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# Visible Light-Mediated Photocatalytic Metal-Free Cross-Coupling Reaction of Alkenyl Carboxylic Acids with Diarylphosphine Oxides Leading to *β*-Ketophosphine Oxides

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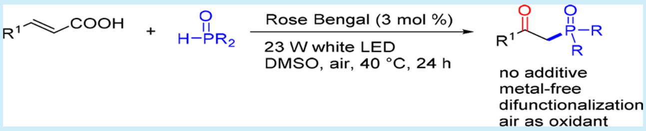
*Supporting Information*

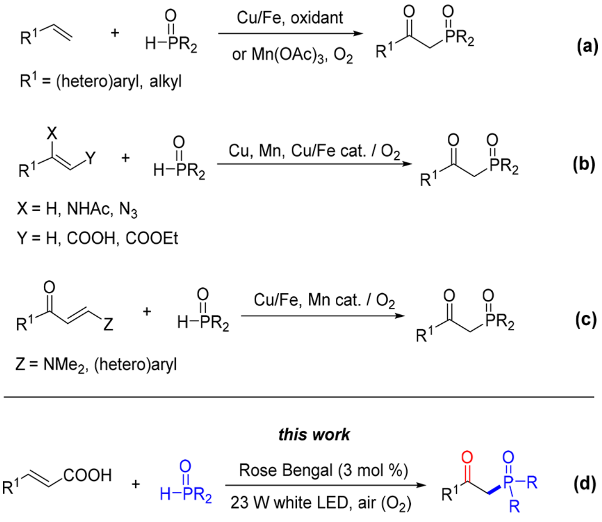
**ABSTRACT:** A new visible light-mediated photocatalytic decarboxylative oxyphosphorylation of cinnamic acids with diarylphosphine oxides is described. This reaction is performed under mild conditions to afford the corresponding *β*-ketophosphine oxides.



*β*-Ketophosphine oxides are important organophosphorus compounds that have been widely used as metal extractants[1] and potential ligands[2] due to their coordination ability. They have also been employed as intermediates for the preparation of important heterocycles,[3] *α*,*β*-unsaturated carbonyl compounds via the Horner−Wadsworth−Emmons (HWE) reactions[4] as well as in other vital chemical transformations.[3a,5] Therefore, the development of methods for their preparation is still of considerable importance. Traditionally, *β*-ketophosphine oxides can be synthesized by the reactions of *α*-haloketones and trialkylphosphites (Arbuzov reaction)[6] or via the acylation of alkylphosphine oxides.[7] Recently, a number of reports have been described based on the transition metal-catalyzed oxyphosphorylation of alkenes and their derivatives (Scheme 1a and 1b).[8,9] Another recent strategy is phosphorylation of *α*,*β*-conjugated alkenes via cleavage of the C = C double bond (Scheme 1c).[10]

Visible light-mediated photoredox catalysis has become a powerful tool in modern synthetic chemistry due to its mild and energy saving nature, environmental friendliness and potential application in industry.[11] This strategy has been employed by several groups in recent years to construct important phosphorylated compounds via C(sp$^2$)−P bond formation.[12] On the other hand, cinnamic acids due to their stability and accessibility, have been employed as viable substrates via extrusion of carbon dioxide, to access a variety of valuable compounds.[13] Our goal is to develop a greener access to *β*-ketophosphine oxides via the visible light-induced photocatalytic reaction of cinnamic acids with H-phosphine oxides. In continuation of our recent efforts on C(sp$^3$)−P bond formation,[8c,14] we report the first example of visible light-mediated photocatalytic metal-free decarboxylative oxyphosphorylation of alkenyl carboxylic acids with diarylphosphine oxides leading to *β*-ketophosphine oxides (Scheme 1d).

**Scheme 1. Synthesis of *β*-Ketophosphine Oxides from Alkenes**



Our initial study focused on the reaction of cinnamic acid (**1a**) and diphenylphosphine oxide (**2a**) under 23W white LED irradiation using Eosin Y as a photocatalyst. To our delight, the desired product **3a** (50%) was obtained in MeCN after 24 h at 40 °C (Table 1, entry 1). The reaction was further investigated in other solvents (Table 1, entries 2−7), and we observed a slightly improved yield (56%) in DMSO (Table 1, entry 2). In order to adhere to the original goal of developing a low-cost and greener protocol, we lowered the amount of Eosin Y to 3 mol % and this led to a further improvement in the yield of **3a** to 67% (Table 1, entry 8). However, a further reduction in the amount of photocatalyst (to 1 mol %) had a detrimental effect on the reaction yield (Table 1, entry 9). In search of a more efficient catalyst, other kinds of organic dyes were evaluated as photoredox catalysts (Table 1, entries 10−13). While all tested catalysts furnished product **3a**, Rose Bengal (RB) was most efficient thus affording **3a** in 70% yield

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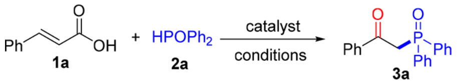
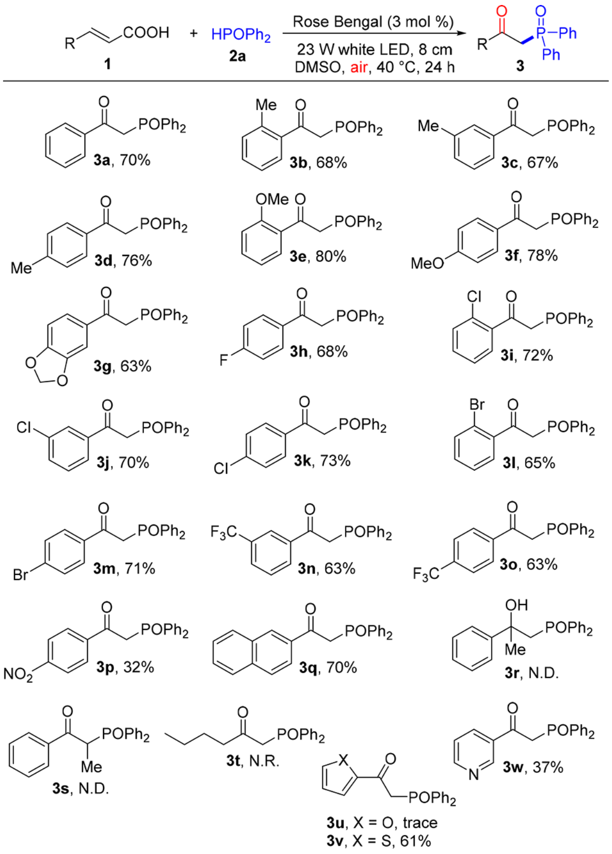
Table 1. Optimization of the Reaction Conditions[a]



| entry | catalyst (mol %) | solvent | yield (%)[b] |
|---|---|---|---|
| 1 | Eosin Y (5) | MeCN | 50 |
| 2 | Eosin Y (5) | DMSO | 56 |
| 3 | Eosin Y (5) | *t*-BuOH | 25 |
| 4 | Eosin Y (5) | EtOH | 23 |
| 5 | Eosin Y (5) | DMF | 47 |
| 6 | Eosin Y (5) | DCE | 33 |
| 7 | Eosin Y (5) | 1,4-dioxane | trace |
| 8 | Eosin Y (3) | DMSO | 67 |
| 9 | Eosin Y (1) | DMSO | 21 |
| 10 | Rose Bengal (3) | DMSO | 70 |
| 11 | fluorescein (3) | DMSO | 62 |
| 12 | rhodamine B (3) | DMSO | 67 |
| 13 | rhodamine 6G (3) | DMSO | 47 |
| 14[c] | Rose Bengal (3) | DMSO | 59 |
| 15[d] | Rose Bengal (3) | DMSO | trace |
| 16 | – | DMSO | NR[e] |
| 17[f] | Rose Bengal (3) | DMSO | 63 |
| 18[g] | Rose Bengal (3) | DMSO | 45 |
| 19[h] | Rose Bengal (3) | DMSO | 33 |
| 20[i] | Rose Bengal (3) | DMSO | trace |
| 21[j] | Rose Bengal (3) | DMSO | 43 |
| 22[k] | Rose Bengal (3) | DMSO | 65 |

[a] Reaction conditions: **1a** (0.25 mmol), **2a** (1 mmol), photocatalyst, solvent (1.5 mL), and irradiation with a 23 W white LED lamp at 40 °C for 24 h under air. [b] Isolated yield. [c] With 16 W green LED. [d] No light. [e] NR means no reaction. [f] **1a**:**2a** = 1:3. [g] **1a**:**2a** = 1:2. [h] At 30 °C. [i] At 20 °C. [j] 12 h. [k] 50 °C.

Scheme 2. Scope of Alkenyl Carboxylic Acids 1[a]



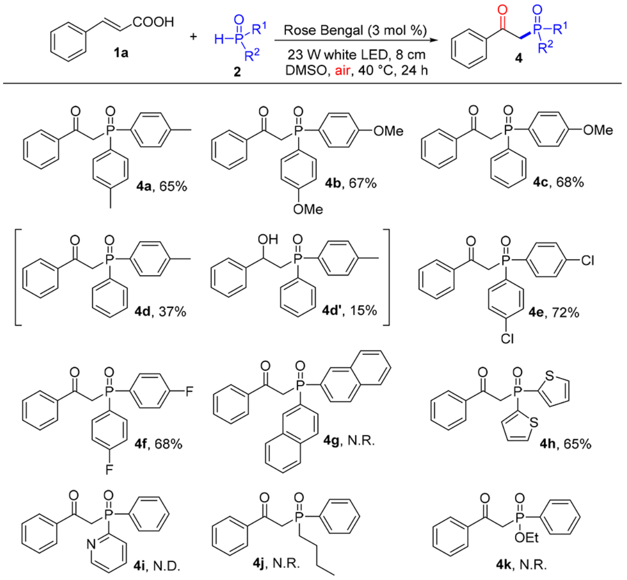
[a] Reaction conditions: **1** (0.25 mmol), **2a** (1 mmol), Rose Bengal (3 mol %) in DMSO (1.5 mL), and irradiation with a 23 W white LED lamp at 40 °C for 24 h under air.

(Table 1, entry 10). Furthermore, when the visible light source was switched to green LED, a lower yield was obtained (Table 1, entry 14). Notably, only a trace amount of **3a** was observed without the LED lamp while there was no reaction in the absence of a photocatalyst (Table 1, entries 15 and 16). After a quick screen of other conditions such as the ratio of reactants, reaction temperature and time (Table 1, entries 17–20), the reaction conditions in entry 10 were chosen as optimal for the investigation of substrate scope.

After the optimized reaction conditions were established, a variety of alkenyl carboxylic acids were subsequently examined. As shown in Scheme 2, substrates with different electronic and steric properties all underwent the reaction to deliver the corresponding difunctionalized products in satisfactory yields. The presence of either electron-donating or halo groups at various positions on the aromatic ring had no obvious effect on the reaction outcome. As a result, good yields of products were observed in all cases (Scheme 2, **3b**–**3m**). The reaction of the trifluoromethyl containing substrates resulted in good yields of the desired products **3n** and **3o**, product **3p** could only be obtained in 32% yield due to the strong electron-withdrawing effect of the 4-nitro group. Meanwhile, the 2-naphthyl containing substrate afforded 70% yield of the corresponding product **3q**. Furthermore, the effect of *α*- and *β*-substituents were investigated. Thus, 3-phenylbut-2-enoic acid, a *β*-methyl substrate, was subjected to the reaction, but unfortunately, the expected product **3r** was not detected. Acetophenone was isolated instead, which might result from cleavage of the C=C double bond. Likewise, no desired product **3s** was detected with 2-methyl-3-phenylacrylic acid, probably due to the steric hindrance caused by the *α*-methyl group. No reaction was observed for aliphatic acrylic acid, probably attributed to the low reactivity of substrate. The reaction of (furan-2-yl) acrylic acid resulted in complicated mixtures of products. The desired product **3u** could only be detected in trace amounts by HRMS. The poor selectivity of the reaction might be attributed to the high reactivity of the starting material. However, the reaction with (thiophen-2-yl) acrylic acid gave the desired product **3v** in 61% yield. In addition, the reactions of substrates such as conjugated (hetero)cyclohexenyl carboxylic acids, heteroaryl carboxylic acids and (*E*)-3-(pyridin-3-yl)acrylic acid were carried out. While the first two types of compounds were inert toward the reaction, (*E*)-3-(pyridin-3-yl)acrylic acid produced the desired product **3w** in 37% yield (see Supporting Informtion, Scheme S1).

Next, the reactions of cinnamic acid with a variety of phosphine oxides were also investigated (Scheme 3). Both electron-rich and halo- containing diaryl phosphine oxides gave the expected products in yields ranging from 65 to 72% (Scheme 3, **4a**–**4c**, **4e**, **4f**). However, phenyl(*p*-tolyl)-phosphine oxide produced a mixture of products **4d** and **4d′** (2.5:1). The reason for this outcome remains unknown. Due to the obvious steric hindrance, the reaction of dinaphthyl phosphine oxide did not take place. Fortunately, di(thiophen-2-yl)phosphine oxide was compatible under the present condition to afford the expected product **4h** in 65% yield. Unfortunately, other phenylphosphine oxides containing the 2-

### Scheme 3. Scope of Diphenylphosphine Oxide Derivatives[a]

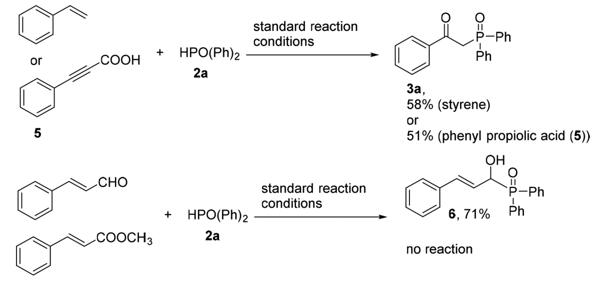


[a]Reaction conditions: **1a** (0.25 mmol), **2** (1 mmol), Rose Bengal (3 mol %) in DMSO (1.5 mL), and irradiation with a 23 W white LED lamp at 40 °C for 24 h under air.

pyridyl, *n*-butyl, and ethoxy groups could not furnish the corresponding products probably due to their lower reactivity.

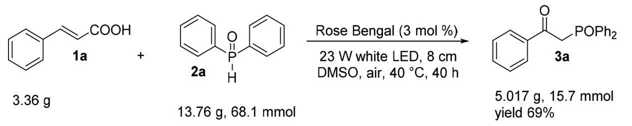
In order to know the generality and limit for this methodology, the reaction of styrene, cinnamaldehyde, methyl cinnamate, and an alkynyl carboxylic acid, phenyl propiolic acid (**5**) with diphenylphosphine oxide (**2a**) were also explored under the same reaction conditions. The results indicate that the reaction with styrene and phenyl propiolic acid (**5**) could all produce 2-(diphenylphosphoryl)-1-phenylethan-1-one (**3a**) in 58% and 51% yields, respectively; however, the reaction with cinnamaldehyde afforded the nucleophilic addition product **6**,[15] and no reaction was observed for methyl cinnamate (Scheme 4).

### Scheme 4. Reaction of Styrene, Cinnamaldehyde, Methyl Cinnamate, and Phenyl Propiolic Acid (5) with Diphenyl Phosphine Oxide



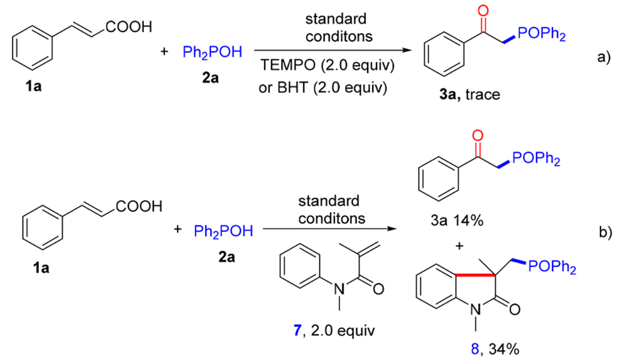
In order to demonstrate the scalability of this visible-light driven photocatalytic reaction, a 5-g scale preparation of *β*-ketophosphine oxide **3a** was performed as illustrated in Scheme 5. Cinnamic acid (**1a**) (3.36 g, 22.7 mmol), diphenylphosphine oxide (**2a**) (13.76 g, 68.1 mmol), Rose Bengal (0.69 g, 0.68 mmol), and DMSO (100 mL) were placed in a crystallizing dish (diameter 8 cm). The mixture was stirred at 40 °C for 40 h under irradiation by a 23 W white LED until the completion of the reaction (monitored by TLC). After workup, the desired product **3a** was obtained in 69% yield (5.017 g).

### Scheme 5. Gram Experiment



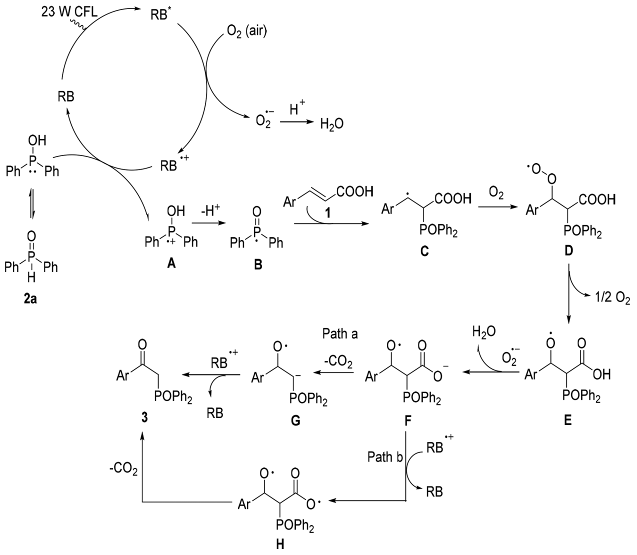
In order to gain an insight into the reaction mechanism, some control experiments were carried out as shown in Scheme 6. Initially, the standard reactions were performed in the presence of either TEMPO or BHT. Although, there was no radical capture in both cases, the suppressed formation of **3a** suggests that radical species were involved in the reaction (Scheme 6a). Delightfully, the participation of phosphoryl radicals was confirmed *via* the reaction of cinnamic acid (**1a**) and diphenylphosphine oxide (**2a**) in the presence of *N*-methyl-N-phenylmethacrylamide **7**. This resulted in only 14% yield of product **3a** along with 34% of phosphoryl radical mediated cyclization product **8** (Scheme 6b). Furthermore, to confirm the presence of superoxide anion radical and diphenylphosphoryl radical, trapping experiments with 5,5-dimethyl-1-pyrroline *N*-oxide (DMPO) were performed. Pleasingly, the EPR experiment detected the presence of the adduct of DMPO−$O_2^{\bullet-}$ (see Supporting Information, Figures S1 and S2 and Scheme S2) and DMPO-$POPh_2^{\bullet}$ (see Supporting Information, Figure S3 and Scheme S3).[16]

### Scheme 6. Control Experiments



On the basis of the results above and studies from literature,[14,16−18] a mechanism is proposed for the visible-light driven photocatalytic decarboxylative oxyphosphorylation of cinnamic acids (Scheme 7). Under visible light irradiation, the RB photocatalyst is induced into its excited state (RB*) which further interacts with oxygen from air to give the superoxide anion radical and RB radical cation ($RB^{\bullet+}$).[16c] Then, **2a** could interact with $RB^{\bullet+}$ to give the radical cation **A** and ground state RB to complete the photoredox cycle. The radical cation **A** could initially undergo loss of proton to afford the key phosphorus-centered radical **B** which subsequently attacks the *α*-position of cinnamic acid **1** to form intermediate radical **C**. Cinnamic acid **1** could not be oxidized by RB* due to its high oxidation potential ($E_{1/2}^{ox}$ = +2.01 V vs SCE in MeCN).[18b] The resultant intermediate radical **C** could react with molecular oxygen to form radical **E** via peroxo radical **D**.[18a] The abstraction of a proton by the superoxide anion radical from **E** would give intermediate **F**,[18b] which can further undergo decarboxylation and electron transfer process to

**Scheme 7. Proposed Mechanism for Decarboxylative Oxyphosphorylation of Cinnamic Acid**



afford product **3** (path a).[18c] Alternatively, **F** can interact with $RB^{\bullet+}$ via a single electron transfer process to give **H** and subsequent decarboxylation would afford **3** (path b).[18b]

In summary, a new visible-light-mediated photocatalytic strategy for the decarboxylative oxyphosphorylation of cinnamic acids with diarylphosphine oxides under air has been developed. This metal-free procedure requires neither oxidant nor additive and is applicable to wide range of substrates including aryl propiolic acids. Notably, the reaction could be carried out on a 5 g scale. This reaction represents a mild and efficient method of preparation of *β*-ketophosphine oxides.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b02639.

Experimental details and spectral data for compounds **3**, **4**, **6**, and **8** (PDF)

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### Notes

The authors declare no competing financial interest.

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