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AROMATIC IONS AS ANODE MATERIALS FOR RECHARGEABLE, AMBIENT-TEMPERATURE BATTERIES.

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BY STANLEY D. JAMES

RESEARCH AND TECHNOLOGY DEPARTMENT

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Two approaches are recommended to containing the aromatics close to a current-collector for extended periods of charge-discharge cycling.

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SUMMARY

The silver-zinc battery, presently the workhorse, high rate secondary battery for naval purposes has a low cycle-life which limits its usefulness. The present work proposes using aromatic ions as anode material in a rechargeable version of the very promising lithium battery. Promising preliminary data are described and the direction of further development is outlined. The work leading to this report was funded by Independent Research Task Area #ZR00130101.

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AROMATIC IONS AS ANODE MATERIALS FOR RECHARGEABLE,
AMBIENT-TEMPERATURE BATTERIESINTRODUCTION

The silver-zinc battery is presently the workhorse high rate secondary battery for Navy uses. It's used for torpedo and DSRV propulsion and in silent running of submarines. It has, however, problems of poor cycle-life, short wet-stand life and the use of an expensive and critical material (silver). The present work seeks to develop a modification of the very promising Li cells⁽¹⁾ as a substitute for silver-zinc batteries. Present Li cells are purely primary and all efforts to make them secondary have not succeeded due to the formation of insulated Li dendrites on recharge.⁽²⁾ This problem appears to be a basic one for the following reasons. Lithium is thermodynamically unstable in all solvents found useful as battery electrolytes and survives in them only because it gets filmed with a thin protective layer of poorly conductive reaction product.⁽¹⁾ Deposition of Li onto this filmed substrate then occurs preferentially at holes or active sites in the insulating film leading to a dendritic type of growth. The Li dendrites have only the most tenuous connection, both mechanical and electrical, to their substrate and themselves in turn become filmed with electrolyte reaction product. This dendritic deposit crumbles very easily. The result is that often only about 50% of the deposited Li can be reoxidized anodically and a cycled Li electrode is soon converted to a useless sludge of inactive material.

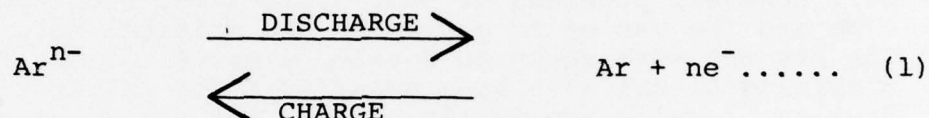
Because of this basic conflict between the need of a protective film on the Li and the deleterious effect of this film

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1. A. N. Dey, "Li Anode Films in Organic and Inorganic Electrolyte Batteries," Thin Solid Films, 43, 131-171 (1977).
 2. J. O. Besenhard "The Effect of Iodide on Li Cycling in Propylene Carbonate," J Electroanal. Chem., 78, 189 (1977).

on Li cycling, we are looking into a non-metallic substitute for Li for a secondary battery. We are studying the use of aromatic radical ions (Ar^{n-}) for this purpose. The idea of using Ar^{n-} as a battery anode occurred to me after a colleague (Isaac Angres) pointed out the ease with which alkali metals donate electrons to unsaturated aromatics. He had earlier cooperated in a study utilizing one such reaction⁽³⁾. In solvents like tetrahydrofuran (THF), alkali metals react with dissolved⁽⁴⁾ unsaturated aromatics (Ar) to give the aromatic ion (Ar^{n-}),



Usually n is one but it can be two or even more. The aromatic anions themselves have reducing power comparable to that of Li and thus are candidate anode materials as follows:



If both Ar and Ar^{n-} are soluble in the battery electrolyte there can be no dendrite problem. Furthermore, a soluble anode material avoids the passivation and consequent voltage-delay which is a bane of the filmed Li anode⁽¹⁾.

A relevant question is whether reaction (1) proceeds fast enough to be useful in a battery providing practical currents. It certainly does at a mercury current-collector since Ar undergoes electron transfer at diffusion-controlled rates at the DME⁽⁵⁾.

The aim of this preliminary study was two fold: firstly, to find whether the high charge-transfer rates observed near the Li potential at Hg can be reproduced at a practical current-collector

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3. W. P. Kilroy and I. Angres, "Determination of Free Li in Li-B Alloys," NSWC/WOL TR 77-129, 10 Nov 1977. Submitted to J. Less Common Metals.
 4. J. F. Garst, Accounts of Chemical Research, 4, 400 (1971).
 5. M. E. Peover, "Electrochemistry of Aromatic Hydrocarbons and Related Substances," in "Electroanalytical Chemistry," A. J. Bard, ed., Volume 2, Marcel Dekker, New York, 1967.

like steel, nickel or carbon. Secondly, whether useful coulombic capacities can be packed into such an electrode. We began with the aromatic, naphthalene (Nap) dissolved in THF which was made conductive with lithium tetrafluoroborate.

EXPERIMENTAL

Feltmetal (FM) Test Electrode

As a porous conductive substrate to both contain the naphthalene solution and collect current from the Nap + ne⁻ ↔ Napⁿ⁻ electrode reaction we used nickel Feltmetal. Feltmetals are conductive mats made by sintering together controlled aggregates of metal fibers. We used a 0.081 inch thick sheet of Ni Feltmetal, FM 1209 from the Brunswick Corp., Milford, Conn. This material has an average pore size of 91 μm (ranging from 45-182 μm) and a ratio of real to geometric area of about 73. Its porosity is 0.86 from which we may calculate theoretical volumetric capacity as follows. If the Feltmetal contains a solution 3.8M in Nap there is 0.0038 moles Nap/cm³ of solution or 0.86 x 0.0038 moles/cm³ of Feltmetal (FM). This corresponds to a capacity of 315 and 631 coulombs/cm³ of FM for a one and two electron change. So even as the divalent anion, such a naphthalene solution has only one twelfth the volumetric capacity of pure Li metal. However, a secondary Li electrode may never be feasible as explained above and other combinations of aromatic and solvent may give higher capacities than Nap/THF.

Materials

The naphthalene used in this work was Eastman Kodak's Recrystallized Grade, m.p. 80-91°C. It was dried by overnight evacuation. Tetrahydrofuran (THF), from Fisher Scientific, Cat. No. T-397, water content 0.01%, was dried over pellets of Linde Type X Molecular Sieve. Lithium tetrafluoroborate was secured from Foote Mineral Co., lot no. 101-1 and was dried to constant weight over Mg perchlorate. To assist in selecting appropriate solutions we characterized the conductance of this salt versus concentration in THF at 25°C (6). Figure 1 shows its specific conductance versus molar concentration. To act as test electrode material we made up a solution of THF, 1.75M (23.2% w/w) in Nap and 0.55M in fluoroborate - solution N. This solution was made unsaturated in

-
6. S. D. James "Conductance of Lithium Tetrafluoroborate Solutions in Tetrahydrofuran," J. Chem. Eng. Data, 23, 313 (1978).

Nap to lessen the likelihood of the FM fibers being coated with an insulating film of Nap during the electrode discharge reaction.

Design of Test Electrode and Cell

Figure 2 shows the test electrode design. The electrode holder is a stainless steel disc (2x0.125") with a 0.97x0.072" hole inset into its center to receive the Feltmetal. A Feltmetal disc was scissored from the 0.081" thick, FM 1209 sheet to just slip into the electrode holder, protruding about 0.01". Two layers of non-woven polypropylene cloth totalling a thickness of 0.016" cover the FM disc to retard loss of its Nap by diffusion or convection into the bulk electrolyte. The steel washer presses 16 mesh SS screen down onto the FM to maintain its electrical contact to the electrode holder. Washer and electrode holder are clamped tightly together with two, alligator-type paper clips (handles removed).

The electrochemical test cell is made of Pyrex glass and contained 100 cm³ of electrolyte under slowly flowing, dried argon gas presaturated with electrolyte vapor. The test electrode was clamped horizontally. The counter electrode, 0.060" thick Li foil pressed into a 9 cm² washer made of Ni Exmet, was clamped about 10mm above the test electrode. Pure Li, plastered over the tip of a 3mm steel (SS) rod, hung midway between test and counter and served as reference electrode. The bulk electrolyte (outside the FM) was THF, 1.42 M in Li fluoroborate. It contained no Nap because this would react with the Li of both counter and reference electrodes giving Li naphthalenide, the active material of the test electrode. A significant and unknown amount of electrode reaction would then occur of the Nap/Napⁿ⁻ couple in the bulk electrolyte at the extensive steel surface of the test-electrode support structure. This would confuse the evaluation of the coulombic efficiency of material within the Feltmetal. We used pure Li on the counter electrode because its electrode reactions, $\text{Li} \rightleftharpoons \text{Li}^+ + \text{e}^-$, do not change or contaminate the electrolyte as those of another metal would. There was sufficient mass of Li in the counter (0.81g) to outlast about 80, 140--coulomb cycles even if none of the Li plating on it were reoxidizable on current-reversal. Charge-discharge curves were obtained using a constant current, regulated power supply and high-impedance strip-chart recorder.

Procedure

The Feltmetal test electrode was prepared in a dry room (2-3% relative humidity) by dripping solution N from a graduated pipette onto the FM disc (sitting in its electrode holder) till excess liquid was just visible. Both added volume and increase in weight of the electrode assembly were recorded. This was

done in a vessel saturated with THF vapor to minimize solvent evaporation from the FM. We found by prior tests that the FM wicked THF efficiently into its pores in amounts both reproducible and consonant with the FM's free volume. After covering the FM disc with the PP cloth soaked with bulk electrolyte, the test electrode was quickly assembled as in Figure 2. It was then immersed, along with the other electrodes in the Nap-free electrolyte in the Pyrex cell. Electrolysis was then begun as soon as possible to minimize prior loss of Nap by diffusion out of the FM into the bulk electrolyte. Our aim was to charge up the Nap electrode by cathodizing it at constant current reducing Nap to Nap^{n-} then reverse the current and characterize the anion's functioning as an anode material.

RESULTS

We measured the solubility of naphthalene in THF at 24.6°C as $3.78 \pm 0.02\text{M}$ and the density of the saturated solution as $0.948 \text{ g}\cdot\text{cm}^{-3}$. The solubility rises with temperature. We did a "quick and dirty" electrolysis in Nap-saturated THF, made conductive with LiClO_4 , to see whether high currents could be obtained at a steel electrode. Current was passed between two SS rods in a test-tube from which air was not rigorously excluded. The current level corresponded to $20\text{mA}\cdot\text{cm}^{-2}$ at the cathode. During passage of current, green solution streamed upward from the cathode and brown steel corrosion products fell from the anode. The catholyte, colored green by the naphthalenide ion, was quickly decolorized by air oxidation after current-interruption. Later tests showed that Nap^{n-} was rapidly oxidized also by the perchlorate ion and we switched to the tetrafluoroborate salt. In summary these preliminary experiments showed us that Nap reduction proceeds at high rates at a practically useful electrode material and guided our choice of Li tetrafluoroborate as supporting electrolyte.

Figure 3 shows first-cycle, charge-discharge curves for the Nap-Feltmetal test electrode. In this run the FM disc contained 0.173g of Nap in 0.748g of solution N, corresponding to a theoretical ($n=1$) coulombic capacity of 130.5 coul. The open-circuit voltage of the Feltmetal was about +3.1V vs the Li reference electrode. Switching on cathodic current at time-zero pushed test electrode voltage down fast toward the Li value but it arrested at +0.4V where $\text{Nap} + e^- \rightarrow \text{Nap}^-$ proceeded to the extent of 18.3 coul. Voltage then fell further to a plateau at the Li potential and the current was switched off after an additional 31.0 coul corresponding to Li deposition. A white layer of plated Li was seen on the test electrode structure. After a 24 min wait at open-circuit we reversed the current at a lower level (4.3 mA per geometric cm^2 of FM). Two voltage-arrests were obtained corresponding to the reverse process: 9.6 coul for Li oxidation

and 2.4 coul for naphthalenide oxidation. From Figure 3 we see firstly that Nap^- was cathodically generated from Nap to the extent of 18.3 in 130.5 coul which is only 14.0% efficient. Secondly, of this 18.3 coul-equivalent of Nap^- , only 2.4 coul was reoxidized on reversing the current, i.e., 13.1%.

Subsequent charge-discharge cycles were even more disappointing; arrests attributable to Nap/Nap^- got shorter and shorter. Naphthalene was obviously escaping rapidly from the FM by a combination of diffusion, convection and electrolysis. In the 42 min that had elapsed between covering the test electrode with Nap-free electrolyte and beginning the first charge of Figure 3, most of the Nap had diffused or convected out of the FM and was lost to the test electrode. Furthermore, electrolysis drives more Nap^- out of the FM cathodically than is returned anodically. So the present test-electrode design has serious defects. The cloth-wrapped FM pad was unable to hold Nap/Nap^- long enough to demonstrate its cycling capability.

Incidentally, Figure 3 also strikingly illustrates how hard it is to efficiently reoxidize electroplated Li since only 9.6 in 31 coul or 31% of the cathodically plated Li was anodically stripped off. This of course is the basic defect of lithium as a secondary electrode, the problem that spawned the present study.

CONCLUSIONS AND RECOMMENDATIONS

1. These preliminary experiments were promising in that high current-densities were obtained at practical current-collectors (steel and nickel). Arrests in the neighborhood of the Li potential corresponding to $\text{Nap} + e^- \rightleftharpoons \text{Nap}^-$ were seen in charge-discharge curves.

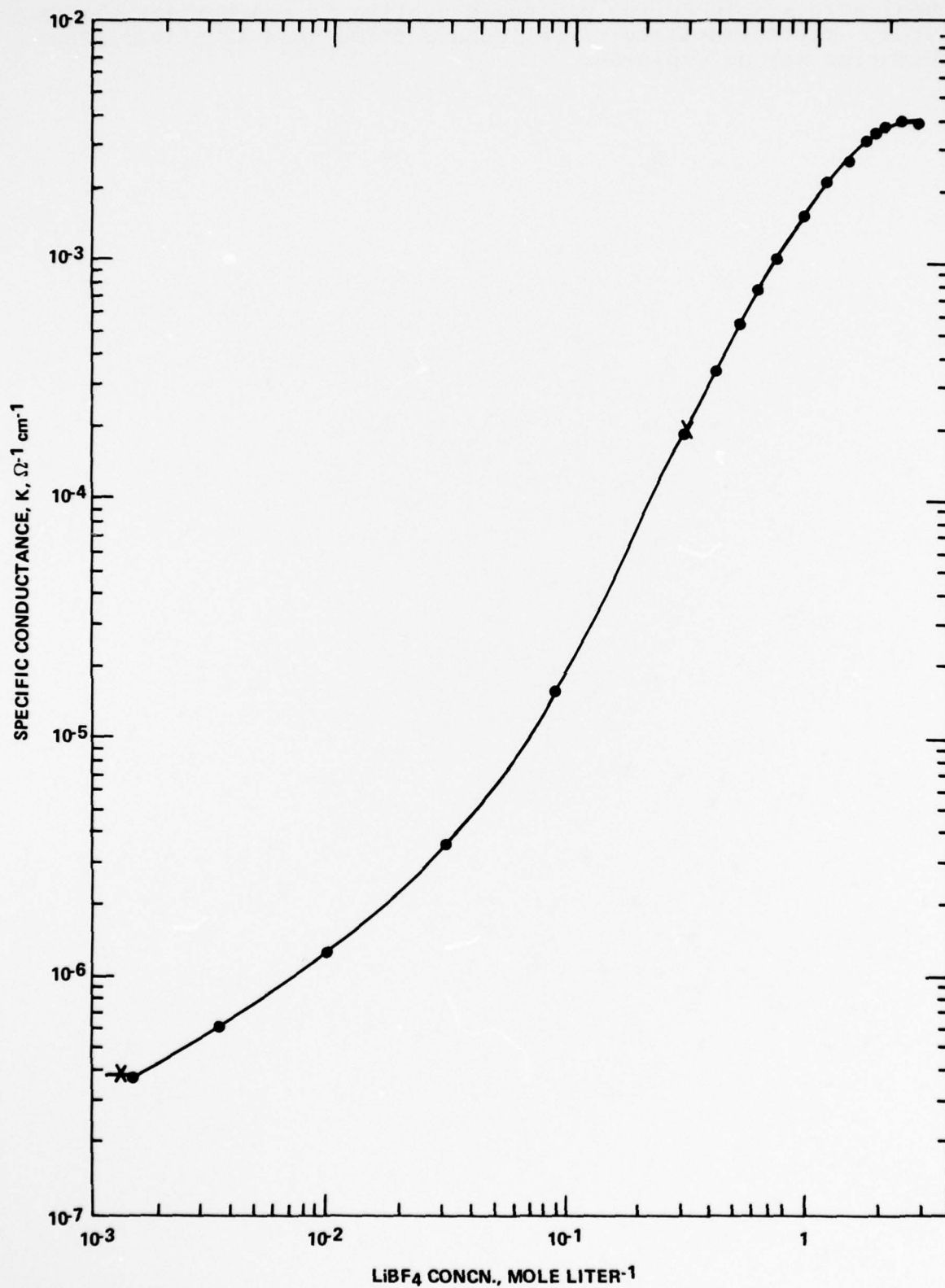
2. However, the test electrode design did not confine naphthalene long enough to demonstrate rechargeability over a number of cycles. It escaped rapidly by a combination of diffusion, convection and electrolysis.

3. We recommend two approaches to containing aromatics close to a current-collector for extended periods of charge-discharge cycling.

A. Find a combination of Ar/Ar^{n-} and electrolyte such that both Ar and Ar^{n-} remain as insoluble solids and may thus be localized as pastes on a metallic grid. One idea along these lines is the use of polymerized aromatics $(\text{Ar})_n$ which will tend to be insoluble solids as the degree of polymerization is raised.

B. Search for a separator film permeable to electrolyte (and thus conductive) but impermeable to soluble aromatics. This film will then confine the aromatics near their current collector. In a battery such a film would prevent self-discharge due to mingling of the anode and cathode materials. The film must be

chemically stable during prolonged cycling in appropriate electrolytes. The suitability of cellophane films used in silver-zinc batteries may be explored.

FIGURE 1 SPECIFIC CONDUCTANCE VERSUS CONCENTRATION FOR LiBF₄ IN THF, 25°C

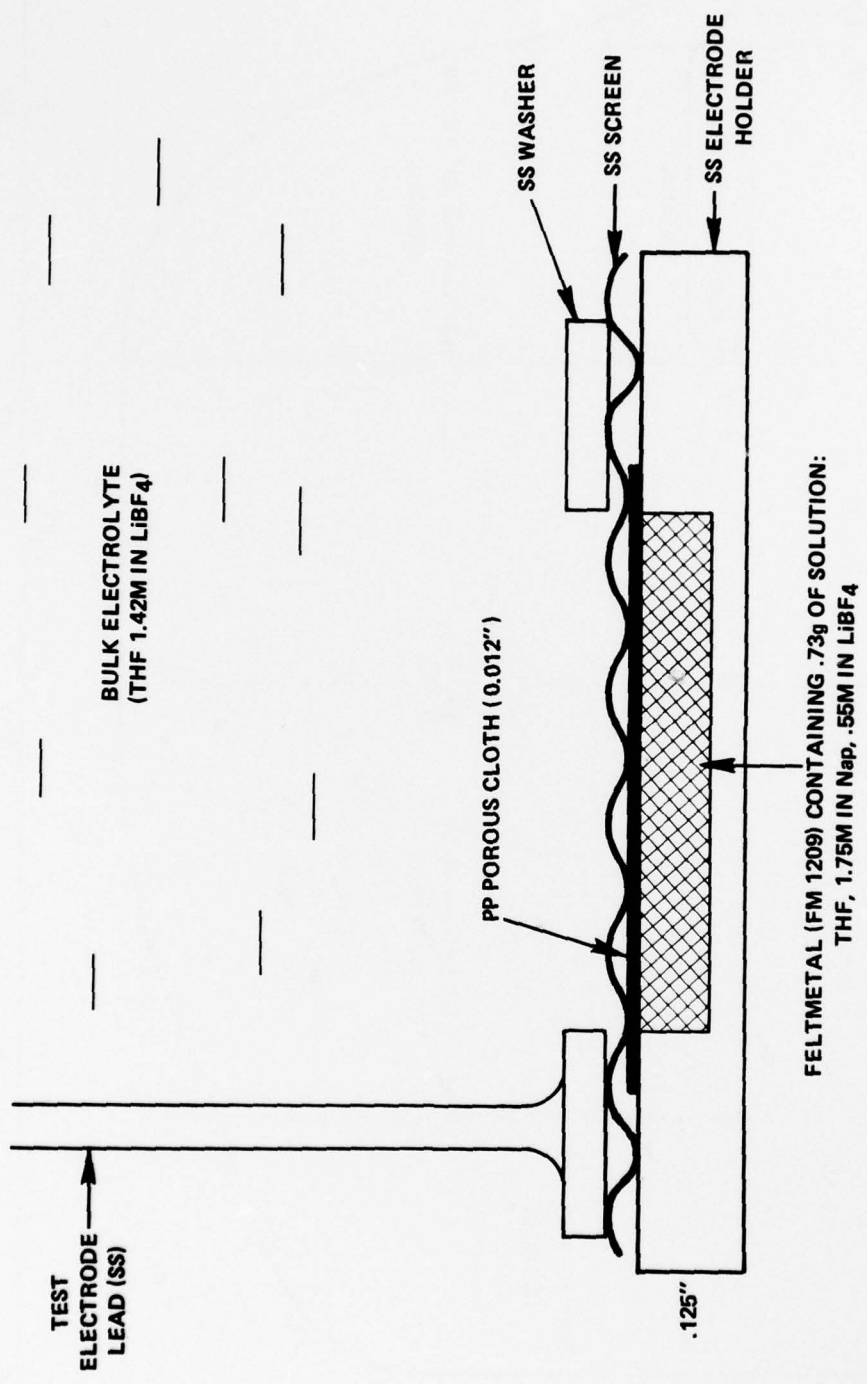


FIGURE 2 NAPHTHALENE TEST ELECTRODE

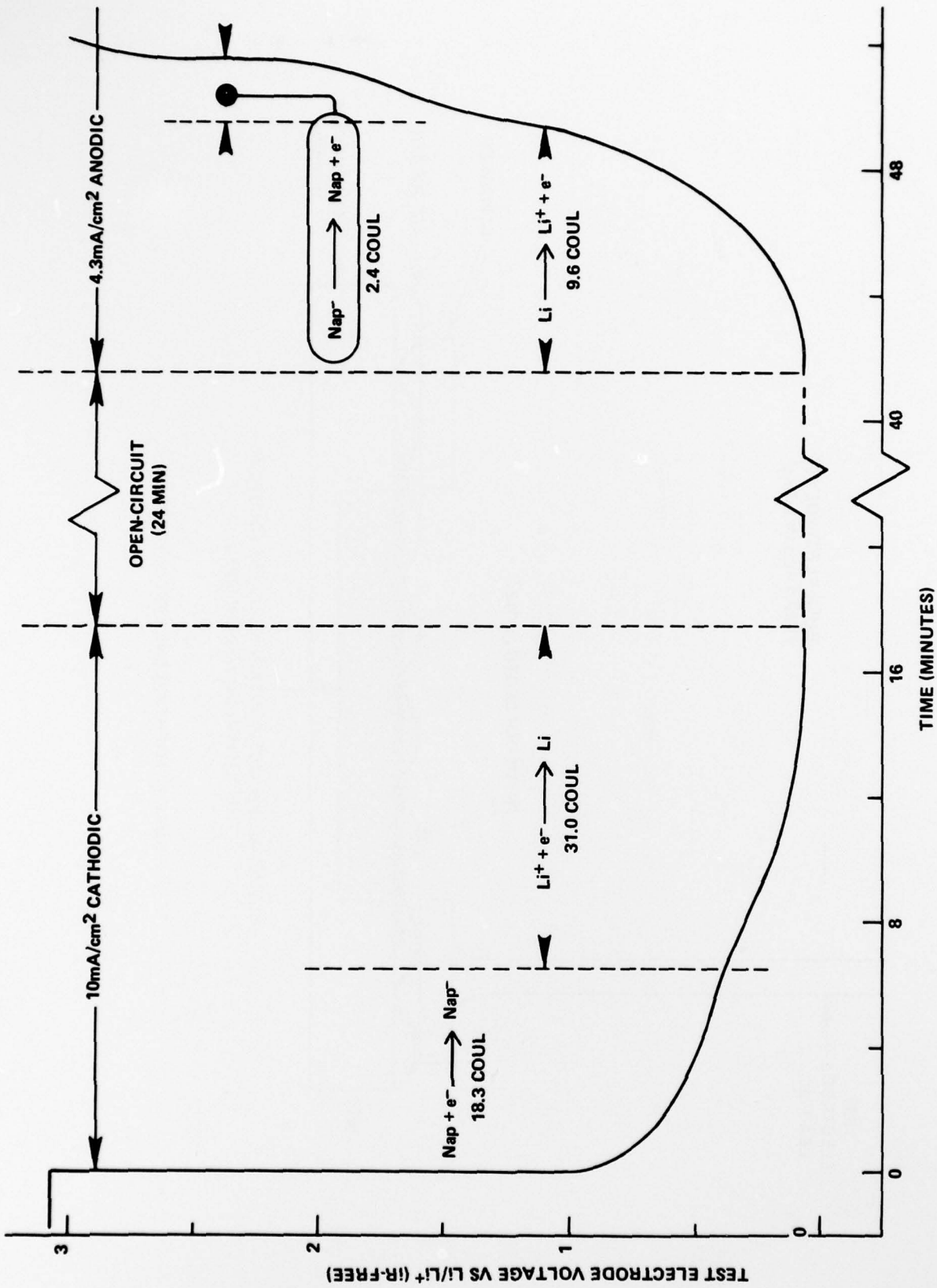


FIGURE 3 FIRST-CYCLE, CHARGE-DISCHARGE CURVE FOR NAPHTHALENE ELECTRODE AT 28°C.
 BULK ELECTROLYTE: 1.42M LiBF₄ IN THF. THEORETICAL CAPACITY: 130.5 COUL (Nap/Nap⁻)

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