

Synthesis of 3-(2-hydroxy-3,4-benzophenyl-5-methoxy)-5-(4-methoxy-phenyl)-1-substituted Pyrazolines

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Abstract

1-(2-hydroxy-3,4-benzophenyl-5-methoxy)-3-aryl-prop-2-ene-1-one & semicarbazide / thiosemicarbazide / phenylhydrazine /isonicotinic acid hydrazide were added to DMF and the mixture was refluxed for about 2 hours . The reaction mixture was cooled and diluted with water. The semisolid so obtained was triturated with ethanol to get a solid which was recrystallised from ethanol-acetic acid mixture to obtain titled pyrazolines

Keywords: Synthesis ,Pyrazolines.

INTRODUCTION

Pyrazoline Derivatives having five membered heterocyclic compounds are known to be biologically active and are important constituents of many pharmaceutical and agrochemical products. Pyrazolines and isoxazoles have been found to be established that introduction of thiophenyl group associated with diverse biological activities and numerous reports have appeared in the literature which highlighted their chemistry and use. Pyrazole derivatives were known to possess many biological derivatives including antibacterial¹ antifungal² antiinflammatory anti-depressant⁴ anti-tumor⁵ musclerelaxant⁶ anti-arthritic⁷ analgesic⁸ and anticonvulsant⁹ properties Also, varied medicinal activities such as anti-inflammatory¹⁰ antibacterial¹¹ Anticonvulsant¹², antibiotic¹³ anti-tubercular¹⁴ antiungal¹⁵ and anxiolytic activity¹⁶

Heterocyclic compounds containing nitrogen are widely distributed in nature and play an important role in the metabolism of all living cells. Present work deals with the synthesis of 3-(2-hydroxy-3,4-benzophenyl-5-methoxy)-5-aryl-1-substituted Pyrazolines and their characterization by elemental analysis, IR, ¹H NMR analysis.

EXPERIMENTAL

All the melting points were taken in silicon oil bath with open capillary tubes and are uncorrected. IR spectra were recorded on a Nicolet-Impact 400 FT-IR spectrometer . ¹ H NMR spectra were recorded on a Bruker AC300 FNMN spectrometer (300 MHz), using TMS as an internal standard. Microanalysis of nitrogen was obtained on Colman 29-N analyzer. Thin Layer Chromatography on silica gel-G, was used to check the purity of the compounds.

Synthesis of 2-Acetyl- 4 methoxy-1-naphthol

2-Acetyl- 4 methoxy-1-naphthol was prepared by refluxing 4-methoxy-1-naphthol with glacial acetic acid in presence of fused ZnCl₂.

Synthesis of 1-(2-Hydroxy-3,4-benzophenyl-5-methoxy)-3-aryl-prop-2-ene-1-one

1 - (2 -Hydroxy -3 ,4 -benzophenyl-5 -methoxy) -3-aryl -prop -2-ene -1-one were synthesized from 2-acetyl-4-methoxy-1-naphthol by condensing it with aromatic aldehydes.

Synthesis of 3-(2-hydroxy-3,4-benzophenyl-5-methoxy)-5-(4-methoxy phenyl)-1-substituted Pyrazolines.

1-(2-hydroxy-3,4-benzophenyl-5-methoxy)-3-aryl-prop-2-ene-1-one & semicarbazide / thiosemicarbazide phenylhydrazine /isonicotinic acid hydrazide were added to DMF and the mixture was refluxed for about 2 hours . The reaction mixture was cooled and diluted with water. The semisolid so obtained was triturated with ethanol to get a solid which was recrystallised from ethanol-acetic acid mixture to obtain titled pyrazolines

Spectral interpretation of (4a)

IR (ν_{max}) (cm⁻¹): 3418(-OH str.) 1320 (C-O-C) , 1570 (C=N str),

NMR (δ ppm): 3.85 (s, 3H, OCH₃) , 3.82 (s, 3H, OCH₃), 11.71 (s, 1H, OH), 5.15 (d, 1H, -CH , J= 12Hz), 6.09 (d, 1H, -CH , J= 12Hz) 6.52-8.3 (m , 14 H, Ar-H)

Spectral interpretation of (4b)

IR (ν_{max}) (cm⁻¹): 3430(-OH str.) 1650 (C=O str), 3218 (NH₂ str.), 1325(C-O-C) , 1572 (C=N str)

NMR (δ ppm): 3.80 (s, 3H, OCH₃) , 3.83 (3H, OCH₃), 6.66-7.44 (m , 9 H, Ar-H) , 7.64 (s, 2H, -NH₂), 11.75 (s, 1H, OH) 5.15 (d, 1H, CH , J= 12Hz), 6.09 (d, 1H, CH , J= 12Hz)

Spectral interpretation of (4c)

IR (ν_{max}) (cm⁻¹): 3345 (OH str), 1322(C-O-C) , 1565 (C=N str), 3212 (NH₂ str.)

NMR (δ ppm): 3.80 (s, 3H, OCH₃) , 3.83 (3H, OCH₃), 7.69 (s, 2H, -NH₂), 11.78 (s, 1H, OH) 5.15 (d, 1H, CH , J= 12Hz), 6.09 (d, 1H,CH, J= 12Hz) , 6.52-8.3 (m ,9 H, Ar-H)

Spectral interpretation of (4d)

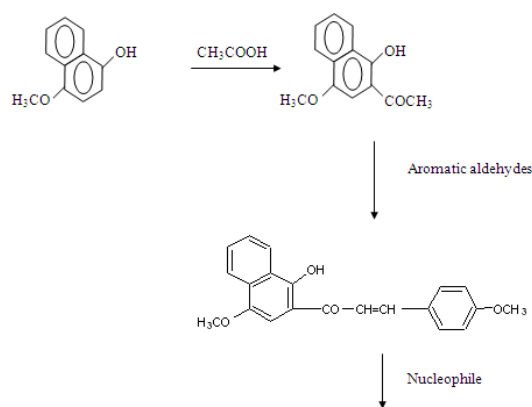
IR (ν_{\max}) (cm^{-1}): 3390 (OH str), 1636 (C=O str) 1572, (C=N str), 1330 (C-O-C)

NMR (δ ppm): 3.80 (s, 3H, OCH_3), 3.83 (3H, OCH_3), 5.13 (d, 1H, -CH, $J=12\text{Hz}$), 6.10 (d, 1H, -CH, $J=12\text{Hz}$), 7.41-7.88 (m, 14H, Ar-H), 12.47 (s, 1H, OH)

Table 1. Physical data of synthesized compounds.

Sr. No.	Compound	R	Melting Point $^{\circ}\text{C}$	% Yield	% Nitrogen		R.F. Value
					Found	Calculated	
1	1	--	98 $^{\circ}\text{C}$	68%	--	--	--
2	2	--	128 $^{\circ}\text{C}$	64%	--	--	--
3	3	--	149 $^{\circ}\text{C}$	58%	--	--	--
4	4a	C_6H_5	198 $^{\circ}\text{C}$	43%	6.59	6.60	0.62
5	4b	CONH_2	213 $^{\circ}\text{C}$	45%	10.73	10.74	0.71
6	4c	CSNH_2	267 $^{\circ}\text{C}$	39%	10.30	10.32	0.55
7	4d	$\text{C}_2\text{H}_4\text{NCO}$	188 $^{\circ}\text{C}$	42%	9.26	9.27	0.58

SCHEME I



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REFERENCES

- [1] Nauduri D & Reddy G.B. *Chem Pharm Bull*, Tokyo, 46,1998,1254.
- [2] Korgaokar S S, Patil P H, Shah MT & Parekh H H, *Indian J Pharm Soc*, 58, 1996,222.
- [3] Nagent R.A, Murphy M, Schlachter S T, Dunn C J, Smith J R, Staite N.D. Galinet A.L, Asar D G & Richard KA, *Med Chem*,36,1993, 134.
- [4] Palaska E, Aytemir M, Uzbay IT & Erol D, Erol D, *Eur J Med Chem*, 36, 2001, 539.
- [5] Taylor E S & Patel H H, *Tetrahedron*, 48, 1992,8089
- [6] Anderson EL, Casey J.E. Greene, Lafferty J L & Reif H.E, *J Med Chem*, 7, 1964, 259.
- [7] Rangari V.Gupta V N & Atal C K, *Indian J Pharm Soc*,52,1990,158
- [8] Manna F, Chimenti F, Bolasco A, Cenjcola ML, Amico M, Parrilo C, Rossi F & Marmo E, *Eur J Med Chem* 27, 1992,633.
- [9] Taylor J, Chandler R, Stauffer J & Harold F US Pat 4871737,1989.
- [10] Hans P & Walter P, US Pat 3668215,1972.
- [11] Hoffer M, US Pat 2721200, 1955.
- [12] Uno H, Kurokawa M, Masuda Y & Nishimura H, *J med Chem*, 22,1979,180.
- [13] Doyle F.P. Betchworth G & Charles J.H. US pat 2996501.1961.
- [14] Haripara K, Patel S, Joshi A & Paresh H, *Indian J Het Chem* 13, 2004,221.
- [15] Sorithiya S D. Patel V B & Parikh A.R. *Indian J.Chemistry*, 36B, 1997,822.
- [16] Wagner E, Becan L & Nowakowska E, *Bio-org Med Chem*, 12 2004,265.