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Visible-light-induced ring-opening cross-coupling of cycloalcohols with vinylazaarenes and enones *via* β-C-C scission enabled by proton-coupled electron transfer

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ABSTRACT

Pyridyl-based ketones and 1,6-diketones are both attractive and invaluable scaffolds which play pivotal roles in the construction and structural modification of a plethora of synthetically paramount natural products, pharmaceuticals, organic materials and fine chemicals. In this context, we herein demonstrate an unprecedented, robust and generally applicable synthetically strategy to deliver these two crucial ketone frameworks *via* visible-light-induced ring-opening coupling reactions of cycloalcohols with vinylazaarenes and enones, respectively. A plausible mechanism involves the selective β-C-C bond cleavage of cycloalcohols enabled by proton-coupled electron transfer and ensuing Giese-type addition followed by single electron reduction and protonation. The synthetic methodology exhibits broad substrate scope, excellent functional group compatibility as well as operational simplicity and environmental friendliness.

The selective C–C bond cleavage has been considered as a unique and robust synthetic tool to streamline the construction of complex molecules *via* the rapid and intriguing skeleton reconstruction and functionalization [1–9]. Nevertheless, the inherent abundance and inertness of C–C bonds in organic molecules render the above task a formidable challenge. In this context, the selective β-C-C scission and functionalization of cycloalcohols *via* the formation of homoenolates [10–19] or keto radical [20–28] has emerged as a burgeoning and prosperous research area over the past few decades. In particular, the later cases involving the β-C-C fragmentation of *O*-centered alkoxy radical to give the distal alkyl radicals have gathered increasing attention among the chemical community [29–33]. Nevertheless, the formation of the alkoxy radical through the homolysis of the alcoholic O–H bond was challengeable owing to the relative high bond dissociation energy. It was not until recently that a number of significant breakthroughs were achieved by the groups of Lectka [34], Chen [23,35,36], Zhu [37–40], Knowles [41–43], Melchiorre [44], Rueping [45,46], Zuo [47–51], Lei [52] and many others [25,53–55] through photocatalytic manner. Noteworthily, the visible-light promoted proton-coupled electron transfer (PCET) by virtue of transferring an electron and proton in a synergistic step, has provided a competent protocol for the homolytic activation of O–H bonds of cycloalcohols under mild and redox-neutral conditions [56–59]. For instance, Knowles and coworkers pioneeringly demonstrated the photocatalytic isomerization reactions (Scheme 1a) [41,42] and an intramolecular ring-expansion [43] of cycloalcohols (Scheme 1b), respectively. As compared to the intramolecular ring-opening reactions of cycloalcohols, the intermolecular scenario has been considered as a more intriguing yet challengeable task. Very recently, Shen and coworkers elegantly reported the visible-light-induced cascade reaction of cyclopropanols with α-trifluoromethylated olefins to access fused *gem*–difluorooxetanes (Scheme 1c) [60]. Despite those aforementioned innovative and prominent advancements, the development of intermolecular ring-opening cross-coupling of cycloalcohols with distinctive olefins such as vinylazaarenes and enones bearing a multitude of crucial functionalities, trigged by PCET mechanism under environmentally benign and cost-effective conditions, remained an unmet and fascinating challenge.

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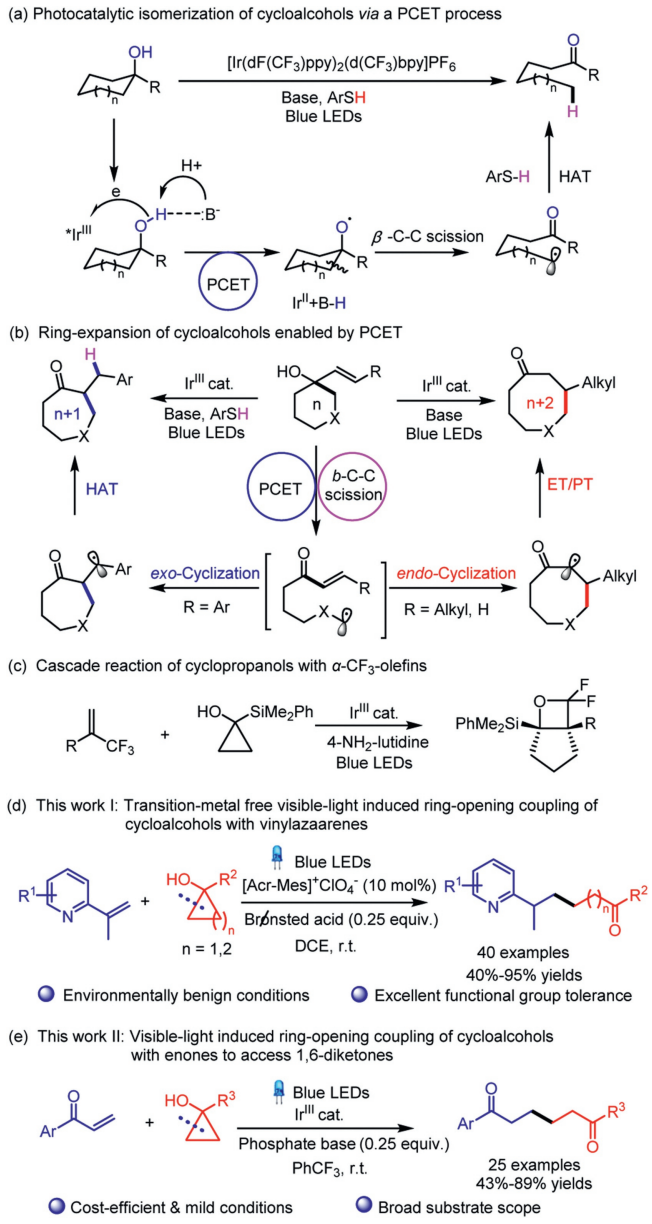


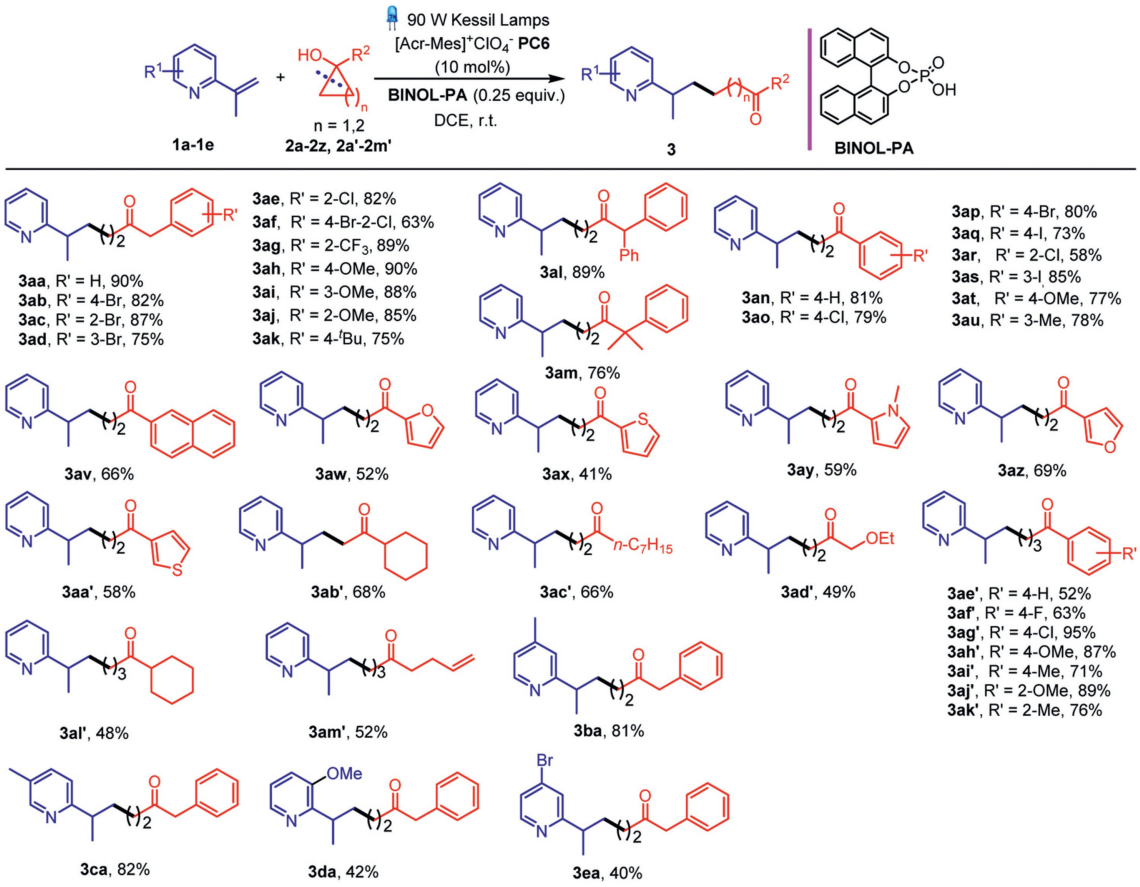
Scheme 1. Strategies for the C–C bond cleavage of cycloalcohols enabled by PCET.

Pyridine-derived scaffolds are widespread subunits in a multitude of natural products, pharmaceuticals, agrochemicals, ligands and functional materials [61–65]. Therefore, the structural modification of pyridine derivatives has been at the forefront of intense research among synthetic community [66–70]. Of note, pyridyl-based carbonyl motifs are of particular synthetic interest owing to their prevalence in myriad bioactive lead molecules. Consequently, the development of straightforward and generally applicable platforms to access such frameworks represents a practical and conceptually crucial pursuit with paramount synthetic value. Recently, Hong and coworkers have developed a regioselective cross-coupling of *N*-amidopyridinium salts with cyclopropanols to access β-carbonyl alkyl-substituted pyridines [66]. The group of Jiang has demonstrated a reductive coupling-enantioselective protonation of α-branched vinylketones with vinylpyridines, delivering pyridyl substituted ketones bearing remote stereogenic centers [67]. Enlightened by those seminal advancements, we herein disclosed a visible-light induced ring-opening cross-coupling of cycloalcohols with vinylpyridines to access a diverse range of δ- or ε-pyridyl substituted ketones with excellent functional group compatibility under environmentally benign metal-free conditions (Scheme 1d).

1,6-Diketones served as pivotal building blocks in the construction of various bioactive carbocyclic and heterocyclic compounds [71–75]. Moreover, the versatile utilities of functionalized 1,6-diketones in a multitude of organic transformations have leveraged their practical synthesis as a highly desired topic of interest. Particularly, unsymmetrical 1,6-diketones are recognized as more appealing yet challenging synthetic targets in contrast to their symmetrical analogues [76–79]. The representative state-of-the-art synthetic methods in this arena involved the C–C bond forming reactions of electrophilic enones with metal-homoenolates [80,81] or β-keto radicals [82–86] derived from siloxycyclopropanes or cyclopropanols. Nevertheless, those methodologies posed a handful of challenges arising from the employment of toxic heavy metals [83,84] or radical initiators [85] as well as the inevitable homocoupling of enones. As such, the quest for catalytic and highly selective protocols for the synthesis of structural diverse unsymmetrical 1,6-diketones under environmentally benign reaction conditions is of inarguably importance. Recently, we have developed a manganese-catalyzed radical mediated methodology to attain various unsymmetrical 1,6-diketones, albeit relative harsh conditions were required [82]. Encouraged by this achievement, we herein demonstrated a photocatalytic ring-opening cross-coupling of cycloalcohols with enones, providing a significant complement to the synthesis functionalized 1,6-diketones with broad substrate scope under mild conditions (Scheme 1e).

We commenced our investigation by screening the optimal reaction conditions using α-methylated 2-vinylpyridine **1a** and 1-benzylcyclopropan-1-ol **2a** as the model substrate (Table S1 in Supporting information for details). To our delight, the transformation proceeded smoothly in the presence of [Ir(dF(CF_3)ppy)$_2$(dtbbpy)](PF_6) (**PC1**) as a photocatalyst and BINOL phosphoric acid **(BINOL-PA)** as an additive using (trifluoromethyl)benzene as the solvent under the irradiation of 90 W Kessil Lamps, furnishing desired product **3aa** in 58% yield (Table S1, entry 1). Other photocatalysts including [Ir(dtbbpy)(ppy)$_2$](PF_6) (**PC2**), *fac*-Ir(ppy)$_3$ (**PC3**) and Eosin Y (**PC4**) gave inferior results attributed to the lower oxidative potentials in comparison with **PC1** (Table S1, entries 2–4). Gratifyingly, transition-metal free photosensitizers such as 4CzIPN (**PC5**) and acridinium salt [Acr-Mes]$^+ClO_4^-$ (**PC6**) with higher oxidizing abilities proved to be efficacious photocatalysts, furnishing **3aa** in 63% and 68% yields, respectively (Table S1, entries 5 and 6). A screening of solvents showed that 1,2-dichloroethane (DCE) was the optimal choice, giving **3aa** in 75% yield (Table S1, entry 14 *vs.* entries 7–13). Further optimization by increasing the loading of **PC6** to 10 mol% resulted a significant improvement to the reaction efficiency, forging **3aa** in 87% yield (Table S1, entry 15). Adjusting the reaction concentration to 0.033 M gave the optimal yield of 90% (Table S1, entry 16). Notably, a gradient decrease of the product yields was observed upon lowering the power of the light source (Table S1, entries 17 and 18). Control experiments showed that a dramatic attenuation of the reaction efficiency was observed without the aid of phosphoric acid (Table S1, entry 19), and the presence of the photocatalyst and irradiation proved to be indispensable for the transformation (Table S1, entries 20 and 21).

Taking advantage of the optimal reaction conditions, we investigated the substrate scope of the cross-coupling reaction of vinylazaarenes with cycloalcohols, as summarized in Scheme 2. A range of benzyl-substituted cyclopropanols were examined, it was observed that a plethora of electron-withdrawing halides on the benzene ring were well tolerated, affording **3ab-3af** in 63%–87% yields. Biologically crucial -CF_3 moiety was amenable, giving **3ag** in 89% yield. Substrates bearing electron-donating groups such as -OMe and -^{t}Bu were viable in the transformation, delivering **3ah-**

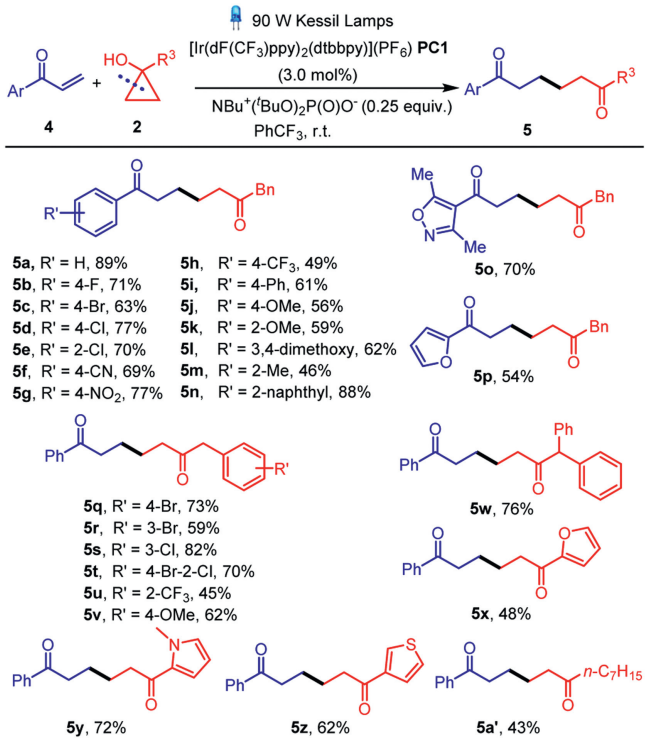


Scheme 2. Substrate scope of the ring-opening coupling reaction of cycloalcohols with vinylazaarenes. Reaction conditions: **1a-1e** (0.2 mmol), **2a-2z**, **2a'–2m'** (0.6 mmol), [Acr-Mes]$^+$$ClO_4^-$ (**PC6**) (0.02 mmol), **BINOL-PA** (0.05 mmol), DCE (6.0 mL), 90 W Kessil Lamps. Isolated yields.

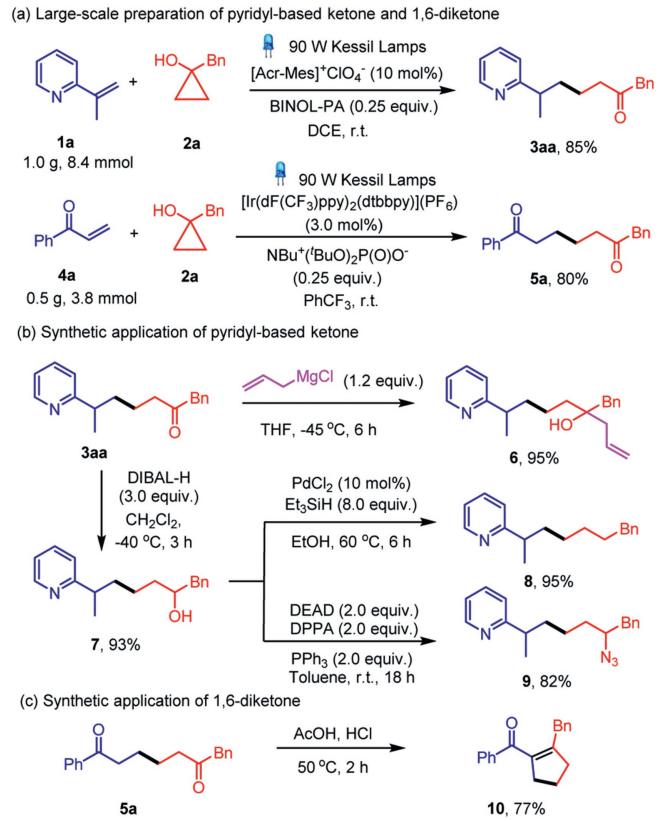
3ak in 75%–90% yields. Steric effect on the phenyl ring of benzyl group did not affect the above transformation, *para*- (**2b**, **2h**, **2k**), *orthro*- (**2c**, **2e**, **2g**, **2j**) and *meta*- (**2d**, **2i**) substituted cyclopropanols as well as disubstituted substrate **2f** all reacted with **1a** smoothly. Substituents such as phenyl and dimethyl on the benzylic position did not attenuate the reaction efficiency, forging **3al** and **3am** in 89% and 76% yields, respectively. Next, it was observed that a range of aryl-substituted cycloalcohols coupled with **1a** smoothly, regardless of their electronic effects or steric patterns, delivering **3an**-**3av** in 58%–85% yields. Heterocyclic substituents including 2-furyl and 2-thienyl were compatible with the standard conditions, forging **3aw** and **3ax** in moderate yields. *N*-Methylpyrrole substituted cyclopropanol also partook in the transformation uneventfully, delivering **3ay** in 59% yield. 3-Furyl and 3-thienyl derived substrates also reacted with **1a** efficiently, forging desired products **3az** and **3aa'** in 69% and 58% yields, respectively. Gratifyingly, cyclopropanols bearing aliphatic substituents such as cyclohexyl, fatty *n*-heptyl and ethoxymethyl were all viable in the ring-opening coupling reaction with **1a**, furnishing **3ab'**, **3ac'** and **3ad'** in 68%, 66% and 49% yields, respectively. To our delight, aside from cyclopropanols, a diverse array of aryl-substituted cyclobutanols bearing substituents with various electronic peculiarities and steric effects proved to be eligible coupling partners of **1a**, delivering **3ae'**–**3ak'** in 52%–95% yields. Cyclobutanols bearing cyclohexyl and unsaturated alkene moieties partook in the reaction smoothly, forging **3al'** and **3am'** in 48% and 52% yields. Next, we examined the substrate scope of 2-vinylpyridines. It was observed that 4-methyl and 3-methyl pyridyl substituted substrates **2b** and **2c** coupled with cyclopropanol **2a** uneventfully, giving rise to **3ba** and **3ca** in 81% and 82% yields. Electron-donating 3-OMe and electron-withdrawing 4-Br substituents on the pyridine ring decrease the reaction efficiencies, furnishing desired products **3da** and **3ea** in moderate yields.

Encouraged by the above conspicuous results, we envisaged that the synthetic strategy could be applied to the ring-opening coupling of cycloalcohols with various enones to access a plethora of 1,6-diketones. Thus, we commenced to screen the optimal conditions to facilitate the transformation (Table S2 in Supporting information for details). Unfortunately, the reaction of 1-phenylprop-2-en-1-one **4a** with cyclopropanol **2a** under the standard conditions in Scheme 2 proved to be futile, giving the desired product **5a** in only 5% yield (Table S2, entry 1). Gratifyingly, replacing the phosphoric acid **BINOL-PA** to a Brønsted base tetrabutylammonium di–*tert*–butyl phosphate NBu_4^+(^{t}BuO)POO^- provided **5a** in 43% yield (Table S2, entry 2). While other photocatalysts did not result in significant increase of the reaction efficiency (Table S2, entries 3–6), it was delightful that **5a** could be furnished efficiently in 74% yield upon the employment of [Ir(dF(CF_3)ppy)$_2$(dtbbpy)](PF_6) (**PC1**) (Table S2, entry 7). The screening of solvents proved that (trifluoromethyl)benzene was the optimal choice, forging **5a** in 89% yield (Table S2, entry 12). Decreasing the amount of **2a** led to lower product yield (Table S2, entry 12 *vs.* entries 13 and 14). The reaction efficiency was attenuated when decreasing the power of the light sources (Table S2, entry 12 *vs.* entries 15 and 16). Control experiments showed that no **5a** was formed in the absence of either the photocatalyst, the Brønsted base or the light irradiation (Table S2, entries 17–19).

With the optimal reaction conditions in hand, we explored the substrate scope of enones and cyclopropanols, as demonstrated in Scheme 3. A series of enones bearing electron-withdrawing



Scheme 3. Substrate scope of the ring-opening coupling reaction of cycloalcohols with enones. Reaction conditions: **4** (0.2 mmol), **2** (1.0 mmol), $[Ir(dF(CF_3)ppy)_2(dtbbpy)](PF_6)$ (**PC1**) (0.006 mmol), $NBu_4^+(^tBuO)_2POO^-$ (0.05 mmol), $PhCF_3$ (4.0 mL), 90 W Kessil Lamps. Isolated yields.

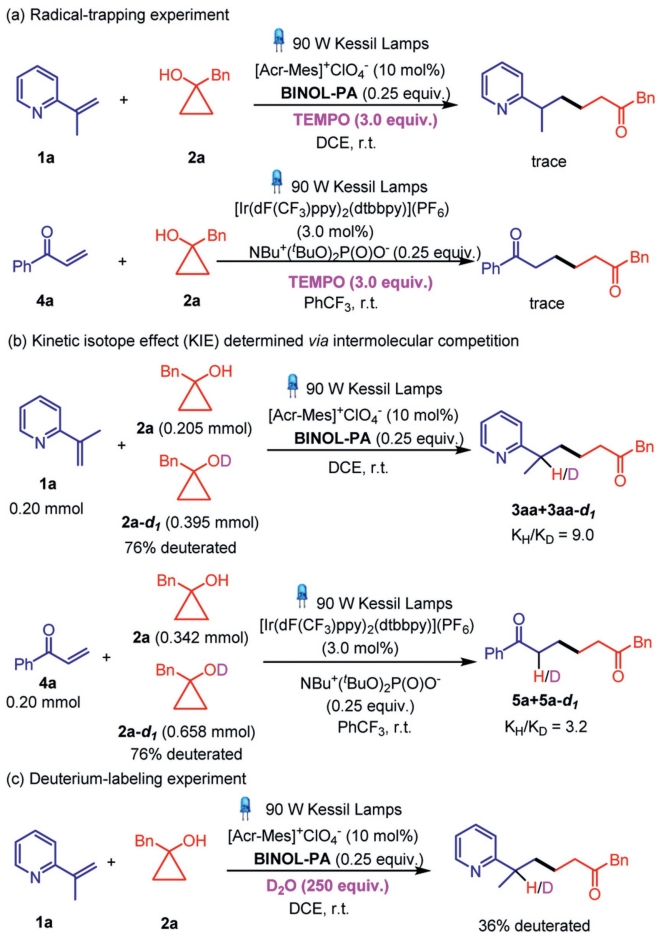


Scheme 4. Large-scale ring-opening coupling reactions and their synthetic applications.

halides, -CN, $-NO_2$, $-CF_3$ substituents on the phenyl ring were well tolerated, generating **5b-5h** in 49%–77% yields. Enones bearing electron-donating groups were also amenable to couple with **2a**, generating **5i-5m** in satisfying yields. Sterically more hindered 2-naphthyl substituent enone proved to be eligible in the transformation, giving **5n** in 88% yield. Delightfully, heterocycle substituted enones bearing 3,5-dimethylisoxazole and furan skeletons coupled with **2a** uneventfully, furnishing **5o** and **5p** in 70% and 54% yields, respectively. The scope of cyclopropanols were proved to be broad as well. Benzyl-substituted cyclopropanols bearing electron-withdrawing groups coupled with **4a** smoothly, forging **5q-5u** in moderate to good yields. The presence of electron-donating –OMe group on the phenyl ring or a –pH at the benzylic position were also viable, furnishing **5v** and **5w** in 62% and 76% yields. Heterocycle-substituted cycloalcohols bearing 2-furyl, *N*-methyl-2-pyrrolyl and 3-thienyl all showed excellent compatibilities with the standard conditions, furnishing **5x-5z** in moderate to good yields. Notably, aliphatic *n*-heptyl substituted cyclopropanol proved to be eligible coupling partner of **4a**, giving rise to **5a'** in 43% yield.

Gratifyingly, the large-scale preparations of pyridyl-based ketone **3aa** and 1,6-diketone **5a** proved to be successful without losing the reaction efficiencies (Scheme 4a). Subsequently, a handful of further transformation of **3aa** and **5a** were carried out to illustrate the synthetic utilities of the aforementioned ring-opening coupling reactions. **3aa** could be converted to synthetically useful allylic alcohol **6** in 95% yield. The carbonyl moiety could also be reduced by DIBAL-H to give pyridyl-based tertiary alcohol **7**, the hydroxyl group could be reduced in the presence of $PdCl_2$ and Et_3SiH to give pyridine derivative **8** in 95% yield. More importantly, the biologically and synthetically prominent pyridyl-containing azide **9** could be prepared efficiently through the Mitsunobu-type S_N2 reaction of **7** (Scheme 4b). The synthetic practicality of the 1,6-diketone **5a** could be highlighted through intramolecular aldol condensation, furnishing cyclic enone **10** in 77% yield (Scheme 4c).

Preliminary mechanistic studies were conducted to elucidate the plausible pathway for the aforementioned ring-opening coupling reactions. It was observed that the reactions of either vinylpyridine **1a** or enone **4a** with cyclopropanol **2a** was hampered in the presence of a radical scavenger 2,2,6,6-tetramethylpiperidine 1-oxyl (TEMPO), along with the interception of the β-keto radical species (see Supporting information for details), implying a plausible radical pathway (Scheme 5a). The kinetic isotope effect (KIE) experiments for the ring-opening coupling reactions of cyclopropanol **2a** and deuterium-labeled **2a-d$_1$** with **1a** or **4a** were conducted, the KIE values were calculated to be 9.0 and 3.2, indicating that the cleavage of the O–H bond in cycloalcohols was rate-determining in both transformations (Scheme 5b) (see Supporting information for details). It was observed that in the presence of external deuterium source D_2O, 36% deuterated **3aa-d$_1$** was isolated *via* the reaction of **1a** with **2a**, suggesting that the C–H bond formation might be attributed to the formation of a radical at the α-position of azaarenes and the ensuing SET reduction and protonation (Scheme 5c). The quantum yields for the ring-opening coupling reaction of cyclopropanol **2a** with **1a** and **4a** were calculated to be 0.36 and 0.25, respectively, implying that both transformations proceeded through a photoredox-catalyzed pathway rather than radical-chain mechanism (see Supporting information for details). The Stern-Volmer experiment for the reaction of **1a** with **2a** showed that a linear quenching was only observed in the presence of both **PC6** and **BINOL-PA**. Similar result was obtained in the reaction of enone **4a** with **2a**, wherein the presence of **PC1** and $NBu_4^+(^tBuO)POO^-$ were both indispensable for the linear quenching. The above observations evidentially supported the PCET process in the ring-opening coupling transformations (see Supporting information for details).



Scheme 5. Preliminary mechanistic studies.

Based on the aforementioned experimental results, we postulated plausible reaction pathways for the ring-opening coupling reactions (Scheme 6). For the ring-opening coupling reaction of vinylpyridines with cycloalcohols (Scheme 6a), the photocatalyst $[Acr\text{-}Mes]^+ClO_4^-$ absorbs a photon under the irradiation of blue LEDs to generate a highly oxidative singlet excited state $^*[Acr\text{-}Mes]^+ClO_4^-$, which reacts with the phosphate and the cycloalcohols **2** through a concerted PCET process to furnish an alkoxyl radical species **A**. Then, the β-C-C scission occurs to forge the distal β- or γ-keto alkyl radical **B**, which would be intercepted by 2-vinylpyridine through a Giese-type radical addition to furnish a tertiary radical **C**. Subsequently, a single electron reduction of **C** generates an anion **D**. Finally, the protonation of **D** gives rise to the desired product **3**. The reaction of cyclopropanols with enones was postulated to proceed *via* a similar mechanism as depicted in Scheme 6b, involving the formation of alkoxyl radical species **A** *via* a PCET process in the presence of excited $^*Ir(III)$-complex and a Brønsted base $NBu_4^+(^tBuO)POO^-$, followed by the β-C-C scission to access radical **B** and its ensuing interception by enones to generate radical **E**, the formation of anion **F** *via* SET, and its protonation to forge 1,6-diketones.

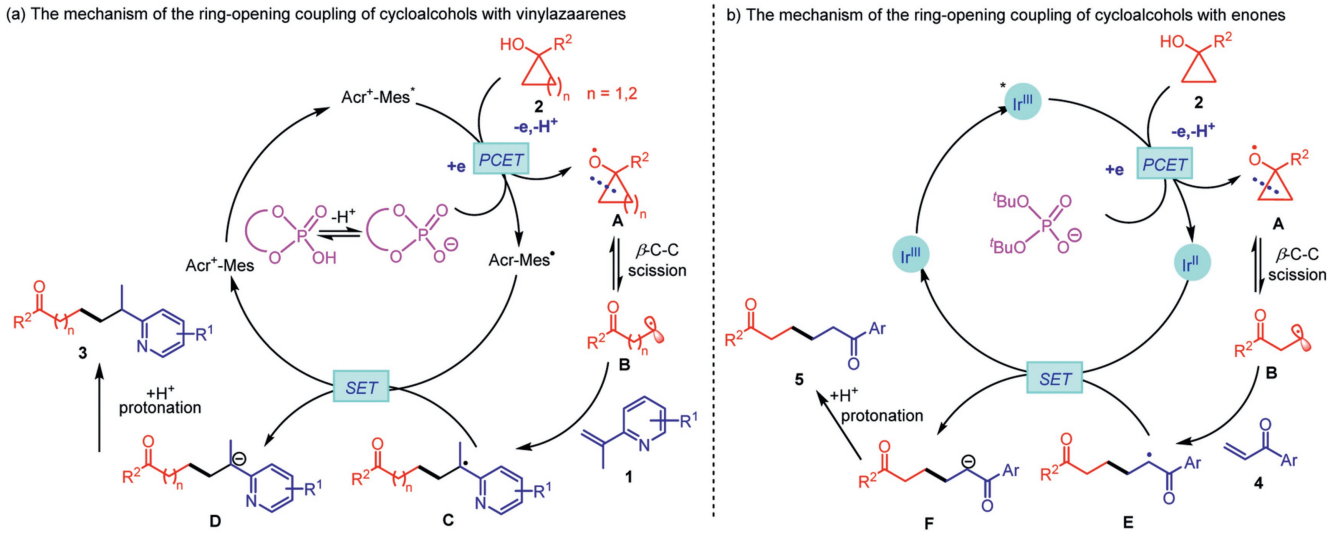
In summary, we have demonstrated unprecedented visible-light-induced intermolecular ring-opening cross-coupling reactions of cycloalcohols with vinylazaarenes or enones. The photoinduced proton-coupled electron transfer and the following β-C-C cleavage played key roles for the formation of the key distal keto alkyl radicals. The operational ease, excellent functional group tolerance and environmentally and mild reaction conditions of those transformations have paved a new avenue for the facile and efficient synthesis of pharmaceutically and synthetically prominent pyridyl-based ketones or 1,6-diketones and their related derivatives.

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.

CRediT authorship contribution statement

Qinghong Zhang: Methodology, Investigation, Formal analysis. **Qiao Zhao:** Methodology, Investigation, Formal analysis, Data curation. **Xiaodi Wu:** Investigation, Formal analysis, Data curation. **Li Wang:** Investigation, Data curation. **Kairui Shen:** Formal analysis, Data curation. **Yuchen Hua:** Formal analysis, Data curation. **Cheng Gao:** Formal analysis, Data curation. **Yu Zhang:** Methodology, Formal analysis. **Mei Peng:** Formal analysis, Data curation. **Kai Zhao:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Investigation, Conceptualization.



Scheme 6. The plausible mechanisms of the ring-opening coupling reactions enabled by PCET.

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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.2024.110167.

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