

# MEASURING ELECTROCHEMICAL POTENTIALS IN BAYER LIQUORS

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## Abstract

Knowledge of the electrochemical potential ( $E_H$ ) is essential to the understanding of redox processes and the speciation of organic compounds and metal ions in Bayer process liquors, and for the development of technologies for the removal of organic compounds by oxidation. Despite this, there is little information in the literature on the measurement of  $E_H$  in Bayer process liquors. This paper reports preliminary studies aimed at filling this gap, and describes the general electrochemical characteristics of a particular Bayer process spent liquor.

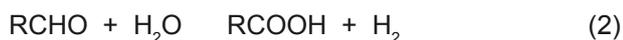
## 1. Introduction

The electrochemical potential ( $E_H$ ) of Bayer liquors (BLs) has so far received little attention in the scientific literature. This is surprising, because  $E_H$  is a key indicator of the species present and the reactions taking place in solution. The species present in BLs include a wide range of organic compounds and metal ions which are of importance to the safety, environmental impact and economic efficiency of the Bayer process. The electrochemical potential is also fundamental in the development of technologies for the destruction of organic compounds by chemical or electrochemical means (Perrotta and Williams, 2009, Thornber and Woods, 1996, Epron et al., 1999) or by catalytic wet oxidation (Eyer et al., 2002).

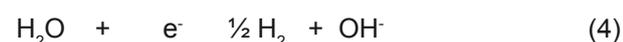
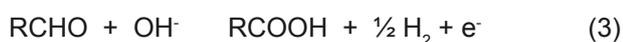
The conditions in Bayer process digestion (high hydroxide concentration and temperature) cause rapid degradation of the organic matter extracted from the bauxite to produce a complex mixture of products (Power and Loh, 2010). The overwhelming majority of these products are carboxylic acids, which are the oxidation products of the degradation processes. The corresponding reduction products include hydrogen (Costine et al., 2010b) and low molecular weight alkanes, alcohols and aldehydes (Galbally, 2004, Galbally, 2008) which report to the vapour. This overall process may be described by the flowing generic reaction:



In this reaction, [Ox] and [Red] denote the oxidized and reduced forms of a redox couple. Water can act as an oxidant under digestion conditions. For example, water can react with certain aldehydes to yield the corresponding carboxylic acid and hydrogen as products (Loh et al., 2010, Costine et al., 2010a, Costine et al., 2010b), as follows:



This overall reaction may be written as two half-reactions involving electron transfer, which also emphasises the base-catalysed nature of the reaction:



In this case,  $\frac{1}{2}$  a mole of hydrogen is produced in each half-reaction, one from the oxidation of the organic molecule and the other from the reduction of water. Similar reactions may result in the production of hydrocarbons, alcohols or carbonyls in place of hydrogen as the reduction products of the degradation of organic compounds. This suggests that the electrochemical potential of the solution in digestion is controlled by the organic degradation processes that occur there, and that it is sufficiently negative for water to be reduced to hydrogen and hydroxyl ions. *This observation has important implications for the oxidation state of metal ions and complexes in solution, and for the types of redox reactions which are possible in the solution.*

Of all the transition metal ions that are known or likely to be present, the only one that appears to have been studied is mercury (Mullett et al., 2007). The behaviour of mercury in the Bayer process has been investigated because of its importance as an environmental pollutant (Mullett et al., 2007). The observation that  $\text{Hg}^0$  is typically present in digestion vapours reveals that the electrochemical potential in digestion must be sufficiently negative for  $\text{Hg}^0$  to be the stable oxidation state. The Pourbaix diagram for mercury at room temperature (Mullett et al., 2007, Takeno, 2005) indicates that, at pH 14.4 (corresponding to the pH in a Bayer digester), the  $\text{Hg}^0/\text{Hg}^{\text{I}}$  equilibrium is close to the  $\text{H}_2/\text{H}_2\text{O}$  equilibrium. The observation that digestion vapours generally contain significant amounts of  $\text{H}_2$  as well as traces of  $\text{Hg}^0$  suggests that this relationship also holds at digestion temperatures, consistent with the proposed role of water as an oxidant in digestion (Loh et al., 2010). This has implications for the speciation of metal ions, most of which are expected on thermodynamic grounds to be present in their reduced forms during digestion. This is illustrated schematically in Figure 1, which shows the speciation at pH 14 of some of the metal ions known to be present in BLs. Note that the ferric ion is not a strong oxidant at pH 14, due to the effect of complexation by  $\text{OH}^-$ . This is an example of the profound effect that complexation can have on the electrochemical behaviour of ions. Complexation can be expected to play a major role in the behaviour of metal ions in BLs due to the large number and variety of organic compounds present.

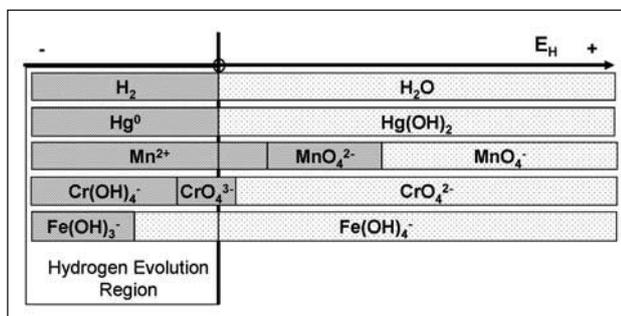


Figure 1: Schematic representation of the speciation of several metal ions under digestion conditions as a function of  $E_H$  at pH 14, constructed from room temperature  $E_H$ /pH diagrams (Takeno, 2005). Pending additional data, it may be assumed that the relativities will broadly hold at higher temperatures.

After digestion, the liquor is cooled to enable recovery of  $Al(OH)_3$  by seeded precipitation (Pearson, 1955). After precipitation the Bayer spent liquor (BSL) is reheated and fed back to digestion. The BSL contains a wide variety of organic compounds, the majority of which are carboxylic acids with molecular weights <300 Da (Power and Loh, 2010).

The  $E_H$  is likely to be higher in BSL than it is in the digester, because:

- $E_H^\circ$  increases linearly as temperature decreases;
- $E_H^\circ = -(RT/F) \cdot pH$ ;
- The redox reactions which occur in digestion produce a liquor containing predominantly products of oxidation (the reduction products having partitioned to the vapour phase);
- Oxidation reactions of the most reactive organic compounds leached from the bauxite and which form mixed potentials (Helburn and MacCarthy, 1994) in digestion are either complete or are much slower at the lower temperatures; and
- The liquor has been exposed to air, particularly in plants using air agitated precipitators.

The organic compounds present are therefore expected to be relatively stable in spent liquor, and the rise in  $E_H$  allows the possibility of changes in the oxidation states of the metal ions and complexes present. For example, mercury may be then present as  $Hg^I$  or  $Hg^{II}$  rather than as  $Hg^0$ , particularly if the liquor has been aerated (Mullett et al., 2007).

A further rise in potential can be expected for liquor that reports to residue storage, due to the combination of lower temperatures and exposure to air and light. From these considerations, the changes in  $E_H$  through the Bayer process cycle may be conceptualised as shown schematically in Figure 2.

To date no measurements specifically focussed on the  $E_H$  of Bayer liquors have been reported to our knowledge. This paper examines some methods for carrying out such measurements, with a view to establishing the means to construct accurate  $E_H$  maps for individual refineries, along the lines of Figure 2. In particular, the use of ferricyanide as a titrant and mediator probe to facilitate  $E_H$  measurements in Bayer process liquors is demonstrated.

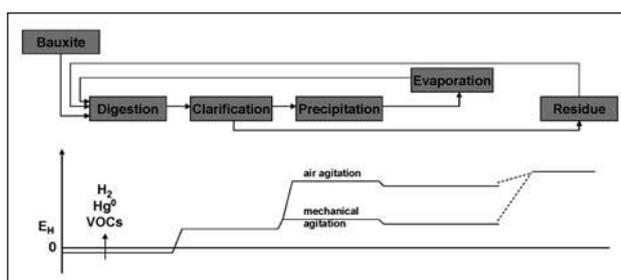


Figure 2: Schematic representation of likely correlation between liquor  $E_H$  and unit process in the Bayer circuit.

This preliminary study examines a BSL at temperatures up to 90 °C. Future work will extend the measurements to other Bayer liquors, and to temperatures typical of Bayer digestion (up to at least 220 °C) using a specially designed electrochemical cell.

The choice of methods, materials and cell configurations for the determination of electrochemical parameters in BLs is limited by the very high alkalinity and concentrations of salts and organic compounds in solution, and the need for measurements at high temperatures. In addition, the complex mixture of organic compounds present results in effects such as adsorption to electrode surfaces which may dominate the observed potentials so that they do not reflect the solution  $E_H$ . These factors limit the number of suitable electrode materials. For example, gold presents difficulties due to the formation of surface oxy-hydroxide layers in alkaline solutions, and carbon electrodes are problematic due to adsorption effects and their reactivity at moderate potentials in strong alkali. No electrode material is immune from these effects. Pt was chosen as the best compromise because of its relative unreactivity in strong alkali, but surface adsorption and oxide formation effects must nevertheless be taken into account.

The main electrochemical methods which were considered for application to  $E_H$  measurements in BLs are summarised in Table 1. A combination of direct potentiometry, cyclic voltammetry, titration and mediator methods was found to be best for determining solution potentials. Pulse and AC methods may be useful in future studies of the kinetics of reactions.

**Table 1: Summary of methods considered for the determination of  $E_H$  in Bayer process liquors**

Method	Advantages	Disadvantages
Direct potentiometry	Simplicity	Uncertain interpretation in presence of irreversible electrode reactions, surface poisoning by adsorption of organics, etc
Cyclic voltammetry	Accurate determination of reversible potentials Information on electrode kinetics	Difficult to distinguish individual redox couples in complex mixtures Subject to surface adsorption effects Requires significant concentrations of electroactive species
Pulse and AC methods	Information on electrode potentials, kinetics and mechanisms	Complex interpretation Requires specialist equipment and techniques
Mediator systems	Detection enhanced by presence of reversible redox couple	Requires mediator which is chemically stable and electrochemically reversible in the required environment
Redox titration	Extends mediator method to provide additional information on nature and kinetics of reactions	More complex than simple mediator Difficult to apply to systems with slow reaction or electron transfer kinetics
Rotating disc voltammetry	Allows mass transport effects to be identified and measured	Requires specialist equipment – difficult to apply in pressure vessels (i.e. to work above atmospheric boiling point)

## 2. Experimental

Sodium hydroxide solutions were prepared from AR sodium hydroxide pellets (>97 % NaOH, <1 %  $Na_2CO_3$ , supplied by Rowe Scientific) and deionised water, and were used without further purification. The Bayer spent liquor was taken from a bulk sample of liquor from an Australian alumina refinery. Potassium ferricyanide solution was prepared from 99 % A.C.S reagent supplied by Sigma Aldrich, and was made up as a 0.1 M stock solution in 4 M NaOH.

Electrochemical measurements were made using a Metrohm Autolab type PGSTAT302N potentiostat. The electrochemical cell was a Metrohm 100 mL glass vessel with water jacket, modified to accommodate a rotating disc electrode and Luggin capillary. The working electrode was a 4.0 mm diameter Pt disc which was mechanically polished to a mirror finish and washed in deionised water prior to use, and the counter electrode was a Pt coil. The reference electrode was an Ionode Ag/AgCl double-junction electrode, connected to the main cell via a Luggin capillary. The capillary tip was positioned within 3 mm of the Pt surface to minimise solution resistance ( $iR$ ) effects. The double junction reference electrode was used to eliminate the possibility of contamination of the Ag/AgCl system, and to minimise the liquid junction potential, which was estimated by comparing the reversible potentials of the ferri/ferrocyanide couple in KCl and NaOH. The reference electrode was maintained at room temperature throughout and was checked before and after each experiment against a standard calomel electrode.

The rotating disc was controlled by a Pine Instruments Digital RDE Controller mounted in a Pine Instruments RDE Stand. Temperature control to  $\pm 0.1$  °C was achieved using a Jubalo water bath with digital controller, and solutions were de-aerated where required by purging with high purity argon for at least 30 minutes.

### 3. Results and discussion

#### 3.1 System validation and background data

A set of preliminary experiments was carried out in the absence of BSL to ensure correct operation of the electrochemical system and to provide background data that could be directly compared with measurement in the presence of BSL. In particular, the behaviour of ferri/ferrocyanide in NaOH solution was investigated to assess its suitability as an indicator and/or titrant at high pH and temperature.

##### 3.1.1 Cyclic voltammetry of Pt in 4M NaOH

Figure 3 shows the cyclic voltammetric response of 4M NaOH at room temperature (22 °C). This concentration was chosen because it approximates the NaOH concentration generally found in BSL. The peaks corresponding to the adsorption and desorption of hydrogen at the electrode surface are evident. The other main features are the decomposition of water to produce hydrogen at potentials below  $-1.1$  V<sup>1</sup> and a broad plateau leading to oxygen evolution at positive potentials.

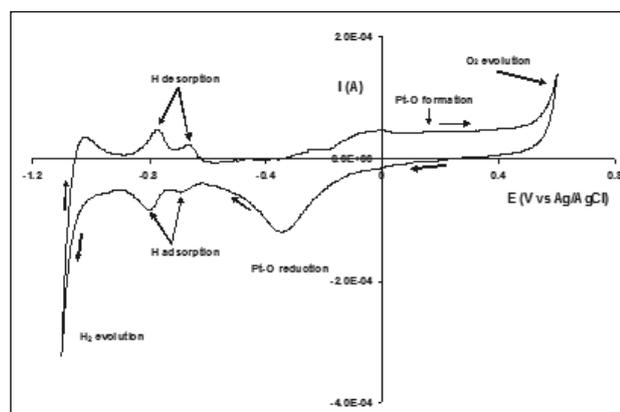


Figure 3: Cyclic voltammogram of 4M NaOH at 25 °C on a Pt disc electrode rotating at 300 rpm.

<> Note: All potentials denoted "E" are potentials measured with respect to a Ag/AgCl reference electrode at room temperature. Potentials denoted "E<sub>H</sub>" are the equivalent potentials relative to a standard hydrogen electrode at the pH and temperature of the solution.

#### 3.1.2 The ferri/ferrocyanide system in 4M NaOH

According to published  $E_{1/2}$ /pH diagrams, ferricyanide is thermodynamically unstable at pH 14 (Takeno, 2005). It was therefore necessary to establish whether or not it is sufficiently kinetically stable to be useful under the conditions required here (i.e. pH 14.4, up to T 90 °C).

Cyclic voltammetry was performed on the ferri/ferricyanide couple in 1 M KCl and 4 M NaOH in the temperature range 25 to 90 °C to check the stability of the complex and to estimate the liquid junction potential for the NaOH solution<sup>9</sup>. Figure 4 shows the cyclic voltammogram at a sweep rate of 100 mV/s at 25 °C, compared with the theoretical curve calculated DigiElch 6 software with literature values for the diffusion coefficients and kinematic viscosity (Opekar and Beran, 1997, Bourne et al., 1985). Apart from a negative offset that can be attributed to the reduction of a surface oxide on the Pt, the experimental data show excellent agreement with the simulated curve. Similar agreement was found at 55 °C, while at 90 °C the offset was somewhat greater although the overall curve shape was preserved.

The values of the charge-transfer rate constant and reversible potential in 4M NaOH at 25 °C were 0.01 cm/s and  $+0.303$  V(Ag/AgCl), respectively. The reversible potential in 1M KCl was  $+0.272$  V(Ag/AgCl), in close agreement with literature values (Bott and Jackson, 1996). The liquid junction potential was therefore  $+31$  mV at 25 °C, and showed an inverse linear relationship with temperature (temperature coefficient =  $-0.91$  mV/°C).

The chemical stability of the ferricyanide was checked by determining the solution concentration from limiting current measurements at a rotating disc electrode. The complex was found to be stable over a period of several hours at 90 °C, but anomalies were noticed with solutions that had been left to stand for more than a day. It is therefore necessary to make up fresh solutions each day.

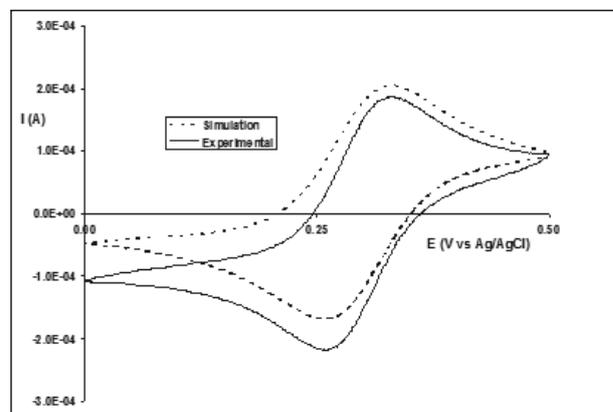


Figure 4: Cyclic voltammetry of 10 mM ferricyanide in 4M NaOH at 25 °C, at a sweep rate of 100 mV/s at a stationary Pt disc electrode. The dashed curve is a steady-state simulation and the full curve shows the experimental results.

#### 3.2 Electrochemistry of Bayer Spent Liquor

The electrochemical characteristics of a sample of BSL were investigated by a variety of techniques. Cyclic voltammetry provides an overview of the electrochemical properties of the solution, and more detail is revealed by potentiometry and potentiometric titration.

##### 3.2.1 Cyclic Voltammetry

The cyclic voltammogram of BSL at 22 °C is shown in Figure 5. The main distinguishing features in relation to pure NaOH under the same conditions (Figure 3) are:

- Hydrogen adsorption is suppressed, as evidenced by flattening and merging of the adsorption and desorption peaks;

- Hydrogen evolution is suppressed and shifted to more negative potentials;
- The Pt-O formation plateau is less well defined;
- A broad oxidation peak appears with a maximum in the vicinity of +0.3 V;
- A bulk oxidation process commences prior to oxygen evolution; and
- The Pt-O reduction peak is smaller.

At 90 °C the oxidation of the bulk organic compounds in BSL is accelerated, as shown by the rapid rise in oxidation current above +0.4 V (Figure 6). The separate oxidation peak at +0.3 V observed in BSL at 22 °C is less prominent at 90 °C, appearing as a barely discernible shoulder on the main peak. At the negative end of the scale, hydrogen evolution is shifted to more negative potentials in BSL compared to NaOH.

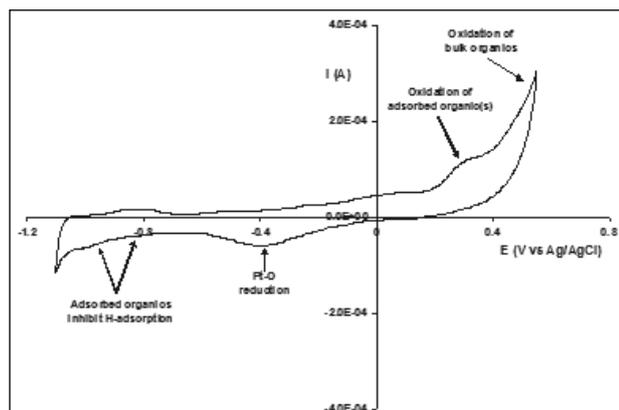


Figure 5: Cyclic voltammogram of 100 % BSL on a Pt electrode at 22 °C, 300 rpm.

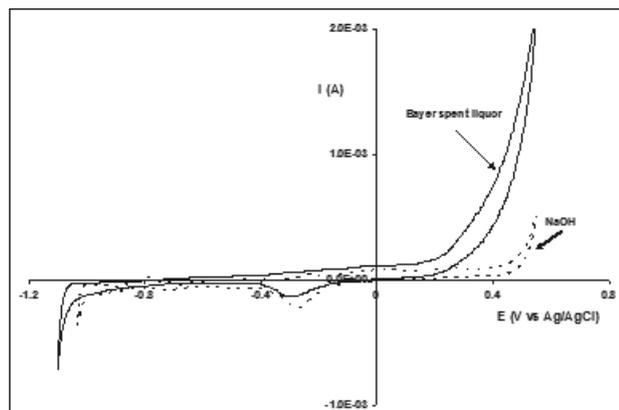


Figure 6: Cyclic voltammogram of 100 % BSL compared with 4M NaOH on a Pt electrode at 90 °C, 300 rpm.

### 3.2.2 Potentiometry, Mediation and Titration

Determination of the solution potential of BSL by simple potentiometry using platinum, gold and carbon electrodes was attempted, but in all cases the potentials were irreproducible and subject to continuous drift. This indicates an absence of reversible couples able to exchange electrons with the Pt surface, which therefore exhibits an ill-defined, time-dependent potential. The addition of a small amount of ferricyanide (0.1 mL of 100 mM solution) had little effect, the ferricyanide simply being reduced to ferrocyanide without materially affecting the disc potential. Adding a larger aliquot (1 mL) of ferricyanide caused the potential to spike upwards rapidly but then to drop below the starting point (at first sight surprising since ferricyanide is an oxidant). This behaviour repeats with subsequent additions of ferricyanide, after which a relatively steady potential is reached, independent of the amount of ferricyanide added.

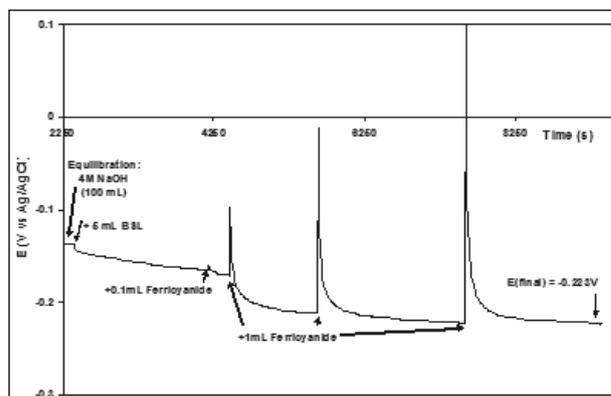


Figure 7: Effect of adding aliquors of 100 mM ferricyanide to 5% BSL in 4 M NaOH at 90 °C. Potential measured at a Pt disc rotating at 1000 rpm.

This effect may be understood in terms of the Nernst equation:

$$E = E^{\circ} - \frac{RT}{F} \ln \left( \frac{[\text{Fe}(\text{CN})_6^{4-}]}{[\text{Fe}(\text{CN})_6^{3-}]} \right) \quad (5)$$

The measured potential,  $E$ , is determined by the  $[\text{Fe}(\text{CN})_6^{4-}]/[\text{Fe}(\text{CN})_6^{3-}]$  ratio, which is itself determined by the reducing ability of the BSL solution. The potential is most sensitive to the very small amount of residual ferricyanide, which sufficient must be present to facilitate a suitable electrode response (in this experiment 0.1 mL is not sufficient). The initial addition causes a sharp increase in potential as the electrode responds to the added ferricyanide ions, and then decays as the ferricyanide is reduced until a potential is reached at which no further reaction is possible. At this point the solution potential is buffered at a potential characteristic of the reducing power of the BSL. Another way of looking at this is that the ferri/ferrocyanide couple is acting as a mediator (Li et al., 2010) between the reductant in solution and the platinum surface. In this way, **the addition of sub-stoichiometric amounts of ferricyanide provides a means of measuring the characteristic electrochemical potentials of Bayer process liquors.**

The measured BSL buffering potential in this case is approximately -0.22 V. This is well positive of the hydrogen evolution region according to Figure 6, consistent with BSL being stable with respect to oxidation by water at this temperature. Higher temperatures and the introduction of organic reducing agents during bauxite digestion can be expected to result in potential which are more negative. Such organic reducing agents include degradation products of sugars and lignins that contain multiple hydroxyl groups (Power and Loh, 2010). As an example, catechol (which has two adjacent hydroxyl groups on a benzene ring) exhibits a buffering potential of approximately -0.35 V in 4 M NaOH at 90 °C. These observations are consistent with the hypothesis of water acting as an oxidant towards organic compounds during bauxite digestion at elevated temperatures (i.e. >140 °C), but this has yet to be demonstrated by measurement.

If ferricyanide additions are continued to the point that all of the oxidisable organic compounds have been consumed, the potential rises rapidly to form the well-known sigmoidal shape of a classical titration curve. At the top of this curve it is the ferri/ferrocyanide couple that buffers the potential. The result of such a potentiometric titration, which enables the concentration of oxidisable organics to be determined, is shown Figure 8.

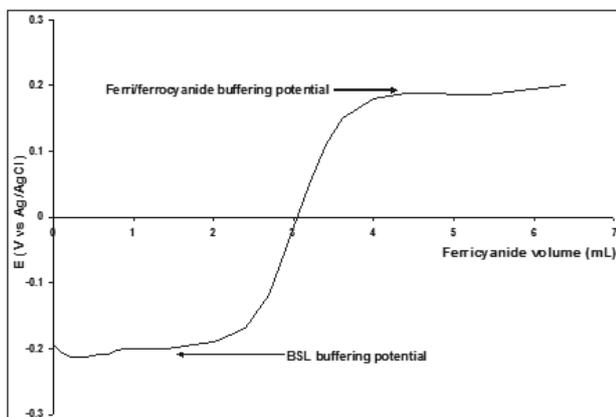


Figure 8: Potentiometric curve for the titration of 100 mL of 5 % BSL in 4 M NaOH with 100 mM ferricyanide at a Pt disc electrode rotating at 1000 rpm.

#### 4. Conclusions

The general electrochemical characteristics of a sample of BSL have been described. Adsorption of organic species from BSL were found to deactivate a Pt electrode in relation to hydrogen evolution. At positive potentials there is evidence of oxidation of surface-adsorbed and dissolved organic compounds, which indicates that removal of organic compounds by anodic oxidation is feasible in principle.

The BSL was found to contain no discernable redox couples, so that direct measurement of a well-defined solution potential was not possible. However, an indirect method for measuring electrochemical potentials in Bayer process liquors has been demonstrated using ferri/ferrocyanide, which has been shown to be suitable for use as a redox mediator and titrant in 4M NaOH up to at least 90 °C. The electrochemical potential for BSL at 90 °C was measured to be -0.22 V with respect to the Ag/AgCl reference. The method can now be applied to extend the investigations to enable mapping of the potential profile for liquor as it proceeds around the Bayer process circuit. Measurements of potentials in the presence of organic compounds from freshly digested bauxite at temperatures up to 220 °C will be possible using a specially designed electrochemical autoclave cell developed at CSIRO.

#### 5. Acknowledgements

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