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RADIATION EFFECTS ON SILVER AND ZINC
BATTERY ELECTRODES. I
INTERIM REPORT
APRIL 1965 TO JULY 1965
PREPARED FOR JET PROPULSION LABORATORY
UNDER CONTRACT NO. 951109

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**ATOMICS INTERNATIONAL****A DIVISION OF NORTH AMERICAN AVIATION, INC.**

**RADIATION EFFECTS ON SILVER AND ZINC
BATTERY ELECTRODES. I**

INTERIM REPORT

APRIL 1965 TO JULY 1965

**PREPARED FOR JET PROPULSION LABORATORY
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WAS 7-100

BY

G. R. ARGUE

H. L. RECHT

W. A. McCOLLUM

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.

AUGUST 16, 1965

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I. SUMMARY

During the initial characterization of silver-zinc cell electrodes, and the study of the effects of gamma radiation on them, excessive growth of dendrites from the zinc electrode occurred. Therefore, cadmium electrodes were substituted for zinc in this phase of the work.

A silver electrode (initial weight = 3.37 gm) which had a capacity of 0.8 AH, lost 54 mg of material after a gamma dosage of 1.1×10^8 rads (H_2O). An identical unirradiated silver electrode at the same temperature ($45^\circ C$) and cycling conditions lost only 4.1 mg material.

Small gamma radiation dosages were found to be sufficient to initiate precipitation of silver metal from the solution containing a charged silver electrode. This effect was observed after only a 5-min irradiation [10^5 rads (H_2O)], and probably would have been apparent after even shorter exposures. The precipitated silver metal appears to be the product of the radiation induced reduction of silver oxides in solution.

Pressure measurements on the irradiated cells did not lead to a consistent pattern in that both pressure increase and decrease were observed. Gas composition data were not obtained during this initial phase of the study.

Numerous technical problems were encountered in the measurement of the ampere-hour capacity of the silver electrode. No definitive data on the effect of radiation on silver electrode capacity were obtained. Redesign of the test cell, with repositioning of the cadmium electrodes, should permit these measurements to be carried out.

II. INTRODUCTION

Storage batteries are used as a source of auxiliary power on almost all space vehicles. These vehicles are exposed to various types of radiation from either cosmic rays or the Van Allen radiation belt. The flight of the SNAP 10A exposed the batteries on board to rather high levels of radiation. During the 43-day life of that mission the batteries, both Ni-Cd and Ag-Zn, received about 10^6 rads (H_2O) total irradiation.

Previous studies⁽¹⁾ have shown that radiation damage to the Ni-Cd system starts at about 10^6 rads. Therefore, it appeared imperative that a similar study be made on the important Ag-Zn system.

III. EXPERIMENTAL

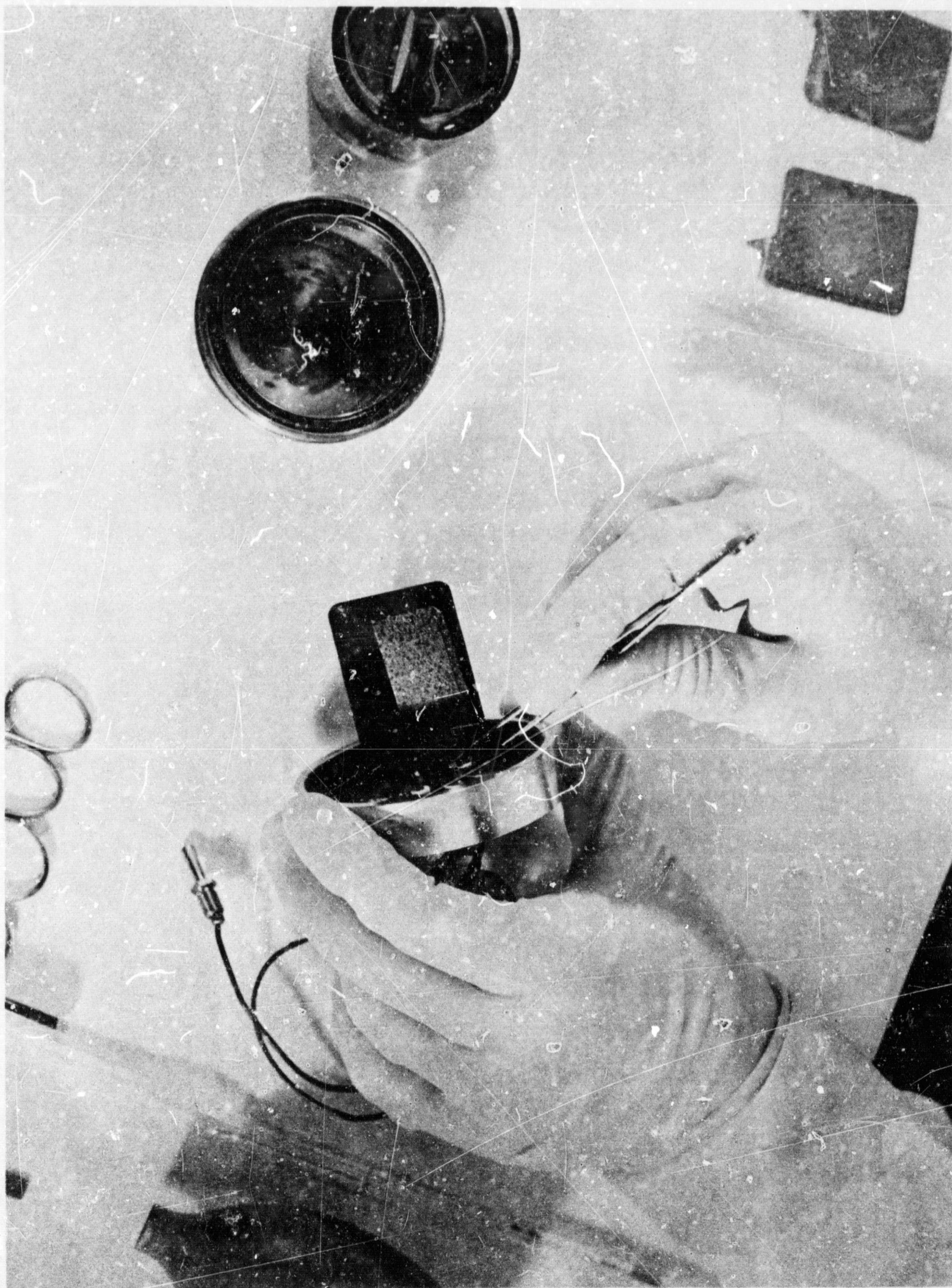
A. CELL DESIGN

A stainless steel cased cell similar to that used in a study⁽¹⁾ on nickel-cadmium electrodes was used in the present investigation. The cell is shown in Figure 1. The top of the cell was fitted with a pressure transducer (C.E.C. Model 2-316) having a pressure range of ± 12.5 psig with a sensitivity of 1 mv/psi. A stainless steel needle valve was also attached to the top of the cell to permit gas chromatographic sampling of evolved gases. The three electrical connections through Stupakoff seals for the three electrodes, namely, the silver, the zinc or cadmium, and the reference Hg/HgO electrode, were also fitted to the top of the cell. The cell top, when screwed onto the base, made a pressure-tight seal with a "Viton" O-ring.

A quartz or polystyrene liner was placed in the stainless steel cell to hold the 50 ml of 40% KOH electrolyte used. A porous quartz disc of about 1/8-in. thickness separated this liner into two compartments; one for the silver electrode and one for the zinc electrode. This liner is shown in Figure 2A. The anode and cathode in each liner were spaced about 0.5 in. apart. The electrodes were held in place by slots in the polystyrene liner.

In those experiments employing cadmium electrodes, three electrodes were used to provide adequate capacity. Initially, these were placed together in one compartment. This configuration did not permit full utilization of the active material on the electrodes. A liner of new design which will have three compartments is shown in Figure 2B. One cadmium electrode will be in each of the two outer compartments and the silver electrode will be in the inner compartment, with quartz disc separators between each of the electrodes. In use, it may be necessary to cut the silver electrode to match the capacity of the two cadmium electrodes.

The silver electrodes were received in the unformed (all metallic) condition and had to be charged before use. A lucite laboratory cell containing two platinum electrodes, one placed on either side of the silver electrode as a counter electrode, was designed for this purpose. This cell is shown in Figure 3.

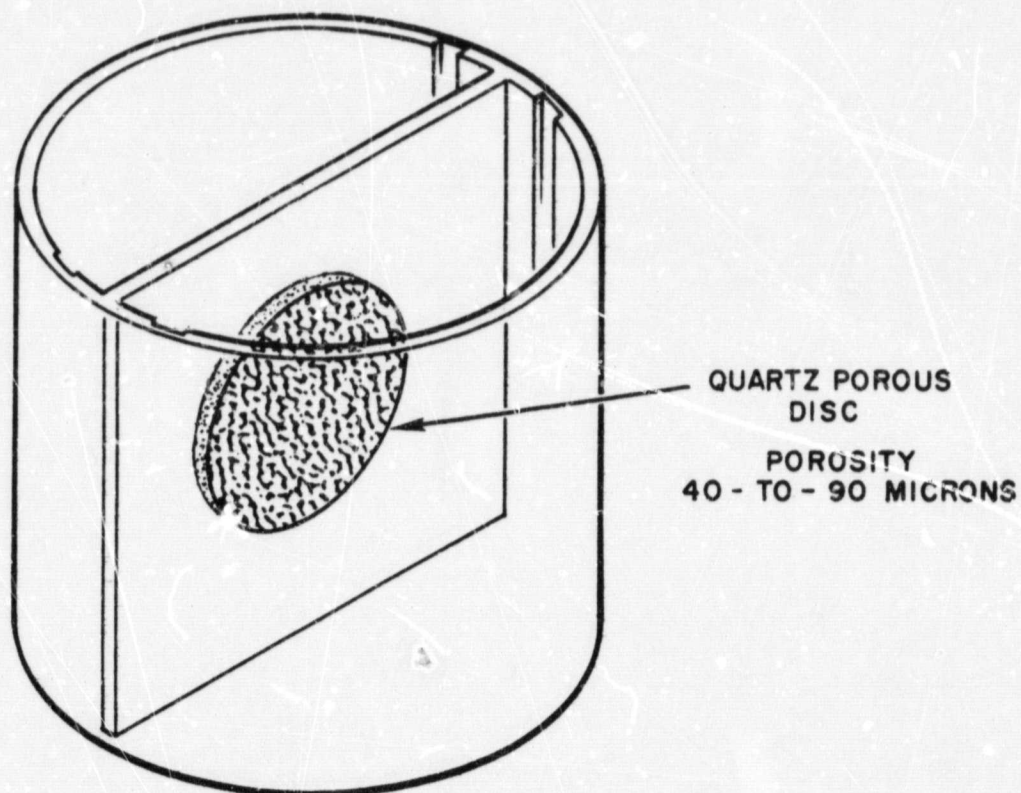


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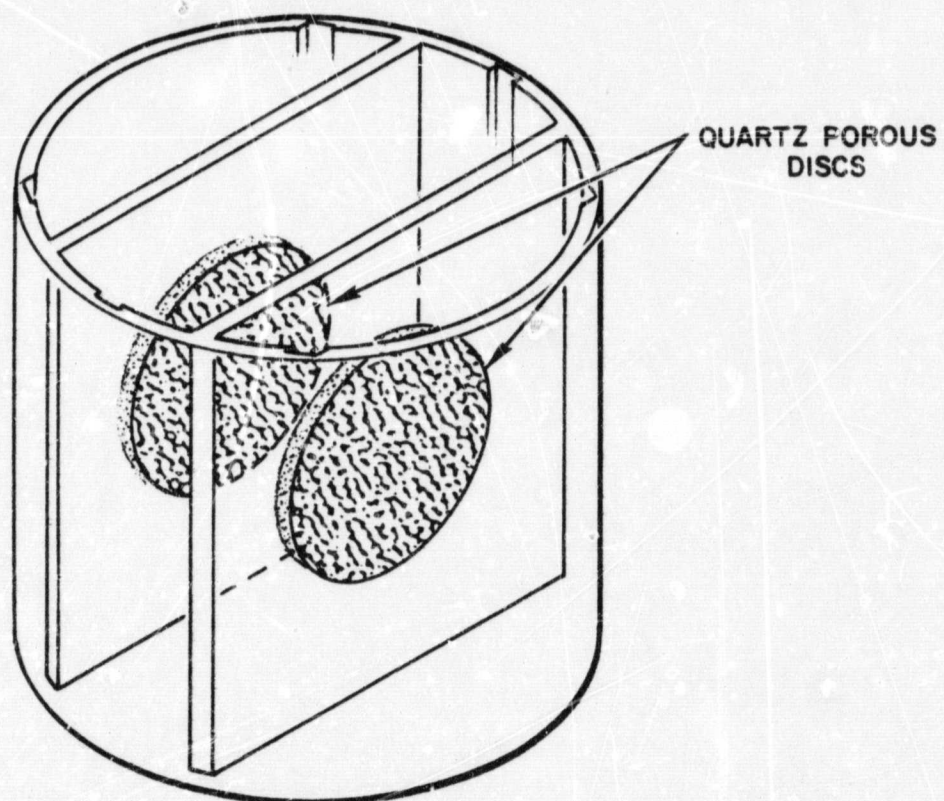
Figure 1. Assembly of Stainless Steel Cell for Silver-Zinc Electrode Studies

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A.



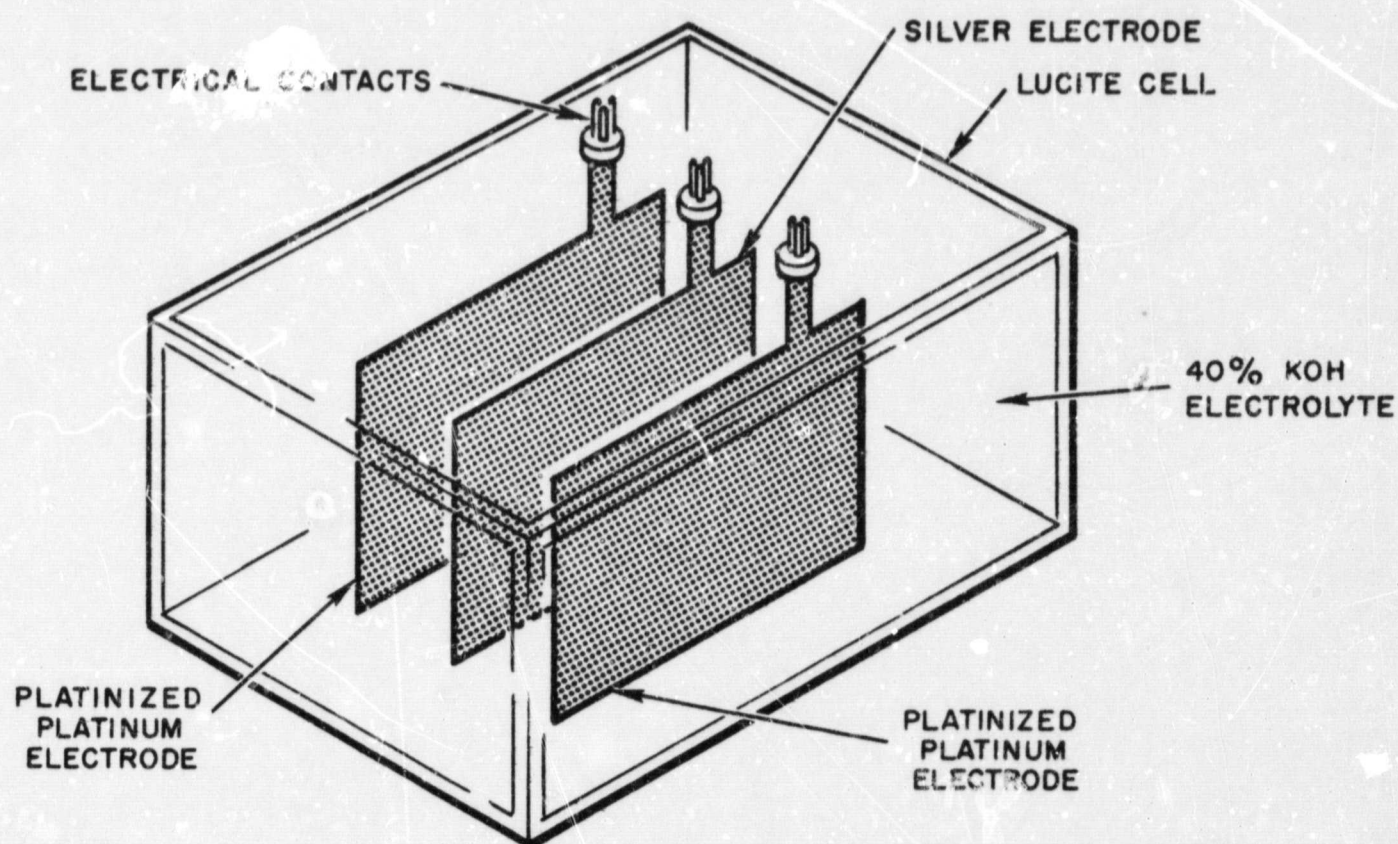
B.

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Figure 2. Polystyrene Cell Liners

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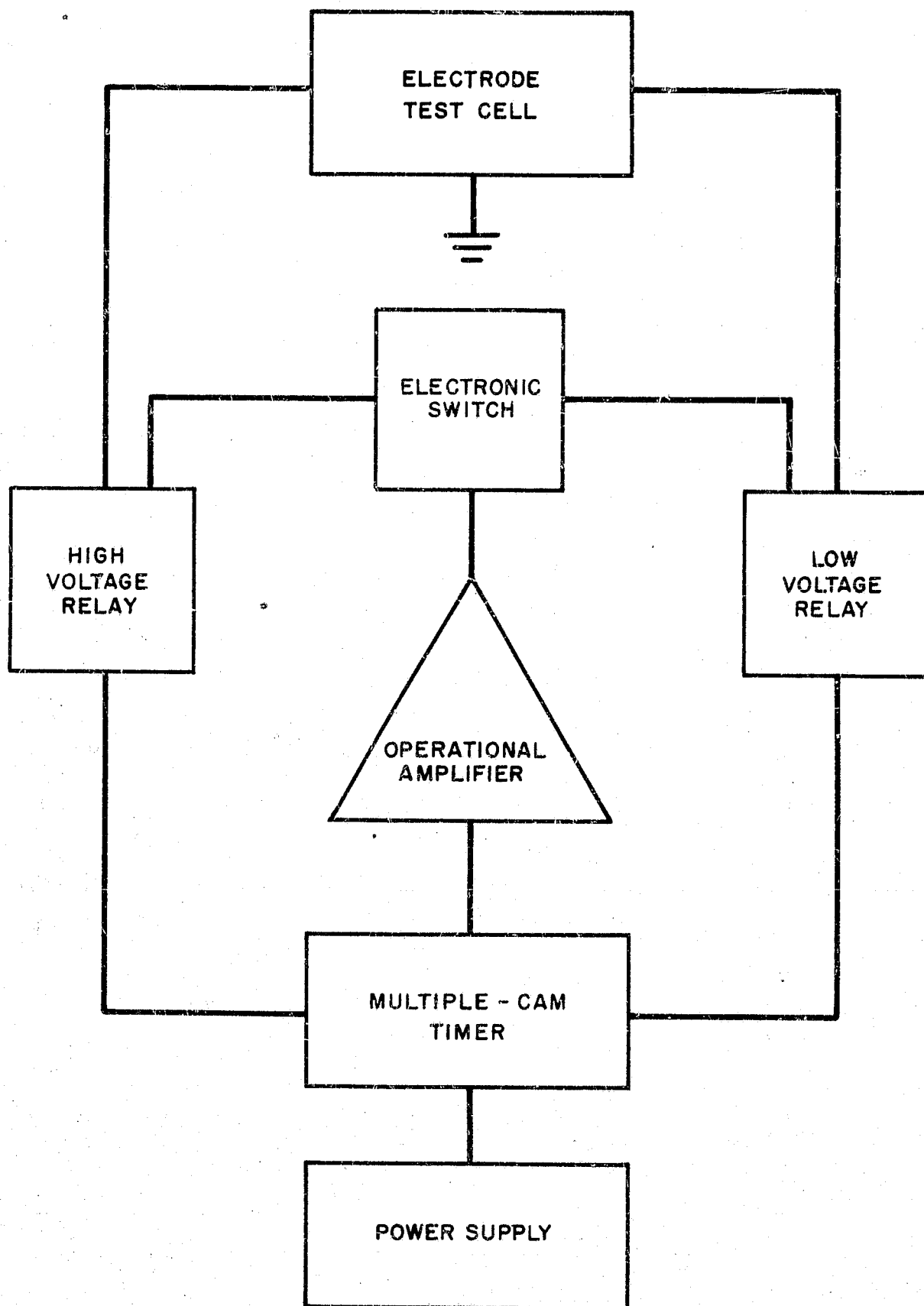
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Figure 3. Silver Electrode Charging Cell

B. CIRCUITS FOR MEASUREMENT AND CONTROL

Two important operations, namely, automatic measurement of electrode capacity and automatic control of current for cycling, required the design and construction of electronic circuits. The circuitry for automatic measurement of electrode capacity required voltage sensing discharge and charge cut-offs for the current as well as a timing arrangement so that the cells could be automatically switched from charge to discharge and vice versa. A block diagram for this circuit is shown in Figure 4. The system was designed so that the cut-off voltages could be set to take into account the IR drop in the cells. The lower voltage point was set for that corresponding to the knee of the discharge, while the upper point was set for a voltage above that corresponding to initiation of gas evolution on charge. For the Ag-Zn system, these voltages are 1.1 volt and 2.1 volt; for the Ag-Cd system, the voltages are 0.8 volt and 1.7 volt, respectively.

Automatic equipment was designed to provide a cycle of 15 min of charge, 15 min on open circuit, 15 min of discharge, and then 15 min on open circuit. This circuit has been described elsewhere.⁽¹⁾



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Figure 4. Block Diagram of Electronic Switching Circuit

C. GAMMA RADIATION SOURCES

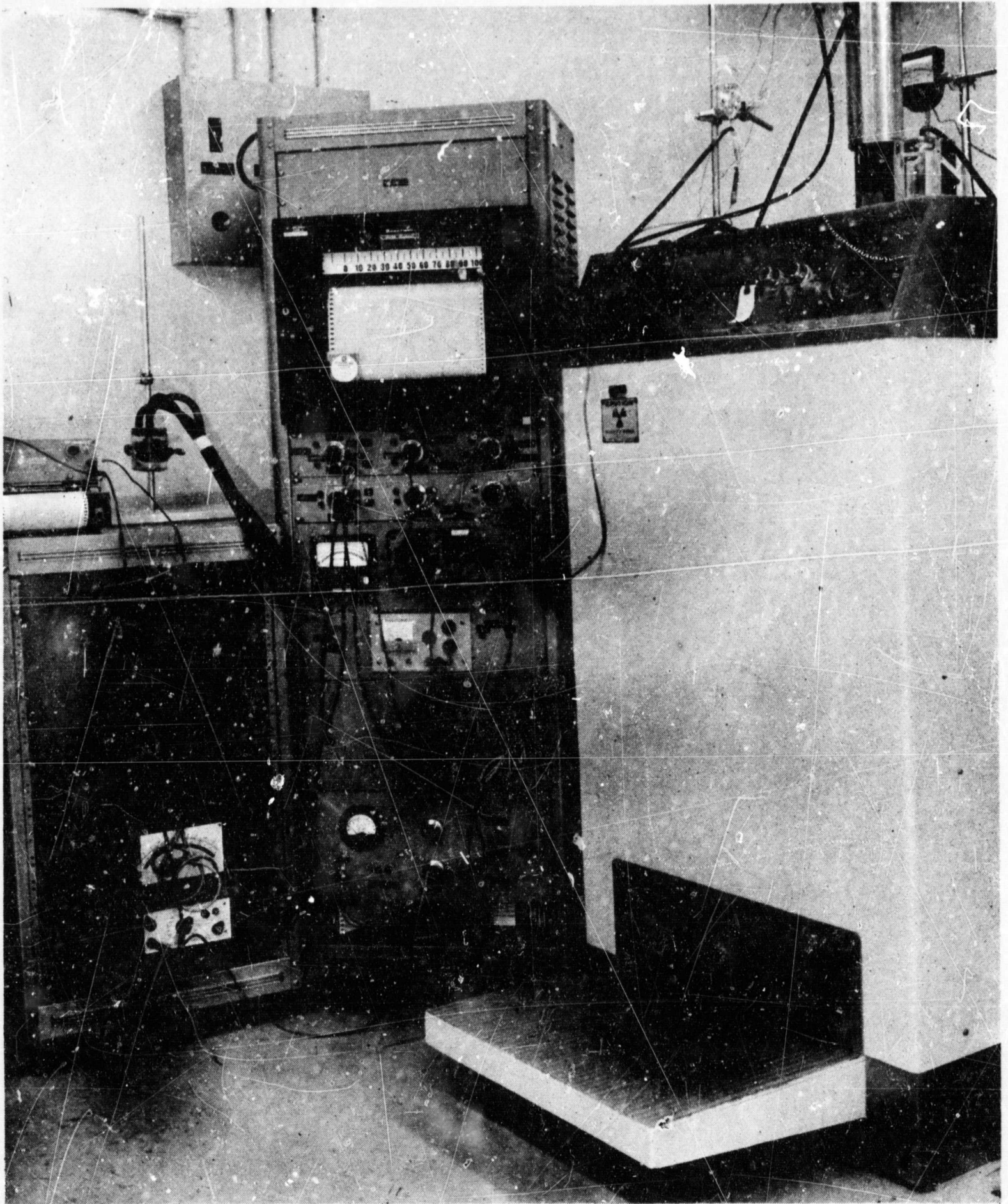
Two Co^{60} gamma sources were available for this study. Source I had a dose rate of 8×10^4 rads (H_2O) per hour at the position of a cell in the source. Source II, rated at 1.2×10^6 rads (H_2O) per hour, was the one used for all runs to date. Total dosages ranged from 7.6×10^7 to 1.06×10^8 rads, corresponding to irradiation periods of from 65 to 89 hours. Figure 5 shows the experimental cell in position ready to be lowered into Source II.

D. ELECTRODE SPECIFICATIONS

The positive (silver) and negative (zinc) electrodes were obtained from Yardney Electric Co. The electrodes which had 0.8 amp hours capacity measured 1.35×1.63 in. Problems were encountered with the zinc electrode with regard to physical deterioration. In order to obtain definitive data on the behavior of the silver electrode undergoing irradiation, it was decided to test silver-cadmium cells during the initial phase of this study. Accordingly, cadmium electrodes of 0.45-amp-hr capacity were ordered from Union Carbide. Cadmium electrodes obtained from Gulton Industries having 0.40-amp-hr capacity were used for the Ag-Cd runs described in this report.

E. ELECTRODE TESTING PROCEDURES

The silver and the zinc electrodes were received in the metallic state. Thus, the silver electrodes had to be charged before incorporating them into Ag-Zn cells. For a typical run, two silver electrodes were charged in the laboratory cell described earlier at a current of 50 ma for 20 hr. One of the silver electrodes and a zinc electrode were then placed in each of two stainless steel cased cells containing 50 ml of an electrolyte of 40% KOH, saturated with ZnO . The cells were then discharged by 10% of their rated capacity at 100 ma, and placed on automatic cycling (as described above) at a current of 100 ma for 24 hr. After this period, the cells were then fully charged and the amp-hr capacity measurements made. This measurement involved a complete discharge at 100 ma to the discharge knee (around 1.0 volt) and then another complete charge at the same current. This charge-discharge cycle was carried out at least twice, although a third measurement was usually required to obtain a consistent ($\pm 2\%$) value for the capacity. The cells were then left at full charge



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Figure 5. Test Cell Installed in Co⁶⁰ Source II

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so that just before the start of the irradiation, they could be discharged to the desired state of charge. After reaching the specified state of charge the cells were placed on automatic cycling. One cell was placed in the radiation source and the second cell placed in a constant temperature bath at 45° (the temperature inside the Co⁶⁰ source).

After the irradiation period of 65 hours at total dose of 7.6×10^7 rads (H₂O), the cells were removed from the source and the bath, and allowed to cool to room temperature. They were then discharged, and capacity measurements were carried out for two or three cycles. The cells were then opened and the electrolyte which might contain sloughed material was removed from the separate compartments. The electrolyte was centrifuged, and the precipitate was washed, dried, and weighed. This material was then submitted for analysis by x-ray diffraction and x-ray fluorescence.

IV. RESULTS

A. CHARACTERISTICS OF ELECTRODES IN PRELIMINARY TESTING

The silver and zinc electrodes specifications received from the manufacturer gave these current specifications:

- 1) Charging current 0.050 amps
- 2) Discharge current 0.150 amps
- 3) Capacity 0.5 amp-hr

The charging mode was carried out initially for 11 hours to overcharge the silver electrodes. However, this amp-hr input was found to be insufficient to charge these silver electrodes. On testing, it was found that 20 hours at 0.05 amps was required before the electrodes appeared to gas, indicating full charge. Using this current, the time period to charge and discharge the cell completely was then in excess of 24 hours. In order to permit a complete cycle to be completed in less than one day, the currents used for this cycle were changed to 0.10 amp for both the charge and discharge phases. The time for a cycle then became about 17 to 20 hours.

It was during these initial capacity measurement cycles that excessive zinc dendrite growth and material loss from the electrode were observed. After two such deep cycles, the zinc electrode was rendered useless for further cycling runs. In two of the earlier irradiations, these cycled zinc electrodes were replaced by new zinc electrodes just before the irradiation. These also exhibited excessive deterioration. At this point in the program, it was decided to substitute cadmium electrodes for the zinc electrodes. The only electrodes available were from nickel-cadmium batteries with a 0.8 amp-hr capacity rating. These cadmium electrodes had about 0.44 amp-hr capacity, which necessitated the use of three cadmium electrodes with each silver electrode to permit the silver electrodes to limit the cell capacity.

B. CAPACITY MEASUREMENTS

Reproducible amp-hr capacity measurements were not obtained on any of the silver-zinc runs, because of the difficulty with the zinc electrodes described above.

Incomplete data were obtained from the initial runs on the silver-cadmium cells as the cadmium electrodes became capacity limiting. This was thought to be due to the inaccessibility of the inner surfaces of the cadmium electrodes. The original design necessitated the stacking of the three cadmium electrodes together, and the closeness of the plates to one another makes the inner surfaces of the plates inaccessible. Redesign of the cells to permit the utilization of the entire capacity of the cadmium electrodes has been completed.

C. IRRADIATION RESULTS

1. Material Loss

No quantitative data were obtained on the loss of material from the zinc electrode as large amounts were lost from both the irradiated and control electrodes due in part to extensive dendrite growth. Material was lost from the silver electrode in all runs. These data are shown in Table 1.

X-ray diffraction and x-ray fluorescence analyses were made on all centrifuged residues. The data from these analyses are shown in Table 2.

Emission spectrographic analyses were made on the electrodes as received, on the irradiated electrodes, and on sloughed materials from selected runs. These data are shown in Table 3.

TABLE 1
MATERIAL LOSS FROM SILVER ELECTRODES

Run *	Total Gamma Dose Dose in Rads (H ₂ O)	Weight of Precipitated Material in Silver Electrode Compartment (mg)	
		Bath Cell	Irradiated Cell
ZG-1	7.8 x 10 ⁷	2.5	42.8
ZG-5	7.8 x 10 ⁷	36.0	77.4
ZG-6	7.8 x 10 ⁷	11.2	78.5
CG-7	7.8 x 10 ⁷	1.3	50.4
CG-8	7.3 x 10 ⁷	9.6	50.6
CG-9	1.07 x 10 ⁸	4.1	54.7

*ZG = Ag-Zn cell
CG = Ag-Cd cell

TABLE 2

X-RAY DIFFRACTION AND FLUORESCENCE ANALYSES
OF MATERIAL LOST FROM ELECTRODE

Run	Electrode Compartment	Detected by X-Ray Diffraction		Determined by X-Ray Fluorescence (wt %)			
		Ag	Zn or Cd	Ag	Zn	or	Cd
ZG-1-Bath	Ag	Ag		99	T		
ZG-1-Bath	Zn		Zn, ZnO	1	99		
ZG-1-Irrad	Ag	Ag		98	2		
ZG-1-Irrad	Zn		Zn, ZnO	2	98		
ZG-5-Bath	Ag	Ag, ZnO		46	54		
ZG-5-Bath	Zn		No analysis made				
ZG-5-Irrad	Ag	Ag		98	2		
ZG-5-Irrad	Zn		ZnO	3	97		
ZG-6-Bath	Ag	Ag		77	23		
ZG-6-Bath	Zn		ZnO	1	99		
ZG-6-Irrad	Ag	Ag		99	T		
ZG-6-Irrad	Zn		ZnO	6	94		
CG-7-Bath	Ag	Ag, CdO		90	1.4		4
CG-7-Bath	Cd		Ag, Cd(OH) ₂	18	1.3		61
CG-7-Irrad	Ag	Ag		99			
CG-7-Irrad	Cd		Cd(OH) ₂ , CdO, Ag	16	1.0		78
CG-8-Bath	Ag	Ag, Cd(OH) ₂		99			1.0
CG-8-Bath	Cd		Cd(OH) ₂ , Ag	40			60.0
CG-8-Irrad	Ag	Ag		99			1.0
CG-8-Irrad	Cd			*			
CG-9-Bath	Ag	Ag, Ag ₂ O		99			1
CG-9-Bath	Cd		Cd(OH) ₂ , Ag	40			60
CG-9-Irrad	Ag	Ag		99			1
CG-9-Irrad	Cd		Cd(OH) ₂ , Ag	40			60

*Samples mixed

TABLE 3
EMISSION SPECTROGRAPHIC ANALYSIS OF ELECTRODE MATERIALS
(wt %)

Elements	Ag Electrode as Received	Ag Electrode After Irradiation	Zn Electrode as Received	Cd Electrode as Received
Ag	M*	M*	0.02	0.005
Al	0.01	<0.01	<0.01	0.01
B	<0.001	<0.001	<0.001	<0.001
Be	<0.001	<0.001	<0.001	<0.005
Ca	<0.01	<0.01	<0.01	0.01
Cd	<0.005	0.02	<0.005	M*
Co	<0.01	<0.01	<0.01	<0.01
Cr	<0.005	<0.005	<0.005	<0.005
Cu	0.05	0.005	0.01	0.002
Fe	0.02	0.02	0.02	0.02
Hg	ND [†]	ND [†]	2.0	-
Mg	0.001	0.001	0.001	0.004
Mn	<0.001	<0.001	<0.001	<0.005
Ni	<0.005	<0.005	<0.005	>1
Pb	<0.005	<0.005	<0.005	0.005
Si	0.05	0.01	0.01	0.01
Ti	0.005	<0.005	<0.005	<0.005
Zn	<0.01	0.2	M*	<0.05
Zr	0.01	<0.005	<0.005	ND [†]

*Indicates a major constituent

†Not detected

2. Pressure Measurements

Pressure transducers were used to monitor the pressure changes in all the irradiated cells. Ambient air was above all cells at the start of the runs. Only initial and final readings of the cell pressure were made due to equipment complications. Continuous recording is planned for the future runs. In the initial runs, only one transducer was available and thus only the irradiated cell was monitored. Runs CG-7 and CG-8 both leaked through a faulty valve; this was

corrected in Run CG-9. A pressure decrease was observed to have occurred in several runs, while one run had a pressure increase of 7.2 lb. These data are shown in Table 4.

TABLE 4
CELL PRESSURE DATA FROM Ag-Zn AND Ag-Cd RUNS

Run	Electrochemical System	Pressure Change in mm of Hg	
		Bath Run	Irradiated Run
ZG-5	Ag-Zn	NM*	-9 mm
ZG-6	Ag-Zn	NM*	-29 mm
CG-7	Ag-Cd	Leaked†	-69 mm
CG-8	Ag-Cd	Leaked†	+362 mm
CG-9	Ag-Cd	+325 mm	-196 mm

*Not measured

†Valve leaked

3. Solution Irradiations

Silver oxides are known to have a slight solubility in 40% KOH. Charged and uncharged single silver electrodes were soaked in separate compartments with aqueous KOH for various periods of time. The electrodes were then removed, and the solutions were irradiated. Material was found precipitated in the solution from the charged electrode after only 5 min or 0.1×10^6 rads exposure. The results from these and similar runs are shown in Table 5.

4. Capacity Measurements

No consistent set of capacity measurements before and after any run was obtained, and none is reported.

Equipment problems and disintegrating zinc electrodes prevented measurements from being carried out for runs ZG-1 through ZG-6. The closeness of packing of the cadmium electrodes in the present cell design has prevented the full capacity of the cadmium electrodes from being realized. This makes these cadmium electrodes capacity limiting, and prevents measurement of the silver electrode capacity.

TABLE 5

SUMMARY OF DATA ON RADIATION INDUCED PRECIPITATION FROM
VARIOUS CATHOLYTES

Pretreatment of Solution	Type of Radiation	Time	PPTe Observe	Weight of PPTe (mg)
Exposed to fully charged silver electrode for 20 hours	$\gamma @ 1.2 \times 10^6$ Rad/hr	5 min	Yes	2.9
		10 min	Yes	3.1
		210 min	Yes	5.7
Exposed to fully charged silver electrode for 40 hours	$\gamma @ 1.2 \times 10^6$ Rads	5 min	Yes	2.3
		10 min	Yes	4.0
		210 min	Yes	7.7
Exposed to uncharged silver electrode for 20 (Cell 1) and 40 (Cell 2) hours	$\gamma @ 1.2 \times 10^6$ Rads	5 min	No	
		10 min	No	
		210 min	No	
Exposed to fully charged silver electrode for 20 hours	Fluorescent light	120 hr	Yes	8.7
Exposed to fully charged silver electrode for 40 hours	Fluorescent light	120 hr	Yes	9.1
Fully charged silver electrode left in KOH	$\gamma @ 1.2 \times 10^6$ Rads	5 min	Yes	NW*
		210 min	Yes	35.2
Silver electrode charged only to point on lower plateau and left in KOH	$\gamma @ 1.2 \times 10^6$ Rads	5 min	Yes	NW*
		210 min	Yes	30.0

*NW = Not Weighed

V. DISCUSSION

A. MATERIAL LOSS FROM ELECTRODES

Material lost from the silver electrode was observed on the bottom of the cell after every irradiation. In an attempt to determine the onset of this phenomenon as a function of total dose, the irradiated cell was removed after several short exposures. It was found that 5 min (the shortest time tested) or 10^5 rads was more than enough to start the precipitation of material. No attempt was made to determine the threshold dosage more accurately or to determine the effect of dose rates on this behavior. The analyses performed on this precipitated material indicated that silver metal was the only silver species present. No material was observed to be precipitated from the electrolyte presoaked with an uncharged silver electrode nor from such solution when the uncharged silver electrode was removed prior to irradiation. However, silver was precipitated upon irradiation of the solution (alone) which had been presoaked with a charged silver electrode. These facts appear to indicate that the soluble species is a silver oxide,* and that radiation reduces silver species in solution to silver metal. Fluorescent light causes precipitation of silver from a solution presoaked with a charged silver electrode, if exposure follows removal of the electrode from the solution. If a charged electrode is soaked in 40% KOH, and the combination is exposed to light for an extended period of time, no precipitate forms. In this case, the reduced species, silver, may react with the AgO of the electrode to form Ag_2O . The fact that this does not happen in the gamma radiation field is probably due to the much faster rate of silver precipitation in this radiation field. The process of precipitation under fluorescent light was much slower than under gamma irradiation. (See Table 5.)

The amount of material lost or precipitated appears to be a function of the total dose, although the rate drops off with time. This process may be explained if one assumes that the rate of solution of silver oxide in KOH is slow. When the cells are placed in the source, the KOH is probably near saturation with respect to the oxides of silver. These immediately undergo radiolytic reduction

*No attempt has been made as yet to determine what the form of the dissolved species is likely to be, i.e., hydroxide, oxide, etc., or whether the initial material is a true solution or a colloid. Also, it is not known if electrochemically prepared Ag oxides are different from those chemically prepared.

and form the precipitate. Thus, the subsequent rate of precipitation is controlled by the rate at which additional oxides dissolve.

B. GAS EVOLUTION

A wide variation in the measured final pressure was observed in these runs. This scatter is not understood at present. The pressure decrease observed in the Ag-Zn runs may be explained by the reaction of zinc with the oxygen from the air over the cells. Gas chromatographic analysis on future runs may resolve this question.

The difference in pressures observed in Runs CG-7 and -8 may be explained by the fact that the same cadmium electrodes were used for both runs. In the second run, the cadmium electrode appears to have lost some of its ability to react with the oxygen. The pressure decrease observed in Run CG-7 is unexplained at present.

C. CAPACITY MEASUREMENTS

As yet no successful capacity measurements on the silver electrode have been made. A new cell liner design and capacity measuring system is planned for the next phase of the program involving these features: (1) triple compartments, with the center compartment for the silver electrode, and the outer two for parallel connected cadmium or zinc electrodes; (2) the compartments will be separated with medium pore fritted quartz discs; and (3) the silver electrode will be physically cut to reduce its size and thus its capacity to make it the limiting electrode in the cell.

The arrangement should permit amp-hr capacity measurements to be made on the Ag electrode with little difficulty.

D. RECOMMENDATION FOR FUTURE WORK

The new cadmium electrodes will be available for the next phase of this study. Incorporation of two of these cadmium electrodes in parallel, one on either side of the silver electrode, should permit the accurate measurement of the silver electrode capacity. These measurements will be carried out at two states of charge, namely, 90% and 60% of the full capacity of the silver electrode. Any effect of radiation on the silver electrode capacity should be studied as a function

of total dose and dose rate, and other studies should be carried out to elucidate the mechanism of any effects observed.

A search will be made to obtain sturdier zinc electrodes so that their characteristics in the radiation field may be studied. This study should examine any effect of radiation on the capacity, the dendrite growth pattern, and on the deterioration processes in general occurring with irradiated zinc electrodes. The parameters of importance for this phase of the study should be state of charge, total dose, and dose rate.

Continuous pressure measurements will be made on both the bath and irradiated cells in this second phase of the study. Gas chromatographic analyses of the gases left in the cells will be made. From the results of the gas analyses, and with a consistent pattern of pressure changes during the irradiations, the mechanisms of the gas generation and utilization processes should be explored.

An attempt should be made to determine the nature of the dissolved silver oxide(s), their rate of dissolution in KOH, and the rate and mechanism of their subsequent precipitation by the gamma radiation. If an additional process is at work leading to material loss from the silver electrodes, this should be determined and its mechanism worked out.

REFERENCE

1. "The Effects of Radiation on Nickel-Cadmium Battery Electrodes. I.," Final Report, June 1963 to April 1965, AI-65-66.