

Synthesis of Crown Ether Rotaxanes

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Introduction

Heavy Metal Cations

- Harmful to the human body as they turn into free radicals and oxidize cells throughout.
- Oxidized cells increase blood acidity and raise lactic acid levels, causing numerous maladies such as cancer.

Critical Need

- A molecule that can sense heavy metal cations in the environment, and possibly trap and remove them.

Crown Ether Rotaxanes

- 18-crown-6 rotaxanes are a new class of molecules that bind to metal cations.
- Consists of a rod shaped molecule (axle), that is threaded through a circular molecule (wheel), which is then locked in place with DCC forming a “pseudo-rotaxane”.
- Crown ether blocking group helps to provide cation selectivity.
- The research conducted derivatized the wheel to enhance the selectivity for heavy metal cations as shown below.

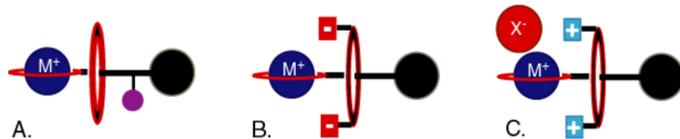


Figure 1: 18-crown-6 rotaxane derivatives.

Methods

Three derivatives of the 18-crown-6 rotaxane (18c6) were synthesized to form rotaxanes that included an acetyl group on the axle (A: Acetyl Rotaxane 18c6), a carboxyl acid group on the wheel (B: Acid-CEHR Rotaxane), and an Arginine group on the wheel (C: Arg-CEHR Rotaxane). Synthesis of the rotaxane requires a multistep approach. ¹H-NMR analyses were used to assess the structures of the intermediate components and final rotaxane.

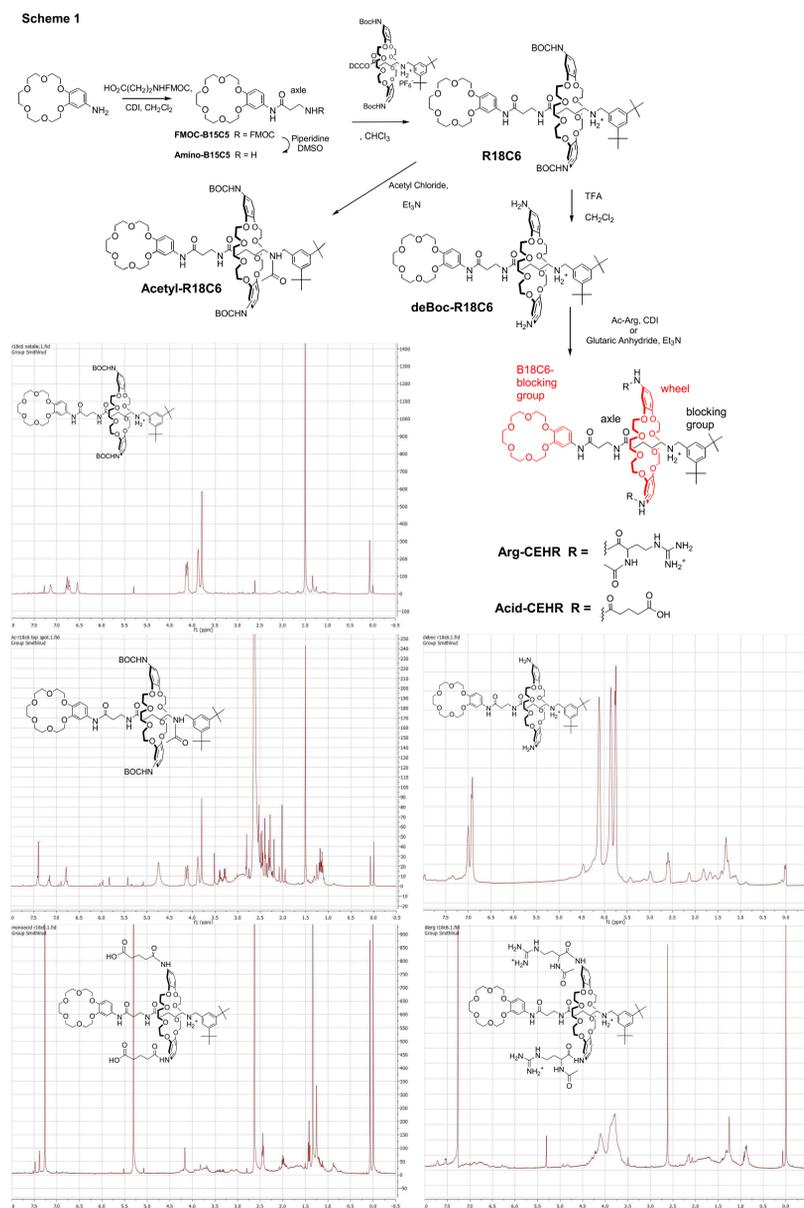


Figure 2: 18c6 Rotaxane, Acetyl Rotaxane 18c6, deBoc Rotaxane 18c6, Acid-CEHR Rotaxane, and Arg-CEHR Rotaxane synthesis scheme and corresponding ¹H-NMR spectroscopy analyses.

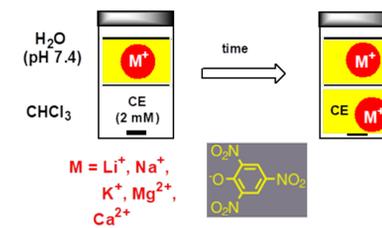
Results

The ¹H-NMR analyses in Figure 3 help verify that the 18-crown-6 rotaxane and its derivatives (Acetyl Rotaxane 18c6, deBoc Rotaxane 18c6, Acid-CEHR Rotaxane, and Arg-CEHR Rotaxane) have successfully been made.

Conclusions

Four crown ether rotaxanes have been synthesized and authenticated by ¹H-NMR spectroscopy. The ability to bind metal cations will be investigated using transfer assays.

Transfer into CHCl₃ Assays



Future Work

Next Steps

- Transferring metal cation-anion complexes into chloroform.

Long Term Goals

- Make sensors for toxic metal cations.

References

Wang, X.; Zhu, J.; Smithrud, D. B. “Investigation of New Host-[2]Rotaxanes that Bind Metal Cations”, *J. Org. Chem.* 2010, 75, 3358-3370.

Acknowledgments

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