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INTRODUCTION

This volume provides abstracts of research work performed by some of the undergraduate students working in chemistry during summer, 2006. The majority of these participants worked under the auspices of the Research Experiences for Undergraduates (REU) Program in Chemistry sponsored by the National Science Foundation (NSF). This 10-week program provides undergraduate students an intensive hands-on research experience that involves them in all phases of the research process. In carrying out their projects, REU participants work alongside Stony Brook's faculty, post-docs, graduate students and other undergraduates. This year, nine summer researchers from as many college campuses were selected for the REU program, from a pool of ~ 200 applicants. The REU summer activities culminate in these research presentations being made to members of the Stony Brook Chemistry Department and the university community on August 4, 2006. The REU students are joined in this symposium by Stony Brook student colleagues.

As you read this collection of the students' abstracts, you will see evidence of their hard work, insight and enthusiasm. You will be impressed at what they have accomplished in only ten weeks. I am confident that these individuals will proceed on to successful research careers.

Robert Kerber
Distinguished Teaching Professor
NSF REU Chemistry Site Co-Director
Stony Brook University

Jennifer Beck
Lock Haven University

Amino Acid-Bearing ROMP Polymers from Bicyclic Compounds

Jennifer Beck, Lock Haven University of Pennsylvania; Kathlyn Parker, Department of Chemistry, Stony Brook University

Ring-opening metathesis polymerization (ROMP) is a synthetic strategy to prepare macromolecules consisting of specific ligands. The ligands, in this case, are peptides effective for interacting sperm to egg receptors for biological chemistry. Ring strain in the monomers ensures the completion of the reaction by pushing the equilibrium process in ROMP towards the formation of the polymers. Different substitution characteristics can also vary the qualities of the product due to stereochemical problems. In addition, ROMP is under thermodynamic control and produces linear material.

Our goal was to synthesize three cycloalkene carboxylic acid monomers and convert them to amino acid-bearing monomers by means of the modified synthetic route with oxalyl chloride. A bicyclooctene carboxylic acid was synthesized, and a bicyclihexene compound made substantial progress in its synthesis of the carboxylic acid. The plan is to convert the carboxylic acid monomers to amides, and then to polymerize the amino acid-bearing monomers with ROMP. Procedures for these monomers were found in literature and prepared using negligible adjustments. The NMR and Infrared spectroscopy instruments were used to verify the purified products. The polymers will later be used for bioassays to determine the valency and spacing of the egg receptor interaction. The National Science Foundation funded the research for this experiment in the Research Experience for Undergraduate program.

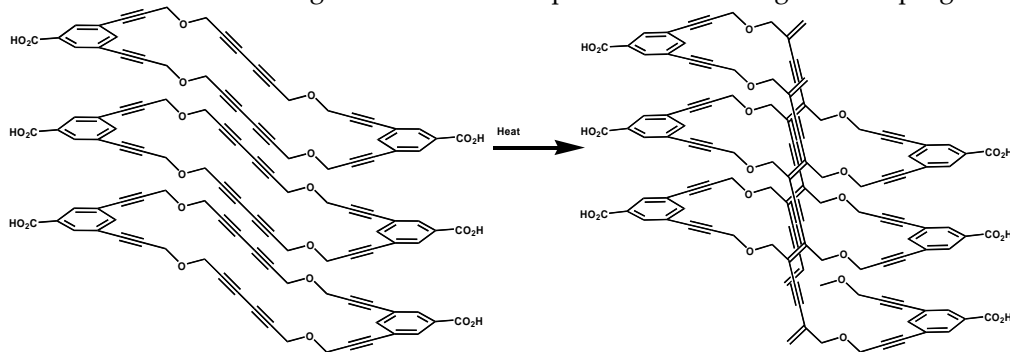
Nathan Burrows Concordia University

Directed Synthesis of a Target Nanotube

Nathan Burrows, *Concordia University, St. Paul, MN*; Zhong Li, Steven Chow, Frank W. Fowler, and Joseph W. Lauher, *Department of Chemistry, Stony Brook University*

Carbon nanotubes are of great research interest because of the many outstanding physical and chemical properties they possess. They are among the stiffest and strongest fibers known, and have remarkable electronic properties and many other unique characteristics. They have an incredible tensile strength of fifty times higher than steel, and yet are much lighter than steel. Nanotubes can also be metallic or semi-conducting depending on their structure. Thus, some nanotubes have conductivities higher than that of copper, while others behave more like silicon. There is great interest in the possibility of constructing nanoscale electronic devices from nanotubes. There are several areas of technology where carbon nanotubes are already being used. These include flat-panel displays, scanning probe microscopes and sensing devices. The unique properties of carbon nanotubes will undoubtedly lead to many more applications. For these reasons they have attracted huge academic and industrial interest, with thousands of papers on nanotubes being published every year. Commercial applications have been rather slow to develop, however, primarily because of the high production costs of the best quality nanotubes. Currently nanotubes can be produced through two methods: arc-evaporation or catalytic growth. However both these methods create single and multiple walled nanotubes of varying diameter all together as a mixture that is extremely difficult to separate and in less than desirable yields. The biggest difficulty in these processes is a lack of control over what nanotubes are constructed.

A directed and controlled method for the synthesis of single walled nanotubes of exact dimensions would be very desirable but difficult. However analogs of nanotubes could be achieved by polymerization of cyclic systems that contain diacetylenes. Besides the issue of synthesizing the cyclic system, organizing these rings for topochemical polymerization would prove difficult however for a new host-guest approach. The host-guest approach is a co-crystal in which the guest molecule is held in arrangement by the host molecule that provides the necessary repeat layering of approximately 5.0 Å with stacking angle of ~ 45° required for polymerization by thermal annealing, UV light and/or Gamma Rays. The current research is to expand this approach from linear diacetylenes to cyclic systems that contain diacetylenes to provide a systematic approach to creating designed nano tubes. Previous host-guest strategies involve hydrogen bond between carboxylic acid and pyridine functional groups on the host molecule an oxalamide derivative, a guest molecule that provides the desired repeat distance. With this in mind, a strategic retro synthesis of a diacetylene macro cycle was developed to include a carboxylic acid on the poles of the macro cycle. Herein is a report on the progress on the synthesis of these macro cycles. This work was funded by the National Science Foundation through their Research Experience for Undergraduates program.



Jean Calderón
University of Puerto Rico

Time-Resolved In Situ Water Gas Shift Reaction Using Ni-MoO₂ as Catalyst

Jean E. Calderón, *University of Puerto Rico at Cayey, Cayey, Puerto Rico*; José A. Rodríguez, Wen Wen, Jonathan C. Hanson, *Department of Chemistry, Brookhaven National Laboratory, NY*

The hydrogen appears as a new source of energy for the future. The Water Gas Shift (WGS, $\text{CO} + \text{H}_2\text{O} \rightarrow \text{H}_2 + \text{CO}_2$) is one of the reactions that the scientific community is examining for the production of pure H₂. To do the WGS reaction faster and with cleaner production of H₂ this process needs a catalyst.

The compound NiMoO₄ was studied as a precursor of catalysts for the Water Gas Shift (WGS) reaction. To obtain a highly active WGS catalyst, the NiMoO₄ compound was partially reduced with H₂ increasing the temperature from 25 to 700 °C in one hour. This process was monitored with the technique of *in-situ* Time-Resolved X-ray diffraction (TR-XRD). With TR-XRD it was found that during this synthesis, around 350 °C, part of the Mo oxide in the compound collapsed and Mo and NiO-Ni were produced in the sample. During the WGS reaction, it was identified the diffraction pattern of Ni-MoO₂ around 400 °C showing that Mo and NiO transformed with the WGS reaction. Around 500 °C the compound produced (Ni-MoO₂) exhibited high activity for the WGS. The same compound (Ni-MoO₂) was used in another trial of the WGS reaction showing great activity and stability at temperatures of 350, 400 and 500 °C.

The key compound of this activity was the mix of MoO₂ with metallic Ni in the mixture. The WGS reaction was investigated on MoO_x compounds (MoO₂ and MoO₃) and for none low or high temperature activity was found. Also with the MoO_x trials for the WGS reaction it was showed that MoO_x compounds are stable under reaction conditions.

Acknowledgement: This research was supported by the US Department of Energy.

Alex Ceballos
Stony Brook University

Substituent Effects on the Stereoselectivity of the 6π Electrocyclic Closure of 1,3,5-Cyclooctatriene to Bicyclo[4.2.0]Octadiene

Alexander Ceballos, *Stony Brook University*; and Nancy Goroff, *Department of Chemistry, Stony Brook University*

Heavily substituted cyclooctatrienes are key intermediates of certain biomimetic pathways where a linear octatetraene will undergo two thermal electrocyclic closures to give a bicyclo[4.2.0]octadiene. This product is the backbone for an interesting spread of natural products; classically, the endiandric acids; and on the forefront, the immunosuppressants SNF4435 C/D. This computational study lays the groundwork for a general understanding of how substituents affect the stereoselectivity of the 6π electrocyclic ring closing of cyclooctatriene (Figure 1). Our current method tests different placements of methyl substituents on the ring. Figure 2 shows compounds modeled by applying a substitution scheme to each parent. The parent cyclooctatrienes are methyl substituted on the sp^3 carbons. The derivatives vary the placement of a methyl group across the conjugated carbons. This is a form of spot-checking for stereoselective activity. The product bicyclo[4.2.0]octadienes can occur as either an 'exo' or 'endo' stereoisomer. 27 methyl substituted cyclooctatrienes were modeled as were their isomeric transition states and products. For each reaction, we attempt to correlate stereoselectivity with steric hindrance during reaction or with substituent crowding on the compact bicyclic product. Geometry, energy and vibrational frequencies were calculated according to Density Functional Theory at the B3LYP/6-31G* level within the program Spartan M04. These electrocyclic closings tend to be more exothermic as more substituents are placed on the ring. Also, various trends have been identified concerning relative stabilities. This project is supported by the NIH MARC program, grant #T34-GM08655.

Figure 1.

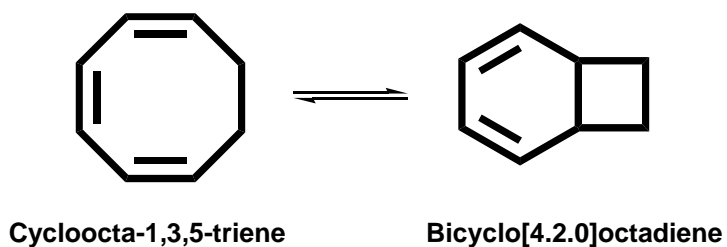
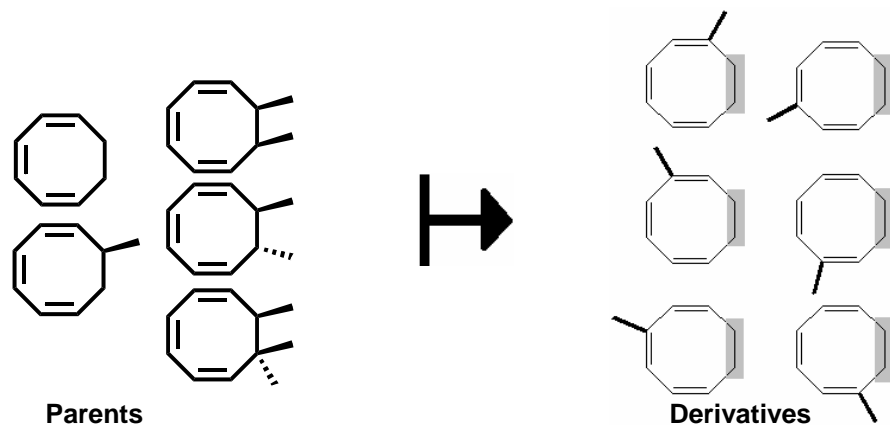


Figure 2.



Ricky Chachra
Stony Brook University

Targeting Influenza with Computational Structure-based Drug Design

Ricky Chachra, *Stony Brook University*; and Robert C. Rizzo, *Department of Applied Mathematics & Statistics, Stony Brook University*

The rapid spread of avian influenza (strain H5N1, “bird flu”) coupled with its high mortality rate and potential for human to human transmission is alarming to health officials who fear a devastating pandemic is inevitable. Although no drug has been approved to specifically treat bird flu, two compounds (Zanamivir and Oseltamivir) are currently approved by the FDA to treat more common human strains and a third compound (Peramivir) is in Phase-1 clinical trials. These compounds are sialic acid mimics that inhibit viral replication through binding to the viral protein neuraminidase. In an effort to determine if current inhibitors will be effective against pathogenic influenza we are using a variety of Molecular Modeling and Simulation tools to construct and refine 3-dimensional models for wild-type and mutant neuraminidase known to cause resistance to current anti-influenza chemotherapeutics. We are using experimental structures of compounds complexed with neuraminidase, from characterized flu strains, to construct homology models of neuraminidase for H5N1 and other strains with unknown 3D structures including a highly pathogenic strain from 1918 termed “Spanish Flu” which killed 20-40 million persons worldwide. The resultant models provide a computational framework for elucidating the molecular determinants that govern binding of inhibitors with neuraminidase so that more potent compounds can be developed. Results from molecular dynamics (MD) and binding energy calculations will be presented.

This work is being supported with funding from the New York State Office of Science Technology and Academic Research (NYSTAR), the Department of Applied Mathematics and Statistics at the Stony Brook University, the Computational Science Center at the Brookhaven National Lab, NVIDIA Corporation, and the Offices of Learning and Research Communities (URECA), Stony Brook University.

Daniel Cohen

SUNY of New Paltz

Synthesis and Modifications of Enantiomerically Pure Biphenols

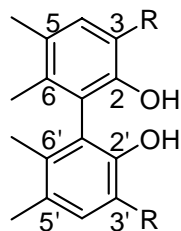
Daniel Tzvi Cohen, *SUNY of New Paltz*; Ce Shi and Iwao Ojima, *Department of Chemistry, Stony Brook University*

Recently, chiral monodentate phosphorus ligands have proven to be highly efficient in various asymmetric transformations. These chiral structures are relatively easy to synthesize as well as modify. It has become an efficient practice to determine the most suitable ligand for a given process rather than trying to develop a universal ligand for different types of asymmetric transformations.

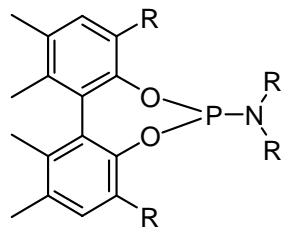
In Professor Ojima's lab, chiral biphenol based monodentate phosphorous ligands have been designed and synthesized. The key feature of the design is that modifications can be introduced at the amine moiety as well as the 3, 3' positions of biphenol moiety. The modifications of biphenol moiety have proven to have great effect on the reactivity and enantioselectivity of various asymmetric transformations.

We will present our results on the synthesis and modifications of these axially chiral biphenols. Large quantity of the chiral biphenol bearing *tert*-butyl on the 3,3' positions have been made, from which H, Me, Br, Phenyl have been incorporated to 3,3' positions through a series of modifications. New modifications on 3,3', positions have been designed and attempts of those new modifications will be summarized.

This work was funded by the National Science Foundation Research Experience for Undergraduates (NSF-REU) 2006 Summer Research Grant, New York State Office of Science, the National Institutes of Health (NIGMS), and the National science foundation.



R = H, Me, Br, ph, t-butyl



R' = Chiral & achiral alkyl

General structure of biphenol and phosphoramidite

Kevin Eccles
Spring Arbor University

Efforts Toward the Synthesis of a Potential New Ligand for Oxygen Activation, and a Bis Glyoxalate Amide Designed to Bind an $i, i + 4$ Cysteine Pair in an Alpha Helical Protein

Kevin Eccles, *Spring Arbor University*; and Dale Drueckhammer, *Department of Chemistry, Stony Brook University*

Synthetic efforts were directed at the biuret functionality of a potential new macrocyclic ligand for an iron-based oxidation catalyst. A synthetic route was pursued that began with reaction of a primary amine with two equivalents of a phosgene-type reagent. In all cases the amine reacted with one equivalent of the reagent to form a urea functionality, but failed to react with the second equivalent to form the biuret compound.

Efforts were also directed toward the synthesis of two different bis glyoxalate amides that were designed using the computer program CAVEAT to position the pair of aldehyde groups in the correct relative position for forming hemithioacetal linkages with the thiol groups of an $i, i + 4$ cysteine pair in an alpha helical protein. Two diamines, piperazine and 1,2-phenylene diamine, were converted to the bis acrylamide and bis bromoacetamide compounds. Reactions were performed to convert the alkene group of the bis acrylamide to the aldehyde using osmium tetroxide and sodium periodate while reactions to convert the bromide to the aldehyde were performed using DMSO and sodium carbonate. All four reactions form highly polar products that are prone to hydrate formation in the presence of water. Purification and characterization of the products are in progress.

Joseph Gaiteri
King's College

Development of Novel Membranes for High Flux Desalination of Seawater

Joseph C. Gaiteri, *King's College*; Hongyang Ma, Benjamin Hsiao, *Department of Chemistry, Stony Brook University*

The existing desalination methods consume a great deal of energy. Our aim is to develop a low-energy method to remove sodium/chloride ions from sea water through the utilization of high flux membranes equipped with a layer of hydrophilic water channels that will facilitate desalination through reverse osmosis. These water channels will be created with carbon nanofibers that have been properly coated with derivatives of the ionic liquid (1-methylimidazolium). As this is an entirely new project, a large part of our research thus far has focused on the creation of the various components of the desalination membranes. Various ionic liquids and powders, such as 3-PEG₇-1-methylimidazolium chloride, 3-butyl-1-methylimidazolium chloride, and 3-dodecyl-1-methylimidazolium chloride, have been successfully synthesized and purified. Moreover, a practical method for anchoring several of these ionic compounds onto the surface of carbon nanofibers has been developed. Recent work has been concerned with coating the surface of polyacrylonitrile (PAN) electrospun membranes with thin layers of cellulose acetate and suspensions containing ionic compound-coated carbon nanofibers. Scanning electron microscopy is our method of choice for determining the efficacy of the various coating techniques, which have thus far been based upon the solvents acetone and 3-butyl-1-methylimidazolium chloride. Conclusions on said techniques will be drawn after a large group of recently prepared samples are viewed under the scanning electron microscope this week.

Megan Johnson
Whittier College

Physical Characterization of Human Islet Amyloid Polypeptide Fibrils using Atomic Force Microscopy

Megan Johnson, Whittier College; Andisheh Abedini, Peter Marek, Chemistry Department, Stony Brook University; Stanislaus Wong, Chemistry Department, Stony Brook University and Condensed Matter Physics at Brookhaven National Laboratory

Amyloidoses diseases affect many in the world today. Type-2 diabetes has become a global health concern of the 21st century. Almost all cases of type-2 diabetes involve a deposition of amyloid fibrils in the beta-islets in the pancreas. These fibril deposits are mostly composed of the human islet amyloid polypeptide (IAPP), a 37 residue polypeptide chain with a disulfide bridge. Islet amyloid and other amyloidoses are created by misfolding of the polypeptide to create a seed. The seed then allows for a more rapid assembly of β -sheets which wrap together to form fibrils. The current studies sought to characterize the fibrils formed by wildtype IAPP and mutants to quantify physical features such as height and length of the individual fibrils that other means of analysis do not offer. Experiments were conducted using Tapping Mode-Atomic Force Microscopy (TM-AFM). We specifically focused on the wildtype of IAPP at pH 4.0, pH 7.4, and pH 8.8 as well as a leucine substituted for an aromatic peptide in a mutant of IAPP (3xL). The pH dependent studies were conducted since previous work shows morphological differences in fibrils as a function of pH. The 3xL mutant was designed to test if aromatics are required for Amyloid formation. Upon analysis, it was found that the wildtype IAPP fibrils at pH 4.0 had an average height of 4.72 ± 0.11 nm and an average length of 688 ± 42 nm. Similarly the 3xL mutant had an average height of 4.40 ± 0.12 nm and an average length of 522 ± 30 nm. The noticeably smaller length could be attributed to the substitution for the aromatic residues, perhaps allowing for closer packing. However, the wildtype IAPP at pH 8.8 had a larger height than both the mutant and the wildtype at a lower pH. Its average height was 5.22 ± 0.25 nm. This was consistent with optical observations of the fibrils. Each type of fibril examined displayed a different optical morphology. The wildtype at pH 4.0 followed previously reported structure of protofibrils forming a super-helical fibril. The wildtype at pH 8.8 showed no super-helical fibrils, but rather a corduroy-type fibril and the mutant showed only straight single fibrils. Currently we have been unable to image fibrils formed by wildtype IAPP at physiological pH using TM-AFM. Future work will strive to fill in the physiological wildtype hole as well as investigate other mutants to gain a larger knowledge of islet amyloid and its mutants to apply to medical research.

This work was funded by the National Science Foundation's Research Experience for Undergraduates program at State University of New York at Stony Brook.

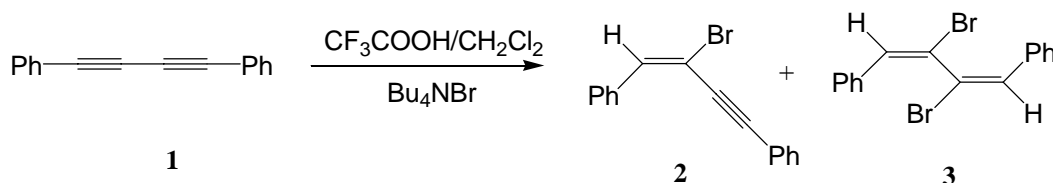
En-Chi Lin
Stony Brook University

Exploration of Conjugated Halogen Compounds

En-Chi Lin, and Nancy S. Goroff, *Department of Chemistry, Stony Brook University*

The main focus of this URECA fellowship was to find an inexpensive synthetic route to 2-bromo-1,4-diphenylbutenyne (**2**). We have targeted enyne **2** as a potential precursor to a polymer called polyacene. Polyacenes are polycyclic aromatic hydrocarbons consisting of fused benzene rings in a rectilinear arrangement. Even though there is a known synthesis for **2**, a simpler and more efficient synthetic route to the monomer is necessary. Hydrobromination of diphenylbutadiyne **1** in $\text{CF}_3\text{COOH}/\text{CH}_2\text{Cl}_2$ solution yields compound **2** (Scheme 1), but **2** can also react under these conditions, leading to a mixture of **2** and diene **3**. Another approach involves preparing **3** and then eliminating one equivalent of HBr using a strong base to make enyne **2**. We have successfully synthesized and purified **3** with an isolated yield of 29%. Further work continues on finding the appropriate base to prepare **2** cleanly.

A second project involved determining the effect of solvent on the ^{13}C NMR of the allene tetraiodopropadiene. Previous studies have shown that iodoalkenes can act as Lewis acids, and that Lewis basic solvents can affect their ^{13}C NMR chemical shifts. Calculations and measurements on tetraiodoethene and tetraiodobutatriene have already been done. This project focuses on confirming the calculations already done on tetraiodopropadiene. Tetraiodopropadiene was prepared by adding iodine and potassium hydroxide to 3-bromo-1-propyne in methanol. Its ^{13}C NMR spectrum has been measured in pyridine and chloroform to determine the solvent effect.



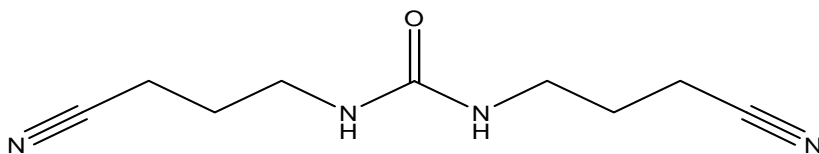
Scheme 1. Synthesis of monomer **2**.

Daniel Miranda
University of Maryland

Creation of Cocrystals with Hosts to Align Diiodobutadiyne for 1,4 Polymerization

*Daniel Miranda, University of Maryland; Liang Luo and Nancy S. Goroff,
Department of Chemistry, Stony Brook University*

The 1,4-polymer of diiodobutadiyne has potential applications as a conducting material and is a precursor of carbyne. For desired polymerization to occur, the monomer needs to be lined up in a crystal by a host molecule with the proper geometry. Ureas and oxalamides through hydrogen bonding form networks with repeat distances near the number required for polymerization. The iodines in diiodobutadiyne can act as Lewis acid acceptors in intermolecular halogen bonding; a process similar to hydrogen bonding. Appropriate Lewis bases include nitrile and pyridyl groups. Hosts containing a hydrogen bonding group, and a Lewis base could be used as hosts to align diiodobutadiyne. The first part of this project dealt with synthesizing two urea hosts and an oxalamide host that have been made before, in order to explore more cocrystal possibilities. Diiodobutadiyne also had to be synthesized to create cocrystals. Finally, a new approach to creating cocrystals was attempted. Previously, cocrystals containing only one host compound were used. However, previous work shows that a combination of a urea and oxalamide will form its own unique hydrogen bonding network, with a geometry intermediate to a pure urea or oxalamide. Therefore, using combinations of various hosts and different ratios, new cocrystals were created mixing urea and oxalamide hosts. The 3 solids were placed in vials, dissolved in methanol, and allowed to crystallize in a dark freezer. This research was funded by a grant from the National Science Foundation through the Research Experience for Undergraduates Program (REU) at Stony Brook University.



N, N'-biscyanopropanyl urea host containing hydrogen bonding urea and Lewis basic nitrile



Diiodobutadiyne with Lewis acidic iodines

Kristen Zeitler
Fairfield University

Synthesis of Dinuclear Rhenium Phenanthroline Complexes for Solar Fuel Production

Kristen Zeitler, Fairfield University; and Andreas Mayr, Department of Chemistry, Stony Brook University

The development of efficient methods for solar fuel production is a major scientific challenge. The light-driven production of fuels from carbon dioxide and other small molecules will benefit society not only by providing a cheap source of fuels, but also by contributing towards the reduction of greenhouse gases. The achievement of this goal will require the availability of effective catalysts for the capture of light and the activation of carbon dioxide and other small molecules.

The aim of the present study is the synthesis of dinuclear rhenium phenanthroline complexes that may be used in the photochemical activation of CO₂ and other small molecules such as CO and N₂. Towards this goal, two rhenium complexes of 4,7-dibromo-3,8-dihexyl-1,10-phenanthroline are connected by means of flexible linkers at the 4,7-positions. This way, the two rhenium phenanthroline units can approach each other face to face, and the two metal centers may be capable of capturing and activating the molecules of interest. Column chromatography, nuclear magnetic resonance spectroscopy and thin layer chromatography were techniques used for isolation, purification, and characterization of reaction products. Further characterization of the photochemical properties of the final products will be performed in future studies. This research is funded by a grant from the National Science Foundation (Research Experiences for Undergraduates) at SUNY Stony Brook

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