Oxygenation

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Synthesis of Oxazoles through Copper-Mediated Aerobic Oxidative Dehydrogenative Annulation and Oxygenation of Aldehydes and Amines**

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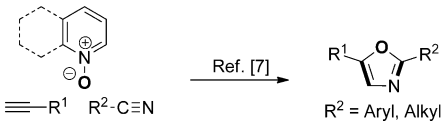
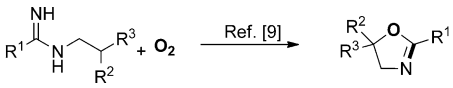
Oxazoles with their 2 and 5 positions substituted with aryl or alkyl groups are regarded as privileged heterocyclic motifs in numerous pharmacologically active synthetic molecules.[1] They are also widespread in bioactive natural products.[2] Subsequently, a lot of new methods have been developed to form oxazoles.[3–6] Among these methods, the intramolecular cyclization of acyclic precursors,[3] the oxidative coupling of amines and prefunctionalized aldehydes or ketones,[4] the oxidation of oxazolines,[5] and other methods were widely used.[6] Practical and efficient approaches to oxazoles from readily available starting material are still desirable.[3–6]

Recently, an elegant work for the formation of oxazoles that are substituted at the 2 and 5 positions was reported by Zhang and co-workers [Eq. (1)].[7] Using the fragment-assembling strategy, they realized the [2+2+1] annulation of



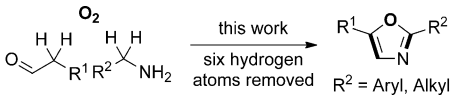
(1)

a terminal alkyne, a nitrile, and a pyridine/quinoline N-oxide as the oxygen source. In light of the increasing demand for environmentally benign organic synthesis and green chemistry, molecular oxygen is probably the ideal terminal oxidant and oxygen atom source for oxygenation, because of its remarkable advantages, such as being inexpensive, having a high atom efficiency, and in most cases with water as the by-product.[8] More recently, Chiba and co-workers[9] developed a significant intramolecular oxygenation approach to 2,5,5-trisubstituted dihydrooxazoles from prefunctionalized N-alkylamidines [Eq. (2)]. Herein, we present an aerobic dehydrogenative annulation and dioxygen activation



(2)

approach to oxazoles from simple and readily available aldehydes, amines, and molecular oxygen [Eq. (3)]. This transformation is highly efficient with the removal of six



(3)

hydrogen atoms, including the cleavage of four C(sp^3)–H bonds.[10] Furthermore, the dehydrogenative coupling strategy[11] and the dioxygen activation of molecular oxygen (1 atm) make this transformation very efficient and practical.

Our studies commenced with the reactions of 2-phenylacetaldehyde (**1a**) and benzylamine (**2a**) in the presence of $CuBr_2$ and K_2CO_3 in toluene at 80 °C under oxygen atmosphere (1 atm). Interestingly, 2,5-diphenyloxazole (**3aa**) was produced in 65% yield (Table 1, entry 1). Other copper salts[12] and bases exhibited lower efficiencies (see Table 1, entries 3 and 4, respectively, and the Supporting Information). Further studies indicated that the presence of a ligand could promote the efficiency of this transformation (Table 1, entries 1 and 5). Among these ligands, pyridine (20 mol%) performed well and significantly increased the yield of **3aa** to 82% (see Table 1, entry 5; for other ligands, see the Supporting Information). Subsequently, the effect of different solvents was surveyed. The reactions in DCE or other solvents resulted in lower yields (see Table 1, entry 8, and the Supporting Information). Significantly, even when this reaction was performed under air, it also worked well and afforded **3aa** in 70% yield (Table 1, entry 9). In contrast, this reaction did not work under argon atmosphere (Table 1, entry 10).

Under these optimized conditions, the scope of substituted amines (**2**) was investigated (Scheme 1). The substituents at the phenyl ring of benzylamines did not affect the efficiency of this transformation, and the desired oxazole products were produced in moderate to good yields. Notably,

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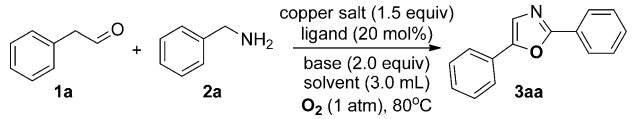
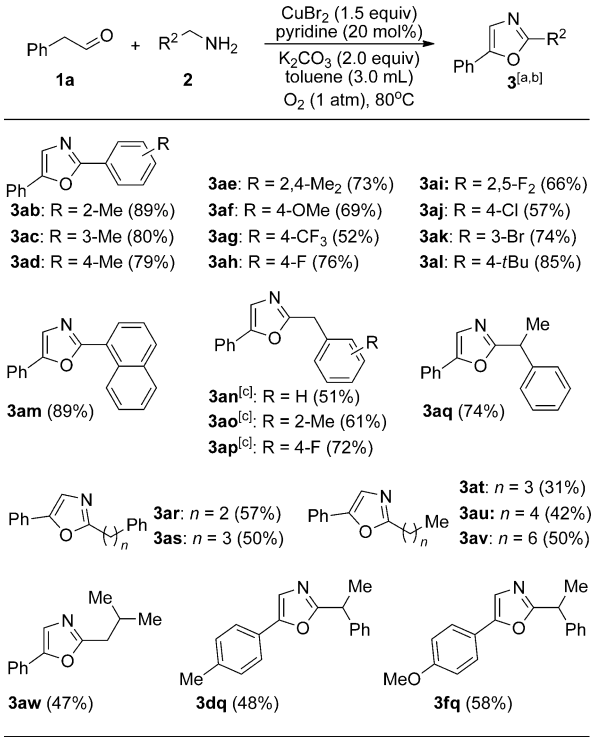
Table 1: The oxidative dehydrogenative annulation and oxygenation of **1a** with **2a**.[a]



Entry	Cu salt	Base	Ligand	Solvent	Yield %[b]
1	$CuBr_2$	K_2CO_3	–	toluene	65
2[c]	$CuBr_2$	K_2CO_3	–	toluene	10
3	$CuCl_2$	K_2CO_3	–	toluene	64
4	$CuBr_2$	Na_2CO_3	–	toluene	21
5	**$CuBr_2$**	**K_2CO_3**	**pyridine**	**toluene**	**82**
6[d]	$CuBr_2$	K_2CO_3	pyridine	toluene	74
7	$CuBr_2$	K_2CO_3	2,2′-bipyridine	toluene	46
8	$CuBr_2$	K_2CO_3	pyridine	DCE	60
9[e]	$CuBr_2$	K_2CO_3	pyridine	toluene	70
10[f]	$CuBr_2$	K_2CO_3	pyridine	toluene	0

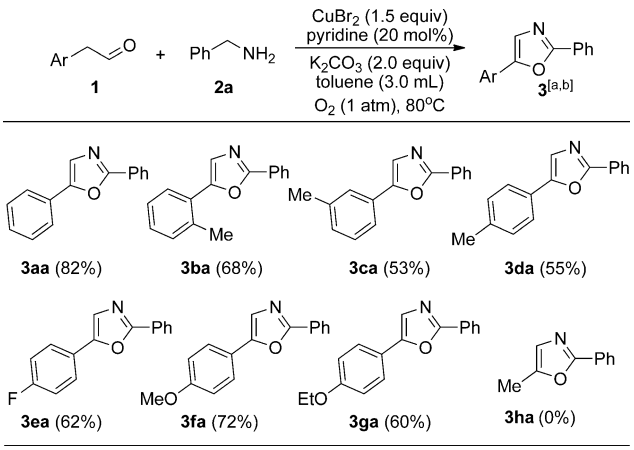
[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), copper salt (0.3 mmol), ligand (0.04 mmol), base (0.4 mmol), solvent (3.0 mL), O_2 (1 atm), 80 °C, 16 h. [b] Yields of isolated products. [c] $CuBr_2$ (0.03 mmol) was used. [d] Pyridine (0.01 mmol) was used. [e] The reaction was carried out under air. [f] The reaction was carried out under argon atmosphere (1 atm). Entry in bold highlights optimized reaction conditions. DCE = dichloroethane.



Scheme 1. Oxidative dehydrogenative annulation and oxygenation of different amines (**2**) with **1a**. [a] Reaction conditions: see Table 1, entry 5. [b] Yields of isolated products. [c] The reaction was stirred for 11 h.

reactions of fluoro-, chloro-, and bromo-substituted benzylamines with **1a** proceeded well and gave the desired oxazole derivative **3ah**, **3ai**, **3aj**, and **3ak** (which could be used for further transformations) in 76%, 66%, 57%, and 74% yield, respectively. The naphthyl-substituted amine was also tolerated in this transformation, generating 2-(naphthalen-1-yl)-5-phenyloxazole **3am** in 89% yield. It is noteworthy that alkyl-substituted amines were also tolerated in this reaction. For example, amines that contained long alkyl chains gave the products in moderate yields (**3ar**–**3aw**). Substituted phenethylamines could be smoothly transformed into the desired products with good yields (**3an**–**3ap**). Furthermore, the steric hindrance did not affect the efficiency (**3aq**, **3dq**, and **3fq**). The present method provides a simple and easily operable protocol for the preparation of 2,5-diaryl- or 2,5-dialkyl-substituted oxazole derivatives from simple and readily available starting materials by employing molecular oxygen as the oxygen source.

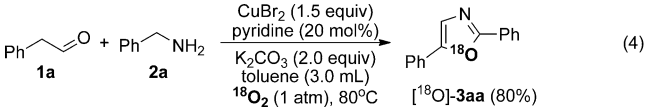
This aerobic oxidative dehydrogenative annulation was further expanded to a range of substituted aldehydes **1** (Scheme 2). The results indicate that reactions of aldehydes with electron-donating groups, such as methoxy, methyl, and ethyloxy, at the aryl ring, proceeded well with moderate to good yields. Moreover, substituents at different positions of



Scheme 2. Oxidative dehydrogenative annulation and oxygenation of different aldehydes (**1**) with **2a**. [a] Reaction conditions: see Table 1, entry 5. [b] Yields of isolated products.

the arene ring (*para, meta*, and *ortho* position) did not affect the efficiency obviously (**3ba**–**3da**). Unfortunately, aliphatic aldehydes did not work under these reaction conditions (**3ha**).

Some control experiments were carried out in order to probe the mechanism of this transformation. The reaction of **1a** and **2a** in the presence of $^{18}O_2$ (1 atm) generated ^{18}O-labeled product [^{18}O]-**3aa** in 80% yield under the standard conditions [Eq. (4), yield determined by HRMS, see the Supporting Information]. In contrast, the similar reaction under $^{16}O_2$ atmosphere but in the presence of $H_2{}^{18}O$ (5.0 equiv) did not afford [^{18}O]-**3aa** [Eq. (5), determined by HRMS, see the Supporting Information]. Both these results



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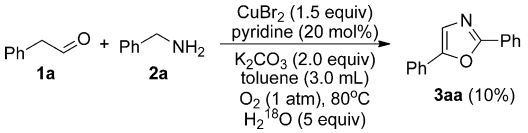
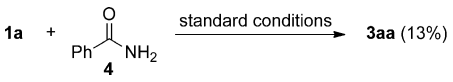


(5)

indicate that the oxygen atom of the oxazole product originated from molecular oxygen. H_2O did not participate in this intermolecular cyclization process.

Furthermore, the reaction of 2-phenylacetaldehyde (**1a**) and benzamide (**4**) under the standard conditions was investigated. Although desired product **3aa** was detected, the yield was only 13% [Eq. (6)], which is much lower than that of the model reaction (82%, see Table 1, entry 5). The



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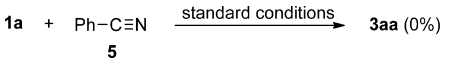
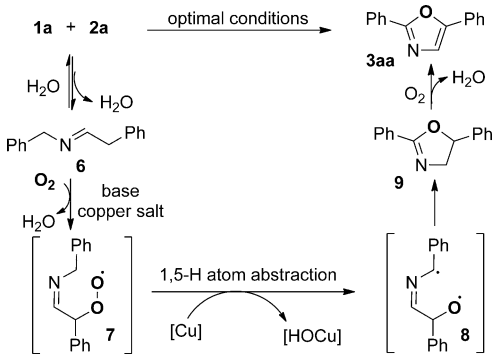
reaction of 2-phenylacetaldehyde (**1a**) and benzonitrile (**5**) under the standard conditions could not generate **3aa** [Eq. (7)], which might exclude **5** as the intermediate of this



(7)

oxidative transformation. These results indicate that benzamide (**4**) may be involved in this process, but they are not the main pathways of this dehydrogenative annulation process.

Although intensive studies of the redox reaction on the copper salt are still ongoing, a possible reaction pathway for the dioxygen activation and oxazole formation is proposed in Scheme 3 on the basis of the above-mentioned results. Aldehydes (**1a**) and anilines (**2a**) initially dehydrate to form imines (**6**).[13] Under basic conditions, imines (**6**) should be oxidized by molecular oxygen to form radical intermediate **7**, a process that should be facilitated by copper salt.[14] Afterward, the 1,5-hydrogen atom abstraction would provide intermediate **8**.[9,15] Subsequent intramolecular radical coupling affords the 4,5-dihydrooxazole intermediate **9**, which



Scheme 3. Possible reaction pathway for dioxygen activation and oxazole formation.

could be easily oxidized to the desired oxazole product **3aa** under oxygen atmosphere.[5f]

In conclusion, we have demonstrated the synthesis of oxazoles through a Cu-mediated aerobic oxidative dehydrogenative annulation of aldehydes, amines, and molecular oxygen by C–H functionalization and dioxygen activation. This transformation is highly efficient with the removal of six hydrogen atoms, including the functionalization of four $C(sp^3)$–H bonds. Furthermore, the dehydrogenative coupling strategy and the dioxygen activation of molecular oxygen (1 atm) make this transformation very practical and atom-economical. Further studies to clearly understand the reaction mechanism and the synthetic applications are ongoing in our laboratory.

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