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Lab Manual for III B.Sc. Chemistry

PRACTICAL ORGANIC CHEMISTRY

DETERMINATION OF BOILING POINT OF LIQUID

Aim :

To determine the boiling point of a liquid.

Principle :

A small amount of the liquid is taken in a test tube with a side tube and fitted with a thermometer. The liquid in the test tube is heated slowly and temperature at which the liquid boils is noted.

Apparatus :

(i). Test tube with a side tube

(ii). Thermometer (110°C)

(iii). Adapter

(iv). Receiver

(v). Stand.

Procedure :

The boiling point apparatus is usually a pyrex test tube with a side tube. The given liquid is taken to about one third of tube with side tube. To promote uniform heating some porcelain pieces are introduced into it. The mouth of the tube is closed with one holed cork carrying the thermometer. The thermometer is so arranged that the tip of the bulb is in level with the side tube of the test tube. The side tube of the apparatus is connected to an adapter which in turn is introduced into a receiver.

The tube is then slowly heated on a wire gauze. The temperature rises gradually and finally the liquid boils and the vapour escapes through the side tube and collects in the receiver in drops. The constant temperature at which the liquid distils steadily is the boiling point of the liquid. When the whole of the liquid has been distilled away heating is stopped. The apparatus is cooled. The experiment is repeated with the collected liquid to get concordant results.

Result :

The boiling point of the given liquid I = °C
The boiling point of the given liquid II = °C
The boiling point of the given liquid III = °C

BROMINATION

1. Preparation of para-Bromo acetanilide

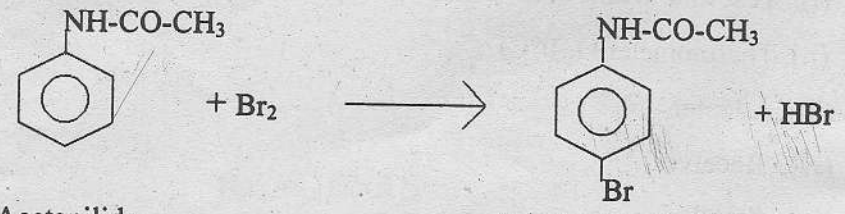
Ex No : Date :

Aim :

To prepare para-bromo acetanilide from Acetanilide.

Principle :

Acetanilide dissolved in glacial acetic acid is brominated by bromine in glacial acetic acid to give para-bromo acetanilide.



Acetanilide p-bromo acetanilide

Chemicals required:

1. Acetanilide ..... 3.5 g
2. Glacial acetic acid ..... 12. 5 ml
3. Bromine .....1.5 ml in 6.5 ml glacial acetic acid.

Procedure :

3.5 g of finely powdered acetanilide is dissolved in 6.5 ml of glacial acetic acid contained in a **conical flask.** The bromine in glacial acetic acid is added gradually from a burette or a separating funnel with constant shaking. When the addition is over, the contents of the flask are kept aside for about 20 minutes. The solution is then poured into about 100 ml of cold water taken in a **beaker,** and a few pieces of ice are added. The mixture is stirred and the precipitated parabromoacetanilide is filtered, washed with cold water and dried. The yield is noted.

About one gram of the compound is recrystallised from alcohol.

Result :

The yield of para-bromoacetanilide. =

Theoretical yield = 2 g.

BROMINATION

2. Preparation of Tribromo aniline

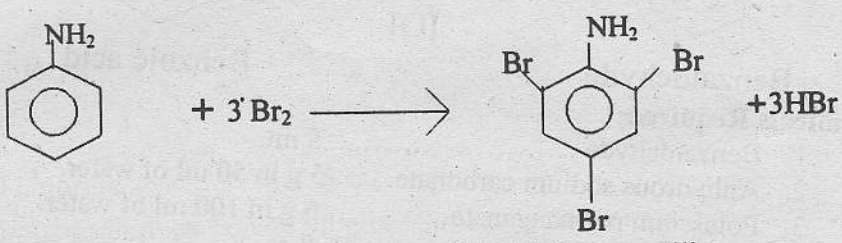
Ex No : Date :

Aim :

To prepare Tribromo aniline from Aniline.

Principle :

Aniline in glacial acetic acid is treated with bromine in glacial acetic acid in the cold when tribromoaniline is formed.



Aniline 2,4,6-tribromo aniline

Chemicals Required :

1. Aniline……2 ml in 5 ml of glacial acetic acid.
2. Bromine…..2.5 ml bromine in 10 ml of glacial acetic acid.

Procedure :

2 ml of aniline is dissolved in 5 ml of glacial acetic acid contained in a **conical flask.** The solution of bromine in glacial acetic acid is added in drops from a **burette** with constant shaking. The addition and shaking are continued until the liquid is coloured light yellow. The mixture is then added to about 75 ml of water. The crystallised tribromoaniline is filtered, washed with a little cold water, dried and the yield is noted.

About one gram of the compound is recrystallised from alcohol.

Result :

The yield of tribromoaniline obtained. =

Theoretical yield = 3.0 g.

OXIDATION

3. Preparation of Benzoic acid

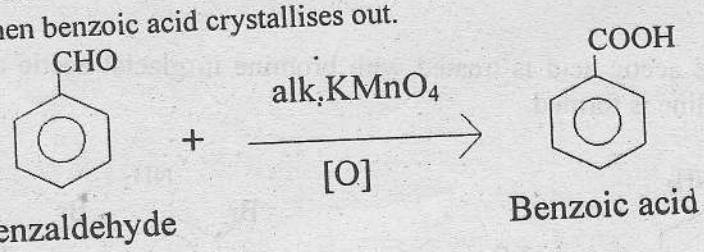
Ex No : Date :

Aim :

To prepare Benzoic acid from Benzaldehyde.

Principle:

Benzaldehyde is mixed with sodium carbonate solution and is oxidised with $KMnO_4$ solution. The sodium salt of benzoic acid formed is acidified with concentrated HCl acid when benzoic acid crystallises out.



Benzaldehyde Benzoic acid

Chemicals Required:

1. Benzaldehyde ..... 5 ml.
2. Anhydrous sodium carbonate.....5 g in 50 ml of water.
3. Potassium permanganate.....6 g in 100 ml of water.
4. Sodium sulphite ..... 8 g.

Procedure:

5 ml of benzaldehyde is added to a solution of 5 g of anhydrous sodium carbonate in 20 ml of water contained in an **R. B. flask.** It is fitted with a reflux **water condenser** and heated. 6 g of $KMnO_4$ in 100 ml of water is added little by little through the condenser until a permanent pink colour persists even after continuous boiling. It is boiled for about 30 minutes. The mixture is now transferred to a **beaker.** About 8 g of sodium sulphite are added. Then concentrated hydrochloric acid is added until the solution is acidic. The solution is cooled, precipitated benzoic acid filtered, washed and dried. The yield of benzoic acid is noted.

About one gram of the compound is recrystallised from boiling water.

Result :

The yield of benzoic acid =

Theoretical yield = 2 g.