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### Ion-Exchange Separation of Silver and Lead

Harovel Grays<sup>a</sup>; Harold F. Walton<sup>a</sup>

<sup>a</sup> UNIVERSITY OF COLORADO, BOULDER, COLORADO

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## NOTE

### Ion-Exchange Separation of Silver and Lead\*

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  $R_F$  approaching unity, while other metals, including lead, had  $R_F$  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 cross-linked polymethacrylic acid, each 100-200 mesh. The columns were 1.1 cm i.d. and 18 to 36 cm long. Silver and lead were introduced as their nitrates. Diethanolamine, reagent grade, was used without further purification. To

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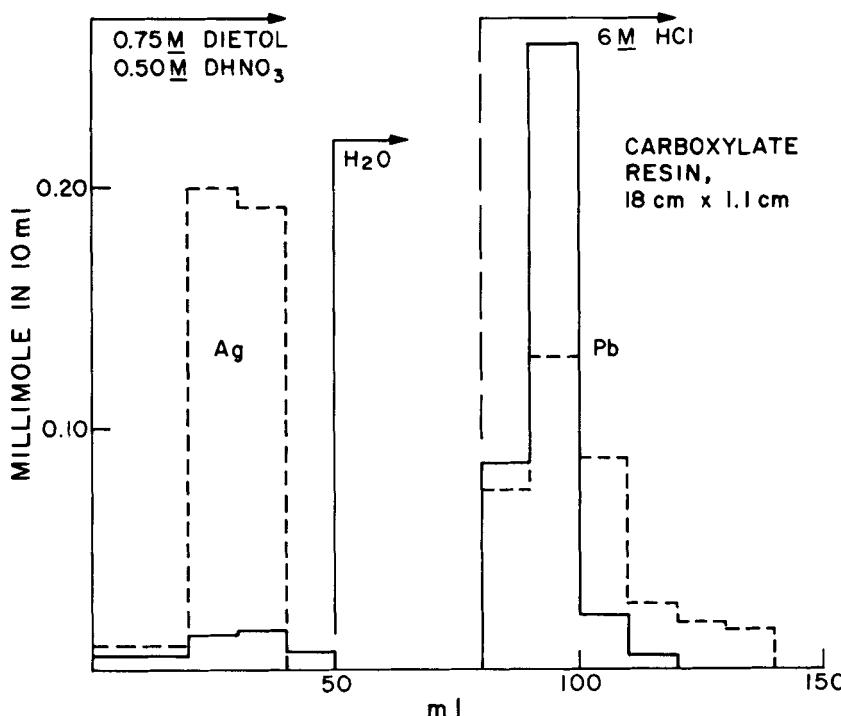


FIG. 1. Separation of silver from lead. Solid curve, 0.048 mmole Ag, 0.350 mmole Pb. Dashed curve, 0.412 mmole Ag, 0.350 mmole Pb.

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 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. The column was regenerated by passing dilute aqueous diethanolamine.

TABLE 1  
Silver-Lead Separation

Column: Bio-Rex 70 carboxylate resin, 100-200 mesh:  $1.1 \times 18$  cm

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

Silver		Lead	
mmole added	Recovery (%)	mmole added	Recovery (%)
0.265	99.5	0.165	99.5
0.412	102	0.350	102
0.048	94	0.350	100.5
0.048	96	0.535	100.7
0.0060	100	0.350	101.5
0.0060	100	2.67	99

Table 1 shows the recoveries of silver and lead from a range of mixtures; Fig. 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, large volumes of solution would have to pass, 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).

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