Preparation of Aspirin

Background

Aspirin has been used for years as a medicine. It is effective in reducing pain (analgesic), reducing inflammation (anti–inflammatory), reducing fever (antipyretic), and thinning blood (anti–coagulant). The chemical name for aspirin is acetylsalicylic acid. It is an ester. Esters are usually prepared from an alcohol and a carboxylic acid using an acid, such as sulfuric, as a catalyst.

$$R-CO_2H+HO-R' \longrightarrow R-CO_2-R'+H_2O$$

In this experiment you will prepare aspirin from salicylic acid and acetic anhydride using sulfuric acid as a cataly

$$\underset{\textit{salicylic acid}}{HO_2C-C_6H_4-OH} + \underset{\textit{acetic anhydride}}{(CH_3CO)_2O} \xrightarrow{acid} \underset{\textit{aspirin}}{HO_2C-C_6H_4O_2CCH_3} + \underset{\textit{acetic acid}}{CH_3CO_2H}$$

In this reaction the –OH on the salicylic acid acts as the alcohol and the acetic anhydride acts like a carboxylic acid.

The aspirin will be prepared by reacting a weighed sample of salicylic acid with an excess of acetic anhydride. The aspirin formed will be contaminated with acetic acid and must be purified by recrystallization. This is accomplished by dissolving the acid in warm ethyl alcohol, adding cold water, and then causing it to recrystallize in an ice bath.

The aspirin is allowed to dry, weighed, and then the percent yield is calculated.

Equipment

From the stockroom:
- Beaker clamp
- filter flask
- Büchner funnel
- ice bath – in lab

From the common drawer:
- ring stand and ring
- wire gauze
- Bunsen burner

From your drawer:
- 125 mL Erlenmeyer flask
- large beaker

Procedure

This procedure must be carried out in the fume hood. Acetic anhydride is an irritant and sulfuric acid is very corrosive.

Record the mass of approximately 6.0 g of salicylic acid in a clean, dry 125 mL erlenmeyer flask. **In the fume hood** add 8 mL of acetic anhydride to the flask and then slowly add 10 drops of concentrated sulfuric acid. Clamp a larger beaker containing some water and a boiling chip or two to a ring stand. Heat the Erlenmeyer flask in this boiling water bath with occasional stirring for 15 minutes. If solid remains, heat it for an additional 15 minutes. Remove the flask and slowly add 20 drops of deionized water to convert any excess acetic anhydride to acetic acid. Add about 20 mL of ice–cold deionized water and cool in an ice bath until crystallization appears to be complete. (Hint: slow rubbing of the bottom of the flask under the solution with a stirring rod sometimes speeds up crystallization.) Assemble a Büchner funnel and filter flask and filter the crystals by vacuum filtration. (Your instructor will demonstrate how to do vacuum filtration.) If you wish to rinse the residue from the flask into the funnel, you may either use the filtrate (the solution in the filter flask) or a small amount of ice–cold deionized water. The filtrate may be disposed of in the hood sink.

The aspirin may be further purified by recrystallization. Dissolve the aspirin in about 20 mL of ethyl alcohol, and warm the mixture slightly by placing it in a beaker of water which has been heated. **DO NOT GET ETHYL ALCOHOL ANYWHERE NEAR A FLAME–HIGHLY FLAMMABLE!!** Stir to dissolve it completely, and then add 50 mL of warm (70°C) deionized water. Cool the mixture in an ice bath until recrystallization is complete. Vacuum filter the product and allow it to dry on the filter paper until the next lab period. The filtrate may be disposed of in the sink. When your aspirin is dry, put it in a weighed plastic vial and weigh it again. Record the mass. Calculate the percent yield.

Solid aspirin should be disposed in the **organic** solid waste