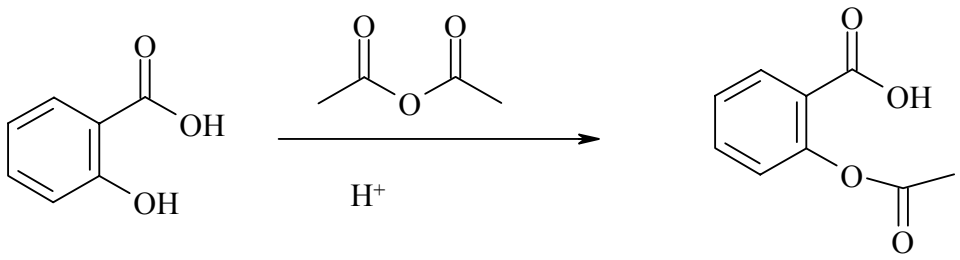
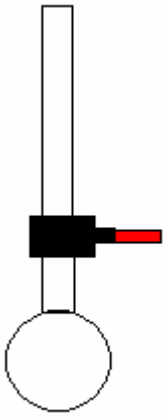
The Synthesis of Aspirin



1. In a round bottom flask, mix 138 mg of salicylic acid with 1 drop of 85% phosphoric acid and 0.30 ml of acetic anhydride. Attach a condenser to your flask to create a reflux apparatus.
2. Heat the mixture at about 90° C in a hot water bath for about 5 minutes. TLC to look for starting material and product. If the starting material has disappeared, continue with the next step. If there is still starting material present, continue the heating process. You may have to add acetic anhydride if you are unable to get rid of the starting material.
3. Add 0.2 ml of water to decompose the un-reacted acetic anhydride.
4. Add 0.3 ml of water.
5. Cool and look for crystals. You may have to induce crystallization by scratching the side of the flask or by adding a seed crystal. Isolate using a Hirsh Funnel.
6. Check MP (128 -137°C)



Calculations: Do not forget to include theoretical yield,, % yield and an Rf calculation.

Post lab Questions:

1. Hydrochloric acid is a strong acid and comes as a 30% solution in water. Why can't we use it instead of phosphoric acid?
2. Write a mechanism for this reaction.
3. How could you further purify the crystals obtained from this synthesis?
4. What is the reaction that occurs in step 3? (Water + acetic anhydride under acidic conditions.) Write an equation but a mechanism is not required.
5. Write a mechanism for the formation of aspirin.
6. How do you account for the smell of vinegar when an old bottle of aspirin is opened? Write an equation and a mechanism.