ADVANCED SCREENING OF ELECTRODE COUPLES

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The chromium (Cr^{3+}/Cr^{2+}) redox couple (electrolyte and electrode) was investigated in order to determine its suitability as negative electrode for the iron (Fe^{3+}/Fe^{2+})-chromium (Cr^{3+}/Cr^{2+}) redox flow battery. Literature search and laboratory investigation established that the solubility and stability of aqueous acidic solutions of chromium(III) chloride and chromium(II) chloride are sufficient for redox battery application.

Four categories of electrode materials were tested; namely, metals and metalloid materials (elements and compounds), alloys, plated materials, and Teflon-bonded materials. In all, the relative performance of 26 candidate electrode materials was evaluated on the basis of slow scan rate linear sweep voltammetry in stirred solution. No single material tested gave both acceptable anodic and acceptable cathodic performance. However, the identification of lead as a good cathodic electrocatalyst and gold as a good anodic electrocatalyst led to the invention of the lead/gold combination electrocatalyst. This type of catalyst can be fabricated in several ways and appears to offer the advantages of each metal without the disadvantages associated with their use as single materials. This lead/gold electrocatalyst has subsequently been tested by NASA-Lewis Research Center in complete, flowing, redox batteries comprising a stack of several cells. A large improvement in the battery's coulombic and energy efficiency has been observed.

In addition to the above, five dissolved organic compounds were tested as possible hydrogen evolution inhibitors, including thiourea, palmitic acid, cetyl alcohol, hexanol and n-propanol. Finally, the cost of large lots of thirteen common chemicals was estimated.
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ABSTRACT

The chromium (Cr\(^{3+}/Cr^{2+}\)) redox couple (electrolyte and electrode) was investigated in order to determine its suitability as negative electrode for the iron (Fe\(^{3+}/Fe^{2+}\))-chromium (Cr\(^{3+}/Cr^{2+}\)) redox flow battery. Literature search and laboratory investigation established that the solubility and stability of aqueous acidic solutions of chromium(III) chloride and chromium(II) chloride are sufficient for redox battery application.

Four categories of electrode materials were tested; namely, metals and metalloid materials (elements and compounds), alloys, plated materials, and Teflon-bonded materials. In all, the relative performance of 26 candidate electrode materials was evaluated on the basis of slow scan rate linear sweep voltammetry in stirred solution. No single material tested gave both acceptable anodic and acceptable cathodic performance. However, the identification of lead as a good cathodic electrocatalyst and gold as a good anodic electrocatalyst led to the invention of the lead/gold combination electrocatalyst\(^7\). This type of catalyst can be fabricated in several ways and appears to offer the advantages of each metal without the disadvantages associated with their use as single materials. This lead/gold electrocatalyst has subsequently been tested by NASA-Lewis Research Center in complete, flowing, redox batteries comprising a stack of several cells. A large improvement in the battery's coulombic and energy efficiency has been observed\(^9\).

In addition to the above, five dissolved organic compounds were tested as possible hydrogen evolution inhibitors, including thiourea, palmitic acid, cetyl alcohol, hexanol and n-propanol. Finally, the cost of large lots of thirteen common chemicals was estimated.
I. INTRODUCTION

The iron (Fe$^{3+}$/Fe$^{2+}$)-chromium (Cr$^{3+}$/Cr$^{2+}$) redox flow battery is an attractive system for bulk energy storage applications\(^1\). The primary objective of this program was to contribute to the development of the redox flow battery concept by investigating the major currently recognized or suspected problems of the chromium electrode. These are: the solubility of reactants, the stability of chromium(II) ion in solution, the efficient electrocatalysis of chromium(III) reduction and chromium(II) oxidation, and the cost and availability of chromium electrolyte.

Concentrated solutions of chromium(II) chloride and chromium(III) chloride in hydrochloric acid are required in order to keep redox flow system costs at a reasonably low level\(^2\). Only a moderate amount of effort in determining solubilities was made under a previous contract\(^3\), so that further work was required.

The long term stability of the aqueous chromium(II) ion is of concern because thermodynamics predict its decomposition. This is illustrated by the Pourbaix diagram of Figure I-1 which shows that Cr$^{2+}$ is unstable with respect to oxidation at low pH. The mode of decomposition is given by:

\[
2\text{Cr}^{2+} + 2\text{H}^+ \rightarrow 2\text{Cr}^{3+} + \text{H}_2
\]  

where \(\Delta G^0 = -18.8 \text{ kcal}\).

The driving force for oxidation of Cr$^{2+}$ by dissolved oxygen is, of course, much larger than this value. It is significant that the process represented by equation (1) is reported to be very slow when chemically pure solutions of Cr$^{2+}$ are carefully stored in the absence of air\(^4,8\).

The fundamental reason for the attractiveness of the Cr$^{3+}$/Cr$^{2+}$ electrode is its negative rest potential with respect to the standard hydrogen electrode over the acid pH range of interest (Figure I-1). For the same reason, difficulties were anticipated in finding stable electrocatalyst materials which exhibit good activity for chromium(III) reduction and chromium(II) oxidation, as well as high hydrogen
overvoltage. By \textit{stable} electrocatalysts is meant immunity from corrosion (most electrode materials oxidize near the potential range where the Cr$^{3+}$/Cr$^{2+}$ redox couple operates). \textit{Good} activity means that the equilibrium exchange current density ($i_o$) for the reaction considered is very high, so that activation polarization will not be a significant source of energy inefficiency in the redox flow battery. A \textit{high} hydrogen overvoltage implies a low value of $i_o$ for the hydrogen evolution reaction so that the coulombic efficiency of the battery remains high. For example, gold had been previously shown\textsuperscript{(3)} to have good activity for the Cr$^{3+}$/Cr$^{2+}$ reaction, but its hydrogen overvoltage is low.

The cost and availability of chromium ores as they relate to redox battery operation had been reviewed by Gahn\textsuperscript{(5)}. The information on the cost of large quantities of commonly available chromium chemicals was compiled.

Based on the above information and prior work, five technical tasks were proposed and carried out as described in the following sections.
FIGURE I-1  Thermodynamic Stability of Fe$^{3+}$/Fe$^{2+}$ and Cr$^{3+}$/Cr$^{2+}$ Redox Couples
II. SOLUBILITY AND STABILITY OF AQUEOUS ACIDIC CHROMIUM(II) CHLORIDE SOLUTIONS

A. Introduction

In a prior contract \(^3\) we were unable to prepare a CrCl\(_2\) solution in 1M HCl as concentrated as one molar starting from samples of solid CrCl\(_2\) obtained from several commercial sources. We became convinced that these commercial samples were not genuine CrCl\(_2\). Therefore, we conducted a detailed literature search on CrCl\(_2\) solubility early in the present program to serve as a guide to the experimental work.

B. Results

The literature search uncovered work done by Lux and Illman\(^8\), which indicated that a solution 3M in CrCl\(_2\) and .2M in Cl\(_2\) can be made by dissolving pure chromium metal in 21\% HCl (approximately 6M). These workers reported that chromium(II) ions remain stable for a long time provided air (O\(_2\)) is excluded.

It was deemed desirable to confirm the preparative method and findings of Lux and Illman. To this end, chromium metal obtained from Atochemicals was dissolved in several concentrations of HCl. The amount of chromium(II) ion produced was determined by potentiometric titration with standard Ce(IV) solution. The final HCl concentration was calculated from the initial HCl concentration and the amount of chromium(II) produced. The results presented in Figure II-1 confirm that solutions up to 3M in CrCl\(_2\) could be prepared in up to 2.5M HCl and establish the suitability for redox battery operation.

Our exploratory attempts to measure the solubility of CrCl\(_2\) at higher HCl concentrations showed that the CrCl\(_2\) solubility is only 0.6M in 5.3M HCl.

The stability of chromium(II) chloride solutions was evaluated by comparison of the amount of chromium(II) ion present in freshly prepared solutions with that in aged solutions. The results are given in Table II-1. The small amount of decomposition observed on long standing is probably due to small intrusion of air. The thermodynamically feasible direct oxidation of H\(^+\) by Cr\(^{2+}\) is kinetically inhibited.
In any event, the observed amount of decomposition is small enough to be manageable on the redox flow system level.

Table II-1 also gives the results of tests on the effect of Cd\(^{2+}\) and Pb\(^{2+}\) ions on the decomposition of Cr\(^{2+}\) ions. These experiments indicate no effect of either ions on decomposition of Cr\(^{2+}\) ions. However, in the case of Pb\(^{2+}\) we noticed the presence of lead metal at the bottom of the reaction chamber, consistent with the thermodynamically favored reduction of Pb\(^{2+}\) by Cr\(^{2+}\) (Figure II-3). Since the total concentration of Pb\(^{2+}\) is very small relative to Cr\(^{2+}\), the results of chemical analysis as reported in Table II-1 are unaffected by Pb\(^{2+}\) within experimental precision.

Knowledge gained about the properties of gold and lead electrodes (vide infra) enabled us to devise a suitable voltammetry experiment to determine if PbCl\(_2\) was present in our CrCl\(_2\) solution in greater than trace quantities. A sample of CrCl\(_2\) solution was separated from the precipitated solid material and added to an electrochemical half-cell already containing CrCl\(_3\) in HCl. Cyclic voltammetry of this solution at a gold foil electrode is illustrated in Figure II-2. Curve A exhibits H\(_2\) evolution as expected if no PbCl\(_2\) is present in solution. As a check, the solution was made 10\(^{-3}\) M in PbCl\(_2\) and further sweeps were made. Curves B-1 and B-2 show the development of a limiting current due to Cr\(^{3+}\) reduction in place of H\(^+\) reduction. This is a consequence of the cathodic deposition of lead.
TABLE II-1

Decomposition of Chromium(II) Chloride

<table>
<thead>
<tr>
<th>Original Solution</th>
<th>4 Days Later</th>
<th>5 Days Later</th>
<th>7 Days Later</th>
<th>9 Days Later</th>
<th>25 Days Later</th>
</tr>
</thead>
<tbody>
<tr>
<td>1M HCl + 2.5M CrCl₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2.3</td>
</tr>
<tr>
<td>1M HCl + 2.5M CrCl₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2.25</td>
</tr>
<tr>
<td>1M HCl + 2.35M CrCl₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2.15</td>
</tr>
<tr>
<td>1M HCl + 2.35M CrCl₂ + 10⁻³M CdCl₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2.3</td>
</tr>
<tr>
<td>1M HCl + .4M CrCl₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>.38</td>
</tr>
<tr>
<td>1M HCl + .4M CrCl₂ + 10⁻³M PbCl₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>.38</td>
</tr>
</tbody>
</table>

* IM HCl + 1.8M CrCl₂                            |              |              |              |              | 1.74          |

* IM HCl + 1.8M CrCl₂ + 10⁻³M PbCl₂              |              |              |              |              | 1.74          |

*Estimated.
Figure II-1

Chromous Chloride Solubility vs.
Hydrochloric Acid Concentration

Figure II-2

Gold Foil Electrode

- IN HCl = 0.08M Cr⁺³
- 0.29M Cr⁺²
- (Pb⁺²)

- IN HCl = 0.08M Cr⁺³
- 0.29M Cr⁺² + 10⁻⁶M Pb⁺²

Sweep Rate: 0.05 Volts/min
Figure II-3  Thermodynamic Stability of Pb/Pb$^{2+}$ and Cd/Cd$^{2+}$ Couples.
III. SOLUBILITY AND STABILITY OF AQUEOUS ACIDIC CHROMIUM(III) CHLORIDE SOLUTIONS

A. Introduction

There was general agreement that acidic CrCl₃ solutions are soluble and stable enough for redox battery application. It was of interest, however, to obtain information on the equilibrium solubility of CrCl₃ in various concentrations of hydrochloric acid.

B. Experimental Results

Saturated solutions of CrCl₃ were prepared by dissolving reagent grade chromic chloride hexahydrate in various concentrations of hydrochloric acid and allowing the supernatant solution to remain in contact with excess solid material at room temperature (25°C) for several days. The solution was then separated from solid and analyzed for chromium using titration and spectrophotometric methods. Results of chromium(III) chloride solubility measurements are given in Figure III-1. In the HCl concentration range of these experiments, the solubility of chromium(III) chloride decreases with increasing HCl concentration.
Figure III.1

Chronic Chloride Solubility vs. Hydrochloric Acid Concentration

GCl\textsuperscript{3} Concentration, Molarity

HCl Molarity

0.0  1.0  2.0  4.0  6.0
IV. ELECTRODE MATERIAL EVALUATION

A. Introduction

Our objective in this portion of the program was to acquire enough knowledge about the electrocatalysis of the Cr\textsuperscript{3+}/Cr\textsuperscript{2+} redox couple to enable us to recommend suitable electrocatalysts for full cell testing at NASA-Lewis Research Center. This task was accomplished by screening a number of candidate electrocatalyst materials. The candidates investigated included metals and metalloid materials, alloys, Teflon-bonded materials and materials plated on various substrates.

Suitable electrocatalysts should meet all of the following criteria:

(1) good activity for Cr\textsuperscript{3+} reduction;
(2) good activity for Cr\textsuperscript{2+} oxidation;
(3) high hydrogen overvoltage; and
(4) good resistance to anodic corrosion.

To screen potentially suitable electrocatalysts, we devised a simple test procedure that consisted of selective slow scan rate linear sweep voltammetry. This procedure provided discriminatory information for the above mentioned four criteria. Most of the materials tested were found unsatisfactory on one or more counts. This required obtaining or preparing and testing of a large number of metals, compounds and alloys in bulk form, supported and composite structures.

B. Screening Approach

The screening was conducted in the following steps:

Step 1. The hydrogen evolution reaction of each electrocatalyst was measured by performing sweeps in 1N HCl. A typical sweep begins at some relatively positive potential where only a small residual current is observed and progresses toward more negative potentials where cathodic current due to hydrogen evolution is eventually observed. The current will rise as the sweep is continued, although no limiting current will be achieved. The hydrogen overvoltage of the
different electrodes tested was compared by noting the potential at which the cathodic current first exceeds the arbitrarily chosen value of 1 mA. This cutoff potential was then compared with the open circuit potential of the Cr\(^{3+}/\)Cr\(^{2+}\) redox couple.

**Step 2.** The activity of electrocatalysts for reduction of Cr\(^{3+}\) was determined in an experiment identical with that performed in Step (1), except that the electrolyte was made .1M in CrCl\(_3\). The total Faradaic current observed in these experiments is usually the sum of the currents due to reduction of Cr\(^{3+}\) and the evolution of H\(_2\). In a few cases there is a contribution from an anodic corrosion process. In one case (bismuth) there is a contribution from a cathodic corrosion process. A well defined limiting current due to Cr\(^{3+}\) reduction was observed when the currents due to competing processes were relatively low. Such cases were judged to have fair activity. An example is Ag-Hg (Figure IV-13). If, in addition to the above, the shape of the current-potential curve indicated low polarization, the electrode material was judged to have good activity for Cr\(^{3+}\) reduction. An example is Pb (Figure IV-3). In some cases no limiting current due to Cr\(^{3+}\) reduction was observed. The electrode materials in question were then categorized as not active. An example is B\(_4\)C as shown by Figure IV-17.

**Step 3.** For completeness, anodic corrosion studies in 1M HCl were done on all the materials tested in Steps (1) and (2). The suitability of the candidates screened was evaluated on the basis of the potential at which anodic current first exceeds the arbitrarily chosen value of 1 mA. This cutoff potential was then compared with the open circuit potential of the Cr\(^{3+}/\)Cr\(^{2+}\) redox couple.

**Step 4.** Those materials which still appeared promising after steps (1), (2) and (3), were investigated for their activity towards the oxidation of Cr\(^{2+}\) using a solution 1N in HCl and .09M in Cr\(^{2+}\). All the materials tested in this step showed a well defined limiting current due to Cr\(^{2+}\) oxidation and were, therefore, judged to have good activity.
Step 5. More detailed investigations were undertaken on selected promising electrode materials emerging from the first four screening steps.

C. Results

The identity of each candidate electrode, a cross-reference to Figures IV (see below), its physical characteristics, and a brief summary of our conclusions as to its performance (e.g. good activity but low hydrogen overvoltage and poor corrosion behavior) in the four screening steps considered are presented in Table IV-1.

Results of the voltammetry experiments on which screening was based are presented in Figures IV-1 to IV-44 which also include simplified Pourbaix diagrams. Inserts and/or captions indicate the conditions under which an experiment was run. Each figure is also marked with one or more of the screening code numbers (1, 2, 3, 4 and 5) in order to indicate the type of information shown. These screening code numbers correspond to the screening steps described in detail in Section IV.B. of this report.

D. Recommendations

Based on the first four screening steps, we selected as the most promising electrocatalysts the combination electrodes Ag/Pb, Cu/Pb and Au/Pb. Further investigation (screening Step 5) indicates that a possible problem with Ag is the irreversible formation of an oxide so that the electrode may lose its good performance on repeated cycling. Both the Au/Pb and Cu/Pb electrode were found to have good resistance to anodic corrosion, but Au/Pb is even more resistance than Cu/Pb. Of course, Au is the most expensive material, but it is felt that loadings at microgram levels will be needed to fabricate practical electrodes. Therefore, in spite of a small cost increase, assured reliability warrants the selection of the Au/Pb electrode as most suitable for full cell testing at NASA-Lewis Research Center.

The investigation of different types of Au/Pb catalysts, including layered (sequential deposition of the two metals), codeposited and underpotential plated was begun in this program. Here codeposition is used in its most general sense -- a
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<th>Anodic Performance</th>
<th>Hydrogen Evolution Overvoltage</th>
<th>Corrosion Characteristics</th>
<th>Figure No.</th>
</tr>
</thead>
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<tr>
<td>Ag</td>
<td>Foil</td>
<td>Good</td>
<td>Good</td>
<td>Low</td>
<td>Fair</td>
<td>IV-1, -2, -29</td>
</tr>
<tr>
<td>Ag</td>
<td>Wire (Ingold)</td>
<td>Good</td>
<td>Good</td>
<td>Low</td>
<td>Fair</td>
<td>IV-11, -32</td>
</tr>
<tr>
<td>Au</td>
<td>Foil</td>
<td>Very Good</td>
<td>Very Good</td>
<td>Low</td>
<td>Good</td>
<td>IV-1, -2, -5</td>
</tr>
<tr>
<td>Bi</td>
<td>Rod, wrapped with Teflon tape to expose end (Ventron)</td>
<td>Good</td>
<td>High</td>
<td>Fair</td>
<td>IV-7, -8</td>
<td></td>
</tr>
<tr>
<td>C (vitreous carbon)</td>
<td>Roughened, rotating disk electrode tip (Beckman)</td>
<td>Fair</td>
<td>Medium</td>
<td>Good</td>
<td>IV-38, -39</td>
<td></td>
</tr>
<tr>
<td>C (graphite)</td>
<td>(Ventron) rod, wrapped with Teflon tape to expose end</td>
<td>Poor</td>
<td>Medium</td>
<td>Good</td>
<td>IV-11, -34</td>
<td></td>
</tr>
<tr>
<td>C (graphite)</td>
<td>(Ultra Carbon) rod, wrapped with Teflon tape to expose end</td>
<td>Fair to Good</td>
<td>Poor</td>
<td>Medium</td>
<td>Good</td>
<td>IV-11, -34</td>
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<tr>
<td>Cd</td>
<td>(Alfa) Foil</td>
<td>Good</td>
<td>High</td>
<td>Very Poor</td>
<td>IV-1, -2, -9</td>
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</tr>
<tr>
<td>Cu</td>
<td>Foil</td>
<td>Good</td>
<td>Low</td>
<td>Fair</td>
<td>IV-1, -2, -4, -11, -14, -30</td>
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</tr>
<tr>
<td>In</td>
<td>(Alfa) Foil</td>
<td>Good</td>
<td>High</td>
<td>Very Poor</td>
<td>IV-1, -2, -10</td>
<td></td>
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<tr>
<td>Electrode</td>
<td>Physical Characteristics</td>
<td>Cathodic Performance</td>
<td>Anodic Performance</td>
<td>Hydrogen Evolution Overvoltage</td>
<td>Corrosion Characteristics</td>
<td>Figure No.</td>
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<td>--------------------------------</td>
<td>--------------------------</td>
<td>------------</td>
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<tr>
<td>Pb</td>
<td>(Fisher) Foil</td>
<td>Very Good</td>
<td>High</td>
<td>Poor</td>
<td></td>
<td>IV-1, -2, -3, -14</td>
</tr>
<tr>
<td>Ch</td>
<td>(Alfa) Supported Foil</td>
<td>Fair</td>
<td>Medium</td>
<td>Fair</td>
<td></td>
<td>IV-6</td>
</tr>
<tr>
<td>SiC</td>
<td>(Ultra Carbon) Rod, wrapped with Teflon tape to expose end only</td>
<td>Poor to Fair</td>
<td>High</td>
<td></td>
<td></td>
<td>IV-16</td>
</tr>
<tr>
<td>Sn</td>
<td>(Ventron) Foil</td>
<td>Fair</td>
<td>Medium</td>
<td></td>
<td></td>
<td>IV-1, -2</td>
</tr>
<tr>
<td>Ti</td>
<td>Mesh</td>
<td>Not active</td>
<td>Low</td>
<td></td>
<td></td>
<td>IV-1</td>
</tr>
<tr>
<td>W</td>
<td>Wire</td>
<td>Not active</td>
<td>Very Low</td>
<td></td>
<td></td>
<td>IV-1</td>
</tr>
<tr>
<td>WO</td>
<td>Wire, heated in flame</td>
<td>Not active</td>
<td>Very Low</td>
<td></td>
<td></td>
<td>IV-1</td>
</tr>
<tr>
<td>Ag/Hg</td>
<td>Etched Ag Foil dipped in Hg</td>
<td>Fair (high polarization)</td>
<td>Very High</td>
<td></td>
<td></td>
<td>IV-12, -13</td>
</tr>
<tr>
<td>Cu/Hg</td>
<td>Cu Foil left in Hg</td>
<td>Fair (high polarization)</td>
<td>Very High</td>
<td>Fair</td>
<td></td>
<td>IV-12, -13</td>
</tr>
<tr>
<td>Cu/Hg</td>
<td>Etched Cu Exmet dipped in 2% Hg(NO₃)₂ · H₂O</td>
<td>Fair (high polarization)</td>
<td>High</td>
<td></td>
<td></td>
<td>IV-12, -13</td>
</tr>
<tr>
<td>Pb/Sn</td>
<td>Wire solder</td>
<td>Good</td>
<td>High</td>
<td></td>
<td></td>
<td>IV-12, -13</td>
</tr>
<tr>
<td>11. ALLOYS</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
### III. Teflon-Bonded Materials

#### A. On Amalgamated Copper Foam

<table>
<thead>
<tr>
<th>Electrode</th>
<th>Physical Characteristics</th>
<th>Cathodic Performance</th>
<th>Anodic Performance</th>
<th>Hydrogen Evolution Overvoltage</th>
<th>Corrosion Characteristics</th>
<th>Figure No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>TaC</td>
<td>Powder, Teflon-bonded flag, Not active</td>
<td></td>
<td>Low</td>
<td></td>
<td></td>
<td>IV-25</td>
</tr>
<tr>
<td>TaN</td>
<td>Powder, Teflon-bonded flag, Not active</td>
<td></td>
<td>Low</td>
<td></td>
<td></td>
<td>IV-19</td>
</tr>
<tr>
<td>TiC</td>
<td>Powder, Teflon-bonded flag, Not active</td>
<td></td>
<td>Very Low</td>
<td></td>
<td></td>
<td>IV-23</td>
</tr>
<tr>
<td>TiN</td>
<td>Powder, Teflon-bonded flag, Not active</td>
<td></td>
<td>Very Low</td>
<td></td>
<td></td>
<td>IV-24</td>
</tr>
<tr>
<td>ZrC</td>
<td>Powder, Teflon-bonded flag, Not active</td>
<td></td>
<td>Low</td>
<td></td>
<td></td>
<td>IV-20, -21</td>
</tr>
<tr>
<td>ZrN</td>
<td>Powder, Teflon-bonded flag, Not active</td>
<td></td>
<td>Low</td>
<td></td>
<td></td>
<td>IV-22</td>
</tr>
</tbody>
</table>

#### B. On Ti Screen

<table>
<thead>
<tr>
<th>Electrode</th>
<th>Physical Characteristics</th>
<th>Cathodic Performance</th>
<th>Anodic Performance</th>
<th>Hydrogen Evolution Overvoltage</th>
<th>Corrosion Characteristics</th>
<th>Figure No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>B4C (Alpha)</td>
<td>Powder, Teflon-bonded flag, Not active</td>
<td></td>
<td>Low</td>
<td></td>
<td></td>
<td>IV-17</td>
</tr>
<tr>
<td>Cu/Pb</td>
<td>Powder, Teflon-bonded flag, Good</td>
<td></td>
<td>Medium</td>
<td></td>
<td></td>
<td>IV-14, -15</td>
</tr>
<tr>
<td>WC</td>
<td>Powder, Teflon-bonded flag, Not active</td>
<td></td>
<td>Low</td>
<td></td>
<td></td>
<td>IV-18</td>
</tr>
</tbody>
</table>

### IV. Plated Materials

#### A. Rotating Disk (Beckman) - Roughened NRE (10 rpm rotation)

<table>
<thead>
<tr>
<th>Electrode</th>
<th>Physical Characteristics</th>
<th>Cathodic Performance</th>
<th>Anodic Performance</th>
<th>Hydrogen Evolution Overvoltage</th>
<th>Corrosion Characteristics</th>
<th>Figure No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag/Pb</td>
<td>Ag⁺, Pb₂⁺ added to electrolyte</td>
<td>Good</td>
<td>Good</td>
<td>High</td>
<td></td>
<td>IV-41</td>
</tr>
<tr>
<td>Au</td>
<td>Ag⁺³ added to electrolyte (10 rpm)</td>
<td>Good</td>
<td>Very Low</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Au/Pb</td>
<td>Au⁺³, Pb₂⁺ added to electrolyte</td>
<td>Good</td>
<td>Good</td>
<td>High</td>
<td></td>
<td>IV-36, -37</td>
</tr>
</tbody>
</table>

---

2A) All powders were commercially obtained materials. In view of their overall unsuitability, no attempts were made to characterize their physical properties such as particle size and surface area.

B) A small quantity of Teflon (usually 20% or less) was used as an inert binder to hold powder materials on amalgamated copper foam screen.
<table>
<thead>
<tr>
<th>Electrode</th>
<th>Physical Characteristics</th>
<th>Cathodic Performance</th>
<th>Anodic Performance</th>
<th>Hydrogen Evolution Overvoltage</th>
<th>Corrosion Characteristics</th>
<th>Figure No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cd</td>
<td>Cd&lt;sup&gt;2+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td></td>
<td>High</td>
<td></td>
<td>IV-38, -39</td>
</tr>
<tr>
<td>Cd/Au</td>
<td>Cd&lt;sup&gt;2+&lt;/sup&gt;, Au&lt;sup&gt;3+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td></td>
<td>High</td>
<td></td>
<td>IV-38, -39</td>
</tr>
<tr>
<td>Cu/Pb</td>
<td>Cu&lt;sup&gt;+&lt;/sup&gt;, Pb&lt;sup&gt;2+&lt;/sup&gt; added to electrolyte (10 rps)</td>
<td>Good</td>
<td>Good</td>
<td>High</td>
<td></td>
<td>IV-40</td>
</tr>
<tr>
<td>Pb</td>
<td>Pb&lt;sup&gt;2+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td></td>
<td>High</td>
<td></td>
<td>IV-35</td>
</tr>
<tr>
<td><strong>B. Graphite Substrate</strong></td>
<td>Rod, wrapped with Teflon tape to expose end</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Au/Pb</td>
<td>Potentiostatic deposition in Au/Pb plating solution</td>
<td>Good</td>
<td>Good</td>
<td>High</td>
<td></td>
<td>IV-26, -27, -28</td>
</tr>
<tr>
<td>Au/In</td>
<td>Au&lt;sup&gt;3+&lt;/sup&gt;, In&lt;sup&gt;3+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td>Good</td>
<td>High</td>
<td></td>
<td>IV-43</td>
</tr>
<tr>
<td>In</td>
<td>In&lt;sup&gt;3+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td></td>
<td>High</td>
<td></td>
<td>IV-42</td>
</tr>
<tr>
<td>Pb</td>
<td>Pb&lt;sup&gt;2+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td></td>
<td>High</td>
<td></td>
<td>IV-34, -42</td>
</tr>
<tr>
<td><strong>C. Other Substrates</strong></td>
<td>Ag foil electrode, Pb&lt;sup&gt;4+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td>Good</td>
<td>High</td>
<td></td>
<td>IV-29</td>
</tr>
<tr>
<td>Ag (Pb plated)</td>
<td>Ag wire electrode, Pb&lt;sup&gt;4+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td></td>
<td>High</td>
<td></td>
<td>IV-31, -32</td>
</tr>
<tr>
<td>Au (Pb plated)</td>
<td>Au foil, Pb&lt;sup&gt;4+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td>Good</td>
<td>Medium</td>
<td></td>
<td>IV-33</td>
</tr>
<tr>
<td>Cu (Pb plated)</td>
<td>Cu foil, Pb&lt;sup&gt;4+&lt;/sup&gt; added to electrolyte</td>
<td>Good</td>
<td>Good</td>
<td>High</td>
<td></td>
<td>IV-30</td>
</tr>
</tbody>
</table>
simultaneous electrodeposition of Au and Pb from a common solution. In such event, an intimately dispersed mixture of Au and Pb will be deposited; and under some favorable, yet undefined, conditions, either a solid solution of Au and Pb or gold/lead intermetallics may be formed. Further work on this aspect of electrocatalysis is anticipated under NASA Contract No. DEN3-97.
Hydrogen Evolution Characteristics of Several Pure Metals in 1N HCl Solution

Figure IV-1

Figure IV-2
**Figure IV-3**

Screening Code 1, 2, 3

**Figure IV-4**

Screening Code 1, 2, 3
Figure IV-5

Figure IV-6
Figure IV-7

After activation at +1.2 volts...

Figure IV-8

After activation at +.4 volts...
**Figure IV-12**

Graph showing the current density vs. potential for different alloys in a solution of 1M HCl.

**Figure IV-13**

Graph showing the potential vs. volts for a solution of 1M HCl + 0.1M CrCl₃ with different alloy electrode materials.
Figure IV-14

Screening Code 1,2

Figure IV-15

Copper/Lead 90/10 Electrode

Screening Code 1,2,3
Figure IV-18

Screening Code 1,2

Figure IV-19

Screening Code 1,2
Screening Code 2

Figure IV-22

Screening Code 2

Figure IV-23
Screening Code 1,2

**Figure IV-24**

TITANIUM NITRIDE ELECTRODE
(20E TFE)

- 1N HCl
- 1N HCl + 0.1M CrCl₃

**Figure IV-25**

TANTALUM CARRIBE ELECTRODE
(20E TFE)

- 1N HCl
- 1N HCl + 0.1M CrCl₃
Figure IV-26

Figure IV-27
Figure IV-26

Au-Pb Co-deposited Electrode (on graphite rod)

Both sweeps obtained in first solution

1N HCl + 0.1M CrCl₃

Second Solution
1N HCl + 0.1M CrCl₃

Third Solution
1N HCl + 0.1M CrCl₃

Fourth Solution
1N HCl + 0.1M CrCl₃
Screening Code 1,2

Figure IV-29

Screening Code 1,2

Figure IV-30
Figure IV-31

**Potential, Volts vs. SCE**

**Screening Code 2,4**

- **ANODIC CURRENT**
- **CATHODIC CURRENT**

![Graph showing current density vs. potential for anodic and cathodic reactions with different solutions.](image)

- **Silver Wire Electrode**
- **Solution:**
  - 1M HCl
  - 10^-4 M Pb^{2+}
  - 0.1M Cr^{2+}
  - 0.1M Cr^{3+}

---

Figure IV-32

**Potential, Volts vs. SCE**

**Screening Code 1,2**

![Graph showing current density vs. potential for anodic and cathodic reactions with different solutions.](image)

- **Silver Wire Electrode**
- **Solution:**
  - 1M HCl
  - 1M HCl + 10^-4 M Pb^{2+}
  - 1M HCl + 10^-4 M Fe^{3+}
  - 1M HCl + 10^-4 M Fe^{3+} + 0.1M Cr^{3+}

---

35
Figure IV-33

Potential Volts vs SCE

**Gold Foil Electrode**

A

1N HCl + 0.08M Cr$^{3+}$

+ 0.29M Cr$^{2+}$ + (Pb$^{2+}$?)

B

1N HCl + 0.08M Cr$^{3+}$

0.29M Cr$^{2+}$ + 10$^{-4}$M Pb$^{2+}$

Sweep Rate: 0.05 Volts/min
Figure IV-34

Screening Code 1, 2

![Graph showing current density vs potential for different solutions]

Graphite Rod Electrode
1. 1M HCl
2. 1M HCl + Pb²⁺
3. 1M HCl + Pb²⁺ + 0.1M Cr⁶⁺
4. 1M HCl + 0.1M Cr⁶⁺
5. 1M HCl right after 4

Potential, Volts vs. SCE

Figure IV-35

Screening Code 2

![Graph showing current density vs potential for different solutions]

Vitreous Carbon Electrode
solution in 1M HCl + 10⁻⁴M Pb²⁺ + 0.1M Cr⁶⁺

- Sweep from 0.45V to -1.53V
- Sweep from -1.33V to -0.45V

Sweep Rate - 0.05V/min.

Potential, Volts vs SCE
Figure IV-36

Screening Code 2

<table>
<thead>
<tr>
<th>Potential, Volts vs SCE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Current Density, mA/cm²</td>
</tr>
</tbody>
</table>

Vitreous Carbon Electrode
Solution: 1M HCl + 1.5M CrCl₃
+ 10⁻⁴ M Au⁺³ + 10⁻⁵ M Pb⁺²
Sweep Rate: 0.05 volts/min
Rotation Rate:
- 40 RPS
- 4 RPS

Figure IV-37

Screening Code 2,4

ANODIC CURRENT
CATHODIC CURRENT

Vitreous Carbon Electrode
Solution: 1M HCl + 10⁻⁴ M Au⁺³
+ 10⁻⁵ M Pb⁺² + 1.1M Cr⁺³ + 0.57M Cr⁺²
Sweep Rate: 0.05 volts/min
Rotation Rate: 10 RPS
Figure IV-38

Figure IV-39
Figure IV-40

Screening Code 2, 3, 4

VITREOUS CARBON ELECTRODE

- IN HCl + 10^{-4} M PbCl_{2}
- + 10^{-4} M CuCl
- + 0.1 M CrCl_{3}
- IN HCl + 10^{-4} M PbCl_{2}
- + 10^{-4} M CuCl
- + 0.09 M CrCl_{3}
- + 0.09 M CrCl_{2}

Figure IV-41

Screening Code 2, 4

VITREOUS CARBON ELECTRODE

- IN HCl + 0.09 M Cr^{3+} + 0.09 M Cr^{2+}
- + 10^{-4} M Ag^{+1} + 10^{-4} M Pb^{2+}
(sweep rate = 0.1 V/min)
- Sweep from -1.25 V to -0.1 V vs. SCE
- Sweep from -0.1 V to -1.25 V

Potential, Volts vs. SCE

Current Density, mA/cm^{2}
Figure IV-42

Screening Code 1, 2

Graphite Rod Electrode
Sweep Rate: 0.05 Volts/min

- IN HCl
- IN HCl + 5 x 10⁻³ M
- Pb²⁺ + 0.1M CrCl₃
- IN HCl + 0.1M CrCl₃
- 4 x 10⁻⁴ M In³⁺

Figure IV-43

Screening Code 2, 4

Graphite Rod Electrode
Sweep Rate: 0.05 Volts/min
Solution: IN HCl + 0.09M
Cr²⁺ + 0.09M Cr³⁻
10⁻⁴ M Au³⁺ + 5 x 10⁻⁴ M In³⁺

First Sweep
Second Sweep
Figure IV-44

VITREOUS CARBON ELECTRODE

1N HCl + 10^{-6} Pb^{2+} + 10^{-4} Au^{3+}
+ 0.09M Cr^{3+} + 0.09M Cr^{2+}

- Sweep Rate = 0.05 V/min
- Sweep rate = 0.025 V/min
V. EVALUATION OF DISSOLVED ORGANIC COMPOUNDS AS HYDROGEN EVOLUTION INHIBITORS

A. Introduction

Five organic compounds mentioned by Vetter\textsuperscript{(6)} as hydrogen evolution inhibitors were tested with the objective of thereby increasing the number of suitable electrocatalysts. The organic inhibitors considered were thiourea, palmitic acid, cetyl alcohol, hexanol and n-propanol.

B. Experimental Results

The first material to be tested was thiourea. Solutions of $10^{-x}$M thiourea ($x = 1, 2, 3, 4, 5$) in 1N HCl were prepared. As shown in Figure V-1, concentrations above $10^{-4}$M enhanced hydrogen evolution. Solutions containing $10^{-4}$ and $10^{-5}$M thiourea in 1N HCl and 0.1M CrCl\textsubscript{3} were tested. The addition of thiourea at these levels had virtually no effect on the gold electrode.

The work reported in the literature on palmitic acid was performed in 6N HCl. In attempting to prepare a $10^{-3}$M solution, we found palmitic acid (as in the case with most aliphatic carboxilic acids) to be virtually insoluble. Upon testing the solution, which was less than $10^{-3}$M palmitic acid, we found that hydrogen evolution was only slightly enhanced as shown in Figure V-2.

We attempted to study the effect of cetyl alcohol addition to a solution of 0.1M CrCl\textsubscript{3} in 1N HCl containing PbCl\textsubscript{2} and AuCl\textsubscript{3}. The purpose was to see if the onset of $H_2$ evolution on the gold-lead plated electrode would be altered by the presence of the cetyl alcohol. We found that cetyl alcohol did not measurably dissolve. Consequently, the current-potential curves showed no discernible change. The cited use of this inhibitor was in 6N HCl, and we suspect that a stronger acid than we employed is required to solubilize cetyl alcohol.

A similar experiment was conducted with hexanol as was performed with cetyl alcohol. The solubility of hexanol also appears to be quite low. When $\sim 10^{-4}$M hexanol was added, no effect on $H_2$ evolution was observed. $10^{-3}$M hexanol also did not show any effect. Upon addition to the cell of an amount of hexanol which would exceed its solubility, it was noted that the electrode appeared to be
poisoned, and the chromic ion reaction was suppressed.

Finally, an experiment was performed to determine whether the addition of n-propanol would have any effect on the hydrogen evolution characteristics of a carbon electrode plated with gold and lead and acting as a catalyst for chromic ion reduction. Linear potential sweeps were applied at a rate of 100 mV/min and limiting currents were observed. The solutions that were tested were

$1N \text{HCl} + 10^{-4}M \text{Pb}^{2+} + 10^{-6} \text{Au}^{3+} + 0.1M \text{Cr}^{3+}$ and the same solution with additions of $10^{-4}M$ n-propanol and $10^{-2}M$ n-propanol. We did not observe any change in the hydrogen evolution characteristics of the electrode with the addition of n-propanol. With this experiment the solution used was freshly prepared, and there did seem to be a problem with plating occurring. We have noticed this before with plating solutions and feel that more investigation into plating could be quite beneficial for future study.

C. Recommendations

Based on the work done so far, a useful hydrogen evolution inhibitor has yet to be identified.
VI. PRELIMINARY COST ANALYSIS OF CHROMIUM CHEMICALS

A. Discussion

Some preliminary cost information was obtained under this contract.

The results are presented in Table I. The (dollar per pound) price of large lots of the various chromium chemicals listed was obtained from Chemical Marketing Reporter. The price of chromium metal was quoted by Union Carbide Corp., Metals Div., Marietta, Ohio. The prices were converted to the units dollar per pound of chromium in order to facilitate comparisons.
TABLE VI-1

COST OF COMMON CHROMIUM COMPOUNDS
(large quantities)

<table>
<thead>
<tr>
<th>Compound</th>
<th>$/lb</th>
<th>$/lb Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potassium Chromate $K_2CrO_4$ (194.2)</td>
<td>.57</td>
<td>1.90</td>
</tr>
<tr>
<td>Potassium Dichromate $K_2Cr_2O_7$ (294.19)</td>
<td>.48</td>
<td>1.36</td>
</tr>
<tr>
<td>Potassium Chromium Sulfate $KCr(SO_4)_2 \cdot 12H_2O$ (499.41) Potash Chromium Alum</td>
<td>.24</td>
<td>2.79</td>
</tr>
<tr>
<td>Sodium Chromate $Na_2CrO_4$ (252.05) Tetrahydrate (161.97) Anhydrous</td>
<td>.32</td>
<td>1.55</td>
</tr>
<tr>
<td>Sodium Dichromate $Na_2Cr_2O_7 \cdot 2H_2O$ (298)</td>
<td>.37</td>
<td>1.06</td>
</tr>
<tr>
<td>Ammonium Dichromate $(NH_4)_2Cr_2O_7$ (252.06)</td>
<td>.78</td>
<td>1.89</td>
</tr>
<tr>
<td><strong>Lead Compounds:</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chrome Yellow $PbCrO_4$ (323.18)</td>
<td>.83</td>
<td>5.16</td>
</tr>
<tr>
<td>Chrome Orange $Pb_2(OH)_2 \cdot CrO_4$ (564.39)</td>
<td>.83-.89</td>
<td>9.01-9.66</td>
</tr>
<tr>
<td>Chrome Green $Fe_4(Fe(CN)_6) \cdot PbCrO_4$ (758)</td>
<td>1.36</td>
<td>19.83</td>
</tr>
<tr>
<td>Chromium Fluoride $CrF_3$ (108.99)</td>
<td>.81</td>
<td>1.70</td>
</tr>
<tr>
<td>Chromium Oxide Green $Cr_2O_3$ (151.99)</td>
<td>2.10</td>
<td>6.14</td>
</tr>
<tr>
<td>Zinc Chromate $ZnCrO_4$ (233.36)</td>
<td>1.00</td>
<td>4.49</td>
</tr>
<tr>
<td>Chromium $Cr$ (52.00)</td>
<td>2.99</td>
<td>2.99</td>
</tr>
</tbody>
</table>
VII. REFERENCES


7. J. Giner and K. Cahill, "Catalyst Surfaces for the Chromous-Chromic Redox Couple" (patent pending).
