

Experiment

Aim: To Prepare and Submit 1-Phenylazo-2-Naphthol from Aniline by diazotization and coupling reactions.

Reference:

1. Kar A. Advanced Practical Medicinal Chemistry, New Age International (P) Limited Publication, New Delhi, Page No. – 136.
- 2.

Requirement:

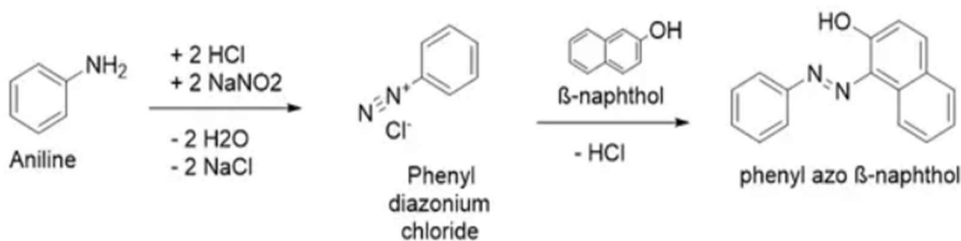
Apparatus: Conical flask, Beaker, Funnel, Filter paper, measuring cylinder, etc.

Chemicals: Aniline – 4.0 g, conc. Hydrochloric acid – 12.8 ml, β -Naphthol -6.24 g, 10% (w/v) Sodium hydroxide solution – 40 ml, Sodium nitrite – 3.2 g

Principle:

Phenyl diazonium chloride is obtained first by the diazotization of aniline with nitrous acid, as explained earlier, which, on coupling with β -naphthol in the presence of NaOH solution, yields the desired coupled product phenyl-azo- β -naphthol. A mole of HCl is eliminated, instantly reacting with NaOH from the medium to produce NaCl and H₂O. Importantly, both diazotization and coupling reactions must be carried out between 0-5°C.

Reaction:



Use: 1-Phenylazo-2-naphthol, also known as **Sudan I**, was historically used as a red dye for textiles and as a food coloring agent. However, its use in food has been restricted in many countries due to concerns about its carcinogenic properties.

Procedure:

1. Weigh 4.0 g (3.92 ml) of aniline and dissolve it in 12.8 ml of concentrated hydrochloric acid (conc. HCl) in a 250 ml beaker. Dilute the aniline solution with 12.8 ml of distilled water.
2. Place the beaker containing the aniline solution in an ice bath and cool it with frequent stirring until the temperature falls below 5°C.
3. Dissolve 3.2 g of sodium nitrite in 15 ml of water in another beaker. Chill the sodium nitrite solution in an ice bath (0–5°C).
4. At intervals, Add the sodium nitrite solution to the aniline solution in small lots (2 ml). Stir vigorously with a glass rod, ensuring the temperature remains below 5°C throughout the addition.
5. After complete addition, test the reaction mixture for the presence of free nitrite by placing a drop on KI-starch paper, which should turn distinctly blue in the presence of free nitrous acid.
6. Dissolve 6.24 g of β -naphthol in a 250 ml beaker containing 40 ml sodium hydroxide solution. Cool the β -naphthol solution in an ice bath (0–5°C).
7. Slowly add the cold diazonium salt solution to the β -naphthol solution with vigorous stirring. Take special care to prevent the temperature of the reaction mixture from rising beyond 5°C. Add crushed ice if necessary.
8. A red color should develop, and crystals of crude phenylazo- β -naphthol should separate. Allow the reaction mixture to stand for 30-40 minutes with stirring to complete the reaction.
9. Filter the red product in a Büchner funnel using a suction pump. Wash the filtered product with ice-cold water and dry it.

Calculation:

The molecular formula of Aniline = $C_6H_5NH_2$

The molecular weight of aniline = 93g/mole

The molecular formula of phenyl azo- β -naphthol = $C_{16}H_{12}N_2O$

The molecular weight of = 248g/Mole

93 g of aniline on reacting with 144 g of β -naphthol yields phenyl azo- β -naphthol =
248 g

.....

<https://www.pharmacareerinsider.com>

.....

.....

Result:

The 1-Phenylazo-2-Naphthol was synthesized, and the percentage yield was found to be...%.

<https://www.pharmacareerinsider.com/>