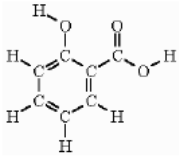
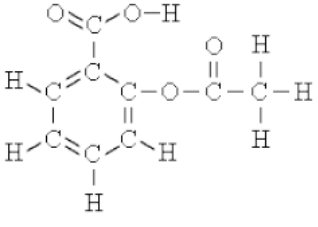
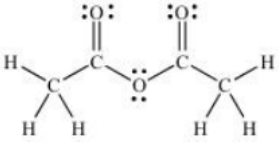
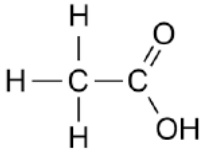
Matter and Motion
Fall 2015
Chemistry Lab 6: Synthesis of Aspirin

Overview: In this experiment we will synthesize an over the counter drug - Aspirin - by the reaction between salicylic acid and acetic anhydride. Salicylic acid can be derived from various plants including willow bark and has been used for its medicinal properties since ancient times. Aspirin has a slight chemical modification that eases some of the side effects of salicylic acid. The modification is the replacement of the hydroxide (-OH) group on the benzene ring (6-carbon ring with delocalize pi bonds) of salicylic acid with an acetyl group (-$COCH_3$). This reaction is known in organic chemistry as esterification. This experiment will give you a brief preview of organic chemistry and give you an opportunity to apply your knowledge of stoichiometry and calculate percent yield.

We will use two starting materials (**reactants**) to make two products. Since aspirin is our product of choice, the second product (acetic acid) is called a byproduct.

Reactants	Products
salicylic acid ($C_7H_6O_3$) 	Aspirin (acetylsalicylic acid) ($C_9H_8O_4$) 
acetic anhydride ($C_4H_6O_3$) 	acetic acid (CH_3COOH) 

PRELAB QUESTIONS
Read the entire lab carefully (including the Post-lab) then complete the Pre-lab before coming to lab. It should be completed in your chemistry lab notebook and presented for a check as you walk in the door.

1. Some of the words in this lab may be new to you. Make a list of any new vocabulary and their definitions.
2. Read the lab carefully. Describe all of the data/measurements you will take during this lab. Be as specific as possible, i.e, include the units used and the number of times each measurement made.
3. Define a "catalyst" in a chemical reaction. Cite your source properly.
4. Write a balanced chemical equation for the synthesis of aspirin.

SAFETY:
- Wear eye protection at all times!
- Beware of reaction fumes! Keep reaction vessels under the hoods at all times!
- Beware of concentrated sulfuric acid! It will burn. And it will ruin your clothes.
- Exercise caution around hot surfaces.

Experimental Procedure (work in pairs)
1. Weigh out 2.0-3.0 g of salicylic acid into a 125 ml Erlenmeyer flask. Record the weight accurately.
2. Measure out 5.0 ml of acetic anhydride (density = 1.08 g/mL). Record the volume accurately. **In the hood** add this to your Erlenmeyer flask.

3. **Wear gloves. Still in the hood**, carefully add 5 drops of concentrated sulfuric acid (a catalyst), to the flask and swirl to mix everything thoroughly.
4. **Still in the hood**, swirl the flask gently till the salicylic acid dissolves. Heat the flask gently on a hot water bath at about 50 °C for at least 10 minutes.
5. Allow the flask to cool to room temperature, during which time the aspirin crystals should begin to form. If it does not, scratch the walls of the flask with a stirring rod to induce crystallization
6. When the crystal formation is complete (usually when the product appears as a solid mass), add 50 mL of DI water and cool the flask in an ice bath.
7. Weigh a filter paper **with** a Buchner funnel and collect the product by vacuum filtration as instructed in lab. Slightly moisten the filter paper to make it "seal" with the funnel. The filtrate can be used to rinse the Erlenmeyer flask repeatedly until all the crystals have been collected.
8. Rinse the crystal several times with *small aliquots* of cold DI water. Allow the air to be drawn through the solid and filter paper for 15 minutes.
9. When the crystals are reasonably dry, weigh the crude product on a watch glass.
10. Take a small amount of your product for the following experiment. Obtain 3 small test tubes. Add 0.5 mL of water to each test tube. Dissolve a small amount of salicylic acid in the first tube. Add a similar amount of your aspirin to the second tube. Keep the third tube as the control. Add one drop of 1% iron(III) chloride solution to each test tube and note the color after shaking. Make inferences based on your observations.

POSTLAB QUESTIONS:

Please show your work/calculations clearly. The Postlab is due in your lab notebook at 9 am Tuesday, Dec. 1st.

1. Write a balanced reaction for the synthesis of aspirin. Keep in mind what is actually a reactant. Should the catalyst be written in the chemical equation?
2. Use your masses of reactants used to determine the limiting reactant.
3. Calculate your theoretical yield of aspirin.
4. Calculate your percent yield by comparing your actual and theoretical yields.
5. Comment on your results from the iron(III) chloride test (step 10 above). Would you consider consuming the aspirin you synthesized? Why or why not?
6. Why does scratching the inside of the reaction flask with a glass rod induce crystallization?