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THE USE OF THE REACTIVE ETHER, TETRAHYDROFURAN (THF) IN RECHARG--ETC(U)

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THE USE OF THE REACTIVE ETHER, TETRAHYDROFURAN (THF)  
IN RECHARGEABLE LITHIUM CELLS

by

K. M. Abraham and J. L. Goldman

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IN RECHARGEABLE LITHIUM CELLS

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SUMMARY

The usefulness of tetrahydrofuran (THF) as a cosolvent with 2-methyl-tetrahydrofuran (2Me-THF)/LiAsF<sub>6</sub> for improving the low temperature rate capability of rechargeable Li cells has been explored. Li/TiS<sub>2</sub> cells utilizing 2Me-THF/LiAsF<sub>6</sub> blended with 15-50 volume-percent THF have shown substantially improved rate characteristics at 0 and -10°C. In addition, the Li electrode in such cells has exhibited practically useful cycling efficiencies.

INTRODUCTION

Practical success with ambient temperature rechargeable Li cells largely depends upon the availability of suitable electrolytes for recharging the Li electrode. Considerable progress has been made recently [1-3]. There are at least two electrolyte systems in which the Li electrode can be recharged with high efficiency. These are the 2Me-THF/LiAsF<sub>6</sub> [4-6] and 1,3-dioxolane/LiClO<sub>4</sub> [7] solutions. Unfortunately, the 1,3-dioxolane/LiClO<sub>4</sub> system has shown a tendency to detonate upon impact and, therefore, has been abandoned [8]. The present electrolyte of choice is 2Me-THF/LiAsF<sub>6</sub> [9-11].



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Li cycles very well in 2Me-THF/LiAsF<sub>6</sub> (1.4M) [4-6], typical F.O.M.<sub>Li</sub>'s (F.O.M.<sub>Li</sub> = total accumulated cell capacity/theoretical Li capacity) being 30-40. However, the solution is impractical below 0°C [6], because of a tendency of the (2Me-THF)<sub>n</sub>LiAsF<sub>6</sub> solvate to crystallize, causing an increase in solution viscosity and a decrease in solution conductivity. A possible approach to improve the low temperature performance of 2Me-THF/LiAsF<sub>6</sub> would be to use a cosolvent (cosolvents) which normally by itself is reactive with Li, but otherwise provide electrolyte solutions with a number of attractive properties required for secondary Li cells. An example is tetrahydrofuran (THF). It forms highly conducting solutions with LiAsF<sub>6</sub>, the specific conductivity of a 1.5M LiAsF<sub>6</sub> solution at 25°C being  $1.60 \times 10^{-2} (\Omega\text{cm})^{-1}$ . This solution maintains good conductivity over the temperature range below room temperature down to -40°C. THF/LiAsF<sub>6</sub> solutions also exhibit relatively good thermal stability. However, THF is reported to react with Li and its solutions do not cycle Li well [12]. Despite this reactivity, when THF is used as a cosolvent with 2Me-THF/LiAsF<sub>6</sub>, the synergistic effects of the blended solution may help retard the kinetics of the Li/THF reaction. The data presented in this paper indicate practical utility for 2Me-THF/LiAsF<sub>6</sub> blended with 15-50 volume-percent THF.

#### EXPERIMENTAL

All experiments were conducted in the absence of air and moisture in a Vacuum-Atmospheres Corporation dry box equipped with a Model He-493 Dri-Train.

Purification of THF and 2Me-THF and preparation of LiAsF<sub>6</sub> solutions were carried out as described previously [5,6,12]. Hermetically sealed Li/TiS<sub>2</sub> cells having 0.68 Ah theoretical (1e<sup>-</sup>/TiS<sub>2</sub>) capacity were used as test vehicles for electrolyte evaluation. The cells were identical to those described previously [6]. The evaluation included extensive galvanostatic cycling at various Li charge densities, rate/capacity characterization at 25, 0 and -10°C, and storage experiments at 50°C. Data collection and retrieval were done with a Bascom-Turner Series 8000 Recorder equipped with microprocessor accessories. The cycling efficiency of the Li electrode was determined using the procedure described in [6].

Conductivity of solutions were measured in a Jones and Bollinger type conductivity cell. The cell was thermostated in a Tenney Environmental Chamber prior to conductivity measurements. The solution resistance was measured with a YSI Model-31 (Yellow Springs Instruments Co., Yellow Springs, OH) conductivity bridge.

The following solutions have been evaluated: 2Me-THF (85 v/o):THF(15 v/o)/LiAsF<sub>6</sub>(1.35M); 2Me-THF(70 v/o):THF(30 v/o)/LiAsF<sub>6</sub>(1.3M); 2Me-THF(50 v/o):THF(50 v/o)/LiAsF<sub>6</sub>(1.5M). These solutions are designated, Blend 85-THF, Blend 70-THF and Blend 50-THF respectively. The results are compared with those of cells containing 2Me-THF/LiAsF<sub>6</sub>(1.4M).

#### RESULTS AND DISCUSSION

Conductivity of Solutions: Figure 1 depicts the conductivities, measured at 25, 0 and -20°C, of THF blended 2Me-THF/LiAsF<sub>6</sub>(1.5M) solutions for several compositions (volume-percent (v/o)) of the two solvents.

The conductivity at all three temperatures decreases in a linear fashion on going from 100 v/o THF to 100 v/o 2Me-THF. In solutions with  $\leq 10$  v/o THF, the  $\text{LiAsF}_6$ -solvate crystallizes at  $-20^\circ\text{C}$ , resulting in considerably reduced conductivities. However, blends containing  $> 30$  v/o THF exhibit conductivities at  $-20^\circ\text{C}$  which are higher than the room temperature conductivity of 2Me-THF/ $\text{LiAsF}_6$ (1.5M). The implication of this for rechargeable Li cells is obvious.

The lower conductivity of 2Me-THF/ $\text{LiAsF}_6$ (1.5M) than that of THF/ $\text{LiAsF}_6$ (1.5M), despite the higher basicity and  $\text{Li}^+$ -complexing ability of 2Me-THF, has been attributed [5] to the 2-methyl substituent on the oxolane ring which perturbs the efficient packing of solvent molecules about  $\text{Li}^+$ , leading to an increased number of contact ion pairs. It appears that below  $0^\circ\text{C}$  the  $\text{LiAsF}_6 \cdot (\text{2Me-THF})_n$  complex has a rather low solubility in 2Me-THF. In a THF(15 v/o):2Me-THF(85 v/o)/ $\text{LiAsF}_6$ (1.5M) solution, the amount of THF is  $\sim 1.2$  moles per mole of  $\text{Li}^+$ . In this blend, probably a  $\text{LiAsF}_6 \cdot (\text{solvent})_n$  complex with at least one THF per  $\text{Li}^+$ , exhibiting higher solubilities in 2Me-THF at low temperatures, is formed. In blends with larger amounts of THF, complexes with more than one THF per  $\text{Li}^+$  may also be present.

Based on what we know about the reactivity of Li with THF/ $\text{LiAsF}_6$  solutions [12], it seems that blends with practical utility in rechargeable Li cells would be those containing 15-50 v/o THF.

Rate/Capacity Behavior of Li/ $\text{TiS}_2$  Cells: The significant effect expected with THF-blended 2Me-THF/ $\text{LiAsF}_6$  solutions, in comparison to 2Me-THF/ $\text{LiAsF}_6$ , is improved rate capability for cells, especially at low tem-

peratures. The data presented in Fig. 2 indicate that this, indeed, is possible. The 2Me-THF/LiAsF<sub>6</sub>(1.4M) cell exhibited practically no capacity at -10°C even at the low discharge rate of 0.5 mA/cm<sup>2</sup>. On the other hand, the Blend 50-THF cell, discharging with 2 mA/cm<sup>2</sup> at -10°C, exhibited a capacity equivalent to 70% of the theoretical cell capacity. The -10°C capacities of the Blend 30-THF and Blend 15-THF cells at 2 mA/cm<sup>2</sup> have been 50 and 16% of the theoretical cell capacity. These higher rate capabilities of cells utilizing the THF blends seem to correlate with the solution conductivities.

At room temperature, the cells with all of the solutions exhibited similar rate capabilities for each current density  $\leq 2$  mA/cm<sup>2</sup>. At higher current densities, however, the THF blends yielded slightly better utilization; the capacity being higher the higher the solution conductivity. The discharges versus current density for a Blend 30-THF cell at room temperature are shown in Fig. 3.

Li Cycling Efficiency: The cycling efficiencies of the Li electrode achieved in cells utilizing the various blends are summarized in Table 1. The data correspond to Li charge densities of 7 and 10.5 mAh/cm<sup>2</sup>. Typical cycling curves are given in Figs. 4 and 5. The cycling ability of the Li electrode is reduced by a significant fraction by the introduction of 15 v/o THF into 2Me-THF/LiAsF<sub>6</sub>. However, on going from blends with 15 to 50 v/o THF, the reduction in cycling efficiency owing to added THF does not follow a linear relationship. Thus, the Li cycling efficiency in both the Blend 50-THF and Blend 30-THF cells is practically the same. It appears



that the 2Me-THF/LiAsF<sub>6</sub> solution-properties, which enable high efficiency Li cycling in that solution dominate the solution properties of the THF/2Me-THF blends.

The F.O.M.Li's achieved in the blended solutions are in the range useful for practical considerations. Cells with attractive low temperature characteristics, although with some sacrifice in cycle life, would be of considerable practical interest.

Cell Storage at 50°C: A knowledge of the storage capability of the cells at elevated temperatures is essential for practical consideration of the solution. The data in Table 2 indicate little effect of storage at 50°C on the cyclability of the cells. The exceptional storageability shown by 2Me-THF/LiAsF<sub>6</sub> cells is in agreement with the good thermal stability of the electrolyte [5]. Prior studies [5] of the thermal stabilities of THF/-LiAsF<sub>6</sub> solutions in presence of Li at 70°C have shown it to be considerably less stable than 2Me-THF/LiAsF<sub>6</sub>. The present findings in cells with the THF blends indicate that either the degradation reactions involving THF are significantly suppressed at 50°C or that the favorable properties of 2Me-THF help retard the kinetics of these Li-THF reactions. Our prior studies [6] with 10 v/o THF blended diethyl ether (DEE)/LiAsF<sub>6</sub> solutions show that the Li cycling efficiency of cells stored at 50°C were markedly lower than that of fresh cells. We ascribed that to the thermal instability of DEE/LiAsF<sub>6</sub> as well as to the increased solubility in polar THF/DEE blends of the LiOC<sub>2</sub>H<sub>5</sub> film on the Li electrode surface. A chemically different Li surface film, probably with low solubility properties, is indicated in 2Me-THF/LiAsF<sub>6</sub> based solutions.

### CONCLUSIONS

The low temperature rate capabilities of rechargeable Li cells with 2Me-THF/LiAsF<sub>6</sub>(1.4M) can be improved by blending the solution with THF. Cells utilizing the blended solutions, despite the reactive THF component, cycle with practically useful Li electrode cycling efficiencies. This is believed to be due to a dominance of the desirable properties of 2Me-THF/-LiAsF<sub>6</sub> helping to retard the kinetics of the Li-THF reactions. Cells utilizing the blended solutions have also shown appreciable storage capabilities at 50°C, indicating practical utility for these solutions.

A general conclusion of the present study is that electrolytes based on mixed solvents at proper ratios may exhibit more desirable solution-properties than solutions based on the individual solvents. Our results also indicate that many of the so-called reactive solvents may be useful for improving the solution-properties of less reactive electrolytes.

### ACKNOWLEDGEMENT

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TABLE 1  
 Li CYCLING EFFICIENCY IN Li/TiS<sub>2</sub> CELLS WITH  
 2Me-THF/LiAsF<sub>6</sub> AND RELATED SOLUTIONS

<u>Electrolyte</u>	<u>Current Density (mA/cm<sup>2</sup>)</u>	<u>Li Charge Density (mAh/cm<sup>2</sup>)</u>	<u>Li Cycling Efficiency (F.O.M. Li)</u>
2Me-THF/LiAsF <sub>6</sub> (1.4M)	{ 1	7	40
	{ 1.5	10.5	32
THF/LiAsF <sub>6</sub> (1.5M)	1	7	3
Blend 15-THF	{ 1	7	23
	{ 1.5	10.5	20
Blend 30-THF	1	7	14
	1.5	10.5	13
Blend 50-THF	2	~6	14

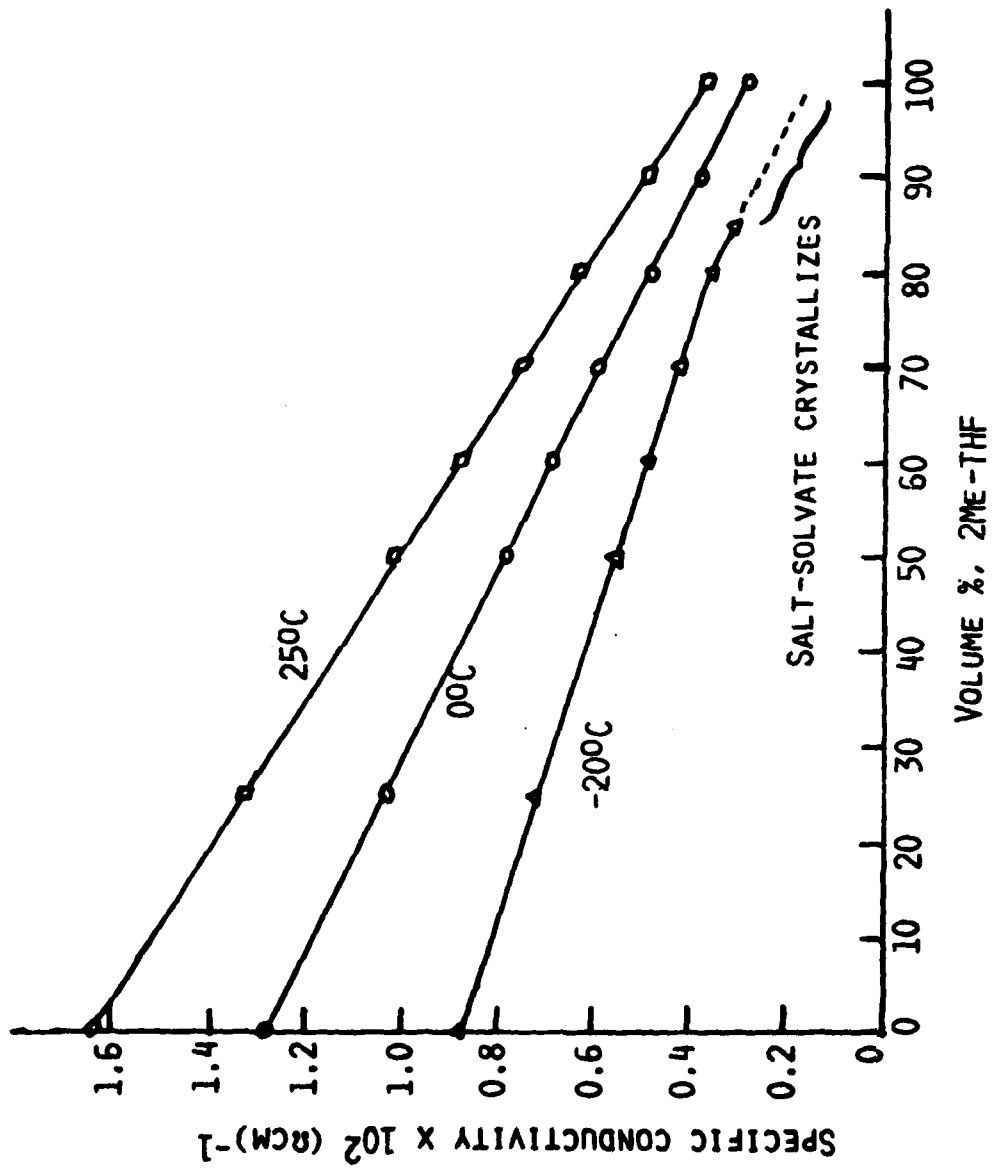
TABLE 2  
STORAGE TEST RESULTS OF Li/TiS<sub>2</sub> CELLS

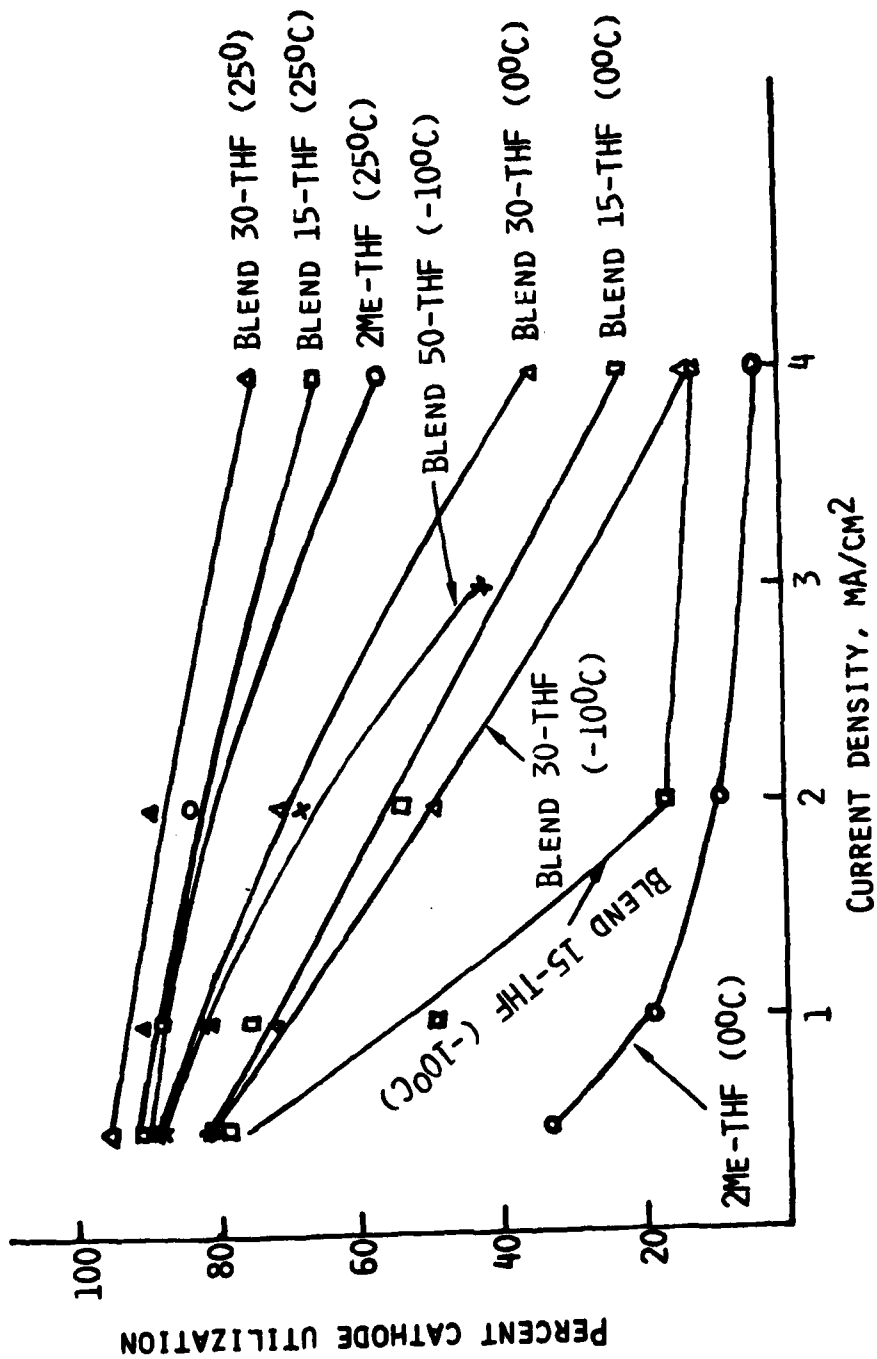
Electrolyte	Days Stored at 50°C	OCV		Capacity (e <sup>-</sup> /TiS <sub>2</sub> ) at 1 mA/cm <sup>2</sup>		Li Cycling Efficiency* (F.O.M.Li)
		Initial, V	Final, V	Typical Fresh Cell	After Storage	
2Me-THF/LiAsF <sub>6</sub> (1.4M)	33	2.4	2.4	0.90	0.90	35
Blend 15-THF	17	2.4	2.4	0.92	0.84	20
Blend 30-THF	17	2.4	2.4	0.95	0.90	13.5

\*Cycling efficiency obtained at 1 mA/cm<sup>2</sup> and 7 mAh/cm<sup>2</sup>.

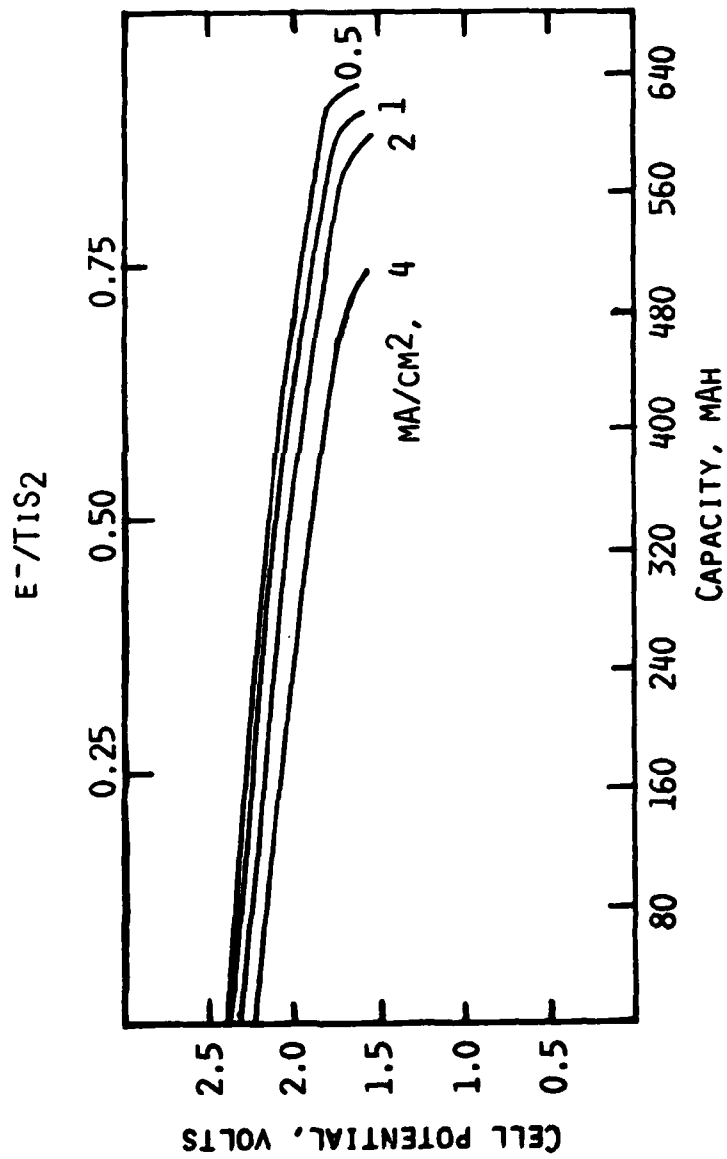
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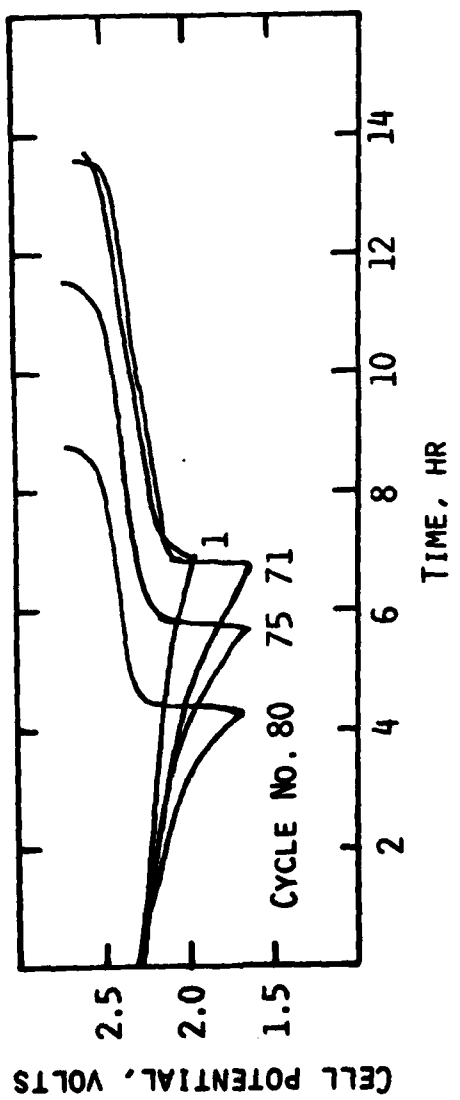
- Fig. 1. Specific conductivity vs volume percent of 2Me-THF in 2Me-THF/THF/LiAsF<sub>6</sub>(1.5M) electrolytes at various temperatures.
- Fig. 2. Rate/capacity data for Li/TiS<sub>2</sub> cells at 25, 0 and -10°C.
- Fig. 3. Cathode utilization vs current density in a Li/Blend 70-THF/TiS<sub>2</sub> cell at 25°C.
- Fig. 4. Typical cycling curves of a Li/Blend 85-THF/TiS<sub>2</sub> Cell. Current density, 1.5 mA/cm<sup>2</sup>. Li charge density, 10.5 mAh/cm<sup>2</sup>. Theoretical Li, 45.5 mAh/cm<sup>2</sup>.
- Fig. 5. Typical cycling curves of a Li/Blend 70-THF/TiS<sub>2</sub> cell. Current density, 1 mA/cm<sup>2</sup>. Li charge density, 7 mAh/cm<sup>2</sup>. Theoretical Li, 45.5 mAh/cm<sup>2</sup>.

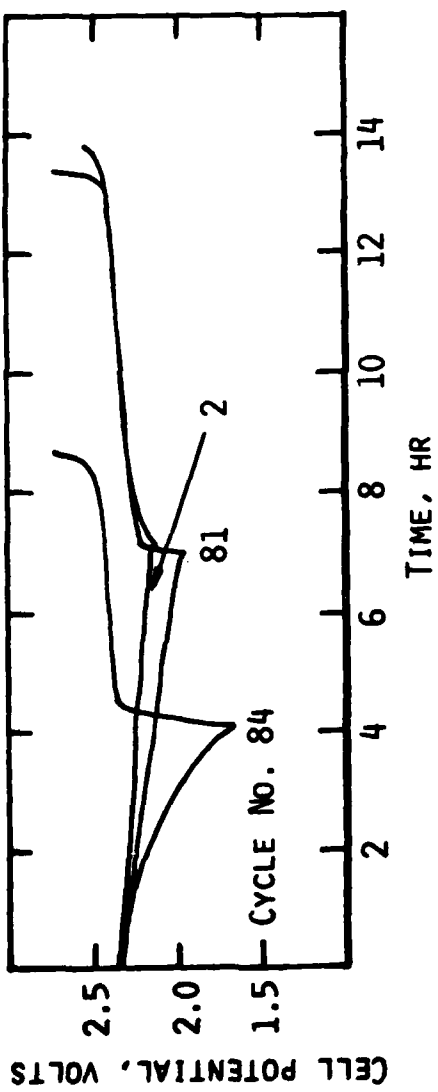












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