

## **Flow Chart Exercise** (Aspirin Synthesis Procedure)

Prepare a flow chart for the following procedure.

### **Synthesis of Aspirin.**

Prepare a hot water bath by placing about 150 mL of water in a 400 mL beaker. Add a boiling chip. Heat the water to boiling on a hot plate. Half fill a 100 mL beaker with ice. Add water to make up to 50 mL.

While the water is heating, accurately weigh about 3 grams of salicylic acid into a 125 mL Erlenmeyer flask. Take the flask to the fume hood where your instructor will add 5 mL of acetic anhydride and 5 drops of concentrated  $\text{H}_2\text{SO}_4$ . Swirl the flask to wet the crystals. When the water is boiling, place the flask in the water bath. Clamp the neck of the flask to the ring stand so that it is secure and does not float in the water bath. Heat for 10 minutes.

Carefully remove the flask from the water bath and add 10 mL of the ice water to decompose any excess acetic anhydride. Chill the solution in an ice bath until the crystallization is complete. Stir occasionally.

Assemble a vacuum filtration set up. Decant the liquid into the funnel leaving the crystalline aspirin in the flask. Add 10 mL of ice water to the flask, swirl, and pour off into the funnel with the aspirin. Use your spatula to scrape the remainder of the aspirin into the funnel. Add an additional 5 mL of ice water to the flask to wash any residual aspirin into the funnel. Leave the suction on in order to draw air through the funnel and dry the aspirin for an additional five minutes.

Release the vacuum by moving the vacuum tubing from the filter flask. Scrape the aspirin onto a pre-weighed watch glass. Weigh the watch glass and crude product to determine the crude yield of the product.

### **Recrystallization of Crude Aspirin.**

Transfer the crude product to a 125 mL Erlenmeyer flask. While swirling the flask, add, by drops, the minimum amount of hot ethanol to dissolve the crude product. Place a boiling stick in the flask. Warm the mixture gently on the hot plate to bring the ethanol just to a boil. Turn off the hot plate. Slowly, add water from a dropper until solid begins to form. Then turn on the hot plate and add an additional few drops of ethanol until the solid redissolves. Remove the flask from the hot plate, stopper it lightly, and let stand to cool. Beautiful needle-like crystals should form. Let the solution stand until next period.

Filter your crystals by vacuum filtration and allow them to dry as much as possible with the vacuum. Transfer the crystals to a pre-weighed weighing bottle and determine the weight of purified product.

**Procedure Writing Exercise**  
(Buffer Preparation and Analysis)

Convert the following procedure into an acceptable laboratory report format.

**Calibrate the pH meter.**

Calibrate the pH meter with the pH 7 and pH 4 standard buffers.

**Determine the concentration of a commercial vinegar solution.**

After calibrating the pH meter, rinse a clean 5 mL pipet with the vinegar and then pipet a 5 mL aliquot into a clean 150 mL beaker. Add about 70 mL of distilled water to the beaker. Rinse the magnetic stir bar with distilled water, and gently place the bar in the beaker. Put the beaker on the magnetic stirrer. Add the titrant to the beaker in small amounts, and record the amount of base added and the resulting pH in your notebook. For a significant portion of the titration there will be a very slow change in pH. Monitor the titration by monitoring the pH. Take a reading every 0.2 pH units or 0.5 mL (which ever occurs first). Record the pH and volume at each increment. Titrate to pH of 12.

**Prepare an acetic acid/acetate buffer solution.**

Using a graduate cylinder, transfer 20 mL of commercial vinegar to a 150 mL beaker. Place the beaker on the magnetic stirrer. Position the electrode in the beaker above the stir bar and turn the stirrer on slowly. Using a graduated cylinder, add 90% of the volume of base required to reach pH 4.74. Then add base from a dropper until you reach a pH of 4.74. Remove the electrode. Set the beaker aside. Label it "stock buffer solution".

**Analysis of buffer solution.**

Pipet a 10 mL aliquot of your buffer solution into a 100 mL beaker. Add 20 mL of water. Titrate the solution with standard 0.2 M HCl to a pH of 2. Be sure to record the exact concentration of the HCl that you used. Note that the pH during this titration will decrease as you add the standard acid. Label this data table "buffer-acid".

Drain the remaining acid from your buret. Rinse it well with tap water then with distilled water. Then rinse it three times with a few milliliters of the standard 0.2 M base solution. Then fill the buret with the 0.2 M base.

Pipet a 10 mL aliquot of your buffer solution. Add 20 mL of water. Titrate the solution with your standard base solution to a pH of 12. Record the exact concentration of the NaOH that you used. Label this data table as "buffer-base".