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Der Pharma Chemica, 2013, 5(1):284-286
(http://derpharmachemica.com/archive.html)

ISSN 0975-413X
CODEN (USA): PCHHAX

Microwave assisted synthesis of styrylisoxazoles under solvent free conditions

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ABSTRACT

Keeping in view of utility of Microwave induced Organic Enhancement (MORE) chemistry, microwave induced preparation of 1- (2-hydroxy phenyl) -5- phenyl - 4-pentene- 1,3- diones in a single pot reaction is reported which on condensation with hydroxylamine hydrochloride under microwave irradiation afforded 3- (2- hydroxyphenyl) –5- styrylisoxazoles in only 100 sec. These compound were characterized on the basis of elemental analysis, IR and NMR spectral data.

Keywords: Styrylisoxazoles, Triton B, Green chemistry, Solvent free conditions, Microwave irradiations

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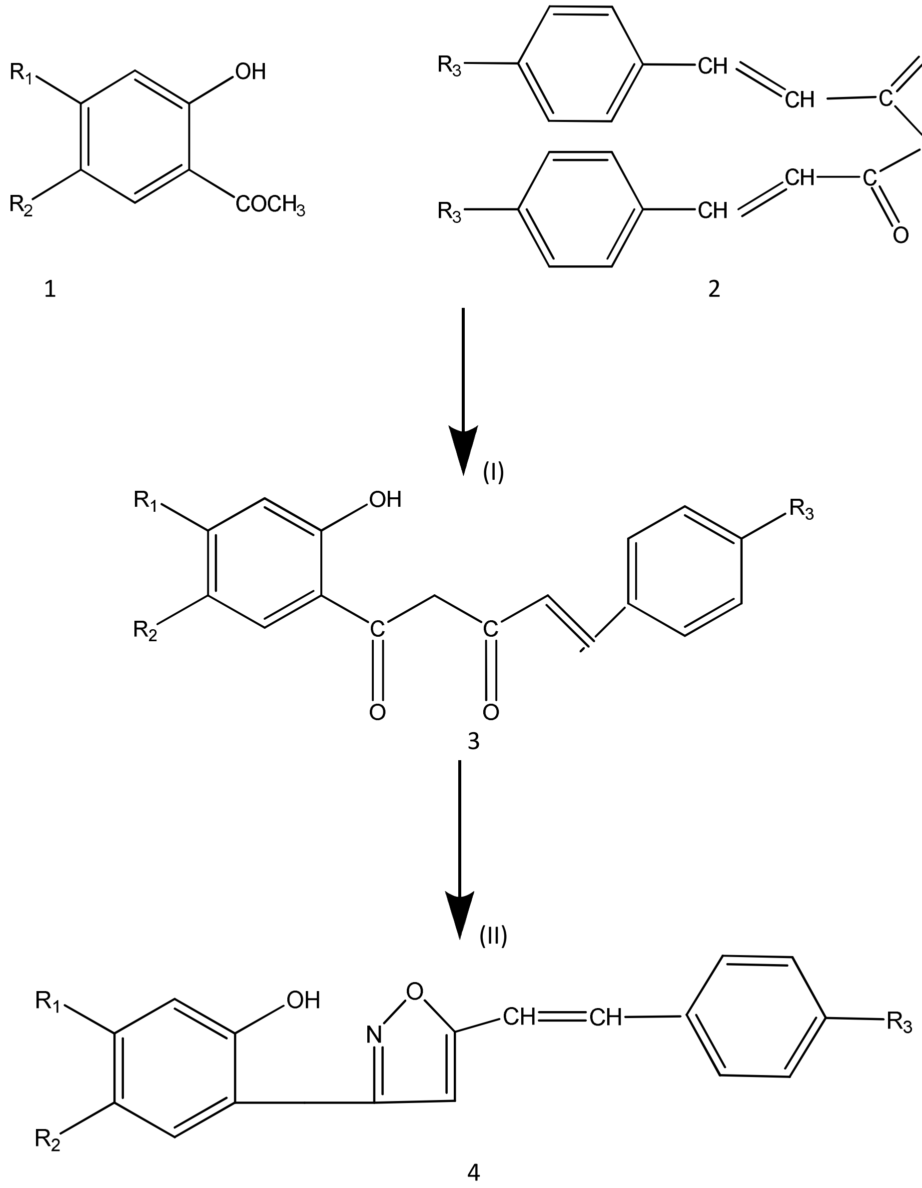
Green and sustainable chemistry has been subject of intensive research in recent years and the studies in this area have led to development of cleaner and relatively benign chemical processes [1,2]. In these much effort has been devoted to microwave assisted organic synthesis (MAOS) which leads to significantly reduced reaction times, enhanced conversions and is environmentally friendly [3-5]. Further isoxazole nucleus is associated with an important class of heterocycles which display interesting biological properties such as antiinflamatory, anti-depressant, antiviral and so on [6-8]. This nucleus alongwith styryl group is likely to be more biologically active. Prompted by these observation a series of styrylisoxazoles were synthesised. These compounds were usually obtained by refluxing 1- (2-hydroxy phenyl) -5- phenyl pent – 4 – ene – 1, 3 – dione with hydroxylamine hydrochloride in pyridine for 5hr [9]. Now first the diones were obtained in a single pot reaction under microwave irradiations [10] and then a simple method had been developed for the condensation of these diones with hydroxylamine hydrochloride to give styrylisoxazoles under microwave irradiations in 90-95% yield.

MATERIALS AND METHODS

Melting points were determined in open capillary tubes and are uncorrected. IR spectra were recorded on Perkin-Elmer Spectrum BX-series FTIR, ^{1}H NMR spectra on Bruker Avance II 400 MHz NMR spectrometer using tetramethylsilane as internal standard and chemical shift values are reported in δ scale. The reaction was carried out in domestic microwave oven (Samsung, Model No. CE II8 KF, output energy 900W, frequency 2450 MHz) using 30% power for all experiments.

General procedure for microwave assisted synthesis of 3- (2- hydroxyphenyl) – 5 - styrylisoxazoles.

A mixture of 1- (2-hydroxy phenyl) – 5 – phenyl pent - 4 – ene – 1, 3-dione (5 mmol) and hydroxylamine hydrochloride (10 mmol) and the base i.e. triton-B adsorbed on flyash (50% composition) was prepared by adding few drops of acetone, air dried and it was taken in a loosely stoppered 100ml R B flask. Then the mixture was subjected to microwave irradiations. Completion of the reaction was checked on TLC and the reaction mixture was dissolved in chloroform. Organic layer was filtered to remove flyash and solvent was distilled off from filterate. The residue was washed with water, dried and recrystalized from methanol to get the desired product. (Scheme -1)



(I) $Ba(OH)_2$ / DMSO (II) $NH_2OH.HCl$

Scheme - 1

Spectral data of compounds 4a to 4f

4a: (R_1= R_2 = R_3 = H), m. p 216 - 17°, (Found C, 77.5; H, 4.6; N, 5.0. $C_{17}H_{13}O_2N$ requires C, 77.6; H, 4.9; N, 5.3%). IR (KBr): 3100, 1640, 1620, 1510, 1450, 1320, 1250, 1130, 950, 800. ^{1}HNMR: 6.80 – 7.50 (12H, m, Ar-H and -CH = CH-) and 9.80 (1 H, s, Ar- OH).

4b: (R_1 = R_3 = H, R_2 = CH_3), m.p 196°, (Found C, 77.6, H, 5.0, N, 4.8. $C_{18}H_{15}O_2N$ requires C, 78.0; H, 5.4; N, 5.1%). ^{1}H NMR: 2.30 (3H, s, - CH_3), 6.95- 7.60 (11H, m, Ar-H and -CH = CH-) and 9.90 (1 H, s, Ar- OH).

4c: (R_1= H, R_2= CH_3, R_3= OCH_3), m.p 197^0, (Found: C, 74.1; H, 5.3; N 4.3. $C_{19}H_{17}O_3N$ requires C, 74.3; H, 5.5, N; 4.6%) ^{1}HNMR: 2.33 (3H, s, - CH_3), 3.60 (3H, s, OCH_3) 6.80 – 7.55 (10H, m, Ar- H and –CH = CH-) and 9.85 (1H, s, Ar-OH)

4d: (R_1 = R_2 = H, R_3 = OCH_3), m.p. 195^0, (Found : C, 73.2; H, 4.6; N, 4.7. $C_{18}H_{15}O_3N$ requires C, 73.7; H, 5.1; N, 4.8%), ^{1}HNMR: 3.50 (3H, s, OCH_3); 6.80-7.50 (11H, m, Ar-H and –CH= CH-) and 9.80 (1H, s, Ar-OH)

4e: (R_1 = CH_3, R_2= R_3 = H), m.p. 225-26^0, (Found : C, 77.5 ; H, 5.0; N, 4.7. $C_{18}H_{15}O_2N$, requires C, 78.0; H, 5.4; N, 5.1%), ^{1}HNMR: 2.42 (3H, s, CH_3), 6.90-7.65 (11 H, m., Ar-H and -CH=CH-) and 9.85 (1H, s, Ar-OH)

4f: (R_1 CH_3, R_2 = H, R_3= OCH_3). m.p. 217-18^0. (Found : C, 74.0; H, 5.1; N, 4.5. $C_{19}H_{17}O_3N$ requires C, 74.3; H, 5.5; N, 4.6%). ^{1}HNMR: 2.45 (3H, s, CH_3), 3.50 (3H, s, OCH_3), 6.85-7.55 (10H, m., Ar-H and -CH=CH-) and 9.80 (1H, s, Ar-OH)

CONCLUSION

The synthetic procedure to get 3- (2- hydroxyphenyl) -5- styrylisoxazoles using microwave irradiation proved to be highly efficient and convenient method for preparation of this important class of compounds. This method is very much superior to previous conventional heating procedure where the reaction takes much longer time using hazardous solvents and yields are significant lower. It is important to say here that shortening of reaction time and improvement in yields in addition to simplicity of procedures obtained with this method may be of importance for the industrial and large scale production of these compounds.

Acknowledgement

The author is thankful to Maharshi Dayanand University Rohtak, India for providing necessary facilities.

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