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Photocatalytic remote alkenylation of hydroxamides with enamides: synthesis of trisubstituted enamides *via* a radical pathway

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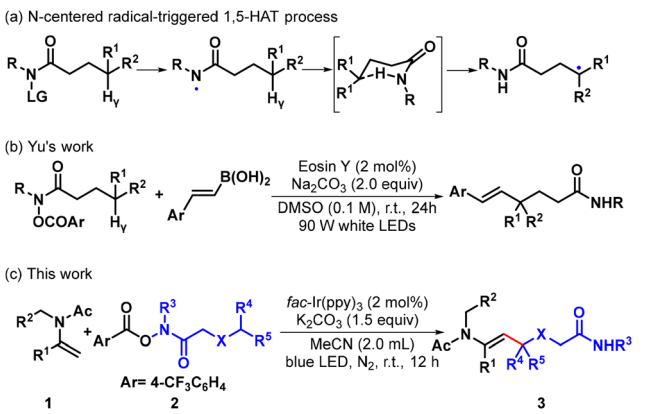
A photocatalytic radical-mediated strategy has been developed for the 1,5-hydrogen atom transfer (1,5-HAT)/alkenylation of hydroxamides with enamides, enabling the efficient synthesis of diverse, previously unreported trisubstituted enamides in moderate to good yields under mild conditions. This *N*-centered radical-triggered γ-C(sp^3)–H alkenylation reaction demonstrates broad substrate scope, excellent functional group tolerance, and remarkable regio- and stereoselectivity. The proposed mechanism involves a sequential cascade of single-electron transfer (SET), 1,5-HAT, radical addition, oxidation, and deprotonation.

The remote direct functionalization of unactivated C(sp^3)–H bonds offers distinct benefits in atom- and step-economy for the rapid synthesis of complex organic molecules.[1] Nevertheless, the high bond dissociation energy (ranging from 90 to 105 kcal mol^{-1}) and the inherently low reactivity of these inert C(sp^3)–H bonds present formidable obstacles to achieving effective C(sp^3)–H functionalization reactions.[2] Among the array of synthetic methods available to chemists, radical-mediated hydrogen atom transfer (HAT) initiated by *N*-centered radical precursors has attracted considerable interest due to its remarkable regioselectivity and chemoselectivity in functionalizing remote inert C(sp^3)–H bonds.[3] A classic example of HAT, 1,5-HAT, dates back to the late 19th century with the Hofmann–Löffler–Freytag reaction.[4] In this reaction, a pivotal carbon-centered radical is ultimately generated through the 1,5-hydrogen atom abstraction by a nitrogen-centered radical (Scheme 1a). After years of dedicated research by chemists, this reaction mode has made significant progress in constructing C(sp^3)–C(sp^3), C(sp^3)–C(sp^2) and C(sp^3)–C(sp) bonds.[5] However, the construction of C(sp^3)–C(sp^2) bonds through the coupling of C(sp^2)–H bonds in alkenes is still rare. In 2018, Yu and co-workers successfully developed a photocatalyzed alkenylation of hydroxamides with alkenylboronic acids (Scheme 1b).[6] Nonetheless, it remains valuable to explore the 1,5-HAT strategy for constructing C(sp^3)–C(sp^2) bonds using simple olefins, thereby circumventing the need to prepare precursors and enabling the efficient synthesis of high-value olefin compounds.

Trisubstituted enamides hold immense potential for widespread applications in medicinal chemistry, natural product research, and materials chemistry.[7] Consequently, the preparation and modification of trisubstituted enamides have been a focal point of chemists' endeavors. In recent years, researchers have continued to validate that using enamides as starting materials and conducting photo-, metal-, or electro-catalytic radical-induced β-C–H functionalization provides a reliable and efficient route for synthesizing these compounds. Thanks to the relentless efforts of numerous researchers, a variety of strategies, including alkylation,[8] acylation,[9] sulfonylation,[10] sulfonamination,[11] silylation,[12] thiocyanation,[13] trifluoromethylthiolation,[14] and bromination,[15] have been developed. Recently, our research group reported the difluoroamidoalkylation and difluoroamidosulfonylation of enamides using



Scheme 1 Synthetic approaches toward trisubstituted enamides.

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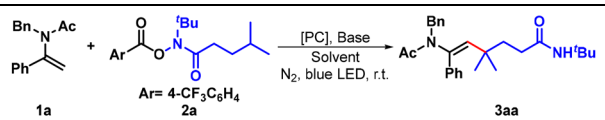
N-allylbromodifluoroacetamides, resulting in the formation of trisubstituted enamides containing a 3,3-difluoro-γ-lactam moiety.[16] Nevertheless, it remains of great significance to further explore novel reaction modes involving enamides to expand the functionalization diversity of trisubstituted enamides. Building on these research backgrounds and as a continuation of our ongoing efforts in the field of radical transformation,[17] we are delighted to report herein a photocatalytic method for remote alkenylation *via* the 1,5-HAT process (Scheme 1c). This approach utilizes enamides **1** and hydroxamides **2** as starting materials, enabling the synthesis of a variety of (*E*)-configured enamides **3** containing bisamide moieties with good yields. Notably, this photocatalytic strategy offers significant advantages, particularly in achieving complete regioselectivity and stereoselectivity.

Initially, the reaction between *N*-benzyl-*N*-(1-phenylvinyl) acetamide (**1a**) and *N*-(*tert*-butyl)-4-methyl-*N*-((4-(trifluoromethyl)benzoyl)oxy)pentanamide (**2a**) was conducted by incorporating *fac*-Ir(ppy)$_3$ (2 mol%) as the photocatalyst, acetonitrile as the solvent, and K_2CO_3 as the base. The reaction mixture was irradiated with 30 W blue LED at room temperature for 12 hours, successfully yielding the desired product **3aa** in 55% (Table 1, entry 1). These promising initial results prompted us to further optimize the reaction conditions for this photocatalytic transformation. Subsequently, a series of photocatalysts, including Eosin Y, Acr-Mes$^+$ClO$_4^-$, 3-CzIPN, 4-CzIPN, and Ir(ppy)$_2$(dtppy)PF$_6$, were evaluated due to their pivotal roles in photocatalytic processes. Among these candidates, the first two failed to promote the reaction, whereas the latter three exhibited relatively modest performance, delivering yields ranging from 45% to 52% (entries 2–6). Next, the influence of various solvents was explored by screening several alternatives. The experimental outcomes revealed the following solvent-dependent yields: dimethyl sulfoxide (DMSO) yielded 44% of the target compound **3aa**, 1,4-dioxane afforded 21%, dichloroethane (DCM) produced 33%, while *N*,*N*-dimethylformamide (DMF) resulted in only a trace amount (entries 7–10). These data collectively demonstrated that none of the tested solvents effectively improved the yield of **3aa**. Furthermore, alternative inorganic bases, including Cs_2CO_3, $NaHCO_3$, pyridine, and *N*,*N*-diisopropylethylamine (DIPEA), were examined, leading to the formation of **3aa** in 23%–53%. Yields. However, none of these bases outperformed K_2CO_3 in terms of yield (entries 11–14). Fortuitously, our efforts to adjust the molar ratio between **1a** and K_2CO_3 to 1 : 1.5 proved fruitful, culminating in a commendable 72% yield of **3aa**.

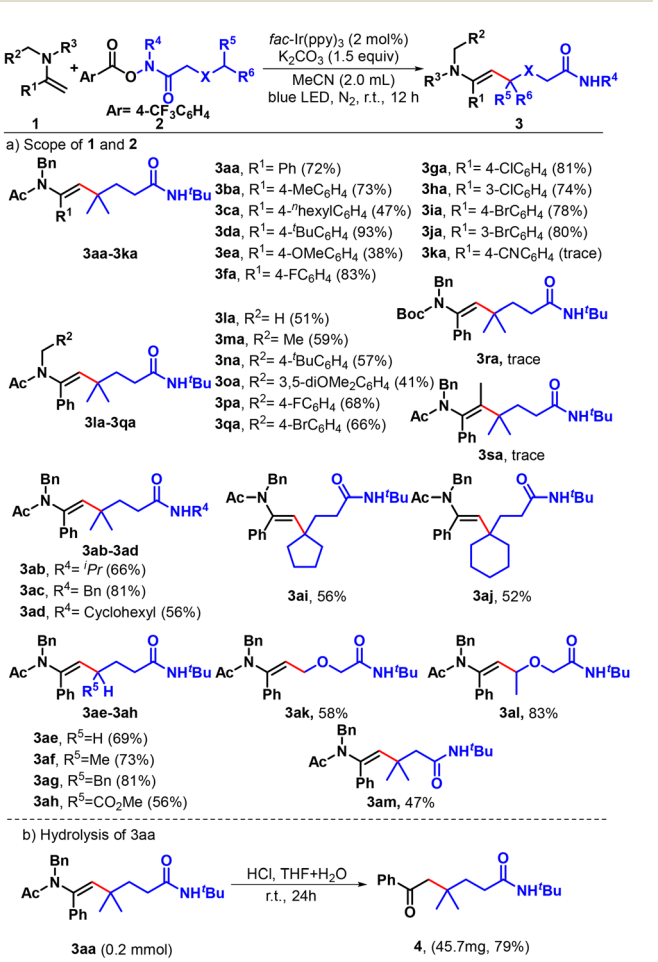
Once we had determined the optimal reaction conditions, our focus turned to assessing the substrate scope and versatility of this photocatalytic 1,5-HAT/alkenylation process, with the results presented in Scheme 2a. Firstly, *N*-((4-(trifluoromethyl)benzoyl)oxy)pentanamide (**2a**) was reacted with enamides **1** that featured different electronic properties and substituent positions (R^1) on the phenyl ring connected *via* the

Table 1 Optimization of the reaction conditions for **3aa** [a]



Entry	[PC]	Solvent	Base	Yield[b] (%)
1	*fac*-Ir(ppy)$_3$	MeCN	K_2CO_3	55%
2	Eosin Y	MeCN	K_2CO_3	Trace
3	Mes-Acr$^+$ClO$_4$	MeCN	K_2CO_3	Trace
4	3-CzIPN	MeCN	K_2CO_3	45%
5	4-CzIPN	MeCN	K_2CO_3	48%
6	Ir(ppy)$_2$(dtppy)PF$_6$	MeCN	K_2CO_3	52%
7	*fac*-Ir(ppy)$_3$	DMSO	K_2CO_3	44%
8	*fac*-Ir(ppy)$_3$	1,4-Dioxane	K_2CO_3	21%
9	*fac*-Ir(ppy)$_3$	DCM	K_2CO_3	33%
10	*fac*-Ir(ppy)$_3$	DMF	K_2CO_3	Trace
11	*fac*-Ir(ppy)$_3$	MeCN	Cs_2CO_3	52%
12	*fac*-Ir(ppy)$_3$	MeCN	$NaHCO_3$	32%
13	*fac*-Ir(ppy)$_3$	MeCN	Pyridine	53%
14	*fac*-Ir(ppy)$_3$	MeCN	DIPEA	23%
15[c]	*fac*-Ir(ppy)$_3$	MeCN	K_2CO_3	72%

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.6 mmol), base (0.6 mmol), [PC] (2 mol%), solvent (2.0 mL), N_2, under 30 W blue LED irradiation at room temperature for 12 h. [b] Isolated yield based on substrate **1a**. [c] K_2CO_3 (0.3 mmol).

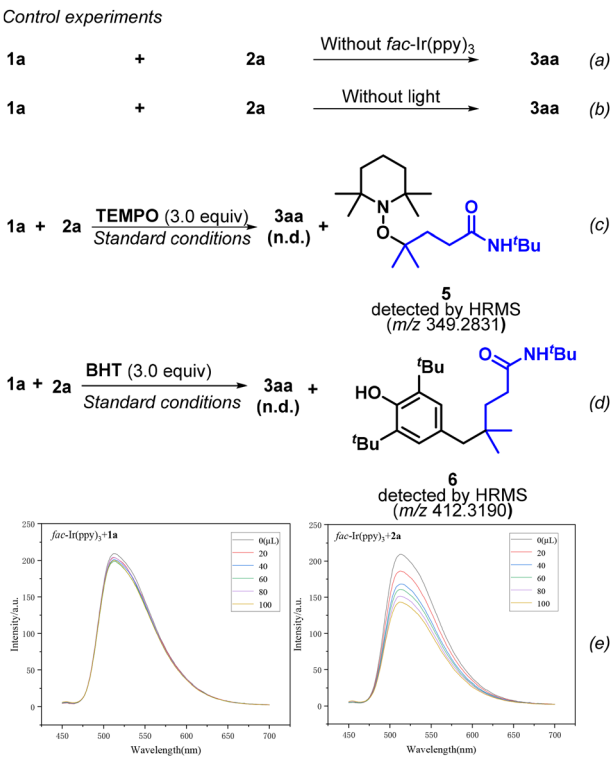


Scheme 2 Substrate scope for the synthesis of **3** and the synthesis of **4**.

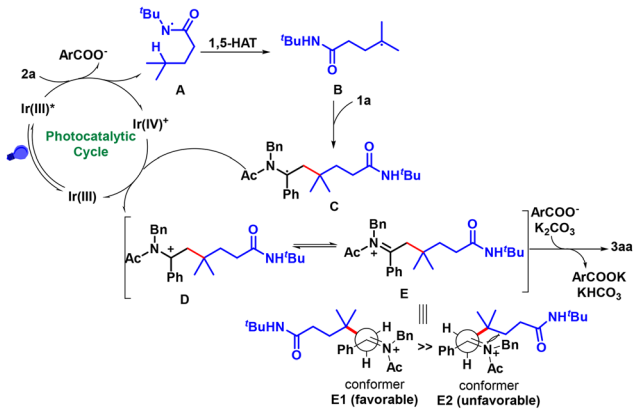
alkenyl group. Remarkably, all of these enamides smoothly underwent the photocatalytic transformation. Under the standard reaction conditions, a broad range of substituents, including methyl (**1b**), *n*-hexyl (**1c**), *t*-butyl (**1d**), methoxy (**1e**), fluoro (**1f**), chloro (**1g–h**), and bromo (**1i–j**) groups, were well-tolerated, and the corresponding products **3ba–3ja** were obtained in 38%–93% yields. Among these functional groups, the *tert*-butyl group was outstanding, as the reaction involving the enamide with this substituent (**1d**) afforded the highest yield (93%) of product **3da**. The strong electron-withdrawing cyanide group present in substrate **1k** exhibited incompatibility with this conversion process. When it came to the R^2 substituents on the methylene group attached to the nitrogen atom, a hydrogen atom (**1l**), methyl group (**1m**), and substituted phenyl groups (**1n–1q**) demonstrated excellent compatibility with this transformation. Consequently, the corresponding trisubstituted enamides **3la–3qa** were isolated in 41%–68% yields. Unfortunately, the substrate equipped with *tert*-butoxycarbonyl (**Boc**, **1r**) as the *N*-protecting group (R^3) was completely ineffective in this reaction. The tetrasubstituted enamide **3sa** utterly failed to be produced when the trisubstituted enamide **1s** was introduced into the reaction. This outcome is likely attributable to the steric hindrance effect. Following that, an investigation was carried out into the possible structural alterations of hydroxamides **2**, choosing **1a** as the reaction partner. When the *tert*-butyl group was swapped with other groups (R^4), such as iso-propyl (**2b**), benzyl (**2c**), and cyclohexyl (**2d**), it had no adverse effect on this alkenylation process, furnishing the target products **3ab–3ad** with yields ranging from 56% to 81%. Notably, in addition to tertiary γ-C (sp^3)–H, primary (**2e**) and secondary (**2f–2h**) γ-C(sp^3)–H were also tested. The results demonstrated that they were well-accommodated in this protocol, enabling the synthesis of bisamide-containing enamides **3ae–3ah** with 56%–81% yields. Moreover, substrates containing cyclopentyl (**2i**) and cyclohexyl (**2j**) groups smoothly underwent this photocatalyzed transformation, yielding **3ai** and **3aj** with yields of 56% and 52%, respectively. Oxa-hydroxamide compounds (**2k–2l**) were all favorably converted into the desired products **3ak** and **3al**, achieving yields of 58% and 83%, respectively. Excitingly, this 1,5-HAT/alkenylation process could be expanded to 1,4-HAT/alkenylation under the standard reaction conditions, resulting in the desired product **3am** with a yield of 47%. Notably, when **3aa** was subjected to a deconstructive hydrolytic reaction in the presence of hydrochloric acid, the hydrolyzed product **4** was obtained with a yield of 79% (Scheme 2b). Regrettably, achieving reaction scale-up proved challenging, as significant quantities of unreacted raw materials are recovered even when performing the reaction on a 5 mmol scale under standard conditions. It is crucial to emphasize that all the isolated products **3** were confirmed to have a complete (*E*)-configuration, as verified through ^{1}H NMR analysis. The precise stereochemical configuration of compound **3ga** was unequivocally determined *via* X-ray diffraction analysis (CCDC 2446825).

To delve deeper into the reaction mechanism of this protocol, a series of control experiments were carried out. Firstly, in the absence of photocatalysts or blue-light irradiation, no target product **3aa** was detected. This finding unequivocally confirms that both the catalyst and light are indispensable for the transformation to occur (Scheme 3a and b). The results of the on–off light experiment further prove the importance of light (Fig. S2). Furthermore, under standard reaction conditions, 3.0 equivalents of a radical scavenger, either 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) or butylated hydroxytoluene (BHT), were added to the reaction system. The reaction was completely halted, as evidenced by the non-detection of the desired product **3aa**. These results strongly suggest that this transformation may proceed *via* a radical pathway. In addition, the identification of the TEMPO-alkyl adduct **5** (*m*/*z* 349.2831) and the BHT-alkyl adduct **6** (*m*/*z* 412.3190) through HR-MS provides further compelling evidence to support this hypothesis (Scheme 3c and d, Fig. S3 and S4). Stern–Volmer fluorescence quenching experiments showed that **2a** had a greater quenching effect on *fac*-Ir(ppy)$_3$* than **1a**, as indicated by the significant drop in *fac*-Ir(ppy)$_3$* fluorescence intensity (Scheme 3e). Specifically, a linear I_0/I-concentration relationship revealed that *fac*-Ir(ppy)$_3$ fluorescence was unchanged with increasing **1a** concentration, and **2a** was likely the quencher (Fig. S5).

By integrating the above-mentioned mechanistic experimental results with insights extracted from existing literature,[4,5,8–16] we propose a plausible reaction mechanism, as illustrated in Scheme 4. Upon exposure to light irradiation, the photocatalyst Ir^{III} undergoes a transition to its excited state, labeled as Ir^{III}*. This excited-state species subsequently reacts with **2a** *via* a single-electron transfer (SET) process,



Scheme 3 Control experiments.



Scheme 4 Probable reaction pathway for the formation of **3aa**.

resulting in the generation of radical **A**, a carboxylate anion ($ArCO_2^-$), and Ir^{IV}. Following this, a 1,5-HAT of radical **A** occurs. During this process, a radical migrates from a nitrogen atom to a carbon atom, leading to the formation of a carbon-centered radical **B**. Radical **B** then participates in a radical addition reaction with the C=C double bonds of **1a**, thereby creating intermediate **C**. Intermediate **C** is then oxidized by Ir^{IV}, which produces a cationic intermediate **D** and regenerates Ir^{III}, thus completing the catalytic cycle. An in-depth analysis of the conformational states of the iminium ion **E**, which exists in equilibrium with **D**, reveals that conformer **E1** is more sterically favorable than **E2**. This preference can be attributed to the reduced $A_{1,3}$-allylic strain in **E1**, and the reduced $A_{1,3}$-allylic strain plays a pivotal role in determining the formation of the (*E*)-configuration of the product. Finally, product **3aa** is produced through the deprotonation of either intermediate **D** or **E** with the aid of K_2CO_3 and $ArCO_2^-$.

Conclusions

In conclusion, we have developed a new visible-light-driven γ-C(sp^3)–H alkenylation for the synthesis of a wide range of trisubstituted enamides *via* a *N*-centered radical-triggered 1,5-HAT strategy starting from readily available hydroxamides and enamides. Notably, this protocol demonstrates exceptional regioselectivity and stereoselectivity in the construction of trisubstituted alkene frameworks, encompassing a wide range of substrates. Moreover, it stands out due to its mild reaction conditions and excellent compatibility with various functional groups. Undoubtedly, the present reaction opens up new avenues for the stereoselective formation of C(sp^3)–C(sp^2) bonds. Currently, our laboratory is actively engaged in further exploring and expanding the application potential of this reaction strategy.

Data availability

The data underlying this study are available in the published article and its SI.

Supplementary information is available. General information, experimental procedure, characterization data and copies of ^{1}H, ^{13}C and ^{19}F NMR spectra. See DOI: **https://doi.org/10.1039/d5ob01116g**.

CCDC 2446825 contains the supplementary crystallographic data for this paper.[18]

Conflicts of interest

There are no conflicts to declare.

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