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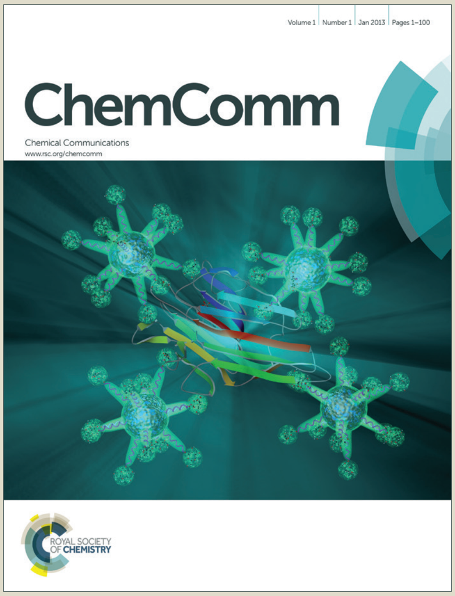

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# Visible Light Mediated Efficient Oxidative Benzylic $sp^3$ C−H to Ketone Derivatives under Mild Conditions using $O_2$

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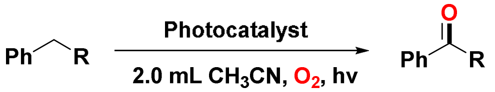
**A photooxygenation of benzylic $sp^3$ C−H reaction has been demonstrated using $O_2$ mediated by visible light. This protocol provides a simple and mild route to ketones from benzylic sp3 C−H bonds. Various benzylic $sp^3$ C−H bonds can be transformed into the desired ketone derivatives in moderate to good yields. The $^{18}O_2$ labelling experiments demonstrated that the oxygen introduced into ketone originated from dioxygen. A plausible mechanism has been proposed accordingly.**

As a representative C-H bond functionalization process, direct benzylic oxidation is one of the most useful and fundamental transformations due to its wide applications in the synthesis of pharmaceuticals and fine chemicals.[1] Up to now, numerous methods have been developed for the oxidation of benzylic $sp^3$ C−H bonds into the corresponding ketones. Traditionally, benzylic oxidation reactions involve the use of stoichiometric amounts metal oxidants.[2] During the last decades, great efforts that combining catalytic amount of transition metal complexes or other additives such as KBr etc with various oxidants like IBX, PhIO, *tert*-butylhydroperoxide (TBHP) and oxone under homogeneous and heterogeneous conditions have been made to modify these oxidative processes.[3] Molecular oxygen ($O_2$) as a cheap, safe and environmentally friendly oxidant has been increasingly used in many common oxidative processes.[4] Recently, direct oxidation of benzylic C–H bond have been well-developed using molecular oxygen as the oxidant, which provided a green route to ketones from benzylic $sp^3$ C−H bonds.[5] However, this area always required transition metal catalyst, additive or high temperature. Thus, the development of simple and mild conditions for selective benzyl oxidation is highly desirable.

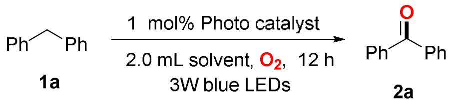
Visible-light, a clean, inexpensive, and 'almost infinitely' available source of energy, has received increasing attentions of the chemistry community. Since 2008, visible-light photoredox catalysis has received widespread research interest and various useful radical chemical transformations have been demonstrated.[6] Based on photo-induced single-electron transfer process, visible-light photocatalysis provide new protocol to C–H bonds functionalization that enable an environmentally friendly approach to the construction of valuable chemicals under mild conditions.[7] The molecular oxygen was also used as the oxidant in many visible light mediated oxidative reactions. In these photocatalytic reactions, two different roles of $O_2$ are included, oxidases and oxygenases. The oxidases means the direct reduction of $O_2$ to water or hydrogen peroxide to couple diverse oxidation reactions, which exists in most visible light mediated oxidative reactions.[7h, 7r, 8] The oxygenases means directly incorporate oxygen atoms into the organic molecule and limited examples have been focused on. In 2012, Xiao and co-workers realized the visible-light initiated aerobic oxidative hydroxylation of arylboronic acids and transform the oxygen of $O_2$ into the product phenol.[9] Herein, we report a photooxygenation of benzylic $sp^3$ C−H reaction using $O_2$ mediated by visible light (Scheme 1). This protocol provides a simple and mild route to ketones from benzylic $sp^3$ C−H bonds.



**Scheme 1.** Visible light mediated efficient oxidative benzylic $sp^3$ C−H to ketones.

The 9-mesityl-10-methylacridinium ion ($Acr^+$–Mes $ClO_4^-$) has been widely applied in many photocatalytic reactions because of strong oxidizing and reducing ability of the photoexcitation of $Acr^+$–Mes $ClO_4^-$.[10] The photocatalytic oxygenation of *p*-xylene, durene and mesitylene has been successfully transformed into the corresponding aldehydes using $Acr^+$–Mes $ClO_4^-$.[11] Though great success has been achieved, the xenon lamp was used and no ketones from benzylic $sp^3$ C−H bonds have been reported. Initially, we started our evaluation of the reaction parameters with diphenylmethane **1a** as the model substrate using $Acr^+$–Mes $ClO_4^-$ as the photocatalyst. To obtain optimized conditions for this reaction, we tried different photocatalyst and a range of solvents. The anticipated benzophenone product **2a** could be achieved in 82% yield in the presence of 1.0 mol% Acr$^+$–Mes $ClO_4^-$ using $CH_3CN$ as the solvent in $O_2$ atmosphere under a 3W blue LEDs (Table 1, entry 1). Other data in Table 1 illustrates the impact of different conditions on the efficiency of this reaction. Other tested photocatalyst such as $Ru(bpy)_3Cl_2$ or Eosin Y got trace or 22% desired product (Table 1, entries 2 and 3). Among the screened solvents, the DMF did not promote the reaction at all (Table 1, entry 5). The DCE can get almost the same result as $CH_3CN$ (Table 1, entry 4). Considering the environment-friendly solvent effect, we chose the $CH_3CN$ as our target solvent to perform the oxidative reaction. In the control experiments, no desired product was observed with neither photoredox catalyst nor light indicating that the photoredox catalysis is essential to this process (Table 1, entries 6 and 8). The reaction did not happen without $O_2$ (Table 1, entry 7).

**Table 1.** Optimization of reaction conditions [a]

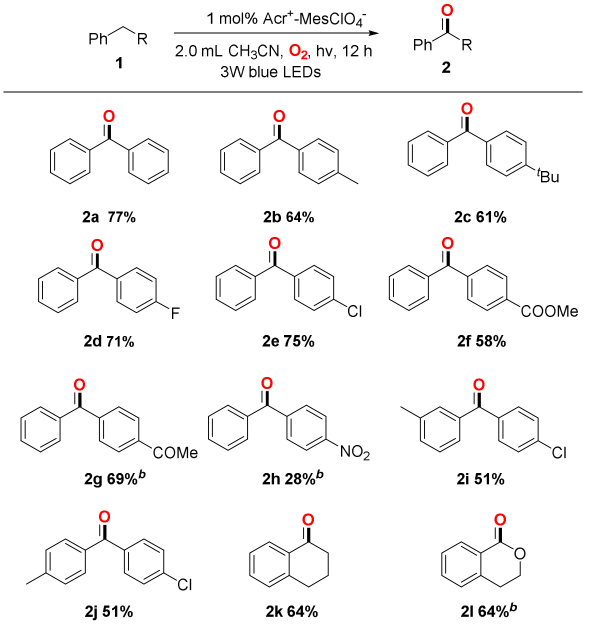


| entry | Photo catatlyst | solvent | yield(%)[b] |
|---|---|---|---|
| 1 | Acr$^+$–Mes | $CH_3CN$ | 82(77) |
| 2 | $Ru(bpy)_3Cl_2$ | $CH_3CN$ | n. d.[c] |
| 3 | Eosin Y | $CH_3CN$ | 22 |
| 4 | Acr$^+$–Mes | DCE | 80 |
| 5 | Acr$^+$–Mes | DMF | n. d.[c] |
| 6[d] | Acr$^+$–Mes | $CH_3CN$ | n. d.[c] |
| 7[e] | Acr$^+$–Mes | CH3CN | n. d.[c] |
| 8 | No | $CH_3CN$ | n. d.[c] |
| 9[f] | Acr$^+$–Mes | $CH_3CN$ | 46 |

[a]Reaction conditions: **1a** (0.50 mmol), photocatalyst (1.0 mol%) in solvent (2.0 mL) at rt in $O_2$ under 3W blue LEDs for 12 h. [b]Determined by GC using biphenyl as an internal standard, the yield in the parentheses is isolated yield. [c]n.d.= no desired product. [d]without light. [e]Under $N_2$ atmosphere. [f]Under Air atmosphere, isolated yield.

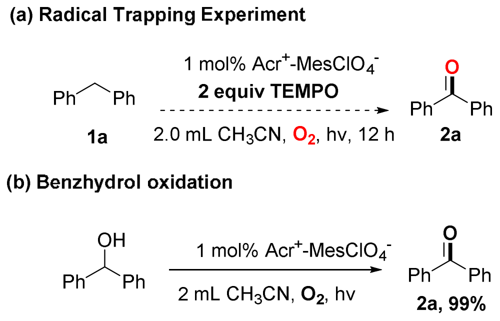
With the optimal conditions established, various benzylic $sp^3$ C−H compounds were investigated and the results are summarized in Table 2. This oxidative reaction was compatible with aryl-Me, $^t$Bu and F groups (**2b–2d**). The C-Cl group was well tolerated in this reaction providing the possibility for further functionalization (**2e**). The electron-withdrawing groups such as COOMe (**2f**) or COMe (**2g**) can also afford the desired ketone products in moderate yields, while the $NO_2$ group afforded a poor yield (**2h**). In addition, this reaction was successfully amenable to the substitutions on the two arenes (**2i, 2j**), which demonstrated a diversity of this protocol. Interestingly, 1,2,3,4-tetrahydronaphthalene could also be employed to get the target alkenylation product without any difficulties (**2k**). Furthermore, the isochromane was also suitable for this reaction and the corresponding products were obtained in 64% yields (**2l**).

**Table 2.** Visible light mediated efficient oxidative benzylic $sp^3$ C−H to ketones [a]



[a] Reactions were conducted at rt for 12 h using **1** (0.50 mmol), Photo catalyst Acr$^+$–$MesClO_4^-$ (1.0 mol%) in $CH_3CN$ (2.0 mL) in $O_2$ atomosphere under 3W blue LEDs. Isolated yields are shown. [b]3.0 mol% Acr$^+$-$MesClO_4^-$.

To gather insights into this oxidative reaction, radical scavengers, such as TEMPO, was employed in the reaction (Scheme 2a). The reaction was completely shut down, which could indicate that this transformation involved radical intermediates. Based on literature, the benzyl alcohol derivative may be a possible intermediate in the formation of ketone. We also used the benzhydrol as the substrate under standard conditions and almost quantitative yield was got (Scheme 2b), which reveals that benzyl alcohol derivative may be an intermediate in this oxidation reaction. The time profile of photocatalytic reaction shown in Figure 1 revealed that the reaction was totally inhibited in the absence of light. This result indicated that continuous irradiation of visible light is essential to this photo-catalytic transformation.



**Scheme 2.** Radical - trapping and benzhydrol oxidation experiments

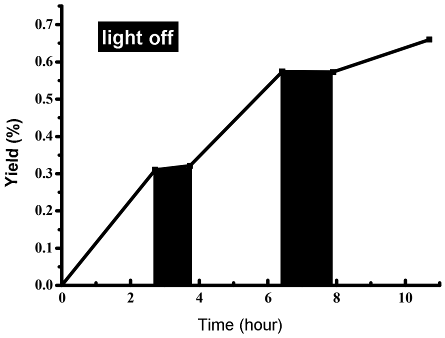


**Figure 1.** Time profile of photocatalytic reaction with and without visible light.

To study where the oxygen comes from, $^{18}O$ labeling experiments were conducted using diphenylmethane **1a** and benzophenone **2a** as substrates. The data listed in Table 3 suggests that carbonyl oxygen on benzophenone **2a** originates from $O_2$, which can readily exchange with water under the visible-light conditions. Firstly, we used the labelling $^{18}O_2$ to do the reaction under the standard conditions and a mixture of of $^{16}O$ and $^{18}O$ was generated, in which the ratio was 0.5: 1.0. When using $H_2{}^{18}O$ to do the labeling experiment, the ratio of $^{16}O$ and $^{18}O$ was 0.6: 1.0. When the diphenyl ketone product **2a** and $H_2{}^{18}O$ was stirring under standard conditions, a mixture $^{16}O$ and $^{18}O$ was also generated. This result revealed that carbonyl oxygen on benzophenone **2a** can exchange with water under the visible-light conditions.[12] In addition, no labeling product was generated when just mixing benzophenone **2a** and $H_2{}^{18}O$ without visible light and photocatalyst. This means the visible light and photocatalyst is essential for the exchange of benzophenone **2a** with water.

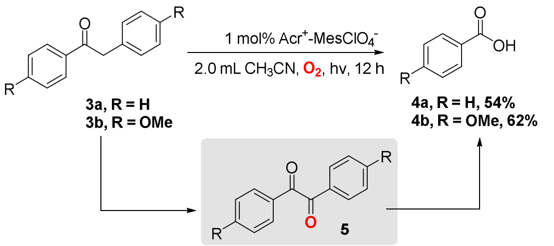
**Table 3.** $^{18}O$ labeling experiment[a]

| entry | substrate (mmol) | $^{18}O$-reagent | product[a] |
|---|---|---|---|
| 1[b] | **1a** (0.2 mmol) | $^{18}O_2$ | **2a** $^{16}O/^{18}O = 0.5 : 1.0$ |
| 2[c] | **1a** (0.2 mmol) | $H_2{}^{18}O$ | **2a** $^{16}O/^{18}O = 0.6 : 1.0$ |
| 3[d] | **2a** (0.2 mmol) | $H_2{}^{18}O$ | **2a** $^{16}O/^{18}O = 0.85 : 1.0$ |
| 4[e] | **2a** (0.2 mmol) | $H_2{}^{18}O$ | **2a** Only $^{16}O$ labeling |

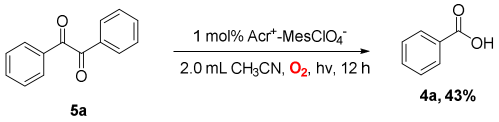
[a] The ratio of $^{16}O$ to $^{18}O$ on products was determined by GC-MS. [b] Reaction conditions: **1a** (0.20 mmol), Photo catalyst Acr$^+$–MesClO$_4^-$ (1 mol%) in $CH_3CN$ (2.0 mL) at rt in $^{18}O_2$ under 3W blue LEDs for 10 h. [c] Reaction conditions: **1a** (0.20 mmol), Photo catalyst Acr$^+$–MesClO$_4^-$ (1 mol%), $H_2{}^{18}O$ (2.0 mmol) in $CH_3CN$ (2.0 mL) at rt in $O_2$ under 3W blue LEDs for 10 h. [d] Reaction conditions: **2a** (0.20 mmol), Photo catalyst Acr$^+$–MesClO$_4^-$ (1 mol%), $H_2{}^{18}O$ (2.0 mmol) in $CH_3CN$ (2.0 mL) at rt in $O_2$ under 3W blue LEDs for 10 h. [e] Reaction conditions: **2a** (0.20 mmol), $H_2{}^{18}O$ (2.0 mmol) in $CH_3CN$ (2.0 mL) at rt for 10 h.

When trying 1,2-diphenylethan-1-one derivatives as the substrates under standard conditions, no target oxygenation product was detected. To our surprise, the benzoic acid derivatives were formed based on GC-MS and NMR spectroscopy (Scheme 3a). In this reaction, we proposed that the benzil derivatives **5** may be the key intermediate to the benzoic acids. To verify our hypothesis, we demonstrate the reaction using benzil **5a** under our standard conditions. The corresponding benzoic acid product **4a** was obtained in 43% yields. Thus, these results indicated that 1,2-diphenylethan-1-one derivatives may be firstly oxidized to benzil derivatives, which under further transformation into benzoic acids.

**(a) Visible Light Mediate Oxidative Reaction of 1,2-Diphenylethan-1-one Derivatives**

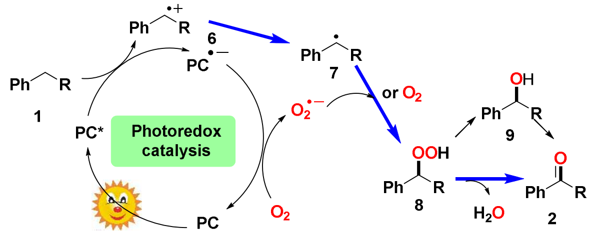


**(b) Visible Light Mediate Oxidative Reaction of Benzil**



**Scheme 3**. Visible light mediated oxidative reaction of 1,2-diphenylethan-1-one derivatives and benzil.

Based on the previous reports[10a] and experimental results, a plausible mechanism is proposed in Scheme 4. Firstly, the photocatalyst Acr$^+$–Mes ClO$_4^-$ is excited by visible light irradiation (3W blue LEDs) to generate the excited species [Acr$^+$–MesClO$_4^-$]*, which then undergoes single electron transfer (SET) process with benzylic sp$^3$ C−H compound to generate radical cation **6**, and [Acr$^+$–MesClO$_4^-$] radical anion. The [Acr$^+$–MesClO$_4^-$] radical anion can be oxidized by $O_2$ to recycle the photocatalytic reaction. The generated radical cation **6** loses one proton to generate the radical **7**, which can react with $O_2$ or $O_2$ radical anion to generate the hydroperoxidate intermediate **8**. This intermediate **8** then loses one water and transformed into the desired ketone product **2**. The hydroperoxidate intermediate **8** may also transformed into the benzyl alcohol derivative **9**, which under further oxidation to ketone **2**.



**Scheme 4**. Proposed mechanism

In conclusion, we have disclosed a photooxygenation of benzylic sp$^3$ C−H reaction using $O_2$ mediated by visible light. This protocol provides a simple and mild route to ketones from benzylic sp$^3$ C−H bonds. Various benzylic sp$^3$ C−H bonds can be transformed into the desired ketone derivatives in moderate to good yields. The $^{18}O_2$ labelling experiments demonstrated that the oxygen introduced into ketone originated from dioxygen. We believe this oxidative reaction mediated by visible light will help chemists to design more and more interesting, useful, and sustainable reactions in the near future.

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## Notes and references

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† Electronic Supplementary Information (ESI) available: Experimental procedure, characterization data, and copies of $^{1}$H, $^{13}$C NMR spectra. See DOI: 10.1039/c000000x/

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