Chinese Chemical Letters 35 (2024) 109843

Contents lists available at ScienceDirect

Chinese Chemical Letters

journal homepage: www.elsevier.com/locate/cclet

Chemoselective photocatalytic sulfenylamination of alkenes with sulfenamides *via* energy transfer

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ARTICLE INFO

Article history:
Received 25 December 2023
Revised 19 March 2024
Accepted 29 March 2024
Available online 29 March 2024

Keywords:
Photocatalysis
Energy transfer
Sulfenamides
Sulfenylamination
Alkenes

ABSTRACT

β-Amino sulfides hold significant biological importance, motivating the development of several methods for sulfenylamination of alkenes. However, these methods often involve a three-component system with limited alkene substrate range. In this study, we present a pioneering two-component approach utilizing readily accessible sulfenamides as efficient difunctionalization reagents. Key to its success is the careful selection of a suitable photosensitizer, which enables precise modulation of sulfenamides by promoting unprecedented energy transfer rather than traditional single-electron oxidation. This novel strategy leads to the concurrent formation of *N*- and *S*-radical species, ensuring high regioselectivity for both electron-neutral and electron-deficient alkenes. As a result, a wide range of valuable β-amino sulfides, including those with congested amine groups, can be readily synthesized. These findings highlight the potential of this method for the efficient synthesis of diverse functionalized β-amino sulfides.

Efforts have been dedicated to the development of methodologies that enable efficient and general synthesis of valuable molecules using readily accessible substrates, while ensuring atom economy. Among these methodologies, sulfenylamination of alkenes [1–9] offers a direct and rapid route to assemble β-amino sulfides, which are prevalent in bioactive compounds (*e.g.*, molecules **I**-**IV**, Scheme 1a) [10–13]. However, this transformation remains challenging, arising from the intrinsic reaction patterns of the two established strategies. The first one involves the utilization of thiiranium intermediates, where alkenes act as nucleophiles and subsequently undergo ring-opening with a separate nucleophile species (Scheme 1b, left) [3–6]. As an alternative, a few examples have explored the addition of thiyl radicals to alkenes, followed by single-electron oxidation and coupling with an additional nucleophile (Scheme 1b, right) [7–9]. These strategies rely on a three-component platform involving alkenes, sulfur (S), and nitrogen (N) sources. Consequently, such approaches suffer from inconvenient manipulations and poor atom economy. Furthermore, the generation of key intermediates like thiiranium or carbon cations, which are crucial for the difunctionalization process, restricts the use of electron-neutral alkenes. To the best of our knowledge, no reports have demonstrated the application of electron-deficient alkenes in sulfenylamination. Additionally, the sulfenylamination of α,α-disubstituted electron-neutral alkenes, which are valuable for constructing highly congested amines, is underdeveloped [9]. This can be attributed to the challenges posed by aza-nucleophilic coupling due to steric hindrance, as well as the propensity of *in-situ*-formed carbon cations for 1,2-elimination. In this context, there is a compelling need to develop a novel strategy that enables the generic sulfenylamination of alkenes in an atom-economical manner, accommodating a wide range of alkene substrates. Such a breakthrough represents a highly desirable pursuit for the scientific community.

In light of these challenges, we proposed a potential solution by investigating the feasibility of double radical-involved sulfenylamination, whereby the addition of thiyl radicals to the C=C bond of alkenes would lead to the formation of a carbon radical that could readily couple with nitrogen radicals (Scheme 1c). This tentative scenario is based on the understanding that radical couplings typically occur with minimal activation energy [14–18], thereby avoiding the formation of competing byproducts *via* carbon cation intermediates. Considering that the α-carbon radical adjacent to electron-withdrawing groups is electrophilic, it is crucial to functionalize the nitrogen radical with electron-donating groups to enhance its nucleophilic ability [19,20]. This approach aligns with

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https://doi.org/10.1016/j.cclet.2024.109843
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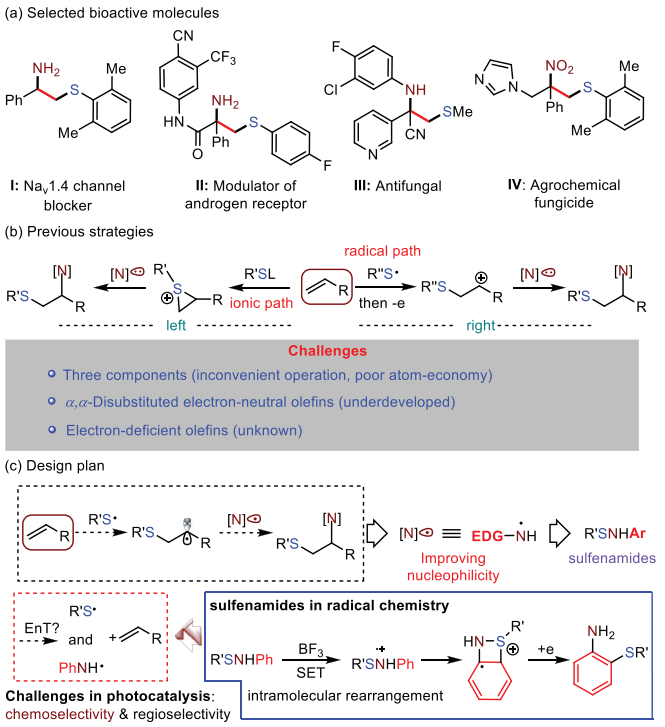
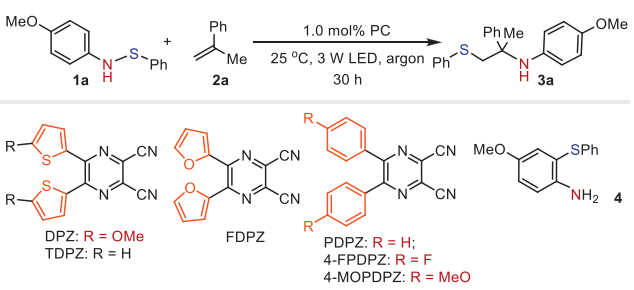


Scheme 1. Development of photocatalytic sulfenylamination of alkenes with sulfenamides as difunctional reagent.

the polar-matching rule, facilitating the efficient recombination of these electronically distinct radicals. Motivated by this concept, we became intrigued by the prospects of utilizing benzenesulfenamides as difunctional partners with alkenes to achieve the desired β-amino sulfides. Notably, since the 1970s [21–26], benzenesulfenamides have been sporadically employed in sulfenylamination of alkenes using Lewis acids as promoters. However, these approaches were limited by the use of thiiranium ions as key transition states, resulting in a severe restriction on the applicable alkene substrates and significantly limiting their synthetic utility. To address this limitation and ensure the generation of both nitrogen and sulfur radicals from benzenesulfenamides, we explored the prospect of harnessing sustainable photocatalytic energy transfer (EnT) for the homolytic cleavage of the N-S bond [27–37]. Notably, the direct irradiation conditions posed challenges due to the potential instability of the reaction products. Additionally, the low oxidation potential of benzenesulfenamides raised the possibility of heterolytic cleavage of the N-S bond *via* single-electron oxidation (SET) of the nitrogen atom by triplet photosensitizers. This SET pathway could lead to an intramolecular rearrangement on the benzene ring (Scheme 1c, bottom) [25,26], underscoring the need for addressing the chemoselectivity issue between EnT and SET pathways. Furthermore, considering the distinct preferences of electron-neutral and electron-deficient alkenes for specific nitrogen and sulfur radicals, achieving regioselectivity in the sulfenylamination process remains elusive [29–37]. These challenges highlight the complexity and importance of developing a novel methodology that addresses these issues, enabling efficient sulfenylamination with broad substrate scope and high regioselectivity.

Motivated by the potential of sulfenamides and the prospects of energy transfer (EnT) synthesis, we set out to test the aforementioned hypothesis. To explore the construction of challenging amines adjacent to congested carbons, we selected *N*-(4-methoxyphenyl)-*S*-phenylthiohydroxylamine (**1a**) and α,α-disubstituted prop–1-en-2-ylbenzene (**2a**) as our model substrates (Table 1). To begin, we evaluated the electrochemical properties

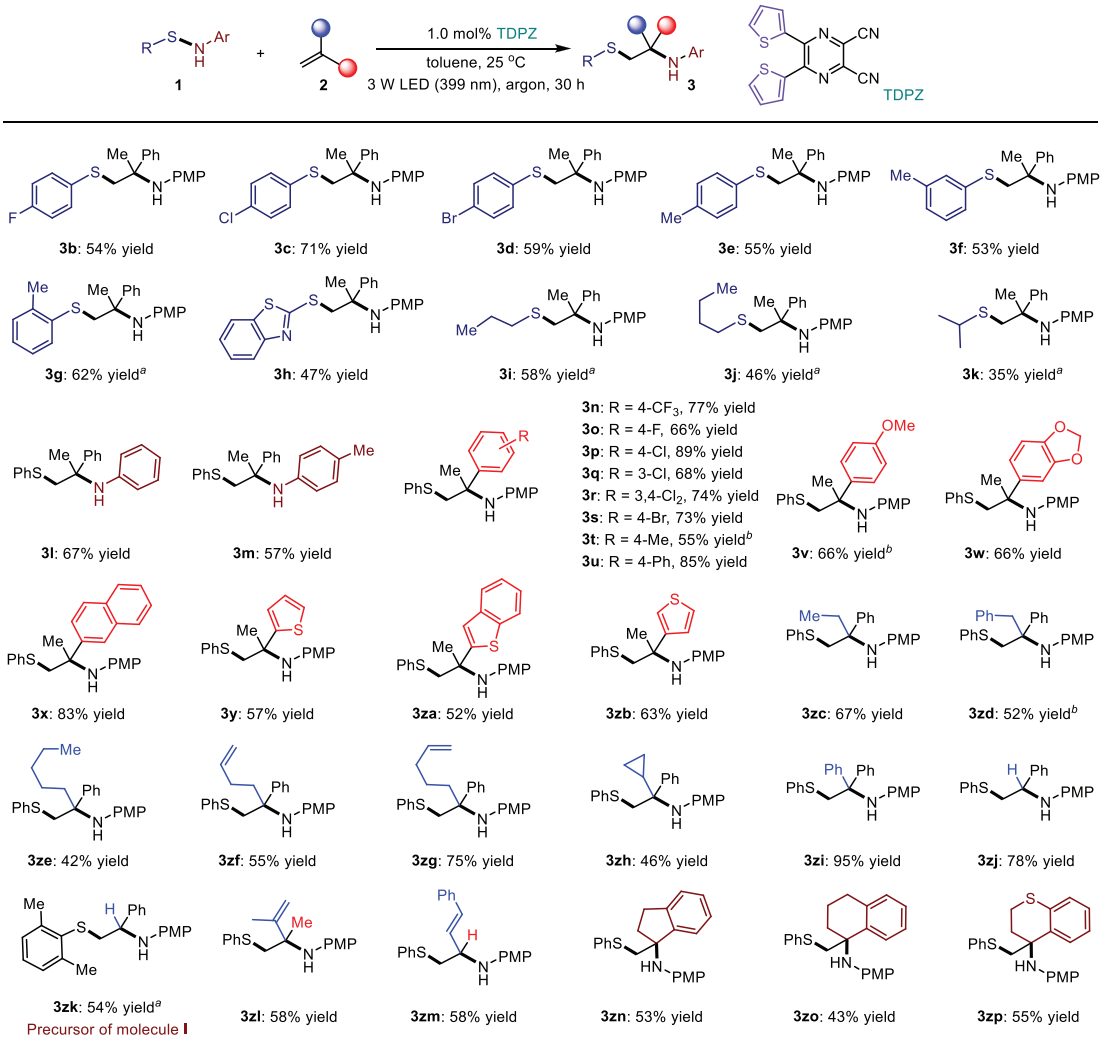
Table 1
Optimization of the reaction conditions.[a]



Entry	PC	Solvent	λ_{max}^{em} of LED	Yield (%)[b]
1	DPZ	CH_3CN	456 nm	5
2	DPZ	Toluene	456 nm	44
3	DPZ	DCM	456 nm	35
4	DPZ	Et_2O	456 nm	10
5	DPZ	EA	456 nm	30
6	Ir[dF(CF_3)ppy]$_2$(dtbbpy)	Toluene	456 nm	65
7	EosinY	Toluene	456 nm	N.R.
8	4CzIPN	Toluene	456 nm	62
9	TDPZ	Toluene	456 nm	70
10	FDPZ	Toluene	456 nm	trace
11	PDPZ	Toluene	456 nm	N.R.
12	4-FPDPZ	Toluene	456 nm	N.R.
13	4-MOPDPZ	Toluene	456 nm	N.R.
14	TDPZ	Toluene	399 nm	90
15	TDPZ	Toluene	369 nm	71
16	No TDPZ	Toluene	399 nm	5
17	TDPZ	Toluene	Dark	N.R.
18[c]	TDPZ	Toluene	399 nm	N.P.

DCM = dichloromethane. EA = ethyl acetate. N.R. = no reaction. N.P. = no product.
[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), PC (0.001 mmol), solvent (3.0 mL).
[b] Yield of **3** was isolated by flash column chromatography on silica gel.
[c] Under air.

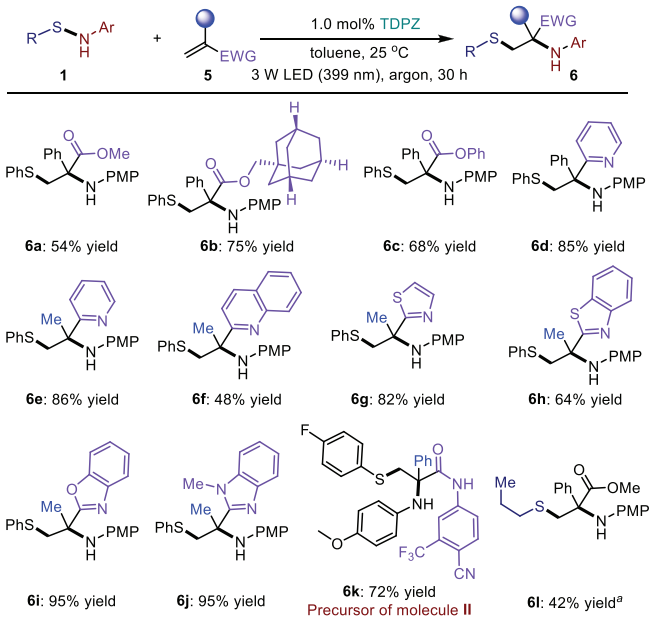
of **1a**, finding that it had an *E*p value of +0.76 and +1.61 *vs.* SCE in CH_3CN, along with an $E_T = 37.2$ kcal/mol (See Supporting information for details). Additionally, we determined the bond dissociation energy (BDE) of the N-S bond in **1a** to be 32.8 kcal/mol (see Supporting information). These results suggested the feasibility of N-S bond homolysis under excitation by energy transfer. Subsequently, we tested our self-developed photosensitizer, DPZ ($E_t(S^*/S^{\bullet-}) = +1.42$ V *vs.* SCE in CH_3CN, $E_T = 46.4$ kcal/mol) [38–40], which has demonstrated widespread utility in visible light-driven photocatalytic reactions [41,42]. Upon irradiation with a 3 W blue LED ($\lambda_{max}^{em} = 456$ nm), we obtained the desired product **3a** in only 5% yield (entry 1, Table 1). In an effort to improve the yield, we screened various solvents (entries 2–5), ultimately finding that toluene led to a yield of 44% (entry 2). Notably, no byproducts such as **4**, which could form through the intramolecular rearrangement of **1a**, were detected. In addition, we tested several other commonly used photosensitizers, including Ir[dF(CF_3)ppy]$_2$(dtbbpy), EosinY, and 4CzIPN (entries 6–8). While the Ir complex resulted in a 65% yield of **3a** (entry 6), we focused our attention on viable dicyanopyrazine-type organophotosensitizers for their clean and environmentally friendly attributes. It is worth mentioning that our ongoing research project involves the synthesis of a series of dicyanopyrazine derivatives by substituting 2-methoxythienyl with other aromatic groups to explore their feasibility as photosensitizers [38]. These derivatives can be conveniently prepared through a one-step synthesis. DPZ was chosen as our initial preference due to its higher catalytic efficiency compared to the others in these specific transformations. To further investigate the potential for catalytic ability, we carefully reexamined various derivatives of DPZ, including TDPZ, FDPZ,



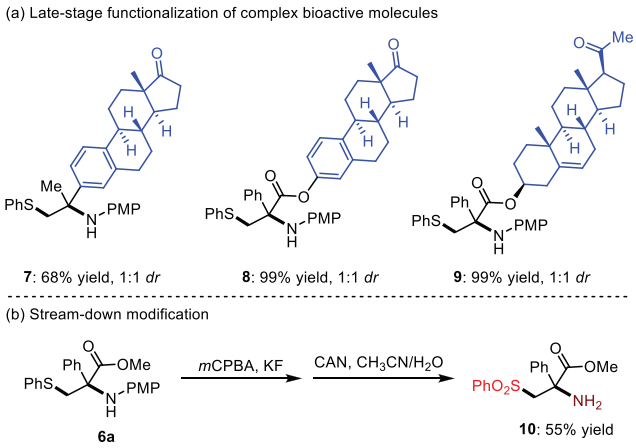
Scheme 2. Substrate scope of electron-neutral alkenes. Standard reaction conditions: on a 0.1 mmol scale, **1** (0.1 mmol), **2** (0.2 mmol) in 3.0 mL toluene at 25 °C. [a] CH_2Cl_2 instead of toluene as the solvent. [b] 2 × 3 W LEDS instead of 3 W LED.

PDPZ, 4-FPDPZ, and 4-MOPDPZ (entries 9–13). Encouragingly, TDPZ ($E_t(S^*/S^{\bullet-}) = +1.90$ V *vs.* SCE in CH_3CN, $E_T = 51.1$ kcal/mol) provided **3a** in a 70% yield, while the other four derivatives were ineffective. Considering that λ_{max}^{abs} of TDPZ is 391 nm, we utilized a 3 W LED with λ_{max}^{em} of 399 nm, which significantly improved the yield of **3a** to 90% (entry 14). Higher energy photons were found to degrade the yield (entry 15). When the transformation was conducted in the absence of TDPZ, minimal product **3a** was obtained (entry 16). No reaction was observed in the dark, underscoring the indispensability of light (entry 17). Finally, the transformation was performed in the presence of an ambient atmosphere, where **3a** could not be detected while with only **1a** completely depleted, most likely caused by the low energy of triplet oxygen leading to various side reactions (entry 18).

Following the establishment of optimized reaction conditions, we investigated the substrate scope of this two-component sulfenylamination protocol (Scheme 2). Initially, we tested the reaction of **2a** with various benzenesulfenamides bearing *S*-aryls with diverse electron-withdrawing and electron-donating groups. This resulted in the formation of products **3b**-**3g** with yields ranging from 53% to 71%. The protocol also tolerated sulfenamides with azaaryls instead of aryls as the *S*-substituent, as demonstrated by the formation of **3h** in a 47% yield. Furthermore, the method proved to be compatible with different aliphatic sulfides, as indicated by the successful synthesis of **3i**-**3k**. It is worth noting that the choice of the 4-methoxyphenyl (PMP) group as an *N*-protecting group serves two purposes: enhancing the nucleophilicity of the putative amino radical and providing an easily removable group. Nevertheless, we explored the substitution of PMP with other aryls, and products **3l**-**3m** exhibited good compatibility with respect to substitutions on the *N*-aryl group. Next, we investigated various electron-neutral alkenes using **1a** as the reaction partner. Notably, a wide range of α-methyl styrenes were well tolerated regardless of electronic properties or substitution patterns on the aromatic rings. This resulted in the formation of products **3n**-**3w** in yields ranging from 55% to 89%. Furthermore, the high chemical yields observed for alkenes with fused aromatic (**3x**) and heteroaromatic (**3y**-**3zb**) ring substituents highlight the generality of this method. We also tested styrenes bearing distinct 2-alkyl substituents, leading to satisfactory yields of products **3zc**-**3zh**. In the case of substrates containing two alkene moieties (**3zf**-**3zg**), sulfenylamination showed a preference for the more electron-rich one. Additionally, when 2-alkyls were replaced by 2-aryls, the reaction proceeded with higher reactivity and chemoselectivity (**3zi**). Simple styrene proved to be a valuable starting material for synthesizing biologically important aryl sulfides with a tertiary carbon atom adjacent to the amine group, as exemplified by the formation of products **3zj** and **3zk** in yields of 78% and 54%, respec-



Scheme 3. Substrate scope with respect to electron-deficient alkenes (on a 0.1 mmol scale). [a] CH_2Cl_2 instead of toluene as the solvent.



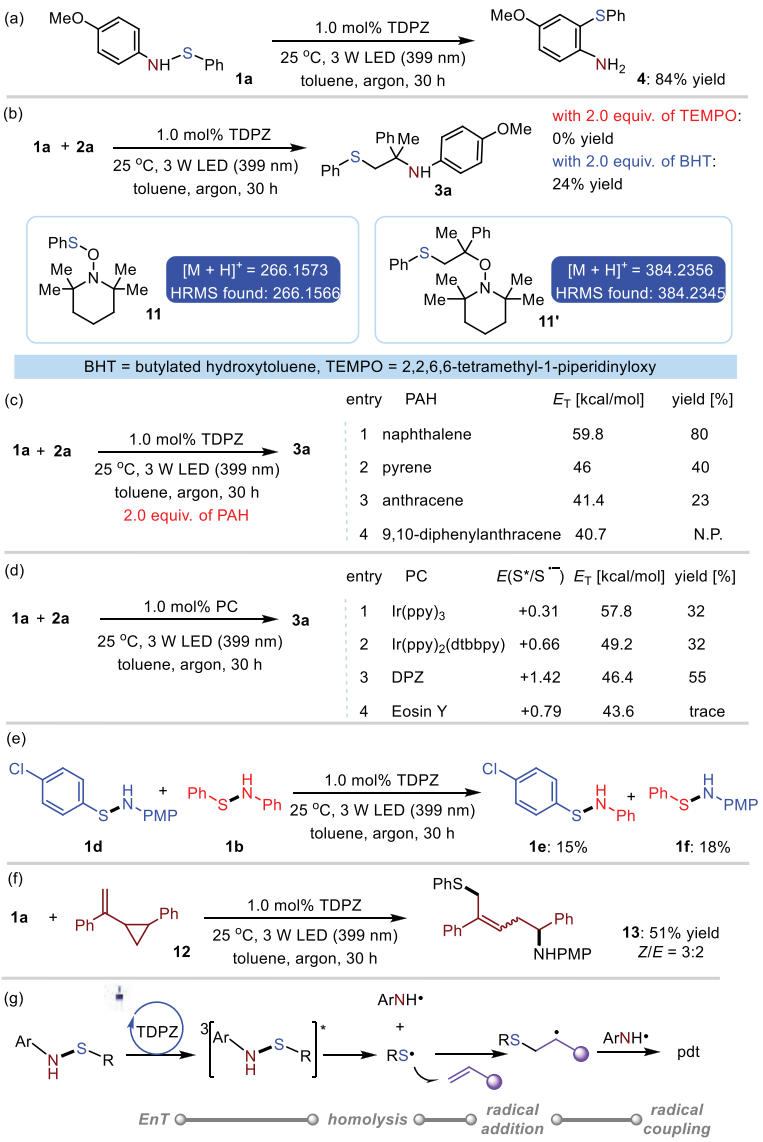
Scheme 4. Exploration of the synthetic utility of the method.

tively. Moreover, it is worth noting that a one-step deprotection of PMP in **3zk** can provide access to a class of bioactive compounds, namely Na_V 1.4 channel blockers (molecule **I**, Scheme 1a). In addition to styrenes, two different 1,3-dienes reacted successfully with **1a**, yielding adducts **3zl**-**zm** with satisfactory yields. Notably, the reaction with terminal alkenes (**3zm**) displayed higher reactivity. Finally, exocyclic C=C bonds, including 5-, 6-, or hetero-rings, were also successfully transformed into sulfenylaminated products (**3zn**-**3zp**) with yields ranging from 43% to 55%. These results highlight the broad substrate scope of the developed sulfenylamination protocol, demonstrating its versatility in synthesizing a variety of valuable products.

Building on the promising results, we expanded the scope of this catalysis platform to investigate the efficiency with electron-deficient alkenes, represented by substrate **5** (Scheme 3). We initially focused on 2-arylacrylates due to their potential in directly synthesizing valuable α-amino acid derivatives that possess a structurally congested amine and a β-thioether motif (*e.g.*, molecule **II**, Scheme 1a). Encouragingly, products **6a**-**c** were obtained with satisfactory yields, regardless of the alkyl and aryl substituents on the esters. Considering the prevalence of imine-containing azaarenes in pharmaceutical compounds and their electron-withdrawing nature [43], we explored the sulfenylamination of **1a** with various vinylazaarenes containing α-aryls or α-alkyls, as well as diverse valuable azaarenes. Consequently, products **6d**-**j** were obtained in yields ranging from 48% to 95%. The broad applicability of electron-withdrawing groups to olefins inspired us to further investigate a specific amide-based olefin, as its corresponding product **6k** could be conveniently deprotected to yield a modulator of the androgen receptor (molecule **II**, Scheme 1a). Furthermore, product **6l** demonstrated the potential of this strategy to assemble alkyl sulfides on these valuable tertiary amines. These findings highlight the robustness and versatility of this catalytic system in facilitating the synthesis of intricate molecules containing electron-deficient alkenes. The successful formation of structurally diverse products further emphasizes the potential of this method in accessing biologically relevant compounds and expanding the synthetic toolbox for complex molecule synthesis.

To assess the generality and potential synthetic applications of this novel methodology, the ability for late-stage functionalization of complex bioactive molecules was investigated, as depicted in Scheme 4a. The objective was to evaluate its versatility in accessing a greater variety of valuable compounds that could be useful for drug development. In this investigation, we successfully assembled estrone and pregnenolone, chosen as representative molecules, onto products **7**–**9** by reacting **1a** with the corresponding electron-neutral and electron-deficient alkenes. This demonstrated the capability of the method to perform late-stage functionalization on complex bioactive molecules. Furthermore, to further confirm the promising synthetic applications of this method, we selected product **5a** and subjected it to oxidation using 3-chloroperoxybenzoic acid (*m*-CPBA) to yield the corresponding sulfone. The *N*-PMP group in the sulfone was then smoothly cleaved using cerium ammonium nitrate (CAN), resulting in the synthesis of sulfone-based α-amino ester **10** in 55% yield over two steps (Scheme 4b). This demonstrates the efficient access to pharmaceutically important molecules **I**-**II** (Scheme 1a) using this methodology. Additionally, this strategy has the potential to address the incompatibility issues observed with *N*-alkyl benzenesulfenamides in the photocatalytic difunctionalization approach. These results highlight the broad synthetic applications and potential for late-stage functionalization of complex bioactive molecules using this methodology. The ability to access pharmaceutically important compounds and overcome compatibility issues further underscores the value and utility of this novel catalytic system in drug development and synthetic chemistry.

In order to gain insight into the mechanistic aspects of this strategy, extensive mechanistic studies were conducted. Initially, control experiments (Table 1, entries 16 and 17) indicated that TDPZ serves as the photosensitizer in the photocatalytic sulfenylamination. However, considering the photochemical and photophysical properties of TDPZ, the photoactivated TDPZ (*TDPZ) could potentially initiate the reactions through either single-electron transfer (SET) or energy transfer (EnT) with benzenesulfenamides. This hypothesis is further supported by comparing the redox potentials and triplet energies of the species involved, as well as the results of the Stern-Volmer experiment. To elucidate the actual excitation mode, a series of elaborate studies were conducted. Firstly, when **1a** was subjected to the established reaction conditions without the presence of alkenes, product **4** was obtained in an 84% yield. This result suggests the occurrence of a competing chemical transformation involving SET of benzenesulfenamides to *TDPZ (Scheme 5a). These findings provide important insights into the mechanistic aspects of this strategy. They



Scheme 5. Mechanistic studies and the plausible mechanism.

indicate that the photoactivated TDPZ can engage in either SET or EnT processes with benzenesulfenamides, thereby initiating the desired transformations. Further investigation and characterization of the reaction intermediates and excited states are necessary to obtain a comprehensive understanding of the underlying mechanisms.

We then conducted several experiments to probe the involvement of radicals in the difunctional sulfenylamination process. Firstly, we attempted the transformation of **1a** with **2a** in the presence of TEMPO as a radical trapping reagent (Scheme 5b, in red). The detection of the trapped products **11** and **11′** by high-resolution mass spectrometry analysis confirmed the formation of thiyl radicals and their viability to undergo addition to olefins. Interestingly, the desired product **3a** was not obtained, indicating that the presence of alkenes leads to a different interaction pattern between the photosensitizer and **1a**, compared to the formation of **4**. We further added 2.0 equiv. of BHT, a radical scavenger, to the reaction. While **1a** was nearly consumed, only a small amount of product **4** was detected, and the yield of **3a** was significantly reduced to 24% (Scheme 5b). This indicates that the difunctional sulfenylamination process involves radical intermediates. To explore the source of radicals, we examined the transformation using various polycyclic aromatic hydrocarbons as energy scavengers for the photoactivated sensitizers (Scheme 5c). Interestingly, it was found that naphthalene, which has a higher triplet energy than DTPZ, did not affect the transformation (entry 1), while 9,10-diphenylanthracene, with an energy similar to that of **1a**, completely suppressed the generation of **3a** while **1a** was consumed (entry 4). We also investigated the transformation using other photosensitizers, and the results are summarized in Scheme 5d. Notably, two Ir complexes with poor oxidizing ability but sufficient triplet energy could yield product **3a** (entries 1 and 2). In contrast, Eosin Y, with a lower E_T of 43.6 kcal/mol, failed to generate **3a** (entry 4). These findings suggest that the reaction relies on the photoexcitation of the sensitizers to generate radicals. In a crossover experiment with two different benzenesulfenamides (**1d** and **1b**) under the reaction conditions, after 30 h, two cross-coupling products (**1e** and **1f**) were obtained (Scheme 5e). Additionally, a radical clock experiment was performed using molecule **12**, which contains an alkene and a cyclopropane ring. As shown in Scheme 5f, a difunctionalized product (**13**) with the opened cyclopropane ring was obtained in 51% yield, suggesting the formation of both *N*- and *S*-radicals in the reaction system. It is worth mentioning that the different chemoselectivity be-

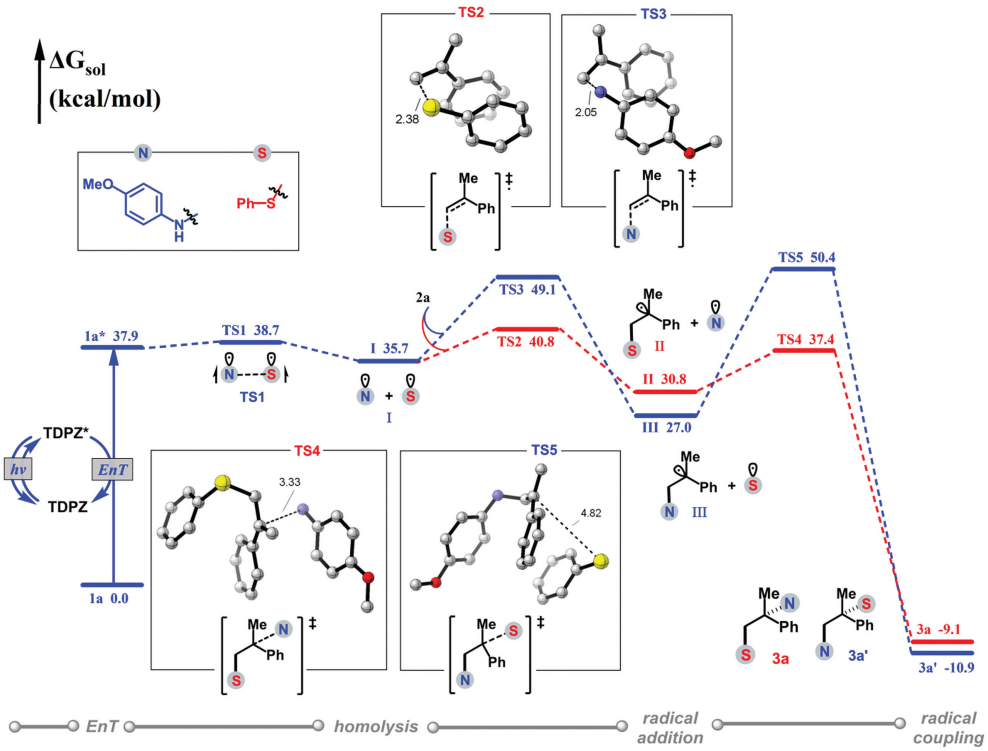


Fig. 1. DFT evaluation of the reaction profile, calculated at the SMD(toluene)-M06–2X/6–31G(d,p) // SMD(toluene)-B3LYP-D3(BJ)/6–31G(d,p) level and 298.15 K.

tween **12** and (1-cyclopropylvinyl)benzene (**3zh**, Scheme 2) probably originates from the considerable instability of the primary carbon radical produced from the latter. It thus readily undergoes intramolecular addition to yield the more stable benzylic radical, which just possesses a similar reactivity to the benzylic radical generated from **12**. In addition, a light on/off experiment was performed, which showed that a radical chain process might be impracticable. Based on these results, a plausible mechanism involving homolysis of the N-S bond through EnT has been proposed (Scheme 5g). In addition to EnT-enabled homolysis, a sequential process of radical addition and radical coupling is responsible for the final formation of the sulfenylamination products. The excellent chemoselectivity observed in this reaction can be attributed to the kinetic preference of the radicals generated *via* EnT for addition to alkenes and/or cross-coupling, compared to the radical cations generated *via* SET that may undergo rearrangements. The efficiency of EnT between the photosensitizer and benzenesulfenamides is crucial for the overall reactivity of the system. These mechanistic insights provide a deeper understanding of the radical-involved difunctional sulfenylamination process and contribute to the development of this efficient and chemoselective synthetic methodology.

To further substantiate the proposed reaction pathways and elucidate the precise regioselectivity (Scheme 5g), density functional theory (DFT) calculations were then performed. As depicted in Fig. 1, the mechanism of the reaction can be reasonably postulated to commence with a triplet-triplet EnT event between triplet excited state of photosensitizer TDPZ and the singlet state of sulfenamide reagent **1a**. Subsequently, a homolysis cleavage of the triplet excited state **1a*** occurs *via* **TS1** (0.8 kcal/mol), leading to the generation of S-radical and N-radical [44]. The ensuing step involves the active S-radical undergoing addition to the terminal carbon of styrene **2a** *via* **TS2**, affording an addition intermediate **II** with a free energy barrier of 5.1 kcal/mol. In contrast, a parallel radical addition involving the N-radical and styrene **2a** *via* **TS3** necessitates surmounting a notably higher free energy barrier of 13.4 kcal/mol. Ultimately, a radical-radical cross coupling event between intermediate **II** and the N-radical *via* **TS4** gave the final product **3a** with a free energy barrier of 6.6 kcal/mol. On the other hand, another analogous radical coupling between intermediate **III** and the S-radical *via* **TS5** necessitates overcoming a significantly higher free energy barrier of 19.6 kcal/mol. Considering the substantial disparity in free energy between **TS2** and **TS3**, as well as between **TS4** and **TS5**, it is apparent that the formation of the product **3a** is kinetically favored. This rationalization effectively accounts for the remarkable regioselectivity that has been observed in this difunctional transformation.

In conclusion, we have developed a novel two-component sulfenylamination of alkenes [45–48] by precisely activating readily prepared sulfenamides through photocatalytic energy transfer. This approach offers several advantages, including high atom economy and exceptional chemoselectivity, making it compatible with a wide range of substrates. Notably, both electron-neutral and unprecedented electron-deficient alkenes can be efficiently incorporated using this catalytic platform. As a result, a diverse array of biologically important β-amino sulfides with secondary or tertiary carbon α to the amine moiety were obtained in high yields. The transition-metal-free nature of this approach holds great potential for the facile and efficient preparation of sulfur-containing α-amino acid derivatives and azaarene-based amines, which are crucial for drug development. The demonstration of sulfenamides' ability to generate both *N*- and *S*-radicals through energy transfer opens up promising avenues for utilizing these valuable building blocks in radical-based chemical synthesis. Furthermore, this methodology can contribute to the synthesis of a wider range of important *N*- and/or *S*-containing molecules. Overall, our developed sulfenylamination strategy showcases a versatile and efficient approach for the incorporation of sulfenamides in synthetic chemistry, with broad applications in pharmaceutical and drug discovery research.

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

Er-Meng Wang: Investigation, Methodology. **Ziyi Wang:** Investigation, Methodology. **Xu Ban:** Writing – review & editing. **Xiaowei Zhao:** Data curation, Funding acquisition. **Yanli Yin:** Conceptualization, Funding acquisition, Supervision, Writing – original draft, Writing – review & editing. **Zhiyong Jiang:** Conceptualization, Funding acquisition, Supervision, Validation, Writing – original draft, Writing – review & editing.

Acknowledgment

We are grateful for financial support from the National Natural Science Foundation of China (Nos. 22171072, 21925103, and 22301061).

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.109843.

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