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## COMMUNICATION

Received 00th January 20xx,<br/>Accepted 00th January 20xxα,β-unsaturated esters enables the polysubstituted γ-lactoneReceived 00th January 20xxformation

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Photoinduced umpolung addition of carbonyl compounds with

We herein report the photoinduced intermolecular umpolung addition of aromatic ketones/aldehydes with  $\alpha$ , $\beta$ -unsaturated esters via ketyl radical intermediates. Following an intramolecular transesterification, a variety of  $\gamma$ -lactone derivatives are readily accessed. Mechanistic investigations demonstrate the significant role of Hantzsch ester, which serves both as the electron and proton donor.

Ketyl radicals are versatile synthons for constructing plenty of functionalized alcohols, especially by ketyl-olefin coupling reactions.<sup>1</sup> However, conversion of carbonyl compounds to ketyl radicals is largely confined by their extremely negative redox potentials (e.g.  $E_{1/2}$  = - 2.11 V vs SCE for acetophenone).<sup>2</sup> Alkali,<sup>3</sup> alkaline earth,<sup>4</sup> and transition metals<sup>5</sup> are the most powerful reducing agents for converting carbonyl compounds to ketyl radicals. Among them,  $SmI_2$  ( $E_{1/2} = -0.89$  V vs SCE) is a frequently used one, which is typically combined with hexamethylphosphoramide (HMPA) for the sake of superior activities as the reduction potential of SmI<sub>2</sub> is dramatically enhanced by this Lewis-basic additive ( $E_{1/2}$  as low as - 1.79 vs SCE).<sup>6</sup> Additionally, the combination of tributyltin hydride (as a hydrogen radical source) and 2,2'-azobisisobutyronitrile (AIBN, as a radical initiator) at high temperatures is also commonly used to generate ketyl radicals from carbonyl compounds.7 Although these methods can effectively convert carbonyls to ketyl radicals, they still have some disadvantages, such as the use of stoichiometric or even excessive amounts of expensive reagents, or the use of chemicals that are extremely harmful to the environment and highly hazardous to health. To circumvent the high activation barrier issues and avoid the use of toxic or costly reducing agents, new procedures for generating ketyl radicals are highly desired. Recently, visiblelight triggered photoredox catalysis represents a unique

excited-state chemistry methodology, which not only enables the discovery of numerous nontraditional bond-forming strategies, but also obviates the need for tough reaction conditions and rigorous laboratory instruments.<sup>8</sup> Photoinduced ketyl radicals enjoy a surge of popularity, which could stem from carbonyl compounds<sup>9</sup> (while most of the existing successful photoinduced ketyl radical reactions mainly involve aldehydes<sup>10</sup> due to the relative high redox potentials), alcohols,<sup>11</sup> acetals<sup>12</sup> and ketals,<sup>13</sup> either catalyzed by Lewis acids or Brønsted acids,<sup>14</sup> getting rid of environmentally unfriendly, harsh and cumbersome reaction conditions.

The radical addition reaction between ketyl radicals and acrylates is one of the most effective methods for the synthesis of y-lactones.<sup>15</sup> Such reactions usually require high temperature to generate ketyl radicals, yet elevated temperatures can also accelerate the polymerization of acrylates, thus resulting in complex side reactions. In this context, the emergence of strategies to produce ketyl radicals by visible-light catalysis makes the synthetic conditions for ylactones mild, excluding the use of free-radical initiators and high temperatures. In 2013, Knowles and coworkers uncovered a new photochemical protocol for the intramolecular ketyl-olefin coupling to produce  $\gamma$ -lactone structure through concerted proton-coupled electron transfer (PCET) pathway (Scheme 1A).16 In 2015, Macmillan and colleagues accomplished the lactonization of diverse alcohols with methyl acrylates via a hydrogen atom transfer (HAT) mechanism (Scheme 1B).<sup>17</sup> Inspired by these outstanding A. Knowles's Photocatalytic Intramolecular Ketyl-Olefin Reductive Coupling





C. This Work: Photoinduced Intermolecular Reductive Coupling of Carbonyl Compounds with Alkenes



 $<sup>\</sup>mbox{Scheme 1}$  Photocatalyzed strategies for  $\gamma\mbox{-lactone}$  synthesis via ketyl radical intermediate.

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### COMMUNICATION

works, we developed a method for synthesizing polysubstituted  $\gamma$ -lactones through intermolecular umpolung addition of ketones with acrylates via photoinduced ketyl radicals (Scheme 1C).

We first investigated the coupling reaction between model substrate acetophenone (1) and  $\alpha$ , $\beta$ -unsaturated ester ethyl 2phenylacrylate (2) in the presence of 1 mol% [Ir(dF(CF<sub>3</sub>)ppy)<sub>2</sub>dtbbpy]PF<sub>6</sub> ([Ir]PF<sub>6</sub>) as the photoredox catalyst and Hantzsch ester (HE1) as the sacrificial reductant. The reaction proceeded smoothly to produce  $\gamma$ -hydroxy ester (3) in 42% yield with only a trace amount of  $\gamma$ -lactone (4) observed (Table 1, entry 1). After screening various factors affecting the reaction efficiency, the standard conditions were determined (Table 1, footnote a). Notably, screening of the additives showed that catalytic amount of Lewis acid like 10 mol%  $B(C_6F_5)_3$  could enhance the yield (Table 1, entry 2). This phenomenon might be attributed to the activation of ketone substrates by Lewis acid, as several previous researches reported.<sup>18</sup> Subsequent screening of different external reductants proved HE1 to be the best hydrogen source with an optimal loading of 2.0 equivalents (Table 1, entries 2-6). After full consumption of the initial reactants, direct addition of a Brønsted acid [1.0 equiv. p-toluenesulphonic acid (pTSA)] into the reaction system realized a nearly quantitative transesterification after stirring for another 24 hours, affording the anticipated y-lactone (4) in 97% yield (Table 1, entry 7; for more details, see ESI<sup>+</sup>, Table S1-S9).

Table 1 Selected optimization experiments<sup>a</sup>

$1 \qquad 2 \qquad 3 \qquad 4$	
$EtO \xrightarrow{Ph} OEt \xrightarrow{b} OPh O \\ HE1 HE2 HE3 TZ$	
Entry Variations from standard conditions $3^{b}$ (%) $4^{b}$	(%)
1 1.0 equiv. of <b>HE1</b> , no B(C <sub>6</sub> F <sub>5</sub> ) <sub>3</sub> 42 tra	ice
2 1.0 equiv. of <b>HE1</b> 67 tra	ice
3 1.0 equiv. of <b>HE2</b> 43 tra	ice
4 1.0 equiv. of <b>HE3</b> 41 tra	ice
5 1.0 equiv. of <b>TZ</b> 61 tra	ice
6 None 95 tra	ice
7 <sup>c</sup> None 0 97	

<sup>o</sup> Standard reaction conditions: a mixture of **1** (0.25 mmol), **2** (0.50 mmol), **HE1** (2.0 equiv.), B(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub> (10 mol%) and **[Ir]PF**<sub>6</sub> (1 mol%) in DMSO (2 mL) was irradiated with 9 W blue LEDs under argon atmosphere at room temperature for 36 hours. <sup>b</sup> Isolated yield. <sup>C</sup> After full consumption of initial reactants, *p*TSA was added and stirred for 24 hours without light irradiation.

Control experiments confirmed the crucial role of photocatalyst, Hantzsch ester, and visible-light because no reaction was observed when lacking any of these components. Besides, when the reaction was performed in air instead of an argon atmosphere, the expected reaction did not occur, indicating that  $O_2$  had an inhibitory effect on the reaction.

With the optimized conditions in hand, we next explored the substrate scope (Scheme 2). Ketones with both mono- and poly-substituted phenyl rings were first surveyed (5-16). Either electron-donating or electron-withdrawing groups were well-

### Journal Name

Page 2 of 6



**Scheme 2** Substrate scope<sup>*a*</sup> <sup>*a*</sup> Reaction conditions: ketone (0.25 mmol), alkene (0.50 mmol). <sup>*b*</sup> Acid catalyzed transesterification in CH<sub>2</sub>Cl<sub>2</sub> instead of DMSO for 24 hours (for details, see ESI<sup>+</sup>). <sup>*c*</sup> Without acid treatment. All yields are isolated yields.

tolerated under the reaction conditions. Sulfur and oxygen containing heteroaryl and other aryl ketones were proved to be competent substrates for the reaction (**17-20**) as well. In addition to abovementioned ketones, various alkyl phenyl ketones also performed well in this reaction, giving the corresponding products (**21-28**) in moderate to excellent yields. Alkyl substituted ketones with bulky structures were also successful candidates, while a small decline in yields was detected (**27** and **28**). Besides, derivatives from complex molecules, such as Nopol and Cholesterol, were feasible substrates as well (**29** and **30**). Subsequently, the scope of the alkene partner was tested (**31-42**). Olefins with aromatic rings Published on 06 October 2020. Downloaded by Universiteit Utrecht on 10/7/2020 12:32:55 PM

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functionalized with a diverse set of distinct substituents all worked well while the alkyl-substituted acrylate **38** was not, emphasizing the importance of the  $\alpha$ -aryl substitution to stabilize the vicinal free radicals. Olefins with other electron withdrawing groups, such as sulfone (**39**), nitrile (**40**) and multi-substituted unsaturated ketone (**41**) were also feasible substrates, providing the corresponding reductive coupling products with acetophenone. However, vinyl amide (**42**) seemed to be unreactive.

To further broaden the generality of this reductive coupling methodology, we selected several aromatic aldehydes as the potential coupling partners to react with ethyl 2-phenylacrylate **2** (Scheme 3). After screening the reaction conditions (see ESI<sup>+</sup>, **Table S10-S11**), we decided to perform this transformation under a mild metal-free conditions (Scheme 3, footnote a). As illustrated in Scheme 3, different aryl aldehydes all worked well, which exhibited a better reactivity compared with some previously reported reductive coupling reactions of aldehydes.<sup>19</sup>



Scheme 3 Substrate scope of aromatic aldehydes<sup>*a*</sup> <sup>*a*</sup> Reaction conditions: a mixture of aldehyde (0.25 mmol), **2** (0.50 mmol), **HE1** (1.5 equiv.) and 4CzIPN (2 mol%) in DMSO (2 mL) was irradiated with 9 W blue LEDs under argon atmosphere at room temperature for 18 hours. Acid catalyzed transesterification was all carried out in CH<sub>2</sub>Cl<sub>2</sub>. All yields are isolated yields. 4CzIPN = 2,4,5,6-tetrakis(carbazol-9-yl)-1,3-dicyanobenzene.

Unfortunately, dialkyl ketones and aliphatic aldehydes failed to undergo the reaction, presumably by virtue of their lower redox potentials (e.g.  $E_{1/2}$  = - 2.33 V vs SCE for cyclohexanone<sup>2</sup>, <sup>20</sup>), and aza-heterocyclic ketones showed no reactivity too. Only in cases where unsuitable alkenes employed, selfcoupling products of acetophenone were observed (unsuccessful substrates, see ESI<sup>+</sup>, 8). In order to get a better conversion efficiency, the transesterification process of some specific substrates proceeded in CH<sub>2</sub>Cl<sub>2</sub> instead of DMSO. Diastereomers of all products are separable by flash chromatography on silica gel except 27 and 28. The relative configuration of the two diastereomers of compound 4 was determined by 2D <sup>1</sup>H-<sup>13</sup>C HMQC and NOESY spectrum (see ESI<sup>+</sup>, Figure S17-S20). The structure of the two diastereomers (13a and 13b) of product 13 was confirmed by single crystal Xray diffraction analysis. The relative configuration of other products was assigned by analogy.

In order to obtain in-depth details of the reaction mechanism, further experiments were carried out (Scheme 4). Radical-trapping experiment was conducted under the

### conditions with radical scavenger Art2 206 hife standard tetramethylpiperidine-1-oxyl (TEMPO). ମନା: ୩୩୫୦୦୦ ଅନିକରେ ଅନ୍ତର୍ବ ଅନ୍ତର୍ବ ଅନ୍ତର୍ବ ଅନ୍ତର୍ବ ଅନ୍ତର୍ବ ଅନ୍ତର୍ବ ଅନ୍ତର୍ବ TEMPO, the reaction was completely inhibited, suggesting a radical pathway. Intriguingly, a cyclic compound (50) was isolated in 30% yield in the radical clock experiment (Scheme 4). We ascribed this observation to a [3+2] cycloaddition between cyclopropyl phenyl ketone 49 and the alkene partner 2 via a radical tandem reaction, which implied the existence of ketyl radical intermediate.<sup>21</sup> Typically, there are two possible radical termination pathways of the acrylate radical intermediate B (Scheme 5): (1) the hydrogen atom transfer (HAT) with Hantzsch ester radical cation;<sup>22</sup> and (2) a radical reduction/protonation sequential process.<sup>23</sup> Given that, we first utilized deuterium-labelled Hantzsch ester as the reductant to testify the feasibility of the HAT pathway. It was found that deuterium was not incorporated in the coupling product at all, indicating that a HAT process might be less prone to happen. Based on this result, a single electron transfer (SET) between the acrylate radical intermediate B and Hantzsch ester radical followed by protonation is, hence, highly possible.13 Moreover, Stern-Volmer fluorescence quenching experiments showed a strong interaction between the photocatalyst and Hantzsch ester, which suggested a SET process (see ESI<sup>+</sup>, 6.1-6.7 for more details).



On the basis of Knowles's work,<sup>24</sup> effective "BDFE" (bonddissociation free energy) of the Ir<sup>II</sup>/**HE1**<sup>+</sup> pair was calculated to be 17 kcal/mol, which is less than the 26 kcal/mol (O-H BDFE for acetophenone ketyl) threshold. Moreover, acid/base addition experiments<sup>9b, 9c</sup> demonstrated that basic compounds suppressed the reaction greatly, verifying the necessity of the Brønsted acidic species **HE1**<sup>+</sup> (see ESI<sup>+</sup>, **6.8** & **6.9**). Collectively, all these results suggested a PCET process.

Considering the above mechanistic studies and literature reports, a plausible mechanism was proposed (Scheme 5). Firstly, visible-light excitation of [Ir]PF<sub>6</sub> forms the active \*Ir<sup>III</sup> species  $[E_{1/2} (*Ir^{III}/Ir^{II}) = + 1.21 \text{ V vs SCE in MeCN}]^{25}$  which is efficiently quenched by Hantzsch ester  $[E_{1/2} (HE1/HE1^{+}) = +$ 0.85 V vs SCE in MeCN] and generates Hantzsch ester radical cation HE1<sup>++</sup>. Next, the resulting  $Ir^{II}$  species  $[E_{1/2}(Ir^{III}/Ir^{II}) = -1.37$ V vs SCE in MeCN]<sup>26</sup> surmounts the thermodynamic barriers and reduces the carbonyl compound (e.g.  $E_{1/2} = -2.11$  V vs SCE for acetophenone 1) to ketyl radical intermediate A via PCET with the activation of the radical cation **HE1**<sup>++</sup> and regenerates  $Ir^{III}$ .<sup>9e,27</sup> Here, the addition of Lewis acid  $B(C_6F_5)_3$  (LA) as described above can slightly improve the yield, which may be partly due to its activation effect on the ketone substrate. Afterwards, radical addition between ketyl radical **A** and  $\alpha$ , $\beta$ unsaturated ester 2 leads to radical intermediate B. Intermediate B is reduced by Hantzsch ester radical HE1<sup>•</sup> via SET, and then protonated to produce product 3, while Hantzsch ester radical HE1 is converted to oxidized Hantzsch ester OxHE1. Finally, product 3 undergoes an acid-catalyzed intramolecular transesterification to furnish the y-lactone product 4.



In summary, we have developed the first intermolecular reductive coupling reaction (umpolung addition) between diverse aromatic carbonyls and  $\alpha$ , $\beta$ -unsaturated esters for the synthesis of  $\gamma$ -lactones via a ketyl radical pathway. Hantzsch ester plays an imperative role in the activation of carbonyl compounds and functioned as the electron and proton donor to facilitate the key SET and PCET processes. The addition of *p*-toluenesulfonic acid successfully promoted the intramolecular transesterification of the coupling products, to afford a wide range of polysubstituted  $\gamma$ -lactones in good yields.

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### **Conflicts of interest**

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There are no conflicts to declare.

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# Table of contents entry

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Photoinduced reductive coupling of carbonyl compounds and  $\alpha$ , $\beta$ -unsaturated esters via ketyl radical intermediates for the synthesis of  $\gamma$ -lactones is described.