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Photoredox-neutral ring-opening pyridylation of cyclic oximes *via* phosphoranyl radical-mediated N–O/C–C bond cleavages and sequential radical-radical coupling

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ABSTRACT

A novel photoredox-neutral ring-opening pyridylation of non-prefunctionalized cyclic oximes has been accomplished through phosphoranyl radical-mediated N–O/C–C bond cleavages followed by radical-radical coupling. This mild acid-, base-, and oxidant-free protocol provides highly site-selective and efficient access to distally pyridylated alkylnitriles, which could be scale-up synthesized and readily converted into skeletally diverse compounds. Notably, the oxidized ground-state photocatalyst generated *via* the SET oxidation of the highly reducing excited-state photocatalyst by cyanopyridines might initiate the following phosphoranyl radical-mediated deoxygenative process.

Pyridines as fundamental structural motifs are ubiquitous in complex natural products, pharmaceuticals, catalysts, ligands, functional materials, pesticides, and so on [1–12]. Over the last few decades, extensive efforts have thereby been devoted to developing a plethora of elegant and state-of-art methodologies for the efficient synthesis of structurally diverse pyridine derivatives [13]. Meanwhile, diverse radical-mediated ring-opening/functionalizations of cyclic oxime derivatives were developed to access distally functionalized nitriles [14–28]. However, to the best of our knowledge, there are only four radical-type examples of the incorporation of cyanoalkyl groups derived from cyclic oxime derivatives into pyridine skeletons to access pyridylated nitriles. In 2017, Guo's group developed Ni-catalyzed Minisci-type cyanoalkylation of heteroaromatic *N*-oxides with electron-poor *O*-pentafluorobenzoyl cyclic oximes at elevated temperature [29], in which there were three examples of pyridine cyanoalkylation with exquisite C-2 selectivity and relatively low efficiency, and also initial oxidation of pyridines to pyridine *N*-oxides need to use the oxidants, thus exhibiting poor functional group compatibility (Scheme 1a). Soon after, Xia group disclosed visible-light-induced photoredox Minisci-type cyanoalkylation of heteroarenes with electron-poor *O*-4-trifluoromethylbenzoyl cyclic oximes in the presence of a stoichiometric amount of trifluoroacetic acid [30], in which there was no need of pre-activation of pyridines ring as their *N*-oxides, and yet only three examples of pyridine cyanoalkylation (two examples blocking competitive C-4 site led to C-2 mono-cyanoalkylated products with moderate yields, the third example blocking one of the competitive C-2 sites led to C-2 and C-4 cyanoalkylated products with poor yields) (Scheme 1b). Alternatively, Xu group exploited Ag-catalyzed Minisci-type cyanoalkylation of heteroarenes with cyclic α-imino-oxy acids under acidic and oxidizing conditions [31], in which there were only two examples of pyridine cyanoalkylation (one example led to mono- and difunctionalized products, the other led to C-2 and C-4 cyanoalkylated products) (Scheme 1c). Besides the three Minisci-type reactions mentioned above, Leonori group established an elegant photoredox-nickel dual-catalyzed ring-opening functionalization of cyclic α-imino-oxy acids in the presence of organic base tetramethyl guanidine [32], in which there were three examples of pyridine cyanoalkylation with exquisite C-2 or C-4 selectivity and moderate yields (Scheme 1d). Although such a dual photoredox-

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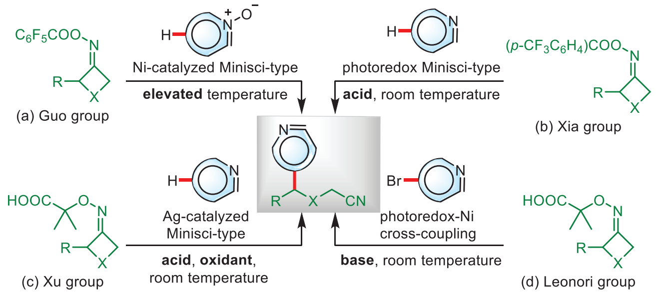


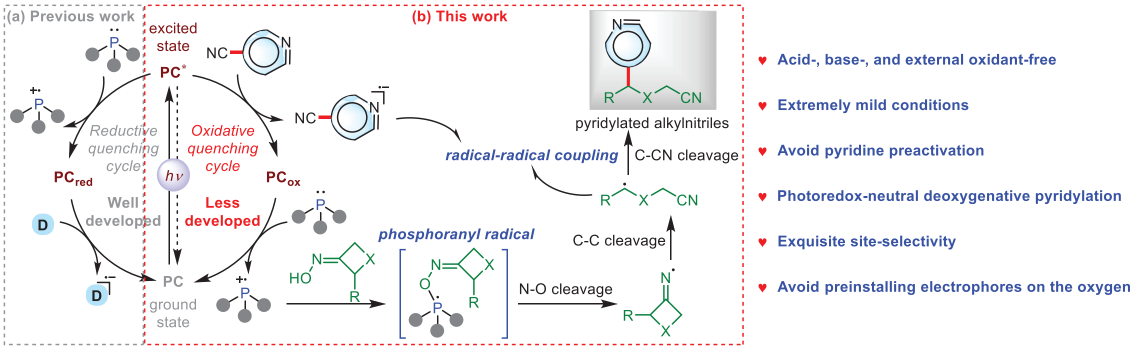
Scheme 1. The already established radical-type ring-opening/pyridylation of cyclic oxime derivatives.

nickel strategy compensated for the inadequacies of the above-mentioned Minisci-type reactions, the specific activation of (hetero)aryl bromides resulted in the addition of the second transition metal catalyst $NiCl_2$·glyme besides the requisite photocatalyst $[Ir(dtbbpy)(ppy)_2]PF_6$. Furthermore, it was worth mentioning that only by voltage-directed installation of appropriate electrophores on the oxygen atom could cyclic oxime derivatives employed in these transformations undergo either SET (single-electron transfer) oxidation or reduction to generate the corresponding cyanoalkyl radicals. All of these limitations are remarkable, and thus the development of highly site-selective, straightforward, and efficient approaches to access structurally diverse pyridylated nitriles from non-prefunctionalized starting materials under mild acid-, base- and oxidant-free conditions is of great synthetic value.

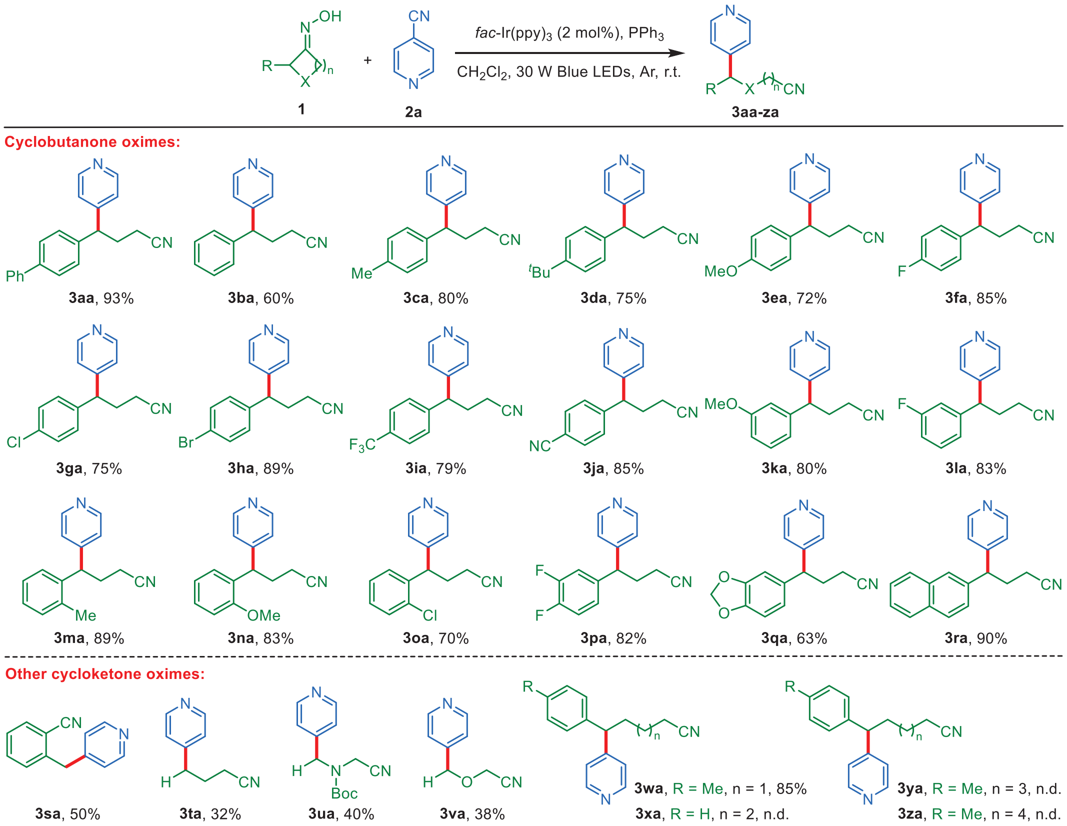
Recently, photopromoted phosphoranyl radical-mediated fragmentation has emerged as a powerful alternative strategy for expedient access to diverse radicals *via* the single-electron deoxygenative process of oxygen-containing substrates such as carboxylic acids, alcohols, oximes, sulfoxides [33–38]. Mechanistically, in almost all cases of the already established methodologies, trivalent phosphorus compounds reductively quenched the highly oxidizing excited-state photocatalysts to yield phosphinium radical cations, which underwent the nucleophilic attack from oxygen-containing substrates and subsequent β-scission to generate the corresponding radical intermediates for further transformations (Scheme 2a). In contrast, owing to the extremely low excited-state oxidation potential of the highly reducing photocatalysts, such photocatalyst-participated phosphoranyl radical-mediated deoxygenative process is still elusive and rare. To the best of our knowledge, there is an exclusive example of photocatalytic sulfoxide deoxygenation using the highly reducing photocatalyst *fac*-$Ir(ppy)_3$ [39], which might be oxidatively quenched by the hypothetical sulfoxide-phosphine adducts to provide the oxidized ground-state photocatalyst for further oxidation of trivalent phosphorus compounds. Inspired by this work and seminal pioneering reports on elegant radical-based *ipso*-functionalizations of cyano(hetero)arenes, we envisaged that other electrophiles such as cyanopyridines instead of the sulfoxide-phosphine adducts were selected to oxidatively quench those highly reducing excited-state photocatalysts **PC***, affording the persistent cyanopyridyl radical anions as well as the oxidized ground-state photocatalysts $\mathbf{PC_{ox}}$. Then, trivalent phosphorus compounds undergo the SET oxidation by such oxidized photocatalysts $\mathbf{PC_{ox}}$ to generate phosphinium radical cations and the ground-state photocatalysts, thus completing a net photoredox-neutral process with only one photocatalyst. Finally, the resulting phosphinium radical cations could further facilitate the initiation of the deoxygenative process from the non-prefunctionalized cyclic oximes to produce the transient cyanoalkyl radicals, which might subsequently couple with the persistent radical anions through the postulated radical-radical coupling pathway to provide the target products. Hence, we report an acid-, base-, and oxidant-free photoredox-neutral ring-opening pyridylation strategy of non-prefunctionalized cyclic oximes to afford distally pyridylated nitriles, which features exquisite site-selectivity, broad substrate scope, and good functional group compatibility (Scheme 2b).

To verify the feasibility of our hypothesis, we initially selected cyclobutanone oxime **1a** and 4-cyanopyridine **2a** as the model substrates to explore the reaction conditions under visible-light irradiation at room temperature. To our delight, we obtained the compound γ-pyridylated alkylnitrile **3aa** in 93% yield after systematic optimization of the reaction parameters, including trivalent phosphorus compounds, photocatalyst, and solvent (Table 1, entry 1). Notably, other electron-rich trivalent phosphorus compounds were also able to facilitate this ring-opening pyridylation reaction albeit with reduced yields (entries 2–6). In contrast, when electron-deficient $P(C_6F_5)_3$ was used in place of PPh_3, the reaction failed to proceed (entry 7). Then, we evaluated the performance of other photocatalysts, highly oxidizing photocatalyst **PC3**–**PC6** was less efficient than highly reducing photocatalyst **PC1** or **PC2** (entries 8–12) [40,41]. Furthermore, solvent screening demonstrated that these reactions performed in other solvents such as MeCN and THF did not obtain as high yields as CH_2Cl_2 and DCE (entries 13–15). As anticipated, control experiments revealed that phosphine, photocatalyst, and light were indispensable for the success of this transformation (entry 16). It was worth mentioning that the desired product **3aa** was obtained in an acceptable yield when the reaction was carried out without argon protection (entry 17). Next, decreasing the loading of the photosensitizer from 2 mol% to 1 mol% could also obtain a comparable high yield (entry 1).

With the optimized reaction conditions in hand, we first examined the scope of cyclic oximes **1** with **2a** as the coupling partner. As highlighted in Scheme 3, 2-aryl-substituted cyclobutanone oximes appear to be effective for this ring-opening pyridylation. A large variety of electron-donating groups and electron-withdrawing groups on the aromatic ring, such as Me, OMe, F,



Scheme 2. Photopromoted phosphoranyl radical-mediated ring-opening pyridylation of non-prefunctionalized cyclic oximes.



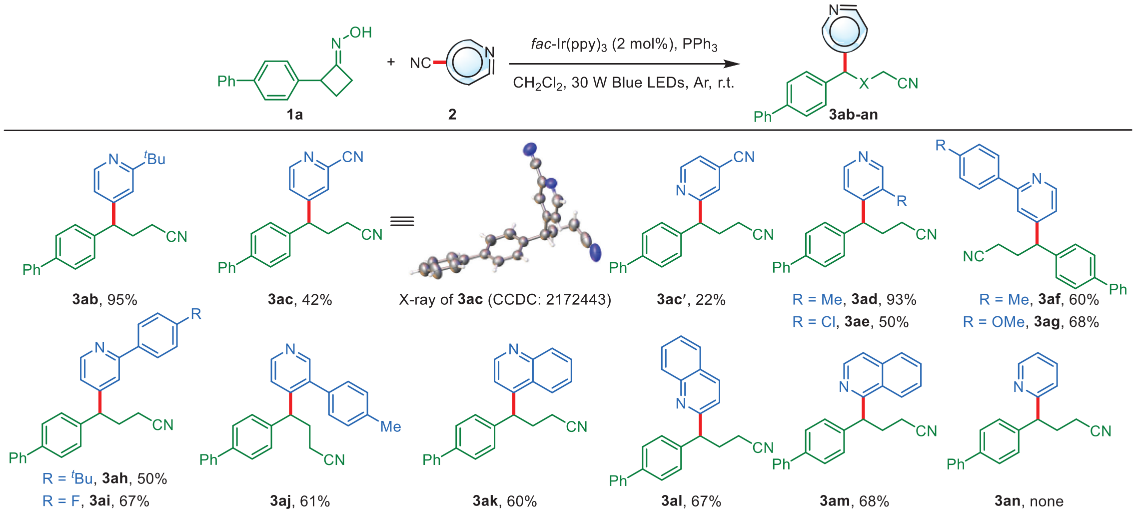
Scheme 3. Substrate scope of the cyclic oximes. Reaction conditions: **1** (0.3 mmol), **2a** (0.2 mmol), *fac*-Ir(ppy)$_3$ (2 mol%), PPh$_3$ (0.6 mmol), CH$_2$Cl$_2$ (4 mL), 30 W blue LEDs, argon atmosphere, r.t., 18 h. Isolated yield is based on **2a**.

Cl, Br, CF$_3$ and CN, were compatible with this transformation. And the position of functional groups on the aromatic ring had a negligible effect on this protocol, affording the corresponding alkylnitriles in moderate to excellent yields (**3aa–3oa**). Furthermore, 3,4-difluoro- or 3,4-methylenedioxy-disubstituted cyclobutanone oximes were used as the substrates in this protocol and successfully converted to the desired alkylnitriles in satisfactory yields (**3pa–3qa**). Cyclobutanone oxime bearing fused aromatic ring was suitable for this protocol to provide the corresponding alkylnitrile **3ra** in an excellent yield of 90%. Interestingly, benzocyclobutanone oxime was also subjected to this transformation, affording a single regioisomer benzonitrile **3sa** in a synthetically useful yield. It was worth mentioning that this photocatalytic system was also amenable to cross-coupling of cyanopyridyl radical anions with *in situ*-generated non-benzyl radicals from cyclic oximes, which gave the desired γ-pyridylated alkylnitriles **3ta–3va** albeit in relatively poor yields. To our delight, the less-strained cyclopentanone oxime also proved to be a competitive coupling partner for this ring-opening pyridylation process and afforded distally δ-pyridylated alkylnitrile **3wa** in a very good yield, while the 6-membered, 7-membered, and 8-membered cyclic oxime substrates failed to provide any desired product (**3xa–3za**).

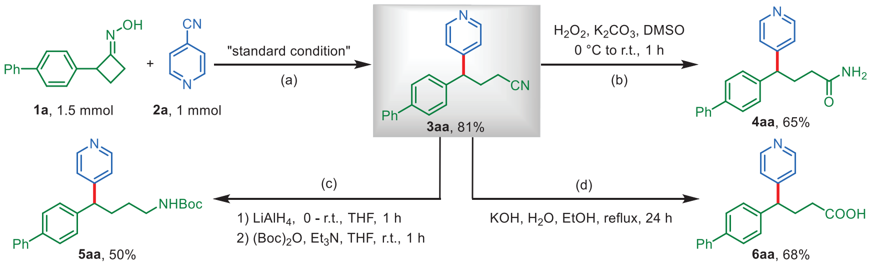
Our attention then turned to evaluating the scope of the cyanopyridines in this transformation with cyclobutanone oxime **1a** as a coupling partner (Scheme 4). A range of 4-cyanopyridines with different electronic groups at the 2- or 3-position of the pyridine ring were tolerated smoothly and gave the expected γ-pyridylated alkylnitriles in acceptable yields (**3ab–3aj**). Notably, 2,4-dicyanopyridine could be employed as a competent substrate to deliver the corresponding product **3ac** with selective coupling at the electron-poor 4-position along with the C2-cyanoalkylated products **3ac’** in 22% yield (see Supporting information for more details). The structure of **3ac** was also unambiguously confirmed by X-ray crystallographic analysis (CCDC: 2172443). In addition to cyanopyridines, other non-pyridine derivatives, such as 4-cyanoquinoline, 2-cyanoquinoline, and 1-cyanoisoquinoline, could participate well in this protocol, thus furnishing the desired γ-heteroarylated alkylnitriles **3ak–3am** in 60%–68% yields. However, no corresponding product **3an** was observed when using 2-cyanopyridine with higher reduction potential ($E_{1/2} = -2.03$ V *vs.* SCE, see Supporting information) instead of 4-cyanopyridine.

In order to validate the synthetic utility of this ring-opening pyridylation, we performed a scale-up model reaction to provide the target alkylnitrile **3aa** in 81% yield (Scheme 5a). Moreover, the cyano moiety of the resulting product is a versatile handle for a wide range of useful transformations. For example, the resulting product **3aa** was efficiently converted into γ-pyridylated amide **4aa** by using basic hydrogen peroxide in dimethyl sulfoxide (Scheme 5b). In addition, the product **3aa** could undergo selective reduction with LiAlH$_4$ and sequential amidation to obtain δ-pyridylated carbamate **5aa** in moderate yield (Scheme 5c). Alternatively, the product **3aa** could be easily transformed into γ-pyridylated carboxylic acid **6aa** *via* basic hydrolysis (Scheme 5d).

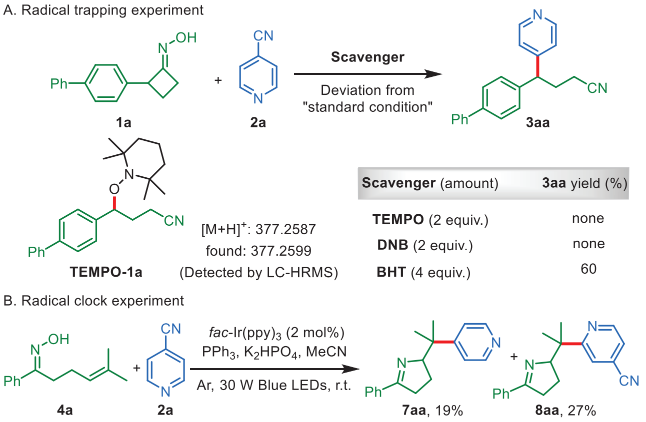
To gain a deeper understanding of the mechanism of this ring-opening pyridylation, we performed several control experiments. As shown in Scheme 6a, when 2 equiv. of the radical scavenger 2,2,6,6-tetramethyl-piperidinyloxy (TEMPO) or electron-transfer scavenger *p*-dinitrobenzene (DNB) were separately added to the model reaction system, the process was completely suppressed. And the corresponding TEMPO-trapped adduct (**TEMPO-1a**) was detected in LC–HRMS spectra. Subsequently, in the presence of the radical scavenger butylated hydroxytoluene (BHT), the model reaction was inhibited to some extent. The above results suggested that a SET/radical process might be involved in this protocol. Moreover, a radical clock experiment was performed to obtain the *ipso*-substituted product **7aa** and Minisci-type product



Scheme 4. Substrate scope of the cyanopyridines. Reaction conditions: **1a** (0.3 mmol), **2** (0.2 mmol), *fac*-Ir(ppy)$_3$ (2 mol%), PPh$_3$ (0.6 mmol), CH$_2$Cl$_2$ (4 mL), 30 W blue LEDs, argon atmosphere, r.t., 18 h. Isolated yield is based on **2**.

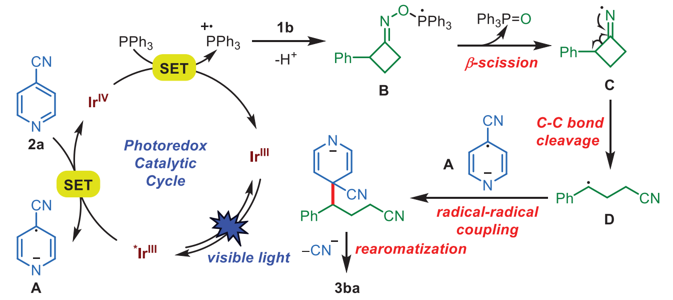


Scheme 5. Scale-up synthesis and synthetic elaboration of the product.



Scavenger (amount)	3aa yield (%)
TEMPO (2 equiv.)	none
DNB (2 equiv.)	none
BHT (4 equiv.)	60

Scheme 6. Mechanistic studies.



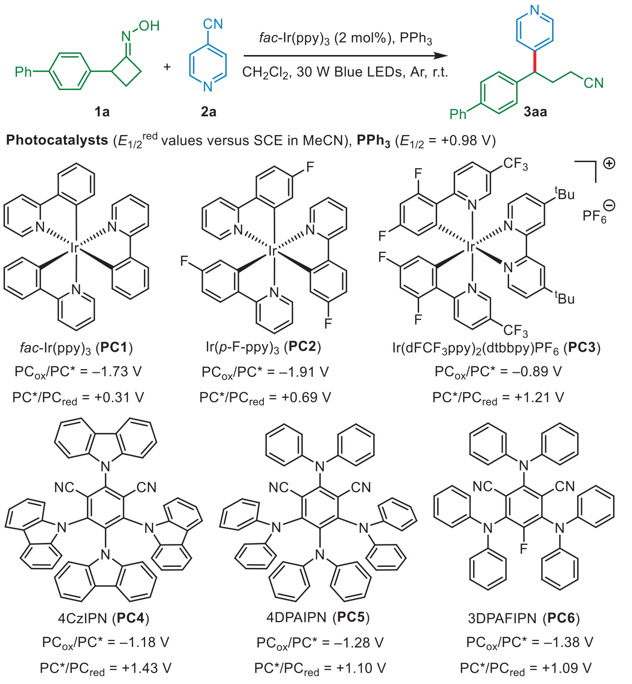
Scheme 7. Plausible reaction mechanism.

8aa by the novel imino-pyridylation reaction of γ,δ-unsaturated oxime **4a** with **2a**, further indicating the radical nature of this transformation (Scheme 6b).

Based on the above-mentioned studies and previous works [42–51], a plausible photoredox cycle was proposed as depicted in Scheme 7. Under the irradiation of blue light, *fac*-Ir(ppy)$_3$ was initially excited to yield the excited-state **fac*-Ir(ppy)$_3$ [$E_{1/2}{}^{red}$ (Ir^{IV}/*Ir^{III}) = −1.73 V *vs.* SCE], which was oxidatively quenched by 4-cyanopyridine **2a** ($E_{1/2}$ = −1.66 V *vs.* SCE) [52] to form the oxidized ground-state *fac*-Ir^{IV}(ppy)$_3$ and the corresponding cyanopyridine radical anion **A**. The resulting oxidized *fac*-Ir^{IV}(ppy)$_3$ [$E_{1/2}{}^{red}$ (Ir^{IV}/Ir^{III}) = +0.77 V *vs.* SCE] could abstract an electron from PPh$_3$ [$E_{1/2}$ = +0.98 V *vs.* SCE] since the subsequent strain-release ring-opening was thermodynamically favorable [53], affording the corresponding phosphinium radical cation and regenerating the ground-state *fac*-Ir(ppy)$_3$. Polar nucleophilic addition of the phosphinium radical cation to cyclobutanone oxime **1b** resulted in the formation of the phosphoranyl radical **B**, which underwent subsequent β-scission to afford the iminyl radical **C** and triphenylphosphine oxide. Then, the strain-relieved C–C bond cleavage of the iminyl radical **C** led to the key cyanoalkyl radical **D**. Finally, the transient cyanoalkyl radical **D** underwent the radical-radical coupling with the persistent radical anion **A** followed by elimination of the cyano anion to obtain the pyridylated alkylnitrile **3ba**.

In summary, we have developed a novel photoredox-neutral ring-opening pyridylation strategy of non-prefunctionalized cyclic oximes to provide distally pyridylated alkylnitriles as well as other heteroarylated alkylnitriles and benzonitriles under acid-, base-, and oxidant-free conditions. Furthermore, the resulting pyridylated nitriles could be scale-up synthesized and also readily converted into skeletally diverse compounds including pyridylated amide,

Table 1
Optimization of the reaction conditions.[a]



Entry	Variations from standard conditions	Yield (%)[b]
1	None	93, 89[c]
2	$PMePh_2$ instead of PPh_3	84
3	PMe_2Ph instead of PPh_3	74
4	P(*p*-tol)Ph_2 instead of PPh_3	89
5	$P(NMe_2)_3$ instead of PPh_3	79
6	$P(OEt)_3$ instead of PPh_3	70
7	$P(C_6F_5)_3$ instead of PPh_3	n.d.
8	**PC2** instead of *fac*-Ir$(ppy)_3$	60
9	**PC3** instead of *fac*-Ir$(ppy)_3$	22
10	**PC4** instead of *fac*-Ir$(ppy)_3$	21
11	**PC5** instead of *fac*-Ir$(ppy)_3$	31
12	**PC6** instead of *fac*-Ir$(ppy)_3$	8
13	DCE instead of CH_2Cl_2	91
14	MeCN instead of CH_2Cl_2	71
15	THF instead of CH_2Cl_2	58
16	no phosphine, photocatalyst, or light	n.d.
17	no argon protection	82

[a] Reaction conditions: **1a** (0.15 mmol), **2a** (0.1 mmol), photocatalyst (2 mol%), trivalent phosphorus compound (0.3 mmol), solvent (2 mL), 30 W blue LEDs, argon atmosphere (Ar), r.t., 18 h. DCE: dichloroethane. THF: tetrahydrofuran. n.d. = not detected.

[b] Yields were determined by ^1H NMR using dibromomethane as an internal standard.

[c] *fac*-Ir$(ppy)_3$ (1 mol%).

pyridylated carbamate, and pyridylated carboxylic acid. The developed protocol confirmed once more that the oxidized ground-state photocatalyst generated *via* the SET oxidation of the highly reducing excited-state photocatalyst by cyano(hetero)arenes could abstract one electron from trivalent phosphorus compound to initiate the phosphoranyl radical-mediated deoxygenation and the following transformation. Additional experiments evaluating further mechanistic research and application of this protocol are currently ongoing in our laboratory.

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.

Acknowledgments

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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.2022.107822.

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