

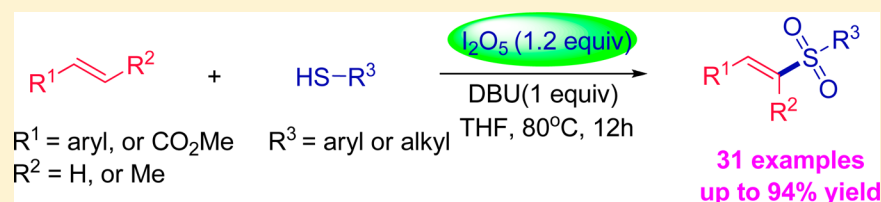
Metal-free Oxidative Coupling of Aromatic Alkenes with Thiols Leading to (*E*)-Vinyl Sulfones

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S Supporting Information

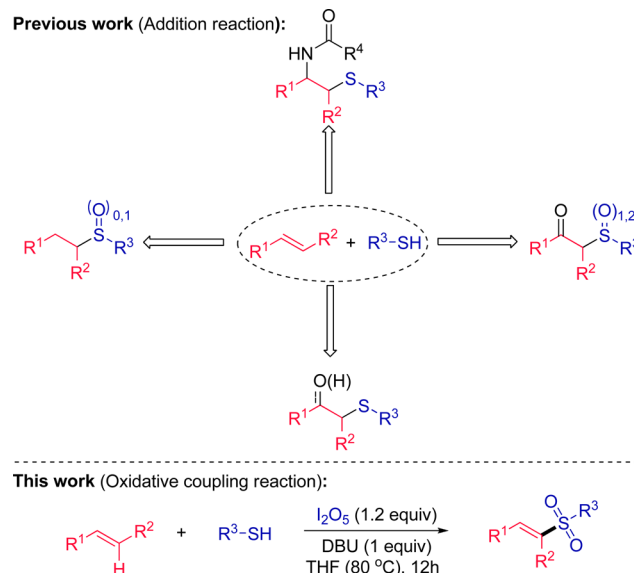


ABSTRACT: A facile I_2O_5 -mediated direct oxidative coupling of aromatic alkenes with thiols toward vinyl sulfones has been developed under metal-free conditions. This methodology provides a convenient and efficient approach to various (*E*)-vinyl sulfones from readily available starting materials with excellent regioselectivity. The present oxidative coupling reaction, not only expands the scope of functionalization of alkenes with thiols, but also makes it a practical and powerful complement to traditional methods for the synthesis of (*E*)-vinyl sulfones.

INTRODUCTION

Sulfur-containing compounds are an important class of chemicals that have wide applications in the field of organic synthesis,¹ materials science,² and medicinal chemistry.³ Thus, the development of a convenient and efficient method for the construction of C–S bond has attracted the increasing synthetic pursuit of chemists in both academic and industrial communities.⁴ In this context, the functionalization of alkenes with thiols represents one of most straightforward and fascinating protocol to access various organosulfur compounds.^{5–10} Over the past decades, some sulfur-containing products, such as thioethers,⁵ sulfoxides,⁶ β -acetamido sulfides,⁷ β -oxy sulfoxides,⁸ β -hydroxy sulfides and sulfoxides,⁹ and β -ketosulfones,¹⁰ have been successfully prepared via the direct addition reaction of alkenes with thiols (Scheme 1). Despite the significance of these functionalization reactions, it is still an attractive but challenging task to develop novel, efficient, and especially, environmentally benign methods to construct other important functional sulfur compounds through direct functionalization of alkenes with thiols. Recently, iodine pentoxide (I_2O_5) as safe and reliable single-electron oxidative surrogates have been increasingly utilized for various synthetically useful transformations because of their ready availability, stability, and low toxicity.¹¹ As part of our continued interest in the construction of sulfur-containing compounds,¹² here, we wish to report a new and facile I_2O_5 -mediated direct oxidative coupling of alkenes with thiols toward (*E*)-vinyl sulfones under metal-free conditions (Scheme 1).

Scheme 1. Methods for the Functionalization of Alkenes with Thiols



Vinyl sulfones as an important class of sulfur-containing compounds play a significant role in pharmaceutical and

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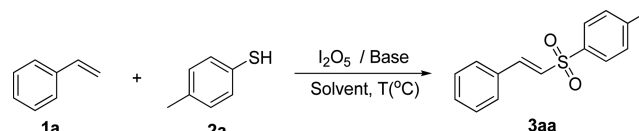
material sciences.¹³ In particular, they can serve as the versatile building blocks to access various useful compounds through a number of organic transformations.¹⁴ The traditional methods to prepare vinyl sulfones are based on the Knoevenagel condensation of aromatic aldehydes with sulfonylacetic acids,¹⁵ Horner–Emmons reaction of carbonyl compounds with sulfonyl phosphoranes,¹⁶ and Wittig reaction.¹⁷ Recently, a series of alternative methods for vinyl sulfones synthesis have also been developed, such as transition-metal-catalyzed the cross-coupling of sulfinate salts with vinyl bromides, vinyl triflates, or vinyl boronic acids;¹⁸ the oxidation of preformed vinyl sulfides with stoichiometric oxidants,¹⁹ the coupling reactions of alkenes or cinnamic acids with sulfonyl halides, sodium sulfonates, and sulfonyl hydrazides;²⁰ and the addition of sulfonic acids, sodium sulfonates, sulfonyl hydrazides, or dimethyl sulfoxide to alkynes.²¹ Nevertheless, most of these reactions might suffer from some limitations, such as the need of inaccessible starting materials, tedious procedures, harsh reaction conditions, low atom economy, and poor regioselectivity or toxic metal catalysts. Therefore, it still remains an attractive task to develop more economic, efficient, and highly selective methods to access vinyl sulfones under metal-free conditions. The present protocol of I_2O_5 -mediated direct oxidative coupling alkenes with thiols provides a facile and efficient approach to various (*E*)-vinyl sulfones in moderate to good yields from simple and commercially available starting materials with high regioselectivity, and does not require the use of any metal-catalysts.

RESULTS AND DISCUSSION

Initially, styrene (**1a**) and 4-methylbenzenethiol (**2a**) were selected as the model substrates to optimize various reaction conditions. As shown in Table 1, none of desired product **3aa** was obtained when the model reaction was performed at room temperature in the presence of I_2O_5 /DBU system (entry 1). To our delight, the yield of desired vinyl sulfone **3aa** was largely improved along with the increasing of the reaction temperature, and the highest yield (55%) of **3aa** was obtained at 80 °C in CH_3CN (entry 4). Subsequently, the screening of a range of solvents demonstrated that the reaction performed in THF was significantly better than those conducted in 1,4-dioxane, DME, DMSO, DMF, EtOH, and H_2O (entries 4–10). Further optimization of various bases showed that DBU was the best choice. Only a trace amount of product or no product was detected when other bases, such as K_2CO_3 , KOH, KO^tBu, and Et_3N , were employed (entries 11–14). The yield of **3a** obviously declined with the decreasing of DBU loading to 0.5 equiv (entry 15) and no conversion was observed in the absence of DBU (entry 16). Next, the investigation on the effect of I_2O_5 loading found that the best yield (87%) was obtained when 1.2 equiv of I_2O_5 was used in the present reaction system (entry 17). Moreover, no conversion was observed when the reaction was performed without I_2O_5 (entry 18).

Under the established reaction conditions, the scope of the I_2O_5 -mediated oxidative coupling of alkenes with thiols for the construction of (*E*)-vinyl sulfones was explored, with the results summarized in Table 2. In general, the reactions of styrene with various aryl thiols bearing electron-rich or electron-poor groups on the aryl rings gave the corresponding products **3aa–3am** in moderate to good yields. It was found that halogen substituents, such as Br, Cl, and F, were all well tolerated, which made this protocol more useful for further structural

Table 1. Optimization of the Reaction Conditions^a

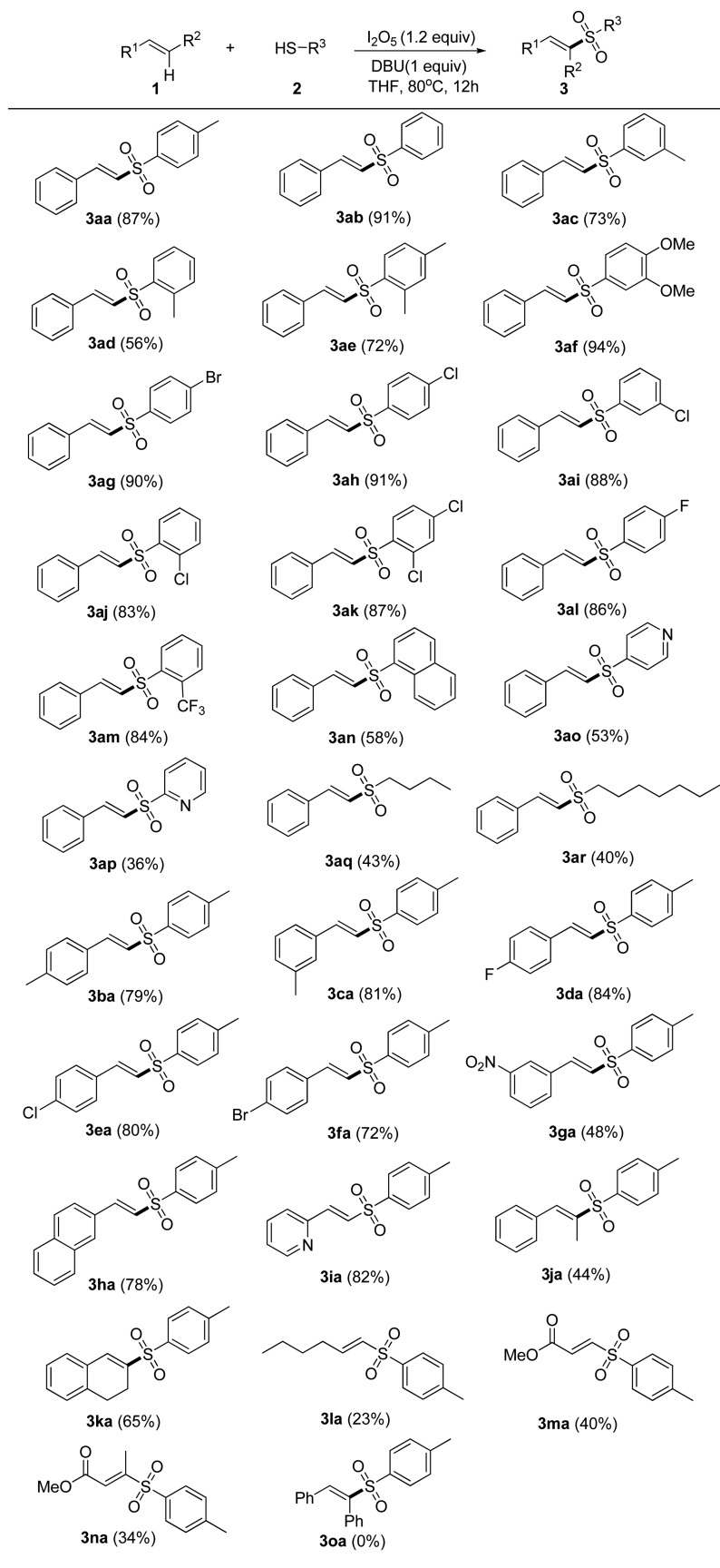


entry	base (equiv)	solvent	T (°C)	yield (%) ^b
1	DBU (1)	CH_3CN	25	0
2	DBU (1)	CH_3CN	50	26
3	DBU (1)	CH_3CN	60	44
4	DBU (1)	CH_3CN	80	55
5	DBU (1)	1,4-dioxane	80	54
6	DBU (1)	DME	80	63
7	DBU (1)	THF	80	75
8	DBU (1)	DMSO	80	58
9	DBU (1)	DMF	80	43
10	DBU (1)	EtOH	80	21
11	DBU (1)	H_2O	80	0
11	K_2CO_3 (1)	THF	80	trace
12	KOH (1)	THF	80	0
13	KO ^t Bu (1)	THF	80	0
14	Et_3N (1)	THF	80	0
15	DBU (0.5)	THF	80	49
16		THF	80	0
17	DBU (1)	THF	80	87 ^c
18	DBU (1)	THF	80	0 ^d

^aReaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), I_2O_5 (1 equiv), base (0–1 equiv), solvent (2 mL), 50–80 °C, 12 h. ^bYields based on **1a**. ^c I_2O_5 (1.2 equiv). ^dWithout I_2O_5 .

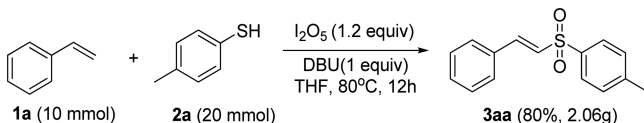
modification (**3ag–3al**). 2-Naphthalenethiol and heterocycle arylthiols, such as pyridine-4-thiol and pyridine-2-thiol, were also compatible with this process, leading to the expected products **3an–3ap** in moderate yields. It is noteworthy that alkyl thiols with long aliphatic chains, such as butane-1-thiol and heptane-1-thiol, could also be used in the reactions to give the corresponding products **3aq** and **3ar** in moderate yields. The scope of this oxidative coupling reaction was further expanded to a variety of alkenes. Aromatic alkenes with electron-donating substituents and with electron-withdrawing substituents were all tolerated under the standard reaction conditions (**3ba–3ga**). As expected, 2-vinylnaphthalene and heterocycle alkene, such as 2-vinylpyridine, could also be unambiguously converted into the desired products **3ha** and **3ia** in 78% and 82% yields, respectively. It should be noted that internal aromatic alkenes (i.e., (*E*)-prop-1-enylbenzene and 1,2-dihydronaphthalene) were also suitable substrates, generating the corresponding products **3ja** and **3ka** in 44% and 65% yields, respectively. Nevertheless, when aliphatic alkene, such as hex-1-ene, was employed in the present reaction system, and the corresponding product **3la** was obtained in relatively low yield. In addition, methyl acrylate and (*E*)-methyl but-2-enoate could also be used in this transformation to produce the desired products (**3ma** and **3na**) in 40% and 34% yields, respectively. Unfortunately, none of the desired product **3oa** was detected when the reaction of stilbene with 4-methylbenzenethiol was conducted under the standard conditions.

Furthermore, to demonstrate the efficiency and practicality of this transformation, a scaled up reaction was conducted between styrene (**1a**) and 4-methylbenzenethiol (**2a**) under the standard conditions. As shown in Scheme 2, the gram-scale synthesis of (*E*)-vinyl sulfone **3aa** was achieved in 80% yield

Table 2. Results for the I_2O_5 -Mediated Oxidative Coupling of Alkenes with Thiols Leading to (*E*)-Vinyl Sulfones^{a,b}

^aReaction conditions: **1** (0.2 mmol), **2** (0.4 mmol), I_2O_5 (0.24 mmol), THF (2 mL), 80 °C, 12 h. ^bYields based on **1**.

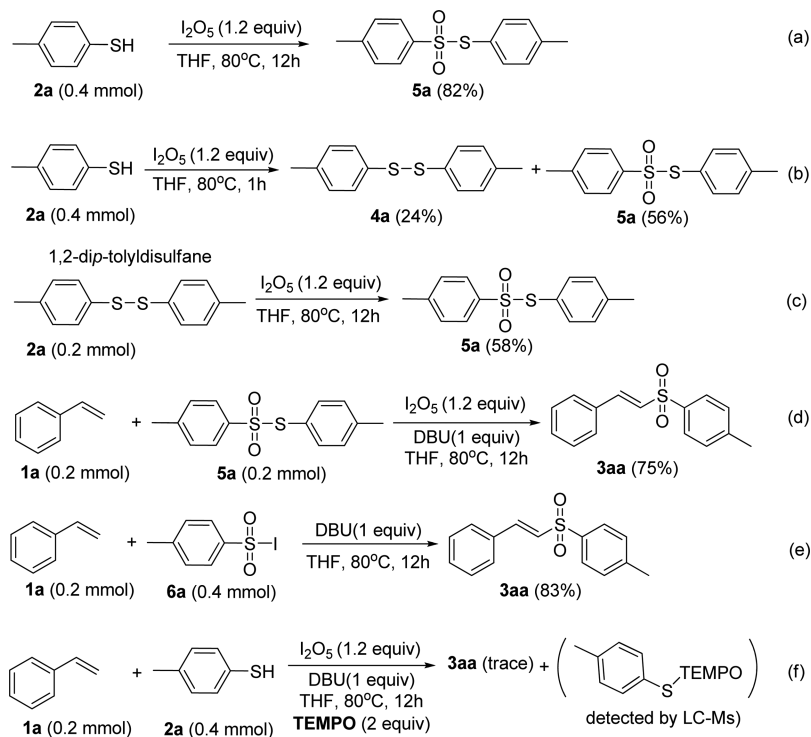
Scheme 2. Gram Scale Reaction



without the significant loss of its efficiency. This result indicates that the present method should be employed as the practical protocol for a large scale synthesis of (*E*)-vinyl sulfones.

In order to gain some further insights into the reaction mechanism, several control experiments were carried out (Scheme 3). Initially, the *p*-tolyl 4-methylbenzenesulfonothioate (5a) was isolated in 82% yield when the reaction of 4-methylbenzenethiol (2a) with I_2O_5 was conducted for 12 h in the absence of styrene and DBU (Scheme 3a). Furthermore, 1,2-dip-tolyldisulfane (4a) was isolated in 24% yield along with *p*-tolyl 4-methylbenzenesulfonothioate 5a (56%) when the reaction of 4-methylbenzenethiol (2a) with I_2O_5 was conducted for 1 h (Scheme 3b). Then, the interaction of 1,2-dip-tolyldisulfane (4a) with I_2O_5 gave *p*-tolyl 4-methylbenzenesulfonothioate (5a) in 58% yield, suggesting that *p*-tolyl 4-methylbenzenesulfonothioate (5a) might be generated from the oxidation of 1,2-dip-tolyldisulfane (4a) (Scheme 3c). Next, we checked the role of *p*-tolyl 4-methylbenzenesulfonothioate (5a) in the formation of product 3aa. As shown in Scheme 3d, the desired product 3aa was obtained in 75% yield when the reaction of styrene (1a) and *p*-tolyl 4-methylbenzenesulfonothioate (5a) was conducted under the standard conditions. Moreover, styrene (1a) reacted with sulfonyl iodide (6a) would lead to the formation of 3aa in 83% yield in the presence of DBU (Scheme 3e). The above results indicated that benzenesulfonothioate and sulfonyl iodide might be the key intermediates in the present reaction system. In addition, the model reaction of 1a and 2a was extremely inhibited by the

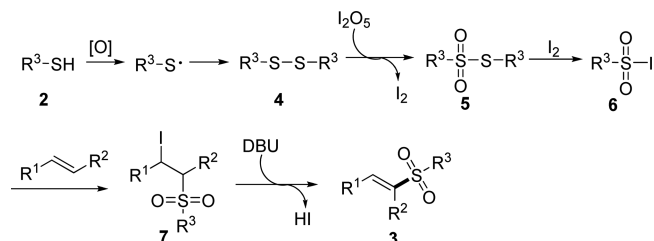
Scheme 3. Control Experiments



addition of radical scavenger 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) and TEMPO-trapped thiyl radical complex (*p*-MePhS-TEMPO) was detected by LC-MS experiment (see Supporting Information), suggesting that the present reaction might proceed via a proposed radical pathway (Scheme 3f).

Based on the above the experimental results and previous reports,^{20,22,23} a possible reaction pathway was proposed as shown in Scheme 4. Initially, thiyl radical was generated from

Scheme 4. Postulated Reaction Pathway



the oxidation of thiophenol 2,^{22a} Subsequently, the dimerization of thiyl radical generated disulfide 4,^{22b} which was further oxidized by I_2O_5 would produce benzenesulfonothioate 5 with the concomitant formation of molecular iodine.²² Then, the interaction of benzenesulfonothioate 5 with iodine gave the sulfonyl iodide 6. Next, the addition of sulfonyl iodide 6 to alkene 1 afforded β-iodo sulfone 7. Finally, the elimination of HI from β-iodo sulfone with the assistance of DBU provided the desired product 3.^{20a,d,23}

CONCLUSIONS

In summary, a novel and facile method has been successfully developed for the construction of (*E*)-vinyl sulfones through metal-free I_2O_5 -promoted direct oxidative coupling of aromatic

alkenes with thiols. Some notable features can be obtained for this reaction, such as operation simplicity, ready availability of starting materials, metal-free, oxidant of low toxicity, and high regio- and stereoselectivity, thus making it a practical and powerful complement to traditional methods for the construction of (*E*)-vinyl sulfones.

EXPERIMENTAL SECTION

General. Chemicals were commercially available and were used without further purification unless otherwise stated. All solvents were used as received without further purification unless otherwise stated. ^1H NMR and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra were obtained in CDCl_3 with TMS as internal standard (500 MHz ^1H and 125 MHz ^{13}C) at room temperature, the chemical shifts (δ) were expressed in ppm and J values were given in Hz. The following abbreviations are used to indicate the multiplicity: singlet (s), doublet (d), triplet (t), quartet (q), doublet of doublets (dd), doublet of triplets (dt), doublet of quartets (dq), and multiplet (m). All first order splitting patterns were assigned on the basis of the appearance of the multiplet. Splitting patterns that could not be easily interpreted were designated as multiplet (m). In a few cases the number of signals in the ^{13}C NMR spectrum is than due, which may be caused by the superimposition of signals. HRMS data were obtained by ESI on a TOF mass analyzer. Column chromatography was performed on silica gel (100–200 mesh).

General Experimental Procedure. In a tube (25 mL), alkene **1** (0.2 mmol), thiol **2** (0.4 mmol), I_2O_5 (0.24 mmol, 80.1 mg), DBU (0.2 mmol, 30 μL), and THF (2 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 80 °C for 12 h. After the reaction, the solvent was removed under vacuum. The residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **3**.

Gram-Scale Reaction. In a tube (100 mL), styrene (10 mmol, 1.15 mL), 4-methylbenzenethiol (20 mmol, 2.5 g), I_2O_5 (12 mmol, 4.0 g), DBU (10 mmol, 1.5 mL), and THF (20 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 80 °C for 12 h. After the reaction, the resulting mixture was concentrated under vacuum and the residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **3aa** in 80% yield (2.06 g).

Reaction of 4-Methylbenzenethiol (2a) with I_2O_5 (12h). In a seal tube (15 mL), 4-methylbenzenethiol **2a** (0.4 mmol, 49.6 mg), I_2O_5 (0.24 mmol, 80.1 mg), and THF (2 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 80 °C for 12 h. After the reaction, the solution was concentrated in vacuum, the product **5a** was isolated in 82% yield (45.6 mg).

Reaction of 4-Methylbenzenethiol (2a) with I_2O_5 (1h). In a seal tube (15 mL), 4-methylbenzenethiol **2a** (0.4 mmol, 49.6 mg), I_2O_5 (0.24 mmol, 80.1 mg), and THF (2 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 80 °C for 1 h. After the reaction, the solution was concentrated in vacuum, the product **4a** and **5a** were isolated in 24% (11.8 mg) and 56% (31.1 mg) yields, respectively.

Reaction of 1,2-Dip-tolyldisulfane (4a) with I_2O_5 . In a seal tube (15 mL), 1,2-dip-tolyldisulfane (**4a**) (0.2 mmol, 49.2 mg), I_2O_5 (0.24 mmol, 80.1 mg), and THF (2 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 80 °C for 12 h. After the reaction, the solution was concentrated in vacuum, the product **5a** were isolated in 58% yield (32.2 mg).

Reaction of Styrene (1a) and *p*-Tolyl 4-Methylbenzenesulfonothioate (5a) under Standard Conditions. In a seal tube (15 mL), styrene (0.2 mmol, 23 μL), *p*-tolyl 4-methylbenzenesulfonothioate (0.2 mmol, 55.6 mg), I_2O_5 (0.24 mmol, 80.1 mg), DBU (0.2 mmol, 30 μL), and THF (2 mL) were added. Then, the tube was sealed and the reaction vessel was allowed to stir at 80 °C for 12 h. After the reaction, the resulting mixture was concentrated under vacuum and the residue was purified by flash column chromatography using a mixture of

petroleum ether and ethyl acetate as eluent to give the desired product **3aa** in 75% yield (38.7 mg).

Reaction of Styrene (1a) with Sulfonyl Iodide (6a) in the Presence of DBU. In a seal tube (15 mL), styrene (0.2 mmol, 23 μL), 4-methylbenzene-1-sulfonyl iodide (0.4 mmol, 112.8 mg), DBU (0.2 mmol, 30 μL), and THF (2 mL) were added. The reaction vessel was allowed to stir at 80 °C for 12 h. After the reaction, the resulting mixture was concentrated under vacuum and the residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent to give the desired product **3aa** in 83% yield (42.8 mg).

Radical Trapping Experiment with TEMPO. In a seal tube (15 mL), styrene (0.2 mmol, 23 μL), 4-methylbenzenethiol (0.4 mmol, 49.6 mg), TEMPO (0.4 mmol, 62.5 mg), I_2O_5 (0.24 mmol, 80.1 mg), DBU (0.2 mmol, 30 μL), and THF (2 mL) were added. The reaction vessel was allowed to stir for 12 h at 80 °C. After the reaction, the solution was concentrated in vacuum, only a trace amount of desired product **3aa** was detected and TEMPO-trapped thyl radical complex (*p*-MePhS–TEMPO) was also detected by LC-MS experiment (see Supporting Information).

(*E*)-(2-(Phenylsulfonyl)vinyl)benzene (3aa). Compound **3aa** was obtained in 87% yield (44.9 mg) according to the general procedure. White solid; mp: 124.0–124.9 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.85 (d, J = 8.3 Hz, 2H), 7.68 (d, J = 15.5 Hz, 1H), 7.51–7.49 (m, 2H), 7.42–7.41 (m, 3H), 7.37 (d, J = 8.1 Hz, 2H), 6.87 (d, J = 15.4 Hz, 1H), 2.46 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 144.4, 141.9, 137.8, 132.5, 131.1, 130.0, 129.1, 128.5, 127.7, 127.6, 21.6; HRMS calc. for $\text{C}_{15}\text{H}_{14}\text{O}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 281.0612; found, 281.0616.

(*E*)-(2-(Phenylsulfonyl)vinyl)benzene (3ab). Compound **3ab** was obtained in 91% yield (44.4 mg) according to the general procedure. Yellow solid; mp: 71.7–73.0 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.96 (d, J = 7. Four Hz, 2H), 7.70 (d, J = 15. Four Hz, 1H), 7.64–7.61 (m, 1H), 7.55 (t, J = 7.3 Hz, 2H), 7.50–7.48 (m, 2H), 7.42–7.38 (m, 3H), 6.86 (d, J = 15. Four Hz, 1H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 142.5, 140.7, 133.4, 132.4, 131.3, 129.4, 129.1, 128.6, 127.7, 127.3; HRMS calc. for $\text{C}_{14}\text{H}_{12}\text{O}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 267.0456; found, 267.04569.

(*E*)-1-Methyl-3-(styrylsulfonyl)benzene (3ac). Compound **3ac** was obtained in 73% yield (37.6 mg) according to the general procedure. Yellow oil. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.75–7.74 (m, 2H), 7.68 (d, J = 15.4 Hz, 1H), 7.50–7.48 (m, 2H), 7.44–7.38 (m, 5H), 6.86 (d, J = 15.4 Hz, 1H), 2.44 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 142.3, 140.5, 139.7, 134.2, 132.4, 131.2, 129.2, 129.1, 128.6, 128.0, 127.4, 124.8, 21.4; HRMS calc. for $\text{C}_{15}\text{H}_{14}\text{O}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 281.0612; found, 281.0613.

(*E*)-1-Methyl-2-(styrylsulfonyl)benzene (3ad). Compound **3ad** was obtained in 56% yield (28.9 mg) according to the general procedure. Yellow oil. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 8.12–8.10 (m, 1H), 7.69 (d, J = 15.4 Hz, 1H), 7.52–7.48 (m, 3H), 7.42–7.37 (m, 4H), 7.30 (d, J = 7.8 Hz, 1H), 6.85 (d, J = 15.3 Hz, 1H), 2.63 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 142.9, 138.3, 138.1, 133.6, 132.6, 132.4, 131.2, 129.5, 129.1, 128.6, 126.7, 20.4; HRMS calc. for $\text{C}_{15}\text{H}_{14}\text{O}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 281.0612; found, 281.0615.

(*E*)-2,4-Dimethyl-1-(styrylsulfonyl)benzene (3ae). Compound **3ae** was obtained in 72% yield (39.1 mg) according to the general procedure. Yellow solid; mp: 74.6–75.3 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.99 (d, J = 8.1 Hz, 1H), 7.66 (d, J = 15.4 Hz, 1H), 7.49–7.48 (m, 2H), 7.42–7.39 (m, 3H), 7.19 (d, J = 8.1 Hz, 1H), 7.10 (s, 1H), 6.83 (d, J = 15.4 Hz, 1H), 2.59 (s, 3H), 2.38 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 144.5, 142.3, 137.9, 135.4, 133.3, 132.5, 131.1, 129.7, 129.1, 128.5, 127.4, 127.1, 21.4, 20.3; HRMS calc. for $\text{C}_{16}\text{H}_{16}\text{O}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 295.0769; found, 295.0771.

(*E*)-1,2-Dimethoxy-4-(styrylsulfonyl)benzene (3af). Compound **3af** was obtained in 94% yield (57.1 mg) according to the general procedure. Yellow solid; mp: 101.9–103.0 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.63 (d, J = 15.5 Hz, 1H), 7.58–7.56 (m, 1H), 7.49–7.47 (m, 2H), 7.42–7.38 (m, 4H), 6.98 (d, J = 8.5 Hz, 1H), 6.86 (d, J = 15.5 Hz, 1H), 3.95 (3H), 3.94 (3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 153.2, 149.3, 141.4, 132.5, 132.3, 131.1, 129.1, 128.5,

127.8, 121.9, 111.0, 109.8, 56.3, 56.3; HRMS calc. for $C_{16}H_{16}O_4SNa$ ($M+Na$)⁺, 327.0667; found, 327.0669.

(E)-1-Bromo-4-(styrylsulfonyl)benzene (3ag). Compound **3ag** was obtained in 90% yield (57.9 mg) according to the general procedure. White solid; mp: 101.2–102.6 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.81 (d, *J* = 8.7 Hz, 2H), 7.70–7.67 (m, 3H), 7.50–7.48 (m, 2H), 7.43–7.40 (m, 3H), 6.83 (d, *J* = 15.4 Hz, 1H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 143.1, 139.8, 132.7, 132.2, 131.5, 129.2, 129.2, 128.7, 126.8; HRMS calc. for $C_{14}H_{11}BrO_2SNa$ ($M+Na$)⁺, 344.9561; found, 344.9563.

(E)-1-Chloro-4-(styrylsulfonyl)benzene (3ah). Compound **3ah** was obtained in 91% yield (50.6 mg) according to the general procedure. White solid; mp: 83.4–84.5 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.89 (d, *J* = 8.7 Hz, 2H), 7.69 (d, *J* = 15.4 Hz, 1H), 7.52 (d, *J* = 8.6 Hz, 2H), 7.49 (d, *J* = 6.4 Hz, 2H), 7.43–7.38 (m, 3H), 6.83 (d, *J* = 15.4 Hz, 1H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 143.1, 140.1, 139.3, 132.2, 131.4, 129.7, 129.2, 129.2, 128.7, 126.9; HRMS calc. for $C_{14}H_{11}O_2ClSNa$ ($M+Na$)⁺, 301.0066; found, 301.0069.

(E)-1-Chloro-3-(styrylsulfonyl)benzene (3ai). Compound **3ai** was obtained in 88% yield (48.9 mg) according to the general procedure. White solid; mp: 71.1–72.7 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.94 (t, *J* = 1.8 Hz, 1H), 7.84 (d, *J* = 7.9 Hz, 1H), 7.71 (d, *J* = 15.4 Hz, 1H), 7.59 (d, *J* = 8.1 Hz, 1H), 7.51–7.48 (m, 3H), 7.44–7.39 (m, 3H), 6.85 (d, *J* = 15.4 Hz, 1H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 143.5, 142.6, 135.6, 133.5, 132.1, 131.5, 130.7, 129.2, 128.7, 127.7, 126.6, 125.8; HRMS calc. for $C_{14}H_{11}O_2ClSNa$ ($M+Na$)⁺, 301.0066; found, 301.0067.

(E)-1-Chloro-2-(styrylsulfonyl)benzene (3aj). Compound **3aj** was obtained in 83% yield (46.1 mg) according to the general procedure. White solid; mp: 93.3–94.0 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 8.23 (dd, *J*₁ = 1.5 Hz, *J*₂ = 8.0 Hz, 1H), 7.77 (d, *J* = 15.4 Hz, 1H), 7.57–7.51 (m, 4H), 7.49–7.46 (m, 1H), 7.44–7.39 (m, 3H), 7.08 (d, *J* = 15.4 Hz, 1H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 145.3, 138.3, 134.6, 132.8, 132.4, 131.9, 131.5, 130.7, 129.2, 128.7, 127.5, 125.3; HRMS calc. for $C_{14}H_{11}O_2ClSNa$ ($M+Na$)⁺, 301.0066; found, 301.0065.

(E)-2,4-Dichloro-1-(styrylsulfonyl)benzene (3ak). Compound **3ak** was obtained in 87% yield (54.3 mg) according to the general procedure. White solid; mp: 80.8–81.4 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 8.16 (d, *J* = 8.6 Hz, 1H), 7.77 (d, *J* = 15.4 Hz, 1H), 7.53–7.51 (m, 3H), 7.46–7.40 (m, 4H), 7.03 (d, *J* = 15.4 Hz, 1H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 145.8, 140.5, 136.9, 133.8, 132.2, 131.8, 131.7, 131.6, 129.2, 128.8, 127.8, 124.9; HRMS calc. for $C_{14}H_{10}O_2Cl_2SNa$ ($M+Na$)⁺, 334.9676; found, 334.9678.

(E)-1-Fluoro-4-(styrylsulfonyl)benzene (3al). Compound **3al** was obtained in 86% yield (45.1 mg) according to the general procedure. White solid; mp: 78.8–80.1 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.98–7.95 (m, 2H), 7.68 (d, *J* = 15.4 Hz, 1H), 7.50–7.48 (m, 2H), 7.43–7.39 (m, 3H), 7.22 (d, *J* = 8.5 Hz, 1H), 6.84 (d, *J* = 15.4 Hz, 1H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 165.6 (d, *J* = 254.5 Hz), 142.7, 136.8 (d, *J* = 3.1 Hz), 132.2, 131.4, 130.5 (d, *J* = 254.5 Hz), 129.2, 128.6, 127.1, 116.8 (d, *J* = 22.5 Hz); HRMS calc. for $C_{14}H_{11}FO_2SNa$ ($M+Na$)⁺, 285.0361; found, 285.0363.

(E)-1-(Styrylsulfonyl)-2-(trifluoromethyl)benzene (3am). Compound **3am** was obtained in 84% yield (52.4 mg) according to the general procedure. Yellow solid; mp: 79.7–81.2 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 8.36 (d, *J* = 7.4 Hz, 1H), 7.90 (d, *J* = 7.2 Hz, 1H), 7.79–7.76 (m, 2H), 7.70 (d, *J* = 15.4 Hz, 1H), 7.50–7.49 (m, 2H), 7.43–7.38 (m, 3H), 6.99 (d, *J* = 15.4 Hz, 1H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 144.4, 139.8 (d, *J* = 1.0 Hz), 133.5, 132.8, 132.3, 131.9, 131.5, 129.2, 129.0, 128.7, 128.4 (q, *J* = 6.2 Hz), 126.9 (q, *J* = 2.7 Hz), 122.7 (q, *J* = 272.3 Hz); HRMS calc. for $C_{15}H_{11}O_2F_3SNa$ ($M+Na$)⁺, 335.0330; found, 335.0331.

(E)-2-(Styrylsulfonyl)naphthalene (3an). Compound **3an** was obtained in 58% yield (34.1 mg) according to the general procedure. Yellow solid; mp: 129.9–131.2 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 8.55 (s, 1H), 8.00–7.97 (m, 2H), 7.92–7.88 (m, 2H), 7.75 (d, *J* = 15.4 Hz, 1H), 7.67–7.60 (m, 2H), 7.49 (d, *J* = 7.5 Hz, 2H), 7.41–7.37 (m, 3H), 6.92 (d, *J* = 15.4 Hz, 1H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 142.6, 137.5, 135.2, 132.4, 132.3, 131.3, 129.7,

129.4, 129.3, 129.2, 129.1, 128.6, 128.0, 127.7, 127.3, 122.6; HRMS calc. for $C_{18}H_{14}O_2SNa$ ($M+Na$)⁺, 317.0612; found, 317.0614.

(E)-4-(Styrylsulfonyl)pyridine (3ao). Compound **3ao** was obtained in 53% yield (25.9 mg) according to the general procedure. Yellow solid; mp: 83.1–84.0 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 8.88 (s, *J* = 6.0 Hz, 2H), 7.81–7.79 (m, 2H), 7.77 (d, *J* = 15.4 Hz, 1H), 7.52–7.50 (m, 2H), 7.48–7.41 (m, 3H), 6.86 (d, *J* = 15.4 Hz, 1H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 151.3, 149.0, 145.2, 131.9, 129.3, 128.9, 125.4, 120.6; HRMS calc. for $C_{13}H_{11}NO_2SNa$ ($M+Na$)⁺, 268.0408; found, 268.0409.

(E)-2-(Styrylsulfonyl)pyridine (3ap). Compound **3ap** was obtained in 36% yield (17.6 mg) according to the general procedure. Yellow solid; mp: 86.7–88.1 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 8.75 (d, *J* = 4.2 Hz, 1H), 8.15 (d, *J* = 7.9 Hz, 1H), 7.98–7.94 (dt, *J*₁ = 1.7 Hz, *J*₂ = 7.8 Hz, 1H), 7.79 (d, *J* = 15.5 Hz, 1H), 7.55–7.51 (m, 3H), 7.45–7.39 (m, 3H), 7.12 (d, *J* = 15.5 Hz, 1H); ¹³C NMR (CDCl₃, 125 MHz, ppm): δ 158.6, 150.4, 145.2, 138.2, 132.4, 131.4, 129.1, 128.8, 127.1, 124.6, 121.9; HRMS calc. for $C_{13}H_{11}NO_2SNa$ ($M+Na$)⁺, 268.0408; found, 268.0411.

(E)-2-(Butylsulfonyl)vinyl)benzene (3aq). Compound **3aq** was obtained in 43% yield (19.2 mg) according to the general procedure. Yellow solid; mp: 83.1–84.0 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.60 (d, *J* = 15.5 Hz, 1H), 7.54–7.52 (m, 2H), 7.45–7.43 (m, 3H), 6.83 (d, *J* = 15.5 Hz, 1H), 3.06 (t, *J* = 8.1 Hz, 2H), 1.85–1.78 (m, 2H), 1.50–1.45 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 144.8, 132.3, 131.3, 129.2, 128.6, 124.7, 55.0, 24.6, 21.7, 13.6; HRMS calc. for $C_{12}H_{16}O_2SNa$ ($M+Na$)⁺, 247.0769; found, 247.0771.

(E)-2-(Heptylsulfonyl)vinyl)benzene (3ar). Compound **3ar** was obtained in 40% yield (21.3 mg) according to the general procedure. Yellow oil. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.60 (d, *J* = 15.5 Hz, 1H), 7.52 (d, *J* = 7.1 Hz, 2H), 7.43 (m, 3H), 6.83 (d, *J* = 15.5 Hz, 1H), 3.06 (t, *J* = 8.1 Hz, 2H), 1.86–1.79 (m, 2H), 1.45–1.40 (m, 2H), 1.34–1.26 (m, 6H), 0.87 (t, *J* = 5.9 Hz, 3H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 144.7, 132.3, 131.3, 129.2, 128.6, 124.8, 55.2, 31.4, 28.7, 28.4, 22.6, 22.5, 14.0; HRMS calc. for $C_{15}H_{22}O_2SNa$ ($M+Na$)⁺, 289.1238; found, 289.1239.

(E)-1-Methyl-4-(4-methylstyrylsulfonyl)benzene (3ba). Compound **3ba** was obtained in 79% yield (42.9 mg) according to the general procedure. Yellow solid; mp: 145.7–147.2 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.84 (d, *J* = 8.3 Hz, 2H), 7.65 (d, *J* = 15.4 Hz, 1H), 7.40–7.35 (m, 4H), 7.21 (d, *J* = 8.0 Hz, 2H), 6.81 (d, *J* = 15.4 Hz, 1H), 2.45 (s, 3H), 2.39 (s, 3H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 144.3, 142.0, 141.7, 138.0, 129.9, 129.8, 129.7, 128.5, 127.7, 126.4, 21.6, 21.5; HRMS calc. for $C_{16}H_{16}O_2SNa$ ($M+Na$)⁺, 295.0769; found, 295.0771.

(E)-1-Methyl-3-(2-tosylvinyl)benzene (3ca). Compound **3ca** was obtained in 81% yield (44.0 mg) according to the general procedure. White solid; mp: 93.4–94.9 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.85 (d, *J* = 8.3 Hz, 2H), 7.65 (d, *J* = 15.4 Hz, 1H), 7.36 (d, *J* = 8.2 Hz, 2H), 7.31–7.29 (m, 3H), 7.25–7.23 (m, 1H), 6.85 (d, *J* = 15.4 Hz, 1H), 2.46 (s, 3H), 2.37 (s, 3H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 144.3, 142.1, 138.8, 137.9, 132.4, 132.0, 130.0, 129.1, 129.0, 127.7, 127.4, 125.8, 21.6, 21.3; HRMS calc. for $C_{16}H_{16}O_2SNa$ ($M+Na$)⁺, 295.0769; found, 295.0767.

(E)-1-Fluoro-4-(2-tosylvinyl)benzene (3da). Compound **3da** was obtained in 84% yield (46.3 mg) according to the general procedure. Yellow solid; mp: 92.5–93.9 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.82 (d, *J* = 8.3 Hz, 2H), 7.62 (d, *J* = 15.4 Hz, 1H), 7.49–7.45 (m, 2H), 7.35 (d, *J* = 8.3 Hz, 2H), 7.08 (t, *J* = 8.6 Hz, 2H), 6.78 (d, *J* = 15.4 Hz, 1H), 2.44 (s, 3H); ¹³C{¹H}NMR (CDCl₃, 125 MHz, ppm): δ 164.3 (d, *J* = 251.3 Hz), 144.5, 140.6, 137.7, 130.6 (d, *J* = 8.7 Hz), 130.0, 128.7 (d, *J* = 8.7 Hz), 127.7, 127.4 (d, *J* = 2.4 Hz), 116.3 (d, *J* = 21.9 Hz), 21.6; HRMS calc. for $C_{15}H_{13}O_2FSNa$ ($M+Na$)⁺, 299.0518; found, 299.0519.

(E)-1-Chloro-4-(2-tosylvinyl)benzene (3ea). Compound **3ea** was obtained in 80% yield (46.7 mg) according to the general procedure. Yellow solid; mp: 143.7–144.8 °C. ¹H NMR (CDCl₃, 500 MHz, ppm): δ 7.84 (d, *J* = 8.3 Hz, 2H), 7.62 (d, *J* = 15.5 Hz, 1H), 7.43 (d, *J* = 8.6 Hz, 2H), 7.39–7.36 (m, 4H), 6.85 (d, *J* = 15.4 Hz, 1H), 2.46 (s,

3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 144.6, 140.4, 137.5, 137.1, 131.0, 130.0, 129.7, 129.4, 128.3, 127.8, 21.6; HRMS calc. for $\text{C}_{15}\text{H}_{13}\text{O}_2\text{ClSNa}$ ($\text{M}+\text{Na}$) $^+$, 315.0222; found, 315.0225.

(*E*)-1-Bromo-4-(2-tosylvinyl)benzene (**3fa**). Compound **3fa** was obtained in 72% yield (48.4 mg) according to the general procedure. Yellow solid; mp: 153.8–155.4 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.85–7.84 (m, 2H), 7.61 (d, J = 15.4 Hz, 1H), 7.55–7.54 (m, 2H), 7.36 (t, J = 8.3 Hz, 4H), 6.86 (d, J = 15.4 Hz, 1H), 2.46 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 144.6, 140.5, 137.5, 132.4, 131.4, 130.0, 129.9, 128.4, 127.8, 125.5, 21.7; HRMS calc. for $\text{C}_{15}\text{H}_{13}\text{BrO}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 358.9717; found, 358.9719.

(*E*)-1-Nitro-3-(2-tosylvinyl)benzene (**3ga**). Compound **3ga** was obtained in 48% yield (29.1 mg) according to the general procedure. Yellow oil. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 8.35 (t, J = 1.9 Hz, 1H), 8.29–8.26 (m, 1H), 7.87–7.86 (d, J = 8.4 Hz, 2H), 7.81 (d, J = 7.8 Hz, 1H), 7.71 (d, J = 15.4 Hz, 1H), 7.62 (t, J = 8.0 Hz, 1H), 7.40 (d, J = 8.2 Hz, 2H), 7.03 (d, J = 15.5 Hz, 1H), 2.48 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 148.7, 145.0, 138.8, 136.9, 134.3, 134.2, 131.1, 130.2, 130.2, 128.0, 125.3, 122.7, 21.7; HRMS calc. for $\text{C}_{15}\text{H}_{13}\text{NO}_4\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 326.0463; found, 326.0465.

(*E*)-2-(2-Tosylvinyl)naphthalene (**3ha**). Compound **3ha** was obtained in 78% yield (48.0 mg) according to the general procedure. Yellow solid; mp: 159.8–160.3 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.93 (s, 1H), 7.87–7.79 (m, 6H), 7.55–7.52 (m, 3H), 7.36 (d, J = 8.3 Hz, 2H), 6.95 (d, J = 15.4 Hz, 1H), 2.44 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 144.4, 142.0, 137.9, 134.5, 133.1, 130.9, 130.0, 129.9, 129.0, 128.7, 127.8, 127.8, 127.7, 127.0, 123.5, 21.6; HRMS calc. for $\text{C}_{19}\text{H}_{16}\text{O}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 331.0769; found, 331.0773.

(*E*)-2-(2-Tosylvinyl)pyridine (**3ia**). Compound **3ia** was obtained in 82% yield (42.4 mg) according to the general procedure. Yellow solid; mp: 91.8–93.4 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 8.63 (d, J = 4.0 Hz, 1H), 7.86 (d, J = 8.4 Hz, 2H), 7.77–7.73 (m, 1H), 7.64 (d, J = 14.9 Hz, 1H), 7.46 (d, J = 14.9 Hz, 1H), 7.42 (d, J = 7.8 Hz, 1H), 7.36 (d, J = 8.0 Hz, 2H), 7.32–7.29 (m, 1H), 2.45 (m, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 151.1, 150.3, 144.6, 140.0, 137.3, 137.0, 132.2, 130.0, 128.0, 125.4, 124.9, 21.6; HRMS calc. for $\text{C}_{14}\text{H}_{13}\text{NO}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 282.0565; found, 282.0567.

(*E*)-1-Methyl-4-(1-phenylprop-1-en-2-ylsulfonyl)benzene (**3ja**). Compound **3ja** was obtained in 44% yield (23.9 mg) according to the general procedure. Yellow solid; mp: 122.16–123.1 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.83 (d, J = 8.2 Hz, 3H), 7.45–7.36 (m, 7H), 2.46 (m, 3H), 2.13 (d, J = 1.4 Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 144.3, 137.6, 137.0, 136.1, 133.9, 129.8, 129.6, 129.2, 128.7, 128.3, 21.6, 13.2; HRMS calc. for $\text{C}_{16}\text{H}_{16}\text{O}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 295.0769; found, 295.0771.

3-Tosyl-1,2-dihydronaphthalene (**3ka**). Compound **3ka** was obtained in 65% yield (36.9 mg) according to the general procedure. Yellow solid; mp: 115.8–116.7 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.80 (d, J = 8.3 Hz, 2H), 7.56 (s, 1H), 7.32 (d, J = 8.1 Hz, 2H), 7.28–7.26 (m, 1H), 7.25–7.21 (m, 2H), 7.12 (d, J = 7.0 Hz, 1H), 2.86 (t, J = 8.1 Hz, 2H), 2.51–2.47 (t, J = 8.1 Hz, 2H), 2.42 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 144.3, 138.5, 136.6, 135.6, 134.8, 131.1, 130.4, 129.9, 129.0, 128.0, 127.8, 127.2, 27.6, 21.7, 21.6; HRMS calc. for $\text{C}_{17}\text{H}_{16}\text{O}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 307.0769; found, 307.0773.

(*E*)-1-(Hex-1-enylsulfonyl)-4-methylbenzene (**3la**). Compound **3la** was obtained in 23% yield (11.0 mg) according to the general procedure. Yellow oil; ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.75 (d, J = 8.3 Hz, 2H), 7.33 (d, J = 8.1 Hz, 2H), 6.99–6.93 (m, 1H), 6.31–6.28 (dt, J_1 = 1.4 Hz, J_2 = 15.1 Hz, 1H), 2.43 (s, 3H), 2.25–2.20 (m, 2H), 1.47–1.41 (m, 2H), 1.36–1.29 (m, 2H), 0.89 (t, J = 7.3 Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 146.7, 144.2, 137.8, 130.6, 129.9, 127.6, 31.2, 29.7, 22.1, 21.6, 13.7; HRMS calc. for $\text{C}_{13}\text{H}_{18}\text{O}_2\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 261.0925; found, 261.0929.

(*E*)-Methyl 3-tosyl acrylate (**3ma**). Compound **3ma** was obtained in 40% yield (19.2 mg) according to the general procedure. Yellow solid; mp: 106.1–107.4 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.80 (d, J = 8.3 Hz, 2H), 7.38 (d, J = 8.2 Hz, 2H), 7.33 (d, J = 15.2 Hz,

1H), 6.80 (d, J = 15.2 Hz, 1H), 3.80 (s, 3H), 2.46 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 164.0, 145.7, 143.7, 135.4, 130.3, 130.0, 128.4, 52.8, 21.7; HRMS calc. for $\text{C}_{11}\text{H}_{12}\text{O}_4\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 263.0354; found, 263.0357.

(*E*)-Methyl 3-tosylbut-2-enoate (**3na**). Compound **3na** was obtained in 34% yield (17.2 mg) according to the general procedure. Yellow solid; mp: 55.2–56.6 °C. ^1H NMR (CDCl_3 , 500 MHz, ppm): δ 7.82 (d, J = 8.3 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 7.23–7.22 (q, J = 1. Five Hz, 1H), 3.78 (s, 3H), 2.46 (s, 3H), 2.33 (d, J = 1. Five Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 125 MHz, ppm): δ 166.2, 145.2, 140.6, 137.7, 137.6, 130.1, 127.7, 53.0, 21.1, 13.3; HRMS calc. for $\text{C}_{12}\text{H}_{14}\text{O}_4\text{SNa}$ ($\text{M}+\text{Na}$) $^+$, 277.0510; found, 277.0513.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.joc.7b00994.

^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR spectra for all compounds (PDF)

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Notes

The authors declare no competing financial interest.

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