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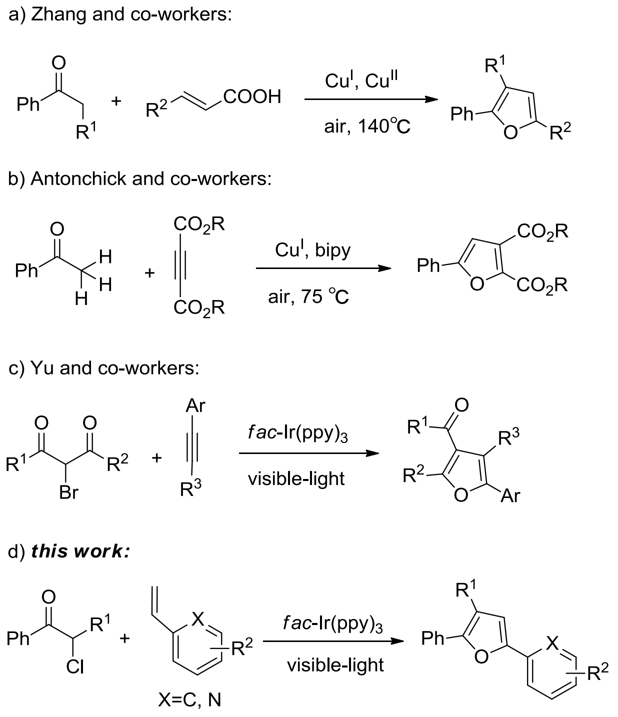
Domino Radical Addition/Oxidation Sequence via Photocatalysis: One-Pot Synthesis of Polysubstituted Furans from α-Chloro-Alkyl Ketones and Styrenes

Shuang Wang,[a] Wen-Liang Jia, [a] Lin Wang,[a] Qiang Liu*[a] and Li-Zhu Wu* [b]

Abstract: A new domino reaction has been developed that allows the combination of styrenes and α-alkyl ketone radicals to afford a wide array of polysubstituted furans in good to excellent yields under mild and simple reaction condition. Key to the success of this novel protocol is the use of photocatalyst *fac*-Ir(ppy)$_3$ and oxidant $K_2S_2O_8$. Mechanistic studies by radical scavenger and photoluminescence quenching suggest that a radical addition/oxidation pathway is operable.

Five-membered heterocycle furans, as fundamental structural units, broadly exist in many bioactive natural products and diverse pharmaceuticals[1] such as furan-2-carbohydrazide and PTTP,[2] which are used as antagonists treating diseases like diabetes and Parkinson. Besides, furan units can be used as versatile synthetic intermediates in the synthesis of complex natural products.[3] For example, double bonds of furans could be utilized in pericyclic reaction and Diels-Alder addition; enol structure could be applied in Michael-type addition. Consequently, the development of efficient methods for the synthesis of multisubstituted furans has gained much attention. Over the past few decades, there are some commonly approaches to polysubstituted furan derivatives such as classical methods of Paal-Knorr/Feist-Benary cyclocondensation[4] and conventional means of metal-mediated cycloisomerization.[5] however, these protocols often suffer from limited availability of functionalized precursors and harsh conditions. Therefore, the development of more efficient and practical strategies to prepare polysubstituted furan derivatives from simple and readily accessible raw materials is still highly sought after.

Over the past century, free-radical chemistry has grown at a dramatic pace within the field of organic chemistry. Among the vast radical libraries, α-ketone radicals are potentially promising intermediates for various transformations such as cyclization,[6] oxidation[7] and addition,[8] which could successfully introduce heteroatom oxygen into compounds. In the recent years, α-aryl ketone radicals, as representative cases of α-ketone radicals, are widely applied to synthesize polysubstituted furans due to the polarity of carbonyl group. For example, in 2013, Zhang and his workers[9] described a novel method for the generation of polysubstituted furans via copper-mediated annulation of simple aryl ketones with α,β-unsaturated carboxylic acids, a protocol that would produce α-aryl ketone radical for constructing a variety of targeted molecules (Scheme **1a**). Later on, Antonchick *et al.*[10] also reported copper-catalyzed radical addition of acetopheones to alkynes and obtained a series of multisubstituted furans, a strategy which also offers α-aryl ketone radical (Scheme **1b**). However, all these systems require either high loading of transition-metal catalysts or radical initiators and high temperature.[11]


a) Zhang and co-workers:

b) Antonchick and co-workers:

c) Yu and co-workers:

d) ***this work:***

Scheme 1. Preparation of polysubstituted furans

Recently, visible-light photocatalysis has offered an economical and environmentally benign platform for the development of free radical reaction. Especially, reductive dehalogenation of electron-deficient haloalkanes has become an efficient and attractive strategy for producing free radicals through photoinitiated single-electron transfer (SET) pathways, due to the ingenious research efforts of some groups, such as MacMillan,[12] Stephenson,[13] Gagne',[14] Yu,[15] and Xiao.[16] By far, our laboratory has also successfully synthesized γ-Lactones,[17] difluoromethylenephosphonyl (DFMP)-substituted aromatic compounds[18] and 6-(DFMP)-substituted phenanthridines[19] by the method of reductive dehalogenation via visible-light photoredox catalysis. Recently, Yu's group[20] has reported a novel method for generating polysubstituted furans using a photoredox neutral coulping of alkynes with 2-bromo-1,3-dicarbonyl compounds (Scheme **1c**). However,

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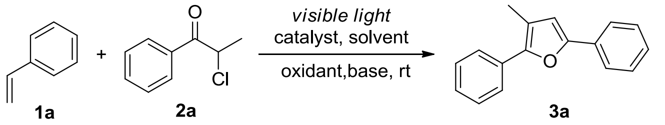
Table 1 Optimization of reaction conditions [a]



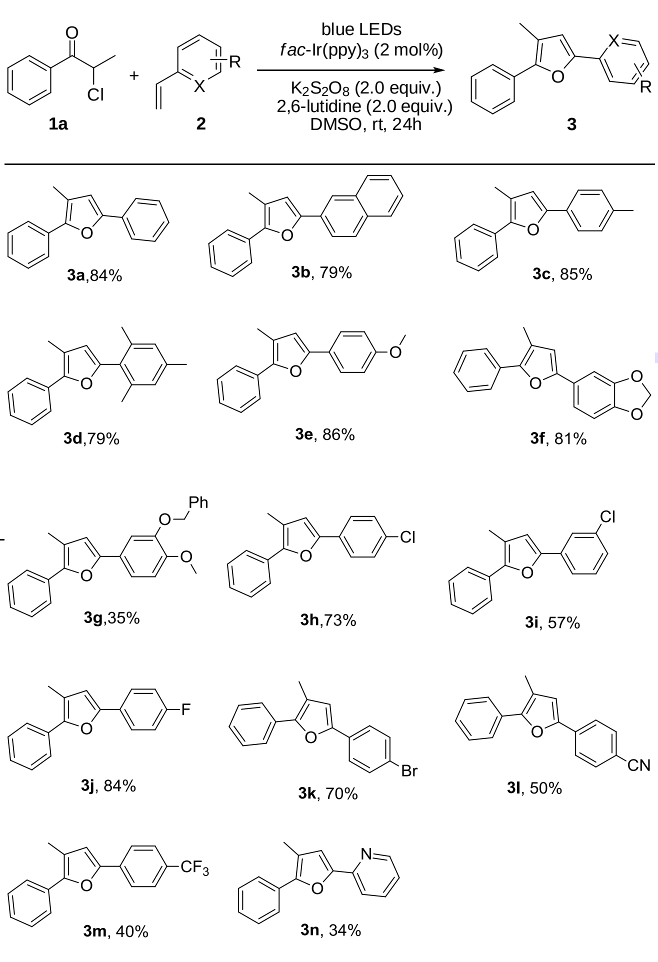
Entry	catalyst	Base[b]	Oxidant[c]	Solvent[d]	Yield (%)[e]
1	*fac*-Ir(ppy)$_3$	Cs_2CO_3	$Na_2S_2O_8$	CH_3CN	21
2	*fac*-Ir(ppy)$_3$	Cs_2CO_3	$Na_2S_2O_8$	acetone	16
3	*fac*-Ir(ppy)$_3$	Cs_2CO_3	$Na_2S_2O_8$	DMF	24
4	*fac*-Ir(ppy)$_3$	Cs_2CO_3	$Na_2S_2O_8$	DMSO	51
5	*fac*-Ir(ppy)$_3$	Cs_2CO_3	$Na_2S_2O_8$	CH_2Cl_2	27
6	*fac*-Ir(ppy)$_3$	K_3PO_4	$Na_2S_2O_8$	DMSO	53
7	*fac*-Ir(ppy)$_3$	KOAc	$Na_2S_2O_8$	DMSO	48
8	*fac*-Ir(ppy)$_3$	K_2CO_3	$Na_2S_2O_8$	DMSO	56
9	*fac*-Ir(ppy)$_3$	2,6-lutidine	$Na_2S_2O_8$	DMSO	71
10	*fac*-Ir(ppy)$_3$	DBN	$Na_2S_2O_8$	DMSO	45
11	*fac*-Ir(ppy)$_3$	2,6-lutidine	$K_2S_2O_8$	DMSO	84
12	*fac*-Ir(ppy)$_3$	2,6-lutidine	$(NH_4)_2S_2O_8$	DMSO	55
13	*fac*-Ir(ppy)$_3$	2,6-lutidine	TBHP	DMSO	36
14	*fac*-Ir(ppy)$_3$	2,6-lutidine	DCP	DMSO	77
15	*fac*-Ir(ppy)$_3$	2,6-lutidine	——	DMSO	51
16[f]	*fac*-Ir(ppy)$_3$	2,6-lutidine	$K_2S_2O_8$	DMSO	23
17	eosin Y	2,6-lutidine	$K_2S_2O_8$	DMSO	N.R.
18	Ru(bpy)$_3$Cl$_2$	2,6-lutidine	$K_2S_2O_8$	DMSO	N.R.
19[g]	*fac*-Ir(ppy)$_3$	2,6-lutidine	$K_2S_2O_8$	DMSO	N.R.
20	——	2,6-lutidine	$K_2S_2O_8$	DMSO	N.R.

[a] Reaction conditions: a mixture of **1a** (0.2 mmol, 1.0 equiv.), **2** (0.4 mmol, 2.0 equiv.), base (0.4 mmol, 2.0 equiv.), oxidant (0.4 mmol, 2.0 equiv.) and photocatalyst (0.004 mmol, 2.0 mol%) in dry solvent (2.0 mL) was irradiated with a 3 W blue LEDs at room temperature for 24 h. The reaction was conducted in a sealed tube full of Ar gas. [b] DBN = 1,5-diazabicyclo[4.3.0]non-5-ene. [c] TBHP = *tert*-butyl hydroperoxide; DCP = dicumyl peroxide. [d] DMF = *N,N*-dimethylformamide; DMSO = dimethylsulfoxide; [e] Isolated yield, N.R. = No Reaction. [f] Exposed in the air. [e] Isolated yield. [g] No irradiation.

when α-bromo-aryl ketones are employed, the products are naphthols instead of polysubstituted furans. Taking inspiration from our previously developed photoredox reductive dehalogenation,[17-19] we recently hypothesized that α-aryl ketone radical could be achieved under much milder conditions from commercially available α-chloro-aryl ketones via visible-light photoredox catalysis and that polysubstituted furans could be achieved by combination of α-chloro-aryl ketones and styrenes via a radical addition/oxidation pathway, a novel method we presented in this study could offer the targeted compounds in just one step(Scheme **1d**).

We initially launched our study by using styrene **1a** (1.0 equiv.), 2-chloro-1-phenylpropan-1-one **2a** (2.0 equiv.) and 2 mol% of *fac*-Ir(ppy)$_3$ in dry CH_3CN. It was encouraging to see that the desired product **3a** was obtained in 21% yield after 24 h of irradiation (blue LEDs, λ = 450 nm) at room temperature when using $Na_2S_2O_8$ (2.0 equiv.) as the oxidant and Cs_2CO_3 (2.0 equiv.) as the base (Table 1, entry 1). Based on the properties of the substrates, the solvent was changed to several other polar aprotic solvents such as acetone, DMSO, DMF and CH_2Cl_2 (Table 1, entries 2-5), and then DMSO was chosen as the ideal organic solvent for the reaction. With the best solvent in hand, other different bases including K_3PO_4, KOAc, K_2CO_3, 2,6-lutidine and DBN were tested (Table 1, entries 6-10), we were delighted to see that the yield of **3a** was greatly improved to 71% in the presence of 2,6-lutidine. Furthermore, some kinds of oxidants containing $K_2S_2O_8$, TBHP and DCP were used in this reaction to replace $Na_2S_2O_8$ (Table 1, entries 11-14), and the yield was further increased to 84% when using $K_2S_2O_8$ as the ideal oxidant.

Table 2. Scope of arylethenes/ heteroarylethenes[a, b]

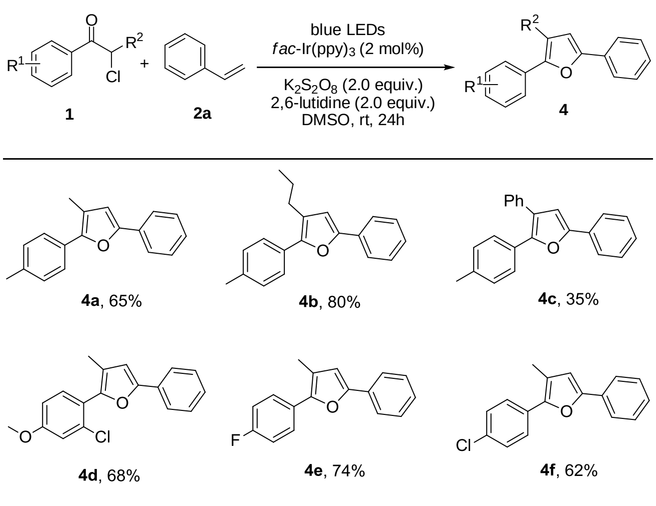


[a] Reaction conditions: a mixture of **1** (0.4 mmol, 2.0 equiv.), **2a** (0.2 mmol, 1.0 equiv.), $K_2S_2O_8$ (0.4 mmol, 2.0 equiv.), 2,6-Lutidine (0.4 mmol, 2.0 equiv.) and *fac*-Ir(ppy)$_3$ (0.004 mmol, 2.0 mol%) in dry DMSO (2.0 ml) was irradiated with a 3 W blue LED lamp at room temperature for 24h. [b] Isolated yield.

To identify the necessity of $K_2S_2O_8$, the reaction was carried out in the absence of oxidant (Table 1, entry 15), and only 51% yield of 3a was obtained. When the reaction was exposed in the air, the yield of the desired product decreased dramatically (Table 1, entry 16). Moreover, the reaction failed in other photocatalysts such as eosin Y and Ru(bpy)$_3$Cl$_2$ (Table 1, entries 17-18). Finally, control experiments showed that no desired products were detected in the absence of light or *fac*-Ir(ppy)$_3$ (Table 1, entries 19-20), indicating that the reaction was a visible-light-driven photoredox system.

Under the optimal conditions, we next investigated the arylethenes scope of this novel and visible-light-driven transformation, as revealed in table 2. A vast variety of substituted arylethenes/heteroarylethenes, including electron-withdrawing and electron-donating groups, could successfully react with 2-chloro-1-phenylpropan-1-one **1**, generating the corresponding products in good to moderate yields (Table 2, **3a-n**, 34-86%). Nevertheless, substrates bearing electron-donating and weak electron-withdrawing substituents (Table 2, **3a**-**f**, **3h** and **3j**-**k**, 73-86%) such as methyl, methoxyl, fluoro, and chloro delivered relatively higher yields than those with strong electron-withdrawing groups (Table 2, **3l**-**m**, 40-50%) such as trifluoromethyl and cyano. On the other hand, sterically demanding substrate (Table 2, **3g**, 35%) had obvious influence on yield. Moreover, the position of substituents on the aryl ring (Table 2, **3h-i**, 57-73%) had certain effect on the yield and the yields for chloro substituted products followed the order of meta<para. Finally, when heteroarylethene was employed as a substrate (Table 2, **3n**, 34%), the product yield was slightly affected, a result attributed to the stability of the pyridine ring in the reaction condition.

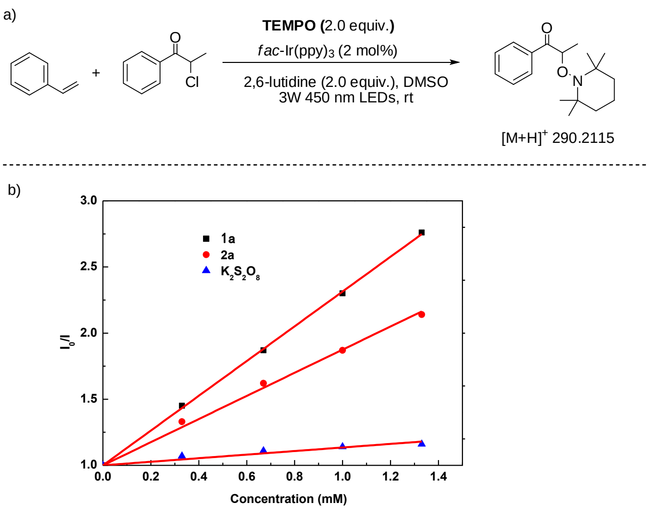
Table 3. Scope of α-chloro arylketones[a, b]



[a] Reaction conditions: a mixture of **1a** (0.4 mmol, 2.0 equiv.), **2** (0.2 mmol, 1.0 equiv.), $K_2S_2O_8$ (0.4 mmol, 2.0 equiv.), 2,6-Lutidine (0.4 mmol, 2.0 equiv.) and *fac*-Ir(ppy)$_3$ (0.004 mmol, 2.0 mol%) in dry DMSO (2.0 ml) was irradiated with a 3 W blue LED lamp at room temperature for 24h. [b] Isolated yield.

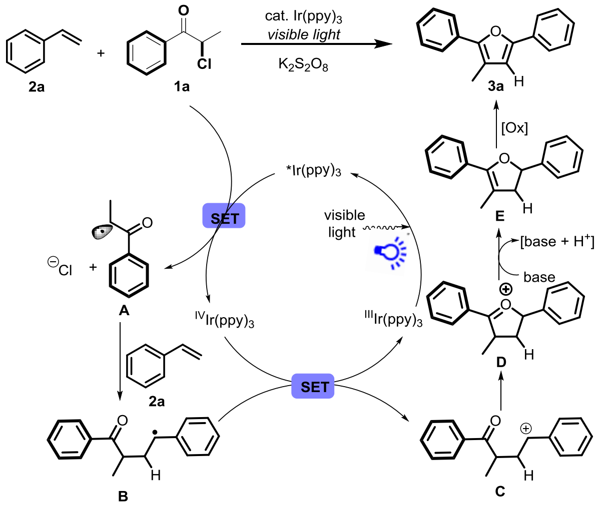
We then turned our attention to the scope of α-chloro-aryl ketones that can participate in this new protocol and function as suitable substrates, as detailed in table 3. It was noteworthy that electron-donating and -withdrawing substituents had little influence on the reaction efficiencies and corresponding α-chloro-aryl ketones were excellent substrates for these reactions (Table 3, **4a-b** and **4d-f**, 65-80%). Notably, α-chloro-aryl ketone bearing a long aliphatic side chain on the α-position offered a desired yield (Table 3, **4b**, 80%). Not surprisingly, α-chloro-aryl ketone bearing a bulk aryl ring was also tolerated under the standard condition (Table 3, **4c**, 35%), albeit the yield is not desirable, presumably owing to the steric hindrance around the site of radical generation.

In order to investigate the mechanism of this transformation, we performed a series of experiments (Scheme 2). When radical inhibitor TEMPO was introduced into the photocatalytic system, no desired product was detected. To our delight, the adduct of the radical inhibitor with 2-chloro-1-phenylpropan-1-one was detected by ESI-HRMS, thus providing plausible evidence of α-aryl ketone radical formation. In addition to the trapping investigation, from the UV-Vis absorption spectra, we can note that only the photocatalyst *fac*-Ir(ppy)$_3$ can be excited under the irradiation of blue LEDs (λ = 450 nm), while both 2-chloro-1-phenylpropan-1-one and styrene are transparent to a photon of blue light (see figure S1 in the Supporting Information). At the same time, the photoluminescence of *fac*-Ir(ppy)$_3$ was quenched by 2-chloro-1-phenylpropan-1-one **1a** with a rate constant of 1.3×10^3 L mol^{-1}(Scheme 2, b). however, the quenching rate constant of *fac*-Ir(ppy)$_3$ with styrene **2a** or $K_2S_2O_8$ was smaller (8.86×10^2 L mol^{-1} and 1.4×10^2 L mol^{-1}). The oxidation potential of the excited *fac*-Ir(ppy)$_3$ is -1.73 V (vs. SCE) [21], which is much lower than the reduction potential of 2-chloro-1-phenylpropan-1-one (-1.48 V, vs. Ag/Ag$^+$, 0.1 M) [22]. Therefore, the significant emission quenching of *fac*-Ir(ppy)$_3$ by **1a** should be attributed to the electron transfer from the excited photocatalyst to **1a**. This finding indicates that the electron transfer from the excited *fac*-Ir(ppy)$_3$ to **1a** is favourable.



Scheme 2. Mechanism investigation (b: Stern-Volmer plots of fluorescence quenching of *fac*-Ir(ppy)$_3$ by **1a**, **2a** and $K_2S_2O_8$)

Based on the above experimental results and previous reports[9, 23], a radical addition/oxidation pathway was proposed in Scheme 3. SET from the excited photocatalyst to **1a** generates electron-deficient α-carbonyl carbon radical **A** along with Ir^{IV}(ppy)$_3$. Intermolecular π-addition of the radical **A** to styrene **2a** produces benzylic radical **B**. Further SET from radical **B** ($E^{ox}_{1/2}$ = 0.73 V, vs. SCE) [21] to Ir^{IV}(ppy)$_3$ ($E^{red}_{1/2}$ = 0.77 V, vs. SCE)[24] gives carbocation **C** and regenerates the photocatalyst. Subsequently, cyclization and deprotonation of **C** aided by the base result in dihydrofuran derivative **E**. Finally, oxidation of intermediate **E** by the excited photocatalyst or $K_2S_2O_8$ provides the desired product **3a**.



Scheme 3. Proposed mechanism

In conclusion, we have developed a novel and straightforward way to synthesis of polysubstituted furans through domino reaction of inexpensive and commercial availably substrates α-chloro-aryl ketones and arylethenes/ heteroarylethenes under visible-light irradiation, a one-pot protocol which exhibits broad scope and wide functional group tolerance. Furthermore, the success of this strategy offered a solid framework for the further development of our research programs in visible-light photocatalysis. The further development of this synthetically valuable photocatalytic processes is a continuing effort in our laboratory.

Experimental Section

General procedure for the preparation of the products: In a 10 mL snap cap vial equipped with a magnetic stirring bar and rubber septum, the $K_2S_2O_8$ (0.4 mmol), *fac*-Ir(ppy)$_3$ (2 mol%), 2,6-lutidine (0.4 mmol), α-chloro-propiophenones **1** (0.4 mmol) were dissolved in dry DMSO (2 mL). The mixture was bubbled with a stream of argon for 30 min via a syringe needle, then the styrenes **2** (0.2 mmol) was added into a snap cap vial through a 100 μL microsyringe. The vial was then irradiated by using two 3W 450 nm blue LEDs. The process of the reaction was monitored by thin-layer chromatography at regular intervals. After 24 hours, H_2O (5.0 mL) was added into the reaction mixture. Then, the mixture was extracted with Et_2O and the combined organic layers were dried over Na_2SO_4 and the solvent was removed under vacuum. The residue was purified by silica gel column chromatography using petroleum/ ethyl acetate to give the desired product.

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Keywords: domino reaction • polysubstituted furans • $K_2S_2O_8$ • room temperature • photocatalysis

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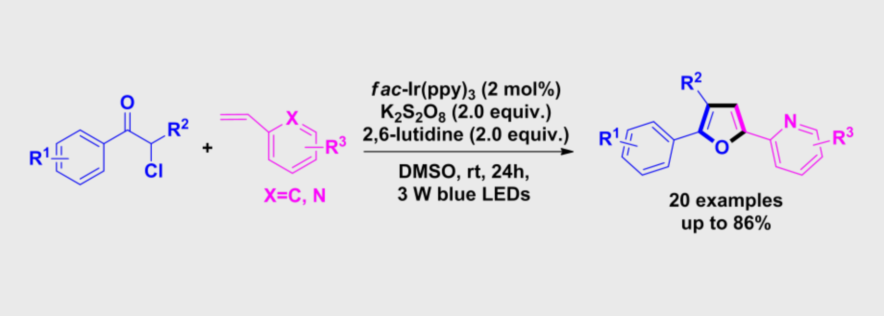

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Domino Radical Addition/Oxidation Sequence via Photocatalysis: One-Pot Synthesis of Polysubstituted Furans from α-Chloro-Alkyl Ketones and Styrenes