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A simple and efficient one step synthesis of 1,3,4-oxadiazoles utilizing polymer-supported reagents and microwave heating

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ABSTRACT

2,5 – disubstituted 1,3,4 –oxadiazoles are synthesized from hydrazine and the corresponding aromatic acids both in the presence and in the absence of clay catalyst.

Key words: 1,3,4-oxadiazole, Microwave irradiation.

INTRODUCTION

1,3,4-Oxadiazoles are a class of heterocycles which have attracted significant interest in medicinal and pesticide chemistry, and polymer and material science[1,2]. Among the 1,3,4-oxadiazoles 2,5-unsymmetrical disubstituted derivatives have attracted considerable attention because of their biological[3] and electrochemical properties[3]. The common synthetic route of these compounds involves cyclisation of diacyl hydrazines with a variety of anhydrous reagents such as thionyl chloride[6], phosphorous pentachloride[7], phosphorous oxy chloride[8], triflic anhydride[9], triphenyl phosphine, polyphosphoric acid[10] and sulphuric acid. The acylation of tetrazoles[11] is another synthetic route of these compounds. They have also been prepared by oxidation of acyl hydrazone with different oxidizing agents.

The title compounds have been synthesized by reacting hydrazides with aromatic carboxylic acid in presence of $POCl_3$ under solvent free conditions using microwave energy. Aromatic hydrazide (1mmole) and aromatic acids (1mmole) were reacted together in presence of $POCl_3$ (1mmole), mixed and irradiated in the microwave oven using 400W power both in absence and presence of catalyst. Cold water (50 ml) was added into the crude product and the product was filtered off and purified by column chromatography.

2-(4'-methoxy phenyl)-5-phenyl 1,3,4-oxadiazole

M.pt 101–102 °C;yield 91%; R_f = 0.37 (ethylacetate–acetone, 8:2); UV (methanol):λ_{max} (log Σ) 262 (2.30) nm^{-1}; IR (KBr) ν_{max}: 3067 (C–H), 1662 (C=N), 1568 (C=C), 1283 (C–O).^{1}H NMR (400 MHz, $CDCl_3$): δ 9.20 (d, 1H, J = 1.1 Hz, H-2'),9.10 (dd, 1H, J = 4.9, 1.6 Hz, H-6'), 8.83 (br d, 1H, J = 8.3 Hz, H-4'), 8.59 (dd, 1H, J = 4.9, 8.3 Hz, H-5'),7.76 (dd, 2H, J = 8.4, 1.2 Hz, H-3"/5"), 7.57 (dd, 2H,J = 8.4, 1.3 Hz, H-2"/6").

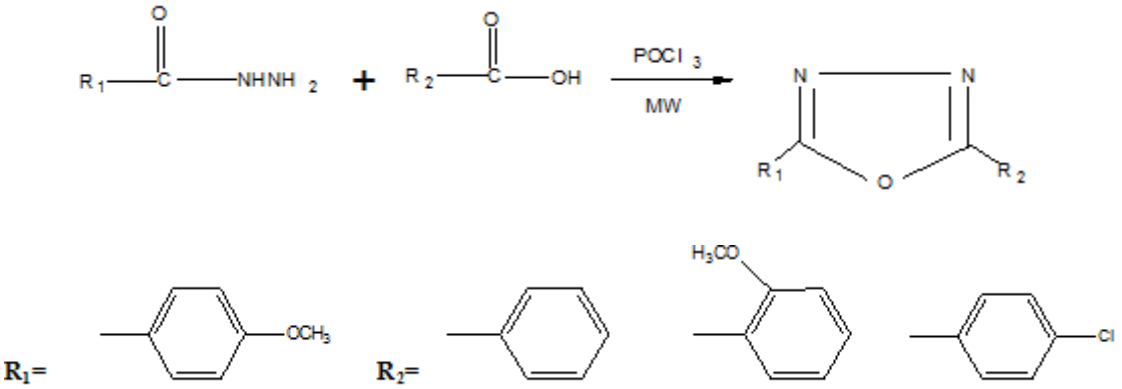
2-(4'-methoxy phenyl)-5-(4'-chloro phenyl) 1,3,4-oxadiazole

M.pt 112–113°C;yield 92%; R_f = 0.34 (ethyl acetate–acetone, 9:1); UV (methanol): λ_{max} (log Σ) 255 (2.32) nm^{-1};IR (KBr) ν_{max}: 3072 (C–H), 1666 (C=N), 1556 (C=C), 1286 (C–O), 831, 658 (C–Cl); ^{1}H NMR (400 MHz, DMSO-d6): δ9.22 (d, 1H, J = 1.1 Hz, H-2'), 9.14 (dd, 1H, $J_{6',5'}$ = 4.9 Hz, H-6'), 8.82 (br d, $J_{4',5'}$ = 8.3 Hz, 1H, H-4'), 8.60 (dd, 1H,J5',

6' = 4.9, $J_{5',4'}$ = 8.3 Hz, H-5'), 7.64 (dd, 2H, $J_{2'',3''/6'',5''}$ = 7.8, $J_{2'',4''/6'',4''}$ = 2.3 Hz,H-2"/H-6"), 7.51 (t, 1H, J = 7.8 Hz, H-4"), 7.39 (t, 2H,J = 7.8, H-3"/5").

2-(4'-methoxy phenyl)-5-(2'-methoxy phenyl) 1,3,4-oxadiazole

M.pt 102–103°C; yield 88% ;R_f = 0.43



Scheme 1:

(ethyl acetate–acetone, 9:1); UV (methanol): λ_{max} (log Σ) 206 (2.41) nm^{-1}; IR (KBr) ν_{max}: 3062 (C–H), 2911 (C–H),1662 (C=N), 1560 (C=C), 1282 (C–O), 607; ^{1}H NMR (400 MHz, DMSO-d6): ä9.13 (d, 1H, J = 1.1 Hz, H-2'), 9.03 (br d, 1H, J = 4.9 Hz, H-6'), 8.77 (br d, 1H,J = 8.3 Hz, H-4'), 8.53 (dd, 1H, J = 4.9, 8.3 Hz, H-5'), 7.48 (br d, 2H, J = 7.8 Hz, H-2"/6"), 7.31 (d, 2H,J = 7.8 Hz, H-3"/5");´ 3.76(s ,3H).

CONCLUSION

Better results were obtained with catalyst also the reaction time was reduced considerably.

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