Exp. 3

Aim: To synthesize Aspirin

Apparatus Required: Measuring cylinders, beaker, conical flask, droppers, glass rod, filter paper, distilled water and other general glassware, Buchner funnel.

Chemicals Required: Salicylic Acid, acetic anhydride, Conc. H_2SO_4, ethanol, methanol, $FeCl_3$.

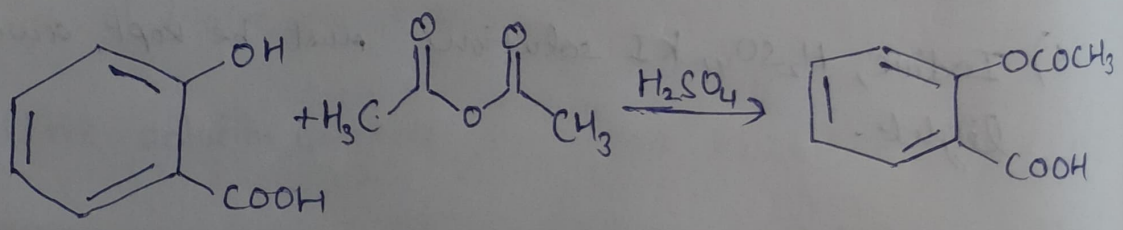
Principle: Aspirin, also called Acetyl Salicyclic Acid is most frequently sold pain reliever in the world, and has been termed as 'Wonder Drug' of the century.

Its preparation is done by salicyclic acid and acetic anhydride in the presence of acid catalyst i.e. H_2SO_4. In the reaction, phenolic hydroxyl group of salicyclic acid is esterified with acetic anhydride to obtain Aspirin.

After it has been prepared, impurities may be there so filtration, then recrystallization and finally validation of purity by $FeCl_3$ test is done.

Reactions:

1)



Procedure:

(A) Synthesis:

1) Take 1.8 g of salicylic acid (Mol. wt. 138.12 g/mol) into 150 ml conical flask.

2) Add 2.7 eq. of Acetic anhydride (Mol. wt. 102.08 g/mol, Density 1.08 g/ml) using a measuring cylinder to the salicylic acid.

3) Now add 5-6 drops of conc. H_2SO_4 and stir until all salicylic acid is dissolved.

4) Allow the reaction mixture to react for 15-20 minutes, leave it undisturbed.

5) After 15 mins, solid Aspirin is formed. Add 50 ml of distilled water & swirl for 2 mins. and then filter using a Buchner funnel.

6) Solid Aspirin can be collected from funnel, after filtration.

(B) Recrystallization:

1) Dissolve the crude product in 7ml of ethanol and 15 ml of distilled water in a beaker.

2) Put it on water bath to heat till you get a clear solution.

3) Take it off from water bath and keep it in ice bath for recrystallization. (for 10-15 mins)

4) Filter again using buchner funnel, and we get the pure white solid Aspirin.

5) Calculate the percentage yield.

6) Determine the melting point of acetylsalicylic acid.

(C) Validation of Purity : ($FeCl_3$ Test)

This test is important to check presence / absence of phenol grp.

since we have done acylation of salicyclic acid phenol has been converted to corresponding acetate.

(i) For salicyclic acid :

it has phenol part so it will show +ve test.

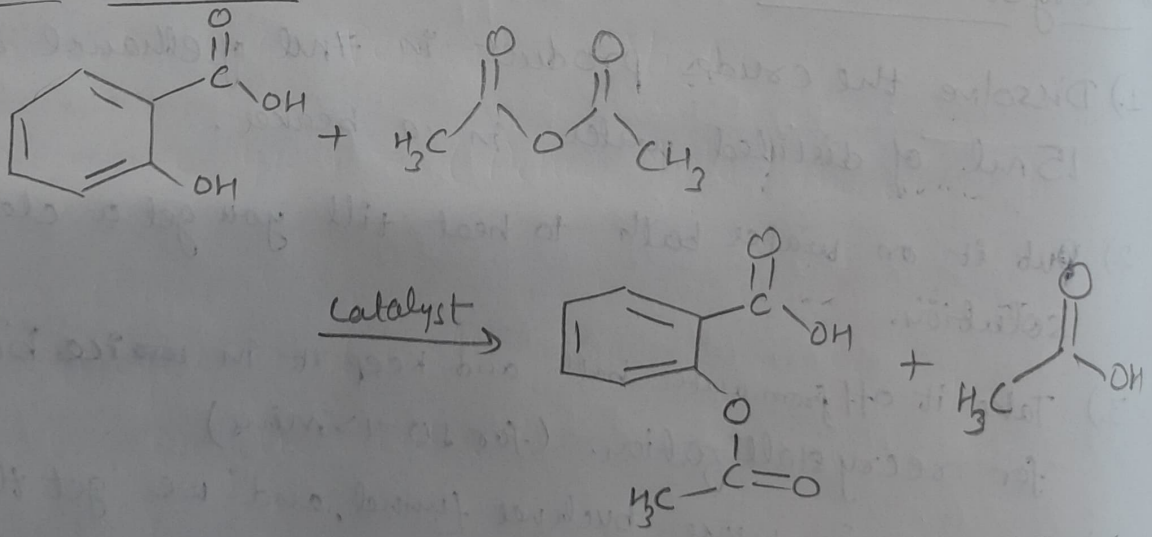
1.) Take a pinch of salicyclic acid
2) dissolve in methanol
3) Add 1-2 drops of $FeCl_3$; it will give a purple-violet colouration, which shows presence of phenol grp.

(ii) For Aspirin

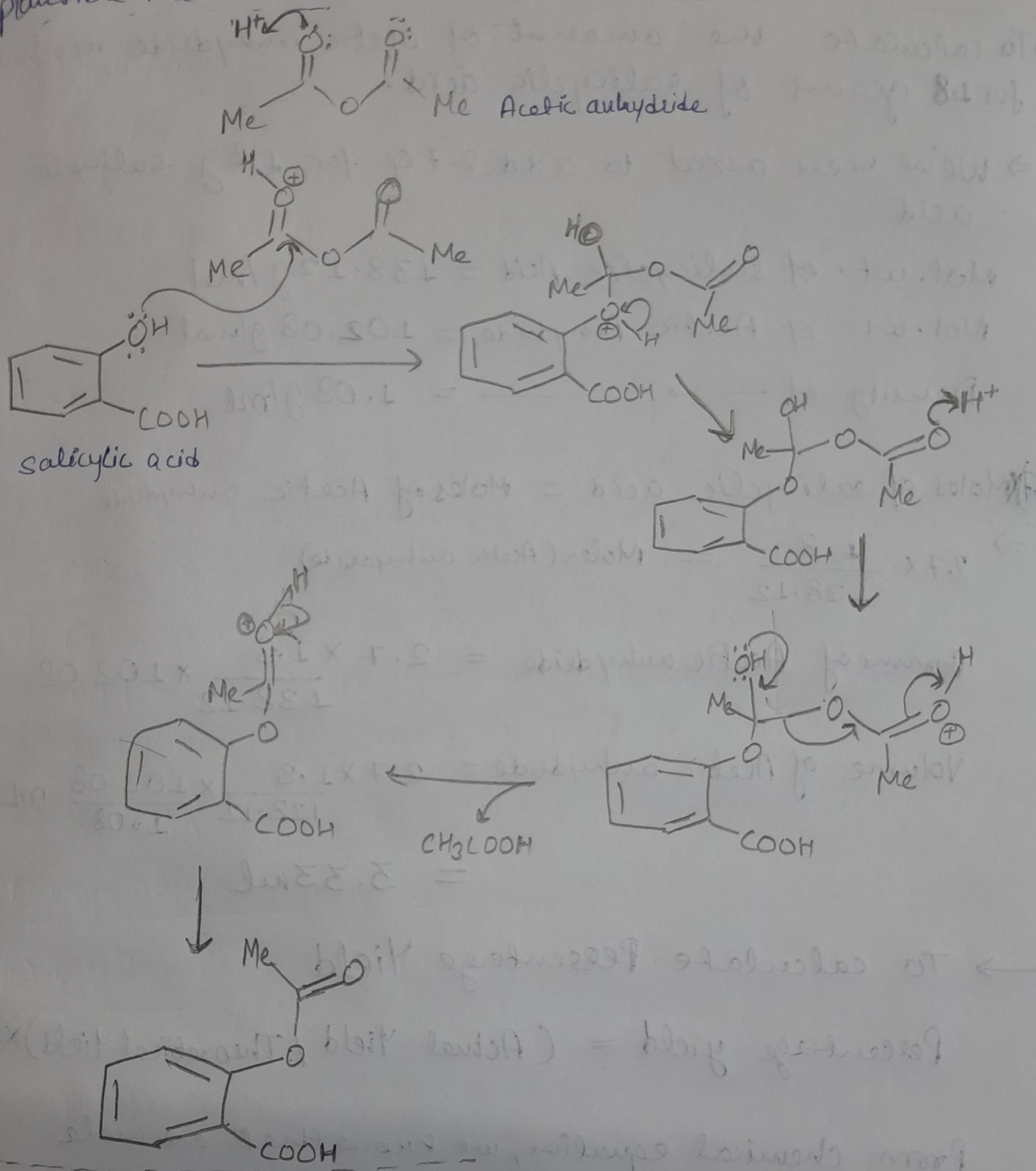
it should give -ve test.

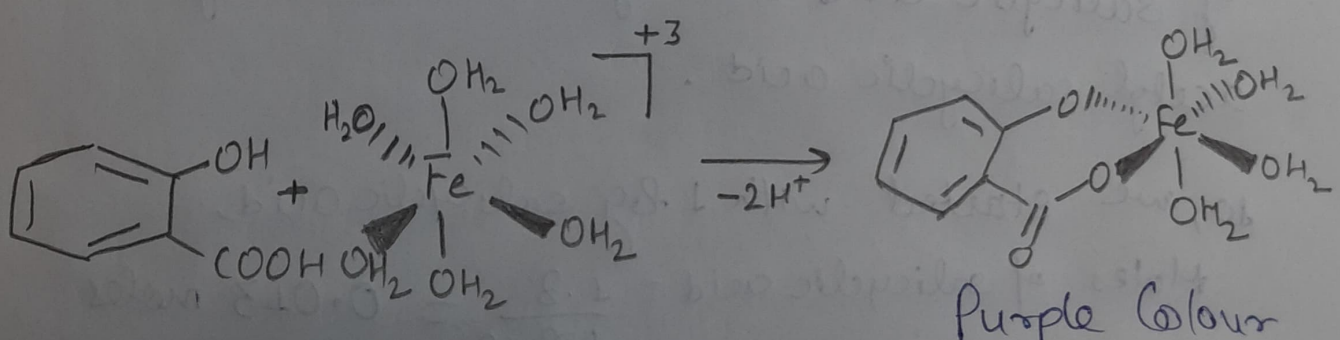
1.) Take a small amount of aspirin prepared
2) dissolve in methanol
3) Add 1-2 drops of $FeCl_3$, colour will not change, It will remain yellow as the colour of $FeCl_3$

Chemical Structures :



plausible mechanism:





Purple Colour

Depending on the conc. of the reagent, mono to trichelate rings may be formed

Observation & Calculations :

→ To calculate the amount of acetic anhydride needed for 1.8 grams of salicyclic acid.

⇒ We've been asked to add 2.7 eq for 1.8 g salicyclic acid

Mol. wt. of salicyclic Acid = 138.12 g/mol

Mol. wt. of Acetic Anhydride = 102.08 g/mol

Density of ——— = 1.08 g/ml

Moles of salicyclic acid = Moles of Acetic anhydride

$$\Rightarrow 2.7 \times \frac{1.8}{138.12} = \text{Moles (Acetic anhydride)}$$

$$\text{Grams of Acetic anhydride} = 2.7 \times \frac{1.8}{138.12} \times 102.08$$

$$\text{Volume of Acetic anhydride} = 2.7 \times \frac{1.8}{138.12} \times \frac{102.08}{1.08} \text{ mL}$$

$$= 3.33 \text{ ml}$$

→ To calculate Percentage Yield

Percentage yield = (Actual Yield / Theoretical Yield) X100

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

so, we started with 1.8g salicyclic acid,

$$\text{Moles of salicyclic acid} = \frac{1.8}{138.12} = 0.013 \text{ moles}$$

Hence we should get 0.013 moles of acetyl salicyclic acid

Mol. wt of acetyl salicylic acid = 180.158 g/mol

0.013 moles = 2.34g of acetyl salicylic acid

↳ Theoretical yield

Actual yield = grams of product obtained by performing reaction

= 1.5g

Percentage Yield = $\frac{1.5}{2.34}$ = 64.10 %

Result:

1.) Aspirin was synthesized from salicylic acid

2) Actual Yield = 1.5g

3) Percentage Yield = 64.10%

4) Melting pt. of Aspirin = 134 – 136°C

5) $FeCl_3$ test confirms the absence of phenolic group in Aspirin prepared.

Precautions:

1. Dry Conical flask should be used
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.