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Metal-Free Remote C−H Bond Amidation of 8-Amidoquinolines on the C5 Position under Mild Conditions

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Supporting Information

ABSTRACT: An efficient and facile process was developed for the remote C−H bond amidation of 8-aminoquinoline scaffolds on the C5 position which is geometric. The method only made use of $PhI(OAc)_2$ as a mediator and showed good tolerance toward numerous dibenzenesulfonimides and amides, giving the corresponding products in moderate to excellent yield.

Quinoline is an important scaffold in various natural products and synthetic compounds with wide applications in medicinal and materials chemistry (Figure 1).[1] As a result, the derivatization of quinolines has been an intensive research focus in synthetic chemistry, which has inspired considerable efforts toward the development of highly efficient strategies for the functionalization of these interesting structural motifs.[2]

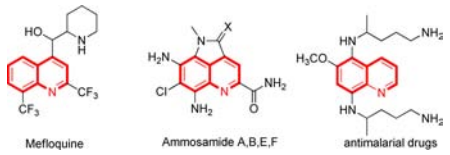
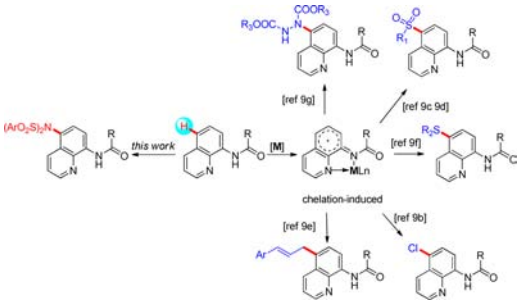


Figure 1. Representative bioactive quinolines.

Although certain functional groups can be introduced into the quinoline scaffold, it often requires harsh conditions or multiple steps in most conventional methods.[3] Recently, significant advances have been made in the direct C−H functionalization of readily available compounds.[4] Especially, transition-metal-catalyzed direct C−H functionalization of the quinoline scaffold has received much attention.[5] Many successful examples in this field typically focusing on the direct functionalization of C−H bonds of quinolines, including at the C2,[6] C4,[7] and C8[8] positions (easily accessible), were reported. Although some approaches for direct construction of C−C bonds at the C5 position of quinolines have been developed,[9] the methodology for the C-5 selective formation of carbon-heteroatom still remains underdeveloped, particularly for C−N bond formation.[9] Thus, it is very necessary to develop a synthetic approach for direct construction of carbon−heteroatom bonds at the C-5 position of quinolines.

Recently, several groups reported remote C−H functionalization of quinolines on the C5 position by metal catalysis (Scheme 1).[9] The first example of quinoline C5-position functionalization

Scheme 1. Direct Modification of C5 Position of Quinolines



was demonstrated by Stahl and co-workers.[9a] Afterward, a series of Cu- or Fe-catalyzed C−H oxidations of 8-aminoquinoline scaffolds have been reported. Zhang and co-workers reported the copper-catalyzed C5-chlorination of quinoline in acetic acid.[9b] Wu[9c] and Liu[9d] developed the copper-catalyzed C5-sulfonylation using arylsulfonyl chlorides, respectively. Zeng and co-workers demonstrated the iron-catalyzed C5-allylation of quinolines with allyl alcohols.[9e] Yin and co-workers developed a C5-chalcogenation using diphenyl disulfide.[9f] Recently, Baidya and co-workers reported a copper-catalyzed C5-amination with azodicarboxylates.[9g] Despite these significant advances, these methods still have some limitations, such as using metal catalysis and high temperature. To the best of our knowledge these are the

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few examples known for the catalytic direct C−H bond functionalization at the C5-position of quinolines. However, there has been no report for the direct C−H bond amidation at the C5-position of quinolines under free-metal catalysis.

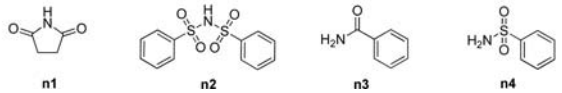
The aryl nitrogen moiety widely exists in pharmaceutically and agrochemically relevant molecules. In recent decades, it has attracted much attention toward constructing C−N bonds of aromatic compounds.[10] As we know, direct oxidative amination reactions[11] of aromatic compounds with metal-free conditions are of high synthetic importance owing to being environmentally benign and practicable. Thus, we became interested in utilizing oxidative amination to realize the C5 position amination of quinoline without metal catalysis. Herein, we report the nonmetal-catalyzed remote C−H amination of quinolines on the C5 position under mild conditions. This remote C−H amination of quinolines can provide a new method for the introduction of sulfonamide into quinoline scaffolds.

Owing to the low toxicity and special reactivity, hypervalent iodine compounds as organic oxidant reagents have been widely used in oxidation reactions.[12] Thus, we commenced our investigation with 8-acylaminoquinoline **1** and phenyliodine(III) diacetate ($PhI(OAc)_2$) as a model substrate (Table 1). At the beginning of our investigation, a nitrogen source was screened. Four different nitrogen sources n1−n4 were used in the reaction of 8-acylaminoquinoline with 2 equiv of $PhI(OAc)_2$ at 100 °C in xylene. However, it was found that n1, n3, and n4 failed to undergo the amination of the C5 position and 8-acylaminoquinoline **1** was recovered after 24 h (entries 1, 3, 4). To our delight, when n2 was used as a nitrogen source the C5 position amination product yield was obtained in 40% and no other position amination product was observed in the reaction (entry 2). This result indicates that the strong electron-withdrawing disulfonyl group facilitates this transformation. Encouraged by this result, we then explored whether other hypervalent iodine compounds can fit this transformation. Unfortunately, no desired product was obtained when $PhI(OAc)_2$ was replaced by Dess-Martin periodinane (entry 5). This result suggests that the hypervalent iodine(III) species may have a key role in this reaction. To verify the essential role of $PhI(OAc)_2$ in the oxidative amination reaction, we then tested a series of oxidants. Some peroxides such as *t*-BuOOH, *m*-CPBA, and H_2O_2 failed to obtain any desired product using the same reaction conditions as those for entry 2 (entries 6−8). Other oxidants often used in $Pd(OAc)_2$-catalyzed C−H activation,[13] such as Oxone, $K_2S_2O_8$, and Ag_2O, also did not provide product **2** (entries 9−11). Using O_2 as the oxidant only could not realize the transformation (entry 12). After confirming the optimal oxidant, several solvents such as MeCN, Dioxane, THF, DMF, 1, 2- dichloroethane, and DME (glycol dimethyl ether) were applied to this reaction (entries 13−18). It was found that using DME as solvent could significantly improve the yield from 40% to 66% (entry 18). Finally, the temperature is also crucial to this reaction. We found that the yield could be improved at lower reaction temperature. The best yield (83%) was obtained at room temperature (entries 19−21).

With the optimized conditions in hand, the scope of the reaction was explored (Scheme 2). The reaction was quite general for a series of diversely substituted 8-aminoquinolines in the presence of 2.0 equiv of $PhI(OAc)_2$ and 1.5 equiv of dibenzenesulfonimide. The carboxamides with alkyl substitutions furnished C5-amino-substituted quinolines **2**, **4**, and **5** in high yields (79−83%). The carboxamide derived from the

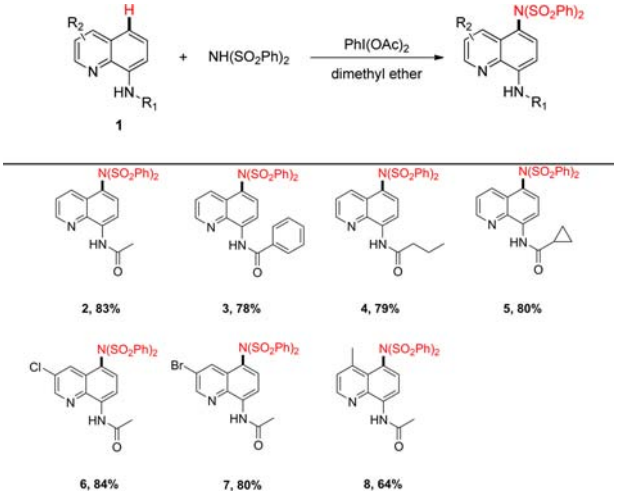
Table 1. Optimization of the Reaction Conditions[a]

entry	oxidant[b]	nitrogen source[c]	solvent	t[°C]	yield[%][d]
1	$PhI(OAc)_2$	n1	xylene	100	n.d.
2	$PhI(OAc)_2$	n2	xylene	100	40%
3	$PhI(OAc)_2$	n3	xylene	100	n.d.
4	$PhI(OAc)_2$	n4	xylene	100	n.d.
5	DMP	n2	xylene	100	n.d.
6	t-BuOOH	n2	xylene	100	n.d.
7	m-CPBA	n2	xylene	100	n.d.
8	H_2O_2	n2	xylene	100	n.d.
9	$K_2S_2O_8$	n2	xylene	100	n.d.
10	oxone	n2	xylene	100	n.d.
11	Ag_2O	n2	xylene	100	n.d.
12	O_2	n2	xylene	100	n.d.
13	$PhI(OAc)_2$	n2	MeCN	100	37%
14	$PhI(OAc)_2$	n2	dioxane	100	56%
15	$PhI(OAc)_2$	n2	THF	100	53%
16	$PhI(OAc)_2$	n2	DMF	100	32%
17	$PhI(OAc)_2$	n2	DCE	100	55%
18	$PhI(OAc)_2$	n2	DME	100	66%
19	$PhI(OAc)_2$	n2	DME	80	68%
20	$PhI(OAc)_2$	n2	DME	50	70%
21	$PhI(OAc)_2$	n2	DME	25	83%



[a]Standard reaction conditions: **1** (0.2 mmol), solvent (1.5 mL), 8 h. [b]Used 2.0 equiv of each oxidant. [c]Used 1.5 equiv of each nitrogen source. [d]Isolated yield after column chromatography. DCE = dichloroethane, DME = glycol dimethyl ether.

Scheme 2. Investigation of Quinoline Scaffolds[a,b]



[a]Experiments were performed with **1** (0.2 mmol), $HN(SO_2Ph)_2$ (0.3 mmol), $PhI(OAc)_2$ (0.4 mmol), in dimethyl ether (1.5 mL) for 8 h at 25 °C. [b]Isolated yields reported.

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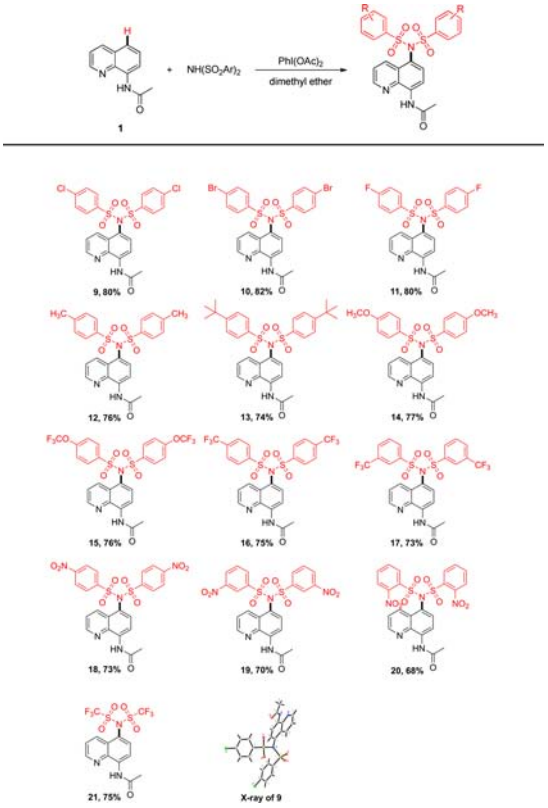
aromatic compound was also a suitable substrate for this reaction, giving product **3** in a 78% yield. Furthermore, the substitution on the quinoline moiety was explored. Both 3-Cl and 3-Br substituted quinoline derivatives **6**, **7** could realize the amination on the C5 position, obtaining yields of 84% and 80%, respectively. However, the amination of 4-CH_3 substitute quinoline derivative **8** was only obtained in a relatively low yield (64%), which could be due to the steric hindrance of 4-CH_3.

The reaction was not restricted only to phenyl-substituted sulfonamide; it worked equally well with other substituted sulfonamides. As demonstrated in Scheme 3, 4-Cl, 4-Br, and 4-F

Scheme 3. Investigation of Dibenzenesulfonimides[a,b]

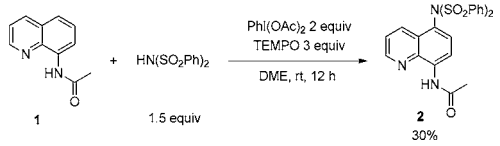


[a]Experiments were performed with **1** (0.2 mmol), $HN(SO_2Ar_2)_2$ (0.3 mmol), $PhI(OAc)_2$ (0.4 mmol), in dimethyl ether (1.5 mL) for 8 h at 25 °C. [b]Isolated yields reported.

substitute dibenzenesulfonimides could react smoothly with **1**, giving 80%−82% yields (**9**−**11**). The structure of compound **9** was confirmed by single-crystal X-ray analysis (see Supporting Information for details). 4-Methyl and 4-tertiary butyl derivatives were also obtained in a relatively low yield (**12**, **13**). Alkoxy substituted dibenzenesulfonimides, such as 4-methoxyl and 4-trifluoromethoxy, gave similar yields (**14**, **15**). The electron-withdrawing substituent derivatives including 4-CF_3, 3-CF_3, 4-NO_2, 3-NO_2, and 2-NO_2 also rendered a good yield under these conditions (**16**−**20**). Encouraged by these outcomes, trifluoromethanesulfonimide was chosen as a nitrogen source and as expected the reaction could also take place under the optimized conditions (**21**).

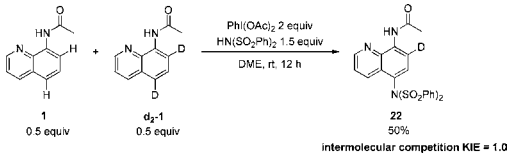
In order to explore the reaction mechanism, 2, 2, 6, 6-tetramethylpiperidine *N*-oxide (TEMPO) was added in the reaction as the radical quencher to investigate whether these reactions proceeded via radical pathways (Scheme 4). With 3

Scheme 4. Radical Inhibition Experiments



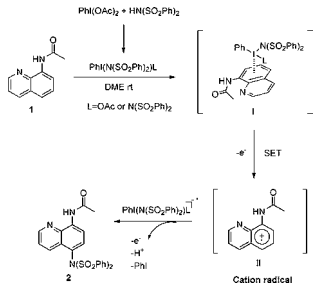
equiv of TEMPO, the yield of the desired product **2** decreased from 83% to 30%. This result suggested that the reaction followed a radical pathway. Furthermore, no kinetic isotope effect (KIE) was observed in an intermolecular competition experiment between amide **1** and the dideuterated substrate d_2-**1** (Scheme 5). This result indicates that the cleavage of the C−H

Scheme 5. Kinetic Isotope Experiment



bond is not the rate-limiting step. Though a full mechanistic understanding of this reaction will require further experimentation, based on the radical inhibition experiments and related results reported by others,[14] a plausible mechanism is illustrated in Scheme 6. The mechanism of this reaction includes a cation

Scheme 6. Proposed Mechanism

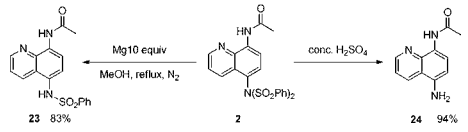


radical intermediate involving SET by the action of $PhI(OAc)_2$. Initially, $PhI(OAc)_2$ reacts with $HN(SO_2Ph)_2$ to generate hypervalent iodine(III) species having $N(SO_2Ph)_2$ ligands by a ligand exchange reaction at the iodine(III) center between OAc group in $PhI(OAc)_2$ and $HN(SO_2Ph)_2$. The activated iodine(III) species induces SET oxidation **1** to produce their cation radical **II** via a **I** complex. The C5 amination transfer toward **II** followed by oxidation and deprotonation gave the amination products along with an iodobenzene coproduct.

Finally, the sulfonamide could be selectively cleaved by different protocols (Scheme 7). Treatment of **2** with magnesium in methanol under reflux produced **23** in 83% yield. The amine **24** could be obtained from **2** in 94% yield with conc. H_2SO_4.

In conclusion, we have developed a novel nonmetal-catalyzed remote C−H amidation of quinolines on the C5 position. This

Scheme 7. Desulfonylation of Sulfonamide



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protocol, which is operationally simple and nonmetal catalyzed, displays a broad substrate scope and uses commercially available dibenzenesulfonimide as the aminating agent under mild conditions. Additional studies investigating the scope for the C−H functionalization with other heteroatoms and their applications in total synthesis are ongoing. Considering the various bioactivities of quinoline scaffolds, further studies to evaluate the antitumor activities of these new compounds are currently in progress.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.6b01980.

Experimental procedures and spectroscopic data for all new compounds (PDF)

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Author Contributions

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Notes

The authors declare no competing financial interest.

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