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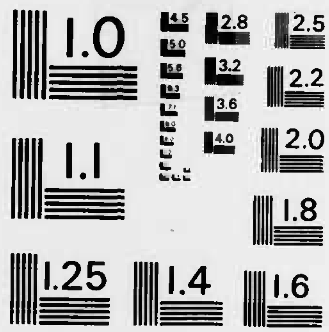


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A "neutral" melt of exactly 1.0:1.0 mole composition of $AlCl_3$:RCl, where $R^+$ is either N-(1-butyl) pyridinium or 1-methyl-3-ethyl imidazolium, has been shown to have an increased electrochemical window of 3.5 and 4.5 V, respectively. System characteristics are described.

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TECHNICAL REPORT NO. 15

# INCREASED ELECTROCHEMICAL WINDOW IN AMBIENT TEMPERATUE NEUTRAL IONIC LIQUIDS

by

Marek Lipsztajn and Robert A. Osteryoung

Prepared for Publication in

The Journal of the Electrochemical Society



Department of Chemistry
State University of New York at Buffalo
Buffalo, New York 14214

January, 1984

# Increased Electrochemical Window in Ambient Temperature Neutral Ionic Liquids

**M. Lipsztojn and R. A. Osteryoung***

*Department of Chemistry, State University of New York, Buffalo, New York 14214*

In the past several years, low temperature molten salt systems composed of aluminum chloride and alkylpyridinium salts have been found (1-3) to be useful solvents for electrochemical and spectroscopic studies of both organic and inorganic species. Recently Wilkes et al (4) have developed a group of dialkyl-imidazolium chloroaluminate melts having a wider cathodic limit than the alkylpyridinium chloroaluminates.

Both cathodic and anodic limits of these solvents depend on the mole ratio of components taken for the preparation of the molten salt. For so-called basic melts ($MC = \frac{n_{AlCl_3}}{n_{RCl}} < 1$) the anodic limit corresponds to the electrooxidation of chloride ions and is identical (5) for both N-n-butylpyridinium chloride (BuPyCl)-$AlCl_3$ and 1-methyl-3-ethylimidazolium chloride (ImCl)-$AlCl_3$ melts. The cathodic limit for basic melts corresponds to the electroreduction of the organic cation ($BuPy^+$ or $Im^+$).

For acidic melts (MC > 1) the anodic and cathodic limits are identical (5) for both RCl-$AlCl_3$ melts (R = $BuPy^+$ or $Im^+$) and correspond to, respectively, tetrachloroaluminate ($AlCl_4^-$) oxidation, to yield chlorine, and heptachloroaluminate ($Al_2Cl_7^-$) reduction, to yield Al.

The electrochemical windows for these melts measured at a tungsten electrode are shown in Figure 1.

The purpose of this communication is to present the great advantage of use of a neutral melt (MC = 1.00); preparation of this melt consists of weighing appropriate amounts of $AlCl_3$ and RCl, and mixing. This roughly neutral melt is subsequently adjusted by addition of small amounts of either $AlCl_3$ or RCl to obtain the proper electrochemical window. As shown in Figure 1 the limits established for neutral melts correspond to anodic oxidation of $AlCl_4^-$ and cathodic reduction of $R^+$, giving a very wide electrochemical window (∿3.6 V for 1:1 $AlCl_3$-BuPyCl and ∿4.4 V for 1:1 $AlCl_3$-ImCl melts respectively). This extension of the electrochemical window enables us to investigate several redox systems inaccessible for study in melts prepared by use of an excess of basic (RCl) or acidic ($AlCl_3$) components. Some of these systems are of fundamental importance in understanding the chemistry of these melts.

As an example, the electrochemistry obtained at a tungsten electrode in a 30 mmol solution of BuPyCl in neutral $AlCl_3$-ImCl melt is presented in Figure 2. The quasi-reversible oxidation of $Cl^-$ is observed (4) as a discrete anodic voltammetric wave, while cathodically the reduction of $BuPy^+$ occurs. More detailed studies of $Cl^-$ oxidation and $BuPy^+$ reduction will be presented in subsequent publications; however it is worth mentioning that this first process is convective diffusion controlled at a rotating disc electrode and that the $Cl^-$ oxidation wave can be very useful in studies of many chemical reactions, particularly those involving chloride complexation with a metal ion.

In a melt symmetric to that discussed above (i.e. a neutral melt with a small excess of $AlCl_3$ added (6)) the convective - diffusion controlled reduction of $Al_2Cl_7^-$ at a rotating disc electrode is observed and this wave can be also very useful.

The very wide electrochemical window of neutral $AlCl_3$-RCl melts appears very promising from the point of view of use as a solvent for new high energy density batteries. Applicability of this system to studies involving redox and conducting polymeric electrodes is in progress (7).

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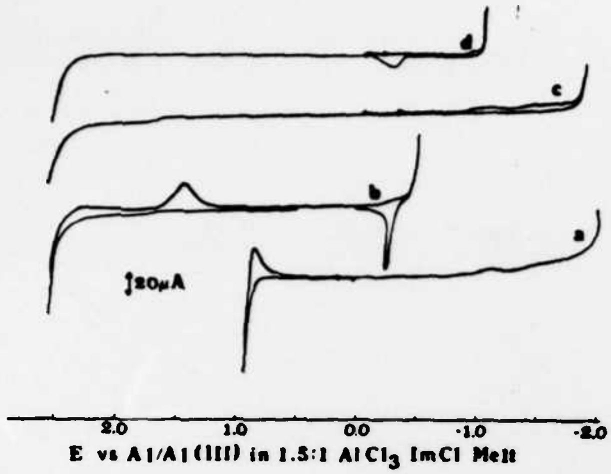
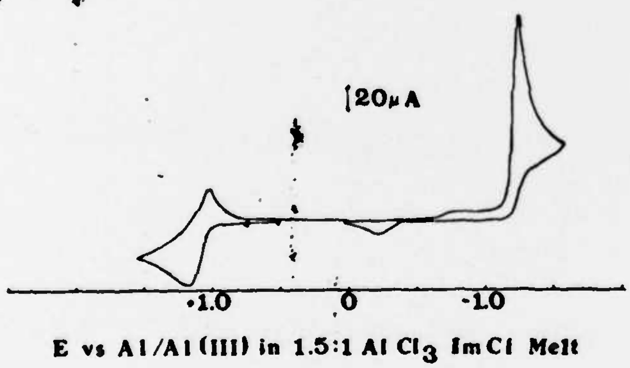
*Electrochemical Society Active Member

Key Words: Fused salts, depolarization, discharge, voltammetry




1. Cyclic voltammetric curves recorded on tungsten electrode (area 0.0784 $cm^2$) in
(a) basic $AlCl_3$-ImCl melt (MC = 0.67)
(b) acidic $AlCl_3$-ImCl melt (MC = 1.2)
(c) neutral $AlCl_3$-ImCl melt (MC = 1.00)
(d) neutral $AlCl_3$-BuPyCl melt (MC = 1.00)
Sweep rate = 0.05 V $s^{-1}$, temperature = 30°C.




2. Cyclic voltammetric curve recorded on tungsten electrode (area 0.0784 $cm^2$) at 30°C in 30 mmol solution of N-n-butylpyridinium chloride in neutral $AlCl_3$-ImCl melt (MC = .994), sweep rate 0.05 V $s^{-1}$.

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