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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,
Robert W. Adler and Warren L. Towle

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

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Over one hundred nonaqueous electrolytes with conductivities in excess of $1 \times 10^{-2} \text{ ohm}^{-1} \text{ cm}^{-1}$ have been developed since the inception of the program. Quantitative determinations of conductance and viscosity as a function of electrolyte concentration have resulted in the development of an empirical equation which enables one to predict conductance at a given concentration within an order of magnitude, provided that the viscosity of the solution and the dielectric constant of the solvent are known.

During the third quarter, eighty-seven anode-electrolyte combinations were studied electrochemically in a screening program which was designed to single out those systems which are most promising for further development. Thus far, four systems have been discovered which could sustain discharges at 100 ma/cm^2 without excessive polarization. An additional forty-one systems could sustain at least 10 ma/cm^2 .

Cathode studies were initiated somewhat later in this period and are presently the subject of intensive investigation.

Chemical stability tests involving anode materials have been carried out in ninety-five of the more highly conductive electrolytes. Cathode stability determinations have been completed in eighteen of the electrolytes thus far.

AUTHOR ↗

INTRODUCTION

The continued 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.

Prior to the third quarter, the experimental work done under the present contract was concerned primarily with the development of highly conductive nonaqueous electrolytes and a clarification of the basic factors governing conductance.

In the past quarter, emphasis has been shifted significantly toward the development of actual batteries. Thus, the work has been extended to electrochemical studies of potential anode and cathode materials in the electrolytes which were developed in the first phase of the program.

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, Robert W. Adler and Warren L. Towle

ABSTRACT

Emphasis was extended to the electrochemical study of prospective electrode-electrolyte systems, in contrast to earlier work which was concerned chiefly with the conductance of nonaqueous electrolytes. Eighty-seven anode-electrolyte systems were screened electrochemically in this period and the development of cathode fabrication techniques was initiated. Chemical stability determinations with anode and cathode materials were carried out in a number of the better electrolytes.

I. OVERALL PROGRESS

I. OVERALL PROGRESS

The experimental progress during the third quarter involved work in the following areas:

1. Electrochemical Half-Cell Screening of Anodes.
2. Electrochemical Studies of Anode Performance in Small Batteries.
3. Fabrication and Electrochemical Studies of Cathodes.
4. Determination of the Chemical Stability of Electrode Materials in Solvents and Electrolyte Solutions.
5. Determination of Conductance and Viscosity as a Function of Electrolyte Concentration.

A. Experimental Approaches.

1. Electrochemical Half-Cell Screening of Anodes.

An effective screening procedure has been adopted for rapidly determining which of the prospective electrode-electrolyte systems are most suitable for the continued development of a high energy density nonaqueous battery.

Evaluation of the individual electrodes is based on the determination of the degree of polarization which an electrode undergoes when it is discharged at different magnitudes of current density in a particular electrolyte.

The standard procedure adopted for this purpose is as follows:

The anode (1 cm² area) under study is made to undergo successive discharges of five minutes duration in combination with a suitable counter electrode at successively increasing values of constant current, corresponding to 0.1, 1, 10 and 100 milliamperes per square centimeter.*

* The system is allowed to stand on open circuit after discharge at each current density until a stable open circuit voltage is observed, i.e. very, very slow change in potential.

The voltage of the test electrode versus a reference electrode is recorded during discharge at each current density and on open circuit. Comparison of these values affords the desired measure of polarization. Current is maintained constant during the discharges by means of the electrochemical kinetics apparatus described in previous reports. All experiments of this kind are performed in an argon atmosphere. Silver-silver chloride is currently being employed as the material for the reference and counter electrodes in all of the screening tests with few exceptions.* This material was chosen because it is believed to possess generally satisfactory characteristics for the work and is convenient to use. In addition, it was thought desirable to employ the same reference and counter electrodes in all systems for the purpose of comparison. Reference electrodes are immersed directly in the test solution without employing a Luggin capillary modification.

In the analysis of the polarization data, evaluation of the individual electrode-electrolyte systems was effected by categorizing each system according to the magnitude of current density, i.e. 0.1, 1, 10 or 100 ma/cm², which the electrode could sustain. The arbitrary basis used for separation into these categories was as follows. If the electrode was observed to be polarized by less than fifty per cent at a given current density, it was judged to be capable of sustaining discharge at that current density. On the other hand, if the degree of polarization was greater than fifty per cent, the system was classified in a lower current density category.** Obviously, since each current density is an order of magnitude greater than the preceding one, classification

* PbO₂ was employed as a counter electrode and as a reference electrode in two of the screening tests.

** The open circuit voltage chosen as a reference for assessing the degree of polarization was the initial value, i.e. the voltage of the anode versus the reference before any discharges were made.

of a system in a particular category does not necessarily represent the upper limit of current density which the system can sustain. Hence, in some cases, where the behavior of a system was particularly interesting, discharges were carried out at current densities other than the standard ones. In such cases, however, the system was still classified under one of the broad categories in the usual manner.

In other systems, borderline cases arose because the degree of polarization varied appreciably during the discharge period at a given current density. Hence, in some cases, polarization was greater than fifty per cent during the first part of the discharge but less than fifty per cent later in the period, or vice versa. In these cases, further arbitrary rules of classification were applied. If the polarization at the end of the discharge period at a given current density was greater than fifty per cent, the system was considered incapable of discharge at that current density. On the other hand, if the polarization was greater than fifty per cent for only a short time at the beginning but less than fifty per cent for the duration of the discharge, the system was considered capable of sustaining discharge at the given current density.

The scope of this screening program will ultimately include all anode and cathode materials which are capable of contributing to the minimum theoretical energy density requirement and all electrolytes developed in the program which exhibited conductivities in excess of $5 \times 10^{-3} \text{ ohm}^{-1} \text{ cm}^{-1}$. Exceptions to the latter are systems which have been judged to be grossly unsuitable by means of stability tests or other experimental data. All electrolyte solutions are prepared in one molal concentration, unless specified otherwise.

2. Electrochemical Studies of Anode Performance in Small Batteries.
During this period, several pilot sized lithium/silver-silver

chloride batteries employing a morpholinium hexafluorophosphate-N-nitrosodimethylamine electrolyte were constructed and discharged. This combination of lithium anode and electrolyte was chosen because of its ability to sustain discharges at high current densities, as demonstrated in the electrochemical screening program. The purpose of this work was twofold.

First of all, it was recognized that in the process of scaling up a small test electrode of one square centimeter area (such as those studied in the screening program) to a size which can be used in an actual battery, the performance characteristics of a system may be altered to some extent. For this reason, the present work was concerned with evaluating the performance of the system under conditions in which the electrodes were ten to twenty times larger than those used in the screening program. Secondly, it was desired to determine how the lithium anode performs when discharged as in an actual battery.*

For this purpose, silver-silver chloride was chosen as a cathode because it was known to possess satisfactory characteristics when coupled with lithium to result in a cell which could be discharged under its own power. Pilot cells of this kind were discharged through resistive loads which were adjusted manually to maintain constant current.

It is to be emphasized that although complete pilot batteries were constructed for this work, the focus of attention was solely on the performance of the lithium anode-electrolyte system. The use of silver-silver chloride as a cathode material was not intended to result in a high energy density battery, but rather it conveniently permitted the study of the lithium electrode when discharged as in an actual battery. Because of the aforementioned

* In the electrochemical screening tests, either the counter or working electrode may become polarized to such an extent at a certain current density that spontaneous discharge is no longer possible. Under these circumstances, the current controlling device drives the discharge at the desired rate regardless of whether the principal electrochemical reaction or a secondary reaction occurs.

considerations, all pilot cells were constructed in a manner such that the number of electrochemical equivalents of silver chloride was appreciably in excess of the lithium so that the performance of lithium would not be inhibited by the limitations of the cathode.

In addition to the cells described above, three small cells involving calcium and magnesium anodes were discharged in morpholinium hexafluorophosphate-acetonitrile electrolytes. The purpose of these experiments was to demonstrate how the anodes performed when discharged as in a battery. The anodes in these cases were of the same size as those employed in the screening program.

3. Fabrication and Electrochemical Studies of Cathodes.

The task of fabricating cathodes which will approach the performance of the better anodes is recognized as a difficult problem. Since the active cathode materials which have been considered thus far are not conductors, they must be combined with a conductive material. Therefore, the method of fabrication is an important factor in determining electrochemical performance. Furthermore, efforts must be directed toward determining the optimum mode of fabrication with each active cathode material in order to effect a valid comparison of these materials. It is also realized that in the cases where a material such as carbon is used as a conductive additive, its chemical and electrochemical properties must be considered when evaluating the overall cathode performance.

With this perspective, the initial work has been concerned with exploring various fabrication techniques to determine how the best performance may be achieved. Cupric fluoride was chosen as the first material for study because of its high theoretical energy density. The first method of construction which was employed involved forming mixtures of cupric fluoride powder and

various conductive materials onto matrices of expanded metal or metal screen. This was effected by compressing the cathode mix into the matrix in a die. The materials which were employed as conductive additives included a carbon black (14R-22) supplied by the Columbian Carbon Company, and metallic copper powder. In order to obtain sufficient mechanical integrity, it was necessary to incorporate an inert binding material into the cathode mixes. For this purpose, a cellulose material (Solka-floc) supplied by the Brown Company was utilized. In these experiments, mixes containing systematically varied proportions of the various constituents were fabricated over a range of pressures.

A technique which is currently being explored experimentally in addition to the method above involves the partial reduction of cupric fluoride to metallic copper. In this work, mixes of cupric fluoride powder and Solka-floc are compressed into expanded metal matrices in a die and subsequently exposed to a current of hydrogen gas at a temperature of ca. 130°C. The degree of reduction varies with the time of exposure.

Other active materials which are being studied at the present time are manganese dioxide, cobalt trifluoride, and lead dioxide.

The method being employed for cathode evaluation is similar to the electrochemical screening procedure for anodes. Evaluation is based on the determination of the polarization which the cathode undergoes when discharged at successively increased values of constant current. For this purpose, lithium, calcium, or magnesium is employed as a counter electrode and silver-silver chloride as a reference electrode.

In addition to the experimental work described above, an intensive effort is being made to develop new approaches to the cathode problem. Some of the possible modes of attack include the following:

- (1) Use of semiconductor electrodes.
- (2) Use of non-stoichiometric compounds.
- (3) Sublimation of one component of a pressed electrode to yield a porous structure.
- (4) Vapor deposition of the electrode components.

4. Determination of the Chemical Stability of Electrode Materials in Electrolyte Solutions.

Stability determinations at room temperature ($27 \pm 3^{\circ}\text{C}$) are divided into two phases. The first step consists of exposing the electrode materials to the various electrolytes for a period of twenty-four hours. After this time, the systems are observed visually. If a gross change in appearance is noted, no further study of stability is carried out.

On the other hand, if only slight or no change in appearance is observed, the second phase consisting of quantitative measurements after an exposure period of two weeks is carried out. For this purpose, the weight of the electrode material and the conductivity of the electrolyte are measured at the beginning and end of the test period. The changes in these values are then determined and visual observations are also recorded. The electrolyte solutions for this work are one molal when not limited by solubility, and the solvents are used as they are received from the suppliers. All tests are carried out in an argon atmosphere.

B. Experimental Results.

1. Electrochemical Half-Cell Screening of Anodes.

A total of eighty-seven anode-electrolyte systems have been screened thus far, involving calcium, lithium and magnesium anodes in electrolytes which in most cases had conductivities in excess of $1 \times 10^{-2} \text{ ohm}^{-1} \text{ cm}^{-1}$. The results are presented in Table I, pages IV-1 through IV-30.

Lithium anodes were found to be capable of sustaining discharges at 100 ma/cm^2 in three different electrolytes:

- (1) Morpholinium hexafluorophosphate-N-nitrosodimethylamine
- (2) Morpholinium hexafluorophosphate-Dimethylformamide
- (3) Tetra-n-propylammonium hexafluorophosphate-N-nitrosodimethylamine

This magnitude of current density is appreciably higher than any reported previously for a nonaqueous system, Although moderate to vigorous gas evolution was observed in all of the above systems, the results clearly demonstrated the capability of lithium for discharge at high rates in nonaqueous electrolytes.

Of greater interest was the performance of calcium in a morpholinium hexafluorophosphate-acetonitrile electrolyte. This system could sustain discharges at 100 ma/cm² and exhibited only a moderate rate of gas evolution.

Forty-one systems were found to be capable of sustaining at least 10 ma/cm² but less than 100 ma/cm². These included nine calcium, twenty-seven lithium and five magnesium anode combinations for which the data are tabulated in Table I, Section II, pages IV-7 through IV-18.

Among these, the following are worthy of particular mention because only slight or no accumulation of corrosion products and electrolyte decomposition was apparent during the screening period:

- (1) Calcium Anode Systems.
 - a. AlCl₃-Acetonitrile
 - b. AlCl₃-N-Nitrosodimethylamine
- (2) Lithium Anode Systems.
 - a. KPF₆-Dimethylformamide
 - b. LiCl-Dimethylformamide
 - c. MgCl₂-N-Nitrosodimethylamine
 - d. (CH₃)₄NPF₆-N-Nitrosodimethylamine
 - e. (n-C₄H₉)₄NCl-Ethylene Carbonate (80%)-Propylene Carbonate (20%)

- f. $(p-C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$ -N-Nitrosodimethylamine
- (3) Magnesium Anode Systems.
- a. $AlCl_3$ -N-Nitrosodimethylamine

The anode-electrolyte combinations which could sustain discharges at a current density of at least 1 ma/cm^2 but less than 10 ma/cm^2 numbered thirty-three in all; ten with calcium, twenty with lithium, and three with magnesium anodes (Table I, Section III, pages IV-19 through IV-27). Many of these show promise for use in batteries which are to be discharged at low rates. In general, the majority of these systems did not suffer appreciable changes in appearance upon discharge.

It should be noted that more electrolytes in the 1 ma/cm^2 category consisted of solutes with inorganic cations in comparison to those in the higher current density divisions in which solutes with quaternary organic cations were more dominant.

Five of the systems studied in this period were qualified as being capable of discharge at 0.1 ma/cm^2 and are tabulated in Table I, Section IV, pages IV-28 and IV-29. In addition, four systems were studied which could not sustain discharge even at the 0.1 ma/cm^2 rate. These are presented in Table I, Section V, page IV-30.

2. Electrochemical Studies of Anode Performance in Small Batteries.

Upon discharging several pilot sized lithium/silver-silver chloride batteries of varying design in a morpholinium hexafluorophosphate-NDA electrolyte, it was apparent that the Ag/AgCl cathode was limiting the performance of lithium, in spite of an appreciable excess of cathode material. However, by use of a rather unique cell design, a lithium anode performance was achieved which was considerably better than any reported in the literature. At a current density of 56 ma/cm^2 , the lithium anode exhibited a potential of greater than 1.6 volts against a silver-silver chloride reference electrode. At current densities of 40 and 20 ma/cm^2 ,

steady discharge potentials of 2.2 and 2.4 volts, respectively, were observed for the lithium electrode, compared to an open circuit potential of 2.95 volts. The discharge curves for this cell at these current densities are presented in Figure 1, page IV-31. The lithium anode was also discharged at a current density of 77 ma/cm^2 for a few minutes. The potential in this case was about 1.0 volts versus the silver-silver chloride reference electrode. The essential features of the cell were as follows:

Two lithium rods, one-half inch in diameter, were inserted perpendicularly through three vertically oriented silver-silver chloride plates which were parallel to each other at a distance of three-eighths of an inch. The silver chloride plates were three inches in width by 4.7 inches in length, and were insulated electrically from the lithium with rubber grommets. This assembly was placed in a hard rubber container, along with the morpholinium hexafluorophosphate-NDA electrolyte. The area of lithium exposed to the electrolyte was sixteen square centimeters. The primary advantage of this cell design was that it made possible a large ratio of cathode to anode area, and at the same time kept the average distance between anode and cathode at a minimum.

Although construction of the cell in this manner permitted discharges at $50\text{-}60 \text{ ma/cm}^2$ with substantial anode voltages, the performance did not equal that observed in the screening test in which a current density of 100 ma/cm^2 was sustained.

In addition to the work described above, three small cells in which a morpholinium hexafluorophosphate-acetonitrile electrolyte was employed were discharged through resistive loads. Here again, attention was focused on anode performance. The anode-cathode electrode combinations are listed below with the corresponding

tables in which the data are presented.

<u>Cell Number</u>		
(1)	Calcium/silver-silver chloride	Table II, page IV-32
(2)	Calcium/lead dioxide	Table III, page IV-33
(3)	Magnesium/silver-silver chloride	Table IV, page IV-34

Silver-silver chloride was used as a reference electrode for all of these cells. In cell number (1), calcium exhibited substantial voltages (-1.6 volts) versus the reference electrode at current densities as high as 120 ma/cm². It may be noted that the anode open circuit potential increased approximately 0.5 volts after discharge for one hour, indicating that discharge improved the electrode in this case. A similar result was observed for cell number (2), although the anode potentials were about 0.2 volts lower than those for cell number (1).

The magnesium anode in cell number (3) did not polarize appreciably at a current density of ca. 9 ma/cm², but at 45 ma/cm² the potential of the anode versus the reference was only about -0.78 volts.

3. Fabrication and Electrochemical Studies of Cathodes.

Initial efforts in the development of cathodes were concerned with the exploration of two different fabrication techniques.

The first technique involved the preparation of forty-one cupric fluoride cathodes by dry-pressing mixtures of the active material and conductive additives as described in the section on experimental approaches. Electrochemical measurements indicated that some of these cathodes were suitable for discharge at low rates (1-5 ma/cm²). However, in most cases, polarization was appreciable even at low current densities. Cupric fluoride cathodes which contained carbon black as the conductive additive performed

better than those which contained metallic copper powder. The cathodes which yielded the best performance were those which had compositions in the following range:

<u>Component</u>	<u>Weight %</u>
CuF ₂	90-94
Carbon black	3-5
Cellulose (Solka-Floc)	3-5

In general, however, the electrochemical performance of a cathode prepared in a given manner was not reproducible within reasonable limits. Because of this difficulty, it was not possible to make a quantitative comparison of the effect of variables such as composition and fabrication pressure.

The second fabrication technique that was studied involved the partial reduction of cupric fluoride pellets to metallic copper, utilizing hydrogen gas. Preliminary work in this area was concerned with determining the conditions under which the desired degree of reduction could be effected and controlled. It was found that the optimum temperature for reduction in a hydrogen atmosphere was ca. 130°C. At temperatures in excess of 140°C, the reduction proceeded too rapidly to be controlled, while at temperatures of 120°C or lower, the rate of reduction was negligible. The time of exposure to the hydrogen atmosphere has been varied between one and two hours. The data for the electrochemical screening measurements of seven cupric fluoride cathodes fabricated in this manner are presented in Table V, Section A, page IV-36.

Obviously, until reproducibility is achieved, a valid comparison of the effects of variation in the fabrication process on electrochemical performance is not possible.

In addition to the work with cupric fluoride, three lead dioxide cathodes were studied electrochemically. The details of construction

and the polarization data are presented in Table V, Section B, page IV-37.

Work has also been started on the fabrication of cobalt trifluoride and manganese dioxide cathodes.

4. Determination of the Chemical Stability of Electrode Materials in Electrolyte Solutions.

a. Anode Materials.

Stability determinations involving calcium, lithium, and magnesium anodes in pure solvents (no solute) have been reported previously. A condensation of these results is presented below for comparison with the current results for stability of the same materials in electrolyte solutions.

<u>Solvent</u>	<u>Anode Material</u>		
	Calcium	Lithium	Magnesium
Acetone	S	U	S
Acetonitrile	S	U	S
Dimethyl Cyanamide	S	U	S
Dimethylformamide	S	U	S
Propylene Carbonate- Ethylene Carbonate	No Test	S	No Test
N-Nitrosodimethylamine	S	S	S

S = No reaction or only slight reaction over 1 to 2 day exposure period.

U = Appreciable reaction over 1 to 2 day exposure period.

Tests of twenty-four hours duration involving calcium, lithium, and magnesium have been carried out for two hundred and sixty-seven (267) anode-electrolyte combinations involving ninety-five (95) of the more highly conductive electrolytes.* The data are presented in Table VI, Section I, pages IV-39 through IV-58.

* In some cases, only one or two of the three anode materials were tested for stability in a particular electrolyte. The remaining tests will be completed shortly.

The number of systems which exhibited no change or only slight changes with each of the anode materials is listed below along with the total number of electrolytes in which each anode material was tested.

<u>Anode Material</u>	<u>Number of Systems Showing No Change or Only Slight Change</u>	<u>Number of Electrolytes Tested</u>
Calcium	59	94
Lithium	15	89
Magnesium	65	84

In general, stability was greater in solutions of inorganic salts such as KPF_6 , $MgCl_2$, $AlCl_3$, and KI , although a significant number of quaternary ammonium organic salt solutions were also quite stable. Acetone and dimethyl cyanamide solutions were unstable toward the anode materials in almost all cases, although there were some exceptions. Solutions in acetonitrile, dimethylformamide, ethylene carbonate-propylene carbonate, and N-nitrosodimethylamine were significantly less reactive.

The second phase of the stability test program, consisting of quantitative measurements after two week exposure is now in progress. Thus far, tests of this kind have been completed for twenty-five anode-electrolyte combinations. The data are presented in Table VI, Section II, pages IV-59 through IV-64. Out of the twenty-five, nine were unstable to an appreciable extent. The remaining sixteen did not undergo appreciable changes in appearance, weight, or conductivity. The table should be consulted for details regarding the systems studied.

b. Cathode Materials.

Stability determinations were carried out with cupric fluoride, manganese dioxide, and cobalt trifluoride in four of the pure solvents (no solute). These tests

consisted of visual observations and conductivity measurements over an exposure period of two weeks. The data are presented in Table VII, page IV-65. No appreciable change in appearance was observed in any of the solvents, but significant changes in conductivity were observed in several cases, particularly in tests with cobalt trifluoride. Manganese dioxide appeared to be the most stable of the three cathode materials on the basis of the visual observations and conductivity measurements.

Tests of twenty-four hours duration have been completed with CuF_2 , MnO_2 and CoF_3 in eighteen of the better electrolytes. Elemental copper and cobalt have also been tested in some of the same electrolytes. The results are listed in Table VIII, pages IV-66 through IV-72.

MnO_2 was found to cause a noticeable change in only three of the solutions. CuF_2 and CoF_3 were more reactive, causing changes of varying degree in several of the solutions. Copper and cobalt metals did not appear to affect any of the electrolytes in which they were tested.

Tests of two weeks duration have not been initiated thus far.

5. Conductance and Viscosity Versus Concentration of Electrolyte Solutions.

The quantitative study of electrolyte properties was confined to one system in this period. This was morpholinium hexafluorophosphate-acetonitrile for which a maximum conductivity of $5.69 \times 10^{-2} \text{ ohm}^{-1} \text{ cm}^{-1}$ was observed at a concentration of 1.565 molar. The results are presented in Table IX, page IV-73.

II. CURRENT PROBLEMS

II. CURRENT PROBLEMS

The development of cathodes which will yield electrochemical performances comparable to the better anodes has already been cited as a major problem. Reproducibility of cathode performance was particularly difficult to achieve in the first attempts at cathode fabrication, and continues to be a problem.

Difficulties were also encountered in some areas where the purity of materials may be important. In this respect, the cupric fluoride powder used in cathode studies was found by X-ray analysis (fluorescence and diffraction) to contain several metals.

Quantitative determinations have not been made, but it has been estimated that the major impurities present were iron and nickel in amounts up to one per cent. It has also been observed that the cupric fluoride material varies from batch to batch as received from the supplier; in some batches, metallic impurities were present which were not found in others. Whether or not the amounts of these impurities which are present are significant from the standpoint of cathode performance remains to be determined.

III. WORK TO BE PERFORMED IN THE NEXT QUARTER

III. WORK TO BE PERFORMED IN THE NEXT QUARTER

A. Cathode Studies.

Efforts will be concentrated on the development of new concepts of cathode design and the continued study of the fabrication techniques which have been utilized thus far.

B. Anode Studies.

Electrochemical screening tests will be completed for calcium, lithium, and magnesium in some of the electrolytes in which these anodes have not been studied before.

A more detailed electrochemical investigation will be carried out with some of the better anode-electrolyte combinations which were screened previously. This work will be directed toward clarifying the electrochemical-kinetic processes which result in the superior anode performances. Electrolytes which are of particular interest include acetonitrile and N-nitrosodimethylamine solutions of morpholinium hexafluorophosphate.

C. Chemical Stability Determinations.

Twenty-four hour stability tests will be carried out with anode and cathode materials in electrolytes which have not been evaluated for stability thus far.

Tests of two weeks duration will also be conducted with anode and cathode materials in some of the electrolytes which were not tested previously.

IV. TEST RESULTS

TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES

		<u>Page</u>
I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm ² .		
A. Calcium Anode Systems.		IV-5
<u>Solvent</u>	<u>Solute</u>	
1. Acetonitrile (AN)	Morpholinium hexafluorophosphate - $\begin{array}{c} \text{O} \begin{array}{l} \diagup \text{CH}_2\text{CH}_2 \\ \diagdown \text{CH}_2\text{CH}_2 \end{array} \text{NH}_2\text{PF}_6 \end{array}$	
B. Lithium Anode Systems.		IV-6
1. Dimethylformamide (DMF)	Morpholinium hexafluorophosphate	
2. N-nitrosodimethylamine (NDA)	Tetra-n-propylammonium hexafluorophosphate - (n-C ₃ H ₇) ₄ NPF ₆	
3. NDA	Morpholinium hexafluorophosphate	
II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm ² .		
A. Calcium Anode Systems.		IV-7
1. AN	AlCl ₃	
2. AN	Tri-n-propylammonium hexafluoroarsenate - (n-C ₃ H ₇) ₃ HNAsF ₆	
3. AN	Tetra-n-propylammonium tetrafluoroborate - (n-C ₃ H ₇) ₄ NBF ₄	
4. AN	N-Phenyl N,N,N,-trimethylammonium hexafluorophosphate - (C ₆ H ₅)(CH ₃) ₃ NPF ₆	
5. DMF	MgCl ₂	
6. DMF	Tetra-n-propylammonium hexafluorophosphate	
7. DMF	Tetra-n-butylammonium chloride - (n-C ₄ H ₉) ₄ NCl	
8. DMF	Tetraphenylphosphonium chloride - (C ₆ H ₅) ₄ PCl	
9. NDA	AlCl ₃	
B. Lithium Anode Systems.		IV-10
1. Dimethyl cyanamide (DMC)	Morpholinium hexafluorophosphate	
2. DMF	KPF ₆	
3. DMF	LiCl	
4. DMF	MgCl ₂	
5. DMF	Tetra-n-butylammonium chloride	
6. DMF	Tetraphenylphosphonium chloride	
7. 80 WT % Ethylene Carbonate (EC); 20 WT % Propylene Carbonate (PC)	AlCl ₃ (1 molal); LiCl (saturated)	

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

		Page
II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm ² . (Continued)		
B. Lithium Anode Systems. (Continued)		IV-10
8.	80 WT % EC; 20 WT % PC	Tetra-n-butylammonium chloride
9.	80 WT % EC; 20 WT % PC	Tetra-n-propylammonium hexafluorophosphate
10.	80 WT % EC; 20 WT % PC	Morpholinium hexafluorophosphate
11.	NDA	AlCl ₃
12.	NDA	N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate - (p-C ₁₂ H ₂₅ C ₆ H ₄ CH ₂)(CH ₃) ₃ NPF ₆
13.	NDA	MgCl ₂
14.	NDA	Morpholinium hexafluorophosphate (0.5 molal) - NaSbF ₆ (0.5 molal)
15.	NDA	Di-n-butylammonium hexafluoroarsenate - (n-C ₄ H ₉) ₂ H ₂ NAsF ₆
16.	NDA	Tetra-n-butylammonium chloride
17.	NDA	Tetramethylammonium hexafluorophosphate - (CH ₃) ₄ NPF ₆
18.	NDA	Tetraphenylarsonium chloride - (C ₆ H ₅) ₄ AsCl
19.	NDA	Tetraphenylphosphonium chloride
20.	NDA	Tetra-n-propylammonium hexafluoroarsenate
21.	NDA	Tetra-n-propylammonium tetrafluoroborate
22.	NDA	N-Benzyl N,N,N-trimethylammonium hexafluoroantimonate - (C ₆ H ₅ CH ₂)(CH ₃) ₃ NSbF ₆
23.	NDA	N-Phenyl N,N,N-trimethylammonium hexafluorophosphate
24.	PC	AlCl ₃ (1 molal); LiCl (saturated)
25.	PC	Morpholinium hexafluorophosphate
26.	Tetramethylurea (TMU)	Morpholinium hexafluorophosphate
27.	TMU	Morpholinium hexafluorophosphate (1 molal); LiCl (0.5 molal)
C. Magnesium Anode Systems.		IV-17
1.	Acetone	N-Phenyl N,N,N-trimethylammonium hexafluorophosphate
2.	AN	Tri-n-propylammonium hexafluoroarsenate
3.	AN	Tetra-n-propylammonium tetrafluoroborate
4.	DMF	MgCl ₂
5.	NDA	AlCl ₃
III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm ² .		
A. Calcium Anode Systems.		IV-19
1.	AN	CsPF ₆ (0.5 molal)
2.	AN	KPF ₆
3.	AN	NH ₄ SO ₃ F
4.	AN	Tetra-n-propylammonium hexafluoroarsenate

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

	<u>Page</u>
III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm ² . (Continued)	
A. Calcium Anode Systems. (Continued)	IV-19
5. AN	Tetra-n-butylammonium chloride
6. DMF	Tetraphenylarsonium chloride
7. 80 WT % EC; 20 WT % PC	Tetra-n-butylammonium chloride
8. NDA	Tetra-n-propylammonium hexafluorophosphate
9. NDA	Tetra-n-butylammonium chloride
10. NDA	Morpholinium hexafluorophosphate
<hr/>	
B. Lithium Anode Systems.	IV-22
1. DMF	Tetra-n-propylammonium hexafluorophosphate
2. 80 WT % EC; 20 WT % PC	KPF ₆
3. NDA	CsPF ₆ (0.5 molal)
4. NDA	FeCl ₃
5. NDA	KAsF ₆
6. NDA	KI
7. NDA	KPF ₆
8. NDA	KPF ₆ (1 molal); LiCl (saturated)
9. NDA	K ₃ Cr(SCN) ₆
10. NDA	NH ₄ SO ₃ F
11. NDA	NaBF ₄
12. NDA	NaPF ₆
13. NDA	NaSbF ₆
14. NDA	NaSbF ₆ (1 molal); LiCl (saturated)
15. NDA	Tri-n-propylammonium hexafluoroarsenate
16. PC	KPF ₆
17. TMU	KPF ₆
18. TMU	LiCl (2.23 molal)
19. TMU	LiCl (1.11 molal)
20. TMU	KPF ₆ (1 molal); LiCl (saturated)
<hr/>	
C. Magnesium Anode Systems.	IV-27
1. Acetone	Morpholinium hexafluorophosphate
2. DMF	Tetra-n-butylammonium chloride
3. 80 WT % EC; 20 WT % PC	Tetra-n-butylammonium chloride

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

	<u>Page</u>
IV. Anode-Electrolyte Systems Capable of Sustaining 0.1 ma/cm ² .	
A. Calcium Anode Systems.	IV-28
<hr/>	
1. AN	NaBF ₄
2. AN	NaPF ₆
3. 80 WT % EC; 20 WT % PC	Tetra-n-propylammonium hexafluorophosphate
4. NDA	Tetraphenylphosphonium chloride
B. Magnesium Anode Systems.	IV-29
<hr/>	
1. DMF	Tetra-n-propylammonium hexafluorophosphate
V. Anode-Electrolyte Systems Which Cannot Sustain Discharges at 0.1 ma/cm ² .	
A. Calcium Anode Systems.	IV-30
<hr/>	
1. AN	NaSbF ₆
2. AN	Di-n-butylammonium hexafluoroarsenate
3. AN	Tetra-n-propylammonium hexafluoroantimonate
4. NDA	MgCl ₂

NOTE: All solutions are of one molal concentration where not limited by solubility, unless specified otherwise.

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm².

A. Calcium Anode Systems.

TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm².
 A. Calcium Anode Systems.

Electrolyte		Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities		Current Density (ma/cm ²)		Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
Solvent	Solute				Initial	Final	Initial	Final			
AN	*	Ag/AgCl	Ag/AgCl	-2.08	-2.05 -2.00 -2.00 -----	0.1 1 10 100	-2.05 -1.94 -1.58 -----	-2.00 -1.90 -1.72 -----	Continuous moderate gassing at calcium electrode. Precipitate formed near surface of the calcium. When this was removed, substantial voltages (-1.50 to -2.16) were observed at 20, 30, 40 and 70 ma/cm ² . This system had been tentatively classified in the 10 ma/cm ² because a temporary failure in instrumentation did not permit measurements at 100 ma/cm ² . Subsequent tests have indicated that the system can sustain 100 ma/cm ² . (Refer to data for discharge of this system through fixed resistors.)		

* Morpholinium Hexafluorophosphate - $O \begin{matrix} \diagup CH_2CH_2 \\ \diagdown CH_2CH_2 \end{matrix} NH_2PF_6$

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm².

B. Lithium Anode Systems.

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

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm². (Continued)
 B. Lithium Anode Systems.

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
1.	DMA	*	Ag/AgCl	Ag/AgCl	-3.20	-3.25	0.1	-3.24	-3.28	Vigorous gassing at lithium electrode on open circuit and during discharge. Lithium was gradually consumed, and a precipitate formed in the solution. The appearance of the reference and counter electrodes was unchanged.
						-3.22	1	-3.25	-3.23	
						-3.18	10	-3.04	-3.00	
						-3.22	100	-1.94	-1.72	
2.	NDA	**	Ag/AgCl	Ag/AgCl	-2.95	-2.98	0.1	-2.92	-2.92	Moderate gassing at lithium electrode on open circuit and during discharge. A slight amount of a precipitate was observed near the lithium electrode. No change was apparent in the reference and counter electrodes.
						-2.95	1	-2.90	-2.88	
						-2.95	10	-2.74	-2.68	
						-2.90	100	-1.80	-1.54	
3.	NDA	*	Ag/AgCl	Ag/AgCl	-3.01	-3.01	0.1	-2.94	-2.94	Vigorous gassing at lithium electrode on open circuit and during discharge. A gelatinous material was formed in solution, and the surface of the counter electrode had deteriorated somewhat.
						-3.00	1	-2.92	-2.92	
						-2.92	10	-2.75	-2.75	
						-2.92	100	-2.34	-2.18	

* Morpholinium hexafluorophosphate - $O \begin{matrix} \text{CH}_2\text{CH}_2 \\ \text{CH}_2\text{CH}_2 \end{matrix} \text{NH}_2\text{PF}_6$

** Tetra-n-propylammonium hexafluorophosphate - $(n\text{-C}_3\text{H}_7)_4\text{NPF}_6$

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm².

A. Calcium Anode Systems.

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm².
A. Calcium Anode Systems.

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
1.	AN	AlCl ₃	Ag/AgCl	Ag/AgCl	-2.53	-2.52	0.1	-2.52	-2.52	No change in the appearance of the entire system. Very slow gassing at calcium at 100 ma/cm ² .
						-2.52	1	-2.49	-2.49	
						-2.53	10	-2.20	-2.20	
						-2.55	100	-1.20	-0.98	
2.	AN	*	Ag/AgCl	Ag/AgCl	-2.09	-2.09	0.1	-2.01	-2.01	Continuous moderate gassing at calcium electrode. Calcium gradually became darker in appearance. No change was apparent in the solution, or in the counter and reference electrodes.
						-2.50	1	-1.98	-1.94	
						-2.50	10	-2.04	-2.00	
						-2.35	100	-1.20	-0.94	
3.	AN	**	Ag/AgCl	Ag/AgCl	-2.45	-2.45	0.1	-2.24	-2.17	Calcium electrode began gassing slowly at 10 ma/cm ² . After this point it continued to gas at all times, even on open circuit. Counter electrode had deteriorated somewhat. Electrolyte and reference electrode were not changed in appearance.
						-2.35	1	-1.68	-2.13	
						-2.35	10	-1.80	-1.96	
						-2.35	100	-0.80	-0.65	
4.	AN	***	Ag/AgCl	Ag/AgCl	-1.84	-1.82	0.1	-1.70	-1.79	Calcium became blackened somewhat upon discharge. Solution became cloudy after 100 ma discharge, apparently because of some deterioration of the counter electrode. Slight gassing of calcium at 100 ma/cm ² .
						-2.03	1	-1.60	-1.57	
						-1.99	10	-1.35	-1.36	
						-1.88	100	-0.85	-0.55	

* Tri-n-propylammonium hexafluoroarsenate - (n-C₃H₇)₃HNAsF₆

** Tetra-n-propylammonium tetrafluoroborate - (n-C₃H₇)₄NBF₄

*** N-Phenyl N,N,N-trimethylammonium hexafluorophosphate - (C₆H₅)(CH₃)₃NPF₆

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 A. Calcium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit		Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
					Initial Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Initial	Final	
5.	DMF	MgCl ₂	Ag/AgCl	Ag/AgCl	-2.34	-2.35 -2.40 -2.33	-2.25 -1.95 Reversed	-2.30 -2.12 -1.40	Calcium became blackened somewhat upon discharge. Slight gassing at calcium at 10 ma/cm ² . At 10 ma/cm ² , potential was constant at about -1.40 after 1 minute. Reference and counter electrodes were deteriorated appreciably.
6.	DMF	*	Ag/AgCl	Ag/AgCl	-2.21	-2.22 -2.31 -2.43 -2.42	-2.00 -1.80 -0.40 -0.60	-2.19 -2.10 -1.92 -0.82	At 10 ma/cm ² , potential rose rapidly from initial low value to -1.92. Very slow gassing at calcium at 10 ma/cm ² . Calcium was blackened and solution became cloudy.
7.	DMF	**	Ag/AgCl	Ag/AgCl	-2.24	-2.30 -2.29 -1.91 -1.15	-2.21 -2.13 -1.80 Reversed	-2.28 -2.21 -1.85 -----	Calcium became appreciably corroded and gassed vigorously upon discharge. Reference and counter electrodes deteriorated somewhat.
8.	DMF	***	Ag/AgCl	Ag/AgCl	-2.05	-2.13 -2.14 -2.15 -2.05	-1.98 -1.60 -1.50 Reversed	-2.05 -2.05 -1.66 -----	Calcium became blackened and the reference and counter electrodes deteriorated somewhat.

* Tetra-n-propylammonium hexafluorophosphate - (n-C₃H₇)₄NPF₆

** Tetra-n-butylammonium chloride - (n-C₄H₉)₄NCl

*** Tetraphenylphosphonium chloride - (C₆H₅)₄PCl

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 A. Calcium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
9.	NDA	AlCl ₃	Ag/AgCl	Ag/AgCl	-2.30	----	0.1	-2.31	-2.31	No change in the appearance of the entire system. Very slow gassing at calcium began at 1 ma/cm ² .
						-2.25	1	-2.25	-2.18	
						-2.12	10	-1.50	-1.45	
						-2.15	100	Reversed	----	
								Polarity		

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm².

B. Lithium Anode Systems.

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit		Open Circuit		Voltage of Anode		Further Observations and Remarks			
					Volage of Anode vs. Reference Electrode at Various Current Densities	Initial Final	Initial	Final	vs. Reference Electrode at Various Current Densities	Initial Final				
5.	DMF	*	Ag/AgCl	Ag/AgCl	-3.08	-3.10	0.1	-3.08	-3.08	-3.08	Lithium became appreciably blackened. Reference and counter electrodes deteriorated somewhat, and the electrolyte became cloudy.			
												1	-3.00	-2.93
												10	-2.35	-1.65
												100	Reversed	----
6.	DMF	**	Ag/AgCl	Ag/AgCl	-2.93	-2.90	0.1	-2.90	-2.88	-2.88	Lithium gassed at moderate rate upon immersion in the electrolyte and became appreciably blackened upon discharge. Brown solid was suspended in the solution. AgCl electrodes deteriorated somewhat.			
												1	-2.84	-2.83
												10	-2.42	-2.47
												100	-0.55	Reversed
7.	80 WT % EC 20 WT % PC	AlCl ₃ -LiCl	Ag/AgCl	Ag/AgCl	-2.92	-2.92	0.1	-2.88	-2.88	-2.88	A solid material was produced during reaction which floated at the surface of the solution. Slow gassing was observed only at 100 ma/cm ² . Lithium also became black at this current density.			
												1	-2.70	-2.70
												10	-1.70	-2.33
												100	Reversed	----
8.	80 WT % EC 20 WT % PC	*	Ag/AgCl	Ag/AgCl	-2.93	-2.89	0.1	-2.88	-2.84	-2.84	No changes in the appearance of the entire system.			
												1	-2.62	-2.67
												10	-1.18	-1.68
												100	Reversed	----

* Tetra-n-butylammonium chloride - (n-C₄H₉)₄NCl
 ** Tetraphenylphosphonium chloride - (C₆H₅)₄PCl

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 B. Lithium Anode Systems.

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
1.	DMC	*	Ag/AgCl	Ag/AgCl	-3.15	-3.12 -3.10 -3.02 -3.00	0.1 1 10 100	-3.12 -3.06 -2.64 -1.20	-3.08 -3.04 -2.40 -0.40	Moderate gassing at lithium electrode initially. This slowly decreased, and finally ceased after discharges were made. At this point lithium had a white film on it. The counter electrode had deteriorated somewhat.
2.	DMF	KPF ₆	PbO ₂	PbO ₂	-3.15	-3.05 -3.00 -2.90 -2.70	0.1 1 10 100	-3.08 -2.80 -2.45 -1.0	-3.03 -2.78 -2.45 -0.4	Solution slowly became cloudy. No gassing at any current density. Lithium remained shiny in appearance. At 20 and 30 ma/cm ² the voltages were -1.6 and -1.2, respectively.
3.	DMF	LiCl	PbO ₂	PbO ₂	-3.08	-2.95 -2.92 -2.80 -2.85	0.1 1 10 100	-2.84 -2.60 -2.20 Reversed Polarity	-2.80 -2.62 -2.18 ----	At 40 ma/cm ² , voltage varied between -1.60 and -1.54. No gassing at any current density. The appearance of the electrodes and electrolyte did not change during the screening period.
4.	DMF	MgCl ₂	Ag/AgCl	Ag/AgCl	-3.10	-3.00 -2.92 -2.84 -2.85	0.1 1 10 100	-3.02 -2.55 -1.94 Reversed Polarity	-2.97 -2.35 -2.05 ----	A black product formed at lithium surface and diffused into the solution. Reference and counter electrodes deteriorated somewhat.

* Morpholinium hexafluorophosphate - $O \begin{matrix} \text{CH}_2\text{CH}_2 \\ \text{CH}_2\text{CH}_2 \end{matrix} \text{NH}_2\text{PF}_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage After Discharge at Various Current Densities		Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
					Initial	Final	Initial	Final	
9.	80 WT % PC	*	Ag/AgCl	Ag/AgCl	-3.33	-3.15	0.1	-3.30	Slow gassing at lithium upon immersion in the electrolyte. Gassing ceased at 10 ma/cm ² . No change in the appearance of the system.
						-3.04	1	-2.98	
						-3.00	10	-2.30	
10.	80 WT % PC	**	Ag/AgCl	Ag/AgCl	-2.97	-3.02	0.1	-2.97	Discharges were repeated at 10 ma/cm ² after the 100 ma/cm ² discharge, and yielded steady voltages of -2.30. At 20 ma/cm ² voltage was steady at -1.74. The appearance of lithium was changed very little after the discharges.
						-2.98	1	-2.88	
						-2.88	10	-1.68	
11.	NDA	AlCl ₃	Ag/AgCl	Ag/AgCl	-2.88	-2.88	0.1	-2.84	White film formed on lithium. No gassing except at 100 ma/cm ² (slow). At 20 ma/cm ² voltage was -2.40; at 40 ma/cm ² voltage was -1.90; at 10 ma/cm ² (repeated) voltage was -2.60
						-2.88	1	-2.80	
						-2.85	10	-2.34	
12.	NDA	***	Ag/AgCl	Ag/AgCl	-3.15	-3.10	0.1	-3.05	Lithium became somewhat darker in appearance. No gassing at any time. No change in appearance of the solution.
						-3.02	1	-2.84	
						-2.90	10	-2.16	

* Tetra-n-propylemmonium hexafluorophosphate (n-C₃H₇)₄NPF₆

** Morpholinium hexafluorophosphate - O < CH₂CH₂ > NH₂PF₆

*** N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate - (p-C₁₂H₂₅C₆H₄CH₂)(CH₃)₃NPF₆

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial		Open Circuit		Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks	
					Open Circuit Voltage of Anode vs. Reference Electrode	Discharge at Various Current Densities	Initial	Final	Initial	Final		
13.	NDA	MgCl ₂	Ag/AgCl	Ag/AgCl	-3.00	-3.00	0.1	-2.80	-2.80	At 40 ma/cm ² voltage was steady at -1.08.		
							1	-2.44	-2.60	At 30 ma/cm ² voltage was steady at -1.48.		
							10	-1.60	-2.26	At 20 ma/cm ² voltage was steady at -2.02.		
							100	Reversed Polarity	----	No gassing at any time. Lithium became darker in appearance at high current densities.		
14.	NDA	*	Ag/AgCl	Ag/AgCl	-3.42	-3.20	0.1	-3.35	-3.10	Moderate gassing at lithium at all times.		
							1	-3.00	-2.40	Rate of gassing decreased somewhat as experiment proceeded. Lithium became dark at high current densities. No change in appearance of the solution.		
							10	-2.20	-2.00			
							100	-0.60	-0.40			
15.	NDA	**	Ag/AgCl	Ag/AgCl	-3.15	----	0.1	----	----	Lithium remains shiny in appearance at 100 ma/cm ² . Moderate gassing at lithium at all times.		
							1	-3.08	-2.94			
							10	-2.78	-2.74			
							100	-1.47	-1.10			
16.	NDA	***	Ag/AgCl	Ag/AgCl	-2.88	-2.82	0.1	-2.80	-2.83	Lithium became darkened in appearance.		
							1	-2.70	-2.72	Slow gassing began at 10 ma/cm ² . No change in appearance of the solution.		
							10	-2.28	-2.30			
							100	Reversed Polarity	----			

* Morpholinium hexafluorophosphate - O $\begin{matrix} \text{CH}_2\text{CH}_2 \\ \diagdown \quad \diagup \\ \text{CH}_2\text{CH}_2 \end{matrix}$ NH₂PF₆ - NaSbF₆

** Di-n-butylammonium hexafluoroarsenate - (n-C₄H₉)₂H₂NAsF₆

*** Tetra-n-butylammonium chloride - (n-C₄H₉)₄NCl

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial		Open Circuit		Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks	
					Open Circuit Voltage of Anode vs. Reference Electrode	Reference Electrode	Discharge at Various Current Densities	Current Density (ma/cm ²)	Initial	Final		
17.	NDA	*	Ag/AgCl	Ag/AgCl	-3.50	-3.10	0.1	-3.40	-2.86	No change in appearance of the solution, counter, and reference electrodes. No gassing at any time.		
18.	NDA	**	Ag/AgCl	Ag/AgCl	-3.05	-3.05	0.1	-3.02	-3.02	White film formed at surface of lithium. Slow gassing observed at 100 ma/cm ² . No change in appearance of the solution. Counter electrode had deteriorated slightly.		
19.	NDA	***	Ag/AgCl	Ag/AgCl	-3.05	-3.05	0.1	-2.98	-2.98	No change in appearance of the solution. Lithium became black in appearance and gassed continuously after 10 ma/cm ² discharge. Counter and reference electrodes had deteriorated somewhat.		
20.	NDA	****	Ag/AgCl	Ag/AgCl	-3.28	-3.05	0.1	-3.25	-2.92	At 150 ma/cm ² voltage varied from -0.40 to -0.24. Lithium remained shiny but was gassing moderately at all times, and was eventually consumed. A brown precipitate formed in the solution.		

* Tetramethylammonium hexafluorophosphate - (CH₃)₄NPF₆

** Tetraphenylarsonium chloride - (C₆H₅)₄AsCl

*** Tetraphenylphosphonium chloride - (C₆H₅)₄PCl

**** Tetra-n-propylammonium hexafluoroarsenate - (n-C₃H₇)₄NAsF₆

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
	NDA	*	Ag/AgCl	Ag/AgCl	-2.95	-2.90 -2.95 -2.95 -2.95	0.1 1 10 100	-2.90 -2.83 -2.50	-2.90 -2.83 -2.54	White film formed at surface of lithium. Very slow rate of gassing at all times. No change in appearance of the solution or in the counter and reference electrodes.
51-AI 22.	NDA	**	Ag/AgCl	Ag/AgCl	-3.20	-3.15 -3.05 -3.05 -2.90	0.1 1 10 100	-3.06 -2.78 -2.36	-3.04 -2.82 -2.12	Lithium became blackened at 100 ma/cm ² . No gassing at any time. No change in appearance of the solution or the counter and reference electrodes.
23.	NDA	***	Ag/AgCl	Ag/AgCl	-3.42	-3.20 -3.05 -3.05 -2.95	0.1 1 10 100	-3.35 -2.84 -2.20 -1.20	-3.10 -2.78 -2.18 -0.8	Solution became dark in color. No gassing was apparent. Lithium became somewhat darker in appearance.
24.	PC	AlCl ₃ -TCl	Ag/AgCl	Ag/AgCl	-2.90	-2.95 -2.90 -2.90 -2.90	0.1 1 10 100	-2.78 -2.70 -1.98	-2.80 -2.78 -2.48	Lithium became dark gray in appearance. Slow gassing at lithium began at 100 ma/cm ² . Counter electrode also gassed at 100 ma/cm ² . No change in appearance of the solution or the counter and reference electrodes.

* Tetra-n-propylammonium tetrafluoroborate - (n-C₃H₇)₄NBF₄

** N-Benzyl N,N,N-trimethylammonium hexafluoroantimonate - (C₆H₅CH₂)(CH₃)₃NSbF₆

*** N-Phenyl N,N,N-trimethylammonium hexafluorophosphate - (C₆H₅)(CH₃)₃NPF₆

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities		Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
						Densities	Densities		Initial	Final	
25.	PC	*	Ag/AgCl	Ag/AgCl	-3.02	-3.05 -2.95 -2.92 -2.90	0.1 1 10 100	-3.10 -2.69 -1.52 Reversed	-3.00 -2.74 -1.84 ----	Lithium became light gray in appearance. No gassing at any time. No change in appearance of the solution or counter and reference electrodes.	
26.	TMU	*	Ag/AgCl	Ag/AgCl	-3.21	-3.20 -3.20 -3.15 -3.12	0.1 1 10 100	-3.14 -3.12 -2.86 Reversed	-3.14 -3.09 -2.89 ----	At 20 ma/cm ² voltage varied from -2.80 to -2.71. At 30 ma/cm ² voltage varied from -2.32 to -1.68. At 40 ma/cm ² voltage varied from -2.08 to -0.04. Moderate gassing at lithium at all times. A gray film formed at the lithium surface.	
27.	TMU	**	Ag/AgCl	Ag/AgCl	-3.10	-3.12 -3.12 -3.12 -3.12	0.1 1 10 100	-3.08 -3.04 -2.74 Reversed	-3.08 -3.06 -2.73 ----	Moderate gassing at lithium electrode at all times. No change in appearance of the solution or reference or counter electrodes.	

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II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm².

C. Magnesium Anode Systems.

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 C. Magnesium Anode Systems

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
1.	Acetone	*	Ag/AgCl	Ag/AgCl	-1.20	-1.15 -1.25 -1.30 -1.20	0.1 1 10 100	-0.92 -0.60 -0.68 Reversed	-0.98 -0.84 -0.68 ----	Magnesium became darkened at 10 ma/cm ² . No change in the appearance of the rest of the system. No gassing at any time.
2.	AN	**	Ag/AgCl	Ag/AgCl	-1.75	-1.78 -1.70 -1.62 -1.60	0.1 1 10 100	-1.76 -1.68 -1.32 Reversed	-1.76 -1.62 -1.41 ----	Continuous gassing (moderate) at magnesium at all times. Magnesium became darkened during discharge. No change in the appearance of the rest of the system.
3.	AN	***	Ag/AgCl	Ag/AgCl	-1.42	-1.35 -1.38 -1.42 -1.55	0.1 1 10 100	-1.32 -1.28 -1.06 -0.36	-1.32 -1.25 -1.04 -0.36	No change in appearance of magnesium except at 100 ma/cm ² (Surface became darkened). Slow gassing at 100 ma/cm ² . A white solid settled at the bottom of the cell.

* N-Phenyl N,N,N-trimethylammonium hexafluorophosphate - (C₆H₅)(CH₃)₃NPF₆

** Tri-n-propylammonium hexafluoroarsenate - (n-C₃H₇)₃HNAsF₆

*** Tetra-n-propylammonium tetrafluoroborate - (n-C₃H₇)₄NBF₄

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm². (Continued)
 C. Magnesium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities		Current Density (ma/cm ²)		Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
						Initial	Final	Initial	Final	Initial	Final	
4.	DMF	MgCl ₂	Ag/AgCl	Ag/AgCl	-1.73	-1.78	0.1	-1.65	-1.70	-1.65	-1.70	Magnesium became blackened and the reference and counter electrodes deteriorated somewhat.
						-1.67	1	-0.90	-1.43	-0.90	-1.43	
						-1.70	10	-0.95	-1.15	-0.95	-1.15	
						-1.67	100	Reversed	-----	-----	-----	
5.	NDA	AlCl ₃	Ag/AgCl	Ag/AgCl	-1.24	-1.39	0.1	-1.22	-1.36	-1.22	-1.36	No changes in the appearance of the entire system. Slow gassing at magnesium at 100 ma.
						-1.39	1	-1.24	-1.31	-1.24	-1.31	
						-1.40	10	-0.97	-0.91	-0.97	-0.91	
						-1.40	100	Reversed	-----	-----	-----	

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm^2 .

A. Calcium Anode Systems.

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm².
A. Calcium Anode Systems.

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities		Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
						Initial	Final		Initial	Final	
1.	AN	CaF ₂	Ag/AgCl	Ag/AgCl	-2.00	-1.95	0.1	-1.66	-1.60	No change in the appearance of the entire system. No gassing at any time.	
						-2.02	1	-1.48	-1.53		
						-1.70	10	Reversed Polarity	----		
2.	AN	KPF ₆	Ag/AgCl	Ag/AgCl	-2.15	-2.05	0.1	-1.84	-1.88	No change in the appearance of the entire system. No gassing at any time.	
						-2.20	1	-1.76	-1.73		
						-2.05	10	-1.52	-0.56		
3.	AN	NH ₄ SO ₄	Ag/AgCl	Ag/AgCl	-2.23	-2.35	0.1	-1.76	-2.24	Calcium became somewhat darkened in appearance upon discharge. No gassing at any time. No change in appearance of the solution or counter and reference electrodes.	
						-2.50	1	-1.24	-2.00		
						-2.40	10	-1.22	-0.29		
4.	AN	*	Ag/AgCl	Ag/AgCl	-2.12	-2.10	0.1	-2.06	-2.03	Calcium became dark gray after the discharge. No gassing at any time. No change in appearance of the solution. Counter electrode had a darker appearance.	
						-2.02	1	-1.88	-1.72		
						-2.10	10	-1.40	-0.78		
					-2.12	100	Reversed Polarity				

* Tetra-n-propylammonium hexafluoroarsenate - (n-C₃H₇)₄NAsF₆

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

Electrolyte		Reference Electrode	Counter Electrode	Initial Open Circuit		Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
Solvent	Solute			Open Circuit Voltage of Anode vs. Reference Electrode	Discharge at Various Current Densities		Initial	Final	
5.	AN	Ag/AgCl	Ag/AgCl	-0.93	-0.80 -0.74 -0.81 ----	0.1 1 10 --	-0.90 -0.73 ----- -----	Calcium became darkened. Reference and counter electrodes deteriorated somewhat upon immersion in the electrolyte.	
6.	DMF	Ag/AgCl	Ag/AgCl	-2.10	-2.28 -2.00 -2.21 -1.78	0.1 1 10 100	-2.20 -1.55 -1.00 ----- -----	Calcium became darkened somewhat. Reference and counter electrodes had deteriorated appreciably. Slow gassing at calcium at 10 ma/cm ² .	
7.	80 WT % EC 20 WT % PC	Ag/AgCl	Ag/AgCl	-2.14	-2.12 -2.13 -2.00 -2.10	0.1 1 10 100	-0.50 -1.40 ----- -----	No change in the appearance of the entire system.	

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* Tetra-n-butylammonium chloride - (n-C₄H₉)₄NCl

** Tetraphenylarsonium chloride - (C₆H₅)₄AsCl

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm². (Continued)
 A. Calcium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial		Open Circuit		Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks			
					Open Circuit Voltage of Anode vs. Reference Electrode	Reference Electrode	Discharge at Various Current Densities	Current Density (ma/cm ²)	Initial	Final				
8.	NDA	*	Ag/AgCl	Ag/AgCl	-1.92	-1.88	0.1	-1.60	-1.55	Moderate gassing at calcium began at 10 ma/cm ² . No change in appearance of the entire system.				
											-2.00	1	-0.50	-1.40
											-2.00	10	-0.78	-1.03
9.	NDA	**	Ag/AgCl	Ag/AgCl	-1.35	-1.13	0.1	-1.05	-1.18	At 1 ma, the potential of calcium vs. the reference was more negative than -0.83 during 80% of the discharge time. No change in appearance of the system except for very slight deterioration of the AgCl electrodes.				
											-1.00	1	-0.25	-0.93
											-1.00	10	Reversed Polarity	----
10.	NDA	***	Ag/AgCl	Ag/AgCl	-2.00	-1.92	0.1	-1.72	-1.70	Continuous moderate gassing at the calcium electrode. Calcium became gray in appearance and a gelatinous material formed in the solution.				
											-2.00	1	-1.44	-1.30
											-2.10	10	Reversed Polarity	----

* Tetra-n-propylammonium hexafluorophosphate - (n-C₃H₇)₄NPF₆

** Tetra-n-butylammonium chloride - (n-C₄H₉)₄NCl

*** Morpholinium hexafluorophosphate - $O \begin{matrix} \text{CH}_2\text{CH}_2 \\ \text{CH}_2\text{CH}_2 \end{matrix} \text{NH}_2\text{PF}_6$

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm^2 .

B. Lithium Anode Systems.

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm². (Continued)
 B. Lithium Anode Systems.

Electrolyte		Counter Electrode	Reference Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
Solvent	Solute						Initial	Final	
1.	DMF	Ag/AgCl	Ag/AgCl	-3.21	-3.20 -3.19 -3.20 -----	0.1 1 10 --	-3.20 -2.10 Reversed Polarity -----	-3.00 -2.70 ----- -----	Vigorous gassing at lithium upon immersion in electrolyte. Most of the lithium was consumed during the test period and a gelatinous precipitate formed in the solution
2.	80 WT % EC 20 WT % PC	Ag/AgCl	Ag/AgCl	-3.25	----- -3.02 -3.00 -3.00	0.1 1 10 100	-2.68 -2.08 -1.48 Reversed Polarity	-2.68 -2.68 -0.88 ----- -----	The appearance of the lithium electrode changed only slightly to a light gray color. No change in appearance of the reference and counter electrodes. Counter electrode gassed slowly at 100 ma/cm ² .
3.	NDA	Ag/AgCl	Ag/AgCl	-3.22	-3.05 -2.95 -2.50 -2.50	0.1 1 10 100	-3.10 -2.60 -2.16 Reversed Polarity	-2.90 -2.58 -1.00 ----- -----	Very slow rate of gassing at lithium electrode at all times. No change in appearance of the reference and counter electrodes or the solution.
4.	NDA	Ag/AgCl	Ag/AgCl	-2.10	-2.38 -2.38 -2.38 -2.38	0.1 1 10 100	-2.13 -2.25 -1.40 Reversed Polarity	-2.32 -2.22 -1.85 ----- -----	Solution was darkened appreciably. Lithium electrode became black and rough. Counter electrode became a dark green color.
5.	NDA	Ag/AgCl	Ag/AgCl	-2.95	-3.10 -3.15 -3.15 -2.65	0.1 1 10 100	-3.03 -2.48 -2.16 Reversed Polarity	-2.88 -2.80 -1.28 ----- -----	No change in appearance of the entire system. No gassing at any time.

* Tetra-n-propylammonium hexafluorophosphate - (n-C₃H₇)₄NPF₆

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
6.	NDA	KI	Ag/AgCl	Ag/AgCl	-2.61	-2.55	0.1	-2.46	-2.32	No change in the appearance of the entire system. No gassing at any time.
						-2.52	1	-1.80	-1.88	
						-2.50	10	-1.18	-0.68	
						-2.40	100	Reversed	----	
7.	NDA	KPF ₆	Ag/AgCl	Ag/AgCl	-3.28	-3.25	0.1	-3.10	-3.10	The appearance of lithium changed to a gray-white color after discharge. No gassing at any time. Repeated discharge at 10 ma/cm ² ; yielded lower, but steady voltages.
						-3.20	1	-2.78	-2.83	
						-2.85	10	-2.04	-1.28	
						-2.35	100	-0.20	-0.30	
8.	NDA	KPF ₆ LiCl	Ag/AgCl	Ag/AgCl	-3.29	-3.28	0.1	-3.20	-3.12	No change in the appearance of the entire system. No gassing. Open circuit voltage failed to return to initial value after discharge at high current densities.
						-3.20	1	-2.60	-2.70	
						----	10	-1.70	-1.32	
						-2.30	100	Reversed	----	
9.	NDA	K ₃ Cr(SCN) ₆	Ag/AgCl	Ag/AgCl	-2.95	-2.95	0.1	-2.86	-2.87	The appearance of lithium became dark gray. No apparent change in the appearance of the solution or the reference and counter electrodes.
						-2.65	1	-2.70	-2.39	
						-2.50	10	-1.84	-1.07	
						-2.50	100	Reversed	----	

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
10.	NDA	NH ₄ SO ₃ F	Ag/AgCl	Ag/AgCl	-3.40	-3.25	0.1	-3.35	-3.20	Moderate gassing at lithium electrode at all times. The appearance of lithium changed to a white color. The appearance of the solution and counter and reference electrodes was unchanged.
						-3.00	1	-3.08	-2.92	
						-3.00	10	-2.54	-1.74	
11.	NDA	NaBF ₄	Ag/AgCl	Ag/AgCl	-3.40	-3.08	0.1	-3.16	-2.80	No change in the appearance of the entire system. No gassing at any time.
						-2.90	1	-2.20	-2.04	
						-2.50	10	Reversed Polarity	----	
12.	NDA	NaPF ₆	Ag/AgCl	Ag/AgCl	-3.52	-3.40	0.1	-3.40	-3.25	No change in the appearance of the entire system. Gassing did not occur at the lithium electrode at any time. Counter electrode gassed moderately at 100 ma/cm ² .
						-2.80	1	-2.94	-2.42	
						-2.85	10	-2.10	-1.52	
13.	NDA	NaSBF ₆	Ag/AgCl	Ag/AgCl	-3.45	-3.05	0.1	-3.36	-2.83	Lithium electrode became somewhat darker in appearance. The rest of the system did not change in appearance. No gassing at any time.
						-3.00	1	-3.20	-2.34	
						-2.85	10	-2.96	-1.35	
						----	100	----	----	

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage		Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
					Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities		Initial	Final	
14.	NDA	NaSBF ₆ LiCl	Ag/AgCl	Ag/AgCl	-3.22	-3.15 -2.95 -2.72 ----	0.1 1 10 100	-2.96 -1.96 -0.84 ----	-2.53 -1.76 -1.10 ----	Lithium became somewhat gray in appearance. The other components of the system were unchanged in appearance. No gassing at any time.
15.	NDA	*	Ag/AgCl	Ag/AgCl	-3.40	-3.00 ---- -3.00 ----	0.1 1 10 100	-3.30 ---- -2.74 -1.44	-2.92 ---- ---- ----	Vigorous gassing at lithium electrode at all times. The discharge at 10 ma/cm ² was interrupted after two minutes. At this point, much of the lithium had been consumed. Discharge was then carried out at 100 ma/cm ² for 20 seconds
16.	PC	KPF ₆	Ag/AgCl	Ag/AgCl	-3.18	-3.13 -3.10 -3.00 -2.70	0.1 1 10 100	-3.00 -2.54 -1.96 Reversed Polarity	-2.95 -2.76 -0.88 ----	The counter electrode gassed vigorously at 10 and 100 ma/cm ² and deteriorated somewhat. Lithium changed to a light gray color.
17.	TMU	KPF ₆	Ag/AgCl	Ag/AgCl	-3.18	-3.12 -3.10 -3.00 -1.50	0.1 1 10 100	-3.40 -2.90 -1.80 Reversed Polarity	-3.40 -3.14 -2.40 ----	Lithium changed to a white appearance. Solution became cloudy in the vicinity of the AgCl electrodes. A repeat of the discharge at 1 ma/cm ² yielded only -0.80 volts at the end.

* Tri-n-propylammonium hexafluoroarsenate - (n-C₃H₇)₃HNAsFe

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm². (Continued)
 B. Lithium Anode Systems. (Continued)

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
18.	TMU	LiCl (1.11 molar)	Ag/AgCl	Ag/AgCl	-2.98	-2.98	0.1	-2.93	-2.93	At 10 ma/cm ² , the entire lithium surface began to blacken. After standing for two hours, lithium was badly corroded and gassed on open circuit. Reference and counter electrodes had also deteriorated somewhat.
					-2.98	-2.98	1	-2.74	-2.74	
					-2.85	-2.85	10	-1.12	-0.84	
					----	----	100	----	----	
19.	TMU	LiCl (2.23 molar)	Ag/AgCl	Ag/AgCl	-3.02	-3.00	0.1	-2.92	-2.89	Reference and counter electrodes had deteriorated somewhat. The appearance of the lithium electrode was not changed appreciably. No gassing at any time.
					-2.95	-2.95	1	-2.40	-2.44	
					-2.93	-2.93	10	Reversed Polarity	----	
					----	----	100	----	----	
20.	TMU	KPF ₆ LiCl	Ag/AgCl	Ag/AgCl	-3.00	-2.98	0.1	-2.94	-2.92	No change in the appearance of the entire system. No gassing at any time.
					-2.92	-2.92	1	-2.76	-2.72	
					-2.90	-2.90	10	-1.53	-1.23	
					-2.90	-2.90	100	Reversed Polarity	----	

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm^2 .

C. Magnesium Anode Systems.

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm². (Continued)
 C. Magnesium Anode Systems.

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit		Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
					Open Circuit Voltage of Anode vs. Reference Electrode	Discharge After Various Current Densities		Initial	Final	
1.	Acetone	*	Ag/AgCl	Ag/AgCl	-1.15	-1.15	0.1	-1.04	-1.06	No change in appearance of the entire system. No gassing at any time.
						-1.25	1	-0.80	-0.95	
						-0.95	10	Reversed Polarity	-----	
						-----	100	-----	-----	
2.	DMF	**	Ag/AgCl	Ag/AgCl	-1.21	-1.22	0.1	-1.20	-1.02	Magnesium became somewhat blackened. Reference and counter electrodes deteriorated somewhat and the solution became cloudy.
						-1.32	1	-0.85	-1.23	
						-1.32	10	-0.05	-0.78	
						-1.23	100	Reversed Polarity	-----	
3.	80 WT % PC 20 WT % DMF	**	Ag/AgCl	Ag/AgCl	-1.27	-1.24	0.1	-0.58	-1.18	Moderate gassing at magnesium at 10 ma. No changes in the appearance of the system.
						-1.27	1	-1.18	-1.16	
						-1.27	10	Reversed Polarity	-0.84	
						-1.22	100	Reversed Polarity	-----	

* Morpholinium hexafluorophosphate - $O \begin{matrix} \text{CH}_2\text{CH}_2 \\ \text{CH}_2\text{CH}_2 \end{matrix} \text{NH}_2\text{PF}_6$
 ** Tetra-n-butylammonium chloride - $(n\text{-C}_4\text{H}_9)_4\text{NCl}$

IV. Anode-Electrolyte Systems Capable of Sustaining 0.1 ma/cm^2 .

A. Calcium Anode Systems.

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

IV. Anode-Electrolyte Systems Capable of Sustaining 0.1 ma/cm².
A. Calcium Anode Systems.

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
1.	AN	NaBF ₄	Ag/AgCl	Ag/AgCl	-2.25	-2.22	0.1	-1.40	-1.64	No visible change in the appearance of the entire system. No gassing at any time.
						-2.22	1	-1.00	-1.04	
						-1.82	10	Reversed Polarity	----	
2.	AN	NaPF ₆	Ag/AgCl	Ag/AgCl	-2.31	-2.33	0.1	-1.76	-1.54	Calcium remained shiny except at edges where it turned black at 100 ma/cm ² . No change in appearance of the rest of the system.
						-2.10	1	-1.60	-1.04	
						-2.10	10	-1.24	Reversed Polarity	
3.	80 WT % EC 20 WT % PC	*	Ag/AgCl	Ag/AgCl	-2.15	-2.21	0.1	-1.22	-1.84	Calcium became darkened somewhat upon discharge. No changes in the appearance of the rest of the system.
						-2.25	1	-0.90	-0.80	
						-2.10	10	Reversed Polarity	--	
4.	NDA	**	Ag/AgCl	Ag/AgCl	-2.24	-2.20	0.1	-2.23	-2.20	Moderate gassing at all times. Calcium became darkened upon discharge. Reference and counter electrodes had deteriorated somewhat.
						-0.89	1	-0.97	-0.84	
						-0.82	10	-0.02	Reversed Polarity	
					--	100	--	--		

* Tetra-n-propylammonium hexafluorophosphate - (n-C₃H₇)₄NPF₆

** Tetraphenylphosphonium chloride - (C₆H₅)₄PCl

IV. Anode-Electrolyte Systems Capable of Sustaining 0.1 ma/cm^2 .

B. Magnesium Anode Systems.

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

IV. Anode-Electrolyte Systems Capable of Sustaining 0.1 ma/cm². (Continued)
 B. Magnesium Anode Systems.

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities		Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
						Initial	Final		Initial	Final	
1.	DMF	*	Ag/AgCl	Ag/AgCl	-1.35	-1.36	0.1	-1.19	-1.19	No change in the appearance of the entire system.	
						-1.36	1	Reversed Polarity	-----		
						-----	--	----	----		
						-----	--	----	----		

* Tetra-n-propylammonium hexafluorophosphate - (n-C₃H₇)₄NPF₆

V. Anode-Electrolyte Systems Which Cannot Sustain Discharges at 0.1 ma/cm^2 .

A. Calcium Anode Systems.

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

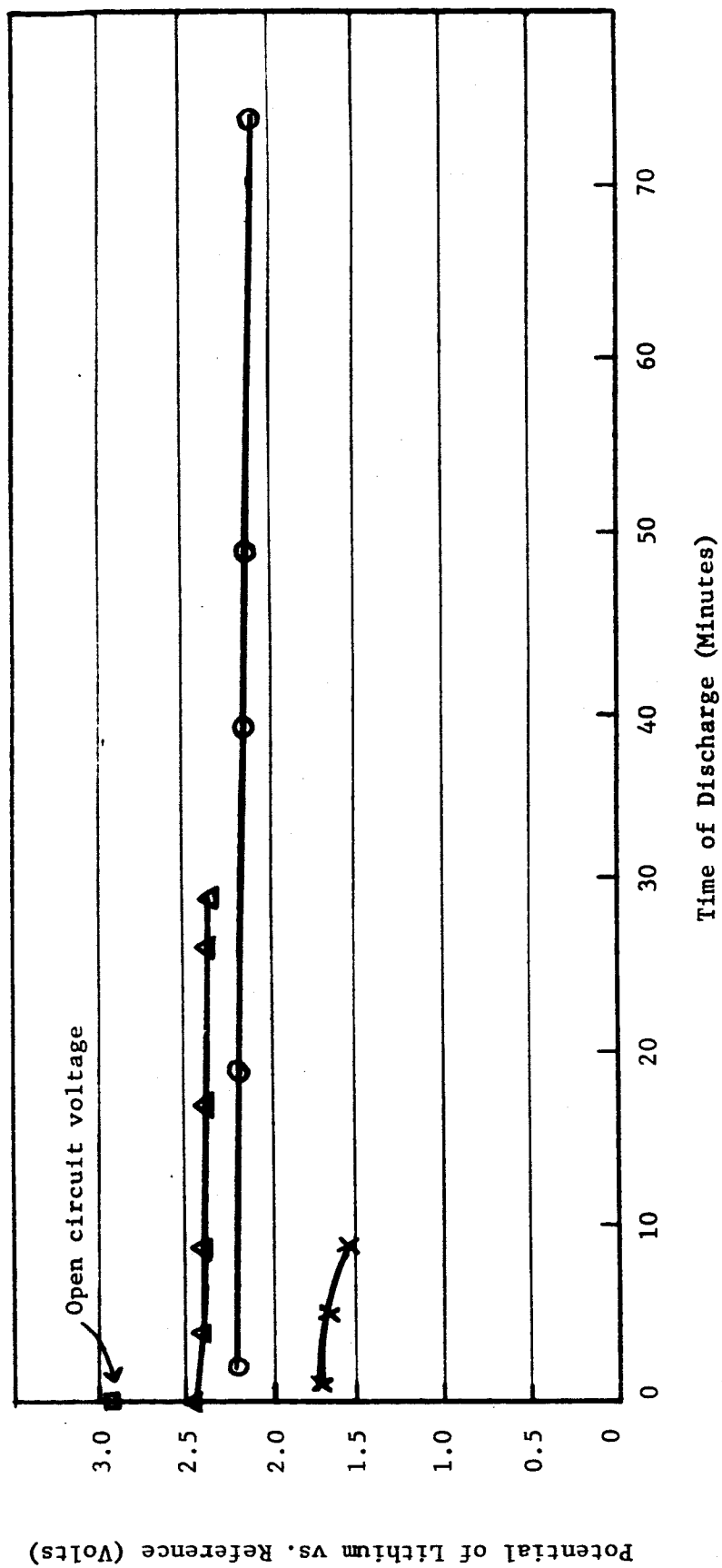
V. Anode-Electrolyte Systems Which Cannot Sustain Discharges at 0.1 ma/cm².
 A. Calcium Anode Systems.

Electrolyte	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm ²)	Voltage of Anode vs. Reference Electrode at Various Current Densities		Further Observations and Remarks
								Initial	Final	
1.	AN	NasbF ₆	Ag/AgCl	Ag/AgCl	-1.59	-0.80 -0.50 ----- -----	0.1 1 10 100	-1.16 Reversed ----- -----	-0.40 ----- ----- -----	Calcium became black in appearance after the discharge at 0.1 ma/cm ² .
2.	AN	*	Ag/AgCl	Ag/AgCl	-1.40	-1.80 -1.85 -1.22 -1.30	0.1 1 10 100	-1.48 -0.52 -0.33 Reversed Polarity	-0.92 -0.81 -0.74 -----	Calcium became slightly darker upon discharge. No gassing at any time. No change in the appearance of the rest of the system.
3.	AN	**	Ag/AgCl	Ag/AgCl	-0.7	-0.75 -0.50 -0.40 -----	0.1 1 10 100	-0.28 -0.16 Reversed Polarity -----	-0.28 -1.00 ----- -----	Calcium became slightly darker during discharge. No change in the appearance of the rest of the system.
4.	NDA	MgCl ₂	Ag/AgCl	Ag/AgCl	-2.12	-2.22 -2.32 -- --	0.1 1 10 100	-1.30 -1.25 -- --	-0.93 Reversed Polarity -- --	No changes in the appearance of the entire system. At 1 ma/cm ² , calcium became polarized appreciably after one minute.

* Di-n-butylammonium hexafluoroarsenate - (n-C₄H₉)₂H₂NAsF₆

** Tetra-n-propylammonium hexafluoroantimonate - (n-C₃H₇)₄NSbF₆

FIGURE 1. DISCHARGE OF LITHIUM/SILVER-SILVER CHLORIDE CELL IN MORPHOLINIUM HEXAFLUOROPHOSPHATE-N-NITROSODIMETHYLAMINE ELECTROLYTE



x = 56 ma/cm² of Lithium.
o = 40 ma/cm² of Lithium.
Δ = 20 ma/cm² of Lithium.

TABLE II. DISCHARGE OF CALCIUM/SILVER-SILVER CHLORIDE CELL IN MORPHOLINIUM HEXAFLUOROPHOSPHATE - ACETONITRILE ELECTROLYTE

Dimensions of calcium electrode = 4 cm x 1 cm.

Active calcium area = 1 cm² (at beginning).

Reference electrode - Ag/AgCl.

Electrolyte concentration - 1 molal.

<u>Time (Minutes)</u>	<u>Calcium vs. Reference (Volts)</u>	<u>Cell Potential (Volts)</u>	<u>Resistance (Ohms)</u>	<u>Current (ma)</u>
0	-1.98	1.98	---	0
6.3	-1.87	1.57	100	15.7
15.0	-1.90	1.65	100	16.5
30.0	-2.00	1.78	100	17.8
41.7	-2.05	1.85	100	18.5
48.4	-2.38	2.15	100	21.5
57.5	-2.31	2.20	100	22.0
59.0	-1.60	1.20	10	120
60.0	-2.50	----	---	0
62.2	-1.85	1.10	10	110
65.0	-2.00	0.98	10	98
66.9	-2.08	0.90	10	90
70.0	-2.08	0.90	10	90
73.4	-2.08	0.92	10	92
78.4	-2.10	0.94	10	94
83.5	-2.10	0.94	10	94
93.5	-2.10	0.95	10	95
103	-2.08	0.95	10	95
117	-2.08	0.95	10	95

TABLE III. DISCHARGE OF CALCIUM/LEAD DIOXIDE
CELL IN MORPHOLINIUM HEXAFLUOROPHOSPHATE -
ACETONITRILE ELECTROLYTE

Dimensions of calcium electrode = 4 cm x 1 cm.

Active calcium area = 1 cm² (at beginning).

Reference electrode - Ag/AgCl.

Electrolyte concentration - 1 molal.

<u>Time (Minutes)</u>	<u>Calcium vs. Reference (Volts)</u>	<u>Cell Potential (Volts)</u>	<u>Resistance (Ohms)</u>	<u>Current (ma)</u>
0	-1.80	2.50	---	0
2.6	-1.80	2.00	100	20.0
3.8	-1.70	1.92	100	19.2
8.3	-1.74	1.82	100	18.2
13.3	-1.80	1.82	100	18.2
18.3	-1.88	1.85	100	18.5
22.5	-2.10	2.30	---	0
23	-1.50	0.85	10	85
25	-1.62	0.52	10	52
31.7	-1.82	0.39	10	39
38.4	-1.88	0.33	10	33
63.4	-1.88	0.30	10	30
70	-1.88	0.29	10	29

TABLE IV. DISCHARGE OF MAGNESIUM/SILVER-SILVER CHLORIDE
CELL IN MORPHOLINIUM HEXAFLUOROPHOSPHATE -
ACETONITRILE ELECTROLYTE

Dimensions of magnesium electrode = 4 cm x 1 cm.

Active magnesium area = 1 cm² (at beginning).

Reference electrode - Ag/AgCl.

Electrolyte concentration - 1 molal.

<u>Time (Minutes)</u>	<u>Magnesium vs. Reference (Volts)</u>	<u>Cell Potential (Volts)</u>	<u>Resistance (Ohms)</u>	<u>Current (ma)</u>
0	-1.20	1.20	---	0
7.9	-1.05	0.81	100	8.1
11.7	-1.05	0.88	100	8.8
16.7	-1.02	0.88	100	8.8
18	-1.25	1.20	---	0
18.2	-0.75	0.42	10	42
21.6	-0.78	0.45	10	45
28.4	-0.75	0.43	10	43
38.4	-0.75	0.41	10	41
39.4	-1.80	1.72	---	0

TABLE V. ELECTROCHEMICAL HALF-CELL
SCREENING OF CATHODES

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TABLE V. ELECTROCHEMICAL HALF-CELL SCREENING OF CATHODES

A. Partially Reduced Cupric Fluoride Cathodes.

Experimental Details: Cathode matrix - nickel expanded metal. Matrix size - 1 cm² (circular).
 Composition of cathode mix: 98% CuF₂, 2% Solka-Floc.
 Dry-press formation pressure: 6,000 lb./cm².
 Thickness of dry-pressed cathode pellets: ca. 1/8".
 Temperature of hydrogen gas for reduction of CuF₂ pellet: 130°C.
 Electrolyte: saturated MgCl₂ - N-nitrosodimethylamine.
 Counter electrode: lithium. Reference electrode: Ag/AgCl.
 Duration of discharge at each current density: 5 minutes.

Cathode Number	Time of Reduction With H ₂ Gas	Voltage of Cathode vs. Reference Electrode at Various Current Densities (ma/cm ²)						Observations
		0	0.1	1	2	10	10	
1	1 hour	Initial	+0.22	+0.38	+0.46	<-2.0	---	The color of the electrolyte solutions changed from yellow to green during discharge of all of these systems.
		Final	+0.17	+0.65	+0.42			
2.	1 hour	Initial	+0.18	+0.14	-0.09	---	<-5.0	It should be noted that in some cases the discharge voltage became more positive than the open circuit voltage, whereas the reverse phenomenon would normally be expected.
		Final	+0.18	+0.13	-0.14			
3.	1 hour	Initial	+0.23	+0.18	-0.13	---	<-10.0	
		Final	+0.16	+0.13	-0.25			
4.	1.5 hours	Initial	+0.16	+0.18	+0.26	<-2.0	---	
		Final	+0.12	+0.22	+0.48			
5.	1.5 hours	Initial	+0.12	+0.07	-0.90	---	---	
		Final	+0.12	+0.08	---			

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

A. Partially Reduced Cupric Fluoride Cathodes. (Continued)

Cathode Number	Time of Reduction With H ₂ Gas	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities (ma/cm ²)					Observations
			0	0.1	1	5	10	
6.	1.5 hours	Initial	+0.13	+0.08	-0.70	---	----	
		Final	+0.13	+0.09	---			
7.	2 hours	Initial	+0.20	+0.25	+0.09	< -2.0	----	
		Final	+0.15	+0.40	+0.31			

B. Lead Dioxide Cathodes.

Experimental Details: Cathode matrix - nickel expanded metal. Matrix size - 1 cm² (circular).
 Composition of cathode mix: 95% PbO₂, 5% Solka-Floc.
 Thickness of dry-pressed cathode pellets: ca. 1/8".
 Counter electrode: lithium. Reference electrode: Ag/AgCl.
 Duration of discharge at each current density: 5 minutes.

Cathode Number	Formation Pressure (lbs./cm ²)	Electrolyte	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities (ma/cm ²)					
				0	0.1	1	5	10	15
1.	4,000	MgCl ₂ -NDA*	Initial	+0.69	+0.77	+0.87	+0.77	+0.40	-0.10
			Final	+0.18	+0.88	+1.02	+0.56	-0.04	-0.64
2.	4,000	MgCl ₂ -DMF**	Initial	+0.56	+0.96	+0.84	+0.58	+0.32	-0.10
			Final	+0.30	+0.99	+0.80	+0.51	+0.08	-0.43
3.	2,000	MgCl ₂ -NDA*	Initial	+0.76	+0.88	+0.85	+0.72	+0.48	+0.12
			Final	+0.28	+0.92	+1.11	+0.69	-0.04	-0.25

In all three of the half-cells described above, the solutions changed to a green color during discharge.

* NDA = N-nitrosodimethylamine.

** DMF = Dimethylformamide

TABLE VI. CHEMICAL STABILITY OF ANODE MATERIALS
IN ELECTROLYTE SOLUTIONS

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I. Tests of Twenty-Four Hours Duration.

A. Stability in Acetone Solutions.

TABLE VI. CHEMICAL STABILITY OF ANODE MATERIALS
IN ELECTROLYTE SOLUTIONS

I. Tests of Twenty-Four Hours Duration.
A. Stability in Acetone Solutions.

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. KPF_6	Ca	Formation of dull, thin film at calcium surface.
	Li	Appreciable consumption of lithium; large amount of gelatinous material formed.
	Mg	Slight formation of white spots on magnesium surface.
2. NaPF_6	Ca	Formation of dull, thin film at calcium surface.
	Li	Appreciable consumption of lithium; large amount of gelatinous material formed.
	Mg	Formation of black spots on magnesium; solution yellowed.
3. $\text{O} \begin{array}{l} \diagup \text{CH}_2\text{CH}_2 \diagdown \\ \diagdown \text{CH}_2\text{CH}_2 \diagup \end{array} \text{NH}_2\text{PF}_6$ (Morpholinium hexafluorophosphate)	Ca	Appreciable consumption of calcium; formation of white solid.
	Li	Appreciable consumption of lithium; large amount of gelatinous material formed.
	Mg	Slight gas evolution observed; no other change.
4. $(\text{C}_6\text{H}_5)(\text{CH}_3)_3\text{NPF}_6$ (N-Phenyl N,N,N-trimethyl- ammonium hexafluorophosphate)	Ca	Formation of dull, thin film at calcium surface.
	Li	Appreciable consumption of lithium; large amount of gelatinous material formed.
	Mg	Formation of gelatinous material and blue color in solution.

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

I. Tests of Twenty-Four Hours Duration.
 A. Stability in Acetone Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
5. $(n-C_3H_7)_4NPF_6$ (Tetra-n-propylammonium hexafluorophosphate)	Ca	Appreciable consumption of calcium; formation of gelatinous material.
	Li	Appreciable consumption of lithium; large amount of gelatinous material formed.
	Mg	No change.

I. Tests of Twenty-Four Hours Duration.

B. Stability in Acetonitrile Solutions.

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

I. Tests of Twenty-Four Hours Duration.
 B. Stability in Acetonitrile Solutions.

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. KPF_6	Ca	No change.
	Li	Appreciable consumption of lithium; cloudy solution.
	Mg	No change.
2. $NaPF_6$	Ca	No change.
	Li	Appreciable consumption of lithium; cloudy solution.
	Mg	No change.
3. $NaSbF_6$	Ca	No change.
	Li	Appreciable consumption of lithium; cloudy solution.
	Mg	No change.
4. $(n-C_3H_7)_4NBF_4$ (Tetra-n-propylammonium tetrafluoroborate)	Ca	Formation of thick film at calcium surface.
	Li	Appreciable consumption of lithium; cloudy solution.
	Mg	Formation of thick film at magnesium surface; solid precipitated.
5. $\begin{matrix} O & \text{CH}_2\text{CH}_2 & \\ & \diagdown & \diagup \\ & \text{CH}_2\text{CH}_2 & \end{matrix} NH_2PF_6$ (Morpholinium hexafluorophosphate)	Ca	Formation of thick film at calcium surface; solid precipitated.
	Li	Appreciable consumption of lithium; vigorous reaction.
	Mg	Formation of thick film at magnesium surface; solid precipitated.

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

I. Tests of Twenty-Four Hours Duration.
 B. Stability in Acetonitrile Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
6. $(C_6H_5)(CH_3)_3NPF_6$ (N-Phenyl N,N,N-trimethyl- ammonium hexafluorophosphate)	Ca	No change.
	Li	Appreciable consumption of lithium; solid precipitated.
	Mg	No change.
7. $(n-C_3H_7)_4NPF_6$ (Tetra-n-propylammonium hexafluorophosphate)	Ca	Appreciable consumption of calcium; solution yellowed.
	Li	Vigorous reaction; heat, gas, and solid product formed.
	Mg	No change.
8. $(n-C_4H_9)_2HNAsF_6$ (Di-n-butylammonium hexafluoroarsenate)	Ca	No change.
	Li	Appreciable consumption of lithium; black solid precipitated.
	Mg	No change.
9. $(n-C_3H_7)_3HNAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	Ca	Formation of thick film at calcium surface; solid precipitated.
	Li	Appreciable consumption of lithium; white solid precipitated.
	Mg	Appreciable consumption of magnesium; solid precipitated.
10. $(n-C_3H_7)_4NAsF_6$ (Tetra-n-propylammonium hexafluoroarsenate)	Ca	Formation of thick film at calcium surface.
	Li	Appreciable consumption of lithium; brown solid produced.
	Mg	Formation of thick, black film at magnesium surface.

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

I. Tests of Twenty-Four Hours Duration.
B. Stability in Acetonitrile Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
11. $(n-C_3H_7)_4NSbF_6$ (Tetra-n-propylammonium hexafluoroantimonate)	Ca	No change.
	Li	Appreciable consumption of lithium; black solid formed.
	Mg	Formation of thick, black film at magnesium surface.
12. $AlCl_3$	Ca	Calcium surface partially darkened.
	Mg	Formation of black spots at magnesium surface.
13. $(CH_3)_4NPF_6$ (Tetramethylammonium hexafluorophosphate)	Ca	No change.
14. $(n-C_4H_9)_4NCl$ (Tetra-n-butylammonium chloride)	Ca	No change.
15. $LiCl$	Ca	No change.
	Mg	No change.

I. Tests of Twenty-Four Hours Duration.

C. Stability in Dimethyl Cyanamide Solutions.

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

I. Tests of Twenty-Four Hours Duration.

C. Stability in Dimethyl Cyanamide Solutions.

1. AlCl_3	Ca	Formation of thick, white film at calcium surface; solution brown.
	Li	Appreciable consumption of lithium; solution black.
	Mg	Appreciable consumption of magnesium; brown cloudy solution.
2. KAsF_6	Ca	Formation of brown spots at calcium surface.
	Li	Formation of thick, gray film at lithium surface.
	Mg	No change.
3. KPF_6	Ca	No change.
	Li	Formation of thick, gray film at lithium surface.
	Mg	No change.
4. NaPF_6	Ca	Slight blackening at calcium surface.
	Li	Formation of dull thin film at lithium surface; solution yellow.
	Mg	No change.
5. NaSbF_6	Ca	Slight blackening at calcium surface.
	Li	Formation of thin, dull film at lithium surface; white solid precipitated.
	Mg	Formation of dull, thin film at magnesium surface.
6. $(n\text{-C}_3\text{H}_7)_4\text{NBF}_4$ (Tetra-n-propylammonium tetrafluoroborate)	Ca	Tan spots formed at calcium surface; solution tan.
	Li	Formation of thick white film at lithium surface; solution black.
	Mg	Formation of thick, dull film at magnesium surface.

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

I. Tests of Twenty-Four Hours Duration
 C. Stability in Dimethyl Cyanamide Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
7. $\text{O} \begin{array}{l} \text{CH}_2\text{CH}_2 \\ \text{CH}_2\text{CH}_2 \end{array} \text{NH}_2\text{PF}_6$ (Morpholinium hexafluorophosphate)	Ca Li	Calcium blackened; solid precipitated. Appreciable consumption of lithium.
8. $(\text{C}_6\text{H}_5)(\text{CH}_3)_3\text{NPF}_6$ (N-Phenyl N,N,N-trimethyl- ammonium hexafluorophosphate)	Ca Li Mg	Thick gray film formed at calcium surface; solution gray. Appreciable consumption of lithium; solution tan. Formation of thick, dull film and black spots at magnesium surface.
9. $(n\text{-C}_3\text{H}_7)_4\text{NPF}_6$ (Tetra-n-propylammonium hexafluorophosphate)	Ca Li Mg	Formation of thin, gray film at calcium surface; yellow solid precipitated. Formation of gelatinous material at lithium surface. No change.
10. $(p\text{-C}_{12}\text{H}_{25}\text{C}_6\text{H}_4\text{CH}_2)(\text{CH}_3)_3\text{NPF}_6$ (N-(p-Dodecylbenzyl)N,N,N- trimethylammonium hexafluorophosphate)	Ca Li Mg	Formation of thick, gray film at calcium surface; solution yellow. Formation of thick, white film at lithium surface; solution brown. Formation of a few spots at magnesium surface.
11. $(n\text{-C}_4\text{H}_9)_2\text{H}_2\text{NAsF}_6$ (Di-n-butylammonium hexafluoroarsenate)	Ca Li	Formation of gray film at calcium surface. Appreciable consumption of lithium; solution brown.

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

I. Tests of Twenty-Four Hours Duration
C. Stability in Dimethyl Cyanamide Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
12. $(n-C_3H_7)_3HNAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	Ca	Formation of thick, gray film at lithium surface.
	Li	Appreciable consumption of lithium; solid precipitated.
	Mg	Formation of a few gray spots at magnesium surface.
13. $(n-C_3H_7)_4NAsF_6$ (Tetra-n-propylammonium hexafluoroarsenate)	Ca	Formation of gray film at calcium surface; solid precipitated.
	Li	Formation of thick, white film at lithium surface; gelatinous material present.
14. $(C_6H_5CH_2)(CH_3)_3NSbF_6$ (N-Benzyl N,N,N-trimethyl-ammonium hexafluoroantimonate)	Ca	Formation of black film at calcium surface; gelatinous material formed.
	Li	Formation of thick, white film at lithium surface; solid precipitated.
	Mg	Appreciable consumption of magnesium; brown solid precipitated.

I. Tests of Twenty-Four Hours Duration.

D. Stability in Dimethylformamide Solutions.

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

I. Tests of Twenty-Four Hours Duration.
D. Stability in Dimethylformamide Solutions.

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. CsPF ₆	Ca	No change.
	Li	Formation of gelatinous material at lithium surface; solution orange.
	Mg	No change.
2. FeCl ₃	Ca	Appreciable consumption of calcium; solution opaque.
	Li	Appreciable consumption of lithium; solution opaque.
	Mg	Appreciable consumption of magnesium; solution opaque.
3. KAsF ₆	Ca	No change.
	Li	Formation of black film at lithium surface; solution orange.
	Mg	No change.
4. K ₃ Cr(SCN) ₆	Ca	Formation of thick gray film at calcium surface.
	Li	Formation of gelatinous material at lithium surface.
	Mg	No change.
5. KI	Ca	No change.
	Li	Formation of gelatinous material at lithium surface.
	Mg	No change.
6. KPF ₆	Ca	No change.
	Li	Formation of gelatinous material at lithium surface.
	Mg	No change.

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

I. Tests of Twenty-Four Hours Duration.
D. Stability in Dimethylformamide Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
7. MgCl ₂	Ca	No change.
	Li	Appreciable consumption of lithium; solution yellow.
	Mg	No change.
8. NH ₄ SO ₃ F	Ca	Slight darkening of calcium surface.
	Li	Formation of thick, gray film at lithium surface; white solid precipitated
	Mg	Slight darkening of magnesium surface; spots at surface.
9. NaBF ₄	Ca	No change.
	Li	Solution colored.
	Mg	No change.
10. NaPF ₆	Ca	No change.
	Li	Appreciable consumption of lithium; solution yellow.
	Mg	No change.
11. NaSbF ₆	Ca	No change.
	Li	Appreciable consumption of lithium; solution orange.
	Mg	No change.
12. (n-C ₃ H ₇) ₄ NBF ₄ (Tetra-n-propylammonium tetrafluoroborate)	Ca	Formation of gray film at calcium surface.
	Li	Formation of gelatinous material at lithium surface.
	Mg	Formation of gray film at magnesium surface; holes in magnesium.

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

I. Tests of Twenty-Four Hours Duration.

D. Stability in Dimethylformamide Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
13. $(\text{CH}_3)_4\text{NPF}_6$ (Tetramethylammonium hexafluorophosphate)	Ca	No change.
	Li	Formation of gelatinous material at lithium surface; solution orange.
	Mg	No change.
14. $\text{O} \begin{array}{l} \diagup \text{CH}_2\text{CH}_2 \diagdown \\ \diagdown \text{CH}_2\text{CH}_2 \diagup \end{array} \text{NH}_2\text{PF}_6$ (Morpholinium hexafluorophosphate)	Ca	Very slow gas evolution.
	Li	Gas evolution at moderate rate.
	Mg	No change.
15. $(\text{C}_6\text{H}_5)(\text{CH}_3)_3\text{NPF}_6$ (N-Phenyl N,N,N-trimethylammonium hexafluorophosphate)	Ca	No change.
	Li	Formation of gelatinous material at lithium surface; orange solution.
	Mg	No change.
16. $(n\text{-C}_3\text{H}_7)_4\text{NPF}_6$ (Tetra-n-propylammonium hexafluorophosphate)	Ca	Formation of a few dark spots at calcium surface.
	Li	Appreciable consumption of lithium.
	Mg	Formation of thin, dark film at magnesium surface.
17. $(p\text{-C}_{12}\text{H}_{25}\text{C}_6\text{H}_4\text{CH}_2)(\text{CH}_3)_3\text{NPF}_6$ (N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate)	Ca	No change.
	Li	Formation of gelatinous material at lithium surface; solution orange.
	Mg	No change.
18. $(n\text{-C}_4\text{H}_9)_2\text{H}_2\text{NAsF}_6$ (Di-n-butylammonium hexafluoroarsenate)	Ca	No change.
	Li	Formation of thick, dull film at lithium surface.
	Mg	No change.

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

I. Tests of Twenty-Four Hours Duration.
D. Stability in Dimethylformamide Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
19. $(n-C_3H_7)_3HNAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	Ca	Formation of black spots at calcium surface.
	Li	Formation of gelatinous material at lithium surface.
	Mg	No change.
20. $(n-C_3H_7)_4NAsF_6$ (Tetra-n-propylammonium hexafluoroarsenate)	Ca	Formation of a few brown spots at calcium surface.
	Li	Appreciable swelling of lithium; gelatinous product.
	Mg	Formation of a few brown spots at magnesium surface.
21. $(C_6H_5CH_2)(CH_3)_3NSbF_6$ (N-Benzyl N,N,N-trimethylammonium hexafluoroantimonate)	Ca	Solution yellow.
	Li	Appreciable consumption of lithium; solution brown.
	Mg	Magnesium black and brittle.
22. $(n-C_3H_7)_4NSbF_6$ (Tetra-n-propylammonium hexafluoroantimonate)	Ca	Calcium blackened; solid precipitated.
	Li	Appreciable consumption of lithium; solution black and cloudy.
	Mg	Magnesium blackened; solution brown and cloudy.
23. $(C_6H_5)_4AsCl$ (Tetraphenyl arsonium chloride)	Ca	No change.
	Li	Formation of gelatinous material at lithium surface; solution orange.
	Mg	No change.

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

I. Tests of Twenty-Four Hours Duration.
D. Stability in Dimethylformamide Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
24. LiCl	Ca	No change.
	Li	Formation of white, gelatinous material at lithium surface.
	Mg	No change.
25. (n-C ₄ H ₉) ₄ NCl (Tetra-n-butylammonium chloride)	Ca	No change.

I. Tests of Twenty-Four Hours Duration.

- E. Stability in Ethylene Carbonate (80 Weight %) - Propylene Carbonate (20 Weight %) Solutions.

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

I. Tests of Twenty-Four Hours Duration.
 E. Stability in Ethylene Carbonate (80 Weight %)-Propylene
Carbonate (20 Weight %) Solutions.

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. AlCl_3	Ca	Calcium surface appeared to be brighter; no other changes.
	Li	Formation of thin, gray film at lithium surface.
	Mg	Slow evolution of gas from magnesium surface; no other changes.
2. KAsF_6	Ca	Slight darkening of calcium surface.
	Li	Formation of dull, thin film at lithium surface.
	Mg	No change.
3. $\text{K}_3\text{Cr}(\text{SCN})_6$	Ca	Appreciable consumption of calcium; gelatinous material present.
	Li	Appreciable consumption of lithium; gelatinous material present.
	Mg	No change.
4. KPF_6	Ca	No change.
	Li	No change.
	Mg	No change.
5. MgCl_2	Li	No change.
6. $\text{O} \begin{array}{l} \text{CH}_2\text{CH}_2 \\ \text{CH}_2\text{CH}_2 \end{array} \text{NH}_2\text{PF}_6$ (Morpholinium hexafluorophosphate)	Ca	Formation of thin, dark film at calcium surface.
	Li	Formation of thick film and gelatinous product at lithium surface.
	Mg	No change.

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

I. Tests of Twenty-Four Hours Duration.
 E. Stability in Ethylene Carbonate (80 Weight %)-Propylene
 Carbonate (20 Weight %) Solutions. (Continued)

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
7. $(C_6H_5)(CH_3)_3NPF_6$ (N-Phenyl N,N,N-trimethyl- ammonium hexafluorophosphate)	Ca	No change.
	Li	No change.
	Mg	No change.
8. $(n-C_3H_7)_4NPF_6$ (Tetra-n-propylammonium hexafluorophosphate)	Ca	No change.
	Li	Thin gray film formed only at edges of lithium strip.
	Mg	No change.
9. $(n-C_4H_9)_4NCl$ (Tetra-n-butylammonium chloride)	Ca	No change.
	Li	Edges of lithium blackened slightly; no other change.
10. LiCl	Ca	No change.
	Li	No change.
	Mg	No change.
11. $(CH_3)_4NPF_6$ (Tetramethylammonium hexafluorophosphate)	Ca	No change.
	Li	No change.

I. Tests of Twenty-Four Hours Duration.

F. Stability in N-Nitrosodimethylamine Solutions.

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

I. Tests of Twenty-Four Hours Duration.

F. Stability in N-Nitrosodimethylamine Solutions.

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. AlCl_3	Ca	No change.
	Li	No change.
	Mg	No change.
2. CsPF_6	Ca	No change.
	Li	Formation of dark, thin film at lithium surface.
	Mg	No change.
3. FeCl_3	Ca	Appreciable consumption of calcium.
	Li	Appreciable consumption of lithium.
	Mg	Appreciable consumption of magnesium.
4. KAsF_6	Ca	Slight formation of brown film at calcium surface.
	Li	Formation of thick, gray film at lithium surface.
	Mg	No change.
5. $\text{K}_3\text{Cr}(\text{SCN})_6$	Ca	Appreciable consumption of calcium.
	Li	Appreciable consumption of lithium.
	Mg	No change.
6. KI	Ca	No change.
	Li	Formation of thick, dark film at lithium surface.
	Mg	No change.

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

I. Tests of Twenty-Four Hours Duration.

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

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
7. KPF_6	Ca	No change.
	Li	Formation of thick, dark film at lithium surface.
	Mg	No change.
8. $MgCl_2$	Ca	No change.
	Li	No change.
	Mg	No change.
9. NH_4SO_3F	Ca	Formation of thick, gray film at calcium surface.
	Li	Very slight consumption of lithium.
	Mg	No change.
10. $NaBF_4$	Ca	No change.
	Li	Formation of thick, gray film at lithium surface.
	Mg	No change.
11. $NaPF_6$	Ca	No change.
	Li	Formation of thin film at lithium surface.
	Mg	No change.
12. $NaSbF_6$	Ca	No change.
	Li	Formation of tan film at edges of lithium strip.
	Mg	No change.

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

I. Tests of Twenty-Four Hours Duration.

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

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
19. $(C_6H_5CH_2)(CH_3)_3NSbF_6$ (N-Benzyl N,N,N-trimethyl- ammonium hexafluoroantimonate)	Ca	No change.
	Li	No change.
	Mg	Formation of black film at magnesium surface; holes in magnesium.
20. $(n-C_4H_9)_4NCl$ (Tetra-n-butylammonium chloride)	Ca	Formation of thin, gray film at calcium surface.
	Li	Formation of thick, white film at lithium surface.
	Mg	No change.
21. $(C_6H_5)_4PCl$ (Tetraphenylphosphonium chloride)	Ca	Formation of thin, dull film at calcium surface.
	Li	Formation of thick, gray film at lithium surface.
	Mg	No change.
22. $(CH_3)_4NPF_6$ (Tetramethylammonium hexafluorophosphate)	Ca	No change.
	Li	No change.
23. LiCl	Ca	No change.
	Li	No change.
	Mg	No change.

I. Tests of Twenty-Four Hours Duration.

G. Stability in Propylene Carbonate Solutions.

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

I. Tests of Twenty-Four Hours Duration.
G. Stability in Propylene Carbonate Solutions.

<u>Solute</u>	<u>Anode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. $\begin{matrix} \text{O} < \text{CH}_2\text{CH}_2 > \text{NH}_2\text{PF}_6 \\ & \text{CH}_2\text{CH}_2 & \end{matrix}$ (Morpholinium hexafluorophosphate)	Ca	Formation of gelatinous material at calcium surface.
	Li	Appreciable consumption of lithium; gelatinous material formed.
	Mg	No change.
2. $(\text{C}_6\text{H}_5)(\text{CH}_3)_3\text{NPF}_6$ (N-Phenyl N,N,N-trimethyl- ammonium hexafluorophosphate)	Ca	Solution tinted blue.
	Li	Formation of gelatinous material at lithium surface.
	Mg	Formation of thin film at magnesium surface.

II. Tests of Two Weeks Duration.

A. Stability in Acetonitrile Solutions.

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

II. Tests of Two Weeks Duration.

A. Stability in Acetonitrile Solutions.

Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance ($\text{ohm}^{-1} \text{cm}^{-1}$)		Visible Change After Two Week Exposure
				Exposed Electrolyte	Blank Electrolyte	
1. KPF ₆	Ca	Before Exposure	1.1338	3.10×10^{-2} (28°C)	3.10×10^{-2} (28°C)	Calcium surface appeared to be slightly duller than initially.
		After Two Weeks	1.1380	3.53×10^{-2} (24°C)	3.48×10^{-2} (24°C)	
2. (C ₆ H ₅)(CH ₃) ₃ NPF ₆ (N-Phenyl N,N,N-trimethylammonium hexafluorophosphate)	Ca	Before Exposure	1.0417	3.96×10^{-2} (26°C)	3.94×10^{-2} (27°C)	A few small brown spots formed at calcium surface.
		After Two Weeks	1.0484	3.97×10^{-2} (24°C)	3.94×10^{-2} (25°C)	

II. Tests of Two Weeks Duration.

B. Stability in Dimethylformamide Solutions.

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

Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance ($\text{ohm}^{-1} \text{cm}^{-1}$)		Visible Change After Two Week Exposure
				Exposed Electrolyte	Blank Electrolyte	
1. MgCl_2	Ca	Before Exposure	2.1523	1.01×10^{-2}	1.01×10^{-2}	No change.
		After Two Weeks	2.2400	1.01×10^{-2}	1.01×10^{-2}	
	Mg	Before Exposure	0.1700	1.01×10^{-2}	1.01×10^{-2}	No change.
		After Two Weeks	0.1892	1.01×10^{-2}	1.01×10^{-2}	
2. $\text{NH}_4\text{SO}_3\text{F}$	Ca	Before Exposure	2.1124	2.09×10^{-2}	2.09×10^{-2}	Entire surface of calcium blackened.
		After Two Weeks	2.1602	2.04×10^{-2}	2.04×10^{-2}	
	Mg	Before Exposure	0.2060	2.09×10^{-2}	2.09×10^{-2}	Large number of black spots formed on magnesium surface.
		After Two Weeks	0.4117	2.01×10^{-2}	2.04×10^{-2}	
3. NaBF_4	Ca	Before Exposure	1.7440	2.02×10^{-2}	2.02×10^{-2}	No change.
		After Two Weeks	1.7715	2.05×10^{-2}	2.04×10^{-2}	
	Mg	Before Exposure	0.1528	2.02×10^{-2}	2.02×10^{-2}	A few small black spots formed on magnesium surface.
		After Two Weeks	0.1829	2.06×10^{-2}	2.04×10^{-2}	
4. NaPF_6	Ca	Before Exposure	2.1598	2.30×10^{-2}	2.30×10^{-2}	No change.
		After Two Weeks	2.1673	2.25×10^{-2}	2.22×10^{-2}	
	Mg	Before Exposure	0.1542	2.30×10^{-2}	2.30×10^{-2}	No change.
		After Two Weeks	0.2088	2.27×10^{-2}	2.22×10^{-2}	

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

II. Tests of Two Weeks Duration.

B. Stability in Dimethylformamide Solutions. (Continued)

Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance ($\text{ohm}^{-1} \text{cm}^{-1}$)		Visible Change After Two Week Exposure
				Exposed Electrolyte	Blank Electrolyte	
5. NaSbF_6	Ca	Before Exposure	2.0969	2.20×10^{-2}	2.20×10^{-2}	Entire surface of calcium blackened.
		After Two Weeks	2.1417	2.20×10^{-2}	2.23×10^{-2}	
	Mg	Before Exposure	0.1324	2.20×10^{-2}	2.20×10^{-2}	No change.
		After Two Weeks	0.1767	2.20×10^{-2}	2.23×10^{-2}	
6. $(\text{n-C}_3\text{H}_7)_4\text{NPF}_6$ (Tetra-n-propylammonium hexafluoro-phosphate)	Ca	Before Exposure	2.4111	1.97×10^{-2}	1.97×10^{-2}	A few black spots formed on calcium surface.
		After Two Weeks	2.5189	1.99×10^{-2}	1.98×10^{-2}	
	Mg	Before Exposure	0.1383	1.97×10^{-2}	1.97×10^{-2}	Appreciable blackening of magnesium; holes in magnesium.
		After Two Weeks	0.1220	1.89×10^{-2}	1.98×10^{-2}	
7. KPF_6	Ca	Before Exposure	1.7519	2.74×10^{-2} (27°C)	2.75×10^{-2} (27°C)	No change.
		After Two Weeks	1.7588	2.25×10^{-2} (25°C)	2.27×10^{-2} (25°C)	
8. $(\text{CH}_3)_4\text{NPF}_6$ (Tetramethylammonium hexafluorophosphate)	Ca	Before Exposure	1.6560	1.19×10^{-2} (17°C)	1.09×10^{-2} (27°C)	No change.
		After Two Weeks	1.6617	1.12×10^{-2} (25°C)	1.11×10^{-2} (25°C)	

II. Tests of Two Weeks Duration.

- C. Stability in Ethylene Carbonate (80 Weight %)-Propylene Carbonate (20 Weight %) Solutions.

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

Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm ⁻¹ cm ⁻¹)		Visible Change After Two Week Exposure
				Exposed Electrolyte	Blank Electrolyte	
1. MgCl ₂	Li	Before Exposure	0.6251	4.52 x 10 ⁻³ (27°C)	3.70 x 10 ⁻³ (27°C)	No change.
		After Two Weeks	0.6528	2.59 x 10 ⁻³ (25°C)	2.85 x 10 ⁻³ (25°C)	
2. KPF ₆	Ca	Before Exposure	1.1016	1.12 x 10 ⁻² (27°C)	1.12 x 10 ⁻² (27°C)	Calcium surface appeared to be slightly duller than initially.
		After Two Weeks	1.1136	1.07 x 10 ⁻² (25°C)	1.09 x 10 ⁻² (25°C)	
3. (n-C ₄ H ₉) ₄ NCl (Tetra-n- butylammonium chloride)	Li	Before Exposure	0.6671	8.20 x 10 ⁻³ (27°C)	8.15 x 10 ⁻³ (27°C)	Slight, partial darkening of lithium surface.
		After Two Weeks	0.7404	7.70 x 10 ⁻³ (25°C)	7.74 x 10 ⁻³ (25°C)	
4. (n-C ₃ H ₇) ₄ NPF ₆ (Tetra-n-propyl- ammonium hexa- fluorophosphate)	Li	Before Exposure	0.6379	1.25 x 10 ⁻² (28°C)	1.25 x 10 ⁻² (28°C)	Formation of thick, gray film at lithium surface. Tan solid formed in solution.
		After Two Weeks	0.8161	8.30 x 10 ⁻³ (25°C)	1.16 x 10 ⁻² (25°C)	

II. Tests of Two Weeks Duration.

D. Stability in N-Nitrosodimethylamine Solutions.

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

II. Tests of Two Weeks Duration.

D. Stability in N-Nitrosodimethylamine Solutions.

Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm ⁻¹ cm ⁻¹)		Visible Change After Two Week Exposure
				Exposed Electrolyte	Blank Electrolyte	
1. AlCl ₃	Li	Before Exposure	0.6442	2.06 x 10 ⁻² (27°C)	2.12 x 10 ⁻² (27°C)	Color of solution changed from yellow to amber.
		After Two Weeks	0.7602	1.92 x 10 ⁻² (24°C)	2.02 x 10 ⁻² (24°C)	
2. MgCl ₂	Ca	Before Exposure	1.0533	1.09 x 10 ⁻² (28°C)	1.09 x 10 ⁻² (28°C)	No change.
		After Two Weeks	1.0788	9.47 x 10 ⁻³ (24°C)	9.42 x 10 ⁻³ (24°C)	
	Li	Before Exposure	0.6608	1.09 x 10 ⁻² (28°C)	1.09 x 10 ⁻² (28°C)	No change.
		After Two Weeks	0.6845	8.13 x 10 ⁻³ (24°C)	9.42 x 10 ⁻³ (24°C)	
3. KPF ₆	Ca	Before Exposure	1.0293	2.00 x 10 ⁻² (28°C)	1.99 x 10 ⁻² (28°C)	Calcium surface appeared to be slightly duller than initially.
		After Two Weeks	1.0449	1.95 x 10 ⁻² (25°C)	1.92 x 10 ⁻² (25°C)	

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

II. Tests of Two Weeks Duration.
D. Stability in N-Nitrosodimethylamine Solutions. (Continued)

Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm ⁻¹ cm ⁻¹) Exposed Electrolyte	Blank Electrolyte	Visible Change After Two Week Exposure
4. (n-C ₄ H ₉) ₄ NCl (Tetra-n-butylammonium chloride)	Ca	Before Exposure	1.0058	1.15 x 10 ⁻² (28°C)	1.16 x 10 ⁻² (27°C)	Calcium surface appeared to be very slightly darker than initially.
		After Two Weeks	1.0073	1.10 x 10 ⁻² (25°C)	1.11 x 10 ⁻² (25°C)	
5. (n-C ₃ H ₇) ₄ NPF ₆ (Tetra-n-propylammonium hexafluorophosphate)	Ca	Before Exposure	1.5178	2.27 x 10 ⁻² (27°C)	2.25 x 10 ⁻² (27°C)	Calcium surface dull and partially blackened. A small amount of a solid product also appeared at the surface.
		After Two Weeks	1.7518	2.29 x 10 ⁻² (25°C)	2.25 x 10 ⁻² (27°C)	
6. O ₂ CH ₂ CH ₂ NH ₂ PF ₆ (Morpholinium hexafluorophosphate)	Ca	Before Exposure	1.1857	2.86 x 10 ⁻² (27°C)	2.87 x 10 ⁻² (27°C)	Formation of large amount of black product at calcium surface. White solid formed in solution.
		After Two Weeks	1.0479	2.70 x 10 ⁻² (25°C)	2.70 x 10 ⁻² (25°C)	

TABLE VII. CHEMICAL STABILITY OF CATHODE MATERIALS
IN NONAQUEOUS SOLVENTS

<u>Cathode Material</u>	<u>Specific Conductance (ohm⁻¹ cm⁻¹) At 25°C - After Two Week Exposure</u>		<u>Visible Change After Two Week Exposure</u>
	<u>Exposed Solvent</u>	<u>Blank Solvent</u>	
a. <u>Stability in Acetonitrile</u>			
CoF ₃	1.13 x 10 ⁻⁴	4.71 x 10 ⁻⁶	No change.
MnO ₂	4.74 x 10 ⁻⁶		No change.
CuF ₂	1.02 x 10 ⁻⁵		No change.
b. <u>Stability in Dimethylformamide</u>			
CoF ₃	1.64 x 10 ⁻⁴	1.54 x 10 ⁻⁶	CoF ₃ somewhat darkened.
MnO ₂	4.86 x 10 ⁻⁶		No change.
CuF ₂	6.80 x 10 ⁻⁶		No change.
c. <u>Stability in Ethylene Carbonate (80 Weight %)- Propylene Carbonate (20 Weight %)</u>			
CoF ₃	2.06 x 10 ⁻⁴	4.92 x 10 ⁻⁶	Solution colored faintly red.
MnO ₂	3.48 x 10 ⁻⁶		No change.
CuF ₂	6.60 x 10 ⁻⁶		No change.
d. <u>Stability in N-Nitrosodimethylamine</u>			
CoF ₃	5.90 x 10 ⁻⁵	2.48 x 10 ⁻⁵	CoF ₃ somewhat darkened.
MnO ₂	3.31 x 10 ⁻⁵		No change.
CuF ₂	2.49 x 10 ⁻⁵		No change.

TABLE VIII. CHEMICAL STABILITY OF CATHODE
MATERIALS IN ELECTROLYTE SOLUTIONS

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I. Tests of Twenty-Four Hours Duration.

A. Stability in Acetonitrile Solutions.

TABLE VIII. CHEMICAL STABILITY OF CATHODE
MATERIALS IN ELECTROLYTE SOLUTIONS

I. Tests of Twenty-Four Hours Duration.
A. Stability in Acetonitrile Solutions.

<u>Solute</u>	<u>Cathode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. MgCl ₂	CuF ₂	Solution color changed from colorless to yellow.
	MnO ₂	No change.
	CoF ₃	Solution color changed from colorless to dark blue.
	Cu	No change.
	Co	No change.
2. KPF ₆	CuF ₂	Solution color changed from colorless to light blue.
	MnO ₂	No change.
	CoF ₃	No change.
	Cu	No change.
3. $\begin{matrix} \text{O} < \text{CH}_2\text{CH}_2 > \text{NH}_2\text{PF}_6 \\ & \text{CH}_2\text{CH}_2 & \end{matrix}$ (Morpholinium hexafluorophosphate)	CuF ₂	Solution color changed from colorless to blue-green; blue-green solid produced.
	MnO ₂	Solution color changed from colorless to gray-green. Small amount of light gray solid produced.
	CoF ₃	No change.
	Cu	No change.
	Co	No change.

TABLE VIII. CHEMICAL STABILITY OF CATHODE
MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

I. Tests of Twenty-Four Hours Duration.
 A. Stability in Acetonitrile Solutions. (Continued)

<u>Solute</u>	<u>Cathode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
4. $(n-C_3H_7)_4NPF_6$ (Tetra-n-propylammonium hexafluorophosphate)	CuF_2	Small amount of yellow solid formed.
	MnO_2	Yellow color of solution appeared to become more intense.
	CoF_3	No change.
	Cu	No change.
	Co	No change.
5. $(n-C_4H_9)_2NH_2AsF_6$ (Di-n-butylammonium hexafluoroarsenate)	CuF_2	Solution color became light blue; flocculent blue solid formed.
	MnO_2	No change.
	CoF_3	No change.
	Cu	No change.

I. Tests of Twenty-Four Hours Duration.

B. Stability in Dimethylformamide Solutions.

TABLE VIII. CHEMICAL STABILITY OF CATHODE
MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

I. Tests of Twenty-Four Hours Duration.
 B. Stability in Dimethylformamide Solutions.

<u>Solute</u>	<u>Cathode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. MgCl ₂	CuF ₂	Solution color became deep amber. Yellow-green solid produced.
	MnO ₂	No change.
	CoF ₃	Solution color became deep blue; blue-green solid produced.
	Cu	No change.
	Co	No change.
2. KPF ₆	CuF ₂	Solution color changed from colorless to light blue. Blue solid formed.
	MnO ₂	No change.
	CoF ₃	Solution color changed from colorless to faint red; CoF ₃ solid somewhat darker.
	Cu	No change.
3. (n-C ₃ H ₇) ₄ NPF ₆ (Tetra-n-propylammonium hexafluorophosphate)	CuF ₂	Solution became yellow-green; small amount of green solid produced.
	MnO ₂	Solution color changed from yellow to amber.
	CoF ₃	Solution color became deep amber.
	Cu	No change.
	Co	No change.
4. $\text{O} \begin{matrix} \diagup \text{CH}_2\text{CH}_2 \diagdown \\ \diagdown \text{CH}_2\text{CH}_2 \diagup \end{matrix} \text{NH}_2\text{PF}_6$ (Morpholinium hexafluorophosphate)	CuF ₂	Solution color became light blue. Blue solid produced.
	MnO ₂	No change.
	CoF ₃	Color of the CoF ₃ solid appeared to darken.
	Cu	No change.

TABLE VIII. CHEMICAL STABILITY OF CATHODE
MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

I. Tests of Twenty-Four Hours Duration.

B. Stability in Dimethylformamide Solutions. (Continued)

<u>Solute</u>	<u>Cathode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
5. $(n-C_4H_9)_2NH_2AsF_6$ (Di-n-butylammonium hexafluoroarsenate)	CuF ₂	Solution color became light green; green solid formed.
	MnO ₂	No change.
	CoF ₃	Solution color became light brown; CoF ₃ solid somewhat darkened.
	Cu	No change.

I. Tests of Twenty-Four Hours Duration.

C. Stability in Ethylene Carbonate (80 Weight %)-Propylene
Carbonate (20 Weight %) Solutions.

TABLE VIII. CHEMICAL STABILITY OF CATHODE
MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

I. Tests of Twenty-Four Hours Duration.
 C. Stability in Ethylene Carbonate (80 Weight %)-Propylene
 Carbonate (20 Weight %) Solutions.

<u>Solute</u>	<u>Cathode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. MgCl ₂	CuF ₂	Solution became orange-yellow colored.
	MnO ₂	No change.
	CoF ₃	Solution became blue colored. Blue solid formed.
	Cu	No change.
2. KPF ₆	CuF ₂	No change.
	MnO ₂	No change.
	CoF ₃	Color of CoF ₃ solid somewhat darkened.
	Cu	No change.
3. (n-C ₃ H ₇) ₄ NPF ₆ (Tetra-n-propylammonium hexafluorophosphate)	CuF ₂	Solution color changed from amber to light yellow. Yellow solid formed.
	MnO ₂	No change.
	CoF ₃	No change.
	Cu	No change.
4. $\begin{matrix} \text{O} & \text{CH}_2\text{CH}_2 & \\ & \diagdown & \diagup \\ & \text{CH}_2\text{CH}_2 & \end{matrix} \text{NH}_2\text{PF}_6$ (Morpholinium hexafluorophosphate)	CuF ₂	No change.
	MnO ₂	No change.
	CoF ₃	No change.
	Cu	No change.

I. Tests of Twenty-Four Hours Duration.

D. Stability in N-Nitrosodimethylamine Solutions.

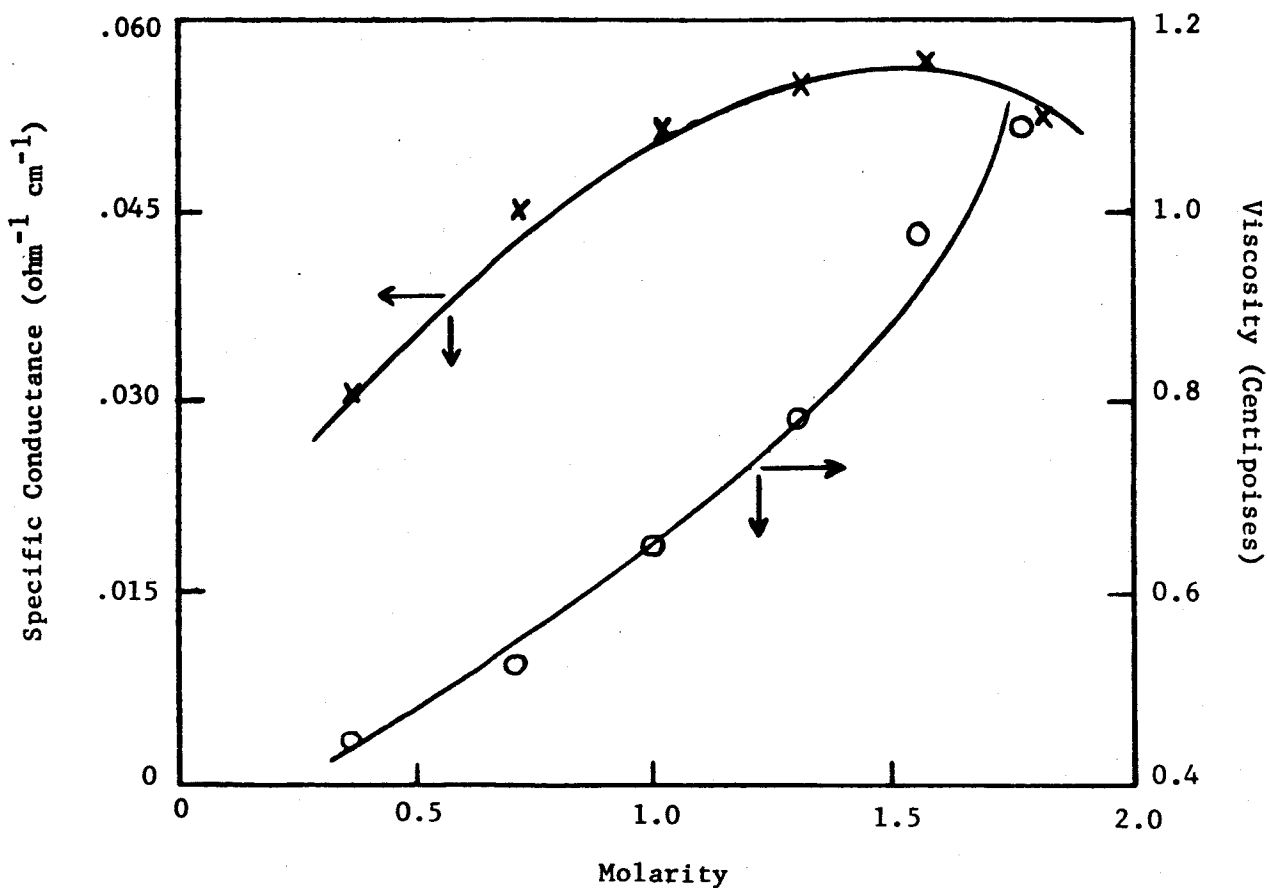
TABLE VIII. CHEMICAL STABILITY OF CATHODE MATERIALS IN ELECTROLYTE SOLUTIONS (Continued)

I. Tests of Twenty-Four Hours Duration.
 D. Stability in N-Nitrosodimethylamine Solutions.

<u>Solute</u>	<u>Cathode Material</u>	<u>Visible Change After 24 Hour Exposure</u>
1. MgCl ₂	CuF ₂	Solution color became amber; green and orange solids formed.
	MnO ₂	No change.
	CoF ₃	Formation of green solid and green solution.
	Cu	No change.
2. KPF ₆	CuF ₂	Color of solution changed from yellow to dark green; gray-green solid formed.
	MnO ₂	No change.
	CoF ₃	Solution color became dark blue-green; black solid formed.
	Cu	No change.
3. (n-C ₃ H ₇) ₄ NPF ₆ (Tetra-n-propylammonium hexafluorophosphate)	CuF ₂	No change.
	MnO ₂	No change.
	CoF ₃	Color of the CoF ₃ solid appeared to be somewhat darkened.
	Cu	No change.
4. $\begin{matrix} \text{O} & \text{CH}_2\text{CH}_2 & \\ & \diagdown & \diagup \\ & \text{CH}_2\text{CH}_2 & \end{matrix} \text{NH}_2\text{PF}_6$ (Morpholinium hexafluorophosphate)	CuF ₂	Solution color became green; green solid formed.
	MnO ₂	No change.
	CoF ₃	Color of the CoF ₃ solid appeared to darken.
	Cu	No change.

TABLE IX. SPECIFIC CONDUCTANCE AND VISCOSITY VS. CONCENTRATION OF MORPHOLINIUM HEXAFLUOROPHOSPHATE-ACETONITRILE SOLUTIONS

Molarity (25°C)	Specific Conductance (ohm ⁻¹ cm ⁻¹) (25°C)	Density (25°C) (g/cc)	Viscosity (25°C) (Centipoise)
0.373	3.05 x 10 ⁻²	0.831	0.447
0.722	4.50 x 10 ⁻²	0.877	0.525
1.021	5.12 x 10 ⁻²	0.920	0.649
1.310	5.47 x 10 ⁻²	0.960	0.782
1.565	5.69 x 10 ⁻²	0.992	0.926
1.811	5.24 x 10 ⁻²	1.024	1.09



V. APPENDIX

V. APPENDIX - DESCRIPTION OF CHEMICAL MATERIALS
AND MATERIAL PREPARATION

The solvents used for electrolyte conductivity determinations, electrochemical tests, and stability determinations were of the highest purity commercially available. The suppliers and specifications of purity for these materials are listed below. In some cases, electrolyte solvents were exposed to lithium metal prior to the experimental work for the purpose of removing any water that might have been present, but, beyond this, no extensive effort has been made to purify or characterize the solvents.

The solutes which were used in the preparation of electrolyte solutions were generally of the best quality available. The specifications of purity provided by the respective suppliers for these materials are also listed subsequently in this section. In some cases, only minimal information is available regarding the purity of the solutes used. This is because of the fact that many of these substances are rather esoteric compounds which have been produced only in limited quantities for research purposes.

Further attempts to characterize these materials have been carried out in a few cases, but no extensive effort has been made toward characterization or purification of solutes. In this respect, continual efforts are being made to obtain materials of higher purity along with more complete specifications of quality from the suppliers.

The suppliers of the calcium, lithium and magnesium metals employed in stability determinations and electrochemical studies are listed below, along with the specifications of purity. Prior to use of these metals in any experiment, the surfaces were scraped or abraded to remove any surface film which did not have the characteristic shiny appearance of a metal. (Calcium and lithium are received and stored in protective oils, but some dullness is produced over extended periods of storage.)

Technical Information on Materials

I. Anodes.

A. Calcium.

1. Supplier - A. D. Mackay.
2. Specifications:
 - a. Sheet, 1/16" thick x minimum width of 3".
 - b. Analysis: Ca, 99.0%; major impurity is Mg.

B. Lithium.

1. Supplier - Foote Mineral Company.
2. Specifications:
 - a. Ribbon, 0.015" thick x 2" wide.
 - b. Analysis: Li, 99.86%; Na, 0.02%; K, 0.07%; N, 0.012%; Cl, 0.04%; Ca, 0.0001%; Fe, 0.0005%; Al, 0.0005%; Si, 0.001%.

C. Magnesium.

1. Supplier - Dow Chemical Co. (Dow Metal Prod.)
2. Specifications:
 - a. Sheet, 0.030" thick.
 - b. Analysis: Mg, 99.80%; Cu, 0.02%; Fe, 0.005%; Pb, 0.01%; Mn, 0.15%; Ni, 0.001%; In, 0.07%.

II. Cathodes and Cathode Products.

A. Lead Dioxide (β -PbO₂).

1. Supplier - Matheson, Coleman and Bell.
2. Specifications:
 - a. Powder.
 - b. Analysis: PbO₂, 95.0%; acid insol., 0.20%; Cl, 0.002%; Mn, 0.0002%; NO₃, 0.02%; SO₄, 0.05%.

B. Cupric Fluoride (CuF₂).

1. Supplier - Ozark-Mahoning.
2. Specifications:
 - a. Powder.
 - b. Analysis: approx. 95% CuF₂; Globe-Union X-ray analysis shows major impurities are Ni, Fe, Cr, Ca and CuF₂·2H₂O.

- C. Cobalt Trifluoride (CoF_3).
1. Supplier - Harshaw.
 2. Specifications:
 - a. Powder.
 - b. Analysis: CoF_3 , 97%; no major impurities found by X-ray.
- D. Manganese Dioxide (MnO_2).
1. Supplier - Manganese Chemical Co.
 2. Specifications:
 - a. Battery grade, type M, powder.
 - b. Analysis: Mn, 59.5%; MnO_2 (based on available oxygen), 88.5%; Fe, 0.25%; H_2O , 3.0%; alkali metals, 0.7%; alkaline earth, 0.4%; heavy metals, 0.01%; insol. in HCl, 0.3%.
- E. Copper.
1. Supplier - Fisher Scientific.
 2. Specifications:
 - a. Electrolytic dust - purified.
 - b. Analysis: no analysis available.
- G. Cobalt.
1. Supplier - Sherritt-Gordon.
 2. Specifications:
 - a. Powder.
 - b. Analysis: Co, 99⁺%.

III. Solvents.

- A. Acetonitrile.
1. Supplier - Matheson, Coleman and Bell.
 2. Specifications:
 - a. Spectroquality.
- B. Dimethylformamide.
1. Supplier - Fisher Scientific.
 2. Specifications:
 - a. Fisher Certified - purity 99⁺% and no H_2O present.
- C. N-Nitrosodimethylamine.
1. Supplier - Aldrich Chemical Co.
 2. Specifications:
 - a. Research grade - free from isomers, homologs and other impurities - suitable for reagent use and more exacting synthesis - pure as is economically feasible.

D. Dimethyl Cyanamide.

1. Supplier - Matheson, Coleman and Bell.
2. Specifications:
 - a. Practical grade - less than 99% pure, boiling point range 162-164°C.

E. Ethylene Carbonate.

1. Supplier - Matheson, Coleman and Bell.
2. Specifications:
 - a. Practical grade - see dimethyl cyanamide, melting point 34-35°C.

F. Propylene Carbonate.

1. Supplier - Matheson, Coleman and Bell.
2. Specifications:
 - a. Practical grade - see dimethyl cyanamide, boiling point 108-110°C (10 mm.).

G. Tetramethylurea.

1. Supplier - Matheson, Coleman and Bell.
2. Specifications:
 - a. Practical grade - see dimethyl cyanamide, boiling point 175.5-177.5°C.

H. Acetone.

1. Supplier - Fisher Scientific.
2. Specifications:
 - a. Spectroanalyzed - 99.9⁺% pure.

IV. Solutes.

A. Aluminum Chloride (AlCl₃).

1. Supplier - Matheson, Coleman and Bell.
2. Specifications:
 - a. Powder, reagent.
 - b. Analysis: heavy metals, 0.002%; Fe, 0.01%; not pp't. by NH₄OH, 0.20%; sulfate, 0.005%.

B. Lithium Chloride (LiCl).

1. Supplier - Matheson, Coleman and Bell.
2. Specifications:
 - a. Regent crystals.
 - b. Analysis: Ca, 0.04%; heavy metals, 0.002%; insol. matter, 0.01%; Fe, 0.002%; Mg, 0.01%; NO₃, 0.005%; KCl + NaCl, 0.5%; SO₄, 0.02%.

C. Ferric Chloride (FeCl_3).

1. Supplier - Fisher Scientific.
2. Specifications:
 - a. Purified, anhydrous, sublimed.

D. Magnesium Chloride (MgCl_2).

1. Supplier - Dow Chemical Co.
2. Specifications:
 - a. Powder.
 - b. Analysis: MgCl_2 , 95% min.; NaCl, 0.2%; CuCl, 0.2%; MgO, 0.2%; Fe, 0.3%.

E. Potassium Hexafluorophosphate (KPF_6).

1. Supplier - Matheson, Coleman and Bell.
2. Specifications:
 - a. 98-100% purity.

The remainder of the solutes tested are classified in a general manner in the following table. They were purchased from three main suppliers who gave the following specifications:

Alpha Inorganics - compounds not otherwise designated have a purity in the range of 97 to 99%.

Aldrich Chemical - unless marked Puriss or Tech., all chemicals are Research Grade (high purity).
- see N-nitrosodimethylamine specifications.

Ozark-Mahoning - in general, all compounds are 95% pure or better based on fluorine analysis.

CHEMICALS SUPPLIED BY:

ALDRICH CHEMICAL CO.	OZARK-MAHONING CO.	ALPHA INORGANICS
<p>Tetra-n-propylammonium tetrafluoroborate Tetraphenylammonium chloride Tetraphenylphosphonium chloride Tetraphenylarsonium chloride Benzyl-N,N,N-trimethylammonium hexafluoroantimonate</p>	<p>Morpholinium hexafluorophosphate Tetra-n-propylammonium hexafluorophosphate Tri-n-propylammonium hexafluoroarsenate N-phenyl-N,N,N-trimethylammonium hexafluorophosphate p-Dodecyl,N,N,N-trimethylammonium hexafluorophosphate di-n-Butylammonium hexafluoroarsenate Tetramethylammonium hexafluorophosphate Tetra-n-propylammonium hexafluoroarsenate Cesium hexafluorophosphate Sodium hexafluorophosphate Tetra-n-propylammonium hexafluoroantimonate</p>	<p>Sodium fluoborate Potassium thiocyanatochromate Potassium hexafluoroarsenate Ammonium fluosulfonate Sodium hexafluoroantimonate</p>

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