Student Name/ ID_____

A] Synthesis of p.Nitroacetanilide

B] Elimination reaction (hydrolysis of p-nitroacetanilide)

BACKGROUND

Principle:

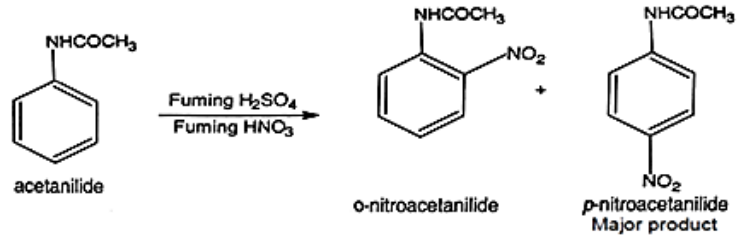
Here in first step electrophilic aromatic substitution (nitration) of nitronium ion is occurring towords para position of acetanilide more than ortho position due to steric reasons. Nitronium ion is the electrophile generates from fuming nitric acid in presence of fuming sulphuric acid. In the second step p-nitroaniline is prepared from p-nitro acetanilide due to hydrolysis of acetate ion from acetamido functional group in presence of concentrated sulphuric acid.[1]

Aim:

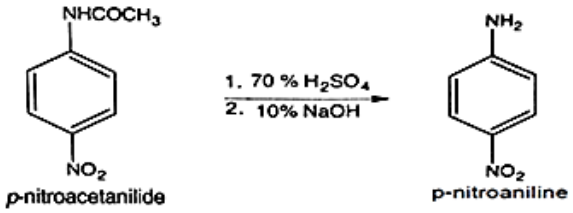
To prepare p-nitro aniline from acetanilide.

Reaction:

Step 1:

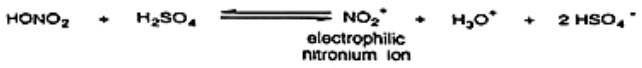


Step 2:



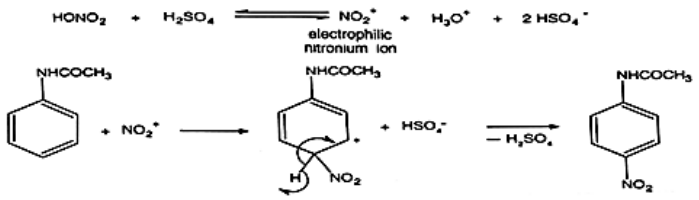
Mechanism:

Step 1:

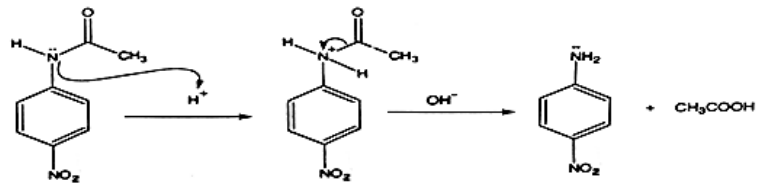


Mechanism:

Step 1:



Step 2:



Use:

It is an intermediate for dyes, pigents, pharmaceuticals (Paracetamol, Phenacetin etc.), pesticides and rubber chemicals.

Chemicals: Acetanilide

Glacial acetic acid

Conc. HNO_3

Conc. H_2SO_4

70% H_2SO_4 solution

10% NaOH solution

Apparatus: Conical flask

Beaker

Pipette

Glass rod

Buchner funnel

PROCEDURE

Step 1: Preparation of p-Nitroacetanilide from Acetanilide.

Add dry acetanilide (25 g) to glacial acetic acid (25 ml) in a beaker and then introduce concentrated sulphuric acid (50 ml) slowly with constant stirring to obtain clear solution. Place the beaker in a freezing mixture of ice and salt to cool the solution below 5 °C. Add a cold mixture of concentrated nitric acid (11 ml) and concentrated sulphuric acid (7 ml) drop wise with constant stirring to a reaction mixture while maintaining the temperature below 5 °C. After adding all the mixed acid, remove the beaker from the freezing mixture and keep it for 1 hr at room temperature. Pour the reaction mixture into an ice cold water (30 ml) to obtain the crude product of *p*-nitroacetanilide. Filter it on suction, wash with cold water till free from acid and recrystallize the pale yellow product from ethanol to get colourless crystalline solid, m.p. 214.5 °C.

Note:
o-Nitroacetanilide remains in the filtrate due to its high solubility in water.

Step 2: Preparation of p–Nitroaniline from p-Nitroacetanilide.

Place 30 g of p-nitroacetanilide and 150 ml of 70% H_2SO_4 (prepared by adding 100 ml conc. acid to 75 ml water carefully) in a round-bottomed flask. Reflux the mixture for 20-30 min. and pour the hot solution into 1000 ml of cold water. Neutralize with 10% NaOH, cool and filter the yellow crystalline product on a Buchner funnel. Wash it thoroughly with water. Recrystallize the product using a mixture of equal volume of rectified sprit and water or from hot water.[2]

Calculation:

Here limiting reagent is acetanilide; hence yield should be calculated from its amount.

Molecular formula of acetanilide = C_8H_9NO

Molecular formula of p-nitroaniline = $C_6H_6N_2O_2$

Molecular weight of acetanilide = 135 g/mole

Molecular weight of p-nitroaniline = 138 g/mole

Theoretical yield:

135 g acetanilide forms 138 g p-nitroaniline

Therefore, 25 g acetanilide will form …….? (X) g p-nitroaniline

X =(138 ×25)/135 = 25.55 g

Theoretical yield = 25.55 g

Practical yield = ————- g

% Yield = (Practical Yield)/(Theoretical Yield) × 100