ORGANIC CHEMISTRY LABORATORY
ACETYLSALICYLIC ACID (ASPIRIN)
"Synthesis Experiment"

(402-247-9858)

Actual Experiment: HAND OUT SHEET

Concepts Include:

Reaction mechanism for preparing aspirin, calculation of excess and limiting reagent, theoretical yield, actual yield, percent yield of aspirin, filtration, recrystallization, and melting point.

Data:

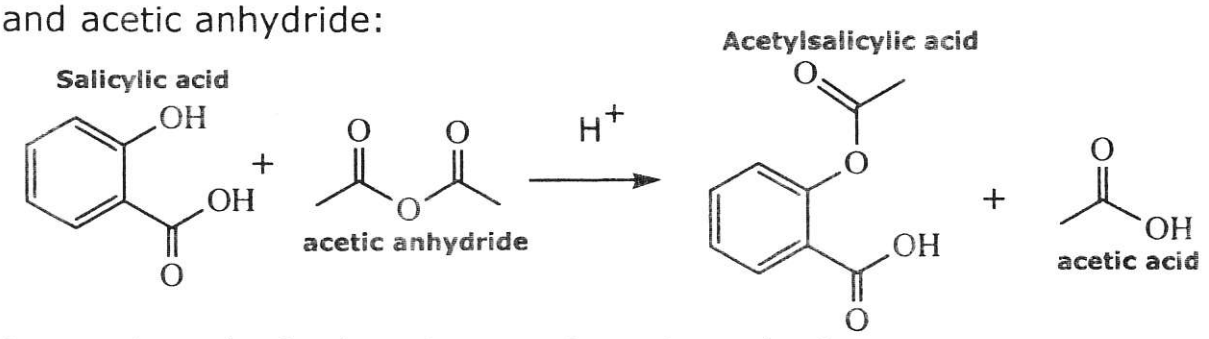
Calculation of reagent in excess and limiting reagent
Theoretical yield aspirin
Actual yield aspirin
Percent yield of aspirin (wet cake)
Percent yield of aspirin (recrystallized)
Melting point recrystallized aspirin

Questions:

Answer questions 1-5.

Acetylsalicylic Acid

Aspirin (acetylsalicylic acid) can be prepared by the reaction between salicylic acid and acetic anhydride:



In this reaction, the hydroxyl group (—OH) on the benzene ring in salicylic acid reacts with acetic anhydride to form an ester functional group. Thus, the formation of acetylsalicylic acid is referred to as an esterification reaction. This reaction requires the presence of an acid catalyst, indicated by the H^+ above the equilibrium arrows.

Procedure

Preparation of Acetylsalicylic Acid (Aspirin). Weigh 2.000g of salicyclic acid (MW= 138.1) and place this in a 125-mL Erlenmeyer flask. Add 5.0 ml of acetic anhydride (MW= 102.1, d 1.08 g/mL), followed by 5 drops of concentrated sulfuric acid, and swirl the flask gently until the salicylic acid dissolves.

Heat the flask gently on the steam bath or in a hot water bath at about 50°C for at least 10 minutes. Allow the flask to cool to room temperature, during which time the acetylsalicylic acid should begin to crystallize from the reaction mixture. If it does not, scratch the walls of the flask with a glass rod and cool the mixture slightly in an ice bath until crystallization has occurred. After crystal formation is complete (usually when the product appears as a solid mass), add 50 mL of water and cool the mixture in an ice bath.

Vacuum Filtration. Collect the product by vacuum filtration on a Büchner funnel The filtrate can be used to rinse the Erlenmeyer flask repeatedly until all crystals have been collected. Rinse the crystals several times with small portions of cold water. Continue drawing air through the crystals on the Büchner funnel by suction until the crystals are free of solvent (5-10 minutes). Remove the crystals for air drying.. Weigh the crude product, which may contain some unreacted salicylic acid, and calculate the percentage yield of crude acetylsalicylic acid(MW 180.2) assuming that the wet cake is about 50% solids.

Acetylsalicylic Acid

Recrystallization. Water is not a suitable solvent for crystallization because aspirin will partially decompose when heated in water. Dissolve the product in a minimum of hot ethyl acetate (no more than 2-3 mL) in a 25-mL Erlenmeyer flask, while gently and continuously heating the mixture on a steam bath or a hot-plate.

When the mixture cools to room temperature, the aspirin should crystallize. If it does not, add petroleum ether to the solution and cool the solution in ice water while scratching the inside of the flask with a glass rod until the product crystallizes. Collect the product by vacuum filtration, using a Büchner funnel. Any remaining material can be rinsed out of the flask with a few milliliters of cold petroleum ether. Dispose of the residual solvents in the waste container for nonhalogenated organic waste.

Determine the melting point of your product. The melting point must be obtained with a completely dried sample. Pure aspirin has a melting point of 135-136°C.

Questions.

1. What is the purpose of the concentrated sulfuric acid used in the first step?
2. What would happen if the sulfuric acid was left out?
3. If you used 5.000 g of salicylic acid and excess acetic anhydride in the preceding synthesis of aspirin, what would be the theoretical yield of acetylsalicylic acid in moles? In grams?
4. Most aspirin tablets contain five grains of acetylsalicylic acid. How many milligrams is this? 1 grain = 0.0648 g
5. If the aspirin crystals were not completely dried before the melting point was determined, what effect would this have on the observed melting point?

Preparation of Acetyl Salicylic Acid

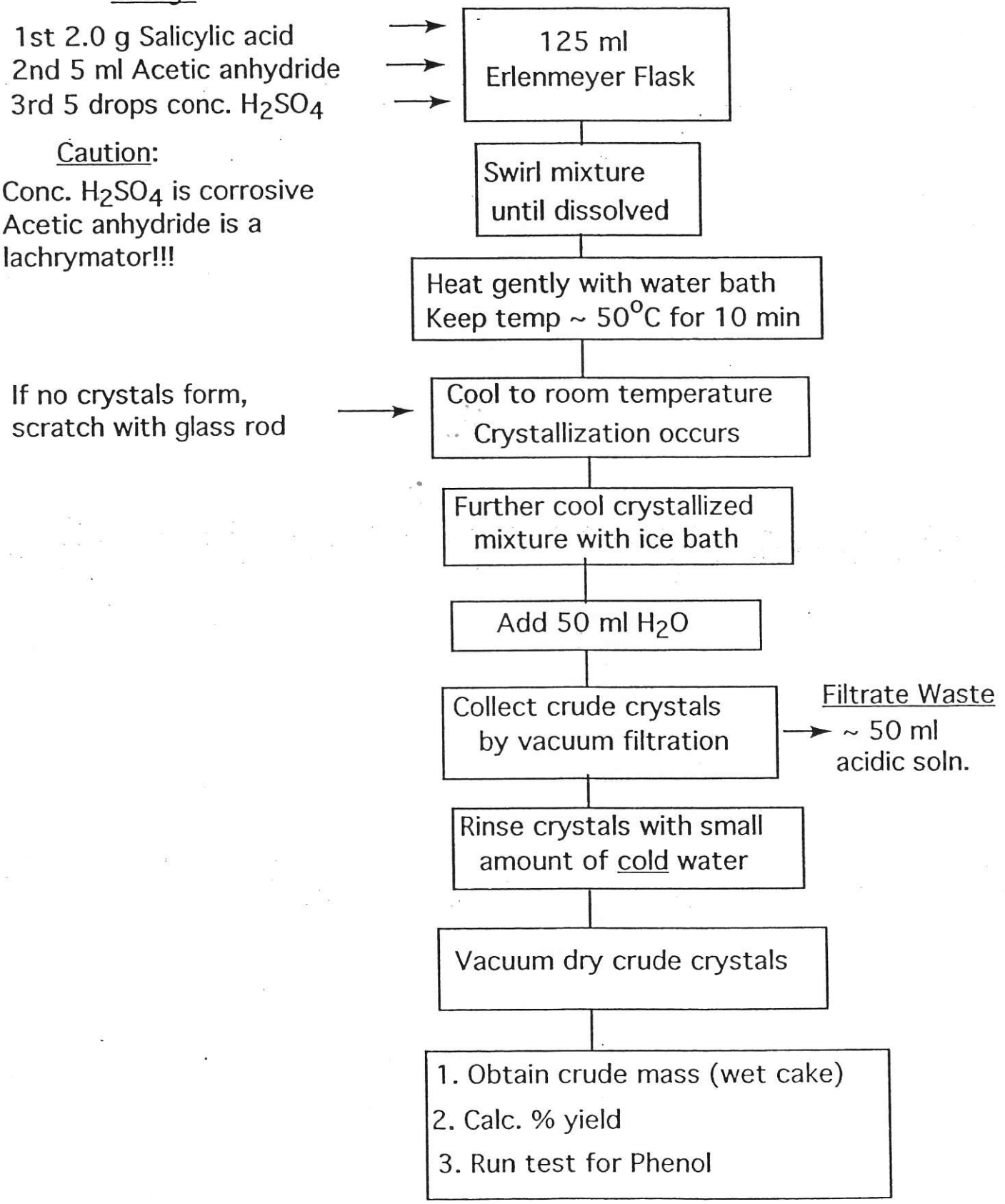
Reaction Flow Diagram and Isolation of Crude Product:

Charge

1st 2.0 g Salicylic acid
2nd 5 ml Acetic anhydride
3rd 5 drops conc. H_2SO_4

Caution:

Conc. H_2SO_4 is corrosive
Acetic anhydride is a lachrymator!!!



Preparation of Acetyl Salicylic Acid
Flow Diagram: Recystrallization of Crude Product