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SPECULAR LITHIUM DEPOSITS FROM LITHIUM HEXAFLUOROARSENATE/DIETH--ETC(U)

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HEXAFLUOROARSENATE/DIETHYL ETHER ELECTROLYTES .

by

V. R. Koch, J. L. Goldman, C. J. Mattos and M. Mulvaney

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SPECULAR LITHIUM DEPOSITS FROM LITHIUM
HEXAFLUOROARSENATE/DIETHYL ETHER ELECTROLYTES

V. R. Koch^{*1}, J. L. Goldman*, C. J. Mattos and M. Mulvaney
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ABSTRACT

A new class of aprotic organic electrolytes in which to cycle the lithium electrode has been developed. Blends of diethyl ether (DEE) and tetrahydrofuran (THF) incorporating LiAsF_6 have been found to afford Li electrode cycling efficiencies in excess of 98%. In addition, specular deposits of up to 10 C/cm^2 may be plated from these systems. The kinetic stability of these blended electrolytes toward Li is thought to be due to the formation of a protective lithium ethoxide film.

INTRODUCTION

Our ongoing search for electrolytes suitable for use in ambient temperature secondary Li batteries has led us to investigate solutions of LiAsF_6 in propylene carbonate (PC) (1), tetrahydrofuran (THF) (2), and 2-methyltetrahydrofuran (2-Me-THF) (3). Average cycling efficiencies for the Li electrode in these media are 84, 88, and 96%, respectively (1 coul Li/cm^2 at 5 mA/cm^2). In this paper, we elaborate on the discovery of a class of diethyl ether (DEE)-based electrolytes which afford cycling efficiencies in excess of 98% (4,5). Moreover, Li plate morphologies are so regular and dendrite-free that they may be deemed specular in appearance.

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Key words: organic, electrolyte, lithium battery.

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Electrolytes comprising DEE have found use in ambient temperature Li primary batteries (6). More recently, Higgins reported on the stability of purified DEE toward Li and found that 1M LiAsF₆/DEE was stable for over one month at 71°C (7). To our knowledge, however, no reports of DEE-based electrolytes employed in secondary Li batteries have surfaced in the open literature.

While a variety of cosolvents have been found to improve the conductivity of LiAsF₆/DEE electrolyte without degrading Li cycling performance (5), the best electrolyte to date comprises a 90:10 V/V mixture of DEE and THF, 2.5M in LiAsF₆. We refer to this mixture as Blend-90 (B-90). This paper explores the variation of Li half-cell cycling efficiency with LiAsF₆ with the addition of asymmetric ether cosolvents.

EXPERIMENTAL

General. All purification procedures subsequent to distillation and the electrochemical experiments were conducted at ambient temperature under Ar atmosphere in a Vacuum-Atmospheres Corp. dry box equipped with a Model He-493 Dri-Train.

Materials. Tetrahydrofuran (THF), acetonitrile (CH₃CN), dimethoxyethane (DME) (Burdick and Jackson, distilled-in-glass), diethyl ether (DEE) (Fisher, anhydrous), lithium hexafluoroarsenate (LiAsF₆) (U.S. Steel Agricultural Chemicals, electrochemical grade), lithium perchlorate (LiClO₄) (Anderson Physics, highest purity) and lithium tetrafluoroborate (Foote Mineral Company) were used as received. Lithium foil (15 mil) was obtained from Foote Mineral Company sealed under Ar. 2-Methyltetrahydrofuran (2-Me-THF) and 1,3-dioxolane (Aldrich) were distilled off CaH₂ under Ar from a

Perkin-Elmer Model 251 Auto Annular Still. The reflux ratio was 5:1; only the middle 60% fraction was collected. Ethylene oxide and dimethyl ether (Matheson Gas Products) were passed through a Drierite column prior to being introduced into an electrolyte. Methyl ethyl ether and methyl n-propyl ether were synthesized and purified according to literature procedures (8).

Activated neutral alumina (Woelm 200 neutral, activity grade Super 1) was used as received and exposed only to the dry box atmosphere. Approximately 1g desiccant per 5 ml solvent was used in a given purification procedure. The first 10% of solvent eluting through the column was discarded.

Blended electrolytes were prepared as follows: A stock solution of 2.5M $\text{LiAsF}_6/\text{THF}$ was prepared according to our alumina-preelectrolysis alumina (APA) method (2b). Aliquots of the stock solution were then added to the appropriate amount of $\text{LiAsF}_6/\text{DEE}$ to give the desired salt concentration and relative solvent volumes. The $\text{LiAsF}_6/\text{DEE}$ solutions were prepared at ambient temperature by adding the salt to DEE passed through alumina.

Cells and Electrodes. Glass rectangular cells (10 x 40 x 60 mm, Vitro Dynamics) were used for the galvanostatic cycling of Li in a half-cell configuration. The working electrode was a 5.5 cm x 3.8 cm x 5 mil strip cut from Ni 200 or electrolytic Cu sheet (Roblinger). The counter electrode was fabricated from 15 mil Li ribbon. Details regarding cell assembly, Li cycling experiments and the galvanostatic pre-electrolysis technique may be found elsewhere (3c).

Photomicrographs of plated Li were obtained by modifying a 32 mm threaded connector (Ace Glass, No. 7644) and mating it with a threaded

Teflon base (Fig. 1). The top of the adapter was fitted with optical glass; a side arm sealed with a septum allowed the cell to be filled with approximately 15 ml electrolyte. The working electrode consisted of a stainless steel shaft (3 mm dia) press fit into polypropylene. Two Li counter electrodes, each 1 cm², were mounted in stainless steel screws. Connections were made at a constant current source at the base of the cell.

Linear sweep experiments employed a 5 mm diameter vitreous carbon disk working electrode (Tokai) sealed in Pyrex. The counter electrode was Li, and the reference electrode was an Ag wire immersed in 0.1M AgNO₃/CH₃CN. The reference electrode was separated from the working and counter electrodes by a fritted compartment.

Chronopotentiometric plating and stripping of Li on a Li substrate was conducted with a constant current power supply and an automatic cycler (both constructed in-house). The latter allowed plating for a given period of time, then stripped to a preset potential (1.0V anodic of the stripping potential). Upon reaching this potential, stripping was terminated and the cell reverted to open circuit until plating again commenced.

The Li on Li cycling procedure was as follows: A known charge of Li, typically 4.5 coul/cm², was plated onto a Ni electrode. Then, a lesser charge (1.1 coul/cm²) was sequentially stripped from and plated to the original Li plate. After the first 1.1 coul/cm² strip, a 3.4 coul/cm² reservoir of excess Li remained. Were each cycle 100% efficient, the cell would cycle indefinitely with a 3.4 coul/cm² reserve of Li (efficiency = $Q_{\text{stripped}}/Q_{\text{plated}}$). Of course, each stripping cycle is <100% efficient which means that each strip cuts into the Li reserve yielding an apparent

"100%" cycle until the Ni substrate is reached. At this point the excess Li is exhausted and one may calculate the average efficiency per cycle, \bar{E} as in Equation 1,

$$\bar{E} = \frac{Q_s - \frac{Q_{ex}}{n}}{Q_s} \quad (1)$$

where Q_s is the charge of Li stripped, Q_{ex} is the amount of excess Li, and n is the number of "100%" cycles.

RESULTS AND DISCUSSION

Cycling Efficiencies of LiAsF₆/DEE:THF Blended Electrolytes. Figure 2 shows the effect on \bar{E} of adding aliquots of 1.5M LiAsF₆/DEE to 1.5M LiAsF₆/THF. From an 88% average cycling efficiency for THF alone, the efficiency values rise to a maximum of about 98% for 1.5M LiAsF₆/DEE:THF (95:5), a Blend-95 electrolyte. Reproducibility for a given batch of electrolyte is excellent. In one experiment, for example, three B-95 cells run simultaneously gave 143, 145, and 147 "100%" cycles. In terms of \bar{E} , this works out to efficiency values of 97.84, 97.87, and 97.89%, respectively.

On going from 1.5 to 2.5M LiAsF₆/DEE:THF electrolyte, even better efficiencies may be obtained due to better electrolyte conductivity. The dependence of \bar{E} on LiAsF₆ concentration for B-90 electrolyte may be seen in Fig. 3. Beyond 2.5M LiAsF₆, electrolyte viscosity increases, thereby reducing the conductivity. The peak in \bar{E} for 2.5 and 1.5M LiAsF₆/DEE:THF occurs in the B-90 to B-95 range. This may reflect the desirability of complexing each Li⁺ with four THF molecules.

The decrease in conductivity on going from 1.5M LiAsF₆/THF to 1.5M LiAsF₆/DEE is present in Fig. 4. Thus, one may wish to trade off

cycling efficiency for better conductivity in battery applications requiring higher rate capabilities.

Specular Li Deposits. In our experience, morphologically smooth, dendrite-free electrodeposits lead to high cycling efficiency. Of all of the electrolytes we have studied (1-3), the LiAsF₆/blended DEE media afford the most adherent and reflective plates. Figure 5 shows how reflectivity is maintained from a bare stainless steel electrode (A), to 2 C/cm² (B), to 10 C/cm² (C) at 1 mA/cm². Beyond 10 C/cm², the Li surface becomes granular in appearance with a corresponding loss in reflectivity. However, dendrite-free plates of up to 50 C/cm² at 1 mA/cm² may be obtained in this cell under flooded electrolyte conditions.

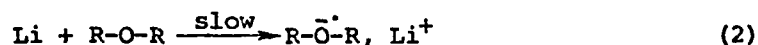
There has been one earlier report of specular Li deposits in the literature. Reddy achieved a reflective 0.9 C/cm² Li plate at 0.1 mA/cm² from a 0.1M LiBr/PC electrolyte (9). The blended LiAsF₆/DEE electrolytes clearly outperform LiBr/PC by an order of magnitude both in rate and in plate thickness.

Effect of Substrate on Cycling Efficiency. Earlier work with Li primary batteries has shown that surface amalgamation of the negative affords a more reproducible cell discharge (10), and better contact between Li and a metallic current collector (11). With this in mind, we set out to explore the effect of a thin Hg film amalgamated onto a Cu substrate. Degreased Cu working electrodes were abraded with alumina in a glove box and then dipped into Hg. A mirror-like Hg film formed on the Cu surface which served as a base onto which Li was plated. In Table 1, we see that presence of the thin Hg film affords almost twice as many cycles from the

3.4 C/cm² base as does bare Ni or Cu. With a thicker Q_{ex} of 16.9 C/cm², over 1000 "100%" cycles were obtained. In this particular experiment, no dendrites were observed until after 450 cycles. This cell was terminated at 1076 cycles due to dendrite bridging, not because the Q_{ex} was exhausted. Thus, one would expect even better cycling behavior by employing separators.

Effect of Ethereal Cosolvents other than THF. In the process of evaluating other ethers as cosolvents with DEE, we noted a striking difference in cycling performance when asymmetric ethers were used. The data in Table 2 show that the addition of methyl ethyl ether or methyl n-propyl ether to LiAsF₆/DEE disastrously reduces the Li cycling efficiency. It is known that R-O-R' bond cleavage is sensitive to variances in the inductive effect of R and R' (12). But on going from Me-O-Me to Me-O-Et to Et-O-Et, changes in the inductive effect or leaving group stability are so slight that the dramatic change in cycling efficiency cannot be explained by C-O cleavage. It is more likely that asymmetric ethers play a subtle but important role in deleteriously affecting Li plate morphology on charge.

Mechanistic Considerations. Earlier work with THF and the methylated THFs indicated that poor cycling efficiency could be ascribed to solvent reduction by Li (2,3). The rate-determining step in this process was taken to be a one-electron transfer from Li to the lowest unfilled molecular orbital (LUMO) of the ether molecule (Equation 2).



Subsequent C-O cleavage and further reduction by Li leads to the observed product stoichiometry (13) (Equation 3).



In a recent paper (3c), we showed that for a series of cyclic ethers, those ethers manifesting kinetic stability to reduction by Li were correspondingly easier to oxidize compared to those ethers which rapidly reacted with Li. We have since performed more accurate linear sweep measurements on several cyclic and acyclic ethers. Rather than measuring potential at an arbitrary current density (3c), we have now determined the peak potential (E_p) of the ether oxidation wave. These data are tabulated in ascending order (Table 3) and are compared to the earlier linear sweep data as well as gas phase ionization potentials from the literature.

We note that variations in the linear sweep data on going from one ether to another are in excellent agreement regardless of experimental procedure. There is, however, little, if any, correlation between the heterogeneous solution phase values and homogeneous gas phase ionization potential data. This most likely reflects differential solvation energies on going from neutral ether to a cation-radical, as well as electrode activation energetics.

Of key importance is the observation that THF and DEE have virtually identical E_p values. Yet as seen in Fig. 2, \bar{E} for 1.5M LiAsF₆/THF is 88% while 1.5M LiAsF₆/DEE gives an \bar{E} of 97%. The IP data are also in conflict with the apparent stability of DEE to Li. DEE is almost 0.1 eV more difficult to oxidize than THF. If the LUMO argument is appropriate, DEE should be easier to reduce than THF.

One possibility is that DEE is in fact reduced as rapidly or even more rapidly than THF. The reduction chemistries yield lithium ethoxide

from DEE and lithium n-butoxide from THF as by-products. Lithium ethoxide is soluble only up to 0.035M in DEE while various lithium butoxides have solubilities in excess of 2M in THF (15). The kinetic stability of DEE and DEE-blends may be due simply to an insoluble, protective lithium ethoxide film, electronically insulating but Li^+ -ion conducting. Since lithium ethoxide is more soluble in solvents more polar than DEE, increasing the relative concentration of THF in the blends results in a more rapid solubilization of the protective ethoxide film. This yields a corresponding decrease in cycling efficiency. Experiments designed to probe the Li surface after storage in a series of blended electrolytes will shed light on this possibility.

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FIGURE CAPTIONS

- Fig. 1 Apparatus used for obtaining photomicrographs of plated Li.
- Fig. 2 Average cycling efficiencies for 1.5 and 2.5M LiAsF₆/DEE:THF as a function of added DEE. $Q_{ex} = 3.4 \text{ C/cm}^2$; $Q_s = 1.1 \text{ C/cm}^2$; $i_p = i_s = 5 \text{ mA/cm}^2$.
- Fig. 3 Effect of LiAsF₆ concentration on \bar{E} for Blend-90 electrolyte.
- Fig. 4 Variation of specific conductance on going from 0 to 100% DEE.
- Fig. 5 Photomicrographs (15X) of specular Li deposits from 2.5M LiAsF₆/B-90 electrolyte. (A) 3 mm diameter stainless steel electrode surface; (B) 2.0 C/cm² electrodeposited Li; (C) 10.0 C/cm² electrodeposited Li. $i = 1 \text{ mA/cm}^2$.

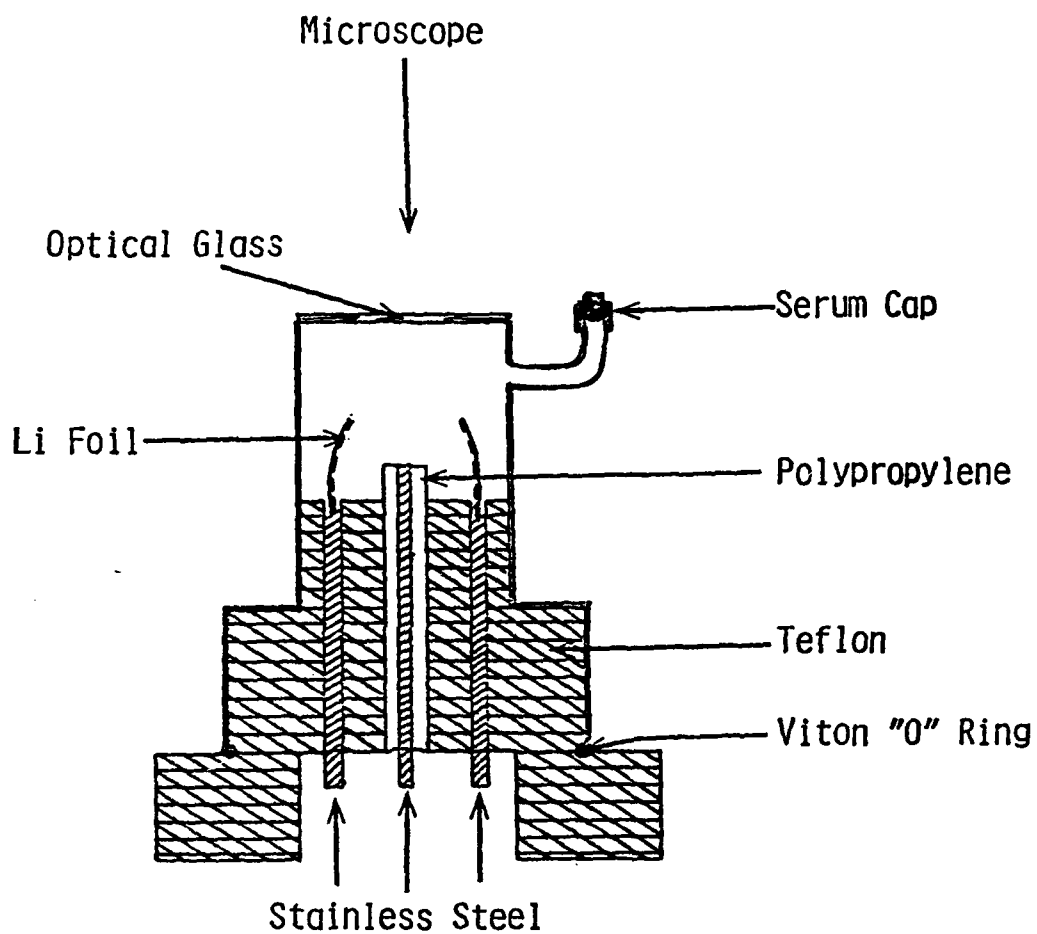


Figure 1

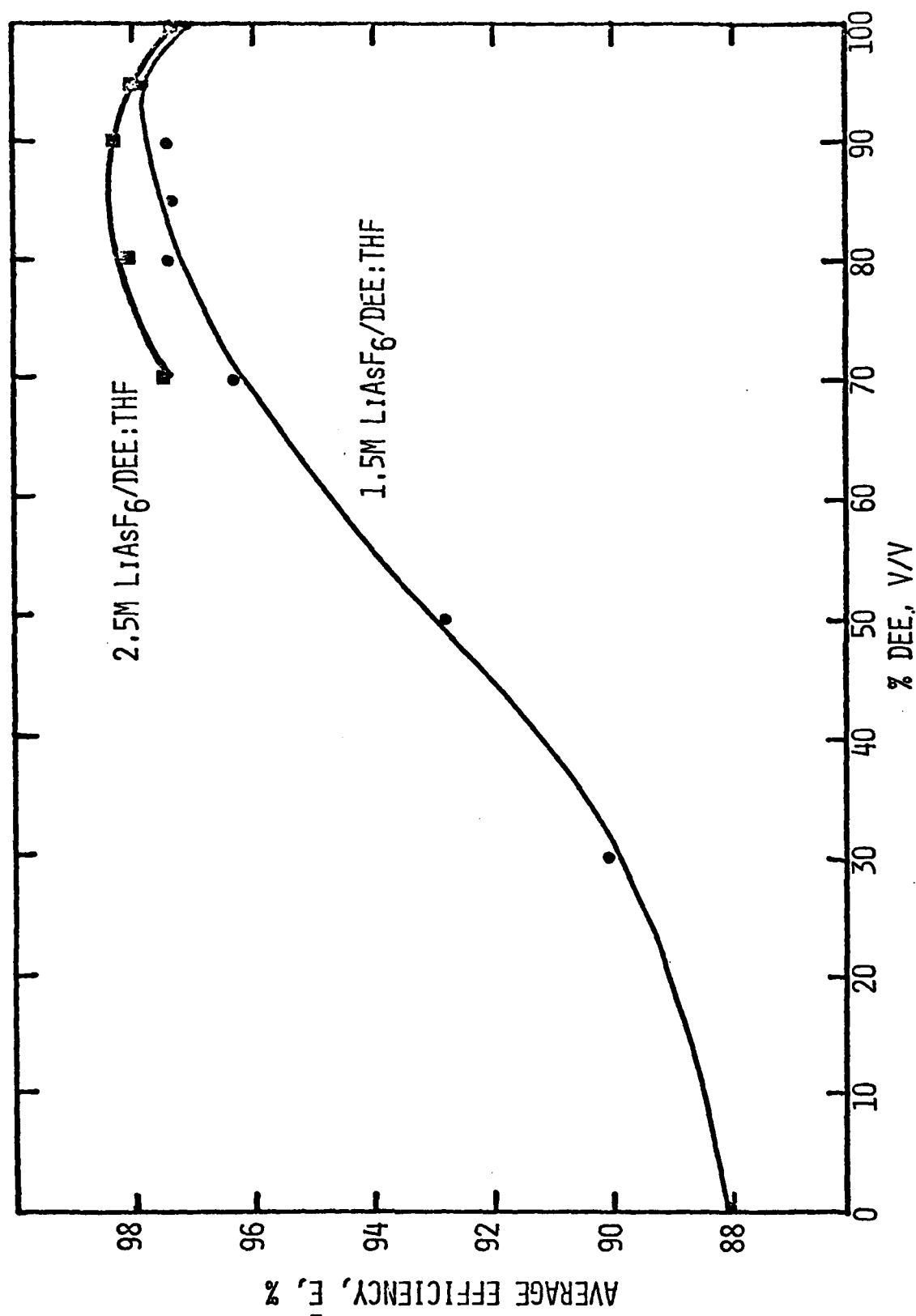


Figure 2

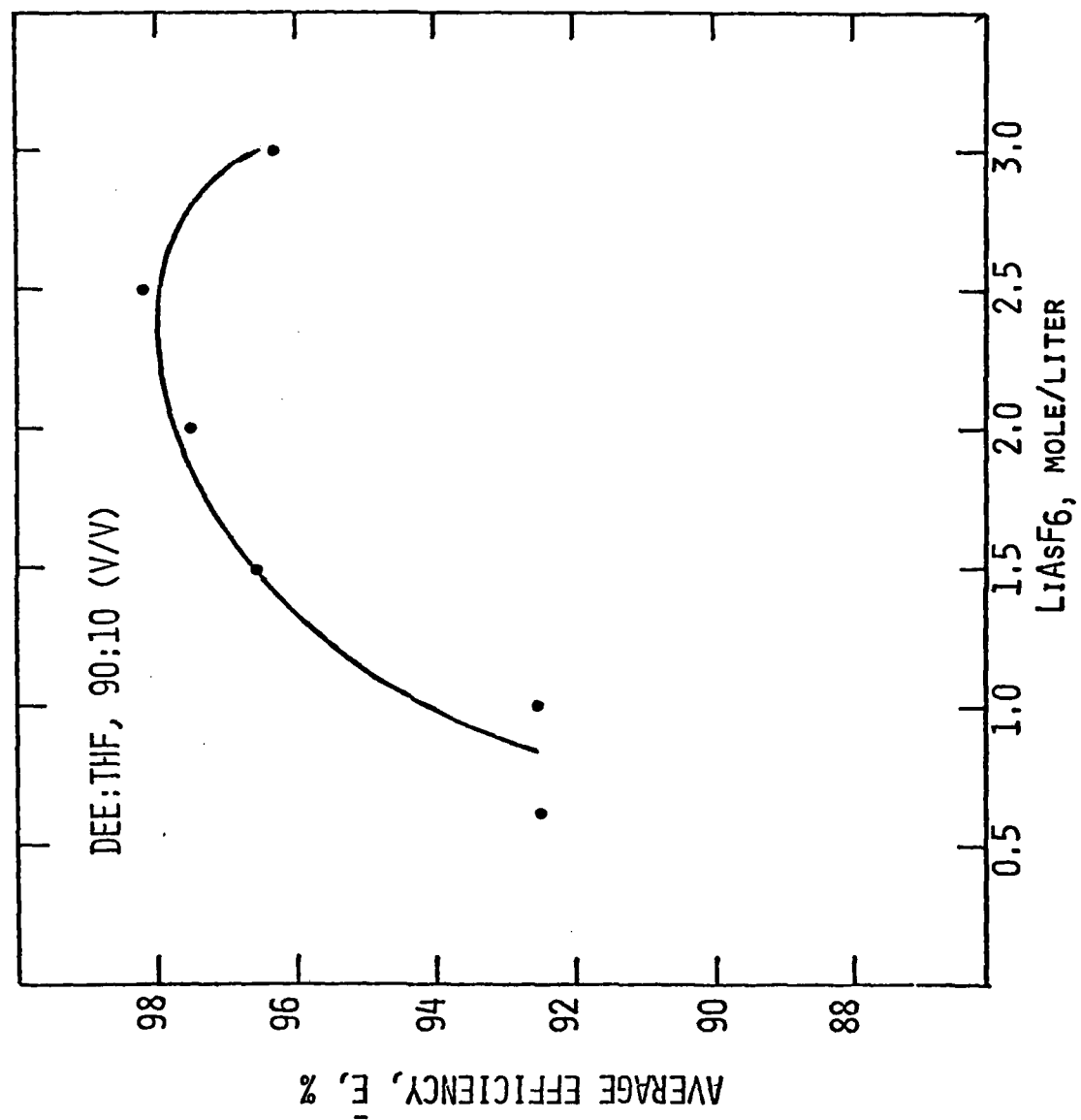


Figure 3

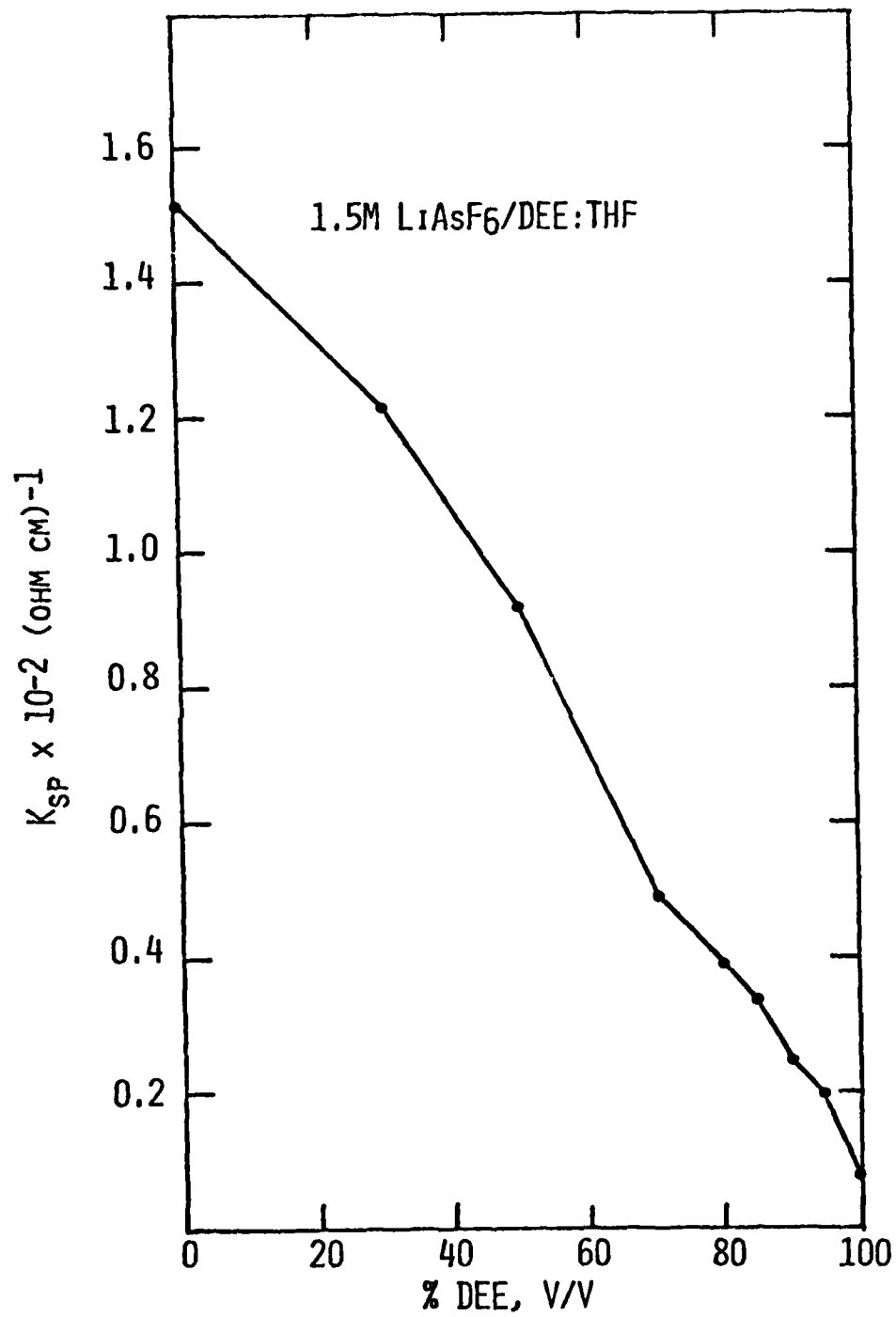


Figure 4

A



B



C

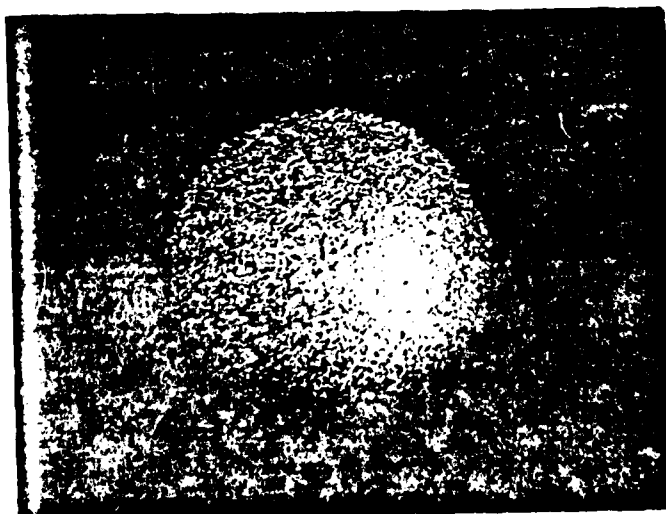


Figure 5

TABLE 1
CYCLING RESULTS FOR 2.5M LiAsF₆/B-90 ELECTROLYTE
ON VARIOUS SUBSTRATES

Substrate	Q_{ex} , coul/cm ²	Number of "100%" Cycles ^a	\bar{E} , %
Ni	3.4	135	97.8
Cu	3.4	126	97.6
Cu/Hg	3.4	233	98.7
Cu	16.9	433	96.5
Cu/Hg	16.9	1076	98.6

^a $Q_s = 1.1$ C/cm²; $i_p = i_s = 5$ mA/cm².

TABLE 2
CYCLING RESULTS FOR ELECTROLYTES COMPRISING
2.5M LiAsF₆/DEE: Cosolvent (90:10)

Cosolvent, 10% V/V	Number of "100%" Cycles ^a	\bar{E} , %
Me-O-Me	123	97.5
Et-O-Et	150	97.9
Ethylene Oxide	137	97.7
Me-O-Et	6	48.5
Me-O-nPr	6	48.5

^a $Q_{ex} = 3.4$ C/cm²; $Q_s = 1.1$ C/cm²; $i_p = i_s = 5$ mA/cm²

TABLE 3
OXIDATION AND IONIZATION POTENTIALS
OF SELECTED ETHERS

<u>Ether</u>	<u>E_p, V vs. $Ag^+/Ag \pm 0.04^a$</u>	<u>E_{100}, V vs. Li^+/Li^b</u>	<u>IP, eV $\pm 0.02^c$</u>
2-Me-THF	2.82	4.15	-
DEE	3.00	-	9.54
THF	3.05	4.25	9.45
DIOX	3.10	4.31	9.52
DME	3.39	-	9.67

^a[ether] = 5 mM in 1M $LiBF_4/CH_3CN$; $v = 100$ mV/sec.

^bPotential at which the current density reached $100 \mu A/cm^2$;
 $v = 100$ mV/sec; Ref. 3c.

^cRef. 14.

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