

## 2. Experiment name: Preparation of Benzotriazole

**Aim:** To prepare benzotriazole from o-phenylenediamine.

**References:** <https://labmonk.com/synthesis-of-benzotriazole>

### Requirements:

Chemicals: o-phenylenediamine, glacial acetic acid, sodium nitrite

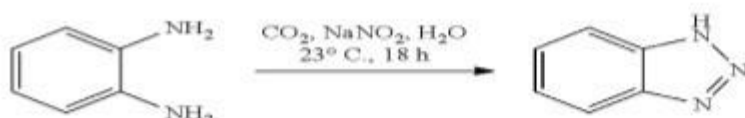
Apparatus: Beaker, Buchner funnel, measuring cylinder, filter paper.

**Procedure:** Dissolve 10.8 g of o-phenylenediamine in a mixture of 12 g of glacial acetic acid and 30 ml of water contained in a 250 ml beaker; slight warming may be necessary. Cool the clear solution to 15 °C, stir magnetically and then add a solution of 7.5 g of sodium nitrite in 15 ml of water in one portion. The mixture gets warm and reaches a temperature of about 85 °C within 2-3 min and then become cool while the colour of the mixture changes from deep red to pale brown. Continue stirring for 15 min, by which the temperature will have dropped to 35-40 °C, and then thoroughly chill in an ice-water bath for 30 min. Collect the product by vacuum filtration of the pale brown solid which separates and wash with three 30 ml portions of ice-cold water.

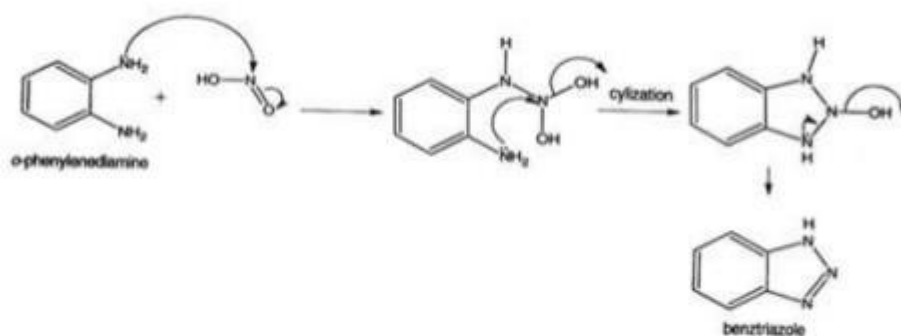
**Recrystallization:** Dissolve the solid in about 130 ml of boiling water, add decolourising charcoal, filter and allow the filtrate to cool about 50 °C before adding a few crystals of the synthesized product (benzotriazole) which have been retained for seeding. Allow the mixture to retain room temperature slowly (to avoid the separation of material as an oil) and then thoroughly chill in ice and collect the benzotriazole which separates as pale straw-coloured needles, m.p. 99-100 °C. A second crop may be obtained by concentrating the filtrate. The yield is about 8 g. The benzotriazole crystallises much more readily from benzene (55 ml) but the material is still slightly coloured. A pure white product can be obtained by sublimation at 90-95 °C at 0.2 mmHg.

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

**Reaction:**



**Mechanism:**



**Conclusion:** Benzotriazole was synthesized and the percentage yield was found to be \_\_\_\_\_%