Experiment: 08

AIM: To prepare and submit benzotriazole from o-phenylenediamine and calculate its percentage yield.

REFERENCES:

1. Vogel's Textbook of Practical Organic Chemistry by Brian S. Furniss, Antony J. Hannaford, Peter W. G. Smith & Austin R. Tatchell; Fifth Edition; Page No.1163.
2. Practical in organic chemistry, by Hitesh G. Raval, Sunil L. Baldania and Dimal A. Shah, Nirav Prakashan, Page No. 303.

REQUIREMENTS:

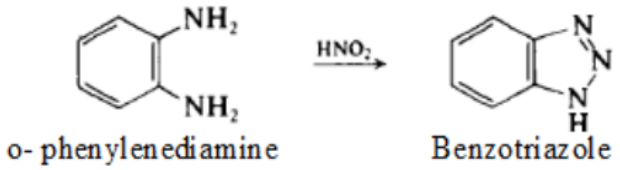
Chemicals: o-phenylenediamine**,** Glacial acetic acid**,** and Sodium nitrite.

Apparatus: Beaker**,** Buchner funnel**,** Measuring cylinder**,** Filter paper, etc.

Principle:

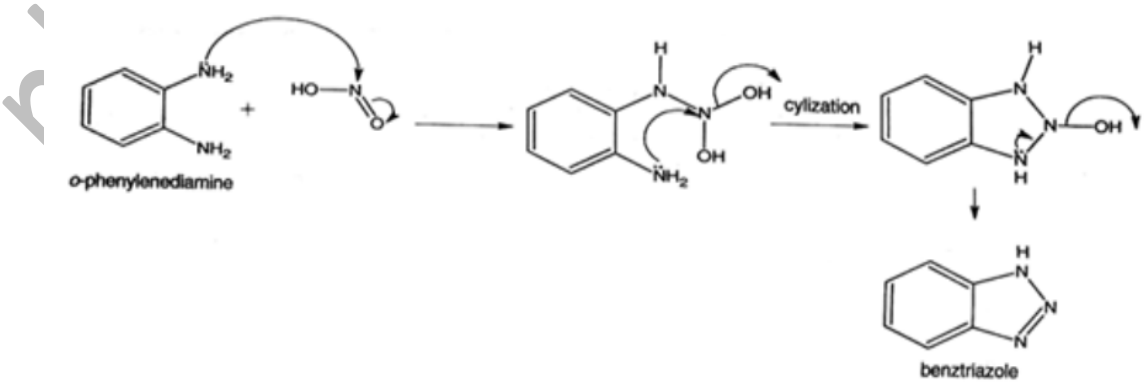
Benzotriazole can be prepared by treating o-phenylenediamine with nitrous acid (liberated during the reaction between sodium nitrite and acetic acid) to form mono-diazonium salt that follows a spontaneous intramolecular cyclization reaction to produce benzotriazole.

Chemical Reaction:



o- phenylenediamine Benzotriazole

Mechanism:



Use:

Used as antifungal, antihypertensive, analgesic etc.

PROCEDURE

1. In a 250-ml beaker, dissolve 10.8 g (0.1 mol) of o-phenylenediamine in a mixture of 12 g (11.5 ml, 0.2 mol) of glacial acetic acid and 30 ml of water. Slight warming may be necessary.
2. Cool the clear solution to 15 °C and stir magnetically.
3. In one portion, add 7.5 g (0.11 mol) of sodium nitrite to 15 ml of water. The mixture will warm up to about 85 °C within 2-3 minutes and then cool down, changing color from deep red to pale brown.
4. Continue stirring for 15 minutes, during which the temperature will drop to 35-40 °C. Chill the mixture thoroughly in an ice-water bath for 30 minutes.
5. Collect the pale brown solid product by vacuum filtration and wash the product with three 30 ml of ice-cold water.

Recrystallization:

1. Dissolve the solid in approximately 130 ml of boiling water and add decolorizing charcoal to the solution.
2. Filter the mixture and allow the filtrate to cool to about 50 °C.
3. Add a few crystals of the synthesized product (benzotriazole) retained for seeding.
4. Let the mixture slowly reach room temperature to avoid separating the material as an oil. Thoroughly chill the mixture in ice.
5. Collect the benzotriazole, which separates as pale straw-colored needles with a melting point of 99-100 °C.

Calculation

Here, the limiting reagent is o-phenylenediamine; yield should be calculated from the amount taken.

$C_6H_8N_2$ = Molecular formula of o-phenylenediamine

$C_6H_5N_3$ = Molecular formula of benzotriazole

Molecular weight of o-phenylenediamine = 108 g/mole

Molecular weight of benzotriazole = 119 g/mole

Theoretical yield:

108 g o-phenylenediamine forms 119 g benzotriazole

Therefore, 10.8 g o-phenylenediamine will form ……? (X) g benzotriazole

$X = (119 \times 10.8)/108 = 11.9$ g

Theoretical yield = 11.9 g

Practical yield = ————————- g

% Yield = (Practical Yield)/(Theoretical Yield) × 100

Result:

Benzotriazole was synthesized, and the percentage yield was found to be……..%.