

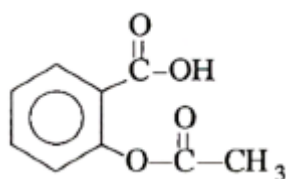
## Experiment 01: SYNTHESIS OF ASPIRIN

### I. Objectives:

- Synthesize acetylsalicylic acid (aspirin) by carrying out a simple organic reaction,
- Separate the product from the reaction mixture by vacuum filtration,
- Purify the product by recrystallization,
- Determine the success of the synthesis by calculating the percentage yield of the product.

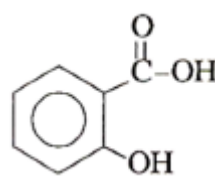
### I. Introduction:

Aspirin is one of the most widely used medications in the world. It is employed as an analgesic (pain relief), an anti-pyretic (fever control) and an anti-inflammatory. More recently, studies have indicated that daily intake of small doses of aspirin can lower the risk of heart attack and stroke in high-risk patients.



Acetylsalicylic acid

(Aspirin)

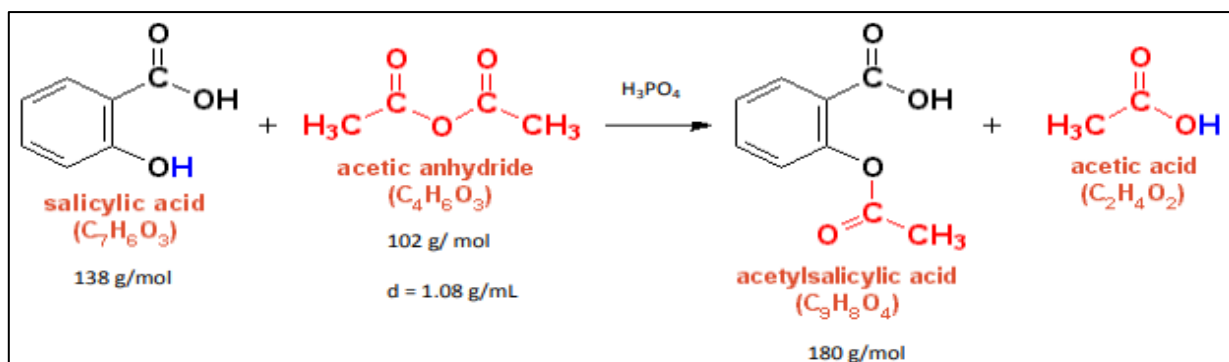


Salicylic acid

The history of aspirin and its precursor dates back to ancient times. Documents attributed to Hippocrates, the father of modern medicine, from the 4th century B.C. refer to the alleviation of pain by chewing on the bark of a willow tree or ingesting a powder made from the bark and leaves of the willow. This remedy was passed on from generation to generation.

### II. Principle:

To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride. A small amount of a strong acid is used as a catalyst which speeds up the reaction. The excess acetic acid will be quenched with the addition of water. The aspirin product is not very soluble in water so the aspirin product will precipitate when water is added. The synthesis reaction of aspirin is shown below:



### III. Equipment and Reagents:

- Salicylic acid
- Acetic anhydride
- Phosphoric acid
- Beaker of tap water
- Reflux heating apparatus
- Distilled water
- Burner
- Stand with iron ring
- Ice bath
- filter paper
- Thermometer 0 to 100 °C.
- Heating magnetic stirrer

### IV. Experimental Procedure :

#### a)- Aspirin Synthesis :

Step	Procedure
1	Measure out 5 g of salicylic acid (SA) and place the SA in to 250-mL Round-bottom flasks.
2	Under the extractor hood, gently and carefully and by using graduated cylinder, add with continuous stirring, 7 mL of acetic anhydride (fume hood) followed by 4 to 5 drops of concentrated phosphoric acid.
3	Place Round-bottom flasks in the water bath and immediately top it with the water condenser then circulate water.
4	Maintain the water bath between 50°C and 60°C with magnetic stirring for 20 minutes approximately. After this, <b>stop</b> the heating and <b>don't stop</b> the circulation of cold water in the refrigerant.
5	After 5 minute, remove the flask from the water bath and add 20 mL of distilled water, in order to destroy excess ethanolic anhydride by hydrolysis. Let the flask cool to room temperature. As the solution cools, crystals of aspirin will appear. Cool the solution further by placing the reaction flask in an ice bath. Chill 5-10 mL of distilled water in a separate container.

6	Set up a Büchner funnel on a vacuum flask connected to a water aspirator. Place the filter paper in the funnel and moisten with distilled water from a squirt bottle. Turn on the aspirator and transfer the aspirin slurry into the funnel. Wash the crystals with 5 mL of the cold water.
7	Transfer the filter paper and aspirin to a pre-weighed watch glass and allow to air dry in your locker until the next lab period.
8	It is safe to discard of the filtrate down the sink with water.

### b)- Recrystallization of the Product:

Step	Procedure
1	Transfer the impure aspirin from the Büchner funnel to a 150-mL beaker. Add 5 mL of 95% ethanol and warm the flask on a hot plate until all of the solid dissolves.
2	Immediately remove the flask from the heat and slowly add 20 mL of cold water.
3	Crystals should form. Chill this solution in an ice-water bath, and collect the crystals using vacuum filtration.

### c) Yield of Purified Aspirin:

Once your aspirin appears to be dry, transfer it to the watchglass. Determine and record the mass of the watch glass and dried aspirin.

$$\% \text{ yield} = \frac{\text{actual mass obtained}}{\text{calculated mass}} \times 100\%$$

### Questions:

1. Give a general description of esterification reaction.
2. Give the brute formula of the reactants and the products.
3. What's the purpose of using a dry glassware?
4. What's the purpose of adding phosphoric acid?
5. Why could the final product have washed with cold water?
6. What is the principle of recrystallization?
7. What is the effective yield of this synthesis?
8. What do you conclude from this experiment?