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Synthesis of chlorinated 3,5 diaryl 2 pyrazrolines

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ABSTRACT

Pyrazolines are important nitrogen containing 5-membered heterocyclic compounds. Various, pyrazoline derivatives have been found to possess considerable biological and pharmaceutical applications as activities, which stimulated the research activity in this field. The chlorinated 3,5-diary-l-2-pyrazolines has been synthesized by reaction of appropriately substituted chloro chalcones and mono-substituted hydrazine's have been synthesized. These compounds were characterized using IR, 1H-NMR and Mass spectra and Elemental analysis. They possess some potent biological activities. Therefore, biological screening of novel compounds has been also done.

Keywords: Pyrazolines, heterocyclic, chlorinated 3,5-diary-l-2-pyrazolines, chloro chalcones, IR, 1H-NMR.

I. INTRODUCTION

α, β Unsaturated ketones (Chalcones) has ability to react with various nucleophilic reagents. Chalcones are extensively used for the synthesis of a variety of biodynamic heterocyclic compound like, Flavone, Isoxazole, coumarin, 3-one pyridines, benzodiazepine, pyrazoline and 1,4-benzodiazipiene etc., and chalcones are said to be building blocks for many heterocyclic compounds. The presence of enone function in the chalcones moiety confers various pharmacological activities. Chalcones are found to possess a wide range of biological activity such as antibacterial antifungal anti-inflammation, anti-tumor, anti-malarial, anticancer, antimicrobial, anti-rhinovirus, anti-picornavirus, herbicidal, insect antifeedant, antiulcer, and antiinvasive. Recently more attention is paid on the synthesis of 2-propene-1-one having pharmacological important hetaryl substituent like thiazoles, 1,8-napthathyridines etc. and the evolution of their therapeutic properties.

The Pyrazole was given by Ludwig Knorr in 1883. Pyrazole refers to the class of simple aromatic ring organic compounds of the heterocyclic series characterized by a 5-membered ring structure composed of three carbon atoms and two nitrogen atoms in adjacent positions. Pyrazolines are prominent nitrogen-containing heterocyclic compounds and therefore, various procedures have been worked out their synthesis. Pyrazole derivatives have a long history of application in agrochemicals and pharmaceutical industry as herbicides and active pharmaceuticals. A systematic investigation of this class of heterocyclic lead revealed that pyrazole-containing pharmacy active agents play an important role in medicinal chemistry. 1,4 Numerous pyrazoline type compounds

have been found to possess useful bioactivity, e.g. antimicrobial, central nervous system and immunosuppressive. Among the various pyrazoline isomers, 2-pyrazolines appear to be the most frequently investigated compounds. As a consequence a large number of 2-pyrazolines have been described in the chemical literature, using different synthetic method for their preparation. An especially popular procedure is based on the reaction of α, β-unsaturated aldehydes and ketones with hydrazine's.

Pyrazoline is a unique template that is associated with several biological activities. Many researchers reported in the literature for different pharmacological activities on pyrazoline compounds synthesized. From the literature, we have concluded that pyrazoline derivatives are having a broad spectrum of biological activities. Pyrazoline and its derivatives constitute an important class of compound in drug research recently reported pyrazoline derivatives have displayed, analgesic antimicrobial, antihypertensive and abortifacient activity. Many new compounds have been made and patented, but still, there are new aspects to explore. Keeping in mind the synthetic importance of α, β-unsaturated ketone and clinical utilities of pyrazoline, we were synthesizing pyrazoline from substituted chalcones.

Present Work:

The present work deals with the synthesis of chalcone and its cyclization with hydrazine to form chlorinated 3,5 diaryl-2 pyrazolines.

The chalcones can be obtained widely by using Claisen-Schmidt condensation. In this condensation the carbonyl compound possessing active methylene group at the α position are condensed with aryl or hetaryl aldehyde in the presence of strong base. Many attempts have been made to modify the condition of Claisen-Schmidt condensation to improves the yield of the product in the reaction mixture. This condensation has been performed in the presence of the different base/catalyst like aqueous potassium hydroxide, barium hydroxide, sodium hydroxide with phase transfer catalyst & bipyridine, triethyl amine, dry hydrochloric acid & sodium acetate. α, β unsaturated ketones can be rapidly synthesized by performing Claisen-Schmidt condensation. Using microwave solid phase system, gel entrapped base catalyst, titanium (IV) chloride, zeolite, & hydrotalcite.

In the present work, the required chalcones were synthesized by condensing substituted acetophenones with aromatic aldehyde in the presence of potassium hydroxide.

This synthetic route was used because of the following advantages.

1. Simple reaction condition
2. Readily availability of starting material & catalyst.
3. Product obtained with good yield.

Thus, obtained chloro chalcone was then used for the synthesis of chlorinated 3,5 diaryl 2 pyrazolines.

Experimental:

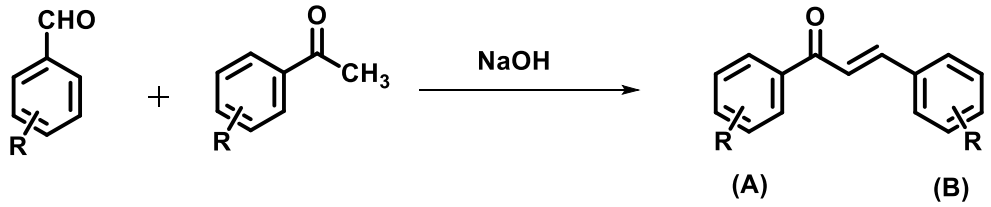
Melting points were determined by the open tube capillary method & are corrected. This purity of the compounds was controlled by thin layer chromatography (TLC) plates (silica gel G) in the solvent system ethyl acetate & -hexane. The spot was located under iodine vapour.

Step-1

General method for synthesis of chlorinated chalcones:

1. 4.4gm solution of NaOH was taken in 40 ml of water & 24.5ml of ethanol was mixed in it.
2. The solution was vigorously stirred & the flask was placed in crushed ice bath.
3. The poured solution was cooled in 10.4 gm of acetophenone (chlorinated) stirred it & 12.1gm of benzaldehyde (chlorinated) was added the reaction mixture was kept at room temperature until the mixture so thick.
4. the mixture was kept in the refrigerator in ice bath for overnight.
5. On the next day solid bar obtained filter the product wash with cold water.

Reaction:



Where,

R=Cl or H

Table Characterization of Synthesis of Chloro Chalcones:

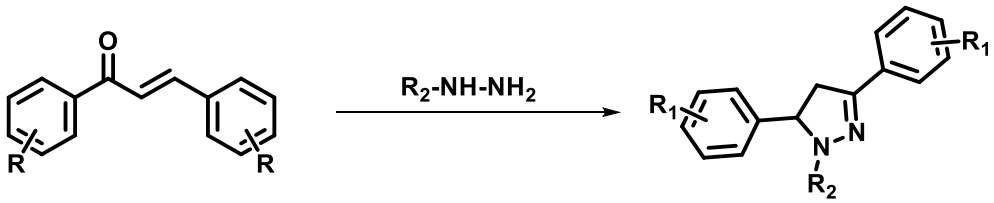
Compound	A/B	M.P/ C	%Yield	Mol. formula
1	A=4 Chlorophenyl B=Phenyl	156-158	70	$C_{15}H_{11}OCL$
2	A=4 Chlorophenyl B=4 Chlorophenyl	190-192	78	$C_{15}H_{10}OCL_2$
3	A=Phenyl B=4 Chlorophenyl	148-150	73	$C_{15}H_{11}OCL$

Step -2

Synthesis of chlorinated 3,5-diaryl-2-pyrazolines:

A mix of chloro chalcone 5.0 millimole, phenyl hydrazine or hydrazine 25.0 millimole & acetic acid 30 ml was heated at reflux for 3 hours then poured on to crushed ice. Solid can be obtained. Washed with cold water & recrystallize in alcohol.

Reaction:



Where,

R=Cl or H

R_2=Ph or H

Characterization Table of Synthesis of Pyrazoline:

Compound	Name	M.P/ C	% Yield	mol. formula
1	1Phenyl, 3(4-Chlorophenyl), 5Phenyl, 2Pyrazoline	132-135	75	$C_{21}H_{17}C_{l}N_{2}$
2	1Phenyl, 3(4Chlorophenyl), 5(4Chlorophenyl), 2Pyrazoline	145-148	83	$C_{21}H_{16}Cl_{2}N_{2}$
3	1Phenyl, 3(Phenyl), 5(4Chlorophenyl), 2Pyrazoline	128-132	70	$C_{21}H_{17}C_{l}N_{2}$

Conclusion:

In conclusion, we have synthesized a systematically substituted series of new, chlorinated 3,5-diaryl-2-pyrazolines for structure activity relationship studies. The 1-substituted-2-pyrazolines are very stable compounds, which renders them beneficial substances for biological or pharmacological trials.

II. REFERENCES

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