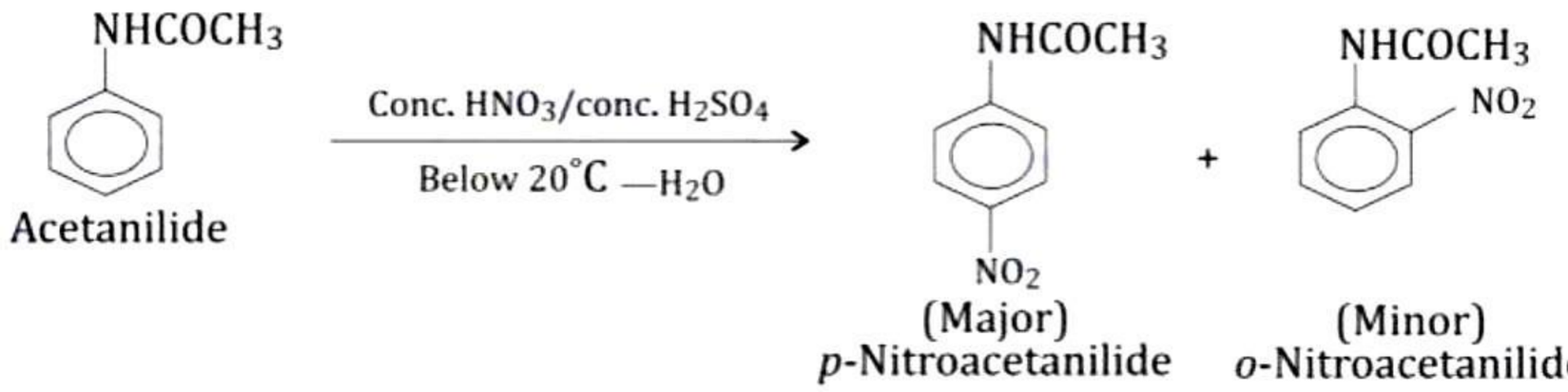
EXPERIMENT

Aim

To prepare a sample of p-Nitro acetanilide from acetanilide.

Theory

The nitration of aniline is difficult to carry out with nitrating mixture (a mixture of conc. H_2SO_4 and conc. HNO_3) since — NH_2 group gets oxidized which is not required. So, the amino group is first protected by acylation to form acetanilide which is then nitrated to give p-nitro acetanilide as a major product and o-nitro acetanilide as a minor product. Recrystallization from ethanol readily removes the more soluble ortho-compound and the pure p-nitro acetanilide is obtained. The chemical equation can be written as:



Material Required

Conical flask (100 ml), beaker (250 ml), measuring cylinder (100 ml), funnel, glass-rod, test-tube, filter-papers, etc.

Acetanilide = 5g

Glacial acetic acid = 5 ml

Conc. H_2SO_4 =10 ml

Fuming HNO_3 = 2 ml

Methylated spirit = 20 ml.

Procedure

1. Take a 100 ml conical flask and add 5 g of powdered acetanilide in it. Add 5 ml of glacial acetic acid and stir the mixture by the use of glass-rod.
2. Place 2 ml of fuming nitric acid in a clean test-tube and cool it in a freezing mixture (ice + salt) taken in a beaker. Carefully add drop by drop 2 ml of cone, sulphuric acid with constant shaking and cooling.
3. Add the remaining 8 ml of conc. H_2SO_4 drop by drop (with cooling under tap water) to the conical flask containing acetanilide and glacial acetic acid. Place the conical flask in a freezing mixture Fig.2. Stir the contents and wait until the temperature becomes less than 5°C.
4. To the cooled contents in the flask add nitrating mixture prepared in step (2) drop by drop with constant stirring. During addition temperature of the mixture should not rise above 10°C. This operation should take about 15 minutes Fig.3.

5. Remove the conical flask from the freezing mixture and allow it to stand for 30 minutes at room temperature.
6. Pour the contents of the flask on the crushed ice in a beaker. Stir it and filter the crude product. Wash thoroughly with cold water to remove acid.
7. Recrystallization of p-nitroacetanilide. Dissolve the crude product obtained above in about 20 ml of methylated spirit. Warm it to get a clear solution. Filter while hot and cool the filtrate in ice. o-Nitroacetanilide goes in the filtrate while p-nitroacetanilide is obtained as colourless crystals on the filter paper. Wash the solid on the filter paper with cold water. Dry the solid, weigh it and record its yield.

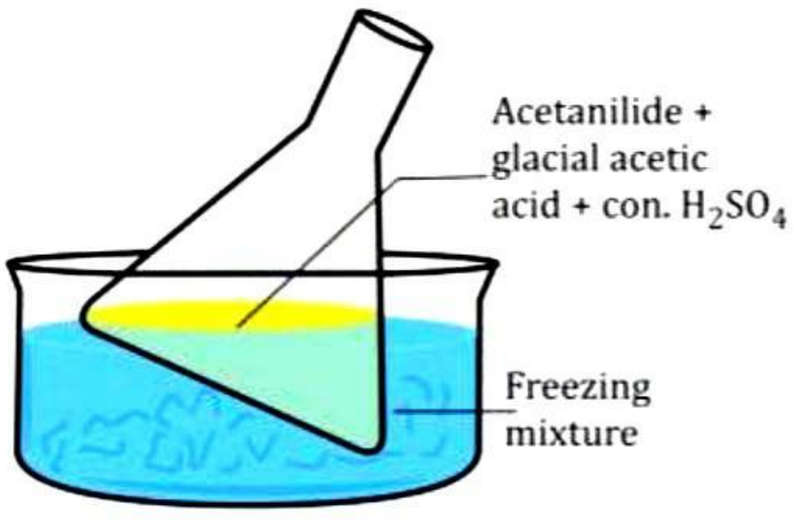


Fig.2. Flask kept in freezing mixture

Fig.3. Preparation of *p*-nitroacetanilide

Result

Weight of p-nitroacetanilide is obtained = ... g

Melting point of the compound is ..... °C

Note: Approximate expected yield is 4 g. The melting point of p-nitroacetanilide is 214°C.

Precautions

1. During addition of nitrating mixture, the temperature of the reaction mixture should not rise above 25°C.
2. Addition of fuming nitric acid should be done drop wise.
3. Do not inhale the vapours of nitric acid as they are very corrosive in nature. Addition of nitrating mixture may preferably be done in a fume-cupboard.

VIVA VOCE

Q 1. What is the chemical structure of acetanilide?

Ans. Acetanilide has the molecular formula C_8H_9NO and consists of an aromatic ring with an acetamide functional group.

Q 2. Why are acetanilide chosen as the starting material for synthesizing p-Nitro acetanilide?

Ans. Acetanilide is chosen due to its availability and the ease with which the amino group on the aromatic ring can be substituted during nitration.

Q 3. Explain the importance of nitration in organic synthesis.

Ans. Nitration introduces a nitro group (NO_2) into an organic compound, providing a way to modify its chemical properties and functionalities.

Q 4. What is the purpose of preparing p-Nitro acetanilide from acetanilide?

Ans. The purpose is to demonstrate the synthesis of a substituted aromatic compound through electrophilic aromatic substitution, a fundamental reaction in organic chemistry. p-Nitro acetanilide is synthesized from acetanilide by introducing a nitro group ($-NO_2$) at the para position of the aromatic ring.

Q 5. Can you outline the reaction mechanism involved in the synthesis of p-Nitro acetanilide from acetanilide?

Ans. The reaction mechanism involves the electrophilic substitution of the aromatic ring in acetanilide by the nitronium ion (NO_2^+), which is generated in situ from the reaction of nitric acid with sulfuric acid. The nitro group ($-NO_2$) is introduced at the para position of the aromatic ring.

Q 6. How would you purify the synthesized p-Nitro acetanilide?

Ans. The synthesized product can be purified by recrystallization. The crude product is dissolved in a suitable solvent at an elevated temperature, and the solution is then allowed to cool slowly, leading to the formation of pure crystals of p-Nitro acetanilide.

Q 7. What analytical techniques can be used to confirm the identity of the synthesized p-Nitro acetanilide?

Ans. Analytical techniques such as melting point determination, infrared spectroscopy (IR), and nuclear magnetic resonance spectroscopy (NMR) can be used to confirm the identity of the synthesized p-Nitro acetanilide by comparing its properties and spectra with those of authentic samples.