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Lewis Acid Catalyzed C-N Bond Formation: Synthesis of Quinoxalines *via* CuX (X = Cl, Br, I) Catalyzed Cyclotrimerization of Alkynes with *o*-Phenylendiamines

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An efficient one-pot cycloisomerization reaction has been developed for the synthesis of quinoxaline derivatives from alkynes and *o*-phenylendiamines using CuX (X = Cl, Br, I) as a catalyst.This method provides a flexible and rapid route to synthesize quinoxaline derivatives.

Keywords: Alkynes, *o*-Phenylendiamines, Cycloisomerization, CuX, Quinoxaline derivatives.

INTRODUCTION

Quinoxaline is a heteroaromatic unit of extensive interests owing to its occurrence in a diverse range of biological activities including antitumor[1], antibacterial, anthelmintic, antiinflammatory, kinase inhibitory and anticancer activities[2]. They also have a wide application in dyes[3], efficient electron luminescent material[4], organic semiconductors[5], DNA cleaving agents[6], photoinitiators in UV-cured coatings[7] and donor materials[8]. Therefore, a number of methods have been developed for the synthesis of substituted quinoxalines[9-13]. Quinoxalines can be prepared from α-hydroxy ketones and 1,2-diamines using transition metal complexes as catalysts[14]. The most common method for their preparation relies on the condensation of an aryl 1,2-diamine with a 1,2-dicarbonyl compound[15]. However, the current studies are mainly focused on the same substrate such as dicarbonyl compounds or α-hydroxy ketones as the starting materials by different methodologies for the synthesis of quinoxaline derivatives[16-19]. Therefore, it is highly significant to construct quinoxaline rings by developing new synthesis methodologies from readily available materials. Recently, the work reported by Chen's group attracted our attention[20]. Chen *et al.*[20] have developed a $Cu(OAc)_2$-catalyzed method for synthesis of quinoxaline by using *o*-phenylenediamines and terminal alkynes in the presence of 3 equiv of DMAP and Cs_2CO_3. However, when we repeat their work, it is found that the product composition is complicated and a large number of the coupling product of phenylacetylene are obtained due to the strong base conditions. Herein, we reported an efficient CuX (X = Cl, Br, I)-catalyzed cycloisomerization reaction of alkynes with *o*-phenylendiamines, leading to the synthesis of quinoxaline derivatives.

EXPERIMENTAL

All compounds are commercially available and were used without further purification. NMR spectra were recorded on a Bruker AVANCE DPX-400 or Bruker AVANCE DRX-500 instrument with TMS as an internal reference. MS measurements were performed on Bruker Reflex III mass spectrometer (ESI). Elemental analyses were done with an ElementarVario Micro Cube in School of Chemistry & Chemical Engineering of Guangxi Normal University, China. Flash chromatography was performed with QingDao silica gel (300-400 mesh).

General procedure for synthesis of quinoxaline derivatives: The reaction mixture of *o*-phenylendiamine (2.5 mmol), alkynes (1 mmol), CuCl (0.1 mmol), chlorobenzene (2 mL) in a 10 mL sealed tube was stirred at 70 °C and monitored periodically by TLC. Upon completion, chlorobenzene was removed under reduced pressure by an aspirator and then the residue was purified by silica gel column chromatography (eluent, PE/EA = 50:1) to afford corresponding quinoxaline derivatives.

2-Phenyl-3-(phenylethynyl)quinoxalne (3aa): Yellow solid, m.p.: 109-111 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.15-8.05 (m, 4H), 7.76-7.72 (m, 2H), 7.60-7.53 (m, 3H), 7.51-7.47 (m, 2H), 7.39-7.25 (m, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.06, 140.97, 140.68, 138.04, 137.61, 132.08, 130.63, 130.25, 129.66, 129.56, 129.27, 128.72, 128.41, 128.12, 121.66, 95.04, 88.33. MS: *m/z* = 307 [M+H$^+$].

2-(*p*-Tolyl)-3-(*p*-tolylethynyl)quinoxaline (3ba): Yellow solid, m.p.: 112-114 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.12-8.10 (m, 2H), 8.05 (d, *J* = 8.1 Hz, 2H), 7.74-7.72 (m, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 7.5 Hz, 2H),7.17 (d, *J* = 7.5 Hz, 2H), 2.48 (s, 3H), 2.37 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.9, 140.9, 140.7, 139.93, 139.78, 138.2, 134.9, 132.0, 130.4, 129.98, 129.65, 129.2, 128.8, 128.68, 127.4, 118.8, 95.3, 88.2, 21.60, 21.44. MS: *m/z* = 335 [M+H$^+$]

2-(4-Ethylphenyl)-3-[(4-ethylphenyl)ethynyl]quinoxaline (3ca): Yellow solid, m.p.: 113-115 °C. ^{1}H NMR (500 MHz,$CDCl_3$): δ 8.15-8.09 (m, 2H), 8.06 (d, *J* = 8.0 Hz, 2H), 7.75 (dd, *J* = 6.2, 3.2 Hz, 2H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 7.9 Hz, 2H), 7.19 (d, *J* = 7.8 Hz, 2H), 2.78 (q, *J* = 7.5 Hz, 2H), 2.66 (q, *J* = 7.6 Hz, 2H), 1.32 (t, *J* = 7.6 Hz, 3H), 1.24 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.0, 146.2, 146.1, 140.9, 140.7, 138.3, 135.1, 132.1, 130.4, 129.9, 129.7, 129.2, 128.7, 128.0, 127.6, 119.0, 95.4, 88.2, 28.9, 28.8, 15.6, 15.0. MS: *m/z* = 363 [M+H$^+$]. Anal calcd for $C_{26}H_{22}N_2$: C, 86.15; H, 6.12; N, 7.73. Found: C, 86.37; H, 6.01; N, 7.62.

2-(4-Methoxyphenyl)-3-[(4-methoxyphenyl)ethynyl]-quinoxaline (3da): Yellow solid, m.p.: 124-126 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.13-8.05 (m, 4H), 7.73 (td, *J* = 1.8 Hz, 6.0 Hz, 2H), 7.49-7.47 (m, 2H), 7.09-7.06 (m, 2H), 6.89-6.86 (m, 2H), 3.91 (s, 3H), 3.82 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 160.90, 160.65, 154.2, 140.7, 138.1, 133.7, 131.2, 130.33, 130.08, 129.87, 129.06, 128.5, 114.1, 113.6, 95.5, 87.8, 55.40, 55.31. MS: *m/z* = 367 [M+H$^+$].

2-(4-Fluorophenyl)-3-[(4-fluorophenyl)ethynyl]quinoxaline (3ea): White solid, m.p.: 212-218 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.12 (m, 4H), 7.79 (m, 2H), 7.49 (dd, *J* = 8.7, 5.4 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 3H), 7.07 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$):δ 164.84, 164.37, 153.9, 141.0, 140.7, 137.70, 134.20, 134.13, 133.75, 131.77, 131.71, 130.87, 130.45, 129.55, 129.27, 128.77, 116.12, 115.94, 115.33, 115.16, 94.0, 88.0. MS: *m/z* = 363 [M+H$^+$].

2-(4-Bromophenyl)-3-[(4-bromophenyl)ethynyl]quinoxaline (3fa): White solid, m.p.: 210-217 °C. ^{1}H NMR (500 MHz, $CDCl_3$):δ 8.13 (dd, *J* = 6.3, 2.7 Hz, 2H), 8.00 (d, *J* = 8.5 Hz, 2H), 7.80 (dd, *J* = 6.4, 3.4 Hz, 2H), 7.70 (d, *J* = 8.4 Hz, 3H), 7.53 (d, *J* = 8.3 Hz, 3H), 7.36 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 141.19, 140.84, 139.3, 136.5, 133.42, 132.39, 131.99, 131.43, 131.30, 131.04, 130.87, 130.64, 129.4, 128.9, 114.0, 94.1, 89.1. MS: *m/z* = 464 [M+H$^+$]. Anal calcd for $C_{22}H_{12}Br_2N_2$: C, 56.93; H, 34.43; N, 6.04. Found: C, 57.25; H, 34.15; N, 6.12.

2-(Thiophen-2-yl)-3-(thiophen-2-ylethynyl)quinoxaline (3ha): Yellow solid,m.p.: 94-96 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.43 (d, *J* = 1.8 Hz, 1H), 8.08-8.04 (m, 2H), 8.01 (d, *J* = 4.2 Hz, 1H), 7.73-7.69 (m, 3H), 7.44 (dd, *J* = 3.0 Hz, *J* = 5.4 Hz, 1H), 7.32 (dd, *J* = 3.0 Hz, 4.8 Hz, 1H), 7.27(d, *J* = 5.1 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 149.33, 140.59, 138.93, 137.13, 131.28, 130.55, 129.96, 129.63, 129.03, 128.81, 128.58, 127.93, 125.87, 125.21,120.78, 90.32, 88.37.

2-(Pent-1-yn-1-yl)-3-propylquinoxaline (3ia): Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$):δ 8.04-7.97 (m, 2H), 7.73-7.64 (m, 2H), 3.15 (dd, *J* = 8.6, 6.9 Hz, 2H), 2.55 (t, *J* = 7.0 Hz, 2H), 1.91 (dd, *J* = 15.2, 7.6 Hz, 2H), 1.74 (dd, *J* = 14.5, 7.2 Hz, 2H), 1.12 (t, *J* = 7.4 Hz, 3H), 1.07 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.55, 140.78, 140.03, 129.85, 129.22, 128.70, 128.56, 97.22, 78.90, 77.25, 77.00, 76.75, 38.34, 29.69, 21.97, 21.78, 21.69, 14.08, 13.64. MS: *m/z* = 239 [M+H$^+$]. Anal calcd for $C_{16}H_{18}N_2$: C, 80.63; H, 7.61; N, 11.76. Found: C, 80.80; H, 7.40; N, 11.80.

2-Hexyl-3-(oct-1-yn-1-yl) quinoxaline (3ja): Colourless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.05-7.95 (m, 2H), 7.68 (m, 2H), 3.20-3.10 (t, *J* = 5.0 Hz, 2H), 2.56 (t, *J* = 7.1 Hz, 2H), 1.85 (m, 2H), 1.76-1.59 (m, 2H), 1.56-1.41 (m, 4H), 1.41-1.29 (m, 8H), 0.91 (t, *J* = 5.0 Hz, 3H), 0.90 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.9, 140.9, 140.7, 140.1, 130.0, 129.3, 128.8, 128.6, 97.6, 78.9, 36.6, 31.8, 31.5, 29.8, 29.5, 28.9, 28.8, 28.4, 22.70, 22.69, 19.9, 14.2. MS: *m/z* = 323 [M+H$^+$].

2-(Butyl)-3-(hex-1-yn-1-yl)quinoxaline (3ka): Colourless oil, ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00 (m, 2H), 7.68 (m, 2H), 3.14 (t, *J* = 7.5 Hz, 2H), 2.57 (t, *J* = 7.2 Hz, 2H), 1.90-1.80 (m, 2H), 1.74-1.64 (m, 2H), 1.60-1.43 (m, 4H), 1.02-0.95(m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ158.7, 140.63, 140.42, 139.9, 129.79, 129.14, 128.58, 128.40, 97.2, 78.6, 36,1, 30.74, 30.19, 22.73, 22.04, 19.2, 13.84, 13.50. MS: *m/z* = 267 [M+H$^+$].

6-Chloro-2-phenyl-3-(phenylethynyl)quinoxaline (3ab): White solid, m.p.: 220-225 °C. ^{1}H NMR (500 MHz, $CDCl_3$):δ 8.13-8.09 (m, 4H), 7.71 (dd, *J* = 8.9, 2.2 Hz, 1H), 7.56-7.58 (m, 3H), 7.50-7.48 (m, 2H), 7.42-7.34 (m, 3H).^{13}C NMR (125 MHz, $CDCl_3$):δ 155.4, 141.4, 139.40, 139.15, 137.5, 136.3, 132.4, 131.7, 130.67, 130.05, 129.96, 129.82, 128.66, 128.35, 127.7, 121.7, 96.1, 88.3. MS: *m/z* = 341 [M+H$^+$]. Anal calcd for $C_{22}H_{13}ClN_2$: C, 77.53; H, 3.84; N, 8.22. Found: C, 77.82; H, 3.69; N, 8.13.

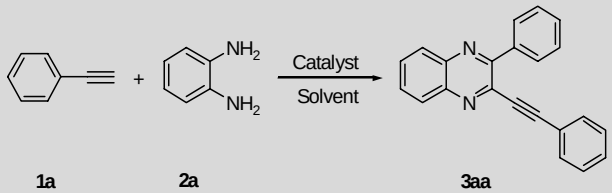
6-Chloro-3-(*p*-tolyl)-2-(*p*-tolylethynyl)quinoxaline (3bb1) and 6-chloro-2-(*p*-tolyl)-3-(*p*-tolylethynyl)quinoxaline (3bb2); 3bb1:3bb2 = 1:1: Yellow solid, m.p.: 150-153 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ8.08-8.04 (m, 4H), 7.65 (dd, *J* = 2.4 Hz, 8.7 Hz, 1H), 7.41-7.33 (m, 4H), 7.17-7.14 (d, *J* = 8.4 Hz, 2H), 2.47 (s, 3H), 2.36(s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ154.89, 141.03, 140.88, 140.20, 140.12, 140.03, 139.30, 139.13, 138.95, 136.14, 135.73, 134.39, 134.33, 132.07, 132.01, 131.25, 130.96, 139.36, 129.77, 129.63, 129.57, 129.22, 128.82, 128.07, 127.38, 118.50, 118.44, 96.12, 95.82, 87.91, 21.63, 21.46. MS: *m/z* = 369 [M+H$^+$].

6-Methyl-2-phenyl-3-(phenylethynyl)quinoxaline (3ac1) and 6-methyl-3-phenyl-2-(phenylethynyl)quinoxaline (3ac2); 3ac1:3ac2 = 2.0:1: Yellow solid, m.p.: 138-142 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.10-8.07 (m, 2H), 8.02 (d, *J* = 9.0 Hz, 1H), 7.89 (s, 1H), 7.57-7.53(m, 4H), 7.49-7.46 (m, 2H), 7.35-7.72 (m, 3H), 2.59 (S, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.5, 141.67, 141.30, 141.11, 139.4, 138.10, 138.02,133.2, 132.8, 132.30, 132.26, 130.64, 130.23, 129.88, 129.80, 129.72, 129.45, 129.0, 128.88, 128.64, 128.49,128.38, 128.32, 127.7, 122.1, 95.0, 88.7, 22.19, 22.13. MS: *m/z* = 321 [M+H$^+$].

RESULTS AND DISCUSSION

To identify the optimal conditions for the reactions, a series of catalysts and solvents were screened (Table-1). Initially, the reaction conditions were optimized starting from alkyne (**1a**) and *o*-phenylendiamine (**2a**) in PhCl at 70 °C with CuCl

TABLE-1
OPTIMIZATION OF QUINOXALINE DERIVATIVES FORMATION[a]



1a **2a** **3aa**

Entry	Catalyst	Solvent	Yield (%)[b]
1	CuCl (5 mol %)	PhCl	82
2	CuCl (10 mol %)	PhCl	90
3	CuCl (20 mol %)	PhCl	91
4	$ZnCl_2$ (10 mol %)	PhCl	NR
5	$FeCl_3$ (10 mol %)	PhCl	NR
6	$BiCl_3$ (10 mol %)	PhCl	NR
7	$CuSO_4$ (10 mol %)	PhCl	45
8	$Cu(OTf)_2$ (10 mol %)	PhCl	56
9	$CuCl_2$ (10 mol %)	PhCl	78
10	CuBr (10 mol %)	PhCl	89
11	CuI (10 mol %)	PhCl	88
12[c]	CuCl (10 mol %)	THF	20
13	CuCl (10 mol %)	Toulene	40
14	CuCl (10 mol %)	$ClCH_2CH_2Cl$	89
15[c]	CuCl (10 mol %)	CH_2Cl_2	87
16	CuCl (10 mol %)	H_2O	33
17	CuCl (10 mol %)	–	37
18	CuCl (10 mol %)	DMSO	NR
19	CuCl (10 mol %)	CH_3NO_2	NR

[a]Reaction conditions: **1a** (1 mmol), **2a** (2.5 mmol) and catalyst in PhCl (2 mL) at 70 °C. [b]Isolated yield of pure product based on alkyne **1a**. [c]The reaction was carried in sealed tube.

(5 mol %) and the desired quinoxaline derivative (**3aa**) was isolated in 82 % yield (Table-1, entry 1). The yield of **3aa** was increased to 90 % when the amount of CuCl was increased to 10 mol % (Table-1, entry 2). However, no further improvement in the yield of **3aa** could be achieved, when the amount of CuCl was increased up to 20 mol % (Table-1, entry 3).The crystallization of quinoxaline **3aa** from anhydrous ethanol gave single crystals suitable for X-ray analysis. Fig. 1 illustrates the molecular structure of quinoxaline **3aa**. $CuSO_4$,$Cu(OTf)_2$ and $CuCl_2$ were also effective, *albeit* affording the products with diminished yields (Table-1, entries 7-9). It is noted that the CuBr and CuI also smoothly promoted the reactions in excellent yields (Table-1, entries 10 and 11). Other Lewis acid catalysts, such as $ZnCl_2$, $FeCl_3$ and $BiCl_3$ did not promote the reaction (Table-1, entries 4-6). In addition, it was found that the solvent played a crucial role in this reaction (Table-1, entries 1 and 12-19). The reactions were obviously restrained when they were performed in tetrahydrofuran or toluene (Table-1, entries 12 and 13). Further inspection of the reaction conditions revealed that this reaction also proceeded efficiently in solvents such as $ClCH_2CH_2Cl$, CH_2Cl_2 (Table-1, entries 14 and 15), whereas DMSO and CH_3NO_2 were found to be unfavourable (Table-1, entries 18 and 19). Furthermore, H_2O as solvent was also able to facilitate the reaction and the reaction could be carried out under solvent-free conditions (Table-1, entries 16 and 17). On the basis of the above experiments, the optimized reaction conditions were summarized as follows: CuCl (10 mol %) in chlorobenzene as solvent.

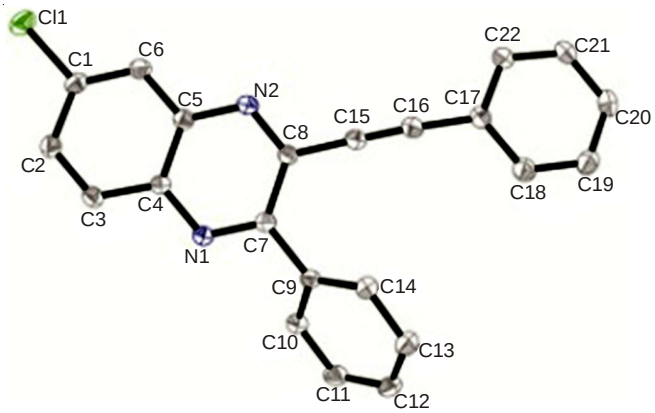
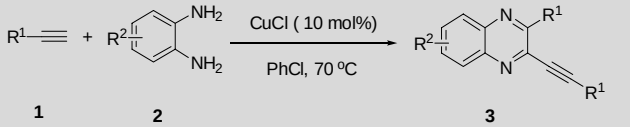


Fig. 1. X-ray Crystal structure of quinoxaline **3aa**. The thermal ellipsoids are at the 50 % probability level

With the optimized reaction conditions in hands, we started to investigate the scope and limitation of this reaction and the results are summarized in Table-2. To our delight, aromatic alkynes bearing both electron-rich and electron-poor moieties gave the corresponding quinoxaline derivatives in good to excellent yields. Substrates possessing electron-donating groups at the aromatic alkyne ring reacted smoothly and afforded the desired products in moderate yields (Table-2, entries 2-4). Other alkynes possessing electron-withdrawing groups at the benzene ring, such as fluoro and bromo also reacted smoothly, providing substituted quinoxaline derivatives in higher yields (Table-2, entries 5 and 6). This reaction was not limited to aromatic alkynes; aliphatic alkynes were also tested and it turned out that they could react smoothly with *o*-phenylendiamines **2** to give quinoxalines (Table-2, entries 9-11). Additionally, alkynes bearing a heterocyclic aromatic substituent such as 2-ethynylthiophene was found to afford

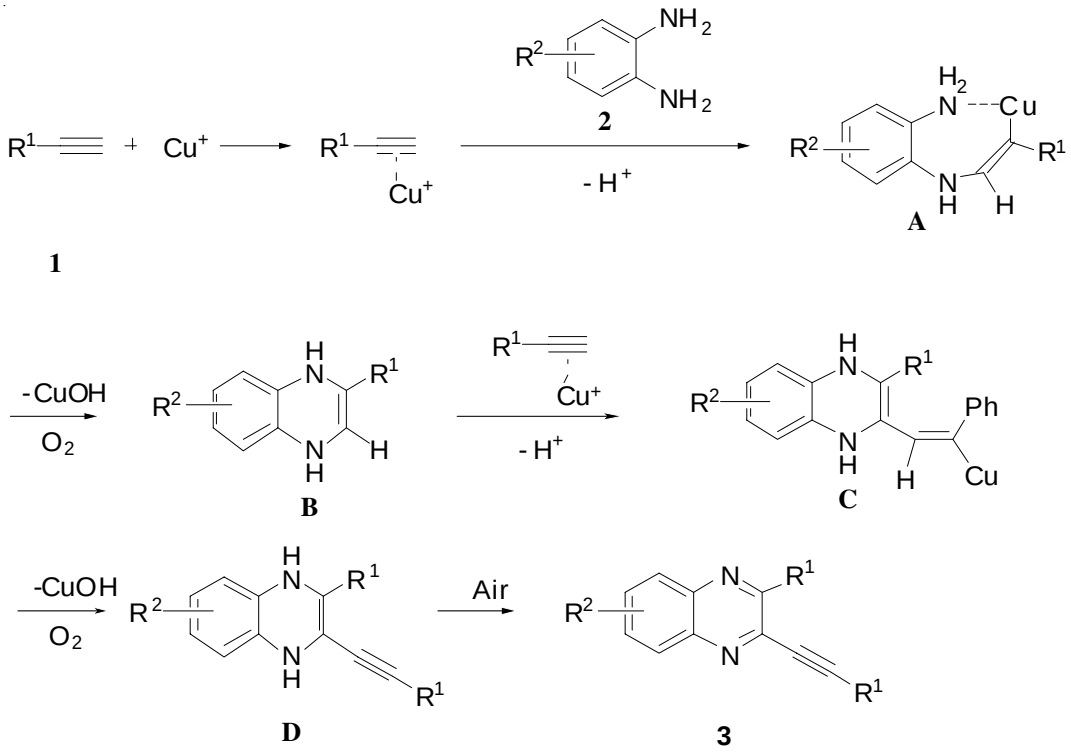
TABLE-2
SYNTHESIS OF QUINOXALINE DERIVATIVES FROM ALKYNES **1** AND *o*-PHENYLENEDIAMINES **2**[a]



1 **2** **3**

Entry	Alkyne	Diamine	Product	Time (h)	Yield (%)[b]
1	$R^1=C_6H_4$	R^2=H	**3aa**	7	90
2	R^1=4-$CH_3C_6H_4$	R^2=H	**3ba**	8	81
3	R^1=-CH_2CH_3 C_6H_4	R^2=H	**3ca**	8	78
4	R^1=4-$OCH_3C_6H_4$	R^2=H	**3da**	12	68
5	R^1=4-FC_6H_4	R^2=H	**3ea**	5	95
6	R^1=4-BrC_6H_4	R^2=H	**3fa**	5	93
7	R^1=TMS	R^2=H	**3ga**	24	Trace
8	R^1=Thienyl	R^2=H	**3ha**	12	58
9	$R^1=C_3H_7$	R^2=H	**3ia**	8	57
10	$R^1=C_6H_{13}$	R^2=H	**3ja**	7	88
11	$R^1=C_4H_9$	R^2=H	**3ka**	7	56
12	$R^1=C_6H_4$	R^2=4-Cl	**3ab**	7	71
13	R^1=4-$CH_3C_6H_4$	R^2=4-Cl	**3bb**	7	65
14	$R^1=C_6H_4$	R^2=4-NO_2	**NR**	24	NR
15	$R^1=C_6H_4$	R^2=4-CH_3	**3ac**	7	91

[a]Reaction conditions: **1** (1 mmol), **2** (2.5 mmol) and CuCl (10 mol %) in PhCl (2 mL) at 70 °C.
[b]Isolated yield of pure product based on alkyne **1**.



Scheme-I: Proposed mechanism

the desired product **3ha** in moderate yield (Table-2, entry 8). Next, the reaction scope of *o*-phenylenediamine was studied (Table-2, entries 12-15). Obviously, electron-rich *o*-phenylenediamine provided the desired products in higher yields than electron-poor *o*-phenylenediamine (Table-2, entry 15 *vs.* entries 12-14).

On the basis of previous work[21-23], our postulated reaction pathways are summarized in **Scheme-I**. The proposed initiated complex **A** would lose H^+ and Cu^+ to give **B**. Subsequently, a second equivalent of alkyne was attacked by **B** to form **C**. After the losing another H^+ and Cu^+, a precursor of quinoxaline **D** was obtained. Next, **D** could be easily aromatized to the target compound quinoxaline **3** by air.

Conclusion

In summary, we have developed an effective CuX (X = Cl, Br, I)-catalyzed cycloisomerization reaction of alkynes with *o*-phenylendiamines to synthesize the quinoxaline derivatives. A wide range of alkynes, bearing not only aryl groups but also alkyl groups, effectively participated in the reactions and the reaction conditions were relatively mild.

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