

# Chapter: 15

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## **AIM: TO PERFORM THE ASPIRIN SYNTHESIS FROM SALICYLIC ACID**

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## **Requirements**

### **Glassware**

- Water bath
- Conical flask
- Test tubes
- Glass rod
- Buckner funnel

### **Chemicals**

- Salicylic acid
- Pyridine
- Acetyl chloride

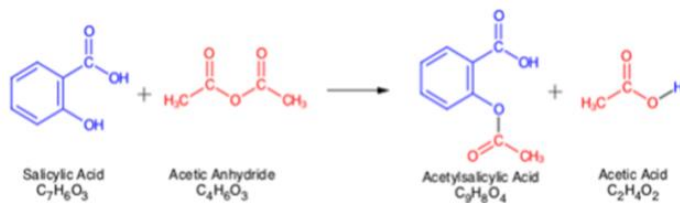
## **Theory**

The process of making aspirin falls within the category of an esterification reaction. The acid derivative acetic anhydride reacts chemically with salicylic acid to convert the hydroxyl group into an ester group. Esterification is a chemical reaction that produces at least one ester product by reacting an organic acid (RCOOH) with an alcohol (ROH) to create an ester (RCOOR) and water.

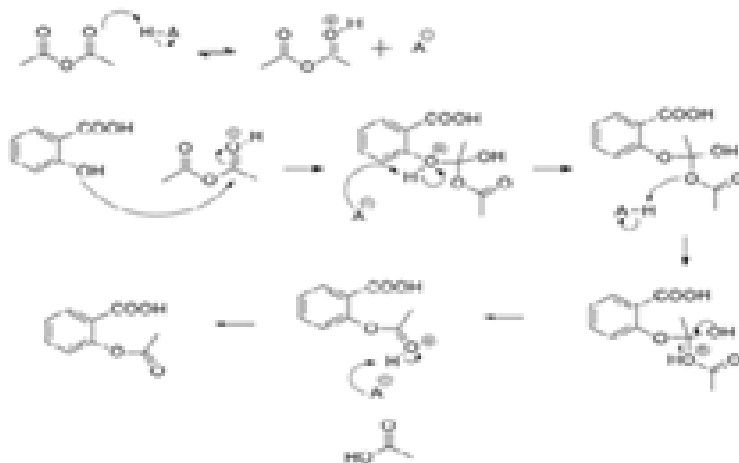
Aspirin is an effective analgesic (pain reliever), antipyretic (fever reducer), and anti-inflammatory (swelling reducer). The analgesic salicylic acid, which is a component of certain plants, was first delivered as sodium salicylate. Chemists searched for a modification that would keep the benefits of salicylic acid while reducing the side effects because it irritates the stomach lining. This

need was met by conversion to the ester, and aspirin (acetylsalicylic acid) proved to be just as effective as sodium salicylate.

## Reaction



## Mechanism



## Physical Properties

### 1. Salicylic acid-

- It is white crystalline powder
- It has no odour
- It tastes bitter first, then sweet

## 2. Acetic anhydride

- Acetic anhydride is a clear, colourless liquid that is soluble in benzene and water and smells strongly of vinegar.

## 3. Aspirin

- White, crystalline, mildly acidic molecule that is an acetyl derivative of salicylic acid.

## Chemical Properties

### 1. Salicylic acid

- Its most important reaction associated with use of salicylic acid is production of aspirin
- It can undergo reaction with organic alcohol groups to produce esters.
- The acidity of salicylic acid is stronger than benzoic acid.
- It becomes darker in color in the sun.

### 2. Acetic anhydride

- Acetic anhydride hydrolyses to give carboxylic acids
- Acid Anhydrides react with alcohols to form esters

### 3. Aspirin

- Aspirin decomposes rapidly in solutions of ammonium acetate or the acetates, carbonates, citrates, or hydroxides of the alkali metals.
- It is stable in dry air, but gradually hydrolyses in contact with moisture to acetic and salicylic acids.

- In solution with alkalis, the hydrolysis proceeds rapidly and the clear solutions formed may consist entirely of acetate and salicylate.

### **Procedure**

1. Take a known amount (say 2.0g) of salicylic acid in a conical flask further add a calculated amount of (say 5 mL) acetic anhydride with constant stirring while you are adding the concentrated sulfuric acid drop by drop (say 5 drops, with a glass- dropper) in a fume hood.
2. Stirring the mixture gently till the salicylic acid dissolves. Maintain the latter between 50° and 60° throughout the flask, occasionally submerging it in cold water if necessary. This is because the heat of the reaction causes the mixture's temperature to rise quickly.
3. After heating the mixture in a steaming water bath for five minutes, cool the solution to 35-45°C by adding ice cold water slowly in the reaction mixture to hydrolyse excess acetic anhydride and a solid mass of aspirin forms. The mixture should then be thoroughly washed with water and drained.
4. Collect the crude aspirin using a funnel or filtration assembly under vacuum and wash with ice-cold water.
5. In the hot air oven, let the products air dry
6. Re-crystallize the end result made from volumes of acetic acid and water.
7. After the product has dried completely, record melting point using capillary method
8. Calculate the percentage yield.

## **Applications**

1. Aspirin is used to ease mild to moderate pain from disorders like muscle pains, toothaches, the common cold, and headaches. It also lowers temperature.
2. It can also be used to treat illnesses like arthritis by lowering pain and swelling.
3. In individuals with cardiovascular disease or who have already experienced a heart attack or stroke, aspirin has been found to be beneficial when administered daily to reduce the risk of heart attack, clot-related strokes, and other blood flow disorders.
4. Aspirin can treat a variety of health conditions, including menstrual cramps, by reducing mild to moderate discomfort, swelling, or both.
5. It lowers men's vulnerability to transient ischemic attacks.