PROPERTIES OF NONAQUEOUS ELECTROLYTES

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FOREWORD

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ABSTRACT

Studies of aprotic electrolytes based on three
solvents: propylene carbonate, dimethyl form-
amide, and acetonitrile, were continued. Character-
ized components were used to prepare the electro-
lyte solutions.

Structural studies of electrolytes containing
lithium perchlorate, lithium chloride, aluminum
chloride, and cupric chloride were performed by
nuclear magnetic resonance.

The physical properties measured included
solvent viscosities, electrolyte conductances to
determine individual ion mobilities, sonic velo-
cities, and transference numbers.

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SUMMARY

Physical properties and structural studies were conducted in the solvents propylene carbonate (PC), dimethyl formamide (DMF) and acetonitrile (AN); the water content of the solvents was in the range of 40 ±20 ppm. The distilled solvent batches were analyzed by vapor-phase chromatography on a routine basis.

The Cl^{35} nuclear magnetic resonance (NMR) was investigated, and approximate chemical shifts in lithium perchlorate solutions were determined. It was confirmed that ClO_4^- ions exist in these solutions. Nuclear magnetic resonance results also revealed the existence of two aluminum species in $AlCl_3$/AN solutions: $Al^{+++}[AN]_n$ and $AlCl_4^-$. The concentration of the former species decreased upon addition of LiCl. A change with time of the H^1 NMR spectrum of dimethyl formamide solutions containing $CuCl_2$ was observed, and this phenomenon is being investigated further.

Viscosity data for the solvents are presented for 25 and 60 C. Conductances were measured of electrolytes containing tetrabutylammonium tetraphenylboride, and tetrabutylammonium bromide to determine individual ion mobilities in lithium bromide and lithium perchlorate solutions. A low mobility of the lithium ion was found in all cases, thus confirming transference experiments. Results of Hittorf experiments with electrolytes containing 0.7 M LiCl + 1 M $AlCl_3$ in propylene carbonate and acetonitrile are consistent with the NMR results, i.e., with the presence of Li^+, Al^{+++}, and $AlCl_4^-$ ions.

Sonic velocities of some electrolytes were determined, and measurements of diffusion coefficients and dielectric constants were initiated.

DESCRIPTION OF PROGRESS

PREPARATION OF ELECTROLYTES

Purification of Solvents

Solvents were purified by distillation and analyzed as described previously (Ref. 1 through 4). Summaries of both the distillation conditions and the analytical procedure normally applied were presented in Ref. 3. The solvent batches used during the report period are listed in Table 1. With one exception, the water content of all solvent batches was 40 ±20 ppm, and no organic impurity above this level was detected.

Attempts to reduce the water content of DMF to 40 ±20 ppm by distillation were temporarily unsuccessful. A step by step monitoring of the purification process on the spinning band column therefore appeared advisable. The following procedure to purify a spectrograde material was employed: (1) a 700 cc batch of reagent grade DMF was dried by allowing it to contact molecular sieves for 3 days, (2) a preliminary batch of DMF was distilled to "wash" the column, and (3) the predried DMF was distilled from ground CaH_2 at reduced pressure. The following results were obtained by vapor-phase chromatographic analysis.

1. Initial sample (DMF #5, spectrograde quality, J. T. Baker, 220 ppm H_2O according to manufacturer's analysis)--690 ppm H_2O, 98 ppm organics

2. Predried sample--58 ppm H_2O, 32 ppm organics

3. First 100 cc of distillate--250 ppm H_2O, 110 ppm organics

4. Second 100 cc of distillate--143 ppm H_2O, 9 ppm organics

5. Collected batch, 400 cc, (DMF #5-2)--65 ppm H_2O, no organics detected

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The results appear to indicate that some water was introduced into the solvent during the distillation, because the water content of the predried starting material was less than that of the distillates obtained. For this reason, the redistillation of a previously purified, unsatisfactory batch was subsequently conducted at atmospheric pressure rather than at reduced pressure, and an acceptable product (DMF #5-1) resulted.

TABLE 1

CHARACTERIZATION OF DISTILLED SOLVENT BATCHES

Solvent Code	H_2O Content, ppm	Organics, ppm
PC #2-6	35	None
PC #2-7	20	35
PC #2-9	20	20
PC #2-10	20	None
PC #2-11	32	None
DMF #4-1	50	None
DMF #5-1	52	26
DMF #5-2	65	9
AN #1-2	40	None
AN #3-1	50	None
AN #4-1	48	None

Synthesis of Cupric Fluoride

The fluorination of copper arsenide (Cu_3As) was investigated in an effort to prepare pure cupric fluoride. Copper arsenide was obtained in 99.99 percent quality. The reaction

$$2\ Cu_3As\ +\ 11\ F_2 \rightarrow 6\ CuF_2\ +\ 2\ AsF_5 \tag{1}$$
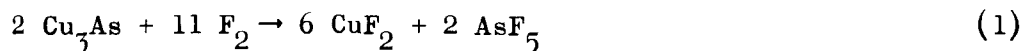
was expected to furnish the desired CuF_2 after removal of the volatile AsF_5.

Four direct fluorination attempts were made: static reactions at ambient and 250 C, and flow-by reactions at ambient and 60 C. In all cases, the Cu_3As appeared to have burned at a very high local temperature causing the copper to form a molten granule or clinker without becoming completely fluorinated.

Preparation of Tetrabutylammonium Tetraphenylboride (TBA·TPB)

Tetrabutylammonium tetraphenylboride was prepared by metathesis of tetrabutylammonium bromide (TBA·Br #1, Columbia Organic Chemicals Company, Inc., polarographic grade) and sodium tetraphenylboride (Na·TPB #1 Baker Adamson Laboratory Chemical, reagent grade) according to a procedure given in Ref. 5. Solutions of TBA·Br and Na·TPB in conductivity water were prepared (0.1 mole in 500 cc). The solution of Na·TPB was allowed to drip through a fine filter into the TBA·Br solution. The bulky white precipitate was filtered and dried. The material was recrystallized three times from acetone and dried under vacuum at room temperature for 6 days. This material was subsequently recrystallized and dried under vacuum at 50 C for 2 days. It was given the designation TBA·TPB #1.

Tetrabutylammonium Bromide (TBA·Br)

Polarographic grade tetrabutylammonium bromide (Columbia Organic Chemicals Company, Inc.), TBA·Br #1, was used for determination of the equivalent conductance at infinite dilution, Λ_0. This chemical, as well as TBA·TPB, was not analyzed because impurities possibly present at moderate concentrations would have negligible effects in these dilute solutions used for determining Λ_0 values.

Tetramethylammonium Fluoride (TMA·F)

TMA·F #2 (Aldrich Chemical Company, Inc.) was used for a determination of the sonic velocity of a TMA·F/PC electrolyte. This chemical has not yet been characterized.

NUCLEAR MAGNETIC RESONANCE STRUCTURAL STUDIES

LiClO$_4$ in Propylene Carbonate, Dimethyl Formamide and Acetonitrile

The Cl^{35} resonance was investigated using a broadline spectrometer and a magnetic field of 10 Kgauss in:

> 1 M LiClO$_4$ #2/PC #2-7
> 1 M LiClO$_4$ #2/DMF #3-3
> 1 M LiClO$_4$ #2/AN #4-1

All three lines exhibit virtually identical characteristics. They are narrow (1 M LiClO$_4$/PC was previously reported in Ref. 4) and have a chemical shift relative to the chloride ion in a 3 M LiCl/H$_2$O reference of approximately 1000 ppm. Calibrations are in progress to determine the magnitude of this shift more accurately.

The narrow line shows that the Cl^{35} site has at least tetrahedral symmetry. Furthermore, the chemical shift of the ClO$_4^-$ ion relative to the chloride ion in concentrated HCl is reported in Ref. 6 as 970 ppm. Thus, both the line characteristics and the chemical shift give evidence of the presence of the expected ClO$_4^-$ ion in all of these specimens

AlCl$_3$ and LiCl in Acetonitrile

The Al^{27} line has been investigated using a broadline NMR spectrometer at a magnetic field of 7.17 Kgauss in 1 M AlCl$_3$ #3/AN #4-1 solutions containing different concentrations of LiCl #2 and LiClO$_4$ #2. Measurements were made initially in specimens contained in screw-top tubes. However, white precipitates formed in some of the specimens, apparently because of screw-top seal

"breathing", thus making the results questionable. New specimens were prepared and the top of the tube was sealed by fusing the glass. No precipitates have formed in these specimens. It had been previously reported (Ref. 4) that there were two aluminum species found in 1 M $AlCl_3$/AN and that the addition of LiCl to the point of saturation removed the coordinating species. It is hypothesized that the following "reaction" occurs in 1 M $AlCl_3$/AN:

$$4 \ AlCl_3 + n \ AN \rightarrow Al^{+++} \ [AN]_n + 3 \ AlCl_4^- \tag{2}$$
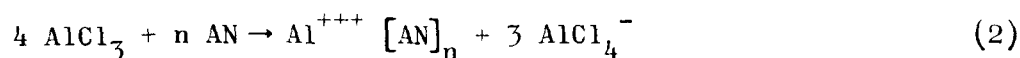
where $[AN]_n$ represents the n acetonitrile molecules coordinated by the Al^{+++} ion ($AlCl_4^-$ may or may not be solvated). This hypothesis is based upon the fact that both Al^{27} lines are quite narrow requiring high symmetry (at least tetrahedral) in the environment of the aluminum site in both Al containing species, and that the approximate relative population as taken from the maximum of the recorded spectra (Fig. 1a) is three to one. Furthermore, the removal of the coordinating species by the addition of LiCl to 1 M $AlCl_3$ can be explained if the chloride ions, introduced by the LiCl, interact with the Al^{+++} to form more $AlCl_4^-$. Another "reaction" which could be hypothesized because it produces highly symmetric species is:

$$2 \ AlCl_3 + n \ AN \rightarrow Al^{+++} \ [AN]_n + AlCl_6^{---} \tag{3}$$
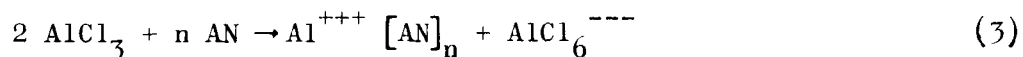
However, the ratio of species in this case is one to one which is not that shown by the Al^{27} spectra. As a further check on the validity of reaction 2, the Al^{27} spectrum was taken for several LiCl concentrations. The results are shown in Fig. 1. Using the maximum derivative as a measure of the line intensity, the ratio of the line intensity from the coordinating species to the total intensity from both species is plotted in Fig. 2 as a function of the LiCl concentration. The solid line represents the theoretical ratio of

The magnetic field is 7.17 Kgauss and increases from left to right. The two lines in (a), (b), and (c) are separated by approximately 0.8 Gauss.

Figure 1. Al^{27} Nuclear Magnetic Resonance in 1 M AlCl$_3$/AN Containing Various Concentrations of LiCl (a) No LiCl, (b) 0.25 M LiCl, (c) 0.5 M LiCl, (d) 0.9 M LiCl (saturated at 25 C)

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THE RELATIVE POPULATION WAS DETERMINED
FROM THE PEAKS OF THE DERIVATIVE CURVES
OF THE SPECTRA SHOWN IN FIG. 1
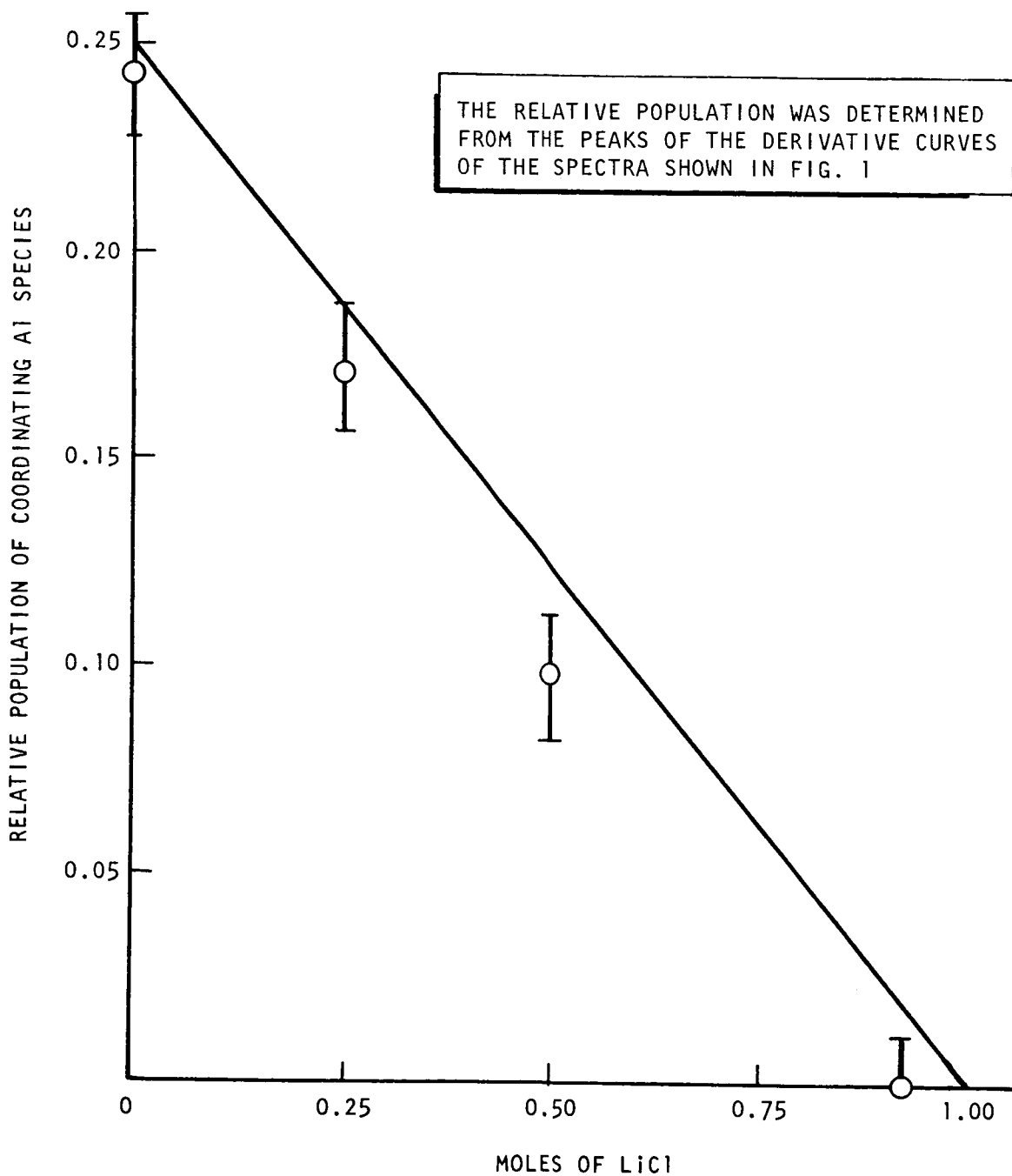
Figure 2. Approximate Relative Population of Coordinating Al Species
in 1 M AlCl$_3$/AN as a Function of Added LiCl.

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coordinating species to total species as based upon the reaction

$$x \text{ LiCl} + \text{Al}^{+++} [\text{AN}]_n \rightarrow x \text{ Li}^+ + \frac{x}{4} \text{AlCl}_4^- + \frac{x}{4} n \text{ AN} + \left(1 - \frac{x}{4}\right) \text{Al}^{+++} [\text{AN}]_n \quad (4)$$
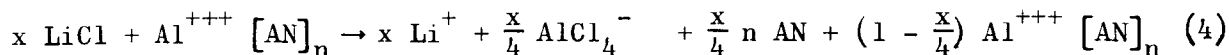
i.e., LiCl interacts to form AlCl_4^- ions. The line width of the coordinating species is somewhat greater than that of the noncoordinating species so that using the maxima of the recorded spectra to determine population ratio should result in an underestimate of the relative coordinating species population and the data points should fall somewhat below the theoretical line. The agreement of the data points with the theoretical curve is quite good, and substantiates reaction 4.

On the basis of these results, another experiment was conducted. As discussed previously, when LiClO_4 is dissolved in acetonitrile ClO_4^- is present. Two specimens were prepared with LiClO_4 rather than LiCl. It was thought that because no Cl^- ions were formed and because ClO_4^- is a relatively inert ion, that the population of the coordinating species would not be reduced showing the importance of the Cl^- in the depletion of the coordinating species. The Al^{27} line is shown in Fig. 3 for 0.5 M LiClO_4 #2 + 1 M AlCl_3 #3/ AN #4-1 and 1 M LiClO_4 #2 + 1 M AlCl_3 #3/AN #4-1. As can be seen, contrary to expectation, these spectra are different from the Al^{27} spectra of 1 M AlCl_3 #3/AN #4-1 shown in Fig. 3a. Also, the H^1 resonance indicates that the population of the coordinating species is decreased by the addition of LiClO_4. Thus, addition of LiClO_4 to 1 M AlCl_3/AN has a similar effect as LiCl but LiClO_4 is not as effective. Whereas 0.9 M LiCl reduces the population of the coordinating species to the point where it cannot be observed, 1 M LiClO_4 reduces the population approximately the same extent as 0.5 M LiCl. These results are tentatively explained on the basis of the formation of $\text{Al}(\text{ClO}_4)_4^-$ or $\text{Al}(\text{ClO}_4)_6^{---}$ species. The ClO_4^- ion is not generally considered to be a complexing species; in these solutions, however, it does appear to complex, though much less effectively than the Cl^- ion. Thus, in the
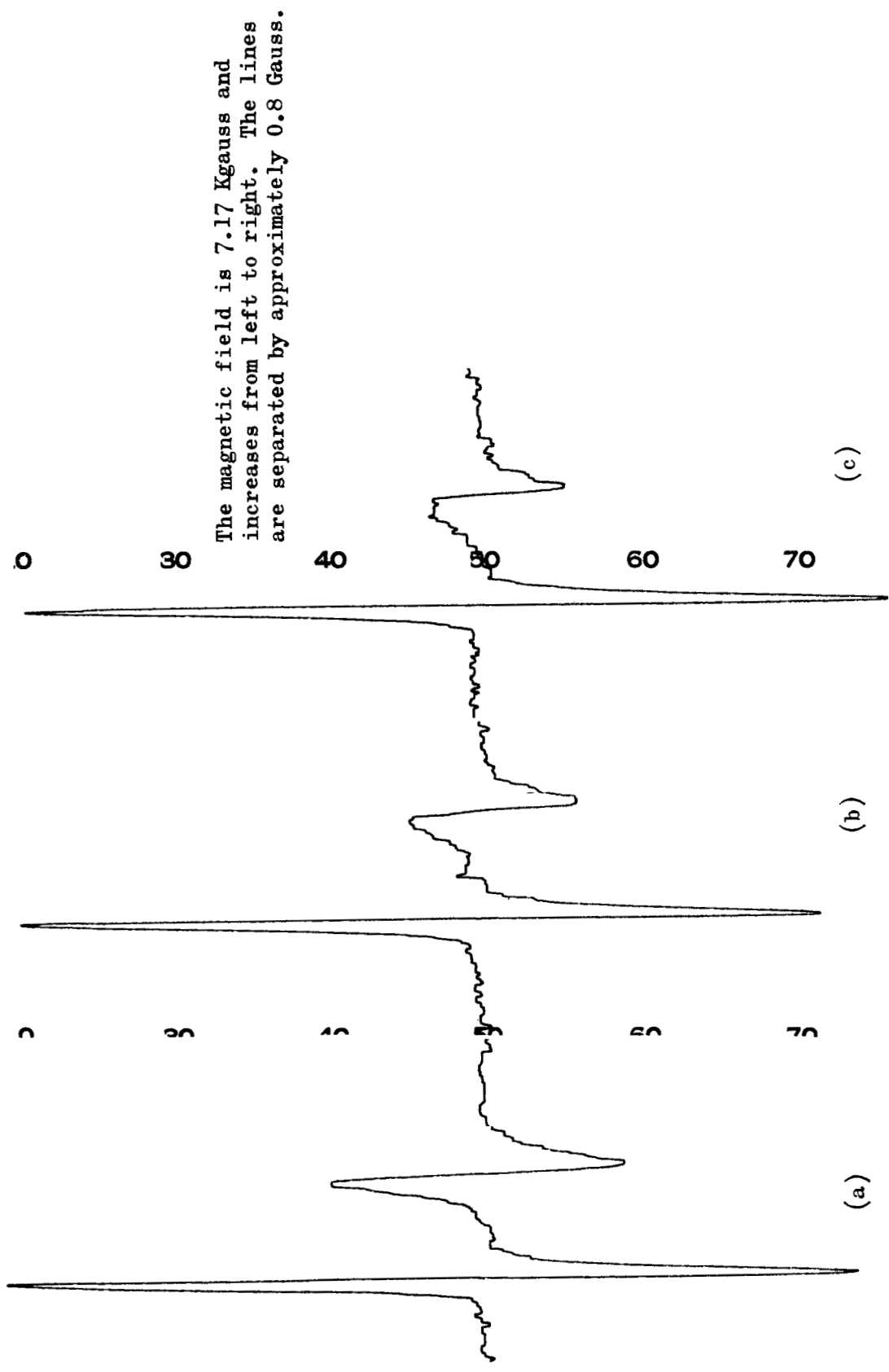
The magnetic field is 7.17 Kgauss and increases from left to right. The lines are separated by approximately 0.8 Gauss.
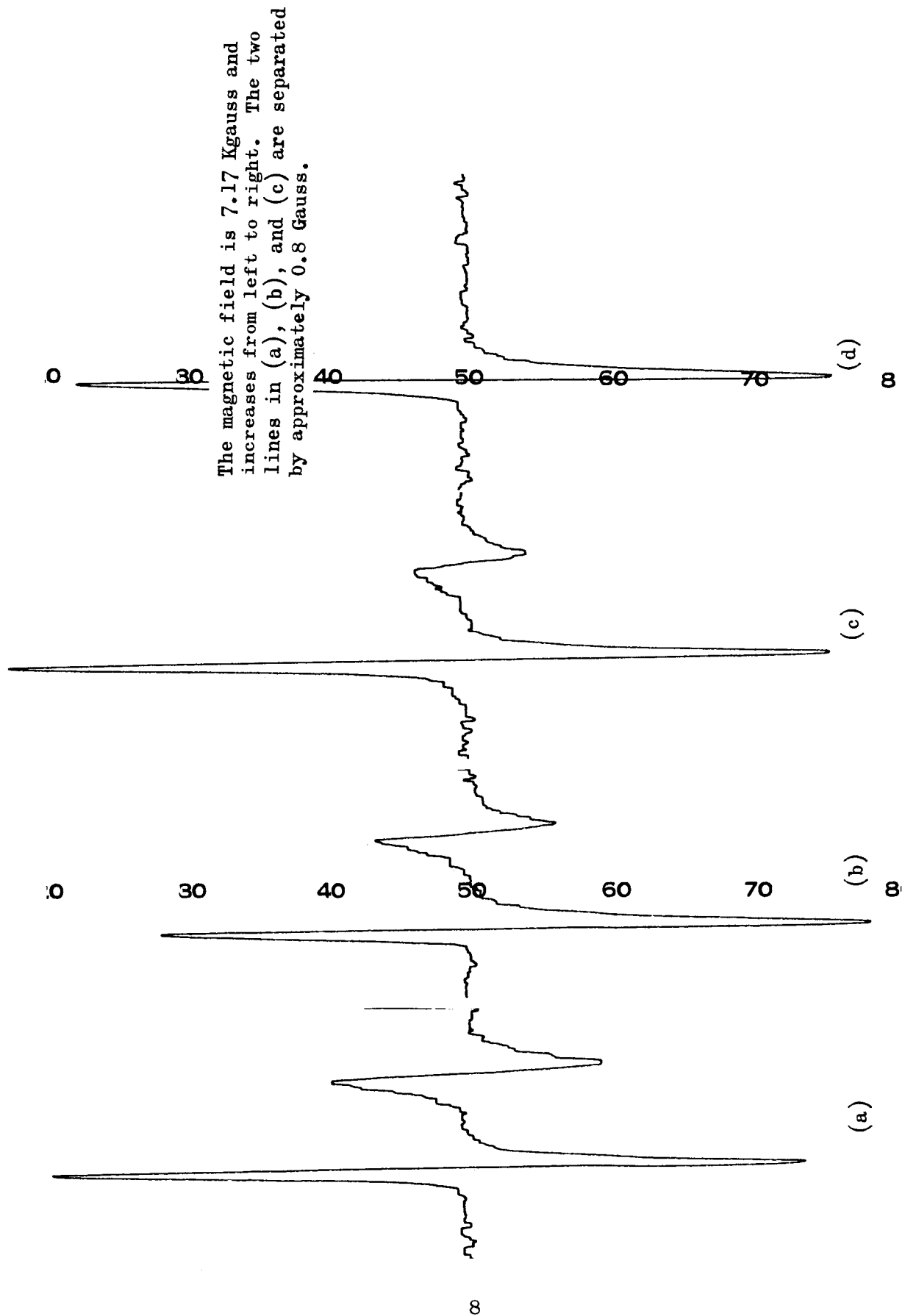
0   30   40   50   60   70

(c)

(b)

(a)

Figure 3. Al^{27} Nuclear Magnetic Resonance in 1 M $AlCl_3/AN$ Containing Various Concentrations of $LiClO_4$, (a) No $LiClO_4$, (b) 0.5 M $LiClO_4$, (c) 1 M $LiClO_4$

1 M $LiClO_4$ + 1 M $AlCl_3$/AN system, there would be three aluminum-containing species: Al^{+++} $[AN]_n$, $AlCl_4^-$, and $Al(ClO_4)_4^-$ (or $Al(ClO_4)_6^{---}$). It was expected that three Al lines would result but this was not observed. This could be explained if the difference of the chemical shift of the Al^{27} line in the anionic species is so small that separate lines could not be resolved. However, unless more information is obtained, this explanation must remain speculative.

$CuCl_2$ and LiCl in Dimethyl Formamide

Because of some unexpected findings, the proton line in the $CuCl_2$ + LiCl/DMF system has been studied extensively during this report period using the high resolution spectrometer. The H^1 spectrum of 1 M $CuCl_2$ #2/DMF #3-3 was reported (Ref. 4) as observed shortly after the specimen was prepared. An experiment was planned wherein LiCl would be added to the 1 M $CuCl_2$/DMF system to determine whether the population of species would be changed as in the LiCl + $AlCl_3$/AN and LiCl + $AlCl_3$/PC systems. A spectrum from the original 1 M $CuCl_2$ #2/DMF #3-3 was recorded as a reference for comparison with that to be obtained from several specimens containing LiCl. The spectrum obtained was, however, completely different from the original spectrum. Whereas the original spectrum consisted of two very broad peaks (refer to Ref. 4, Fig. 3), the new spectrum as shown in Fig. 4 is very similar to that obtained from pure DMF (refer to Ref. 4, Fig. 6) except that the lines are somewhat broadened, and there are small downfield shifted peaks similar to those found when $AlCl_3$ is dissolved in DMF (however, the shifts observed in the 1 M $CuCl_2$/DMF are considerably larger). Thus, over an extended period of time (approximately 4 months) the species present in this specimen had apparently changed.

New specimens were prepared to verify this change. In the new 1 M $CuCl_2$ #2/ DMF #5-2 the spectrum was virtually identical to that found in the first

Vertical arrows point out lines not found
in pure DMF. Magnetic field increases from
left to right.
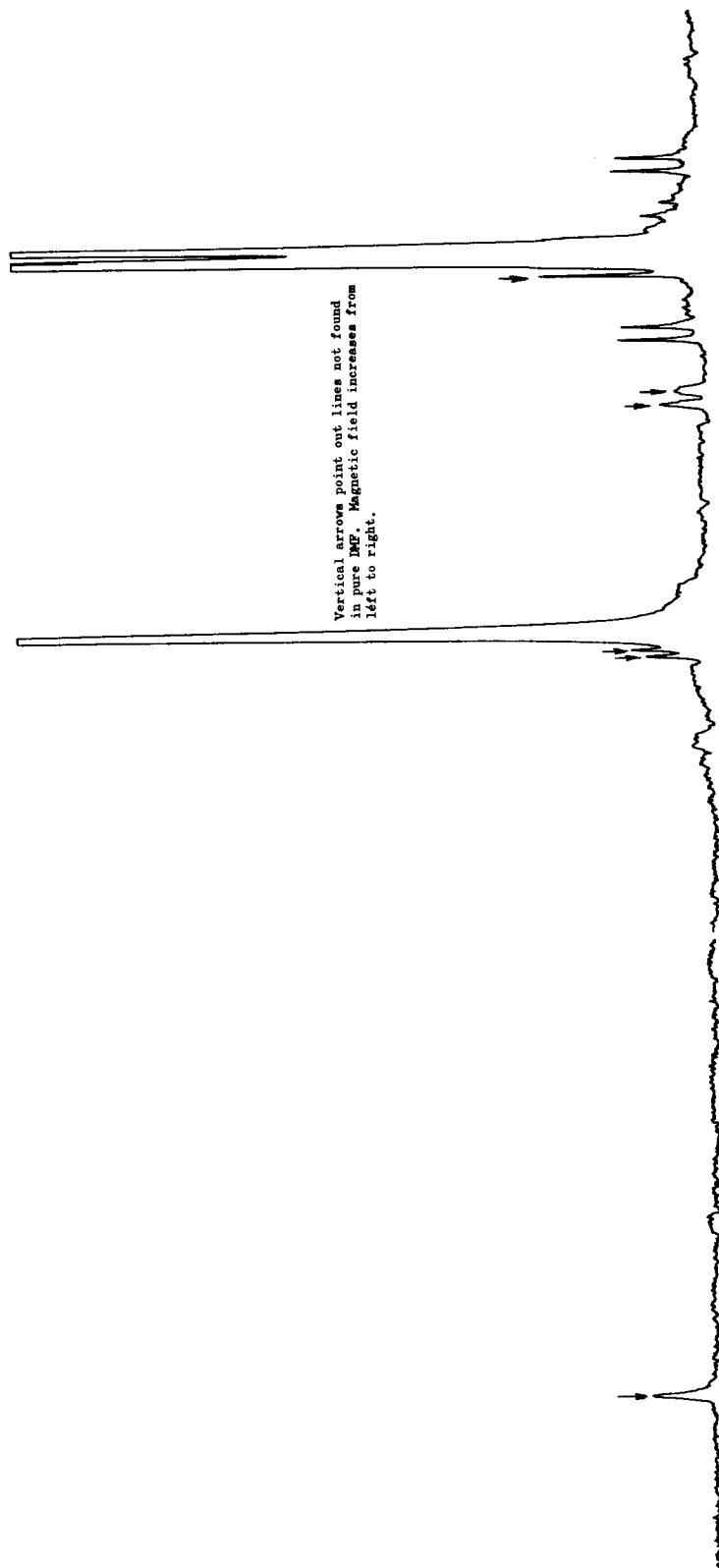
Figure 4. Proton Spectrum (High Resolution) in 1 M CuCl$_2$/DMF,
4 Months After Preparation

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1 M CuCl$_2$ #2/DMF #3-3, namely, two very broad peaks (one small and one large). The spectra from freshly prepared 1 M CuCl$_2$ #2/DMF #5-2, 0.5 M LiCl #2 + 1 M CuCl$_2$ #2/DMF #5-2, 1 M LiCl #2 + 1 M CuCl$_2$ #2/DMF #5-2 and 2 M LiCl #2 + 1 M CuCl$_2$ #2/DMF #5-2 shown in Fig. 5 through 8 are all similar except that as the concentration of LiCl is increased, the shift of the small peak from the large peak decreases and the line width decreases. The spectra from these specimens are now being recorded frequently to follow any changes. For example, the spectra obtained from these specimens 1 month after preparation are shown in Fig. 9 through 12. Thus far, the shift of the downfield peak in every specimen except the 2 M LiCl + 1 M CuCl$_2$/DMF specimen has been decreasing. In the 2 M LiCl + 1 M CuCl$_2$/DMF specimen, the shift has remained constant. These later spectra also show the appearance of additional small peaks. The shift of these peaks is also being determined. A plot of the major peak shifts as a function of time, to date, is presented in Fig. 13. Of particular interest is the fact that the shifts in 1 M CuCl$_2$/DMF, 0.5 M LiCl + 1 M CuCl$_2$/DMF, and 1 M LiCl + 1 M CuCl$_2$/DMF are approaching that of the 2 M CuCl$_2$/DMF specimen.

Magnetic Field Increases
From Left to Right
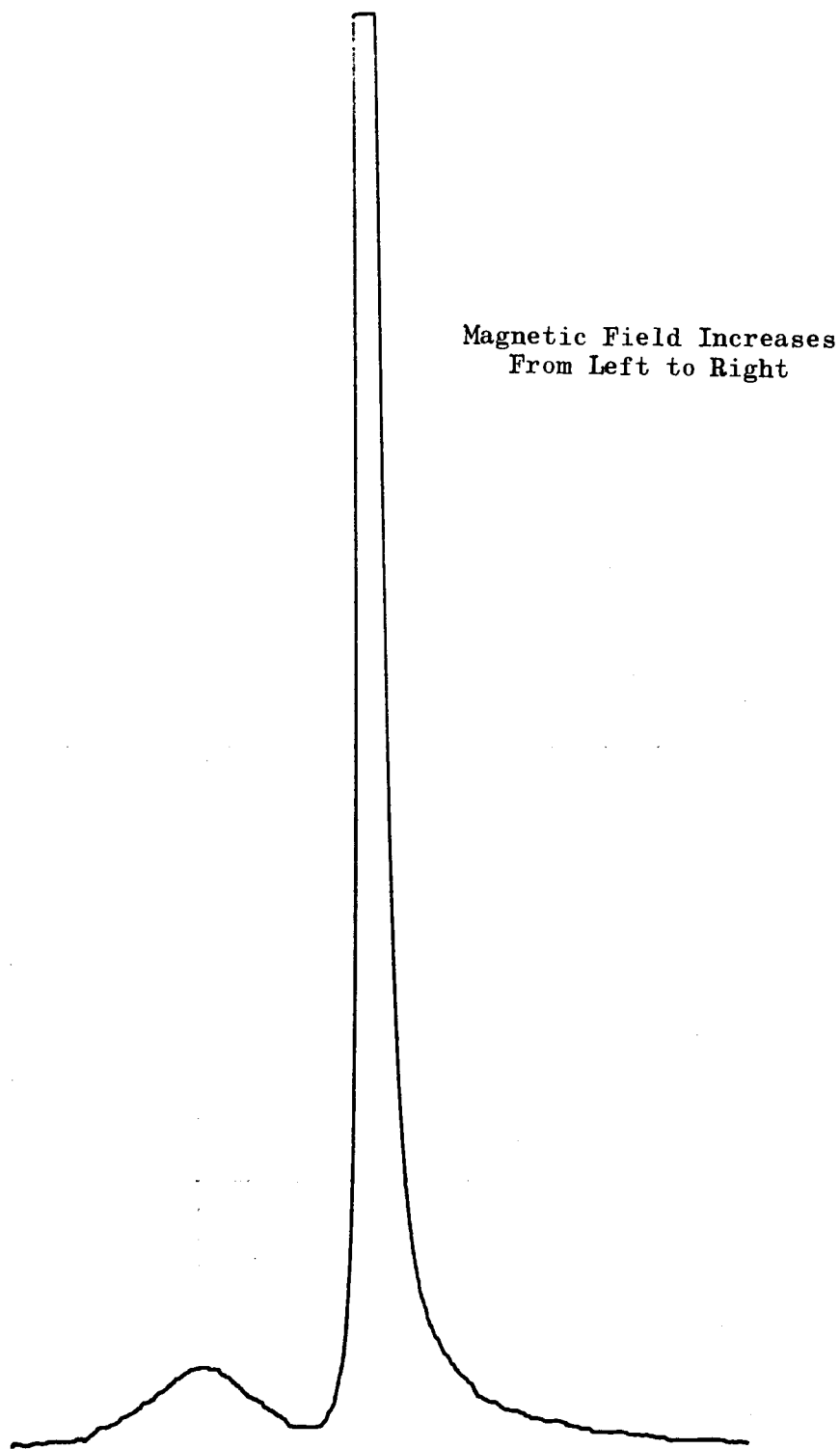
Figure 5. Proton Spectrum in 1 M $CuCl_2$/DMF,
Freshly Prepared Specimen

Magnetic Field Increases
From Left to Right
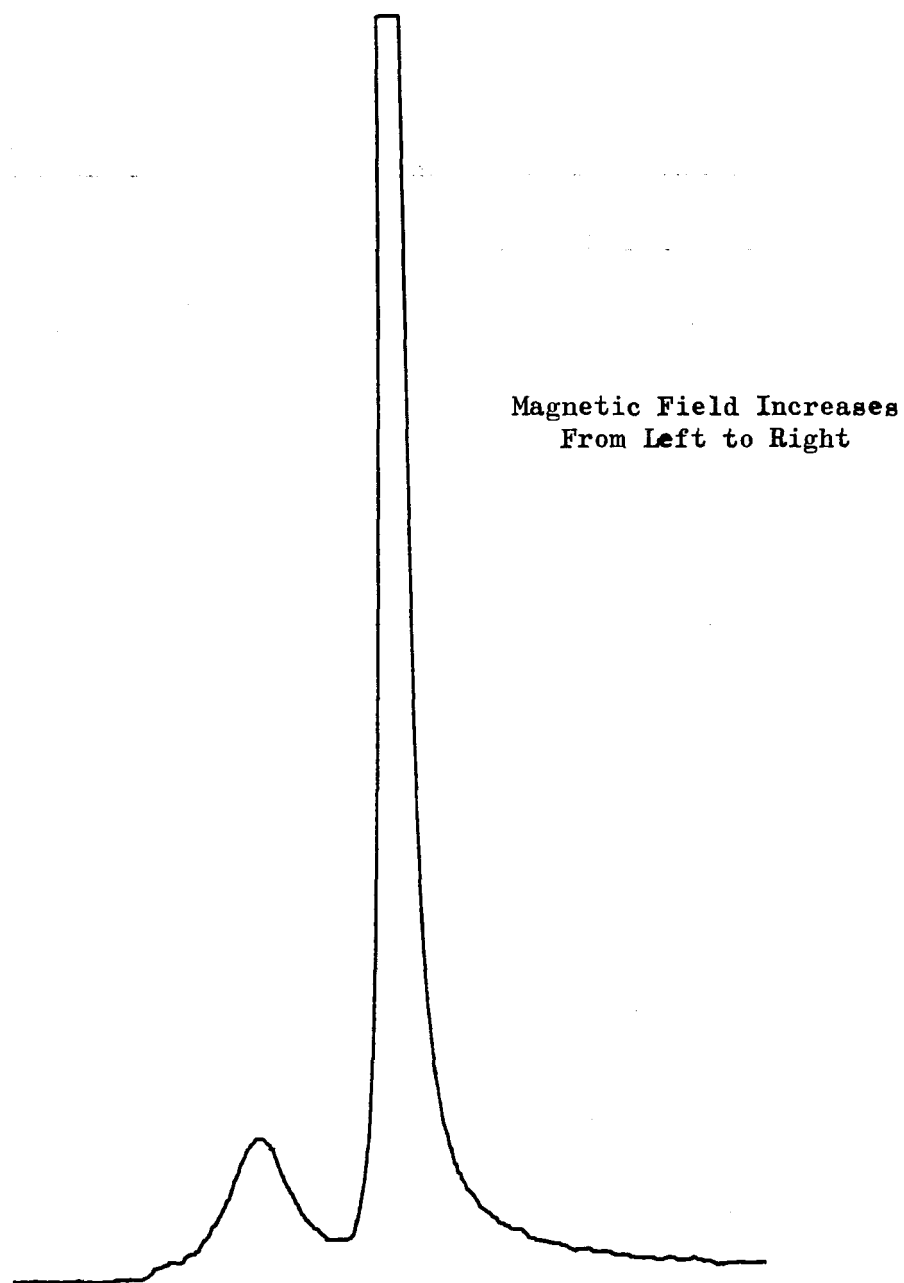
Figure 6.   Proton Spectrum in 0.5 M LiCl + 1 M $CuCl_2$/DMF, Freshly Prepared Specimen

Magnetic Field Increases
From Left to Right
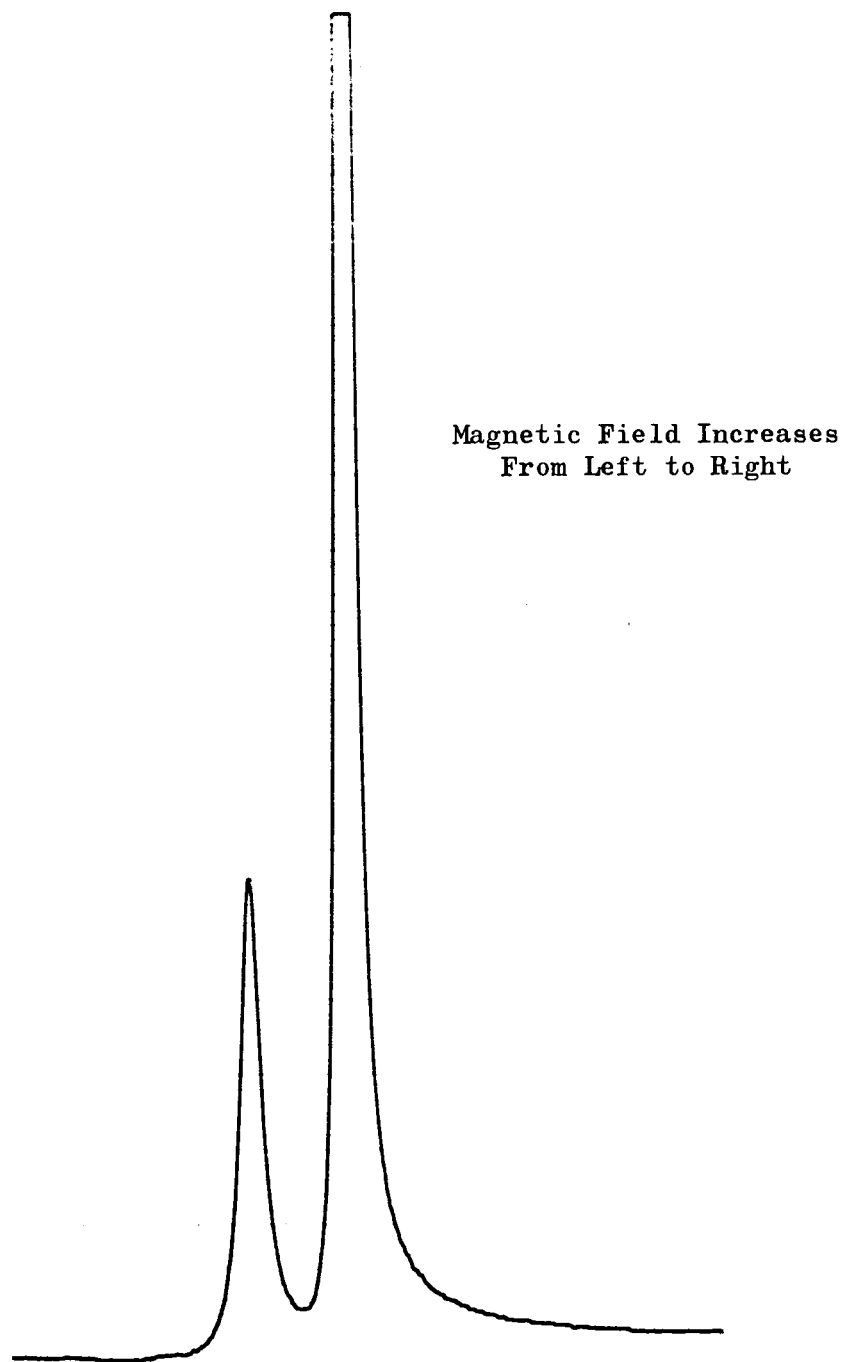
Figure 7.   Proton Spectrum in 1 M LiCl + 1 M CuCl$_2$/DMF,
Freshly Prepared Specimen

Magnetic Field Increases
From Left to Right
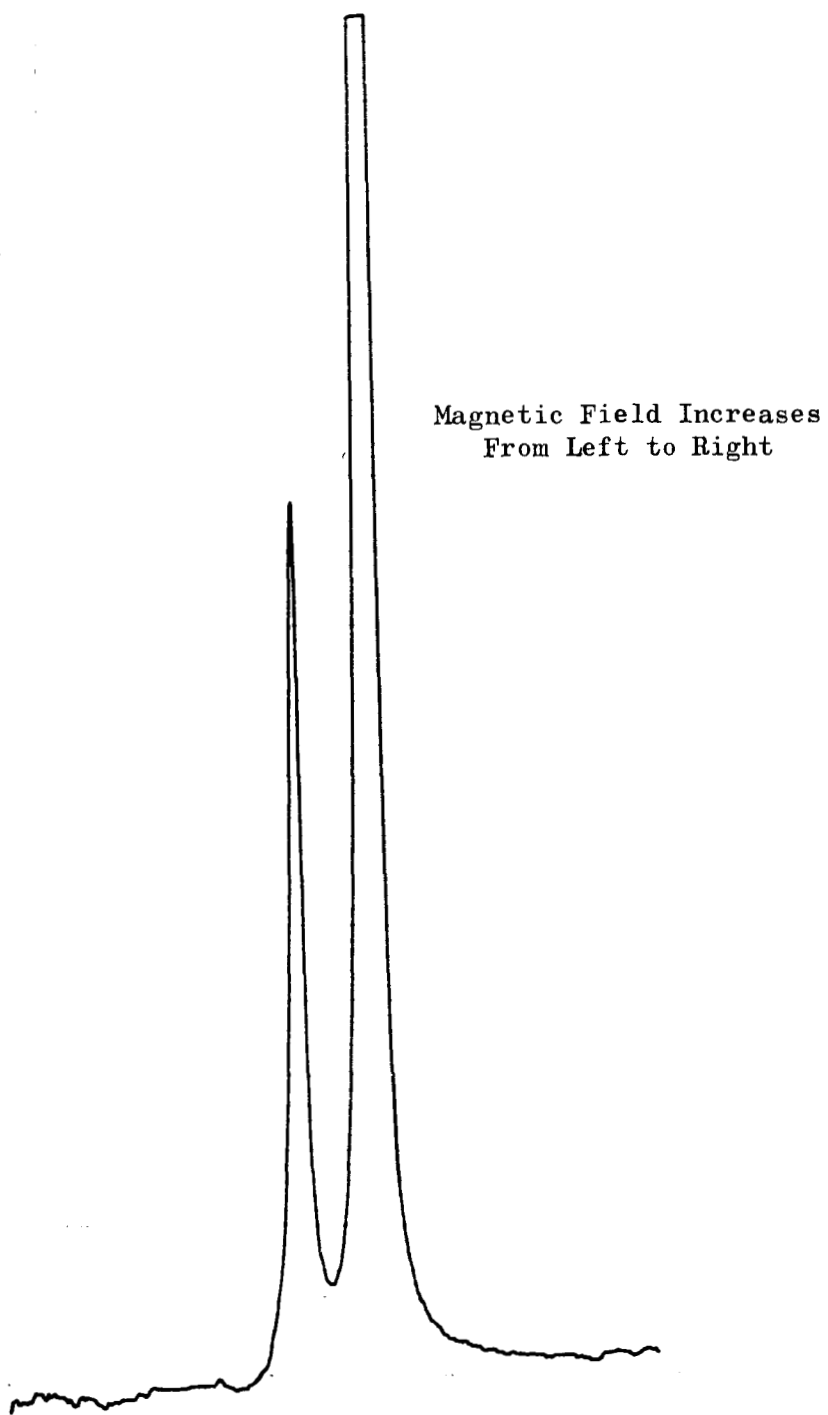
Figure 8. Proton Spectrum in 2 M LiCl + 1 M CuCl$_2$/DMF,
Freshly Prepared Specimen

Magnetic Field Increases
From Left to Right
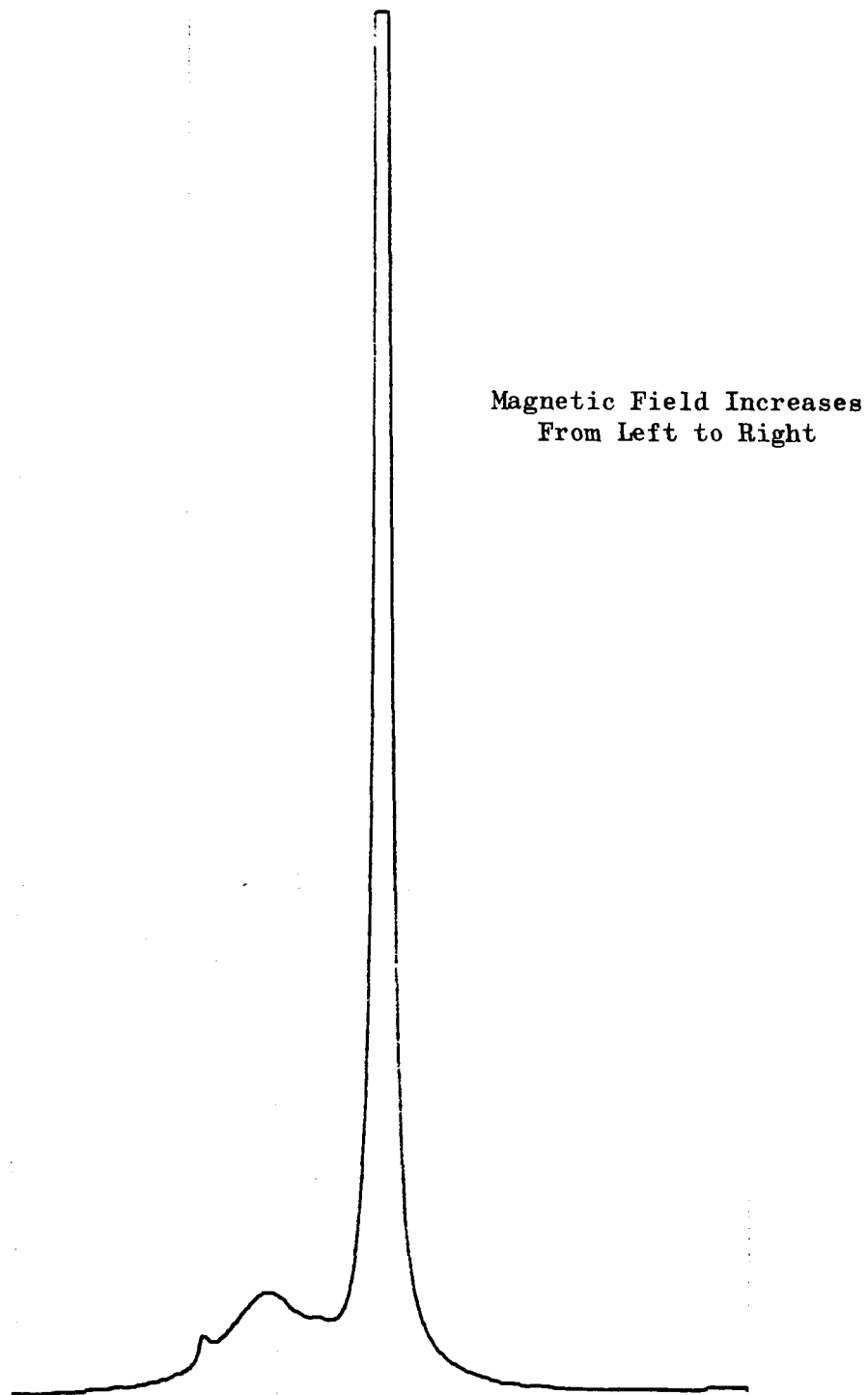
Figure 9. Proton Spectrum in 1 M CuCl$_2$/DMF, 1 Month
After Preparation

Magnetic Field Increases
From Left to Right
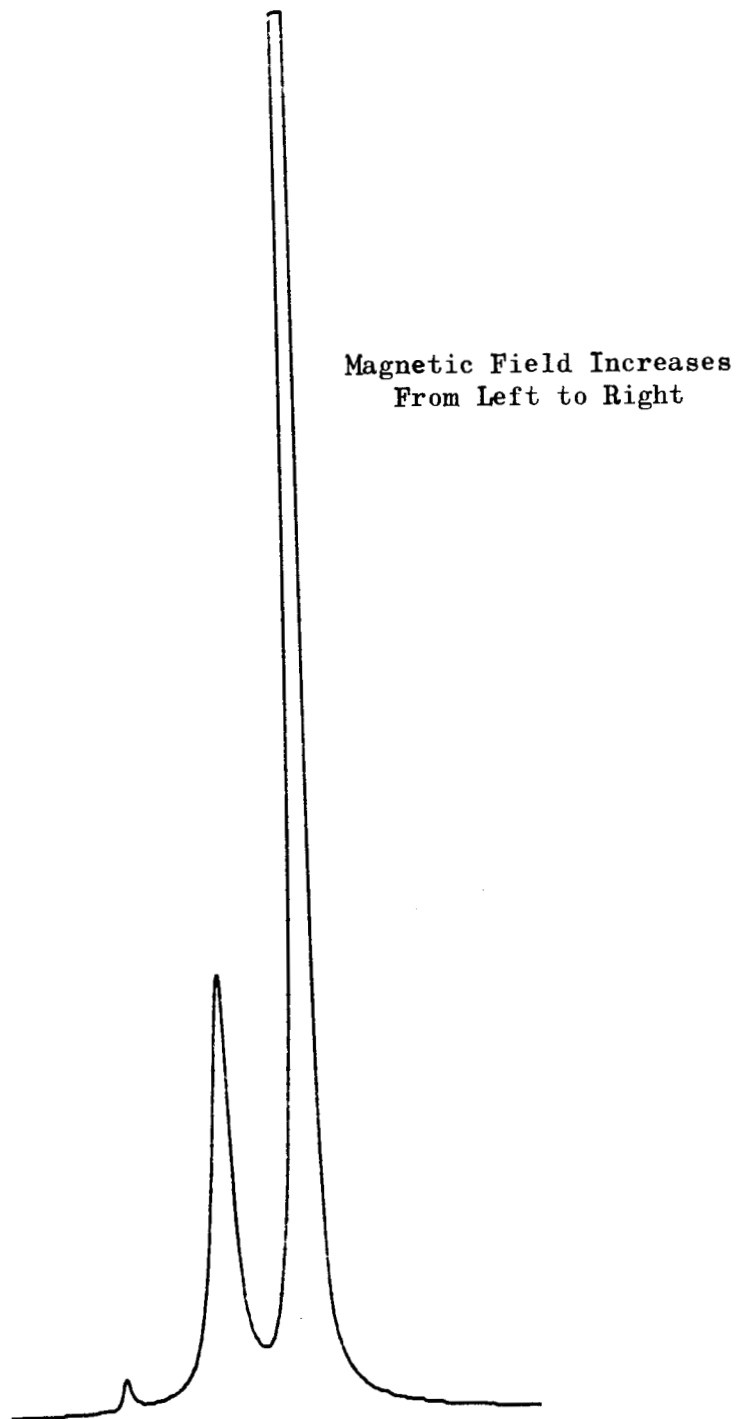
Figure 10. Proton Spectrum in 0.5 M LiCl + 1 M $CuCl_2$/DMF,
1 Month After Preparation

Magnetic Field Increases
From Left to Right
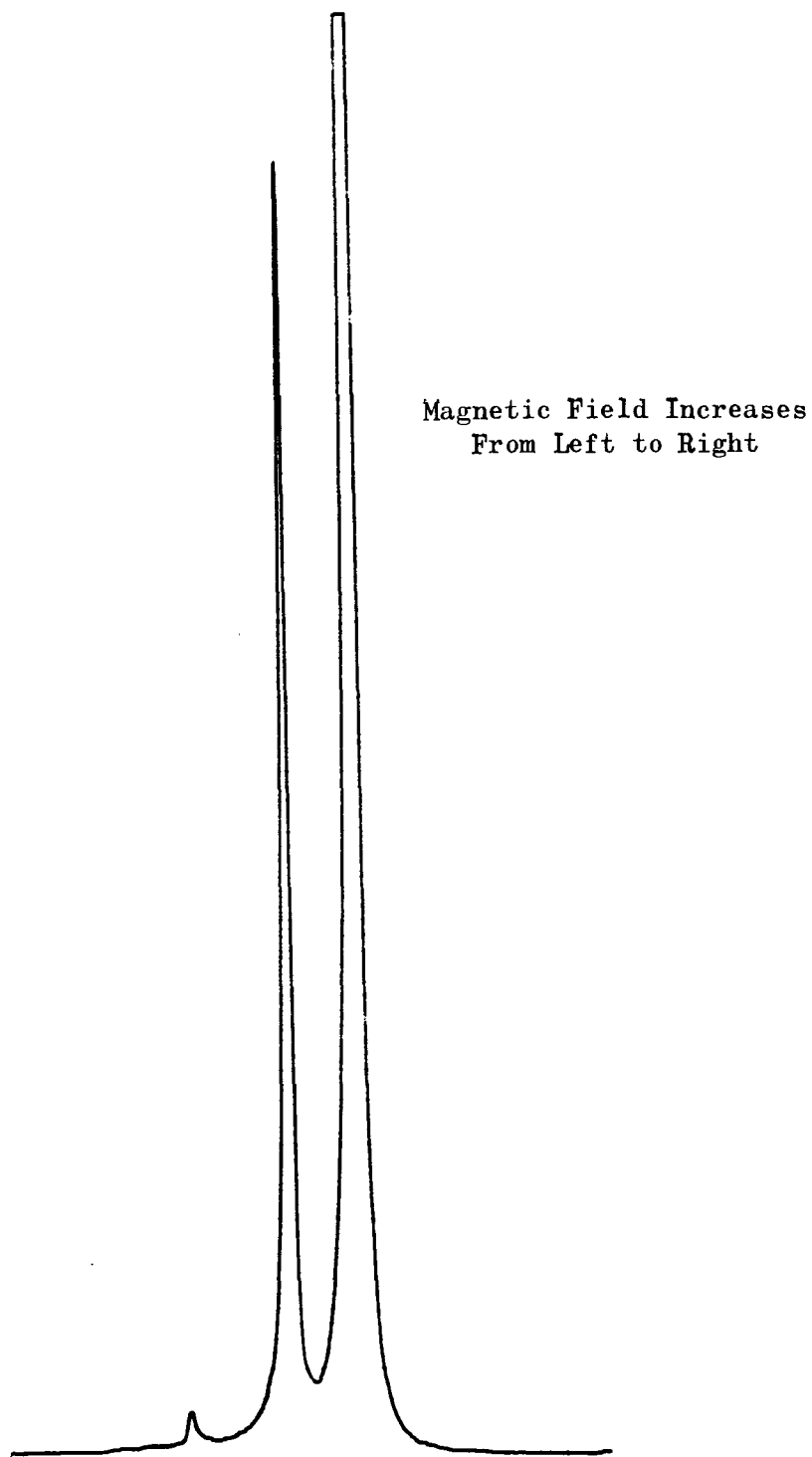
Figure 11. Proton Spectrum in 1 M LiCl + 1 M $CuCl_2$/DMF, 1 Month After Preparation

Magnetic Field Increases
From Left to Right
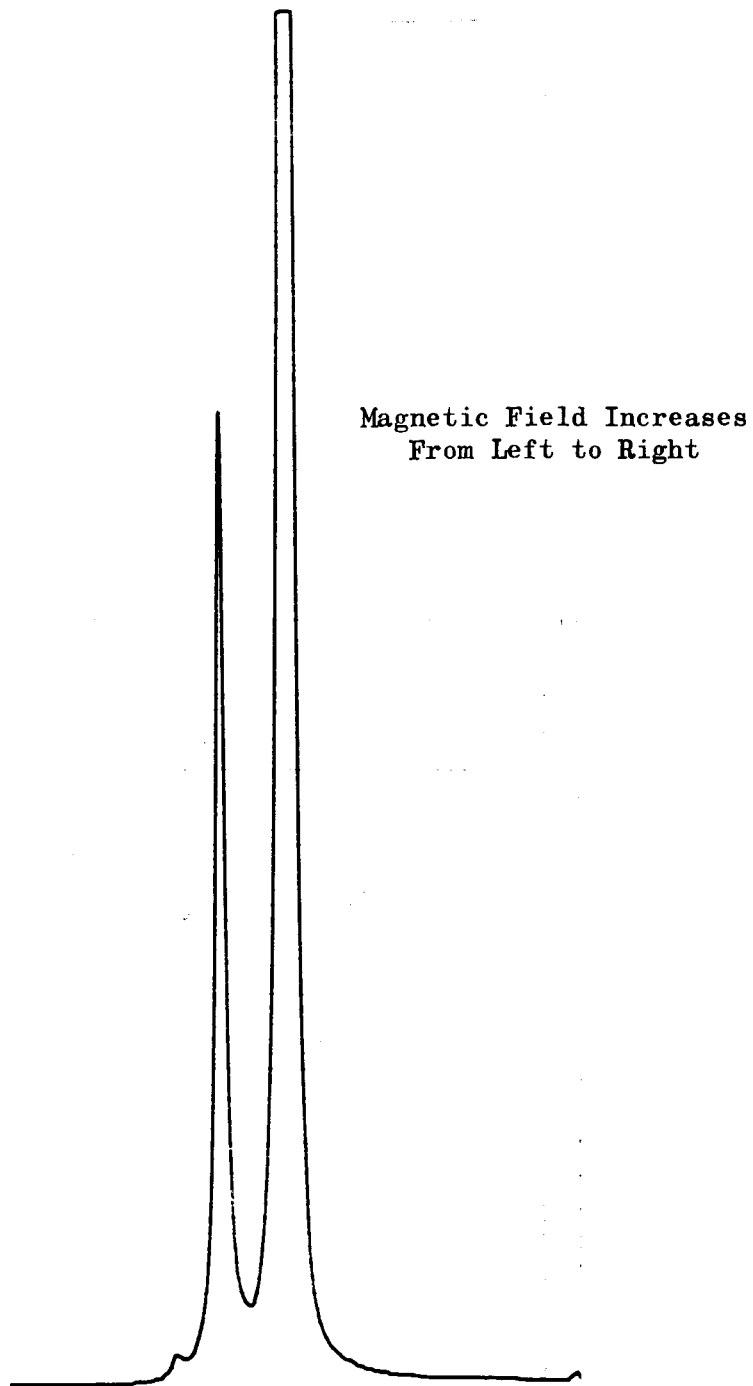
Figure 12. Proton Spectrum in 2 M LiCl + 1 M $CuCl_2$/DMF,
1 Month After Preparation

Figure 13.   Shift of Downfield Proton Peak as a Function of Time After Preparation of Various Concentrations of LiCl #2 in 1 M CuCl$_2$ #2/DMF #5-2

PHYSICAL PROPERTY DETERMINATIONS

Viscosity Measurements

Viscosity measurements on pure solvents were extended to 60 C as required for the interpretation of conductance data. The results, along with the 25 C values, are presented in Table 2.

Solubility Measurements

The solubility of LiCl in 1 M $LiClO_4$/PC #2-10 + 1000 ppm H_2O was determined as 0.071 M by chloride ion titration.

TABLE 2

VISCOSITIES OF SOLVENTS AT 25 and 60 C

Solvent	Temperature, C	Density, gm/cc	Viscosity, millipoise
PC #2-6	25	1.203	24.8*
PC #2-7	60	1.161	13.3
DMF #4-1	25	0.944	7.93**
DMF #5-1	60	0.910	5.35
AN #1-2	25	0.777	3.36*
AN #3-1	60	0.737	2.63

*Ref. 3
**Ref. 2

Conductance Measurements

The conductance measurements made during this report period are presented in Tables 3 through 10 and in Fig. 14 through 21.

The equivalent (molar) conductance values at infinite dilution, Λ_0, of solutions containing tetrabutylammonium tetraphenylboride (TBA·TPB) are of interest, because the anion and the cation of this compound have equal mobilities. This has been assumed by Fuoss and Hirsch (Ref. 7), and they used this compound to determine the molar conductances of individual ions in propylene carbonate.

The presently determined Λ_0 value (Fig. 14) of 17.2 $ohm^{-1}equ^{-1}cm^2$ at 25 C compares reasonably well with their Λ_0 value of 17.144 ±0.012 which was obtained by a more sophisticated extrapolation procedure (Ref. 7).

Prue and Sherrington (Ref. 8) based their determination of individual ion mobilities in dimethyl formamide on the Λ_0 value for tetrabutylammonium triphenylborofluoride, as suggested by Fowler and Kraus (Ref. 9). Their value for the molar conductance of the tetrabutylammonium ion ($\Lambda_0^+ = 26.2$ $ohm^{-1}equ^{-1}cm^2$) compares with the present value of $\Lambda_0^+ = 25.5$ determined by use of TBA·TPB (see below). Because the triphenylborofluoride anion is smaller than the tetraphenylboride anion, the somewhat higher value obtained by Prue and Sherrington can be explained. Fuoss and Hirsch believe that the requirement of equal size of anion and cation is much more approximated in tetrabutylammonium tetraphenylboride than in tetrabutylammonium triphenyl-borofluoride.

The equivalent conductances at infinite dilution calculated for individual ions from the preceding data are listed in Table 11 for propylene carbonate, in Table 12 for dimethyl formamide, and in Table 13 for acetonitrile. The values for compounds from which the values for individual ions were calculated

TABLE 3

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ)

OF TBA·TPB #1/PC #2-11

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
0.004566	0.06760	6.983×10^{-5}	15.2	1.228×10^{-4}	26.8
0.002282	0.04778	3.618×10^{-5}	15.7	6.351×10^{-5}	27.6
0.001142	0.03380	1.876×10^{-5}	16.2	3.290×10^{-5}	28.4
0.000571	0.02390	9.706×10^{-5}	16.5	1.700×10^{-5}	28.9
0.000286	0.01691	5.064×10^{-6}	16.7	8.853×10^{-6}	29.2
0.000143	0.01196	2.674×10^{-6}	16.7	4.671×10^{-6}	29.2
0		2.899×10^{-7}	Extrapolated: $\Lambda_o = 17.2$	4.899×10^{-7}	Extrapolated: $\Lambda_o = 30.0$

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

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CONCENTRATION (C), MOLAR

0.0004   0.0016   0.0036

$\Lambda_o = 30.0$

60 C

$\Lambda_o = 17.2$

25 C

EQUIVALENT CONDUCTANCE (Λ), OHM^{-1} EQU^{-1} CM^2

30
29
28
27
26

17.0

16.0

0   0.02   0.04   0.06

CONCENTRATION ($\sqrt{c}$), MOLAR$^{1/2}$
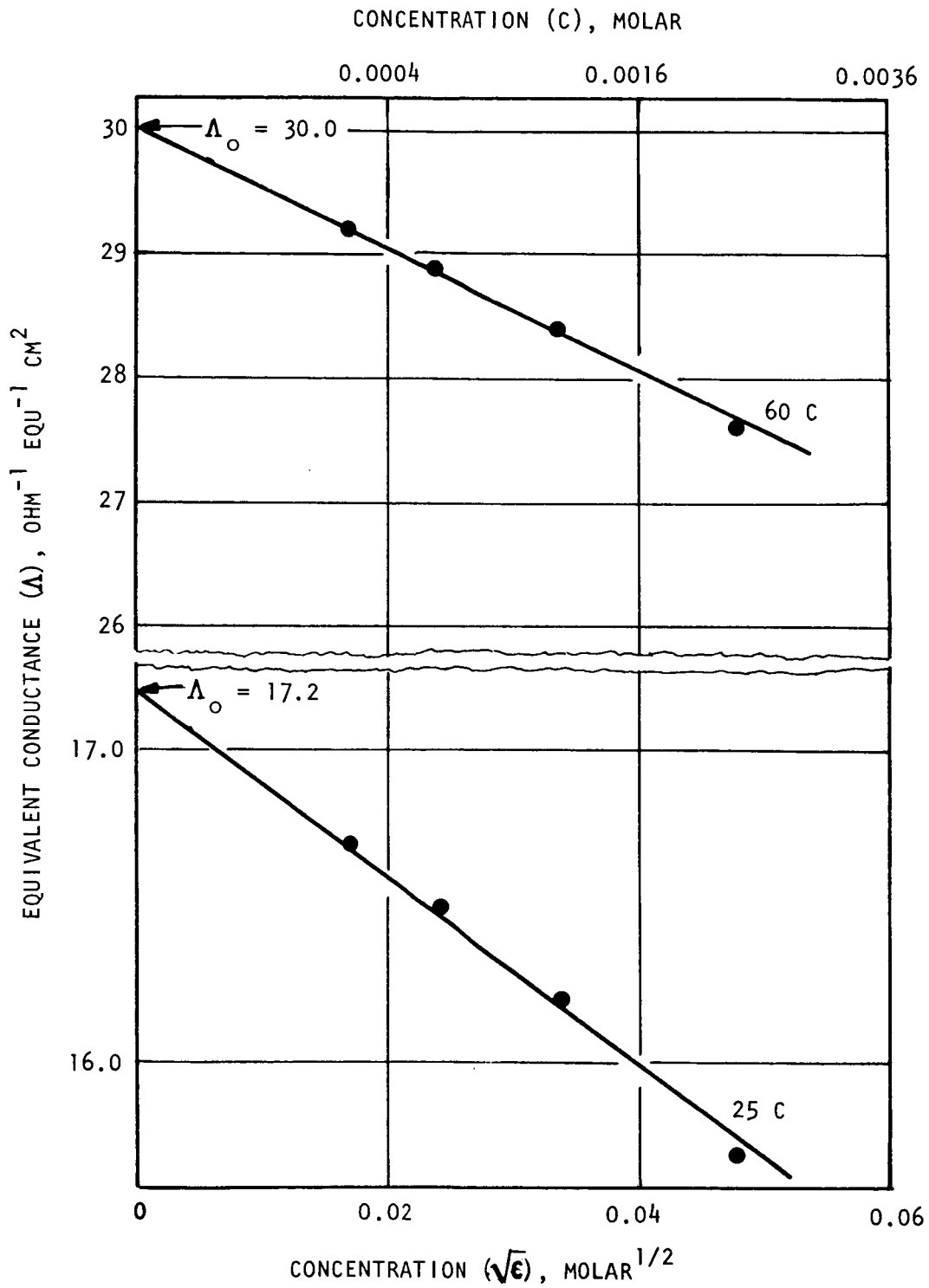
Figure 14.   Equivalent Conductance of TBA·TPB
in PC at 25 and 60 C

TABLE 4

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ)

OF TBA·TPB #1/DMF #5-1

Concentration (C), molar	$\sqrt{C}, \frac{1}{2}$ molar	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
0.005355	0.07285	2.300×10^{-4}	43.6	3.263×10^{-4}	60.7
0.002677	0.05171	1.203×10^{-4}	44.6	1.711×10^{-4}	63.4
0.001339	0.03659	6.273×10^{-5}	46.2	8.916×10^{-5}	65.6
0.000669	0.02587	3.261×10^{-5}	47.4	4.624×10^{-5}	67.2
0.000335	0.01830	1.728×10^{-5}	48.8	2.451×10^{-5}	69.3
0.000167	0.01292	9.193×10^{-5}	49.5	1.305×10^{-5}	70.3
0		9.266×10^{-7}		1.312×10^{-6}	
			Extrapolated: $\Lambda_o = 51.0$		Extrapolated: $\Lambda_o = 72.6$

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

CONCENTRATION (C), MOLAR

0.0004 0.0016 0.0036
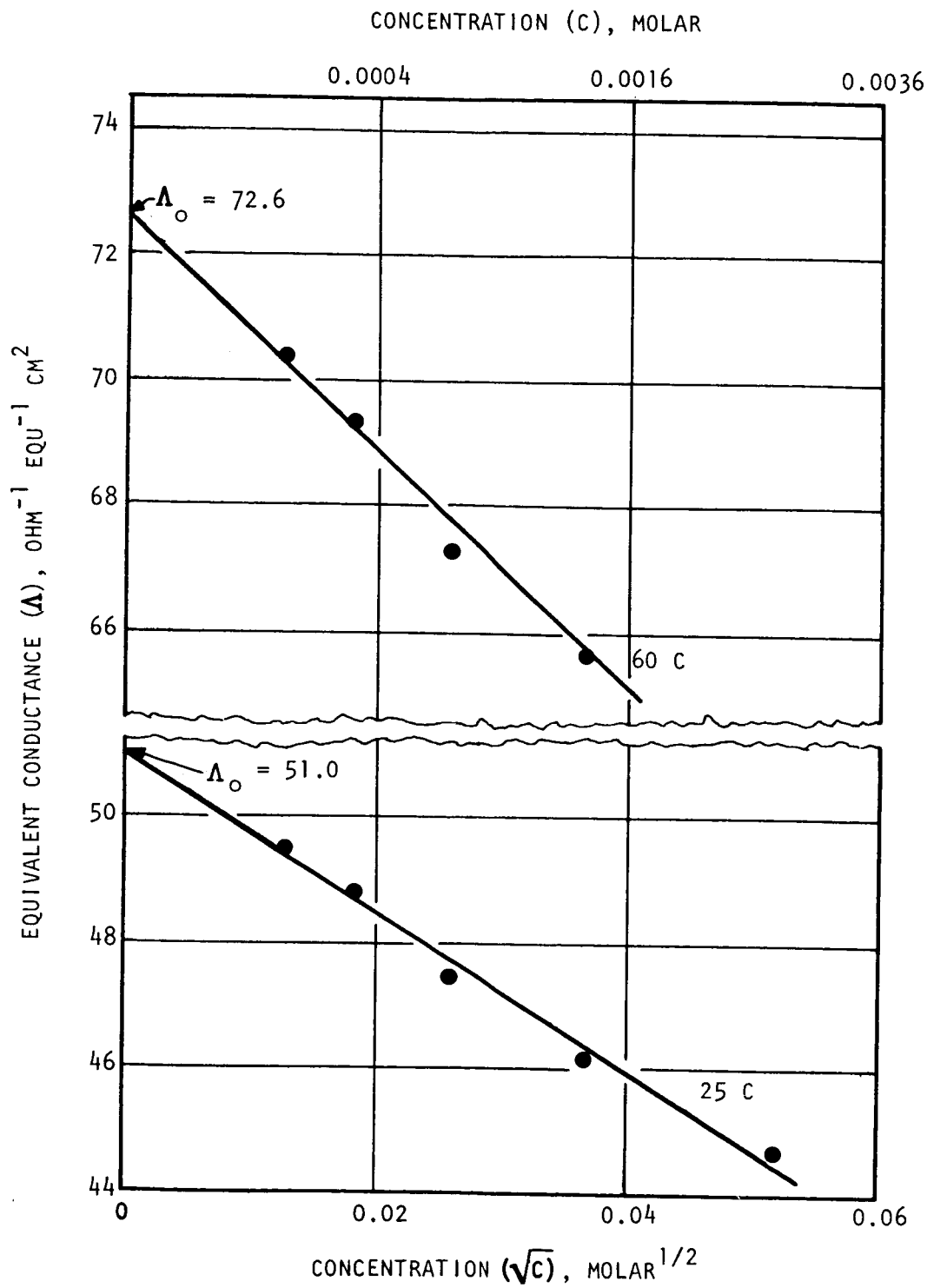
Figure 15. Equivalent Conductance of TBA·TPB in DMF at 25 and 60 C

TABLE 5

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ)

OF TBA·TPB #1/AN #4-1

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
0.003416	0.0584	3.498×10^{-4}	102.2	4.532×10^{-4}	132.4
0.001708	0.0413	1.820×10^{-4}	106.1	2.361×10^{-4}	137.6
0.000854	0.0292	9.453×10^{-5}	109.8	1.225×10^{-4}	142.3
0.000428	0.0207	4.879×10^{-5}	112.2	6.335×10^{-5}	145.7
0.000214	0.0146	2.535×10^{-5}	114.9	3.279×10^{-5}	148.6
0.000107	0.0103	1.326×10^{-5}	116.7	1.713×10^{-5}	150.7
0		7.747×10^{-7}		1.004×10^{-6}	
			Extrapolated: $\Lambda_o = 119.6$		Extrapolated: $\Lambda_o = 154.6$

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

Figure 16. Equivalent Conductance of TBA·TPB in AN at 25 and 60 C

TABLE 6

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ)

OF TBA·Br #1/PC #2-10

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
0.01044	0.1022	2.555×10^{-4}	24.4	4.343×10^{-4}	41.5
0.00522	0.07225	1.329×10^{-4}	25.3	2.263×10^{-4}	43.1
0.00261	0.05108	6.859×10^{-5}	26.0	1.165×10^{-4}	44.1
0.001305	0.03611	3.544×10^{-5}	26.5	6.020×10^{-5}	45.0
0.0006525	0.02554	1.836×10^{-5}	26.8	3.126×10^{-5}	45.7
0.0003262	0.01806	9.673×10^{-6}	27.0	1.652×10^{-5}	46.2
0.0001631	0.01277	5.282×10^{-6}	27.1	9.007×10^{-6}	46.3
0		8.562×10^{-7}		1.463×10^{-6}	
			Extrapolated: $\Lambda_o = 27.5$		Extrapolated: $\Lambda_o = 47.3$

Λ*: Equivalent conductance, corrected for conductance of pure solvent
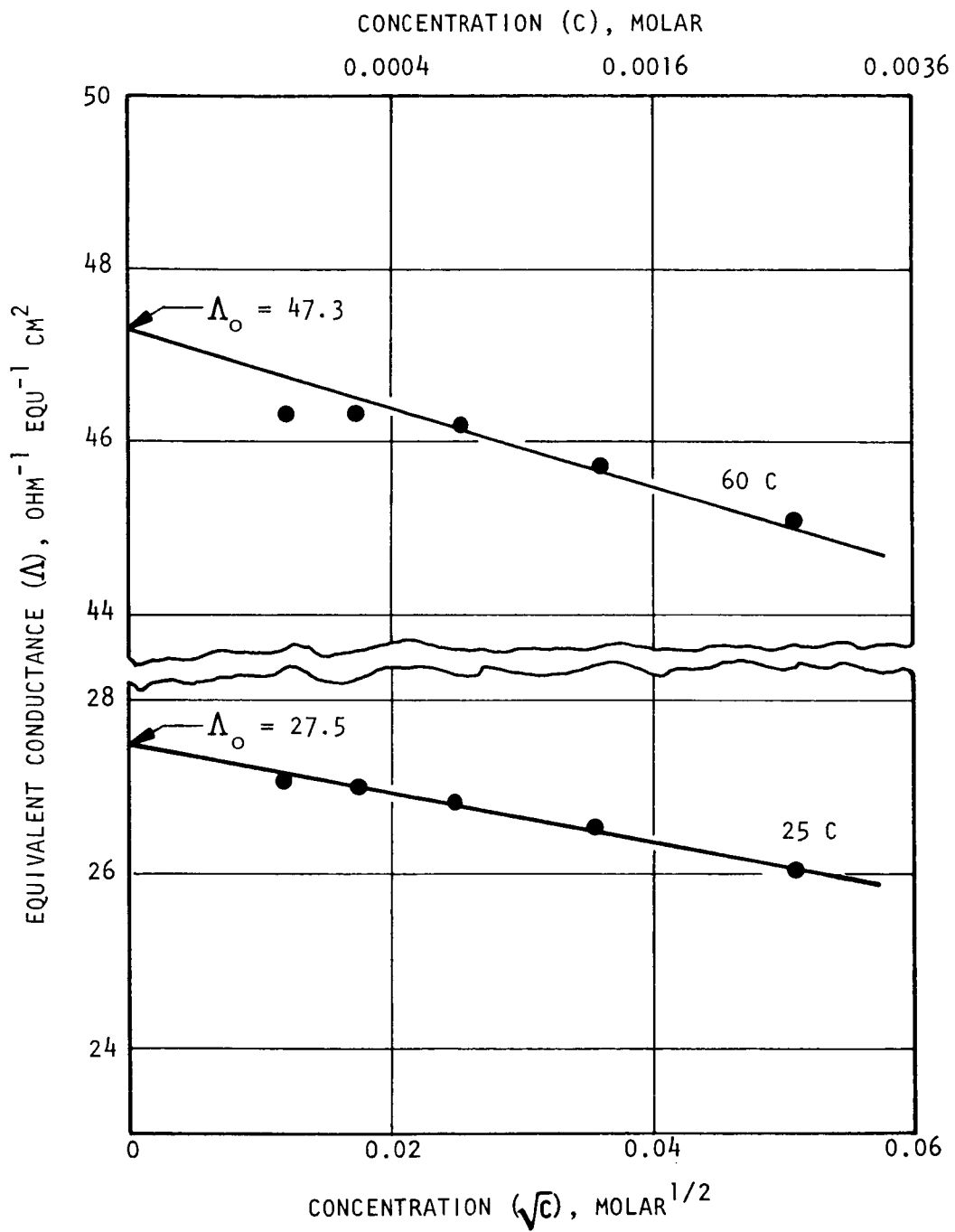
Figure 17.   Equivalent Conductance of TBA·Br
in PC at 25 and 60 C

TABLE 7

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ)

OF TBA·Br #1/DMF #5-2

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
0.00520	0.0721	3.544×10^{-4}	68.0	4.901×10^{-4}	94.1
0.00260	0.0510	1.863×10^{-4}	71.4	2.597×10^{-4}	99.5
0.00130	0.0361	9.725×10^{-5}	74.3	1.357×10^{-4}	103.6
0.000650	0.0255	5.133×10^{-5}	77.9	7.184×10^{-5}	109.1
0.000325	0.01803	2.690×10^{-5}	80.6	3.770×10^{-5}	113.0
0.000162	0.01275	1.358×10^{-5}	79.3	1.898×10^{-5}	110.8
0		6.870×10^{-7}	Extrapolated Λ_o = 87.0	9.749×10^{-7}	Extrapolated Λ_o = 122.4

Λ*: Equivalent conductance, corrected for conductance of pure solvent
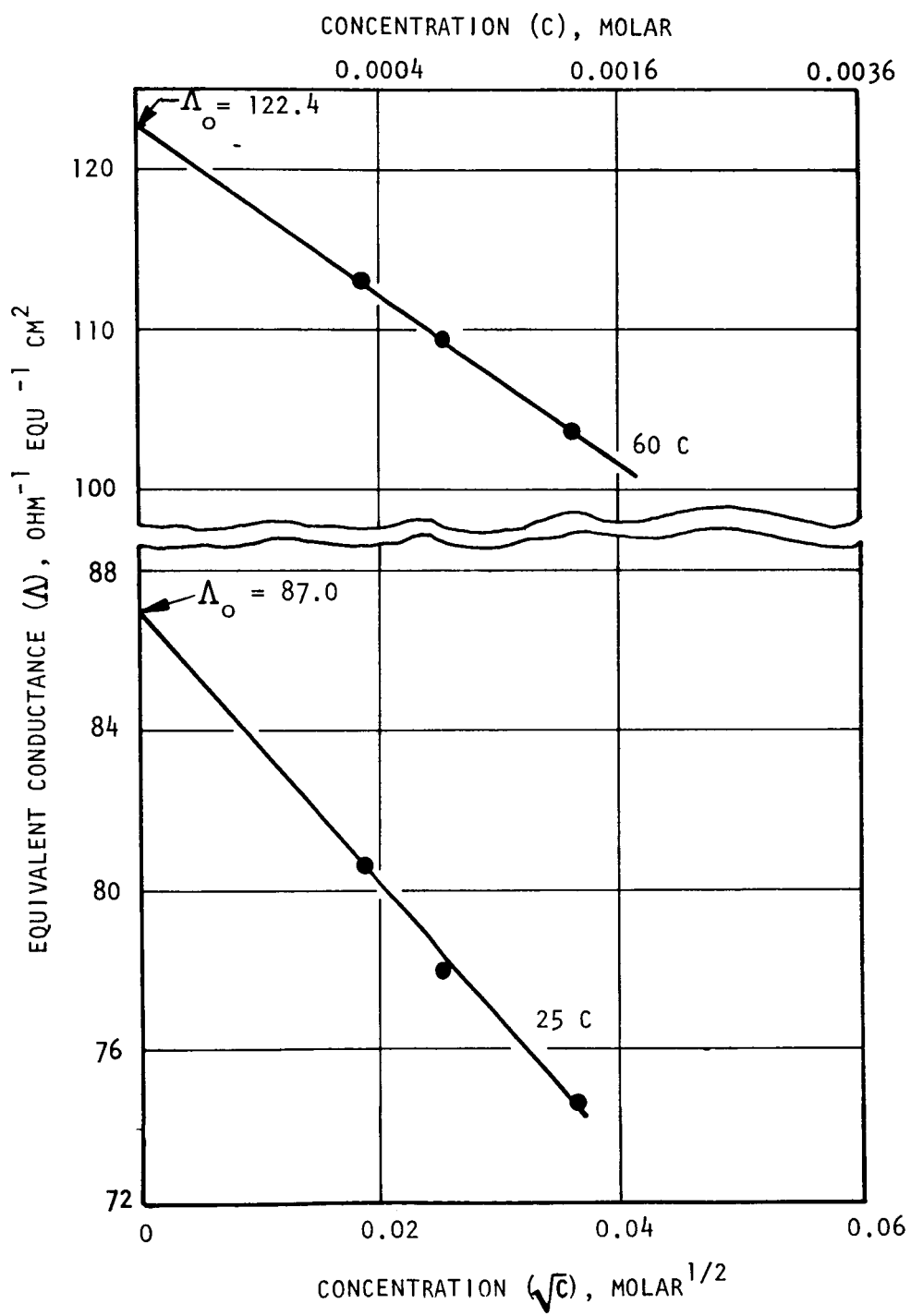
Figure 18.   Equivalent Conductance of TBA·Br in DMF
at 25 and 60 C

TABLE 8

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ)

OF TBA·Br #1/AN #4-1

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
0.006112	0.0782	8.189×10^{-4}	133.9	1.045×10^{-3}	170.8
0.003056	0.0552	4.306×10^{-4}	140.6	5.524×10^{-4}	180.4
0.001528	0.0391	2.250×10^{-4}	146.7	2.895×10^{-4}	188.8
0.000764	0.0276	1.163×10^{-4}	151.2	1.492×10^{-4}	194.0
0.000382	0.0195	5.969×10^{-5}	154.2	7.679×10^{-5}	198.4
0.000191	0.0138	3.074×10^{-5}	156.8	3.951×10^{-5}	201.6
0		7.850×10^{-7}	Extrapolated: $\Lambda_o = 161.6$	9.968×10^{-7}	Extrapolated: $\Lambda_o = 207.8$

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

36
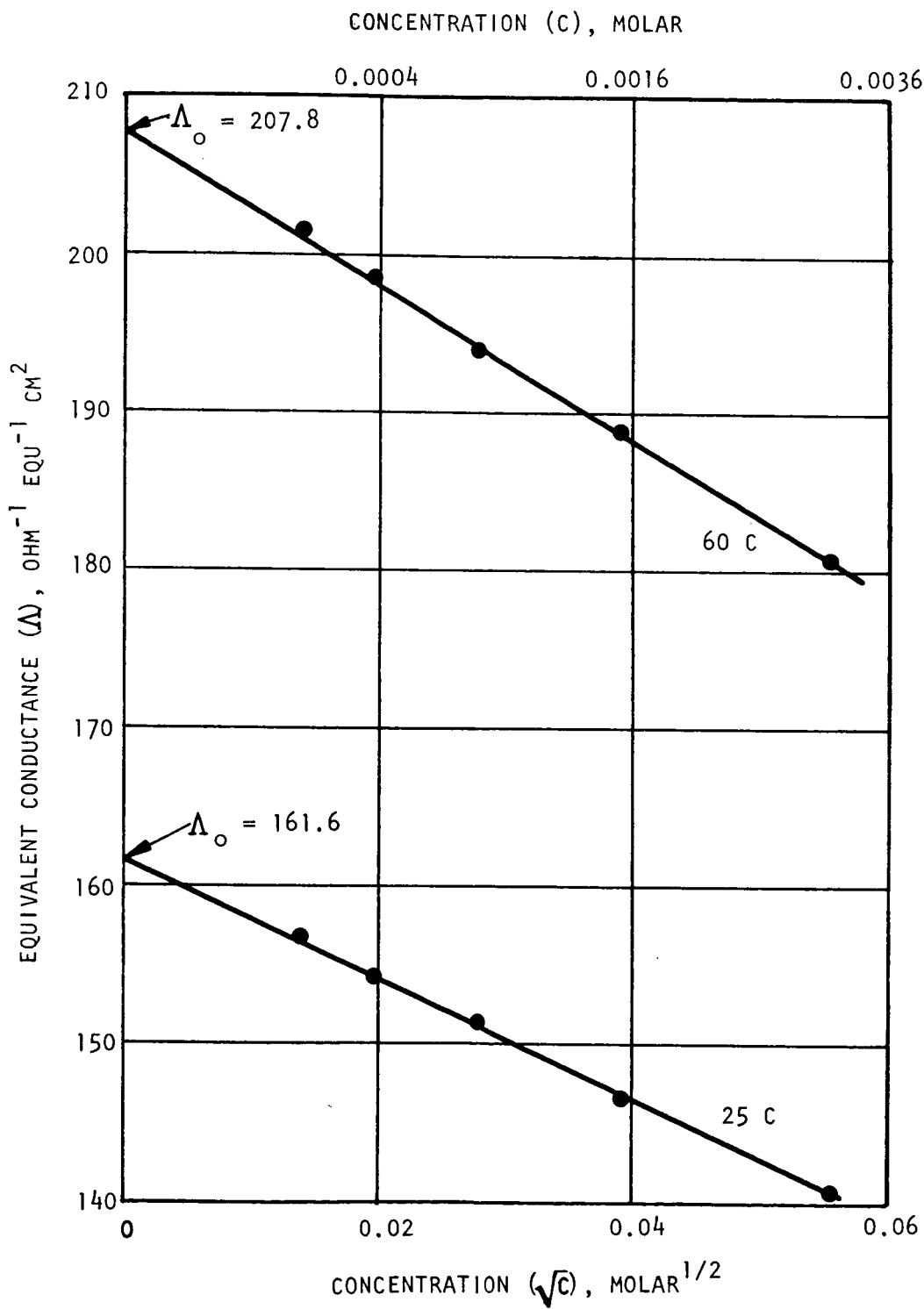
Figure 19.   Equivalent Conductance of TBA·Br in AN
at 25 and 60 C

TABLE 9

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ)

OF LiBr #2/AN #4-1

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
0.003846	0.0620	3.444×10^{-4}	89.4	4.260×10^{-4}	110.5
0.001923	0.0439	1.937×10^{-4}	100.4	2.428×10^{-4}	125.7
0.000961	0.0310	1.051×10^{-4}	108.6	1.330×10^{-4}	137.4
0.000481	0.0219	5.547×10^{-5}	113.8	7.065×10^{-5}	144.9
0.000240	0.0155	2.870×10^{-5}	116.5	3.598×10^{-5}	146.0
0		7.422×10^{-7}		9.471×10^{-7}	
			Extrapolated: $\Lambda_o = 127.2$		Extrapolated: $\Lambda_o = 163.8$

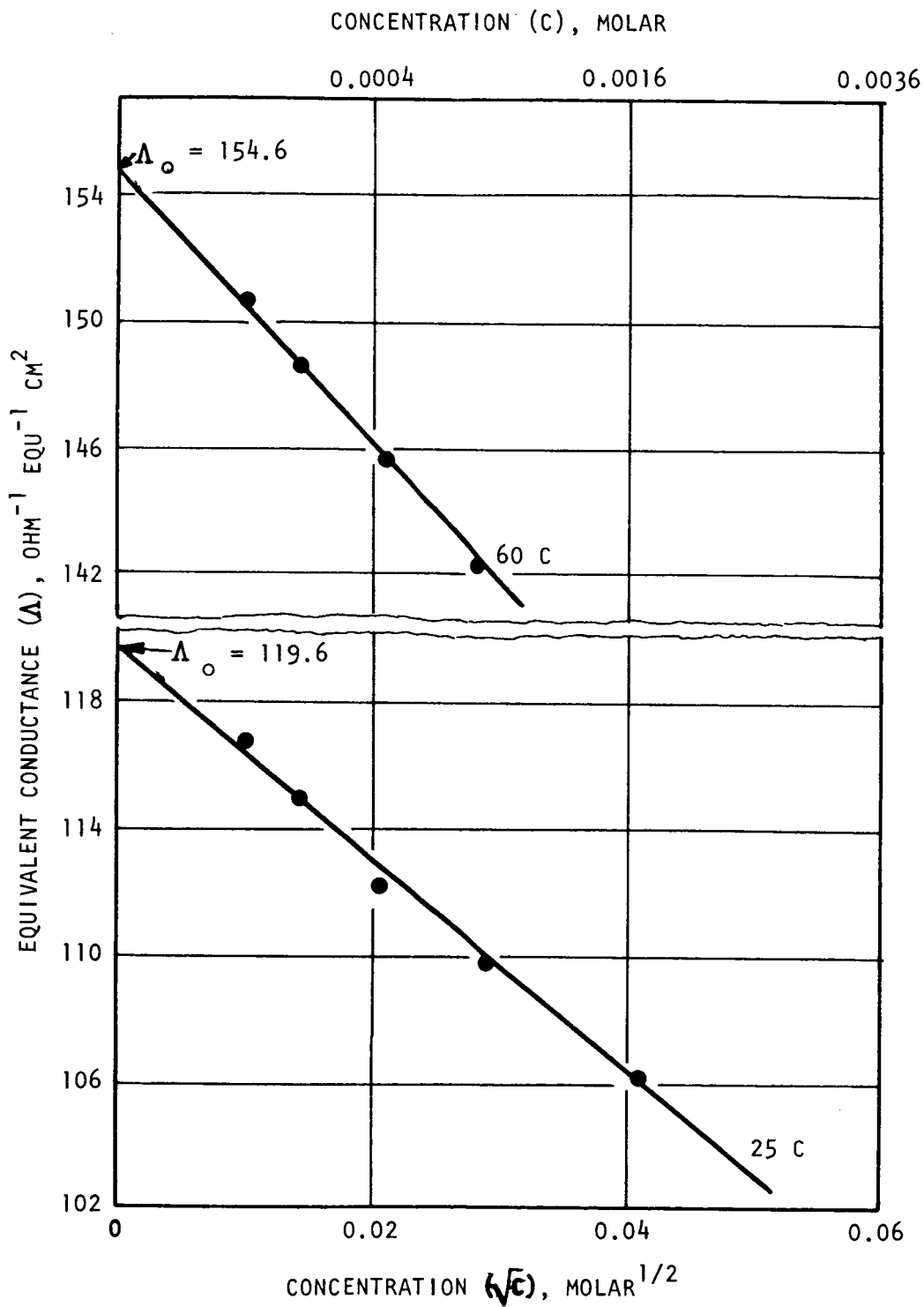Λ*: Equivalent conductance, corrected for conductance of pure solvent
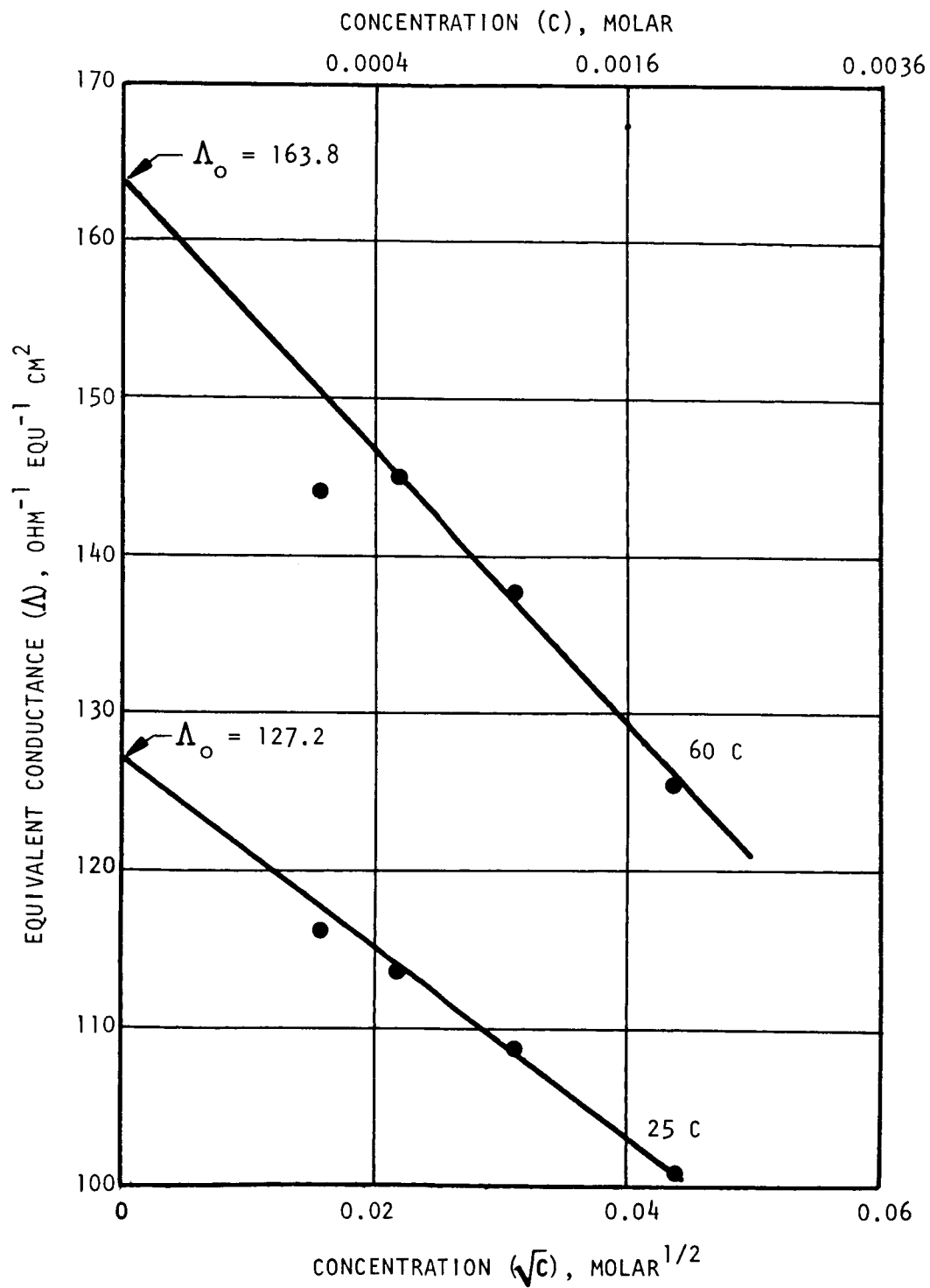
Figure 20. Equivalent Conductance of LiBr
in AN at 25 and 60 C

TABLE 10

SPECIFIC CONDUCTANCE (λ) AND EQUIVALENT CONDUCTANCE (Λ)

OF LiClO$_4$ #2/DMF #5-2

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
0.00668	0.0817	4.474 x 10^{-4}	66.9	6.186 x 10^{-4}	92.4
0.00334	0.0578	2.338 x 10^{-4}	69.8	3.237 x 10^{-4}	96.6
0.001670	0.0417	1.208 x 10^{-4}	71.9	1.678 x 10^{-4}	99.8
0.000835	0.0289	6.272 x 10^{-5}	74.1	8.723 x 10^{-5}	103.1
0.0004175	0.0204	3.292 x 10^{-5}	76.9	4.538 x 10^{-5}	106.0
0.0002087	0.0144	1.698 x 10^{-5}	77.4	2.358 x 10^{-5}	107.3
0.0001044	0.0102	8.867 x 10^{-6}	77.1	1.235 x 10^{-5}	107.6
0		8.233 x 10^{-7}		1.158 x 10^{-6}	
			Extrapolated: Λ_o = 80.4		Extrapolated: Λ_o = 111.1

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

CONCENTRATION (C), MOLAR

0.0004   0.0016   0.0036

$\Lambda_o = 111.1$

110

60 C

100

EQUIVALENT CONDUCTANCE (Λ), OHM^{-1} EQU^{-1} CM^2

90

80

$\Lambda_o = 80.4$

25 C

70

60

0   0.02   0.04   0.06

CONCENTRATION ($\sqrt{C}$), $MOLAR^{1/2}$

Figure 21.   Equivalent Conductance of $LiClO_4$ in DMF at 25 and 60 C

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TABLE 11

EQUIVALENT CONDUCTANCE AT INFINITE DILUTION (Λ_0) FOR SEVERAL SOLUTES AND INDIVIDUAL IONS IN PROPYLENE CARBONATE

Solute	25 C		60 C	
	Λ_0, ohm^{-1}equ^{-1}cm^2	Λ_0^+ or Λ_0^-, ohm^{-1}equ^{-1}cm^2	Λ_0, ohm^{-1}equ^{-1}cm^2	Λ_0^+ or Λ_0^-, ohm^{-1}equ^{-1}cm^2
TBA·TPB	17.2 17.144 ±0.012 (Ref. 7)		30.0	
TBA$^+$		8.6 8.57 (Ref. 7)		15.0
TPB$^-$		8.6 8.57 (Ref. 7)		15.0
TBA·Br	27.5		47.3	
Br$^-$		18.9		32.3
LiBr	26.2		44.8	
Li$^+$		7.3		12.5
LiClO$_4$	25.6		43.1	
ClO$_4^-$		18.3		30.6
LiCl	26.2 (Ref. 3)		44.9 (Ref. 3)	
Cl$^-$		18.9		32.4
TMA·PF$_6$	33.9 (Ref. 3) 36 at 25.5 ±0.5 C (Ref. 10)		57.6	

TABLE 12

EQUIVALENT CONDUCTANCE AT INFINITE DILUTION (Λ_0) FOR SEVERAL SOLUTES AND INDIVIDUAL IONS IN DIMETHYL FORMAMIDE

	25 C		60 C	
Solute	Λ_0, ohm^{-1}equ^{-1}cm^2	Λ_0^+ or Λ_0^-, ohm^{-1}equ^{-1}cm^2	Λ_0, ohm^{-1}equ^{-1}cm^2	Λ_0^+ or Λ_0^-, ohm^{-1}equ^{-1}cm^2
TBA·TPB	51.0		72.6	
TBA$^+$		25.5 26.2 (Ref. 8)		36.3
TPB$^-$		25.5		36.3
TBA·Br	87.0		122.4	
Br$^-$		61.5		86.1
LiBr	79.1		111.0	
Li$^+$		17.6 25.0 (Ref. 8)		24.9
LiClO$_4$	80.4 77.42 (Ref. 8)		111.1	
ClO$_4^-$		62.8		86.7
LiCl	84.0 (Ref. 2) 80.15 (Ref. 8)		118.0 (Ref. 2)	
Cl$^-$		66.4 55.1 (Ref. 8)		93.0
TMA·PF$_6$	90.5 (Ref. 3)		126.0 (Ref. 3)	

TABLE 13

EQUIVALENT CONDUCTANCE AT INFINITE DILUTION (Λ_o) FOR SEVERAL
SOLUTES AND INDIVIDUAL IONS IN ACETONITRILE

	25 C		60 C	
Solute	Λ_o, ohm^{-1}equ^{-1}cm^2	Λ_o^+ or Λ_o^-, ohm^{-1}equ^{-1}cm^2	Λ_o, ohm^{-1}equ^{-1}cm^2	Λ_o^+ or Λ_o^- ohm^{-1}equ^{-1}cm^2
TBA·TPB	119.6 119.60 (Ref. 11)		154.6	
TBA$^+$		59.8		77.3
TPB$^-$		59.8		77.3
TBA·Br	161.6		207.8	
Br$^-$		101.8		130.5
LiBr	127.2		163.8	
Li$^+$		25.4 77.25 (Ref. 12)		33.3
LiClO$_4$	172 183.25 (Ref. 12)		220	
ClO$_4^-$		147 106 (Ref. 13)		187
TMA·PF$_6$	402 (Ref. 3)		503 (Ref. 3)	

are also listed. Values for the tetramethylammonium ion and the hexafluoro-phosphate ion cannot be given yet, because an appropriate link, e.g., data on $LiPF_6$, $TMA \cdot ClO_4$, or $TMA \cdot Br$, is missing.

The most significant result is the low mobility of the lithium ion in all three solvents. From the preceding results, transference numbers for the Li^+ ion in $LiClO_4$ solutions can be calculated, $t_+ = 0.28$ and 0.29 in propylene carbonate at 25 and 60 C, respectively; $t_+ = 0.22$ in dimethyl formamide at 25 and 60 C; and $t_+ = 0.15$ in acetonitrile at 25 and 60 C. The values obtained for 1 molar solutions by Hittorf experiments at room temperature are $t_+ = 0.19$ in propylene carbonate (Ref. 4); $t_+ = 0.25$ in dimethyl formamide (Ref. 4); and $t_+ = 0.32$ in acetonitrile (presented later in this report). If it is considered that relatively large experimental uncertainties are involved in the Hittorf experiments, at least the values for propylene carbonate and dimethyl formamide are in good agreement with the ones cal-culated at infinite dilution. The deviation of the two values is somewhat larger in the case of acentonitrile. A higher ionic conductance has been given in the literature $[\Lambda_0^+ = 77.25 \; ohm^{-1}equ^{-1}cm^2$ (Ref. 12)$]$. This value is based on the ionic conductance of the perchlorate ion, $\Lambda_0^- = 106$, as given in an early paper by Lange (Ref. 13). Lange does not include a value for the lithium ion in his list; his value for the potassium ion in aceto-nitrile is $\Lambda_0^+ = 77$, and the corresponding value for lithium may very well be lower. A possible cause for the discrepancies may consist in a signifi-cant difference of the water content of the solvents used for the studies.

The Walden product $(\Lambda_0 \cdot \eta)$ was calculated for some salts and ions; the values are listed in Table 14. The Walden product is approximately identical for $TBA \cdot TPB$ in all three solvents. This is consistent with the assumption that the ions of this salt are unsolvated. The bromide ion and the lithium ion appear to be solvated to a greater extent in acetonitrile than in the other solvents, whereas the perchlorate ion does not show significant differences.

A more detailed evaluation will be given when the data are more complete.

TABLE 14

WALDEN PRODUCT $(\Lambda_o \cdot \eta)$ FOR SEVERAL SOLUTES AND IONS IN PC, DMF, AND AN

Solute	Solvent	T, C	η, millipoise	Λ_o, ohm^{-1}equ^{-1}cm^2	$\Lambda_o \cdot \eta$
TBA·TPB	PC	25	24.8	17.2	427
		60	13.32	30.0	400
	DMF	25	7.93	51.0	404
		60	5.35	72.6	388
	AN	25	3.36	119.6	402
		60	2.63	154.6	407
TBA·Br	PC	25	24.8	27.5	682
		60	13.32	47.3	630
	DMF	25	7.93	87.0	690
		60	5.35	122.4	655
	AN	25	3.36	161.6	543
		60	2.63	207.8	547
Br$^-$	PC	25	24.8	18.9	469
		60	13.32	32.3	430
	DMF	25	7.93	61.5	488
		60	5.35	86.1	461
	AN	25	3.36	101.8	342
		60	2.63	130.5	343
Li$^+$	PC	25	24.8	7.3	181
		60	13.32	12.5	167
	DMF	25	7.93	17.6	140
		60	5.35	24.9	133
	AN	25	3.36	25.4	85
		60	2.63	33.3	88
ClO$_4^-$	PC	25	24.8	18.3	454
		60	13.32	30.6	408
	DMF	25	7.93	62.8	498
		60	5.35	86.7	464
	AN	25	3.36	147.0	494
		60	2.63	187.0	492

Transference Experiments

The Hittorf cells and the procedure used in these experiments were described previously (Ref. 3).

The results obtained with a 1 M $LiClO_4$/AN electrolyte are summarized in Table 15. A transference number of $t_+ = 0.38$ was calculated from the loss of lithium in the anolyte. Assuming no deposition of lithium at the cathode, the increase in the concentration of the lithium in the catholyte corresponds to $t_+ = 0.26$. The average value of $t_+ = 0.32$ is selected as the final value.

TABLE 15

HITTORF EXPERIMENTS WITH 1 M $LiClO_4$/AN

Solute	Solvent	Total Charge, coulombs	Loss of Li in Anolyte, moles	t_+ (From Anolyte Data)	Gain of Li in Catholyte, moles	t_+ (From Catholyte Data)
1 M $LiClO_4$ #2	AN #3-1	370	$1.46 \times 10^{-3}*$	0.38	$9.91 \times 10^{-4}**$	0.26

*Determined concentration of anolyte at conclusion of experiment: 0.951 M
**Determined concentration of catholyte at conclusion of experiment: 1.035 M

Two Hittorf experiments were performed with 1 M $AlCl_3$ + 0.7 M LiCl/PC; the results are summarized in Table 16. The interpretation of the results is only qualitative or semiquantitative because more than one Al species probably exists. Aluminum was found to accumulate in the anode compartment during the experiment; lithium migrated away from the anode. The results

TABLE 16

HITTORF EXPERIMENT WITH ELECTROLYTES CONTAINING

LiCl AND AlCl$_3$ IN PC AND AN

Run No.	Solutes	Solvent	Total Charge, coulombs	Change of Lithium Content in Anolyte	Change of Aluminum Content in Anolyte
1	0.7 M LiCl #2 + 1 M AlCl$_3$ #3	PC #2-10	432	-5.8×10^{-4} mole[a] (corresponds to n x 56 coulombs)	$+3.3 \times 10^{-3}$ mole[b] (corresponds to n x 318 coulombs)
2	0.7 M LiCl #2 + 1 M AlCl$_3$ #3	PC #2-10	461	-9.5×10^{-4} mole[c] (corresponds to n x 92 coulombs)	$+2.52 \times 10^{-3}$ mole[d] (corresponds to n x 244 coulombs)
3	0.7 M LiCl #2 + 1 M AlCl$_3$ #3	AN #4-1	461	-1.75×10^{-3} mole[e] (corresponds to n x 175 coulombs)	$+3.04 \times 10^{-3}$ mole[f] (corresponds to n x 294 coulombs)

[a] Determined concentration of lithium at conclusion of experiment: 0.68 M

[b] Determined concentration of aluminum at conclusion of experiment: 1.11 M

[c] Determined concentration of lithium at conclusion of experiment: 0.67 M

[d] Determined concentration of aluminum at conclusion of experiment: 1.08 M

[e] Determined concentration of lithium at conclusion of experiment: 0.64 M

[f] Determined concentration of aluminum at conclusion of experiment: 1.105 M

are consistent with the quantitative formation of $AlCl_4^-$ from Cl^- ions introduced by the LiCl with some positive Al species remaining, resulting in an electrolyte composition of

$$0.7 \text{ M Li}^+ + 0.925 \text{ M AlCl}_4^- + 0.075 \text{ M Al}^{+++}/\text{PC}$$

or

$$0.7 \text{ M Li}^+ + 0.85 \text{ M AlCl}_4^- + 0.15 \text{ M AlCl}_2^+/\text{PC}$$
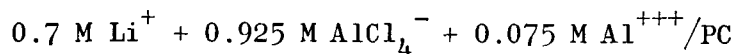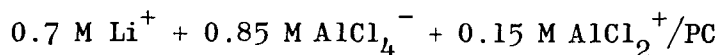
The former is consistent with NMR results previously reported (Ref. 4). Two Al^{27} species are formed in 1 M $AlCl_3/\text{PC}$, and addition of LiCl to the solution converted one of these species to the other. Assuming the species to be $AlCl_4^-$ and complexed Al^{+++}, it would require 1 M LiCl to convert all Al containing species of a 1 M $AlCl_3$ solution to $AlCl_4^-$. Thus in 0.7 M LiCl + 1 M $AlCl_3/\text{PC}$ most of the Al species are expected to be converted to $AlCl_4^-$ but there should be some complexed Al species remaining.

Results similar to these were obtained with a 1 M $AlCl_3$ + 0.7 M LiCl/AN electrolyte; they are also presented in Table 16.

Sonic Velocity Determination

One method of determining the primary solvation number for electrolytes requires a knowledge of compressibility values in the pure solvent and the electrolyte solutions. The compressibilities are obtained by measuring the speed of sound in the liquids of interest and then using the following relationship

$$\beta_s = \frac{1}{\rho c^2}$$

where

β_s = adiabatic compressibility

ρ = density

C = sonic velocity

The apparatus illustrated in Fig. 22 is being used to measure the velocity of sound in various nonaqueous solutions of interest. The electronic equipment consists of a Sperry type UR Reflectoscope, a Tektronix model 535A oscilloscope, and a 5 MHz lithium sulphate ultrasonic transducer. The Reflectoscope generates a 5 MHz pulse-modulated radio-frequency signal which is fed simultaneously to the transducer and the oscilloscope. The sound waves, emanating from the transducer, travel through a known distance of liquid to the bottom of the cell at which point they are reflected back to the transducer. The initial and reflected waves are displayed on the oscilloscope, thus allowing measurement of time required for the ultrasonic waves to traverse the known distance of test fluid.

The distance between the transducer face and the bottom of the cell was determined by calibration of the apparatus at 25 C using distilled water as a standard (Ref. 14). During the calibration runs and subsequent runs, the cell was allowed to reach thermal equilibrium in a mineral oil bath which was thermostated at 25 ±0.01 C. The estimated accuracy of the values determined is 0.2 percent.

Sonic velocity measurements at 25 C were conducted in pure PC and three solutions. The results of these measurements are presented in Table 17. An interpretation of the results will be made after more data have been collected.

Measurements of Diffusion Coefficients

The method described by Wall (Ref. 15 through 17) is being investigated. It involves measurement of the weight change of a suspended disk as a

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TEFLON
O-RING

ULTRASONIC
TRANSDUCER

TEST
FLUID

SONIC VELOCITY CELL

RADIO
FREQUENCY

REFLECTOSCOPE

OSCILLOSCOPE

SYNCHRONIZATION
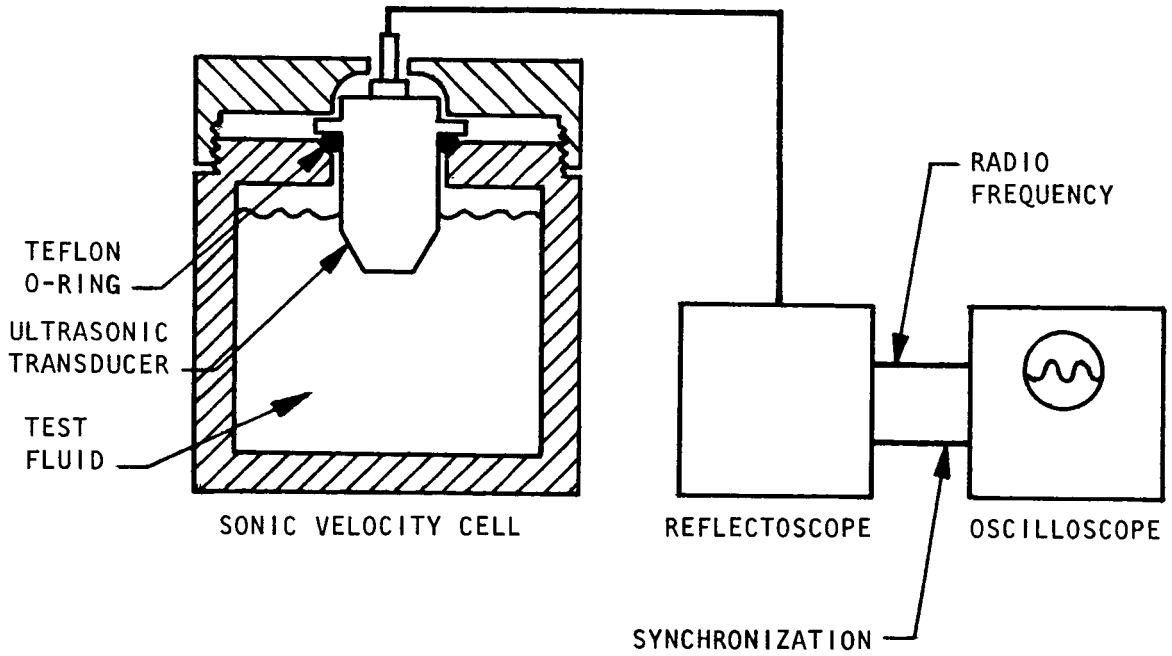
Figure 22.   Determination of Sonic Velocities

function of time. Disks recommended by Wall have been obtained and are being calibrated.

TABLE 17

VELOCITY OF SOUND IN PROPYLENE CARBONATE ELECTROLYTES

Solute	Solvent	Sonic Velocity, m/sec at 25 C
None	PC #2-11	1440
1.0 M LiClO$_4$ #2	PC #2-11	1481
0.125 M TMA·PF$_6$ #1	PC #2-11	1438
0.0227 M TMA·F #2	PC #2-11	1443

An attempt is being made to determine diffusion coefficients of cathodically active species in solution by chronopotentiometry. In a 1 M LiClO$_4$ + 0.0014 M CuCl$_2$/PC electrolyte, a correlation between transition times and the presence of copper species could not be made. It is intended to study next a LiCl/DMF electrolyte containing various amounts of CuCl$_2$.

Measurement of Vapor Pressure

A gas-saturation method was investigated to measure vapor pressures. Nitrogen was bubbled through a wash-bottle containing DMF, and the solvent was condensed in a liquid nitrogen trap. Solvent corresponding to only 30 percent of the vapor pressure of DMF was recovered. An improved saturator is being designed.

Measurement of Dielectric Constants

The microwave method used (at approximately 10 and 25 GHz) is based on measurement of standing wave position and amplitude. This wave results

from interference of a reference wave and the wave traveling through the sample.

Calculations have been carried out to relate the measured quantities to the dielectric constant K ($K = K' - iK''$, where K' is the dispersive part and K'' the absorptive part). Graphs of amplitude vs phase with K' and K'' as parameters have been computed for $70 > K' > 40$, $40 > K'' > 20$ at a frequency of 8.490 GHz.

Additonal computation will be needed to extend the range to lower K' and K'' values.

For the 10 GHz setup, an E-H tee is used to split the wave into reference and sample waves. This apparatus has been partially checked out. Using a ferrite isolator and a phase shifter, the coupling initially noticed between the reference and sample arm has been reduced. The isolator is employed to attenuate to a large extent the wave reflected from the sample cell. Measurements have shown no significant coupling between the two arms. The dielectric constant (K') of water could be obtained to approximately a 3 percent accuracy; the absorptive part, however, appears to be 20 percent low. Work is in progress to decrease the error in K''.

WORK PLANNED FOR NEXT QUARTER

PREPARATION OF ELECTROLYTES

Work on this task will continue. Further solutes such as BF_3, PF_5, TMA·F, and CuF_2 will be characterized.

STRUCTURAL STUDIES

Work on the $LiCl + CuCl_2$ system will continue. This will include continued investigation of the H^1 NMR spectra and an initiation of the investigation of EPR spectra. The Cu^{63} resonance will be also studied if possible. Low temperature H^1 spectra from $LiCl + AlCl_3/AN$ will be investigated. The measurement of the chemical shift of Cl^{35} and Li^7 in electrolytes containing $LiClO_4$ and $LiCl$ will continue.

PHYSICAL PROPERTY DETERMINATIONS

Determinations of physical properties will continue. Individual ion mobilities of some additional ions will be determined. Sonic velocity measurements will be extended to electrolytes based on DMF and AN. Initiated work on measurement of diffusion coefficients, heats of solution, and dielectric constants will be pursued further.

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13. ABSTRACT

Studies of aprotic electrolytes based on three solvents: propylene carbonate, dimethyl formamide, and acetonitrile, were continued. Characterized components were used to prepare the electrolyte solutions.

Structural studies of electrolytes containing lithium perchlorate, lithium chloride, aluminum chloride, and cupric chloride were performed by nuclear magnetic resonance.

The physical properties measured included solvent viscosities, electrolyte conductances to determine individual ion mobilities, sonic velocities, and transference numbers.

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Nonaqueous Electrolytes						
Propylene Carbonate						
Dimethyl Formamide						
Acetonitrile						
Lithium Perchlorate						
Lithium Chloride						
Aluminum Chloride						
Cupric Chloride						