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Visible-Light-Mediated Aerobic Oxidation of Organoboron Compounds Using in Situ Generated Hydrogen Peroxide

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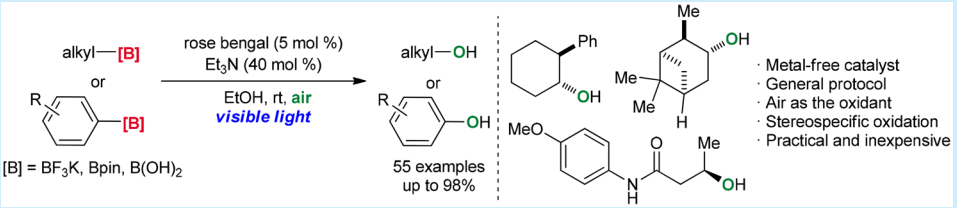
Supporting Information



ABSTRACT: A simple and general visible-light-mediated oxidation of organoboron compounds has been developed with rose bengal as the photocatalyst, substoichiometric Et_3N as the electron donor, as well as air as the oxidant. This mild and metal-free protocol shows a broad substrate scope and provides a wide range of aliphatic alcohols and phenols in moderate to excellent yields. Notably, the robustness of this method is demonstrated on the stereospecific aerobic oxidation of organoboron compounds.

Aliphatic alcohols and phenols are highly important and valuable compounds, which are widely present in the structures of numerous natural products and pharmaceuticals and are employed as versatile intermediates in organic synthesis.[1] Accordingly, there is continuing interest in the development of synthetic methods for their preparation. Among various methods known,[2] the oxidative hydroxylation of boronic acids has provided a particularly effective avenue to access aliphatic alcohols and phenols. Various reaction conditions for the oxidative hydroxylation of boronic acids have been established in the past decades. Most of these methods rely on the use of stoichiometric oxidants such as hydrogen peroxide,[3] TBHP,[4] oxone,[5] benzoquinone,[6] *m*CPBA,[7] hypervalent iodine,[8] amine oxide,[9] and $NaBO_3$.[10] Compared to these stoichiometric oxidants, molecular oxygen (O_2 and air) is a more appealing oxidant because of its abundance and its environmentally friendly and inexpensive character.[11] Based on this concept, several reaction systems for aerobic oxidation hydroxylation of boronic acids have been developed.[12] However, these protocols involve the use of transition-metal catalysts, the need for a specialized reaction setup, or the use of stoichiometric reductants. Moreover, their applicability is limited to only boronic acids.

In recent years, photoredox catalysis has been introduced to aerobic oxidative hydroxylation of aryl boronic acids, which has provided a simple and sustainable synthetic route to phenols.[13] Pioneering work by Xiao and Jørgensen demonstrated a visible-light-mediated aerobic oxidative hydroxylation of aryl boronic acids using $Ru(bpy)_3Cl_2$ as a catalyst.[14] Later, various photocatalysts including metal organic framework (MOF),[15] porous organic framework (POF),[16] cationic polycarbazole network (CPOP),[17] quantum dots (QDs),[18] and organic photocatalysts[19] have been successfully applied to this transformation. Despite the high efficiency, these protocols are associated with one or more limitations such as the use of expensive metal catalysts, tedious syntheses of the catalysts, high cost, prolonged reaction time, the need for pure O_2, and so on. More importantly, in these protocols, the scope of organoboron compounds is mostly limited to arylboronic acids. Furthermore, other synthetic challenges in photomediated oxidative hydroxylation also include achieving oxidative hydroxylation of tetravalent organoboron compounds (e.g., alkyl- or aryltrifluoroborates), which, however, has been unexplored thus far.

Considering the above limitations and challenges, we decided to develop a simple, general, and practical photocatalytic method to realize oxidative hydroxylation of a broad range of organoboron compounds. We believe the key to achieve our goal lies in an appropriate choice of photocatalytic system. We also noticed that all the aforementioned visible-light-mediated oxidative hydroxylation reactions consistently employ a stoichiometric amine (2−5 equiv) as a sacrificial electron donor to complete the photoredox catalytic cycle.[14−19] In this process, the O_2 molecule can be activated by receiving an electron to transform it to superoxide anion radicals, which then reacted with arylboronic acids to afford the phenols. In fact, besides the superoxide anion radical, hydrogen peroxide is also one of the important reactive oxygen species in many photomediated aerobic oxidation reactions.[20] Based on this, we reasoned a different reaction pathway where in situ generated hydrogen peroxide might act as a highly efficient and powerful oxidant to oxidize various organoboron compounds directly. However, hydrogen peroxide generated

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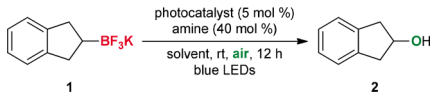
under photocatalysis is generally considered to be a useless byproduct and thus has not been further applied in organic synthesis. This is partly due to the fact that the generation of hydrogen peroxide is inefficient under the current photocatalytic systems. In this contribution, we report a general and practical visible-light-mediated aerobic oxidative hydroxylation of organoboron compounds using in situ generated hydrogen peroxide. The significance of this work presented here is 3-fold: (1) We discover a simple and highly efficient metal-free photocatalytic system to generate hydrogen peroxide. This photocatalytic system employs commercially available and inexpensive organic dye, rose bengal, as the photocatalyst, substoichiometric Et_3N as the electron donor, and air as the oxidant. (2) The present protocol exhibits a broad substrate scope and high functional-group tolerance, which enables oxidative hydroxylation of a wide range of organoboron compounds. (3) These reactions are operationally simple and amenable to large-scale synthesis. In contrast to methods using hydrogen peroxide as oxidant, this novel process avoids potential safety hazards from the transport, storage, and handling of bulk hydrogen peroxide.

Our studies started with the aerobic oxidative hydroxylation of alkyltrifluoroborate **1** (Table 1). We found that 5 mol % rose bengal along with 2.0 equiv of Et_3N catalyze the oxidative hydroxylation of **1** in the presence of air as the oxidant in EtOH under 12 W blue LED irradiation to give the alcohol **2** in 93% yield (entry 1). We believe that catalytic amounts of Et_3N might be sufficient for this transformation since tertiary amines underwent three successive photodegradation processes under photoredox catalysis to generate enough hydrogen peroxide. To test this hypothesis, we performed the reaction using 40 mol % of Et_3N, and **2** was smoothly obtained in 95% yield (entry 2). Other common oganic photocatalysts were then evaluated, and these results show that rose bengal performed best (entries 3–5). Switching the solvent from EtOH to aprotic solvents such as DMF, CH_3CN, or DCE has a negative effect on the yield of **2** (entries 6–8). The influence of various amines on this reaction was also investigated, and we found that iPr_2NEt is also an efficient electron donor, but DABCO and DBU resulted in low yields (entries 10 and 11). Control experiments were performed and confirmed that the photocatalyst, visible light, and amine were all essential for product formation (entries 12–14).

With the optimized conditions in hand, the scope of this transformation was examined (Scheme 1). In general, this protocol has a very broad scope. A range of primary alkyltrifluoroborates possessing different chain lengths underwent this oxidative hydroxylation smoothly to produce the corresponding alcohols in 78–98% yields (**3**–**21**). Moreover, various secondary and tertiary alkyltrifluoroborates reacted with high efficiency, affording products **22**–**28** in good to excellent yields. Notably, this reaction showed excellent functional-group compatibility: ether (**13**, **14**), ester (**15**, **16**, **19**), amide (**17**), ketone (**25**, **28**), alkyne (**19**), alkene (**20**, **21**), and even heterocycles (**15**, **16**, **18**, **26**) were well-tolerated. To show the practical usefulness of this method, we

Table 1. Optimization of Reaction Conditions[a]

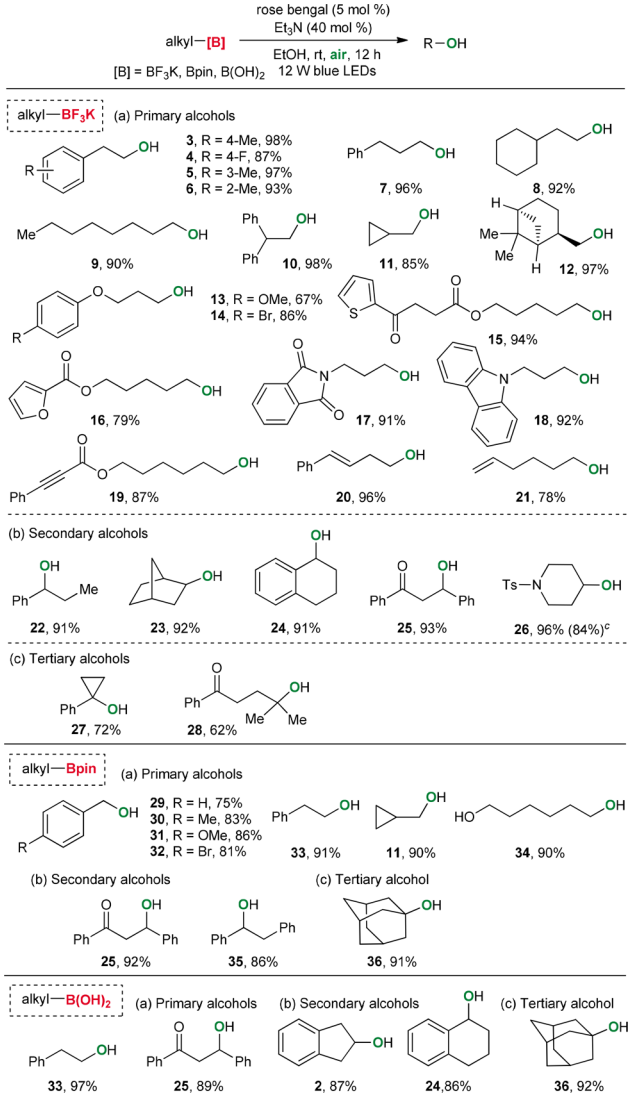


1 2

entry	photocatalyst	amine	solvent	yield[b] (%)
1[c]	rose bengal	NEt_3	EtOH	93
2	**rose bengal**	**NEt_3**	**EtOH**	**95**
3	fluorescein	NEt_3	EtOH	80
4	rhodamine B	NEt_3	EtOH	60
5	eosin Y	NEt_3	EtOH	81
6	rose bengal	NEt_3	DMF	trace
7	rose bengal	NEt_3	CH_3CN	45
8	rose bengal	NEt_3	DCE	42
9	rose bengal	iPr_2NEt	EtOH	92
10	rose bengal	DABCO	EtOH	27
11	rose bengal	DBU	EtOH	60
12	none	NEt_3	EtOH	0
13[d]	rose bengal	NEt_3	EtOH	0
14	rose bengal	none	EtOH	0

[a]Reaction conditions: **1** (0.5 mmol), photocatalyst (5 mol %), and amine (40 mol %) in solvent (3.0 mL) were irradiated with 12 W blue LEDs at room temperature under air for 12 h. [b]Isolated yields. [c]Using NEt_3 (2.0 equiv). [d]In the dark.

Scheme 1. Scope of Alkylboron Compounds[a,b]



[a]Reaction conditions: see entry 2, Table 1. [b]Isolated yields. [c]0.86 g of **26** prepared.

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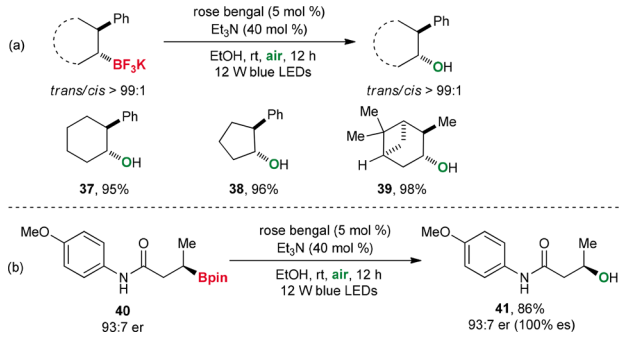
carried out a reaction on a 4.0 mmol scale and isolated **26** in 84% yield (0.86 g).

Encouraged by these results, we next explored the scope of this reaction with respect to alkylboronic pinacol esters. As shown in Scheme 1, a series of primary, secondary, and tertiary alkylboronic pinacol esters were smoothly converted into the desired products in good to excellent yields (75−92%). When various alkylboronic acids were employed as the substrates, alcohols were obtained in high yields (86−97%). Notably, aliphatic alcohols, especially benzylic alcohols and secondary aliphatic alcohols, are generally susceptible to oxidation conditions, which could be readily transformed into the corresponding aldehydes or ketones.[21] Advantageously, these alcohols are able to survive under our photocatalytic oxidation conditions.

We then tested the stereoselectivity of this oxidative hydroxylation reaction. Pleasingly, the reactions conducted with *trans*-alkyltrifluoroborate substrates proceeded with complete stereoselectivity to produce the corresponding products **37**−**39** in excellent yields (Scheme 2a). Assignment

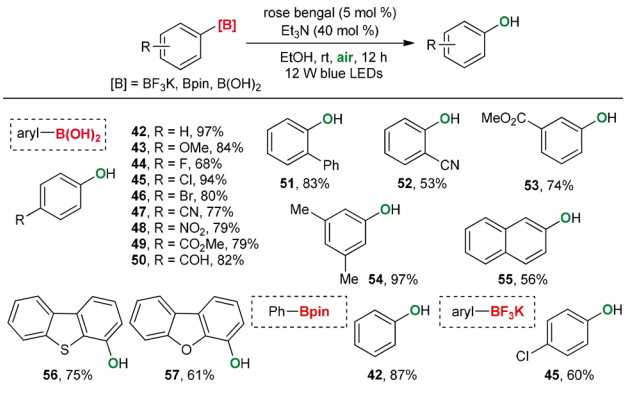
Scheme 2. Stereospecific Aerobic Oxidative Hydroxylation



of the relative configuration was based on ROESY analysis of crude products. To evaluate the stereochemical course of this reaction further, we prepared enantioenriched **40**.[22] It is found that **40** reacted with complete stereochemical fidelity to give the alcohol **41** in 94% yield (Scheme 2b).

After successfully investigating various alkylboron compounds, we turned our attention to evaluating arylboron compounds. As shown in Scheme 3, various aryl boronic acids bearing electron-donating and electron-withdrawing substituents were readily oxidized in an efficient manner (**42**−**54**). A

Scheme 3. Scope of Arylboron Compounds[a,b]

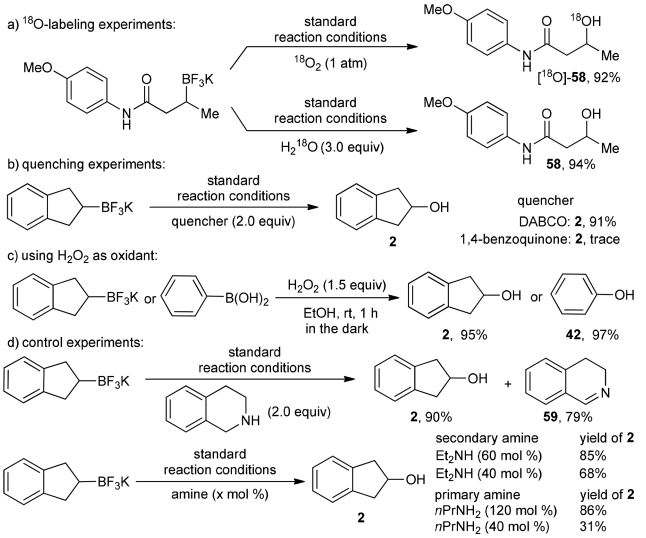


[a]Reaction conditions: see entry 2, Table 1. [b]Isolated yields.

naphthol can be obtained in moderate yield (**55**). Moreover, heteroaryl substrates were also competent substrates, and phenols **56** and **57** were obtained in good yields. Finally, both phenylboronic acid pinacol ester and aryltrifluoroborate were successfully employed and delivered the corresponding phenols **42** and **45** in good yields.

Mechanistic studies were performed to shed light on the mechanism of this reaction. Initially, two ^{18}O-labeling experiments were performed to verify the origin of the oxygen atom (Scheme 4a). When this reaction was conducted under an $^{18}O_2$

Scheme 4. Mechanistic Experiments



atmosphere, an ^{18}O-labeling product [^{18}O]-**56** was obtained in 92% yield. However, in the presence of $H_2{}^{18}O$ (3.0 equiv), no [^{18}O]-**56** was observed. The above results confirm that O_2 (air) is the source of the oxygen atom of the alcohol product. The singlet oxygen or superoxide anion radical would be the reactive oxygen species responsible for this oxidative hydroxylation. To verify the presence of these reactive oxygen species in this reaction system, we conducted quenching experiments (Scheme 4b). The addition of 1,4-diazabicyclo[2.2.2]-octane (DABCO), which is known as a strong physical quencher of singlet oxygen,[23] did not decrease the rate of oxidation. However, this reaction did not work well in the presence of 1,4-benzoquinone as a superoxide quencher,[24] thus indicating that the superoxide anion radical is produced from O_2 in our reaction system.[25] Furthermore, the formation of H_2O_2 was detected in a typical iodometric experiment (see the Supporting Information (SI) for details). Direct use of H_2O_2 as the oxidant furnished the products in excellent yields, which suggested that H_2O_2 is the responsible oxidant in this photocatalytic system (Scheme 4c). To confirm the role of amine, some control experiments were carried out (Scheme 4d). When 1,2,3,4-tetrahydroisoquinoline was employed instead of Et_3N, imine **59** was obtained in 79% yield, suggesting that oxidation of amine can take place in this reaction. Moreover, we found that this reaction proceeds efficiently in the presence of an appropriate amount of secondary or primary amine. Finally, quantum yield measurements were performed. A quantum yield (Φ) of 0.05 was calculated (λ = 525 nm), thereby implying that a radical chain process is not involved in the reaction (see the SI for details).[26]

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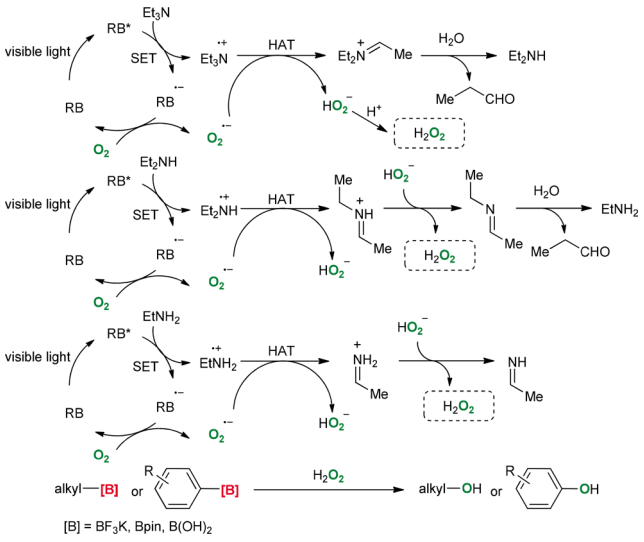
Based on these experimental observations, a plausible reaction mechanism is proposed in Scheme 5. The mechanism

Scheme 5. Proposed Reaction Mechanism



includes three successive photodegradation processes of Et_3N. At first, rose bengal (RB) is excited under visible-light irradiation followed by rapid intersystem crossing to give its long-lived triplet state species RB*, which oxidizes Et_3N through a single electron transfer (SET) process and subsequently reduces the O_2 molecule to a superoxide anion radical. This process allows the regeneration of RB. Next, the formed amine radical cation gets oxidized by a superoxide radical anion by a hydrogen-atom transfer (HAT) pathway to produce an imine with a concomitant generation of H_2O_2.[27] Hydrolysis of the imine affords the secondary amine Et_2NH, which then undergoes a similar photodegradation process to give a primary amine $EtNH_2$ as well as H_2O_2. Finally, photodegradation of $EtNH_2$ takes place in a similar manner to deliver another part of H_2O_2. The resulting H_2O_2 oxidizes various organoboron compounds to afford the desired products.

In summary, we have presented a simple and mild visible-light-mediated aerobic oxidative hydroxylation of organoboron compounds using air as oxidant under organic photocatalysis at room temperature. Remarkably, this protocol shows a broad substrate scope and good functional-group tolerance. A range of aliphatic alcohols and phenols can be readily obtained in moderate to excellent yields. Importantly, we demonstrated that chiral alcohol can be prepared stereospecifically using our method. All of these advantages as well its mild and environmentally friendly conditions and high efficiency make this protocol highly practical.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b02095.

Experimental details and characterization data for the products (PDF)

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Notes

The authors declare no competing financial interest.

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