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Copper mediated C–H amination with oximes: en route to primary anilines†

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Here we report an efficient Cu(I)-mediated C–H amination reaction with oximes as amino donors to introduce NH₂ groups directly. Various strongly coordinating heterocycles including quinoline, pyrimidine, pyrazine, pyrazole and triazole were tolerated well. The potential utility was further demonstrated in a late-stage modification of telmisartan (an antagonist for the angiotensin II receptor).

Introduction

Primary anilines are important structural motifs present in many natural and pharmaceutical compounds (Fig. 1).¹ For example, retigabine is an anticonvulsant used as an adjunctive treatment for partial epilepsies in treatment-experienced adult patients.² Anileridine, as a member of the piperidine class of analgesic agents, is used as an analgesic drug.³ Mesalazine is an aminosalicilate anti-inflammatory drug used for treating inflammatory bowel disease,⁴ and lenalidomide is a tumour necrosis factor (TNF) inhibitor and can be used to treat multiple myeloma.⁵ The development of efficient and practical

approaches to accessing this type of compound is in high demand and remains a challenge in organic synthesis.⁶

Recently, C–H amination *via* a transition-metal catalyzed C–H activation process has attracted considerable attention and has been proven to be a useful method for preparing amine molecules.⁷ Among these C–H amination reactions, only a few examples were able to directly introduce an unprotected amino group to form primary anilines.⁸ These reactions can be classified into two approaches: non-directed and directed (Scheme 1). The former exploits the innate reactivity of the substrates. For example, the non-directed amination of arenes involving photocatalysis was realized by the Tung^{9a} and Nicewicz^{9b} groups independently.

Fig. 1 Selected drug molecules containing primary anilines.

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Scheme 1 The direct synthesis of primary anilines by C–H amination.

Transition metal-catalyzed C–H aminations of arenes were also reported by the two groups of Falck^{9c} and Morandi.^{9d} However, controlling the positional selectivity effectively remains a great challenge in these reactions. In recent years, the use of directing groups has become a reliable approach to realizing selective C–H functionalization.¹⁰ However, few examples of C–H amination for preparing primary anilines are known. The use of chalcogenide amine donors is largely limited to alkyl amines.¹¹ In 2014, the Zhu group reported an example employing TMSN₃ as a nitrogen source to form primary anilines.^{12a} Uchiyama and co-workers developed another amination protocol which used BnONH₂ as aminating agent and involved deprotonative cupration with a stoichiometric amount of (TMP)₂Cu(CN)Li₂.^{12b} More recently, Bolm and coworkers have developed a new reagent, dibenzothiophene sulfoximine, as an NH₃ surrogate to synthesize primary anilines.^{12c} However, site selective C–H amination in late-stage heterocycle drug discovery is always a thorny problem because heterocycles compete with directing groups for coordination to metal catalysts, which may result in the poisoning of the catalyst or the functionalization of an undesired C–H bond. Herein, we report for the first time a facile Cu(I)-mediated C_{sp²}-H amination using oxime derivatives as an aminating agent, in which the free amine products can be easily obtained upon *in situ* hydrolysis. The potential use of this method in late-stage diversification for drug discovery was exemplified by the directed amination of telmisartan.

Oxime derivatives as a class of versatile building block have been applied to a number of transition metal-catalyzed C–H functionalizations.¹³ Due to the N–O σ bond (~ 57 kcal mol^{−1}) being weaker than normal C–X (X = C, N, O) σ bonds, oxime derivatives could not only act as amino sources but also as oxidants. Based on our previous work¹⁴ and oxime chemistry reported by other groups,¹³ we propose a Cu(I) to Cu(III) catalytic cycle (Scheme 1): the first step is the oxidative insertion of the Cu(I) species into the N–O bond to give the Cu(III) intermediate **A**. After coordination of the bidentate directing group oxazoline-amide to the Cu(III) complex **A**, C–H activation occurs to generate the intermediate **B**. Subsequent reductive elimination affords imine compound **C** and regenerates Cu(I). Finally, imine compound **C** can be hydrolyzed to form primary amine **3** and a ketone.

Results and discussion

Based on this hypothesis, we initiated our studies by investigating the C–H amination of *N*-arylbenzamide, substrate **1a**, with oxime **2a**. Encouragingly, we found that C–H amination of **1a-A** with 1.5 equiv. of **2a** occurred in the presence of 20 mol% CuOAc in DMSO at 100 °C over 8 h, and the desired aminated product **3a-A** was obtained in 28% yield after acid hydrolysis. The structure of the product **3a-A** was confirmed by X-ray crystallography (Table 1). Other copper catalysts were also investigated (Table 1, entries 1–4), and CuOAc was still found to be the optimal one. Subsequently, we investigated the influence of the directing groups (DG) (Table 1, entries 5–9) on the efficiency of the amination reaction. We found that the use of substrate **1a-B**, containing the methyl-substituted oxazoline DG **B**, afforded the

Table 1 Optimization of reaction conditions^a

Entry	DG	[Cu] (equiv)	Base	Temp, °C	Yield, % ^b
1	A	CuOAc (0.2)	-	100	28
2	A	CuBr (0.2)	-	100	24
3	A	CuCl (0.2)	-	100	25
4	A	Cu(OAc) ₂ (0.2)	-	100	21
5	B	CuOAc (0.2)	-	100	35
6	C	CuOAc (0.2)	-	100	20
7	D	CuOAc (0.2)	-	100	20
8	E	CuOAc (0.2)	-	100	21
9	F	CuOAc (0.2)	-	100	n.d.
10	B	CuOAc (0.3)	-	100	40
11	B	CuOAc (0.3)	K ₂ HPO ₄	100	49
12	B	CuOAc (1.0)	K ₂ HPO ₄	100	52
13	B	CuOAc (1.5)	K ₂ HPO ₄	100	58
14	B	CuOAc (1.5)	K ₂ HPO ₄	90	78 (73) ^c
15	B	CuOAc (1.5)	K ₂ HPO ₄	80	66
16	B	CuOAc (1.5)	K ₂ HPO ₄	70	59

A: R = H
B: R = Me
C: R = *i*-Pr
D: R = *t*-Bu
E: R = Ph

F

^a Reaction conditions: (1) **1a** (0.1 mmol), **2a** (0.15 mmol), [Cu], base (0.1 mmol), DMSO (1 mL), N₂, 8 h; (2) 2 M HCl (2 mL), rt, 20 min. ^b Yield determined by ¹H NMR analysis of crude reaction product using CH₂Br₂ as internal standard. ^c Isolated yield is given in parentheses.

aminated product **3a-B** in 35% yield. Interestingly, the use of Daugulis's 8-aminoquinoline DG under the same conditions did not provide any desired product (entry 9). This result is consistent with our early observation.¹⁴ The yield was improved to 40% by further increasing the amount of CuOAc to 30 mol%. After screening a series of bases (see ESI[†]), a higher yield of 49% was obtained using 1 equiv. of K₂HPO₄ as a base. Considering that the product binds to the catalyst better than the starting material, increasing the amount of copper may be beneficial to the reaction. Indeed, we found that when 1.5 equiv. of CuOAc was used, the yield could be increased to 58% with full conversion of substrate **1a** (Table 1, entry 13), which implied that the substrate partially decomposed at this temperature. Finally, we screened the temperature (Table 1, entries 14–16), and found that a satisfactory yield of 78% could be obtained at 90 °C (entry 14).

3a-A

After the optimal reaction conditions were found, we proceeded to examine the substrate scope. As shown in Table 2,

Table 2 The scope of C–H amination^{a,b}

^a Reaction conditions: (1) **1a–1t** (0.1 mmol), **2a** (0.15 mmol), CuOAc (0.15 mmol), K₂HPO₄ (0.1 mmol), DMSO (1 mL), N₂, 90 °C, 8 h; (2) 2 M HCl (2 mL), rt. 20 min. ^b Isolated yield. ^c Yields of reactions using 0.3 equiv. of CuOAc are shown in parentheses.

a wide variety of substituted benzamides were reactive. 2-Methyl substituted substrate **1b** gave the aminated product in a moderate yield of 54%. Various electron-donating and -withdrawing substituents including methyl **1c**, *t*-butyl **1d**, methoxyl **1e**, fluoro **1f**, chloro **1g**, bromo **1h**, acetyl **1i**, trifluoromethyl **1j**, phenyl **1k** and vinyl **1l** at position 4 of the benzamides were compatible in the reaction, affording the desired products in moderate to good yields. When position 3 of the benzamides was substituted with methyl, methoxyl or fluoro groups, C–H activation took place at positions 2 and 6 to give two regioisomeric products, with the less hindered position 6-aminated product as the major one. In the case of the benzamide bearing 3-CF₃ (**3p**), only the position 6-aminated product was obtained. Gratifyingly, this copper-mediated C–H amination protocol was compatible with a wide variety of heterocycles, including pyridines, quinoline, and benzothiophene-derived substrates (**3q–t**). Notably, 0.3 equiv. of CuOAc was sufficient to enable the amination reaction (the yields are also shown in parentheses), and the yields were even higher than those obtained in the reactions using 1.5 equiv. of CuOAc in some cases (**3g**, **3h** and **3t**).

Inspired by previous examples overcoming the positional selectivity with substrates containing strongly coordinating heterocycles,¹⁵ we subsequently examined whether the C–H amination system could override the heterocycle-directed

cyclometalation (Table 3). We chose *para*-(2-pyridyl)benzamide as a test substrate. Under the standard conditions, C–H amination proceeded exclusively at the position *ortho* to the amide group, providing the desired product in 71% isolated yield. The structure of **5a** was confirmed using X-ray crystallography. Furthermore, when pyridine was replaced with other medicinally important heterocycles, including quinoline, pyrimidine, pyrazine, pyrazole and triazole, the amide group was still an effective directing group, and the desired aminated products were formed in moderate to good yields (38–72%). Furthermore, reactions using 0.3 equiv. of CuOAc as the catalyst were also examined (the yields are also shown in parentheses).

To probe the reaction mechanism, we measured intra- and intermolecular kinetic isotope effects. Significant isotope effects were observed in both cases (Scheme 2). In addition, the addition of the radical scavenger 2,2,6,6-tetramethylpiperidine-*N*-oxyl (TEMPO) did not affect the reaction efficiency under optimized conditions, which indicates that a radical pathway is unlikely (Scheme 3). These combined data support a mechanism which involves a copper-mediated C–H cleavage step rather than an electrophilic aromatic substitution (SEAr) or radical pathway. Therefore, we propose a Cu(I) to Cu(III) mechanism as shown in Scheme 1.

We also conducted the reaction on a gram scale with 0.3 and 1.5 equiv. of CuOAc and **3a** was obtained in 45% and 62% yield respectively, and most of the benzophenone was recovered (see ESI†). Removal of this amide-oxazoline DG was demonstrated by treating product **3a** with 2 N KOH/EtOH at 80 °C to release the corresponding carboxylic acid and oxazolyamide DG in good yields (Scheme 4). Interestingly, when the aminated products were treated with concentrated HCl at 190 °C, a series of different substituted primary anilines were obtained with complete removal of the amide-oxazoline part (Table 4).¹⁶ In particular, primary anilines containing heterocycles are accessible *via* this approach, making it a good complement to early reported nondirected C–H aminations.⁹

Table 3 Overcoming the limitation of heterocycles in C–H amination^{a,b}

^a Reaction conditions: (1) **4a–4g** (0.1 mmol), **2a** (0.15 mmol), CuOAc (0.15 mmol), K₂HPO₄ (0.1 mmol), DMSO (1 mL), 90 °C, 6 h, N₂. (2) 2 M HCl (2 mL), rt. 20 min. ^b Isolated yield. ^c Yields of reactions using 0.3 equiv. of CuOAc are shown in parentheses.

Scheme 2 (a) Intramolecular kinetic isotope effect (KIE); (b) inter-molecular KIE.

Scheme 3 The effect of TEMPO.

Scheme 4 Removal of the directing group.

Table 4 The transformation of the aminated product^{a,b}

^a Reaction conditions: 3, 5 (0.03 mmol), conc. HCl (0.5 mL), 190 °C, 3 h, air. ^b Isolated yield.

To further demonstrate the viability of the C–H amination reactions for late-stage diversification in drug discovery, we chose telmisartan, an antagonist for the angiotensin II receptor,

Scheme 5 Late-stage diversification of telmisartan.

for functionalization (Scheme 5). As we mentioned earlier,^{15f} there are multiple reactive C–H bonds in this molecule, which significantly enhance the difficulty of selective functionalization of the target C–H bond. By installing the directing group amide oxazoline into the molecule, we were pleased to find that C–H amination occurred exclusively at the amide-directed position, while other C–H functionalization products were not observed. Furthermore, the resulting aminated product was treated under acidic or alkaline conditions to afford two derivatives **8k** and **6b**, respectively.

Conclusions

In summary, we have developed a Cu(I)-mediated C–H amination reaction for the synthesis of primary anilines. A catalytic cycle involving Cu(I) to Cu(III) was proposed, and the oxime derivatives not only act as amino sources but also as oxidants. This practical reaction is compatible with various functional groups as well as diverse strongly coordinating heterocycles. The potential utility of this method was demonstrated by the late-stage diversification of the drug molecule telmisartan.

Conflicts of interest

The authors declare no conflict of interest.

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