Extraction Chromatographic Studies of Rf Homologs Using Crown Ether Based Resins

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Separation Requirements

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

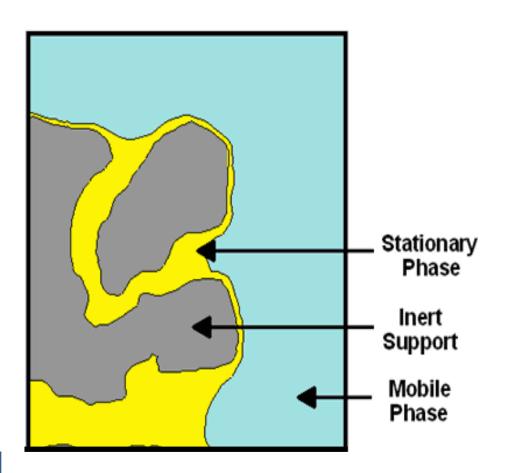
Extraction Chromatography fulfills all of these





Extraction Chromatography¹

Surface of Porous Bead



$$D_{w} = \frac{A_{r}}{m_{r}} \div \frac{A_{s}}{v_{s}}$$

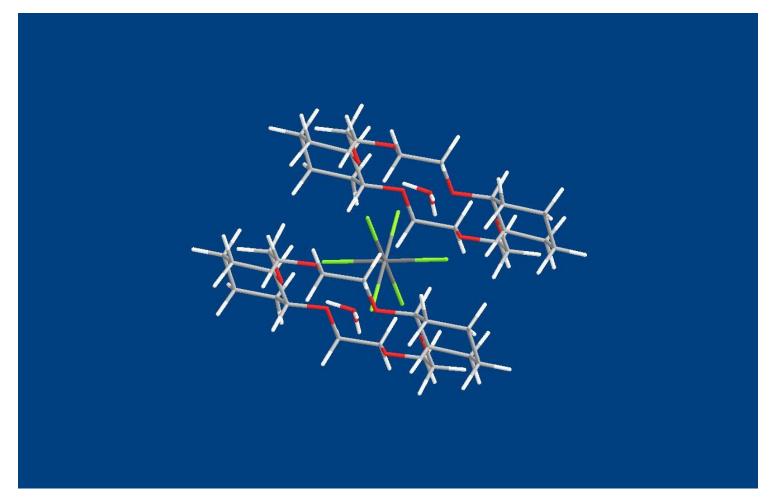
$$A_r = A_o - A_s$$

http://www.eichrom.com/products/extraction.cfm November 2009





Why Crown Ethers?



Courtesy of Ralf Sudowe





Previous Work – Commercially Available Resins

- No difference in separation of Zr and Hf due to:
 - [Crown Ether]
 - Solvent (octanol and decanol)





In House Synthesized Resins

In house DC18C6 Resin

- ~0.75 M DC18C6
- Various solvent
- Free resin

In house DB18C6 Resin

- ~0.75 M DB18C6
- dichloromethane solvent
- Free resin

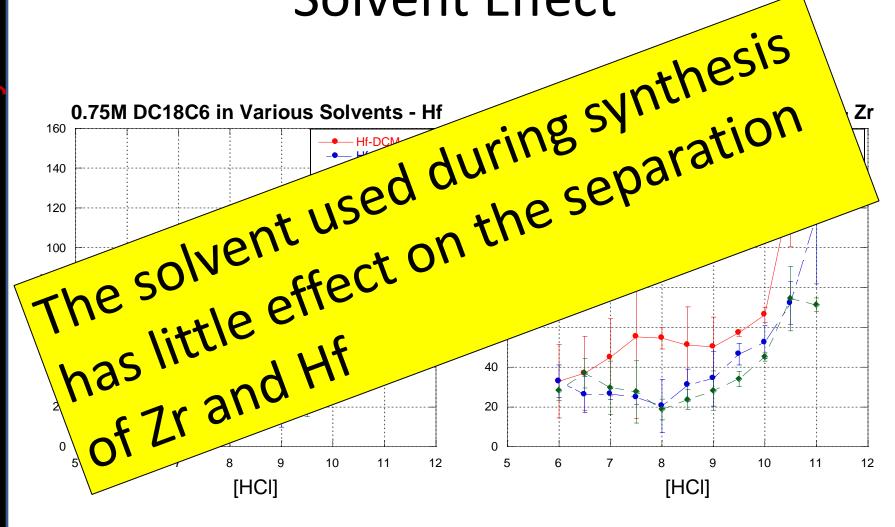




Solvent Effect



Solvent Effect





U.S. Department of Energy

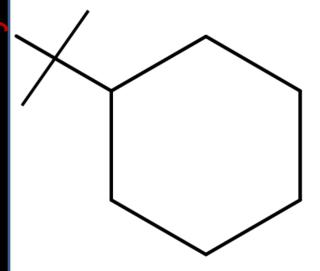


Functional Group Effect



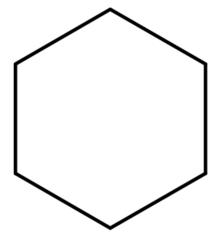


Functional Groups



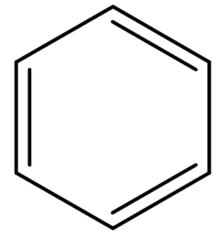
Eichrom's Pb Resin

- ~0.75 M DtBC18C6
- 1-Octanol solvent
- Free resin
- •2 mL pre-packed



In House DC18C6 Resin

- ~0.75 M DC18C6
- DCM solvent
- Free resin
- •2 mL pre-packed



In House DB18C6 Resin

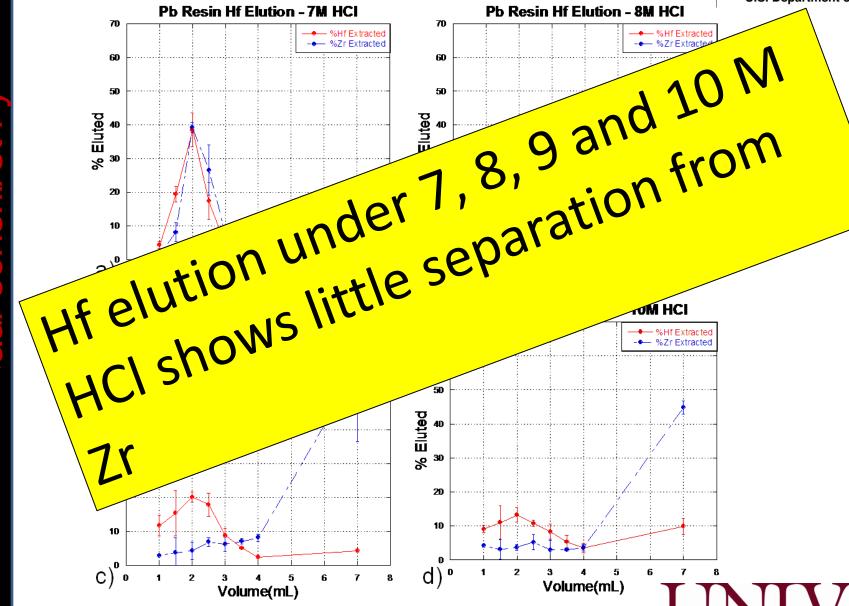
- ~0.75 M DB18C6
- DCM solvent
- Free resin
- •2 mL pre-packed



Error bars are standard deviation of 3 trials

Eichrom's Pb Resin





Error bars are standard deviation of 3 trials

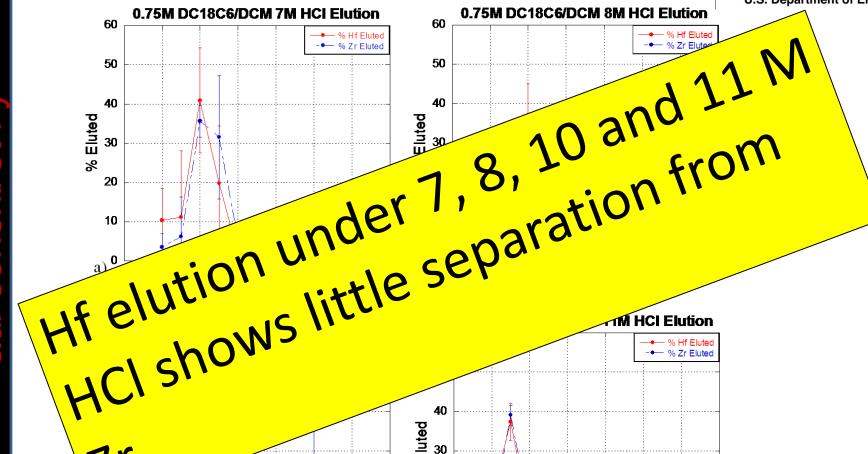
ACS National Meeting Fall 2011

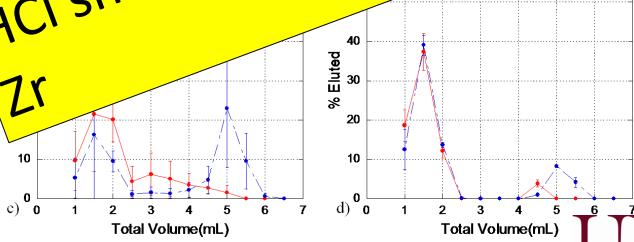
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DC18C6 Resin







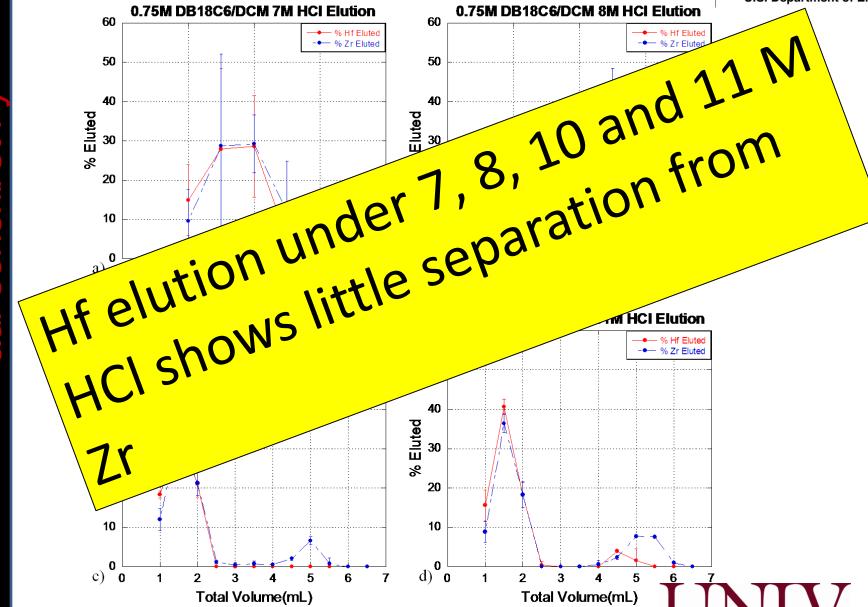
ACS National Meeting Fall 2011

Error bars are standard deviation of 3 trials

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DB18C6 Resin





Error bars are standard deviation of 3 trials

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Conclusions

 Solvent has little effect on separation of Zr and Hf

 0.75 M DtBC18C6, DC18C6 and DB18C6 show no promise for application to Rf chemistry

 Most likely due to the crown's inability to form the needed sandwich structure on the resin



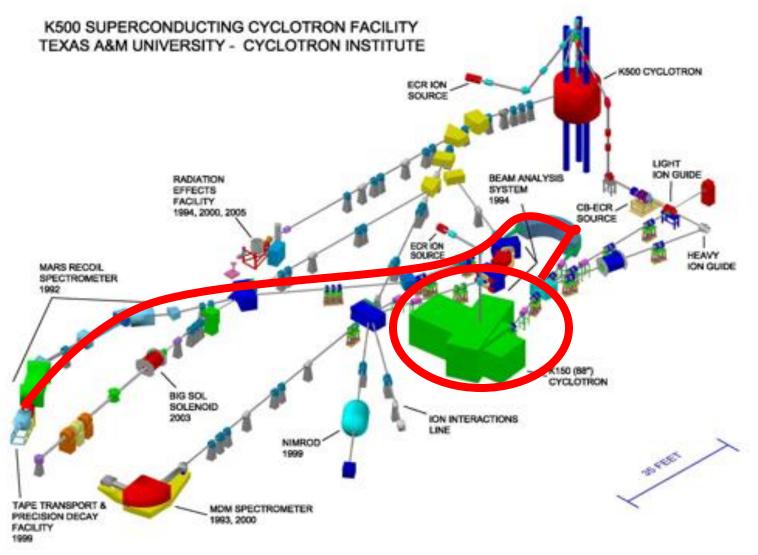
Future Directions





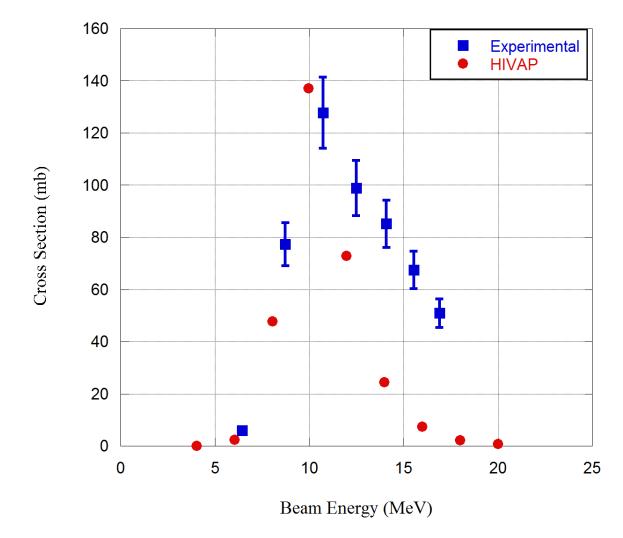


Why Texas A&M





Large Scale Production of ¹⁷⁵Hf



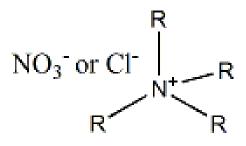






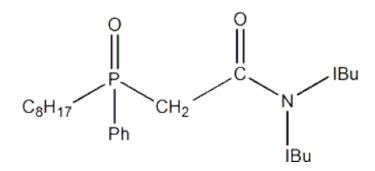
Future Chemical Systems

- Eichrom's TEVA Resin
 - Quaternary amine
 - HCl and HNO₃solvents



$$R = C_8 H_{17} \text{ or } C_{10} H_{21}$$

- Eichrom's TRU Resin
 - CMPO
 - HCl and HNO₃solvents



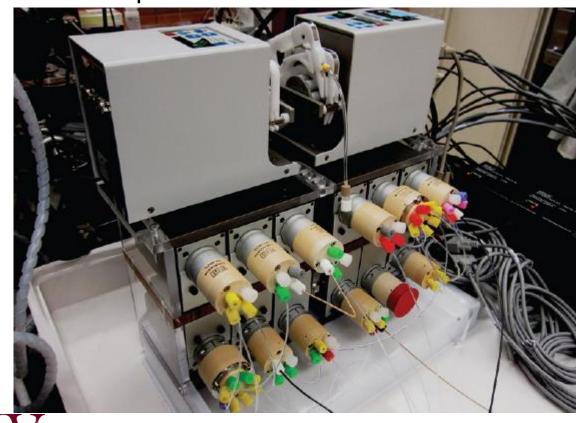






Off-line to On-Line Chemistry

If any of the extraction systems exhibit large separation factors of $^{95}Zr/^{175}Hf$ automated separations will be conducted with accelerator produced ^{85}Zr and ^{169}Hf

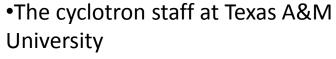




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