

Extraction Chromatographic Studies of Rf homologs with Eichrom's Pb and Sr Resins

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Separation Requirements

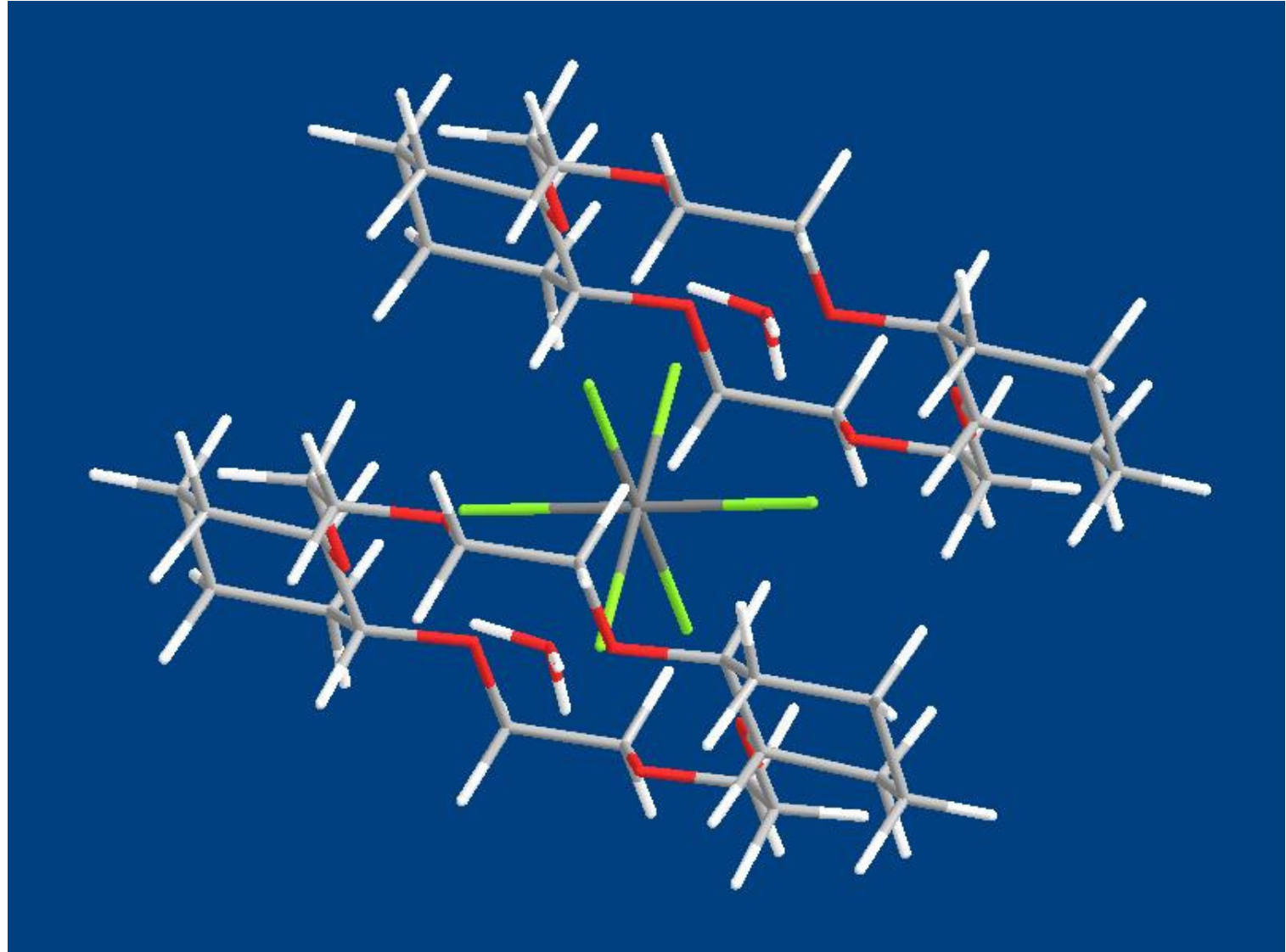
- Rapid
- Large number of exchange steps
- Highly Selective
- Preferably a continuous process
- Samples easily prepared for α spec

**Extraction Chromatography
fulfills all of these**

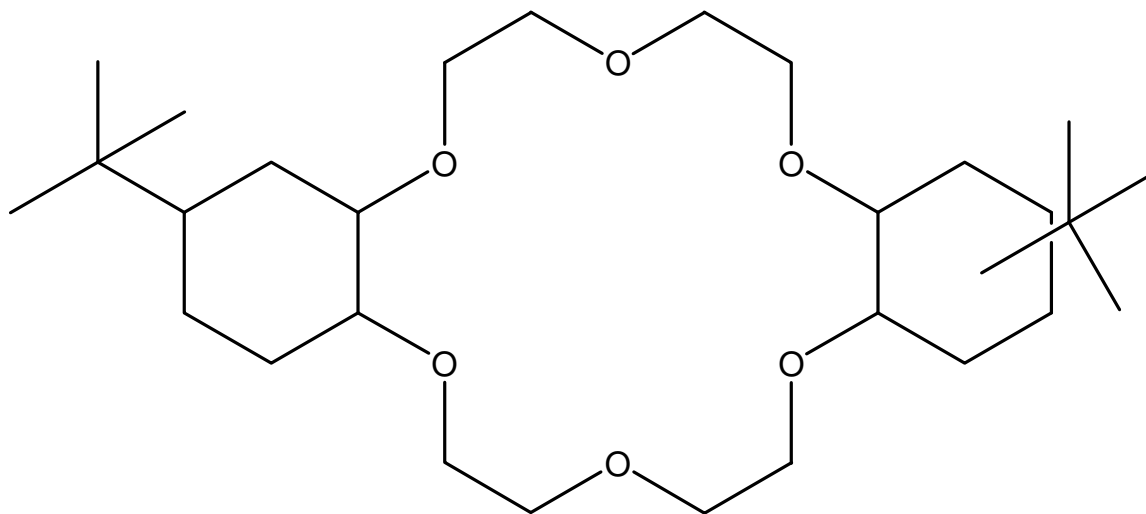
Background

- Crown Ethers have been shown to extract Rf homologs¹
 - Physical pre-separation is necessary due to the extraction mechanism
- Complex formation is dependent on solution conditions
 - $2[\text{crown ether} \cdot \text{H}_3\text{O}^+]$ responsible for extraction
 - $[\text{MCl}_6]^{2-}$ is extracted
 - $[\text{MCl}_6]^{2-}$ does not form below 6M HCl

Extraction Mechanism



Eichrom's Pb and Sr Resins



Pb Resin

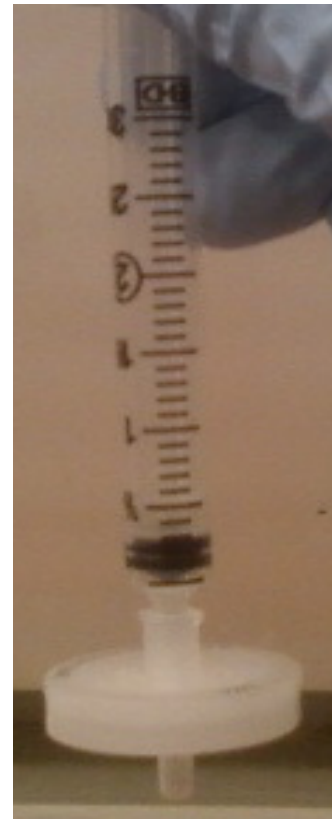
- 0.75M crown ether
- Isodecanol solvent
- Free resin
- 2mL pre-packed cartridges

Sr Resin

- 1.0M crown ether
- 1-octanol solvent
- Free resin
- 2mL pre-packed cartridges

Batch Studies

- $\sim 3\text{cps } ^{95}\text{Zr}$ and $\sim 2\text{cps } ^{175}\text{Hf}$ placed in a PPE 15 mL centrifuge tube
- Samples evaporated to dryness, reconstituted with HCl solution
- Samples counted using HPGe gamma spectroscopy for 30 minutes
- 10 – 20mg of resin added
- Mixed for 1 hour
- Filtered using a PPE syringe filter and placed in a clean PPE 15mL centrifuge tube
- Samples counted for 30 minutes



Results

- Absorption expressed in terms of k' values, number of free column volumes to peak maximum²
- k' value can be determined from D_w
 - Dependent on the resin (multiplication factor)³

Resin	Correction Factor (F)
Pb	0.55
Sr	0.46

$$k' = D_w \times F$$

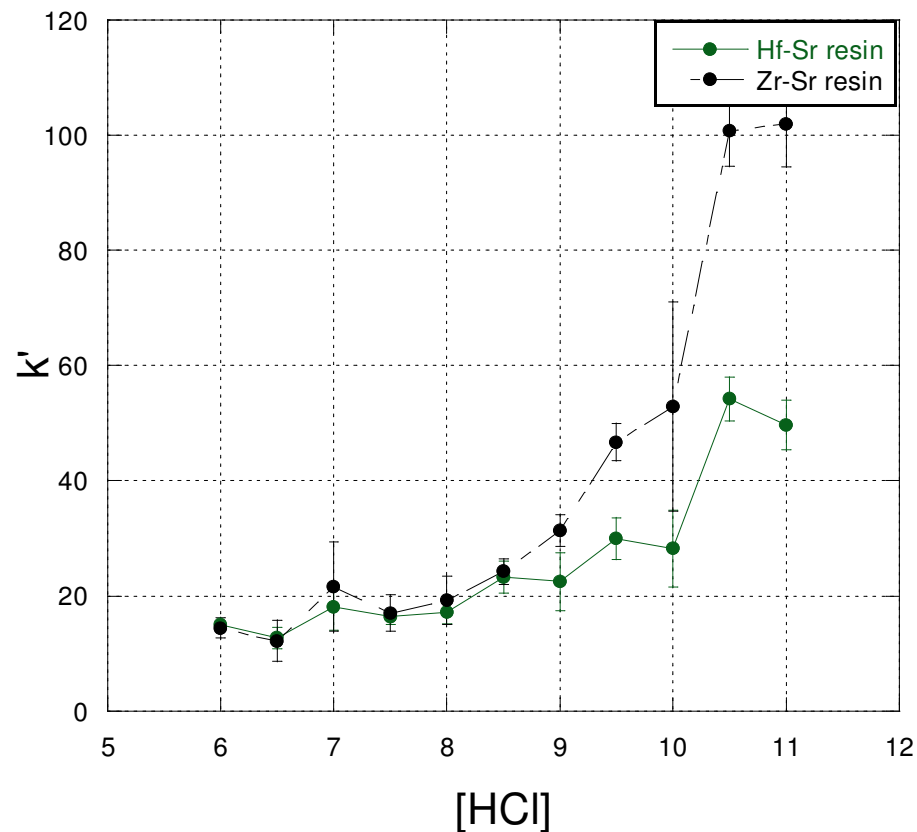
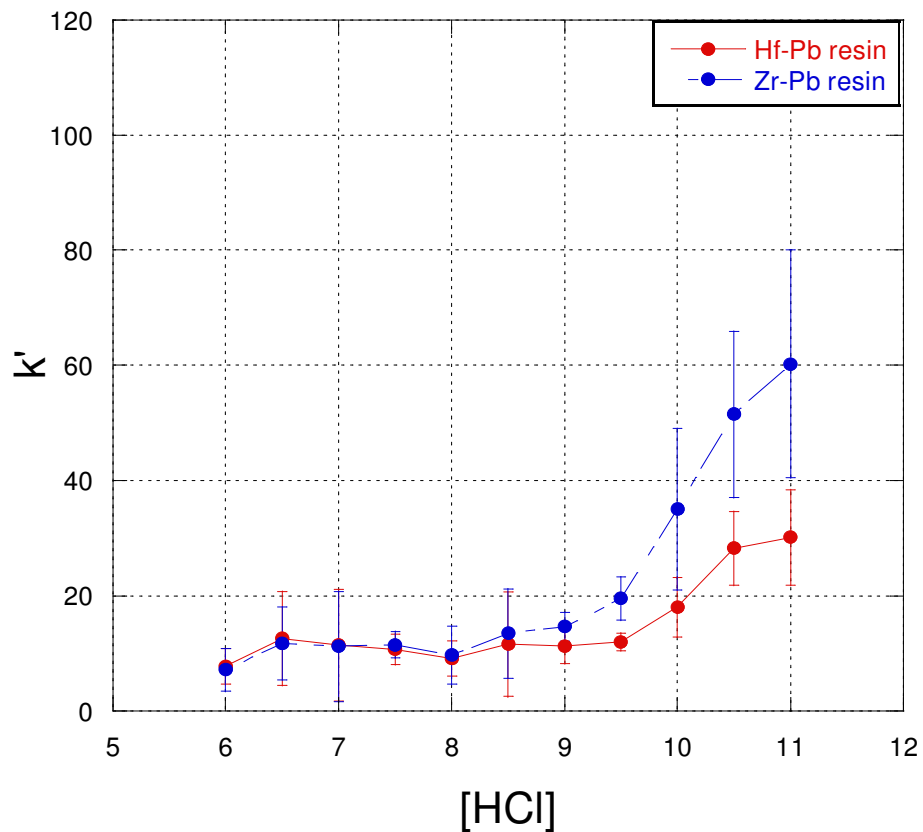
$$D_w = \frac{A_r}{m_r} \div \frac{A_s}{v_s}$$

$$A_r = A_o - A_s$$

²Horwitz, E.P., Chiarizia, R., Dietz, M.L., Solvent Extr. Ion Exch. **10**, 313-336 (1992)

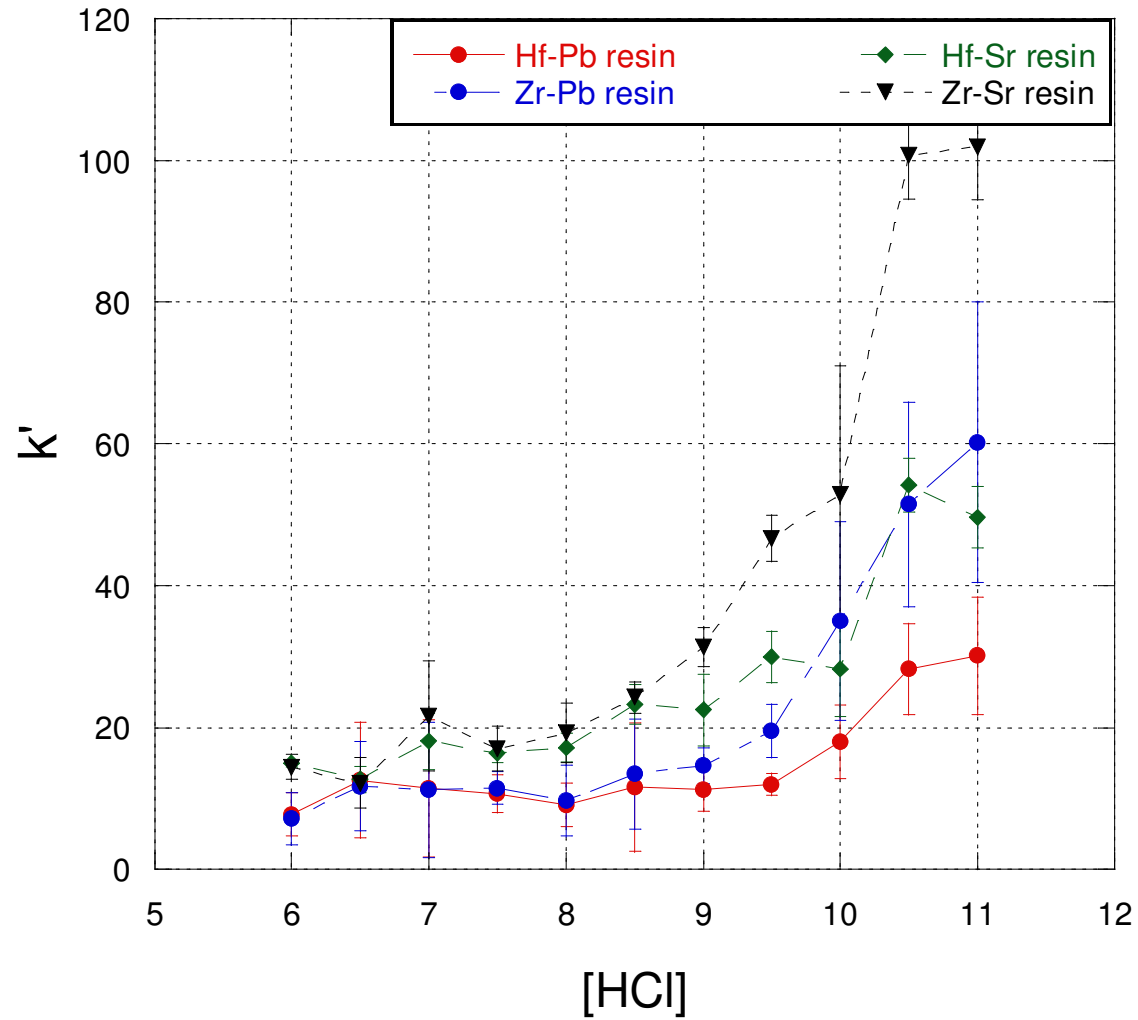
³Eichrom Website: "Extraction Chromatography of Actinides and Selected Fission Products: Principles and Achievement of Selectivity" August 2008

Batch Results



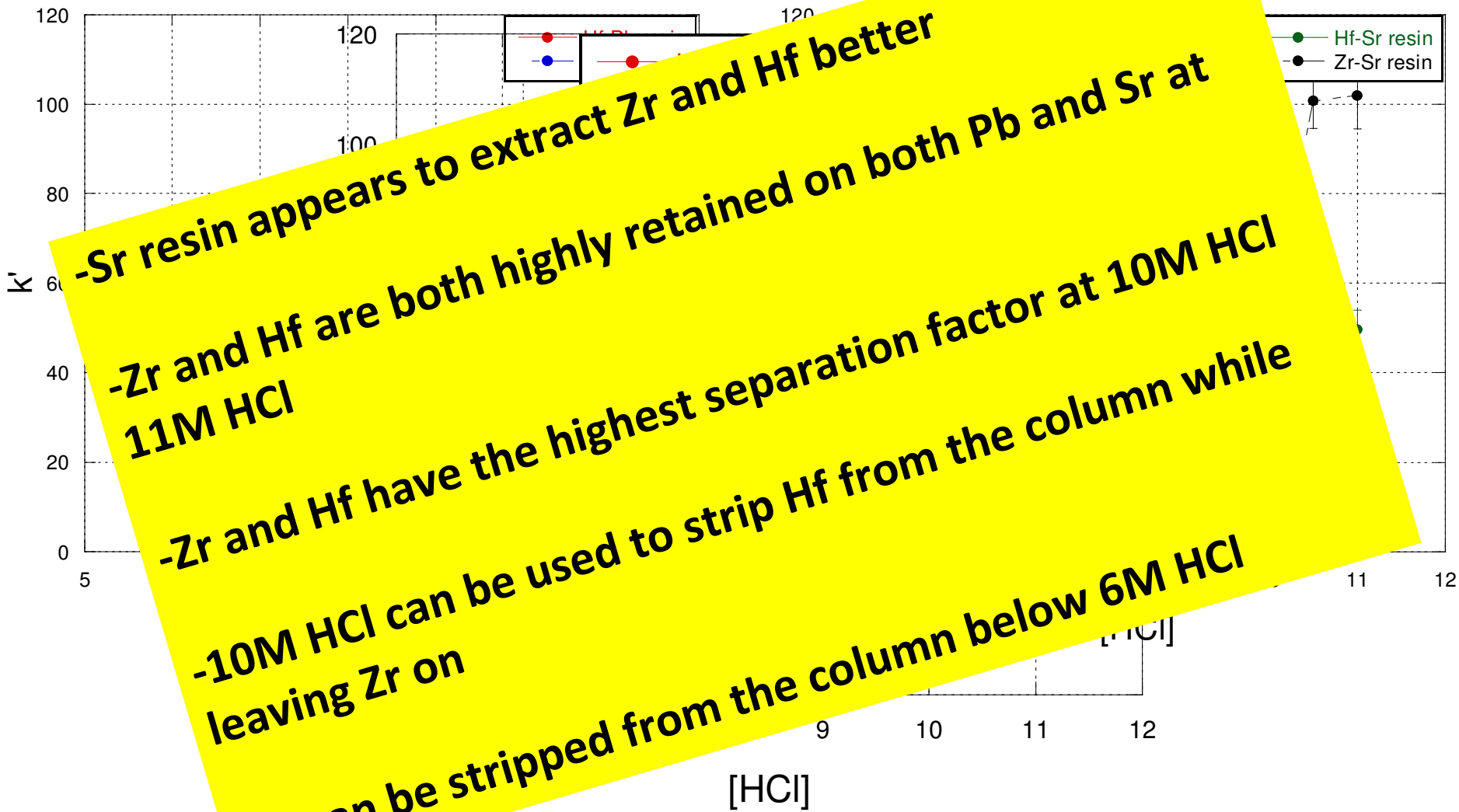
Error bars are standard deviation of 3 trials

Batch Results



Error bars are standard deviation of 3 trials

Batch Results



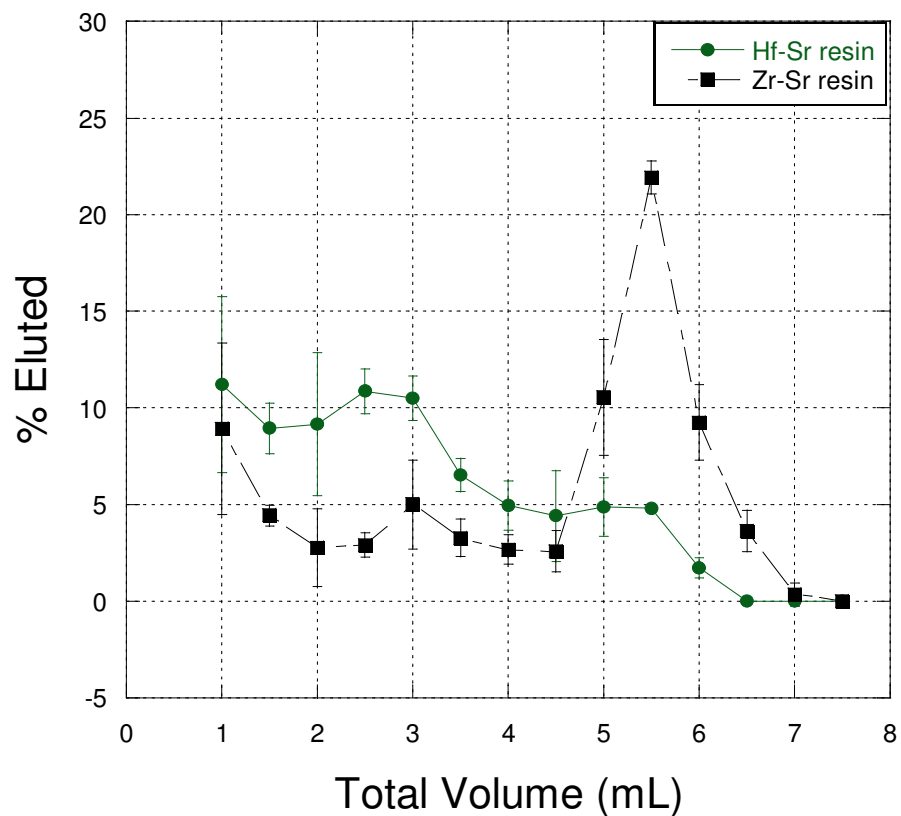
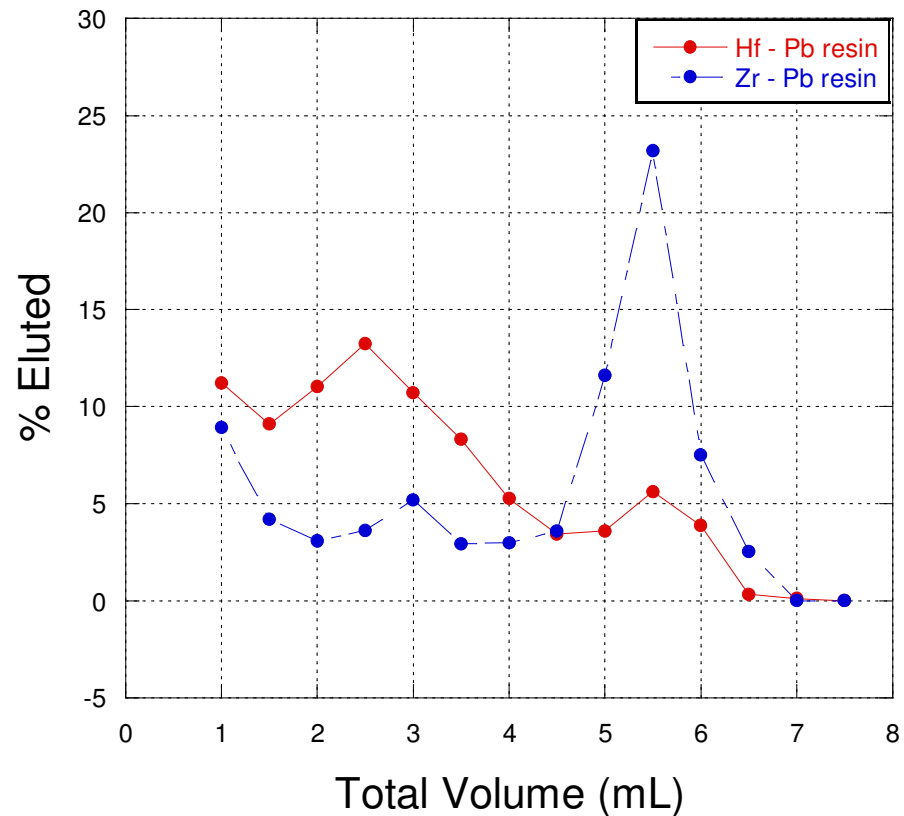
Error bars are standard deviation of 3 trials

Column Studies

- $\sim 3\text{cps } ^{95}\text{Zr}$ and $\sim 2\text{cps } ^{175}\text{Hf}$ placed in a PPE 15mL centrifuge tube
- Samples evaporated to dryness, reconstituted in 11M HCl
- Samples counted using HPGe gamma spectroscopy for 30 minutes
- Columns conditioned with 5mL 11M HCl
- Columns loaded
- Hf eluted with 3mL 10M HCl
- Zr eluted with 3mL 3M HCl
- Each fraction evaporated to dryness, reconstituted in 1mL acid then counted for 30m using HPGe gamma spectroscopy

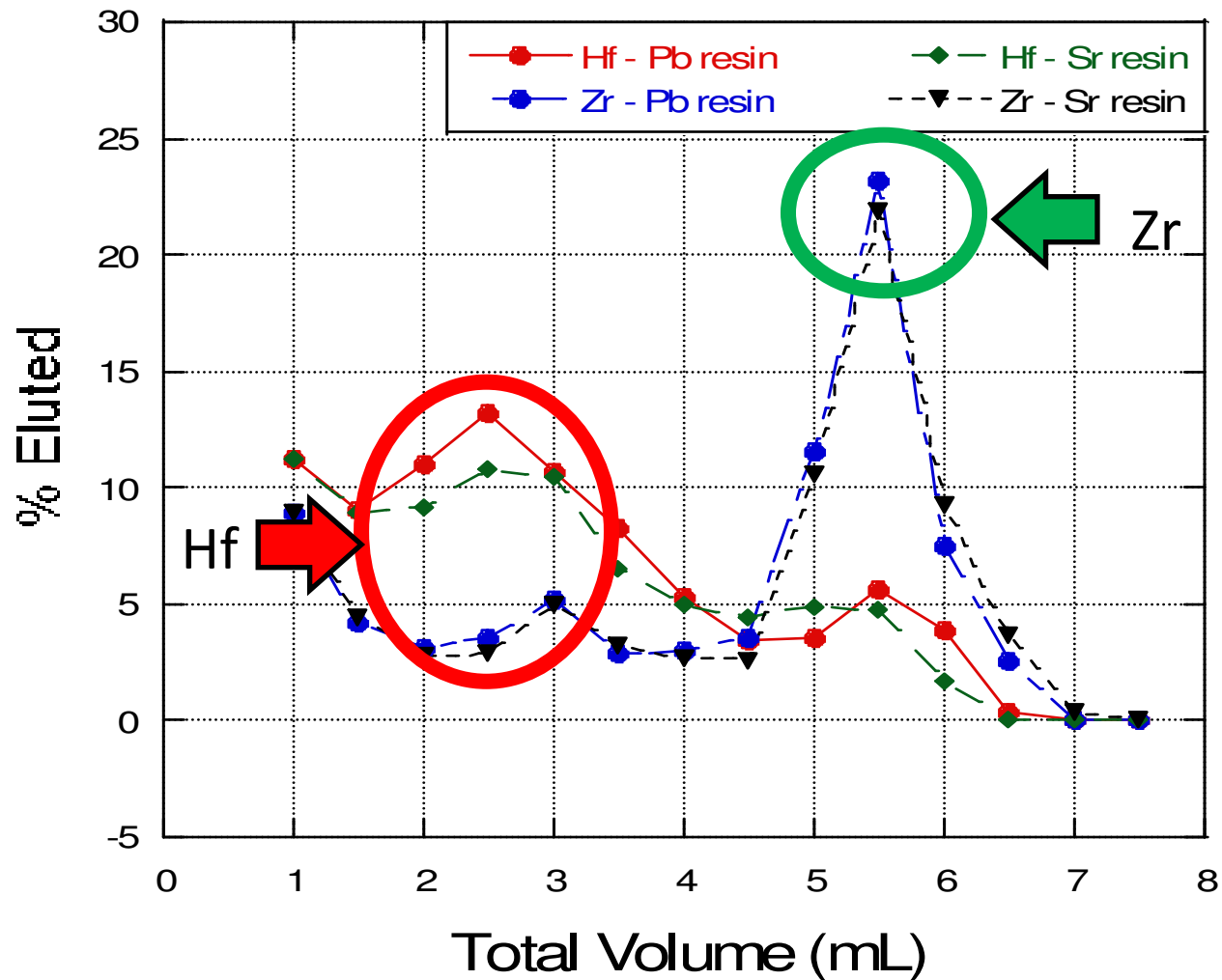


Column Results



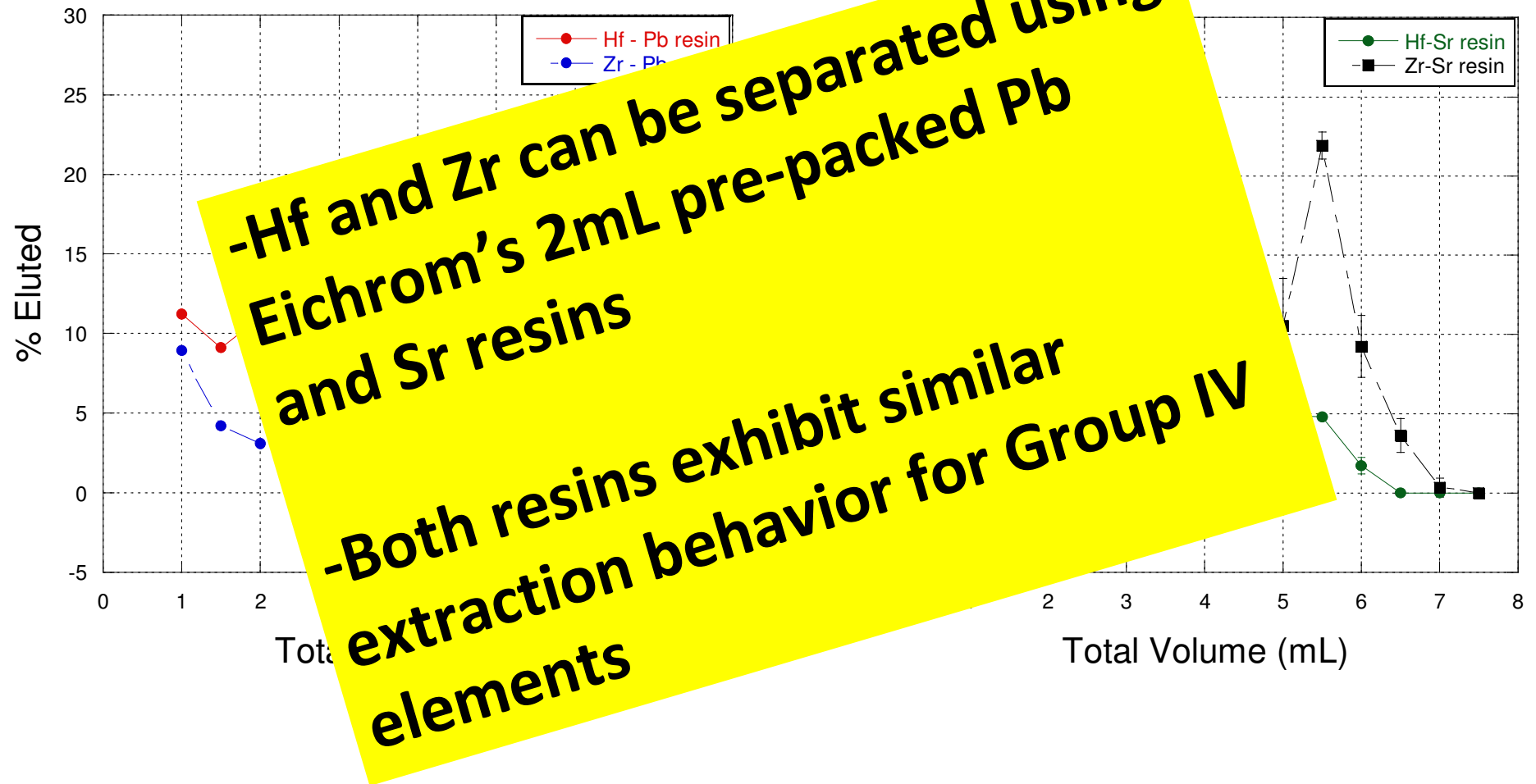
Error bars are standard deviation of 3 trials

Column Results



Error bars are standard deviation of 3 trials

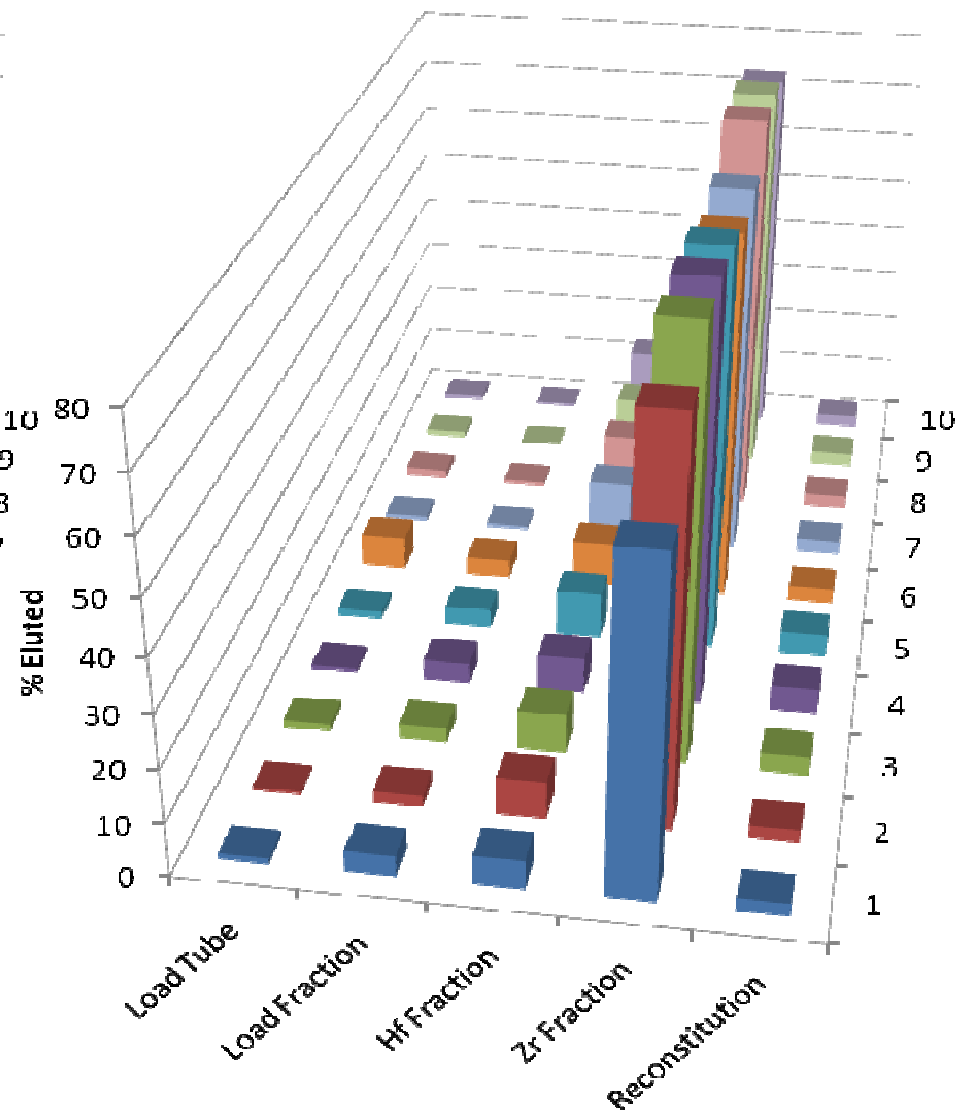
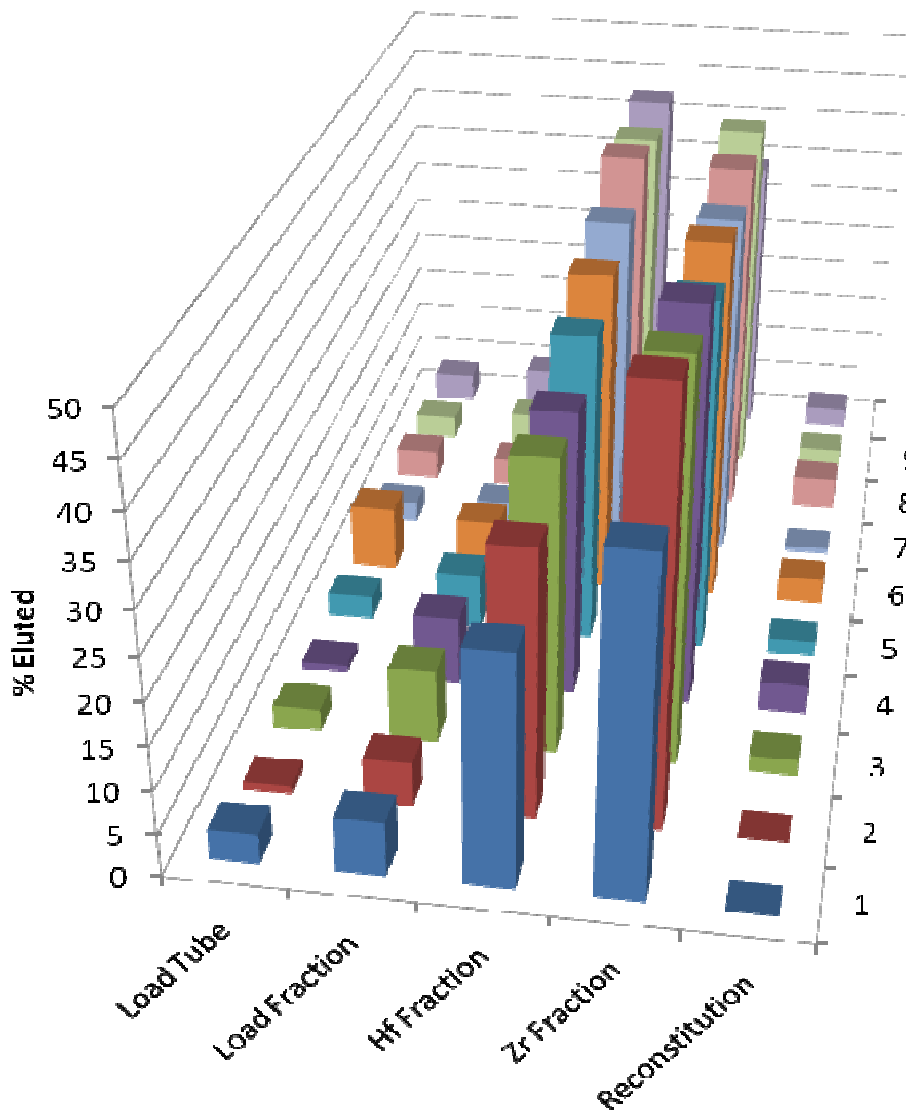
Column Results



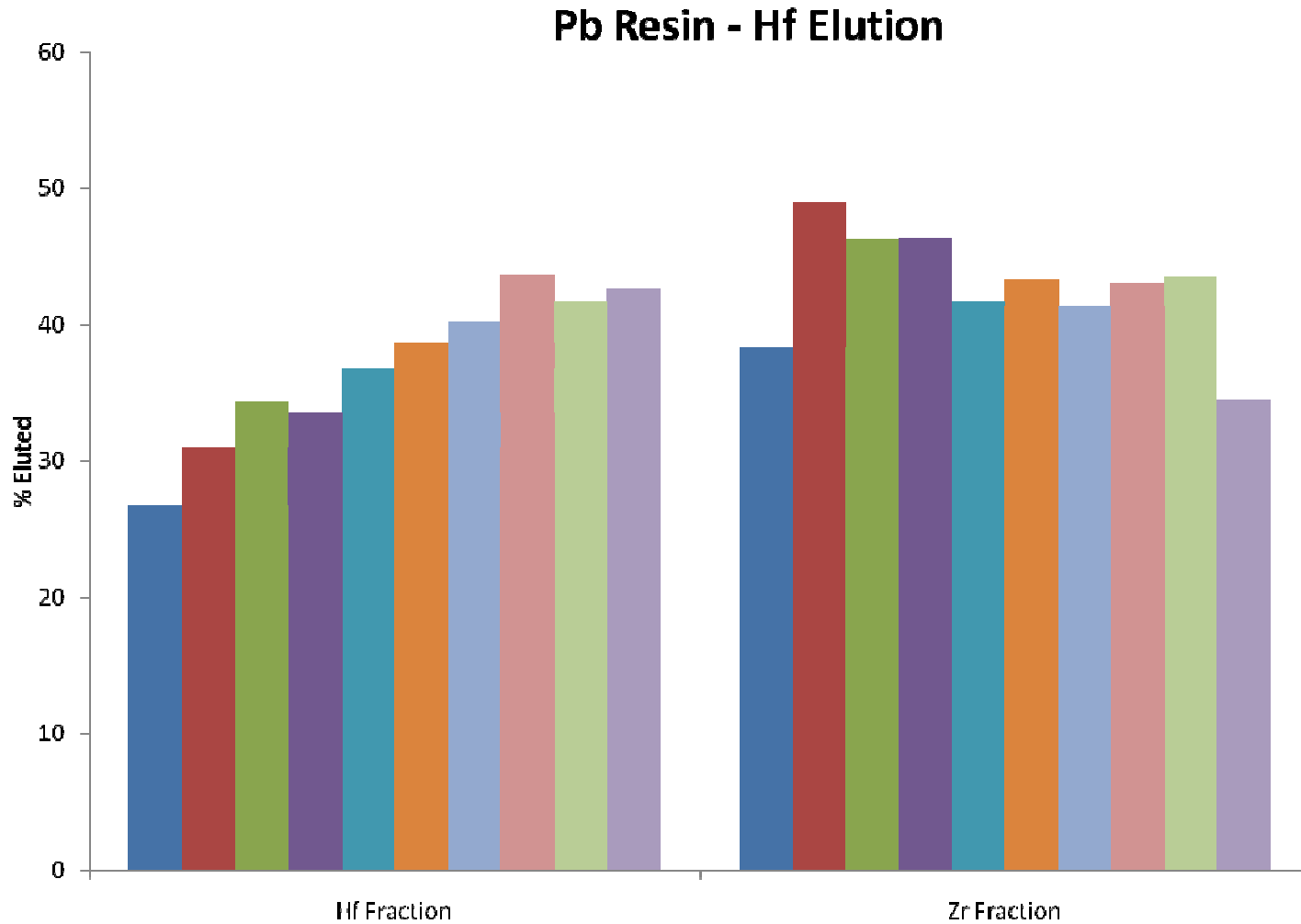
Pb Resin Reusability

Pb Resin - Hf Elution

Pb Resin - Zr Elution

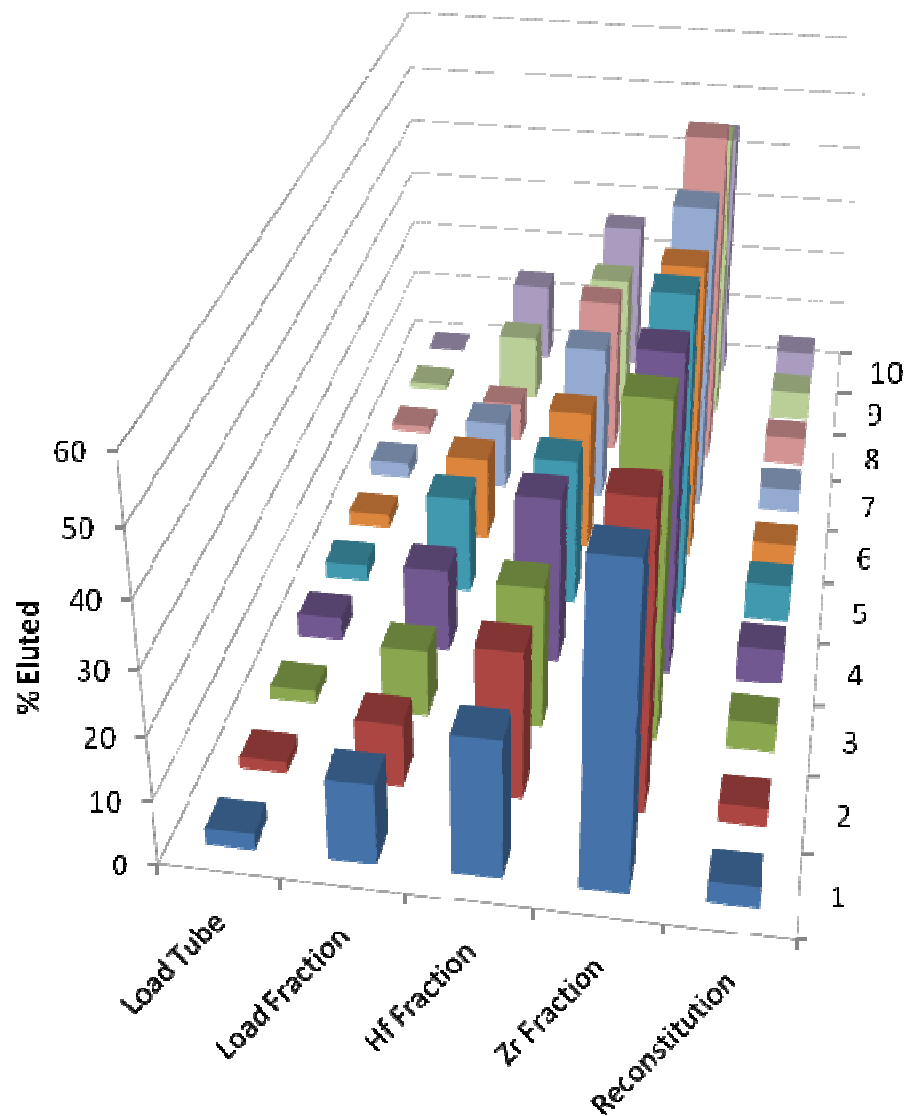


Pb Resin Reusability

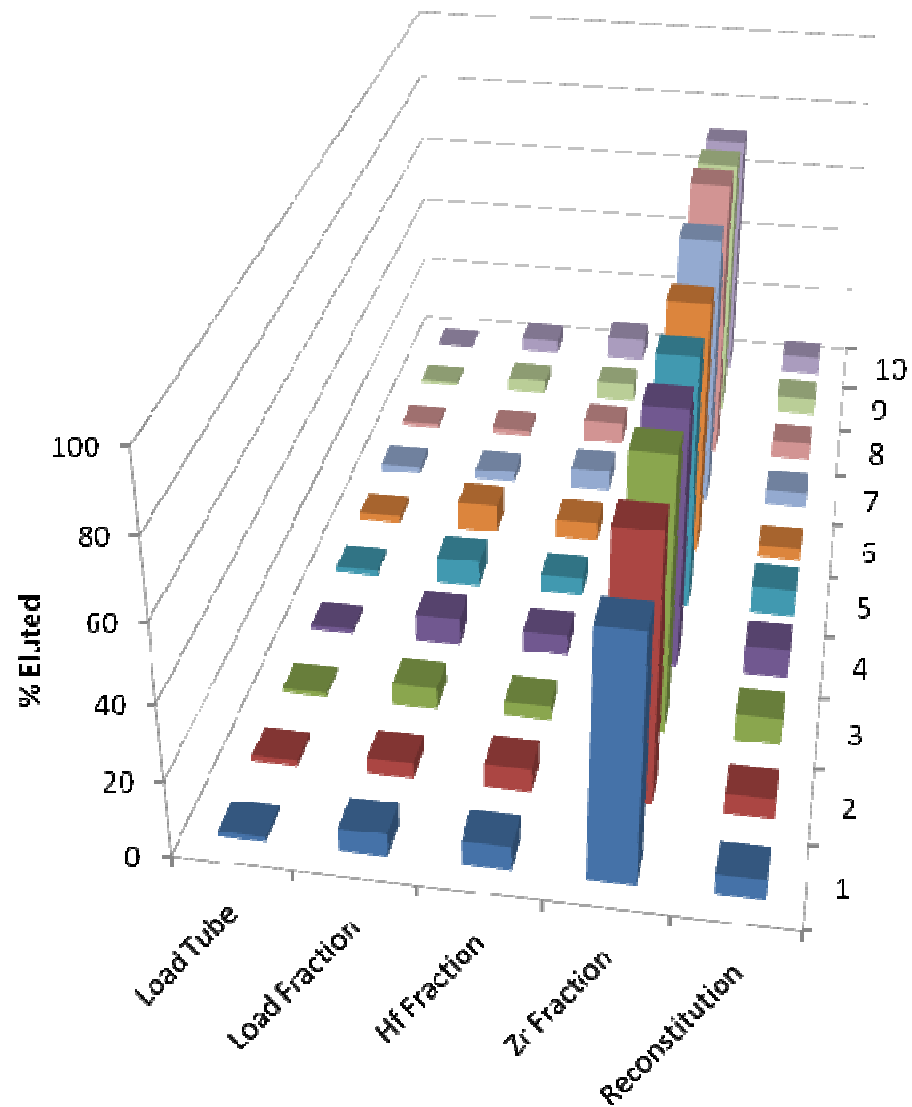


Sr Resin Reusability

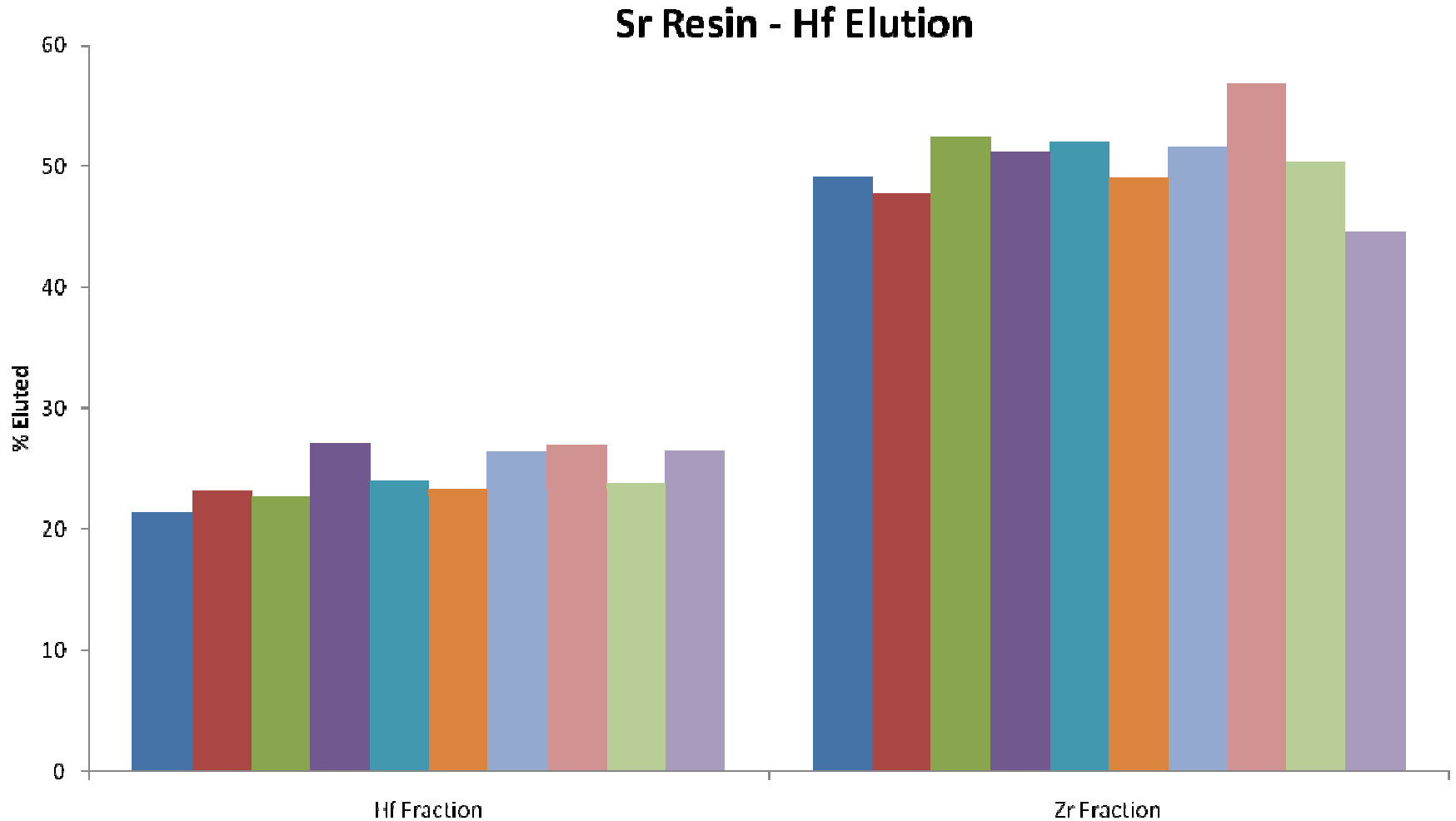
Sr Resin - Hf Elution



Sr Resin - Zr Elution



Sr Resin Reusability



Conclusions

- Group IV sorption onto crown ether based resins has been studied for TAn applications
- A separation procedure for Group IV elements using Eichrom's Pb and Sr resins has been established
 - 11M HCl load solution, Hf elution with 10M HCl, Zr elution with 3M HCl
- Each resins cannot be re-used.

Future Work

- Elution of Hf with 5mL of 10M HCl
 - If successful do a reusability study
- Extend the crown ether resin work
 - ~0.75M DC-18-C6 in 1-octanol
 - ~0.75M DC-18-C6 in isodecanol
 - ~0.35 M DB-18-C6 in 1-octanol
 - ~0.35M DC-18-C6 in 1-octanol
- Bleeding studies over the course of 5 weeks

Acknowledgements

- Tom O'Dou, Trevor Low, Julie Bertoia, Mary Turner

