Ion-Exchange Separation of Silver and Lead

*This work was supported by the U. S. Atomic Energy Commission, Contract AT(ll-l)499.

Harovel Grays and Harold F. Walton
University of Colorado, Boulder, Colorado

Summary

Silver and lead were separated on a column of carboxylic cation-exchange resin using a diethanolamine-diethanolammonium nitrate solution to elute silver. Lead was eluted with hydrochloric acid.

Recently we showed that solutions of diethanolamine salts containing excess base could separate metals by chromatography on cation-exchange resins (1). Copper(II) was not bound to the resin at all, but stayed in solution as an uncharged complex (2). In this way copper could be separated from large excesses of other metals. Another metal that was very weakly held was Ag. Chromatography on resin-impregnated paper showed that Ag(I) had
DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.
DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.
RF approaching unity, while other metals, including lead, had RF values of 0.5 or less. Because of the practical utility of separating silver from lead we decided to explore column separations of these elements based on the paper chromatography results.

EXPERIMENTAL

The resins were Bio-Rad AG 50W-X8, a sulfonated polystyrene cation-exchange resin, and Bio-Rex 70, a crosslinked polymethacrylic acid, each 100 - 200 mesh. The columns were 1.1 cm internal diameter and 18 to 36 cm long. Silver and lead were introduced as their nitrates. Diethanolamine, reagent grade, was used without further purification. To analyze the effluents, silver was titrated with thiocyanate after acidifying with nitric acid, and lead was titrated with EDTA.

The first experiments were made with sulfonated polystyrene resin and eluents containing diethanolamine and its nitrate in concentrations about 1 M and 0.5 M, respectively. Silver was eluted satisfactorily within 1 - 2 void column volumes, and it was free from lead. Lead, however, was retained very strongly, and large volumes of 1 - 5 M nitric or hydrochloric acid were needed to remove it from the column.

The resin Bio-Rex 70 has weakly acidic carboxyl groups, and can therefore be efficiently regenerated by acid. Columns of this resin were tested. Again, silver was eluted rapidly and
was free from lead. Lead could now be eluted completely within 2 - 3 void column volumes by nitric or hydrochloric acid, hydrochloric acid being more effective. After use of the column was regenerated by passing dilute aqueous diethanolamine.

Table I shows the recoveries of silver and lead from a range of mixtures; Figure 1 shows elution curves for two mixtures, one containing a large excess of lead.

DISCUSSION

This technique is better suited to separating small amounts of lead from large amounts of silver than vice versa. To recover small proportions of silver one would have to pass large volumes of solution, and the silver would be recovered as a very dilute solution. The high viscosity of the diethanolamine eluent is a drawback that could probably be overcome by raising the temperature. However, the separation does appear to be clean and complete, and the indications are that copper would be the only "heavy metal" accompanying the silver.

For removing traces of silver from lead an extraction procedure is probably better, such as the dithizone extraction of Jangida et al. (3).


TABLE 1

Silver - Lead Separation

Column: Bio-Rex 70 carboxylate resin, 100-200 mesh: 1.1 x 18 cm

Eluents: (a) Diethanolamine base, 0.75 M; nitrate, 0.50 M
(b) Water
(c) HCl, 6 M

<table>
<thead>
<tr>
<th>SILVER:</th>
<th>LEAD:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mmol added</td>
<td>Recovery, %</td>
</tr>
<tr>
<td>0.265</td>
<td>99.5</td>
</tr>
<tr>
<td>0.412</td>
<td>102</td>
</tr>
<tr>
<td>0.048</td>
<td>94</td>
</tr>
<tr>
<td>0.048</td>
<td>96</td>
</tr>
<tr>
<td>0.0060</td>
<td>100</td>
</tr>
<tr>
<td>0.0060</td>
<td>100</td>
</tr>
</tbody>
</table>
MILLIMOLE IN 10 ml

0.10

0.20

0.75M DIETOL

0.50M DHNO3

50

H2O

100

150

Ag

CARBOXYLATE RESIN,
18 cm x 1.1 cm

Pb

6M HCl