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Alkylarylation of alkenes with arylsulfonylacetate as bifunctional reagent *via* photoredox radical addition/Smiles rearrangement cascade

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ABSTRACT

The radical difunctionalization of alkenes with sulfonyl bifunctional represents a powerful and straightforward approach to access functionalized alkane derivatives. However, both the mechanistic activation mode and the substrate scopes of this type of radical difunctionalizations are still limited. We demonstrate herein a modular photoredox strategy for the difunctionalization of alkenes, employing arylsulfonyl acetate as the bifunctional reagent. This approach involves a radical addition/Smiles rearrangement cascade process, offering a robust alternative for the synthesis of valuable γ,γ-diaryl and γ-aryl esters. A complementary oxidative bifunctional reagents activation mode is identified to govern the radical cascade reactions, facilitating the simultaneous incorporation of aryl and carboxylate-bearing alkyl groups into the alkenes with excellent diastereoselectivity. Noteworthy features of this method include mild reaction conditions, organophotocatalysis, high atom- and step-economy, excellent functional group compatibility and great structural diversity.

Difunctionalization of alkenes provides a robust tool for converting simple alkenes into complex molecules, which is of high interest and challenging from both academic and industrial perspectives [1–7]. The simultaneous formation of multiple bonds using just one bifunctional reagent has become state-of-the-art in achieving this target [8–17]. Numerous examples of bifunctional reagents-mediated difunctionalization of alkenes have been disclosed in recent decades due to the rapid development of electrocatalysis [18–20] and photocatalysis [18,21–30]. Among them, the sulfonyl bifunctional reagent stands out for enabling alkene difunctionalization *via* a radical-induced functional group migration process, proving to be one of the most efficient approaches [31–34]. In general, those reactions could be categorized mechanistically into the following two types: (1) Single-electron oxidation of the electron-rich alkenes to generate the key active radical cation species. Then, bifunctional reagents nucleophilic attach the radical cation and engage in the following intramolecular Smiles rearrangement to achieve the difunctionalization (Fig. 1A) [35–37]. (2) Instead of alkene oxidation, single-electron reduction of sulfonyl alkyl bromide bifunctional reagents yields the electrophilic radical species, which subsequently undergo radical addition to styrenes. Then, the benzyl radical goes through *ipso* attack, leading to 1,4-functional group migration to deliver the difunctionalized products (Fig. 1B) [32,33,38–46]. Pioneered by Stephenson and Zhu, various efficient desulfonylative difunctionalizations of alkenes with these activation modes have been reported for incorporating amino or alkyl units and (hetero)aryl units across alkenes. In addition, an asymmetric radical difunctionalization was also realized by the Nevado group, employing chiral sulfinamide as the auxiliary [47,48]. Nevertheless, both the mechanistic activation mode and the substrate scopes in alkene radical difunctionalizations are still limited. More easily accessible and functional group-compatible bifunctional reagents, as well as complementary mechanistic activation modes, are still highly desirable.

Recently, arylsulfonylacetate has been independently developed as a bifunctional reagent for the difunctionalization of unsaturated carbon-carbon bonds by both our research group [49,50] and others [51]. As an extension of our ongoing exploration into radical chemistry [52–57], we conceived a strategy employing arylsulfonylacetate as a bifunctional reagent for the difunctionalization of styrenes to access γ,γ-diaryl and γ-aryl ester compounds, which are frequently encountered in bioactive compounds and nat-

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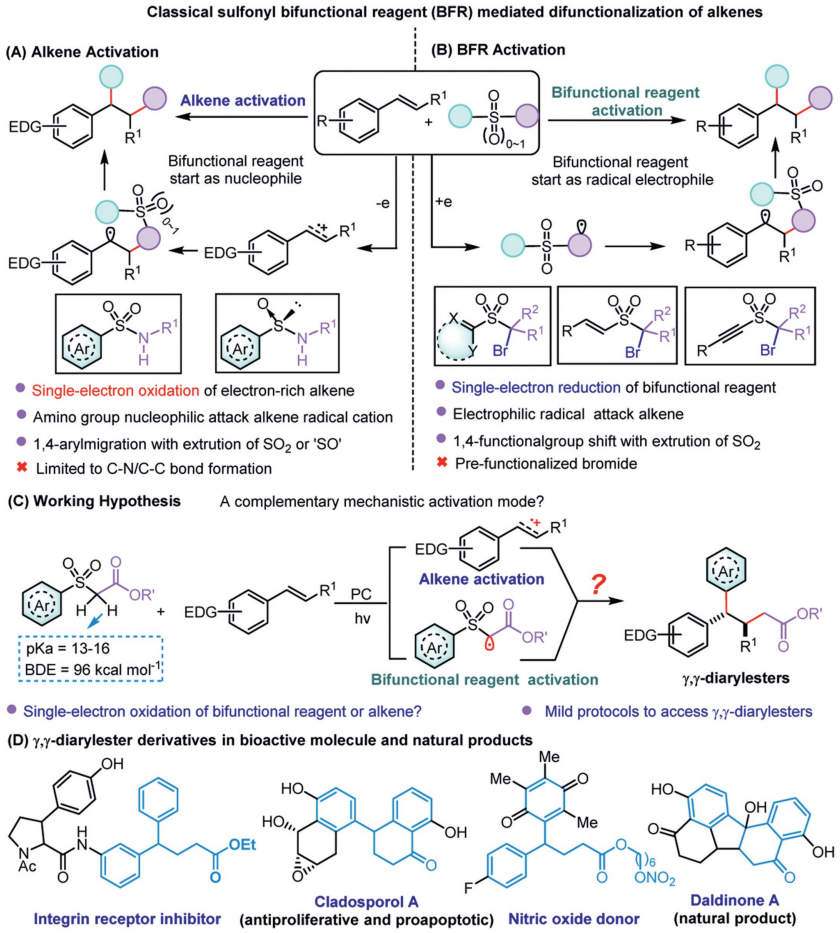


Fig. 1. Previous work on sulfonyl bifunctional reagent mediated difunctionalization of alkenes and our working hypothesis.

ural products (Fig. 1D) [58–60]. Subsequently, a crucial mechanistic question arose, specifically concerning the activation mode that would govern the anticipated transformation (Fig. 1C). This consideration holds significant implications for both the substrate scope and the overall efficiency of the proposed method. Theoretically, the acidic proton (p*K*a 13–16) in the active methylene site would be readily deprotonated under basic conditions to give a nucleophilic enolate, which would trap alkene radical cation *via* alkene activation mode. Conversely, the bond dissociation energy of the C–H bond at the active methylene site falls within the range of approximately 96 kcal/mol, rendering selective hydrogen atom abstraction (HAA) thermodynamically feasible [50]. Then, the generation of a highly electrophilic radical species, facilitated through either a suitable hydrogen atom abstraction (HAA) or a single electron oxidation process appears operational to initiate the difunctionalization *via* bifunctional reagent activation mode.

To test the feasibility of our hypothesis and inspire the future development of novel bifunctional reagents for alkene difunctionalization, we selected arylsulfonyl acetate **1aa** and *trans*-anethole **2aa** as model substrates for our initial investigations. To our delight, when 4CzIPN was employed as the photocatalyst and K_3PO_4 as the base, the desired γ,γ-diaryl ester **3aa** could be formed in 76% yield with excellent diastereoselectivity (Fig. 2A). Then, cyclic voltammetry (CV) measurements were carried out to elucidate the oxidative process underlying this photocatalytic event. Compound **1aa** displayed the first distinct oxidation at $E_{p/2} = +\ 0.68$ V (*vs.* SCE in CH_3CN) in the presence of base, while that of *trans*-anethole **2aa** was determined to be $E_{p/2} = +\ 1.30$ V (*vs.* SCE in CH_3CN) which is consistent with the reported data (Fig. 2B) [61,62]. Given the redox potential of the photocatalyst 4CzIPN ($^*E_{1/2\ (PC^*/PC^-)} = +1.35$ V *vs.* SCE in MeCN) enables the thermodynamically favorable oxidation of both reactants, further experiments were performed. Stern-Volmer fluorescence quenching experiments revealed a notably faster quenching rate for **1aa** in the presence of a base compared to **2aa**, while pure **1aa** is entirely unresponsive (Fig. 2C). Moreover, upon the introduction of 2.0 equiv. of the radical scavenger TEMPO into the reaction mixture, the reaction was entirely inhibited, concomitant with the detection of TEMPO adduct **5** (Fig. 2D). These results demonstrate that the crucial radical species derived from bifunctional reagent **1aa** actively participated in this cascade transformation. As anticipated, $Ir(ppy)_3$ possessing a relatively low oxidative potential ($E_{Ir(II)/Ir(III)^*} = +0.31$ V) [63,64] exhibited completely inertness in the reaction. While $Ru(bpy)_3Cl_2$ with an oxidation potential positioned between those of **1aa** and **2aa** was employed as the photocatalyst, the desired product **3aa** can be obtained with a 72% yield. Intriguingly, switching the photocatalyst to Mes-Acr^+-$MeClO_4^-$ or TPT which were commonly used for alkene radical cation species generation [65–70], resulted in no detection of **3aa** (Fig. 2E). Taken together, these data provide support for single-electron oxidation of bifunctional reagent **1aa** to initiate this radical difunctionalization reaction.

Further optimization of reaction parameters, including the photocatalyst, base, and solvent, led us to identify the optimal conditions to achieve an 84% yield of product **3aa** with >20:1 diastereoselectivity (For details, see Supporting information). Control experiments indicated that the photocatalyst, light irradiation, and $K_3PO_4{\cdot}3H_2O$ were all ciritical to the cascade transformation, as the absence of any of these components resulted in no detection of

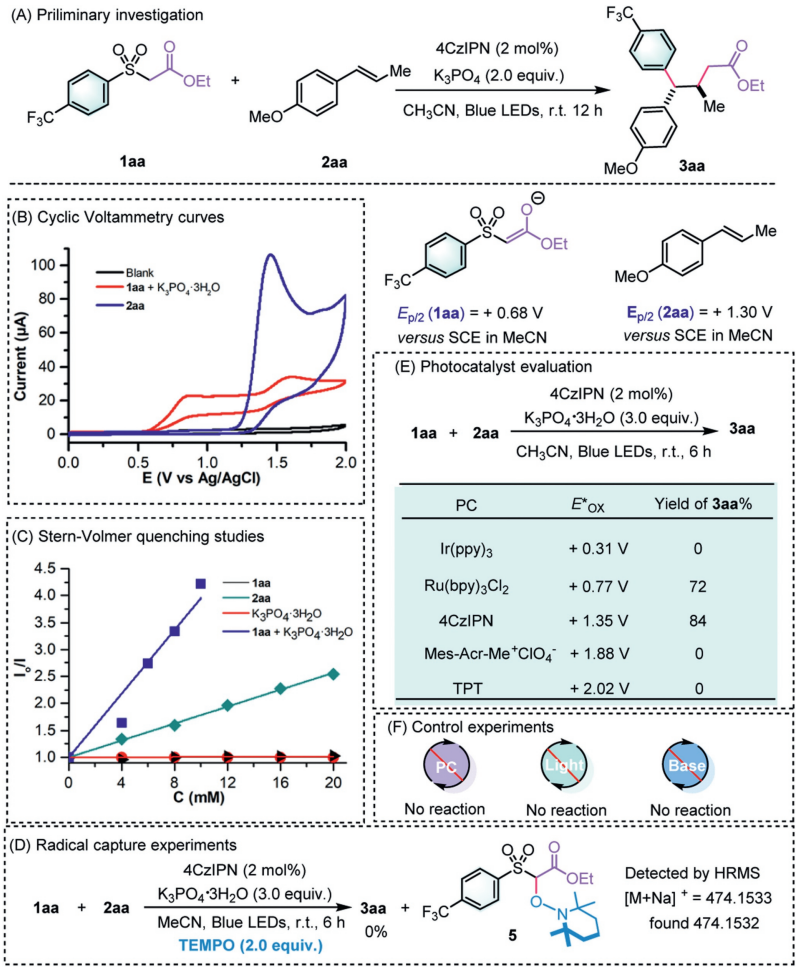
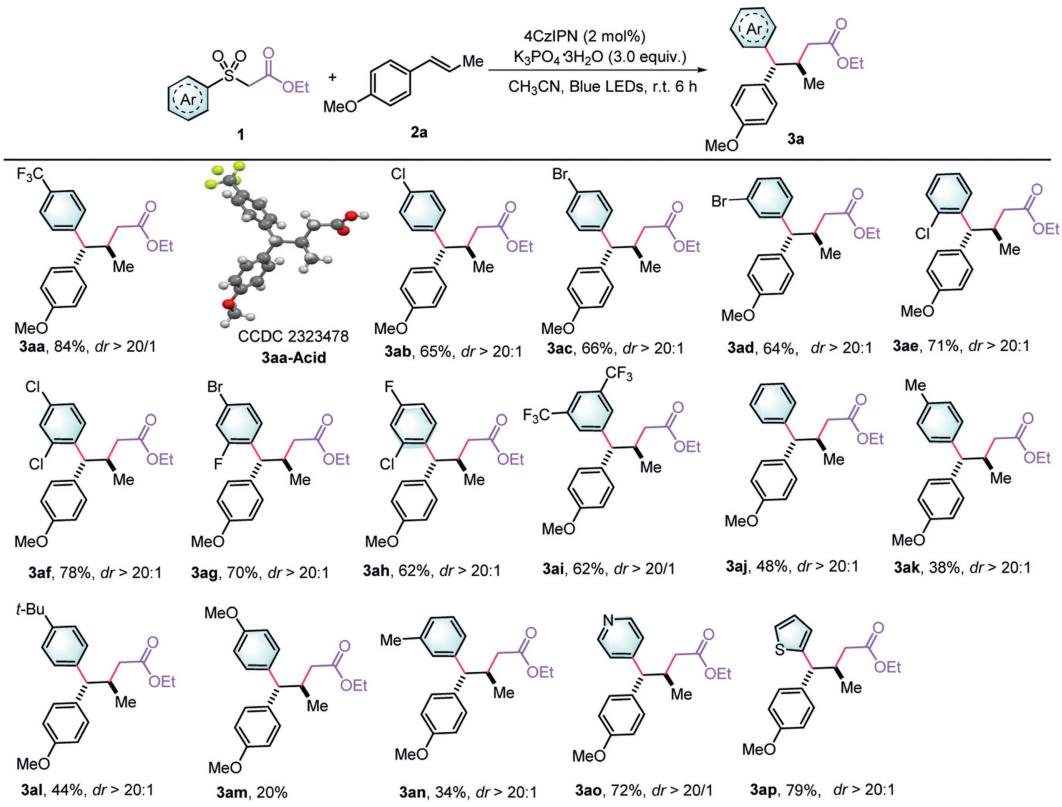


Fig. 2. Preliminary investigation and proof of the concept.

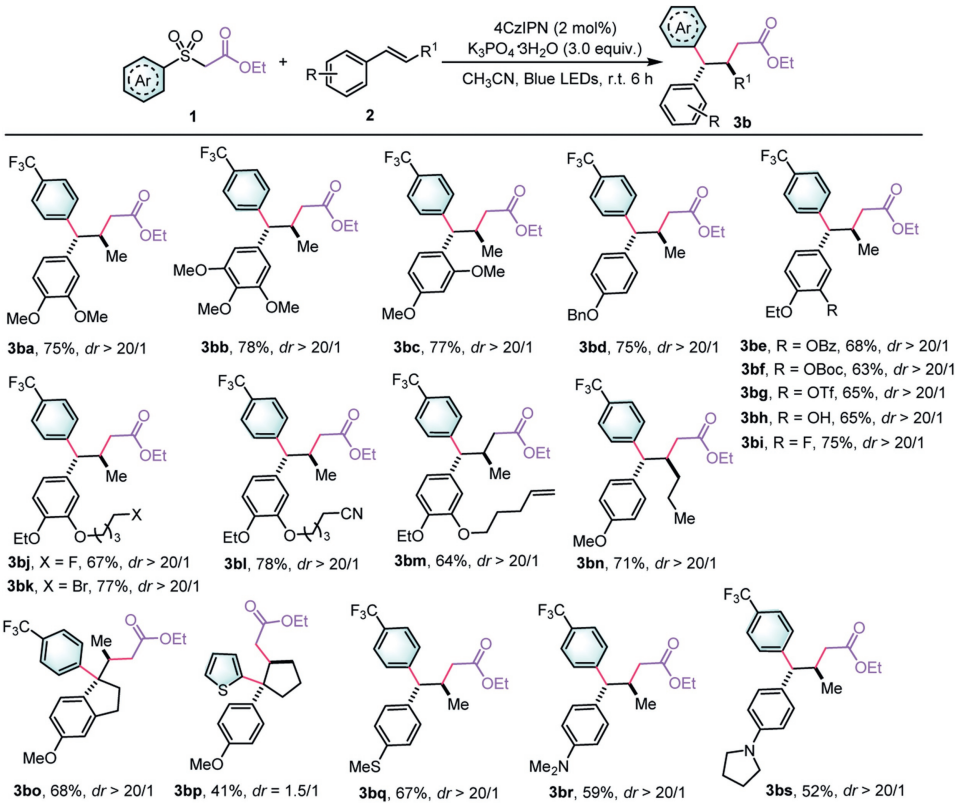
the desired product (Fig. 2F). To the best of our knowledge, this reaction not only presents a novel alternative for the consecutive formation of multiple C–C bonds, leading to the synthesis of γ,γ-diaryl esters but also introduces a complementary mechanistic activation mode for the sulfonyl bifunctional reagent-mediated difunctionalization of alkenes.

With the optimal conditions in hand, our initial focus was on exploring the structural diversity concerning the migrating aromatic ring in arylsulfonylacetate bifunctional reagents (Scheme 1). Phenyl rings containing various substituents, including electron-donating groups such as methyl, *tert*–butyl, and methoxy, as well as electron-withdrawing groups (halogens), were all found to be compatible to provide the corresponding γ,γ-diarylesters (**3aa**-**3ap**) in 20% to 84% yields with excellent diastereoselectivity. It is worth noting that the structure of **3aa** was ambiguously confirmed by X-ray analysis of the corresponding acid derivative (CCDC:2323478). Generally, bifunctional reagents featuring electron-withdrawing groups on the phenyl rings exhibited more efficient reactions with alkenes, resulting in higher yields of the corresponding γ,γ-diarylesters compared to those with electron-donating groups. This phenomenon was attributed to the favorable π-π stacking interaction between the electron-deficient aryl ring of the bifunctional reagent and the electron-rich PMP rings [71]. This interaction is believed to contribute to lowering the energy barrier for the aryl migration process [36]. Significantly, functional groups like halides, amenable to subsequent derivatization, were well-tolerated, yielding the corresponding products in good yields. Furthermore, heteroaryl groups such as pyridine and thiophene were found to be compatible under the standard conditions, yielding γ,γ-diarylesters **3ao** and **3ap** in moderate yields.

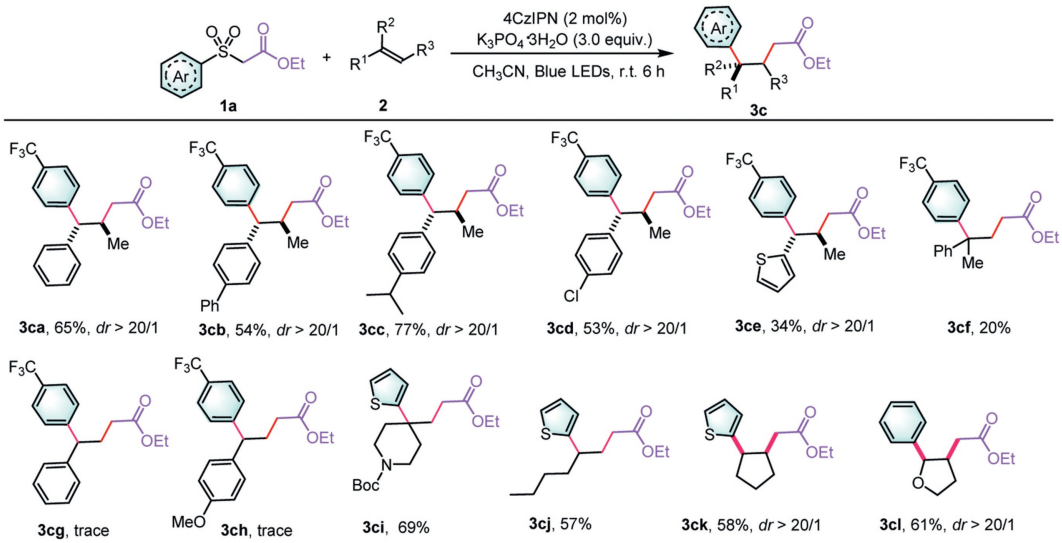
We extended our investigation to explore the diversity of substituents in the electron-rich internal alkenes (Scheme 2). Both mono- and multi-substituted alkoxy phenyl rings in olefins resulted in the production of the target products with high yields (**3ba-3bd**). Notably, various protecting groups for the phenol moiety, including benzoyl, Boc, and triflate, were all compatible under the standard conditions (**3bd-3bg**). Particularly, the free hydroxyl group remained intact, yielding the corresponding difunctionalized product **3bh** in a moderate yield of 65%. Furthermore, alkyl chains containing halide and cyano functionalities were also well-tolerated under the standard conditions, affording the products **3bj-3bl**. It is noteworthy that an electron-rich styrene containing a terminal alkene chain exhibits pronounced selectivity to give the expected product **3bm** in moderate 65% yield. This selectivity can be attributed to the favorable π-π stacking interactions within the electron-deficient aromatic phenyl ring of **1a** and the electron-rich alkene **2m**. Increasing the length of the alkyl chain in the internal alkene showed negligible impact on the transformation (**3bn**). Trisubstituted internal alkene also proved to be effective substrate under the standard conditions, yielding the difunctionalized product **3bo** bearing a quaternary center with moderate yield and diastereoselectivity. Internal alkene was also examined, but poor reactivity and diastereoselective was observed (**3bp**). This was attributed to the steric hindrance of the cyclic internal alkene for both the radical addition and aryl migration process. Furthermore, methylthiol and dialkylamino groups were proven to be suit-



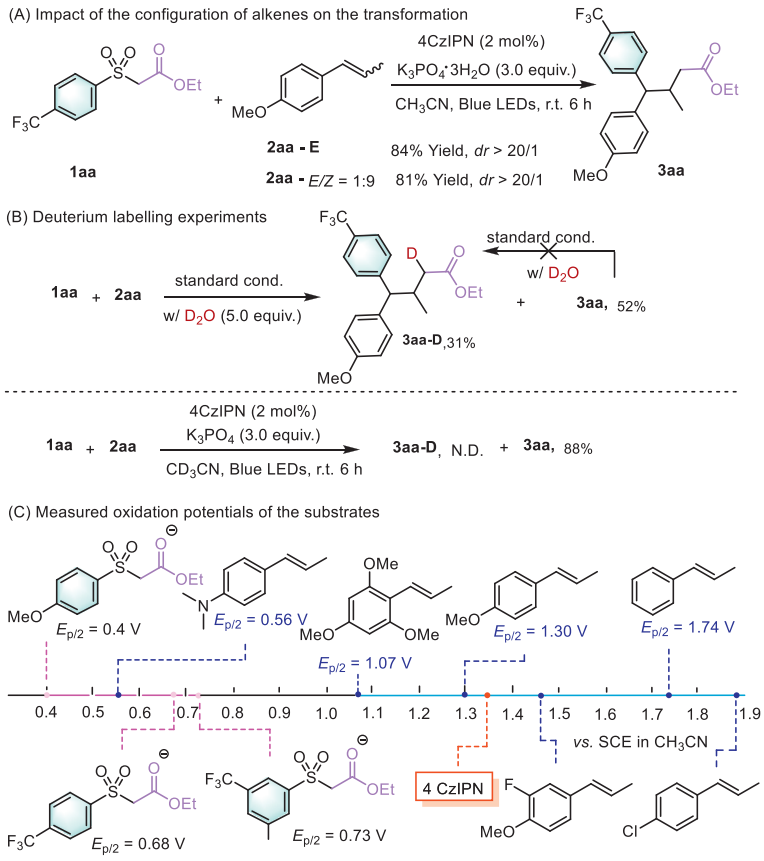
Scheme 1. Variation of the arylsulfonylacetates: reactions were carried out on 0.2 mmol scale and isolated yields are given. *dr* was determined by crude NMR spectroscopy, for details see Supporting information.



Scheme 2. Variation of the electron-rich alkenes: reactions were carried out on 0.2 mmol scale and isolated yields are given. *dr* was determined by crude NMR spectroscopy, for details see Supporting information.



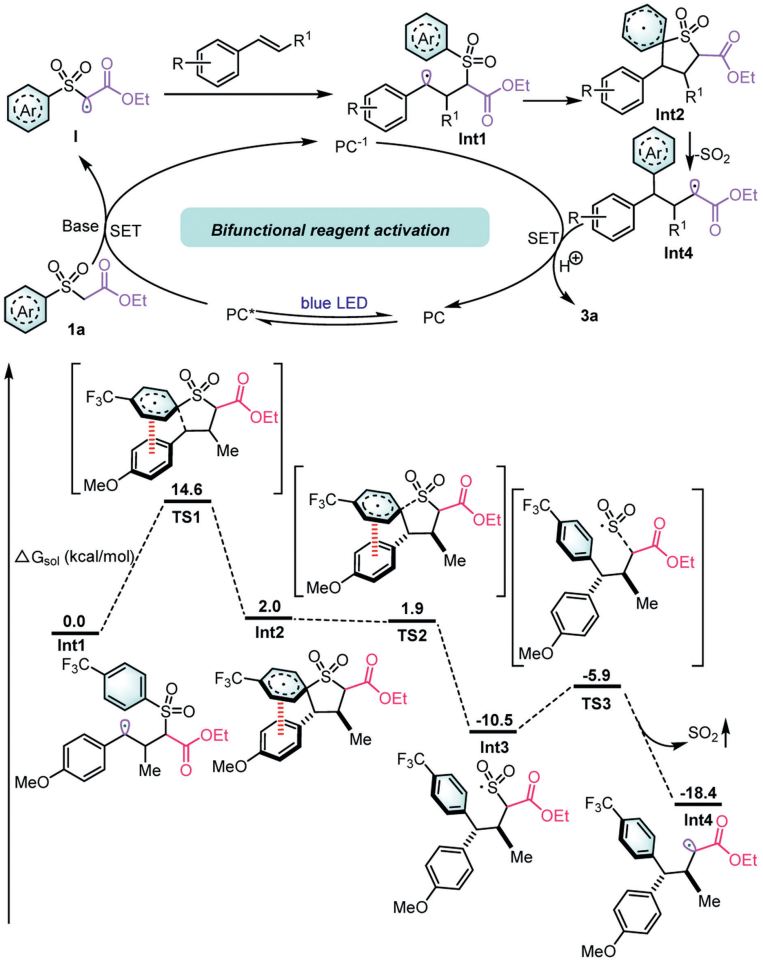
Scheme 3. Variation of the electron-rich alkenes: reactions were carried out on 0.2 mmol scale and isolated yields are given. *dr* was determined by crude NMR spectroscopy, for details see supporting information.



Scheme 4. Mechanism investigation.

able electron-donating groups under this photoredox conditions, furnishing the corresponding γ,γ-diarylesters (**3bq-3bs**) in moderate yields with good diastereoselectivities.

Considering the initiation of the reaction through the bifunctiomal reagent activation mode, it can be inferred that radical acceptors are not restricted solely to electron-rich anethole derivatives (Scheme 3). To validate our hypothesis, (*E*)–prop-1-en-1-ylbenzene, with an oxidation potential $E_{p/2} = +1.74$ V *vs.* SCE in CH_3CN surpassing that of the excited photocatalyst 4CzIPN ($E_{1/2\ (PC^*/PC^-)} = +1.35$ V *vs.* SCE in MeCN), was subjected to the standard conditions. As expected, the desired difunctionalized product **3ca** was formed in a moderate yield of 65%. Substituents bearing different electronic propertied were also effective precursor for the preparation of the γ,γ-diarylesters **3cb-3d** In addition, heterocycle substituted internal alkene can also be tolerated under the standard conditions, affording the thiophene substituted product **3ce**



Scheme 5. Proposed mechanism.

with a yield of 34%. The disubstituted terminal alkene exhibited relatively low reactivity to produce a quaternary-carbon-bearing γ,γ-diarylester **3cf** in 20% yield. Furthermore, styrenes turned out to be completely inert under the standard conditions. Moreover, this photocatalyzed radical addition/Smiles rearrangement cascade reaction is applicable to a variety of unactivated olefins, such as terminal and internal alkenes, yielding the corresponding alkylarylated products (**3ci**-**3cl**) with moderate yields. This underscores the synthetic versatility of our approach. Notably, cyclopentene and 1,2-dihydrofuran selectively generated *cis*-products, which likely attributed to the formation of the most stable *cis*-fused intermediates from these substrates [51].

To gain additional insights in the reaction mechanism, several control experiments were performed (Scheme 4). First, standard conditions were applied to two independent experiments using *trans*-anethole and a 1:9 mixture of *trans*- and *cis*-anethole. In both cases, identical products were formed in comparable yields, with almost identical diastereoselectivity. This indicated that a stepwise process is involved in the reaction. In addition, a deuterium labelling experiments was performed. Introducing 5 equiv. of D_2O to the reaction mixture under standard conditions resulted in the isolation of 31% yield of deuterium-labeled **3aa**-D, along with the formation of 52% yield of **3aa**. However, treating **3aa** with D_2O under the standard conditions did not lead to the detection of **3aa**-D. Furthermore, when the reaction was performed in CD_3CN with K_3PO_4 as the base, no deuterium was observed on the product. Considering the rather hydridic nature of the proton on substrate **1aa** and radical polarity-match effect, we concluded that a radical polar crossover-enabled protonation might be operational in this cascade process. Moreover, we further measured the oxidation potentials of the substrates. Generally, all the arylsulfonyl acetates exhibited good compatibility with the redox potential of the photocatalyst, ranging from 0.4 V to 0.73 V, which is considerably lower than that of alkenes (1.07–1.89 V). It is worth mentioning that all the oxidation potentials for the arylsulfonyl acetates were determined in the presence of a base. This aligns with their effective fluorescence quenching observed in experiments involving a base, which indicated a deprotonation and oxidation sequence is involved. Interestingly, the dialkylamino-substituted phenyl alkene exhibited an oxidation potential of 0.56 V, even lower than that of compound **1aa**. Further fluorescence quenching experiments indicated that these two alkenes are much more efficient quenchers of the photocatalyst (see Supporting information). These results suggest that different mechanisms might be operating in the cascade transformation depending on the olefinic partner (Fig. S12 in Supporting information).

Drawing upon the control experiments and prior literatures [35–37], a plausible mechanism for the majority of the difunctionalization was proposed, as depicted in Scheme 5. And it was further confirmed by density functional theory (DFT) calculations. Initially, the photocatalyst (PC) is irradiated by blue light to its excited state, which undergoes a single electron transfer event with **1a** in the presence of base to afford alkyl radical **I**. Electrophilic radical addition to the double bond of alkene forms benzyl radical **Int1**. Subsequently, radical **Int1** undergoes *ipso*-radical addition to the aryl ring *via* a π-π stacked transition state **TS1** with a Gibbs

free energy barrier of 14.6 kcal/mol, delivering the spiroradical intermediate **Int2**. Fragmentation, accompanied by the extrusion of SO_2 *via* a stepwise sequence involving **TS2** and **TS3**, produces alkyl radical **IV**. The exergonicity of this transition was computed to be −20.4 kcal/mol. The transient radical **Int4** is then reduced *via* another SET event from **PC-1** followed by protonation to give the desired alkene **3**, concomitantly regenerating PC to complete the photo-redox cycle. For the dialkylamino-substituted alkenes, which represent some of the electron-rich alkenes with relatively low oxidation potentials, the alkene activation mode might be involved, and a detailed discussion of the reaction mechanism is proposed in Fig. S12 (Supporting information).

In conclusion, we have unveiled a novel oxidative bifunctional reagent activation mode for the alkylarylation of alkenes under photoredox conditions using arylsulfonyl acetate as the bifunctional reagent. This metal-free radical process enables the simultaneous incorporation of carboxylate-bearing alkyl groups and (hetero)aryl rings into a wide range of olefins, thereby facilitating the synthesis of a diverse library of synthetically valuable γ,γ-diarylester derivatives. This method features mild reaction conditions, high atom- and step-economy, excellent functional group compatibility and great structural diversity. Given the current easy availability of arylsulfonylacetate bifunctional reagents, along with the ubiquity of alkenes as feedstock substrates, we anticipate this method would serve as a highly enabling platform for research endeavors aimed at synthesizing synthetic useful γ,γ-diarylester and γ-arylesters in a single operation. The success of this strategy utilizing bifunctional reagents for the difunctionalization of alkenes is expected to stimulate further investigations into this concept.

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.

CRediT authorship contribution statement

Chonglong He: Investigation, Data curation. **Yulong Wang:** Methodology, Investigation. **Quan-Xin Li:** Software, Investigation. **Zichen Yan:** Methodology, Formal analysis, Data curation. **Keyuan Zhang:** Methodology, Data curation. **Shao-Fei Ni:** Writing – review & editing, Writing – original draft, Formal analysis. **Xin-Hua Duan:** Writing – original draft, Supervision, Project administration. **Le Liu:** Writing – review & editing, Writing – original draft, Supervision, Formal analysis, Conceptualization.

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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.2024.110253.

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