Title: Engineering Organic Light-Emitting Devices

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Appropriate Level: High School Regents Chemistry, or any advanced high school Chemistry course

Abstract: It is important for students to be made aware of current technological and commercial breakthroughs in their current field of study. This serves both to educate on the practical nature of science and to motivate students by introducing them to possible career options. Organic light-emitting diodes are making great strides in an effort to replace current displays such as those found in cell phones, MP3 players, and even television screens. These organic devices also may have a future serving as white-light sources with lower energy consumption and greater efficiency. This activity allows students to create a simple organic light-emitting device.

Time requirement: 90 – 120 minutes, spread over two days.

NYS Standards Met: Process Skills: S1.1, S2.1, S2.3, S3.3, 3.2vii
Performance Indicators: 3.2d, 3.2e, 3.2g

Sections to Document: Teacher Tips
Student Section

Required Materials:
Ruthenium-tris(2,2′-bipyridyl) dichloride (Sigma-Aldrich #544981)
Ammonium tetrafluoroborate
Gallium-indium eutectic (Sigma-Aldrich # 495425)
Indium-tin oxide coated glass slides
Polyvinyl alcohol
4.5-volt power supply
Cotton swabs

Suggested Materials:
Homemade spin coater
Beral pipettes
Oven (or controllable hot plates)

TIPS FOR THE TEACHER / INSTRUCTOR

Engineering an Organic Light-Emitting Diode

Placement in the Curriculum

Organic Light-Emitting Diodes require *a minimum* of basic understanding of several core chemistry concepts. As such, if this activity is being performed in a first-year Chemistry course, it should be done near the completion of the course. Using the New York State Regents Chemistry course core curriculum as a guideline, this experiment works best during the Oxidation-Reduction unit. The ruthenium complex is the chemical involved in both the oxidation and reduction half-reactions as it supplies a means for the electrons to move between electrons with just one compound (as opposed to the need for two separate compounds in most OLEDs).

Lab Techniques

Students must be familiar with using digital balances and Beral pipettes (or similar method for dispensing small amounts of liquid). Other steps in the lab all require careful attention to detail, but are not singularly complex.

Lab preparation

As with many chemistry labs, this lab requires some teacher setup prior to lab. Depending on the amount of time available for experimentation and the skill level of your students, more or less setup can be done prior to lab.

Preparation of $[Ru(bpy)_3](BF_4)_2$

The ruthenium complex is commercially available through supplies such as Sigma-Aldrich as the chloride salt (Ruthenium-tris(2,2′-bipyridyl) dichloride, Prod. #93307). The hexafluorophosphate salt is also available at this time, but the anion plays a large role in the success of the reaction. The end goal is to use tetrafluoroborate as the anion, but that still requires an ion-transfer reaction.

Synthesis

Dissolve approximately 0.5 g [Ruthenium-tris(2,2′-bipyridyl) dichloride] in 50 ml of distilled water. Dissolve approximately 0.5 g of ammonium tetrafluoroborate (NH_4BF_4) in the same solution. An orange precipitate should begin to form. This solid is your desired product. Allow ample time for the recrystallization to reach completion. Collect the crystals via vacuum filtration and wash with ethanol to remove as much water as possible.

Quantities needed

The quantities in the above synthesis are guidelines. The mass of the reagents can be changed proportionately depending on how many devices you plan to create. Since the tetrafluorborate ion is replacing the chloride ions, the theoretical yield is slightly greater than the mass of the Ruthenium-tris(2,2′-bipyridyl) dichloride dissolved in the first step of the synthesis, so you should expect to recover a similar, if not slightly greater, amount of product crystals.

The directions given in the lab are for generating 3.0 ml of solution with which to create OLEDs. If using a spin coater and one coat, 3.0 ml should be enough solution for 5-6 devices. Keep this in mind when preparing the stock ruthenium solutions.

Tips for smooth procedures and making adjustments

Several steps to the lab can be adjusted if necessary, based on class size and equipment available in the lab. Each adjustment will have some effect on the final outcome, so proceed with caution.

20 mg/ml PVA solution

Based on the time available in lab, it may be wise to prepare a stock batch of the polyvinyl alcohol solution ahead of time. The speed at which the PVA dissolves varies with its molecular weight, and sometimes needs to be stirred for up to an hour while being heated to 80°C (but not higher, so as not to boil to mixture). This can be done ahead of time in one liter batches to save lab time.

Filtering the PVA

If using a large molecular weight PVA you may find an undissolved film left behind. For this reason, it is best to filter the PVA solution before adding the ruthenium complex. During the initial research, a syringe and a 0.45чm PVDF filter were used, but using vacuum filtration will also work.

Cleaning the slides

In a research lab, much more time is spent on this step of the procedure to make sure the slides are quite clean. These simple steps are used to make sure the slides are void of fingerprints and other dirt and oil.

In the drying step, nitrogen gas would work best, but cans of compressed air are a viable alternative. This ensures that water droplets do not dry leaving odd patterns on the slides.

Applying the ruthenium layer

Using a spin coater is the ideal method for applying this ruthenium layer. A simple device can be made using an 80mm fan and simple circuitry. If a spin coater is not available, Q-tips and a heat gun can work to a certain degree:

> Use a Q-tip to apply some of the Ruthenium-tris(2,2′-bipyridyl) tetrafluoroborate to the glass slide and then dry with a heat gun for 3-5 minutes.

Drying the ruthenium layer

The presence of water plays a large role in decreasing the lifetime of OLEDs, if the water does not cause them to fail completely. Drying the slides in an oven at 120°C overnight is the preferred method. Alternatively, hotplates can be used if the temperature can be steadied near 120°C. If not, extended drying under a heat gun can be used. The temperature should not be allowed to rise above this temperature due to the behavior of polyvinyl alcohol at high temperatures.

Making electrical contacts

Several top electrodes were tested, and the gallium-indium eutectic (Sigma-Aldrich product #495425) works best. The main difficulty when working with the eutectic is that care needs to be observed since its melting point is around 16°C. Students must remember they have a liquid contact on the slide and cannot flip the slide over for viewing.

The contact works best if one lead is touching the indium-tin oxide layer and one lead if touching the eutectic. If the ruthenium layer completely covers the slide, one edge of the ruthenium layer should be wiped clean with water and a Q-tip to expose a strip of ITO.

Safety

There are no known safety hazards at this time. The eutectic can be disposed of in the garbage cans. Students should take care to wash their hands if their skin comes into contact with any of the chemicals in the experiment.

Name: ________________________

Date: ___________ Period: ______

Engineering an Organic Light-Emitting Diode

Introduction

Organic light-emitting diodes (OLEDs) are among a lengthy list of hot technologies currently being researched among the world's top universities and corporations. In order to develop new state-of-the-art products, researchers must always stay on the cutting-edge and continue to explore new ideas. Organic light-emitting devices are currently being explored as possible replacements for various displays and monitors (they are already used as the screens on some digital cameras, MP3 players and stereo faceplates), as well as a possible replacement for the common light bulb.

Organic light-emitting diodes have several advantages over traditional LEDs and other display technologies, such as LCD (liquid crystal display) screens. First, organic devices emit their own light (remember our discussions about atomic structure and fireworks!) so there is no need for backlighting. Second, the devices can be manufactured using solution processing techniques, including the use of ink-jet printer technology. This allows for quicker, more reliable manufacturing.

In this lab, you are going to manufacture a simple OLED and test its intensity and lifetime, two desirable criteria for analyzing such devices. These OLEDs are sensitive, and the procedures must be followed carefully.

Procedure

A. Solution Preparation

1. Dissolve 0.060 g PVA (polyvinyl alcohol) into 3.00 ml of distilled water. Heating the mixture up to 80°C and stirring can increase the rate of dissolution.
2. Filter the PVA solution to remove any chunks. An inline filter works best, but if one is not available use vacuum filtration.
3. Dissolve 0.060 g ruthenium (II) tetrafluoroborate $\mathbf{[Ru(bpy)_3](BF_4)_2}$ into the PVA solution.

4. Record the exact masses of the PVA and $[Ru(bpy)_3](BF_4)_2$ used and calculate the (wt%) of each solute.

B. Preparation of the slide

Gloves should be worn at all times when handling the slides.

1. The slides must be cleaned sequentially with three separate solutions. First, using a cotton swab, wash both sides of the ITO (indium-tin oxide) slide with ammonia-based glass cleaning solution.
2. Next, using a fresh cotton swab, wash both sides of the slide with isopropanol.
3. Third, with a new swab, wash both sides of the slide completely with distilled water.
4. Dry the slide using a can of compressed air.
5. Use double-stick tape to attach the ITO slide to the middle of the spin coater's fan.
6. Using a Beral pipette drop approximately 0.4 ml $[Ru(bpy)_3](BF_4)_2$ / PVA solution onto the slide. Close the cover and turn the spin coater on for 30 seconds.
7. Remove your slide and remove the tape. Place your slide in the oven to bake overnight at 120°C.

C. Testing your device (Day 2)

1. If you slide is completely covered with the organic layer, use a cotton swab and distilled water to expose a small section of the ITO layer.
2. Use a fresh cotton swab to drop a dot of gallium-indium eutectic (alloy) to add the top electrode.
3. Touch one lead of a 4.5-volt power supply to the ITO glass (not the coating) and gently touch the other lead to the gallium-indium eutectic.

Tip: Raising the glass slide slightly should allow you to better see the light while keeping the eutectic from running down the slide!

4. Record observations of what happens while the voltage is applied. Make sure to comment on color, intensity, lifetime and any other significant information.

Data

Mass of PVA used:

Mass of $[Ru(bpy)_3](BF_4)_2$ used:

Concentrations of the solution, in wt% (show your work):

Observations:

Questions:

1. What effect do you think humidity has on OLED lifetime? Why?

2. Name two existing technologies which utilize organic light-emitting diodes and propose one future application. You may need to utilize the internet and remember the main benefits are low power consumption and they produce their own light.

3. The ruthenium complex acts as both the cathode and the anode in this OLED. Explain how this enables a flow of electrons from the top electrode to the indium-tin oxide layer on the glass.