

## SOLID ELECTROLYTE INTERPHASE (SEI) ELECTRODES PART VI: CALCIUM-Ca(AlCl<sub>4</sub>)<sub>2</sub>-SULFURYL CHLORIDE SYSTEM

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### Summary

The electrochemistry of the Ca/Ca(AlCl<sub>4</sub>)<sub>2</sub>-sulfuryl chloride (SC) system and its capability of functioning as a primary cell were studied. The OCV of this cell is about 3.4 V and it exhibits a rather flat discharge curve of 3.1 - 2.9 V at current densities of 0.4 - 2.0 mA cm<sup>-2</sup>. Cell capacity is 35 - 45 mA h cm<sup>-2</sup> (for 1.1 mm thick porous carbon cathodes). Cell failure results from anodic polarization. Cells were found to resist charging and reversal abuses and no indication of calcium deposition during reversal was found. It was found that the calcium anode is covered by a thin film (most likely CaCl<sub>2</sub>) which serves as a SEI. Its thickness on fresh electrodes is about 15 - 30 Å and it increases with time. The rate determining step for the calcium dissolution process is the ionic migration through the SEI. The ionic resistivity of the SEI is about  $4 \times 10^9 \Omega \text{ cm}$ .

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### Introduction

The lithium-thionyl chloride (TC) cell has the highest energy density of all commercially available primary cells. However, the major obstacle to the marketing of a high-rate type cell and, in particular, a high-rate type multi-cell battery, is its known explosion hazard during reversal or charging [1 - 3]. The basic reason for this is the fact that lithium is deposited at low overpotentials, on itself, or on a passivated cathode [4]. In order to develop a safer system, both the Ca/LiAlCl<sub>4</sub>-TC [5, 6] and the Ca/Ca(AlCl<sub>4</sub>)<sub>2</sub>-TC [4, 5, 7 - 10] systems were studied. The use of the LiAlCl<sub>4</sub> electrolyte in the calcium anode cell does not eliminate the deposition of lithium on the anode during charging or into the cathode during reversal [4].

The Ca/Ca(AlCl<sub>4</sub>)<sub>2</sub>-thionyl chloride cell has been proved to be a safer alternative to high-rate lithium cells [4, 7 - 9]. When it is fully developed, this cell is expected to have about the same energy density as the Li-SO<sub>2</sub> system [4]. Laboratory prototype C-size "jelly roll" Ca/Ca(AlCl<sub>4</sub>)<sub>2</sub>-TC cells with 100 cm<sup>2</sup> electrode areas delivered 3.5 A h at 0.125 A, and 2.05 A h at 0.8 A [8]. Unlike lithium cells it resists charging and reversal abuses. The

steady-state value of the current during charging or reversal abuse tests at 25 V was lower than 20 mA. Its shelf life is expected to be about two years at room temperature [8].

The calcium-sulfuryl chloride cell has a higher OCV (3.35 - 3.4 V) [11, 12] than calcium-TC (3.25 V) [4]. The theoretical energy density for a two electrons per molecule reduction is about the same for both TC and SC. The Ca/1.2M  $\text{Ca}(\text{AlCl}_4)_2$ -SC cell exhibits a stable discharge voltage plateau of 3 - 3.2 V (at current densities of 0.4 - 2 mA  $\text{cm}^{-2}$ ) for about 50 to 70% of the service life [11], and cell failure is found to result from anodic polarization. Cell capacity at current densities of 0.4 - 2 mA  $\text{cm}^{-2}$  was 35 - 45 mA h  $\text{cm}^{-2}$  for a 1.1 mm thick Teflon-bonded carbon cathode [11]. The conductivity of  $\text{Ca}(\text{AlCl}_4)_2$ -SC solutions is about one third that of TC solutions [11]. Binder *et al.* [12] found that at moderate current densities, the cathodes of Ca-SC cells formulated with small volumes of  $\text{LiAlCl}_4$  electrolyte provide as little as 30% of the capacity normally recorded for similar lithium cells, while the results for cells formulated with  $\text{Ca}(\text{AlCl}_4)_2$  were even poorer.

Calcium-TC and calcium-SC as well as lithium cells belong to the family of nonaqueous batteries called Solid-Electrolyte-Interphase (SEI) batteries [13, 14]. These batteries have anodes that are always covered by a film which separates them from the solution. This film has the properties of a solid electrolyte, that is, it is a very poor electronic conductor but a rather good ionic conductor. The resistivity of this SEI is in the range  $10^7 - 10^{10} \Omega \text{ cm}$  [13, 14]. The "diode" behaviour of the Ca/ $\text{Ca}^{2+}$ -TC system (*i.e.*, delivery of high current densities in the anodic direction and high resistance in the cathodic direction) was concluded to be the result of almost pure anionic ( $t_- = 1$ ) conduction of  $\text{CaCl}_2$ -SEI [4, 5, 7, 15].

The goal of this work was to study the electrochemistry of the Ca/ $\text{Ca}(\text{AlCl}_4)_2$ -SC system and to evaluate its capability of functioning as a primary cell.

## Experimental

All solutions were prepared in an argon-filled glove box. Sulfuryl chloride (Fluka, puriss) was vacuum distilled. Aluminium chloride (Fluka, AR) was vacuum sublimed, and calcium chloride (Frutarom AR) was vacuum dried for 24 h at 200 °C. Sulfuryl chloride was added to  $\text{AlCl}_3$  and stirred for 24 h; then the solution was filtered through a glass filter paper. Calcium chloride (*ca.* 20% excess) was slowly added to this solution which was then stirred for 24 h. The final concentrations of Ca and Al were determined by atomic absorption spectrophotometry. Conductivity measurements were performed in 10 ml cells with Rotaflo stopcocks and platinized platinum electrodes using a standard conductivity meter (Radiometer - Copenhagen). The temperature was controlled (to  $\pm 1$  °C) by a refrigerated circulating bath (RTE-4, Neslab).

The calcium metal (Roc/Ric, 99%) was first degreased in AR acetone, abraded with sandpaper, annealed at 700 - 750 °C for two hours, and transferred to a glove-box where it was again abraded to the final thickness of about 0.47 mm.

The electrochemical experiments were carried out using hermetically sealed glass cells, which are described in detail in ref. 4. The cell consists of a 1.0 - 1.2 mm-thick Teflon-bonded carbon cathode having an area of 5 or 6 cm<sup>2</sup> and a 0.47 mm-thick calcium anode of the same area. Some of the cells also contained a 2 - 3 mm wide calcium reference electrode. A piece of non-woven, 0.18 mm-thick glass paper separated the electrodes and two 316 stainless-steel plates supported them. The discharge tests were performed by loading the cell with a suitable resistor and the results were recorded by a (Fluke 2240B) datalogger. An Elron (model CHG-1) galvanostat was used in polarization, charging, and reversal tests. The results were recorded on a Yokogawa (type 3046) strip-chart recorder. Reaction resistance and electrode interfacial capacitance were determined by a pulse galvanostatic technique [16]. A home-made pulse galvanostat and a Tektronix 564 storage oscilloscope were used in these experiments.

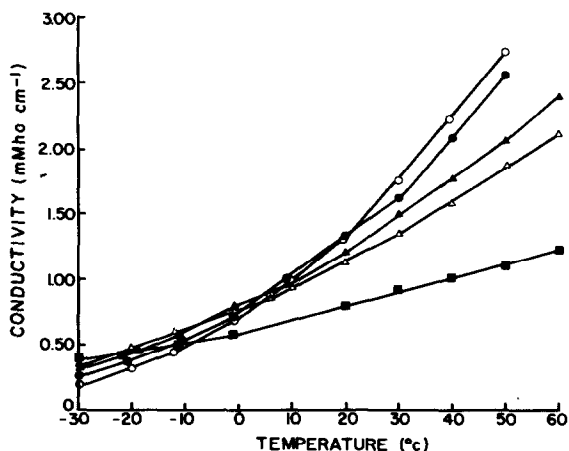


Fig. 1. Specific conductivity of  $\text{Ca}(\text{AlCl}_4)_2\text{-SO}_2\text{Cl}_2$  solutions as a function of temperature. ○, 1.35M; ●, 1.20M; ▲, 1.00M; △, 0.90M; ■, 0.60M.

## Results

### Conductivity measurements

The conductivity of  $\text{Ca}(\text{AlCl}_4)_2\text{-SC}$  solution was measured from -30 to +60 °C for an electrolyte concentration range of 0.6 - 1.35M. The effect of temperature at constant electrolyte concentration can be seen in Fig. 1. Over the whole concentration range the conductivity increases with temperature. Above 30 °C the conductivity increases with concentration while at -30 °C it decreases with concentration. This can be more clearly seen in Fig. 2. Over

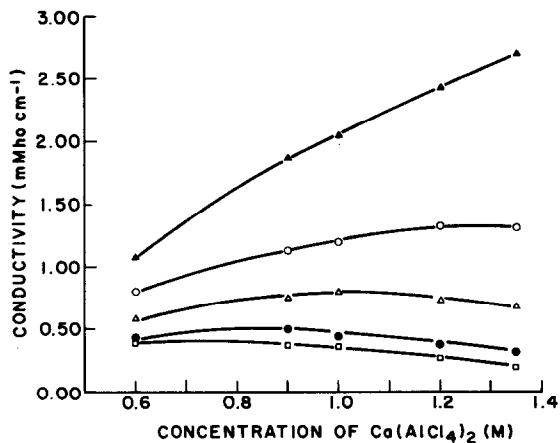


Fig. 2. Specific conductivity of  $\text{Ca}(\text{AlCl}_4)_2$  solutions as a function of concentration.  $\square$ ,  $-30^\circ\text{C}$ ;  $\bullet$ ,  $-20^\circ\text{C}$ ;  $\triangle$ ,  $-1^\circ\text{C}$ ;  $\circ$ ,  $20^\circ\text{C}$ ;  $\blacktriangle$ ,  $50^\circ\text{C}$ .

the temperature range  $-20 - +20^\circ\text{C}$  the conductivity *vs.* concentration plots have maxima which move toward higher concentration as the temperature is increased. It is important to notice that no salt precipitation was observed even at the highest concentration and the lowest temperature.

### Kinetic measurements

As soon as the cell was filled with electrolyte, pulse galvanostatic experiments were periodically conducted. From the data obtained, micropolarization curves were drawn. Typical anodic micropolarization curves are shown in Fig. 3. The reaction resistance was calculated from these curves. It should be emphasized that a linear relationship between the anodic overpotential and the current density is valid up to 200 mV for electrodes which were immersed for about 20 h.

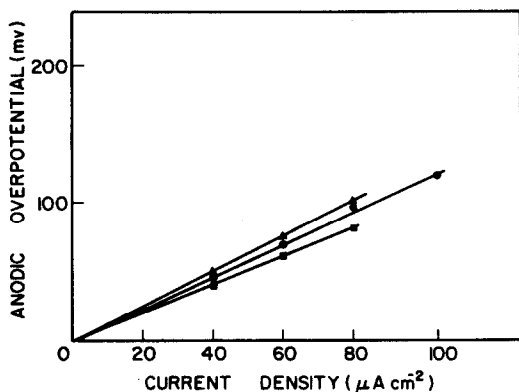


Fig. 3. A plot of the *IR*-free anodic overpotential *vs.* current density at different storage times;  $1.35\text{M Ca}(\text{AlCl}_4)_2$ .  $\bullet$ , 1.2 h;  $\blacksquare$ , 6.3 h;  $\blacktriangle$ , 23.3 h.

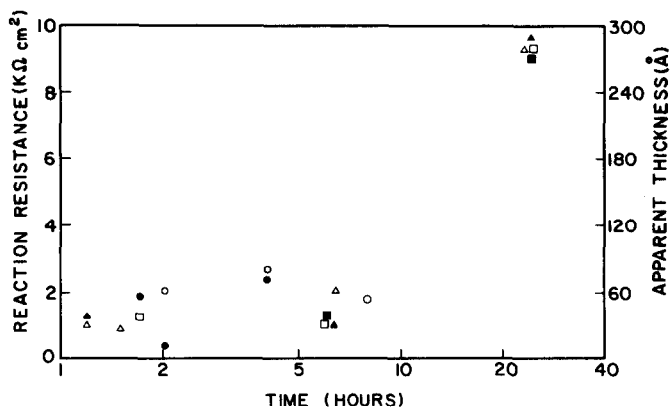


Fig. 4. Reaction resistance and apparent SEI thickness vs. storage time. Thickness:  $\Delta$ , 1.35M;  $\square$ , 1.50M;  $\circ$ , 1.20M. Resistance:  $\blacktriangle$ , 1.35M;  $\blacksquare$ , 1.50M;  $\bullet$ , 1.20M.

The apparent thickness of the SEI was calculated from differential electrode capacitance using an equation for a parallel-plate capacitor [16] where the value of the dielectric constant of the SEI was assumed to be ten. The differential capacitance of the calcium electrode was calculated from the initial slope of the potential/time transient. The dependence of the reaction resistance and the apparent thickness of the SEI on immersion time are shown in Fig. 4. During the first ten hours of immersion the apparent thickness of the SEI is about 30 - 80 Å while the reaction resistance is about 500 - 2500  $\Omega \text{ cm}^2$ . After 24 h of immersion the thickness rises to 280 Å and the reaction resistance to 9000  $\Omega \text{ cm}^2$ . After longer periods of time the thickness of the SEI increases further and no reproducible and stable transients were recorded. This may partially result from damage to the relatively thick passivating layer by the short current pulses.

### Cell tests

The corrosion rate of calcium in SC solutions is faster than that in TC solutions. After a few days of immersion of Ca in 0.6 - 1.35M  $\text{Ca}(\text{AlCl}_4)_2$ -SC solutions at room temperature, pits start to develop near the edges of the sample. The fastest corrosion takes place at the solution/gas interface. This leads to a disconnection of the Ca anode from the current collector and to premature failure of the cell.

The initial OCV of fresh cells immediately after filling with electrolyte was 2.5 - 2.8 V, and it rose to 3.0 - 3.25 V after 5 min and to 3.35 - 3.4 V after 12 - 18 h. The cells were discharged across a constant load within 1 - 2 days from the time of filling with electrolyte. Cells with 0.9 - 1.35M  $\text{Ca}(\text{AlCl}_4)_2$  were discharged at room temperature at c. ds. of 0.4 - 2 mA  $\text{cm}^{-2}$ . For 50 °C discharge tests a higher electrolyte concentration of 1.5M was chosen as its conductivity is higher. Cells exhibit a flat discharge curve of 2.9 - 3.1 V

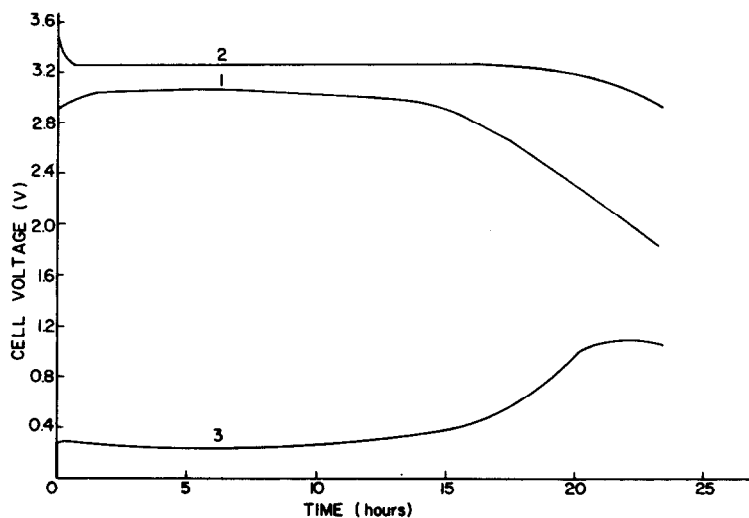


Fig. 5. Constant load ( $330 \Omega$ ) discharge curve of a calcium-sulfuryl chloride cell ( $1.35M \text{Ca}(\text{AlCl}_4)_2$ ) at room temperature, electrode area:  $5 \text{ cm}^2$ . 1, cell voltage; 2, reference-cathode; 3, reference-anode.

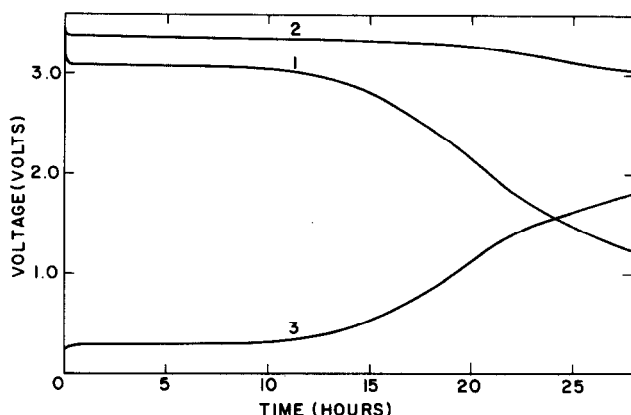


Fig. 6. Constant load ( $330 \Omega$ ) discharge curve of a  $\text{Ca}-1.5M \text{Ca}(\text{AlCl}_4)_2\text{-SC}$  cell at  $50^\circ \text{C}$ , electrode area:  $5 \text{ cm}^2$ . 1, cell voltage; 2, reference-cathode; 3, reference-anode.

depending on the c. d. and temperature of discharge. Although the theoretical capacity of the anode was  $97 \text{ mA h cm}^{-2}$ , cells delivered only  $35 - 45 \text{ mA h cm}^{-2}$  (to a cut-off voltage of  $2 \text{ V}$ ). Discharge of cells at high c. ds. results in a large loss of capacity and lower working voltage. A cell with  $1.2M$  electrolyte which was discharged at  $10 \text{ mA cm}^{-2}$  maintained a stable voltage of  $2.65 \text{ V}$  for only  $30 \text{ min}$  and then the voltage declined sharply. This cell delivered only about  $10\%$  of the capacity measured on  $1.2 \text{ mA cm}^{-2}$  discharge tests. In order to find the limiting capacity factor, cells having calcium reference electrodes were assembled and tested, both at room temperature with  $1.35M$  electrolyte (Fig. 5) and at  $50^\circ \text{C}$  with  $1.5M$  electrolyte (Fig. 6).

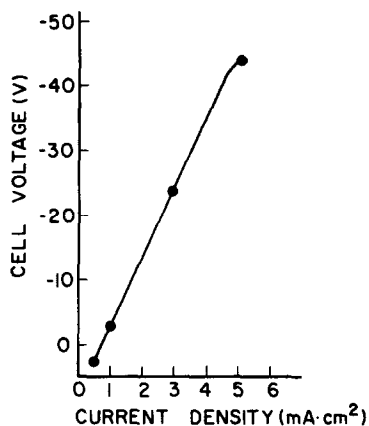


Fig. 7. Reversal test of a fully discharged cell (1.20M  $\text{Ca}(\text{AlCl}_4)_2$ ). The cell was fully discharged at  $1.5 \text{ mA cm}^{-2}$  prior to reversal test.

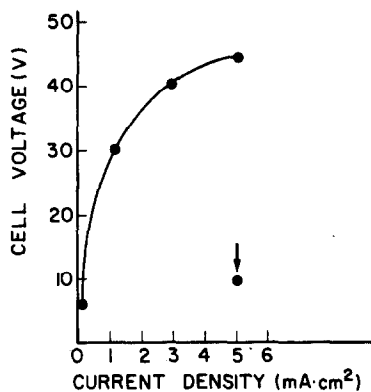


Fig. 8. Charge test of a discharged cell (1.20M electrolyte) at room temperature.

It can be seen that for up to 20 h of discharge the cathode reference potential is above 3.2 V at room temperature and above 3.3 V at 50 °C, and decreases only slightly later. This means that during most of the service life of the cell, the cathode potential is close to the OCV of the cell. The anodic polarization is about 0.3 V during the first 10 h of discharge (at both temperatures) and rises sharply to above 1 V after 20 h of discharge. In parallel with this development of anodic polarization, the cell voltage drops until the cell fails (Figs. 5 and 6).

Inspection of fully discharged cells showed that the anodes were covered by a hard, black material. The edges of the anode were corroded (or preferentially anodically dissolved) and, in some cases they had disintegrated and pieces were missing. In some of the cells the calcium strip which connected the anode to the tungsten rod broke during discharge.

The cells were found to resist successfully galvanostatic charging and reversal (overdischarge) abuse tests. No indication of calcium deposition on the cathode or significant gas evolution were observed during these tests. Figure 7 shows a plot of the current density *vs.* reverse voltage of a cell which contained 1.2M electrolyte and was fully discharged at  $1.5 \text{ mA cm}^{-2}$  prior to the reversal test. The reverse voltage increases linearly with the current density. However, a very high reverse voltage is needed to force current through the cell. At  $-30 \text{ V}$  the current density is only  $3.5 \text{ mA cm}^{-2}$ . Immediately after interrupting a reversal test, the cell voltage jumps to above 2 V, indicating no calcium deposition on the cathode. Similar results were obtained in charging abuse tests. A very high voltage was needed to force a "charging" current through the cell (Fig. 8). At a voltage as high as 30 V the current density was only  $1.5 \text{ mA cm}^{-2}$ . However, at a c.d. above  $5 \text{ mA cm}^{-2}$  the voltage rose to about 43 V, and after a few seconds at this voltage it suddenly and sharply fell to about 10 V and stayed at this level.

## Discussion

The solubility of  $\text{Ca}(\text{AlCl}_4)_2$  in SC is above 1.35M over the temperature range  $-30 - 0^\circ\text{C}$  and above 1.5M at  $20 - 50^\circ\text{C}$ . The conductivity of these solutions is about one third that of  $\text{Ca}(\text{AlCl}_4)_2$ -TC solutions and about one tenth that of  $\text{LiAlCl}_4$ -TC solutions over the temperature range  $-30$  to  $+50^\circ\text{C}$ . This limits the rate capability of this system and makes it inferior to the other two. Calcium-TC cells deliver 60% of their capacity at rates as high as  $11 \text{ mA cm}^{-2}$  [4] and can function at an even higher rate ( $50 \text{ mA cm}^{-2}$ ) in their reserve type version [7]. The Ca-SC cell, on the other hand, delivers only 10% of its capacity at  $10 \text{ mA cm}^{-2}$ . The conduction mechanism of the electrolyte appears to be complex, as might be expected for low dielectric constant solvents [17]. In such solvents it is expected that aggregates of several ions will be formed. The conductivity of  $0.4 - 0.7\text{M}$   $\text{Ca}(\text{AlCl}_4)_2$ -TC solutions increases with decrease in temperature [4]. This was explained by the formation at lower temperatures of larger ionic aggregates which have a smaller association constant. The behaviour of the conductivity of  $0.6\text{M}$   $\text{Ca}(\text{AlCl}_4)_2$ -SC solution at temperatures below  $0^\circ\text{C}$  may be partially attributed to such an effect. However the major parameter affecting the conductivity of the SC solutions seems to be the dependence of their viscosity on electrolyte concentration and on temperature. Concentrated SC solutions are highly viscous, especially at low temperatures. Therefore, the increase in conductivity with temperature (Fig. 2) is attributed mostly to the increase in the viscosity.

The activation energy ( $E_a$ ) for conduction was calculated from Arrhenius plots. Typical Arrhenius plots are shown in Fig. 9. The activation energy decreases from 2.04 kcal/mol for 1.35M solution to 0.98 kcal/mol for 0.6M

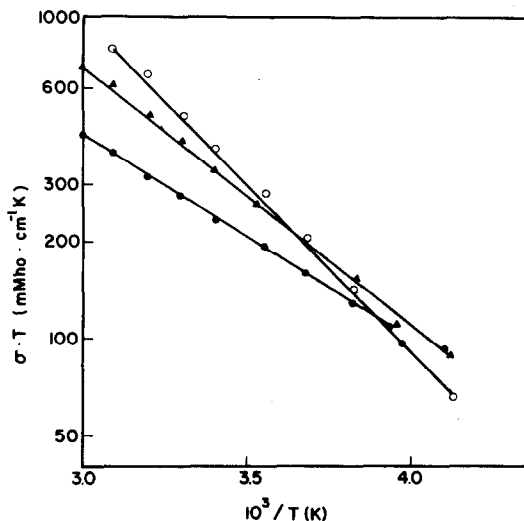


Fig. 9. Arrhenius plots for conductivity of  $\text{Ca}(\text{AlCl}_4)_2$  solutions. ●, 0.60M; ▲, 0.90M; ○, 1.20M.



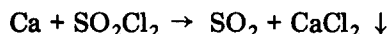
TABLE 1

The activation energy for conductance in  $\text{Ca}(\text{AlCl}_4)_2$ -TC solutions

Concentration (M)	Activation energy (kcal/mol)
0.60	0.98
0.90	1.52
1.00	1.73
1.20	2.08
1.35	2.04

solution (Table 1). A similar decrease of  $E_a$  with decrease in electrolyte concentration was found for  $\text{LiAlCl}_4$ -TC solutions [18]. In the case of 0.4 - 0.7M  $\text{Ca}(\text{AlCl}_4)_2$ -TC solutions, an apparent "negative" energy of activation was observed (*i.e.*, the conductivity increases with decrease in temperature). These phenomena observed in the three systems seem to result from the dependence of the size (and therefore the concentration) of the ionic aggregates on temperature.

Calcium is thermodynamically unstable with respect to SC. On immersion of calcium in SC solutions it immediately attacks the solvent. The proposed [12] cell reaction is:



Calcium chloride is insoluble in SC and therefore it precipitates on the anode surface to form a passivating layer. It was concluded that for many SEI battery systems [14] this passivating layer consists of two parts. One, thin and compact (25 - 5000 Å), close to the surface of the anode, serves as the SEI. On top of it a thick, porous, secondary layer is developed.

The fact that the anodic overpotential *vs.* c. d. plot is linear up to 200 mV indicates that the Tafel slope for this process is above 1 V. Therefore it can be concluded [14] that the rate-determining step for the anodic dissolution process of calcium is the ionic migration through the SEI. The SEI resistivity fluctuates between  $5 \times 10^8$  and  $5 \times 10^9 \Omega \text{ cm}$  in the first 30 h of immersion. Similar values were measured in the Ca-TC system.

The corrosion rate of calcium in SC solutions is greater than in TC solutions. This causes premature failure in many cells as a result of mechanical breaks in the calcium foil at the solution/gas interface. This may be partly explained by the difference in the preparation procedures for TC and for SC solutions. The TC solutions were refluxed [4], while the SC solutions were not. As a result, it is possible that the SC solutions contain a higher concentration of hydrolysis products of the electrolyte. In addition, we found that SC is more harmful to other cell materials and tools than is TC.

The OCV of the Ca-SC cell is only 100 - 150 mV higher than that of the Ca-TC cell. However, the working potential at low c. d.s is similar while

at high c. d.s that of the Ca-TC cell is higher. This finding is at variance with the polarization curves measured by Binder *et al.* [12]. This discrepancy may result from the difference in the electrolyte concentration, since we used twice as high a concentration. The increase of the OCV with aging of freshly filled cells is a common phenomenon in many liquid cathode cells such as Li, Mg, Ca-TC and SC systems. It results from the decrease of the corrosion rate with increase in the thickness and the order of the SEI.

The Ca/Ca(AlCl<sub>4</sub>)<sub>2</sub>-SC cell exhibits, at low c.d.s a flat discharge curve for up to about two thirds of its service life. While the cathodic polarization is small (100 - 150 mV) for most of the discharge time, an anodic overpotential is developed after 20 - 25 mA h cm<sup>-2</sup> (50% of cell capacity) and it is responsible for cell failure both at room temperature and at 50 °C. The thick passivating layer found on the anode in *post mortem* examination of discharged cells supports the conclusion that anode passivation during discharge is responsible for cell failure. Similar heavy anodic polarization was observed in the discharge of Ca-TC cells having high electrolyte concentration (1.9M Ca(AlCl<sub>4</sub>)<sub>2</sub>) [15]. The process leading to the development of the anodic overpotential in Ca-SC and Ca-TC [15] cells having a concentrated electrolyte may be similar to those responsible for the voltage delay phenomenon observed in many lithium cells. This type of polarization is associated [14] with the build-up of electrolyte concentration in the pores of the secondary passivating layer which covers the anode. This, in turn, causes a further increase in the viscosity of the solution, in the pores of the secondary layer and, as a result, the IR voltage drop across this layer increases. If the electrolyte concentration near the SEI/secondary layer interface exceeds the solubility of the electrolyte salt, it will precipitate in this region. This precipitation process may lead to blocking of the anode and thus to a high IR voltage drop. This problem can be overcome, at least to some extent, by decreasing the viscosity of the solution [14]. In the Ca-TC case this was done by reducing the electrolyte concentration from 1.9 to 0.9M [11].

The hazardous properties of lithium high-rate cells make the safety features of calcium cells very attractive. As for the Ca/Ca<sup>2+</sup>-TC cell, it was found that passing a "charging" or reversal current through Ca/Ca(AlCl<sub>4</sub>)<sub>2</sub>-SC cells requires a very high voltage (Figs. 7 and 8). Although in the SC system the current density values are higher than those for the parallel TC system, they are an order of magnitude or more lower than those for the lithium TC cells. In addition, no indications of calcium deposition during reversal were observed. The sharp drop of voltage in "charging" at 5 mA cm<sup>-2</sup> most likely results from a dielectric breakdown of the SEI [4, 7].

## Conclusion

The OCV of the Ca(AlCl<sub>4</sub>)<sub>2</sub>-SC cell is 3.35 - 3.4 V, 0.1 - 0.15 V higher than that of the parallel TC cell. However, its working potential is similar at low current densities and lower at high current densities. The conductivity of

$\text{Ca}(\text{AlCl}_4)_2$ -SC solutions is one third that of the parallel TC solutions, which makes it inferior with respect to high-rate capability. Like the TC cell, the SC cell resists charging and reversal abuses, but the current density passed through the cell during these tests is an order of magnitude higher. The corrosion of calcium in SC solutions is faster than in TC solutions. The  $\text{Ca}/1.2\text{M}$   $\text{Ca}(\text{AlCl}_4)_2$ -SC cell exhibits a stable discharge voltage plateau for up to 50% of its service life, which later falls as a result of anodic polarization and is ultimately responsible for cell failure.

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