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Synthesis of 3-(2'-hydroxy-3'-nitro-5'-methylphenyl)-5-(aryl/heteryl) -2-pyrazoles

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ABSTRACT

The present study deals with the synthesis and characteristics of 3-(2'-hydroxy-3'-nitro-5'-methylphenyl)-5-(aryl/heteryl) pyrazoles synthesized from 1-(2'-hydroxy-3'-nitro-5'-methylphenyl)-3-aryl/heteryl-2-propen-1-ones by reaction with hydrazine hydrate in ethanol. The structures of synthesized compounds have been established by spectral (IR, NMR, etc.) and elemental analysis.

Key words: Chalcone, pyrazoles.

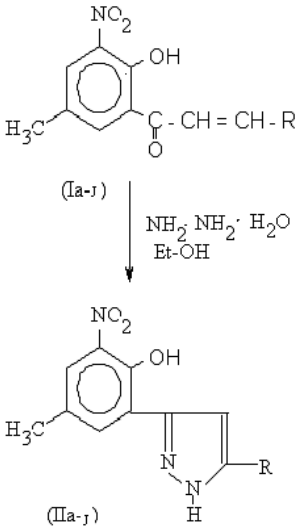
Pyrazoles have been studied because of their wide range biological and pharmacological activities. These compounds have been found to be effective as antimicrobial, antiinflamatoryl[1], herebicidals[2], antibacterial[3] etc. The diverse properties of pyrazoles have promoted to synthesis some new pyrazoles.

Melting points are uncorrected. The IR spectra of some of the representative compounds from the series were recorded on PERKIN ELMER IR Spectrometer -450. The NMR spectra of few representative compounds were studied in $CDCl_3$ on Bruker Avance II 400 NMR Spectrometer using TMS as internal standard.Purity of compounds was checked by TLC.

Synthesis-3-(2-Hydroxy-3-Nitro-5-MethylPhenyl)-5-(Aryl/Heteryl)-2-Pyrazoles.

1-(2'-hydroxy-3'-nitro-5'-methylphenyl)-3-phenyl-2-propen-1-one (0.01 mole) treated with hydrazine hydrate (0.012 mole) in 25 ml of ethanol and reaction mixture was refluxed for 2-3 hours. Then reaction mixture was cooled, poured in ice cold water. The separated solid product was filtered washed with water, dried and recrystallized from proper solvent. Similarly all the other compounds of the series were also prepared by the above procedure. The IR spectra shows the presence of absorption band in the region 3600-3300 cm^{-1} for (N-H) stretching vibrations characteristic band of pyrazole ring. The absorption at 1600 cm^{-1} is due to C=N stretching. The absorption in the region 3360-3380 cm^{-1} is due to –OH group.

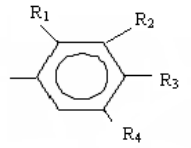
The NMR spectra of 3-(2-hydroxy-3-nitro-5-methyl phenyl)-5-(p-dimethylamino phenyl)-2–pyrazole exhibited signals at δ NMR (δ ppm): The 6 protons of dimethyl amino [-N $(CH_3)_2$] were observed at 3.06 δ. The aromatic protons were absorbed at 6.81-7.91 δ and the signal due to phenolic (-OH) proton was seen at 9.82δ (s).



Scheme 1

Table 1

R =



S. No.	Compounds	R_1	R_2	R_3	R_4
1	IIa	-H	-H	$-N(CH_3)_2$	-H
2	IIb	-H	-H	-H	-H
3	IIc	$-NO_2$	-H	-H	-H
4	IId	-H	-H	$-OCH_3$	-H
5	IIe	-Cl	-H	-H	-H
6	IIf	-H	-H	-Cl	-H
7	IIg	In IIg, **R**= Furfuryl Ring			
8	IIh	-H	$-CH_2-O-CH_2-$	-H	
9	IIi	-H	-H	-OH	-H
10	IIj	-H	-OH	-H	-H

Table 2: Characterization data of 3-(2′-hydroxy-3′-nitro-5′-methylphenyl)-5-(Aryl/Heteryl) -2-Pyrazoles

S. No.	Comp. No.	M. P. ^0C	Yield %	Molecular formula	Anal. found (Calcd) % Nitrogen
1.	IIa	270	80	$C_{18}H_{18}O_3N_4$	9.20 (9.42)
2.	IIb	280	70	$C_{16}H_{13}O_3N_3$	6.05 (6.75)
3.	IIc	240	75	$C_{16}H_{12}O_5N_4$	12.05 (12.21)
4.	IId	221	72	$C_{17}H_{15}O_3N_3$	9.80 (9.85)
5.	IIe	116	76	$C_{16}H_{12}O_3N_3Cl$	7.20 (7.37)
6.	IIf	180	66	$C_{16}H_{12}O_3N_3Cl$	7.20 (7.37)
7.	IIg	153	72	$C_{14}H_{11}O_4N_2$	8.20 (8.37)
8.	IIh	285	56	$C_{17}H_{14}O_6N_2$	8.28 (8.35)
9.	IIi	145	62	$C_{16}H_{13}O_4N_3$	5.48 (5.87)
10.	IIj	132	78	$C_{16}H_{13}O_4N_3$	5.48 (5.87)

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