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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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FOURTH QUARTERLY REPORT
APRIL – JUNE, 1965

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SUMMARY

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Eighty-five new anode-electrolyte combinations were studied in the continuation of a screening program designed to single out those systems which are most promising for further development. Among these, eight anode systems could sustain discharges at 100 ma/cm^2 without excessive polarization. An additional forty-two systems could sustain at least 10 ma/cm^2. Linearly varying potential and coulombic efficiency studies were initiated to provide a more extensive electrochemical evaluation of some of the anode systems which had exhibited promising characteristics in preliminary studies.

Cupric fluoride cathodes prepared by dry-press techniques exhibited unsatisfactory electrochemical characteristics. Cathodes of lead dioxide and manganese dioxide yielded performances which were reproducible and superior to those of cupric fluoride, but did not perform well enough to be used in a high energy density battery. Preliminary studies of soluble cathode depolarizer cells indicated that utilization of cathodes of this kind might be feasible, provided that it is possible to separate a soluble cathode depolarizer from an anode by some means without affecting cell performance.

Chemical stability tests involving prospective electrode materials in various electrolytes were carried out, and solvent purification by vacuum distillation was undertaken to determine if trace impurities in the solvents can affect electrochemical characteristics significantly.

Author

INTRODUCTION

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

Preliminary electrochemical evaluations of calcium, lithium and magnesium anodes in various electrolytes have yielded several promising combinations thus far and studies have been initiated to evaluate some of these systems more thoroughly.

Efforts to improve the electrochemical characteristics of dry-pressed cupric fluoride cathodes have been unsuccessful. Concurrently, consideration has been given to possible approaches for eliminating some of the difficulties associated with conventional techniques of cathode fabrication.

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ABSTRACT

Electrochemical half-cell screening of 85 anode-electrolyte combinations was carried out and coulombic efficiency and linearly varying potential studies of anode systems were initiated. Cathodes of CuF_2, CoF_3, PbO_2 and MnO_2 were tested for electrochemical capability and a study was made of the electrochemical characteristics of cells which utilized soluble cathode depolarizers.

Compatibility tests involving electrode materials were completed in several electrolytes. In addition, a study to determine the effect of distillation on solvent and electrolyte properties was undertaken.

I. OVERALL PROGRESS

I. OVERALL PROGRESS

Progress during the fourth quarter encompassed work in the following areas:

1. Electrochemical half-cell screening of anodes.
2. Linearly varying potential studies of anodes.
3. Coulombic efficiency studies of anodes.
4. Development of cathodes. Fabrication and electrochemical studies.
5. Determination of the chemical stability of electrode materials in electrolyte solutions.
6. Determination of the specific conductance of electrolyte solutions.
7. Purification of solvents.

Experimental Approaches and Results.

1. Electrochemical Half-Cell Screening of Anodes.

a. Approach. The procedure adopted for rapid, preliminary evaluation of prospective anode-electrolyte systems on the basis of polarization measurements was described in detail in the Third Quarterly Report. This work was continued during the fourth quarter to include eighty-five systems which had not been studied previously. In addition, the method was applied to evaluating the effect of modifying the electrolytes for certain of the systems which had been studied previously. Of specific interest were electrolytes typified by morpholinium hexafluorophosphate-N-nitrosodimethylamine with a lithium anode. This system can sustain discharge at 100 ma/cm^2 but the anode is attacked by the electrolyte. It was observed, however, that the reaction ceased after several hours and that the specific conductance of the resultant solution was still in excess of 1 x 10^{-2} ohm^{-1} cm^{-1}, although the value was approximately 30% lower than the value prior to exposure to lithium. Consequently, it was decided to determine how a lithium anode would perform if the electrolyte were pretreated with lithium until gas evolution ceased. Several other similar systems were selected for the same kind of study.

b. Results. The screening data for the eighty-five new anode systems are given in Table I, pages IV-1 through IV-34. Among these, eight systems could sustain discharge at 100 ma/cm^2. Forty-two others were qualified as being capable of discharge at a current density of at least 10 ma/cm^2, but less than 100 ma/cm^2. The remainder of the systems exhibited excessive polarization at current densities of 10 ma/cm^2 and were classified as 1, 0.1, or less than 0.1 ma/cm^2 capability systems.

The polarization data for lithium anodes screened in three electrolytes which had been pretreated with lithium metal are presented in Table II, pages IV-35 through IV-38, where details of the pretreatment are given along with the results for the same systems when the electrolytes were not pretreated. In each case, high current densities (10-100 ma/cm^2) were sustained and excessive decomposition of the anode did not occur as it had in the untreated electrolytes. Thus far, a positive identification has not been made of the products which are formed upon pretreating the electrolytes with lithium, but work of this kind will be carried out in the future.

2. Linearly Varying Potential Studies of Anodes.

a. Approach. In previous half-cell screening studies, constant current techniques have been utilized almost exclusively. Efforts have now been initiated to provide a more detailed characterization of the electrochemical processes governing the performance of promising anode-electrolyte systems. For this purpose, controlled potential methods have been adopted. In particular, the linearly varying potential (LVP) technique has been selected to determine whether or not the electrochemical reaction at a given anode involves any reactions other than oxidation of the metal. Other reactions may arise either from oxidation of the metal through more than one simple reaction, depending upon the nature of the particular electrolyte and the electrode, or from oxidation of a component of the system, other than the metal, which is capable of being oxidized in the range of potentials investigated. Reactions of the latter type could involve impurities in the electrode or electrolyte, or the electrolyte itself.

In the linearly varying potential method, the potential of a small test electrode of precisely defined area is increased and then decreased as a linear function of time. By starting the scan at a potential corresponding to the open circuit voltage of the electrode being studied, and continuing in a given direction, the current will rise to a peak and thereafter either level off or decrease. The rise in current observed is a function of the kinetics, i.e., the activation polarization of the reaction. The decay is attributable to the fact that the reaction rate has increased with the changing potential to the point that the transport of reactants to the electrode surface (or of products away from the electrode surface) has become slower than the electrode process and, thus, has limited the passage of current. However, if a second reaction can occur in the range of potential where the first reaction is transport limited, the current may begin to increase again as the potential is continuously changed and result in a second peak or inflection in the current versus potential curve.

The electrochemical kinetics apparatus employed in this work has been described in previous reports. The high scanning rates which can be achieved with this equipment permit detection of secondary reactions which might not be ascertainable from current-voltage curves obtained at lower sweep rates.

The anode-electrolyte systems to be studied by the linearly varying potential technique were chosen on the basis of previous half-cell screening evaluations. Systems capable of sustaining high current densities (100 ma/cm^2) are being examined first.

b. Results. A linearly varying potential study was made on the anode system of Mg in an electrolyte of 1 molal $AlCl_3$ in acetonitrile. The counter and reference electrodes were Ag/AgCl in all cases. The results obtained using sweep rates of 30, 100 and 330 mv/sec. are shown in Figure 1, page IV-39. At first, 26.45 mm^2 of Mg surface were in contact with the solution. However, the currents obtained were too large to allow any definitive examination of the system. Therefore, the area of the Mg surface in contact with the electrolyte was decreased to 7.55 mm^2 before the scans depicted by curve II were obtained. Curve II is typical of the results obtained at sweep rates of 30, 100 and 330 mv/sec. Curve III represents a final sweep at 30 mv/sec. At the conclusion of the experiment, the Mg surface area had decreased to approximately 2.9 mm^2. These data indicate that only one reaction is taking place at the anode, presumably the oxidation of Mg.

3. Coulombic Efficiency Studies of Anodes.

a. Approach. Coulombic efficiency determinations were initiated for anode-electrolyte systems which sustained current densities of 100 ma/cm^2 in the half-cell screening evaluations. The procedure employed for these studies consisted of discharging the anode (1 cm^2 initial area) at a constant current of 50 milliamperes in combination with a silver-silver chloride cathode until all of the anode had been consumed. These discharges were not forced, but were allowed to proceed spontaneously through a variable resistor implemented by a transistor circuit which automatically adjusted the resistance to maintain constant current.

b. Results. Initial efforts to determine the coulombic efficiencies of promising anode systems were unsuccessful because the cathodes became polarized so badly that spontaneous discharge at the chosen rate of 50 ma was not possible. Subsequently, a large silver chloride electrode was employed which functioned effectively.

Efficiency determinations were limited to one system in this period, magnesium in aluminum chloride-acetonitrile electrolyte. Magnesium electrodes weighing 0.11 grams were discharged to the limit of their capacity at 50 milliamperes. This discharge rate corresponded to a current density of 50 ma/cm^2 initially and higher current densities as the magnesium was consumed. The discharge curves for two trials with this system are presented in Figure 2, page IV-40. In both cases, a coulombic efficiency of 74% was achieved, based on a two electron change for magnesium. In one case, a small fragment of magnesium was observed to break off the lead at the end of the discharge, indicating that a higher efficiency could have been achieved if this had not happened.

4. Development of Cathodes. Fabrication and Electrochemical Studies.

a. Approaches. On the basis of its high theoretical energy density capabilities, cupric fluoride was the first material selected for cathode development. Since this substance and most of the other active cathode materials which were initially considered are not conductors (and must, therefore, be effectively combined with conductive additives), the mode of fabrication is important in determining electrochemical performance.

Initially, cathodes consisting of cupric fluoride and conductive additives (carbon black or metallic copper powder) were fabricated by dry-press techniques and the effects of varying composition and formation pressure were studied. These electrodes were observed to polarize appreciably even at low current densities (1 ma/cm^2) and, in general, the electrochemical characteristics were not reproducible. The limitations of these cathodes were believed to result primarily from insufficient intimate contact of the active material with the conductive substrate. Subsequent cathode development was approached in two different ways; part of the effort was concerned with developing means of achieving more intimate contact of active materials with the conducting substrates and with controlling other factors which determine electrode performance, such as porosity. Concurrently, attention was given to modes of attack which may make it possible to circumvent the difficulties and limitations associated with incorporating an inert, conductive element with the active material.

Regarding the more conventional approach to electrode fabrication, improved performance was sought during this period by continued study of the fabrication technique involving the partial reduction of cupric fluoride with hydrogen gas (cf. Third Quarterly Report). The degree of reduction to copper metal varies with the time of exposure. The metal formed in this manner should provide the necessary conductive substrate.

Another phase of this work was concerned with developing means of increasing and controlling the porosity of the electrodes to determine if this would have a beneficial effect on electrode performance. The method adopted for this purpose consisted of subliming a solid substance from a dry-pressed cathode pellet under reduced pressure. Several possible sublimable substances were considered on the basis of known vapor pressure-temperature characteristics. After a preliminary experimental study, phenol was chosen for use.

Fabrication of cupric fluoride cathodes by a filter pad technique reported by another research group was started. However, no electrochemical data have been generated thus far because of a delay in the acquisition of materials. With respect to developing concepts which may eliminate some of the problems and limitations associated with conventional approaches to cathode fabrication, two distinct possibilities were explored.

The first of these involves the use of high energy density cathode materials which are non-stoichiometric compounds. Materials of this kind are often conductive in themselves and may eliminate the need to employ inert, conductive additives and the accompanying fabrication problems. The initial experimental work in this area involved the preparation and preliminary electrochemical evaluation of cathodes of lead dioxide and manganese dioxide. These materials have high theoretical energy density characteristics and are known non-stoichiometric compounds. In addition, a theoretical study and literature survey is being conducted to select other possible high energy density materials which are non-stoichiometric compounds.

The second approach explored was concerned with determining the feasibility of utilizing soluble cathode depolarizers. Essentially, a cathode of this kind consists of a concentrated solution of the active cathode material in contact with an inert conductor. Utilization of an electrode of this kind offers the following advantages over solid cathodes:

1) When the active material is in the dissolved state, the problems of high resistance and low efficiency resulting from insufficient contact with the conducting element may be avoided.

2) In the fabrication of solid electrodes, it is desirable to make the structure sufficiently porous both to permit diffusion of the electrolyte into the inner electroactive sites and to create an adequate number of such sites. When the active material is in the dissolved state, this problem is also avoided.

An experimental study was undertaken to determine whether or not cells employing soluble cathode depolarizers are (1) operable, and (2) capable of delivering high currents. It was believed that if cells could be made which satisfied these initial requirements, further consideration might be given to this approach, provided that some means of separating a soluble cathode depolarizer from an anode without affecting cell performance is possible.

The selection of the first active cathode materials for determining the feasibility of the soluble cathode approach was based upon the following considerations.

Free energy calculations for the reaction --

$$2Li + H_2O \longrightarrow H_2 \uparrow + Li_2O$$

yield a theoretical energy density in excess of 1,000 watt-hours per pound. In this case, the voltages provided by lithium (or calcium) are such that, theoretically, if an inert electrode is operating at a potential where it can function as a hydrogen evolution cathode, the Li-H_2O couple (or Ca-H_2O) could still deliver over 200 watt-hours per pound. The Li-H_2O system was not chosen for the initial experiments, however, because of the high reactivity of these materials toward one another. The study of this system would require effective separation of the reactants, which was not the immediate objective of these tests.

Instead, it was proposed to use an electrolyte capable of furnishing hydrogen ions in which calcium or lithium is somewhat stable. In this situation, a soluble cathode depolarizer capable of furnishing hydrogen ions could function as a hydrogen evolution cathode at an inert electrode with a low hydrogen overpotential. Since the purpose of the experiments was to demonstrate operability, the depolarizer was not required to be of low equivalent weight.

The first material selected for this purpose was morpholinium hexafluorophosphate. In previous anode half-cell screening tests in which solutions of this substance were employed as the electrolyte, it had been observed that gas evolution occurred at the cathode as the cathode polarized. The assumption that this gas was hydrogen was based on the fact that the cathode voltage was about -1.0 versus a silver-silver chloride reference, which could permit the hydrogen evolution reaction to proceed. Morpholinium hexafluorophosphate satisfied the additional requirement of high solubility in nonaqueous solvents, forming highly conductive solutions. Although solutions of the substance are somewhat reactive toward calcium and lithium, the rate of reaction is not prohibitive for these experiments.

The first cells constructed which employed this material as a soluble cathode depolarizer were essentially of the reserve activated type, i.e., the anode and an inert conducting electrode (connected through the external circuit) were positioned in the same container. The cell was then activated by addition of a solution of the cathode depolarizer-electrolyte and discharge was carried out until virtually all of the anode had been consumed. The anode material employed in the first cells of this type was calcium; acetonitrile was used as the solvent for the morpholinium hexafluorophosphate. Silver foil and carbon were employed initially as the inert electrodes. Subsequently, materials were utilized which favor the hydrogen evolution reaction, i.e., conductors were chosen for which the hydrogen overpotential is small. These included platinized platinum, platinized carbon and sintered nickel.

In some of the experiments, additional sources of hydrogen ions were added to the morpholinium hexafluorophosphate solution to determine what effect they would have on the cell performance, i.e., small quantities of water or aqueous sulfuric acid were added to the cathode solution.

Upon completion of the study of several small, reserve activated cells of the type described above, the construction of larger, more elaborate cells was undertaken. In these cells, the anode and cathode solutions were situated in two different compartments, separated from one another by a thin, permeable carbon wall. In the anode compartment, an electrolyte was used that did not cause self-discharge of the anode. The use of porous carbon to separate the anolyte and catholyte limited the rate of interdiffusion of the two solutions, and thereby reduced the rate of attack of the anode by the soluble cathode material. The carbon divider was also utilized as the inert electrode for the cathode.

Lithium and calcium anodes were used in these cells and the substances employed as cathode depolarizers included nonaqueous solutions of morpholinium hexafluorophosphate and aqueous solutions of sulfuric acid. In addition, two cells were tested that employed solutions of cuprous chloride and cupric chloride as the cathode depolarizers.

b. Results.

1) Preparation of Porous Cathodes by Sublimation. Upon completion of a preliminary experimental study, phenol was selected as a possible substance to be sublimed from dry-pressed cathodes for the purpose of increasing porosity. Subsequent experiments indicated that approximately 92% of the phenol initially present* in cupric fluoride pellets could be removed by sublimation in a vessel in which the pressure was reduced to 0.5-1 mm of mercury. This degree of removal was effected by maintaining the temperature of the water bath surrounding the vessel at approximately 26°C for seventeen hours. The decrease in weight of the pellets after this interval was assumed to represent the weight of phenol which had been sublimed. Microscopic examination of the pellets revealed the presence of depressions or void spots which had not been observed prior to sublimation. Extended treatment of the pellets at reduced pressure beyond the initial seventeen hour period did not result in further significant weight losses even at temperatures of 40°C. However, when the method was applied to cobalt trifluoride pellets, virtually 100% removal of phenol was effected.

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* Initial composition of cupric fluoride pellets: 80% CuF_2, 16% phenol and 4% Solka-Floc binder.

The results of this work indicated that the method could be useful for controlling the porosity of electrodes. Although no visible evidence of reaction was observed upon combining phenol with cupric fluoride or cobalt trifluoride, it was realized that the possibility of interactions of these materials should be considered in the further evaluation of the method.

Porous cupric fluoride cathodes prepared by subliming phenol were subsequently treated with hydrogen gas to effect partial reduction of the active material for the purpose of providing a conductive substrate. The evaluation of these electrodes is presented below, where the further application of the partial reduction technique is discussed.

2) <u>Cupric Fluoride and Cobalt Trifluoride Cathodes Treated with Hydrogen Gas</u>. Preliminary investigation of this technique during the third quarter indicated that partial reduction of cupric fluoride pellets with a current of hydrogen gas could best be controlled if the gas were maintained at a temperature of ca. 130°C. Partial reduction to metallic copper was confirmed qualitatively by X-ray diffraction analysis. Attempts to fabricate cupric fluoride cathodes with improved electrochemical characteristics by this technique, however, were unsuccessful. Polarization was extensive even at current densities of 1 ma/cm^2 for each of several electrodes prepared by this method. The electrochemical characteristics did not differ significantly for any of the electrodes, with the exception that the initial open circuit voltages differed in some cases.

The polarization data for two cupric fluoride cathodes of this kind are presented in Table III, section A, page IV-42. These results are typical of all those obtained for cupric fluoride cathodes in this particular series of experiments.

The partial reduction technique was also applied in the fabrication of cobalt trifluoride cathodes. Initial attempts to partially reduce this material with hydrogen were unsuccessful, as confirmed by X-ray diffraction analysis. Prior to obtaining this information, however, three cobalt trifluoride cathodes were tested for electrochemical capability. The data for these electrodes are presented in Table III, section B, page IV-43. The electrochemical characteristics were poor, being comparable to those of the cupric fluoride electrodes.

3) Electrochemical Screening of Lead Dioxide and Manganese Dioxide Cathodes. The results for electrodes of PbO_2 and MnO_2 are listed in Table III, sections C and D, respectively, pages IV-44 and IV-45. The electrochemical characteristics of these electrodes were quite reproducible in two trials and were superior to those of cupric fluoride and cobalt trifluoride. Performance, however, was not satisfactory for a high energy density battery, since the degree of polarization became quite high at a current density of 5 ma/cm^2. Coloration of the electrolytes and solid product formation were also observed in these tests.

The electrolytes which have been used in the preliminary electrochemical evaluation of CuF_2, CoF_3, PbO_2 and MnO_2 cathodes were chosen as being representative of some of the better electrolytes which were developed in the earlier phases of the program (conductance studies and anode screening).

Although the nature of the electrolyte is highly important in determining electrode performance, this effect cannot be adequately evaluated until methods of cathode fabrication are developed which permit effective utilization of a given active cathode material. Reproducibility of electrode performance in a given electrolyte must also be achieved before comparisons can be made with other electrolytes. Because these requirements have not been satisfied by most of the cathodes tested to date, electrochemical studies have been confined to electrolytes utilizing two representative solutes: $MgCl_2$ and N-phenyl N,N,N-trimethylammonium hexafluorophosphate.

4) Electrochemical Studies of the Feasibility of Utilizing Dissolved Active Cathode Materials. Nineteen cells which employed soluble cathode depolarizers were constructed and discharged in this period. The details of construction and the discharge data are presented in Table IV, pages IV-46 through IV-75, wherein each cell is designated by the anode and active cathode material.

The first cells were of the reserve activated type described in the section on experimental approaches. These were small cells (anodes and cathodes of ca. 1 cm^2 area) and were comprised of calcium anodes and morpholinium hexafluorophosphate cathode-electrolytes. The cells which performed best among these exhibited voltages of ca. 1.4 volts while delivering currents of approximately 15 milliamperes. At somewhat higher currents (20-25 ma), voltages as high as 1.25 volts were observed. At still higher currents (40-60 ma), the corresponding voltages were about 0.5 volts in the better cells.

Cells utilizing inert cathode current collectors of platinized platinum, platinized carbon or sintered nickel performed better than those which used pure carbon, as was expected. In general, the performance of cells which used platinized inert electrodes was better than those employing sintered nickel.

Other small cells were also assembled with calcium anodes and morpholinium hexafluorophosphate cathode solutions, but differed from the previous cells in that water or aqueous sulfuric acid was added to the cathode depolarizer solution. Addition of these materials enhanced the performance of the cells, although they caused the calcium to self-discharge at a greater rate.

One small cell (1 cm^2, anode) of the reserve activated type with lithium as the anode and morpholinium hexafluorophosphate solution as the cathode exhibited voltages of 1.70 to 1.00 volts while delivering a current varying between 17 and 10 milliamperes.

The remaining cells were all of the second general type described in the section on experimental approaches, i.e., the anode and cathode compartments were separated from one another by a platinized porous carbon divider. Several variations of this basic design were employed, the details of which are presented with the discharge data. In the cells in which lithium was employed as the anode, the area of the anode was much larger than had been utilized in the initial cells.

Among these, the best performances were attained when lithium was utilized as the anode and aqueous sulfuric acid, cuprous chloride or cupric chloride solutions as the cathode. In some cases, cell voltages in excess of two volts were maintained over discharge periods of several hours at a discharge rate of ca. 70 milliamperes. In other cases, discharges were carried out at much higher rates (200-500 milliamperes) for periods of up to one hour. Voltages varying between 2.0 and 1.0 were observed under these conditions in the better cells. In still other cases, voltages varying between 2.95 and 2.50 were observed at lower discharge rates (25-50 milliamperes).

The effects of variation in cell design on performance are too numerous to mention here and, indeed, since determining the feasibility of this type of operation was the major purpose of these experiments, an analysis of these effects is not appropriate at this time.

In view of the fact that cells of this kind were operable and capable of delivering substantial currents, it was concluded that further consideration might be given to this approach, provided that some means of separating a soluble cathode depolarizer from an anode without affecting cell performance is possible.

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

a. Approach. The procedure adopted for stability determinations at room temperature was described in the Third Quarterly Report. Normally, if an electrode-electrolyte system exhibits reactivity during an initial twenty-four hour exposure period, no further study of stability is made. In the past quarter, however, some of the anode systems tested for two week periods had exhibited instability in previous twenty-four hour tests. Longer observations were desired because the systems exhibited high electrochemical capability in half-cell screening tests and because it was observed that reaction ceased after an initial exposure period.

Twenty-four hour tests were carried out with CuF_2, CoF_3, MnO_2 cathode materials, and copper metal in several electrolytes which had not been tested previously.

b. Results. The data for two week stability tests involving calcium, lithium or magnesium anodes in sixteen electrolytes are listed in Table V, pages IV-65 through IV-85. In the case of the systems which had exhibited some reactivity in previous twenty-four hour tests, decomposition of the anode materials was appreciable. However, since other tests have indicated that reaction ceases in systems of this kind after an initial exposure period (cf. half-cell screening experiments, Table II), it is evident that the stability test procedure should be modified. In the future, systems of this kind will be pretreated with the given anode material until reaction ceases, and after this time, the resultant solution (filtered) will be tested for stability with a fresh strip of the anode material.

The data for cathode stability tests of twenty-four hours duration involving CuF_2, CoF_3, MnO_2 and copper metal in several electrolytes are given in Table VI, pages IV-86 through IV-93.

6. Determination of the Specific Conductance of Electrolyte Solutions. Since the inception of the electrochemical screening program for anode-electrolyte systems, several electrolytes have been utilized for which the conductance values had not been determined previously. These electrolytes were chosen because of their similarity to some of the other electrolytes which have been employed. The conductance values were determined as a matter of routine at the time of the electrochemical screening tests and have been combined in Table VII, pages IV-94 and IV-95, for presentation at this time. Values varied from 4.20×10^{-2} to 4.52×10^{-3} ohm^{-1} cm^{-1}.

7. Purification of Solvents.

a. Approach. The solvents utilized for electrolytes in the various phases of the experimental program have been of the highest purity commercially available (cf. Third Quarterly Report for names of suppliers and suppliers' specifications of purity). Controls for checking the consistency of the purity of solvents as received from the suppliers have been effected by regularly measuring the conductivity of the solvents and of the electrolytes prepared from them. In the course of making these control measurements in the various phases of the experimental work, i.e., electrochemical half-cell screening, chemical stability determinations and conductance studies, a high degree of reproducibility of electrolyte conductivity has been observed. Thus, for example, repeated determinations of the conductivity of 1 molal morpholinium hexafluorophosphate-dimethylformamide solutions used in various studies during the past several months have yielded the following values:

Specific Conductance (ohm^{-1} cm^{-1})
2.57×10^{-2}
2.68×10^{-2}
2.68×10^{-2}
2.89×10^{-2}
2.66×10^{-2}

A comparable degree of reproducibility has generally been observed for the other electrolytes studied in the program. These results attest to the reliability of the quality of the solvents and solutes utilized. Conductance values determined for pure solvents (no solute) have agreed with literature values within an order of magnitude. Since literature values reported by various investigators differed among themselves by as much as an order of magnitude for the same solvents (high purity), a much closer agreement would not generally be expected.

The conductance of solvents being used is typically in the range 10^{-6} - 10^{-7} ohm^{-1} cm^{-1}, while the corresponding electrolyte conductances are of the order of 10^{-2} ohm^{-1} cm^{-1}. It is apparent that solvent conductivity variations of an order of magnitude will contribute negligibly to electrolyte conductance in their systems, except in possible cases where a solvent impurity could have a synergistic effect on electrolyte conductance.

In accord with Technical Direction No. 1, work has been undertaken to determine whether or not the presence of residual (trace) impurities, such as water, in the solvents can influence the electrochemical and chemical behavior of electrodes to a significant degree. An additional objective of this work is to provide a more extensive characterization of solvent purity.

Initial purification will be accomplished by distillation. A vacuum distillation apparatus has been set up for this purpose (see Figure 3, page IV-96). The distillation apparatus includes a 1000 ml distilling flask and a vacuum-jacketed column which is strip-silvered (supplied by the Scientific Glass Co.). This column has an internal diameter of 2.5 cm. and is packed with a 90 cm. bed of glass helices 1/8" in diameter. Distillations were carried out at a 1:1 reflux ratio. This ratio was maintained by means of a swinging funnel fitted with a soft iron core mounted in this distillation head. An electromagnet regulated by a GE type TSA-14 timing device moves this funnel into position for either total reflux or collection. The timer cycle of 20 seconds is divided into the desired ratio of total reflux to collection, in this case, 1:1. Variations up to a ratio of 1:100 with 1% accuracy are possible by appropriate setting of the timer. Distillate was collected in a flask designed to allow transfer to the argon atmosphere dry box without exposure to the atmosphere or interruption of the distillation process. Vacuum is maintained with a No. 1400B Welch Duoseal pump capable of achieving a pressure of 1 micron. Vapor pressure is measured by a closed-end manometer mounted on the distillation flask.

Vapor pressure in the system during distillation was dependent upon the solvent being distilled and was kept as low as proper reflux conditions would permit. In each case, 700 ml were distilled; the first and last 100 ml fractions were discarded. The solvents were characterized by recording the refractive index and specific conductivity of each of the collected fractions. Specific conductivities were measured in a dry argon atmosphere and all fractions were stored in an argon atmosphere between tests. Refractive indices and specific conductivities will be measured one, two, four, twelve and twenty-four weeks after distillation.

Anode screening tests in electrolytes made from N,N-dimethylformamide distilled at a reflux ratio of 1:1 were carried out in two cases to permit comparison with earlier tests in which the solvent had not been distilled.

b. Results. In order to determine the fractionating capabilities of the distillation apparatus, a known mixture consisting of 250 ml of water and 250 ml of acetone was distilled at 743 mm. Hg using a 1:1 reflux ratio. The distillate was collected in 50 ml fractions. The refractive indices obtained for the first five fractions matched that of pure acetone. After 250 ml had been collected, the next 50 ml fraction was found to have a refractive index of 1.333 at 20^{o}C. Since this is the refractive index of pure water, a clean separation had been obtained.

Prior to setting up the fractionating column, a 475 ml sample of N,N-dimethylformamide (DMF) was distilled at a pressure of 2 mm. Hg using a 1:1 reflux ratio. The temperature of the vapor was approximately 26°C throughout the distillation. The first 100 ml were discarded and the next 125 ml fraction was transferred in vacuo to an argon atmosphere dry box. The specific conductivity of this fraction was 6.92×10^{-7} ohm^{-1} cm^{-1} (27°C) as compared to an average value of 2.64×10^{-6} ohm^{-1} cm^{-1} (27°C) for the undistilled solvent.

A 1 molal solution of N-phenyl-N,N,N-trimethylammonium hexafluorophosphate in the distilled DMF did not differ in conductivity from a similar solution prepared with the undistilled solvent. When the above solutions were used in anode screening tests, only minor differences in behavior were noted (Table VIII, page IV-97 and IV-98).

Using the fractionating column, a second sample of DMF was distilled from calcium hydride. The refractive indices and specific conductivities were determined for the second through sixth 100 ml fractions; the specific conductivities were measured in an argon atmosphere. These samples were stored in an argon atmosphere and the measurements repeated after one week had elapsed (Table IX, page IV-99). It is evident that the refractive indices for the various fractions were fairly consistent while the specific conductivities showed greater variation. This variation in conductivity can be attributed, at least in part, to the fact that the measurements are being made at the lower operating limit of the conductivity equipment.

A sample of acetonitrile was distilled in the same manner as the second DMF sample. The results for refractive indices and conductivities are given in Table IX, page IV-99. Because of the large fluctuations in vapor pressure during the distillation, the test will be repeated with a new sample.

The data thus far indicate that vacuum distillation has only a small effect on the solvents thus purified. Refractive indices differ only slightly, if at all, from the values before distillation. Conductivity decreases initially but it begins to increase again with time. Tentatively, it seems that distillation yields a pure material and changes upon standing should indicate the stability of the purified solvent.

II. CURRENT PROBLEMS

II. CURRENT PROBLEMS

The major problem continues to be that of developing a cathode with suitable characteristics for a high energy density battery.

III. WORK TO BE PERFORMED

III. WORK TO BE PERFORMED IN THE NEXT QUARTER

As a result of the extension and expansion of the present contract, work will be carried out at a higher level of activity and will include the following areas in the next quarter.

1. Anodes.

Linearly varying potential and coulombic efficiency studies will be made of stable anode-electrolyte systems capable of sustaining 100 ma/cm^2 discharges.

2. Cathodes.

Emphasis will be placed on developing cathodes from high energy density compounds which are non-stoichiometric. Cathodes fabricated from such materials will be evaluated electrochemically and the study to select other materials of this kind will be continued.

Possible means of separating soluble cathode depolarizers from anodes without detracting from cell performance will also be investigated. Presently, two possibilities are being considered. The first of these involves the use of selective ion exhange membranes which could effect the desired separation while permitting passage of an electrolyte species to perform the electrolytic conductance function. The second possibility involves the use of of a conductive porous container for a liquid cathode depolarizer, which would be designed in a manner such that the cathode depolarizer would diffuse out of the container at the same rate that it was consumed in an electrochemical reaction occurring at the outer surface.

In addition, electrochemical evaluations will be made of cupric fluoride paste electrodes fabricated by a filter press technique.

3. Electrolytes.

Purification of solvents by vacuum distillation will be continued. Characterization will be effected by refractive index and conductivity measurements.

A detailed study of the properties of electrolytes which contribute to good electrode performance will be initiated. Of specific interest are electrolytes made from solutes such as morpholinium hexafluorophosphate, in which anodes of lithium and calcium have exhibited superior electrochemical characteristics. This work will include the study of electrolyte properties in general, and in this respect, will be a continuation of the work carried out in an earlier phase of the program.

4. Stability of Electrode Materials in Electrolyte Solutions.

Stability tests will be carried out with calcium, lithium, and magnesium in electrolytes which have not been tested previously.

5. Construction of Batteries.

Several experimental cells will be constructed and tested for electrochemical capability. The choice of electrode combinations and electrolytes will be made from systems exhibiting promising characteristics in half-cell screening tests.

6. Compatibility of Battery Components.

Tests will be initiated to determine the compatibility of various battery separator, case and lead components with electrolytes.

IV. TEST RESULTS

TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES

Page

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm^2

A. Calcium Anode Systems. IV-6

Solvent	Solute
1. Acetonitrile (AN)	Tetra-n-propylammonium hexafluorophosphate - $(n\text{-}C_3H_7)_4NPF_6$
2. AN	Ammonium hexafluorophosphate - NH_4PF_6

B. Lithium Anode Systems. IV-7

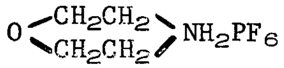
Solvent	Solute
1. Dimethylformamide (DMF)	Di-n-butylammonium hexafluoroarsenate - $(n\text{-}C_4H_9)_2NH_2AsF_6$
2. DMF	Tri-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_3NHAsF_6$
3. DMF	N-phenyl N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$
4. DMF	Ammonium hexafluorophosphate
5. DMF	$KAsF_6$

C. Magnesium Anode Systems. IV-9

Solvent	Solute
1. AN	$AlCl_3$

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2.

A. Calcium Anode Systems. IV-10

Solvent	Solute
1. AN	Tetramethylammonium hexafluorophosphate - $(CH_3)_4NPF_6$
2. DMF	Tetramethylammonium hexafluorophosphate
3. DMF	Morpholinium hexafluorophosphate -  $O\genfrac{<}{>}{0pt}{}{CH_2CH_2}{CH_2CH_2}NH_2PF_6$
4. DMF	$NaPF_6$
5. DMF	$KAsF_6$
6. DMF	KPF_6
7. DMF	LiCl
8. DMF	N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate - $(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$
9. DMF	N-phenyl N,N,N-trimethylammonium hexafluorophosphate

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

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

Page

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)

A. Calcium Anode Systems (Continued) IV-10

10.	Ethylene Carbonate - Propylene Carbonate - 80 WT % EC - 20 WT % PC	$AlCl_3$
11.	80 WT % EC - 20 WT % PC	KPF_6
12.	80 WT % EC - 20 WT % PC	Morpholinium hexafluorophosphate
13.	80 WT % EC - 20 WT % PC	N-phenyl N,N,N-trimethylammonium hexafluorophosphate
14.	80 WT % EC- 20 WT % PC	Tetramethylammonium hexafluorophosphate
15.	N-Nitrosodimethylamine - (NDA)	N-phenyl N,N,N-trimethylammonium hexafluorophosphate
16.	NDA	KPF_6
17.	NDA	Tetramethylammonium hexafluorophosphate

B. Lithium Anode Systems IV-15

1.	DMF	Tetra-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_4NAsF_6$
2.	DMF	Tetra-n-propylammonium hexafluoroantimonate - $(n\text{-}C_3H_7)_4NSbF_6$
3.	DMF	$NaBF_4$
4.	DMF	$NaPF_6$
5.	DMF	Tetramethylammonium hexafluorophosphate
6.	80 WT % EC- 20 WT % PC	Di-n-butylammonium hexafluoroarsenate
7.	80 WT % EC - 20 WT % PC	N-phenyl N,N,N-trimethylammonium hexafluorophosphate
8.	80 WT % EC - 20 WT % PC	Tetramethylammonium hexafluorophosphate
9.	80 WT % EC - 20 WT % PC	Tri-n-propylamlonium hexafluoroarsenate

C. Magnesium Anode Systems IV-18

1.	AN	Ammonium hexafluorophosphate
2.	AN	N-Phenyl N,N,N-trimethylammonium hexafluorophosphate
3.	AN	Morpholinium hexafluorophosphate
4.	AN	KPF_6
5.	AN	Tetramethylammonium hexafluorophosphate

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

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

			Page
II.	Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)		
	C. Magnesium Anode Systems (Continued)		IV-18
6.	AN	Tetra-n-propylammonium hexafluorophosphate	
7.	DMF	KPF_6	
8.	DMF	LiCl	
9.	DMF	N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate	
10.	DMF	Tetramethylammonium hexafluorophosphate	
11.	DMF	Morpholinium hexafluorophosphate	
12.	DMF	Tetra-n-propylammonium hexafluoroarsenate	
13.	80 WT % EC- 20 WT % PC	$AlCl_3$	
14.	NDA	$MgCl_2$	
15.	NDA	N-phenyl N,N,N-trimethylammonium hexafluorophosphate	
	D. Lithium-Magnesium Alloy Anode Systems		IV-22
1.	NDA	Morpholinium hexafluorophosphate	
III.	Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm^2.		
	A. Calcium Anode Systems.		IV-23
1.	DMF	Di-n-butylammonium hexafluoroarsenate	
2.	DMF	Tri-n-propylammonium hexafluoroarsenate	
3.	80 WT % EC - 20 WT % PC	LiCl	
4.	80 WT % EC - 20 WT % PC	Di-n-butylammonium hexafluoroarsenate	
5.	80 WT % EC - 20 WT % PC	Tri-n-propylammonium hexafluoroarsenate	
6.	NDA	LiCl	
7.	NDA	N-(p-Dodecylbenzyl) N,N,N-trimethylammonium hexafluorophosphate	
	B. Lithium Anode Systems		IV-25
1.	80 WT % EC - 20 WT % PC	LiCl	
2.	NDA	LiCl	
	C. Magnesium Anode Systems		IV-26
1.	AN	Di-n-butylammonium hexafluoroarsenate	

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

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

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

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

Page

V. Anode-Electrolyte Systems Which Cannot Sustain 0.1 ma/cm^2. (Continued)

A. Magnesium Anode Systems (Continued) IV-33

3.	80 WT % EC - 20 WT % PC	Tetramethylammonium hexafluorophosphate
4.	NDA	Di-n-butylammonium hexafluoroarsenate
5.	NDA	Tri-n-propylammonium hexafluoroarsenate
6.	NDA	Tetra-n-propylammonium hexafluorophosphate
7.	NDA	KPF_6
8.	NDA	Morpholinium hexafluorophosphate

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm^2.

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm^2.

A. Calcium Anode Systems.

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
1.	AN	*	Ag/AgCl	Ag/AgCl	-2.10	-2.13	0.1	-2.03	-2.04	At 1, 10 and 100 ma, a loosely adhering gray solid formed on the calcium. At 100 ma, gas evolved at the calcium electrode. No change in the appearance of the solution or counter and reference electrodes.
						-2.30	1	-1.75	-1.98	
						-2.32	10	-1.58	-1.72	
						-2.27	100	-1.10	-1.27	
2.	AN	**	Ag/AgCl	Ag/AgCl	-2.13	-2.14	0.1	-2.12	-2.12	Slight gas evolution occurred when the calcium was immersed in the solution. A slight amount of gray solid formed at the anode upon discharge. The counter electrode gassed at 100 ma and the solution became turbid.
						-2.14	1	-2.08	-2.09	
						-2.20	10	-1.74	-1.95	
						-2.21	100	-1.12	-1.45	

* Tetra-n-propylammonium hexafluorophosphate - $(n\text{-}C_3H_7)_4NPF_6$

** Ammonium hexafluorophosphate - NH_4PF_6

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

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm^2. (Continued)
B. Lithium 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	
1.	DMF	*	Ag/AgCl	Ag/AgCl	-3.32	-3.32	0.1	-3.32	-3.32	Vigorous gassing at lithium electrode at all times. Discharges at each current density were limited to two minutes instead of the usual five minutes because the rate of self-discharge appeared to be quite high. Brown product formed at lithium surface.
						-3.31	1	-3.30	-3.29	
						-3.26	10	-3.13	-3.15	
						-3.17	100	-2.32	-2.38	
2.	DMF	**	Ag/AgCl	Ag/AgCl	-3.52	-3.52	0.1	-3.52	-3.52	Vigorous gassing at lithium electrode at all times. Rate of gassing decreased slightly as the test progressed. Discharges at each current density were limited to two minutes because of high apparent rate of self-discharge.
						-3.51	1	-3.50	-3.48	
						-3.28	10	-3.08	-2.95	
						-3.26	100	-2.28	-2.35	
3.	DMF	***	Ag/AgCl	Ag/AgCl	-3.12	-3.18	0.1	-3.12	-3.17	Moderate rate of gassing at lithium after 4 minutes at 100 ma, accompanied by blackening of the lithium. Slow gassing continued on open circuit. The counter electrode deteriorated slightly. No change in appearance of the solution or reference electrode.
						-3.19	1	-3.03	-3.06	
						-3.20	10	-2.77	-2.85	
						-3.18	100	-1.65	-1.77	

* Di-n-butylammonium hexafluoroarsenate - $(n\text{-}C_4H_9)_2NH_2AsF_6$

** Tri-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_3NHAsF_6$

*** N-phenyl N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

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

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm^2. (Continued)
B. Lithium Anode Systems. (Continued)

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
4.	DMF	*	Ag/AgCl	Ag/AgCl	-3.10	-3.03	0.1	-3.08	-3.04	Vigorous gas evolution occurred when the lithium was immersed. The solution became slightly turbid and turned yellow-brown by the 10 ma discharge. A new lithium anode was inserted for the 100 ma discharge because the first anode had dissolved completely. The counter electrode gassed heavily at 100 ma and the anode potential fluctuated considerably. A strong ammonia odor was detected.
						-3.02	1	-3.03	-3.02	
						-3.00	10	-2.96	-2.90	
						-2.99	100	-2.00	-1.80	
5.	DMF	$KAsF_6$	Ag/AgCl	Ag/AgCl	-3.48	-3.46	0.1	-3.42	-3.37	Gas evolution occurred at the lithium anode and at the counter electrode at 100 ma. The solution turned black and turbid.
						-3.43	1	-3.03	-3.10	
						-3.37	10	-2.98	-2.98	
						-3.33	100	-2.22	-2.32	

* Ammonium hexafluorophosphate - NH_4PF_6

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

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm^2. (Continued)

C. Magnesium Anode Systems.

	Electrolyte				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	Reference Electrode	Counter Electrode				Initial	Final	
1.	AN	$AlCl_3$	Ag/AgCl	Ag/AgCl	-1.79	-1.82	0.1	-1.79	-1.82	No changes in the appearance of the entire system.
						-1.82	1	-1.74	-1.75	
						-1.82	10	-1.63	-1.63	
						-1.82	100	-0.95	-1.08	

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2.

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

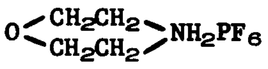
TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES (Continued)

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2.
A. Calcium Anode Systems.

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
1.	AN	*	Ag/AgCl	Ag/AgCl	-2.00	-1.97	0.1	-1.94	-1.92	After the 100 ma discharge, a small amount of white solid was suspended in the electrolyte. No change in appearance of calcium or counter and reference electrodes.
						-2.07	1	-1.65	-1.77	
						-2.08	10	-1.45	-1.34	
						-2.10	100	Reversed Polarity	----	
2.	DMF	*	Ag/AgCl	Ag/AgCl	-2.32	-2.25	0.1	-2.32	-2.27	At 50 ma, voltage was initially -1.00 but reversed to +0.25 within 30 seconds. Calcium became blackened upon discharge at 0.1 ma.
						-2.17	1	-2.15	-2.12	
						-2.49	10	-1.50	-1.96	
						-2.22	100	-0.50	Reversed Polarity	
3.	DMF	**	Ag/AgCl	Ag/AgCl	-2.44	-2.39	0.1	-1.98	-1.98	At 50 ma, voltage varied between -0.60 and -0.79. Black material formed at calcium surface and diffused into the solution.
						-2.53	1	-1.65	-1.92	
						-2.57	10	-1.00	-1.63	
						-2.48	100	Reversed Polarity	----	

* Tetramethylammonium hexafluorophosphate - $(CH_3)_4NPF_6$

** Morpholinium hexafluorophosphate -



$O\begin{matrix}\diagup CH_2CH_2 \diagdown \\ \diagdown CH_2CH_2 \diagup\end{matrix}NH_2PF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2.(Continued)
A. Calcium Anode Systems. (continued)

	Electrolyte				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	Reference Electrode	Counter Electrode				Initial	Final	
4.	DMF	$NaPF_6$	Ag/AgCl	Ag/AgCl	-2.25	-2.48	0.1	-2.05	-2.04	Calcium blackened during the 10 ma and 100 ma discharges and gas evolution began at the counter electrode at 100 ma. No change in the appearance of the reference electrode or the solution.
						-2.57	1	-2.04	-2.03	
						-2.63	10	-1.97	-1.88	
						-2.65	100	Reversed Polarity	---	
5.	DMF	$KAsF_6$	Ag/AgCl	Ag/AgCl	-2.18	-2.23	0.1	-1.75	-1.75	Calcium blackened during the 10 ma and 100 ma discharges and gassed at 100 ma. The counter electrode began gassing at 100 ma with the formation of a black solid.
						-2.43	1	-1.60	-1.72	
						-2.48	10	-1.48	-1.56	
						-2.36	100	-0.25	-0.24	
6.	DMF	KPF_6	Ag/AgCl	Ag/AgCl	-2.36	-2.38	0.1	-2.07	-2.04	At 100 ma calcium blackened, the counter electrode gassed vigorously, and the solution changed from colorless to brown.
						-2.45	1	-2.00	-1.98	
						-2.58	10	-1.87	-1.84	
						-2.63	100	-1.14	-1.03	
7.	DMF	LiCl	Ag/AgCl	Ag/AgCl	-2.22	-2.17	0.1	-2.10	-2.15	Both counter and reference electrodes deteriorated to some extent. Beginning at the 1 ma discharge, the calcium gassed continuously. At 10 ma, the calcium blackened and a brown turbidity appeared in the solution around it.
						-1.95	1	-2.05	-2.10	
						-1.80	10	-1.78	-1.11	
						-1.85	100	Reversed Polarity	----	

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
A. Calcium Anode Systems (Continued)

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
8.	DMF	*	Ag/AgCl	Ag/AgCl	-2.16	-2.34	0.1	-2.02	-2.02	At 100 ma, calcium blackened, the solution changed from colorless to a turbid brown, and the counter electrode deteriorated slightly.
						-2.41	1	-2.02	-2.02	
						-2.42	10	-1.80	-1.82	
						-2.42	100	-0.65	Reversed Polarity	
9.	DMF	**	Ag/AgCl	Ag/AgCl	-1.88	-1.87	0.1	-1.70	-1.82	After the 10 ma discharge, the calcium gassed at all times. It also blackened with the formation of a black turbidity under it. At 50 ma, voltage varied between -1.0 and -1.38. No changes in counter or reference electrodes.
						-2.03	1	-1.40	-1.82	
						-2.23	10	-1.00	-1.77	
						-2.43	100	Reversed Polarity	----	
10.	80 WT % EC 20 WT % PC	$AlCl_3$	Ag/AgCl	Ag/AgCl	-2.57	-2.57	0.1	-2.55	-2.57	Calcium gassed slowly at all times. No changes in appearance of the solution or counter and reference electrodes.
						-2.57	1	-2.52	-2.53	
						-2.57	10	-2.18	-2.20	
						-2.57	100	Reversed Polarity	----	
11.	80 WT % EC 20 WT % PC	KPF_6	Ag/AgCl	Ag/AgCl	-2.57	-2.32	0.1	-1.74	-1.81	Moderate gassing at calcium at 100 ma. No changes in the appearance of the solution or counter and reference electrodes.
						-2.28	1	-1.72	-1.72	
						-2.23	10	-1.50	-1.49	
						-2.39	100	Reversed Polarity	----	

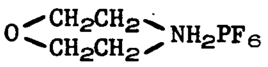
* N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate - $(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$

** N-phenyl N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
A. Calcium Anode Systems. (Continued)

	Electrolyte: Solvent	Electrolyte: 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: Initial	Voltage of Anode vs. Reference Electrode at Various Current Densities: Final	Further Observations and Remarks
12.	80 WT % EC 20 WT % PC	*	Ag/AgCl	Ag/AgCl	-2.32	-2.24 -2.47 -2.48 -2.48	0.1 1 10 100	-2.18 -1.85 -1.65 Reversed Polarity	-1.94 -2.04 -1.35 ----	Calcium gassed slowly at all times and became darkened at 10 ma.
13.	80 WT % EC 20 WT % PC	**	Ag/AgCl	Ag/AgCl	-2.04	-2.09 -2.13 -2.23 -2.20	0.1 1 10 100	-1.99 -1.85 -1.35 Reversed Polarity	-2.06 -1.90 -1.32 ----	Calcium began gassing at 0.1 ma and continued to do so at all times until coated gray-black during the 100 ma discharge. No changes in the appearance of the solution or counter and reference electrodes.
14.	80 WT % EC 20 WT % PC	***	Ag/AgCl	Ag/AgCl	-2.07	-2.11 -2.17 -2.18 -2.15	0.1 1 10 100	-1.53 -1.53 -1.35 Reversed Polarity	-1.64 -1.56 -1.34 ----	At 100 ma, calcium gassed vigorously and a gray film formed at the surface. No changes in the appearance of the solution or counter and reference electrodes.
15.	NDA	**	Ag/AgCl	Ag/AgCl	-1.83	-1.77 -1.88 -2.08 -2.10	0.1 1 10 100	-1.58 -1.26 -1.25 Reversed Polarity	-1.54 -1.43 -1.32 ----	At 10 ma, calcium began to gas and continued to do so at all times; solid formed at the calcium surface. No changes in the appearance of the solution or counter and reference electrodes.

* Morpholinium hexafluorophosphate - 
$O\begin{matrix}CH_2CH_2\\CH_2CH_2\end{matrix}NH_2PF_6$

** N-phenyl N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

*** Tetramethylammonium hexafluorophosphate - $(CH_3)_4NPF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
A. Calcium Anode Systems. (Continued)

	Electrolyte				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	Reference Electrode	Counter Electrode				Initial	Final	
16.	NDA	KPF_6	Ag/AgCl	Ag/AgCl	-2.30	-2.18	0.1	-1.50	-1.62	Calcium became blackened during the discharges.
						-2.13	1	-1.40	-1.43	
						-2.10	10	-1.27	-1.28	
						-2.15	100	Reversed Polarity	----	
17.	NDA	*	Ag/AgCl	Ag/AgCl	-2.18	-2.24	0.1	-1.54	-1.69	Moderate rate of gas evolution at calcium at 10 ma/cm^2. Calcium somewhat darkened. Gas evolution at counter electrode.
						-2.28	1	-1.50	-1.63	
						-2.32	10	-1.23	-1.10	
						-2.30	100	Reversed Polarity	----	

* Tetramethylammonium hexafluorophosphate - $(CH_3)_4NPF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
B. Lithium Anode Systems.

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
1.	DMF	*	Ag/AgCl	Ag/AgCl	-3.23	-3.20	0.1	-3.22	-3.22	The solution became slightly yellow during the 1 ma discharge and a red-brown solid began forming at the lithium anode which remained suspended in the electrolyte. No change in the appearance of the counter reference electrodes.
						-3.07	1	-3.17	-3.05	
						-3.05	10	-2.57	-2.76	
						-3.04	100	Reversed Polarity	---	
2.	DMF	**	Ag/AgCl	Ag/AgCl	-3.23	-3.20	0.1	-3.23	-3.20	A black, loosely adherent solid formed on the lithium anode during discharge. No change in the appearance of the counter and reference electrodes or in electrolyte.
						-3.18	1	-3.14	-3.13	
						-3.20	10	-2.80	-2.95	
						-3.07	100	-0.85	Reversed Polarity	
3.	DMF	NaBF$_4$	Ag/AgCl	Ag/AgCl	-3.31	-3.31	0.1	-3.18	-3.20	Gas evolution and deterioration occurred at the counter electrode at 100 ma and the solution became slightly yellow. No change in the appearance of the lithium or reference electrodes.
						-3.27	1	-2.80	-3.15	
						-3.20	10	-2.68	-2.88	
						-3.10	100	-1.40	-1.20	
4.	DMF	NaPF$_6$	Ag/AgCl	Ag/AgCl	-3.58	-3.51	0.1	-3.43	-3.43	Gas evolution at the counter electrode occurred at 100 ma. The solution turned slightly yellow and a brown solid formed at the lithium anode.
						-3.53	1	-3.18	-3.38	
						-3.30	10	-2.60	-2.65	
						-3.10	100	-1.20	-0.60	

* Tetra-n-propylammonium hexafluoroarsenate -$(n\text{-}C_3H_7)_4NAsF_6$

** Tetra-n-propylammonium hexafluoroantimonate -$(n\text{-}C_3H_7)_4NSbF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
B. Lithium Anode Systems. (Continued)

	Electrolyte Solvent	Electrolyte 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 Initial	Voltage of Anode vs. Reference Electrode at Various Current Densities Final	Further Observations and Remarks
5.	DMF	*	Ag/AgCl	Ag/AgCl	-3.41	-3.42	0.1	-3.35	-3.35	At 100 ma the counter electrode gassed vigorously and became blackened. A brown, non-adherent product formed at the lithium electrode which produced a turbidity in the solution. The lithium remained bright in appearance.
						-3.41	1	-3.08	-3.21	
						-3.36	10	-2.74	-2.83	
						-3.03	100	-0.55	Reversed Polarity	
6.	80 WT % EC 20 WT % PC	**	Ag/AgCl	Ag/AgCl	-3.12	-3.09	0.1	-3.11	-3.09	Vigorous gassing at lithium electrode at all times. At 100 ma, lithium blackened and gas evolution occurred at the counter electrode. No changes in the appearance of the solution or reference electrode.
						-3.08	1	-3.06	-3.05	
						-3.08	10	-2.81	-2.87	
						-2.99	100	-1.18	Reversed Polarity	
7.	80 WT % EC 20 WT % PC	***	Ag/AgCl	Ag/AgCl	-2.96	-2.98	0.1	-2.95	-2.98	Lithium gassed slowly at all times and became darkened. No changes in the appearance of the solution or counter and reference electrodes.
						-2.98	1	-2.90	-2.92	
						-3.00	10	-2.53	-2.63	
						-2.98	100	Reversed Polarity	----	
8.	80 WT % EC 20 WT % PC	*	Ag/AgCl	Ag/AgCl	-3.26	-3.20	0.1	-3.13	-3.04	No changes in the appearance of the entire system.
						-3.14	1	-2.85	-2.87	
						-2.98	10	-2.45	-2.42	
						-3.00	100	Reversed Polarity	----	

* Tetramethylammonium hexafluorophosphate - $(CH_3)_4NPF_6$

** Di-n-butylammonium hexafluoroarsenate - $(n\text{-}C_4H_9)_2NH_2AsF_6$

*** N-phenyl N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
B. Lithium Anode Systems. (Continued)

	Electrolyte				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	Reference Electrode	Counter Electrode				Initial	Final	
9.	80 WT % EC 20 WT % PC	*	Ag/AgCl	Ag/AgCl	-3.20	-3.17	0.1	-3.20	-3.18	Lithium gassed moderately at all times. No changes in the appearance of the solution or counter and reference electrodes.
						-3.11	1	-3.13	-3.10	
						-3.04	10	-2.82	-2.83	
						-2.98	100	Reversed Polarity	----	

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* Tri-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_3NHAsF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)

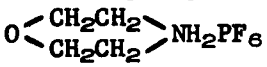
C. Magnesium Anode Systems.

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
1.	AN	*	Ag/AgCl	Ag/AgCl	-1.58	-1.53	0.1	-1.55	-1.50	Gas evolution occurred at the magnesium anode during discharge. A loosely adherent gray solid formed on the magnesium at 100 ma.
						-1.48	1	-1.24	-1.35	
						-1.60	10	-1.13	-1.13	
						-1.57	100	-0.75	-0.73	
2.	AN	**	Ag/AgCl		-1.25	-1.17	0.1	-1.23	-1.20	At 50 ma, voltage varied between -0.78 and -0.68. A black solid formed at magnesium surface which did not adhere. The solution became brown colored and turbid.
						-1.13	1	-1.10	-1.13	
						-1.42	10	-1.00	-0.96	
						-1.50	100	-0.58	-0.49	
3.	AN	***	Ag/AgCl		-1.23	-1.20	0.1	-1.18	-1.21	At 50 ma, voltage varied between -1.05 and -0.81. Magnesium became blackened and gassed on open circuit after the discharges. Solution became cloudy.
						-1.22	1	-1.15	-1.19	
						-1.28	10	-0.90	-1.06	
						-1.68	100	-0.40	-0.50	
4.	AN	KPF$_6$	Ag/AgCl	Ag/AgCl	-1.28	-1.22	0.1	-1.14	-1.12	At 100 ma, magnesium darkened and the counter electrode gassed vigorously. No changes in the appearance of the solution or counter and reference electrodes.
						-1.24	1	-1.11	-1.11	
						-1.41	10	-1.04	-1.04	
						-1.52	100	-0.47	-0.34	

* Ammonium hexafluorophosphate - NH_4PF_6

** N-Phenyl N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

*** Morpholinium hexafluorophosphate -



$O\begin{smallmatrix} \diagup CH_2CH_2 \diagdown \\ \diagdown CH_2CH_2 \diagup \end{smallmatrix}NH_2PF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
C. Magnesium Anode Systems. (Continued)

	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	
5.	AN	*	Ag/AgCl	Ag/AgCl	-1.23	-1.23	0.1	-1.15	-1.13	No change in the appearance of the entire system.
						-1.16	1	-0.98	-0.95	
						-1.24	10	-0.78	-0.77	
						-1.12	100	Reversed Polarity	----	
6.	AN	**	Ag/AgCl	Ag/AgCl	-1.27	-1.32	0.1	-1.15	-1.25	At 100 ma, magnesium gassed and a solid formed at the surface. No change in the appearance of the counter or reference electrode.
						-1.32	1	-1.07	-1.25	
						-1.30	10	-0.98	-1.05	
						-1.75	100	-0.50	-0.40	
7.	DMF	KPF_6	Ag/AgCl	Ag/AgCl	-1.24	-1.13	0.1	-1.15	-1.13	At 100 ma, the counter electrode gassed vigorously. The appearance of the magnesium, the solution, and the reference electrode was unchanged.
						-1.19	1	-1.08	-1.12	
						-1.40	10	-1.02	-1.03	
						-1.63	100	Reversed Polarity	----	
8.	DMF	LiCl	Ag/AgCl	Ag/AgCl	-1.72	-1.68	0.1	-1.67	-1.68	The counter and reference electrodes both deteriorated to some extent. Magnesium blackened and gassed continuously after discharge at 10 ma. At 100 ma, a brown turbidity appeared around the magnesium.
						-1.21	1	-1.58	-0.90	
						-1.55	10	-1.00	-1.18	
						-1.40	100	Reversed Polarity	----	

* Tetramethylammonium hexafluorophosphate - $(CH_3)_4NPF_6$

** Tetra-n-propylammonium hexafluorophosphate - $(n\text{-}C_3H_7)_4NPF_6$

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

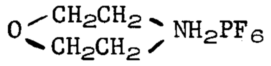
II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
C. Magnesium Anode Systems. (Continued)

	Electrolyte				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	Reference Electrode	Counter Electrode				Initial	Final	
9.	DMF	*	Ag/AgCl	Ag/AgCl	-1.50	-1.38 -1.57 -1.68 -1.60	0.1 1 10 100	-1.19 -1.18 -1.00 Reversed Polarity	-1.23 -1.20 -1.07 ----	Counter electrode deteriorated. No change in appearance of the magnesium, the reference electrode, or the solution.
10.	DMF	**	Ag/AgCl		-1.20	-1.28 -1.27 -1.25 -1.08	0.1 1 10 100	-0.90 -0.80 -0.70 Reversed Polarity	-1.20 -1.15 -0.93 ----	Magnesium became darkened slightly at high current densities. Solution color deepened somewhat. Counter and reference electrodes deteriorated somewhat.
11.	DMF	***	Ag/AgCl		-1.56	-1.59 -1.58 -1.50 -1.56	0.1 1 10 100	-1.53 -1.10 -0.70 -0.60	-1.57 -1.47 -1.30 Reversed Polarity	Magnesium became darkened and began to gas at 10 ma.
12.	DMF	****	Ag/AgCl	Ag/AgCl	-1.58	-1.60 -1.60 -1.40 -1.58	0.1 1 10 100	-1.54 -1.54 -1.38 Reversed Polarity	-1.55 -1.58 -1.46 ---	The magnesium anode became slightly coated. No change in the appearance of the counter and reference electrodes or in the electrolyte.

* N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate - $(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$

** Tetramethylammonium hexafluorophosphate - $(CH_3)_4NPF_6$

*** Morpholinium hexafluorophosphate -



$$O\begin{matrix}CH_2CH_2\\CH_2CH_2\end{matrix}NH_2PF_6$$

**** Tetra-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_4NAsF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
C. Magnesium Anode Systems. (Continued)

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
13.	80 WT % EC 20 WT % PC	$AlCl_3$	Ag/AgCl	Ag/AgCl	-1.68	-1.80	0.1	-1.68	-1.77	Magnesium began gassing at 1 ma and continued to do so at all times. No change in the appearance of the solution or counter and reference electrodes.
						-1.80	1	-1.70	-1.70	
						-1.77	10	-1.25	-1.32	
						-1.77	100	Reversed Polarity	----	
14.	NDA	$MgCl_2$	Ag/AgCl	Ag/AgCl	-0.90	-1.07	0.1	-0.88	-0.88	No change in the appearance of the entire system.
						-1.13	1	-0.60	-0.96	
						-1.26	10	-0.50	-0.70	
						-1.23	100	Reversed Polarity	----	
15.	NDA	*	Ag/AgCl	Ag/AgCl	-1.57	-1.60	0.1	-1.56	-1.58	Magnesium began to gas during the 0.1 ma discharge and continued to do so at all times until the current was increased to 100 ma. No changes in the appearance of the solution or counter and reference electrodes.
						-1.57	1	-1.51	-1.33	
						-1.49	10	-1.30	-1.13	
						-1.47	100	Reversed Polarity	----	

* N-phenyl N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

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

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2. (Continued)
D. Lithium-Magnesium Alloy* Anode System.

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
1.	NDA	**	Ag/AgCl	Ag/AgCl	-2.32	-2.24	0.1	-2.32	-2.24	Continuous gassing occurred at the anode. The reversal in polarity at 100 ma occurred in less than one minute. The counter electrode began gassing at 100 ma. No change in the appearance of the reference electrode.
						-2.12	1	-2.23	-2.12	
						-1.98	10	-2.02	-1.89	
						-1.97	100	-0.63	Reversed Polarity	

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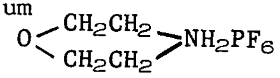
* 60 wt. % lithium; 40 wt. % magnesium

** Morpholinium hexafluorophosphate -



$O\begin{matrix}CH_2CH_2\\CH_2CH_2\end{matrix}NH_2PF_6$

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

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

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

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

A. Calcium Anode Systems.

	Electrolyte Solvent	Electrolyte 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: Initial	Voltage of Anode vs. Reference Electrode at Various Current Densities: Final	Further Observations and Remarks
1.	DMF	*	Ag/AgCl	Ag/AgCl	-1.53	-1.51	0.1	-1.35	-1.20	Calcium darkened at 10 ma. No change in the appearance of the solution or counter and reference electrodes.
						-1.85	1	-0.84	-1.08	
						-2.13	10	-0.25	-0.13	
						-2.59	100	Reversed Polarity	----	
2.	DMF	**	Ag/AgCl	Ag/AgCl	-1.84	-1.82	0.1	-1.52	-1.63	At 1 ma, calcium became blackened. No change in the appearance of the solution or counter and reference electrodes.
						-1.90	1	-1.08	-1.02	
						-2.08	10	-0.20	-0.15	
						-2.17	100	Reversed Polarity	----	
3.	80 WT % EC 20 WT % PC	LiCl	Ag/AgCl	Ag/AgCl	-2.43	-2.43	0.1	-2.26	-2.31	No change in the appearance of the entire system.
						-2.45	1	-1.90	-2.08	
						-2.45	10	Reversed Polarity	----	
						----	100	----	----	
4.	80 WT % EC 20 WT % PC	*	Ag/AgCl	Ag/AgCl	-1.79	-1.82	0.1	-1.68	-1.73	Calcium began to gas during 10 ma discharge and continued to do so at all times. Calcium surface became light brown in appearance. No change in the appearance of the solution or reference and counter electrodes.
						-2.28	1	-1.68	-1.68	
						-2.20	10	-0.45	-0.30	
						-2.20	100	Reversed Polarity	----	

* Di-n-butylammonium hexafluoroarsenate - $(n\text{-}C_4H_9)_2NH_2AsF_6$

** Tri-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_3NHAsF_6$

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm^2. (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 Initial	Final	Further Observations and Remarks
5.	80 WT % EC 20 WT % PC	*	Ag/AgCl	Ag/AgCl	-2.13	-2.40	0.1	-1.18	-1.87	Calcium darkened; counter electrode deteriorated. No change in the appearance of the solution or the reference electrode.
						-2.37	1	-1.70	-1.70	
						-2.08	10	-0.32	-0.20	
						-1.75	100	Reversed Polarity	----	
6.	NDA	LiCl	Ag/AgCl	Ag/AgCl	-2.30	-2.30	0.1	-2.18	-2.12	No change in the appearance of the entire system.
						-2.30	1	-1.20	-1.92	
						-2.30	10	Reversed Polarity	----	
						----	100	----	----	
7.	NDA	**	Ag/AgCl	Ag/AgCl	-2.22	-2.27	0.1	-1.60	-1.69	No change in the appearance of the entire system.
						-2.40	1	-0.70	-1.49	
						-2.18	10	Reversed Polarity	----	
						----	100	----	----	

* Tri-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_3NHAsF_6$

** N-(p-Dodecylbenzyl) N,N,N-trimethylammonium hexafluorophosphate - $(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm^2. (Continued)
B. Lithium Anode Systems.

	Electrolyte				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	Reference Electrode	Counter Electrode				Initial	Final	
1.	80 WT % EC 20 WT % PC	LiCl	Ag/AgCl	Ag/AgCl	-2.83	-2.83	0.1	-2.80	-2.80	No change in the appearance of the entire system.
						-2.83	1	-2.58	-2.62	
						-2.82	10	-0.80	-0.68	
						-2.82	100	Reversed Polarity	----	
2.	NDA	LiCl	Ag/AgCl	Ag/AgCl	-2.84	-2.84	0.1	-2.82	-2.83	Lithium became dull gray in appearance. No change in the appearance of the solution or counter and reference electrodes.
						-2.84	1	-2.62	-2.60	
						-2.81	10	-0.50	-0.35	
						-2.78	100	Reversed Polarity	----	

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm^2. (Continued)
C. Magnesium Anode Systems.

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
1.	AN	*	Ag/AgCl	Ag/AgCl	-0.74	-0.78	0.1	-0.68	-0.60	Magnesium became dull gray in appearance. No changes in the appearance of the solution or counter and reference electrodes.
						-0.90	1	-0.40	-0.47	
						-0.93	10	-0.35	Reversed Polarity	
						----	100	----	----	
2.	AN	**	Ag/AgCl	Ag/AgCl	-1.09	-1.10	0.1	-1.09	-1.09	A reversal in polarity occurred after 15 seconds during the 10 ma/cm^2 discharge. No change in the appearance of the system.
						-1.08	1	-1.05	-1.03	
						-1.05	10	-0.15	Reversed Polarity	
						---	100	---	---	
3.	DMF	$NaPF_6$	Ag/AgCl	Ag/AgCl	-1.69	-1.73	0.1	-1.30	-1.52	Magnesium darkened slightly. No change in the appearance of the counter and reference electrodes or the electrolyte.
						-1.71	1	-1.08	-1.27	
						-1.70	10	Reversed Polarity	---	
						---	100	---	---	
4.	80 WT % EC 20 WT % PC	LiCl	Ag/AgCl	Ag/AgCl	-1.30	-1.33	0.1	-1.25	-1.26	No change in the appearance of the entire system.
						-1.60	1	-1.00	-0.98	
						-1.62	10	Reversed Polarity	----	
						----	100	----	----	

* Di-n-butylammonium hexafluoroarsenate - $(n\text{-}C_4H_9)_2NH_2AsF_6$

** Tetra-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_4NAsF_6$

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

III. Anode-Electrolyte Systems Capable of Sustaining 1 ma/cm^2. (Continued)
C. Magnesium Anode Systems. (Continued)

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
5.	80 WT % EC 20 WT % PC	*	Ag/AgCl	Ag/AgCl	-1.20	-1.00 -1.28 -1.32 ----	0.1 1 10 100	-0.78 -0.67 Reversed Polarity ----	-0.81 -0.72 ---- ----	Magnesium blackened. No change in the appearance of the solution or counter and reference electrodes.
6.	80 WT % EC 20 WT % PC	**	Ag/AgCl	Ag/AgCl	-1.53	-1.44 -1.40 -1.53 -1.58	0.1 1 10 100	-1.50 -1.32 -0.65 Reversed Polarity	-1.45 -1.33 -0.50 ----	Magnesium gassed slowly at all times. No change in the appearance of the solution or counter and reference electrodes.
7.	80 WT % EC 20 WT % PC	***	Ag/AgCl	Ag/AgCl	-1.23	-1.27 -1.57 -1.60 ----	0.1 1 10 100	-0.72 -0.63 Reversed Polarity ----	-0.82 -0.62 ---- ----	Black streaks appeared at the magnesium surface and the counter electrode deteriorated somewhat. No change in the appearance of the solution or the reference electrode.
8.	80 WT % EC 20 WT % PC	KPF_6	Ag/AgCl	Ag/AgCl	-1.38	-1.38 -1.58 -1.54 ----	0.1 1 10 100	-0.72 -0.72 Reversed Polarity ----	-0.88 -0.80 ---- ----	No change in the appearance of the entire system.

* Di-n-butylammonium hexafluoroarsenate - $(n\text{-}C_4H_9)_2NH_2AsF_6$

** N-phenyl N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

*** Tri-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_3NHAsF_6$

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

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

C. Magnesium Anode Systems. (Continued)

	Electrolyte				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	Reference Electrode	Counter Electrode				Initial	Final	
9.	NDA	LiCl	Ag/AgCl	Ag/AgCl	-1.00	-1.32	0.1	-0.65	-0.87	No change in the appearance of the entire system.
						-1.32	1	-0.43	-0.52	
						-1.00	10	Reversed Polarity	----	
						----	100	----	----	

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

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

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

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

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
1.	DMF	*	Ag/AgCl	Ag/AgCl	-2.17	-2.02	0.1	-1.50	-1.90	No changes in the appearance of the entire system.
						-2.05	1	-1.04	-0.55	
						-2.04	10	Reversed Polarity	---	
						---	100	---	---	
2.	NDA	**	Ag/AgCl	Ag/AgCl	-1.68	-1.73	0.1	-1.53	-0.95	Calcium blackened. No change in the appearance of the solution or the counter and reference electrodes.
						-1.88	1	-0.75	-0.59	
						-1.80	10	-0.75	-0.15	
						-1.87	100	Reversed Polarity	----	
3.	NDA	***	Ag/AgCl	Ag/AgCl	-1.48	-1.58	0.1	-1.05	-0.78	No change in the appearance of the entire system. The discharge at 10 ma was discontinued after one minute.
						-1.67	1	-0.48	-0.50	
						-1.98	10	-0.05	-0.14	
						----	100	----	----	

* Tetra-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_4NAsF_6$

** Di-n-butylammonium hexafluoroarsenate - $(n\text{-}C_4H_9)_2NH_2AsF_6$

*** Tri-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_3NHAsF_6$

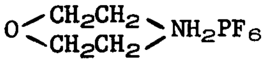
TABLE I. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES (Continued)

IV. Anode-Electrolyte Systems Capable of Sustaining 0.1 ma/cm^2. (Continued)
B. Magnesium Anode Systems.

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
1.	DMF	*	Ag/AgCl	Ag/AgCl	-1.47	-1.47	0.1	-1.45	-1.47	Magnesium gassed at 10 ma and black streaks appeared at its surface. No change in the appearance of the solution or counter and reference electrodes.
						-1.47	1	-0.90	-0.63	
						-1.32	10	-0.40	-0.53	
						-1.29	100	Reversed Polarity	----	
2.	DMF	KAsF$_6$	Ag/AgCl	Ag/AgCl	-1.10	-1.37	0.1	-0.60	-1.19	Magnesium blackened during the 10 ma and 100 ma/cm^2 discharges. At 10 ma the anode potential fluctuated continuously between reversal and -0.42 V. No change in the appearance of the counter and reference electrodes or the electrolyte.
						-1.47	1	-0.55	-0.46	
						-1.43	10	-0.42	-0.42	
						-1.04	100	Reversed Polarity	---	
3.	80 WT % EC 20 WT % PC	**	Ag/AgCl	Ag/AgCl	-1.22	-1.20	0.1	-0.85	-0.99	Counter electrode had partially deteriorated. No change in the appearance of the rest of the system.
						-1.18	1	Reversed Polarity	----	
						----	10	----	----	
						----	100	----	----	
4.	NDA	***	Ag/AgCl	Ag/AgCl	-0.78	-0.79	0.1	-0.60	-0.50	No change in the appearance of the entire system.
						-0.88	1	Reversed Polarity	----	
						----	10	----	----	
						----	100	----	----	

* N-phenyl N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

** Morpholinium hexafluorophosphate -



$$O\left\langle\begin{matrix}CH_2CH_2\\CH_2CH_2\end{matrix}\right\rangle NH_2PF_6$$

*** N-(p-Dodecylbenzyl) N,N,N-trimethylammonium hexafluorophosphate - $(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$

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

IV. Anode-Electrolyte Systems Capable of Sustaining 0.1 ma/cm^2. (Continued)
B. Magnesium Anode Systems. (Continued)

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
5.	NDA	*	Ag/AgCl	Ag/AgCl	-0.92	-1.03	0.1	-0.70	-0.57	No change in the appearance of the entire system.
						-0.93	1	Reversed Polarity	----	
						----	10	----	----	
						----	100	----	----	

* Tetramethylammonium hexafluorophosphate - $(CH_3)_4NPF_6$

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

IV. Anode-Electrolyte Systems Capable of Sustaining 0.1 ma/cm^2. (Continued)
C. Lithium-Magnesium Alloy* Anode System.

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
1.	NDA	**	Ag/AgCl	Ag/AgCl	-1.20	-1.13	0.1	-1.08	-0.90	No change in the appearance of the system.
						-1.20	1	Reversed Polarity	---	
						---	10	---	---	
						---	100	---	---	

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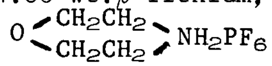
* Foote Alloy LA 141 consisting of 14.00 wt.% lithium; 84.70 wt.% magnesium; 1.00% aluminum; 0.30% impurities

** Morpholinium hexafluorophosphate -



$O\langle{}^{CH_2CH_2}_{CH_2CH_2}\rangle NH_2PF_6$

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

Each system is classified under the current density at which the anode exhibited less than 50% polarization of the initial open circuit potential.

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

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

A. Magnesium Anode Systems.

	Electrolyte				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	Reference Electrode	Counter Electrode				Initial	Final	
1.	DMF	*	Ag/AgCl	Ag/AgCl	-1.15	-1.08	0.1	-0.70	Reversed Polarity	No change in the appearance of the entire system.
						----	1	----	----	
						----	10	----	----	
						----	100	----	----	
2.	DMF	**	Ag/AgCl	Ag/AgCl	-1.34	-1.27	0.1	-0.64	-0.64	No change in the appearance of the entire system.
						-1.42	1	Reversed Polarity	----	
						----	10	----	----	
						----	100	----	----	
3.	80 WT % EC 20 WT % PC	***	Ag/AgCl	Ag/AgCl	-1.93	-1.32	0.1	-0.90	-0.67	Magnesium became blackened and gassed during discharge. No change in the appearance of the solution or reference and counter electrodes.
						-1.30	1	-0.55	-0.75	
						-1.85	10	-0.28	Reversed Polarity	
						----	100	----	----	
4.	NDA	*	Ag/AgCl	Ag/AgCl	-1.15	-0.88	0.1	-0.78	Reversed Polarity	No change in the appearance of the entire system.
						----	1	----	----	
						----	10	----	----	
						----	100	----	----	

* Di-n-butylammonium hexafluoroarsenate - $(n\text{-}C_4H_9)_2NH_2AsF_6$

** Tri-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_3NHAsF_6$

*** Tetramethylammonium hexafluorophosphate - $(CH_3)_4NPF_6$

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

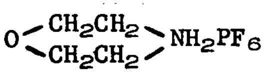
V. Anode-Electrolyte Systems Which Cannot Sustain 0.1 ma/cm^2. (Continued)
A. Magnesium Anode Systems. (Continued)

	Electrolyte							Voltage of Anode vs. Reference Electrode at Various Current Densities		
	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)	Initial	Final	Further Observations and Remarks
5.	NDA	*	Ag/AgCl	Ag/AgCl	-1.07	-0.95	0.1	-0.65	Reversed Polarity	Polarity reversed after 45 seconds at 0.1 ma. No change in the appearance of the entire system.
						----	1	----	----	
						----	10	----	----	
						----	100	----	----	
6.	NDA	**	Ag/AgCl	Ag/AgCl	-0.83	-0.79	0.1	-0.50	Reversed Polarity	Polarity reversed after 2 minutes at 0.1 ma. No change in the appearance of the entire system.
						----	1	----	----	
						----	10	----	----	
						----	100	----	----	
7.	NDA	KPF$_6$	Ag/AgCl	Ag/AgCl	-1.13	-0.90	0.1	-0.40	+0.33	No changes in the appearance of the system.
						-0.98	1	+0.75	+0.98	
						----	10	----	----	
						----	100	----	----	
8.	NDA	*	Ag/AgCl	Ag/AgCl	-0.73	-0.75	0.1	+0.80	+1.60	No changes in the appearance of the system.
						----	1	----	----	
						----	10	----	----	
						----	100	----	----	

* Tri-n-propylammonium hexafluoroarsenate - $(n\text{-}C_3H_7)_3NHAsF_6$

** Tetra-n-propylammonium hexafluorophosphate - $(n\text{-}C_3H_7)_4NPF_6$

*** Morpholinium hexafluorophosphate -



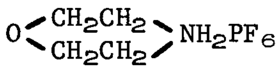
$$O\begin{matrix} \diagup CH_2CH_2 \diagdown \\ \diagdown CH_2CH_2 \diagup \end{matrix}NH_2PF_6$$

TABLE II. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES IN PRETREATED ELECTROLYTES

Page

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm^2.

A. Lithium Anode Systems. IV-36

	Solvent	Solute
1.	Dimethylformamide (DMF)	NH_4PF_6 - Ammonium hexafluorophosphate
2.	N-Nitrosodimethylamine (NDA)	 $O(CH_2CH_2)_2NH_2PF_6$ - Morpholinium hexafluorophosphate

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2.

A. Lithium Anode Systems. IV-38

	Solvent	Solute
1.	NDA	$(n\text{-}C_3H_7)_3NHAsF_6$ - Tri-n-propylammonium hexafluoroarsenate

TABLE II. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES IN PRETREATED ELECTROLYTES

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm^2.
A. 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: Initial	Final	Further Observations and Remarks
1.	DMF	*	Ag/AgCl	Ag/AgCl	-3.18	-3.17	0.1	-3.18	-3.17	Lithium blackened and the solution became turbid during discharge. No change in the appearance of the counter and reference electrodes.
						-3.18	1	-3.14	-3.15	
						-3.19	10	-2.97	-3.01	
						-3.15	100	-2.02	-1.93	

* NH_4PF_6 - Ammonium hexafluorophosphate (1 molal). The solution was pretreated with lithium metal until gas evolution ceased. An appreciable quantity of gas was evolved (ammonia odor) and the color of the electrolyte changed from colorless to yellow. No solid was formed. The conductance of the resultant solution was 2.09×10^{-2} ohm^{-1} cm^{-1} as compared to 2.96×10^{-2} ohm^{-1} cm^{-1} for the untreated solution.

The results for the same system (not pretreated with lithium) are listed below. Conductance of the electrolyte was 2.96×10^{-2} ohm^{-1} cm^{-1}.

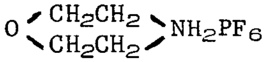
DMF	**	Ag/AgCl	Ag/AgCl	-3.10	-3.03	0.1	-3.08	-3.04	Vigorous gas evolution occurred when the lithium was immersed. The solution became slightly turbid and turned yellow-brown during the 10 ma discharge. A new lithium anode was inserted for the 100 ma discharge because the first anode had dissolved completely. The counter electrode gassed heavily at 100 ma and the anode potential fluctuated considerably. A strong ammonia odor was detected.
					-3.02	1	-3.03	-3.02	
					-3.00	10	-2.96	-2.90	
					-2.99	100	-2.00	-1.80	

** NH_4PF_6 - Ammonium hexafluorophosphate (1 molal)

TABLE II. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES IN PRETREATED ELECTROLYTES (Continued)

I. Anode-Electrolyte Systems Capable of Sustaining 100 ma/cm^2. (Continued)
A. 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: Initial	Final	Further Observations and Remarks
2.	NDA	*	Ag/AgCl	Ag/AgCl	-2.90	-2.91	0.1	-2.90	-2.91	Counter electrode gassed at 100 ma. No change in the appearance of the lithium, the reference electrode, and the electrolyte.
						-2.91	1	-2.87	-2.88	
						-2.90	10	-2.72	-2.68	
						-2.88	100	-1.60	-1.64	

* 
$O\langle{}^{CH_2CH_2}_{CH_2CH_2}\rangle NH_2PF_6$ - Morpholinium hexafluorophosphate (1 molal). The solution was pretreated with lithium metal until gas evolution ceased. This reaction resulted in the formation of a gelatinous yellow solid in addition to the evolution of an appreciable quantity of gas. After filtering the solution, it had a specific conductance of 1.81 x 10^{-2} ohm^{-1} cm^{-1} as compared to 2.74 x 10^{-2} ohm^{-1} cm^{-1} for the untreated solution.

The results for the same system (not pretreated with lithium) are listed below for comparison. The conductance of the solution in this case was 2.80 x 10^{-2}.

	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: Initial	Final	Further Observations and Remarks
	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	

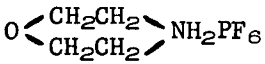
** 
$O\langle{}^{CH_2CH_2}_{CH_2CH_2}\rangle NH_2PF_6$ - Morpholinium hexafluorophosphate (1 molal)

TABLE II. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES IN PRETREATED ELECTROLYTES (Continued)

II. Anode-Electrolyte Systems Capable of Sustaining 10 ma/cm^2.

A. 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: Initial	Voltage of Anode vs. Reference Electrode at Various Current Densities: Final	Further Observations and Remarks
1.	NDA	*	Ag/AgCl	Ag/AgCl	-2.90	-2.90	0.1	-2.88	-2.88	During the 100 ma discharge, both the lithium and the counter electrodes gassed lightly. A gray-white film formed at the lithium surface and the counter electrode became blackened. No change in the appearance of the reference electrode or the electrolyte.
						-2.90	1	-2.81	-2.83	
						-2.90	10	-2.48	-2.64	
						-2.83	100	-1.20	-1.00	

* (n-C_3H_7)$_3$NHAsF$_6$ - Tri-n-propylammonium hexafluoroarsenate (1 molal). The solution was pretreated with lithium until gas evolution ceased. In addition to evolving an appreciable amount of gas, this reaction resulted in the formation of a second liquid which was immiscible with the rest of the solution and amounted to ca. 10% of the total volume. Solid was also formed. After filtering the solution, it had a specific conductance of 1.85 x 10^{-2} ohm^{-1} cm^{-1} (larger liquid phase) as compared to 2.36 x 10^{-2} ohm^{-1} cm^{-1} for the untreated solution. The combination of the two immiscible liquids was used for the screening test, but the electrodes were not in contact with the smaller (upper) phase.

The results for the same system (not pretreated with lithium) are listed below for comparison. Conductance of the electrolyte was 2.35 x 10^{-2} ohm^{-1} cm^{-1}.

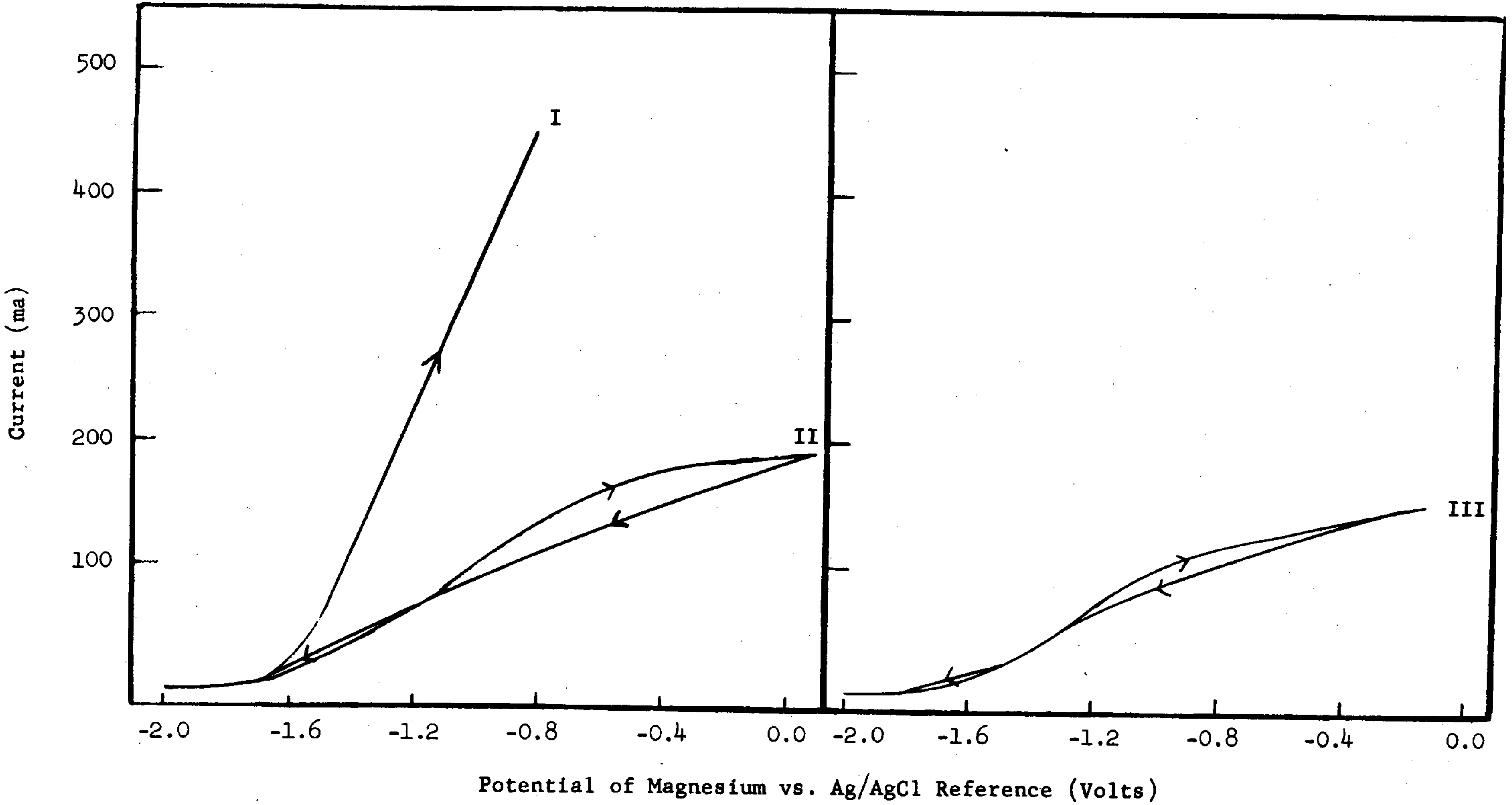
	Solvent	Solute	Reference Electrode	Counter Electrode	Initial Open Circuit Voltage of Anode vs. Reference Electrode	Open Circuit Voltage After Discharge at Various Current Densities	Current Density (ma/cm^2)	Initial	Final	Further Observations and Remarks
	NDA	**	Ag/AgCl	Ag/AgCl	-3.40	-3.00	0.1	-3.30	-2.92	Vigorous gassing at lithium electrode at all times. No discharge was carried out at 1 ma, and at 10 ma the discharge was interrupted after two minutes. At this point, much of the lithium had been consumed. Discharge was then carried out at 100 ma for 20 seconds.
						----	1	----	----	
						-3.00	10	-2.74	----	
						----	100	-1.44	----	

** (n-C_3H_7)$_3$NHAsF$_6$ - Tri-n-propylammonium hexafluoroarsenate (1 molal)

FIGURE 1. LINEARLY VARYING POTENTIAL SCANS OF MAGNESIUM ANODE IN ALUMINUM CHLORIDE-ACETONITRILE ELECTROLYTE

Current versus Potential of Magnesium Anode.

I. 30 mv/sec. sweep rate*
II. 100 mv/sec. sweep rate**
III. 30 mv/sec. sweep rate***



* Total Mg surface = 26.5 mm^2.

** Mg surface changed to 7.6 mm^2 at the start of this series of scans. Scans at 30 and 330 mv/sec. gave similar curves.

*** After this run, the surface of the Mg had decreased to 2.9 mm^2.

FIGURE 2. COULOMBIC EFFICIENCY DETERMINATION FOR MAGNESIUM IN ALUMINUM CHLORIDE-ACETONITRILE ELECTROLYTE

Potential of Magnesium Anode versus Ampere-Hours of Discharge.

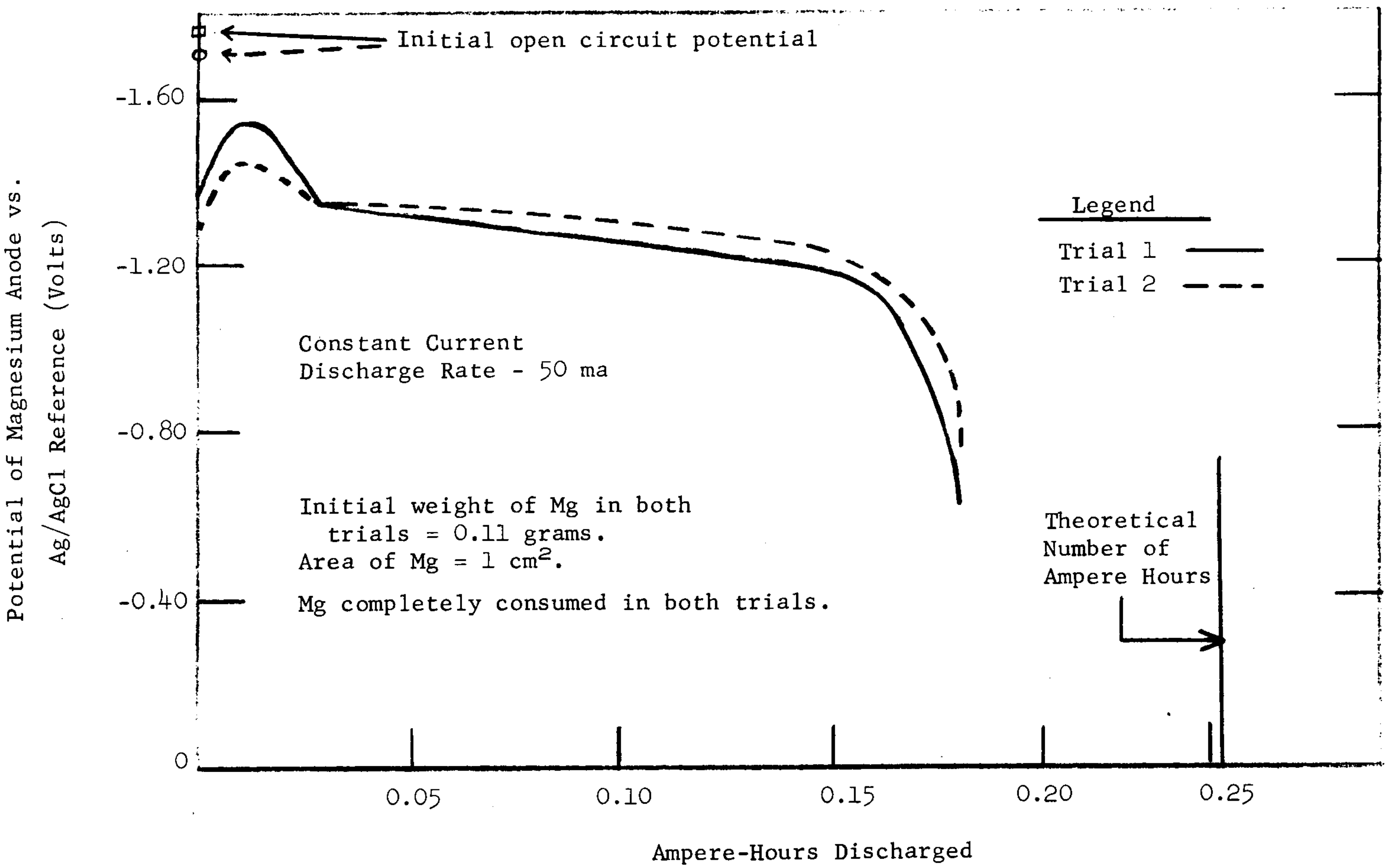


TABLE III. ELECTROCHEMICAL HALF-CELL SCREENING OF CATHODES

TABLE III. ELECTROCHEMICAL HALF-CELL SCREENING OF CATHODES

A. Cupric Fluoride Cathodes Prepared by Subliming Phenol from Electrode Pellets, Followed by Partial Reduction with Hydrogen Gas.

Experimental Details:

Cathode matrix - silver expanded metal, 1 cm^2 area (circular).
Composition of cathode mix - 79.7% CuF_2, 16.1% phenol, 4.2% Solka-Floc.
Weight of cathode mix - 0.5 grams. Thickness of pellet - ca. 1/8" (4,000 lb./cm^2 formation pressure).
Sublimation of phenol - sublimed at ca. 40°C, 0.5 mm Hg until constant weight attained (ca. 16 hours). Approximate percentage of initial phenol remaining after sublimation was 7%.
Temperature of hydrogen gas for partial reduction of CuF_2 pellets - 130°C.
Electrolyte - 1 molal $(C_6H_5)(CH_3)_3NPF_6$*-dimethylformamide.
Counter electrode - lithium. Reference electrode - Ag/AgCl.
Duration of discharge at each current density - 5 minutes (unless noted otherwise).

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) 0	0.1	1	Observations and Remarks
1	1.3 hours	Initial	+0.11	+0.07	$<$ -1.0 (5 seconds)	Although the fabrication procedure was the same in both cases, visual inspection indicated that the two electrodes had been reduced to different degrees with hydrogen. During discharge of these electrodes, the electrolyte color changed from green to yellow.
		Final	-0.02	-0.09	----	
2	1.3 hours	Initial	+0.44	+0.08	$<$ -1.0 (3 seconds)	
		Final	-0.47	-0.10	----	

---

* N-Phenyl N,N,N-trimethylammonium hexafluorophosphate

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

B. Cobalt Trifluoride Cathodes.

Experimental Details:

Cathode matrix - nickel expanded metal.
Matrix size - 1 cm^2 (circular).
Composition of cathode mix - 97% CoF_3, 3% Solka-Floc.
Thickness of dry-pressed pellets - ca. 1/8".
Temperature of hydrogen gas for partial reduction of CoF_3 pellets to provide conductive substrate - 130°C.*
Electrolyte - 1 molal $(C_6H_5)(CH_3)_3NPF_6$**-acetonitrile.
Counter electrode - magnesium. Reference electrode - Ag/AgCl.
Duration of discharge at each current density - 5 minutes (unless noted otherwise).

Cathode Number	Time of Treatment With H_2 Gas	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities (ma/cm^2)			Observations and Remarks
			0	0.1	1	
1	0.5 hours	Initial	-0.14	-0.24	-1.00	In each case the electrolyte took on a green coloration around the cathode after discharge for approximately one minute. Gas evolution was observed at each of the cathodes during discharge at 1 ma/cm^2. Discharges at this current density were discontinued at the time that gas evolution was noted.
		Final	-0.11	-0.42	----	
2	1.0 hours	Initial	-0.16	-0.27	-0.75	
		Final	-0.11	-0.37	-0.92 (1 minute)	
3	1.5 hours	Initial	-0.12	-0.25	-0.84	
		Final	-0.08	-0.40	-0.94 (30 seconds)	

---

* Qualitative X-ray analyses performed at a later date for CoF_3 cathodes prepared in the same manner as those above indicated that treatment with hydrogen gas had not resulted in the formation of metallic cobalt.

** N-Phenyl N,N,N-trimethylammonium hexafluorophosphate

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

C. Lead Dioxide Cathodes.

Experimental Details:

Cathode matrix - silver expanded metal, 1 cm^2 area (circular).
Composition of cathode mix - 95% β-PbO_2, 5% Solka-Floc.
Dry-press formation pressure - 3,000 lb./cm^2.
Thickness of dry-pressed pellet - ca. 1/8".
Counter electrode-lithium. Reference electrode - Ag/AgCl.
Electrolyte - 1 molal $(C_6H_5)(CH_3)_3NPF_6$*-dimethylformamide.
Length of discharge at each current density - 5 minutes (unless noted otherwise).

Cathode Number	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities (ma/cm^2) 0	0.1	1	5	10	Observations and Remarks
1	Initial	+0.44	+0.44	+0.40	-0.20	-0.80	The solution color changed from green to dark blue upon insertion of the cathode in both of the tests. During discharge, the solution color changed to gray and a large amount of a yellow-brown solid formed in the solution.
	Final	-0.47	+0.40	-0.02	-0.86	-1.00 (1 minute)	
2	Initial	+0.43	+0.45	+0.38	-0.15	-0.86	
	Final	-0.40	+0.38	-0.05	-0.91	-1.00 (1 minute)	

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* N-Phenyl N,N,N-trimethylammonium hexafluorophosphate

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

D. Manganese Dioxide Cathodes.

Experimental Details:

Cathode matrix - silver expanded metal, 1 cm^2 area (circular).
Composition of cathode mix - 95% MnO_2, 5% Solka-Floc.
Dry-press formation pressure - 6,000 lb./cm^2.*
Thickness of dry-pressed pellet - ca. 1/8".
Counter electrode - lithium. Reference electrode - Ag/AgCl.
Electrolyte - 1 molal $(C_6H_5)(CH_3)_3NPF_6$**-dimethylformamide.
Length of discharge at each current density - 5 minutes.

Cathode Number	Time of Measurement	Voltage of Cathode vs. Reference Electrode at Various Current Densities (ma/cm^2)				Observations and Remarks
		0	0.1	1	5	
1	Initial	+0.44	+0.44	+0.36	-0.05	The solution became turbid and a brown solid settled out during discharge in both of the tests. No change in the appearance of the cathode was observed in either test.
	Final	-0.17	+0.41	+0.11	<-1.0	
2	Initial	+0.44	+0.43	+0.39	-0.31	
	Final	-0.15	+0.42	+0.19	-1.09	

---

* An electrode fabricated at 4,000 lb./cm^2 did not have sufficient mechanical integrity; the cathode material did not adhere well to the matrix.

** N-Phenyl N,N,N-trimethylammonium hexafluorophosphate

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS

NOTE: In some cases the voltage of the anode versus the reference electrode does not appear in the tables for all points of a discharge. In these instances, the cells were being discharged overnight and only the cell voltage was recorded.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS

1. Calcium-Morpholinium Hexafluorophosphate.

Anode - calcium; area, 1 cm^2; weight, ca. 0.22 grams.

Cathode - electrode of silver foil; area, 17 cm^2; weight, 0.63 grams. Active cathode material, morpholinium hexafluorophosphate (dissolved), 6 molal in acetonitrile; weight of solution, 28 grams.

Electrolyte - 6 molal morpholinium hexafluorophosphate in acetonitrile (i.e. the cathode solution was utilized as the electrolyte).

Weight of glass container - 31 grams.

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
--	2.20	---	0	-2.20
0	1.10	100	11	-2.00
17	0.98	100	9.8	-1.97
38	1.01	100	10.1	-1.99
54	1.07	100	10.7	-2.06
72	1.11	100	11.1	-2.11
80	1.12	100	11.2	-2.11
81	1.90	---	0	-2.25
85	2.20	---	0	-2.28
86	1.12	100	11.2	-2.09
91	1.05	100	10.5	-2.09
92	1.90	---	0	-2.25
93	0.95	50	19	-2.00
94	0.90	50	18	-2.00
107	0.82	50	16.4	-2.03
133	1.77	---	0	-2.28
134	0.53	10	53	-1.82
160	0.50	10	50	-1.80
180	0.13	2	65	-1.58
276	0.82	50	16.4	-2.02
350	0.73	50	14.6	-1.90
428	0.62	50	12.4	----

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

2. Calcium-Morpholinium Hexafluorophosphate.

Anode - calcium; area, 1 cm^2; weight, ca. 0.25 grams.

Cathode - electrode of platinized platinum; area, 1 cm^2; weight, 1.12 grams. Active cathode material, morpholinium hexafluorophosphate (dissolved), 6 molal in acetonitrile; weight of solution, 25.1 grams.

Electrolyte - 6 molal morpholinium hexafluorophosphate in acetonitrile (i.e. the cathode solution was utilized as the electrolyte).

Weight of glass container - 30.8 grams.

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
0	2.50	---	0	-1.85
1.5	1.15	100	11.5	-1.65
2.1	1.05	100	10.5	-1.75
3	1.10	100	11.0	-1.77
18	1.20	100	12.0	-1.92
37.5	1.19	100	11.9	-1.94
53	1.20	100	12.0	-1.96
55.5	1.73	---	0	-2.10
57	1.05	50	21.0	-1.88
74.5	1.04	50	20.8	-1.87
88.7	1.02	50	20.4	-1.87
93.5	1.70	---	0	-2.15
94	0.60	10	60	-1.63
95.2	0.60	10	60	-1.70
108	0.18	2	90	-1.55
137	0.10	1	100	-1.50
238	0.04	1	40	-1.00
239	1.45	---	0	-2.14

TABLE IV . DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

3. Calcium-Morpholinium Hexafluorophosphate.

Anode - calcium; area, 1 cm^2; weight, ca. 0.24 grams.

Cathode - electrode of carbon; area, 4.8 cm^2; weight, 1.31 grams. Active cathode material, morpholinium hexafluorophosphate (dissolved), 6 molal in acetonitrile; weight of solution, 26.2 grams.

Electrolyte - 6 molal morpholinium hexafluorophosphate in acetonitrile (i.e. the cathode solution was utilized as the electrolyte).

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
0	2.20	---	0	-1.85
0.6	0.40	100	4.0	-1.80
4.6	0.42	100	4.2	-1.84
23.4	0.16	10	16.0	-1.70
35.7	0.24	10	24.0	-1.80
49.5	0.30	10	30.0	-1.85
54	0.31	10	31.0	-1.88
955	>0	10	>0	-0.98
970	1.38	---	0	-2.19

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

4. Calcium-Morpholinium Hexafluorophosphate.

Anode - calcium; area, 1 cm^2; weight, ca. 0.23 grams.

Cathode - electrode of sintered nickel; area, 1 cm^2. Active cathode material, morpholinium hexafluorophosphate (dissolved), 2.5 molal in acetonitrile.

Electrolyte - 2.5 molal morpholinium hexafluorophosphate in acetonitrile (i.e. the cathode solution was utilized as the electrolyte).

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
0	1.90	---	0	-2.10
0.3	0.88	100	8.8	-1.79
1.3	0.84	100	8.4	-1.92
4.1	0.82	100	8.2	-2.00
5.4	0.89	100	8.9	-2.08
8.2	0.90	100	9.0	-2.12
10.6	0.90	100	9.0	-2.12
17.6	0.90	100	9.0	-2.10
21.3	0.90	100	9.0	-2.10
33	0.91	100	9.1	-2.10
124	0.97	100	9.7	-2.18
156	1.00	100	10.0	-2.21
184	1.01	100	10.1	-2.22
218	1.00	100	10.0	-2.10
285	0.98	100	9.8	-2.05
340	0.94	100	9.4	----
400	0.94	100	9.4	----
520	0.92	100	9.2	----
640	0.88	100	8.8	----
760	0.80	100	8.0	----
820	0.70	100	7.0	----
940	0.45	100	4.5	----
1060	0.28	100	2.8	----
1200	0.20	100	2.0	----

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

4. Calcium-Morpholinium Hexafluorophosphate. (Continued)

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
1320	0.12	100	1.2	----
1380	0.11	100	1.1	----
1381	1.95	---	0	-2.50

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

5. Calcium-Morpholinium Hexafluorophosphate.

Anode - calcium; area, 1 cm^2; weight, ca. 0.24 grams.

Cathode - electrode of sintered nickel; area, 1 cm^2. Active cathode material, morpholinium hexafluorophosphate (dissolved), 2.5 molal in acetonitrile, 22 ml.

Electrolyte - 2.5 molal morpholinium hexafluorphosphate in acetonitrile (i.e. the cathode solution was utilized as the electrolyte).

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
0	1.51	---	0	-1.72
1	0.49	50	9.8	-1.71
8	0.58	50	11.6	-1.87
27	0.63	50	12.6	-1.91
65	0.69	50	13.8	-1.95
120	0.72	50	14.4	----
300	0.71	50	14.2	----
420	0.67	50	13.4	----
540	0.64	50	12.8	----
600	0.62	50	12.4	----
660	0.56	50	11.2	----
720	0.42	50	8.4	----
780	0.36	50	7.2	----
840	0.30	50	6.0	----
888	0.30	50	6.0	----

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

6. Calcium-Morpholinium Hexafluorophosphate.

Anode - calcium; area, 0.5 cm^2; weight, ca. 0.15 grams.

Cathode - electrode of sintered nickel; area, 1 cm^2. Active cathode material, morpholinium hexafluorophosphate (dissolved), 2.5 molal in acetonitrile; volume, 25 ml.

Electrolyte - 2.5 molal morpholinium hexafluorophosphate in acetonitrile (i.e. the cathode solution was utilized as the electrolyte).

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
0	1.70	---	0	-1.78
0.8	0.70	100	7.0	-1.65
9.2	0.70	100	7.0	-1.80
15.4	0.75	100	7.5	-1.89
48.4	0.82	100	8.2	-1.96
77.5	0.90	100	9.0	-2.03
91.5	0.91	100	9.1	-2.04
124	0.92	100	9.2	-2.04
180	0.94	100	9.4	----
300	0.92	100	9.2	----
420	0.88	100	8.8	----
540	0.84	100	8.4	----
660	0.80	100	8.0	----
760	0.78	100	7.8	----
765	0.68	100	6.8	----
840	0.62	100	6.2	----
960	0.56	100	5.6	----
1044	0.30	100	3.0	----

Virtually all calcium consumed.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

7. Calcium-Morpholinium Hexafluorophosphate.

Anode - calcium; area, 1 cm^2; weight, ca. 0.23 grams.

Cathode - electrode of platinized carbon; area, 1 cm^2. Active cathode material, morpholinium hexafluorophosphate (dissolved), 6 molal in acetonitrile.

Electrolyte - 6 molal morpholinium hexafluorophosphate in acetonitrile (i.e. the cathode solution was utilized as the electrolyte).

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
0	1.83	---	0	-1.34
1.7	1.45	100	14.5	-1.82
7.2	1.31	100	13.1	-1.87
7.4	1.81	---	0	-2.14
7.7	1.25	50	25.0	-1.75
11.7	1.09	50	21.8	-1.74
13.7	1.09	50	21.8	-1.76
13.9	1.75	---	0	-2.14
14.4	0.55	10	55.0	-1.43
15	0.33	10	33.0	-1.09
16.7	0.45	10	45.0	-1.31
19.7	0.50	10	50.0	-1.38
20.1	1.80	---	0	-2.22
20.3	1.45	100	14.5	-2.00
20.8	1.40	100	14.0	-2.00
25.8	1.37	100	13.7	-1.98
36	1.27	100	12.7	-1.94
65	1.20	100	12.0	----
305	0.80	100	8.0	----
545	0.70	100	7.0	----
665	0.60	100	6.0	----
965	0.40	100	4.0	----
973	2.20	---	0	-2.19
975	0.20	100	2.0	----
1025	0.20	100	2.0	----

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

8. Calcium-Morpholinium Hexafluorophosphate + Water.

Anode - calcium; area, 3.8 cm^2; weight, 0.87 grams.

Cathode - electrode of platinized platinum; area, 1 cm^2; weight, 1.02 grams. Active cathode material, morpholinium hexafluorophosphate (dissolved), 6 molal in acetonitrile plus 2.5 ml. of water.

Electrolyte - 6 molal morpholinium hexafluorophosphate in acetonitrile plus 2.5 ml. of water (i.e. the cathode solution was utilized as the electrolyte).

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)
0	2.70	---	0
0.2	1.58	100	15.8
1.6	1.53	100	15.3
6.9	1.40	100	14.0
11.7	1.31	100	13.1
19.7	1.13	100	11.3
28	0.26	100	2.6
28.5	----	100	----
29.6	1.48	---	0
30	0.45	200	2.2
38	Silver wire reference electrode inserted; on open circuit, cathode vs. Ag = -2.10 volts, calcium vs. Ag = -2.40 volts.		
41	1.42	---	0

Vigorous gas evolution was observed when the calcium electrode was placed in the electrolyte.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

9. Calcium-Morpholinium Hexafluorophosphate + Sulfuric Acid.

Anode - calcium; area, 3.7 cm^2; weight, 0.83 grams.

Cathode - electrode of platinized platinum; area, 1 cm^2; weight, 1.02 grams. Active cathode material, morpholinium hexafluorophosphate (dissolved), 6 molal in acetonitrile, plus 2.5 ml. of 4.4 molar H_2SO_4.

Electrolyte - the cathode solution was utilized as the electrolyte.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)
0	2.80	---	0
0.3	1.90	100	19.0
1.7	1.73	100	17.3
7.1	1.38	100	13.8
12	1.16	100	11.6
20	0.95	100	9.5
28	0.81	100	8.1
28.7	----	---	0
29.7	1.76	---	0
30.7	1.79	---	0
31	1.18	200	5.9
50	0.94	200	4.7
64	0.84	200	4.2
82	0.76	200	3.8
90	1.48	---	0
91	0.88	200	4.4
99	0.79	200	4.0
120	0.67	200	3.4
121	1.40	---	0

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

10. Calcium-Sulfuric Acid.

Anode - calcium; area, 1 cm^2; weight, ca. 0.24 grams.

Cathode - H_2SO_4 (ca. 4 molar) contained in a porous carbon vessel which was platinized on the inside. This vessel also served as the current collector.

Electrolyte - 2.5 molal morpholinium hexafluorophosphate-acetonitrile.

Cell structure - The anode and the carbon vessel were positioned in a beaker containing the electrolyte.

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
0	2.20	---	0	-1.94
3	2.20	---	0	-2.07
4	1.49	100	14.9	-1.60
9.3	1.45	100	14.5	-1.92
25	1.23	50	24.6	-1.85
37.3	0.78	50	15.6	-1.72
58	0.76	50	15.2	-1.72
89	0.62	50	12.4	-1.65
143	0.57	50	11.4	-1.63
158	0.52	50	10.4	-1.60
180	0.43	50	8.6	-1.50
195	0.37	50	7.4	-1.41
211	0.22	50	4.4	-1.20
222	0.18	50	3.6	-1.22
223	1.54	---	0	-2.22

All calcium was consumed.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

11. Calcium-Sulfuric Acid.

Anode - calcium; area, 1 cm^2; weight, ca. 0.24 grams.

Cathode - H_2SO_4 (ca. 4 molar) contained in a porous carbon vessel which was platinized on the outside. This vessel also served as the current collector.

Electrolyte - 2.5 molal morpholinium hexafluorophosphate-acetonitrile.

Cell structure - The anode and the carbon vessel were placed in a beaker containing the electrolyte.

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Calcium vs. Reference (Volts)
0	2.47	---	0	-1.92
0.9	1.68	100	16.8	-1.67
2.1	1.65	100	16.5	-1.83
4.9	1.53	100	15.3	-1.80
16	1.54	100	15.4	-1.93
19.6	1.82	---	0	-2.13
20	1.30	50	26.0	-1.80
24	1.34	50	26.8	-1.90
28	1.40	50	28.0	-1.95
32	1.84	---	0	-2.13
32.5	0.80	10	80	-1.55
34	0.85	10	85	-1.67
40	0.80	10	80	-1.68
40.8	1.78	---	0	-2.10
41	0.29	2	145	-1.33
42	0.32	2	160	-1.40
59	0.30	2	150	-1.50
82	0.24	2	120	-1.55
97	0.22	2	110	-1.48
98	0.18	2	90	-1.52
99	1.70	---	0	-2.20

Calcium virtually all consumed.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

12. Lithium-Morpholinium Hexafluorophosphate.

Anode - lithium; area, 1 cm^2; weight, ca. 0.075 grams.

Cathode - electrode of sintered nickel; area, 1 cm^2. Active cathode material, morpholinium hexafluorophosphate (dissolved), 1 molal in EC-PC*, volume, 15 ml.

Electrolyte - the cathode solution was utilized as the electrolyte.

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Volts)
0	2.99	---	0	-3.26
4	1.70	100	17.0	-2.95
5	1.38	100	13.8	-2.82
29	1.34	100	13.4	-2.78
38	1.34	100	13.4	-2.80
60	1.20	100	12.0	----
120	1.10	100	11.0	----
180	1.00	100	10.0	----
300	0.90	100	9.0	----
420	0.80	100	8.0	----
660	0.70	100	7.0	----
900	0.60	100	6.0	----
1050	0.70	100	7.0	----
1200	0.70	100	7.0	----
1201	1.85	---	0	-2.97

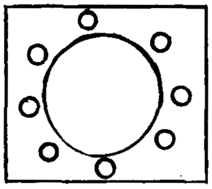
---

* 80% Ethylene Carbonate - 20% Propylene Carbonate.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

13. Lithium-Sulfuric Acid.

Cell structure - Holes were drilled in a porous carbon block to obtain the configuration illustrated below:


(Top View)

The large central hole was 1" in diameter and 3.5 cm. deep. Eight 1/8" diameter holes, 3.9 cm. deep were situated symmetrically about the large hole at a distance of 0.1 cm from the periphery. The surface of the holes was lightly platinized. The weight of this structure was 144 grams.

Anode - lithium rod; dimensions, 1/2" diameter, 3 cm. length. This was positioned in the central hole which contained the electrolyte, 1 molal $MgCl_2$-N-nitrosodimethylamine.

Cathode - H_2SO_4 (ca. 4 molar) was placed in each of the 1/8" diameter holes. The carbon structure served as the current collector for the cathode.

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Volts)
0	3.32	---	0	-2.70
0.2	2.20	100	22.0	-1.98
1.6	2.07	100	20.7	-2.08
3.8	1.96	100	19.6	-2.10
6.5	1.85	100	18.5	-2.18
8.7	1.80	100	18.0	-2.23
11.5	1.74	100	17.4	-2.29
16	1.74	100	17.4	-2.33
21	1.68	100	16.8	-2.38
21.2	2.84	---	0	-2.80
21.7	2.90	---	0	-2.80
22	1.40	50	28.0	-2.10
22.4	1.33	50	26.6	-2.18
24	1.28	50	25.6	-2.23
24.6	2.80	---	0	-2.80
25	2.88	---	0	-2.80
25.5	0.53	10	53.0	-1.58
25.8	0.50	10	50.0	-1.68
26.6	0.45	10	45.0	-1.67

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

13. Lithium-Sulfuric Acid. (Continued)

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Volts)
30.4	0.34	10	34.0	-1.65
30.8	2.80	---	0	-2.78
32.5	3.08	---	0	-2.80
33	2.68	500	5.4	-2.68
35	2.62	500	5.3	-2.67
40	2.58	500	5.2	-2.68
40.5	2.84	---	0	-2.80
41.5	3.10	---	0	-2.80
42	2.40	250	9.6	-2.54
43.5	2.23	250	8.9	-2.58
50	2.47	250	9.9	-2.62
52	2.54	250	10.2	-2.63
67	2.40	250	9.6	-2.68
105	2.28	250	9.1	----
165	2.12	250	8.5	----
225	1.92	250	7.7	----
285	1.72	250	6.9	----
345	1.52	250	6.1	----
405	1.36	250	5.4	----
465	1.20	250	4.8	----
525	1.04	250	4.2	----
585	0.90	250	3.6	----
650	0.50	250	2.0	----
651	0.20	250	0.8	abrupt drop in cell voltage
770	0.14	250	0.6	----
977	0.12	250	0.5	-1.60
979	2.20	---	0	----

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

14. Lithium-Sulfuric Acid.

Cell design - A porous carbon vessel containing 4.4 molar H_2SO_4 as the active cathode material was positioned in a polyethylene container between two lithium electrodes which were immersed in a morpholinium hexafluorophosphate-N-nitrosodimethylamine electrolyte. The carbon vessel served as the cathode current collector.

Description of cell components -

A. Carbon vessel:

	External	Internal
length	5.64 cm.	5.0 cm.
width	3.64 cm.	3.0 cm.
depth	3.52 cm.	3.2 cm.

i.e. the walls of the vessel were approximately 0.32 cm. in thickness. Weight of vessel = 44.5 grams.

B. Polyethylene cell container:

length = 12.6 cm.
width = 7.6 cm.
depth = 4.5 cm.

Weight of container plus cover = 57 grams.

C. Anodes:

Lithium was cast in the form of oblong blocks around a length of nickel screen 1 cm. in width. The dimensions of the blocks were:

length = 5 cm.
width = 2.6 cm.
depth = 2.6 cm.

Two such blocks were employed in the cell. The weights of lithium in these blocks were 19.5 and 20 grams.

The weight of each nickel screen was 1.5 grams.

D. 4.4 molar H_2SO_4 - 45 ml; weight, 57 grams.

E. 1 molal morpholinium hexafluorophosphate - 100 ml.

F. Total weight of cell = approximately 300 grams.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

15. Lithium-Sulfuric Acid.

Cell components were the same as for No. 14, except for the following changes:

A. The sides of the carbon vessel were made thinner so that they were 0.15 cm. thick. Weight of vessel = 34.2 grams.

B. $(C_6H_5)(CH_3)_4NPF_6$-N-nitrosodimethylamine (1 molal) was used as the electrolyte instead of morpholinium hexafluorophosphate-N-nitrosodimethylamine.

C. The lithium electrodes were the same ones that had been used in cell No. 14. (Only a small fraction of the lithium capacity had been used.) Each lithium electrode was scraped to remove reaction products. The weight of each lithium electrode (including nickel screen) was approximately 22 grams.

D. Total weight of cell = 296 grams.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Ag/AgCl Reference (Volts)
0	3.35	---	0	-2.85
2.5	1.80	10	180	-2.80
3.9	1.30	5	260	----
4.7	1.25	5	250	----
7.5	1.20	5	240	----
8.4	1.65	10	165	-2.83
12.4	1.60	10	160	----
18	1.57	10	157	-2.88
19	1.20	5	240	----
19.3	1.15	5	230	----
---	1.50	10	150	----
22	1.85	20	92.2	----
22.6	1.88	20	94.0	-2.90
25	1.90	20	95.0	-2.91
35	1.89	20	94.5	-2.91
50	1.85	20	92.5	-2.90
100	1.78	20	89.0	-2.88
127	1.55	20	77.5	-2.88
128	----	---	0	-2.93
138	2.70	---	0	----
139	2.00	20	100	-2.85

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

15. Lithium-Sulfuric Acid. (Continued)

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Ag/AgCl Reference (Volts)
140	2.20	40	55.0	----
145	2.23	40	55.8	-2.87
157	2.25	40	56.3	-2.87
172	2.25	40	56.3	-2.85
187	2.27	40	56.7	-2.83
201	2.30	40	57.5	-2.89
207	2.08	20	104	-2.86
219	2.04	20	102	-2.85
230	2.19	30	73.0	-2.84
240	2.10	30	70.0	----
360	2.00	30	66.6	----
480	1.90	30	63.2	----
600	1.80	30	60.0	----
720	1.80	30	60.0	----
840	1.70	30	56.6	----
1020	1.60	30	53.2	----
1080	1.60	30	53.2	----
1100	----	30	----	-2.72
1125	1.60	30	53.2	----
1126	2.20	---	0	-2.78

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

16. Lithium-Sulfuric Acid.

Cell components were the same as for No. 15. The same lithium anodes were used after scraping to remove products formed during the discharge of cell No. 15.

The N-nitrosodimethylamine electrolyte and the sulfuric acid were replenished.

Hexane was poured over the NDA electrolyte (immiscible) to prevent evaporation.

Total weight of cell = 310 grams.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Volts)
0	2.80	---	0	-2.82
1	2.30	30	76.9	-2.67
2	2.16	30	72.0	-2.68
22	2.12	30	70.7	-2.69
35	2.12	30	70.7	-2.70
53	2.13	30	71.0	-2.70
72	2.13	30	71.0	-2.71
142	2.10	30	70.0	-2.73
165	2.06	30	68.8	-2.72
245	1.98	30	66.0	-2.70
295	1.94	30	64.7	-2.70
307	1.03	5	206	-2.40
312	1.85	30	61.7	-2.63
352	1.88	30	62.7	-2.68
432	1.82	30	60.6	-2.65
447	1.81	30	60.2	-2.65
459	1.81	30	60.2	-2.63
480	1.79	30	59.6	----
600	1.65	30	55.0	----
720	1.60	30	53.3	----
960	1.58	30	52.6	----
1080	1.50	30	50.0	----
1320	1.40	30	46.5	----
1440	1.30	30	43.4	----

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

16. Lithium-Sulfuric Acid. (Continued)

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Volts)
1600	1.20	30	40.0	----
1840	1.00	30	33.3	----
2080	1.00	30	33.3	----
2220	0.90	30	30.0	----
2460	0.80	30	26.6	----
2940	0.80	30	26.6	----
3060	0.80	30	26.6	----
3300	0.70	30	23.3	----
3420	0.60	30	20.0	----
4380	0.60	30	20.0	----
5100	0.50	30	16.6	----
5760	0.50	30	16.6	----
5761	2.35	---	0	-2.98

At this time, the discharge was interrupted. A large amount of white solid had formed on the lithium electrode. This was removed by scraping and the discharge continued.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Volts)
5768	2.29	---	0	-3.00
5769	1.65	30	55.0	-2.90
5770	1.55	30	51.7	-2.89
5780	1.28	30	42.6	-2.60
5793	1.10	30	36.6	-2.30
5796	0.10	1	100	-1.81
5801	1.00	30	33.3	----
5877	0.90	30	30.0	----
6168	0.75	30	25.0	----
6305	0.75	30	25.0	----
6665	0.70	30	23.3	----
6905	0.62	30	20.6	----
7115	0.60	30	20.0	----
7135	0.60	30	20.0	----
7137	1.38	---	0	-2.68

TABLE IV . DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

17. Lithium-Sulfuric Acid.

Cell structure - A polyethylene container was partitioned into three compartments by use of two carbon dividers, which also served as current collectors for the cathode.

The middle compartment contained 1 molal $(C_6H_5)(CH_3)_3NPF_6$-N-nitrosodimethylamine electrolyte, along with a lithium anode. The two outer compartments contained 4.4 molar sulfuric acid as the active cathode material.

Description of Cell Components -

A. Polyethylene container:

Dimensions: Base = 7.2 x 7.2 cm.
Top = 8.2 x 8.2 cm.
Depth = 6 cm.

B. Carbon dividers: (Helwig Carbon - 34151)

Dimensions: Length = 7.65 cm.
Width = 5.05 cm.
Thickness = 0.3 cm.

Pre-treatment: The plates were electrolyzed in sulfuric acid prior to use for the purpose of increasing the porosity of the carbon. Following this, the plates were platinized.

The weights of the two plates were 19.1 and 18.7 grams. These dividers were bonded to the container with epoxy glue at a distance of 1 cm from each end at the base.

C. Lithium electrode - the same two lithium blocks which had been utilized in cells No. 14, 15 and 16 were used. (Only a small fraction of the lithium had been consumed thus far.) The blocks were placed one on top of the other in the central compartment of the cell container.

Total weight of lithium, 38.46 grams.

Total weight of nickel screen, 3.00 grams.

D. Cathode solution - 4.4 molar H_2SO_4; total of 63 ml. divided between two compartments.

E. Electrolyte - 100 ml. of 1 molal $(C_6H_5)(CH_3)_3NPF_6$-N-nitrosodimethylamine.

F. Total weight of assembled cell = 304.4 grams.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

17. Lithium-Sulfuric Acid. (Continued)

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Ag/AgCl)
0	3.10	---	0	----
1.7	3.07	---	0	-2.78
2.6	2.95	100	29.5	-2.72
5	2.84	100	28.4	-2.70
8.4	2.78	100	27.8	-2.71
11.5	2.68	100	26.8	-2.68
11.8	2.60	50	52.0	-2.63
15	2.50	50	50.0	-2.58
18	2.50	50	50.0	-2.61
18.8	2.52	50	50.3	-2.65
21.6	2.50	50	50.0	-2.65
21.8	2.44	30	81.4	-2.62
24.8	2.39	30	79.7	-2.57
28	2.33	30	77.7	-2.52
31.8	2.25	30	75.0	-2.45
32	2.19	20	109	-2.40
35	2.10	20	105	-2.34
38	2.01	20	100	-2.26
38.4	1.82	10	182	-2.10
41.7	1.65	10	165	-1.95
41.9	1.35	5	170	-1.70
44.8	1.20	5	240	-1.82
45	0.80	2	400	-1.17
45.5	0.73	2	365	-1.11
48.4	0.62	2	310	-1.00
49.8	0.62	2	310	-1.00
50	0.37	1	370	-0.72
53	0.34	1	340	-0.68

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

17. Lithium-Sulfuric Acid. (Continued)

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Ag/AgCl)
56.5	1.93	---	0	-2.10

At this point, the discharge was discontinued. H_2SO_4 had leaked around and through the dividers to an appreciable extent, causing corrosion of the lithium. The lithium surface was scraped clean, the electrolytes were replenished, and discharge resumed.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Ag/AgCl)
0	1.0	1	1000	-2.00
1	0.95	1	950	-2.08
3.9	0.70	1	700	-1.70
4.7	1.40	---	0	-1.90

Acid leaked around carbon dividers and corroded lithium again.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

18. Lithium-Cuprous Chloride.

Cell structure - exactly the same as for cell No. 15. (Carbon vessel contains cathode solution.)

Anode - Two lithium blocks used in previous cells (surfaces scraped).

Weights of lithium blocks (including nickel screen) = 21.13 grams and 19.96 grams.

Cathode - Cu_2Cl_2 (dissolved), 1 molal in dimethylformamide. 45 ml. used.

Electrolyte - 1 molal $(C_6H_5)(CH_3)_3NPF_6$-N-nitrosodimethylamine.

Weight of carbon vessel = 37.74 grams.

Total weight of cell = 283.1 grams.

Reference electrode - Ag/AgCl.

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Ag/AgCl (Volts)
0	2.79	---	0	-2.80
0.9	2.80	---	0	-2.80
1.1	2.55	100	25.5	-2.76
2.4	2.50	100	25.0	-2.77
4.3	2.45	100	24.5	-2.78
9.9	2.41	100	24.1	-2.79
10.3	2.22	50	22.2	-2.77
11.4	2.15	50	21.5	-2.78
15	2.11	50	21.1	-2.79
20	2.10	50	21.0	-2.79
20.1	----	30	----	----
20.2	1.91	30	63.7	-2.77
25	1.83	30	61.0	-2.78
30	1.81	30	60.2	-2.78
30.1	1.73	25	69.4	-2.77
34.2	1.70	25	68.0	-2.78
40	1.69	25	67.6	-2.78
40.5	1.60	20	80.0	-2.77
43.8	1.57	20	78.6	-2.78
49.7	1.53	20	76.5	-2.78

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

18. Lithium-Cuprous Chloride. (Continued)

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Ag/AgCl (Volts)
50	1.27	10	127	-2.74
54.7	1.14	10	114	-2.73
59.8	1.10	10	110	-2.73
60	0.82	5	164	-2.70
60.5	0.80	5	160	----
64.5	0.74	5	148	-2.70
66.2	0.73	5	146	-2.70
70	----	100	--	-2.80
70.5	1.95	100	19.5	-2.81
75	2.19	100	21.9	-2.82
80	2.23	100	22.3	-2.82
85	2.27	100	22.7	-2.81
90	2.30	100	23.0	-2.82
90.2	2.00	30	86.7	-2.79
95	1.83	30	61.0	-2.79
100	1.78	30	59.3	-2.79
105	1.73	30	57.6	-2.79
113	1.68	30	56.0	-2.79
127	1.62	30	54.0	-2.80
138	1.56	30	52.0	-2.79
149	1.54	30	51.3	-2.80
150	----	---	0	----
151	2.28	---	0	-2.87
152	2.38	---	0	-2.88
158	2.48	---	0	-2.88

Observation: No insoluble corrosion products had formed at the lithium surface.

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

19. Lithium-Cupric Chloride.

Cell structure - A cylindrical carbon vessel containing an aqueous solution of cupric chloride dihydrate ($CuCl_2 \cdot 2H_2O$) was positioned between two lithium electrodes which were immersed in an ethylene carbonate-propylene carbonate electrolyte in a polyethylene container. The carbon vessel served as the cathode current collector.

Description of cell components -

A. Carbon vessel:

Dimensions:
External diameter = 5 cm.
Length = 3.5 cm.
Internal diameter = 4 cm.
Internal length = 3 cm.

Weight of vessel = 47.7 grams.

B. Polyethylene container:

Weight = 39.7 grams.

C. Lithium anodes - The block anodes from cell No. 18 were utilized again.

Weights of lithium anodes, 18.50 and 18.25 grams.

Weights of nickel screens, 1.50 and 1.50 grams.

D. Cathode solution - 1 molal $CuCl_2 \cdot 2H_2O$ in water. Volume employed = 50 ml.

E. Electrolyte - 1 molal $(C_6H_5)(CH_3)_3NPF_6$ in ethylene carbonate (80%)-propylene carbonate (20%). Volume employed = 100 ml.

Total weight of assembled cell = approximately 300 grams.

Reference electrode - Ag/AgCl

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

19. Lithium-Cupric Chloride. (Continued)

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Reference (Volts)
0	----	---	0	----
2	2.83	---	0	-2.72
2.6	2.30	100	23	-2.40
4.5	2.25	100	22.5	-2.45
13.4	2.13	100	21.3	-2.43
23.2	1.91	100	19.1	-2.42
47.6	1.77	100	17.7	-2.50
54	1.75	100	17.5	-2.51
56	1.83	100	18.3	-2.52
74.2	1.63	100	16.3	-2.48
83	----	---	0	-2.55
84.7	2.27	---	0	-2.64
86	1.80	100	18.0	-2.38
91	1.73	100	17.3	-2.49
98.5	1.79	100	17.9	-2.47
113	1.68	100	16.8	-2.47
122	1.69	100	16.9	-2.46
150	1.67	100	16.7	-2.45
157	1.68	100	16.8	-2.46
174	1.72	100	17.2	-2.44
197	1.65	100	16.5	-2.43
209	1.61	100	16.1	-2.43
245	1.40	100	14.0	----
425	1.20	100	12.0	----
545	1.20	100	12.0	----

TABLE IV. DISCHARGE OF CELLS UTILIZING DISSOLVED ACTIVE CATHODE MATERIALS (Continued)

19. Lithium-Cupric Chloride. (Continued)

Time (Minutes)	Cell Voltage	Resistance (Ohms)	Current (ma)	Lithium vs. Refefence (Volts)
665	1.00	100	10.0	----
905	1.00	100	10.0	----
1025	1.00	100	10.0	----
1085	1.10	100	11.0	----
1110	1.10	100	11.0	----
1111	1.58	---	0	-2.35
1118	1.96	---	0	-2.39
1127	2.03	---	0	-2.39

TABLE V. CHEMICAL STABILITY OF ANODE MATERIALS IN ELECTROLYTE SOLUTIONS

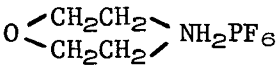
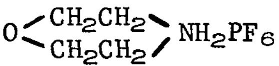
Tests of Two Weeks Duration.			Page
A.	Stability in Acetonitrile Solutions.		IV-78
	Solute	**Anode Material**	
1.	$(n\text{-}C_4H_9)_2NH_2AsF_6$ (Di-n-butylammonium hexafluoroarsenate)	Ca Mg	
2.	$AlCl_3$	Mg	
3.	 $O\langle {}^{CH_2CH_2}_{CH_2CH_2} \rangle NH_2PF_6$ (Morpholinium hexafluorophosphate)	Ca	
4.	$(n\text{-}C_3H_7)_4NPF_6$ (Tetra-n-propylammonium hexafluorophosphate)	Ca	
B.	Stability in Dimethylformamide Solutions.		IV-80
1.	$(CH_3)_4NPF_6$ (Tetramethylammonium hexafluorophosphate)	Mg	
2.	$(n\text{-}C_4H_9)_2NH_2AsF_6$ (Di-n-butylammonium hexafluoroarsenate)	Ca Li Mg	
3.	 $O\langle {}^{CH_2CH_2}_{CH_2CH_2} \rangle NH_2PF_6$ (Morpholinium hexafluorophosphate)	Ca Mg	
4.	$(C_6H_5)(CH_3)_3NPF_6$ (N-phenyl N,N,N-trimethylammonium hexafluorophosphate)	Ca Li Mg	
5.	$(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	Ca Li Mg	
C.	Stability in 80 WT % Ethylene Carbonate-20 WT % Propylene Carbonate Solutions.		IV-83
1.	$(C_6H_5)(CH_3)_3NPF_6$ (N-phenyl N,N,N-trimethylammonium hexafluorophosphate)	Ca Li Mg	

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

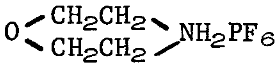
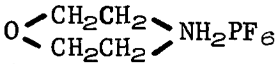
			Page
C.	Stability in 80 WT % Ethylene Carbonate-20 WT % Propylene Carbonate Solutions. (Continued)		IV-83
2.	$(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	Ca Mg	
3.	 $O\langle{}^{CH_2CH_2}_{CH_2CH_2}\rangle NH_2PF_6$ (Morpholinium hexafluorophosphate)	Ca Mg	
D.	Stability in N-Nitrosodimethylamine Solutions.		IV-85
1.	$(n\text{-}C_4H_9)_2NH_2AsF_6$ (Di-n-butylammonium hexafluoroarsenate)	Mg	
2.	 $O\langle{}^{CH_2CH_2}_{CH_2CH_2}\rangle NH_2PF_6$ (Morpholinium hexafluorophosphate)	Li Mg	
3.	$(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	Li	

TABLE V. CHEMICAL STABILITY OF ANODE MATERIALS IN ELECTROLYTE SOLUTIONS

Tests of Two Weeks Duration.

A. Stability in Acetonitrile Solutions.

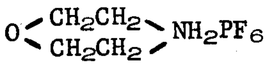
	Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Two Week Exposure
1.	$(n\text{-}C_4H_9)_2NH_2AsF_6$ (Di-n-butyl-ammonium hexa-fluoroarsenate)	Ca	Before Exposure	0.5840	4.17×10^{-2} (25°C)	4.06×10^{-2} (25°C)	Darkening of the entire calcium surface; thick black film formed on part of the calcium.
			After Two Weeks	0.6217	4.34×10^{-2} (25°C)	4.25×10^{-2} (25°C)	
		Mg	Before Exposure	0.2140	4.12×10^{-2} (25°C)	4.06×10^{-2} (25°C)	Formation of thin, white film on part of the magnesium surface.
			After Two Weeks	0.2202	4.38×10^{-2} (25°C)	4.25×10^{-2} (25°C)	
2.	$AlCl_3$	Mg	Before Exposure	0.6123	2.87×10^{-2} (28°C)	2.94×10^{-2} (28°C)	A loosely adhering black solid formed at the magnesium surface.
			After Two Weeks	0.5880	2.65×10^{-2} (26°C)	2.84×10^{-2} (26°C)	
3.	 $O<\begin{smallmatrix}CH_2CH_2\\CH_2CH_2\end{smallmatrix}>NH_2PF_6$ (Morpholinium hexafluorophos-phate)	Ca	Before Exposure	0.2826	4.61×10^{-2} (27°C)	4.44×10^{-2} (27°C)	Some consumption of the calcium. Formation of a thick, black film on the calcium surface. White solid formed in the solution.
			After Two Weeks	0.1848	4.64×10^{-2} (26°C)	4.20×10^{-2} (26°C)	

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

Tests of Two Weeks Duration. (Continued)

A. Stability in Acetonitrile Solutions. (Continued)

	Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Two Week Exposure
4.	$(n\text{-}C_3H_7)_4NPF_6$ (Tetra-n-propyl-ammonium hexa-fluorophosphate)	Ca	Before Exposure	0.3387	4.15×10^{-2} (27°C)	4.05×10^{-2} (27°C)	Formation of a thin, gray film on the calcium. The calcium had a duller appearance. Slight amount of orange-brown solid formed in solution.
			After Two Weeks	0.3319	4.10×10^{-2} (26°C)	4.01×10^{-2} (26°C)	

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

Tests of Two Weeks Duration. (Continued)
B. Stability in Dimethylformamide Solutions.

	Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Two Week Exposure
1.	$(CH_3)_4NPF_6$ (Tetramethyl-ammonium hexa-fluorophosphate)	Mg	Before Exposure	0.2091	1.15×10^{-2} (26°C)	1.15×10^{-2} (26°C)	No change.
			After Two Weeks	0.2210	1.10×10^{-2} (25°C)	1.10×10^{-2} (25°C)	
2.	$(n\text{-}C_4H_9)_2NH_2AsF_6$ (Di-n-butyl-ammonium hexa-fluoroarsenate)	Ca	Before Exposure	0.5633	2.33×10^{-2} (25°C)	2.38×10^{-2} (25°C)	Calcium surface somewhat darkened.
			After Two Weeks	0.6018	2.28×10^{-2} (25°C)	2.33×10^{-2} (25°C)	
		Li	Before Exposure	0.7214	2.33×10^{-2} (27°C)	2.35×10^{-2} (27°C)	Extensive consumption of the lithium. Remaining lithium covered with a thick, yellow-brown solid. The original colorless solution became an opaque red-brown. Some black solid was present.
			After Two Weeks	3.9874	1.29×10^{-2} (25°C)	2.31×10^{-2} (25°C)	
		Mg	Before Exposure	0.2055	2.34×10^{-2} (25°C)	2.38×10^{-2} (25°C)	No change.
			After Two Weeks	0.2106	2.29×10^{-2} (25°C)	2.33×10^{-2} (25°C)	

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

Tests of Two Weeks Duration. (Continued)
B. Stability in Dimethylformamide Solutions. (Continued)

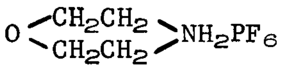
	Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Two Week Exposure
3.	 $O\langle{}^{CH_2CH_2}_{CH_2CH_2}\rangle NH_2PF_6$ (Morpholinium hexafluorophosphate)	Ca	Before Exposure	0.5656	2.71 x 10^{-2} (26°C)	2.68 x 10^{-2} (26°C)	Formation of thick white film at calcium surface. Solution cloudy.
			After Two Weeks	0.8281	2.65 x 10^{-2} (25°C)	2.65 x 10^{-2} (25°C)	
		Mg	Before Exposure	0.2232	2.73 x 10^{-2} (26°C)	2.68 x 10^{-2} (26°C)	Entire surface of magnesium was blackened. Some white solid was also formed.
			After Two Weeks	0.3167	2.68 x 10^{-2} (25°C)	2.65 x 10^{-2} (25°C)	
4.	$(C_6H_5)(CH_3)_3NPF_6$ (N-phenyl N,N,N-trimethylammonium hexafluorophosphate)	Ca	Before Exposure	0.4221	2.21 x 10^{-2} (27.5°C)	2.25 x 10^{-2} (27.5°C)	Calcium became slightly dull.
			After Two Weeks	0.4198	2.15 x 10^{-2} (25°C)	2.15 x 10^{-2} (25°C)	
		Li	Before Exposure	0.4824	2.12 x 10^{-2} (26°C)	2.14 x 10^{-2} (26°C)	Complete consumption of the lithium. Change in the solution from clear and colorless to opaque orange-brown. Considerable amount of orange-white solid formed in solution.
			After Two Weeks	---	1.61 x 10^{-2} (26°C)	2.08 x 10^{-2} (26°C)	
		Mg	Before Exposure	0.6225	2.23 x 10^{-2} (27.5°C)	2.25 x 10^{-2} (27.5°C)	Magnesium became slightly dull.
			After Two Weeks	0.6217	2.19 x 10^{-2} (25°C)	2.15 x 10^{-2} (25°C)	

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

Tests of Two Weeks Duration. (Continued)
B. Stability in Dimethylformamide Solutions. (Continued)

	Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Two Week Exposure
5.	$(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	Ca	Before Exposure	0.3011	2.20×10^{-2} (27°C)	2.20×10^{-2} (27°C)	Formation of a thick, black film on the calcium. A white solid formed in solution.
			After Two Weeks	0.3717	2.06×10^{-2} (26°C)	2.15×10^{-2} (26°C)	
		Li	Before Exposure	0.6745	2.20×10^{-2} (27°C)	2.22×10^{-2} (27.5°C)	Extensive consumption of the lithium. Remaining lithium covered with a thick, yellow-brown solid. The original light orange solution separated into a small, clear, colorless upper layer and an opaque, dark brown lower layer.
			After Two Weeks	4.9617	1.65×10^{-2} (26°C)	2.15×10^{-2} (26°C)	
		Mg	Before Exposure	0.6375	2.20×10^{-2} (27°C)	2.20×10^{-2} (27°C)	No change.
			After Two Weeks	0.6399	2.15×10^{-2} (26°C)	2.15×10^{-2} (26°C)	

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

Tests of Two Weeks Duration. (Continued)
C. Stability in 80 WT % Ethylene Carbonate-20 WT % Propylene Carbonate Solutions.

	Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Two Week Exposure
1.	$(C_6H_5)(CH_3)_3NPF_6$ (N-phenyl N,N,N-trimethylammonium hexafluorophosphate)	Ca	Before Exposure	0.4858	1.23 x 10^{-2} (28°C)	1.23 x 10^{-2} (28°C)	Formation of a black film which partially covered the calcium surface.
			After Two Weeks	0.4993	1.19 x 10^{-2} (25°C)	1.19 x 10^{-2} (25°C)	
		Li	Before Exposure	0.5461	1.22 x 10^{-2} (26°C)	1.20 x 10^{-2} (26°C)	Formation of a thick, gray film on the lithium surface. Some white solid was present in solution.
			After Two Weeks	1.1810	1.13 x 10^{-2} (26°C)	1.13 x 10^{-2} (26°C)	
		Mg	Before Exposure	0.6127	1.23 x 10^{-2} (28°C)	1.23 x 10^{-2} (28°C)	Gray spots appeared at random over approximately 30% of the magnesium surface.
			After Two Weeks	0.6151	1.19 x 10^{-2} (25°C)	1.19 x 10^{-2} (25°C)	
2.	$(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	Ca	Before Exposure	0.2633	1.23 x 10^{-2} (28°C)	1.23 x 10^{-2} (28°C)	The calcium dulled and was partially covered with a gray-black film.
			After Two Weeks	0.2704	1.15 x 10^{-2} (26°C)	1.15 x 10^{-2} (26°C)	
		Mg	Before Exposure	0.5785	1.26 x 10^{-2} (28°C)	1.23 x 10^{-2} (28°C)	No change.
			After Two Weeks	0.5824	1.16 x 10^{-2} (26°C)	1.15 x 10^{-2} (26°C)	

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

Tests of Two Weeks Duration. (Continued)
C. Stability in 80 WT % Ethylene Carbonate-20 WT % Propylene Carbonate Solutions. (Continued)

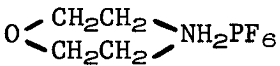
	Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Two Week Exposure
3.	 $O\langle^{CH_2CH_2}_{CH_2CH_2}\rangle NH_2PF_6$ (Morpholinium hexafluorophosphate)	Ca	Before Exposure	0.5134	1.33×10^{-2} (28°C)	1.28×10^{-2} (28°C)	Formation of a thick, black film on the calcium surface.
			After Two Weeks	0.5331	1.27×10^{-2} (25°C)	1.26×10^{-2} (25°C)	
		Mg	Before Exposure	0.6284	1.34×10^{-2} (28°C)	1.28×10^{-2} (28°C)	The magnesium surface became slightly covered with black spots.
			After Two Weeks	0.6560	1.28×10^{-2} (25°C)	1.26×10^{-2} (25°C)	

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

Tests of Two Weeks Duration. (Continued)

D. Stability in N-Nitrosodimethylamine Solutions.

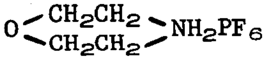
	Solute	Anode Material	Time of Measurement	Weight of Anode Material (Grams)	Specific Conductance (ohm^{-1} cm^{-1}) Exposed Electrolyte	Specific Conductance (ohm^{-1} cm^{-1}) Blank Electrolyte	Visible Change After Two Week Exposure
1.	$(n\text{-}C_4H_9)_2NH_2AsF_6$ (Di-n-butyl-ammonium hexa-fluoroarsenate)	Mg	Before Exposure	0.2033	2.37 x 10^{-2} (26°C)	2.40 x 10^{-2} (26°C)	No change.
			After Two Weeks	0.2062	2.31 x 10^{-2} (25°C)	2.36 x 10^{-2} (24°C)	
2.	 $O{<}^{CH_2CH_2}_{CH_2CH_2}{>}NH_2PF_6$ (Morpholinium hexafluorophos-phate)	Li	Before Exposure	0.5365	2.74 x 10^{-2} (26°C)	2.59 x 10^{-2} (26°C)	Some consumption of the lithium. Formation of thin, gray film at lithium surface. White gelatinous suspension in the solution.
			After Two Weeks	0.3488	1.81 x 10^{-2} (26°C)	2.68 x 10^{-2} (26°C)	
		Mg	Before Exposure	0.2048	2.65 x 10^{-2} (26°C)	2.70 x 10^{-2} (26°C)	No change.
			After Two Weeks	0.2089	2.67 x 10^{-2} (25°C)	2.67 x 10^{-2} (25°C)	
3.	$(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propyl-ammonium hexa-fluoroarsenate)	Li	Before Exposure	0.6344	2.36 x 10^{-2} (27°C)	2.38 x 10^{-2} (27°C)	Formation of a thin, gray film on the lithium surface. Original yellow-orange solution separated into a small, clear, colorless upper layer and a larger slightly yellow-brown lower layer. A dark brown solid formed.
			After Two Weeks	0.6347	1.85 x 10^{-2} (25°C)	2.30 x 10^{-2} (25°C)	

TABLE VI. CHEMICAL STABILITY OF CATHODE MATERIALS IN ELECTROLYTE SOLUTIONS

TABLE VI. CHEMICAL STABILITY OF CATHODE MATERIALS IN ELECTROLYTE SOLUTIONS.

Tests of Twenty-Four Hours Duration.

A. Stability in Acetonitrile Solutions.

Solute	Cathode Material	Visible Change After 24 Hour Exposure
1. $AlCl_3$	CuF_2	Solution color changed from colorless to green.
	MnO_2	Solution color changed from colorless to tan.
	CoF_3	Solution color changed from colorless to brown. CoF_3 color changed from brown to blue.
	Cu	No change.
2. LiCl	CuF_2	No change.
	MnO_2	No change.
	CoF_3	No change.
	Cu	No change.
3. $(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$ (N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate)	CuF_2	Solution color changed from faint yellow to colorless.
	MnO_2	Solution color changed from faint yellow to colorless.
	CoF_3	Solution color changed from faint yellow to colorless.
	Cu	Solution color changed from faint yellow to colorless.
4. $(CH_3)_4NPF_6$ (Tetramethylammonium hexafluorophosphate)	CuF_2	No change.
	MnO_2	No change.
	CoF_3	No change.
	Cu	No change.

TABLE VI. CHEMICAL STABILITY OF CATHODE MATERIALS IN ELECTROLYTE SOLUTIONS

Tests of 24 Hours Duration.

A. Stability in Acetonitrile Solutions. (Continued)

	Solute	Cathode Material	Visible Change After 24 Hour Exposure
5.	$(C_6H_5)(CH_3)_3NPF_6$ (N-Phenyl N,N,N-trimethylammonium hexafluorophosphate)	CuF_2	Solution color changed from clear gray to blue-green.
		MnO_2	Solution color changed slightly from clear gray to light blue.
		CoF_3	Solution color changed from clear gray to orange-yellow.
		Cu	No change.
6.	$(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	CuF_2	No change.
		MnO_2	Solution color deepened.
		CoF_3	No change.
		Cu	No change.

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

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

Solute	Cathode Material	Visible Change After 24 Hour Exposure
1. LiCl	CuF_2	Solution color changed from colorless to orange. CuF_2 changed from gray to yellow.
	MnO_2	No change.
	CoF_3	Solution color changed from colorless to dark blue. CoF_3 color changed from brown to black.
	Cu	No change.
2. $(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$ (N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate)	CuF_2	No change.
	MnO_2	No change.
	CoF_3	No change.
	Cu	No change.
3. $(CH_3)_4NPF_6$ (Tetramethylammonium hexafluorophosphate)	CuF_2	Color of CuF_2 changed from light gray to darker gray.
	MnO_2	No change.
	CoF_3	Solution changed from colorless to faint red; CoF_3 color darkened.
	Cu	No change.
4. $(C_6H_5)(CH_3)_3NPF_6$ (N-Phenyl N,N,N-trimethylammonium hexafluorophosphate)	CuF_2	No change.
	MnO_2	No change.
	CoF_3	Solution color changed from clear gray to clear brown. CoF_3 color darkened.
	Cu	No change.
5. $(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	CuF_2	No change.
	MnO_2	Solution color somewhat deeper.
	CoF_3	Solution color somewhat deeper; CoF_3 color darker.
	Cu	No change.

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

Tests of Twenty-Four Hours Duration. (Continued)

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

Solute	Cathode Material	Visible Change After 24 Hour Exposure
1. $AlCl_3$	CuF_2	Solution color changed from light brown to deep amber. CuF_2 color changed from gray to black.
	MnO_2	Solution color changed from light brown to dark green.
	CoF_3	Solution color changed from light brown to dark blue. CoF_3 color changed from brown to blue.
	Cu	No change.
2. LiCl	CuF_2	Solution color changed from colorless to orange-yellow. CuF_2 color changed from gray to yellow-green.
	MnO_2	No change.
	CoF_3	Solution color changed from colorless to blue.
	Cu	No change.
3. $(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$ (N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate)	CuF_2	No change.
	MnO_2	No change.
	CoF_3	CoF_3 color darkened.
	Cu	No change.
4. $(CH_3)_4NPF_6$ (Tetramethylammonium hexafluorophosphate)	CuF_2	No change.
	MnO_2	No change.
	CoF_3	CoF_3 color darkened.
	Cu	No change.

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

Tests of 24 Hours Duration. (Continued)
C. Stability in Ethylene Carbonate (80%)-Propylene Carbonate (20%) Solutions. (Continued)

Solute	Cathode Material	Visible Change After 24 Hour Exposure
5. $(C_6H_5)(CH_3)_3NPF_6$ (N-Phenyl N,N,N-trimethylammonium hexafluorophosphate)	CuF_2	Solution color changed slightly from light gray to light green.
	MnO_2	Solution color became green.
	CoF_3	CoF_3 color darkened appreciably; solution color became yellow.
	Cu	No change.
6. $(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	CuF_2	No change.
	MnO_2	No change.
	CoF_3	Solution color changed from yellow to brown; CoF_3 darkened.
	Cu	No change.
7. $(n\text{-}C_4H_9)_2H_2NAsF_6$ (Di-n-butylammonium hexafluoroarsenate)	CuF_2	No change.
	MnO_2	No change.
	CoF_3	No change.
	Cu	No change.

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

Tests of Twenty-Four Hours Duration. (Continued)
D. Stability in N-Nitrosodimethylamine Solutions.

Solute	Cathode Material	Visible Change After 24 Hour Exposure
1. $AlCl_3$	CuF_2	CuF_2 color changed from light gray to light brown.
	MnO_2	Solution color changed from amber to deep yellow-green.
	CoF_3	Solution color changed from amber to dark green; CoF_3 color changed from brown to blue.
	Cu	Solution color changed from amber to orange.
2. LiCl	CuF_2	Solution color changed from yellow to amber. CuF_2 color changed from gray to yellow.
	MnO_2	No change.
	CoF_3	Solution color changed from yellow to blue-green.
	Cu	No change.
3. $(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$ (N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate)	CuF_2	Solution color changed from yellow to yellow-green.
	MnO_2	Solution color changed from yellow to yellow-green.
	CoF_3	Solution color changed from yellow to yellow-green.
	Cu	Solution color changed from yellow to yellow-green.

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

Tests of 24 Hours Duration. (Continued)
D. Stability in N-Nitrosodimethylamine Solutions. (Continued)

	Solute	Cathode Material	Visible Change After 24 Hour Exposure
4.	$(CH_3)_4NPF_6$ (Tetramethylammonium hexafluorophosphate)	CuF_2	No change.
		MnO_2	No change.
		CoF_3	CoF_3 color darkened.
		Cu	No change.
5.	$(C_6H_5)(CH_3)_3NPF_6$ (N-Phenyl N,N,N-trimethylammonium hexafluorophosphate)	CuF_2	No change.
		MnO_2	No change.
		CoF_3	CoF_3 color darkened appreciably.
		Cu	No change.
6.	$(n\text{-}C_3H_7)_3NHAsF_6$ (Tri-n-propylammonium hexafluoroarsenate)	CuF_2	No change.
		MnO_2	No change.
		CoF_3	CoF_3 color darkened.
		Cu	No change.
7.	$(n\text{-}C_4H_9)_2H_2NAsF_6$ (Di-n-butylammonium hexafluoroarsenate)	CuF_2	No change.
		MnO_2	No change.
		CoF_3	No change.
		Cu	No change.

TABLE VII. SPECIFIC CONDUCTANCE OF SOLUTIONS

Solute	Specific Conductance (ohm^{-1} cm^{-1})	Visual Observations		
		Solution	Solid	Others
	a. Acetonitrile			
$AlCl_3$	3.18×10^{-2} (26°C)	Clear	None	Exothermic heat of dissolution
NH_4PF_6 (Ammonium hexafluorophosphate)	4.20×10^{-2} (24°C)	Clear	White	None
$(n\text{-}C_4H_9)_4NCl$ (Tetra-n-butylammonium chloride)	2.23×10^{-2} (26°C)	Clear, orange	None	None
	b. Dimethylformamide			
NH_4PF_6 (Ammonium hexafluorophosphate)	2.96×10^{-2} (24°C)	Clear, colorless	Small amount, white	None
	c. 80% Ethylene Carbonate-20% Propylene Carbonate			
* $MgCl_2$	4.52×10^{-3} (27°C)	Turbid	Large amount, white	None
$(n\text{-}C_3H_7)_4NPF_6$ (Tetra-n-propylammonium hexafluorophosphate)	1.20×10^{-2} (27°C)	Clear, orange	None	None
$(n\text{-}C_4H_9)_4NCl$ (Tetra-n-butylammonium chloride)	8.15×10^{-3} (27°C)	Clear, orange	None	None
	d. N-Nitrosodimethylamine			
$AlCl_3$	2.06×10^{-2} (27°C)	Clear, tan	Slight amount, white	Exothermic heat of dissolution
$FeCl_3$	1.72×10^{-2} (26°C)	Clear, brown	Tan	None
** $K_3Cr(SCN)_6$	1.94×10^{-2} (30°C)	Clear, purple	None	None
* KI	8.82×10^{-3} (30°C)	Clear, orange	Large amount, white	None
* $MgCl_2$	1.08×10^{-2} (27°C)	Clear, yellow	Large amount, white	None

---

All solutions were one molal unless noted to be saturated or of another concentration.

* saturated

** 0.5 molal

TABLE VII. SPECIFIC CONDUCTANCE OF SOLUTIONS (Continued)

Solute	Specific Conductance (ohm^{-1} cm^{-1})	Visual Observations		
		Solution	Solid	Others
	d. N-Nitrosodimethylamine (Continued)			
* $(CH_3)_4NPF_6$ (Tetramethylammonium hexafluorophosphate)	8.21×10^{-3} (26°C)	Clear, yellow	Large amount, white	None
$(C_6H_5)(CH_3)_3NPF_6$ (N-Phenyl N,N,N-trimethylammonium hexafluorophosphate)	2.32×10^{-2} (28°C)	Dark green	Small amount, dark	None
$(p\text{-}C_{12}H_{25}C_6H_4CH_2)(CH_3)_3NPF_6$ (N-(p-Dodecylbenzyl)N,N,N-trimethylammonium hexafluorophosphate)	1.13×10^{-2} (26°C)	Clear, yellow	Tan	None
$(n\text{-}C_3H_7)_4NAsF_6$ (Tetra-n-propylammonium hexafluoroarsenate)	2.08×10^{-2} (26°C)	Clear, brown	Tan	None
$(C_6H_5)_4AsCl$ (Tetraphenylarsonium chloride)	1.32×10^{-2} (26°C)	Clear, yellow	White	None

---

All solutions were one molal unless noted to be saturated or of another concentration.

* saturated

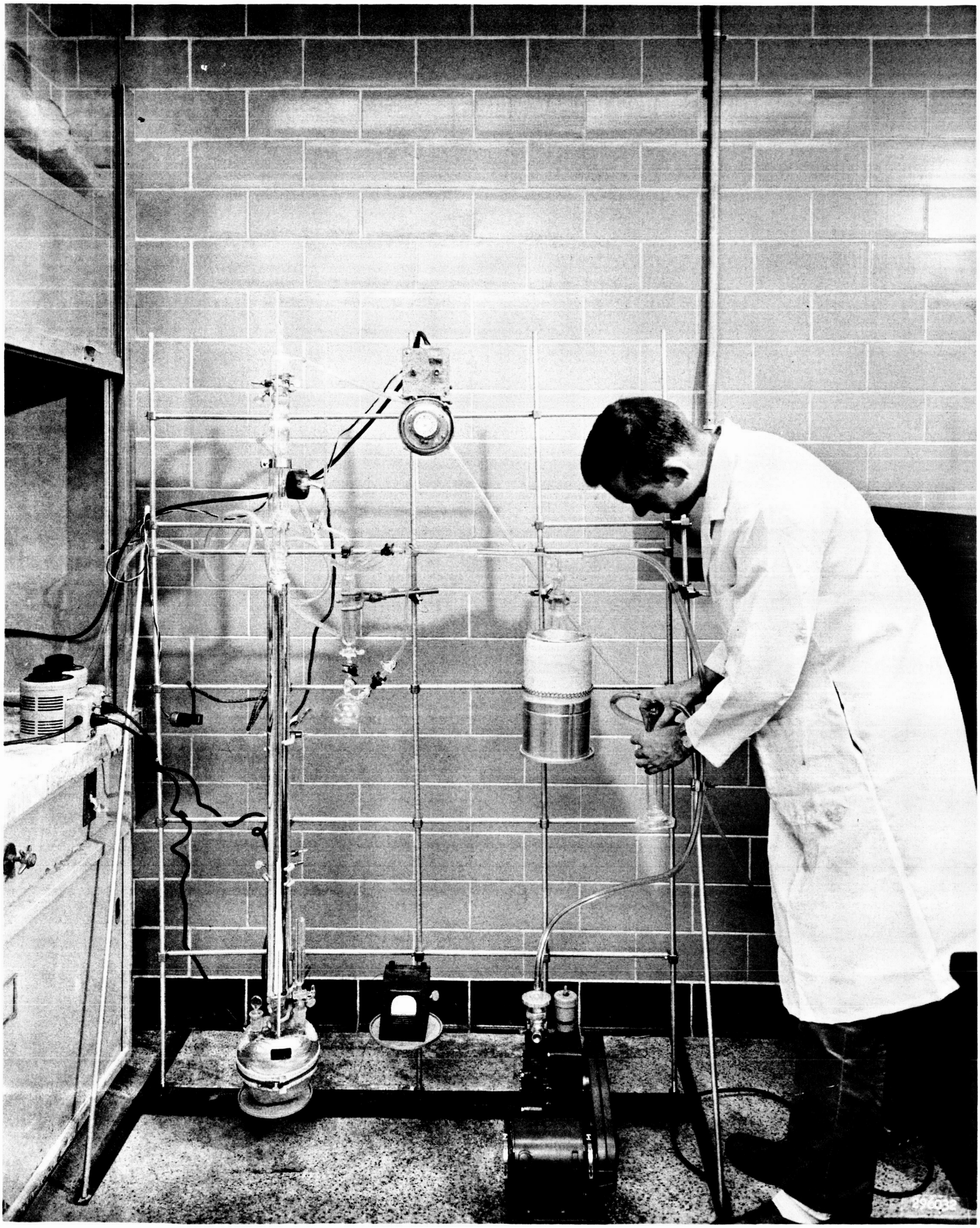


FIGURE 3. VACUUM DISTILLATION APPARATUS FOR PURIFICATION OF SOLVENTS

TABLE VIII. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES IN PURIFIED N,N-DIMETHYLFORMAMIDE

	Electrolyte				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
	Anode	Solvent	Solute	Reference Electrode	Counter Electrode				Initial	Final	
1.	Li	DMF (Not Distilled)	*	Ag/AgCl	Ag/AgCl	-3.12	-3.18	0.1	-3.12	-2.17	The lithium anode began gassing moderately after four minutes of the 100 ma discharge and turned black. Slight gassing continued on open circuit. The counter electrode deteriorated slightly. No change in the appearance of the solution or the reference electrode. System capable of sustaining 100 ma/cm^2.
							-3.19	1	-3.03	-3.06	
							-3.20	10	-2.77	-2.85	
							-3.18	100	-1.65	-1.77	
	Li	DMF (Distilled)	*	Ag/AgCl	Ag/AgCl	-3.08	-3.10	0.1	-3.06	-3.07	Slight gassing at the lithium anode began during the 10 ma discharge. At 100 ma, the gassing rate was moderate and lithium blackened. Slight gassing continued on open circuit. The counter electrode deteriorated slightly. No change in the appearance of the solution or the reference electrode. System capable of sustaining 100 ma/cm^2.
							-3.12	1	-2.90	-2.96	
							-3.16	10	-2.68	-2.84	
							-3.17	100	-1.90	-2.06	

* N-phenyl-N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

TABLE VIII. ELECTROCHEMICAL HALF-CELL SCREENING OF ANODES IN PURIFIED N,N-DIMETHYLFORMAMIDE (Continued)

Electrolyte								Voltage of Anode vs. Reference Electrode at Various Current Densities		
Anode	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)	Initial	Final	Further Observations and Remarks
2. Mg	DMF (Not Distilled)	*	Ag/AgCl	Ag/AgCl	-1.47	-1.47	0.1	-1.45	-1.47	Gassing occurred at the magnesium anode during the 10 ma discharge and black streaks appeared on its surface. No change in the appearance of the solution or the counter and reference electrodes. System capable of sustaining 0.1 ma/cm^2.
						-1.47	1	-0.90	-0.63	
						-1.32	10	-0.40	-0.53	
						-1.29	100	Reversed Polarity	----	
Mg	DMF (Distilled)	*	Ag/AgCl	Ag/AgCl	-1.43	-1.43	0.1	-1.40	-1.43	Gassing occurred at the magnesium anode during the 10 ma discharge and black streaks appeared on its surface. No change in the appearance of the solution or the counter and reference electrodes. System capable of sustaining 1 ma/cm^2.
						-1.42	1	-0.68	-1.41	
						-1.35	10	-0.48	-0.46	
						-0.85	100	Reversed Polarity	----	

* N-phenyl-N,N,N-trimethylammonium hexafluorophosphate - $(C_6H_5)(CH_3)_3NPF_6$

TABLE IX. SPECIFIC CONDUCTIVITIES AND REFRACTIVE INDICES OF DISTILLED SOLVENTS

					After One Week		
Fraction	n_D	Vapor Temp.	Vapor Pressure (mm of Mercury)	Specific Conductance (ohm^{-1} cm^{-1})	n_D	Specific Conductance (ohm^{-1} cm^{-1})	Change in Conductivity
				a. Acetonitrile*			
Blank	1.3415^{25}	---	---	2.13×10^{-6} (25°C)	---	---	---
1. (100 ml)	Discard	25°C	56 mm	---	---	---	---
2. (100 ml)	1.3414^{25}	28-29°C	54 mm	7.25×10^{-7} (27°C)	---	---	---
3. (100 ml)	1.3412^{25}	25-26°C	50 mm	8.58×10^{-7} (25°C)	---	---	---
4. (100 ml)	1.3415^{25}	26-27°C	28-29 mm	3.10×10^{-7} (25°C)	---	---	---
5. (12 ml)	1.3416^{25}	26-27°C	28-29 mm	---	---	---	---
				b. N,N-Dimethylformamide**			
Blank	1.4281^{25}	---	---	1.30×10^{-6} (25°C)	1.4281^{25}	1.33×10^{-6} (27°C)	0.03×10^{-6}
1. (100 ml)	Discard	56°C	14 mm	---	---	---	---
2. (100 ml)	1.4279^{25}	51-52°C	13 mm	1.65×10^{-6} (26°C)	1.4281^{25}	5.91×10^{-6} (25°C)	4.26×10^{-6}
3. (100 ml)	1.4280^{25}	52°C	11 mm	6.66×10^{-7} (26°C)	1.4282^{25}	1.78×10^{-6} (26°C)	1.11×10^{-6}
4. (100 ml)	1.4279^{25}	50°C	10 mm	5.34×10^{-6} (26°C)	1.4282^{25}	8.25×10^{-6} (26°C)	2.91×10^{-6}
5. (100 ml)	1.4281^{25}	46°C	9 mm	4.46×10^{-6} (26°C)	1.4281^{25}	7.95×10^{-6} (26°C)	3.49×10^{-6}
6. (100 ml)	1.4279^{25}	37-38°C	7 mm	7.43×10^{-7} (26°C)	1.4280^{25}	2.82×10^{-6} (26°C)	2.08×10^{-6}

---

* $n_D^{16.5} = 1.34596$, Handbook of Chem. and Physics., Chem. Rubber Co., 44th Ed.
$L = 5.9 \times 10^{-8}$ ohm^{-1} cm^{-1}, J. Electrochem. Soc., 112, 104, (1965)

** $n_D^{25} = 1.4269$, DMF, Dupont Product Information Bulletin
$n_D^{25} = 1.4294$, J. Electrochem. Soc., 99, 28 (1952)
$L = 0.6 - 2.0 \times 10^{-7}$ ohm^{-1} cm^{-1}, J. Phys. Chem., 59, 16-17, (1955)

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