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One-Pot Synthesis of C3-Alkylated Imidazopyridines from α-Bromocarbonyls under Photoredox Conditions

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A convenient strategy is presented for the synthesis of C3-alkylated imidazopyridines through one pot condensation and alkylation of α-bromocarbonyl compounds with 2-aminopyridines. A series of C3-alkylated imidazopyridines were obtained in moderate to high yields. This strategy was also elaborated to enable the synthesis of zolpidem in short steps, which otherwise require multistep synthesis.

Imidazo[1,2-*a*]pyridine is considered as a "drug bias" heterocyclic skeleton since compounds bearing this heterocycle exhibit a wide range of pharmacological activities, such as anti-inflammatory,[1] anti-ulcer,[2] antiviral activity,[3] anticancer,[4] etc.[5] A series of marketed drugs containing imidazo[1,2-*a*]pyridine core are listed in Figure 1, including zolpidem, alpidem, saripidem, necopidem, minodronic acid, olprinone, etc, and most of them are C3-alkylated imidazo[1,2-*a*]pyridines (Figure 1). Therefore, developing the efficient synthesis of C3-alkylated imidazo[1,2-*a*]pyridines is of great important in organic synthesis.

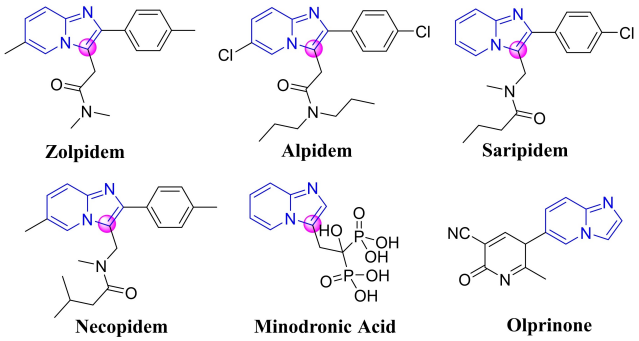
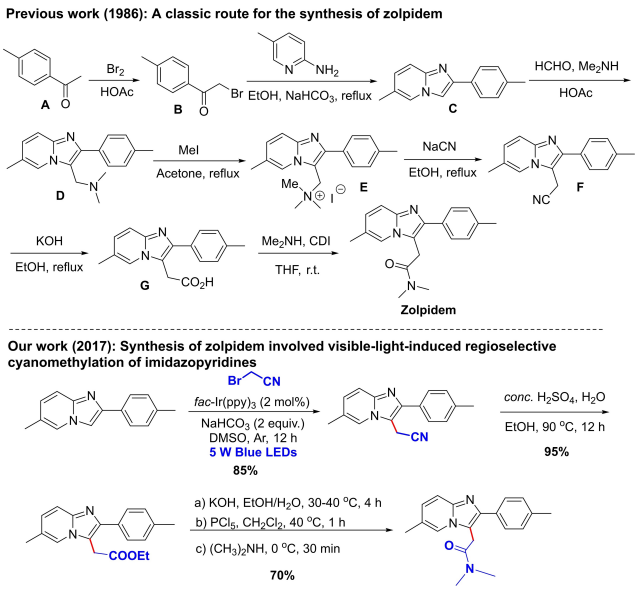


Figure 1. Representative pharmaceuticals containing imidazo[1,2-*a*]pyridine core.

Traditional processes involved the construction[6] of imidazo[1,2-*a*]pyridines and their C3-[7a–e] or C5-[7f] functionalization. For example, a classic route to synthesize zolpidem involved seven steps (Scheme 1). Bromination of 4-methyl acetophenone followed by the condensation with 5-methyl-2-aminopyridine gave 6-methyl-2-(*p*-tolyl)imidazo[1,2-*a*]pyridine (**C**). The intermediate *N*,*N*-dimethyl amino imidazopyridine derivative **D** was obtained from a Mannich reaction of imidazopyridine **C**, formaldehyde and dimethylamine. The subsequent reaction of **D** and methyl iodide generated quaternary ammonium salt **E**. Compound **E** underwent nucleophilic substitution with sodium cyanide to give the 3-cyanomethylated imidazopyridine **F**.[8] In 2009, Satyanarayana and co-workers modified this route to four stages.[9] However, toxic reagents CH_3I and NaCN were still used. In 2017, to overcome this limitation, our group developed a visible-light-induced regioselective cyanomethylation reaction



Scheme 1. Synthesis of zolpidem in different routes.

of imidazopyridines using bromoacetonitrile as the cyanomethyl source and applied this method to the synthesis of zolpidem (Scheme 1).[10]

In order to improve the reaction efficiency to synthesize C3-functionalized imidazo[1,2-*a*]pyridines, a variety of straightforward methods such as multicomponent reactions and "one-pot" reactions were developed in the past decades. In 1998, Groebke and co-workers made a seminal contribution to three component reaction of 2-aminopyridines, aldehydes, and isonitriles for the direct formation of 3-amino-substituted imidazo[1,2-*a*]pyridines (Scheme 2a).[11a] In 2010, the groups of

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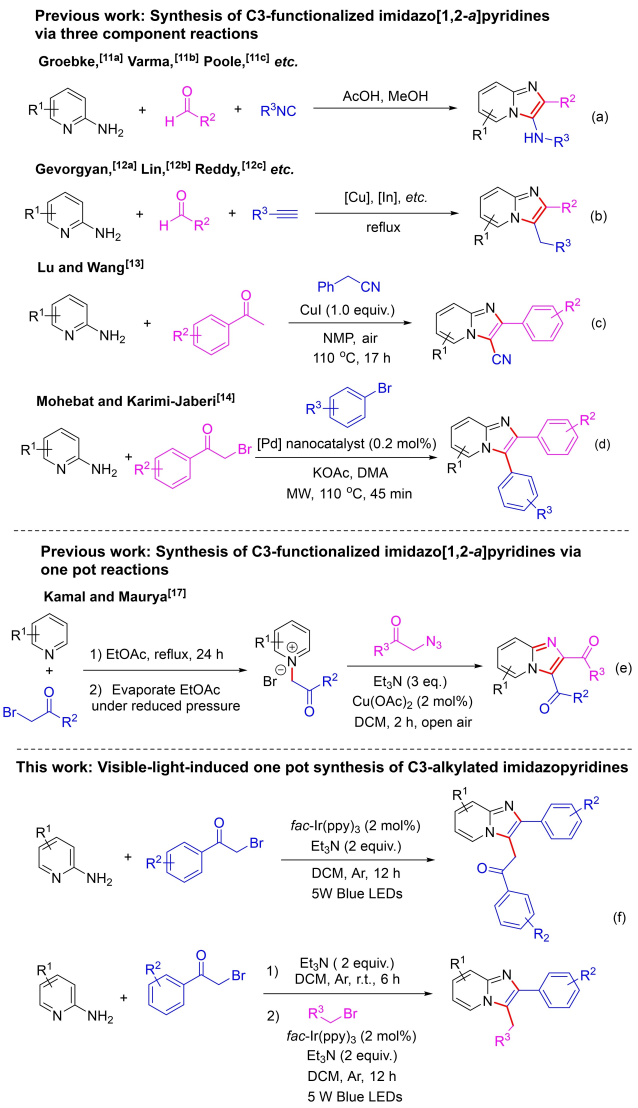
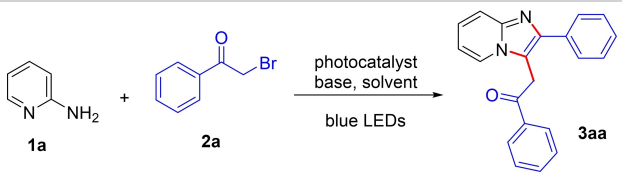


Scheme 2. Synthesis of C3-functionalized imidazo[1,2-*a*]pyridines via three component reactions or one pot reactions.

Gevorgyan[12a] and Lin[12b] respectively developed copper-catalyzed three component reaction of 2-aminopyridines, aldehydes and alkynes to synthesize C3-alkylated imidazo[1,2-*a*]pyridines (Scheme 2b). Benzyl cyanide is an operational safe cyanating reagent. In 2015, Lu, Wang, et al. described a copper-mediated three-component reaction to access 3-cyanoimidazo[1,2-*a*] pyridines from 2-aminopyridines, acetophenones and benzyl cyanide (Scheme 2c).[13] In 2018, Mohebat, Karimi-Jaberi and co-worker reported a three component reaction of 2-aminopyridines, 2-bromo-1-arylethanones and aryl bromides catalyzed by palladium nanoparticles for the synthesis of 2,3-diarylimidazo[1,2-*a*]pyridine derivatives under microwave irradiation (Scheme 2d).[14] In 2020, Singh and co-workers delivered a visible-light-induced three component reaction of benzylamine, 2-aminopyridine and *t*-butyl isocyanide for the synthesis of 3-aminoimidazo[1,2-*a*]pyridines.[15] Moreover, In(III)/Cu(I) co-catalyzed three component reaction of malonic esters, benzaldehydes and 2-aminopyridines to give 3-alkoxycarbonylated Imidazopyridines was developed by Chuah et al.[16] Recently, One pot reaction is also an operationally simple procedure. In 2015, a $Cu(OAc)_2$–Et_3N mediated one-pot strategy for the synthesis of imidazo[1,2-*a*]pyridine derivatives was reported by the group of Kamal and Maurya (Scheme 2e).[17] Thomas et al. reported an one pot protocol for Imidazo[1,2-a]pyridine derivatives formation from 2-aminopyridine and the in-situ generated α-bromoketone.[18] Encouraged by the above excellent works and based on our previous research on the synthesis of C3-functionalized imidazo[1,2-*a*]pyridines,[10,19] we herein demonstrated a visible light-induced one pot condensation of α-bromocarbonyl compounds and 2-aminopyridines cascade the subsequent C3-alkylation to generate C3-alkylated imidazopyridines (Scheme 2f).

Initially, 2-aminopyridine (**1a**) and α-bromoacetophenone (**2a**) were selected as model substrates to test the feasibility of the condensation and alkylation reaction (Table 1). Gratifyingly, the reaction indeed occurred in the presence of *fac*-Ir(ppy)$_3$ (2 mol%) as the photocatalyst and Et_3N (2 equiv.) as a base under 5 W blue light-emitting diodes (LEDs) bulb irradiation in DCM at room temperature and gave the C3-alkylated imidazopyridine **3aa** in 80% yield (entry 1). However, only a trace amount of **3aa** was observed when this reaction was carried out in the absence of a base (entry 2). Several other commonly used bases including $NaHCO_3$ and K_2CO_3 were then investigated, and lower yields of **3aa** were obtained (entries 1, 3 and 4). As for solvents, DCM was superior to other solvents such as DCE, EtOAc and DMF (entries 1, 5–7). Then a variety of photocatalysts were screened, and the results showed that Ir(dtbbpy)(ppy)$_2$(PF_6) only resulted in the product **3aa** in 43%

Table 1. Optimization of the reaction conditions.[a]



Entry	Catalyst	Solvent	Base	Yield [%]
1	***fac*-Ir(ppy)$_3$**	**DCM**	**Et_3N**	**80**
2	*fac*-Ir(ppy)$_3$	DCM	–	trace
3	*fac*-Ir(ppy)$_3$	DCM	$NaHCO_3$	41
4	*fac*-Ir(ppy)$_3$	DCM	K_2CO_3	42
5	*fac*-Ir(ppy)$_3$	DCE	Et_3N	55
6	*fac*-Ir(ppy)$_3$	EtOAc	Et_3N	51
7	*fac*-Ir(ppy)$_3$	DMF	Et_3N	33
8	Ir(dtbbpy)(ppy)$_2$(PF_6)	DCM	Et_3N	43
9	4CzIPN	DCM	Et_3N	74
10	eosin Y	DCM	Et_3N	70
11[b]	*fac*-Ir(ppy)$_3$	DCM	Et_3N	0
12[c]	*fac*-Ir(ppy)$_3$	DCM	Et_3N	0
13[d]	*fac*-Ir(ppy)$_3$	DCM	Et_3N	34
14[e]	*fac*-Ir(ppy)$_3$	DCM	Et_3N	32

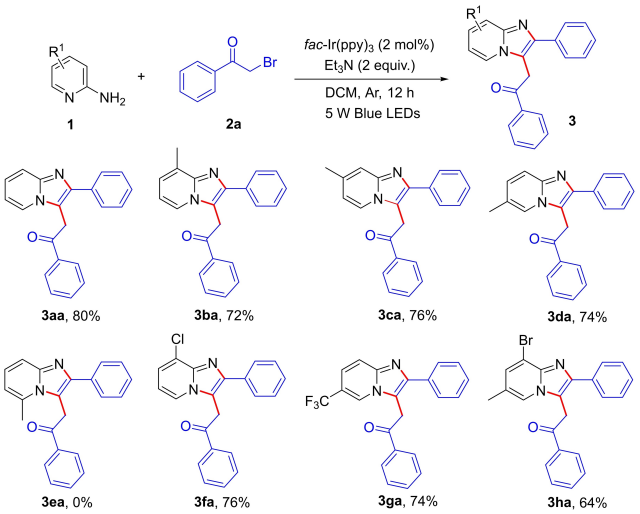
[a] Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2a** (0.6 mmol, 3.0 equiv.), photocatalyst (2 mol%), base (0.4 mmol, 2.0 equiv.), solvent (2 mL), irradiated with 5 W blue LEDs (wavelength range 460–480 nm) under Ar at room temperature for 12 h. [b] No light. [c] No photocatalyst. [d] irradiated with 5 W white LEDs. [e] Under air.

yield, while organic photocatalysts such as eosin Y and 4CzIPN displayed comparable catalytic activity in this reaction (entries 8–10). In addition, no desired product was observed in the absence of visible light irradiation or photocatalyst (entries 11 and 12). Irradiation with white LEDs led to a lower yield of 34% (entry 13). The formation of product **3aa** significantly reduced when the reaction was carried out under air instead of argon, which might be due to the inhibition of oxygen to the generation of the radical intermediate (entry 14).

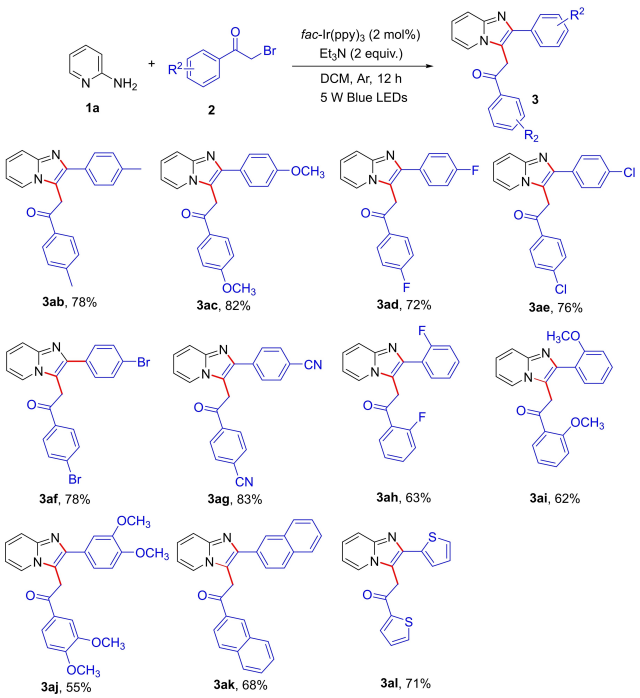
With the optimized reaction conditions in hand, the scope of the 2-aminopyridines were evaluated (Scheme 3). The results showed that 2-aminopyridines with various substituents at the C3-, C4- or C5- position reacted smoothly with α-bromoacetophenone to give the corresponding products in moderate to high yields (**3ba**–**3da**, **3fa**–**3ha**). However, this reaction was relatively sensitive to a substituent at the C6-position of pyridine ring, which failed to enable the formation of **3ea**, presumably owing to the steric hindrance. In addition, when 2-aminoquinoline was employed as the reactant, a mixture that was hard to separate was obtained.

Next, we evaluated various α-bromoketones to examine the generality and limitations of the reaction for the synthesis of C3-alkylated imidazopyridines, and the results were summarized in Scheme 4. Substrates with electron-donating groups (–Me, –OMe) or electron-withdrawing groups (–F, –Cl, –Br, –CN) on the *para*-position of benzene ring could be transferred to the corresponding products in good to excellent yields (**3ab**–**3ag**). Furthermore, substrates bearing -F or -OMe at the *ortho*-position of benzene ring could also provide the desired products **3ah** and **3ai** in 63% and 62% yields, respectively. Moreover, 3,4-dimethoxy or β-naphthyl-substituted substrates were all tolerated, providing the imidazo[1,2-*a*]pyridine derivatives **3aj** and **3ak** in satisfactory yields. In addition, the product **3al** bearing thienyl moiety was also obtained in 71% yield.

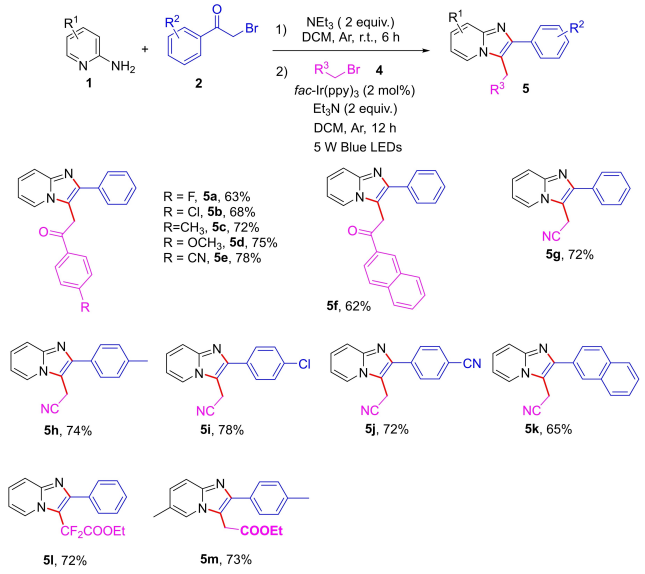
Interestingly, α-bromocarbonyl compounds **2** and alkyl bromides **4** could be successively added to achieve one pot relay processes for the facile synthesis of other C3-alkylated imidazopyridines (Scheme 5). Alkyl bromides **4** with electron-withdrawing



Scheme 3. Scope of 2-aminopyridines. Reaction conditions: **1** (0.2 mmol, 1.0 equiv.), **2a** (0.6 mmol, 3.0 equiv.), *fac*-Ir(ppy)$_3$ (2 mol%), Et$_3$N (0.4 mmol, 2.0 equiv.), DCM (2 mL), irradiated with 5 W blue LEDs under Ar at room temperature for 12 h.



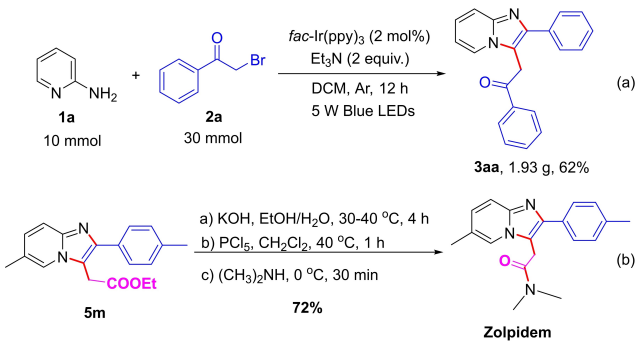
Scheme 4. Scope of various α-bromoketones. Reaction conditions: **1a** (0.2 mmol, 1.0 equiv.), **2** (0.6 mmol, 3.0 equiv.), *fac*-Ir(ppy)$_3$ (2 mol%), Et$_3$N (0.4 mmol, 2.0 equiv.), DCM (2 mL), irradiated with 5 W blue LEDs under Ar at room temperature for 12 h.



Scheme 5. Scope of various substituents on the alkyl bromides. Reagents and conditions: 1) The solution of **1** (0.2 mmol, 1.0 equiv.), **2** (0.2 mmol, 1.0 equiv.) and Et$_3$N (0.4 mmol, 2.0 equiv.) in DCM (2 mL) was stirred under Ar at room temperature for 6 h. 2) *fac*-Ir(ppy)$_3$ (2 mol%) and alkyl halides **4** (0.3 mmol, 1.5 equiv.) were added to the above mixture and the reaction mixture was irradiated with 5 W blue LEDs under Ar at room temperature for 12 h.

groups (–F, –Cl, –CN) or electron-donating groups (–Me, –OMe) on the *para*-position of aromatic ring could be transferred to the corresponding products **5a**–**5e** in good to high yields (63–78%). Bromomethyl 2-naphthyl ketone (**4f**) were also tolerated well in this one pot reaction and 62% yield of **5f** was obtained. This protocol was further extended to other alkyl bromides like bromoacetonitrile, $BrCF_2COOEt$ and $BrCH_2COOEt$ to enlarge the generality of the protocol. To our delight, A series of C3-alkylated imidazopyridines **5g**–**5m** were obtained in satisfactory yields (65–78%).

To demonstrate the practicality of this one pot protocol, the reaction was performed on gram-scale with 2-aminopyridine (**1a**, 0.94 g, 10 mmol) and α-bromoacetophenone (**2a**, 5.97 g, 30 mmol), providing the desired product **3aa** in 62% yield (1.93 g) (Scheme 6a). Notably, zolpidem could be easily obtained by the hydrolysis of imidazopyridine ester **5m** and subsequent amidation according to the procedure of Namboothiri[20] and our previous work[10] (Scheme 6b).



Scheme 6. Gram-scale reaction and further transformation of the product **5m** to zolpidem.

The Stern-Volmer experiment showed that the luminescence of *fac*-Ir(ppy)$_3$ could be easily quenched with an increase in the concentration of α-bromoacetophenone (**2a**) (Figure 2 and Figure 3, for details see the Supporting Information). Several control experiments were performed to study the mechanism of this visible-light-induced condensation and alkylation for the synthesis of C3-alkylated imidazopyridines (Scheme 7. For details see the Supporting Information). 2-Phenylimidazo[1,2-*a*]pyridine (**7**) was obtained through the condensation of 2-aminopyridine and α-bromoacetophenone in the absence of light and a photoredox catalyst (Scheme 7-(1)). The direct C3-alkylation of 2-phenylimidazo[1,2-*a*]pyridine with α-bromoacetophenone took place under the photoredox catalysis (Scheme 7-(2)). The above results shown that the condensation and alkylation processes occurred independently. Moreover, when a radical scavenge 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) was added to the reaction mixture of **2a** and 2-phenylimidazo[1,2-*a*]pyridine (**7**) under the same conditions, the C3-alkylation reaction was completely suppressed, and the TEMPO-adduct **6a** was isolated in 68% yield (Scheme 7-(2)). Meanwhile, when TEMPO was added to the reaction mixture of **1a** and **2a** under the standard reaction conditions, the TEMPO-adduct **6a** and 2-phenylimidazo[1,2-*a*]pyridine (**7**) could be isolated in 20% and 46% yields respectively (Scheme 7-(3)). These results suggested that the photocatalytic reaction probably proceeded via a radical pathway.

Based on the above results and related reports, a possible mechanism of the reaction is proposed in Scheme 8. Initially, the displacement of the bromine atom of α-bromoacetophenone by the nitrogen atom of pyridine ring led to the formation of pyridinium salt **H**, which was subjected to a ring closure to construct the imidazopyridine **7**.[21] The *fac*-Ir(III)(ppy)$_3$ was converted to the excited state [*fac*-Ir(III)(ppy)$_3$]* upon the irradiation of 5 W blue LEDs. A single electron transfer (SET) from Ir*(III) to α-bromoacetophenone resulted in the formation of Ir(IV) and radical intermediate **I**.[22] The regioselective addition of the electron-

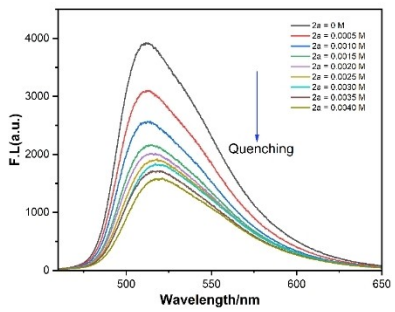


Figure 2. Emission spectra of *fac*-Ir(ppy)$_3$ at different concentrations of **2a**.

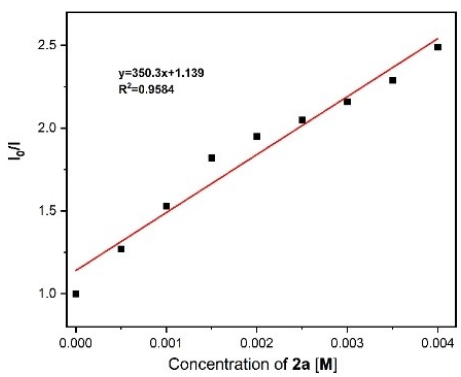
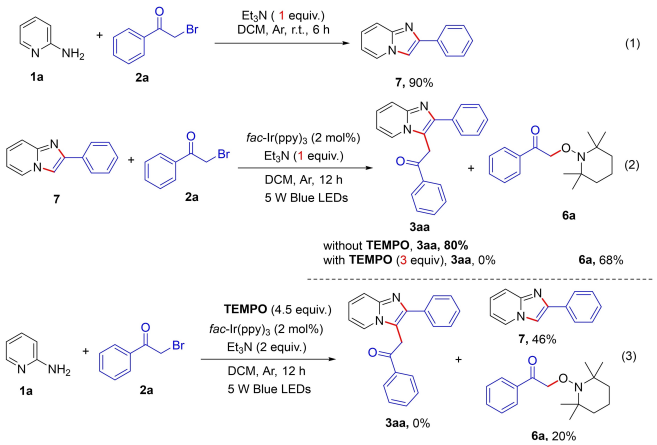
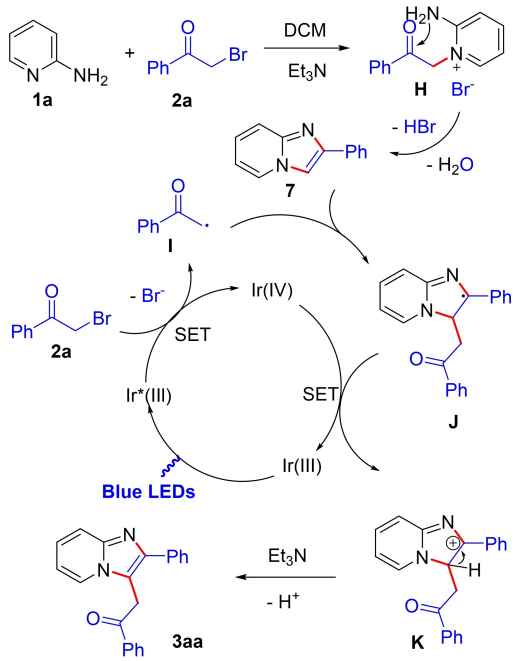


Figure 3. Stern-Volmer plot of *fac*-Ir(ppy)$_3$ at different concentrations of **2a**.



Scheme 7. Control experiments.



Scheme 8. Proposed reaction mechanism.

deficient radical **I** to the electron-rich C3-position of imidazopyridine **7** furnished a new radical **J**, which was oxidized by the Ir(IV) to produce the carbocation **K** and regenerate the Ir(III) photocatalyst via the second SET process. Finally, the deprotonation of **K** with the aid of the base Et_3N produced the desired product **3 aa**.

In summary, we have presented an expedient one pot condensation and alkylation of 2-aminopyridines with a wide variety of α-bromocarbonyl compounds for the construction of C3-alkylated imidazopyridines under visible-light photoredox catalysis. This new protocol exhibits remarkable features, such as short synthetic route, readily accessible substrates, operational simplicity, and mild reaction conditions. This strategy provided an attractive route for the rapid synthesis of zolpidem and can be extended further to obtain other C3-functionalized imidazo[1,2-*a*]pyridine derivatives.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: α-Bromocarbonyls · C3-Alkylated imidazopyridines · Photoredox catalysis · Visible light · Zolpidem

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