In the first week of a two-week experiment you will synthesize aspirin (acetyl salicylic acid). The following week you will determine the percent yield of your aspirin and then analyze your product for purity by thin layer chromatography and by a melting point determination.

Though esters can be produced from the direct esterification of an alcohol and a carboxylic acid in the presence of an acid catalyst, typically sulfuric acid, the present method uses a derivative of acetic acid (acetic anhydride) to form more quickly an acetate ester with salicylic acid (the alcohol donor of the ester) as shown below:

![Diagram of the reaction]

**Procedure:**

1. Place ~50 mL of distilled water into a 250 mL beaker, add a couple of boiling chips and heat to boiling. Also put ~25 mL of distilled water in a 50 mL Erlenmeyer flask into an ice water bath using another 250 mL beaker.

2. Write your group number in pencil on a piece of filter paper. Tare the balance and weigh the filter paper to the nearest 0.001 g; record this value in your notebook. Obtain ~1 gram of salicylic acid and place it into a tared plastic weigh boat. Weigh the salicylic acid to the nearest 0.001 g and record this value in your notebook.

3. Place the salicylic acid into a clean dry medium test tube. Add 2 mL of acetic anhydride and 2 drops of concentrated sulfuric acid. Caution: acetic anhydride produces irritation and necrosis of tissues in liquid or in vapor state. Avoid contact with skin and eyes. Do this addition in the hoods.
4. Place the test tube containing the reaction mixture into the boiling water bath. Stir the mixture vigorously with a clean glass rod while in the boiling water bath. Be careful not to break the test tube.

5. After all of the solid has dissolved, remove the test tube from the boiling water bath and let it cool to room temperature. Place the test tube into the ice water bath. If crystals do not form, induce crystallization by scratching the inside of the test tube with a glass rod. When crystallization is complete add 10 mL of the ice cold water.

6. Fold your weighed, initialed filter paper so that it is fluted (your instructor will demonstrate this) and place it into a short stem funnel.

7. Collect the solid on the filter paper in the funnel. Rinse the solid with 2 or 3 small (5 mL) portions of ice cold water; be sure to let the water drain through the filter between additions of the rinses.

8. Carefully remove the filter paper from the funnel and spread it out on a piece of paper towel. Set the paper towel with the filter paper in a drawer and allow it to dry until the next laboratory period.

9. Determine the gram formula weight for salicylic acid. Using the mass of your salicylic acid, determine the number of moles in the reaction. Determine the gram formula weight for the acetyl salicylic acid product. Finally, calculate the mass of product expected if all of the salicylic acid is converted to acetyl salicylic acid.

10. Your instructor will demonstrate how to prepare a sample for a melting point determination and the use of the melting point apparatus. Practice taking melting points using the salicylic acid.

11. The following week carefully remove the filter paper containing the aspirin from the drawer and weigh it to the nearest 0.001 g; record this value in your notebook. Save the aspirin for further analysis.