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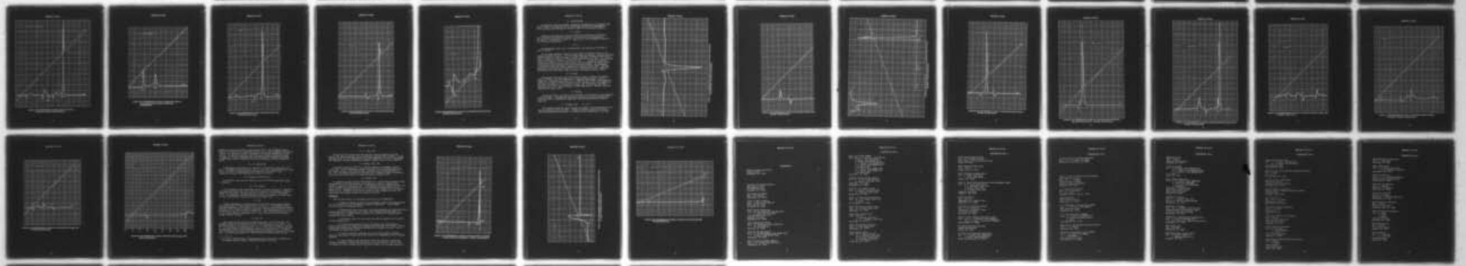

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THERMAL ANALYSIS OF SOME COMPONENTS OF THE Li-So$_2$ CELL

BY WILLIAM P. KILROY STEVEN DALLEK

RESEARCH AND TECHNOLOGY DEPARTMENT

13 NOVEMBER 1978

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Safety problems associated with heat generation in the Li/SO_2 system were investigated using differential scanning calorimetry. Various binary mixtures of cell components were found to undergo explosive combustion. Ignition temperatures and cell temperature increases were determined. Passivation of some exothermic reactions were observed on addition of other cell components to some binary mixtures.

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SUMMARY

The $Li-SO_2$ battery is currently being considered by the Navy for future use in mines. However, this battery has experienced safety problems where cells have exploded, vented toxic fumes, and have caught fire.

This report represents preliminary studies on the thermal compatibility of the lithium anode with various cell components and some of the expected discharge products.

The work leading to this report was jointly supported by the Naval Sea Systems Command under Task Area Number 202 and by Independent Research Task Area Number ZR00130101.

SG Fishman
(Acting for)
J. R. DIXON
By direction

PREFACE

We wish to thank Mr. R. Walk and Mr. W. Ebner of the Honeywell Power Sources Center for supplying us with the various components comprising the cathode collector.

CONTENTS

ILLUSTRATIONS

ILLUSTRATIONS (Cont.)

THERMAL ANALYSIS OF SOME COMPONENTS OF THE Li-SO_2 BATTERY SYSTEM

INTRODUCTION

Increases in the temperature of a battery during either storage or discharge can be expected to occur as a result of environmental, mechanical, electrical or chemical occurrences. Since safety problems associated with some lithium batteries are often accompanied by an increase in temperature, thermal studies on the chemical components is an important first step in understanding the safety question. The explosion hazard can be attributed to exothermic reactions that occur in a cell. These chemical reactions often require thermal energy for activation, and once activated can then either be violently exothermic or can create local "hot spots" that may trigger another reaction into producing a thermal runaway.

This report is a preliminary thermal investigation of the compatibility of various components of the Li-SO_2 battery in order to identify potentially explosive reactions and the temperatures at which these occur. Due to the numerous components in the Li-SO_2 system including choice of several solutes (LiBr, $LiAsF_6$, etc.) and solvents CH_3CN (AN) and propylene carbonate (PC) , the complexity of combinations was restricted to reactions involving mixtures of lithium with initial components or with some expected products.

The experimental approach involved differential scanning calorimetry and thermogravimetric analysis.

EXPERIMENTAL

Apparatus. Differential scanning calorimetry (DSC) was employed to measure enthalpy changes during controlled heating of known masses of elemental lithium with either single or multiple components of the Li-SO_2 system. DSC measurements were obtained between 25°C and 350°C at a programmed rate, using a calibrated DuPont Model 990 DSC instrument in which a flowing atmosphere of dry argon was maintained.

One inch on the ordinate corresponds to 2mv or approximately 50°C. Accurate sample temperatures were obtained from the millivolt output of the chromel-alumel thermocouple of the instrument.

Thermogravimetric analysis (TGA) using a DuPont Model 951 was employed to elucidate compositional changes resulting from mass loss on heating.

Cell Design. The selection of a sample container presented two immediate problems, the reactivity of lithium with the container and the confinement of volatile liquids.

Thermal studies involving lithium with solid components were performed by hermetically sealing the mixture in a DuPont type cell (Fig. 1A), specially fabricated from Armco iron.

In thermal analysis involving volatile liquids, a cell must be designed that is (a) tightly sealed so as to prevent the slow loss of gaseous products and (b) capable of withstanding the internal pressure that develops over the desired temperature range to be studied.

Slowly leaking containers result in spurious endotherms whereas pressure bursts occur when the cell container can no longer maintain internally generated pressures.

Two cell designs were employed to study the volatile liquid system.

Figure 1B shows the dimensions of the 95 microliter "welded" cell, consisting of a type 303 stainless steel cover and pan. Both parts were cleaned and then sealed by resistance welding, employing copper electrodes and approximately 17 joules of welding energy for each weld.

A Perkin Elmer "O-ring" 304 stainless steel cell (Fig. 1C) was also employed. It has a 75 microliter capacity with a viton rubber O-ring seal capable of containing a pressure limit of 24 atmospheres.

Reagents. Acetonitrile was triply distilled as previously reported(1) and stored over molecular sieves. Anhydrous 99.98% SO_2 was condensed under an argon atmosphere. Lithium hexafluoroarsenate, obtained from U. S. Steel, was used throughout. All solids were vacuum dried at the appropriate temperature. Webril non-woven polypropylene, Shawinigan black, and Teflon were obtained from Honeywell Power Sources.

Sample Preparation. All of the samples were weighed, mixed, and sealed in the DSC cells in a dry room maintained at 2% relative humidity. The solid samples were pressed and sandwiched in a single piece of 99.9% lithium (Foote Mineral Company).

Tetraacetonitrilolithium hexafluoroarsenate(2) was prepared by mixing 1:4 mole ratio of lithium hexafluoroarsenate and acetonitrile. An exothermic reaction occurred producing the tetrasolvated salt.

Solutions of SO_2 in AN were prepared by passing SO_2 gas over a weighed sample of AN, followed by cooling in an acetone-dry ice bath, weighing, equilibrating to room temperature and reweighing.

---

1. G. A. Forcier and J. W. Olver, "Simplified Purication of Acetonitrile for Electroanalytical Applications," Anal. Chem. 37 (11), 1447, 1965.

2. W. P. Kilroy, "Solubility and Solvate Formation fo Lithium Hexafluoroarsenate in Acetonitrile," Journal of Solution Chemistry, 6 (7), 487, 1977.

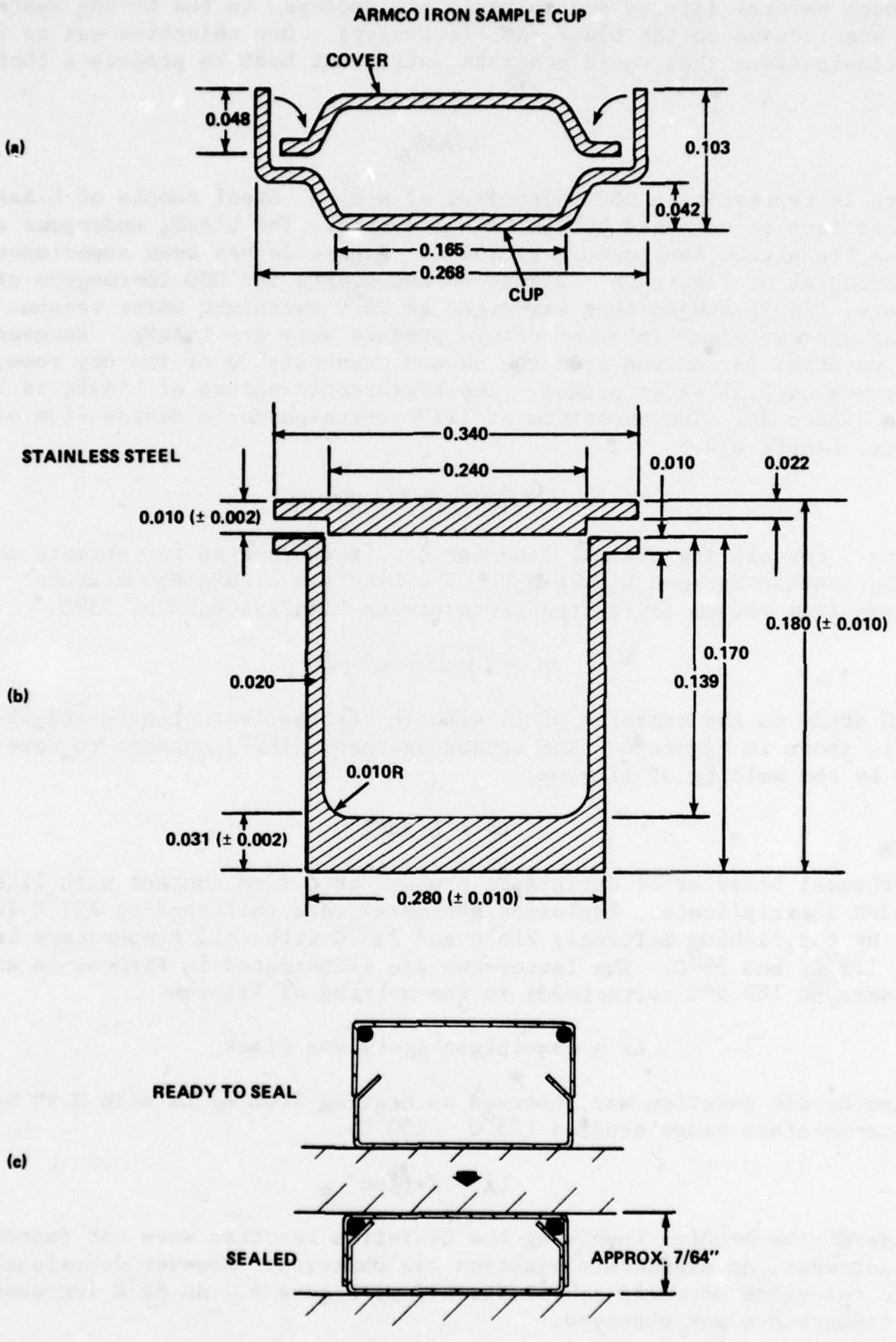

FIGURE 1 CELL DESIGNS EMPLOYED IN THE DSC STUDIES. (A) DUPONT TYPE, (B) WELDED TYPE, (C) PERKIN ELMER O-RING TYPE

OF RESULTS

Although several solutes and solvents are employed in the Li-SO_2 system, our attention was focused on the $LiAsF_6$-AN electrolyte. Our objective was to identify chemical combinations that could generate sufficient heat to produce a thermal runaway.

$LiAsF_6$

Figure 2a represents a DSC thermogram of a U. S. Steel sample of $LiAsF_6$ that was prepared from an unopened bottle in a dry room. The $LiAsF_6$ undergoes a reversible phase transition that occurs at 258°C. Figure 2a has been superimposed on the thermogram of Figure 2b. Figure 2b represents the DSC thermogram of the tetrasolvate, $LiAsF_6 \cdot 4CH_3CN$ that was dried at 25°C overnight under vacuum. Such drying was performed in an effort to produce very dry $LiAsF_6$. However, if the dried material is removed from the vacuum oven outside of the dry room, TGA analysis revealed 2.5% water pickup. The hygroscopic nature of $LiAsF_6$ is illustrated in Figure 2b. The endotherm at 117°C corresponds to dehydration of the monohydrate, $LiAsF_6 \cdot H_2O$.

$Li + LiAsF_6 + LiAsF_6 \cdot H_2O$

Figure 3 reveals the thermal behavior of lithium heated in intimate contact with $LiAsF_6$ containing some $LiAsF_6 \cdot H_2O$. The $LiAsF_6$ - $LiAsF_6 \cdot H_2O$ mixture was prepared from vacuum drying the tetrasolvate $LiAsF_6 \cdot 4CH_3CN$ at 25°C.

$Li + LiAsF_6 \cdot 4CH_3CN$

A DSC study on the reaction of Li with the tetrasolvate $LiAsF_6 \cdot 4CH_3CN$ (wet with AN) is shown in Figure 4. The second exotherm (180°) appears to have been initiated by the melting of lithium.

$Li + LiAsF_6$

The thermal behavior of dry $LiAsF_6$ pressed into firm contact with lithium was recorded in triplicate. Explosive exotherms were initiated at 197°C (apparently triggered by the lithium melting); 216°C and 236°C with cell temperature increases of 177°C, 179°C, and 36°C. The latter two are illustrated in Figures 5A and 5B. The endotherm at 180.5°C corresponds to the melting of lithium.

Li + Shawinigan Acetylene Black

No exothermic reaction was observed on heating 3.60 mg Li with 0.90 mg carbon over the temperature range studied (25°C - 250°C).

Li + Teflon

Exothermicity studies involving the Li-Teflon reaction were not reproducible. In some instances, no exothermic reaction was observed. However occasionally, exothermic reactions occurred as illustrated by Figure 6. An 87°C increase in the cell temperature was observed.

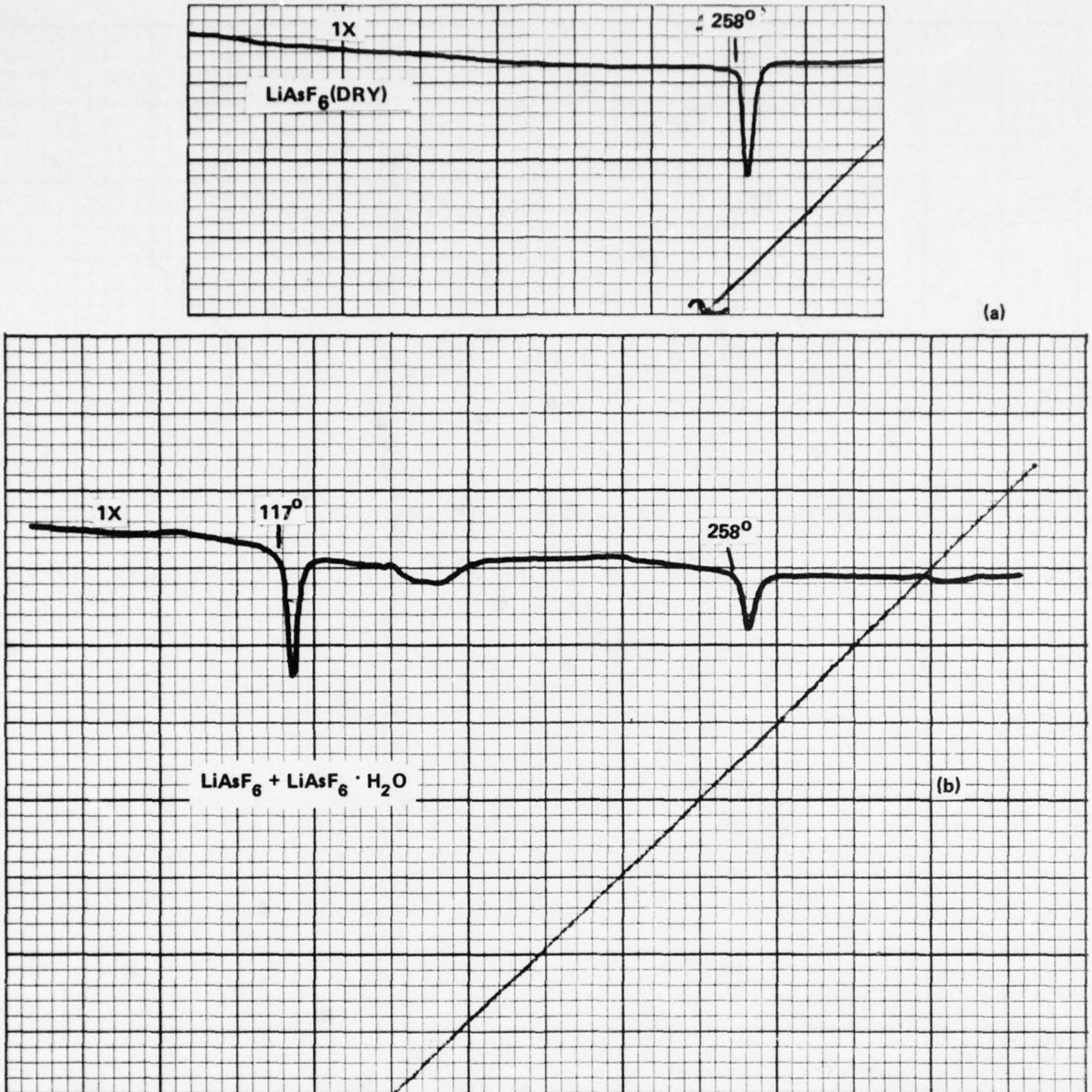

FIGURE 2 DSC THERMOGRAM OF (a) DRY $LiAsF_6$, (b) WET $LiAsF_6$ PREPARED FROM IMPROPER DRYING OF $LiAsF_6 \cdot 4CH_3CN$; 10°C/MIN; SENSITIVITY 1.0 MCAL/SEC/IN

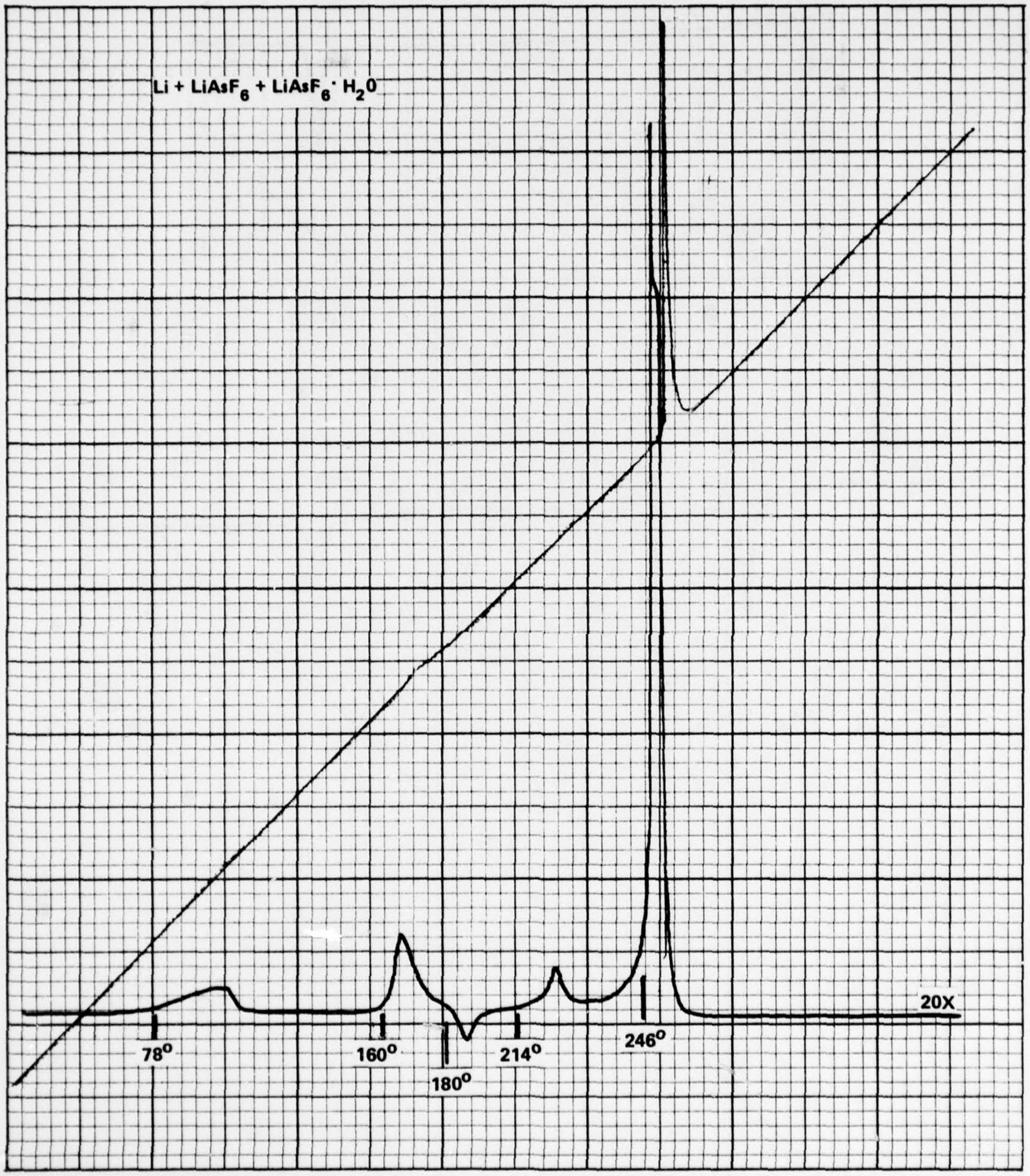

FIGURE 3 DSC THERMOGRAM OF Li+LiAsF$_6$+LiAsF$_6$· H$_2$O 1.52 MG Li, 3.18 MG SALT; 10°C/MIN; 20.0 MCAL/SEC/IN

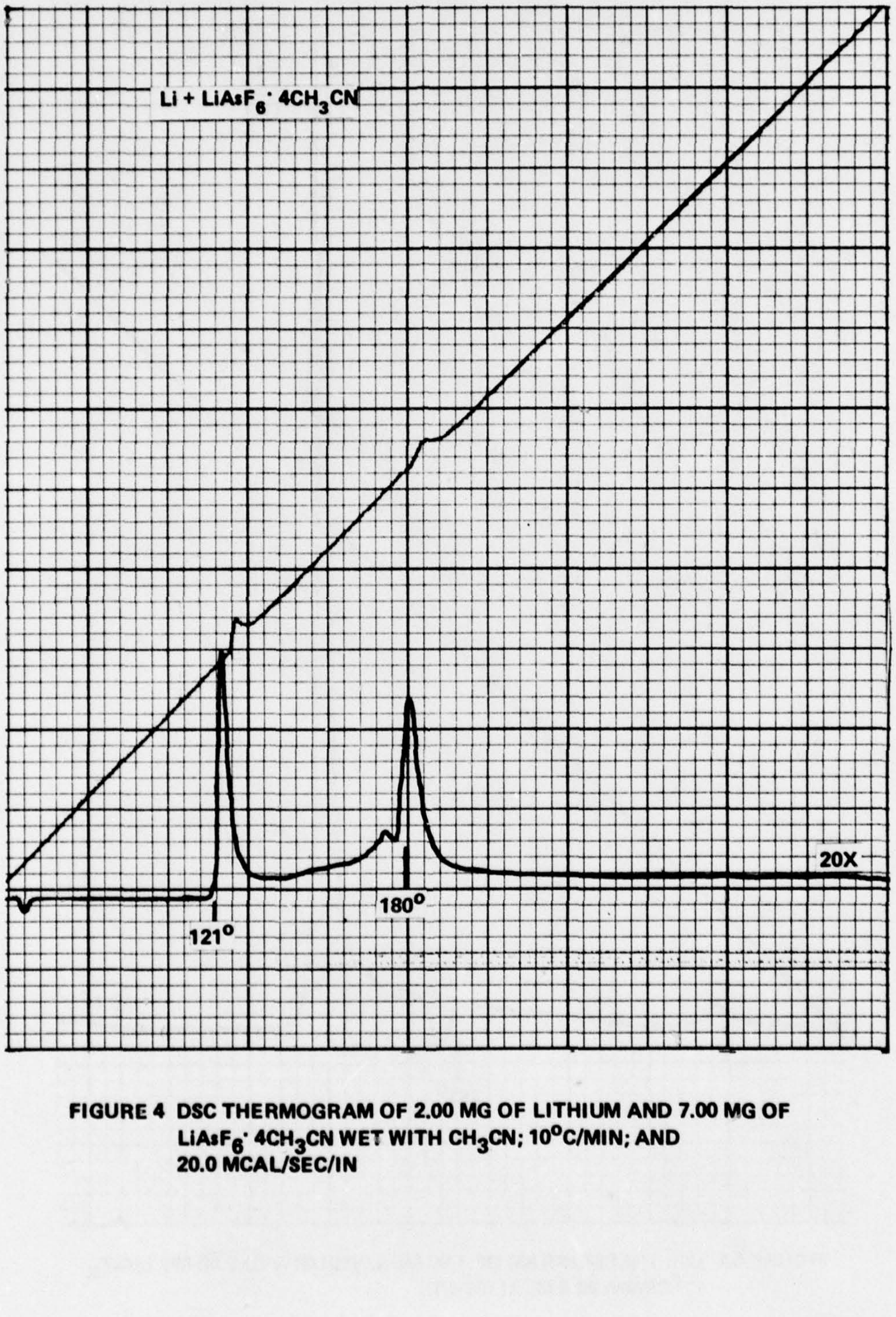

FIGURE 4 DSC THERMOGRAM OF 2.00 MG OF LITHIUM AND 7.00 MG OF LiAsF$_6$ · 4CH$_3$CN WET WITH CH$_3$CN; 10°C/MIN; AND 20.0 MCAL/SEC/IN

FIGURE 5A DSC THERMOGRAM OF 1.90 MG LITHIUM AND 5.00 MG LiAsF$_6$
10°C/MIN; 20.0 MCAL/SEC/IN

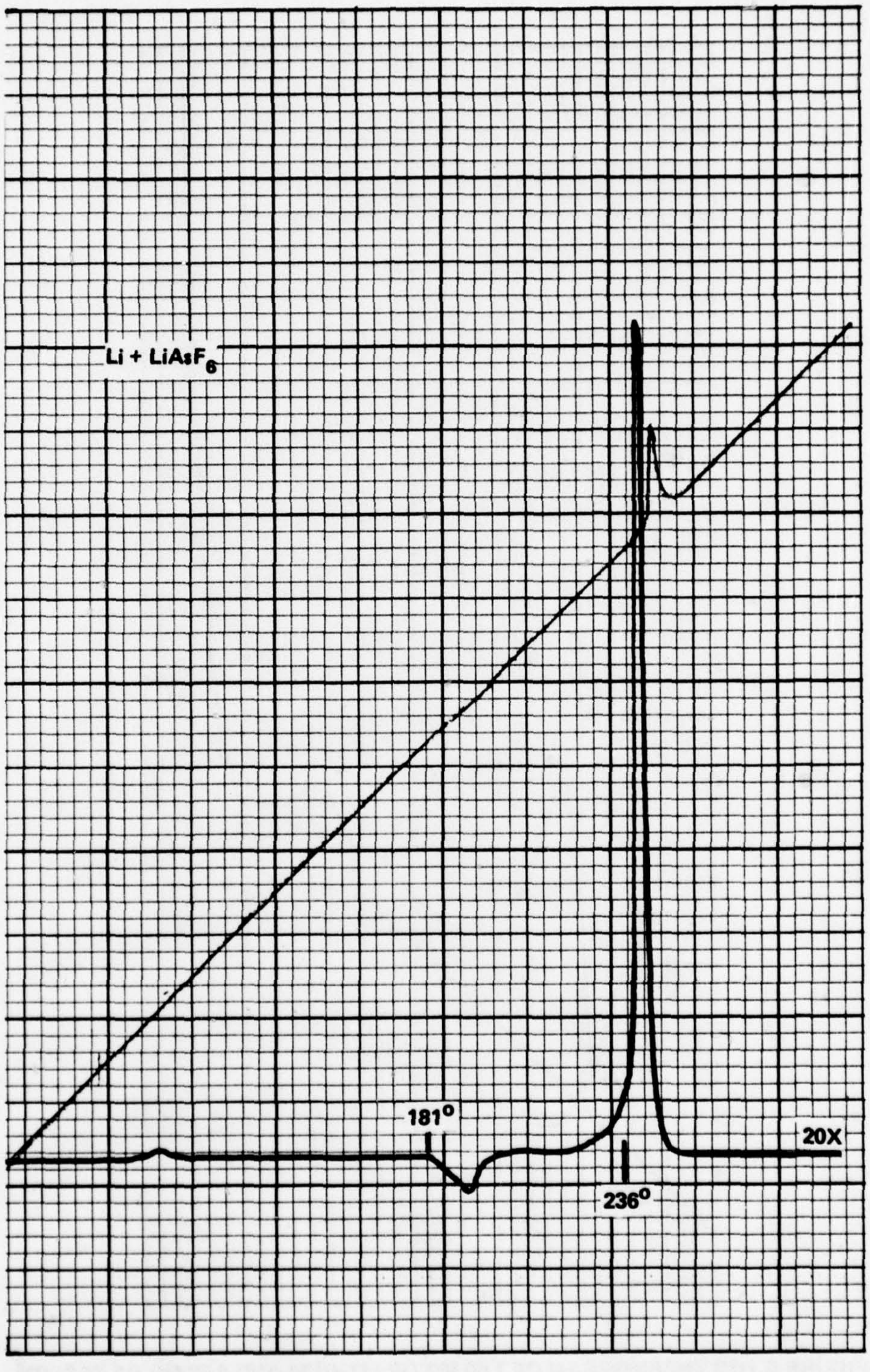

FIGURE 5B DSC THERMOGRAM OF 2.00 MG OF LITHIUM AND 1.80 MG $LiAsF_6$ 10°C/MIN; 20.0 MCAL/SEC/IN

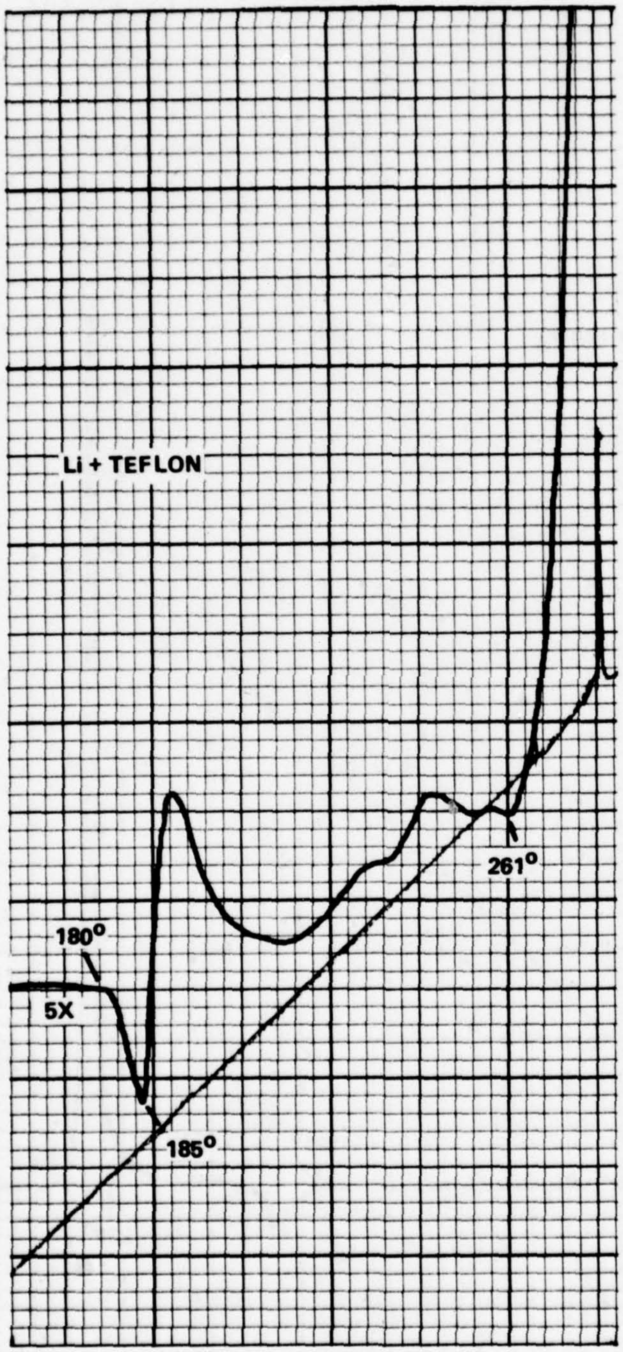

FIGURE 6 DSC THERMOGRAM OF 1.60 MG OF LITHIUM AND 4.20 MG OF TEFLON
10°C/MIN; 5.0 MCAL/SEC/IN

Li + Polypropylene

No exothermic reactions between lithium and polypropylene were observed from 25°C to 300°C. Polypropylene has an endothermic transition at approximately 160°C followed by the melting of lithium at 180°C as illustrated by Figure 7.

Li + LiBr

Samples of LiBr that were dried at 110°C and vacuum dried at 210°C gave similar unexpected exotherms occurring at 142°C and at 131°C respectively. The former is illustrated by Figure 8. Impurities or the deliquescent nature of the LiBr may be responsible.

Li + S

Two endothermic peaks occur corresponding to the melting of two forms of sulfur (Fig 9A).

A very large exothermic reaction occurs between lithium and sulfur in the probable formation of Li_2S. If the reactants "wet" one another, a violent exothermic reaction begins at 142°C resulting in a 120°C temperature increase in the cell as shown in Figure 9B. However, if the lithium and sulfur do not "wet", the exothermic reaction was observed to occur at 235°C (Fig 9A). In another experiment, lithium was pressed to give a large surface area, was immersed in liquid SO_2 for 15 minutes, and was then mixed with sulfur and firmly pressed. The thermogram illustrated by Figure 9C shows a combustion occurring at about 124°C. The heat generated by this reaction increased the cell temperature by 305°C. Apparently, the passivating film formed by SO_2 on lithium is destroyed by molten sulfur.

Li + $Li_2S_2O_3$

Thermograms before and after vacuum drying $Li_2S_2O_3$ confirmed the complete removal of water. The DSC thermogram of lithium and dry $Li_2S_2O_3$ is shown in Figure 10A. Exothermic reactions occur at 152°C and 239°C with a large explosive exotherm occurring at 244°C resulting in a 203°C increase in the cell temperature. The effect of moisture is illustrated in Figure 10B. Thiosulfate containing moisture gives rise to small exotherms at lower temperatures. However, explosive combustion no longer occurs.

Li + $Na_2S_2O_4$

Sodium dithionite, containing 2.5% thiosulfate and 7% bisulfite, was sandwiched in lithium metal. Although some exothermic peaks were observed at approximately 198°C and 222°C, no significant temperature increase was observed as shown in Figure 11.

Li + $Li_2SO_3 \cdot x\ H_2O$ $(x = 0, 1)$

The exothermic behavior shown in Figure 12A appears to be attributable to the reaction between lithium and water. Drying the hydrate at 206° for two hours under vacuum removed the moisture as evidenced by the disappearance of the DSC

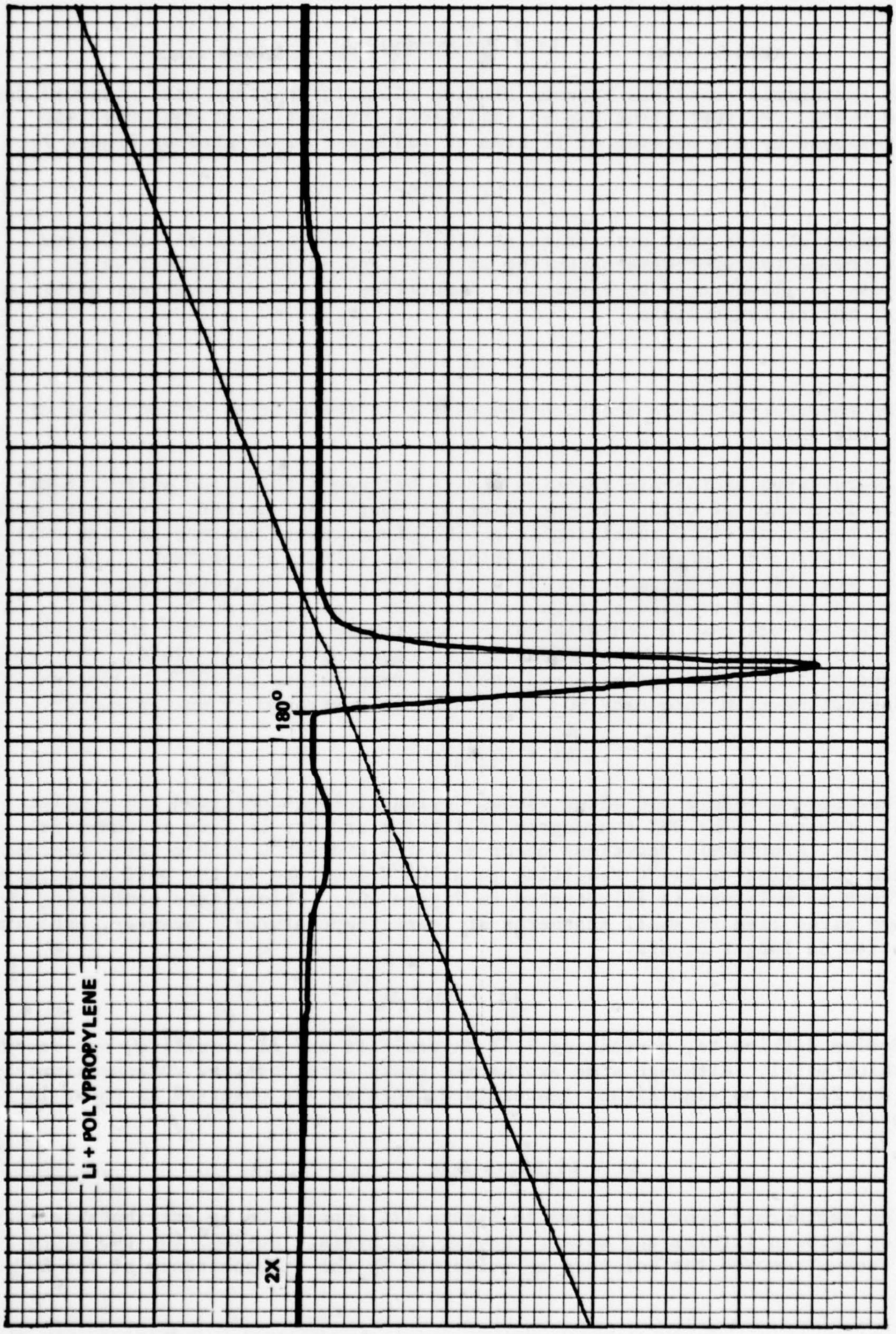

FIGURE 7 DSC THERMOGRAM OF 2.30 MG OF LITHIUM AND 1.60 MG OF NON-WOVEN POLYPROPYLENE. 10°C/MIN; 2.0 MCAL/SEC/IN

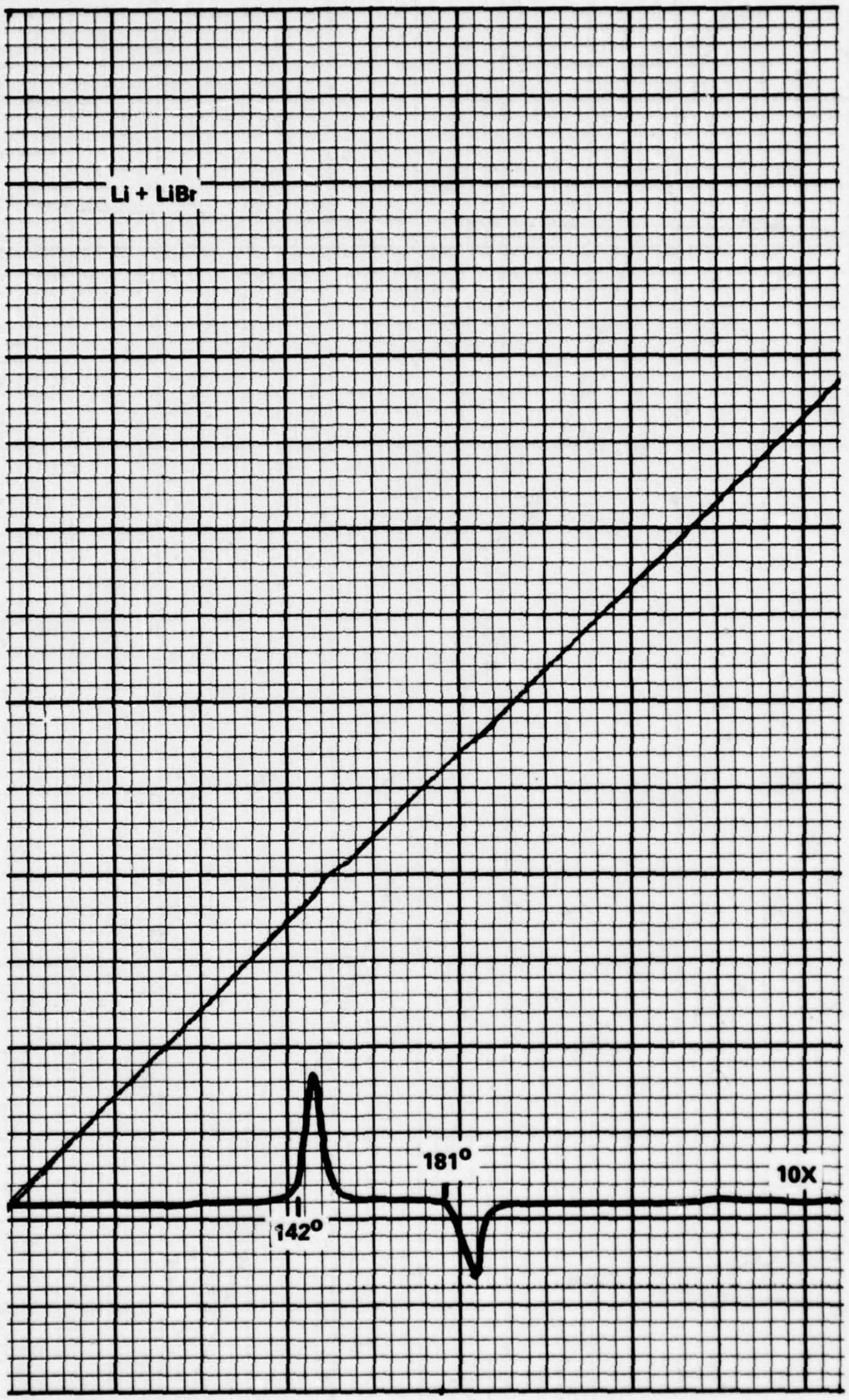

FIGURE 8 DSC THERMOGRAM OF 1.60 MG OF LITHIUM AND 2.20 MG OF LiBr; 10°C/MIN; 10.0 MCAL/SEC/IN

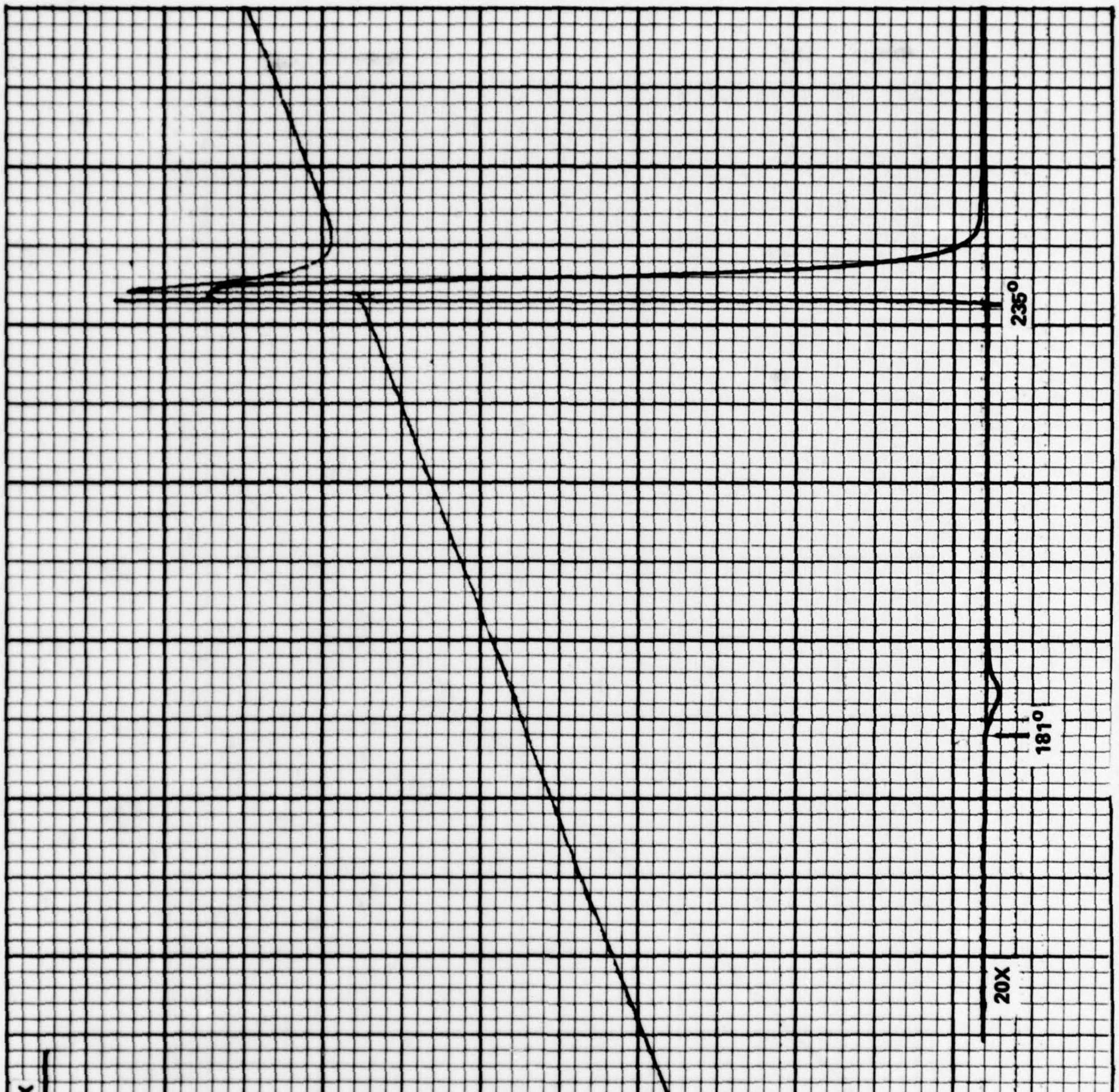

FIGURE 9A DSC THERMOGRAM OF 0.50 MG OF LITHIUM AND 3.10 MG OF SULFUR
10°C/MIN; 1.0 AND 20.0 MCAL/SEC/IN

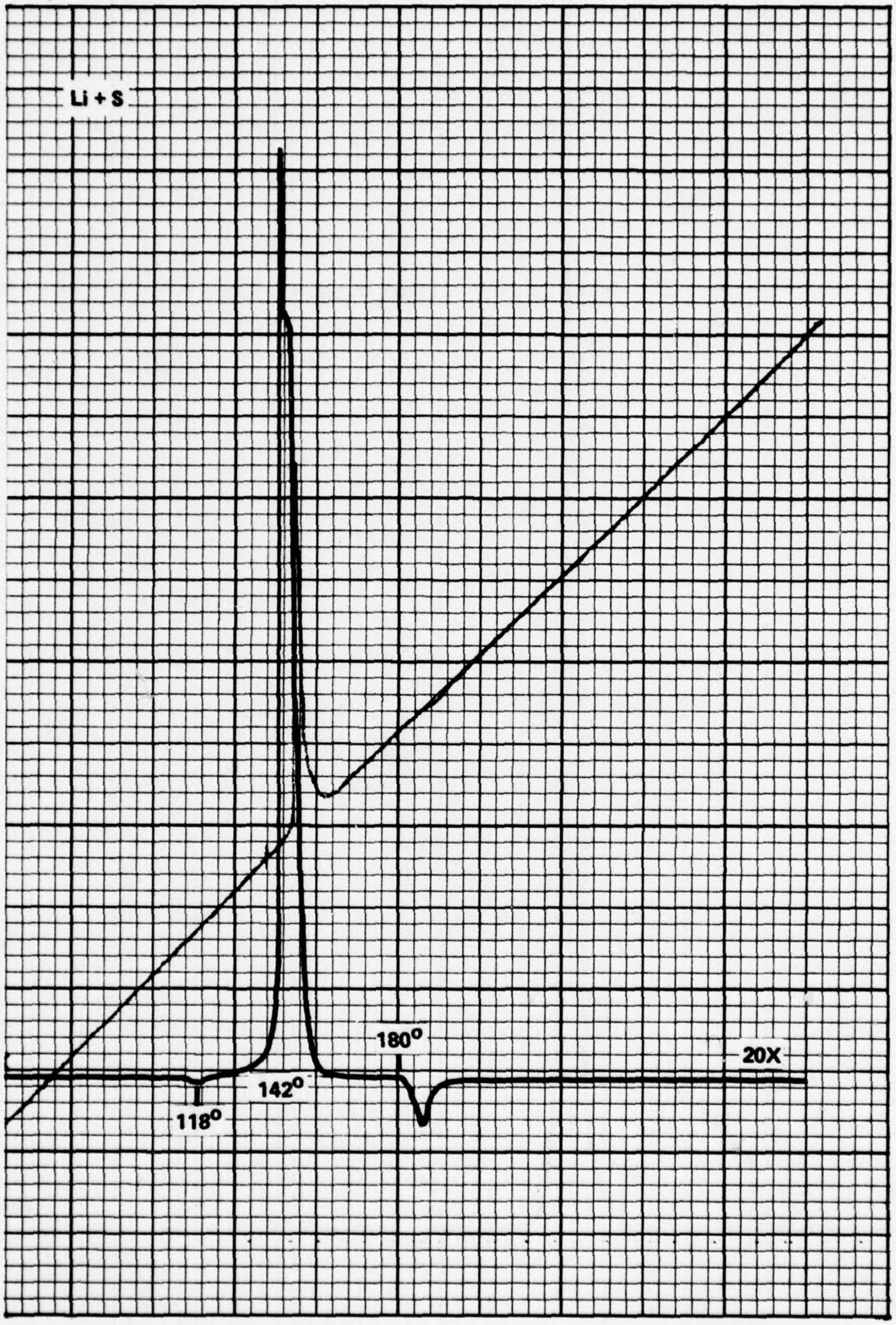

FIGURE 9B DSC THERMOGRAM OF 2.7 MG OF LITHIUM AND 2.2 MG OF SULFUR
10°C/MIN; 20.0 MCAL/SEC/IN

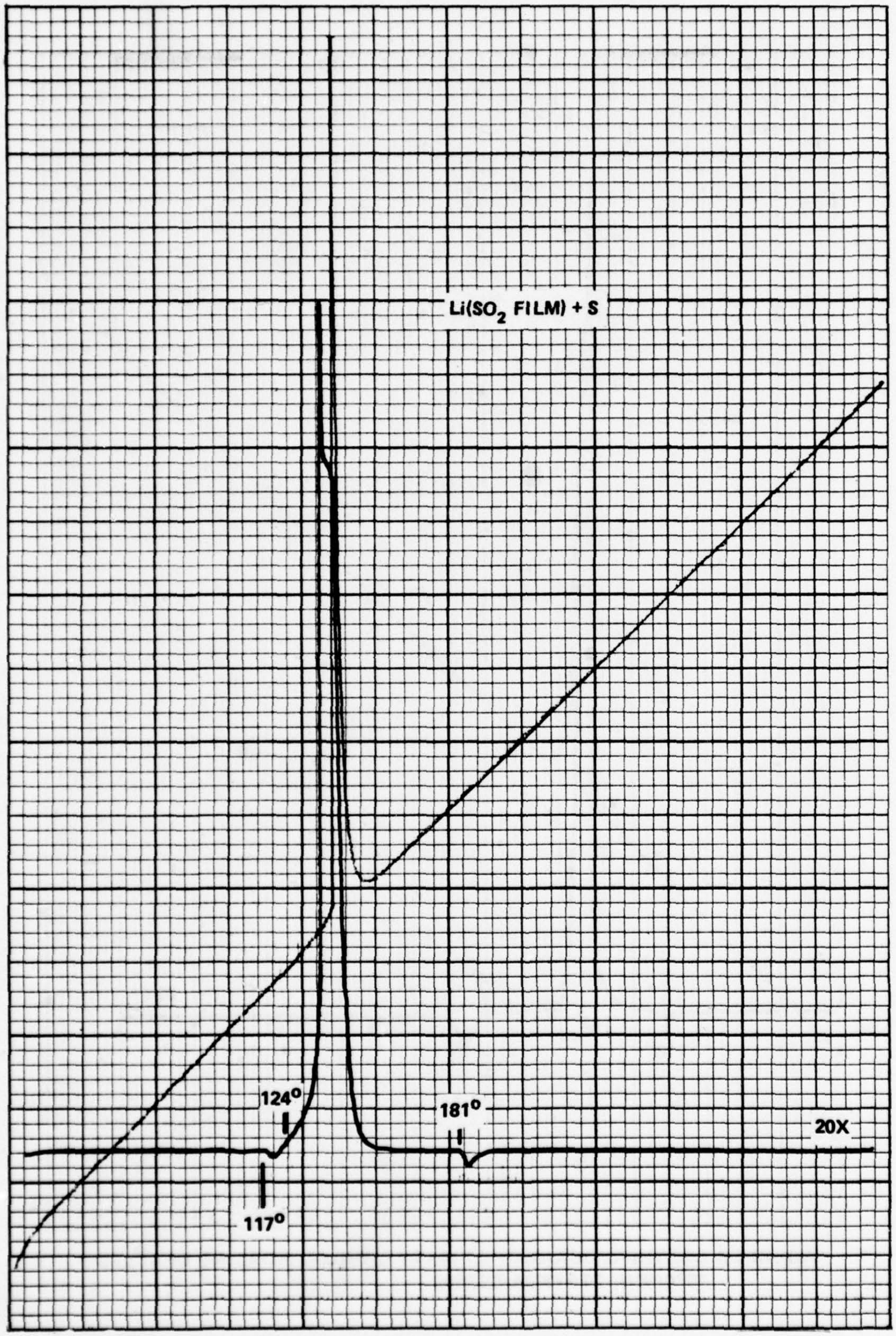

FIGURE 9C DSC THERMOGRAM OF 2.00 MG OF LITHIUM (COATED WITH 0.30 MG OF SO_2) AND 5.05 MG OF SULFUR. 10°C/MIN; 20.0 MCAL/SEC/IN

FIGURE 10A DSC THERMOGRAM OF 3.40 MG OF LITHIUM AND 6.50 MG OF $Li_2S_2O_3$. (DRY) 10°C/MIN; 20.0 MCAL/SEC/IN

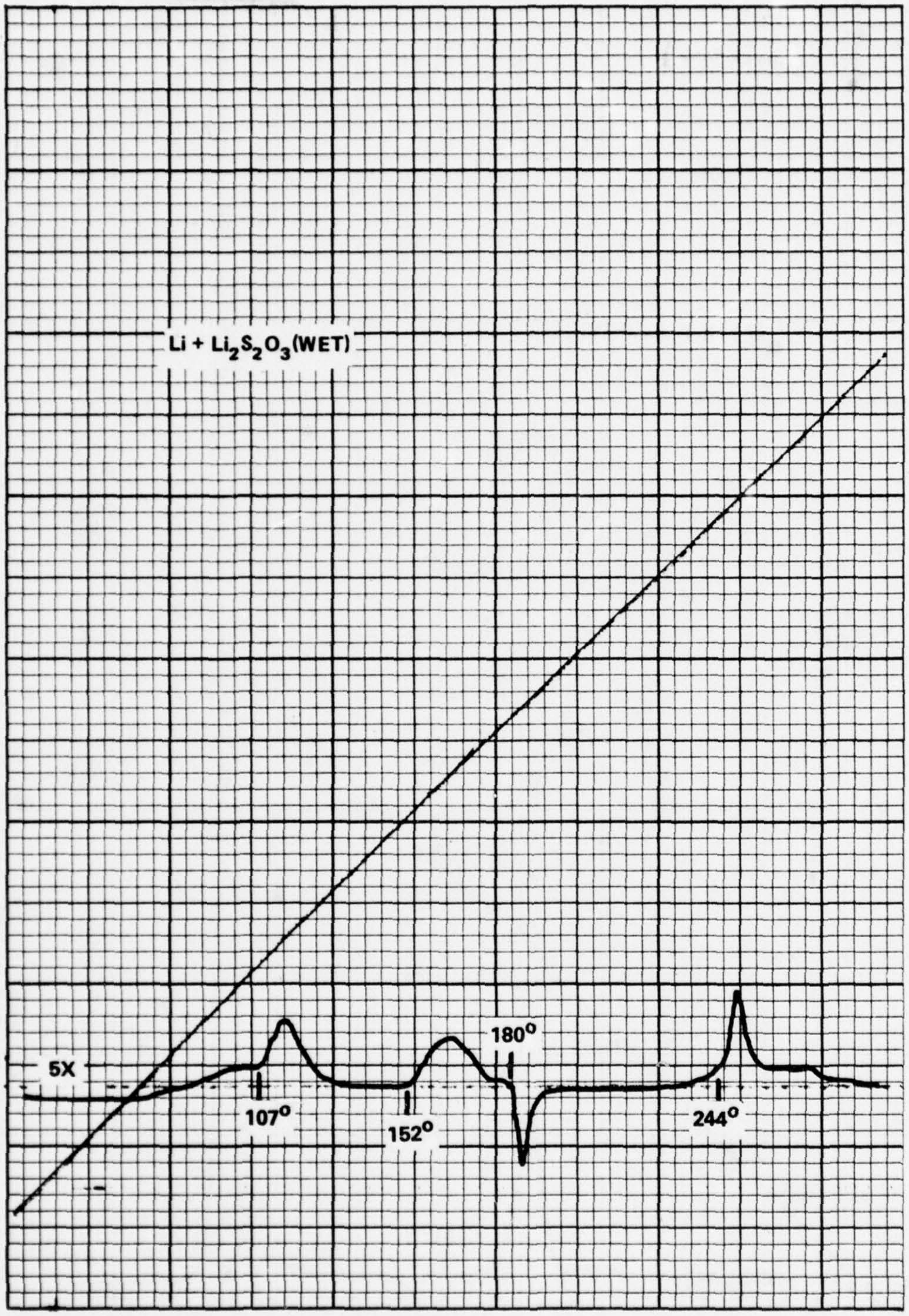

FIGURE 10B DSC THERMOGRAM OF 1.50 MG OF LITHIUM AND 3.20 MG OF $Li_2S_2O_3$. (WET) 10°C/MIN; 5.0 MCAL/SEC/IN

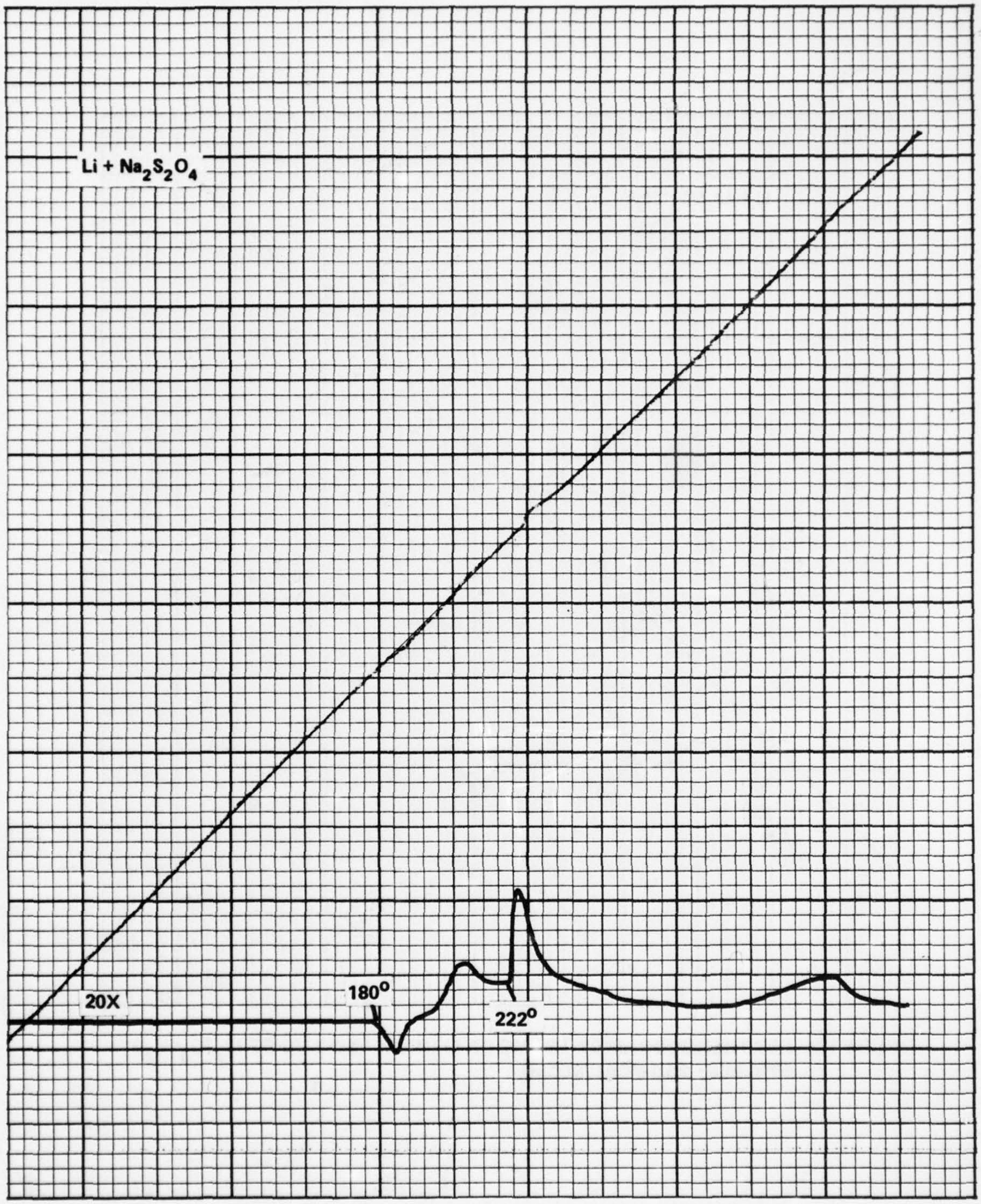

FIGURE 11 DSC THERMOGRAM OF 1.80 MG OF LITHIUM AND 4.30 MG OF SODIUM DITHIONITE. 10°C/MIN; 20.0 MCAL/SEC/IN

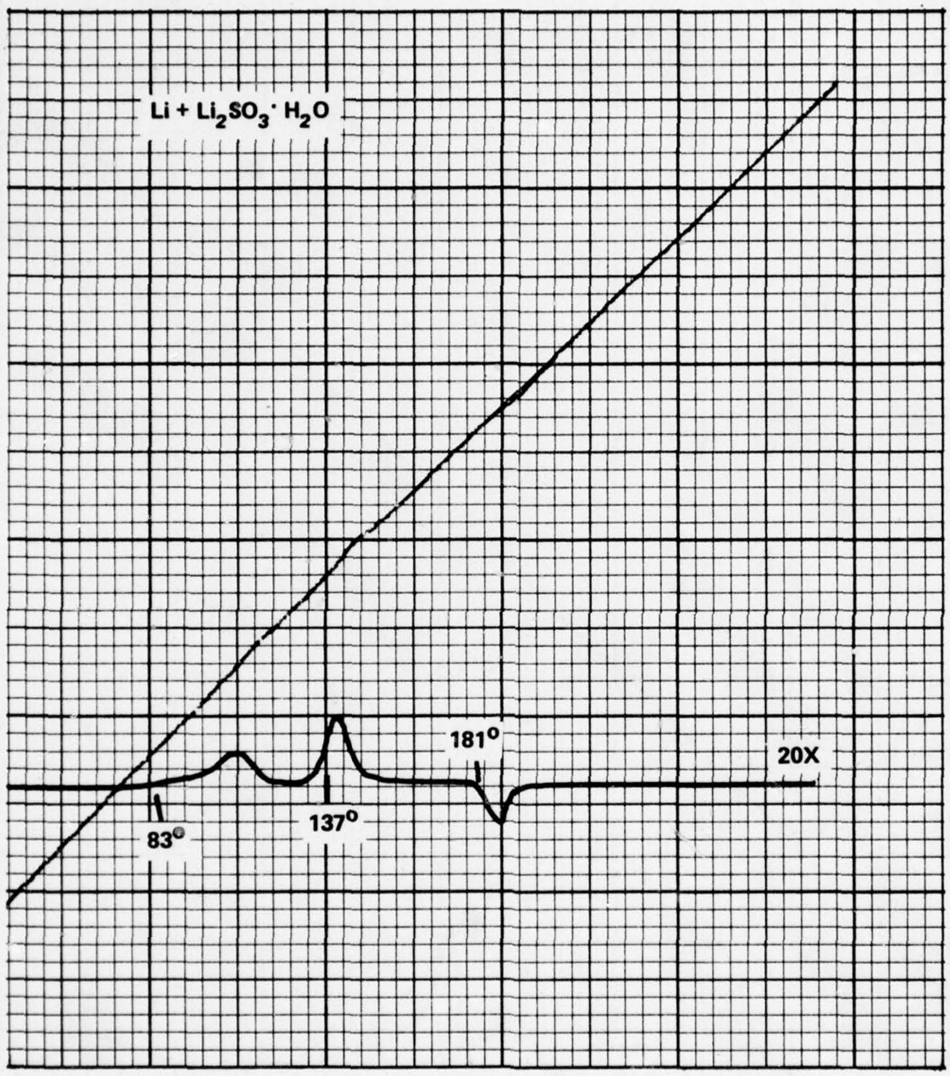

FIGURE 12A DSC THERMOGRAM OF 1.90 MG OF LITHIUM AND 2 MG OF $Li_2SO_3 \cdot H_2O$ 10°C/MIN; 20.0 MCAL/SEC/IN

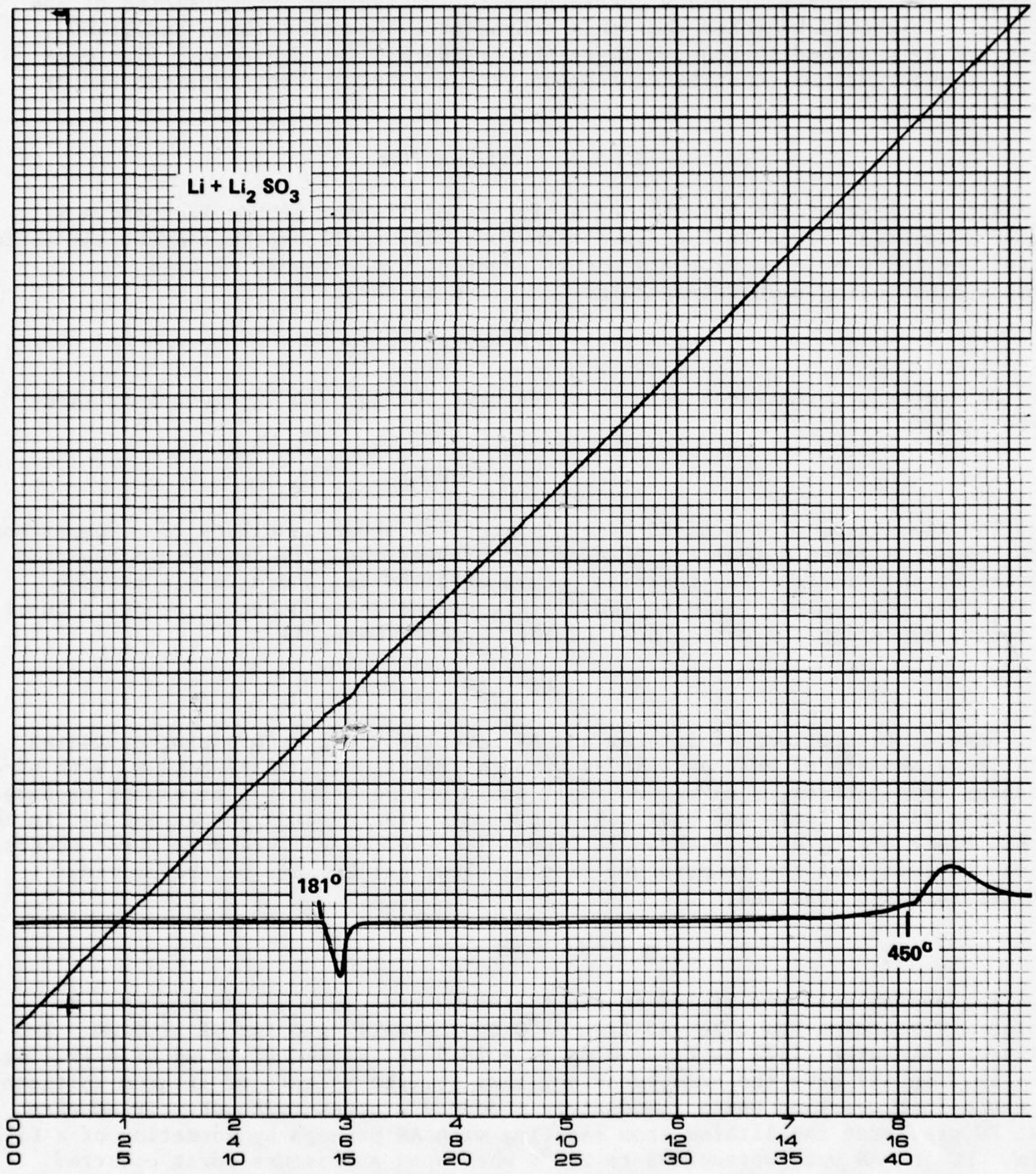

FIGURE 12B DSC THERMOGRAM OF 2 MG OF LITHIUM AND 2 MG OF Li_2SO_3. 10°C/MIN; 20.0 MCAL/SEC/IN endotherm previously recorded at approximately 145°C. The thermogram shown in Figure 12B reveals that no exothermic reaction occurs between anhydrous Li_2SO_3 and lithium. The exotherm at 450°C corresponds to the thermal decomposition of the sulfite. It should be noted that this nonreaction between lithium and Li_2SO_3, confirmed by a second thermogram where the $Li_2SO_3 \cdot H_2O$ was dehydrated five days at 120°C under vacuum, contrasts with the very exothermic reaction reported by Dey(3).

Li + S + Cathode Mix

A thermogram of the behavior of a mixture of lithium with sulfur and a 4:1 mix of Shawinigan black-Teflon gives rise to a very small exotherm after the sulfur melts. At 153°C a large exotherm occurs apparently the result of Li_2S formation producing a 78°C temperature increase in the cell.

Li + propylene carbonate (PC)

No exothermic reactions were observed over the 25°-250°C temperature range investigated.

Li + PC + $LiAsF_6$

The thermogram of the Li-PC-$LiAsF_6$ mixture is shown in Figure 13. Comparison with Figure 5A reveals that the PC does not prevent the very exothermic reaction between Li and $LiAsF_6$. The reaction occurs at 216°C as it did in the absence of PC. The temperature increase immediately causes an endothermic pressure burst to occur, rupturing the o-ring seal of the Perkin Elmer cell.

Li + AN

Several thermograms of lithium and AN revealed slowly increasing exotherms with large amounts of heat associated with the reaction. Unfortunately, with increasing temperatures, the "welded" cell could not maintain the pressures produced. This gave rise to leaks and spurious endotherms. The thermogram carried out in the Perkin Elmer o-ring cell is shown in Figure 14. Before the pressure burst occurs, it can be seen that the temperature rise is slow, unlike some of the previous thermal runaways observed.

Li + AN + PC

The o-ring cell was filled in the following order: PC, lithium, AN. No exothermic reaction was observed from 25°C to the melting point of lithium. Two different mixtures (1.22 mg Li-41.4 mg PC - 13.5 mg AN) and (1.92 mg Li - 19.7 mg PC - 14.9 mg AN) gave the same behavior, that is, the melting of lithium initiated an exothermic reaction which immediately produced a pressure burst. It appears that PC prevented the lithium from reacting with AN perhaps by formation of a Li_2CO_3 film. PC and AN were unreactive to 200°C whereupon a pressure burst occurred.

---

3. A. N. Dey, "Sealed Primary Lithium-Inorganic Electrolyte Cell," Research and Development Technical Report, DELET-TR-74-0109-F, July 1978.

$Li + S + SO_2 + AN$

Thermal analysis revealed that SO_2 prevented reaction between Li and AN. However, SO_2 does not prevent the very exothermic lithium-sulfur reaction. A large exotherm occurs at 147°C approximately the same temperature as the Li-S thermogram. The temperature increase immediately produced a pressure burst in the o-ring cell.

$Li + LiAsF_6 + SO_2 + AN$

Isothermal heating at 130°C for 15 minutes produced no exothermic reaction of any kind. The low temperature exotherm observed in the Li-$LiAsF_6$ reaction was not visible nor were any Li-AN reactions, confirming the passivating effect of SO_2. This is further illustrated by the DSC curve in Figure 15 where an exothermic reaction at the melting point of lithium initiates a pressure burst.

$Li + LiAsF_6 + AN$

A nonexplosive broad exotherm characteristic of a slow reaction between lithium and AN starts at 75°C and continues to the melting of lithium. Another broad exotherm is observed starting at 221°C. Although possibly a Li-$LiAsF_6$ reaction, this latter exotherm is not characteristic of the explosive exotherm observed in the absence of AN. Presumably the kinetics of the reaction is slowed by a simultaneous Li-AN reaction, depletion of most of the lithium or that $LiAsF_6$ is now less concentrated at the lithium surface due to partial solution.

CONCLUSION

From the DSC studies, the following conclusions can be summarized:

(1) Thermal runaways produced by the exothermic reaction occurring between two solid chemicals can be initiated at different temperatures. A good example is the reaction of lithium and sulfur.

(2) Welded cells and o-ring cells were found inadequate for studying volatile liquids beyond the melting point of lithium. Spot welding invariably gives rise to a leaking cell. A better welding technique or a different cell design may be adequate for future studies.

(3) The proper choice of electrolyte salts may be essential in the safety of some lithium cells.

(4) In reactions involving lithium with some solids, the presence of moisture may produce low temperature exotherms involving water and lithium. However moisture may also severely reduce the exothermicity of another reaction. This is illustrated in Figures 10A and 10B.

(5) Several hazardous conditions may result from extremely exothermic reactions that involve lithium with either initial cell components or cell discharge products.

(6) Some components have beneficial effects by reducing or preventing exothermic reactions. SO_2 and PC both prevent the Li-AN reaction. AN may reduce the kinetics of the Li-$LiAsF_6$ reaction and water hinders the Li-$Li_2S_2O_3$ combustion.

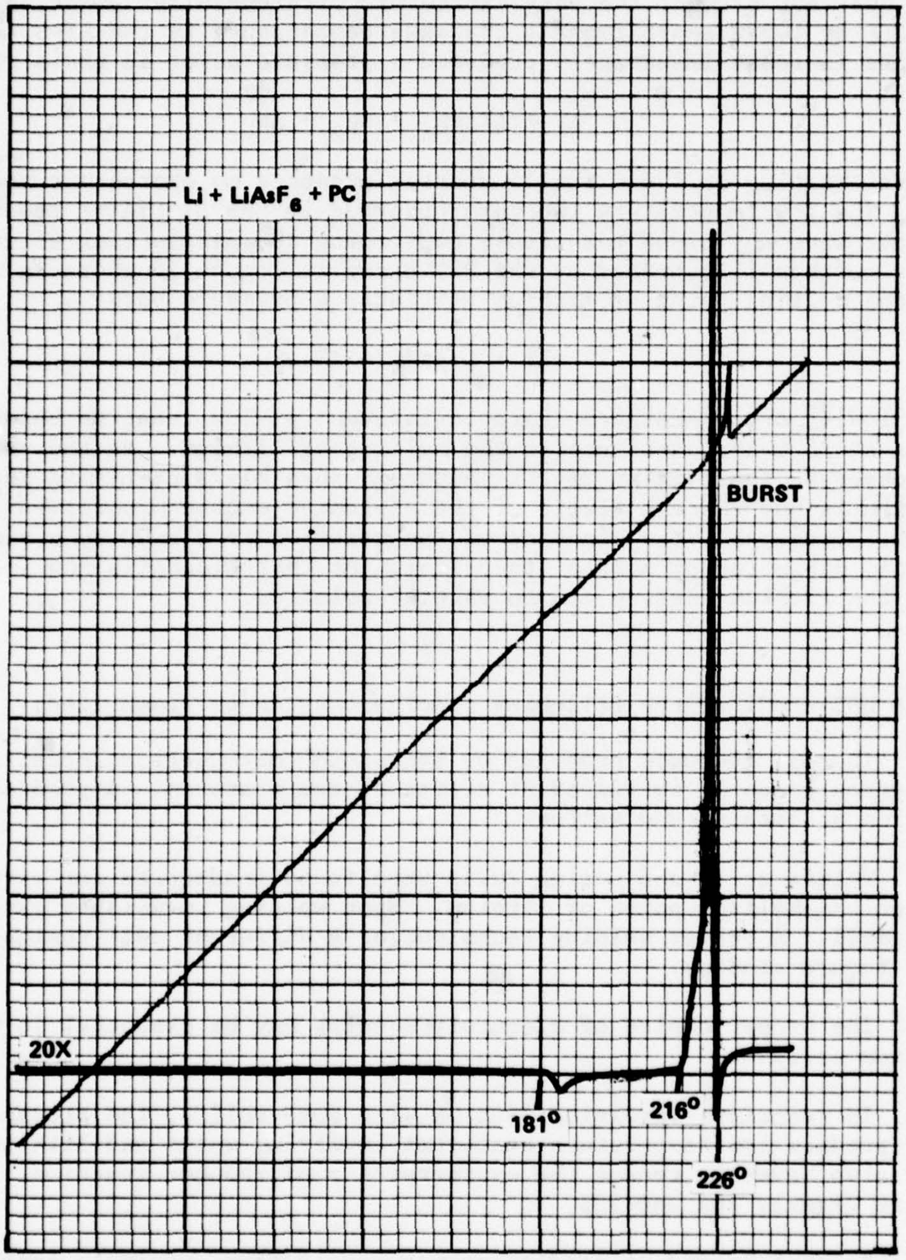

FIGURE 13 DSC THERMOGRAM OF 1.39 MG OF LITHIUM, 2.10 MG OF LiAsF6 AND 31.0 MG OF PROPYLENE CARBONATE. 10°C/MIN; 20.0 MCAL/SEC/IN

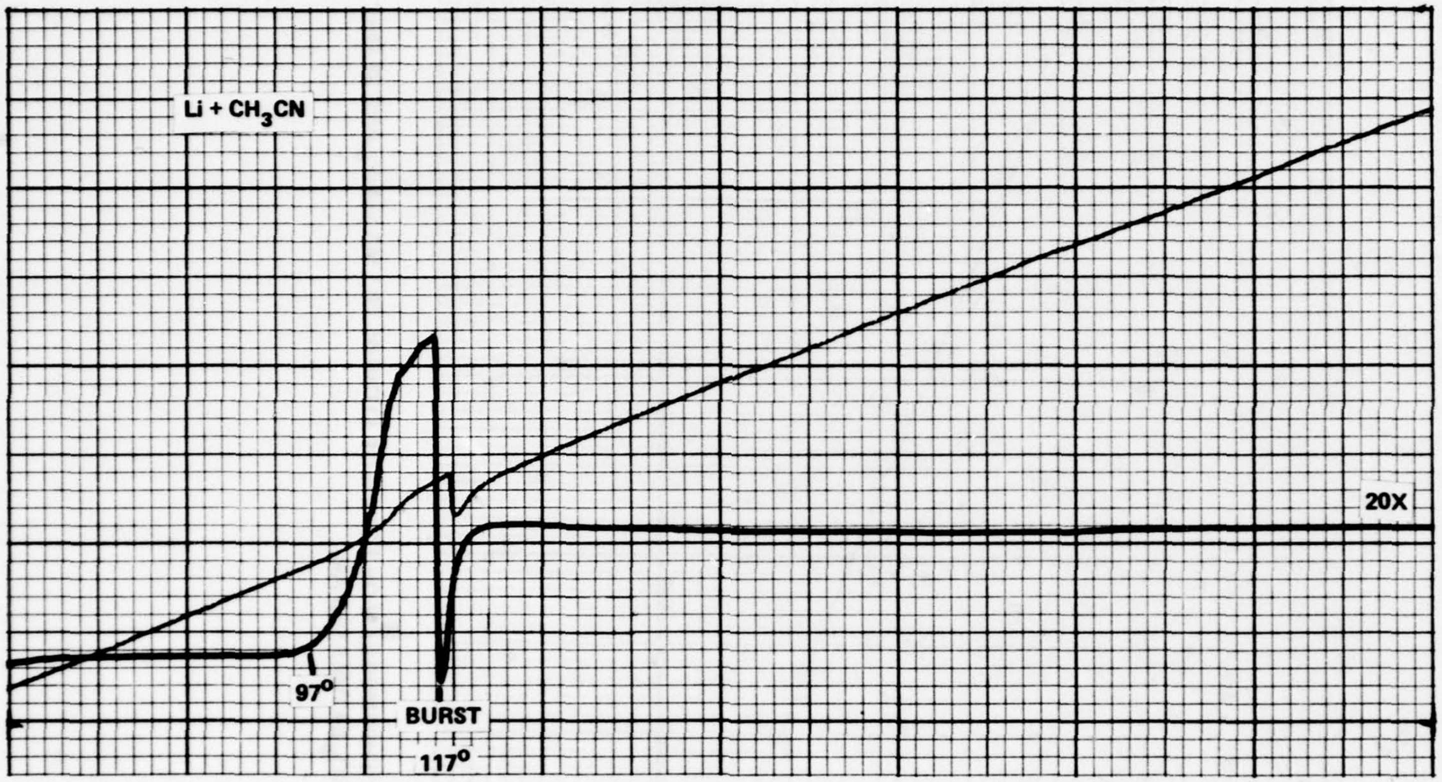

FIGURE 14 DSC THERMOGRAM OF 2.01 MG OF LITHIUM AND APPROXIMATELY 38 MG OF CH_3CN. 20°C/MIN; 20.0 MCAL/SEC/IN

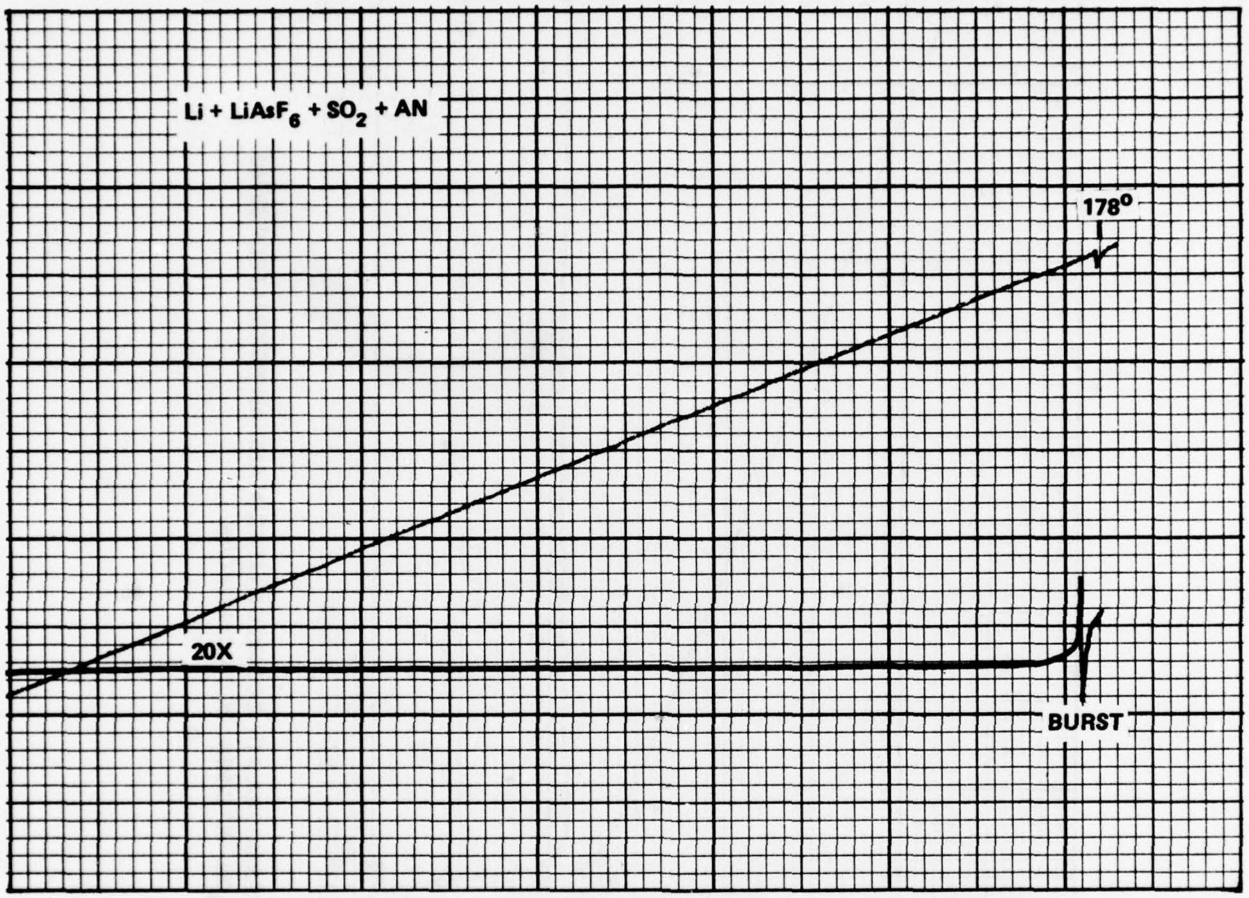

FIGURE 15 DSC THERMOGRAM OF 2.30 MG OF LITHIUM, 10.1 MG $LiAsF_6$ AND 38 MG OF SO_2 SATURATED AN

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