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A Carbonylation Approach Toward Activation of C_{sp2}-H and C_{sp3}-H Bonds: Cu-Catalyzed Regioselective Cross Coupling of Imidazo[1,2-*a*]pyridines with Methyl Hetarenes

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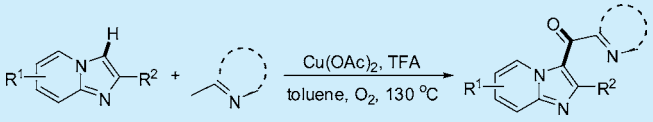
Supporting Information

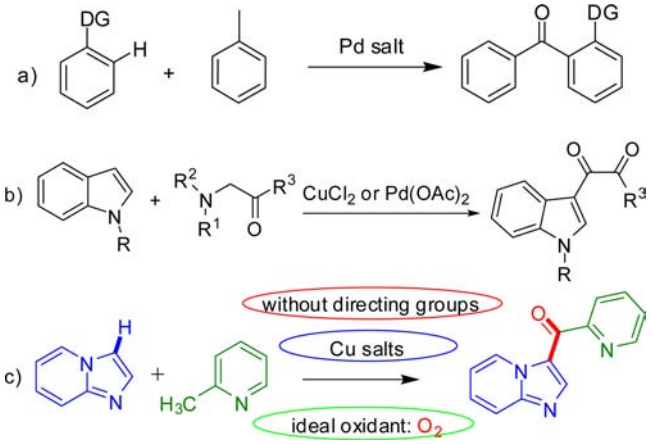
ABSTRACT: An efficient copper-catalyzed selective cross coupling of imidazo[1,2-*a*]pyridines with methyl hetarenes has been reported. This transformation opened a new route to synthesize the C-3 carbonyl imidazo[1,2-*a*]pyridine derivative, which is a common structural motif in natural products and pharmaceuticals. ^{18}O-labeling experiments indicated that the oxygen source of products originated from O_2.



Transition metal-catalyzed carbon−carbon bond formation[1] is one of the most powerful tools in organic synthesis. During the last several decades, C−H activation, especially transition metal-catalyzed C−H activation,[2] has gained considerable attention in constructing carbon−carbon bonds for the preparation of useful compounds. Most recently, the versatility of transition metal catalysts in activating both C_{sp2}-H and C_{sp3}-H for subsequent carbon−carbon bond formation[3] in one reaction has allowed the discovery of a variety of new reactions. This type of direct cross-coupling of two "unactivated" agents is appealing because of their high atom efficiency and easy accessibility of starting materials. The carbonylation approach[4] toward the activation of C_{sp2}-H and C_{sp3}-H bonds has attracted interesting research (Scheme 1a and b)[5] and become a challenging goal due to the prevalent presence of the carbonyl group,[6] which should work nicely in each step during the reaction.

Imidazo[1,2-*a*]pyridines and their derivatives represent an important class of compounds[7] that are closely related to the life sciences.[8] Recent studies have indicated that these molecules show unique bioactivities and chemical properties[9] that lead them to broad applications in modern organic synthesis,[10] medicinal chemistry,[11] and materials chemistry. Although extensive study in this area has been conducted,[12] the development of new routes is still highly desirable for the synthesis of functionalized imidazo[1,2-*a*]pyridines. For example, carbonyl-substituted imidazo[1,2-*a*]pyridines are desirable target molecules due to their potential bioactivities. As a consequence, the development of improved methods for the synthesis of carbonyl-substituted imidazo[1,2-*a*]pyridine derivatives via direct cross-coupling methods that avoid organometallic reagents have become important research fields. Actually, designing an efficient cross-coupling reaction for selective construction of imidazo[1,2-*a*]pyridines[13] continues to attract our attention. Currently, our interest is focused on developing a facile strategy to construct carbonyl imidazo[1,2-*a*]pyridine derivatives via cross-coupling of C_{sp2}-H and C_{sp3}-H bonds (Scheme 1c).

Scheme 1. Carbonylation Strategy Toward Activation of C_{sp2}-H and C_{sp3}-H bonds



For the reaction conditions to be standardized, a series of experiments were performed with variation of the reaction parameters, including the oxidant, additive, solvent, catalyst loading, and temperature, for a representative cross-coupling of imidazo[1,2-*a*]pyridine (**1a**) with 2-methylpyridine (**2a**). The results are presented in Table 1. Initial experiments were carried out using 0.5 mmol **1a**, 0.7 mmol **2a**, 0.15 mmol AcOH, and toluene (3 mL) together with 10 mol % $Cu(AcO)_2$ as catalyst in a sealed tube at 130 °C under oxygen for 12 h (Table 1, entry 1). Desired product **3a** was formed in 36% yield. Other catalysts, such as $Cu(OTf)_2$, $CuCl_2$, $CuBr_2$, CuBr, and CuI, were examined (Table 1, entries 2−6). The results indicated

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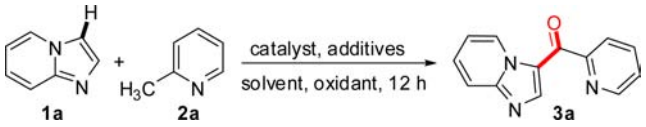
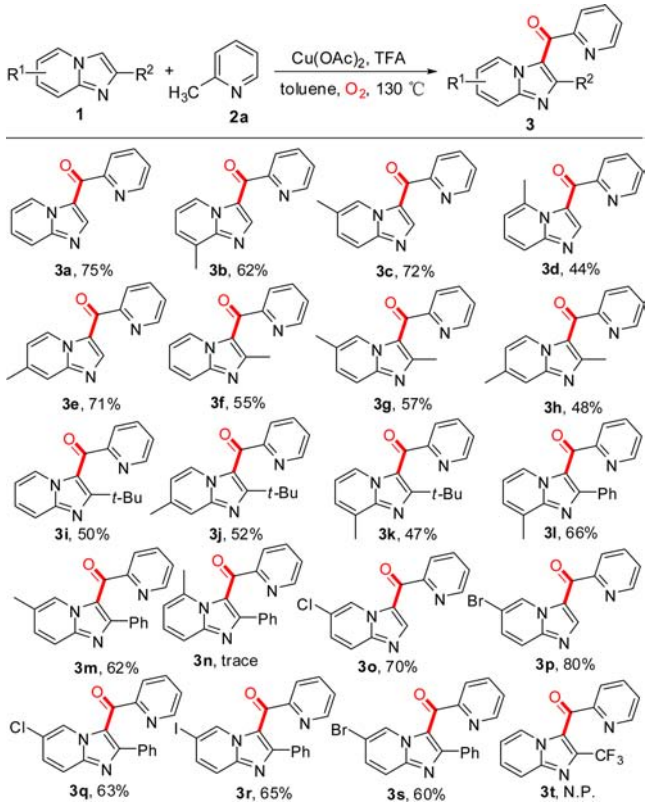
Table 1. Optimization of Reaction Conditions[a]



entry	catalyst	oxidant	additive	solvent	temp	yield(%)[b]
1	$Cu(OAc)_2$	O_2	HOAc	toluene	130	36
2	$Cu(OTf)_2$	O_2	HOAc	toluene	130	28
3	$CuCl_2$	O_2	HOAc	toluene	130	15
4	$CuBr_2$	O_2	HOAc	toluene	130	<10
5	CuBr	O_2	HOAc	toluene	130	13
6	CuI	O_2	HOAc	toluene	130	10
7[c]	$Cu(OAc)_2$	O_2	HOAc	toluene	130	trace
8	$Cu(OAc)_2$	TBHP	HOAc	toluene	130	35
9	$Cu(OAc)_2$	$K_2S_2O_8$	HOAc	toluene	130	N.P.
10	$Cu(OAc)_2$	DDQ	HOAc	toluene	130	19
11	$Cu(OAc)_2$	AgOAc	HOAc	toluene	130	30
12	$Cu(OAc)_2$	O_2	TFA	toluene	130	82
13	$Cu(OAc)_2$	O_2	K_2CO_3	toluene	130	N.P.
14	$Cu(OAc)_2$	O_2	$AlCl_3$	toluene	130	N.P.
15	$Cu(OAc)_2$	O_2	$ZnCl_2$	toluene	130	N.P.
16	$Cu(OAc)_2$	O_2	TFA	DMF	130	trace
17	$Cu(OAc)_2$	O_2	TFA	DMSO	130	trace
18	$Cu(OAc)_2$	O_2	TFA	dioxane	130	trace
19	$Cu(OAc)_2$	O_2	TFA	toluene	150	77
20	$Cu(OAc)_2$	O_2	TFA	toluene	100	56
21	$Cu(OAc)_2$	O_2	TFA	toluene	rt	N.P.

[a]Reaction conditions: **1a** (0.5 mmol), **2a** (0.7 mmol), catalyst (10 mol %), additives (0.15 mmol), oxidant (1.0 mmol), solvent (3.0 mL), rt−150 °C, 12 h, carried out in a sealed tube (25 mL). [b]GC yield. [c]O_2 (500 mL).

Scheme 2. Cross-Coupling of Imidazo[1,2-*a*]pyridines with 2-Methylpyridine[a]



[a]Isolated yields.

that $Cu(OAc)_2$ was most effective among the metal catalysts. Only a trace of product **3a** was formed when the reaction was carried out in the presence of $Cu(OAc)_2$ and AcOH using an oxygen ballon (500 mL) (Table 1, entry 7). Different oxidants, such as TBHP, $K_2S_2O_8$, DDQ, and AgOAc, were tested, and experiments indicated that O_2 gave the best yield compared with those of other oxidants (Table 1, entries 8−11). For the yield to be improved, other additives were employed. To our delight, desired product **3a** was generated in 82% yield (Table 1, entry 12) using TFA as an additive, which indicated that a strong acid could promote the reaction. However, when K_2CO_3, $AlCl_3$, or $ZnCl_2$ was employed as the additive, no product **3a** was obtained (Table 1, entries 13−15). Among the solvents tested, toluene was shown to be the most effective in comparison to DMF, DMSO, and dioxane (Table 1, entries 16−18). A decreased yield was obtained after increasing the reaction temperature to 150 °C or lowering the reaction temperature to 100 °C (Table 1, entries 19 and 20). No product **3a** was detected when the reaction was performed at room temperature (Table 1, entry 21).

For the scope of this Cu-catalyzed selective cross-coupling reaction to be examined, different imidazo[1,2-*a*]pyridine derivatives were employed under the conditions optimized above. The results are outlined in Scheme 2. Our experiments were smooth under the optimized conditions in most cases and provided the C-3 carbonylation imidazo[1,2-*a*]pyridines in moderate to good yields. A variety of substituents, such as 8-Me, 7-Me, 6-Me, and 5-Me, on the pyridine ring of imidazo[1,2-*a*]pyridine were well-tolerated under the optimized conditions. Interestingly, F, Cl, Br, and I substituted on the pyridine ring of imidazo[1,2-*a*]pyridine were also performed under these conditions and led to a beneficial effect on the reaction outcome, which made further functionalization possible to prepare complex compounds. Furthermore, most of the groups, such as 2-Me, 2-Ph, and 2-*t*-Bu, on the imidazo ring of imidazo[1,2-*a*]pyridine worked very well. Except for 5-methyl-2-phenylimidazo[1,2-*a*]pyridine being used as the substrate, only a trace amount of desired product **3n** was detected by GCMS, which was caused by steric hindrance of the substrate. However, imidazo[1,2-*a*]pyridines with electron-withdrawing groups (CF_3) at C-2 could not perform the carbonylation with **2a**.

The scope of this Cu-catalyzed highly regioselective coupling reaction has been further explored by reacting imidazo[1,2-*a*]pyridines with different methyl hetarenes; the results are summarized in Scheme 3. As expected, various 2-methylpyridines with electron-donating methyl or ethyl groups have coupled with imidazo[1,2-*a*]pyridine to furnish the corresponding C-3 carbonylation products in moderate to good yields under the standard reaction conditions (**4a**−**4f**). However, only a trace amount of desired product **4n** was detected by GCMS. We were delighted to find that the presence of electron-withdrawing groups (**4g**−**4i**) on the pyridine ring, such as Br and Cl, were tolerated in the reaction and afforded the desired products smoothly. This new transformation was further found to be successfully applied to catalyze the carbonylation of imidazo[1,2-*a*]pyridine with 2-methylquinoline, 2-methylpyrazine, 2-chloro-3-methylpyrazine, or 2,5-dimethylthiazole, affording the desired products in moderate to good yields (**4j**−**4m**). These results showed that this cross-coupling

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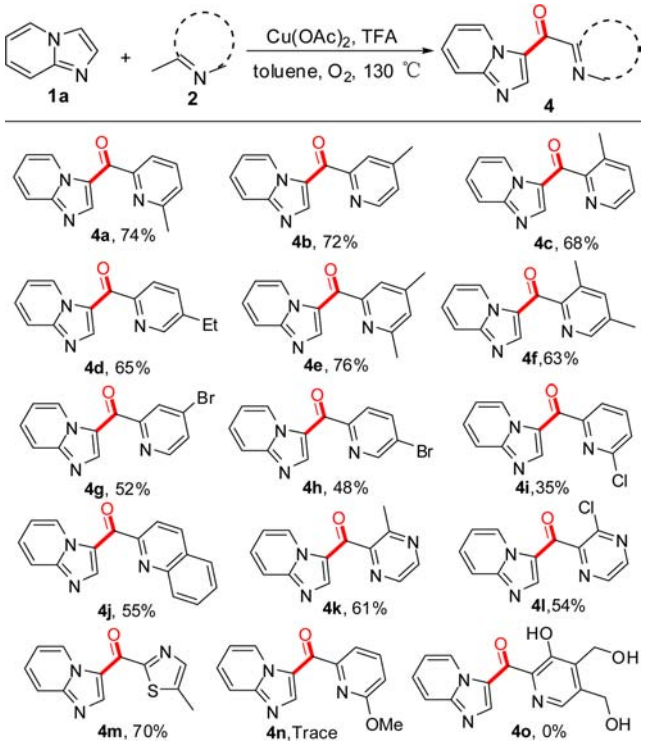
Scheme 3. Cu-Catalyzed Carbonylation of Imidazo[1,2-*a*]pyridine with Methyl Hetarenes[a]



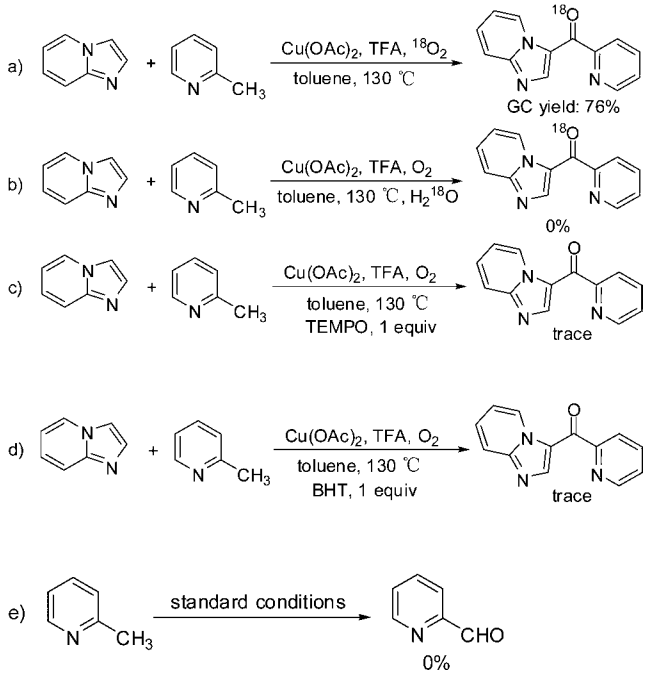
[a]Isolated yields.

reaction turned out to be a versatile process. All of the results indicated that this process was highly regioselective for C-3 carbonylation. Vitamin B6 was next used as a substrate to test the Cu-catalyzed carbonylation reaction. Unfortunately, product **4o** was not formed under the optimized conditions.

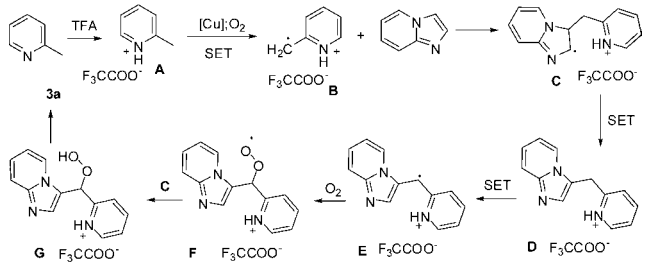
For more insight into the Cu-catalyzed carbonylation approach to be obtained, several control experiments were performed. First, a labeling experiment with $^{18}O_2$ as oxidant was conducted in the reaction, and ^{18}O-labeled product **3a** was obtained (Scheme 4a). In addition, $H_2{}^{18}O$ was added to the reaction (Scheme 4b), and ^{18}O-labeled product **3a** was not detected by GCMS. Those results indicated that the carbonyl oxygen atom of **3a** originated from O_2. To confirm an organic radical species involved in the overall process, we then carried out the radical capture reactions by adding a radical-trapping reagent (TEMPO) (Scheme 4c) or a radical inhibitor (BHT) (Scheme 4d) to the reaction system, and desired product **3a** was not detected. These observations demonstrated that there should be a radical pathway in this reaction. As shown in Scheme 2e, the initial step of the reaction was coupling of **1a** with **2a** and not the formation of the benzaldehyde.

On the basis of the results provided above and the results of related reports,[14] we have proposed a mechanism for this transformation, which is shown in Scheme 5. Initially, intermediate **A** is formed by interaction between 2-methylpyridine and TFA. Then, intermediate **A** underwent single electron transfer (SET) oxidation in the presence of Cu(II) species to form radical **B**. Radical **B** could directly add to imidazo[1,2-*a*]pyridine **2a** to give radical **C**, which then underwent a sequential single electron transfer (SET) process and proton transfer to generate intermediate **E**. Subsequently, intermediate **E** captured O_2 to generate radical **F**, which reacted with another

Scheme 4. Control Experiments for Investigation of the Mechanism



Scheme 5. Possible Mechanism



intermediate **D** to give intermediate **G**. Finally, desired product **3a** was formed and released a TFA.

In conclusion, we have developed a novel copper-catalyzed carbonylation of imidazo[1,2-*a*]pyridines with methyl hetarenes. The C_{sp2}-H functionalization approach was featured with high regioselectivity without any directing group. The mechanism was investigated, which suggested that this transformation should be a radical pathway and that the carbonyl oxygen atom of **3a** originated from oxygen. This Cu-catalyzed strategy tolerated a broad range of substrates and provided a new route to prepare carbonyl imidazo[1,2-*a*]pyridines, which should be significant for the construction of imidazo[1,2-*a*]pyridine libraries.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b01588.

Experimental procedures and spectral data (PDF)

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Notes

The authors declare no competing financial interest.

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