

Research Experience For Teachers II
Cluster Project
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The research of the cluster group involved the synthesis of tungsten based clusters. After synthesis of the tungsten cluster, the linking of these clusters together to form a crystalline network is being studied. It is the hope of the group that this linking will be achieved to a degree sufficient to provide interesting electrical properties. The delocalized electrons from the tungsten metal along with the pi electrons from the connector ligands make for potentially exciting chemistry for the future.

Although a high school chemistry teacher, most of what I experienced this summer was not familiar. From the scope of the facilities, to the chemistry being done, I was in a completely different element.

My research director, Frank DiSalvo, told me that I would begin my summer doing what all his new graduate students did, preparing their own sample of tert-butylpyridine tungsten chloride. The equipment, instruments, and facilities being so intimidating, I was glad to learn I would be working closely with two graduate students, Ellen Scheuer and Catherine Oertel.

After completing the lab safety training I proceeded doing research. The first week, I did some solid-state chemistry. This reaction between tungsten hexachloride and bismuth metal required the use of a specially designed glass tube made by the glassblower. One of the reactants was kept in the argon glove box, and was added to the tube inside the box. This was my first exposure to working in an inert atmosphere. At first I found the glove box and the attached antechamber under vacuum to be very intimidating. I would use it on an almost daily basis and soon became comfortable not only with its use but also bringing items in and out. I even witnessed the changing of the gloves, quite an involved process. This glass tube was sealed under vacuum. This first reaction also involved using a furnace, which was programmed to take a specific time to warm up to the desired temperature, remain at that temperature for so many hours, and then how long the cool down should take.

The sealed glass tube was scored with a saw and cut open. The tungsten dichloride was the product of interest. It was reacted with hot, concentrated hydrochloric acid. The acid separated the tungsten chloride (W_6Cl_{12}) ringed cluster into another cluster, $(H_3O)_2W_6Cl_8^+Cl_6^-$, one step closer to the desired product. This intermediate, was reacted with tetrabutylammonium chloride hydrate, after both reactants had been dissolved in ethyl alcohol. A thick, soft yellow, precipitate was formed, dibutylammonium tungsten chloride. These crystals were recrystallized by dissolving them in hot acetone. They are soluble in hot acetone but not room temperature acetone. As it cooled, the yellow crystals formed. The beaker was allowed to cool and placed in the refrigerator overnight. This batch of product appeared to come out particularly well. A sample was prepared and an X-ray crystallography scan confirmed this.

The procedures above were repeated and a second batch of the butylammonium tungsten chloride cluster was prepared. Most of the reactions took twenty-four to ninety-six hours, so as one step of the first batch was being processed, another step of the second batch was underway. It turned out to be fortuitous for me that a second crop was available.

When the beautiful, yellow, butylammonium tungsten chloride cluster was reacted in the next step of the process with potassium sulfur hydride, potassium tert-butoxide, and 4-tert-butylpyridine for the final step in the preparation of butylammonium tungsten chloride cluster the realities of research hit. Something went wrong.

The work up of the desired product was not going according to plan. The filtration wasn't working. A very fine powder appeared to be present that was interfering with the separation of the cluster. After aborted vacuum filtration with a frit, many trips to the centrifuge also proved fruitless. Very little product was recovered, and an NMR confirmed that it was, as we feared, not very pure. After hypothesizing about the unexpected results, it was decided that the second batch of yellow butylammonium tungsten chloride crystals would be divided and two separate reactions would be performed in case the same problems should occur.

Fortunately, the work up for the second batch of tert-butyl pyridine cluster went well, and the reddish cluster collected in the frit almost effortlessly. An NMR sample showed the expected peaks, with no surprises.

Having regained faith in our procedure, the other half of the second batch of butylammonium tungsten chloride crystals were processed to make the desired TBP cluster.

The last step of this research that I was able to participate in was the attempt to link these tert-butyl pyridine tungsten chloride cluster together with bipyridine. The two lone pairs of electrons, one of each nitrogen atom in the bipyridine allows for the linking of clusters.

In addition to the time spent with the DiSalvo cluster group I also participated in some of the RET activities that were planned. A Powerpoint and an Excel workshop were held, graduate students doing outreach did some demonstrations and taught lessons on various topics. "Fun Talks" were held weekly, and the RET I teachers gave weekly presentations each Friday.

This RET II was an experience that I will treasure. I am indebted to the members of the DiSalvo group for sharing their enthusiasm, talents, knowledge, expertise, and time with me for six weeks. I learned a great deal of chemistry that I hopefully will share with my students. I also did some chemistry. This lab experience was the best part. I have a much better understanding of how a chemistry lab operates and how chemistry is done at the university level.