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铜催化亚砜叶立德与邻苯二胺[4＋2]环加成反应

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摘要 采用廉价易得的过渡金属铜盐作为催化剂, 实现了多种不同类型的亚砜叶立德与邻苯二胺的[4＋2]环加成反应. 实验结果表明在以 10 mol%醋酸铜[$Cu(OAc)_2$]为催化剂, 1.0 equiv.醋酸钠(NaOAc)为添加剂, 1,2-二氯乙烷(DCE)为溶剂, 80 ℃搅拌 24 h 的条件下, 目标产物 2-苯基喹喔啉的分离收率可达 93%. 该方法具有金属催化剂廉价易得、反应条件温和、底物普适性好、官能团容忍度高等显著优点.

关键词 铜催化; [4＋2]环加成; 亚砜叶立德; 喹喔啉

Copper-Catalyzed [4＋2] Annulations of Sulfoxonium Ylides and *o*-Phenylenediamines

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Abstract Herein, a novel [4＋2] annulation of a wide range of sulfoxonium ylides and *o*-phenylenediamines was reported, which was catalyzed by cheap and commercially available copper catalyst. The research results indicated that under the optimal reaction conditions, the isolated yield was up to 93% with 10 mol% $Cu(OAc)_2$ as catalyst, 1.0 equiv. of NaOAc as additive and 1,2-dichloroethane (DCE) as solvent at 80 ℃ for 24 h. This method features cheap and commerial available catalyst, mild reaction conditions, broad substrate scope and excellent functional groups tolerance.

Keywords copper catalysis; [4＋2] annulations; sulfoxonium ylides; quinoxaline

1 Introduction

Quinoxaline derivatives are recognized as a significant class of nitrogen heterocycles due to their unique biological activities including antitumor, antibacterial and anti-inflammatory.[1-3] Besides, quinoxaline skeletons were also widely applied in the field of dyes and pigments, organic semiconductors, solar cells and so on.[4-6] Therefore, tremendous efforts have been devoted in the exploitation of efficient synthesis of quinoxalines. In recent years, the majority protocols to construct quinoxaline involved the annulation of 1,2-diamines with ketone derivatives, alkenes, alkynes, and other reagents.[7-10] In addition, more and more transition-metal-catalyzed reactions and photochemical or electrochemical reactions could also achieve the facile synthesis of quinoxaline scaffolds.[11-14] Despite the above elegant works, the establishment of more effective routes with the utilization of mild reaction conditions is still in high demand.

In 1964, Corey's group[15] reported the first example of formation and photochemical rearrangement of sulfoxonium ylides. Since then, this reagent has been thoroughly exploited as versatile building blocks in organic synthesis. Generally, the sulfoxonium ylides possessed the advantages of easily preparation, crystalline and bench-stable, comparable reactivity with *α*-diazocarbonyl compounds, thermodynamic stability and security in utilization.[16-18] Thus, sulfoxonium ylides have been widely employed in various reactions, including C—H activation, olefination, cycloaddition, X—H insertion and others.[19-22] Moreover, considerable organic chemists were devoted to establishing more new and practical reactions of sulfoxonium ylides in recent years. The outstanding works predominantly focus on the construction of nitrogen-contain-

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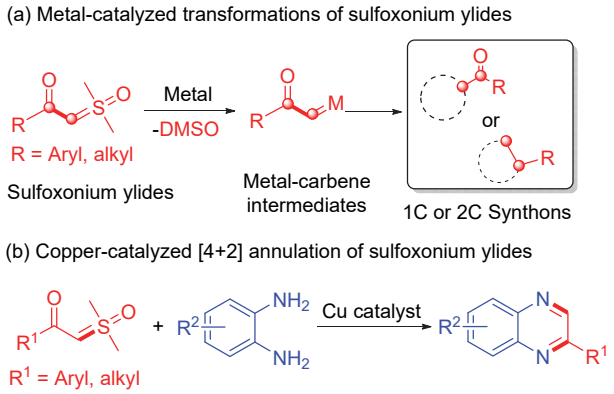
ing heterocycles such as pyrroles, indoles, quinolines, quinoxalines, oxazoles.[23-28] As shown in Scheme 1a, sulfoxonium ylides could serve as one- or two-carbon synthons under metal catalysis via metal-carbene intermediates. For example, Wu's group[24f] reported the Rh-catalyzed [1+5] and [1+4] cycloaddition of sulfoxonium ylides with *N*-arylamidines for the synthesis of indoles and quinazolines. Additionally, Liu and coworkers[29] accomplished the application of sulfoxonium ylides as two carbon synthons for the preparation of pyrazolo[1,2-*a*]cinnolines. However, expensive and detrimental metal catalysts, comparatively harsh reaction conditions limited the wide application of sulfoxonium ylides in organic synthesis. Herein, a copper-catalyzed [4+2] annulations of sulfoxonium ylides and *o*-phenylenediamines were disclosed with the advantages of cheap metal catalyst, mild reaction conditions, broad substrate scope and excellent functional groups tolerance (Scheme 1b).

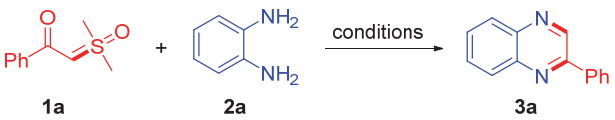


Scheme 1 Protocols for sulfoxonium ylides transformations

2 Results and discussion

Based on the previous works,[20] sulfoxonium ylides (**1a**) and the commercially readily available *o*-phenylenediamine (**2a**) were selected as standard substrates to investigate the workable reaction conditions. Firstly, $Pd(OAc)_2$ was selected as the catalyst, CsOAc as the additive and 1,2-dichloroethane (DCE) as the solvent. However, the desired products **3a** could not be obtained. Several other types of metal catalyst were then examined. It was indicated that the catalytic efficiency of low cost copper salts was comparable with expensive Ir or Rh catalyst (Table 1, Entries 1～4). Subsequently, a series of additives were probed, including NaOAc, KOAc, $NaHCO_3$, Na_2CO_3. NaOAc was identified as the most effective additive by enhancing the formation of **3a** to 85% yield (Table 1, Entries 5～8). Following these exciting results, various solvents screening investigations displayed that other solvents could not improve the generation of expected quinoxaline product (Table 1, Entries 9～13). In further examination, controlled experiments revealed that copper catalyst was indispensable in this reaction (Table 1, Entries 9～13). The further temperature evaluation illustrated that 80 ℃ was the optimal reaction temperature (Table 1, Entries 15～16). Finally, different reaction atmosphere was examined (Table 1, Entries 17～18), which indicated that the reaction could proceed efficiently under air atmosphere.

Table 1 Optimization of reaction conditions[a]



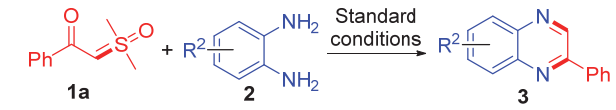
Entry	Catalyst	Additive	Solvent	Yield[b]/%
1	$Pd(OAc)_2$	CsOAc	DCE	0
2	$[Ir(cod)Cl]_2$	CsOAc	DCE	60
3	$(Cp^*RhCl_2)_2$	CsOAc	DCE	58
4	$Cu(OAc)_2$	CsOAc	DCE	54
5	$Cu(OAc)_2$	NaOAc	DCE	85
6	$Cu(OAc)_2$	KOAc	DCE	62
7	$Cu(OAc)_2$	$NaHCO_3$	DCE	43
8	$Cu(OAc)_2$	Na_2CO_3	DCE	30
9	$Cu(OAc)_2$	NaOAc	DCM	35
10	$Cu(OAc)_2$	NaOAc	MeCN	42
11	$Cu(OAc)_2$	NaOAc	THF	50
12	$Cu(OAc)_2$	NaOAc	Toluene	58
13	$Cu(OAc)_2$	NaOAc	Dioxane	38
14	—	CsOAc	DCE	0
15[c]	$Cu(OAc)_2$	NaOAc	DCE	93
16[d]	$Cu(OAc)_2$	NaOAc	DCE	80
17[e]	$Cu(OAc)_2$	NaOAc	DCE	95
18[f]	$Cu(OAc)_2$	NaOAc	DCE	24

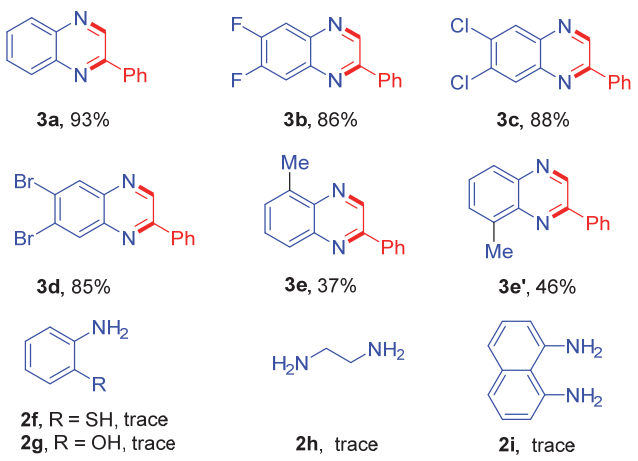
[a] Reaction conditions: unless otherwise noted, all reactions were performed with **1a** (0.20 mmol), **2a** (0.3 mmol, 1.5 equiv.), catalyst (0.10 equiv.), additives (0.20 mmol, 1.0 equiv.), in solvent (2.0 mL) at 60 ℃ for 24 h under air. [b] Isolated yield. [c] Reaction at 80 ℃. [d] Reaction at 100 ℃. [e] Reaction under O_2 at 80 ℃. [f] Reaction under N_2 at 80 ℃.

With the optimal reaction conditions obtained, we set out to investigate the substrate scope of this annulation process. Diverse types of *o*-phenylenediamines were examined as shown in Table 2. Delightfully, different symmetrical di-halogen-substituted *o*-phenylenediamines were well-tolerated and providing the desired quinoxalines successfully in high yields (**3a**～**3d**). When it came to the example of 2-methyl-*o*-phenylenediamine (**2e**), two regioisomers (**3e** and **3e'**) were generated, which could be isolated by column chromatography both in moderate yields. To our disappointment, other di-nucleophilicity substituted substrates (**2f**～**2i**) were incompatible for the optimal reaction conditions.

To further demonstrate the broad application of this reaction, diverse types of sulfoxonium ylides were then evaluated (Table 3). A wide variety of sulfoxonium ylides bearing different substituents could be converted smoothly into the corresponding products in moderate to excellent yields. Generally, substrates bearing electron-withdrawing groups, such as halogen, nitrile groups were more effective than those with electron-donating groups (**4a**～**4h**). Whereas the *ortho*-methyl substituted **4i** and **4o** were remarkably affected by steric hindrance, the yields of the desired products were decreased to 70% and 60%, respec-

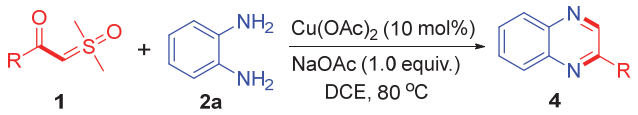
Table 2 Substrate scope of *o*-phenylenediamines[a,b]

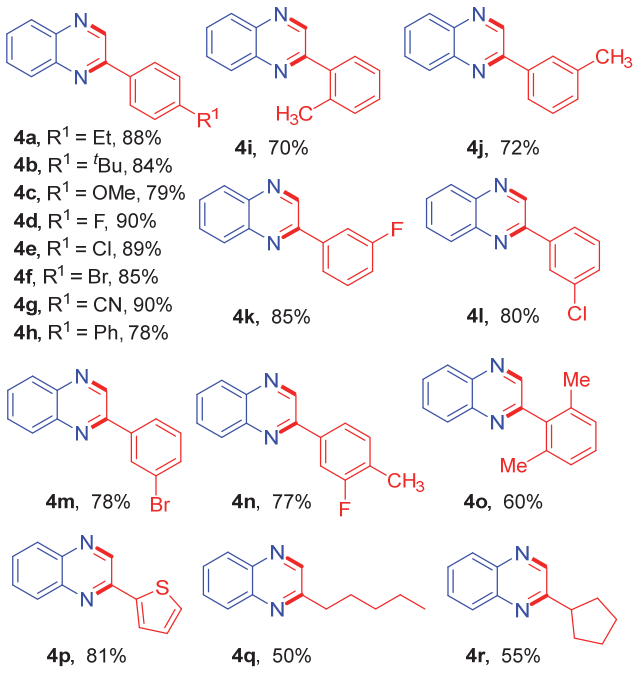




[a] General reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2** (0.3 mmol, 1.5 equiv.), $Cu(OAc)_2$ (10 mol%), NaOAc (0.2 mmol, 1.0 equiv.), DCE (2 mL), stirred at 80 ℃ for 24 h under air; [b] Yield of isolated products based on **1**.

Table 3 Scope of sulfoxonium ylides[a,b]

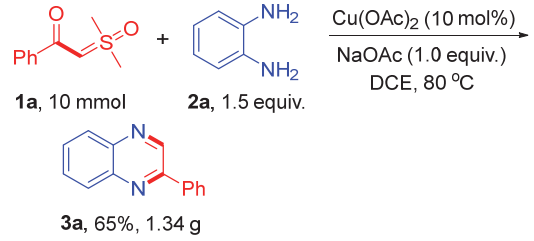




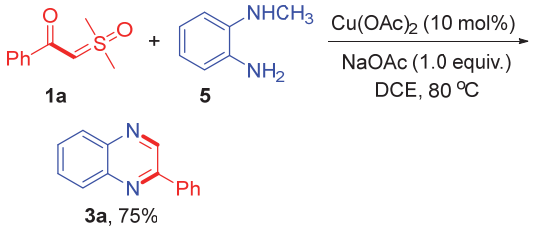
[a] General reaction conditions: **1** (0.2 mmol, 1.0 equiv.), **2a** (0.3 mmol, 1.5 equiv.), $Cu(OA)_2$ (10 mol%), NaOAc (0.2 mmol, 1.0 equiv.), DCE (2 mL), stirred at 80 ℃ for 24 h under air; [b] Yield of isolated products based on **1**.

tively. Aside from mono-substituted derivatives, multiple-substituted sulfoxonium ylides were also compatible in this reaction system to give the desired products **4n** and **4o** in moderate yields (77% and 60%). Moreover, in the case of alkyl-sulfoxonium ylides substrates, both long-chain alkanes (**4q**) and cycloalkanes (**4r**) delivered acceptable results.

Encouraged by this practically synthesis of various quinoxalines, the gram scale experiment was conducted to confirm the significance of this reaction. To our delight, 1.34 g of **3a** was isolated as expected for the subsequent synthetic elaboration (Eq. 1). It was noteworthy mentioning that the *N*-protected substrate **5** was also suitable in this reaction, yielding **3a** in 75% yield (Eq. 2).

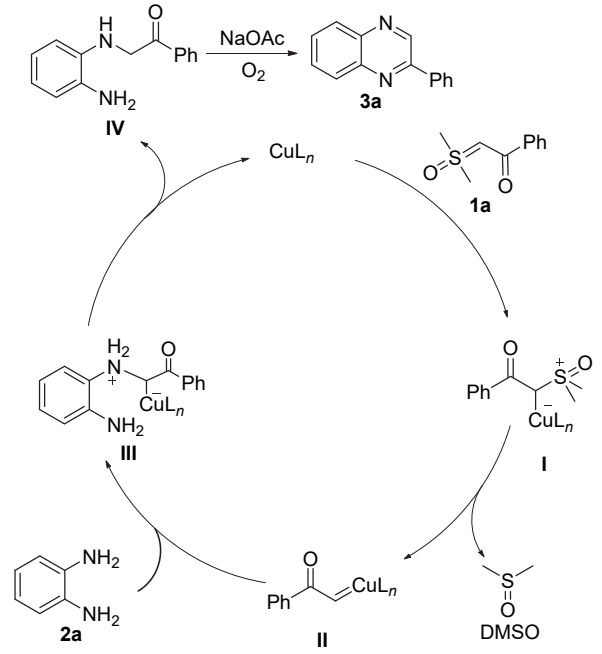


(1)



(2)

Based on the previous reported literatures,[20,22,26] the plausible mechanism was depicted in Scheme 2. At first, the two-carbon synthon *β*-keto sulfoxonium ylide **1a** was converted into highly activated copper carbene species **I** with the assistance of copper salts. After elimination of



Scheme 2 Proposed reaction mechanism

dimethyl sulfoxide (DMSO), the copper carbene intermediate **II** was generated. Next, this copper carbene species inserted into the N—H bonds of *o*-phenylenediamines to generate intermediates **III**. Then, intermediate **III** converted into the intermediate **IV** successfully with the regeneration of active copper catalyst. Finally, after three crucial process including intramolecular nucleophilic addition, dehydration and oxidative aromatization in turn, the desired product **3a** was rapidly generated.

3 Conclusions

In conclusion, a novel and feasible copper catalyzed approach for the synthesis of quinoxaline products has been established. This reaction system is compatible with extensive substrates of sulfoxonium ylides and *o*-phenylenediamines. Furthermore, the utilization of cheap commercially available copper catalysis and moderate reaction conditions as well as the practical scale-up synthesis exhibit great potential and inspiration for the construction of quinoxaline skeletons.

4 Experimental section

4.1 General experimental information

^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer and the frequencies for ^{1}H NMR and ^{13}C NMR test are 400 MHz and 100 MHz, respectively except for products **3c**, **3e**, **3e'**, **4f**. ^{1}H NMR and ^{13}C NMR spectra of products **3c**, **3e**, **3e'**, **4f** were recorded on a Bruker AMX-500 spectrometer and the frequencies for ^{1}H NMR and ^{13}C NMR test are 500 MHz and 125 MHz, respectively. The chemical shifts were reported with TMS as internal standard. Melting points were tested in an X-4A instrument without correcting temperature. Sulfoxonium ylides[20] were synthesized following literature processes. All other chemicals and solvents used in the experiments were obtained from commercial sources and used directly without further treatment.

4.2 General procedure for the synthesis of amidines

To a 25 mL of sealed tube were added **1** (0.2 mmol, 1.0 equiv.), **2** (0.3 mmol, 1.5 equiv.), $Cu(OAc)_2$ (10 mol%), NaOAc (1.0 equiv.) and DCE (2 mL). Then the reaction was stirred at 80 ℃ for 24 h. Upon completion, the reaction mixture was diluted with EtOAc and washed with water for three times. The organic phases were combined and concentrated, and the residue was purified with silica gel chromatography to deliver the pure products with the elution of mixed petroleum ether/ethyl acetate.

2-Phenylquinoxaline (**3a**):[26a] Yellow solid (38.3 mg, 93% yield), m.p. 80～81 ℃ (lit.[26b] 75～77 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.32 (s, 1H), 8.20～8.18 (m, 2H), 8.16～8.10 (m, 2H), 7.79～7.71 (m, 2H), 7.58～7.51 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.78, 143.29, 142.25, 141.50, 136.71, 130.22, 130.13, 129.57, 129.48, 129.09, 129.05, 127.50.

6,7-Difluoro-2-phenylquinoxaline (**3b**):[26a] Yellow solid (41.6 mg, 86% yield), m.p. 122～123 ℃ (lit.[35] 122～124 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.27 (s, 1H), 8.16～8.14 (m, 2H), 7.89～7.81 (m, 2H), 7.56～7.52 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.62 (dd, J=256.2, 15.7 Hz), 152.04 (dd, J=256.0, 15.9 Hz), 151.93 (d, J=3.2 Hz), 143.31 (d, J=3.1 Hz), 139.64 (d, J=10.4 Hz), 138.72 (d, J=10.1 Hz), 136.07, 130.49, 129.17, 127.40, 115.11 (dd, J=17.2, 1.5 Hz), 114.78 (dd, J=17.3, 1.8 Hz).

6,7-Dichloro-2-phenylquinoxaline (**3c**):[26a] Yellow solid (48.2 mg, 88% yield), m.p. 153～154 ℃ (lit.[26b] 151～153 ℃); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.29 (d, J=1.6 Hz, 1H), 8.23 (d, J=1.8 Hz, 1H), 8.20 (d, J=1.8 Hz, 1H), 8.17～8.15 (m, 2H), 7.57～7.54 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 152.53, 144.18, 141.01, 140.18, 135.89, 134.86, 133.94, 130.72, 130.12, 129.71, 129.22, 127.51.

6,7-Dibromo-2-phenylquinoxaline (**3d**): Yellow solid (61.5 mg, 85% yield), m.p. 173～176 ℃ (lit.[30] 173～176 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.31 (s, 1H), 8.44～8.40 (m, 2H), 8.18～8.16 (m, 2H), 7.57～7.55 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.61, 144.28, 141.52, 140.69, 135.90, 133.58, 133.14, 130.77, 129.24, 127.54, 126.96, 125.96.

5-Methyl-2-phenylquinoxaline (**3e**): Yellow solid (16.3 mg, 37% yield), m.p. 104～105 ℃ (lit.[26b] 120～122 ℃); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.33 (s, 1H), 8.27～8.25 (m, 2H), 7.96～7.94 (m, 1H), 7.64～7.61 (m, 2H), 7.58～7.55 (m, 2H), 7.53～7.50 (m, 1H), 2.83 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.18, 142.59, 141.63, 141.32, 137.98, 137.04, 130.16, 130.02, 129.28, 129.07, 127.44, 126.86, 17.08.

8-Methyl-2-phenylquinoxaline (**3e'**): Yellow solid (20.2 mg, 46% yield), m.p. 106～107 ℃ (lit.[26b] 107～109 ℃); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.33 (s, 1H), 8.20 (d, J=8.4 Hz, 2H), 8.01 (d, J=8.4 Hz, 1H), 7.67 (t, J=8.0 Hz, 1H), 7.59～7.56 (m, 3H), 7.53～7.51 (m, 1H), 2.83 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 151.37, 142.39, 141.97, 140.73, 137.30, 136.88, 130.07, 129.64, 129.13 (2C), 127.52, 127.49, 17.30.

2-(4-Ethylphenyl)quinoxaline (**4a**):[31] Yellow solid (41.2 mg, 88% yield), m.p. 77～78 ℃ (lit.[36] 74.7～75.5 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.30 (s, 1H), 8.14～8.08 (m, 4H), 7.76～7.68 (m, 2H), 7.37 (d, J=8.0 Hz, 2H), 2.73 (q, J=8.0 Hz, 2H), 1.28 (t, J=8.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.78, 146.69, 143.24, 142.25, 141.34, 134.13, 130.10, 129.46, 129.20, 129.00, 128.63, 127.46, 28.69, 15.37.

2-(4-(*tert*-Butyl)phenyl)quinoxaline (**4b**):[31] Yellow oil (44.0 mg, 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.31 (s, 1H), 8.15～8.09 (m, 4H), 7.77～7.69 (m, 2H), 7.58 (d, J=8.0 Hz, 2H), 1.38 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 153.53, 151.78, 143.24, 142.29, 141.31, 133.88, 130.12, 129.49, 129.24, 128.98, 127.24, 126.08, 34.78, 31.16.

2-(4-Methoxyphenyl)quinoxaline (**4c**):[26a] Yellow solid (37.3 mg, 79% yield), m.p. 104～105 ℃ (lit.[26b] 99～101 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.27 (s, 1H), 8.15 (d, J=8.0 Hz, 2H), 8.09 (t, J=8.0 Hz, 2H), 7.74～7.69

(m, 2H), 7.06 (d, J=7.6 Hz, 2H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.42, 151.42, 142.90, 142.26, 141.00, 130.18, 129.30, 129.12, 129.06, 128.90, 128.84, 114.53, 55.35.

2-(4-Fluorophenyl)quinoxaline (**4d**):[26a] Yellow solid (40.3 mg, 90% yield), m.p. 120～122 ℃ (lit.[26b] 120～122 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.25 (s, 1H), 8.18～8.15 (m, 2H), 8.11～8.08 (m, 2H), 7.75～7.73 (m, 2H), 7.22 (t, J=8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.13 (d, J=250.8 Hz), 150.57, 142.77, 142.06, 141.36, 132.79 (d, J=3.2 Hz), 130.28, 129.47, 129.42, 129.33, 129.02, 116.10 (d, J=21.8 Hz).

2-(4-Chlorophenyl)quinoxaline (**4e**):[26a] Yellow solid (42.7 mg, 89% yield), m.p. 132～134 ℃ (lit.[26b] 138～139 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.28 (s, 1H), 8.16～8.10 (m, 4H), 7.81～7.74 (m, 2H), 7.53 (d, J=8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.55, 142.80, 142.18, 141.57, 136.55, 135.11, 130.45, 129.76, 129.55, 129.36, 129.09, 128.73, 127.35.

2-(4-Bromophenyl)quinoxaline (**4f**):[26a] Yellow solid (48.3 mg, 85% yield), m.p. 137～138 ℃ (lit.[33] 119～122 ℃); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 9.28 (s, 1H), 8.14～8.10 (m, 2H), 8.07 (d, J=8.0 Hz, 2H), 7.80～7.73 (m, 2H), 7.68 (d, J=8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 150.56, 142.70, 142.16, 141.58, 135.53, 132.30, 130.45, 129.78, 129.55, 129.09, 128.94, 124.95.

4-(Quinoxalin-2-yl)benzonitrile (**4g**):[26a] Yellow solid (41.6 mg, 90% yield), m.p. 193～194 ℃ (lit.[37] 192～194 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.26 (s, 1H), 8.25 (d, J=8.0 Hz, 2H), 8.08 (t, J=6.4 Hz, 2H), 7.78～7.73 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.48, 142.68, 142.14, 142.01, 140.75, 132.79, 130.75, 130.48, 129.75, 129.21, 127.95, 118.40, 113.63.

2-([1,1'-Biphenyl]-4-yl)quinoxaline (**4h**): Yellow solid (44.0 mg, 78% yield), m.p. 127～128 ℃ (lit.[33] 118～120 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.38 (s, 1H), 8.29 (d, J=8.0 Hz, 2H), 8.15 (dd, J=16.4, 8.0 Hz, 2H), 7.81～7.75 (m, 4H), 7.68 (d, J=8.0 Hz, 2H), 7.49 (t, J=8.0 Hz, 2H), 7.40 (t, J=8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.41, 143.21, 142.97, 142.36, 141.52, 140.20, 135.56, 130.32, 129.60, 129.53, 129.09, 128.90, 127.93, 127.84, 127.82, 127.14.

2-(*o*-Tolyl)quinoxaline (**4i**): Yellow solid (30.8 mg, 70% yield), m.p. 75～76 ℃ (lit.[26b] 88～89 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.91 (s, 1H), 8.05 (d, J=8.0 Hz, 2H), 7.69～7.66 (m, 2H), 7.45 (d, J=8.0 Hz, 1H), 7.27～7.26 (m, 3H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.90, 145.84, 141.94, 140.98, 137.07, 136.50, 131.13, 130.13, 129.92, 129.62, 129.47, 129.32, 129.10, 126.24, 20.25.

2-(*m*-Tolyl)quinoxaline (**4j**): Yellow solid (31.7 mg, 72% yield), m.p. 82～83 ℃ (lit.[26b] 84～86 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.30 (s, 1H), 8.13 (dd, J=16.4, 8.0 Hz, 2H), 8.01 (s, 1H), 7.96 (d, J=8.0 Hz, 1H), 7.79～7.71 (m, 2H), 7.45 (t, J=7.6 Hz, 1H), 7.33 (d, J=8.0 Hz, 1H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 152.00, 143.46, 142.25, 141.49, 138.89, 136.68, 130.95, 130.19, 129.53, 129.41, 129.06, 128.99, 128.15, 124.64, 21.51.

2-(3-Fluorophenyl)quinoxaline (**4k**): Yellow solid (38.1 mg, 85% yield), m.p. 80～81 ℃ (lit.[32] 113～115 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.29 (d, J=8.0 Hz, 1H), 8.16～8.10 (m, 2H), 7.96 (d, J=8.0 Hz, 2H), 7.80～7.75 (m, 2H), 7.56～7.48 (m, 1H), 7.26～7.18 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 163.44 (d, J=246.8 Hz), 150.45, 142.98, 142.22, 141.82, 139.03 (d, J=7.6 Hz), 130.73 (d, J=8.1 Hz), 130.53, 129.97, 129.70, 129.17, 123.07, 117.18 (d, J=21.4 Hz), 114.52 (d, J=23.1 Hz).

2-(3-Chlorophenyl)quinoxaline (**4l**):[31] Yellow solid (38.4 mg, 80% yield), m.p. 132～134 ℃ (lit.[26b] 107～109 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.26 (s, 1H), 8.20 (s, 1H), 8.12 (t, J=8.0 Hz, 2H), 8.03 (s, 1H), 7.80～7.73 (m, 2H), 7.47 (d, J=4.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: ^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.17, 142.79, 142.09, 141.68, 138.39, 135.25, 130.44, 130.26, 130.10, 129.88, 129.59, 129.07, 127.57, 125.41.

2-(3-Bromophenyl)quinoxaline (**4m**): Yellow solid (44.3 mg, 78% yield), m.p. 131～133 ℃ (lit.[32] 131～132.3 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.27 (s, 1H), 8.37 (s, 1H), 8.15～8.08 (m, 3H), 7.81～7.73 (m, 2H), 7.63 (d, J=8.0 Hz, 1H), 7.41 (t, J=8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.10, 142.82, 142.11, 141.74, 138.67, 133.04, 130.52, 130.47, 129.91, 129.61, 129.10, 125.89, 123.40.

2-(3-Fluoro-4-methylphenyl)quinoxaline (**4n**):[26a] Yellow solid (36.7 mg, 77% yield), m.p. 130～131 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.25 (s, 1H), 8.11 (t, J=8.0 Hz, 2H), 8.04 (d, J=7.2 Hz, 1H), 7.95 (s, 1H), 7.78～7.70 (m, 2H), 7.16 (t, J=8.0 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.80 (d, J=249.5 Hz), 150.90, 142.91, 142.11, 141.29, 132.47 (d, J=3.6 Hz), 130.81, 130.75, 130.28, 129.40, 129.01, 126.67 (d, J=8.6 Hz), 125.82 (d, J=17.6 Hz), 115.70 (d, J=23.0 Hz), 14.66.

2-(2,6-dimethylphenyl)quinoxaline (**4o**):[34] Yellow solid (28.1 mg, 60% yield), m.p. 101～103 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.21 (s, 1H), 8.16 (d, J=8.0 Hz, 2H), 7.89 (s, 1H), 7.84 (s, 1H), 7.56～7.49 (m, 3H), 2.50 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.96, 142.30, 141.21, 140.78, 140.47, 140.10, 137.07, 129.79, 129.02, 128.61, 128.07, 127.34, 20.31.

2-(Thiophen-2-yl)quinoxaline (**4p**):[26a] Yellow solid (34.4 mg, 81% yield), m.p. 117～119 ℃ (lit.[26b] 119 ℃); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.21 (s, 1H), 8.05 (d, J=8.0 Hz, 2H), 7.83 (s, 1H), 7.74～7.65 (m, 2H), 7.53 (d, J=4.8 Hz, 1H), 7.18 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 147.28, 142.15, 142.03, 141.95, 141.23, 130.34, 129.75, 129.10, 129.03, 128.39, 126.89.

2-Pentylquinoxaline (**4q**):[33] Yellow oil (20.0 mg, 50% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.74 (s, 1H), 8.09～8.03 (m, 2H), 7.75～7.67 (m, 2H), 3.01 (t, J=8.0 Hz, 2H), 1.87～1.82 (m, 2H), 1.40～1.36 (m, 4H), 0.93～0.89 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.62, 145.75, 142.12, 141.11, 129.81, 129.07, 128.80, 128.77, 36.41, 31.53, 29.14, 22.40, 13.89.

2-Cyclopentylquinoxaline (**4r**):[26a] Yellow oil (21.8 mg, 55% yield), ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.77 (s, 1H), 8.07～8.02 (m, 2H), 7.74～7.66 (m, 2H), 3.47～3.39 (m, 1H), 2.23～2.18 (m, 2H), 1.99～1.90 (m, 4H), 1.79～1.76 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 160.71, 145.38, 142.08, 141.23, 129.70, 129.01, 128.95, 128.70, 46.14, 33.10, 25.94.

Supporting Information The ^{1}H NMR and ^{13}C NMR spectra of **3a**～**4r**. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn/.

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