SYNTHESIS OF ASPIRIN

EXPERIMENTAL TECHNIQUES REQUIRED

Recrystallisation (T 2), filtration (T 3), melting point determination (T 4), yield calculation (T 14)

OTHER DOCUMENTS : Aspirin report (WORD)

EXPERIMENTAL PROCEDURE

- **Work in a fumehood**
- **Acetic anhydride is toxic, corrosive and a lachrymator (causes tears) and a skin irritant. Avoid contact with skin, eyes and clothing.**
- **USE ACETIC ANHYDRIDE IN A FUMEHOOD**
- **Ethanol is flammable.**

Place 1g of salicylic acid and 2mL of acetic anhydride in a large test tube (18mm x 150mm). Add 0.2g of **anhydrous** sodium acetate and stir. Place the test tube in a beaker of hot water (or a steam bath) until all the solid material has dissolved (use a glass rod to stir / crush the solid if needed, it may take 15 mins. or longer). When all the solid has dissolved, pour the solution into a 50mL Erlenmeyer flask containing 10mL of water, and rinse the test tube with a small amount of water. Swirl the flask to aid hydrolysis of excess acetic anhydride, and cool thoroughly in an ice-water bath (15-30 min) to induce crystallisation. If crystals haven't started to grow after 20 mins, then vigorous scratching of the inside of the flask with a glass rod *may* be necessary to induce crystallisation. Collect the crystallised solid in a Büchner funnel using vacuum filtration and then rinse with small portions of ice cold water. Collect your crude product and record your crude yield.

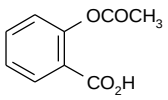
In three test tubes containing 5mL of water, separately dissolve a few crystals of phenol (1st tube), salicylic acid (2nd tube), and your crude product (3rd tube). Add about 10 drops of 1% ferric chloride solution to each tube and note the color of each tube (use these results to determine the purity of your crude product).

Use a hot water bath to heat some ethanol. Place your remaining crude aspirin in a large test tube and stir with a **minimum volume of hot ethanol** (this will vary depending on your crude yield but it is typically about 2-3 mL per gram of crude product). Add 10 mL water to the ethanol / aspirin solution. Heat the resulting turbid solution / oily liquid in the hot water bath until the solution clarifies then allow it to cool to room temperature (5-10 mins.). Now cool it in an ice-water bath to induce crystallisation, scratching with a glass rod if needed. After crystallisation is complete (at least 10 mins more), collect the product by vacuum filtration washing with small portions of ice cold water. Perform the ferric chloride test on your purified product (see above) to test for the presence of unreacted salicylic acid. Allow the crystals to dry completely before weighing them, calculating the percentage yield and determining the

melting point (3 determinations: one rapid, then two more slowly). Make sure you show your properly labeled product to your instructor. Don't throw your sample away as you will need to use a sample of it in the chromatography experiment.

Typical Sample Label:

MARY DOE



wt: 1.9 g
mp: 120-121° C
% yield: 50%

ASPIRIN

CLEAN UP

- The aqueous mother liquor should be transferred to the aqueous waste container.
- Melting point capillaries MUST be placed in the special bottles provided.

REPORT

Before writing any Chem 351 laboratory report, we strongly recommend that you review section 8 in the introductory section of the student laboratory manual that discusses how to write reports and/or from "writing reports" on the course website. Students often don't get the grades they would like because they make errors that are addressed in that section of the manual. These are avoidable errors.

The report is to be completed in the aspirin report templates provided (WORD).

Remember that more it not necessarily better. It is important to be accurate and concise rather than verbose and vague. Proper English should be used and it should be written in your own words.