TO: KSI/Scientific & Technical Information Division
   Attn: Miss Winnie M. Morgan

FROM: GP/Office of Assistant General Counsel for Patent Matters

SUBJECT: Announcement of NASA-Owned U.S. Patents in STAR

In accordance with the procedures agreed upon by Code GP and Code KSI, the attached NASA-owned U.S. Patent is being forwarded for abstracting and announcement in NASA STAR.

The following information is provided:

U.S. Patent No. : 3,826,726

Government or Corporate Employee : U.S. Government

Supplementary Corporate Source (if applicable) : 

NASA Patent Case No. : Lew-19906-1

NOTE - If this patent covers an invention made by a corporate employee of a NASA Contractor, the following is applicable:

YES / / NO [X]

Pursuant to Section 305(a) of the National Aeronautics and Space Act, the name of the Administrator of NASA appears on the first page of the patent; however, the name of the actual inventor (author) appears at the heading of column No. 1 of the Specification, following the words "...with respect to an invention of..."

Bonnie L. Woerner
Enclosure
SUMMARY OF THE INVENTION

An apparatus for depositing elements by irradiating liquids. Ultra pure elements are precipitated from aqueous solutions. The apparatus comprises a reaction chamber into which is introduced a liquid containing metal ions. The liquid is irradiated with high energy particles. This promotes a chemical reaction causing metals to precipitate or separate out from the solutions. The process is performed under high temperature, normal pressure, and under an inert atmosphere. A scavenger additive is unnecessary. The method can be used for preparing pure metals from solutions containing several metal ions. The process is also useful for depositing adherent metal coatings on substrates as a radiation chemistry method for producing pure metals in an improved method of preparing catalysts. Another object of the present invention is to provide a low temperature process for preparing pure metals from solutions. The temperature of the solution being limited by the freezing point of the solution. The radiation method is to provide an improved method of preparing catalysts. The method is also useful for depositing adherent metal coatings on substrates as a radiation chemistry method for producing pure metals.
When formed by irradiating a solution of an electrolyte, particularly lead form macro sponge-like conglomerates. The reaction with the environment is minimized. The product wherein the solvent is a dry organic liquid.

Metals occurs at a low enough temperature where condensation due to diffusion of container material and/or reaction with the environment is minimized. The product 75

is isolated as a precipitate; reactants and unwanted reaction products remain in solution. Also easily purified metal salts may be used.

Samples of silver, copper and nickel were all found to be 99.9+ pure by chemical analysis. In each metal, X-ray diffraction showed that only the metal was present.

DESCRIPTION OF ALTERNATE EMBODIMENT

Active metals, those which reduce water, and anhydrous compounds are best prepared from dry organic liquids. Antimony and some anhydrous lower valence metal halides were prepared in accordance with the invention using dry organic liquids as solvents. Antimony (III) chloride (SbCl₃) was selected as the metal salt because of its solubility in a wide variety of organic liquids and because of the relative convenience in handling antimony metal.

The yield of antimony upon irradiation of SbCl₃ solutions in dry organic liquids is shown in Table II. As in the preferred embodiment the electron energy was 2 mev. The concentration of the SbCl₃ was 0.25 M, the current was 20 µA., and the dose was 0.20 coulomb.

### TABLE II.—ANTIMONY YIELD

<table>
<thead>
<tr>
<th>Organic liquid</th>
<th>n-G(Sb)</th>
<th>Purity of Sb, percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methanol</td>
<td>2.4</td>
<td>99.9+</td>
</tr>
<tr>
<td>Di-butyl sulfoxide</td>
<td>1.0</td>
<td>99.9+</td>
</tr>
<tr>
<td>Tetrahydroluim</td>
<td>1.0</td>
<td>98.6</td>
</tr>
<tr>
<td>Cyclonaphthol</td>
<td>0.9</td>
<td>99.0</td>
</tr>
<tr>
<td>Amyl acetate</td>
<td>0.7</td>
<td>94.6</td>
</tr>
<tr>
<td>Quinolione</td>
<td>0.4</td>
<td>99.0</td>
</tr>
<tr>
<td>Dibutyl phthalate</td>
<td>0.3</td>
<td>99.0</td>
</tr>
<tr>
<td>2-methyl phenol</td>
<td>0.2</td>
<td>99.0</td>
</tr>
<tr>
<td>Oleic acid</td>
<td>0.2</td>
<td>99.0</td>
</tr>
<tr>
<td>Toluene</td>
<td>0.1</td>
<td>99.0</td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td>0.2</td>
<td>99.0</td>
</tr>
<tr>
<td>Acetic acid</td>
<td>0.2</td>
<td>99.0</td>
</tr>
<tr>
<td>2,3-dimethylpropane</td>
<td>0.1</td>
<td>99.0</td>
</tr>
<tr>
<td>Glycolol</td>
<td>0.1</td>
<td>99.0</td>
</tr>
</tbody>
</table>

While several embodiments of the invention have been described it will be appreciated that various modifications may be made without departing from the spirit of the invention or the scope of the subjoined claims. For example, other metals including zinc and cadmium have also been deposited from dry organic liquids.

What is claimed is:

1. A radiation chemical process for producing pure metals comprising the steps of adding an oxidizing species scavenger to a liquid which decomposes when irradiated with high energy particles producing long lived reducing species and oxidizing species, dissolving at least one metal compound in said liquid to form a solution, placing said solution in an inert atmosphere at ambient temperature and pressure, irradiating said solution with high energy particles while in said inert atmosphere at ambient temperature and pressure thereby decomposing said liquid whereby said oxidizing species are scavenged and at least one metal is reduced and precipitates from said solution as a pure metal, and removing said pure metal from said solution.

2. A radiation chemical process as claimed in claim 1 wherein a salt of a metal is dissolved in the solvent and said metal precipitates from said solvent.

3. A radiation chemical process as claimed in claim 1 wherein a plurality of metal compounds are dissolved in the solvent and a predetermined metal precipitates from said solvent.

4. A radiation chemical process as claimed in claim 1 wherein the solution is irradiated with an electron beam having an accelerating voltage between 0.5 and 2.5 mev.

5. A radiation chemical process as claimed in claim 4 wherein the solution is irradiated with an electron beam having an accelerating voltage of about 2 mev.

6. A radiation chemical process as claimed in claim 1 wherein the solvent is a dry organic liquid.
7. A radiation chemical process as claimed in claim 6 wherein antimony (III) chloride is dissolved in the dry organic liquid and antimony precipitates from the solution during irradiation.

8. A radiation chemical process as claimed in claim 1 wherein the solvent is water which forms hydrogen atoms, hydrated atoms, and hydroxyl radicals during irradiation.

9. A radiation chemical process as claimed in claim 8 wherein the solvent contains a hydroxyl radical scavenger.

10. A radiation chemical process as claimed in claim 9 wherein the solvent contains hydroxyl radical scavengers selected from the group consisting of primary alcohols, secondary alcohols, polyhydroxyl alcohols, and aldehydes.

References Cited
UNITED STATES PATENTS

3,073,766 1/1963 Bown et al. 204—157.1 H
3,104,216 9/1963 Ruskin 204—157.1 H

HOWARD S. WILLIAMS, Primary Examiner