LAB MANUAL

B. Sc. III

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AYODHYA

Synthesis of Acetanilide from Aniline

OBJECT: To synthesize Acetanilide from Aniline

Chemicals Required:

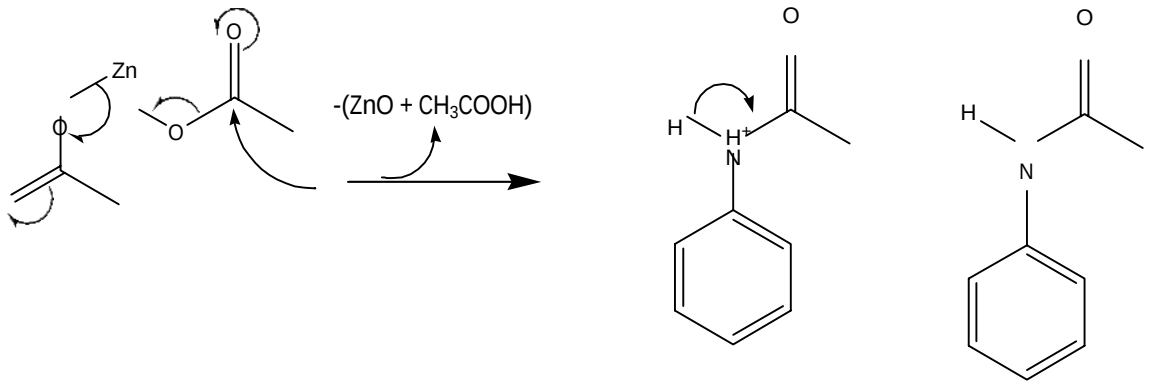
Aniline	- 5 ml
Glacial acetic acid	- 15 ml
Zinc dust	- 2 g

1.A mixture of aniline (5 ml) and zinc dust (2.0 g) in acetic acid (15 ml) in a 100 ml round bottom flask was heated over a gentle flame using water condenser. Heating was continued for about 2 hrs.
2.The reaction mixture was then carefully poured in cold water (100 ml) in a 250 ml beaker with cooling and vigorous stirring. The shining crystals of acetanilide were separated slowly.
3.After 15 min. the acetanilide crystals were collected by filtration. The solid crystals were washed over the Buchner funnel with water and the product was dried (yield, 10 gm). It

was crystallized in boiling water.

M.p. 114 ^{0}C.

Mechanism:



Synthesis of m- dinitrobenzene from m- nitrobenzene

OBJECT: To synthesize m- dinitrobenzene from nitrobenzene

Apparatus: Round bottom flask (RBF), Air condenser, beaker, suction pump.

Chemicals required:

Nitrobenzene - 7 ml

Fuming nitric acid - 9 ml

Conc H_2SO_4 -15ml

Procedure: 1. Prepare nitrating mixture by placing 9ml of fuming nitric acid in a clean dry Round bottom flask (RBF). To this carefully add, with shaking 15ml of concentrated H2SO4 and a fragment of porcelain.

2. To the nitrating mixture add 7ml nitrobenzene in very small lots with constant shaking. After Adding whole of nitrating mixture shake the Round bottom flask (RBF) for 10 minutes.

3. Fix the air condenser and heat the flask on boiling water bath for 1 hour. Shake the flask vigorously from time to time throughout this period of heating.

4. After heating allow the Round bottom flask (RBF) to cool to room temperature.

5.Finally pour this mixture carefully with vigorous stirring into a beaker containing crushed ice. The heavy oily dinitrobenzene will rapidly solidify.

Re-crystallization: Rectified spirit or alcohol.

Melting Point: 89-90^{0}C.

Synthesis of m-nitroaniline from m- dinitrobenzene

OBJECT: To synthesize m-nitroaniline from m-dinitrobenzene.

Apparatus: 500ml beaker sand bath burette, glass funnel.

Chemicals required: 5g m-dinitrobenzene, 2g-sulphur, 8 g-sodium sulphide,

Procedure: 1. Preparation of sodium disulphide: Add 2 g of finely powdered sulphur to a solution of 8 g of crystalline sodium sulphide in 60ml water. Boil the mixture gently for a few a minutes until a clear solution of sodium disulphide is obtained.

2. Heat 5g of pure m-dinitrobenzene in 150ml of water in a 500ml beaker on sand bath until the water boils gently.

3. Transfer the sodium disulphide solution into a burette and clamp the burette in position such that the end of the burette is immediately above the beaker.

4. Allow the sodium disulfide solution to fall drop by drop into boiling water at such a rate that the total addition takes 10-15minutes throughout this period keep the molten dinitrobenzene continuously dispersed as fine drops and not allowed to settle to the bottom.

5. When the addition of sodium disulphide is complete, boil the solution gently for a 20 minutes and quickly filer the solution using a hot water funnel.

6. A small quantity of elementary sulphur remains in the filter paper. The pale brown filtrate rapidly deposits yellow crystals of m-nitroaniline.

Re-crystallization: Hot water.

Melting point: 114^0C