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CONFIDENTIAL

## DEVELOPMENT OF A BIPOLAR CELL FOR LITHIUM PRODUCTION

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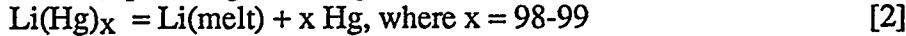
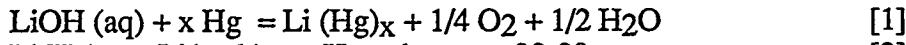
We report development and bench-scale testing of an electrolytic process for reduction of LiOH to lithium metal through an amalgam intermediate. The amalgam is formed in an aqueous-electrolyte cell and stripped in a molten salt cell using a LiI-CsI eutectic at 225 C. Total energy efficiency is >70%. The process obviates high temperature materials problems, chlorine evolution and anhydrous feedstocks. While the principle is proven, sustained operation of the cell is now needed to obtain statistical data on reliability and maintainability.

### INTRODUCTION

Lithium metal is produced industrially by the electrolysis of a fused salt consisting of LiCl and KCl at about 450 C. The process has several shortcomings. The required feedstock (purified, anhydrous LiCl) is expensive and difficult to handle. The evolution of molten lithium and chlorine gas in close proximity presents a potential explosion hazard. The elevated temperatures lead to problems of materials stability and to contamination of the lithium product with cell degradation products. The energy efficiency of the electrolysis process is low (~50%). These problems motivated us to develop an alternative process of increased safety and lower cost (1,2).

### TECHNICAL APPROACH

We have developed and tested on the bench scale a process which uses electrolysis to reduce aqueous solutions of various lithium compounds to metal through the intermediate formation of a 2-3 at.-% Li amalgam (Figure 1). The amalgam is produced in an aqueous cell at 30-40 C, and then refined across a LiI-CsI eutectic (melting point, 218 C) at 225 C. The amalgam circulates continuously between the two cells with a counter current exchange of heat between the streams exiting and entering the higher temperature cell. The aqueous cell and molten salt cell reactions are, respectively:



Water may be reduced at the amalgam, consuming generally less than 1% of the current:



In principle, the amalgam can be formed from any soluble lithium compound, of which LiOH and Li<sub>2</sub>CO<sub>3</sub> are the most likely industrial feedstocks. The amalgam is a single electrode which separates two cells and sustains a reduction reaction [1] at one electrolyte interface and an oxidation reaction [2] at the other interface, with an internal transfer of current. Such electrodes are called "bipolar." Amalgam electrodes have been used to purify lithium compounds and to separate the two isotopes of lithium (3).

The mercury is encapsulated in this process by an impervious molten salt or aqueous solution. The presence of trace amounts of lithium (ca. 100 ppm) protects the mercury and containment vessels from oxidation, by lowering the electrode potential of the amalgam to below -2 V vs. Hg/HgO (4).

Benefits sought from this process include (1) reduced materials stability problems through lower temperature operation; (2) scale-down potential for specialty applications; (3) use of aqueous lithium solutions as feedstock; and (4) elimination of evolution of chlorine and associated hazards. The objectives of the development project were to establish proof of principle through (1) identifying a suitable molten salt composition; (2) measurement of process efficiencies; (3) and demonstration of stable operation on the bench scale (30 A).

## APPARATUS

### Aqueous subsystem

The aqueous cell uses a 100-cm<sup>2</sup> ring-shaped amalgam cathode ("race track" configuration), through which amalgam is flowed in one direction between two ports in the floor of the nickel-plated stainless steel track. The anode is a nickel-plated 304 stainless steel electrode. Electrolyte flows outwards between the closely spaced electrodes at right angles to the track. This imparts a spiraling flow profile to the amalgam.

The cell is constructed of Teflon (PTFE) and has an acrylic lid allowing observations. The electrolyte temperature is controlled in an external portion of the flow circuit in a concentric tube heat exchanger. The pump provides flows of 20-60 ml/s, giving an average linear velocity of 1-3 cm/s above the amalgam cathode (Re ~ 600, based on 1 cm separation and 3 cm/s). The gas volume above the electrolyte is minimized and is slowly purged with nitrogen or argon to avoid accumulation of hydrogen.

### Molten salt subsystem

The molten salt cell consists of a race track amalgam anode of the size and shape of the aqueous cell cathode. The track is machined into a 316 stainless steel cell body. (Figure 3) The anode is a 316 stainless steel lid of the cell. The lid and body are separated by a boron nitride (BN) gasket sealed with a high-temperature silicone cement. The gasket supports a BN skirt which prevents shorting of the lithium to the cell body. The cell is thermally insulated and is externally heated and controlled at a temperature of 225 C.

Our cell contains a small isolated cathode chamber. This chamber can be used as a lithium metal reference electrode, or (in the work reported here) as a subscale cathode.

Prior to electrolysis, the LiI-CsI eutectic is externally melted and transferred to the argon-filled cell. This hot transfer partially vaporizes any mercury present in the cell and will unavoidably contaminate the first production of lithium. The molten salt subsystem also has provisions for argon displacement of lithium melt from the cell, and for automatic transfer of amalgam to (or from) the cell from an external reservoir. In this bench unit, active thermal control is used throughout. Industrial units would be air-cooled and would rely on waste process heat to maintain molten salt operating temperatures.

### Amalgam transfer subsystem

The cells share a common amalgam electrode. This bipolar electrode provides both thermal and chemical isolation of the molten salt and the aqueous electrolytes. The thermal isolation is effected by a counter current exchange of heat between amalgam streams entering and leaving the high temperature cell in two concentric tube units. Part of the lower-temperature heat exchanger is rigorously controlled at 110 C, which allows any (inadvertently) entrained water to be evaporated and removed from the cell through a vent on the stream leaving the aqueous cell.

We rely on the wetting of nickel by mercury to prevent entrainment of electrolyte into the amalgam stream exiting the aqueous cell. Before loading amalgam into the nickel-plated race track of the aqueous cell, the nickel is polarized to reduce any nickel oxide surface layers. After a few minutes of cathodic polarization in LiOH solution, a single drop of mercury will rapidly spread over surface to form a permanent seal. Without this procedure, a film of water creeping over the nickel surfaces will be reduced by the lithium amalgam to form LiOH, which draws in yet more water between amalgam and plumbing by osmosis. Eventually, this leads to a clogging of the circuit with solid LiOH.

The hydrostatic level of mercury in all three subsystems is constant. The inert gas is vented through condensers (e.g., stainless steel ball bearings in a water cooled tube) followed by activated charcoal filters. The entire system is supported on a massive 1 cm deep steel catch tray filled with 6 mm ball bearings. The heat capacity of this tray is intended to mitigate any catastrophic breach of the amalgam containment by rapidly lowering the amalgam temperature and vapor pressure.

## EUTECTIC DEVELOPMENT

A molten salt suitable for this process must have thermodynamic stability in the presence of lithium and lithium amalgam, and a melting point somewhat in excess of that of lithium (180 C) to assure a liquid product. Electrolysis in the molten salt cell should result only in lithium deposition or dissolution. The operating temperature of the cell should be as low as possible to avoid excessive mercury vapor pressures.

### Experimental Studies of the LiI-CsI System

No known composition fills all these requirements, but phase equilibrium calculated by Sangster and Pelton (5) for the system LiI-CsI suggested a eutectic at  $216 \pm 50$  C for 2:1 mole ratios of LiI to CsI. This theoretical phase diagram was calculated from excess enthalpy measurements provided by Melnichak and Kleppa (6) using reasonable assumptions about the excess entropy of mixing in the LiI-CsI system (assumed to be zero). Therefore, we chose to determine the melting point and eutectic composition experimentally to determine whether this composition could be used in our process.

The composition  $(\text{LiI})_{0.66} (\text{CsI})_{0.34}$  was prepared in an argon glove box using anhydrous, high purity materials provided by Johnson Matthey (Alfa Aesar; LiI: anhydrous, stock No 44159; CsI, ultra dry, Stock No. 35729). This composition was melted under argon; arrests in the cooling curve were found near 218 C. Further tests on a number of 120 mg specimens using differential thermal analysis (using indium as an internal melting point standard) established that the eutectic melting point is 217-218 C, and the eutectic composition contains 64.2 at.-% LiI.

Inadvertent contamination of the DTA samples with water lowered melting point to as low as 175 C. Repeated DTA cycles under dry argon flow raised the melting temperature to approach that of the anhydrous sample. In the presence of lithium, any trace water initially present in the melt would be react producing  $\text{Li}_2\text{O}$  and hydrogen gas. This should cause no operational problems provided that the oxide content does not precipitate or greatly alter the melting point.

#### Thermodynamic stability of the eutectic

Although cesium is generally base to lithium in halide melts, it is possible that some cesium might be reduced to form a liquid Cs-Li alloy at a sufficiently polarized lithium cathode. Besides contaminating the product, deposition of cesium would progressively alter the melt composition and jeopardize the process. The amounts of Cs and Li in equilibrium with  $\text{LiI-CsI}$  salt mixtures were calculated from the equilibrium constants for the following equation:



Thermodynamic functions for  $\text{Cs(l)}$  and  $\text{Li(l)}$  were taken from the CODATA tables (7) and thermodynamic functions for  $\text{LiI(l)}$  and  $\text{CsI(l)}$  were taken from Pankratz (8). Combining these data and extrapolating the data to lower temperatures for  $\text{CsI(l)}$  and  $\text{LiI(l)}$ , an equilibrium constant can be calculated. These results are given as a function of temperature in Table 1. The excess enthalpy data given by Melnichak and Kleppa (6) can be used to relate the eutectic composition to the activities of the salt constituents. At a composition of  $\text{X}_{\text{LiI}} = 0.66$  and  $\text{X}_{\text{CsI}} = 0.34$ , the activities of the salts at 500 K are calculated to be about  $a_{\text{LiI}} = 0.0407$  and  $a_{\text{CsI}} = 0.0248$ . This assumes a regular solution behavior for the salt system. The composition of the metal is primarily Li. Therefore, the activity of Li is taken to be unity. At 500 K, the activity coefficient at infinite dilution for Cs is calculated from assessed solubility data by Bale (9) to be  $5.7 \times 10^{10}$ .

**Table 1. Equilibrium compositions of Cs in Li in equilibrium with the  $\text{CsI-LiI}$  eutectic salt composition.**

T (K)	$K_{\text{eq}}$	$a_{\text{Cs}}$	$x_{\text{Cs}}$
470	$5.3 \times 10^6$	$1.1 \times 10^{-8}$	$2.0 \times 10^{-19}$
490	$2.5 \times 10^6$	$2.5 \times 10^{-8}$	$4.3 \times 10^{-19}$
510	$1.2 \times 10^6$	$5.0 \times 10^{-8}$	$8.8 \times 10^{-19}$
530	$6.3 \times 10^5$	$9.6 \times 10^{-8}$	$1.7 \times 10^{-18}$

From the data in Table 1, the maximum Cs impurity in the Li product would be  $3.2 \times 10^{-11}$  ppm-wt at 530 C. These calculations also assume that there is very little or no solubility of  $\text{LiI}$  and  $\text{CsI}$  in the Li metal product. The solubility data of  $\text{CsI}$  in Cs given by Bredig, et al. (10) indicate that the  $\text{CsI}$  solubility in  $\text{Cs(l)}$  at 500 K is about  $1.8 \times 10^{-2}$  mol-% which is small but significant. No solubility data for  $\text{LiI}$  in  $\text{Li(l)}$  are available. However, there are solubility data by Bredig, et al.(11) for  $\text{NaI}$  in  $\text{Na(l)}$  which should approximate the amounts expected for  $\text{LiI}$  in  $\text{Li(l)}$ . The data of Bredig, et al.(11) indicate a solubility of  $\text{NaI}$  in  $\text{Na(l)}$  of about  $7.7 \times 10^{-12}$  mole percent at 500 K. Taking the reduced activity  $\text{LiI}$  in the molten salt eutectic, the solubility of  $\text{LiI}$  would be about  $3.1 \times 10^{-12}$  mole percent in the

Li product and the solubility of CsI in the Li product will be much less (probably several orders of magnitude) than  $4.4 \times 10^{-4}$  mol-% which is the CsI solubility in Cs(l) expected for the reduced CsI activity.

### Transport of mercury

The presence of lithium provides cathodic protection against the corrosion of the mercury in the molten salt cell. From considerations of the electrode potential differences between a 1.33 at.-% Li amalgam and Hg/HgCl, the equilibrium concentration of  $\text{Hg}^+$  (or  $\text{Hg}_2^{2+}$ ) in the melt is extraordinarily low. Mercury may still be transported from anode to cathode at the vapor pressure of the operating temperature within any bubbles that might form on the surface of the amalgam by reduction of trace water. Finally, if the salt is initially loaded into the cell containing some mercury, the latter will be partly vaporized and contaminate the cathode current collector and any lithium formed thereafter. Initial contamination would dissipate after prolonged electrolysis.

A potentially serious contamination would result if reduced mercury had an appreciable solubility in the melt, allowing diffusion across the melt as a neutral species. In principle, mercury may diffuse through molten salt as a mono-atomic gas.

## EXPERIMENTAL RESULTS

### Mercury transpiration experiments

Having found no literature data on mercury solubility or diffusion in low temperature halide melts, we conducted preliminary transpiration experiments. The eutectic used (2.4% KCl, 47.8% LiI, 49.8% KI) was an earlier candidate for this process before development of the LiI-CsI formulation). A 15 ml tungsten crucible under argon containing 2 cm deep layer of molten salt was injected with 2 ml of Hg/Li amalgam and then heated to 300 C; after purging with argon for 18 h at a rate of 18 ml/min, the purge gas was diverted to a gas scrubbing tube containing activated charcoal for 3.5 h. The charcoal was leached with dilute nitric acid, followed by ICP/MS determination of total Hg in the charcoal to be 300 ng (above background of 75 ng in the charcoal and < 10 ng in the acid leach). If the collection efficiency were 100% (unlikely!), then the flux of Hg cross the 2 cm melt would be  $1.6 \times 10^{-12}$  g/cm<sup>2</sup>-s, indicating a 70 ppb contamination of lithium when produced at 3 kA/m<sup>2</sup>. While the transport rate of mercury is not likely to be significant, these results should be taken with caution. We did not independently determine an efficiency of the charcoal for mercury capture at such very low levels.

### Aqueous Cell Efficiencies

The electrical and coulombic efficiencies of lithium deposition into a flowing amalgam were determined using the aqueous cell after isolation from the rest of the system. The amalgam (2.5 kg) was circulated continuously between the 100 cm<sup>2</sup> cathode and an external loop at a rate of 1 ml/s. The 4M LiOH electrolyte (3 L volume) was circulated through an external heat exchanger and allowed to impinge upon and flow radially over the 100 cm<sup>2</sup> nickel-plated anode. Coulombic efficiency of lithium deposition (moles of lithium deposited per equivalent of charge-passed) was determined by measuring the total volume of gas  $\Delta V$  (anodic oxygen plus cathodic hydrogen) evolved over a time interval  $\Delta t$  of

electrolysis at constant current, I. The efficiency  $\epsilon$  follows from combining the ideal-gas and Faraday laws:

$$\epsilon = 3/2 - (2F/I)[(P - P_{H_2O})/RT] \Delta V/\Delta t \quad [5]$$

where F and R are the Faraday and ideal gas constants; P is pressure; T is absolute temperature. This technique was calibrated with internal evolution of hydrogen from an auxiliary nickel electrode and found to be accurate to within 0.5%.

We determined efficiencies at candidate operating conditions by continuously depositing lithium into a fixed volume of flowing mercury, allowing lithium concentration to increase up to several atomic percent. The results of one such experiment is given in Table 2. The amalgam concentration is generally in excess of the equilibrium solubility of about 1.5 at.- %. The lithium might be entrained as crystallites of a lithium-mercury intermetallic compound ( $LiHg_3$ ) or present as a metastable solution. Operation at 30 A ( $3 \text{ kA/m}^2$ ) and an amalgam concentration  $[Li] = 3 \text{ at.-\%}$  offers a reasonable balance between cell voltage and coulombic efficiency. Higher-than-equilibrium concentrations of lithium-amalgams formed by electrolysis have been observed by others [3].

**Table 2. Efficiency and cell voltages for lithium deposition at 28-30 C for increasing  $[Li]$  for 2 cm separation of  $100 \text{ cm}^2$  anode and cathode.**

Current A	coulombic efficiency, %	Charge passed, coulombs	$[Li]$ at.- %	Cell voltage V
10.0	99	0	0	3.52
10.0	65.6	22.8	1.96	3.56
15.7	96.4	26.4	2.26	3.95
20.3	99.3	29.9	2.56	4.21
25.3	98.1	31.4	2.70	4.36
29.9	83.5	33.2	3.85	4.43
34.8	68.8	38.4	3.29	4.54
40.1	62.5	40.1	3.44	4.64

#### Integrated System

At this writing, we have conducted the first tests of the integrated system consisting of aqueous cell, molten salt cell, and amalgam transfer subsystems, using a computerized control of critical temperatures. The  $LiI-CsI$  melt was transferred hot into the molten salt cell under continuous argon purge, to form an electrolyte 2 cm thick over a ~1% lithium amalgam (maintained at this concentration by operation of the aqueous cell). The temperature of the cell was controlled at 225 C, the temperature of the vented exchanger was controlled at 110 C; and the temperature of the aqueous cell was controlled at 30 C. The amalgam exchange rate was 0.3 ml/s between the aqueous and molten salt cells. Figure 5 presents the cell voltages of the salt, aqueous and bipolar system. The molten salt cell was operated up to  $4 \text{ kA/m}^2$  without encountering a mass transport limitation indicated by non-linear polarization curves. The process was well behaved and stable over ~10 hours of operation.

## DISCUSSION

Closer spacing of the electrodes in both cells should allow us to achieve 5 V total voltage at 3 kA/m<sup>2</sup> (in the aqueous cell). At this voltage, overall energy efficiency is ~70%. The actual efficiency will evolve from a trade study of energy, manpower and capital costs. The primary economic advantages of this process derive from direct use of a common aqueous feed stock (lithium carbonate or lithium hydroxide), and from the low operating temperature and consequently few materials stability problems.

The highly linear polarization curves for both cells suggests a straight-forward control strategy using two separate power supplies connected to a common grounded amalgam. Since the current in the molten salt cell is carried only by lithium electrolysis, we would monitor depletion of the lithium from the amalgam from a drop in current at fixed cell voltage. (The voltage of the cell would be fixed at a level far below that required for mercury dissolution, and electrode potentials of both lithium and amalgam would be monitored by a captive amalgam reference electrode). Based on the *current* measurement, we would feed back a signal to control the *voltage* applied to the aqueous cell. The charge flowing through the aqueous cell will in general be slightly larger than that flowing through the salt cell, because of the coulombic inefficiency of the aqueous cathode.

There are three potential safety issues that require special attention in the scale-up of the process: water entrainment into the amalgam, mercury loss, and inadvertent mixing of molten lithium and amalgam. The first potential hazard can be avoided by careful wetting of the nickel tray and connecting tubing by the amalgam, but rigorous control of the temperature at a point in the vented heat exchanger is essential.

Our system supports a common mercury level in aqueous cell, molten salt cell and vented heat exchanger, and detectors are installed to monitor the amalgam level and shut down the system following changes which might indicate a clog or a leak.

As part of our program we have determined losses of mercury by evaporation from each of the subsystems (2). The only significant anticipated loss is by evaporation into the hydrogen bubbles formed at the amalgam surface in the aqueous cell. Without filters, this amounts to 0.3 g/day per square meter of amalgam electrode producing 18 kg/day of lithium product. (The EPA allowable loss is 1.38 g/day). This level can be reduced and the mercury recovered using special Hg filters. The worst case leakage would involve a slow leak with continuous evaporation of hot mercury from fine droplets. For catastrophic rupture the cell, a massive porous heat sink filled silicone oil should be provided to quench and encapsulate any spill.

The inadvertent mixing of amalgam and lithium would result in an exothermic reaction of about 23 kcal/mol. It will be necessary to maintain a comfortable separation of 2 cm between electrodes, to minimize the volume of amalgam in the cell to a depth (1-2 mm). Alternatively, we could introduce a physical barrier to mixing such as a porous or woven-fabric yttria separator or vertical baffles.

The greatest barrier to mass transport is the diffusion of lithium into the amalgam near the amalgam/electrolyte interface in the aqueous cell. While not limiting on the bench scale, transport will be an issue with any scale-up design. Empirical knowledge or computer-modeled predictions of amalgam and electrolyte velocity profiles near their common interface will be needed.

## CONCLUSIONS

We have established that lithium can be produced at high energy efficiency (>70%) in a bipolar electrolysis cell through the intermediate production of an amalgam. We have

determined the melting point and composition of a LiI-CsI eutectic having thermodynamic stability with respect to lithium and from which lithium can be deposited. Process control should be straight-forward because of the large time constant and the linear behavior of the system. Mercury emissions through normal operation now meet EPA standards, and can be reduced further to almost any reasonable level through regenerative filters. Catastrophic losses (such as cell or plumbing rupture) can be mitigated through various rapid thermal quenching and liquid encapsulation schemes. The process avoids the far more serious hazards associated with the high temperatures and chlorine evolution of the current industrial process, and obviates the use of expensive anhydrous feedstocks.

The bipolar cell process should produce very pure lithium as might be needed in certain chemical reagents. The process readily scales down, and might be useful in in-plant recovery of lithium scrap from battery manufacture; in recovery of lithium leached from recycled batteries; and in isotopic separation. Most important, its low operating temperature, high energy efficiency and acceptance of primary aqueous feedstocks make it a potential candidate for the next generation of lithium production cells.

The next stage of development of this process is sustained operation on the 30-100 A scale, which will yield statistical data on process reliability and maintainability. This work will no doubt uncover unforeseen failure modes along with ways to mitigate them.

#### ACKNOWLEDGMENTS

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## FIGURES

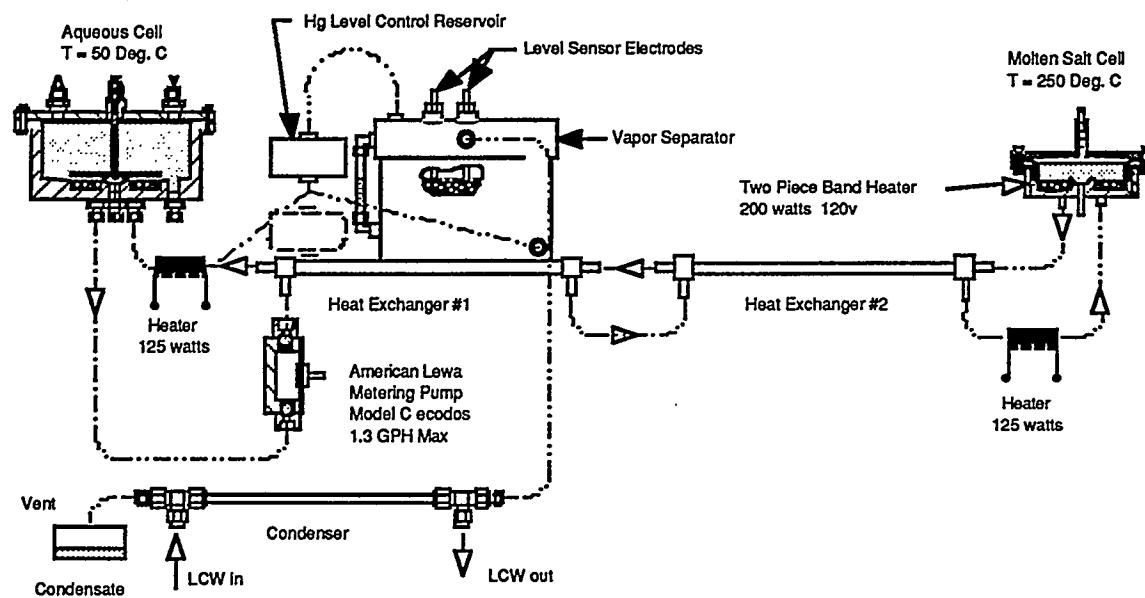


Figure 1. The lithium electrolysis systems consists of an aqueous cell and a molten salt cell sharing a circulating bipolar amalgam electrode. Two counter-current heat exchangers are used on the amalgam circuit, one of which is controlled at 110 C and vented (through a condenser) to prevent inadvertent entrainment of water into the molten salt cell.

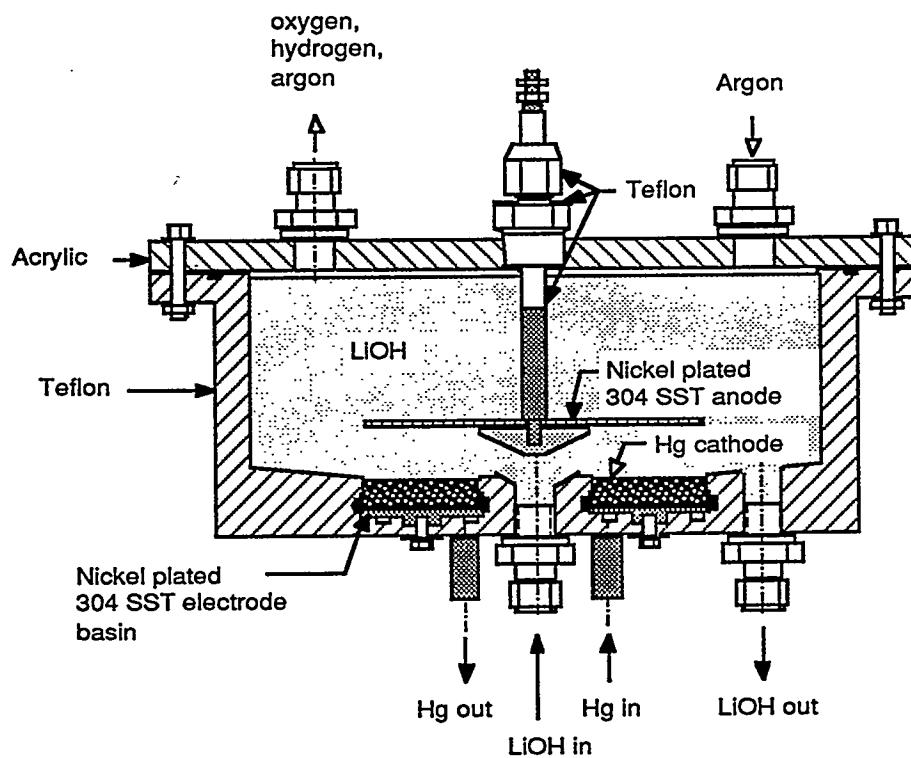


Figure 2. The aqueous cell consists of an oxygen-evolving nickel electrode and an amalgam cathode which circulates around a circular ring depression.

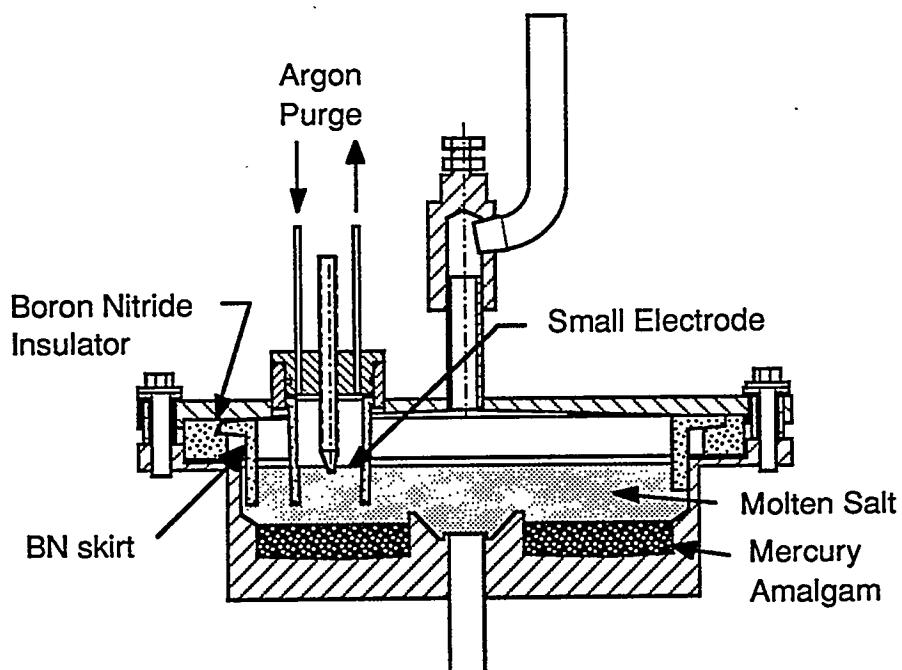


Figure 3. The molten salt cell features a "race track" amalgam anode, and a molten lithium cathode, which floats on the molten salt. To initiate cathodic reduction, a stainless steel pin conductor is polarized.

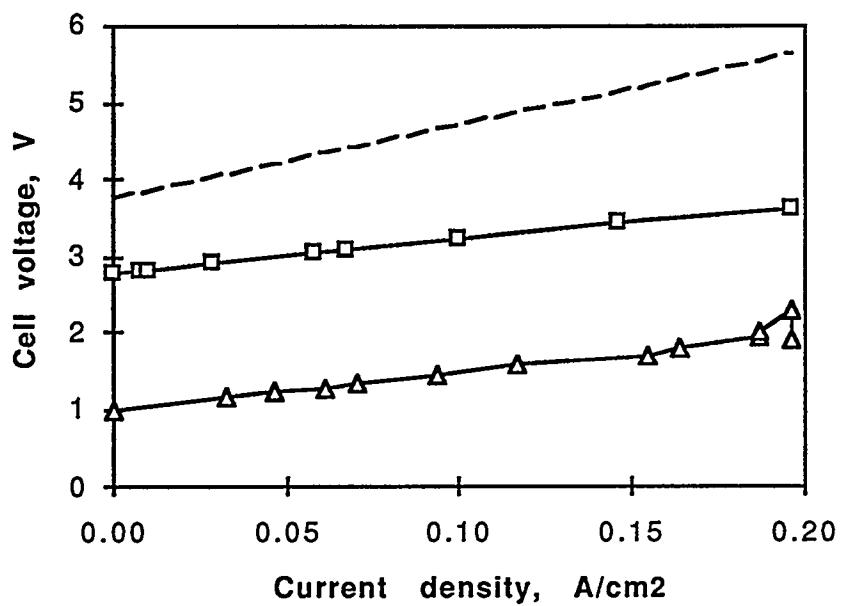
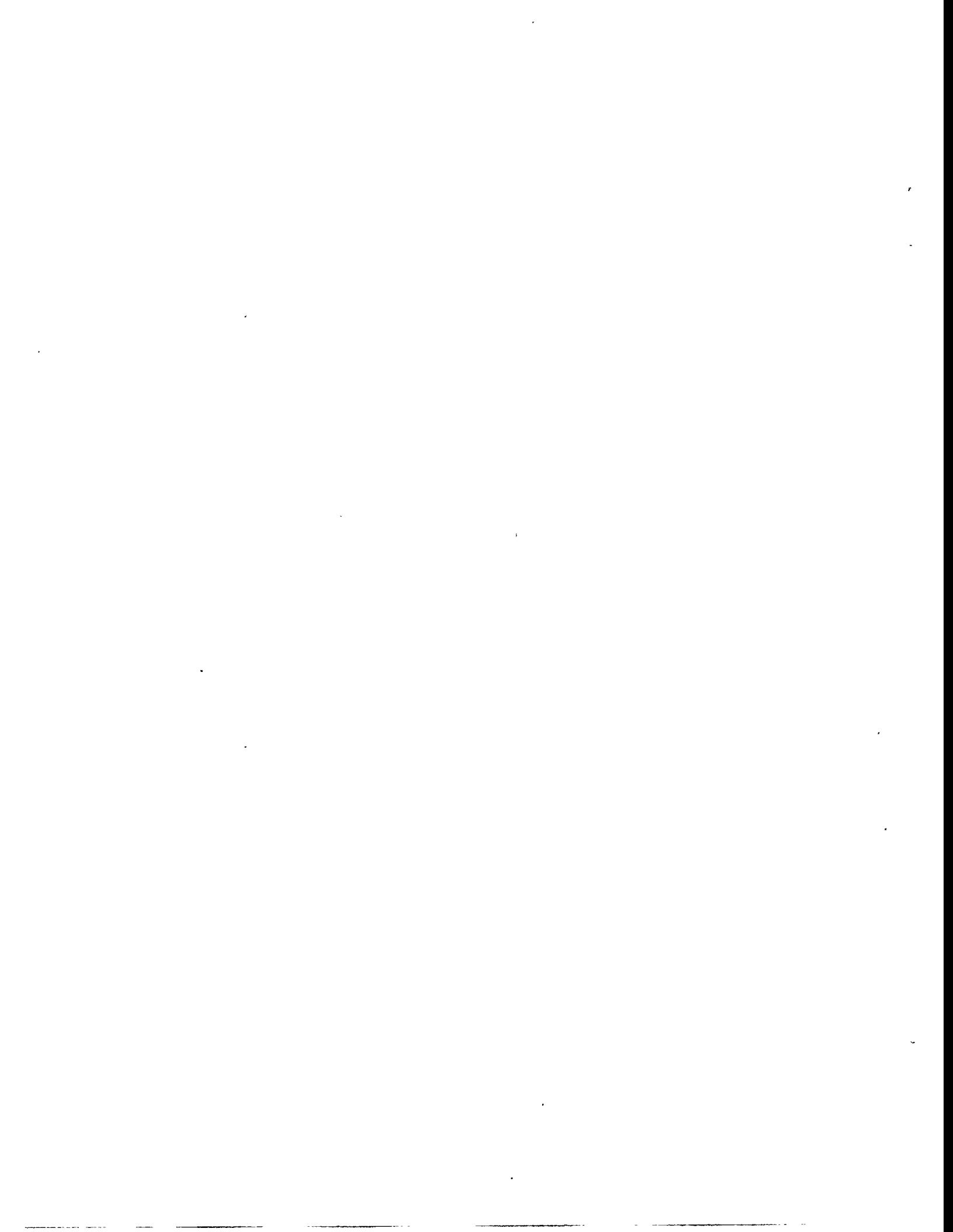


Figure 4. Molten salt cell voltage (at 225 C) , aqueous cell voltage (30-35 C) and the total bipolar cell voltage is plotted against current density.





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