

On the development of a rapid method for the determination of Sr and Pb in water samples based on TK100/1 Resins

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Introduction

The crown-ether based SR Resin is frequently used for the separation and determination of Pb-210 in aqueous samples via liquid scintillation (LSC) or gas proportional counting (GPC). The resin only shows significant Pb (and Sr) retention at moderate to high acid concentrations; it does thus not allow for direct loading of Pb from acidified or raw water samples, making the additional use of pre-concentration steps such as ion exchange or co-precipitation necessary. In order to simplify and quicken the Pb-210 determination an extraction chromatographic resin (TK100 Resin) allowing the direct load of Pb from water samples and its subsequent purification on the same resin has been developed and characterized. The TK100 Resin is based on the same crown-ether used in the SR Resin; accordingly its selectivity and robustness against interferences from common matrix elements, such as e.g. Ca and Mg, are similar to that of the SR Resin, however, including HDEHP into its composition allows extracting Pb at a much wider range of pH.

TK100 Resin

- Original project Sr via DGT (Diffuse Gradients in Thin films)
 - Bio-availability of Sr
 - Additional project: rapid methods for Sr in aqueous samples
- Keep Sr Resin selectivity (crown ether), increase uptake pH range
- 1st approach: Replace 1-Octanol by HDEHP
- Screening of several test resins (varying HDEHP contents)
- Characterisation of best suited resin prototype
- Elution studies

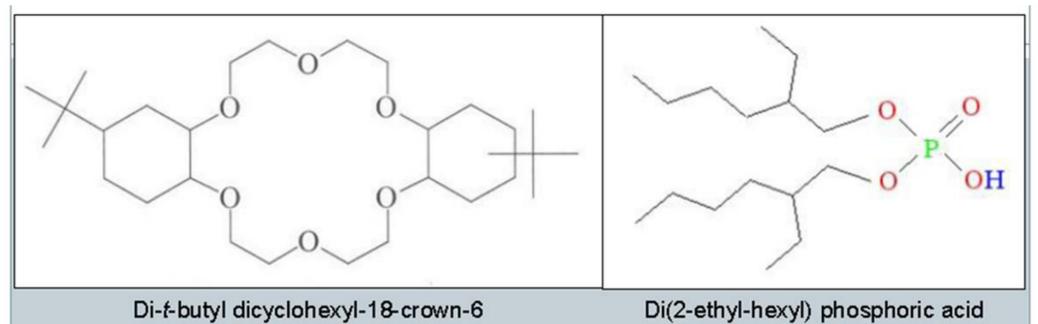


Figure 1: Extractant system TK100 Resin

Column elution studies

➤ Column elution study

- Test samples:
 - pH7, 1 mg Sr, 100 mg Ca, 5 mg K, 0.1 mg Pb, Y, U per sample
 - 1L samples, loaded in 100 mL aliquots
 - 2 mL columns (650 mg resin)
 - Vacuum supported separation, 5 mL/min
 - ICP-MS measurement of effluents
 - Load and first rinse at pH 7
 - K and Ca direct breakthrough during load
 - Sr breakthrough after 500 – 600 mL
 - Y removed with 8M HNO₃
 - Sr elution with 2M HCl
 - Pb and U co-eluted with 6M HCl
 - Pb/U separation via oxalates under evaluation

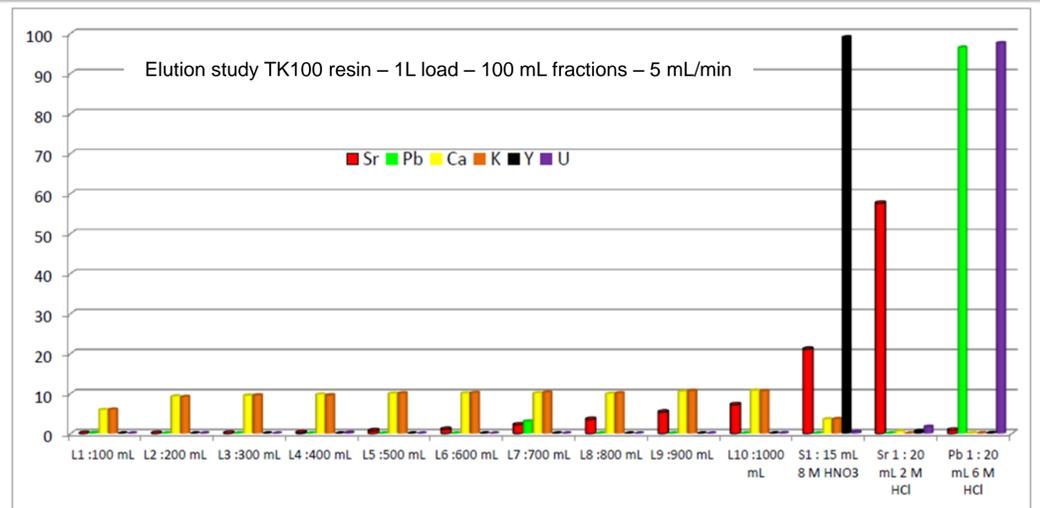


Figure 2: Column elution study TK100 resin

Disc elution studies

- Discs allow for faster flow rates
- 47 mm Ø discs based on TK100 resin
- Test on 1L water samples:
 - pH 7, 1 mg Sr, 250 mg Ca, 5 mg K, 0.1 mg Pb, Y, U per sample
 - Flow rate ~30 ml/min, gravity flow
 - Effluents analyzed by ICP-MS
 - Pb uptake (ICP-MS) >95%
 - Rinsing steps to be optimized
- Aim: direct measurement of loaded discs
- Analogue to Tc-99/TEVA disc method
- Test on 5L water samples:
 - pH 7, 1 mg Sr, 500 mg Ca, 25 mg K, 0.5 mg Pb, Y, U per sample
 - Load in five 1L aliquots, analysis of effluents by ICP-MS
 - Flow rate ~30 ml/min, gravity flow
 - Pb uptake during load ≥ 95%

On-going work

- Optimization of rinsing volumes (partial Pb breakthrough during rinse)
- Application to high-salt waters
- Application to larger water samples
- Higher flow-rates
- LSC counting of loaded discs

Ionic liquids

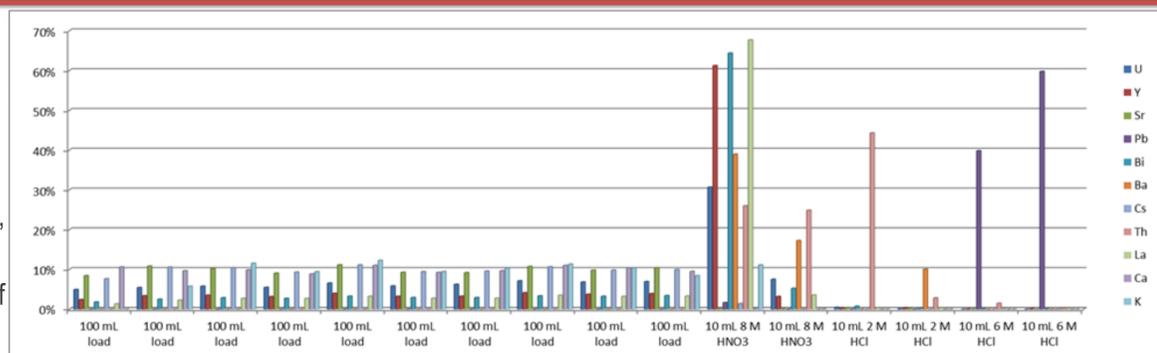


Figure 3: Elution study, selected elements, CE/EMIM resin

- HDEHP replaced by ionic liquids
 - Different ionic liquids under evaluation
- Elution studies show improved selectivity for Pb
- Pb breaks through earlier than on TK100 during sample load -> lower loading volumes

Conclusions

- Introduction of HDEHP and RTILs results in extended uptake pH range (pH 8 to high nitric acid) for Sr and Pb
- Direct extraction of Pb (and Sr) from non-acidified water possible
- Tests in column and disc form
- High Pb uptake, Pb separation possible
- Discs for increased flow rates and direct LSC
- Use of RTIL increases Pb selectivity but lowers sample load volume

Literature

1. Surman J. et al.: Development of a Rapid Strontium-90 Determination Method for Environmental Waters using a new Sr Selective Resin and Measurement by LSC. Presented at the LSC 2013 conference, 18 – 22.03.13, Barcelona, Spain
2. Dirks C. et al.: On the development of a method for the rapid determination of Pb-210 in water samples based on a new Sr selective resin. Presented at the NORM 7 conference, 22 – 26.04.2013, Beijing China
3. Carina Dirks, Jake Surman, Jackie Pates, Steffen Happel: Rapid determination of Pb-210 and Sr-90 in water samples using new crown-ether based extraction chromatographic resins, oral presentation, TrisKem International UGM, 6.10.14, Moscow (RU), http://www.triskem-international.com/ru/iso_album/8_rapid_determination_of_pb-210_and_sr-90_in_water_samples_using_new_crown-ether_based_extraction_chromatographic_resins.pdf