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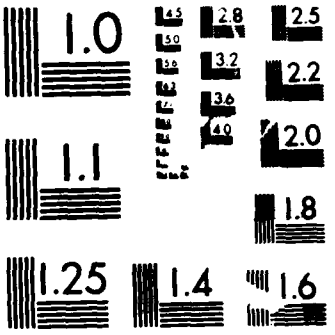

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ELECTROLYTE CONDUCTANCE IN THE $LiAsF_6$-1,1,2,2-TETRACHLOROETHANE-COSOLVENT SYSTEM

BY YVONNE TRAN PATRICIA H. SMITH KATHLEEN M. O'NEILL
STANLEY D. JAMES

RESEARCH AND TECHNOLOGY DEPARTMENT

1 AUGUST 1987

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Electrolyte Conductance in the $LiAsF_6$-1,1,2,2-Tetrachloroethane Cosolvent System

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Conductance was measured at lithium hexafluoroarsenate dissolved in binary mixtures of 1,1,2,2-tetrachloroethane with the following cosolvents: methyl formate, tetrahydrofuran, ethyl acetate, γ-butyrolactone, propylene carbonate, ethylene glycol dimethyl sulfite. The conductivity measurements were conducted at -30, 0, 25°C.

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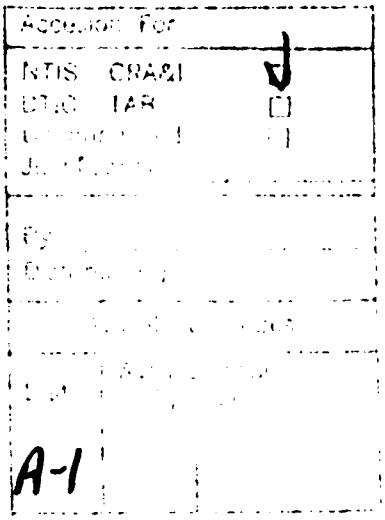
FOREWORD

In recent work in our laboratories, 1,1,2,2-tetrachloroethane was selected as an alternate cathode material for possibly safer lithium batteries. However, it is a poor solvent medium for lithium salts. A cosolvent is needed to dissolve the salt and raise the conductivity of the electrolyte. A number of non-aqueous dipolar aprotic liquids were investigated as possible cosolvents for 1,1,2,2,-tetrachloroethane. This report presents the conductivity study of these electrolyte systems as a function of temperature between -20 and 25°C.

This work was supported by the Independent Research and Exploratory Development programs at NSWC.

Approved by:

JACK R. DIXON, Head
Materials Division



CONTENTS

ILLUSTRATIONS

TABLES

INTRODUCTION

In search of a lithium battery system safer than the present energetic SO_2 and sulfur oxychlorides, we investigated 1,1,2,2-tetrachloroethane as a new cathode because of its low toxicity and reduced shock sensitivity with lithium.[1] Though this halocarbon liquid is an active cathode, it is a poor solvent for lithium salts. Therefore, the viability of this new cathode demands the presence of a cosolvent.

Our investigation focused on finding halocarbon/cosolvent/ $LiAsF_6$ systems that are highly conductive over the temperature range of -20 to 25°C. This report presents the results of our studies on conductivities of $LiAsF_6$ solutions in the binary mixtures of 1,1,2,2-tetrachloroethane and the following cosolvents: acetonitrile, methyl formate, tetrahydrofuran, ethyl acetate, γ-butyrolactone, propylene carbonate and dimethyl sulfite.

EXPERIMENTAL

The reagents used for this experiment were:

$LiAsF_6$, Electrochemical Grade, U.S. Steel Agri-Chemicals

ethyl acetate (EtOAc), anhydrous 99 percent; γ-butyrolactone (γBL), 99 percent; tetrahydrofuran (THF), 99.9 percent; 1,1,2,2-tetrachloroethane (TCE), 98 percent; dimethyl sulfite (DMSI), 99.7 percent: Aldrich Chemical Co.

propylene carbonate (PC); water percent 0.006 percent: Burdick and Jackson Laboratories

acetonitrile (AN), 99 percent: Fisher Scientific

methyl formate (MF): MCB MX-1041-30 Honeywell Grade.

A Yellow Spring Instrument borosilicate dip-type cell (model no. 3403) connected to a YSI Conductance Resistance Meter (model no. 34) was used to measure the resistance of the solution. The YSI model 34 applies a 1 volt square wave to the sample under test. The model 3403 cell constant was determined to be 1.10 cm^{-1}. The specific conductance of the electrolyte was calculated using the resistance-specific conductance relationship,[2]

$$\kappa = C / R \quad (\Omega^{-1}\ cm^{-1}) \qquad (1)$$

where κ = specific conductance (Ω^{-1} cm^{-1})
C = cell constant (cm^{-1})
R = measured resistance (Ω)

Most of the solution resistances (R) measured in this work were below 2000 Ω where the YSI model 34's output frequency is 2000 Hz. In the few measurements above 2000 Ω the frequency was 177 Hz.

Experiments were performed in a dry room in which the humidity did not exceed 0.5%. TCE-cosolvent mixtures were made up in terms of volume percent, e.g. a 25% TCE-75% THF mixture was made by combining 25 volumes of TCE with 75 volumes of THF. Solubility tests were then made to estimate maximum $LiAsF_6$ concentration achievable in these TCE/cosolvent solutions at room temperature. After this concentration was determined, a concentrated $LiAsF_6$ stock solution was prepared for subsequent dilution with the TCE-cosolvent mixture.

The prepared solutions were transferred into 16 x 100 mm Pyrex test tubes. A 5 ml sample was required for the conductivity test. Protected with Parafilm, these test tubes were placed in a constant temperature bath for at least an hour to reach thermal equilibrium at the set temperature. The constant temperature bath was a Haake A81 equipped with a digital read-out monitoring the temperature with an accuracy of $\pm 0.01^{o}C$. The bath liquid was a glycol-alcohol-water mixture appropriate to the particular temperature. The temperatures selected for the conductivity measurements were 25, 0 and $-20^{o}C$.

Conductivity measurements were performed as follows: The conductivity cell was placed in the vessel that contained the solution studied. After 1 to 2 minutes, the constant solution resistance was read from the YSI Conductance Resistance Meter display. Using the known cell constant, the specific conductance of the electrolyte was obtained using equation (1). After each reading, the conductivity cell was first rinsed with the pure cosolvent then with acetone afterwards to remove any adhering salt. A heat gun was employed to air-dry the conductivity cell. Specific conductances were plotted against molar concentrations at 25, 0 and $-20^{o}C$ to generate Figures 1 through 9.

RESULTS AND DISCUSSION

Tables 1 through 9 list the conductivity data at 25, 0 and $-20^{o}C$. Figures 1 through 9 are plots of the specific conductances versus molar concentrations at these temperatures. Conductance data were measured up to the limit of salt solubility. A maximum specific conductivity was obtained for most plots. The solubility of $LiAsF_6$ in TCE solutions containing 25% THF, 25% MF or 50% AN is low. In these cases (Figures 2, 4 and 8) no maxima were observed in the accessible concentration range.

On cooling below room temperature, solutions of $LiAsF_6$ in TCE mixtures containing 50% γBL, 50% THF or 50% AN become supersaturated. At $0^{o}C$, 50% AN solution experiences salt precipitation at concentrations of 0.392 M or greater. At $-20^{o}C$, similar behavior is observed in 50% THF solution at molar concentrations above 1.35 M. Conductance data of 50% γBL solution with concentrations above 0.752 M are not reproducible at $0^{o}C$ due probably to salt separation. At $-20^{o}C$, this solution freezes at 0.846 M. In all cases,

electrolyte conductance decreases with decreasing temperature as shown in Figures 1 through 9. Lowering the temperature causes a decrease in ionic mobility. At 25°C the ranking of the highest specific conductivities obtained with 50% 1,1,2,2-tetrachloroethane, 50% cosolvent solutions is arranged as PC < γBL < THF ≃ DMSI < EtOAc < MF ≃ AN. At 0°C, the conductivity ranking is PC < γBL < DMSI < THF < EtOAc < AN < MF. At -20°C, the conductivity order is PC < γBL < DMSI < THF < EtOAc < MF.

To understand this ordering, a comparison was made between the maximum specific conductance of the solution and its calculated dielectric constant and viscosity. (The dielectric constant ϵ and the viscosity η of the electrolyte are known to strongly affect conductivity by influencing the number and mobility respectively of ionic species.) The following equation[3] were used to generate Table 10:

$$\text{dielectric constant of mixture} = y_1\epsilon_1 + y_2\epsilon_2 + \ldots$$

where y_i, ϵ_i are volume fraction and dielectric constant of component i

$$\text{viscosity of mixture} = \eta_1^{x_1}\eta_2^{x_2}\ldots$$

where x_i, η_i are mole fraction and viscosity of component i

Although there was no direct correlation, one trend was observed; the viscosity of the cosolvent (and hence the solution) is more important than the dielectric constant of cosolvent/solution. Thus PC or γBL solutions were not the most conductive even though they have the highest dielectric constants.

We observed a decrease in solution conductivity as we decreased the volume percent of the cosolvent in 1,1,2,2-tetrachloroethane mixtures. Figures 1 through 4 illustrate this effect in methyl formate and tetrahydrofuran. As discussed above, the controlling factor is probably the higher viscosity of mixtures rich in tetrachloroethane. We also observed that for cosolvents of high dielectric constants, the highest electrolyte conductivities occurred at lower salt concentration (C < 1.0 M) as in PC, γBL, AN and DMSI, while with lower dielectric constant cosolvent (MF, THF and EtOAc) the maxima in conductance shifted to salt concentrations above 1.0 M. This is illustrated in Table 11. Similar observations on other systems have been reported in the literature.[3]

CONCLUSIONS

$LiAsF_6$ solutions in 50:50 volume mixtures of 1,1,2,2-tetrachloroethane with acetonitrile, methyl formate are more conductive than such solutions with propylene carbonate, γ-butyrolactone, tetrahydrofuran, dimethyl sulfite and ethyl acetate. At 25°C, the maximum specific conductances of 50 volume percent mixtures of 1,1,2,2-tetrachloroethane with acetonitrile, and methyl formate solutions are 12.6 x 10^{-3} $\Omega^{-1}cm^{-1}$ at 0.653 M, and 12.5 x $10^{-3}\Omega^{-1}cm^{-1}$ at 1.40 M respectively.

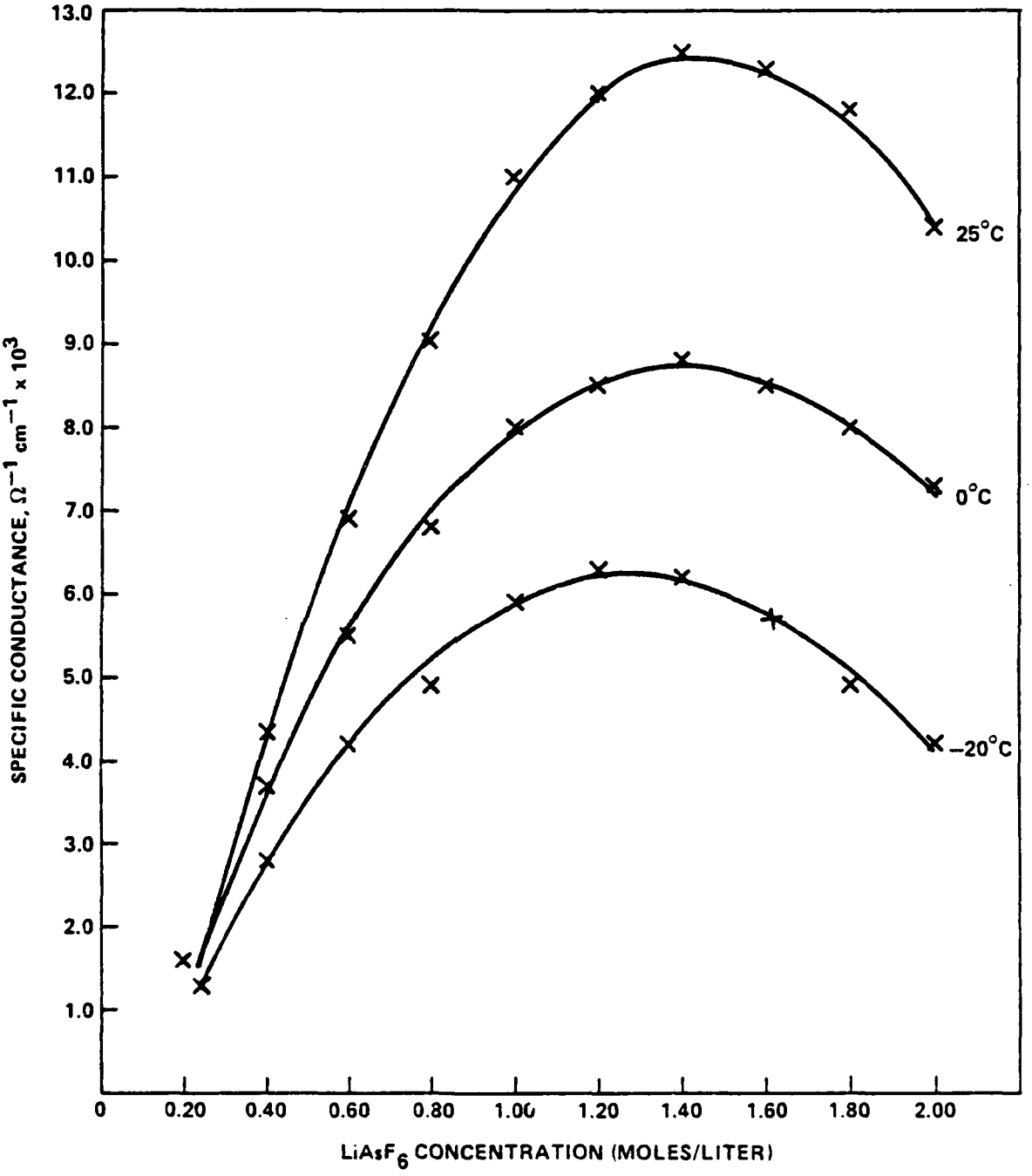


FIGURE 1. SPECIFIC CONDUCTANCE OF 50% 1,1,2,2-TETRACHLOROETHANE, 50% METHYL FORMATE SOLUTIONS

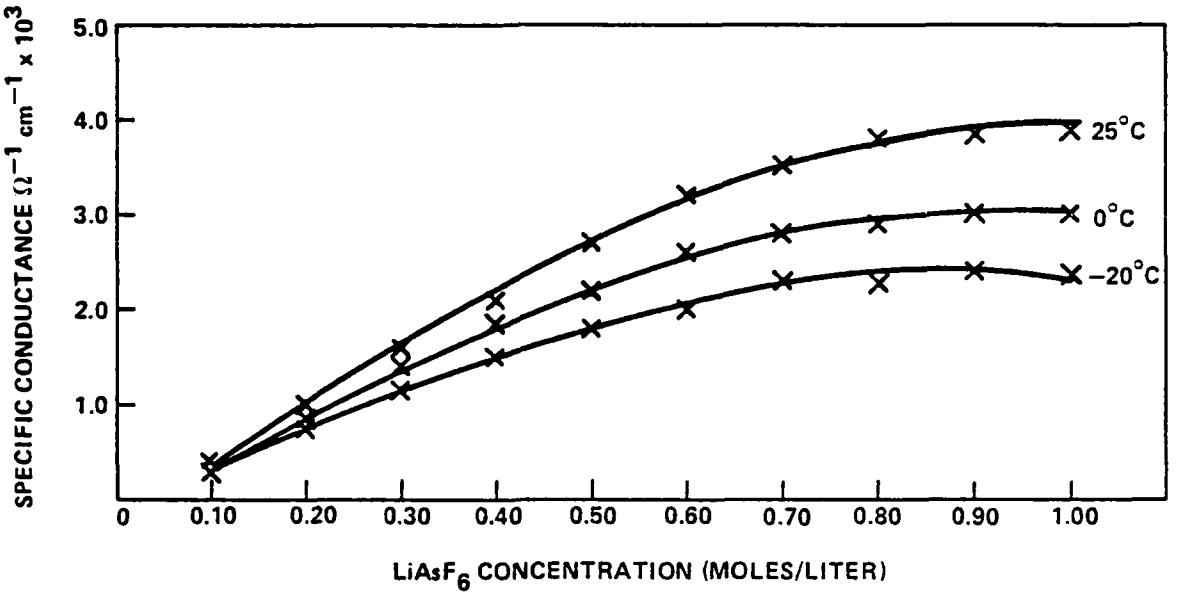


FIGURE 2. SPECIFIC CONDUCTANCE OF 75% 1,1,2,2-TETRACHLOROETHANE, 25% METHYL FORMATE SOLUTIONS

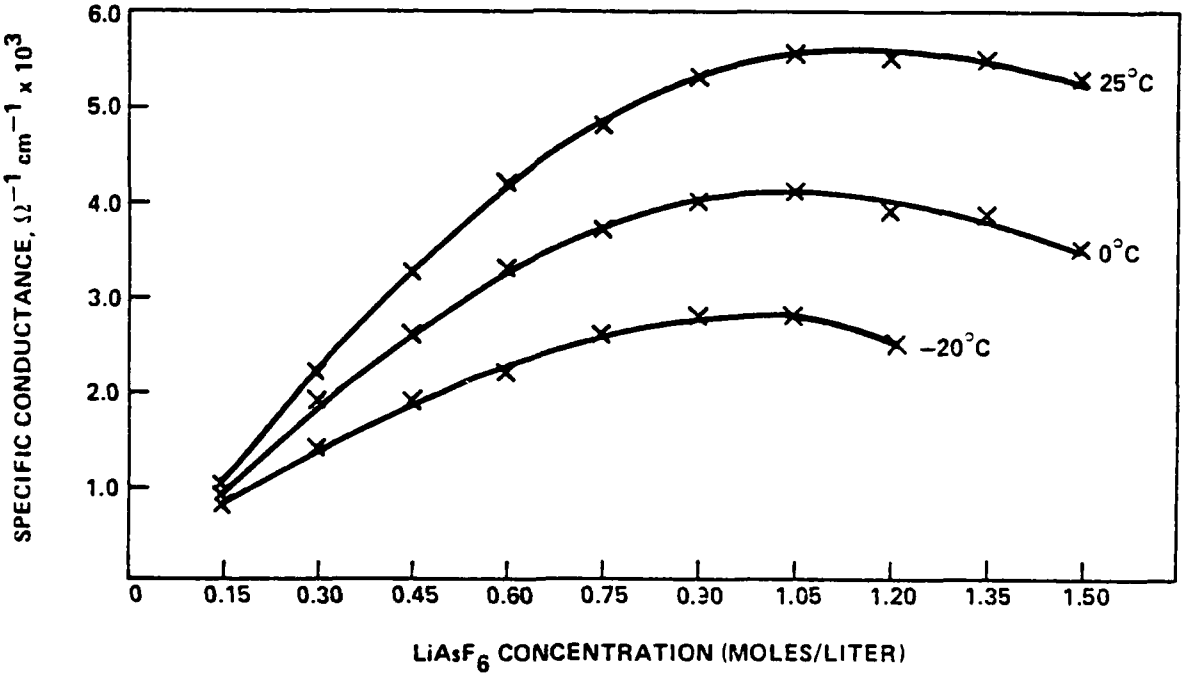


FIGURE 3. SPECIFIC CONDUCTANCE OF 50% 1,1,2,2-TETRACHLOROETHANE, 50% TETRAHYDROFURAN SOLUTIONS

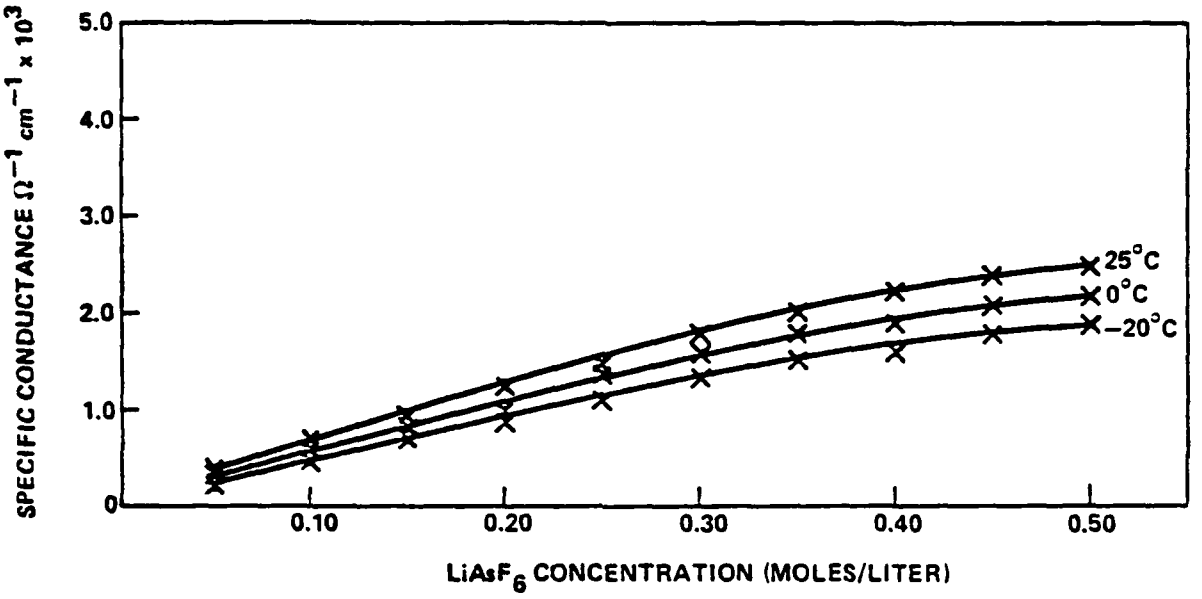


FIGURE 4. SPECIFIC CONDUCTANCE OF 75% 1,1,2,2-TETRACHLOROETHANE, 25% TETRAHYDROFURAN SOLUTIONS

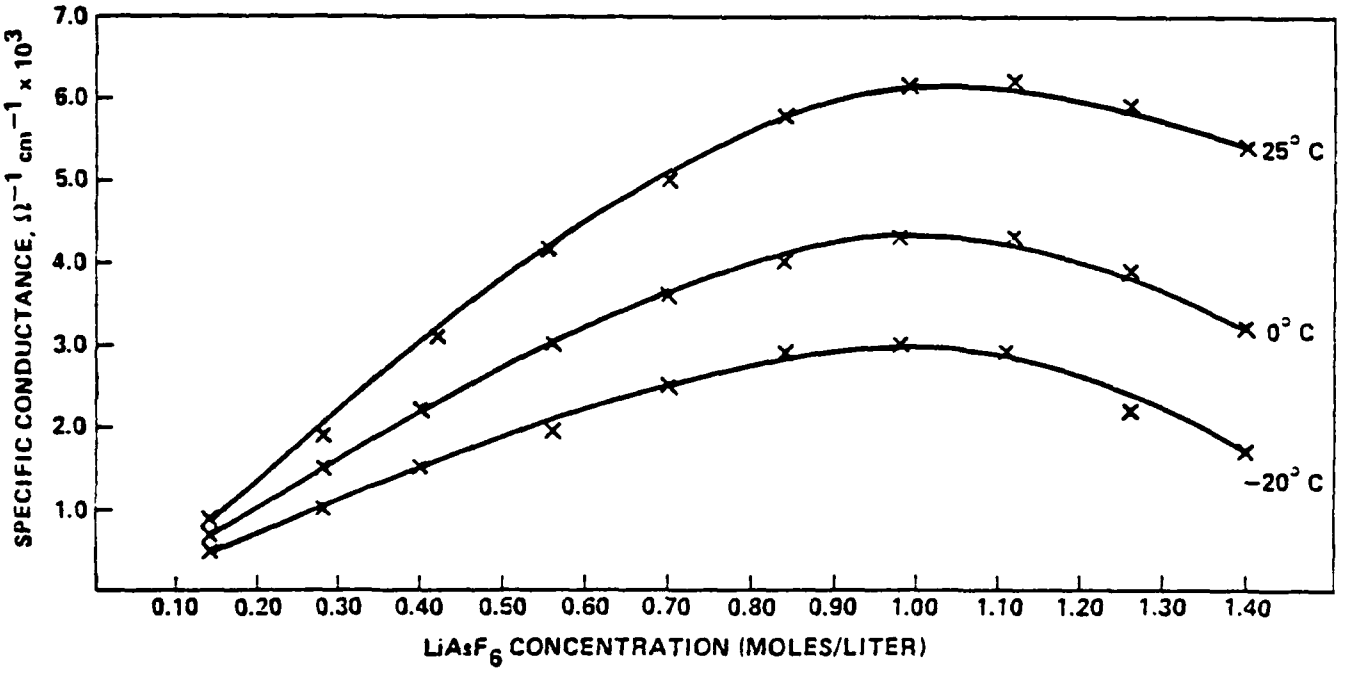


FIGURE 5. SPECIFIC CONDUCTANCE OF 50% 1,1,2,2-TETRACHLOROETHANE, 50% ETHYL ACETATE SOLUTIONS

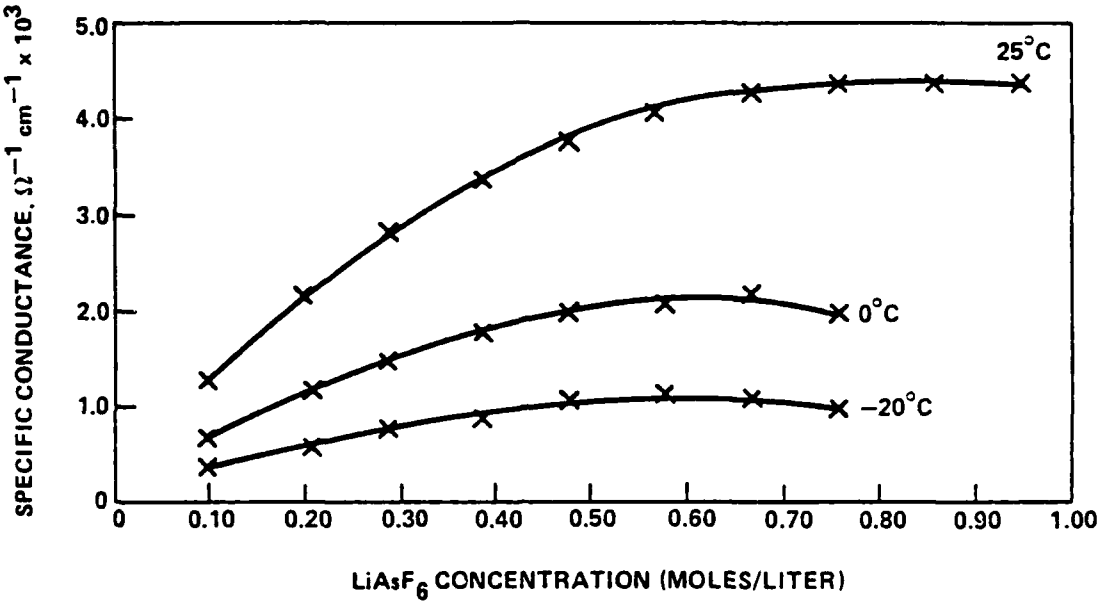


FIGURE 6. SPECIFIC CONDUCTANCE OF 50% 1,1,2,2-TETRACHLOROETHANE, 50% γ-BUTYROLACTONE SOLUTIONS

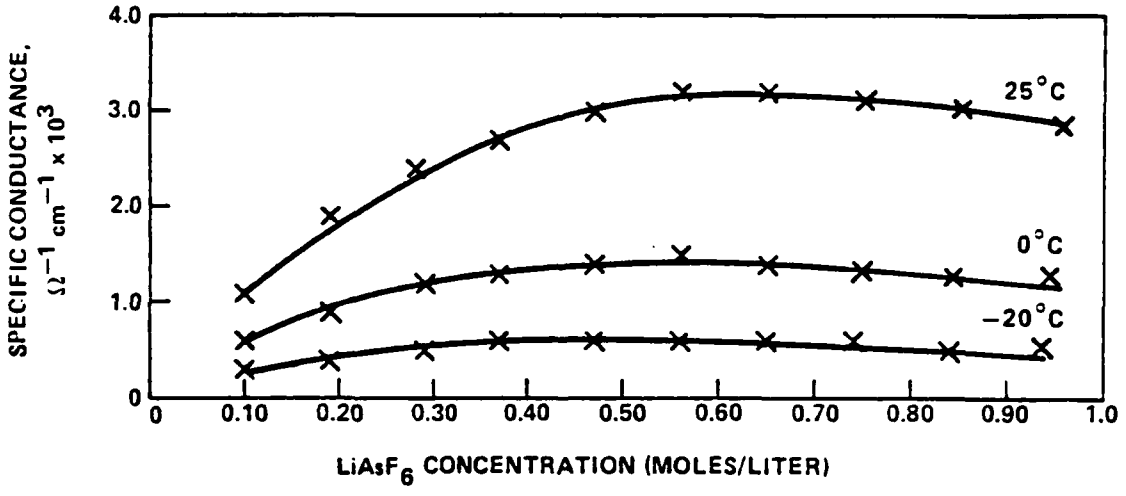


FIGURE 7. SPECIFIC CONDUCTANCE OF 50% 1, 1, 2, 2-TETRACHLOROETHANE, 50% PROPYLENE CARBONATE SOLUTIONS

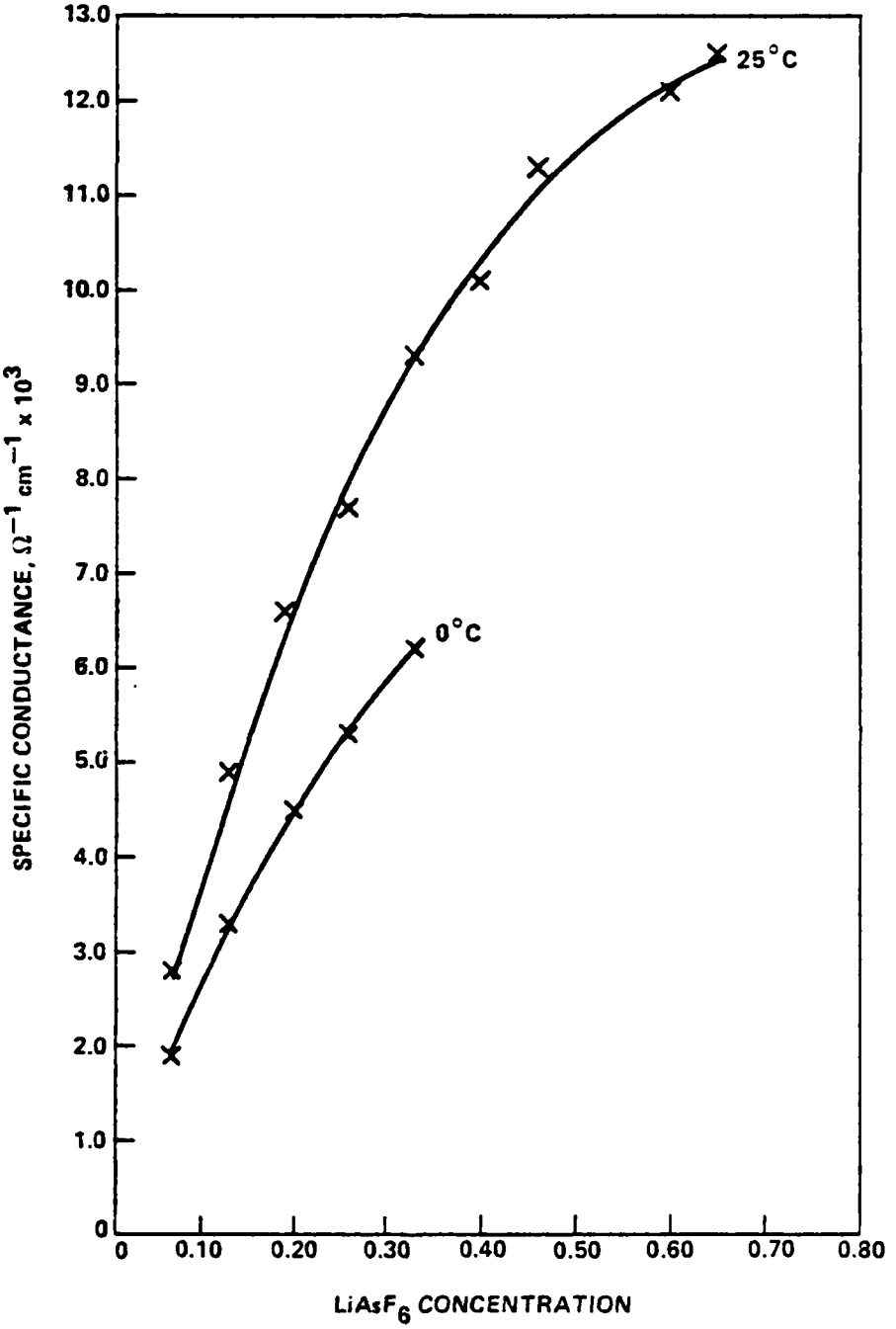


FIGURE 8. SPECIFIC CONDUCTANCE OF 50% 1,1,2,2-TETRACHLOROETHANE, 50% ACETONITRILE SOLUTIONS

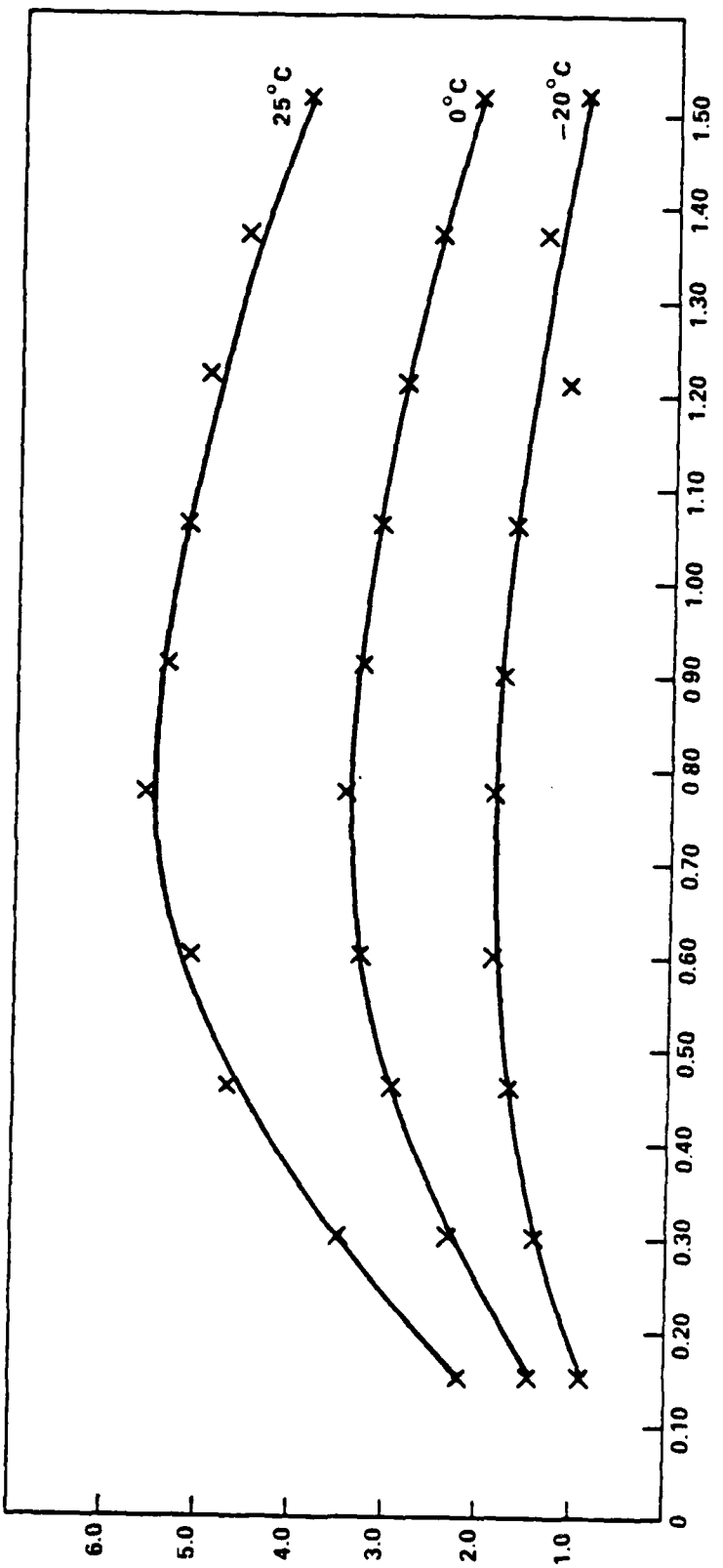


FIGURE 9. SPECIFIC CONDUCTANCE OF 50% 1,1,2,2-TETRACHLOROETHANE, 50% DIMETHYL SULFITE SOLUTIONS

TABLE 1. CONDUCTANCE OF $LiAsF_6$ IN 50% 1,1,2,2-TETRACHLOROETHANE, 50% METHYL FORMATE

SALT CONCENTRATION (MOLES/LITER)	SPECIFIC CONDUCTANCE, $\Omega^{-1}\,cm^{-1} \times 10^3$		
	−20°C	0°C	25°C
0.200	1.27	1.61	1.57
0.400	2.80	3.72	4.28
0.600	4.17	5.50	6.89
0.800	4.87	6.83	9.05
1.00	5.88	7.97	11.0
1.20	6.32	8.53	12.0
1.40	6.18	8.80	12.5
1.60	5.19	8.46	12.3
1.80	4.85	7.97	11.8
2.00	4.18	7.24	10.4

TABLE 2. CONDUCTANCE OF $LiAsF_6$ IN 75% 1,1,2,2-TETRACHLOROETHANE, 25% METHYL FORMATE

SALT CONCENTRATION (MOLES/LITER)	SPECIFIC CONDUCTANCE, $\Omega^{-1}\,cm^{-1} \times 10^3$		
	−20°C	0°C	25°C
0.100	0.326	0.375	0.404
0.200	0.741	0.857	0.973
0.300	1.15	1.38	1.58
0.400	1.50	1.84	2.13
0.500	1.77	2.24	2.66
0.600	2.03	2.58	3.22
0.700	2.28	2.82	3.53
0.800	2.28	2.86	3.79
0.900	2.44	3.00	3.85
1.00	2.38	3.02	3.85

TABLE 3. CONDUCTANCE OF $LiAsF_6$ IN 50% 1,1,2,2-TETRACHLOROETHANE, 50% TETRAHYDROFURAN

SALT CONCENTRATION (MOLES/LITER)	SPECIFIC CONDUCTANCE, $\Omega^{-1}\ cm^{-1} \times 10^3$		
	−20°C	0°C	25°C
0.150	0.762	0.945	1.07
0.300	1.39	1.87	2.18
0.450	1.86	2.61	3.24
0.600	2.18	3.30	4.16
0.750	2.58	3.73	4.82
0.900	2.75	3.99	5.30
1.05	2.76	4.06	5.54
1.20	2.63	3.89	5.50
1.35	–	3.85	5.48
1.50	–	3.47	5.30

TABLE 4. CONDUCTANCE OF $LiAsF_6$ IN 75% 1,1,2,2-TETRACHLOROETHANE, 25% TETRAHYDROFURAN

SALT CONCENTRATION (MOLES/LITER)	SPECIFIC CONDUCTANCE, $\Omega^{-1}\ cm^{-1} \times 10^3$		
	−20°C	0°C	25°C
0.0500	0.251	0.336	0.370
0.100	0.550	0.612	0.718
0.150	0.701	0.828	0.949
0.200	0.909	1.07	1.27
0.250	1.14	1.37	1.51
0.300	1.35	1.57	1.78
0.350	1.49	1.76	2.01
0.400	1.61	1.88	2.26
0.450	1.81	2.12	2.40
0.500	1.96	2.24	2.55

TABLE 5. CONDUCTANCE OF $LiAsF_6$ IN 50% 1,1,2,2-TETRACHLOROETHANE, 50% ETHYL ACETATE

SALT CONCENTRATION (MOLES/LITER)	SPECIFIC CONDUCTANCE, $\Omega^{-1}\,cm^{-1} \times 10^3$		
	−20°C	0°C	25°C
0.140	0.498	0.697	0.851
0.280	1.04	1.49	1.96
0.420	1.55	2.24	3.13
0.560	1.96	3.04	4.14
0.700	2.52	3.63	5.07
0.840	2.86	4.04	5.76
0.980	3.02	4.30	6.11
1.12	2.93	4.28	6.18
1.26	2.23	3.93	5.88
1.40	1.82	3.19	5.42

TABLE 6. CONDUCTANCE OF $LiAsF_6$ IN 50% 1,1,2,2-TETRACHLOROETHANE, 50% γ-BUTYROLACTONE

SALT CONCENTRATION (MOLES/LITER)	SPECIFIC CONDUCTANCE, $\Omega^{-1}\,cm^{-1} \times 10^3$		
	−20°C	0°C	25°C
0.0940	0.383	0.720	1.28
0.188	0.626	1.20	2.20
0.282	0.774	1.53	2.84
0.376	0.898	1.80	3.45
0.470	1.06	2.00	3.81
0.564	1.17	2.17	4.10
0.658	1.15	2.19	4.30
0.752	1.09	2.08	4.40
0.846	–	–	4.42
0.940	–	–	4.38

TABLE 7. CONDUCTANCE OF $LiAsF_6$ IN 50% 1,1,2,2-TETRACHLORGETHANE, 50% PROPYLENE CARBONATE

SALT CONCENTRATION (MOLES/LITER)	SPECIFIC CONDUCTANCE, $\Omega^{-1}\ cm^{-1} \times 10^3$		
	-20°C	0°C	25°C
0.0935	0.266	0.587	1.14
0.187	0.444	0.915	1.88
0.280	0.521	1.16	2.42
0.374	0.603	1.34	2.77
0.408	0.610	1.38	3.00
0.561	0.599	1.44	3.16
0.654	0.586	1.39	3.17
0.748	0.542	1.33	3.14
0.842	0.572	1.32	3.07
0.935	0.547	1.28	2.90

TABLE 8. CONDUCTANCE OF $LiAsF_6$ IN 50% 1,1,2,2- TETRACHLOROETHANE, 50% ACETONITRILE

SALT CONCENTRATION (MOLES/LITER)	SPECIFIC CONDUCTANCE, $\Omega^{-1}\ cm^{-1} \times 10^3$	
	0°C	25°C
0.0653	1.89	2.78
0.131	3.27	4.89
0.196	4.44	6.63
0.261	5.34	7.75
0.327	6.18	9.32
0.392	–	10.1
0.457	–	11.3
0.522	–	11.6
0.588	–	12.1
0.653	–	12.6

TABLE 9. CONDUCTANCE OF $LiAsF_6$ IN 50% 1,1,2,2-TETRACHLOROETHANE, 50% DIMETHYL SULFITE

SALT CONCENTRATION (MOLES/LITER)	SPECIFIC CONDUCTANCE, $\Omega^{-1}\ cm^{-1} \times 10^3$		
	−20°C	= 0°C	25°C
0.152	0.878	1.44	2.23
0.304	1.41	2.33	3.51
0.456	1.72	2.93	4.66
0.608	1.86	3.27	5.07
0.760	1.89	3.45	5.61
0.911	1.82	3.36	5.39
1.06	1.70	3.14	5.20
1.22	1.15	2.90	4.95
1.37	1.32	2.56	4.58
1.52	1.04	2.18	4.04

TABLE 10. CORRELATION OF HIGHEST OBTAINABLE $LiAsF_6$ ELECTROLYTE CONDUCTANCES WITH CALCULATED VALUES OF DIELECTRIC CONSTANT AND VISCOSITY OF 50% TETRACHLOROETHANE/COSOLVENT MIXTURES AT 25°C

COSOLVENT	COSOLVENT		MIXTURE		HIGHEST CONDUCTANCE
	ϵ^*	η^*,cP	ϵ	η,cP	$\kappa(\Omega^{-1}\ cm^{-1} \times 10^3)$
AN	35.95	0.3409	22.1	0.598	12.6
MF	8.05(20°C)	0.3298	8.18	0.622	12.5
EtOAc	6.02**	0.4546**	7.11	0.902	6.18
DMSI	22.5	0.7715	15.3	1.156	5.61
THF	7.39(23.3°C)	0.46	7.80	0.843	5.50
γBL	39.1	1.72	23.6	1.79	4.42
PC	64.4†	2.53†	36.3	2.20	3.17
1,1,2,2,-TCE	8.2†	1.844†			

* Reference 3 unless otherwise noted

** Reference 4

† Reference 5

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