

# “Sustainable and Eco-Friendly Approach For Synthesis Of Substituted N-Phenyl Pyrazolines By Using Diacetoxy IodoBenzene ”

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## Abstract :

The rising awareness for more sustainable technologies have focused attention on the use of atom efficient catalytic methodologies for the manufacture of fine chemicals and pharmaceuticals. Pyrazolines are prominent nitrogen containing heterocyclic compounds. Numerous pyrazoline type compounds have been found to possess useful bioactivity such as fungicidal, local anaesthetics, anticonvulsant, antifertile, CNS stimulant, antidiabetics, antiinflammatory, cardiovascular agents<sup>1-8</sup>.

**Keywords:** aromatic aldehyde and ketones, Chalcones, Diacetoxy IodoBenzene

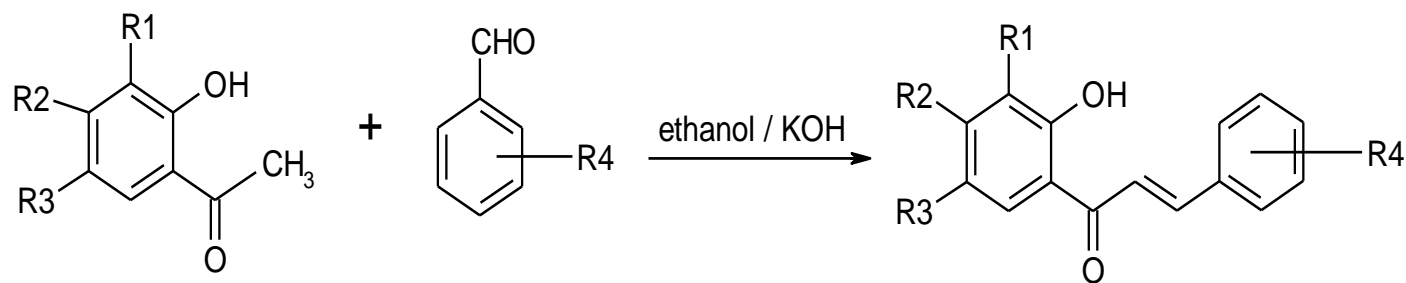
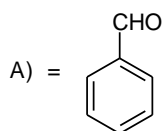
## Green Chemistry :

Also called sustainable chemistry, is a chemical philosophy encouraging the design of products and processes that reduce or eliminate the use and generation of hazardous substances. As a chemical philosophy, green chemistry derives from organic chemistry, inorganic chemistry, biochemistry, analytical chemistry, and even physical chemistry. However, the philosophy of green chemistry tends to focus on industrial applications. Click chemistry is often cited as a style of chemical synthesis that is consistent with the goals of green chemistry. The focus is on minimizing the hazard and maximizing the efficiency of any chemical choice.

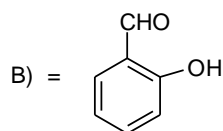
Examples of applied green chemistry are supercritical water oxidation, on water reactions and dry media reactions. Bioengineering is also seen as a promising technique for achieving green chemistry goals.

Another aspect which is receiving increasing attention is the use of alternative reaction media that circumvent the problems associated with many of the traditional volatile organic solvents. The use of non-conventional reaction media also provides opportunities for facilitating the recovery and recycling of the catalyst. The state of the art in the use of alternative reaction media for green, sustainable organic synthesis is reviewed. Liquid-liquid biphasic catalysis provides an industrially attractive method for the recovery and recycling of catalysts as an alternative to the more traditional solid heterogeneous catalysts. Various approaches to liquid liquid biphasic catalysis aqueous biphasic, fluorous biphasic, supercritical carbon dioxide, ionic liquids and various combinations thereof are reviewed and compared.

Recent advances in the development of environmentally friendly recyclable reagents and catalytic systems based on hypervalent iodine like diacetoxy iodo benzene (DIB). These efficient and ecofriendly reagents and catalytic systems are now widely used in organic synthesis for various oxidative transformations of organic substrates<sup>9-14</sup>

**Synthesis of substituted chalcones from aromatic aldehyde and ketones :****SCHEME - I**

Benzaldehyde

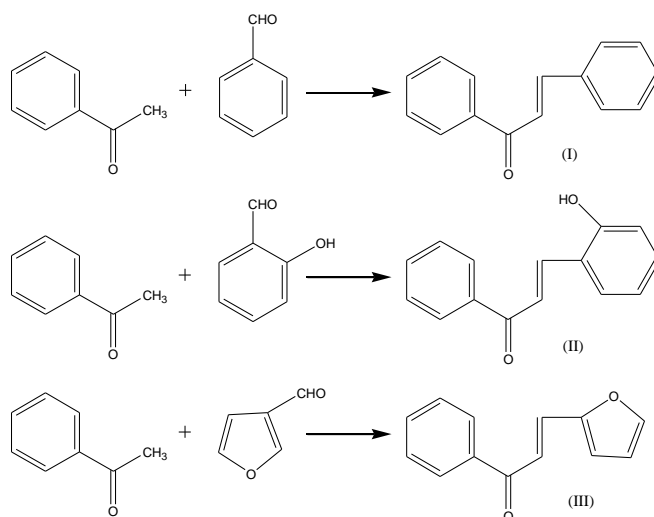


salicylaldehyde

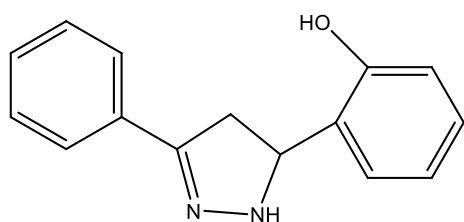
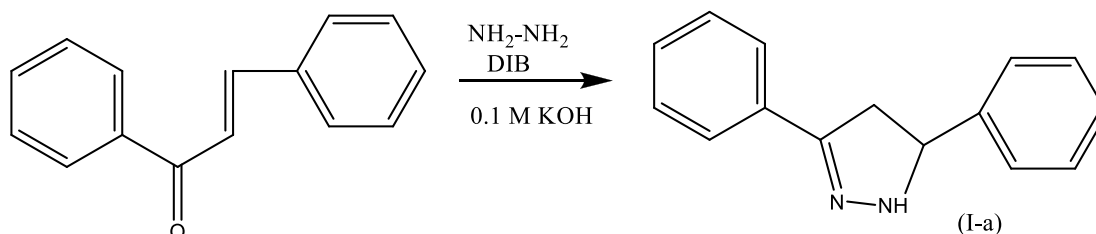
A mixture of substituted acetophenones and aryl aldehydes was stirred in ethanol and then an aqueous solution of potassium hydroxide was added to it. The mixture was kept over night at room temperature and then it was poured into crushed ice and acidified with dilute hydrochloric acid. The chalcone derivative precipitates out as solid. Then it was filtered and crystallized from ethanol (**Scheme-I**).

**Representative experimental procedure for synthesis of chalcones :**

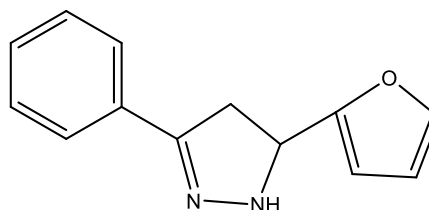
A mixture of substituted acetophenones (0.01 mole) and aryl aldehydes (0.01 moles) was stirred in ethanol (30 mL) and then an aqueous solution of potassium hydroxide (15 mL) was added to it. The mixture was kept over night at room temperature and then it was poured into crushed ice and acidified with dilute hydrochloric acid. The chalcone derivative precipitates out as solid. Then it was filtered and crystallized from ethanol (Scheme I).



Pyrazolines are prominent nitrogen containing heterocyclic compounds. Numerous pyrazoline type compounds have been found to possess useful bioactivity such as fungicidal, local anaesthetics, anticonvulsant, antifertile, CNS stimulant, antidiabetics, antiinflammatory, cardiovascular agents. Benzothiazepine systems are known to be biologically active and are important constituents of many pharmaceutical role as anticoagulant, antihypertensive and antidepressant compounds. The exploitation of simple molecules of heterocycles is a worthy contribution to the field of medicinal chemistry. This contribution prompted us to synthesize different pyrazolines and benzothiazepine with a view of obtaining antibacterial agents.



(IIa)



(III a)

### General procedure for synthesis of n-phenyl pyrazolines derivatives from Chalcone and its derivatives:

Substituted 2-propen-1-one i.e. chalcone (0.008mol) in 20ml of 1,4-dioxane, hydrazine (0.024mol) were added. To these mixtures diacetoxy iodobenzene in 0.1N KOH was added and the contents were allowed to get reflux for 4 hr. On cooling to room temperature the contents were poured on crushed ice. As a result the solid products substituted-pyrazolines and were obtained which were recrystallized using ethanol.

### RESULT AND DISCUSSION :

COMPOUND	PHYSICAL STATE	MELTING POINT	Yield (%)	MOLECULAR FORMULA
I-a	Dark Yellow crystal	162 <sup>0</sup> C	80	C <sub>21</sub> H <sub>18</sub> N <sub>2</sub>
II-a	Dark Yellow crystal	158 <sup>0</sup> C	78	C <sub>21</sub> H <sub>18</sub> ON <sub>2</sub>
III-a	Dark Yellow crystal	156 <sup>0</sup> C	76	C <sub>21</sub> H <sub>16</sub> ON <sub>2</sub>

**I-a) IR v (KBr) :** 1501, 1365, 1119, 1065 cm<sup>-1</sup>.

**II-a) IR v (KBr) :** 1501, 1362, 1103, 1070 cm<sup>-1</sup>.

**III-a) IR v (KBr) :** 3481, 1614, 1465, 1376, 1357, 1074 cm<sup>-1</sup>.

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