

Synthesis and Characterization of some novel Pyrazoline derivatives

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ABSTRACT: In present study, the new series of acetyl Pyrazoline derivatives (2a-e) have been synthesized by conventional method in excellent yield and less reaction time using ethanol via cyclization reaction of Chalcones (1a-e), hydrazine hydrates and few drops of acetic acid. The Characterizations of Pyrazolines have been established on the basis of IR, ¹NMR and elemental analysis.

KEYWORDS: Chalcone, Hydrazine hydrate, Pyrazoline, spectral analysis.

1. INTRODUCTION

The strategic development of a clean procedure for the preparation of heterocyclic compounds is a major challenge in modern heterocyclic chemistry in view of the environmental, practical and economic issues. Pyrazolines are an important class of heterocyclic compounds containing two nitrogen atoms in the five membered ring and are reported to have anti-microbial [1], anti-inflammatory [2], anti-cancer [3], anti-oxidant [4], anti-viral [5], anti-depressant [6].

In many cases Pyrazolines are found in applications such as dyestuff, analytical reagents, and in the agricultural industry. Therefore, the pyrazoline moiety has a predominant role in medicinal chemistry since it is part of many scaffolds which show an impressive array of activity.

Moreover, the substituted 2-pyrazolines have found application as activators for polymerization [7] dyes for wool, nylon [8], as electro photographic conductors [9]. Pyrazoline derivatives are the electron rich nitrogen heterocycles which play an important role in the diverse biological activities. Consider attention has been focused on pyrazoline and substituted pyrazoline [10], due to their interesting biological activities [11].

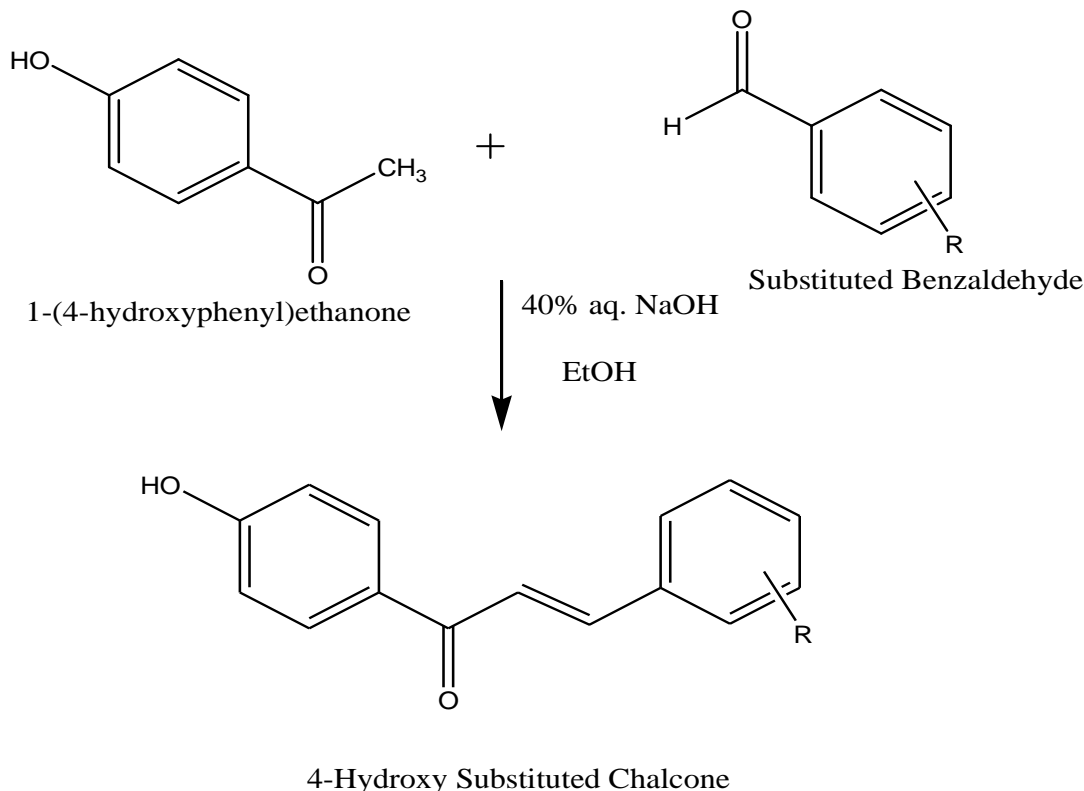
Therefore in the view of above facts, we report the reaction of different chalcone derivatives with phenyl Hydrazine hydrochloride to form pyrazolines. The structures of various synthesized compounds were assigned on the basis of elemental analysis, IR and ¹H NMR spectral data.

II. METHODS AND EXPERIMENTAL

Melting points were determined by Thieles tube method (Table 1) and were uncorrected. ¹H NMR spectra were recorded on Bruker AC 400 MHz, spectrometer using TMS (Tetra methyl silane) as an internal standard in CDCl₃. FT-IR spectra were recorded on MB 3000 (Make; ABB Bomem, Canada).

2.1 Preparation of 4-Hydroxy Substituted chalcone:

4-Hydroxy acetophenone (1 gm) was dissolved in about 10 ml ethyl alcohol and anisaldehyde or benzaldehyde were dissolved in about 20 ml ethyl alcohol this solution placed in ice cold water bath (to maintained the cold condition) and add a 10 ml of 40% NaOH solution in 3 to 4 installment with vigorous stirring. The semisolid material form was kept overnight. It was acidified with dil. HCl. The solid obtained was wash with water and recrystallized from ethanol to get a yellowish colour crystalline compound.



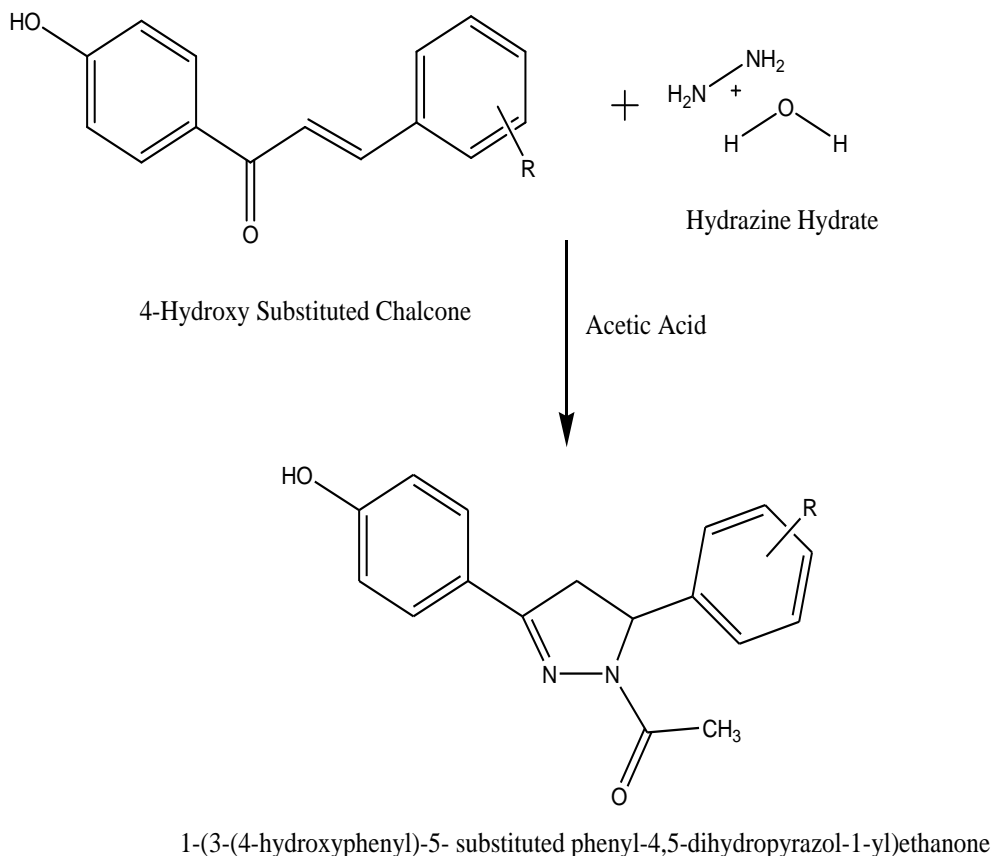
Scheme 1

2.2 Preparation of 1- acetyl-3-(4-Hydroxy phenyl)-5-(substituted phenyl)- Δ^2 - pyrazolines:

A mixture of 4-hydroxy substituted chalcone (0.2 gm) and hydrazine hydrate (0.1 ml) in 5 ml acetic acid was heat in microwave at 25 °C about 1 min. in 3 installments. The solution becomes somewhat cold at room temperature then added crush ice in this mixture was washed with water and recrystallization from ethanol get a yellowish crystalline compound.

Table: 1 Synthesized Pyrazoline Derivative.

Compounds	R1	R
2a	4-hydroxy phenyl	phenyl
2b	4-hydroxy phenyl	4-methoxy phenyl
2c	4-hydroxy phenyl	4-chloro phenyl
2d	4-hydroxy phenyl	Furan
2e	4-hydroxy phenyl	4-hydroxy phenyl



Scheme 2

III. RESULT AND DISCUSSION

New pyrazoline derivatives (2a-e) were synthesized by cyclization of substituted chalcone derivatives in presence of hydrazine hydrate (Figure 1). Physicochemical properties of synthesized compounds were determined in terms of melting point & % yield (Table 2).

Synthesized compounds were also characterized by using FT-IR and ¹H-NMR.

1-Acetyl-3-(2-hydroxyphenyl)-5-(phenyl)- Δ^2 -pyrazoline (2a):

This compound was obtained as yellowish orange colour solid in 60% yield, m.p. 210 °C; ¹H-NMR (δ): 2.26 (3H, s, -COCH₃), 3.19 (1H, t, -CH), 2.46 (2H, d, -CH₂), 4.50 (1H, s, -OH), 6.70-7.90 (9H, m, Ar-H). IR (cm⁻¹): 3509 (O-H str. of alcohol), 3140 (aromatic C-H str.), 1654 (C=C str.), 1087 (C=C bend. Aromatic), 1715 (C=O str.), 857 (C-H bend), 1660 (aliphatic C=N str.).

1-Acetyl-3-(2-hydroxyphenyl)-5-(4-methoxy phenyl)- Δ^2 -pyrazoline (2b):

This compound was obtained as yellow colour solid in 65% yield, m.p. 194 °C; ¹H-NMR (δ): 2.38 (3H, s, -COCH₃), 3.10 (1H, t, -CH), 2.54 (2H, d, -CH₂), 4.42 (1H, s, -OH), 3.85 (3H, s, -OCH₃), 6.80-8.10 (8H, m, Ar-H). IR (cm⁻¹): 3487 (O-H str. of alcohol), 3150 (aromatic C-H str.), 1640 (C=C str.), 1050 (C=C bend. Aromatic), 1710 (C=O str.), 850 (C-H bend), 1680 (aliphatic C=N str.), 1180 (C-O str. of O-CH₃).

1-Acetyl-3-(2-hydroxyphenyl)-5-(4-Chloro phenyl)- Δ^2 -pyrazoline (2c):

This compound was obtained as orange colour solid in 70% yield, m.p. 205 °C; ¹H-NMR (δ): 2.30 (3H, s, -COCH₃), 3.55 (1H, t, -CH), 2.34 (2H, d, -CH₂), 4.52 (1H, s, -OH), 6.90-8.40 (8H, m, Ar-H). IR (cm⁻¹): 3387 (O-H str. of alcohol), 3110 (aromatic C-H str.), 1630 (C=C str.), 1030 (C=C bend. Aromatic), 1730 (C=O str.), 850 (C-H bend), 1685 (aliphatic C=N str.), 860 (C-Cl str.).

1-Acetyl-3-(2-hydroxyphenyl)-5-(furan)- Δ^2 -pyrazoline (2d):

This compound was obtained as orange colour solid in 60% yield, m.p. 165 °C; ¹H-NMR (δ): 2.35 (3H, s, -COCH₃), 3.39 (1H, t, -CH), 2.30 (2H, d, -CH₂), 4.62 (1H, s, -OH), 6.85-8.20 (7H, m, Ar-H) . IR (cm⁻¹): 3347 (O-H str. of alcohol), 3160 (aromatic C-H str.), 1610 (C=C str.), 1010 (C=C bend. Aromatic), 1701 (C=O str.), 809 (C-H bend), 1650 (aliphatic C=N str.), 1230 (C-O str.).

1-Acetyl-3-(2-hydroxyphenyl)-5-(4-hydroxyphenyl)- Δ^2 -pyrazoline (2e):

This compound was obtained as pale yellow colour solid in 62% yield, m.p. 120 °C; ¹H-NMR (δ): 2.31 (3H, s, -COCH₃), 3.34 (1H, t, -CH), 2.20 (2H, d, -CH₂), 4.62-4.86 (2H, s, -OH), 6.75-8.00 (8H, m, Ar-H) . IR (cm⁻¹): 3550 (O-H str. of alcohol), 3130 (aromatic C-H str.), 1630 (C=C str.), 1050 (C=C bend. Aromatic), 1721 (C=O str.), 869 (C-H bend), 1662 (aliphatic C=N str.), 1198 (C-O str.).

Table: 2 Physical constant of synthesized pyrazoline derivative.

Compound	Melting point	% Yield
2a	210 ⁰ C	60
2b	194 ⁰ C	65
2c	205 ⁰ C	70
2d	165 ⁰ C	60
2e	120 ⁰ C	62

IV.CONCLUSION

In Summary, we have described synthesis of novel Pyrazolines compounds derived from substituted chalcones and hydrazine hydrate in ethanol. After that the compounds were purified by crystallization. The structures of the synthesized compounds were established on the basis of spectral data from ¹H NMR, IR spectroscopic techniques.

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