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MICROWAVE ASSISTED SYNTHESIS OF N-PHYNYLPYRAZOLINES DERIVED FROM BIS-CHALONES

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Abstract: In the present communication, reported the easy, convenient route for the synthesis of *N*-phenyl pyrazolines from α,β-unsaturated ketone (bis-chalcones). The reaction was done in ethanol solvent through microwave irradiation. The reaction time, yield and purity of synthesized compounds were notable advantages of present method. All synthesized compounds were confirmed on the basis of IR, ^{1}H NMR, ^{13}C NMR and CHN analysis.

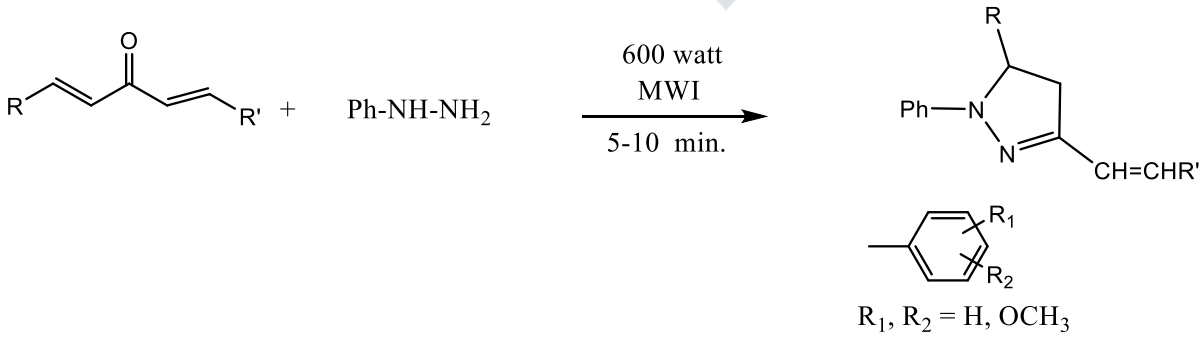
INTRODUCTION

Amongst five-membered heterocycles, Nitrogen containing heterocyclic compounds represents a wide range of pharmacological applications and pronounced biological properties [1-8]. Pyrazolines are useful synthons in organic chemistry and also important in the development of theory in heterocyclic chemistry [9-10]. A classical synthesis of these compounds involves the condensation of α,β-unsaturated carbonyl compounds with hydrazines [11-12].

The three components one step synthesis has great current interest towards development of novel synthetic organic compounds. One of the three component one step reaction involve, synthesis of *N*-phenyl pyrazolines in which α,β–unsaturated ketone reacts with phenyl hydrazine under microwave irradiation.

2. MATERIAL AND METHODS
CHEMICAL AND INSTRUMENTATION

All chemicals, solvents and reagents used in the present study were of analytical grade purchased from Sigma, SD Fine, or Spectrochem. Melting points were determined in an open capillary tube and are uncorrected. The reactions were carried out in ethanol solvent (10 mL:10 mL, v/v) using 200. Purification of the compounds was indicated using TLC (mixture of ethyl acetate and hexane, 0.20 mL:0.20 mL, vv). IR spectra were recorded in KBr pellets on a Perkin-Elmer FT-IR Shimadzu spectrometer. ^{1}H and ^{13}C NMR spectra were obtained in DMSO-*d*6 on Avance 300 MHz spectrometer using TMS as an internal standard. The mass spectra were recorded on EI-Shimadzu-GC-MS spectrometer.



Scheme **1**: Synthesis of N-phenyl pyrazolines derived from bis-chalcones

General Procedure for synthesis of *N*-phenylpyrazolines derived from bis-chalones:
In 50 mL round bottom flask, a mixture of α,β-unsaturated ketones(0.01 mol), phenyl hydrazines(0.01mmol) was dissolved in ethanol (20 mL) by warming. The resulting reaction solution were irradiated under microwave irradiation for 5-10 min. The progress of reaction was indicated by TLC. After completion of reaction the precipitate formed was filtered through simple büchner funnel under vacuum pressure and crystallized from ethanol to yield 2-pyrazolines (Scheme **1**).

3a. 4,5-dihydro-1,5-diphenyl-3-styryl-1H-pyrazole:

Yield: 75%, m.p. 203-205 ºC.IR (KBr): 1635 (C=O), 1588 (C=N) cm-1. 1H NMR (300 MHZ, DMSO-*d6*) δ 3.21 (dd, *J* = 5.0, 17.8 HZ, 1H, HA), δ 3.65 (dd, *J* = 12.0, 17.8 HZ, 1H, HB), δ 5.57 (dd, *J* = 5.1, 12.1 HZ, 1H, HX), δ 6.74 (d, *J* = 16.2 HZ,1H, Hα), δ 7.17 (d, *J* = 16.2 HZ, 1H, Hβ), δ 7.29-7.74 (m, 15H, Ar-H). m/z 324.43 (100%).

3b. 3-(4-methoxyphenyl)-4,5dihydro-5-(4-methoxystyryl)-1-phenyl-1H-pyrazole:

Yield: 78%, m.p. 184-187 ºC. IR (KBr): 1632 (C=O), 1580 (C=N) cm-1. 1H NMR (300 MHZ, DMSO-*d6*) δ 3.70 (s, 6H, *two* OCH_3), δ 3.24(dd, *J* = 5.0, 17.8 HZ, 1H, HA), δ 3.69 (dd, *J* = 12.0, 17.8 HZ, 1H, HB), δ 5.57 (dd, *J* = 5.1, 12.1 HZ, 1H, HX), δ 6.78 (d, *J* = 16.2 HZ,1H, Hα), δ 7.13 (d, *J* = 16.2 HZ, 1H, Hβ), δ 7.24-7.54 (m, 13H, Ar-H). m/z 384.48 (100%).

3. CONCLUSION

In summary, present communication reported the synthesis of *N*-phenyl pyrazolinesderivatives starting from α,β-unsaturated ketone in ethanol under MWI. The advantage of methods has notable advantages which includes clean reaction procedure, easy isolation of products, short reaction time and no need of special apparatus device.

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