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Synthesis and Characterization of 3-(1-hydroxy naphthalene-2-yl)-5-(furan-2-yl)-1-substituted pyrazolines

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ABSTRACT

2-acetyl-1-naphthol 2 is prepared by Modified Nenchi's method which on treatment with furfuraldehyde and KOH gives 1-(1-hydroxy naphthalen-2-yl)-3-(furan-2-yl) prop-2-ene-1-ones 3 in excellent yield. The chalcone 3 when subjected to hydrazine / phenyl hydrazine/ semicarbazide / 2,4 dinitro phenyl hydrazine / isonicotinic acid hydrazide in DMF solvent gives 3-(1-hydroxy naphthalene-2-yl)-5-(furan-2-yl)-1-substituted pyrazolines 4, 5, 6, 7 and 8 in 35-45% yield. The structural assignments to the compounds 4, 5, 6, 7 and 8 are based on their elemental analysis and spectral data.

Key words: Synthesis, Characterization, Pyrazolines.

INTRODUCTION

The literature survey reveals that pyrazoline derivatives have been studied extensively because of their ready accessibility, diverse chemical reactivity, broad spectrum of biological activity[1-9] and variety of Industrial applications[10-12]. Pyrazolines with sulphonamidoaryl substituent at 3-position show cerebroprotective[13], antidepressant activity[14], anti-implantation activity[15], hypoglycemic activity[16]. Due to this vital biological roll of pyrazoline derivatives, it was thought of interest to synthesize titled pyrazoline derivatives . Thus we present herein the synthesis of the titled compounds.

It has been observed that substituted flavanones are the best starting compounds for the preparation of 4-aroyl derivatives of pyrazoline. Present work deals with the synthesis of some new pyrazolines and their characterization by spectral analysis (IR, ^{1}H NMR)

EXPERIMENTAL

All the melting points were taken in silicon oil bath with open capillary tubes and are uncorrected. ^{1} H NMR spectra were recorded on a Brucker AC300 FNMR spectrometer(300MHz), using TMS as an internal standard. IR spectra were recorded on a Nicolet-Impact 400 FT-IR spectrometer. Thin

Layer Chromatography on silica gel-G, was used to check the purity of the compounds. Microanalysis of nitrogen was obtained on Colman 29-N analyzer.

Experimental Section

Procedure for the synthesis of 2-acetyl-1-naphthol 2

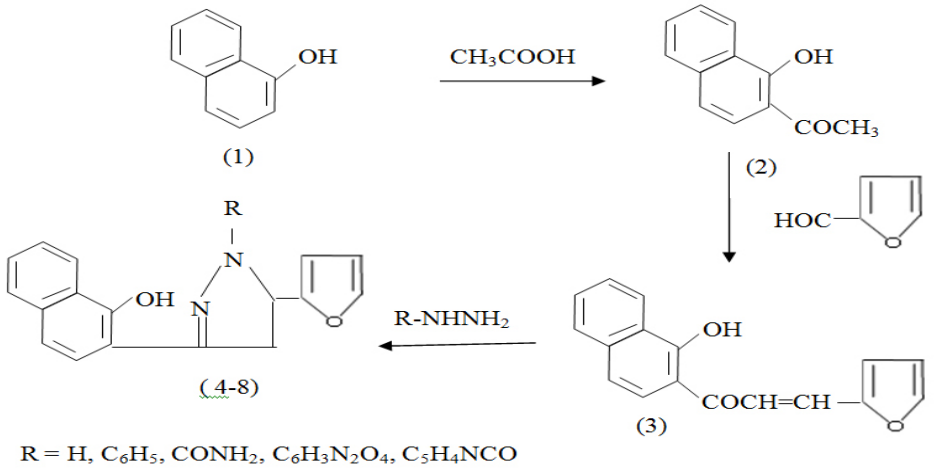
In hot glacial acetic acid (80ml), fused $ZnCl_2$(50 gm) was added and refluxed till dissolved, then powdered 1-naphthol (30gm)was added and the mixture was refluxed for about 8 hours then cooled & poured in acidulated water. The solid obtained was filtered, washed, dried and recrystallized from rectified spirit to obtain compound 2. Physical data of the compound is given in table 1 .

2: IR (KBr) : 1650 (C=O), 3412 (-OH)

NMR ($CDCl_3$ + DMSO-d6) : δ 2.35 (S,3H,CH_3), δ 7.11-6.88 (m, 6H, Ar-H), ´ 9.83 (S,1H,-OH)

Table 1: Physical and analytical characterization data of newly synthesized compounds

S. No.	Comp ound No	Molecular formula	R	Melting Point ^{0}C	% Yield	% Nitrogen Found	% Nitrogen Calculated	R.F. Value
1	2	$C_{12}H_{10}O_2$	-	98^{0}C	72%	-	-	
2	3	$C_{17}H_{12}O_3$	-	126^{0}C	71%	-	-	
3	4	$C_{17}H_{14}N_2O_2$	H	110^{0}C	38%	10.00	10.07	0.79
4	5	$C_{23}H_{18}N_2O_2$	C_6H_5	100^{0}C	40%	7.81	7.91	0.88
5	6	$C_{18}H_{15}N_3O_3$	$CONH_2$	140^{0}C	45%	12.99	13.08	0.87
6	7	$C_{23}H_{16}N_4O_6$	$C_6H_3N_2O_4$	120^{0}C	40%	12.56	12.61	0.78
7	8	$C_{23}H_{17}N_3O_3$	C_5H_4NCO	117^{0}C	35%	10.91	10.97	0.84



Synthesis of 1-(1-hydroxy naphthalen-2-yl)-3-(furan-2-yl) prop-2-ene-1-one (3)

2-acetyl-1-naphthol (0.01mole) and furfuraldehyde (0.02 mole) were added in ethanol solvent (20ml). To this mixture KOH (10%, 10ml) solution was added dropwise with constant stirring. The reaction mixture was kept overnight. Then the mixture was poured over crushed ice & little HCl. The product was filtered and recrystallized from ethanol to obtain the compounds (3). The physical data is given in Table 1

3: IR (KBr):1650 (C=O), 3412(-OH), 1155(C-O-C) NMR ($CDCl_3$ + DMSO-d6) : δ 6.98-7.46 (m,9H, Ar-H), δ 8.057 (d,1H =CH) , δ 8.099(d,1H =CH) δ 9.63 (S,1H,-OH)

Synthesis of 3 - (1-hydroxy naphthalen – 2 – yl)-5-(furan-2-yl) - 1-substituted pyrazolines (4-8)

1-(1-hydroxy naphthalen-2-yl)-3-(furan-2-yl) prop-2-ene-1-ones (0.01mole) & hydrazine / phenyl hydrazine/ semicarbazide / 2,4 dinitro phenyl hydrazine / isonicotinic acid hydrazide (0.01 mole) were added to DMF (20 ml) 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 titled pyrazolines in 45-51% yield . The physical data is given in Table 1

4: IR (KBr):3000 (N-H), 3402(-OH), 1151(C-O-C) NMR ($CDCl_3$ + DMSO-d6) : δ 6.90-7.80 (m,6H,
Ar-H), δ 8.051 (d,1H =CH), δ 8.091(d,1H =CH) δ 9.62 (S,1H,-OH)

5: IR (KBr):1600 (C=C), 3410(-OH), 1148(C-O-C) NMR ($CDCl_3$ + DMSO-d6) :δ 6.89-7.90 (m,11H, Ar-H), δ 8.049 (d,1H =CH) , δ 8.081(d,1H =CH) δ 9.53 (S,1H,-OH)

6: IR (KBr):1723 (C=O), 3405(-OH), 1160(C-O-C) NMR ($CDCl_3$ + DMSO-d6) : δ 6.81-7.40 (m,6H, Ar-H), δ 8.041 (d,1H =CH) , δ 8.079(d,1H =CH) δ 9.49 (S,1H,-OH), 6.6 (S,2H,NH_2)

7:IR(KBr):1530,1550(NO_2),3412(OH), 1155(C-O-C) NMR ($CDCl_3$ + DMSO-d6) : δ 6.81-7.60 (m,9H, Ar-H), δ 8.045 (d,1H =CH) , δ 8.081(d,1H =CH) δ 9.43 (S,1H,-OH)

8: IR (KBr):1611 (C=O), 3477(-OH), 1170(C-O-C) NMR($CDCl_3$ + DMSO-d6) : δ 6.95-7.81 (m,10H, Ar-H), δ 8.043 (d,1H =CH) , δ 8.083(d,1H =CH) δ 9.50 (S,1H,-OH)

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