

Aim: To prepare and submit 1,3-diphenyl pyrazole from diphenyl hydrazone and a vicinal diol.

References:

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, Tert-butyl hydroperoxide, Acetyl acetone, Sodium chloride, Ethyl acetate, Sodium sulphate.

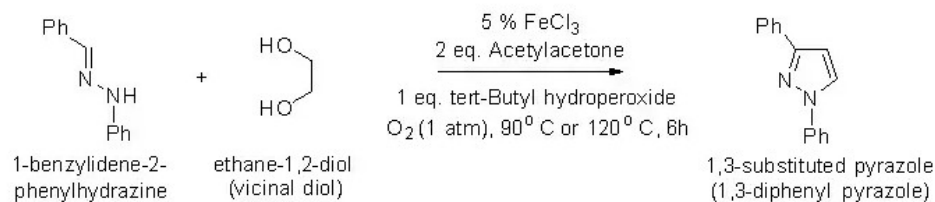
Apparatus:

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 the presence of ferric chloride and tert-butylhydroperoxide (TBHP) which is also called the regioselective synthesis of substituted pyrazole.

Reaction:



Use:

Can be used as an antibacterial and antiviral agent.

Procedure:

About 4.55 g of 1-benzylidene-2-phenyl hydrazine is dissolved in 25 ml of vicinal diol and ferric chloride (5 mol%). Then, another solution of tert-butyl hydroperoxide (5.3 g) in 25 mL of acetylacetone is added. The mixed solution is 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 sulfate, and evaporated under a vacuum. About 3.15 g of the final product is found with m.p. 185 °C.

Calculation

– Molecular Formula of 1-benzylidene-2-phenyl hydrazine = $C_{13}H_{12}N_2$

– MF of 1,3-diphenyl pyrazole = $C_{15}H_{12}N_2$

– Molecular weight 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 = $(\text{Practical yield} / \text{Theoretical yield}) \times 100$
 $= (\dots / 5.1) \times 100 = \dots\%$

Result:

The percent yield of 1,3-diphenyl pyrazole is 61.75 % with m.p. 185 °C.