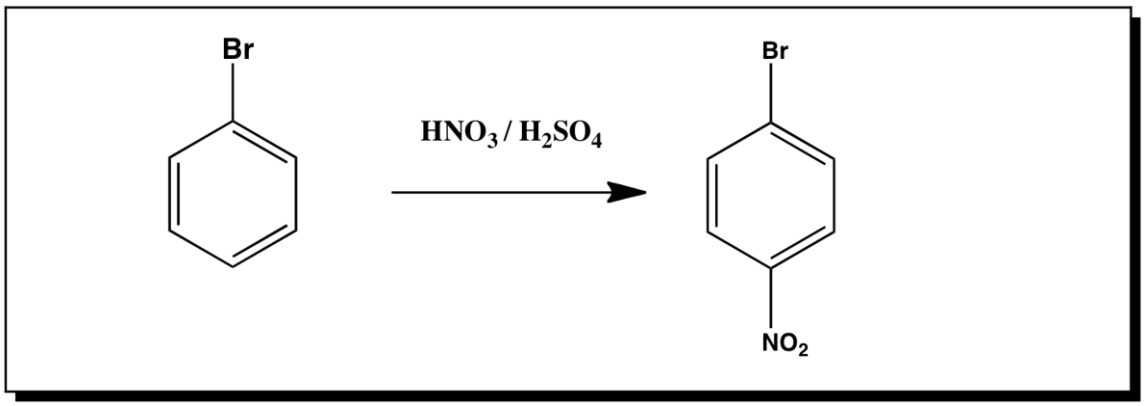
Exp. 5 Nitration of Bromobenzene



Techniques

Ice bath cooling, recrystallization, thin layer chromatography (TLC)

Equipment

Dropping funnel, vacuum filtration set-up, magnetic stirrer with oil bath and thermometer.

Chemicals

Bromobenzene, nitric acid, sulfuric acid, ethanol, hexane/ethylacetate eluent

Safety

Conc. nitric and sulfuric acid and especially the mixture of both are extremely corrosive liquids which have to be handled with special care and should not come in contact with skin or clothes! Spills must be diluted with water before wiping up. Paper towels are burned to char.

Procedure:

• 10 ml of conc. sulfuric acid are filled into a 100ml double-necked flask equipped with the joint thermometer and stir bar and cooled down in ice.

• Then carefully add 10 ml of the nitric acid slowly with stirring and let the mixture come to room temperature.

• The flask is fitted with a dropping funnel and 10 ml of bromobenzene are added slowly in small portions over the course of 10 min.

• Maintain the temperature around 60°C (a cold water bath should be prepared).

• When the addition is complete, heat the mixture for 30min to 60°C.

• Let the reaction mixture cool down and pour it into a beaker with ~100 ml of stirred ice water.

• Vacuum filtrate the crude nitration product and wash it thoroughly with water to remove most of the acid.

• When the product is almost dry, transfer it into a round-bottom flask and recrystallize it from ethanol.

• Vacuum filtrate the crystals and wash them with some ice-cold ethanol. Keep the filtration liquid for TLC.

• Develop a TLC of your purified product and compare it with the mother liquor. (hexane/ethylacetate 9:1 as eluent)

Tasks

1. Determine the yield of the air-dried crystals.
2. Determine the melting point of the purified product.
3. Visualize the side products by TLC

Melting point of 4-Nitro-bromobenzene = 124-126°C

Protocol **Exp. 5** **Name/Date:**

Aim:

Apparatus and Materials:

Theory and Mechanism:

Procedure and Observations: