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Microwave Induced Synthesis of Some Novel Heterocycles From Substituted Chalcones

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Various substituted chalcones have been converted into 1,3,5-tri substituted pyrazolines. These pyrazolines have been obtained by reacting chalcones with isoniazide and thiosemicarbazide in stoichiometric proportion in microwave oven. The reaction mixture was irradiated within microwave oven for 1 min at 150 watt. The yields obtained with these techniques are higher and purity of compound obtained is better as compared to classical method of synthesis. The structures of all these pyrazolines Δ^2 have been conformed on the basis of elemental analysis and spectroscopic data.

Key Words: Synthesis of 1,3,5-tri substituted pyrazolines, Microwave irradiation.

INTRODUCTION

Synthesis of several chalcone are reported in literature[1,2]. In recent past researchers have been activity engaged in the synthesis of various heterocycles having more than one hetero atom with structural and functional novelty[3-8]. In fact amongst substituted heterocycles, benzimidazole, thiazole, isoniazid have gained great interest due to their successful application in clinical pharmaceutical chemistry.

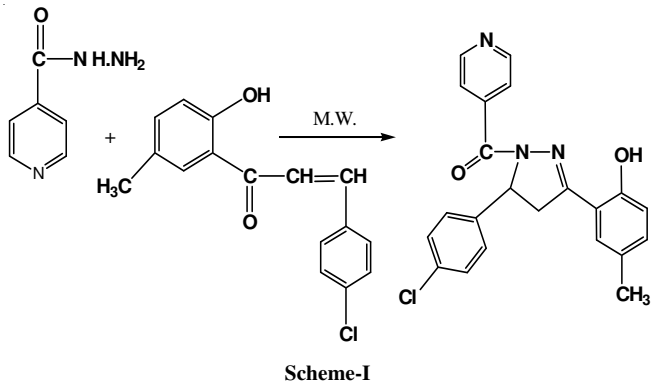
Heterocyclic compound are widely used in pharmaceutical industry for the manufacture of large number of common drugs. Heterocyclic nuclei has been reported to have biological activities like antihistamic, analgesic and hypothermic, antiinflammatiry, antitumor, antifungal, antibacterial and anti-tubercular and variety of physiological properties like hyphotics, analgesic, local and spiral anesthetic CNS-stimulant, anticancer and anti HIV[9-12].

In fact amongst several substituted heterocycles, isoniazid, thiosemicarbazide have gained great intrest due to their application in clinical and pharmaceutical chemistry. Pyrazolines substituted on nitrogen exibit tautomerisum are stable, are called Δ^2-pyrazoline.

EXPERIMENTAL

The melting points are recorded using hot paraffin bath and are uncorrected.

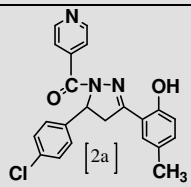
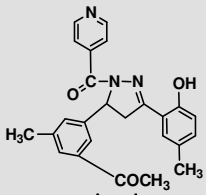
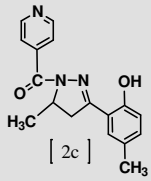
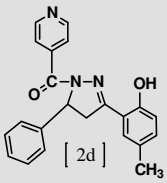
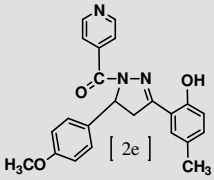
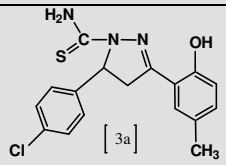
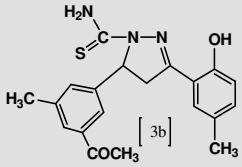
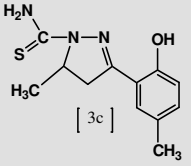
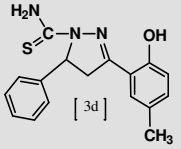
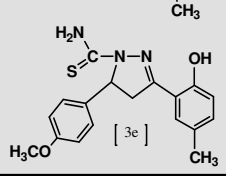
Reaction of isoniazid with 1-*p*-chlorophenyl-3-(2-hydroxyl-5-methyl phenyl chalcone (2a): Isoniazid (0.005 mol) and 1-*p*-chlorophenyl-3-2-hydroxy-5-methyl phenyl chalcone (0.005 mol) in (1-2 drops) ethanol was added to it moisten the mixture. The beaker was covered with a watch glass and irradiated in a microwave oven for 1 min. After completion of reaction the beaker was remove from the oven and mixture was cooled to room temperature, the product was crystallized from ethanol (**Scheme-I**). Yield: 79 % and m.p. was found to be 130 ºC. Molecular formula of synthesized compound was found to be $C_{20}H_{16}N_3O_2Cl$. The physical characteristics of other derivatives are given in Table-1.



Scheme-I

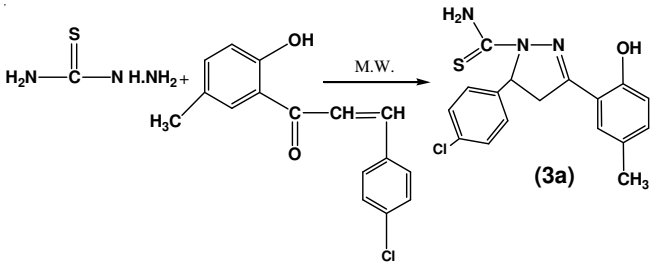
The IR spectral analysis of the product indicated the presence of ν(OH) 3460.5 cm^{-1}, ν(C=O) 1645.7 cm^{-1}, ν(N-N) 1213.0 cm^{-1}, ν(C-N) 1340.9 cm^{-1}. The NMR spectral analysis of the product indicated the presence of 12.48 (1H, bs, OH); 7.92-8.76 (4H, d, Py-H); 7.08-7.83 (12H, m, Ar-H); 6.85-6.87 (2H, d, $-CH_2$); 2.48 (3H, s, $-CH_3$).

TABLE-1
PREPARATION OF 1,3,5-TRI SUBSTITUTED PYRAZOLINE USING ISONIAZID (**2a-e**) THIOSEMICARBAZIDE (**3a-e**) AND DIFFERENT TYPE OF CHALCONE

Compound No.	1,3,5-Tri substituted Δ^2-pyrazolines	Time (min)	Yield (%)	Elemental analysis (%) calcd. (found)			
				C	H	N	S
2a	 [2a]	2	87	67.86 (66.45)	4.11 (3.15)	10.79 (10.20)	–
2b	 [2b]	1.5	90	72.99 (72.60)	5.10 (4.80)	10.21 (9.70)	–
2c	 [2c]	2	56	69.62 (69.40)	5.11 (4.70)	14.33 (13.80)	–
2d	 [2d]	2.4	67	74.36 (73.12)	4.78 (4.00)	11.83 (11.70)	–
2e	 [2e]	2.2	37	71.68 (71.45)	4.93 (4.50)	10.90 (10.20)	–
3a	 [3a]	2	66.35	9.47 (59.02)	4.08 (3.60)	12.24 (12.00)	9.32 (10.35)
3b	 [3b]	2.1	69.97	65.75 (65.25)	5.02 (4.85)	11.50 (10.97)	8.76 (8.65)
3c	 [3c]	1.5	70	58.29 (58.15)	5.26 (5.14)	17.00 (16.80)	12.95 (12.75)
3d	 [3d]	2.5	73.2	66.01 (65.58)	4.85 (4.48)	13.59 (13.27)	10.35 (10.16)
3e	 [3e]	2.1	47.8	63.71 (63.59)	5.01 (4.00)	12.15 (12.15)	9.43 (9.20)

Compound 2b: The IR spectral analysis of the product indicated the presence of ν(OH) 3514 cm^{-1}, ν(C=O) 1665.3 cm^{-1}, ν(N-N) 11.90 cm^{-1}, ν(C-N) 1373.5 cm^{-1}. The NMR spectral analysis of the product indicated the presence of 11.82 (1H, b s, OH); 8.72-8.76 (4H, d, Py-H); 7.07-7.74 (12H, m, Ar-H); 3.35 (2H, d, $-COCH_3$); 2.31-2.49 (3H, s, $-CH_3$).

Reaction of thiosemicarbazide with 1-*p*-chlorophenyl-3,2-hydroxy-5-methyl phenyl chalcone: Thiosemicarbazide (0.005 mol) and 1-*p*-chlorophenyl-3-2-hydroxy-5-methyl phenyl chalcone (0.005 mol) in (1-2 drops) ethanol was added to moisten the mixture. The beaker was covered with a watch glass and irradiated in a microwave oven for 1 min. After completion of reaction the beaker was remove from the oven and mixture was cooled to room temperature, the product was crystallized from ethanol (**Scheme-II**). Yield: 66.35 % and m.p. was found to be 140 ºC, molecular formula of the synthesized compound 1-thioamido-3-(2-hydroxy-5-methyl phenyl-5-*p*-chlorophenyl pyrazolines (**3a**) was found to be $C_{17}H_{14}N_3OSCl$. The physical characteristics of other derivatives are given in Table-1.



Scheme-II

Compound 3a: The IR spectral analysis of the product indicated the presence of ν(OH) 3366 cm^{-1}, ν(-NH) 3262.3-3174.9 cm^{-1}, ν(N-N) 1213 cm^{-1}, ν(C-N) 1340 cm^{-1}. The NMR spectral analysis of the product indicated the presence of 12.59 (1H, b s, OH); 7.83-7.92 (2H, d, NH_2); 7.31-7.66 (12H, m, Ar-H); 2.35 (3H, s, $-CH_3$).

Compound 3b: The IR spectral analysis of the product indicated the presence of ν(OH) 3350 cm^{-1}, ν(-NH) 3262.1-3174.9 cm^{-1}, ν(N-N) 1215.6 cm^{-1}, ν(C-N) 1343.2 cm^{-1}. The NMR spectral analysis of the product indicated the presence of 12.59 (1H, b s, OH); 7.88-7.92 (2H, d, NH_2); 7.29-7.66 (12H, m, Ar-H); 2.34 (3H, s, $-CH_3$).

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