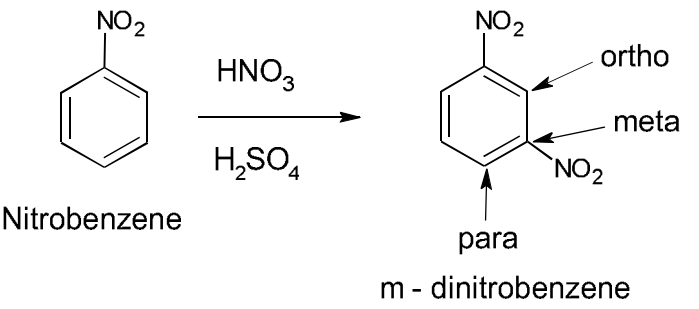
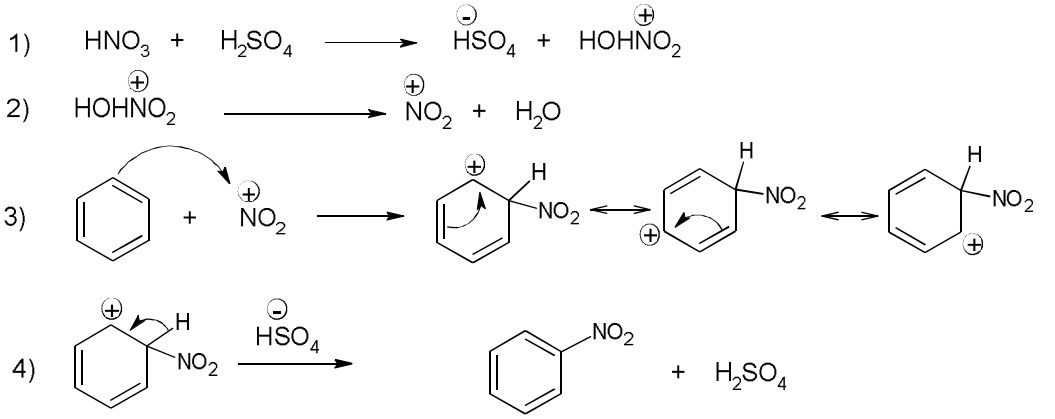


The nitration mechanism of benzene :



Chemical and Apparatus

Benzene, conc.HNO_3, conc.H_2SO_4, cold distilled water, ethanol, ice path, Round bottom, condenser, funnel, electric heater, beaker, balance, filter paper, oven,

Safety Note

Caution: Avoid contact with the acids used in this experiment and the reaction product. Prevent contact with the skin, eyes, and clothing; work in the hood. An acid spill is neutralized using solid sodium carbonate or bicarbonate. The reaction is highly exothermic.

Concentrated nitric acid and concentrated sulfuric acid are both strong oxidizers, and strongly corrosive--wear gloves while handling them, and avoid breathing their vapors.

Experimental No. (10) Nitration of benzene

Procedure of Experimental

1- In hood, prepare a mixture of (4 mL) conc. HNO_3 and (4 mL) conc. H_2SO_4 in a beaker. Cool beaker to room temperature (30^0C) by means of a ice bath, , remove the beaker from the ice bath, wipe off the outside of the conical flask, and clamp it to hotplate .

2- To the conical flask, add (0.5 mL) benzene **DROPWISE** over a period of about 5-10 min; gently swirling the tube to mix the contents after each addition. Keep the reaction mixture between 50 - 55 °C. **DO NOT ALLOW THE REACTION MIXTURE TO EXCEED 60 °C.**

3- After the addition is completed and the exothermic reaction has subsided, put the mixture in the round bottom with condenser and heat the round for (45 min) in a hot water bath, maintaining the temperature in the reaction below 90 °C during this period.

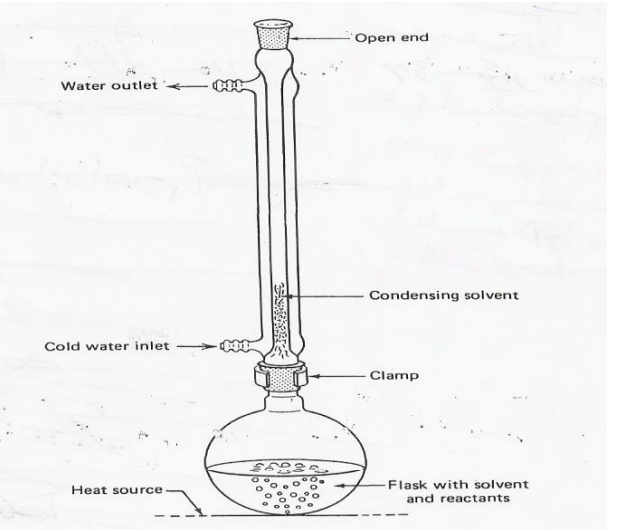


Figure show the reflux of reaction mixture

4- Cool the conical flask in an ice bath to room temperature.

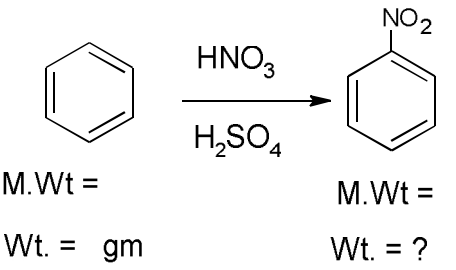
5- Pour the reaction mixture into 100 mL of distilled water which is in a 150 mL beaker.

6- Isolate the crude product by filtration.

7- Wash the filter cake thoroughly with cold (0-10°C) distilled water and dry the filter in the oven.

8- The crude product may be purified by recrystallization by hot ethanol, then determine the weight, melting point, and percentage yield.

Calculation



$$\text{Theoritical weight of nitobenzene} = \frac{\text{Wt of benzene} \times \text{M.Wt of nitrobenzene}}{\text{M.Wt of benzene}}$$

Experimental weight = weight of product with filter paper – weight of filter paper of nitrobenzene

$$\% \text{ of nitrobenzene} = \frac{\textbf{Experimental weight}}{\textbf{Theoritical weight}} \times 100$$

Questions for discussion

1. Which is nitrated faster? toluene or nitrobenzene, toluene or phenol ? Explain.
2. List three combinations of reagents used for nitration of aromatic compounds?
3. Why is concentrated sulfuric acid employed in this reaction? What is the electrophile that is produced by the reaction of sulfuric acid and nitric acid/
4. Why is it important to maintain the reaction temperature low and the addition of nitric acid-sulfuric acid mixture carried out slowly?
5. Explain why concentrated H_2SO_4, not concentrated HCl, was used in this experiment?