Exp.08: preparation of Aspirin

Objectives:

1. To synthesize aspirin from salicylic acid and acetic anhydride.

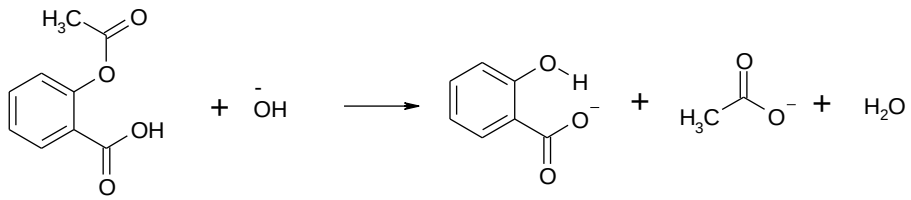
Introduction

Most drugs are chemical compounds, which are described as "organic compounds" because they are comprised primarily of the elements carbon, hydrogen and oxygen. The present experiment will be the synthesis of a familiar organic compound called **aspirin**. The common chemical name is **acetylsalicylic acid.**Aspirin is one of the most widely used, commercially available pharmaceutical drugs in the world. Its properties make it a powerful analgesic (pain reliever), antipyretic (fever reducer) and anti-inflammatory (reduces swelling) drug.

Preparation:

The preparation of Aspirin is an example of an organic synthesis called **esterification**. Esterification is the acid catalyzed reaction of a carboxyl (-COOH) group and an -OH group of an alcohol or phenol to form a carboxylate ester. In the synthesis of aspirin the -OH group is the **phenolic -OH** group attached to ring of the salicylic acid. The **acetyl group, $-COCH_3$** comes form acetic anhydride, and the reaction is catalyzed by **sulfuric acid**.

This reaction is quite simple and gives a good yield of the product. Aspirin, although it is soluble in hot alcohol, is not soluble in water. Consequently, the final product will be filtered from an aqueous solution, and washed with cold water, and thenair-dried.



Aspirin Salicylate ion acetate ion

The salicylate ion forms the therapeutic part of the aspirin. Aspirin passes through the acidic stomach contents ad does not ionize and form the salicylate ion until it reaches the alkaline conditions in the intestines. Here it is hydrolyzed to form the acetate and salicylate ion. The latter ion is absorbed through the intestinal wall. The equation depicting this reaction is given below.

The final step in the commercial synthesis of aspirin involves the reverse of this reaction. The salicylic acid is treated with acetic anhydride, which is a more reactive compound than the acetate ion. Catalytic amounts of sulfuric acid make the reaction proceed very rapidly. This is the synthesis step you will perform in this assignment.

Experimental Procedure:

Step	Procedure
1	Place 5.0 g of salicylic acid in a 100 mL Erlenmeyer flask.
2	Add, with continuous stirring, 10 mL of acetic anhydride (fume hood) followed by 1 mL of concentrated sulfuric acid.
3	Stir the mixture gently observing the rise in temperature to 70-80 °C while the salicylic acid dissolves.
4	After 10 minutes the solution cools by adding ice then Stir the mixture again for 10 minutes after that solid mass of aspirin will be formed.
5	Collect the crude aspirin using a Buchner funnel and wash with ice-cold water
6	Air-dry the products, weigh, and calculate the yield.

Laboratory Report

Name: ---------- Date: ----------

Experiment Subject: ----------

- Reaction:

Calculations:

Compound	Mol. Formula	Mol. Wight	Moles	Wight, mg	Density	Volume	Limiting reagent

Purification:

Recrystallization solvent: ----------

Purity check by melting range: ----------

TLC: ----------

Physical Data (Product):

State: ---------- Melting Point: ----------

Color: ---------- Solubility: ----------

Yield:

Characterization:
