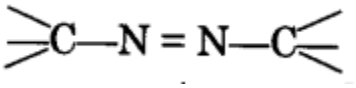
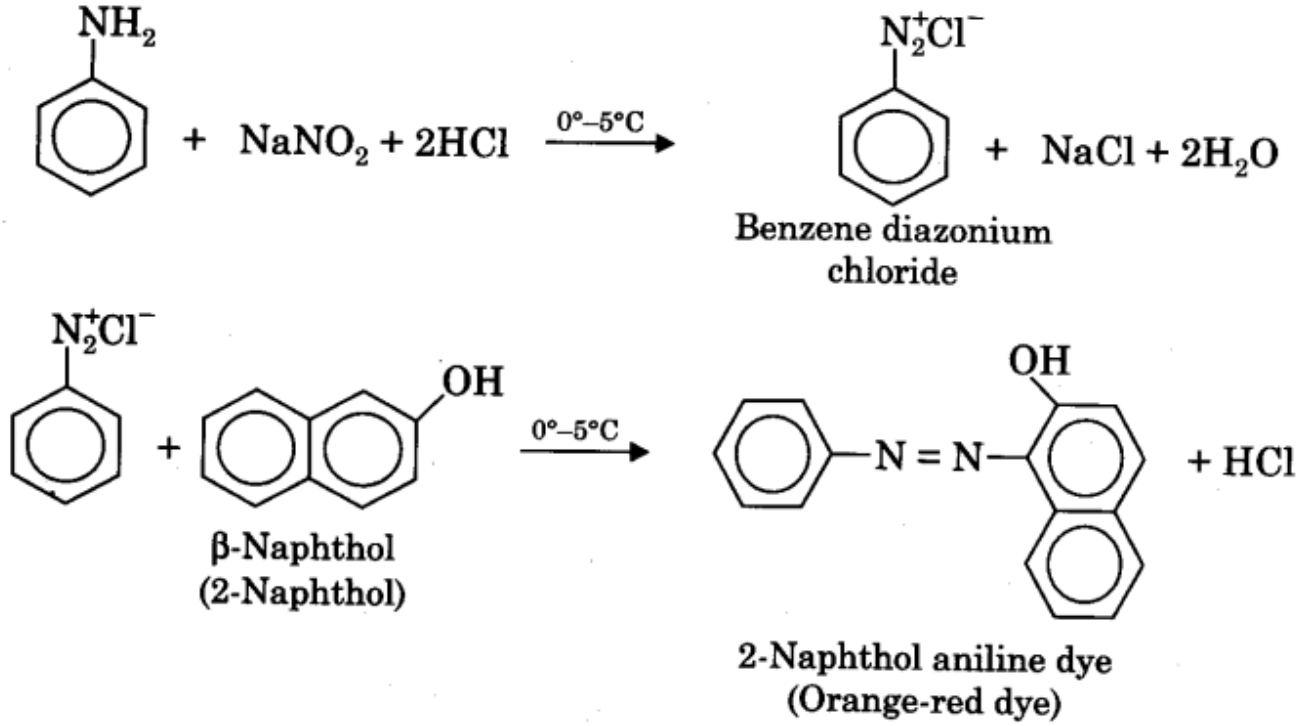
To Prepare 2-Naphthol Aniline Or Phenyl-azo-β-Naphtholdye

Theory

2-Naphthol aniline dye or Phenyl-azo-β-naphthol is an orange-red dye. It belongs to a large class of azo-compounds, all of which contain the characteristic grouping



Azo compounds are all coloured compounds. For the preparation of this dye, aniline is diazotised and then diazonium salt thus obtained is subjected to coupling reaction with 2-naphthol.



Apparatus

One 100 mL conical flask, one 100 mL beaker, one 250 mL beaker, ice-bath, glass-rod, Buchner funnel, water pump.

Chemicals Required

Aniline = 4.5 ml
Sodium nitrite = 4g
2-Naphthol = 7 g

Cone, hydrochloric acid = 10 ml
Glacial acetic acid = 40 ml

Procedure

1. Take a 100 ml conical flask and add 4.5 ml of aniline, 10 ml of cone. HCl and 20 ml of water. Cool this solution to 5°C by placing the conical flask in a trough containing ice- cold water.
2. In a 100 ml beaker dissolve 4 g of sodium nitrite in 20 ml of water and cool this solution also to 5°C.
3. Now slowly add sodium nitrite solution to the solution of aniline in cone. HCl.
4. Dissolve 7.0 g of 2-naphthol in 60 ml of 10% NaOH solution taken in a 250 ml beaker and cool this solution to 5°C by placing in an ice bath. Some crushed ice may be added directly to fecilitate cooling.
5. Now add the diazotised solution very slowly to the naphthol solution with constant stirring. The mixed solutions immediately develop a red colour and the phenyl-azo-β- naphthol rapidly separates as orange-red crystals.
6. When the addition of diazo solution is complete, allow the mixture to stand in ice-salt mixture for 30 minutes, with occasional stirring. Filter the solution through a buchner funnel under suction from the pump. Wash the phenyl-azo-β-naphthol with water and dry the crystals obtained by pressing between the folds of filter paper.
7. Recrystallise the product from glacial acetic acid. Filter the crystals obtained at the pump. Wash with a few ml of ethanol to remove acetic acid. Phenyl-azo-β-naphthol is obtained as orange-red crystals. Expected yield is 3 g and melting point is 133°C.

Result

Weight of phenyl-azo-β-naphthol obtained as orange-red crystals = …….g.
Melting point of phenyl-azo-β-naphthol is…….°C.

Precautions

1. The solution of the aniline hydrochloride should be cooled to 5°C, and this temperature should be maintained throughout the addition of the sodium nitrite solution.
2. Addition of sodium nitrite should be very slow because the reaction is exothermic and may cause the temperature to rise.
3. Always add diazonium chloride solution to β-naphthol solution for dye formation and not vice-versa.