

## SYNTHESIS AND CHARACTERIZATION OF SOME PYRAZOLINE DERIVATIVES

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**Abstract:** In present study, the synthesis of pyrazoline derivatives by the reaction of chalcones with phenyl hydrazine hydrochlorides in ethanol under reflux conditions in good yields. All the prepared compound were characterized by spectral studies.

**Keywords:** pyrazoline, chalcone, phenyl hydrazine, spectral analysis

### INTRODUCTION

The prevalence of pyrazoline core in biologically active molecules has stimulated the need for an efficient way to make the heterocyclic lead. Pyrazoline has attracted attention and medicinal chemists for both with regard to heterocyclic chemistry and the pharmacological activities associated with them. The chalcones are most commonly synthesized via Claisen-Schmidt reaction of an aromatic aldehyde with acetophenones, which undergo cyclization reaction with phenyl hydrazine hydrochlorides in ethanol to synthesize pyrazoline derivatives. Pyrazoline is an important class of heterocyclic compounds containing two nitrogen atoms in the five-membered ring and are reported to have anti-inflammatory<sup>1,2</sup>, antibacterial<sup>3</sup>, antioxidant<sup>4</sup>, antimicrobial<sup>5-6</sup>. Pyrazoline products nitrogen heterocycles consider rich electron (neutrophil) which help to show a significant part in the various biological activities<sup>7-8</sup>, anticancer activity<sup>9-10</sup>, Antimalarial activity<sup>11</sup>. A popular method for synthesizing these chemicals involves the reaction of aromatic ketones and aldehydes in the presence of base. These compounds in this process and can be converted into 2-pyrazolines by utilizing cyclizing agent such as acetic acid<sup>12</sup>. Due to these properties, pyrazolines have garnered interest in medicinal chemistry for drug development.

### EXPERIMENTAL

#### Materials and methods

All the reagents and solvent used were of laboratory grade. The synthesis of new products was monitored by TLC. Melting points were determined by Thiele tube method. The IR spectra were recorded on a Shimadzu instrument using KBr pellets. <sup>1</sup>H NMR spectra were recorded on Bruker Avance 400 MHz spectrophotometer using DMSO-d<sub>6</sub> as solvent and TMS as internal standard (chemical shift are expressed in ppm).

#### Synthesis of ligands

The compounds were synthesized in two steps:

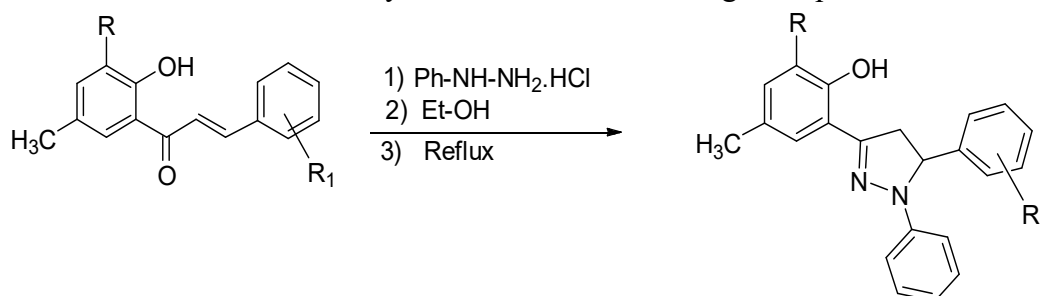
##### Step I: Synthesis of Chalcone

A mixture of 0.01 mol 2-hydroxy-3-nitro-5-methyl acetophenone and 0.01 chloro-

substituted aldehyde and indole-3-carboxaldehyde were taken in ethanol. 20% NaOH solution added slowly and stirred at room temperature for 4-5h. The mixture was kept overnight and then it was poured into crushed ice and acidified with dilute hydrochloric acid. It was filtered, dried in hot air oven and weighed. The crude product was further purified using ethanol.

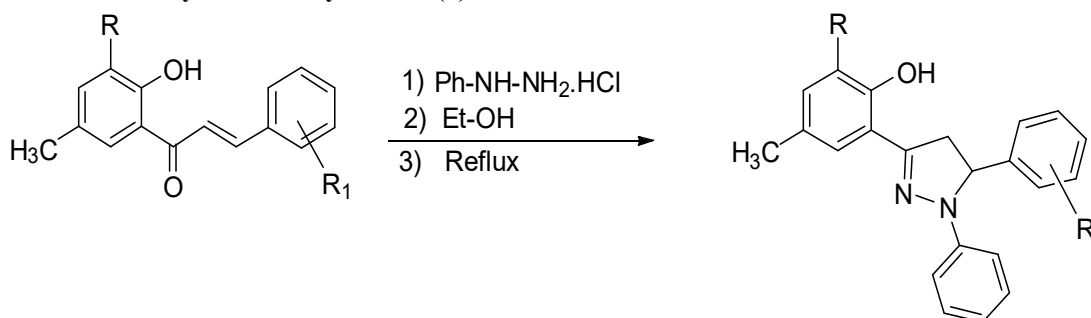
## Step II: Synthesis of 2-pyrazoline derivatives

A solution of synthesized chalcone (0.01mol) and Phenyl Hydrazine Hydrochloride (0.02mol) were dissolved in ethanol (25ml). The reaction mix. was refluxed for 1.5hrs. The reaction mix. Was cooled and decomposed it into ice-cold water. The product precipitates were refined, laundered with distilled water and re-crystallized from ethanol to get the product.



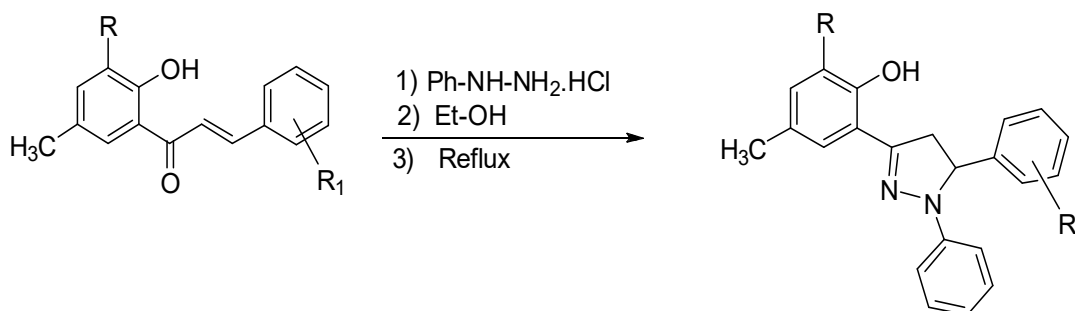
Ligand R R<sub>1</sub>  
a -NO<sub>2</sub>-2-Cl

Scheme 1: Synthesis of Pyrazoline (a)



Ligand R R<sub>1</sub>  
b -NO<sub>2</sub> 3-Cl

Scheme 2: Synthesis of Pyrazoline (b)



Ligand R R<sub>1</sub>  
c -NO<sub>2</sub>Indole-3-carboxy aldehyde  
Scheme 3: Synthesis of Pyrazoline (c)

## RESULT AND DISCUSSION

**Table: Physical and analytical data of synthesized compounds**

Compound	molecular Formula	M.P. (°C)	Yield (%)	Element % Found (Calculated)			
				C	H	N	Cl
aC <sub>12</sub> H <sub>18</sub> ClN <sub>3</sub> O <sub>3</sub>	114	87	64.73	4.40	10.31	8.62	
bC <sub>12</sub> H <sub>18</sub> ClN <sub>3</sub> O <sub>3</sub>	130	72	64.74	4.41	10.28	8.62	
c	C <sub>24</sub> H <sub>20</sub> N <sub>4</sub> O <sub>3</sub>	119	89	69.82	4.89	15.25	----

The structure of newly synthesized compounds was characterized by <sup>1</sup>H NMR and IR spectral data

### 2-(5-(2-chlorophenyl)-1-phenyl-4,5-dihydro-1H-pyrazol-3-yl)-4-methyl-6-nitrophenol

IR (KBr, cm<sup>-1</sup>): 3768 ( Strong intramolecular H-bonded -OH), 1691.57 (-C=N), 3082.225 (-C-H aromatic), 2226.01 (-C-H aliphatic), 1138.00 (-C-N), 1043.49 (-C-O), 754.17(-C-Cl ), 1593.20(-NO<sub>2</sub> asymmetric),1475.54(-C=C),1313.52(-NO<sub>2</sub> symmetric); <sup>1</sup>H-NMR(400 MHz,(CDCl<sub>3</sub>) 2.2 (s, Ar-CH<sub>3</sub>), 4.1(t, pyrazoline nucleus), 4.7 (d, pyrazoline nucleus), 4.8 (s,1H, phenolic -OH),7-8.1(m, 11H,Ar-H)

### 2-(5-(3-chlorophenyl)-1-phenyl-4,5-dihydro-1H-pyrazol-3-yl)-4- methyl-6-nitrophenol

IR (KBr, cm<sup>-1</sup>): 3741 ( Strong intramolecular H-bonded -OH), 1691.57 (-C=N), 3068.7(-C-H aromatic), 2926.01(-C-Haliphatic),1138.00 (-C-N), 1045.49 (-C-O), 752.24(-C-Cl),1539.20(-NO<sub>2</sub> asymmetric),1473.62(-C=C); <sup>1</sup>H-NMR(400 MHz,(CDCl<sub>3</sub>) 2.2 (s, Ar-CH<sub>3</sub>), 4.1(t, pyrazoline nucleus), 4.7 (d, pyrazoline nucleus), 4.89 (s,1H, phenolic -OH),7-8.1(m, 11H,Ar-H)

### 2-(5-(1H-indol-3-yl)-1-phenyl-4,5-dihydro-1H-pyrazol-3-yl)-4-methyl-6-nitrophenol

IR(KBr, cm<sup>-1</sup>): 3741(Strong intramolecular H-bonded -OH),3385.07(N-H), 3056.10(-C-H aromatic),2926.01(-C-Haliphatic),1249.87(-C-N-stretching),1531.48(-NO<sub>2</sub>asymmetric),1450.47 (-C=C-),1342.26(-NO<sub>2</sub> symmetric); <sup>1</sup>HNMR(400MHz,CDCl<sub>3</sub>)δ(ppm):2.4(s,Ar-CH<sub>3</sub>),2.32(t, pyrazoline nucleus),2.2(d,pyrazoline nucleus),7.8(s,1H,N-H).4.7(s,1H,Phenolic

-OH).6.9-8.4(m, 1H,Ar-H)

## CONCLUSION

In Summary, we have described synthesis of pyrazolines compounds derived from substituted chalcones and hydrazine hydrate in ethanol. After that the compounds were purified by crystallization. The structure of the synthesized compounds was established on the basis of spectral data from <sup>1</sup>H NMR, IR spectroscopic techniques.

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