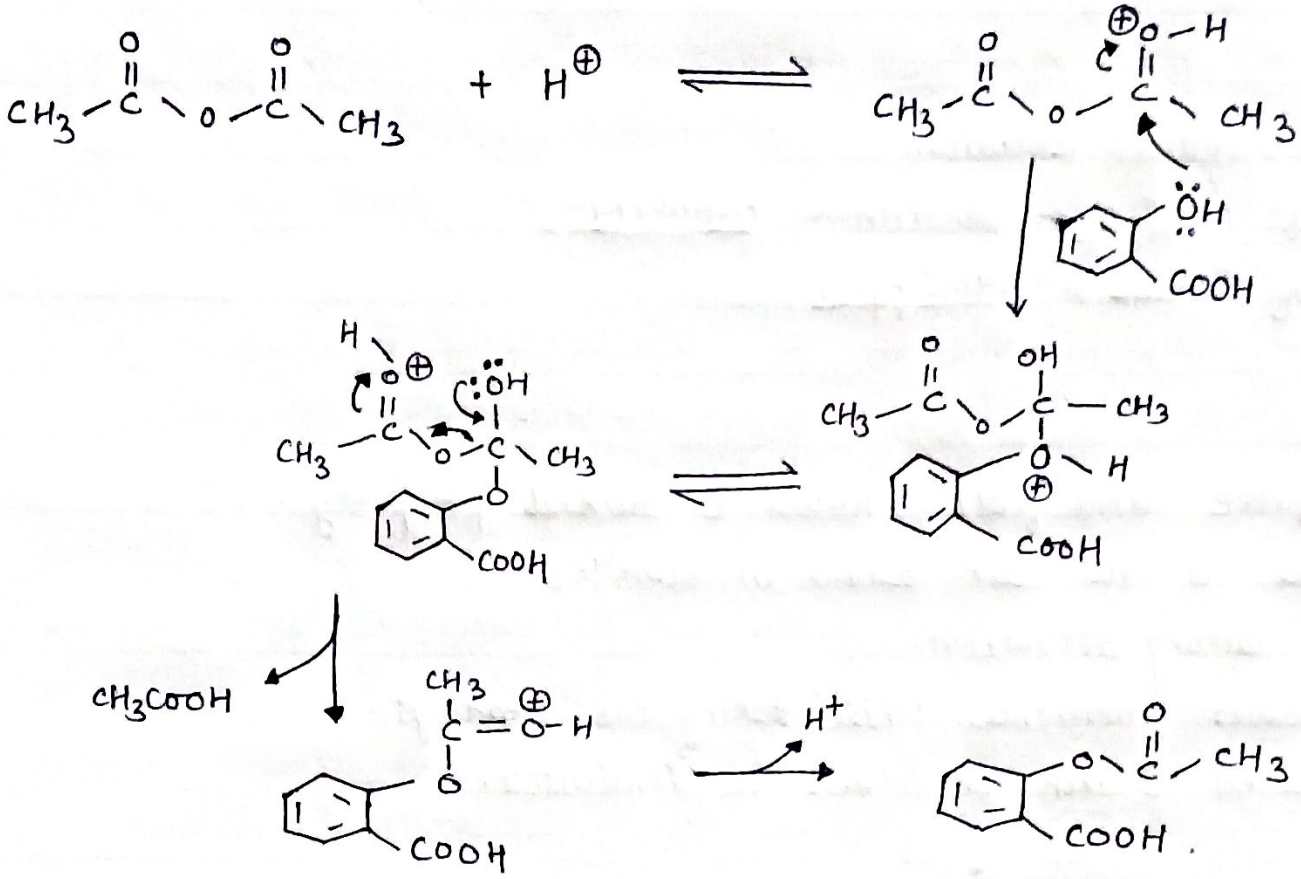
Mechanism of the Reaction :



Aspirin

Date 26.12.2021

Expt. No. 3 Page No. 7

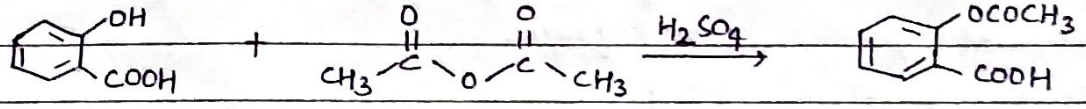
Aim: To Synthesize Aspirin

Apparatus Required: Conical flask, measuring cylinder, beaker, glass rod, filter paper and other general glasswares.

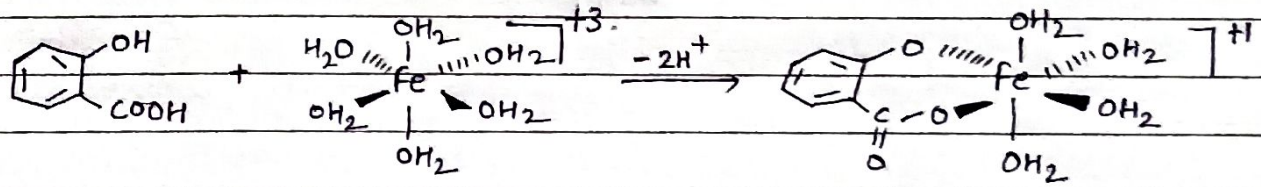
Chemicals Required: Salicylic Acid, acetic anhydride, conc. Sulphuric acid, ethanol, methanol and $FeCl_3$.

Principle: Preparation of the derivative of a functional group compound. Here, phenolic group in salicylic acid is esterified with acetic anhydride in the presence of an acid catalyst, ie, H_2SO_4 to obtain 2-acetoxy benzoic acid (Aspirin). Aspirin is the most frequently sold painkiller in the world. It is used to treat patients with cardiovascular disease. It works as an analgesic and antipyretic.

Reactions:



Salicylic Acid Acetic Anhydride Aspirin.



Purple colour.

Procedure:

(A) Synthesis:

(1) Take X(=2g grams of salicylic acid (Md wt 138.12 g/mol) and transfer

Teacher's Signature

Observations and Calculations

- To calculate the amount of Acetic Anhydride

Salicylic acid = 1.8 g.

Moles of Salicylic Acid = 1.8 / 138.12 = 0.01303 moles.

So, moles of acetic anhydride = 2.7 × 0.01303 = 0.03518 moles.

Grams of Acetic Anhydride = 0.03518 moles × 102.08 g/mole

= 3.59 grams.

Acetic Anhydride is a liquid. So, we can calculate volume.

Volume = 3.59 / 1.08 = 3.324 ml

- To calculate percentage yield.

From the chemical equation, we know that one mole salicylic acid will give one mole acetyl salicylic acid.

So, we should get 0.01303 moles of acetyl salicylic acid.

molecular weight of acetyl salicylic acid = 180.158 g/mol

∴ 0.01303 moles = 2.347 grams of Acetyl Salicylic Acid.

Theoritical yield = 2.347 grams

Actual yield = 1.5 grams.

So, Percentage yield = $\frac{1.5}{2.347} \times 100$ = 63.91 %.

it to a dry 150 ml conical flask.

(2) Add 2·7 equivalents of acetic anhydride (M.wt 102·08 g/mol, Density 1·08 g/ml) using a measuring cylinder. Now add 5-6 drops of conc. H_2SO_4 and stir until all salicylic acid is dissolved.

(3) Leave all the reaction mixture undisturbed for 15-20 minutes.

(4) Add 50 ml of water to the flask and swirl for two minutes and filter using a Buchner funnel.

(5) Collect the solid from the filter paper.

(B) Recrystallisation.

(1) Dissolve the crude product in 7 ml of ethanol in a beaker and add 15 ml of distilled water. Heat on water bath until we get a clear solution.

(2) Allow the solution to cool in an ice bath without disturbing. Pure acetyl salicylic acid crystallises.

(3) Filter the pure product and dry it by placing in between the sheets of filter paper.

(4) The crystallised pure material was weighed and report the percentage yield.

(C) Validation of Purity

A few crystals of the compound contained in test tube were dissolved in 0·5 ml of methanol. A few drops of $FeCl_3$ were added. No purple color was obtained, which suggests the absence of salicylic acid as an impurity. The same test was carried out for salicylic acid. In this case, intense purple colouration was observed on adding $FeCl_3$ solution.

As a characteristic physical property, melting point of pure compound was determined.

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Results:

(1) Acetyl salicylic acid was synthesized from salicylic acid.
(2) Actual yield : 1.50 g
(3) Percentage yield : 63.91%
(4) Melting point of acetyl salicylic acid : 134-136 °C
(5) Ferric chloride test confirms the absence of phenolic group in acetyl salicylic acid.

Precautions:

(1) Dry conical flask should be used for salicylic acid.
(2) measuring cylinder should be used for acetic anhydride.
(3) Conc. H_2SO_4 should be used with care.
(4) Care should be taken to isolate crystals of aspirin as far as possible.