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Visible-Light-Mediated Nickel(II)-Catalyzed C−N Cross-Coupling in Water: Green and Regioselective Access for the Synthesis of Pyrazole-Containing Compounds

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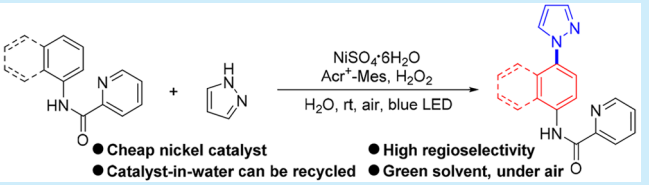
Supporting Information

ABSTRACT: A regioselective green approach for the nickel-(II)-catalyzed C−N cross-coupling between arylamines and pyrazoles through a photoredox process is reported. Moderate to good yield was observed for this reaction, performed in water under air at room temperature. This strategy provides a powerful tool for the green synthesis of pyrazole-containing bioactive molecules. In addition, a single-electron-transfer mechanism is proposed in this report.

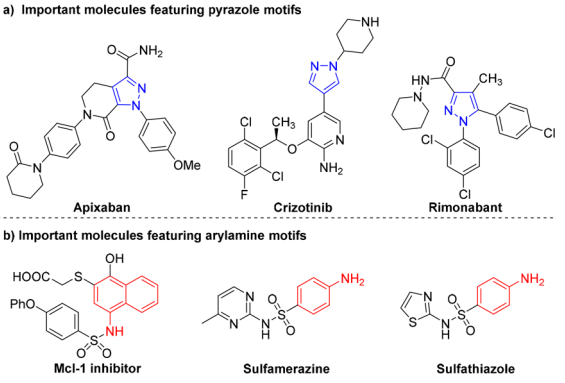


Transition-metal-catalyzed C−H functionalization has emerged as a reliable strategy for rapid synthesis of organic intermediates because of its high synthetic efficiency and atom economy.[1] In particular, oxidative C−H/N−H cross-coupling is a hot topic of current research because nitrogen-containing compounds widely exist in many pharmaceuticals and natural products, and much attention has been paid to it by chemists.[2] Over the past decade, the field of metal-catalyzed oxidative C−H/N−H cross-coupling has progressed with novel developments using various transition-metal catalysts including palladium,[3] copper,[4] and other metals.[5] Nickel, as an inexpensive and abundant catalyst, has recently received considerable attention in C−H bond functionalization,[6] with minimal reports to extend this method to alternative bond formation. In particular, the construction of C−N bonds via nickel-catalyzed C−H functionalization has rarely been reported.[6a,b]

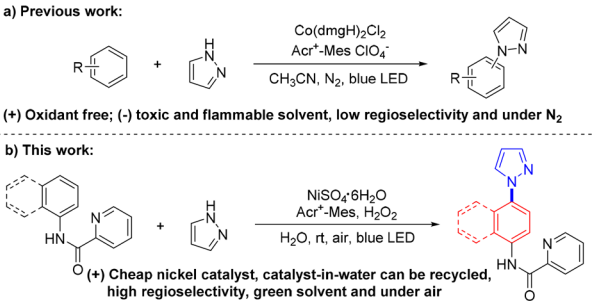
Pyrazole frameworks have been of great interest in recent years because of their frequent appearances in bioactive compounds (Scheme 1a).[7] Consequently, it is important to develop simple and efficient strategies for the preparation of functionalized pyrazoles.[8] In 2017, Niu and co-workers reported a photoinduced, oxidant-free, oxidative C−H/N−H cross-coupling between arenes and azoles (Scheme 2a).[8a] Despite this crucial advancement in the field, such reactions use toxic and flammable solvents, achieve low regioselectivity, and require an N_2 atmosphere. Because of these prerequisites, these reactions fail to meet demands of effective and efficient green chemistry. In recent years, many efforts have also been conducted toward the direct modification of arylamines[9] as a critical scaffold in the manufacturing of many important pharmaceutical molecules (Scheme 1b).[10]

Recently, photocatalysis has become a hot area of research because it is a powerful tool in green organic synthesis with lower energy consumption and higher atom economy. The preeminent advances have been showed by MacMillan[11] and other groups.[12] In addition, an increasing number of aqueous phase reactions recently have been gradually established for the synthesis of organic compounds because water is acknowl-

Scheme 1. Representative Examples of Important Molecules



Scheme 2. C−H/N−H Cross-Coupling between Arenes and Azoles



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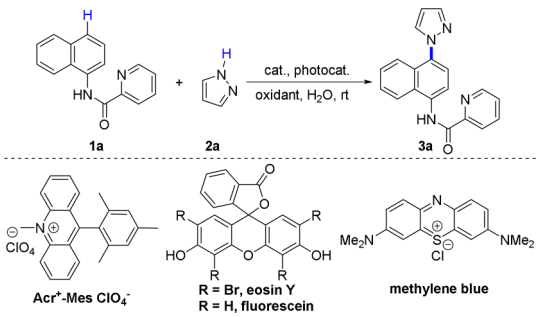
edged as a cheap, green, and nontoxic solvent.[13] The emergence of such a technique sets the foundation for the development trend of organic synthesis and catalysis fields in the future. Herein, we demonstrate the green synthesis of pyrazole-containing compounds via visible-light-mediated nickel(II)-catalyzed C−N cross-coupling in water under mild conditions (Scheme 2b).

Photocatalysts including Acr^+-$MesClO_4^-$, eosin Y, fluorescein, and methylene blue have been widely used in photocatalysis due to the advantage of low price.[14] Hence, the reaction between amide (**1a**) and pyrazole (**2a**) was initially performed in H_2O in the presence of $NiCl_2$ (15 mol %), Acr^+-$MesClO_4^-$ (5 mol %), and $K_2S_2O_8$ (0.4 mmol) under irradiation of 12 W blue LED. The desired product **3a** was isolated in 23% yield (Table 1, entry 1). Subsequently, various

Table 1. Optimization of Reaction Conditions[a,b]



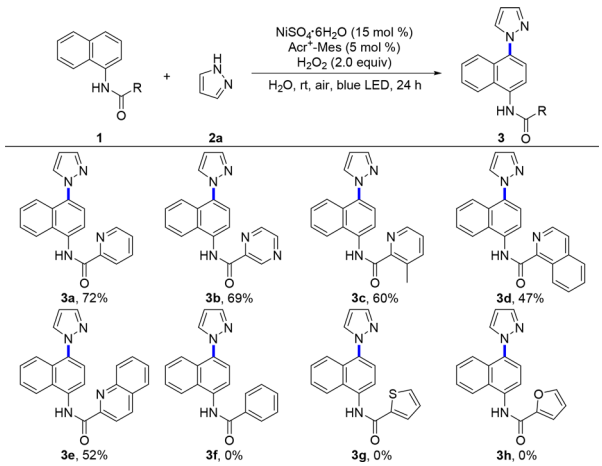
entry	catalyst	photocatalyst	oxidant	yield[b] (%)
1	$NiCl_2$	Acr^+-$MesClO_4^-$	$K_2S_2O_8$	23
2	$NiBr_2$	Acr^+-$MesClO_4^-$	$K_2S_2O_8$	32
3	NiI_2	Acr^+-$MesClO_4^-$	$K_2S_2O_8$	0
5	$NiSO_4{\cdot}6H_2O$	Acr^+-$MesClO_4^-$	$K_2S_2O_8$	52
6	$Ni(OTf)_2$	Acr^+-$MesClO_4^-$	$K_2S_2O_8$	37
7		Acr^+-$MesClO_4^-$	$K_2S_2O_8$	0
8	$NiSO_4{\cdot}6H_2O$	Acr^+-$MesClO_4^-$	$Na_2S_2O_8$	46
9	$NiSO_4{\cdot}6H_2O$	Acr^+-$MesClO_4^-$	Oxone	0
10	$NiSO_4{\cdot}6H_2O$	Acr^+-$MesClO_4^-$	TBHP	42
11	$NiSO_4{\cdot}6H_2O$	Acr^+-$MesClO_4^-$	H_2O_2	72
12	$NiSO_4{\cdot}6H_2O$	Acr^+-$MesClO_4^-$	DTBP	21
13	$NiSO_4{\cdot}6H_2O$	Acr^+-$MesClO_4^-$	O_2	13
14	$NiSO_4{\cdot}6H_2O$	Acr^+-$MesClO_4^-$		0
15	$NiSO_4{\cdot}6H_2O$	eosin Y	H_2O_2	59
16	$NiSO_4{\cdot}6H_2O$	fluorescein	H_2O_2	0
17	$NiSO_4{\cdot}6H_2O$	methylene blue	H_2O_2	0
18	$NiSO_4{\cdot}6H_2O$		H_2O_2	0
19[c]	$NiSO_4{\cdot}6H_2O$	Acr^+-$MesClO_4^-$	H_2O_2	70

[a]Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), catalyst (15 mol %), photocatalyst (5 mol %), blue LED (12 W), oxidant (0.4 mmol), H_2O (4 mL), rt, air, 24 h. [b]Isolated yields based on **1a**. [c]Under N_2 atmosphere.

nickel catalysts including $NiBr_2$, NiI_2, NiF_2, and $NiSO_4{\cdot}6H_2O$ as well as $Ni(OTf)_2$ were screened (Table 1, entries 2−6). The yield of compound **3a** was enhanced to 52% when $NiSO_4{\cdot}6H_2O$ was utilized as catalyst. Subsequently, the effects of oxidants on influencing reaction yields were investigated. Oxidants such as $Na_2S_2O_8$, Oxone, *tert*-butyl hydroperoxide (TBHP), H_2O_2, di-*tert*-butyl peroxide (DTBP), and O_2 (Table 1, entries 8−13) were observed to demonstrate interesting improvement in yields. The yield of product **3a** was increased to 72% by employing H_2O_2 as the oxidant. Expectedly, no target product was detected in the absence of catalyst, photocatalyst, or oxidant (Table 1, entries 7, 14, and 18). The yield was not increased when the reaction was performed under N_2 atmosphere (Table 1, entry 19). Further tests on different photocatalysts and light sources also did not improve the yield (see the Supporting Information).

After the reaction conditions were optimized, efforts were focused on determining better directing groups for the C−N cross-coupling (Scheme 3). Some amides, prepared from the

Scheme 3. Investigation of Directing Group[a,b]



[a]Reaction conditions: **1** (0.2 mmol), **2a** (0.4 mmol), $NiSO_4{\cdot}6H_2O$ (15 mol %), Acr^+-$MesClO_4^-$ (5 mol %), blue LED (12 W), H_2O_2 (0.4 mmol), H_2O (4 mL), rt, air, 24 h. [b]Isolated yields based on **1**.

amidation of aromatic acid and naphthylamine, were employed as substrates. Product **3a** was obtained at 72%, yield while other substrates with *N*-containing directing groups were converted into the corresponding products with low yields (**3b−e**). Regrettably, products (**3f−h**) could not be detected. These phenomena revealed that the N of pyridine moiety played an important role in this reaction. On the basis of these results, picolinamide (PA) is likely a good choice for directing C−N cross-coupling reactions.

Next, the substrate scope was explored (Scheme 4). Generally, pyrazole derivatives bearing different substituent groups could be transformed into related products in moderate to good yields. Pyrazoles with methyl and phenyl groups were compatible, affording the corresponding products in 62−74% yields (**3i**, **3j**, **3m**, and **3p**). Halogen-containing substrates, which could be further functionalized, were also tolerated under standard conditions (**3n**, **3o**, **3q**, and **3r**). Moreover, benzotriazole, which could be utilized as a corrosion inhibitor of copper, was also found to be compatible and offer the desired product in 44% yield (**3s**).[15] Additionally, other naphthalene amides could also be converted into the corresponding products in acceptable yields by using pyrazole derivatives as copartners (**3t−x**). Interestingly, two reactions were carried out by using Triton X-100[13b] and Nok[13c] as surfactant, respectively, and product **3w** could be obtained in higher yields. Unfortunately, it was observed that pyrazole derivatives with electron-withdrawing groups (−CN and $-NO_2$), could not undergo the C−N cross-coupling reaction (**3k**, **3l**). The unique role of aniline derivatives in pharmaceutical agents encouraged the attempt to apply these conditions to aniline derivatives. Initial reactions show an

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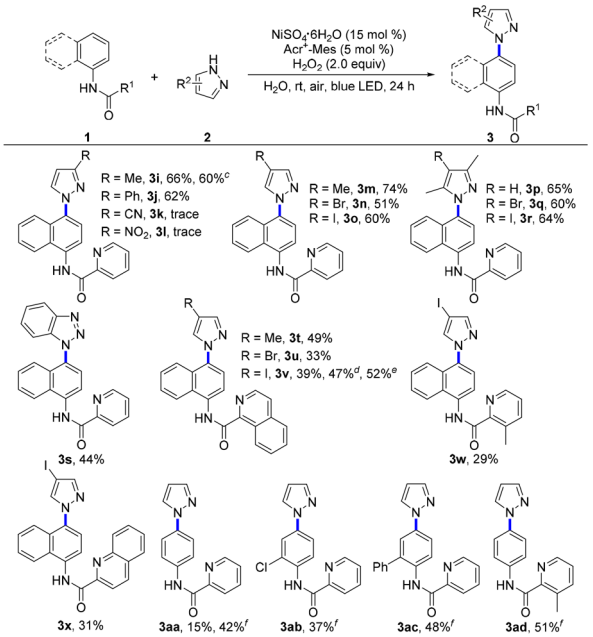
Scheme 4. Substrate Scope[a,b]

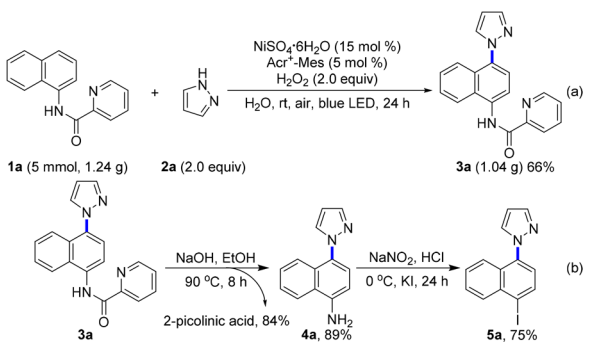


[a]Reaction conditions: **1** (0.2 mmol), **2** (0.4 mmol), $NiSO_4·6H_2O$ (15 mol %), Acr^+-$MesClO_4^-$ (5 mol %), blue LED (12 W), H_2O_2 (0.4 mmol), H_2O (4 mL), rt, air, 24 h. [b]Isolated yields based on **1**. [c]Reaction was performed on a 1 mmol scale. [d]Triton X-100/water (2 wt %). [e]Nok/water (2 wt %). [f]**2a** (0.8 mmol), H_2O_2 (0.8 mmol), 48 h.

isolation of product **3aa** at low 15% yield when *N*-phenylpicolinamide was prepared from the amidation of picolinic acid and aniline. To improve the yield, amounts of pyrazole and H_2O_2 were increased to 4 equiv. Furthermore, the reaction time was extended to 48 h. The yield of product **3aa** was improved to 42%. Other substrates were also converted into corresponding products in acceptable yields (**3ab**–**ad**). Unfortunately, this new reaction condition did not improve the reactivity of naphthalene amides in this C–N cross-coupling reaction.

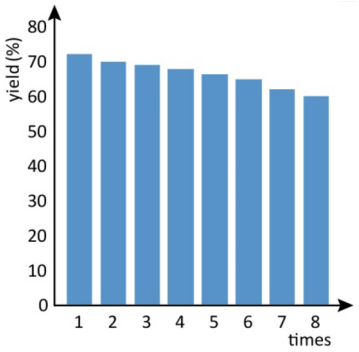
To show the application value of this method, a gram-scale synthesis was performed, and the product **3a** was isolated in 66% yield (Scheme 5a). Subsequently, hydrolysis product **4a** and 2-picolinic acid was obtained in 89% and 84% yields, respectively, through a hydrolysis reaction. Then the hydrolysis product **4a** was transformed into iodine-containing product which could be used as pyrazole-containing intermediate to form C–C bonded compounds by the Suzuki–Miyaura coupling, the Heck reaction, and the Sonogashira reaction (Scheme 5b).

Scheme 5. Synthetic Applications



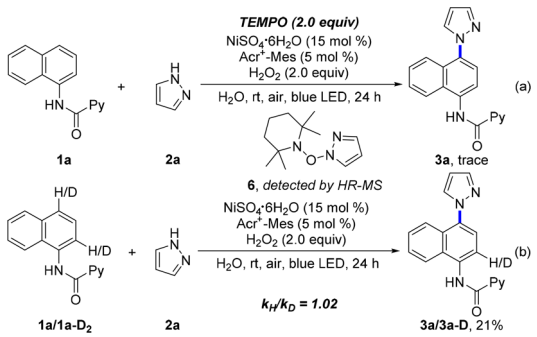
Our goal is to develop a green and atom-economic strategy for the preparation of pyrazole-containing compounds. Therefore, the recycling of catalyst-in-water was studied. According to the difference of solubility to nickel(II) salt and organic compounds in H_2O and EtOAc, the mixture was extracted with EtOAc after the transformation was completed. Then, the catalyst-in-water (contains nickel(II) salt and H_2O) could be retrieved by an easy phase separation. Finally, the retrieved catalyst-in-water was reutilized in the next round by the addition of starting materials. The activity of the catalyst remained stable after eight recycles (Scheme 6).

Scheme 6. Recycling Catalyst-in-Water System



Mechanistic studies on the investigation of molecular structure, radical inhibition experiment, and kinetic isotope effect (KIE) were extensively researched. First, the analogue substrates failed to afford the desired products **3ae** and **3af** (see the SI). These results indicated that the NH of the amide moiety and the N of the pyridine moiety (Scheme 3) were indispensable in this C–N cross-coupling. In addition, when TEMPO was used as radical inhibitor, the coupling reaction was absolutely suppressed, and an adduct (**6**) of TEMPO and pyrazole radical was detected by high-resolution mass spectrometry (HRMS) (Scheme 7a). These results suggest

Scheme 7. Mechanism Studies



the presence of a radical pathway that might be responsible for this reaction. Subsequent tests in kinetic isotope effect (KIE) provided a low ratio (k = 1.02) (Scheme 7b), revealing that the process of C–H bond cleavage was not the rate-determining step.[16]

Finally, according to the results of mechanism studies and previous works,[17] a reasonable mechanism was presented (Scheme 8). Initially, the photocatalyst (PC) was excited by

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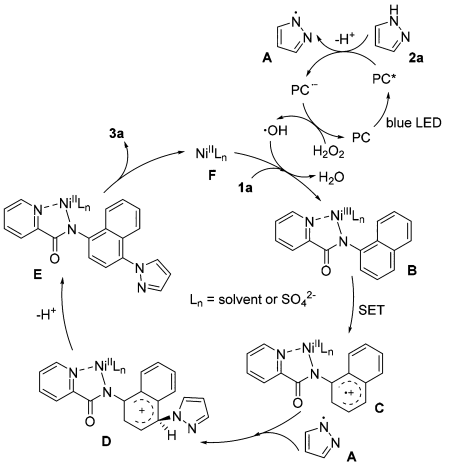
Scheme 8. Plausible Mechanism



visible light irradiation to form the excited species PC*, which underwent the single-electron transfer (SET) process with pyrazole (**2a**) to generate pyrazole radical **A** and $PC^{\bullet-}$. Subsequently, $PC^{\bullet-}$ was oxidized by H_2O_2 to generate PC with the formation of hydroxyl radical. Meanwhile, $Ni^{II}L_n$ combined with substrate **1a** followed by oxidation of the hydroxyl radical to generate aryl-$Ni^{III}L_n$ complex **B** and H_2O. Then, aryl-$Ni^{II}L_n$ complex **C** was formed through the process of SET. Next, pyrazole radical **A** attacked aryl-$Ni^{II}L_n$ complex **C** to generate aryl-$Ni^{II}L_n$ complex **D**. After the formation of aryl-$Ni^{II}L_n$ complex **E** via deprotonation, target product **3a** was obtained through a metal-dissociation process.

In conclusion, a visible-light-mediated nickel(II)-catalyzed protocol for C−N cross-coupling between arylamine and pyrazole is reported. This method affords an efficient and green solution for the construction of pyrazole-containing bioactive molecules from simple starting materials. A single-electron-transfer process was also deduced to be critical in this C−N cross-coupling reaction.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b01395.

Experimental procedures, characterization data, and ^1H and ^{13}C NMR spectra for the synthesized compounds (PDF)

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Notes

The authors declare no competing financial interest.

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