I. Preparation of drugs/intermediates

1. Experiment name: Preparation of Benzimidazole.

Aim: To synthesize Benzimidazole from o-phenylenediamine.

References:https://labmonk.com/synthesis-Benzimidazole-from-o-phenylenediamine.

Requirements:

Chemicals: o-phenylenediamine, formic acid, sodium hydroxide.

Apparatus: Beaker, Round bottom flask, Buchner funnel, measuring cylinder, filter paper.

Procedure:

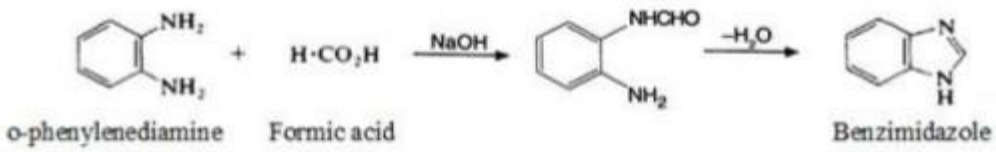
Dissolve 27gm of o-phenylenediamine in a round bottomed flask of 250ml and add 17.5gm of formic acid. Heat the mixture on a water bath at 100◦c for 2hrs. Cool and add 10% sodium hydroxide solution slowely, with constant rotation of the flask, until the mixture is just alkaline to litmus. Filter off the synthesized crude benzimidazole by using the pump, wash with ice cold water, drain well and wash again with 25ml of cold water.

Recrystallization with dissolve the synthesized product in 400ml of boiling water, add 2gm of decolorizing carbon and digest for 15min. filter rapidly through a preheated Buchner funnel and a flask at the pump. Cool the filtrate to about 10◦c, filter off the benzimidazole, wash with 25ml of cold water and dry at 100◦c. The yield of pure benzimidazole, m.p. 171-172◦c is 25gm.

Principle:

The two carbon-nitrogen bonds in benzimidazole when disconnected give o-phenylenediamine and formic acid. Therefore, synthesis of benzimidazole is affected by simply heating the o-phenylenediamine and formic acid together (condensation type of reaction)

Reaction:



o-phenylenediamine Formic acid Benzimidazole