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LITHIUM SULFURYL CHLORIDE BATTERY

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Conductivities in the range 7×10^{-4} to 11×10^{-4} ohm^{-1} cm^{-1} have been measured for solutions of $LiAlCl_4$ (0.1 to 1.0 mol per liter) in SO_2Cl_2 at 30°C. A four electrode conductivity probe was used to give measurements independent of frequency in the range studied, 500 Hz to 20,000 Hz.

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1. Introduction

The conductance of battery electrolyte should be large to avoid internal resistive losses during current passage. Since the maximum current which may be drawn from a cell is resistance limited, the conductivity of electrolyte becomes an important parameter for optimum cell design. As is well known, the concentration of electrolyte in several types of aqueous batteries is chosen primarily for maximum conductivity. Accordingly, work on this project was directed towards obtaining conductivity data for a practical range of concentrations of $LiAlCl_4$ (0.1-1.0M) dissolved in SO_2Cl_2. In order to obtain data rapidly, a novel procedure was devised which yielded good results with relatively simple equipment. A four-point dipping conductivity probe was developed to supplement the two-point method (described in ECOM75-1676-1) which included a large frequency effect associated with the low dielectric constant of SO_2Cl_2. The details of the conductivity probe design, its calibration and use in measuring conductivity in $LiAlCl_4$ -SO_2Cl_2 solutions is described in the following sections.

2. Conductivity Cell

2.1 Method

In the four-point method, two outer electrodes carry current while two inner electrodes are used to measure a resulting potential difference. A fixed geometry was provided by adding pyrex cross-braces to the arrangement of four electrodes, as shown in Fig. 1. Each electrode consists of a platinum wire sealed through a pyrex glass capillary. For simplicity of operation, the container for electrolytes under study was chosen to be an ordinary laboratory beaker of 250 ml capacity. Various $LiAlCl_4$ concentrations were prepared by directly adding amounts of $LiAlCl_4$ to the SO_2Cl_2 initially contained within the beaker. These operations were conducted in a nitrogen filled glove box (LabconCo). This procedure is the reverse of the method used to prepare a series of known concentrations by successive dilutions. However, for glove box operations, there is a distinct advantage with the increasing concentration method since a limited amount of solvent can be used continuously in place. Of course, a sample is obtained after each addition has been thoroughly dissolved with the aid of a Teflon-coated magnetic stirrer in the beaker. These samples are stored for subsequent analysis.

The additions of $LiAlCl_4$ were made by melting this material from its storage tube directly into the beaker containing the SO_2Cl_2 solvent. A heating tape was used to melt a selected section of the solid $LiAlCl_4$ from its storage tube. No weighing procedure is necessary in this method, thus avoiding the necessity of either cumbersome transfer equipment for weighing outside of the glove box or the need to incorporate a balance within the glove box. Even though the concentration is not determined until after the withdrawn samples have been analyzed, this reverse method is rapid and convenient. Because each sample is taken immediately following a conductivity determination, no special effort is needed to prevent solvent loss by splashing during subsequent $LiAlCl_4$ addition. Since samples are withdrawn from homogeneously mixed electrolyte, only the level of liquid in the beaker is diminished. Typically, the sample volume was one ml, withdrawn from approximately 200 ml of electrolyte.

A possibility for error exists due to solvent evaporation during the conductivity measurement sequence for each composition. However, the increase in $LiAlCl_4$ concentration due to loss of SO_2Cl_2 is minimized by reducing the conductivity measurement time to less than one minute. During this time, several data points were obtained at selected frequencies to verify that the potential between the inner two probes was, in fact, independent of frequency.

2.2 Construction of Four-Point Probe Electrodes

As shown in Fig. 1, the two current carrying electrodes (identified as 3 in Fig. 1) and the two potential probe electrodes (inner electrodes, identified as 2 in Fig. 1) are platinum, sealed in pyrex capillary tubing. Pyrex cross-braces sealed to the capillaries provided structural rigidity, thus allowing the cell constant to be maintained after calibration (identified as 4 in Fig. 1). Platinum rods, as 0.25 cm diam. were used for the outer electodes and 0.037 cm diam. platinum wires were used for the inner electrodes. Mercury was poured into each glass tube, and clean, abraded pure copper wires were immersed into the mercury (labelled 5 in Fig. 1).

2.3 Calibration of Four-Point Probe Electrodes

The four-point probe electrode (Fig. 1) was calibrated and tested with the known conductivity of potassium chloride solutions (0.01 N and 0.1 N). A 250 ml beaker was used for the cell because of the simplicity of operation in the glove box where the four-point assembly, used as a dip cell, could be moved aside to allow fresh addition of $LiAlCl_4$ to the SO_2Cl_2 solvent. With a constant current applied to the outer electrodes, the electrical field around the platinum tips of the outer electrodes was intercepted by the two inner "point" probes. The potential drop, V_2 (see Sec. 2.4), between the two inner probes is due to the resistance of solution and theoretically should be constant for constant current flowing in the cell according to Ohm's law. However, the electrical field is influenced by the proximity of non-conducting boundaries such as the upper surface of electrolyte and the bottom of the pyrex beaker. Thus, the potential drop, V_2, depends upon the distance between the electrode and the bottom of the beaker.

The range of spacing giving rise to a constant potential difference between the two inner "point" probes was determined while a constant current was flowing through the cell for 0.01N potassium chloride solution. To do this, the potential difference, V_2, between two inner probes was measured for different spacings between the four-point array and the bottom of the beaker at constant frequency (1000 Hz). A constant potential region was obtained for spacings in the range from 1.2 to 2.6 cm. The four-point probe electrode array was maintained at the center of the beaker during these measurements, with the beaker filled to a height of 4.3 cm from the bottom.

A second determination was made of the position dependence of the four-point array, using 0.1 N KCl as electrolyte. The range of heights for which the inner probe potential difference, V_2, was constant was from 1.0 to 2.6 cm from the bottom of the 250 ml beaker, in agreement with results obtained with 0.01N KCl. Further details of the calibration procedure are given in the following section.

2.4 Measurement and Calibration Circuit

For the calibration of the four-point electrode array in standard solutions of KCl and for measurements of the conductivity in the $LiAlCl_4$-CO_2Cl_2 system, the outer electrodes (3 in Fig. 1) were in series with a current limiting resistance of 20,000 ohms and a precision resistor (Type 1434-N, General Radio Co.) of 1000 ohms. Voltage was applied from a Hewlett-Packard Signal Generator (model 3310A). Potential differences were determined with the aid of a digital multimeter (Fluke 8000A). Current through the resistance train was determined from the potential difference, V_3, across the 1000 ohm resistance standard and was generally maintained in the range 0.5 to 1.5 x 10^{-4} Amp. For each selected frequency in the range 500 to 20,000 Hz provided by the signal generator, the digital multimeter was used to determine the potential difference between the outer electrodes, V_1, as well as V_2, the potential difference between the inner pair of electrodes.

In the glove box arrangement, all leads from the four-point array were attached to a terminal strip mounted on the inside wall which was, in turn, connected through the box wall to matching external connections.

Table 1 is a condensation of potential difference data obtained at $27^{o}C$ with a fixed frequency of 1000 Hz for two concentrations of KCl. Constant current was maintained at 1.5×10^{-4} Amp, with $V_3 = 150$ mV across the 1000 ohm standard resistor. The potential difference of 3.2 mV at 1.6 cm spacing for 0.1 NKCl may be compared with the value of 20.0 mV for 0.01 N KCl. As expected, the more dilute electrolyte has a lower conductivity and the ratio of conductivities is in close agreement with the ratio of potential differences, i.e., 28.7/3.2 = 8.97, whereas the conductivities are in the ratio 8.77, in fair agreement to within 2.2 per cent. This agreement may be considered as a general measure of the accuracy of the conductivity measurement possible with the smooth platinum electrodes of the four-point probe array. Such accuracy is not sufficient to permit the refined measurements needed for evaluating theories of electrolytic conductance but is sufficient for the engineering design of optimum battery and cell configurations.

2.5 Determination of Cell Constant

As determined empirically from the potential difference data for V_1 and V_2 as a function of vertical spacing in the electrolyte-filled beaker (Sec. 2.4), a distance of 2 cm, which lies in a region of constant V_2, was selected for the calibration measurements. The potential difference, V_2, was frequency independent, as expected, (Table 2) and allowed the cell constant to be calculated for 0.01 N KCl solution of a known conductivity by using the flux relation:

$$J = \frac{i}{A} = -K \frac{\partial V}{\partial X}$$

TABLE 1

Effect of Position on Four-Point Probe Measurements

Constant current, $i = 1.5 \times 10^{-4}$ Amp, $V_3 = 150$ mV

Constant frequency, $f = 1{,}000$ Hz. $T = 27^{\circ}C$

	Potentials			
	0.1 N KCl		0.01 N KCl	
Spacing (cm.)	V_1(mV)	V_2(mV)	V_1(mV)	V_2(mV)
0.1	12.9	4.9	102.0	34.2
0.2	11.7	4.0	100.5	32.9
0.5	11.1	3.5	97.1	30.1
1.0	10.8	3.2	95.8	29.0
1.6	10.8	3.2	95.7	28.7
2.2	10.7	3.2	96.0	28.8
3.0	10.9	3.3	97.8	29.4

where J is current density, i is current, A is area, X is spacing and V is voltage. For 0.01 N KCl, K = 0.0014975 $\mathrm{ohm}^{-1}\mathrm{cm}^{-1}$ at 27^{0}C. V_2 was measured to be 28.7 mV (Table 1). The flux relation may be written in the following form:

$$c' = \frac{K\ V_2}{i}$$

where c' is the cell constant. With the current, i, kept constant at 1.5×10^{-4} Amp,

$$c' = \frac{0.0014975\mathrm{ohm}^{-1}\mathrm{cm}^{-1} \times 28.7 \times 10^{-3}\ V_oH}{1.5 \times 10^{-4}\ \mathrm{Amp}}$$

$$c' = 0.228\ \mathrm{cm}^{-1}$$

The potential difference, V_2, was then measured for 0.1N KCl, keeping the same experimental arrangement. Since the geometry of the cell was not changed, the above cell constant, c', was used to calculate the conductance of 0.1 N KCl.

V_2 was measured to be 3.2 mV (Table 1) with current constant, equal to 1.5×10^{-4} Amp. Using the cell constant 0.288 cm^{-1}, the conductance of 0.1 N KCl is 0.0135 $\mathrm{ohm}^{-1}\mathrm{cm}^{-1}$ at 27^{o}C, in good agreement with 0.013663 $\mathrm{ohm}^{-1}\mathrm{cm}^{-1}$, the accepted value.

Assuming the cell constant to be correct as determined with 0.01 N KCl, the measured conductivity for 0.1 N KCl is in error by 1.4% (negative).

A similar comparison made on the basis of V_2 measurements at a selected position of 1.6 cm drove the beaker (Sec. 2.4) indicated an accuracy of 2% could be expected for conductivity measurements using the four-point probe and beaker combination with SO_2Cl_2-$LiAlCl_4$ mixtures in the glove box.

2.6 Frequency Dependence

The existence of double layer capacitance at the electrode-electrolyte interface is the cause of a frequency dependent component of the cell impedance in two-point conductivity measurements. Because the dielectric constant of SO_2Cl_2 is 10 in contrast with the value of 80 for water, a small value of interfacial capacitance is expected (on the order of 2 to 3 $\mu F\ cm^{-2}$). Thus, a large contribution to impedance will occur even at higher frequencies where the effect is negligible in aqueous electrolytes. The expedient of decreasing the capacitive impedance term by increasing the electrode area through platinization was not used because of possible catalytic promotion of SO_2Cl_2 decomposition.

Because the inner probes of the four-point probe does not carry current, the potential difference measured by the inner probes, V_2, should be independent of frequency. This was checked by positioning the probe in a region demonstrated to be unaffected by vertical position (Sec. 2.5) and determining the current and outer and inner electrode potential differences for a range of frequencies. Two concentrations of KCl were used in the 250 ml beaker used previously (Sec. 2.5), filled to a level of 4.3cm. in each case, with the bottom of the electrode array positioned 2 cm. above the bottom of the beaker.

Table 2 shows results for the inner probe potential difference, V_2, and the outer, current carrying probe potential difference, V_1, for a constant cell current of 1.5×10^{-4} Amp (V_3 = 150 mV across 1000 ohm). Over the frequency range from 500 Hz to about 8000 Hz, the outer electrode potential difference V_1 shows considerable variation, decreasing by approximately a threefold change for 0.1 N KCl. The variation of V_1 is less for 0.01 N KCl and occurs in a smaller frequency range, from 500 to about 1500 Hz.

For both concentrations of KCl shown in Table 2, the inner probe potential difference, V_2, is shown to be essentially independent of frequency in the range 500 to 20,000 Hz. With the cell constant thus verified over the indicated frequency range, the four-point probe array was ready for use in the glove box with SO_2Cl_2-$LiAlCl_4$ electrolytes.

TABLE 2

Four-point Probe Calibration Measurements at 27°C

Constant spacing between the electrode and bottom of the cell, 2.0 cm.
Constant current $i = 1.5 \times 10^{-4}$ Amp; $V_3 = 150$ mV

	0.1 N KCl		0.01 N KCl	
Frequency (Hz.)	V_1 (mV)	V_2 (mV)	V_1 (mV)	V_2 (mV)
500	27.9	3.2	103.7	28.8
1000	18.1	3.2	99.2	28.8
1500	14.9	3.2	97.9	28.7
2000	13.5	3.2	97.2	28.7
4000	11.6	3.2	96.3	28.6
6000	11.2	3.2	95.9	28.6
8000	10.9	3.2	95.9	28.7
10000	10.8	3.2	95.8	28.7
12000	10.7	3.2	95.8	28.8
14000	10.7	3.2	95.8	28.8
16000	10.6	3.2	95.8	28.8
20000	10.6	3.2	96.0	28.8

3. Experimental Procedure

3.1 Preparation of Electrolyte:

Lithium Aluminum Chloride in Sulphuryl Chloride
($LiAlCl_4$-SO_2Cl_2)

Sulphuryl chloride was obtained from ROC/RIC, Belleville, N.J. and was purified before use for conductivity measurement. A vacuum line was used to facilitate degasification, dehydration, and distillation, as described below.

In an access bulb (250 ml capacity) connected to the vacuum line, 100 ml of SO_2Cl_2 was stored over approximately 20 grams of Linde molecular sieves type 4A for two days for dehydration. The solvent was degasified by freeze-pump-thaw cycles. SO_2Cl_2 was then distilled into a container on the vacuum line containing freshly cut lithium chips (approximately 8x3 mm) to further dehydrate the solvent. SO_2Cl_2 was kept in contact with the lithium chips for at least two days. Several batches prepared in this manner were transferred to the glove box (argon flushed) and stored for subsequent addition to the conductivity cell.

Pure anhydrous $LiAlCl_4$ was obtained from Anderson Physics Laboratory (Urbana, Ill.) in the form of a fused mass (250 gram) sealed in a glass ampule (8 in. long, 1-½ in. diam.) and was used without any further purification. The seal of the glass used ampule was broken inside the Labconco glove box under argon and the ampule was heated by wrapped electrical tape. The molten $LiAlCl_4$ (m.pt. = $144^{\circ}C$) was readily collected in the 250 ml beaker used with the four-point probe array and was dissolved rapidly with the aid of a Teflon coated magnetic stirrer. The 1 ml samples withdrawn after each addition of $LiAlCl_4$ did not change the 4.3 cm depth of solution significantly and no attempt was made to add fresh SO_2Cl_2 to counteract what little evaporation took place.

3.2 Analysis of Electrolyte Composition

The 1 ml samples pipetted from the conductivity cell were stored in sealed tubes within the glove box until the finish of $LiAlCl_4$ additions to the main stock of electrolyte and corresponding conductivity measurements. These sample tubes were taken out in a closed glass container, which was designed so that it could be connected to the vacuum line to evaporate SO_2Cl_2 from the tubes. Following this step, the tubes plus $LiAlCl_4$ residue were accurately weighed. This procedure was followed by thorough washing with water to remove the $LiAlCl_4$, after which, the tubes were dried and again weighed to permit calculation of the $LiAlCl_4$ by weight difference. A Mettler H 16 type analytical balance was used to weigh the tubes. Because of the small difference in weight for the residue of $LiAlCl_4$ corresponding to the lowest concentration, great accuracy cannot be expected for this method. Blank determinations with 1 ml of pure SO_2Cl_2 indicated an overall uncertainty of ±0.005 gm. Accordingly, the lower concentration values are most in doubt. This is unfortunate because of the need for accuracy in both concentration and conductivity determination in the low concentration region for the comparison of data with the predictions of theories of conductance in limitingly dilute electrolytes. Nevertheless, for the larger concentrations of practical significance to battery applications, the uncertainty in concentration at the level of 1 mole per liter of $LiAlCl_4$ in SO_2Cl_2 was only ±3 per cent.

4. Results and Discussion

In the determination of conductivity for each concentration of $LiAlCl_4$ in SO_2Cl_2, the frequency range from 500 to 20,000 Hz was spanned with selected frequencies as indicated in Table 2. As for the calibration studies with aqueous KCl solutions, the inner probe potentials were essentially independent of frequency. Uncertainties in the third significant figure (0.1 mV level) were averaged out by combining V_2 (inner probe) values over the entire frequency range, i.e. twelve individual conductivity measurements were averaged for each concentration. The results of conductivity measurement are shown in Table 3.

The measured value of specific conductivity for pure SO_2Cl_2 (1.8×10^{-7} ohm^{-1} cm^{-1} at 27°C) is only in fair agreement with a value of 2×10^{-8} ohm^{-1} cm^{-1} at 20°C, cited in Sec. 1.2.1 in a previous report of this contract (ECOM 75-1676-1). In the range 0.1 to 1 mole per liter of $LiAlCl_4$, the conductivity is on the order of 10^{-3} ohm^{-1} cm^{-1}, approximately one order of magnitude lower than is desirable for battery electrolyte. The equivalent conductance ranges from 1 to 2 ohm^{-1} cm^{+2} eq^{-1}, low in comparison aqueous electrolyte values which for KCl is 129 at 0.1 equi. per liter.

TABLE 3

Conductivity in the System $LiAlCl_4$-SO_2Cl_2

Concentration of $LiAlCl_4$ mol/liter	Specific Conductivity k, $ohm^{-1}cm^{-1}$	Temperature °C
0.0	1.8×10^{-7}	27.0
0.06 ±.03	$0.76\ (\pm .04) \times 10^{-4}$	30.0
0.51 ±.03	$11.1\ (\pm .1) \times 10^{-4}$	33.5
0.62 ±.03	$7.40 (\pm .04) \times 10^{-4}$	32.0
0.74 ±.03	$10.3 (\pm .2) \times 10^{-4}$	31.0
0.96 ±.03	$7.22 (\pm .07) \times 10^{-4}$	31.0

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