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MICROWAVE SYNTHESIS OF 3-(2'-HYDROXY-5'-METHYL-PHENYL)-5-ARYL –ISOXAZOLES

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ABSTRACT

ß-diketone obtained by esterification of substituted acetophenone with aromatic acids followed by B.V. Transformation, which on microwave irradiation with hydroxylamine hydrochloride , give 3 - (2'– Hydroxy - 5' – Methyl – Phenyl)-5-Aryl – Isoxazoles (2a-f). The structures of these compounds were established by elemental, chemical and spectral analysis (IR, NMR). The properties of these compounds are found to be similar to the compounds obtained by refluxing method.

Key Words: *Microwave Synthesis, Isoxazoles.*

INTRODUCTION

Environmental pollution is the biggest challenge of the present era. It is the duty of every individual to protect the surrounding area somehow or the other. To promote the environment friendly process, it has been emphasized to use alternate way of the synthesis of the organic compounds. Microwave dielectric heating[1-2] is not only avoid environmental pollution[3-6] but also enhances the rate of the reactions. Since last decade the reaction rate has been accelerated by using this method.

Isoxazoles play an important role in the pharmaceuticals, possessing antifungal[7], and antitubercular activity[8] . Hence it was thought to synthesize substituted pyrazoles by heating with microwave radiation.

ß-diketones and Hydroxy Chalcones are best precursors of the Isoxazole derivatives in different solvents like pyridine, DMF, ethanol, acetic acid etc. obtained by the refluxing method. In continuation of research work present work deals with the Microwave synthesis of 3-(2'-Hydroxy-5'-Methyl-Phenyl)-5-Aryl –Isoxazoles (2a-f) and their characterization by elemental analysis, IR, ^{1}H NMR analysis and comparison their properties with the compounds synthesize by the usual method[9-15].

EXPERIMENTAL

The melting points of the synthesized compounds 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 Brucker AC300 FNMR spectrometer (300MHz), using TMS as an internal standard. Microanalysis of nitrogen was obtained on colman 29-N analyzer. The purity of the compounds was checked by Thin Layer Chromatography on silica gel-G.

Preparation of 1,3-Diaryl-propane-1,3-dione (1a): 2'-hydroxy-5'-methyl-acetophenone esterifies with benzoic acid to give the product which give 1,3-Diaryl-propane-1,3-dione (1a) by B.V. transformation. The product was washed and recrystallized with ethanol. The structures of these compounds were confirmed by chemical and spectral analysis. m.p.146^{0}C. Yield (70 %).

Spectral interpretation of (1a):

IR (ν_{max}) (cm^{-1}): 1549 v(-CH=CH-), 1646 v(C=O), 1186 v(-C-O stretching),

NMR δ ppm: 2.32 (s, 3H, CH_3) , 2.53 (s, 2H, $-CH_2-$), 13.61 (s, 1 H, -OH), 6.71-7.89 (m, 8H, Ar-H),

Similarly 1,3-Diaryl-propane-1,3-diones (1b-f) were prepared and their physical data is given in Table-1.

Preparation of 3-(2'-Hydroxy-5'-Methyl-Phenyl)-5-Phenyl –Isoxazoles (2a):

1,3-Diaryl-propane-1,3-dione (1a) mixed hydroxylamine hydrochloride (1:1) and irradiated with microwave radiation, in house hold 2450 Hz Microwave oven at 600 watts for 3 minutes to give 3-(2'-Hydroxy-5'-Methyl-Phenyl)-5-Phenyl –Isoxazole (2a). The product was washed and recrystallized with ethanol. The structures of these compounds were confirmed by chemical and spectral analysis. m.p.184 ^{0}C. Yield (88 %).

Spectral interpretation of (2a)

IR (ν_{max}) (cm^{-1}): 3156 cm^- v (-OH phenolic), 2861 v (-CH), 1638 v (>C=N), 1472 v (-C=C-), 1290 v (-C-O).

NMR δ ppm: 2.34 (s, 3H, $-CH_3$) , 3.68 (s,1H, -CH) , 6.75-7.57 (m, 8 H, Ar-H), 10.2 (s, 1H , Ar-OH).

Similarly 3-(2'-Hydroxy-5'-Methyl-Phenyl)-5-Aryl –Isoxazoles (2b-f) were prepared and their physical data is given in Table-2.

RESULTS AND DISCUSSION

Isoxazoles obtained from ß-diketones and hydroxylamine hydrochloride by microwave irradiation found same characteristics to that of compounds prepared by refluxing method . The rate of the organic reaction is accelerated and product obtained in 4 minutes which was 6 hours in the routine method. % Yield of the product obtained was also found more than the usual method. Hence this method is quite beneficial to the usual method as it avoid environmental pollution.

Table-1: PHYSICAL DATA OF ß-DIKETONES .

Compound (Mol. Formula)	R_1	R_2	R_3	m. p ^{0}C	Yield %
1a ($C_{16}H_{14}O_3$)	H	H	H	146	70
1b ($C_{16}H_{13}O_3Br$)	Br	H	H	153	73
1c ($C_{16}H_{13}O_3Cl$)	Cl	H	H	168	69
1d ($C_{16}H_{13}O_3Cl$)	H	H	Cl	180	77
1e ($C_{17}H_{16}O_4$)	OCH_3	H	H	167	64
1f ($C_{17}H_{16}O_4$)	H	H	OCH_3	170	74

Table- 2:PHYSICAL DATA OF ISOXAZOLES.

Compound (Mol. Formula)	R_1	R_2	R_3	m. p	Yield Old (M/W)	%N
				^{0}C	%	Found (Calculated)
2a ($C_{16}H_{13}O_2N$)	H	H	H	184	75 (88)	5.58 (5.51)
2b ($C_{16}H_{12}O_2NBr$)	Br	H	H	193	78 (86)	4.24(4.19)
2c ($C_{16}H_{12}O_2NCl$)	Cl	H	H	173	69 (84)	4.90 (4.98)
2d ($C_{16}H_{12}O_2NCl$)	H	H	Cl	178	76 (86)	4.90 (4.87)
2e ($C_{17}H_{15}O_3N$)	OCH_3	H	H	196	71 (90)	4.98 (4.87)
2f ($C_{17}H_{15}O_3N$)	H	H	OCH_3	207	79 (87)	4.98 (4.91)

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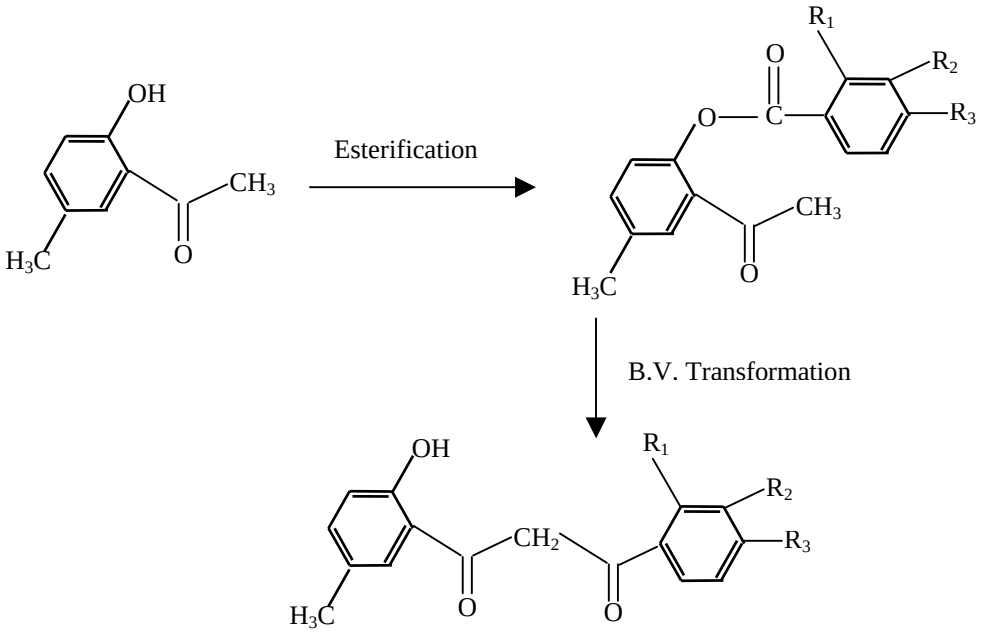
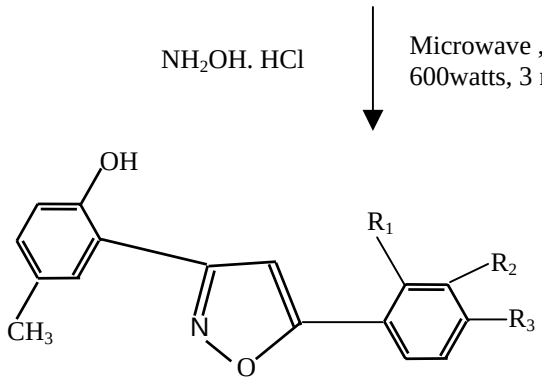
SCHEME I



(1a-f)



(2a-f)

R_1= -H, -OCH_3 ,-Cl ,-Br , R_2= -H, , R_3= -H, -OCH_3 ,-Cl .

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