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PROPERTIES OF NONAQUEOUS ELECTROLYTES

THIRD QUARTERLY REPORT

(20 December 1966 to 19 March 1967)

By
Rudolf Keller
James N. Foster
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Prepared For

National Aeronautics and Space Administration

Contract NAS3-8521

Rocketdyne
A Division of North American Aviation, Inc.
Canoga Park, California

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Technical Management
NASA Lewis Research Center
Cleveland, Ohio
Space Power Systems Division
Mr. Robert B. King

Rocketdyne
A Division of North American Aviation, Inc.
Canoga Park, California

FOREWORD

This report was prepared under G.O. 8852 in compliance with Article VI and Paragraph B of Contract NAS3-8521 for the National Aeronautics and Space Administration, Lewis Research Center, Cleveland, Ohio. The work was conducted in the Chemical and Material Sciences Department of Rocketdyne's Research Division, during the period 20 December 1966 through 19 March 1967.

ABSTRACT

A complete vapor phase chromatographic analysis of acetonitrile was conducted. A method was established for the routine characterization of purified solvent batches of propylene carbonate, dimethyl formamide and acetonitrile; the water content of each solvent is determined quantitatively to ±5 to 10 ppm.

Structural studies of electrolytes containing tetramethylammonium hexafluorophosphate, tetramethylammonium fluoride, and $AlCl_3$ were performed by nuclear magnetic resonance.

The physical property studies included measurements of viscosities, conductances, transference numbers, and solubilities of a number of electrolytes.

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SUMMARY

Acetonitrile (AN) was completely analyzed by vapor phase chromatography; no organic impurity at a level greater than 10 ppm was found in the distilled product. A convenient vapor phase chromatographic analysis method is now available to determine the water content in each batch of distilled solvent.

Nuclear magnetic resonance structural studies revealed that tetramethylammonium hexafluorophosphate ($TMA \cdot PF_6$) was present in propylene carbonate (PC) and dimethylformamide (DMF) as TMA^+ and PF_6^- ions. $AlCl_3$ coordinates with DMF molecules at the oxygen site rather than the nitrogen site; between 4 and 8 (probably 6) moles of coordinated solvent are produced per dissolved mole of $AlCl_3$.

Viscosities and conductances were measured for a number of electrolytes containing $TMA \cdot PF_6$, LiCl, and/or $AlCl_3$. Solubilities are listed for such electrolytes, and also for some others containing LiF, TMA·F (tetramethylammonium fluoride), and $CuCl_2$. Transference number determination by the Hittorf method indicated a low transference number for the cation in LiCl/DMF and the presence of a negatively charged copper species in such an electrolyte which also contained $CuCl_2$.

DESCRIPTION OF PROGRESS

PREPARATION OF ELECTROLYTES

Purification of Solvents

Solvents were purified by distillation as described previously (Ref. 1 and 2). A summary of the distillation conditions normally applied is presented in Table 1.

TABLE 1

DISTILLATION PROCEDURES FOR PURIFICATION OF SOLVENTS

Solvent	Grade	Drying Agent	Column	Condition
PC	Practical	CaH_2	Spinning Band	5 to 10 mm Hg; reflux ratio: 30/1
DMF	Spectro quality	Multrathanel	Spinning Band (or Vigreux)	25 mm Hg; reflux ratio: 10/1
AN	Reagent	P_2O_5	Spinning Band	Atmospheric pressure; dry nitrogen; reflux ratio: 25/1

The solvents are usually purified in batches of approximately 500 milliliters. The vapor phase chromatographic analysis method used for routine characterization of the solvents is subsequently described. The analysis results of the individual solvent batches used during the program are presented in Table 2. The water content of the distilled product is normally between 20 and 50 ppm. A content of organic imputities between 0 and 40 ppm is normally found by the vapor phase chromatographic analysis on a Porapak Q column.

TABLE 2

CHARACTERIZATION OF DISTILLED SOLVENT BATCHES

Solvent Code	H_2O Content, ppm	Organics, ppm
PC #2-2	55	20
PC #2-3	25	None*
PC #2-4	20	15
PC #2-5	20	35
PC #2-6	35	None
DMF #1-2	20	140
DMF #3-2	40	None
DMF #4-2	100	None
DMF #3-3	45	35
AN #1-2	40	None
AN #3-1	50	None

*No organic impurity at a level of 10 ppm or greater was detected in the analysis.

Analysis of Acetonitrile by Vapor Phase Chromatography

Two samples of AN, AN #1-2 and AN #3-1, have been completely characterized by vapor phase chromatography. Three columns were employed to detect various species as outlined in Table 3.

TABLE 3

COLUMNS EMPLOYED IN THE CHARACTERIZATION OF ACETONITRILE

Column*	Temperature, C	Detector	Species Characterized
Porapak Q	150	Cross section	Water
Apiezon L	40	Flame ionization	Lower boiling materials
Apiezon L	100	Flame ionization	Higher boiling materials
Carbowax 20M	40	Flame ionization	Lower boiling materials
Carbowax 20M	100	Flame ionization	Higher boiling materials
Porapak Q	140	Flame ionization	Acrylonitrile and very volatile materials

*Hydrogen was used as the carrier gas, its flow rate was 25 cc/min

A sample of AN containing 120 ppm water was analyzed with a Porapak Q column; a chromatogram is presented in Fig. 1. Water is not completely separated from AN under the conditions utilized for the determination of water in PC and DMF. More complete separation can be obtained by decreasing either the column temperature or the sample size. Decreasing the column temperature causes the water peak to tail; decreasing the sample size decreases the amount of water being determined.

Under the experimental conditions employed, the water peak tails somewhat and does not return to the baseline before the initial portion of the AN peak. However, the water peak can be extrapolated to the baseline and its area measured. Under these conditions, the optimum sensitivity is obtained.

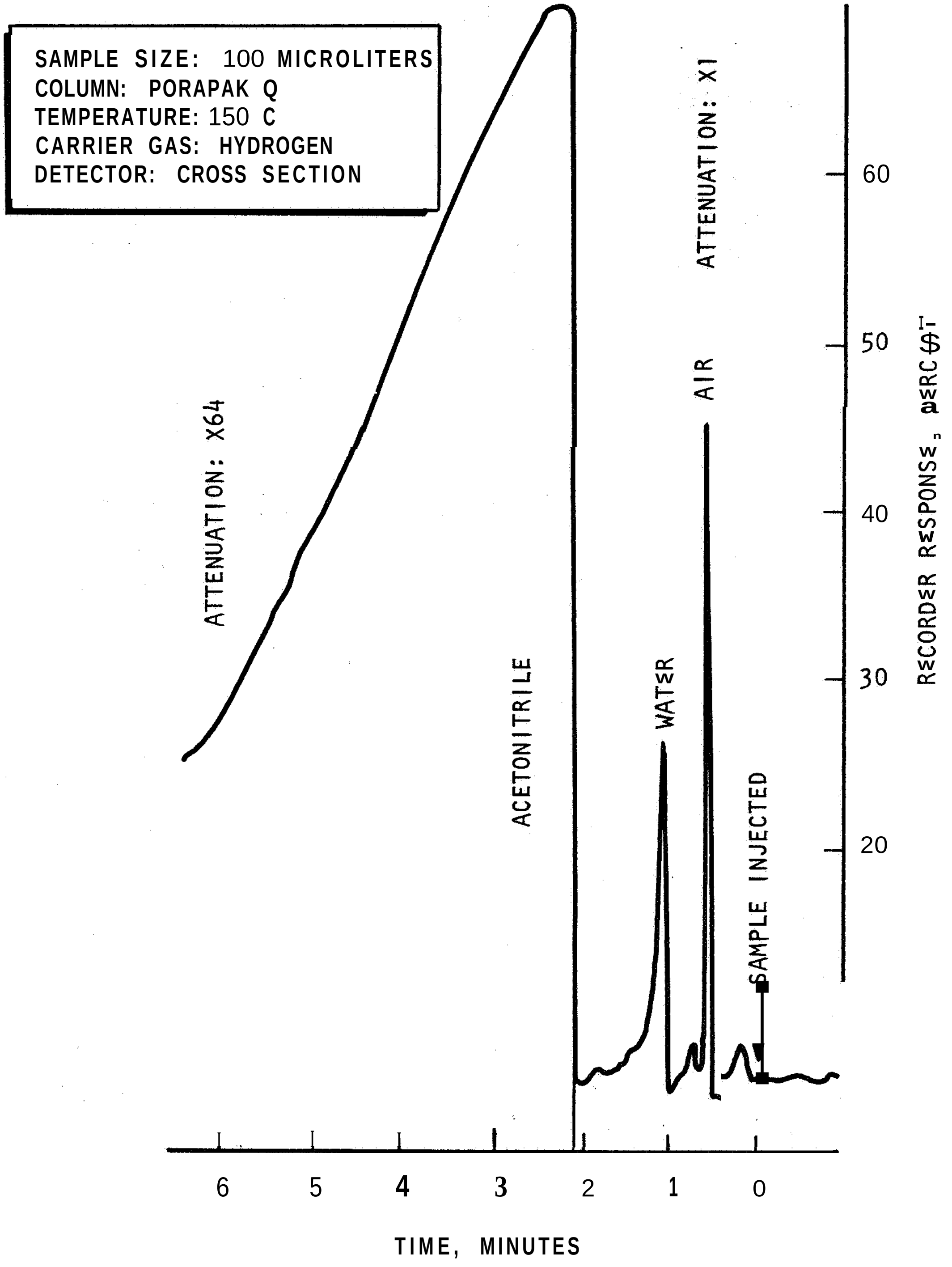

Figure 1. Initial Portion of Chromatogram of Acetonitrile Containing 120 ppm Water

Known amounts of water were added to a sample of AN and the areas of the resulting water peaks were measured. The area of the water peak for various additions of water is presented in Table 4. The response for water is 112 ppm/cm^2.

TABLE 4

AREA OF WATER PEAK FOR SOLUTIONS OF ACETONITRILE CONTAINING VARIOUS AMOUNTS OF WATER

H_2O added, ppm	H_2O peak area, cm^2
0*	1.1
196	3.0
408	4.9
645	6.7
1130	10.8

*The AN to which the water was added contained some water. Based on the response of 112 ppm/cm^2, the AN contained 120 ppm water.

This response corresponds to 8.7 micrograms H_2O/cm^2. This value is greater than the responses found for PC (6.0) and DMF (6.4). The difference is probably caused by the fact that the extrapolation of the water peak did not include all of the peak area under the acetonitrile peak. However, the assumed area of the water peak was found to be proportional to the water content of the AN and can be used to determine the water concentration in AN samples. The lower limit for the determination of water in AN appears to be 10 ppm.

Three columns were used for the characterization of organic impurities in AN. Carbowax 20M and Apiezon L columns were used for the reasons discussed in a previous report (Ref. 2) under the characterization of DMF. A third column, a 6-foot Porapak Q column, was used to determine acrylonitrile and other possible volatile impurities in AN. The latter column gives an excellent separation of AN and acrylonitrile. The retention times at 140 C of AN and acrylonitrile are 6.5 and 10.5 minutes, respectively. This separation is important because acrylonitrile is usually found as an impurity in AN. Furthermore, since AN is eluted very rapidly from the Apiezon L and Carbowax 20M columns, good separation between AN and any volatile impurities may not occur. Because AN is not eluted as rapidly on Porapak Q, volatile impurities should be separated.

Some chromatograms of purified AN are presented in Fig. 2 through 4; the chromatograms obtained with AN #3-1 were virtually identical. Two additional chromatograms were run at 100 C on the Carbowax 20M and Apiezon L columns. Each chromatogram was recorded for 60 minutes to allow any higher boiling impurities to be eluted. No peaks were evident on any of the chromatograms.

If an impurity were present at the 100 ppm level, a peak approximately one-fifth the size of the AN should be observed on the chromatogram at the X0.2 attenuation. Because no such peak appeared, there are no impurities in the AN with concentrations greater than 100 ppm.

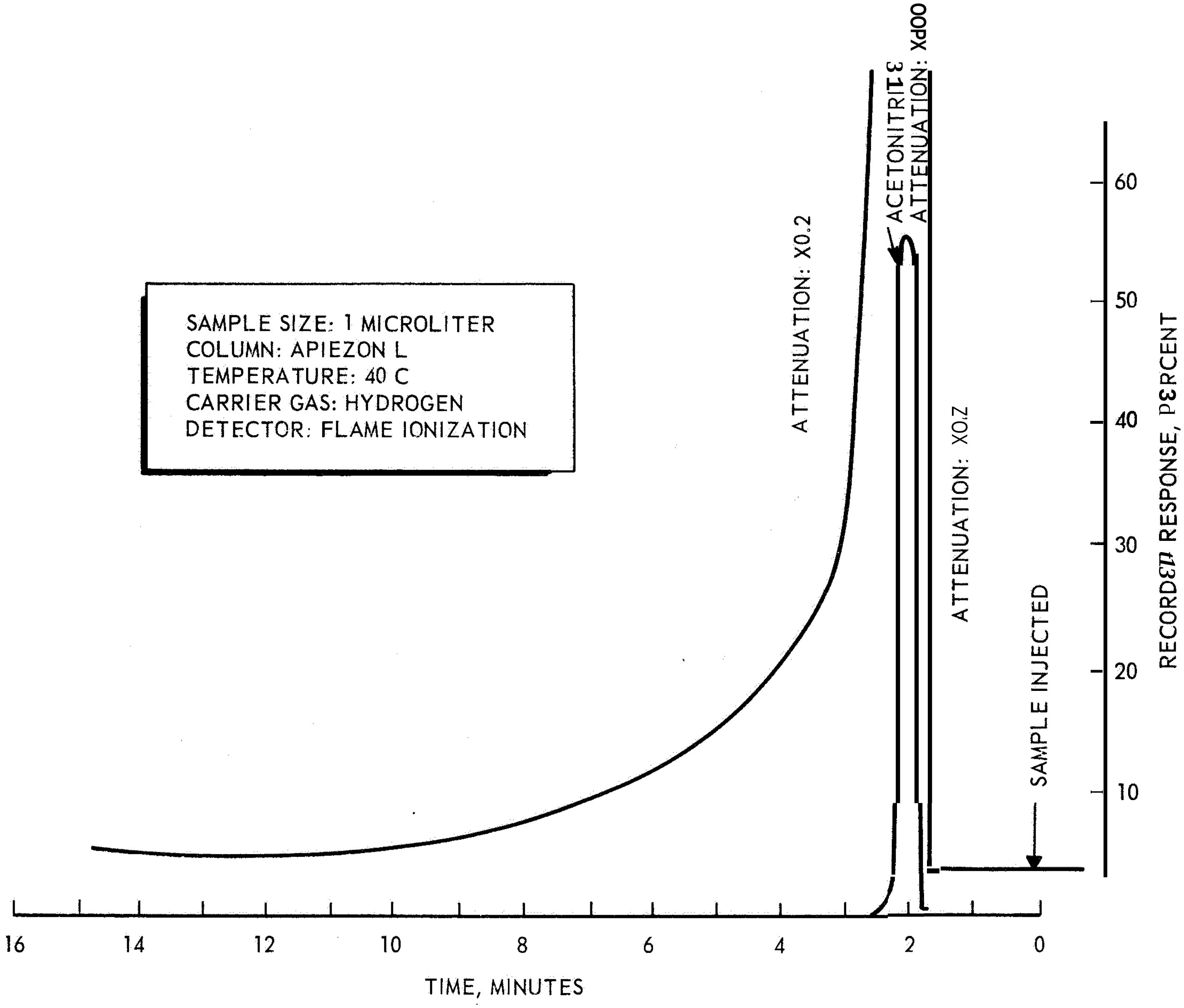

Figure 2. Chromatogram of Acetonitrile, AN #1-2, on Apiezon L

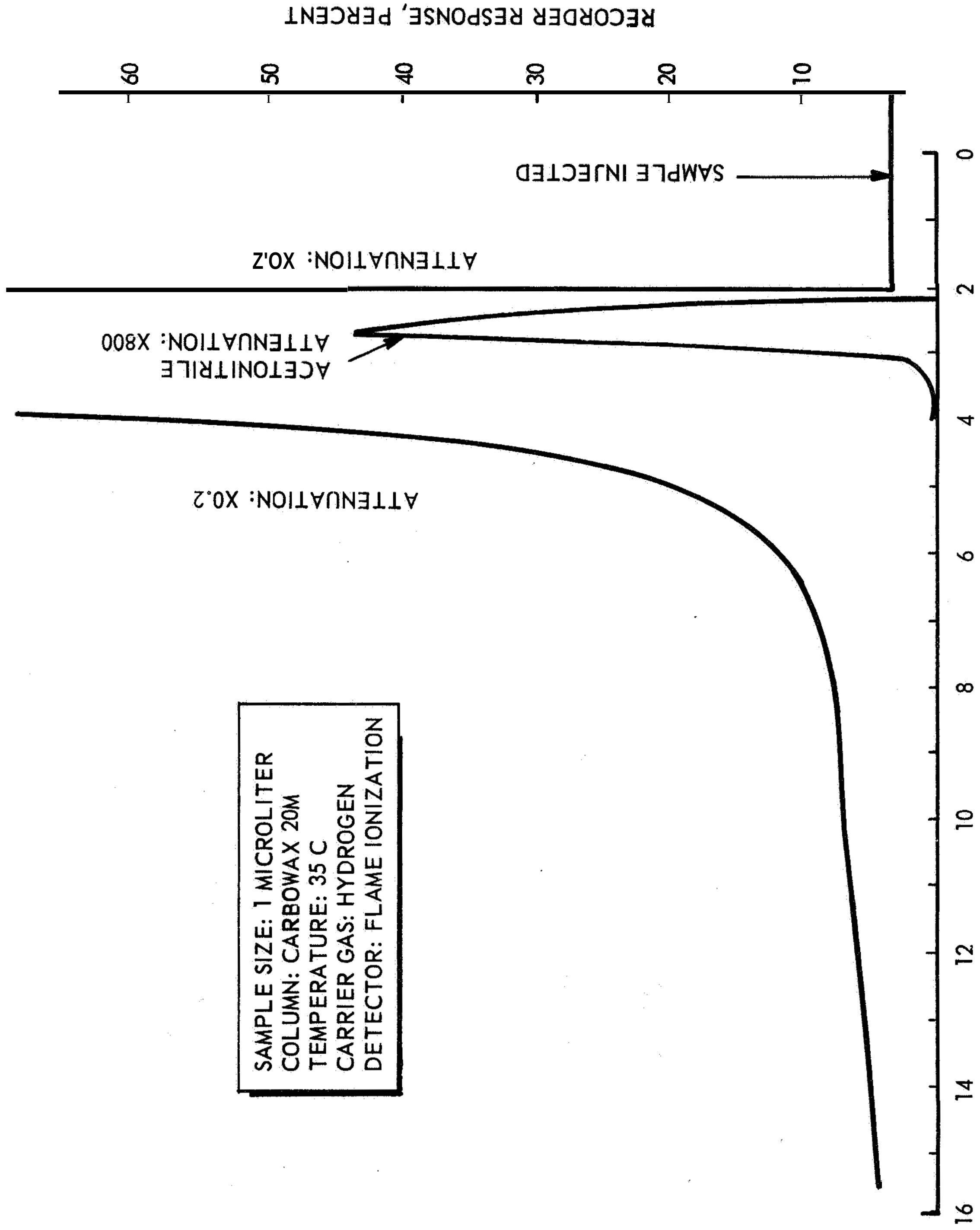

Figure 3. Chromatogram of Acetonitrile, AN #1-2, on Carbowax 20M

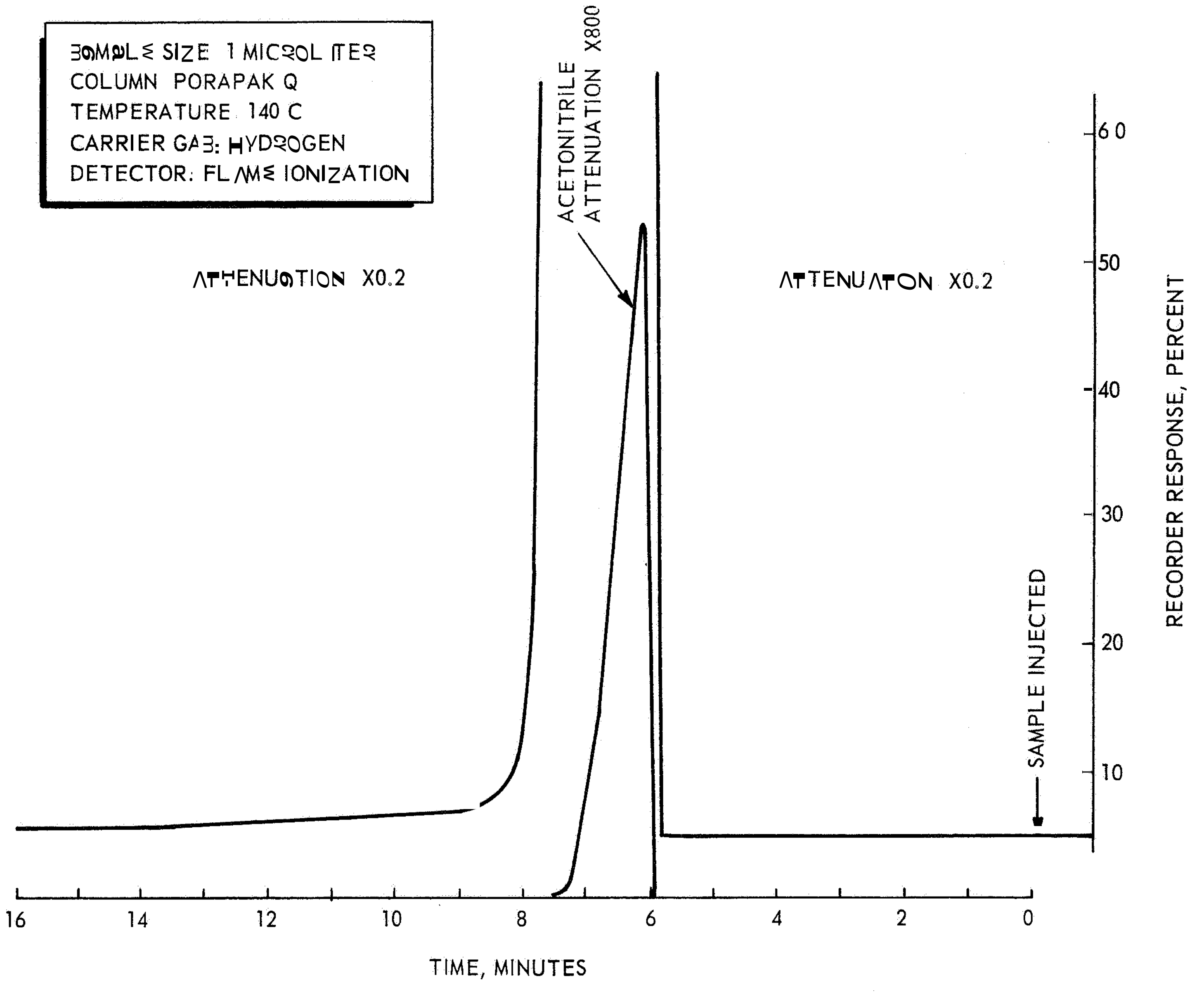

Figure 4. Chromatogram of Acetonitrile, AN #1-2, on Porapak Q

Routine Characterization of Distilled Solvents

Vapor phase chromatographic procedures for the determination of water and organic impurities in all three solvents (PC, DMF, and AN) have been developed. Table 5 presents the experimental parameters for the routine analysis applied to monitor the quality of the distilled solvents.

TABLE 5

EXPERIMENTAL PARAMETERS AND RESPONSE FOR THE ROUTINE DETERMINATION OF WATER IN PROPYLENE CARBONATE, DIMETHYL FORMAMIDE, AND ACETONITRILE

Conditions*	Solvent		
	PC	DMF	AN
Sample Size, microliters	100	100	100
Column Dimension	3/16 inch by 6 feet	3/16 inch by 6 feet	3/16 inch by 6 feet
Column Packing	Porapak Q	Porapak Q	Porapak Q
Column Temperature, C	165	165	150
Injector Temperature, C	175	175	175
Carrier Gas	Hydrogen	Hydrogen	Hydrogen
Flowrate, cc/min	25	25	25
Detector	Cross Section	Cross Section	Cross Section
Detector Temperature, C	165	165	165
Response (by weight), ppm/cm^2	50	67	112
Response, micrograms H_2O/cm^2	6.0	6.4	8.7

*Chromatograph: Aerograph 660, Wilkens Instrument & Research, Inc.
Recorder: Leeds & Northrup Speedomax G; 750 microvolts full scale, 1/2 inch per minute

This routine analysis does not constitute a complete characterization but furnishes data in regard to the most important impurity, water, as well as the most likely organic impurities. The lower limit for the quantitative determination of water is approximately 5 ppm in PC and in DMF, and 10 ppm in AN.

NUCLEAR MAGNETIC RESONANCE STRUCTURAL STUDIES

$TMA \cdot PF_6$* and $TMA \cdot F$** in Propylene Carbonate and Dimethyl Formamide

It is expected that dissolving $TMA \cdot PF_6$ and $TMA \cdot F$ would result in the formation of $(TMA)^+$ and $(PF_6)^-$ and F^- ions, respectively. To verify this, both the F^{19} resonance and H^1 resonance.were investigated in PC and DMF solution containing these solutes; the solutions were identical to the diluted solutions used for solubility determinations (Table 8). The F^{19} resonance in both PC and DMF containing $TMA \cdot PF_6$ is an equal intensity doublet. No other lines were observed. Because P has a spin of 1/2, spin-spin interaction should lead to an equal intensity doublet. Furthermore, all F^{19} in $(PF_6)^-$ are chemically equivalent; therefore, complex spectra would be only due to spin-spin interactions. Thus, the finding of only an equal intensity doublet is consistent with the assumption that the ion containing F^{19} is the $(PF_6)^-$ ion.

Because of the relatively low solutility, these lines were rather weak. The even lower solubility of $TMA \cdot F$ in PC and DMF and the smaller number of fluorine atoms per solute anion resulted in no F^{19} line being observed for these solutions.

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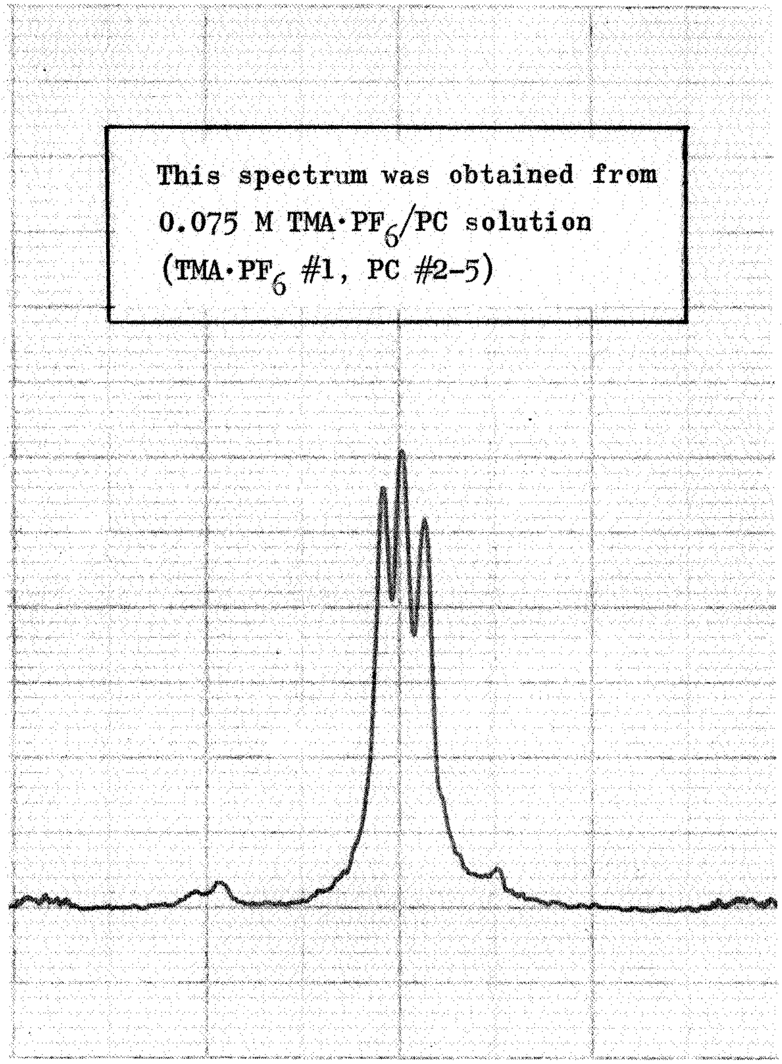
*$TMA \cdot PF_6$ = tetramethylammonium hexafluorophosphate
**$TMF \cdot F$ = tetramethylammonium fluoride

In contrast, the proton line due to the addition of TMA·PF$_6$ and TMA·F was observed in all solutions. It should be noted that TMA·F contains the same number of protons as TMA·PF$_6$. Therefore, the low solubility of TMA·F did not prevent the proton resonance line from being observed as was the case with F^{19} resonance. In all cases the H^1 line consisted of an equal intensity triplet with a splitting of less than 1 Hz as shown in Fig. 5. The H^1 resonance has been observed for NH$_3$ (Ref. 3) and consists of an equal intensity triplet with the splitting of 46 ±2 Hz between adjacent lines. This pattern is explained on the basis that all H^1 are equivalent and split into a triplet by virtue of a spin-spin interaction with N^{14} which has a spin of 1. By analogy, because all H^1 are equivalent in a (TMA)$^+$ ion and because interaction with N^{14} would yield a triplet as observed, the observations are consistent with what is expected for a (TMA)$^+$ ion. The splitting is much less for (TMA)$^+$ than for NH$_3$ because the splitting must bridge another bond due to the carbon in the methyl groups.

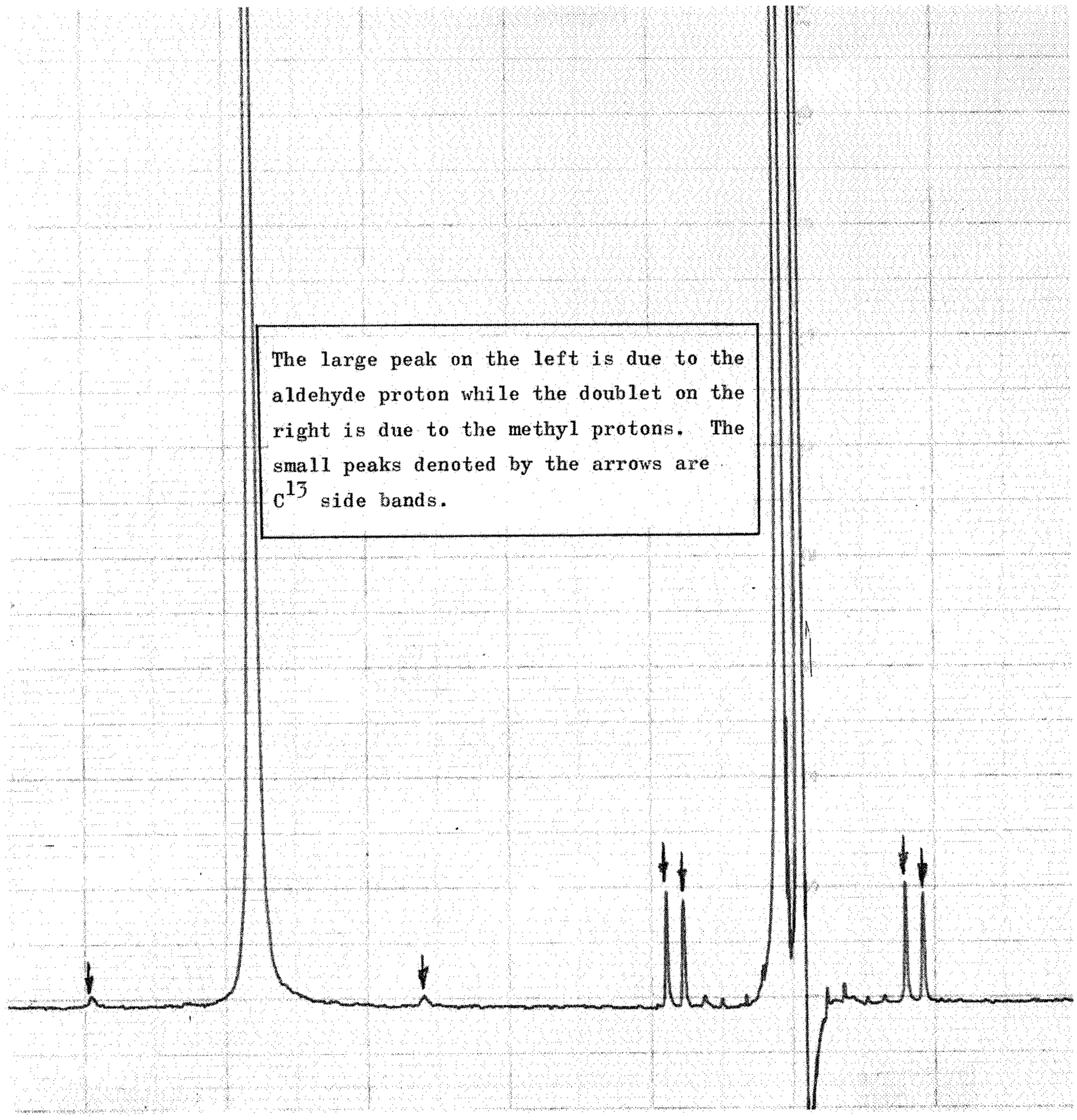
AlCl in Dimethyl Formamide

To date, the nuclear magnetic resonance structural studies based upon observation of resonance patterns of the solvent have been concentrated on observations in DMF containing AlCl$_3$. The H^1 spectrum of pure DMF (Fig. 6) is relatively simple. However, under closer scrutiny the spectrum due to the methyl protons is more complex. Effort has been focused on AlCl$_3$ in DMF because of the observation of displacement of the H^1 lines by coordination with AlCl$_3$. This has been previously reported by Fratiello et al. (Ref. 4). A spectrum of DMF containing 0.0528 M AlCl$_3$ is shown in Fig. 7. The downfield shift of the aldehyde proton is greater than that of the methyl protons. Furthermore, the



This spectrum was obtained from 0.075 M TMA·PF_6/PC solution (TMA·PF_6 #1, PC #2-5)

Figure 5 . H^1 Spectrum for $(TMA)^+$



The large peak on the left is due to the aldehyde proton while the doublet on the right is due to the methyl protons. The small peaks denoted by the arrows are C^{13} side bands.

Figure 6. H1 Spectrum for Pure DMF

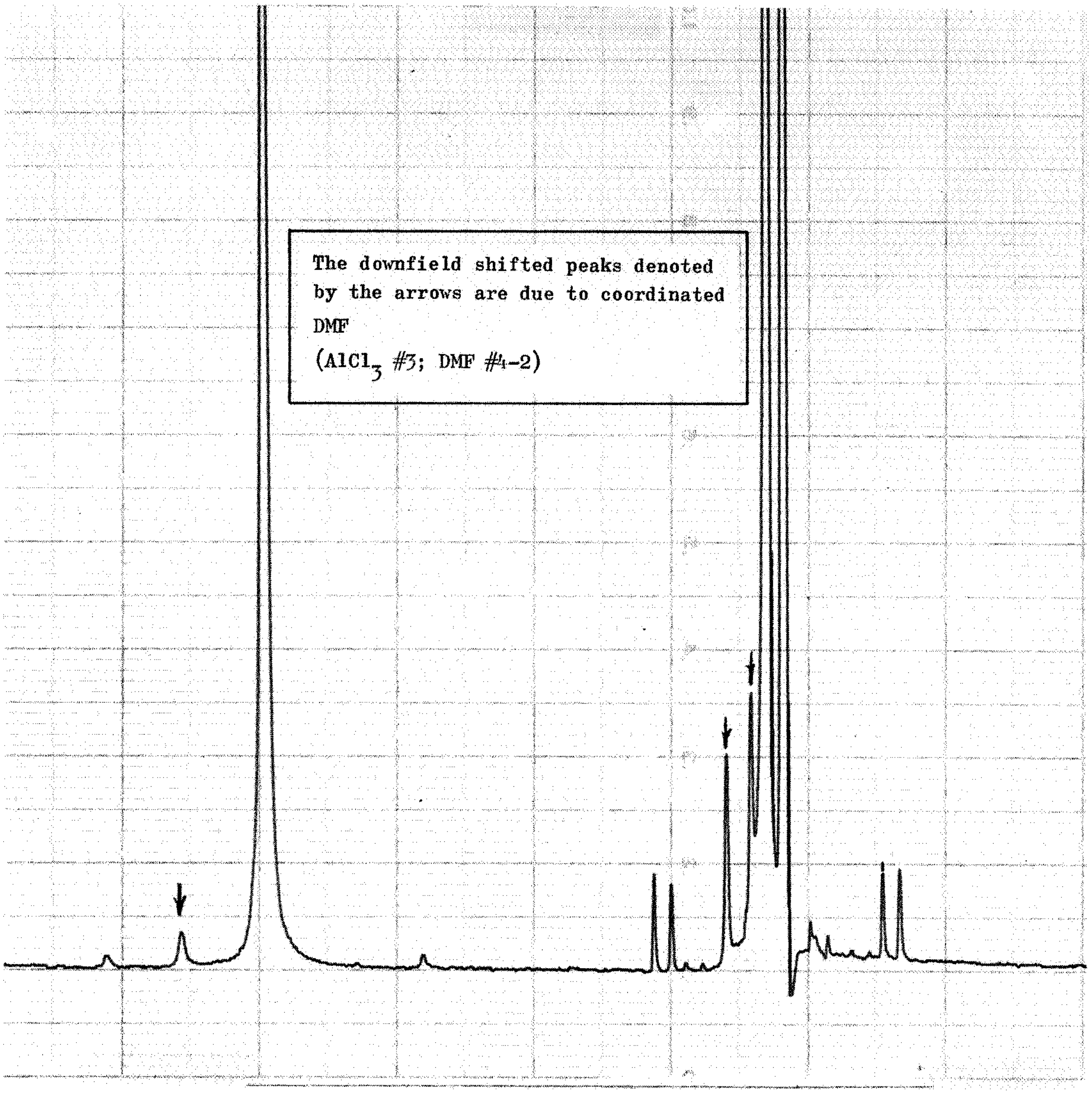


Figure 7. H^1 Spectrum for 0.0528 M $AlCl_3$/DMF

methyl protons are not shifted the same amount. These shifts have been measured and the results. 51.8 Hz for the aldehyde proton and 25.5 Hz and 21.2 Hz for the methyl protons, are in general agreement with the results of Ref. 4. The interpretation of these results, presented in Ref. 4, is that $AlCl_3$ coordinates the DMF molecules at the oxygen site rather than the nitrogen site.

The H^1 resonance pattern obtained from DMF containing 1 M LiCl is the same as for pure DMF. No downfield peaks are observed. Thus, either LiCl does not coordinate, or the effect upon the electron structure is too small to be observed. Furthermore, the H^1 resonance pattern in DMF containing both 0,0528 M $AlCl_3$ and 0.0528 M LiCl is the same as that found in 0.0528 M $AlCl_3$/DMF.

More detailed study of the methyl doublet in DMF revealed that each peak of the doublet is itself a doublet as shown in Fig. 8 . The complete methyl proton spectrum of 0.0528 $AlCl_3$/DMF is shown in Fig. 9 . The secondary splitting is 0.63 Hz in both pure DMF and the uncoordinated DMF in 0.0528 M $AlCl_3$/DMF. However, the splitting in the coordinated DMF is 0.85 Hz. This difference has not yet been completely interpreted. The splitting is probably due to spin-spin interaction with the aldehyde proton. When the DMF is coordinated, the chemical environment of the methyl locations is altered as is shown by the fact that each line of the methyl doublet is shifted downfield. It is, therefore, reasonable to expect that the spin-spin coupling in the coordinated DMF is different from the uncoordinated DMF.

The ratio of the intensity of the coordinated DMF peaks to the uncoordinated DMF peaks is a measure of the coordination number. In an attempt to determine how many moles of DMF are coordinated by 1 mole of $AlCl_3$, the

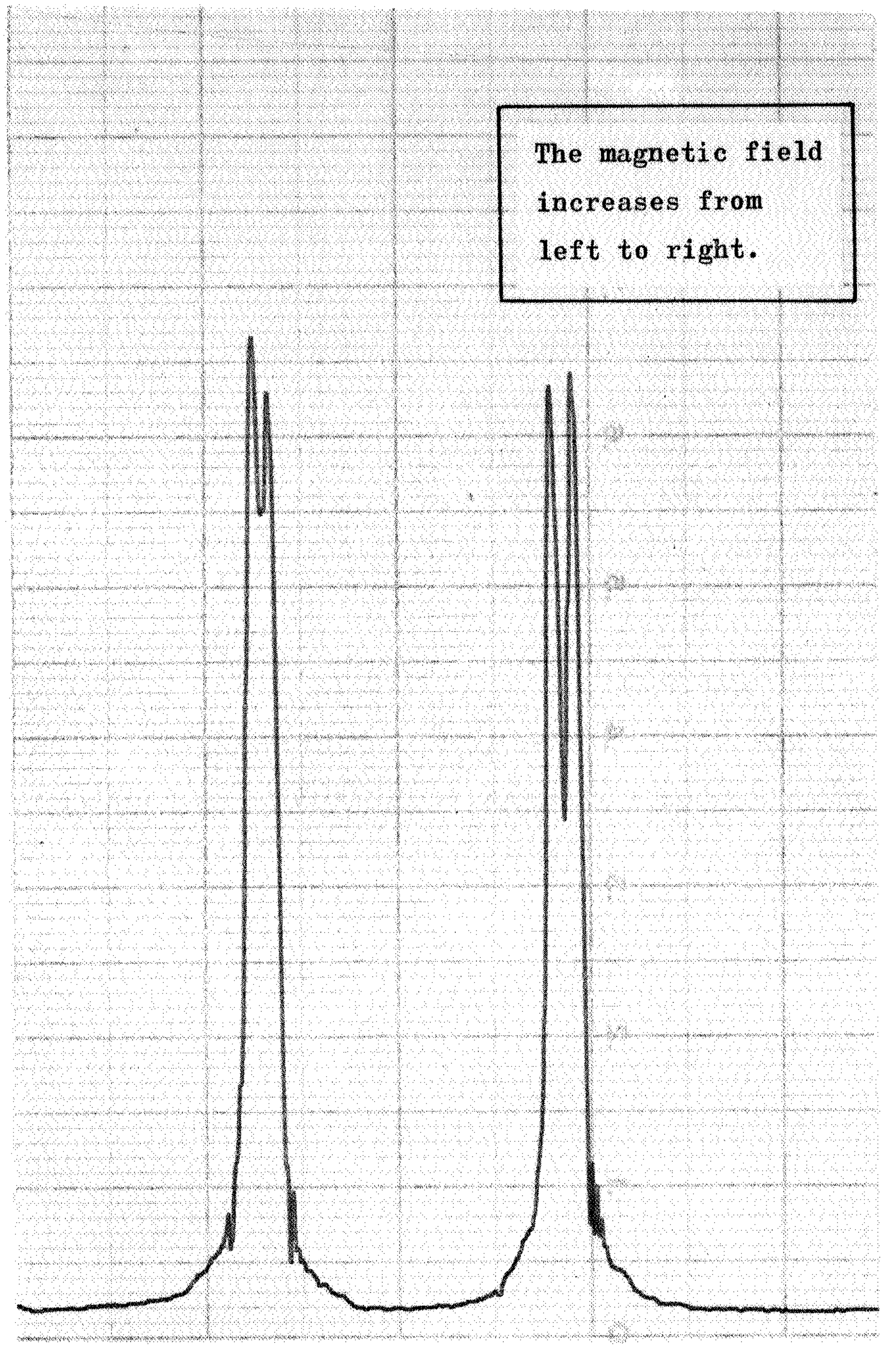


Figure 8. H^1 Spectra, Methyl Doublet at High Resolution, in Pure DMR,

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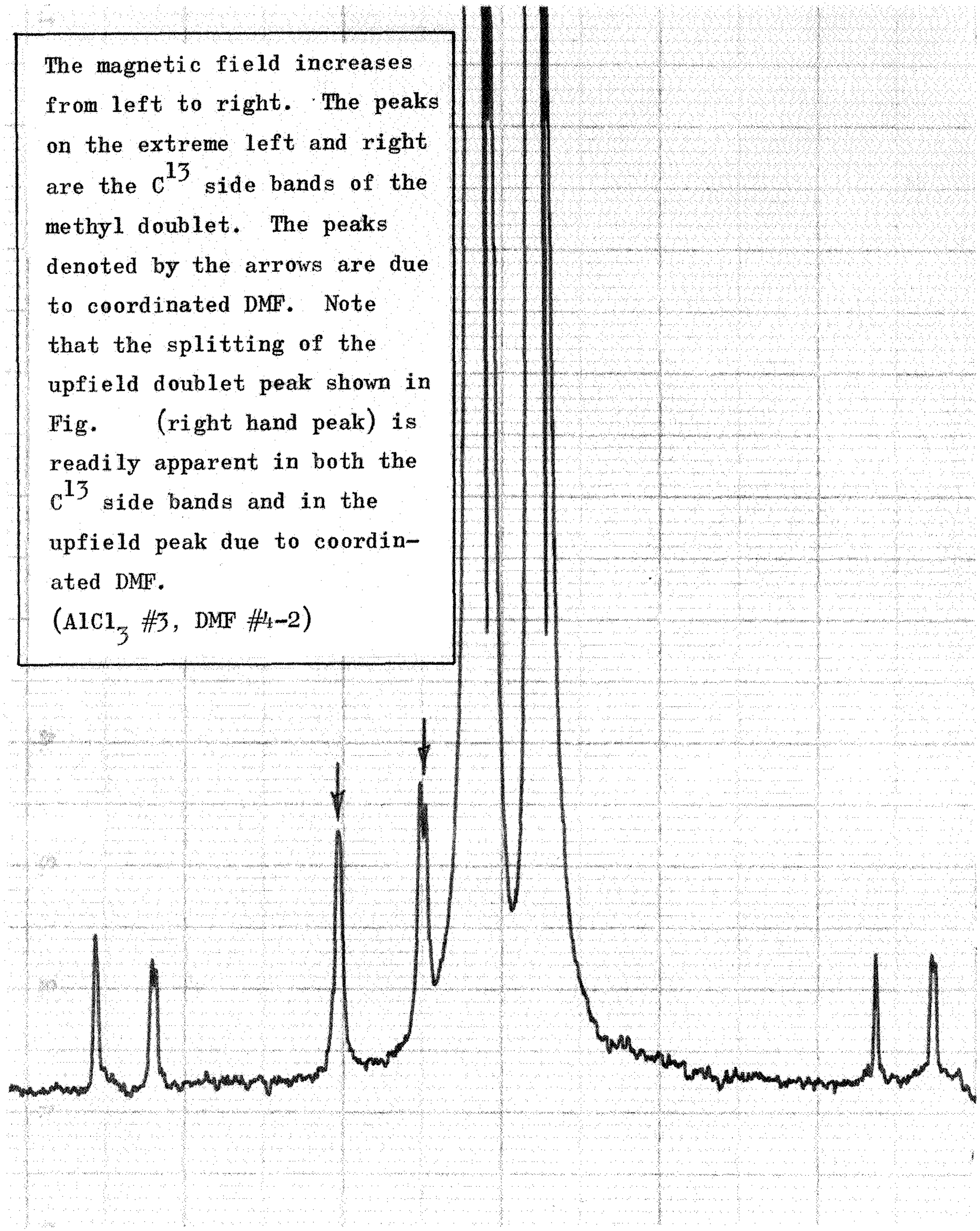

The magnetic field increases from left to right. The peaks on the extreme left and right are the C^{13} side bands of the methyl doublet. The peaks denoted by the arrows are due to coordinated DMF. Note that the splitting of the upfield doublet peak shown in Fig.   (right hand peak) is readily apparent in both the C^{13} side bands and in the upfield peak due to coordinated DMF.
($AlCl_3$ #3, DMF #4-2)

Figure 9. H^1 Spectrum of Methyl Protons in 0.0528 M $AlCl_3$/DMF

areas of the uncoordinated lines were compared with the areas of the coordinated DMF lines. This comparison is subject to rather large errors because the coordinated lines are relatively weak and a greater fraction of the signal is lost in the noise. This would lead to an underestimate of the coordination number. The area comparison yields a coordination number of approximately 4 assuming a one to one correspondence of the amount of $AlCl_3$ dissolved in solution to the coordinated peak intensity. Because of the errors involved, this is interpreted as showing that the coordination number is greater than 4. Judging from the line intensity, the error can not be as high as 100 percent; therefore, the coordination number must be less than 8. Thus, the results suggest the most likely coordination number to be 6, which is not an unreasonable result.

PHYSICAL PROPERTY DETERMIMATIONS

Viscosity Measurements

The results of viscosity measurements made during this report period are presented in Table 6. The experimental procedure was described previously (Ref. 2). The solutions were prepared in a dry box and brought to temperature equilibrium while enclosed in a volumetric flask with a polyethylene bag containing dry nitrogen as an additional protective seal (for saturated solutions the handling procedure was the same as described in the Solubility Measurements section). The solutions were exposed to the atmosphere only while viscosity and density measurements were being made.

The 'densities necessary for these determination were measured using a Christian Becker Chainomatic Density Balance. This instrument allows rapid and accurate measurements of densities to three decimal points.

TABLE 6

VISCOSITY DETERMINATIONS AT 25 C

Solute	Concentration	Solvent	Density, gm/cc	Viscosity, millipoise
None		PC #2-6	1.203	24.8
None		AN #1-2	0.777	3.36
$AlCl_3$ #3*	1.03 M	PC #2-6	1.257	57.2
LiCl #2 +$AlCl_3$ #3	Saturated 1.03 M	PC #2-6	1.257	71.6
$AlCl_3$ #3 +LiCl #2	Saturated 1.00 M	DMF #3-2	0.984	22.8
TMA·PF_6 #1	Saturated	PC #2-4	1.209	26.6
TMA·PF_6 #1	Saturated	DMF #4-2	0.964	8.99
TMA·PF_6 #1	Saturated	AN #1-2	0.787	3.57

*All solutions of $AlCl_3$/PC and LiCl+$AlCl_3$/PC have a brownish tinge when prepared very slowly with stirring.

For example, the density of pure PC obtained by the pycnometer technique was 1.200 gm/ml, while the density obtained with the density balance was 1.203. This accuracy is entirely adequate for viscosity measurements.

Solubility Measurements

The results of solubility measurements made during this report period are presented in Table 7. Saturated solutions were prepared in a dry box by adding an excess of salt to the solvent of interest. The flasks were then sealed with a glass stopper and the neck of the flask enclosed in a polyethylene bag containing dry nitrogen. The flasks were removed from the dry box and placed in an 80 C bath for several hours and then allowed to equilibrate in the constant temperature baths at 25 and 60 C, respectively, for several days.

In general, the solubilities of the chlorides were determined by dilution of 10-cc samples of saturated solution with distilled water and titrating with standard $AgNO_3$.

The lithium fluoride solutions were analyzed for lithium content by atomic absorption spectroscopy. The procedure consisted of diluting the saturated solution with solvent and comparing its atomic absorption spectra with calibration curves obtained with known solutions of LiCl or $LiClO_4$ in the corresponding solvent. No response indicating lithium was obtained with LiF/PC; the lowest detectable value was estimated to be 5×10^{-6} molar. This value is lower than the values of 6×10^{-5} molar (Ref. 5) and 2×10^{-5} molar (Ref. 1) which were estimated from conductivity data.

TABLE 7

SOLUBILITY DETERMINATIONS

Solute	Solvent	Temperature, C	Procedure	Solubility, molar
LiCl #2	PC #2-2	25	Chloride Titration	3.8×10^{-2}
		60	Chloride Titration	3.1×10^{-2}
LiCl #2	1 2 M $AlCl_3$/PC #2-[illegible]	25	Chloride Titration	6.6×10^{-1}
		60	Chloride Titration	7.5×10^{-1}
$AlCl_3$ #3	1 M LiCl/DMF #1-4	25	Chloride Titration	8.8×10^{-2}
LiCl #2	AN #1-2	25	Chloride Titration	2.55×10^{-2}
		60	Chloride Titration	1.42×10^{-2}
LiF #2	PC #2-4	25	Atomic Absorption	$< 5 \times 10^{-6}$
		60	Atomic Absorption	$< 5 \times 10^{-6}$
LiF #2	DMF #1-2	25	Atomic Absorption	3.18×10^{-5}
		60	Atomic Absorption	5.34×10^{-5}
LiF #2	AN #1-2	25	Atomic Absorption	2.2×10^{-5}
		60	Atomic Absorption	3.2×10^{-5}
$TMA \cdot PF_6$ #1	PC #2-4	25	Nuclear Magnetic Resonance	1.5×10^{-1}
		60	Nuclear Magnetic Resonance	2.2×10^{-1}

TABLE 7

(Concluded)

Solute	Solvent	Temperature, C	Procedure	Solubility, molar
MA F_6 #1	DMF #4-2	25	Nuclear Magnetic Resonance	2.4×10^{-1}
		60	Nuclear Magnetic Resonance	3.6×10^{-1}
TMA F_6	AN #1-2	25	Evaporation to Dryness	1.0×10^{-1}
		60	Evaporation to Dryness	1.8×10^{-1}
MA #1	P< #2-Σ	25	Nuclear Magnetic Resonance	6.5×10^{-2}
TMA #1	DMF #4-2	25	Nuclear Magnetic Resonance	2.8×10^{-2}
#2	P< #2-4	25	Chloride Titration	4.9×10^{-3}
		60	Chloride Titration	4.2×10^{-3}
#2	DMF #1-2	25	Chloride Titration	1.30
		60	Chloride Titration	2.68
#2	1 M LiCl/DMF #4-2	25	Chloride Titration	1.04
		60	Chloride Titration	1.34

The F^{19} and H^1 nuclear magnetic resonances were used to determine the amounts of $TMA \cdot PF_6$ and $TMA \cdot F$ dissolved in DMF and PC. Quantitative analyses were obtained by comparing the line intensity from unknown, diluted samples with that from a sample having a known concentration, The results are shown in Table 8. Comparison of the results obtained by F^{19} and H^1 analysis for $(PF_6)^-$ and $(TMA)^+$ (the assignment of ion species is discussed in this report) ions reveals good agreement for $TMA \cdot PF_6$ dissolved in DME. Although the concentrations obtained for $(TMA)^+$ are consistently lower than the results obtained for $(PF_6)^-$ the difference is within the limits of error (Table 8). For $TMA \cdot PF_6$ dissolved in PC, the agreement is within the limits of error for the saturated solution at 25 C sample but not €or the saturated solution at 60 C sample. The source of this discrepancy is not known. Again the concentrations determined by H^1 analysis are consistently lower than those determined by F^{19} analysis. Because the H^1 lines are more complex than the F^{19} lines, more confidence should be credited to the F^{19} analyses.

In the case of $TMA \cdot PF_6/AN$, preliminary results were obtained by evaporation of 10-cc samples to dryness and weighing the dried salt. These results will be compared with those obtained by nuclear magnetic resonance procedures.

Conductance Measurements

Conductivity values of solutione were determined in Freas cells as described previously (Ref, 1). The conductivity measurements taken during this report period are summarized in Table 9 and are discussed on the following pages.

Results obtained with electrolytes containing LiCl and/or $AlCl_3$ in PC are presented in Tables 10 through 13 and Fig. 10 through 14. In the

TABLE 8

SOLUTE CONCENTRATIONS DETERMINED BY NUCLEAR MAGNETIC RESONANCE

Solvent	Solute	Dilution"	Ion	Concentration of Saturated Solutions, molar
DMF #4-2	$TMA \cdot PF_6$ (0.101 M)		$(PF_6)^-$	
	$TMA \cdot PF_6$ (Saturated at 25 C)	1:1	$(PF_6)^-$	0.262 ±0.018
	$TMA \cdot PF_6$ (Saturated at 60 C)	5:1	$(PF_6)^-$	0.379 ±0.030
PC #2-4	$TMA \cdot PF_6$ (0.0994 M)		$(PF_6)^-$	
	$TMA \cdot PF_6$ (Saturated at 25 C)	1:1	$(PF_6)^-$	0.152 ±0.013
	$TMA \cdot PF_6$ (Saturated at 60 C)	4:1	$(PF_6)^-$	0.234 ±0.025
DMF #4-2	$TMA \cdot PF_6$ (0.101 M)		$(TMA)^+$	
	$TMA \cdot PF_6$ (Saturated at 25 C)	1:1	$(TMA)^+$	0.223 ±0.025
	$TMA \cdot PF_6$ (Saturated at 60 C)	5:1	$(TMA)^+$	0.337 ±0.034
PC #2-4	$TMA \cdot PF_6$ (0.0994 M)		$(TMA)^+$	
	$TMA \cdot PF_6$ (Saturated at 25 C)	1:1	$(TMA)^+$	0.142 ±0.015
	$TMA \cdot PF_6$ (Saturated at 60 C)	4:1	$(TMA)^+$	0.176 ±0.020

*Saturated solutions were diluted with pure solvent and the concentrations of such solutions were determined.

TABLE 8

(Concluded)

Solvent	Solute	Dilution*	Ion	Concentration of Saturated Solutions, molar
DMF #4-2	TMA·F (Saturated at 25 C)	1:1	$(TMA)^+$	0.028 ±0.005
PC #2-3	TMA·F (Saturated at 25 C)	1:1	$(TMA)^+$	0.065 ±0.010

*Saturated solutions were diluted with pure solvent and the concentrations of such solutions were determined.

TABLE 9

SUMMARY OF CONDUCTANCE MEASUREMENTS

Solutes	Concentration	Solvent	λ^*(25 C), $ohm^{-1} cm^{-1}$	λ (60 C), $ohm^{-1} cm^{-1}$
LiCl #2-1	Dilution series	PC #2-2		
LiCl #2-1	Saturated	PC #2-2		
$AlCl_3$ #3	Dilution series	PC #2-3		
$AlCl_3$ #3	Dilution series	PC #2-5		
LiCl #2-1 + $AlCl_3$ #3	Dilution series	PC #2-5		
LiCl and/Or $AlCl_3$	Dilution series	DMF		
LiCl #2-1	0.0528 M	DMF #3-2	1.97×10^{-3}	2.48×10^{-3}
LiCl #2-1 + $AlCl_3$ #3	0.0528 M 0.0528 M	DMF #3-2	3.79×10^{-3}	2.95×10^{-3}
LiCl #2-1 + $AlCl_3$ #3	1 M Saturated	DMF #3-2	7.77×10^{-3}	1.19×10^{-2}
$TMA\cdot PF_6$ #1	Dilution series	PC #2-5		
$TMA\cdot PF_6$ #1	Saturated	PC #2-5	2.71×10^{-3}	6.75×10^{-3}
$TMA\cdot PF_6$ #1	Dilution series	DMF #1-2		
$TMA\cdot PF_6$ #1	Saturated	DMF #1-2	1.11×10^{-2}	1.93×10^{-2}
$TMA\cdot PF_6$ #1	Dilution series	AN #1-2		
$TMA\cdot PF_6$ #1	Saturated	AN #1-2	1.14×10^{-2}	2.26×10^{-2}
$CuCl_2$ #2	Saturated	DMF #1-2	5.95×10^{-3}	9.06×10^{-3}

$^*\lambda$: Specific conductance

TABLE 10

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ) OF LiCl/PC AT 25 AND 60 C

Concentration (C), molar	$\sqrt{C}$, molar$^{1/2}$	λ (25 C), ohm^{-1} cm^{-1}	Λ^* (25 C), ohm^{-1} equ^{-1} cm^2	λ (60 C), ohm^{-1} cm^{-1}	Λ^* (60 C), ohm^{-1} equ^{-1} cm^2
0.0265	0.163	1.459×10^{-4}	5.49	2.719×10^{-4}	10.2
0.0053	0.0728	5.828×10^{-5}	10.9	1.079×10^{-4}	20.2
0.00265	0.0515	3.600×10^{-5}	13.5	6.717×10^{-5}	25.0
0.00106	0.0326	1.825×10^{-5}	16.9	3.439×10^{-5}	31.6
0.000530	0.0230	1.075×10^{-5}	19.7	2.011×10^{-5}	36.3
0.0001766	0.0133	4.261×10^{-6}	22.5	7.769×10^{-6}	39.0
0		2.858×10^{-7}		8.846×10^{-7}	
			Extrapolated: $\Lambda_o = 26.2$		Extrapolated: $\Lambda_o = 44.9$

*Λ: Equivalent conductance, corrected for conductance of pure solvent

TABLE 11

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ) OF $AlCl_3$/PC AT 25 AND 60 C

Concentration (C), molar	$\sqrt{C}$, $molar^{1/2}$	λ(25 C), $ohm^{-1} cm^{-1}$	Λ^* (25 C), $ohm^{-1} equ^{-1} cm^{2}$	λ (60 C), $ohm^{-1} cm^{-1}$	Λ^* (60 C), $ohm^{-1} equ^{-1} cm^{2}$
1.00	1.00	6.960×10^{-3}	6.96	1.309×10^{-2}	13.09
0.261	0.5108	3.227×10^{-3}	12.36	5.567×10^{-3}	21.33
0.0522	0.2285	8.218×10^{-4}	15.73	1.390×10^{-3}	26.62
0.01044	0.1022	1.716×10^{-4}	16.42	2.838×10^{-4}	27.16
0.00522	0.07220	7.618×10^{-5}	14.56	1.275×10^{-4}	24.37
0.00261	0.05108	3.110×10^{-5}	11.85	5.576×10^{-5}	21.24
0.00130	0.03605	1.416×10^{-5}	10.75	2.538×10^{-5}	19.28
0.000522	0.02285	5.421×10^{-6}	10.04	9.636×10^{-6}	17.85
0.000124	0.0114	1.351×10^{-6}	9.46	2.291×10^{-6}	15.91
0		1.784×10^{-7}		3.185×10^{-7}	

$^*\Lambda$: Equivalent conductance, corrected for conductance of pure solvent

TABLE 12

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ) OF $AlCl_3$/PC AT 25 AND 60 C

Concentration (C), molar	$\sqrt{C}$, $molar^{1/2}$	λ (25 C), $ohm^{-1}cm^{-1}$	Λ* (25 C), $ohm^{-1}equ^{-1}cm^{2}$	λ (60 C), $ohm^{-1}cm^{-1}$	Λ* (60 C), $ohm^{-1}equ^{-1}cm^{2}$
0.0913	0.302	1.356×10^{-3}	14.8	2.300×10^{-3}	25.2
0.0183	0.135	2.883×10^{-4}	15.7	4.767×10^{-4}	26.0
0.00913	0.0955	1.307×10^{-4}	14.2	2.111×10^{-4}	23.0
0.00456	0.0676	4.992×10^{-5}	10.8	8.853×10^{-5}	19.1
0.00228	0.0478	2.077×10^{-5}	8.83	3.853×10^{-5}	16.4
0.00114	0.0338	1.161×10^{-5}	9.62	2.074×10^{-5}	17.2
0.000570	0.0239	6.807×10^{-6}	10.8	1.340×10^{-5}	21.5
0.000285	0.0169	6.682×10^{-6}	21.2	1.330×10^{-5}	42.66
0.000143	0.0119	5.576×10^{-6}	34.6	1.104×10^{-5}	69.23
0		6.289×10^{-7}		1.136×10^{-6}	

Λ*Equivalent conductance, corrected for conductance of solvent

TABLE 13

SPECIFIC CONDUCTANCE (κ) AND EQUIVALENT CONDUCTANCE (Λ) OF LiCl+$AlCl_3$/PC AT 25 AND 60 C

Concentration (c), molar*	$\sqrt{c}$, $\text{molar}^{1/2}$	λ (25 C), $\text{ohm}^{-1}\text{cm}^{-1}$	Λ^{**} (25 C), $\text{ohm}^{-1}\text{equ}^{-1}\text{cm}^{2}$	λ(60 C), $\text{ohm}^{-1}\text{cm}^{-1}$	Λ^{**} (60 C), $\text{ohm}^{-1}\text{equ}^{-1}\text{cm}^{2}$
1.00*	1.00	6.578×10^{-3}	6.58	1.292×10^{-2}	12.9
0.200	0.447	3.003×10^{-3}	15.0	5.188×10^{-3}	25.9
0.0400	0.200	7.515×10^{-4}	18.8	1.270×10^{-3}	31.8
0.00400	0.0632	8.595×10^{-5}	21.5	1.449×10^{-4}	36.2
0.00200	0.0447	4.480×10^{-5}	22.3	7.521×10^{-5}	37.4
0.00100	0.0316	2.354×10^{-5}	23.4	3.929×10^{-5}	39.0
0.000500	0.0224	1.232×10^{-5}	24.3	2.068×10^{-5}	40.8
0.000250	0.0158	6.814×10^{-6}	26.6	1.158×10^{-5}	44.9
0.000125	0.0112	3.788×10^{-6}	28.9	6.509×10^{-6}	49.7
0		1.752×10^{-7}		2.988×10^{-7}	
			trapolated: $\Lambda_o = 34.5$		Extrapolated: $\Lambda_o = 61.7$

*The initial solution was 1 molar with respect to $AlCl_3$ and saturated with respect to LiCl (0.78 molar LiCl) The other solutions were obtained by diluting this solution. The concentrations listed refer to the $AlCl_3$ concentration.

**Λ: Equivalent conductance, corrected for conductance of pure solvent

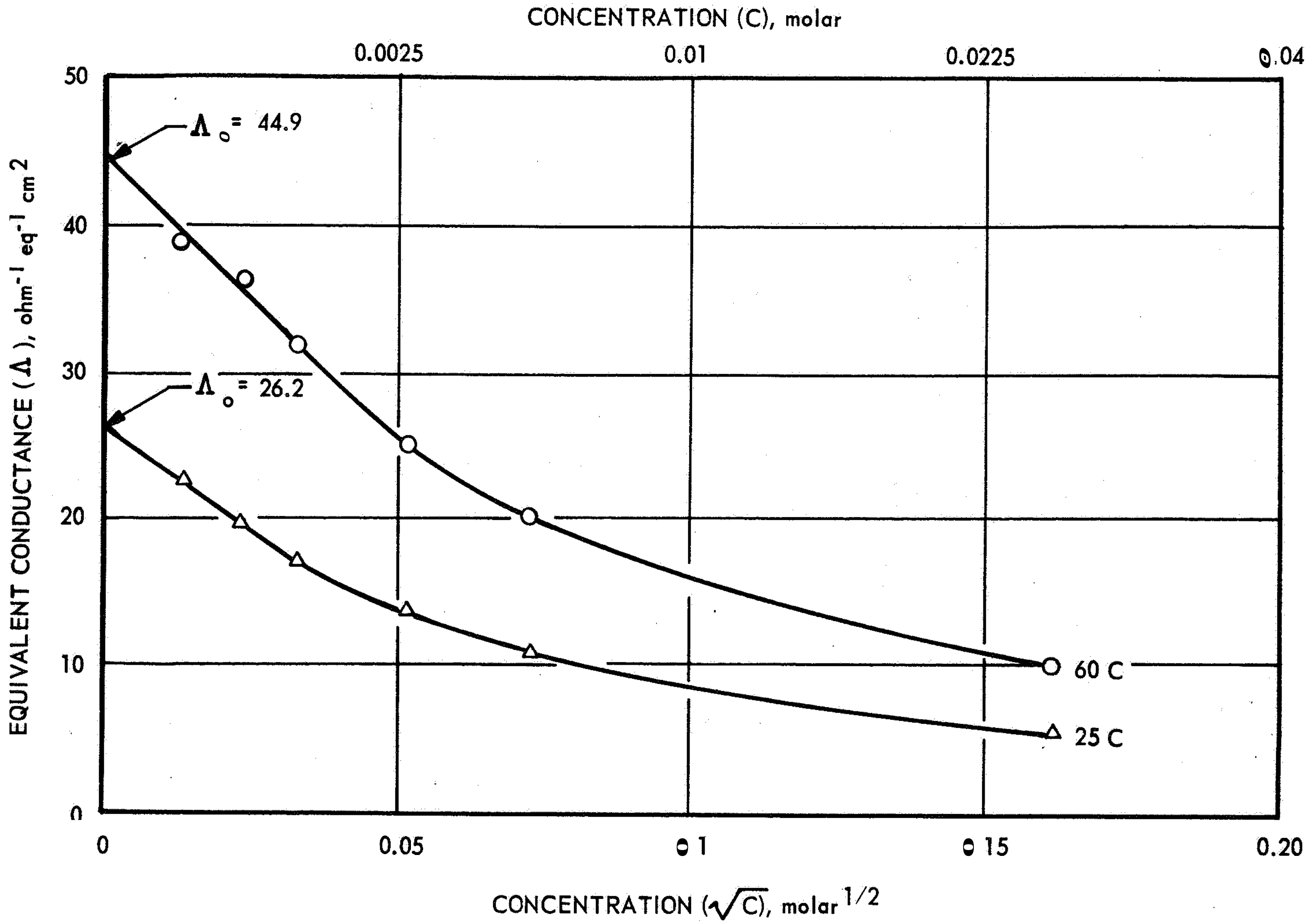

Figure 10. Equivalent Conductance of LiCl in Propylene Carbonate at 25 and 60 C

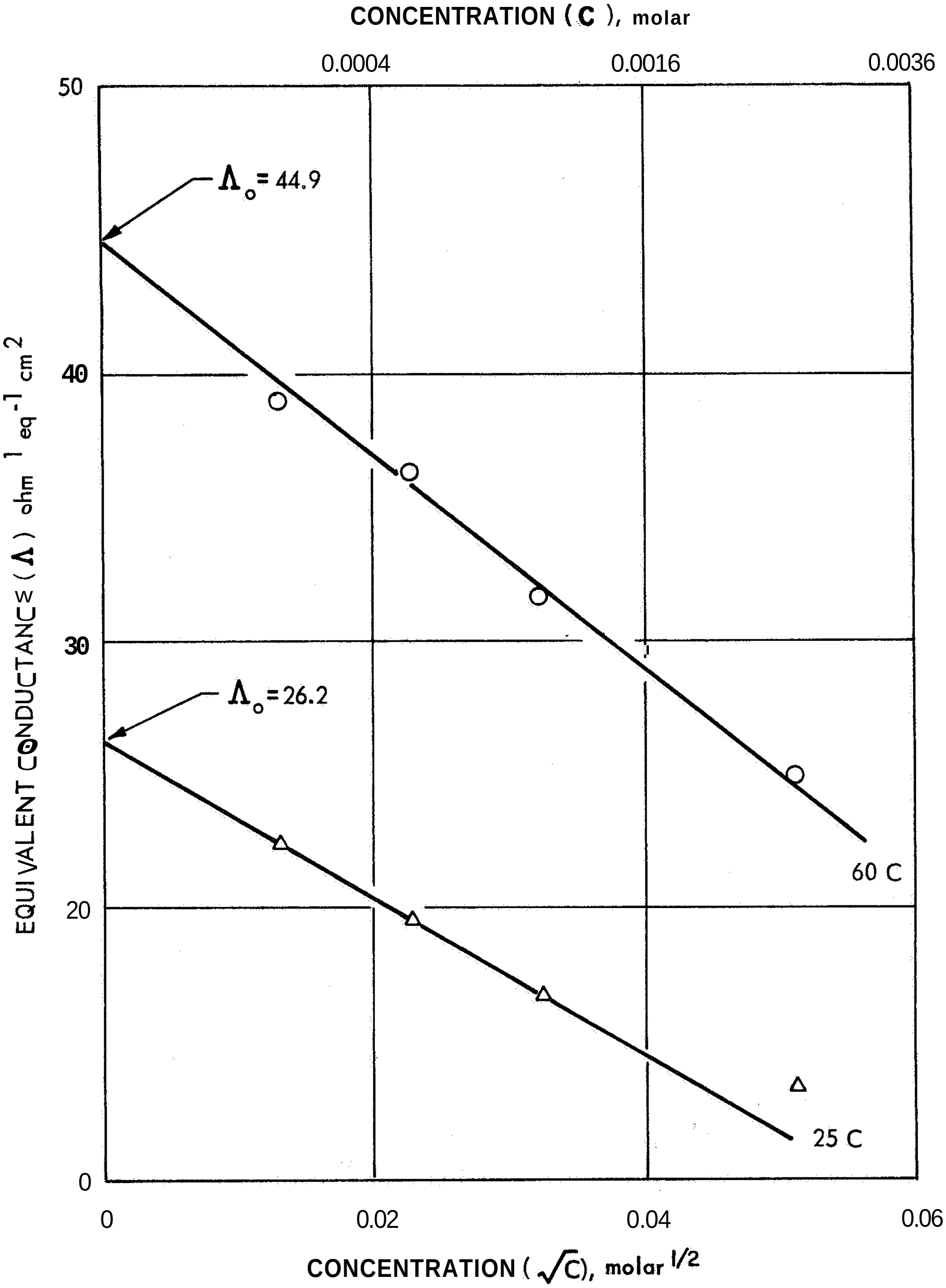

Figure 11. Equivalent Conductance of LiCl in Propylene Carbonate at 25 and 60 C

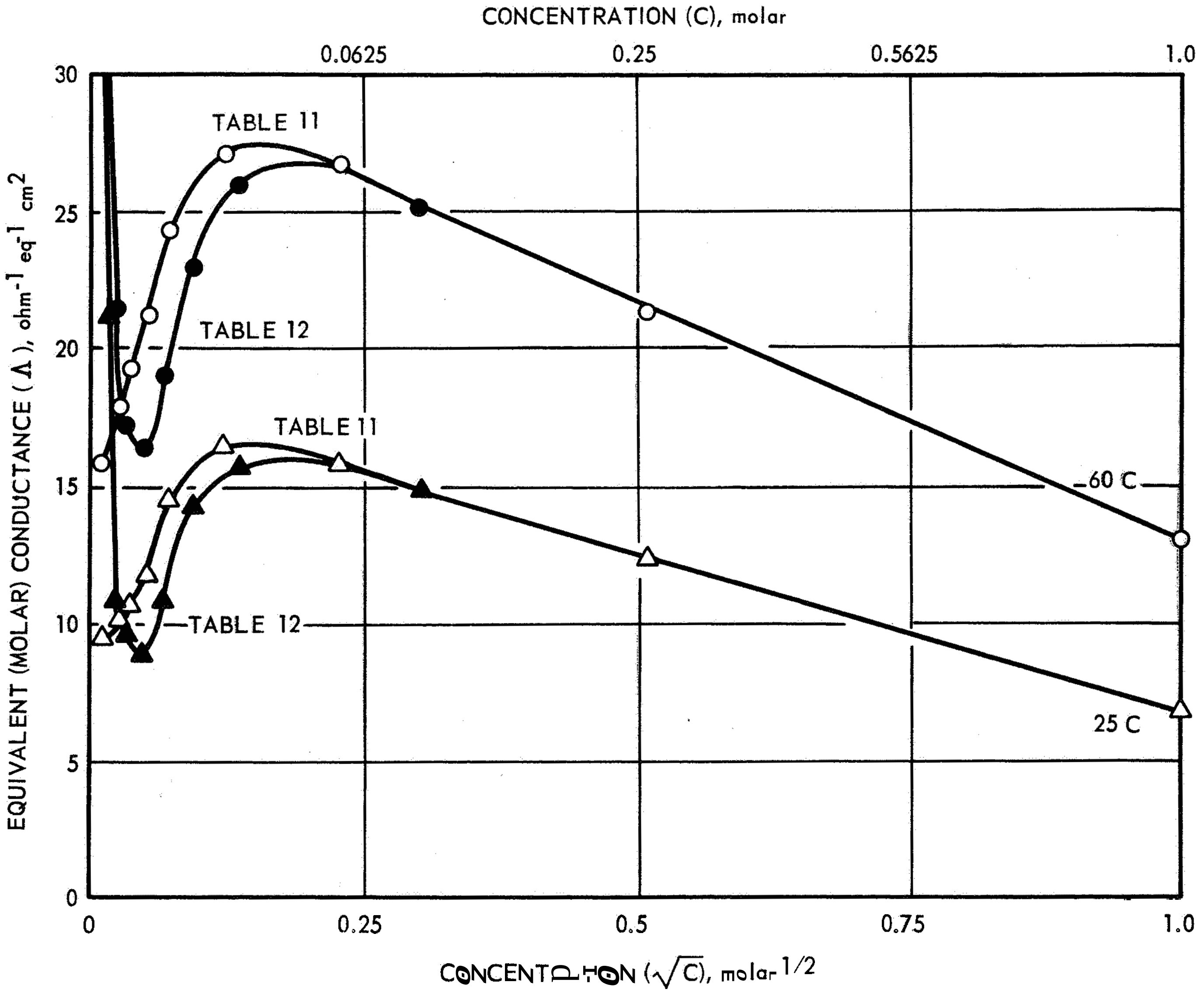

Figure 12 . Equivalent (Molar) Conductance of $AlCl_3$ in Propylene Carbonate at 25 and 60 C

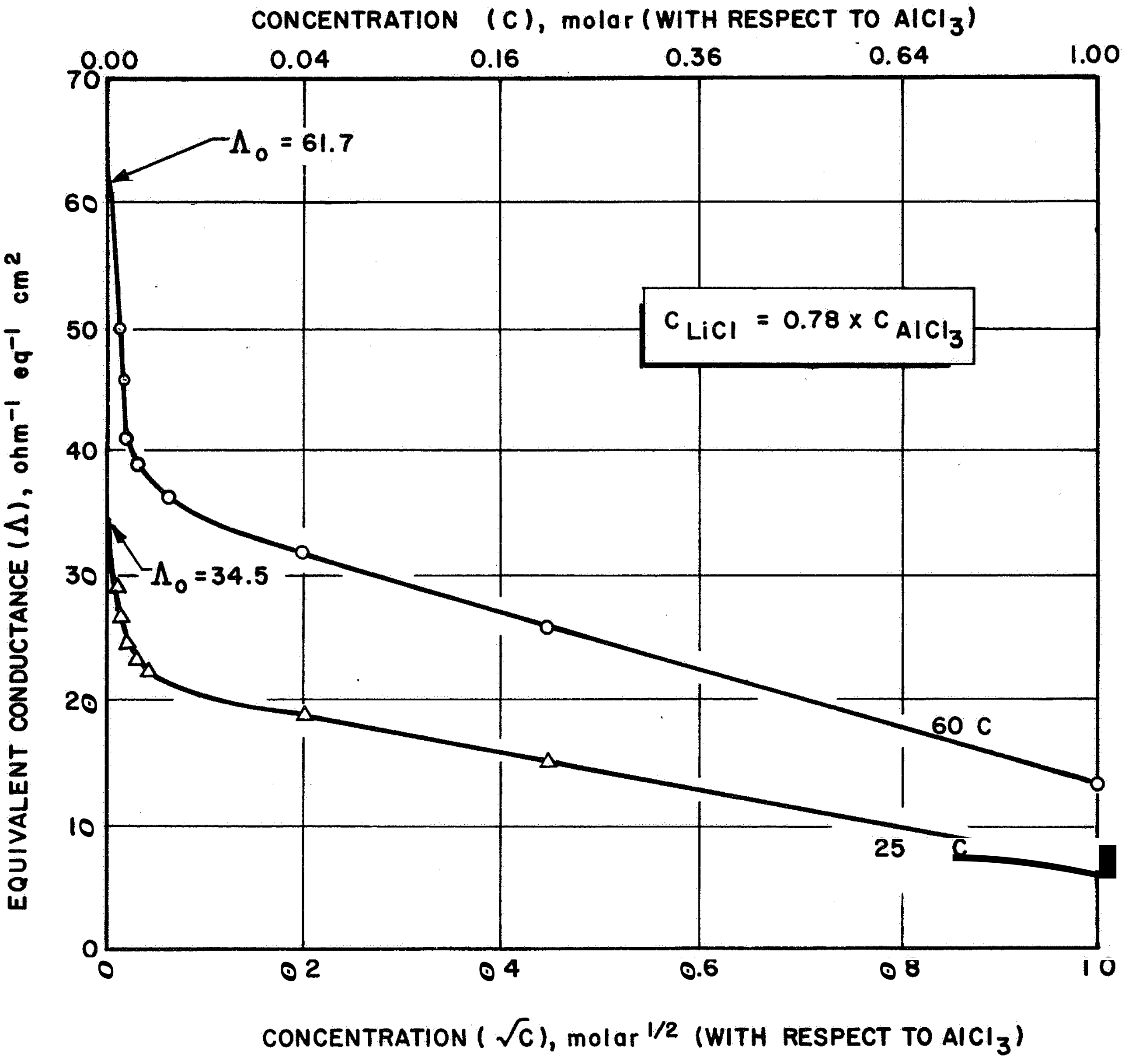

Figure 13. Equivalent (Molar) Conductance of LiCl + AlCl$_3$ in Propylene Carbonate at 25 and 60 C

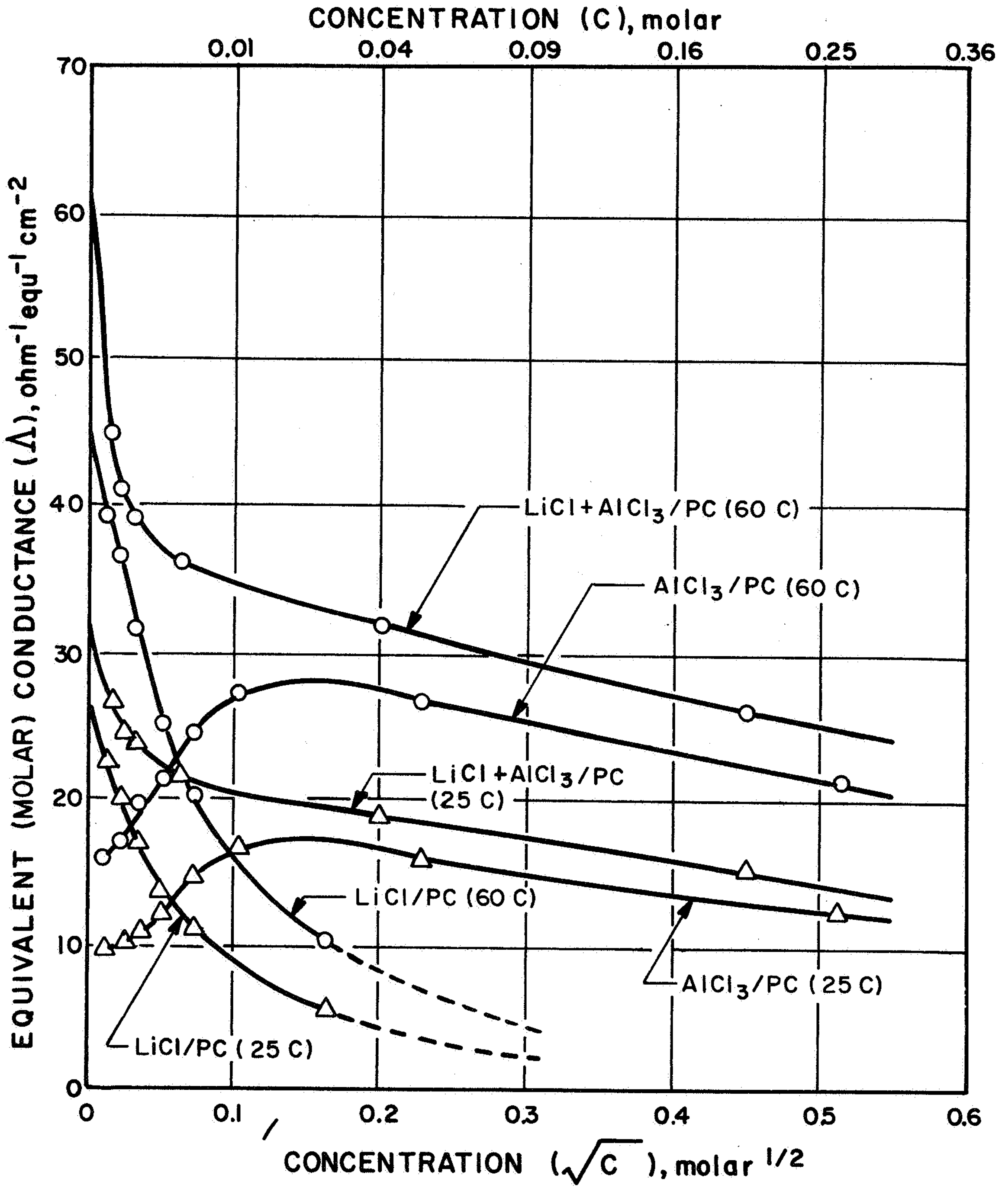

Figure 14. Comparison of Equivalent (Molar) Conductances of Electrolytes Containing LiCl and/or $AlCl_3$ in PC at 25 and 60 C

case of LiCl/PC (Fig. 10 and 11) Λ plotted vs $\sqrt{C}$ results in a straight line at low concentrations, and an extrapolation to zero concentration is readily obtained: Λ_0 = 26.2 $ohm^{-1}equ^{-1}cm^2$ at 25 C and Λ_0 = 44.9 $ohm^{-1}equ^{-1}cm^2$ at 60 C. For the $AlCl_3$/PC system, two sets of measurements were taken in addition to the results reported earlier (Ref. 1). In the first set (Table 11), the dilution series was initiated with a 1 molar solution which was brownishly discolored; in the second set (Table 12), the dilution was initiated with a 0.1 molar solution which was only slightly tinted. A maximum and a minimum of the equivalent conductance was observed for $AlCl_3$/PC with decreasing solute concentration (Fig. 12), although in one case the minimum was only fragmentally indicated as a slight change in the slope of the curve. An unambiguous interpretation of this behavior has not been Pound; the occurrence of the minimum at low concentrations may be connected with the water content of the solvent, which was approximately 20 ppm (corresponding to a concentration of 0.001 molar). A study of this effect as a function of the water content could result in clarification, but such a study is beyond the scope of the present work. A reasonable extrapolation of the equivalent conductance to zero concentration cannot be made from the present measurements. For the electrolyte containing LiCl and $AlCl_3$, Λ_0 of 34.5 and 61.7 $ohm^{-1}equ^{-1}cm^2$ have been obtained at 25 and 60 C, respectively, but the extrapolation is somewhat uncertain.

A comparison of the three electrolytes is illustrated in Fig. 14. There appears to be an additive effect in the molar conductances when LiCl is added to $AlCl_3$/PC. At low concentrations, the high conductance of LiCl/PC predominates; at higher concentrations, the conductance curves of $AlCl_3$/PC and of the mixed electrolyte approach each other because the contribution of LiCl becomes small, A chemical interaction resulting in the formation of new species as indicated by the increase of the solubility of LiCl is not readily evidenced by the present conductance results. Nuclear magnetic resonance studies may produce more direct, less ambiguous information in this respect.

It has been found previously (Ref. 2) that the sum of the Λ_o values for LiCl/DMF and $AlCl_3$/DMF electrolytes did not equal the Λ_o value obtained for the LiCl + $AlCl_3$/DMF mixed electrolyte. Some of the extrapolations to zero concentration were questionable because linearity at low concentrations was not obtained. Therefore, equivalent conductance values are compared at a specific concentration, 0.05 molar. The values on which the comparison is based are presented in Table 14. The majority of the values were obtained by interpolation of conductance curves. In the case of LiCl/PC, an extrapolation was made, because 0.05 molar designates a concentration beyond the solubility of LiCl in PC. The sum of the Λ values for a 0.0528 M LiCl/DMF and a 0.0528 M $AlCl_3$/DMF solution is $37.4 + 53 = 90.4\ ohm^{-1}equ^{-1}cm^2$, but the Λ value for a 0.0528 M LiCl + 0.0528 M $AlCl_3$/DMF solution is 71.8 $ohm^{-1}equ^{-1}cm^2$. However, this latter solution is expected to have a higher ionic strength, and one should rather take into account the Λ values at 0.106 molar; if it is assumed that the two solutes do not chemically interact, some interaction will be reflected nevertheless in a change of the activity of the individual components. If as a first approximation, the Λ values of 0.106M LiCl/DMF and of 0.106 M $AlCl_3$/DMF are added, 74 $ohm^{-1}equ^{-1}cm^2$ results, which closely approximates the value for the 0.0528 M LiCl + 0.0528 M $AlCl_3$/DMF mixture (Λ = 71.8 $ohm^{-1}equ^{-1}cm^2$ at 25 C), and a similar agreement is found also for values obtained at 60 C. This result gives no indication of any interaction between the two solutes, LiCl and $AlCl_3$, such as:

$$Li^+ + Cl^- + 1/2\ AlCl_2^+ + 1/2\ AlCl_4^- \longrightarrow Li^+ + AlCl_4^-$$

This appears to apply also for values obtained in PC, although the extrapolation of Λ values for LiCl/PC is uncertain. Because a 1 M $AlCl_3$/PC solution saturated with LiCl (~ 0.8 molar) was used for the dilution series, the Λ values of 0.09 molar LiCl/PC and $AlCl_3$/PC solutions

TABLE 14

COMPARISON OF CONDUCTANCE VALUES OF 0.05 M LiCl, 0.05 M $AlCl_3$, AND 0.05 M LiCl + 0.05 M $AlCl_3$ SOLUTIONS IN PC AND DMF

Solution	Concentration, molar	Λ* (25 C), $ohm^{-1} equ^{-1} cm^2$	Λ* (60 C), $ohm^{-1} equ^{-1} cm^2$	Determination
LiCl/PC	0.04	4.2	8.0	(e)
	0.09	2.2	4.2	(e)
	0	26.2	44.9	(e)
$AlCl_3$/PC	0.05	15.8	26.8	(i)
	0.09	14.8	25.3	(i)
LiCl + $AlCl_3$/PC	0.04 + 0.05	18.4	31.0	(i)
	0	34.5	61.7	(e)
LiCl/DMF	0.0528	37.4	46.9	(m)
	0.106	31	38	(i)
	0	84.0	118	(e)
$AlCl_3$/DMF	0.0528	53	71	(i)
	0.106	43	58	(e)
	0	220	280	(e)
LiCl + $AlCl_3$/DMF	0.0528 + 0.0528	71.8	93.7	(m)
	0	260	325	(e)

*Λ: Equivalent (molar) conductance, corrected for conductance of pure solvent

NOTE: m = measured value i = interpolated value e = extrapolated value at 25 C (2.2 and 14.8 $ohm^{-1}equ^{-1}cm^{2}$, respectively) were compared with the respective values for 0.04 M LiCl + 0.05 M $AlCl_3$/PC (Λ = 18.4 $ohm^{-1}equ^{-1}cm^{2}$ at 25 C). No indication of a direct interaction between LiCl and $AlCl_3$ in PC can be obtained from the preceding comparison, although absence of such an interaction is certainly not proven,

Specific conductance and equivalent conductance values for electrolytes containing $TMA \cdot PF_6$ are presented in Tables 15 through 17 and Fig. 15 through 20. Extrapolation of the equivalent conductance to infinite dilution yielded values which suggest the presence of $TMA \cdot PF_6$ as a 1-1 electrolyte forming $(CH_3)_4N^+$ and PF_6^- ions in all three solvents. This is in agreement with the results of the nuclear magnetic resonance structural studies.

Measurement of Transference Numbers by the Hittorf Method

The cell designed for these experiments is shown in Fig. 21 and 22. The cell design of Wall, Stent, and Ondrejcin (Ref. 6) was modified to allow easy immersion of the lower parts of the cell into an oil bath. The experiments are performed at room temperature; however, the electrolyte warms under current flow because of the relatively low specific conductance of the electrolytes under study, and performing the experiment at a completely controlled, constant temperature is not possible. It appears that cooling of the lower cell parts in a bath is efficient enough to prevent excessive temperature increases in the cell. Cooling of only the lower parts of the cell also furnishes a stable density distribution in the electrolyte. The three compartments are filled with a total of approximately 90 milliliters of electrolyte and the electrolyte is raised to the desired level by applying suction to the center arm. At the conclusion of the experiment, the stopcock is reopened to separate catholyte and anolyte from the electrolyte contained in the middle compartment. The total loss of solute in the cathode and anode compartment is determined by analysis, and the transference numbers are calculated.

TABLE 15

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ) OF $TMA \cdot PF_6/PC$ AT 25 AND 60 C

Concentration (C), molar	$\sqrt{C}$, molar$^{1/2}$	λ (25 C), $ohm^{-1} cm^{-1}$	Λ* (25 C), $ohm^{-1} equ^{-1} cm^2$	λ (60 C), $ohm^{-1} cm^{-1}$	Λ* (60 C), $ohm^{-1} equ^{-1} cm^2$
0.09942	0.315	2.236×10^{-3}	22.48	3.776×10^{-3}	37.97
0.0165	0.129	4.503×10^{-4}	27.09	7.637×10^{-4}	46.03
0.00828	0.0910	2.333×10^{-4}	28.10	3.965×10^{-4}	47.75
0.00414	0.0644	1.212×10^{-4}	29.14	2.056×10^{-4}	49.41
0.00207	0.0439	6.218×10^{-5}	29.77	1.059×10^{-4}	50.69
0.00104	0.0322	3.203×10^{-5}	30.44	5.462×10^{-5}	51.87
0.000518	0.0228	1.690×10^{-5}	31.65	2.876×10^{-5}	53.80
0.000259	0.0161	8.830×10^{-6}	32.14	1.506×10^{-5}	54.69
0.000129	0.0114	4.601×10^{-6}	31.63	7.841×10^{-6}	53.62
0		5.083×10^{-7}		9.030×10^{-7}	
			Extrapolated: $\Lambda_o = 33.9$		Extrapolated: $\Lambda_o = 57.6$

*Λ: Equivalent conductance, corrected for conductance of pure solvent

TABLE 16

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ) OF TMA·PF_6/DMF AT 25 AND 60 C

Concentration (C), molar	$\sqrt{C}$, $molar^{1/2}$	λ (25 C), $ohm^{-1} cm^{-1}$	Λ* (25 C), $ohm^{-1} equ^{-1} cm^2$	λ (60 C), $ohm^{-1} cm^{-1}$	Λ* (60 C), $ohm^{-1} equ^{-1} cm^2$
0.1010	0.318	5.760×10^{-3}	57.04	7.806×10^{-3}	77.29
0.0101	0.100	7.466×10^{-4}	73.80	1.028×10^{-3}	101.6
0.00505	0.0710	3.905×10^{-4}	77.06	5.393×10^{-4}	106.4
0.00252	0.0502	2.040×10^{-4}	80.27	2.826×10^{-4}	111.2
0.00126	0.0355	1.059×10^{-4}	82.80	1.467×10^{-4}	114.7
0.000631	0.0251	5.479×10^{-5}	84.66	7.622×10^{-5}	117.8
0.000315	0.0177	2.866×10^{-5}	86.60	3.975×10^{-5}	120.3
0.000158	0.0126	1.505×10^{-5}	86.58	2.084×10^{-5}	120.1
0		1.375×10^{-6}		1.875×10^{-6}	
			Extrapolated: Λ_o = 90.5		Extrapolated: Λ_o = 126

*Λ : Equivalent conductance, corrected for conductance of pure solvent

TABLE 17

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ) OF TMA·PF_6/AN AT 25 AND 60 C

Concentration (C), molar	$\sqrt{C}$, $molar^{1/2}$	λ (25 C), $ohm^{-1} cm^{-1}$	Λ* (25 C), $ohm^{-1} equ^{-1} cm^2$	λ (60 C), $ohm^{-1} cm^{-1}$	Λ* (60 C), $ohm^{-1} equ^{-1} cm^2$
0.07097	0.266	8.648×10^{-3}	121.8	1.070×10^{-2}	152.0
0.007097	0.0842	2.180×10^{-3}	306.9	2.733×10^{-3}	385.5
0.003548	0.0596	1.161×10^{-3}	326.7	1.463×10^{-3}	411.8
0.001774	0.0421	6.121×10^{-4}	344.1	7.726×10^{-4}	434.5
0.000888	0.0296	3.181×10^{-4}	356.4	4.030×10^{-4}	451.8
0.000444	0.0219	1.652×10^{-4}	368.5	2.085×10^{-4}	465.5
0.000222	0.0149	8.571×10^{-5}	378.7	1.076×10^{-4}	476.6
0.000111	0.0105	4.396×10^{-5}	381.3	5.450×10^{-5}	474.4
0		1.645×10^{-6}		1.844×10^{-6}	
			Extrapolated: Λ_o = 402		Extrapolated: Λ_o = 503

*: Equivalent conductance, corrected for conductance of pure solvent

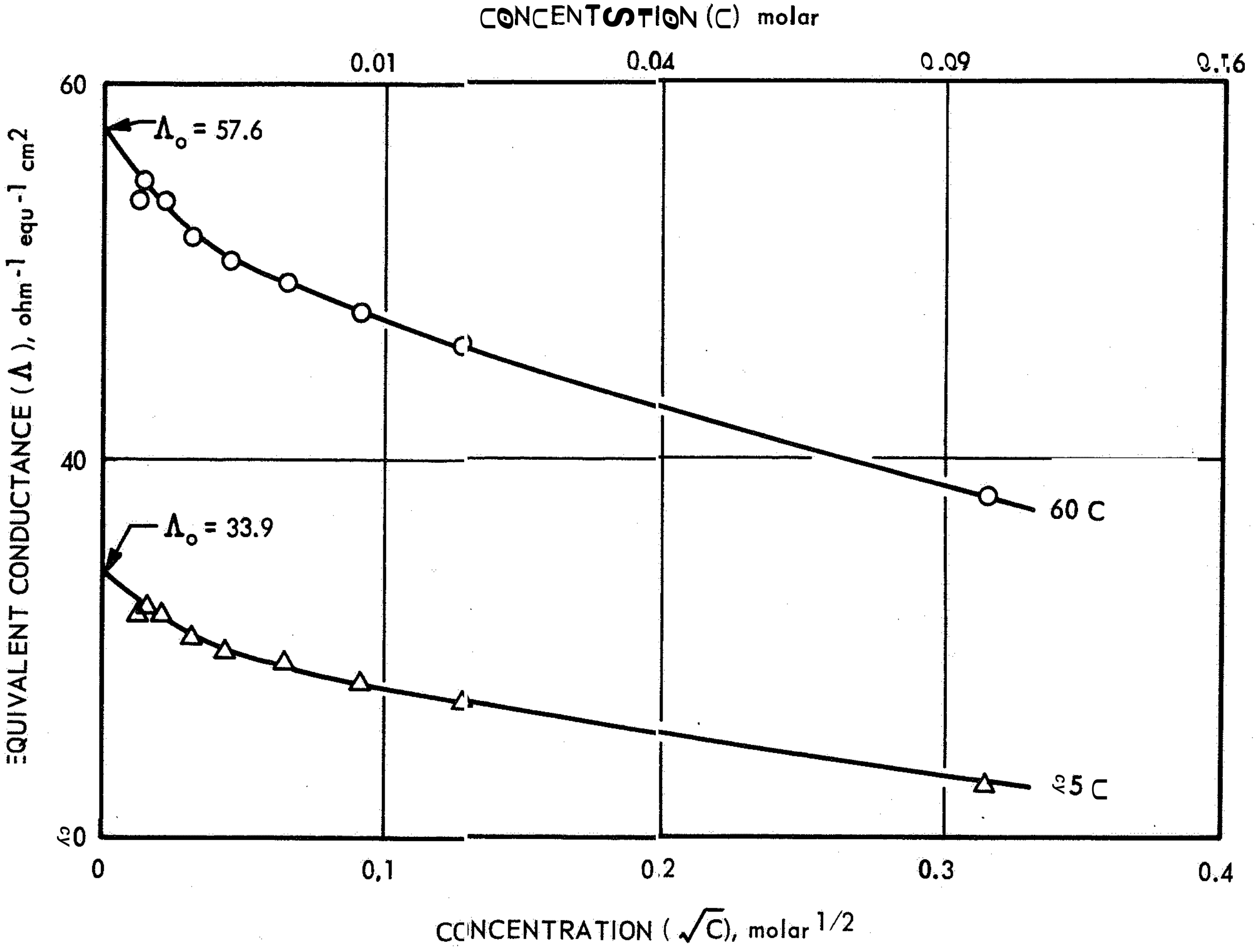

Figure 15. Equivalent Conductance of TMA·PF_6 in PC at 25 and 60 C

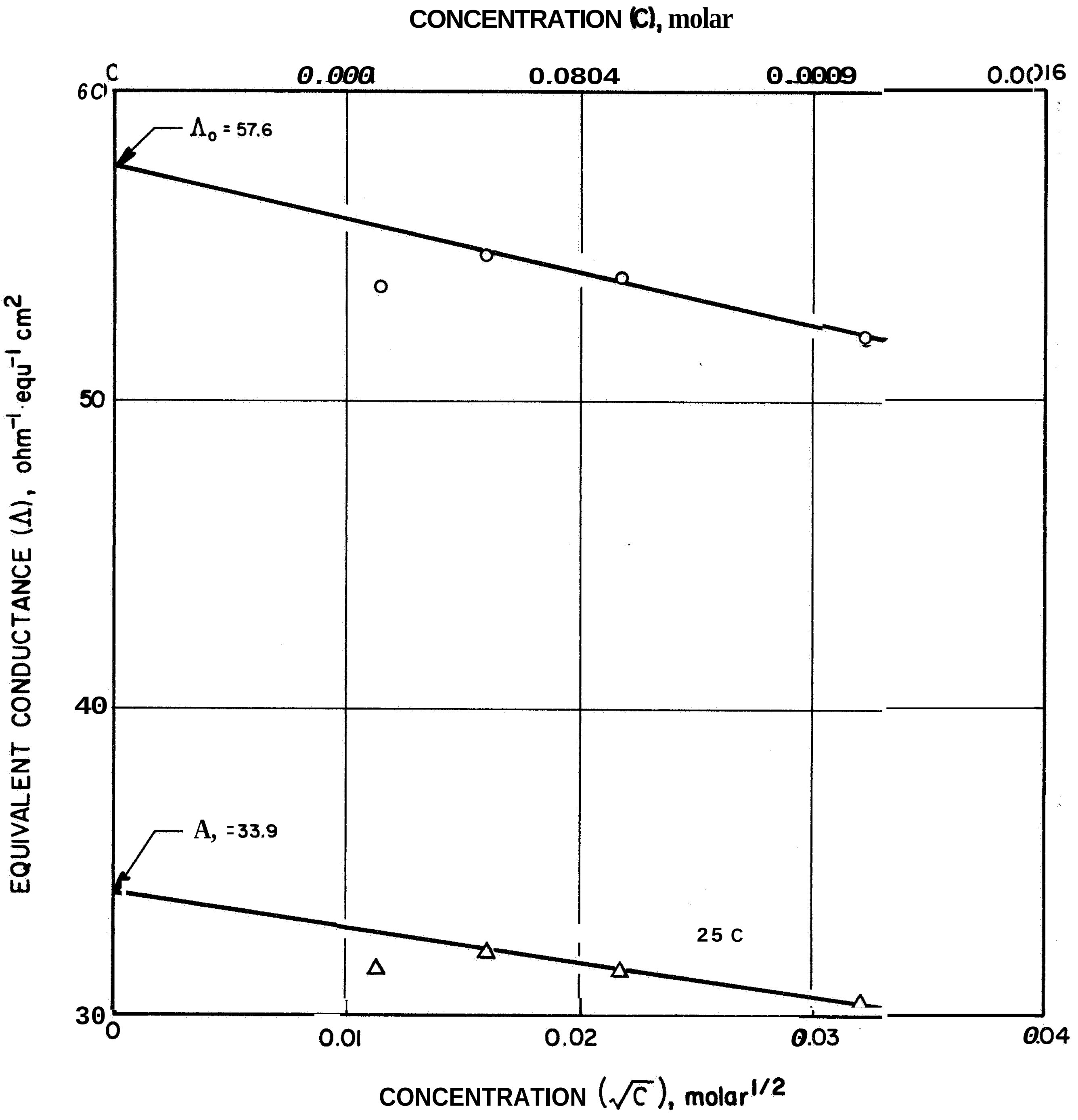

Figure 16. Equivalent Conductance of TMA·PF_6 in PC at 25 and 60 C

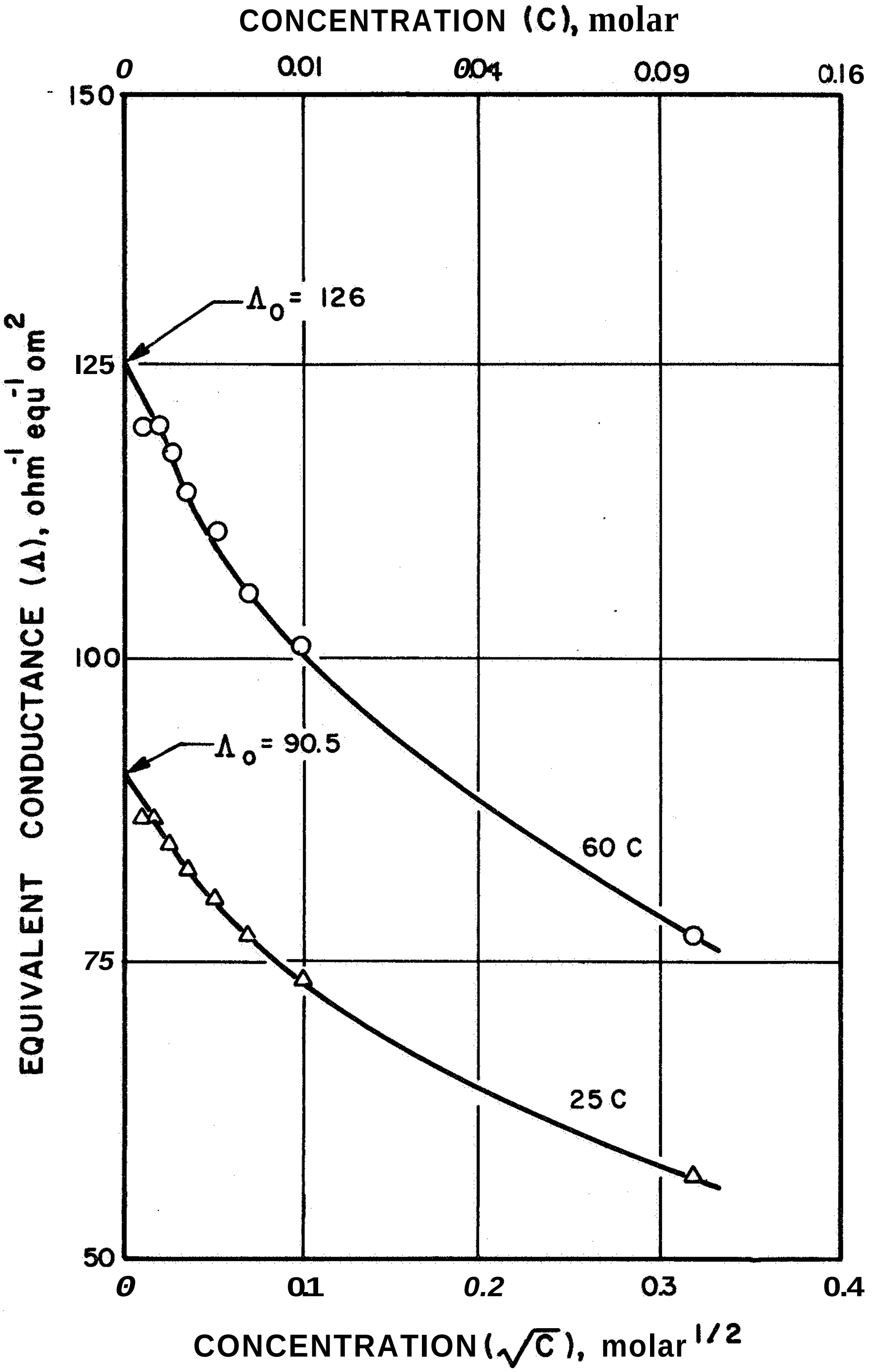

Figure 17. Equivalent Conductance of TMA·PF_6 in DMF at 25 and 60 C

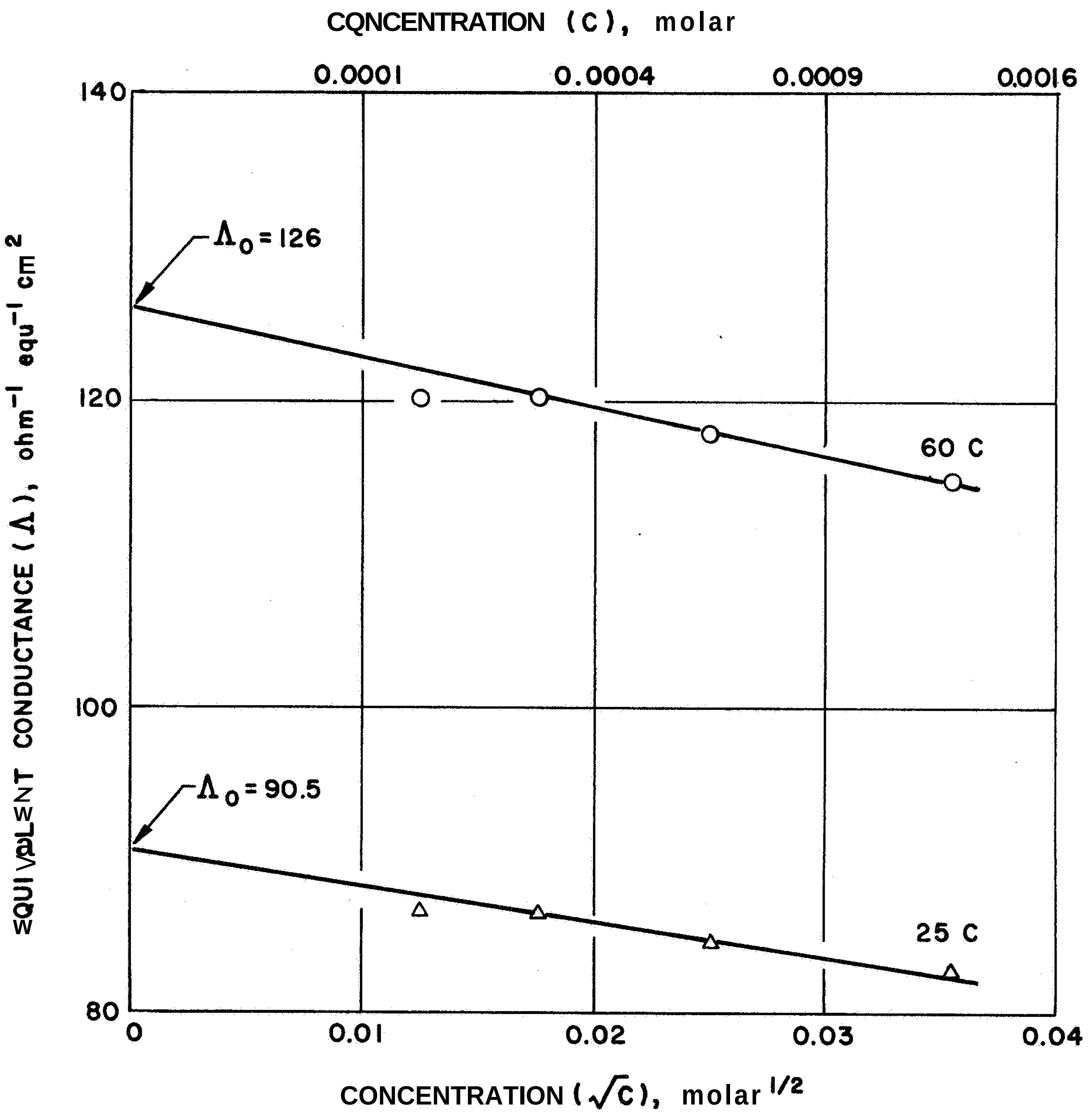

Figure 18. Equivalent Conductance of TMA·PF_6 in DMF at 25 and 60 C

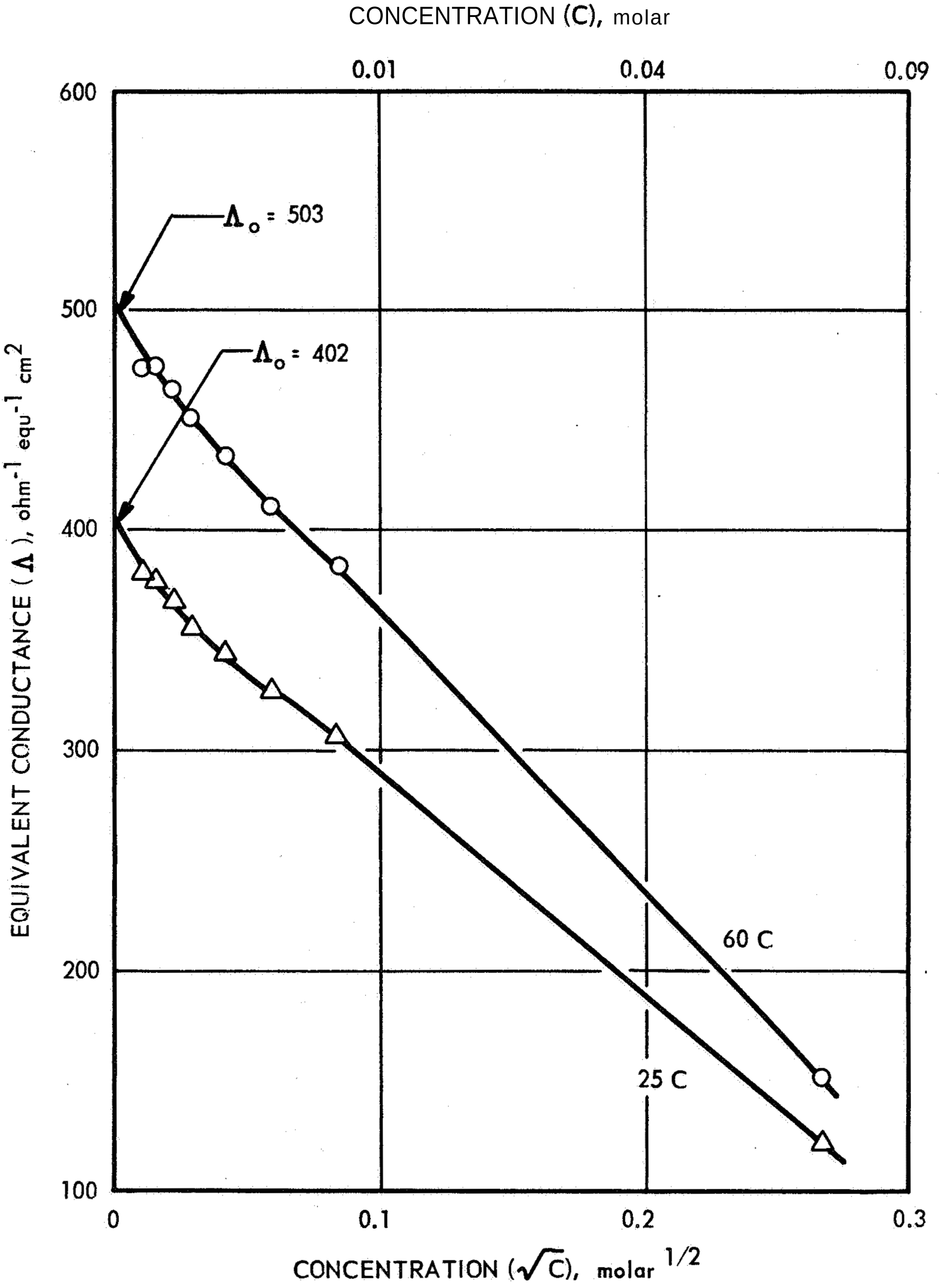

Figure 19. Equivalent Conductance of TMA·PF_6 in *AN* at 25 and *60* C

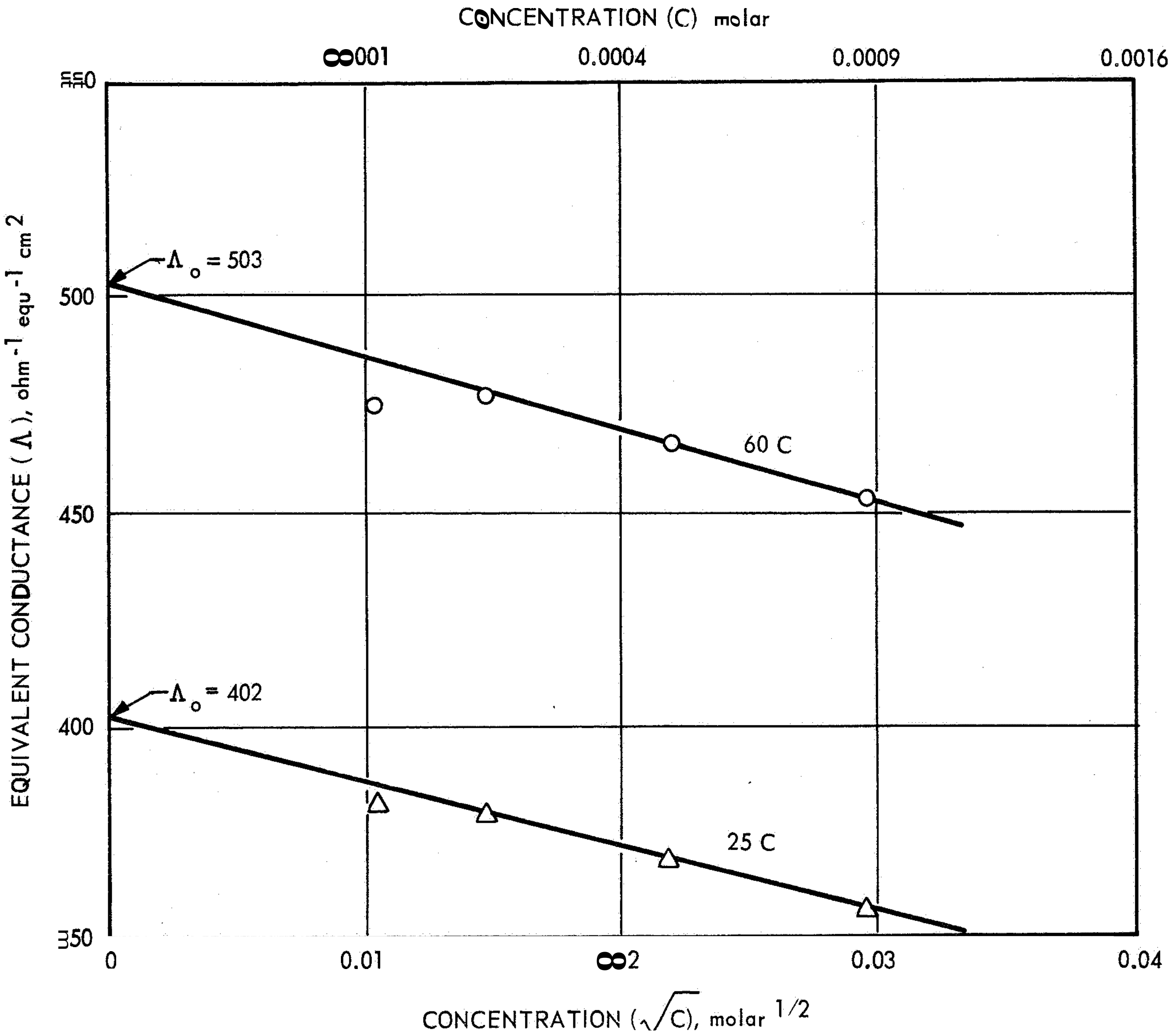

Figure 20. Equivalent Conductance of $TMA \cdot PF_6$ in AN at 25 and 60 C

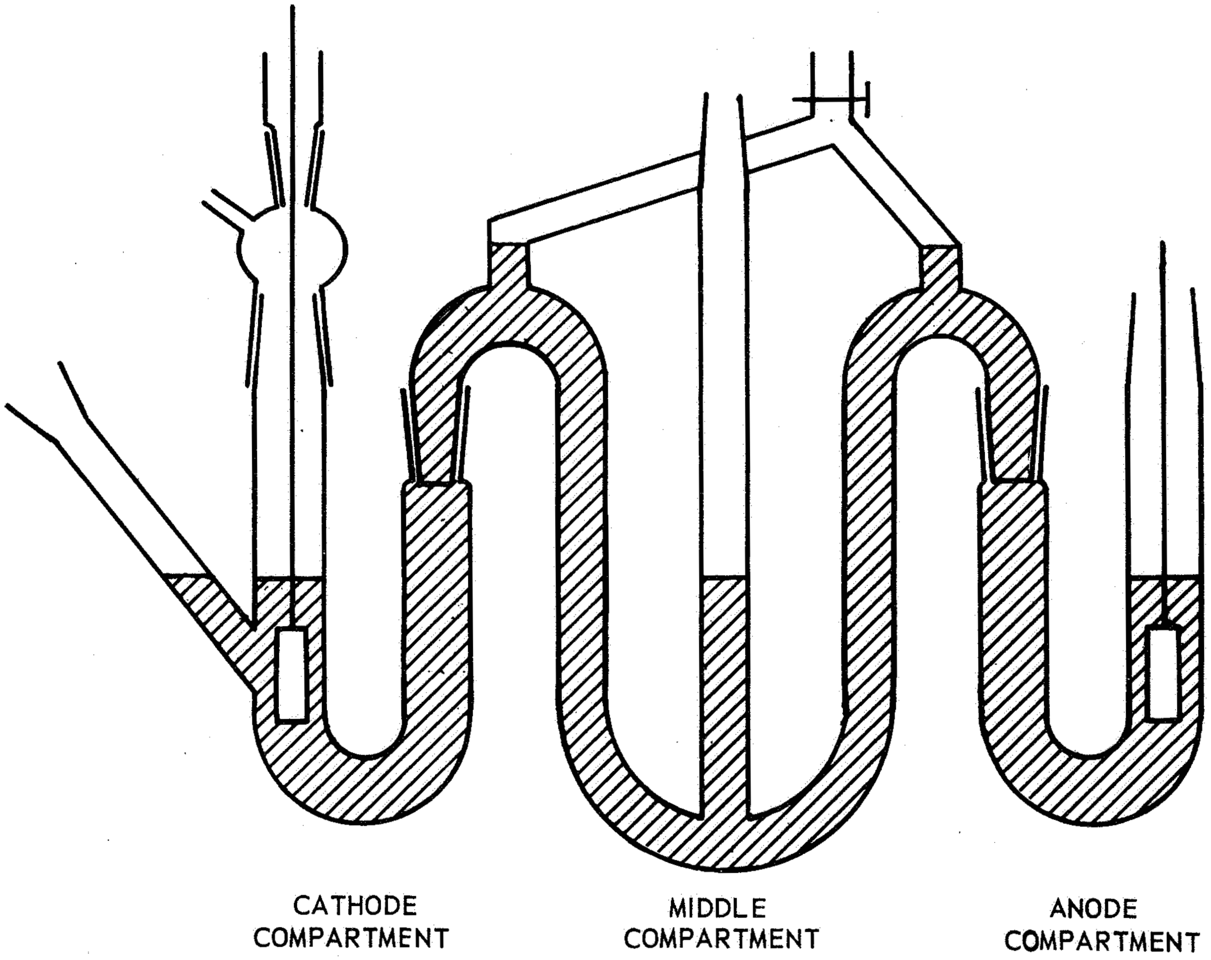

Figure 21. Hittorf Cell Schematic

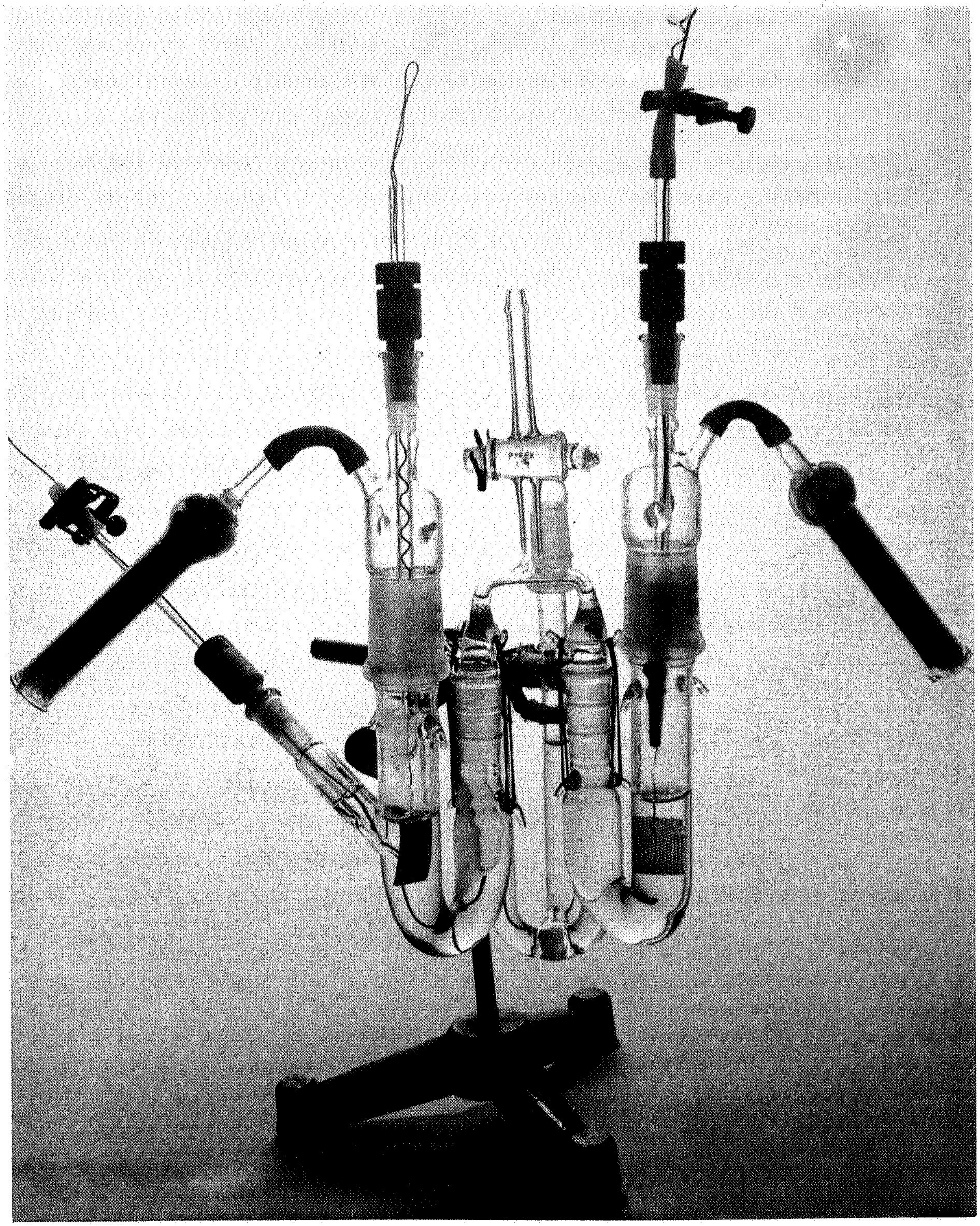

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Figure 22. Hittorf Cell

LiCl/DMF was the first system investigated. The experimental conditions and results are summarized in Table 18. A silver sheet anode was used. According to weight loss determinations, the anodic dissolution of the silver was quantitative, silver chloride being soluble in the electrolyte (it appears that this anodic reaction could be recommended for use in a coulometer). When the samples were diluted with water, AgCl precipitated quantitatively. Nickel screen cathodes were used, except in one case (run No. 4) where a copper sheet cathode was substituted. A lithium deposit was observed only in two cases (runs No. 1 and 5). In runs No. 2 through 4 a reaction of the solvent appeared to occur exclusively, and the resulting reducing compounds presented difficulties in the chloride analysis. Oxidation of the samples with H_2O_2 improved the reproducibility of the analysis results, but spurious results were still obtained (e.g., $t_- > 1$). The nature of the cathodic products and the conditions under which they form were not investigated (a pretreatment of the nickel substrate may be an important factor). If chlorine-containing organic species were formed, the decrease in chloride ion concentration would not result from ion transport alone. For these reasons, some of the transference numbers for the anion determined from the change in catholyte composition (runs No. 2 through 4) appear to be in error. There are two independent determinations of the transference numbers; (1) from changes of catholyte composition and (2) from change of anolyte composition. Unfortunately, the concentration changes of the anolyte (measured after precipitation of AgCl) are relatively small, because $t_- >> t_+$, and the limited accuracy of the analysis leads to a relatively high uncertainty in the transference numbers calculated from anolyte data.

Because of these difficulties and limitations, only the results of runs No. 1 and 5 are considered; t_+ and t_- are corrected to a sum of 1 (Table 18). An accuracy of ±0.05 is estimated for the transference

TABLE 18

DETERMINATIONS OF TRANSFERENCE NUMBERS (t) OF LiCl IN DMF BY THE HITTORF METHOD

Run No.	Solute	Concentration, molar	Solvent	Cell Current, milliamperes	Electrolysis Duration, hours	t_+ Calculated From Anolyte	t_- Calculated From Catholyte	t_+ Corrected	t_- Corrected
1	LiCl #2-1	1.25	DMF #3-1	10	6	0.27[a]	0.73[b]	0.2	0.73
2	LiCl #2-1	1	DMF #3-1	12	9-1/4	0.15	0.82		
3	LiCl #2-1	1	DMF #4-2	15	9	0.15	1.12		
4	LiCl #2-1	1	DMF #3-3	12	8	0.25	1.71		
5	LiCl #2-1	0.1	DMF #4-2	2.2	8	0.24[c]	0.70[d]	0.27	0.73

[a] Amount of chloride removed from anolyte (after precipitation of AgCl): 0.61 x 10^{-3} mole

[b] Amount of chloride removed from catholyte: 1.64 x 10^{-3} mole

[c] Amount of chloride removed from anolyte (after precipitation of AgCl): 1.6 x 10^{-4} mole

[d] Amount of chloride removed from catholyte: 4.6 x 10^{-4} mole

numbers for LiCl/DMF, $t_+ = 0.27$ and $t_- = 0.73$; these compare with $t_+ = 0.31$ and $t_- = 0.69$ as calculated from ion mobilities given by Prue and Sherrington (Ref. 7).

A mixed electrolyte containing 0.1 M $CuCl_2$ and 1 M LiCl in DME was also studied by the Hittorf method. It was found that the silver anode dissolved with coulombic efficiencies greater than 100 percent; an open circuit corrosion of the silver was verified. Because no copper deposit was observed, it may occur as follows:

$$Ag + Cu^{++} \rightarrow Ag^{+} + Cu^{+}$$

The anolyte was analyzed for the copper content and an accumulation of copper was found as shown in Table 19. The analysis result of run No. 1 is too low because some copper species seem to have coprecipitated with the silver chloride upon dilution of the samples with water (the silver chloride appeared bluish). For run No. 2, addition of ammonium hydroxide prevented this. Accumulation of copper in the anode compartment indicates the presence of negatively charged copper species in the electrolyte. Assuming a quantitative formation of $CuCl_4^{-2}$, the electrolyte would have a composition of 1.0 M Li^+ + 0.8 M Cl^- + 0.1 M $CuCl_4^{-2}$. If it is assumed, furthermore, that $CuCl_4^{-2}$ has twice the ion mobility of Cl^- because of the higher charge, and that the mobility of Li^+ is one-third of that of Cl^-, 27 percent of the current would be transported by the $CuCl_4^{-2}$. The experimental figure of n x 14.6 percent for the more reliable result of run No. 2 therefore indicates that n = 2 and copper chloride is present in the form of $CuCl_4^{-2}$ in LiCl/DMF. The preceding calculation is only a first approximation; the actual situation may be more complex because of ion pair formation, mixtures of various species, etc.

TABLE 19

HITTORF EXPERIMENT WITH AN ELECTROLYTE CONTAINING 1 M LiCl and 0.1 M $CuCl_2$ in DMF

Run No.	Accumulated Cu in Anolyte, moles	Corresponding Charge**, coulombs	Total Charge, coulombs	Percentage of Current Transported by Cu Species
1	2.16×10^{-4}*	n x 20.8	288	n x 7.2
2	3.92×10^{-4}	n x 37.8	258	n x 14.6

*Coprecipitation occured during analysis

**n = charge of copper complex

WORK PLANNED FOR NEXT QUARTER

PREPARATION OF ELECTROLYTES

The distillation of solvents will continue. The purity of the distilled products will be checked on a routine basis by chromatography on a Porapak Q column.

The analysis of all major solutes to be used on the program will be completed.

NUCLEAR MAGNETIC RESONANCE STRUCTURAL STUDIES

High resolution nuclear magnetic resonance studies of solvents containing solutes will continue in both LiCl + $AlCl_3$/PC and LiCl + $AlCl_3$/AN and will Be initiated for solutions of copper halides. The studying of solutes by wide line techniques will be initiated, with $AlCl_3$ and LiCl in PC and DMF being enphasized. Structural information obtained during these studies will be correlated with the physical and electrochemical properties of the electrolytes.

PHYSICAL PROPERTY DETERMINATIONS

Determinations of physical properties will continue. Solubility studies of lithium fluoride and lithium chloride, as well as of the copper halides in electrolyte solutions will be emphasized. In some cases, the influence of water on these solubilities will be investigated. Other measurements, such as the measurement of sonic velocities and of diffusion coefficients will be initiated.

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PROPERTIES OF NONAQUEOUS ELECTROLYTES

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content of each solvent is determined quantitatively to ±5 to 10 ppm. Structural studies of electrolytes containing tetramethylammonium hexafluorophosphate ($TMA \cdot PF_6$), $TMA \cdot F$ and $AlCl_3$ were performed by nuclear magnetic resonance. The physical property studies included measurements of viscosities, conductances, transference numbers, and solubilities of a number of electrolytes.

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UNCLASSIFIED

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Nonaqueous Electrolytes Acetonitrile Propylene Carbonate Dimethyl Formamide Tetramethylammonium Hexafluorophosphate						

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