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Catalytic, Metal-free Sulfonylcyanation of Alkenes via Visible light Organophotoredox Catalysis

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The group transfer radical addition of olefins with tosyl cyanide has been accomplished via visible light-induced organophotoredox catalysis. A diverse array of olefins is amenable to this protocol, furnishing β-sulfonyl nitriles with excellent efficiency under metal-free and redox-neutral conditions. A closed catalytic cycle is operative in this transformation, providing complementary reactivity to the classic radical chain process.

Alkenes are abundant and privileged building blocks in chemical and material industries, and recently difunctionalization of alkenes has been redeemed as a powerful strategy for building up molecular complexity from simple starting materials in an economical and straightforward fashion.[1] Besides the organometallic additions,[1a-c] radical–based additions to alkenes also represent a versatile approach to functionalize alkenes with the simultaneous and regioselective installation of two functionalities.[2] One of the most widely employed transformations of this type is the atom transfer radical addition (ATRA) reactions of olefins with alkyl halides, which enables the formation of C-C and C-X bonds in an atom- and step-economic way.[3] Recent effort has been devoted to develop efficient ATRA reactions employing safer reagents and environmental friendly and mild conditions. Seminal works by the group of Stephenson[4] and others[5] have elegantly demonstrated the application of visible light-induced photoredox catalysis[6], a powerful strategy for the development of useful transformations, to facilitate ATRA addition to alkenes with alkyl halides, advancing the ATRA technology with solar energy in a simple and efficient way. More recent advances have revealed that the ability of organic photoredox catalysts to facilitate ATRA reactions[7] as well as atom transfer radical polymerizations[8] under metal-free conditions. To the best of our knowledge, however, visible light-mediated ATRA-type group transfer radical addition of alkenes remained underexplored, although this approach would enable the net addition of two functional groups beyond halogen atoms under mild conditions.[9]

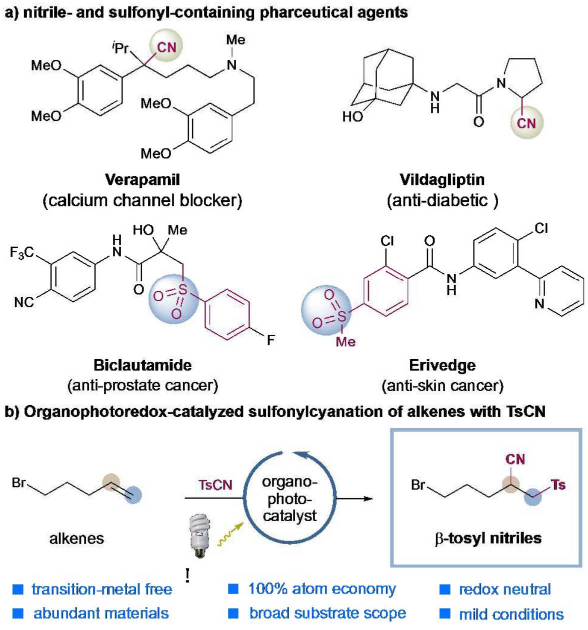

Figure 1. Organophotoredox catalyzed sulfonylcyanation of alkenes.

The alkyl nitriles[10] and sulfones[11] are important motifs prevalent in pharmaceuticals, agrochemicals, natural products, and materials. Several drugs, such as anti-diabete drug vildagliplin, anti-breast cancer drug anastrazole, calcium channel blocker verapamil, and anti-prostate cancer drug bicalutamide, all contain the key nitrile or sulfone moieties. Moreover, nitriles are versatile precursors for aldehydes, carboxylic acids, amides, and other important functionalities in organic synthesis.[12] As such, the concomitant incorporation of a cyano and sulfonyl group into one molecule, the regioselective sulfonylcyanation of alkenes, would serve as an attractive entry to the synthesis of alkyl nitriles and sulfones. Herein, we describe the organo-photoredox catalyzed regioselective sulfonylcyanation of alkenes under mild, metal-free, and redox-neutral conditions, providing facile access to a wide range of α-sulfonyl nitriles with excellent atom and step economy.

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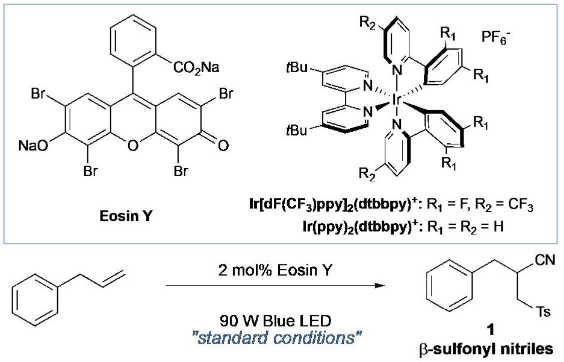
Table 1. Optimization of the reaction conditions [a]



Entry	Variation from the standard conditions	Yield of 1
1	none	90%
2	Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)$^+$, instead of Eosin Y	53%
3	Ru(phen)$_3$Cl$_2$, instead of Eosin Y	74%
4	Ir(ppy)$_2$(dtbbpy)$^+$, instead of Eosin Y	73%
5	benzophenone, instead of Eosin Y	37%
6	CH$_3$CN, instead of DMSO	19%
7	acetone, instead of DMSO	37%
8	ethyl acetate, instead of DMSO	12%
9	air, instead of N$_2$ atmosphere	23%
10	w/o cat	0
11	w/o light	0
12	w/o light, 50 °C	3%

[a]Reaction conditions: allylbenzene (0.1 mmol), TsCN (0.3 mmol), DMSO [1.2 M], Eosin Y (2 mol%), N$_2$, 90W blue LED, 20h. [b]Determined by gas chromatography with an internal standard.

Tosyl cyanide has been widely used as a cyanating agent in radical processes, nevertheless producing tosylate as chemical waste.[13] Utilizing TsCN as cyano trapping agent as well as sulfonyl radical[4a, 14] source would be advantageous to atom economy of reactions. Indeed, the groups of Fang and Barton independently described the radical addition of alkenes with TsCN in the presence of UV-light irradiation or radical initiators, proceeded by a radical chain propagation process.[15] While the requirement of an excess of alkene substrates limited the utility of these transformations in late-stage functionalizations of complex molecules. With this in mind, we sought to develop a catalytic sulfonylcyanation of alkenes via an alternative approach – a non-chain mechanism that may provide opportunities to tune the reaction processes through catalyst evaluation, and to increase the synthetic practicality by employing substrate as the limiting reagent. We reported the development, application, and the mechanistic studies of this transformation in this communication.

Evaluation of this photoredox sulfonylcyanation strategy was examined with allylbenzene as the model substrate. As shown in Table 1, irradiation with a 90W blue LED of a solution of allylbenzene and TsCN in the presence of 2 mol% Eosin Y in DMSO at room temperature delivered the desired β-sulfonyl nitrile product **1** in 90% yield (Table 1, entry 1). Other metal-based photoredox catalysts, such as Ir[dF(CF$_3$)ppy]$_2$(dtbbpy)PF$_6$, Ir(ppy)$_2$(dtbbpy)PF$_6$, and Ru(phen)$_3$Cl$_2$ that have been commonly employed in visible light induced ATRA reactions,[4, 5b] also promoted this transformation with slightly diminished efficiency (entries 2-4). However, the use of benzophenone resulted in a dramatic decrease in efficiency (entry 5). Not surprisingly, solvents played a big influence on the reaction efficiency, with polar solvent DMSO providing the best result. The employment of less polar solvents had a deleterious effect, and significantly low yields were obtained in these cases (entries 6-8). This reaction is sensitive to air, indicating a radical process involved in this transformation (entry 9). Furthermore, control experiments revealed that importance of both photocatalyst and visible light to the desired transformation, as no product formations were observed in the absence of photocatalyst or light (entries 11 and 12). Heating the reaction mixture under dark conditions couldn't promote this transformation (entry 13).

As revealed in Table 2, a diverse array of unactivated and activeted alkenes can undergo the desired regioselective sulfonylcyanation with excellent efficiency under the metal-free and redox-neutral conditions. The mild reaction conditions are compatible with a wide range of functional groups, such as ketones, esters, amides, bromides, chlorides, and boronic esters, providing a versatile platform for further synthetic manipulations (products **4**–**14**, 63%–97% yields). Remarkably, both alkyl halides and alkyl boronic esters were competent substrates, affording the desired products in high to excellent yields, where the halogen and boronic atoms remained intact under visible light irradiation (products **4**, **9**–**10**, 63%–95% yields). The resulting products can be further employed to cross-coupling reactions via transition metal catalysis, highlighting complementary ability of this photoredox technique. With respect to the terminal alkenes, 1,1-di-substituted alkenes in both cyclic and acyclic forms could undergo the radical addition smoothly with efficient formation of quaternary carbon stereocenters, and substituents at the α-position of alkenes has little effect on the reaction yields (products **11**–**12**, **16**, 63%–80% yields). Besides terminal alkenes, less reactive internal alkenes, including cyclopentenes and cyclohexenes, proved to be highly efficient in this transformation, delivering the corresponding nitriles in good efficiency and moderate stereoselectivity (products **13**–**15**, 60%–85% yields). Furthermore, naturally occurring bicyclic alkenes, such as (+)-β-pinene and (+)-α-pinene, were shown to be viable substrates, furnishing the ring-opening products **16** and **17** in 63% and 85% yields respectively, further indicating the radical nature of this protocol. Gratifyingly, the sulfonylcyanation reaction of allylbenzene in a 4 mmol scale furnished the desired product **1** in

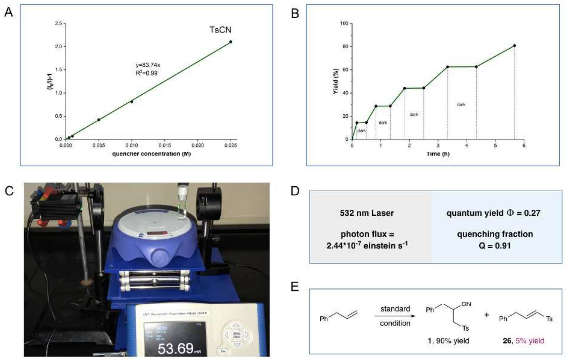


Figure 2. Mechanistic Studies. A) Quenching study. B) Light-dark Experiments; C) Laser set-up; D) Calculations of quantum yield and quenching fraction; E) Determination of byproduct.

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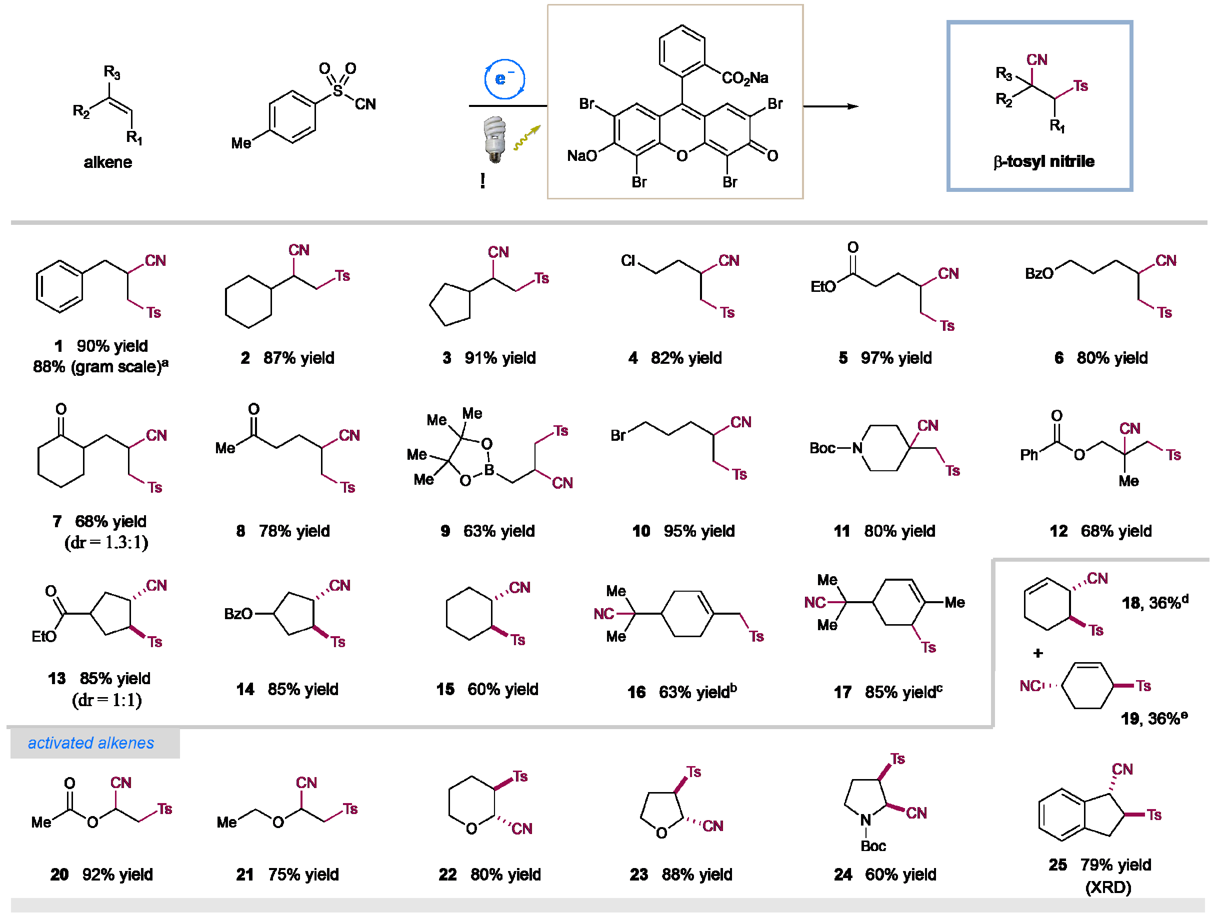

Scheme 1. Organophotoredox-catalyzed sulfonylcyanation of alkenes. All yields are isolated yields; Diastereoselectivity was determined by ^{1}H NMR analysis after isolation. ^{a}4 mmol scale; ^{b}From β-pinene; ^{c}From α-pinene; ^{d}From 1,3-cyclohexadiene.

1.05 g, 88% yield, further indicating the synthetic potential of this photoredox manifold.

Moreover, this protocol could be further applied to activated alkene system, including cyclic and acyclic vinyl ethers, affording a variety of α-oxo nitriles in good to excellent yields (products **20**–**23**, 60–92% yield). Notably, enamides such as Cbz-protected dihydropyrrole can be regioselectively functionalized, furnishing the α-amino nitriles in synthetic useful yield (product **24**, 60% yield). The resulting β-Amino nitrile is a key structural core found in a number of medicinal agents including anti-diabetic drug Vildagliplin. Additionally, we found dienes and aryl alkenes could be employed with equal efficiency, as exemplified by 1,3-cyclohexadiene and indene, to furnish the corresponding allyl and benzyl nitriles (products **18**–**19**, **25**, 72–79% yield).

To probe the mechanism of this photoredox transformation, a number of preliminary mechanistic studies were conducted. Stern–Volmer fluorescence quenching experiments clearly demonstrated that the excited state of photocatalyst Eosin Y can be quenched by TsCN (Figure 2A). Next, the light-dark experiment was conducted using alternative intervals of light and dark, and the formation of product was only feasible during periods of irradiation while no products formed during periods of dark (Figure 2B). Furthermore, we conducted the quantum yield measurements for this photochemical reaction of allylbenzene and TsCN using a commercial 532 nm laser (photon flux of $2.44*10^{-7}$ Einstein s^{-1}) (Figure 2C).[16] The calculated quantum yield value of Φ = 0.273, indicating the involvement of long radical chain progress is unlikely (Figure 2D). To further preclude the occurrence of quite short chain process, we calculated the hypothetical chain length of this reaction, with an assumption that the product was formed via radical chain progress. The hypothetic chain length of 0.283, calculated using quantum yield and quenching fraction, is less than unity, which is not suggestive of a radical chain process. Moreover, 5% yield of byproduct **26**, formed via elimination of carbon cation intermediate, can be isolated under the standard condition, suggesting that a radical-polar crossover mechanism is operative (Figure 2E).

On the basis of these observation, a proposed radical-polar crossover mechanism is depicted in Scheme 1. Irradiation of Eosin Y^{2-} with visible light could form an excited singlet state of *Eosin Y^{2-},

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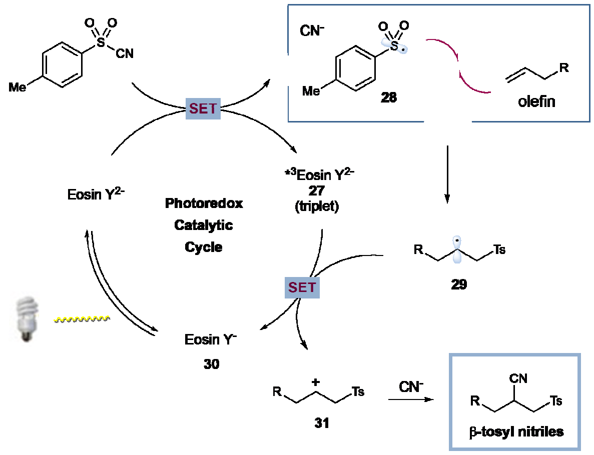

Scheme 2. Proposed mechanism.

which undergoes intersystem crossing to generate the long-lived triplet state $*^3$Eosin Y^{2-} **27**. The highly reducing excited state of **27** $[E_{1/2}(\mathrm{EY}^{*}/\mathrm{EY}^{+\bullet}) = -1.15\ \mathrm{V\ vs\ SCE\ in\ MeCN}]$[17] could facilitate the single-electron reduction of TsCN ($E^{\mathrm{red}}{}_{1/2} = -0.78$ V vs SCE in CH_3CN, see ESI for details) to generate the sulfonyl radical **28** and the oxidized catalyst **29**. The resulting electrophilic sulfonyl radical **28** should undergo facile addition to alkene, generating the first C-$S(O_2)$ bond and an alkyl radical species **30**. At this stage, we hypothesized that the alkyl radical **30** would undergo single-electron transfer (SET) with the oxidized catalyst **29** $[E_{1/2}(\mathrm{EY}^{+}/\mathrm{EY}^{+\bullet}) = +0.76\ \mathrm{V\ vs\ SCE\ in\ MeCN}]$[17] to form an alkyl cation species **31** and regenerate the ground-state eosin Y to close the catalytic cycle. Finally, rapid trapping of carbon cation **31** by the anion CN would yield the desired product.

In conclusion, we have demonstrated the regioselective sulfonylcyanation of alkenes with TsCN via visible light-mediated organophotoredox catalysis. This metal-free and redox-neutral protocol is applicable to a wide array of alkenes containing many important functional groups, incorporating both sulfone and nitrile moieties with excellent atom economy. A closed catalytic cycle is operative in this transformation, providing complementary reactivity to the classic radical chain process.

Notes and references

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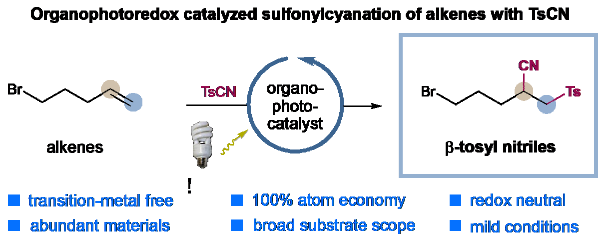
Graphic Abstract



A catalytic, redox-neutral group transfer radical addition of olefins with tosyl cyanide via visible light-induced organophotoredox catalysis has been described.