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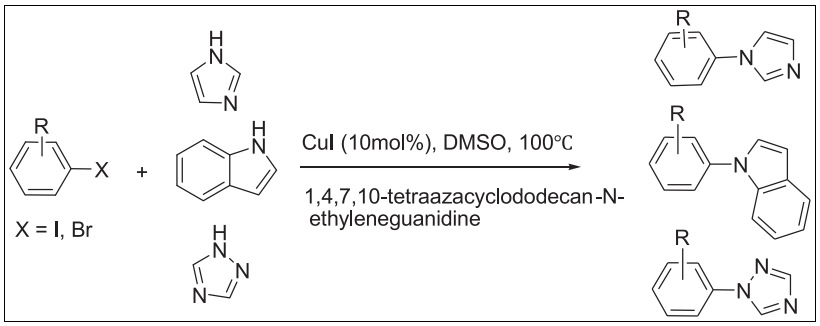

An efficient copper-catalyzed N-arylation reactions of imidazole, indole, and triazole with aryl or heteroaryl halides using cyclen derivatives as efficient organic base and ligand at moderate temperature have been investigated. The cross-couplings proceed smoothly with good to excellent yields.

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INTRODUCTION

Copper-assisted Ullmann-type coupling reactions are valuable transformations for organic synthesis. Researchers have extensively applied these reactions in both academic and industrial settings. However, for a long time, these reactions had been carried out at high temperature (normally above 150°C) and many functional groups could not be tolerated and therefore their usage was greatly limited. In addition, these reactions often require the use of stoichiometric amounts of copper reagents, which, on scale, leads to problems of waste disposal [1,2]. To overcome these drawbacks, important developments have been made on modification of the catalytic system such as improving the solubility of the copper source via addition of a wide range of ligands. Diamines [3–5], amino acids [6–8], 1,10-phenanthrolines [9], diols and triols [10–12], diketones [13], ketone-esters [14], amino-alcohols [15], diimines [16], imine-amine [17], diethylsalicylamide [5], etc. were found to be effective ligands for the copper-catalyzed Ullmann arylation reaction. In addition, in most reported methods of Ullmann reactions, inorganic base, which make the transformation suffer heterogeneous reaction in organic solvents, were commonly used. Only a few organic bases, such as 1,8-diazabicyclo[5.4.0]undec-7-ene [18], tetraalkylammonium salts [19,20], alkoxide [21], and tetraethylenepentamine [22] have been reported. Therefore, it is necessary to explore more efficient organic base to simplify the process. Herein, we reported our preliminary investigations on the use of cyclen derivatives as base and ligand for the Cu-catalyzed N-arylation of heterocycles.

RESULT AND DISCUSSION

Usually, the ligand and base play an important role for a successful Cu-catalyzed C–N formation reaction. At the outset of this study, we found that treatment of iodobenzene (1 equiv) with imidazole (1 equiv), 10 mol% CuI as catalyst, cyclen (**1a**, 2 equiv) as a base and ligand in DMSO at 100°C for 12 h gave the good result, furnishing N-phenylimidazole in 82% yield (entry 1). Encouraged by this result, different cyclen derivatives (**1a–f**) were tested under same conditions. The results are shown in Table 1.

According to the results in Table 1, it was observed clearly that the base plays a very important role in the reaction. We observed that there was no coupling product when only using CuI. In presence of **1a–f**, all reactions were carried out smoothly and gave the desired N-phenylimidazole in 51–92% yields. **1f** with strong alkaline side chain was the best, **1b** with CO_2H group only gave the moderate yield. Therefore, we could speculate that the main contribution of **1** in this reaction was as base, and followed by as ligand, which can promote the dissolution of CuI in DMSO. In addition, the effects of the amount of **1f** were investigated. The 2 equiv **1f** was appropriate (entry 7). The copper source such as $CuCl_2$, CuBr, $Cu(OAc)_2$, and CuO has been studied in this reaction, all copper sources can afford satisfactory results, CuI turns out to give the best result (entries 7, 10–13).

Therefore, the optimal results have been obtained when imidazole (1.0 equiv) and aryl halide (1.0 equiv) were allowed to react with **1f** (2.0 equiv), CuI (0.1 equiv) in DMSO 2 mL at 100°C.

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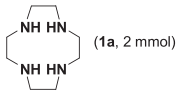
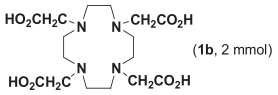
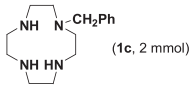
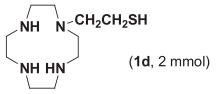
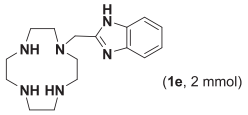
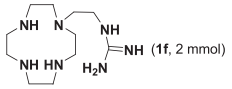
Table 1

Screening reaction conditions for N-arylation of imidazoles with iodobenzene.[a]

Entry	[Cu]	Compound **1** (mol%)	Time (h)	Yield (%) [b]
1	CuI	 (**1a**, 2 mmol)	12	82
2	CuI	 (**1b**, 2 mmol)	12	51
3	CuI	 (**1c**, 2 mmol)	12	83
4	CuI	 (**1d**, 2 mmol)	12	88
5	CuI	 (**1e**, 2 mmol)	12	87
6	CuI	 (**1f**, 2 mmol)	12	92
7	CuI	**1f** (1 mmol)	12	85
8	CuI	**1f** (3 mmol)	12	92
9	$CuCl_2$	**1f** (2 mmol)	18	85
10	CuBr	**1f** (2 mmol)	16	88
11	$Cu(OAc)_2$	**1f** (2 mmol)	16	86
12	CuO	**1f** (2 mmol)	20	75
13	CuI	—	12	Trace

[a]Reaction conditions: iodobenzene (1 mmol), imidazole (1 mmol), [Cu] (0.1 mmol), and **1a–f** (2 mmol) in 2 mL in DMSO at 100°C.
[b]Isolated yield.

With the optimized conditions in hand, we examined the scope of the substrates for this catalyst system. As shown in Table 2, both aryl iodides and bromides gave the coupling products under the optimized conditions. But the aryl iodides react more quickly than aryl bromides with the same nucleophile and afford higher yield. The electronic nature of the aryl halides was also found to have no obvious effect on the reaction yield (entries 2, 4-7). The obviously reduced yields of imidazole and 2-iodotoluene, 2-nitroiodobenzene suggest that the adverse impact of steric hindrance exists in the current system (entries 3 and 9). The catalyst system developed was also successfully amenable to the N-arylation of indole and triazole (entries 16-21).

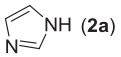
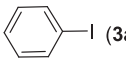
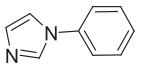
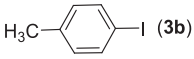
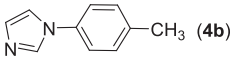
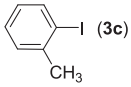
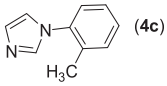
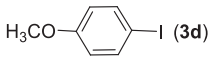
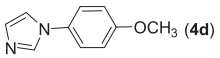
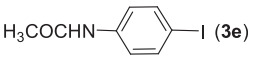
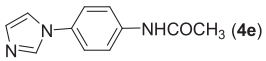
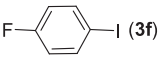
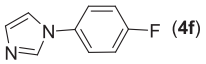
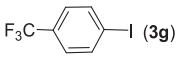
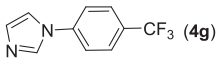
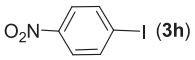
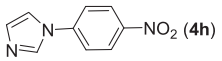
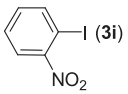
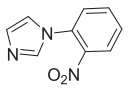
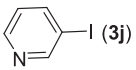
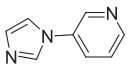
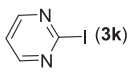
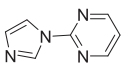
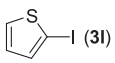
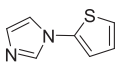
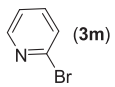
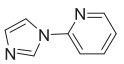
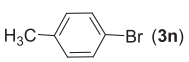
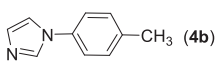
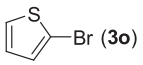
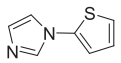
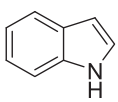
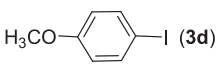
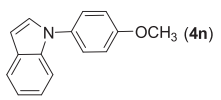
On the basis of the oxidative addition/reductive elimination mechanism [23] and the facts that copper ions can form chelates with cyclen derivatives, we put forward possible catalytic cycles for our reactions as depicted in Scheme 1. The chelation of Cu(I) with a cyclen derivative makes Cu(I) species more reactive toward the oxidative addition, or/and stabilize the oxidative addition intermediates **A**, thereby promoting the coupling reaction. The mechanism outlined in Scheme 1 could be used to explain the phenomena observed, for example, the order I > Br for ease of halogen displacement from the aromatic ring. But, more detailed mechanistic studies should be carried out to clarify these issues.

CONCLUSIONS

We have demonstrated a novel CuI-catalyzed method for the N-arylation of nitrogen heterocycles with aryl halides using cyclen derivatives as base and ligand that

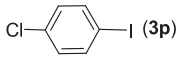
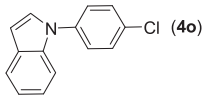
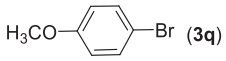
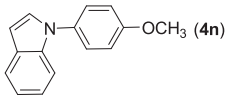
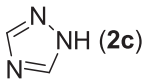
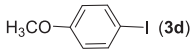
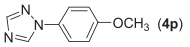
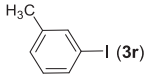
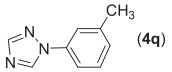
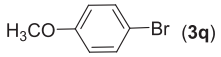
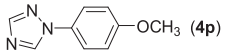
Table 2

CuI-catalyzed N-arylation of imidazole, indole, and triazole with various halides.[a]

Entry	Nitrogen heterocycle	ArX	Product	Yield (%)[b]
1	 (**2a**)	 (**3a**)	 (**4a**)	92
2		 (**3b**)	 (**4b**)	93
3		 (**3c**)	 (**4c**)	82
4		 (**3d**)	 (**4d**)	95
5		 (**3e**)	 (**4e**)	90
6		 (**3f**)	 (**4f**)	84
7		 (**3g**)	 (**4g**)	89
8		 (**3h**)	 (**4h**)	79
9		 (**3i**)	 (**4i**)	72
10		 (**3j**)	 (**4j**)	89
11		 (**3k**)	 (**4k**)	91
12		 (**3l**)	 (**4l**)	87
13		 (**3m**)	 (**4m**)	80
14		 (**3n**)	 (**4b**)	82
15		 (**3o**)	 (**4l**)	79
16	 (**2b**)	 (**3d**)	 (**4n**)	94

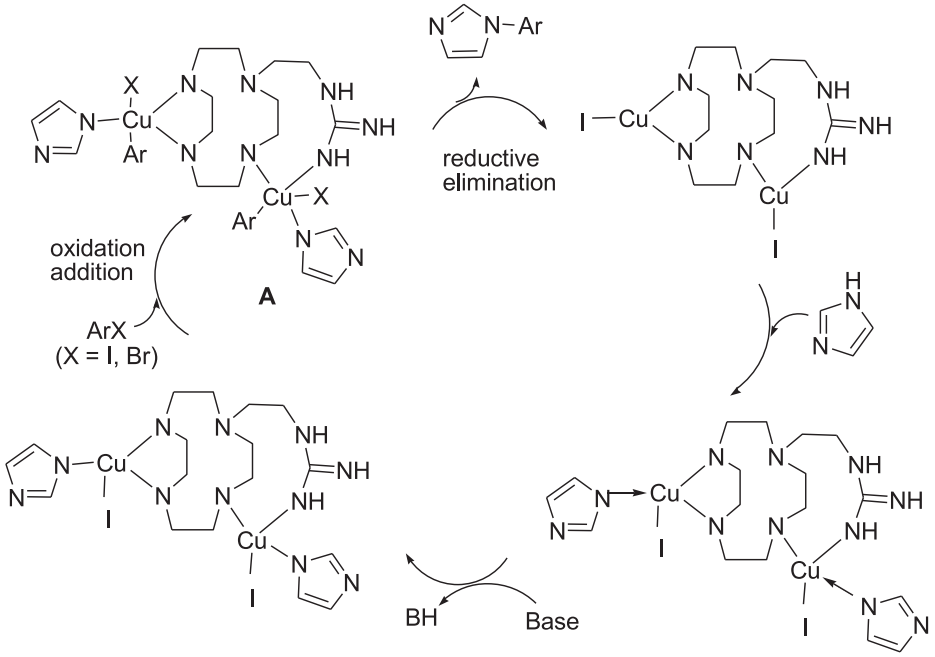
(*Continues*)

Table 2
(Continued)

Entry	Nitrogen heterocycle	ArX	Product	Yield (%)[b]
17		 (3p)	 (4o)	91
18		 (3q)	 (4n)	86
19	 (2c)	 (3d)	 (4p)	93
20		 (3r)	 (4q)	89
21		 (3q)	 (4p)	85

[a]Optimized reaction conditions.
[b]Isolated yields.

Scheme 1



proceeded in good to excellent product yields. The present protocol is applicable to a variety of nitrogen heterocycles and aryl halides containing electron-donating and electron-withdrawing properties. A detailed examination of the scope of the present reaction is currently underway.

EXPERIMENTAL

The reagents were purchased from Merck, ^{1}H and ^{13}C spectra were recorded in $CDCl_3$ on a 400 MHz instrument at 400 MHz (^{1}H) and 100 MHz (^{13}C) with TMS as internal standard; melting points were recorded on X4 microscope; HRMS were obtained

on OA-TOF mass spectrometer; Elemental analyses were performed using C, H, and N elemental analyzer; Flash column chromatography was performed with 300-mesh and 400-mesh silica gel.

General procedure for the synthesis of compounds 4. Aryl or heteroaryl halides (1 mmol), imodazole, indole or triazole (1 mmol), [Cu] (0.1 mmol), **1a–f** (2 mmol) and 2 mL DMSO were added to a 10 mL flask, which was subsequently capped with a rubber balloon. The mixture was stirred in a preheated oil bath at 100°C for appropriate time. After cooling the mixtures to the room temperature and partitioned between water and ethyl acetate, the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with 10% hydrochloric acid and water, dried over Na_2SO_4, and concentrated *in vacuo*. The residuals were purified by silica-gel column chromatography to afford corresponding products. The aqueous phase adjusted pH = 11 with 15% NaOH solution, ethyl acetate extraction, concentrate, and recycle **1a–f**.

1-(Phenyl)-1H-imidazole (4a). Colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.86 (s, 1H), 7.50–7.21 (m, 7H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 137.2, 135.5, 130.3, 129.8, 127.4, 121.4, 118.2. Anal. Calcd for $C_9H_8N_2$: C, 74.98; H, 5.59; N, 19.43. Found: C, 74.96; H, 5.58; N, 19.41.

1-(4-Methylphenyl)-1H-imidazole (4b). White solid, mp:45–46°C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.80 (s, 1H), 7.26–7.18 (m, 6H), 2.39 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 137.4, 135.6, 134.9, 130.3, 130.2, 121.3, 118.3, 20.9; Anal. Calcd for $C_{10}H_{10}N_2$: C, 75.92; H, 6.37; N, 17.71. Found: C, 75.93; H, 6.38; N, 17.71.

1-(2-Methylphenyl)-1H-imidazole (4c). White oil; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.58 (s, 1H), 7.35–7.32 (m, 2H), 7.30–7.27 (m, 1H), 7.22–7.20 (m, 2H), 7.05 (s, 1H), 2.18 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 137.3, 136.5, 133.7, 131.1, 129.2, 128.6, 126.7, 126.4, 120.3, 17.4; Anal. Calcd for $C_{10}H_{10}N_2$: C, 75.92; H, 6.37; N, 17.71. Found: C, 75.91; H, 6.38; N, 17.71.

1-(4-Methoxyphenyl)-1H-imidazole (4d). Colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.74 (s, 1H), 7.30–7.27 (m, 2H), 7.19–7.17 (m, 2H), 6.98–6.96 (m, 2H),3.83 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 158.8, 135.7, 130.6, 129.9, 123.0, 118.6, 114.8, 55.4; Anal. Calcd for $C_{10}H_{10}N_2O$: C, 68.95; H, 5.79; N, 16.08. Found: C, 68.94; H, 5.78, N, 16.06.

1-(4-Acetamidophenyl)-1H-imidazole (4e). White solid, mp:164–166°C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 10.08 (s, 1H), 8.16 (s, 1H), 7.70 (d, J = 8.8 Hz, 2H), 7.66 (s, 1H), 7.55 (d, J = 9.2 Hz, 2H), 7.08 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 168.9, 138.8, 135.8, 132.5, 130.2, 121.3, 120.3, 118.3, 24.2; Anal. Calcd for $C_{11}H_{11}N_3O$: C, 65.66; H, 5.51; N, 20.88. Found: C, 65.78; H, 5.55; N, 20.68.

1-(4-Fluorophenyl)-1H-imidazole (4f). Colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.78 (s, 1H), 7.37–-7.33 (m, 2H), 7.21–7.14 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 162.8, 160.3, 134.7 (J = 194.4 Hz), 130.3, 123.4 (J = 8.2 Hz), 118.5, 116.6 (J = 22.7 Hz); Anal. Calcd for $C_9H_7FN_2$: C, 66.66; H, 4.35; F, 11.72; N, 17.27. Found: C, 66.67; H, 4.35, F, 11.71; N, 17.27.

1-(4-Trifluoromethylphenyl)-1H-imidazole (4g). White solid, mp:69-70°C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.93 (s, 1H), 7.76 (d, J = 8.0 Hz, 2H), 7.53 (d, J = 8.0 Hz, 2H), 7.34 (s, 1H), 7.72 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 139.9, 135.4, 131.1, 129.5 (J = 29.6, 62.5 Hz), 127.2, 123.6 (J = 272.1, 549.0 Hz), 121.2, 117.8; HRMS (M^+): Calcd for $C_{10}H_7F_3N_2$ 212.0561, found 212.0560.

1-(4-Nitrohenyl)-1H-imidazole (4h). White solid, mp:201–202°C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.39 (d, J = 9.0 Hz, 2H), 7.99 (s, 1H), 7.58–7.61 (m, 2H), 7.39 (s, 1H), 7.29 (s, 1H), 6.46 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 146.5, 142.2, 132.0, 126.0, 121.3, 117.9, 105.0; HRMS (M^+): Calcd for $C_9H_7N_2O_2$ 189.0538, found 189.0538.

1-(2-Nitrohenyl)-1H-imidazole (4i). White solid, mp:79-80°C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.01 (d, J = 8.1 Hz, 1H), 7.73–7.77 (m, 1H), 7.61–7.62 (m, 2H), 7.48 (d, J = 7.9 Hz, 1H), 7.22 (s, 1H), 7.08 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 140.3, 132.2, 126.7, 125.5, 125.3, 124.6, 123.6, 120.3, 115.3; HRMS (M^+): Calcd for $C_9H_7N_2O_2$ 189.0538, found 189.0538.

1-(Pyridine-3-yl)-1H-imidazole (4j). Yellow oil; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.71 (d, 1H), 8.60–8.58 (m, 1H), 7.84 (s, 1H), 7.42–7.39 (m, 1H), 7.42–7.39 (m, 1H), 7.27–7.25 (m, 1H), 7.21 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 148.7, 142.8, 135.5, 133.8, 131.1, 128.8, 124.2, 118.0; HRMS (M^+): Calcd for $C_8H_7N_3$ 145.0640, found 145.0640.

2-(1H-imidazol-1-yl)-pyrimidine (4k). White solid, mp:122–123°C;^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.68–8.67 (d, 2H), 8.60 (s, 1H), 7.87 (t, 1H), 7.19–7.15 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 158.7, 154.7, 136.1, 130.6, 118.8, 116.5; HRMS (M^+): Calcd for $C_7H_6N_4$ 146.0592, found 146.0591.

1-(Pyridine-2-yl)-1H-imidazole (4l). Yellow oil; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.64 (s, 1H), 8.35 (s, 1H), 7.82–7.77 (m, 1H), 7.64–7.63 (t, 1H), 7.36–7.33 (m, 1H), 7.24–7.19 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 149.1, 138.9, 134.9, 130.5, 121.9, 116.0, 112.2; HRMS (M^+): Calcd for $C_8H_7N_3$ 145.0640, found 145.0641.

1-(Thien-2-yl)-1H-imidazole (4m). Yellow oil; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.43 (dd, 1H), 7.24–7.17 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 136.2, 135.7, 129.9, 127.1, 121.3, 118.4, 113.1; HRMS (M^+): Calcd for $C_7H_6N_2S$ 150.0252, found 150.0251.

1-(4-Methoxyphenyl)-1H-indole (4n). Yellow solid, mp:48–50°C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.69 (d, J = 7.3 Hz, 1H), 7.40–.48 (m, 3H), 7.28 (d, J = 3.0 Hz, 1H), 7.13–7.23 (m, 2H), 7.04 (d, J = 8.7 Hz, 2H), 6.66 (d, J = 3.0 Hz, 1H), 3.86 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 158.6, 136.7, 133.3, 129.4, 128.8, 126.4, 122.6, 121.5, 120.5, 115.2, 111.8, 103.3, 56.1; HRMS (M^+): Calcd for $C_{15}H_{13}NO$ 223.0997, found 223.0997.

1-(4-Chlorophenyl)-1H-indole (4o). Yellow oil; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.69 (d, J = 7.1 Hz, 1H), 7.43-7.53 (m, 5H), 7.17–7.30 (m, 3H), 6.69 (d, J = 2.8 Hz, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 138.6, 135.9, 132.2, 130.0, 129.5, 127.9, 125.7, 122.8, 121.5, 120.8,111.5, 104.2; HRMS (M^+): Calcd for $C_{14}H_{10}ClN$ 227.0502, found 257.0503.

1-(4-Methoxyphenyl)-1H-1,2,4-triazole (4p). White solid, mp : 87–88°C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.55 (s, 1H), 8.10 (s, 1H), 7.58 (d, J = 8.8 Hz, 2H), 7.02 (d, J = 8.9 Hz, 2H), 3.87 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 159.6, 152.7, 141.4, 130.6, 122.0, 115.0; HRMS (M^+): Calcd for $C_9H_9N_3O$ 175.0746, found 175.0747.

1-m-Tolyl-1H-1,2,4-triazole (4q). Colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 8.52 (s, 1H), 8.07 (s, 1H), 7.48 (s, 1H), 7.42 (d, J = 8.1 Hz, 1H), 7.35 (d, J = 7.7 Hz, 1H), 7.17 (d, J = 7.5 Hz, 1H), 2.40 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 152.7,141.1, 140.2, 137.1, 129.8, 129.2, 120.9, 117.3, 21.6; HRMS (M^+): Calcd for $C_9H_9N_3$ 159.0800, found 159.0797.

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