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Visible-light induced tandem radical cyanomethylation and cyclization of *N*-aryl acrylamides: access to cyanomethylated oxindoles†

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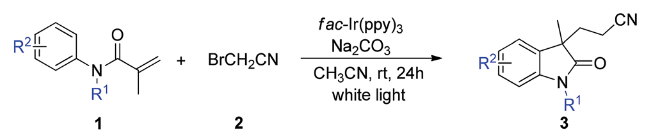
A visible-light induced cyanomethylation of *N*-aryl acrylamides with bromoacetonitrile followed by intramolecular cyclization has been explored. This transformation exhibits a wide substrate scope and significant functional group tolerance, providing a facile synthetic approach and highly efficient access to cyanomethylated oxindoles.

Oxindoles are an ubiquitous heterocycles motif in many natural products, pharmaceuticals and agrochemicals.[1] Moreover, oxindoles have significant biological activities and wide-ranging applications in organic synthesis.[2] Therefore, the search for sustainable and more efficient methods for the preparation of oxindoles is of constant interest. Recently, catalytic difunctionalization of alkenes emerged as an attractive strategy for accessing structurally diverse heterocyclic compounds,[3] for example, a tandem radical addition/cyclization of *N*-aryl acrylamides that provides an elegant method for the construction of the 3,3-disubstituted oxindole skeleton has been reported;[4] and the radical process involving alkylarylation,[5] diarylation,[6] arylnitration,[7] arylphosphorylation,[8] aryltrifluoromethylation,[9] and azidoarylation[10] of *N*-aryl acrylamides have since been disclosed by several groups, allowing the effective formation of the oxindole framework.

Cyanomethylation oxindoles are of significant interest because cyanomethylation reaction is considered to be a privileged reaction which resulted products can be utilized as key intermediates in drug synthesis.[11] Some successful examples synthesizing cyanomethylated oxindoles have been reported in recent years.[12] For example, in 2011, Liu discovered a novel Pd-catalyzed oxidative method to afford nitrile-bearing indolinones, which involves α-C–H activation of both aniline and acetonitrile by the aid of stoichiometric $PhI(OCO^tBu)_2$ and AgF.[12a] Subsequently, You and Zhu demonstrated Cu and Fe-catalysed 1,2-cyanoalkylarylation of *N*-aryl acrylamides for the construction of cyanomethylation of oxindoles using acetonitrile as cyanomethyl source, respectively.[12b,12e] Sheng also developed cyanomethylation of activated alkenes through a radical pathway using AIBN as the radical initiator.[12g] Nevertheless, stoichiometric amount of transition metals or promoter mediates are required in aforementioned examples. A milder and more efficient method for the synthesis of functionalized oxindoles is still highly desirable.

Nowadays, the visible-light photoredox catalysis strategy has been identified as a uniquely powerful and straightforward tool for synthetic transformations in organic chemistry, owing to its high efficiency and environmentally friendly mild reaction conditions.[13] Several groups have been synthesized the oxindole derivatives by the means of UV light or visible-light photoredox catalysis.[14] However, the visible-light photoredox catalysis approaches for their preparation of cyanomethylated oxindoles are extremely limited. As far as we know, only one example involving a visible-light catalyzed cyanomethylated of oxindoles has been reported by the Li group,[12c] in which moderate yields and equivalent of $4\text{-}MeOC_6H_4N_2BF_4$ reagent was used as promoter. As part of our ongoing interest in visible light photoredox catalytic reactions,[15] we present a novel visible-light induced radical addition/cyclization cascade cyanomethylation of *N*-aryl acrylamides for the synthesis of valuable cyanomethyl-containing oxindoles using bromoacetonitrile as cyanomethyl radical source (Scheme 1).

Initially, we investigated this reaction using *N*-methyl-*N*-phenylmethacrylamide (**1a**) and bromoacetonitrile (**2**) as the



Scheme 1 Visible-light induced tandem cyanomethylation and cyclization of *N*-aryl acrylamides.

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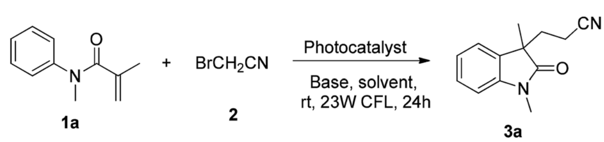
starting materials with K_2CO_3 as the base and *fac*-Ir(ppy)$_3$ (2 mol%) as the catalyst. To our delight, the transformation proceeded smoothly after 24 h of irradiation with a 23 W household fluorescent lamp (CFL) in CH_3CN at room temperature, affording the desired product **3a** in 76% yield (Table 1, entry 1). When this reaction was performed in the absence of base, only 15% yield of **3a** was obtained, and most of the *N*-methyl-*N*-phenylmethacrylamide **1a** was recovered (Table 1, entry 2). Encouraged by this result, the reaction conditions of this cascade were further optimized. Firstly, we screened the catalysts and found that other photoredox catalysts, such as [Ir(dtbbpy)(ppy)$_2$][PF$_6$], Ru(bpy)$_3$Cl$_2\cdot$6H$_2$O, and Eosin Y, resulted in low reaction efficiency (Table 1, entries 3–5). Then we screened different bases (Table 1, entries 6–12). It turned out that Na_2CO_3 was the best base among the inorganic and organic bases tested, giving the product with 93% yield. A survey of commonly used solvents, such as DMF, DMSO, $CHCl_3$, MeOH, and THF, were tested (Table 1, entries 13–17). However, reaction in none of the above solvents afforded higher yield than that in CH_3CN. The highest yield was achieved when 2 equiv. of bromoacetonitrile was used (Table 1, entry 19). Increased to 4 equiv. or reduced to 1.5 equiv. of bromoacetonitrile led to a lower yield of 91% and 71%, respectively (Table 1, entries 18 and 20). Control experiments suggested that photocatalyst and irradiation are indispensable to this transformation (Table 1, entries 21 and 22).

Table 1 Optimization of reaction conditions[a]



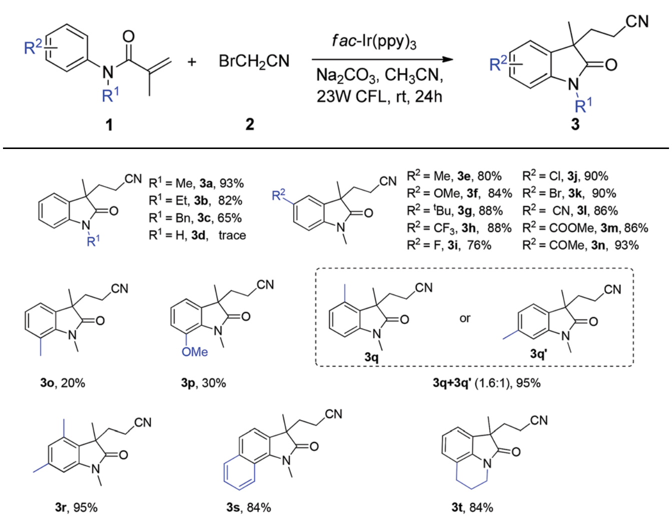
Entry	Photocatalyst	Base	Solvent	Yield[b] (%)
1	*fac*-Ir(ppy)$_3$	K_2CO_3	CH_3CN	76
2	*fac*-Ir(ppy)$_3$	None	CH_3CN	15
3	[Ir(dtbbpy)(ppy)$_2$][PF$_6$]	K_2CO_3	CH_3CN	12
4	Ru(bpy)$_3$Cl$_2\cdot$6H$_2$O	K_2CO_3	CH_3CN	3
5	Eosin Y	K_2CO_3	CH_3CN	5
6	*fac*-Ir(ppy)$_3$	Li_2CO_3	CH_3CN	79
7	*fac*-Ir(ppy)$_3$	Na_2CO_3	CH_3CN	93
8	*fac*-Ir(ppy)$_3$	$NaHCO_3$	CH_3CN	82
9	*fac*-Ir(ppy)$_3$	Na_2HPO_4	CH_3CN	71
10	*fac*-Ir(ppy)$_3$	K_3PO_4	CH_3CN	41
11	*fac*-Ir(ppy)$_3$	KOAc	CH_3CN	46
12	*fac*-Ir(ppy)$_3$	NEt_3	CH_3CN	29
13	*fac*-Ir(ppy)$_3$	Na_2CO_3	DMF	86
14	*fac*-Ir(ppy)$_3$	Na_2CO_3	DMSO	69
15	*fac*-Ir(ppy)$_3$	Na_2CO_3	$CHCl_3$	87
16	*fac*-Ir(ppy)$_3$	Na_2CO_3	MeOH	22
17	*fac*-Ir(ppy)$_3$	Na_2CO_3	THF	85
18[d]	*fac*-Ir(ppy)$_3$	Na_2CO_3	CH_3CN	91
19[e]	*fac*-Ir(ppy)$_3$	Na_2CO_3	CH_3CN	95(93)[c]
20[f]	*fac*-Ir(ppy)$_3$	Na_2CO_3	CH_3CN	71
21	None	Na_2CO_3	CH_3CN	0
22[g]	*fac*-Ir(ppy)$_3$	Na_2CO_3	CH_3CN	0

[a] Reaction conditions: **1a** (88 mg, 0.5 mmol), **2** (180 mg, 1.5 mmol, 3 eq.), catalyst (2 mol%), base (1 mmol, 2 eq.), solvent (5 mL), rt, 24 h, under N_2 atmosphere. [b] Determined by ^1H NMR analysis with benzyl ether as an internal standard. [c] The value in parentheses was isolated yield. [d] 4 equiv. of **2** was used. [e] 2 equiv. of **2** was used. [f] 1.5 equiv. of **2** was used. [g] In the dark.

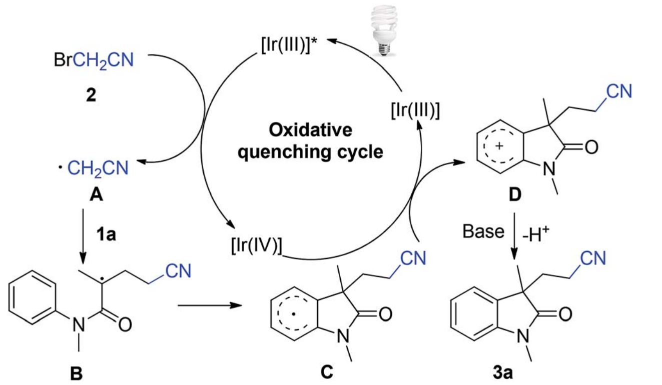
With the optimized reaction conditions in hand, we evaluated the scope of acrylamides with **2** (Table 2). Initially, the examination of different N-protecting groups revealed that methyl-protected (**1a**) was still the best choice, similar ethyl-protected (**1b**) and benzyl-protected (**1c**) substrate gave slightly reduced yields, whereas the reactions of N–H derivatives failed (**1d**). Gratifyingly, various functional groups were well tolerated, and both electron-donating group (*e.g.*, Me, OMe, ^{t}Bu) and electron-withdrawing group (*e.g.*, CN, COOMe, COMe) substituents at the *para* position of the aniline moiety proceeded efficiently to afford the cyclized products **3e–n** in moderate to good yields. Notably, halogen functional groups such as F, Cl, and Br were well-tolerated leading to the corresponding halogen-substituted cyanomethylation of oxindoles in good yields (76–90%, **3i–k**, Table 2), which offered the potential for further synthetic elaboration. For the *N*-aryl acrylamides containing *ortho*-position substituent groups exhibited a particularly distinct steric hindrance effect, and lower yields were observed as a result (Table 2, **3o–p**). *N*-Aryl acrylamides bearing a *meta*-substituent underwent cyanomethylation smoothly to give a mixture product of isomers in 95% yield with poor regioselectivity (**3q** : **3q′** = 1.6 : 1). Moreover, 3,5-dimethyl *N*-aryl amides **3r** also underwent the tandem reaction smoothly. In addition, naphthalene and tetrahydroquinoline derivative were also viable substrates to provide the corresponding oxindoles **3s** and **3t** with the same yield of 84% (Table 2, **3s–3t**).

To gain additional mechanistic insights, 3 equiv. of TEMPO relative to **1a** was added to the reaction system, no desired product **3a** was observed and starting material was recovered,

Table 2 Scope of acrylamides[ab]



[a] Reaction conditions: **1** (0.5 mmol), $BrCH_2CN$ (120 mg, 2 equiv.), base (2 equiv.), catalyst (2 mol%), CH_3CN (5 mL), irradiation with a 23 W household light bulb, rt, 24 h. [b] Yields of isolated products.



Scheme 2 Proposed plausible mechanism.

indicating that a radical process is probably involved in this reaction. On the basis of above results and previous literature reports,[11] a plausible mechanism was proposed (Scheme 2).

Initially, iridium catalyst was excited to generate the excited species *fac*-[Ir(ppy)$_3$]* under visible light irradiation, which then undergoes single electron transfer (SET) process with bromoacetonitrile **2** to generate cyanomethyl radical **A** and Ir^{IV} metal complex. Subsequently, the addition of cyanomethyl radical **A** to activated alkene **1a** afforded alkyl radical **B**, followed by intramolecular cyclization with aryl ring lead to intermediate **C**, which was further oxidized through SET process to give key carbocation **D** and regenerated the photocatalyst. Finally, deprotonation of **D** in the presence of a base gave the desired product **3a**.

Conclusions

In summary, we have disclosed an operationally convenient visible-light photocatalytic tandem cyanomethylation of *N*-aryl acrylamides using available bromoacetonitrile as starting material. The protocol presents a mild and efficient to furnish a variety of functionalized oxindoles. Both electron donating and electron withdrawing groups on the *N*-aryl acrylamides are tolerated in the reaction, and the corresponding products were obtained in moderate to good yields.

Conflicts of interest

There are no conflicts to declare.

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