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Photoinduced carbonylative synthesis of β -trifluoromethylated thioesters through 1,2-trifluoromethylation and carbonylation of alkenes

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Among carboxylic acids, β -trifluoromethylated carboxylic acid derivatives are important components of drug molecules and have always been one of the research hotspots. In this work, a novel visible light-induced metal-free carbonylation method for the synthesis of β -trifluoromethylated thioesters has been developed. This method has been demonstrated to be an efficient procedure for synthesizing a variety of trifluoromethyl-containing carbonylated compounds. The synthesis of a variety of β -trifluoromethylated

thioesters was accomplished through the utilization of abundant unactivated alkenes and Langlois' reagent, under mild conditions. Concurrently, this method demonstrates good functional group tolerance with medium to excellent yields and good regioselectivity. It is noteworthy that the utilization of Langlois' reagent as a trifluoromethyl source is both cost-effective and environmentally sustainable, with thiosulfonates serving as both endogenous oxidants and sulfur sources.

Green foundation

1. A novel visible light-induced metal-free carbonylation method for the synthesis of β -trifluoromethylated thioesters.
2. Good functional group tolerance with medium to excellent yields and good regioselectivity, using thiosulfonates as both oxidant and sulfur sources.
3. Improve the reaction efficiency and lower CO pressure for further research.

Introduction

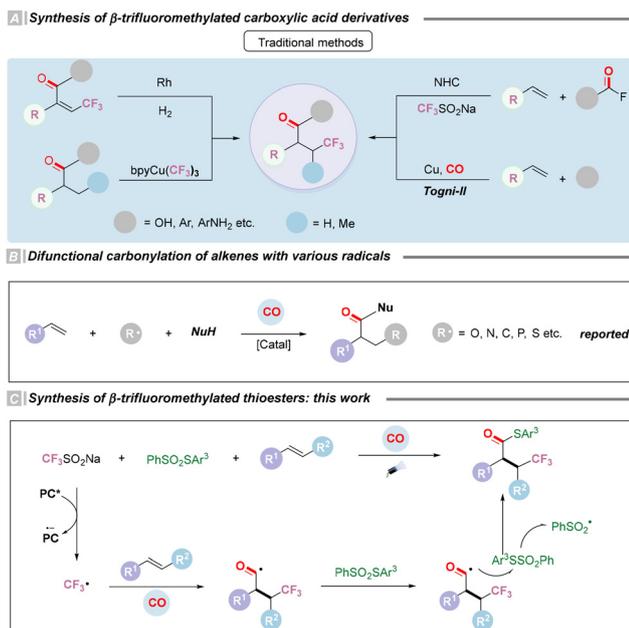
The trifluoromethyl group (CF_3) is a very important moiety in medicinal chemistry due to its physical and biological properties such as good permeability, lipophilicity, metabolic stability, binding selectivity, and bioavailability.^{1–5} In the past few decades, chemists have been working on the introduction of the trifluoromethyl group into organic molecules, and significant progress has been made. Among them, β -trifluoromethylated carboxylic acid derivatives have been one of the important research areas as an important component of drug molecules, for example, β -trifluoromethylated phenylpropionic acid derivatives, which can act as agonists on human peroxisome proliferator-activated receptors.⁶ Therefore, in recent years, chemists have been exploring various efficient and mild methods for the synthesis of β -trifluoromethylated carboxylic acid derivatives. For example, the preparation of

β -trifluoromethylated carboxylic acid derivatives *via* rhodium-catalyzed asymmetric hydrogenation of α - or β -trifluoromethylated acrylic acid was reported in 2013.⁷ β -Trifluoromethylated carboxylic acid derivatives prepared by the conversion of $\text{C}(\text{sp}^3)\text{-H}$ through trifluoromethylation using $\text{CF}_3\text{-Cu}^{\text{III}}$ complexes were reported in 2020.⁸ In the same year, a synergistic NHC/photoredox catalytic system for the synthesis of β -trifluoromethylated alkyl aryl ketones using Langlois' reagent as a CF_3 source was also developed.⁹ In 2021, the synthesis of β -trifluoromethylated acid derivatives by transition metal-catalyzed carbonylation and other methods using Togni II as the CF_3 source was achieved as well (Scheme 1A).¹⁰ Consequently, there is significant interest in exploring green and efficient methods for synthesizing β -trifluoromethylated acid derivatives. On the other hand, thioesters represent significant active structural units that are prevalent in natural products and drug molecules. These compounds are of particular importance as intermediates within the domains of synthetic chemistry and biochemistry, owing to their distinctive chemical and biological properties.^{11–16} Thioesters demonstrate stability in ambient air, thus functioning as reliable acyl donors in the controlled synthesis of ketones, esters, amides,

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Scheme 1 Difunctionalization of alkenes *via* radical addition.

and aldehydes.^{17–26} This property confers upon them a significant role in organic synthesis, providing valuable tools and strategies for chemists. Hence, the development of a new procedure for the synthesis of β -trifluoromethylated thioesters is of significant interest.

Carbon monoxide gas (CO) is an inexpensive bulky industrial compound, and numerous new carbonylation reactions with it have been achieved during the past few decades.^{27–30} Among these achievements, a significant development is the radical carbonylation, which can proceed without a metal catalyst and is promoted by light or heat. On the other hand, alkenes represent a chemical raw material with important applications in chemical synthesis, due to their prevalence and ease of procurement. The difunctionalization reaction involving an alkene can introduce two distinct functional groups on the carbon–carbon double bond concurrently. This reaction is not only a highly efficient method for synthesizing multifunctional compounds, but also an effective approach for constructing complex molecular structures.^{31,32} In recent years, alkene difunctionalized carbonylation reactions *via* transition metal catalysis or photocatalysis were reported, such as those involving perfluoroalkyl, sulfur, and phosphine groups (Scheme 1B).^{33–40} The activation of organic molecules using photocatalysts has been established as a powerful strategy for triggering new chemical reactions in organic synthesis. The reaction conditions are generally mild and environmentally benign, with excellent tolerance of functional groups and high reactivity. However, relatively fewer studies have been reported on intermolecular carbonylative difunctionalization induced by visible light under metal-free conditions.

In accordance with the findings of preceding studies, a range of reactions involving CF_3 radical sources has been docu-

mented, such as $\text{CF}_3\text{SO}_2\text{Cl}$, CF_3I , Umemoto's reagent, and Togni II reagent.^{41–45} However, these CF_3 reagents still have certain disadvantages, such as the corrosiveness and volatility of $\text{CF}_3\text{SO}_2\text{Cl}$, the difficulty of handling CF_3I as a gas, and the fact that Umemoto and Togni II reagents require many synthesis steps and result in a high synthesis cost and low atom efficiency. Consequently, it is advantageous to select a cost-effective and readily manageable source of CF_3 radicals. In this study, Langlois' reagent ($\text{CF}_3\text{SO}_2\text{Na}$) was identified as a promising source of CF_3 radicals, owing to its affordability and accessibility. Additionally, thiosulfonates are considered to be an optimal selection of bifunctional reagents, primarily due to the favorable leaving properties of benzenesulfonates and their capacity to function as both a sulfur source and an oxidizing agent for the photosensitizer, through the generation of an aryl sulfonyl radical. In this study, we present a novel method for synthesizing β -trifluoromethylthioesters under mild conditions *via* a visible light-induced metal-free reaction (Scheme 1C). A variety of β -trifluoromethylated thioesters were accomplished through the utilization of abundant unactivated alkenes and with cost-effective Langlois' reagent and thiosulfonates as both endogenous oxidants and sulfur sources. This method demonstrates good functional group tolerance with medium to excellent yields and good regioselectivity.

Results and discussion

In order to explore the synthesis of β -trifluoromethylated thioesters, but-3-en-1-ylbenzene (**1a**), *S*-phenyl benzenesulfonothioate (**2a**), and $\text{CF}_3\text{SO}_2\text{Na}$ (**3a**) were selected as substrates for the template reaction, 4-CzIPN (1,2,3,5-tetrakis(carbazol-9-yl)-4,6-dicyanobenzene, 2,4,5,6-tetrakis(9*H*-carbazol-9-yl) isophthalonitrile) was chosen as the photocatalyst, and EA was used as the solvent under a carbon monoxide (CO) pressure of 40 bar (Table 1). Initially, a series of substrate ratios were screened, and it was determined that 72% yield of the target product **4ae** was obtained at a ratio of 1 : 1.5 : 2 (Table 1, entry 1). Further reactions with different photocatalysts and without a photocatalyst were conducted (Table 1, entries 2–5). It was ascertained that 4-CzIPN as the photocatalyst yielded the highest yield of the desired product **4ae**. Furthermore, the absence of a photocatalyst resulted in the non-detection of the target product. Then, an additional series of solvents was tested (Table 1, entries 6–9), and it was determined that EA was still the optimal solvent for this transformation. Following the screening of a number of other conditions, the optimal conditions were finally determined (Table 1, entry 1). Notably, only a trace amount of the desired product was detected when the reaction was tested under 5 bar of carbon monoxide.

Under the optimal conditions, the generalizability of substrates for the reaction was checked. Initially, a series of unactivated alkenes were checked (Table 2). The tested straight-chain alkylated alkenes reacted smoothly with $\text{CF}_3\text{SO}_2\text{Na}$ (**2a**)



Table 1 Optimization of reaction conditions^a

Entry	Photocatalysts	Solvent	Yield (%)
1	4-CzIPN	EA	72 (70) ^b
2	PC1	EA	17
3	Ru(bpyl ₃)Cl ₂ ·6H ₂ O	EA	19
4	PC2	EA	57
5	Without photocatalyst	EA	N.D.
6	4-CzIPN	DMF	19
7	4-CzIPN	MeCN	29
8	4-CzIPN	THF	20
9	4-CzIPN	DCM	36

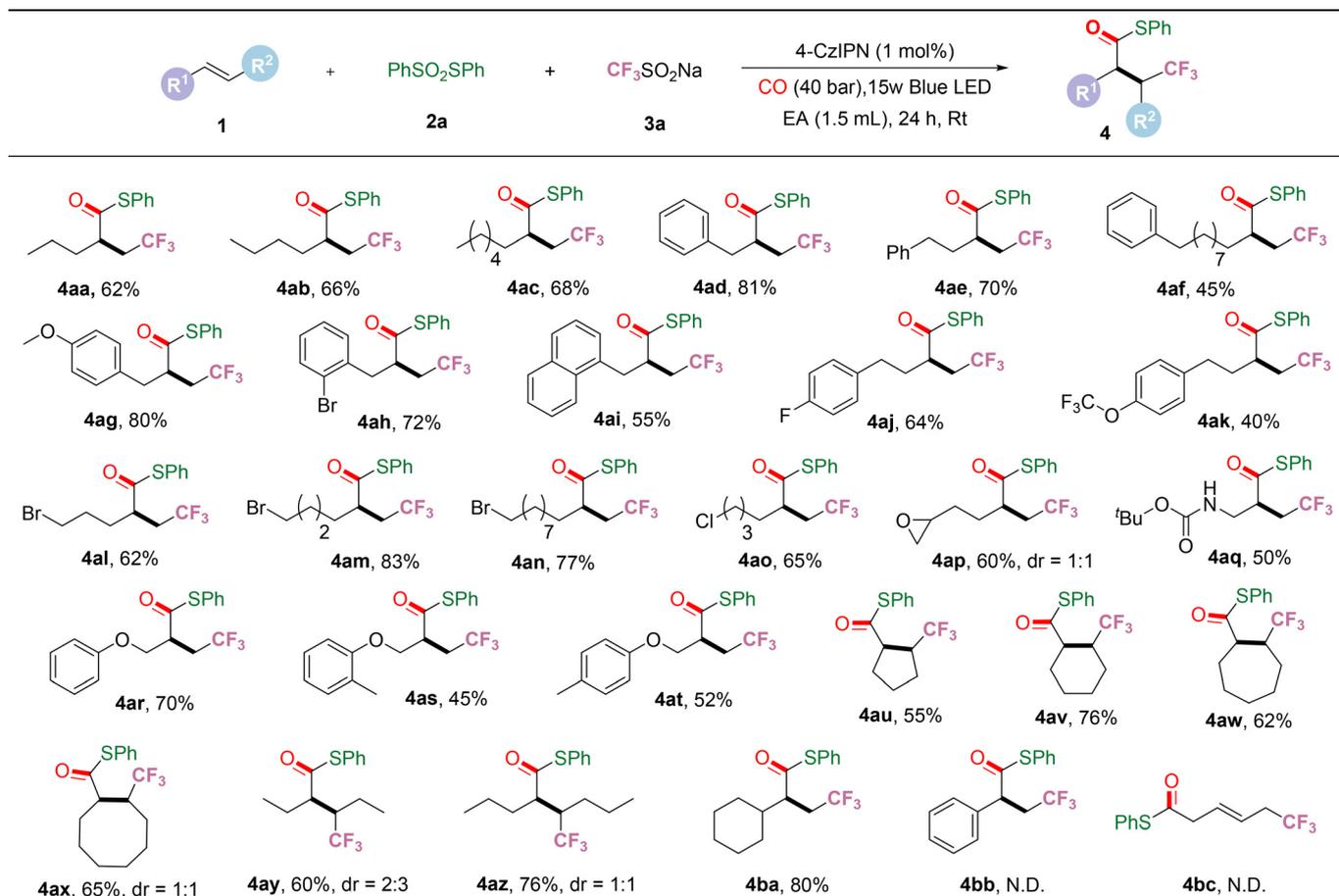
^a Reaction conditions: **1a** (0.1 mmol), **2a** (0.15 mmol), **3a** (0.2 mmol), 4-CzIPN (1 mol%), EA (1.5 mL), Rt, 24 h, and 15 w blue LEDs. The yields were determined by GC using dodecane as the internal standard. ^b Isolated yields. EA: ethyl acetate. DMF: *N,N*-dimethylformamide. DCM: dichloromethane.

and *S*-phenyl benzenesulfonothioate (**3a**). Pent-1-ene, hex-1-ene, and oct-1-ene all gave satisfactory yields of the desired products (**4aa–4ac**). Substrates comprising a phenyl group on the straight-chain alkyl groups were also investigated, with examples including allylbenzene, but-3-en-1-ylbenzene, and undec-10-en-1-ylbenzene. It was observed that the yield decreased in proportion to the length of the carbon chain (**4ad–4af**). Allylbenzenes with a range of substituents on the phenyl group were tested. A substrate with methoxy at the *para*-position and bromide at the *ortho*-position yielded optimal results, while a moderate yield was obtained when the phenyl group was replaced with a naphthalene group (**4ag–4ai**). Subsequently, but-3-en-1-ylbenzene substituted with fluoride and trifluoromethoxy groups was tested, and good yields were obtained for substrates that are *para*-fluoride-substituted. However, relatively low yields were obtained for those that are *para*-trifluoromethoxy-substituted (**4aj** and **4ak**). In the cases of bromo/chloro-substituted unactivated alkenes with varying chain lengths, satisfactory yields of the corresponding products were obtained (**4al–4ao**). Moderate to good yields of the desired products (**4ap** and **4aq**) were obtained when unactivated alkenes substituted with propylene oxide or *tert*-butyl carbamate groups were used. Substrates containing phenoxy groups were then checked, and moderate to good yields were obtained under our standard conditions (**4ar–4at**). Internal olefins as an important class of alkenes were tested without exception. Internal cyclic olefins from five-membered to eight-

membered rings were transformed successfully and gave the corresponding β -trifluoromethylated thioesters in moderate to good yields (**4au–4ax**). Chain-type internal olefins, such as (*E*)-hex-3-ene and (*E*)-oct-4-ene, were then checked under the same conditions, and good yields of the targeted compounds were obtained without any problem (**4ay–4az**). Finally, 80% of the desired β -trifluoromethylated thioester (**4ba**) can be produced from vinylcyclohexane under our standard conditions. However, the reactions failed when styrene or buta-1,3-diene was tested as the substrate (**4bb–4bc**).

Subsequently, the universality testing of thiosulfonates with CF₃SO₂Na (**2a**) and but-3-en-1-ylbenzene (**3a**) was performed (Table 3). Various substitutions at the *para*-position of the phenyl group were tested in the first stage. Electron-donating groups, including methyl, isopropyl, *tert*-butyl, and methoxy, were well tolerated and gave the desired products in moderate to good yields (**4ca–4cd**). Substrates with *para*-electron-withdrawing groups, including bromide, chloride, trifluoromethyl, and cyano, were tested as well, and moderate to good yields of the target products were obtained (**4ce–4ch**). Then, substrates at *meta*- and *ortho*-substitutions were checked, and moderate to excellent yields of the corresponding products (**4ci–4cl**). As an example of heterocyclic substrates, a thiophene-substituted thiosulfonate was tested but only a trace amount of the target product (**4am**) was detected. Finally, alkyl-substituted thiosulfonate substrates were also examined. However, only a trace amount of the desired product



Table 2 Substrate scope of unactivated alkenes^a

^a Reaction conditions: **1** (0.1 mmol), **2a** (0.15 mmol), **3a** (0.2 mmol), 4-CzIPN (1 mol%), EA (1.5 mL), Rt, 24 h, and 15 w blue LEDs. Isolated yields.

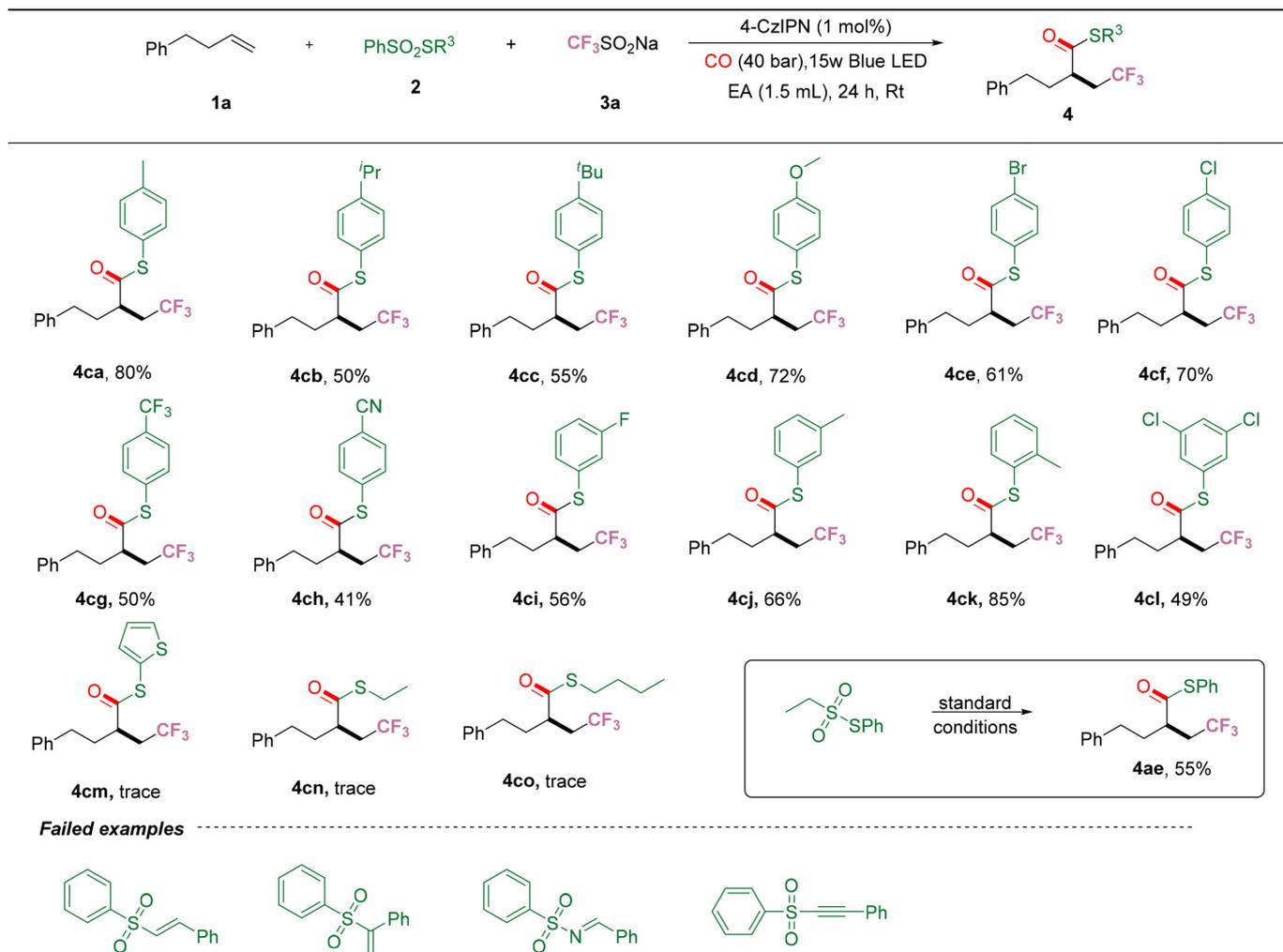
was formed when ethyl- and butyl-substituted thiosulfonates were checked (**4cn**, **4co**). Notably, 55% yield of **4ae** was produced when an ethylsulfonyl-substituted thiosulfonate was used as the substrate. We then proceeded to consider the suitability of other sulfonate analogs for this system, but the attempt failed with (*E*)-2-(phenylsulfonyl)vinylbenzene, (1-(phenylsulfonyl)*v*-inyl)benzene, (*E*)-*N*-benzylidenebenzenesulfonamide, and (phenylethynyl)sulfonylbenzene as the substrates.

In order to get some understanding of the reaction pathway of this transformation, control experiments were conducted (Scheme 2). Initially, radical inhibitors were added to the model system under our standard conditions, and the desired transformation was completely terminated when TEMPO ((2,2,6,6-tetramethylpiperidin-1-yl)oxyl) or BHT (2,6-di-*tert*-butyl-4-methylphenol) was added (Scheme 2A). The results imply that radical intermediates might be involved during the transformation. If we replace the thiosulfonate with diphenyl disulfide, the target product can still be isolated in 32% yield (Scheme 2B). In an attempt to substitute sodium trifluoromethylsulfinate with perfluoroiodobutane, 30% yield of the desired product (**4da**) was formed in the presence of DIPEA (*N*,

N-diisopropylethylamine) as a reducing agent (Scheme 2C). This reveals that the photosensitiser was important for CF₃SO₂Na activation. As one of the advantages of thioesters, the produced compound can be easily transformed into the corresponding ester in the presence of nucleophiles. As we expected, 70% yield of β-trifluoromethylated ester (**4ea**) was generated in the presence of methanol and a base (Scheme 2D). The reaction was also demonstrated to be easily scalable (Scheme 2E).

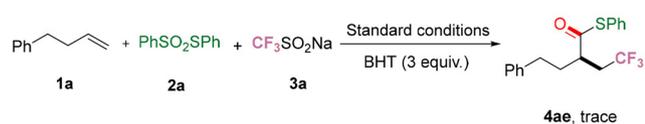
Based on our results and understanding, a possible reaction pathway is proposed (Scheme 3). First, compound **2a** has been shown to produce a trifluoromethyl radical intermediate upon oxidation by a photosensitiser, accompanied by the release of SO₂. Then, the generated trifluoromethyl radical adds to the alkene **1** starting material to yield a new carbon radical. After the capture of a carbon monoxide molecule, acyl radical intermediate **A** will be formed. Finally, the acyl radical results in the formation of the target product through the trapping of aryl sulfur from sulfonyl sulfide. The released sulfonyl radical subsequently serves as an oxidising agent, enabling the oxidation of the photosensitiser to complete the cycle and generate PhSO₂Na as the side product.



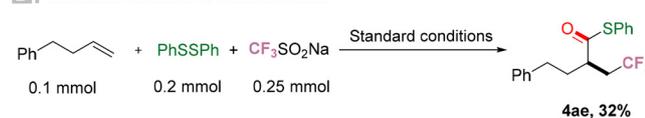
Table 3 Substrate scope of thiosulfonates^a

^a Reaction conditions: **1a** (0.1 mmol), **2** (0.15 mmol), **3a** (0.2 mmol), 4-CzIPN (1 mol%), EA (1.5 mL), Rt, 24 h, and 15 w blue LEDs. Isolated yields.

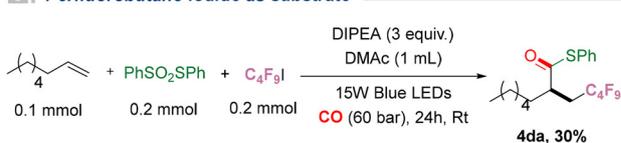
A Radical trapping experiments



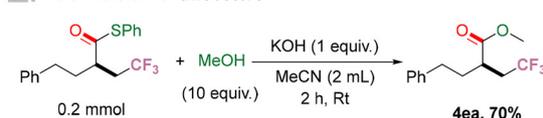
B Disulfide instead of thiosulfonate



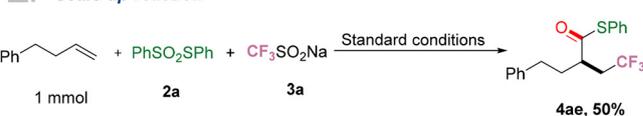
C Perfluorobutane iodide as substrate



D Conversion of thioesters

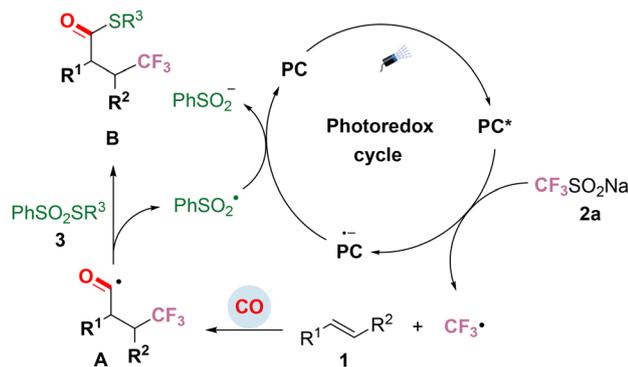


E Scale-up reaction



Scheme 2 Control experiments and synthetic applications.





Scheme 3 Proposed mechanism.

Conclusions

In summary, a new method for synthesising β -trifluoromethylated thioesters has been developed. The reaction proceeds *via* the addition of a trifluoromethyl radical to unactivated alkenes, followed by the capture of the aryl sulfur from the thiosulfonate by acyl radicals in the presence of carbon monoxide to afford valuable β -trifluoromethylated thioesters in good yields under metal-free conditions. The reaction demonstrates good tolerance towards various functional groups. It is noteworthy that thiosulfate salts function as bifunctional reagents in this system, providing the sulfur source and generating aryl sulfonyl radicals that act as endogenous oxidizing agents.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information: general comments, general procedure, analytical data, and NMR spectra. See DOI: <https://doi.org/10.1039/d5gc04881h>.

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