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Rapid extraction of Sr and Pb by ion-specific chromatography for thermal ionization mass spectrometry: an update

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Abstract: A modification to a laboratory procedure is reported for sequential separation of Sr and Pb using Sr-Resin™. The modification entails adding 125 µL of inert resin substrate to the bottom of a 125 µL column of Sr-Resin™. The inert resin substrate adsorbs crown-6 ether removed from the column during extraction of Pb in 6 N HCl. The absence of the crown-6 ether increases the solubility of the 6 N HCl eluant, and results in a more quantitative recovery of the Pb concentrate and improved analysis by thermal ionization mass spectrometry.

Résumé : La modification d'une procédure de laboratoire visant à séparer Sr et Pb au moyen d'une résine (Sr-Resin™) est décrite. Elle consiste à ajouter 125 µL d'un substrat de résine inerte dans le fond d'une colonne de 125 µL de Sr-Resin™. La résine inerte adsorbe un éther couronne-6 prélevé dans la colonne durant l'extraction de Pb dans du HCl 6 N. L'absence d'éther couronne-6 augmente la solubilité de l'éluant HCl 6 N et permet de récupérer une plus grande quantité de Pb et d'améliorer l'analyse par spectrométrie de masse à ionisation thermique.

Table 1. Results of column tests on Pb concentrate viscosity and blanks.

	Column 1	Column 2	Column 3	Column 4
volume of Inert Pre-Filter Material™ (µL)	0	25	60	125
volume of Sr-Resin™ (µL)	250	225	190	125
Character of dried-down 6 N HCl eluant	Viscous, gel-like concentrate. Insoluble in water. Difficult to introduce into capillary pipette tip.	Viscous, gel-like concentrate. Insoluble in water. Difficult to introduce into capillary pipette tip.	Viscous concentrate. Water soluble. Difficult to remove from capillary pipette tip.	Water-soluble concentrate. Flows readily in capillary pipette tip.
Pb column procedure blank (pg)	81	90	101	79

INTRODUCTION

A procedure for sorption and sequential removal of Sr and Pb from Sr-Resin™ (Eichrom Industries Inc., Darien, Illinois) for thermal ionization mass spectrometer (TIMS) analysis at the Geological Survey of Canada was described by Thériault and Davis (1999). The Sr-Resin™, a bis-t-butyl-cis-dicyclohehano-18-crown-6 ether (Horowitz et al., 1991, 1994), has the capability of selectively retaining Sr and Pb over a broad range of nitric acid concentration, and allows for separation of these elements from a wide variety of matrix constituents in solution. A problem noted with this method is that the Pb concentrate obtained after drying down the 6 N HCl eluant formed a relatively insoluble and viscous residue that caused significant problems for loading the sample for mass spectrometer analysis.

The insoluble, gel-like character of the Pb concentrate is thought to be caused by the actual stripping of the active ingredient crown-6 ether from the Sr-Resin™ substrate during elution of Pb with 6 N HCl (L. Jassin, Eichrom Industries Inc., pers. comm., 1999). Inert Pre-Filter Material™ (Eichrom Industries Inc., Darien, Illinois), the organic substrate of Sr-Resin™, was packed at the bottom of columns and tests were conducted to assess its ability to adsorb organics stripped from the overlying Sr-Resin™ bed. The following describes a simple modification to the method presented in Thériault and Davis (1999) that reduces this viscosity problem.

MODIFIED CHEMICAL PROCEDURE

The method described by Thériault and Davis (1999) used columns with 250 µL of Sr-Resin™ over a bed length of 30 mm. This resin bed yields a minimum working capacity of approximately 225 µg of Sr, based on specifications by Eichrom Industries Inc. of 10 to 20% of the total column capacity of 8.9 mg Sr/mL of resin bed.

Four columns were tested to assess the capability of Inert Pre-Filter Material™ to adsorb crown-6 ether stripped from Sr-Resin™ with 6 N HCl. Inert Pre-Filter Material™ beads 100 to 150 µm in diameter were added in volumes varying from 0 µL to 125 µL to empty pre-shrink PFTE-Teflon™ columns with 30 mm bed lengths. The column volumes were then adjusted to 250 µL by adding Sr-Resin™. Test results are presented in Table 1. Sample material was not introduced to the column, but reagents were added as outlined in Table 2, simulating column conditions during sample processing and ensuring that the only Pb removed with 6 N HCl is from the chromatographic materials. The beakers placed beneath the columns to collect 6 N HCl eluant were spiked with ^{208}Pb, thus allowing measurement of Pb blanks associated with column procedure. After evaporation, the 6 N HCl eluant from columns 1 and 2, charged with 0 µL and 25 µL of Inert Pre-Filter Material™ respectively, were similar to those described by Thériault and Davis (1999) in which Pb concentrates collected after addition of 6 N HCl formed a viscous, gel-like material that was insoluble in water and very difficult to remove with a teflon capillary pipette tip typically used for loading Pb onto Re filaments for TIMS analysis. Column 3 charged with 60 µL of Inert Pre-Filter Material™ yielded a less viscous Pb concentrate than columns 1 and 2 that was partly soluble in water and removable from the collection beaker using a capillary pipette tip for loading onto a Re filament. Column 4, charged with 125 µL Inert Pre-Filter Material™, yielded a nonviscous concentrate that was readily introduced in a capillary pipette tip and easily loaded onto a Re filament. Measured Pb blanks for the four test columns ranged from 79 to 101 pg, with no clear relationship between the amount of Inert Pre-Filter Material™ used and blank. These Pb blanks are generally lower than the 79 to 183 pg procedural blanks reported by Thériault and Davis (1999) for 30 mm columns charged with Sr-Resin™ only. The results suggest that Pb blank levels are not related to the relative amounts of Inert Pre-Filter Material™ and Sr-Resin™ used.

Table 2. Procedure for extracting Sr and Pb using 125 µL Sr-Resin™- 125µL Inert Pre-Filter Material™ columns.

Column preparation
Wash Sr-Resin™ with 6 N HCl, H_2O, and 3 N HNO_3
Add 125 µL Inert Pre-Filter Material™ to columns
Add 125 µL Sr-Resin ™ to columns
Wash twice with 3 mL 6 N HCl, 3 mL H_2O
Equilibrate columns with 3 mL 3.2 N HNO_3
Sample loading, and Sr and Pb collection
Add sample as 1 mL 3.2 N HNO_3
Add 5 mL 3.2 N HNO_3
Insert Sr collection beakers
Collect Sr with 3 mL H_2O
Add 0.01 mL H_3PO_4 to Sr collection beakers
Insert Pb collection beakers
Collect Pb with 4 mL 6.2 N HCL
Add 0.01 mL HNO_3 to Pb collection beakers
Evaporate Sr and Pb concentrates to dryness

SUMMARY

Modifications brought to a chromatographic column procedure to sequentially extract Sr and Pb from a nitric acid solution using Sr-Resin™ ion-specific resin result in a nonviscous Pb concentrate, improving upon an earlier version of the procedure (Thériault and Davis, 1999). The updated procedure is outlined in Table 2.

A volume of 125 µL Inert Pre-Filter Material™ packed beneath 125 µL Sr-Resin™ was sufficient to yield a nonviscous Pb concentrate after elution with 6 N HCl. The entire Pb concentrate obtained from elution with 6 N HCl readily loads onto Re filaments and produces stable signals of appropriate intensity and durability for high precision Pb isotope measurements by TIMS analysis. Although the amount of Sr-Resin™ recommended in the updated procedure is reduced by 50%, the active resin volume of 125 µL yields a minimum column working capacity of approximately 112 µg of Sr, an amount of recovered material that exceeds requirements for Sr and Pb TIMS analysis. The lowest procedural Pb blank obtained is approximately 80 pg and does not appear to be related to the amount Sr-Resin™ or Inert Pre-Filter Material™ used in the columns.

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