KONGUNADU ARTS AND SCIENCE COLLEGE

(AUTONOMOUS)

Re-accredited by NAAC with A+ Grade - 4th cycle, College of Excellence - UGC

COIMBATORE -29.

DEPARTMENT OF CHEMISTRY

Supported by : DBT STAR College Scheme

LAB MANUAL & SOP

(STANDARD OPERATING PROCEDURE)

CORE PRACTICAL- V : APPLICATION ORIENTED PRACTICALS

Name : …….

Class : …….

Roll No. :…….

www.kongunaducollege.ac.in

TABLE OF CONTENTS

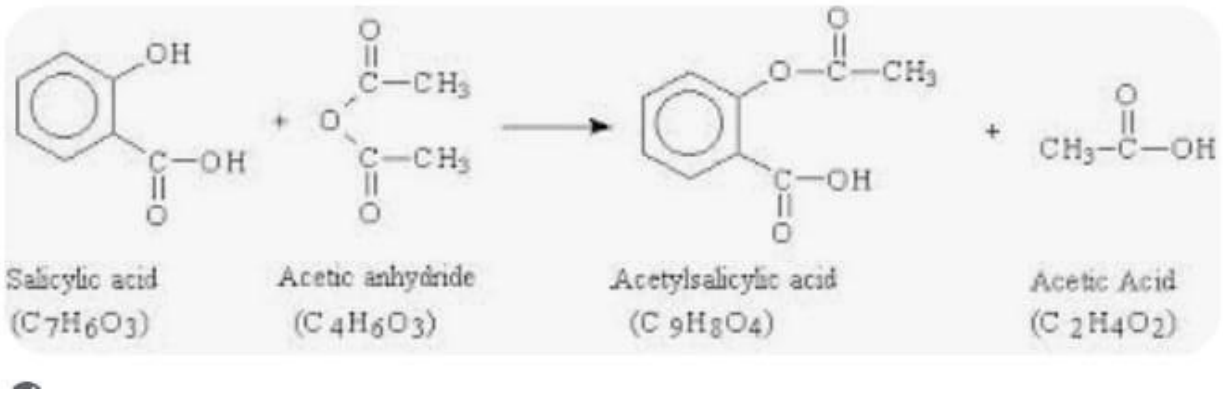
PREPARATION OF ACETYL SALICYLIC ACID FROM SALICYLIC ACID

AIM:

To prepare acetyl salicylic from salicylic acid.

PRINCIPLE:

Salicylic acid is a phenolic acid .The phenolic group can easily be acetylated using acetic anhydride or acetyl chloride .When salicylic acid is boiled with acetic anhydride ,acetyl salicylic acid is obtained.



CHEMICALS REQUIRED:

1. Salicylic acid – 1 g
2. Acetic anhydride – 2 ml
3. Conc.H2SO4 – 1 drop
4. Ethanol (Rectified spirit)– 7 ml

PROCEDURE:

Salicylic acid is mixed with acetic anhydride in a 100 ml conical flask and 1 drop conc.H2So4 is added. The mixture is shaken well and heated gently on a water bath at 50°c – 60° c for about 10-15 minutes with occasional shaking .Then the contents are cooled under the tap water .The acetyl salicylic acid that separates out is filtered off at the pump, washed with water ,dried ,filtered and weighed.

RECRYSTALLISATION:

The crude acetyl salicylic acid is recrystallised as follows. About 1 g of the sample is dissolved in the minimum amount of hot ethanol and then poured into 40 ml of water in a 250 ml beaker .The turbid solution is then warmed to get a clear solution .The resulting clear solution is filtered and allowed to cool slowly. When heat of acetyl salicylic acid separates out. It does not posses at definite melting point.
[decomposes at 128° c to 135° c]

RESULT:

The yield of acetyl salicylic acid =

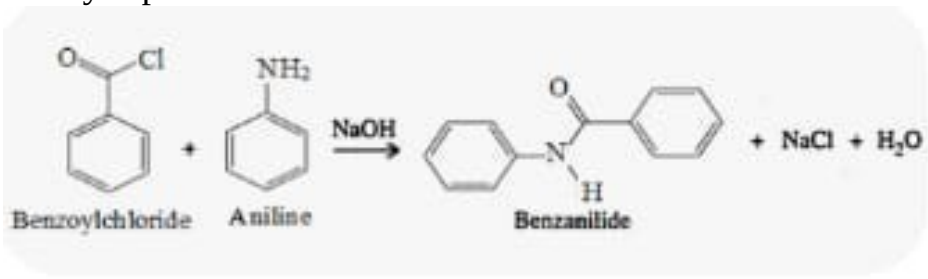
PREPARATION OF BENZANILIDE FROM ANILINE (SCHOTTEN – BAUMANN REACTION)

AIM:

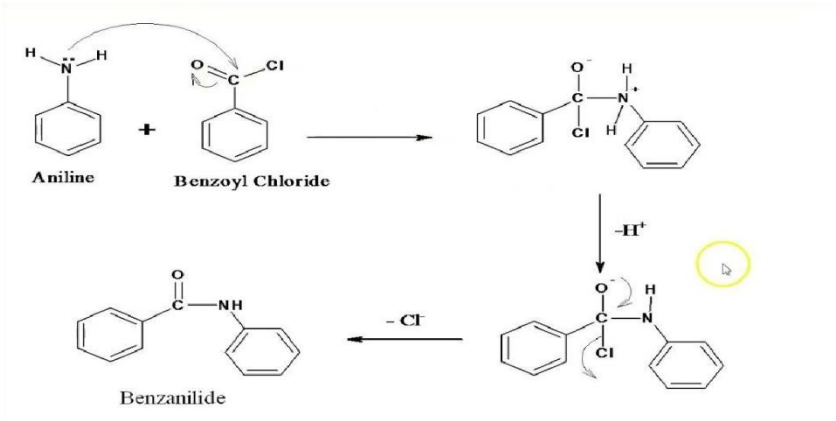
To prepare a pure sample of crystals of benzanilide from aniline.

PRINCIPLE:

Aromatic amines in general react with benzoyl chloride in aqueous medium to yield the respective benzanilides. In schotten - baumann methods of Benzylation the amine compound is treated with a slight excess of sodium hydroxide solution and benzoyl chlriode or vigorous shaking, benzoylation takes place readily and the product respectively separates as a solid.



The mechanism of the reaction is as follows:



CHEMICALS REQUIRED:

1.Aniline – 2 ml

2.Benzoyl chloride – 3.5 ml

3. 10 % of NaOH 0- 25 ml

PROCEDURE:

2.5 ml of aniline and 25 ml of 10% NaOH are placed in a 100 ml conical flask and stoppered well 3.5 ml of benzoyl chloride are introduced into the flask and stoppered well for 10 to 15 minutes. The progress of the reaction is known by its exothermal character. Completion of the reaction is tested by smelling the presence of benzoyl chloride in the flask. If the smell persists, add a little more sodium hydroxide solution and the continue the shaking for the white crystals. The white crystals of benzanilide are filtered at the pump, washed well with water and dried

RECRYSTALLISATION:

The little amount of the sample is recrystallised from hot methylated spirit.It is filtered through hot water funnel.The colourless crystals are separated and dried in air.

RESULT:

The yield of benzanilide =

PREPARATION OF PARABROMO ACETANILIDE FROM ACETANILIDE

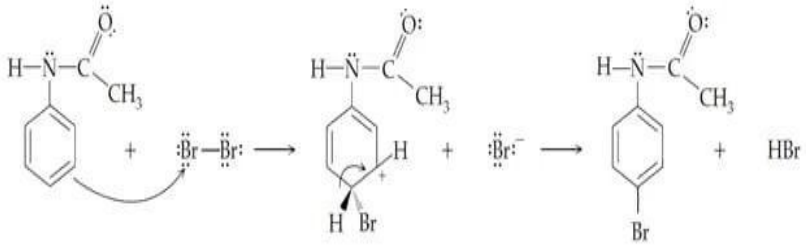
AIM:

To prepare pure crystals of parabromo acetanilide from acetanilide.

PRINCIPLE:

Primary amines do not yield monosubstituted products by direct reaction with reagents such as bromine (or) nitration mixture.

When the amine group is protected as in the case of acetanilide. The substituted reaction readily occurs with bromine. Acetanilide product produces pbromo acetanilide as the main product. Through a little amount of O- isomer on recystallisation.



MATERIALS REQUIRED:

1.Acetanilide -1 g

2.Acetic acid – 3 ml

3. Bromine acetic acid – 7 ml

PROCEDURE:

About 1 g of finely powdered acetanilide is dissolved in 5 ml of glacial acetic acid in a RB flask. In another conical flask , 7 ml of bromine in acetic acid is transferred into a burette (or) separating funnel. The bromine solution is added slowly into the RB flask. Containing acetanilide and shaken well to make through mixing. The flask may be cooled in ice cold water. When all the bremine has been added. The solution will be have an orange colour due to ercess of bromine. The RB flask is well shaken and the contents are poured into a beaker containing 200ml water. The precipitate P-bromo acetanilide is filtered at the pump using buchner funnel and dried over. At the filter paper, the colourless sample of P – bromo acetanilide is obtained.

RECRYSTALLISATION:

A small amount of crude acetanilide is dissolved in a dilute solution of methylated spirit and recrystallised. The colourless crystals of P- bromo acetanilide are separated and dried.

RESULT:

The yield of P – bromo acetanilide =

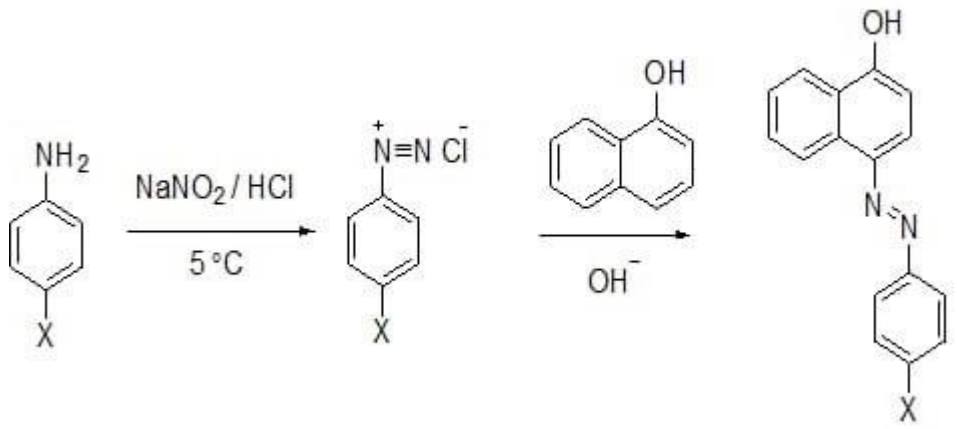
PREPARATION OF P-NITROPHENYL AZO-ß-NAPHTHOL

AIM:

To prepare pure sample of p-nitrophenyl azo-b-naphthol by treating pnitrophenyl and -b-naphthol in alkaline medium.

PRINCIPLE:

P-nitro aniline on diazolysation. Followed by coupling with b-naphthol in alkaline medium gives the azodye.P-nitrophenyl azo-b-naphthol.



MATERIALS REQUIRED:

1.P-Nitro aniline

2.Conc.H2SO4

3.Sodium Nitrate

4.B- Naphthol

5.Sodium acetate

6.Sodium hydroxide

PROCEDURE:

The p-nitro aniline is mixed with the diluted acid in a 250 ml beaker and cooled in a freezing mixture of ice and salt.The nitrite solution is added slowly and carefully from a dropping funnel.At first the P- nitro aniline dissolves,and a pasty mass of diazo compound and separates.The sodium acetate solution of BNapthol in alkali is diluted to 50 ml in a 400 ml beaker.The diazo solution is then taken and mixed with stirring coupling takes place and a scarlet precipitate of the dye is obtained.After keeping dye about 10 minutes.The dye is filtered at pump,washed with hot water and dried.

RESULT:

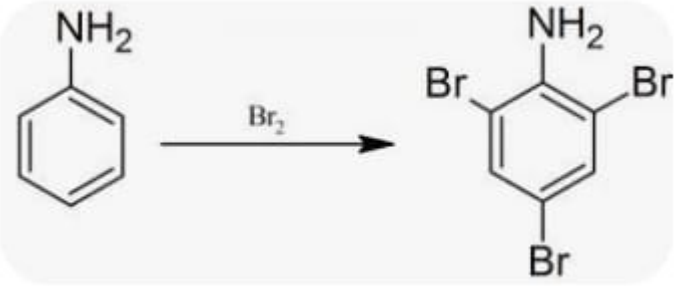
The yield of P-nitro azo-b-naphthol =

PREPARATION OF TRI – BROMO ANILINE FROM ANILINE

AIM:

To prepare a pure sample of tribromo aniline from aniline.

PRINCIPLE:



Aniline is easily brominated by treating with bromine in glacial acetic acid to yield 2,4,6-tri-bromo aniline

MATERIALS REQUIRED:

1. Aniline -2.5 ml
2. Acetic acid(glacial)-9.5 ml
3. Bromine in glacial CH3COOH – 4.2 ml BR2 / 10 ml of CH3COOH(glacial)

PROCEDURE:

In a R.B Flask 2.5 ml of aniline is dissolved in 9.5 ml of glacial acetic acid.In a separating funnel ,4.2 ml of bromine liquid is mixed with 10 ml of glacial acetic acid and stopped well.Slowly and carefully.The bromine solution is added to the aniline taken in the R.B Flask since a reaction is exothermic after each addition of bromine the flask is cooled in ice water.When all the bromine has been added.The final product is yellow in colour.The contents of flask are poured into a beaker containing 200 ml of water and stirred ware the precipitate tribromo aniline is filtered at the pump ,washed with cold water and dried in air.

RECRYSTALLISATION:

A Part of the sample is recrystallised fron rectified spirit solution fine yellow crystals of tribromo aniline are separated and dried.

RESULT:

The yield of Tri-bromo aniline =

ISOLATION

ISOLATION OF A CITRIC ACID FROM LEMON

AIM:
To prepare a isolation of citric acid from lemon.

REQUIRED MATERIALS:

1. Lemon juice
2. Calcium Chloride(10%)
3. Dil.Ammonia
4. Dil.H2S04

PROCEDURE:

About 100 ml of either lemon juice (or) pine apple juice is taken and diluted with double the amount of H2O.The juice is neutralized byb adding Dil.ammonium slowly with constant stirring.The citric acid present in the juice is now converted into ammonium citrate .About to the solution of ammonium citrate. About 2 ml of 10% aqueous solution of acid is added.The resulting solution is heated to boiling and set aside for 20 minutes. A white precipitate of tricalcium citrate is formed.The precipitate is washed and decanted from the tricalcium citrate.Citric acid can be obtained by treating with dilute H2SO4.The white precipitate of CASO4 formed is filtered and the filtrate is concentrated where crystals of citric acid appears.

RESULT:

The amount of citric acid obtained from lemon taken =