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Synthesis and characterization of pyrazoline derivatives obtained from 4-bromo-naphthalen-1-ol

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ABSTRACT

1-(4-bromo-1-hydroxynaphthalen-2-yl)-ethanone have been synthesized from 4-bromo-naphthalen-1-ol by refluxing it with glacial acetic acid in presence of fused $ZnCl_2$ by modified Nenchis method. 1-(4-bromo-1-hydroxy-naphthalen-2-yl)-3-aryl-prop-2-en-1-one were synthesized from 1-(4-bromo-1-hydroxy naphthalen-2-yl)-ethanone by condensing it with aromatic aldehydes. Then these newly synthesized 1-(4-bromo-1-hydroxy-naphthalen-2-yl)-3-aryl-prop-2-en-1-one were cyclized with nucleophiles like phenyl hydrazine / semicarbazide / thiosemi carbazide in DMF solvent and refluxed for 2 hours . The cooled reaction mixture was diluted with water the semisolid so obtained was triturated with ethanol to get a solid which was recrystallised from ethanol–acetic acid mixture to get pyrazoline derivatives. The synthesized compounds were characterized by elemental analysis, 1H NMR, IR Spectroscopy.

Key words: Synthesis , characterization, IR Specra, NMR Spectra, pyrazolines.

INTRODUCTION

In recent scenario heterocycles play a major role in drug synthesis in that respect pyrazoline derivatives plays a significant role among other heterocycles. From the literature survey, in recent years pyrazoline derivatives have attracted considerable interest because of their therapeutic and pharmacological properties. Pyrazolines are well known heterocyclic compound and important nitrogen-containing five-membered hetero- cyclic compounds and various methods have been worked out for their synthesis[1 - 4].Various pyrazoline derivatives are important biological agents and a significant amount of research activity has been directed towards this class of compounds[5-8]. In particular, they show , antimyco-bacterial[9], anti-inflammatory, analgesic[10-12] antidepressant activities[13], bioactive heterocycles[14-17], central nervous system stimulant and immuno suppressive, [18] antimicrobial[19] and antibacterial activities[20].

Synthesis charecterization and biological evaluation of pyrazoline derivatives becomes favorate field for many investigator their efforts are quite significant in literature.Hence, a series of novel pyrazoline derivatives from 4-bromo-naphthalen-1-ol has been synthesized.

MATERIALS AND METHODS

The melting points (ºC) were recorded by open capillary method and are uncorrected. IR spectra (υ max in cm-1) were recorded on a Shimadzu FTIR 8300 spectrophotometer using KBr pellets. The 1H NMR spectra were recorded on aDRX-300 (300 MHZ) instrument using $CDCl_3$ as solvent (chemical shift in δppm), and TMS as internal standard. Thin Layer Chromatography on silica gel-G, was used to check the purity of the compounds.

Method and Discussion of result:

Synthesis of 1-(4-bromo-1-hydroxynaphthalen-2-yl)-ethanone (2)

1-(4-bromo-1-hydroxynaphthalen-2-yl)-ethanone was prepared by refluxing 4-bromo-naphthalen-1-ol with glacial acetic acid in presence of fused $ZnCl_2$.

Synthesis of 1-(4-bromo-1-hydroxy-naphthalen-2-yl)-3-aryl-prop-2-en-1-one (3-6)

1-(4-bromo-1-hydroxynaphthalen-2yl)-2-aryl-prop-2-en-1-one were synthesized from 1-(4-bromo-1-hydroxynaphthalen-2-yl)-ethanone by condensing it with aromatic aldehydes.

Synthesis of pyrazoline derivatives (7-18)

1-(4-bromo-1-hydroxynaphthalen-2yl)-2-aryl-prop-2-en-1-one phenyl hydrazine / semicarbazide / thiosemi carbazide were added to DMF and refluxed for 2 Hours . The cooled reaction mixture was diluted with water the semisolid so obtained was triturated with ethanol to get a solid which was recrystallised from ethanol–acetic acid mixture to get pyrazoline derivatives.

Table 1. PHYSICAL DATA OF SYNTHESIZED COMPOUNDS

Sr.No.	Compound No	R_1	R_2	R_3	Molecular formula	Melting Point ^0C	% Yield	% Nitrogen		R.F. Value
								Found	Calculated	
1	3	$-OCH_3$	-H	--		124^0C	55%	--	--	--
2	4	$-OCH_3$	$-OCH_3$	--		120^0C	53%	--	--	--
3	5	-H	-OH	--		140^0C	57%	--	--	--
4	6	-OH	-H	--		144^0C	59%	--	--	--
5	7	$-OCH_3$	-H	C_6H_5	C26H21BrN2O2	205^0C	43%	5.90	5.92	0.54
6	8	$-OCH_3$	$-OCH_3$	C_6H_5	C27H23BrN2O3	203^0C	45%	5.53	5.57	0.61
7	9	-H	-OH	C_6H_5	C25H19BrN2O2	217^0C	42%	6.07	6.10	0.63
8	10	-OH	-H	C_6H_5	C25H19BrN2O2	225^0C	38%	6.09	6.10	0.57
9	11	$-OCH_3$	-H	$-CONH_2$	C21H18BrN3O3	290^0C	40%	9.51	9.55	0.58
10	12	$-OCH_3$	$-OCH_3$	$-CONH_2$	C22H20BrN3O4	283^0C	43%	8.93	8.94	0.62
11	13	-H	-OH	$-CONH_2$	C20H16BrN3O3	278^0C	41%	9.83	9.86	0.55
12	14	-OH	-H	$-CONH_2$	C20H16BrN3O3	285^0C	44%	9.85	9.86	0.54
13	15	$-OCH_3$	-H	$-CSNH_2$	C21H18BrN3O2S	185^0C	42%	9.20	9.21	0.59
`4	16	$-OCH_3$	$-OCH_3$	$-CSNH_2$	C22H20BrN3O3S	179^0C	43%	8.63	8.64	0.63
15	17	-H	-OH	$-CSNH_2$	C20H16BrN3O2S	188^0C	45%	9.48	9.50	0.62
16	18	-OH	-H	$-CSNH_2$	C20H16BrN3O2S	197^0C	41%	9.47	9.50	0.57

Spectral interpretation of (7)

IR (ν_{max}) (cm^{-1}) : 3337 (OH, str), 3217 (N-N pyrazoline), 1588 (C=N str), 3013 (CH str in Ar)

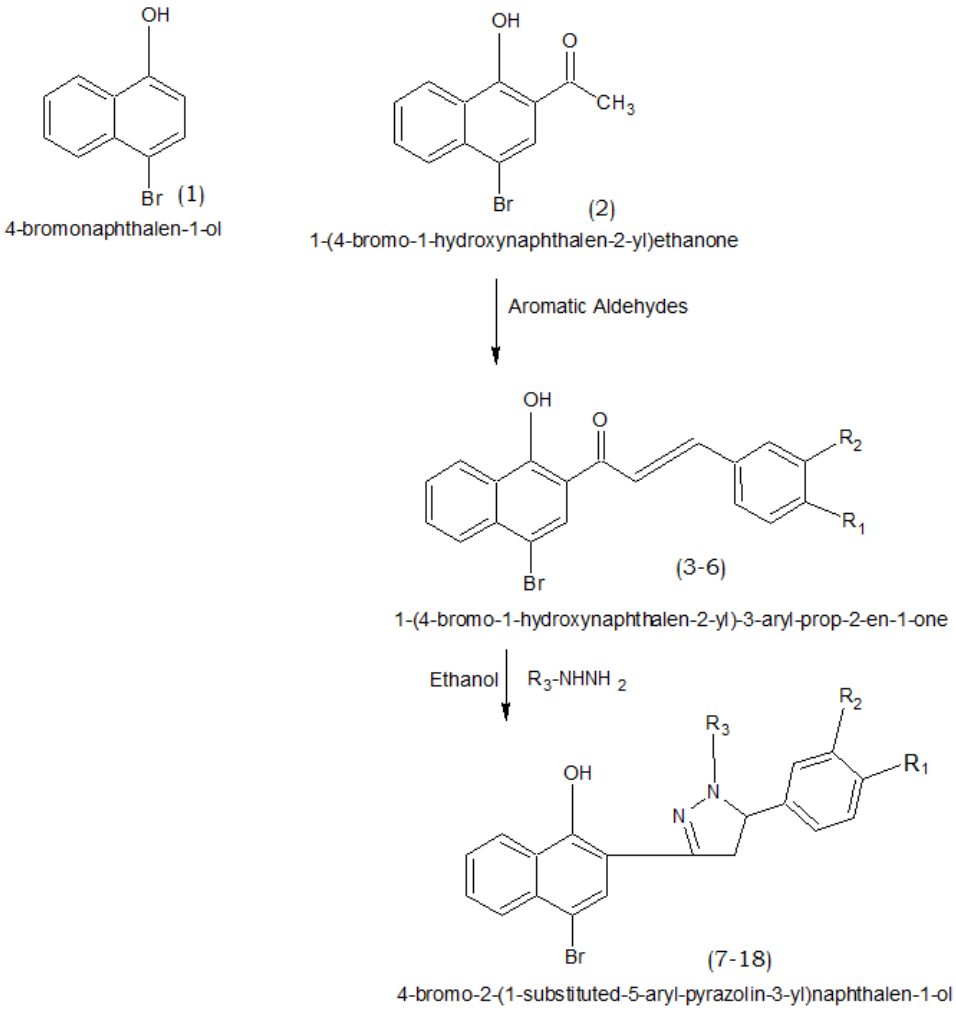
NMR (δ ppm): 9.51 (s, 1H, OH), 3.077–3.154 (dd, 1H, H_A), 3.618-3.718 (dd, 1H, H_B) , 5.297-5.252, (dd, 1H, H_X) , 8.17- 8.73 (m, 14Ar-H) , 3.75 (s, 3H, OCH_3).

Spectral interpretation of (12)

IR (ν_{max}) (cm^{-1}): 3333 (OH str), 3221 (N-N pyrazoline), 1583 (C=N str), 1663 (NH bend)

NMR (δ ppm): 3.087–3.162 (dd, 1H, H_A) , 3.626-3.726 (dd, 1H, H_B) , 5.205-5.260, (dd, 1H, H_X) 8.13- 8.78 (m, 9 Ar-H) , 10.08 (s, 1H, OH),8.98 (s, 1H, OH) ,6.73 (s, 2H, NH_2).

SCHEME



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