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PROPERTIES OF NONAQUEOUS ELECTROLYTES

FIRST QUARTERLY REPORT

(20 June 1966 to 19 September 1966)

By
Rudolf Keller
James N. Foster
Jack M. Sullivan

Prepared For

National Aeronautics and Space Administration

Contract NAS3-8521

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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

N67 11351

EXPENDITURES UNDER CONTRACT NAS3-8521

The expenditures and commitments during the period from 20 August to 17 September 1966 were:

501 man-hours
$6841

The total expenditures and commitments up to 17 September 1966 were:

1351 man-hours
$18,918

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 Chemistry Section of Rocketdyne's Research Division, during the period 20 June through 19 September 1966.

ABSTRACT

Propylene carbonate, dimethyl formamide, and acetonitrile were purified by distillation and the water content of these solvents was determined by gas chromatography. Solutes were purchased in the highest purity possible.

Conductance measurements and solubility determinations were made on some electrolytes containing lithium salts and aluminum chloride.

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SUMMARY

This program was initiated to generate data and concepts which will provide a better understanding of organic, aprotic electrolyte systems. Measurements of physical properties, such as conductance, solubility, viscosity, vapor pressure, sonic velocity, heat of solution, etc. will be made with pure solutions which have been well-characterized with regard to minor chemical constituents. The structure of solutions will also be studied by spectroscopic techniques.

Main emphasis during this report period was placed on procuring, purifying, and characterizing the electrolyte constituents. Solvents were distilled with a high-plate column and analyzed by vapor phase chromatography. Techniques for the chemical analysis of solids are discussed.

Conductance measurements, including extrapolation to infinite dilution, were made with some electrolytes; also, solubility values were determined.

INTRODUCTION

Investigations currently being carried out in the field of nonaqueous electrochemistry are being seriously hampered by the general lack of knowledge of the properties or characteristics of nonaqueous electrolytes. Solubility, solvation effects, transport properties, and the structure of these electrolytes are not well understood. It is the objective of this program to generate data and concepts which will lead to a better understanding of the organic, aprotic electrolyte systems. Both the primary and secondary high-energy battery development will benefit from the results of this program. Measurements of physical properties, such as conductance, solubility, viscosity, vapor pressure, sonic velocity, heat of solution, etc., will be made with well-characterized solutions. The structure of solutions will be studied by spectroscopic techniques.

The following chemicals will be studied: propylene carbonate (PC), dimethyl formamide (DMF), and acetonitrile (AN) as solvents; lithium perchlorate, lithium chloride, lithium fluoride, tetramethylammonium fluoride, tetramethylammonium hexafluorophosphate, and morpholinium hexafluorophosphate as electrolyte salts; aluminum chloride, boron trifluoride, and water as coordinating agents; and cupric fluoride and cupric chloride as electroactive (cathodic) compounds.

DESCRIPTION OF PROGRESS

PREPARATION OF ELECTROLYTES

Emphasis is being placed on procuring, purifying, and characterizing all electrolyte constituents so that a high degree of reliability can be assigned to the physical property data that will be obtained. It is desirable to use chemicals with less than 100 ppm water, to know what other impurities are present above the somewhat arbitrary level of 100 ppm, and to reduce these contaminations whenever possible. The sources, purification, analyses, and handling of these chemicals will now be described in detail.

Sources for Chemicals

A search for suppliers of high-purity chemicals was made, and the chemicals were ordered as listed in Table 1. Although a purity of 99.99 percent is desirable, many compounds are not available at such high purity and some are available only at a prohibitive cost.

Purification of Solvents

The solvents were purified by distillation from appropriate drying agents using a spinning band distillation column (Stanford Glassblowing Laboratory). This apparatus consisted of a vacuum-jacketed, silvered column equipped with a helical condenser. The column was equipped with an auxiliary nitrogen inlet to ensure an inert atmosphere and a vacuum

TABLE 1

SOURCES FOR CHEMICALS

Chemical	Purity	Supplier
PC	Technical	Eastman
	Reagent	Chemicals Procurement Laboratories
DMF	Spectroquality	J. T. Baker
	Spectroquality	Matheson, Coleman and Bell
AN	Spectroquality	Matheson, Coleman and Bell
$LiClO_4$	99.75 Percent	Foote Mineral Co.
	99.9 Percent	Atomergic Chemetals Co.
LiCl	99.75 Percent	Foote Mineral Co.
	99.9 Percent	Research Inorganic Chemicals
	99.99 Percent	Atomergic Chemetals Co.
LiF	99.8 Percent	Foote Mineral Co.
	99.99 Percent	Research Inorganic Chemicals
$(CH_3)_4NF$	Not stated	Ozark-Mahoning
$(CH_3)_4NPF_6$	Not stated	Ozark-Mahoning
Morph. PF_6	98 Percent	Ozark-Mahoning
$AlCl_3$	99.999 Percent (for Al)	Rocky Mountain Research
BF_3	99.5 Percent	Matheson
CuF_2	99.5 Percent (special quality)	Ozark-Mahoning

TABLE 1

(Concluded)

Chemical	Purity	Supplier
$CuCl_2$	Reagent	Fisher Scientific
	99 Percent	Matheson, Coleman and Bell
LiBr	99.99 Percent	Atomergic Chemetals Co.
$LiBF_4$	95 Percent	Ozark-Mahoning
$(CH_3)_4N\ BF_4$	Melting point 418 C	Ozark-Mahoning

pressure regulator to provide a constant pressure. In general, the solvents were purified by two distillations at reflux ratios of 10/1 and 50/1, respectively.

During this report period, major emphasis was placed on the purification of propylene carbonate (practical grade). For at least 2 days, 500-milliliter batches of propylene carbonate were allowed to stand over $CaSO_4$ (Drierite). The solvent was decanted into a dry distillation flask (dried at 250 C) and distilled at a reflux ratio of 10/1. The middle fraction was collected in a dry receiver and stored over $CaSO_4$. The distillate was again decanted from the $CaSO_4$ and distilled from CaH_2 at a reflux ratio of 50/1. A typical list of column conditions for the final distillation are:

Pressure, mm Hg	11 to 13
Pot Temperature, C	115 to 116
Column Temperature, C	103 to 105
Band Speed, rpm	Approximately 2000
Reflux Ratio	50/1

Initial purification also involved passage of the once-distilled propylene carbonate through a Linde 5A molecular sieve column. However, recent findings indicated that this procedure has the maleficent effect of increasing the number and concentration of impurities (Ref. 1); therefore, it has been discontinued.

A water content of less than 50 ppm was indicated for the first batches analyzed by gas chromatography. A later batch showed a water content of 100 ppm, whereas the undistilled product contained 560 ppm H_2O.

Acetonitrile (spectro grade) was purified by distilling it twice from P_2O_5 at reflux ratios of 15/1 and 50/1, respectively. Typical column conditions for the final distillation are:

Pressure	Atmospheric
Pot Temperature, C	83
Column Temperature, C	80.5 to 80.9
Band Speed, rpm	Approximately 2000
Reflux Ratio	50/1

Water removal may also be facilitated by storage of the solvent over $CaSO_4$ prior to distillation. Future procedures will incorporate this step.

To date, approximately 700 milliliters of N-N, dimethyl formamide (reagent grade) have been purified. The initial procedure involved a single distillation from P, P′ diphenyl methane diisocyanate (Multrathane M) under the following column conditions:

Pressure, mm Hg	48
Pot Temperature, C	84
Column Temperature, C	70 to 71
Band Speed, rpm	Approximately 2000
Reflux Ratio	30/1

Gas chromatographic analysis of the DMF indicated that the water content was decreased by the distillation from 1100 ppm to 350 ppm. A better starting material (spectro grade) will be available in future work.

Conductometric data and gas chromatographic analysis (see below) indicate that further purification steps are necessary since the water content should be reduced to 100 ppm or less if possible. Future procedures will involve storage of the solvent over $CaSO_4$, followed by distillations from CaH_2 and Multrathane M at reflux ratios of 10/1 and 50/1, respectively.

Analysis of Solvents by Vapor-Phase Chromatography

Analysis of the solvents has been divided into a first phase involving the determination of water, and a second phase involving the determination of organic impurities. Because the amount of water present in the solvents is most likely to influence the electrochemical properties of the solvents, the water concentration was determined first.

Separation of water from dimethyl formamide and propylene carbonate is relatively simple because of the large differences in the boiling points. During distillation of propylene carbonate in the initial phase of the program, the concentration of water was monitored using a column packed with 10-percent Quadrol on Teflon-6. This column was used because it had been employed on a previous project and was known to give good peaks for small quantities of water. Although the Quadrol column can be used to monitor volatile impurities in propylene carbonate, it is not suitable for the complete characterization of propylene carbonate because of the bleeding of the liquid phase at temperatures greater than 150 C. It is very unlikely that sharp peaks can be obtained for propylene carbonate on any column with a temperature less than 200 C.

Furthermore, an attempt was made to use the Quadrol column to monitor the efficiency of the distillation of acetonitrile, but the column could not separate acetonitrile and water.

A review of the literature showed that propylene carbonate had been analyzed on a column packed with 10-percent Carbowax K1540 on Teflon-6 (Ref. 2 and 3). This column gave sharp peaks for water and for propylene carbonate. A column was prepared with 10-percent Carbowax 20M on Teflon-6 following the directions of Kirkland (Ref. 4). This column can be used for the characterization of organic impurities in propylene carbonate in the second phase; however, the column does not separate acetonitrile and water, and, consequently, cannot be used for determination of water in acetonitrile.

Porapak Q, a material similar to that described by Hollis (Ref. 5) and Hollis and Hayes (Ref. 6), will separate water from most organic compounds. Lower molecular weight compounds, such as propane, are eluted prior to water, whereas heavier compounds, such as acetone, are eluted subsequent to water. The water peak is sharp and well defined, and since water is eluted prior to most organic compounds, the determination of trace amounts of water in organic solvents is made easier. On the other hand, if water were eluted after the solvent, then water would probably appear as a broad peak superimposed on the tail of the solvent peak.

The water content of several samples of dimethyl formamide and propylene carbonate was determined using the Porapak Q column. Aliquots of the samples were injected into an Aerograph 660 gas chromatograph equipped with a cross-section detector. The experimental conditions are given in Table 2 .

TABLE 2

EXPERIMENTAL CONDITIONS FOR DETERMINATION OF WATER

Column	6-foot length, 3/16-inch-diameter, stainless-steel tube packed with Porapak Q
Hydrogen Flow, cc/min	25
Injector Temperature, C	165
Detector Temperature, C	175
Column Temperature, C	165
Sample Size, microliters	100

Figure 1 shows the beginning of a chromatogram of a sample of propylene carbonate which was obtained under these conditions. The first peak represents the elution of air; both the second and third peaks represent water. That this is so was shown by adding a small amount of water to the sample, whereupon the size of both peaks was increased. Although the areas under the peaks varied between aliquots, the total area was constant. The appearance of two peaks for water may have been due to the relatively large sample size. The appreciable quantity of propylene carbonate used may have coated the Porapak Q and altered the characteristics of Porapak Q temporarily. If this was the case, then part of the water in the sample may have reached the column before the propylene carbonate and become eluted in the normal manner. Then the remaining water in the sample would have reached the column after propylene carbonate and become eluted from a column which is coated with propylene carbonate. This will be verified by injecting smaller aliquots of propylene carbonate (approximately 1 microliter). If the hypothesis is correct, then only one peak will be obtained. If it is incorrect, the second peak could have resulted from decomposition or hydrolysis of the solvent.

Some samples of propylene carbonate gave peaks which were eluted after the water peak. Undoubtedly, these peaks represent organic impurities and they will be characterized in the second phase. Propylene carbonate was eluted after approximately 2 hours at 165 C; therefore, several samples could be injected for the determination of water prior to elution of propylene carbonate. However, the water peaks broaden and their retention times increase with injection of each successive sample. Nevertheless, three or four samples can be injected without broadening the peaks excessively. After a series of samples had been analyzed, the column was heated to 250 C for 40 minutes and cooled to 165 C before analyzing the next set of samples.

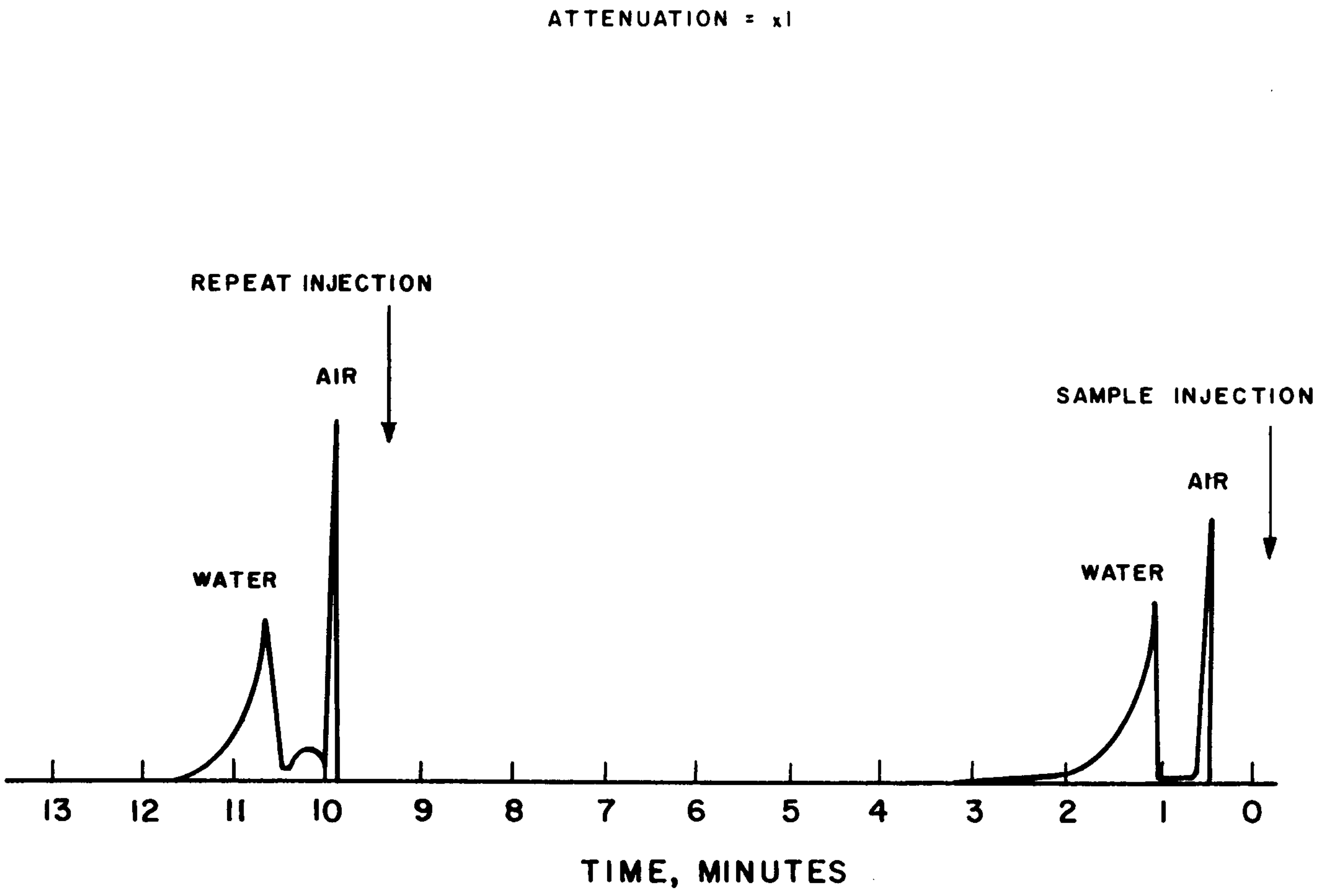

Figure 1. Vapor-Phase Chromatogram of Purified Propylene Carbonate (100-ppm Water); Initial Portion

The response of water in propylene carbonate was determined using two standard additions of water to propylene carbonate. A 0.1-microliter aliquot of water was added to 1.8474 grams of distilled propylene carbonate; this is equivalent to adding 54 ppm water to the sample. A 1-microliter aliquot of water was subsequently added to 1.063 grams of the above solution, giving a new solution of 1000 ppm of water. The two solutions gave response factors of 0.017 and 0.020 cm^2/ppm, respectively. Because a smaller relative uncertainty may be associated with the larger concentration of water, the factor of 0.020 cm^2/ppm was used to relate the area of the water peaks and the water concentration. An additional calibration was made by determining the amount of water in' acetone by a similar procedure. This calibration also gave a factor of 0.020 cm^2/ppm when 1000 ppm water had been added.

The amount of water in dimethyl formamide was determined under the same conditions as those used with propylene carbonate. The response factor of 0.020 cm^2/ppm was used, although it was not explicitly determined in DMF. The initial portion of the chromatogram of a sample of distilled dimethyl formamide is shown in Fig. 2 . Since dimethyl formamide was eluted after 14 minutes, only two water determinations could be made before the solvent was eluted. Therefore, after two analyses, the column was heated to 250 C for 20 minutes and cooled to 165 C.

The chromatograph peaks for pure water were very sharp, whereas the peaks corresponding to water in the solvents were broader. However, with 100-microliter samples, it was possible to determine water concentrations greater than 30 ppm. Since the water peak was broadened apparently by the large sample size, it is reasonable to expect that

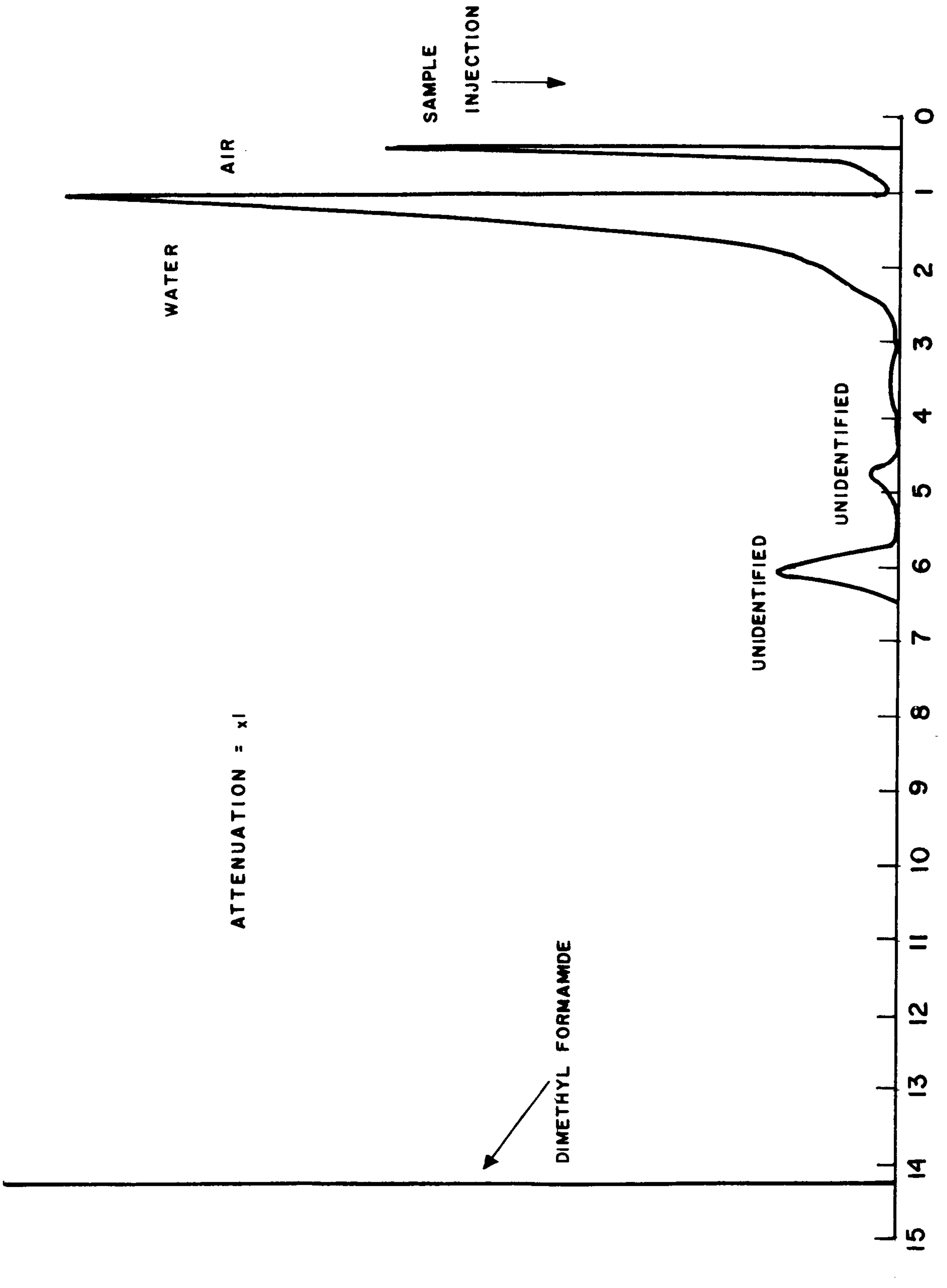

Figure 2. Vapor-Phase Chromatogram of N-, N- Dimethyl Formamide (350-ppm Water); Initial Portion

peaks corresponding to organic material would also be broadened. Furthermore, a 100-microliter sample would have flooded the column to such an extent that compounds eluted at approximately the same time as the solvent would be obscured by the solvent peak. Thus, to obtain sharp peaks and to avoid flooding the column, smaller sample sizes are necessary. The smaller sample sizes necessitate the use of a detector that is more sensitive than the cross-section detector. Consequently, the Aerograph 660 has been equipped with a hydrogen flame-ionization detector and is currently being used to determine the organic impurities in the solvents.

Analysis of Solutes

A comprehensive analysis of the solutes is difficult since a very large number of impurities may be present. Several methods of analysis are being investigated to characterize the solutes. At the present time, it is planned to use spark-source mass spectrometry to perform the major portion of the analysis. The analysis of the metal ions will be duplicated by emission spectroscopy, and the other elements or moieties will be analyzed as necessary by techniques such as spot tests. If spark-source mass spectrometry is found unsuitable, emission spectroscopy will be used to characterize metals, and other tests will be performed to characterize the remaining elements.

Spark-source mass spectrometry is probably the only technique that may be used for the simultaneous determination of practically all inorganic and some organic impurities. In this method, an electric spark is used to volatilize and ionize the sample. The ions produced by the spark are then accelerated by an electric potential and separated by a magnetic field according to their mass-to-charge ratio. The quantity of ions produced in the spark and, therefore, the amount of material in the original sample is determined by the density of exposure on a photographic plate. Thus, the method is both qualitative and quantitative. The method has been used to determine impurities in the part-per-billion range (Ref. 10), and has adequate sensitivity for this program. Under suitable conditions, this method is capable of giving an elemental analysis and the actual chemical moiety. For instance, if a peak is found for chlorine at mass 35, further examination of the spectrogram can indicate whether chlorine is present in the sample as chloride ion or some other species such as chlorate ion.

One of the most important advantages of the method is that basically, no element is overlooked. Some elements, however, may be obscured because of the cracking pattern of the major components. As an example, consider the determination of trace amounts of chloride in lithium perchlorate. In the cracking pattern of perchlorate, Cl^+ ions predominate with increasingly smaller amounts of ClO^+, ClO_2^+, ClO_3^+, and ClO_4^+ ions (Ref. 8). If chloride ions were present in the sample as an impurity, then the chloride ion would produce a peak at mass 35 in addition to the peak already expected from the perchlorate ion in the sample. However, the increment due to the chloride would probably be very small compared to the corresponding amount of chloride ions produced by the cracking of the perchlorate. In this case, an additional analysis for chloride would be performed.

Emission spectroscopy is a common technique that may be employed to determine many metals at levels less than 100 ppm. Table 3 gives some lower limits as obtained by Pacific Spectrochemical Laboratory. Additional tests would have to be devised for determining elements such as mercury, potassium , tantalum, cerium, cesium, dysprosium, and the nonmetallic elements.

TABLE 3

LOWER LIMIT FOR DETECTION OF VARIOUS METALS IN LITHIUM CARBONATE MATRIX

(Obtained from Pacific SpectroChemical Laboratory)

Element	Lower Limit, ppm
Aluminium	2
Antimony	40
Arsenic	300
Barium	100*
Beryllium	0.3
Bismuth	1
Boron	30
Cadmium	30
Calcium	0.05
Chromium	1
Cobalt	3
Columbium	40
Copper	0.3
Gallium	2
Germanium	5
Gold	10
Indium	3
Iron	3
Lead	20*
Magnesium	0.1
Manganese	1
Mercury	200
Molybdenum	5
Nickel	3
Palladium	2
Phosphorus	1000
Platinum	5
Potassium	300
Rubidium	20
Silicon	5
Silver	0.3
Sodium	100
Strontium	3
Tantalum	200
Thallium	100

*Special spectrographic techniques are available which may determine smaller quantities of the metal.

TABLE 3

(Concluded)

Element	Lower Limit, ppm
Thorium	100
Tin	5
Titanium	4
Tungsten	100
Vanadium	5
Zinc	100
Zirconium	5

Additional information on the nonmetallic elements could be obtained by methods such as spot tests. Many of the spot tests described by Feigl (Ref. 9) require less than 2 micrograms of material dissolved in 0.05 milliliter of solution, and in most cases, one-tenth of that concentration is required. To detect an impurity at the 100-ppm level, a solution containing 400 gm/l might be necessary. With the possible exception of lithium fluoride and copper fluoride, it should be possible to prepare a sufficiently concentrated aqueous solution for spot testing of the solutes of interest.

Handling of Chemicals and Glassware

Pure chemicals were handled exclusively in an inert-atmosphere box under a dry nitrogen atmosphere. A dry bag was set up to store bottled chemicals under dry nitrogen to minimize water uptake; this could occur by breathing as the bottles experience temperature fluctuations.

The glassware used for storing solutions was prepared according to the following procedure. After cleaning in hot nitric acid-water (1:1), the glassware was rinsed thoroughly with deionized water, dried in a regular oven, and finally heated to 250 C in a special oven for several hours under a current of dry nitrogen. After the glassware was dried in such a manner, it was immediately transferred to the inert-atmosphere box.

Electrolyte solutions were prepared in the dry box as follows. First, the salts were placed in stoppered volumetric flasks and weighed roughly in the inert-atmosphere box. Then, the flasks were removed and accurately weighed on an analytical balance. Finally, the flasks were returned to the dry box where the solvents were added.

Preparation of $AlCl_3$/PC Solutions

Difficulties in preparing $AlCl_3$/PC solutions have been reported (Ref. 10 to 12), because when aluminum chloride is added to propylene carbonate, considerable heat is produced. The temperature of the solution can rise locally to such an extent that a dark-colored decomposition product forms. To minimize this effect, aluminum chloride was added carefully to propylene carbonate under vigorous stirring; the solution produced was light colored. The addition of $AlCl_3$ to frozen PC led also to only light-colored solutions. Another very promising approach was investigated to eliminate this discoloration. Aluminum chloride was placed in liquid nitrogen, and then propylene carbonate was added under vigorous stirring to form a slurry. The solution which forms upon evaporation of the nitrogen and after thawing was colorless. This approach, however, was not actually applied, because it is not certain how much water would be introduced with the liquid nitrogen.

$AlCl_3$/PC solution showed a tendency to change conductance on standing, particularly at the elevated temperature of 60 C. In one case, a solution containing $AlCl_3$ and LiCl became dark colored after an overnight period at 60 C, although the conductivity did not change very much.

PHYSICAL PROPERTY DETERMINATIONS

Measurements were made of the electrolyte conductivity and solubility in certain solute-solvent combinations. These tests were preliminary since the salts and solvents did not have the desired high degree of purity. Nevertheless, these tests have helped in establishing techniques and in checking out equipment. If differences in the data become apparent later, when chemicals with high purities are obtained, this information will be useful in understanding these electrolytes.

Conductance Measurements

Table 4 summarizes specific conductance values obtained. Conductivity values of solutions were determined in Freas cells with platinized platinum electrodes and cell constants of approximately 0.4 cm^{-1}. Cell constants were determined accurately with standard aqueous KCl solutions. Normally, the conductivity cells were filled with 10 milliliters of solution, but the cell constants were also determined for a 7-milliliter filling and will be used when only small amounts of solution are available. Measurements were made in constant-temperature oil baths at 25 ±0.02 C and at 60 ±0.1 C.

To determine the conductivity of pure solvents and of very dilute solutions, the cell was washed several times with the solvent. It was found that leaving the cell overnight filled with the solvent, was more effective for this purpose than repeated washings for short intervals. The cells were never washed again with water after the initial preparation.

The conductance values listed in Table 4 for the purified solvents represent the lowest values observed so far on this program. Conductance values obtained with different batches occasionally varied by a factor of up to 2. Harris reported a conductance value of 2×10^{-7} $ohm^{-1}cm^{-1}$ for propylene carbonate (Ref. 13). Lockheed chose an upper

TABLE 4

SPECIFIC CONDUCTANCE VALUES AT 25 C

Solute	Solvent	Concentration (C)	Specific Conductance (λ), $ohm^{-1} cm^{-1}$
	PC	Purified solvent	7.6×10^{-8} 1.75×10^{-7} (at 60 C)
	DMF	Purified solvent	1.1×10^{-6}
	AN	Purified solvent	5.1×10^{-7}
LiF	PC	Saturated at 25 C	5.8×10^{-7}
LiCl	PC	Saturated at 25 C	1.70×10^{-4}
$LiClO_4$*	PC	1.11 molar	4.97×10^{-3}
$LiClO_4$*	PC	1.11 to 0.000196 molar	See Fig. 3 and 4
$LiClO_4$	DMF	1.19 molar	2.10×10^{-2}
$LiClO_4$	DMF	1.19 to 0.000076 molar	See Fig. 5 and 6
LiCl	DMF	1.08 molar	8.46×10^{-3}
LiCl	DMF	1.08 to 0.00035 molar	See Fig. 7 and 8
$AlCl_3$	PC	1.015 molar	7.00×10^{-3} 1.33×10^{-2} (at 60 C)
$AlCl_3$	PC	1.015 to 0.000065 molar	See Fig. 9
$LiCl \cdot AlCl_3$	PC	1 molar $AlCl_3$, saturated with LiCl	6.57×10^{-3}

*Results presented in the informal Monthly Progress Report No. 2 contained a computation error in C.

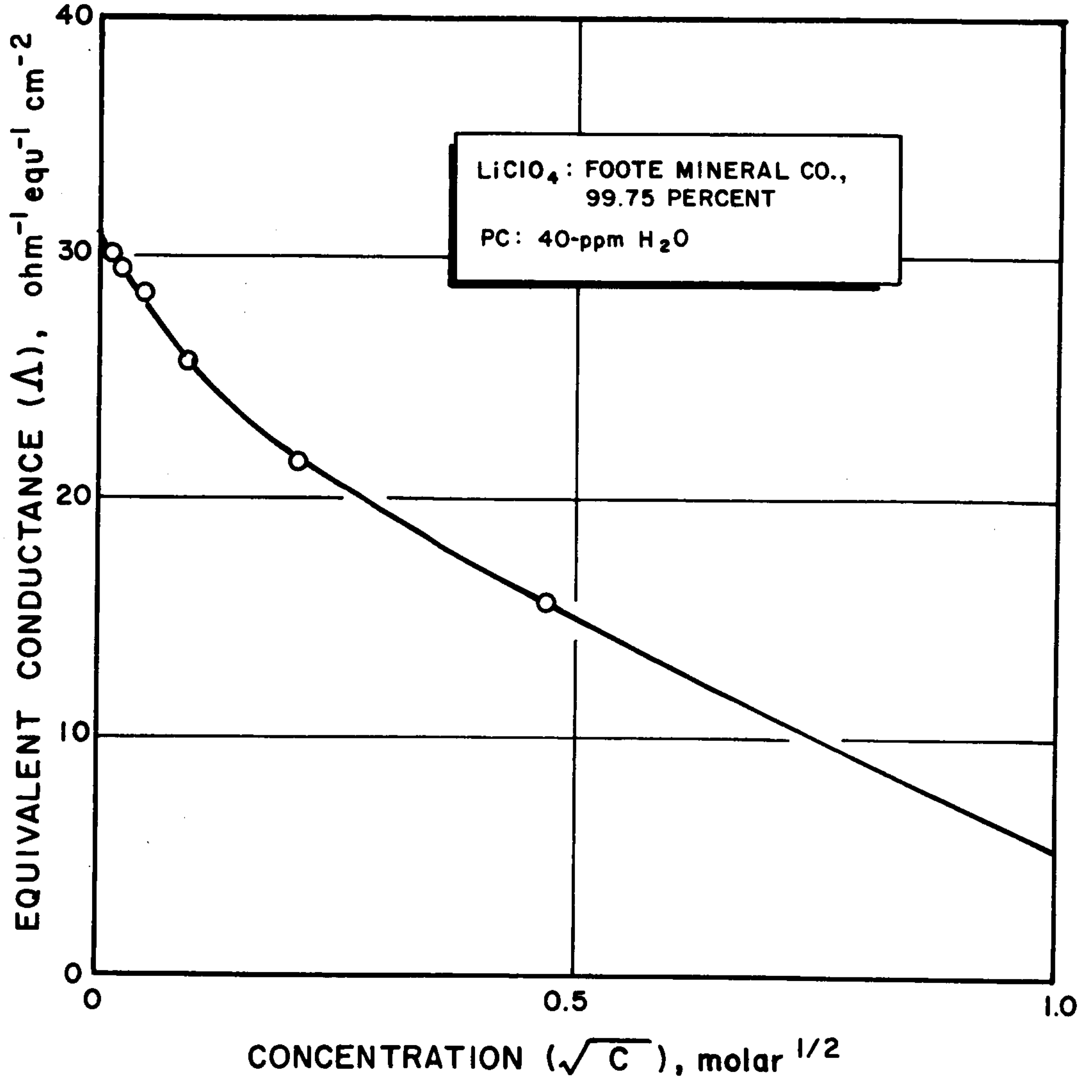

Figure 3. Equivalent Conductance of $LiClO_4$ in PC at 25 C

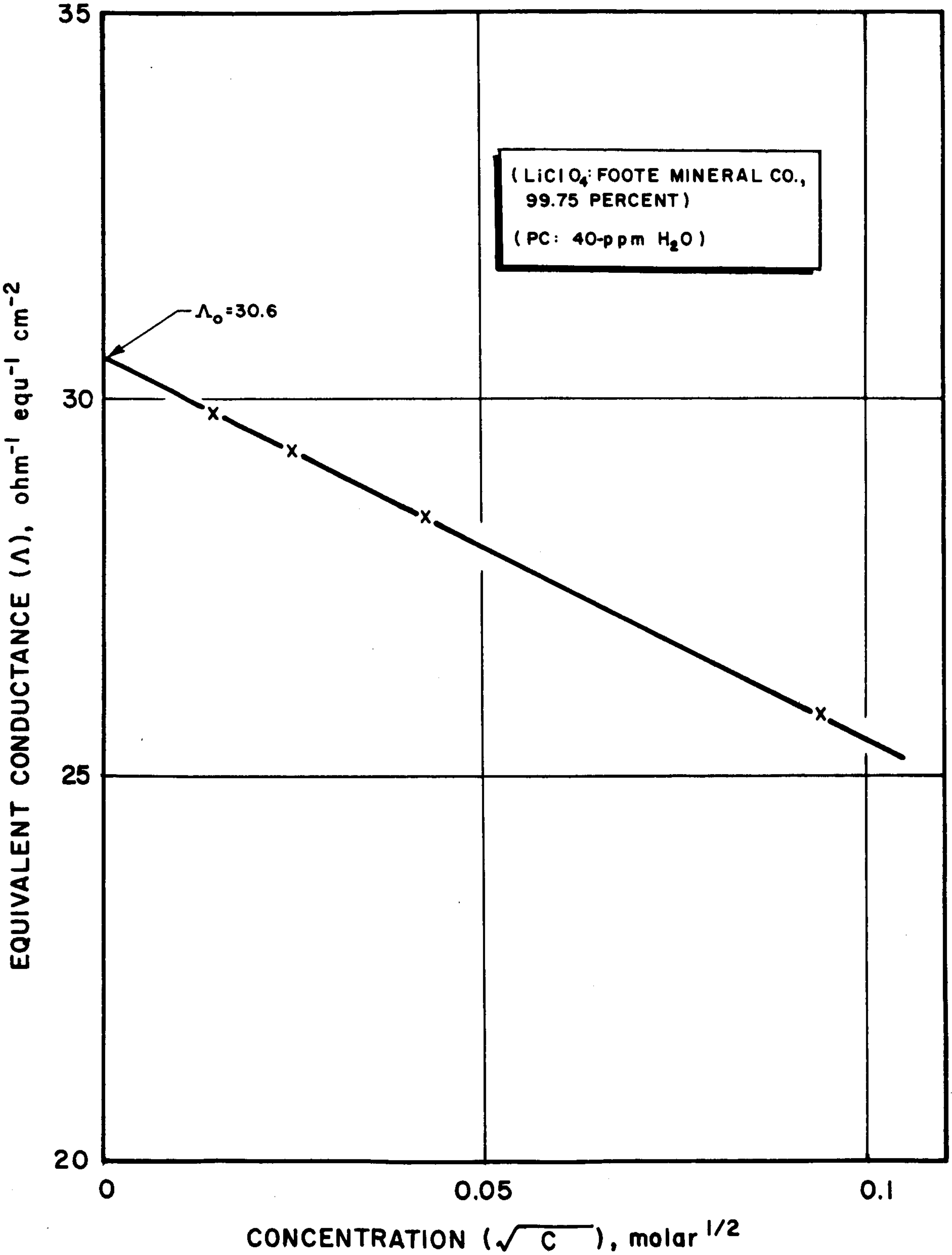

Figure 4. Equivalent Conductance of $LiClO_4$ in PC at 25 C

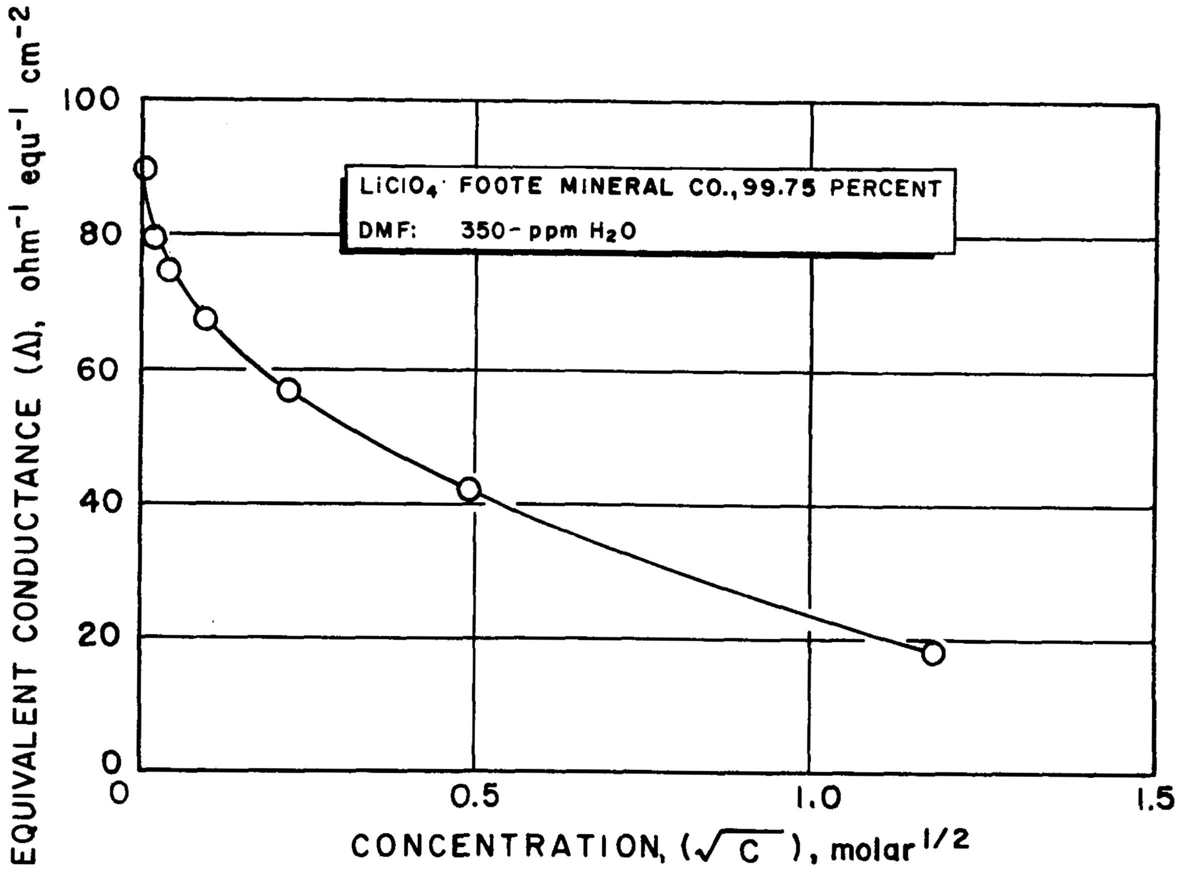

Figure 5. Equivalent Conductance for $LiClO_4$ in DMF at 25 C

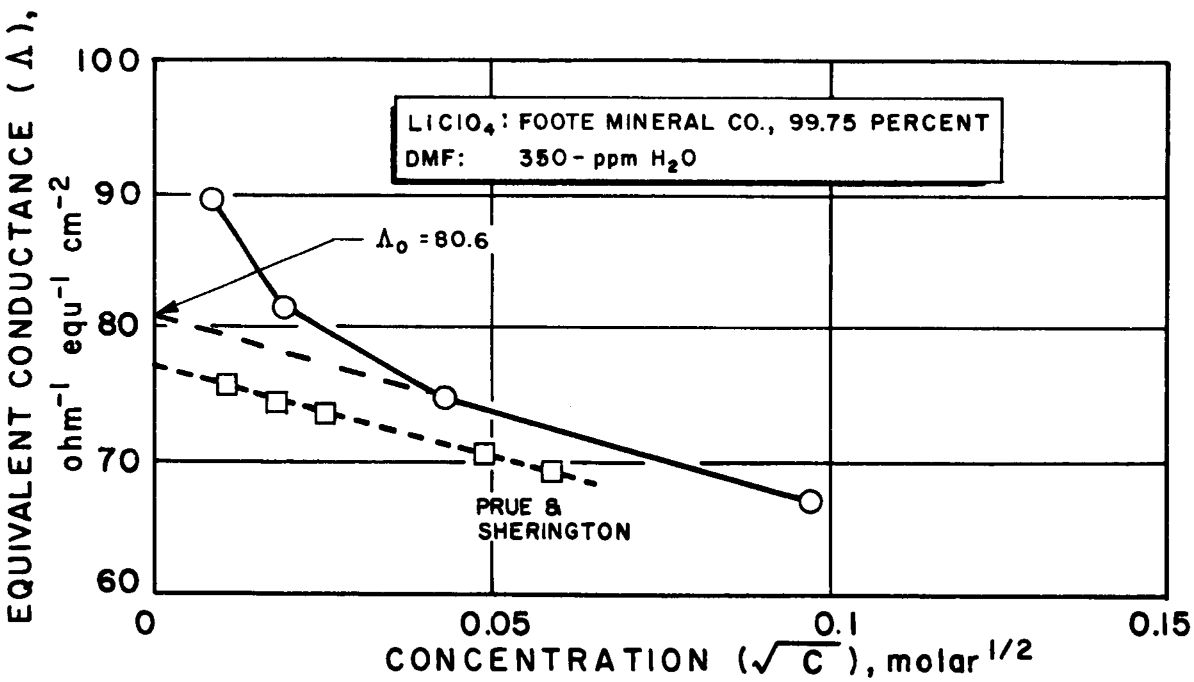

Figure 6. Equivalent Conductance of $LiClO_4$ in DMF at 25 C

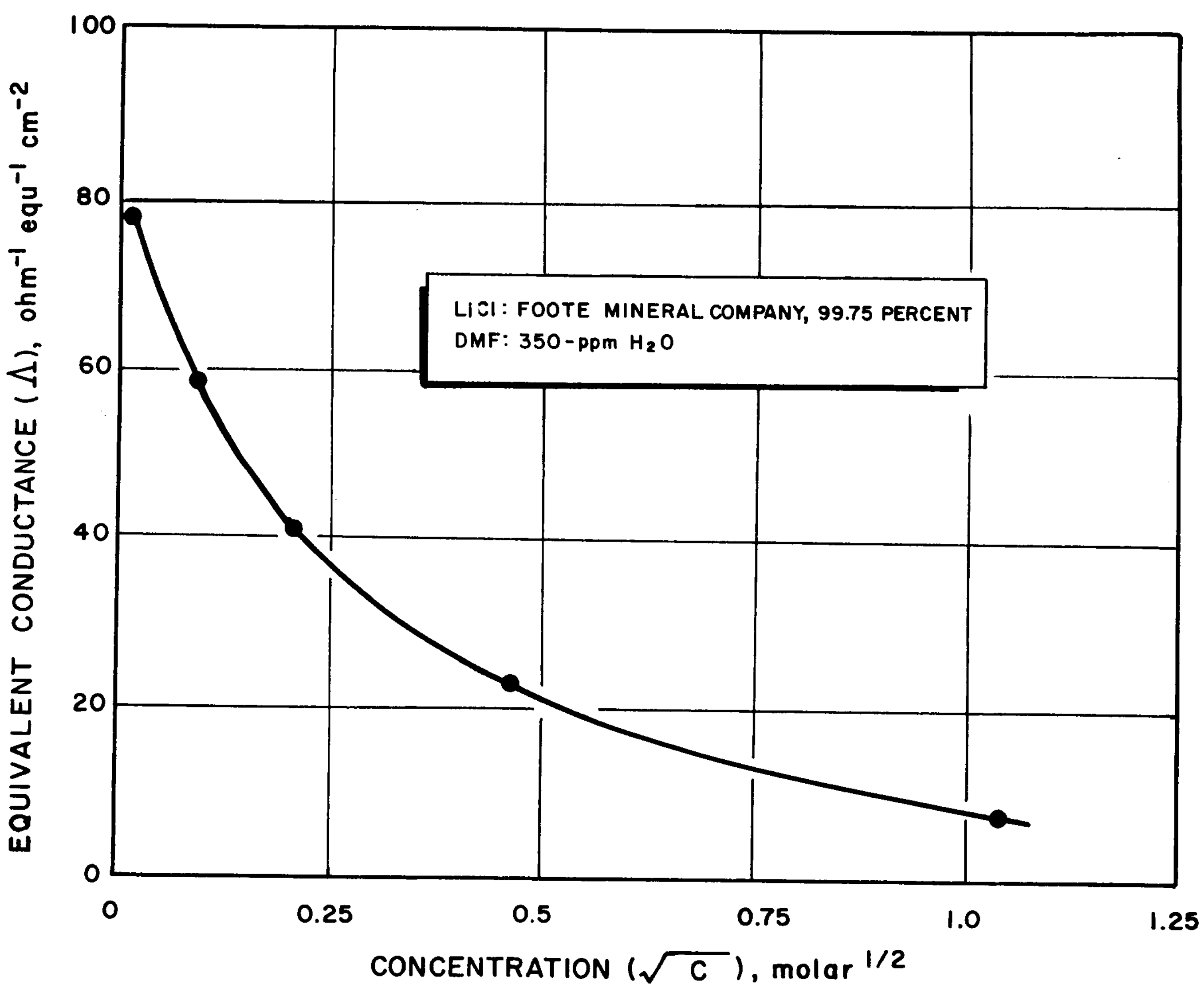

Figure 7. Equivalent Conductance of LiCl in DMF at 25 C

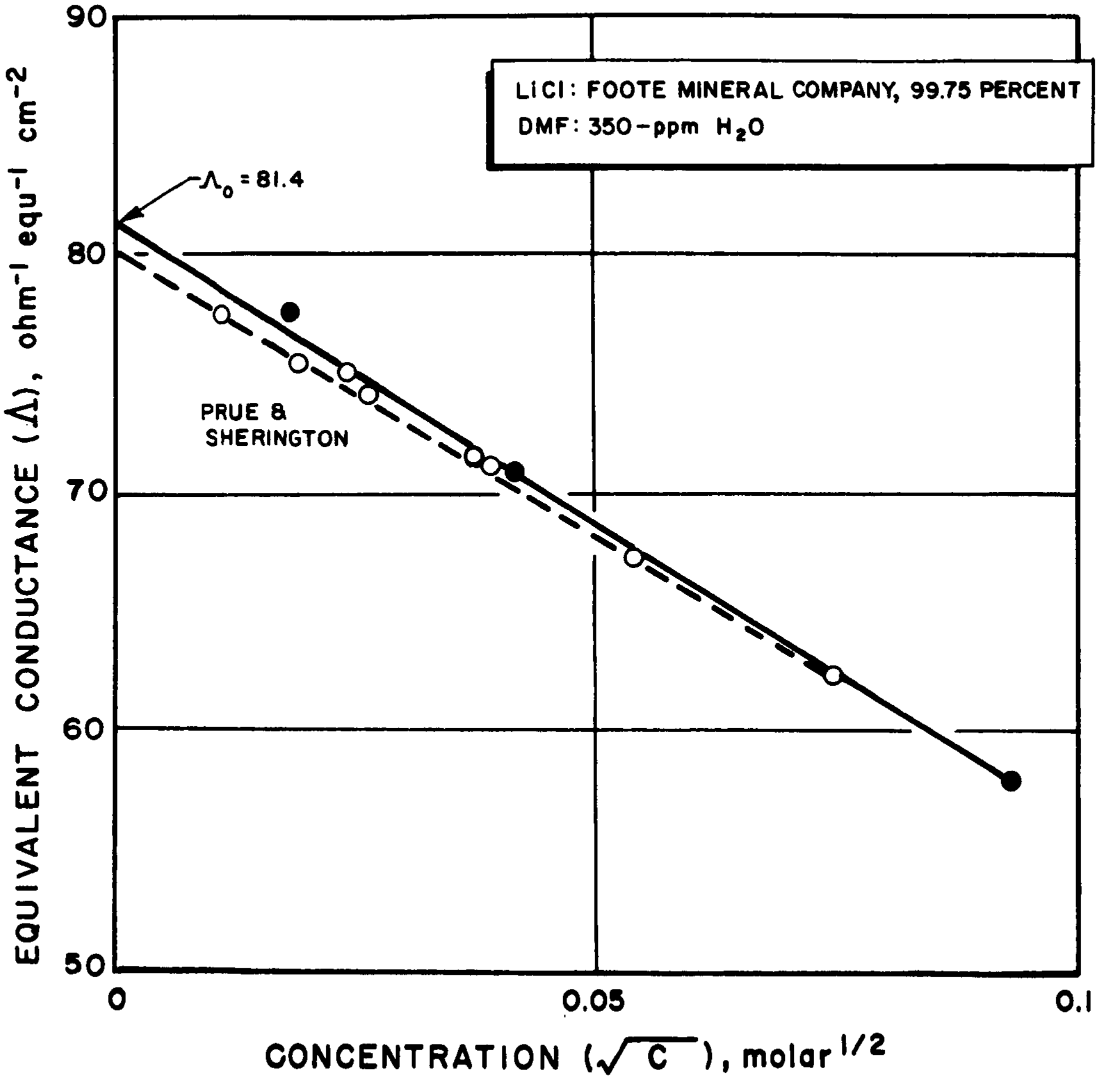

Figure 8. Equivalent Conductance of LiCl in DMF at 25 C

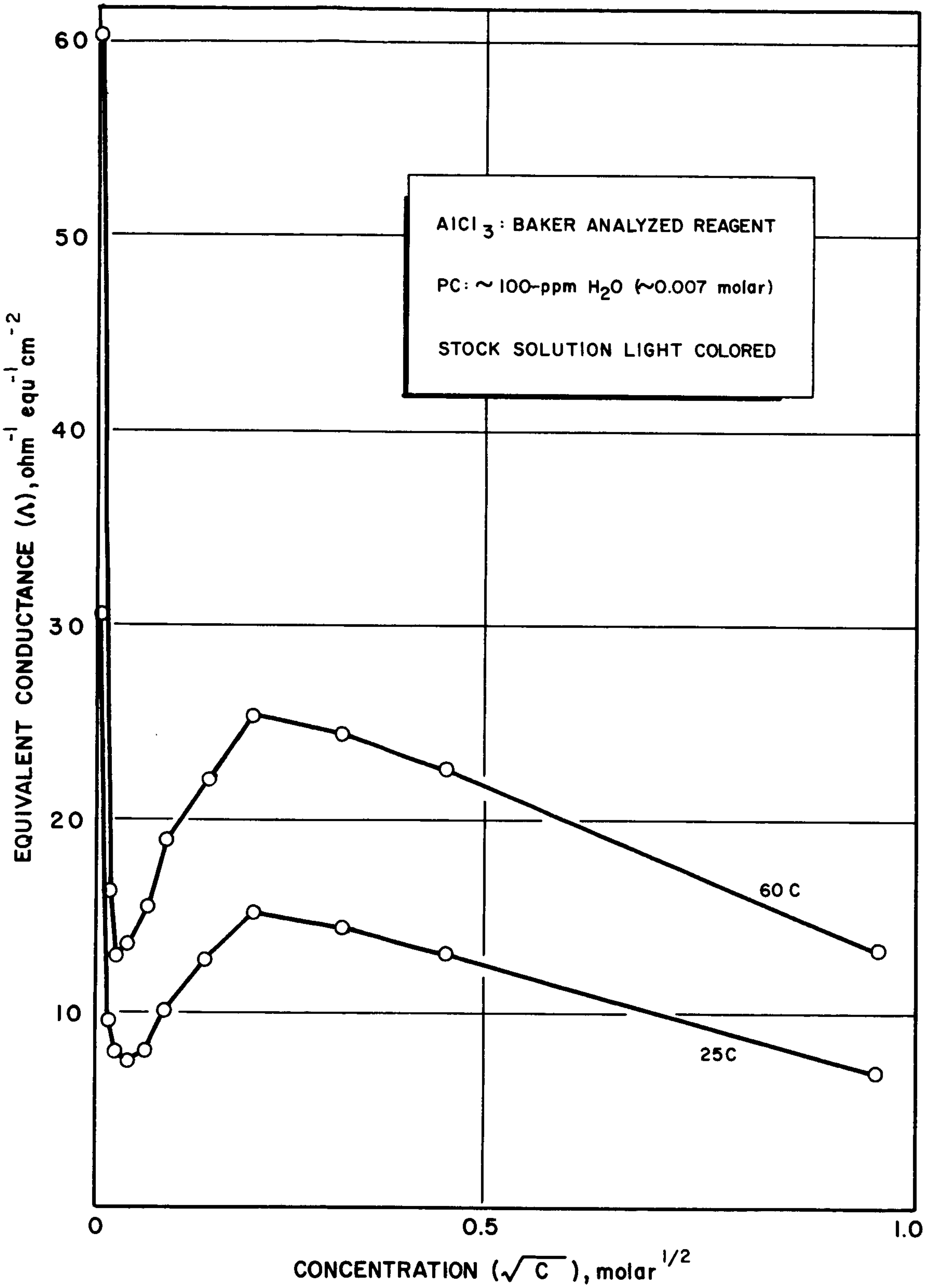

Figure 9. Equivalent Conductance of $AlCl_3$ in PC

limit of 5×10^{-7} $ohm^{-1}cm^{-1}$ for the propylene carbonate used in some of their investigations (Ref. 14). Literature values for dimethyl formamide and acetonitrile are 0.6×10^{-7} $ohm^{-1}cm^{-1}$ (Ref. 15), and 5×10^{-8} $ohm^{-1}cm^{-1}$ (Ref. 16), respectively.

Conductance at Infinite Dilution

A series of solutions having different concentrations were prepared: $LiClO_4$ in PC, $LiClO_4$ in DMF, LiCl in DMF, and $AlCl_3$ in PC. The conductivities were measured as described, and the results are presented in Fig. 3 through 9 . The chemicals used for these tests did not have the high, known degree of purity desirable. Nevertheless, a linear relationship between equivalent conductance and the square root of concentration was obtained in the case of $LiClO_4$/PC, and good agreement with literature data (Ref. 17) was obtained in other cases, particularly for LiCl/DMF.

The equivalent conductance values, Λ , are all corrected for the conductance of the pure solvent. They reflect only that part of the solution conductivity which was contributed by the ionic solute. The correction may introduce relatively high uncertainties at low concentrations, because the conductance of the pure solvent is subject to some variation. This fact is probably solely responsible for the deviation of the curves (Fig. 6 and 8) at low concentrations. It is clear that a highly pure, low-conductive solvent is desirable for such measurements.

A very interesting relationship between equivalent conductance and concentration was obtained for an $AlCl_3$/PC electrolyte (Fig. 9).

The equivalent conductance showed a maximum around a concentration of 0.04 molar, and a minimum at about 0.002 molar. A similar behavior was found by Lockheed (Ref. 8). At very low concentrations, a great increase in the equivalent conductance was observed. This had not been reported previously. The concentration of water was 100 ppm or 0.007 molar, and it is not known to what extent the presence of water determined the conductivity characteristics.

Solubility Determinations

Saturated solutions were prepared in volumetric flasks. The solvents were heated with an excess of salt, shaken well, and cooled down in a constant-temperature bath. Samples of the saturated solution were taken with pipettes. This simple technique seemed adequate since the solubility value had not changed after a LiCl/PC sample had been centrifuged.

The results of the solubility determinations are summarized in Table 5 .

Solubility of LiF in PC. The solubility of LiF in propylene carbonate was found to be very low. A saturated solution showed a conductivity of 5.8×10^{-7} ohm^{-1}cm^{-1} at 25 C compared with a solvent conductivity of 9×10^{-8} ohm^{-1}cm^{-1}. Bauman, Chilton, and Mauri (Ref. 17) estimated the solubility of LiF in propylene carbonate to be 1.5×10^{-4} gm/100 ml (6×10^{-5} molar), based upon a conductivity value of 1.99×10^{-6} ohm^{-1}cm^{-1} and a $\Lambda_0 = 28$ ohm^{-1} equiv.$^{-1}$cm^{2}. According to our conductivity value, the solubility of LiF is 2×10^{-5} molar, i.e., lower than the referenced value.

A direct method to determine the solubility of LiF in PC is preferred. An attempt was made to determine the lithium content of a saturated LiF

TABLE 5

SOLUBILITY DETERMINATIONS SUMMARY

Solute	Solution	Solubility
LiF	PC	2×10^{-5} molar at 25 C (estimated from conductivity)
LiCl	PC	0.056 molar at 25 C
LiCl	1 molar $LiClO_4$/PC	0.038 molar at 25 C 0.037 molar at 60 C
LiCl	1 molar $AlCl_3$/PC	0.78 molar at 25 C 0.80 molar at 60 C

solution by flame photometry. The signal received was at the limit of detectability. A colorimetric method which is being used at Rocketdyne as a routine analysis for determining small amounts of fluorine in water (Ref. 19) was tried, but this method was not directly applicable. The presence of propylene carbonate causes interfering color changes of the zirconium-dye lake. However, the method may be applied if the sample is pretreated. Another alternative is to use atomic absorption*; this method may furnish sufficient sensitivity for analysis of trace amounts of lithium as they are present in a saturated LiF/PC solution.

Solubility of LiCl in PC, in 1 Molar $LiClO_4$/PC, and in 1 Molar $AlCl_3$/PC. The solubility of LiCl in propylene carbonate at 25 C was determined by a potentiometric titration with aqueous $AgNO_3$ solution. It was found to be 0.056 molar, as compared to a literature value of 0.057 molar (Ref. 14). An attempt to determine the solubility by evaporation to dryness resulted in a value which was too low, namely 0.031 molar; it was noticed that the residue was not hygroscopic in contrast to the original solute.

The solubility of LiCl in molar $LiClO_4$/PC solution was also determined by titration. It was 0.038 molar at 25 C and 0.037 molar at 60 C, i.e., the solubility did not change significantly with the temperature rise.

A similar observation was made in a 1 molar $AlCl_3$/PC solution. The solubility for LiCl in this solution was 0.78 molar at 25 C and 0.80 molar at 60 C.

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*An atomic absorption apparatus will be delivered to Rocketdyne soon.

Discussion

Many data of properties of nonaqueous electrolytes have been obtained in the past with solutions which were not sufficiently characterized and often impurity effects may have affected the results significantly. It is, therefore, desirable to eliminate such uncertainties as far as possible.

An example taken from this report may serve to illustrate how impurities may introduce some ambiguity. Figures 5 and 6 show an attempt to determine the equivalent conductance for $LiClO_4$ in DMF. A high conductivity value for the solvent can be caused by a high water level or by other impurities. This introduces uncertainty into conductivities measured at low $LiClO_4$ concentrations so that accurate extrapolation of conductivities to infinite dilution cannot be performed.

In determining absolute ion mobilities by the above method, it is crucial to have a very pure solvent. On the other hand, a moderate contamination of the $LiClO_4$ by some LiBr, for example, may not interfere significantly with this measurement. However, if, for example, solubility of CuF_2 is determined, traces of bromide ions may alter the solubility substantially.

Interesting results were indicated by the preliminary conductivity measurements. For the LiCl-DMF system, good agreement was obtained with literature values of Prue and Sherrington (Ref. 17), despite a fairly high water content determined in our DMF. These authors found no evidence for any preferential solvation of the ions in DMF by water. Aluminum chloride displayed an abnormal behavior; its equivalent conductance showed a minimum with decreasing concentration. A definite conclusion concerning possible species present at low concentrations can not be reached yet, because the water level of the solvent was too high.

Lithium fluoride seems to have a very low solubility in propylene carbonate. This fact is important since LiF is one of the major possible electrode products in high-energy battery systems under consideration.

LITERATURE STUDY

A McBee index card system has been devised to collect and store information on properties of organic electrolytes. Particular emphasis was placed on the solute-solvent combinations considered in this program. A systematic review of the literature was started, emphasizing previous NASA high-energy battery contracts.

WORK PLANNED FOR NEXT QUARTER

PURIFICATION AND ANALYSIS OF CHEMICALS

The distillation of solvents will be perfected to reduce contamination to acceptable levels. The water content of solvents will be determined on a routine basis by vapor-phase chromatography and by Karl Fischer titration. The organic contaminants in distilled solvents will be measured by the former technique.

Based upon the methods of salt analysis presented in the text, contamination levels will be established and recommendations will be made to the contract monitor as to the degree of purification which will be required. Only when salts and solvents have been characterized and purified to an acceptable level will the physical property determinations be carried further.

PHYSICAL PROPERTY DETERMINATIONS

The first solute-solvent combinations to be investigated will be LiCl/DMF and LiCl-$AlCl_3$/PC. These will be studied by nuclear magnetic resonance (NMR), by conductance measurements, and by solubility determinations.

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The expenditures and commitments during the period from 20 August to 17 September 1966 were:

501 man-hours
$6841

The total expenditures and commitments up to 17 September 1966 were:

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UNCLASSIFIED

Security Classification

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(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

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3. REPORT TITLE

PROPERTIES OF NONAQUEOUS ELECTROLYTES

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*

Quarterly Report (20 June through 19 September 1966)

5. AUTHOR(S) *(Last name, first name, initial)*

Keller, R.; Foster, J. N.; Sullivan, J. M.

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13. ABSTRACT

Propylene carbonate, dimethyl formamide, and acetonitrile were purified by distillation and the water content of these solvents was determined by gas chromatography. Solutes were purchased in the highest purity possible. Conductance measurements and solubility determinations were made on some electrolytes containing lithium salts and aluminum chloride.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Nonaqueous Electroyltes						
Propylene Carbonate						
Dimethyl Formamide						
Acetonitrile						
Lithium Salts						
Aluminum Chloride						

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