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Visible-Light Photoredox-Catalyzed α-Regioselective Conjugate Addition of Allyl Groups to Activated Alkenes

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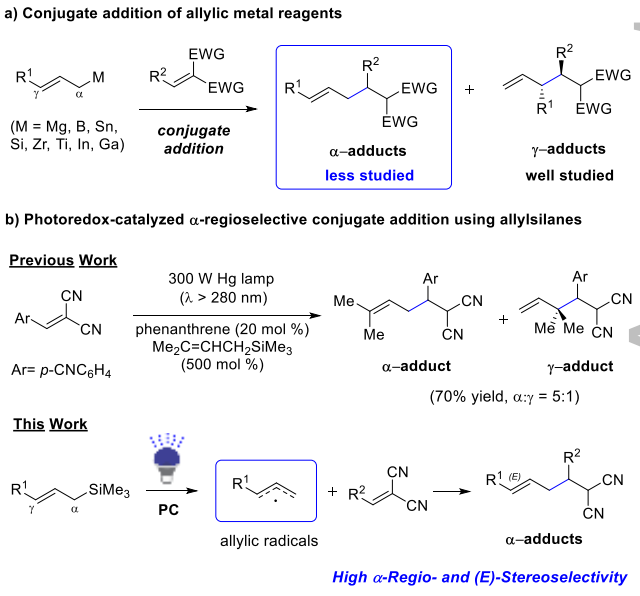
Abstract. The α-regioselective conjugate addition of allyl groups to activated alkenes is a poorly explored area of research. Herein, we report an α-adduct and (*E*)-isomer selective conjugate addition of allylsilanes to activated alkenes by visible-light photoredox catalysis. The reaction involves allylic radicals that can be generated from allylsilanes through a photoinduced single-electron transfer mechanism.

Keywords: photoredox catalysis; allylation; allylsilane; conjugate addition; α-regioselectivity

Carbon–carbon bond formation is essential for the construction of organic compounds such as natural products, pharmaceuticals, agrochemicals, and organic materials. Allylation is one of the most important methods for C–C bond formation in organic synthesis, mainly owing to its regioselectivity, stereoselectivity, and good functional-group tolerance and the easy transformation of allyl moieties into various functional groups.[1] Especially, carbonyl allylation has been well developed using a variety of allylic metal reagents and catalysts, with good regioselectivity, diastereoselectivity, and enantioselectivity being achieved.[1a-c, 1f] However, the allylation of α,β-unsaturated carbonyl, nitro, and nitrile compounds with allylic metals presents more complex regioselectivity and stereoselectivity issues. Thus, the selectivity of 1,2-addition and 1,4-addition processes is highly dependent on allylic metals and substrates.[2] To date, research on conjugate addition has mainly focused on γ-addition with controlled enantioselectivity[3] or diastereoselectivity[4] (Scheme 1 a). The selective α-addition in the conjugate addition of allyl groups has been rarely reported,[5] and remains a challenging subject. Therefore, we focused on developing a highly α-regioselective allylation in conjugate addition processes.

Allylsilanes are useful allylation reagents because of their thermal stability, inertness in oxygen and water, and good reactivity. The Hosomi–Sakurai reaction is a representative allylation using allylsilanes,[6] whose reaction mechanism involves a carbocation intermediate that is stabilized by β-silicon effect[7] as the driving force. The selective 1,4-addition of allylsilanes with high γ-addition and *anti*-selectivity has been reported.[6a, c, d, 8] In addition, allylsilanes are used as allylic radicals precursors for radical-mediated allylations. From allylsilanes, which have lower oxidation potentials than alkenes owing to the β-silicon effect,[7] allylic radicals are generated by photoinduced single-electron oxidation and desilylation. Mariano et al.[9] and Mizuno et al.[5] reported the generation of allylic radicals from allylsilanes by single-electron transfer (SET) using UV irradiation in the presence of an organic photosensitizer or donor–acceptor interaction. Interestingly, this radical-mediated conjugate addition provided α-adducts as the major product, which is the reverse regioselectivity to that observed in the Hosomi–Sakurai reaction (Scheme 1 b).



Scheme 1. Conjugate addition of allylic groups to activated alkenes

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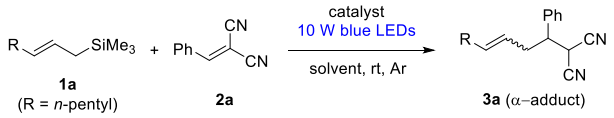
This α-selectivity is due to steric effects during the radical addition to activated alkenes. We were inspired by this allylic radical reaction for the development of an α-selective conjugate allylation. Although Mizuno and coworkers developed α-regioselective conjugate allylation using allylsilanes, this reaction was realized using a high photosensitizer loading and high-energy source (300-W Hg lamp, λ > 280 nm), and only one example with moderate selectivity (5:1) was reported. Therefore, the development of an α-selective conjugate allylation that proceeds under mild reaction conditions with high regioselectivity and good functional-group tolerance is required.

Recently, visible-light photoredox catalysis has emerged as a powerful tool in chemical transformations.[10] Wu and Glorius groups reported an efficient allylation using allylic radicals that are generated *in situ* from electron-rich alkenes by a visible photoredox-catalyzed oxidation/deprotonation process.[11] However, this reaction is limited to electron-rich alkenes. Previously, we developed a neutral silicon-based traceless activation group to generate stabilized radicals by photoinduced SET, and applied it in the visible-light photoredox-catalyzed Giese reaction of stabilized radicals with alkenes.[12] Following this, we expanded the use of our recently developed silicon-activation groups to the formation of allylic radicals. Although Wu and coworkers recently reported visible-light photoredox-catalyzed allylation of activated alkenes using allylsilanes, they did not deal with regioselective and stereoselective allylation and reported only reactions with unsubstituted allylsilane.[13] Herein, we report an α-adduct and (*E*)-isomer selective conjugate addition of allylsilanes to activated alkenes by metal-free visible-light photoredox catalysis.

Allyltrimethylsilanes are good precursors of allylic radicals. They can be easily prepared from allylic alcohols and stand out as the most atom economic allylic radical surrogates among the allylsilane family. We commenced the investigation of the photoredox-catalyzed allylation by using *n*-pentyl-substituted allyltrimethylsilane **1a** and benzalmalononitrile **2a** as model substrates in MeOH:MeCN (1:1) under irradiation with 10 W blue LEDs. The oxidation potential of allylsilane **1a** was measured by cyclic voltammetry (+1.39 V vs SCE in MeCN). Therefore, photocatalysts having high oxidation power were investigated in the initial study (Table 1, entries 1–4). The desired allylation product was obtained in 29% yield when using $Ir(dF(CF_3)ppy)_2(dtbpy)PF_6$ (Table 1, entry 3) and in 56% yield with Fukuzumi acridinium salt (Acr^+-Mes) (Table 1, entry 4). We found that the α-adduct (α:γ = >20:1) and (*E*)-isomer was the major product. This enhanced regioselectivity compared to Mizuno's allylation (α:γ = 5:1) is most likely due to mechanistic differences. Thus, Mizuno's group proposed a radical coupling mechanism of allylic radicals with anion radicals of benzalmalononitrile, whereas a radical addition mechanism of allylic radicals to benzalmalononitrile is more likely in our case.

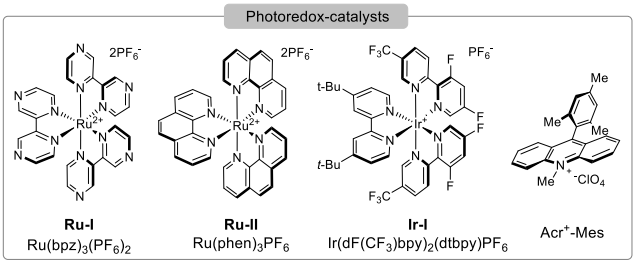
Table 1. Optimization of the reaction conditions[a]



1a (R = *n*-pentyl) **2a** **3a** (α–adduct)

entry	**1a** (mmol)	**2a** (mmol)	catalyst (mol %)	solvent (1:1) (M)	time (h)	yield (%)[b]
1	0.2	0.4	Ru-I (1.0)	MeOH:MeCN (0.1)	24	nd
2	0.2	0.4	Ru-II (1.0)	MeOH:MeCN (0.1)	24	nd
3	0.2	0.4	Ir-I (1.0)	MeOH:MeCN (0.1)	24	29
4	0.2	0.4	Acr^+-Mes (1.0)	MeOH:MeCN (0.1)	24	56
5	0.2	0.4	Acr^+-Mes (2.5)	MeOH:MeCN (0.1)	24	52
6	0.2	0.4	Acr^+-Mes (1.0)	MeOH (0.1)	24	50
7	0.2	0.4	Acr^+-Mes (1.0)	DCE (0.1)	24	32
8	0.4	0.2	Acr^+-Mes (1.0)	MeOH:DCE (0.1)	24	42
9	0.4	0.2	Acr^+-Mes (1.0)	MeOH:DCE (0.2)	24	54
10	0.4	0.2	Acr^+-Mes (1.0)	MeOH:DCE (0.2)	60	77
11	**0.6**	**0.2**	**Acr^+-Mes (1.0)**	**MeOH:DCE (0.2)**	**32**	**79(77)[c]**
12[d]	0.6	0.2	Acr^+-Mes (1.0)	MeOH:DCE (0.2)	32	nd
13	0.6	0.2	-	MeOH:DCE (0.2)	32	nd
14[e]	0.6	0.2	Acr^+-Mes (1.0)	MeOH:DCE (0.2)	32	55

Photoredox-catalysts



Ru-I $Ru(bpz)_3(PF_6)_2$ **Ru-II** $Ru(phen)_3PF_6$ **Ir-I** $Ir(dF(CF_3)bpy)_2(dtbpy)PF_6$ Acr^+-Mes

[a]Reaction conditions: **1a**, **2a**, catalyst, solvent with 10 W blue LEDs (452 nm) irradiation at room temperature under argon atmosphere in pressure tube. [b]Yield determined by ^1H NMR (internal standard: methyl benzoate). [c]Isolated yield, >20:1 α:γ, 16:1 *E*:*Z* [d]Under air atmosphere. Acr^+-Mes (9-mesityl-10-methylacridinium perchlorate), nd = not detected.

We then proceeded to further optimize the reaction conditions by using Fukuzumi acridinium salt as a photocatalyst and surveyed the catalyst loading and solvents (Table 1, entries 5–8, see SI for details); however, no significant improvement in yield was observed. We found 100% conversion of allyltrimethylsilane **1a** into the corresponding allylation products and byproducts, the latter mixtures of alkenes being generated by the protonation of the allylic radical. Therefore, we changed the ratio of allyltrimethylsilane **1a** and benzalmalononitrile **2a** from 1:2 to 2:1 to improve the yield. Further test of solvents and reaction time resulted in the 1:1 mixture of MeOH and DCE with long reaction times providing the best yield (Table 1, entry 10). However, the reaction time of 60 h was inefficient. Upon increasing the amount of **1a** to 3 equiv, the reaction time decreased dramatically to 32 h, with a modest increase in yield (Table 1, entry 11). We performed a control experiment in the absence of photocatalyst and light to check the dependency of the reaction on both parameters. In either case, the desired product was not observed, which strongly indicates that the presence of light and a photocatalyst is required for the reaction to occur.

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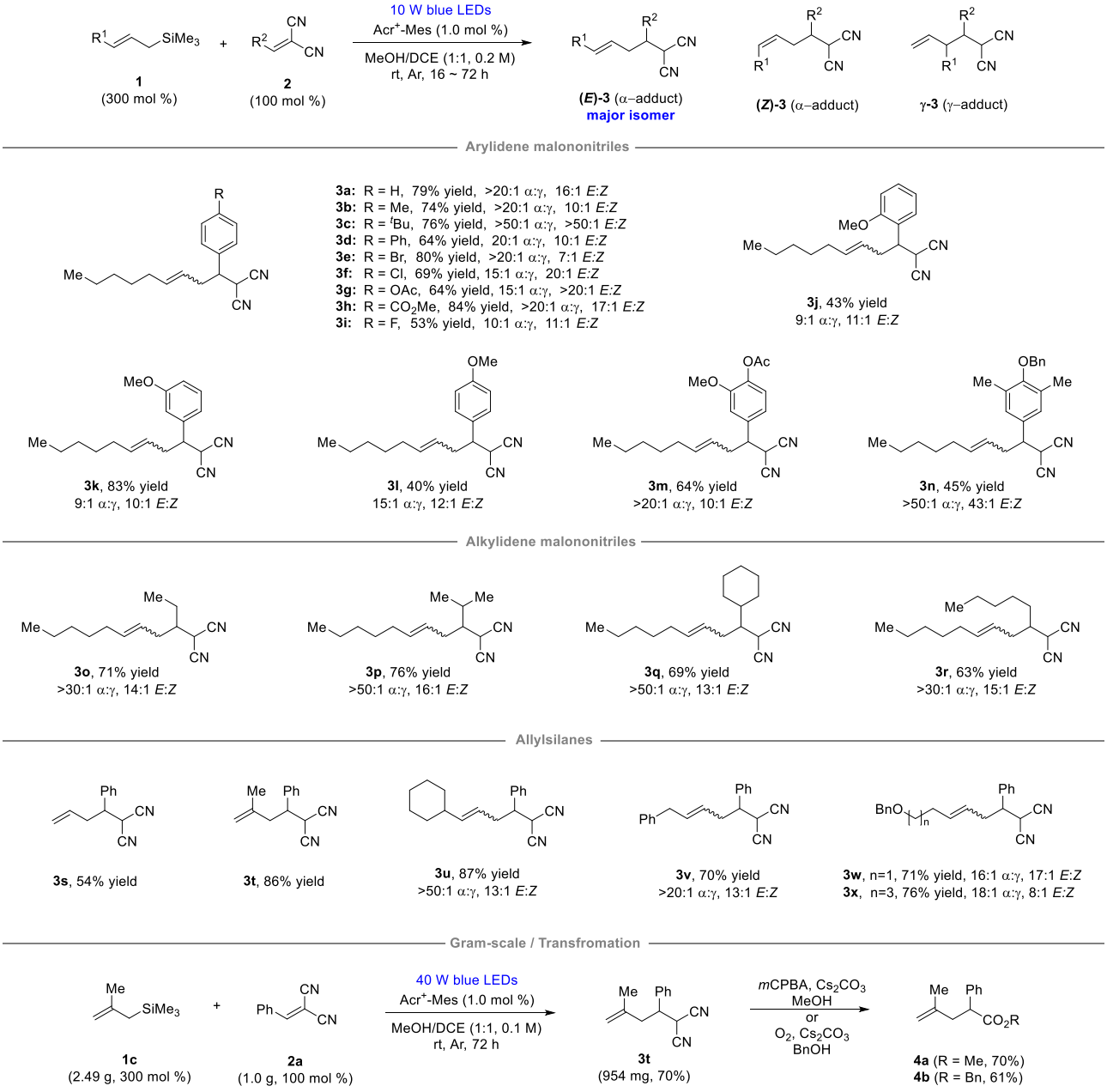
Table 2. Substrate scope, gram-scale reaction, and dehomologation of the allylation product[a]



[a]Reaction conditions: Table 1, entry 11, Isolated yield.

With the optimized conditions, we investigated the scope of several arylidene and alkylidene malononitriles (Table 2). Arylidene malononitriles (**2a**–**2n**) bearing electron-withdrawing and electron-donating substituents performed well in the reaction, providing good to moderate yields with high regioselectivities and stereoselectivities (α:γ = 9:1 ~ >50:1, E:Z = 7:1 ~ >50:1) (**3a**–**n**). A variety of functional groups were tolerated in the arylidene malononitrile substrates; methyl, tert-butyl, and phenyl substitutions delivered good yields with excellent regioselectivities and stereoselectivities (**3b**–**3d**, α:γ = >20:1, E:Z = 10:1 ~ >50:1). Photolabile halogen functional groups such as bromo, chloro (**3e**, **3f**), and strong electron-withdrawing groups (**3h**–**3i**) were well tolerated under the developed reaction conditions and afforded good to excellent regioselectivities and stereoselectivities. Allylation with arylidene malononitriles containing ortho-, meta-, and *para*-substituted methoxy functional groups performed well, with the meta-methoxy-substituted benzalmalononitrile producing the best result, whereas *ortho*- and *para*-methoxy substituents gave moderate yields and good selectivity. These results show that the presence of an electron-donating group in *ortho* and *para* position positions of arylidene malononitriles decreases the reactivity. Arylidene malononitriles containing disubstituted and trisubstituted arenes also underwent the reaction, and gave the desired product

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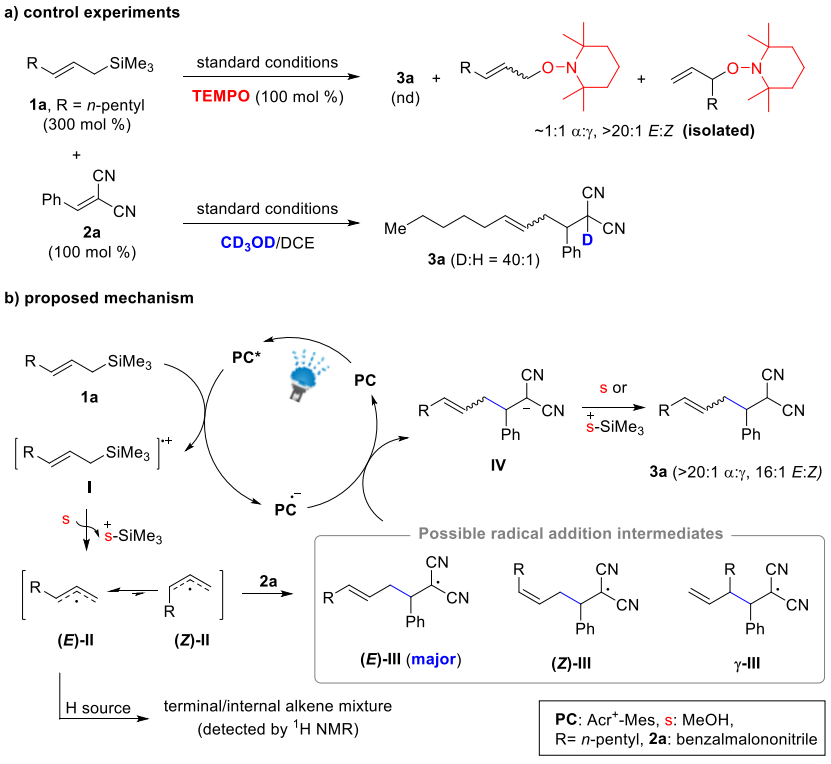
in moderate to good yields with exclusive formation of the α-adduct. Moreover, alkylidene malononitriles (**2o**–**2r**) reacted to provide the desired allylation products in good yields and high regioselectivities (**3o**–**3r**). In general, arylidene and alkylidene malononitriles bearing bulky substituents showed excellent regioselectivities. We next explored a variety of allylsilanes (**1b**–**1g**) to evaluate the generality of the allylation reaction. The allylation of various silanes (**1b**–**1g**) with benzalmalononitrile **2a** provided the desired product with moderate to good yields and good regioselectivities. Although allyltrimethylsilane **1b** has a high oxidation potential (E = 1.61 V vs. SCE), Fukuzumi salt (Acr$^+$-Mes), having an oxidation potential E = 2.06 V vs SCE, was able to oxidize **1b** to generate the allylic radical. Methallyltrimethylsilane **1c** was more efficient as an allyl radical source than allyltrimethylsilane **1b**, providing good yields. In addition, we performed a gram-scale reaction of **1c** with **2a** to investigate the possibility of a scale-up. The scale-up reaction required high-power blue LEDs (40 W) and longer reaction times (72 h) and resulted in a slight decrease in the yield of allylation product **3t**. The malononitrile group in **3t** was transformed into esters (**4a, 4b**) by oxidative dehomologation.[11a,14] Cyclohexyl-substituted allylsilane **2d** provided the corresponding coupling product **3u** with excellent yield and regioselectivity. Phenyl- and benzyl-protected alcohols in allylsilanes were tolerated in the present reaction conditions. Unfortunately, we have not been able to expand on allylation with less activated alkenes such as acrylonitrile and methyl acrylate under optimized conditions.

Because we considered that the occurrence of a radical mechanism is important to achieve an α-addition selective allylation, we decided to confirm that allylsilanes were oxidized by the photocatalyst, and that the allyl radical intermediates were produced under the reaction conditions. We determined the oxidation potential of allylsilanes by cyclic voltammetry. The measured oxidation potential was less than 1.6 V, which means that the excited photocatalyst (Acr$^+$-Mes*) can oxidize allylsilanes. In addition, Stern–Volmer fluorescence quenching experiments were conducted with allylsilane **1a** and benzalmalononitrile **2a**. It was found that only allylsilane **1a** quenched the excited photocatalyst, whereas benzalamalononitrile **2a** did not (detailed in SI). This result reveals that a SET mechanism between allylsilane **1a** and the photocatalyst occurs under blue LEDs irradiation. We were able to isolate an allylic radical–TEMPO complex (α:γ = ~1:1, E:Z = > 20:1) in a radical trapping experiment using TEMPO. This result provides strong evidence that the photocatalyzed allylation involved an allylic radical intermediate (Scheme 2 a).

Based on our control experiments and the literature,[5,9] we propose the reaction mechanism depicted in Scheme 2 b. Initially, the single-electron oxidation of allylsilane **1a** by excited Fukuzumi acridium (Acr$^+$-Mes*) generates an allylic cation radical **I**, which is desilylated by a MeOH molecule to produce allylic radical **II**. Substituted allylic radicals are in equilibrium with (*E*)-isomer **(*E*)-II** and (*Z*)-isomer **(*Z*)-II**, and **(*E*)-II** is the thermodynamically predominant isomer.[15]



Scheme 2. Control experiments and proposed reaction mechanism

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The less hindered terminal position of **(*E*)-II** is then added to benzalamalononitrile **2a** to provide α-adduct **(*E*)-III** as the major product. As minor products, α-adduct **(*Z*)-III** and γ-adduct **γ-III** are generated from (*Z*)-allylic radical **(*Z*)-II**, and by addition of allylic radical **II** at the γ-position, respectively. Moreover, allylic radicals **II** are converted into alkenes by the proton source as byproducts, which were detected by ^{1}H NMR spectroscopy. Adducts **III** are converted into anion intermediate **IV** by single-electron reduction from the reduced photocatalyst (Acr•-Mes), and the photocatalyst is regenerated. Finally, the desired product **3a** is produced by the protonation of anion **IV** by methanol. We confirmed the proton source by performing an isotope labeling experiment using deuterated methanol (CD_3OD) (Scheme 2 a). In addition, we performed on–off experiments and determined quantum yield (Φ = 0.0077) to verify the proposed mechanism (detailed in SI).[16]

In conclusion, we have developed a visible-light photoredox-catalyzed regioselective and stereoselective allylation of activated alkenes. This reaction proceeds under mild conditions, i.e., metal- and additive-free photoredox catalysis at room temperature and tolerates various functional groups. Especially, this conjugate allylation provides high α-regioselectivities and (*E*)-stereoselectivities and moderate to good yields. The investigation of the mechanism provides evidence for a photoinduced allylic radical-mediated reaction mechanism. These results present a new strategy for the α-allylation reaction using allylsilanes.

Experimental Section

General procedure for conjugate addition of allylsilanes 1 to alkenes 2

To a re-sealable pressure tube (13 × 100 mm) with a magnetic stir bar was charged with **1** (0.6 mmol, 3.0 equiv), **2** (0.2 mmol, 1.0 equiv) and 9-mesityl-10-methylacridinium perchlorate (Acr$^+$-Mes) (0.82 mg, 0.002 mmol, 1.0 mol %) under argon atmosphere. The reaction mixture was dissolved by degassed methanol and DCE (1:1, 1 mL, 0.2 M for **2**). The mixture was irradiating with 2 × 5W blue LEDs using our customized milligram scale reaction set up [as shown in Figure S2 (a)] under constant stirring condition at room temperature (20 ~ 30 °C) for 16 ~ 72 h. After finishing the stipulated time, the solvent was removed under reduced pressure and residue was purified by flash column chromatography on silica gel to afford the corresponding allylation product **3**.

Acknowledgements

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Visible-Light Photoredox-Catalyzed α-Regioselective Conjugate Addition of Allyl Groups to Activated Alkenes

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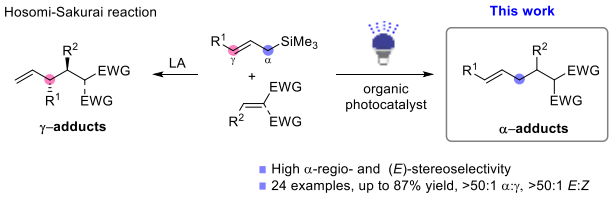

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