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A NOVEL AND CONVENIENT METHOD FOR THE SYNTHESIS OF 3, 5-DIARYLISOXAZOLES

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Abstract: α,β-Unsaturated oximes of chalcones on treatment with 2,3-Dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) in methanol furnish 3,5-disubstituted isoxazoles in good yield.

Isoxazoles are important class of heterocycles used as intermediates for natural product synthesis and building blocks for construction of new molecular system[1]. Various methods comprising α,β-unsaturated oximes have been adopted for the synthesis of isoxazoles. These methods include iodine/potassium iodide[2], N-bromosuccinimide[3], Palladium Complex in presence of sodium phenoxide[4], Lead(IV)acetate[5] and tetralin(pyridine)Cobalt(II)dichromate (TPCD)[6] as reagents. Herein we report a novel convenient procedure (Scheme 1) for the synthesis of 3,5-diarylisoxazoles by oxidative cyclisation of chalcone oximes by 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) in methanol. A total of six 3,5-diarylisoxazoles were prepared in 60-75% under same conditions from the corresponding substituted chalcone oximes (Table 1).

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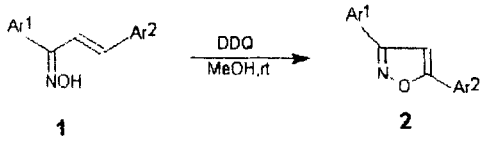

1 2

1,2	Ar^1	Ar^2	1,2	Ar^1	Ar^2
a	Ph	Ph	d	$4\text{-}CH_3OC_6H_4$	Ph
b	Ph	$3,4(OCH_2O)C_6H_3$	e	$4\text{-}CH_3OC_6H_4$	$4\text{-}CH_3OC_6H_4$
c	Ph	PhCH=CH	f	$4\text{-}CH_3OC_6H_4$	$3,4(OCH_2O)C_6H_3$

Scheme 1

Table 1. 3,5 –diarylisoxazoles **2a-f** Prepared

Product	Yield(%)	mp(°C)	Mol.F or lit.mp(°C)
2a	75	140	141[6]
2b	72	120	120[6]
2c	70	130	134[6]
2d	74	120	120-121[8]
2e	64	140	141-142[9]
2f	71	152	$C_{16}H_{15}NO_4$ (295)

However, extension of this methodology for oxidative cyclisation of cinnamaldehyde oxime, benzalacetone oxime and β-ionone failed. Thus, probably for effective cyclisation with DDQ presence of two phenyl groups at 1,3-positions of chalcones is necessary. A report[7] using DDQ for oxidative conversion of isoxazolidines to isoxazolines has recently appeared. In conclusion, the reaction presents a general and a convenient procedure for the

Table 2. Spectroscopic Data of New Compound **2f**

Compound[a]	IR (KBr) ν (cm^{-1})	^{1}H NMR ($CDCl_3$/TMS) δ,J(Hz)	^{13}C NMR ($CDCl_3$/TMS) δ	MS m/z (%)
2f	1619, 1514, 1450, 1269, 1058, 813	3.86(s,3H,OCH_3), 6.04(s,2H,OCH_2O), 6.63(s,1H,CH), 6.88(d,2H,9Hz), 6.99(d,2H,9Hz), 7.26-7.78(m,3H,ArH)	55.43, 77.10, 96.39, 101.68, 106.23, 108.88, 114.37, 120.52, 121.74, 121.80, 128.25, 148.30, 149.30, 161.06, 162.64, 169.64	149(100), 239, 280(4), 295(M^+,52)

[a]Satisfactory microanalysis were obtained

preparation of 3,5-diarylisoxazoles. Comparing with other methods mentioned above our method is a very mild method with known commercial reagent and in good yields.

All melting points are uncorrected and measured by normal thiels tube method. IR spectra were recorded on a FT-IR spectrophotometer. ^{1}H NMR were recorded at 300mhz.

EXPERIMENTAL

Preparation of Isoxazoles 2; General Procedure:

A mixture of chalcone- oxime **1**(1 mmol), DDQ(2 mmol) was stirred overnight in methanol (5 mL). The reaction mixture was concentrated in vacuo. The residue was taken up in CH_2Cl_2 (20 mL) and washed with 2M NaOH. The organic phase was dried (Na_2SO_4) and concentrated. Purification column chromatography over silica gel with petroleum ether/ethyl acetate (4:1) furnished the product **2**. For analysis, the product was recrystallised using an appropriate solvent.

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REFERENCES:

1 (a)Dominguez, E.; Ibeus, E.; Matrinez de margorta, E.; Palacious, J. K.; San Marka, E. *J.Org.Chem.* **1996**, 61, 5435. (b)Buron, C.; Kairon, L.E.; Hslu, A. *Tetrahedron lett.* **1997**, 38, 8027. (c)Grunanger, P.; Vitafinzi, P. *Isoxazoles In the Chemistry of Heterocyclic compounds*, Taylor, E.C.; Ed., Wiley Interscience: New York, **1991**; Vol. part I, pp 1-416.

2 Buchi, G.; Vederas, J.C. *J.Am.Chem.Soc.* **1972**, 94, 9128.

3 Maeda, K.; Hosokawa, T.; Murahashi, S.I.; Moritani, J. *Tetrahedron Lett.***1973**, 51, 5075.

4 Hanson, J.F.;Strong, S.A. *J.Heterocycles Chem.* **1977**, 14, 1289.

5 Sharma, J.C.; Roginder, S.; Berger, D.D.; Kale. A.V. *Indian J.Chem.***1986**, 25B, 437.

6 Wei, X.; Fang, J.; Hu, Y.; Hu, H. *Synthesis* **1992**, 1205.

7 Li, P.; Gi, H-J.; Zhao, K. *J.Org Chem.* **1998**, 63, 336.

8 Beam, C.F.; Dyer, C.D.M.; Schwarz, R.A.and Hauser, C.R. *J.Org.Chem.* **1970**, 35, 1806.

9 Ichinose, N.; Mizuno, K.; Tamai, T.; Otsuji, Y. *Chem.lett.* **1988**, 233.

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