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A PROGRAM TO DEVELOP A HIGH-ENERGY DENSITY PRIMARY BATTERY WITH A MINIMUM OF 200 WATT HOURS PER POUND OF TOTAL BATTERY WEIGHT

by

William E. Elliott, James R. Huff, Gerald L. Simmons,
Guy D. McDonald, Judith L. Jamrozy and Warren L. Towle

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SEVENTH QUARTERLY REPORT
January 1 - March 31, 1966

A RESEARCH REPORT FROM

GLOBE-UNION INC.

ELECTRONICS BATTERY SPECIAL PRODUCTS

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Technical Management
Space Power Systems Division
National Aeronautics and Space Administration
Lewis Research Center, Cleveland, Ohio
Mr. Robert B. King

GLOBE-UNION INC.
Applied Research
900 East Keefe Avenue
Milwaukee, Wisconsin 53201

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* "As received" $LiPF_6$.

SUMMARY

During the Seventh Quarter, investigation of the Li/LiClO$_4$-propylene carbonate (PC)/CuF$_2$, Li/LiPF$_6$-N-nitrosodimethylamine (NDA)/CuF$_2$ and Li/LiPF$_6$-NDA/AgO systems was emphasized. Controlled current half-cell testing of CuF$_2$ and AgO cathodes was used to evaluate factors in cathode construction such as substrate, conductive additives, fabrication pressure and temperature, and the weight ratio of active material to substrate. The tests showed that the weight ratio of active material to substrate could be increased from 2:1 to 10:1 without significant increase in IR losses. A dependence of cathode performance on fabrication pressure was also noted. Low pressures result in poor mechanical integrity while pressures which are too high create an impenetrable electrode face. For this series of tests, a pressure of 2,000 lb/cm^2 appeared to be the optimum.

Constant current voltammetric studies yielded information about cathode performance in different electrolytes. Best results were obtained in the LiPF$_6$-NDA electrolyte. Cupric fluoride cathodes discharged vs. lithium in excess amounts of 1.0 m LiPF$_6^{*}$-NDA and 1.0 m LiClO$_4$-PC yielded coulombic efficiencies of 42.7 and 35.2%, respectively. Average cell voltage was 1.3 V for the LiPF$_6^{*}$-NDA system and 1.18 V for the LiClO$_4$-PC system. It was also found that solute purity significantly affected cathode performance in the LiPF$_6$-NDA electrolyte. In constant current discharges of full cells, CuF$_2$ and AgO cathodes operated at more positive voltages and yielded higher coulombic efficiencies when the LiPF$_6$-NDA electrolyte was prepared from "as received" LiPF$_6$. For example, cupric fluoride cathodes discharged in 1.0 m LiPF$_6$-NDA yielded coulombic efficiencies of 99.9% and 42.7% for the "as received" and dried solutes, respectively. Average cell voltage was 0.4 V more positive for the LiPF$_6^{**}$-NDA system. A second effect noted for the LiPF$_6^{**}$-NDA electrolyte was that reducing

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* The LiPF$_6$ was dried at 110°C in a nitrogen atmosphere before use.

** "As received" LiPF$_6$.

the volume of electrolyte decreased coulombic output considerably. This was not the case when dried $LiPF_6$ was used. Apparently, some component of the $LiPF_6$-NDA electrolyte, removed by drying the $LiPF_6$, is involved in the electrochemical reaction. This hypothesis was confirmed by full cell discharges with inert working electrodes and by linearly varying potential (LVP) studies.

The organic cathode material, trichloroisocyanuric acid (ACL-85) was also investigated. Attempts were made to fabricate a pellet-type ACL-85 cathode to eliminate the problems associated with dissolved cathodes, i.e., diffusion limitation and filming of the working electrode. The use of pellet-type cathodes is dependent on finding a suitable electrolyte in which the cathode would be sparingly soluble. Solubility studies were initiated to find such an electrolyte.

LVP scans were made of Li, AgO, and $Ni(OH)_2$ electrodes. The LVP technique was also used to investigate various dimethylformamide, N-nitrosodimethylamine, and propylene carbonate electrolytes for possible decomposition. One molal solutions of KPF_6 and $LiPF_6$* in NDA produced reduction peaks in the -1.0 to -2.0 V range.

Stability tests were conducted for Li, CuF_2, and AgO in 1.0 m $LiPF_6$-NDA prepared from "as received" and dried solutes. Aside from partial solubility of AgO and CuF_2 in the electrolytes, no extreme changes were noted. Specific conductivities of the solutions were fairly constant.

Several separators were studied for use with the $Li/LiPF_6$-NDA/CuF_2 and $Li/LiPF_6$-NDA/AgO systems. These included microporous rubber, Versapor 6429 epoxy filters (Gelman), and glass fiber (Gelman). The latter seemed most promising because of its low resistance, high percent void volume, and stability in the electrolyte.

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* "As received" $LiPF_6$.

INTRODUCTION

The objective of this research is to develop a primary battery with an energy density of at least 200 watt-hours per pound of total battery weight.

Preliminary evaluations have been made of various anode-electrolyte and cathode-electrolyte combinations using controlled current voltammetry and the linearly varying potential method. This has led to the selection of several complete systems which will be subjected to a more thorough investigation.

In the future, emphasis will be placed on further characterization of these systems. Concurrently, the compatibility of electrode materials with the electrolytes and losses on standing will be investigated.

A PROGRAM TO DEVELOP A HIGH-ENERGY DENSITY PRIMARY BATTERY WITH A MINIMUM OF 200 WATT HOURS PER POUND OF TOTAL BATTERY WEIGHT

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ABSTRACT

Characterization of electrode reactions using the linearly varying potential technique is described for lithium anodes and nickel hydroxide and silver oxide cathodes.

Controlled current voltammetric studies of cupric fluoride cathodes were conducted in a variety of electrolytes. Seventy-two constant current discharges of full cells containing cobaltic fluoride, cupric fluoride, and silver oxide cathodes in $LiClO_4$-propylene carbonate and $LiPF_6$-N-nitrosodimethylamine electrolytes were completed.

Limited testing of the organic cathode material, trichloroisocyanuric acid, was undertaken.

Linearly varying potential scans of dimethylformamide, N-nitrosodimethylamine, and propylene carbonate electrolytes are presented to illustrate the extent of electrolyte interaction in full cell discharges. Compatibility studies for lithium, cupric fluoride, and silver oxide in $LiPF_6$-N-nitrosodimethylamine are described.

I. OVERALL PROGRESS

I. OVERALL PROGRESS

The experimental work during the Seventh Quarter encompassed work in the following areas:

- A. Anodes.
  - 1. Linearly varying potential studies.
- B. Inorganic Cathodes.
  - 1. Voltammetric studies at controlled current.
    - a. Half-cell testing.
    - b. Coulombic efficiency studies.
  - 2. Linearly varying potential studies.
- C. Organic Cathodes.
  - 1. Voltammetric studies at controlled current.
    - a. Half-cell testing.
    - b. Coulombic efficiency studies.
- D. Electrolytes.
  - 1. Linearly varying potential studies of electrolyte decomposition.
  - 2. Chemical stability of electrode materials in electrolyte solutions.
- E. Separators.

A. Anodes.

1. Linearly Varying Potential Studies.

This quarter, several systems were studied intensively in preparation for assembling a prototype battery. These systems consisted of lithium anodes, cupric fluoride and silver oxide cathodes, and $LiClO_4$-propylene carbonate (PC) and $LiPF_6$-N-nitrosodimethylamine (NDA) electrolytes. The $LiPF_6$-NDA electrolytes were prepared from "as received" and dried* $LiPF_6$. Constant current discharges of full cells in the "as received" $LiPF_6$ electrolyte in some cases yielded coulombic efficiencies in excess of 100%. It was postulated that an impurity removed in drying $LiPF_6$ was contributing to the discharge.

A series of linearly varying potential (LVP) scans were made of Li electrodes in 1.0 m $LiClO_4$-PC and 1.0 m $LiPF_6$-NDA. (The LVP technique is described in the Fourth Quarterly Report.) These are depicted in Figures 1-a through 1-e, pages IV-1 through IV-6**. A comparison of Li electrodes in 1.0 m $LiPF_6$("as received")-NDA and 1.0 m $LiPF_6$(dried)-NDA is depicted in Figure 1-a, page IV-2. In both electrolytes, a single oxidation wave is present in the -3.0 to -2.0 V range. The wave occurs approximately 0.5 V more positive in the dried $LiPF_6$ electrolyte.

A scan of Li in 1.0 m $LiClO_4$-PC is depicted in Figure 1-b, page IV-3. The oxidation wave for Li occurs at more positive voltages, i.e. -2.0 to -1.0 V, than in the $LiPF_6$-NDA electrolytes. In addition, the slope of the curve is not as steep. This could be attributed to filming of the anode surface, diffusion limitation, or slower reaction rate in the $LiClO_4$-PC electrolyte.

LVP scans of Li electrodes which had been exposed to 1.0 m $LiPF_6$-NDA for approximately twenty-four hours are shown in Figures 1-c and 1-d, pages IV-4 and IV-5. Li performance

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* $LiPF_6$ was dried by heating to 110°C in a nitrogen atmosphere.

** The interpretation of these scans should be based on the initial slope only. The anode areas were not equal in every case, thus causing differences in peak intensity.

was significantly affected only in the dried $LiPF_6$ electrolyte. The oxidation wave was shifted to more positive voltages after the twenty-four hour exposure period.

Further LVP studies of Li electrodes are planned to confirm the results obtained thus far.

B. Inorganic Cathodes.

1. Voltammetric Studies at Controlled Current.

a. Half-Cell Testing.

Two series of half-cell tests were run this quarter. The first was designed to evaluate cathode construction and cathode performance in various electrolytes.* The second series showed the effect of increasing the weight ratio of active material to substrate from 2:1 to 10:1.

Half-cell testing of cupric fluoride cathodes gave the following information about cathode construction:

(1) Substrate. A comparison of Nos. 5 and 6, Table I, pages IV-7 through IV-13, shows that the cathode pressed on copper screen operated at voltages 0.1 to 0.3 V more positive than the cathode pressed on silver expanded metal.

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* A comparison of Ag and Ag/AgCl reference electrodes was also included in these tests. Deterioration of Ag/AgCl reference electrodes in certain electrolytes required a change to Ag reference electrodes. Comparing Nos. 3a and 3b, Table I, pages IV-7 through IV-13, it can be seen that voltage readings vs. Ag were 0.1 to 0.2 v more positive than the Ag/AgCl readings.

(2) Method of Fabrication., i.e., hot- or cold-pressing. Cathode No. 9, Table I, was pressed on the substrate without the application of heat. A hot-pressed cathode tested in the same electrolyte during the Sixth Quarter operated at voltages approximately 0.2 V more positive than the cold-pressed electrode.

(3) Conductive Additives. A comparison of Nos. 4 and 5, Table I, indicates that the addition of acetylene black to copper fluoride results in a more positive open circuit voltage and less polarization under load.

The first series of tests was also used to evaluate cathode behavior in various electrolytes. Cupric fluoride cathodes polarized less in 1.0 m $LiPF_6$-N-nitrosodimethylamine (NDA) than in 1.0 m $LiClO_4$-propylene carbonate (PC). Compare Nos. 2 and 5, Table I at current densities less than 10 ma/cm^2. (Above this current density the tests were conducted in different ways.) At a discharge current of 7.2 ma/cm^2, the cathode in $LiClO_4$-PC polarized to -0.99 V. At the same current density, the cathode in $LiPF_6^*$-NDA was operating at -0.03 V with slight polarization. Further tests indicated that the behavior of cupric fluoride in $LiPF_6$-NDA is dependent upon whether the $LiPF_6$ is dried in a nitrogen atmosphere or used "as received." A cupric fluoride cathode tested in $LiPF_6$("as received")-NDA could sustain 10 ma/cm^2 before polarizing to -1.03 V. In the $LiPF_6$(dried)-NDA electrolyte, polarization to -0.99 V occurred during the 3 ma/cm^2 discharge (See Nos. 7 and 8, Table I).

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* "As received" $LiPF_6$.

Further evaluation of cathode construction and of cathode behavior in different electrolytes was accomplished by constant current voltammetric tests. These are discussed in the next section.

In assembling a battery it would be necessary to increase the weight ratio of active material to substrate thus increasing theoretical watt-hours per pound of total battery weight. A series of half-cell tests was run to determine whether this increase in weight of active material would cause significant IR losses. The data, which are presented in Table II, pages IV-14 through IV-17, indicate that increasing the active material to substrate ratio does not cause severe IR losses for cupric fluoride cathodes. One silver oxide cathode with a 10:1 active material to substrate ratio was tested. Polarization was not excessive in two minute discharges at current densities as great as 20 ma/cm^2.

Other factors affecting electrochemical performance in these tests were fabrication pressure and temperature. Cupric fluoride cathodes were pressed at 500, 1,000, 2,000, and 3,000 lb/cm^2. Cathodes pressed at 2,000 lb/cm^2 gave the best overall performance (compare Nos. 2 and 3 and Nos. 6 and 7, Table II). A comparison of Nos. 2 and 10, Table II, shows that the cathode pressed at ambient temperature polarized less than a hot-pressed electrode at the same current densities. This behavior is not consistent with previous testing and will have to be confirmed.

b. Coulombic Efficiency Studies.

Constant current* voltammetry was used to evaluate cathodes under full cell conditions. These "coulombic efficiency"

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* Constant current was maintained in most cases by a transistorized variable resistor apparatus. When a power supply was used, the term "forced discharge" is applied.

tests provided information about the active materials, cathode construction, and electrolyte effects. This quarter seventy-two coulombic efficiency tests were completed for cobalt trifluoride, cupric fluoride, and silver oxide cathodes vs. lithium in various electrolytes. The results are presented in Table III, pages IV-18 through IV-29. Plots of cathode and cell voltage vs. percent theoretical coulombs for selected tests are depicted in Figure 2, pages IV-31 through IV-42.

Comparison of CoF_3, CuF_2, and AgO cathodes are depicted in Figures 2-a and -b, pages IV-31 and IV-32. Cold-pressed CoF_3 and CuF_2 cathodes containing acetylene black (3 wt. %) and Solka-Floc (3 wt. %) were discharged in excess amounts of 1.0 m $LiPF_6^*$-NDA at approximately 2 ma. Coulombic efficiencies were 129.1% and 12.1%, respectively. CuF_2 (97 wt. % CuF_2, 3 wt. % acetylene black) and AgO (97 wt. % AgO, 3 wt. % Solka-Floc) cathodes discharged in minimal amounts of 1.0 m $LiPF_6^*$-NDA at approximately 2 ma yielded efficiencies of 33.6% and 79.0%, respectively. In general, AgO cathodes were superior to CuF_2 cathodes.

Coulombic efficiency tests were run in 1.0 m $LiClO_4$-PC and $LiPF_6$-NDA electrolytes. The $LiPF_6$-NDA electrolytes were prepared from "as received" and dried solutes. In Figure 2-c, page IV-33, discharges of hot-pressed CuF_2 cathodes (97 wt. % CuF_2, 3 wt. % acetylene black) in the three electrolytes are depicted. Coulombic efficiencies were 99.9%, 42.7%, and 35.2% in $LiPF_6^*$-NDA, $LiPF_6^{**}$-NDA, and $LiClO_4$-PC, respectively. These results show the

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* "As received" $LiPF_6$.

** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

superiority of the $LiPF_6$-NDA electrolyte. They also indicate the presence of a reducible impurity in the $LiPF_6$-NDA electrolyte presumably removed in drying the $LiPF_6$.

To determine whether the impurity could be water, water was placed on the surface of a CuF_2 cathode (97 wt. % CuF_2, 3 wt. % acetylene black) during a discharge in 1.0 m $LiPF_6^*$-NDA. This caused a temporary increase in cathode voltage but did not result in the high efficiencies obtained when "as received" $LiPF_6$ was used (see Figure 2-d, page IV-34).

To further substantiate the presence of an electroactive contaminant, tests were run in minimal amounts (ca. 3 ml) of electrolyte. As can be seen in Figures 2-e through 2-h, pages IV-35 through IV-38, decreasing the amount of $LiPF_6^{**}$-NDA from 15 to 3 ml decreased the coulombic efficiencies obtained for CuF_2 and AgO cathodes by more than 50%. The drop in coulombic efficiency was much less for $LiClO_4$-PC and $LiPF_6^*$-NDA.

Discharges of inert silver and carbon working electrodes vs. lithium in $LiPF_6^{**}$-NDA were also run (see pages IV-30 and IV-43). The Li/1.0 m $LiPF_6^{**}$-NDA/Ag system produced 106.4 coulombs at an average current density of 1 ma/cm^2. Two cell voltage plateaus are present at 2.2 and 1.6 V. Coulombic output and operating voltages for the Li-carbon system were considerably lower. Again, the presence of an electroactive impurity seems highly probable. It is

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* The $LiPF_6$ was dried in a nitrogen atmosphere before use.

** "As received" $LiPF_6$.

unlikely that the solvent is being reduced since 15 ml of NDA would theoretically produce 4×10^4 coulombs assuming a two electron reduction. An intensive investigation to determine the nature of the impurity is underway.

2. Linearly Varying Potential Studies.

During the sixth quarter, a cold-pressed nickel hydroxide cathode (80 wt. % $Ni(OH)_2$, 10 wt. % acetylene black, 10 wt. % Solka-Floc) was tested in a full cell discharge vs. lithium in $LiClO_4$-propylene carbonate. This cathode yielded a coulombic efficiency of 21.4% at a current density of 1.5 ma/cm^2. Average cell voltage was only 1.2 V.

This quarter, a nickel hydroxide thin film electrode was prepared by depositing $Ni(OH)_2$ on nickel metal from a 1.0 M $Ni(NO_3)_2$ solution. Linearly varying potential scans were made of both types of $Ni(OH)_2$ cathodes to determine whether the thin film electrode would exhibit better electrochemical characteristics.

Linearly varying potential scans of the cold-pressed $Ni(OH)_2$ electrode in 1.0 m $LiPF_6^*$-NDA exhibited reduction waves in the -1.0 and -3.0 V regions. Scans of the $Ni(OH)_2$ thin film in 1.0 m $LiClO_4$-PC contained waves at -1.2 and -2.0 V. A comparison of the scans of $Ni(OH)_2$ with linearly varying potential scans of $LiPF_6^*$-NDA and $LiClO_4$-PC indicates that the second reduction waves for both $Ni(OH)_2$ electrodes can probably be attributed to reduction of an electrolyte component.

The linearly varying potential scans of $Ni(OH)_2$ cathodes are depicted in Figures 4-a and -b, pages IV-45 and IV-46.

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* "As received" $LiPF_6$.

Linearly varying potential scans were also made of hot-and cold-pressed silver oxide* electrodes in 1.0 molal $LiPF_6^{**}$-NDA. These are depicted in Figure 4-c, page IV-47. Two reduction waves are present in the scans of both electrodes. The first waves occur in the 0.0 to -0.5 V range. Peak cathodic current of the initial wave was higher for the hot-pressed AgO electrode. Comparison with scans of 1.0 m $LiPF_6^{**}$-NDA indicates that again the second reduction waves can probably be attributed to reduction of an electrolyte component.

C. Organic Cathodes.

1. Voltammetric Studies at Controlled Current.

a. Half-Cell Testing.

Past testing of organic cathode materials has been carried out almost exclusively with the cathode material dissolved in the electrolyte. However, the electrochemical performance of these dissolved compounds might be limited by diffusion or by filming of the inert current collector. These problems could possibly be eliminated by using a solid organic electrode.

Three pellet-type organic electrodes were prepared from mixtures of trichloroisocyanuric acid (ACL-85)(94 wt. %), acetylene black (3 wt. %), and Solka-Floc (3 wt. %). Half-cell tests were run in $AlCl_3$-acetonitrile, $LiPF_6^{**}$-dimethylformamide, and $LiPF_6^{**}$-N-nitrosodimethylamine electrolytes. The cathodes disintegrated during the tests because of their solubility in the electrolytes. Open circuit voltages were nearly one volt more negative than those obtained for dissolved ACL-85 (ca. + 0.9 V). The results of these tests are given in Table IV-A, pages IV-49 and IV-50.

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* For preparation see Jacob Kleinberg, <u>Inorganic Synthesis</u>, Vol. 4, page 12. Analysis of the compound showed that the sample contained 84.96 wt. % Ag. Ag_2O and AgO contain 93.1 and 87.1 wt. % Ag, respectively.

** "As received" $LiPF_6$.

Because ACL-85 was highly soluble in the electrolytes mentioned above, an investigation was initiated to find suitable electrolytes in which solid organic cathodes would be sparingly soluble. ACL-85 was added to acetonitrile, N-methylformamide, dimethylformamide, N-nitrosodimethylamine, ethylene carbonate-propylene carbonate (80/20 wt. %), propylene carbonate, and methylene chloride. No reaction occurred with the latter three solvents. The stability of ACL-85 in the other solvents was at least doubtful. The solubility of ACL-85 in ethylene carbonate-propylene carbonate (EC-PC) and propylene carbonate (PC) was 0.3 g/cc and 0.2 g/cc, respectively, at ambient temperature. ACL-85 was insoluble in methylene chloride (MC). The results of adding ACL-85 to various PC and MC electrolytes are given below:

Electrolyte (1 Molal)		Solubility of ACL-85 in the Electrolyte at Ambient Temperature (ca. 25°C)	Observations
Solvent	Solute		
MC	$AlCl_3$	Slightly Soluble.	A milky white solution formed on addition of ACL-85.
MC	LiCl	Insoluble.	No reaction.
MC	$(CH_3)_4NCl$	Insoluble.	No reaction.
MC	$(C_4H_9)_4NCl$	------	Gassing and precipitate formation occurred when ACL-85 was added.
MC	$(C_6H_5)_4PCl$	Soluble.	The solution turned yellow when ACL-85 was added.
MC	$(C_4H_9)_4NI$	------	Gassing and precipitate formation occurred when ACL-85 was added.
PC	LiCl	0.06 g/cc	No reaction.

Half-cell screening of soluble organic cathodes was also continued this quarter. ACL-85 was tested at two concentrations in $LiPF_6^*$-NDA using HP-10 Speer carbon working

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* The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

and counter electrodes. A saturated (<0.2 m) ACL-85 system could sustain a discharge of at least 10 ma/cm^2 before polarizing to voltages more negative than -1.0 V (vs. Ag). A 0.01 molal ACL-85 system polarized rapidly at a discharge rate of 2.5 ma/cm^2. A comparison of ACL-85 at different concentrations in LiPF$_6$-NDA and -DMF is given below. The half-cell tests for ACL-85 in LiPF$_6$-DMF were described in the Sixth Quarterly Report.

Concentration of ACL-85 in the Electrolyte	Electrolyte (1 Molal)	Working and Counter Electrodes	Reference Electrode*	Current Density Which Could Be Maintained Before the Cathode Voltage Became More Negative Than -1.0 V
0.01	LiPF$_6^{**}$-NDA	HP-10 Speer Carbon	Ag	0.5 ma/cm^2
Sat. (<0.2 m)	LiPF$_6^{**}$-NDA	"	Ag	10 ma/cm^2
0.1	LiPF$_6^{***}$-DMF	"	Ag/AgCl	2.5 ma/cm^2
****1.0	LiPF$_6^{***}$-DMF	"	Ag/AgCl	40 ma/cm^2

The results of the half-cell screening of dissolved organic cathodes are presented in Table IV-B, pages IV-51 and IV-52.

b. Coulombic Efficiency Studies.

The coulombic output of ACL-85 at varying concentrations in 1 molal AlCl$_3$-acetonitrile (AN) and LiPF$_6^{**}$-dimethylformamide (DMF) electrolytes was determined this quarter. Magnesium and

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* For a comparison of voltage measurements vs. Ag and Ag/AgCl see page 3.

** The LiPF$_6$ was dried at 110°C in a nitrogen atmosphere before use.

*** "As received" LiPF$_6$.

**** Gas evolution occurred in the electrolyte initially and during the discharge.

lithium anodes were utilized in $AlCl_3$-AN and $LiPF_6$-DMF, respectively. Speer carbon working electrodes and silver reference electrodes were employed. The discharges were held at constant current with a power supply. Average current densities ranged from 1 to 5 ma/cm^2. The highest coulombic efficiency obtained this quarter was 22.0%* for 0.01 molal ACL-85 discharged at approximately 1 ma/cm^2 in $LiPF_6$**-DMF. The percent efficiency was calculated to a cut-off cell voltage of 0.2 V.

The results of the ACL-85 coulombic efficiency tests are given in Table V, pages IV-53 through IV-55. Two previously reported tests, in which $LiPF_6$**-N-nitrosodimethylamine electrolytes were utilized, are included for comparison purposes.

D. Electrolytes.

1. Linearly Varying Potential Studies of Electrolyte Decomposition.

Constant current voltammetric studies have shown that in some cases reduction of an electrolyte component may be contributing to the number of coulombs obtained. For this reason, LVP studies were made of electrolytes to determine whether reduction would occur in the voltage range of interest, i.e., approximately -1.0 to -2.0 V.

This quarter a series of LVP scans was made of dimethylformamide (DMF), N-nitrosodimethylamine (NDA), and propylene carbonate (PC) solutions of various solutes. These are depicted in Figures 5-a through 5-j, pages IV-56 through IV-68. Inert, nonporous pyrolytic graphite working electrodes were used in most cases. Previous LVP studies have shown that the use of metallic

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* Calculated on the basis of a 6 electron change per molecule.

** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

working electrodes often causes waves due to metallic oxide reduction (see Figures 5-i and 5-j, pages IV-67 and IV-68). Porous carbon electrodes are unsuitable because the effective surface area is not reproducible.

Of the electrolytes scanned, only 1.0 m $LiPF_6^*$- and KPF_6-NDA exhibited significant reduction peaks in the -1.0 to -2.0 V range. The KPF_6-NDA peak was located at -1.5 V and the $LiPF_6^*$-NDA peak at -1.4 V. Peak intensities were in excess of 10 ma/cm^2 (see Figure 5-g, page IV-65).

Further LVP studies were made of the $LiPF_6^*$-NDA electrolyte which has been used extensively in recent electrochemical testing. It has been shown that some impurity, removed in drying the solute, is electrochemically active. The LVP scans depicted in Figures 6-a through 6-e, pages IV-69 through IV-74, were made to determine whether this impurity could be water. As can be seen from Figure 6-a, page IV-70, the addition of water to 1.0 m $LiPF_6^*$-NDA intensifies only the second peak at -2.4 V. The -1.4 V peak is of primary interest since reduction at this voltage could significantly affect full cell discharges. Figures 6-c and 6-d, pages IV-72 and IV-73, show the effect of adding water "scavengers," i.e., Li and P_2O_5, to the electrolyte. Again, the second reduction peak is affected.

A comparison of LVP scans of $LiPF_6 \cdot H_2O$-NDA and $LiPF_6$-NDA prepared from "as received" and dried solutes is shown in Figure 6-e, page IV-74.

Further testing of the $LiPF_6$-NDA electrolyte is planned for the future to confirm results obtained this quarter and to identify the cause of the first reduction wave.

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* "As received" $LiPF_6$.

2. Chemical Stability of Electrode Materials in Electrolyte Solutions.

Because of the emphasis on the Li/$LiPF_6$-NDA/CuF_2 and Li/$LiPF_6$-NDA/AgO systems, stability tests are being carried out for the electrode materials in pure NDA and in one molal $LiPF_6$-NDA solutions prepared from "as received" and dried solutes. Changes in specific conducitivity during a four week period were recorded for the electrolytes exposed to Li, CuF_2, and AgO. These conductivity measurements, together with visible observations of change, are presented in Table VI, pages IV-75 through IV-80. Specific conductivities were fairly constant over a four week period indicating that the electrode materials did not react extensively with the electrolytes. However, shelf-life tests for complete battery systems are necessary to confirm the electrochemical stability of the electrode materials.

The stability of lithium in methylene chloride was also studied over a two week period. Methylene chloride is presently being investigated for use with organic cathodes because of their low solubility in the solvent. A piece of lithium remained unchanged after two weeks exposure to MC.

E. Separators.

This quarter, a Versapor 6429 epoxy membrane (Gelman) was tested for use with dissolved cathode systems using the procedure described in the Sixth Quarterly Report. The cell used to test the membrane consisted of a lithium anode, a $CuCl_2 \cdot 2H_2O$ dissolved cathode, a copper working electrode, and a 1.0 m $(C_6H_5)(CH_3)_3NPF_6$-dimethylformamide electrolyte. The membrane did not prevent copper migration and thus proved unsatisfactory. X-ray diffraction analysis of the anode after discharge showed that copper had been deposited on the lithium surface. The results of this test are given in Table VII, pages IV-81 and IV-82.

Separators were also investigated for use in the Li/$LiPF_6$-NDA/CuF_2 and Li/$LiPF_6$-NDA/AgO systems in which the cathode is not dissolved. Separators for these systems must be stable in $LiPF_6$-NDA and must prevent anode attack. In addition, they must possess the following characteristics: low density, high percent void volume, and low resistance.

A list of the separators tested is given below:

Separator	Thickness (cm)	Weight (g/cm^2)	Volume of Electrolyte Absorbed Per cm^2*
1. Glass fiber (Gelman)	0.004	0.01	0.06
2. Versapor 6429 epoxy (Gelman)	0.006	0.02	0.01
3. Microporous rubber	0.011	0.05	0.11
4. Microporous rubber	0.020	0.09	0.18

All of the materials appeared to be stable in 1.0 m $LiPF_6^{**}$-NDA. Testing of the separators was accomplished by clamping a soaked separator between the electrodes and measuring the voltage drop from open circuit as a 2 to 5 ma/cm^2 load was applied. Resistance followed the order: microporous rubber > Versapor 6429 epoxy > glass fiber. Because of its low density, high void volume, and low resistance, glass fiber appears to be the most promising separator material. Full cell testing of complete battery systems and shelf-life tests will be necessary to evaluate the separating characteristics of the materials.

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* Approximate values.

** "As received" $LiPF_6$.

II. CURRENT PROBLEMS

II. CURRENT PROBLEMS

The major problem will be that of characterizing electrode materials and electrolytes to be used in complete battery systems.

III. WORK TO BE PERFORMED

III. WORK TO BE PERFORMED

A. During the Next Quarter.

During the next quarter, studies of Li/AgO and Li/CuF_2 systems will be continued. Of particular interest are the Li/AgO and Li/CuF_2 couples in a $LiPF_6$-N-nitrosodimethylamine electrolyte. The Mg/$AlCl_3$-acetonitrile/CuF_2 system and several organic cathode systems (trichloromelamine, trichloroisocyanuric acid) will also be investigated.

Experimentation will be divided into the following areas:

1. Further characterization of electrode materials and electrolytes.

2. Compatibility studies of electrode materials and battery components in the electrolyte.

3. Electrochemical testing of complete systems.

B. During the Next Month.

Emphasis will be placed on further study of the Li/$LiPF_6$-NDA/CuF_2 and Li/$LiPF_6$-NDA/AgO systems. This will involve efforts in the following areas:

1. Further characterization of electrode materials and the electrolyte.

2. Compatibility studies of electrode materials and battery components in the electrolyte.

3. Electrochemical testing of complete systems.

The investigation of organic cathode systems will be continued on a limited basis.

IV. TEST RESULTS

FIGURE 1. LINEARLY VARYING POTENTIAL STUDIES OF LITHIUM ANODES

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* "As received" and dried $LiPF_6$.

** "As received" $LiPF_6$.

*** Dried $LiPF_6$.

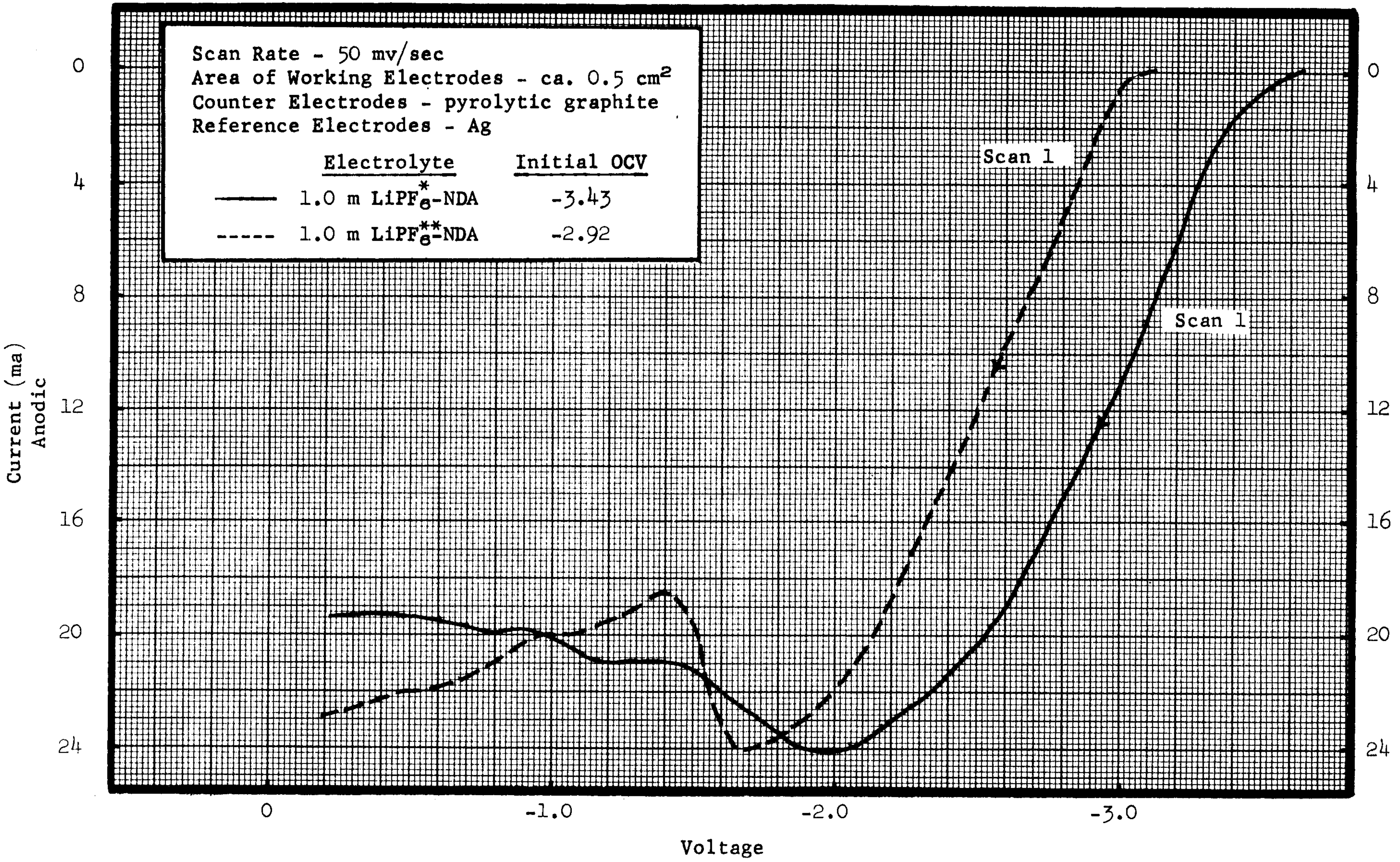

FIGURE 1. LINEARLY VARYING POTENTIAL STUDIES OF LITHIUM ANODES

Figure 1-a. The Effect of Drying the $LiPF_6$ Solute.

* "As received" $LiPF_6$.

** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

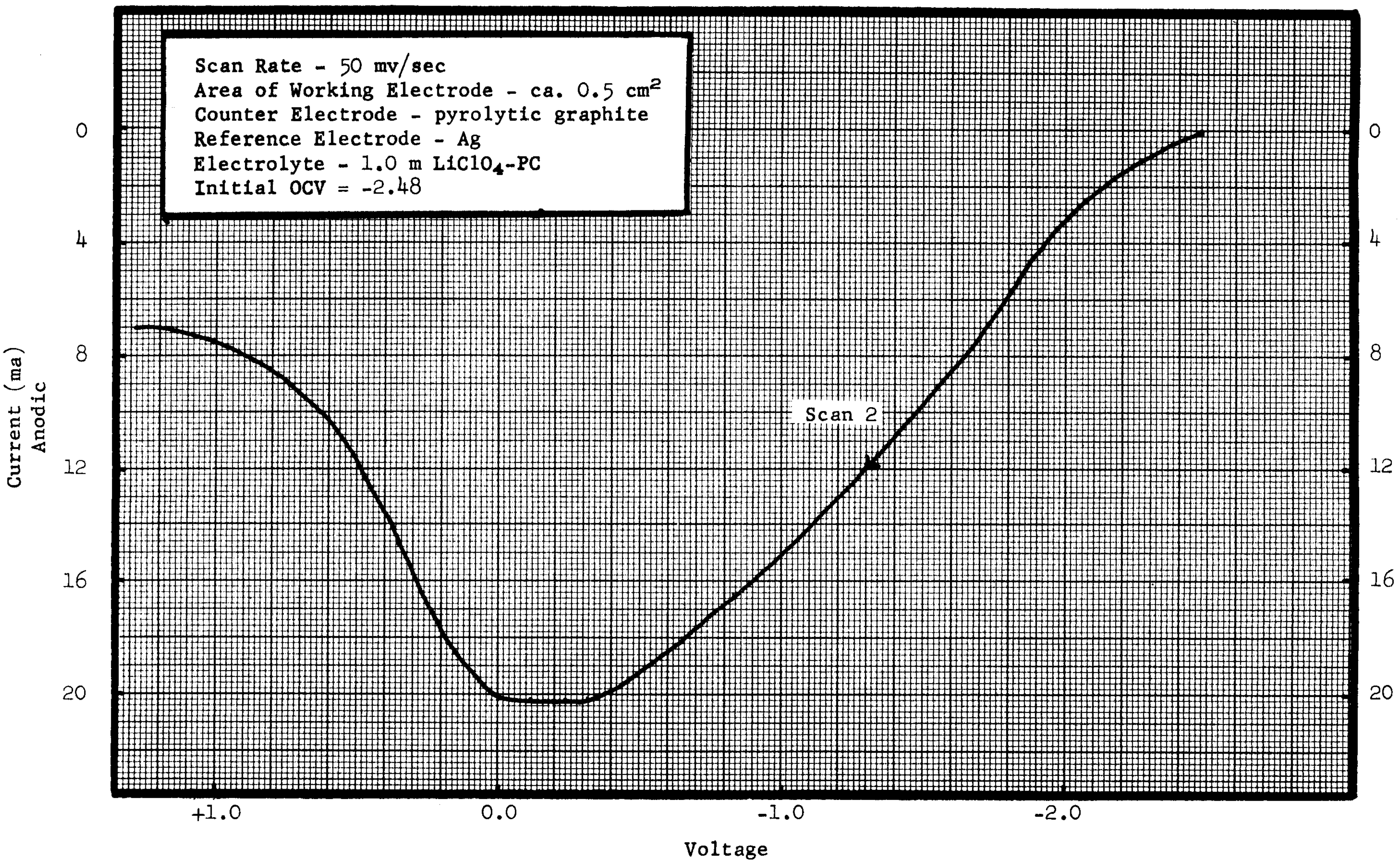

FIGURE 1. LINEARLY VARYING POTENTIAL STUDIES OF LITHIUM ANODES (Continued)

Figure 1-b. The Effect of Using a LiClO$_4$-Propylene Carbonate Electrolyte.

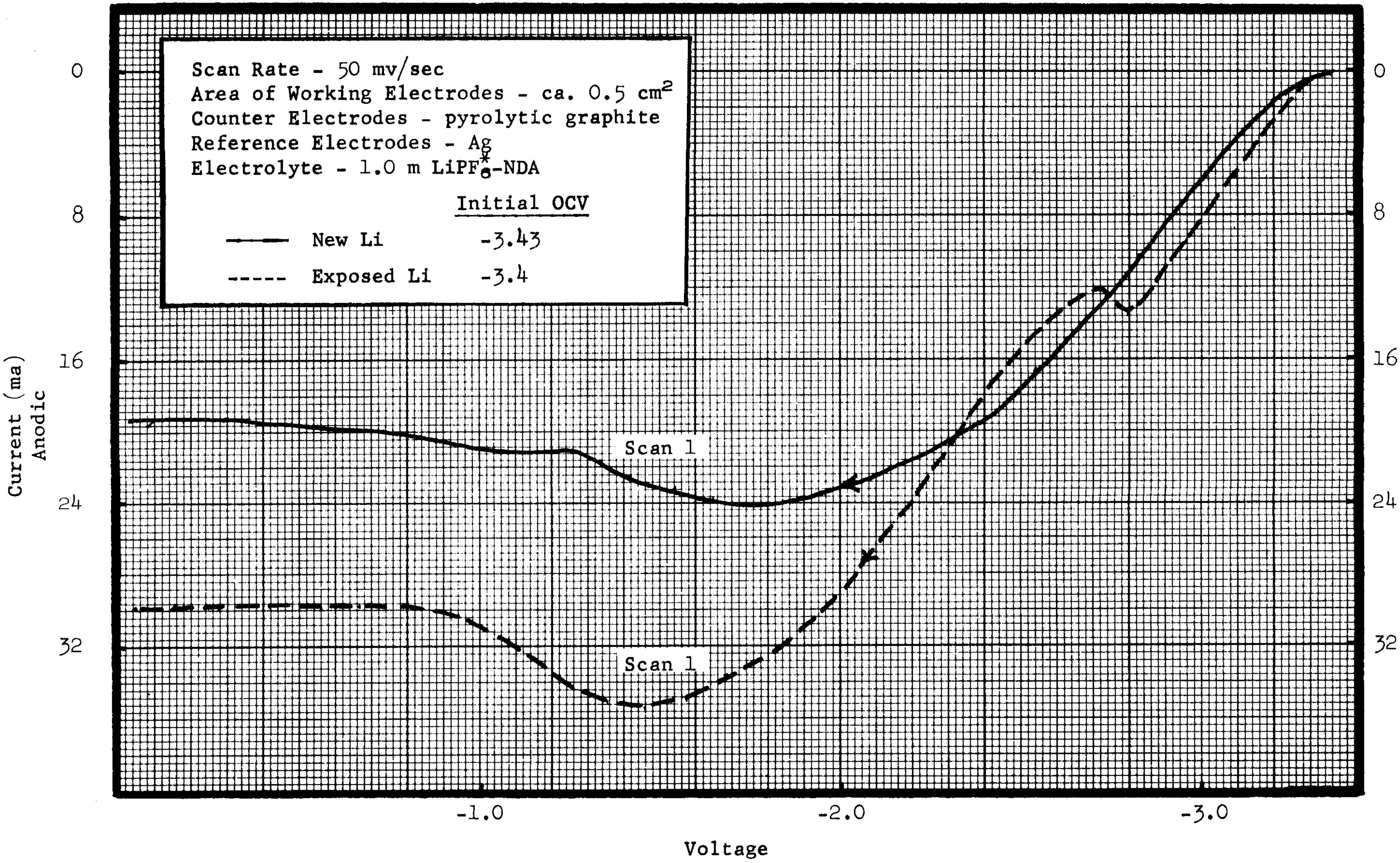

FIGURE 1. LINEARLY VARYING POTENTIAL STUDIES OF LITHIUM ANODES (Continued)

Figure 1-c. LVP Scans Before and After Twenty-Four Hour Exposure to the Electrolyte.

* "As received" $LiPF_6$.

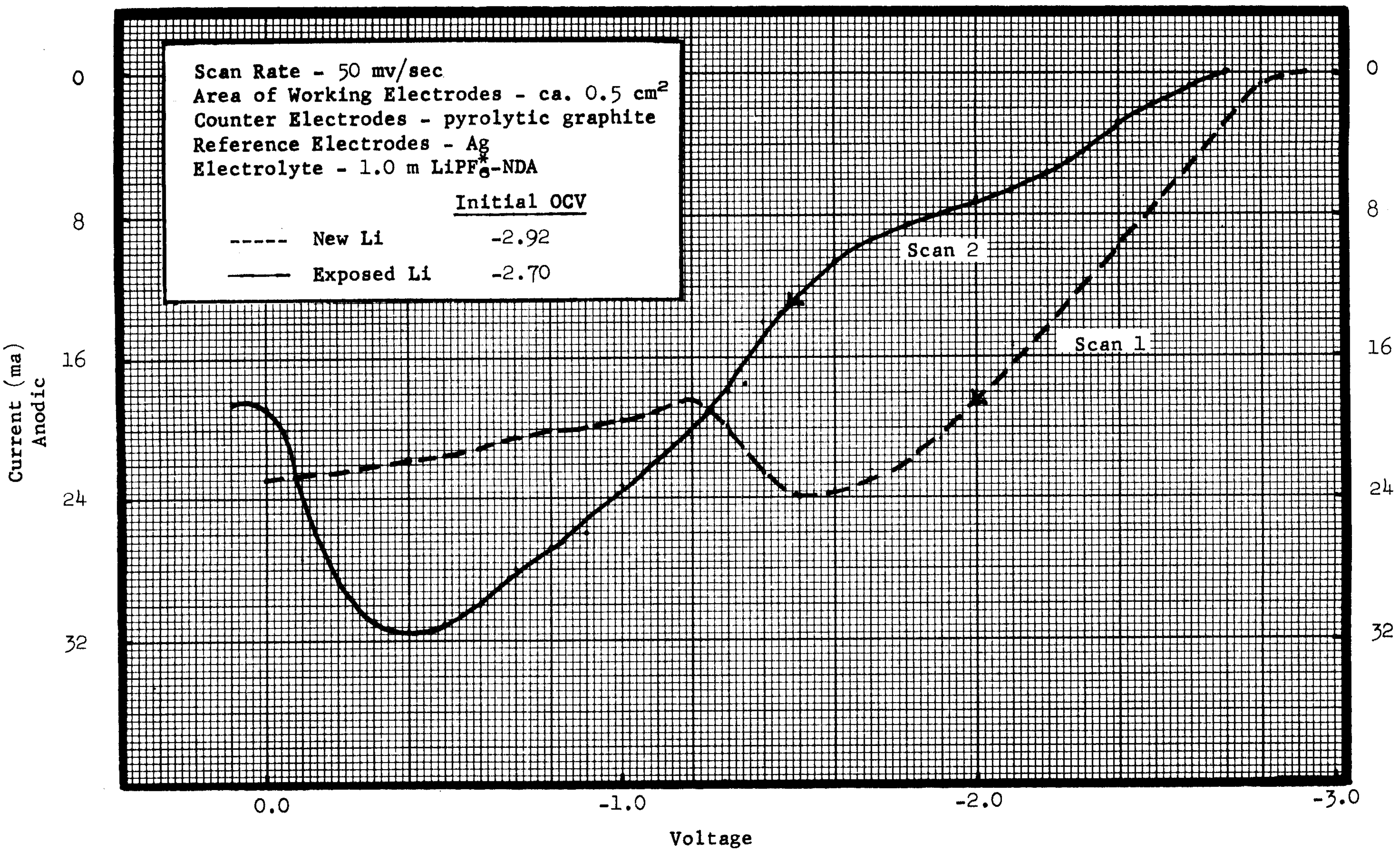

FIGURE 1. LINEARLY VARYING POTENTIAL STUDIES OF LITHIUM ANODES (Continued)

Figure 1-d. LVP Scans Before and After Twenty-Four Hour Exposure to the Electrolyte.

* The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

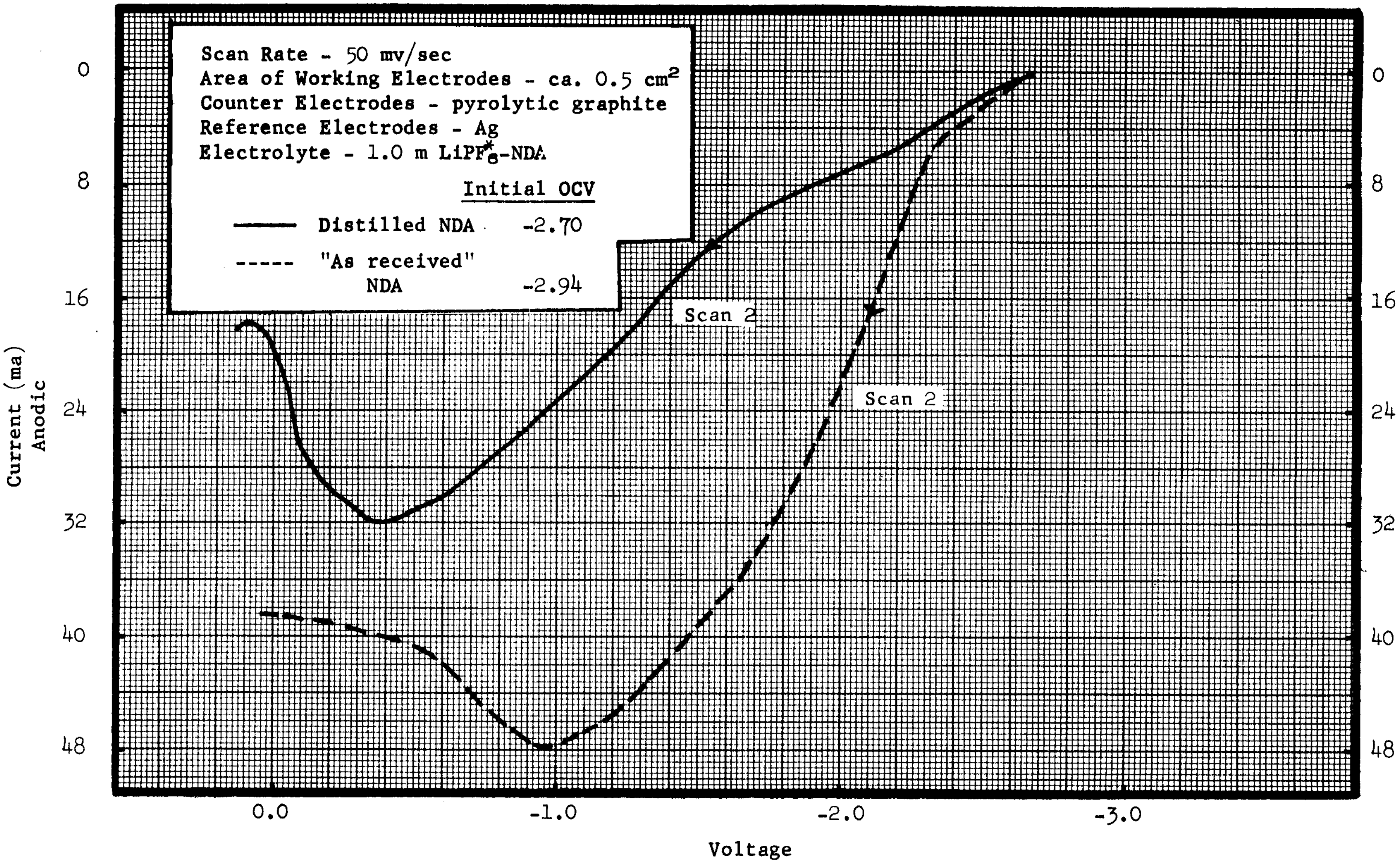

FIGURE 1. <u>LINEARLY VARYING POTENTIAL STUDIES OF LITHIUM ANODES</u> (Continued)

Figure 1-e. LVP Scans After Twenty-Four Hour Exposure to One Molal $LiPF_6^*$-N-Nitrosodimethylamine Prepared from Distilled and "As Received" Solvent.

* The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

TABLE I. ELECTROCHEMICAL HALF-CELL TESTING OF INORGANIC CATHODES

TABLE I. ELECTROCHEMICAL HALF-CELL TESTING OF INORGANIC CATHODES

A. Physical Details

	Active Cathode Material	Composition of Cathode Mix	Matrix	Method of Preparation	Electrolyte (1 Molal)	Counter Elec- trode	Reference Electrode
1.	CuF_2	100% CuF_2.	Cu screen	A small amount of CuF_2 was hot-pressed on the matrix (2.8 cm^2) at 1,400 lb/cm^2. Pressure was applied while the sample was heated to 150°C. As soon as temperature was reached the heating was stopped and the sample was cooled rapidly under pressure. One side of the electrode was masked with tape.	$LiClO_4$-PC	HP-10 Speer Carbon	Ag
2.	CuF_2	CuF_2 (97 wt. %), acetylene black (3 wt. %).	Cu screen	The mix was ball-milled in hexane and dried in air at 110°C to remove the solvent. A small amount of the mixture was hot-pressed on the matrix (2.8 cm^2) at 1,400 lb/cm^2. Pressure was applied while the sample was heated to 150°C. As soon as temperature was reached the heating was stopped and the sample was cooled rapidly under pressure. One side of the electrode was masked with tape.	$LiClO_4$-PC	Li	Ag/AgCl
3a.	CuF_2	CuF_2 (97 wt. %), acetylene black (3 wt. %).	Cu screen	Same as No. 2. The electrode was not masked with tape.	$LiClO_4$-PC	HP-10 Speer Carbon	Ag

TABLE I. ELECTROCHEMICAL HALF-CELL TESTING OF INORGANIC CATHODES (Continued)

A. Physical Details (Continued)

	Active Cathode Material	Composition of Cathode Mix	Matrix	Method of Preparation	Electrolyte (1 Molal)	Counter Electrode	Reference Electrode
*3b.	CuF_2	CuF_2 (97 wt. %), acetylene black (3 wt. %).	Cu screen	Same as No. 2. The electrode was not masked with tape.	$LiClO_4$-PC	HP-10 Speer Carbon	Ag/AgCl
4.	CuF_2	100% CuF_2.	Cu screen	Same as No. 1.	$LiPF_6^{**}$-NDA	HP-10 Speer Carbon	Ag
5.	CuF_2	CuF_2 (97 wt. %), acetylene black (3 wt. %).	Cu screen	Same as No. 2.	$LiPF_6^{**}$-NDA	Li	Ag/AgCl
6.	CuF_2	CuF_2 (97 wt. %), acetylene black (3 wt. %).	Ag expanded metal	Same as No. 2.	$LiPF_6^{**}$-NDA	Li	Ag/AgCl
7.	CuF_2	CuF_2 (97 wt. %), acetylene black (3 wt. %).	Cu screen	Same as No. 2 but the matrix area was 1.0 cm^2.	$LiPF_6^{**}$-NDA	Li	Ag
8.	CuF_2	CuF_2 (97 wt. %), acetylene black (3 wt. %).	Cu screen	Same as No. 2 but the matrix was 1.0 cm^2.	$LiPF_6^{***}$-NDA	Li	Ag
9.	CuF_2	100% CuF_2.	Cu screen	A small amount of CuF_2 was pressed on the matrix (2.8 cm^2) at 1,400 lb/cm^2. The sample was not heated.	$(C_6H_5)(CH_3)_3NPF_6$-DMF	Li	Ag/AgCl

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* This test was run simultaneously with No. 3a to determine the difference between Ag and Ag/AgCl reference electrodes.

** "As received" $LiPF_6$.

*** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

TABLE I. ELECTROCHEMICAL HALF-CELL TESTING OF INORGANIC CATHODES (Continued)

B. Electrochemical Details

Active Cathode Material	Electrolyte (1 Molal)	Counter Electrode	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities* (ma/cm^2)							
				0	0.04	0.4	1.1	1.8	3.5	5.4	7.2
1. CuF_2	$LiClO_4$-PC	HP-10 Speer Carbon	Initial	+0.49	+0.48	+0.41	**	+0.11	-0.46	-0.90	-1.50
			Final	+0.19	+0.47	+0.36	**	-0.32	-1.07	-1.50	-2.46
				0	0.04	0.4	1.1	1.8	3.5	5.4	7.2
2. CuF_2	$LiClO_4$-PC	Li	Initial	+0.46	**	**	-0.58	-0.65	-0.76	-0.85	-0.95
			Final	0.0	**	**	-0.61	-0.67	-0.79	-0.9	-0.99***
				8.9	10.7	12.5	14.3	16.1	17.8	21.4	25.0
			Initial	**	-0.78	**	-0.94	**	-1.16	-0.95	-1.30
			Final	**	-0.92	**	-1.28	**	-1.6	-1.63	-2.1

Note: A new reference electrode was inserted for the 21.4ma/cm^2 discharge.

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* Duration of discharge at each current density - 5 minutes (unless noted otherwise).

** Not run.

*** The circuit was opened for 1-1/4 hours before continuing the test.

TABLE I. ELECTROCHEMICAL HALF-CELL TESTING OF INORGANIC CATHODES (Continued)

B. Electrochemical Details (Continued)

Active Cathode Material	Electrolyte (1 Molal)	Counter Electrode	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities* (ma/cm^2)							
				0	0.04	0.4	1.1	1.8	3.5	5.4	7.2
3a. CuF$_2$	LiClO$_4$-PC	HP-10 Speer Carbon	Initial	+0.39	+0.38	+0.37	**	+0.13	-0.40	-0.56	-1.00
			Final	+0.07	+0.36	+0.28	**	-0.57	-0.94	-1.22	-1.58
				8.9	10.7	12.5	14.3	16.1	17.8	21.4	25.0
			Initial	-1.15	**	**	**	**	**	**	**
			Final	-2.18	**	**	**	**	**	**	**
				0	0.04	0.4	1.1	1.8	3.5	5.4	7.2
3b. CuF$_2$	LiClO$_4$-PC	HP-10 Speer Carbon	Initial	+0.40	+0.39	+0.27	**	-0.15	-0.50	-0.64	-1.10
			Final	+0.21	+0.39	+0.15	**	-0.74	-1.11	-1.37	-1.73
				8.9	10.7	12.5	14.3	16.1	17.8	21.4	25.0
			Initial	-1.30	**	**	**	**	**	**	**
			Final	-2.30	**	**	**	**	**	**	**

---

* Duration of discharge at each current density - 5 minutes (unless noted otherwise).

** Not run.

TABLE I. ELECTROCHEMICAL HALF-CELL TESTING OF INORGANIC CATHODES (Continued)

B. Electrochemical Details (Continued)

Active Cathode Material	Electrolyte (1 Molal)	Counter Electrode	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities* (ma/cm^2)							
				0	0.04	0.4	1.1	1.8	3.5	5.4	7.2
4. CuF_2	$LiPF_6^{**}$-NDA	HP-10 Speer Carbon	Initial	-0.22	-0.22	-0.24	***	-0.32	-0.39	-0.43	-0.58
			Final	-0.29	-0.23	-0.26	***	-0.35	-0.62	-0.85	-1.01
				8.9	10.7	12.5	14.3	16.1	17.8	21.4	25.0
			Initial	-0.65	-0.80	-0.80	-0.90	-1.10	-1.20	***	***
			Final	-1.16	-1.25	-1.45	-1.73	-1.87	-2.09	***	***
			Note:	The electrolyte began gassing during the 1.8ma/cm^2 discharge.							
				0	0.04	0.4	1.1	1.8	3.5	5.4	7.2
5. CuF_2	$LiPF_6^{**}$-NDA	Li	Initial	+0.2	***	***	+0.16	+0.15	+0.09	***	-0.03
			Final	+0.16	***	***	+0.16	+0.15	+0.09	***	-0.03
				8.9	10.7	12.5	14.3	16.1	17.8	21.4	25.0
			Initial	***	-0.15	***	-0.28	***	-0.41	-2.0	***
			Final	***	-0.18	***	-0.40	***	-1.70	-3.15 (3 min.)	
			Note:	The electrolyte turned dark green during the discharges and slight gassing occurred at discharges greater than 10.7ma/cm^2.							

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* Duration of discharge at each current density - 5 minutes (unless noted otherwise).

** "As received" $LiPF_6$.

*** Not run.

TABLE I. ELECTROCHEMICAL HALF-CELL TESTING OF INORGANIC CATHODES (Continued)

B. Electrochemical Details (Continued)

	Active Cathode Material	Electrolyte (1 Molal)	Counter Electrode	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities* (ma/cm^2)							
					0	0.04	0.4	1.1	1.8	3.5	5.4	7.2
6.	CuF_2	$LiPF_6^{**}$-NDA	Li	Initial	+0.10	***	***	***	0.0	-0.22	***	-0.32
				Final	-0.06	***	***	***	-0.17	-0.25	***	-0.34
					8.9	10.7	12.5	14.3	16.1	17.8	21.4	25.0
				Initial	***	-0.42	***	-0.54	***	-0.76	-1.0	***
				Final	***	-0.45	***	-0.66	***	-1.24	-2.7	***
					0.0	3.0	5.0	10.0	15.0	20.0	25.0	
7.	CuF_2	$LiPF_6^{**}$-NDA	Li	Initial	-0.23	-0.29	-0.42	-0.84	-1.1	-1.3	-1.5	
				Final	-0.3	-0.38	-0.77	-1.03	-1.23	-1.52	-1.9	
8.	CuF_2	$LiPF_6^{****}$-NDA	Li	Initial	-0.28	-0.4	-0.6	-1.38	-1.70	***	***	
				Final	-0.35	-0.99	-1.19	-1.70	-2.4 (1 min.)	***	***	
					0	0.04	0.4	1.1	1.8	3.5	5.4	7.2
9.	CuF_2	$(C_6H_5)CH_3)_3$ NPF_6-DMF	Li	Initial	-0.37	-0.08	-0.40	***	-1.2	-1.40	-1.60	-1.7
				Final	-0.8	-0.17	-0.97	***	-1.46	-1.62	-1.8	-1.91
					8.9	10.7	12.5	14.3	16.1	17.8	21.4	25.0
				Initial	-1.8	***	***	***	***	***	***	***
				Final	-1.92	***	***	***	***	***	***	***

---

* Duration of discharge at each current density - 5 minutes (unless noted otherwise).

** "As received" $LiPF_6$

*** Not run.

**** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

TABLE II. HALF-CELL TESTING OF INORGANIC CATHODES: THE EFFECT OF VARYING THE WEIGHT RATIO OF ACTIVE MATERIAL TO SUBSTRATE

Code

SF	Solka-Floc
AB	Acetylene black
DM	Cathode components were dry-mixed
WM	Cathode components were ball-milled in hexane and dried to remove the solvent
AT	Ambient temperature
Cu-E	Copper expanded metal
Ag-E	Silver expanded metal
I	Initial
F	Final

NOTE:

1. Li counter electrodes were used for all of the tests.
2. 1.0 m $LiPF_6^*$-N-nitrosodimethylamine was used as the electrolyte in all of the tests.

---

* "As received" $LiPF_6$.

TABLE II. HALF-CELL TESTING OF INORGANIC CATHODES: THE EFFECT OF VARYING THE WEIGHT RATIO OF ACTIVE MATERIAL TO SUBSTRATE

	Composition of Cathode Mix (Weight Percent)			Method of Preparation								Voltage of Cathode Vs. Ag Reference Electrode at Various Current Densities* (ma/cm^2)						
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature ($^\circ$C)	Weight of Mix (g)	Substrate [Area (cm^2)]	Weight of Mix/Weight of Substrate	Cathode Thickness (in)	Time of Measurement	0.0	1.0	5.0	10.0	20.0	50.0	75.0
1.	CuF_2 (94)	SF (3)	AB (3)	WM	500	AT	0.2164	Cu-E (1.0)	5.2	0.047	I	-0.2	-0.23	-0.31	-0.43	-0.62	-1.15	**
											F	-0.27	-0.24	-0.35	-0.48	-0.88	-2.45	**
2.	CuF_2 (94)	SF (3)	AB (3)	WM	1,000	AT	0.1948	Cu-E (1.0)	4.6	0.040	I	-0.2	-0.22	-0.27	-0.34	-0.60	-1.5	**
											F	-0.16	-0.21	-0.27	-0.39	-0.76	-2.0	**
3.	CuF_2 (94)	SF (3)	AB (3)	WM	2,000	AT	0.1935	Cu-E (1.0)	4.6	0.038	I	+0.24	+0.19	+0.01	-0.16	-0.46	-1.2	**
											F	-0.06	+0.15	-0.05	-0.22	-0.66	-2.2	**
4.	CuF_2 (94)	SF (3)	AB (3)	WM	3,000	AT	0.1778	Cu-E (1.0)	4.2	0.035	I	-0.17	-0.18	-0.25	-0.33	-0.52	-1.1	**
											F	-0.2	-0.15	-0.26	-0.39	-0.76	-2.5 (1.5 min.)	**
5.	CuF_2 (94)	SF (3)	AB (3)	WM	1,000	AT	0.0951	Cu-E (1.0)	2.3	0.021	I	-0.22	-0.25	-0.32	-0.40	-0.50	-1.0	**
											F	**	-0.26	-0.32	-0.48 (3 min.)	-0.77	-1.95 (1.5 min.)	**
6.	CuF_2 (94)	SF (3)	AB (3)	WM	2,000	AT	0.0884	Cu-E (1.0)	2.1	0.019	I	+0.01	+0.01	-0.24	-0.35	-0.5	-1.2	**
											F	-0.23	+0.01	-0.28	-0.43	-0.89	-2.15 (0.5 min.)	**

* Duration of discharge at each current density - 2 minutes (unless noted otherwise).
** Not run.

TABLE II. HALF-CELL TESTING OF INORGANIC CATHODES: THE EFFECT OF VARYING THE WEIGHT RATIO OF ACTIVE MATERIAL TO SUBSTRATE (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation								Voltage of Cathode Vs. Ag Reference Electrode at Various Current Densities* (ma/cm²)						
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm²)	Temperature (°C)	Weight of Mix (g)	Substrate [Area (cm²)]	Weight of Mix/Weight of Substrate	Cathode Thickness (in)	Time of Measurement	0.0	1.0	5.0	10.0	20.0	50.0	75.0
7.	CuF_2 (94)	SF (3)	AB (3)	WM	3,000	AT	0.0865	Cu-E (1.0)	2.1	0.017	I	-0.1	-0.11	-0.21	-0.32	-0.53	-1.3	**
											F	-0.22	-0.14	-0.24	-0.4	-0.88	-2.25 (0.5 min.)	**
8.	CuF_2 (94)	SF (3)	AB (3)	WM	500	AT	0.0725	Cu-E (1.0)	1.7	0.020	I	-0.18	-0.19	-0.16	-0.22	-0.37	-0.72	**
											F	-0.14	-0.13	-0.17	-0.28	-0.52	-1.95	**
9.	CuF_2 (94)	SF (3)	AB (3)	WM	2,000	150	0.4120	Cu-E (1.0)	9.4	0.054	I	-0.2	-0.18	-0.28	-0.38	-0.5	-1.4	**
											F	-0.28	-0.1	-0.38	-0.85	-1.3	-2.4 (1 min)	**
10.	CuF_2 (94)	SF (3)	AB (3)	WM	1,000	150	0.2043	Cu-E (1.0)	4.9	0.030	I	-0.2	-0.2	-0.22	-0.36	-0.84	**	**
											F	-0.32	-0.20	-0.38	-0.82	-2.3	**	**
11.	CuF_2 (94)	SF (3)	AB (3)	WM	1,000	150	0.1825	Cu-E (1.0)	4.3	0.028	I	-0.18	-0.19	-0.25	-0.26	-0.44	-1.3	**
											F	-0.20	-0.2	-0.27	-0.39	-0.75	-2.6 (1 min)	**
12.	CuF_2 (94)	SF (3)	AB (3)	WM	1,000	150	0.1731	Cu-E (1.0)	4.1	0.027	I	-0.20	-0.2	-0.26	-0.32	-0.60	-1.3	**
											F	-0.29	-0.22	-0.28	-0.46	-1.1	-2.95 (1 min)	**

* Duration of discharge at each current density - 2 minutes (unless noted otherwise).

** Not run.

TABLE II. HALF-CELL TESTING OF INORGANIC CATHODES: THE EFFECT OF VARYING THE WEIGHT RATIO OF ACTIVE MATERIAL TO SUBSTRATE (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation			Weight of Mix (g)	Substrate [Area (cm^2)]	Weight of Mix/Weight of Substrate	Cathode Thickness (in)	Time of Measurement	Voltage of Cathode Vs. Ag Reference Electrode at Various Current Densities* (ma/cm^2)						
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (°C)						0.0	1.0	5.0	10.0	20.0	50.0	75.0
13.	AgO (97)	SF (3)		DM	1,400	AT	0.4274	Ag-E (1.0)	10.3	0.045	I	**	+0.08	-0.02	-0.12	-0.42	-1.5	-1.45
											F	+0.05	+0.08	-0.04	-0.2	-0.63	-1.55	-1.9 (1 min)

* Duration of discharge at each current density - 2 minutes (unless noted otherwise).

** Not run.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES

Code

SF - Solka-Floc

AB - Acetylene black

DM - Cathode components were dry-mixed

WM - Cathode components were ball-milled in hexane and dried to remove the solvent

AT - Ambient temperature

Cu-E - Copper expanded metal

Cu-S - Copper screen

Ag-E - Silver expanded metal

NDA - N-Nitrosodimethylamine

PC - Propylene carbonate

NOTE:

1. Lithium anodes were used for all of the tests.
2. Coulombic efficiencies were calculated to a cut-off voltage of 0.2 V for the cell.
3. Equivalent weights were based on the following:

Active Material	Electron Change Per Molecule
CoF_3	3
CuF_2	2
$CuCl_2$	2
AgO	2
SbF_3	3

4. The discharges were allowed to proceed spontaneously through a transistorized variable resistor unless noted otherwise.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES

	Composition of Cathode Mix (Weight Percent)			Method of Preparation					Electrolyte (1 Molal)						Coulombs			
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature ($^\circ$C)	Substrate [Area (cm^2)]	Reference Electrode	Solute	Solvent	Volume of Electrolyte (ml)	Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Theoretical	Actual	% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
1.	CoF_3 (94)	SF (3)	AB (3)	DM	10,000	AT	Ag-E (1.0)	Ag	$LiPF_6$*	NDA	15.0	0.90	2.2	1.66	86.9	112.2	129.1**	Not definitive.
2.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiClO_4$	PC	15.0	1.54	2.3	1.7	148.6	94.1	63.3	CuF_2, LiF
****3.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiClO_4$	PC	15.0	2.11	1.9	1.5	203.6	106.0	52.1	CuF_2, Cu***
4.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiClO_4$	PC	2.9	1.94	1.8	1.5	187.2	97.2	51.9	CuF_2, Cu***, LiF
****5.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiClO_4$	PC	3.0	1.83	2.0	1.7	176.6	108.0	61.2	CuF_2, Cu***, LiF
6.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag/AgCl	$LiClO_4$	PC	15.0	1.88	12.1	1.18	181.4	63.9	35.2	CuF_2, Cu ***
****7.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiClO_4$	PC	15.0	2.32	9.7	1.3	223.9	65.2	29.1	CuF_2, $CuF_2 \cdot 2H_2O$, Cu***

* "As received" $LiPF_6$.
** Apparently material other than the active cathode material is contributing to the number of coulombs obtained.
*** Substrate metal.
**** Forced discharge.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation					Electrolyte (1 Molal)						Coulombs			
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (^oC)	Substrate [Area (cm^2)]	Reference Electrode	Solute	Solvent	Volume of Electrolyte (ml)	Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Theoretical	Actual	% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
8.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiClO_4$	PC	3.0	2.18	10.6	1.1	210.4	63.6	30.2	CuF_2, Cu*
**9.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiClO_4$	PC	3.0	1.96	10.0	1.3	189.1	65.4	34.6	CuF_2, $CuF_2 \cdot 2H_2O$, Cu*, LiF
10.	CuF_2 (80)	SF (10)	AB (10)	DM	10,000	AT	Cu-E (1.0)	Ag/AgCl	$LiClO_4$	PC	16.0	0.89	1.98	2.08	85.9	64.1	74.6	Not determined.
11.	CuF_2 (80)	SF (10)	AB (10)	DM	10,000	AT	Cu-E (1.0)	Ag/AgCl	$LiClO_4$	PC	16.0	0.44	2.27	1.7	42.7	24.5	57.4	Not determined.
**12.	CuF_2 (90)	SF (10)		DM	10,000	AT	Cu-S (1.0)	Ag/AgCl	$LiClO_4$	PC	15.0	0.85	4.32	1.69	82.0	38.9	47.4	Not determined.
13.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiClO_4$	PC	15.0	2.36	2.42	1.26	227.7	55.6	24.4	CuF_2, Cu, Ag*
**14.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiClO_4$	PC	15.0	1.97	1.98	1.49	190.1	83.8	44.1	CuF_2, Cu, Ag*

* Substrate metal.

** Forced discharge.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation					Electrolyte (1 Molal)						Coulombs			
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (°C)	Substrate [Area (cm^2)]	Reference Electrode	Solute	Solvent	Volume of Electrolyte (ml)	Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Theoretical	Actual	% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
15.	CuF$_2$ (100)				1,400	150	Ag-E (2.8)	Ag	LiClO$_4$	PC	2.9	1.90	1.77	1.4	183.4	50.1	27.3	CuF$_2$, Cu, Ag*
**16.	CuF$_2$ (100)				1,400	150	Ag-E (2.8)	Ag	LiClO$_4$	PC	2.9	1.78	1.90	1.4	171.8	54.7	31.9	CuF$_2$, LiF, Ag*
17.	CuF$_2$ (100)				1,400	150	Ag-E (2.8)	Ag	LiClO$_4$	PC	15.0	1.96	9.3	0.96	189.1	31.8	16.8	CuF$_2$, Ag*
**18.	CuF$_2$ (100)				1,400	150	Ag-E (2.8)	Ag	LiClO$_4$	PC	15.0	1.93	9.9	0.87	186.2	33.3	17.9	CuF$_2$, CuF$_2\cdot$2H$_2$O, CuOHF
19.	CuF$_2$ (100)				1,400	150	Ag-E (2.8)	Ag	LiClO$_4$	PC	3.0	2.00	9.5	1.02	192.5	30.8	16.0	CuF$_2$, CuF$_2\cdot$2H$_2$O
**20.	CuF$_2$ (100)				1,400	150	Ag-E (2.8)	Ag	LiClO$_4$	PC	3.0	1.71	10.1	1.00	165.0	36.4	22.0	CuF$_2$, CuF$_2\cdot$2H$_2$O, Ag*
21.	CuF$_2$ (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	LiPF$_6$***	NDA	15.0	1.88	1.92	1.8	181.4	195.8	****107.9	CuF$_2$, Cu*, LiF

* Substrate metal.
** Forced discharge.
*** "As received" LiPF$_6$.
**** Apparently material other than the active cathode material is contributing to the number of coulombs obtained.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation			Substrate [Area (cm^2)]	Reference Electrode	Electrolyte (1 Molal)			Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Coulombs		% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (^{o}C)			Solute	Solvent	Volume of Electrolyte (ml)				Theoretical	Actual		
*22.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6$**	NDA	15.0	1.95	1.9	1.8	188.2	213.8	113.6***	CuF_2, $CuF_2 \cdot 2H_2O$, Cu****, LiF
23.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6$**	NDA	3.1	1.79	2.8	1.55	172.7	58.1	33.6	CuF_2, $CuF_2 \cdot 2H_2O$, Cu****, LiF
*24.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6$**	NDA	3.0	1.64	1.9	1.43	158.3	74.2	46.9	CuF_2, $CuF_2 \cdot 2H_2O$, Cu****, LiF
25.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6$**	NDA	15.0	1.93	9.8	1.8	186.2	238.1	127.9***	CuF_2, Cu****, LiF
26.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag/ AgCl	$LiPF_6$**	NDA	15.0	1.74	11.1	1.74	167.9	165.2	99.9	CuF_2, Cu****, LiF
*27.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6$**	NDA	15.0	1.77	9.8	1.75	170.8	272.2	159.4***	CuF_2, Cu****,

* Forced discharge.

** "As received" $LiPF_6$.

*** Apparently material other than the active cathode material is contributing to the number of coulombs obtained.

**** Substrate metal.

TABLE III. <u>COULOMBIC EFFICIENCIES OF INORGANIC CATHODES</u> (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation					Electrolyte (1 Molal)						Coulombs			
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (^oC)	Substrate [Area (cm^2)]	Reference Electrode	Solute	Solvent	Volume of Electrolyte (ml)	Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Theoretical	Actual	% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
28.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6^*$	NDA	3.0	2.09	10.9	1.6	201.7	72.6	36.0	CuF_2, Cu**, LiF
***29.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6^*$	NDA	3.0	1.62	10.1	1.33	156.3	47.9	30.6	CuF_2, $CuF_2 \cdot 2H_2O$, Cu**, LiF
30.	CuF_2 (94)	SF (3)	AB (3)	WM	2,000	AT	Cu-E (1.0)	Ag	$LiPF_6^*$	NDA	10.0	3.58	2.00	1.65	345.5	12.4	3.6	CuF_2, $CuF_2 \cdot 2H_2O$, LiF
31.	CuF_2 (94)	SF (3)	AB (3)	WM	3,000	AT	Cu-E (1.0)	Ag	$LiPF_6^*$	NDA	10.0	3.29	2.11	1.58	317.5	38.4	12.1	CuF_2, Cu**, LiF
32.	CuF_2 (97)		AB (3)	WM	1,400	150	Ag-E (2.8)	Ag	$LiPF_6^*$	NDA	15.0	1.98	2.15	2.05	191.1	199.4	104.3****	CuF_2, Cu, LiF
***33.	CuF_2 (97)		AB (3)	WM	1,400	150	Ag-E (2.8)	Ag	$LiPF_6^*$	NDA	15.0	2.43	2.0	2.1	234.5	161.6	68.9	CuF_2, $CuF_2 \cdot 2H_2O$, $CuOHF \cdot CuF_2$, Cu, LiF

* "As received" $LiPF_6$.

** Substrate metal.

*** Forced discharge.

**** Apparently material other than the active cathode material is contributing to the number of coulombs obtained.

TABLE III. COLUMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation					Electrolyte (1 Molal)						Coulombs			
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (^oC)	Substrate [Area (cm^2)]	Reference Electrode	Solute	Solvent	Volume of Electrolyte (ml)	Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Theoretical	Actual	% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
34.	CuF_2 (97)		AB (3)	WM	1,400	150	Ag-E (2.8)	Ag	$LiPF_6$*	NDA	3.0	1.78	2.39	1.51	171.8	68.0	39.6	CuF_2, $CuF_2 \cdot 2H_2O$, Cu, LiF, Ag**
***35.	CuF_2 (97)		AB (3)	WM	1,400	150	Ag-E (2.8)	Ag	$LiPF_6$*	NDA	3.0	1.94	1.94	1.65	187.2	71.7	38.3	CuF_2, Cu, LiF
36.	CuF_2 (97)		AB (3)	WM	1,400	150	Ag-E (2.8)	Ag	$LiPF_6$*	NDA	15.0	1.84	9.2	1.77	177.6	211.4	****119.0	CuF_2, Cu, LiF, Ag**
***37.	CuF_2 (97)		AB (3)	WM	1,400	150	Ag-E (2.8)	Ag	$LiPF_6$*	NDA	15.0	1.88	9.53	1.52	181.4	140.1	77.2	CuF_2, Cu, LiF
38.	CuF_2 (97)		AB (3)	WM	1,400	150	Ag-E (2.8)	Ag	$LiPF_6$*	NDA	3.1	1.93	9.9	1.39	186.2	61.8	33.2	CuF_2, Cu, LiF, Ag**
***39.	CuF_2 (97)		AB (3)	WM	1,400	150	Ag-E (2.8)	Ag	$LiPF_6$*	NDA	3.0	2.06	10.3	1.70	198.8	76.6	38.5	CuF_2, Cu, LiF
40.	CuF_2 (80)	SF (10)	AB (10)	DM	5,000	AT	Ag-E (1.0)	Ag/ AgCl	$LiPF_6$*	NDA	16.0	0.78	1.56	2.02	74.8	104.2	****139.3	Not determined.

* "As received" $LiPF_6$.
** Substrate metal.
*** Forced discharge.
**** Apparently material other than the active cathode material is contributing to the number of coulombs obtained.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation					Electrolyte (1 Molal)						Coulombs			
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm²)	Temperature (°C)	Substrate [Area (cm²)]	Reference Electrode	Solute	Solvent	Volume of Electrolyte (ml)	Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Theoretical	Actual	% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
41.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag/AgCl	$LiPF_6$*	NDA	12.0	1.30	1.73	1.73	125.4	83.6	66.6	CuF_2, Cu**, LiF
***42.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag/AgCl	$LiPF_6$*	NDA	12.0	1.80	1.96	1.90	173.7	101.1	58.2	CuF_2, Cu**, LiF
43.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag	$LiPF_6$*	NDA	2.9	1.68	1.78	1.73	162.1	75.3	46.4	CuF_2, Cu** LiF
44.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag/AgCl	$LiPF_6$*	NDA	2.9	1.37	2.09	1.80	132.2	53.4	40.4	CuF_2, Cu** LiF
***45.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag	$LiPF_6$*	NDA	3.0	1.95	1.92	1.43	188.2	81.6	43.3	CuF_2, $CuF_2 \cdot 2H_2O$, Cu**, LiF
46.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag/AgCl	$LiPF_6$*	NDA	12.0	1.36	10.2	1.42	131.2	71.6	54.6	CuF_2, Cu** LiF
***47.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag/AgCl	$LiPF_6$*	NDA	12.0	1.21	9.9	1.41	116.8	80.2	68.7	CuF_2, Cu** LiF

* The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.
** Substrate metal.
*** Forced discharge.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation					Electrolyte (1 Molal)						Coulombs			
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (^oC)	Substrate [Area (cm^2)]	Reference Electrode	Solute	Solvent	Volume of Electrolyte (ml)	Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Theoretical	Actual	% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
48.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag/ AgCl	$LiPF_6$*	NDA	2.9	1.48	9.76	1.48	142.8	63.2	44.3	CuF_2, Cu** LiF
49.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag	$LiPF_6$*	NDA	2.9	1.13	10.01	1.40	109.0	55.9	51.2	CuF_2, Cu** LiF
***50.	CuF_2 (100)				1,400	150	Cu-S (2.8)	Ag	$LiPF_6$*	NDA	2.9	1.58	9.9	1.31	152.5	50.5	33.1	CuF_2, $CuF_2\cdot 2H_2O$, Cu**, LiF
51.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6$*	NDA	14.1	2.16	9.5	1.5	208.4	94.1	45.2	CuF_2, Cu**, Ag
52.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6$*	NDA	15.0	2.27	10.6	1.3	219.1	93.5	42.7	CuF_2, Cu**, LiF
53.	CuF_2 (97)		AB (3)	WM	1,400	150	Cu-S (2.8)	Ag	$LiPF_6$*	****NDA	15.0	1.73	9.1	1.34	166.9	83.0	49.7	CuF_2, Cu**, LiF
54.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiPF_6$*	NDA	15.0	1.74	1.54	1.74	167.9	97.9	58.3	CuF_2, LiF, Ag**

* The $LiPF_6$ was dried at 110^oC in a nitrogen atmosphere before use.

** Substrate metal.

*** Forced discharge.

**** Water was added to the electrolyte after 60 minutes of discharge to determine its effect on electrochemical performance.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation			Substrate [Area (cm^2)]	Reference Electrode	Electrolyte (1 Molal)			Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Coulombs		% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (^oC)			Solute	Solvent	Volume of Electrolyte (ml)				Theoretical	Actual		
*55.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiPF_6$**	NDA	15.0	2.07	1.91	1.65	199.8	117.0	57.2	CuF_2, Cu, LiF
56.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiPF_6$**	NDA	3.0	2.10	1.6	1.4	202.6	92.8	45.8	CuF_2, Cu, Ag***
*57.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiPF_6$**	NDA	3.0	1.88	2.0	1.6	181.4	88.0	48.5	CuF_2, Cu, LiF, Ag***
58.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiPF_6$**	NDA	15.0	1.76	9.76	1.34	169.8	94.3	55.5	CuF_2, $CuF_2 \cdot 2H_2O$, Cu, LiF
*59.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiPF_6$**	NDA	15.0	1.95	10.3	1.3	188.2	92.7	49.3	CuF_2, Cu, LiF, Ag***
60.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiPF_6$**	NDA	3.0	1.89	10.2	1.3	182.4	80.2	44.0	CuF_2, $CuF_2 \cdot 2H_2O$, Cu, LiF, Ag***

* Forced discharge.
** The $LiPF_6$ was dried at 110^oC in a nitrogen atmosphere before use.
*** Substrate metal.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation					Electrolyte (1 Molal)						Coulombs			
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (^oC)	Substrate [Area (cm^2)]	Reference Electrode	Solute	Solvent	Volume of Electrolyte (ml)	Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Theoretical	Actual	% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
*61.	CuF_2 (100)				1,400	150	Ag-E (2.8)	Ag	$LiPF_6$**	NDA	3.0	1.48	10.2	1.6	142.8	44.7	31.3	CuF_2, Cu, LiF, Ag***
62.	CuF_2 (80)	SF (10)	AB (10)	DM	8,000	AT	Ag-E (1.0)	Ag	$LiPF_6$**	NDA	2.4	0.44	2.53	1.8	42.5	15.9	37.5	Not determined.
*63.	CuF_2 (80)+ $CuCl_2$ (10)	SF (10)		DM	10,000	AT	Cu-S (1.0)	Ag/AgCl	$LiClO_4$	PC	15.0	0.467 0.045	4.59	1.19	49.4	21.5	43.5	Not determined.
*64.	CuF_2 (70)+ $CuCl_2$ (20)	SF (10)		DM	10,000	AT	Cu-S (1.0)	Ag/AgCl	$LiClO_4$	PC	15.0	0.65 0.14	4.23	1.53	76.2	40.9	53.6	Not determined.
65.	AgO (97)	SF (3)		DM	10,000	AT	Ag-E (1.0)	Ag	$LiClO_4$	PC	15.0	0.63	4.8	0.83	60.8	7.2	11.8	Not determined.

* Forced discharge.
** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.
*** Substrate metal.

TABLE III. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

	Composition of Cathode Mix (Weight Percent)			Method of Preparation					Electrolyte (1 Molal)						Coulombs			
	Active Material	Binder	Conductive Additives	Mixing	Pressure (lb/cm^2)	Temperature (^oC)	Substrate [Area (cm^2)]	Reference Electrode	Solute	Solvent	Volume of Electrolyte (ml)	Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Theoretical	Actual	% Coulombic Efficiency	Components of Cathode After Discharge as Shown by X-ray Diffraction
*66.	AgO (97)	SF (3)		DM	1,400	50	Ag-E (1.0)	Ag	$LiPF_6$**	NDA	15.0	0.5	0.93	2.01	48.3	87.6	181.4***	Not determined.
*67.	AgO (97)	SF (3)		DM	1,400	50	Ag-E (1.0)	Ag	$LiPF_6$**	NDA	15.0	0.6	9.8	1.64	57.9	129.9	224.4***	Ag****
68.	AgO (97)	SF (3)		DM	10,000	AT	Ag-E (1.0)	Ag	$LiPF_6$**	NDA	15.0	0.48	1.88	1.78	46.3	102.2	220.7***	Not definitive.
69.	AgO (97)	SF (3)		DM	10,000	AT	Ag-E (1.0)	Ag	$LiPF_6$**	NDA	2.2	0.45	2.01	1.71	43.4	34.3	79.0	Not definitive.
70.	AgO (97)	SF (3)		DM	10,000	AT	Ag-E (1.0)	Ag	$LiPF_6$**	NDA	15.0	0.46	9.4	1.63	44.4	109.4	246.4***	LiF, Ag****
71.	AgO (95)	SF (5)		DM	8,000	AT	Ag-E (1.0)	Ag/AgCl	$LiPF_6$**	NDA	16.0	0.46	1.86	2.29	44.4	167.3	376.8***	Not determined.
72.	AgO (87)+ SbF_3 (10)	SF (3)		DM	10,000	AT	Ag-E (1.0)	Ag	$LiClO_4$	PC	15.0	0.90 0.11	5.0	1.04	97.5	4.8	4.9	Not determined.

* Forced discharge.
** "As received" $LiPF_6$.
*** Apparently material other than the active cathode material is contributing to the number of coulombs obtained.
**** Substrate metal.

SUPPLEMENT TO TABLE III: CONSTANT CURRENT BEHAVIOR OF $LiPF_6^*$-N-NITROSODIMETHYLAMINE ELECTROLYTE WITH INERT WORKING ELECTRODES

	Working Electrode [Area (cm^2)]	Reference Electrode	Electrolyte (1 Molal)			Milliequivalents of Active Material	Average Current (ma)	Average Cell Voltage	Coulombs		% Coulombic Efficiency
			Solute	Solvent	Volume of Electrolyte (ml)				Theoretical	Actual	
1.	Pyrolytic graphite (1.76)	Ag	$LiPF_6^*$	NDA	15.0	----	1.66	1.10	-----	56.3	------
2.	Ag expanded metal (2.8)	Ag	$LiPF_6^*$	NDA	15.0	----	2.77	1.91	-----	106.4	------

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* "As received" $LiPF_6$.

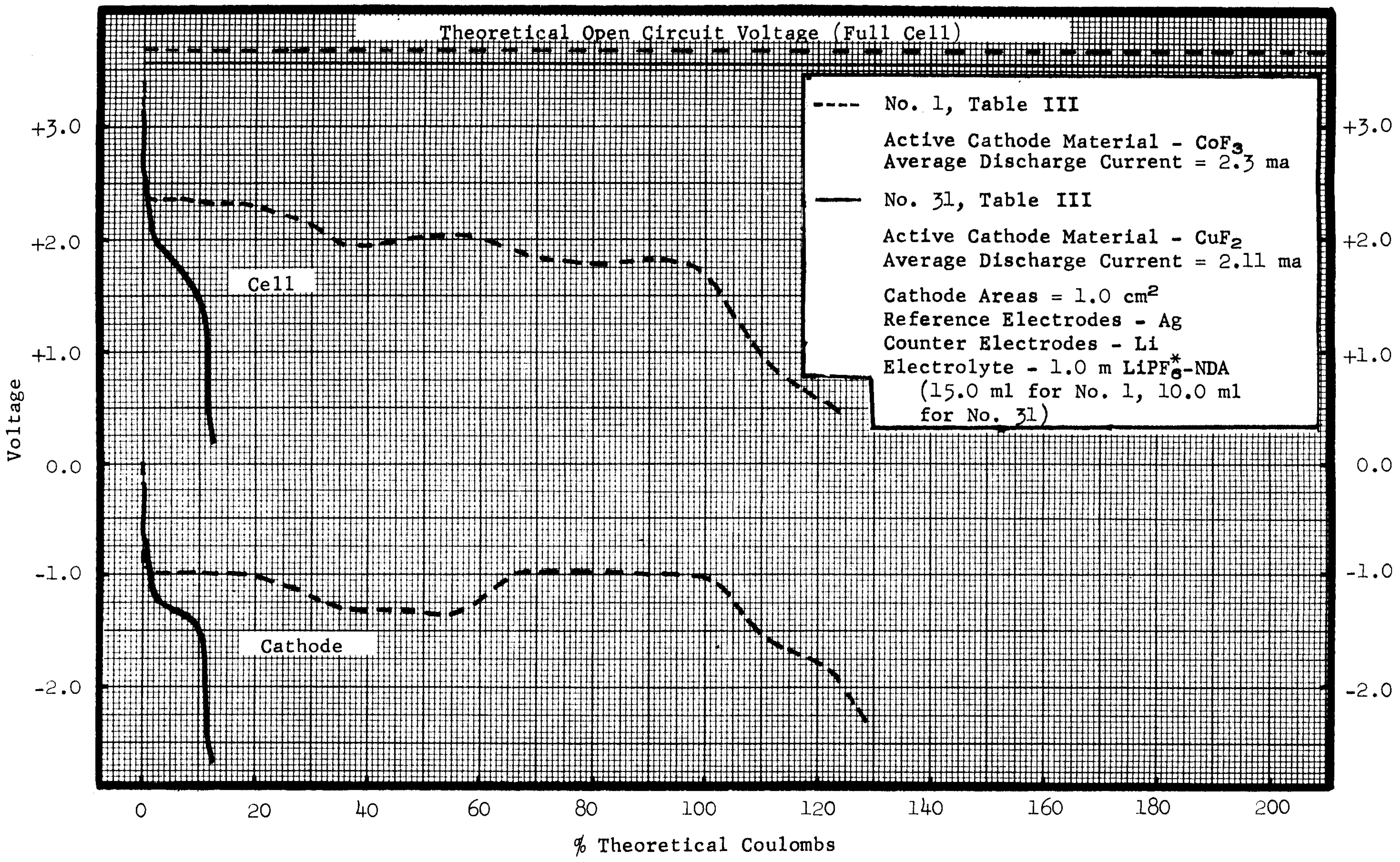

FIGURE 2. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES

Figure 2-a. Cobalt Trifluoride (94 Wt.%) Plus Acetylene Black (3 Wt. %) and Solka-Floc (3 Wt.%) Cold-Pressed on Silver Expanded Metal and Cupric Fluoride (94 Wt. %) Plus Acetylene Black (3 Wt. %) and Solka-Floc (3 Wt. %) Cold-Pressed on Copper Expanded Metal.

* "As received" LiPF$_6$.

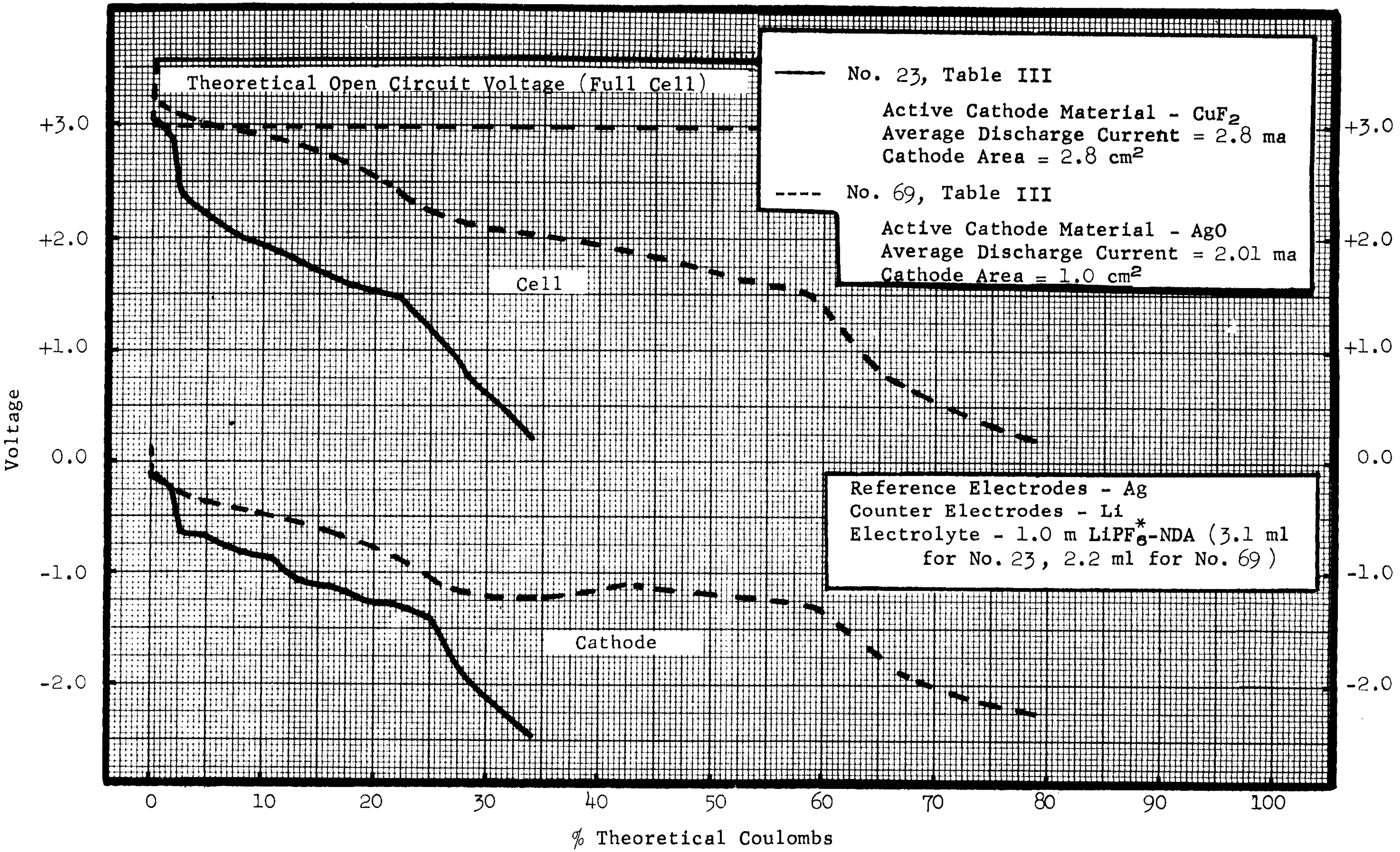

FIGURE 2. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

Figure 2-b. Cupric Fluoride (97 Wt. %) Plus Acetylene Black (3 Wt. %) Hot-Pressed on Copper Screen and Silver Oxide (97 Wt. %) Plus Solka-Floc (3 Wt. %) Cold-Pressed on Silver Expanded Metal.

* "As received" $LiPF_6$.

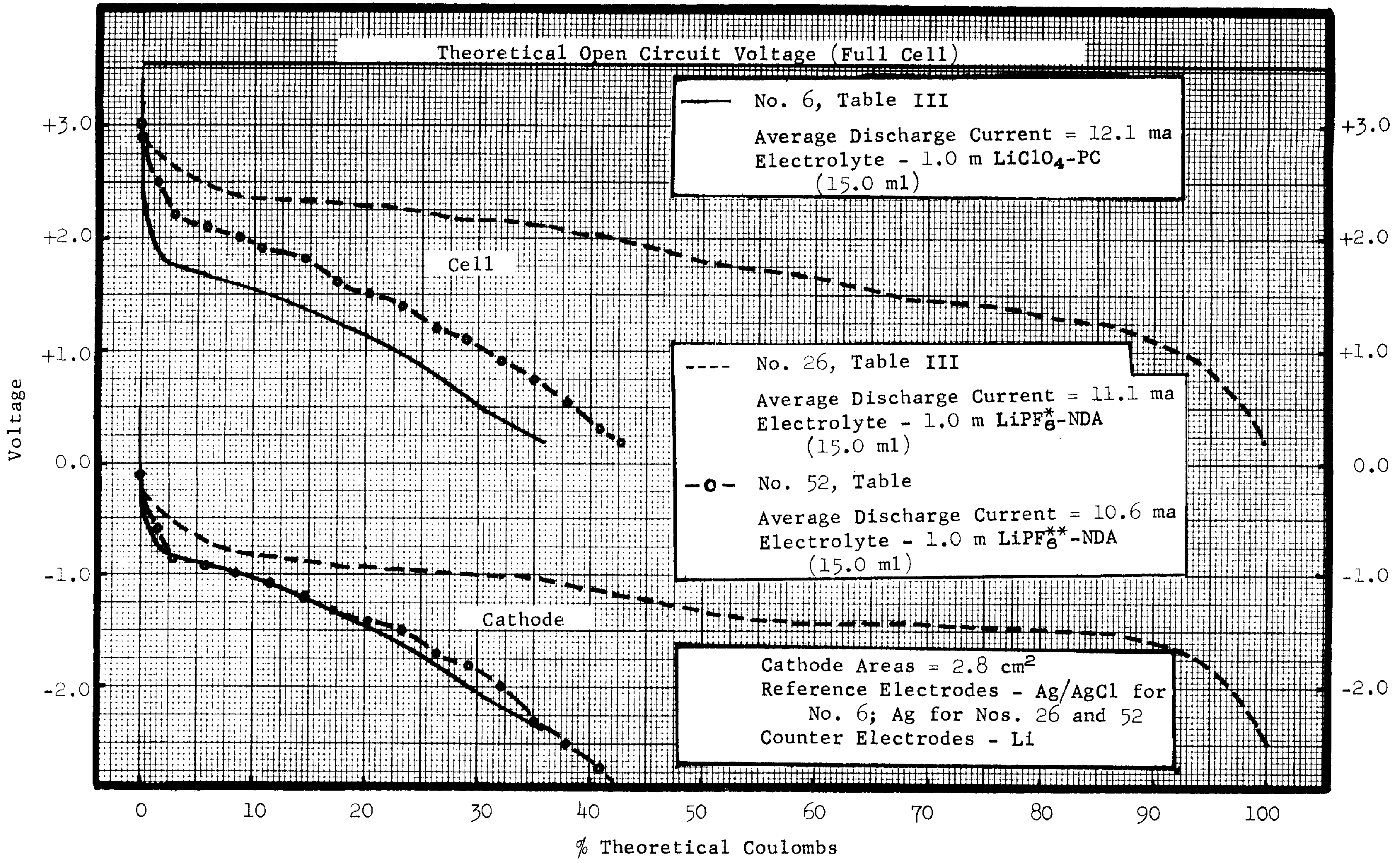

FIGURE 2. <u>COULOMBIC EFFICIENCIES OF INORGANIC CATHODES</u> (Continued)

Figure 2-c. Cupric Fluoride (97 Wt. %) Plus Acetylene Black (3 Wt. %) Hot-Pressed on Copper Screen. A Comparison of Discharges in Different Electrolytes.

* "As received" $LiPF_6$.

** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

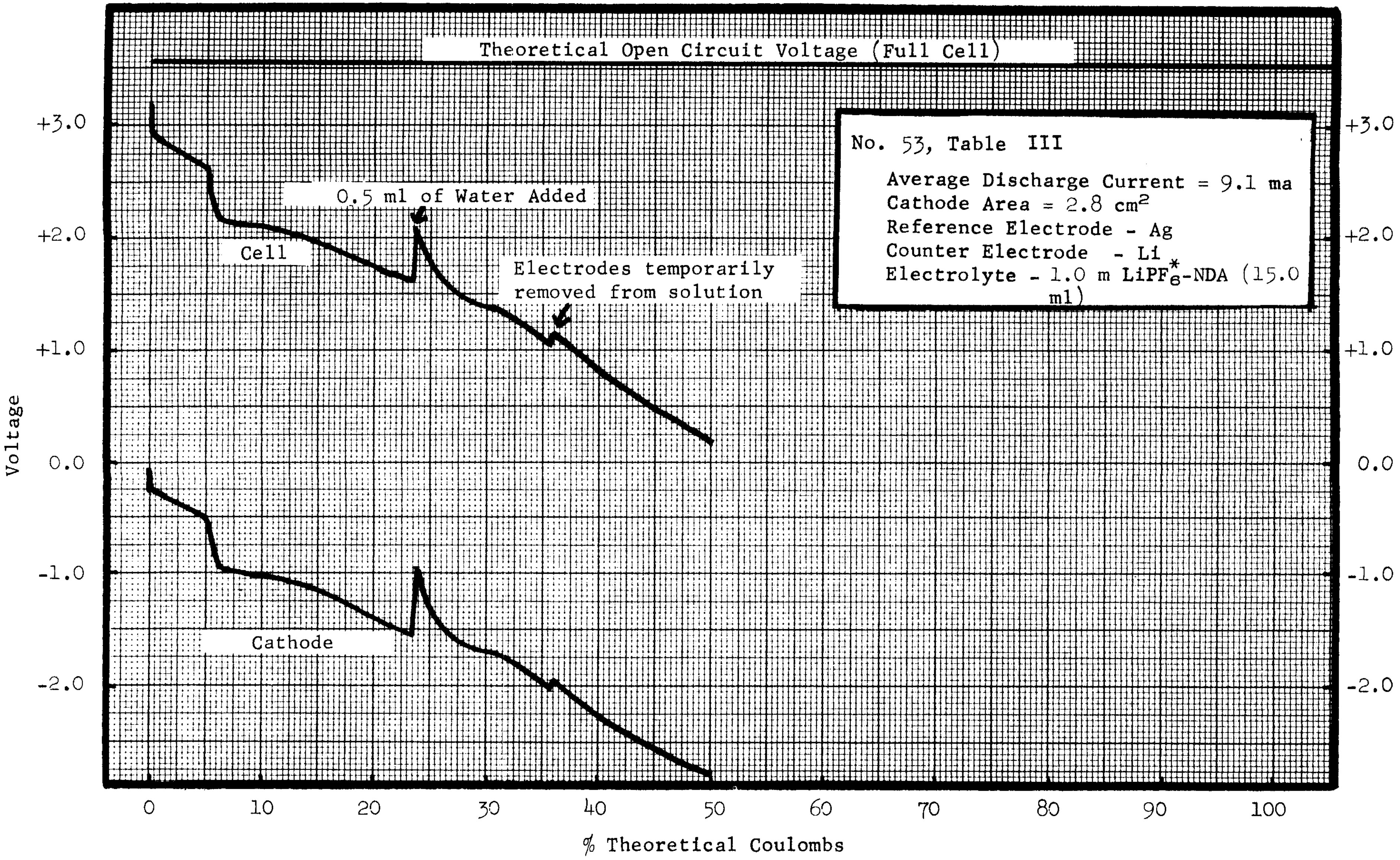

FIGURE 2. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

Figure 2-d. Cupric Fluoride (97 Wt. %) Plus Acetylene Black (3 Wt. %) Hot-Pressed on Copper Screen. The Effect of Adding Water During the Discharge.

* The LiPF$_6$ was dried at 110°C in a nitrogen atmosphere before use.

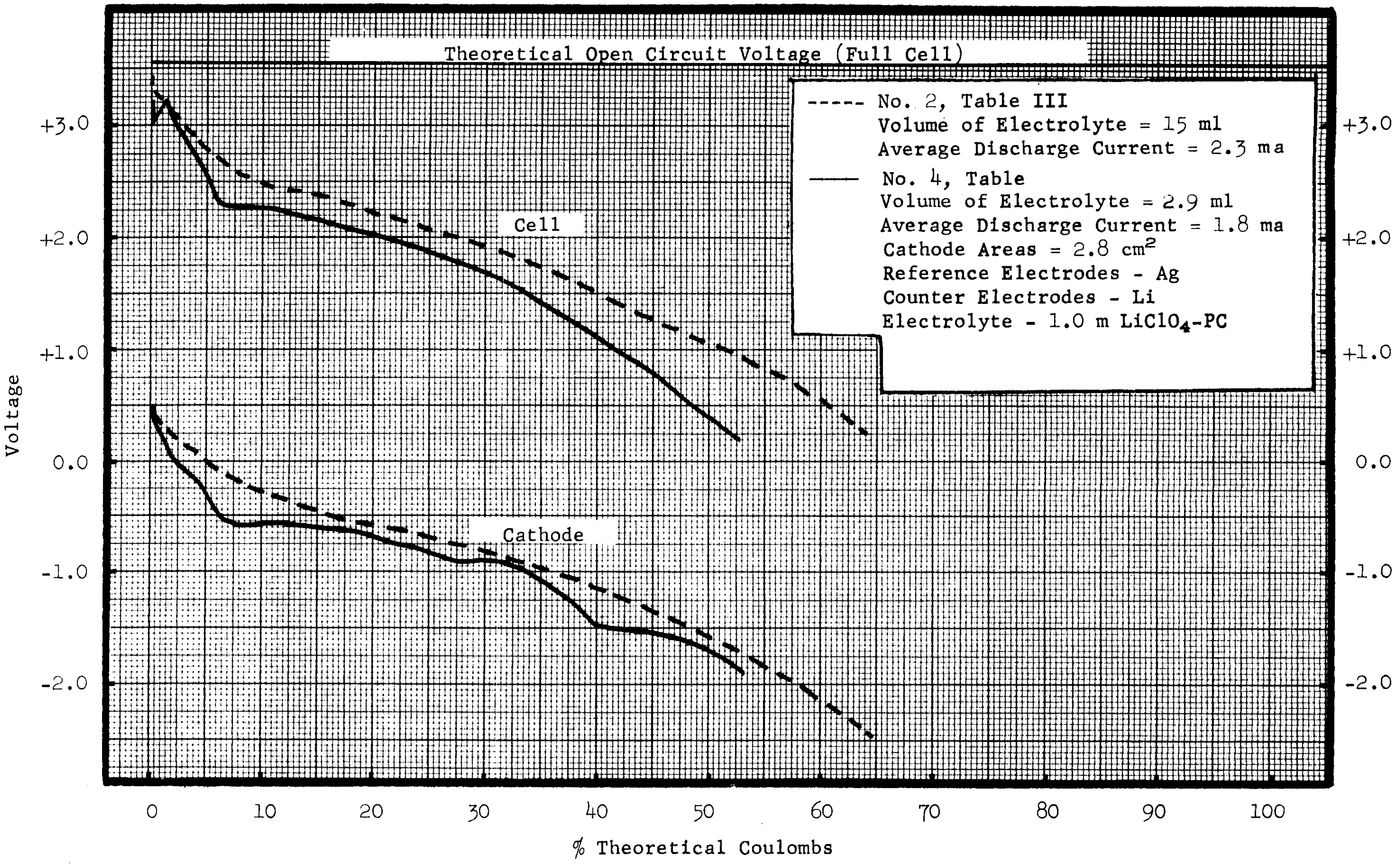

FIGURE 2. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

Figure 2-e. Cupric Fluoride (97 Wt. %) Plus Acetylene Black (3 Wt. %) Hot-Pressed on Copper Screen. A Comparison of Discharges in Different Amounts of Electrolyte.

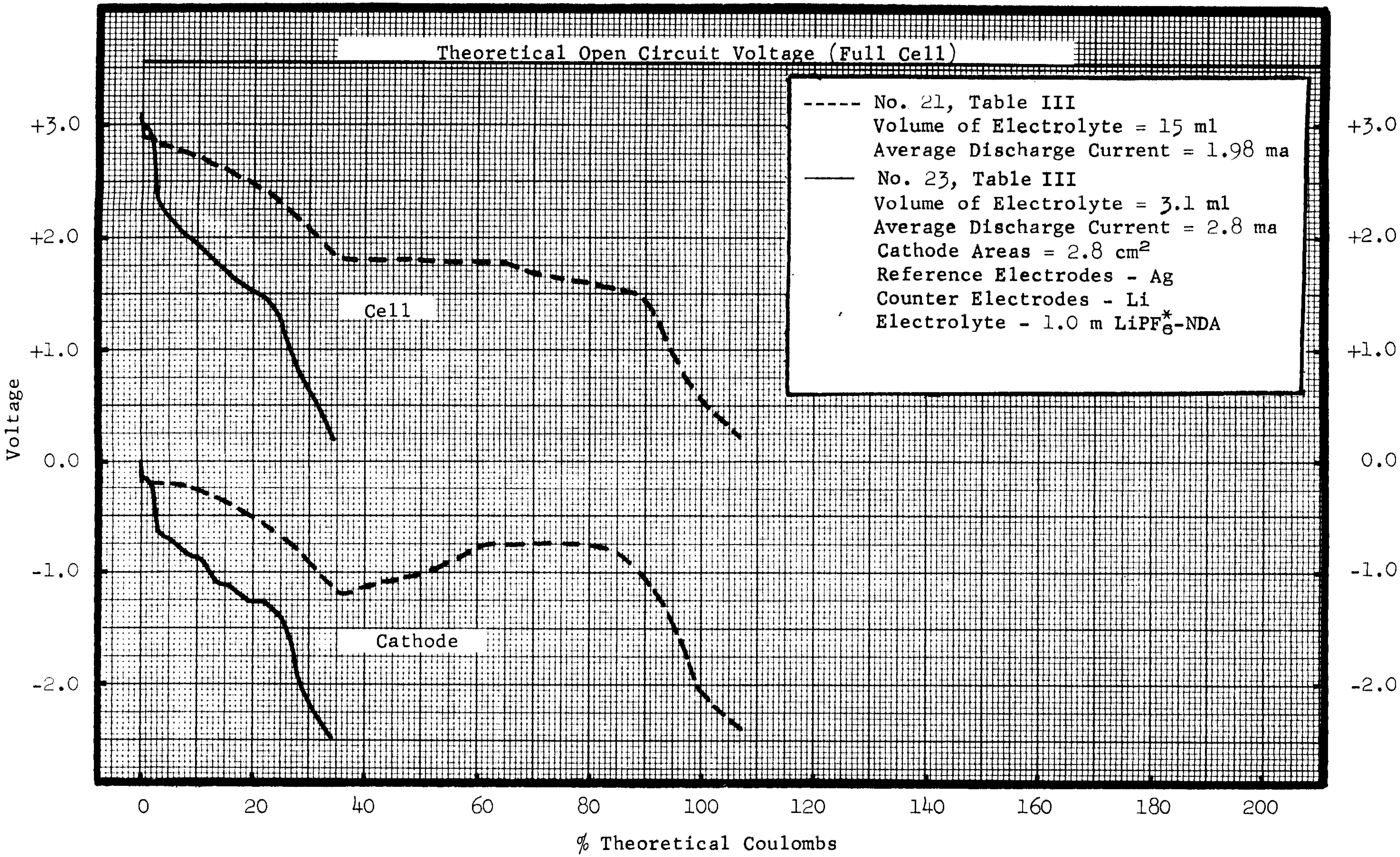

FIGURE 2. <u>COULOMBIC EFFICIENCIES OF INORGANIC CATHODES</u> (Continued)

Figure 3-f. Cupric Fluoride (97 Wt. %) Plus Acetylene Black (3 Wt. %) Hot-Pressed on Copper Screen. A Comparison of Discharges in Different Amounts of Electrolyte.

* "As received" $LiPF_6$.

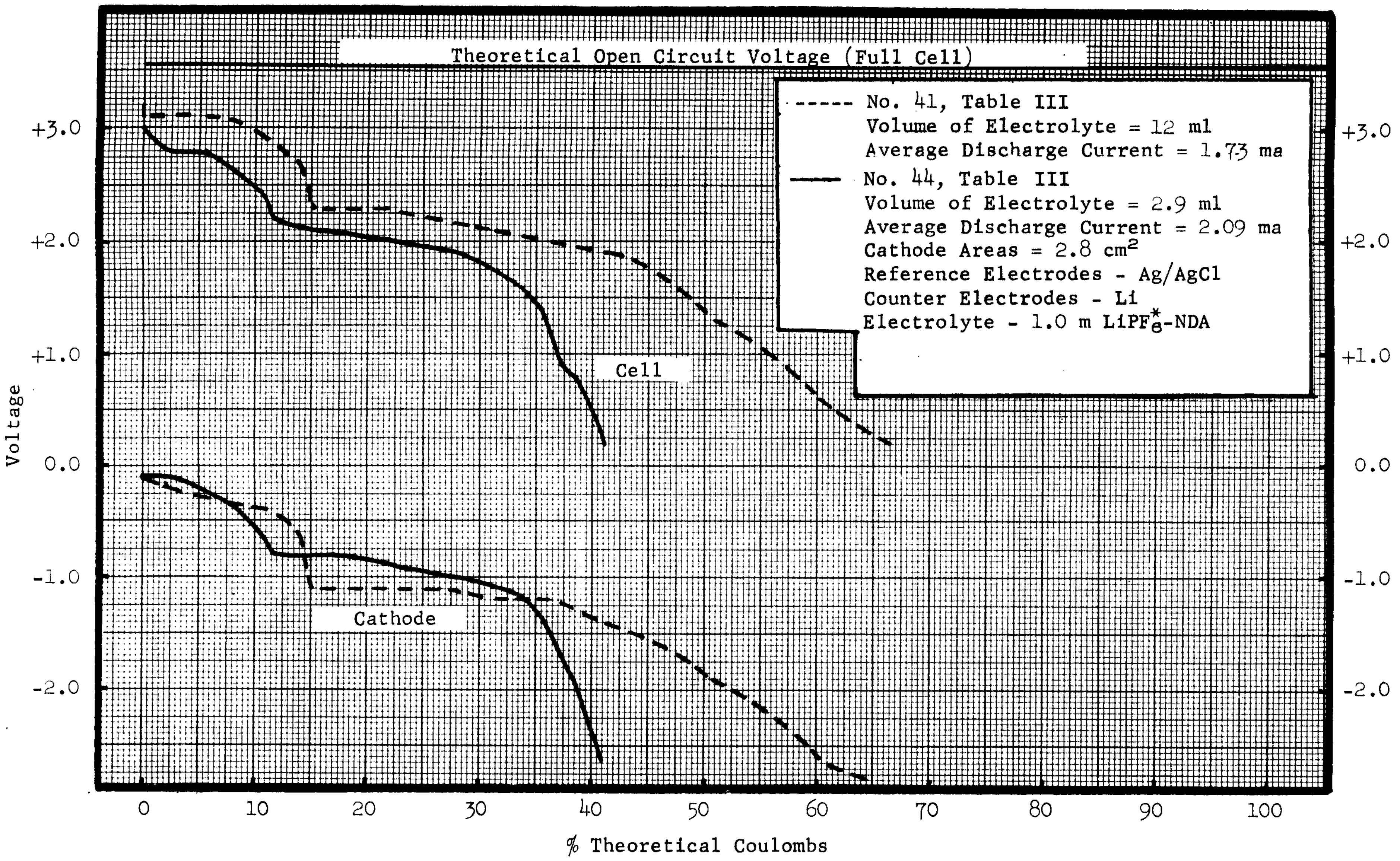

FIGURE 2. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

Figure 2-g. Cupric Fluoride Hot-Pressed on Copper Screen. A Comparison of Discharges in Different Amounts of Electrolyte.

* The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

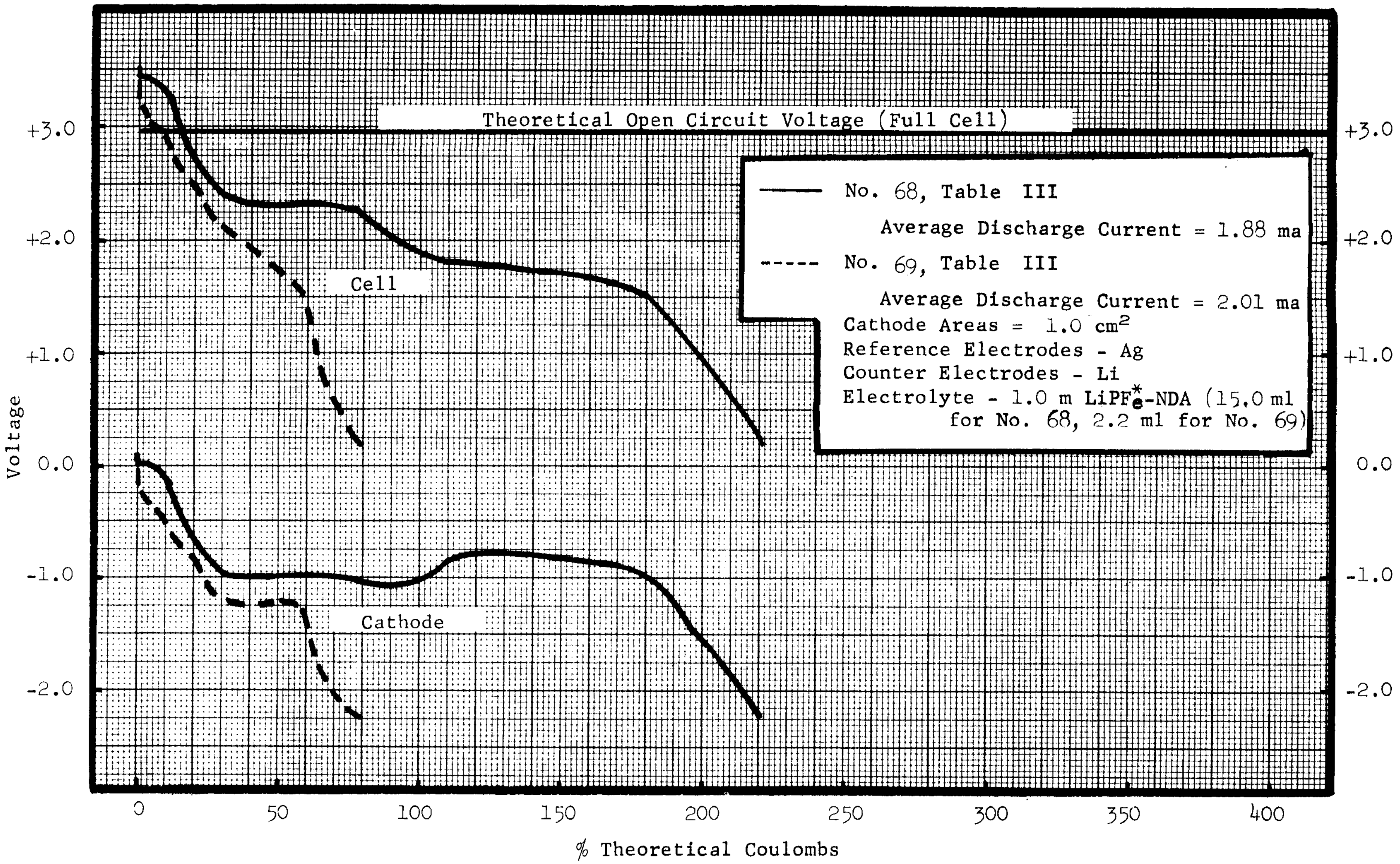

FIGURE 2. <u>COULOMBIC EFFICIENCIES OF INORGANIC CATHODES</u> (Continued)

Figure 2-h. Silver Oxide Plus Solka-Floc (3 Wt. %) Cold-Pressed on Silver Expanded Metal. A Comparison of Discharges in Different Amounts of Electrolyte.

* "As received" $LiPF_6$.

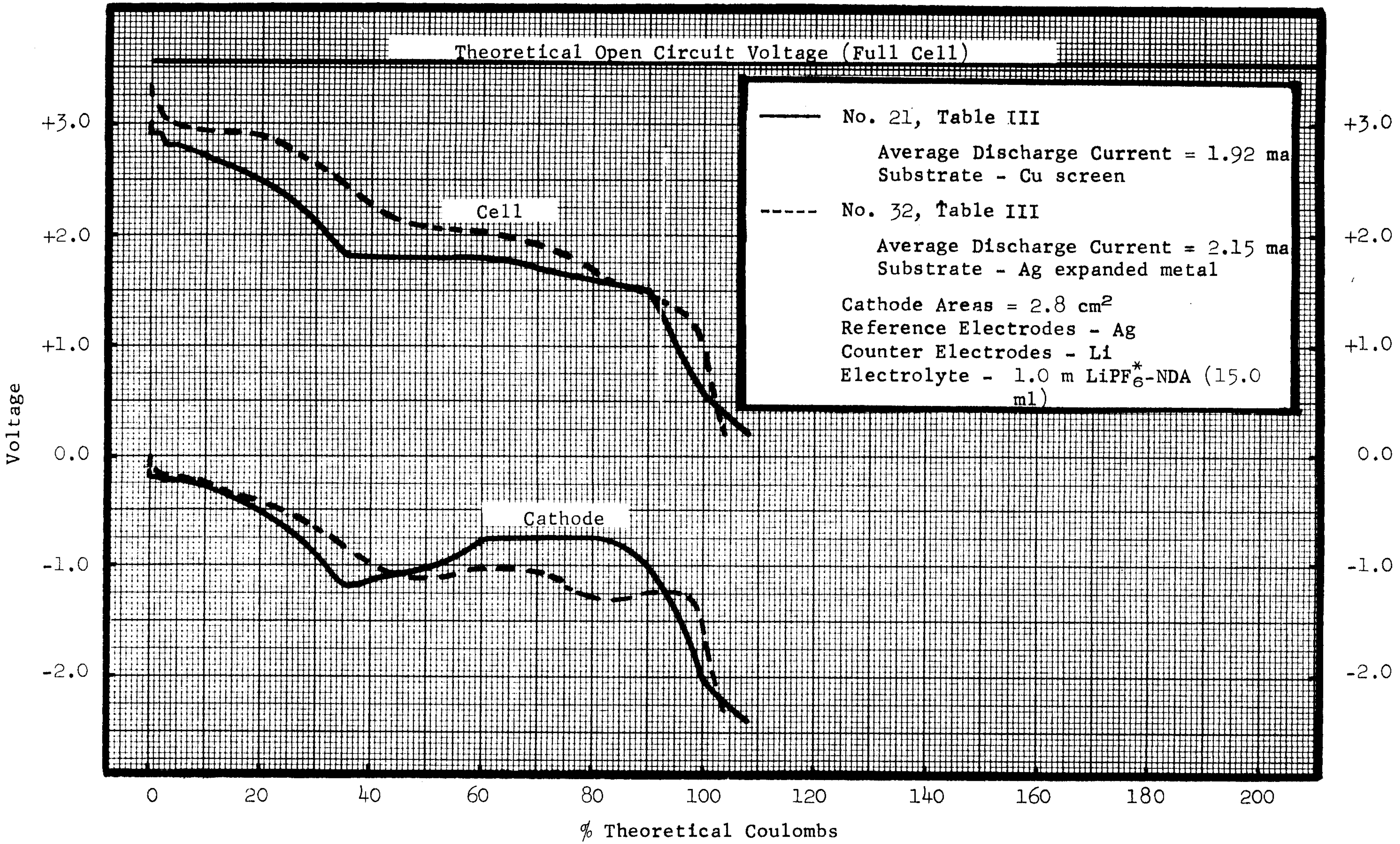

FIGURE 2. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

Figure 2-1. Cupric Fluoride (97 Wt. %) Plus Acetylene Black (3 Wt. %) Hot-Pressed on Copper Screen and Silver Expanded Metal. A Comparison of Different Substrates.

* "As received" LiPF$_6$.

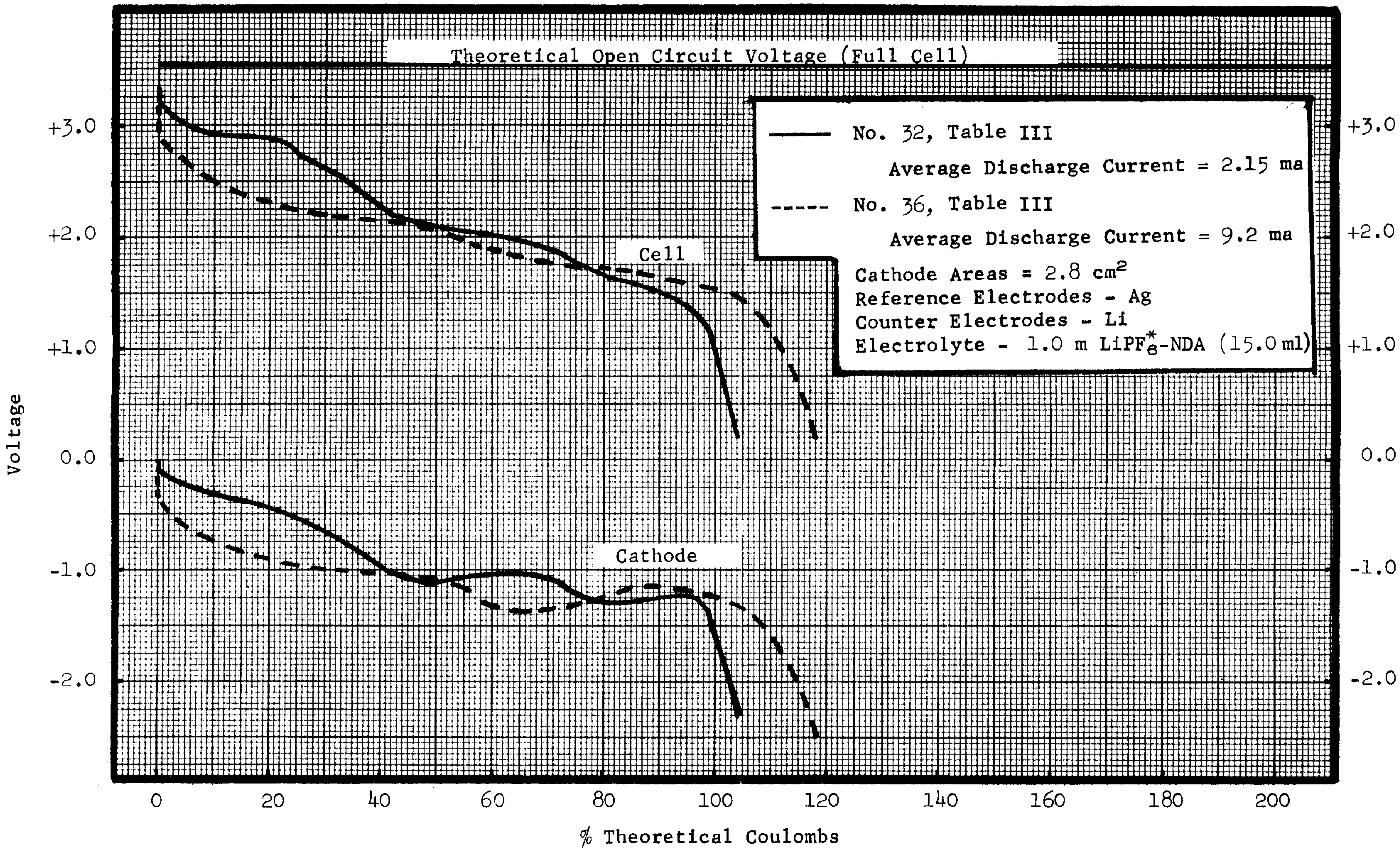

FIGURE 2. <u>COULOMBIC EFFICIENCIES OF INORGANIC CATHODES</u> (Continued)

Figure 2-j. Cupric Fluoride (97 Wt. %) Plus Acetylene Black (3 Wt. %) Hot-Pressed on Silver Expanded Metal. A Comparison of Discharges at Different Current Densities.

* "As received" $LiPF_6$.

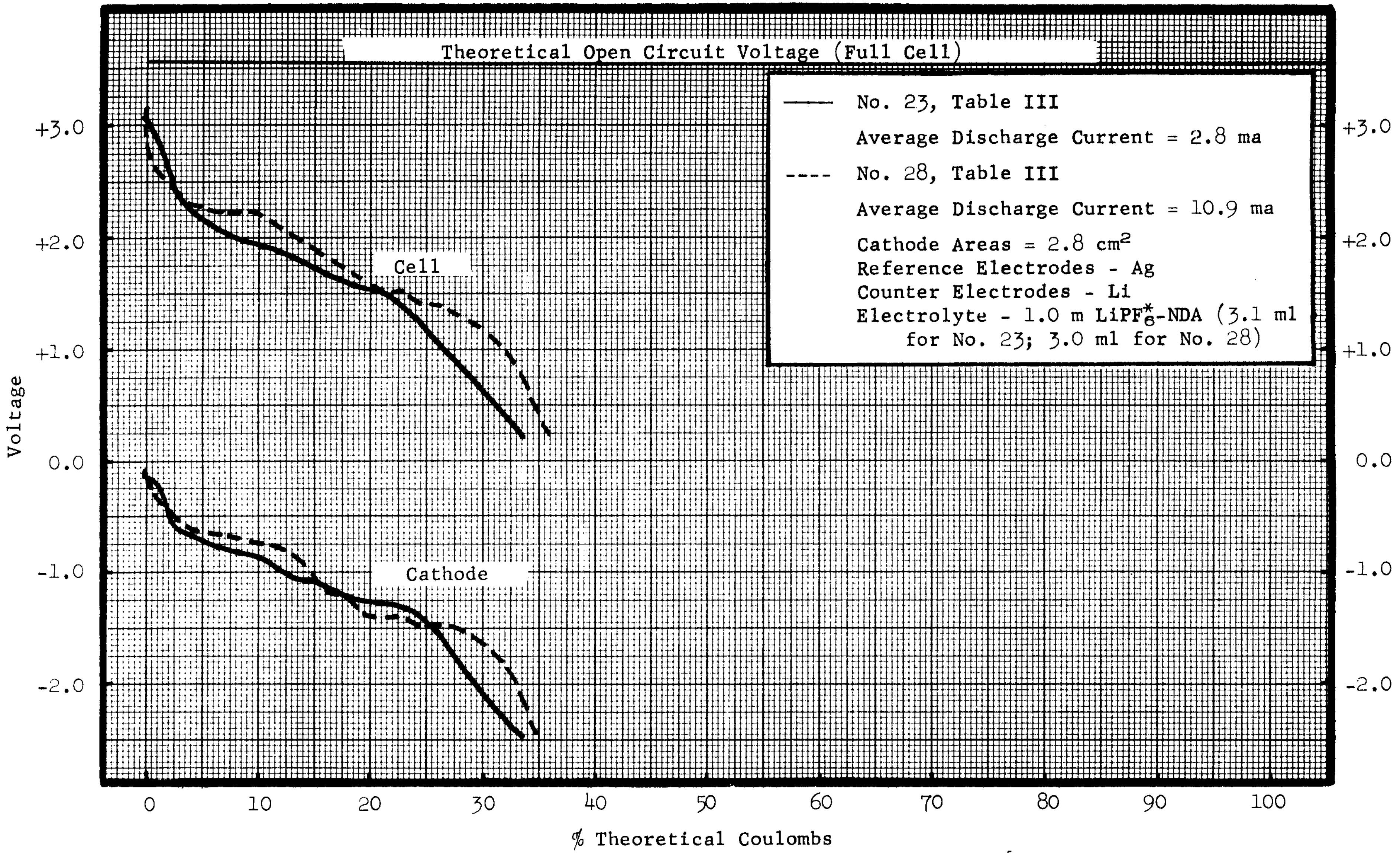

FIGURE 2. COULOMBIC EFFICIENCIES OF INORGANIC CATHODES (Continued)

Figure 2-k. Cupric Fluoride (97 Wt. %) Plus Acetylene Black (3 Wt. %) Hot-Pressed on Copper Screen. A Comparison of Discharges at Different Current Densities.

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* "As received" $LiPF_6$.

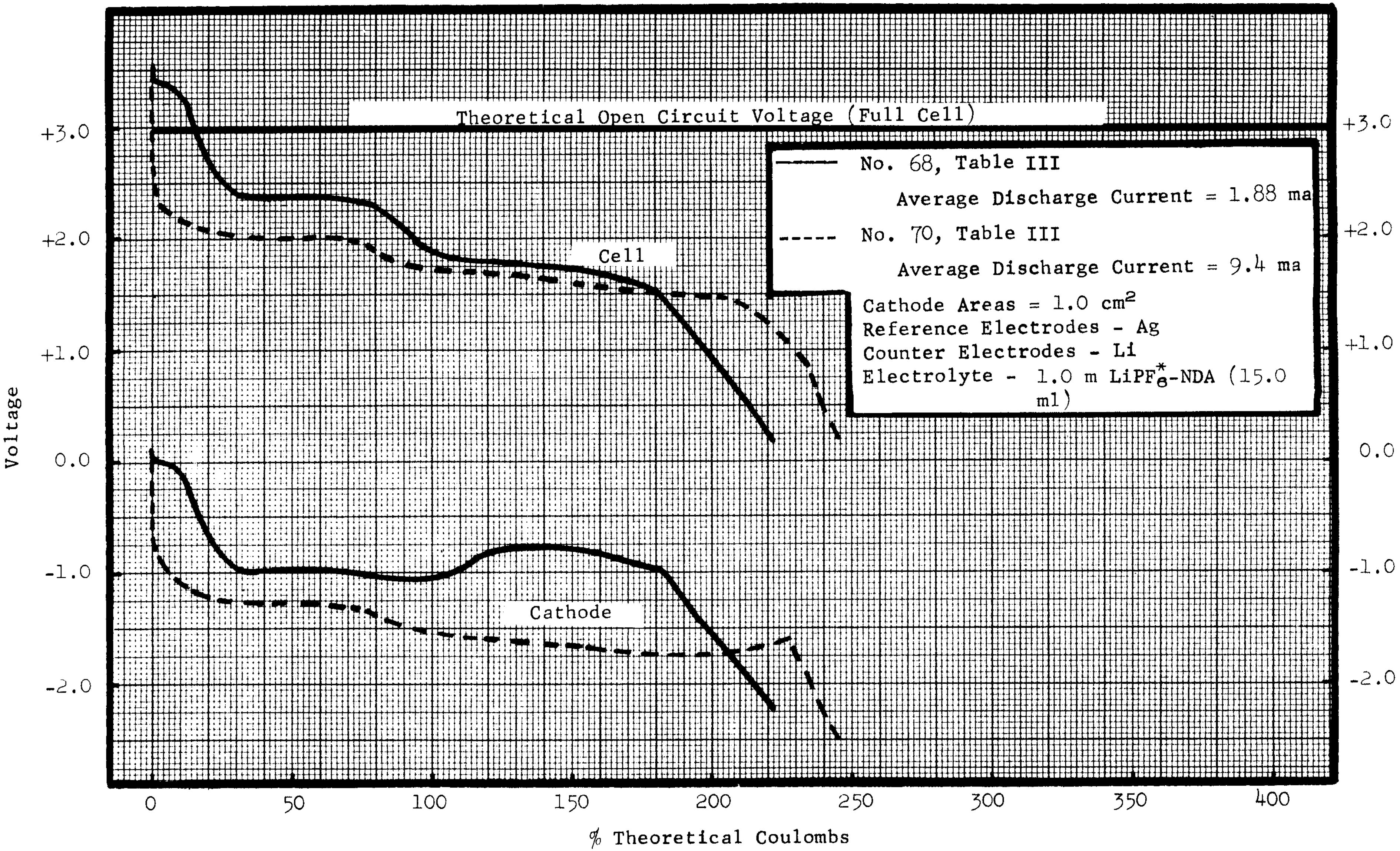

FIGURE 2. <u>COULOMBIC EFFICIENCIES OF INORGANIC CATHODES</u> (Continued)

Figure 2-1. Silver Oxide Plus Solka-Floc (3 Wt. %) Cold-Pressed on Silver Expanded Metal. A Comparison of Discharges at Different Current Densities.

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* "As received" $LiPF_6$.

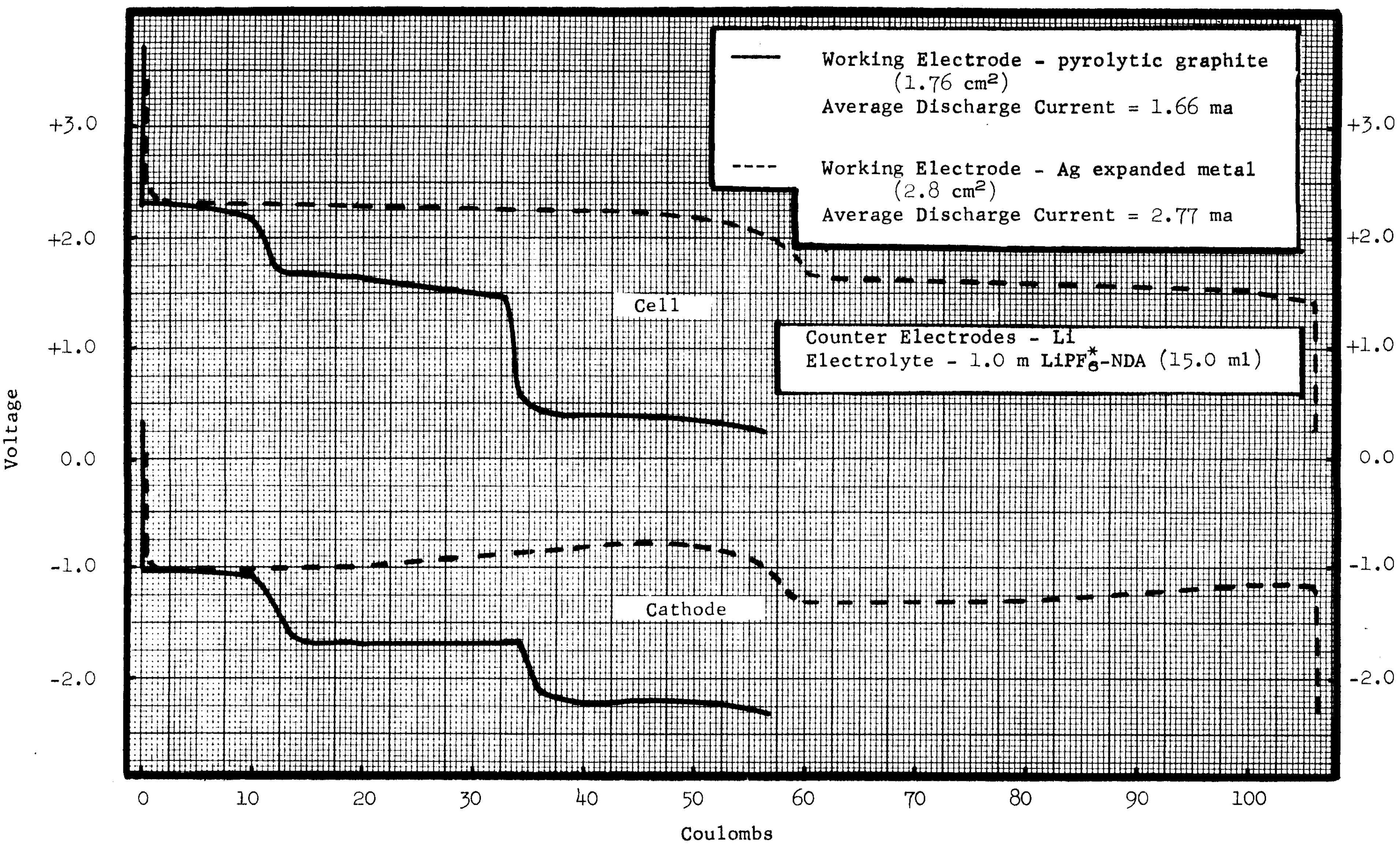

FIGURE 3. CONSTANT CURRENT BEHAVIOR OF $LiPF_6^*$-N-NITROSODIMETHYLAMINE ELECTROLYTE WITH INERT WORKING ELECTRODES

* "As received" $LiPF_6$.

FIGURE 4. LINEARLY VARYING POTENTIAL STUDIES OF INORGANIC CATHODES

	Cathode	Electrolyte (1 Molal)	Page
a.	Nickel Hydroxide Cold-Pressed on Copper Expanded Metal.	$LiPF_6^*$-N-Nitroso-dimethylamine	IV-45
b.	Nickel Hydroxide Thin Film.	$LiClO_4$-Propylene Carbonate	IV-46
c.	Silver Oxide Hot- and Cold-Pressed on Silver Expanded Metal.	$LiPF_6^*$-N-Nitroso-dimethylamine	IV-47

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* "As received" $LiPF_6$.

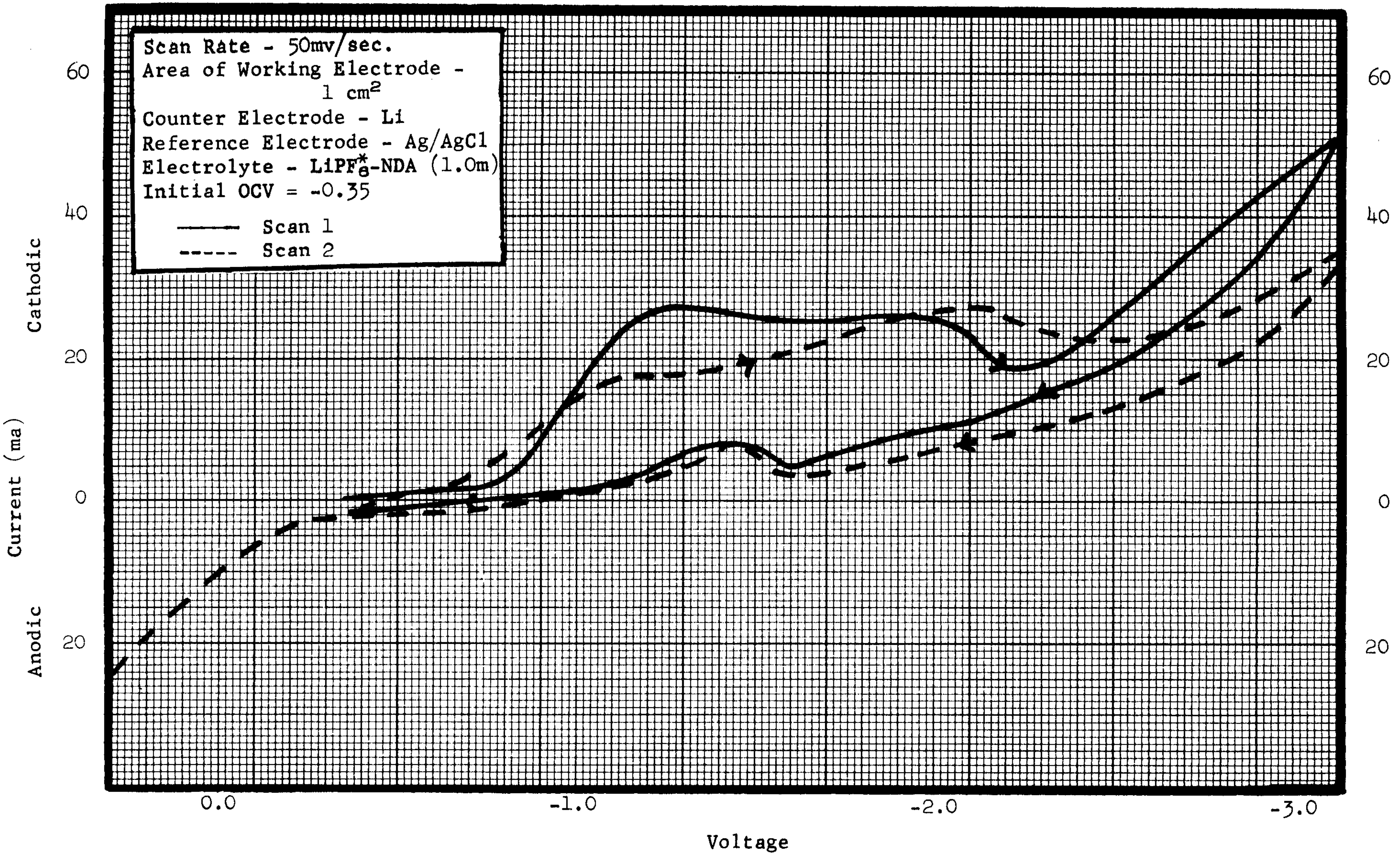

FIGURE 4. LINEARLY VARYING POTENTIAL STUDIES OF INORGANIC CATHODES

Figure 4-a. Nickel Hydroxide Cold-Pressed on Copper Expanded Metal.

* "As received" $LiPF_6$.

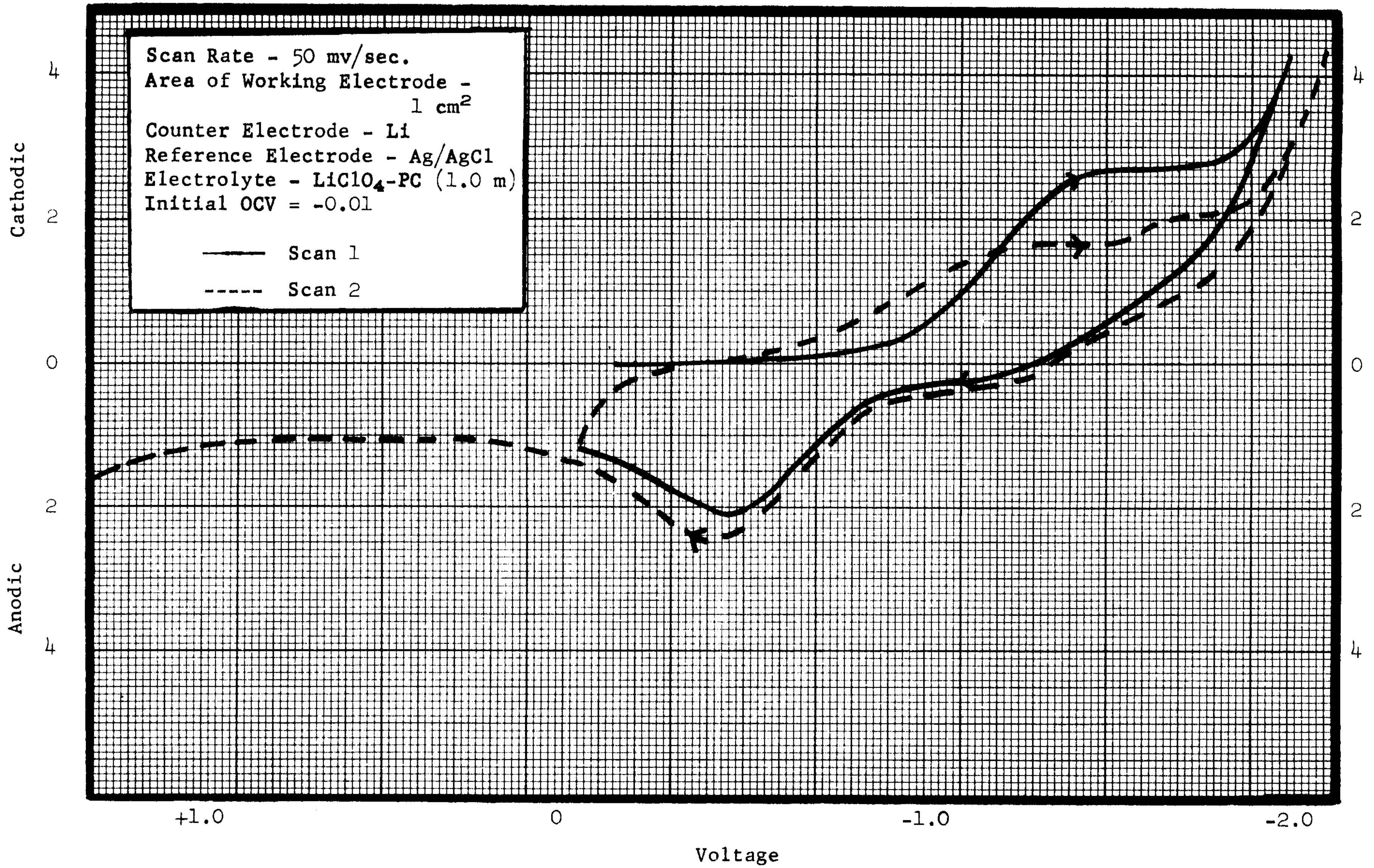

FIGURE 4. LINEARLY VARYING POTENTIAL STUDIES OF INORGANIC CATHODES (Continued)

Figure 4-b. Nickel Hydroxide Thin Film Formed on Nickel by Electrolysis of 1M $Ni(NO_3)_2$.

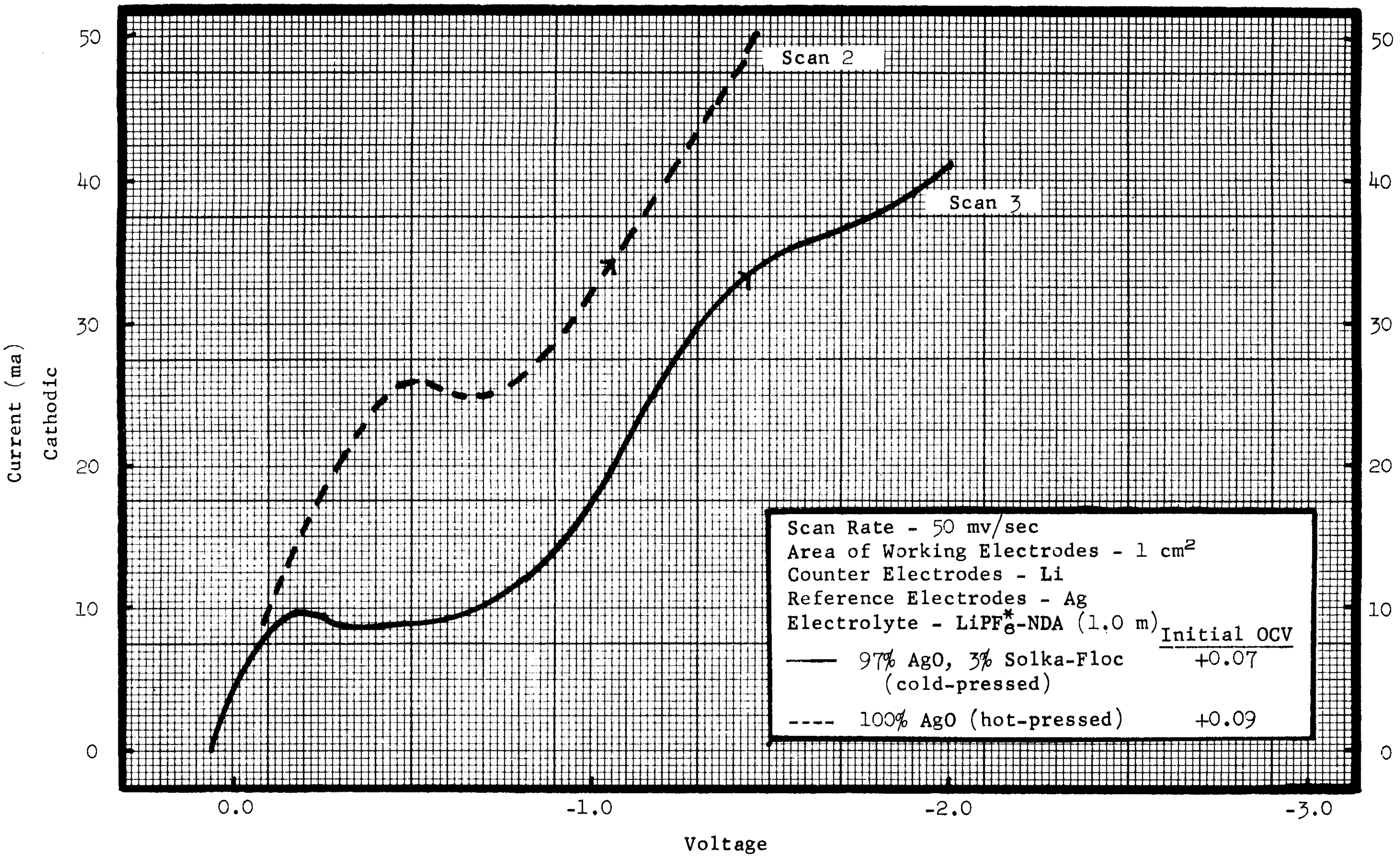

FIGURE 4. LINEARLY VARYING POTENTIAL STUDIES OF INORGANIC CATHODES

Figure 4-c. Silver Oxide Hot- and Cold-Pressed on Silver Expanded Metal.

* "As received" $LiPF_6$.

TABLE IV. ELECTROCHEMICAL HALF-CELL TESTING OF ORGANIC CATHODES

TABLE IV. ELECTROCHEMICAL HALF-CELL TESTING OF ORGANIC CATHODES

A. Pressed Organic Cathodes

1. Physical Details

	Active Cathode Material	Composition of Cathode Mix	Matrix	Method of Preparation	Electrolyte (1 molal)	Counter Electrode	Reference Electrode
a.	ACL-85	ACL-85 (94 wt. %), acetylene black (3 wt. %), Solka-Floc (3 wt. %).	Ag expanded metal	A small amount of the mixture was pressed on a 1cm^2 matrix at a pressure of 10,000 lb/cm^2. One side of the electrode was masked with tape.	$AlCl_3$-AN	Mg	Ag
b.	ACL-85	Same as "a."	Ag expanded metal	Same as "a."	$LiPF_6^*$-DMF	Li	Ag
c.	ACL-85	Same as "a."	Ag expanded metal	Same as "a " but the pressure applied to the electrode was 9,000 lb/cm^2.	$LiPF_6^*$-NDA	Li	Ag

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* "As received" $LiPF_6$.

TABLE IV. ELECTROCHEMICAL HALF-CELL TESTING OF ORGANIC CATHODES (Continued)

A. Pressed Organic Cathodes (Continued)

2. Electrochemical Details

	Active Cathode Material	Electrolyte (1 molal)	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities* (ma/cm^2							
				0	3.0	5.0	10.0	20.0	30.0	40.0	60.0
a.	ACL-85	$AlCl_3$-AN		Note: The cathode disintegrated upon insertion into the electrolyte; therefore, the test was discontinued.							
b.	ACL-85	$LiPF_6$-DMF	Initial	-0.51	**	-0.54	-0.58	-0.78	**	-1.24	-1.34
			Final	-0.5	**	-0.53	-0.66	-0.83	**	-1.12	-3.5 (3 min.)
				Note: The cathode disintegrated considerably in the electrolyte. At the end of the discharges the lithium was black and the electrolyte was yellow.							
c.	ACL-85	$LiPF_6$-NDA	Initial	-0.5	-0.6	-0.66	-0.76	-0.92	-1.1	-1.36	**
			Final	-0.68	-0.63	-0.71	-0.81	-0.99	-1.3	-2.5 (1 min.)	**
				Note: The cathode disintegrated considerably in the electrolyte. The lithium remained unchanged but the reference electrode became covered with a brown-black film.							

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* Duration of discharge at each current density - 5 minutes (unless noted otherwise).

** Not run.

TABLE IV. ELECTROCHEMICAL HALF-CELL TESTING OF ORGANIC CATHODES (Continued)

B. Dissolved Organic Cathodes

1. Physical Details

	Active Cathode Material	Concentration (Molal)	Electrolyte (1 Molal)	Working Electrode	Counter Electrode	Reference Electrode
a.	ACL-85	0.01	LiPF$_6^*$-NDA	HP-10 Speer Carbon	HP-10 Speer Carbon	Ag
b.	ACL-85	Sat. (< 0.2m)	LiPF$_6^*$-NDA**	HP-10 Speer Carbon	HP-10 Speer Carbon	Ag
c.	ACL-85	Sat. (< 0.2m)	LiPF$_6^*$-NDA***	HP-10 Speer Carbon	HP-10 Speer Carbon	Ag

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* The LiPF$_6$ was dried in a nitrogen atmosphere before use.
** Electrolyte was prepared just prior to testing.
*** Electrolyte after three weeks.

TABLE IV. ELECTROCHEMICAL HALF-CELL TESTING OF ORGANIC CATHODES (Continued)

B. Dissolved Organic Cathodes (Continued)

2. Electrochemical Details

	Active Cathode Material (Concentration)	Electrolyte (1 molal)	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities* (ma/cm^2)									
				0	0.05	0.5	2.5	5.0	10.0	12.5	20.0	25.0	30.0
a.	ACL-85 (0.01m)	$LiPF_6$**-NDA	Initial	+0.91	+0.90	+0.84	+0.73	+0.65	***	***	***	***	***
			Final	+0.91	+0.89	+0.78	-1.30	-2.18	***	***	***	***	***
b.	ACL-85 (Sat.)	$LiPF_6$**-NDA	Initial	+1.01	+1.00	+0.95	+0.82	+0.89	+0.66	***	+0.23	***	***
			Final	+1.06	+1.00	+0.89	+0.94	+0.87	+0.54	***	-1.95	***	***
				A second series of discharges was run with new working electrodes for each discharge.									
			Initial	***	***	***	***	***	***	***	+0.15	-0.09	-0.59
			Final	***	***	***	***	***	***	***	-0.82	-1.71	-4.58
				Note: Gassing occurred in the electrolyte initially and during the discharges with the evolution of heat.									
c.	ACL-85 (Sat.)	$LiPF_6$**-NDA	Initial	+0.81	+0.79	+0.67	-0.30	-0.70	***	-1.02	***	-1.50	***
			Final	+0.61	+0.77	+0.27	-0.66	-0.84	***	-1.30	***	-3.67	***
				Note: Gassing occurred in the electrolyte initially and during the discharges.									

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* Duration of discharge at each current density - 5 minutes (unless noted otherwise).

** The $LiPF_6$ was dried in a nitrogen atmosphere before use.

*** Not run.

TABLE V. COULOMBIC EFFICIENCIES* OF DISSOLVED ORGANIC CATHODES

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* Coulombic efficiencies were calculated to a cut-off voltage of 0.2V for the cell.

TABLE V. COULOMBIC EFFICIENCIES OF DISSOLVED ORGANIC CATHODES

Trichloroisocyanuric Acid (ACL-85)

A. Physical Details

	Working Electrode	Counter Electrode	Reference Electrode	Electrolyte (1 molal)	Volume of Electrolyte (ml)
1.	HP-10 Speer Carbon ($2cm^2$)	Mg	Ag	$AlCl_3$-AN	12
2.	HP-10 Speer Carbon ($2cm^2$)	Mg	Ag	$AlCl_3$-AN	12
3.	HP-10 Speer Carbon ($2cm^2$)	Mg	Ag	$AlCl_3$-AN	12
4.	HP-10 Speer Carbon ($2cm^2$)	Li	Ag	$LiPF_6^*$-DMF	12
5.	HP-10 Speer Carbon ($2cm^2$)	Li	Ag	$LiPF_6^*$-DMF	12
6.	HP-10 Speer Carbon ($2cm^2$)	Li	Ag	$LiPF_6^*$-DMF	12
7.	HP-10 Speer Carbon ($2cm^2$)	Li	Ag	$LiPF_6^*$-DMF	12
**8.	HP-10 Speer Carbon ($2cm^2$)	Li	Ag	$LiPF_6^*$-NDA	12
**9.	HP-10 Speer Carbon ($2cm^2$)	Li	Ag	$LiPF_6^*$-NDA	12

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* The $LiPF_6$ was dried in a nitrogen atmosphere before use.

** Reported in the Sixth Quarterly.

TABLE V. COULOMBIC EFFICIENCIES OF DISSOLVED ORGANIC CATHODES (Continued)

Trichloroisocyanuric Acid (ACL-85)

B. Electrochemical Details*

	Weight of Active Material (g)	Milli-equivalents**	Average Current (ma)	Average Cell Voltage	Duration of Test (Minutes)	Coulombs		% Coulombic Efficiency
						Theoretical	Actual	
1.	0.027	0.70	1.94	0.80	38	67.6	4.42	6.5
2.	0.027	0.70	9.9	0.76	12	67.6	7.13	10.5
3.	0.2776	7.17	1.96	1.5	195	691.9	22.9	3.3
4.	0.032	0.83	1.85	0.78	159	80.1	17.65	22.0
5.	0.032	0.83	10.2	1.1	3	80.1	1.84	2.3
6.	0.337	8.71	2.02	1.48	1104	840.5	133.8	15.9
7.	0.337	8.71	9.9	1.3	272	840.5	161.6	19.2
8.	0.035	0.90	1.90	0.88	530	86.9	60.4	69.6
9.	0.035	0.90	10.1	0.64	17	86.9	10.3	11.9

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* All of the discharges were forced.

** Calculated on the basis of a 6 electron change per molecule.

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION

	Solvent	Solute	Working Electrode	Counter Electrode	Page
a.	Dimethylformamide (DMF)	LiCl, $LiPF_6$*	Pyrolytic graphite	Pyrolytic graphite	IV-57
b.	N-Nitrosodimethylamine (NDA)	LiCl, $LiPF_6$*	Pyrolytic graphite	Pyrolytic graphite	IV-59
c.	Propylene Carbonate (PC)	$LiClO_4$, LiCl	Pyrolytic graphite	Pyrolytic graphite	IV-61
d.	DMF, NDA, PC	LiCl	Pyrolytic graphite	Li	IV-62
e.	DMF, NDA, PC	$LiPF_6$*	Pyrolytic graphite	Li	IV-63
f.	DMF	LiCl, $LiPF_6$*, $(C_6H_5)(CH_3)_3NPF_6$	Pyrolytic graphite	Li	IV-64
g.	NDA	KPF_6, $LiPF_6$*, $(CH_3)_4NPF_6$	Pyrolytic graphite	Li	IV-65
h.	PC	$LiClO_4$, LiCl, $LiAlCl_4$, $LiPF_6$*	Pyrolytic graphite	Li	IV-66
i.	NDA	$LiPF_6$*	Copper, Silver	Lithium	IV-67
j.	PC	$LiPF_6$*, $LiClO_4$	Platinum	Platinum	IV-68

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* "As received" $LiPF_6$.

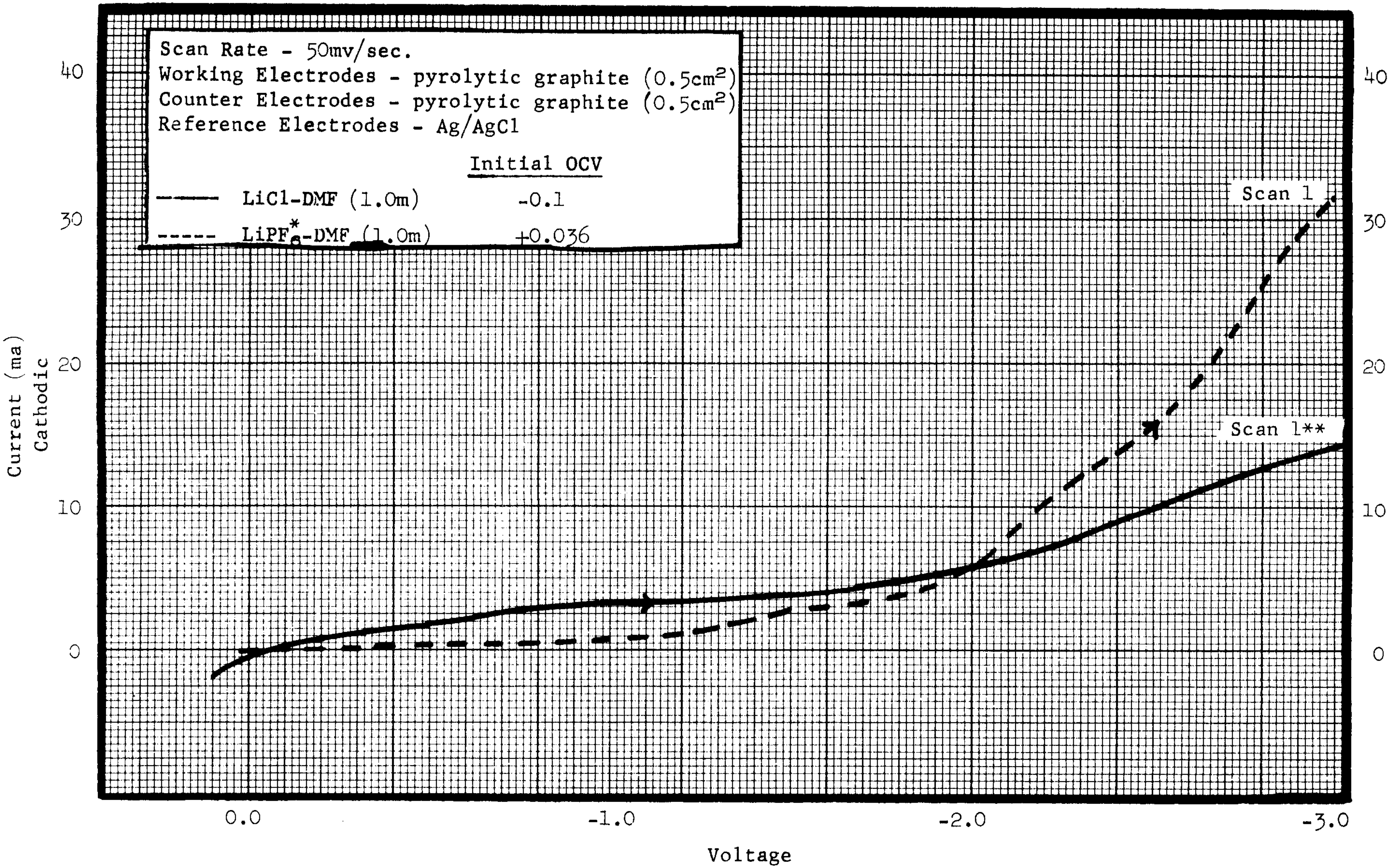

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION

Figure 5-a. Dimethylformamide Solutions of Various Solutes.

* "As received" $LiPF_6$.

** Initial anodic current may be due to products formed while establishing the appropriate current sensitivity for the test.

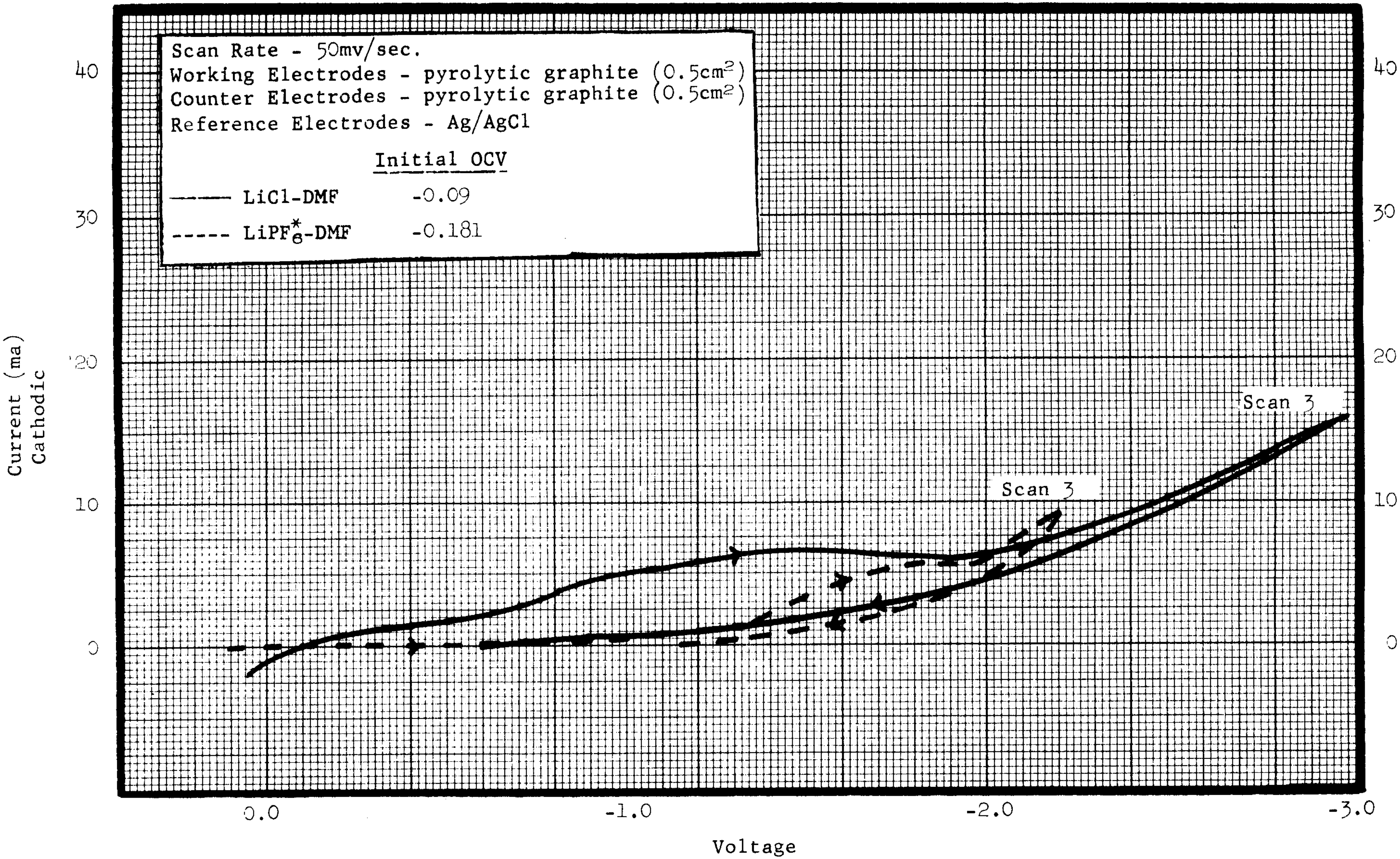

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION (Continued)

Figure 5-a (Con't.) Dimethylformamide Solutions of Various Solutes.

* "As received" LiPF$_6$.

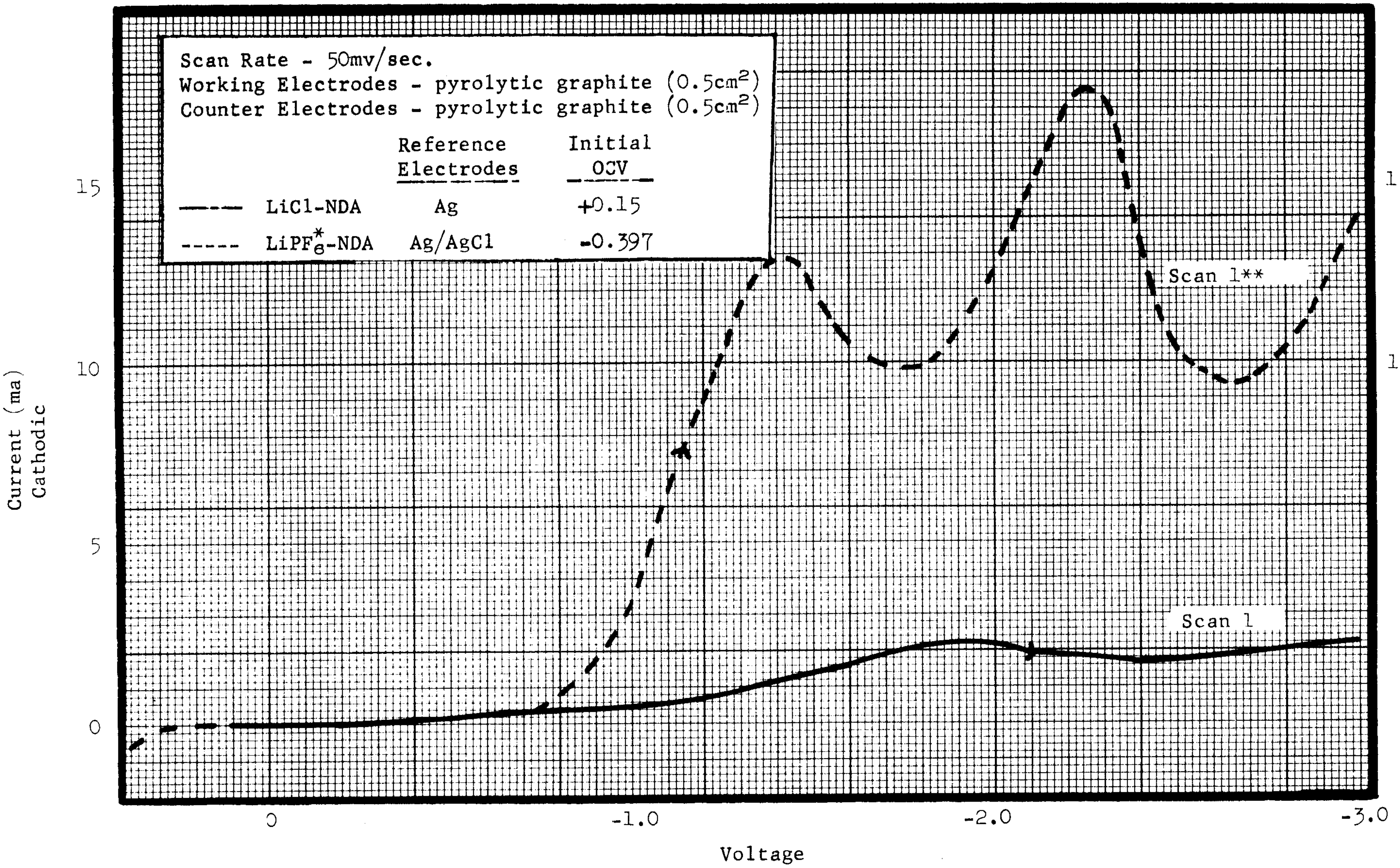

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION (Continued)

Figure 5-b. N-Nitrosodimethylamine Solutions of Various Solutes.

* "As received" $LiPF_6$.

** Initial anodic current may be due to products formed while establishing the appropriate current sensitivity for the test.

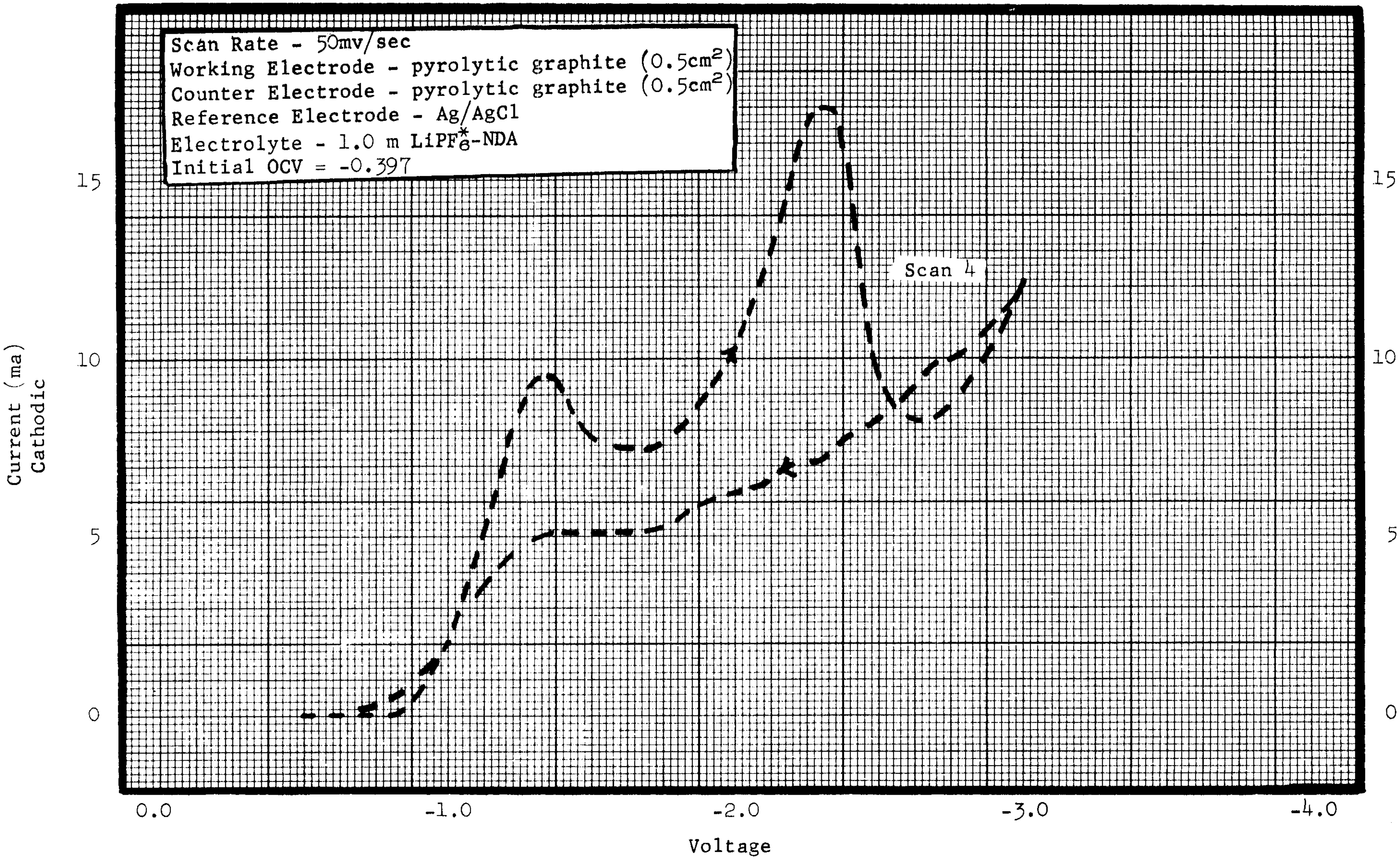

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION (Continued)

Figure 5-b. (Con't.) N-Nitrosodimethylamine Solutions of Various Solutes.

* "As received" $LiPF_6$.

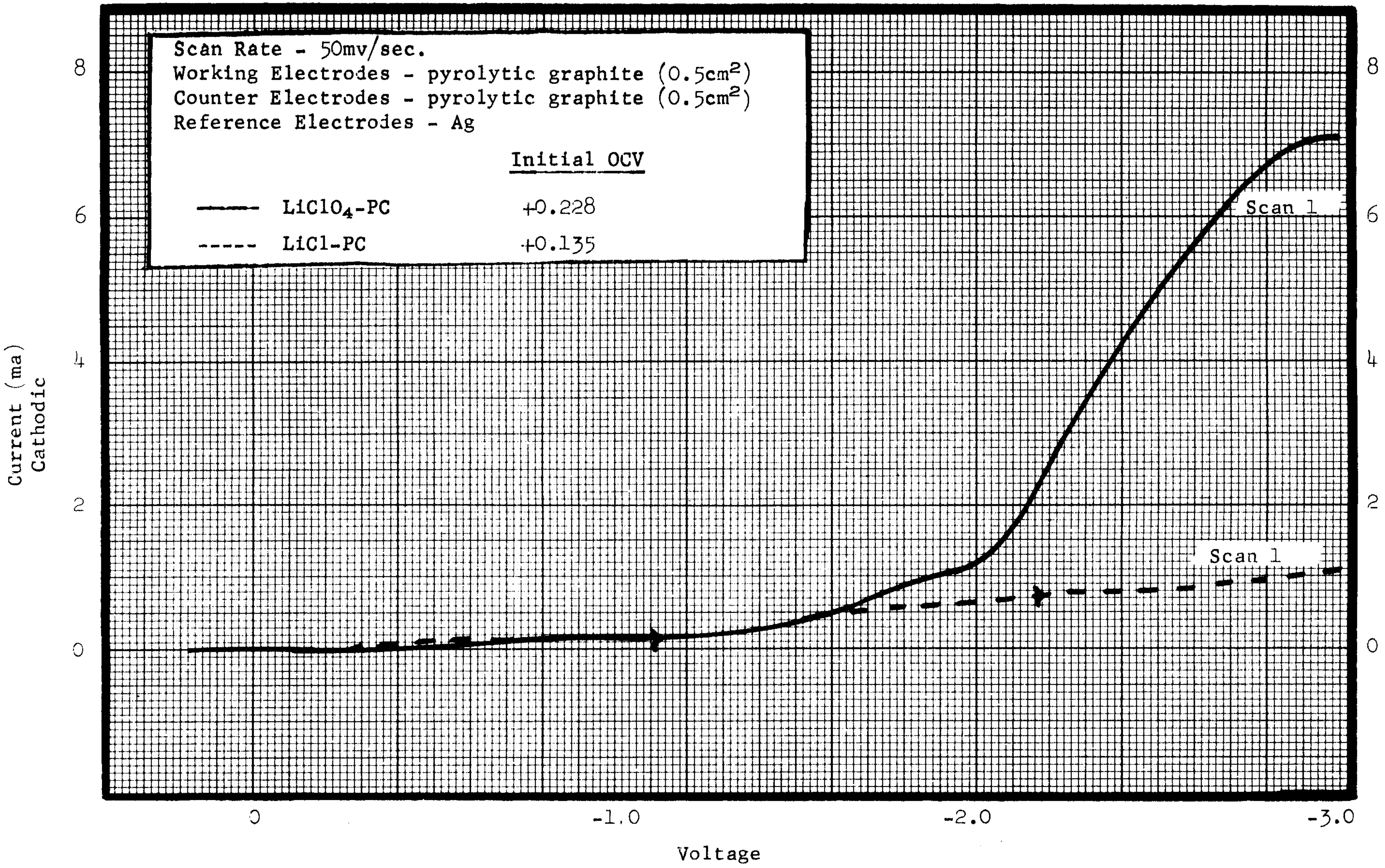

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION (Continued)

Figure 5-c. Propylene Carbonate Solutions of Various Solutes.

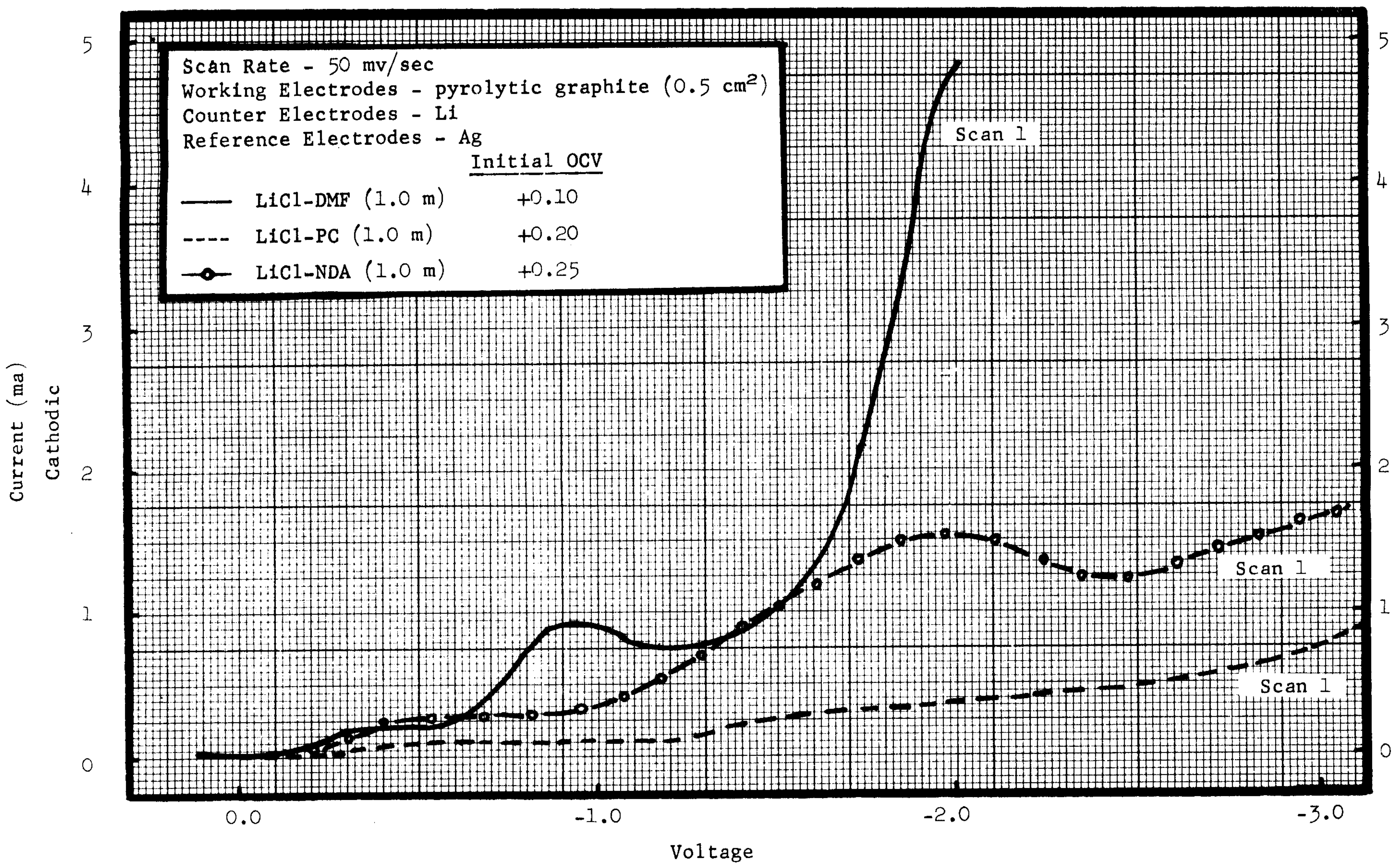

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION

Figure 5-d. LiCl in Various Solvents

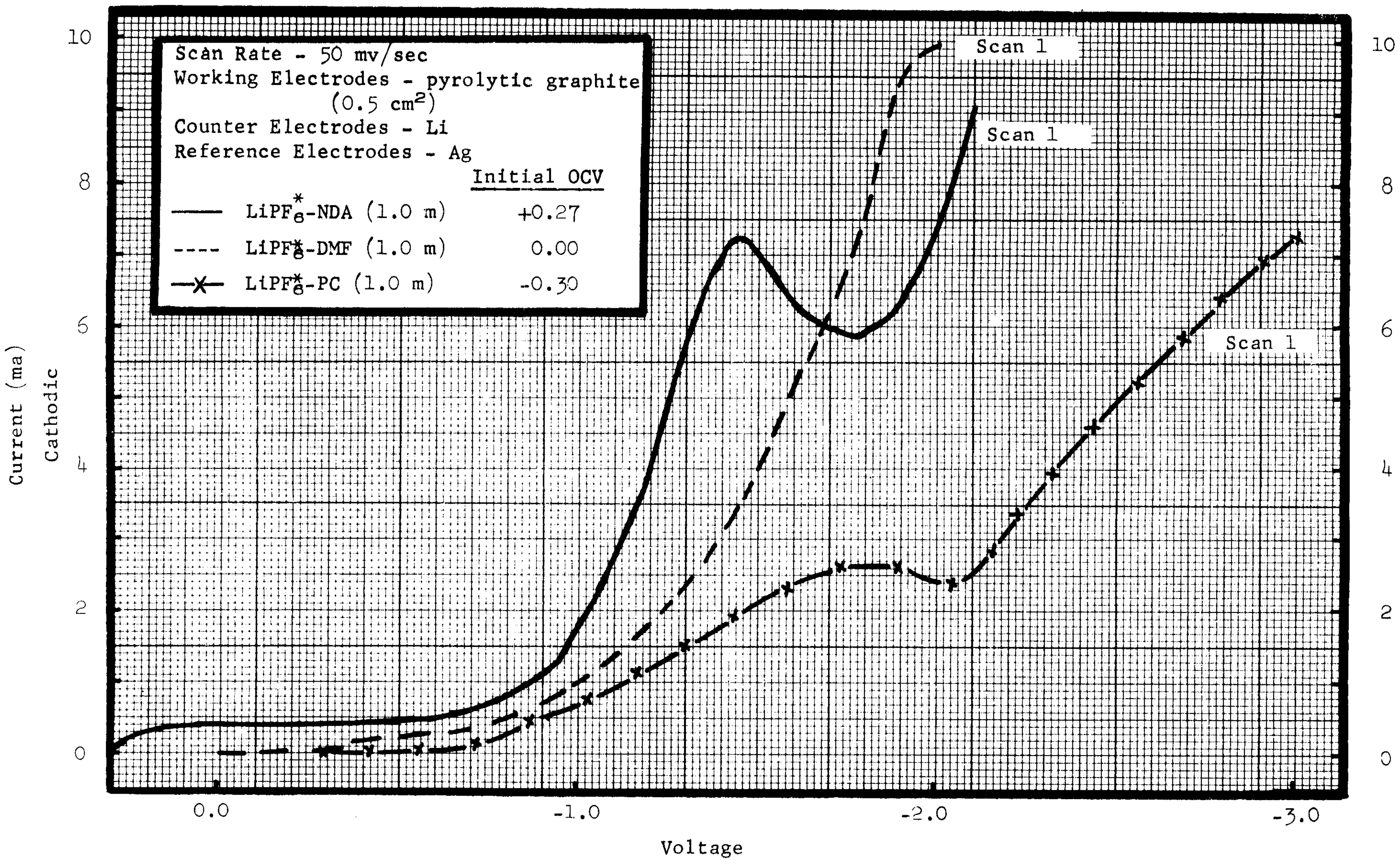

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION (Continued)

Figure 5-e. $LiPF_6^*$ in Various Solvents.

* "As received" $LiPF_6$.

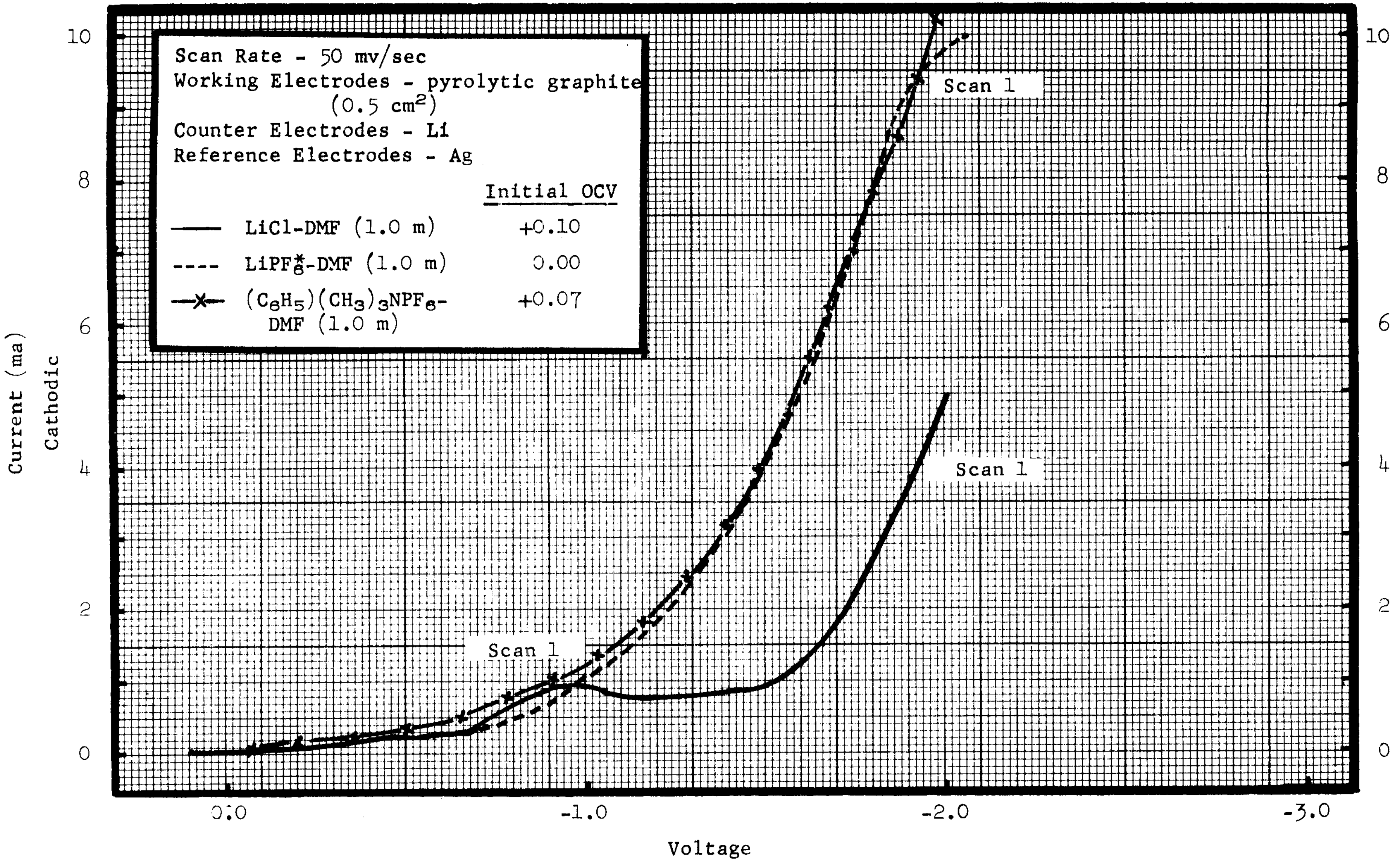

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION (Continued)

Figure 5-f. Dimethylformamide Solutions of Various Solutes.

* "As received" $LiPF_6$.

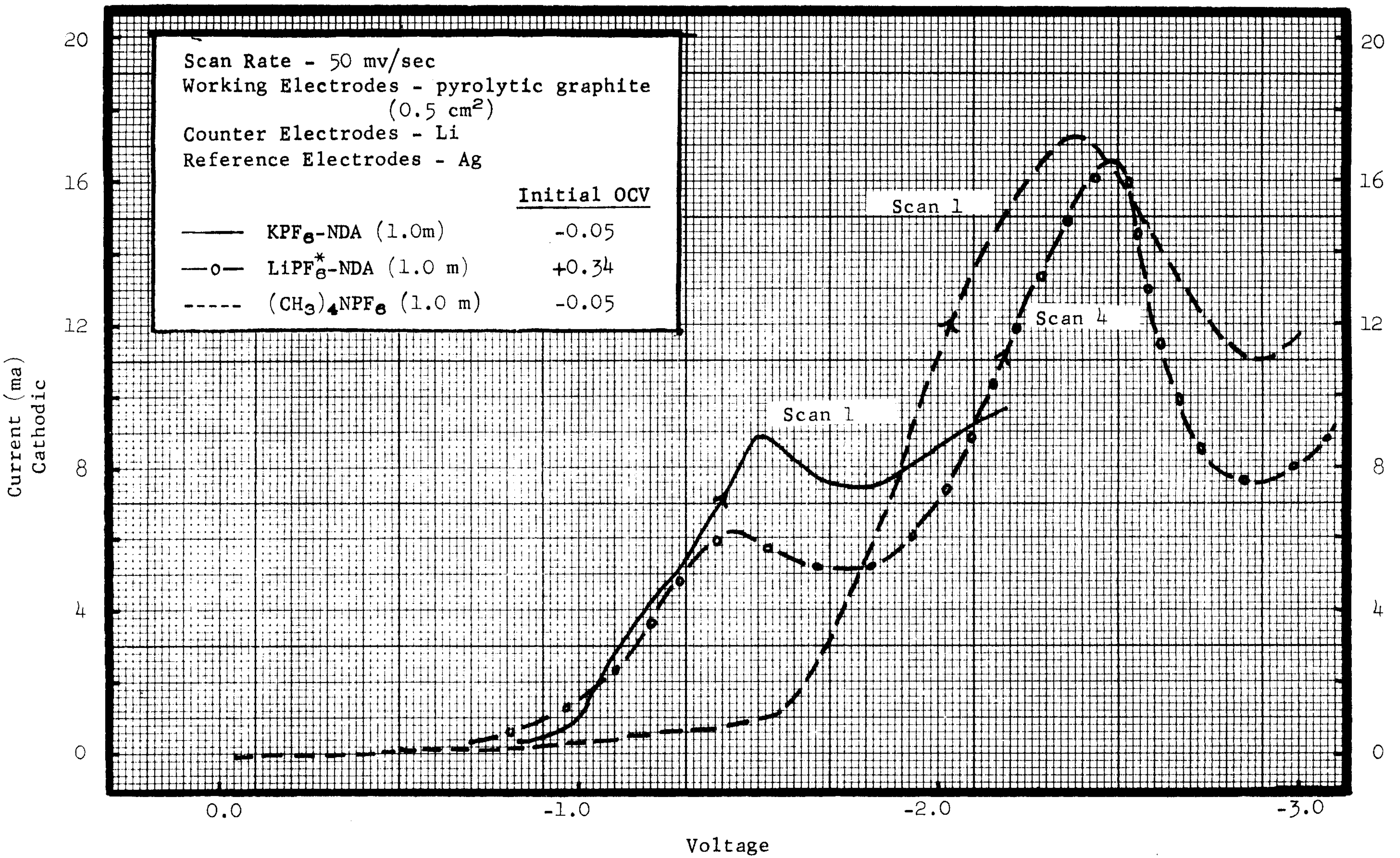

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION (Continued)

Figure 5-g. N-Nitrosodimethylamine Solutions of Various Solutes.

* "As received" $LiPF_6$.

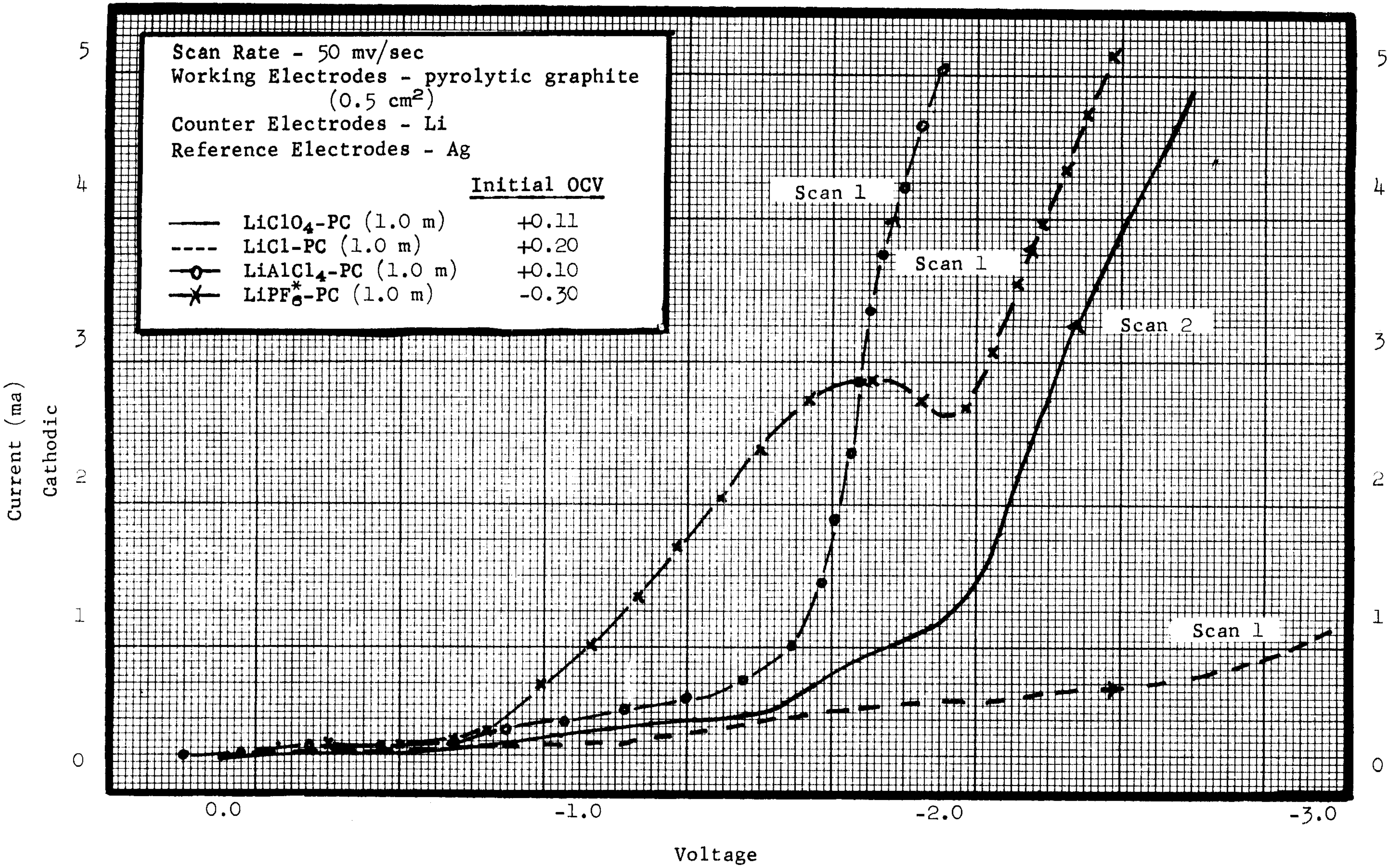

FIGURE 5. <u>LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION</u> (Continued)

Figure 5-h. Propylene Carbonate Solutions of Various Solutes.

* "As received" $LiPF_6$.

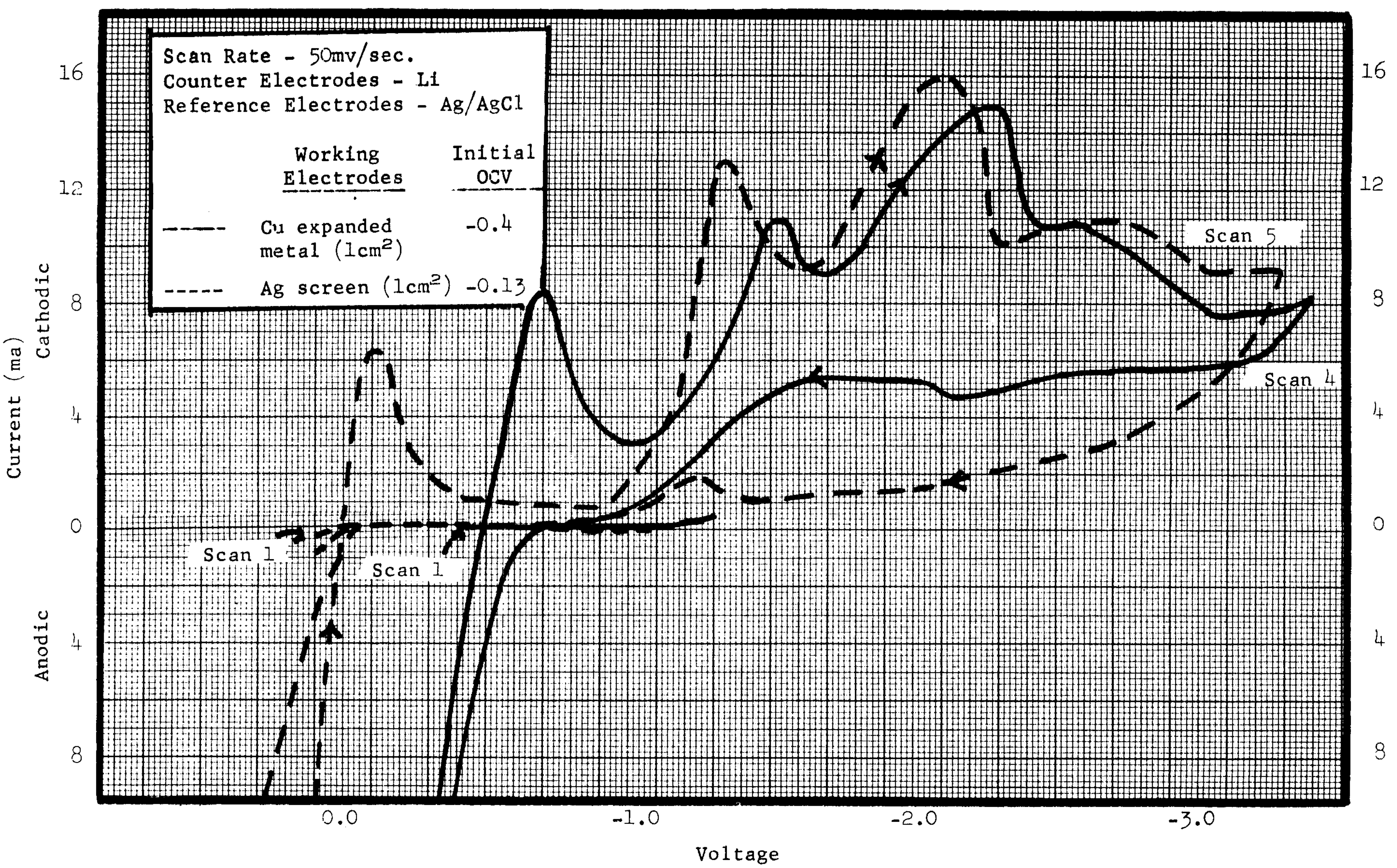

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION (Continued)

Figure 5-i. LVP Scans of 1 Molal $LiPF_6^*$-N-Nitrosodimethylamine Using Different Working Electrodes.

* "As received" $LiPF_6$.

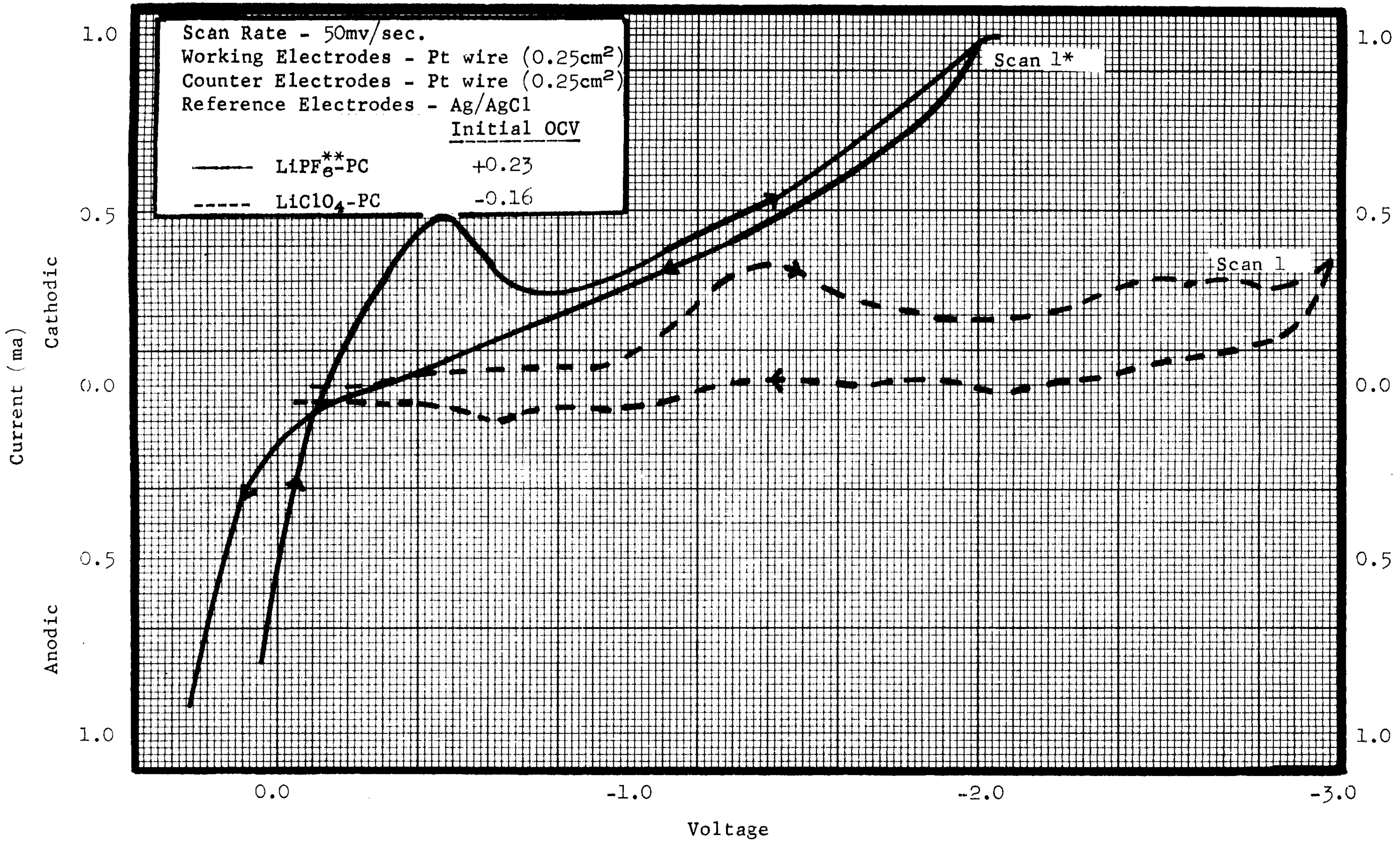

FIGURE 5. LINEARLY VARYING POTENTIAL STUDIES OF ELECTROLYTE DECOMPOSITION (Continued)

Figure 5-j. Propylene Carbonate Solutions of Various Solutes.

* Initial anodic current may be due to products formed while establishing the appropriate current sensitivity for the test.

** "As received" $LiPF_6$.

FIGURE 6. LINEARLY VARYING POTENTIAL STUDIES TO DETERMINE THE IMPURITY PRESENT IN $LiPF_6^*$-N-NITROSODIMETHYLAMINE

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* "As received" $LiPF_6$.

** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

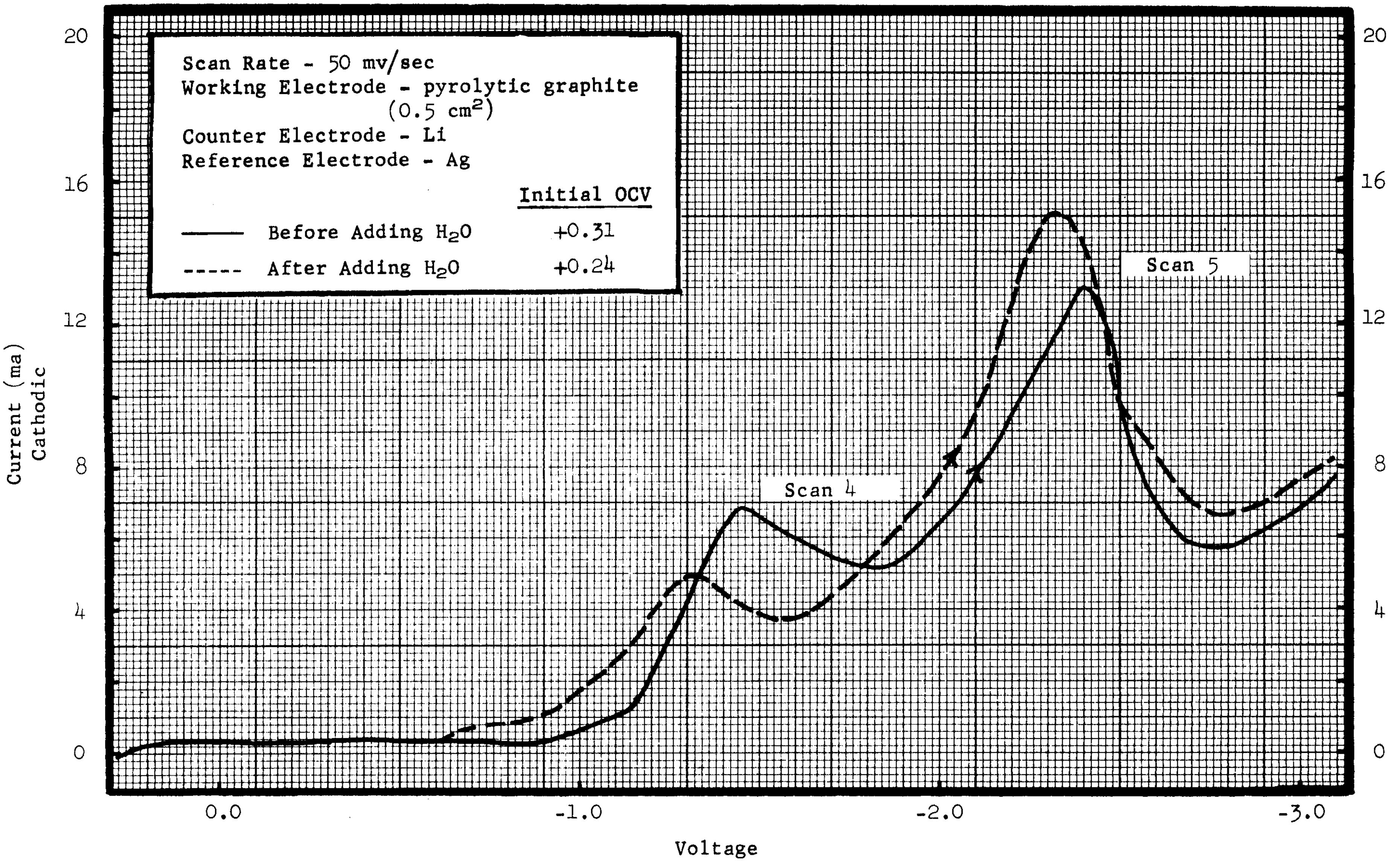

FIGURE 6. LINEARLY VARYING POTENTIAL STUDIES TO DETERMINE THE IMPURITY PRESENT IN $LiPF_6^*$-N-NITROSODIMETHYLAMINE

Figure 6-a. The Effect of Adding Water to One Molal $LiPF_6^*$-N-Nitrosodimethylamine.

* "As received" $LiPF_6$.

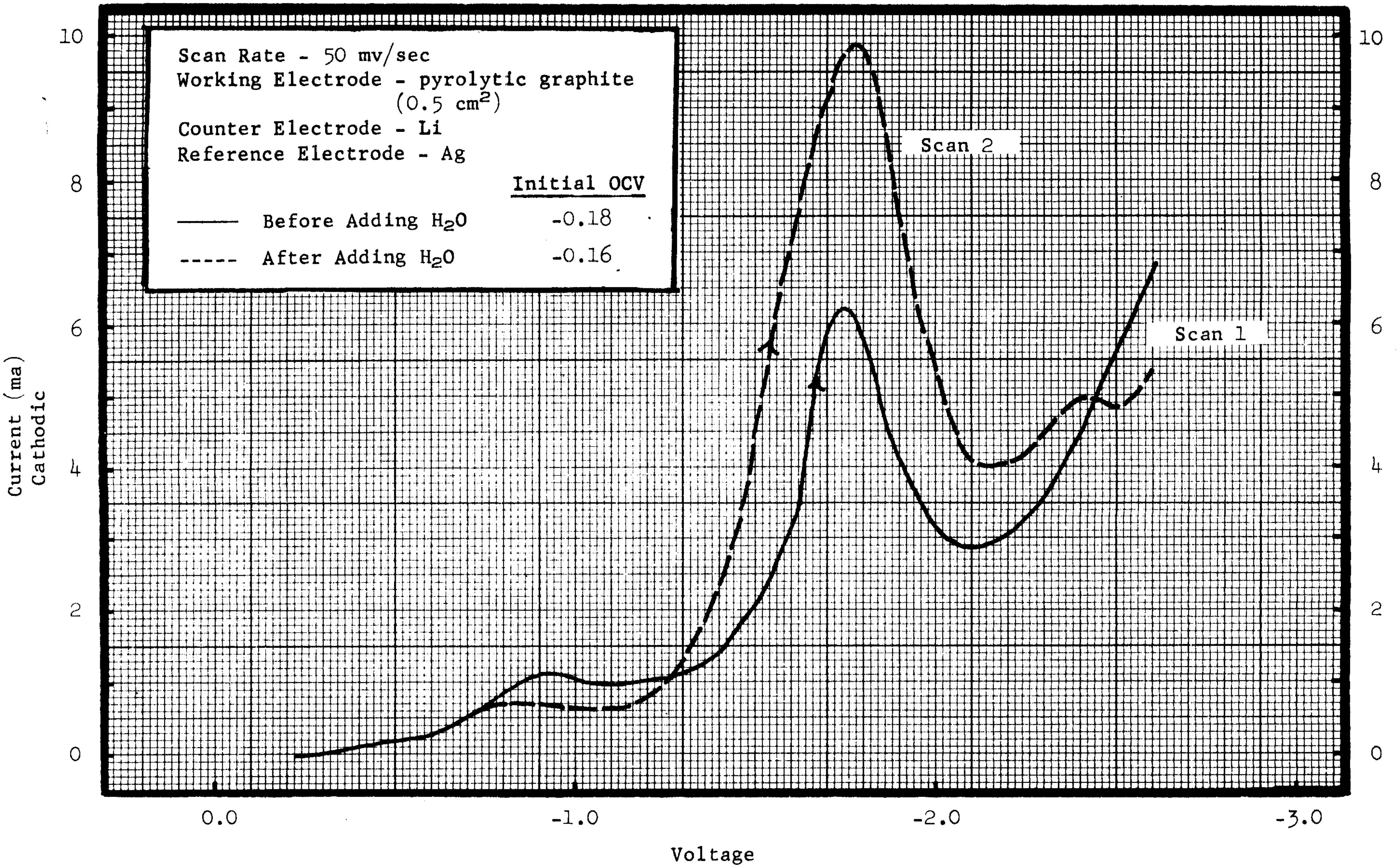

FIGURE 6. LINEARLY VARYING POTENTIAL STUDIES TO DETERMINE THE IMPURITY PRESENT IN $LiPF_6^*$-N-NITROSODIMETHYLAMINE (Continued)

Figure 6-b. The Effect of Adding Water to One Molal $LiPF_6^{**}$-N-Nitrosodimethylamine.

* "As received" $LiPF_6$.

** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

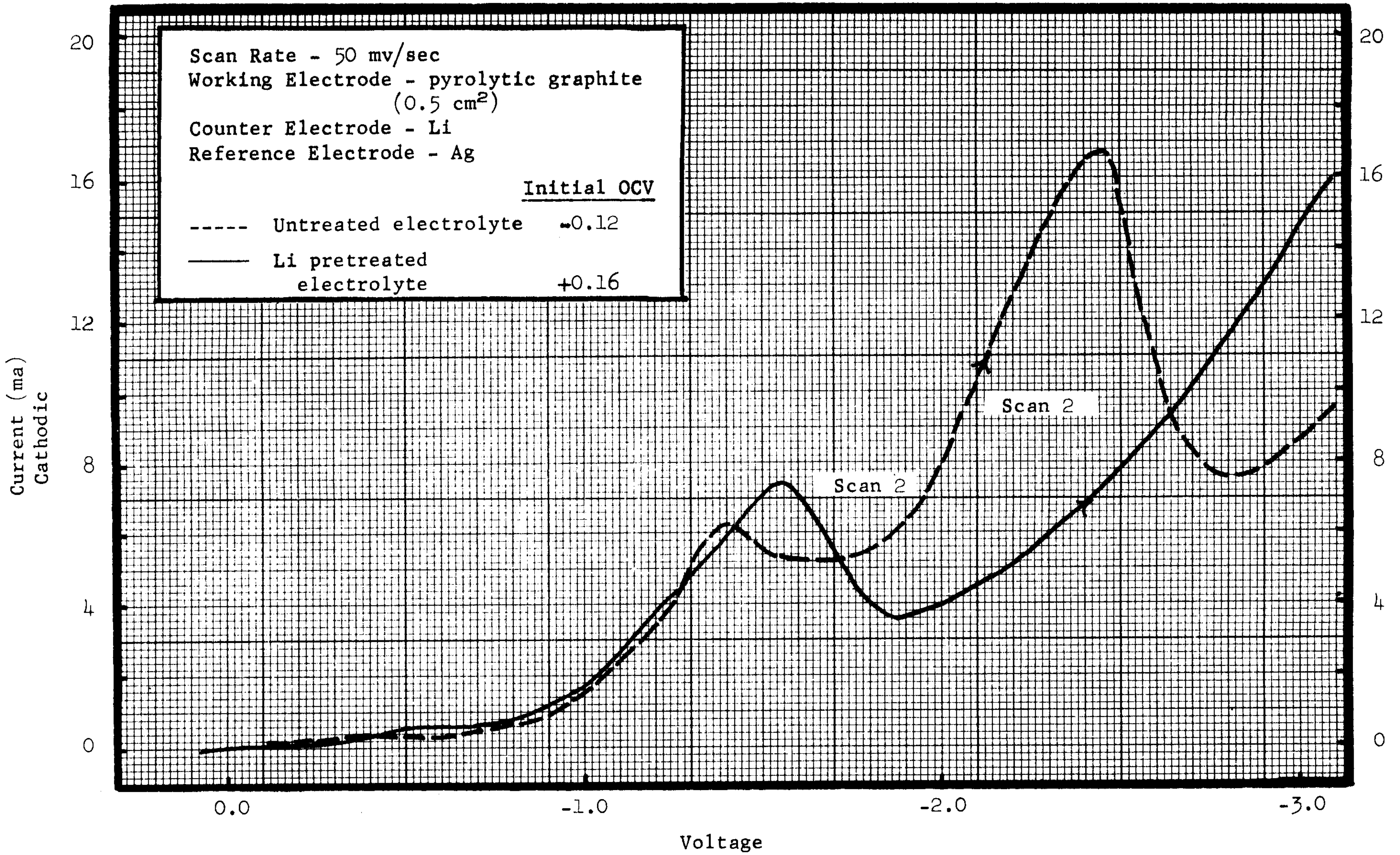

FIGURE 6. <u>LINEARLY VARYING POTENTIAL STUDIES TO DETERMINE THE IMPURITY PRESENT IN $LiPF_6^*$-N-NITROSODIMETHYLAMINE</u> (Continued)

Figure 6-c. The Effect of Pretreating One Molal $LiPF_6^*$-NDA With Lithium.

* "As received" $LiPF_6$.

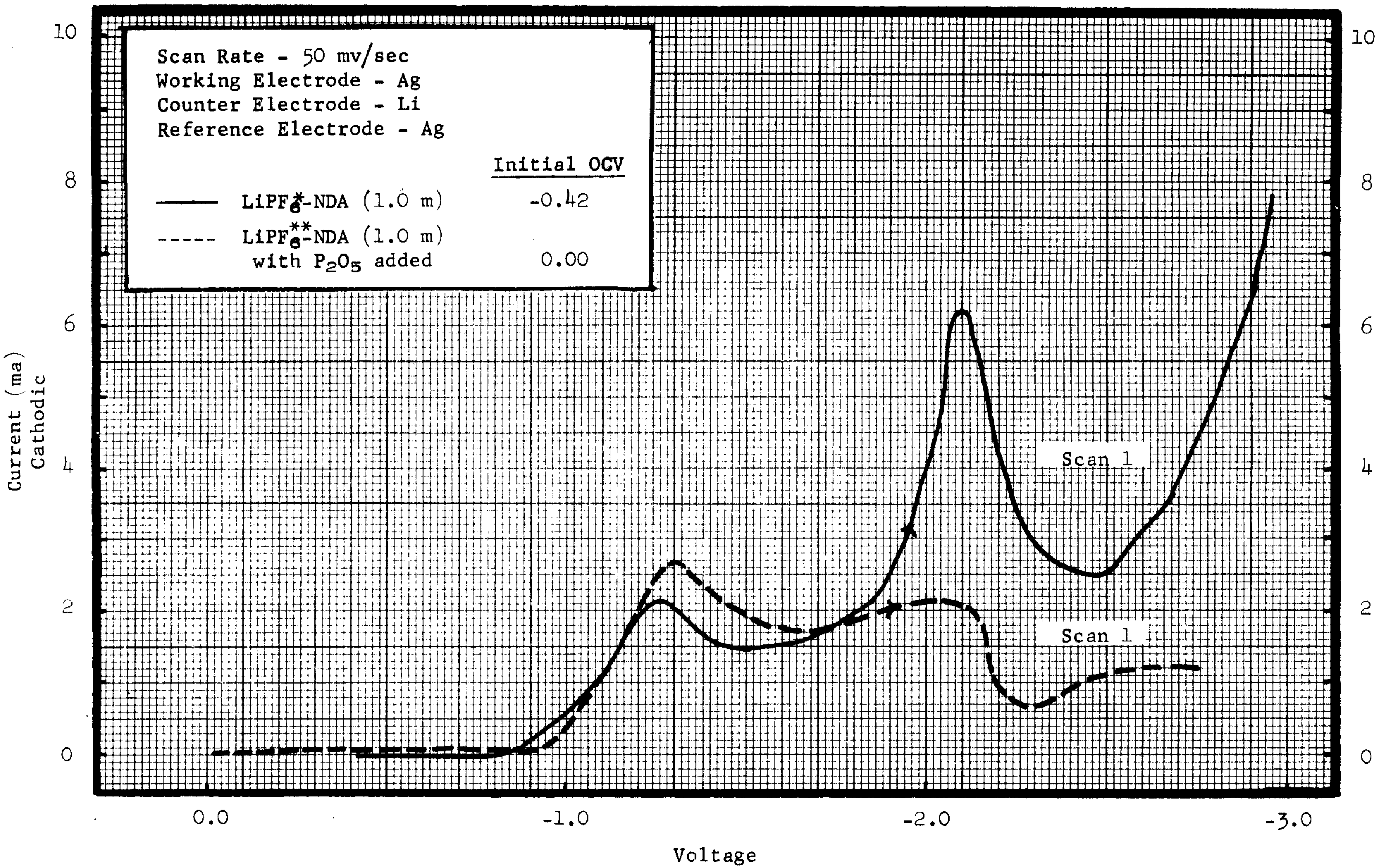

FIGURE 6. LINEARLY VARYING POTENTIAL STUDIES TO DETERMINE THE IMPURITY PRESENT IN LiPF$_6^{**}$-N-NITROSODIMETHYLAMINE (Continued)

Figure 6-d. The Effect of Adding Phosphorous Pentoxide to One Molal LiPF$_6^{**}$-N-Nitrosodimethylamine.

* The LiPF$_6$ was dried at 110°C in a nitrogen atmosphere before use.
** "As received" LiPF$_6$.

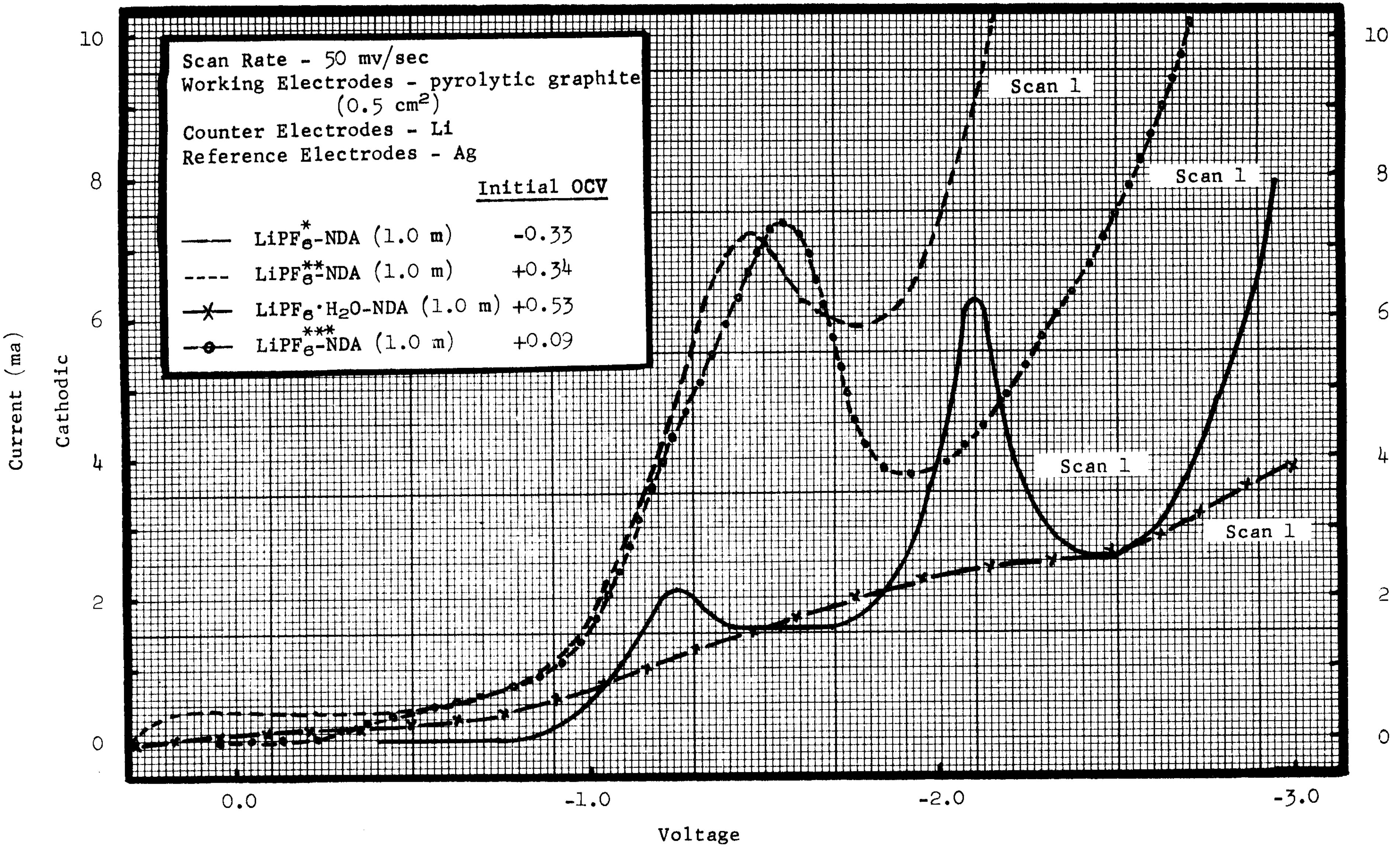

FIGURE 6. LINEARLY VARYING POTENTIAL STUDIES TO DETERMINE THE IMPURITY PRESENT IN $LiPF_6^{**}$N-NITROSODIMETHYLAMINE (Continued)

Figure 6-e. $LiPF_6$- and $LiPF_6 \cdot H_2O$-N-Nitrosodimethylamine Electrolytes.

* The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.
** "As received" $LiPF_6$.
*** The $LiPF_6$(as received)-NDA solution was pretreated with Li for 48 hours before use.

TABLE VI. CHEMICAL STABILITY OF ELECTRODE MATERIALS IN ELECTROLYTE SOLUTIONS

A. Stability in N-Nitrosodimethylamine Solutions

	Solute	Electrode Material	Time of Measurement	Weight of Electrode Material (g)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Blank Electrolyte	Visible Change After Exposure
1.	None	Li	Initial	0.1632	1.07×10^{-4} (24°C)	1.08×10^{-4} (24°C)	A gray film formed on the lithium after one week. No further changes noted to date.
			After Two Days	*	1.02×10^{-4} (26°C)	1.31×10^{-4} (26°C)	
			After One Week	*	1.17×10^{-4} (26°C)	1.56×10^{-4} (26°C)	
			After Three Weeks	*	1.29×10^{-4} (24°C)	1.44×10^{-4} (24°C)	
2.	$LiPF_6$ (as received)	Li	Initial	0.2069	2.08×10^{-2} (24°C)	2.03×10^{-2} (24°C)	Gassing occurred initially. A white film formed on the lithium during the first day. No further changes noted to date.
			After Two Days	*	1.98×10^{-2} (26°C)	2.03×10^{-2} (26°C)	
			After One Week	*	1.98×10^{-2} (26°C)	2.06×10^{-2} (26°C)	
			After Three Weeks	*	1.96×10^{-2} (24°C)	2.03×10^{-2} (24°C)	
3.	$LiPF_6$**	Li	Initial	0.2205	1.90×10^{-2} (24°C)	1.87×10^{-2} (24°C)	Gassing occurred initially. A white film formed on the lithium during the first day. No further changes noted to date.
			After Two Days	*	1.82×10^{-2} (26°C)	1.88×10^{-2} (26°C)	
			After One Week	*	1.85×10^{-2} (26°C)	1.17×10^{-2} (26°C)	
			After Three Weeks	*	1.81×10^{-2} (24°C)	1.77×10^{-2} (24°C)	

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* The stability tests are still in progress. Weight loss will be calculated at the end of the tests.

** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

TABLE VI. CHEMICAL STABILITY OF ELECTRODE MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

A. Stability in N-Nitrosodimethylamine Solutions (Continued)

Solute	Electrode Material	Time of Measurement	Weight of Electrode Material (g)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Exposure
4. None	CuF_2	Initial	0.4576	1.05×10^{-4} (24°C)	1.08×10^{-4} (24°C)	No changes noted to date.
		After Two Days	*	1.26×10^{-4} (26°C)	1.31×10^{-4} (26°C)	
		After One Week	*	1.42×10^{-4} (26°C)	1.56×10^{-4} (26°C)	
		After Three Weeks	*	1.71×10^{-4} (24°C)	1.44×10^{-4} (24°C)	
5. $LiPF_6$ (as received)	CuF_2	Initial	0.3179	1.98×10^{-2} (24°C)	2.03×10^{-2} (24°C)	The cathode pellet remained intact. The electrolyte changed from yellow to green after two days. No further changes noted to date.
		After Two Days	*	1.81×10^{-2} (26°C)	2.03×10^{-2} (26°C)	
		After One Week	*	2.07×10^{-2} (26°C)	2.06×10^{-2} (26°C)	
		After Three Weeks	*	1.91×10^{-2} (24°C)	2.03×10^{-2} (26°C)	
6. $LiPF_6$**	CuF_2	Initial	0.1932	1.89×10^{-2} (24°C)	1.87×10^{-2} (24°C)	The cathode pellet remained intact. The electrolyte changed from yellow to green after two days. No further changes to date.
		After Two Days	*	1.86×10^{-2} (26°C)	1.88×10^{-2} (26°C)	
		After One Week	*	1.89×10^{-2} (26°C)	1.17×10^{-2} (26°C)	
		After Three Weeks	*	1.83×10^{-2} (24°C)	1.77×10^{-2} (24°C)	

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* The stability tests are still in progress. Weight loss will be calculated at the end of the tests.

** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

TABLE VI. CHEMICAL STABILITY OF ELECTRODE MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

A. Stability in N-Nitrosodimethylamine Solutions (Continued)

Solute	Electrode Material	Time of Measurement	Weight of Electrode Material (g)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Exposure
7. None	AgO	Initial	0.3591	7.96×10^{-5} (29°C)	8.03×10^{-5} (29°C)	The cathode pellet remained intact. The electrolyte changed from yellow to green after four weeks. No further changes noted to date.
		After One Day	*	9.59×10^{-5} (26°C)	8.22×10^{-5} (26°C)	
		After One Week	*	1.07×10^{-4} (27°C)	8.58×10^{-5} (27°C)	
		After Two Weeks	*	1.14×10^{-4} (28°C)	1.12×10^{-4} (28°C)	
		After Three Weeks	*	1.22×10^{-4} (26°C)	1.07×10^{-4} (26°C)	
		After Four Weeks	*	1.45×10^{-4} (27°C)	1.11×10^{-4} (27°C)	
8. $LiPF_6$ (as received)	AgO	Initial	0.4480	2.04×10^{-2} (29°C)	2.04×10^{-2} (29°C)	Moderate gassing occurred initially. A white precipitate formed during the first day. The cathode pellet disintegrated considerably. No further changes noted to date.
		After One Day	*	2.00×10^{-2} (26°C)	1.98×10^{-2} (26°C)	
		After One Week	*	2.03×10^{-2} (27°C)	2.06×10^{-2} (27°C)	
		After Two Weeks	*	2.07×10^{-2} (28°C)	2.06×10^{-2} (28°C)	
		After Three Weeks	*	2.06×10^{-2} (26°C)	1.95×10^{-2} (26°C)	
		After Four Weeks	*	2.05×10^{-2} (27°C)	1.94×10^{-2} (27°C)	

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* The stability tests are still in progress. Weight loss will be calculated at the end of the tests.

TABLE VI. CHEMICAL STABILITY OF ELECTRODE MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

A. Stability in N-Nitrosodimethylamine Solutions (Continued)

Solute	Electrode Material	Time of Measurement	Weight of Electrode Material (g)	Specific Conductance (ohm^{-1} cm^{-1})		Visible Change After Exposure
				Exposed Electrolyte	Blank Electrolyte	
9. LiPF$_6$*	AgO	Initial	0.4071	1.86×10^{-2} (29°C)	1.93×10^{-2} (29°C)	Slight gassing occurred initially. A small amount of white precipitate formed after two weeks. The cathode pellet remained intact. No further changes noted to date.
		After One Day	**	1.89×10^{-2} (26°C)	1.83×10^{-2} (26°C)	
		After One Week	**	1.90×10^{-2} (27°C)	1.91×10^{-2} (27°C)	
		After Two Weeks	**	1.96×10^{-2} (28°C)	1.84×10^{-2} (28°C)	
		After Three Weeks	**	1.91×10^{-2} (26°C)	1.87×10^{-2} (26°C)	
		After Four Weeks	**	1.94×10^{-2} (27°C)	1.92×10^{-2} (27°C)	
10. LiPF$_6$ (as received)	CuF$_2$ + Li	Initial	0.0691(Li), 0.2372*** (CuF$_2$)	2.06×10^{-2} (28°C)	2.01×10^{-2} (28°C)	Gassing occurred initially and the solution turned green. The cathode pellet disintegrated completely after four weeks. No further changes noted to date.
		After One Day	**	1.95×10^{-2} (27°C)	1.96×10^{-2} (27°C)	
		After One Week	**	1.98×10^{-2} (26°C)	1.91×10^{-2} (26°C)	
		After Two Weeks	**	2.01×10^{-2} (27°C)	2.01×10^{-2} (27°C)	
		After Three Weeks	**	1.97×10^{-2} (27°C)	2.06×10^{-2} (27°C)	
		After Four Weeks	**	1.89×10^{-2} (27°C)	2.01×10^{-2} (27°C)	

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* The LiPF$_6$ was dried at 110°C in a nitrogen atmosphere before use.

** The stability tests are still in progress. Weight loss will be calculated at the end of the tests.

*** Includes substrate weight.

TABLE VI. CHEMICAL STABILITY OF ELECTRODE MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

A. Stability in N-Nitrosodimethylamine Solutions (Continued)

	Solute	Electrode Material	Time of Measurement	Weight of Electrode Material (g)	Specific Conductance (ohm^{-1} cm^{-1})		Visible Change After Exposure
					Exposed Electrolyte	Blank Electrolyte	
11.	$LiPF_6$*	CuF_2 + Li	Initial	0.0643(Li), 0.2523** (CuF_2)	1.89×10^{-2} (28°C)	1.94×10^{-2} (28°C)	The solution turned green when the cathode was inserted. After four weeks the cathode pellet had disintegrated completely. No further changes noted to date.
			After One Day	***	1.92×10^{-2} (27°C)	1.92×10^{-2} (27°C)	
			After One Week	***	1.91×10^{-2} (26°C)	1.86×10^{-2} (26°C)	
			After Two Weeks	***	1.87×10^{-2} (27°C)	1.93×10^{-2} (27°C)	
			After Three Weeks	***	1.93×10^{-2} (27°C)	1.90×10^{-2} (27°C)	
			After Four Weeks	***	1.90×10^{-2} (27°C)	1.92×10^{-2} (27°C)	
12.	None	AgO + Li	Initial	0.0756(Li), 0.3237(AgO)	1.09×10^{-4} (29°C)	8.03×10^{-5} (29°C)	The cathode pellet remained intact. After four weeks the solution had changed from yellow to yellow-green and was slightly turbid. No further changes to date.
			After One Day	***	1.06×10^{-4} (26°C)	8.22×10^{-5} (26°C)	
			After One Week	***	1.21×10^{-4} (27°C)	8.58×10^{-5} (27°C)	
			After Two Weeks	***	1.27×10^{-4} (28°C)	1.12×10^{-4} (28°C)	
			After Three Weeks	***	1.27×10^{-4} (26°C)	1.07×10^{-4} (26°C)	
			After Four Weeks	***	1.37×10^{-4} (27°C)	1.11×10^{-4} (27°C)	

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* The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

** Includes substrate weight.

*** The stability tests are still in progress. Weight loss will be calculated at the end of the tests.

TABLE VI. CHEMICAL STABILITY OF ELECTRODE MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

A. Stability in N-Nitrosodimethylamine Solutions (Continued)

	Solute	Electrode Material	Time of Measurement	Weight of Electrode Material (g)	Specific Conductance (ohm^{-1} cm^{-1})		Visible Change After Exposure
					Exposed Electrolyte	Blank Electrolyte	
13.	$LiPF_6$ (as received)	AgO + Li	Initial	0.0921(Li) 0.2960(AgO)	2.05×10^{-2} (29°C)	2.04×10^{-2} (29°C)	Moderate gassing occurred initially. A white precipitate formed during the first day. After four weeks the solution was dark brown and turbid. A black precipitate was present. No further changes noted to date.
			After One Day	*	1.96×10^{-2} (26°C)	1.98×10^{-2} (26°C)	
			After One Week	*	1.99×10^{-2} (27°C)	2.06×10^{-2} (27°C)	
			After Two Weeks	*	1.97×10^{-2} (28°C)	2.06×10^{-2} (28°C)	
			After Three Weeks	*	1.93×10^{-2} (26°C)	1.95×10^{-2} (26°C)	
			After Four Weeks	*	1.93×10^{-2} (27°C)	1.94×10^{-2} (27°C)	
14.	$LiPF_6$**	AgO + Li	Initial	0.0904(Li) 0.4209(AgO)	1.94×10^{-2} (29°C)	1.93×10^{-2} (29°C)	Slight gassing occurred initially. The lithium was black after one week. After four weeks the solution was dark green and a black precipitate was present. No further changes noted to date.
			After One Day	*	1.94×10^{-2} (26°C)	1.83×10^{-2} (26°C)	
			After One Week	*	1.91×10^{-2} (27°C)	1.91×10^{-2} (27°C)	
			After Two Weeks	*	1.96×10^{-2} (28°C)	1.84×10^{-2} (28°C)	
			After Three Weeks	*	1.79×10^{-2} (26°C)	1.87×10^{-2} (26°C)	
			After Four Weeks	*	1.95×10^{-2} (27°C)	1.92×10^{-2} (27°C)	

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* The stability tests are still in progress. Weight loss will be calculated at the end of the tests.

** The $LiPF_6$ was dried at 110°C in a nitrogen atmosphere before use.

TABLE VII. FULL CELL TESTING OF SEPARATOR MATERIALS

Epoxy Membrane Separator

Cell Details:

Anode - lithium. Area = ca. 1cm^2.
Cathode - $CuCl_2 \cdot 2H_2O$ (saturated) in 1 molal N-phenyl-N,N,N-trimethylammonium hexafluorophosphate $[(C_6H_5)(CH_3)_3NPF_6]$-dimethylformamide (DMF).
Cathodic current collector - copper foil. Area = ca. 1cm^2.
Electrolyte - 1 molal $(C_6H_5)(CH_3)_3NPF_6$-DMF.
Membrane - Versapor epoxy No. 6429 (Gelman).
Reference electrodes - Ag screen. Area = ca. 0.5cm^2.
Cell construction - The membrane was placed between two polypropylene blocks. The blocks were bolted together so that a 1x1x1cm well was located on each side of the membrane.

Time (Minutes)	Cell Voltage	Anode Voltage (vs. Ag Reference Electrode*)	Cathode Voltage (vs. Ag Reference Electrode**)	Anode Reference Electrode vs. Cathode Reference Electrode	Current (ma)
0	3.32	-3.07	-0.06	-0.32	0.0
0	2.50	-2.52	-0.27	-0.27	2.0
4	2.55	-2.50	-0.23	-0.28	2.0
7	1.71	-2.23	-0.50	+0.08	5.0
12	1.69	-2.29	-0.48	+0.16	5.0
14	1.67	-2.29	-0.46	+0.18	5.0
17	0.30	-1.67	-0.80	+0.61	10.0
22	0.11	-1.43	-0.70	+0.63	8.6

The circuit was opened for five minutes and the discharges were repeated.

Time (Minutes)	Cell Voltage	Anode Voltage (vs. Ag Reference Electrode*)	Cathode Voltage (vs. Ag Reference Electrode**)	Anode Reference Electrode vs. Cathode Reference Electrode	Current (ma)
27	2.50	-2.72	-0.17	+0.09	2.0
31	2.50	-2.72	-0.15	+0.07	2.0
32	1.65	-2.32	-0.31	+0.37	5.0
37	1.50	-2.16	-0.31	+0.33	5.0
39	0.10	-1.10	-0.47	+0.53	8.4
43	0.10	-1.10	-0.47	+0.53	7.7

---

* Located in the anolyte.
** Located in the catholyte.

TABLE VII. FULL CELL TESTING OF SEPARATOR MATERIALS (Continued)

Epoxy Membrane Separator (Continued)

Time (Minutes)	Cell Voltage	Anode Voltage (vs. Ag Reference Electrode*)	Cathode Voltage (vs. Ag Reference Electrode**)	Anode Reference Electrode vs. Cathode Reference Electrode	Current (ma)
The cell was allowed to discharge at a maximum current level for approximately two hours.					
169	0.09	-0.40	-0.23	+0.09	5.0

Observations: Leakage through the membrane was visually apparent. The original anolyte was clear and colorless. At the end of the discharges it was dark green. A black solid, identified as Cu by X-ray diffraction, was deposited on the anode.

---

* Located in the anolyte.

** Located in the catholyte.

V. DISTRIBUTION LIST

DISTRIBUTION LIST

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