THE GSFC BATTERY WORKSHOP

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Goddard Space Flight Center
Greenbelt, Maryland

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THE GSFC BATTERY WORKSHOP

NOVEMBER 19 – 20, 1974

Gerald Halpert
Chairman

November 1974

GODDARD SPACE FLIGHT CENTER
Greenbelt, Maryland
PREFACE

This is a verbatim transcript of the Annual Battery Workshop held at Goddard Space Flight Center, November 19-20, prepared by Ace-Federal Reporters of Washington, D.C. The material covered at the workshop includes a discussion of the low cost/standardization program, test and flight experience, storage experience and manufacturing improvements, materials and cell components, analysis and new developments in the Ni/H₂ system. The meeting was attended by cell and battery manufacturers, users and interested government personnel. A list of attendees appears in Appendix A.
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Each year we take another stride forward toward our objective of improving reliability of the nickel-cadmium cell. Our aim may be directed at the aerospace application but our interest is improvement for every application, including calculators.

To achieve these goals requires communication between manufacturers, users, and government personnel. We believe that the success that has been achieved is due to you, the audience, who has actively participated through your presentations, questions, and comments during our past workshops.

The program for this, the sixth annual workshop, includes a discussion of the new, low-cost standardization approach to cells and batteries. This short discussion will be followed by testing and analysis and other improvements of the cells, and will be topped off by some discussion of new developments in nickel-hydrogen.

We hope that this workshop will be another step forward toward our improvement.

Now, without further ado, I would like to introduce this morning's speaker. To kick off this workshop meeting today we have with us William H. Woodward, the Director of the Space Propulsion and Power Division, Office of Aeronautics and Space Technology. Bill has been associated with the space business for some time, since he joined NACA in 1943. He continued in aerospace when NACA became NASA in 1958, and has been associated with space power since 1961.

Bill is here today to give us an insight into NASA's role in battery development, and to look into his crystal ball and tell us what the future holds. We have asked him to provide the activation energy necessary to get this workshop off to a thermodynamically favorable start.

I am pleased to present William H. Woodward.
NASA's ROLE AND OBJECTIVES: W. H. WOODWARD/NASA-HQ

WOODWARD:  O.K.

With that kind of an introduction, I don't quite know how to start. The right thermal environment is hot air, perhaps. I've got lots of that.

I'm a little shocked at how long I've been around this business, but I find myself in the unenviable position of speaking on a topic to a bunch of experts that I don't know very much about, so let me proceed. I don't think you need much activation but maybe a small prod in one or two directions, and then we will proceed.

Also, let me add on behalf of NASA Headquarters a welcome to you to this annual workshop. We appreciate your making your time available and we hope that you will find the time well spent. For our part, the fact that we keep holding these meetings each year is the best evidence of their value to us. And in addition, I'm assured that the progress made over the years in the quality and performance of rechargeable space batteries is due in some part to meetings such as this.

Of course, sitting in my downtown office and looking at some of the reports and receiving an occasional briefing from Chuck McKenzie and so forth, I don't find the rate of progress to be exceptionally high. No doubt that is because I don't appreciate all the detailed efforts that must be exerted in each task to make it successful.

Be that as it may, I get the impression that I've heard about separators, seals, operational lab quality control problems for as long as I've been associated with the battery program, and that goes back to 1961. It is almost as though such problems are a virus, momentarily and just barely under control, but ready to mutate into a new and a virulent strain at an embarrassing moment. And for my own sake, I just wish that we would be able to rectify this situation.
I've been looking for reasons as to why this situation exists, assuming that my impressions are correct, and I find that one, perhaps the outstanding reason, is the historical precedent.

There were so many crises at the beginning of the space age that firefighting has become a way of life in battery R&D, with little effort left over for agency-wide long-range planning. That's the reason there was a little in-house get-together yesterday afternoon to see whether we could step back from the day-to-day problems a bit and focus on some longer-range goals.

I hope and trust that this workshop would be able to give adequate attention to this subject.

Of particular importance in this regard is the future of the ni-cad program. I understand that nickel-hydrogen and silver-hydrogen cells are becoming fashionable systems to work on. Aside from the fact that we haven't the resources to support everyone working on the same systems, I must ask the question, perhaps somewhat plaintively, what's wrong with the ni-cads? After all, we have years and millions of dollars of hard-won experience with them. In addition, they probably will continue to be the workhorse space battery for many years, and are finding their way into the planetary program.

Finally, advances in ni-cad technology presumably will easily find their way into the ground systems such as the computers as we heard earlier.

I submit to you that it may be worth our while to consider ni-cads from a long-range, non-crisis point of view before hurrying on to the next green pasture.

I mentioned the word "resources," near and dear to every R&D gentleman. And I must admit part of the reason for the slow progress that I'm unhappy about must be the rapid decline of the available funding from perhaps six million in the OASD program at the height of our glory to the current one and a half million per fiscal year for electrochemical R&D contracts and grants. Realistically, I cannot promise an increase. In fact, I will have to say
that all our programs are constantly being screened for possible cuts.

Unfortunately, I must also confess that I and the people around me are running out of ideas -- begging your pardon, Mr. Cohn -- for justifying our battery program. On one hand, not many spacecraft failures can be attributed to batteries. On the other, batteries are not the limiting technology for future missions and it is very difficult to quantify the benefits of improved performance in batteries. We badly need a better justification for our battery program; otherwise, I fear that our budgets will continue to shrink as the competitive pressure for other research areas becomes more intense.

Perhaps this is right and proper. Maybe we have reached the happy point and the batteries are about good enough, but we should not permit it to occur without proper analysis and argument. Any help that you can give us in this regard will be appreciated.

Finally, to not be misunderstood, when I emphasize the ni-cads I did not mean to say that we should not be looking at higher performance systems such as the silver-hydrogen or the nickel-hydrogen. I'm trying to say that we must keep a balance in our program and before we discard something like the ni-cad system I think we should be satisfied that it indeed makes a great deal of sense and is not just for current fashion.

With these few and short words, and I don't know how much incentive I've managed to insert, now let me wish you an interesting and successful time at this workshop.

Thank you.

HALPERT: Thank you, Bill.

Bill said he would entertain some questions from the floor if there were any with regard to his comments. Does anybody want to take a chance?

(No response.)

Thank you, Bill.
III  **LOW COST STANDARDIZATION PROGRAM**

With that we will move on into our next part of the meeting which is kind of an introduction to the new low-cost standardization approach. We will have kind of a brief, three-phase discussion.

Tom Hennigan will bring us up to date on the background of the low-cost program. Flo' r Ford will discuss the present situation in the ad hoc committee and where we are going, and I'll try and talk about the new cell or the cell standardization objectives and approach that we are interested in.

III.1 BACKGROUND: T. HENNIGAN/GSFC

HENNIGAN: In May of 1972, Dr. Low of NASA, who is Deputy Administrator, initiated a program to reduce the cost of spacecraft. This included a cost reduction of component subsystems and total system and test and evaluation associated with the spacecraft; reduction of cost of documentation was also a goal.

Lewis Research Center was also given the responsibility of organizing a power working panel for solar battery and power conditioning committees to determine methods to achieve cost reduction.

Goddard was given the responsibility for the battery work. A committee was formed with the following people: Trout of Johnson, Paschal of Marshall, Kraus, formerly of JPL, Rene of Langley, Miller, Wright-Patterson, Billerbeck of Comsat, and Bill Webster and myself from Goddard.

The first task was to set up a catalog of all types of batteries flown on spacecraft. This would include the design of the cells of batteries, how the batteries were used, and the cost in particular, a breakdown of recurring and non-recurring costs, costs of testing, design, management and so forth.

This type of cost breakdown was very difficult to get.
It was decided that the catalog would not be useful for the purpose of determining and reducing the cost of spacecraft.

A second task was to report to the power working panel a best estimate of cells of battery sizes required, based on the NASA mission model and some Air Force requirements; in other words, should standard cell sizes be recommended for the pre-shuttle and post-shuttle periods.

A study was made by Bill Webster of Goddard to determine the ampere hour sizes of nickel-cadmium cells that had been used in quantity.

May I have the first slide?

(Slide 1.)

On this slide the results of the survey are shown. It covered 120 spacecraft and about 20,000 cells. As you can see, the 6, 12, and 20 sizes were used extensively.

The 4-1/2 ampere hour size is a cylindrical cell used primarily on Nimbus and was not considered to be a standard size.

May I have the next slide, please?

(Slide 2.)

Now based on this, in the nickel-cadmium area we would have nominal or standard sizes of 6, 20, 50, and 100. These would expand in height only or decrease in height to get -- like for a 6, to get a 3 to 8, so your footprint would always remain the same. And say for the 20, 50, and you could go up to 120 ampere hours.

We did recommend some types of designs: for the small size, the box, which has been used a lot for small cells, or the fin, like on SMS; and again on the 20, a nominal size of box or truss-type. The OGO is a truss-type battery.

Then for the 50's we went to a module size and for the 100 we used the four-cell module designed by Grumman for
the space station.

For the silver-cadmium these four sizes have been flown: 3, 5, 10, and 16. And it didn't seem like there was going to be much use for any sizes larger than that, and 10 would probably be the most popular size for the next few years.

The encapsulated design was adequate.

For silver-zinc, it has such a limited use that it did not seem worthwhile to go into a standard design.

For small sizes, at least less than 16 ampere hours, we could use the encapsulated design, the same as the silver-cadmium.

Then after the study, it was required that we come up with some designs for the pre-shuttle range and we kind of did away with the up and down, or the height of the battery and went to four sizes. These four sizes would furnish the cells that we need from 1980 because the highest power range for NASA will be two kilowatts. In the 1980's they are forecasting some large ones of 10 to 25 kilowatts, but we don't know if they are going to go.

So the sizes that we picked were 6, 12, 20, and 50, the latter being optional.

I think Floyd Ford has more to say about these sizes in his talk.

Thank you.

III.2 ORGANIZATION AND GOALS: F. FORD/GSFC

FORD: Thank you.

My name is Floyd Ford. I would like to give you a summary, more or less a debriefing on the current status of the low-cost effort, particularly related to our thrust in low-cost batteries.
(Slide 3.)

After the conclusion of the study that Tom talked about, with certain recommendations, the Low Cost Systems Office at NASA Headquarters was somewhat reorganized and as its present status it has a Low Cost Systems Office with four major standard equipment panels.

The equipment panels are represented by NASA people from the various centers and by Air Force people, at least on some of the panels.

I would like to draw your attention particularly to the power equipment panel which at this time is represented by NASA people only. But under the power equipment panel they have established three ad hoc teams, one for power processing, one for solar cells, and the third one for batteries.

The purpose of these teams is to advise and to consult with the power equipment panel on what work needs to be done and more specifically, how to achieve the goals of reducing the cost of spacecraft batteries.

(Slide 4.)

The objective, obviously, is to reduce the cost of spacecraft batteries, which is only one of the many components of the spacecraft. But to save millions of dollars, you have to learn how to save dollars, and that's where we're starting that here today, is learning how to save a few dollars a day, add on to that tomorrow, and then ultimately reduce the over-all cost of a subsystem.

Now the goal is to adopt a standard family of nickel-cadmium cells. We have chosen the 6, 12, 20 and 50's, and I think the previous discussion gave you an idea of why we have selected those to standardize on.

From that establishing of standard cells we will proceed to establish standard battery packages for that family of cells.

Then we will work on standardizing the charge control
**LOW COST BATTERY PROGRAM**

**INITIAL STUDY**

**NICKEL CADMIUM**

<table>
<thead>
<tr>
<th>CATEGORY</th>
<th>NOMINAL SIZE</th>
<th>CAPACITY RANGE</th>
<th>PACKAGING</th>
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</thead>
<tbody>
<tr>
<td>I</td>
<td>6</td>
<td>3 TO 8</td>
<td>BOX OR FIN</td>
</tr>
<tr>
<td>II</td>
<td>20</td>
<td>9 TO 10.5</td>
<td>BOX OR TRUSS</td>
</tr>
<tr>
<td>III</td>
<td>50</td>
<td>16 TO 80</td>
<td>TRUSS OR MODULE</td>
</tr>
<tr>
<td>IV</td>
<td>100</td>
<td>81 TO 120</td>
<td>FOUR CELL MODULE</td>
</tr>
</tbody>
</table>

**SILVER CADMIUM**

STANDARD SIZES: 3AH, 5AH AND 10AH

ENCAPSULATED DESIGN ADEQUATE

**SILVER ZINC**

LIMITED USE: STANDARD CELL DESIGNS NOT REQUIRED FOR SMALL SIZES < 16AH USE ENCAPSULATION SIMILAR TO NICKEL CADMIUM

**OBJECTIVE**

- REDUCE COST OF SPACECRAFT BATTERIES

**GOALS:**

- ADOPT STANDARD FAMILY OF NICKEL CADMIUM CELLS SIZES (4, 12, 20 AND 30 AMPERE-HOUR)
- ESTABLISH STANDARD BATTERY PACKAGES FOR FAMILY OF CELLS
- STANDARDIZE ON BATTERY CHARGE CONTROL TECHNIQUES
- USE STANDARD BATTERIES ON OVER 80% OF U.S. SATELLITES

**COST SAVINGS BENEFIT:**

- SIGNIFICANT REDUCTION IN DOCUMENTATION ASSOCIATED WITH EACH NEW PROGRAM
- ELIMINATES ENGINEERING DESIGN TIME PRESENTLY REQUIRED ON EACH PROGRAM
- ELIMINATES NEED FOR BATTERY QUALIFICATION ON EACH PROGRAM
- REDUCES AMOUNT OF BATTERY TESTING ON NEW PROGRAMS

**FIGURE 3**

**NASA HEADQUARTERS**

LOW COST SYSTEMS OFFICE

STANDARD EQUIPMENT PANELS

COMMUNICATION & DATA HANDLING

POWER

PROPULSION

STABILIZATION & CONTROL

GROUND SUPPORT EQUIPMENT

**FIGURE 4**
techniques for batteries. And when I say "standardize charge control," this probably is one of the biggest challenges because of the variations you get from cells, and also the variations from manufacturer to manufacturer of cell characteristics.

Of course the over-all goal, and it's a projection, that at least from the NASA viewpoint, that we will probably be able to use the standard batteries in over 80 percent of our missions.

The cost-savings benefit: Well, first of all we're still talking about percentage-wise, but the cost-savings in documentation is a fairly small percentage of the total cost of the battery. Nevertheless, you have to start there in order to achieve a standard battery configuration.

The other thing: It eliminates the engineering design time presently required for each program.

Another potential cost-savings is the need for requalification.

The concept is simply this: Once we have built a battery and qualified it to an over-all signature level, both mechanical and thermal, the signature level that we choose will be one that will encompass the requirements of a multitude of launch vehicles including the shuttle, because we are not just talking about the pre-shuttle era but we're talking about the post-shuttle application, too.

Along these lines, we have had some discussion recently on the man rating aspect of nickel-cadmium batteries to be flown or at least be handled in the payload area of the shuttle. This is an area we still have not certified significantly to our satisfaction yet, but the indications are now that the type of batteries we have been flying on the scientific series of satellites, namely hermetically sealed units, we may be looking at pressure relief configurations either on a cell and/or the battery level.

Now of course the follow-on which may result in the
most significant cost saving is the reduction in amount of battery testing that is associated with each new program.

Now I stand here before you and admit this is an ambitious program but I also have to make you aware of the fact -- something you are well aware of, I'm sure, that with the cost escalation that is associated with everything today we have got to do things that we have not done in the past. We have got to be able to get more effective use of our dollars or else we are going to cost ourselves right out of the aerospace business, and along with that we will cost ourselves out of a job.

(Slide 5.)

I would like to show you the present membership of the ad hoc battery team, and I have a main objective in doing this. One is to familiarize those who are not aware of the existence of this team, and so also give you a point of contact for any input you may have, or any suggestions you may have along the low-cost standard battery approach.

For the sake of the record I will read the names: Tom Hennigan, Goddard Space Flight Center; myself, Floyd Forc; John Rubencer, Ames Research; Uchiyama, from Jet Propulsion Laboratory; Paschal from Marshall; Barry Trout from from Houston, Johnson Space Center; Jim Been from Lewis Research Center; James Bene from Langley; Bill Nagle from Lewis; Gerry Halpert from Goddard.

And the Air Force participation is Captain Straton, Dave Pickett, Robert Kerr, and Harry Killian from Aerospace.

At this time Gerry Halpert will give you the status of the current, on-going program, and define some of the most -- what we call the intermediate objectives of the next couple of years.
III.3 APPROACH/OBJECTIVES OF 20ah CELL PROGRAM:
G. HALPERT/GSFC

(Slide 6.)

In terms of our standard 20 ampere hour cell, we call out our objective to produce a standardized design 20 ampere hour cell with a specific envelope configuration. What we intend to do is to establish one MCD manufacturing control document with each manufacturer so that we can refer to that one document to purchase all of our 20 ampere hour cells. We don't want to change the design every time we buy a new cell, so we are going to work it out so that there is one MCD with each manufacturer.

The design will be for a standard family, although this procurement will be only for the 20 ampere hour cell; that is, the present design. We would like the MCD to serve as a link toward designing the standard family as well so that a minimum change in the MCD would be required to change the family or to change the cell size.

We would like to be able to have the cells in a package, an envelope that would fit into a standardized battery package; that is, no matter what manufacturer we buy from, that cell will fit into a standardized battery.

One very important goal is to have reproduceable cells from each manufacturer so that we again, buying one this month and buying one next month or next year, we are fairly sure or we would like to be assured that we get the same product with essentially the same characteristics.

That is, reproduceable cells from lot to lot is the important thing. Obviously, we don't expect the manufacturers to be able to give us the same properties as other manufacturers. Each would hopefully have each characterized so that we would know what the manufacturing characteristics are for the nickel-cadmium cell.

At the very end we want to develop a source
control document for identifying qualified suppliers, and salient characteristics of each supplier's cell. This would be a document like a mil. spec where we would buy a 20 ampere hour cell to this mil. spec or this SCD, we'll call it now, for NASA's use. And that SCD will describe the manufacturers who can produce this cell of the quality we want, that we would like. And then it would be up to the procurement people to handle it from there.

(Slide 7.)

Our approach is two-phased. First, documentation; that is, to produce a MCD which will meet the requirements that we would like to see, all up-to-date specification and documentation, tests on quality and so on, based on the present 74-15,000 specification that we have.

We will be very happy to throw out that 74-15,000 specification, and I'm sure many of you feel the same way, if we have an MCD we can rely on. We don't want to use our spec; we want to use your spec, but we want you to stick to it. That's the approach there.

The second phase will be hardware to produce 20 ampere hour cells based on an MCD, the number to be determined some time later. But the idea is now, once you've shown you can have the documentation, let's produce it to that documentation.

(Slide 8.)

The specifics of the documentation would be to have in there the cell design, the assembly instructions, the testing, the inspection, the drawings and document control for those drawings so that we don't change drawings or modify them without adequate knowledge or description.

The data sheets ought to be in there, the quality assurance provisions, and what we think would be a good step is provisions for a data log in which all data for the cell would go into, and that would be delivered at the end of the program. In other words, we don't want loose papers, we don't want loose sheets. We would have a log
AD HOC BATTERY TEAM MEMBERS

NASA
THOMAS J. HENNING
GODDARD SPACE FLIGHT CENTER
FLOYD L. FORD
GODDARD SPACE FLIGHT CENTER
JOHN RUBENZER
AMES RESEARCH CENTER
R. A. UCYTIIIA
JET PROPULSION LABORATORY
L. E. PASCAL
GEORGE C. MARSHALL
BARRY TROUZ
JOHNSON SPACE CENTER
J. BBN
LEWIS RESEARCH CENTER
JAMES RHEE
LANGLEY RESEARCH CENTER
WILLIAM N. NAGLE
LEWIS RESEARCH CENTER
GERALD MALPOTT
GODDARD SPACE FLIGHT CENTER

AIR FORCE
CAPT. J. R. STRATON
SAMSDYLEX
DAVID F. PIETI
WRIGHT-PATTERSON AIR FORCE BASE
ROBERT L. KEEF
WRIGHT-PATTERSON AIR FORCE BASE
HARRY KILLIAN
AEROSPACE CORPORATION

FIGURE 5
STANDARD 20 AH CELL

OBJECTIVE
TO PRODUCE A STANDARD DESIGN 20 AH CELL WITH SPECIFIC ENVELOPE CONFIGURATION

GOALS
1. ESTABLISH AN MCD WITH EACH MANUFACTURER THAT WILL SERVE AS A STANDARD FOR FUTURE CELL PROCUREMENT
2. STANDARD FAMILY OF 6, 12, 20, 50 AH CELLS
3. INTERCHANGEABILITY OF CELLS IN A STANDARD BATTERY PACKAGE
4. REPRODUCIBLE CELLS WITHIN ONE MANUFACTURER
5. REPRODUCIBLE CELLS FROM LOT TO LOT
6. DEVELOPMENT OF A SOURCE CONTROL DOCUMENT (SCD) FOR IDENTIFYING QUALIFIED SUPPLIERS AND SALIENT CHARACTERISTICS OF EACH SUPPLIER'S CELL

FIGURE 6

APPROACH

• PHASE A. DOCUMENTATION
TO PRODUCE COMPLETE, UP-TO-DATE, ACCURATE MCD BASED ON THE GSFC 74-15000 SPECIFICATION THAT WILL BE CONSIDERED STANDARD MCD

• PHASE B. HARDWARE
TO PRODUCE 20 AH CELLS BASED ON THE STANDARD MCD THAT WILL BE CAPABLE OF ASSEMBLY INTO A STANDARD BATTERY DESIGN

FIGURE 7

PHASE A. DOCUMENTATION

THE MCD IS TO INCLUDE:
• CELL DESIGN
• ASSEMBLY INSTRUCTIONS
• MECHANICAL & ELECTRICAL TEST PROCEDURES
• INSPECTION PROCEDURES
• DRAWINGS AND DRAWING DOCUMENT CONTROL SYSTEM
• DATA SHEETS
• QUALITY ASSURANCE PROVISIONS
• PROVISIONS FOR DATA LOG

FIGURE 8
which starts with the very first certification by the vendors that the manufacturers deal with, down to the final testing that the cell manufacturers do.

(Slide 9, 9a, 9b, 9c.)

The size envelope for the 20 that we're talking about looks like this, and I won't bother to read all those things off. I think you can see them. This will be in the minutes.

We notice down here to the left I have two requirements listed, or three requirements: that the minimum capacity be 20 ampere hours, maximum 24. We'd like to standardize on something we can keep within certain limits and not put in extra or additional loading that we really do not desire.

Again, a maximum weight here would be one kilogram.

Tom has worked up not only this one but the cell dimensions or the envelope for the 6, the 12's, and the 50's, and we would hope to be able to put this in the document even though you don't see it here today so that you will have something to work from in the future.

(Slide 10.)

In the hardware, in the phase B section where the cells are manufactured, we're going to implement a buy-off review. This is a review after plate tests are done; after the manufacturer has procured his basic materials, the separator and cans and so on, we would have a buy-off review to see whether the materials used in the cells will meet the design that he has said he is going to meet; that is, is the capacity right, is the loading right, is the separator right in terms of its characterization and so on. So here we would buy off the components essentially before we would allow the cells to be built.

The samples would follow and then we would go along in the usual way with all the manufacturing and the testing, including some vibration tests.
Finally, the delivery of the data log with the cells and then a chance for the manufacturer to submit finally an MCD which we can call the right MCD.

I guess one of the things I have not mentioned is the fact that there would be three MCD's, two that NASA would get and one that will be maintained by the manufacturer himself with all of the proprietary documents. That MCD, the master MCD we'll call it, would stay at the manufacturer's facility and not be deliverable. A suitable NASA official -- a designated official would have access to that MCD, and anyone else whom he and the manufacturer recognize as a qualified individual.

Therefore, the manufacturer would not have to give out any proprietary information that he desired not to.

The two deliverable MCD's would be those in which the proprietary documents were called out with their dates and revision numbers, but that would be all.

(Slide 11.)

The concept in reviewing again the MCD's based on the 74-15,000 spec: The maximum use of existing design documents and drawings is not a development. We don't want to start from the beginning. This is what you have already, what the manufacturer has already, and then modified or revised to meet the present design.

There will be no exceptions to the spec unless satisfactorily identified, and the proprietary documents will be in the master MCD, again access to that through a Goddard representative. And all the future procurements will be based on that standard MCD.

(Slide 12.)

Finally, when we talk about using these cells now, we have cells in-house and we want to build them to the standard battery, a motherhood statement with regard to standardized battery considerations because we really have not finalized these at all.
It includes consideration of cell-to-battery weight ratio, the ease of manufacture of the battery, the ease of assembly and disassembly, the complexity of the system, the thermal design, the mechanical design, and I guess I could go on with several more, but these cells will be used in the standard battery program, the standard battery design which will come at a later time.

(Slide off.)

That really concludes the discussion of the low-cost program but I'm sure Floyd, Tom, and myself would be very happy to answer questions with regard to this if there are any at the present time.

CORBETT: I guess I have kind of a loaded question, Gerry. That is: Do you have any plans for specific battery mechanical design which will include requirements on the cells? The first thing that comes to mind is some of the things that really crank the cell design requirements are the battery package, the pre-load design, and then you have to have enough strength in the side walls so that the cells don't buckle under the pre-load.

For this reason, for example, you have 6 ampere hour cells that can be bought with anything from I guess 19 to 30 mil can thickness. If it's a welded can it can be pretty uniform. If it's a drawn can it is kind of thick on the edges and that gives you more strength. You know, there are all these kinds of interface considerations that tend to relate to better mechanical designs.

So could you comment on that?

HALPETER: With regard to the mechanical design of the cell, we'd fix on one design and allow for certain modifications in the can wall which is, of course, not an electrochemical problem but a mechanical problem based on what a manufacturer or what a battery requirement is.

If it is for a vibration problem where the cell has to be thicker, or some kind of g forces, then that would be -- certainly a modification would be involved.
What we don't want to do is to change the loading, change the plate, change the structure of the cell so that the characteristics are different. That's what we really want to be the same.

CORBETT: This is a critical cost element though because one of the things I guess that really hits the manufacturers is having to keep an inventory or else to order so many different kinds of cans, styles and terminals and that sort of thing, both welded and drawn. And it is not only a cost problem but a schedule problem, too.

FORD: Yes, Bob, your question is very relevant and it is one that we have looked at and are still looking at.

Specifically, the answer to your question is that the standard cell will be just that. It will be designed to accommodate all the environments that we can anticipate for it for a large percentage of satellites. It will not be, necessarily, designed to accommodate a particular -- like an impact design for lander -- you know, high g levels.

We are trying to achieve a high percentage of use of the standard battery.

Now the standard cell will be as the manufacturer proposes. I would like to make a point though about this weight.

We have had three meetings of the ad hoc battery team. At the last meeting we reviewed-- In a two-day session we reviewed at least a dozen different battery designs that have flown or at least are going to fly within the next two years, but most of those we have had considerable experience with.

It is my honest opinion that some of those batteries could be dropped out of a C-5-A and land on the ground and still stay intact, and I'm talking about from a considerable altitude.

I think that we have to optimize the mechanical design but it was the general consensus of the ad hoc battery team that while weight should be a consideration
in the battery design, it should not be given the primary consideration.

Now we're faced with this philosophy that things that fly on the shuttle will have a weight problem. I don't think that we can live by that real hard. I think that we have to consider optimizing weight but yet not cutting down weight sufficiently that we may have a marginal design for a good percentage of our applications.

I guess one other point.

Bill Limberis, will you stand up?

He is the Program Manager for Power Subsystems, Low Cost Systems Office at NASA/HQ.

One other point about this: The mechanics of implementing the standard design is still in the mill. In other words, we come up with the standard configuration of the cell. Gerry has mentioned the SCD. Ultimately we will have something in the follow-on that will define the standard battery packages.

Now the project -- I'm speaking for NASA, not the Air Force. The NASA projects will be required to review the standard component list and I am now not talking about just batteries. You saw the other panels that are working there, and there are other groups that are working on standards like the standard tape recorder, the standard spacecraft computer.

The future projects will have to show cause why they can't use the standard configuration, whether it be a battery, a tape recorder, or a computer, or a propulsion system. The incentive for the projects to use the standard components is primarily the cost factor, that they don't have to pay for the development.

If they show an incompatibility with the use of any of
these standard components, then the project will have to absorb the cost of either modifying or whatever they require, or even going out and maybe developing a new one in order to meet their needs.

Bill, do you have any other comment along those lines?

LIMBERIS: No, I think you summarized it pretty well.

There will be a catalog of standard equipment which by direction of Dr. Low Deputy Administrator will be used by every project unless a waiver is requested. According to what Floyd has been saying, we're trying to develop a standard 20 ampere hour cell that any project, by accessing our catalog system, can procure and be sure of getting the same cell each time.

GANDEL: I'm Marty Gandel, Lockheed.

Floyd, if you look at, say, the 20 ampere hour cell, what's the projected usage that is— How many programs do we have to look forward to in the next, say, five years or ten years, if you want to restrict it to NASA programs, if that's easier, that would use the 20 ampere hour cell?

Let's just take a number. Maybe there are 10 programs, 20 programs, whatever it happens to be. Now with that you associate a certain amount of non-recurring engineering that would be spent on that program if you didn't have the standard cell.

Now working with the so-called standard cell, can you quantify the amount of engineering that would be saved?

Now what I'm looking against, and this is being hyper-critical— I'm trying to be very critical of it. If we look at the list of people on your ad hoc committee, there are no manufacturers that aren't the major users, these people in the government agencies.

And so I toss one back to Mr. Woodward now:

When we look at the over-all economics, we have the same number of people employed in the same number of
agencies overlooking all of these things, even though the number of programs goes down. Now at some point there is a crossover between the staff costs and the "cost savings" programs against the cost of those programs if you didn't have cost savings.

I think somewhere you've got to make a systems analysis and see what you're getting for the effort.

I've made kind of a speech out of this thing, Floyd. Does it make sense?

FORD: It certainly does.

Well, I'm not sure exactly what the question was but I think I understood your statement very well.

I have to point out, as I think I did a little earlier, that the cost savings that we're looking for isn't necessarily with the standard cell, particularly the standard 20. We're looking at the total program as far as the overall cost savings. And quite frankly, if I had to stand--I could not stand here and justify the program, based on the 20 ampere hour cell cost savings alone. It's the total picture, including all the way through to delivery of batteries to the spacecraft.

But in order to get a standard battery you have to start with the cell, with some defined footprint. And the reason we chose the 20 is because looking at NASA's productions and looking at the projections the Air Force has provided us with, is that it is going to be one of the major users. That is the first cell. That doesn't mean we're going to stop there.

We hope to, immediately thereafter, proceed to develop the 6, 12, and even the 50.

Now interestingly enough, the 50 ampere hour sizes, as far as the NASA projections are concerned, in the next ten years still will be few in number. Their projection of use is predominantly in the 12 to 20 ampere hour. And I think if they look at the -- what do they call that? --
the mission model that NASA has made up, and look at the types of spacecraft, the power requirements, you're talking about power in the range of a half a kilowatt to a two-kilowatt system. But there are still a large number of spacecraft down in the 100-watt range, the 100- to 200-watt range.

But the best data we have says that the 20 will be the biggest user for NASA in the next five years, and I believe that is consistent with the Air Force projection. However, their projection shows use of the larger cells; that's the reason the 50 is included in the program.

WOODWARD: If I might get at it from a different angle, I sympathize with your comments that maybe we've got too many people beating a small problem to death, and maybe we should go back and look at our balance of people.

The comment that we do not have industry people on the panel and we need that input is well-taken. There is another angle I would like to just suggest.

I said earlier that these problems of quality control and seal problems and operational problems keep turning up and they cost us money. Now if we can find a way to get to automation or at least making the same doggoned thing the same way each time with a high degree of confidence, I think it would help. Let me draw an analogy. Every time I do I get in trouble but I'll try it again.

The American automobile engine is a pretty good engine as far as the engine itself. I don't mean the accessories. When I was young and gay, yes, we overhauled engines at 40,000 miles and we were using two quarts of oil and so forth and so on per thousand.

I don't think you'll find, if you think about it, very many modern automobile engines that are giving any internal troubles with the main power plant like that, upwards of 100,000 miles. So I got a little interested to find out why this is so. And at least one authority tells me that this is because they've gotten the man out of the production process. Those engines are made by automated
machinery. They make them to close tolerance. When they are through, the machines grade each part for dimensional tolerances and they just don't stay with the tolerances; they match each part to each part as best they can, all done automatically. The end result is the machine is a remarkably consistent beast.

I'd like to see, or would suggest that maybe as a part of this standardization effort an unexpected and hopefully useful offshoot will be standardization in the manufacturing process that will give us a better and more uniform product.

It's just a thought, but I think we need it. Again, let me reiterate it seems about other year in a program review somebody stands up and says "Hey, we've got quality control problems, and this must be costing us money. It must be costing us time and effort."

DUNLOP: Dunlop, from Comsat.

Bill, I don't know what your age is but what would happen if they were to have standardized the engine back in 1928?

WOODWARD: I don't know, sir. However, we've been at the ni-cad business since 1960-something-or-other. And if you look at the progress that's been made, we've gone from a life of a few months up to like five years and the watt hours per pound have increased.

HENNIGAN: The usable has increased.

WOODWARD: Yes, the depth of discharge has increased and I think we're getting in the ni-cad business to a state of maturity that perhaps is analogous to the automobile engine in the late '40's and early '50's when they started trying to automate.

DUNLOP: That's probably the area where the question lies. If you standardize the separator, for example, and it's a bad separator, you still have a bad separator.

WOODWARD: Okay. I don't think we would have made as
much progress as we have—Although I keep insisting it is not fast enough, I don't think we would have made as much if we didn't have fundamentally a decent battery.

HAAS: Haff, Philco-Ford.

Floyd, I've been wondering what degree of cost analysis has gone into the justification for standardizing cells and batteries. Are we talking about $20,000 for ten programs, a few hundred thousand dollars in the next five years? Or are we talking about $200,000 per program for ten programs?

HENNIGAN: I've got a slide here I'll show you.

(Slide 13.)

Now these are some battery costs. Now we have to watch out how we use these numbers because, as was said before, it is very difficult to get costs on batteries, on battery programs.

The people who are familiar with these programs can correct me if I'm wrong but the Mariner/Mars, that consists of ten batteries.

The Mercury one was three.

OAO was three batteries in two packages.

OSO-I -- Do you remember how many batteries that was?

WEBSTER: You mean actually flown?

HENNIGAN: Assembled and tested for the program.

WEBSTER: Approximately five, I think.

HENNIGAN: Okay.

The Air Force project was 17 batteries.

Project B was 27.
PHASE B. HARDWARE

THE PRODUCTION OF 20 Ah CELLS MANUFACTURED TO THE
STANDARD MCD SHALL INCLUDE:
- Buyoff Review
- Submit Samples
- Flooded Cell Tests
- Cell Manufacture
- Cell Electrical Test
- Cell Vibration Test
- Delivery of Data Log
- Delivery of Cells
- Final Revision of MCD

FIGURE 10

CONCEPT

- Use 74-15090 as basis for MCD
- Maximum use of existing design documents and drawings
- Exceptions to spec allowed is identified and satisfactorily defended
- Proprietary documents will be included in the master MCD to be held at manufacturer facility
- Access to master MCD through GSFC authorized representative
- Future procurements will specify the standard MCD

FIGURE 11

STANDARD BATTERY CONSIDERATIONS

- Cell/Battery Weight Ratio
- Ease of Manufacture
- Ease of Assembly & Disassembly
- Complexity
- Thermal Design
- Mechanical Design

FIGURE 12

BATTERY COSTS

<table>
<thead>
<tr>
<th>Project/Program</th>
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<tr>
<td>Mariner Mars 71</td>
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<td>Mariner Venus Mercury</td>
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<td>Project A (USAF)</td>
<td>759K</td>
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<tr>
<td>Project B (USAF)</td>
<td>931K</td>
</tr>
<tr>
<td>IMP H</td>
<td>14K</td>
</tr>
<tr>
<td>RAE</td>
<td>20K</td>
</tr>
</tbody>
</table>

FIGURE 13
IMP-H, that's the cost of one battery.

And RAE, that's the cost of one battery.

And those last two are to launch. Some of the other ones I don't know if they include the integration costs on some of these things.

But I did look at the cost of man-hours to make a design, do the drawings, assemble the battery, vibrate it, thermal vacuum it for a couple of weeks, and do some other electrical testing. I can't remember the numbers but if you have all that stuff available, the drawings and the design, you don't vacuum it and you don't vibrate it and you do minor electrical test after you build the battery, it was a cost reduction of about 45 percent, and I think that was the biggest area of cost reduction.

I know an awful lot of electrical tests, vibration and so forth, are done on batteries, but it seems if you use the same design over and over again you have some confidence in it. For instance, on the IMP space batteries, we've made those so much, we put them together and capacity check them and that's it.

WEBSTER: Tom, was that the extent of the cost analysis?

HENNIGAN: Yes.

WEBSTER: Okay. Thank you.

HOWARD: Let me ask you from your experience, what's the most costly element of the battery as far as your experience, if you have to single out one area?

WEBSTER: The most cost of the cost of the item is the testing, the testing of the cells, the testing of the battery, and the tabulation and the gathering of the data, the log books the vendor -- the manufacturer's testing and software involved in the program.

FORD: Would anyone else like to comment on that question?
LACKNER: There is one comment I would like to make.

It seems that the manufacturers have said that making batteries for satellites is small volume, low cost to them in their total production line, and it is a bit of a nuisance.

Now can we guarantee that we will be buying the number of batteries to maintain a production team year-round so they do qualify people to make it, that the quality control will be consistent by saying "Okay, we have ten people in the plant who will work on nothing but aerospace batteries and they'll have their heart and soul in it," instead of working on it for a set program and then another group will work on it two years later.

There is where your inconsistency in quality control will come out.

FORD: That's like asking me if I can guarantee the space program is going to continue for ten years.

No, I don't think-- I want to make a point to your question. It is simply that we will not have any change from the status quo as far as those battery teams at the various -- or the production teams at the various sources that make these cells today.

LACKNER: But if they have to pay these people and they are not working on a set program, they are liable to let them go over and put them on something else.

FORD: But now does this standardization change the present status quo? That's what I fail to see.

LACKNER: You're standardizing on components and you are either saying that we've got the process under such control that any human operator can work on it, which is what I believe the prior comment was.

We do know that quality control does make a difference. If you can get the same team working on it all the time they will be looking at matters that may not be documented just by experience.
WEBSTER: I would like to comment on why the cost of the IMP battery is so low, 14 K. Not only do we just capacity check it but we do put it through a subsystem thermal vacuum and magnetic acceptability test and we’ve used the standard document for this for about six years now, and that’s, I believe, one of the big cost savings.

We spent approximately a week going through magnetic measurements, thermal vac and vibration at the subsystem level, but the cost of that is only $2,000 because there is no documentation required. It exists. And this is the principal cost, I think, of standardization.

Every time you talk about overseeing by the government, re-writing by industry, we’re generating new specifications and new documents for every single battery. And I really feel that an immediate cost savings would be in that area, and we’ve demonstrated it in-house.

KRAUSE: Krause, from Hughes.

Floyd, I don’t know if this is the right place to ask the question but some of the industry people are good-sized users of boosters which are supplied by NASA and I was wondering how much pressure or filtering down of this drive for standardization of some of the components might NASA exert on the users of the boosters to try to get them to optimize their savings, make them simpler, or in some way provide a better use of the boosters.

FORD: I’m not aware that there will be any pressure exerted by NASA from the Low Cost Systems Office on people that use—You’re talking about people who use the Delta, this type vehicle, to put up commercial satellites?

KRAUSE: Yes, or Centaur or any others.

FORD: I’m not aware where there will be any impact at all on the commercial aspect or the non-NASA users of the launch vehicles.

MICKELSON: Mickelson, of Convair.

...when you selected your various battery sizes for your
standard program, did you consider the reliability of the various sizes? In other words, is one size more reliable than the other, or what have you?

FORD: No. Those size selections were based predominantly on projections of spacecraft power system requirements and the ampere hour capacity or the watt hour capacity required to fulfill those requirements.

Speaking for myself, I'm not aware of where we have data that shows one size cell or one size battery, restricting that to the prismatic design, is more reliable than another size. For instance, I don't know that anyone has shown that a 20 is more reliable than a 12, or vice versa.

I. M. SCHULMAN: Schulman, TRW.

Floyd, are you going to factor in life requirements into your studies, the very fact that you have some vehicles that have a requirement of six months and other vehicles five to seven years?

FORD: Only to the extent that the standard battery design will have specified certain guideline parameters for its use. For instance, if you have a 20, you have a six-months mission which-- Incidentally, they are few and far between these days, but using that for an example, if you have a six-month mission versus a two-year mission, then the user obviously would be able to take more advantage of that size and the usable capacity. He could get six months versus two years.

So the answer to your question is no in one respect, yes in the other. But bear in mind this end product is still to be used by the various people in the aerospace industry.

I will mention again and encourage you to contact the members on the ad hoc battery team for any and all inputs in this area of standardization.

BARNETT: Barnett, Rockwell.
I'm not encouraging it but has NASA given any consideration to GF-ing cells?

FORD: Yes, and the answer is no, we will not. That's the purpose of the SCD.

WOODWARD: Can I ask another question maybe of the industry people, and I think the NASA people will shudder, but let me ask it.

You raised a point that our production requirements are so low it is not worth the time, trouble, and effort on the part of industry. Fine, if that's so. Okay, can we use your standard equipment you're producing for ground use? Why can't we use it? Is it not of good enough quality now to meet our needs? If so, what can we do about improving it?

Surely it would help you in your end of the business in selling things on the ground.

This is an area that we've been wondering about for quite a while. Why can't we make better use of the available -- let's call them civilian ground cell technologies.

FORD: Any questions or comments?

CORBETT: Corbett, Lockheed.

I was going to comment originally when Joe Lackner made the comment about battery standardization. I think the point he had in mind was when you go to a cell vendor and ask him to build a battery, the first time around they'll say "We don't like that because it is not a high-volume item for us. We don't have the specialists in-house."

My comment to Joe was going to be if it's a pain for the three or four ni-cad manufacturers who are around, it is even more so for the 15 or so contractors who make batteries. It may be a once-a-year thing.

I think it would be valuable if we could get a couple
of the battery vendors fully into the standardization program, not only for cells but for battery assemblies because I think there is a volume there.

We have been producing at Lockheed silver-zinc battery cells for boosters and limited-life spacecraft, and Marty Gandel probably has the figures on the top of his head, but there have been hundreds, probably thousands of batteries produced and at this point it is probably as standard a battery as you're going to get in the space business. It is so standard the battery people don't get involved with procurement or the tests. In many cases, the thing may go right down to the base for integration and in fact, as recently as I guess the late '60's, we were even doing this with the ni-cad cells and batteries.

But I think what is a real question to ask here if one is a customer, are you going to allow these things to be done?

We already have a military customer who does allow it to be done. He says "Okay, if that's what it takes to make a low-cost battery, then I'm willing to say that I don't want to look at the battery data or I'm not going to put my consultants on your deal."

I don't think NASA is anxious to put out that kind of a policy which would enable the industry to respond to it with a low-cost battery.

FORD: A good point.

Any other questions or comments?

MICKELSON: Mickelson, Convair.

In your committee how closely are you working with people who generate environmental requirements, for example, vibration requirements, because so often they come along with a design requirement and you pick out this part and you test it and it's all qualified. Then you look at the vibration requirement and it is under the scope of the new vibration requirements.

How closely are you going to attack that? That means
you are going to have to turn around and test that.

FORD: We have mechanical people as well as people from T&E, the Test and Engineering group here at Goddard that look at that.

It is interesting to note that to contrast the difference from the military requirement -- and correct me, John, if I'm wrong here -- and the Air Force requirement -- military versus NASA, NASA requires usually a random and sinusoidal vibration test on practically every subcomponent and I believe currently you only require --

STRATON: We require both.

FORD: Okay. I thought you only required random.

STRATON: We require both random and sinusoidal. It depends upon the program in some cases.

FORD: The area we will be scrutinizing the hardest will be the testing. After all, if we have made any progress, and I believe that we have in the last five years or the last ten years in the quality of the cell, then once you have sealed that cell you either have a cell or you don't. And without the benefit of a valid accelerated test technique at this point, the additional testing you can do on the battery, from my viewpoint, is strictly a workmanship testing, not just the cells but a workmanship test primarily of the battery fabrication.

I think from that viewpoint with the good high confidence level in the discrete cells that you procure, I think you can seriously consider reducing the amount of testing at the battery level and simply make it a workmanship test environment.

HENDEE: We're talking about lowering the cost of cells as far as commercial satellites go. Probably my break-even point would be for every additional year of life I can get out of the battery I can spend a million dollars for that battery.

You figure out the vehicle, the launch vehicle, the
satellite cost and, say, project it on a seven-year lifetime. It doesn't take too much to figure out--It's better than a million dollars a year for better battery tests. I think it is probably the limiting life at the moment of the commercial spacecraft.

FORD: I commented in a meeting we had yesterday, an internal meeting, that typically our experience is with the scientific theory satellites where they are experimental in nature that the batteries usually are a very minor problem during the life of those scientific satellites, simply because the experiment usually is of an experimental nature.

However, we are also dealing now in quite a volume of operational satellites, namely, the weather satellites, so life is becoming more and more important. and the experiment or the device being flown is still called an experiment but it is no longer an experiment; it's a proven device. It has been flown on one of the early Nimbus or on one of the other experimental spacecraft and they know it will operate for a long time.

Now in those programs we are beginning to see that the batteries are showing significant deterioration. I'm talking about three-, four-, five-year satellites, not two-year satellites. But the batteries are showing as a weak element in the system simply because, unlike the experimental series, the operational satellites are designed to operate for several years.

I can honestly say that I am not aware of any failure that we've had of an operational satellite because of the batteries. We've had some deterioration, very obvious deterioration because of battery performance after about four years.
For our next session on Test and Flight Experience Bill Webster of our Electrochemical Section will be the chairman. I introduce you to Bill Webster.

WEBSTER: Good morning. Thank you, Gerry.

Now we move from the future and philosophy into the present and facts.

This section will be on tests, their results, raw tests, and some flight experiences. It will go on after lunch. We've had some additional speakers. We will have a total of eight speakers. The topics to be covered I will enumerate and they will be in the following order:

State of charge by phase-measuring techniques, by Ed Dowgiallo from Fort Belvoir;
A short test by Sam Bogner;
Improvements in short testing by Dr. Scott of TRW;

Accelerated test program currently underway at Crane will be presented by Don Mains of the U. S. Navy Ammunitions Depot at Crane.

I will present some in-house characterization of the OSO flight cells.

Then we will have the effects of reconditioning presented by Irv Shulman of TRW.

Floyd Ford of Goddard will be presenting a paper titled "Power Module Development Using Third Electrode Control."

So without further ado I would like to introduce our first speaker, Ed Dowgiallo of Fort Belvoir on phase measurements and state of charge.
IV.1 PHASE MEASUREMENTS AND STATE OF CHARGE:
E. DOWGIALLO/F. BELVOIR

DOWGIALLO: What we have tried to do is to relate some electrical parameter to state of charge. Since many of the batteries in the Army ground paravehicle propulsion applications are found to be sealed and only accessible at the two terminals, a means of automatic and continuous monitor of electrical parameter-related state of charge is very desirable.

An analog signal proportional to this parameter would be useful and a field gauge as a means of ending charges as an early diagnostic for possible detrimental changes and for various control schemes.

Previously, to measure battery complex impedences, a bridge circuit was nulled to various AC frequencies during DC discharges typical for an application. Parameter failures were calculated for an assumed equivalent circuit based on the bridge setting.

Measurements were time-consuming and difficult to automate.

A new unit that measures gain and phase was selected to overcome this. The unit is a Hewitt-Packard gain phase meter model 3575-A. It does not require frequency tuning or amplitude setting. Display resolution is .1 of a degree. The first slide, please.

(Slide 14.)

This is a block diagram of the circuit we used. What we have here is a charge loop and a discharge loop, and we're programming a .5 amp RMS current from the battery which is to say to and from the battery at all times. That's generated by programming this programmable load bank with a sine wave.

The charge supply charges the system at a current level determined by this programmable load bank which operates at a constant current and load.

In discharge this programmable load bank is in
operation, programmed by a discharge current level which is added to the sine wave.

We have passively coupled amplifiers here, one on the shunt monitoring current, the other monitoring AC voltage across the battery, and these are fed into two inputs, A and B, and then they are converted to phase and analog signal proportionate to the phase angle so that we could record continuously the phase angle change. And also we can record even the amplitude of A or B on the data acquisition system.

The phase lock generator was used because the principle of operation of this particular gain phase meter, it takes its readings about zero crossing point, so most noise seems to be in that region and it seems to generally sink with the line.

So what we did was use the phase lock generator such that our readings were not taken where the noise was. We can shift it relative to the line.

The batteries that we used in this were what I guess are non-standard 15 ampere ni-cads, high rate types.

Our purpose here was not so much to find out if this would work with ni-cads. I just used the ni-cad batteries because they were convenient and relatively inexpensive and they could sit around the lab for a while in lulls, because this was the Director's in-house project, and would not be harmed.

Our purpose here was to see if this would work with lead-acid cells after we worked out the technique with the ni-cads, and then to go to some molten salt cells. We just briefly touched that as a matter of interest to this group, I guess.

So what we actually did then was do measurements on three systems, the ni-cads, the lead-acid, both the SI starting light ignition automotive type and the fork lift type, the fork lift battery type, and molten salt cells that we have a contract with the Sohio Company to produce. These are a lithium-tellurium-tetrachloride
system.

Slide two, please.

(Slide 15.)

This shows the phase angle change during charge versus the amp hours at 12, 60, and 400 hertz for four ni-cad cells in series at 1.5 amps for 13 hours. At 60 hertz the change can be seen to be fairly linear in the region after about three amp hours.

Slide three shows the discharge.

(Slide 16.)

This is the phase angle change versus discharge amp hours at three frequencies for four ni-cad cells in series at 7.3 amps to one volt per cell. We went to one volt per cell to see what happened when we went beyond 1.1.

And you can see at about 15 amp hours what happens when you approach or go through the knee.

It is interesting here I guess to note the 60 and 400 hertz almost have a straight-line relationship.

Slide four, please.

(Slide 17.)

This shows the phase angle versus amp hours discharged at 50 hertz for four ni-cad cells at three currents. That is 4.4 amps.

Note the knee at 14 amp hours again. It slopes very slightly with the discharge rate. However, the phase change per current level is approximately linear at .25 degrees for 5 amps.

These effects could be compensated for in a fuel gauge over a wide range of discharge currents.

Slide five.
This shows the overlap of charge and discharge as well as the linearity for two ni-cad cells in series at 1.5 amps. And you can see on charge we go up this line here and on discharge we start up here because of the inefficiency of the cell, and we come back down here, so they overlap fairly well.

This is amp-hours in, and coming right back down, amp-hours out.

Next slide, please.

One comment, too. I guess we were wondering about what would happen on open circuit stand times for this type of measurements and we let it stand at zero, three, and 20 hours, and we noticed that generally there might be about a .2 of a degree shift in the beginning of charge.

And we did not test it extensively but it seems that the current level has an effect on this.

Phase angle change versus discharge amp-hours out per cycle for two ni-cads at 1.5 amps at charge and discharge. We charge at 1.5 and discharge at 1.5. This is the lowest rate recommended by the manufacturer. These were done at 40 degrees Centigrade to 1.1 volts per cell.

The purpose here was to determine if phase change varied with capacity decrease. By operating at below current, 1.5 amps, and high temperature, a coarse deposit of large crystals are formed on the cadmium plate according to the literature. This causes a reversible battery capacity degradation called memory effect.

Phase range and total capacity followed each other as the cells degraded for at least 22 cycles that we ran. This correspondence would be important for a fuel gauge application. If this held for actual load profiles a gauge would actually get true readings of remaining capacity.
Next slide.

(Slide 20.)

A simple electrical circuit for the cell would consist of a resistance, \( R \), and a small series inductance cells, typically \( 10^{-6} \) henrys or connectors, internal leads, and electrolyte resistance in series with the parallel combination of a large capacitor, \( C \), which typically is several farads, and a small resistance, \( R_b \).

The capacitor is a lump representation of that portion of the double layer between the electrodes and the electrolytes which is, to some extent, affected by ionic absorption and reaction kinetics.

The small resistance represents the effects of activation and concentration polarization. The results here are purely empirical.

The double layer is thought to be the main component varying with state of charge. At frequencies above 12 hertz the combined effect of double air and sound inductance produces a positive inducted phase angle, so at most of the frequencies we were operating we had an inductor.

It is interesting to note that when series ni-cad cells were removed, the phase angle became less positive, which would be logical.

Next we'll take a brief look at the lead-acid system.

Next slide, please.

(Slide 21.)

Here we find the phase angle versus amp-hours out of a six-volt, 67-amp-hour SLI battery at 17 amps. This is a four-hour rate and 3.5 amps there at the tail end, the 20-hour rate. And so we went to one volt per cell in each case, at each rate.

I think it is interesting to see that the 400 hertz,
FIGURE 18

FIGURE 19

FIGURE 20

FIGURE 21
while it is not a straight-line relationship, at least it does seem to follow some reasonable relationship.

Also, we broke this down, the 400 hertz, just to see what was happening, into the reactive and resistive components. And the reactive component, while it is fairly flat until you get out to where that knee begins to occur in the amp-hours there, the resistive component followed conductivity.

Next slide.

(Slide 22.)

Here we have phase angle versus amp-hours on a 150 amp-hour forklift truck battery at the six hour -- It's 150 amp-hours at the six hour rate; that's how they rate it. It's a forklife truck battery.

We charged it at eight amps to five volts; it's 2.5 volts per cell, and discharged it at 25 amps to 3.5 volts.

I think it is interesting here to note the linearity of the charge curve to the onset of gassing. Now generally it is thought that gassing starts at about 2.2 volts so you can't usually observe it until a little after that, and somewhere between 2.2 and 2.3 you are definitely in gassing. And I think you can see the effect that has on the curve.

You can see that the discharge is similar; at least you might say it's a similar direction, but the charge is actually in the opposite direction with a ni-cad, which is interesting.

Also, phase measurements seem to give some more meaningful correlations than just simple amplitude measures across the battery with lead-acids which didn't seem to change very much.

Next slide.

(Slide 23.)
This is a-- Just take a brief look. We had a molten salt cell being tested to near the end of its cycle life, and so we hooked up our test equipment to it to see what would happen.

Here we charged it at five amps to 3.25 volts and discharged it to two volts, and this is the discharge results. Normally the cell does not discharge below 2.4 volts, so you can see that after 2.4, we have a noticeable change. Apparently there we're going through a window in which some constituents are tied up.

We tried normally not to go through that but I wanted to see what would happen here if we did.

In conclusion, the ni-cad system demonstrated that phase change does relate to the capacity available even under conditions where cycle degradation is significant.

Use of phase measurements for state of charge and end of charge control appears possible. The SLI lead-acid battery, 400 hertz, decreased continuously with ampere-hours out while the forklift battery had a fairly linear relationship on charge.

The molten salt system's phase angle batteries decreased and reaction changes can be detected. Phase measurements may be useful as a diagnostic tool for state-of-charge control schemes, ending charges or discharges for various studies in the molten salt and lead-acid systems.

That's it.

WEBSTER: Thank you, Ed.

Are there any questions?

GASTON: Gaston, Grumman.

The nickel-cadmium cells you used for testing, were these the open type or were they the sealed type?

DOWCIALLO: They were the vented sealed type.
GASTON: Would you expect a similar phase-change correlation in sealed cells?

DOWGIALLO: There is reason to believe there would be.

WEBSTER: How do you think the effect of oxygen pressure as you saw in the lead-acid, your gassing taking place--- Do you think you would pick this up?

DOWGIALLO: There is reason to believe you would pick that up, yes. There has other work been done in other areas that would make you think that that's true.

CORBETT: At the frequency that you're working at, when you see a change in phase angle, isn't that really--- or let's say doesn't that really reflect the change in cell resistance balanced against the relatively constant series inductance that you have in the cell?

In other words, doesn't it--- It is amplification of the resistance factor mostly, isn't it?

DOWGIALLO: They are both in there. If you sit down and work it out you end up being--- My phase readings were inducted above 12 Hertz so it is not so much resistance, I would think, as the inductance effect combined with the capacities at that frequency.

CORBETT: What I meant was it is my experience that the inductance, regardless of the state of charge, if you just look at---

DOWGIALLO: It is fixed.

CORBETT: And you still have the 20 db line; okay. And so what is really controlling the phase is what is the resistance more than anything. It is even less than the capacitance.

What I'm trying to say is the capacitance is insignificant.

DOWGIALLO: I thought it was the capacitance, myself.
In fact, in the lead-acid system we did break it down into its components and we found that the resistive component followed the conductivity exactly; at least what I would think from looking at some of the references on that, it seemed to follow those curves while the reactive component, at least at 400 hertz, it seemed to vary somewhat.

It's fairly flat. It comes down like this and it's fairly flat, and then it begins to go down like that. (Demonstrating.) You can probably work out some kind of a system if you had some circuit that would take those two things and know where you are at any point and give you a readout.

CORBETT: I think if you did some analyses that assumed that you had a frequency-dependent resistor, you know, kind of like a third-order term, it would make a lot more sense. I think the literature has reported that there is that kind of an effect. It is not an RCL sort of thing.

ROGERS: Rogers, Hughes Aircraft.

I noticed in the original circuit I believe you used two amplifiers with five microfarad and 15,000 ohm resistors where you get phase shift at about 60 hertz. Is that intentional?

DOWGIALLO: No, that's not intentional. The real purpose there was just simply to decouple the DC component.

ROGERS: In that event, if your capacitor is a typical capacitor of that size, it's not going to be terribly stable and you're going to get some phase shift just from room temperature drifting.

DOWGIALLO: We put this into the oven, the particular capacitors that we used. I forget what they were but we did try several capacitors and we had this in an oven and checked, and it did not have a significant effect.

STOFEL: Stofel, Hughes Aircraft.

We have had some experience at Hughes similar to this.
Also, I had some at Aerospace before going to Hughes. And in both cases we saw a fairly large difference between the cell, depending upon what its previous nickel history has been.

That one Vugraph you had there--

DOWGIALLO: I went to an extreme on the one there, low current, very low current, high temperature.

STOFEL: I guess my question is have you tried varying other things with ni-cads? Do you have a bigger experience on different types of cycles?

DOWGIALLO: No. This was a rather limited time-type thing. We did not go into it-- I hoped somebody would go on with it.

STOFEL: One comment or question that was raised-- The experiences I've had happened to be in the 50 amp-hour, 45 amp-hour type cells, and they were sealed, flight-type ni-cads, and indeed there's a phase shift and an amplitude shift.

There is some question whether it is usable as a full gauge, as you call it, because of the dependency upon past history. It may, however, be a good diagnostic -- perhaps a bit expensive but a good diagnostic tool in terms of knowing something about your battery in addition to the state of your charge.

DOWGIALLO: We looked into some relatively inexpensive circuits and there apparently are some-- We did try one phase detector circuit with two off-amps and you actually had a window controlled by one, say A, with the current input which would control the B input to the other one, turning it on and off, and had an integrator after that one which would give you voltage proportional to phase, and it was very inexpensive. And there are a lot of other ways to do it.

STOFEL: With regard to that, at the Aerospace the work was such that we talked to a circuit designer and he happened to have a phase detector he was using for another
purpose. It was essentially a flight-qualified little unit. It weighed ounces at most, used low power, and if the basic electrochemistry was such that you could use it as a fuel gauge, there would be no difficulty interrogating the battery during charge or discharge as well as quiescent periods.

So it's a good approach if the battery will only be reproduceably, and that evidence we didn't--

DOWGIALLO: That would require quite a bit of testing I would feel.

GROSS: Gross, Boeing Company.

This basic approach does have some very interesting possibilities. It was pursued at Boeing for applications not with batteries but for the purpose of characterizing surfaces of aluminum, and it has been reported in the literature.

It was carried out in extremely great detail to a very complete state I thought. Some of the things that were done were to use an extremely wide range of frequencies and to break out the resistive and capacitive inducted elements.

And as Bob Corbett pointed out, resistance is frequency-dependent and this must be accounted for.

For those who might be interested in pursuing this further I'd be glad to find the reference for them.

WEBSTER: Any further questions?

Ed, thank you very much.

Our next speaker will be Sam Bogner of JPL, and he'll be talking about our short test, charge retention test, authored by Bogner and Uchiyama.
My discussion this morning is on the internal short testing of nickel-cadmium cells. A large portion of this work was to support the Viking project. Jet Propulsion Laboratory is responsible for the Viking orbiter system which is part of the over-all Viking project managed for NASA by the Viking project office at the Langley Research Center.

This particular discussion was presented earlier this year at the IECEC but it was suggested, since there were not too many battery personnel at that meeting, that it be presented again. Also, it missed the publication of that meeting, so I do have some papers with more data than I will present here up on the front table if anybody wants to pick one up.

Next slide, please.

There were about a one to two percent failure

An internal short test, I think as most of you know, has been used by most users of ni-cad cells, and the subject has been discussed at many of the Goddard Space Flight battery workshop meetings. But really, there has not been much data published and put in the literature on some of the results of the tests.

I couldn't find, actually, the origin of the tests. Perhaps somebody in this room knows how it originated. On past programs we had about a one to two percent failure
rate on cells passing the short test, and the cause of the failures were not always determined.

On the Viking orbiter '75 program we ran into about a six percent failure rate on the first lot of 100 cells, but that wasn't so bad. The problem was that they were failing after they were built into batteries, and the construction of this battery is pretty difficult to remove cells from, so the project management became quite concerned.

We are also dealing with a new cell size which is rated at 30 ampere-hours and actually delivers approximately 36 ampere-hours. Initially there were 100 cells built for development cells, and the flight program involved approximately 350 cells.

The battery was assembled and tested and the cells were purchased by TRW as the subcontractor to Electro-optics Systems who was the power subsystems contractor.

Next slide, please.

(Slide 27.)

Okay. As we stated before, we had about six failures out of the first 100 flight cells that we received and four of these failures occurred after the batteries were assembled.

We removed some of these cells from the batteries and also some of the failed cells that we found in the screening test for autopsy, and we'll show a few pictures of the cell autopsies a little later.

We contacted several other users around the country to see if they were having similar problems. We held a technical review at the manufacturer, and out of those discussions and meetings we implemented a quick test program.

Okay, the next slide, please.

(Slide 28.)
INTRODUCTION

INTERNAL SHORT TEST USED BY MOST USERS
SUBJECT DISCUSSED AT GSFC BATTERY WORKSHOP
LITTLE TEST DATA PUBLISHED
ORIGIN OF TEST UNKNOWN
FAILURE RATE 1-2 PERCENT
CAUSE OF FAILURE NOT ALWAYS DETERMINED
ON VO75 PROGRAM 6 PERCENT
NEW CELL SIZE 30 Ah 100 DEVELOPMENT CELLS
TRW PROCURED FLIGHT CELLS 350

FIGURE 26

AGENDA

- INTRODUCTION
- PROBLEM INVESTIGATION
- SUMMARY OF CELL AUTOPSIES
- TEST PROGRAM & RESULTS
- CONCLUSIONS

FIGURE 25

PROBLEM INVESTIGATION

6 FAILURES LOT OF ABOUT 100 CELLS
4 OF FAILURES AFTER BATTERY ASSEMBLY
PERFORMED CELL AUTOPSY
CONTACTED OTHER USERS
TECHNICAL REVIEW AT MANUFACTURER
IMPLEMENTED TEST PROGRAM

FIGURE 27
This is a quick summary of the results of the cell autopsy. It was thought that possibly they might have high nitrate content which would have some effect on the charge retention or the short test, but they now showed that the nitrate content was very low; the carbonate content was relatively low.

We did find that all plate thicknesses were at the top side of the limits for cell assembly. The negative plate loading in some cell lots was slightly over specification. Generally, cell construction and the workmanship was good.

One thing we found was cadmium deposits as stripes, and we have a picture of that, in the separators, and these were new cells and normally you don't see cadmium in the separator on new cells that have had approximately only 30 cycles.

In the area of these cadmium deposits on the separator opposite on the negative plate we found discolored surface hydrates and we also found interstitial material in the separator which was a cadmium compound.

In one cell we found a little tear in the separator, and in one of the developmental cells we found a plate-to-plate short.

Next slide, please.

(Slide 29.)

This is the cadmium deposit that we saw in some of the cells that failed the short test. We also saw similar deposits in cells that passed the test but they weren't quite as heavy as this one.

You're looking at the bottom right here.

We also saw some stripes-- I don't have a picture of it -- near the top.

Next slide, please.
This is an SEM photo of the material in the separator. We didn't analyze exactly what the material is but we do know it is a cadmium compound, probably cadmium hydroxide. If it had been cadmium when we opened the cell it was done in the atmosphere so it would have oxidized right away.

The next slide --

-- shows a photograph of the plate-to-plate shorts. There you can see a positive plate sticking up out of the separator bag and touching the negative terminal coming up, and there is also one here, and possibly one over here.

Now that particular cell passed the internal short test and it was discovered about six months later.

Next slide, please.

The test methods that we used on this program were basically those that TRW had used and also JPL had used on a previous Mariner-Mars '71 program.

Basically the cell is discharged at the C/2 rate to approximately one volt, and then it is shorted with a half ohm resistor for 16 hours, or to a .2 volts and then it is dead shorted for 16 hours. Then it is given a charge at 15 amps for 60 seconds and it's placed on an open circuit stand and the voltage at the end of the 24-hour stand period is supposed to be about 1.19 volts.

This particular test is performed after 31 cycles at 75 percent delta discharge, four cycles a day.

The other primary test which is being used in the industry I guess is the Goddard Space Flight Center test and the one used at NAD Crane on all their testing where the cell is discharged and is shorted with one ohm for 16
hours and then placed on an open circuit stand, and then the voltage at the end of 24 hours must be above 1.15 volts.

Then we started doing a new test, a charge retention test. In the past a lot of people have referred to the short test as the charge retention test, and I would like to make sure we distinguish between the two because the short test I don't believe has much relationship to the actual charge retention of the cell.

So in the charge retention test we charge the cell up completely from zero to ten range at 20 hours, and stand open circuit for seven days, and this was usually at room temperature, and then we record the open circuit voltage at 12-hour intervals during the seven-day stand, and then the cell is discharged at the C/2 rate, and that capacity cycle is then compared to a previous capacity cycle done on the cell. And if you want to make a double-check you can also run a second capacity cycle after you do the charge stand period.

next slide.

(Clipse 33.)

This is the outline of the test program that we went through. We had 48 cells ready to go in the batteries when we started running into our problems so we performed the short tests on those again, and a seven-day charge retention test.

We had two batteries assembled. We performed the short test and also the seven-day charge retention test on those. Then we had seven cells that we did more extensive testing on. We tried both the VO-75 short test, the Goddard short test, the charge-acceptance seven-day stand, and an additional 31 cycles, and then we repeated those tests again after going through an additional 31-cycle period.

We had 14 cells at JPL that we ran similar tests on. At least seven of those cells were of the flight variety
and the other cells were part of the 100 developmental cells that had been built a year prior to the flight cells.

We also had two '71 batteries that had been on test for about four years and these were still on a trickle charge, and we decided to see how they would perform on these tests.

And then Crane had two cells, and Dr. Floyd Ford, and these cells were designated out of a pack, 50-B, at Crane, and the test data on these cells is in one of their reports.

These two cells had exceeded 29,000 cycles at 25 percent depth of discharge. They were six ampere-hour cells and they had been on a 90-minute cycle. So you can see the tests that were performed on those cells at Crane.

Okay, some of the test results then. Next slide, please.

(Slide 34.)

This summarizes the seven-day charge retention tests that were run.

On the 48 cells, all of those were between 83 and 86 percent charge retention. The two batteries of course was based on the first cells to go to one volt, and they were 85 and 87 percent.

The seven cells, after an additional 31 cycles, ranged from 68 to 83 percent, and two cells out of that group of seven failed the short test.

The 14 cells that we tested at JPL-- We were trying to determine a method possibly to accelerate the shorting or the problem that was causing the apparent shorts, so they were put on an accelerated charge. They were charged at three amps at 120 degrees for 28 days, and then they performed a short test and charge retention test.

They show a high charge retention result but that was because they actually gained in capacity during the 28-day
charge.

And then the two cells at Crane that had gone through 29,000 cycles only had 55 percent charge retention in the seven-day stand, but both of those cells passed the short test.

We performed both types of short tests, the Goddard method and the TRW method, on a lot of these tests and in all cases when a cell failed one test it failed the other. If they passed, they passed both tests.

Next slide, please.

(Slide 35.)

This just shows a typical plot of the open circuit voltage during the seven-day stand test. Some people have also suggested that you can use -- and I guess that's in the most recent Goddard spec now, that the cells out of a given test group should be plus or minus five millivolts of the average.

Generally you'll see slightly less than a ten millivolt spread out of a large group of cells on the seven-day stand test. Actually, the temperature I think plays an important part, so although this test temperature was over a three degrees C. range, I think you should attempt -- If you're really going to spec them you should hold that temperature to plus or minus one degree.

Next slide, please.

(Slide 36.)

This is a plot of the cell potential at the end of the seven-day stand test versus the capacity retention. These two cells were the ones that failed the short test. All the other cells passed that test.

It looks like we've got a real good correlation here. However, this test was repeated. Another 31 cycles was put on it, except one cell was removed for autopsy, and we didn't get any correlation on the next test.
TEST METHODS
VOLTAGE DECAY (TRW/JPL)
- DISCHARGE CELL
- SHORT WITH 0.5 OHM 16 h
- DEAD SHORT FOR 16 h
- CHARGE AT 15 A FOR 60 sec
- OPEN CIRCUIT STAND
- AT 24 h VOLTAGE MUST BE ≥ 1.19
  *(PERFORMED AFTER 31 CYCLES AT 75% DOD)*
VOLTAGE RISE (GSFC/NAD CRANE)
- DISCHARGE CELL
- SHORT WITH 1 OHM 2 h
- OPEN CIRCUIT STAND
- AT 24 h VOLTAGE MUST BE ≥ 1.15
- CHARGE RETENTION
- CHARGE CELL C/10 20 h
- STAND OPEN CIRCUIT 7 DAYS
- RECORD OCV 12 h INTERVALS
- DISCHARGE CELL C/2

FIGURE 32

TEST RESULTS
7 DAY CHARGE RETENTION

<table>
<thead>
<tr>
<th>48 CELLS</th>
<th>TEST</th>
<th>TEST</th>
<th>TEST</th>
<th>TEST</th>
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<tr>
<td>TRW</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>2 BATTERIES</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>GSFC/31</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

14 CELLS | X | X | X | X | X |

2 BATTERIES | X | X |

NAD CRANE | X | X | X | X |

FIGURE 33

OUTLINE OF TEST PROGRAM

<table>
<thead>
<tr>
<th>VOLT</th>
<th>SHORT</th>
<th>TEST</th>
<th>TEST</th>
<th>TEST</th>
<th>TEST</th>
<th>TEST</th>
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<tr>
<td>2</td>
<td>BATTERIES</td>
<td></td>
<td></td>
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<td></td>
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</tr>
<tr>
<td>GSFC/31</td>
<td></td>
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<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
</tbody>
</table>

14 CELLS | | | | | | | |

2 BATTERY | | | | | | | |

NAD CRANE | | | | | | | |

FIGURE 33

CELL VOLTAGE, V

7 DAY STAND OCV vs TIME
(TEMP. 24±3°C)
24 LOT 8 CELLS

FIGURE 35
At the cell autopsy it was difficult to look in and say "There's the short."

Next slide, please.

(Slide 37.)

Now we thought that we were getting identical cells on the flight build as the developmental cells, and this is a plot of the discharge of some of the typical cells after the 28-day three-amps charge at 120 degrees.

You can see on the developmental cells you've got a typical discharge but on the flight cells we've got a dip out here at about the last 25 percent on the discharge curve, so the cells weren't identical and I don't know the specific reason for that. People have made suggestions.

Next slide, please.

(Slide 38.)

This is just a typical plot of two of the cells that failed the charge retention or the short test. One of them shows a plot of the Goddard short test and the other is a TRW short test where a little bit of charge is put back in.

The interesting thing about this is on the Goddard test, the cell voltage actually recovered and then dropped back down, so if it had a short in it makes you wonder why it would recover from a short down with the resistor.

Next slide, please.

(Slide 39.)

This slide summarizes the charge efficiency test. Some people had suggested that at the low end or zero state of charge, the charge efficiency was a possible reason for the cell not passing the short test, and they also indicated that the charge efficiency was probably low.
7 CELL TEST
CAPACITY RETENTION vs CELL POTENTIAL AT 7 DAYS

![Graph showing capacity retention vs cell potential.](image)

FIGURE 36

CELL VOLTAGE BEHAVIOR DURING SHORT TEST

SHOWS GOOD CELL 6 FAILED CELL FOR BOTH TESTS

![Graph showing cell voltage behavior.](image)

FIGURE 38

TYPICAL DISCHARGE CURVES OF CELLS AFTER 28 DAYS CHARGE

CHARGE 3A & 43°F C
DISCHARGE 15A 0°F C

![Graph showing typical discharge curves.](image)

FIGURE 37

TEST RESULTS CONTINUED

CHARGE EFFICIENCY
C/2 FOR 1 & 2 MIN 0.90 TO 0.74V
C/10 FOR 8 & 12 MIN 0.20 TO 0.14V

RESULTS VARYED 15 TO 40%
RATE HAD NO EFFECT
TIME HAD NO EFFECT
NO CORRELATION WITH FAILED CELLS

![Text box with test results.](image)

FIGURE 39
So the tests that we ran were a C/2 for one and two minutes, and a C/10 for six and 12 minutes.

Now in the past programs the C/10 rate for six minutes had been used, and on the Viking program it was changed to C/2 for one minute. The reason for that was stated that the charge efficiency would be better at a higher rate.

Well, the results varied from 15 to 42 percent. The rate didn't seem to make much difference and whether you went one minute, two minutes, six minutes, or 12 minutes didn't seem to make much difference. So the charge time had no effect.

Actually there was no correlation between the charge acceptance and the failed cells.

Actually the two cells that had been on the 29,000 cycle test at Crane gave about ten percent charge efficiency and they passed the test.

Next slide, please.

(Slide 40.)

We decided to take a look at what size short are we really detecting on these tests, so on the seven-day charge stand test or the charge retention test, how sensitive is this test?

Well, if we assume in our past testing that the lowest cell lost 12 percent beyond the normal loss of, say, 80 percent, you can calculate that this comes out to about 50 ohms short.

On the short test, if we assume that 30 percent of our charge is going into charging the electrode and assume that the cell voltage during the 24-hour stand is at least one volt, and assume that there is no self-discharge, the result is approximately a 300-ohm short. So this gives you some idea of the magnitude of the short detectible by the two different tests.
I don't know how you would really calculate the size of the short that you would get with the Goddard test since they put no charge back into the cell.

Okay, next slide.

(Slide 41.)

I have mixed emotions over the short test and I don't think that the testing we did here really came up with any solid conclusions although we found some interesting data.

Anyhow, the short test I do not feel is absolute but it is still one way of doing a screening test.

The charge retention test, the resolution is not as great as on the short test. It requires more time, but it is a good backup test to give you some indication maybe of the size of the short if you fail the short test.

I think we need more test data, particularly on failed cells. Usually the problem in the past has been that when you get a failed cell, the project office wants to know, you know, what's wrong with it. Why did it fail? And you tear them apart and you lose the cell.

So we've tried to save some of the cells that have failed on this test for additional testing.

And then, you know, what size short is really detectible? What does a 300-ohm short look like when you open up a cell? Does the size of the short change with life? If we could guarantee that the size of the short wouldn't change with life, I'm sure on many of the programs that you could fly cells with .1 ohm resistance.

Thank you.

WEBSTER: Thank you, Sam.

KRAUSE: Krause, Hughes.

Sam, did you find any relationship between either the ability to pass the short test or the ability to retain
SIZE OF SHORT DETECTABLE

7 DAY CHARGED STAND
LOWEST CELL LOST 12% (Ah)
50 OHM SHORT

SHORT TEST
ASSUME: 30% CHARGE EFF
1.0 V AVE OVER 24 h
NO SELF DISCHARGE
RESULT: 300 OHM SHORT

FIGURE 40

CONCLUSIONS

SHORT TEST
NOT ABSOLUTE
STILL WORTHY OF DOING

CHARGE RETENTION TEST
RESOLUTION NOT AS GREAT AS S.T.
REQUIRES MORE TIME
GOOD BACK UP TEST

NEED MORE TEST DATA PARTICULARLY ON FAILED CELLS
WHAT SIZE SHORT SAFE TO FLY ?
WHAT SIZE SHORT IS REALLY DETECTABLE ?
WHAT DOES A 300Ω SHORT LOOK LIKE ?
DOES SIZE OF SHORT CHANGE WITH LIFE ?

FIGURE 41
charge with time to the interelectrode spacing in the packs and the pack thickness and the swelling of depression?

BOGNER: Yes. In the paper there's a little bit of data where we compressed the cells and ran the short test and also ran the short test with the cells free-standing and there was a difference.

We repeated the test again and that clouded the results.

DE BAYLO: Paul DeBaylo of Fairchild Industries.

I have two questions. The first question relates to one of your early slides with the shorted plates on which you made a comment that about six months prior to detection of the short you had run an internal short test. I'm assuming you did not go through any shake, rattle, roll of the cell or any real usage between the times you ran the internal short test and the time that it was detected.

I was interested in whether you ran any kind of peak load test like a 3-C discharge at the time you ran the internal short test to try to pick up some defect like that.

BOGNER: No, we didn't run a high rate discharge. The cells went through a 31-cycle approximately 75 percent depth of discharge before we ran the test.

They did not go through a shake, rattle and roll test other than the fact that they were transported from TRW to JPL, and I suppose they got some vibration there. And they were stored at JPL in a shorted condition, so perhaps during the storage period this plate swelled a little bit and came in contact.

DE BAYLO: The second question I have, and maybe a comment relating to some of my experience.

I'm kind of interested in whether you have any basic
experience for running, say, a Goddard-type internal short test based on previous cell history, in other words what the cell has gone through prior to running that test.

The reason I'm making this comment, I notice difference in recovery voltage if you run the test after, say, 30 cycle -- 50 percent depth of discharge cycling versus a virgin cell.

Also, we had performed internal short tests on the ATS-6 batteries very successfully several times during our acceptance testing. We had then installed the batteries on the spacecraft and subjected the spacecraft to spacecraft level vibration.

We decided to reperform this test.

Basically what we did is the batteries were on the spacecraft supporting various RFI type tests, discharging at various rates and so forth, with really no plan of battery profile, if you want to call it that. And we tried to repeat this internal short test. We discharged-- Rather than a C/2 rate, we discharged like an amp, an amp and a half, varying rates because we had various loads on, to an average cell voltage of probably 1.1 volt.

We then applied the one ohm across each cell for 16 hours. We opened the circuit and then we failed miserably this internal short test, and we started scratching our heads and we said, "Hey, we've got problems."

Yet we had passed the same identical test four or five times during acceptance testing.

We then ran a modified version of one of your internal short tests where, after shorting the cells out for at least an hour, we had placed a charge back into the cell for about five minutes, and we let it sit open circuit for 24 hours, and then we examined the cell voltages at the end of that period and they were all above what we considered an acceptable limit, proving the cells themselves were not really bad.

I was just kind of curious what your experience along
Bogner: It's been similar. It does make some difference on how the battery in a previous test has -- on previous cells, but I can't give you explicit numbers.

Webster: There is one comment on the ATS program, that all the cells failed so immediately we suspected the test, not the cells. And then we applied what I call -- which I didn't see up there -- the five-minute charge which is similar to the technique which used -- where they put five minutes of charge on there, and they all passed.

But yours truly had a heart attack because that was the last test before the spacecraft left for the Cape, and Von Braun and the Indian Ambassador were having pheasant under glass, and I knew that all the batteries had just failed.

Ford: I would like to point out something. I have a question for you, Sam, but let me make my point first.

In reference to the ATS battery, I would point out those cells had fairly low-level precharge and the current indication from other programs that we've seen is that the state of charge on that negative electrode certainly is a factor in the open circuit recovery test.

I think that's what we were seeing as a result of low precharge on the ATS cells, that in the low-rate discharge which really depleted the negative as well as the positive, influenced that open-circuit test.

Bogner: You must be in bad shape in the precharge. You don't have that much cadmium in your precharge to affect the test.

Ford: That's debatable, too.

I would like to ask you a question, though, particularly in line with what you were referring to as your developmental cells versus your flight. And I think Stan
alluded to this point but I would like to pursue it a little more in detail.

Have you done a statistical analysis, a worst-case analysis of interelectrode spacing taking into consideration positive plate expansion and the dynamic behavior of the cells? Have you really looked at that on the developmental cell versus the flight cell and if so, what is the interelectrode spacing in the worst case?

BOGNER: Five to six mils.

FORD: After you have run tests on the cells?

BOGNER: Yes. Well, the 31 cycles or maybe 60 cycles on a couple of cells. They were autopsied and the plate thicknesses were measured. And after the swelling we got at that time, it was around five to six mils. spacing.

J. H. SCHULMAN: Schulman, PSI.

Have you looked at the time constant for open circuit recovery, and would you care to comment on what the mechanisms might be that would affect the time constant for recovery of the open circuit stand?

BOGNER: Well, one would think that-- We tried a little bit of that using three plate cells and trying to get a reference electrode, but I'm not too confident of the results we got.

We attempted to look at it but we didn't go into it in enough detail. I kind of think that part of the problem may be due to the positive electrode and as Floyd pointed out, if you get problems with precharge in the cadmium electrode, it could be causing problems.

I don't have an answer.

CORBETT: Sam, I think it is kind of instructive that in your data you have examples of cells that passed either test A or test B or both; in other words, you've got every kind of possibility there. You actually had
cells that passed the short test and didn't pass the charge retention or, let's say, didn't give a good charge retention, and you have cells that gave good charge retention but also failed one or both of the short tests.

For example, I guess you had two cells from Crane that did pass the short test but had poor charge retention.

BOGNER: That was kind of mysterious. Those two cells had been on 29,000 cycles. They were autopsied. I don't have all the data here. But when you look at those separators -- and we've seen pictures before -- the separators were black with cadmium.

CORBETT: Well, to me what is kind of interesting about that is --

BOGNER: They passed the short test but the charge retention was only 55 percent.

CORBETT: But what's the test criterion? What you're getting is a final voltage. It is not a voltage drop or it is not dependent on how the voltage rises. You know, the final test criterion is what is the voltage, 1.15, 1.19, or whatever it happens to be. And there are really two parameters involved.

One is resistance of the cell or the resistance of the supposed short which is in parallel with the cell, and the absolute voltage, and it seems possible to have a cell with some sort of a partial short but with a fairly high initial voltage so that it decays to something reasonable.

And it is also possible to have something like a cell without a very high initial voltage and perhaps less of a partial short, less of a shunt load, and have, you know, comparable voltages on those two cells.

So I would like to put out a couple of propositions.

One is if a cell is completely discharged it will probably fail the short test. I think we've all had this experience. If you keep a cell shorted for two weeks, it
will never come up, or for three weeks. You know there is some time period at which you can insure that a cell will not respond.

BOGNER: Yes. You should give it a couple of cycles before you test it.

CORBETT: And usually when we're judging plate capacity and battery performance, we usually think a battery is good if we get all of the capacity out at some fairly high voltage, in other words, if there is a very small amount of distributed resistance in the plate itself.

Okay, so if you get all the capacity out real fast with a very small voltage drop, then it seems to me with that in mind that the perfect battery would probably fail this test because under load it would lose all of its capacity very quickly in something like the Goddard test and fail to come up, and it would probably also fail the Hughes or JPL type of test because when you dead short it you take all the capacity out and what you put back in is below what is a good reaction voltage or something.

So I think the problem is in the test itself, which is probably a kind of a dumb thing to say as a conclusion to all of this, but I think a real problem is we're looking at an absolute voltage; that is, trying to deduce from an absolute voltage what is the shunt load.

It seems to me you ought to be looking at the actual shape of the voltage rise or something that relates to the time constant of the cell itself. I think the charge retention test is probably a lot more instructive if you are worried about cell reliability.

FORD: With regard to that interelectrode spacing, I have noticed over a few hundred cells on some programs in-house over the last couple of years that the ones that seem to fail the short test, the 24-hour short test initially are always those that apparently are slightly convex. More than the others they're bulged.

We get cells, an occasional lot of cells, that have fairly thick plates and the cells are bulged somewhat and
those are restrained and the short test is run, and they always fail the test.

If their cycles are reconditioned they are fine after that, but if you take a cell that is not bulging and one that is and you do the same thing to it, the one that is bulging seems to have a lower voltage, always initially especially. There seems to be some relationship between the compression in the pack and the results of the short tests.

BOGNER: All the cells bulged, like 20 to 30 mils. beyond the original spec size.

KIPP: From a manufacturer's standpoint we do have some experience that I think is relevant and that is that with our experience, when you build cells, when you start out with an interelectrode spacing of five to six mils., all things being equal you will have a higher incidence of charge retention failures, regardless of what tests you run on those cells versus cells that you build when you start with an interelectrode spacing of around eight mils.

There is enough data to show that that's a fact.

POWELL: Powell, of Powell Corporation.

What was the separator?

BOGNER: A standard pellon separator.

KRAUSE: One comment about the origin of the short test. I cannot attest to the veracity of the comment but I have a recollection that about two years ago at this workshop I think Lou Belove mentioned that he recalls it being a test that was originated at Sonotone to look for copper in the cells as a result of contamination from seals way back when.

As I say, I don't know if it is true but that is what he commented.
I. M. SCHULMAN: Schulman, TRW.

I go back almost as far as he does. I think the cell was originated by one Walter Scott at the Applied Physics Lab in around 1958.

FORD: One other question. Can you give us any significance to the fact that some of the numbers you put up there are different from the flight cells as far as the upper limits on the loading and so forth versus the increase in percentage of the cell failing the short test?

BOGNER: I can't tell you right off-hand.

GROSS: Another useful source of cell shorting information lies in the data on the cells that are cycling prior to running any specific cell shorting test. In the testing we do we use primarily digital data systems which give you information periodically. This of course misses most of the data.

However, if you put a brushline recorder on the data and you follow the voltage time history exactly, on cells which are likely to have shorts you will find transients in the data which show that shorts are developing in the cell and that they are burning out, and this transient looks like a voltage dip and then later on, the voltage comes up as the short burns out and the new voltage will be slightly less than the original voltage prior to transient.

This shows up especially well on cells which are deliberately being tested to accelerate the shorting condition.

RAMPEL: Guy Rampel, General Electric.

The impression was left that in some six-hour cells there was very little precharge and that in some way may have affected the Goddard short test.

I would like to point out, though, that there probably was residual precharge available as such but not electrochemically available, and it should generate a
potential for it.

FORD: Correction, the low precharge was in 12 ampere-hour cells.

WEBSTER: Correction, 15.

CORBETT: Just a brief comment about the sensitivity of the Goddard test, Sam.

Once I tried with some 20 amp-hour SAFT cells to measure what kind of current was available after they were shorted for something -- I think like 24 or 48 hours, and with a 20 amp-hour cell I found that the cell behaved almost as a constant current source and would furnish about four milliamps.

So if you thought that a tenth of a volt would be a significant drop or something that you pick up, that means that test would be sensitive to something like two or three K, a two or three thousand ohm resistor in shunt, but that would depend upon the cell.

The actual sensitivity of the test in that case was really cell-dependent and dependent on how much current was coming out of the cell when it has been so-called shorted or totally discharged, which is almost never.

BOGNER: Ron Banes at JPL did some testing by putting various resistors on-- These were 20 ampere-hour cells that he tested, and I think the result he got was it showed that in a 24-hour period you would be down below 1 to 1.5 volts at around a 300 ohm resistor. So it was interesting that his results came out close to what were calculated.

BAER: Baer, Goddard.

I would like to verify -- I also ran some tests on the Goddard short test and I found they were detectible at 300, 350 ohm short.

I'd also like to comment on that one curve you showed that began to recover and then fell off. I've
also observed that when you have a resistor across the cell of the order of 300 ohms. I have also observed that. But I haven't seen it fall off quite as rapidly; it was more of a gradual decay, and gradually, maybe at the end of a 24-hour period it will be down under 1 volt. So it would recover, and then decay.

BOGNER: This was a typical plot of two cells and if you plot a couple of other cells that failed you would probably get an entirely different plot.

LACKNER: There was some comment made on bulged cells and I think I would like to follow that up.

How was the bulge detected on an open circuit? On the stand? Because we are also told to restrain the cell walls.

BOGNER: This is detected as they're received at the receiving inspection.

LACKNER: They came in bulged?

BOGNER: Yes. And they are not restrained during shipment.

LACKNER: So this isn't a case of the plates growing in size? It could be a gas pressure?

BOGNER: It is not believed to be gas pressure and it was believed to be due to the packaging. Like I say, when we tried to look at all the data that we could, manufacturing data, all the plates were on the high side of the specification in loading and also the thickness.

WEBSTER: Can I ask you to hold questions now, and we'll move on to Dr. Scott.

Thank you, Sam, very much.
Dr. Scott's presentation is going to be improvements in the short test. He's got the answers.

IV.3 IMPROVEMENTS IN THE SHORT TEST: SCOTT/TRW

Dr. Scott.

SCOTT: All the discussion that has been going on here—I've been impatiently holding my tongue because of the data that I am going to show you now.

After the results that Sam reported, we got to scratching our heads and decided that one of the things that we probably needed more than anything else to try to unravel some of these questions was a more scientific method of detecting shorts, at least more definitive and more data than a single point after 24 hours or after one week of open circuit stand.

So under the sponsorship of JPL, TRW undertook a small study of possible approaches to improving methods of test for shorts in nickel-cadmium cells. This study is still underway. A final report will be issued early next year, and so what I have to show you now is interim data as a preview of things to come, and I think the data is interesting.

(Slide 42.)

The scope of the work involves all three of the test methods previously discussed: the so-called 24-hour voltage decay test; the 24-hour voltage recovery; and the seven-day charged stand.

We are including in the study the possible effects of cell history prior to testing, the effect of conditioning immediately prior to conducting the test, parameters used during testing, calibration of sensitivity, and consideration of acceptance criteria.

I'm going to give you just a very brief presentation here today and not try to be exhaustive, so what I would like to do is first present our tentative conclusions and
then I'll show you the data, a sampling of the data that we have so far.

(Slide 43.)

The study is not confined to experimental work we are doing presently. It involves a survey of the literature and summary of what is the current state of the art in terms of test methods of experience. And this indicates that, as no surprise to us nor anyone else, that history of the cell can affect the results of the test and partly because of that, some form of conditioning is required for reliable and reproduceable results of short testing.

We looked at this question of what charge rate to use during the 24-hour voltage decay test and have concluded that of the various reasonable rates to use, that the old stand-by C/10 rate is probably the best one of many to choose from.

Also, we looked at a C/10 charge rate of charge inputs, charge times varying from three minutes up to 12 minutes, and we have concluded, believe it or not, that the old stand-by, five or six minute charge input, appears to be as good as any other and probably optimum in terms of ease of handling and in terms of sensitivity to resistive loads on the cell.

We have looked at the voltage, open circuit voltage over time periods extending over a period of three days; that is, beyond the normal 24-hour period, in order to see whether anything interesting is happening beyond the normal 24-hour time limit.

And generally speaking, as some of the data here will indicate, it looks like if the time is available that a 48-hour stand time is probably more generally useful than 24 hours, and it is more useful at 72 hours in terms of optimizing the effectiveness of the test.

Also, from the data it is pretty obvious that a single point determination and single point pass/fail criterion simply is too crude and is inadequate for the purpose that the test is being used for, which goes far
beyond the original purpose of the test, I would say, of trying to detect whether there is copper in the cell or whether a piece of separator is being pierced by a wire or part of the plates.

I think the intention is to try to push this test as any other test, diagnostic test, to its reasonable limits in order to have a much more useful diagnostic tool, and I think that we are on the road to being able to do that with some of the results that we are getting here.

Now the approach that we're using involves taking more frequent readings than a single reading at 24 hours, namely, a voltage scan at one-hour intervals during the entire test period. The voltages are scanned automatically in the particular case of the present study with a digital data system and the charge removed from the cell during those periods is, as far as I can tell, absolutely negligible as far as any impact on the test results is concerned.

As a matter of fact, as we may get into a little bit later, in the course of testing for the sensitivity of the method by applying ex-channel resistors, we have found that for any cell up to a 30 ampere-hour size that, over a period of 72 hours, any resistance higher than 10,000 ohms has a completely negligible effect on the results, so that this allows for quite a variety of test instruments and sampling times to be used to increase the amount of data that one obtains during this test interval.

So the over-all method that we used was to obtain frequent data and then to plot the data on a scale involving the logarithm of the stand time, and then to look at the results in that format.

The reason that we decided to do this was based on some results that were published a number of years back by Conway and co-workers in which they showed that the potential of the positive electrode decayed on a straight-line function of the logarithm of time over a period of many days if the system was pure and under control.

And we reasoned that since it is pretty well known
that a cadmium electrode in good condition and in a proper state of charge is a fairly constant potential under these same conditions, that therefore, if the cell truly had no resistive shorting paths, that therefore the cell potential should also decay as a function of the logarithm of time.

So we just took that approach and plotted the data and took a look at what it looked like.

I think you'll see that in general the data looks pretty good on such a plot.

(Slide 44.)

Here is the results of three different tests. As you can see, the data is plotted. As you can see, the data almost up to 72 hours is shown there. This is test data using the decay, the voltage decay test in which all cells were initially charged at a C/10 rate for six minuter and then allowed to stand.

The cells involved were General Electric cells, 24-hour rated capacity. The cells were newly received and had not been put through any cycling after receipt from the supplier. They had gone through, of course, the normal acceptance testing at the supplier. They had been shorted approximately three weeks, I believe, from the time that they were shipped from the manufacturer.

Three curves are shown here. Curve A is the plot of the data when the test was applied immediately after removing the shorts without any conditioning, cycling, or whatever following the shorting period. And you can see the slope.

Curve B was a test repeated after a single cycle consisting of charging the cell for 24 hours at a C/10 rate, discharging at C/2 to one volt, applying a quarter ohm resistor to less than .1 of a volt, and then shorting the cells for 16 hours. You can see that the curve is rotated at about that point. (Indicating.)

Interestingly enough, the initial voltage has come down; the final voltage has gone up.
Curve C is the same test repeated on the same cells after a second 24-hour cycle involving C/10 charge, discharge, a quarter ohm resistor, and so forth. Now you can see the curve is rotating about that point and the initial voltage is going back up again, but the final voltage at the end of about 72 hours hasn't changed very much.

This data indicates the average of approximately 48 cells, all of which were part of the same manufacturing lot and in this plot and in subsequent ones that I'll show you, a single point indicates that the spread of voltages was not more than ten millivolts at any point.

When the spread was greater than that, I have indicated that by a vertical bar which extends to include the maximum and the minimum cell voltages.

(Slide 45.)

One of the questions that came up during the Viking cell program was whether the 31 cycles that we applied to these cells had any effect on the results of this type of test. So the cells, the data for which you just saw, were then put through the normal acceptance test and a 31-cycle burn-in, and then retested with the same approach.

Not all the same data is shown here as before.

The curve with the open circles is the one for the cells with no resistors applied. This graph, by the way, shows some of the first calibration data that we obtained on these cells, where we put a 1,000 ohm resistor across the cells and the 330 ohm resistor across.

Now if you compare this with the previous slide you will notice that this curve has come down about 15 to 20 millivolts from the previous one prior to this amount of testing. Also, we don't have as straight a line any more; we have what looks like two lines intersecting at approximately six hours. But then also we have something else. We have a droop out here beyond 48 hours. I will talk about that more a little bit later.

Then we started calibrating at this point and you
SHORT TEST METHOD STUDY

SCOPE

THREE ASPECTS INCLUDED

- 24-HOUR VOLTAGE DECAY TEST
- 24-HOUR VOLTAGE RECOVERY TEST
- 7-DAY CHARGED STATE

ASPECTS INCLUDED

- CELL HISTORY PRIOR TO TESTING
- CONDITIONING PRIOR TO TESTING
- METHOD PARAMETERS
- CALIBRATION OF SENSITIVITY
- ACCEPTANCE CRITERIA

FIGURE 42

SHORT TEST METHOD STUDY

TENTATIVE CONCLUSIONS FOR THE 24-HOUR DECAY TEST

- HISTORY CAN AFFECT RESULTS
- CONDITIONING IS REQUIRED
- C/10 CHARGE RATE OPTIMUM
- 5- TO 8-MINUTE CHARGE TIME OPTIMUM
- 48-HOUR STAND TIME PREFERABLE
- SINGLE-POINT VOLTAGE CRITERIA IS INADEQUATE

FIGURE 43

FIGURE 44

FIGURE 45
see that the curve here for the 1,000 ohm external resistor across the cells might easily be considered in the same population at the end of 24 hours as cells with no resistor. So that that begins to indicate, I think, what the potential sensitivity of this method is as a function of time. That is, it looks like at the end of 24 hours one would tend to accept cells that had the effective resistance of 1,000 ohms, but if you carried the test out here to 48 hours or even beyond, then the difference between these cells and these cells I think is clearly evident.

So this indicates the possible value of extending the time period beyond 24 hours, and even beyond 48.

At 330 ohms it is obvious within 24 hours that there is an obvious and real difference between these two curves. However, it is interesting to note that-- You'll notice the bottom of my chart is 1.18 volts. If one were to choose, as certain procedures do, a criterion of, say, 1.16 volts, then one would pass these cells at 24 hours with the equivalent of 330 ohms.

So you see, without having this kind of data to look at, selecting a single point voltage at the end of any particular time period has a very limited application as far as knowing exactly what kind of resistance you're looking at.

(Slide 46.)

One of the things we were interested in in the study was what the effect of long-term storage may be on the results of such a test, so we had a group of 12 ampere-hour cells, General Electric cells, that had been on shorted storage for three years. We happened to have them in a cabinet.

This curve shows the effect of our so-called standard test conditions for the voltage decay test which is the upper curve, C/10 for six minutes. And then in this particular case we tested out the effects of dropping the charge input by reducing the charge time to three minutes in order to see whether we might be able to possibly increase the sensitivity of the test by doing that, and we
got this lower curve.

Now here the upper curve seems to take a break here beyond about 48 hours. The lower curve seems to be rolling off beyond about 24 hours. Both of these were with no external resistors added and I believe that one might, with additional data, interpret this droop here at the end in terms of a certain equivalent resistance.

I think, generally speaking, this droop here may indicate a resistance of the order of a couple of thousand ohms across these cells internally.

I have not tried to do a complete interpretation of this data at this point in time. I am just showing you some typical results for whatever they are worth right now.

(Slide 47.)

I don't want to be partial to any particular manufacturer of cells but it just so happened that the scope of the program was such that we were limited to testing on just cells that we happened to have in-house during the period of the contract, and these were limited at the time to those available of Gulton and General Electric cells. We did not have any new Gulton cells to test at the time while this testing was going on, but here is some data for some Gulton 12 ampere hour cells after one and a half years of shorted storage.

And as indicated here, curve A is after C/10 charge for six minutes.

Curve B explored the effect of increasing the charge time to 12 minutes. And you can see that the effect was to raise the voltage level and in this case, instead of the curve turning down out here, there seems to be a flattened portion.

In general, it looks like one way of telling the difference between cells that appear to be in good condition with no signs of shorts, either that the curve on a log time basis is either straight or it's convex, it's concave upward and whenever you have a curve that is
concave downward, you might begin to suspect that something else is going on within the cell and is acting like a short.

(Slide 48.)

Here is some resistive calibration data, again for Gulton cells, in this case after three years of shorted storage. All these curves were run with the so-called standard set of conditions, C/10 charge for six minutes.

Curves A and C, the circles open and closed, indicate the range of curves that we got with no external resistors applied just to indicate what the variabilities from one group of cells to the other might be.

Curve B, for a 1,000 ohm resistor on the cells. And you can see here again at the end of 24 hours 1,000 ohm resistance would essentially have no difference between those and cells with no resistance applied at all except possibly the fact that here we are beginning to see a spread of more than 10 millivolts.

Still, if one were to apply, let's say, a 1.16 or 1.18 criterion, you might be tempted to accept all those cells.

Now 1,000 ohm resistance may indeed be perfectly acceptable for a given application, so you have to know what resistance you're trying to look at. Obviously, by the time you get out here to 48 hours, you have a distinct drop in the curve. It is beginning to be concave downward and I think the presence of a resistor then becomes obvious.

Here again is a curve for 500 ohms and you can see how clearly distinguishable that curve is.

Now most of the work to date has been with the so-called voltage decay test. We are now in the process of doing similar work with the voltage recovery test and also with the seven-day charged/stand test. We are looking at the exact shape of these curves and getting more quantitative data on what their inherent sensitivity might be.
I only have one set of curves to show you at this time. It's rather incomplete. But here's a set of curves for a voltage recovery test involving Gulton 15 ampere-hour cells.

(Slide 49.)

Here again the cells that we had to work with had been on short storage for three years and that may affect the results to some degree. But shown here are results for no external resistor, the circles for 500 ohm external load and for a 1,000 ohm external load.

I don't really know exactly what to make of this data yet. It appears that generally speaking, the cells with the lighter loads rose faster toward their final value than those with the 500 ohm load, but there's a lot of overlap and I'm not just sure what we are going to find out in terms of the over-all shape of the curve.

As you can see, this data happens to stop at 24 hours because all the cells except one -- this was just one low one here -- had reached a certain limit but we are extending this test also to 72 hours to see whether we can find out anything more.

So the outcome, hopefully, of this work is going to be some much greater insight into what is going on during this kind of testing, maybe a better way to present and display the data, and hopefully more knowledge as to how to interpret the data, and some recommendations for improving our approach to making these kinds of tests.

Thank you.

WEBSTER: Thank you, Dr. Scott.

Are there questions?

J. H. SCHULMAN: Joe Schulman, PSI.

Did you look into the question of how long you should leave the cell shorted?
SCOTT: To a limited extent. It is obvious that if you do-- I guess you're talking about leaving the short on after you do the discharge just prior to putting in the short charge. Is that right?

J. H. SCHULMAN: Yes.

SCOTT: We have looked at that, extending over a period from four hours to 24 hours, and don't see any difference; as long as you do a conditioning charge in the manner that I have indicated, it does not appear to -- in that range does not appear to make any difference.

J. H. SCHULMAN: What about open circuit recovery? Does it have any effect on that?

SCOTT: I don't know yet. We are looking at that, yes. Obviously again if you-- We do know, as other people have indicated, that if you don't do a full charge and a discharge within a short period of time prior to that voltage recovery test you can get any results you want.

In fact, just for information, when we took cells off of storage where they had been shorted anywhere up to three years, we immediately did a voltage recovery test every time, and usually the results were that the voltages never recovered to more than .2 or .3 of a volt after those long shorted periods.

So we know that that voltage recovery test following a long short down is not an indication of anything except the cell is completely discharged.

SULKES: Sulkes, USAEDC.

On the voltage recovery test or I should say voltage decay test, have you ever considered running it at low temperature so that the cell itself discharged portion would be separated out?

SCOTT: Yes, indeed. I think that's an obvious thing to do. We were not able to do it within the scope of this particular program but if anyone wishes to separate those two even more clearly, certainly that is
be done at low temperatures.

KRAUSE: Dr. Scott, if we continue to use these kinds of tests like the voltage decay test and we find some cells have 500 ohms apparently on them and others perhaps one K ohm, could you venture an opinion as to what the useful life of that cell might be? What is really normal? What do we do with these results in terms of screening cells?

SCOTT: Well, I was not prepared to venture such an opinion right now. I don't really know clearly what the answer to that question is.

The answer tends to lie— Well, let's say the question, if you have X ohms now, is that going to remain the same or go up or go down with further use of cycling of the cell? I am not prepared to say where it is going to go.

Generally speaking, I think there is a feeling that the longer the life or the greater the cycling, the greater the chance that the cell has some kind of resistive or internal discharge path, but I'm not convinced that that's the case.

DUNLOP: Two groups of data you presented showed one group of cells where there was sort of a tail-off at the end after about 48 hours, and another group of cells in which -- these were two different manufacturers -- in which they tend to be curving up, or there was not this tailing-off.

Now did you do a further analysis to try to determine if, for example, the question of the point that was brought up about the spacing, or what were the differences, did you find any differences between the two cells? Or do you account for the differences between the cells?

SCOTT: First of all your last question first.

No, we have not looked into the question of what the interplate spacing is in any differences between the cells, at least so far in the program. That was not part of the original purpose of the study. However, it certainly is a key point.
I think though that the reason for the difference in the shape of the curves that we are seeing may not be -- well, may or may not be associated with the spacing, but I think in the case where we saw the curves convex upward, I believe all those cells were in better condition. There was reason to believe that they were newer cells.

In fact, the only cells I believe that we saw of that shape were newer cells. All of the cells that had been stored over a long period of time tended to show some roll-off, at least after 48 hours, which indicates to me that there is something behaving as at least a small, barely detectable shorting mechanism going on inside the cell.

What I'm saying is that -- Well, one point I said I was going to comment on but didn't, and that is that it is interesting that if you plot and lock carefully at the shape of the curves plotted on a semi-long plot as we have here, for the curves with resistive loads for a certain number of hours they fall away -- a real good, straight line on a semi-long plot, and only after X hours do they begin to roll off and go down.

So that depending upon the size of the resistor you have, even on this kind of plotting you cannot distinguish, based on a straight line between a resistive load and no resistive load. You have to wait until that straight-line portion -- until you come to the end of that and then you can see the roll-off and that would be the value of extending the period of the test beyond 24 hours, it looks like right now.

ARMANTROUT: Armantrout, Philco.

On this particular batch of cells, I am familiar with them from my employment with TRW. I know they were shorted for this period of time.

What conditioning did you do to the cells prior to doing the standard charge retention test in the way of cycling? That's one question.

The other question is: They previously had an AC impedance test. Have you attempted to correlate any of
those results with the results you're getting now?

SCOTT: Your first question: Most of those results that I'm showing you here follow one cycle, a 24-hour-plus cycle prior to doing the charge retention test. It looks like one cycle, done properly, is adequate for conditioning for performing this test. You can do more but it doesn't seem to make any difference.

We did measure impedance, AC impedance of most of these cells from which the data you have was shown, after removing them from storage and taking off the shorts, after the first conditioning cycle following the short down, and those impedances were within the normal range that we see on fairly new cells.

For example, on the 12 ampere-hour cell, it is about four to five milliohms; on a 24 ampere-hour cell it is two to three milliohms.

CORBETT: I was very interested in the comment you made when John Armantrout asked his question, and that is -- or I guess it was just before that, what you said about the voltage on a semilog plot being very linear and then having a sudden dropoff.

If you would plot that on a linear time scale it would be much like what you get on a true charge retention test. It's a voltage time test for a battery discharge and you're simply, I believe, running out of capacity. You know, you've got some fixed shunt load and you're running out of capacity.

SCOTT: On a linear time scale it's a linear S-shaped discharge curve.

CORBETT: Right.

And if the question is you don't know what kind of capacity is in the thing in the beginning, then how do you know what kind of a shunt load is on it at the end? In other words, I think we're really back at the same problem again.
If you don't know how much capacity is there and what the cell internal resistance is, how can you make this test really meaningful?

And the follow-up to that is you made a comment about one lot of cells could be projected if you had a 1.19 criterion, that they would be accepted -- rejected with 1.19, and they would be accepted with a 1.16 volt criterion.

My question is why would you want to reject them with a 1.16 criterion, or a 1.10 or a 1.19 in terms of real usage?

SCOTT: Your first question is-- I think it may turn out that you have to accept a certain equivalent resistance in the cell in order to get you out of the noise level introduced by residual charge in the cell. I don't know what that number is, but I would hope that one could say, well, for a given size cell I need to detect -- I would like to detect 500 ohms. If I can detect that or anything lower it's all right, and anything above that I will accept.

Well, maybe by doing that you could avoid argument as to what the effect of the exact state of the charge is. That's a tricky point which is still debatable.

I think every user is still going to have to exercise some judgment as to what kind of criteria he wants to apply, and all that we are trying to do is really just to provide a data base on which to do some more cogitating.

On your second question-- I've forgotten the second question.

CORBETT: Why would you reject cells with a 1.16 criterion?

SCOTT: Well, it looks to me like if you had these full curves to look at, why not? That is, I think if you didn't have these full curves to look at, you could have -- a 1.16 could correspond to a 500 ohm resister whereas a
1.19 or 1.20 or even higher could correspond to two or three thousand ohms.

If you had your druthers, wouldn't you rather reject cells that had the highest equivalent resistance that you can?

CORBETT: If you know that they are brand-new, when you have a very limited amount of initial testing and initial cycling on the cells when the tests are made.

I think one of the problems we have is that we get some cycling done at the cell vendor and we come in and we match cells in-house, and you may keep the battery around for some time and you run the tests before you fly, and I'm not sure it is as valid a test then as it was when the cells were initially procured.

SCOTT: Well, you raise a good question. I'm not sure that the test—Probably the test is going to be a lot more valid and less argumentative when it is done on a brand-new cell than at any time thereafter. It could turn out that way. It looks like that is what is going to happen.

CORBETT: Cells certainly run for years after they have a very significant shunt resistance. You can put 100 ohms across them and they will just run for years and years if you have a trickle charge on your system, which most power systems have.

WEBSTER: The last question by Ford and then we have to break for lunch.

FORD: So far you have addressed yourself to the ohmic aspect, the resistance aspect of this testing. Have you considered the implications that the test results may be telling you about the electrochemical aspects of the cell as far as the amount of electrochemical precharge, and I use that term to clarify the term "precharge," electrochemical versus chemical.

Also, do you plan to look into the effect that the state of charge on the negative electrode has on either
type of test?

SCOTT: Certainly I recognize that the state of charge of the negative can have an impact theoretically. We have had problems in the past which suggest that that's the only explanation for what we see, is that with inadequate precharge when you discharge the cell all the way down to zero, if you are negative-limited, the negative potential controls the cell voltage and you don't know what the test means under those conditions.

However, the program that we are working on is very limited in scope and we won't be able to consider those refinements.

WEBSTER: Thank you, Will.

(Whereupon, at 12:45 p.m., the workshop was recessed to reconvene at 1:30 p.m. the same day.)
IV.4 THE ACCELERATED TEST PROGRAM: D. MAINS/NAD CRANE

We have approximately five more speakers left, and our first speaker will be Don Mains from Naval Ammunition Depot at Crane, Indiana, and he will be addressing the subject of accelerated tests.

MAIN: Thank you, Bill.

For the past several years we've been telling you about the accelerated test program, its progress at Crane. I'm glad to announce that it is under way, and we do have some preliminary results that I would like to present today so that we all see where we stand.

(Slide 50.)

To start off with I'll give a quick run-down on what we're talking about in the program.

The cells as they're received are fabricated into packs, and then each one of them runs through the baseline test. This gives us a basic package of information that is identical as far as environmental conditions on each battery.

Then they go into cycling on their given conditions. From that point then they're either removed per schedule which was developed through NASA and the Air Force or they fail, either with pressure transducers on them or without. And this then governs what type of post-cycling analysis is done, finally ending up with a physical and chemical analysis of the cell itself.

In addition to this, then, the data is analyzed for the statistical results that we can arrive at.
At the present time we have the star point in normal tests and have started some of the fractional factorial programs on the test. So this gives you some idea of how many batteries we have on test, also the relative number of cycles at test conditions ranging from about five to a little over 1800 at the present time.

When we receive the cells we also receive information, or all the test results from the manufacturer. So what we have done is taken a look at some of this data, correlated the pressure versus voltage -- I'm sorry; precharge versus voltage. We have the precharge, the amount of potassium hydroxide, and the potassium hydroxide concentration. This is to give you an idea of how the results are distributed for the precharge here.

For the amount of potassium hydroxide versus voltage.

And for the concentration of potassium hydroxide.

The voltage data did not reveal any major significant differences among the three different groups of data. However the results on the percent of KOH did give the most surprising, in that 30 percent seemed to give a lower voltage level than the other two. We seemed to have a low point at about 30.

We also looked at the same data compared to pressure. This was for the amount of potassium hydroxide.

This one is on the amount of precharge versus the
FIGURE 50

TESTING FLOW CHART

- Baseline
- Cycling
- Pressure
- Failures
- Post Cycles
- Physical
- Cell Failures

FIGURE 51

PACK NO. | CYCLES TO DATE | REMARKS
--- | --- | ---
65A | 5 | baseline
65B | 13 | 60°C increase chs 14
57A | 378 | baseline
59A | 6 | baseline
72A | 1381 | baseline
72A | 1293 | estimated - 200 cycles
73A | 527 | baseline
74A | 473 | start point
75A | 596 | baseline
74A | 486 | baseline
75A | 561 | baseline
92A | 25 | center points
92A | 3 | baseline
93A | 1903 | normal test conditions
94A | 912 | baseline
95A | 1900 | factorial baseline
96A | 909 | baseline

FIGURE 52

Preecharge (amp-hrs)

FIGURE 53

CC of Kon versus voltage (5 ± 1)

VOLTAGE

PRECHARGE VERSUS VOLTAGE (5 ± 1)
And this one is on the concentration of potassium hydroxide versus pressure.

Again, the differences are not very significant. But you can see that the 38 percent does give a considerably higher pressure, also a lot larger spread on the data.

We've done a similar analysis on the test results that G.E. did on the 16-hour charge. By the way, this was on the 72-hour charge on G.E. We did the same thing for the 16-hour charge that G.E. ran at 22 degrees Centigrade and compared it with the test results that we had on a similar charge at 20 degrees Centigrade, and the data came out, for all practical purposes, identical.

One last item: The purpose of the whole program is to come up with eventually with a prediction to failure.

This, I want to say, is a very preliminary approach on this; the first column being the pack numbers, the second being the scheduled failures. These are the numbers we have used on these particular packs to gauge out our removal cells. The numbers were arrived at by Dr. Landers of the Air Force.

The Crane predictions to failure use the information that Dr. Landers had arrived at. Plus we've taken the results of the first five cycles and inputted them using the Lannon technique, or the voltage to 1.25 volts to come up with the new predictions.

As more data becomes available, and as we begin to get failures, these will continually be refined. But I wanted you to see at least some of the results we are getting, and the idea that to the present time we have begun to predict. How good these are, only time will tell.
<table>
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**FIGURE 58**
IV.5 OSO TEST RESULTS: W. WEBSTER/GSFC

WEBSTER: Thank you, Dom.

The next topic of conversation is the results of the OSO characterization. These tests were performed by myself at Goddard.

The cells were procured by Hughes according to a specification which was modeled around the Goddard specification. It has a Hughes number of RS 31331-132. Essentially they have to have a negative-to-positive ratio greater than 1.5. It also specified that the precharge would be — represented a minimum of 35 percent of the excess negative.

Typically the cells had around 3.3 ampere hours of precharge, and they were a nominal 12. The manufacturer was General Electric.

Also these cells were stored, some of them were stored for prolonged periods of time in a nitrogen atmosphere.

The original contract was for an order of about 300 cells, and we envisioned at first having three satellites. The program has now been reduced to one. So we have an excess of cells that are qualified that are on storage now down at G.E.

Also I might mention that this technique has also been used, and quite successfully so, with Gulton Industries on the ATS-F program. We store a couple of hundred cells up there. They're stored up there in an Argon atmosphere, and are activated when we need them.

This makes for several advantages. One, you have a quick turn-around time when you need cells, and, two, if a program phases out essentially you have virgin cells, because they haven't been activated, and they become available for another program.

We ran a series of tests prior to conditioning. Let
me tell you what we're conditioning for and what tests they are.

This spacecraft consists of a voltage clamp system which has eight levels which, in origin, evolved around the QAO philosophy. However the voltage levels were curtailed quite a bit. It was found that on QAO the upper levels were never used, so that they were down-shifted to give us lower levels. The designation became 00, 0, 1, on up to 6.

In addition, the original intent of the program was to have a third electrode. The third electrode, however, for cost considerations, was deleted. So the actual spacecraft which will be launched, possibly in March of this year, will have simply a voltage clamp system, a charge rate of C/10 and eight levels, with no third electrode.

However, since we went through the characterization, accumulated the data, we're going to present it here, and then possibly people can make use of it.

Our initial conditioning of these cells at C/20 yielded 15.7 amper hour and very low pressures. At the end of 48 hours' charge we got in the neighborhood of 13 to 18 pounds per square inch absolute. Then they were subjected to short test, which they all passed.

The next part of our characterization was to subject the cells to a decade box for determining the optimum resistor to be used for the third electrode signal.

(Slide 59.)

The results of these tests are shown on this curve.

The way that this test was performed is that the cells were charged at a very low constant current and allowed to come to equilibrium for twenty-four hours before the data was obtained. The data was obtained by switching a variable resistor box in increments of 1, 2, 5 and 10 units of resistance. And then it would go 100, 1000, 10,000. And these data points were acquired at three different temperatures, 0 degrees, 15 and 25 degrees C. No attempt was made
to keep the pounds per square inch constant. But they came in pretty close. You can see it ranges from 23 to 28 pounds per square inch.

So you see what people have shown before; that is, that as the temperature becomes colder the signal becomes lower.

Also we are interested in obtaining—— We plot this resistance versus the power, not versus the millivolts. And this is because we feel that the power gives us the greatest sensitivity, that is, for one pound we see the greatest change.

(Slide 60.)

Next I was interested in—— The cell pack consisted of five cells, four of which were third electrode, so I was interested in what was the variability of signal under any one condition. So here we had constant temperature, current, .4 amp. The voltage had reached an equilibrium, it was not clamped, of 1.46 volts. And this is how the third electrode signal varied in the temperature in the pack from cell to cell.

And just a general statement I could make at this point is that if you were considering cells for flight use you would probably throw out the top cell: it seems way out of line with the rest.

(Slide 61.)

After completing that, our next step was to run a 0° 70-hour overcharge test. And the results of these will become very important. I wish you would kind of look at this and remember it, because as the characterization tests conclude, we had trouble with this particular test. So this is the initial findings.

Our voltage peaked at 1.52 and then came down. And our pressure stabilized at about 54 or 55 pounds per square inch absolute. And that shows—— It's plotted versus ampere hours in. It was a C/10 charge rate, 0 degrees C. for 72 hours.

Our next step was to run a series of standard capacity
checks; 'standard' meaning charging at C/10 rate at 0, 15 degrees C. and 25 degrees C., and discharge at C/2.

We found that at 25 degrees C. we got 15 and 0.5 ampere hours out, and at 15 degrees C. we got 15 ampere hours out, and at 0 degrees we got 14.3 ampere hours out, with the undercharge pressure being approximately 30 psi.

Our next step in the characterization was to run a burn-in cycle. The purpose of the burn-in cycle was to stabilize the cells prior to characterizing the cells for C to D ratio versus various voltage limits on the charger.

We ran approximately two hundred cycles at what we call Level 3 -- I'm sorry; Level 4, which was equivalent to 1.448 volts per cell at 15 degrees C. The emphasis will be on 15 degrees C. because that is the projected operating temperature of the spacecraft.

Our C to D ratio, while holding all these things constant -- that is, the voltage to charge current, the temperature -- ranged, in C to D ratio, over 200 cycles from 106 percent to 110 percent.

Having completed this, we went into the actual voltage characterization of the cells for the charger.

(Slide 62.)

The results are shown on this slide.

The charge rate for the spacecraft is 3.2 amps, the discharge rate is also 3.2 amps. The depth of discharge is 16 percent. It's a 36-minute discharge and a 60-minute charge cycle. And we characterized the three different temperatures, 0, 15 and 25 degrees.

The various charger levels would be represented -- 00 would be the first one you would read, 0, 1, 2, 3, all the way up to 6.

As I stated before, 15 degrees C. is the temperature at which the spacecraft would operate, so that's the one
RELATIONSHIP BETWEEN SIGNAL-ELECTRODE AND RESISTANCE AS A FUNCTION OF TEMPERATURE

1.100

FIGURE 61

FIGURE 59

PROJECT OSO-I

OD

(PRCHARGE TO DISCHARGE RATIOS OBTAINED FOR VARIOUS CONDITIONS OF SIGNAL-ELECTRODE VOLTAGE, CURRENT, & TEMPERATURE

1.41.1 = 3.2 a

FIGURE 60

CEU VOLTAGE

FIGURE 62

VARIABILITY OF SIGNAL-ELECTRODE UNDER CONDITIONS OF CONSTANT CURRENT, CELL VOLTAGE & TEMPERATURE

FIGURE 59

FIGURE 60
that we're most interested in.

To fill in that curve some more with some more characteristics: it was noted that between Level 00 and Level 3 the pressure stayed below 1 atmosphere; that is, the highest it got was 11.8.

The end of charge current between Level 00 and Level 3 ranged between a half of an amp and .4 amp. Then at Levels 4, 5 and 6, the last three data points on each curve, at 15 degrees C. in particular, the end of charge current was, at Level 4, .7 amp, at Level 5, 1.04 amps, at Level 6, 1.28 amps. And the pressure increased correspondingly: 17 psi, 23 and 31.

Without going into the details of what the characteristics were for the other temperatures -- that is, 0 and 25 degrees, it suffices to say that the currents were in the same general region, and also that the end of charge pressures were in the same general region; that is, generally speaking, out of these eight levels we have very low pressure cells.

Now what is also interesting to see is that up to about Level 4, which would be this point here, we're maintaining the cells at no more than about 110 percent of the spacecraft operating conditions. It's anticipated at the present time that the spacecraft will be launched either on Level 2 or Level 3, so that's 00, 0, 1, 2, 3, somewhere in this region we're talking about 107 percent C to D ratio initially.

Upon completion of this characterization of the cells at these voltage limits-- let me state also that these readings of the C to D ratio are accurate to 1 percent. That was our electric ampere hour integrator. And also, we would not go from one level to another until we maintained stability for a period of approximately ten orbits.

After completing this test we performed a spacecraft capacity discharge; that is to say, the cells were at 15 degrees C. and we discharged at the 3.2 amp rate until we reached 1 volt, the undervoltage point on the space-
craft. We obtained 14.3 ampere hours out.

We then performed a reiteration of the baseline capacity check at 15 degrees C. and obtained 15 ampere hours.

Then in trying to repeat the initial 0 degree over-charge test we had our problems.

(Slide 63.)

This is an attempt at the same test under the same condition, that is, C/10, 0 degrees C. And we are going to try to charge the cell for seventy-two hours. You can see what happened after approximately fourteen hours, where the ampere hours in was approximately 18 to 19 ampere hours in. The voltage had stabilized, but the pressure had gone up to about 72 psia. So we terminated the test by discharging at C/2, shorting the cells overnight, and then trying it again at C/20.

(Slide 64.)

At C/20, the same problem. It didn't go away. We had our safety limit set, an electronic pressure transducer over the top of the cell, at 75 psi. The voltage had tapered down to 1.49 but the pressure had continued to increase.

So then the question came, Is it oxygen or is it hydrogen?

Next you see here a curve, the pressure decay. This is open circuit decay, so it would lead one to believe that it was oxygen, indeed. It dropped down to less than an atmosphere in eight hours.

We have no explanation. We do have some more chemical analysis for salts in the cells which will be given tomorrow as a general package of analysis of approximately twenty cells we have at Goddard. It details such things as negative-to-positive ratio, capacity, and everything is what everyone thinks it should be.

We're going to fly the cells: we have no reason not to.
Why I say that is, we've had the cells on life cycle tests at Goddard, and now they're on test at Crane. For anyone interested in following the test at Crane, it's Pack 7C, and it has approximately 4000 cycles on it at 10°C, and Level 3.

In-house, we ran approximately 4000 cycles on some cells which Hughes had already run approximately 1500 cycles on. The results I have on this chart.

(Slide 65.)

This is operating under what we envision the nominal spacecraft conditions being: 15 degrees C, Level 3.

You can see the cycle numbers on the side: 200, 1200, 2100, 3100, 3900, and the note at the bottom indicating that 1500 cycles had already been run at Hughes. The end of discharge voltages are kind of typical, they're decreasing as they should, with life.

End of charge is clamped at Level 3, as I indicated, and it is spread on either side. The end of charge current is indicated as approximately .6 through all cycles. The pressure doesn't seem to be increasing or decreasing, it's approximately at one atmosphere. And the C to D ratio is fluctuating slightly between 107 and 109 percent.

So these cells seem to operate fine under their intended use. However they do not pass the cold temperature overcharge test. There is nothing in the analysis, and it was built according to essentially the Goddard spec. So we have no answer, and I just pass this information on for what it's worth.

That concludes my talk.

Any questions or comments?

KIPP: Did you have a real need to run the O degree C. overcharge at the C/10 rate? Was there a program need for that? Or was it just an arbitrary number?
WEBSTER: It was an arbitrary number.

KIPP: I was thinking of that versus 20, which is the normal Goddard test.

WEBSTER: It was arbitrary, but you will see that—Well, you don't have the background. But I do. It was run one time mistakenly at G.E. at C/20, and the data came in about the same level, about 13 pounds to 30 pounds, around that range, over the entire cell base. And we tried to repeat it at C/20 here, and of course we went—we would probably have gone over 75. We had not stabilized; we just stopped there for safety reasons.

FORD: I'd like to make the point that C/10 is very safe. It isn't a Goddard requirement.

WEBSTER: It's a Hughes requirement.

IRV SCHULMAN: I think, with respect to Mr. Kipp's question, just recently we have seen a phenomenon—exactly similar to this one. This was on a E.O. program where we tested some Gulton cells at 0 degrees C. at C/10 for sixteen hours. And they passed that test. But subsequent tests failed. The pressures went up. And we have no explanation for it.

WEBSTER: Was it also oxygen pressure, as indicated?

IRV SCHULMAN: It was oxygen pressure, yes.

WEBSTER: Okay. Thank you.

GROSS: Bill, did you cycle the battery at the low temperature before you started the overcharge test?

WEBSTER: No. This was—In fact, the place that it occurred was right after the—We had just finished our characterization, which probably went in the sequence of 15, 25 and then 0. And then we did a couple of capacity checks, as I said, at various temperatures. So we had not cycles at cold temperature prior to running it. However on subsequent testing we tried this about four times. So that
should suffice as some type of cycling. And it would never pass.

GROSS: Prior operation at higher temperature makes it difficult to get good performance at low temperature.

WEBSTER: Marty?

GANDEL: How well were you holding at 0 degree C. temperature?

WEBSTER: Unbelievably well. One degree. This is an extremely accurate set of experimental data. The ampere hour integrator was accurate to 1 percent, the current regulation was accurate to a hundredth of a percent, the temperature chamber was accurate to plus or minus 1 degree.

Fred?

BETZ: Concerning this characterization testing, the cycling you were running, about how many cycles did you run before you got equilibrium at each voltage?

WEBSTER: Typically, I'd say it would take approximately twenty-four hours after the cells had seen all this exercising; you know, we went through the burn-in and things like that. Once we got to characterize each level we would stay at it approximately one day, then observe the results for less than a 1 percent. Plus or minus 1 percent was our criterion. When the results did not deviate more than plus or minus 1 percent on the C to D ratio, then we said that's a good data point.

BETZ: Did you discharge for capacity at that point, so that you have capacity versus--

WEBSTER: No. We discharged at the end. That's what I meant when I said I ran the spacecraft capacity. And we only got out 14 ampere hours at that point.

BETZ: Could I look at your data later?

WEBSTER: Sure.
We hope to get out an X report on this in a lot more detail with a lot more data by March.

CORBETT: Bill, what was your DOD when you were characterizing the--

WEBSTER: 16 percent; which is what the spacecraft will operate at.

CORBETT: The other question I had: On the over-charge voltage problem, do you have any reason to believe that those cells are wetter than normal?

WEBSTER: Than normal? They have 35 c.c.s of KOH in there, according to the manufacturer.

Unfortunately, our chemical analysis, the way it was being run in-house at that time, was forcing an answer. And we came up with about 40 c.c.s.

What I mean by 'forcing,' they just analyzed for OH ions, and they assumed that it was 31 percent, rather than doing a weight determination and then analyzing for OH ions and coming up with a correct percent.

So forcing it and saying it is 31 percent, when we analyzed it it came out to be approximately 40 c.c.s, as opposed to the manufacturer who said 30. So I think the truth is probably very close in there.

And we don't have teflonated negatives, I don't believe.

So it's just another phenomenon.
IV.6 EFFECT OF RECONDITIONING: I. SCHULMAN/TRW

WEBSTER: Our next speaker will be Irv Schulman of TRW, and he will be addressing the subject of the effect of reconditioning.

IRV SCHULMAN: This afternoon I wish to report on the Fleet SATCOM battery test, in which the objective was to determine the effect of reconditioning cells during the eclipse period.

The Fleet SATCOM battery program, which is an Air Force program, the battery itself is made up of twenty-four nickel-cadmium cells, 24 ampere hour nominal capacity, and the cells are General Electric cells. There is electronic protection for overcharge, for both overcharge and cell reversal.

I might say the principal investigator on this program is Mr. Jim Pierce of TRW Battery Group.

The test itself was what we consider to be an accelerated life test, accelerated to simulate a five-year operation. We accelerated by cutting down on the total day period to both 12 and 16 hours, as I'm going to explain in a few moments. And we also cut down the solstice season from three months to approximately five days.

Now in this program there were two 12-cell battery packs, of which one pack was reconditioned after each eclipse season; and I will go through the reconditioning program in a few moments.

(Slide 65.)

This Vugraph describes the ten eclipse seasons of 45 charge/discharge cycles. And you can see, if you loop down to 22 and 23 and back up to 45 you will notice that seasons 1 to 5 -- on the righthand side, the seasons, it shows you the discharge time in minutes, and on the left you see seasons 1 to 5 with a cycle of approximately twelve hours, in seasons 6 to 10 with a cycle of sixteen hours.

Now let me explain that.
In Seasons 1 to 5 we considered that the battery was new, all batteries were in operation, and that the power supply, the solar array, was fresh. When we got to Seasons 6 to 10 we figured that the solar array was now degraded. And you will see when I describe -- well, it was degraded. And one cell in one battery had failed, in one of the three batteries on the vehicle.

Now since there is a constant power load, our systems people, with their programs, determined that the depth of discharge would increase from 63 percent depth of discharge in Seasons 1 to 5 to approximately 75 percent DOD in Seasons 6 to 10. Also, during the first five seasons the charging rate was at 2 amperes, and in Seasons 6 to 10 it was 1.6 amperes, again simulating a degraded array.

The discharges I mentioned were constant power discharges varying from 20 to 72 minutes.

The method of charging is a constant current charge to a temperature compensated voltage limit, and at the currents I mentioned, both 2 amps and 1.6 amps, after which we then switch to a trickle charge of .24 amperes, which is approximately the C/100 rate, for the remainder of the charging period.

Now another systems variation was the temperature variation that we imposed on this program.

(Slide 66.)

The baseplate temperatures were varied to simulate battery temperatures during mission. During the discharge the cells baseplate temperatures were kept at 21°C., and during charge they were lowered to 7°C.

Now when the battery reached to within about a half a volt of the temperature compensated voltage limit the baseplate temperature, again it was changed to 16°C. During the trickle charge it was again lowered to 7°C.

Now, again, these are all systems requirements that the vehicle will see.
We also had thermal switches to change the charge rate to a trickle charge if the battery temperature ever reached 27 degrees Centigrade.

This test was started in January of this year and is still going on. The tenth season was completed yesterday, and unfortunately the results have not been plotted for the tenth season, but I think I can explain them to you.

(Slide 67.)

The results are reasonably dramatic. Here is the reconditioned system for the first five seasons, and then, of course, with an increase in the depth of discharge there is the expected drop. And the curve continues down this way. The tenth season is right in line with these results. So there has been no drooping away. So the tenth season was as expected.

As far as the unreconditioned batteries, they started out at a lower level, but then with the increase in depth of discharge they come down dramatically. And, again, the tenth season follows right in line.

Now the requirement on the system is a 1.0 volts per cell end point. And it would seem to me that this experiment points out certainly the beneficial effects of reconditioning.

Thank you.

Any questions?

KRAUSE: Irv, what kind of reconditioning procedure did you use?

IRV SCHULMAN: I'm sorry; you're absolutely right. I forgot to tell you about that. Okay, so we'll continue.

Reconditioning is performed, of course, on one of the 12-cell packs at the end of each eclipse season, and the other pack receives the normal recharge and remains on trickle charge until the start of the following season.
The reconditioning of one pack went this way:

At the end of the last discharge of the eclipse season the battery is recharged in a normal manner, at a constant current of 1.6 or the 2.0 amps, depending on the season, to the temperature compensated voltage limit. It is then discharged at a constant power of 190 watts, a constant power load of 190 watts, until the first cell goes below 1 volt. Then discharge is then terminated and a 25 ohm resistor is connected across this 12-cell pack until its voltage reached 12 volts or until one cell reached 0.5 volts.

That ended the reconditioning, and the pack was then recharged and remained on trickle charge until the following season.

So there is no real attempt to go down to a zero situation. However it still seemed to be an effective method of reconditioning these packs.

I also might mention that although we simulated a cell failure there have been no cell failures in this program.

Now we hope to continue on with this far beyond the tenth season, although we have met the primary objectives of the program.

CORBETT: What was the actual test temperature?

IRV SCHULMAN: They varied all over the place.

CORBETT: What was the control temperature? What did you attempt to keep it at?

IRV SCHULMAN: Well, we kept it, as I showed you,--

CORBETT: Well I guess I must have missed that.

(Slide 66.)

IRV SCHULMAN: You can see the base plate temperature and the battery temperature up above it. And the reason for this, although it may seem peculiar, is that this was
a system requirement, this is what the Thermal people told us the system would actually impose on the battery itself.

CORBETT: Okay.

KRAUSE: Irv, was there a difference between the reconditioned and unreconditioned battery in the C-to-D ratio? In other words, it was going to some control limits. Were they behaving differently on charge?

IRV SCHULMAN: Not as far as I know. I don't have the actual C-to-D ratio. I think it is about, something a little below 1.10. But I don't have those figures. If you will give me a ring I can get them for you.

HELLER: I'm curious as to your rationale for reconditioning immediately after eclipse rather than before.

IRV SCHULMAN: Well I guess it was after the use of the system. There is really no reason to go one way or the other that I can give you; it's just probably a very arbitrary choice.

CORBETT: Another piece of data: We had an accelerated test similar to this as far as the profile, the actual discharge profile that has been on for about three and a half years. And we're running SAFT cells at 20C., -20, and zero. And we've had a need to recondition the cells at 20 about six times to keep above 1.1 volts at end of discharge. The packs at -20 and zero have not had to be reconditioned.

IRV SCHULMAN: Yes; but, you know, this test seems to be run in a reasonably favorable situation. It's run between--

CORBETT: It's moderately warm, I take it?

IRV SCHULMAN: It's from 7 degrees up to about 20-some-odd degrees. By today's standards I think it's reasonable.

I think, however, the experiment does point out that there is some benefit to the reconditioning.
FORD: I'd like you to comment, if you could, on just what reconditioning is doing. Is it really improving the energy storage content of the cells to 1 volt, or is it just a redistribution of the energy relative to the volts?

IRV SCHULMAN: My answer would only be a function of my vivid imagination.

My own opinion is that it improves the effectiveness of recharge. In other words, this thing may— You know, when you run a cycling program like this there's nothing that says your recharge is optimum to start with: there's no way of telling. Therefore you may effectively do the same thing by increasing the amount of recharge that you're putting into the cells which aren't being reconditioned.

I think reconditioning just improves their effectiveness.

HENDEE: I think you have to be very careful when you're discussing the effects of reconditioning. As some of you may know, we have a test program in which we've been doing real time testing on the same cells we have up in orbit. And now we have just been through our sixth season, and we have discharged these cells down to a low level and, exactly as you say, we got a great improvement in the end of discharge voltage. It's like where they're running just over 1.15 volts at the end of the normal eclipse, they rise up, after the number of cycles, as high as, say, 1.2 volts.

But, in fact, one interesting thing we found is that if you discharge the cells down to 1 volt, which we consider about the lowest acceptable voltage without the danger of going into reversal on some cells, we have no reversal protection. In fact, if you do a second discharge to that same voltage the capacity to that voltage does not change at all; if anything, there's a slight drop.

So although, provided you have adequate capacity in the cells, you can certainly improve your end of discharge voltage. It does nothing for you in the end of life in increasing the effective capacity of the cells.
IRV SCHULMAN: I think these results—I have to go along with the results I have shown. And it certainly seems sufficiently to raise the end of discharge voltage to give me a better feeling about the reliability of the system as a whole.

HENDEE: It certainly increases your end of discharge voltage when you have adequate capacity reserve.

IRV SCHULMAN: The end of discharge, going through cycles 6 to 10, is 75 percent of nominal capacity. And that's pretty deep depth of discharge.

NAPOLI: Does your spacecraft have the capability of monitoring the cell, the actual cell voltages during the discharge?

IRV SCHULMAN: I don't know. I would think not.

NAPOLI: If you're looking at the terminal voltage how do you know if one of these cells reaches—

IRV SCHULMAN: This is the way this particular test was run. There are other schemes of running reconditioning.

NAPOLI: I'm talking about in orbit, now.

IRV SCHULMAN: Well, in orbit there are other methods. One method is to put a high enough—Of course you have more time to put a high enough resistor in series with the batteries, so that if there is a reversal it is reversal at a very low rate.

Now we also have reason to believe that there is a recombination if you get the rate low enough. The hydrogen can recombine if the rate is low enough.

There is other work being done which I am not prepared to report on in this session. But we have a program going on investigating that phenomenon at TRW.

WEBSTER: Thank you, Irv.
Our next speaker will be Floyd Ford of Goddard Space Flight Center, and he will be presenting some data on 24-hour tests, I assume at Crane. Is that correct?

FORD: Yes.

For the benefit of those synchronous orbit buffs, I would like to share with you some data that we have generated on a 24-hour orbit test. And the reason I found this interesting is simply because, unlike synchronous orbit, we're experiencing 60 percent depth of discharge on a daily basis. And, in a nutshell, we have seen cells go for three years, which is over 1100 cycles, in synchronous orbit on a daily eclipse.

I'm just going to hit the highlights, because this test data is available from Crane by request through the appropriate channels at Crane. So I will just share with you a few moments of some of the summary, that we have seen.

(Slide 68.)

Incidentally, these are 6-ampere hour cells. They are pre-1970 vintage. And in case you don't know what that means, it means they're not built like they are today.

They are made by General Electric. They're some of the first cells that we got from G.E., with the nickel braze seal that we're very pleased with.

One group of these cells was put on a 24-hour charge, and it's basically a C/25 constant current with no voltage limit, and a C/2 discharge for one hour, for 60 percent.

There were four packs, and I won't even discuss the fact of 40 degrees C. for obvious reasons. It failed after just a little over a year of testing.

What I would like to do is bring to your attention some of the results that we've seen, particularly after about two and a half years.
You might note the end of discharge voltage, A-1. This is what we call our pre-life test discharge which is usually run at the rate that we recycle at as compared with one year, one and a half, two, two and a half.

Within 60 days of the test these voltages were down to the value indicated in one year. So, in a nutshell, what we can say is about 85 to 90 percent of the end of discharge voltage degradation in this cycle regime occurred in less than three months. And after that time we see a fairly-- You see, the numbers bounce around. I can't explain why they do, other than the fact that we select discrete cycles to look at and there is no attempt to average the end of discharge voltage over over the increment of cycles at that given time in the life of it.

I point out another fact which I want to make significant-- of significance that I will indicate later is the fact that pack 75-E was initially started at minus 20 degrees C. and we found out for this type of regime that the cells were almost impossible to operate there, both from a charge and discharge viewpoint. It was very difficult to get them charged without getting excessively high voltage and we could not sustain a full 60-minute discharge. But they were at minus 20 for about two to three months in the early life of the cells, the significance of which will come out in a few minutes.

THOMPSON: Why were they better at zero degrees than ten degrees?

FORD: Why were they better at zero than ten?

THOMPSON: Yes, why?

FORD: Because inherently batteries seem to operate longer at zero degrees C.

THOMPSON: How about that.

(Slide 69.)

100
FORD: What I've used here is what I consider the worst degradation which was at 20 degrees C. to show you, one, what the precycling discharge looks like on day one versus what it looks like in one year, which is this curve here (indicating), also followed by what it looks like in two and a half years, and the end of discharge criterion in this case is one volt per cell, of which you can see the amount of capacity degradation between the day one and two and a half years has dropped just slightly below rated value which is six ampere-hours.

What is also indicated here is a condition which we ran the test for with a specific application in mind, one of where you are using the value to supply full spacecraft load and then switch down to an essential or survival mode where the power may drop by a factor of ten, and that's what we attempted to represent.

That six is not six amps; that's .6. The decimal place got dropped a little bit, so that's 3.6 amps, two hundred voltage at one volt, and then dropping down to .6 of an amp, to show that the total capacity--

Keeping this in mind, let me move to the next one.

(Slide 70.)

This shows the performance of all three packs, using that type of test condition.

The interesting thing is that the total capacity or at least rated capacity is still in the cell, particularly as noted in .6 degrees C., that the seven and a half ampere-hours is practically that that we got at day one capacity but it is not available at the 3.6 amp rate.

I think this suggests a very high degree of polarization effect with cycle life, meaning that if you really need the capacity or if you have to have the capacity, it may not be available at the nominal rate but it certainly will be available at a much lower rate, in this case by reducing by a factor of ten.

(Slide 71.)
END OF DISCHARGE VOLTAGE DEGRADATION DURING
2.5 YEARS CYCLING AT 60% DOD
(24 HOUR CYCLE - 1 HOUR DISCHARGE)

<table>
<thead>
<tr>
<th>TIME ON TEST</th>
<th>123B (0°C)</th>
<th>76E (10°C)</th>
<th>1086 (20°C)</th>
<th>916 (40°C)</th>
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<tr>
<td>(YEARS)</td>
<td>Voltage at 80% Depth-of-Discharge</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>1.230</td>
<td>1.171(1)</td>
<td>1.246</td>
<td>1.144</td>
</tr>
<tr>
<td>1</td>
<td>1.129</td>
<td>1.166</td>
<td>1.113</td>
<td>0.910</td>
</tr>
<tr>
<td>1.5</td>
<td>1.096</td>
<td>1.157</td>
<td>1.048</td>
<td>0.947</td>
</tr>
<tr>
<td>2.0</td>
<td>1.129</td>
<td>1.116</td>
<td>1.106</td>
<td>FAILED</td>
</tr>
<tr>
<td>2.5</td>
<td>1.135</td>
<td>1.115</td>
<td>1.142</td>
<td>FAILED</td>
</tr>
</tbody>
</table>

NOTE (1) PACK 75E LIFE TEST INITIATED AT -20°C. DURING FIRST YEAR, TEMPERATURE CHANGED TO +10°C.

FIGURE 68

BATTERY DEGRADATION ON 24 HOUR ORBIT TEST NAD-CRANE

FIGURE 69

AMPERE HOUR CAPACITY AFTER CYCLING
2.5 YEARS AT 60% DOD
(G.E. 6.0 AMPERE-HOUR RATED)

<table>
<thead>
<tr>
<th>PACK #</th>
<th>TEMPERATURE</th>
<th>TOTAL AMPERES-HOURS TO 1.0 V @ 3.6 AMPERES</th>
<th>.6 AMPERES</th>
</tr>
</thead>
<tbody>
<tr>
<td>123B</td>
<td>0°C</td>
<td>5.78</td>
<td>7.65</td>
</tr>
<tr>
<td>76E</td>
<td>10°C</td>
<td>4.79</td>
<td>6.34</td>
</tr>
<tr>
<td>1086</td>
<td>20°C</td>
<td>4.75</td>
<td>6.27</td>
</tr>
</tbody>
</table>

FIGURE 70

FIGURE 71
Now I would like to go one step further and show you, after three years, the data I showed you for two and a half years, after three years what analysis revealed about the cell.

It is interesting to note, like synchronous orbit cells that we have subjected to teardowns, there were no appreciable evidence of the cadmium migration as exhibited by the graying of the separator. I will show you some examples of the separator later.

What I would like to point out, though, is very indicative of segments orbit testing, is the degradation. If you look very closely along the edge of the plate you will see where the active material has actually flaked off, and we found quite a bit of this in the cell.

But this pack was also tested first at-- Let me see if I happen to have the slide-- No, I don't have the other one.

This is an indication of what happens at zero, but the thing that is of interest is the fact that I mentioned earlier that it was tested at minus 20 and then placed at 10 degrees C., which showed significantly more deterioration than the pack did at zero degrees C. And we concluded from this that the deterioration on the plus 10 degrees C. test pack really occurred at minus 20 and was not attributed to the fact that it was at the plus 10 degrees C.

Now there will be some discussion tomorrow by a gentleman who is here today, and I won't go into it, where he is going to discuss what he considers some of the things that are affecting positive plate deterioration with the life, particularly in synchronous orbit.

(Slide 72.)

To show you a comparison, this is a cell from 20 degrees C. There was very little evidence of positive plate deterioration. It simply suggests that positive plate deterioration as applicable to synchronous orbit failure mode is much more prevalent as the temperature is
decreased.

Now, you're ready to question immediately, hey, I thought the colder we operated batteries, the longer they lasted. Well, I think that is generally true, but the colder you operate batteries at synchronous orbit, apparently the less you can overcharge them, and the exact mechanism that is causing the plate deterioration we're not sure of, and we're not going into that further; I don't want to preempt the discussion that comes up tomorrow.

But there is an indication that there are two or three factors that affect the positive plate deterioration.

I would like to emphasize from my experience with this test and other synchronous orbit teardowns that the failure mode that you have to contend with in synchronous orbit batteries is the plates themselves and not the limitation as the separator experiences in low-earth-orbit application.

(Slide 73.)

Speaking of separator, this shows you typical material after three years. I believe that was the 20 degree C. pack-- No, that's the zero degree C. pack.

(Slide 74.)

There is some evidence of migration in the 20 degree C. pack; nothing that I would call significant after three years of testing.

Thank you.

WEBSTER: Any questions?

CORBETT: Was that a C/20 charge rate on those?

FORD: Yes.

CORBETT: That's continuous all the time?
FORD: Continuous except for one 23-hour charge, one-hour discharge, and the charging is always C/20. And I make it clear I'm not recommending that. I'm saying that's the test condition that we're using.

CORBETT: We're making a test that is very similar. I'm sure you know about it by now. And we set the recharge coefficient at what it had to be to maintain and end-of-discharge voltage to some reasonable level, and I think we ended up with a charge and discharge rate at minus 20 temperature of something like 105 percent. And this ends up being something like a C/35 rate, and we still have those cells after about three years.

So I think it kind of agrees with what you say about the overcharge problem.

FORD: Minus 20 testing, I find with the ni-cads with the present design anyway, is a very difficult environment in which to get long-term satisfactory performance, particularly if you try to overcharge.

There is just one cardinal criterion you have to accept at below zero and even at zero, but you just don't overcharge the cell.

CORBETT: We use the potential control system where we can go to the C/35 limit but the pack itself can limit its own voltage to somewhere around 1.54 volts.

FORD: I can't remember the details but I know that we tried at minus 20 under one of the Crane tests where we set the C-to-D ratio up at 100 percent just to see how long the cells would run, and I know it was in excess of three months, which indicates a pretty high charge acceptance and charge efficiency at that rate.

And I think that is one of the explanations you have of why you don't overcharge because practically everything you pass through the cell at that temperature is being stored in an active mode.

NAPOLI: Joe Napoli.
Could you venture to guess or extrapolate from the two and a half years of testing what that translates to in actual synchronous orbit?

FORD: I knew somebody was going to ask that.

No, because I looked at this in light of the experience we have on synchronous orbit tests, and I've even plotted the data points as the data points we've plotted, and I'm sure you remember the curves you've gotten from us, and will be shown tomorrow also. But the answer, no.

I really would like to think we could extrapolate this but today I don't have the means to do it.

NAPOLI: It's not really an accelerated test compared to the program that the gentleman described earlier on the accelerated test plan that he had run?

FORD: Accelerated relative to true synchronous, equatorial synchronous?

NAPOLI: Right.

FORD: I could say in a sense it's accelerated and yet I don't know what the exact relationship is to the true synchronous profile because we're experiencing 365 discharges a day here, and in a synchronous orbit you get 88 of which they are not constant.

I think I've said it before and I really feel that the true synchronous orbit regime in some ways is one of the better environments that we've subjected batteries to, simply because as you go into that eclipse, each day you go a little bit deeper into the capacity and you get the effect of reconditioning to voltage for the subsequent discharge.

And I think from that viewpoint that the synchronous orbit is a pretty mild application where it is not a fixed depth of discharge every day. From my experience, the fixed depth of discharge are the most difficult to deal with in those situations where, on non-periodic
occasions you have the need to go beyond that point.

That's where we always run into a problem, is not maintaining end of discharge voltage, you know, for a fixed depth. It's where we have the need to go beyond that depth of discharge we find the bottom falls out, or at least voltage-wise it falls out.

DUNLOP: Did you measure the thickness of those points?

FORD: Yes. In fact, I believe the chemical analysis that will be discussed tomorrow includes these cells, and I believe it includes one of the virgin cells-- No, it doesn't. We have a virgin cell, one that was not cycled, that we haven't finished the analysis on, but we plan to do it. so we are looking at that, but I don't have that data here. Some of it will be discussed tomorrow.

WEBSTER: Thank you, Ford.

IV.8 POWER MODULE DEVELOPMENT: R. CORBETT/LOCKHEED

Our last speaker of this session will be Bob Corbett of Lockheed, and he will be talking about power module development using third electrode control.

CORBETT: What I really expected to talk on today was some third electrode testing that we got under what you might call accelerated test conditions, and this is in conjunction with a development program we had about two years ago, starting about two years ago, to develop a charge controller for a fairly old Lockheed power system, one that has been around for a long time, and one that has had some problems at high temperatures because of the problem of setting ten compensated battery voltage limits.

If you set them too high you tend to go into overcharge; if you set them too low you can't get the amp-hour utilization out of the battery. So we got a third electrode that was good for that application, and I've got some data on those accelerated tests.
However, I got kind of turned on this morning after hearing Mr. Woodward, who I consider really had some kind of refreshing comments to make, and I think we had some good discussion on low-cost systems after this, and it occurred to me that a lot of what we've done in this particular development meets the requirements for a low-cost program.

I don't know if there's anybody still interested in low cost. Now that all the battery engineers have left the room I think it might be worth taking a look at.

Most of this was presented at the IECEC and I won't dwell on it because it is not all strictly battery stuff. I would like to go through a bunch of slides rather quickly, just to fully advise you on what we did, and maybe after I've gone through them, to make some comments about one low-cost battery and charge-control systems, and what I think means low cost and also to discuss some of the accelerated test data and how the third electrode is holding up.

(Slide 75.)

The first slide is just a picture of a power module upside down, --

-- as it might very well be in space, in fact. Maybe the JPL people would be interested in what the polarization terms would be under those conditions.

This is a 2.6 ampere-hour battery in a power subsystem. The base plate that that is mounted on is part of the spacecraft, and that's the whole power subsystem right there.

The battery charge controller is a card on the end of the battery. There are two cards that take care of the instrumentation and the other power subsystem control and monitoring functions.

(Slide 76.)
The next slide is I think a picture of the battery itself.

The charge controller-- One interesting thing about this as relates to cost, the charge controller is tested with the battery as soon as the charge controller itself is built in manufacturing so the battery goes through its test with the charge controller, and it goes through cycling tests where you essentially look for some output out of the electronics that says indeed that charge has been shut off.

So the only thing missing from this is the actual power control switches which the charge controller drives.

The board is actually two charge controllers. One is based on a third electrode and the other is based on a multiple level system very similar to the OAO or I guess the Fleet SATCOM which you saw before.

You see the card on the left there.

We can go to the next slide right away if you will, please.

(Slide 77.)

That's the unit that the battery is mounted on, and that's a separately tested unit. You have this little power control setup that's tested and then the charge controller is tested and then the battery is tested with the charge controller on it and they all get mated at the spacecraft level.

We can go to the next slide.

(Slide 78.)

This kind of shows how it all happens. I think what is important here is that you've got a test at every stage so you haven't cut down on the amount of testing you do, I don't believe, but you've kind of broken the tests out into small enough segments that if you have a problem, it is much easier to check out. You don't end
up testing one giant module and trying to find out if you have a battery problem at the spacecraft level; neither do you wait until the battery is on the spacecraft before you find out whether it will work with the electronics.

Next slide.

(Slide 79.)

This is the charge control board. If it looks kind of symmetrical, it should because the charge controllers are quite similar.

Basically it is op and hysteresis kind of stuff where you shut off at one voltage and you have a certain amount of drop before you can reset the charge.

I think on the next slide we may have a functional diagram of how it actually works.

(Slide 80.)

That's a battery schematic and there are two third electrodes per battery, each of which is command-selectable so you have your choice, and we have thermostats and the usual kinds of things.

The third electrode, as I mentioned, are two per battery and they're switchable. You either switch one or another into the third electrode charge controller. The third electrode charge controller works on both the battery voltage and third electrode input.

There are other charge controller backups that work only on battery voltage.

Next slide, please.

(Slide 81.)

This is some performance test data taken early in the accelerated test period where we had a charge controller on the test setup, and this is data at a couple
of different rates.

I guess the only thing this really says is that the third electrode is not as sensitive to rate as battery voltage itself is, and it still gives you a fairly good ramp regardless of what the rate is. These are all at 25 degrees C.

I think in the next slide we show the variations with temperature. You’ve seen this kind of a thing before.

(Slide 82.)

We are charging here to basically the same cutoff level.

On of the things that was said this morning was the -- or I guess Floyd actually mentioned it, and that is the problem of how do you customize controller to the batteries. And it's a cost problem when you have to re-characterize the cells every time you get a new lot, and it seems to me that's been partially solved on programs like Fleet SATCOM and OAO where we have gone to multiple-level control.

And I think the only limitation on that is even when you've got control over the voltage level you can still miss them. You can still have voltage slopes which are so slight, that have insufficient rise to them that they cannot be effective controllers, and I think that is where the third electrode is useful.

So in our particular system we have four commands in the charge controller. And in the case of the primary charge controller we use the four commands to change the levels. The third electrode is switched off. In the backup we use the same things for the temp-compensated voltage level.

(Slide 83.)

These are systems test data that illustrate the variations of both battery voltage and third electrode
with temperature and it points out the third electrode is virtually useless in a range of 10 degrees up to probably about 30 or 40.

And it indicates also that battery voltage at fairly high temperatures is not a very good control signal because of the absence of slope.

Next slide, please.

(Slide 84.)

So the idea I was trying to tell in this slide, and it really isn't told because it doesn't differentiate between the amplitude of a signal and the slope that you get from a control signal, but basically there is a kind of a complementary between a third electrode and battery voltage.

One has a fairly good rise at the high temperature and the other is fairly reliable at the low temperature as far as indicating the end of charge signal. So we simply combined them in the primary charge controller on this particular power module.

Next slide, please.

(Slide 85.)

FORD: Why don't we take a coffee break and come back and you can finish your talk?

(Recess.)

WEBSTER: All right, Bob, we'll continue.

Bob represents our last speaker in this session, and then we will be moving on to Floyd's session, I guess, next.

CORBETT: Well, as I was saying before I was so tactfully interrupted, we have two charge controllers on this battery. One has a third electrode; one doesn't.
There's a story behind that that has to do with how nervous people feel about the third electrode as a control device and whether you should rely upon it or not. I think if you had it to do over you might just find they were exactly the same.

(Slide 86.)

The point of this, this primary curve, the one that has less slope, is the temperature-compensated voltage level at which charge is controlled by the primary charge controller. That's one signal level.

The other four levels off and on are the levels that are used in the backup controller, and you'll notice two things:

One is they are generally kind of lower than the primary and they have a lot more slope. And the reason for that is when you don't have a third electrode, you have to do what is traditionally done and that is to kind of overcompensate on the temperature to make sure that you don't get into the problem of a runaway battery because of a little extra heat injection from the spacecraft or some similar problem.

So what this gave us -- and I don't have the control diagrams here because I didn't think they were appropriate for a battery discussion. But what this gives you is a kind of a sharing, a kind of an oaring of the third electrode and the temp-compensated voltage signal which is dependent upon temperature and rate. At the relatively high temperatures and the relatively low rates, the third electrode takes over. At the relative high rates and low temperatures the battery voltage takes over.

It is difficult to say exactly when or how the respective sensors are responsible for control. We have seen during systems test that in changing temperature or in changing a regimen from one set of conditions to another that the third electrode may be used during the transient period. That is, there may be three or four cycles when you're changing conditions where the third electrode is really necessary.
And then once you get down to a balanced cycle again, then the battery voltage with the temperature compensation is the one that is really responsible for your control.

Now this has some advantages because it means that you really don't have to design your battery voltage levels for safety. You basically design them for amp-hours.

In practice, we've found out in the systems test that at rates around C/4 above 80 degrees, the third electrode pretty much has control, and below that, the battery voltage.

In the next slide we've got some additional data that comes from the systems test.

(Slide 87.)

This is from the two battery systems so these are battery voltages from a parallel battery system, two batteries in parallel.

We've got three temperatures: 19, 77, and 110. This, by the way, is the range we had for the design requirements and you can understand why we wanted to go to the third electrode.

These represent your third electrode signals. I think there's a divisor of 13 involved there so it is somewhere around a 270 millivolt signal, and that's our lowest third electrode level.

So there isn't really much of a difference between the third electrode voltage once you get up above room temperature as a practical control signal.

On the next slide I think I have some more systems test data that is in the same vein.

(Slide 88.)

You can see there that at the— It is probably not
apparent but at the higher temperatures you are actually under the control of the third electrode. I think that is kind of self-explanatory; I won't go into it any more.

I think I've got a couple of more slides, possibly. If I don't, a couple of Vufoils.

Maybe I can have the next one?

(Slide 89.)

This is more of the same kind of systems test data. What you've got there-- This is kind of interesting. You've got three different levels. As I mentioned, we got four levels. In the primary it's third electrode and the backup. It's battery voltage.

And this is the effect of level one. Let's see. I guess this is inverted. This is actually level three. This is level two and level one. And what you get is more of an overcharge so as has been pointed out, the actual operation and use of this system is kind of like an overcharge control. It is a recharge control at the upper end, and we haven't had the experience with this to know exactly how you change levels, but I'm sure it's, you know, based on a combination of looking at the spacecraft thermal data and the kind of end of charge or on-orbit testing discharge performance of the battery which would enable you to select exactly what kind of levels you wanted.

(Slide 90.)

This is more of the same kind of systems test data. This happens to be at room temperature; the other one was at 110. And you get the same kind of effect with the level change there; a little bit more overcharge, a little bit more of an end of -- higher end of charge voltage using the higher third electrode signal.

So it is about a 25 percent higher signal and it gives you about a five percent higher recharge coefficient.
And so the battery sets its own cycling regimen based on its capacity. We did that with a development test cell which I don't have any data on today, and that ran about a year. I think it was something like 3400 cycles, with an average DOD of about 85 percent.

The third electrode characteristics did not change throughout that entire test. The mode of failure was a cadmium short right from positive to negative. We opened up the cell and there was a little black spot where it appears as if some cadmium hydroxide had accumulated and later reduced, and we had a direct short which could not be removed by overcharge.

We did it with a subsequent pack, and this is one of the production group of cells. These are G.E. 6's, and this is a test at a relatively early point.

We're shutting off at 260 millivolts. We're getting an end of charge voltage of about 1.44; fairly good charge efficiency, something like, I guess, close to 90 percent; 106 percent recharge. And we're getting 70.8 percent DOD so it's about, I guess, 4.2 amp-hours out.
Now we're getting 1.151 end of discharge voltage. Again, the control cutoff is just preset; there is no change there.

The capacity is up somewhat, indicating that we're overcharging more so we are getting some advantage of the overcharge in terms of improved capacity, but we don't like the end of discharge voltage.

Let me just lay this on here for a comparison.

(Overlay 95.)

The third electrode, in terms of its shape, has been changed very much, and I really haven't thought about this data. I don't know whether it is a third electrode sensitivity change or whether it is a kind of an offset change in the pressure of the cell, whether we're recombining better than we were before or something of that nature which is dropping our signal down.

But in any case, a decreasing third electrode signal is giving us a higher end of charge voltage, a higher amp-hour input which is giving you a higher amp-hour output.

Let me show you what happened on readjustment. These are pretty much equilibrium data and we're not playing with the thing.

A readjustment was made right after the 2900 cycles or right around 3,000 cycles and we got back to a 1.47 cutoff but we had to drop down to a 225 millivolt third electrode cutoff.

Now comparing that with what we had before we made the correction --

(Overlay 96.)

-- you see that the battery voltage curves haven't changed very much.

In fact, if I slide it over, the actual slope of
the third electrode slope hasn't changed very much either so it appears to be kind of an offset change in the signal, either because of the electrode itself or perhaps because of an offset change or kind of a bias change in the pressure.

So now to compare 3,000 cycles one year for a thing which-- This is essentially a full discharge; it is not a hundred percent DOD but it is all that the battery can give at these kinds of rates for both charge and discharge.

(Overlay 97.)

This is kind of a comparison of where we were new and where we are after 3,000 cycles, and it says it really hasn't changed all that much if you neglect this offset change in sensitivity.

Now relating that to the charge controller, the level in this case a 260 would be a level two control for our particular controller. 220 would be a level one.

So this would mean that after one year of accelerated cycling, which perhaps is equivalent to perhaps two years or three years at 25 percent DOD, we ought to have to flick the switch and drop it down one level on the third electrode to maintain the same kind of recharge coefficient.

This is continuing. The voltage is not degrading; if anything, it is improving somewhat at the end of the discharge.

I'm very interested to see how this is going to continue.

That's the end of my presentation.

WEBSTER: Thank you, Bob.

Any questions?

SCOTT: Scott, TRW.
What style of third electrode was in the cells? In other words, how is it positioned with respect to the plate stack?

CORBETT: This is on the edge. It's normal to the plates. It is right against the side of the plates.

WADHAM: I'm very interested in your comment that you attributed the short circuit due to the cadmium migration. I've been asking around quite a bit to find out about this, and so far, you're the first person I've come across who positively states that cadmium migration would produce this.

A lot of people theorize this; you read it in textbooks and so on, that cadmium migration causes problems. But when you get specific questions you can't get a positive answer and say "Yes, we have identified this as cadmium migration."

How well did you analyze the short circuit to determine that it was really cadmium migration and wasn't some other short circuit which was causing the problem?

CORBETT: We did not analyze it very well at all. In fact, I probably only make the statement because I'm out of the main stream. I don't tend to read very much literature any more but I identified just based on the fact that for one, cadmium to me is the only really mobile material in the cell in terms of the fact that it can be dissolved in KOH.

From what I know if it, it is white when it is discharged -- I mean charged, and it is black when it gets reduced. And this was very black material. It looked kind of like carbon black.

I don't know. Are there any experts who can say how positive an identification that is?

WADHAM: I'd certainly be glad to hear from any experts on that.

CORBETT: How about Guy?
Guy Rampel is begging off on this one.

RAMPEL: I'm not that expert, but I would say if it is black it also could have been nickel oxide, and the best thing to do would be to analyze it. It might have been nickel oxide.

CORBETT: Is that mobile within the cell? Do you get migration of nickel oxide?

RAMPEL: Charged nickel oxide is quite mobile and can travel.

CORBETT: I was kind of elated by the fact that the cell failure occurred before the third electrode began to fall apart; mainly because I built a power subsystem around it I would not want to see the third electrode degrading very much.
V STORAGE EXPERIENCE AND MANUFACTURING IMPROVEMENTS: FORD/GSFC

FORD: The rest of the afternoon will be spent on storage experience and also manufacturing improvements and without further comment I would like to introduce Joel Lackner from the Defense Research Board of Canada. He is going to talk about battery maintenance in synchronous orbit.

V.1 BATTERY MAINTENANCE IN SYNCHRONOUS ORBIT: LACKNER/DRE

LACKNER: This paper will describe the results of some experiments at DREO by Ron Haines and myself.

The experiments were designed to determine a good method of maintaining ni-cad satellite cells in synchronous orbit.

Now our previous experience being with four satellites in near-earth orbit. One of the significant differences between near-earth orbit and synchronous orbit operation is that batteries in near-earth orbit are exercised or cycled more frequently and at limited overcharge whereas in synchronous orbit, the battery is usually called upon to provide power only during the eclipse cycle of 45 days occurring twice a year.
In between eclipse seasons the satellite is in continuous sunlight for 135 days, during which time battery power is usually not required in any significant amount. Consequently, the battery could be either dormant or subjected to some form of prolonged overcharge.

The depth of discharge on synchronous satellites tends to be greater, in the order of six percent DOD, with 80 percent maximum.

Loss of battery can also be more disastrous than for the near-earth orbit.

Some of the problems incurred with maintaining ni-cad batteries in either the open circuit or continuous trickle charge mode were discussed last year by COMSAT and Hughes Aircraft. The significant differences between those tests and ours are that our main charge was always high rate, C/10.

The tests at REO covered nine eclipse seasons spread over four and a half years of real time testing. The tests were done on the ground on a simulated eclipse cycler operating at room temperature, 65 to 75 Fahrenheit.

The objectives of the tests were to study the charge efficiency at high rate, C/10, and low rates of charge, C/27 and C/40, and also the charge acceptance as a function of the rate and amount of overcharge.

The aim was to evaluate the ability of varying charging regimes to maintain the battery without the benefit of reconditioning procedures in between eclipses. No specific reconditioning procedure was put into the program so as to simplify the battery management from an operational point of view.

To be consistent with the desire for low-cost satellite cells and to expedite delivery, the test cells were of a standard commercial D cell size but having the following special characteristics:

High rate charge capability; they could be charged
at C/3 and continuously at C/10.

The had a high negative-to-positive plate ratio of two-to-one; low carbonate level; and a polypropylene separator.

Seven battery packs of five cells each were subjected to seven different charge modes.

(Slide 98.)

The charge control was accomplished using cadmium, cadmium hydroxide coulometers with a DRL design. The coulometers were used on four of the battery packs: one, two, four and five.

The coulometer signal indicated the end of the main charge and initiated the overcharge mode as noted in the Vugraph.

Now the main charge is defined here as the period of time necessary to return a hundred percent of the amp-hour capacity withdrawn in the previous discharge.

Following the main charge the specific overcharge mode was initiated.

Although these tests were run on the ground, DREO and Communication Research Center have had several years' experience on continuous high rate charging at C/10 with or without coulometer charge control on the Alouette ISTS series with a reasonably good degree of success.

The coulometer was merely used for convenience rather than as a specific test in itself. There are various other types of charge control devices which could have been used.

(Slide 99.)

Battery packs one and six behaved as expected, showing high rate charging is essential and that even on overcharge a high rate is required with an adequate amount of overcharge.
Battery pack four proved that an adequate overcharge at a high rate was needed, and that overcharge currents of as low as C/100 could be noted using just a coulometer control.

Battery pack three was too low a charge rate, especially in main charge. That's where the charge rate was C/27 continuous.

On battery number four it just went up to a hundred percent put back in and then there was no overcharge. It was just at a very trickle of about C/100.

Battery pack two is overcharged at C/40.

Battery pack five is C/27.

One is-- Battery pack five is C/27, continuous.

And battery pack one is C/27 on a pulse regime.

Number six is the interesting one because we've got a continuous C/10 DC charge from day one until nine eclipse cycles.

Number two behaved well for five eclipse cycles and then tapered off as we'd expected.

Number five stayed reasonably constant.

Number seven is an odd one to figure out, but possibly points out that some open circuit stand isn't too good.

May I have the next Vugraph?

(Slide 100.)

This is the ninth eclipse cycle. We haven't included three and four because they dropped out. And number six is still up there, and five, one, and seven.

Number two is just barely holding in.
Now we picked day 25 out of the 45-day eclipse cycle because we've gone past the peak and you're still going with about a 71-minute discharge with a six percent depth of discharge, C/2.

If we may have the next Vugraph it will summarize some points.

(Slide 101.)

This is the voltage of the battery packs after 60 percent depth of discharge had been taken out on day 25. And we have one, three, five, six, seven, and nine eclipse seasons.

You will notice that battery packs three and four didn't even meet the first eclipse season.

Just prior to the sixth eclipse season, we did a reconditioning on battery packs two, three, and four to see if we could bring them back. It didn't do any good for number four, but it managed to improve three for that one eclipse season, and just maintained number two.

You will notice number seven has got a very odd characteristic in that for the first three eclipse seasons, it had a low voltage and then it hit a peak and stayed relatively steady.

Number six just had a steady decline.

And number five was about the steadiest of them all.

What happened to number seven I don't know. This is the one that had a C/10 charge but it was a five-minute charge on, 16 minutes off, giving you an average of C/27.

Now in conclusion, it would appear that the high-rate charging on the order of C/10 is more efficient than C/27 or C/40, and a reasonable amount of overcharge at a charge rate above the trickle charge is advisable to maintain capacity.
**FIGURE 98**

**TEST OBJECTIVES**

(a) To study charge efficiency at high rate C/10 and low rates of charge C/27 and C/40.

(b) To study charge acceptance as a function of the amount of overcharge.

(c) To study the advantages of pulsed charging.

**FIGURE 99**

**PROGRAM 1**

**BATTERY 1**

Charge 400 mA Coulometer to signal end of each charge and overcharge at average C/27.

**PROGRAM 2**

**BATTERY 2**

Same as Program 1 except overcharge averages C/40.

**PROGRAM 3**

**BATTERY 3**

Main charge and overcharge at continuous pulsing 400 mA to give average C/27.

**PROGRAM 4**

**BATTERY 4**

Charge at 400 mA. Coulometer to signal end of main charge and to control pulse repetition frequency in overcharge.

**PROGRAM 5**

**BATTERY 5**

Main charge 400 mA. Coulometer to signal end of main charge and to reduce charge current to C/27 continuously.

**PROGRAM 6**

**BATTERY 6**

Main charge and overcharge 400 mA. (Approximately C/10).

**PROGRAM 7**

**BATTERY 7**

Main charge and overcharge 400 mA pulsed to average C/27. (5 min "on" 10 min "off")

**FIGURE 100**

**VOLTAGE AFTER 60% RESIDUAL**

<table>
<thead>
<tr>
<th>Battery</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voltage</td>
<td>5.85</td>
<td>5.80</td>
<td>5.20</td>
<td>5.15</td>
<td>5.10</td>
<td>4.80</td>
<td>4.75</td>
<td>4.90</td>
</tr>
</tbody>
</table>

**FIGURE 101**
It should be noted that the cell design has to be compatible with the charge maintenance routine or disaster may occur. Cells with too low a negative-to-positive plate ratio wouldn't be able to tolerate high rate charging for long periods, especially during overcharge, without generating high pressures, high end-of-charge voltages. Cells with nylon will undoubtedly suffer from effects of oxidation on prolonged charging and there could also be some thermal balance problems unless the heat generated on overcharge could also be accommodated by the spacecraft. Now despite the above design considerations, it would still be possible to give the standard cells a high rate of charge for the major portion of the main charge prior to the overcharge region, and we should try to design our cells and maintain maintenance routines in which a way as to get the best out of the Ni-Cad system. The system can offer a high rate charge acceptance and it is inherent and is utilized every day in aircraft batteries and consumer appliances, so why not in satellite batteries, the main drawback being that there is a slight weight penalty due to lower watt-hour-to-pound value. Now these tests were completed two weeks ago so we haven't got a chemical analysis of the cells and we are wondering whether we should terminate them. So if there are any questions on what is the chemical analysis, we haven't yet done it.

FORD: Are there any questions?

GROSS: You specified in the cells you used that they be designed for high overcharge capability, high rate charge capability, so this connotes that the plate would probably be extremely thin.

Now if that's the case then you would expect that such a cell would have lower charge efficiency than the thick-plated cell. So therefore the fact that you need a high charge rate with these cells to have the efficiency you need may not be a valid conclusion for the plated cells.

LACKNER: You can achieve a high negative-to-positive ratio other ways as well.
V.2 EFFECT OF LONG TERM ACTIVATED SHORTED STORAGE: SCOTT/TRW

SCOTT: I'm going to talk about inactive storage on the ground rather than storage in orbit, as may be obvious from the title.

In connection with certain defects type programs, we have been feeling the pressure to stretch out certain cell procurements, spacecraft builds, and spacecraft storage over longer and longer periods of time, and it seems that every year the same question comes up:

We have certain cells on storage; are the cells still good? How much longer will they be good? And how much longer can they be stored and still meet mission requirements?

There has been a certain amount of data available for our cells that are stored one or two years. I would like to now present some results of work that is still ongoing that is applicable to cells that have been stored for as long as four years, shorted.

(Slide 102.)

The program in general is summarized here. The range of cells that we are looking at extends from six months to four years. At the moment we are looking at General Electric and Gulton cells. That data that I have to show you today is all on General Electric cells. We
will be getting similar data on Gulton cells.

Testing involves certain tests performed on the cells as cells, as whole cells, and then subsequent to that, teardown of certain cells and analysis and inspection of the cell internal parts.

Our general approach is to compare what we see after X years of shorted storage with what we know about those or similar cells when they were first put into storage.

Now granted our data bank on some of these cells, especially those that are four years old, is very limited, but we have done our best to surmise and synthesize data that might be typical of cells when they were first put in storage.

(Slide 103.)

The G.E. cells that were actually subjected to test and analyses are summarized here. There are seven cells, several different capacities, activation dates ranging from 69 to 72, the total life since activation up to five years, and the storage time one, two, and four years.

Now, rather than go through an exhaustive recitation of the data, I would just like to quickly summarize our general results and then show you some specific data on one interesting aspect of storage that we have come up with.

(Slide 102.)

Briefly put, we have seen essentially no significant changes that we can attribute to shorted storage up to four years with regard to leakage conditions around the terminals, or cell impedance following a few cycles to condition the cell, end of charge voltages or end of charge pressures compared to what they were during initial acceptance testing of those cells, maybe a slight increase in end of charge voltage but nothing to get excited about.

Discharge capacities were within the same range, in
general, that we observed when the cells were first put in storage.

After, the cells that were subjected to certain electrical tests were then torn down and inspection of the inside of the cells indicated that the cells looked, both visually and under low-power microscopic examination, to be about the same as they were -- as cells look today when they are new and as far as we can remember as to what they looked like up to four years ago.

A general analysis of certain selected physical and chemical characteristics of the plates, including thickness measurements, density, porosity, active material content, were all within the same range as they were when the cells were first started.

Now, wettability of the separator: We don't really have good data on wettability measurements for cells that were new four years ago, but the wettability appeared to be adequate.

The condition of the separator was quite good, certainly not outside of the range that we see on brand-new cells as we see them from the manufacturer.

We did a limited amount of electrochemical capacity and charge acceptance measurements on the negative plates. We were primarily concerned with the negatives because at least all of our capacity measurements indicated, from an electrochemical standpoint, that there was no problem with the positive electrodes in these cells.

The flooded hydrogen free charge acceptance of the sample negative plates that we tested was quite adequate and the discharge capacities were what we would expect to see in new cells.

There was, however, one type of change which I think is interesting, that we are somewhat concerned about, although this change does not appear to affect the immediate charge and discharge characteristics and other directly measurable characteristics of the cell.
One thing we have not done and hope to do is to do some extended cycling or life-testing of some of these older cells to see whether there are any effects that might develop from more extended cycling. We have not done that type of testing so far.

The area that I would like to end up on is that of electrolyte content and analysis of the separators and the plates. For this study we did not use the approach of extracting the entire plate stack and removing all of the electrolyte from the plate stack as a whole.

We separated the plates under nitrogen, individually removed plates and separators, and conducted electrolyte analysis on individual plates and individual separators so we could get some idea of what the distribution of electrolyte was in the cells from plate to plate and from separator to separator.

Generally speaking, there was some variation within any one cell, at least from one plate to another, one negative plate to another, or one positive plate to another, but that variation was small.

(Slide ’04 )

The results I would like to show you for four plates. This is out of a typical cell.

One of those, 02405, is one of the five-year-old cells that was stored for four years. I have expressed the electrolyte analysis in terms of total alkalinity which is the sum of hydroxide and carbonate equivalence, expressed them in terms of a unit of area of the plate material in the cell in order to sort of have a number which is independent of the size of the cell.

Without any numbers to compare exactly, to compare these numbers to, it is hard to judge but it is interesting that a theoretical analysis of the electrolyte distribution in these cells which assumes that the same amount of electrolyte is absorbed by each of the negative plates and each of the positive plates plus the two separator layers associated with the positive plate, if
you make that assumption, then you calculate, assuming that the amount of electrolyte in these cells, that the original volume was two and a half times the nominal cell capacity and that 34 percent KOH was used, making all those assumptions, you come out with the calculation that that number that you see up there should be 20 millequivalents for negatives and 18 millequivalents for positives.

So at least these numbers are the right order of magnitude for the plates.

The relatively high level for percent of carbonate, particularly in the negative plates, is of some concern although of course the exact effect of carbonate is controversial. I don't know what these numbers would be for a new cell although we do have some data for recently-obtained cells but the recently-obtained cells were put through a decarbonating process by the manufacturer, and these older cells were not, as far as I know, so that I don't have any real good older data to compare to.

(Slide 105.)

Finally, the most interesting set of data I think is for the analysis of the separators. These were separators that were removed from the cell immediately after disassembly, were immersed in distilled water immediately and titrated separately for -- well, titrated two steps, by a standard procedure for hydroxide and for carbonate.

The cells are of decreasing age as they are listed here. The two cells at the top were the oldest; cell 180 was next; and 025-06 was the newest cell.

Notice particularly, going from newer cells to older cells, in general the level of total millequivalents per unit area of separator material ranges over a factor of about three downward with age.

At the same time, the percentage of carbonate goes up.

Again, making the same type of assumptions
### Figure 102

**Test Cell Identification**

<table>
<thead>
<tr>
<th>Serial No.</th>
<th>Rated Capacity (Ah)</th>
<th>Activation Date</th>
<th>Time Since Activation (Yrs)</th>
<th>Storage Time (Yrs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>013-01</td>
<td>15</td>
<td>1969</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td>024-05</td>
<td>15</td>
<td>1968</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td>100-01</td>
<td>12</td>
<td>1968</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td>102-01</td>
<td>12</td>
<td>1968</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td>102-102</td>
<td>24</td>
<td>1971</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>105-102</td>
<td>24</td>
<td>1971</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>005-05</td>
<td>24</td>
<td>1972</td>
<td>2</td>
<td>1</td>
</tr>
</tbody>
</table>

**Figure 103**

**Cell Serial Number**

<table>
<thead>
<tr>
<th>Cell Serial No.</th>
<th>Sample No.</th>
<th>Total (OH⁻ + CO₃²⁻)</th>
<th>CH⁻</th>
<th>CO₃²⁻</th>
<th>Equivalent % CO₃²⁻</th>
</tr>
</thead>
<tbody>
<tr>
<td>013-05</td>
<td>1</td>
<td>2.21</td>
<td>1.61</td>
<td>0.60</td>
<td>27.1</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>2.14</td>
<td>1.60</td>
<td>0.54</td>
<td>25.2</td>
</tr>
<tr>
<td>024-05</td>
<td>1</td>
<td>2.36</td>
<td>2.44</td>
<td>0.90</td>
<td>32.8</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>2.34</td>
<td>2.37</td>
<td>0.99</td>
<td>32.7</td>
</tr>
<tr>
<td>100-01</td>
<td>1</td>
<td>6.55</td>
<td>4.00</td>
<td>1.15</td>
<td>18.0</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>6.15</td>
<td>4.95</td>
<td>1.20</td>
<td>19.3</td>
</tr>
<tr>
<td>102-01</td>
<td>1</td>
<td>4.80</td>
<td>4.25</td>
<td>0.35</td>
<td>11.7</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>6.67</td>
<td>5.90</td>
<td>0.77</td>
<td>11.5</td>
</tr>
</tbody>
</table>

**NOTE:** This value, expressed in the units used, allows direct comparison between different cell sizes.
regarding the initial distribution of electrolyte in the cell, it appears that these numbers here (indicating) are about what you would expect in a new cell, so that these lower values here represent a distinct loss of electrolyte from the separator, at least a loss of alkalinity.

We did not do a direct determination of weight loss or water content or other associated things which might give us a more detailed idea of what is actually happening here, but this change with storage life was the only significant shift in characteristics of the cell that we could find during our analysis.

One, I guess, point I did not make -- I guess it was not listed on that list of other characteristics -- was that in these cells we did measure the AC impedance of the cells following the electrical part of this test procedure and found it to be slightly higher than what we would expect in a new cell, like 25 percent or 50 percent higher.

Now, maybe when you're talking about percentage changes, it could be that 25 or 50 percent is quite significant: certainly if that reflects a drying state of separator it is consistent with the change in alkalinity content that is shown on this slide.

That concludes what I have to say on that.

DE BAYLO: What was your storage environment?

SCOTT: Up until about a year ago, it was about 70 degrees, plus or minus a few degrees. Then for the last year it has been about 60 degrees, plus or minus five.

BOGNER: Two questions.

Did you say that you calculated the total quantity of electrolyte in the cell?

SCOTT: No. We assumed an average value equal to two and a half times the cell rated capacity in milliliters.
BOGNER: With your analysis, though, you could not calculate the quantity of electrolyte that you really found in the cell?

SCOTT: Well, yes, we could have, by multiplying by the total plate area and the total separator area; assuming that it was uniform we could have calculated back. I did not do that.

It would have indicated proportionately— Well, you see, since the change was so much greater in the separator than in the plates, you would have to make a separate calculation and then add them up. I did not do that.

BOGNER: Were the cells discharged or charged?

SCOTT: They were discharged.

WADHAM: Did you find any signs of cadmium migration in these cells?

SCOTT: No more than we see in a new cell, which is some slight. You see it sometimes at the corners; sometimes there's a few pressure points that you can see, but no different than in cells that have, say, completed acceptance testing and might have gone through 10 or 20 cycles.

KRAUSE: I think I have a recollection that most of the G.E. cells over the past four or five years have been running around 3 c.c.'s per rated ampere-hour. I was just wondering if that might have some effect.

SCOTT: You've got to distinguish between what is initially put in and what you get when you finally get the cell. They are not the same.

DUNLOP: I guess the point I'm most curious about, I didn't get the time frame again. On the last slide you showed you said the time frame was the older cells to the newest. What was that time frame?

SCOTT: The one shorted longest had been shorted
for four years. They were a total of about five years old. They were made in '69.

DUNLOP: They were made in '69, so that was just a time sequence that you showed there? Those cells were all different times?

SCOTT: There were two cells that were five years old, I think two cells, two or three. And there were two or three that were three years old. And then there was one or so that was one year old.

DUNLOP: Well, I guess I would like to make a couple of comments here about what we've observed, and we're going to have a discussion on it tomorrow. But the initial numbers appeared to be -- well, I would say seemed to be quite reasonable.

We observed number of, typically, 11 percent of carbonate in the electrolyte initially. We have shorted cells for two years, but we have observed some increase but it is not anything like the numbers you're looking at.

If I read you right you're looking at numbers like 28 percent, 37 percent carbonate.

SCOTT: Those are from the ones that have been stored for four years. They were quite a bit lower-- Of course the ones that were only two years old were about the same numbers that you're talking about. They were in the order of 15 percent.

DUNLOP: It's surprising. Do you have any explanation for that seemingly very large increase in the carbonate percent?

SCOTT: Off-hand, if you're saying how that could occur during shorted storage per se, I don't have an explanation.

DUNLOP: Have you done any other analysis on your cell to try to correlate whether it's possibly from-- It's surprising you would have that much carbonate in your plates to begin with, I guess is what we're curious about,
and we're wondering where the heck this is coming from.

FORD: I want to clear up a point here. The implication is that this carbonate level, particularly on the older cell, is the result of storage and I don't know how you distinguish whether it's the result of storage or whether it was there to begin with because the higher numbers that I saw are not unlike some of the data we get from -- some recent data that we got from Crane on cells that were put into the test program out there in '69 and '70. So I don't know how you draw a line and say that the carbonate is increasing because of the storage, or where it is just reflecting that the carbonate level was that much higher back then than it is today.

I think the latter may be true. I don't think the data has indicated that the storage period increases the carbonate.

SCOTT: I think I said we have essentially no data for carbonate levels in new cells as of four or five years ago.

DUNLOP: Well, the initial levels on cells we got four or five years ago was about -- The amount that you measure by extraction of electrolyte is about 11 or 12 percent. That's what you measure on day one.

The same cells measured today, when they've been operated in a real time sense, measure about 18 percent. These numbers here are just twice as much as anything we have ever seen before, and I'm just curious as to whether-- Your comment was you don't think it has anything to do with the storage mode or something to that effect.

My comment is we have never seen numbers like this on a lot of cells that we've looked at, whether we're storing them shorted or whether we're running them on a real time basis. We have never seen anything higher to date than 18 percent carbonate, and that's after five or six years of real time testing of cells that were built in the same time frame at the same initial levels.

So what I guess I'm asking is I don't know whether the numbers you're talking about are real or not. They
seem like they are exceedingly high. I don't think you
can run a cell very well at that level.

I guess I don't know what I'm really asking.

SCOTT: I understand your point, Jim. You know, if
some of you have some reservations as to whether or not
we might be inadvertently contaminating our samples by
exposure to air, I can only assure you that we stood on
our head to not contaminate them by our handling proce-
dures.

In fact, one of the things we did was develop a
curve by which we could predict if we had to bring the
sample, the separator sample, out into the air. We ran
a series of tests exposing separators wet with KOH to air
for different lengths of time and plotted a curve and
extrapolated them all back to zero time.

I think it is good data. I don't have an explana-
tion for it. I can say, however, that we have had simi-
lar on older cells, especially those that have had more
cycle life on them. We don't consider 35 equivalent per-
cent exceptionally high.

DUNLOP: Let me make one more comment. If you have
levels like 35 percent carbonate, the next step is to try
to figure out whether that carbonate came from the plate
or whether it came from degradation of the separator. If
it came from degradation of the separator there is an oxi-
dation mechanism and you can determine that effect by
simply looking at the state of charge. You should get a
change in the state of charge of your negative electrode.

That's one way to try to determine whether or not it
is an oxidation mechanism or a carbonate in the plates.

SCOTT: Assuming that there is no mechanism for
oxidizing the separator in a shorted cell, I don't know how
that difference could arise from shorted storage.

LACKNER: Lackner, Canadian Defense Research.
I'm a little bit confused here. I was first under the impression that you had one lot of cells that you kept for five years and at periodic intervals you analyzed them, say four months up to five years.

Now are you saying, in reply to Jim Dunlop, that it is not all one lot of cells that you're testing?

SCOTT: That's right.

LACKNER: It could be a different manufacturer?

SCOTT: It's not one lot.

LACKNER: So how can you correlate things if they're not one lot?

SCOTT: I'm not trying to correlate anything. I'm trying to give you the data flat out. I don't know what the interpretation is at the moment, but all I'm saying is by normal acceptance-type tests we can find no appreciable problems of performance of cells that are five years old that have been stored shorted for four years.

And so, by our normal procedure, we would try them. I don't know what is going to happen.

LACKNER: All I'm suggesting is if you had bought them all in one lot and then tested them with time, taking four months, one year, a year and a half, and five-year storage, you would have had a more meaningful correlation.

SCOTT: Yes. I would like to have that kind of advanced planning and visibility involved here, but it never seems to happen.

MARGERUM: Margerus, from Hughes.

Did you say they were nylon separators?

SCOTT: They are all nylon separators, yes.
MARGERUM: And although you tested the cells for acceptance, had you run any long enough to know they do last?

SCOTT: No, none of the eight cells have been carried past just a repeat of acceptance testing which involves three or four cycles.

HARSH: I'm not too familiar with the term that you are using, or the units, "millequivalents," but I notice when you showed the plates from the oldest cells to the newest cells it appeared that there was not much difference between. I believe they were all on the order of 20 or whatever that number was.

And then on the other slide for the separator there was a difference on the order of six to two.

I wonder if you would explain where the rest of the electrolyte is.

SCOTT: I wish I knew, Bill. I would like to investigate that but right now I don't know.

KRAUSE: Is the manufacturer in agreement, or have you talked to him about the original electrolyte quantity in these cells some way to determine that perhaps a little better? If you assume that there was only 2.5 level of c.c.'s per amp-hour it would factor all those numbers up slightly.

SCOTT: I agree. I have not tried to do that. I think the change of a factor of three in the separator is the thing that I would like to point to first, and I'm sure that the level of electrolyte was not a factor of three less four years ago than it is today.

KRAUSE: I was not questioning the level of the separator. We see the same thing in our analyses, that with time the separator dries out, but rather that those percentage numbers of the equivalents of carbonate could be explained in terms of what Jim Dunlop sees. He has never seen anything higher than 18 percent. We don't see numbers anywhere near that high either.
If you assume there is less electrolyte then of course the percentage of carbonate that you determine is increased. That percentage number looks higher.

SCOTT: No, that's an equivalent percent based on total alkalinity. What I mean is that's the equivalence of carbonate divided by the total of OH plus carbonate.

KRAUSE: In weight percent.

SCOTT: No, in equivalent percent, the number of equivalence. You see, the presumption is that if you start out with pure KOH in a cell and you do not -- let's say you do not oxidize any separator, that all the carbonate has to come from metastasizing initially insoluble carbonates in the plates and for every equivalent of carbonate you exchange an equivalent of OH, so your total alkalinity remains the same.

Now we did check that point and indeed, at least in some of the newer cells where we were more sure of the amount of electrolyte initially there, we did confirm the fact that it looked like the total alkalinity was the same as it was initially, and we just exchanged some carbonate for some OH.

BOGNER: I just want to add one data point.

On a cell that had been cycled about 11,000 cycles, about 54 percent-- not 11,000, 1,100 cycles at 54 percent DOD, a 20 ampere-hour cell, it came out with 46 percent concentration of carbonate.

SCOTT: And no sign of a deteriorative separator in that cell, as I recall.

BOGNER: There was heavy cadmium deposits on it.

MC DERMOTT: When you found, going from the new to the old cells, that you went from something like six millequivalent down to two, were you making a direct correlation between wetness and dryness with those numbers, or did you actually take some weight measurements? Are you using millequivalents as sort of a measure of
wetness and dryness?

SCOTT: Well, the terms wetness and dryness are, of course, rather subjective unless you back them up with some analysis.

As far as I know, there isn't much quantitative analysis that goes along to say what is wet and what is dry. This is some of the, you know, some of the -- a few pieces of data along those lines that tends to define what is wet and what is dry.

All I can tell you is that the separators in those older cells appeared dryer. They did not appear bone dry by any means, but they tended to stick a little bit more, but not a lot more than the newer cells, and they just had a dryer appearances if you are used to, you know, comparing one with the other.

But what's dry and what's wet from a subjective point of view I'm sure I don't know in terms of electrolyte content.

FORD: Thank you, Will.

I think that during this past talk a couple of very pertinent questions were raised, and we have some data we will try to get together for tomorrow's session, which is a computer printout that Cran's made summarizing all the analyses they've done on a large number of cells, including three or four manufacturers. and I can't recall the numbers but I recall -- I'm not even sure of the units right now but we'll try to get the data for you tomorrow because I know some of the numbers were in the high 20's in the carbonate analysis, and I think it would be interesting to correlate that with this because the higher
numbers are definite for cells that have been on test for longer periods.

That doesn't necessarily say it was in the cell to start with, or that it came from the cell during the operation, but I think it would be of interest to compare those numbers and we'll try to get them for tomorrow's meeting.

Okay, I would like to move on. The first two discussions we've had were on storage. The next one is on manufacturing and processing. It is by Dave Pickett of the Air Force Aeropropulsion Lab, and the subject is Electrochemical Impregnation of Nickel Plaque with Nickel Hydroxide.
V.3 ELECTROCHEMICAL IMPREGNATION OF NICKEL PLAQUE WITH NICKEL HYDROXIDE: PICKETT/AFAPL

PICKETT: In response to some of the questions that were raised this morning, there are some people who are still interested in research on ni-cad batteries.

In fact, we have had an in-house program going for the past three or four years in which we have been looking at possible ways to improve the energy density of the ni-cad system and to better fabricate the plates or develop new methods for fabricating plates.

At the initiation of this program we thought we would first look at the electrochemical methods that had been proposed such as the Candler process and generally speaking, our first entry into the program was just to educate ourselves on how to make ni-cad batteries and plates and, in general, acquire an education.

We first looked at the electrochemical methods, as I stated, and we looked at -- or we considered methods that had been published in the literature by McIlery and Coburn and at the present time that we were initiating this study, we had a program going on at General Electric in which Dr. Kroger was in the process of developing the electrochemical method for impregnating positive plates.
I don't plan to talk about the negative plate process at this time. I will just limit myself to the positive and concentrate on that.

I would like to say that we've been helped to a great extent by Bell Telephone Laboratories in this work and when Dick Beauchamp's paper came out in the extended abstracts of the Electrochem Society in the fall of '70, we were quite interested, and we went and talked with him and he was of considerable help to us.

Our first entry into this field of electrochemical impregnation, we had bought a number of plastic tanks to try the low temperature processes that I just spoke of but the Beauchamp process is a high temperature process in which boiling solutions are used and sodium nitrite additive incorporated. And so we were at odds as to what to do with the plastic tanks.

So we found out by adding ethanol to the solution that we could lower the boiling temperature and use the plastic tanks quite well. And as a result of this we came up with another electrochemical method of impregnating positive plates.

Contrary to what some people may think, we did not develop the ethanol process just so we could drink in the lab.

But I would like to present to you at this time some comparison of the data that we have got with these various methods, and I think the first Yucograph shows some of the work that we did with impregnating nickel electrodes with just plain nickel hydroxide and not using any additives such as cobalt, et cetera.

(Slide 106.)

This shows the loadings that we obtained. This is just on a beaker level and these are three by three inch plaques coined about ten mils. on the edge, ten mils.
deep and a hundred mils wide.

I would like to point out here that we used nickel-copper electrodes in this process. In the Beauchamp process one normally uses an inert electrode such as platinum or platinum-plated titanium. We tried this and we got reasonable results with it but we thought we got a little better results if we used a nickel-copper electrode, so we proceeded with that.

You will notice here that we did get very good loadings with the process, on the order of 2.2 to 2.6 grams per c.c. of void in the plaque, depending on which manufacturer's plaque we used. And the porosity did have some bearing on that.

I have calculated current efficiencies for this deposition but I will wait until I get to the end of the presentation to discuss these, and maybe do a comparison between them and the ethanol process.

(Slide 107.)

These plates were given some formation cycles to see how they performed, and we also included a plate from a 34 ampere-hour vented ni-cad cell for comparison.

This (indicating) is cell five. Cell five is a conventional plate out of an aircraft battery.

Now these are flooded, positive-limited cells. They are three-plate cells, two plates being negative electrodes and one plate being positive, and they were first charged at 100 percent overcharge at the one-hour rate. And then the subsequent charges and discharges were made at the one-hour rate with 20 percent overcharge.

And after about ten or so cycles you can see that some of the electrodes that were electrochemically impregnated are beginning to fall off a bit in capacity but the other electrode keeps on going.

Well, there's a possible explanation for this on the next slide, but I would like to point out that in these impregnations I used a reagent grade nickel nitrate and
there is no cobalt or any impurities in this at all.

Okay, once we added the cobalt we got a little different results and I will just show one slide here to sum up the over-all performance of what we got from these formation cycles.

(Slide 108.)

The number of cycles that you see over here are those at which the capacity peaked out and then started to degrade.

The utilization of active material is not hear what you get in a normal positive plate.

We tried ten percent cobalt in the active material; in other words, the active material was ten percent cobalt-hydroxide, and 90 percent nickel-hydroxide.

(Slide 109.)

We obtained these loadings with various plaques. Now we have here displayed 15, 20, 30, and 40 mil plaques and we loaded those to a reasonable level, the 15 mil plaques.

We did another experiment and we tried to see how efficient the plates were at the various loadings, and you will notice loading times vary and the amount of deposit varies.

I think the interesting thing here, though, is that the capacity retention with respect to overdischarge or overcharge is much better with the cobalt added.

You see, here is the previous slide --

(Overlay 110.)

-- and then the one with the cobalt in there.

You got a much better capacity.

Now of course you see that this electrode is getting
TABLE 1
LOADING CHARACTERISTICS OF NICKEL HYDROXIDE ELECTRODES

IMPPREGNATED FROM AQUEOUS SOLUTIONS

<table>
<thead>
<tr>
<th>Plaque</th>
<th>Weight Before</th>
<th>Thickness</th>
<th>Plaque *</th>
<th>Current Density</th>
<th>Time</th>
<th>Loading</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plaque</td>
<td>Impregn.</td>
<td>Deposit</td>
<td>Before</td>
<td>Porosity</td>
<td>Deposit</td>
<td>Density</td>
</tr>
<tr>
<td>(30 mil plaque, Manufacturer A)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NW-30</td>
<td>16.659</td>
<td>9.372</td>
<td>0.010</td>
<td>0.73</td>
<td>2.59</td>
<td>0.65</td>
</tr>
<tr>
<td>NW-30</td>
<td>17.549</td>
<td>9.346</td>
<td>0.265</td>
<td>2.40</td>
<td>2.30</td>
<td></td>
</tr>
<tr>
<td>NW-30</td>
<td>18.800</td>
<td>9.072</td>
<td>0.071</td>
<td>2.43</td>
<td>0.30</td>
<td>2.30</td>
</tr>
<tr>
<td>NW-30</td>
<td>19.385</td>
<td>9.744</td>
<td>0.032</td>
<td>2.52</td>
<td>2.30</td>
<td></td>
</tr>
<tr>
<td>(30 mil plaque, Manufacturer B)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NW-30</td>
<td>17.378</td>
<td>9.094</td>
<td>0.010</td>
<td>0.73</td>
<td>2.59</td>
<td>0.30</td>
</tr>
<tr>
<td>NW-30</td>
<td>17.941</td>
<td>9.373</td>
<td>0.265</td>
<td>2.40</td>
<td>2.30</td>
<td></td>
</tr>
<tr>
<td>NW-30</td>
<td>14.791</td>
<td>7.250</td>
<td>0.020</td>
<td>0.73</td>
<td>2.59</td>
<td>0.30</td>
</tr>
<tr>
<td>NW-30</td>
<td>14.425</td>
<td>7.250</td>
<td>0.020</td>
<td>2.59</td>
<td>2.30</td>
<td></td>
</tr>
</tbody>
</table>

* Porosity for manufacturer A was supplied by vendor, Porosity for manufacturer B was measured using water imbibition.

FIGURE 106
FORMATION CYCLES FOR NICKEL HYDROXIDE ELECTRODES, IMPREGNATED FROM AQUEOUS SOLUTIONS ELECTRODE: CHEMICALLY CHARGED: CHARGE CUT, CHARGE DISCHARGE, CHARGE, DISCHARGE: NO COBALT.

FIGURE 107

- X - CELL 1
- O - CELL 2
- A - CELL 3
- B - CELL 4
- C - CELL 5

TABLE 2
LOADING CHARACTERISTICS OF NICKEL HYDROXIDE ELECTRODES

IMPPREGNATED FROM AQUEOUS SOLUTIONS

WITH COBALT PRESENT

<table>
<thead>
<tr>
<th>Plaque</th>
<th>Weight Before</th>
<th>Thickness</th>
<th>Plaque *</th>
<th>Current Density</th>
<th>Time</th>
<th>Loading</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plaque</td>
<td>Impregn.</td>
<td>Deposit</td>
<td>Before</td>
<td>Porosity</td>
<td>Deposit per</td>
<td>Density</td>
</tr>
<tr>
<td>(50 mil plaque, Manufacturer B)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NW-35</td>
<td>8.202</td>
<td>1.947</td>
<td>0.040</td>
<td>66.2</td>
<td>2.16</td>
<td>0.30</td>
</tr>
<tr>
<td>NW-35</td>
<td>9.257</td>
<td>2.057</td>
<td>0.070</td>
<td>2.34</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NW-35</td>
<td>9.828</td>
<td>3.766</td>
<td>0.080</td>
<td>2.34</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NW-35</td>
<td>10.405</td>
<td>4.594</td>
<td>0.090</td>
<td>2.34</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(50 mil plaque, Manufacturer B)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NW-25</td>
<td>13.354</td>
<td>5.455</td>
<td>0.025</td>
<td>78.2</td>
<td>2.34</td>
<td>0.35</td>
</tr>
<tr>
<td>NW-25</td>
<td>13.354</td>
<td>5.455</td>
<td>0.025</td>
<td>2.34</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NW-25</td>
<td>17.751</td>
<td>8.599</td>
<td>0.034</td>
<td>2.34</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NW-25</td>
<td>20.525</td>
<td>9.224</td>
<td>0.034</td>
<td>2.34</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NW-25</td>
<td>20.525</td>
<td>9.224</td>
<td>0.034</td>
<td>2.34</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* Porosity for manufacturer A was supplied by vendor, Porosity for manufacturer B was measured using water imbibition.
about 9-1/2 ampere-hours per cubic inch, and that seems a bit unbelievable, and we don't always do that. But in this particular case that's what we got.

(Slide 111.)

Here are some of the results with the 20- and 30-mil plaques, just for some comparison.

(Slide 112.)

Here again is a 3 mil plaque and the curve on the bottom is for the aircraft cell again.

And so forth.

Well, anyway, to sum it all up, this is what the results look like.

(Slide 113.)

The number of cycles we got on the plate before the capacity began to fall off increase considerably, and this depended upon which manufacturer's plaque we used to some extent.

I might say at this point that manufacturer A's plaque was made from 255 Inco powder whereas the other was the normal 287, I believe, that is used.

The percent utilizations of active material are quite phenomenal, and of course this can't be explained on one electron change occurring in the discharge reaction.

We would also like to explain that during the cycling of these plates that eventually you see the thing peaking out up here.

(Slide 114.)

It goes on out but it doesn't fall off completely to zero. The plate capacity does remain up there with the increasing overcharge versus cycle life.

(Slide 115.)
FORMATION CYCLES FOR NICKEL HYDROXIDE ELECTRODES IMPREGNATED FROM AQUEOUS SOLUTIONS ELECTRO-
CHEMICALLY 1C CHARGE, C DISCHARGE, 30% OVER-
CHARGE, 10% OF ACTIVE MATERIAL, COBALT HYDROXIDE
ELECTRODE THICKNESS = 0.010 - 0.020

FORMATION CYCLES FOR NICKEL HYDROXIDE ELECTRODES IMPREGNATED FROM AQUEOUS SOLUTIONS ELECTRO-
CHEMICALLY 1C CHARGE, C DISCHARGE, 30% OVER-
CHARGE, 10% OF ACTIVE MATERIAL, COBALT HYDROXIDE
ELECTRODE THICKNESS = 0.010 - 0.020

FORMATION CYCLES FOR NICKEL HYDROXIDE ELECTRODES IMPREGNATED FROM AQUEOUS SOLUTIONS ELECTRO-
CHEMICALLY 1C CHARGE, C DISCHARGE, 30% OVER-
CHARGE, 10% OF ACTIVE MATERIAL, COBALT HYDROXIDE
ELECTRODE THICKNESS = 0.010 - 0.020

FORMATION CYCLES FOR NICKEL HYDROXIDE ELECTRODES IMPREGNATED FROM AQUEOUS SOLUTIONS ELECTRO-
CHEMICALLY 1C CHARGE, C DISCHARGE, 30% OVER-
CHARGE, 10% OF ACTIVE MATERIAL, COBALT HYDROXIDE
ELECTRODE THICKNESS = 0.010 - 0.020

FIGURE 110

FIGURE 112

FIGURE 111

FIGURE 113
Now for some comparison with the ethanol process:

These are pretty much the same. There isn't much difference here. The loadings are quite good. Here I've also included the amount of thickening that we get on the plate as a result of the impregnation. This is usually not more than a couple of mils but there are exceptions. (Indicating.)

And if you can control your impregnation conditions you can avoid this.

(Slide 116.)

Okay, the formation cycles again. The bottom curve is from the aircraft cell again, and this is the same cycling regime that I gave you for the other plates.

The performance of these plates was about the same as we had with the aqueous solution.

We also tried various additives in the plates such as manganese and cobalt and cadmium, et cetera, to see if we could improve the capacity of the plates any.

(Slide 117.)

These are some of the plates we tried. We had no problem loading to about the same levels with this. The thickness increase in the plaque wasn't that great. We tried one solution in which we had increased concentration of nickel and we got more thickening there in the plaque. That's plaque number 13A-30, the last one. (Slide 118.)

Anyway, the results of these showed that the additives did help to some extent to stabilize the capacity and the lower curves going on down here are just almost in the inverse order as I presented them to you. This one (indicating) is the one with the manganese in it. This one (indicating) is manganese and cobalt. This is the one, the last one, with just cobalt. And then this one here (indicating) has the cadmium and cobalt combination.

(Slide 119.)
### Table 8

**Performance of Nickel Hydroxide Electrodes Impregnated from Aqueous Solutions with Cobalt Present**

<table>
<thead>
<tr>
<th>Plaque Thk.</th>
<th>Measured</th>
<th>Utilization</th>
<th>A-10°</th>
<th>A-5°</th>
<th>Thickness</th>
<th>Cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>(A-H)</td>
<td>(A-H)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>.05 mil plaque, Manufacturer B</td>
<td>1.580</td>
<td>1.825</td>
<td>105.8</td>
<td>9.98</td>
<td>62.2</td>
<td>2.7</td>
</tr>
<tr>
<td>.10 mil plaque, Manufacturer A</td>
<td>1.580</td>
<td>1.825</td>
<td>105.8</td>
<td>9.98</td>
<td>62.2</td>
<td>2.7</td>
</tr>
<tr>
<td>.15 mil plaque, Manufacturer B</td>
<td>2.475</td>
<td>2.545</td>
<td>105.6</td>
<td>10.6</td>
<td>65.2</td>
<td>10.9</td>
</tr>
<tr>
<td>.20 mil plaque, Manufacturer B</td>
<td>2.790</td>
<td>2.545</td>
<td>105.5</td>
<td>7.33</td>
<td>65.1</td>
<td>7.3</td>
</tr>
</tbody>
</table>

**Figure 114**

### Table 4

**Loading Characteristics of Nickel Hydroxide Electrode Impregnated from Ethanol Solutions**

<table>
<thead>
<tr>
<th>Plaque Wt.</th>
<th>After Wt.</th>
<th>Thickness</th>
<th>Crystals</th>
<th>Current Loading</th>
<th>Deposition Before</th>
</tr>
</thead>
<tbody>
<tr>
<td>(grams)</td>
<td>(grams)</td>
<td>(inches)</td>
<td>(mil)</td>
<td>(amps/m²)</td>
<td>Deposition Time</td>
</tr>
<tr>
<td>.05 mil plaque</td>
<td>1.500</td>
<td>1.125</td>
<td>5.0</td>
<td>0.0229</td>
<td>0.0364</td>
</tr>
<tr>
<td>.10 mil plaque</td>
<td>1.500</td>
<td>1.125</td>
<td>5.0</td>
<td>0.0229</td>
<td>0.0364</td>
</tr>
<tr>
<td>.15 mil plaque</td>
<td>1.500</td>
<td>1.125</td>
<td>5.0</td>
<td>0.0229</td>
<td>0.0364</td>
</tr>
</tbody>
</table>

**Figure 116**

### Table 5

**Various Additives Present**

<table>
<thead>
<tr>
<th>Plaque Wt.</th>
<th>After Wt.</th>
<th>Thickness</th>
<th>Crystals</th>
<th>Current Loading</th>
<th>Deposition Before</th>
</tr>
</thead>
<tbody>
<tr>
<td>(grams)</td>
<td>(grams)</td>
<td>(mil)</td>
<td>(mil)</td>
<td>(amps/m²)</td>
<td>Deposition Time</td>
</tr>
<tr>
<td>D-8 - 30</td>
<td>11.175</td>
<td>1.125</td>
<td>5.0</td>
<td>0.0229</td>
<td>0.0364</td>
</tr>
<tr>
<td>D-8 - 30</td>
<td>11.175</td>
<td>1.125</td>
<td>5.0</td>
<td>0.0229</td>
<td>0.0364</td>
</tr>
</tbody>
</table>

**Figure 117**
This kind of summarizes it up as to what we were getting in the performance of these things. Although there is no great improvement in what we were getting with just cobalt, it didn't hurt it any.

(Slide 120.)

This shows the current efficiency for loading the plaques from aqueous solutions using the 15 mil plaque.

Now I would like to say at this point that the efficiency that you get for the loading, regardless of which methods you use, depends upon the plaque porosity, the amount of loading, and I would imagine on the pH, so the solution to some extent, although I have not investigated this.

But the maximum efficiency we were getting with the 15 mil plaque is about 35 percent. This is measured as ampere-hours of nickel-hydroxide which we put in over the amper-hours of current that we used to deposite it, times a hundred.

On the ordinate we have loading in terms of ampere-hours per cubic inch theoretical.

On a 40-mil plaque, this is much more efficient. We got as high as about 55 percent efficiency.

With the ethanol process on a 30-mil plaque we got about 25 percent efficiency.

And with the aqueous solution, it was somewhere around 35.

(Slide 121.)

This is charging efficiency of some cells we made up with the plates from the aqueous solution and what I have plotted here is charge efficiency, the charge in over the charge out times a hundred over the state of charge or percent of full charge of the cell. And I think the curve speaks for itself. We got quite good charging efficiency with these plates.
These are plates with the ten percent cobalt hydroxide in active material.

I would like to say that our idea that the ten percent -- it was not our idea to put the ten percent cobalt in there. This has been looked at to some extent in the past. Harvey Seider and co-workers at Gulton some years back looked at the effects of cobalt and manganese and Hans Kroger investigated the effect of cobalt in the nickel-hydroxide electro program that General Electric had with us.

But I do think the data shows that, number one, if I may make some conclusions based on this limited data for just bigger level production, that you do get a good plate with electrochemically impregnated plaque and under some circumstances it could be superior to your conventional impregnation process.

Number two, it's a much easier process. We can do the deposition in anywhere from one to two and a half hours and it also protects the integrity of the sinter in that you don't get corrosion of the plaque during the impregnation process to the extent that you do in a conventional vacuum impregnation.

We have scaled the process up to building 20 amper-hour vintage cells in-house, and these are currently being tested on cycle life. We have a contracted program with Ilia Tech in which they are building the 15 amper-hour cells using this process.

And Bell Telephone has scaled their process up and they are producing cells with it so it has proven to be -- both methods have proven to be very viable techniques for impregnating nickel electrodes and they both work quite well.

We have also made some plates for various people on Air Force contracts for use in nickel-hydrogen cells, and the results of those tests aren't in yet, all of them aren't, so I won't comment any further on that, and I will conclude my presentation on that note.

FORD: Are there questions?
NAGLE: Why did you choose ten percent?

PICKETT: Because of some work that Hans Kroger did at General Electric, and this is in the final report that he wrote.

NAGLE: Harvey's work showed 17.

PICKETT: Well, that's for a vacuum impregnated process though. And Kroger's results are in conflict with that, and I think we'll go along with those.

THORNELL: During formation, was there any change in plate dimension?

PICKETT: There was some. Of the values that I show on some of these --

(Slide.)

... the thickness gain that you see here is the difference between the initial plaque thickness and the final electrode thickness. And all the calculations that I did based on ampere-hour per cubic inch are based on this final thickness.

It depends on what type of plaque you use how much thickening you get during the formation, but you do get some and it is not as much as you get with a conventionally impregnated plate.

FORD: Any other questions?

(No response.)

(Whereupon, at 5:00 p.m., the meeting was adjourned to reconvene at 9:00 a.m. the following day.)
HALPERT: Good morning. I want to welcome you all back to the second session of the 1974 Battery Workshop.

We will continue with the session of storage experience and manufacturing improvements with Floyd Ford as chairman.

FORD: Thank you, Gerry.

V.4 PLATE LOADINGS FOR THE F"E CELL: FORD/GSFC

I saved this. I think it might have been a Vugraph intended for today's meeting; if someone would like to come up and claim it. It's interesting what you find on tablecloths after technical people get together.

Several people asked me yesterday, "I thought we were going to talk about plate loadings" well the answer is we're going to talk about plate loadings. But my emphasis this morning, contrary to what it has been for the last three or four years in the workshop, I'm not going to be criticizing the battery manufacturers; I'm going to be pointing my fingers at the users. And I challenge you to take heed of what I'm trying to tell you.

There has been an evolution process on nickel-cad cells that has gotten to what I consider a very disturbing situation. The evolution process is simply that this question about nameplate versus actual capacity.

We've all seen 20-ampere hour cells and other sizes, but using 20-amp as an example, go from 24, 25, 28, as high as 29 ampere hours. And there is some time correlation with this happening, or with this event.

Now we're going to be talking about improving energy density this afternoon, and I contend there are ways -- and things are going to be discussed today -- to improve the energy density of Ni-cad. But I also contend you don't improve energy density of Ni-cad by stuffing more of the same stuff into the same size package. And that's what I'd like to talk about this morning.

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I talked about, yesterday, some test experience we've had in the 24-hour orbit. Well, that test, even though it was on 6-ampere-hour cells, has a very relevant aspect to one of Goddard's missions.

We have coming up a launch in 1976 of one satellite called the International Ultraviolet Explorer. It will be placed in synchronous altitude but it will not be over the equator, it will be over the northern hemisphere. And, as such, it will experience an average of a 60-minute eclipse period every day. We have a three-year design life, a five-year goal.

Now based on the data I presented yesterday, I think the technology is here to give us this.

I also think that we have certain pieces of information, certain things that we can do to further enhance that three-life and possibly achieve the five-year design goal. And this is related to the way the cell is built: more specifically, to the amount of actual capacity that is put into the cell.

And pursuing, trying to achieve the five-year design goal of this mission, we looked at the things we have learned about cell failures related to the synchronous type orbit. And I will refer to this as a synchronous orbit because it's a unique synchronous orbit in many respects.

We have seen indicators, as I mentioned yesterday, that the positive plates are really deteriorating, and they deteriorate under conditions -- well, the deterioration is somewhat of a function of the condition of how they're used. And the speaker who follows me will talk about that to some extent.

But in looking at the information we had, we said "What can we do to enhance the life of the cell? What can we do to try to achieve the design life of five years?"

Well, we figured that-- and I don't think anyone disagrees with this philosophy: the more electrolyte you can put in the cell the better it's going to last. And I think
in the past couple of workshops we've seen where there has been data to correlate dryness in a separator with cadmium migration.

This particularly came out in our polypropylene work. As most people are aware, polypropylene will not contain the same amount of electrolyte as a nylon, and consequently cells seem to run dryer.

(Slide 125.)

We started off with the design goal to achieve a reduction in positive plate loading of 90 percent of nominally specified. We also chose to go with the teflonation of negative plates. And I've just told you who makes the cell, because it's a General Electric 12-ampere hour cell nameplate capacity. The reason for the teflonation, other than the fact that we know from experience with the teflonation process with the nylon separator we can get more electrolyte in a cell than those cells made without the teflonation of the negative electrode.

As far as the teflonation, as related to cadmium migration that we see so pronounced in near earth orbit, I really cannot say that the teflonation is going to help us in a synchronous orbit other than the fact it did provide for additional electrolyte capability that I don't think we would have.

But as a result of the two top items we ended up with cells that were able to get approximately a 40 percent increase in the amount of electrolyte that had nominally been used in a 12-ampere hour nameplate capacity.

Now I point out we did not change the loading on the negatives. This was only directed at positives. However it's my conclusion that once you can reduce the loading on the negative you can reduce the loading on the positive.

I think we also have to look deeply at the negative, because if there's one thing that's clear in my mind it's that the utilization that you get, both initially and long term, is going to be directly related to how the plates are
loaded; not necessarily how as much as the amount of loading that's put in.

So what I am prepared to show you this morning is more or less a summary of the results that we have gotten both from manufacturing tests -- and I'll hit the highlights of those because I don't want to go into all the details -- also the acceptance test or the initial evaluation tests that the cells went through at Crane.

These cells, three types of them, are now on test at Crane, life test in the 24-hour orbit regime. We also have a pack in-house under test looking at a mission profile as the IUE will provide the batteries during the eclipse. And we've noticed a few interesting things.

(Slide 126.)

Let's start and look on a comparative basis. And I use the OSO-I simply because we have quite a bit of test data and experience on OSO-I cells. I believe Bill Webster presented electrical test data on that yesterday. And later on this morning you'll see some chemical analysis on the OSO-I cell. But I used it because it's a standard 12-ampere hour using nominal parameters, as you would expect if you went in and bought a nameplate 12 from General Electric.

What is interesting, and what I find significant in comparing these -- and I used three points: what I call -- what the manufacturer refers to as ECT, sometimes called formation, but this is the flooded plate test. This is on plates prior to packing them into the cell. I chose a point called mid-acceptance test simply as a comparison. Then I chose the final acceptance test by the manufacturer. And this is the last test just prior to shipment.

Now what's interesting is the trend you see on the conventional loaded cells, starting off with as high as 16.5 ampere hours as the average, or the mean. And I show the number of units here, because we're not comparing an equal number of units, so the standard deviation may reflect that. But what's interesting is the delta decrease and the
mean capacity of the group of cells from the start of the first electrical test, which is a flooded, to the finished final cell test.

Here you have approximately 1.5 ampere hours lost. Here you have—You start with about 14.8 to 15, in that range, and you end up with about 1.5. There’s approximately a half an ampere hour lost in that process.

(Slide 127.)

The next Vugraph is taken out of the test report that should be issued shortly from Crane, showing the three sets of conditions: The 23°C. capacity C/10 for 24 hours, showing what the cells were -- and it's interesting to note we have cells now as high as 15 ampere hours; the average is running about 14.8 for the twenty cells, also the 0°C. capacity test, which is C/20 for sixty hours. And I did not show this in comparison with IUE because I don't have a one-to-one comparison on that. But the main reason for showing you this particular slide and the performance indicated is to point out the 35°C. capacity data.

Now this is the second time in a group of cells that we have seen where a large increase in the electrolyte shows a significant increase in the performance of the cell at elevated temperature.

I think if you look back through the proceedings of last year's workshop, or the previous year, I mention a program where we had undertaken on a Gulton 20-ampere hour cell to evaluate increasing the electrolyte quantity in cells. And the numbers were like we used, standard nominal specified as a control. We had a 20 percent increase and a 40 percent increase. And under similar test conditions at Crane we noticed that at the 35°C. capacity test, the cells with the more electrolyte showed increasingly better performance, particularly those with 40 percent, almost the same as these, having equal capacity under 35°C. as they did at 20°C.

Now I still have a nameplate rating. We have reduced the positive loading to 90 percent of the specified value.
SPECIAL CELL DESIGN PARAMETERS
IUE PROTOTYPE CELLS
(GE 12 Ah)

- POSITIVE PLATE LOADING 90% OF NOMINAL SPECIFIED
- TEFLOMATIATION OF NEGATIVE PLATES
- KOH AMOUNT INCREASED APPROXIMATELY 40% OVER NOMINAL VALUE SPECIFIED

FIGURE 125

EFFECTS OF POSITIVE PLATE LOADING ON CELL AMPERE-HOUR PERFORMANCE
(12 AMPERE-HOUR RATED)

<table>
<thead>
<tr>
<th>MANUFACTURER'S TEST</th>
<th>STANDARD LOADING (OSO-I)</th>
<th>10 PERCENT REDUCTION IN LOADING (IUE)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MEAN Ah</td>
<td>STD. DEV.</td>
</tr>
<tr>
<td>ECT CAPACITY (FLOODED)</td>
<td>16.52</td>
<td>3.67</td>
</tr>
<tr>
<td>MID-ACCEPTANCE TEST CAPACITY</td>
<td>15.90</td>
<td>4.64</td>
</tr>
<tr>
<td>FINAL ACCEPTANCE TEST CAPACITY</td>
<td>15.05</td>
<td>4.51</td>
</tr>
</tbody>
</table>

FIGURE 126

IUE PROTOTYPE CELLS
AMPERE-HOUR CAPACITY SUMMARY
INITIAL EVALUATION TEST
NAD-CRANE

<table>
<thead>
<tr>
<th>TEST CONDITION</th>
<th>AMPERE-HOURS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>MAXIMUM</td>
</tr>
<tr>
<td>20°C CAPACITY C/10 FOR 24 HRS.</td>
<td>15.3</td>
</tr>
<tr>
<td>0°C CAPACITY C/20 FOR 60 HRS.</td>
<td>14.4</td>
</tr>
<tr>
<td>35°C CAPACITY (26.7)</td>
<td>15.4</td>
</tr>
</tbody>
</table>

FIGURE 127
But I'm still getting 14 or 15 ampere hours out of the cell. Now I have the cell on cycle right now, and I don't have any life test data on it at this point to speak of. I will have that, and hope to have it reported to you at the next workshop.

The other set of conditions that's most interesting -- and again I point this out -- is the voltage and pressure characteristics.

Now to run a cell wetter than what our past experience has indicated, there's no question in my mind that we've got to rethink our present criteria for pressure requirements and just what good cells are. I've heard people say "I have cells operating with low pressure. Gee, they look good."

I'm not convinced that low pressure cells are all that good. They can be, if you know why they are running low. But on the other hand I know the Goddard spec, it has a 50 psig maximum pressure. To get wetter cells we're going to have to go to 70 to 85 psig.

The other thing you're not going to be able to do is to take the present technology, meaning the wetter cells, and operate them the old way with a lot of overcharge. I think the evidence is mounting that you don't need the overcharge. And where cells are running high on electrolyte or where you have put a significant amount of electrolyte above that which you experienced in the past, you've got to rethink your philosophy about how you charge the cells.

I was talking to a couple of people yesterday about this. And one of the things that is coming more and more to mind is that eventually we may be flying cells that are very close to flooding. In order to do that you've got to have a way to control overcharge. And we've got to be more precise than what we are now doing. And I think pressure transducers may be coming back into style; not on every cell, but using selected cells and going with a pressure transducer type control. And I say this simply because in the experience we've had with these cells on test, with
that 40 percent increase in electrolyte these cells are very near flooded. And I'll give you an example of how we know that; simply because a number of the cells have third electrodes located down one of the narrow faces. At 0°C. during the overcharge test at Goddard -- well, first at Crane, we noticed some unusual performances on those cells, indicating that we were getting saturation of the third electrode from oxygen pressure, or we were getting flooding.

Well, we did an experiment here where we had the cells fully charged at C/20 at zero, and after about forty hours we just reached down and laid them over on their sides. The results were phenomenal. The pressure, which had stabilized, took off again. It took about a period of about six or eight hours before the pressure restabilized very close to where it was initially.

When we first saw that, it was very difficult to explain. But when you think about what's happening to a near flooded cell -- and I don't think it is flooded; I think if you tried to pour electrolyte out you might get a few drops. But I think what really is happening is in a gravitational field with this type of cell you're getting the effects of the gravity pulling the electrolyte toward the bottom and settling. As soon as you rotate it in the gravitational field you wet the surface of the negative electrodes and immediately affect the recombination rate.

I think this is what contributed to the pressure increase. The interesting thing is it increased for a period of three or four hours and then it gradually came back down and stabilized very close to what it was prior to disturbing the equilibrium of the pack in the gravity field.

The other thing I find interesting about the cells is the voltage characteristic, the overcharge voltage at zero. This is not the peak, incidentally, this is the end of charge for sixty hours. But the peak is still below 150. And it kind of puts a question, in my mind at least, and in validates some of my previous thinking, that if you have a lot of electrolyte in the cells they're going to run higher in overcharge voltage, particularly at low temperature.
I can tell you this, that we are presently cycling this five-cell pack from the group of cells that this data represents at 0°C. at 140 -- I'm sorry; at 148 volts per cell. Again, I would never have thought I could recharge a cell at 0°C for that condition, particularly when you're charging at the lower rates using C/10 and C/20. But indications are we can go as low as 1.47 and still get an adequate C-to-D ratio in a 24-hour orbit period.

In summary, while I am not standing before you and saying I have all the answers on loading, I'm saying we have demonstrated that we can cut back to about 90 percent on the positive. We have yet -- I have yet to see a sacrifice that I've to take in cell capacity.

The question I leave you with is, How much further can I cut back and still maintain probably 2 or 3 percent above nameplate capacity?

I think the long term effect of this will prove out that the cells are going to operate better, they're going to show less deterioration under the similar regime that we have tested. And I think that part of the problem associated with the positive electrode, which may be attributed to electrolyte redistribution, will further be minimized with these wet cells.

Thank you.

Are there questions?

GROSS: Floyd, did you note any difference in polarization on charge and discharge of these cells?

FORD: Under a specific regime, or just in general?

GROSS: I presume you've been testing them; is that right?

FORD: Yes.

GROSS: How does the charge and discharge voltage compare with the conventional 12's?
FORD: The charge voltages are a little bit lower. I haven't looked at the details of the discharge voltage to that degree. I've looked at the charge voltages on these 100 percent cycle tests, and also I've looked at the charge voltages during cycling on a 24-hour orbit. And, as I indicated, the fact that we're down -- that we're cycling at the 1.48 level and still getting adequate C-to-D ratio, why, I'm sure, based on the data, I think we could go to 1.47 volts per cell and still not be undercharging the cell at 0°C.

I don't know how much lower I can go. But we're going to be exploring that.

So the answer to your question is, Yes, particularly on charge I think they have a little lower charge voltage than what we've experienced, comparing it with a so-called "standard" cell made by the normal process.

KRAUSE: Floyd, we've been running some cells lately that are not especially optimized for electrolyte level. But they do have extremely low carbonates on purpose. We have the manufacturer go through a process to reduce the carbonates. And the voltage levels are very similar to what you're getting. So I'm wondering how much you can attribute this lower charge voltage to the additional electrolyte; or perhaps you also have low carbonates in the cell.

FORD: Are you asking me did they go through a decarbonation step in these cells?

KRAUSE: Yes.

FORD: I don't believe they did.

The answer has just been verified, that they did not. There were no special manufacturing procedures implemented to keep the carbonate level below nominal. -- you know, what you would expect in other cells.

KRAUSE: As I say, apparently just by getting very low carbonates we can also reduce the charge voltages and pressures.
FORD: Yes. And I think if you look at the fact of the significant increase in electrolyte amount, that if you had the same amount of carbonate you had in previous cells you still would have more electrolyte available for the reaction.

KRAUSE: Yes, I agree.

FORD: Okay.

So the end effect may be about the same.

McHENRY: About six years ago I guess, we had the first battery workshop. And when I was here it seemed they were having trouble with their building pressure in their cells. And what they were doing was using 20-ampere hours cells for a 3-ampere hour job. And in order to get into the package the weight and the size and all that sort of stuff, to get 20 ampere hours in it, you have to stuff as much as you can into the electrode. You have to cut your negative-to-positive ratio way down. And you make a cell which can't be overcharged. It builds up pressure and the thing fails.

You only wanted three ampere hours. Why we couldn't have made a 10-ampere hour cell in the same size—just don't load the plates so heavily. A heavily loaded negative will flood quicker. The pores are smaller. Then if you have troubles with your nylon separator — of course you don't use that any more anyway, but it will degrade and uses up some of your oxygen. This unbalances the state of charge. You find that your positive now is — the negative is used up on charge before the positive. If you had only made a negative that was twice as big as the positive in capacity you could make errors of having a little degradation, your separator losing 15 or 20 percent of the negative, and you'd still be all right.

But what we've tried to do is stuff energy density into the can, and at the expense of life. Well life is what you need, not energy density.

If you're going to use a 20-ampere hour cell you could have had a 10-ampere hour cell. You only wanted three. You only use these things down to 15 or 20 percent
of their capacity. Why not 60 percent of the capacity and make something that works?

The basic philosophy of the battery company -- and this is because of the market, which demands high energy density -- we are not a market, we don't spend that much money. The toy manufacturers and the electric shaver people are the ones who make the money on it. You can't expect the battery company to go into business to lose money just for us. Their basic philosophy is to stuff as much in as they can. And that's what they can sell.

But we must say, "Look, we don't want it. What we want is a long life cell. We are willing to take less energy density. Just don't impregnate it fifteen times," or whatever it is. I guess eight times, or something. Six or five times. We will now have a 12-ampere hour instead of a 20, and it will last.

We just keep doing the same dumb thing, insisting on that very high energy density. Tell them you don't want it. "What I want is life." And then see what he can do.

I know we could do an awful lot better with these lower capacities than we can with high capacities. If you just look at the electrode you can see little crystals, you can see pores and everything else. And you look at the scanning electron microscope pictures and there's a world of difference between them.

You must give up this energy density if you're going to get reliability. You don't have to give up an awful lot but you've got to give up some. You can't play on the borderline.

That's it.

FORD: Thank you.

I mentioned earlier that I did not find fault with the manufacturers. I would like to say the only fault I find with them in this area is simply that they yielded to the pressure of the user to do this.
VI MATERIALS AND CELL COMPONENTS: CHAIRMAN G. HALPERT

HALPERT: We'll go into our next session now on Materials and Cell Components. And I might mention that we were very fortunate here at Goddard this past summer to have had two faculty fellows from local universities working with us on two distinct problems, both of whom you will hear from this morning.

The first you'll hear from right now, Dr. Pat McDermott of Coppin State College in Baltimore. He was a Morgan State College fellow here this summer.

VI.1 POSITIVE PLATES EVALUATION AFTER HIGH DOD AND LOW TEMPERATURE: P. McDERMOTT/COPPIN STATE

McDERMOTT: Okay. We're looking at a series of synchronous orbit tests conducted on 6-amp hour G.E. cells at Crane. And it was demonstrated that the principal failure mechanism of the cell subjected to low temperature at high rates of discharge seemed to be the positive plates. The physical inspection of the failed plates showed a deterioration of the sintered nickel, the active material.

(Slide 128.)

There's a loosening, a crumbling of the active material, an actual flaking and peeling away of the substrate. And although all of the test cells seemed to show some form of deterioration, the most extreme cases seemed to be the ones at 0°C., 60 and 80 percent depth of discharge.

Most of the deterioration occurred around the edges, and particularly along the top edge near the tab, and along the uncoined edges.

(Slide 129.)

We looked at cells under different test regimes. I might show six of these cells.
Packs 1 to 4 had a percent depth of discharge of 40 percent. The temperature ranged from 40 down to -20. Two cells that we were particularly interested, at the bottom of 5 and 6, were 0°C. and 60 and 80 percent depth of discharge. The charging rate was C/30 constant current except for the low temperature there, a slightly higher charge rate, and then switched down to C/30 by the coulometer.

(Slide 130.)

The capacities of the cell--This is discharge time to .5 volts.--showed that the Synch 1 and Synch 6 there at the bottom—Synch 1 is the 40°, 40 percent depth of discharge. It shows very low capacity.

Synch 6—now this is the pack where a cell actually failed there at the eleventh eclipse season. This was the 0° 80 percent depth of discharge.

Synch 5—Well, Synch 4 was -20°, 40 percent depth of discharge. This did not perform very well.

And Synch 5, 0°, 60 percent, did fairly well.

The two at the top, Synch 2 and Synch 3, were 40 percent depth of discharge. Synch 2 was 25°, and Synch 3 was 0°.

So if we would generalize somewhat from these results we'd see that -20° and 40 seemed to perform poorly, and that in looking at the cells at 0°, this is Synch 3 and Synch 5 and Synch 6, they seemed to perform better at the lower depths of discharge; that is, 40 percent performs the best, and then 50 and then 80; which would stand to reason.

(Slide 131.)

Then we looked at some 12-ampere-hour cells. These were also under synchronous orbit, a 140-day daylight period with 42 days of eclipse, running from the 1st to the 18th day for 12 minutes discharge up to 72 minutes, and a capacity check in the middle of the dark period.
There was a plateau for about eight days where there was a 72-minute discharge, and then a capacity check was run in the middle of that.

The 12-ampere hour cells were also under a synchronous orbit regime. We found that the -- we looked at two cells where the fill tubes had broken. These cells were not ready to be taken off yet. The two cells did fail mechanically: there was no internal failure but just a mechanical breakage of the fill tube. So they were taken off.

And we looked at these this summer also.

This 12-ampere hour cell at 20°, 80 percent depth of discharge, showed some deterioration of the positive plates, not quite as bad as the 80 percent depth of the 6-ampere hour cell.

(Slide 132.)

You can see there's quite a bit of crumbling and deterioration here, whereas on the 12-ampere-hour, 80 percent, not so much.

(Slide 133.)

We also looked at a 0°C. 60 percent depth of discharge 12-amp cell. And the plates looked quite good relative to other plates that we had seen.

So trying to assess what was going on here I tried to look for some explanation of the differences between either the charge/discharge regimes or the various parameters of the cell. And it looked as if there was one primary difference between the two tests, the 6-ampere hour and the 12-ampere hour. And one was the charging rate. I mentioned previously that the 6-ampere hour cells were charged at C/30 constant current whereas the 12's were charged at a higher rate during the early stages of charging. But then they were stepped down to C/60 trickle charge when they started reaching full charge.

I also noted a difference between the 60 percent depth
FIGURE 132

FIGURE 133

These are cells which failed due to full-time break then were removed from the pack.
plate, which looked pretty good -- and this is in the 12-ampere hour cell -- and the 80 percent, and found there again that the charge rate for the 60 percent depth was much lower than the 80 percent depth. So it seems that the charging mode, and particularly the overcharge, could be a factor in the deterioration of the positive plate.

In looking through the literature to find if there were some theoretical explanation for this I found references to the fact that overcharge tends to shorten cell life considerably, but not too much information on why this was. So I'd like to hazard at least a couple of guesses as to why this might be happening.

I offer this to stimulate rebuttal, so that I might be able to find what the real mechanism is and finish this report.

No. 1 on my list was the possible culprits would be gassing. This would be related directly to the rate of charge and the overcharge. And just examining some of these plates and looking at the blistering and the pimples that occur along the coining, and so forth, it looks as if -- just physical inspection -- it looks as if the gas in the active material is actually distorting the substrate. Theoretically the sintered nickel, the porosity should be sufficient to allow the gas to escape. But it's possible that due to recrystalization of the active material, or for some reason, that this gas might be trapped and that there might be difficulty in getting the gas through the plate.

I was talking to Dr. Gross last night. He mentioned that he had done some theoretical studies which indicate that the problem would be accentuated particularly as the gassing took off; that is, when you have a bend in the gassing curve, that the highest stress would occur at that point.

Other possible mechanisms which might be responsible for the rupture of the cross linkages or the tearing of the sinter away from the plate, might be the expansion and
contraction of the active material as it undergoes charge and discharge. Now this would be exacerbated by having depth of discharge where the active material would be going all the way down to discharge, and also by low temperature in the sense that the high acceptance --- probably the highest acceptance of charge for these cells would be around 0° Centigrade so you would have this big swing of active material from charge to discharge at 0°.

Thermal expansion and contraction of the nickel sintered active material could possibly account for it.

Migration of electrolyte in and out of the positive electrode which would cause swelling and possible deterioration or the corrosion of the nickel sinter itself. All of these are just possible means. I'm looking for other possible mechanisms to explain this.

In order to test some of these hypotheses out, we put two cells on test during the summer. These are six ampere-hour G.R. cells. They are both at 0° and 50 percent depth of discharge.

The only difference between the two is the charging mechanism. One is on constant current charge at C/30 and the other is constant current with a pressure trip, so when the pressure rises five pounds per square inch it will trip down to C/60. So we'll be looking at these cells for a couple of months and then tear the cells apart and look at the positive plate deterioration.

I also constructed another test cell which is now in test. We haven't really analyzed the data yet, but it is a cell constructed by attaching an external lead to every positive and negative plate, and we can continually monitor the voltage across each positive-negative plate system or couple, and also measure impedance and so forth.

And the idea is to try to look at the inner-dynamics of the cell during charge and discharge, look at plate capacity insofar as this is possible, and then tear the cell down at the end and see if the plate capacity of the cell operating as -- as an operating cell looks any different from the plate capacities of the plates outside because generally we look at plate capacity after the cell is torn
We've gotten some interesting results shown in some plates falling off, the capacity falling off quite rapidly during discharge. And it would be interesting to see what happens when we tear it apart.

In summary I would say the examination of the 12- and the 6-ampere-hour cells showed severe deterioration of the positive plates, particularly those cycled at 80 percent depth, and I think there might be several ways of attacking this problem.

One would be to look at the charging mode and try to limit overcharging as much as possible, maybe consider lowering the depth of discharge in the operating system, and looking also at other factors which Floyd mentioned and which we'll be looking at; that is, plate loading, amount of electrolyte, and the contamination, the carbonate contamination.

So that's pretty much a summary of the work we did this summer.

HALPERT: Are there any questions for Pat?

SCOTT: Is it not true that the plate that you showed us with the blistering was from only one of the cells on that 90 percent depth of discharge 0° test at Crane?

MC DETMOTT: Yes.

SCOTT: Is it also not true that there was only one or two plates in that cell that showed that condition?

MC DETMOTT: Let's see. We had photographs of--

Floyd, how many plates were actually in those?

FORD: In the cells?

MC DETMOTT: No, in the photographs, when they physically torn the cells down at Crane and looked at the plates?

FORD: I don't know how many photographs we had within
each cell, but we had photographs of various cells, plates out of cells of each test group.

MC DERMOTT: And they all showed some deterioration.

FORD: Yes, there was some deterioration.

SCOTT: Well, have not only two cells of the original five cells on that 0° 30 percent depth of discharge test been torn down to date? Or is there more--

FORD: That's the 12 ampere-hour cells. Of the 12 ampere-hour cells there have been only two cells pulled and those cells failed for mechanical reasons, not what we attribute to a normal failure mode.

Now if your question is whether that was indicative of one or two plates out of a cell, the answer is I don't know. I would have to go back and check. I don't think they would isolate a plate and say this is typical.

SCOTT: Well, my impression is that that occurrence that was shown in that photograph is a random condition and not typical of plates operating under that mode and therefore, I question whether that occurrence should be the cause for zeroing in on that particular kind of a condition.

MC DERMOTT: You're talking about the 6's?

SCOTT: Right. That's where that particular blistered plate came from, was it not?

MC DERMOTT: The 12 which I tore apart. All the positive plates showed some deterioration, some more than others, but that was the second slide of the plates that I showed.

FORD: But not to the extent that you showed it with the 6. The 6 were shown-- It was the worst case that was photographed out of the group of 6's, but there were six cells opened, one out of each test condition that he talked about, and the degree of positive plate deterioration varied from whichever cell or whichever test group the cell came from.
COHN: Cohn, NASA.

I think you're on the right track when you're saying that there are probably a number of reasons as to why you have observed what you have observed. I think it will be very dangerous to conclude that the deeper the depth of discharge, the worse the wear on the plates is, which is I think what Willard Scott was trying to tell you, too.

Things are not simple; things are very complicated, and as you change the structure and as you change the operating of your cells and of your batteries you will undoubtedly find that they behave differently.

There has been a lot said about that already. It is not very simple.

Off the record.

(Discussion off the record.)

On the record.

'V C DIONISOT: You're saying that depth of discharge really has little to do with plate deterioration.

COHN: That I'm saying is this: If you will look in the 1963 NASA Headquarters Workshop on Batteries you will find one of the men questioning the relationship between life and depth of discharge. There was no answer then and there is no answer now.

The indications are that life does not suffer from depth of discharge if the battery or the cell is built right and if it is used right.

For example, in charging, it has been well established that if you charge at about the two c. to four c. rate up to the point at which you get the first indication of pressure evolution, you get a much more efficient charge even though it's a faster charge, which means you don't have the temperature problem. It's an endothermic process so you don't get any rise in temperature, you don't get any gasping. And if you manage to do your charging properly, if you cut down on your plate loading, if you increase on
on the amount of electrolyte, if you do a number of other things right that we talked about already, it may turn out that you are actually better off. In fact, I'm convinced that you will be better off.

If you look at the record of Skylab where the problem was insufficient depth of discharge rather than too deep depth of discharge because of various reasons, you will undoubtedly find that you're better off with a deeper discharge by properly managing the battery.

MC BERGOTT: That's what I was suggesting by the charging mechanism, that the passing I felt was probably the predominant feature of this deterioration.

FORD: A point just came to mind, and I would like to tap somebody's memory that was on the Power Sources Symposium this spring.

Didn't the gentleman from Wright-Patterson give a report or give a paper on the effects of current density on the cell degradation, looking at the-

Dave, was this Gerry Miller? Yes, gave a paper showing that there was a direct relationship between the current density and depth of discharge, that they go hand-in-hand, and the effects of degradation on cell performance.

PICKETT: Miller did give a paper, as you suggest, with that information in it, and I don't know the details of it so I can't comment any further, but there was one given.

COHN: I don't doubt for a minute that he had such data, and that the data were proper. What I would like to ask is whose cells, how were they built, and how were they operated, in detail; not only depth of discharge, not only rate of discharge, but also amount of recharge, rate of recharge, amount of overcharge, the various controls on this for temperature, pressure and so on.

There is no simple answer, and there is no simple datum that will give us a solution to our problems.

What I'm saying is I'm sure that when you do those right you have to know all the circumstances, including what
what the manufacturer did, and what the people in the lab did, in order to appreciate what really happens.

MC DERMOTT: Could I comment briefly? I still think you're saying that going from 60 to 80 does exacerbate problems in other areas. In other words you're saying control the cell perfectly and you won't have any problems, but if you do have variance in temperature and so forth, that the depth of discharge could exacerbate that problem.

HAINES: I wish Dr. Cohn would change his mind about off-the-record remarks. I do hope you're not suggesting that we burp these things.

On other comment before I come to my question:

It sounds as though we're having a session back home with Dr. McHenry's remarks about excess negative, increasing the amount of excess negative, which was exactly what Floyd Ford was doing when he cut back on his positive material. But I want to ask you a question:

Have you done any of this evaluation on the vented ni-cad battery, particularly at low rate pretested discharge and then high recharge characteristics? This is a field where the aircraft industry is vitally interested, and if you're getting this sort of degradation with the low charge rates and what I would call very high load temperatures, we're in real trouble in the aircraft industry.

MC DERMOTT: I've done no work on vented cells.

HAINES: I would like to know whether this is something that is peculiar to the scaled cell in starved electrolyte condition or whether it is something which is prevalent in all varieties of ni-cad batteries.

SCOTT: Scott, TRU.

In my experience, the problem is worse in flooded conditions than it is in starved conditions so that may be some answer to the previous question.
We saw that kind of comparison in the study we made of coining versus no coining last year, in which we looked at various ways of accelerating this kind of deterioration of the positive plates and we saw the worst, the most rapid deterioration under flooded conditions.

I would like to make another comment or raise another question.

I still believe that the specific form of blistering that you're seeing in the cells, particularly the one that you showed the photograph of, is not generic to this particular operating mode. I believe that is more a function of a particular problem possibly in plate manufacturing. That should not be considered as inherent in operating at deep depth of discharge at low temperatures.

I say that for various reasons, one of which is that we have conducted tests in which we have operated the cells at a constant 80 percent depth of discharge for every cycle, one cycle a day, and we have run them up to 600 cycles in this way. And under those conditions, with about C/10 charge rate with some control of overcharge, we saw no blistering whatsoever of the plates. And these were G.E. plates made by at least a similar process compared to the ones that were in those tests.

So we did see a weakening of the positive plates. We did see some crumbling of the edges, but no blistering whatsoever. So I think that what you showed there was probably in that cell at day one, because sometimes you can open new cells and see blistered plates.

MC HENRY: Too bad Dean Maurer is not here because he has much more of this data in his head, but we had run electrochemically impregnated cobalt-containing electrodes with various amounts of cobalt in them. We ran them at the 10 C rate, not C/10. They got something like 2,000 to 3,000 cycles at that rate, a hundred percent discharge with a hundred percent overcharge.

You discharge them down to half a volt and then you run them a hundred percent overcharge, and then you discharge them down. The whole cycle takes something like 1 2 to 15 minutes and they just go on and on and on, and they don't
blister.

And I've seen other electrodes blister but I think one of the problems is some of the impregnation methods corrode part of the sinter and make it weaker. And then if you look at the photographs you'll see there aren't any pores in them. I don't know how the gas would get out.

An electrochemically impregnated electrode looks essentially like a covered-over piece of sinter. It looks like the sinter except it has crystals all over it. You can actually see down into the pores, but some of the other electrodes I've seen, vacuum-impregnated, the material is not spread over the sinter. It's just plastered into the pores and it looks like what someone did, just plaster something in there, and it is no wonder they fall apart. And they do.

And I think it is much worse in flooded condition. There is nothing to support them. I've had them at C/10 fall apart on me. I was cycling some just to characterize the thing and they all fell apart.

So depending on how you make the things, you can get all kinds of troubles. And I think overcharge is bad for a bad electrode but for a good electrode you can just worry go ahead.

THIERFELDER: Thierfelder, General Electric.

There were some cells on test for more than ten years out at Crane and I was wondering if any of those had been opened up and looked at, or if they are planned to be.

FORD: Yes. I believe there were some molten cells.

THIERFELDER: C-512's.

FORD: We did open one of the cells and I have photographs of it. I was not prepared to present them here.

THIERFELDER: They were near-earth orbit vs. synchronous orbit.
FORD: Yes, and the failure was attributed to the separator eaten up with cadmium. When you pulled it apart the separator just stuck right onto the negative.

The positive plates looked relatively good. There was some indication of deterioration, but nothing like we're talking about here.

NAPOLI: Ford, in his earlier presentation, indicated that you can get 14 ampere-hours out of a 12-ampere-hour cell, and we have also been talking about depths of discharge. Are we all talking about a common basis for calculating the depth of discharge, that is, rated or actual? Are we all working the same goal here, or starting from the same square one?

FORD: Well, depth of discharge as we normally refer to it is based on nameplate capacity. Now the answer to the question is no, we don't all work from that base.

NAPOLI: The gentleman who indicated like 30 percent depth of discharge, is he going on nameplate?

"C DURKOTT: That's nameplate, yes.

NAPOLI: So you could have had the 12 ampere-hour cells; you could have had indeed 14 ampere-hours, but you don't usually use that actual for determining depth of discharge. Is that true?

(Slide 131.)

FORD: That's the similar data that was shown on the 6, showing the performance of the 12 ampere-hour cells, and I don't know that this is self-explanatory to everyone, but Pat explained what happened on the eclipse. But what we're looking for is a line here right at 72 minutes. Will the cells support that rate, or that depth of discharge at the 72-minute period?

The interesting thing when you talk about capacity--When you talk about basing depth of discharge on actual capacity, I have one question for you. What's the actual capacity of this pack right here? (Indicating.) What do
I use as a baseline? After two years in orbit, do I say I have a 19 ampere-hour cell, because that's what it is. Are we going to change our groundrules as we fly the spacecraft? I don't think so.

The other question is what causes that phenomenon. If we assume that our capacity is determined by the positive plates, then the positive plates must be changing, growing, doing something.

CROSS: The most significant thing to me in the comparison of those two charts is that the six ampere-hour cells which were tested to the same regime as the 12's, the capacity fell with time whereas with the 12's it stayed the same, ignoring the high temperature and the low temperature tests.

I would think we would need an explanation for this.

FORD: There is no question about it, the 12's are performing much better than the sixes. As far as an explanation, the sixes are late '66, early '67 vintage cells. The 12's are '69 cells. There is two to three years difference in the manufacturing period.

The data that have come out later, the 12's were testing out not to 12's you're flying today; at least if you're flying a C/7.17 the plate dimensions on those are entirely different from the plate dimensions you're getting today, so there are many factors that you have to consider.

But also the charge regimes on the 12's--As Pat indicated, these 12's are charged back in the range of C/10. Initially some of them are as high as C/5 until we get the third electrode signal and then we trip to C/50 and we've found that C/50 was adequate in all cases except above 70 degrees C. And we considered that really not a trickle charge but more a sustaining charge than anything else.

But we are not holding these cells on constant current overcharge day in and day out when they are not going through the eclipse period. That's the only explanation I can offer.
VI.2 PRELIMINARY REPORT ON CHEMICAL DEGRADATION OF NYLON:
J.D. MARGEZUJ; "S. LIN/HUGHES

HALPERT: Our next speaker on the subject of Materials and Cell Components will be J. David Margerum from Hughes.

MARGERUM: I want to thank NASA for inviting us to share these very preliminary results with you in an informal fashion. The people in our Space Communications group at Hughes are very interested in the problem of having long-time satellites, ones that last for seven years or longer, and they didn't tell it to us in those terms but I guess Sciesz who was saying it was maybe worth a million dollars or an extra year of battery sort of makes the point that if you can make a battery last longer, if that's the limiting component, then it is certainly worth a lot of money in a communications satellite.

They noticed that the research labs were doing things in liquid crystals and polymers and things, but weren't doing anything in battery studies, and so they invited us to get our feet wet in this area, and after a few months of getting our feet wet, I discovered it is like stepping into a bath of KOH. But anyway, that's the present situation.

What we would like to do is look at a long-term study of some of the fundamental limitations or possible limitations in the lifetime of batteries and in looking at it from what we could do, especially from the chemical standpoint first, we decided that we would like to begin studies on the limitations of the nylon separator as one possible area of study, so that's the point at which we have begun.

(Slide 134.)

This is somewhat in a position like the old jokester who says "Don't stop me if you've heard this one before," because you people have been talking about this thing for a long time already, and you've had many advocates of the problems with the nylon separators as being possible life-
time limitations. You've had discussions by Dunlop and by Mauer and other people in your workshops and elsewhere, pointing out these problems. But let me put this on here to begin with.

The nylon, if it hydrolyzes— This has been one proposed mechanism: that is, the nylon hydrolyzes first as shown on the slide and then reacts electrochemically to degrade in the cell. That is, the hydrolysis product reacts in the cell at the nickel electrode and is oxidized either— It reacts with oxygen at the nickel electrode or it reacts with the nickel electrode and is oxidized by the nickel electrode directly. Both of those have been suggested.

And then this leads to the formation of carbonate and other products, and then this is a possible limitation to the lifetime of the batteries.

I would like to point out, in reference to the talk that Scott gave yesterday, that if such a hydrolysis occurs, that you are consuming the OH ion, and so if you start out with a given concentration of carbonate, and if you really have an appreciable hydrolysis of the nylon over a long period of time, that you are increasing the ratio of carbonate to OH ion even though nothing else is happening, even if there is no other production of carbonate because you in fact are consuming OH ion as the process goes along.

If you also have the electrochemical formation of carbonate, you are further reducing the concentration of OH ion as well as increasing the carbonate formation. So that's another area that you might want to consider in regard to this mechanism.

Now the reason this has been suggested as a mechanism, as I understand it from what I've been able to glean out of reading your workshop proceedings and so on, is that if you do an accelerated test on a battery and take the complete battery and heat it at various temperatures, you will find the thing degrades and that there is a -- quote -- "activation energy" -- unquote -- for some reaction which is really not known because it is rather complex and has not been analyzed in detail in the battery, and that
activation energy has about 14, 15 kilocalories per mol figure.

What we would like to do is look at this sort of thing from the chemical standpoint and decide what is this mechanism, which is the rate producing step, both before electrochemical reaction and after electrochemical reaction, and what are the products, and what does the whole thing look like from the chemical standpoint?

Now I must confess that at this point we have just begun on this, but I thought I would like to share with you where we are at this point.

We are looking at Pellon-2595, a triple-washed sample as is used in the Hughes 24 amp-hour cells, and the question came up as to what is this? And we asked Pellon and they said it's nylon.

And we said 6, or 6X?

And they said that was proprietary.

So among other things we're doing is looking at an investigation of what we're starting with. Now we haven't completely analyzed it but let me point out a few things.

It is nylon 6 predominately. If you do a hydrolysis at high temperatures and then analyze with a gas chromatograph the product, you find nylon 6 is the primary monomer. We found less than .2 percent nylon 6X if there was any there at all.

We looked also at the monomer content of it by dissolving it in formic acid and looking at the gas chromatograph and we found less than .2 percent of free monomer that is Caprolac-10.

And we also heated it in water, boiling water, and after about seven hours there was about .4 percent weight reduction. We did not identify what that product is yet, but it is also an indication that there is not much free monomer in the sample to begin with.
We sent some samples out for atomic emission spectroscopy. We thought we might look for zinc or something like that. The analysis really wasn't a big enough sample apparently to detect zinc accurately, but they did show about 3,000 parts per million of titanium. I'm not quite sure what significance that has.

(Slide 135.)

Now there is an alternate mechanism that I would like to call to your attention. This is a mechanism that has been studied and reported in the Journal of Polymer Chemistry, studies on the heating of nylon in the presence of oxygen in water.

What this says is that oxidation may be the rate-determining step in this reaction. First we get oxidation at the carbon -- next to the nitrogen, and then that is followed by hydrolysis and you get adipic acid as well as adiponitrile acid. And then of course this also then would react with OH ion to use up OH ion. So this is an oxidating hydrolysis and it also uses up OH ion. And again, you would have products that could react at the electrode in the actual battery.

Now what we thought we would do initially is to try to evaluate which one of these mechanisms is going on. We just started this, but let me tell you the sort of thing we're doing.

We are starting to look outside of the battery, sort of like an idealized situation, and get some idea if you can look at the actual chemical reactions first, without looking at the electrode effects. So this is a setup diagram here.

We put the nylon in that excess of KOH, a large excess of KOH, and control the gas by bubbling either oxygen or nitrogen through the sample and heating at constant temperature and then look at what happens to the nylon. And then we intend to look at what happens to the products that were formed as we carry this reaction on. This is just our initial kind of studies in this area.

(Slide 136.)
PROBABLE MECHANISMS OF NYLON 6 DEGRADATION

HYDROLYSIS

\[
\begin{align*}
\text{H} & \quad N - \text{ICH}_2\text{C} - C \quad \rightarrow \quad n \cdot \text{H}_2\text{O} \\
= & \quad \text{NH}_2 - \text{ICH}_2\text{C} - \text{COOH} \\
= & \quad \text{NH}_2 - \text{ICH}_2\text{C} - \text{COO}^- \quad + \quad \text{H}_2\text{O}
\end{align*}
\]

FIGURE 134

OXIDATIVE HYDROLYSIS

\[
\begin{align*}
\text{H} & \quad N - \text{ICH}_2\text{C} - C \quad \rightarrow \quad n \cdot \text{O}_2 \\
= & \quad \text{H}_2\text{O} - \text{C} - \text{O} - \text{H}_2 \text{C} - \text{C} \quad \rightarrow \quad n \cdot \text{H}_2\text{O} \\
= & \quad \text{HCOO}^- - \text{ICH}_2\text{C} - \text{COOH} \quad + \quad \text{NH}_3 \\
= & \quad \text{HCOO}^- - \text{ICH}_2\text{C} - \text{COO}^- \quad + \quad 2\text{H}_2\text{O}
\end{align*}
\]

FIGURE 135

REACTION FLASK FOR NYLON REACTION

WATER CONDENSER

GAS INLET

H_2O

Ni PLATED STAINLESS STEEL FLASK

SAMPLE

34% KOH

Ni WIRE

CONSTANT TEMPERATURE BATH

FIGURE 136
To give you some idea of what happens to the nylon, if you heat this at 115 degrees Centigrade for various lengths of time in such reactor -- and this had a small amount of nitrogen bubbling through it -- what you find is after a few days it all falls apart, and there is no question but what it is degrading. And if there is a free sample of nylon that is not contained in any way, why you get nylon fibers all through the reaction vessel after about two or three days.

(Slide 137.)

These are pictures of the nylon as it was taken out and washed and dried and looked at, and it has obviously deteriorated.

Now one of the things that is going to happen as this reaction continues is that the nylon will become soluble partly as it degrades.

(Slide 138.)

Let me point out this, that as far as we know, the attack of OH ion on nylon is a random attack anywhere along the chain and there is no more probability that it attacks one end than the other, as far as I know, of the polymer chain, and there is no unzipping characteristic in the nylon so it's a random attack.

Therefore, with a fairly long chain length as you have in KOH and the reaction proceeds, what you expect to see is a decrease in the molecular weight of the sample, and this has been reported. It has been studied, at least by nylon samples in batteries.

I think a couple of years ago it was reported at a NASA meeting that the molecular weight distribution of the nylon did decrease to lower molecular weights as you go on.

So in the cell, in the conditions that we are studying, we expect to see that as it reacts, the rate at which it dissolves should increase because the chain links will be getting smaller and they will be reaching the point where you're more likely to knock off an end group or you are more apt to get a ligamer that is soluble.
WASHED AND DRIED NYLON SAMPLES AFTER REACTION IN 34% KOH AT 115°C

FIGURE 137

REACTION OF NYLON SEPARATOR IN 34% KOH AT 100°C

FIGURE 138
And so we expect to see an accelerated effect as the reaction continues, which is the curve we have here. (indicating.)

I do want to say that we have just really begun this work, that this is not at all conclusive. The error bar here between these two runs happens to be fairly substantial and it is only out at those points that we can really see if there is a difference between oxygen and nitrogen.

Preliminary results indicate that there is a difference and that the sample is decomposing more rapidly in the presence of oxygen, and we need to actually confirm that yet.

Then we have identified aminocaprolic acid as one of the products in the electrolyte, the KOH sample, and we will be looking in much more detail with both gas and liquid chromatography at the other reaction products that we find, and we will also be analyzing the nylon for its change in molecular weight and for other physical properties.

That is sort of a status report on where we are now.

Thank you very much.

PALMER: In the equipment you show, you can see oxygen bubbling through a sort of a separate piece of equipment, and then an atmosphere of oxygen. Is there a gaseous oxygen in contact with the sample, or is it simply a soluble oxygen situation?

HARGERUM: In this case this is a large excess of KOH and it is soluble oxygen that we're concerned with, not gaseous oxygen.

PALMER: My suggestion here would be that if there is an oxygen effect in real life you're likely to get a mix of liquid electrolyte and gaseous oxygen in a washing back and forth situation, and that this is going to be vastly more destructive because the solubility of oxygen in KOH or...
these temperatures is pretty low so you are not going to get a great deal of oxygen in that type of a situation, I would not think.

NARGERUH: You're quite correct. We understand that. We wanted to begin the study at some place where we more or less knew what the concentration was. At 100 degrees it is about $5 \times 10^{-5}$ molar. As you decrease -- in KOH.

As you decrease the temperature, actually the oxygen solubility increases and the degradation may not be temperature-dependent in the normal activation energy plot. It may be you have to take into account the rate of hydrolysis constant times $"$ ion concentration, and also the rate of hydrolysis which would include the concentration of oxygen.

So we intend to do that. We would like to then follow up with the sort of thing you're talking about, looking at it in the condition in which there is gaseous -- In a wetted nylon sample such as in a battery, we intend to follow it up this way.

The advantage in doing it this way first is at least we are not changing the $\Omega^+$ ion concentration as the reaction goes. If we get some sort of a decomposition rate here, we can understand from the chemical standpoint what is happening.

Now one thing I didn't point out. I feel all of this has already been done before, but we can't find it in the literature. And maybe by telling people we're starting this we'll smoke out some data and perhaps people will either tell me what it is or else they will perhaps publish it if they haven't already.

GASTOJ: I recall a workshop meeting several years ago where Bob Steinhauer did report the degradation of nylon in the presence of oxygen. It was done by a study for the Air Force, and I believe it was for the 15 amp-hour cell.

And I think one of the conclusions was that the nylon does hydrolyze at temperatures as low as 100 degrees F.
which we could not confirm in our study. So that work was done and it was done at Hughes.

MARGERUM: Bob was one of the guys who encouraged us to do this, and he knows in detail what we're doing. When we ask such questions as what is the rate-determining step, what are the products and so forth, as far as I know that has not been published.

GASTON: Now I have another comment. You mentioned possibly the presence of zinc in the Pellon separator. Yes, there is, and we did find it several years ago, that it is relatively high, and I think you see some good data, the OAO battery data analysis program which was published. And you will find -- I was amazed -- that it is relatively high, but of course that was done three or four years ago. It might have changed.

HAINES: I think what you're doing is pretty good work but I have a feeling you're flogging a dead horse. We do have a separator which is much more resistant than the Pellon, namely, polypropylene, and I think what we should be doing is spending some time on the polypropylene, to make the fibers just a little bit more hydrocrilic.

MARGERUM: Yes. We don't intend to limit our studies to nylon. It just looked like a-- Since there are many satellites that have nylon and will be using nylon for a while, this is something that we thought we should look at.

HAINES: The ones that are up there now we can't do anything about, but we can do something about the next generation to go up.

MARGERUM: I would like to see some data showing some tests showing that the lifetime improvement of the nylon separators after as many years as the nyons have been up.

HAINES: Canada has several satellites in orbit at the present time with polypropylene. One of them was turned off after ten years. They were tired of getting data from it.
J. H. SCHULMAN: What is the Q-10 of the reaction, do you know? Is it linear with temperature?

MARGERUM: I didn't understand what you meant.

J. H. SCHULMAN: The Q-10; in other words, the increase in the reaction rate. In other words, if you go from your three-day degradation to 115 degrees C. back down to 25 degrees C., every drop of 10 degrees --

MARGERUM: We intend to plot that out as a function of temperature. We've just begun this and I don't know what that actual temperature --

J. H. SCHULMAN: For that type of material do you know whether it would be a Q-10 of two or three for that type of molecule?

MARGERUM: We could guess but I'm not going to.

HALPERT: We have two more papers on separators and the answers may come up in those.
VI.3 SEPARATOR EVALUATION: T. HENNIGAN/GSFC

The next speaker is from our own home base here, Tom Hennigan, who is going to talk on separator evaluation, some work that is being done.

HENNIGAN: We have been reporting for the last four years on various separator tests. These have included polypropylene, and we have been trying to determine from a screening test which is run at the Bureau of Standards, can we pick a good separator and a bad separator.

I think we showed a few years ago we can pick a very bad one. We can pick good ones, but not the degree of goodness we would like to tie to.

A couple of years ago we put it on a microfiber polypropylene made by Hercules, and this looked like-- It had characteristics similar to nylon as far as retaining electrolyte during cycling and wetting and so forth.
Unfortunately, Hercules was not interested in this business at all, and took the machine apart, --

-- even the whole battery, because they were not interested in it.

At that time the Grace Chemical Company was interested in it because they were in the separator area anyhow, and we got a series of separators from those people, which are also microfibrous, and we have screened those. And we have gotten some other polypropylenes, and we also got a nylon.

Now the nylon is a very good separator. We've been using it for years and you can knock it but one thing I would like to point out is we have sole source in separators and if anything ever happens to that one, everybody is going to scramble in a hurry.

So I think if we get a nylon or a polypropylene, I think it is worth the effort.

(Cline 139.)

We have some cells built by Eagle-Picher. They are six ampere-hour cells and we're running them at Crane. We just started this about two months ago. There are the Crane pack numbers, and the suppliers, the four Graces, Hercules -- that's similar to the one we tested before -- another Grace, a Vex 1242 which is made by Gaff which is irradiated, RAI, and grafted.

This is the Canadian-- Well, we call it the Vex ISIS which Mr. Haines just talked about. And of course we have Pellon as a control -- that's a nylon -- and nylon Grace also.

Now we did give them capacity checks before we started but we held that goal to 152 per cell, and some of them cut off.

Now this is an average of five cells, the third column over, and some are somewhat low, but we wanted to see how they would cycle anyhow, so we put them on a cycle regime, the same as last time, 25 percent depth of discharge, 25
### SEPARATOR PROGRAM

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Notes: PP POLYPROPYLENE N NYLON

**FIGURE 139**
degrees Centigrade, and a 1.41 voltage limit, and they are overcharging at about 110 percent. They start a little high but they level off between 107 and 110 percent. And that's the number of cycles we have to date, as of last week anyhow, and there have been no failures.

What we intend to do is vary 2,000 cycles, take out one cell, take it apart, and analyze the separator for electrolyte retention. That's about it. Thank you. Are there any questions?

LACKNER: You haven't any cycles for our beautiful polypropylene.

HENNIGAN: We just haven't started it, but it is started. It was started last Friday.

LACKNER: Okay. It creates the wrong impression.

HENNIGAN: Well, how about the Pellon? Any questions?

WEBSTER: Tom, what attempt have you made to keep the interelectrode spacing the same, the thickness of the separator material the same?

HENNIGAN: Well, that's kind of tough when you get all these different materials from different companies. You can play around with-- All these are samples. The only one we paid for was the Wex 1242-RAI.

SCOTT: Do those numbers in the far right-hand column indicate how many cycles they are still going at, or when they failed?

HENNIGAN: No, they are still going.

SCOTT: All of them are still going?

HENNIGAN: Right.
HALPERT: Maybe some questions with regard to the Grace material will be described by Nigel Palmer who is the Technical Director of Fiber Specialties at Grace. He is going to talk about permanently wettable polypropylene separators.

PALMER: First let me make a couple of general comments and that is that what I'm going to say is quite impromptu. I hadn't any thought of saying anything until last Friday when we had a rather relevant patent issue on this subject and this prompted me to call Gerry and suggest that if they were talking about separators that I might be able to give a bit of background information on our separator, not so much to give data on cycle performance and things like this, which is principally being generated by NASA and by various cell manufacturers, but rather to give people who are working with our separators some idea, some background information on how it is put together, what gives it the various properties we think it has, and so they can interpret their results and in turn, working with us, can further the optimization process. As a result of this being impromptu, I don't have any slides or anything like that, for which I apologize.

The other thing I might just say by point of introduction also is that as some of you may know, we are separator people. We have been this for about 25 years, but we are quite new to alkaline separators and in fact only got in it as a sort of what I have to admit was something of an afterthought about three years ago as a result of work we were doing for our principal separator business, which was lead acid.
An having sort of gotten into it, we have kind of gotten interested and involved, and we think we're making an improved product.

I think from the comments there have been already this morning there is a general feeling or agreement among at least a large number of people that polypropylene separators will give improved performance in nickel-cadmium batteries in terms of life, in terms of chemical resistance, and in terms of ability to handle high temperatures. And the down side has been the unreliability of wetting and in particular, the non-permanence of wetting.

This is something that we initially found out again when we were looking at a non-woven product from a lead acid aspect but I think it is common to polypropylene in general, and I think the problem frankly is that polypropylene is an extremely hydrophobic material that, although you can wet it very easily for immediate wet-out -- a thousand things will give it initial wetting -- it is very difficult to get it to stay. You have to go usually to things like grafting and so on, which is tricky.

Let me talk a little bit about the process we've got and how we think we have overcome this wetting problem.

Basically our process, which is a method we licensed from Exxon on an exclusive basis for separators-- We spin our own material. We don't take staple and create a non-woven material. We take the basic polymer and spin it and the process basically involves extruding filaments through a series of fine nozzles and then impinging on those fibers as they come out, a very high velocity stream of air at a high temperature.

This results in a rapid attenuation of these fibers to a microfiber situation, and you're talking about fiber diameters in the one to three micron region. We can make them smaller and we can make them coarser. There are questions of economics that come into play really on a fiber diameter.

The process is not grossly different from that that Hercules used in the material that was talked about earlier.
The advantage of that, from a product point of view, of course is that we have control over the fiber properties, both physical and chemical, as well as the obvious control that anybody has over the actual lay-down aspects of the web itself once it's made.

Now from a product point of view, I identify a number of key factors that I think are different.

First, and most important in my opinion, is the permanent wetting aspect of the product. The other thing I mentioned about the fine fiber diameters which I think there are mixed feelings about. In general it tends to contribute to absorptivity and to wicking and to resistance to cadmium migration, but I think the verdict is not really in on that yet.

Another factor which I think is of some importance is the fibers are continuous rather than discontinuous, which means you tend not to have things falling off.

The next factor is there is no binder involved, so you have a purity possibility you probably lack with materials that are dry laid and bonded.

Going on to the aspect of the wettability, I mentioned that we started this program with lead acid and we found fairly early on that although we had great electrical resistance and good initial performance, the batteries would start failing and they would start shorting and doing funny things on a rather unpredictable basis.

And when we looked into it we really concluded that the problem was the loss of wetting in a situation where you had gassing; in other words, in a charging situation or in a situation where electrolyte levels could drop, anything like that, you would tend to get a loss of wetting in those areas. It wouldn't come back.

So what we did was to develop what we call the internal-external wetting approach and basically what it involves is injecting a suitable wetting material, surfactant, into the polymer during its fiber formation. We select this material, in addition to doing the things right in the
battery, we selected it to be compatible with the polamer while it is being made -- in other words, a single-phase situation -- but incompatible when it's cold.

Then you have a situation where you get in effect a ballooning process, a gradual exudation of this material to the surface of the polamer or the fiber. And this provides, we believe, a permanent wettable surface to the polamer and as far as we can tell, certainly on the tests that we have run over a period now of about three years -- not, I might say, in nickel-cadmium that long -- has shown no loss of this property.

I can't say at what point this would disappear. We don't think -- I think there are some theoretical reasons why it would never entirely disappear.

We have a second -- I mentioned the internal-external. The external refers to a second surfactant component which we add to the outside of the separator to improve the initial wetting characteristics and particularly how fast the separator wets.

For the fine fiber and a fine pore size you tend to wet somewhat slowly and obviously, particularly if vacuum impregnations are being used, this can be important in commercial cells, so we have come up with a package which we think covers both bases.

In terms of data, really I cannot give at this point any data; in fact, I won't be able to because we don't test nickel-cadmium cells. We rely on our various customers to do this.

But what I can say is that we have been testing the separator material of this type in lead acid batteries that we do test, both in field and in lab conditions, for about three years, and have established that the wettability has been retained during this period.

Secondly, we have done artificial tests where we have, for example, washed off the wetting material, the surface wettability with solvents, for example, and then
demonstrated to our satisfaction that the wettability re-
covered under conditions that would simulate cycling in a
cell.

So we believe that this will compensate for this prob-
lem that has been the traditional problem with polypropy-
lené and we're quite optimistic that this will give every-
body a better polypropylene separator.

On last comment is that at this point we are not in a
commercial situation. We are still in an advanced develop-
ment stage. We are making the material on a pilot basis and
it will be rather more than a year before we are in a posi-
tion to supply this material commercially. But during this
period we are certainly very happy to work with people and
supply samples and tailor the product to whatever degree is
necessary.

That is really all I have to say.

HALPERT: Any questions?

GASTON: I just have one comment. You mentioned the
wet-out characteristics. In the aerospace battery business
we're not interested in fast wet-out. That got us into
difficulty with the nylon several years ago, so that is not
the most important criterion.

The next thing is I have a question. You referred to
lead acid batteries. Do you also use polypropylene?

PALMER: No, we don't use them-- Excuse me. We are
developing a separator for lead acid which would also be
polypropylene. We have pretty well homed in on that material
as the best material for a combination of chemical and physi-
cal and economic reasons. So although in principle we can
make this type of material with a lot of different polamers,
polypropylene is going to be the principal material, and we
are developing separators, polypropylene separators, for
lead acid batteries.

GASTON: Is it possible to combine this with the sampl-
ing that is going on for nickel-cadmium cells? It might
make it then,dollar-wise,more attractive for use in both
types of batteries. Could the same separator, the same type,
be used for nickel-cadmium?
PALMER: No, not precisely.

GASTON: If it could, it would be advantageous.

PALMER: Well, let me say in this regard that because of the volume consideration, there is no way you could justify the venture on the basis of nickel-cadmium alone, and this is perhaps why we're only just getting into the nickel-cadmium business, is because until we had a product that had some commonality, we couldn't justify developing a separate product.

But having done so, you can say that the lead-acid business will justify the basic economics and then, as a separate product which admittedly has to be optimized for nickel-cadmium, you can develop the separate product. This is our approach and this is what we've done.

There are differences for nickel-cadmium, obviously, in terms of not only thicknesses and so on, but wetting systems are different and optimized for the KOH and so on. But the basic process will apply to both.

GASTON: Now I have one more comment. I found the Hercules microfiber was a little brittle. It is difficult to handle, particularly in larger cells. It might be important.

If the fiber is too brittle, too fine, it might fall apart if you tried to wet the material --

PALMER: I think it is a good comment. Let me say that we are working to improve the strength but, like Herculon, I don't have any size numbers of comparison whether we are stronger or weaker. But in general, this type of material will not have the high strength, the higher strength of the more conventional fibers.

It's a characteristic of the fiber-making process which does not have the cold draw in it and hence it will be weaker; there's no two ways about it, and it is foolish to pretend that it will be otherwise.

We believe from what we've done and from what we've learned so far that we can meet satisfactory levels of
strength for people's manufacturing operations.

**KLEIN:** Can you tell us what the surfactant is and how stable that is in the KOH environment?

**PALMER:** Sure. Actually what I'll tell you is some of the types of surfactants we use. I'm not going to tell you specifically which ones, but referring to the patent, typically what we have used is non-ionic surfactants of the epoxy type.

I think if you want, I can refer you to this patent and you will get a list of the types of materials that have been tested, which are given as examples. I can give the number. The number is 3847676.

I think it really in a sense is the text of my talk. You will find a lot more detail that is worth giving in that, and to follow up on your question in terms of stability, all evidence we have is that these materials are stable. We have selected them with this in mind.

Like a lot of things, the ultimate test -- I won't say the acid test, but the alkaline test, if you like --

-- is several years down the pike. But all our tests say it's fine, but that's not the problem.

**HALPERT:** I think I want to make just one comment. Tom tells me that samples of that material have been given to the Bureau of Standards for evaluation. The results have already appeared in the latest NBS Report.
Okay. The next speaker, to move from the separator
data to the seals, Peter Mayer of the Ontario Research
Foundation is going to give us some aspects of glass-metal
interfaces in seals for nickel-cadmium batteries.

MAYER: I really don't know how to introduce this talk
because, as you understand, I was not present at the previ-
ous workshops. But the glass-to-metal seal was flagged
down for Ni-cad battery application.

We looked into these problems of the properties, or
the change in the properties of the glass-metal interface
in the seals for Ni-cad applications specifically as to how
these interfaces react in 30 percent KOH aqueous solutions.
The reason for this was, first, it was challenging,
because there's a common opinion that this is the weakest
part, the glass-metal seals, for application to Ni-cad
batteries.

Second, glass-to-metal seals are much cheaper, much
more economically made than ceramic-to-metal seals. And
as I understand from talking to various people yesterday,
the ceramic-to-metal seal is overengineered, it's too
safe for various lower-cost applications. Therefore we
looked at this problem again. This paper is co-authored
by Mr. Lackner from the Defense Research Board of Canada
and Dr. Murphy and Dr. Topping from the Ontario Research
Foundation.

(Slide 140.)

The first slide shows our experimental set-up.

The first objective of the work was to investigate
the reactions between the glasses and metals. A number of
mechanisms have been postulated in the past, but direct
experimental confirmation of these reactions was not pub-
lished. So we set up a EMS cell where we used glass melt
at high temperature and we immersed two metal electrodes
into these glass melts.
One of them, the metal, we were looking for the reaction with glasses. The second was platinum which acted in this case as a pseudo-oxygen electrode.

(Slide 141.)

This slide shows some results of our experiment where you can see EMF measured by using the experimental setup on the previous slide as a function of concentration, in this case of nickel oxide in soda disilicate glass at various temperatures.

(Slide 142.)

This slide shows EMF measured again in soda disilicate glass as a function of concentration of cobalt oxide this time, and as a function of temperature.

(Slide 143.)

This slide shows the over-all cell reaction which is identified from these experiments. Not surprisingly, the combined reaction is a simple oxidation of metal, in this case the cathode, to ionize to enter the glass as a positive cation, as a cation and giving away electrons.

These electrons are recombining at the positive electrode. The oxygen is absorbed by the glass melt.

These experiments have shown that there is a considerably large driving force for the reaction, for the oxidation reaction, occurring if glass and molten metal are brought together at high temperature. Because it is a thermodynamically unstable situation. A stable situation would be metal oxide dissolved in a glass or metal oxide-glass, solution of the glass and the oxide.

However, these reaction mechanics are established from the EMF technique which is a no-current technique, which means there was no current going between this and this, the electrodes; therefore this is a thermodynamic equilibrium. Only the driving forces behind these reactions were measured.
In actual glass-to-metal sealing practice these galvanic cells are shorted through the graphite jig, and there some changes in reaction mechanisms are occurring. The driving force behind the reaction is simple oxidation of metal to the oxide. However because of the mobility of the alkali ions in the glass, these are moving and have a very large electric potential toward the anode, in actual sealing practice it could be the more normal part of the seal.

This slide shows that because of the high concentration of the alkalis at the anode interface new compounds which are easily soluble in water or 30 percent KOH are forming.

An additional effect is increase in alkali concentration at one of the metal-glass interfaces is causing crystallization of the glass, which results in a formation of very low corrosion resistant residue of glass at this interface again.

The next objective of our investigation was to investigate how the corrosion properties of the common sealing glass are changing with addition of transition metal oxide since this situation occurs at the negative electrode.

(Slide 144.)

This slide shows that with addition of nickel oxide to the common sealing glass, you can see that the resistance to the KOH corrosion at 90° -- these are the two temperatures we are investigating for the reaction kinetics, this is time and this is the rate loss in milligrams per square area. And what we found is that nickel addition is decreasing quite considerably the resistance of the glass to the KOH corrosion.

(Slide 145.)

A similar effect is found as shown here by addition of cobalt oxide. Of course the effect is not as pronounced.
This slide shows the effect of the addition of $\text{Fe}_2\text{O}_3$ to the glass, or the influence on corrosion resistance to 30 percent KOH.

In all three cases we found that the corrosion resistance of the glass is decreasing by increasing the concentration of transition metal oxide in this glass. And from this we concluded that the reason why some glass-to-metal seals failed if they are applied in nickel-cadmium batteries is there is a formation of this highly soluble alkali silicate compounds at one of the interfaces.

We have investigated further the influence of transition metal oxide on the sealing glass.

This slide shows a hypothetical case taken from a paper by Bosque, where he shows the adherence of the glass to the metal is increased by increasing, or saturation -- by increasing the concentration to the saturation point of the glass by the oxide of the substrate metal. We can see in all cases the contact angle from the drop test was decreasing with the increasing of the oxide content in the soda disilicate glass.

However, looking at the modified Dupre equation which is shown on the next slide --

--we can see that not only do interfacial energies play a role of adherence of glass to metal, but also the difference in thermal contraction from the sealing temperature to the room temperature.

And we have found very interesting reasons which are shown on the next three slides.
The expansion coefficient of the soda disilicate glass is changing by addition of transition metal oxide. It could be shown that the thermal contraction mismatch, if there is no nickel oxide present in the soda disilicate glass, for example, would be in excess of a thousand parts per million. However, as we approach the saturation point of nickel in the soda disilicate glass the difference in thermal contraction would be less than 100 parts per million.

This would mean -- in common sealing practice this would indicate good thermally matched glass to metal seal.

(Slide 149.)

This shows a similar effect of cobalt oxide on the thermal expansion coefficient.

(Slide 150.)

This slide shows the similar effect of Fe₃O₄. In effect we are getting a greater seal going from the pure glass join to at the glass-metal interface.

In answer, I would like to point out that we have shown that glass-to-metal seal for a fraction in nickel-ceramic batteries could still be considered as a viable alternative to the ceramic -- the expensive ceramic-to-metal seal.

Thank you.

QUESTION: Any questions?

CONCEPT: I just wanted to make sure that underflow your weight loss curves correctly. By increasing the concentration of the metal oxide you actually decrease the weight loss. Which means you increase the resistance of the seal.

TAYLOR: Right.

CONCEPT: I thought I heard you say the degradation of the seal was worse with the transition metal oxide, but you really meant the other.
MAYER: The decreasing corrosion rate would indicate better resistance of the seal.

CORBETT: So higher concentrations are better?

MAYER: Yes; because KOH attacks common sealing glass, mainly in the silica network. So by addition of the higher charge cations the corrosion rate goes down, and there is less chance for KOH attack of the silica.

CORBETT: Have you had some opportunity to build some seals using this kind of approach?

MAYER: Yes. And we are testing them at the present time. I am not prepared to report any results yet.

ROGERS: You pointed out that the coefficient of expansion between the metal and glass match. I don't think that implies the necessary match over the entire sealing range. They match at whatever temperature you were pointing out.

In other words, rarely glass-to-metal match over the entire range.

(Slide.)

MAYER: (At the easel) This is the temperature and this is the changing length per unit length. Let's say that the curve looks like this, and the point which we are usually considering in the glass-to-metal sealing is the so-called Tg where the glass becomes rigid. So the sealing is done at a much higher temperature. And the seal, together with the glass, is cooled from this temperature, and the stresses are set up at this point.

Now the metal can contract more or less. But there is an accepted factor, or accepted sort of limit for matched seals in glass-to-metal sealing practice. For the matched seal the thermal contraction difference should be less than 100 parts per million, which is the difference here.

For a compression seal the limit is about 1000 parts per million.
STOPEL: I hold in my hand a plastic type compression seal. This seal is cheap, easy to make, and is very versatile. Can anyone tell me why it's no good?

CORBETT: It's too cheap.

MAYER: The bonds in organic seals, glass-to-metal, ceramic-to-metal seals, are formed at high temperature. The activation energies for formation of these bonds are very high. And usually the bonds are of two types: covalent or ionic.

In the case of a mechanical seal by the best estimation it would be a Van der Waal type of bonding between the metal and the plastic. And we know the Van der Waal bonds and many others are likely to be lower in strength than covalent or ionic.

In the case of glass-to-metal you have ionic type of bonds. Therefore the bond between the metal and the separator, or the dielectric is much more permanent than in the case of an inorganic seal. Because there's only we can exert that kind of pressure mechanically.

However these seals have definite applications for specific purposes.

FORD: I have a question. Where can I buy that seal? Where do I buy it on the market? Where do I buy it?

LACKNER: We may have an answer for you. Wait until next year.

If you talk to us we may figure something out.

HENNIGAN: Twice we tried to get in the seal business, once with T.I. and once with Tyco. Both times they went out. Maybe we'll try again. It's not like we don't like it. But we haven't been able to keep anybody in business.
with that seal.

FORD: Maybe that's telling you something.

McHENRY: To start off with, I don't believe T.I. went out of business, the battery business, because of the seal. They did try to make a sterilized seal, which did not work. They tried to make a plastic seal and it did not work?

HENNIGAN: It worked.

McHENRY: And they went out of business because of that?

HENNIGAN: How would you test T.I. cells?

McHENRY: Has anyone ever seen a single one of those plastic seals? Has anyone here worked with them at all? I've had a guy out at Jet Propulsion made a few of them, and I don't know of anyone who has ever made one that fit. You can take a torch to it and you'll ruin it if you overheat it.

But we've never had one that failed, other than when the guy put it into the oven and cooked it; which he's not supposed to do.

I've seen them fail when you take a torch to the--when you compress it and you find a leak in your can and you try to weld the lid back on, after you compress the seal. And it will leak if you heat it after you compress it. But you're supposed to weld the top on and then crimp the seal.

We've had them in the old Telstar batteries. And I haven't looked in about three or four years, but they went about ten years without a single leaker. All ceramic seals and all the glass seals had failed.

There were four hundred glass seals, all of which failed in a few years. The ceramic seals, within maybe
four or five years they were all gone. Not a single one of the other type seal failed.

They are reasonabl, cheap to make. Now making one of them is real expensive: you have to make a mold. Just making one can of beer is awfully expensive, too. If you want 6x1 over radical 2 ounces of beer, the guy would have to -- he couldn't make it, not one. Just changing the size of a can of beer would make it terribly expensive. But, you know, if you make them by the thousands and millions it's a lot cheaper if you want to make that many.

I guess for about ten dollars apiece you can make the seals in a very small amount, 100 or 200, something like that. And the thing works perfectly fine.

JOE SCHULMAN: Over a long period of time won't gases leak out?

McHENRY: I've done a little looking at-- We put them on the helium leak test. You cannot detect it.

JOE SCHULMAN: How long? That period of time?

MCHENRY: Leak tests, to ensure the rate of leakage.

JOE SCHULMAN: I understand. But if you put it on one side then you have to wait. You don't wait for a few minutes, you wait for days and weeks as it slowly drips through.

MCHENRY: I can comment on that.

We've been experimenting with these seals on nickel-hydrogen cells where we're pressure cycling with hydrogen between 100 and 500 psi. I guess the oldest seals that we have must have gone through literally thousands of these kinds of pressure cycles, and we don't see any leakage.

JOE SCHULMAN: How did you determine that you didn't have leakage?

MCHENRY: We know how much hydrogen we got-- On long
term we do initial leak testing with a helium leak detecting device. Then we're running cells that have a finite quantity of hydrogen in them. And we're not losing hydrogen.

Then we also do some leak testing after we've run the cell, just for helium leak rate.

As far as we're concerned, the seals work very well in all respects.

HALPERT: I'd like to go on to our next subject.

UCHIYAMA: Let me ask a question.

A reference was made to the Jet Propulsion Laboratory and the Ziegler seal. I would like to mention that as a part of JPL's responsibility to NASA for its deep space mission we are looking at long life batteries, and some of the candidate components which might be used in such a battery. The Ziegler seal is such a candidate, and we have had some headers built with such seals and we are incorporating these into actual cells.

The header which Stockel showed a few moments ago, I believe, is one which he has built for JPL. To date, the cost of fabricating such a header has been low and the procedure has been simple. We hope to present data on these cells at the next GSFC workshop.

Preliminary test data on the integrity of these seals have been reported in a JPL publication available to any interested personnel; the results confirm the data published by Bell personnel in the Journal of the Electrochemical Society.

HALPERT: Harvey Frank has a report on some of this work in a JPL report.

FORD: One point I want to make: We buy cells, and we buy a complete assembly. I buy cells from the manufacturer that offers them to us, and we usually buy them with the headers they offer.
Now would a manufacturer stand up and tell me that I can buy a cell with a Ziegler seal on it today?

That's when we'll use them, when you can go to the manufacturer and buy cells with Ziegler seals; that's when you'll find them coming into aerospace, and not before.

There's a lot been said about the technology. I don't think the technology is even in question. But the commercial availability is what the question is on the Ziegler seal today. And when it becomes commercially available on Nicad cells you'll see them put into use.

HALPERT: Okay. Thank you, Floyd.

I'd like to go into the next subject, otherwise we're not going to get our work accomplished.

Moving now into analysis, the next speaker is Gert Van Ommering from COMSAT who is going to talk about his analysis of cadmium plates.
VI.6 ANALYSIS OF CADMIUM PLATES: VAN OMMERING/COMSAT

VAN OMMERING: I just want to clear up a problem that came up yesterday concerning carbonate buildup in cells stored passively for some period of time.

Dr. Scott presented some numbers that showed carbonate in the order of 25 to 30 and 35 equivalent percent, and Jim Dunlop stood up and said that we saw only about 10 or 11 percent. It turns out we're talking about different percentages. Ours are simple weight percentages on the basis of total electrolyte weight.

I did a quick calculation on our numbers, and it turns out that if we do it on an equivalent percent basis we see levels of about 25 percent carbonate in cells that have been stored shorted for three years. And it varies from about 25 to 27. So it's pretty much in the same range that you were talking about. We see the same kind of level also in cells that we stored open for the same amount of time.

So what we're concluding from that -- and whether it's a 100 percent valid conclusion we're not quite sure -- is that this carbonate is coming from plates and is slowly leaching out with time. Even though for some people it is not that clear that it can happen. But if you look at it thermodynamically it will eventually happen. The tendency is there for the carbonate to move out of the plates into the electrolyte.

(Slide 152.)

On this graph we show on the top curve some of the data that we have presented before on carbonate increase in cells that have been run in the Intelsat-4 real time simulation test. The top curve gives the numbers that we presented to measure a rate of about 1.16 grams per year increase.

It's interesting to note that if you look at the legend at the bottom, that the increase turns out to be practically the same for cells that have been trickle charged as compared to cells that have been stored open.
circuit during the storage season. Initially it looked like the trickle charged cells were building up carbonate much faster than the open circuit cells were. That must have been the initial scattering data. It looks like everything works out pretty well, and you can draw a beautiful straight line for this.

Then we also draw a beautiful curved line through just about three points which describe this passive storage. Those are the initial points right on a 'Y' axis here. Those are measured initially on new cells. The carbonate level is about 5.

And these little dashes here show points that were measured on cells that had been stored for respective two and three years. We drew kind of an arbitrary curve through that and subtracted that curve from the top one, and we came up with a corrected line at the bottom which shows the actual increase of about .82 grams per year that we can attribute to some type of oxidation that occurs in the cell. And that number fits pretty well with the precharge data that we have on the cells also. The increase in precharge is very low.

So that sets that straight.

The other story that I have concerns the cadmium electrodes in cells that we have taken out of the real time test that we're running. For people who haven't been here before, let me briefly say we're running a real time test on nickel-cadmium batteries. One pack of batteries is stored in a storage season between eclipses, open circuit, with periodic recharge every thirty days. Another pack is trickle charged continuously during the storage season. And the eclipses that are run are exactly the same. The tests are primarily intended to evaluate the effects of various storage modes and to see whether they have any detrimental effect on the batteries which would limit their life in orbit.

We recently took some cells out of these tests.

(Slide 153.)
In the trickle charge test we see this kind of behavior during the eclipse. The points that are marked there show the highest cell of the five cells that are remaining in the test in terms of voltage at the end of the longest eclipse, or in terms of voltage at the end of the eclipse charge.

The lowest point at each eclipse day shows the lowest cell in that group. And the little dots in there are the average for the five cells in the pack.

The spread here for the trickle charge cell is fairly small. It looks like it's about a 30-millivolt difference maximum for the eight points between the best top cell and the lowest cell. And the average voltage for the longest eclipse day is about 1.17, which is fairly good, especially if you compare it with the packs that had been stored in open circuit mode.

(Slide 154.)

You see the results are a lot more erratic. Apparently there's one cell that looks very good, and that shows a very nice smooth curve there at the top. That one is performing fairly well. But there is a considerable amount of scattering in the data on the worst cell, and the spread is quite significant. It's more like 200 millivolts, whereas it was 30 millivolts on the other test.

Also, the average voltage found in the longest eclipse day is something like 1.10 instead of 1.17.

This shows that the open circuit storage does have some bad effects. The little dots that are circled here in these two graphs show the value of the voltage for the particular cell that we pulled out of each test in the most recent seasons.

(Slide 155.)

This shows the discharge curves on those two cells plus the discharge curve on the new cell which was run a long time ago.
The top curve here is for the new cell. The other two curves -- this is for the trickle charged cell run at the same rate, the same kind of test. You see the capacity was decreased quite significantly. And the voltage loss is also fairly large. But it is not as bad as the open circuit cell which has lost even more capacity and more voltage.

These cells do seem to be negative limited to some extent, especially the open circuit cell is negative limited when you go to discharge. Although generally when you go into discharge you find that you're generating both hydrogen and oxygen. So it is not really a matter of the capacity not being there. It is there. You can use it; but at a very low rate. You can't really use the capacity at the full rate that you would like to use normally.

The amount of oxygen that we generate over discharge does not correspond exactly to the amount of current we're putting in.

So either we're still discharging some of the cadmium in the plate electrochemically or we're recombining some of the oxygen that we generate in the reverse with metallic cadmium that is around but that for some reason cannot be discharged electrochemically. Maybe because it's drying out or something of that nature.

We opened these cells up for complete chemical analysis. And I'm not going to present everything here, because everything has not been quite completed.

(Slide 156.)

This is not such a very clear picture of cadmium plates from these cells that were pulled out. The one on the left is a trickle charged, one of the trickle charged cells. It looks fairly clean. There is some irregularity on there that kind of got washed out of the picture. But compared to the open circuit stored cell it looks very clean. There's practically no separator sticking to the plate. In the open circuit cell there's an awful lot of separator that you simply cannot get off.
There's quite a bit of non-uniformity in the open circuit cell. You can see apparently there are some high pressure areas, or high air pressure areas, where the electrolyte preferably is, and where possibly the cadmium moves around more and kind of attaches the separator to the plate.

The positive plates look very good. They generally don't have any separator sticking to them. For the trickle charged cell they look very uniform in color, but for the open circuit cell they show the same kind of pattern that you see on the cadmium plate. It generally seems to be somewhat darker, the areas where you see the separator on the cadmium plate. And these two things generally cover it very well. I don't have any pictures unfortunately, I didn't bring any plates either, which I probably should have done.

You saw these little lines drawn across these plates. These two particular plates were cut up into twelve little pieces because we saw this non-uniformity and we wanted to check out what actually was happening in each of these squares.

We cut these things up and did a separate analysis of each of those pieces.

(Slide 157.)

This slide kind of refers to that. The same kind of pattern is shown here. And this particular slide shows the thickness variation across the plate as well as the thickness difference between the open circuit storage and trickle charge storage.

The upper numbers in blue are the ones that we measure right after the plate has been taken out of the cell. You can see that in the trickle charged cell the numbers are fairly uniform. There's really not very much difference between the thicknesses. This is in hundredths of millimeters.

On the open circuit storage cell, for one thing the numbers are somewhat higher, or quite a bit higher. And
they are less uniform. You can see in the area that corresponded to the black area on the plate where not much separator was sticking that the numbers, the thickness numbers are fairly low. Of course people will say that that is due to the separator being there on the plate, and you will automatically measure a somewhat thicker plate at that point.

That's true. But the reason for that is that the cadmium did come out of the plate at those points, and it does attach the separator to the plate. So there is an indication here of cadmium migration being worse in certain areas of the plate than in other areas.

After extraction of the plates we again measured the thickness. And it's shown here in the bottom figures that are colored in red.

You can see that there is a slight drop in thickness on the trickle charged plate, which you would probably expect. But it's fairly uniform. And the open circuit plate, we find that the thickness has increased tremendously, and is practically the same as for the trickle charged plate after its extraction has been done. So it shows that the center of the thing actually does not expand; it is simply cadmium covered all over it, and the sinter stays intact.

(Slide 158.)

We also did on these twelve separate pieces a measurement of the amount of cadmium that is in a discharged state in these various areas. And again there was a very uniform distribution in the trickle charged plate. There's a maximum of 5 percent difference.

If you look at the open circuit plate there's a much larger variation. And the interesting thing is that there is more discharged material in the areas that were not covered with the separator, where the separator did not attach. That would almost indicate that the places where the separator does not attach show a better utilization of cadmium as the plate was discharged, before the cell was
opened up. But you cannot be 100 percent sure of that.

There's also the possibility that this area was dry and that that was the reason why the separator did not attach there so much; that the other areas of the plates were utilized quite a bit, but simply cannot be utilized very far because of pore blockage, et cetera. So the actual utilization of the plate would take place in these areas that show bad migration. And then when you go into reversal and generate oxygen, then there's a fairly dry area of the plate which is at this point (indicating). It probably has a bit of charged cadmium in it, but it can fairly easily recombine with the oxygen that's around it. So at this point you cannot really be quite sure of what is going on there.

But we feed that the second explanation, the recombination in reversal, is somewhat more likely.

(Slide 159.)

We took some of these plates and cross-sectioned them and looked at them under fairly high magnification. And this picture shows some interesting things.

This is the trickle charged cell. We cross-sectioned the plate, and you can see here on the top picture it shows an overall material distribution. You can see the center grid, a little piece of it, right there. And then we have--We're kind of looking through a perforated hole in the thing. And here you see the sinter with the actual material.

The middle picture shows the distribution of the cadmium. You can see it is really not very uniform. There are some areas where the cadmium accumulates much more strongly. But it doesn't look too bad. And you can also see that the outline of the plate is still very similar to the top picture. So practically there's no cadmium coming out of the side of the plate; which, of course, also is shown easily by the thickness measurements.

So this just confirms what we already said.
The bottom picture shows the sinter basically. It shows the distribution of nickel. And you would see, if you superimposed these two pictures, it would fit quite nicely.

There are fairly bid voids in the sinter which correspond to the high concentration of cadmium; which is what you would expect.

We were a little surprised to see that the sinter is that non-uniform and does have some big gaps.

(Slide 161.)

Now for the open circuit cell things look a little different.

The top picture again shows the overall distribution of material. You see there's quite a bit of stuff outside this plate. To get a good feeling for how much that is, you can compare the top two pictures. The middle one shows cadmium, the bottom one shows nickel. If you compare those two with the nickel you can see that the same outline exists for this edge he.e, but that there's an awful lot of material outside, which is very dense if you compare it to some of the other areas within the plate.

So it is very likely that this contributes a lot to the reduced utilization in those areas where the migration is severe. There's a lot of blocking there.

The thickness of this layer, you see, is something like 30 microns. It's quite a bit.

We took some surface pictures of both types of plates to see what this material on the outside really looks like. And we have two magnifications.

(Slide 162.)

One of them is-- I don't know what the magnification is, but the scale is shown here. On the left we have the trickle charged cell. It's not that easy to see, but you can see that the grain structure is fairly fine, fairly
small grains around, really no big ones to be seen anywhere.

On the open circuit storage cell you can see that the crystals are quite a bit larger. You can see some of the fibers also embedded in the material on the outside of the plate.

This was kind of interesting, so we blew it up a little bit more. And you see horrible things.

(Slide 163.)

On the trickle charged plate, again, the crystals could be estimated at about 1 micron, which is not particularly huge. But on the open circuit plate we find some monsters.

There's one which, if you compare the scale, is almost 40 to 50 microns big, which is incredibly huge. I don't think anywhere we have seen anything reported on cadmium recrystallization where anybody showed crystals this big in an actual cell. At least I haven't seen anything. I know there has been a good bit of work done by Fritz Will, for instance, on that kind of thing. But those were not really real electrodes. And the type of crystal sizes he was looking at were on the order of 5 microns, which already gave a fantastic reduction in capacity. They were actually non-useable.

So here we are building up some real big ones. And the thickness of this one crystal here is about 30 microns that we measured as the thickening on the plate. So you can see it has almost some single crystals, or big crystal sitting on the outside, held onto the plate by the nylon.

We're not sure yet what the base structure is that you see behind the crystals. This surface could be hydroxide, it could be metallic cadmium. We're going to check that out. And you probably may hear about that next year.

So this is what we have been seeing, and it just confirms what we have already seen before, or have said before, that the open circuit storage does give you problems in
terms of this cadmium migration much worse than the trickle charged cells. Open circuit storage looked good for a while because we thought there was less carbonate buildup with the open circuit storage compared to trickle charged. But, as you saw in the first slide that I showed, that's not really true any more. And there was probably some early confusion in the data. So that's not really valid at this point.

So it looks like open circuit storage really doesn't have any advantage over trickle charge. Trickle charge seems to maintain the plate in a better manageable condition.

There is some spread in voltage, as you can see from the early slides. There's some loss of capacity, some loss in voltage after something like five years. But it is still bearable compared to the open circuit ones. --that is, that the trickle charge is quite a bit better.

I guess that just about covers what I wanted to talk about.

HALPERT: Any questions?

COHN: Without doubting the validity of your data, it reminds me in a way of the chemist who made the experiments with skotch and soda in which he got a terrible hangover, and bourbon and soda. And you know the rest of the story.

The message you were giving us I believe was that you prefer trickle charge storage over open circuit storage. And the message I would like to leave with you is, perhaps you're oversimplifying things.

As for the conditions under which you operated, there's no argument that your conditions for trickle charge storage were better than your conditions for open circuit storage. However, one has to know what kind of cells you used, how you treated these cells, and what conditions. And it may well be that an open circuit storage with different treatment of different cells might give you the exact opposite conclusion than you have drawn from your data.

VAN OMMERING: I'm limiting my conclusion to this
particular test. I'm not saying this as a general thing.

WADHAM: On Mr. Cohn's point, COMSAT hasn't mentioned that it has also been testing our cells. And we find exactly the same results with a different design of plate, a different batch -- their's are 1969 cells, ours are 1970, I think it is, or '71 cells. The whole design of the plates was changed by G.E., as I think most people know, aroundabout 1969. And on open circuit storage our cells are, if anything, worse.

VANOMMERING: I guess that kind of limits it then to synchronous operation of G.E. cells.

COHN: Let me ask a question now. Have you got any data on the amount of overage that you get in both regimes. How does the amount of overcharge compare in both regimes? I guess the amount of discharge would be the same. But the amount of overcharge might be very different.

DUNLOP: First of all, to go back, these tests were started in 1969. And at the time we started this test I don't think we were drinking either skotch or bourbon. We were trying to simulate a test that we felt was valid because these were the two choices that we had. And we unfortunately chose the open circuit. We didn't start out here trying to prove anything; we started out with the two storage modes, realizing in the satellite that we had that the storage mode we were using was the mode that we have now shown is where our problems were.

To explain the overcharge: we knew exactly what we were doing: there's a 10 percent overcharge at a C/12 rate, and we switched back to a C/36 rate during each eclipse operating day. That's for the eclipse period.

The only other difference is the one that was mentioned here, in the storage mode. One cell is charged in the open circuit mode with recharge every 30 days, and the other is on trickle charge. That's the mode these two cells have been running in, and they've been running in now for nine eclipse seasons, or eight eclipse seasons.
The other point is that the depth of discharge is 60 percent of the rated capacity on the 72-minute eclipse day. I think there is no doubt that the results we see in this test on those particular plates, and the experimental results that we have seen on the Telsat plates, which are -- I'm talking about the negative plates, the negative electrodes, in both cases we are seeing this significant expansion of the cadmium electrode, particularly at the bottom part of the electrode.

I don't want to go into why it's occurring at the bottom part. But we don't get this expansion on the plates that we are trickle charging in the Intelsat-8 plates. We don't know o- telesat because we don't have any comparable type of test data.

There is no question in the data that this open circuit stand mode is a great way to go are far as cadmium crystals are concerned.

CORBETT: I have a question about your data. But first let me say I think it's a very interesting piece of work and that it sheds some light on some problems that I think a lot of battery engineers have been experiencing without explanation, like why system test batteries on satellites tend to die if you leave them alone. And people have been asking, you know, "What have those guys been doing to the battery?" And there have been a number of problems around the company, both on the military, on the NASA side, with respect to system test batteries.

The question I have, it's kind of a subtle point, but on your first chart where you showed the regions of the plate and the difference in cadmium -- the difference in thickness, a comparison of the thickness both after removal of the hydroxide and prior, I notice that the actual thicknesses were slightly lower for the open circuit storage cells. Is that a significant statistical difference; and, if so, why do you think that is?

VAN ONMERING: That is a difference that you generally expect to see from plate to plate. That is something that does happen. There is some non-uniformity in the sinter
CORBETT: They all seem to be in the seventies on the right and the eighties on the left.

VAN OMMERING: They were about 77 or 78 on trickle charge, and 79, 80 on-- 79, 80 on trickle charge and 77, 78 on open circuit. That's the normal variation. We have been seeing that all along.

FORD: While we didn't go into the detail to show the redistribution of the material in the cells, there was experimental data presented in one of these sessions two to three years ago that compared the effect of open circuit, what we call the random open circuit, with trickle charge, with discharge and shorting. And it happens this confirms exactly what Jim and Gert here have reported; that the open charge stands are a very unhealthy environment for the battery.

With that as a comment I'd like to ask a couple of questions. One is, Did you look at how much separator-- or how much electrolyte was in a separator with respect to each test condition? Was there a significant difference?

VAN OMMERING: No. When we analyze a cell what we generally do is we open it up and then extract the entire cell. So we really don't have any data on where the electrolyte is, whether it's in the negatives, the positives, or separator, or how much exactly in each of these three components. Unfortunately we don't have that.

FORD: One of the things we found out in the storage test, where we were simulating ground storage conditions, not like your in-flight storage, was that the amount of electrolyte that was retained in the separator was the lowest in trickle charged cells. Now I don't think we realize yet what the long term implications of that may be. But it's the only problem, or the only indication of a problem with trickle charges I've seen so far--and I'm talking about moderate rates, now, C/30. --is associated with the redistribution of electrolyte. And also with that redistribution of electrolyte the cells did show an increase
in capacity to 1 volt, indicating a possible expansion of a positive electrode.

In your discharge data, your voltage profile data, did you indicate in your talk if you determined which was the limiting electrode in reversal?

VAN OMMERING: Well it generally looks like it is the negative one. Because you see some oxygen as soon as you go into reversal. But you generally also see some hydrogen. And it's really extremely hard to detect which is going first.

In this kind of a tapering down thing the electrode cannot quite support the discharge rate that you're using, so you get kind of a mixed reaction. And it seems to happen on the negative as well as on the positive, though on the positive it's to a smaller extent. But there is a utilization problem at the end; it's not a straight cut-off, it's kind of a slow tapering from one mode to the next.

FORD: In your chemical analysis of the negative for charged material, as far as total charged cadmium, is there any distinction made between the two groups, or did you notice any difference?

VAN OMMERING: Well the problem is that you have a hard time comparing cells like these, using, for instance, a bar graph as we have been doing in the past. We cannot really compare very easily because we don't know exactly what goes on during this reversal. But what we generally try to do is we collect all the gas in reversal, and you generally find an imbalance either way. Either there is more hydrogen or more equivalents of hydrogen and oxygen, or the other way around.

Using that kind of a calculation with an estimate of the overall positive capacity you can arrive at a bar graph for the negative electrode. And at that point you could make a decent comparison of how much you have in each electrode for these different cells. You really can't do it at this time. We're still working on that.
FORD: One more question and then I'll stop.

On the large crystals that you referred to, are those fairly sized crystals, or is there any indication that the crystals are built up by layers like you would expect in Formica or something like that?

VAN OMMERING: You often see that in pictures of potassium hydroxide crystals. You see this kind of step structure.

I think Will, in his paper, showed that generally happened after exposure to air, or something like that. It somehow made it worse. I don't know what that means. I guess it means that there might be some dehydration of the hydroxide to oxide, or something like that.

We don't see these steps very clearly. There is a very vague indication that there might be some ridges on that surface, but our pictures don't show a very regular pattern of ridges as some of Will's pictures show.

THIERFELDER: Have you investigated whether these large crystals can be broken down by reconditioning, or are they permanent?

VAN OMMERING: Well they're almost permanent. Theoretically you can show that you should be able to bring cadmium back to the plate and slowly dissolve these big crystals by very long continuous charge. That should be a possible way of bringing those back.

With big crystals like this it might take years.

DUNLOP: I'd like to make a couple of comments.

In regard to some of the questions that Floyd Ford asked, in terms of the original redistribution of cadmium, that's the first point: where you see that -- where we cut those plates up into twelve different sections and analyzed for the total amount of cadmium in each section was the same. So where you see that expansion occurring in the
bottom of the plate it isn't a slumping phenomenon that you normally associate with something like the zinc electrode, it is simply the movement of the cadmium from inside the plate to the surface. But we find it's the same total amount of grams of cadmium per gram of plate material in each one of those twelve sections, whether we're talking about open circuit or whether we're talking about trickle charge.

The other point is, in answer to one of the other questions: in that plate, in terms of the utilization of the amount of residual charged cadmium remaining, it turned out that in the plate that was trickle charged we were able to utilize 70 percent of the active material uniformly in every one of those twelve sections.

In the plate that was open circuit we were able to utilize approximately 70 percent of the active material in the upper part of the plate and only 53 percent of the active material in the bottom part of the plate. That meant there was a remaining large amount of charged cadmium, about 50 percent of the active material in the bottom part of that plate that was charged cadmium which we could not electrochemically discharge.

VAN OMMERING: That refers to utilization in the cell. If you would run these things flooded things might be different.

DUNLOP: But at the same time it's very likely that this is a masking effect going on, too, in the bottom part of that plate.

HENDEE: Jim kept side-stepping it, but I'd like to put out a general question: What causes the stratification from top to bottom?

VAN OMMERING: Do I have to answer that?

What you want to hear is, It's gravity.
HENDEE: That's not what I want to hear. I want to hear the truth.

VAN OMMERING: Nobody knows the truth. I think gravity is a good candidate mechanism for this thing.

It is obvious that -- well, in all these cells, if you look at them, for some strange reason the bottom is always the one that shows the most migration. So it makes you think about gravity.

I wish somebody accidentally had run some of these things upside down. It would be interesting.

But there are other possibilities. The plates are not uniform in thickness over the entire area. And the way they are compressed in the can, it is possible that there are some high pressure areas.

And of course you would expect that there is somewhat more expansion in the center of the cell. So that you would expect the high pressure areas to kind of occur around the perimeter of the electrodes. In those areas you would tend to contain somewhat more electrolyte than in that low pressure area. That would possibly accelerate cadmium migration. And as soon as you get cadmium in the separator it would improve the electrolyte holding capability of the separator at that point even more.

So this is the kind of effect that would accelerate itself. But it has to be a concentration of electrolyte, whether it's to high pressure areas where the electrolyte concentrates or whether it's to gravity. It might be a combination of both, and probably is.

HENDEE: Well then probably the conclusion is, even if initially it was gravity and it were to be transferred to zero g, that the process would probably be self-perpetuating and continue in the general frame of reference, that the bottom would be expanding.

VAN OMMERING: That's true. The cell has been-- Assuming that mechanism would be correct, the cell is going
to run a little while at the ground, and then it's launched to a zero g environment. The effect would still be stronger in that particular area where some migration had occurred already.

Of course, in a satellite you're not running in a zero g environment actually; you're running at a tenth g.

DUNLOP: Approaching this from the other direction, it is interesting that you don't notice the expansion on plates that are trickle charged. Therefore you could assume, you can start postulating, Why not? And that's a reverse way of approaching the same problem.

It may mean that when you do trickle charge plates you may have a mechanism by which you redistribute the electrolyte more uniformly throughout your cell so that you're able to more uniformly discharge your plate. Because there's an obvious indication that you are -- that these plates that are expanding, that you are getting electrolyte redistribution, by whatever method, in the bottom of the cell, and that in doing that that's the part of the plate that's probably being utilized, and that's why you get the cadmium movement. And the whole thing is self-perpetuating in a way.

That also brings up a second point.

When you do try and analyze the separators and look for the electrolyte, where do you look? Do you look at the top part of the cell? Do you look at the bottom part of the cell?

His question is a rather interesting one, because I don't think that electrolyte in that open circuit stand cell is distributed very uniformly in that separator.

BENE: On your recharge on the cells open circuit, what was your charge regime for that?

VAN OMMERING: I think C/12 to 10 percent.

DUNLOP: C/24 for one day.
VAN OMMERING: Before the capacity measurement.

BENE: Before the battery was discharged?

DUNLOP: I guess I missed your point.

BENE: Well the cells you have on open circuit stands, when do you recharge?

DUNLOP: Every thirty days, C/24 for twenty-four hours.

BENE: So it's possible you have gotten more recharge in those cells than you got on a trickle charge cell.

DUNLOP: Not recharge; because the trickle charge is more overcharged than a 1-day C/24.

BENE: In one sense you can look at it that way, yes. But you maybe generate more oxygen with the C/24 for twenty-four hours in those cells than in the other.

DUNLOP: I don't know why that would be a problem with cadmium, offhand.

BENE: Well let me ask another question, then.

Were these drawn cans or welded cans?

VAN OMMERING: Welded cans.

FORD: There's one condition that you're overlooking, though. When you go open circuit charge the oxygen recombines and the cell ends up in a hard vacuum, and you have an environment where there is no oxygen recombination mechanism taking place, contrasting to trickle charging.

Now I think this is where you've got to look to find some solutions to this problem, if you really want to solve it. And this is one of the things that occurred to us in looking at the random storage mode, is that there were weekends and seven days on end and those batteries were on open circuit. You come in and hit that battery with a charge.
For practical purposes the electrodes are charged. And what's going to happen? Something has got to go. The reaction isn't a normal reaction if you start from way down and come back up again. You have a different environment that the cell is seeing internally, and you've got to look at it in that sense.

I think you have to look at the effects of oxygen in the cell and what it does to the cadmium.

VAN OMMERING: If you're worried about the cell being fully charged and then being overcharged extensively when you recharge it-- Is that your worry?

FORD: No; I'm worrying about the fact that if you go open circuit on a fully charged battery, say for twenty-four hours, if you test some of your pressure gauges and you know when you come in after open circuit on a week-end the cells are in a good hard vacuum, what happens to a cell that for all practical purposes-- Those electrodes haven't self-discharged. You hit it with a charge again. You're in overcharge already. The recombination mechanism that suppresses the potential of the cell in overcharge isn't there. What's happening to the cadmium metal during that period?

VAN OMMERING: One thing I have to say is that the cell is not exactly on open circuit during this period. There's a very small load across it, extremely small. So the thing does discharge.

JOE SCHULMAN: I do know if you try to grow salt crystals-- something like that in a beaker that if you keep wiggling the jar, keep moving it, they'll never grow. If everything stands perfectly still it'll grow.

Could it be that on open circuit there's no mechanical motion of the fluid, where in trickle charge you have a little bit of transfer of material?

VAN OMMERING: I don't know if that directly keeps the crystals from growing. I think the slow discharge which occurs during the open circuit is really what's responsible for the crystals getting a chance to grow. I think that's
the main mechanism.

HALPERT: Thank you very much.

Gentlemen, we'd like to let you go earlier this after-
noon, so if we can get back here and start exactly
at one-thirty I think we can get everything completed in
time.

So please come back and let's be ready to go at one-
thirty.

VAN OMMERING: I would like to say that most of this
work was done by Marty Earl of COMSAT Labs.

(Whereupon, at 12:45 p.m., the workshop was recessed,
to reconvene at 1:30 p.m., the same day.)
VI.7 A NOVEL METHOD OF PLATE CARBONATE DETERMINATION:
H. KROGER/GE

HALPERT: Our afternoon session will begin where we left off, on chemical analysis, and our first speaker this afternoon is Hans Kroger of General Electric. He is going to speak on a novel method of plate carbonate determination.

KROGER: I am going to present to you a piece of supporting development work which was done at the Advanced Engineering Laboratory of General Electric Battery Business Department, and which subsequently led to a novel method of determination of carbonates on plates for alkaline cells.

Conventionally the substances to be analyzed for carbonates on plate materials for alkaline cells is acid digested in a closed system and the resulting carbon dioxide is then swept out and absorbed in a barium hydroxide solution. The resulting carbonate of barium is collected and pre-weighted, filtered in crucibles, and carefully washed and dried to weight constancy.

This procedure, including the preparation and associated cleanup operations, is rather time-consuming. The method does not lend itself to a large amount of samples to be analyzed in a given span of time.

Last year at this time and place we were introduced to a new concept of carbonate determination by Clint Jones of Goddard. He replaced the carbon dioxide collection by means of barium hydroxide by a condensation of the CO₂ at liquid nitrogen temperatures, and a subsequent quantitative determination in a calibrated gas chromatograph.

This is a rather elegant approach. However, it did not produce a significant saving of time.

In our department we have historically always used the conventional method of CO₂ absorption in barium
hydroxide. However, as I said, this procedure is slow and expensive.

When we needed a new, time-efficient method, the feasibility of the gas chromatograph approach was ruled out very quickly for reasons of cost and long lead time to get one, and then there are apparently two other avenues that remain, namely, absorption of the liberated CO₂, an excess of caustic and the subsequent quantitative determination by means of the well-known double-end point acid-based titration or the absorption of carbon dioxide in a known amount of caustic where a color change of an appropriate indicator would permit a go-no go decision with respect to permissible carbonate levels.

We started our investigation with the first approach since it promised to be more beneficial with regard to quantitative information. We prepared a .2 normal or .1 molar aqueous solution of potassium carbonate, and this was used for testing and calibration purposes.

When directly titrated with a standard one-tenth normal hydrochloric acid, we consistently obtained the concentration of five milliliter reagent between the two end points for each five milliliter of the potassium carbonate solution.

Subsequently this standard carbonate solution was then used in the existent equipment to try our concept.

(Slide 164.)

Now these are our results. The column, "Vessels," stands for the number of wash bottles connected in series. The volume is the amount of absorbing liquids in milliliters. The normality is the strength of the liquid calculated from the initial KOH concentration. And the diluting water and the yield is the amount of carbon dioxide to recover expressed in percent of the amount given.

The first trial with one wash bottle only containing 125 milliliters of about .02 normal KOH yielded only about 40 percent of the CO₂, the first line there.

Certain improvement in the collection of CO₂, 52

225
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FIGURE 164
percent, was observed when two vessels connected in a series were used. That contained about the same amount of caustic as used in the first trial, however at a lower concentration.

It should be mentioned here if two vessels were used before the titration was done, the liquids of the two vessels were connected and the vessels carefully washed out.

Based on these two observations we got only 40 and 52 percent when low concentration solutions were used. This ruled out the application of the go-no go decision using a change of indicator in the solution.

Then in the next trial we increased the concentration of the KOH by a factor of two and again we only got 50 percent of the theory.

When we then further increased the concentration by a factor of ten we came up to 88 percent and in a series of trials we were not able to increase to a hundred percent recovery.

It was then felt that obviously our method of titration was responsible for these shortcomings; namely, we titrated the bulk of the unreacted KOH with one normal hydrochloric acid until the phenosaline indicator became colorless. This is the normal practice.

Then we continued with one-tenth normal acid until the change of the second, the methyl red indicator. The use of the stronger acid in reaching the first end point apparently led to continued overshooting of the first mark and consequently to too low yields in between the two points.

The adverse phenomenon of the first end point overshooting could be overcome in the following manner:

The titration with normal hydrochloric acid was terminated about one milliliter prior to reaching the first end point; that is, at a consumption when the intensity of the color of the phenol-telahene solution, or indicator, begins to fade.
The change to colorless is then achieved with one-tenth normal acid. At this point the second indicator is added and the titration is, with the tenth normal acid, resumed and the second end point is reached.

Applying this new practice and a further increase in the caustic concentration, we applied it to a series of further trials with standard potassium samples. We found that these five milliliter runs required a mean of 4.9 milliliter of that one-tenth normal acid with a standard deviation of only .03 milliliters. This is typically the hundred percent recovery as seen in the last line of the figure.

In a typical plate carbonate determination we used sample sizes of ten square centimeters or tenths of a square decimeter. In this case, each milliliter of the tenth normal acid consumption would be equivalent to 138 milligrams of potassium carbonate per square decimeter where all carbonates are calculated as potassium carbonate.

Thus the conception of the trials method that has been implemented in our quality control plate laboratory for both positive and negative electrodes. The inherent savings in preparation and clean-up time can amount to three or four times more samples to be run in a given period of time.

In summary, I have described to you how we modify the HO end point method for total alkalinity, carbonates, and then successfully applied it to the determination of carbon dioxide liberated from electrodes for alkaline cells.

The new method is fast, accurate, and has good economic aspects.

Thank you.

HALPERT: Are there questions?

Bill Webster.

WEBSTER: Yes.
You referenced time. What time does it take to run a sample from start to finish?

KROGER: It takes usually—Digestion and flushing over take somewhere between 10 to 15 minutes. Then you can set aside the solution and charge the train again and start it. As a matter of fact, one operator operates two trains at one time. And the titration itself maybe is five minutes, so we cut the time from originally 30 to 35 minutes per run to about 20 minutes with two at the same time.

WEBSTER: How long do you think the Jones’ technique takes?

KROGER: Pardon me?

WEBSTER: How do you think the Jones' technique takes?

KROGER: I asked him last year and he said 30 to 35 minutes.

WEBSTER: I just wanted to make that point. It is definitely a cost saving since you don't have to have a chromatograph.

KROGER: I was last year quite intrigued by the elegance of his method but I specifically asked him how long one determination lasts and he said, well, 30 to 35 minutes.

WEBSTER: Thank you.

HALPENG: Any other questions?

STOCEL: My pencil couldn't write fast enough. Do you have the thing written up so I could get it in the short term before the notes come out for the workshop? It is very timely from my point of view.

KROGER: I have only my original manuscript here, but maybe I can send you one if you'll give me your address.
Our next speaker is one who joined us this summer on a NASA-ASEE summer fellowship program from Catholic University, Mr. Lee May, who is going to talk about the role of iron in the nickel-cadmium cell.

VI.8 THE ROLE OF IRON IN THE NICKEL CADMIUM CELL:
L. MAY/CATHOLIC U.

MAY: All through the literature on the nickel-cadmium battery there have been allusions to the role of iron in the performance of the cell. If I may go back to perhaps what some would regard as the prehistoric age of the nickel-cadmium battery, I would go back to Thomas Edison who is quoted in several books, for example, as stating that there is a decrease in the electrochemical capacity of nickel oxide electrodes when small amounts of ferric hydroxide are added to various electrolytes.

Jungner, who I guess is the inventor of the nickel-cadmium battery, also claimed that the iron in solution combines with nickel at the positive electrode to form a nickel-iron compound that is inactive electrochemically. And actually I think in his original patent he proposed making a mixture of cadmium and iron plate although he did state that iron affects the capacity and he restricted the mixture of cadmium to iron to 85 to 15 percent, certain structural properties of this cadmium-iron.

Vinal, in his book in 1940, claims that iron in electrolyte affects the positive plate resulting in a loss of activity. Sanel, in 1939, in an abstract to the Electrochemical Society, stated that the presence of iron in cadmium plates did not affect the charging and discharging curves but that the beneficial effect of lithium was to counteract the deleterious effect of traces of iron.

Up until that time there was no actual facts and figures to support these contention. Perhaps the first study was made in 1966, and I would like to show you some of the results.

(Slide 165.)
Troilius and Alfelt built their own cell and used radioactive iron-59 to trace the movement of the iron throughout the cell and as you can see when we’re dealing with the components of their positive electrode assembly, we find that with the number of cycles, the amount of the radioactive iron increased.

The ordinate is the weight of iron in milligrams per plate. And you also note that the contamination of the positive plate -- electrode, excuse me -- increased with temperature.

A second experiment is they doped the cadmium electrode with radioactive iron and they determined the amount of iron which appeared in the positive electrode. As you can see, as the number of cycles increased we have an increase in the amount of iron in the positive plate. And again there is an increase as the temperature increases.

They also measured the effect on the capacity in a nickel-cadmium cell which they constructed themselves, and here they plotted the capacity versus the number of cycles. The top curve represents a cell in which no iron was added. The second curve represents putting one bit in the positive electrode. You see there is a diminution in the capacity.

The difference between the standard and adding the cadmium to the plate represents going from about two percent, diminishing to about three and a half percent.

When they doped the nickel electrode with iron they found that the capacity changed with the number of cycles and the diminution in terms of percent went from about 12 percent to about 32 percent. These figures are evaluated from the curve which appears in the publication so it is not to be taken exactly right.

Some additional evidence concerning this was in a paper in the same symposium by Harivel and company. They doped the electrode with different ratios of iron to nickel so we go from -- The top is the smallest amount of iron, to the bottom, which is the highest amount of iron.

And in the second and third columns you have the
capacity which they measured and the percent change is in the fourth column. You will note that at low concentrations of iron, we have a greater percent change than we have as the concentration of iron in the plate increases.

In a report by Carson and company there is some little data relative to this. In this case they took the original nickel plate and after six cycles they measured the capacity. This is the capacity range. And they measured the average gassing rate which is given -- the units are given here -- against percent charge. And these are the figures which they report.

Then they impregnated this plate with ferric nitrate to about .002 percent iron and after 12 cycles they show that there is a slight decrease in the capacity. And in the lower part of the column you will note that -- let's take the more dramatic decrease, which was in the case of 90 percent state of charge. There is a diminution of 25 percent of the gassing rate; at 75 percent, there is a diminution of 19 percent, and actually at a 60 percent state of charge there is an increase of 18 percent in the system.

All of this of course suggests that iron may have a role in the activity of the nickel-cadmium battery.

Falk and Salk in 1969 in their book, Alkaline Storage Battery, reviewed some of this work and quote Stringfellow who says that the loss of capacity in the positive plate is due to iron poisoning.

They also state, and I quote:

"For a long time, iron has been recognized as a harmful impurity in the nickel-hydroxide electrode."

Later they state that the effect of iron -- or conclude that the effect of iron is a reduction of the oxidized state of the nickel oxide electrode and a reduction of the oxygen over voltage.

I set out to give some preliminary information on
whether we could detect iron in a new cell as compared to an aged cell and to see what -- just to get an analytical method for this.

I would like to first point out that in the company log of Eagle-Picher they do give some analyses for iron. For example, in the negative plaque they find that the iron percentage is .002 to .001 percent in the separator, depending upon which separator they use. All being variations of nylon, they find .002 to .004 percent.

In another analysis on three cells they give the range of iron in the nickel powder that they use for preparing the electrodes is .02 to .04 percent. And the figure for iron in the pennon separator is .003 percent.

The specification provided for Aerospace nickel-cadmium cells specifies the maximum iron content should be .01 percent which is the average assay given by the International Nickel Company.

I took two cells and analyzed using atomic adsorption the amount of iron in various parts of the cell.

(Slide 168.)

The virgin plate you see is a SART cell and the amount the analysis -- The last figure gives the percent iron I found, .02, in the positive sinter and .005 in the negative sinter.

I took what was said to be an aged plate, a Culton cell. You will find if we compare the positive sinter in the aged with the positive sinter in the virgin cell there is roughly a doubling of the amount of iron.

In the negative sinter we also find an increase of roughly three times.

In the cell that I took apart, the separator was attached to the negative electrode and I just removed the mass, which was easily removed with a nickel spatula, and I found that the percent of iron on that was .021.

There was some brownish material on the plastic
cover of the cell and the percent iron in that plastic material which you can see was only nine milligrams came to 4.3 percent.

Merely for uniformity or complete analysis, I analyzed the soluble iron in the electrolyte which came to 9.3 micrograms per liter.

The details and the references that I have mentioned previously will be found in this report.

So the indication, at least from the literature and these preliminary analyses suggests that there is a migration of iron and that there is some deleterious effect of iron on the performance of the cell.

Perhaps study is needed now to evaluate whether in truth iron does contribute and some of the factors that I would like to suggest would be, one, since the data suggests that the effect of iron increases as the concentration of the iron decreases in the nickel electrode, one would have to study the effect of concentration and determine the limits in which there is a deleterious effect.

Secondly, the position of iron in the cell. There is evidence that the migration of the iron varies with the position of the iron in the components of the cell.

Third, the effect of temperature to show the result of Troilius and Alfelt. They felt that the rate of contamination of iron did increase with temperature, and also perhaps it would affect the electrochemical capacity.

And finally, the type of purging, the sequence and the scheduling may affect the movement of the iron and its effect on the electrochemical capacity.

HALPERRY: Are there any questions for Dr. May?
HALPERT: There is a question.

GROSS: I thought what was significant was the effect of cadmium in the positive. There were very small amounts of cadmium that were shown to have a significant capacity reduction. Cadmium is sometimes placed in the positive electrode to give reversal protection.

I had always been of the impression that this -- that it was of negligible importance.

MAY: I think this slide shows the effect of cadmium on the positive.

(Slide 166.)

If you look at the figures, it is rather difficult because there are no actual figures; it is just a plot that Troilius and Alfelt get. And if you calculate by this rough measurement it is a very little-- Well, it would support your notion that it will maintain the same capacity.

GROSS: Well, it suggests to me that one should look very carefully at the use of cadmium as an ingredient in a positive electrode for reversal protection. This is now done in some cell designs.

MAY: Of course I was interested in the iron.

GROSS: Yes.

ICKFIT: I showed some data yesterday where the addition of cadmium to the positive material enhanced the performance to some extent. This was on the order of somewhere around seven percent. Other people have indicated to me that this does occur.
VI.9 CHEMICAL ANALYSIS PROCEDURE AT GSFC

MAY: If you'll be patient, there was a paper that summarized the effect of various foreign ions. This was a paper by Casey and all, The Journal of the Electrochemical Society in 1965, and they state that cadmium has no effect. This is their data which refers back to two other references. If you wish I can show you that.

HALPERT: The next speaker is Bill Webster who is going to discuss some of the results of the chemical analysis on the nickel-cadmium cells here at Goddard.

WEBSTER: This work was performed under a contract that we have, or a grant, I should say, with the Federal City College. On this program at the present time we have two graduate chemists working at Goddard, one at a 40-hour week and one at a 20-hour week, approximately.

We have in the past year come out with an analysis of about 20 cells and we thought this 'could be an opportunity to make you aware of these results.

Also, there was an X report which was put out which was a pre-print. It was very limited distribution. This was our first attempt at documenting what we were doing here.

Since then, it has gone through a dynamic evolution and we came out with another one which represented our best attempts at the end of August, which is available here and which has been distributed widely.

Let me state that this is undergoing changes and that we will come out with an addendum to this. Just as we heard a talk by Dr. Kroger of G.E., we will probably be continually evolving better and more efficient techniques.

Let me discuss first of all, before I get into the results, the addendum that will be coming out to this X report.

Let me state first of all our differences with how we are going to do things from now on. We now have three S\textsuperscript{3}XHLET extractors. Our intent in the future is to take a cell apart and upon taking it apart in an inert
atmosphere, to place the positive electrodes in one
SOXHLET, the negative in another, and the separator in
the third, so that we may get three separate aliquots
which we may analyze for amount of electrolyte and extent
of carbonate.

Also, our technique for electrolyte analysis has
been modified as a result of discussing with Jim Dunop
of COMSAT their way of doing it. I believe last year the
technique involved forcing an answer; that is to say we
assumed that the percent concentration of the electro-
lyte was what the manufacturer put in and then we would
analyze for the number of OH ions present and then calcu-
late the number of c.c.'s of electrolyte in the cell.

From now on, and this is in this procedure, we will
first do a weight determination of the cell, then an OH
determination, and then come up with a figure which gives
us the actual percent of the electrolyte at the time that
this sample is analyzed.

This is significant, from what you've been hearing
today, with respect to the role of OH ions in the reaction
with the separator. For instance, if we have cells which
have been out at Crane on test for five years, yes, they
might have started out at 31 percent KOH but it would
certainly be interesting to know what they have in them
now.

First I would like to show you a summary of the
results of the chemical analysis of some cells. These
will be representative of all three manufacturers.

(Slide 169.)

What we have here is a list of the projects from
which the cell had its origin, the capacity. These cells
all are G.E. I have another slide which shows a few
Eagle-Picher and a few ATS.

We have the test history. The term "virgin" is used
to describe simply plates. Sometimes we receive histori-
cal samples when we have cells made and that is repre-
"virgin."
"Acceptance test" means in most cases that it had received an acceptance test at the manufacturer and possibly also at the prime contractor.

And then the last category is self-explanatory, which is the actual test history. Most of these cells represent cells which have been on test at Crane.

You can see that we have analyzed or reported the results here: the total negative capacity, the total positive capacity, not what chemical species this is in. We have done this. We will be coming out with an X report in detail on our findings, probably in the spring of the year.

But just to give you a highlight, we reported these numbers.

One cell of particular interest I would think is the 1966 historical. We just used that term. It is one we have had in-house. It is being shorted. It was not built to any particular specification. It was what was coming out of G.E. in 1966. It went through acceptance test and that's all.

And you will observe that it has a 1.37 negative to positive ratio.

Also we are in the process of moving our laboratory so we had to interrupt our analysis but you see on the slide there are three nickel braised seals. We have a fourth which will be analyzed after the first of the year, and this will fall in the category of a cell which has been through acceptance test only so we will be able to get a comparison then in characteristics of carbonate contamination, electrode growth, and things of this nature versus the cells which have undergone three years' cycling.

(Slide 170.)

This is more of the same material but for Eagle-Picher and for Gulton Industries. The designation 7-P means that they are lot 7 plates; this was not a cell--it was a three ampere-hour cell that contained
approximately 10.26 ampere-hours of negative and ended up with a negative-positive ratio of 1.62.

Down under the Gulton Industries we have two ATSF cells indicated by their serial number which had been stored by different techniques. One was activated, stored shorted and wet for a period of two years. The other was stored at the manufacturer for a period of two years in argon atmosphere before being activated and going through acceptance test.

And then the top ATS there is an example of the positive plate and the negative plate; historical materials.

(Slide 171.)

Next I would like to show you the results on the same cells of our carbonate analysis. And this is by Mr. Jones-- Let me give credit where credit is due.

This work is a combination of a lot of people's energies: Floyd Ford, Gerry Halpert and myself have had inputs to the procedures, and so have quite a few people in industry who have contributed their ideas.

The actual work in the laboratory has been done by Mr. Clinton Jones of Federal City College, and Mr. Ifechi Opalui of Federal City College, also.

This is using Halpert/Jones technique as reported last year for the carbonate determination.

In the case of the nickel braise results, the percent potassium carbonate in the electrolyte, we received these cells from Crane and Crane had already performed the analysis out there so we had to input their data into our program, and these are the numbers that we came up with.

Of interest is the AE cell, atmospheric explorer. That represents lot 3, serial number 89. It had been through acceptance test. Note the extremely low nickel carbonate, cadmium carbonate, and percentage of carbonate in the electrolyte. And I understand from talking to
Gerry Halpert, the project engineer at Goddard on this program, that this cell had seen a special step at G.E. which I'm not familiar with. I don't think it is significant right now, but it is one step anyway for removing carbonate from the cells, and it seems to work.

Without going into the details, the data is furnished and is obvious there.

I'd say this is strictly a weight percent. We were talking yesterday about Dr. Scott's figures versus COMSAT's figures and so forth. This is the percent potassium carbonate by weight over the percent of electrolyte. However, I would caution one thing: This data is forced again because we assume that we had -- was it 31 percent, Gerry? -- 31 percent potassium carbonate in all the cells and so we ended up analyzing for the OH ions and then coming up with a number of c.c.'s. So we are taking our number for potassium carbonate and putting it over a c.c. value converted to grams, using a density figure of 1.3 for 31 percent potassium carbonate.

If, indeed, on some of the older cells after some cycling, the concentration is not 31 percent, then that density figure is incorrect. But this is making available-- I think it is realistic and representative of what is in the cell. And in the future, our work will be based strictly on a weight determination of electrolyte and will be a little bit more accurate.

(Slide 172.)

More data. This is Eagle-Picher and Gulton Industries, also. The term "dry" is self-explanatory. This was a strict determination on plates. They were not in a cell so there was no electrolyte to analyze.

The next area of concern or change to this document will be-- In here is reference to a technique for doing nitrate determination which we are all familiar with, which is known as the Kjeldahl technique. We don't do that.
### Chemically Determined Ni-Cd Cell Capacity & Plate Ratios

<table>
<thead>
<tr>
<th>PROJECT</th>
<th>MANUFACTURER'S CAPACITY (AH)</th>
<th>TEST HISTORY</th>
<th>NEGATIVE CAPACITY (AH)</th>
<th>POSITIVE CAPACITY (AH)</th>
<th>NEG/POS RATIO</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACCELERATED TEST</td>
<td>6</td>
<td>VIRGIN</td>
<td>18.62</td>
<td>9.46</td>
<td>1.76</td>
</tr>
<tr>
<td>Ni BRAZE SEAL (1023)</td>
<td>6</td>
<td>3 YR/20 HR, 6CP/80%</td>
<td>17.04</td>
<td>10.50</td>
<td>1.67</td>
</tr>
<tr>
<td>Ni BRAZE SEAL (1042)</td>
<td>6</td>
<td>3 YR/20 HR, 18CP/80%</td>
<td>16.36</td>
<td>9.56</td>
<td>1.77</td>
</tr>
<tr>
<td>Ni BRAZE SEAL (1048)</td>
<td>6</td>
<td>3 YR/20 HR, 20CP/80%</td>
<td>17.98</td>
<td>12.33</td>
<td>1.46</td>
</tr>
<tr>
<td>AE (240)</td>
<td>6</td>
<td>ACCEPTANCE</td>
<td>21.74</td>
<td>16.99</td>
<td>1.25</td>
</tr>
<tr>
<td>OBD-1 (1420)</td>
<td>12</td>
<td>9.5 YR/20 MIN, 10CP/80%</td>
<td>38.30</td>
<td>19.80</td>
<td>1.95</td>
</tr>
<tr>
<td>OBD-1 (2371)</td>
<td>12</td>
<td>VIRGIN</td>
<td>23.74</td>
<td>19.30</td>
<td>1.22</td>
</tr>
<tr>
<td>SYNC CRANE (924)</td>
<td>12</td>
<td>9 YR/SYNC, 6CP/80%</td>
<td>38.08</td>
<td>19.58</td>
<td>1.93</td>
</tr>
<tr>
<td>SYNC CRANE (924)</td>
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<td>ACCEPTANCE</td>
<td>29.57</td>
<td>19.09</td>
<td>1.30</td>
</tr>
<tr>
<td>HISTORICAL</td>
<td>12</td>
<td>ACCEPTANCE</td>
<td>31.74</td>
<td>23.18</td>
<td>1.37</td>
</tr>
</tbody>
</table>

**Figure 169**

### Carbonate Analysis of Nickel-Cadmium Cells

<table>
<thead>
<tr>
<th>PROJECT</th>
<th>MANUFACTURER'S CAPACITY (AH)</th>
<th>TEST HISTORY</th>
<th>% NICKLE CARBONATE IN ACTIVE MATERIAL</th>
<th>% CADMIUM CARBONATE IN ACTIVE MATERIAL</th>
<th>% POTASSIUM CARBONATE IN ELECTROLYTE</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACCELERATED TEST</td>
<td>6</td>
<td>VIRGIN</td>
<td>8</td>
<td>4</td>
<td>DRY</td>
</tr>
<tr>
<td>Ni BRAZE SEAL (1023)</td>
<td>6</td>
<td>3 YR/20 HR, 6CP/80%</td>
<td>8</td>
<td>2</td>
<td>7</td>
</tr>
<tr>
<td>Ni BRAZE SEAL (1042)</td>
<td>10</td>
<td>3 YR/20 HR, 10CP/80%</td>
<td>10</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>AE (240)</td>
<td>6</td>
<td>ACCEPTANCE</td>
<td>1</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>OBD-1 (1420)</td>
<td>12</td>
<td>9.5 YR/20 MIN, 10CP/80%</td>
<td>13</td>
<td>2</td>
<td>15</td>
</tr>
<tr>
<td>OBD-1 (2371)</td>
<td>12</td>
<td>VIRGIN</td>
<td>19</td>
<td>12</td>
<td>DRY</td>
</tr>
<tr>
<td>SYNC CRANE (924)</td>
<td>12</td>
<td>9 YR/SYNC, 6CP/80%</td>
<td>13</td>
<td>2</td>
<td>15</td>
</tr>
<tr>
<td>HISTORICAL</td>
<td>12</td>
<td>ACCEPTANCE</td>
<td>10</td>
<td>5</td>
<td>15</td>
</tr>
</tbody>
</table>

**Figure 171**

### Chemically Determined Ni-Cd Cell Capacity & Plate Ratios

<table>
<thead>
<tr>
<th>PROJECT</th>
<th>MANUFACTURER'S CAPACITY (AH)</th>
<th>TEST HISTORY</th>
<th>NEGATIVE CAPACITY (AH)</th>
<th>POSITIVE CAPACITY (AH)</th>
<th>NEG/POS RATIO</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMS (7P)</td>
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<td>VIRGIN</td>
<td>10.25</td>
<td>6.32</td>
<td>1.62</td>
</tr>
</tbody>
</table>

**Figure 170**

### Carbonate Analysis of Nickel-Cadmium Cells

<table>
<thead>
<tr>
<th>PROJECT</th>
<th>MANUFACTURER'S CAPACITY (AH)</th>
<th>TEST HISTORY</th>
<th>% NICKLE CARBONATE IN ACTIVE MATERIAL</th>
<th>% CADMIUM CARBONATE IN ACTIVE MATERIAL</th>
<th>% POTASSIUM CARBONATE IN ELECTROLYTE</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMS (7P)</td>
<td>3</td>
<td>VIRGIN</td>
<td>10</td>
<td>4</td>
<td>DRY</td>
</tr>
<tr>
<td>SEPARATOR EVAL (27)</td>
<td>6</td>
<td>VIRGIN</td>
<td>10</td>
<td>8</td>
<td>DRY</td>
</tr>
</tbody>
</table>

**Figure 172**
Mr. Halpert has procured a Taylor-Betz coulometric apparatus for doing nitrate determinations so we will be applying this technique in the future for the nitrate determination.

The next area in this report which should be discussed is the electrochemical test as opposed to chemical analysis. Mr. Ford and myself concocted a scheme which we thought would be a nice way of insuring stability in steady state data for electrochemical determination.

This is the plates in the flooded state, the negative-to-positive ratio, the amount of recharge and things like this.

We had a slight error in our original concept and this will be the cause for the addendum to our procedure, and I will show you what this error is and how it affects the results.

(Slide 173.)

The error concerns how we determine the base line capacity. We made a supposition which was incorrect. We said all right, let's say if the cell is a nominal 12 ampere-hour cell, let's charge it up for 200 percent 24 ampere-hours, the C/10 rate before we start to determine the negative-to-positive ratio.

That's not sufficient. It is fine for the positive plate as demonstrated by these results here, so we will leave the procedure as 200 percent at the C/10 rate as indicated at the bottom of the picture.

But if you will note, we have one column which gives the manufacturer's nominal capacity. Then we have what we found to be present chemically in the cell, and then we have also what we got out electrochemically by putting in 200 percent of the nominal, and the percent utilization.

There's a second step to this process. We do two things essentially. One is we determine a base line capacity which is then calculated into an equation which are mentioned in the report which is used to perform a
three-cycle step for getting the steady state negative-to-positive ratio and the ratio is determined on the third cycle.

But if you have not fully charged up the cell in the first place, then your subsequent data will not reflect what really happens and that will be obvious by this slide. When we talk about the negative electrode, then we get into trouble.

(Slide 174.)

This is an AE cell, G.E. 6 ampere-hour. It's the one we've been following right through the sequence.

The chemically determined negative plate capacity was 21.47 ampere-hours. Now we assumed that this was a 6 and you charge it up 200 percent and that would give you approximately 12 ampere-hours, yet the cell had 21 ampere-hours, so that is where the test was in error.

The base line electrochemical data for approximately 200 percent charge is shown there. We put in 13 ampere-hours. We got out 6.9, or only a 32 percent utilization.

We modified the procedure so that we charged at C/10 until we put 400 percent in, discharged, and we get a 61 percent utilization.

So we will recommend that if this procedure is followed for determining negative-to-positive ratio, that you charge at 200 percent for the positive electrode and at 400 percent for the negative electrode.

I believe that concludes the material I have.

HALPERT: Any questions?

SCOTT: I noticed that some of your chemically determined positive capacities run almost twice the rated. It seems almost like maybe even the 200 percent charge might not be quite enough for the positive.
WEBSTER: This is an area of consideration. However, we take this into consideration, if you will read the procedure, under our steady state determination. As I said, we run three cycles. When we run these three cycles we take the base line capacity and factor in 120 percent of that number.

We then put another 20 percent more in over what we originally had determined as the base line capacity.

It's not obvious. It sounds like a bunch of gibberish, but if you read the procedure you will see our sequence and our equations. And we use one to determine -- to give us a starting point because there is, as everyone knows, no relationship between the nominal capacity and what is actually the cell, so that just actually gives us the base line capacity.

Then we apply an inefficiency -- if you want to call it -- factor and we charge an additional 20 percent.

RAMPEL: You said 31 percent potassium carbonate. You meant to say 31 percent potassium hydroxide.

The other thing is in charging up the negative for this procedure, I would definitely recommend that you insert either cellophane or RAI 2291 in order to prevent the oxygen from discharging the negative plate as you are charging it.

WEBSTER: Thank you, Guy.

KLEIN: On your accounting for your analysis you said you averaged maybe ten percent nickel carbonate in the active material.

WEBSTER: That's correct.

KLEIN: This chemical capacity number, what is that based on?

WEBSTER: Okay, that's pure chemical; that is not electrochemical. That is the result of following the procedure in here for determining the amount of active material.
This is not black and it is not electrochemical. It does not take into account, let's say, utilization or electrochemical efficiencies.

KLEIN: Okay. Does that number include the nickel active material? Is this a form of carbonate? That includes the carbonate?

WEBSTER: Yes.

KLEIN: So on that basis, utilization of the non-carbonate form of the active nickel would be higher on this line?

WEBSTER: It would have to be.

Also, last year— I might like to clarify one thing -- the question came up with regard to how do we know that we have all of the carbonate out of the plates? I guess it wasn't obvious what our test sequence was.

We go through a Soxhlet extraction and of course your carbonate is soluble in hot water, and we made a study of the extract from the Soxhlet and we now recommend a 48-hour extraction to make sure that you have indeed removed all of the carbonate and all the O{sup+} from the plates.

WADHAM: How critical do you think it is that you open the cells up in an inert atmosphere before you transfer to a Soxhlet?

WEBSTER: It's like chicken soup. It won't hurt.

WADHAM: You open it up in there and then as rapidly as you can you transfer it to the Soxhlet in an inert atmosphere. You don't think it would have any degrading effect on your analysis?

WEBSTER: You would have to do a quality control study on it I would think.

HALPERT: Can I clarify that for one moment?
The plates are packed in a plastic plyofilm bag in the chamber and moved to the SOXHLET and while there is nitrogen passing through the SOXHLET, the bag is opened and the plate is dropped in and immediately sealed so we minimize to any extent the amount of--

WADHAM: I was wondering how critical it would be if you got air into the plates.

WEBSTER: How much carbon monoxide would you absorb as a result of having the sinter structure pass through the air is the question briefly. I don't know.

SCOTT: As I mentioned yesterday, we took a look at that and found that within a period of several minutes at least, there was negligible effect on the plates but there was a definite effect on separators. Since it takes some time to take a cell apart and to remove the separator, I would think that within a period of a few minutes you would see a significant error in carbonate pickup in the separators.

"We found like five to ten percent error within five minutes' exposure of separators to the air."

WEBSTER: Years ago at Goddard, an experiment was run in which sintered plates were exposed to the air and a solution of KOH was exposed to the air, and the carbonate contamination of the sintered plates was quite high. The pickup by electrolytes sitting in the beaker was very low because it is a surface reaction and there wasn't much surface area exposed.

WADHAM: I was more concerned with what effect it might have on the cadmium, whether that might get converted to cadmium oxide or cadmium hydroxide during that time.

WEBSTER: Well, in our procedure, that opportunity is not afforded because we are in an inert atmosphere.

FORD: I would like to point out for those who haven't already made the connection between the left-hand title there on all these Vugraphs versus some of the previous discussions we've had, the nickel braided cells
are the ones I discussed about the 24-orbit test results. OSO cells are on test at Crane. Practically every one of these cells is on life test at Crane, so we are making an attempt to correlate the electrical performance with the electrochemical analysis that we're getting.

THORNELL: On your three-capacity cycle have you recorded capacity for positive and negative?

WEBSTER: Yes, we do. What we use is a three-electrode sample, three positive electrodes, three negatives, and we record the ampere-hours in, ampere-hours out at the end of each cycle. And we are doing a statistical analysis to see if it is really necessary.

This is a very creative and dynamic program and it changes as we go along and as we've learned, and on those cells where we had sufficient charge to take care of the material that was actually there, we would find that the variation between cycle one and cycle three was on the order of one percent drift from cycle to cycle.

THORNELL: Another thing on your separation, keep away from collophane. It might cloud the capacity on the calcium; it's sort of beneficial.

WEBSTER: Right now we're using— Clint, what are we using in there?

The simulator we use between the positive and the negative when we're going to flood and electrochemical test, it looks like a PVC grid material, something like this that is being used at the present time.

HALPERT: These plates are run flooded and they do have a separation which is KOH inert. I'm not exactly sure, I think Nexar, but I'm not certain about that.

Thank you, Bill.

WEBSTER: Excuse me. There is one last thing.

I asked the fellows should I show any plate thicknesses and they said, "Oh, no, of course not. No one is interested in that data."
Then of course Jim Dunlop of COMSAT said gee, he'd like to see some so I hurried up and made some this morning. Let me show it.

(Slide 175.)

I think the most significant thing that you can see up there right now is-- This is an average value and no attempt was made at statistical analysis or variation, and things like this will be done in our X report when it comes out on this subject, as to where the samples were made and what the plus or minus is.

But I find it interesting to look at the 1966 historical cell from General Electric and see that the positive plate was 42 mils thick, and this is an uncycled cell.

Also, note that we do have some growth. For instance, the OSO-I cell 2-37 had an initial positive plate thickness of 28-1/2 mils and the cell 1-400 shows a thickness of 30 mils, and those cells experienced approximately 900 cycles, and there's a couple of mil growth in the positive.

HALPERT: Our next speaker is also going to talk about nickel-cadmium cell analysis, Ed Stofel.
VI.10 CELL ANALYSIS: STOFL/HUGHES

STOFL: Hughes started in on a cell analysis of the type we've been listening to about four years ago. It started pretty much as a hobby of Bob Steinhauer's and Al Heller's. It then was picked up on IR&D and carried that way for a while, and about the last year or year and a half, we've been getting most of our money from program offices and to that extent, maybe we are doing things a little bit different than what some of the other people have stated here.

We live now in a situation where we're trying to save time and money and meet schedules and cell analysis is here as far as we at Hughes are concerned. Maybe if you're doing 20 cells here at Goddard you may be in the same position, but we get asked questions by program managers who know very little or nothing about batteries: what have you done with my money?
## EVALUATION OF POSITIVE PLATE ELECTROCHEMICAL TEST PROCEDURE

<table>
<thead>
<tr>
<th>PROJECT</th>
<th>MANUFACTURER'S CAPACITY (A)</th>
<th>CHEMICAL CAPACITY (A)</th>
<th>ELECTROCHEMICAL CAPACITY (A)</th>
<th>PERCENT UTILIZATION</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMS (171)</td>
<td>3 EP</td>
<td>6.32</td>
<td>4.58</td>
<td>72</td>
</tr>
<tr>
<td>ATS - F (571)</td>
<td>15 GI</td>
<td>26.73</td>
<td>18.18</td>
<td>69</td>
</tr>
<tr>
<td>ATS - F (1721)</td>
<td>15 GI</td>
<td>28.34</td>
<td>21.16</td>
<td>75</td>
</tr>
<tr>
<td>ACCELERATED TEST</td>
<td>8 GE</td>
<td>9.68</td>
<td>7.08</td>
<td>75</td>
</tr>
<tr>
<td>Ni BRAZE (5231)</td>
<td>8 GE</td>
<td>10.60</td>
<td>7.21</td>
<td>69</td>
</tr>
<tr>
<td>Ni BRAZE (5232)</td>
<td>8 GE</td>
<td>9.56</td>
<td>7.10</td>
<td>74</td>
</tr>
<tr>
<td>SYNC CRANE (5040)</td>
<td>12 GE</td>
<td>19.96</td>
<td>14.95</td>
<td>78</td>
</tr>
<tr>
<td>O3O - 1 (I - 400)</td>
<td>12 GE</td>
<td>19.90</td>
<td>15.18</td>
<td>78</td>
</tr>
</tbody>
</table>

* BASELINE ELECTROCHEMICAL 1-200% CHARGE

**FIGURE 173**

## EVALUATION OF NEGATIVE PLATE ELECTROCHEMICAL TEST PROCEDURE

### I. BACKGROUND:
- PROJECT AE
- G.E. 6 Ah
- SN 3 - 89

### II. CHEMICALLY DETERMINED NEGATIVE PLATE CAPACITY:
21.47 Ah

### III. BASELINE ELECTROCHEMICAL (~200%) CHARGE:
- Ah IN: 12.00
- Ah OUT: 8.90
- % UTILIZATION: 32%

### IV. BASELINE ELECTROCHEMICAL (~400%) CHARGE:
- Ah IN: 24.96
- Ah OUT: 12.98
- % UTILIZATION: 61%

**FIGURE 174**

### AVERAGE PLATE THICKNESS

<table>
<thead>
<tr>
<th>PROJECT</th>
<th>NEGATIVE</th>
<th>POSITIVE</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACCELERATED TEST</td>
<td>32.40 mils</td>
<td>31.40 mils</td>
</tr>
<tr>
<td>Ni BRAZE SEAL (5231)</td>
<td>39.72</td>
<td>37.95</td>
</tr>
<tr>
<td>Ni BRAZE SEAL (5232)</td>
<td>33.83</td>
<td>32.94</td>
</tr>
<tr>
<td>Ni BRAZE SEAL (5231)</td>
<td>35.43</td>
<td>34.08</td>
</tr>
<tr>
<td>A E (5-48)</td>
<td>37.01</td>
<td>30.40</td>
</tr>
<tr>
<td>O3O - 1 (5-420)</td>
<td>34.53</td>
<td>30.11</td>
</tr>
<tr>
<td>O3O - 1 (6-53)</td>
<td>33.40</td>
<td>28.50</td>
</tr>
<tr>
<td>SYNC CRANE (5040)</td>
<td>30.02</td>
<td>24.54</td>
</tr>
<tr>
<td>SYNC CRANE (5232)</td>
<td>28.51</td>
<td>24.87</td>
</tr>
</tbody>
</table>

HISTORICAL: 35.43  42.38

<table>
<thead>
<tr>
<th>PROJECT</th>
<th>NEGATIVE</th>
<th>POSITIVE</th>
</tr>
</thead>
<tbody>
<tr>
<td>SMS (11)</td>
<td>40.47</td>
<td>39.80</td>
</tr>
<tr>
<td>ATS - F (571)</td>
<td>37.20</td>
<td>31.20</td>
</tr>
<tr>
<td>ATS - F (572)</td>
<td>38.54</td>
<td>38.34</td>
</tr>
<tr>
<td>ATS - F (572)</td>
<td>37.07</td>
<td>39.01</td>
</tr>
</tbody>
</table>

**FIGURE 175**
And what it really amounts to is often we have to tell him or we should tell him -- we are not always that frank but we should tell him that we raise more questions than we have answered.

I wanted to point that out because I think that some other companies are moving in the direction of doing more cell analysis of an analytical nature, not failure analysis. I think a lot of people have been doing that for years. But this business of trying to use cell analysis to try to learn something, as you get into it and you start getting funding from where the money really is in the program offices, watch out.

(Slide 176.)

The first thing to notice about this slide, as a matter of fact, is not even the words and the fact that it is colored and it has the standard Hughes format. We at Hughes have standardized on a lot of things, not on battery cells yet, but we have on Vugraphs. And the justification for this is that we can take the standard format and reshuffle them and have another presentation tomorrow on a slightly different subject and use these Vugraphs all over again.

I want to point this out in particular because some of the Vugraphs you'll see, as well as some other ones, are the same ones that go to program management and try to tell them what they're getting for their dollars spent on cell analysis.

Getting a little bit more into detail, what we're attempting to do first of all is better prediction of life. We believe that there is money to be made or saved, however you want to look at it, if we can get longer-lived batteries.
WE have yet to know of a situation of any spacecraft that has failed because of a wear-out degradation mode of the batteries. There have been some battery problems on spacecraft, and I am not talking just about Hughes; I'm talking about generally. They have been problems of inconvenience or problems like, I believe, the OAO or one of the spacecraft had a thermal runaway or something like that.

But in terms of wear-out modes we have yet to know of a situation where this actually happened, but we worry about it.

In general, besides predicting life, we also are looking for those failure modes. The reason we're looking for them is hopefully some day to come up with a better way of designing satellites or at least specifying them. I think the cell vendors are probably as well aware as we are that a lot of things in our ni-cad cell specifications today, we don't really know why they're in there. They're like chicken soup that was mentioned a few minutes ago; it doesn't seem to hurt until you get down to asking the cell manufacturer whether he really reads the fine print over and over again until he memorizes it, or whether he has time to do that.

We would like to simplify our specifications. It would be ideal. If it means have a more complex specification, however, to get long life, that's the way it will have to be.

Our approach is very much like you have been hearing. We examine the cells with a whole series of tests. We write up a report, typically per cell, aimed at the particular program office involved.

We also, as our own little battery section task, compile this and try to compare notes. We have some programs that are synchronous obviously at Hughes; we also have some that are not synchronous. We have polypropylene committed to flight on some programs. We have a test, for example, where we have an equal number of polypropylene and nylon separator-type cells running at 90 degrees after they've been running for about like two years.
We've pulled cells new. We've pulled a pair of cells off after 500, approximately, cycles; after approximately 1,000; after approximately 15,000; after 2,000. We're about ready to pull some more, so we can get some time history from that.

We've been looking at some of the cells from the Intelsat-4 program that Stan Krause reported on, the electrical performance here last year, and so forth. That's the type of data that we are looking at.

(Slide 177.)

Our procedure looks like this, as an overview. We claim very little originality in terms of techniques. We have borrowed or stolen wherever we can. I think due acknowledgement should go certainly to Battelle, obviously to NASA/Goddard, to COMSAT certainly, and G.F. has been very helpful in giving us some ideas, too, on some of our procedures.

Those are the principal ones but we have also asked other people for help.

The way this box is spread out, this flow diagram, it is one that happens to fit the procedures the way we do them at Hughes. I'm not saying it is the way everything has to be done. It just is taking the over-all things that have been talked about here at NASA/Goddard and putting them into the Hughes environment and using the people available.

We consider this first box very important. We have the impression that some people neglect that. We have found the cell manufacturers very helpful; once they understand why we want to bother them by getting all these data, they are generally very helpful in getting this data.

Our representatives at the manufacturers hopefully spend most of their time and don't interfere too much with the production people, but we have a standard form which is about three pages long, a fill-in quiz, so to speak, per cell, giving the detailed history and why we're going to be doing this particular analysis.
Physical measurements is fairly simple. We measure the weight compared to the weight at which it left the cell vendor. We measure the thickness, both in the restrained condition and in the unrestrained condition because we're going to use that later on in order to calculate the thickness of the separator, an item which we feel is rather important from some of the results we've seen.

We also look for potential electrolyte leakage at this point.

Electrical normalization: This I suspect is a controversial area. Our present standard procedures -- and like Bill mentioned, I'm afraid our standard procedures are a growing thing but we have to tell our program managers we're doing things by standard procedures or they tear their hair out.

But essentially our standard procedures include six cycles to one volt as well as a full discharge reconditioning. If we don't do this we can see definite effects of near-term history when we open up the cell. And the part that is controversial is should you open up the cell as is, and attempt to make some sense out of that, or should you give it a reconditioning, or whatever you want to call it, a normalization procedure, and do it on that basis?

The first approach of opening up the cell immediately gives you a hope of catching the short-term transients, that effects that are in the cell that can easily be wiped out by reconditioning and so forth.

The other approach gives you a more basic or fundamental viewpoint of what the somewhat longer-term effects are, what we've been calling longer-term effects. We have some reason to be suspicious that even the longer-term effects are not so long-term.

We talk about putting a cell through an acceptance test at maybe 30, 40, 50 cycles because it takes that long to equilibrate. And then we take an old cell that has been cycling maybe 2,000 cycles and we give it six cycles
cycles and we say well, we've put that back into an equi-
librated position. And I suspect we have not really.

I am not telling most of you anything new when I
point out that the battery is a very dynamic situation.
I do tell our program managers that a typical spacecraft
like an L-SAT-4 which is -- we have some larger and some
smaller-- during maximum eclipse that evening in the fall,
in the winter, has about six pounds of material up there
that are going through chemical reactions and we're ex-
pecting that to stay in one place and it doesn't, parti-
cularly; it migrates around and does things like this.
It's a very dynamic situation.

I think we have to be much concerned when we compare
notes with each other what we have done here. (Indicating.)

In terms of measuring the positive capacity starred
and the pre-charge we follow what we believe is still the
COMSAT procedure, at least what they had in the Power
Sources Conference a few years ago, and published. We
have some questions on that but generally we are getting
reproducible results off of that, but it depends some-
what on what you're doing here. (Indicating Box No. 3.)

With regard to cell disassembly, we do it under the
nitrogen environment, a glove box. We keep the glove box
about 70 to 90 percent humid so we don't get too much
evaporation. We do pull the cell apart at this particu-
lar time, separating it out into positive plates in one
package, negative plates in another, and separators in the
third. And in fact we have an allocation that is a little
bit more complicated than that, but that's basically what
it is.

So when we come down here to the SOXHLET, we've been
doing for quite a period of time separate SOXHLET's on
negatives, positives, and the separators. We take un-
SOXHLET-ed plates, two each on the negatives and two each
on the positives, and do the electrochemical cycling on
that.

We support our analysis down here by atomic adsorp-
tion. Generally it is pretty good. I caution you, though,
if you are not doing the work yourself, as we personally don't -- we do it in other parts of Hughes, or send it out -- be sure you send controls along because we've had some erroneous results.

We have also done it by EDTA and find good results that way, too.

We do emission spec generally. On an optional basis we do scanning electron microscopy. We do the NO\textsubscript{x} analysis and we do a separator weight analysis.

(Slide 178.)

As a general detail on the chemical and physical I want to point out a couple of items. We take a six-pack of negatives. That's what goes into our SOXHLET. We weigh it with electrolyte. We extract it. We then weigh it soaked with as much water as possible, squeegee it off to make it reproduceable; we vacuum dry it and weight it again. And out of that we get not only the total weight but the electrolyte that was in there. We have a pretty good indication of what the void volume is. We have a more elegant way of doing void volume with vacuum impropagation and that sort of stuff and using a chlorinated diaine. But on a routine basis, trying to do things cheap, we get a good first approximation just using water.

We go through fairly standard analysis. I think you've heard enough about that. I won't take time to go into detail.

Routinely, we do, however, do check for potassium as well as the OH and carbonate by titration. We happen to use a pH meter rather than any other indicator. We also now are checking potassium so that our bookkeeping all comes out. We're doing basically double-entry bookkeeping here.

(Slide 179.)

We get results that are pretty much like what has been reported but I want to point out that when you start looking at a variety of cells you do see things that raise questions, as I pointed out.
Ni-Cd ENERGY STORAGE ANALYSIS

PURPOSES
- PROVIDE BETTER MEANS OF PREDICTING BATTERY PERFORMANCE OVER LONG LIFE
- PROVIDE MEANS OF DETECTING FAILURE MODES
- PROVIDE QUANTITATIVE BASIS FOR FUTURE IMPROVEMENTS TO Ni-Cd DESIGN

APPROACH
- EXAMINE CELLS WITH SERIES OF TESTS (ELECTRICAL, CHEMICAL, ELECTROCHEMICAL, METALLURGICAL) SUFFICIENTLY PRECISE TO DETECT SMALL DEVIATIONS FROM NORMAL CELL BEHAVIOR
- COMPILe INFORMATION FROM CELL EXAMINATIONS AS FUNCTION OF CELL LIFE TO DETECT WEAROUT MODES AND CAUSES OF CATASTROPHIC FAILURES

Ni-Cd CELL ANALYSIS STANDARD PROCEDURE DIAGRAM

PROCEDURE FOR PHYSICAL AND CHEMICAL ANALYSIS OF NEGATIVE ELECTRODES

ELECTROCHEMICAL DISCHARGE OF NEGATIVE ELECTRODE WITHOUT CELL RECONDITIONING
This is pretty much what we see in a discharged reversal out. We measure the amount of hydrogen. It is only hydrogen coming off. We use a gas chromatograph.

We have been-- This is a real cell trace; this is another real cell trace. They both came from the same program. They did have different-- They were essentially the same cells, as far as I know, when they left the manufacturer but they did have different cycling histories.

In this one it gives only indication of being negative limited. There was no gas coming out whatsoever, no bubbles, no nothing.

On this one here we had a situation where this was all hydrogen gas. It was only at this point, toward the end, that we begin to get an indication of any oxygen coming out at all.

I just point out that here are two cells that give somewhat different results. We're not sure yet what it all means but it does occur. We have to be alert to that type of thing.

(Slice 180.)

Another thing, we talk about the electrochemical capacity of the plates. We do use a membrane, incidentally, in terms of getting our measurements.

The positive we find fairly reproducible; the negative is dependent upon several things, depending upon whether you have a saturated or non-saturated KOH solution with cadmium hydroxide or not. As you cycle these things more than one time in a beaker, the capacity moves in. But we also see in some cases, a step-- Font talked a little bit more about this last year at this conference, what that step might mean.

But the point that we want to point out is that as we cycle it a couple of times, it goes from this to that condition. What does that mean? What value should we plug into our life predictions in terms of using what we would say to some extent is a conventional approach in
life prediction of saying what's the utilization of all this?

(Slide 181.)

This is pretty straightforward. Incidentally, in both the positives and negatives when we do our cycling in a beaker we do it under a nitrogen atmosphere. Bob Steinhauer reported this at the Electric Chem Society a few years ago.

This is pretty straightforward. The available pre-charge we feel is very sensitive to the immediate past history of the cell and so is the unavailable precharge, the ratio of these, the amount that you pick out, one or the other. It is also dependent upon the rate that depends upon some crystal structure items I will touch on briefly.

This is obtained by subtracting one number from another so it really depends upon what electrochemical measurement we get. And in that Vugraph I just pointed out before with the double plateau, which plateau should we take as the end point?

We've gotten into some discussions of the physical chemistry significance of the two, and so it goes.

Sometimes we of course find cadmium out on the separator and elsewhere, and so that is a way of accounting for everything.

(Slide 182.)

Here is a familiar looking Vugraph. I didn't know it was going to look all that familiar. But when you open up a cell on the East Coast it looks pretty much sometimes like when you open up a cell on the West Coast.

This scale is wrong. I will correct it before I turn the notes in. That should be a tenth of a millimeter. I got carried away with the zero and since these Vugraphs were made up while I was already on the road I didn't get a chance to catch it.
But it's about the same size crystals that I think I picked off of Van Ommering's Vugraph.

When I say "top view," this is the view of the separator when you look at it microscopically on the general area, the surface area of the electrode. If you take the electrode which we routinely do, and break it like so (demonstrating), that leaves you a broken surface exposed and it is undeformed other than indention, and it is a pretty weak indention.

You look at it in cross-section, so now you're looking at what had been the visible surface before. It is just represented by that line, and this is the broken surface.

The same is true here and if you see that this is submicron-sized particles, this all down in here is submicron, but here you have the 20, 30, 40 micron diameter boulders right at the surface and only at the surface.

If you come in with an EDAX and sit here for a while and take an X-ray scan of your nickel-cadmium ratio here, here and here, you see that the cadmium has gone down here.

I will not truly make a prediction that this is good or bad to the surface but once this layer is built up we won't see it changing all that much with time. It is not necessarily bad.

I do want to make sure that all of us who are using SEM recognize that this is a very powerful tool. I've used SEM on a lot of things for six or seven years now, I guess, ever since it came out, and I've seen the reaction, what these beautiful pictures do to the program manager who doesn't really know what they mean.

And I hope Van Ommering will take this as advice, but be very careful when you say this is terrible, because it is not necessarily terrible.

I was telling Jim a little bit of my experience many years ago when we were looking at integrated circuits and all hell would break loose at what was really very normal devices, just because microscopically they looked
more-- It's a situation of looking at the playmate of the month now with a microscope and finding out she has skin blemishes. You know, suddenly she is terrible where yesterday she was beautiful.

So be careful on that or we're going to be chasing an awful lot of false information.

(Slide 183.)

With regard to your cadmium migration generally I just want to point to this as representative. It is certainly not the only way we see things.

This is a positive plate. This is a separator that was next to the positive plate. It is much darker on the other side. That had a perfectly good charge retention or did not have a short.

I might also point out that -- not microscopically, it would be on a fine scale -- the dimples in the positive plate are reproduced. Here they are reproduced as light material. On other cases we have seen then where the dimples were reproduced on dark material with the space in between being light.

We think, as I said before, that the amount of pressure on the separator is very important, or the amount it is compressed, however you want to look at it.

I might point out also that there's a band here that has less migration. There's been discussion about whether things are sticking up down at the bottom more than they do at the top. This we have found on some cells but it is certainly not universal. In fact, we have found it reversed on some cells.

I suspect that pressure might be more important than gravity. The dryness of the cell probably plays a role in all this.

(Slide 184.)

Where we have been what I would say successful in answering pretty much questions to the program manager's
VARIATIONS OBSERVED IN PRECHARGE MEASUREMENTS FOLLOWING COMPLETE DISCHARGE

FIGURE 180

CADMIUM DISTRIBUTION WITHIN Ni-Cd CELL

FIGURE 181

CHARGE MANAGEMENT EFFECTS ON Cd(OH)$_2$ CRYSTAL SIZE

FIGURE 182

NYLON WRAPPER AND NICKEL ELECTRODE

FIGURE 183
satisfaction is when we have had a problem. Here's an example of a problem area.

We had a cell. This particular cell was one that passed all of our usual electrical leakage tests, C/10 for five minutes, holding up the voltage and all that sort of stuff, and we did not find anything wrong with it until we were monitoring the voltage on the shake test which we normally do. I suspect many of the rest of you people do also.

And we saw a glitch in the voltage and we went back and tried to reproduce it, and when all was said and done we pulled the cell apart, being very careful to find out where this possible short might be. We have techniques for that but I won't take too much time since I'm supposed to hurry.

But over-all, what I'm saying is apparently from the manufacturing this one out of a hundred or one out of 200 and so forth occasionally slipped by. It had some material that got compressed on its surface and under vibration apparently it was being able to just barely force its way through the separator.

This is a very satisfying thing to show to a program manager, and then he comes along and says, "Does that mean we should reject the cell?"

Well, this sort of thing happens very infrequently. We have one of our statisticians kind of looking at this type of thing as a hobby, but he really doesn't know what to do with it. And if anybody has got some ideas I'd be happy to talk to them at the coffee break because it is the type of thing that the program manager wants to do. He wants to get the Quality people in right away.

I think what it might tell us as battery specialists is that some of these shake, rattle and roll tests that we do are probably worthwhile to keep on eye on when they do pick up things like this and say, "Aha, maybe it is successful."

I see some of my customers here staring me in the
face and they are probably wondering, "Gee, how many get
by without picking them," but I don't frankly know. I
may not be as reassuring as you'd like but we're trying.

(Slide 185.)

Here's another situation, a little different one.
This one did not pass charge retention. It just barely
didn't pass charge retention-- Excuse me. I'd better
be careful. It just barely did not pass the short test.
And looking at it very carefully, meaning that we pried
things apart one plate at a time, we realized that the
voltage we were monitoring on the cell, the cell package,
changed and we knew that we had the right plate.

We looked at it with the SEM and found a particle
in there and did an X-ray scan and found copper in there.
It was in impurity that had somehow or other gotten onto
this one plate somewhere in the manufacturing process.
We did talk to the manufacturer about it. We expressed
concern and hopefully has cleared up the matter and it
won't happen again.

So in conclusion I would like to say that we at
Hughes take the cell analysis very seriously. We appreciate
all the work that has been done at the various
agencies or COMSAT where the techniques have been de-
veloped. We mostly are a user in that case but generally
we're kind of pleased.

The only problem is that we often raise more ques-
tions to our management than we are able to answer.

WADIHAM: In your reconditioning procedure before
you test the cells, you say to about six cycles to one
volt. Now on most of the current satellite designs
this is an impractical thing to do on the spacecraft.

How can you justify examining the cell which is
being reconditioned in a way that it is not possible for
the cell to see that sort of reconditioning on the space-
craft? Surely we're interested in seeing what the cell
looks like under realistic conditions, what would be
DETECTION OF COPPER IN PARTICLE IMBEDDED IN NYLON SEPARATOR OF Ni-Cd CELL

IMPURITY PARTICLE IMBEDDED IN NYLON SEPARATOR

COPPER K$_\alpha$ SCAN OF IMPURITY PARTICLE

FIGURE 184

DAMAGED CORNER OF POSITIVE ELECTRODE

CORNER VIEW

EDGE VIEW

FIGURE 185

258A
happening on the spacecraft.

STOFEL: That's a good question. First of all, there is some question in my mind whether or not one can discharge a battery to one volt safely in a spacecraft. I don't think that's impossible by any means, but more importantly, what does all this analysis mean? What does a chart like this mean? Where do those numbers come from?

(Slide 181.)

The precharge is measured after we have completely discharged the cell. We don't do that intentionally in the spacecraft so that's already-- To get this kind of number we have done something to the cell that we don't do in space anyhow. And this is why I say it's controversial.

What does the physical chemistry of what's happening-- Why do we want precharge in there is the first place? The fact that we can change precharge so readily by just going to deeper cycles like this I think are questions that I can't answer at this time.

But with regard to whether we should be doing it -- quote -- like we do on a spacecraft -- unquote -- we don't pull cells apart in the spacecraft. There is no way that we can destructively analyze these cells and be able to say that it is like we do in a spacecraft.

Now among things that I did not mention, for example, we're very much interested in non-destructive testing. The comments yesterday with regard to looking for state of charge using the AC impedance basically is what amounts to a phase shift. You look at phase angle instead. But we're going back and looking at that fairly seriously, not as a state of charge indicator but as an indication -- as a diagnostic tool.

We've just about completed all the mechanical details, O-ring seals and things like this, that we can take a standard battery that is normally ready to fly in space and puncture a hole inside of our nitrogen chamber, put a reference electrode down in there and help settle some of the questions that are raised by this
by this pre-charge measure; things like this.

But there is no way that I know of, or the techniques that people generally are using, that you can say, "Gee, I want to do it like we do it on the spacecraft."

Your point is well taken and I've heard it very many times from our program management people, and we go round and round and round on that.

WADHAM: I don't think the point about measuring available pre-charge on the discharge cycle-- Obviously you have to discharge the cell to do this. I don't think that point is relevant because in the worst case, what you're interested in in the spacecraft is when is your cell going to go into reversal; what happens when it goes into reversal? And this is the point when one cell goes through that discharge cycle.

Another thing is I've given stand curves which show quite distinctly on all cells that you keep on discharging one volt or a half a volt a number of times, and it is well-known and it has been discussed here before that if you keep on discharging the one volt, each time you discharge to one volt you get a little bit of improvement but that first discharge curve is for real; the first one, when that cell maybe goes first into reversal, that is a real curve. It's not an artificial one.

STOFFEL: You know you are welcome for you and I to discuss this at any length. I think I am being pressed here though is what it amounts to.

HALPERT: I am going to turn the program over to Tom for his session on new developments.
VII NEW DEVELOPMENTS IN BATTERIES: T. HENNIGAN/CHAIRMAN

HENNIGAN: We have a special trust here this afternoon, Bill Harsch, who is going to talk on three things all at the same time, 21-hour per pound ni-cad, nickel-hydrogen development, and electrochemical impregnation, in particular the Bell process.

HARSCH: I'm going to be rather brief this afternoon. I have to catch a plane, and if you miss the only one to Joplin, there isn't another one for a month.

VII.1 20 WATT HOUR/POUND NICKEL CADMIUM CELL: HARSCH/E-P

I want to start out with our recent developments in nickel-cadmium, that is, the light-weight, 20-watt-hour per pound nickel-cadmium. The development program was done with Philco-Ford Corporation, the cells that we are building and have built for use on the NATO-3 satellite.

(Slide 186.) Briefly, this is the cell configuration. It is 2.97 inches wide, .88 inches thick, and 4.5 inches to the top of the cell case with an over-all height of 5.0.

(Slide 187.)

This is a summary of the design details or cell characteristics.

It has a nominal positive capacity of 23 ampere-hours and a nominal negative capacity of 38 ampere-hours.

We have a required ratio of 1.35 to one, and the nominal ratios on the flight cells are 1.6 to one. Operating pressure is a maximum of 75, and so on and so forth. The cell weight, the nominal cell weight is 583 grams, which corresponds to 23 watt hours per pound.

(Slide 188.)
**FIGURE 186**

**FIGURE 187**

<table>
<thead>
<tr>
<th>Item</th>
<th>Item</th>
<th>WEIGHT</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. POSITIVE CAPACITY (NOMINAL)</td>
<td>23 A.M.</td>
<td>216 GMS</td>
</tr>
<tr>
<td>2. REQUIRED POSITIVE CAPACITY</td>
<td>21 A.M.</td>
<td>216 GMS</td>
</tr>
<tr>
<td>3. NEGATIVE CAPACITY (NOMINAL)</td>
<td>38 A.M.</td>
<td>74 GMS</td>
</tr>
<tr>
<td>4. REQUIRED NEGATIVE CAPACITY (1.35 RATIO)</td>
<td>32 A.M.</td>
<td>74 GMS</td>
</tr>
<tr>
<td>5. NEGATIVE/POSITIVE RATIO (NOMINAL)</td>
<td>1.60:1</td>
<td>50 GMS</td>
</tr>
<tr>
<td>6. REQUIRED MINIMUM RATIO</td>
<td>1.35:1</td>
<td></td>
</tr>
<tr>
<td>7. OPERATING PRESSURE</td>
<td>LESS THAN 75 PSIG</td>
<td>15 GMS</td>
</tr>
<tr>
<td>8. MAXIMUM ALLOWABLE PRESSURE</td>
<td>75 PSIG</td>
<td></td>
</tr>
<tr>
<td>9. OPERATING TEMPERATURE (CAPABILITY)</td>
<td>10-100 DEGREES F</td>
<td></td>
</tr>
<tr>
<td>10. REQUIRED OPERATION TEMPERATURE</td>
<td>10-80 DEGREES F</td>
<td></td>
</tr>
<tr>
<td>11. CELL WEIGHT (NOMINAL)</td>
<td>105 GMS</td>
<td></td>
</tr>
<tr>
<td>12. ENERGY DENSITY</td>
<td>15 WH/LBS</td>
<td></td>
</tr>
</tbody>
</table>

**FIGURE 188**
Again, here's a summary of the weight figures. The positive weight is 210, the negative 216. Electrolyte, 74. Cell case, 58. Cover, 16. Separators, nine, which is 583 grams or 1.28 pounds.

(Slide 189A, B, C, D)

I have some characteristic data at the various temperatures, namely, 30, 40, 70, and 80 degrees F. Rather than go through those, I think I'll just leave the Vugraph and if you're interested you can read it in the minutes.

These cells are qualified now. We've run through the qualification tests. We have built the first few lots of flight cells that have been delivered to Philco-Ford.

Philco-Ford is taking these cells and packaging them in a 20-cell battery.

(Slide 190.)

This has a packaging weight of five percent and a total battery weight of 26.87 pounds, which corresponds to, I believe, about 21-1/2 or 22 watt hours per pound.

Okay, I'm ready to change subjects.

Any questions on this particular matter?

COHN: What sort of cycle life are you driving at?

HARSCH: Do you want to answer that question?

HAAS: Haas, Philco-Ford.

A seven-year sequence orbit. We are currently running life tests. In about a year we will have the back-to-back eclipse season data for about seven years.

COHN: And what is your maximum depth of discharge?

HAAS: The maximum as directed by the customer is 0 percent. It's a military-type satellite.
### 16°F. PERFORMANCE TEST

**PROCEDURE:**
Charge 50 hours, rate 0.75 amperes/discharge to 1.28 volts, rate 1.2 amps.

**REQUIREMENTS:**

<table>
<thead>
<tr>
<th>Capacity (min.)</th>
<th>16 AM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voltage (max. on charge)</td>
<td>1.520 VOLTS</td>
</tr>
<tr>
<td>Pressure (max.)</td>
<td>75 PSIG</td>
</tr>
</tbody>
</table>

**Performance:** 95% acceptance level

<table>
<thead>
<tr>
<th>Capacity (nominal)</th>
<th>22.9 AM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low voltage</td>
<td>21.9 AMP</td>
</tr>
<tr>
<td>High voltage</td>
<td>23.9 AMP</td>
</tr>
<tr>
<td>Pressure (nominal)</td>
<td>69 PSIG</td>
</tr>
<tr>
<td>Low pressure</td>
<td>63 PSIG</td>
</tr>
<tr>
<td>High pressure</td>
<td>88 PSIG</td>
</tr>
</tbody>
</table>

**FIGURE 189A**

### 20°F. PERFORMANCE TEST

**PROCEDURE:**
Charge 50 hours, rate 1.5 amperes/discharge to 1.28 volts, rate 1.9 amps.

**REQUIREMENTS:**

<table>
<thead>
<tr>
<th>Capacity (min.)</th>
<th>22 AM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voltage (max. on charge)</td>
<td>1.460 VOLTS</td>
</tr>
<tr>
<td>Pressure (max.)</td>
<td>75 PSIG</td>
</tr>
</tbody>
</table>

**Performance:** 95% acceptance level

<table>
<thead>
<tr>
<th>Capacity (nominal)</th>
<th>29.7 AM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low voltage</td>
<td>22.8 AMP</td>
</tr>
<tr>
<td>High voltage</td>
<td>31.5 AMP</td>
</tr>
<tr>
<td>Pressure (nominal)</td>
<td>64 PSIG</td>
</tr>
<tr>
<td>Low pressure</td>
<td>65 PSIG</td>
</tr>
<tr>
<td>High pressure</td>
<td>75 PSIG</td>
</tr>
</tbody>
</table>

**FIGURE 189B**

---

262A

*ORIGINAL PAGE IS OF POOR QUALITY*
HARSCH: I'm going to talk about the continuous electrochemical process.

The process itself is the Bell process that has been described before at this meeting, and at some other meetings around the country so I will not go into the actual chemistry of the process. I believe everybody knows how it operates.

In particular, what I would like to show is how you take this process and put it into a continuous process and manufacture cells and batteries.

Western Electric has awarded us a contract to set up the electrochemical processing equipment and to manufacture batteries for use in the Bell Telephone system. The battery that we are presently building at AP is a five-cell, 34 ampere-hours sealed nickel-cadmium.

May I have the first slide?

(Slide 191.)

The process starts, of course, with the making of the raw sinter material. This slide here shows the sintering furnace and the slurry technique used in the continuous process.

As the plaque comes out of the furnace it is rolled and then run through our coining operation which coins the specific size plate that we're going to impregnate and also attaches a tab to each plate but maintains it in a roll.

Next slide, please.

(Slide 192.)

The rolled plaque with the tabs attached is placed
on the impregnation equipment. The plates enter the trough through a slotted spillway and as they come through, there is a carousel, a cammed carousel which picks up each individual tab to make an electro-
connection.

The boiling nitrates are held in an insulated tank and are continually circulated through the impregnation trough. Temperature and pH are continually -- are controlled automatically.

The current then is passed through the plaque in the impregnation trough and the carousel indexes itself every so many minutes, according to the amount of pickup that we want.

(Slide 193.)

These are the storage tanks with the boiling solutions. The solutions are continuously run through the trough which is up here on the carousel. The temperature, pH, and what-have-you are automatically adjusted over at this end.

(Slide 194.)

What you can see in the corner here is the wheel with the sintered material and tabs attached. It is fed in through this slot in the trough. This cammed carousel clamps each tab as it is going through, and it runs in for a given distance and stops and then indexes every two to four minutes with current being passed through it.

Then it comes out the other end back here.

(Slide 195.)

We have our operator adjusting the rates. This gives you the other angle of the machine as it is coming in this part of the carousel and out the other side.

Next slide, please.
Finally the end roll, which is the impregnated plaque.

At this point that roll of impregnated plaque is transferred to a formation machine, which I don't have pictures of. It is a very similar arrangement as the impregnation where the plaque is picked up again and made an electrical connection, it's passed through the various tanks, charged and discharged, washed, scrubbed, and finally passes through an oven and dried.

At this point the plaques are then cut into plates and grouped for cells. Next slide, please.

(Slide 196.)

This is a slide of the cell testing. Now that we have them in cells we are testing, using cold plates. And also we have individual charge/discharge circuitry to automatically cut the cells in and out with their own timer.

(Slide 197.)

These are the cells. The battery itself is a 5-cell pack. The five cells are electrically connected in series using a bolt-on type intercell connector. Next slide, please.

(Slide 198.)

The 5-cell pack is then put between molded plastic restraining plates and slid into a stainless steel can. Next slide, please.

(Slide 199.)

This is the completed battery here ready for shipment. If there aren't any questions on that I would just want to show for the benefit of you people-- Is there a question?

CORBETT: Are those Ziegler seals?

HARSCH: A modification of the Ziegler seal, yes.
VII.3 NICKEL HYDROGEN CELLS: HARSCH/E.P.

For the benefit of the people who were at the Hughes design review last week, Hughes and their design review with Wright-Patterson Air Force Base, Don Warnock. And in that review they talked about cylindrical 50-ampere hour cell that they had designed and were in the process of building.

(Slide 200.)

These cells are built in conventional laboratory bombs mainly because the lightweight hardware was not available.

Basically the plates are what they refer to as pineapple shape. They have a rather large hole in the center.

Can we have the next picture on, and we can see the plates better.

(Slide 201.)

Howard Rogers said they were a half a bagel.

The electrical connection of the plate is made internally. Here's the tab material and the various separators. It's a little hard to see on the photograph, but there's a mandrill, a plastic mandrill here that has the shape of the separator and the plate. The plates are slid in the mandrill and built into this stack, like this. (Indicating)
There's a given amount of compression put on them by the end plates. The terminals here are rather long, to accommodate the container. They weld right to the top of this case.

Can we have the next one, please?

(Slide 202.)

Basically the pack configuration looks like this. And eventually this pack will go into a cylindrical container, a thin-walled cylindrical container with terminals coming out the top of the cells.

We now have the tooling -- I think it is finished -- and we will have the capability of building the lightweight hardware within another week, at least using Inconel 625 or Inconel 718 in deep-drawn thin-walled containers of about 20 to 25 mills.

There's only one other thing that I wanted to show on those two slides there.

(Slide 203.)

This, of course, most of you recognize as being a COMSAT design, of which we are delivering cells, I believe, this week. It's COMSAT's Inconel can with a single terminal. And on this versus the other design, the only difference is the way the plates -- the shape of the plates and the way they're mounted on the center rod. They have end plates also, which are slid into the bomb.

This is the completed cell here. This is the final cell configuration.

That's all I have.

Hennigan: Any questions?

Betz: On the electrochemical impregnation, is there a substrate? You're actually sintering -- I should say sintering using a slurry method, did you say?
HARSCH: Yes, that's right.

BETZ: What's your substrate material?

HARSCH: Nickel mesh.

BETZ: You're not using a perforated sheet?

HARSCH: No.

BETZ: Question No. 2: Do you use the electrochemically impregnated plate in the nickel hydrogen?

HARSCH: We haven't yet, simply because we're trying to get into full production for Western Electric and make deliveries on the committed batteries.

HENNIGAN: Any other questions?

(No response)

HENNIGAN: Thank you, Bill. You can catch your airplane now.

Our next speaker is Howard Rogers from Hughes Aircraft, who's going to talk about absorption of oxygen on the nickel electrode. This has to do with nickel hydrogen cells.
ROGERS: I might make one comment considering the last question that was asked on the electrochemical process. The Tyco cells that were made for us on the Air Force program were electrochemically impregnated on the same equipment that Eagle-Picher is now using. And these cells we have tested, which are nickel-hydrogen cells.

(Slide 204.)

I'm going to be talking about what we refer to as the chemisorbed oxygen in nickel-oxygen cells. I did a short series of experiments to find out what were the conditions of formation. We wanted to know something about the thermal, particularly the thermal, and also the electrical phenomena that were associated with this chemisorbed oxygen.

We defined chemisorbed oxygen simply in electrical terms as that capacity between 1 and zero volts; in other words, we simply took the cells down into reversal and measured the capacity between 1 volt and zero volts.

(Slide 205.)

This is a typical capacity test. And this is representative of almost any of the nickel-hydrogen cells we have tested thus far. What we did was to discharge the cell at a C/2 rate down to zero volts. We then changed to a C/10 rate and continued the discharge down to zero-volt level and sometimes beyond.

Had we continued the discharge at the C/2 rate, the dotted line shows what it would have looked like, and has looked like. That is not theoretical, that's an actual line.

You can clearly see that there's a step at the C/10 rate as a prime function of this. And it's also quite simple to measure the capacity.

(Slide 206.)
The cell used for these experimental runs was an ERC 50-ampere hour boilerplate cell, and was equipped with a stack thermocouple at the center for temperature measurements.

We made four runs. The first one is what we refer to as the base run, the charge rate of C/10. It was a 16-hour charge. And the discharge rate was kept at C/2.

The second run involved the same exact technique, with a 24-hour delay between charge and discharge.

The third run we stepped up the charge rate to the C/2 level to improve charge efficiency. But there was no overcharge. We simply calculated the amount of charge based on the actual capacity of the cell.

Finally, the fourth run: we simply cut that value in half. So we got essentially half charge.

The cells were run essentially at room temperature and they were actually externally cooled. And they were taken into reversal.

(Slide 207.)

Now I think the primary, the most important thing to see on this particular slide is the temperature, and that is the internal stack temperature, where we got a 24-degree rise in temperature primarily during the time that the cell went below 1 volt.

You will notice at the C/2 rate you don't see that clear a plateau. But there's obviously something happening between about 1 volt and three-quarters of a volt.

Let me see if I can superimpose the next one, which involves a delay.

(Slide 208.)

I think the most important thing here, while we did get some loss in capacity, we lost most of the plateau around
CHEMISORBED OXYGEN IN NiH₂ CELLS

OBJECTIVES OF EXPERIMENTS

- DETERMINE CONDITIONS OF FORMATION
- INVESTIGATE THERMAL AND ELECTRICAL PHENOMENA ASSOCIATED WITH IT

DEFINITION

- ARBITRARILY DEFINED AS THAT CAPACITY BETWEEN 1.0 AND 0.0 V

EXPERIMENTAL RUNS

ERC CELL

- 50 A-HR BOILER PLATE
- STACK THERMCOUPLE – CENTER

CONDITIONS

<table>
<thead>
<tr>
<th>RUN</th>
<th>OBJECTIVE</th>
<th>CHARGE RATE</th>
<th>DISCHARGE RATE = C/2</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>BASE RUN</td>
<td>C/10</td>
<td>70°F COOLANT WITH COOLING COILS ON CASES</td>
</tr>
<tr>
<td>2</td>
<td>24 HR DELAY</td>
<td>C/10</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>NO OVERCHARGE</td>
<td>C/2</td>
<td>CELLS TAKEN INTO REVERSAL</td>
</tr>
<tr>
<td>4</td>
<td>50% CHARGE</td>
<td>C/2</td>
<td></td>
</tr>
</tbody>
</table>

RUN 1 DISCHARGE

TYPICAL CAPACITY TEST

FIGURE 204

FIGURE 205

FIGURE 206

FIGURE 207
.9 volt level, or .8 level. We got some temperature increase but nowhere near as much, particularly if you look at the area under the curve.

(Slide 209.)

Now as I mentioned, in the next case we gave the cell a C/2 charge, not a full charge but exactly the full ampere hour rating of the cell. And, if you notice, there's a distinct plateau around .8 volt and a substantial temperature rise, indicating the chemisorbed oxygen effect.

(Slide 210.)

If you compare this to the one where we charged only approximately half-way, we got no plateau that anyone could see, and a very small temperature rise.

(Slide 211.)

Now to summarize the results: the amount of chemisorbed oxygen we saw was up to 20 percent of the normal capacity of the cell.

The formation of this chemisorbed oxygen is obviously connected with the state of charge that you reach. We found that it was generated both at C/2 and C/10 rates. And we got a very large reduction when we had a 24-hour period between charge and discharge.

If you look at what point in the charge cycle oxygen is being generated, we can definitely associate this phenomenon with that generation of oxygen.

Finally, the voltage range of this chemisorbed oxygen appears to be .8 to .88 volts.

(Slide 212.)

Conclusions from this would be that at the normal discharge rate we get a significant amount of self-heating in the stack. It's probably fast enough to avoid much of a vapor transfer problem due to the heat, but that one could
RUN 2 DISCHARGE

RESULTS

- AMOUNT OF CHEMISORBED OXYGEN UP TO ~ 20% OF NORMAL CAPACITY
- FORMATION OF CHEMISORBED OXYGEN DEPENDENT UPON STATE OF CHARGE
- GENERATED AT BOTH C/2 AND C/10 CHARGE RATES
- REDUCED BY DELAY BETWEEN CHARGE AND DISCHARGE
- ASSOCIATED WITH OXYGEN GENERATION
- VOLTAGE RANGE OF MAIN CHEMISORBED OXYGEN DISCHARGE IS 0.80 TO 0.88 V

RUN 3 DISCHARGE

RUN 4 DISCHARGE

FIGURE 208

FIGURE 210

FIGURE 209

FIGURE 211
CONCLUSIONS

- AT NORMAL DISCHARGE RATES, PRODUCES SIGNIFICANT SELF-HEATING IN STACK
- PROBABLY FAST ENOUGH TO AVOID MUCH WATER VAPOR LOSS
- POSSIBLE CUMULATIVE CELL DAMAGE FROM HEAT, ESPECIALLY WITH NYLON SEPARATOR
- MAY PROVIDE ADDITIONAL CAPACITY IN BATTERY
- SIGNIFICANT EFFECT ON MAXIMUM DESIGN PRESSURE AND USE OF PRESSURE AS STATE OF CHARGE INDICATOR (CHEMISORBED OXYGEN CAUSES EQUIVALENT INCREASE IN HYDROGEN PRESSURE)

FIGURE 212
worry about damage from the heat if you had, say, a nylon separator.

It can provide an additional capacity if you have a series of cells, because it does involve some additional capacity with a small voltage drop. That, of course, is assuming we're only losing one cell, or one cell loses capacity. And there is a very significant effect on the maximum design pressure of the case, and the use of pressure as a state of charge indicator.

One of the advantages of nickel-hydrogen is certainly going to be associated -- certainly is going to be affected by chemisorbed oxygen. Because you do get an equivalent increase in hydrogen pressure with the oxygen you store.

Thank you.

HENNIGAN: Any questions?

COHN: I missed something somewhere. Where do you get the absorbed oxygen? What evidence do you have that it is absorbed oxygen?

ROGERS: I don't. And I think I pointed out in the beginning that I was merely using it as a definition. People have talked about it that way. And I simply defined it as that plateau.

We made no attempt in this to prove whether it was or wasn't; we were interested in the effects.

CORBETT: Is what you describe peculiar to the nickel-hydrogen cells, do you believe; or is it the same thing we've been seeing in Nicad cells for a long time?

ROGERS: I think it's the same thing.

CORBETT: I was wondering, then, why this hasn't come up in the coulometric literature published by various people, why that hasn't shown up. It sort of hits me cold.

ROGERS: I don't know. It certainly has appeared here.
And it has been very obvious. There's no question about it being here. I really couldn't answer for what's in the literature.

DUNLOP: Which positives were those?

ROGERS: The positives here are ERC plates. I guess they're Gould plates.

We have seen this, incidentally, in the Air Force cells as well, which were the Tyco electrochemically impregnated plates. It seems to be very similar.

HENNIGAN: Any other questions?

(No response)

HENNIGAN: The coffee has arrived, so we'll take a 15-minute break. We do have two more presentations afterwards, one by Stokel from COMSAT on nickel-hydrogen and Parry on inorganic separators.

(Recess)
VII.5 NICKEL HYDROGEN CELL TESTING: J. STOCKEL/COMSAT

Joe Stockel of COMSAT who's going to speak on the nickel-hydrogen cell testing.

STOCKEL: Gentlemen, today I'd like to give a short summary of some of our nickel-hydrogen testing. May I have the first slide, please?

(Slide 213.)

This is the inside of a 50-ampere hour nickel-hydrogen cell provided to us by Energy Research. As you can see here, the stack is mounted to the dome here by four tie rods and the negative and positive feed-throughs are through the top here.

This was put into an electroform nickel can. Recently we've sort of gone away from the electroform nickel can and to Inconel-718 cans. The feed-through goes -- one feed-through out the top and one through the bottom. The internal mounting I believe will be essentially the same.

(Slide 214.)

This slide shows the design that COMSAT is currently pursuing. As you can see here, this of course is the pressure vessel. And we do have the Ziegler seal here at the top.

The positive feed-through here is right here. And the current is actually collected at a bus bar arrangement; which you probably can't see too well here; as opposed to some design where they actually take and bend each tab from each electrode.

In our new design we're actually going to have a
feed-through out the top and out the bottom.

The cell is held in pressure by these plastic end plates, with a tie rod down through the center.

(Slide 215.)

This slide shows some of our cells on test. Actually we've been using a water bath to control the temperature, and we run the cells -- we do control the water in the bath at 20°C.

You can see most of our cells do have pressure gauges on them, and these are the lightweight cells in the bath, and the ones over here are some of our small boilerplate cells.

(Slide 216.)

This slide shows some discharge, end-of-discharge voltages and end-of-charge voltages for the small boilerplate cells. These cells were -- they all had SAFT-type positive electrodes. On the ones you can see here, we did use polypropylene as a separator, and it didn't go any more than about 12 or 13 hundred cycles. And these were at a deep depth of discharge. They were on a 3-hour cycle about 85 percent depth of discharge, with about a 15 percent overcharge.

Now the polypropylene, as you can see, did not do too well. We took it out of the cell and looked at it, and it just would not absorb any water whatsoever.

The nylon cells did run to about 1500 cycles. There were two of them. And, once again, both of those failed, as you can see by the indication of the end of discharge voltage and the charge voltage rising. The impedance check on the cells showed that the impedance had gone high.

I did take one cell apart. I opened it, I should say. And I refilled it with electrolyte and put it back on test, and it's well over 4000 cycles now.
The cells with the fuel cell type separator, the KT and the asbestos, seemed to run fairly well. And to date now they're still going, and they have well over -- just about 4000 cycles on them. Once again, they're at the deep depth of discharge and 15 percent overcharge.

We also ran a cell -- I don't have it shown here -- that did have electrochemically impregnated electrodes with a nylon separator. Now that cell ran 2000 cycles, which we stopped for reasons that -- well, it didn't fail; we had to stop it. An examination of that electrode showed that it did not expand whatsoever. And I think that's pretty significant that we were able to run the electrochemical impregnated electrode with the nylon separator and have it run that good.

Next slide.

(Slide 217.)

This next slide just shows some of our 50-ampere hour lightweight cells that have been running since probably, I think they were started about the beginning of this year. They are the 50-ampere hour, they're run at 60 percent depth of discharge.

We had run this cell, this particular cell, on a 3-hour cycle with a 10 percent overcharge. It had the electroformed nickel can, and the stack was actually put together at COMSAT and delivered to Energy Research and put into their electroformed nickel can.

The negative electrode was from ERC, and it was a SAFT-positive, and there was a nylon 2505 separator. To date that cell has about 1600 cycles on it.

But what we did do right here, this curve here, we did change from a 3-hour cycle to a 6-hour cycle. It appeared that the 3-hour cycle was overstressing the cell somewhat: they were getting fairly hot. So we did switch over to a 6-hour cycle. The next slide is another cell.

(Slide 218.)
It is similar to that, as you can see. We did run it on a 3-hour cycle for a while and did switch over to a 6-hour cycle. The performance is, as you can see here, fairly good.

(Slide 219.)

This data was taken just from a 50-ampere -- from our lightweight cells, both the 35 and 50 ampere hour cells. These are early in life, about the first or second discharge cycle. And it just shows the difference in the separators that are being considered: the nylon, the asbestos and the KT. And, of course, nylon does give the better discharge performance, with the asbestos falling somewhere in between the nylon and the KT.

I guess that's about all.

HENNIGAN: Any questions?

HAINES: How many cycles do you have on this thing now?

STOCKEL: Well the two cells that I showed up there, like I say, they have about -- they are 50-ampere hour size, and they are running 60 percent depth of discharge and 15 percent overcharge on a 6-hour cycle. And one has 1400 and the other has about 1650 cycles on them. And they are running in that temperature range.

Those were the first two cells.

KLEIN: I guess they were about a year old?

STOCKEL: Exactly. They'll be a year old in January.

HOLLECK: Joe, I was wondering, I don't know whether I saw that right. There's one asbestos cell. It looked to me like it had a void around 1.1, on the slide where you had the polypropylene failing.

STOCKEL: That was KT and -- well, asbestos was in there, very close.
FIGURE 217

FIGURE 218

FIGURE 219

NI-H\textsubscript{2} LIGHTWEIGHT CELLS

277A ORIGINAL PAGE IS OF POOR QUALITY
HOLLECK: I was wondering why that appeared to be even much lower than this one.

DUNLOP: The problem with the boilerplate cells, in those we used leads that have a fairly high impedance. In the 50-ampere hour cells we have a tab design and the impedance is significantly lower. But in the boilerplate type cells there are fairly small feed-through terminals and wires and they cause a fair amount of drop.

STOCKEL: All these cells do have the plastic type pressure seal. We've had very good luck so far with these seals in the nickel-hydrogen cell. And we did -- as I guess was mentioned before, we did supply these seals to JPL. And we are interested in the technology. And if anyone is interested in acquiring any, or any more information on it, I'll be glad to talk to them if they will contact me.

HENDEE: On your life test, especially the polypropylene separators that failed out a little early, did you have that plate stack spring-loaded or was it a fixed pressure?

STOCKEL: That was only what we call a single electrode cell. It only had two electrodes back-to-back. To date we only have one cell that has asbestos in a large multi-electrode stack running.

HENDEE: Is that spring-loaded?

STOCKEL: No. None of them are.

HENNIGAN: What kind of polypropylene was it?

STOCKEL: I think it was Pellon. We had Hercules around, but I believe that was Pellon.

LACKNER: On the variations in the voltage on your discharge curve, what is it that causes that different voltage drop? Is it the amount of electrolyte, is it a resistance in the separators?

STOCKEL: Yes, I believe it is.
LACKNER: Which? The amount of electrolyte?

STOCKEL: No; the resistance in the separator. They're all loaded— These cells are loaded differently than the nickel-cadmium.

When I load a cell I will pull a vacuum and then completely flood the interior of the cell, and then remove the electrolyte either by charging the cell or by pulling a vacuum, or in some way getting the excess back out.

LACKNER: Now, rather than taking the polypropylene separator, all the different separators you get, if you had asked the separator manufacturer -- and there are a number of them -- to give you a particular resistance value, then you may not get into a case where you say polypropylene or asbestos or KT is up or down, and there may be some other characteristics you would want with those three.

This may be a point to look at.

STOCKEL: Yes.

HAINES: Did you extract that polypropylene? In other words, did you extract the wetting agent out of it before you used it?

STOCKEL: No, I did not. I used it as I got it.

KLEIN: I was going to make a comment on this point of the different resistances in the separators. It's a question of the pore size and the pore size distribution that is drastically different between the asbestos or potassium titanate and the Pellon. So the tortuosity factor is much greater. And actually, even though we pay that resistance penalty with the so-called fuel cell grade materials, we think that's favorable because having as fine a pore size may retain the electrolyte seemingly better than the open mesh woven materials in this application.

LACKNER: That may well be true. But I think if you will check with Dr. Landem at Wright-Patterson -- he did a lot of work with aircraft batteries, and he could actually
get -- it was RAI in this case -- separators anywhere from 10 milli-ohm to 60 milli-ohm, depending upon what he wanted. It was a question of engineering them to the application.

Polypropylene can do the same thing. We can engineer it to the application. And it isn't that-- The tortuosity is whatever it happens to be.

Now if you're unhappy with the tortuosity you can still make some changes. It's a question of communication with the manufacturer.

HENNIGAN: What I would like to do is get into the next speaker. And after that you're welcome to stay here and discuss any topics that you want.

Our next speaker is John Parry from Arthur D. Little, and he's going to talk on inorganic separators for the silver-zinc cells.
PARRY: I want to describe briefly some work we've done for NASA on the organic separator of the silver-zinc cell. Essentially this is a follow-on to a paper that John Bozak of NASA-Lewis gave at the workshop last year, where he described some of the details of the structure of this particular separator.

(Slide 220.)

I would like to say quickly that the objectives of our work were to identify those factors and mechanisms that give the separator its good characteristics in the silver-zinc cell. I define those good characteristics as good conductivity and effective barrier capability to silver diffusion, and relatively long life.

Our approach to this was to look at structure, primarily with the scanning electron microscope. And since it was described last year in a fair amount of detail I don't intend to repeat that.

We looked at diffusivity, we looked at conductivity, we looked at transport numbers, and we looked at silver migration and retention to see what we could understand about this separator.

(Slide 221.)

I would like to remind you of what this structure looks like. It consists of asbestos, which is used in practice in the form of a bag or envelope and lightly coated with polyphenolene oxide which gives it an extra degree of resistance to potassium hydroxide. This is coated with a mixture composed of the 3420-25 powder. This is a zirconia prepared in a proprietary manner. Some potassium titanate needles and polyphenolene oxide and a polyvalic plasticizer, which is a commercial plasticizer, Emery Industries' P9-750 which, as far as I can tell, is composed of some high molecular weight di-carbasylic acids. All this is mixed up in chloroform to create a slurry for coating the asbestos.
I think I should point out at this point that the plasticizer is not a plasticizer \textit{per se}. When the chloroform comes out the PPO and the plasticizer are, to a large extent, immiscible, they do segregate when the solvent dries out.

I want to draw attention to this particular point at the bottom, the cuticle, since this will appear in some of the tables that I'm going to show.

This I interpret as the skin, which is entirely the organic component, as opposed to the 3420-25 powder KT which forms on the surface of this coating when it dries out. And what I'm going to try and show is that most of the attractive properties of the separator derive from the cuticle and not from the inorganic components.

(Slide 222.)

Since what I'm going to do is throw a lot of numbers at you I think it's only fair to put up the principal conclusions first, and then perhaps the numbers will be a little easier to digest.

As I've already said, the attractive characteristics of the separator seem to derive from the organic components and not the inorganic. So that the "inorganic" title is a misnomer. But I would suggest that we retain it because we're at least familiar with it.

The barrier properties actually derive from this thin skin on the surface of the coating which becomes a semi-permeable membrane on exposure to KOH. KOH reacts with the polymeric plasticizer. It's a quantification reaction which I think results in the creation of the semipermeable membrane.

Another point which has to be added to this is that this thin membrane is by no means perfect. It has quite a large number of holes in it, and some cracks and other physical defects. As used in practice, these separators are used as two envelopes, one around each bag -- one around each electrode. And I think this has the effect of
eliminating most of the effects of the physical defects.

(Slide 223.)

These are measurements that we made of diffusivity. And I would just make one point here.

The first few months of the program we did spend looking at the inorganic components of the separator, looking for surface charge on the zirconia. We did some electrophoretic mobility measurements, and we tried to look at ion exchange capacity on the powder. We spent quite a bit of time before we got to measuring the diffusivity as a function of temperature in KOH. And since we tried to do these measurements with the separator as received, we discovered that there were very rapid changes in properties as a function of time and determined the extent of this chemical reaction between the plasticizer and the KOH.

We then set out to do these experiments in which all the separators are pretreated at 80°C for 48 hours in KOH prior to making any physical measurements on them. And for this particular slide, the numbers I want to look at in detail are to compare this figure of 1.42 here for a single separator measured with 45 percent KOH at 26°C and two of these separators put face-to-face where this number is reduced to .25. So we have almost a factor of four or five between these—More than a factor of 5 between these two numbers when we put a double separator in instead of a single.

This I think is indicative of the number of defects that occur in the separator as made.

(Slide 224.)

Again some more diffusivity measurements. And the only figures I want to draw attention to are this one here for the double separators with the coating in contact; the same figure from the previous slide; and this one here for the double separator where we actually removed the surface skin. And this is a technique one of our coatings chemists uses.
OBJECTIVE
TO IDENTIFY AND UNDERSTAND THE MECHANISMS INVOLVED IN THE
CONDUCTION AND BARRIER PROPERTIES OF THE SEMI-FLEXIBLE
MEMBRANE SEPARATOR

APPROACH
STRUCTURE
DIFFUSIVITY
CONDUCTIVITY
TRANSPORT NUMBERS
SILVER IONIZATION AND RETENTION

FIGURE 220

PRINCIPAL CONCLUSION

- THE ATTRACTIVE CHARACTERISTICS OF THIS SEPARATOR DERIVE FROM
  THE ORGANIC COATING
- THE BARRIER PROPERTIES OF THE SEPARATOR DEPEND ON THE FORMATION -
  ON EXPOSURE TO POTASSIUM HYDROXIDE - OF A THIN SEMI-PERMEABLE
  MEMBRANE AT THE SURFACE OF THE COATING (CUTICLE).
- THE MEMBRANE IS NOT PERFECT AND IS SUBJECT TO HETEROGENEOUS PHYSICAL
  DEFECTS

FIGURE 222

TABLE 1
DIFFUSIVITY OF H2S IN FLEXIBLE LAMINATED CONSTRUCTION

<table>
<thead>
<tr>
<th>Concentration (Molar)</th>
<th>10°C</th>
<th>15°C</th>
<th>20°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1E (1E.66)</td>
<td>1.04</td>
<td>1.07</td>
<td>0.84</td>
</tr>
<tr>
<td>3E (3E.66)</td>
<td>0.63</td>
<td>0.39</td>
<td>0.40</td>
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<tr>
<td>1E (1E.90)</td>
<td>0.28</td>
<td>0.25</td>
<td>0.26</td>
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</tbody>
</table>

For double separator in 4E H2S at 20°C Diffusion rate = 0.25 x 10⁻⁶

FIGURE 223
By putting sticky tape down on the surface he's able to take off this surface film without really physically damaging the rest of the structure.

I think this emphasizes the importance of this thin surface skin on the characteristics of these membranes.

(Slide 225.)

These are measurements of resistivity. And I want you to look at the last two columns where we're comparing the resistivities of the double and single separator. To some extent the difference is not so great here. The experimental conditions are different. In the diffusion measurements there's a 14 molar concentration difference between the two sides of the separator, and I think this shows up the defects in bad light. In resistivity the concentration is the same on either side. Nevertheless, the defects have a lower resistivity.

(Slide 226.)

One other set of resistivity data, where I just want to look at the figures that we have over on this far side, and not deal with the rest of the data at the top.

Essentially what we can do is peel this membrane apart, and we can strip off the asbestoes from the back--Let me start at the beginning.

This is the complete separator, first of all, .4824. We can then strip off the asbestoes from the back of the separator. We can also get rid of the cuticle on the front by the sticky tape technique that I talked about. And we can take these numbers here and, by subtraction, determine the contributions to total resistance of the various layers of the separator.

You can see from this that the very thin cuticle on the surface does make a very significant contribution to the total resistivity of the separator.

(Slide 227.)
Table 4

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Fraction Volume</th>
<th>Specific Impedance</th>
<th>Specific Impedance of Double Volume</th>
<th>Specific Impedance of Double Volume</th>
<th>Specific Impedance of Double Volume</th>
<th>Specific Impedance of Double Volume</th>
<th>Specific Impedance of Double Volume</th>
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Table 5

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<th>Specific Impedance of Double Volume</th>
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<th>Specific Impedance of Double Volume</th>
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FIGURE 224

FIGURE 225

FIGURE 226

FIGURE 227
Some quick comments on silver diffusion and silver retention. There's a massive amount of data on here. But the only point I want to draw attention to is that except in some special cases the concentrations in the downstream solution here are really too low, in most cases, to be significant.

These are silver radio tracer measurements. In other words, the separator is a very effective barrier for silver migration across the cell.

I might add, at the bottom here in this line we have separators taken from a cell that had a total of a thousand days wet stand life with routine synchronous orbit cycling in that period, and there was no movement of silver even through those separators.

(Slide 228.)

These are short term tests. And I just want to single out two points here.

First of all, we're only looking at the upstream separators. Where we looked at the doubles, the downstream separators showed no silver content at all. But the silver retention in the cell did depend on which way we oriented the separator itself. There is significant chemical interaction between the soluble silver and the organic components of the cell, and particularly if that separator has not been pretreated there's an even bigger interaction.

We did attempt to look at how the various components of the separator did soak up silver. And this is shown here.

(Slide 229.)

First of all we find that -- this is silver taken up from the solution. It's an easy way to measure it. The 3420-25 powder showed very little pickup, a slight surface adsorption. The polyphenolene oxide powder, one of the raw materials, showed quite significant pickup. The PPO cast film also showed quite significant pickup. Once we mixed
SILVER UPTAKE BY SEPARATOR COMPONENTS

CONTROL
3420 25
PPO POWDER
PPO FILM
PPO + PLASTICIZER FILM
COATING LAYER OF SEPARATOR
ASBESTOS + PPO FROM SEPARATOR

24 Hours

Counts Per Min of Solution

500

1000

FIGURE 229
the PPO and the plasticizer that number goes up quite considerably. And, of course, these are virtually components of the coating layer of the separator except that they're present in higher surface area form. And here the PPO distributed on the asbestoes is also a very high surface area.

So there's a considerable chemical sink in the separator for the removal of silver from solution. And I think this is a big component of the overall reaction. But there is a fundamental barrier to silver diffusion as well at the surface.

Thank you.

HENNIGAN: Are there any questions?

CORBETT: How would the resin itself with the plasticizer be as a film separator, given your conclusion that the barrier properties come from the organics themselves?

PARRY: I think you need more than just barrier properties in a silver-zinc separator. You need an electrolyte reservoir as well. This is the function of the asbestoes.

I think the inorganic components of the coating provide a very good mechanical key for the thin film at the surface to the asbestoes physical support. I think they are an important part of the separator in that context.

Within the narrow definition of this piece of work, the inorganic component does not seem to contribute to the real mechanistics of the separator itself.

We've just started doing some work with respect to the zinc electrode. I might revise my opinion in that direction. There appears to be an interaction between the inorganic component and zinc. I don't want to say more than that at this time.

GANDEL: What temperature was that last slide?

PARRY: The last was at 26°C.
MENNIGAN: Wasn't there a reaction between the original separator and the zinc?

PARRY: We did some quick gassing experiments of powdered zine and the 3430-35 powder, and there is slight hydrogen evolution, yes.

KLEIN: What's the pore size distribution of this thin plastic film that you have there?

PARRY: I don't know, Marty. I guess it is down in the hundredths of angstrom region.

POWELL: What's the thickness of the asbestos layer?

PARRY: The asbestos, if I remember correctly, is 12 mills --10 mills; thank you, Marty. 10 mills. The coating builds up to about 4 mills on the surface.

The coating itself, from our analysis, turns out to be 50 percent porous.

HENNIGAN: Any other questions?

(No response)

HENNIGAN: Thank you very much, John.

HALPERT: I want to thank you all for coming. I hope we have made another step forward, as we hoped we would at the beginning.

I want to thank all of our chairman -- Floyd, Tom and Bill -- for their help. And Mr. Bloom, who has been talking into that machine for two solid days, we want to thank you very much. And also our people back there in the recording studio, who are in the other building, Tom Dixon, thank you very much.

Have a good trip home.

(Whereupon, at 4:25 p.m., the 1974 Battery Workshop was concluded.)
APPENDIX A

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