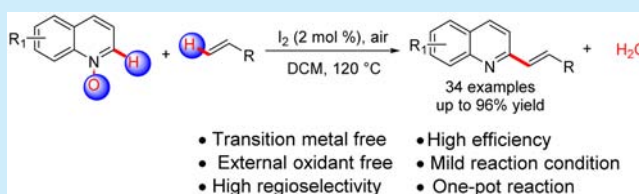


Iodine-Catalyzed Direct C–H Alkenylation of Azaheterocycle N-Oxides with Alkenes

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Supporting Information

ABSTRACT: An efficient and regioselective alkenylation of azaheterocycle N-oxides with alkenes catalyzed by iodine under metal- and external oxidant-free reaction conditions has been developed. A variety of (*E*)-2-styrylazaheterocycles have been produced in moderate to excellent yields. The mechanistic exploration indicated that the N-oxide group played dual roles as both the directing group and an internal oxidant in this catalytic cycle.



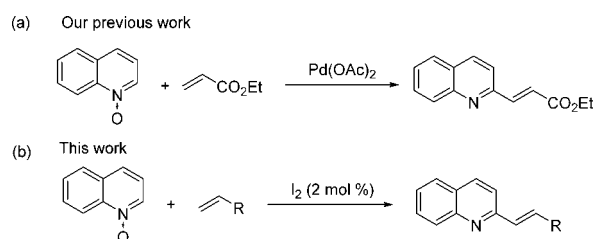
Direct C–H bond activation has attracted great interest in organic synthesis as the most efficient and straightforward method to build C–C and C–heteroatom bonds.¹ The high bond energy, weak reactivity, and poor regioselectivity of the C–H bond have led to very harsh reaction conditions. Transition metal-catalyzed C–H functionalization through cyclometalation has become an ideal strategy to overcome these substantial challenges.² For instance, the transition-metal catalyzed oxidative Heck reaction has been widely used to create functionally diverse and structurally complicated compounds.³ Generally, an external oxidant is required to regenerate the active catalytic species, which would provide a stoichiometric amount of the reduced external oxidant as waste and reduce the overall “greenness” of the process. Our group has previously developed a novel alkenylation of quinoline N-oxides catalyzed by palladium in which the N-oxides served as an oxidizing directing group (ODG) and additional oxidants were avoided (Scheme 1a).⁴ The clear advantages of ODGs, which serve as both directing group and oxidant, thereby simplifying reaction systems and making reactions more concise and efficient, have attracted interest in C–H activation. Further progress has been achieved by the Yu,⁵ Ackermann,⁶ Hartwig,⁷ Li,⁸ Shi,⁹ Glorius,¹⁰ and other groups.¹¹ However, the use of transition metals may cause potential

contamination of the products, which is particularly significant in the pharmaceutical industry¹² and advanced functional materials.¹³ Therefore, C–H activation reactions under metal-free conditions are highly desirable.¹⁴

Iodine has concurrently been developed as a newly emerging nonmetal catalyst due to its low toxicity, convenient after-treatment, and ease of removal, and it has emerged as one of the most active research topics in organic synthesis.¹⁵ Our interest in clean cross-dehydrogenative coupling reactions under external oxidant-free conditions spurred us to test the feasibility of iodine-catalyzed oxidative alkenylation.^{14d,16} Herein, we embarked on the development of a highly efficient and selective iodine-catalyzed direct alkenylation of azaheterocycle N-oxides with alkenes as part of our continuing work. In this transformation, the substrate scope was successfully extended to low activity alkenes, and the N–O group served as an oxidizing directing group and avoided the use of a metal catalyst (Scheme 1b).

The condensation of quinoline N-oxide **1a** and styrene **2a** was chosen as an initial model reaction to screen various reaction parameters (Table 1). In an initial attempt, **1a** was treated with **2a** in the presence of I₂ (10 mol %) in toluene at 120 °C under air for 22 h. As a result, the desired dehydrogenative coupling product **3aa** was formed in 57% yield (Table 1, entry 1). The molecular structure of product **3aa** was confirmed by single crystal X-ray diffraction analysis (SI, Figure S1) and NMR spectra. Next, various solvents were screened (entries 2–6). Among them, DCM gave the best results to afford **3aa** in 83% yield. Decreasing the reaction time to 5 h gave the same result (entry 7). Pleasingly, 2 mol % I₂ was sufficient for this transformation, affording the coupling product **3aa** in 83% yield (entries 8–11). No target product was observed in the absence of iodine (entry 12) and in the presence of KI instead of I₂.

Scheme 1. Oxidative Alkenylation Reaction under External Oxidant-Free Conditions



Received: November 21, 2016

Published: January 23, 2017

Table 1. Optimization of the Reaction Conditions for Quinoline N-Oxide 1a with Styrene 2a^a

entry	I ₂ (mol %)	solvent	time (h)	yield (%) ^b
1	10	toluene	22	57
2	10	1,4-dioxane	22	71
3	10	MeCN	22	82
4	10	DCE	22	62
5	10	DCM	22	83
6	10		22	75
7	10	DCM	5	83
8	15	DCM	5	82
9	5	DCM	5	85
10	2	DCM	5	83
11	1	DCM	5	43
12	0	DCM	5	n.d. ^c
13	2	DCM	24	58 ^d
14	2		24	69 ^e
15	2	DCM	24	67 ^e
16	2	DCM	24	58 ^f
17	2	DCM	5	66 ^g
18	2	DCM	5	35 ^h

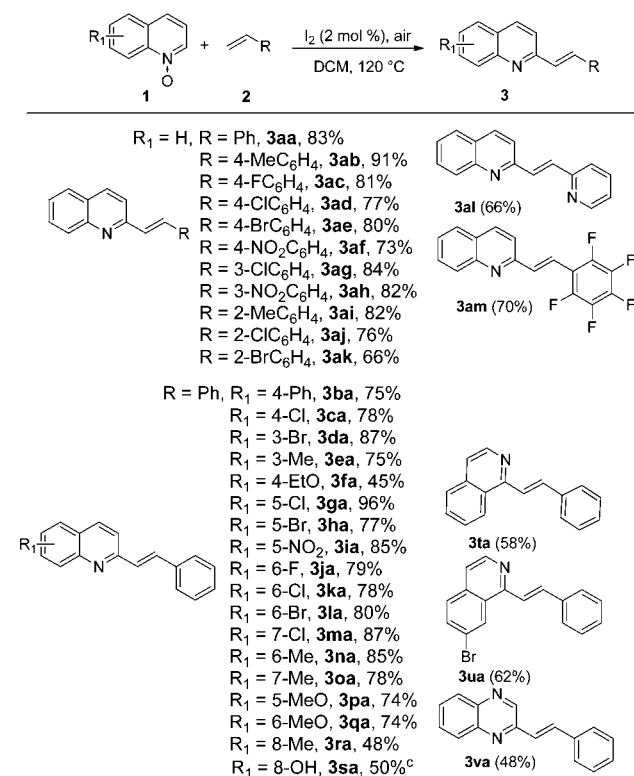
^aReaction conditions: **1a** (0.2 mmol), **2a** (2.0 mmol), solvent (0.5 mL), under air, 120 °C. ^bIsolated yield. ^cn.d. = not detected.

^dSealed container. ^eN₂. ^fO₂. ^g**2a** (1.0 mmol). ^h**2a** (0.4 mmol).

The transformation was incomplete after 24 h in a sealed container in air as well as under N₂ and O₂ atmospheres (entries 13–16). Decreasing the loading of **2a** to 1.0 or 0.4 mmol led to lower yield of 66% and 35%, respectively (entries 17 and 18).

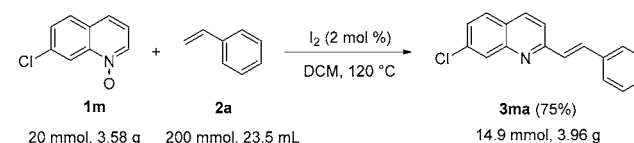
With the optimized conditions set, the substrate scope of this transformation was investigated (Scheme 2). A range of alkenes were tested with quinoline N-oxide (**1a**). Styrenes with electron-donating and electron-withdrawing substituents (such as Me, F, Cl, Br, and NO₂) at the para-position of the benzene ring gave the desired products in excellent yields (**3aa–af**). Generally, styrenes with electron-donating groups gave higher yields than those with electron-withdrawing groups (**3aa–ab** vs **3ac–af**). The efficiency of the alkenylation was not significantly affected by steric hindrance (**3aa–ah** vs **3ai–ak**). For instance, 4-chlorostyrene (**2d**) gave a similar yield to 2-chlorostyrene (**2j**). Furthermore, 2-vinylpyridine and 2,3,4,5,6-pentafluorostyrene could give the corresponding C2-alkenylated products in 66% and 70%, respectively (**3al** and **3am**). However, aliphatic alkene is not suitable for this reaction system. For example, no desired product was observed when vinylcyclohexane was used as a reactant under the standard reaction conditions.

We next investigated a range of heteroaromatic N-oxides to react with styrene (**2a**) under the optimized conditions (Scheme 2). Electronically and sterically diverse quinoline N-oxides all participated smoothly to provide the targets in good to excellent yields (**3ba–sa**). A strong electron-withdrawing group such as 5-nitroquinoline (**2i**) generated the product **3ia** in 85% yield. C8-substituted quinoline N-oxides (**2r** and **2s**) provided the desired products in 48% (**3ra**) and 50% (**3sa**) yields. Isoquinoline N-oxide (**2t**), 6-bromoisoquinoline N-oxide (**2u**), and quinoxaline N-oxide (**2v**) are also suitable for this catalytic system and generated the desired products in 58%, 62%, and 48% yields (**3ta**, **3ua**, and **3va**). Pyridine oxides instead of quinoline-oxide could not give the desired product.

Scheme 2. Substrate Scope of Heteroaromatic N-Oxides with Alkenes^{a,b}

^aReaction conditions: **1** (0.2 mmol), **2** (2.0 mmol), DCM (0.5 mL), 120 °C, 5 h, air. ^bIsolated yield. ^cTwenty-four hours.

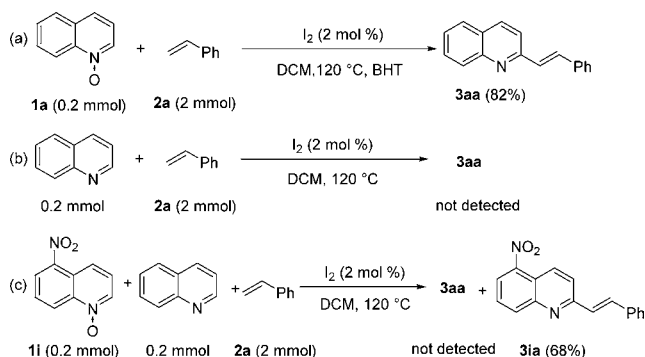
Compounds containing the 7-chloro-2-styrylquinoline moiety are always present in leukotriene receptor antagonists.¹⁷ To demonstrate the synthetic utility of this alkenylation reaction, a gram-scale reaction of 7-chloroquinoline N-oxide (**1m**) and styrene (**2a**) was carried out. As shown in Scheme 3, (E)-7-chloro-2-styrylquinoline (**3ma**) was obtained in 75% yield.

Scheme 3. Gram-Scale Reaction of **1m** with **2a**

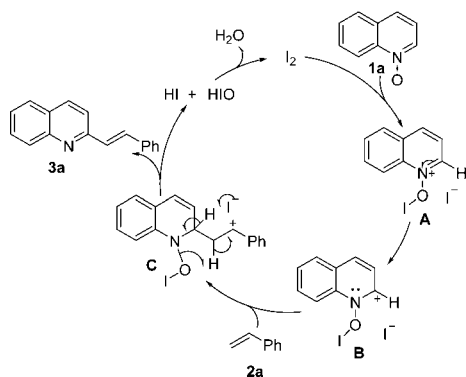
To clarify the reaction mechanism, control experiments were performed (Scheme 4). Addition of the radical scavenger BHT (2,6-di-*tert*-butyl-4-methylphenol) to the reaction mixture afforded the target product **3aa** in 82% yield (Scheme 4a), suggesting the impossibility of a radical process. When quinoline was used instead of quinoline N-oxide as the reactant under the standard reaction conditions, no desired **3aa** was found, which indicated that the N–O group played an important role in this transformation (Scheme 4b). Moreover, when a mixture of 5-nitroquinoline 1-oxide **1i** and quinoline (**1**:**1**) were added to the reaction system, only **3aa** was detected (Scheme 4c), further indicating the crucial role of N-oxide.

Based on the obtained results and the literature,¹⁸ a plausible reaction mechanism is proposed (Scheme 5). Initially, the oxygen atom in quinoline N-oxide (**1a**) was attacked by the iodine, leading to the formation of quaternary ammonium salt intermediate **A**.

Scheme 4. Control Experiments



Scheme 5. Plausible Reaction Mechanism



Subsequently, the complex **A** was converted to intermediate **B** through intramolecular rearrangement. Next, the intermediate **B** could convert into intermediate **C** through nucleophilic addition of styrene (**2a**) at C2 of quinoline. Finally, the rearrangement of intermediate **C** gave the product **3aa**, HOI, and HI. Meanwhile, the generated HI was oxidized by HOI to produce iodine for the next catalytic cycle.

In summary, a mild, simple, and efficient protocol for the iodine-catalyzed C2-alkenylation of azaheterocycle N-oxides with alkenes under air atmosphere and metal-free conditions has been developed. In this approach, a broad range of (*E*)-2-styrylazaheterocycles is conveniently obtained in moderate to excellent yield. This protocol is a convergent one-pot cascade sequence, and the N-oxides served as both substrates and internal oxidants. Further exploration of the synthetic potential of the N-oxide-mediated iodine-catalyzed direct C–H bond activation is underway in our laboratory.

■ ASSOCIATED CONTENT

S Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: [10.1021/acs.orglett.6b03399](https://doi.org/10.1021/acs.orglett.6b03399).

General experimental procedure and characterization data of the products (PDF)
CIF data of **3aa** (CIF)

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

We greatly acknowledge partial financial support from the NSF of China (21572072), the Science and Technology Innovation Program of the Universities of Henan Province (16HAS-TIT007), and Zhengzhou University

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