Synthesis of p-NO₂ Acetanilide

Reaction Equation:

$$\begin{array}{c} O \\ HN-C-CH_3 \\ \hline \\ HNO_3 \\ \hline \\ H_2SO_4 \\ \end{array}$$

Cautions: This reaction is an exothermic reaction. It may occur an explosion if the formation of polynitro derivatives and temperature of the reaction medium is not controlled.

Requirements:

Acetanilide

Acetic Acid

Nitric Acid

Sulfuric Acid

NaHCO₃ (%10)

EtOH

Procedure:

- Place the thermometer and dropping funnel in a 250 ml two-neck balloon. Add 15 ml acetic acid and 1 g of precisely weighed acetanilide.
- Add 25 ml of concentrated sulphuric acid with stirring.
- Cool the clear reaction medium in 3 parts of ice + 1 part of NaCl bath.
- Prepare a mixture of 5 ml HNO₃ and 3 ml H₂SO₄.
- Add the sulfonitric mixture slowly with stirring after the temperature of the reaction medium has decreased to 0-2 °C. Maintain the temperature below 10 °C during the addition.
- After all of the nitrification reagent has been added, remove the balloon containing the reaction mixture from the freezing mixture and allow it to stand at room temperature for half an hour.
- Pour the reaction mixture into 250 g of crushed ice contained in a beaker with stirring.
- Allow it to stand for 5-10 minutes.
- Filter it through Buchner funnel and allow it to dry.
- Wash it with distilled water then 40 ml of NaHCO₃ (%10) and again with water.

Melting point and yield: 215-217 °C, %50-55