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Modular synthesis of 1,4-diketones through regioselective bis-acylation of olefins by merging NHC and photoredox catalysis

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ABSTRACT

Efficient and modular synthesis of structurally diverse 1,4-diketones from readily available building blocks represents an essential but challenging task in organic chemistry. Herein, we report a multi-component, regioselective bis-acylation of olefins by merging NHC organocatalysis and photoredox catalysis. With this protocol, a broad range of 1,4-diketones could be rapidly assembled using bench-stable feedstock materials. The robustness of this method was further evaluated by sensitivity screening, and good reproductivity was observed. Moreover, the diketone products could be readily converted into functionalized heterocycles, such as multi-substituted furan, pyrrole, and pyridazine. Mechanistic investigations shed light on the NHC and photoredox dual catalytic radical reaction mechanism.

As one of the fundamental goals of advanced synthesis and catalysis, developing efficient, economical, and modular synthesis of complex value-added molecules in an environmentally friendly manner has always attracted the attention of organic chemists [1–4]. Consequently, green catalytic strategies, including organocatalysis [5–15] and photoredox catalysis [16–18], have become the main pillars of modern organic synthesis. Although significant advancements have been achieved in each catalytic system, the combination of organocatalysis and photoredox catalysis in a synergetic way is still in its infancy, and its synthetic potential remains open-ended [19–26]. Therefore, merging organo- and photoredox catalysis to allow diversity-oriented synthesis of valuable molecules is highly desirable.

Ketones represent one of the most prevalent structural motifs in various bioactive natural products and pharmaceutical agents [27–31]. They also serve as necessary starting materials for assembling structurally complex molecules. In particular, the 1,4-diketones, frequently used for diverse cyclization reactions, have played a crucial role in advancing heterocyclic chemistry and thus accelerating the discovery of novel agrochemicals, drug molecules, and dye materials. (Scheme 1a) [32–35]. Traditional preparation methods of 1,4-diketones typically involve forming one chemical bond and connecting two functionalized keto-substrates. As illustrated in Scheme 1b, there are two main established strategies, namely as 2,3-disconnection and 3,4-disconnection pathway, which include the classical nucleophilic substitution reaction [36–38], oxidative cross-coupling of metal enolates [39–44], and Stetter-type addition [45–54], respectively. In recent years, radical bis-acylation of simple olefins, based on a more efficient 1,2- and 3,4-disconnection strategy, has emerged as a powerful tool for rapidly synthesizing 1,4-diketones. For instance, in 2020, Xia reported an elegant example of using 4-benzoyl-dihydropyridines (DHP) as bis-acylating reagents to react with styrene in the presence of nickel catalysts under visible light irradiation [55]. Soon afterward, Wu and Xiao's groups independently utilized α-keto acids and olefins to achieve the target bis-acylation reactions by using photoredox/N(n-Bu)$_4^+$ catalytic system and photoredox/chromium dual catalysis, respectively [56,57]. Later, Feng and co-workers disclosed that acyl fluorides were also suitable substrates for the bis-acylation process upon visible light-promoted NHC catalysis [58]. Our group has recently developed a conceptually-different oxidative radical NHC catalysis to achieve 1,4-diketone synthesis through bis-acylation of olefins by using various commercially available aldehydes as acylating substrates [59]. Despite this, all of the above works belong to the two-component reaction manifold, which could only allow the synthesis of symmetric 1,4-diketones (Scheme 1c, left). In par-

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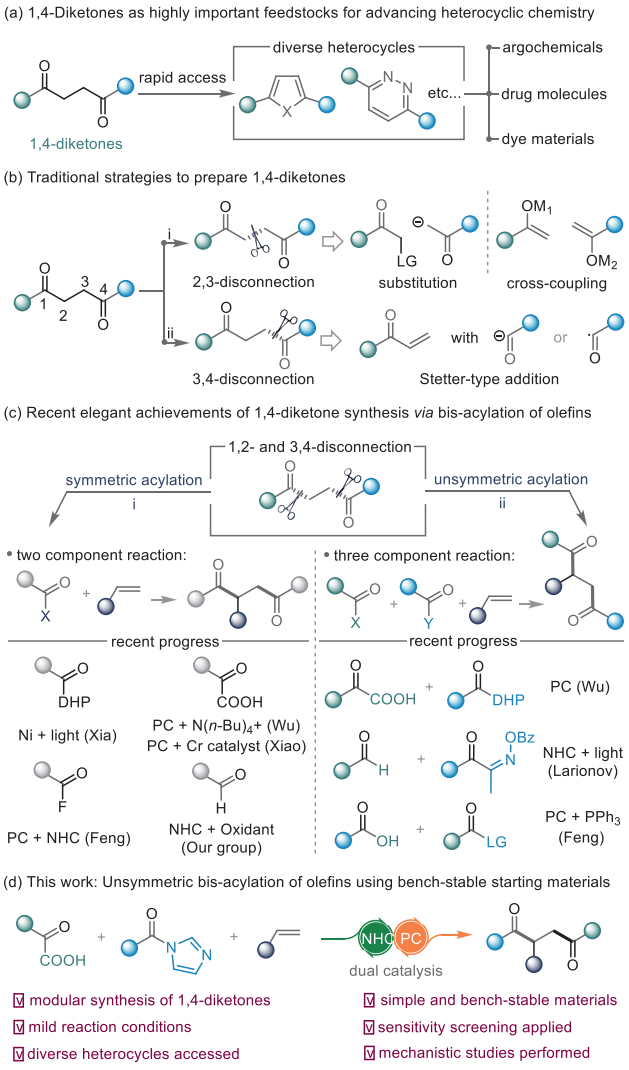
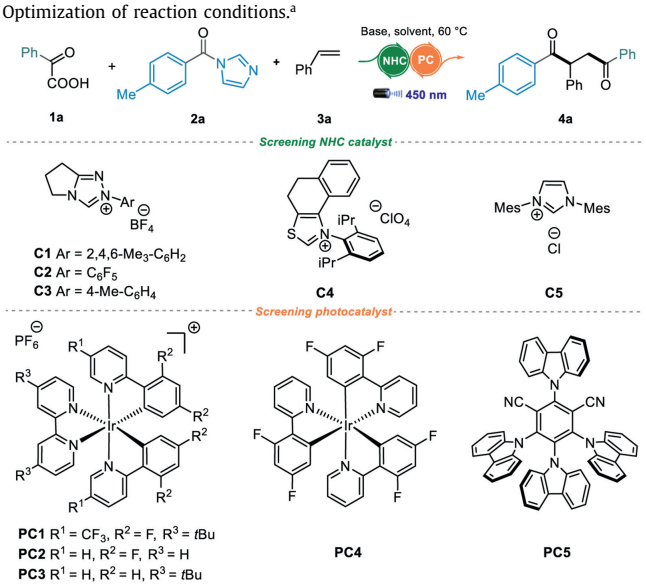


Scheme 1. Overview of the 1,4-diketone synthesis and our motivation of this study.

allel, the first example of an unsymmetric synthsis of 1,4-diketones was reported by Wu and co-workers, and they realized the three-component reactions of α-keto acids, 4-benzoyl-DHP, and simple styrene under photoredox catalysis [56]. After that, more catalytic synthesis methods were reported; for example, Larionov disclosed an elegant visible light-induced NHC-catalyzed radical coupling of aldehyde, oxime, and styrene to access a spectrum of 1,4-diketone products (Scheme 1c, right) [60]. Inspired by these advancements, we envisaged that the unsymmetric olefin bis-acylation process might be also achieved by merging NHC organocatalysis and photoredox catalysis [61,62]. As part of our continuing interests in NHC radical organocatalytic synthesis [6,63–72], we herein disclose the modular synthesis of 1,4-diketones through regioselective bis-acylation of olefins by using the bench-stable α-keto acids [27,73] and acyl imidazoles upon organo- and photoredox dual catalysis [74–76]. This protocol could offer a straightforward route to diketones featuring excellent functional group tolerance under mild conditions.

We commenced the investigations by selecting α-keto acid **1a**, acyl imidazole **2a**, and styrene **3a** as model substrates, using a blue light-emitting diode (LED) as the visible-light source. To our delight, the corresponding 1,4-diketone **4a** was smoothly isolated by using a triazolium salt catalyst **C1** in dichloromethane at 60 °C, albeit with poor yield (Table 1, entry 1). Next, several solvents

Table 1
Optimization of reaction conditions.[a]

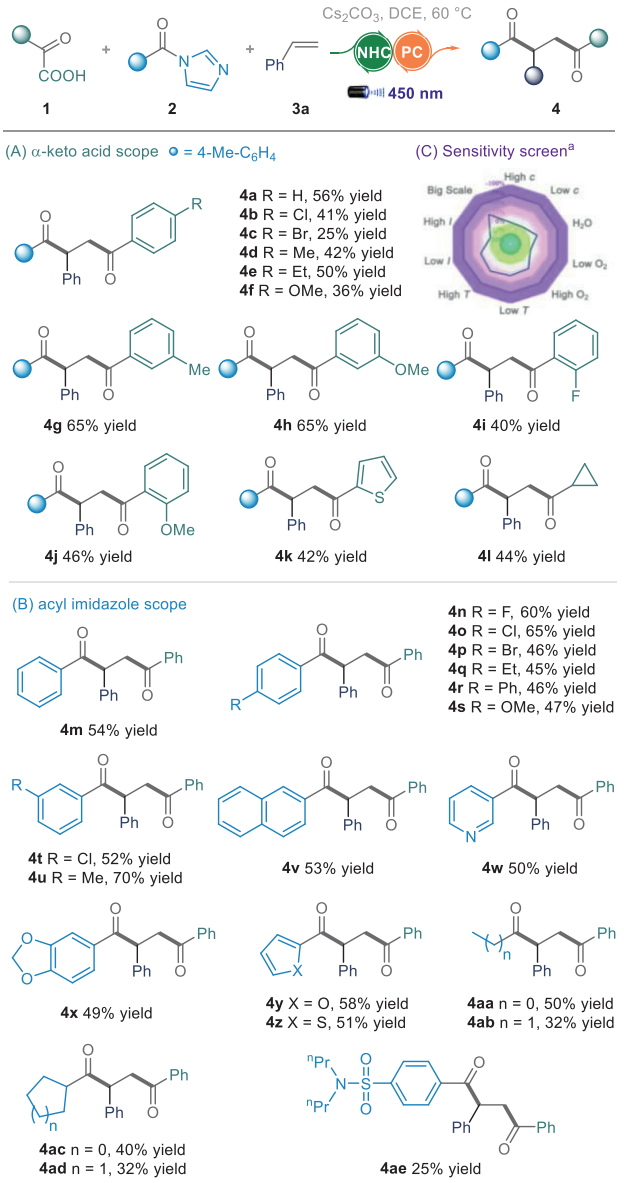


Entry	NHC	PC	Solvent	Base	Yield (%) [b]
1	**C1**	**PC1**	DCM	Cs_2CO_3	38
2	**C1**	**PC1**	Tol	Cs_2CO_3	14
3	**C1**	**PC1**	MeCN	Cs_2CO_3	41
4	**C1**	**PC1**	DCE	Cs_2CO_3	56
5	**C1**	**PC1**	DMF	Cs_2CO_3	25
6	**C2**	**PC1**	DCE	Cs_2CO_3	<5
7	**C3**	**PC1**	DCE	Cs_2CO_3	37
8	**C4**	**PC1**	DCE	Cs_2CO_3	N.R. [c]
9	**C5**	**PC1**	DCE	Cs_2CO_3	N.R. [c]
10	**C1**	**PC2**	DCE	Cs_2CO_3	20
11	**C1**	**PC3**	DCE	Cs_2CO_3	9
12	**C1**	**PC4**	DCE	Cs_2CO_3	N.R. [c]
13	**C1**	**PC5**	DCE	Cs_2CO_3	8
14	**C1**	**PC1**	DCE	DBU	23
15	**C1**	**PC1**	DCE	K_3PO_4	37
16	**C1**	**PC1**	DCE	CsF	21
17	**C1**	**PC1**	DCE	NaOAc	N.R. [c]
18 [d]	**C1**	**PC1**	DCE	Cs_2CO_3	31
19 [e]	**C1**	**PC1**	DCE	Cs_2CO_3	27
20 [f]	**C1**	**PC1**	DCE	Cs_2CO_3	31
21 [g]	**C1**	**PC1**	DCE	Cs_2CO_3	12
22 [h]	**C1**	**PC1**	DCE	Cs_2CO_3	23

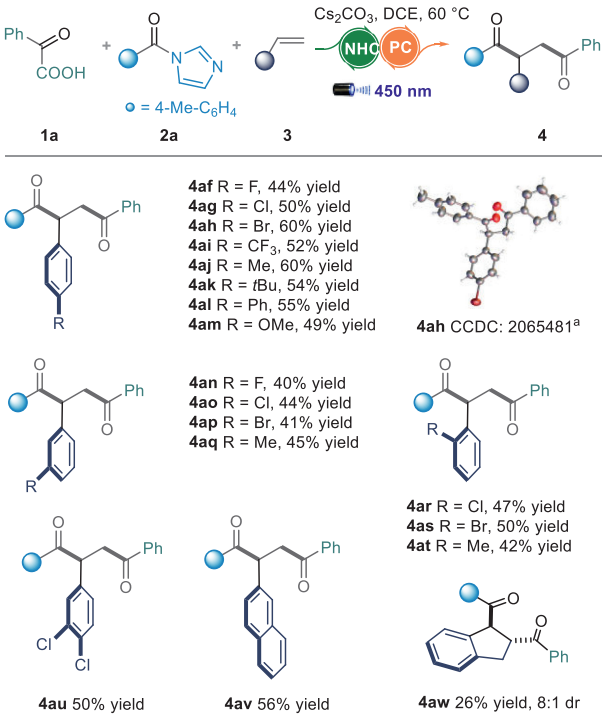
[a] Reactions condition: **1a** (0.10 mmol), **2a** (0.20 mmol), **3a** (0.20 mmol), base (0.12 mmol), PC (0.002 mmol) and NHC (0.02 mmol) in 1.0 mL solvent, irradiation with blue LEDs at 60 °C for 2 h.
[b] Isolated yield of **4a**.
[c] No reaction.
[d] With 2.2 mmol Cs_2CO_3.
[e] With 3.2 mmol Cs_2CO_3.
[f] With 0.001 mmol PC.
[g] **1a** (0.20 mmol), **2a** (0.10 mmol,), **3a** (0.20 mmol).
[h] **1a** (0.20 mmol), **2a** (0.20 mmol), **3a** (0.10 mmol).

were screened in the presence of NHC catalyst **C1**, photocatalyst **PC1**, and Cs_2CO_3 at 60 °C. As shown in entries 2–5, dichloroethane (DCE) provided the best result. Subsequently, various NHC catalysts, including triazolium, thiazolium, and imidazolium salts, were tested in this dual catalytic system, but poor results were generally observed (entries 6–9).

The catalytic efficiencies of triazolium salts **C2**–**C3** were unsatisfactory and delivered **4a** in poor yield (entries 6 and 7). Other catalysts cannot promote the radical target transformation (entries 8 and 9). Further screening of photocatalysts did not improve the reaction outcome (entries 10–13). Then, the effect of various inorganic and organic bases was investigated, but no better results were obtained (entries 14–17). Moreover, we screened the amount



Scheme 2. Substrate scope of α-keto acids **1** and acyl imidazoles **2** for the synthesis of 1,4-diones. Reactions condition: **1** (0.10 mmol), **2** (0.20 mmol), **3a** (0.20 mmol), Cs_2CO_3 (0.12 mmol), **PC1** (0.002 mmol) and NHC **C1** (0.02 mmol) in 1.0 mL DCE, irradiation with blue LEDs at 60 °C for 2 h. [a] *I*, intensity; *T*, temperature; Big scale, 2.0 mmol scale; *c*, concentration.



Scheme 3. Substrate scope of the styrenes **3** used for the synthesis of 1,4-diones. Reactions condition: **1a** (0.10 mmol), **2a** (0.20 mmol), **3** (0.20 mmol), Cs_2CO_3 (0.12 mmol), **PC1** (0.002 mmol) and NHC **C1** (0.02 mmol) in 1.0 mL DCE, irradiation with blue LEDs at 60 °C for 2 h. [a] The representative product **4ah** was determined by X-ray diffraction analysis, and other products were tentatively assigned by analogy.

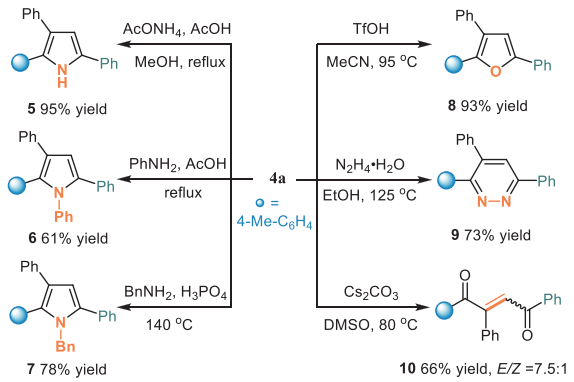
of base and photocatalyst (entries 18–20), and the substrate loading (entries 21 and 22). Unfortunately, the yield did not improve. Furthermore, we screened the substrates with different leaving groups, such as benzoyl fluoride, benzoic anhydride. However, no significant promotion in conversion was observed (for more details, see Supporting information).

With the optimized conditions in hand, we investigated the substrate scope by testing various α-keto acids **1** and acyl imidazoles **2** (see Supporting information for details). As exhibited in Scheme 2A, a range of α-keto acid substrates bearing either electron-deficient or electron-rich aryl substituents could afford the desired 1,4-diketones **4a–4j** in 25%–65% yields. The thienyl and cyclopropyl substituted α-keto acids were also suitable substrates for this target reaction (**4k–4l**).

In addition, the transformation also tolerated a broad range of acyl imidazoles **2** with various electronic and steric properties, offering the 1,4-diones in moderate yields (Scheme 2B). In general, the steric and electronic nature of substituents on the aryl ring did not significantly affect the reaction outcome (**4m–4u**). Furthermore, 2-naphthyl and heteroaryl acyl imidazoles were also suitable for the transformation to afford products **4v–4z,** respectively. Gratifyingly, a collection of the challenging alkyl acyl imidazoles also proved compatible with this catalytic system (**4aa–4ad**). Moreover, this organocatalytic system was successfully applied to the late-stage functionalization of acyl imidazole derived from probenecid, delivering the target product **4ae** in acceptable yields. Furthermore, we applied a condition-based sensitivity screening approach (Scheme 2C) [77], and this reaction exhibited medium sensitivity to water, oxygen, temperature, and light intensity. Hence, an appropriate performance of this reaction requires the assurance of suitable light intensity and an inert atmosphere.

Then, the reactions with various styrenes **3** were examined under the established conditions. As shown in Scheme 3, the styrenes bearing electron-withdrawing or electron-donating substituents at *para*- or *meta*-position could all participate in this reaction and offer the products **4af–4aq** in acceptable yields. Moreover, the structure of **4ah** was unambiguously confirmed by X-ray analysis. It is noteworthy that the *ortho*-substituted styrenes could smoothly participate in this reaction, affording the desired products respectively (**4ar–4at**). The disubstituted styrene could also participate in the reaction affording **4au** in moderate yield. In addition, the catalytic system was suitable for naphthyl substrate and afforded the corresponding product **4av**. The cyclic 1*H*-inene could also participate in this reaction, delivering product **4aw** in 26% yield with 8:1 diastereoselectivity.

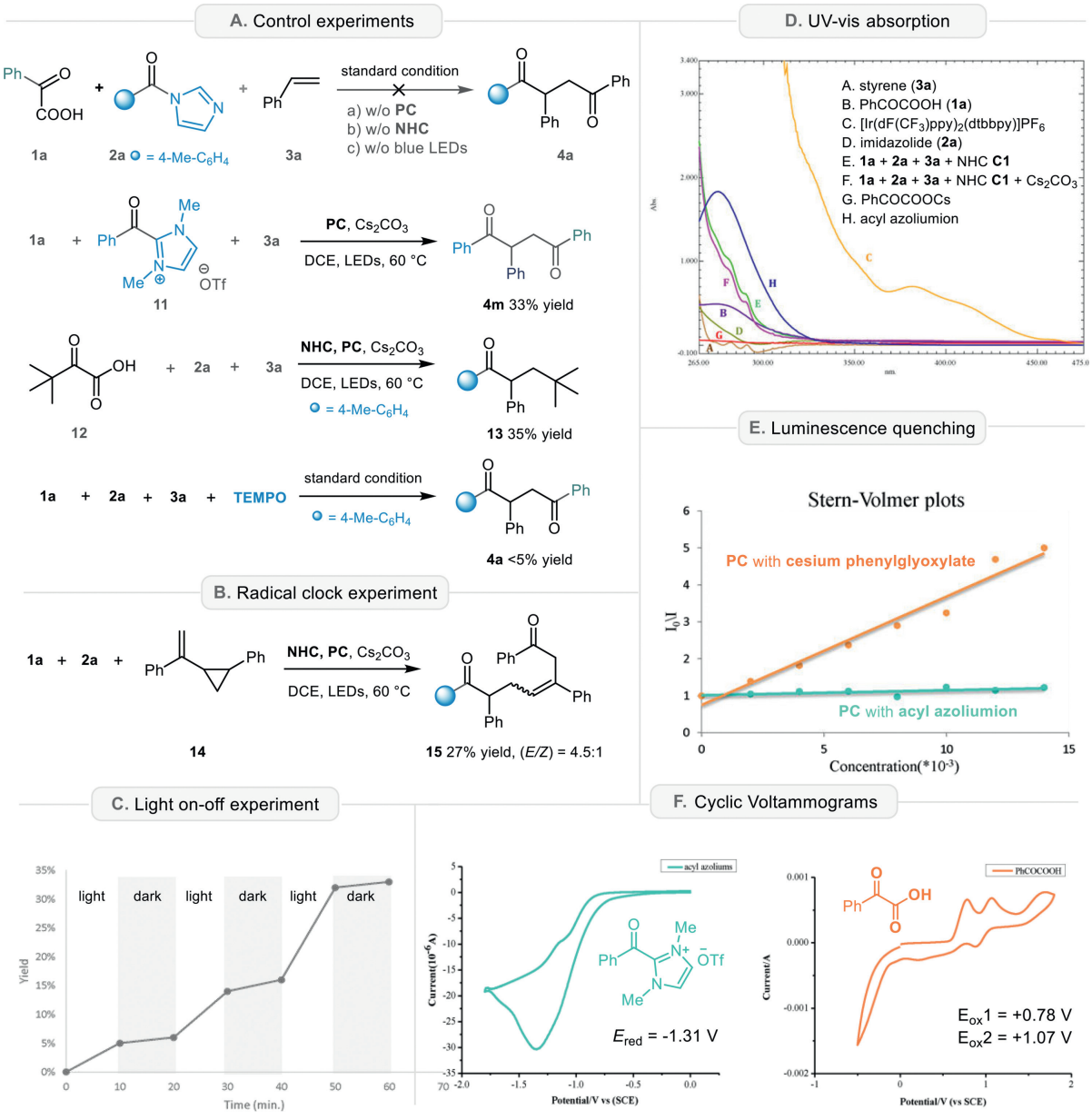
To showcase the synthetic value of these 1,4-diketones, we investigated various function group transformations. The products of five-membered heteroarenes **5–8** were obtained by treating the 1,4-diketone **4a** under acidic conditions. Treating **4a** with $N_2H_4{\cdot}H_2O$ and EtOH, a pyridazine **9** was obtained in good isolated



Scheme 4. Further synthetic applications.

yield. In addition, the oxidative dehydrogenation of **4a** can quickly occur with a catalytic amount of Cs_2CO_3, delivering product **10** as a mixture of 7.5:1 *trans/cis* isomers in moderate yield (Scheme 4).

In order to gain insights into the dual catalytic system, we performed a series of mechanistic investigations to elucidate the possible reaction mechanism (Scheme 5) [78–81]. First, control experiments indicated that blue LEDs, NHC, and PC were indispensable for this radical organocatalytic reaction. Then, in the presence of α-keto acid **1a** and styrene **3a**, the direct use of acyl azolium **11** could also provide desired product **4m** in 33% yield (Scheme 5A). This result indicated that the acyl azolium sepeices generated from an imidazolide and NHC is the reactive intermediate. Furthermore, when 3,3-dimethyl-2-oxobutanoic acid **12** was used, alkylative acylation product **13** was obtained (Scheme 5A). Moreover, the catalytic process could be completely suppressed by adding TEMPO under standard condition (Scheme 5A). Then, a radical-clock experiment with the vinylcyclopropane substrate **14** afforded ring-opening product **15** (Scheme 5B). These results indicated that the corresponding catalytic reaction might proceed through a radical process. Next, a light on-off experiment embodied the products only formed during the constant irradiation (Scheme 5C). This result indicated that the reaction underwent a photo-catalytic process.



Scheme 5. Experimental studies for mechanistic investigation.

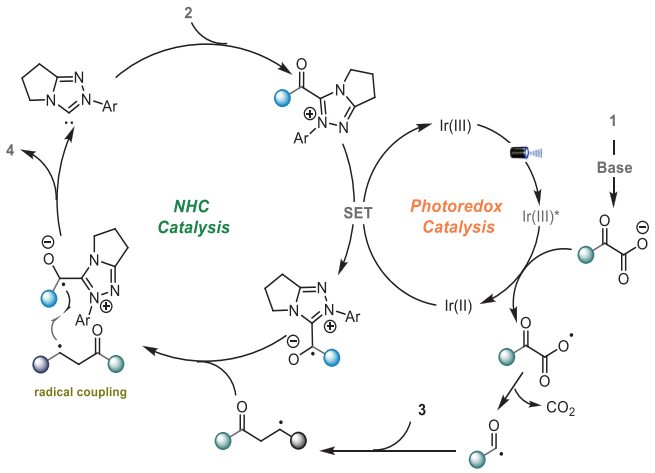


Fig. 1. Proposed reaction mechanism.

Moreover, there was a significant absorption of the photocatalyst $[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$ in the UV–vis absorption spectrum. By contrast, when the substrates, NHC catalyst, or their combination were tested, no absorption was observed in the spectrum of visible light (Scheme 5D). This result indicated that the visible light excitation of the photocatalyst initiated the photocatalytic process. Besides, we performed Stern-Volmer fluorescence quenching experiments. As illustrated in Scheme 5E, the excited photocatalyst **PC1** could be readily quenched by Cesium phenylglyoxylate rather than acyl azolium. According to the cyclic voltammetry experiments (Scheme 5F), we proposed a tentative mechanism in which the excited state of Ir(III)* photocatalyst ($E_{1/2}{}^{III*/II} = +1.21$ V *vs.* SCE) reacted with α-keto acid **1a** [$E_{ox}1$ ($PhCOCOO^-$) = + 0.78 V *vs.* SCE] to form the PhCOCOO· and Ir(II) photocatalyst. Then, the acyl azolium **11** ($E_{red} = -1.31$ V *vs.* SCE) oxidized Ir(II) ($E_{1/2}{}^{III/II} = -1.37$ V *vs.* SCE) to regenerate the ground state of Ir(III) photocatalyst.

Based on the above experimental studies, a plausible mechanism for the catalytic transformation is proposed in Fig. 1. Initially, the base-mediated deprotonation of α-keto acid **1a** provided α-oxocarboxylate. Meanwhile, the ground-state Ir(III) catalyst was excited as a single-electron oxidant Ir(III)* under blue LEDs irradiation. Then, the α-oxocarboxylate was oxidized through a single electron transfer, and give acyl radical species through decarboxylation. Subsequently, acyl radical added to styrene **3** to give a benzyl radical. Meanwhile, the ketyl radical and a ground-state Ir(III) catalyst was generated through the single-electron transfer from Ir(II) photocatalyst to acyl azolium species. Finally, the cross-coupling of these two radical intermediates afforded 1,4-diketone product and released the free carbene catalyst.

In summary, we have developed a multi-component, regioselective bis-acylation of olefins by merging NHC organocatalysis and photoredox catalysis. With the reported synthetic approach, a broad range of 1,4-diketones was easily prepared using readily accessible starting materials under mild conditions. Interestingly, the diketone products could be readily converted into multi-substituted furan, pyrrole, and pyridazine. Moreover, some mechanistic experiments shed light on understanding this NHC and photoredox dual catalytic radical process. Further investigations on the development of new chiral NHC catalysts and their applications in asymmetric radical transformations are underway in our laboratory, and the results will be reported in due course.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2023.108271.

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