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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, Judith L. Jamrozy, Guy D. McDonald,
Gerald L. Simmons, Jorge E. A. Toni and Warren L. Towle**

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

EIGHTH QUARTERLY REPORT

April 1 - June 30, 1966

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ELECTRONICS

BATTERY

SPECIAL PRODUCTS

EIGHTH QUARTERLY REPORT

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SUMMARY

The electrochemical characteristics of lithium in a number of electrolytes and magnesium in aluminum chloride-acetonitrile were investigated using the voltammetric sweep method. Best performance, as indicated by open circuit potentials and initial slopes of current-potential curves, was obtained in N-nitrosodimethylamine electrolytes. Detailed studies of lithium in lithium hexafluorophosphate^a-N-nitrosodimethylamine (NDA) at various voltage scan rates showed two distinct oxidation processes. The first reaction is not affected by agitation and, therefore, is not affected by mass transport. The second reaction, superimposed on the first at more positive voltages, is affected by agitation and appears to be transport limited. It is postulated that initial oxidation of the lithium is followed by a lithium oxidation process requiring transport of some species to or from the electrode.

Half-cell screening tests were run on lithium anodes in thirteen electrolytes (using the procedure described in the Third Quarterly Report) to determine performance capabilities at current levels between 10ma/cm² and 100ma/cm² and to compare performance in distilled and undistilled solvents. Trends noted were better performance in electrolytes prepared from distilled solvents and the relationship of solute cation size to anode polarization. In the series lithium, sodium, potassium and cesium hexafluorophosphate, least polarization occurred with the smallest cation, lithium. Several of the lithium screening tests were run in electrolytes which appear promising for use with organic cathode systems.

The inorganic cathode materials silver oxide (AgO) and silver oxynitrate (Ag₇O₈NO₃) were investigated in a lithium hexafluorophosphate^a-NDA (vacuum distilled) electrolyte using both the voltammetric sweep method and constant current half-cell screening tests. The two materials were both

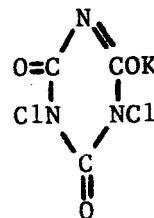
^a The electrolyte was pretreated with lithium to remove acidic impurities and was filtered before use.

reduced at approximately -0.2V (vs. Ag), but the cathodic current peaks were greater for silver oxynitrate. However, in half-cell tests, the silver oxynitrate performance was only slightly better than that of silver oxide. The difference between a commercially obtained silver oxide and silver oxide prepared in our laboratory was also investigated. X-ray patterns showed that the commercial sample was Ag_2O whereas the material we prepared showed only the pattern of AgO . X-ray patterns for silver oxynitrate confirmed its composition. A voltammetric sweep study of cupric fluoride in aluminum chloride-acetonitrile was attempted, but the cathode disintegrated before the scans were completed and the test will have to be repeated.

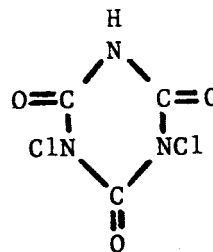
The screening of several organic cathode materials in a variety of electrolytes was initiated this quarter. The screening program included compatibility studies with lithium and magnesium and electrochemical half-cell testing. The organic compounds investigated were:

1. Triazinetrione Derivatives^a

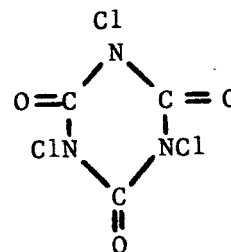
a. ACL-59



b. ACL-70

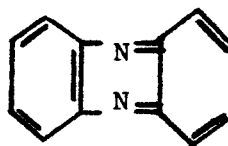


c. ACL-85

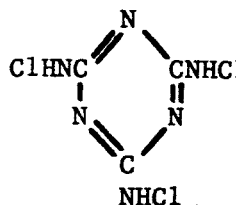


^a Obtained from the Monsanto Chemical Co.

2. Phenazine



3. Trichloromelamine (TCM)



The systems lithium/ACL-59, ACL-70, and trichloromelamine in lithium chloride-aluminum chloride-propylene carbonate^a and ACL-59 in tetramethylammonium hexafluorophosphate-ethylene carbonate (80 weight percent)-propylene carbonate (20 weight percent) appeared to be stable on the basis of visual observations and open circuit voltage stability over a four week period. Half-cell tests were run for these systems using pelletized organic cathodes containing acetylene black and a binder. The most promising combination was an ACL-70 cathode in lithium chloride-aluminum chloride-propylene carbonate.^a Operating cathode voltages vs. a Ag/AgCl reference electrode were +0.9, +0.8, and +0.7V at 0.5, 1.0, and 2.0ma/cm², respectively. Polarization to negative voltages occurred at a current density of 7ma/cm².

Voltammetric sweep studies to determine the nature of the electroactive impurities in lithium hexafluorophosphate (as received from the supplier) were continued. It has been shown previously that of the two reduction waves produced in lithium hexafluorophosphate-N-nitrosodimethylamine, one is caused by proton reduction. The exit gases from the drying of lithium hexafluorophosphate under nitrogen which may contain either a volatile impurity or decomposition product exhibited acidic properties when absorbed in water. Hydrofluoric and hexafluorophosphoric acid were investigated as possible impurities. Thus far, hexafluorophosphoric acid gives performance most closely resembling that of the impurities in lithium hexafluorophosphate.

^a This electrolyte was prepared by saturating the propylene carbonate with lithium chloride and then slowly adding enough aluminum chloride to make a 1 molal solution.

A more precise study of the lithium hexafluorophosphate impurities was made by the voltammetric sweep method using a pyrolytic graphite disc of fixed area as the working electrode and a silver/silver chloride reference electrode with a nonaqueous salt bridge.^a The studies showed that both reduction waves for lithium hexafluorophosphate-N-nitrosodimethylamine are adsorption dependent.^b Fast scan rates decreased adsorption possibilities and only residual current was produced. The scans also showed a marked difference in the amounts of impurities present in various lithium hexafluorophosphate samples. While the actual identification of the electroactive impurities using the voltammetric sweep technique seems beyond the scope of this project, the method may prove useful as a check on solute purity.

In addition to solute purity, chromatographic analysis of the N-nitrosodimethylamine solvent was made. A column packing of 10% Carbowax 20M on Fluoropak 80 was used and the temperature was changed by program from 50° to 120°C. The only impurity peak found was identified as water.

A number of complete battery tests were run this quarter. Preliminary test batteries containing 1.0cm² electrodes were assembled for magnesium/silver oxide, magnesium/cupric fluoride, magnesium/cupric chloride, lithium/silver oxynitrate, and lithium/silver oxide couples in various electrolytes. Results were often erratic, possibly because of electrolyte evaporation in these small cells, but information was obtained about separators, cathode composition and fabrication, and the effect of the presence of impurities in the electrolytes.

Test batteries containing 2" x 2" electrodes and a minimum of dead weight were assembled for the systems lithium/lithium hexafluorophosphate-N-nitrosodimethylamine/silver oxide or cupric fluoride. Best results obtained this month were 108 and 81.4 watt-hours per pound of total battery weight for the silver oxide system discharged at 0.5 and

^a This reference electrode was designed and tested this quarter using a voltammetric method. It was found to give more precise voltage measurements than the silver reference electrodes normally used.

^b The lithium hexafluorophosphate as received from the supplier shows two reduction waves.

5.0ma/cm², respectively, to a cell voltage of 1.5V.

Stability studies for various electrode-electrolyte combinations were continued. Evaluation was based on visual observations, changes in electrolyte conductivity, changes in electrode open circuit potentials, and standloss of assembled test batteries.

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 are being subjected to a more thorough investigation.

Emphasis is now being placed on further characterization of these systems. Concurrently, the compatibility of electrode materials with the electrolytes and losses on standing will be investigated.

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ABSTRACT

Characterization of electrode reactions using the voltammetric sweep method is described for lithium and magnesium anodes and cupric fluoride, silver oxide (AgO), and silver oxynitrate cathodes.

Galvanostatic studies (half-cell screening tests) were made for thirteen anode-electrolyte and three cathode-electrolyte combinations.

Constant current full cell discharges of thirteen preliminary test batteries (1cm^2 electrodes) were completed for magnesium/silver oxide, magnesium/cupric fluoride, magnesium/cupric chloride, lithium/silver oxynitrate, and lithium/silver oxide couples in various electrolytes. Forty-one test batteries ($2'' \times 2''$ electrodes, minimum "dead weight") were assembled for the systems lithium/lithium hexafluorophosphate-N-nitrosodimethylamine/silver oxide or cupric fluoride. The best watt-hours per pound obtained were 108 for a discharge at the 125 hour rate ($0.5\text{ma}/\text{cm}^2$) to a cell voltage of 1.5V.

Screening of the organic cathode materials, ACL-59^a, ACL-70^a, ACL-85^a, trichloromelamine, and phenazine was accomplished in compatibility tests with lithium and magnesium anodes and in half-cell tests.

Voltammetric sweep studies of the impurities in lithium hexafluorophosphate (as received from the supplier) were continued. A qualitative analysis

^a Supplied by the Monsanto Chemical Co.

was made of distilled N-nitrosodimethylamine by means of gas chromatography.

Compatibility studies of lithium, cupric fluoride, and silver oxide in lithium hexafluorophosphate-N-nitrosodimethylamine are described.

I. OVERALL PROGRESS

I. OVERALL PROGRESS

The experimental work done during the eighth quarter encompassed work in the following areas:

- A. Anodes.
 - 1. Voltammetric Sweep Studies.
 - 2. Electrochemical Half-Cell Screening Tests.
- B. Cathodes.
 - 1. Inorganic.
 - 2. Organic.
- C. Electrolytes.
 - 1. Voltammetric Sweep Studies of the Electroactive Impurities in Lithium Hexafluorophosphate.
 - 2. Chromatographic Studies of N-Nitrosodimethylamine.
- D. Complete Battery Tests.
 - 1. Preliminary Battery Tests.
 - 2. Test Batteries.
- E. Chemical Stability of Electrodes in Electrolytes.
- F. Reference Electrode.

A. Anodes.

1. Voltammetric Sweep Studies.

Voltammetric sweep studies have been made on lithium and magnesium anodes in various electrolytes in order to obtain a better insight into the performance of these anodes. Studies were made on lithium in the electrolytes potassium hexafluorophosphate-N-nitrosodimethylamine (NDA), phenyl-trimethylammonium hexafluorophosphate-NDA, morpholinium hexafluorophosphate-NDA, lithium hexafluorophosphate^a-NDA, and lithium hexafluorophosphate^a-propylene carbonate (PC) and on magnesium in the electrolyte

^a The lithium hexafluorophosphate was dried at 110°C under nitrogen.

aluminum trichloride-acetonitrile.

Figure 3-a, page IV-1, contains plots of the voltammetric sweep studies of lithium anodes in the above electrolytes. The geometric areas of the anodes were only approximately the same and therefore the currents obtained can only be qualitatively compared. The highest open circuit potentials (the most negative values relative to a silver reference electrode) were obtained when lithium hexafluorophosphate was used as the solute, e.g., -3.38V and -3.19V for lithium in the electrolytes lithium hexafluorophosphate^a-NDA and lithium hexafluorophosphate^a-PC, respectively.

The potential changes (toward more positive voltages) from open circuit potential before any appreciable current was obtained were greater than 100mv in all electrolytes except lithium hexafluorophosphate^a and morpholinium hexafluorophosphate in NDA. In these latter electrolytes, currents of 50ma/cm² were obtained before more than a 50mv change had occurred.

The greatest initial slope of the current-potential curves was obtained in morpholinium hexafluorophosphate-NDA and the curve with the least slope was obtained in lithium hexafluorophosphate^a-PC (60 and 0.5ma/V, respectively). The scan for lithium hexafluorophosphate^a-PC demonstrates what a drastic effect a change in solvent can have on the slope of the current-voltage curve. The propylene carbonate shows a definite inhibiting effect on the rate of the reaction, thereby severely limiting the current levels at which Li can be operated in this system before polarization becomes extreme.

The voltammetric sweep studies on magnesium anodes relative to a silver reference electrode in aluminum trichloride-acetonitrile (AN) electrolyte are shown in Figure 3-b, Page IV-2. The initial open circuit voltage for magnesium in this electrolyte was -1.70V (vs. Ag). The change in

^a The lithium hexafluorophosphate was dried at 110°C under nitrogen.

potential from this open circuit voltage before a current of 5ma ($30\text{ma}/\text{cm}^2$) was attained was slight (less than 30mv), and the initial slope was approximately 17ma/V compared to the average slope of about 55ma/V for lithium anodes in NDA electrolytes. Magnesium aged in this electrolyte for 24 hours before scanning gave the same open circuit voltage, -1.70V vs. Ag, but slightly lower currents at each potential. The lower currents obtained on the aged electrode may have been caused by the black film which had formed on the magnesium during the ageing process.

A detailed study of the electrochemical behavior of lithium was made in 1m lithium hexafluorophosphate^a-N-nitrosodimethylamine (NDA). In this study, the lithium was held in a cup electrode. Mounting the metal in this manner prevented any gross changes in the area of the lithium electrode during the tests which might affect the results. The cup was made from a small piece of 1/8" teflon tubing (see Figure 1 on page 4) and a glass rod containing a platinum contact sealed inside it. This cup electrode filled with lithium performed very satisfactorily as an electrode and had the advantages over previously used electrodes of ease of preparation and relatively constant geometric area maintained even as the lithium electrode reacted. Figure 3-c, page IV-3 shows the oxidation processes observed with the lithium electrode operated in lithium hexafluorophosphate^a-N-nitrosodimethylamine. Two distinct oxidation processes can be discerned. The first one, with larger slope, appears to be an oxidation which is unaltered by agitation of the electrolyte and thus is not subject to mass transport effects. The second process which is superimposed on the first (after -2.5V) is a transport limited oxidation and is markedly affected by agitation. This oxidation must require the transport of some species to or from the electrode.

^a The electrolyte was pretreated with lithium to remove acidic impurities and was filtered before use.

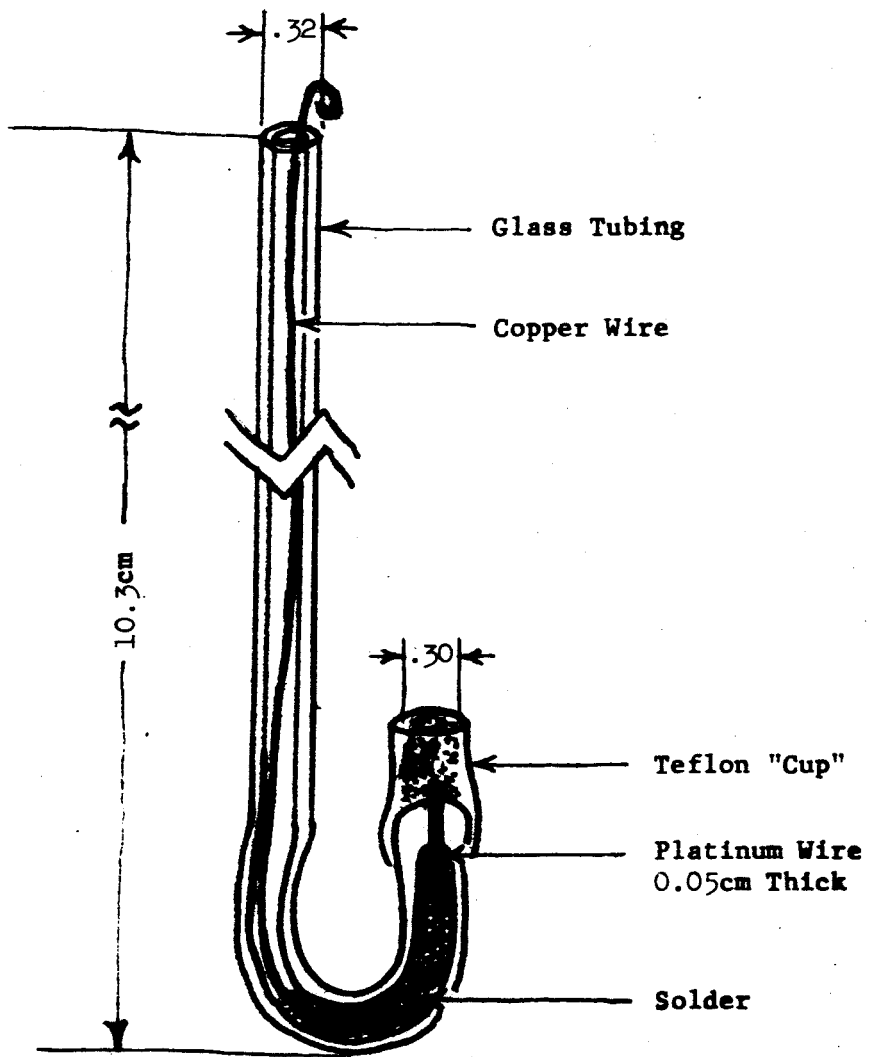


FIGURE 1. THE CUP ELECTRODE

2. Electrochemical Half-Cell Screening of Anodes.

Half-cell screening tests of lithium anodes were run on 13 electrolytes using the procedures described in the Third Quarterly Report. Of these 13 systems, nine were considered for use with inorganic cathodes and four with organic cathodes. The tests on the nine systems for use with inorganic cathodes are similar to previously reported tests except that distilled solvents were used and, in some cases, additional voltage values were obtained at intermediate current densities.

Of the nine systems considered for use with inorganic cathodes, seven demonstrated 100ma/cm² capabilities. Only the systems, 1m tetra-n-propylammonium hexafluorophosphate-dimethylformamide and 1m phenyl-trimethylammonium hexafluorophosphate-NDA, were not capable of maintaining this current density.

A comparison of the data shows that the use of distilled solvents yielded better performance for the lithium anode. For example, lithium in potassium hexafluorophosphate-distilled NDA showed less polarization and more negative voltages (relative to a silver-silver chloride reference electrode) at each current density than did lithium in potassium hexafluorophosphate-undistilled NDA.

In present tests run in distilled solvents, the performance of the lithium anode seemed to be related to the size of the cation in the solute series LiPF₆, NaPF₆, KPF₆, CsPF₆ with the smallest cation, lithium, giving the least polarization of the lithium anode at various current densities.

In addition to the nine half-cells mentioned above, anode half-cell tests were run in saturated solutions of MgCl₂-80 weight percent ethylene carbonate/20 weight percent propylene carbonate^a (ECPC), lithium chloride-methylene chloride^a (MC),

^a Saturated solutions were less than 1 molal.

$(\text{CH}_3)_4\text{NCl-MC}^{\text{a}}$ and 1 molal $\text{LiAlCl}_4^{\text{b}}$ -propylene carbonate (PC). These electrolytes were investigated for possible use with organic cathode systems. Lithium anodes had not previously been screened in these electrolytes and, therefore, their capabilities were determined. The results of the half-cell test are given in Table I, page IV-8. The anode-electrolyte systems of lithium in $\text{MgCl}_2\text{-ECPC}$ and $\text{LiAlCl}_4\text{-PC}$ were the only ones which behaved in a manner that warrants further investigation. They were capable of sustaining at least 10ma/cm^2 before polarizing to a voltage less than 50 percent of the initial open circuit voltage. In view of the stability and performance of organic cathode materials in these electrolytes which are discussed below, these systems are suitable for testing in full cells.

B. Cathodes.

1. Inorganic Cathodes.

A comparison was made of a commercial sample of silver oxide and a silver oxide prepared in our laboratory using the procedure described in Inorganic Synthesis, Volume 4, in order to determine which material would give the best performance. The silver oxides were blended with Solka-Floc (10% by wt.) and electrodes formed by hot-pressing these mixtures (at 50°C and 10,000 lb.) onto 1cm^2 silver expanded metal matrices. Voltammetric sweep studies in lithium hexafluorophosphate^c-NDA gave reduction waves at approximately -0.2V (vs. Ag) for both materials. The peak cathodic current densities at this potential were 9.5ma/cm^2 for the commercial sample and 4.5ma/cm^2 for our material, as shown in Figure 4-a, page IV-9. The results of a potentiostatic

^a Saturated solutions were less than 1 molal.

^b The LiAlCl_4 was prepared by heating 1:1 molal ratio of LiCl and AlCl_3 in an argon atmosphere until fusion occurred.

^c The lithium hexafluorophosphate was used "as received" from the supplier.

test of these two materials where the electrodes were on open circuit and a potential of -0.2V (vs. Ag) was applied and held for two minutes are depicted in Figure 4-b, page IV-10. An initial current density of 14.6ma/cm² was obtained for the commercial material, while our material delivered 9.8ma/cm² initially. However, the commercial material electrode dropped to 2ma/cm² in 15 seconds and had leveled out at 0.90ma/cm² after two minutes while our material was at 3ma/cm² after 15 seconds and was level at 2.1ma/cm² after two minutes. The difference in performance was accounted for to some degree by the X-ray analyses obtained for the two materials. Our material gave a diffraction pattern corresponding to that reported in the literature for AgO whereas the commercial "AgO" gave only the lines for Ag₂O. Since better performance was obtained with the laboratory-prepared material, it will be used in all subsequent tests.

A sample of silver oxynitrate (Ag₇O₃NO₃) was obtained from Mr. H. Leibecki, NASA Lewis Research Center, in order to compare its performance with that of silver oxide (AgO). Voltammetric sweep studies and half-cell screening tests were carried out with both materials in lithium-hexafluorophosphate^a-NDA. The silver oxide electrodes were prepared as described above. The silver oxynitrate electrodes were prepared by cold-pressing the material (at 10,000 lb.) directly onto a 1cm² silver expanded metal matrix. Three-fourths of the 1cm² electrode was removed for the voltammetric studies leaving an electrode with an approximate geometric area of 0.25cm². The voltammetric scans of this electrode in LiPF₆^a-NDA showed a reduction wave at approximately -0.2V (vs. Ag) similar to that found for AgO. A comparison of the voltammetric scans for the two materials is shown in Figure 4-c, page IV-11. A potentiostatic test similar to the one run on the two silver oxides with a two minute hold at -0.2V (vs. Ag) gave an initial current density of 20ma/cm² and,

^a The lithium hexafluorophosphate was used "as received" from the supplier.

after two minutes, a level current density of $11.2\text{ma}/\text{cm}^2$ had been reached. The shape of the curve was the same as that obtained for the two oxides. The X-ray pattern of the silver oxynitrate agreed with both the pattern furnished by Mr. Leibecki and the pattern reported in the literature.

A comparison of the half-cell tests for silver oxide (AgO) and silver oxynitrate (see Table II, page IV-12) shows slightly better performance for the silver oxynitrate at lower current densities; however, at higher current densities, the silver oxide exhibits less polarization. The results for both materials are comparable to our best results for inorganic cathode materials to date.

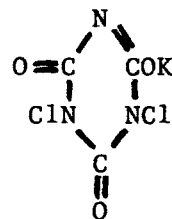
Voltammetric sweep studies were carried out with a cupric fluoride cathode containing 90 wt. percent cupric fluoride, 5 wt. percent acetylene black, and 5 wt. percent Solka-Floc which was prepared by hot-pressing the mix (at 150°C , 10,000 lb.) onto a 1cm^2 copper expanded metal matrix. The electrode disintegrated before the scans were completed, therefore, the test will be repeated with an electrode with greater mechanical integrity.

2. Organic Cathodes.

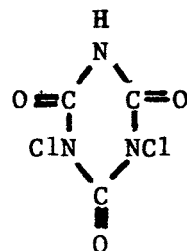
The screening of several organic compounds in a variety of electrolytes to determine their feasibility for use as cathode materials was initiated this quarter. The screening program included compatibility studies with lithium and magnesium, and electrochemical half-cell testing. The organic compounds investigated are listed below:

1. Triazinetrione Derivatives^a

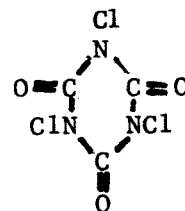
a. ACL-59



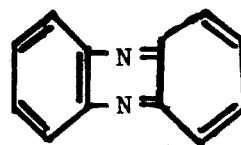
b. ACL-70



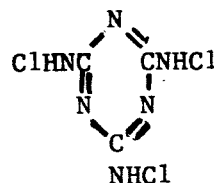
c. ACL-85



2. Phenazine



3. Trichloromelamine (TCM)



The first step in the screening program was the determination of the compatibility of these organic cathode materials with lithium and magnesium anodes. Since many of the organic compounds are soluble in the electrolytes investigated, the compatibility tests were performed by exposing the anode materials to organic material-electrolyte solutions and

^a Obtained from the Monsanto Chemical Company.

periodically measuring the anode and cathode open circuit voltage and making visual observations. In preliminary tests of this type, anode, reference electrode, and inert cathodic current collector were all immersed in the solution for the entire test period. The systems listed below were eliminated because of extreme anode corrosion after a period of one week:

| <u>Anode Material</u> | <u>Organic Depolarizer</u> | <u>Electrolyte (1 Molal)</u> |
|-----------------------|----------------------------|---|
| Mg | ACL-85 (0.1m) | AlCl ₃ -Acetonitrile (AN) |
| Mg | TCM (0.1m) | AlCl ₃ -AN |
| Li | ACL-85 (0.1m) | (C ₄ H ₉) ₄ NCl-80 Wt. % Ethylene Carbonate (EC), 20 Wt. % Propylene Carbonate (PC) |
| Li | TCM (0.1m) | (C ₄ H ₉) ₄ NCl-80 Wt. % EC, 20 Wt. % PC |

In later stability tests, only the anode material remained in solution for the entire test period. The cathodic current collectors (pyrolytic graphite) and reference electrodes (Ag/AgCl) were inserted only for open circuit measurements. This modification in procedure was made to eliminate the possibility of erroneous voltage readings due to filming of the current collector or corrosion of the reference electrode. The results for these tests can be found in Table III, pages IV-14 through IV-21. On the basis of open circuit readings and visual observations, only four systems appear to be stable. These are:

| <u>Anode Material</u> | <u>Organic Depolarizer</u> | <u>Electrolyte (1 Molal)</u> |
|-----------------------|----------------------------|--|
| Li | ACL-59 (1.0m) | LiCl-AlCl ₃ -PC ^a |
| Li | ACL-59 (1.0m) | (CH ₃) ₄ NPF ₆ -80 Wt. % EC, 20 Wt. % PC |
| Li | ACL-70 (1.0m) | LiCl-AlCl ₃ -PC ^a |
| Li | TCM (0.2m) | LiCl-AlCl ₃ -PC ^a |

^a This electrolyte was prepared by saturating the propylene carbonate with lithium chloride and then slowly adding enough aluminum chloride to make a 1 molal solution.

It is interesting to note that in each of these four systems, sufficient organic depolarizer was added to make up the given concentration but the solutions apparently would have been saturated at lower concentrations. Thus, it might be possible to study the organic materials as solid cathodes in these electrolytes.

Half-cell tests were run on the four stable systems using pelletized cathodes prepared from slurries of the organic depolarizer, acetylene black, and filter paper pulp. Silver matrices were used to hold the cathode mix. The most promising combination was an ACL-70 cathode in one molal $\text{LiCl-AlCl}_3\text{-PC}^a$. Operating voltages for this cathode system were +0.9, +0.8, and +0.7V (vs. Ag/AgCl) at 0.5, 1.0 and 2.0ma/cm², respectively. The ACL-70 cathode polarized to negative values at a current density of 7ma/cm². A half-cell test was also run for an ACL-59 cathode in one molal $\text{LiAlCl}_4^b\text{-PC}$ for comparison with the $\text{LiCl-AlCl}_3\text{-PC}^a$ electrolyte. The performance of this electrode in $\text{LiAlCl}_4^b\text{-PC}$ was not as good as in $\text{LiCl-AlCl}_3\text{-PC}^a$. The results of the half-cell tests are given in Tables IV-a and IV-b on pages IV-22 through IV-26. These tables also include several dissolved organic depolarizer systems which later proved to be unstable.

C. Electrolyte.

1. Voltammetric Sweep Studies of the Electroactive Impurities in Lithium Hexafluorophosphate.

Electrochemical studies on lithium hexafluorophosphate have been continued during the past quarter to determine the nature of the electroactive impurities present in this material, when

^a This electrolyte was prepared by saturating the propylene carbonate with lithium chloride and then slowly adding enough aluminum chloride to make a 1 molal solution.

^b The LiAlCl_4 was prepared by heating a 1:1 mole ratio of LiCl and AlCl_3 in an argon atmosphere until fusion occurred.

received, which contribute to the electrochemical performance of the cathodes discharged in electrolytes containing this solute.

Prior to this month, it was shown that of the two reduction waves found in the voltammetric sweeps of the electrolyte composed of lithium hexafluorophosphate (as received)-N-nitrosodimethylamine, one wave was due to proton reduction. This wave was enhanced by the addition of water and decreased by the addition of drying agents such as lithium metal or phosphorous pentoxide.

The exit gasses from the drying of lithium hexafluorophosphate under nitrogen which may contain either a volatile impurity or decomposition product exhibited acidic properties when absorbed in water. A titration of the aqueous solution of the exit gasses obtained when 55.5g of lithium hexafluorophosphate was dried required 18.8 milliequivalents of base. Sodium bifluoride, NaHF_2 , and hexafluorophosphoric acid (65% aqueous solution) were investigated in hopes of providing further information about the nature of the electroactive impurities in lithium hexafluorophosphate.

Sodium bifluoride had only a limited solubility in N-nitrosodimethylamine (NDA), less than 0.3m. A voltammetric sweep study of a saturated solution of sodium bifluoride in NDA is shown in Figure 5-a, page IV-27. The low currents obtained are probably due to the low conductivity of the electrolyte. Figure 5-b on page IV-28, shows the effect of saturating a solution of "as received" lithium hexafluorophosphate-NDA with sodium bifluoride. The initial wave, the reduction of interest, showed no enhancement by this addition and it is therefore doubtful that the bifluoride ion or any of its possible dissociation products, e.g., HF, are the electroactive impurities in lithium hexafluorophosphate.

Voltammetric sweep studies of 0.1m and 1.0m hexafluorophosphoric acid in NDA are shown in Figure 5-c, page IV-29. The peak positions in these solutions are not dissimilar from those found in "as received" lithium hexafluorophosphate-NDA. The addition of hexafluorophosphoric acid to dried^a lithium hexafluorophosphate-NDA (see Figure 5-d, page IV-30) enhances the initial peak considerably. Similarly, the addition of hexafluorophosphoric acid to "as received" lithium hexafluorophosphate-NDA (as shown in Figure 5-e, page IV-31) gave enhancement of the initial peak. Of the materials tested to date, hexafluorophosphoric acid gives performance most closely resembling that shown by the electroactive impurities found in lithium hexafluorophosphate as received from the supplier.

Attempts were made to duplicate the work on lithium hexafluorophosphate and hexafluorophosphoric acid in propylene carbonate (PC) as solvent in place of NDA. The electrochemical tests were hampered by severe filming of the electrodes which led to irreproducible results. After each scan, the working electrode was covered with a heavy blue-gray film. Repeated scans gave different peak heights, peak positions and sometimes no peaks at all. These results are quite different from those obtained in NDA. Because of the problems encountered in propylene carbonate, the studies of lithium hexafluorophosphate and hexafluorophosphoric acid in this solvent were discontinued.

A more detailed study of the electroactive impurities in lithium hexafluorophosphate in NDA was made using a pyrolytic graphite disk with a geometric area of 7.8mm^2 as a working electrode, silver-silver chloride reference electrode with a nonaqueous salt bridge (see Section F), and a pyrolytic graphite rod counter electrode. The solutions to be investigated were purged with nitrogen prior to investigation to eliminate any oxygen

^a The lithium hexafluorophosphate was dried at 110°C under nitrogen.

contamination. The solutions were kept under a nitrogen blanket during the tests. This precaution was taken because preliminary studies showed that while oxygen did not have a pronounced influence on the reduction waves found in lithium hexafluorophosphate solutions, a small wave was present prior to the reduction peaks of lithium hexafluorophosphate when samples were exposed to oxygen.

Two solutions were investigated, lithium hexafluorophosphate as received from the supplier dissolved in N-nitrosodimethylamine (NDA) and a similar solution neutralized with lithium metal. The voltammetric sweep studies of the neutralized solution showed only one reduction peak which apparently corresponds to the second peak found in the unneutralized solution (see Figure 5-f, page IV-32). Since adsorption is time, potential and concentration dependent, the linearly varying potential techniques allowed the study of the two solutions in more detail by varying the first two parameters over a wide range. In Figure 5-g, page IV-33, curve 1 shows the cyclic voltammetric scan obtained at a scan rate of two volts per second on an electrode which had only been held at potentials more negative than -1.5V relative to the Ag/AgCl reference electrode. Under these conditions, only residual current is obtained. When this same electrode was held at -0.76V for 5 to 10 seconds, curve 2 is typical of the results obtained when the electrode was scanned towards more negative potentials. The peak height was found to be related to the holding time and to the potential applied (for potentials more positive than -1.5V). The peak height is limited to a maximum value which is determined by the equilibrium for adsorption. During the holds, the current was very close to zero and was cathodic.

In the unneutralized solution, both reduction processes found were shown to be adsorption dependent. The voltammetric studies of this solution are shown in Figure 5-h, page IV-34. If the

adsorption of the impurities on the working electrode was prevented through the use of very fast scan rates, only residual current was obtained between -1.5V and -2.5V vs. Ag/AgCl (curve 1). Holds for 500 and 800 seconds at -1.5V gave appreciable adsorption of one species and both resulted in scans represented by curve 2. A rapid scan from -0.5V to -2.5V gave curve 3 which shows only residual current between -0.5V and -1.5V and some reduction of the species adsorbed at voltages more positive than -1.5V. Again, a 500 second hold at -0.5V resulted in curve 4 where both species have been adsorbed and their reduction waves appear.

When the adsorbed species are reduced, it is possible to reoxidize them at more positive potentials. Figure 5-i on page IV-35 shows these anodic and cathodic scans. The anodic peak at +0.8V (vs. Ag/AgCl) is only obtained if the first impurity is reduced (cycle 1) and the peak at +0.3V appears when the second species is reduced (cycle 2). The actual identification of the impurities responsible for these peaks appears to be a problem not within the scope of this project. The first reduction peak seems to be related to the acidic impurity, the concentration of which varies markedly from one lot of lithium hexafluorophosphate to another. Neutralization of this acidic impurity with lithium removes the material giving rise to the wave at -1.1V vs. Ag/AgCl (see Figure 5-f, page IV-32) and extends the region where only residual current is apparent to cover the range from 0 to -1.5V (vs. Ag/AgCl). Thus, linear sweep voltammetry or cyclic voltammetry can be used to provide a rapid and simple check of the composition of lithium hexafluorophosphate solutions as each new lot of solute is received and used.

2. Chromatographic Studies of N-Nitrosodimethylamine.

In addition to solute purity determinations, chromatographic analysis of the N-nitrosodimethylamine solvent was made. A

column packing of 10% Carbowax 20M on Fluoropak 80 was used and the temperature was changed by program from 50° to 120°C at 40° per minute. The chromatogram of distilled N-nitrosodimethylamine (NDA) is shown in Figure 6, page IV-36. The attenuations used are shown next to the appropriate peaks. Only three peaks appear in the chromatogram. The first peak, which occurs immediately after injection, is an air peak. The second peak was shown to be water and the third peak is N-nitrosodimethylamine. To confirm the identification of the second peak, a synthetic mixture of 99.9 percent distilled NDA with 0.1 percent water was made. The chromatogram of this mixture showed no new peaks and the second peak in the chromatogram is approximately twice as large as it was in the first chromatogram. Further studies of N-nitrosodimethylamine are planned to quantitatively determine the water content of this distilled solvent.

D. Complete Battery Tests.

1. Preliminary Test Batteries.

Several small test cells containing 1cm² electrodes were assembled to test separator types and thickness, ratio of active material weight to substrate weight, and general fabrication technique. The performance of these test batteries was, in general, quite erratic with frequent discontinuities in the data caused by short circuits, loss of electrolyte or changes in electrodes or clamping pressures. From these small test cells, it was found that an active material weight to matrix weight of 10:1 could be used without any appreciable increase in resistance in the electrode. These small test cells also showed that Gelman's glass fiber separators gave better performance than the previously tested microporous rubber separators. The glass separators also had the advantages of less weight and greater electrolyte absorbing ability. The information gained through these small test cells was used in the construction of batteries containing 2" x 2" electrodes.

Additional small test cells containing 1cm^2 electrodes were assembled to test the effect on performance of various compositions of cathode mixtures in various battery systems. The variables investigated were the effect of the addition of varying amounts of binder and conductive additives, the use of different anode materials and the use of various electrolytes.

As a rule, test electrodes containing acetylene black (Shawinigan) as a conductive additive gave slightly better performance. Cells containing the greater amount of acetylene black usually gave better high voltage performance. This is what would be expected if silver oxide was fairly resistive.

The performance of silver oxide electrodes (our material pressed at $2,000\text{ lb/cm}^2$ at 50°C onto a 1cm^2 silver expanded metal matrix) in 1m AlCl_3 -acetonitrile (AN) and 1m AlCl_3 , 0.1m LiPF_6 -AN were compared. It was postulated that the cathode performance might be improved by the addition of lithium hexafluorophosphate because of the good performance of silver oxide electrodes in other electrolytes containing lithium hexafluorophosphate. A brief summary of the test results can be found in Table V, pages IV-37 to IV-40. It is interesting to note that the addition of acetonitrile saturated with lithium hexafluorophosphate (0.1 molal) improved the magnesium voltages somewhat, but the silver oxide polarized more severely than in the absence of lithium hexafluorophosphate. The addition of additional acetonitrile saturated with lithium hexafluorophosphate to the $\text{Mg}/1\text{ molal AlCl}_3$, 0.1 molal LiPF_6 -AN/AgO system after the 75 minute test depolarized the cathode and its voltage returned to approximately -0.5V . Evidently some constituent of this solution was capable of reducing the cathode polarization. The anode voltage also improved upon addition of the LiPF_6 -AN solution, returning to -1.79V .

The performance of magnesium anodes was investigated in 1m $(n\text{C}_3\text{H}_7)_4\text{NPF}_4$ -acetonitrile (AN), 0.5m $(n\text{C}_3\text{H}_7)_4\text{NBF}_4$, 0.5m AlCl_3 -AN, 1m AlCl_3 -AN, 1m $\text{LiAlCl}_4^{\text{a}}$ -AN, 1m morpholinium hexafluorophosphate-AN, 1m $(n\text{C}_3\text{H}_7)_4\text{NPF}_6$ -AN and 0.5m $(n\text{C}_3\text{H}_7)_4\text{NPF}_6$, 0.5m AlCl_3 -AN. These electrolytes were chosen for the small test cells because previous half-cell tests showed that magnesium was capable of delivering at least 10ma/cm² in these electrolytes. In all these tests, silver oxide cathodes were used. The best magnesium performance was obtained in 0.5m $(n\text{C}_3\text{H}_7)_4\text{NPF}_6$, 0.5m AlCl_3 -AN which gave an open circuit value of -1.9V (vs. Ag/AgCl) and a potential of -1.9V (vs. Ag/AgCl) for 12 minutes under a load of 5ma/cm² (see Table V, page IV-38). The performance of the silver oxide cathodes in these electrolytes was relatively poor.

Small test cells were also run to compare the performance of silver oxide in 1m AlCl_3 -AN and 1m $\text{LiAlCl}_4^{\text{a}}$ -AN. The cathode performance was better in the AlCl_3 -AN electrolyte (see Table V, page IV-38). Cupric fluoride was also tested in the 1m $\text{LiAlCl}_4^{\text{a}}$ -AN electrolyte. Its performance was poorer than that of the silver oxide.

One test cell using a 1cm² silver oxynitrate cathode in excess 1m LiPF_6^{b} -NDA was run. The cathode had an open circuit voltage of +0.28V (vs. Ag/AgCl) and ran for five hours at 10ma/cm² (200 hour rate) before polarizing to -1.3V vs. Ag/AgCl (1.5V for the full cell). This is comparable to the coulombic results obtained with AgO. The better performance expected on the basis of voltammetric sweep and half-cell tests cited earlier was not realized.

^a The LiAlCl_4 was prepared by heating a 1:1 mole ratio of LiCl and AlCl_3 in an argon atmosphere until fusion occurred.

^b The LiPF_6 was used "as received" from the supplier.

2. Test Batteries.

The investigation of test batteries containing 2" x 2" electrodes and a minimal amount of dead weight was initiated. Silver oxide and copper fluoride cathodes were used with lithium anodes, Gelman glass fiber separators and a one molal lithium hexafluorophosphate^a-N-nitrosodimethylamine (NDA) electrolyte. The variables of cathode composition and cell configuration were examined. The results of these tests are given in Table VI, pages IV-41 through IV-46.

Several variables which markedly effected the overall performance of these cells have been isolated. A correlation has been found between the method employed in mixing the active cathode material and the performance of the cathode. Free tumbling of the ingredients or blending them under hexane produced better performance for the silver oxide electrodes than when the materials were ball-milled. One possible explanation for this may be that the fibers of the Solka-Floc binder are broken down by ball-milling and the electrodes containing these broken fibers do not achieve adequate electrolyte transfer from the solution to the interior of the electrode.

When silver oxide produced in this laboratory was used as the active cathode material, it gave better performance in full battery tests than did the commercial material. As previously noted, the commercial sample of "AgO" was actually Ag₂O and the difference in performance could be attributed to the difference between AgO and Ag₂O.

The examination of discharged cells has shown that non-uniform surface of electrodes results in uneven consumption of the electrode materials. The high points of the electrodes come in contact with the electrolyte-laden separator and are consumed first. The lower spots on the electrode faces are utilized only with difficulty. Changes in pressing the electrodes and the use of more even clamping should remedy this problem.

^a The lithium hexafluorophosphate was used either "as received" from the supplier or after a pretreatment as specified in Table VI, pages IV-41 to IV-46.

Even with the problems mentioned above, the coulombic efficiencies of active materials were as high as 70 percent at a 0.5ma/cm² discharge rate and as high as 60 percent at 5ma/cm². These efficiencies are reproducible and, although efficiencies of over 90 percent are desirable, the slight decrease with a tenfold increase in current density is encouraging.

In certain tests, where a new batch of LiPF₆ was used to prepare the electrolyte, a vigorous reaction with the electrodes was noted. Gas evolution occurred and in some cases enough heat was evolved to melt the plastic bag containing the battery. Shelf life tests using the same electrolyte gave very poor results due to the severe attack of the electrodes by this electrolyte. A titration of a sample of the new batch of LiPF₆ showed it contained a significantly higher level of the acidic impurity than previous batches of this material. It was therefore deemed necessary to improve and regulate the purity of the lithium hexafluorophosphate either by purifying commercial supplies or by preparing the compound ourselves.

Purification of the lithium hexafluorophosphate could be accomplished most readily by recrystallization from an appropriate solvent. Plans have been made to try this approach with the supplies on hand.

A literature search has revealed the following methods for preparing lithium hexafluorophosphate. These are as follows:

- (1) $PCl_5 + 6 LiF = LiPF_6 + 5 LiCl$
- (2) $LiF + 5 HF + PCl_5 = LiPF_6 + 5 HCl$
- (3) $PF_5 + LiF \xrightarrow[Steel Bomb]{HF Anhyd., 12 Hr. 250^\circ C} LiPF_6$
- (4) $3 P_2O_5 + 10 BF_3 + 6 LiF = 6 LiPF_6 + 5 B_2O_3$

Of these methods, only (1) does not involve the use of relatively hazardous materials or techniques. However, method (1) produces the lowest purity LiPF_6 since it is very difficult to separate the LiCl from the product.

An alternative method for preparing LiPF_6 would involve the metathetical reaction of a lithium salt and a hexafluorophosphate compound in a suitable solvent to form an insoluble product and LiPF_6 . This method for preparing LiPF_6 is currently being investigated.

In spite of the problems encountered using this latest batch of LiPF_6 , some results of interest were noted. A battery discharged at the 60 hour rate ($1\text{ma}/\text{cm}^2$) to 2.0V gave 72.2 watt-hours per pound of total battery weight as compared to 100 watt-hours per pound for a similar battery discharged at the 120 hour rate ($0.5\text{ma}/\text{cm}^2$). A battery discharged at the 6 hour rate ($10\text{ma}/\text{cm}^2$) to 1.5V yielded 41.8 watt-hours per pound of total battery weight. The electrolyte used in this last test was pretreated with lithium to remove any acidic impurities. No attack of the electrodes was observed. A similar battery with three weight percent of acetylene black added to the cathode mix delivered 81.4 watt-hours per pound of total battery weight when discharged at the 12.5 hour rate ($5\text{ma}/\text{cm}^2$) to 1.5V.

E. Chemical Stability of Electrodes in Electrolytes.

Stability tests started during the previous quarter (see the Seventh Quarterly Report) on the stability of lithium, copper fluoride, and silver oxide in $\text{LiPF}_6\text{-NDA}$ were terminated during this quarter. The specific conductance of the electrolyte remained relatively constant over the entire test period indicating that no extensive interaction between the electrode materials and the electrolyte to give conductive species had occurred. On prolonged standing, the cathode pellets in many cases lost much of their mechanical integrity. The data for the stability tests are presented in Table VII, pages IV-47 through

IV-59. Half-cell tests were run on the lithium anodes after the stability tests were completed. The results of these tests are given in Table VIII, page IV-60. The lithium aged in the electrolyte prepared from LiPF_6 dried at 110°C under nitrogen gave better performance than the lithium aged in electrolyte prepared from LiPF_6 used as received from the supplier. It is interesting to note that CuF_2 in LiPF_6 -NDA underwent a 50% weight loss with "as received" solute whereas there was very little change with the dried solute (see pages IV-49 and IV-50).

A second series of tests was initiated this quarter in which electrode stability was evaluated by periodically measuring open circuit voltages and making visual observations. The open circuit readings were used to augment the visual observation. If a marked change in an electrode's open circuit potential occurred it was assumed that either the electrode had been attacked by the electrolyte or that it had been filmed. Copper fluoride and silver oxide electrodes in one molal aluminum trichloride-acetonitrile and lithium and silver oxide electrodes in one molal lithium hexafluorophosphate-N-nitrosodimethylamine (NDA) were tested in this manner. Plots of the open circuit voltages versus time are depicted in Figures 7-a through 7-c, pages IV-61 through IV-63. Cathode open circuit voltages were relatively stable in both electrolytes over a period of at least two weeks. Open circuit values for lithium in LiPF_6 -NDA prepared from "as received" and vacuum-dried LiPF_6 dropped approximately 2.0V in less than one day. Drying the LiPF_6 solute at 110°C in a nitrogen atmosphere decreased the reactivity of the lithium with the electrolyte and eliminated this rapid initial voltage drop.

Lithium and silver oxide electrodes were also exposed to one molal LiPF_6 (dried under nitrogen at 110°C)-NDA which contained hexafluorophosphoric acid,^a (approximately 0.4 weight percent HPF_6), a possible impurity in "as received" lithium hexafluorophosphate.

^a The hexafluorophosphoric acid added to the electrolyte was a 65 percent aqueous solution.

The HPF_6 addition had little effect on the silver oxide and lithium open circuit voltages over a two week period (see Figure 7-c, page IV-63). Apparently, any Li reaction with this small an amount of material is not detrimental to the Li surface.

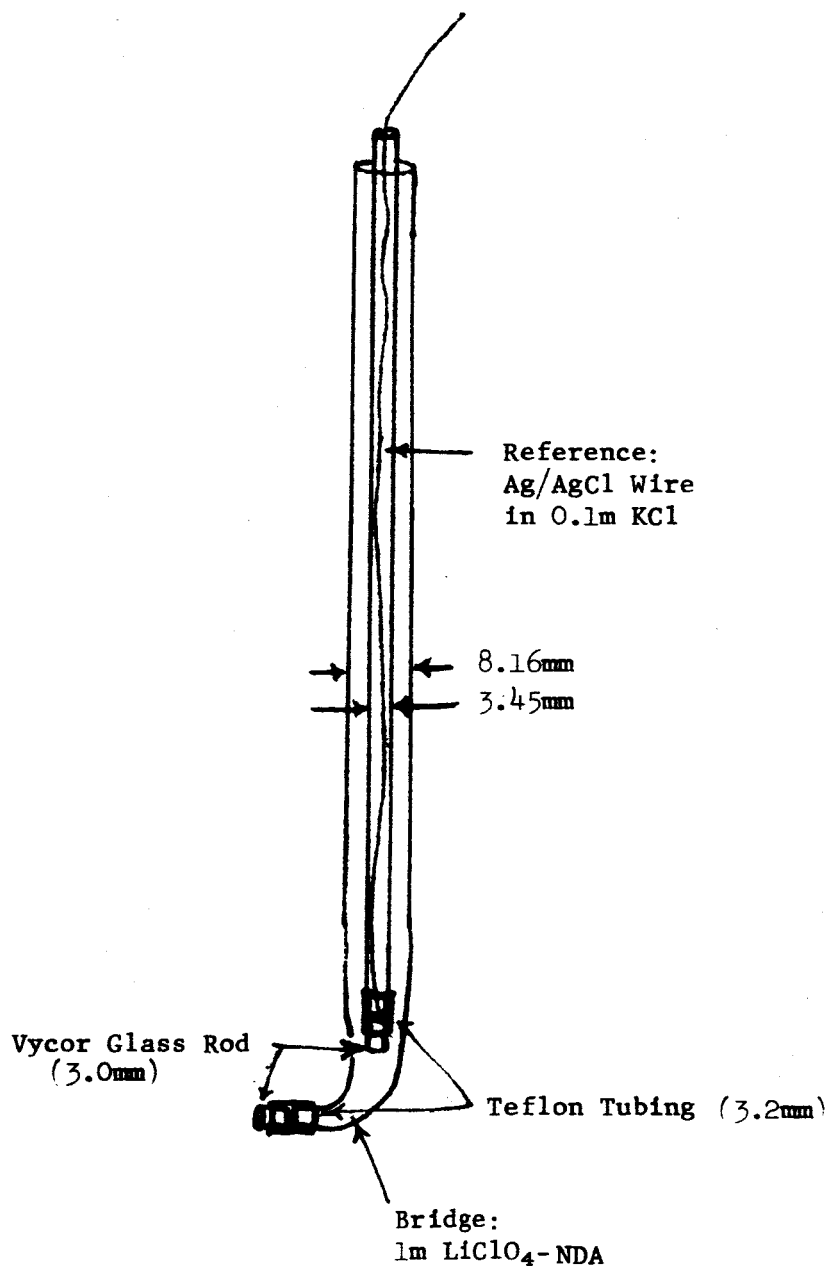
A single preliminary shelf-life study was run in which a completely assembled 2" x 2" cell was allowed to stand for five days before discharge. A total of 77.8 watt-hours per pound was obtained at cell voltages greater than 1.5V. This is 72% of the 108 watt-hours per pound obtained from a fresh cell of similar construction and composition discharged at the same rate to the same voltage. Shelf-life studies were also run on completely assembled 2" x 2" test cells containing LiPF_6 -NDA electrolyte which was prepared with the batch of LiPF_6 which contained a large amount of an acidic impurity (approximately 20%). The watt-hours per pound of total battery weight obtained above 1.5V was 75.4, 32.8, 10.6 and 9.9 for zero, one, two and three weeks of activated standing.

F. Reference Electrode Study.

Silver electrodes are widely used as reference electrodes in non-aqueous solutions. The stability of the potential of these electrodes is in doubt. An alternative electrode was constructed which had the simplicity and versatility of the silver electrode, but which demonstrated considerably greater potential stability than the silver electrode. This electrode will be used in place of silver whenever precise potential measurements are required.

Figure 2, page 24, is a schematic drawing of this alternative reference electrode. It consists of a conventional silver-silver chloride electrode in an aqueous 0.1M KCl solution separated from the working electrolyte by a nonaqueous salt bridge (1M LiClO_4 -NDA). A small piece of porous glass rod (Vycor No. 7930) is used to isolate the KCl solution from the salt bridge and the salt bridge from the solution of interest. The lower end of the salt bridge is curved

FIGURE 2. SILVER-SILVER CHLORIDE REFERENCE ELECTRODE



to permit positioning of the reference electrode very close to the working electrode. The salt bridge is designed for easy cleaning after it has been contaminated by ions from the working electrolyte and the reference electrode.

The reversibility of this electrode was investigated using the classical method of applying small overpotentials to both sides of the equilibrium values and determining if there is any hysteresis. For this experiment, the cell consisted of the electrode shown in Figure 2 as working electrode, a saturated Calomel Electrode (Beckmann 39170) as reference, a pyrolytic graphite rod^a as counter electrode and 1M LiPF₆-N-nitrosodimethylamine^b as electrolyte. The overpotentials were applied using potentiostatic equipment and were measured using a digital voltmeter through the use of a load resistor. The overpotentials were applied manually and the measurements were made within 10-20 seconds (after a steady value was achieved). The results are shown in Figure 8, page IV-64.

On the first run, a maximum hysteresis of only 0.6mv was observed. All subsequent runs duplicated this hysteresis curve. The potential, as determined from Figure 8, of the Ag/AgCl (0.1m KCL), 1m LiClO₄-NDA reference electrode is $-0.0249 \pm 0.0003V$ relative to the saturated Calomel Electrode (SCE). For our experiments, a value of -0.025V versus SCE will be used.

The same reversibility test was attempted with a silver electrode. No steady potential value could be obtained so that it was impossible to determine its reversibility or its potential.

^a The pyrolytic graphite was Speer Carbon No. 580.

^b The electrolyte was neutralized with excess Li before use. Distilled NDA was also used.

II. CURRENT PROBLEMS

II. CURRENT PROBLEMS

The major problem continued to be that of characterizing electrode materials and electrolytes to be used in complete battery systems. Purity of electrolyte solutes also remains a serious problem which we are attempting to overcome.

III. WORK TO BE PERFORMED

III. WORK TO BE PERFORMED

A. During the Next Quarter.

Studies of the lithium-silver oxide system will be continued with particular emphasis on the performance of this couple in lithium hexafluorophosphate-N-nitrosodimethylamine electrolyte. The performance of this couple will also be investigated on a limited scale in dimethylsulfoxide and propylene carbonate.

The investigation of the performance of silver oxynitrate will be continued. The long range compatibility of electrodes in electrolytes will be further investigated.

B. During the Next Month.

Emphasis will be placed on obtaining better current performance from the Li/LiPF₆-NDA/AgO system. The long range compatibility tests on this system will be completed during this month.

Half-cell tests will be run on silver oxide and lithium in LiClO₄-dimethylsulfoxide (DMSO), phenyltrimethylammonium hexafluorophosphate-DMSO and LiPF₆-DMSO.

The search for a satisfactory separator for use with soluble cathodes will be continued.

Cyclic voltammetric sweep studies will be run on lithium and AgO in LiAlCl₄-PC and LiPF₆-NDA.

IV. TEST RESULTS^a

^a Unless otherwise indicated, vacuum distilled solvents were used in all tests.

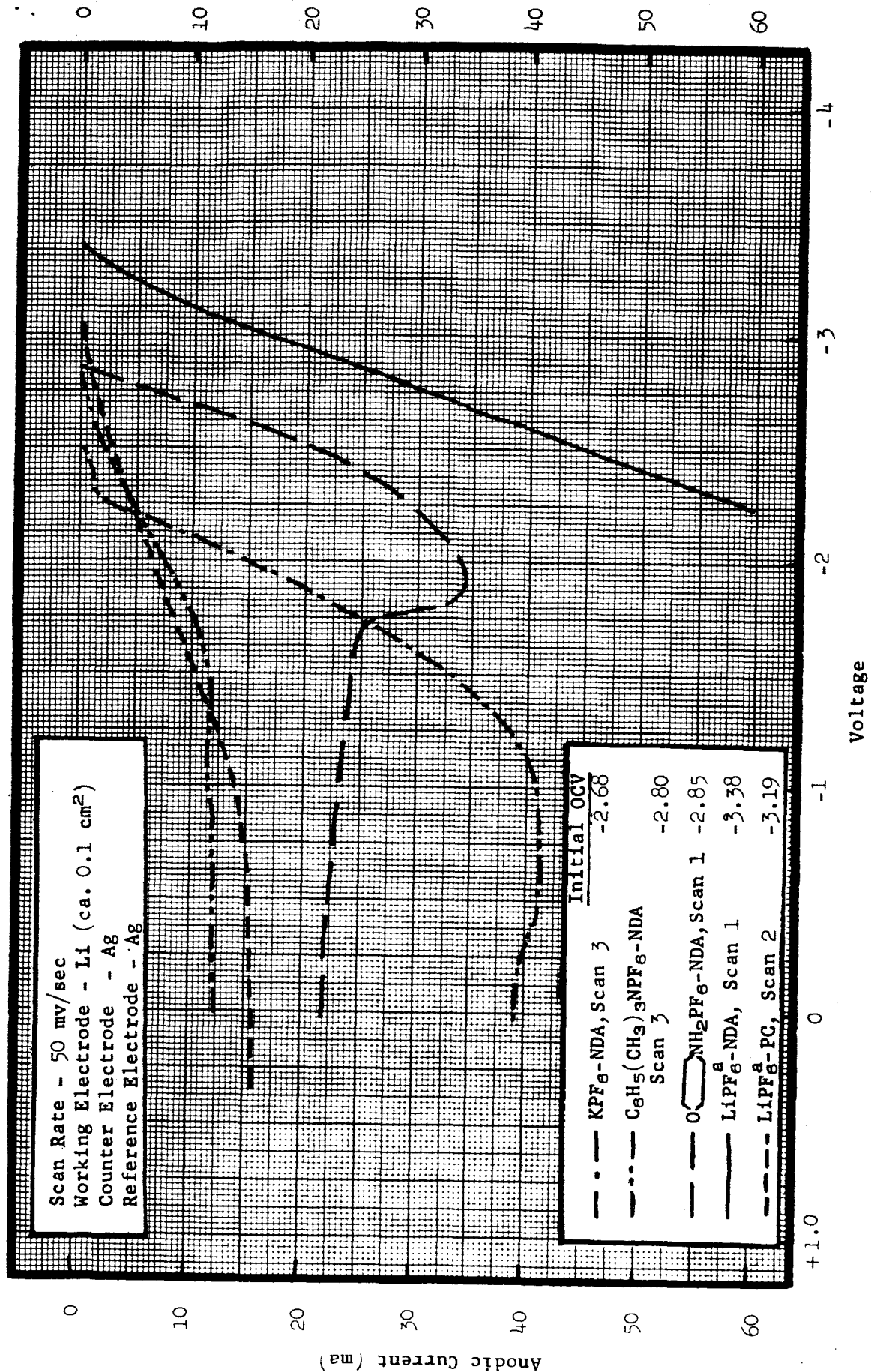


FIGURE 3. VOLTAMMETRIC SWEEP STUDIES OF ANODES

Figure 3-a. Voltammetric Sweep Studies of Lithium in Various Electrolytes.

The initial potential for each electrode was set equal to the open circuit voltage and the scan taken towards more positive voltages.

^a LiPF₆ used was dried at 110°C under nitrogen.

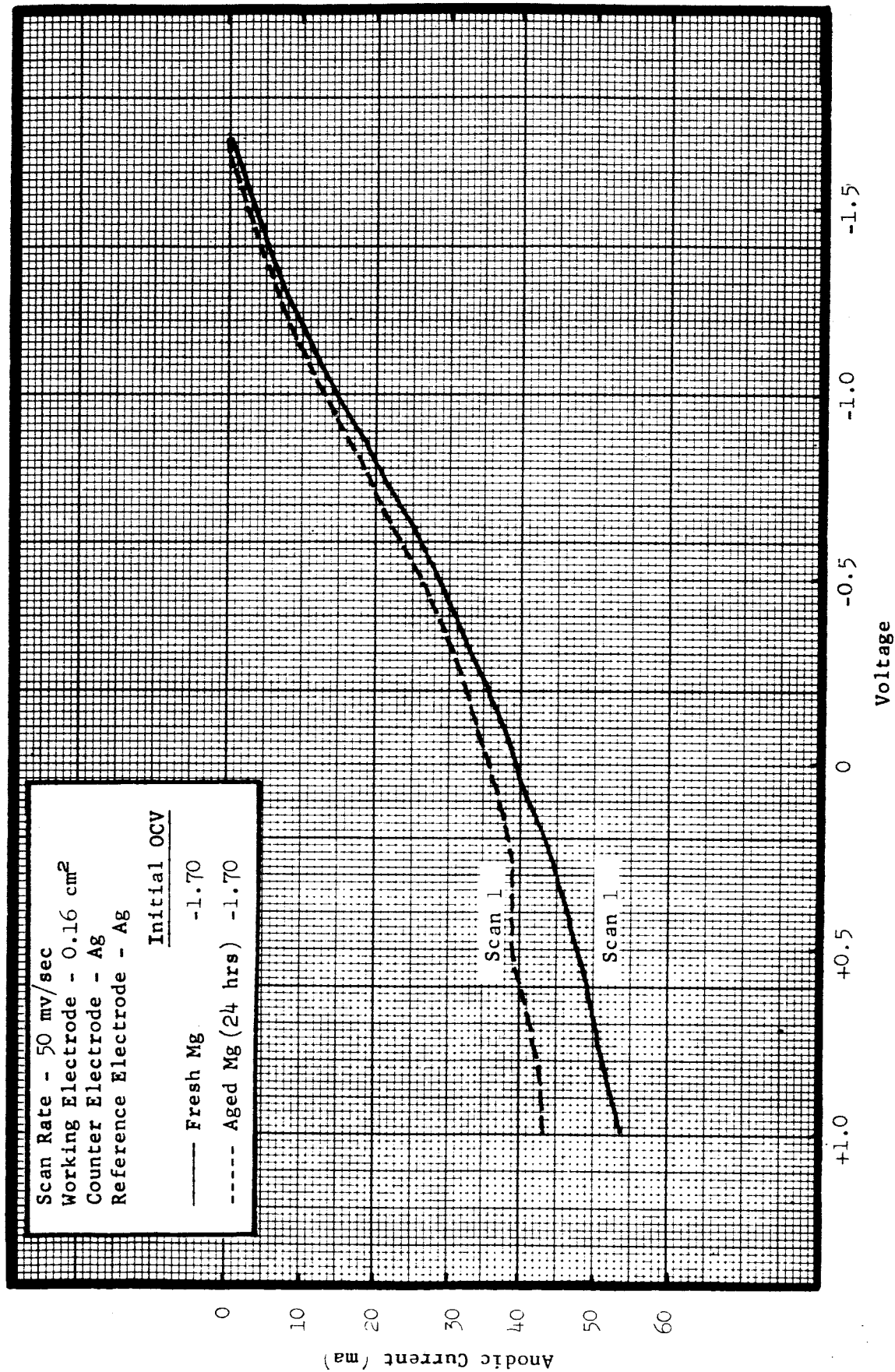


FIGURE 3. VOLTAMMETRIC SWEEP STUDIES OF ANODES (Continued)

Figure 3-b. Voltammetric Sweep Studies of Magnesium in 1m Aluminum Trichloride-Acetonitrile. The initial potential for each electrode was set equal to the open circuit voltage and the scan taken towards more positive voltages.

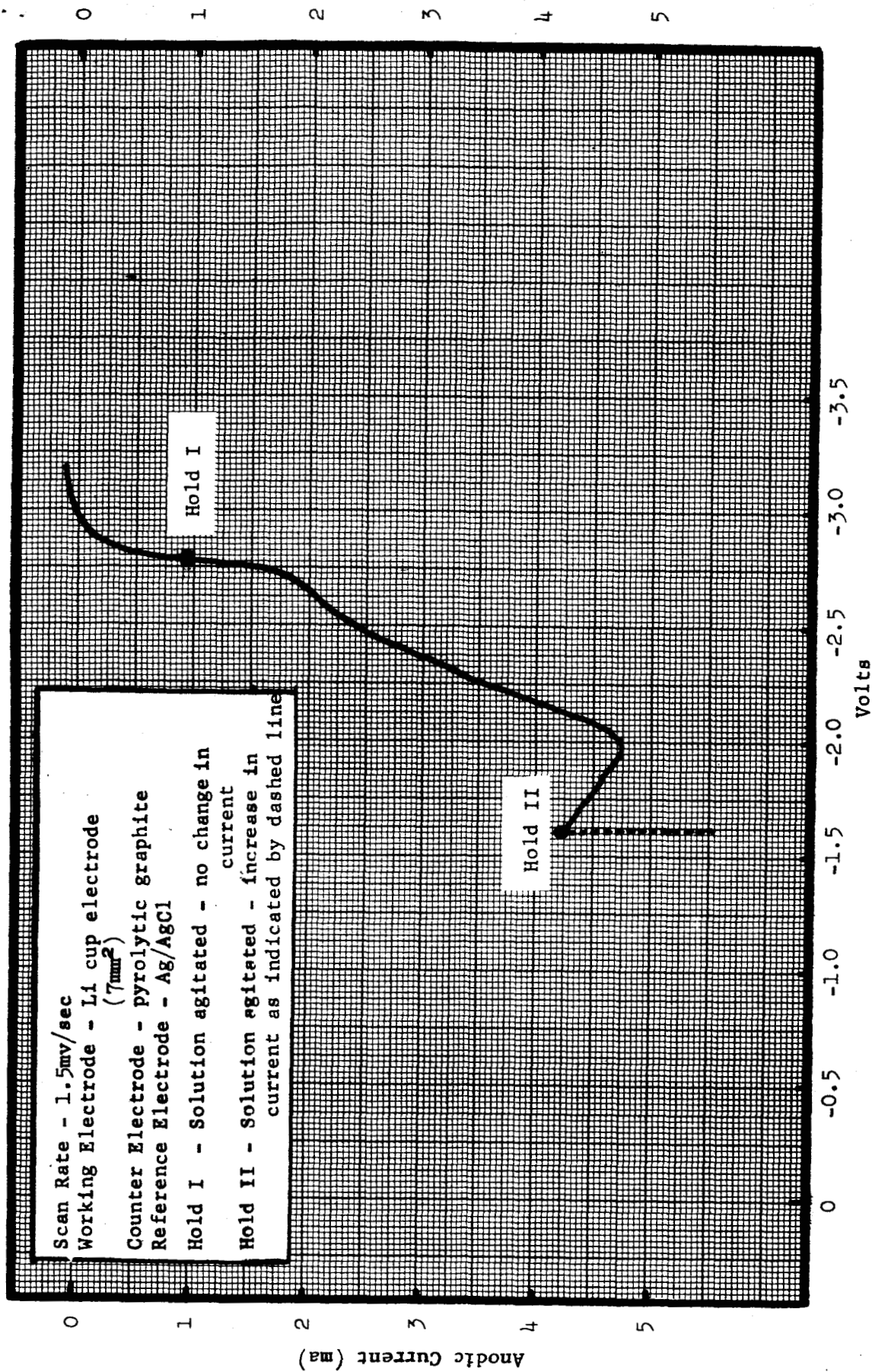


FIGURE 3. VOLTAMMETRIC SWEEP STUDIES OF ANODES (Continued)

Figure 3-c. Electrochemical Study of Lithium in Lithium Hexafluorophosphate-N-Nitrosodimethylamine^a. The initial potential for the electrode was set at -3.2V vs. Ag/AgCl and the scan run towards more positive voltages. At the Hold I point, the potential was held constant for approximately 30 seconds, then the scan was continued to Hold II when the potential was again held constant for 30 seconds.

^a The LiPF₆-N-nitrosodimethylamine solution was pretreated with lithium prior to this study.

TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES

A. Lithium Anode Systems.

| <u>Solute</u> | <u>Solvent</u> | <u>Page</u> |
|--------------------------|---|-------------|
| 1. $(n-C_3H_7)_4NPF_6$ | dimethylformamide (DMF) | IV-5 |
| 2. $(n-C_3H_7)_3NHAsF_6$ | N-nitrosodimethylamine (NDA) | IV-5 |
| 3. $(n-C_3H_7)_4NPF_6$ | NDA | IV-5 |
| 4. $C_6H_5(CH_3)_3NPF_6$ | NDA | IV-6 |
| 5. $KAsF_6$ | NDA | IV-6 |
| 6. $NaPF_6$ | NDA | IV-6 |
| 7. KPF_6 | NDA | IV-7 |
| 8. KPF_6 | NDA (undistilled) | IV-7 |
| 9. $CsPF_6^a$ | NDA | IV-7 |
| 10. $MgCl_2^a$ | 80 Weight Percent Ethylene Carbonate (EC), 20 Weight Percent Propylene Carbonate (PC) | IV-8 |
| 11. $LiCl^a$ | Methylene Chloride (MC) | IV-8 |
| 12. $(CH_3)_4NCl^a$ | MC | IV-8 |
| 13. $LiAlCl_4$ | PC | IV-8 |

^a Electrolyte was saturated at one molal.

TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES

A. Lithium.

| Electrolyte Solute-Solvent | Reference Electrode | Counter Electrode | Current Densities (ma/cm ²) | Voltage of Anode vs. | | Open Circuit Voltage After Discharge | Observations & Remarks |
|--|------------------------|----------------------|---|-------------------------------------|-------|--|--------------------------------|
| | | | | Reference Electrode ^a | Final | | |
| | | | | Initial | Final | | |
| 1. 1m(n-C ₃ H ₇) ₄ NPF ₆ - DMF | Ag/AgCl | Ag/AgCl | 0.0 | -3.04 | -3.04 | | |
| | | | 10 | -2.76 | -2.65 | -3.02 | |
| | | | 20 | -2.20 | -1.95 | -2.98 | |
| | | | 30 | -2.12 | -1.43 | -2.95 | Test stopped after 40 seconds. |
| | | | 50 | -1.72 | -1.05 | -2.98 | Test stopped after 10 seconds. |
| | | | 100 | -1.50 | -1.00 | ----- | Test stopped after 14 seconds. |
| | | | 0.0 ^b | -3.22 | | | |
| | | | 0.1 | -3.20 | -3.00 | -3.19 | |
| | | | 10 | -2.10 | -2.70 | -3.20 | |
| | | | | | | | |
| 2. 1m(n-C ₃ H ₇) ₃ NHAsF ₆ - NDA | Ag/AgCl | Ag/AgCl | 0.0 | -3.00 | -3.00 | | |
| | | | 10 | -2.90 | -2.85 | ----- | |
| | | | 100 | -1.97 | -1.81 | -2.85 | |
| | | | 0.0 ^b | -3.30 | | | |
| | | | 0.1 | -3.30 | -2.90 | -3.00 | |
| | | | 10 | -2.74 | ----- | | |
| | | | 100 | -1.44 | ----- | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| 3. 1m(n-C ₃ H ₇) ₄ NPF ₆ - NDA | Ag/AgCl | Ag/AgCl | 0.0 | -2.83 | -2.83 | | |
| | | | 40 | -2.27 | -2.04 | -2.82 | |
| | | | 50 | -2.06 | -1.76 | ----- | |
| | | | 60 | -1.94 | -1.66 | -2.80 | |
| | | | 75 | -1.70 | -1.60 | ----- | |
| | | | 100 | -1.38 | -1.35 | -2.80 | |
| | | | 0.0 ^b | -3.00 | | | |
| | | | 0.1 | -2.92 | -2.92 | -2.95 | |
| | | | 1.0 | -2.90 | -2.88 | -2.95 | |
| | | | 10 | -2.74 | -2.64 | -2.90 | |
| 100 | -1.80 | -1.54 | | | | | |

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

^b Test previously reported in Third Quarterly Report at which time undistilled solvents were being used.

TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES (Continued)

A. Lithium. (Continued)

| Electrolyte Solute-Solvent | Reference Electrode | Counter Electrode | Current Densities (ma/cm ²) | Voltage of Anode vs. Reference Electrode ^a Initial Final | Open Circuit Voltage After Discharge | Observations & Remarks |
|--|---------------------|-------------------|---|---|--------------------------------------|------------------------|
| 4. 1mC ₆ H ₅ (CH ₃) ₃ NPF ₆ -NDA | Ag/AgCl | Ag/AgCl | 0.0 | -2.83 | | |
| | | | 20 | -2.35 | -2.52 | -2.82 |
| | | | 50 | -2.13 | -2.22 | -2.84 |
| | | | 75 | -1.95 | -1.23 | -2.84 |
| | | | 100 | -1.78 | -0.50 | -2.84 |
| | | | 0.0 ^b | -3.25 | | |
| | | | 0.1 | -3.25 | -3.10 | -3.20 |
| | | | 1.0 | -2.84 | -2.78 | -3.05 |
| | | | 10 | -2.20 | -2.18 | -3.05 |
| | | | 100 | -1.20 | -0.80 | -2.95 |
| 5. 1mKAsF ₆ -NDA | Ag/AgCl | Ag/AgCl | 0.0 | -3.10 | | |
| | | | 20 | -2.77 | -2.71 | -3.09 |
| | | | 50 | -1.75 | -2.18 | -3.09 |
| | | | 100 | -0.80 | -1.00 | -2.95 |
| | | | 0.0 ^b | -3.05 | | |
| | | | 0.1 | -3.03 | -2.88 | -3.15 |
| | | | 1.0 | -2.48 | -2.80 | -3.15 |
| | | | 10 | -2.16 | -1.28 | -2.65 |
| | | | 100 | ----- | ----- | ----- |
| | | | 6. 1mNaPF ₆ -NDA | Ag/AgCl | Ag/AgCl | 0.0 |
| 0.1 | -3.24 | -3.24 | | | | -3.24 |
| 1.0 | -3.16 | -3.20 | | | | -3.24 |
| 10 | -2.73 | -2.81 | | | | -3.08 |
| 100 | -1.68 | -1.85 | | | | -2.75 |
| 0.0 ^b | -3.30 | | | | | |
| 0.1 | -3.24 | -3.25 | | | | -3.40 |
| 1.0 | -2.94 | -2.42 | | | | -2.80 |
| 10 | -2.10 | -1.52 | | | | -2.85 |
| 100 | ----- | ----- | | | | ----- |

Test stopped after 30 seconds.

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

^b Test previously reported in Third Quarterly Report at which time undistilled solvents were being used.

TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES (Continued)

A. Lithium. (Continued)

| Electrolyte Solute-Solvent | Reference Electrode | Counter Electrode | Current Densities (ma/cm ²) | Voltage of Anode vs. | | Open Circuit Voltage After Discharge | Observations & Remarks | | | |
|-------------------------------|------------------------|----------------------|--|-------------------------|--------------------|--|------------------------|-------|-------|--|
| | | | | Initial | Final ^a | | | | | |
| 7. 1mKPF ₆ -NDA | Ag/AgCl | Ag/AgCl | 0.0 | -3.23 | -3.19 | -3.22 | | | | |
| | | | 0.1 | -3.23 | -2.88 | -3.11 | | | | |
| | | | 1.0 | -2.95 | -2.71 | -3.05 | | | | |
| | | | 10 | -2.55 | -1.24 | -3.00 | | | | |
| | | | 100 | -1.24 | -3.20 | | | | | |
| | | | 0.0 ^b | -3.10 | -3.10 | -3.20 | | | | |
| | | | 0.1 | -2.70 | -2.83 | -2.85 | | | | |
| | | | 1.0 | -2.04 | -1.28 | -2.35 | | | | |
| | | | 10 | -2.04 | -0.30 | ----- | | | | |
| | | | 100 | -0.20 | ----- | ----- | | | | |
| | | | 8. 1mKPF ₆ -NDA ^c (undistilled) | Ag/AgCl | Ag/AgCl | 0.0 | -3.12 | -3.07 | -3.12 | |
| | | | | | | 0.1 | -3.08 | -2.86 | -3.09 | |
| 1.0 | -2.90 | -2.58 | | | | -2.92 | | | | |
| 10 | -2.30 | -1.42 | | | | ----- | | | | |
| 100 | -1.10 | ----- | | | | ----- | | | | |
| 0.0 | -3.34 | -3.32 | | | | -3.33 | | | | |
| 9. 0.5mCsPF ₆ -NDA | Ag/AgCl | Ag/AgCl | 0.1 | -3.34 | -3.15 | -3.29 | | | | |
| | | | 1.0 | -3.21 | -2.56 | -3.04 | | | | |
| | | | 10 | -2.59 | -1.52 | -2.96 | | | | |
| | | | 100 | -1.15 | -3.10 | | | | | |
| | | | 0.0 ^b | -3.10 | -2.90 | -3.05 | | | | |
| | | | 0.1 | -2.60 | -2.58 | -2.98 | | | | |
| 1.0 | -2.16 | -1.00 | -2.50 | | | | | | | |
| 10 | ----- | ----- | ----- | | | | | | | |
| 100 | ----- | ----- | ----- | | | | | | | |

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

^b Test previously reported in Third Quarterly Report at which time undistilled solvents were being used.

^c This test is a repeat of the preceding test.

TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES (Continued)

A. Lithium. (Continued)

| Electrolyte Solute-Solvent | Reference Electrode | Counter Electrode | Current Densities (ma/cm ²) | Voltage of Anode vs. | | Open Circuit Voltage After Discharge | Observations & Remarks |
|--|------------------------|----------------------|---|--|----------|--|--|
| | | | | Reference Electrode ^a Initial | Final | | |
| 10. MgCl ₂ - 80 Wt. % EC 20 Wt. % PC | Ag/AgCl | Ag/AgCl | 0.0 | -2.90 | -2.83 | -2.90 | The electrolyte was clear with a heavy gelatinous precipitate. No visible changes during the discharge. |
| | | | 0.1 | -2.83 | -2.69 | -2.87 | |
| | | | 1.0 | -2.68 | -2.54 | -2.84 | |
| | | | 5 | -2.50 | -2.43 | -2.83 | |
| | | | 10 | -2.36 | Reversed | -2.79 | |
| | | 50 | -1.66 | polarity after 1.5 minutes | | | |
| 11. LiCl-MC | Ag/AgCl | Ag/AgCl | 0.0 | -2.81 | | -2.70 | Clear yellow solution with white precipitate. No visible changes during the discharge. |
| | | | 0.1 | Reversed | polarity | | |
| 12. (CH ₃) ₄ NCl-MC | Ag/AgCl | Ag/AgCl | 0.0 | -2.87 | | -2.87 | Clear yellow solution with large amount of suspended white solid. No visible change during the discharge. |
| | | | 0.1 | Reversed | polarity | | |
| 13. LiAlCl ₄ -PC | Ag/AgCl | Ag/AgCl | 0.0 | -2.80 | -2.72 | -2.80 | No gassing occurred. |
| | | | 1.0 | -2.72 | -2.52 | -2.79 | |
| | | | 5 | -2.47 | -2.28 | -2.79 | |
| | | | 10 | -2.28 | -1.98 | -2.78 | |
| | | | 20 | -2.04 | Reversed | -2.76 | |
| | | 30 | -1.79 | polarity | | | |

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

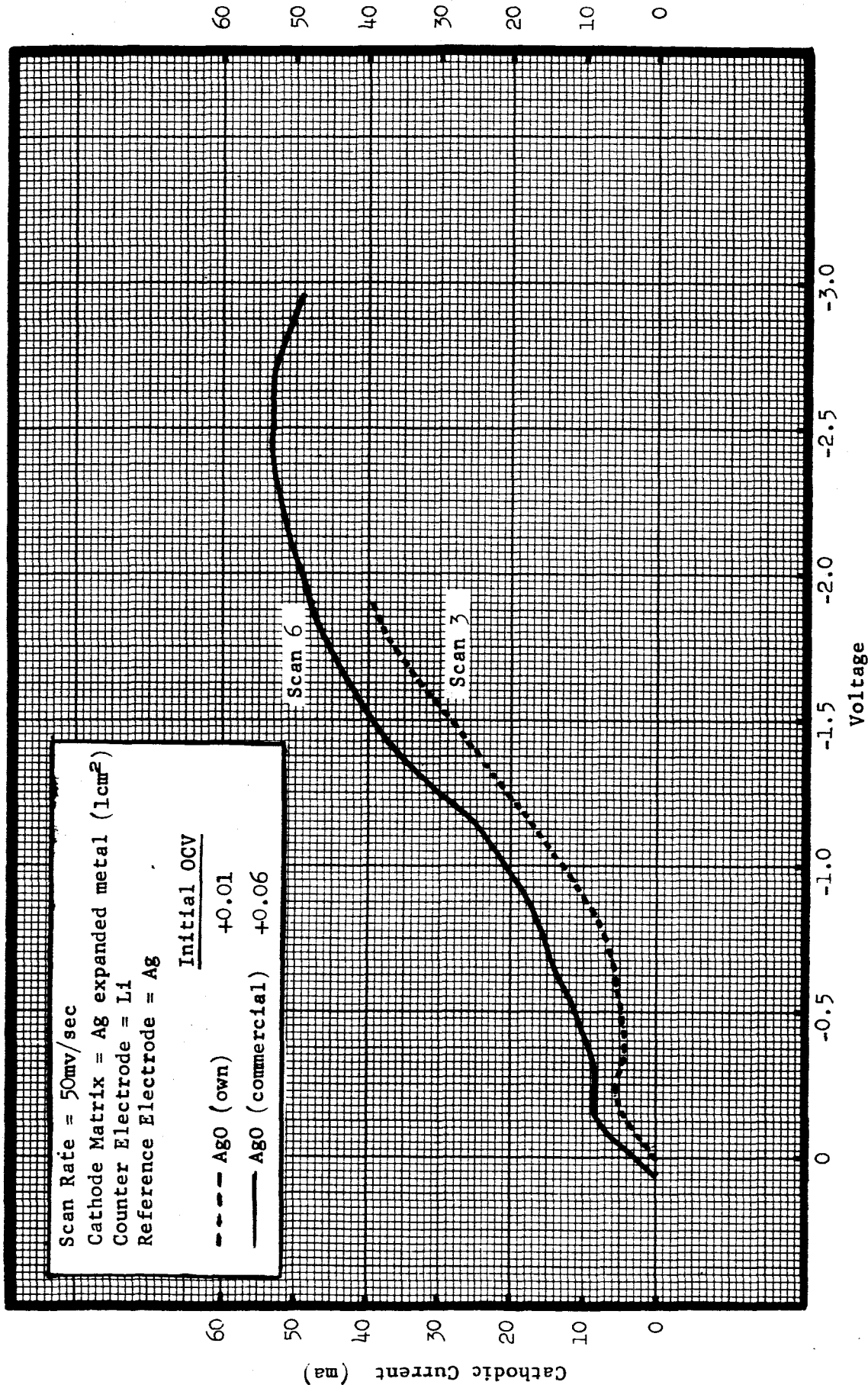


FIGURE 4. VOLTAMMETRIC STUDIES OF CATHODES

Figure 4--a. Voltammetric Sweep Studies of Commercially Obtained and Laboratory Prepared Silver Oxides in 1mLiPF₆-N-Nitrosodimethylamine.

The initial potential for each electrode was set equal to the open circuit voltage and the scans run towards more negative voltages.

^a "As received" LiPF₆ was used.

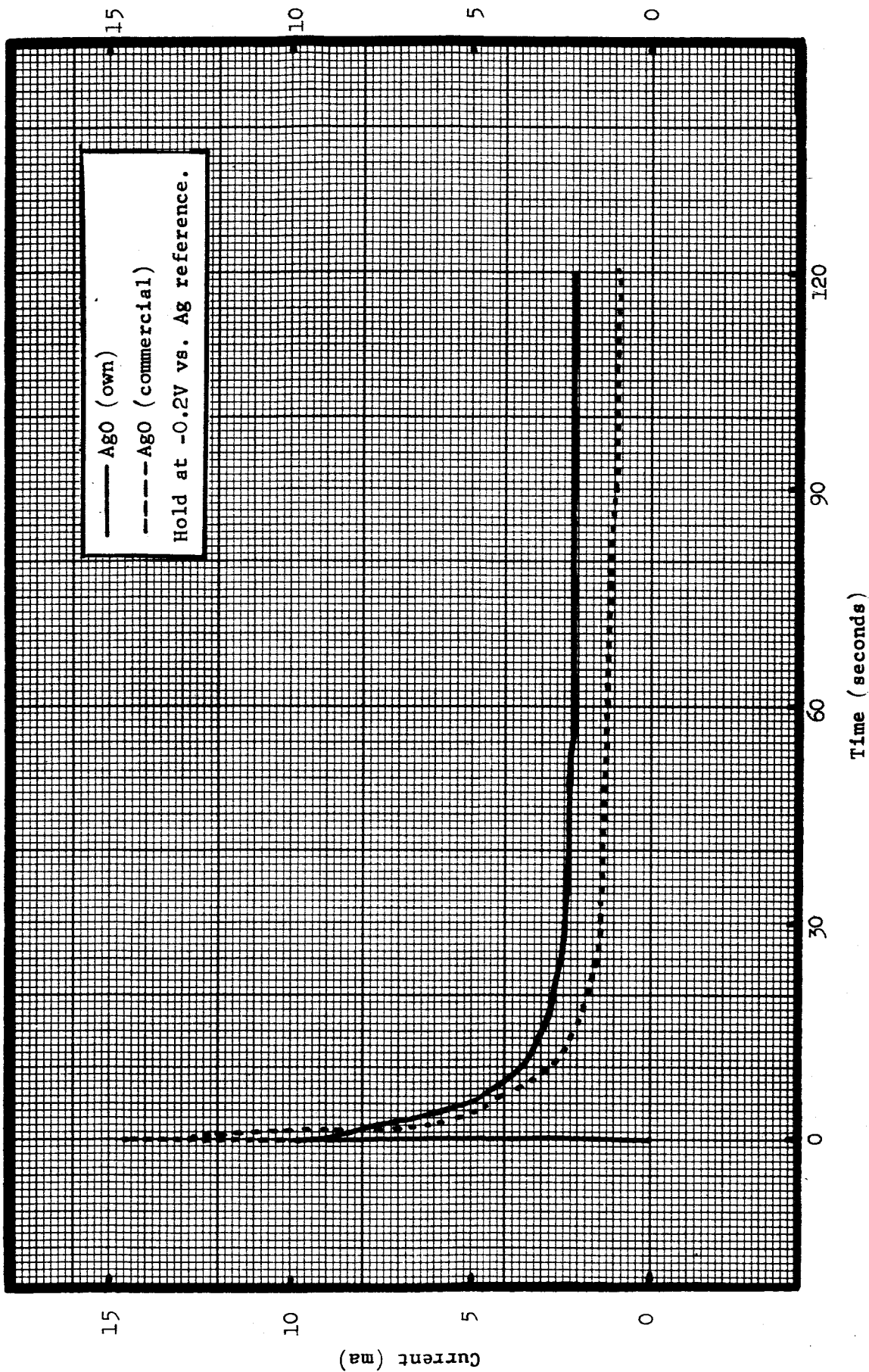


FIGURE 4. VOLTAMMETRIC STUDIES OF CATHODES (Continued)
 Figure 4-b. Potentiostatic Studies of Commercially Obtained and Laboratory Prepared Silver Oxides
 in 1mLiPF₆-N-Nitrosodimethylamine.

a "As received" LiPF₆.

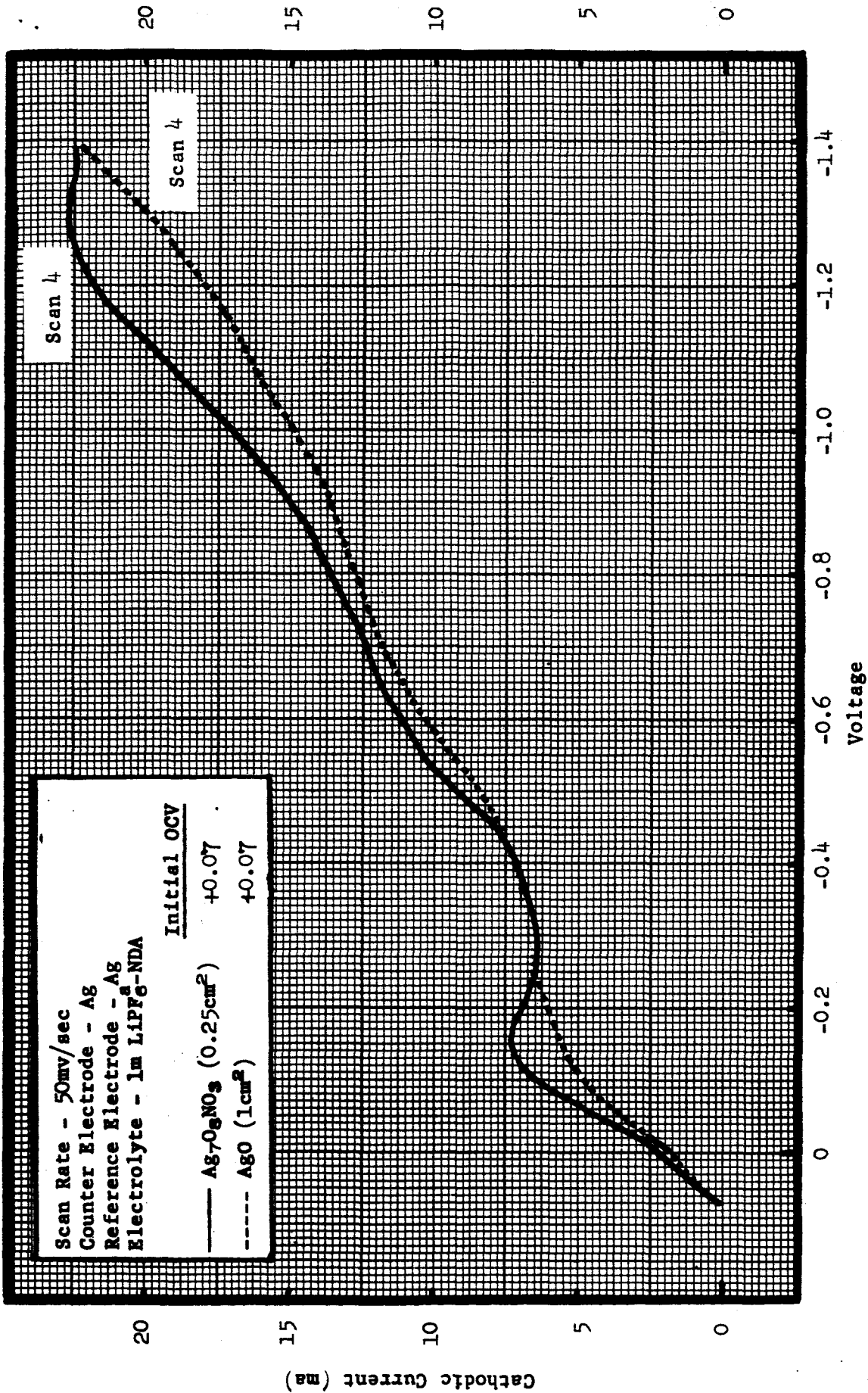


FIGURE 4. VOLTAMMETRIC STUDIES OF CATHODES (Continued)

Figure 4-c. Voltammetric Sweep Studies of Silver Oxynitrate and Silver Oxide (Laboratory Prepared) in LiPF₆-N-Nitrosodimethylamine.

The initial potential for each electrode was set equal to the open circuit voltage and the scans run towards more negative voltages.

^a Lithium hexafluorophosphate was used "as received."

TABLE II. ELECTROCHEMICAL HALF-CELL SCREENING OF INORGANIC CATHODES

A. Physical Details

| Active Cathode Material | Composition of Cathode Mix | Matrix | Method of Preparation | Electrolyte (1 Molal) | Counter Electrode | Reference Electrode |
|-------------------------|------------------------------------|--------|--|-----------------------|-------------------|---------------------|
| 1. $Ag_7O_8NO_3$ | 95% $Ag_7O_8NO_3$ 5% Solka-Floc | Ag | Mix was pressed on 1 cm^2 matrix at a pressure of 10,000 lbs/ cm^2 | $LiPF_6^a$ -NDA | Ag | Li |
| 2. $Ag_7O_8NO_3$ | 100% $Ag_7O_8NO_3$ | Ag | " | $LiPF_6^b$ -NDA | Ag/AgCl | Li |
| 3. AgO | 100% AgO | Ag | " | $LiPF_6^b$ -NDA | Ag/AgCl | Li |

^a $LiPF_6$ was used "as received" from the supplier.

^b Electrolyte was neutralized with lithium and filtered before using.

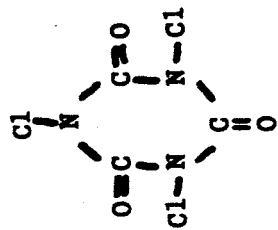
TABLE II. ELECTROCHEMICAL HALF-CELL SCREENING OF INORGANIC CATHODES
(Continued)

| A. Electrochemical Details | | | | | | | | | |
|--|----------------------------|---------------------|-------------------|---|---|---------------|--------------------------------------|---|--|
| Electrode | Electrolyte Solute-Solvent | Reference Electrode | Counter Electrode | Current Densities (ma/cm ²) | Voltage of Cathode vs. Reference Electrode ^a | Initial Final | Open Circuit Voltage After Discharge | Observations & Remarks | |
| 1. Ag ₇ O ₈ NO ₃ | LiPF ₆ NDA | Ag | Li | 0.0 | +0.21 | ----- | ----- | | |
| | | | | 0.1 | +0.17 | +0.15 | +0.15 | Anode and cathode gassed moderately at all current densities. | |
| | | | | 1.0 | +0.14 | +0.13 | +0.15 | | |
| | | | | 10 | -0.05 | -0.38 | +0.11 | | |
| | | | | 20 | -0.50 | -0.98 | +0.07 | | |
| 2. 100% Ag ₇ O ₈ NO ₃ | LiPF ₆ NDA | Ag/AgCl | Li | 0.0 | +0.61 | ----- | ----- | | |
| | | | | 1.0 | +0.59 | +0.53 | +0.57 | Electrolyte turned cloudy when 5ma load was applied. | |
| | | | | 5 | +0.43 | +0.12 | +0.45 | | |
| | | | | 10 | +0.09 | -0.31 | +0.31 | | |
| | | | | 20 | -0.07 | -1.35 | +0.03 | | |
| 3. 100% AgO | LiPF ₆ NDA | Ag/AgCl | Li | 0.0 | +0.59 | ----- | ----- | | |
| | | | | 1.0 | +0.54 | +0.41 | +0.51 | Solution turned cloudy when 20ma load was applied. | |
| | | | | 5 | +0.29 | +0.08 | +0.42 | | |
| | | | | 10 | -0.04 | -0.21 | +0.34 | | |
| | | | | 20 | -0.42 | -0.68 | +0.32 | | |
| | | | | 30 | -0.60 | -1.07 | +0.24 | | |
| | | | | 40 | -0.50 | -1.66 | +0.28 | | |
| | | | | 50 | -0.50 | -2.33 | +0.24 | | |

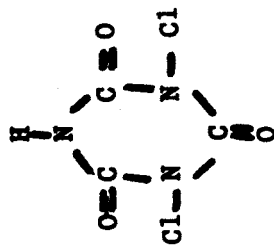
^a Duration of discharge at each current density - 5 minutes (unless otherwise noted).

TABLE III. COMPATIBILITY STUDIES: ANODES IN ORGANIC CATHODE SOLUTIONS

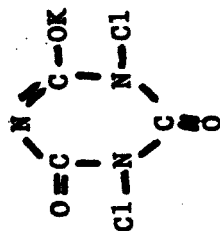
Code



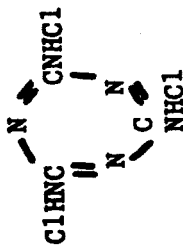
ACL-85^a



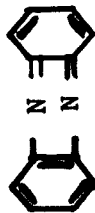
ACL-70^a



ACL-59^a



Trichloromelamine



Phenazine

^a Obtained from Monsanto Chemical Company

TABLE III. COMPATIBILITY STUDIES: ANODES IN ORGANIC CATHODE SOLUTIONS

| Active Cathode Material (Concentration) | Electrolyte (1 Molal) | Anode Material | Electrode | Open Circuit Voltages | | | | | | | | Observations |
|---|---|----------------|------------------|-----------------------|----------------|----------------|----------------|----------------|----------------|--|--|--|
| | | | | Initial | One Day | One Wk. | Two Wks. | Three Wks. | Four Wks. | | | |
| 1. ACL-59 (1.0m) | LiCl-AlCl ₃ -Li PC ^a | Li | Anode Cathode | -2.83 +1.02 | -2.81 +1.08 | -2.82 +1.00 | -2.69 +1.05 | -2.83 +0.97 | -2.77 +0.88 | | | The original electrolyte was light brown and slightly turbid. After adding ACL-59, the solution was opaque yellow-green. The ACL-59 did not dissolve completely. At the end of the test, the solution consisted of a light-green gel covered with a small amount of clear yellow-green liquid. A heavy white precipitate was present. The lithium was covered with a loosely-adhering light green gel. |
| 2. ACL-59 (1.0m) | MgCl ₂ -80 Wt. % EC, 20 Wt. % PC ^b | Li | Anode Cathode | -2.58 +1.26 | -2.88 +1.04 | -2.80 +1.06 | -2.21 +1.06 | -2.75 +1.05 | -2.64 +1.09 | | | The original electrolyte was white and turbid. After adding ACL-59, the solution was opaque yellow-green. The ACL-59 did not dissolve completely. At the end of the test, the solution was almost solidified. The lithium was covered with an uneven light green film and appeared to be diminished in size. |

^a This electrolyte was prepared by saturating the propylene carbonate with lithium chloride and then slowly adding enough aluminum chloride to make a 1 molal solution.

^b The electrolyte was saturated at 1 molal. The solution was centrifuged and decanted away from undissolved solute prior to use.

TABLE III. COMPATIBILITY STUDIES: ANODES IN ORGANIC CATHODE SOLUTIONS (Continued)

| Active Cathode Material (Concentration) | Electrolyte (1 Molal) | Anode Material | Electrode | Open Circuit Voltages | | | | Observations | | |
|---|--|----------------|------------------|-----------------------|----------------|----------------|----------------|----------------|----------------|--|
| | | | | Initial | One Day | One Wk. | Two Wks. | | Three Wks. | Four Wks. |
| 3. ACL-59 (1.0m) | (CH ₃) ₄ NPF ₆ ⁻ 80 Wt. % EC 20 Wt. % PC ^a | Li | Anode Cathode | -2.97 +0.90 | -2.96 +0.81 | -2.92 +0.93 | -2.78 +0.92 | -2.80 +1.00 | -2.80 +0.97 | The original electrolyte was clear and colorless. The ACL-59 was only slightly soluble. At the end of the test, the lithium was covered with a black film. No other changes occurred. |
| 4. ACL-70 (1.0m) | LiCl-AlCl ₃ -PC ^b | Li | Anode Cathode | -2.70 +1.27 | -2.85 +1.08 | -2.79 +1.08 | -2.69 +1.04 | -2.87 +1.00 | -2.55 +1.12 | The original electrolyte was light brown and slightly turbid. After adding ACL-70, the solution was opaque yellow-white. At the end of the test, the solution consisted of a light green gel with a heavy white precipitate. The lithium was covered with a loosely adhering, light green gel. |

^a The electrolyte was saturated at 1 molal. The solution was centrifuged and decanted away from undissolved solute prior to use.

^b This electrolyte was prepared by saturating the propylene carbonate with lithium chloride and then slowly adding enough aluminum chloride to make a 1 molal solution.

TABLE III. COMPATIBILITY STUDIES: ANODES IN ORGANIC CATHODE SOLUTIONS (Continued)

| Active Cathode Material (Concentration) | Electrolyte (1 Molal) | Anode Material | Electrode | Open Circuit Voltages | | | | | | | | Observations |
|---|---|----------------|------------------|-----------------------|----------------|----------------|----------------|----------------|----------------|--|---|--------------|
| | | | | Initial | One Day | One Wk. | Two Wks. | Three Wks. | Four Wks. | | | |
| 5. ACL-70 (1.0m) | MgCl ₂ -80 Wt. % EC, 20 Wt. % PC ^a | L1 | Anode Cathode | -2.96 +1.00 | -2.99 +0.96 | -2.77 +1.11 | -2.74 +0.43 | -2.74 +0.51 | -2.73 +0.52 | | The original electrolyte was white and turbid. After adding ACL-70, the solution was opaque yellow-green. At the end of the test, the solution consisted of a light green slurry covered with a small amount of clear yellow-green liquid. The lithium was covered with an uneven light green film. | |
| 6. ACL-70 (1.0m) | (CH ₃) ₄ NPF ₆ - 80 Wt. % EC ^a 20 Wt. % PC | L1 | Anode Cathode | -2.91 +0.20 | -3.13 +0.82 | | | | | | The original electrolyte was clear and colorless. After adding ACL-70, the solution was opaque white. At the end of the test, the solution was turbid yellow-green with a white precipitate. The anode was covered with white crystals and was partially corroded. | |

^a The electrolyte was saturated at 1 molal. The solution was centrifuged and decanted away from undissolved solute prior to use.

TABLE III. COMPATIBILITY STUDIES: ANODES IN ORGANIC CATHODE SOLUTIONS (Continued)

| Active Cathode Material (Concentration) | Electrolyte (1 Molal) | Anode Material | Electrode | Open Circuit Voltages | | | | | | | | Observations |
|---|---|----------------|------------------|-----------------------|----------------|----------------|----------------|----------------|----------------|--|---|--------------|
| | | | | Initial | One Day | One Wk. | Two Wks. | Three Wks. | Four Wks. | | | |
| 7. ACL-85 (0.2m) | LiCl-AlCl ₃ -PC ^a | Li | Anode Cathode | -3.00 +1.09 | -2.93 +1.11 | -2.90 +1.01 | -2.83 +0.97 | -2.88 +0.38 | -2.87 +0.27 | | The original electrolyte was light brown and slightly turbid. After adding ACL-85, the solution was yellow-green. At the end of the test, the solution was turbid yellow with a slight white precipitate. The lithium was covered with an uneven white film. | |
| 8. ACL-85 (0.2m) | $ \begin{array}{c} \text{CH}_2\text{CH}_2 \\ \quad \\ \text{O} \quad \text{NH}_2\text{PF}_6 \\ \quad \\ \text{CH}_2\text{CH}_2 \\ \text{-PC} \end{array} $ | Li | Anode Cathode | -3.11 +0.98 | -3.19 +0.97 | -2.97 +0.41 | | | | | The original electrolyte was clear and colorless. After adding ACL-85, the solution was clear yellow. The lithium gassed slightly on immersion. At the end of the test, the solution was dark and viscous with a slight yellow precipitate. The lithium was partially consumed. | |

^a This electrolyte was prepared by saturating the propylene carbonate with lithium chloride and then slowly adding enough aluminum chloride to make a 1 molal solution.

TABLE III. COMPATIBILITY STUDIES: ANODES IN ORGANIC CATHODE SOLUTIONS (Continued)

| Active Cathode Material (Concentration) | Electrolyte (1 Molal) | Anode Material | Electrode | Open Circuit Voltages | | | | | | | | Observations |
|---|--|----------------|------------------|-----------------------|----------------|----------------|----------------|----------------|----------------|--|--|--------------|
| | | | | Initial | One Day | One Wk. | Two Wks. | Three Wks. | Four Wks. | | | |
| 9. ACL-85 (0.2m) | MgCl ₂ -80 Wt. % EC 20 Wt. % PC ^a | Li | Anode Cathode | -2.97 +1.03 | -2.89 +1.08 | -2.87 +0.99 | -2.87 +0.92 | -2.83 +0.94 | -2.72 +0.93 | The original electrolyte was clear and colorless. ACL-85 dissolved completely. The lithium gassed slightly on immersion. At the end of the test, the solution was turbid orange with a slight yellow precipitate. The lithium was gray with white crystals and was partially consumed. | | |
| 10. ACL-85 (0.2m) | (CH ₃) ₄ NPF ₆ ⁻ 80 Wt. % EC 20 Wt. % PC ^a | Li | Anode Cathode | -2.87 +1.08 | -2.91 +1.08 | -2.88 +0.42 | -2.80 +0.28 | -2.87 +0.22 | -2.75 +0.27 | The original electrolyte was white and turbid. After adding ACL-85, the solution was opaque yellow-green. At the end of the test, the solution was orange-brown and gelled at the surface. A heavy white precipitate had formed. The lithium appeared unchanged. | | |

^a The electrolyte was saturated at 1 molal. The solution was centrifuged and decanted away from undissolved solute prior to use.

TABLE III. COMPATIBILITY STUDIES: ANODES IN ORGANIC CATHODE SOLUTIONS (Continued)

| Active Cathode Material (Concentration) | Electrolyte (1 Molal) | Anode Material | Electrode | Open Circuit Voltages | | | | | | | Observations |
|---|--|----------------|------------------|-----------------------|----------------|----------------|----------------|----------------|----------------|--|--------------|
| | | | | Initial | One Day | One Wk. | Two Wks. | Three Wks. | Four Wks. | | |
| 11. ACL-85 (0.2m) | $\begin{array}{c} \text{CH}_2\text{CH}_2 \\ \\ \text{O} \\ \\ \text{CH}_2\text{CH}_2 \\ \\ \text{NH}_2\text{PF}_6 \end{array}$ - 80 Wt. % EC, 20 Wt. % PC | L1 | Anode Cathode | -3.23 +0.82 | -3.03 +1.10 | -2.90 +0.98 | -2.87 +0.36 | -2.72 +0.26 | -2.73 +0.21 | The original electrolyte was clear and colorless. After adding ACL-85, the solution was clear yellow. The lithium gassed slightly on immersion. At the end of the test, the solution was dark brown with yellow crystals and was partially corroded. | |
| 12. Phenazine (1.0m) | MgCl ₂ - 80 Wt. % EC, 20 Wt. % PC ^a | L1 | Anode Cathode | -2.97 +0.02 | -2.85 +0.56 | -2.86 +0.18 | -2.86 +0.25 | -2.81 +0.18 | -2.77 +0.30 | The original electrolyte was white and turbid. On adding phenazine, the solution formed a yellow-orange suspension. At the end of the test, the solution was a viscous, yellow-orange slurry containing some black particles. The lithium was coated with a gelatinous film. | |

^a The electrolyte was saturated at 1 molal. The solution was centrifuged and decanted away from undissolved solute prior to use.

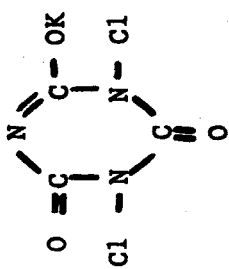
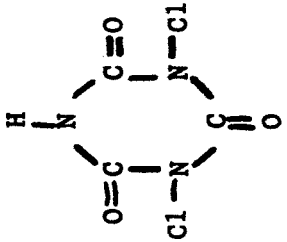
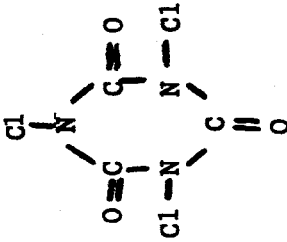

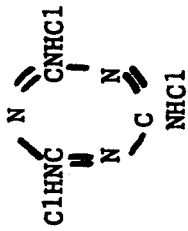
TABLE III. COMPATIBILITY STUDIES: ANODES IN ORGANIC CATHODE SOLUTIONS (Continued)

| Active Cathode Material (Concentration) | Electrolyte (1 Molal) | Anode Material | Electrode | Open Circuit Voltages | | | | Observations | | |
|---|---|----------------|------------------|-----------------------|----------------|----------------|----------------|----------------|----------------|--|
| | | | | Initial | One Day | One Wk. | Two Wks. | | Three Wks. | Four Wks. |
| 13. Tri-chloro-melamine (0.2m) | LiCl-AlCl ₃ -PC ^a | Li | Anode Cathode | -2.87 +1.09 | -3.04 +0.82 | -2.92 +1.00 | -2.87 +1.03 | -2.83 +1.01 | -2.75 +1.01 | The original electrolyte was light brown and clear. After adding TCM, the solution was opaque light green. At the end of the test the electrolyte was gelled. The lithium was covered with a yellow gelatinous film. |
| 14. Tri-chloro-melamine (0.2m) | MgCl ₂ - 80 Wt. % EC, 20 Wt. % PC ^b | Li | Anode Cathode | -2.93 +1.01 | -2.89 +0.80 | -2.82 +0.81 | -2.81 +0.97 | -2.79 +0.88 | -2.74 +0.82 | The original electrolyte was clear and colorless. After adding TCM, the solution was opaque yellow-white. At the end of the test, the solution was opaque orange and almost solidified. The lithium was gray-green and was partially corroded. |

^a This electrolyte was prepared by saturating the propylene carbonate with lithium chloride and then slowly adding enough aluminum chloride to make a 1 molal solution.

^b The electrolyte was saturated at 1 molal. The solution was centrifuged and decanted away from undissolved solute prior to use.

TABLE IV. ELECTROCHEMICAL HALF-CELL TESTING OF ORGANIC CATHODES

| Code | Chemical Structure | Code | Chemical Structure | Code | Chemical Structure | |
|------|---|---------------------|--|---------------------|---|---------------------|
| |  | ACL-59 ^a |  | ACL-70 ^a |  | ACL-85 ^a |
| |  | |  | | | |
| | Phenazine | | Trichloromelamine | | | |
| EC | - Ethylene carbonate | | | | | |
| PC | - Propylene carbonate | | | | | |
| AB | - Acetylene black | | | | | |
| FP | - Filter paper pulp | | | | | |
| BM | - Ball-milled in heptane, filtered, and vacuum-dried | | | | | |
| BL | - Blended in heptane, filtered, and vacuum-dried | | | | | |
| TCM | - Trichloromelamine | | | | | |
| PG | - Pyrolytic graphite | | | | | |

^a Obtained from Monsanto Chemical Company.

TABLE IV. ELECTROCHEMICAL HALF-CELL TESTING OF ORGANIC CATHODES

A. Dissolved Organic Cathodes.

| Cathode (Molar Concentration) | Electrolyte (1 Molal) | | Current Collector | Reference Electrode | Counter Electrode | Current Density (ms/cm ²) | Voltage of Cathode Vs. Reference Electrode | | Open Circuit Voltage After Discharge | Observations and Remarks |
|-------------------------------|--------------------------|----------------------------------|-------------------|---------------------|-------------------|---------------------------------------|--|----------------|--------------------------------------|---|
| | Solvent | Solute | | | | | Initial | Final | | |
| 1. ACL-85 (0.1) % PC | 80 Wt. % EC, 20 Wt. % PC | MgCl ₂ ^b | PG | Ag/AgCl | Li | 0.0 | +1.08 | ----- | ----- | The original electrolyte was opaque white and extremely viscous. After adding ACL-85 the solution was light green. No visible changes occurred during the discharges. |
| | | | | | | 0.1 | +1.07 | +1.05 | +1.08 | |
| | | | | | | 1.0 | +0.91 | +0.88 | +1.08 | |
| | | | | | | 5.0 | +0.44 | -3.82 (3 min.) | +1.08 | |
| 2. Phenazine (1.0) % PC | 80 Wt. % EC, 20 Wt. % PC | MgCl ₂ ^{b,c} | PG | Ag/AgCl | Li | 0.0 | +0.05 | ----- | ----- | The original electrolyte was clear. A yellow-orange suspension formed when phenazine was added. No visible changes occurred during the discharges. |
| | | | | | | 0.1 | -0.01 | -0.02 | +0.06 | |
| | | | | | | 1.0 | -0.20 | -2.4 (2 min.) | -0.03 | |
| | | | | | | 5.0 | ----- | ----- | ----- | |
| 3. TCM (0.1) % PC | 80 Wt. % EC, 20 Wt. % PC | MgCl ₂ ^b | PG | Ag/AgCl | Li | 0.0 | +0.99 | ----- | ----- | The original electrolyte was white and viscous. A yellow-white gel formed when TCM was added. The gel adhered to all three electrodes. The solution was deep yellow in the vicinity of the reference electrode after the discharges. No other changes occurred. |
| | | | | | | 0.1 | +0.91 | +0.87 | +1.00 | |
| | | | | | | 1.0 | +0.62 | -0.52 | +0.93 | |
| | | | | | | 5.0 | ----- | -2.0 (40 sec.) | +0.92 | |

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

^b The electrolyte was saturated at 1 molal.

^c The electrolyte was centrifuged and decanted away from the undissolved solute.

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

B. Pelletized^d Organic Cathodes.

| Cathode Composition (Wt. %) | | Electrolyte (1 Molal) | | Reference Electrode | Counter Electrode | Current Density (ma/cm ²) | Voltage of Cathode Vs. Reference Electrode ^a | | Open Circuit Voltage After Discharge | Observations and Remarks | |
|-----------------------------|------------------------------|-----------------------|-------------------------|--|-------------------|---------------------------------------|---|-------|--------------------------------------|--------------------------|--|
| | | Solvent | Solute | | | | Initial | Final | | | |
| 1. ACL-59 (80) | AB (10) FP (10) Binder | Prep. of Mix | 80 Wt. % EC, 20 Wt % PC | MgCl ₂ ^{b, c} | Ag/AgCl | Li | 0.0 | +0.8 | ---- | ---- | The original electrolyte was slightly turbid. The cathode pellet disintegrated slightly on immersion. No visible changes occurred during the discharges. |
| | | | | | | | 0.1 | +0.02 | -0.03 | +0.07 | |
| | | | | | | | 0.5 | -0.22 | -0.14 | +0.02 | |
| | | | | | | | 1.0 | -0.30 | -0.27 | 0.00 | |
| | | | | | | | 2.0 | -0.47 | -0.63 | 0.00 | |
| 5.0 | -0.7 | -2.0 | 0.00 | | | | | | | | |
| 2. ACL-59 (80) | AB (10) FP (10) Binder | Prep. of Mix | 80 Wt. % EC, 20 Wt % PC | (CH ₃) ₄ NPF ₆ ^{b, c} | Ag/AgCl | Li | 0.0 | +0.57 | ---- | ---- | The original electrolyte was clear and colorless. The cathode pellet disintegrated slightly during the discharges. No other changes occurred. |
| | | | | | | | 0.1 | +0.56 | +0.48 | +0.54 | |
| | | | | | | | 0.5 | +0.50 | +0.34 | +0.50 | |
| | | | | | | | 1.0 | +0.40 | +0.24 | +0.48 | |
| | | | | | | | 2.0 | +0.12 | +0.13 | +0.46 | |
| 5.0 | -0.18 | -0.12 | +0.44 | | | | | | | | |
| 7.0 | -0.2 | -0.32 | +0.42 | | | | | | | | |
| 10.0 | -0.57 | -0.56 | +0.40 | | | | | | | | |
| 15.0 | -0.60 | -0.90 | +0.36 | | | | | | | | |
| 20.0 | -1.00 | -1.60 | +0.39 | | | | | | | | |

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

^b The electrolyte was saturated at 1 molal.

^c The electrolyte was centrifuged and decanted away from the undissolved solute.

^d Pressed at 10,000 lb/cm² on silver expanded metal.

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

B. Pelletized Organic Cathodes (Continued)

| Cathode Composition (Wt. %) | | Prep. of Mix | Electrolyte (1 Molal) | | Reference Electrode | Counter Electrode | Current Density (mA/cm ²) | Voltage of Cathode Vs. Reference Electrode ^a | | Open Circuit Voltage After Discharge | Observations and Remarks |
|-----------------------------|---------------------|--------------|-----------------------|---------|---------------------|-------------------|---------------------------------------|---|---------------|--------------------------------------|--|
| Active Material | Conductive Additive | | Binder | Solvent | | | | Solute | Initial | | |
| 3. ACL-59 (80) | AB (10) | FP (10) | BM | PC | Ag/AgCl | Li | 0.0 | +0.89 | ----- | ----- | The original electrolyte was light brown and clear. A few gas bubbles formed on the cathode surface during the discharges. At the end of the test, the cathode had disintegrated slightly and the lithium was covered with a thin gray film. |
| | | | | | | | 0.1 | +0.87 | +0.87 | +0.89 | |
| | | | | | | | 1.0 | +0.79 | +0.72 | +0.88 | |
| | | | | | | | 5.0 | +0.60 | -2.00 (3 min) | +0.88 | |
| 4. ACL-59 (80) | AB (10) | FP (10) | BM | PC | Ag/AgCl | Li | 0.0 | +0.92 | ----- | ----- | The original electrolyte was light brown and clear. No visible changes occurred during the discharges. |
| | | | | | | | 0.1 | +0.87 | +0.80 | +0.88 | |
| | | | | | | | 0.5 | +0.72 | -0.20 | +0.48 | |
| | | | | | | | 1.0 | -0.40 | -0.43 | +0.42 | |
| | | | | | | 2.0 | -0.65 | -2.80 | -0.03 | | |

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

^d Pressed at 10,000 lb/cm² on silver expanded metal.

^e Prepared by fusing a 1:1 mole ratio of LiCl and AlCl₃.

^f This electrolyte was prepared by saturating the propylene carbonate with lithium chloride and then slowly adding enough aluminum chloride to make a 1 molal solution.

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

B. Pelletized^d Organic Cathodes (Continued)

| Cathode Composition (Wt. %) | | | | Prep. of Mix | Electrolyte (1 Molal) | | Reference Electrode | Counter Electrode | Current Density (mA/cm ²) | Voltage of Cathode Vs. Reference Electrode ^a | | Open Circuit Voltage After Discharge | Observations and Remarks |
|-----------------------------|---------------------|---------|--|--------------|-----------------------|--------------------------|---------------------|-------------------|---|--|--|---|--|
| Active Material | Conductive Additive | Binder | | | Solvent | Solute | | | | Initial | Final | | |
| 5. ACL-70 (80) | AB (10) | FP (10) | | BL | PC | LiCl-f AlCl ₃ | Ag/AgCl | Li | 0.0 0.1 0.5 1.0 2.0 5.0 7.0 10.0 | +0.96 +0.95 +0.90 +0.84 +0.77 +0.61 +0.52 +0.38 | ----- +0.96 +0.94 +0.87 +0.96 +0.83 +0.74 +0.49 +0.23 -1.14 | ----- +0.96 +0.96 +0.96 +0.96 +0.96 +0.96 +0.96 +0.96 | The original electrolyte was light brown and slightly cloudy. The cathode disintegrated slightly on immersion and gas bubbles formed on the surface during the discharges. No other changes occurred. |
| 6. TCM (80) | AB (10) | FP (10) | | BL | PC | LiCl-f AlCl ₃ | Ag/AgCl | Li | 0.0 0.1 0.5 1.0 2.0 5.0 7.0 10.0 | +0.91 +0.34 +0.15 -0.12 -0.65 -1.05 -1.05 -1.26 | ----- +0.32 +0.09 -0.13 -0.32 -0.63 -0.76 -0.96 | ----- +0.40 +0.46 +0.46 +0.58 +0.56 +0.49 ----- | The original electrolyte was light brown and slightly turbid. The cathode disintegrated slightly on immersion. At the end of the test, the electrolyte had darkened slightly and the lithium was partially covered with black particles. |

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

^d Pressed at 10,000 lb/cm² on silver expanded metal.

^f This electrolyte was prepared by saturating the propylene carbonate with lithium chloride and then slowly adding enough aluminum chloride to make a 1 molal solution.

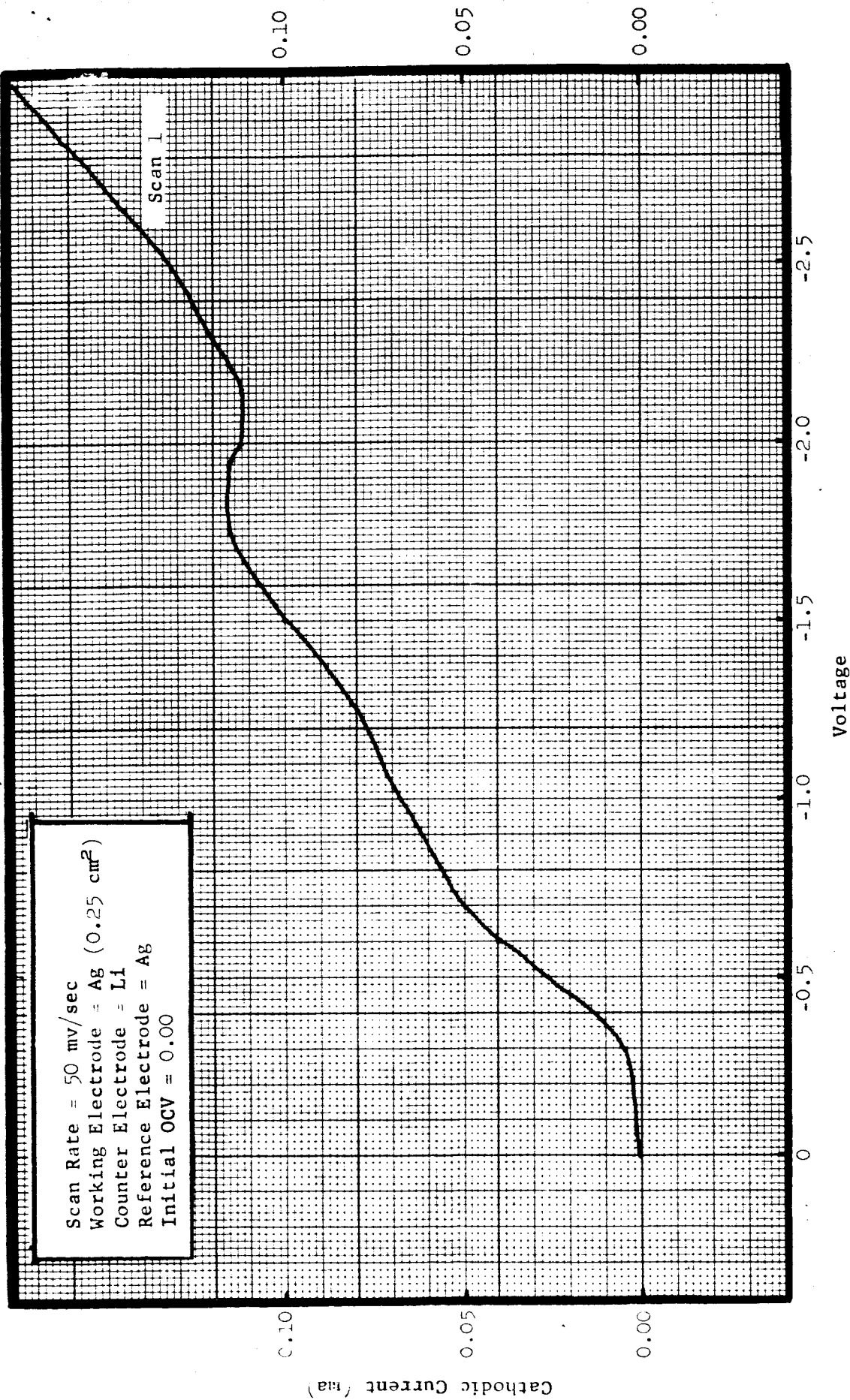


FIGURE 5. VOLTAMMETRIC SWEEP STUDIES OF ELECTROACTIVE IMPURITIES IN LITHIUM HEXAFLUOROPHOSPHATE

Figure 5-a. Voltammetric Sweep Study of Saturated Sodium Bifluoride in N-Nitrosodimethylamine. The initial potential was set equal to the open circuit voltage and the scan run towards more negative voltages.

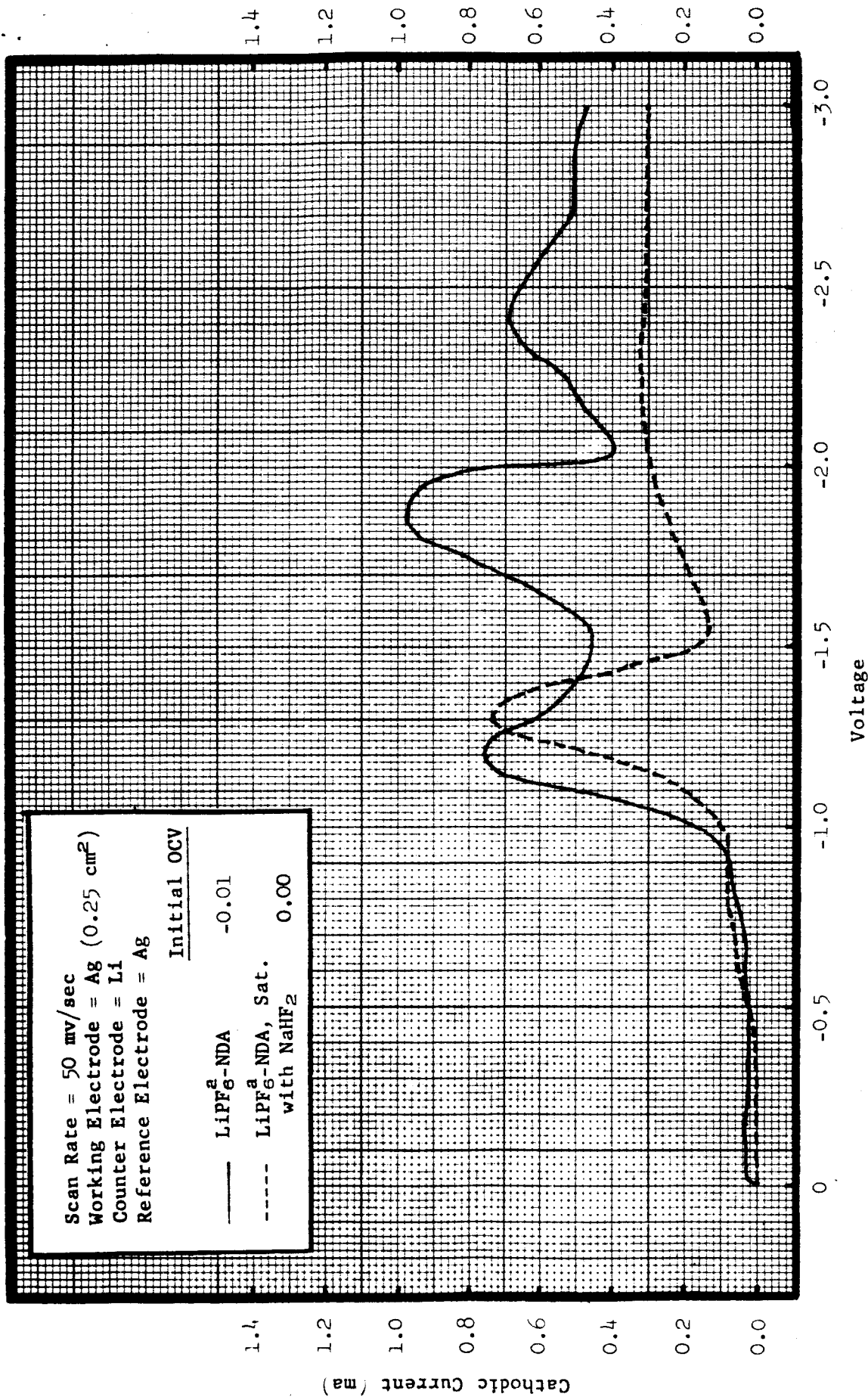


FIGURE 5. VOLTAMMETRIC SWEEP STUDIES OF ELECTROACTIVE IMPURITIES IN LITHIUM HEXAFLUOROPHOSPHATE (Continued)

Figure 5-b. Voltammetric Sweep Study of 1m LiPF₆-NDA and 1m LiPF₆-NDA Saturated with Sodium Bifluoride. The initial potentials were set equal to the open circuit voltages and the scans run towards more negative voltages.

^a LiPF₆ used "as received."

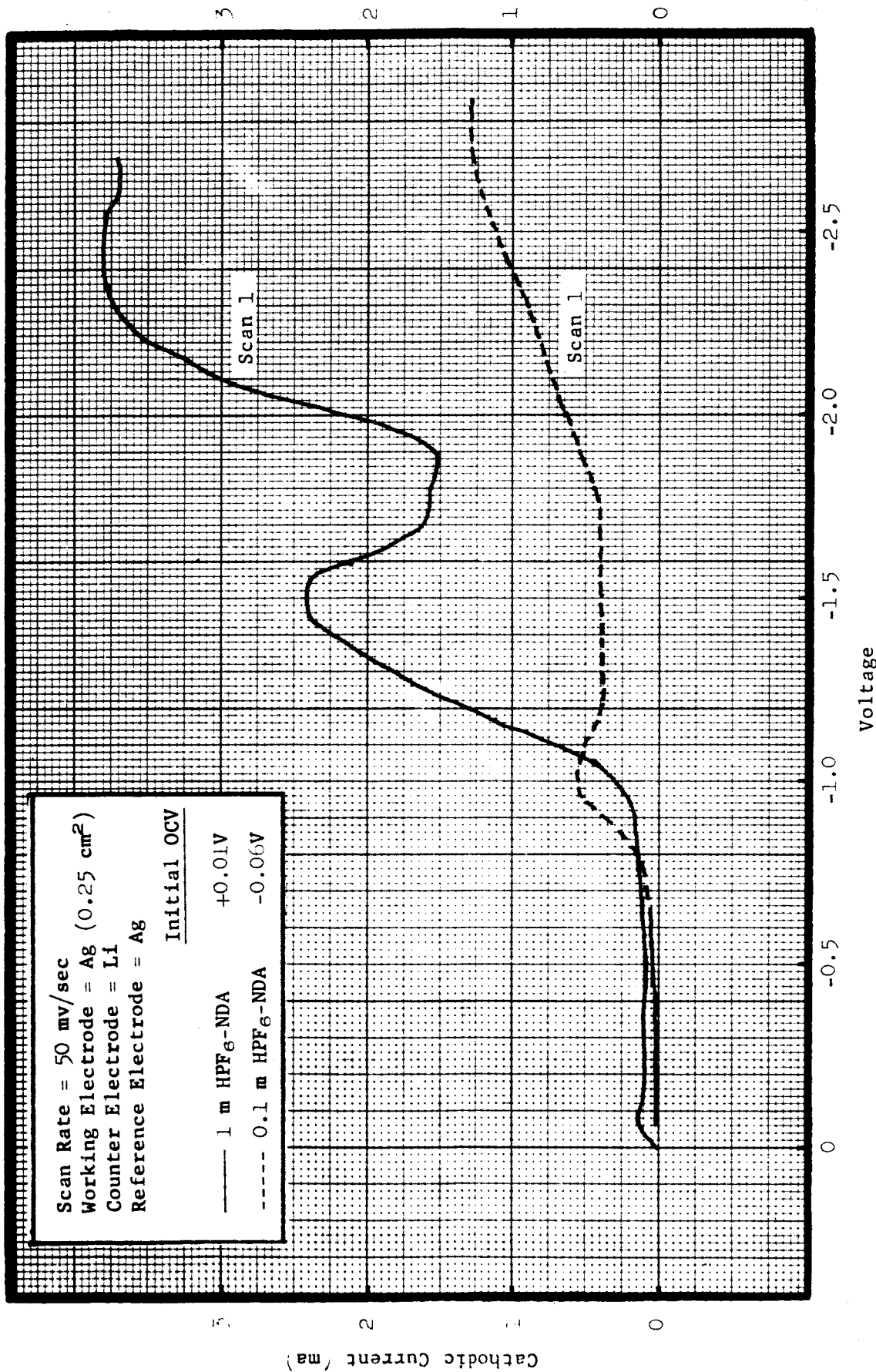


FIGURE 5. VOLTAMMETRIC SWEEP STUDIES OF ELECTROACTIVE IMPURITIES IN LITHIUM HEXAFLUOROPHOSPHATE (Continued)

Figure 5-c. Voltammetric Sweep Study of Hexafluorophosphoric Acid^a in N-Nitrosodimethylamine. The initial potentials were set equal to the open circuit voltages and the scans run towards more negative voltages.

^a Hexafluorophosphoric acid used was a 65% aqueous solution.

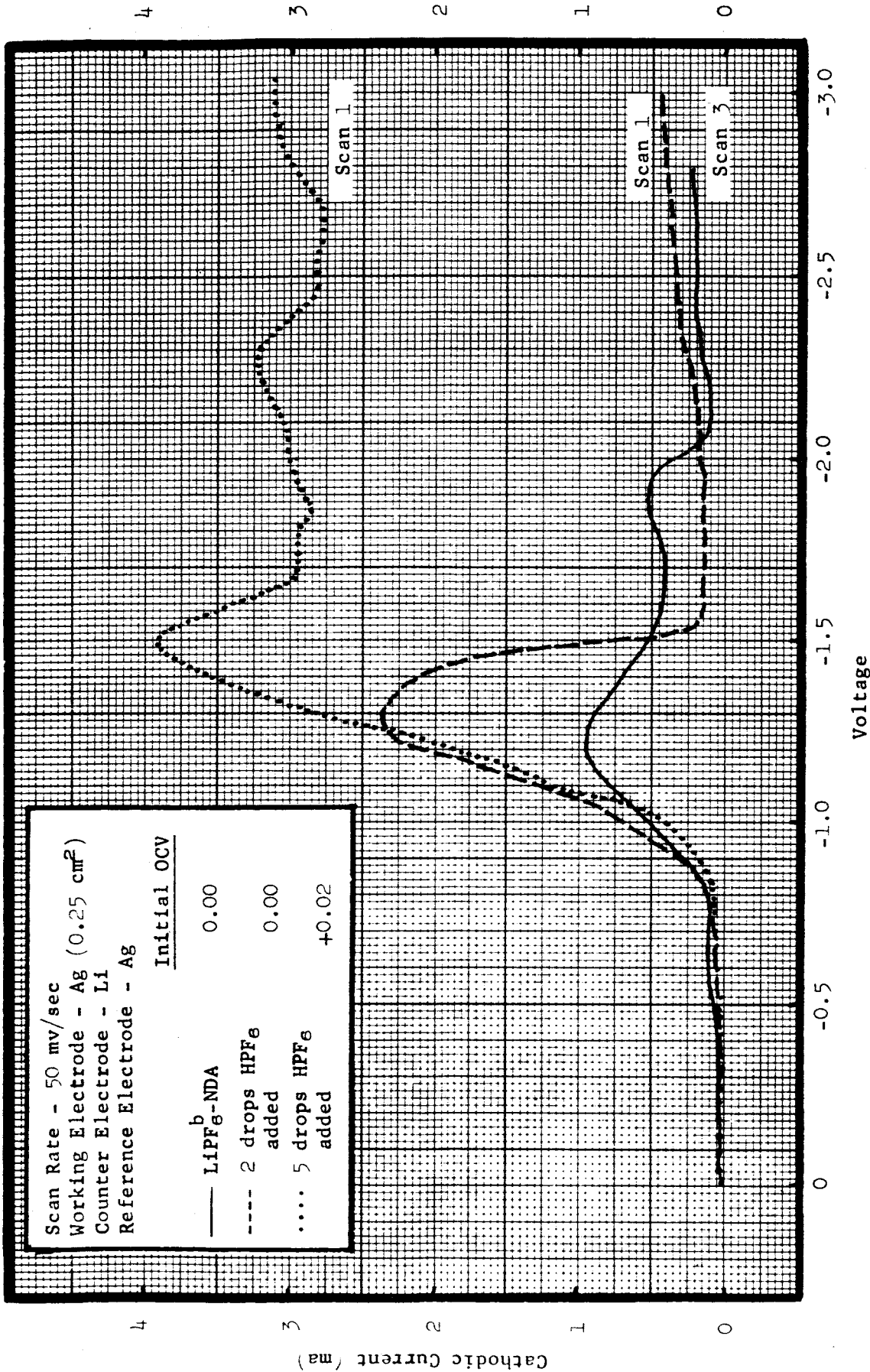


FIGURE 5. VOLTAMMETRIC SWEEP STUDIES OF ELECTROACTIVE IMPURITIES IN LITHIUM HEXAFLUOROPHOSPHATE (Continued)

Figure 5-d. Voltammetric Sweep Study of Adding Hexafluorophosphoric Acid^a to LIPF₆^b-N-Nitrosodimethylamine. The initial potentials were set equal to the open circuit voltages and the scans run towards more negative voltages.

^a Hexafluorophosphoric acid used was a 65% aqueous solution.

^b LIPF₆ used was dried at 110°C under nitrogen.

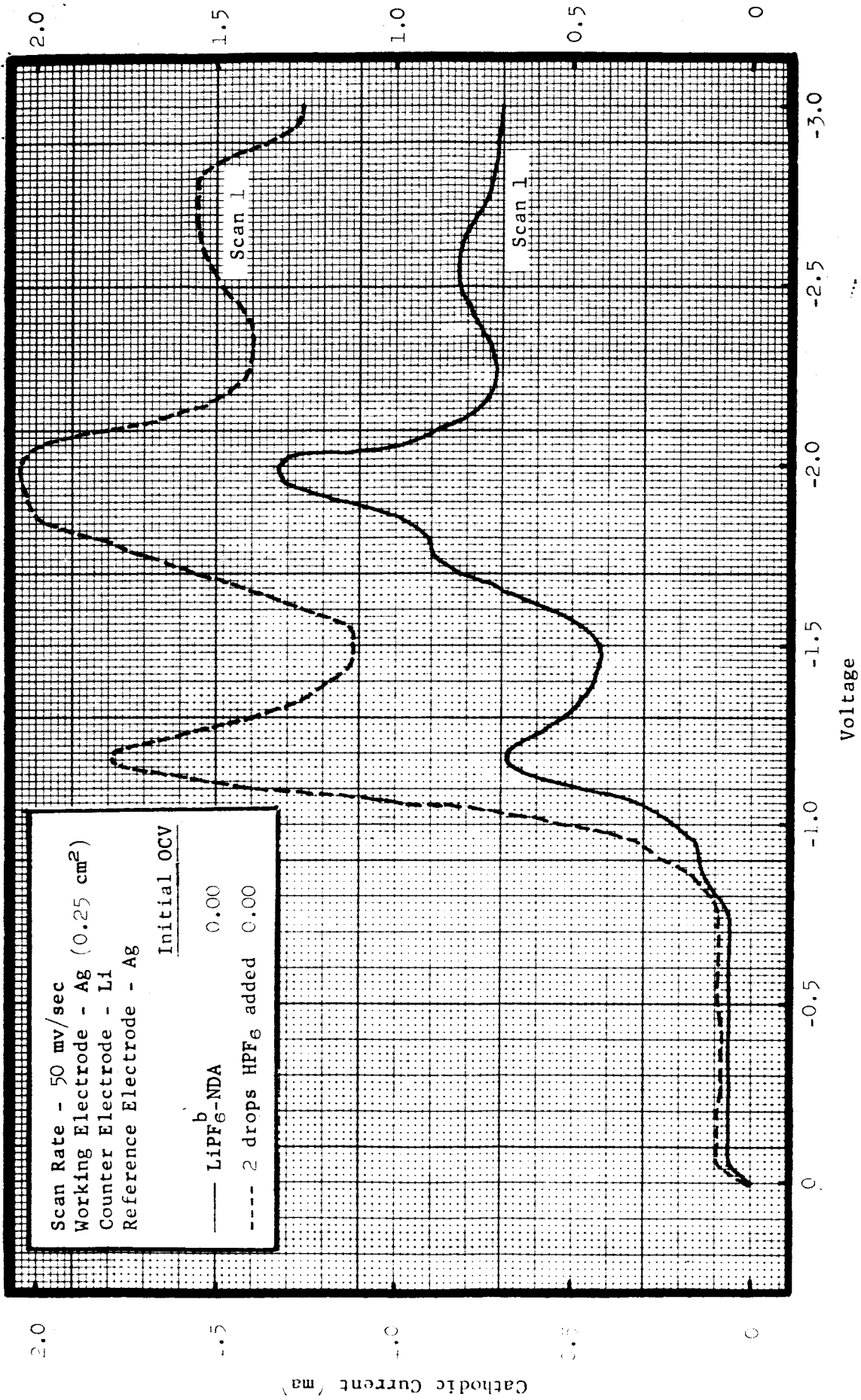
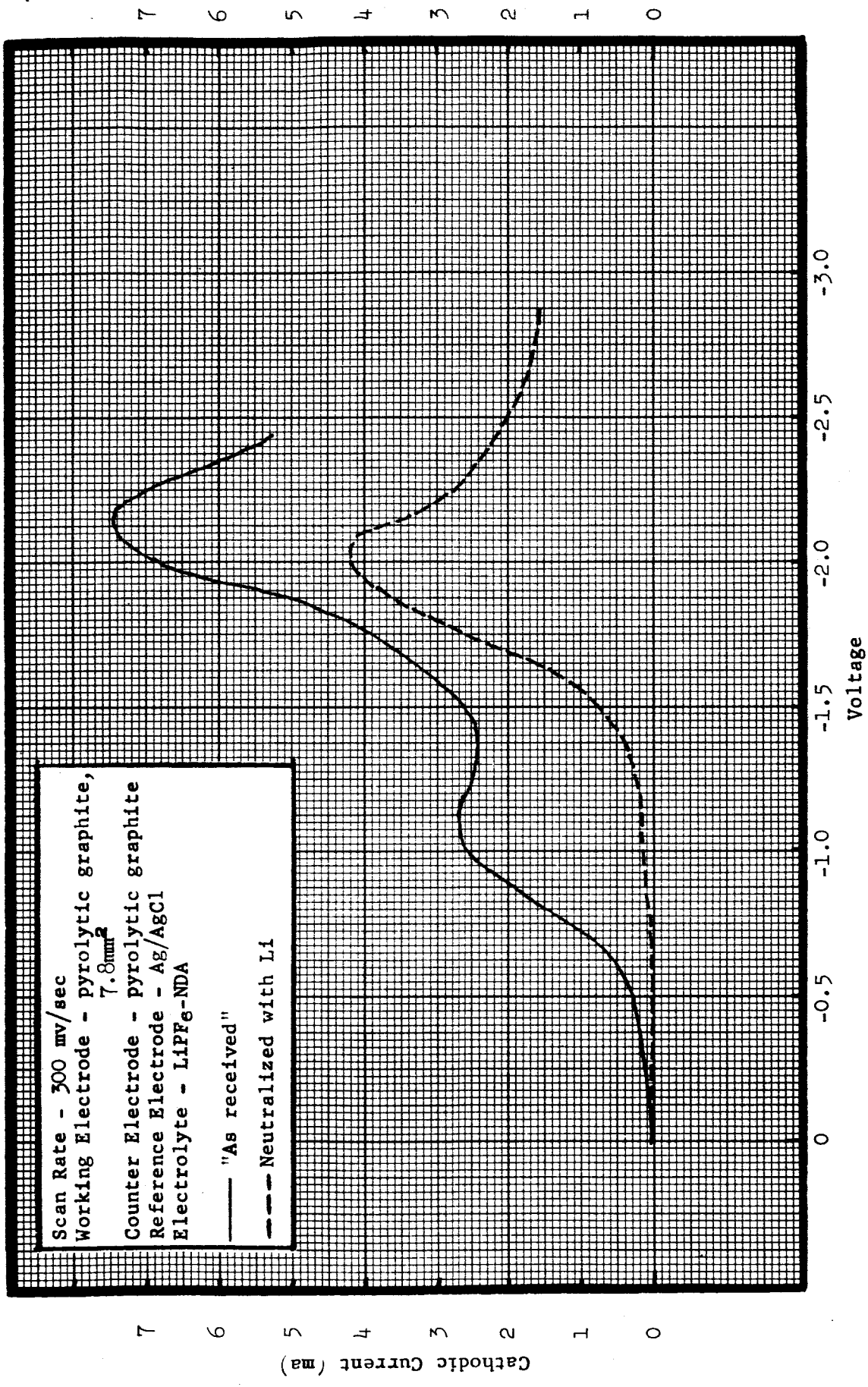


FIGURE 5. VOLTAMMETRIC SWEEP STUDIES OF ELECTROACTIVE IMPURITIES IN LITHIUM HEXAFLUOROPHOSPHATE. (Continued)

Figure 5-e. Voltammetric Sweep Study of Adding Hexafluorophosphoric Acid^a to LiPF₆-N-Nitrosodimethylamine. The initial potentials were set equal to the open circuit voltages and the scans run towards more negative voltages.

^a Hexafluorophosphoric acid used was a 65% aqueous solution.

^b LiPF₆ used "as received."



Scan Rate - 300 mv/sec
 Working Electrode - pyrolytic graphite,
 7.8mm²
 Counter Electrode - pyrolytic graphite
 Reference Electrode - Ag/AgCl
 Electrolyte - LiPF₆-NDA
 — "As received"
 - - - Neutralized with Li

FIGURE 5. VOLTAMMETRIC SWEEP STUDIES OF ELECTROACTIVE IMPURITIES IN LITHIUM HEXAFLUOROPHOSPHATE (Continued)

Figure 5-f. The Effect of Neutralizing the Lithium Hexafluorophosphate-N-Nitrosodimethylamine Solution with Lithium Metal.
 The initial potentials were set equal to the open circuit voltages and the scans run towards more negative voltages.

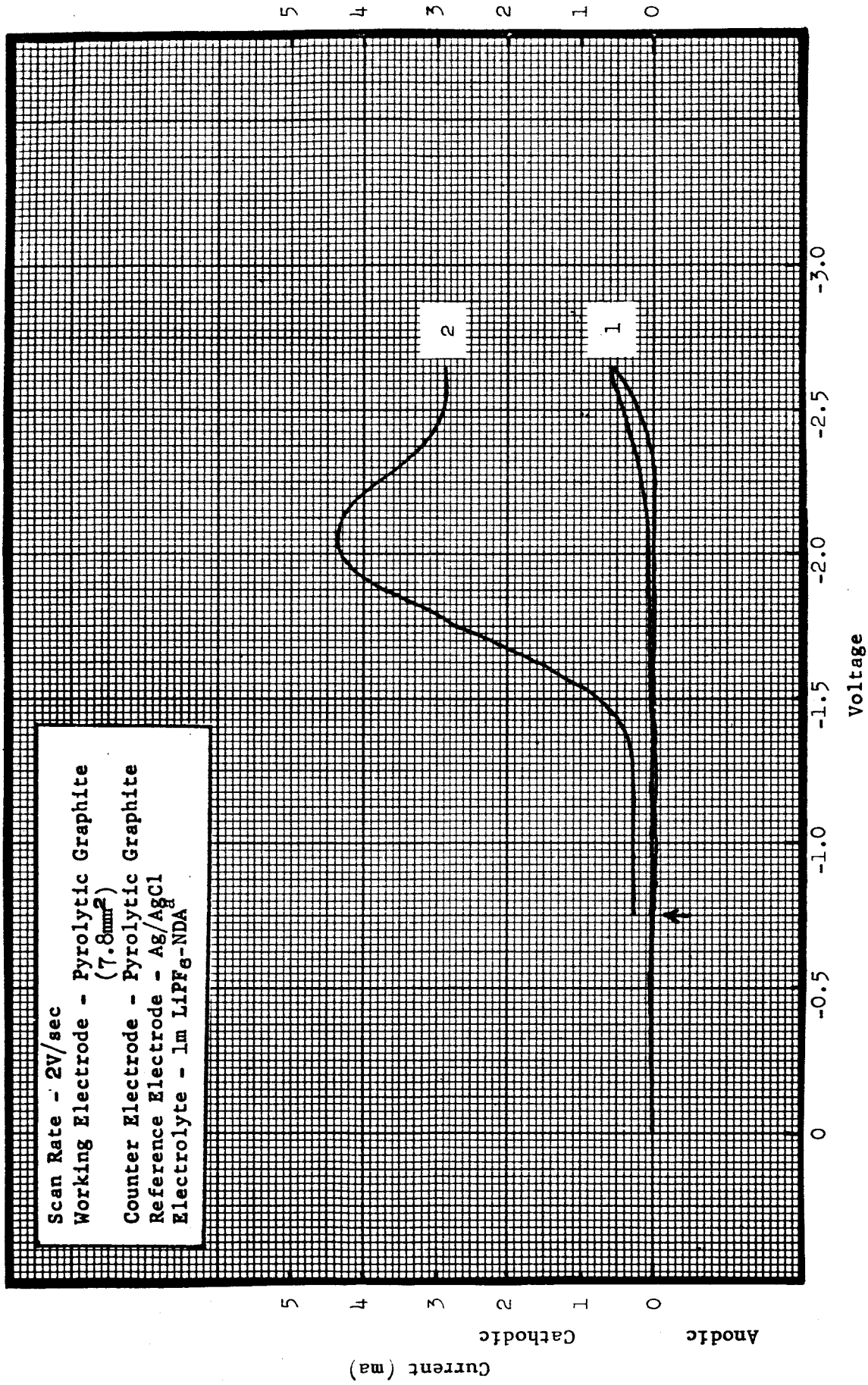


FIGURE 5. VOLTAMMETRIC SWEEP STUDIES OF ELECTROACTIVE IMPURITIES IN LITHIUM HEXAFLUOROPHOSPHATE (Continued)

Figure 5-g. Reduction of the Absorbed Species Found in Lithium Hexafluorophosphate-N-Nitrosodimethylamine. Curve 1 was obtained when the electrode had never been held at potentials more positive than -1.5V (vs. Ag/AgCl). Curve 2 is typical of the scans obtained when the electrode was held at -0.76V (vs. Ag/AgCl) for 5 to 10 seconds. (Voltage indicated by the arrow.)

^a The LiPF₆-NDA was neutralized with an excess of lithium prior to the electrochemical testing.

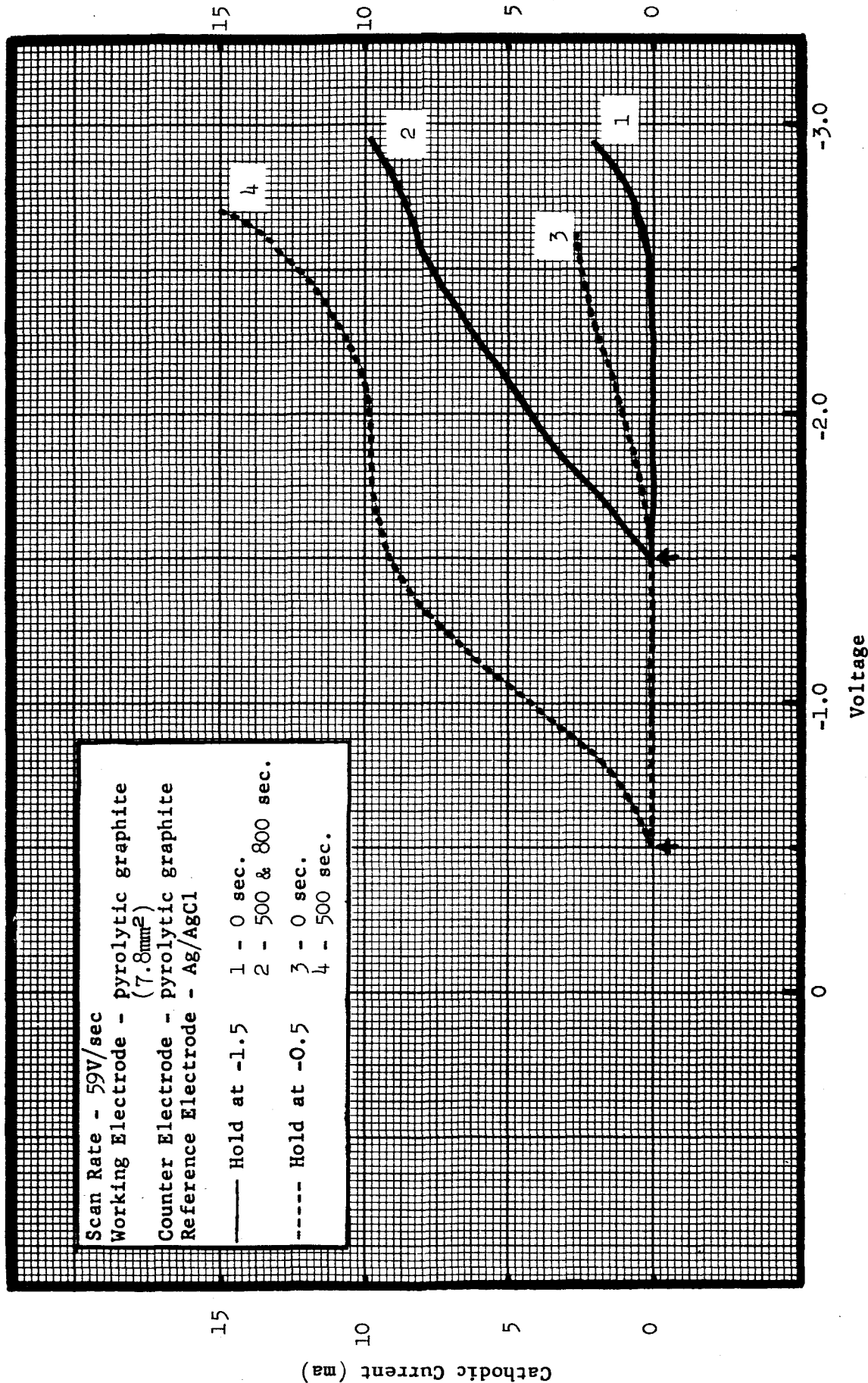


FIGURE 5. VOLTAMMETRIC SWEEP STUDIES OF ELECTROACTIVE IMPURITIES IN LITHIUM HEXAFLUOROPHOSPHATE (Continued)

Figure 5-h. Voltammetric Sweep Studies of the Electroactive Impurity Present in Lithium Hexafluorophosphate-N-Nitrosodimethylamine.

Curves 1 and 3 were obtained by making rapid scans over the voltage ranges indicated. Curves 2 and 4 represent the results obtained when the electrode was held at the potentials indicated by the arrows for the given lengths of time.

^a LiPF₆ used "as received."

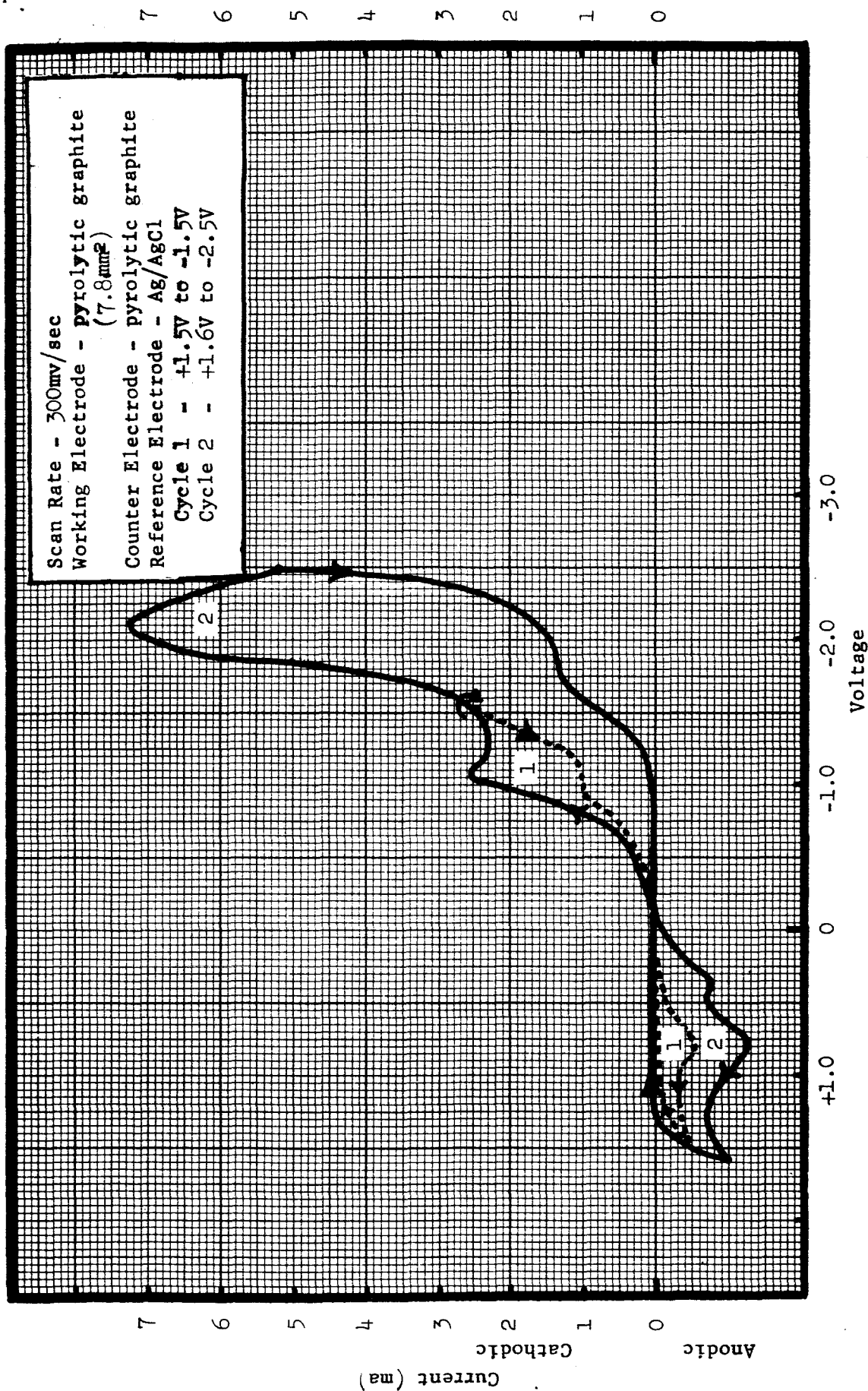


FIGURE 5. VOLTAMMETRIC SWEEP STUDIES OF ELECTROACTIVE IMPURITIES IN LITHIUM HEXAFLUOROPHOSPHATE (Continued)

Figure 5-1. Cyclic Voltammetric Study of the Electroactive Impurities Present in Lithium Hexafluorophosphate^a-N-Nitrosodimethylamine.

The changes in the anodic portion of the scan as a function of increasing reduction are demonstrated by the differences between cycles 1 and 2.

^a LiPF₆ used "as received."

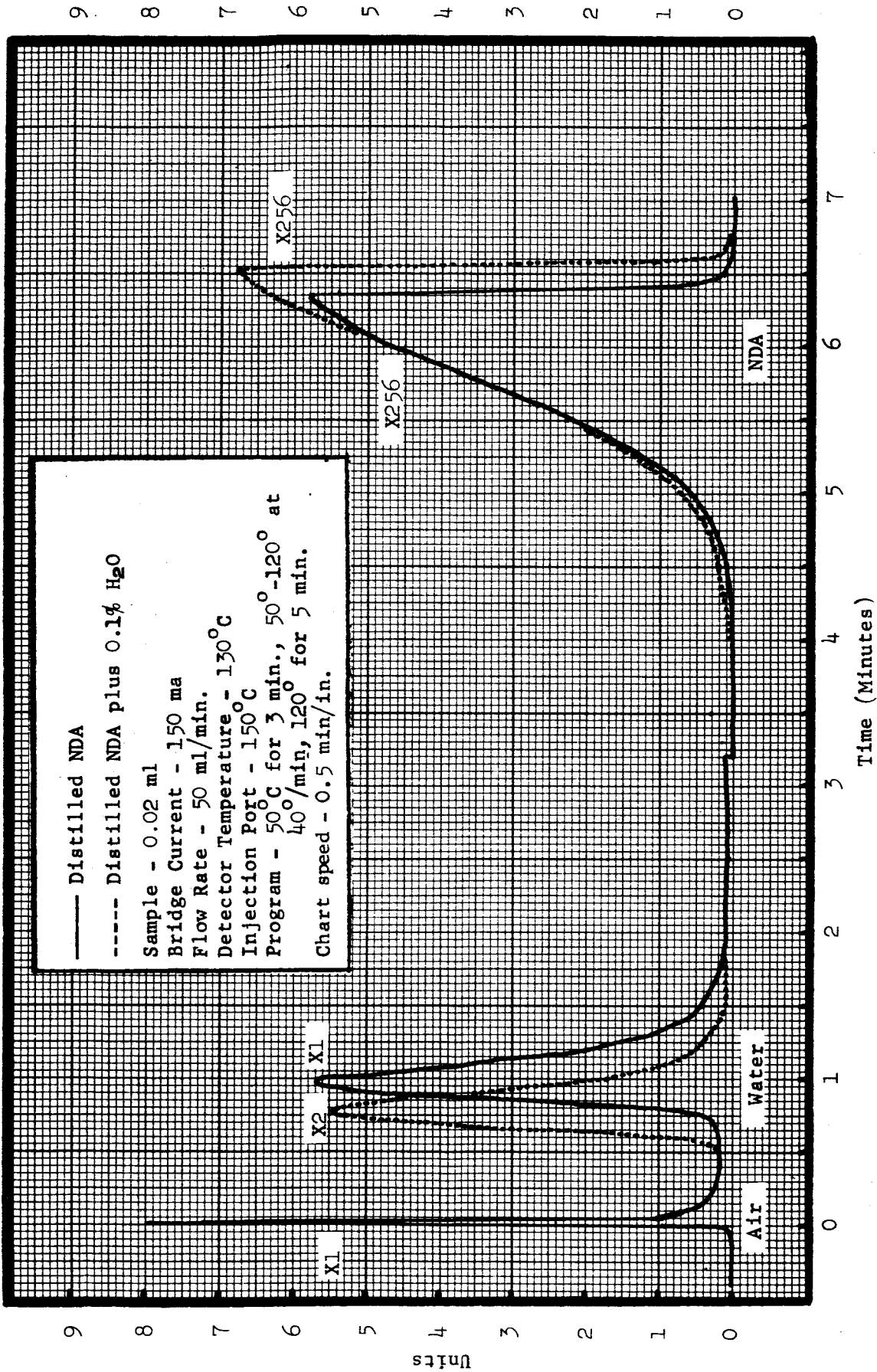


FIGURE 6. GAS PHASE CHROMATOGRAMS OF N-NITROSODIMETHYLAMINE

The column was 1.5 ft. of stainless steel packed with 10% Carbowax 20M on Fluoropak 80. The attenuations used are those shown by each peak.

TABLE V. PRELIMINARY BATTERY TESTS

A. Physical Details

| | Anode | | Cathode | | Electrolyte | | Solvent | | | |
|-----|--------------|------------|----------|---------|---|-------------------------|---------|-----------|--|------------------------------|
| | Material (g) | Weight (g) | Material | Wt. (g) | Composition | Size (cm ²) | | Vol. (ml) | | |
| 1. | Mg | 0.103 | 1 | 0.406 | AGO ^a (90%) | FP ^b (10%) | 1 | 15 | 1m(n-C ₃ H ₇) ₄ NBF ₄ | Acetonitrile (AN) |
| 2. | Mg | 0.100 | 1 | 0.400 | AGO (90%) | FP (10%) | 1 | 15 | 0.5m(n-C ₃ H ₇) ₄ NBF ₄ 0.5m AlCl ₃ | AN |
| 3. | Mg | 0.100 | 1 | 0.405 | AGO (90%) | FP (10%) | 1 | 15 | 1m AlCl ₃ | AN |
| 4. | Mg | 0.103 | 1 | 0.410 | AGO (90%) | FP (10%) | 1 | 15 | 1m LiAlCl ₄ | AN |
| 5. | Mg | 0.102 | 1 | 0.401 | AGO (90%) | FP (10%) | 1 | 15 | 1m Morpholinium hexafluorophosphate | AN |
| 6. | Mg | 0.100 | 1 | 0.393 | AGO (90%) | FP (10%) | 1 | 15 | 1m(n-C ₃ H ₇) ₄ NBF ₄ | AN |
| 7. | Mg | 0.100 | 1 | 0.397 | AGO (90%) | FP (10%) | 1 | 15 | 0.5m(n-C ₃ H ₇) ₄ NBF ₄ 0.5m AlCl ₃ | AN |
| 8. | Mg | 0.100 | 1 | 0.405 | AGO (90%) | FP (10%) | 1 | 15 | 1m AlCl ₃ 0.1m LiPF ₆ ^c | AN |
| 9. | Li | --- | 1 | 0.582 | Ag ₇ O ₈ NO ₃ (100%) | | 1 | 15 | 1mLiPF ₆ ^c | N-nitrosodimethylamine (NDA) |
| 10. | Mg | 0.100 | 1 | 0.398 | CuF ₂ (90%) | FP (5%) | 1 | 15 | 1m LiAlCl ₄ | AN |
| 11. | Mg | 0.100 | 1 | 0.270 | CuCl ₂ (100%) | | 1 | 15 | 1m AlCl ₃ | AN |

^a AGO for all these tests was prepared in our laboratory.

^b FP stands for Filter Paper Pulp.

^c LiPF₆ was used "as received" from the supplier.

TABLE V. PRELIMINARY BATTERY TESTS (Continued)

B. Electrochemical Details.

| Cell | Cell | | Electrolyte | Current Density (ma/cm ²) | Time (Min) | Voltage ^a | |
|------|-------|---------|--|--|---------------|---------------------------|-----------------------------|
| | Anode | Cathode | | | | Anode Vs. Ag/AgCl/Ref. | Cathode Vs. Ag/AgCl/Ref. |
| 1. | Mg | AgO | 1m(n-C ₅ H ₇) ₄ NBF ₄ -AN | OC | 0 | 1.6 | +0.43 |
| | | | | 5 | 0 | 1.1 | 0.0 |
| | | | | 2 | 50 | 0.25 | -0.6 |
| 2. | Mg | AgO | 0.5m(n-C ₅ H ₇) ₄ NBF ₄ -AN 0.5m AlCl ₃ | OC | 0 | 1.9 | +0.06 |
| | | | | 5 | 0 | 1.6 | -0.1 |
| | | | | 10 | 180 | 1.3 | -0.41 |
| 3. | Mg | AgO | 1m AlCl ₃ -AN | OC | 0 | 1.7 | -0.02 |
| | | | | 5 | 0 | 1.5 | -0.21 |
| | | | | 10 | 22 | 1.4 | -0.21 |
| 4. | Mg | AgO | 1m LiAlCl ₄ -AN | OC | 0 | 1.9 | +0.09 |
| | | | | 5 | 0 | 1.2 | -0.4 |
| | | | | 10 | 60 | 0.89 | -0.6 |
| 5. | Mg | AgO | 1m morpholinium hexafluorophos- phate-AN | OC | 0 | 1.3 | +0.09 |
| | | | | 5 | 0 | 1.2 | +0.02 |
| | | | | 10 | 27 | 1.1 | -0.09 |
| 6. | Mg | AgO | 1m(n | OC | 0 | 1.3 | +0.32 |
| | | | | 5 | 0 | 1.1 | -0.01 |
| | | | | 10 | 7 | 1.1 | 0.10 |
| | | | | OC | 0 | 0.7 | -0.43 |
| | | | | 10 | 60 | 0.87 | -0.25 |
| | | | | | 90 | 0.32 | -0.70 |

^a Voltage readings were taken after indicated time had elapsed.

TABLE V. PRELIMINARY BATTERY TESTS (Continued)

B. Electrochemical Details. (Continued)

| Cell | Cell | | Electrolyte | Current Density (ma/cm ²) | Time (Min) | Voltage ^a | |
|------|-------|--|--|--|---------------|---------------------------|-----------------------------|
| | Anode | Cathode | | | | Anode Vs. Ag/AgCl/Ref. | Cathode Vs. Ag/AgCl/Ref. |
| 7. | Mg | AgO | 0.5m(n-C ₃ H ₇) ₄ NBF ₄ -AN | OC | 0 | -1.9 | +0.09 |
| | | | 0.5m AlCl ₃ | 5 | 0 | -1.9 | -0.26 |
| | | | | 10 | 12 | -1.9 | -0.6 |
| | | | | 10 | 0 | -1.8 | -0.7 |
| | | | | | 60 | -1.8 | -1.1 |
| | | | | | 120 | -1.7 | -1.3 |
| | | | | | 180 | -1.7 | -1.4 |
| 8. | Mg | AgO | 1m AlCl ₃ -AN | 0 | 0 | -1.9 | +0.9 |
| | | | 0.1m LiPF ₆ ^b | 5 | 0 | -1.75 | -0.5 |
| | | | | 4.5 | 30 | -1.79 | -0.5 |
| | | | | 4.5 | 75 | -1.68 | -1.57 |
| 9. | Li | Ag ₂ O ₈ NO ₃ | 1m LiPF ₆ ^b -NDA | OC | 0 | -3.4 | +0.28 |
| | | | | 5 | 0 | -3.3 | +0.19 |
| | | | | 10 | 10 | -3.3 | +0.12 |
| | | | | 10 | 0 | -3.2 | +0.06 |
| | | | | 60 | 60 | -3.2 | -0.16 |
| | | | | 120 | 2.9 | -3.2 | -0.38 |
| | | | | 180 | 2.2 | -3.2 | -1.0 |
| | | | | 240 | 1.8 | -3.1 | -1.3 |
| | | | | 300 | 1.5 | -2.9 | -1.3 |
| | | | | 360 | 1.2 | -2.2 | -1.0 |
| | | | | 420 | 0.6 | -2.2 | -1.6 |
| | | | | 480 | 1.1 | -2.7 | -1.7 |
| | | | | 540 | 1.4 | -2.7 | -1.2 |
| | | | | 660 | 1.1 | -2.6 | -1.2 |
| | 720 | 0.9 | -2.5 | -1.4 | | | |
| | 780 | 0.8 | -2.3 | -1.4 | | | |
| | 840 | 0.7 | -2.1 | -1.3 | | | |
| | 900 | 0.4 | -2.1 | -1.5 | | | |
| | 960 | 0.1 | -2.1 | -1.9 | | | |

^a Voltage readings were taken after indicated time had elapsed.

^b LiPF₆ was used "as received" from the supplier.

TABLE V. PRELIMINARY BATTERY TESTS (Continued)

B. Electrochemical Details. (continued)

| | Cell | | Electrolyte | Current Density (ma/cm ²) | Time (Min) | Cell | Voltage ^a | |
|-----|-------|-------------------|----------------------------|--|---------------|------|---------------------------|-----------------------------|
| | Anode | Cathode | | | | | Anode Vs. Ag/AgCl/Ref. | Cathode Vs. Ag/AgCl/Ref. |
| 10. | Mg | CuF ₂ | 1m LiAlCl ₄ -AN | OC | 0 | 1.5 | -1.8 | -0.33 |
| | | | | 5 | 0 | 1.1 | -1.6 | -0.5 |
| | | | | | 65 | 0.19 | -1.7 | -1.6 |
| 11. | Mg | CuCl ₂ | 1m AlCl ₃ -AN | OC | 0 | 1.4 | -1.7 | -0.46 |
| | | | | 5 | 0 | 1.2 | -1.7 | -0.6 |
| | | | | 10 | 10 | 1.2 | -1.7 | -0.6 |
| | | | | 10 | 10 | 1.1 | -1.7 | -0.7 |
| | | | | 66 | 66 | 1.2 | -1.7 | -0.7 |
| | | | | 106 | 106 | 1.1 | -1.7 | -0.7 |
| | | | | 139 | 139 | 1.0 | -1.7 | -0.7 |
| | | | | 150 | 150 | 0.9 | -1.7 | -0.8 |
| | | | | 177 | 177 | 0.8 | -1.7 | -0.9 |
| | | | | 186 | 186 | 0.7 | -1.7 | -1.0 |
| | | | | 193 | 193 | 0.7 | -1.7 | -1.1 |
| | | | | 298 | 298 | 0.6 | -1.7 | -1.1 |

^a Voltage readings were taken after indicated time had elapsed.

TABLE VI. CONSTANT CURRENT DISCHARGES OF TEST BATTERIES

| | <u>Code</u> |
|----|---------------------|
| SF | - Solka-Floc |
| FP | - Filter Paper Pulp |
| BM | - Ball Milled |
| TM | - Tumbled |
| BL | - Blended |

TABLE VI. CONSTANT CURRENT DISCHARGES OF TEST BATTERIES^a

VI-a. Test Batteries With Silver Oxide (AgO) Cathodes.

| 1. | Li Anode Wt. (g) | AH | Composition (Wt. %) | | | Prep. of Mix | Wt. (g) | AH | Cell Wt. (g) | Current Density (ma/cm ²) | Watt-Hour Per Pound | | Total Weight to 1.5V |
|----|---------------------|------|---------------------|--------|----|--------------|---------|------|--------------|---------------------------------------|---------------------|---------|----------------------|
| | | | AgO | Binder | SF | | | | | | Active Mat. to 2V | to 1.5V | |
| | | | | | | | | | | | | | |
| 1. | 0.85C | 3.28 | 80 | 20 | | BM | 3.23 | 0.75 | 0.5 | 36.2 | 41.4 | 12.8 | 14.6 |
| | | | | SF | | | 3.19 | 0.74 | | | | | |
| 2. | 0.836 | 3.23 | 90 | 10 | | BL | 3.72 | 0.86 | 0.5 | 119 | 170 | 53.1 | 76.2 |
| | | | | FP | | | 3.71 | 0.86 | | | | | |
| 3. | 0.851 | 3.29 | 95 | 5 | | BL | 3.59 | 0.82 | 0.5 | 100 | 141 | 45.7 | 64.1 |
| | | | | FP | | | 3.66 | 0.85 | | | | | |
| 4. | 0.896 | 3.46 | 90 | 10 | | TM | 3.70 | 1.60 | 0.5 | 210 | 233 | 91.0 | 101.0 |
| | | | | FP | | | 3.66 | 1.58 | | | | | |
| 5. | 0.900 | 3.48 | 80 | 20 | | TM | 3.71 | 1.60 | 0.5 | 209 | 234 | 85.9 | 96.0 |
| | | | | FP | | | 3.74 | 1.62 | | | | | |
| 6. | 0.905 | 3.49 | 95 | 5 | | TM | 3.71 | 1.60 | 0.5 | 193 | 218 | 87.8 | 99.8 |
| | | | | FP | | | 3.71 | 1.60 | | | | | |
| 7. | 0.906 | 3.50 | 90 | 10 | | BL | 3.88 | 1.68 | 0.5 | 225 | 243 | 100 | 108 |
| | | | | FP | | | 3.82 | 1.65 | | | | | |
| 8. | 0.846 | 3.27 | 90 | 10 | | BL | 3.81 | 1.65 | 0.5 | 147 | 175 | 65.1 | 77.8 |
| | | | | FP | | | 3.82 | 1.65 | | | | | |
| 9. | 1.01 | 3.89 | 90 | 10 | | BL | 3.88 | 1.68 | 0.75 | 172 | 200 | 92.8 | 108 |
| | | | | FP | | | 4.11 | 1.78 | (23 Hrs) | | | | |
| | | | | | | | | | 1.50 | | | | |
| | | | | | | | | | (12.5 Hrs) | | | | |

^a These batteries all contained three electrodes (2" x 2"). The AH figures indicated whether these were two anodes or two cathodes. The electrolyte was 1m LiPF₆(as received)-N-nitrosodimethylamine unless otherwise stated. The separators were microporous glass. The cathode matrix was silver expanded metal.

^b In tests 1 through 3, the silver oxide used was obtained commercially. The AgO for all other tests was prepared in this laboratory according to the method given in Inorganic Synthesis, Volume 4, page 12.

^c Battery was assembled in activated condition and allowed to stand for 5 days before discharging it.

TABLE VI. CONSTANT CURRENT DISCHARGES OF TEST BATTERIES^a (Continued)

| VI-a. Test Batteries With Silver Oxide (AgO) Cathodes. (Continued) | | AgO Cathode ^b | | | | | | | | | | |
|--|--------------|--------------------------|------------------------|-----------------|--------------|--------------|--------------------|---|----------------------|---------|-------------------------------|-------------------|
| Li Anode Wt. (g) | AH | Composition (Wt. %) | | Prep. of Mix | Wt. (g) | AH | Cell Wt. (g) | Current Density (ma/cm ²) | Watt-Hour Per Pound | | Total Weight to 2V to 1.5V | |
| | | AGO | Binder | | | | | | Active Mat. to 2V | to 1.5V | | |
| 10. 1.03 | 3.98 | 90 | 10 FP | BL | 3.72 3.84 | 1.61 1.66 | 16.4 | 2.5 | 32.2 | 49.7 | 16.9 | 26.1 |
| 11. 1.01 | 3.89 | 85.7 | 9.5 ^g FP | BL | 3.72 3.31 | 1.61 1.43 | 17.9 | 0.75 | 105 | 137 | 47.7 | 62 |
| 12. 1.04 | 4.00 | 90 | 5 ^g FP | BL | 3.75 3.78 | 1.62 1.63 | 16.8 | 1.5 | 131 | 144 | 67.8 | 74.9 |
| 13 ^e . 0.999 | 3.86 | 90 | 10 SF | BL | 3.59 3.58 | 1.56 1.55 | 17.0 | 1 | 147.6 | 154 | 72.2 | 75.4 ^f |
| 14 ^e . 0.853 | 3.30 | 92 | 5 ^g FP | BL | 3.76 3.70 | 1.63 1.60 | 17.5 | 5 | 58.7 | 102.6 | 27.8 | 48.7 |
| 15 ^e . 0.837 | 3.23 | 90 | 10 FP | BL | 3.75 3.76 | 1.62 1.63 | 17.0 | 10(11 min) 5(114 min) | 29.6 | 63.0 | 14.7 | 31.4 |
| 16 ^h . 0.910 | 3.52 | 90 | 10 FP | BL | 3.75 3.74 | 1.62 1.62 | 18.8 | 10 | 29.3 | 95.2 | 12.9 | 41.8 |
| 17 ^h . 0.972 0.993 | 3.75 3.83 | 90 | 10 FP | BL | 7.50 | 3.25 | 18.5 | 5 | 25.2 | 45.4 | 13.3 | 23.9 |

^a These batteries all contained three electrodes (2" x 2"). The AH figures indicated whether these were two anodes or two cathodes. The electrolyte was 1M LiPF₆ (as received)-N-nitrosodimethylamine unless otherwise stated. The separators were microporous glass. The cathode matrix was silver expanded metal.

^b In tests 1 through 3, the silver oxide used was obtained commercially. The AgO for all other tests was prepared in this laboratory according to the method given in Inorganic Synthesis, Volume 4, page 12.

^d LiPF₆ solute vacuum-dried.

^e Electrolyte reacted with electrodes causing heat evolution.

^f Test terminated when cell voltage reached 1.8V.

^g The balance of the mixture was made up with acetylene black.

^h Electrolyte pretreated with lithium before use in batteries.

ⁱ The circuit was opened several times during the discharge.

TABLE VI. CONSTANT CURRENT DISCHARGES OF TEST BATTERIES^a (Continued)

| VI-a. Test Batteries With Silver Oxide (AgO) Cathodes. (Continued) | | Li Anode | | Composition (Wt. %) | | Prep. of Mix | | Cell Wt. (g) | Current Density (ma/cm ²) | Watt-Hour Per Pound | | |
|--|-------|----------|--------|---------------------|-----------------|--------------|------|--------------|---------------------------------------|---------------------|----------------------|-------|
| Wt. (g) | AH | AgO | Binder | AgO | Binder | Wt. (g) | AH | (g) | (ma/cm ²) | Active Mat. to 2V | Total Weight to 1.5V | |
| 18. | 0.939 | 3.63 | | 90 | 10 | BL | 7.53 | 3.26 | 17.4 | 1 | 125.2 | 69.9 |
| | 0.950 | 3.67 | | | FP | | | | | | | j |
| 19. ^d | 0.981 | 3.79 | | 95 | 5 | BL | 7.48 | 3.24 | 17.2 | 5 | 23.8 | 46.0 |
| | 0.994 | 3.84 | | | FP | | | | | | | 13.4 |
| 20. | 0.847 | 3.27 | | 95 | 5 | BL | 3.79 | 1.64 | 16.4 | 5 | 69.0 | 103.8 |
| | | | | | FP | | 3.72 | 1.61 | | | | 35.1 |
| 21. ^d | 0.929 | 3.59 | | 90 | 10 | BL | 7.52 | 3.25 | 17.7 | 10(11 min) | 14.4 | 29.1 |
| | 0.976 | 3.77 | | | FP | | | | | 5(39 min) | | 7.8 |
| 22. | 0.813 | 3.14 | | 87 | 10 ^g | BL | 3.79 | 1.64 | 17.6 | 5 | 104.1 | 171.1 |
| | | | | | FP | | 3.77 | 1.63 | | | | 49.5 |
| 23. | 0.844 | 3.28 | | 90 | 10 | BL | 3.77 | 1.63 | 16.6 | 5 | 11.0 | 144.1 |
| | | | | | FP | | 3.77 | 1.63 | | | | 5.5 |
| 24. ^k | 0.862 | 3.35 | | 87 | 10 ^g | BL | 3.86 | 1.67 | 18.8 | 5 | 5.3 | 8.9 |
| | | | | | FP | | 3.79 | 1.64 | | | | 2.4 |
| 25. | 0.916 | 3.56 | | 80 | 20 | BL | 3.77 | 1.63 | 17.9 | 5 | 57.0 | 90.2 |
| | | | | | FP | | 3.79 | 1.64 | | | | 27.0 |

^a These batteries all contained three electrodes (2" x 2"), two cathodes and one anode. The electrolyte was 1m LiPF₆(as received)-N-nitrosodimethylamine. The separators were microporous glass. The cathode matrix was silver expanded metal.

^b In tests 1 through 4, the silver oxide used was obtained commercially. The AgO for all other tests was prepared in this laboratory according to the method given in Inorganic Synthesis, Volume 4, page 12.

^d LiPF₆ solute vacuum-dried.

^g The balance of the mixture was made up with acetylene black.

^j Test terminated when cell voltage reached 2.0V.

^k The electrolyte in this test was LiClO₄(as received)-propylene carbonate.

TABLE VI. CONSTANT CURRENT DISCHARGES OF TEST BATTERIES^a (Continued)

| VI-a. Test Batteries With Silver Oxide (AGO) Cathodes. (Continued) | | AGO Cathode ^b | | | | | | | | | | |
|--|------|--------------------------|-----------------------|-----------------|--------------|--------------|--------------------|---|----------------------|-------------------------|-------|------|
| Li Anode Wt. (g) | AH | Composition (Wt. %) | | Prep. of Mix | Wt. (g) | AH | Cell Wt. (g) | Current Density (ma/cm ²) | Watt-Hour Per Pound | | | |
| | | AGO | Binder | | | | | | Active Mat. to 2V | Total Weight to 1.5V | | |
| 26. 0.838 | 3.26 | 77 | 20 ^g FP | BL | 3.80 3.78 | 1.64 1.64 | 18.9 | 5 | 99.5 | 154.6 | 44.4 | 68.8 |
| 27. 0.866 | 3.36 | 87 | 8 ^g FP | BL | 3.82 3.85 | 1.65 1.67 | 18.7 | 5 | 31.1 | 146.0 | 14.2 | 66.7 |
| 28 ^h 0.999 | 3.86 | 90 | 10 FP | BL | 3.59 3.58 | 1.55 1.55 | 17.0 | 1 | 147.6 | 154 | 72.2 | 75.4 |
| 29. 0.972 | 3.75 | 90 | 10 FP | BL | 3.60 3.59 | 1.56 1.55 | 17.5 | 1 | 32.8 | 69.3 | 15.5 | 32.8 |
| 30. 0.901 | 3.48 | 90 | 10 FP | BL | 3.60 3.61 | 1.56 1.56 | 16.95 | 1 | ----- | 21.7 | ----- | 10.6 |
| 31. 0.962 | 3.71 | 90 | 10 FP | BL | 3.61 3.62 | 1.56 1.57 | 17.3 | 1 | ----- | 20.6 | ----- | 9.9 |

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^a These batteries all contained three electrodes (2" x 2"). The AH figures indicated whether these were two anodes or two cathodes. The electrolyte was 1M LiPF₆ (as received)-N-nitrosodimethylamine unless otherwise stated. The separators were microporous glass. The cathode matrix was silver expanded metal.

^b In tests 1 through 3, the silver oxide used was obtained commercially. The AGO for all other tests was prepared in this laboratory according to the method given in Inorganic Synthesis, Volume 4, page 12.

^g The balance of the mixture was made up with acetylene black.

^h Cell numbers 28 through 31 were assembled at the same time. Cell 29 was discharged after one week; cell 30 after two weeks; and cell 31 after three weeks.

TABLE VI. CONSTANT CURRENT DISCHARGES OF TEST BATTERIES^a

VI-b. Test Batteries with Cupric Fluoride Cathodes

| | Li Anode | | CuF ₂ Cathode | | | | Cell Wt. (g) | Current Density (ma/cm ²) | Watt-Hour Per Pound | | | | | |
|-----|----------|------|--------------------------|--------|--------------|---------|--------------|---------------------------------------|---------------------|-------------|-------|---------|------|------|
| | Wt. (g) | AH | Composition (Wt. %) | | Prep. of Mix | Wt. (g) | | | AH | Active Mat. | | | | |
| | | | CuF ₂ | Binder | | | | | | AB | to 2V | to 1.5V | | |
| 1. | 0.856 | 3.38 | 94 | 3 SF | 3 | BM | 5.46 | 2.88 | 24.1 | 0.5 | 13.7 | 20.0 | 3.6 | 5.2 |
| 2. | 0.848 | 3.28 | 94 | 3 SF | 3 | BM | 4.77 | 2.52 | 22.3 | 0.5 | b | b | b | b |
| 3. | 0.857 | 3.31 | 94 | 3 SF | 3 | BM | 6.14 | 3.24 | 16.6 | 0.5 | 54.1 | --- | 22.8 | --- |
| 4. | 0.916 | 3.54 | 90 | 5 FP | 5 | BL | 6.05 | 3.19 | 18.4 | 0.5 | 156 | 189 | 58.9 | 71.7 |
| 5. | 0.910 | 3.51 | 85 | 10 FP | 5 | BL | 5.94 | 3.13 | 18.3 | 0.5 | 129 | 167 | 48.3 | 62.4 |
| 6. | 0.924 | 3.57 | 90 | 5 FP | 5 | BM | 6.37 | 3.36 | 17.7 | 0.5 | 78.4 | 101 | 32.4 | 41.7 |
| 7. | 0.901 | 3.48 | 75 | 20 FP | 5 | BL | 6.16 | 3.25 | 19.8 | 0.5 | 108 | 125 | 38.5 | 44.5 |
| 8. | 0.903 | 3.49 | 85 | 10 FP | 5 | BM | 6.08 | 3.21 | 18.1 | 0.5 | 152 | 179 | 58.4 | 69.0 |
| 9. | 1.03 | 3.99 | 94 | 3 SF | 3 | BM | 5.69 | 3.00 | 14.6 | 0.75 | 14.8 | 18.2 | 6.6 | 8.1 |
| 10. | 1.04 | 4.00 | 90 | 5 FP | 5 | BM | 7.59 | 4.01 | 16.7 | 0.8 | 103 | 136 | 53.1 | 70.0 |

^a These batteries all contained three electrodes (2" x 2"), two cathodes and one anode. The electrolyte was 1m LiPF₆(as received)-N-nitrosodimethylamine. The separators were microporous glass. The cathode matrix was copper expanded metal.

^b Cell was placed on open circuit several times; therefore, watt-hours per pound were not calculated.

^c A silver expanded metal matrix was used.

TABLE VII. 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 ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure | | | |
|-------------------|--------------------|------------------------------------|----------------------------------|---|---------------------------------|--|---------------------------------|---------------------------------|--|
| | | | | Exposed Electrolyte | Blank Electrolyte | | | | |
| 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 visible changes occurred. | | | |
| | | 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) | | | | |
| | | After Four Weeks | | 1.38×10^{-4} (29°C) | 1.65×10^{-4} (29°C) | | | | |
| | | After Nine Weeks | | 1.66×10^{-4} (26°C) | 1.77×10^{-4} (26°C) | | | | |
| | | After Fourteen Weeks | | 1.62×10^{-4} (27°C) | 1.97×10^{-4} (27°C) | | | | |
| | | After Fifteen Weeks | | 1.65×10^{-4} (25°C) | 2.01×10^{-4} (25°C) | | | | |
| | | After Sixteen Weeks | | 1.69×10^{-4} (25°C) | 2.11×10^{-4} (25°C) | | | | |
| | | After Seventeen Weeks | | 1.70×10^{-4} (23°C) | 2.05×10^{-4} (23°C) | | | | |
| | | After Eighteen Weeks | 0.1840 | 1.71×10^{-4} (24°C) | 2.07×10^{-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. After fourteen weeks a white precipitate had formed. No further visible changes occurred. |
| | | | | 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) | | | | |
| After Four Weeks | | | | 1.94×10^{-2} (29°C) | 2.06×10^{-2} (29°C) | | | | |

TABLE VII. 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 ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|--------------------|--------------------|---------------------------------|----------------------------------|---|---------------------------------|--|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 2. (Continued) | Li | After Nine Weeks | | 1.97×10^{-2} (26°C) | 2.04×10^{-2} (26°C) | |
| | | After Fourteen Weeks | | 2.03×10^{-2} (27°C) | 1.59×10^{-2} (27°C) | |
| | | After Fifteen Weeks | | 1.87×10^{-2} (25°C) | 2.02×10^{-2} (25°C) | |
| | | After Sixteen Weeks | | 1.92×10^{-2} (25°C) | 2.03×10^{-2} (25°C) | |
| | | After Seventeen Weeks | | 1.94×10^{-2} (23°C) | 2.04×10^{-2} (23°C) | |
| | | After Eighteen Weeks | 0.2230 | 1.96×10^{-2} (24°C) | 1.86×10^{-2} (24°C) | |
| | | 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. After fourteen weeks a white precipitate had formed. No further visible changes occurred. |
| | | 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) | |
| After Four Weeks | | 1.85×10^{-2} (29°C) | 1.93×10^{-2} (29°C) | | | |
| 3. LiPF_6 | Li | After Nine Weeks | | 1.82×10^{-2} (26°C) | 1.92×10^{-2} (29°C) | |
| | | After Fourteen Weeks | | 1.87×10^{-2} (29°C) | 1.96×10^{-2} (28°C) | |
| | | After Fifteen Weeks | | 1.81×10^{-2} (25°C) | 1.80×10^{-2} (25°C) | |

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

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

A. Stability in N-Nitrosodimethylamine Solutions. (Continued)

| Solute | Reference Material | Time of Measurement | Weight of Electrode Material (g) | Specific Conductance ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure | |
|------------------------------------|--------------------|---------------------------------|----------------------------------|---|---------------------------------|--|---|
| | | | | Exposed Electrolyte | Blank Electrolyte | | |
| 3. (Continued) | Li | After Sixteen Weeks | | 1.80×10^{-2} (25°C) | 1.78×10^{-2} (25°C) | | |
| | | After Seventeen Weeks | | 1.84×10^{-2} (23°C) | 1.78×10^{-2} (23°C) | | |
| | | After Eighteen Weeks | 0.2384 | 1.76×10^{-2} (24°C) | 1.71×10^{-2} (24°C) | | |
| 4. None | CuF ₂ | Initial | 0.4576 | 1.05×10^{-4} (24°C) | 1.08×10^{-4} (24°C) | The cathode pellet turned green immediately. No further visible change occurred. | |
| | | 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) | | |
| | | After Four Weeks | | 2.21×10^{-4} (29°C) | 1.65×10^{-4} (29°C) | | |
| | | After Nine Weeks | | 2.59×10^{-4} (26°C) | 1.72×10^{-4} (26°C) | | |
| | | After Fourteen Weeks | 0.4685 | 2.79×10^{-4} (27°C) | 1.97×10^{-4} (27°C) | | |
| | | Initial | 0.3179 | 1.98×10^{-2} (24°C) | 2.03×10^{-2} (24°C) | | The electrolyte changed from yellow to green after two days. After fourteen weeks a dark green precipitate had formed. No further visible changes occurred. |
| | | 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} (24°C) | | | | |
| After Four Weeks | | 2.13×10^{-2} (29°C) | 2.06×10^{-2} (29°C) | | | | |
| 5. LiPF ₆ (as received) | CuF ₂ | | | | | | |

TABLE VII. 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 ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|----------------------|--------------------|----------------------|----------------------------------|---|---------------------------------|---|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 5. (Continued) | CuF ₂ | After Nine Weeks | | 1.97×10^{-2} (26°C) | 2.04×10^{-2} (26°C) | |
| | | After Fourteen Weeks | 0.1461 | 1.88×10^{-2} (27°C) | 1.59×10^{-2} (27°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. After fourteen weeks a blue-green precipitate had formed. No further visible changes occurred. |
| | | 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) | |
| | | After Four Weeks | | 1.93×10^{-2} (29°C) | 1.93×10^{-2} (29°C) | |
| | | After Nine Weeks | | 1.88×10^{-2} (26°C) | 1.92×10^{-2} (26°C) | |
| | | After Fourteen Weeks | 0.1942 | 1.72×10^{-2} (27°C) | 1.96×10^{-2} (28°C) | |

^a The LiPF₆ was dried at 110°C in a nitrogen atmosphere before use.

TABLE VII. 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 ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|----------------------------------|------------------------------------|---------------------|--|---|------------------------------|--|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 7. LiPF_6 (as received) | CuF_2 & Li (2 electrodes) | Initial | 0.2372 ^c (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. |
| | | After One Day | 0.0691 (Li) | 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) | |
| | | | | | | |
| 8. LiPF_6 ^b | CuF_2 & Li (2 electrodes) | Initial | 0.2523 ^c (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. |
| | | After One Day | 0.0643 (Li) | 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) | |
| | | | | | | |

^b The LiPF_6 was dried at 110°C in a nitrogen atmosphere before use.

^c Includes substrate weight.

TABLE VII. 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 ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|----------------------------------|--------------------|---------------------------------|----------------------------------|---|---------------------------------|--|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 9. None | AgO | Initial | 0.3591 | 7.96×10^{-5} (29°C) | 8.03×10^{-5} (29°C) | The electrolyte changed from yellow to green after four weeks. After ten weeks a dark precipitate had formed and the cathode pellet was disintegrating. No further visible changes occurred. |
| | | 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) | |
| | | After Five Weeks | | 1.58×10^{-4} (29°C) | 1.24×10^{-4} (29°C) | |
| | | After Six Weeks | | 1.68×10^{-4} (28°C) | 1.24×10^{-4} (28°C) | |
| | | After Seven Weeks | | 1.72×10^{-4} (27°C) | 1.36×10^{-4} (28°C) | |
| | | After Eight Weeks | | 1.85×10^{-4} (26°C) | 1.25×10^{-4} (25°C) | |
| | | After Nine Weeks | | 1.96×10^{-4} (25°C) | 1.25×10^{-4} (25°C) | |
| | | After Ten Weeks | | 2.03×10^{-4} (26°C) | 1.26×10^{-4} (26°C) | |
| | | After Eleven Weeks | | 2.06×10^{-4} (25°C) | 1.30×10^{-4} (25°C) | |
| | | After Twelve Weeks | | 2.20×10^{-4} (27°C) | 1.47×10^{-4} (27°C) | |
| | | After Thirteen Weeks | | 2.26×10^{-4} (26°C) | 1.39×10^{-4} (26°C) | |
| After Fourteen Weeks | | 2.24×10^{-4} (22°C) | 1.50×10^{-4} (22°C) | | | |
| After Fifteen Weeks ^b | | 2.44×10^{-4} (23°C) | 1.48×10^{-4} (23°C) | | | |

^b Weight change of AgO cannot be calculated because pellet disintegrated.

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

A. Stability in N-Nitrosodimethylamine Solutions. (Continued)

| Solute | Material | Time of Measurement | Weight of Electrode Material (g) | Specific Conductance ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|-----------------------------------|----------|---------------------------------|----------------------------------|---|---------------------------------|--|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 10. 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 after two weeks. |
| | | 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) | |
| | | After Five Weeks | | 2.08×10^{-2} (29°C) | 2.06×10^{-2} (29°C) | |
| | | After Six Weeks | | 2.07×10^{-2} (28°C) | 2.03×10^{-2} (28°C) | |
| | | After Seven Weeks | | 2.00×10^{-2} (26°C) | 1.87×10^{-2} (26°C) | |
| | | After Eight Weeks | | 2.05×10^{-2} (26°C) | 2.00×10^{-2} (26°C) | |
| | | After Nine Weeks | | 2.05×10^{-2} (26°C) | 1.90×10^{-2} (26°C) | |
| | | After Ten Weeks | | 2.05×10^{-2} (26°C) | 1.90×10^{-2} (26°C) | |
| | | After Eleven Weeks | | 1.96×10^{-2} (25°C) | 1.94×10^{-2} (25°C) | |
| | | After Twelve Weeks | | 1.98×10^{-2} (27°C) | 2.02×10^{-2} (27°C) | |
| | | After Thirteen Weeks | | 2.01×10^{-2} (26°C) | 1.89×10^{-2} (26°C) | |
| After Fourteen Weeks | | 1.88×10^{-2} (22°C) | 1.59×10^{-2} (22°C) | | | |
| After Fifteen Weeks ^b | | 1.82×10^{-2} (23°C) | 1.88×10^{-2} (23°C) | | | |

^b Weight change of AgO cannot be calculated because pellet disintegrated.

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

A. Stability in N-Nitrosodimethylamine Solutions. (Continued)

| Solute | Reference Electrode | Weight of Electrode Material (g) | Time of Measurement | Specific Conductance ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|------------------------------------|---------------------------------|----------------------------------|---------------------|---|---------------------------------|--|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 11. LiPF ₆ ^a | AgO | 0.4071 | Initial | 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 visible change occurred. |
| | | | 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) | |
| | | | After Five Weeks | 1.91×10^{-2} (29°C) | 1.93×10^{-2} (29°C) | |
| | | | After Six Weeks | 1.97×10^{-2} (28°C) | 1.42×10^{-2} (28°C) | |
| | | | After Seven Weeks | 1.97×10^{-2} (27°C) | 1.82×10^{-2} (27°C) | |
| | | | After Eight Weeks | 1.94×10^{-2} (26°C) | 1.88×10^{-2} (26°C) | |
| | | | After Nine Weeks | 1.80×10^{-2} (26°C) | 1.91×10^{-2} (25°C) | |
| | | | After Ten Weeks | 1.93×10^{-2} (26°C) | 1.95×10^{-2} (26°C) | |
| | | | After Eleven Weeks | 1.88×10^{-2} (25°C) | 1.90×10^{-2} (25°C) | |
| After Twelve Weeks | 1.95×10^{-2} (27°C) | 1.85×10^{-2} (27°C) | | | | |
| After Thirteen Weeks | 2.01×10^{-2} (26°C) | 1.88×10^{-2} (26°C) | | | | |

^a The LiPF₆ was dried at 110°C in a nitrogen atmosphere before use.

TABLE VII. 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 ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|-----------------|-----------------------------|----------------------------------|-----------------------------------|---|---------------------------------|---|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 11. (Continued) | AGO | After Fourteen Weeks | | 1.86×10^{-2} (22°C) | 1.79×10^{-2} (22°C) | |
| | | After Fifteen Weeks ^b | | 1.81×10^{-2} (23°C) | 1.82×10^{-2} (23°C) | |
| 12. None | AGO & Li (2 elec-trodes) | Initial | 0.3237 (AGO) 0.0756 (Li) | 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. After ten weeks a dark precipitate had formed. No further visible change occurred. |
| | | 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) | |
| | | After Five Weeks | | 1.45×10^{-4} (29°C) | 1.24×10^{-4} (29°C) | |
| | | After Six Weeks | 0.0554 ^c (Li) | 1.46×10^{-4} (28°C) | 1.24×10^{-4} (28°C) | |
| | | After Seven Weeks | | 1.52×10^{-4} (27°C) | 1.36×10^{-4} (28°C) | |
| | | After Eight Weeks | | 1.56×10^{-4} (26°C) | 1.25×10^{-4} (25°C) | |

^b Weight change of AGO cannot be calculated because pellet disintegrated.

^c Lithium was not visibly attacked up to this time. However, between tests it apparently worked loose from the connecting clip and dropped into solution. To avoid disturbing the test excessively, a new piece of lithium was used.

TABLE VII. 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 ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|--|-----------------------------|----------------------|-----------------------------------|---|---------------------------------|---|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 12. (Continued) AgO & Li (2 elec-trodes) | AgO & Li (2 elec-trodes) | After Nine Weeks | | 1.60×10^{-4} (26°C) | 1.25×10^{-4} (25°C) | |
| | | After Ten Weeks | | 1.66×10^{-4} (26°C) | 1.26×10^{-4} (26°C) | |
| | | After Eleven Weeks | | 1.76×10^{-4} (25°C) | 1.30×10^{-4} (25°C) | |
| | | After Twelve Weeks | | 1.80×10^{-4} (27°C) | 1.47×10^{-4} (27°C) | |
| | | After Thirteen Weeks | | 1.87×10^{-4} (26°C) | 1.39×10^{-4} (26°C) | |
| | | After Fourteen Weeks | | 1.82×10^{-4} (22°C) | 1.50×10^{-4} (22°C) | |
| 13. LiPF ₆ (as received) & Li (2 elec-trodes) | AgO & Li (2 elec-trodes) | After Fifteen Weeks | 0.0813 (Li) ^b | 1.85×10^{-4} (23°C) | 1.48×10^{-4} (23°C) | |
| | | Initial | 0.2960 (AgO) 0.0921 (Li) | 2.05×10^{-2} (29°C) | 2.04×10^{-2} (29°C) | After one week the lithium turned black. After four weeks the solution's color changed from yellow to yellow-green and it was slightly turbid. The AgO pellet remained intact for six weeks after which time it began to disintegrate slowly. |
| | | 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) | |

^b Weight change cannot be calculated because pellet disintegrated.

TABLE VII. CHEMICAL STABILITY OF ELECTRODE MATERIALS IN ELECTROLYTE SOLUTIONS

A. Stability in N-Nitrosodimethylamine Solutions. (Continued)

| Solute | Electrode Material | Time of Measurement | Weight of Electrode Material (g) | Specific Conductance ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure | |
|-----------------|-------------------------|----------------------|----------------------------------|---|---------------------------------|---------------------------------|--|
| | | | | Exposed Electrolyte | Blank Electrolyte | | |
| 13. (Continued) | AgO & Li (2 electrodes) | After Five Weeks | | 1.87×10^{-2} (29°C) | 2.06×10^{-2} (29°C) | | |
| | | After Six Weeks | | 1.82×10^{-2} (28°C) | 2.03×10^{-2} (28°C) | | |
| | | After Seven Weeks | | 1.86×10^{-2} (26°C) | 1.87×10^{-2} (26°C) | | |
| | | After Eight Weeks | | 1.77×10^{-2} (26°C) | 2.00×10^{-2} (26°C) | | |
| | | After Nine Weeks | | 1.85×10^{-2} (26°C) | 1.90×10^{-2} (26°C) | | |
| | | After Ten Weeks | | 1.83×10^{-2} (26°C) | 1.90×10^{-2} (26°C) | | |
| | | After Eleven Weeks | | 1.74×10^{-2} (25°C) | 1.94×10^{-2} (25°C) | | |
| | | After Twelve Weeks | | 1.69×10^{-2} (27°C) | 2.02×10^{-2} (27°C) | | |
| | | After Thirteen Weeks | | 1.88×10^{-2} (26°C) | 1.89×10^{-2} (26°C) | | |
| | | After Fourteen Weeks | | 1.66×10^{-2} (22°C) | 1.59×10^{-2} (22°C) | | |
| | | After Fifteen Weeks | | 0.1327 (Li) ^b | 1.85×10^{-2} (23°C) | 1.88×10^{-2} (23°C) | |

^b Weight change of AgO cannot be calculated because pellet disintegrated.

TABLE VII. 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 ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|-----------------------|-------------------------|------------------------------|----------------------------------|---|------------------------------|--|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 14. LiPF_6^a | AgO & Li (2 electrodes) | Initial | 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. After seven weeks the lithium showed signs of extensive disintegration. |
| | | After One Day | 0.2350 (Li) ^b | 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) | |
| | | After Five Weeks | | 1.88×10^{-2} (29°C) | 1.93×10^{-2} (29°C) | |
| | | After Six Weeks | | 1.93×10^{-2} (28°C) | 1.42×10^{-2} (28°C) | |
| | | After Seven Weeks | | 1.86×10^{-2} (27°C) | 1.82×10^{-2} (27°C) | |
| | | After Eight Weeks | | 1.85×10^{-2} (26°C) | 1.88×10^{-2} (26°C) | |
| After Nine Weeks | | 1.93×10^{-2} (26°C) | 1.91×10^{-2} (25°C) | | | |
| After Ten Weeks | | 1.87×10^{-2} (26°C) | 1.95×10^{-2} (26°C) | | | |

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

^b Weight change of AgO cannot be calculated because pellet disintegrated.

TABLE VII. 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 ($\text{ohm}^{-1} \text{cm}^{-1}$) | | Visible Change After Exposure |
|-----------------|-------------------------|----------------------|----------------------------------|---|---------------------------------|-------------------------------|
| | | | | Exposed Electrolyte | Blank Electrolyte | |
| 14. (Continued) | AgO & Li (2 electrodes) | After Eleven Weeks | | 1.76×10^{-2} (25°C) | 1.90×10^{-2} (25°C) | |
| | | After Twelve Weeks | | 1.87×10^{-2} (27°C) | 1.85×10^{-2} (27°C) | |
| | | After Thirteen Weeks | | 1.94×10^{-2} (26°C) | 1.88×10^{-2} (26°C) | |
| | | After Fourteen Weeks | | 1.79×10^{-2} (23°C) | 1.79×10^{-2} (23°C) | |
| | | After Fifteen Weeks | 0.2350 | 1.74×10^{-2} (23°C) | 1.82×10^{-2} (23°C) | |

TABLE VIII. HALF-CELL TESTS ON AGED LITHIUM^a

| Solvent | Solute | Electrolyte | | Current Density (ma/cm ²) | Voltage of Anode | | |
|---------|--------------------------------|---------------------|-------------------|---------------------------------------|------------------|-------|--------------------------------------|
| | | Reference Electrode | Counter Electrode | | Initial | Final | Open Circuit Voltage After Discharge |
| 1. NDA | LiPF ₆ ^b | Ag/AgCl | Ag/AgCl | 0 | -3.30 | | |
| | | | | 1 | -3.30 | -3.25 | -3.31 |
| | | | | 5 | -3.31 | -3.06 | -3.31 |
| | | | | 10 | -2.75 | -2.34 | -3.00 |
| 2. NDA | LiPF ₆ ^c | Ag/AgCl | Ag/AgCl | 0 | -2.95 | | |
| | | | | 1 | -2.68 | -2.80 | -2.95 |
| | | | | 5 | -2.25 | -1.96 | -2.90 |
| | | | | 10 | -2.18 | -0.82 | -2.88 |

^a The lithium anodes were aged in the electrolyte for 18 weeks before this test (see Table VII, numbers 2 and 3).

^b The LiPF₆ used was dried at 110°C.

^c The LiPF₆ was used "as received."

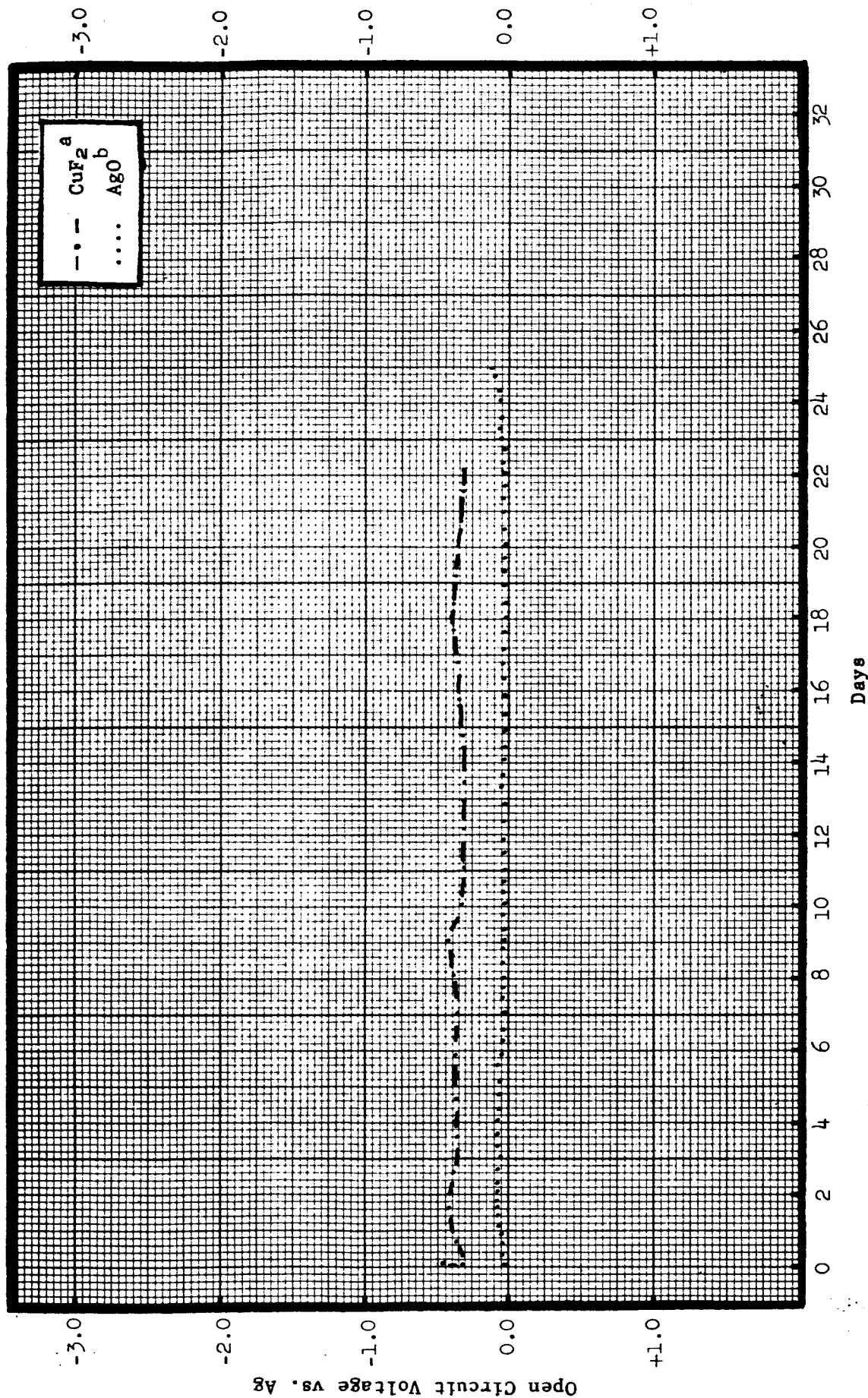


FIGURE 7. COMPATIBILITY STUDIES OF ELECTRODES IN ELECTROLYTES

Figure 7-a. Cathodes in 1m AlCl₃-Acetonitrile.

- a CuF₂ was pressed on 1cm² Ag expanded metal substrate. A slight gray precipitate formed after one day. At the end of the test the weight loss of active material was 0.1343g.
- b AgO was pressed on 1cm² Ag expanded metal substrate. Slight gassing occurred initially and the electrode changed from black to white. A slight gray precipitate formed after one day. No appreciable change in open circuit voltage occurred between the 32nd and 63rd day of the stability tests.

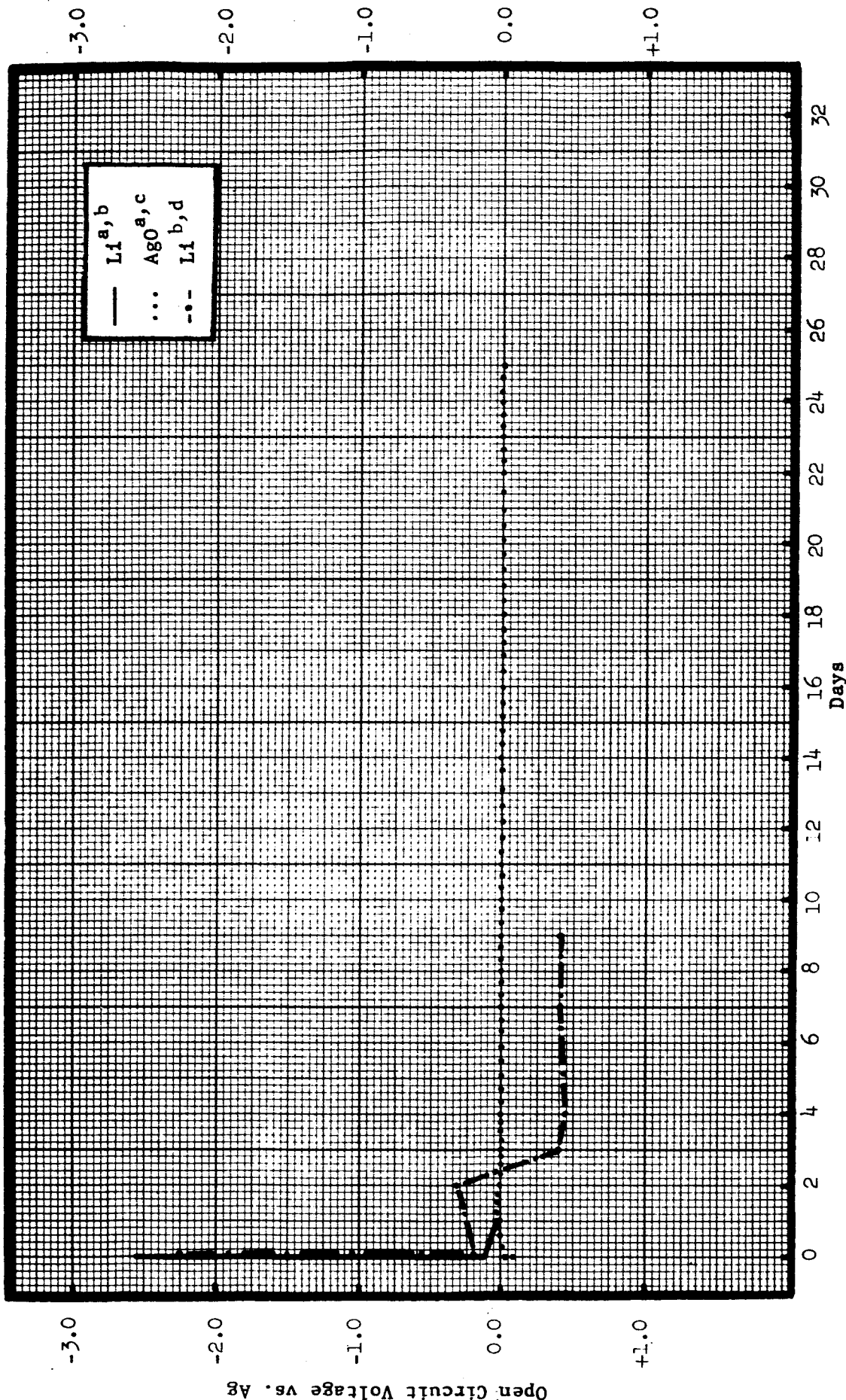


FIGURE 7. COMPATIBILITY STUDIES OF ELECTRODES IN ELECTROLYTES (Continued)

- a The electrolyte was prepared from $1m \text{ LiPF}_6\text{-N-Nitrosodimethylamine}$.
substrates.
- b Vigorous gassing occurred initially and a white precipitate formed.
- c Moderate gassing occurred initially and an orange film formed on the electrode. The solution darkened after 10 days.
- d The electrolyte was prepared from vacuum-dried LiPF_6 .

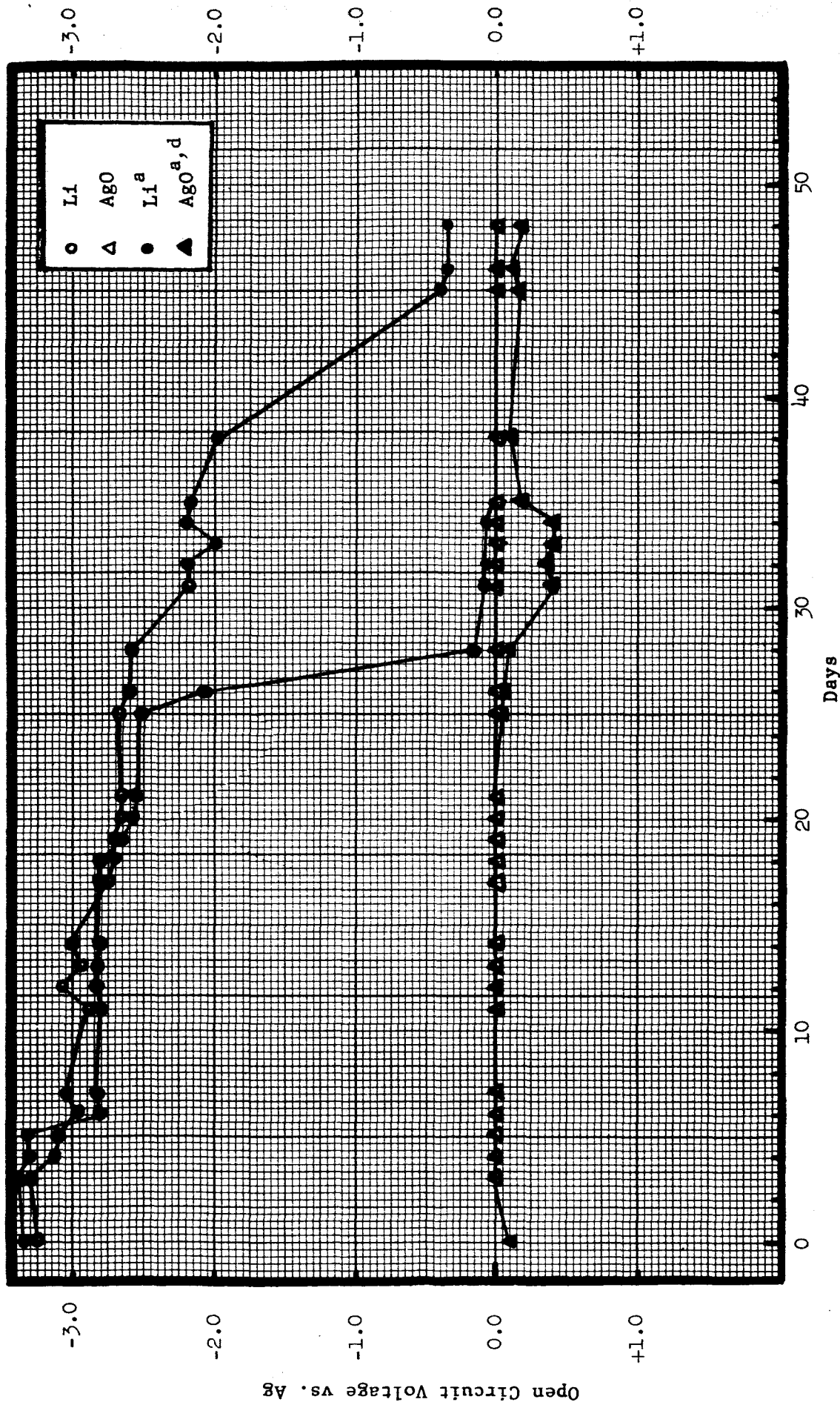


FIGURE 7. COMPATIBILITY STUDIES OF ELECTRODES^b IN ELECTROLYTES (Continued)

Figure 7-c. Electrodes in 1m LiPF₆-N-Nitrosodimethylamine.

- a Electrolyte contained ca. 0.4 weight percent HPF₆. A 65 percent aqueous solution of HPF₆ was used.
- b Pressed on 1cm² Ag expanded metal substrates.
- c Dried at 110°C in a nitrogen atmosphere.
- d No change in open circuit voltage occurred between the 32nd and the 63rd day.

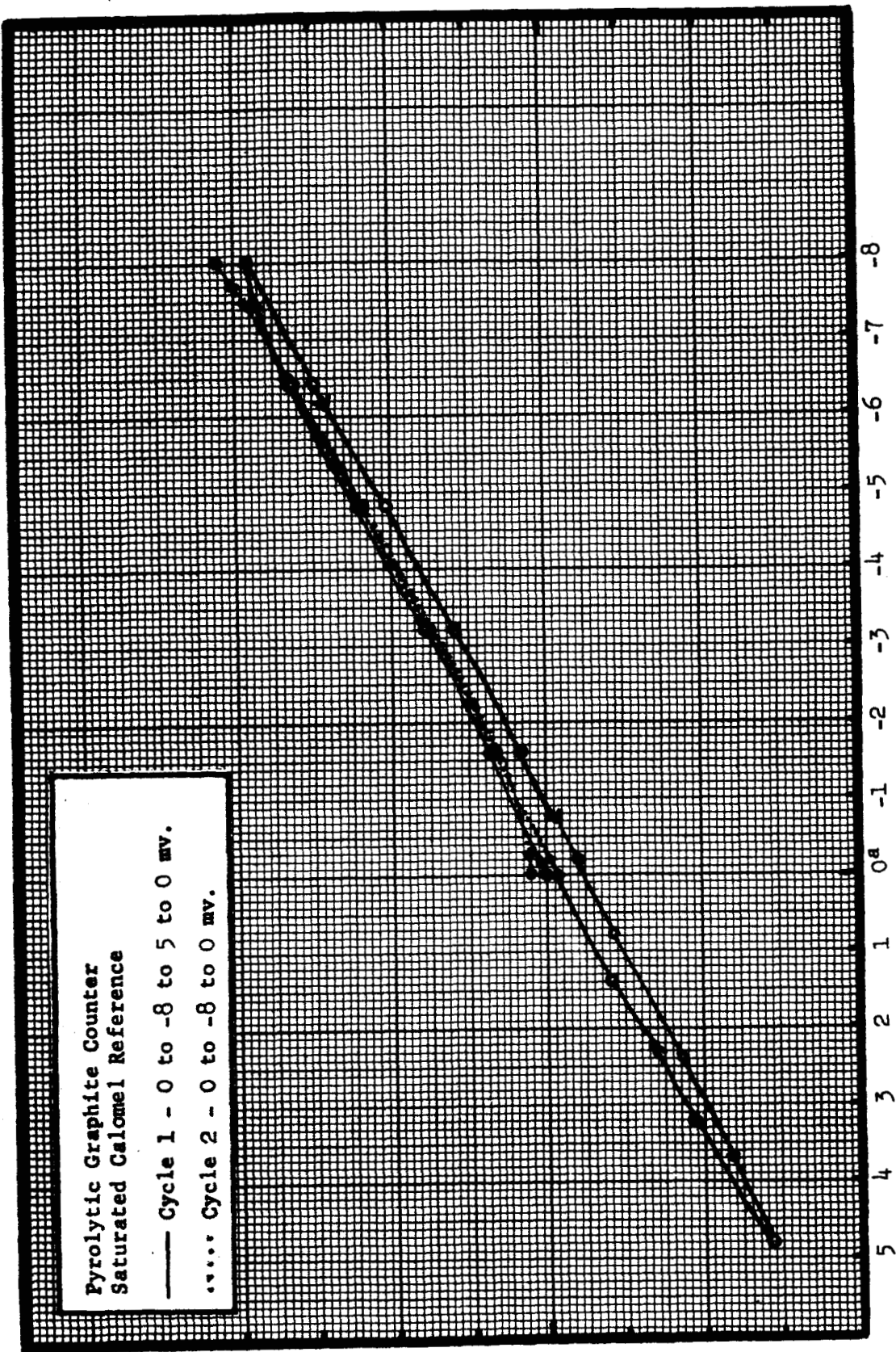


FIGURE 8. REFERENCE ELECTRODE STUDY - HYSTERESIS OF SILVER-SILVER CHLORIDE ELECTRODE

^a Open circuit value of this reference electrode is -24.9mv versus a saturated calomel reference electrode.

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