

Experiment 20: Preparative Chromatography for Pb

SYNOPSIS: Isotopic determination of lead often requires a purified sample containing only lead, since the main experimental difficulty is not in the separation and quantification of lead in general from other metals, but in lead from itself. Lead is quantitatively separated from other metal species by formation of PbBr_3^- which is exchanged onto an anion exchange column and subsequently eluted with nitric acid. The nitric acid both competes for the exchange sites, and alters the distribution of lead bromide species.

READINGS Read pages 331-334 in Critical Reviews. Attached is an article for a preparative method used by geochemists who are interested in very small, very pure lead samples for geochronological measurements of the earth's age.

Instruments:

AG1-X8 resin of 200-400 mesh
Glass tube 15 mm i.d. and 250 mm long
#2 glass scintered stopper

Solutions

1 M HBr
0.2 M HBr + 0.5 M HNO_3
0.05 M HBr + 0.5 M HNO_3

Procedure

1. Mix 5 g AG1-X8 resin with water to a slurry.
2. Place the glass sinter at the bottom of the glass column
3. Place the resin slurry into the column
4. Pass 100 mL 1M HBr through the column to convert the column to the Br form. Expect about a 5 mm change in the height of the column due to shrinkage.
5. Dissolve lead containing sample in 25 mL of 1M HBr
6. Pass Pb mixture onto the column.
7. Rinse the column with 200 mL of 0.2 M HBr+0.5 M HNO_3 to remove other metals species.
8. Collect the lead from the column by passing 200 mL 0.05 M HBr + 0.5 M HNO_3 through the column. Collect the entire volume from the column
9. The collected solution can be measured directly on the AA since it is high in nitrate and low in bromide.