

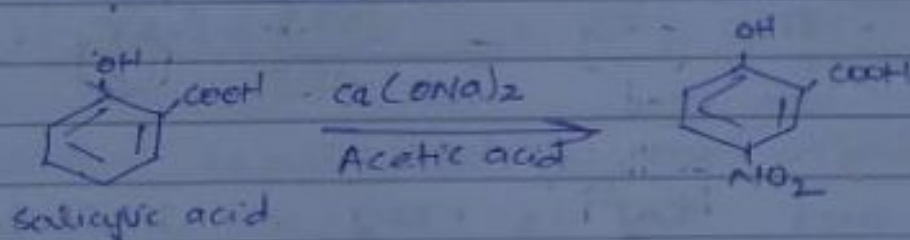
Experiment No. 1

Procedure for the synthesis of nitrophenol by aromatic electrophilic substitution reaction (nitration).

Theory:-

There are many methods for the synthesis of organic reagents. Some are conventional methods that are hazardous to human health. Another method is green chemistry method that is environment friendly and economic as well.

Chemical Reaction:-



Apparatus:-

Beakers, test tubes, water bath, stirrer volumetric cylinder, volumetric flask

Chemicals Required:-

Calcium nitrate tetrahydrate = 1.5g

Acetic acid = 5ml

Salicylic acid = 1g

Procedure:-

- ① The apparatus was washed and dried carefully.

- ② 1.5g of calcium nitrate was dissolved in warm acetic acid (5ml).
- ③ 1g of salicylic acid was also added to it. Mixture was heated in a boiling water bath (maintained at 80°C) for one minute.
- ④ when salicylic acid dissolved completely, solution becomes dark red.
- ⑤ Solution was then immediately poured in a 10ml of ice cold water.
- ⑥ The resultant turbid dark red solution placed in refrigerator.
- ⑦ After four hours, the yellow crystals were obtained. The crystals were separated and washed with minimum amount of ice cold water and then dried.

precautions:-

- ① yield of reaction mainly depends on the amount of reaction mixture and solubility.
- ② Minimum amount of water should be used for washing.

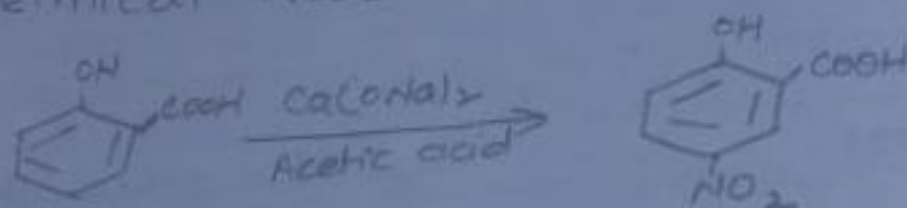
Result:-

Nitration of phenol takes place with good yield.

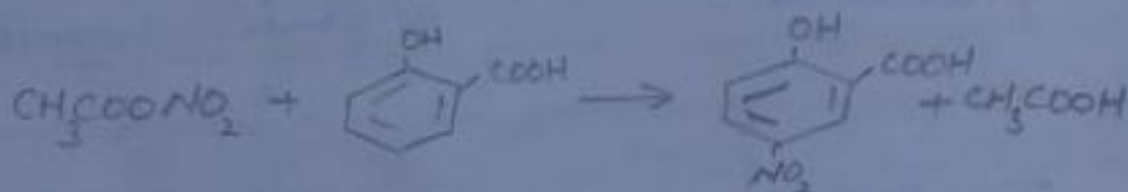
Experiment No. 1

Nitration of Salicylic acid by green procedure.

Chemical Reaction:-



Mechanism:-



Observations and Calculations

→ Limiting reactant (theoretical yield):-

Salicylic acid : product

13.8g : 18.3g

1g : $\frac{18.3}{13.8}$

= $\boxed{1.32g}$

Experiment No. 2

Procedure for the synthesis of salicylic acid

Calcium nitrate

Product

164g

:

183

1g

:

$\frac{183}{164}$

1.5g

:

$\frac{183}{164} \times 1.5$

=

1.67g

Acetic acid

:

product

120

:

183

1

:

$\frac{183}{120}$

5.25

:

$\frac{183}{120} \times 5.25$

=

8.00g

As salicylic acid produce least amount of product that is 1.32g, so it is limiting reagent.

Actual yield:-

wt. of filter paper = 1.20g

wt of filter paper + ppt = 1.89g

wt of precipitates = $1.89 - 1.20 = 0.69g$

Actual yield = 0.69g

% yield :-

$$\% \text{ yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100$$

$$= \frac{0.60}{1.32} \times 100 = 45.4\%$$

Result:-

% age yield of product was 45.4%.

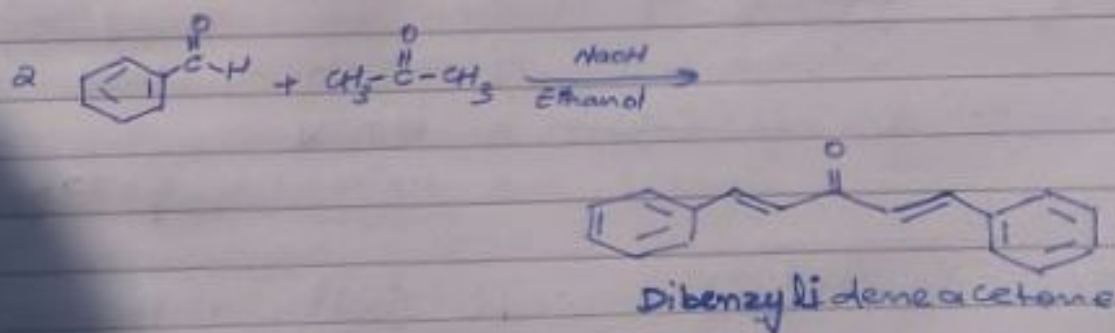
Experiment NO. 2

Synthesis of dibenzylideneacetone through base catalysed aldol condensation reaction.

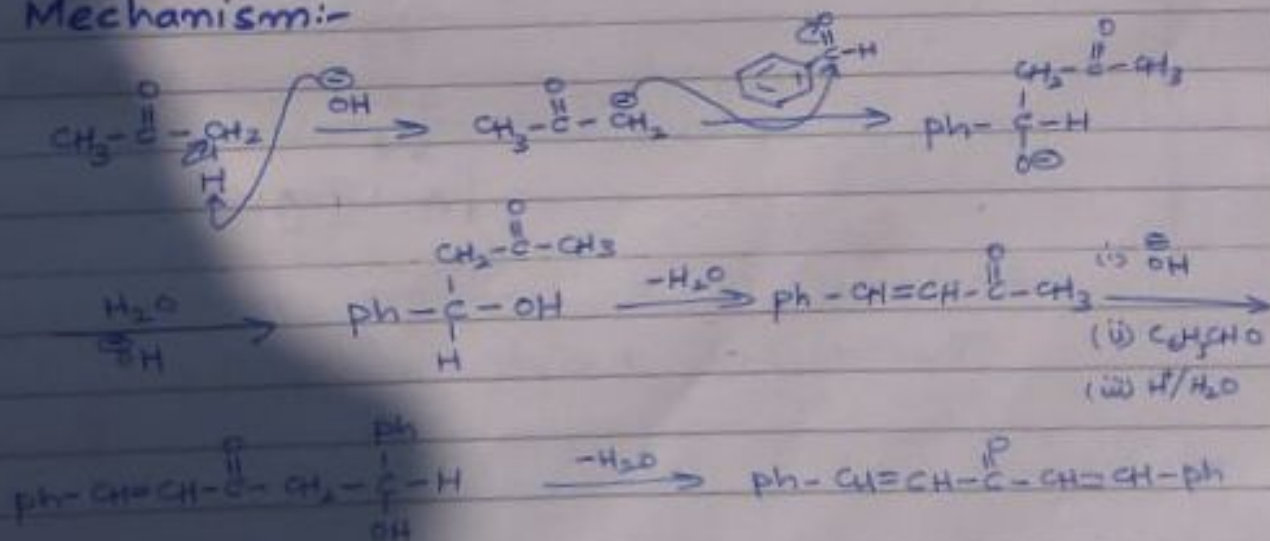
Chemicals required:-

- Acetone 1 ml (0.89g)
- Benzaldehyde 3.8 ml (3.9g)
- NaOH 30 ml of 10% soln

Chemical Equation:-



Mechanism:-



procedure:-

- ① All the apparatus was washed and cleaned properly.
- ② In a conical flask fitted with a cork, benzaldehyde (1ml), acetone (3.8ml) and methylated spirit or alcohol (15ml) was shaken together for 2 minutes.
- ③ 10% NaOH solution was added and shaken for about 10 minutes.
- ④ Reaction mixture was cooled in ice and pale yellow solid was filtered.
- ⑤ precipitates were washed and dried with it. It is recrystallized from ethanol having m.p. $120-122^{\circ}$.

Results:-

Synthesis of dibenzalpropenone was completed with 64% yield.

Experiment No. 2

Synthesis of dibenzal propanone.

Chemicals Required:-

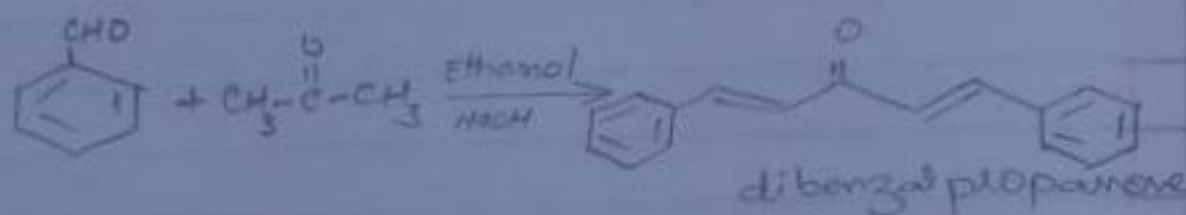
Benzaldehyde = 3.5 ml

Acetone = 1 ml

Methanol = 15 ml

10% NaOH = 30 ml

Chemical Reaction:-



Observations and Calculations

Molecular wt. of product = 234 g

Molecular wt. of benzaldehyde = 106 g

Volume used of benzaldehyde = 3.8 ml

$$d = \frac{m}{V} \Rightarrow d \times V = m$$

$$m = d \times V$$

$$= 1.004 \times 3.8 = 3.86 \text{ g}$$

Actual yield = 1.89g

$$\% \text{ age yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100$$

$$= \frac{1.89}{3.18} \times 100$$

$$= 59.4\%$$

Result:-

The %age yield of dibenzal propenone was 59.4%.

Experiment no. 3

03-01-19

preparation of Benzil from Benzoin

Apparatus:-

water bath, Round bottom flask, Buchner funnel, cotton wool, Burner, Tripod stand etc

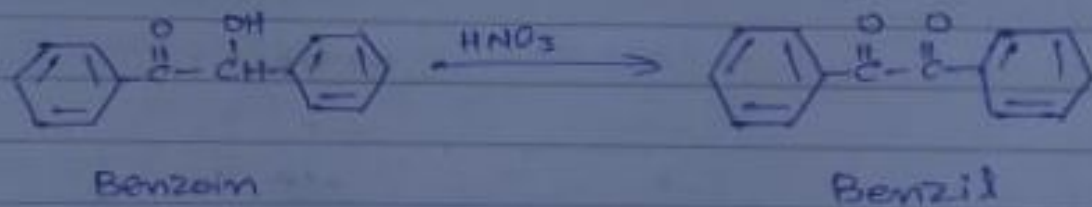
chemicals Required:-

Benzoin = 4.0g

conc. HNO₃ = 1.4 ml

Ethanol = 10mm

Chemical Equation:-

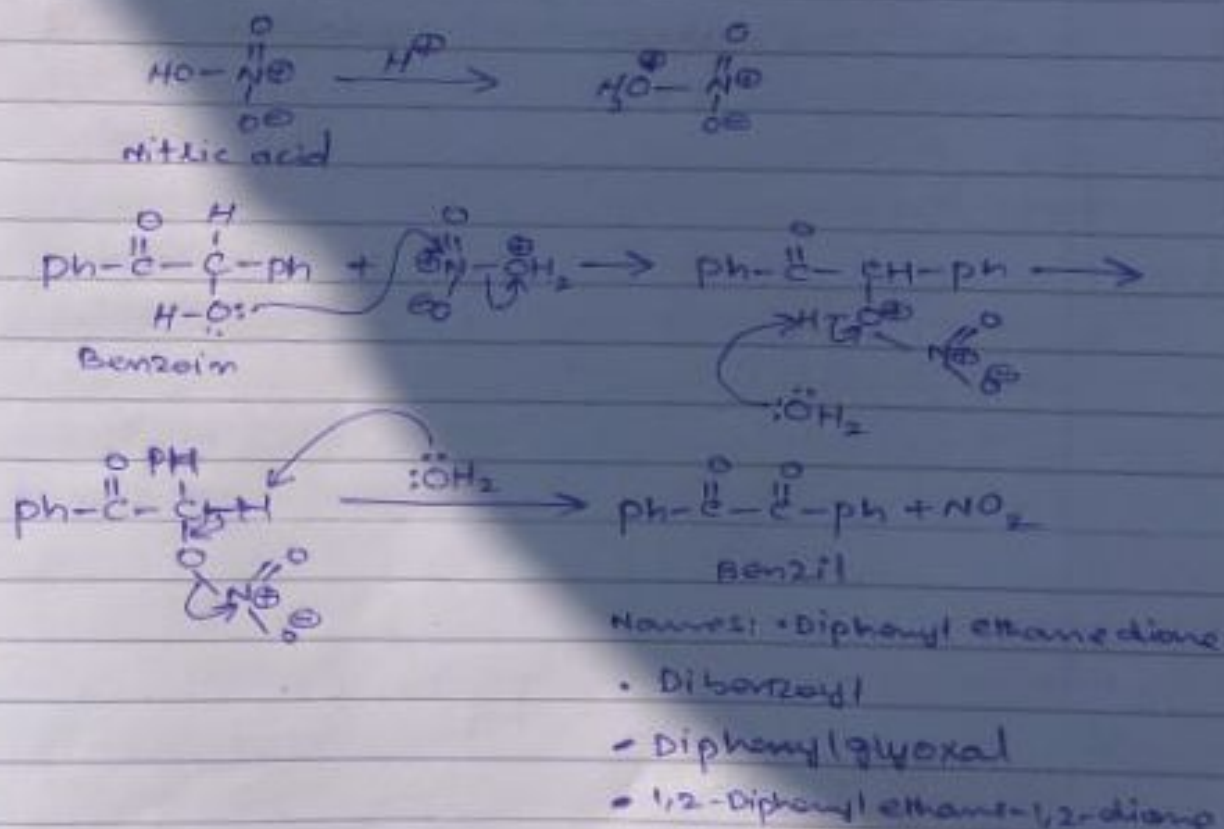


procedure:-

- ① The mixture of 4g of benzoin and 14ml of concentrated HNO_3 was heated on a steam bath (water bath) for 10-12 minutes. The reaction was carried out in fume hood because vapours produced are very toxic.
- ② Once the reaction was completed, 75ml H_2O was added and was cooled to room temperature and stirred for a minute or two to coagulate the precipitated product.

- ③ The product was collected by filtering the mixture and was pressed to remove moisture.
- ④ The product was recrystallized from 10ml ethanol, once the product was dissolved, water was added dropwise to reach the cloud point. The product was allowed to recrystallize.
- ⑤ The product was filtered to obtain pure product.
- ⑥ The mppt was determined and theoretical yield was calculated.

Mechanism:-



Result:-

The benzil was prepared by the oxidation of benzoin and % yield was 82%.

03-10-19

Experiment NO. 3

Preparation of Benzil from benzoin.

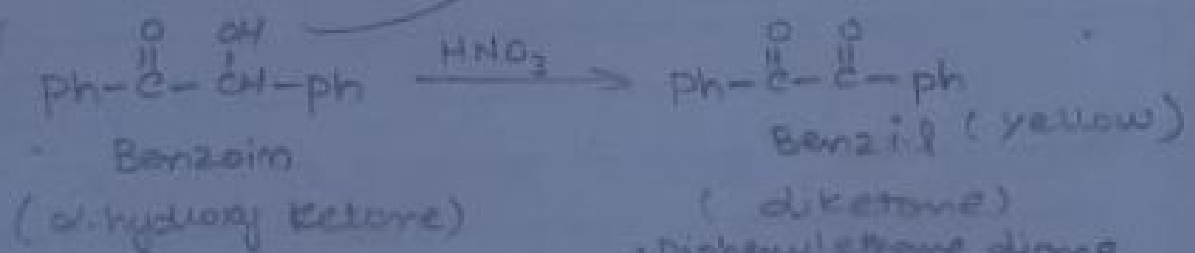
Chemicals Required:-

Benzoin = 4.0g

conc. HNO_3 = 14 ml

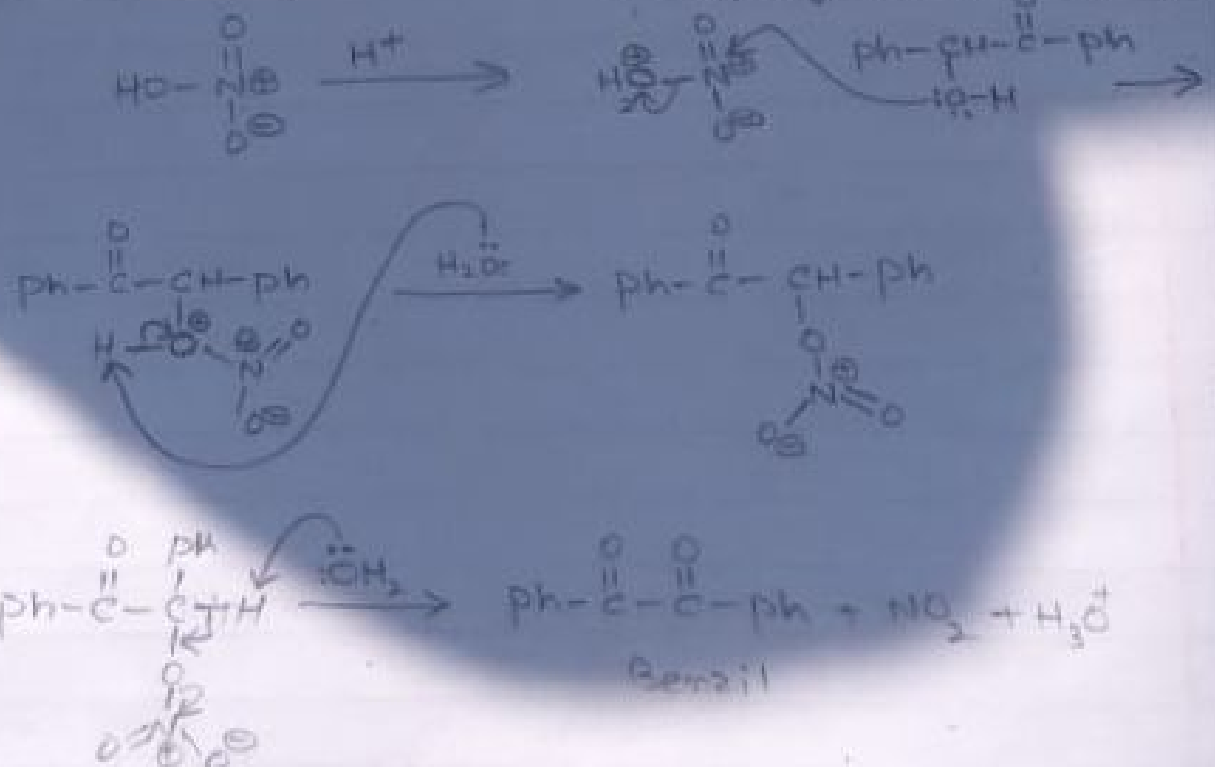
Ethanol = 10 ml

Chemical Equation :-



- 2-Hydroxy-1,2-diphenylethane
- 2-Hydroxy-2-phenylacetophenone
- Benzoylphenyl carbinol

Mechanism:-



Theoretical yield:-

$$\text{Molar mass of benzoin} = 212.24 \text{ g/mol}$$

$$\text{Molar mass of benzil} = 210.2 \text{ g/mol}$$

$$\text{Molar mass of } \text{HNO}_3 = 63.01 \text{ g/mol}$$

$$\text{Density of } \text{HNO}_3 = 1.51 \text{ g/cm}^3$$

$$d = \frac{m}{V}$$

$$m = d \times V$$

$$= 1.51 \times 14 = 21.14 \text{ g}$$

Limiting reactant:-

Benzoin : Benzil

$$212 : 210$$

$$1 : \frac{210}{212}$$

$$4 : \frac{210}{212} \times 4 = \boxed{3.96 \text{ g}}$$

HNO_3 : Benzil

$$63 : 210$$

$$1 : \frac{210}{63}$$

$$21.14 : \frac{210}{63} \times 21.14 = \boxed{71.33 \text{ g}}$$

⇒ Since benzoin produces least amount of product, so it is limiting reactant

Theoretical yield = 3.9 g

Actual yield = 3.2 g

$$\% \text{ yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100$$

$$= \frac{3.2}{3.9} \times 100 = \boxed{82\%}$$

Result:-

The benzil was prepared by the oxidation of benzoin and % yield was 82%.

Experiment No. 4

Synthesis of dihydropyrimidinone through green procedure:-

water bath, Round bottom flask, glass rod, filter paper, funnel, Burner, Tripod stand etc.

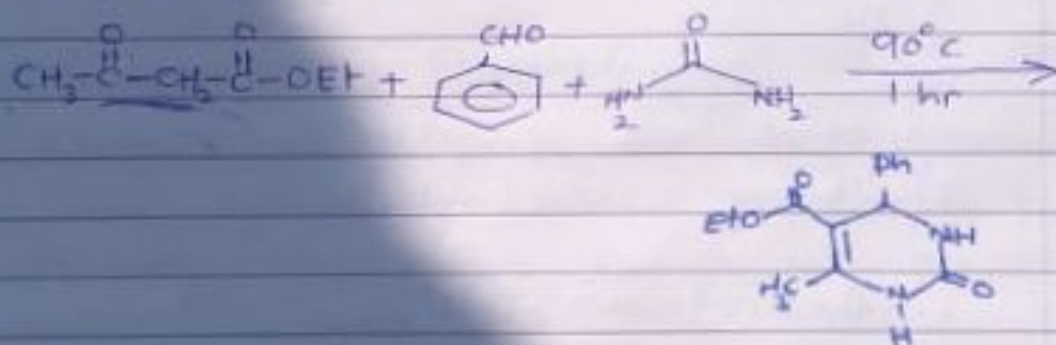
Chemicals Required

Ethyl acetate = 1.3g

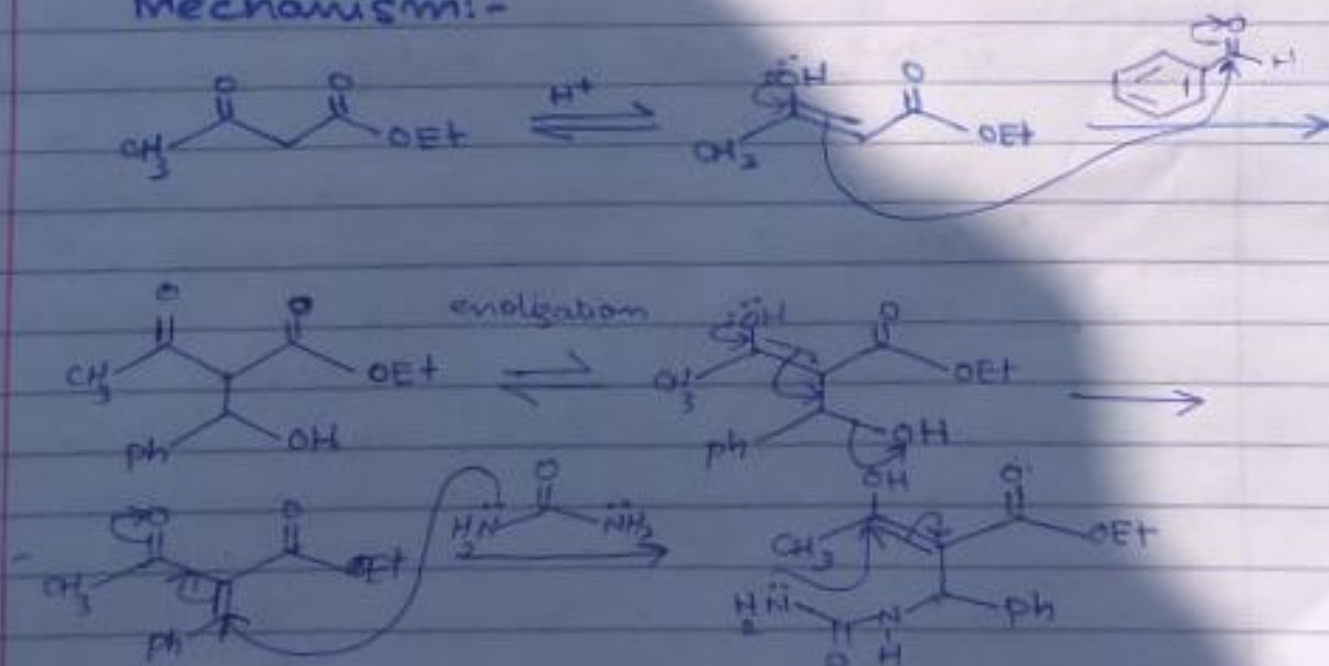
Benzaldehyde = 1.1g

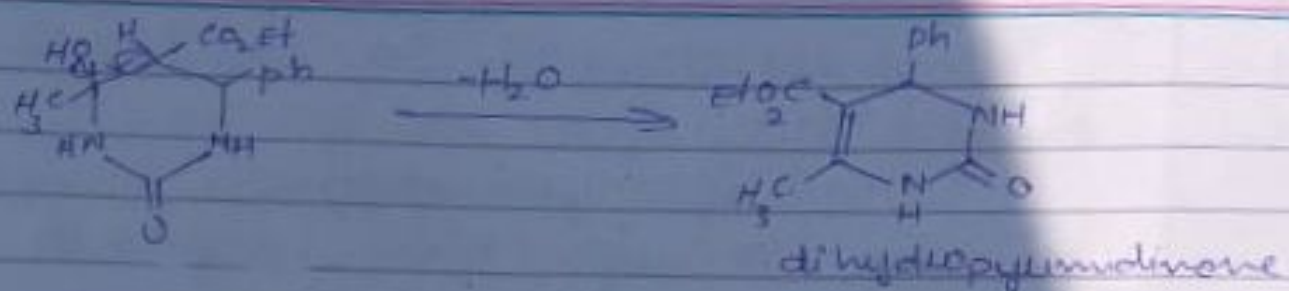
Urea = 0.7g

Chemical Equation

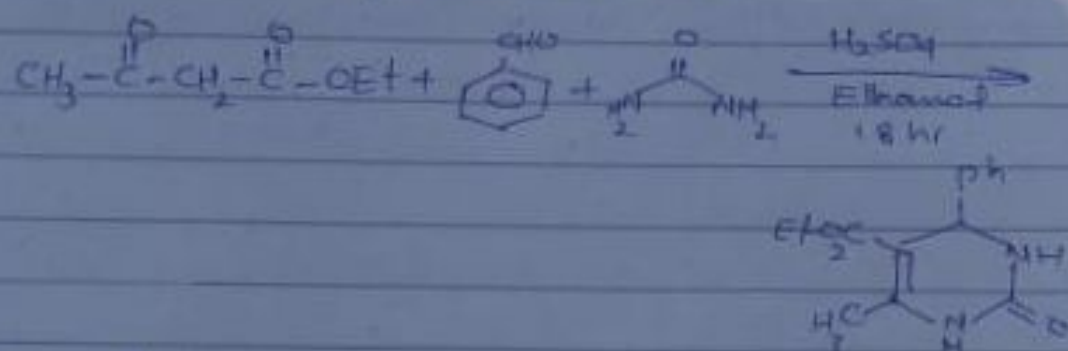


Mechanism:-





conventional procedure:-



Procedure:-

- ① The mixture of benzaldehyde, ethylacetoacetate and urea taken in round bottom flask was shaken by hand for 2 minutes.
- ② The reaction mixture was then heated in a water bath at 90°C for one hour.
- ③ with the progress of reaction a solid started to deposit and after one hour the flask was full of solid.
- ④ The solid was taken out carefully with spatula or spoon in a conical flask.
- ⑤ The yellow solid was washed with cold water (1ml) and then recrystallized from rectified spirit to give a colourless solid (m.p = $201-202^{\circ}\text{C}$)

Advantages of Green procedure:

- use of no hazardous organic solvent
- NO requirement of catalyst
- Faster reaction

RESULT:-

Dihydropyrimidinone was prepared with %age yield 74%.

Experiment No. 4

Synthesis of dihydropyrimidinone through green procedure.

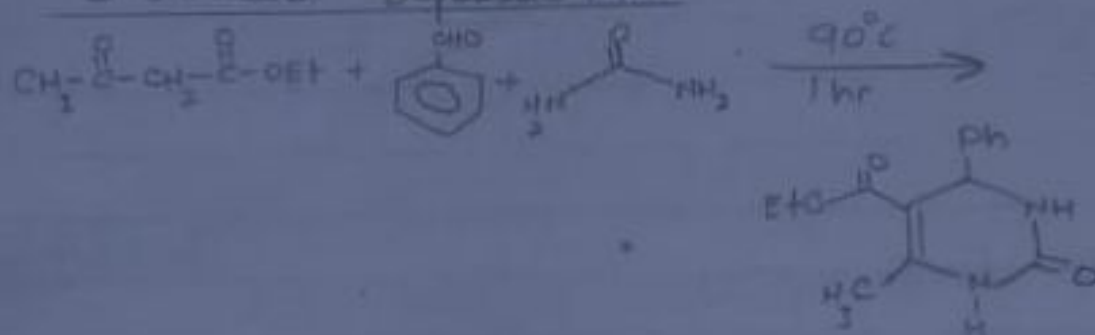
Chemicals Required:-

Ethyl acetate = 1.3g

Benzaldehyde = 1.1g

urea = 0.7g

Chemical Equation:-



Observations and Calculations

Molecular mass of ethyl acetoacetate = 118g/mol

Molar mass of Benzaldehyde = 106g/mol

Molar mass of urea = 60g/mol

Molar mass of product = 260g/mol

Theoretical yield:-

Acetoacetic Ester : product

118 : 260

1 : $\frac{260}{118}$

1.3 : $\frac{260}{118} \times 1.3 = \boxed{2.86g}$

Experiment 10.2

$$\begin{array}{lcl}
 \text{Benzaldehyde} & : & \text{product} \\
 106 & : & 234 \\
 1 & : & \frac{234}{106} \\
 3.96 & : & \frac{234}{106} \times 3.96 \\
 & & = 8.74g
 \end{array}$$

Molecular wt of acetone = 58g

density of acetone = 0.79 g/cm^3

Volume used of acetone = 1ml

$$m = d \times V$$

$$= 0.79 \times 1 = 0.79g$$

Acetone : product

$$58 : 234$$

$$1 : \frac{234}{58}$$

$$0.79g : \frac{234}{58} \times 0.79$$

$$= \boxed{3.19g}$$

Acetone is a limiting reactant.

Theoretical yield = 3.18g

Benzaldehyde : product

$$106 : 260$$

$$1 : \frac{260}{106}$$

$$1.1 : \frac{260}{106} \times 1.1 = \boxed{2.69g}$$

Urea : product

$$60 : 260$$

$$1 : \frac{260}{60}$$

$$0.7 : \frac{260}{60} \times 0.7 = \boxed{3.03g}$$

- since Benzaldehyde produces least amount of product, so it is a limiting reactant.

$$\text{Theoretical yield} = 2.69$$

$$\text{Actual yield} = 2.09$$

$$\% \text{ yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100$$

$$= \frac{2}{2.69} \times 100$$

$$= 74\%$$

RESULT:-

Dihydroxypyrimidinone was prepared through green procedure with %age yield 74%.

Experiment no. 5

109-01-19

Synthesis of cyclohexanone adipic acid from cyclohexanone.

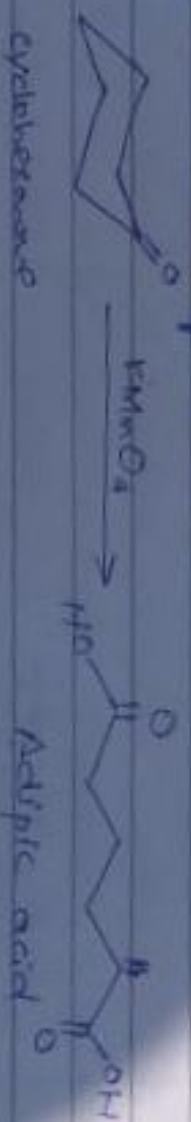
Apparatus:-

Erlenmeyer flask, glass rod, filter paper, conical flask, funnel, burner, tripod stand etc

Chemicals Required:-

- cyclohexanone = $0.0025 \text{ mole} = 0.245 \text{ g/mole}$
 $V = d \times m = 0.948 \times 0.245 = 0.232 \text{ ml}$
- $\text{KMnO}_4 = 2\% = 0.005 \text{ mole} = 0.74 \text{ g/mole}$
 $V = d \times m = 2.7 \times 0.74 = 2 \text{ ml}$
- $\text{NaOH (10\%)} \text{ solution}$

Chemical Equation:-



Adipic acid:-

Adipic acid is an important bulk chemical for the production of nylon 6,6. Industrial process uses the nitric acid oxidation of cyclohexanone or cyclohexanol. Nitric oxidizing agent do not react with ketones, but do react with aldehydes. In this case, a very strong oxidizing agent is used to form diacid, while breaking carbon-carbon bond.

Procedure:-

- ① In a 50ml Erlenmeyer flask place 0.0025 moles of cyclohexanone and a solution of 0.0050 moles of KMnO_4 in 15ml of H_2O .
- ② The solution was made slightly basic by adding 3 drops of 10% NaOH .
- ③ The solution was stirred gently for 30 minutes at room temperature, and then was placed in a boiling water bath for 20 minutes. After each 5 minutes, the solution was checked to see if the oxidizing agent had been used up, by taking a drop of the reaction mixture and transferring it onto a filter paper. The appearance of a purple ring around a dark center was the indication of remaining KMnO_4 .
- ④ If after 20 minutes, the oxidizing agent persists, it should be decomposed by addition of a small amount of Sodium bisulfite.
- ⑤ The solid produced was filtered and excess of manganese(IV) oxide was removed. The solid was washed with 2ml of hot water. The filtrate was collected.
- ⑥ The filtrate was transferred to a small beaker and volume was concentrated to less than 5ml.
- ⑦ 12M concentrated HCl was added till the

pH became acidic to litmus paper. Another 10 drops of 12M HCl were added and product was precipitated out.

(8) The solution was cooled to room temperature, the precipitated product was collected by vacuum filtration.

(9) The product was dried in air and melting point was found.

RESULT:-

The adipic acid was prepared with %age yield 65%.

Experiment No. 5

10-01-19

Synthesis of adipic acid from
cyclohexanone.

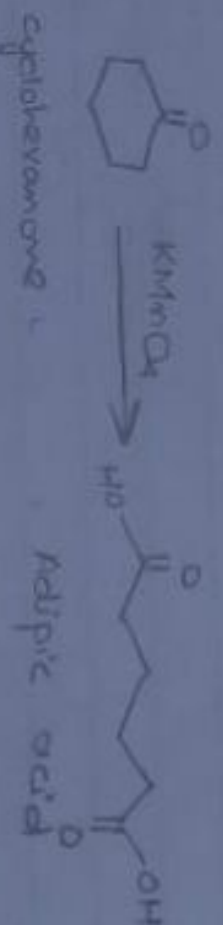
Chemicals Required:-

Cyclohexanone = 0.0025 mole

KMnO_4 = 0.005 mole

10% NaOH solution

Chemical Equation:-



Observations and Calculations

Molar mass of cyclohexanone = 98 g/mol

Molar mass of KMnO_4 = 148 g/mol

Molar mass of Adipic acid = 146 g/mol

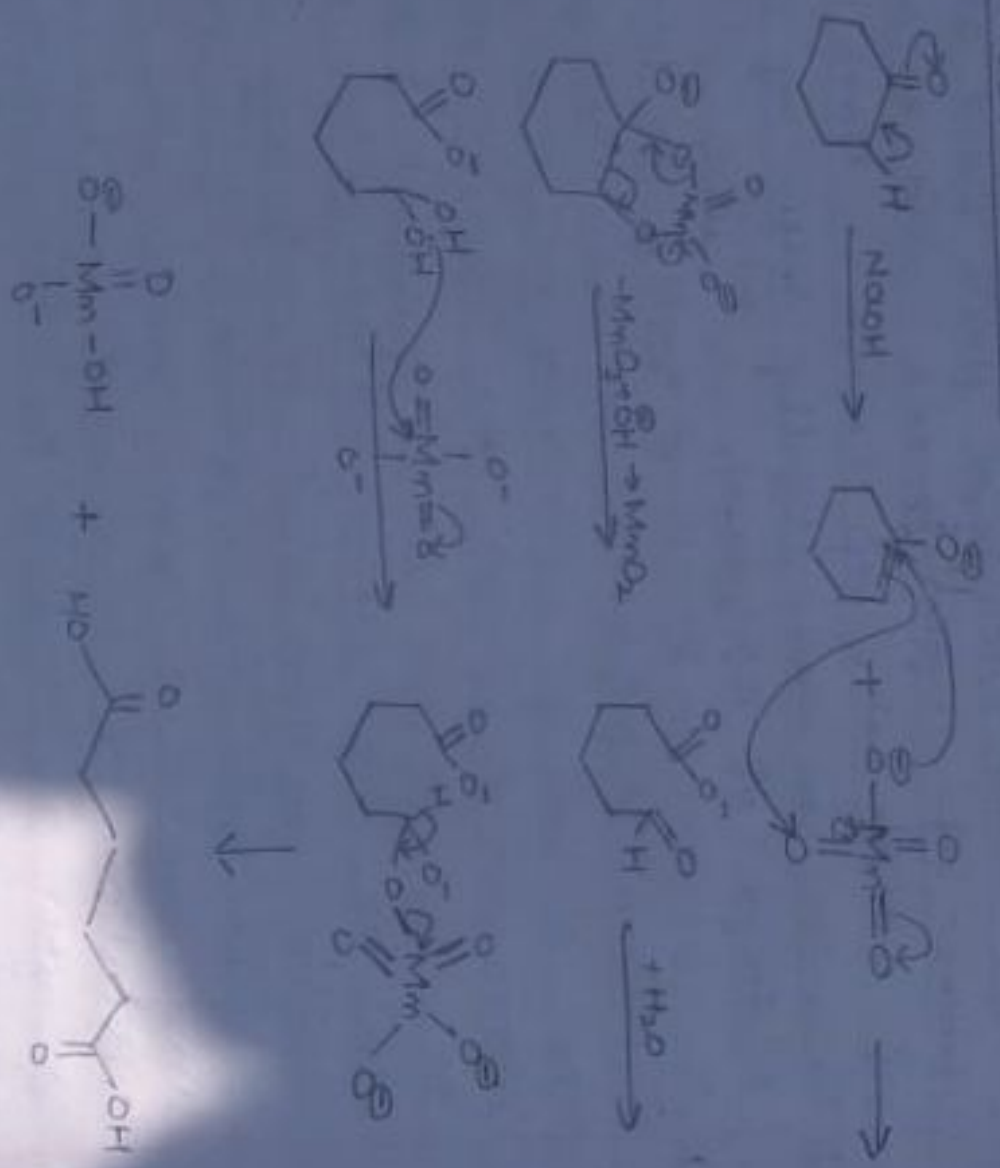
Theoretical yield:-

cyclohexanone : Adipic acid

$$\begin{array}{ccc} 98 & : & 146 \\ 1 & : & \frac{146}{98} \end{array}$$

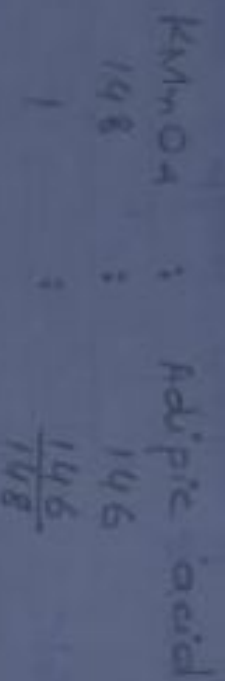
$$0.0245 \quad : \quad \frac{146}{98} \times 0.0245$$
$$= \boxed{0.365 \text{ g}}$$

Mechanism :-



RESULT :-

The adipic acid was prepared with % age yield of 65%...



$$0.74 \times \frac{146}{148} \times 0.74 = \boxed{0.739 \text{ g}}$$

• Since cyclohexanone produces least amount of product, so it is a limiting reagent.

$$\text{Theoretical yield} = 0.365 \text{ g}$$

$$\text{Actual yield} = 0.24 \text{ g}$$

% Yield:-

$$\% \text{ age yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100$$

$$= \frac{0.24}{0.365} \times 100 =$$

$$= 65 \%$$

RESULT:-

Adipic acid was prepared with %age yield 65%.

Experiment No. 6

17-01-19

Preparation of Benzilic acid from Benzil.

Apparatus:-

Round bottom flask, Reflux set, cotton
therm stand, glass rod, beakers, funnel, filter
paper, chemical flask, oven, ice bath
burner, tripod stand etc

Chemicals Required:-

Benzil = 2-3g

Methanol = 12ml

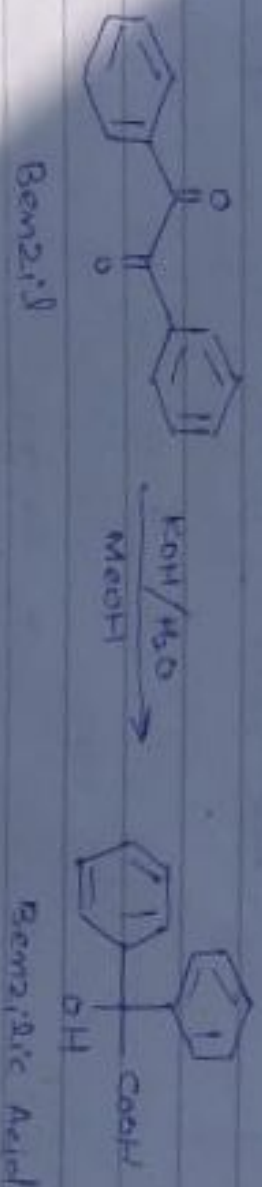
KOH = 6ml

Hot H₂O = 2ml

Distilled H₂O = 120ml

H₂PO₄ = 6ml

Chemical Equation:-



Principle:-

α -diketones (Benzil) upon refluxing
with hydroxide ion (acts as nucleophile)
gives benilic acid. This is called Benzil
Benzilic acid rearrangement.

Procedure:-

- ① 100ml round bottom flask containing 12 ml of methanol was taken. 2.5g of benzal was dissolved in methanol (just if necessary).
- ② 6-8ml of KOH solution was added to the reaction flask, while stirring the contents.
- ③ Condenser was fitted to the reaction flask, reaction mixture was refluxed for 30 minutes, the initial black brown colouration changed to brown colouration.
- ④ The reaction mixture was transferred to evaporating dish and evaporated till the solid layer appeared on liquid surface.
- ⑤ The reaction mixture was cooled by the means of ice bath under the residue solidified.
- ⑥ The precipitated solid (potassium benzoate) was washed with 2ml of ice-cold 95% of aq. methanol.
- ⑦ The precipitates were then dissolved in 2ml of hot water and filtered with double filter paper.

⑧ The filtrate was acidified by adding kmol of phosphoric acid in Erlenmeyer flask.

⑨ The filtrate was allowed to cool to room temperature and in ice bath. for pure oxalates.

⑩ The precipitated benzoic acid was collected by filtering the reaction mixture

⑪ The precipitates were dried, weighed and %age was calculated.

RESULT:-

The benzoic acid was prepared with %age yield 66.4%.

Experiment No. 6

17-01-19

Preparation of Benzoic acid from Benzal

Chemicals Required:-

Benzal = 2.3g

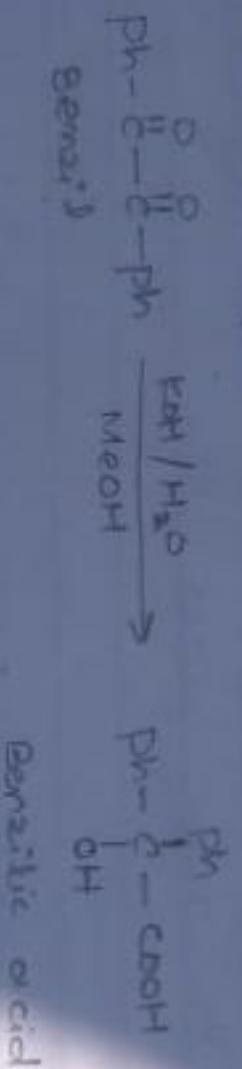
Methanol = 12 ml

KOH = 6 ml (0.448g)

Hot H_2O = 2 ml

H_3PO_4 = 6 ml

Chemical Equation:-



Observations and Calculations

Molar mass of Benzal = 210 g/mol

Molar mass of KOH = 56 g/mol

Molar mass of CH_3OH = 32 g/mol

Molar mass of Benzoic acid = 228 g/mol

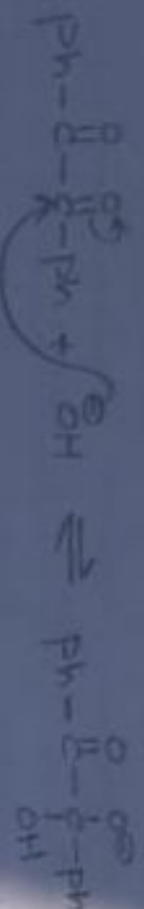
Theoretical Yield:-

Benzal : Benzoic acid

$$\begin{array}{r} 210 \quad : \quad 228 \\ 1 \quad : \quad \frac{228}{210} \end{array}$$

Mechanism:-

1- step one: adduct formation



2- step two: rearrangement step (rate determining step)

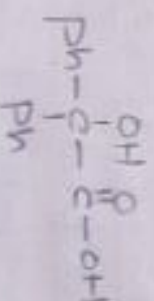


* Formation of C=O group provides the driving force for the rearrangement.

③ step three: protonation/salt formation

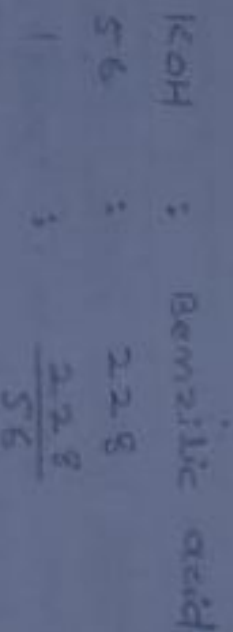


Potassium
benzoate



Benzoic acid

$$2.3 \text{ g} \times \frac{228}{210} = 2.49 \text{ g}$$



$$0.448 \text{ g} \times \frac{228}{56} = 1.824 \text{ g}$$

• Since KOH produces least amount of product, so it is a limiting reactant.

Theoretical yield = 1.824 g

Actual yield = 1.21 g

$$\begin{aligned} \% \text{ age yield} &= \frac{\text{Actual yield}}{\text{Theoretical yield}} \times 100 \\ &= \frac{1.21}{1.824} \times 100 \\ &= 66.4\% \end{aligned}$$

RESULT:-

The benzilic acid was prepared with %age yield 66.4%.