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LONG CYCLE LIFE SECONDARY LITHIUM CELLS UTILIZING
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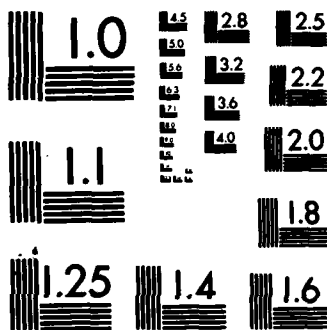
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TECHNICAL REPORT NO. 12

LONG CYCLE LIFE SECONDARY LITHIUM CELLS UTILIZING
TETRAHYDROFURAN

By

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Cycle lives of secondary Li cells utilizing THF/LiAsF ₆ solutions have been significantly improved by the use of small amounts of additives such as 2,methyl-furan, 2,methyl-thiophene or similar unsaturated organic compounds. A most important consequence of the discovery has turned out to be the practical utility of the mixed electrolyte, 50 v/o THF:50 v/o 2Me-THF/LiAsF ₆ (1.5M). This solution combined with ~3.5 v/o 2Me-furan has enabled 5 A-hr Li/TiS ₂ cells to exhibit more than 200 cycles at 60% depth-		

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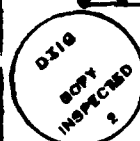
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of-discharge. This cycle life is twice that achieved with 2Me-THF/LiAsF₆(1.5M). In addition, the mixed electrolyte cells have shown low temperature discharge capabilities superior to those containing either THF/LiAsF₆(1.5M) or 2Me-THF/LiAsF₆(1.5M).

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LONG CYCLE LIFE SECONDARY LITHIUM CELLS
UTILIZING TETRAHYDROFURAN

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The tetrahydrofuran(THF)/LiAsF₆ electrolyte solution, except for its high reactivity with Li (1), is highly desirable for use in ambient temperature rechargeable Li cells (2). In a recent patent application (3), we have disclosed that the use of unsaturated organic additives such as 2-methyl-furan (2Me-F) enables high efficiency Li cycling in THF/LiAsF₆. A most important consequence of this discovery has turned out to be the utility of the high rate, low temperature mixed solution, 50:50 THF:2Me-THF/LiAsF₆(1.5M) (2,4,5), in 5 A-hr Li/TiS₂ cells to achieve, for the first time, more than 200 cycles of 60% depth-of-discharge (d.o.d). The Li anode cycling efficiency in these 5 A-hr cells exceeded 97%. We now believe that this mixed electrolyte is significantly better than 2Me-THF/LiAsF₆ for use in ambient temperature secondary Li cells. The relevant data are reported in this communication.

In prior publications (6-8), we have reported on the superior ability of the 2Me-THF/LiAsF₆ solution to cycle Li with high efficiencies which span the range of 96-97.5% (Figure of Merit, F.O.M._{Li} = 25-40) for Li charge densities of 7-10 mA-hr/cm². A persistent impurity in distilled 2Me-THF is 0.2-0.4 volume-percent (v/o) of 2Me-F (4,7,9), the unsaturated analog of 2Me-THF (see Table 2 for structural formulas). In fact, most of the published

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results discussing the use of 2Me-THF/LiAsF₆ in rechargeable Li cells have dealt with an electrolyte solution containing ~0.2-0.4 v/o 2Me-F. This has come about because of the difficulty of removing the 2Me-F by fractional distillation of the solvent.

Recently we have found that this 2Me-F can be completely converted into 2Me-THF by hydrogenating the "impure solvent" over a Pt catalyst at ambient temperature and pressure (3). Li/TiS₂ laboratory cells using the 2Me-F-free-2Me-THF/LiAsF₆ solution, to our surprise, have exhibited inferior cycle lives; typically, about half of that of cells utilizing the "regular electrolyte" with 0.2-0.4% 2Me-F. The cycle tests at 1 mA/cm² and 7 mA-hr/cm² were performed in 0.68 A-hr Li/TiS₂ laboratory test cells, identical to those we have previously described in ref. 7. Indeed, the cycle numbers for the various cells reported in Table 1 are directly comparable to those presented in Figures 4 and 5 of ref. 7. It should be noted that all cell capacities mentioned in this paper are based on 1e⁻/TiS₂. As discussed in ref. 7, the theoretical Li capacity in a cell is much larger than the TiS₂ capacity. Following the surprise with the 2Me-F-free-electrolyte, we deliberately prepared 2Me-F-containing solutions by adding 0.3, 0.6 or 1.2 v/o of 2Me-F into the solution in neat 2Me-THF, obtained via the hydrogenation procedure. Performance of laboratory Li/TiS₂ cells employing these solutions is summarized in Table 1. It is clear that 0.3 to 0.6 v/o of 2Me-F doubles the cycle life of 2Me-THF/LiAsF₆-based cells.

An obvious question is whether 2Me-THF/LiAsF₆ is less reactive in secondary Li cells than THF/LiAsF₆, as previously claimed (8). The answer

is yes, although about half of the cycles of the cell presented in Figure 5 of ref. 7 appears to be due to the effect of 2Me-F.

We have also found 2Me-F to improve substantially the cycle life of cells utilizing THF/LiAsF₆. Some relevant data are given in Table 1. Cells utilizing THF/LiAsF₆ plus 0.5 v/o 2Me-F exhibit about 100 cycles as opposed to 7 in 2Me-F-free cells. Interestingly enough, a significant increase in cycles is not seen at the higher 2Me-F concentrations of 1, 1.5 and 2 v/o. It appears from the data obtained from both THF and 2Me-THF cells, that there is a stoichiometric relationship between the amount of effective 2Me-F and the Li being electrochemically cycled. Our present data indicate that about 0.4 mmoles of 2Me-F is required per 1 A-hr of Li for the manifestation of an optimum additive effect in THF/LiAsF₆. This Li-to-additive mole ratio has been satisfactorily tested in 5 A-hr prismatic Li/TiS₂ cells (vide-infra). The practical significance of this discovery is very clear. For the first time, high rate, low temperature discharge and long cycle life have all been achieved in the same electrolytes, namely THF/LiAsF₆ and its blends.

We believe that 2Me-F is effective as a cycle life enhancing additive because it forms a protective film on the Li surface. The film may be of the solid electrolyte (10) or of the polymer electrolyte (11) type, enabling Li discharge and charge, while preventing or significantly slowing down direct chemical reactions at the Li surface. Further studies to fully elucidate the chemistry of the additive effect are in progress.

In laboratory test cells, the 50:50 THF:2Me-THF/LiAsF₆(1.5M) electrolyte solution with 0.5 v/o 2Me-F has given cycle lifes mid-way between

those given by cells containing the parent solutions plus the additive (see Table 1). However, in 5 A-hr prismatic Li/TiS₂ cells this order of additive effects changes. In these larger cells, employing a practical Li-to-electrolyte ratio, the mixed THF:2Me-THF solution plus 2Me-F has produced the longest cycle life. Thus a 5 A-hr Li/TiS₂ cell (12) utilizing a solution of the composition, THF (48.3 v/o):2Me-THF(48.3 v/o):2Me-F(3.4 v/o)/LiAsF₆ (1.5M), has given more than 225 cycles when cycled at a d.o.d. of 60% (Figure 1). As indicated earlier, the amount of the additive required is determined by the total Li in the cell, and the larger additive concentration in these cells is due to the larger Li to electrolyte ratio. Under similar cycling regimes, 2Me-THF/LiAsF₆ cells gave 125 cycles and the THF/LiAsF₆ cells 120 cycles. In all cases, cell failure occurred due to increased impedance and dendrite shorting. The blended solution, in addition, has exhibited relatively good thermal stability (see Table 1) and its low temperature performance is superior to that of the parent solutions. A 5 A-hr Li/TiS₂ cell employing the blended solution has yielded ~2.5 Ahr at -20°C at 2mA/cm² (2,4,5). For the same temperature and current density, capacities were non-existent in the cells utilizing the parent solutions. The superior -20°C performance of the mixed solution over THF/LiAsF₆, despite the higher conductivity of the latter (2), may be related to the more desirable Li⁺-solvates in the blend, permitting better Li⁺ diffusivities and a consequently high rate capability. The 2Me-THF system is impractical below 0°C because of the formation of sparingly soluble Li⁺-solvates (2,7).

In exploring the field, we have discovered other additives as well suitable for improving the cycle life of THF/LiAsF₆-based secondary Li cells. Some examples are given in Table 2. To date 2Me-F appears to be the most effective. Detailed studies of the various additives are in progress.

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FIGURE CAPTION

Figure 1. Typical cycles of a 5 A-hr theoretical capacity Li/TiS₂ cell utilizing THF(48.3 v/o):2Me-THF(48.3 v/o):2Me-F(3.4 v/o)/LiAsF₆(1.5M). Current: Cycles 1 and 100, id = 0.5A, ic = 0.25A; Cycles 160 and 203, id = ic = 0.25A. All cycling was performed to a depth-of-discharge of 60% (3A-hr).

TABLE CAPTIONS

Table 1 Effect of 2-Methyl-furan (2Me-F) on the Cycle Life of Li/TiS₂ Laboratory Cells^(a)

Table 2 Effect of Several Additives^(a) on the Cycle Life of Li/TiS₂ Laboratory Cells Utilizing THF/LiAsF₆(1.5M)

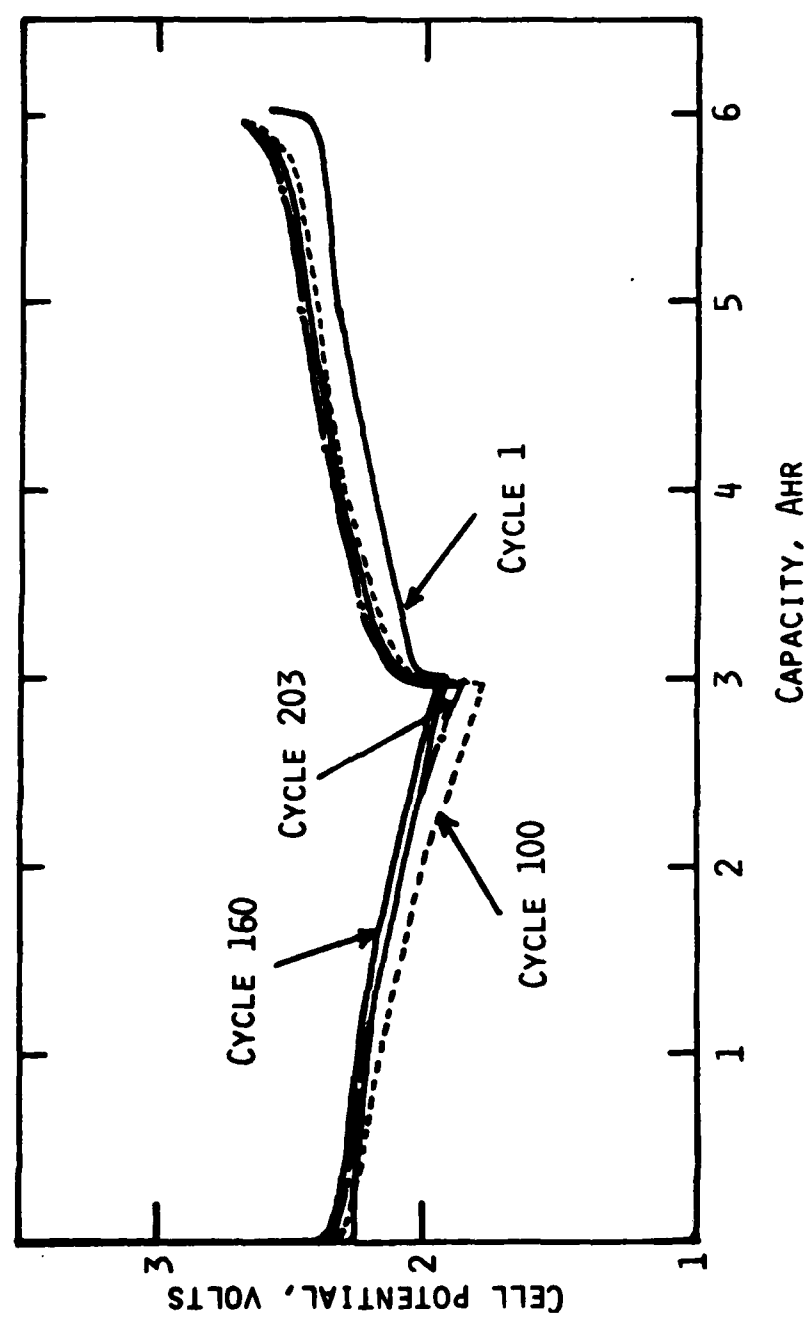


FIG. 1

Cell No.	Electrolyte	Amount of 2Me-F v/o	Number of Cycles ^(b)
98-18	2Me-THF ^(c) / LiAsF ₆ (1.4M) ↓	0.0	96
98-07		0.3	150
98-26		0.6	177
98-29		1.2	198
140-19	THF / LiAsF ₆ (1.5M) ↓	0.0	7
35-10		0.5	111
35-32 ^(d)		0.5	92
98-06		1.0	96
98-11		1.5	110
98-15		2.0	105
101	2Me-THF (50 v/o) :	0.5 ^(e)	145
	THF (50 v/o) / LiAsF ₆ (1.5M)		
102	2Me-THF (50 v/o) :	1.0	140
	THF (50 v/o) / LiAsF ₆ (1.5M)		

(a) Each type of cell has been cycled at least in duplicate. Temperature, 25°C.

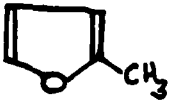
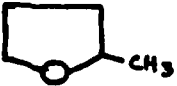
(b) Obtained at 1 mA/cm² for 7 mA-hr/cm²; compare Fig. 5 in Ref. 7.

(c) Obtained by hydrogenating distilled 2Me-THF containing 0.2-0.4 v/o 2Me-F over Pt catalyst and subsequently preparing the LiAsF₆ solution.

(d) Stored 14 days at 50°C prior to cycling.

(e) Stored for 1 month at 50°C prior to cycling. Unstored cells gave ~10 cycles less.

Table 1

<u>Additive(c)</u>		<u>Percent by Volume (v/o)</u>	<u>Number of Cycles(b)</u>
None	-	-	7
Furan,		0.5	46
2-Methyl-furan,		0.5	110
2,5-dimethyl-furan,		0.5	37
2,5-dimethyl-thiophene,		0.5	38
4,5-dihydropyran,		0.5	32
3,4-dihydrofuran,		0.5	28
2-methyl-THF,		0.5	8

(a) Additional additives are given in our patent (3).

(b) Obtained at 7 mA-hr/cm² and 1 mA/cm² (7).

(c) It appears that at least one double bond in the ring is necessary.

Table 2

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