

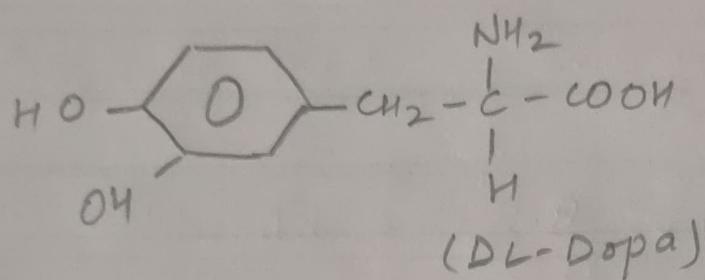
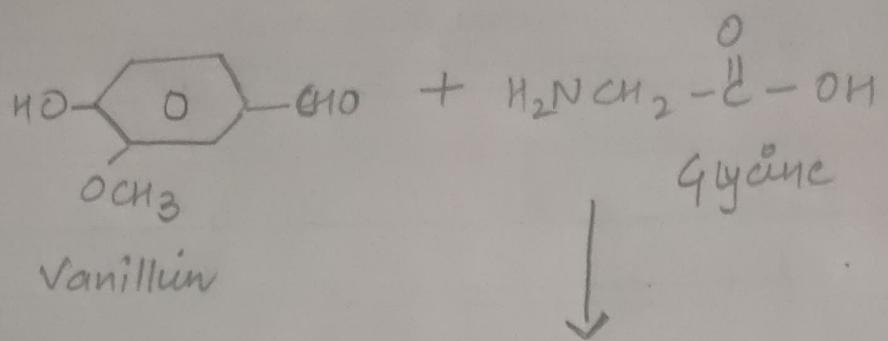
Object:- To synthesize and characterize levodopa from vanillin.

Reference:- Kae Ashutosh "Medicinal chemistry" 5th edition, New age International publisher.

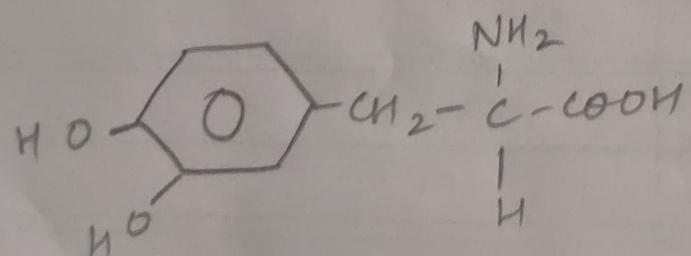
Requirements :- Glass rod, spatula, conical flask, round bottom flask, pipette, measuring cylinder, funnel

Theory:-

It is considered as one of the most important drug for the treatment of parkinsonism. It is also been used successfully to control the neurological symptoms arising from chronic manganese poisoning, which incidentally resemble those of parkinsonism. Since dopamine does not cross the blood brain barrier. It has no therapeutic utility. However its immediate metabolic product, levodopa is transported into the brain by large neutral amino acid transporter &



Hydrolysis with aq. HBr
 Resolution by d-phenyl ethylamine.
 conversion to DL-N-acetyl 3-methoxy 4 acetoxy - phenylalanine.



Levodopa

and permeates into tissue, where it is decarboxylated. L-DOPA is rapidly absorbed by active transport GI tract & once absorbed; it is 95% decarboxylated in the periphery.

D,L-dopa may be first prepared from vanillin and glycine which is then converted to D,L-N-acetyl-β-methoxy-4-acetoxy phenylalanine. The resulting product is then resolved by means of α-phenyl ethyl amine which upon hydrolysis with aq. HBr forms L-DOPA.

Procedure:-

- (1) Mix 3 gm of Vanillin with 2.33 ml of glycine in 10 ml methanol in a round bottom flask and shake.
- (2) This mixture is allowed to reflux and boil under for 2 hours, then cool it to room temperature.
- (3) Then reaction mixture is mixed thoroughly with 15 ml of water and allow to stand for 30 minutes.
- (4) Then filter the solution under vacuum to remove the insoluble product.

152.15 g of vanillin \rightarrow 197.18 gm of levodopa

$$3 \text{ gm vanillin} = \frac{197.18}{152.15} \times 3$$

$$\text{Theoretical yield} = 3.8 \text{ gm}$$

$$\text{Practical yield} = 2.3 \text{ gm}$$

$$\% \text{ yield} = \frac{2.3}{3.8} \times 100 = 73.68\%$$

(5) Treat the filtrate with conc. HCl, cool on ice water filter off the precipitate & then recrystallize.

Result:- % yield of Levodopa was found to be 73.68 %.

Object:- To synthesize & characterize benzanilide from aniline & benzoyl chloride.

Reference:- Kau Ashutosh "Medicinal Chemistry"
5th edition, New age International
Publisher.

Requirements :- measuring cylinder, conical flask, beaker, glass rod.

Theory:-

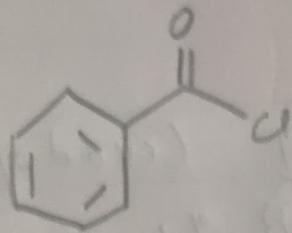
Benzanilide is the organic compound with the formula $C_6H_5CO-NH-C_6H_5$.

It is a white solid; commercially available, it may be prepared by heating benzoic acid with aniline.

Insertion of benzoyl moiety instead of an active hydrogen atom present in primary amino or secondary amino group is usually termed as benzoylation reaction. This particular reaction essentially bears a close resemblance to the phenomena of acetylation except that in this specific instance the reagent is which reacts in the presence of 10% NaOH & not benzoic anhydride.



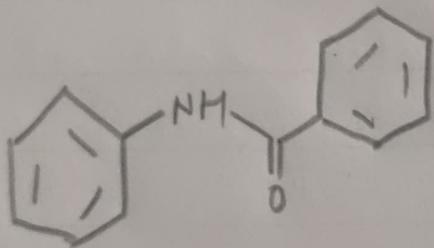
Aniline



Benzoyl
chloride



10% Ag·NaOH



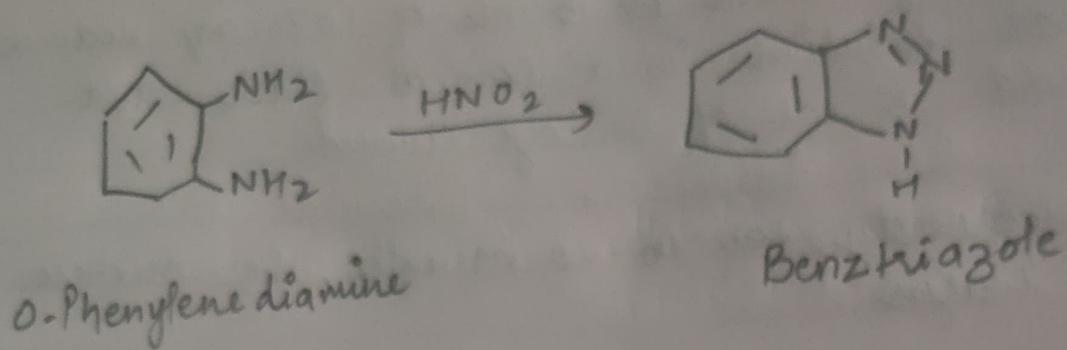
Benzanilide

Procedure :-

- (1) Add 1 ml of aniline to 15 ml of 10% aq. sodium hydroxide solution in a conical flask, and then add 5 ml of benzoyl chloride.
- (2) Shake vigorously for 15-20 minutes, the mixture becomes warm.
- (3) The crude benzoyl derivative separates as a white powder, when the reaction is complete, filter off the product at the pump, & then thoroughly wash with water.
- (4) Recrystallise the benzanilide from hot methylated spirit, as the benzanilide may crystallise rapidly as the solution cools.

Result:- % yield of benzanilide was found to be
71%

9-8722



Object:- To synthesize & characterize benzotriazole.

Reference:- Kae Ashutosh "Medicinal Chemistry"
15th edition, New age International
publisher.

Requirements :- beaker, glass rod, pipette, measuring cylinder, ice bath, spatula.

Theory:-

It is a heterocyclic compound containing 3 'N' atoms, with the chemical formula $C_6H_5N_3$.

This aromatic compound is colorless & polar and can be used in various fields.

Benzotriazole features two fused rings.

Its five membered ring can exist in tautomers;

These are used as restrainer in photographic emulsions & as a reagent for the analytical determination of silver.

Its derivatives & their effectiveness as drug precursors are also used in various fields.

108.1 g of o-phenyldiamine \rightarrow 119.12 gm of benzimidazole

$$10.8 \text{ gm} \rightarrow \frac{119.12}{108.1} \times 10.8$$

Theoretical yield = 11.9 gm

Practical yield = 9.9 gm

$$\% \text{ yield} = \frac{9.9}{11.9} \times 100 = 83.19\%$$

Procedure :-

- (1) Dissolve 10.8 gm of o-phenylenediamine in a mixture of 12 g of glacial acetic acid & 30 ml of water in 250 ml beaker, warm slightly.
- (2) cool the clear solution to 15°C, stir magnetically and then add a solution of 7.5 gm of sodium nitrite in 15 ml of water, the rxn mixture becomes warm, within 2-3 minutes temp. reaches to about 85°C & then begins to cool while the colour changes from deep red to pale brown.
- (3) Continue stirring for 15 min, the temp. will have a drop to 35-40°C & then thoroughly chill in a ice-water bath for 30 min.
- (4) Collect by vacuum filtration the pale brown solid which separates out & wash with 3 30 ml portions of ice-cool water.
- (5) Dissolve the solid in about 130 ml of boiling water, add decolorising charcoal, filter & allow the filtrate to cool to about 60°C & add few crystals of crude benzotriazole for seeding.
- (6) Allow the mixture to attain room temperature.

Slowly & then thoroughly chill in ice & collect the Benzotriazole which separates as pale pale yellow or pale straw - coloured needles.

Result:- % yield of benzotriazole was found to be 83.19 %.

Object:- To synthesize & characterize chlorobutanol from acetone.

Reference:- Pandya Surendra Nath "A text book of medicinal chemistry Volume - I , publisher Varanasi.

Requirements :- Round bottom flask, glass rod, measuring cylinder, spatula.

Theory .

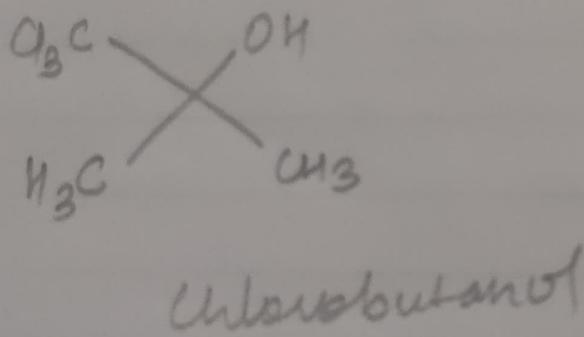
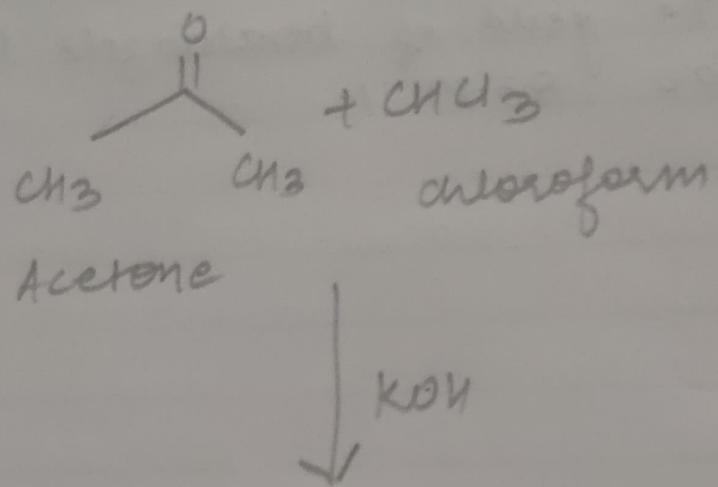
chlorobutanol (Chloro - 2 - methyl - 2 - propanol) is a preservative, sedative, hypnotic & weak local anaesthetic similar in nature to chloral hydrate.

It has anti bacterial & antifungal properties .

Chlorobutanol is typically used at a conc. of 0.5% where it lends long term stability to multi-ingredient formulations . It is a white volatile solid with a menthol-like odor .

chlorobutanol is highly toxic to the liver, is a skin irritant and a severe eye irritant .

It is formed by exn of chloroform & acetone in presence of KOH or NaOH .



Expt. No.

Procedure:-

Add 11 ml of chloroform and 1 gm of solid potassium hydroxide in a round bottom flask & shake until the potassium hydroxide dissolves. To this add 14 ml of acetone & was again shaken for 15 minutes. Then keep it aside for half an hour & crystals of chlorobutanol separates out.

Result:- % yield of chlorobutanol was found to be 74%.

Object:- To synthesize & characterize Sulfanilamide from aniline acetanilide

Reference:- Kas Ashutosh "Medicinal chemistry"
5th edition new age International
Publishers.

Requirements:- Conical flask, beaker, glass rod,
ice bath, Spatula, measuring cylinder,
filter paper

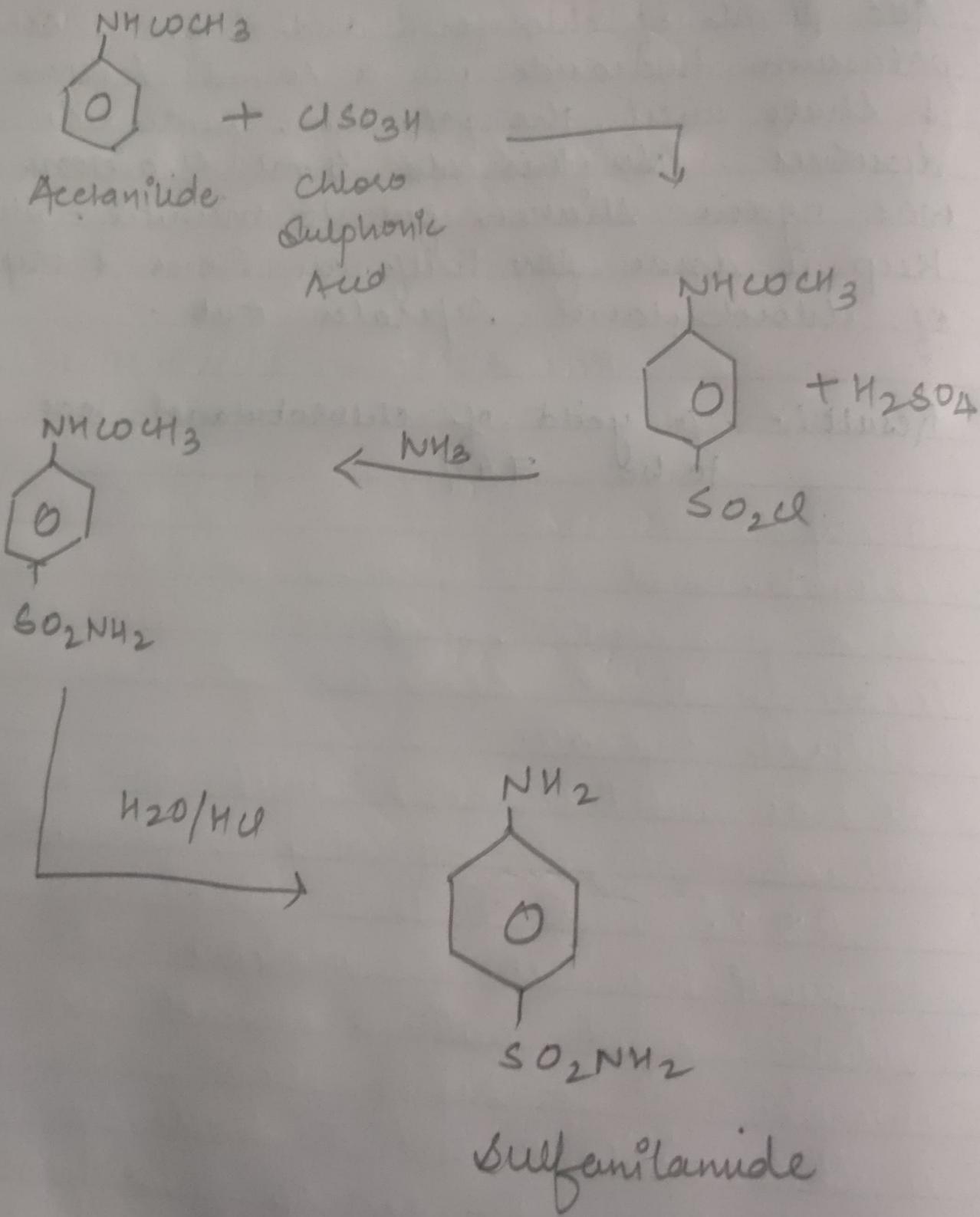
Theory:-

Sulfanilamide is an organic sulfur compound structurally similar to p-aminobenzoic acid (PABA) with anti-bacterial property.

Sulfanilamide competes with PABA for the bacterial enzyme dihydro pterotate synthase, thereby preventing the incorporation of PABA into dihydrofolic acid, the immediate precursor of folic acid.

It is primarily used today for treatment of yeast infections.

Sulfanilamide was first prepared in 1908 by Austrian chemist Paul Josef Jakob Gelmo (1879 - 1961), as a part of dissertation for



doctoral degree from Technische Hochschule of Vienna. It was patented in 1909. Powdered Sulfanilamide was used by the Allies in World War II to reduce infection rates & reduction in mortality rates.

Procedure:-

- (1) Add 25g of dry powdered acetanilide to 63 ml of chlorosulfonic acid in 250 ml of conical flask. Heat the contents to $60 - 70^{\circ}\text{C}$ for 2 hours.
- (2) cool the mixture & then pour it carefully on to the crushed ice, whereby the $\text{P-}\alpha\text{-acetamido benzyl sulphonyl chloride}$ separates out as a white solid.
- (3) filter off the product & use it as raw material for next step.
- (4) Place the above obtained salt in 500 ml of conical flask & add 120 ml of conc. ammonia.
- (5) Stir the mixture at 70°C for 30 min with occasional stirring.

135.1 gm of Acetanilide \rightarrow 172.2 gm of Sulphanilamide

$$25g \rightarrow \frac{172.2}{135.1} \times 25$$

Theoretical yield - 31.8 gm

Practical yield - 26.2 gm

$$\% \text{ yield} = \frac{26.2}{31.8} \times 100 \\ = 82.3\%$$

- (6) Cool the mixture & make it just acidic with dil. H_2SO_4 .
- (7) filter off the ppt. p-acetamido benzene Sulphonamide at pump & wash it well with cold water.
- (8) Take the product from above step & mix with HCl (10 ml conc. + 20 mL H_2O) & boil this mixture under reflux for 1 hr.
- (9) Filter the boiling solution & add Na_2WO_4 until the effervescence stops & Sulphanilamide is ppt. as white powder.
- (10) cool the mixture & filter off the Sulphanilamide obtained, wash with H_2O & dry.

Result:- % of Sulphanilamide was found to be
82.31.

Object:- To synthesize & characterize Kiphenyl imidazole from benzil.

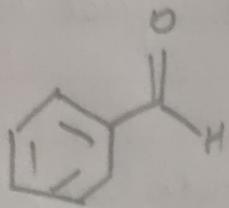
Reference :- Kar Ashutosh "Medicinal Chemistry"
5th edition, new age International
publisher.

Requirements:- beaker, measuring cylinder, glass rod, conical flask.

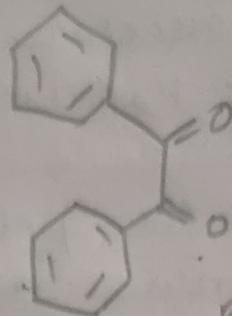
Theory:-

This drug was synthesized by refluxing benzoin, Benzaldehyde & ammonium bi carbonate in equimolar quantities.
The Mannich bases were synthesized by using abstractable 'H' present in 2,4,5-Kiphenyl imidazole because various drugs obtained from Mannich reaction have proved more effective & less toxic than their parent drugs.

It is mainly used as fungicides & antifungal, antiprotozoal & antihypertensive medications. Imidazole is a part of the theophyllin molecule found in tea leaves & coffee beans.



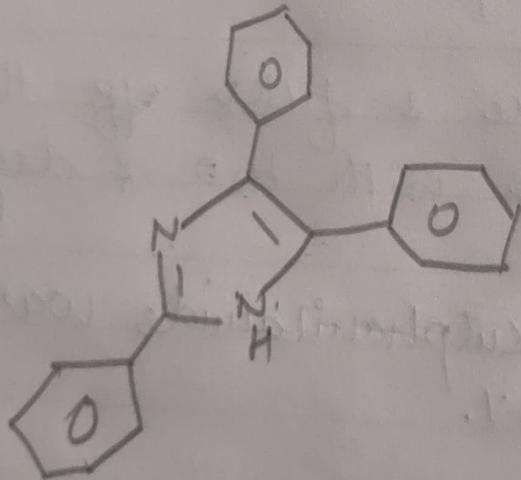
Benzaldehyde



Benzil

+ 2NH₄OAc

EtOH, reflux



triphenyl - imidazole

Procedure

- (1) To a reflux apparatus, add 107 ml of glacial acetic acid & 11 gm of ammonium bicarbonate until CO_2 evolves out.
- (2) Add 2.25 gm of dry benzil & 2.5 ml of benzaldehyde.
- (3) Heat under reflux for 2 hrs. cool the solution down & pour into 300 ml of ice or chilled water.
- (4) Neutralize the mixture with ammonium, filter & wash with H_2O .
- (5) Recrystallize the product from 1:4 ratio of H_2O ethanol.

Result: = % yield of triphenylimidazole was found to be 87%.

210.23 g of benzil \rightarrow 296.4 g of triphenylimidazole

$$2.25 \text{ g} = \frac{296.4}{210.23} \times 2.25$$

Theoretical yield = 3.1 gm

Practical yield = 2.7 gm

$$\% \text{ yield} = \frac{2.7}{3.1} \times 100 = 87\%$$