Chemistry
How to Synthesize Aspirin, Light-Harvesting Dyes, Secret Inks, and Much More.

DEPARTMENT OF CHEMISTRY
July 8 - 14, 2018
July 15 - 21, 2018
Pre-College Summer @ UConn, Chemistry

Chemistry: How to Synthesize Aspirin, Light-Harvesting Dyes, Secret Inks, and Much More!

PROGRAM OVERVIEW

Be Part of the solution!

Pre-College Summer Chemistry provides a strong emphasis on hands-on age-appropriate laboratory experiments and interaction with experienced faculty. Participants will get a feel for the magic of making new compounds and see bench-top wet chemistry and advanced chemical instrumentation first-hand. Fun and interactive discussion will provide the required background knowledge and will further summarize and assess in-lab experiences. We will explore the areas of chemistry that touch on daily life: synthetic food additives, drugs (aspirin), colors in paints, solar energy, and batteries. Students will have the opportunity to view and interact with a number of impressive chemistry magic demonstrations... and food chemistry, the labors of which will be enjoyed. It's going to be a great week filled with exploration, new experiences, and fun!

All safety equipment necessary for being in our state-of-the-art teaching laboratories (gloves, goggles and lab coats) will be provided. Due to chemistry laboratory safety regulations, students will be required to wear long pants and closed toed shoes during the entire chemistry course (the chemistry building is air conditioned).
THE INSTRUCTORS
FOR THE ECE PRE-COLLEGE SUMMER CHEMISTRY EXPERIENCE:

Clyde Cady, Ph.D., is a Visiting Assistant Professor in the Department of Chemistry. Dr. Cady received his undergraduate degree from the University of Minnesota where he was introduced to bioinorganic chemistry, the chemistry of the elements as pertaining to life events. He moved to Connecticut for his Ph.D. at Yale University where he studied photosynthesis. After postdoctoral studies at Uppsala University, Sweden, and Rutgers University he started teaching general and inorganic chemistry at UConn. His special interests are directed toward introducing pre-college students to the thrills of experimental chemistry.

Christian Brückner, Ph.D., is a Professor in the Department of Chemistry. After his undergraduate studies in chemistry and biology at the Rheinisch-Westfälische Technische Hochschule (RWTH) in Aachen, Germany, Dr. Brückner received his Ph.D. degree in synthetic heterocycle chemistry from the University of British Columbia, Canada. After a postdoctoral stay at the University of California, Berkeley, he joint UConn. His research interests span the chemistry of porphyrins and related dye macrocycles and their utilization in a number of medicinal and technical applications. For the past nine years, he was the Associate Department Head for Teaching. He also led the NSF-funded departmental undergraduate summer research (REU) program.
LABORATORY

T209 (2nd floor teaching wing)
Located in the new chemistry building, UConn Storrs campus

Proper laboratory attire (long pants and closed-toed shoes) is mandatory. Lab coats and safety goggles will be provided. (for more safety information, see below)

CONTACTS: INSTRUCTORS

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CONTACTS: TEACHING ASSISTANTS

Anne Mirich

Seth March
PROGRAM SCHEDULE OVERVIEW

Sample Schedule:

7:30 AM – 8:30 AM  Breakfast
9:00 AM – 12:00 PM  Class
12:00 PM – 1:50 PM  Lunch
2:00 PM – 4:00 PM  Workshops on Mondays, Wednesdays and Friday; Class on Tuesdays and Thursday
4:30 PM – 6:00 PM  Free/Study time and optional activities
6:00 PM – 7:00 PM  Dinner
7:00 PM – 10:00 PM  Free/study time and optional activities; Students must be on their floors by 10 PM
10:30 PM  Students must be in their rooms
Laboratory: Chemistry Department
T209 (2nd floor teaching wing)

Monday
8:45 – 11:45 am  Welcome
Safety instruction
Synthesis of Aspirin

Tuesday
8:45 – 11:45 am  Extraction of Ultramarine from Lapis Lazuli
2:15 – 4:00 pm  The Golden Penny

Wednesday
8:45 – 11:45 am  Synthesis of a Superconductor
Phosphorescent Emissions of a Metal Complex

Thursday
8:45 – 11:45 am  Making of a Dye-Sensitized Solar Cell (Grätzel Cell)
2:15 – 4:00 pm  Chemistry Lecture (What freshman chemistry is like)

Friday
8:45 – 11:45 am  Food Chemistry
Pre-College Summer Laboratory Safety Policies and Procedures

Before You Start Work in the Laboratory:

- Make sure you are familiar with the location of the following safety equipment: the eye wash fountains, emergency shower, first aid kit and emergency exits.
- Read the experiment you will be performing before coming to lab. This will allow you to familiarize yourself with the chemicals and techniques being utilized in the experiment.
- Material safety data sheets (MSDS) for all chemicals are available in the stockroom or online at the UConn EH&S web site (www.ehs.uconn.edu).
- Food, drink and chewing gum are never allowed in the laboratory.
- I-Pods, CD players, MP3 players, cell phone, pagers and other electronic devices may not be used at any time in the laboratory.

Laboratory Attire:

<table>
<thead>
<tr>
<th>EYES:</th>
<th>Approved eye protection will be provided on the first day of class.</th>
</tr>
</thead>
<tbody>
<tr>
<td>BODY:</td>
<td><strong>Long pants must be worn at all times in the lab. Please make sure you bring a pair of sweat pants you can wear over your shorts if you do not want to wear pants after lab is over.</strong></td>
</tr>
<tr>
<td>FEET:</td>
<td><strong>Shoes that completely cover the feet are required. Socks are not an acceptable substitute for proper footwear or full length pants. Ballet shoes, sandals, flip-flops, clogs, and flats that leave the foot exposed are not allowed.</strong></td>
</tr>
</tbody>
</table>

*If you do not come in proper attire you will be dismissed from the laboratory!*

Also note:

- Lab coats will be provided on the first day and should be worn when in the laboratory.
- Gloves will be provided in the lab. Wear gloves whenever directed to do so but remember to remove your gloves before going into the hallway.
- Loose fitting clothes represent a fire/spill hazard and should be avoided.
- Long hair must be tied back while in the laboratory to prevent exposure to flame or chemicals.
Synthesis of Aspirin

Purpose

Chemical synthesis is the purposeful execution of chemical reactions to obtain one or more products. Chemical reactions are processes that lead to the transformation of one set of chemical substances to another. This happens by chemical manipulations usually involving one or more reactions. Chemical synthesis is one of the core disciplines in chemistry (others are chemical analysis, for example) and form the basis for the pharmaceutical and petrochemical industries. In this lab we will synthesize a drug – Aspirin – from a naturally occurring starting material.

Introduction

Salicylic acid and its esters are found in several plants. They are naturally occurring pain killers (analgesics) for humans. The salicylic acid found in willow bark and leaves (Salix species) has been used medicinally by native people all over the world but it wasn’t until the 1800s that the active components were isolated.

Salicylic acid is very hard on the stomach. Thus, salicylic is converted to its acetyl ester (acetylsalicylic acid), also known under its trade name Aspirin, firstly synthesized in 1897 along the synthetic route we will explore.

The Experiment

The synthesis of aspirin from salicylic acid is a so-called esterification reaction. Esters are formed from the reaction of an alcohol and a carboxylic acid. Salicylic acid is a bifunctional compound. It is a phenol (an aromatic alcohol) and a carboxylic acid (R-COOH) or carboxylic acid derivative. In the presence of acetic anhydride, acetylsalicylic acid is formed by the esterification of the alcohol functionality. Chemical formula can represent the reaction as follows:
Besides aspirin, acetic acid is a product of this reaction. This product is readily soluble in water and will be removed when the product is filtered. Dilute acetic acid is vinegar.

Have you ever noticed that a bottle of aspirin tablets can have a distinct vinegar smell? As the aspirin tablets age the acetylsalicylic acid reacts with moisture and decomposes to acetic acid and salicylic acid. If your bottle of aspirin is old and has a strong vinegar smell you are more likely to experience the unwelcome side effects seen with salicylic acid. Check the expiration date; you may want to throw it away. Many manufacturers now coat the aspirin tablets to prevent decomposition.

### Materials

**Equipment**

- 125-mL Erlenmeyer flask
- 400-mL beaker
- Heat source
- Thermometer
- Dispensing pipet
- Crystallizing dish
- Glass rod
- 10-mL graduated cylinder
- 50-mL graduated cylinder
- Buchner funnel
- Filter flask
- Small test tubes (3)

**Chemicals**

- Salicylic acid
- Acetic anhydride
- Concentrated sulfuric acid (98%)
- 0.25 M iron(II) nitrate in 0.5 M hydrochloric acid
- Ethanol

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**CAUTION**

*Perform all experiments under the fume hood.*

Concentrated sulfuric acid is highly corrosive. You must handle it with greatest care. Inform the instructor of any spill, drop on your skin, or clothes.

Acetic anhydride is an irritant and combustible liquid. Handle under fume hood.

Make sure you wear your goggles throughout the experiment.*
Procedure

A. Synthesis of Aspirin

Using a 400-mL beaker, set up a water bath and begin heating it with a Bunsen burner. You want the water to reach a temperature of about 75°C.

While the water is heating, weigh 2.0 g (0.015 mole) of salicylic acid crystals and place them in a 125 mL Erlenmeyer flask. Add 5 mL (0.05 mole) of acetic anhydride, followed by 1 to 2 drops of concentrated sulfuric acid and gently swirl the flask until the salicylic acid dissolves.

Heat the flask gently in the water bath for 10 to 15 minutes and stir occasionally.

Allow the flask to cool to room temperature, during which time the acetylsalicylic acid should begin to crystallize from the reaction mixture. Cool the mixture slightly in an ice bath until complete crystallization has occurred. If it does not, carefully scratch the inside walls of the flask with a glass rod; this often induces the formation of crystals.

After crystallization is complete - usually, the product will appear as a solid mass when crystallization has become complete, add 50 mL of water and cool the mixture in an ice bath.

To collect the product by vacuum filtration, set up a filtration apparatus using a Buchner funnel (your instructors will demonstrate how to use a Buchner funnel). Do not forget to use a filter paper in the funnel and record its weight beforehand! Little ice-cold water can be used to rinse the Erlenmeyer flask out to collect all the crystals.

Mass weighing paper: ______________________________

Rinse the crystals (the filter cake in the filter) several times with small portions of ice-cold water. Continue drawing air through the crystals for air drying.

Meanwhile, weigh a watch glass and record its mass. After a few minutes, remove the vacuum and place the filter paper and crude product on the watch glass. Place under a heat lamp to continue drying. Weigh the crude, dried product.
Mass crude product + paper: ____________________________

Mass aspirin: ____________________________

**B: Testing Purity of the Product**

The reaction may not have gone to 100% completion (or some product may have reacted backwards). Either way, some salicylic acid may still be present in your crude product. This can be analyzed using a convenient color reaction: salicylic acid produces a red color when reacted with iron(III) ions, acetyl salicylic acid does not (can you now guess which portion of the molecule in salicylic acid reacts with the iron to produce the color?)

Perform the following procedure for testing the purity of your product:

- Weigh out 0.10 +/- 0.01 g of your product into a 100 mL beaker
- Add 5 mL ethanol to dissolve product
- Add 5 mL of a solution of 0.25 M Iron(III) nitrate in 0.5 M HCl
- Add 40 mL of deionized water
- Compare the colors of your solutions. The more red, the more unreacted salicylic acid does the sample contain.

This method can also be used to quantify the amount of impurity present using a spectrometer, an instrument that quantifies the color density.

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**WASTE DISPOSAL**

Water from hot water baths ONLY goes down the drain (let them cool first). All other chemical/reagent waste to the dedicated waste containers. Filter paper goes into the regular trash, NOT the waste containers.
Extraction of Ultramarine from Lapis Lazuli

Purpose

Extraction and purification of valuable chemicals found in nature are very important processes in chemistry. In this lab we will look at an unusual and historic example of both extraction/purification and synthesis as it relates to solid state chemistry. Sometimes we get a bit to caught up in how awesome modern science is. Just because some hundred years ago people were still contracting polio, dying of small infections, and traveling by horse doesn’t mean that the science they did was not impressive. In fact, considering the primitive tools available and the limited knowledge about the underlying processes makes the accomplishments of our forefathers all the more impressive.

In this lab we will experience the ancient ultramarine extraction method from impure lapis.

Introduction

In the late middle ages, the cutting edge of visual entertainment often consisted of paintings (what would we ever do without Instagram?). Before the 1600's, all of the colors used in paintings came from natural pigments. The hardest (and one of the most beautiful) of all colors to obtain was a deep blue. This could only be obtained by crushing Lapis Lazuli stone. Because pure blue lapis lazuli is a semi-precious gem stone that was mainly found in modern day Afghanistan, it is
understandable that it was rare. The blue color obtained from Lapis is known as ultramarine. The rarity of ultramarine lead to a very high price for the pigment: its price at some time around 1300 was listed as 8 ducats per ounce for very fine ultramarine. A ducat is about 3.5 g of gold. This means that 1 ounce of ultramarine was worth slightly more than one ounce of gold!

Originally, a pure blue lapis lazuli stone was crushed to make the ultramarine pigment, but its high price drove people to look for ways of extracting it from impure Lapis ores that were contaminated with white (calcium carbonate), sodalite (dark blue), and gold (iron pyrite) particles. Grinding the impure ore did not provide the desired pure, deep blue, but a greyish powder. Thus, in an example of market-driven research, someone developed a method to separate the calcium carbonate and iron pyrite from the blue pigment ultramarine.

Even with the advanced purification methods developed in the middle ages and renaissance periods ultramarine was still expensive. In the middle ages it was reserved for painting the robes of the Virgin Mary or the swaddling clothes of the Baby Jesus. Even by the 1600’s it was still pretty rare, Vermeer’s Girl with Pearl Earring (show on on the right) is a good example of how it was used as an accent. As you can imagine there were a lot of other things that people wanted to paint blue but they couldn’t afford the pigment.

In 1824 a French society offered a large prize (think X-prize) for the development of an artificial Ultramarine. A few years after the prize was offered, a process was found leading to what is called “French Ultramarine” a synthetic version of the pigment derived from lapis lazuli.

(images from Wikipedia: https://en.wikipedia.org/wiki/Lapis_lazuli)
The Experiment

In this lab we will isolate ultramarine from lapis by a procedure that is analog to that used in the middle ages. Where is the chemistry in this? The blue color in ultramarine comes from a cage of aluminum and silicon that protects a group of sulfur atoms, as shown in the figure below. The trapped sulfur atoms create the blue color but are also very unstable, they are only present because of the protecting cage. The rest of our lapis lazuli rock is made up of CaCO$_3$ (limestone) and Fe$_2$S$_3$ (fools gold). The first thing that a chemist thinks to do when trying to get rid of a carbonate like limestone is to dissolve it in acid but in this case we can’t because the H$^+$ in the acid is small enough to get inside the ultramarine cage and everything turns white. The middle ages chemist first trying to separate the blue color probably was rather upset when they turned everything white. Because we can’t use the simple dissolve in acid method we have to use a more advanced chemical procedure called an extraction. In this extraction we are trying to separate the negatively charged sulfur cage from the non-charged limestone and fools gold. Normally when we do this all of our chemicals are liquids and we can separate things by using two different solvents, like oil and water. In this case we have the added complication of having a solid but the principle is the same. We have a big ball of wax (which is non-charged like an oil) and this ball of wax is in a water and NaOH solution (which is charged). If we had liquids we would just mix the two together but because we have solids we have to smush up the ball of wax manually. As we smush the wax we expose the wax with all of our stone inside to the water solution. At the same time the outer layer of the wax is dissolved off as it turns into soap. As the outer layer of wax dissolves the non-charged limestone and fools gold bury themselves deeper in the wax because they are non-charge and like to stay in the non-charged wax rather than go into the water. At the same time the charged Ultramarine cage is given the same choice of wax or water and because it has a charge it chooses water. So after a lot of work we end up with a bunch of wax that has mostly limestone and fools gold and water that the blue ultramarine powder.
Tuesday 8:45 – 11:45 am

Materials

![Materials and Equipment](image)

**Equipment**

- Hot plate
- Medium evaporating dish
- Wooden dowel
- Mortar and pestle

**Chemicals**

- 1.85 g Gum Rosin
- 950 mg Gum Acacia
- 1.10 g Paraffin wax
- 5.0 g Lapis powder (125 microns or smaller)
- 75-100 drops mineral oil
- 75 mL 10 mM aqueous NaOH deionized (DI) water

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**CAUTION**

Be careful when heating up the dishes; they may be too hot to touch with your bare hands!

NaOH is caustic and particularly dangerous when splashed into your eyes. Make sure you wear your goggles throughout the experiment.
Procedure

Place gum rosin, gum acacia and wax in a small evaporating dish and heat on a hot plate until melted (Picture 1). Be careful not to burn the wax.

Grind the lapis lazuli powder to make an even finer powder (picture 2).

Mix thoroughly and add the ground and sieved lapis powder (Picture 3), mixing until uniform blue.

Add 15 drops mineral oil and remove from heat and cool (Picture 4).

Once the wax/three sap mixture is
cool enough to touch with gloves you can remove from the evaporating dish and work it into a ball with your gloved hands. The ball should be soft enough to mold easily. If too hard, work in an extra 5 drops of mineral oil.

You are going to want to deform the wax ball using your wooden dowels (Picture 5), make sure it is soft enough but still forms a ball.

Place ball back in evaporating dish and add NaOH solution (also called lye). Press and deform the ball with the wooden dowels in the solution (Picture 6). The lye solution should slowly (20-30 min) extract out the blue color, which will accumulate in the bottom of the evaporating dish.

The blue solution can be decanted off to collect some of the ultramarine. You should see it collecting in the bottom of the dish. Decant off the cloudy solution to remove most of the wax and add a small amount of DI water (Picture 7).

Swirl the solution to observe the dark blue solid you have extracted and note how hard it was to get a few mg (Picture 8).
WASTE DISPOSAL

Place all wastes in the provided waste containers.
The Golden Penny

Purpose

Electroplating one metal on another is a common and industrially important electrochemical process that deposits a metal from a metal salt solution onto a solid surface, also often a metal. Alloys are solid mixtures of two (or more) metals (or other elements) that have a different property than either pure components. In this lab we will perform an electroplating process – we will deposit zinc onto a copper penny, followed by an alloying process where we alloy the layer of zinc with the underlaying copper to form a brass-plated penny.

Introduction

The applications of electroplating is very broad, ranging from chrome plating a car bumper (in the old days when chrome was still on the bumber) to plating a layer of gold on a silver ring, to making a replica of a priceless piece of art (electrotyping). Electroplating processes are often driven by electricity; it is an electrochemical process. Normally, we deposit a thin layer of metal on a surface using electrical current but there are other methods. In this lab we will use heat and the electrochemical potential between the metals involved to drive the electroplating redox process instead of electricity.

Layering two different types of metal on top of one another is very useful but metals in their pure form are not always possessing all desirable properties. Thus much of our daily life is dependent upon homogenous mixtures of metals called alloys (technically they are solutions of one metal in the other). These alloys have distinctly different appearances and properties when compared to the pure metals. Pure iron is soft while steel, a mixture of carbon and iron, can be very hard – though it may readily form rust. Adding in a little bit of chromium may make stainless steel. Bronze, one of the first alloys made, is made of copper and tin. Brass is a mixture of zinc and copper, which is often used in decorations. We will here alloy zinc and copper to form brass.
The Experiment

This experiment will be plating zinc onto a copper penny. However, since zinc mixes with the copper forming brass, we are taking this experiment one step beyond electroplating, we will alloy the zinc and copper by heating them up, thus plate the penny with brass. Copper has the typical red color, zinc is silver-grey, and brass is yellow (or golden), clearly showing what type of metal or allow is on the surface.

Equipment

250 mL beaker
Hot plate
bunsen burner
tongs

Chemicals

1 clean, polished penny
5 g of zinc powder
24 g of sodium hydroxide pellets
100 mL of DI water

CAUTION

Sodium hydroxide pellets are strongly caustic. Use gloves when handling them or handling your penny wetted by the hot lye solution. Your eyes are particularly susceptible to damage by sodium hydroxide (lye).

Make sure you wear your goggles throughout the experiment.

Be sure to do the heating inside of the fume hood.

Be careful when using the Bunsen burner. Only use the burner in the hood, make sure there is no combustible material in the hood at the same time and never leave the flame on unsupervised.

Procedure

1. Electroplating the Copper Penny with Zinc.

In a 250-mL beaker dissolve the sodium hydroxide in the water. Add the 5 g of zinc and heat on the hot plate until boiling. As the zinc dissolves, hydrogen will
be given off and you will observe the solution fizzing. Drop in your penny, making sure the penny is in contact with some of the solid zinc in the bottom of the beaker. After 2 or 3 minutes remove the penny using tongs. Be careful the solution is hot and very corrosive. Drop the penny into a beaker with water and then rinse the penny under running water and dry with a paper towel. Remove the sodium hydroxide solution from the heat so that it can cool down.

You now have a silver penny created by plating a layer of zinc on top of the copper.

2. Alloying the Zinc on the Copper Penny to form Brass.

Take the silver penny with a pair of long metal tongs and carefully heat it in a Bunsen burner flame. Heat the silver penny gently, zinc melts at a low temperature (mp = 420 °C; compare to the mp of copper of 1085 °C) and boils off before the copper melts (bp 907 °C). As the zinc melts and alloys with the underlying copper, the penny should turn the golden color of brass. Place the golden penny in a beaker of clean water in order to cool it and then dry with a paper towel.

WASTE DISPOSAL

Place the COLD caustic wastes very carefully in the provided waste containers (avoid splashing) and rinse the beakers with copious amounts of water.
Synthesis of a Superconductor

Purpose

This experiment illustrates the synthesis of a defect perovskite structure which shows superconductive properties at low temperatures, demonstrated by a levitation in a magnetic field experiment.

Introduction

The phenomenon of superconductivity refers to the observation that certain materials possess zero electrical resistance; electricity flows through them without heating up the material. This phenomenon was first observed in mercury in 1911. A large number of other elements and alloys were subsequently shown to exhibit this property, but the early examples required temperatures in the liquid helium range (T_c < 4.2 K). Superconductive magnets have been built and commercialized for devices such as NMR and MRI instruments. The major hindrance to the widespread use of superconductivity is the cost of maintaining the liquid helium or supercritical helium temperatures. Because of the great technical advantages in having electromagnets operate without resistance (they generate large magnetic fields without warming up or wasting energy), many research efforts were placed on finding ‘high-temperature’ superconductive materials.

For many years, the “holy grail” was to develop a material with a superconductive transition temperature transition temperature above 77 K, the boiling point of liquid nitrogen. This would make the cryogenic technology much less expensive and open up a number of technically attractive uses for superconductivity.
In the 1980s, a number of metal oxides were found that pushed the superconducting temperature up above 77 K. The most famous of these are the ytterbium, barium copper oxides shown in the figure below.

![Diagram of YBa\textsubscript{2}Cu\textsubscript{3}O\textsubscript{x}](https://en.wikipedia.org/wiki/Yttrium_barium_copper_oxide#/media/File:Ybco002.svg)

This special type of structure called a perovskite is critical to the superconductivity of the compound and the bigger the crystal of this structure the better the superconductor. In order to make really big crystals of this perovskite we heat up our pellets to high temperature and then slowly cool them just like slowly evaporating the water from a sugar solution gives you really big sugar crystals.

**The Experiment**

A high-temperature solid state synthesis of the superconductive material Yttrium barium copper oxide, demonstrated by levitation in a magnetic field experiment.
Equipment

Mortar and Pestle
½ inch pellet die set

Chemicals

0.56 g yttrium (III) oxide
2.61 g Barium nitrate
1.65 g Cu$_2$(OH)$_2$CO$_3$

Reaction equation:

\[ 4 \text{ BaCO}_3 + Y_2(\text{CO}_3)_3 + 6 \text{ CuCO}_3 + (1/2 - x) \text{ O}_2 \rightarrow 2 \text{ YBa}_2\text{Cu}_3\text{O}_{7-x} + 13 \text{ CO}_2 \]

CAUTION

The tube furnace used in this experiment is heated to almost 1000°C. This is above the melting temperature of most glass and considerable above the flash point of most paper. Your instructor or TA will place your sample inside and remove your sample from the tube furnace. Keep all materials away from the tube furnace and do not touch it when it is operating.

Cryogenics: Liquid nitrogen is -196°C and will cause extreme frostbite almost immediately. Use only under the supervision of your instructor or TA. Remember liquid nitrogen is just many degrees below body temperature as fire is above body temperature. Liquid nitrogen should be treated with just as much care as an open flame. It is just as dangerous.

Procedure

Synthesis of the YBa$_2$Cu$_3$O$_x$ Perovskite

The yttrium, barium, and copper are reacted in the ratio of 1 to 2 to 3 moles. This reaction begins with 0.56 g of yttrium (III) oxide, an amount of Ba(NO$_3$)$_2$ and basic CuCO$_3$.

The weighed samples of the reagents are ground well in a clean mortar and placed in an unglazed crucible in a muffle furnace at 950 °C for two hours. Decomposition of both the carbonate and nitrate will lead to bubbling, and as a result the reaction mixture must be reground following the heating.

Place the ground reaction mixture in a pellet press 1/2" in diameter. Use a pressure of about five tons to make the pellet. Place the pellet in a quartz tube
heated in a tube furnace under slowly flowing oxygen. This refiring and slow cooling is essential to develop the superconductive property. The temperature should be held at 950 °C for about an hour then decreased from 950 °C to 250 °C over a period of three to four hours (or longer).

**Testing its Superconductivity by Utilization of the Messner Effect**

The mutual repulsion of a permanent magnet and a superconductor is called the Meissner effect. The magnetic flux of the magnet will not penetrate the superconducting material. The magnet will, therefore, be levitated if the material on which it is resting becomes a good strong superconductor. Place the pellet at the bottom of a clear Dewar flask or an insulated container made from Styrofoam coffee cups. If the latter is used, a small plastic bottle or a combination of a piece of plastic and glass wool can be used to raise the pellet near the top of the container. Care must be taken to make sure the pellet is not tilted. Carefully place a small magnet on top of the pellet. Slowly pour liquid nitrogen into the Dewar flask or Styrofoam cup. The magnet will levitate if the pellet becomes superconductive.

**WASTE DISPOSAL**

Place all solid wastes into the designated chemical waste containers.
Phosphorescent Emissions of the Metal Complex Tris(2,2’-bipyridine)ruthenium(II)

Purpose

Demonstration of how chemical, and electrical energy, can be turned into light through the fluorescent properties of chemicals and materials.

Introduction

Fluorescent materials as well as other light emitting compounds are becoming increasingly industrially important. LEDs and phosphor coated fluorescent lights are now our main light sources in the home and new medical technologies use fluorescent molecules to track cancers in our bodies. One of the most studied fluorescent molecules known is Ruthenium Tris bipyridine. In this experiment we will explore how to turn chemical potential energy into light using this extensively studied molecule.

The Experiment

In our reaction mixture we have K$_2$S$_2$O$_8$ (an electron acceptor) and magnesium metal (an electron donor). These two compounds together represent an electrochemical potential, like a battery or an electrical current. The problem is that it is hard for electrons to pass between them, kind of like when a battery is in the package and not hooked up to anything. There is a lot of potential energy but nothing is happening. When we add the ruthenium compound to this mixture it
acts as a mediator to move the electrons from the magnesium metal to the persulfate. The ruthenium is like hooking up the battery to a light bulb.

Materials

Equipment

125 mL erlenmyer flask

Chemicals

30 mL DI water
25 mg [Ru(C_{10}H_{8}N_{2})_{3}]Cl_{2}·6H_{2}O
330 mg K_{2}S_{2}O_{8}
2M HCl

CAUTION

Heat: As reaction progresses the solution will heat up, just like a light bulb. Be careful not to burn yourself and only hold the flask at the top away from the hot water solution.
2 M HCl is strong acid if it comes in contact with your skin immediately rinse the exposed area with water.

**Procedure**

Weigh out approximately 0.025 g [Ru(C\(_{10}H_8N_2\)_3]Cl\(_2\)·6H\(_2\)O into a 10 mL beaker and transfer with a small amount of water to a 125 mL conical flask. Add 28 mL water and 0.33 g K\(_2\)S\(_2\)O\(_8\) to the flask along with a Teflon stirring bar, and stir the solution on a magnetic stirrer. Adjust the pH to approximately 1 by the addition of 2 M hydrochloric acid. Now add approximately 12 small pieces of magnesium to the stirred solution. Observe in a darkened room.

![Chemical Structure](image)

**WASTE DISPOSAL**

All waste should be placed in the appropriately labeled waste bottle located in the lab.
Making of a Dye-Sensitized Solar Cell
(Grätzel Cell)

Purpose

Making a solar cell using conductive glass (which we made) and an inorganic dye which we also made. A combination of lab experiments to show modern renewable energy technology uses inorganic chemistry.

Introduction

The next page illustrates how the dye-sensitized solar cell functions. The student creates a thin film between two slides of conductive glass. The conductive glass aids in collecting electrons from the materials that will be placed in between the two glass slides (shown as the top plate in the second illustration on the next page) will be coated with titanium dioxide (TiO₂) by the teacher or student beforehand. The student will stain the plate by soaking it either in porphyrin or raspberry juice. The dye and the TiO₂ together will constitute a film about 10 micrometers thick.
The Experiment

Materials

**Equipment**

2 pieces of FTO conductive glass

**Chemicals**

2 g TiO₂

3 mL vinegar
Experimental Procedure

Preparation of Titanium Dioxide

Measure out 2 g of titanium dioxide on the scale using weighting paper and place into a mortar and pestle. Measure out 3 mL of vinegar and add 1 mL to the titania powder, thoroughly mixing afterwards. Repeat until all 3 mL are added. Add 2 or 3 drops of provided colorless soapy water to the solution and let the suspension sit for 15 minutes.

Making the Solar Cell

Clean both 1” square glass slides gently with ethanol and Kim wipes. Identify which side is conductive by using a multimeter. Place the glass slides on the bench, orienting one glass slide with its conductive side up, and another immediately below it with its conductive side down. Carefully keeping the slides touching, tape them to the lab bench using scotch tape, as shown, leaving most of the surface of the slides exposed and only covering a thin strip (2 mm wide) on either side. Place a third piece of tape over the top of the conductive slide, as shown, covering a larger area (about 5-6 mm wide):
Keep in mind that our goal in this step is to get a nice, smooth titania film on the conducting slide; any film on the non-conducting slide does not matter. Using a metal spatula take a small amount of the titania suspension you made earlier and spread it in a thin line just below the last piece of tape, on the conductive slide. Immediately take a glass stir rod, held horizontal as shown, and in contact with the tape, and slide it in order to spread the suspension smoothly, first moving downwards, then reversing direction. Do this 2-3 times or until the film on the conducting slide is smooth. Add more of the suspension if necessary, but do it quickly, as it will dry rapidly. If something goes wrong, you can carefully wipe the slides off and try again.
Carefully, remove the top piece of tape first, and then place your finger on the uncoated area where the tape just was, being sure not to touch the film. If you are worried that you may accidentally touch the film, ask an instructor for help. While the conductive side is held down this way, have someone else very carefully pull the other pieces of tape off, peeling from the top down. The non-conducting slide will come loose at some point. Take the slide whose conductive side has been coated with the titania film and cover it with a glass petri dish. You may leave it on the lab bench for this. Finish removing the tape from the other slide, and gently wash the titania suspension off of it and into the sink. Use ethanol and kim wipes to finish the cleaning, and clean the glass rod in the same way.

Once you are done cleaning things, the coated glass slide you placed under the petri dish on the lab bench should be dry. Take the dried glass slide and find an instructor to help you put it into an oven at 450 °C or use a blow dryer. The slides will be taken out of the oven by the instructors after 30 minutes of heating and allowed to cool.

Take a small petri dish and, using an eyedropper, put a small amount of blackberry juice or porphyrin solution on it. Take your completely cooled titanium dioxide coated glass slide and place it onto the dye in the dish, with the titania coated side face down. Make note of the time. While letting the film soak, take the other glass slide, identify the conductive side, and coat it with graphite using a pencil tip, lightly shading it. Make sure the slide is completely coated in graphite, including the corner.
By this time, your titania coated slide should have soaked for at least 10 minutes. Examine the slide from the back as it sits in the petri dish and ensure that the titania film now has a deep purple color throughout. If it does not, wait until it does. Holding from the edge, be sure to carefully wash the film thoroughly with deionized water. Once done, repeat using ethanol.

The next step is to construct the cell by taking the graphite-coated slide and placing it facedown on top of the dyed side of the other slide so that they are just offset, as shown in the figure below. Binder clips should be used to attach the two.

Once your cell is clipped together, you will need to add the electrolyte solution. Holding the cell such that one of the steps is facing upwards towards you, put on to two drops of the iodide electrolyte solution at the point where the two slides meet. You will see that the solution is slowly drawn into the cell by capillary action. In order to ensure that the solution completely saturates the cell, loosen one of the binder clips, then clip it on again, then loosen the other binder clip and
clip it on again. Repeat until the cell is clearly saturated with the iodine solution. Test in the dark and in the light using the multi-meter.

**WASTE DISPOSAL** Dispose of TiO2/fruit juice solution as directed by your Instructor/TA. Return the clean conductive glass pieces for reuse.
Molecular Gastronomy aka Food Chemistry

Purpose

The purpose of this experiment is to examine the chemistry present in everyday food. Recently the term molecular gastronomy has started to be used in reference to scientific style cooking but most of the equipment, techniques and methods used by these innovative chefs are right out of the chemistry laboratory.

Introduction

Increasingly food ingredient lists that you find in the supermarket look more and more like lists of chemicals. Who better to teach you a bit about what these things are than a chemist? Chemistry can be used in the kitchen to give you all the characteristics you want in a type of food without the natural tradeoffs. We will make a thick and creamy ice cream without using eggs or much cream by using plant based thickeners. Food scientists and chefs can also reduce costs by substituting chemical compounds that taste the same but cost less than the real thing, most people never notice until they read the label. Taken to the extreme molecular gastronomy can even turn your food into something completely new. Just freezing something in liquid nitrogen can completely change the flavor and texture of food and is something never observed in normal kitchens.

Molecular gastronomy is all about applying the techniques and practices of the chemistry lab to the kitchen and edible food.

The Experiments

We will use molecular gastronomy techniques to make ice cream as well as frozen foams.
Liquid Nitrogen Ice Cream:

Materials

120 g Sugar
0.4 g Locust bean gum
0.4 g Xanthan gum
0.1 g Carrageenan, Kapppa
550 g Milk
165 g Heavy cream
8 g Vanilla bean seeds (or 5 tsp Vanilla extract)
50 g Milk solids

CAUTION

Cryogenics: Liquid nitrogen is -196°C and will cause extreme frostbite almost immediately. Use only under the supervision of your instructor or TA. Remember liquid nitrogen is just many degrees below body temperature as fire is above body temperature. Liquid nitrogen should be treated with just as much care as an open flame. It is just as dangerous.

Experimental Procedure

Start large water bath heating to 95°C so that it is ready for later.

Mix all the dry powders together in your zip lock bag. Add milk and cream to the dry powders, seal the bag and carefully mix them all together. Suspend the bag filled with the mixture in the hot water bath and allow to sit for 30 minutes.

While waiting for ice cream base to heat move on to Root beer cryosicle.

After ice cream base has been heated for approximately 30 minutes remove from the water bath and place in a bowl chilled with ice. Add the Vanilla while the solution is still hot. Let the ice cream base cool while you whisk the solution. The cooled solution can then be cooled into ice cream using liquid nitrogen.

For more information on this ice cream preparation see:
http://www.chefsteps.com/activities/egg-free-vanilla-ice-cream
Root Beer Cryosicle:

Materials

285 g Root Beer Syrup
120 g Heavy cream
(recipe can be doubled and still fit in the whipping siphon)

CAUTION

Cryogenics: Liquid nitrogen is -196°C and will cause extreme frostbite almost immediately. Use only under the supervision of your instructor or TA. Remember liquid nitrogen is just many degrees below body temperature as fire is above body temperature. Liquid nitrogen should be treated with just as much care as an open flame. It is just as dangerous.

Experimental Procedure

Mix two ingredients together and place in the whipping siphon. Seal siphon and vent two CO$_2$ canisters into the siphon. Gently shake the siphon and then empty a third canister into the siphon. Check to see if you now have a foam. If solution is still runny add half of another canister and shake more. Check and repeat as necessary. After you confirm Foam formation you can place the siphon in an ice bath.

Chill a metal spoon in Liquid nitrogen and place a dollop of the foam on the spoon. Chill the base of the foam in the spoon by dipping in Liquid Nitrogen. Place a wooden stick into the top of the foam to act as a handle and then fully immerse the foam to freeze in the stick. Spoon extra Liquid nitrogen over the foam ball to make sure the outer surface is frozen. Remove foam by the attached stick.

Check foam to ensure that no liquid Nitrogen is trapped in foam ball. As a safety precaution, count to 5 after removing foam from liquid nitrogen before placing in your mouth.

For more information on this experiment see: http://www.chefsteps.com/activities/root-beer-float-cryosicle
Candy Bar Crunch:

Materials

Small candy bars
Liquid nitrogen

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CAUTION

Cryogenics: Liquid nitrogen is -196°C and will cause extreme frostbite almost immediately. Use only under the supervision of your instructor or TA. Remember liquid nitrogen is just many degrees below body temperature as fire is above body temperature. Liquid nitrogen should be treated with just as much care as an open flame. It is just as dangerous.

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Experimental Procedure

Place various fun size candy bars into liquid nitrogen. Allow to sit for about 1 minute. Hit candy bar with a spoon to shatter it and place the pieces on top of ice cream.

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WASTE MANAGEMENT

None of the experiments will generate any noxious wastes. Dispose of all uneaten food items in to the regular trash bin.