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FULL PAPER

Photocatalytic Dual Decarboxylative Alkenylation Mediated by Triphenylphosphine and Sodium Iodide

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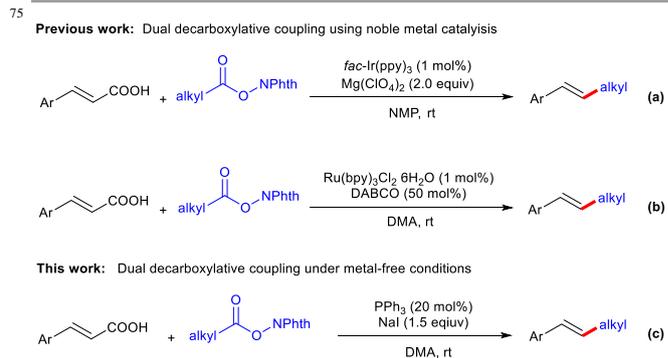
An efficient photocatalytic dual decarboxylative alkenylation of α,β -unsaturated carboxylic acids and alkyl *N*-hydroxyphthalimide (NHP) esters mediated by triphenylphosphine and sodium iodide has been developed.

This protocol proceeds under 456-nanometer irradiation by visible blue light in the absence of transition metals or organic dye based photoredox catalysts. The reaction is successfully applied to a wide range of redox-active esters derived from aliphatic carboxylic acids (1°, 2° and 3°) and α -amino acids, enabling transformations of diverse α,β -unsaturated carboxylic acids to α,β -alkylated styrenes with high efficiency and excellent selectivity under mild conditions.

Carboxylic acids and their derivatives are a highly important class of platform molecules which have broad applications in biological and chemical synthesis because they are abundant, low cost, easy to store, and simple to handle.¹ One of the most functional transformations of carboxylic acids and their derivatives is decarboxylative cross-coupling, in which undergoes C-C bond cleavage to liberate CO₂ along with the construction of new C-C or C-heteroatom bond.²⁻⁴ Generally, the decarboxylative cross-coupling process are achieved by transition metal-catalyzed extrusion of CO₂ to form nucleophilic organometallic intermediates.⁵⁻⁷ Despite these achievements obtained in this area, the current methods require metal catalysis or high temperature. The development of a mild and environmentally-benign route to form this process is highly desired.

In recent years, visible-light-induced radical decarboxylative functionalization of carboxylic acids and their derivatives has emerged as a powerful method for the construction of C-C bond owing to their sustainability, practicality, and environmental friendliness.⁸⁻¹⁰ The radical intermediates formed from carboxylic acids can directly participate in cross-coupling under

photocatalytic conditions, among them, *N*-hydroxyphthalimide (NHP) esters were demonstrated to be very efficient alkyl source in C(sp³)-C bond formation.^{11,12} In 2017, Duan and co-workers^{12a} reported a visible-light-mediated decarboxylative coupling between alkyl NHP esters and α,β -unsaturated carboxylic acids in the presence of *fac*-Ir(ppy)₃ and Mg(ClO₄)₂ (scheme 1a). In the same year, Xu, Zhang and co-workers^{12b} described a similar visible-light-mediated dual decarboxylative reaction using Ru(bpy)₃Cl₂ as the photocatalyst and DABCO as the base for this transformation (scheme 1b). Although these two methods provide an efficient way to construction of α,β -alkylated styrenes, they require noble metal catalysis. Recently, Shang, Fu and co-workers^{12c} developed a new photocatalytic decarboxylative alkenylation reactions of *N*-(acyloxy)phthalimide derived from α -amino and α -hydroxy acids with 1,1-diarylethene, and with cinnamic acid derivatives. The advantage of this reaction was using sodium iodide and triphenylphosphine instead of a traditional dye- or metal as redox catalysts for the synthesis of allylic amine or allylic ether derivatives, but a wide range of alkyl NHP esters did not involved. Therefore, we envisaged that using triphenylphosphine and sodium iodide to achieve decarboxylative alkenylation of α,β -unsaturated carboxylic acids with alkyl *N*-hydroxyphthalimide esters, which applied to a wide range of redox-active esters derived from aliphatic carboxylic acids (1°, 2° and 3°) and α -amino acids.



Scheme 1 Photocatalytic dual decarboxylative alkenylation.

Herein, we report a new, metal-free photocatalytic dual decarboxylative alkenylation between α,β -unsaturated carboxylic acids and alkyl NHP esters in combination of

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triphenylphosphine and sodium iodide (scheme 1c). It should be noted that a wide range of redox-active esters derived from aliphatic carboxylic acids (1°, 2° and 3°) as well as α -amino acids were found to be compatible. This method offers an efficient way to access α,β -alkylated styrenes with highly regio- and stereo-specificity.

The optimized reaction conditions for dual decarboxylative alkenylation using 4-methoxycinnamic acid **1a** and *N*-acyloxypthalimide **2a** as the model reaction partners (Table 1). To our delight, the desired decarboxylative coupling product **3aa** was isolated in 90% yield under blue LED irradiation of 20 mol% PPh₃ and 1.5 equiv of NaI in DMA at room temperature for 10 h (entry 1). Other iodides, including LiI, KI and *n*-Bu₄NI were examined: each of which exhibited good catalytic activity but was less efficient than NaI (entries 2-4). Other halides such as NaF, NaCl and NaBr were entirely ineffective (entry 5). Next, we screened a series of phosphines with different electronic and steric properties, the result showed that PPh₃ was the most efficient catalyst for stabilizing the iodine radical as a R₃P[•] species (entries 6-8). Among the solvents examined, DMA was the best option compared with other solvents such as DMSO, DMF, 1,4-dioxane or MeCN (entries 9-12). All three components phosphine, sodium iodide and irradiation were essential for the reaction (entries 13-15). Gratifyingly, the reaction (entry 16) scale up to 1 g of acid **1a** was successful to construct **3aa** in 85% yield for 36 h, (more details see the SI).

Table 1 Screening of optimal reaction conditions ^a



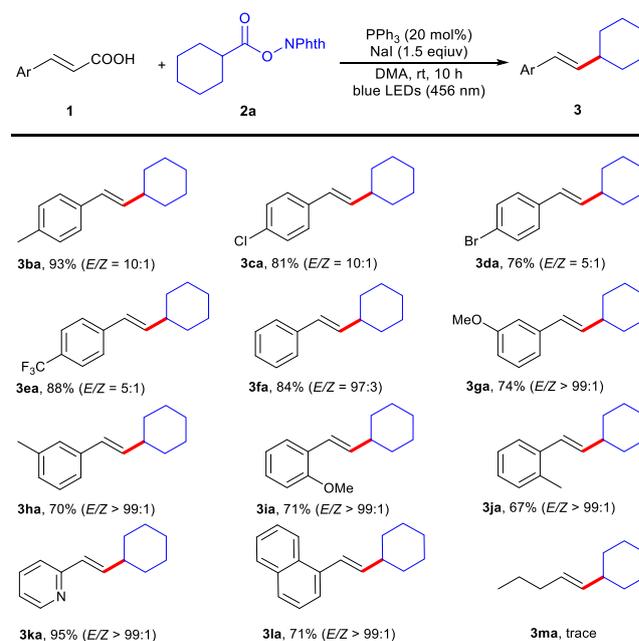
Entry	Variation from the optimal conditions	Yield [%]
1	none	90
2	LiI instead of NaI	40
3	KI instead of NaI	57
4	<i>n</i> -Bu ₄ NI instead of NaI	60
5	NaF, NaCl, NaBr instead of NaI	<5%
6	P(4-MeC ₆ H ₄) ₃ instead of PPh ₃	81
7	P(4-FC ₆ F ₄) ₃ instead of PPh ₃	84
8	PCy ₃ instead of PPh ₃	33
9	DMSO instead of DMA	62
10	DMF instead of DMA	60
11	1,4-dioxane instead of DMA	43
12	MeCN instead of DMA	35
13	without NaI	0
14	without PPh ₃	0
15	without blue LEDs	0
16 ^b	none	85

^a Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), NaI (1.5 equiv), PPh₃ (20 mol%), DMA (2 mL), room temperature, argon, and 10 h. *E/Z* was determined by ¹HNMR analysis of the crude mixture. ^b **1a** (1 g) and 36 h.

With the optimized reaction conditions in hand, we exploited the substrate scope of α,β -unsaturated carboxylic acids **1** in the coupling with alkyl NHP esters **2a**. As shown in Table 2, a wide range of cinnamic acids **1b-1e** with both

electron-donating and electron-withdrawing substituents at the *para*-position of the phenyl ring all reacted smoothly with alkyl NHP esters **2a** to give the corresponding styrenes **3ba-3ea** in 76-93% yields. The cinnamic acid **1f** with no group on the phenyl ring was well tolerated. Furthermore, *ortho*-substituted and *meta*-substituted cinnamic acids **1g-1j** were also suitable substrates in this transformation, but these showed lower reactivity than *para*-substituted cinnamic acids. To our delight, the electron-withdrawing substrate such as 3-(pyridin-2-yl)acrylic acid **1k** gave **3ka** in 95% yield. 3-(Naphthalen-2-yl)acrylic acid **1l** was also an effective substrate in this protocol, and the desired product **3la** was isolated in 71% yield. Unfortunately, 2-hexenoic acid **1m** was not suitable for this reaction, possibly due to the lower stability of the alkyl radical compared with the benzylic radical.

Table 2. Variations of α,β -unsaturated carboxylic acids (**1**) ^a

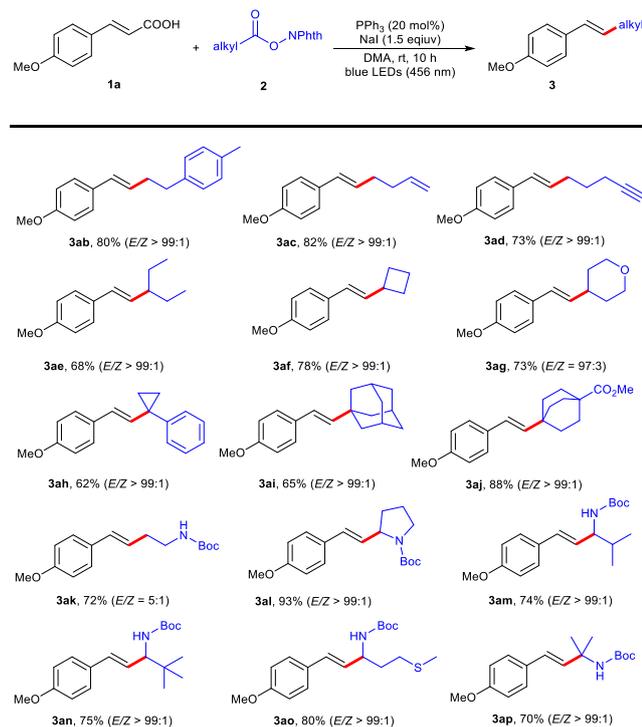


^a Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), NaI (1.5 equiv), PPh₃ (20 mol%), DMA (2 mL), room temperature, argon, and 10 h. *E/Z* was determined by ¹HNMR analysis of the crude mixture.

Subsequently, we examined the scope of alkyl NHP esters in this decarboxylative alkenylation reaction. As outlined in Table 3, A variety of functionalized alkyl NHP esters **2b-j** comprising 1°, 2°, and 3° alkyl groups were all suitable substrates and provided **3ab-3aj** in moderate to good yields. The 1° alkyl functional groups, such as aryl **2b**, terminal alkene **2c**, terminal alkyne **2d**, led to **3ab-3ad** in 73-82% yield. The 2° alkyl groups containing linear alkyl **2e**, cycloalkyl **2f** and heteroatoms on the aliphatic ring **2g** also worked well to give the desired products **3ae-3ag** in good yields. 1-Phenylcyclopropyl or adamantyl-possessing NHP esters **2h-2i** were delivered in moderate yields, but 3° bridge ring-containing NHP ester **2j** furnished **3aj** in high

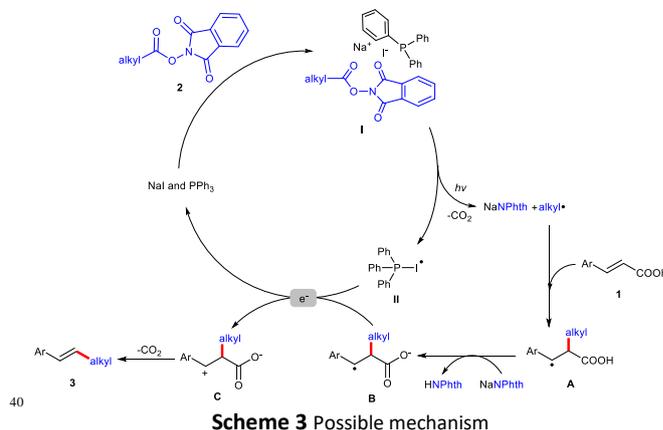
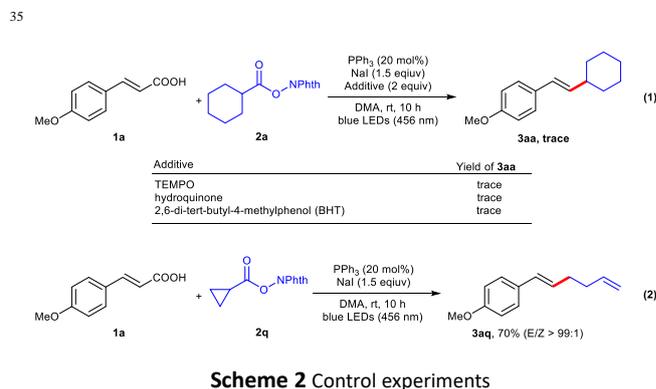
yield, probably due to the electron-withdrawing ester group. Alkyl NHP esters **2k-2p** derived from various natural and unnatural amino acids were reactive to give substituted allylic amine product **3ak-3ap**. Cinnamic acids could be transformed into their amine derivatives in one step, demonstrating the potential of this method for rapid, post-synthetic natural products or drug modification.

Table 3. Variations of the alkyl NHP esters (**2**)^a



^a Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), NaI (1.5 equiv), PPh₃ (20 mol%), DMA (2 mL), room temperature, argon, and 10 h. *E/Z* was determined by ¹HNMR analysis of the crude mixture.

radical intermediate **A**. Deprotonation of the intermediate **A** with NaNPhth gives HNPhth and the radical intermediate **B**, which direct oxidized by Ph₃P-I[•] species **II** to produce the intermediate **C**, finally the intermediate **C** could release CO₂ to deliver the desired product **3**.



In summary, we have developed a new visible-light-induced dual decarboxylative alkenylation of α,β -unsaturated carboxylic acids with alkyl *N*-hydroxyphthalimide esters through in combination of triphenylphosphine and sodium iodide. This reaction provided a facile and efficient way for the preparation of α,β -alkylated styrenes. Moreover, the reaction features mild conditions, good functional group tolerance and excellent stereoselectivity.

Conflicts of interest

There are no conflicts to declare.

Acknowledgment

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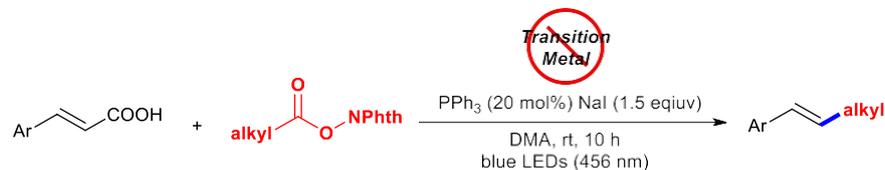
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Photocatalytic Dual Decarboxylative Alkenylation Mediated by Triphenylphosphine and Sodium Iodide

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● General: 28 examples, up to 95% yield

● Regio- and stereo-specificity using the NaI/PPh₃ catalysis

A new transition-metal-free photoredox decarboxylative alkylation of α,β -unsaturated carboxylic acids and alkyl

N-hydroxyphthalimide (NHP) esters by NaI/PPh₃ catalysis has been developed.