

**AIM:** To prepare 1,3-diphenyl pyrazole from diphenyl hydrazone and vicinal diol.

**REFERENCES:**

1. N. Panda, A. K. Jena, J. Org. Chem., 2012, 77, 9401-9406. 2. Practical Heterocyclic Chemistry by A. O. Fitton and R. K. Smalley Academic Press London and New York, Page. 25.

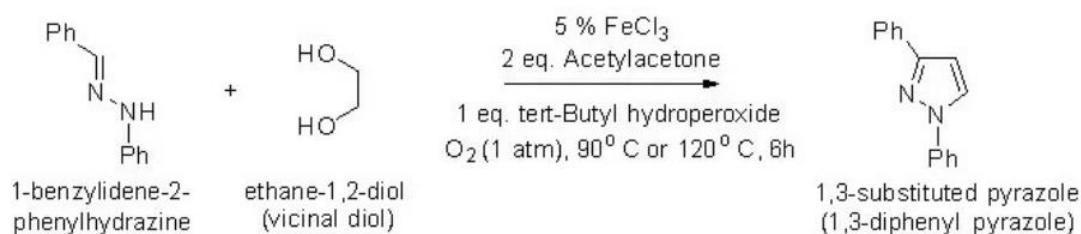
**REQUIREMENTS:**

**Chemicals:** 1-benzylidene-2-phenyl hydrazine, Ethane-1,2-diol(ethylene glycol), Ferric chloride, tertbutyl hydroperoxide, Acetyl acetone, Sodium chloride, Ethyl acetate, Sodium sulfate.

**Apparatus:** Water bath, Beaker, Measuring cylinder, Thermometer, Stirrer, Separatory funnel, Buchner funnel, etc.

**PRINCIPLE:** 1,3-substituted pyrazole is prepared by cyclization of diarylhydrazone and vicinal diol in presence of ferric chloride and tert-butyl hydroperoxide (TBHP) which is also called regioselective synthesis of substituted pyrazole.

**Reaction:**



**PROCEDURE:** About 4.55 g of 1-benzylidene-2-phenyl hydrazine is dissolved in the solution of 25 ml of vicinal diol and ferric chloride (5 mol %). Then another solution of tert-butyl hydroperoxide (5.3 g) in 25 ml of acetyl-acetone is added to it. Mix solution is kept maintained at a temperature range of 90 to 100° C. The mixed solution is left to reach room temperature and stirred for 6 hours. Content is poured into water and extracted with ethyl acetate three times. The combined organic solution is washed with water, then with a saturated solution of sodium chloride, passed through sodium sulphate and evaporated under vacuum. About 3.15 g of the final product is found with m.p: 185°C.

**Calculation of yield:**

– Molecular Formula of 1-benzylidene-2-phenyl hydrazine = C<sub>13</sub>H<sub>12</sub>N<sub>2</sub>

– Molecular Formula of 1,3-diphenyl pyrazole = C<sub>15</sub>H<sub>12</sub>N<sub>2</sub>

– Molecular weight (MW) of 1-benzylidene-2-phenyl hydrazine = 196 g/ mol

– MW of 1,3-diphenyl pyrazole = 220 g/ mol

196 g of 1-benzylidene-2-phenyl hydrazine yields 1,3-diphenyl pyrazole = 220 g

4.55 g of 1-benzylidene-2-phenyl hydrazine shall yield 1,3-diphenyl pyrazole =  $(220 / 196) \times 4.55 = 5.1$  g Therefore,

Theoretical yield of 1,3-diphenyl pyrazole = 5.1 g

If reported Practical yield = ..... g Then, Percentage Practical yield =  $(\text{Practical yield} / \text{Theoretical yield}) \times 100$

**RESULT:** The percent yield of 1,3-diphenyl pyrazole is ..... % with m.p. ....°C.