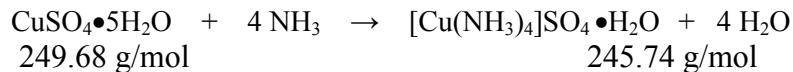


Experiment 9

Synthesis of a Copper Coordination Complex and Aspirin with Demonstrations of the Synthesis of Nylon, Bakelite, and Polyvinyl Alcohol Slime

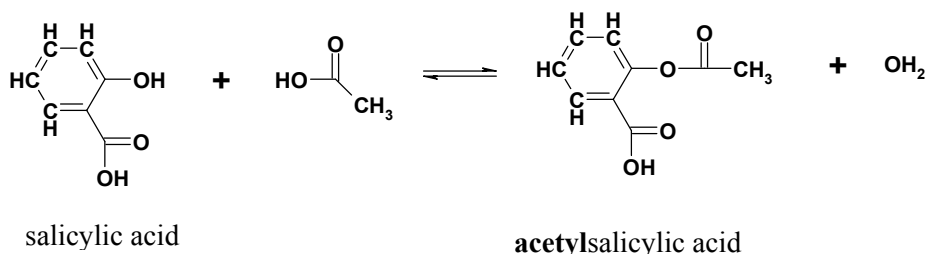
Synthesis of tetraamminecopper(II) sulfate, [Cu(NH₃)₄]SO₄

The reaction for making tetraamminecopper(II) sulfate and some molar masses are:

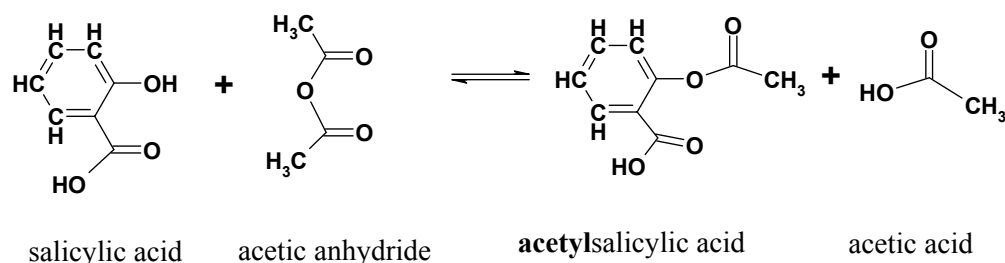


Synthesis of Aspirin

Organic syntheses often involve the addition of a functional group to a molecule. An example is the synthesis of acetylsalicylic acid, the compound known as aspirin. Aspirin can be synthesized by combining salicylic acid with acetic acid. The reaction results in the formation of an ester (see table of functional groups shown).



A preferred method for synthesizing aspirin is to use acetic anhydride instead of acetic acid. Phosphoric acid can also be added to catalyze the reaction.



Procedure

Eye protection must be worn during this lab. Students work in pairs.

Use graduated cylinders for all volume measurements.

Concentrated ammonium hydroxide should be added in the fume hood to reduce the emission of ammonia vapor in the laboratory.

Part 1: Synthesis of an Inorganic Compound

Eye protection must be worn during this lab. Students work in pairs.

1. Obtain about 3 g of copper(II) sulfate pentahydrate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ using an analytical balance. Record the color and mass of the compound.
2. Dissolve the compound in 15 mL of distilled water in a 125 mL Erlenmeyer flask. Use a magnetic stir bar to ensure that the complex dissolves completely. Note the color of the solution in the Data Table.
3. Once the solid has completely dissolved, add 15 mL of concentrated ammonium hydroxide (in the fume hood) with stirring and note any color change in the Data Sheet.
4. To induce crystal formation, add 15 mL 95% ethanol with stirring and note what happens in the solution. Is this reaction exothermic or endothermic?
5. Allow the crystals to settle for 15 min.
6. Obtain 10 mL of 95% ethanol to wash the product.
7. Collection of the crystals is performed using a Hirsh funnel apparatus with filter paper. The apparatus should be attached to the laboratory vacuum line.
8. Wet the filter paper with 5 mL of ethanol to seat it against the holes in the funnel, and turn on the vacuum. Use a stirring rod to get as much of the solid transferred from the beaker to the funnel. Allow the suction to remain on until the flow of liquid through the funnel ceases.
9. Wash the crystals by slowly dropping 10 mL of ethanol using a plastic pipette.
10. Allow the suction to continue for 10-15 minutes.
11. Place an 8 cm filter paper on a watch glass and measure its mass. Record this mass in the Data Sheet.
12. Turn off the vacuum and carefully remove the filter paper with the solid on it from the Hirsh funnel. Place the solid, without its filter paper, on the 11 cm filter paper that was weighed with the watch glass.
13. Place the watch glass with the product into the drying oven for 45 min. After removing the watch glass from the oven, allow it to cool to room temperature.
14. Measure the mass of the watch glass with the product and record the value in the Data Sheet.

Part 2: Synthesis of an Organic Compound

Eye protection must be worn during this lab. Students work in pairs.

Start by heating 300-mL of water in a 600-mL beaker to a temperature of between 75-80 deg C.

While the water is heating, obtain 2.5 g of salicylic acid that is in a 125-mL Erlenmeyer flask.

Add 5 mL of acetic anhydride to the Erlenmeyer flask followed by 5 drops of 85% phosphoric acid. Heat the Erlenmeyer flask in the 600 mL beaker for about 10 min, **stirring the contents occasionally**.

Remove the flask from the hot water bath and immediately add, drop by drop, being extremely careful, 2 mL of water to the flask. **Some spattering is to be expected so be very careful not to burn yourself.**

When the reaction in the flask subsides, add 30-40 mL of ice water (ice mixed with water) to the reaction mixture and wait until you see a milky cloudiness. This is the beginning of crystallization. It may help initiate crystallization if you scratch the wall of the flask at the solution's surface with a stirring rod.

After you get the cloudiness, place the flask in another 600-mL beaker that contains an ice/water mixture. Rotate the flask in the ice water to ensure uniform cooling and completeness of precipitation. After you think that crystallization is complete, you will filter the product through a Hirsh funnel. To do this, put a piece of filter paper at the bottom of the funnel, wet it with water, and turn on the vacuum. Pour the aspirin slurry into the funnel using a stirring rod to transfer as much aspirin as possible. After all the water has been drawn through the funnel, turn off the vacuum.

Pour an additional 25 mL of cold water into the funnel and carefully stir to wash the product. Restart the vacuum. When all of the water has been removed, transfer the product to a dry piece of filter paper and allow it to dry at room temperature for a least 25 min.

You can tell if you have washed the aspirin enough if it has no smell of acetic acid (vinegar). If you could remove all traces of acetic acid, you could press the aspirin into tablets and use it as an analgesic. However, your aspirin is still a bit crude, so the instructor will collect it for use in the organic chemistry lab. The organic students will further purify the aspirin by re-crystallization.

Data Sheet
Experiment 9
Synthesis of an Inorganic and Organic Compound

Synthesis of Tetraamminecopper(II) sulfate monohydrate

	Mass and % yield	Colors	
Mass of copper(II) sulfate pentahydrate	g	solid	solution
Mass of product + watch glass and filter paper	g	No entry	
Mass of watch glass and filter paper	g	No entry	
Mass of product, tetraamminecopper(II) sulfate	g	In solution	solid
Is the reaction exothermic or endothermic? (Circle one)	Exothermic	Endothermic	
Theoretical yield	g	No entry	
Percent yield	%	No entry	

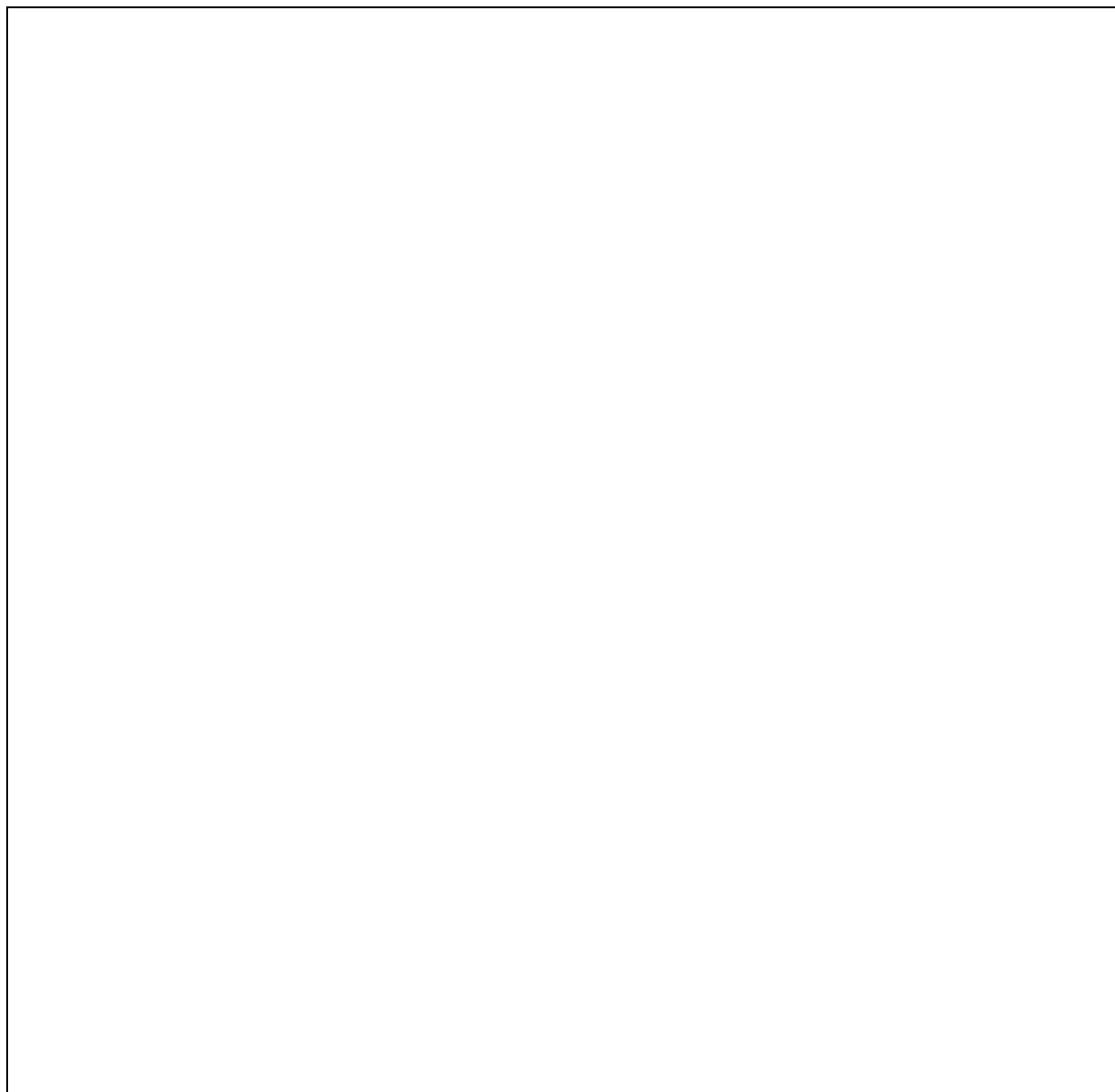
Organic Molecules (These will be passed around in small bottles for you to look at and smell.)

Substance	Chemical Structure	Functional Group	Odor	Uses
Acetone				
Ethanol				
Isopropanol				
Acetic acid				
Benzaldehyde				
Ethyl acetate				
Amyl acetate				
Phenol				
Methyl salicylate				
Trimethylamine				
Camphor	ketone			

Synthesis of Nylon 610

This will be done as a demonstration. Your instructor will pour hexamethylenediamine (also known as 1,6-hexanediamine, $C_6H_{16}N_2$) down the wall of a slightly tilted beaker that contains an aqueous solution of sebacyl chloride (also known as decanedioyl dichloride, $C_{10}H_{16}Cl_2O_2$), to form two layers. A polymer film of nylon will form between the two liquids. This nylon is called nylon 610 because there are six carbon atoms in the hexamethylenediamine and 10 carbon atoms in the sebacyl chloride.

This reaction is an example of a condensation polymerization. See p 1069 in your textbook for a photograph of the reaction. (In the photograph, they are making nylon 66. Can you see why?) Your instructor will write the equation for the reaction on the chalk board. Copy it in the box below.



e at

Procedure

On a tray inside the hood, add the following to a 600-mL beaker:

- 80 mL of liquefied phenol (C_6H_5OH)
- 80 mL of formaldehyde (HCHO)

- 40 mL of glacial acetic acid
- 30 mL of concentrated hydrochloric acid

Using a stirring rod, stir the mixture for about one minute and leave the stirrer in the beaker. **Close the hood and stand away and wait.** This reaction takes a few minutes, so be patient. A solid pink phenol–formaldehyde condensation polymer forms with some spattering. The reaction is very exothermic. You can read more about Bakelite on page 1064 in your textbook and on the web site <http://www.let.uu.nl/ams/xroads/bakelite.htm>. The chemistry of the reaction is rather difficult and will not be discussed in this lab. After the reaction is completed:

- Add cold water to the beaker to dilute any unreacted material.
- You can poke holes in the Bakelite with a glass rod, but do not touch it with your bare hands.
- The Bakelite can be discarded in the trash after being washed.

Synthesis of Polyvinyl Alcohol Slime — Although not dangerous, this will done as a demonstration.

Materials

A 4% solution of sodium tetraborate (Borax). This solution can be prepared by dissolving 4 grams of Borax in 100 mL of water.

A 4% solution of polyvinyl alcohol. This solution is prepared by dissolving 4 grams of polyvinyl alcohol (PVA) in 100 mL of water. The PVA does not dissolve well so heating and stirring are required. For detailed instructions on how to dissolve PVA, see http://www.murraystate.edu/kate/Magic/polyvinyl_alcohol.htm.

Note: PVA comes in a variety of average molar masses. Our PVA has an average molar mass of between 124,000 and 186,000 g/mol. Higher molar masses produce a "stiffer" slime while lower molar masses produce a more fluid, i.e., "slimier" slime. About 345 million pounds of PVA is produced each year. Search www.google.com for more information on PVA.

Procedure

The alcohol and borax solutions should be mixed in the ratio of about 200 mL of PVA solution to 15 mL of borax solution. Prior to mixing, food coloring can be added to the PVA solution to make different colors of slime. Best results are obtained if the borax solution is added to the PVA solution. The 200:15 ratio can be achieved by the following English volume measurements:

- (1) one-half cup of PVA solution to one tablespoon of borax solution.
- (2) one-quarter cup of PVA solution to 1.5 teaspoons of borax solution.

Polyvinyl alcohol is a polymer, a very long molecule rather like a very long piece of string. The borax joins the polymer molecules together to make a large network— rather like a flexible scaffolding. This is called cross-linking. This turns the water dissolved polymer into a gel (the cross-linked polymer incorporates water into its matrix) which in this case is called Slime.

Note: The reaction is rather slow. Wait about ½ hour for the sliminess to develop.

Nice Things About Slime

- Materials can be cleaned up with hot water and soap or detergent.
- Slime can be stored for fairly long periods of time in sealed plastic sacks to prevent evaporation.

- Slime is not poisonous, but since it does contain small quantities of a mildly toxic material (Borax), we would not advise leaving it around unattended children.
- Slime is fun. Students like to play with it, while teachers like to "work" with it. Hospitals use "soluble" laundry bags which also consist of PVA.
- Boric acid and Boraxo seem to work as well as Borax.
- Slime is used to "fix" holes in flat tires.

In the box below, copy the polymer structure of polyvinyl alcohol from the chalkboard.

