

Synthesis of a Cyclam Precursor en Route to CO₂ Reduction Catalysts

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Introduction:

metal cations some of which are able to act as electrocatalysts propane was carried out successfully. The for CO₂ reduction. The focus of this project was to synthesize the backbone of the cyclam ligand, 1, 3-bis(2-aminoethylamino) propane, "tet-2,3,2," ² as it is relatively expensive to purchase. Eventually, this molecule will be transformed into a Cfunctionalized cyclam, bearing a linker/anchor group. The transition metal cyclam catalysts can then be immobilized on semiconductor surfaces for light-driven CO₂ reduction to generate carbon monoxide as a solar fuel precursor.



Figure 1. Desired product 1,3-bis (2-aminoethylamino) propane. Experimental Design:

In a simple one step synthesis, ethylenediamine, ethanol, and 1,3-dibromopropane, were combined with excess potassium hydroxide under constant temperature. The product was then vacuum filtered. Ethylenediamine and ethanol impurities were removed by vacuum distillation. The final product was collected by a second vacuum distillation at 130°C.²



Figure 2. Synthetic route to1,3-bis(2-aminoethylamino)propane, "tet-2,33."

Results & Discussion:

A cyclam N₄-macrocycle is known to bind strongly to transition The synthesis of 1,3-bis (2-aminoethylamino) percent yield obtained was 31.8%. The H NMR and C NMR show characteristic peaks confirming the product was obtained in high purity.





Future Work:

Modifications could be made to the experimental conditions to improve the yield of the tetra amine. Continuing with the synthesis of cyclam ligand by attaching the metal center and linker groups to reduce CO₂ is also another possibility for future work.



Figure 3. N₄-macrocycle linked to a semi-conductor nanoparticle Conclusion:

The desired "tet 2,3,2" was successfully and efficiently synthesized. Although percent yield was lower than the 50% literature value, the H NMR and C NMR suggest the compound was obtained in high purity according to the synthetic method entailed in this project.

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References:

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