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Novel an Environmentally Benign Strategy for Synthesis of 2, 5 Diaryl Oxazole Derivatives

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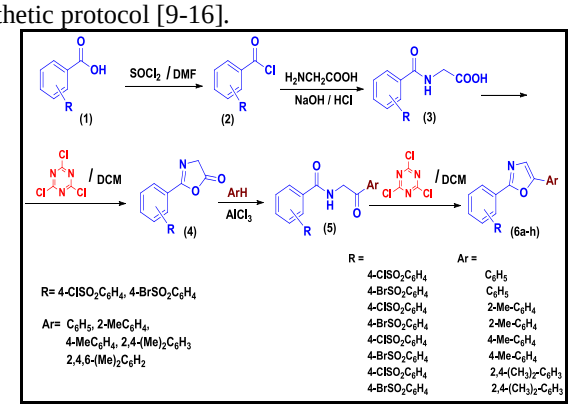
ABSTRACT

Hippuric acid **1** cyclized by using cyanuric acid (TCT) as environmentally benign catalyst to 2-phenyloxazol-5(4H)-one **2** further treated with aromatic aldehyde-Friedel Craft reaction to benzoylaminoketones **3** and finally cyclized using the same reagent (TCT) gives good yields of substituted 2,5 diaryloxazoles **6a-h** under the reflux condition.

Introduction

Oxazole scaffold are present in a large amount of natural products [1-2] possessing a wide range of pharmacological activities such as the treatment of inflammation (anti-inflammation)[3]. Phenyl substituted oxazoles as having anti-bacterial activity [4]. 2-alkyl and 2-cycloalkyl-4,5 phenyloxazoles as intermediates used as analgesic and antipyretic activity [5]. 4-substituted chloro- or 4-bromo-benzenesulfonyl)-phenyl] such 2,5-diaryloxazoles 6a-h (Fig. **1**) which are potential fluorescent sensors, laser dyes, and scintillators for detecting nuclear radiations[6]. The previously reported method for the synthesis of 2, 5 di-aryl oxazole derivatives using phosphorous oxy chloride and other halogenated reagents [7-8]. To overcome these hazardous reagent or solvent for the synthesis of 2, 5 di-aryl oxazole derivatives using cyanuric chloride as environmentally benign catalyst. In continuation with our interest as a part of green synthetic protocol [9-16].



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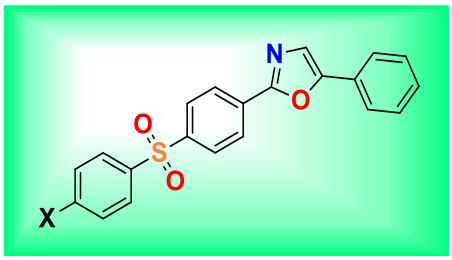
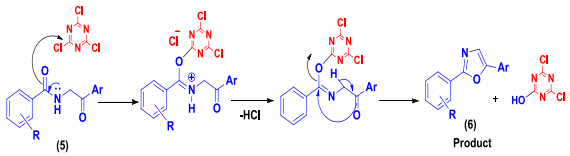


Fig 1. Target Molecule



Scheme 2. Plausible Mechanism for step 5-6: Synthesis of 2-(4-((4-chlorophenyl)sulfonyl)phenyl)-5-phenyloxazole

Results and discussion

At first, we choose environmentally benign reagent-trichloro triazine (TCT) and screened with various solvents with respect to different reaction condition for the cyclization of hippuric acid 3 (Table 1) and 4-chloro/bromo-N-(2-oxo-2-phenylethyl) benzamide 5 (Table 2). For the first model reaction of Hippuric acid (0.011mol) 3a, cyanuric chloride (0.035 mol) and solvent 8ml, reflux under the conventional method an excellent yield was obtained in a short reaction time using dichloromethane (DCM) as a solvent (Table 1 entry 2). While, in dimethyl form amide (DMF), chloroform, dimethyl sulfoxide, toluene gave corresponding yield and ethanol gave very poor yield (Table 1 entry 1, 3, 4, 5, 6).

Table 1. Screening of solvents with cyanuric chloride for the synthesis of 2-(4-((4-chlorophenyl)sulfonyl)phenyl)oxazol-5(4H)-one 4a

Entry	Catalyst	Solvent	[a]Reaction condition			
			Time(min)		Yield[b](%)	
			a	b	a	b
1.	Cyanuric chloride	DMF	60	70	30	50
2.	Cyanuric chloride	DCM	50	60	73	92
3.	Cyanuric chloride	Chloroform	60	70	65	89
4.	Cyanuric chloride	DMSO	60	70	35	65
5.	Cyanuric chloride	Toluene	60	70	32	58
6.	Cyanuric chloride	Ethanol	60	120	28	40
7.	Cyanuric chloride	Water	60	120	00	00

[a]Conventional Method: Hippuric acid(0.011mol) 3a, Cyanuric chloride (0.035 mol) and Solvent 8ml, 100ºC; [b]Isolated yield.

If we increase or decrease the time of reaction, there is no significant effect on yield of product. Herein we observed that product 4 too unstable and difficult for isolation, presumably partly reverting to hippuric acid and undergoing self-condensation if overheating it decomposes [18].

Table 2. Screening of solvents with cyanuric chloride for the synthesis 2-(4-((4-chlorophenyl)sulfonyl)phenyl)-5-phenyloxazole 6a

Entry	Catalyst	Solvent	[a]Reaction condition			
			Time(h)		Yield[b] (%)	
			a	b	a	b
1.	Cyanuric chloride	DMF	5	6	30	50
2.	Cyanuric chloride	DCM	4	5	**97**	93
3.	Cyanuric chloride	Chloroform	4	5	62	73
4.	Cyanuric chloride	DMSO	4	5	60	69
5.	Cyanuric chloride	Toluene	5	6	56	63
6.	Cyanuric chloride	Ethanol	5	6	28	36
7.	Cyanuric chloride	Water	6	7	00	00

[a]Conventional Method: 4-((4-chlorophenyl)sulfonyl)-N-(2-oxo-2-phenylethyl)benzamide (0.022mmol) **5a**, Cyanuric chloride (0.044mmol) and Solvent 15ml, 100ºC; [b]Isolated yield.

For the second model reaction of 4-((4-chlorophenyl)sulfonyl)-N-(2-oxo-2-phenylethyl)benzamide (0.022mmol) 5a, cyanuric chloride (0.044mmol) and solvent 15ml, reflux under the conventional method an good yield was obtained in a short reaction time using dichloromethane (DCM) as a solvent (Table 2 entry 2). While, in dimethyl form amide (DMF), chloroform, dimethyl sulfoxide, toluene gave corresponding yield and ethanol gave very poor yield (Table 1 entry 1, 3, 4, 5, 6). If we increase or decrease the time of reaction, there is no significant effect on yield of product. The products 4 and 6 were obtained in good yield in less time of reaction using an eco-friendly reagent compared to reported method [8]. Thus, we decide to carry out reactions in DCM as a solvent in cyanuric chloride as catalyst under conventional method. All the examples were tested reasonably good to excellent yield (Table 3). Here, we replaced hazardous, toxic organic solvents and reagent or catalyst, one of the most important goals in Green synthesis, which are environmental friendly falling in the domain of Green Chemistry.

Experimental section

General Procedure

Starting materials were commercially available. The major chemicals were purchased from Sigma Aldrich and Avra labs. Reaction courses were monitored by TLC on silica gel precoated F254 Merck plates. Melting points were recorded on SRS Optimelt, melting point apparatus and these are uncorrected. UV spectra with a Perkin-Elmer Lambda spectrophotometer. IR spectra were recorded with an FT-IR instrument, (υ_{max} in cm^{-1}), ^1H spectra were recorded on a Bruker spectrometer 300 MHz and 75 MHz for ^{13}C-NMR spectra. Chemical shifts are reported as δ_{ppm} units.

Table 3. ***2-(4-((4-chloro/bromophenyl)sulfonyl)phenyl)-5-phenyloxazole* 6**

Sr. no.	Compound	R	Ar	Yield (%)	M.P.(ºC) Found	M.P.(ºC) Reported[8]
1	**6a**	4-$ClSO_2C_6H_4$	C_6H_5	97	201-102	200
2	**6b**	4-$BrSO_2C_6H_4$	C_6H_5	94	184-185	185
3	**6c**	4-$ClSO_2C_6H_4$	4-Me-C_6H_4	92	234-235	236
4	**6d**	4-$BrSO_2C_6H_4$	4-Me-C_6H_4	93	235-236	235
5	**6e**	4-$ClSO_2C_6H_4$	2,4-$(Me)_2$-C_6H_3	91	152-153	153
6	**6f**	4-$BrSO_2C_6H_4$	2,4-$(Me)_2$-C_6H_3	89	201-201	202
7	**6g**	4-$ClSO_2C_6H_4$	2,4,6-$(Me)_2$-C_6H_3	92	163-164	164
8	**6h**	4-$BrSO_2C_6H_4$	2,4,6-$(Me)_2$-C_6H_3	90	178-179	179

General procedure for the synthesis of Hippuric acid (3a)

The acid chlorides 2a-h were prepared from the reported method [17] from the starting of benzoic acids 1a-h. Glycine (20mmol) in 1N sodium hydroxide was cooled at 0-5º C and the cold solution was added drop wise to the solution of acid chloride 2a in 25 ml of dichloromethane (DCM). The reaction mixture was stirred continued for one hour. The aqueous layer was separated and acidified with 2N hydrochloric acid, collect the product by filtration and recrystalized from ethanol. The chloro compound 3a was obtained in Yield=89% for $C_{15}H_{12}ClNO_2S$. FTIR (cm^{-1}) 1728(O-C=O), 1645(-NH-C=O, amide), 1540(-NH), 3340(-NH), 1150, 1325(SO_2), 2398(-COOH); ^{1}HNMR (δppm, DMSO-d6); 8.21(t, 1H, *J*= 5.6); 7.95 (d, 2H, *J*= 8.5); 7.90 (d, 2H, *J*= 8.5), 7.86 (d, 2H, *J*= 8.6), 7.36 (d, 2H), 4.36 (d, 2H, *J*= 5.6); ^{13}C-NMR (δppm, DMSO-d6); 136.69, 130.19, 127.98, 144.10, 141.56, 128.25, 129.16, 136.89, 168.89, 173.98, 41.98.The same procedure was used for compound 3b.

General procedure for the synthesis of Oxazolone or Cyclization of Hippuric acid (4a)

A mixture of hippuric acid (0.011mol) 3a, cyanuric chloride (0.035mol) and dichloromethane (8ml) was added and the contents of solution was heated under the reflux condition for the appropriate time (Table 1). Progress the reaction was monitored by thin layer chromatography (ethyl acetate: n-hexane). The solid obtained, crystallized from ethyl alcohol, colorless crystal of compound 4a. Yield= 92% and mp: 181-182ºC; for $C_{15}H_{10}ClNO_4S$. FTIR (cm^{-1}) 1816(C=O), 1665(C=N), 1152, 1322 (SO_2); ^{1}HNMR (δppm, DMSO-d6); 8.16(d, 2H, *J*= 8.2); 8.12 (d, 2H, *J*= 8.3); 8.06 (d, 2H, *J*= 8.6), 7.72 (d, 2H, *J*= 8.6), 4.62 (s, 2H); ^{13}C-NMR (δppm, DMSO-d6); 170.79, 165.18, 143.78, 139.41, 139.8, 136.63, 129.96, 129.42, 128.59, 128.15, 55.19. The same procedure was used for compound 4b.

General procedure for the synthesis of 4-((4-chlorophenyl)sulfonyl)-N-(2-oxo-2-phenylethyl)benzamide(5a)

The azlactone (5mmol) 4a in 25 ml of toluene or xylene in excess amount was added portion wise with (15mmol) of anhydrous aluminum chloride at room temperature. After the addition, the reaction mixture was continued under stirring for 18 hrs. The reaction mixture was then poured over crushed ice with hydrochloric acid and the organic component was extracted with ethyl acetate, washed with water and dried. The solvent was removed under the vacuum pressure and finally the product was crystallized from ethyl alcohol colorless needle was obtained. Yield=83% and mp: 202-204 ºC for $C_{21}H_{16}ClNO_4S$. FTIR (cm^{-1}) 3389(NH), 1648, 1692(C=O), 1152, 1324 (SO_2); ^{1}HNMR (δppm, DMSO-d6); 7.73(d, 2H, *J*= 8.6); 8.00-8.12 (m, 2H); 8.12 (d, 2H, *J*= 9.0), 8.09 (d, 2H, *J*= 9.0), 9.16 (t, *J*= 5.5), 4.81 (d, *J*= 5.5),; ^{13}C-NMR (δppm, DMSO-d6); 170.79, 165.18, 143.78, 139.41, 139.8, 136.63, 129.96, 129.42, 128.59, 128.15, 55.19. The same procedure was used for compound 5b.

General procedure for the synthesis of Substituted 2, 5-diphenyloxazoles (6a)

A mixture of 4-substituted *N*-(2-oxo-2-phenylethyl) benzamide (0.022mmol) 5, cyanuric chloride (0.044mmol) and dichloromethane (15ml) was added and the contents of solution was heated under the reflux condition for the appropriate time (Table 2). Progress the reaction was monitored by thin layer chromatography (ethyl acetate: n-hexane), washed with water followed by sodium bicarbonate. The solid obtained, crystallized from ethyl alcohol, Yield= 89-97%; Compound (*6a):* colorless crystal; Yield=97%; mp: 201-102ºC for $C_{21}H_{14}ClNO_3S$; ^{1}HNMR (δppm, DMSO-d6); 6.98(s,1H, N-CH=CH); 7.40-8.05 (m, 5H,Ar-H); 7.92 (d, 2H, Ar-H), 7.83 (d, 2H,Ar-H), 7.89 (d, 2H, Ar-H), 7.49(d,2H,Ar-H),; ^{13}C-NMR (δppm, DMSO-d6); 122.3, 125.3, 125.1, 128.7, 1228.5, 128.3, 128.1, 128, 129.9, 129.7, 129.5, 129.4, 129.2, 129, 135.3, 139.5, 139.2. 141.2, 148.9, 162.2

Spectral Characterization Data

(3a) 2-(4-((4-chlorophenyl)sulfonyl)benzamido)acetic acid (3a)

Yield=89% for $C_{15}H_{12}ClNO_5S$. FTIR (cm^{-1}) 1728(O-C=O), 1645(-NH-C=O, amide), 1540(-NH), 3340(-NH), 1150, 1325(SO_2), 2398(-COOH); ^{1}HNMR (δppm, DMSO-d6); 8.21(t, 1H, *J*= 5.6); 7.95 (d, 2H, *J*= 8.5); 7.90 (d, 2H, *J*= 8.5), 7.86 (d, 2H, *J*= 8.6), 7.36 (d, 2H), 4.36 (d, 2H, *J*= 5.6); ^{13}C-NMR (δppm, DMSO-d6); 136.69, 130.19, 127.98, 144.10, 141.56, 128.25, 129.16, 136.89, 168.89, 173.98, 41.98.

(4a)2-(4-((4-chlorophenyl)sulfonyl)phenyl)oxazol-5(4H)-one

Yield= 92%; mp: 181-182ºC; for $C_{15}H_{10}ClNO_4S$. FTIR (cm^{-1}) 1816(C=O), 1665(C=N), 1152, 1322 (SO_2); ^{1}HNMR (δppm, DMSO-d6); 8.16(d, 2H, *J*= 8.2); 8.12 (d, 2H, *J*= 8.3); 8.06 (d, 2H, *J*= 8.6), 7.72 (d, 2H, *J*= 8.6), 4.62 (s, 2H); ^{13}C-NMR (δppm, DMSO-d6); 170.79, 165.18, 143.78, 139.41, 139.8, 136.63, 129.96, 129.42, 128.59, 128.15, 55.19.

(5a)4-((4-chlorophenyl)sulfonyl)-N-(2-oxo-2-phenylethyl)benzamide

Yield=83% and mp: 202-204ºC for $C_{21}H_{16}ClNO_4S$. FTIR (cm^{-1}) 3389(NH), 1648, 1692(C=O), 1152, 1324 (SO_2); ^{1}HNMR (δppm, DMSO-d6); 7.73(d, 2H, *J*= 8.6); 8.00-8.12 (m, 2H); 8.12 (d, 2H, *J*= 9.0), 8.09 (d, 2H, *J*= 9.0), 9.16 (t, *J*= 5.5), 4.81 (d, *J*= 5.5),; ^{13}C-NMR (δppm, DMSO-d6); 170.79, 165.18, 143.78, 139.41, 139.8, 136.63, 129.96, 129.42, 128.59, 128.15, 55.19.

(6a)2-(4-((4-chlorophenyl)sulfonyl)phenyl)-5-phenyloxazole

Yield=97%; mp: 201-102ºC for $C_{21}H_{14}ClNO_3S$; ^{1}HNMR (δppm, DMSO-d6); 6.98(s,1H, N-CH=CH); 7.40-8.05 (m, 5H,Ar-H); 7.92 (d, 2H, Ar-H), 7.83 (d, 2H,Ar-H), 7.89 (d, 2H, Ar-H), 7.49(d,2H,Ar-H),; ^{13}C-NMR (δppm, DMSO-d6); 161.9, 148.9, 141.2, 139.2, 139.5, 135.3, 129.9, 129.7, 129.5, 129.4, 129.2, 128.5, 128.3, 128.1, 128, 128.7, 125.3, 125.1, 122.3.

(6b)2-(4-((4-bromophenyl)sulfonyl)phenyl)-5-phenyloxazole

Yield=94%; mp: 184-185ºC for $C_{21}H_{14}BrNO_3S$; ^{1}HNMR (δppm, DMSO-d6); 6.98(s,1H, N-CH=CH); 7.40-8.05 (m, 5H,Ar-H); 7.91 (d, 2H, Ar-H), 7.86 (d, 2H,Ar-H), 7.73 (d, 2H, Ar-H), 7.68(d,2H,Ar-H),; ^{13}C-NMR (δppm, DMSO-d6); 7.92 (d, 2H, Ar-H), 7.83 (d, 2H,Ar-H), 7.89 (d, 2H, Ar-H), 7.49(d,2H,Ar-H),; ^{13}C-NMR (δppm, DMSO-d6); 161.9, 149.6, 141.2, 140.2, 135.5, 135.3, 135, 130.3, 130, 129.7, 129.2, 128.9, 128.7, 128.5, 128.3, 128.1, 128., 128, 125.3, 125.1, 122.2.

(6c)2-(4-((4-chlorophenyl)sulfonyl)phenyl)-5-(p-tolyl)oxazole

Yield=92%; mp: 234-235ºC for $C_{22}H_{16}ClNO_3S$; ^{1}HNMR (δppm, DMSO-d6); 6.98(s,1H, N-CH=CH); 7.63 (d, 2H,Ar-H); 7.22 (d, 2H, Ar-H), 2.30 (s, 1H, CH_3-Ar), 7.92 (d, 2H, Ar-H), 7.86(d,2H,Ar-H), 7.89(d,2H,Ar-H), 7.48(d,2H,Ar-H); ^{13}C-NMR (δppm, DMSO-d6); 161.9, 149.6, 141.2, 139.3, 139, 135.2, 131.3, 129.7, 129.5, 128.3, 129.2, 129.1, 129, 128.7, 128.5, 128.3, 128, 124.5, 124.2, 122.2, 21.2

(6d)2-(4-((4-bromophenyl)sulfonyl)phenyl)-5-(p-tolyl)oxazole

Yield=93%; mp: 235-236ºC for $C_{22}H_{16}BrNO_3S$; ^{1}HNMR (δppm, DMSO-d6); 6.98(s,1H, N-CH=CH); 7.63 (d, 2H, Ar-H); 7.23 (d, 2H, Ar-H), 2.32 (s, 3H, CH_3-Ar), 7.82 (d, 2H, Ar-H), 7.92(d,2H, Ar-H), 7.72(d, 2H, Ar-H), 7.68(d, 2H, Ar-H); ^{13}C-NMR (δppm, DMSO-d6); 161.9, 149.5, 141.2, 139.2, 139, 135.2, 132.2, 131.3, 129.7, 129.5, 129.2, 129.2, 129, 128.4, 128.3, 128.3, 128, 124.2, 124.2, 122.1, 21.2

(6e)2-(4-((4-chlorophenyl)sulfonyl)phenyl)-5-(2,4-dimethylphenyl)oxazole

Yield=91%; mp: 152-153ºC for $C_{23}H_{18}ClNO_3S$; ^{1}HNMR (δppm, DMSO-d6); 6.98(s, 1H, N-CH=CH); 7.52 (s, 1H, Ar-H); 7.03 (s, 1H, Ar-H), 7.12 (s, 1H, Ar-H), 2.32 (s, 3H, CH_3-Ar), 2.49 (s, 3H, CH_3-Ar), 7.86 (d, 2H, Ar-H), 7.92 (d, 2H, Ar-H), 7.48 (d, 2H, Ar-H), 7.88 (d, 2H, Ar-H); ^{13}C-NMR (δppm, DMSO-d6); 161.9, 149.2, 141.2, 139.2, 139, 138.2, 136.3, 135.3, 131.7, 131.3, 129.8, 129.6, 129.4, 129, 128.7, 128.5, 128.3, 128.1, 128.4, 126.2, 122.3, 21.2, 19.1

(6f)2-(4-((4-bromophenyl)sulfonyl)phenyl)-5-(2,4-dimethylphenyl)oxazole

Yield=89%; mp: 201-201ºC for $C_{23}H_{18}BrNO_3S$; ^{1}HNMR (δppm, DMSO-d6); 6.98(s,1H, N-CH=CH); 7.52 (s, 1H, Ar-H); 7.02 (s, 1H, Ar-H), 7.09 (s, 1H, Ar-H), 2.31 (s, 3H, CH_3-Ar), 2.48(s, 3H, CH_3-Ar), 7.82 (d, 2H, Ar-H), 7.89(d, 2H, Ar-H), 7.68 (d, 2H, Ar-H), 7.70 (d, 2H, Ar-H); ^{13}C-NMR (δppm, DMSO-d6); 161.9, 149.2, 141.3, 138.3, 135.6, 135.1, 132.2, 131.2, 130.3, 130, 128.8, 128.4, 128.2, 128.1, 127.9, 127.5, 126.3, 122.2, 21.3, 19.2

6g)2-(4-((4-chlorophenyl)sulfonyl)phenyl)-5-mesityloxazole

Yield=92%; mp: 163-178ºC for $C_{24}H_{20}ClNO_3S$; ^{1}HNMR (δppm, DMSO-d6); 6.98(s,1H, N-CH=CH); 2.52 (s, 9H, CH_3-Ar); 6.91 (s, 2H, Ar-H), 7.82 (d, 2H, Ar-H), 7.91(d, 2H, Ar-H), 7.48(d, 2H, Ar-H), 7.86 (d, 2H, Ar-H). ^{13}C-NMR (δppm,

DMSO-d6); 161.9, 149.2, 141.2, 139.4, 139.1, 138.3, 136.2, 135.5, 135.2, 129.7, 129.5, 129.2, 128.9, 128.8, 128.6, 128.5, 128.3, 128.2, 128, 127.8, 127.2, 21.2, 19.3, 19.1

(6h)2-(4-((4-bromophenyl)sulfonyl)phenyl)-5-mesityloxazole

Yield=90%; mp: 178-179ºC for $C_{24}H_{20}BrNO_3S$; ^{1}HNMR (δppm, DMSO-d6); 6.98(s,1H, N-CH=CH); 2.52 (s, 9H, CH_3-Ar); 6.90 (s, 2H, Ar-H), 7.83 (d, 2H, Ar-H), 7.91(d, 2H, Ar-H), 7.68(d, 2H, Ar-H), 7.71 (d, 2H, Ar-H). ^{13}C-NMR (δppm, DMSO-d6); 161.9, 149.2, 141.2, 140.1, 138.2, 136.2, 135.3, 132.3, 132.1, 130.2, 130, 128.9, 128.8, 128.7, 128.6, 128.4, 128.2, 128, 127.5, 122.2, 21.2, 19.2, 1

Conclusions

In the present work we have developed a new methodology for the synthesis of reported 2-(4-((4-chlorophenyl)sulfonyl)phenyl)-5-phenyloxazole from starting hippuric acid and substituted aromatic compound using trichloro-triazene (TCT) as environmentally benign catalyst under the conventional method in less time of reaction with good to excellent yield of product.

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