Oriental Journal of Chemistry Vol. **24**(2), 775-776 (2008)

Microwave-assisted synthesis of 3-(2'-hydroxy-3'-nitro-5'-methylphenyl)-5-(aryl/heteryl) isoxazolines

S.B. BORUL[1], R.U. PATHAN[2] and S.V. AGARKAR[3]

Anuradha Engineering College, Chikhli-443201 Dist- Buldana (India)
[2]Rahimkhan Usmankhan Pathan, At post- Mangrul Navghare,
Tq- Chikhli, Dist- Buldana - 444 301 (India).
[3]Suryakant Bhanudas Borul, At post- Isrul,Tq -Chikhli, Dist- Buldana - 443 201 (India).

(Received: May 11, 2008; Accepted: June 23, 2008)

ABSTRACT

The present study deals with the microwave-assisted synthesis and characteristics of 3-(2'-hydroxy-3'-nitro-5'-methylphenyl)-5-(aryl/heteryl) isoxazolines synthesized from 1-(2'-hydroxy-3'-nitro-5'-methylphenyl)-3-aryl/heteryl-2-propen-ones by reaction with hydroxylamine hydrochloride and sodium acetate in ethanol. All synthesized compounds characterized by IR, and NMR.

Key words: Microwave, Chalcone, Isoxazoline.

INTRODUCTION

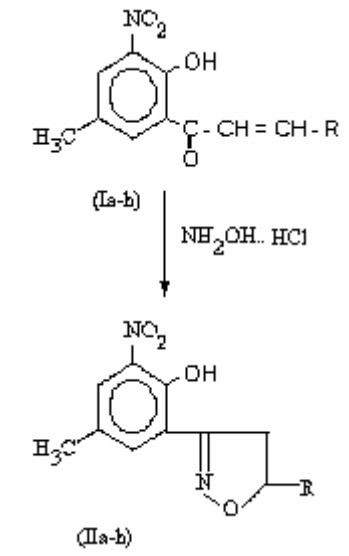
Isoxazolines possesses wide range biological and pharmacological activities. These compounds have been found to be effective as antimicrobial, antiinflamatoryl[1], herebicidals[2], antibacterial[3] etc. The diverse properties of isoxazolines have promoted to synthesize some new isoxazolines.

EXPERIMENTAL

IR spectra were recorded on Perkin-Elmer Infrared spectrophotometer .NMR spectra were recorded on Bruker Avance-II 400MHz spectrophotometer. All reagents used were of analar grade.

***Synthesis3-(2-Hydroxy-3-Nitro-5-MethylPhenyl)-5-(Aryl/Heteryl) Isoxazolines.**

1-(2'-hydroxy-3'-nitro-5'-methylphenyl)-3-phenyl-2-propen-1-one (0.01 mole) treated with hydroxylamine hydrochloride (0.0015 mole) and sodium acetate (0.002 mole) in 15 ml of ethanol and reaction mixture was irradiated under microwave at 640 power. Then reaction mixture was cooled,



Scheme 1

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 bond at 1260 cm^{-1} has been assign to N-O-C stretching vibrations. The absorption at 1465 cm^{-1} is characteristic bond of isoxazoline 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.

Table 1: Characterization data of 3-(2'-hydroxy-3'-nitro-5'- methyl phenyl)-5- (aryl/heteryl) Isoxazolines

S. No.	Entries	Molecular formula	R	m.p. (°C)	Yield (%)	Time required (Min.)
1.	IIa	$C_{18}H_{19}O_4N_3$	Dimethyl amino Phenyl	126	84	4
2.	IIb	$C_{16}H_{14}O_4N_2$	Phenyl	192	78	2
3.	IIc	$C_{16}H_{13}O4N_3$	2-Nitro Phenyl	205	88	3
4.	IId	$C_{17}H_{16}O_5N_2$	P-methoxy phenyl	210	86	2.5
5.	IIe	$C_{16}H_{12}O_4N_2Cl$	2-chloro Phenyl	178	80	3
6.	IIf	$C_{16}H_{12}O_4N_2Cl$	4-chloro Phenyl	184	70	1.5
7.	IIg	$C_{14}H_{12}O_5N_2$	Furfuryl	102	75	4
8.	IIh	$C_{17}H_{14}O_6N_2$	PiperonalPhenyl	187	77	3
9.	IIi	$C_{16}H_{13}O_5N_2$	P-hydroxyPhenyl	110	70	2
10.	IIj	$C_{16}H_{13}O_5N_2$	M-hydroxyPhenyl	168	92	4

The NMR spectra of 3-(2-hydroxy-3-nitro-5-methyl phenyl)-5-(p-dimethylamino phenyl) –isoxazolines exhibited signals at δ NMR (δ ppm):2.47δ (s, 3H –CH_3); 3.23δ (dd, 1 –CH H_A); 3.64δ (dd, 1 –CH H_B); 4.90δ (dd, 1H –CH H_X); 3.06δ (s, 6H –$N(CH_3)_2$); 6.8-8.5δ (m, 6H Ar-H); 12.6δ (s, 1H -OH).

R=

P-Dimethylamino, Phenyl, Furfural, O-Nitro phenyl, P-Methoxy Phenyl, 2-Chloro Phenyl, 4-Chloro phenyl, Piperonal

ACKNOWLEDGEMENTS

The authors are thankful to Dr.J. D. Dhake Tech. Director Anuradha Engineering College Chikhli for providing necessary laboratory facilities.

REFERENCES

1. B. Shivkumar, Nargund, *Indian J. Heterocycl. Chem.* **8**(1): 27-30 (1998).
2. V. R. Naik, *Asian J. Chem.,* **12**(4): 1358-13609 (2000), *Chem. Abstr.* **16**: 222650m, 134 (2001).
3. Kucherov V. F. J. Gen Chem., USSR, **21**: 1145 (1951). *Chem. Abstr.* **46**: 5043 (1952).
4. Shur M and Israelstam S.S., *J. Chem,* **33**: 3015 (1968).
5. Caddick S, Tetrahedron, **51**: 10403 (1995).
6. Hayes B L, Microwave Synthesis: Chemistry at the speed of light, (CEM Publishing, USA), 2002.
7. Rajanarendar E and Ramu K, *Indian J Heterocyclic Chem.,* **13**: 73 (2001).