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Chinese Chemical Letters

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An electron donor–acceptor photoactivation strategy for the synthesis of *S*-aryl dithiocarbamates using thianthrenium salts under mild aqueous micellar conditions☆

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ARTICLE INFO

Article history:
Received 29 December 2022
Revised 18 March 2023
Accepted 28 March 2023
Available online 30 March 2023

Keywords:
S-Aryl dithiocarbamates
Visible-light
EDA complex
Thianthrenium salts
Micellar photocatalysis

ABSTRACT

An eco-friendly and convenient method is developed herein for the synthesis of *S*-aryl dithiocarbamates *via* visible-light-induced SET process of an EDA complex between thianthrenium salt functionalized arenes and dithiocarbamate anions under mild aqueous micellar conditions. This strategy indirectly realizes the method for constructing *S*-aryl dithiocarbamates through site-selective C—H functionalization of arenes. Most importantly, the reaction proceeded smoothly without addition of any photocatalyst, and the by-product thianthrene is recycled in quantity, ultimately minimizing the production of chemical waste. This protocol provides a promising synthesis candidate for the construction of valuable *S*-aryl dithiocarbamates, which also opens up a new avenue for micellar photocatalysis.

The dithiocarbamate skeleton is one of the most important sulfur-containing organic frameworks, showing various biological activities such as anticancer [1,2], anti-infection [3], antibacterial [4], and antitumor properties (Scheme 1A) [5]. Furthermore, dithiocarbamates are also widely used as versatile synthetic building blocks in organic synthesis [6]. In light of their widespread utilities, the development of novel and convenient methods for the synthesis of these useful sulfur-containing compounds continues to motivate a large number of scientists. The classical approaches toward *S*-aryl dithiocarbamates are the reaction of amines with thiophenols and thiophosgene or the treatment of isothiocyanates with thiophenols [7,8]. Recently, copper-catalyzed Ullmann-type coupling between iodoarenes or aryl boronic acids with sodium dialkyldithiocarbamates or tetraalkylthiuram disulfides has also been developed for the preparation of *S*-aryl dithiocarbamates [9–13]. For example, Bolm and Dong demonstrated an elegant copper-catalyzed system towards *S*-aryl dithiocarbamates using commercially available thiuram disulfide reagents [14]. Nevertheless, most of these methods still involve the use of toxic metal catalysts, special ligands, multi-step synthetic sequences, or harsh reaction conditions, which would limit their practical utility. Therefore, the development of an efficient, facile, and eco-friendly approach to *S*-aryl dithiocarbamates is still highly desirable. As a practical organic synthesis strategy, multicomponent reactions (MCRs) have attracted extensive attention because it improves the atomic economy and step economy of the reaction process, thus minimizing pollution and reducing costs [15,16]. In 2008, Ranu and co-workers reported an efficient method for the synthesis of *S*-aryl dithiocarbamates through copper nanoparticle-catalyzed three component condensation of arylhalides, carbon disulfide and amines in water (Scheme 1B) [17]. In 2016, Qi *et al.* demonstrated a similar process utilizing aryl boronic acids as the aryl reagents by copper catalysis (Scheme 1B) [18]. Very recently, Murarka and Singh developed an elegant multicomponent coupling between diaryliodonium triflates, amines, and carbon disulfide for the preparation of *S*-aryl dithiocarbamates under mild conditions (Scheme 1B) [19]. Furthermore, Biju and Jindal reported a facile method for the synthesis of *S*-aryl dithiocarbamates by three-component

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☆ Dedication to Prof. Lixin Dai on the Occasion of His Centenary Birthday.
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https://doi.org/10.1016/j.cclet.2023.108403
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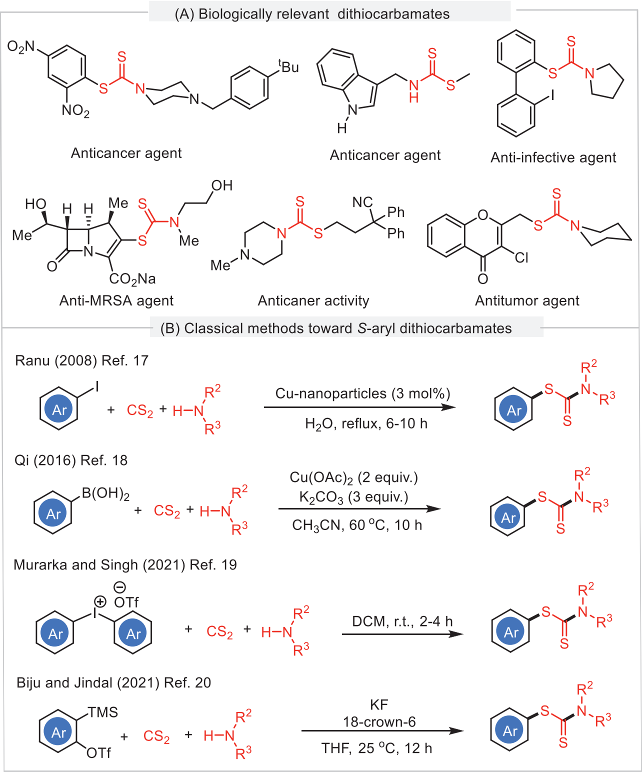
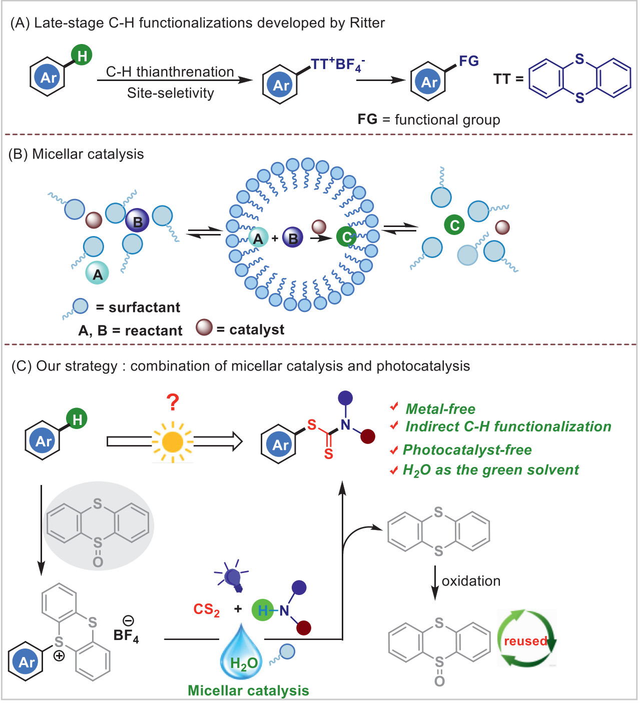


Scheme 1. (A) Biologically relevant *S*-aryl dithiocarbamates, (B) Classical methods toward *S*-aryl dithiocarbamates.



Scheme 2. (A) Late-stage C-H functionalization of arens; (B) The reaction mode of micellar catalysis; (C) Our strategy for the synthesis of *S*-aryl dithiocarbamates from thianthrenium salt functionalized arenes.

coupling of arynes, CS_2 and aliphatic amines under metal-free conditions (Scheme 1B) [20]. Although great achievements have been made, developing an alternative approach to *S*-aryl dithiocarbamates with high efficiency and generality is still of great interest.

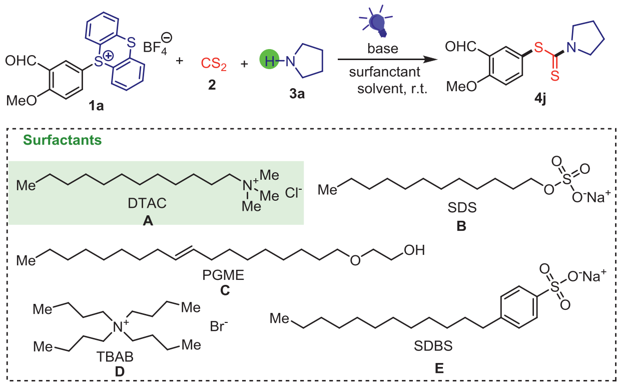
In recent years, the direct C—H functionalization of unreactive C—H bonds provides a valuable tool for the construction of C—C bonds and C—heteroatom bonds [21–25]. However, the main challenge for this straightforward strategy is the site-selectivity. The existence of the directing group together with transition-metal catalysis could effectively solve this problem, but the removal or transformation of the directing groups is a intractable task [26]. Notably, Ritter and co-workers demonstrated an efficient and convenient site-selective C—H functionalization of arenes by thianthrenation process affording many chances for late-stage functionalization of arenes without a directing group (Scheme 2A) [27]. Recently, desulfurization couplings using thianthrenium salts for the construction of C—C bonds and C—heteroatom bonds have been extensively studied [28–46]. Undoubtedly, the C-H bond functionalization is the most efficient and direct method to construct *S*-aryl dithiocarbamates (Scheme 2C).

Over the past decade, visible-light photoredox catalysis has emerged as an attractive strategy for organic transformations *via* photoinduced energy transfer (EnT), hydrogen atom transfer (HAT), or electron transfer (SET) processes as well as their combinations [47–56]. In this regard, electron donor-acceptor (EDA) complexes which possess typical red-shifted charge-transfer transition bands, can effectively induce inner-sphere electron transfer upon their photoexcitation to generate radicals [57–59]. Considering the special properties of thianthrenium salts and their susceptibility to undergo single electron reduction, we envisage that *S*-aryl dithiocarbamates could be constructed starting from readily available arenes *via* indirect C-H bond functionalization (Scheme 2C).

On the other hand, water is an ideal green non-toxic reaction medium, which is environmentally friendly and safe to handle. However, performing organic reactions in water is often challenging due to the immiscibility of organic molecules in aqueous media, and this has brought to light the use of surfactants as micellar catalytic approach (Scheme 2B) [60–63]. Micelles can effectively accumulate reactants and catalysts in their inner region, forming a higher local concentration, which can facilitate organic transformations effectively. Over the past few decades, tremendous progress has been made in "micellar catalysis" since it affords a sustainable alternative to various conventional organic synthesis [64–67]. As a continuous interest of our research in sulfur chemistry and photochemical reactions [68–79], herein, we would like to report a novel and efficient visible-light-induced SET process of an EDA complex between thianthrenium salt functionalized arenes and dithiocarbamate anions under mild aqueous micellar conditions for the synthesis of *S*-aryl dithiocarbamates (Scheme 2C).

We firstly embarked on optimization studies through reacting aryl thianthrenium salt **1a**, carbon disulfide **2** and pyrrolidine **3a** in DMSO by using K_2CO_3 as a base at room temperature with irradiation of a 20 W blue LED lamp. To our delight, the desired *S*-aryl dithiocarbamate **4j** could be obtained in 70% isolated yield (Table 1, entry 1). Encouraged by this result, further optimization of different reaction parameters including the solvents, bases and surfactnts was carried out. Initially, seven solvents, such as NMP, 1,4-dioxane, THF, MeCN, DMF, DCM, and DCE were evaluated, and none of these solvents are as effective as DMSO. In addition, the yield of product **4j** was not improved with the increase of the water content in DMSO (Table 1, entry 9). We found that the yield

Table 1
Screening for the optimal conditions.[a]



Entry	Surfactant	Base	Solvent	Yield of **4j** (%)[b]
1	None	K_2CO_3	DMSO	70
2	None	K_2CO_3	NMP	55
3	None	K_2CO_3	1,4-Dioxane	40
4	None	K_2CO_3	THF	32
5	None	K_2CO_3	CH_3CN	45
6	None	K_2CO_3	DMF	Trace
7	None	K_2CO_3	DCM	Trace
8	None	K_2CO_3	DCE	Trace
9	None	K_2CO_3	DMSO/H_2O (1:1)	67
10	None	K_2CO_3	H_2O	65
11	**A**	K_2CO_3	H_2O	82
12	**B**	K_2CO_3	H_2O	80
13	**C**	K_2CO_3	H_2O	79
14	**D**	K_2CO_3	H_2O	70
15	**E**	K_2CO_3	H_2O	75
16	**A**	K_2CO_3	H_2O	Trace[c]
17	**A**	None	H_2O	Trace
18	**A**	Na_2CO_3	H_2O	73
19	**A**	Cs_2CO_3	H_2O	60
20	**A**	DBU	H_2O	35
21	**A**	DABCO	H_2O	32
22	**A**	NaOH	H_2O	55
23	**A**	DMAP	H_2O	42
24	**A**	Et_3N	H_2O	50
25	**A**	K_2CO_3	H_2O	40[d]
26	**A**	K_2CO_3	H_2O	68[e]
27	**A**	K_2CO_3	H_2O	51[f]
28	**A**	K_2CO_3	H_2O	0[g]
29	**A**	K_2CO_3	H_2O	Trace[h]

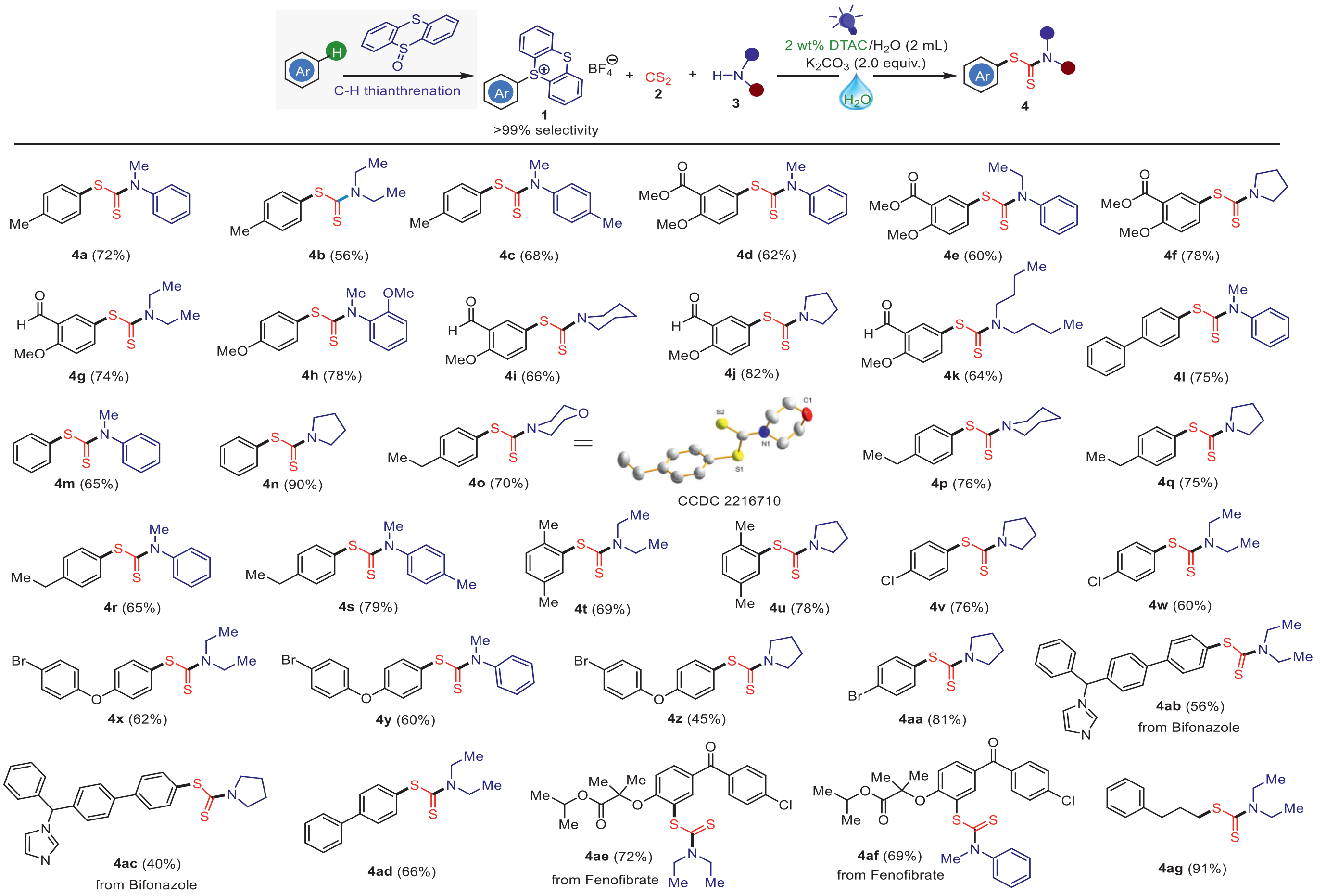
[a] Reaction conditions: **1a** (0.2 mmol), **2** (0.6 mmol), **3a** (0.4 mmol), base (0.4 mmol), 2 wt% sulfactant/H_2O (2 mL) at room temperature under irradiation with a 20 W blue LED (455 nm) for 12 h.
[b] Isolated yield.
[c] No light.
[d] Eosin Y was used as the photocatalyst.
[e] 4CzIPN was used as the photocatalyst.
[f] $Ir[dF(CF_3)ppy]_2(dtbbpy)BF_6^-$ was used as the photocatalyst.
[g] At 40 °C, no light.
[h] At 60 °C, no light.

of **4j** reached 65% when water was used as the solvent (Table 1, entry 10). Inspired by micellar catalysis, various surfactants were screened in order to achieve the optimum conditions. To our delight, dodecyl trimethyl ammonium chloride (DTAC) **A** show the highest activity, delivering the product **4j** in 82% isolated yield (Table 1, entries 11–15). Next, we examined different bases including K_2CO_3, Na_2CO_3, Cs_2CO_3, DBU, DABCO, NaOH, DMAP, Et_3N, and K_2CO_3 was more efficient (Table 1, compare entries 11, and 17–24). Furthermore, control experiments indicated that light irradiation is an essential factor in the present transformation (Table 1, entry 16). In addition, control experiments had shown that adding additional photocatalysts did not improve the reaction efficiency (entries 25–27, Table 1). Finally, controlled experiments showed that raising the reaction temperature in the absence of light resulted in lower yields (entries 28 and 29, Table 1).

After establishing the optimal conditions (Table 1, entry 11), the scope and generality of the present reaction was investigated (Scheme 3). To our delight, various aryl thianthrenium salts **1** which were directly synthesized from diverse arenes reacted efficiently with carbon disulfide **2** and secondary amines **3**, affording the target products **4** in moderate to excellent yields (**4a**–**4af**). The secondary amines including aliphatic and aromatic ones were well tolerated under the standard conditions, showing no obvious difference in reactivity. There is no significant difference in the electron-effect of the substituted groups in secondary aromatic amines, including electron-rich, and -neutral ones (compare **4a**, **4c**, **4d** and **4h**). In addition, a series of cyclic and linear amines coupled with carbon disulfide and aryl thianthrenium salts to produce the desired products in good yields. Subsequently, the scope of aryl thianthrenium salts was explored. To our satisfaction, the present photochemical reaction tolerated an array of structurally and electronically diverse aryl thianthrenium salts. Notably, the electron-withdrawing groups (-Cl, -Br) or electron-donating groups (-Me, -Et, -OMe) present at any position of the phenyl group of arylsulfonium salts showed similar reactivity. Most noteworthy is the EDA complex photoactivation method was also applied to the late-stage functionalization of pharmaceuticals or their derivatives (**4ab**, **4ac**, **4ae** and **4af**). A series of valuable functional groups including alkoxy, alkyl, ester, halides, and imidazole are well tolerated under the standard conditions leaving ample room for further modifications. To our delight, alkyl thianthrenium salt was also well tolerated in the present tranformaiton, leading to the *S*-alkyl dithiocarbamate **4ag** in 91% isolated yield.

To show the synthetic application of this EDA complex photoactivation method, a gram-scale reaction was carried out by using substrates **1a** (9 mmol), CS_2 (27 mmol), and pyrrolidine (**3a**) under irradiation of two 20 W blue LED lamps (Scheme 4a). To our delight, the reaction proceeded well by delivering **4j** in 63% isolated yield, suggesting that this photochemical system is easy to scale up. Importantly, thianthrene **5** could be well recovered which subsequently was oxidized into thianthrene 5-oxide **6** in quantitative yield (Scheme 4a) [80,81]. Furthermore, a sunlight-promoted experiment also afforded **4j** in 60% isolated yield (Scheme 4b). The sustainability of the reaction media was also evaluated using the model reaction under the optimized conditions. After completion of the reaction, the mixture was extracted with ethyl acetate and only fresh reagents (**1a**, CS_2 and **3a**) were added to the DTAC/H_2O solution for the next photochemical reaction cycle. Excitingly, the chemical process showed no significant loss of activity after five cycles as illustrated in Scheme 4c. Notably, the reaction also proceeded smoothly when 4-chlorophenol was used as the substrate, and the thioether product **4ah** was obtained in 80% isolated yield (Scheme 4d). Therefore, this developed protocol can be well applied to the synthesis of thioethers. These experimental results indicate the potential value of this synthesis strategy.

To illuminate the mechanism of the reaction, radical trapping experiments were conducted (Scheme 5). When radical-trapping reagents 2,2,6,6-tetramethylpiperidin-1-yl)oxidanyl (TEMPO) and 1,1-diphenylethylene were added into the present reaction system, the transformation was significantly suppressed, which implied that the reaction might proceed *via* a free radical pathway. In addition, the formation of trapping product of aryl radical was detected by HRMS analysis, suggesting the generation of aryl radicals in the EDA complex photoactivation transformation.



Scheme 3. The substrate scope. Reaction conditions: **1** (0.2 mmol), **2** (0.6 mmol), **3** (0.4 mmol), K_2CO_3 (0.4 mmol), 2 wt% sulfactant/H_2O (2 mL) at room temperature under irradiation with a 20 W blue LED (455 nm) for 12 h. Isolated yield.

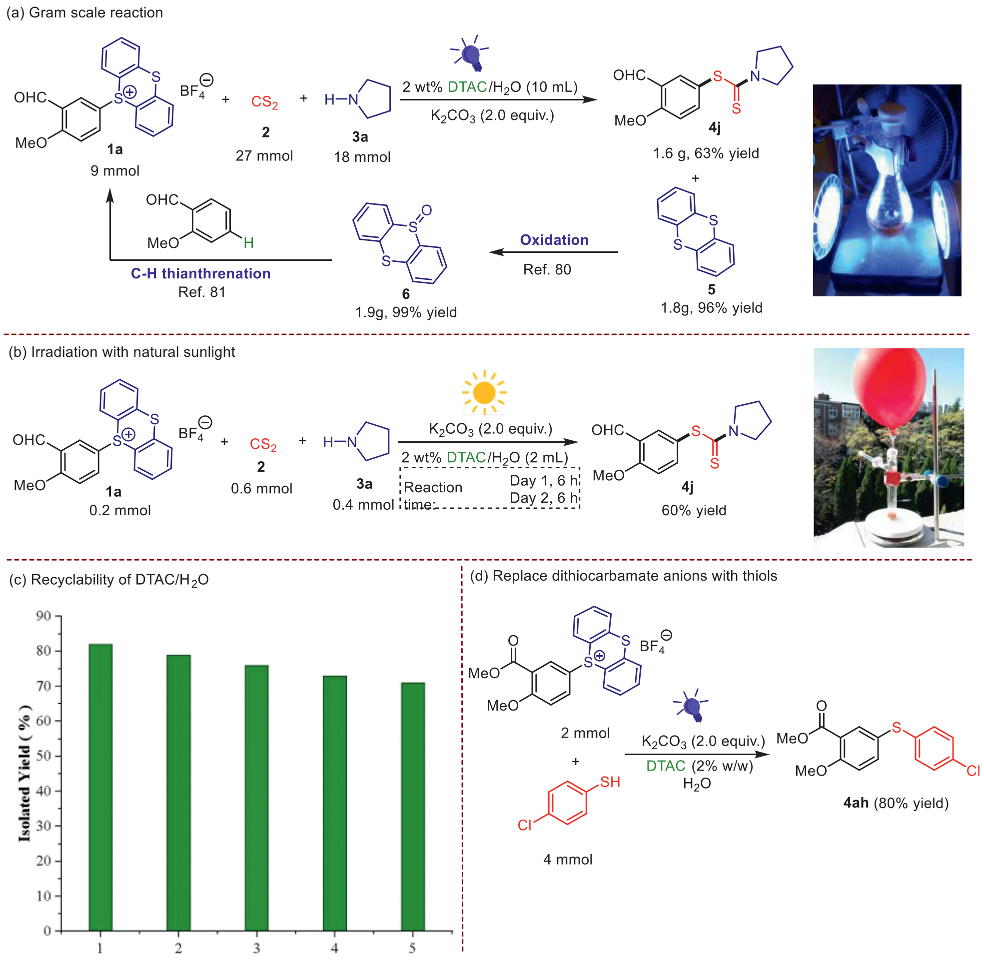
In order to verify the formation of an EDA complex in the current transformation, some preliminary experiments were performed (Fig. 1). When dithiocarbamate anion **8** generated in situ was added to a solution of aryl thianthrenium salt **1a**, the solution transformed from pale colourless to yellow color. Furthermore, the UV-vis absorption of different combinations of the reactants were measured. As shown in Fig. 1a, neither aryl thianthrenium salt **1a**, carbon disulfide **2** + pyrrolidine **3a**, nor **1a**+**2**+**3a** showed obvious absorption beyond 400 nm, but a mixture of thianthrenium salt **1a**, carbon disulfide **2**, pyrrolidine **3a** and K_2CO_3 showed a significant bathochromic shift. In addition, a molar ratio of 1:1 between **1a** and **8** was observed by using Job's method of continuous variations (Fig. 1b). Finally, ^{1}H NMR titration experiments were performed as shown in Fig. 1c. Upon addition of **8** generated in situ to **1a** in $CDCl_3$, the proton signals of **1a** showed significant upfield shift, indicating the formation of an EDA complex between **1a** and **8** in the photochemical process.

Based on the above preliminary finding and previous related reports [82–85], a plausible mechanism hypothesis for the EDA complex photoactive transformation is thus presented in Scheme 6. Initially, carbon disulfide **2** reacts with amines **3** in the presence of K_2CO_3 under mild aqueous micellar conditions to give the corresponding dithiocarbamate anion **A**. Subsequently, the electron-rich dithiocarbamate anion **A** and the electron-poor aryl thianthrenium salt **1** associate to form an EDA complex **B**. Upon irradiation with visible-light, a SET (single electron transfer) transformation from **A** to **1** to give an aryl radical **D**, a thiyl radical **E**, and thianthrene **5**. Finally, the aryl radical **D** couples with the thiyl radical **E**, delivering the coupling product **4**.

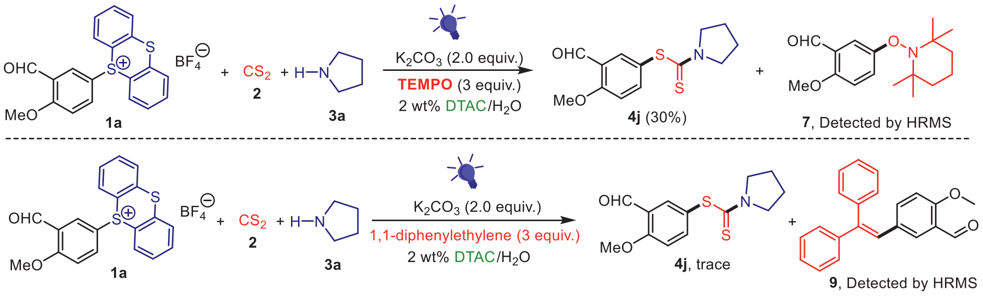
In conclusion, we have successfully developed a novel and efficient visible-light-induced EDA complex process for the synthesis of *S*-aryl dithiocarbamates under mild aqueous micellar conditions. The corresponding products were obtained in moderate to good yields with excellent functional group tolerance. Some important features of the present protocol involve: (a) using water as an environmentally friendly reaction medium; (b) using readily available thianthrenium salts, carbon disulfide and amines as the starting materials; (c) external photocatalyst-free; (d) gram-scale synthesis; and (e) ease of operation. The advantages of the developed method meet the requirements of sustainable and green chemistry. Thus, we expect that the present study will be found wide applications in pharmaceutical chemistry and organic synthesis.

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.



Scheme 4. Synthetic applications.



Scheme 5. Radical trapping experiment.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (No. 22271170), and the Scientific Research Foundation of Qingdao University of Science and Technology.

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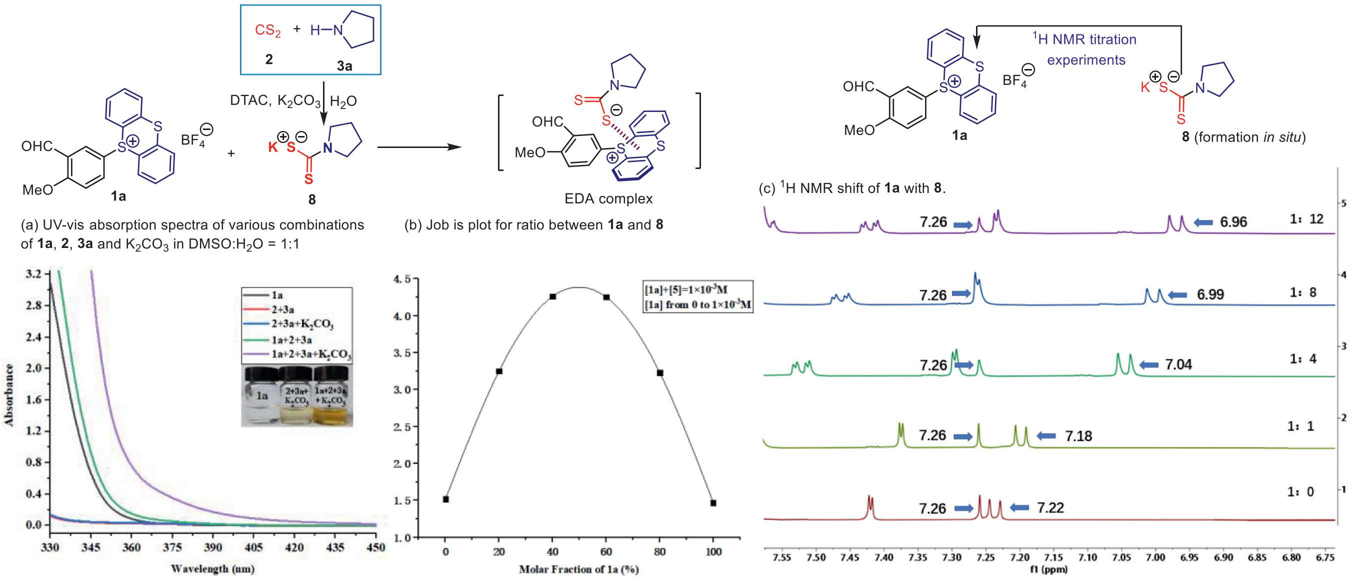
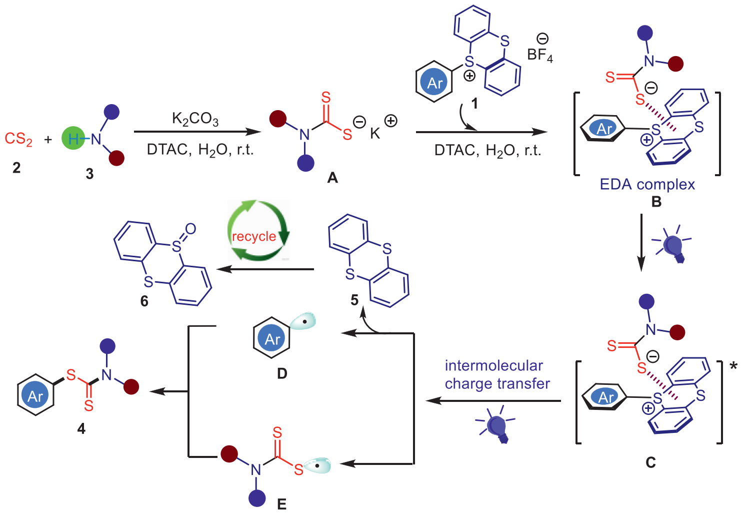


(a) UV-vis absorption spectra of various combinations of **1a**, **2**, **3a** and K_2CO_3 in DMSO:H_2O = 1:1

(b) Job is plot for ratio between **1a** and **8**

(c) ^1H NMR shift of **1a** with **8**.

Fig. 1. Mechanistic studies.



Scheme 6. Possible reaction pathway.

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