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# Synthesis of Sulfones and Sulfonamides via Sulfinate Anions: Revisiting the Utility of Thiosulfonates

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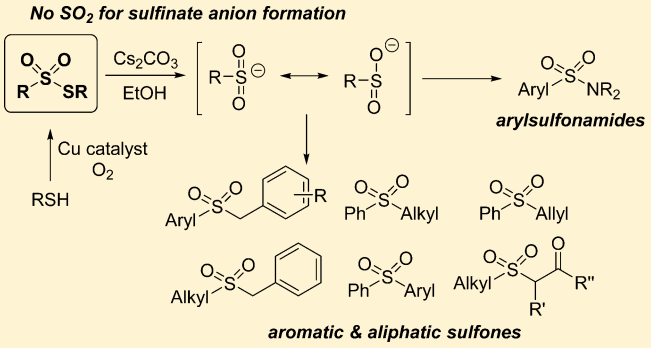
Supporting Information

**ABSTRACT:** Simple and high-yielding strategies for the production of a variety of sulfones and sulfonamides, using thiosulfonates synthesized by copper-catalyzed aerobic dimerization, are reported. Although thiosulfonates are an old class of compound, practical methods for their synthesis and utilization have not been rigorously developed. In this study, we revisit the reactions of easily accessible thiosulfonates to form sulfinate anions. Because of the similar reactivity of thiosulfonates and metal sulfinates derived from toxic $SO_2$, thiosulfinates are proposed to be stable, nontoxic alternatives to metal sulfinate salts.

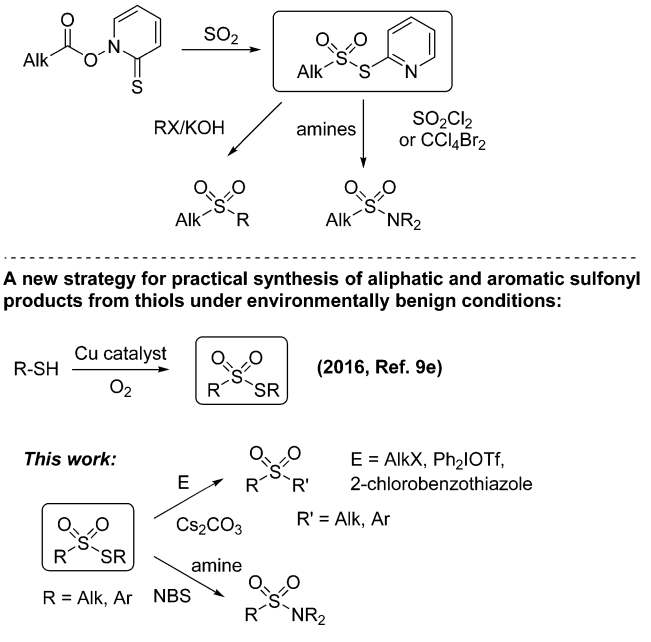



## INTRODUCTION

Sulfinates are versatile reagents that are used in transition-metal-catalyzed cross-coupling reactions to produce synthetically and pharmaceutically useful sulfones and sulfonamides.[1,2] Although sulfinates were reported in the early 1900s, their use in chemical reactions has drawn little attention due to the limited accessibility of sulfinate reagents. Until DABSO (DABCO·$(SO_2)_2$; DABCO = 1,4-diazabicyclo[2.2.2]octane) and potassium metabisulfite ($K_2S_2O_5$) were introduced in metal-catalyzed coupling reactions, the main sources of sulfinates were commercially available sodium sulfinates and $SO_2$ gas derived sulfinates.[3−5] The limited commercial availability of sodium sulfinates and the toxicity of $SO_2$ gas used for the generation of metal sulfinates[6] has resulted in the slow growth of sulfinate chemistry. Since Willis and Mascitti introduced stable, nontoxic sulfinates, generated from DABSO and $K_2S_2O_5$, a range of sulfinate derivatives and their reactions have been extensively reported.[7]

In their pioneering work, Barton and co-workers studied the preparation and synthetic utility of alkyl *S*-pyridyl thiosulfonates (Scheme 1).[8] These intermediates were synthesized from aliphatic esters of *N*-hydroxy-2-thiopyridone esters and $SO_2$ gas under anaerobic cryogenic photolytic conditions. The thiosulfonates were then treated with alkyl halides or *N*-haloamines (generated in situ from amines) to form sulfones and sulfonamides, respectively. Recently, our research group has reported one-pot, copper-catalyzed aerobic coupling reactions using thiols to prepare synthetically challenging organosulfur compounds.[9] In particular, it was found that alkyl and aryl thiols undergo copper-catalyzed oxidative dimerization to form thiosulfonates.[9e] We envisioned that the latter could be cleaved by nucleophiles to generate sulfinate anions, which would then react in one pot with various electrophiles to afford sulfones or with amines to form sulfonamides. Thus, in this study, we present a new strategy for the preparation of alkyl and aryl sulfones and sulfonamides based on the use of thiosulfonate intermediates. Compared with Barton's protocol, it avoids using toxic $SO_2$ gas, anaerobic cryogenic photolytic conditions, and the preparation of *N*-hydroxy-2-thiopyridone esters. In addition,

**Scheme 1. Synthesis of Sulfones and Sulfonamides via Thiosulfonate Intermediates**

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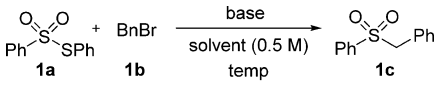
it expands the scope of the thiosulfonate-based transformations to the aromatic chemical space.[10] Taking into account their ready accessibility and nonhazardous properties, the proposed use of thiosulfonates provides a practical platform for convenient synthesis of a variety of sulfones and sulfonamides without isolation of sulfinate salts.

## RESULTS AND DISCUSSION

The reaction of phenyl thiosulfonate **1a** and benzyl bromide **1b** was investigated in the presence of organic and inorganic bases (Table 1). First, guanidine- and amidine-type bases such as

**Table 1. Optimization of the Synthesis of 1c**




| entry | base (equiv) | solvent | temp (°C) | yield (%) |
|---|---|---|---|---|
| 1 | TBD (2) | toluene | 100 | 89 |
| 2 | DBU (2) | toluene | 100 | 81 |
| 3 | TMG (2) | toluene | 100 | 70 |
| 4 | Me-TBD (2) | toluene | 100 | 73 |
| 5 | DIPEA (2) | toluene | 100 | 71 |
| 6 | DMAP (2) | toluene | 100 | 0 |
| 7 | DABCO (2) | toluene | 100 | 0 |
| 8 | $K_2CO_3$ (2) | toluene | 100 | 5 |
| 9 | $Cs_2CO_3$ (2) | toluene | 100 | 97 |
| 10 | TBD (2) | $CH_3CN$ | 80 | 91 |
| 11 | TBD (2) | THF | 65 | 99 |
| 12 | TBD (2) | EtOH | 90 | 87 |
| 13 | $Cs_2CO_3$ (2) | EtOH | 90 | 99 |
| 14 | $Cs_2CO_3$ (2) | THF | 65 | 91 |
| 15 | $Cs_2CO_3$ (1) | EtOH | 90 | 99 (61)[a] |
| 16 | TBD (1) | THF | 65 | 81 |
| 17 | $Cs_2CO_3$ (1) | EtOH | rt | 88 |

[a]0.5 equiv of $Cs_2CO_3$.

1,5,7-triazabicyclo[4.4.0]dec-1-ene (TBD), 1,8-diazabicyclo-[5.4.0]undec-7-ene (DBU), 1,1,3,3-tetramethylguanidine (TMG), and 7-methyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene (Me-TBD) were examined. As listed in Table 1, the reaction in the presence of TBD afforded **1c** in the highest yield (entries 1−4). Diisopropylethylamine (DIPEA), *N*,*N*-dimethylamino-pyridine (DMAP), and 1,4-diazabicyclo[2.2.2]octane (DABCO) were also used in the reactions, affording **1c** in good yields (entries 5−7). The inorganic bases $K_2CO_3$ and $Cs_2CO_3$ exerted very different effects, forming **1c** in 5% and 97% yields, respectively (entries 8 and 9). We next turned our attention to solvent effects. Using TBD, **1c** was formed in yields of 91% in $CH_3CN$, 99% in THF, and 87% in EtOH (entries 10−12). Using $Cs_2CO_3$, yields of 99% (EtOH) and 91% (THF) were obtained for **1c** (entries 13 and 14). As almost complete conversion was observed with 2 equiv of bases, lower amounts of base (TBD and $Cs_2CO_3$) were also tested. When 1 equiv of $Cs_2CO_3$ was used, the excellent yield of **1c** was retained, but 1 equiv of TBD and 0.5 equiv of $Cs_2CO_3$ gave somewhat reduced yields (entries 15 and 16). The reaction was run at room temperature, showing a slightly decreased yield (88%, entry 17).

Based on the results listed in Table 1, we next turned our attention to explore the substrate scope under two sets of optimum reaction conditions, namely $Cs_2CO_3$ in EtOH and TBD in THF. A range of electrophiles including benzyl bromide derivatives, alkyl bromides, allyl bromide, α-bromo ketones and esters, 2-chlorobenzothioazole, and diphenyliodonium salts, were examined for aryl and alkyl sulfone formation, as shown in Figure 1.

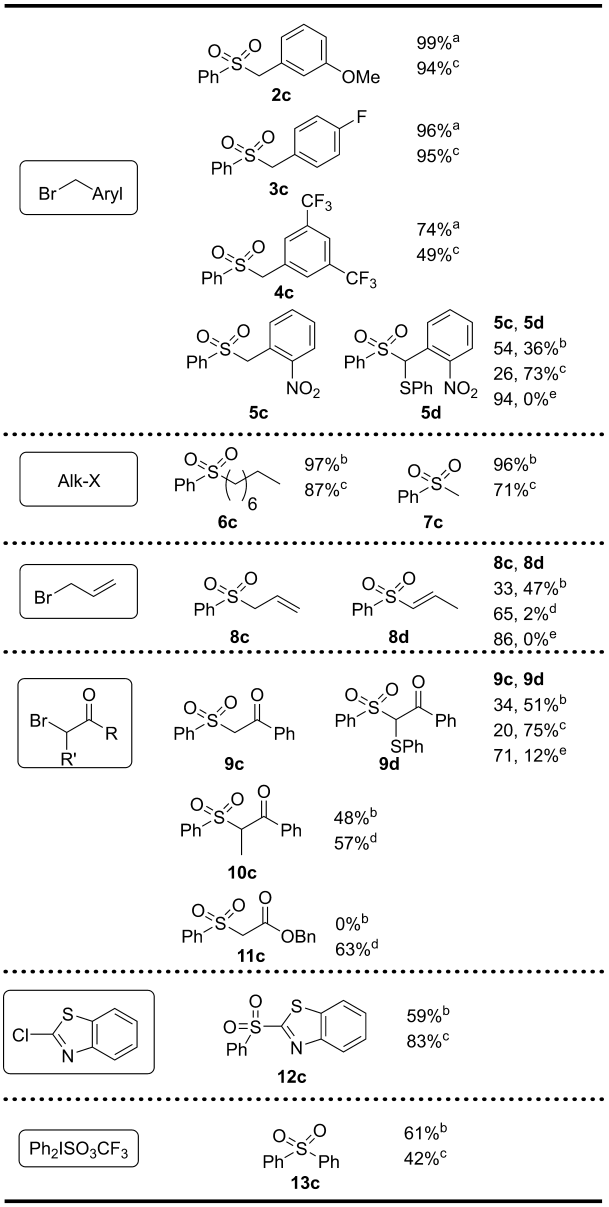



**Figure 1.** Sulfone substrate scope.

3-Methoxy- and 4-fluorobenzyl bromides reacted with thiosulfonate **1a** to afford sulfones **2c** and **3c**, respectively, in excellent yields under $Cs_2CO_3$- and TBD-mediated reaction conditions. Meanwhile, the reactions of 3,5-bis(trifluoro-methyl)benzyl bromide gave comparatively low yields of **4c**. Interestingly, 2-nitrobenzyl bromide afforded the desired sulfone **5c** along with the side product **5d**, resulting from sulfenylation, in good yields, but in ratios that depended on the reaction conditions. Presumably, the higher acidity of **5c**, assisted by the $NO_2$ group, promoted further sulfenylation. In the presence of acetyl acetone, sulfenylation was completely



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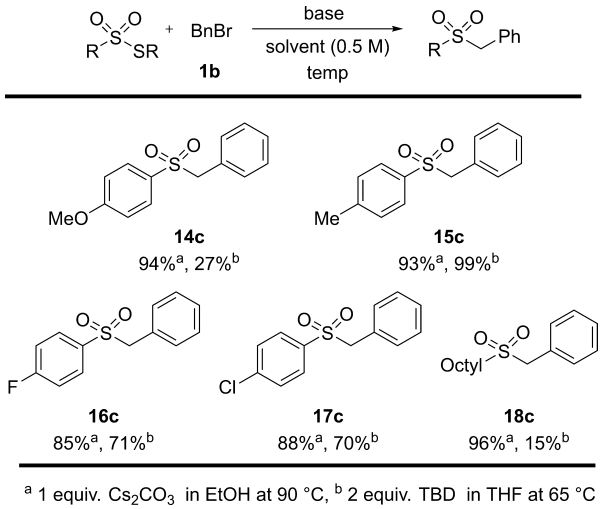
inhibited to afford **5c** as a single product in 94% yield. As alkyl derivatives, octyl bromide and methyl bromide were also tested, affording the desired products **6c** and **7c**, respectively, in excellent yields. When allyl bromide was employed in this transformation, allyl-substituted sulfone **8c** was formed, along with vinyl sulfone **8d**, when $Cs_2CO_3$ was used as the base. Presumably, the acidic nature of the $\alpha$-protons in the allylsulfones induced the formation of the vinyl sulfone.[11] Interestingly, once again, the addition of acetylacetone promoted the formation of **8c** only. $\beta$-Keto sulfones were prepared from thiosulfonates using $\alpha$-bromo ketones and esters.[12] Similar to the reactions of 2-nitrobenzyl bromides, $\beta$-keto sulfone **9c** and $\alpha$-sulfenylated **9d** were isolated. The formation of **9d** is the result of the displacement of the sulfinate moiety at the divalent sulfur atom of the thiosulfonate with a carbon nucleophile of the desired product. Depending on the reaction conditions, **9c** or **9d** could be formed as the major product. $\alpha$-Methylated $\beta$-keto sulfone **10c** and $\beta$-ester **11c** did not undergo further sulfenylation. Heterocyclic and aromatic sulfones were synthesized using 2-chlorobenzothiazole and diphenyliodonium triflate, respectively. The reaction of thiosulfonate **1a** with 2-chlorobenzothiazole afforded the desired heterocyclic sulfone **12c** in good yields. The phenyl cation, generated from diphenyliodonium triflate, also participated in these reactions to give the desired diphenyl sulfone **13c** in good yields. The reaction of bromobenzene as an example of aromatic halides was tested, resulting in no product formation.

Subsequent to electrophile screening, the nucleophilic reactivities of these diverse thiosulfonates were assessed (Figure 2). Various thiosulfonates were subjected to the base-mediated



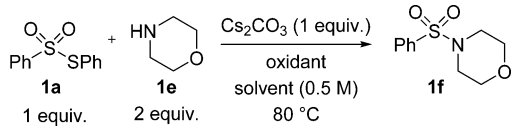

[a] 1 equiv. $Cs_2CO_3$ in EtOH at 90 °C, [b] 2 equiv. TBD in THF at 65 °C

**Figure 2.** Examples of sulfones synthesized from various thiosulfonates and **1b**.

reaction with benzyl bromide **1b**. The methoxy-substituted thiosulfonate was converted into **14c** in 94% yield using $Cs_2CO_3$, but the TBD-mediated reaction was not as effective. A methyl-substituted thiosulfonate participated in the reaction with **1b** to afford **15c** in excellent yields for each base. Fluoro- and chloro-substituted thiosulfonates reacted to form sulfone **16c** and **17c**, respectively, in good yields, but slightly lower than those of the methyl-substituted derivative. As an example of an aliphatic system, octyl thiosulfonate was reacted in the presence of $Cs_2CO_3$ and TBD to afford **18c** in 96% and 15% yields, respectively.

Next, the reactions of thiosulfonate **1a** and morpholine **1e** were attempted in the presence of $Cs_2CO_3$ (1 equiv) and *N*-bromosuccinimide (NBS) (Table 2). As shown during the

**Table 2. Optimization of the Synthesis of 1f**




| entry | oxidant (equiv) | solvent | yield (%) |
|---|---|---|---|
| 1 | NBS (2) | DMSO | 40 |
| 2 | NBS (2) | DMF | 74 |
| 3 | NBS (2) | EtOH | 96 |
| 4 | NBS (2) | $CH_3CN$ | 75 |
| 5 | NBS (2) | THF | 61 |
| 6 | NBS (2) | DCE | 57 |
| 7 | NBS (2) | toluene | 46 |
| 8 | $I_2$ (2) | EtOH | 92 |
| 9 | NCS (2) | EtOH | 66 |
| 10 | NBS (1) | EtOH | 75 |
| 11 | NBS (2) | EtOH | 94[a] (74)[b] |
| 12 | NBS (1.5) | EtOH | 98[a] |
| 13 | NBS (2) | EtOH | 84[c] |

[a]1.5 equiv of **1e**. [b]1.1 equiv of **1e** [c]The reaction was run at room temperature.

formation of the sulfones (above), $Cs_2CO_3$ (1 equiv) provided generally good yields under most reaction conditions. Hence, we used $Cs_2CO_3$ throughout the optimization of the sulfonamide synthesis. To induce halogenation, NBS (2 equiv) was added in various solvents (DMSO, DMF, EtOH, $CH_3CN$, THF, DCE, and toluene; entries 1−7). In EtOH, **1f** was obtained in the highest yield (96%, entry 3). When $I_2$ was used instead of NBS, a comparable yield was obtained (92%, entry 8). The reaction using *N*-chlorosuccinimide (NCS) also gave the desired product **1f** in 66% yield (entry 9). When the amounts of NBS and **1e** were reduced to 1 and 1.1 equiv, respectively, the lower yields of **1f** were observed, but in the presence of 1.5 equiv of NBS and **1e**, the high yield of **1f** observed in entry 3 was retained (98%) (entries 10−12). To investigate the effect of temperature, the reaction was run at room temperature, and **1f** was formed at 84% yield (entry 13).

Using the optimized reaction conditions listed in Table 2, various amines and thiosulfonates were reacted to form sulfonamides (Figure 3). Cyclic and acyclic *sec*-amines participated in the reaction to afford the desired sulfonamides **2f**, **3f**, and **4f** in good yields. The reactions of benzylamine and butyl amine with **1a** provided the desired products **5f** and **6f** in slightly lower yields than was observed for the *sec*-amines. In the case of aromatic amines, aniline and 2-aminopyridine were examined under the reaction conditions. Both compounds underwent bromination on the aromatic ring, resulting in no S−N bond formation. Various thiosulfonates were also subjected to the indicated oxidative amination with morpholine to afford aromatic sulfonamides **7f−10f** in good to excellent yields.

Plausible mechanisms for these transformations are shown in Scheme 2. In the presence of $Cs_2CO_3$ in EtOH or TBD in THF, nucleophilic bases attack the divalent sulfur atom to release the sulfinate anion, which is trapped by a carbon electrophile to afford the corresponding sulfone.[7] In the case of sulfonamides, the amines undergo halogenation by NBS or $I_2$, prior to addition



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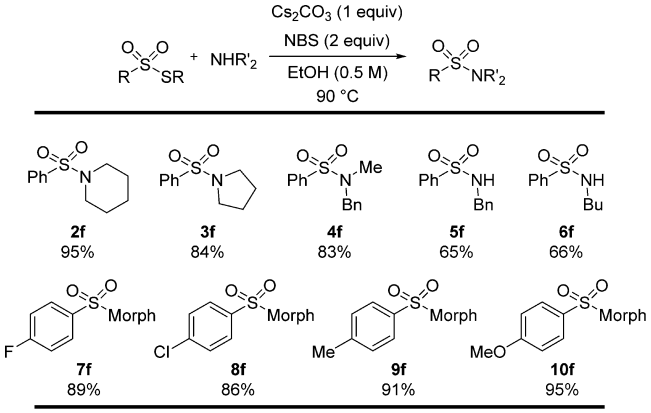
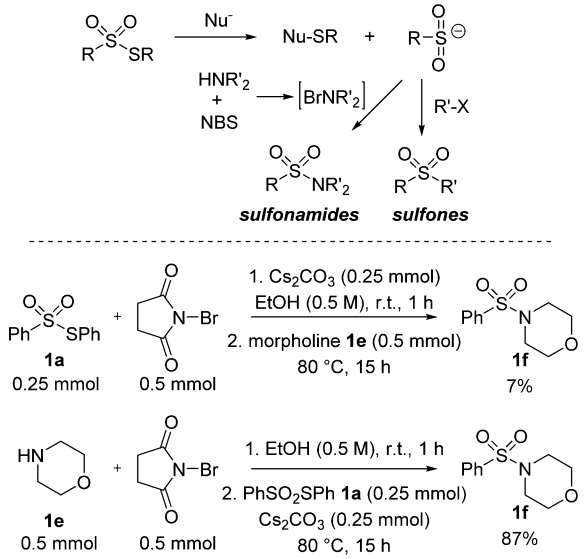



**Figure 3.** Substrate scope in the formation of sulfonamides.

**Scheme 2. Mechanisms for Sulfone and Sulfonamide Formation via Sulfinate Anions and Control Experiments**




by the sufinate anion, which is supported by control experiments (Scheme 2).[13] When a solution containing a thiosulfonate and NBS was stirred prior to the addition of morpholine, the desired product **1f** was obtained in 7% yield; however, when the reaction of morpholine and NBS was conducted prior to the addition of the thiosulfonate, **1f** was isolated in 87% yield. Because halogenation of amines occurs prior to the reaction with sulfinate anions, sulfenamides formed by the addition of amines to divalent sulfur of thiosulfonates were not observed.[14]

## CONCLUSION

We have demonstrated that thiosulfonates participate in reactions with electrophiles and amines to afford a range of sulfones and sulfonamides in good to excellent yields. In the presence of nucleophilic bases, sulfinate anions are expelled during the reaction, which react then with benzyl bromides, alkyl bromides, allyl bromide, phenacyl bromide, and electron-deficient aromatic compounds to afford sulfones, while sulfonamides are formed in the presence of amines and NBS. Compared to metal sulfinates derived from the reaction of organometallic compounds and $SO_2$, thiosulfonates are easily synthesized by catalytic aerobic dimerization and show good reactivity with various electrophiles and amines. Thiosulfonates are stable, nontoxic alternatives to metal sulfinates.

## EXPERIMENTAL SECTION

**General Procedure for the Synthesis of Thiosulfonates.**[9e] Thiophenol (159 μL, 1.5 mmol) and benzyl alcohol (52 μL, 0.50 mmol) were added to a mixture of CuI (4.8 mg, 0.025 mmol) and TBD (7.1 mg, 0.05 mmol) in THF (1 mL). The resulting solution was stirred at 65 °C for 18 h under 1 atm of oxygen. The reaction mixture was cooled, and the solvent was removed. The residue was purified by column chromatography on a silica gel eluting with hexane and ethyl acetate to afford the desired product.

**General Procedure for the Synthesis of Sulfones.** *Method A.* Benzyl bromide (60 μL, 0.50 mmol) was added to a mixture of *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) and $Cs_2CO_3$ in a sealed tube. After 0.5 mL of EtOH was added to the reaction, the vessel was closed and the mixture stirred at 90 °C for 16 h. The reaction mixture was cooled, diluted with DCM, and filtered. The filtrate was taken and concentrated through a rotary evaporator. The residue was purified by column chromatography on a silica gel column eluting with 3% ethyl acetate in hexane to afford the desired product. [Depending on the nature of the carbon electrophiles and amines, the amount of $Cs_2CO_3$ used was either 1 or 2 equiv (with respect to the thiosulfonate) to obtain the optimum yield.]

*Method B.* Benzyl bromide (60 μL, 0.50 mmol) was added to a mixture of *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) and TBD in a sealed tube. After addition of 0.5 mL of THF, the reaction vessel was closed and stirred at 65 °C for 16 h. The reaction was cooled and concentrated through a rotary evaporator. The residue was purified by column chromatography on a silica gel column eluting with 3% ethyl acetate in hexane to afford the desired product. [Depending on the nature of the carbon electrophiles and amines, the amount of TBD used was either 1 or 2 equiv (with respect to the thiosulfonate) to obtain the optimum yield.]

*(Benzylsulfonyl)benzene (**1c**).*[15] The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with benzyl bromide (60 μL, 0.50 mmol) to form **1c** as a white solid. Mp: 148−150 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.63−7.56 (3H, m), 7.45−7.41 (2H, m), 7.33−7.22 (3H, m), 7.08 (2H, d, $J$ = 8.0 Hz), 4.30 (2H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz) $\delta$: 137.7, 133.6, 130.7, 128.8, 128.7, 128.6, 128.5, 128.0, 63.0. HRMS (EI, $m/z$): calcd for $C_{13}H_{12}O_2S$ $[M]^+$ 232.0558, found 232.0555. FTIR (neat): 3030, 2963, 1302, 1152, 1126 cm$^{-1}$.

*1-Methoxy-3-((phenylsulfonyl)methyl)benzene (**2c**).*[16] The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with 3-methoxybenzyl bromide (71 μL, 0.50 mmol) to form **2c** as a white solid. Mp: 112−114 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.66−7.58 (3H, m), 7.47−7.44 (2H, m), 7.15 (1H, t, $J$ = 8.0 Hz), 6.86−6.83 (1H, m), 6.65−6.60 (2H, m), 4.28 (2H, s), 3.71(3H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 159.4, 137.8, 133.6, 129.5, 129.3, 128.8, 128.6, 123.1, 115.9, 114.7, 62.9, 55.3 ppm. HRMS (FAB, $m/z$): calcd for $C_{14}H_{15}O_3S$ $[M + H]^+$ 263.0742, found 263.0741. FTIR (neat): 3061, 2977, 1299, 1132 cm$^{-1}$.

*1-Fluoro-4-((phenylsulfonyl)methyl)benzene (**3c**).*[17] The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with 4-fluorobenzyl bromide (63 μL, 0.50 mmol) to form **3c** as a white solid. Mp: 167−168 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.65−7.59 (3H, m), 7.49−7.45 (2H, m), 7.08−7.04 (2H, m), 6.97−6.93 (2H, m), 4.28 (2H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 164.2 (d, $J$ = 246 Hz), 137.7, 133.8, 132.6 (d, $J$ = 9.1 Hz), 129.0, 128.6, 124.0, 115.8 (d, $J$ = 22 Hz), 62.1. HRMS (FAB, $m/z$): calcd for $C_{13}H_{12}FO_2S$ $[M + H]^+$ 251.0542, found 251.0540. FTIR (neat): 3061, 2945, 1303, 1150 cm$^{-1}$.

*1-((Phenylsulfonyl)methyl)-3,5-bis(trifluoromethyl)benzene (**4c**).* The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with 3,5-bis-(trifluoromethyl)benzyl bromide (96 μL, 0.50 mmol) to form **4c** as a white solid. Mp: 147−149 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.84 (1H, s), 7.69−7.64 (3H, m), 7.53−7.49 (4H, m), 4.41 (2H, s). $^{13}C\{^1H\}$-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NMR ($CDCl_3$, 100 MHz): 137.0, 134.4, 132.0 (q, J = 33.4 Hz), 130.9 (2C), 129.3, 128.5, 122.8 (q, J = 271 Hz), 122.7 (m), 62.1. HRMS (FAB, *m/z*): calcd for $C_{15}H_{11}F_6O_2S$ $[M + H]^+$ 369.0384, found 369.0385. FTIR (neat,): 3061, 2932, 1330, 1128 $cm^{-1}$.

*1-Nitro-2-((phenylsulfonyl)methyl)benzene (**5c**) and ((2-Nitrophenyl)(phenylsulfonyl)methyl)(phenyl)sulfane (**5d**).* The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with 2-nitrobenzyl bromide (110 mg, 0.50 mmol) to afford the easily separable mixture of **5c** and **5d**. **1-Nitro-2-((phenylsulfonyl)methyl)benzene (5c).**[18] Mp (light yellow solid): 114−116 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.97 (1H, dd, *J* = 8.0, 1.2 Hz), 7.69−7.66 (2H, m), 7.64−7.59 (2H, m), 7.55−7.44 (4H, m), 4.94 (2H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz) *δ*: 149.2, 137.8, 134.2, 134.1, 133.3, 130.1, 129.2, 128.3, 125.4, 122.9, 58.7. HRMS (FAB, *m/z*): calcd for $C_{13}H_{12}NO_4S$ $[M + H]^+$ 278.0487, found 278.0485. FTIR (neat): 3068, 2924, 1529, 1352, 1320, 1147 $cm^{-1}$. **((2-Nitrophenyl)(phenylsulfonyl)methyl)(phenyl)sulfane (5d).** Mp (yellow solid): 116−118 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.96 (1H, d, *J* = 8.0 Hz), 7.92 (1H, d, *J* = 8.4 Hz), 7.75−7.68 (3H, m), 7.61−7.51 (2H, m), 7.43 (2H, t, 7.2 Hz), 7.30−7.19 (5H, m), 6.65 (1H, d, *J* = 1.6 Hz). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 148.6, 136.8, 134.2, 133.4, 132.5, 132.3, 132.2, 130.1, 129.3 (2C), 128.9, 128.8, 127.0, 124.9, 70.2. HRMS (FAB, *m/z*): calcd for $C_{19}H_{16}NO_4S_2$ $[M + H]^+$ 386.0521, found 386.0518. FTIR (neat): 3065, 2919, 1528, 1348, 1322, 1150 $cm^{-1}$.

*(Octylsulfonyl)benzene (**6c**).*[19] The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with octyl bromide (88 μL, 0.50 mmol) to afford **6d** as liquid. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.92 (2H, dd, *J* = 8.0, 1.6 Hz), 7.68−7.63 (1H, m), 7.59−7.55 (2H, m), 3.10−3.06 (2H, m), 1.74−1.67 (2H, m), 1.36−1.23 (10H, m), 0.86 (3H, t, *J* = 7.2 Hz). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 139.1, 133.5, 129.2, 127.9, 56.4, 31.8, 29.1, 29.0, 28.4, 22.8, 22.7, 14.2. HRMS (FAB, *m/z*): calcd for $C_{14}H_{23}O_2S$ $[M + H]^+$ 255.1419, found 255.1418. FTIR (neat): 3065, 2927, 1306, 1147 $cm^{-1}$.

*(Methylsulfonyl)benzene (**7c**).*[15] The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with methyl iodide (32 μL, 0.50 mmol) to form **7c** as a white solid. Mp: 87−89 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.96 (2H, dd, *J* = 8.4, 0.8 Hz), 7.69−7.64 (1H, m), 7.60−7.56 (2H, m), 3.06 (3H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 140.4, 133.6, 129.3, 127.2, 44.6. HRMS (FAB, *m/z*): calcd for $C_7H_9O_2S$ $[M + H]^+$ 157.0323, found 157.0322. FTIR (neat): 3023, 2927, 1307, 1147 $cm^{-1}$.

*(Allylsulfonyl)benzene (**8c**) and (Prop-1-en-1-ylsulfonyl)benzene (**8d**).* The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with allyl bromide (45 μL, 0.50 mmol) to form the inseparable liquid mixture of **8c** and **8d**.[20] **(Allylsulfonyl)benzene (8c).** $^1H$ NMR ($CDCl_3$, 400 MHz): 7.89 (2H, dd, *J* = 8.4, 1.2 Hz), 7.68−7.63 (1H, m), 7.59−7.54 (2H, m), 5.85−5.74 (1H, m), 5.36 (1H, dd, *J* = 10.0, 4.8 Hz), 5.18−5.12 (1H, m), 3.83 (2H, dd, *J* = 7.6, 0.8 Hz). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 138.1, 133.7, 128.9, 128.3, 124.7, 124.5, 60.9. HRMS (FAB, *m/z*): calcd for $C_9H_{11}O_2S$ $[M + H]^+$ 183.0480, found 183.0478. FTIR (neat): 3066, 2920, 1308, 1146 $cm^{-1}$.

*1-Phenyl-2-(phenylsulfonyl)ethan-1-one (**9c**) + 1-Phenyl-2-(phenylsulfonyl)-2-(phenylthio)ethan-1-one (**9d**).* The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with 2-bromoacetophenone (102 mg, 0.50 mmol) to form the easily separable mixture of **9c** and **9d** as a white solid. **1-Phenyl-2-(phenylsulfonyl)ethan-1-one (9c).**[21] Mp: 95−97 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.95−7.88 (4H, m), 7.68−7.59 (2H, m), 7.56−7.52 (2H, m), 7.50−7.45 (2H, m), 4.74 (2H, s). $^{13}C\{^1H\}$-NMR ($CDCl_3$, 100 MHz): 187.8, 138.6, 135.6, 134.3, 134.1, 129.2, 129.1, 128.8, 128.4, 63.4. HRMS (FAB, *m/z*): calcd for $C_{14}H_{13}O_3S$ $[M + H]^+$ 261.0585, found 261.0583. FTIR (neat): 3064, 2947, 1680, 1323, 1154 $cm^{-1}$. **1-Phenyl-2-(phenylsulfonyl)-2-(phenylthio)ethan-1-one (9d).**[22] Mp: 135−136 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 8.02 (2H, dd, *J* = 8.4, 1.2 Hz), 7.89 (2H, d, *J* = 8.0 Hz), 7.70−7.44 (8H, m), 7.37−7.28 (3H, m), 5.79 (1H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 189.0, 136.3, 134.9, 134.4, 134.3, 133.5, 131.9, 130.6, 129.4, 129.3, 129.1, 128.8, 128.6, 75.6. HRMS (FAB, *m/z*): calcd for $C_{20}H_{17}O_3S_2$ $[M + H]^+$ 369.0619, found 369.0618. FTIR (neat): 3062, 1682, 1323, 1151 $cm^{-1}$.

*1-Phenyl-2-(phenylsulfonyl)propan-1-one (**10c**).*[23] The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with 2-bromopropiophenone (78 μL, 0.50 mmol) to form **10c** as a white solid. Mp: 78−80 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.98 (2H, dd, *J* = 7.2, 1.2 Hz), 7.80 (2H, dd, *J* = 7.2, 1.2 Hz), 7.66−7.58 (2H, m), 7.54−7.45 (4H, m), 5.19 (1H, q, *J* = 6.8 Hz), 1.58 (3H, d, *J* = 7.2 Hz). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 192.3, 136.1, 136.0, 134.2, 134.0, 129.7, 129.1, 128.9, 128.7, 65.1, 13.4. HRMS (EI, *m/z*): calcd for $C_{15}H_{14}O_3S$ $[M]^+$ 274.0664, found 274.0667. FTIR (neat): 3064, 2941, 1682, 1309, 1149 $cm^{-1}$.

*Benzyl 2-(Phenylsulfonyl)acetate (**11c**).* The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with benzyl bromoacetate (81 μL, 0.50 mmol) to afford **11c** as liquid. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.86 (2H, d, *J* = 8.0 Hz), 7.64 (1H, t, *J* = 7.6 Hz), 7.51−7.47 (2H, m), 7.36−7.34 (3H, m), 7.27−7.25 (2H, m), 5.11 (2H, s), 4.15 (2H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz) *δ*: 162.1, 138.5, 134.4, 134.2, 129.2, 128.7, 128.6, 128.5, 128.4, 68.1, 61.1. HRMS (EI, *m/z*): calcd for $C_{15}H_{14}O_4S$ $[M]^+$ 290.0613, found 290.0610. FTIR (neat): 3065, 2943, 1739, 1327, 1151 $cm^{-1}$.

*2-(Phenylsulfonyl)benzo[d]thiazole (**12c**).* The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with 2-chlorobenzothiazole (67 μL, 0.50 mmol) to form **12c** as a white solid. Mp: 159−161 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 8.17−8.13 (3H, m), 7.96 (1H, dd, *J* = 7.6, 0.8 Hz), 7.68−7.64 (1H, m), 7.59−7.50 (4H, m). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 167.1, 152.8, 138.4, 137.0, 134.5, 129.5, 128.9, 127.9, 127.5, 125.5, 122.2. HRMS (FAB, *m/z*): calcd for $C_{13}H_{10}NO_2S_2$ $[M + H]^+$ 276.0153, found 276.0151. FTIR (neat): 3092, 3061, 1329, 1316, 1159 $cm^{-1}$.

*Sulfonyldibenzene (**13c**).*[24] The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with diphenyliodonium trifluoromethanesulfonate (220 mg, 0.50 mmol) to form **13c** as a white solid. Mp: 123−125 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.96−7.93 (4H, m), 7.58−7.48 (6H, m). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 141.5, 133.2, 129.3, 127.6. HRMS (FAB, *m/z*): calcd for $C_{12}H_{11}O_2S$ $[M + H]^+$ 219.0480, found 219.0479. FTIR (neat, $cm^{-1}$): 3065, 1309, 1154 $cm^{-1}$.

*1-(Benzylsulfonyl)-4-methoxybenzene (**14c**).*[25] The representative experimental procedures were applied to *S*-4-methoxyphenyl 4-methoxybenzenesulfonothiolate (77.6 mg, 0.25 mmol) with benzyl bromide (60 μL, 0.50 mmol) to form **14c** as a white solid. Mp: 101−103 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.53 (2H, dd, *J* = 6.8, 2.0 Hz), 7.31−7.24 (3H, m), 7.09 (2H, d, *J* = 6.8 Hz), 6.89 (2H, dd, *J* = 6.8, 2.0 Hz), 4.28 (2H, s), 3.85 (3H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 163.6, 130.8, 130.7, 129.3, 128.6, 128.5, 128.4, 114.0, 63.2, 55.8. HRMS (EI, *m/z*): calcd for $C_{14}H_{14}O_3S$ $[M]^+$ 262.0664, found 262.0664. FTIR (neat): 3031, 2975, 1315, 1296, 1153 $cm^{-1}$.

*1-(Benzylsulfonyl)-4-methylbenzene (**15c**).*[26] The representative experimental procedures were applied to *S*-*p*-tolyl 4-methylbenzenesulfonothioate (69.6 mg, 0.25 mmol) with benzyl bromide (60 μL, 0.50 mmol) to form **15c** as a white solid. Mp: 146−148 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.50 (2H, d, *J* = 8.4 Hz), 7.32−7.22 (5H, m), 7.09 (2H, d, *J* = 6.8 Hz), 4.28 (2H, s), 2.41 (3H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz) *δ*: 144.6, 134.9, 130.8, 129.4, 128.6, 128.5, 128.4, 128.2, 62.9, 21.8. HRMS (EI, *m/z*): calcd for $C_{14}H_{14}O_2S$ $[M]^+$ 246.0715, found 246.0714. FTIR (neat): 3031, 2974, 2924, 1303, 1155 $cm^{-1}$.

*1-(Benzylsulfonyl)-4-fluorobenzene (**16c**).*[17] The representative experimental procedures were applied to *S*-4-fluorophenyl 4-fluorobenzenesulfonothioate (71.6 mg, 0.25 mmol) with benzyl bromide (60 μL, 0.50 mmol) to form **16c** as a white solid. Mp: 155−157 °C. $^1H$ NMR ($CDCl_3$, 400 MHz): 7.63−7.59 (2H, m), 7.34−7.24 (3H, m), 7.13−7.06 (4H, m), 4.31 (2H, s). $^{13}C\{^1H\}$NMR ($CDCl_3$, 100 MHz): 166.9 (d, 54 Hz), 133.7 (d, *J* = 3.1 Hz), 131.5 (d, *J* = 9.1 Hz), 130.7, 128.8, 128.6, 127.9, 116.3 (d, *J* = 21.9 Hz), 63.1. HRMS (EI, *m/z*): calcd for $C_{13}H_{11}FO_2S$ $[M]^+$ 250.0464, found 250.0463. FTIR (neat): 3057, 2942, 1315, 1149, 1085 $cm^{-1}$.



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*1-(Benzylsulfonyl)-4-chlorobenzene (**17c**).*[25] The representative experimental procedures were applied to *S*-4-chlorophenyl 4-chlorobenzenesulfonothioate (79.8 mg, 0.25 mmol) with benzyl bromide (60 μL, 0.50 mmol) to form **17c** as a white solid. Mp: 144−146 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.54 (2H, dd, $J$ = 8.8, 2.0 Hz), 7.41 (2H, dd, $J$ = 8.8, 2.4 Hz), 7.35−7.25 (3H, m), 7.09 (2H, d, $J$ = 7.6 Hz), 4.31 (2H, s). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz) $\delta$: 140.4, 136.2, 130.8, 130.1, 129.2, 128.9, 128.7, 127.8, 63.0. HRMS (EI, $m/z$): calcd for $C_{13}H_{11}ClO_2S$ [M]$^+$ 266.0168, found 266.0172. FTIR (neat): 3065, 2943, 1314, 1151 cm$^{-1}$.

*((Octylsulfonyl)methyl)benzene (**18c**).* The representative experimental procedures were applied to *S*-octyl octane-1-sulfonothiolate (80.6 mg, 0.25 mmol) with benzyl bromide (60 μL, 0.50 mmol) to form **18c** as a white solid. Mp: 70−72 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.41−7.38 (5H, m), 4.22−4.19 (2H, m), 2.82−2.78 (2H, m), 1.81−1.75 (2H, m), 1.36−1.25 (10H, m), 0.87 (3H, t, $J$ = 5.6 Hz). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz): 130.5, 129.0, 128.9, 128.1, 59.5, 51.2, 31.9, 29.2, 29.1, 28.6, 22.8, 22.0, 14.3. HRMS (EI, $m/z$): calcd for $C_{15}H_{24}O_2S$ [M]$^+$ 268.1497, found 268.1500. FTIR (neat): 3025, 2954, 2916, 1317, 1299, 1123 cm$^{-1}$.

**General Procedure for the Synthesis of Sulfonamides.** Morpholine (44 μL, 0.50 mmol) was added to a mixture of *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol), NBS (90 mg, 0.5 mmol), and $Cs_2CO_3$ (82.2 mg, 0.25 mmol) in 0.5 mL of EtOH in a sealed tube. The tube was closed and stirred at 80 °C for 16 h. After completion of the reaction, the mixture was was cooled, diluted with DCM, and filtered. The filtrate was concentrated through a rotary evaporator and purified by column chromatography on a silica gel column eluting with 6% ethyl acetate in hexane to afford the desired product.

*4-(Phenylsulfonyl)morpholine (**1f**).*[27] The representative experimental procedure was applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with morpholine (44 μL, 0.50 mmol) to obtain **1f** as a white solid in 96% yield. Mp: 119−121 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.77−7.75 (2H, m), 7.65−7.61 (1H, m), 7.58−7.54 (2H, m), 3.74 (4H, t, $J$ = 4.8 Hz), 3.00 (4H, t, $J$ = 4.8 Hz). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz): 135.0, 133.1, 129.1, 127.8, 66.2, 46.2. HRMS (EI, $m/z$): calcd for $C_{10}H_{13}NO_3S$ [M]$^+$ 227.0616, found 227.0617. FTIR (neat): 2921, 2856, 1449, 1350, 1169 cm$^{-1}$.

*1-(Phenylsulfonyl)piperidine (**2f**).*[27] The representative experimental procedure was applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with piperidine (50 μL, 0.50 mmol) to obtain **2f** as a white solid in 95% yield. Mp: 92−94 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.77−7.74 (2H, m), 7.61−7.57 (1H, m), 7.55−7.51 (2H, m), 2.99 (4H, t, $J$ = 5.2 Hz), 1.67−1.61 (4H, m), 1.45−1.40 (2H, m). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz): 136.2, 132.5, 128.9, 127.6, 47.1, 25.4, 23.7. HRMS (EI, $m/z$): calcd for $C_{11}H_{15}NO_2S$ [M]$^+$ 225.0824, found 225.0826. FTIR (neat): 2945, 2840, 1445, 1336, 1168 cm$^{-1}$.

*1-(Phenylsulfonyl)pyrrolidine (**3f**).*[27] The representative experimental procedure was applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with pyrrolidine (42 μL, 0.50 mmol) to obtain **3f** as a white solid in 84% yield. Mp: 52−54 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.86−7.81 (2H, m), 7.62−7.58 (1H, m), 7.57−7.51 (2H, m), 3.28−3.22 (4H, m), 1.79−1.72 (4H, m). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz): 136.7, 132.5, 128.9, 127.4, 48.1, 25.4. HRMS (EI, $m/z$): calcd for $C_{10}H_{13}NO_2S$ [M]$^+$ 211.0667, found 211.0664. FTIR (neat): 2980, 2886, 1445, 1337, 1159 cm$^{-1}$.

*N-Benzyl-N-methylbenzenesulfonamide (**4f**).*[27] The representative experimental procedure was applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with N-benzylmethylamine (65 μL, 0.50 mmol) to obtain **4f** as a white solid in 83% yields. Mp: 91−93 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.85−7.83 (2H, m), 7.63−7.53 (3H, m), 7.35−7.25 (5H, m), 4.14 (2H, s), 2.60 (3H, s). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz) $\delta$: 137.2, 135.5, 132.7, 129.1, 128.6, 128.3, 127.9, 127.4, 54.3, 34.5. HRMS (EI, $m/z$): calcd for $C_{14}H_{15}NO_2S$ [M]$^+$ 261.0824, found 261.0822. FTIR (neat): 2968, 2860, 1446, 1341, 1166 cm$^{-1}$.

*N-Benzylbenzenesulfonamide (**5f**).*[28] The representative experimental procedure was applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with benzylamine (55 μL, 0.50 mmol) to obtain **5f** as a white solid in 65% yield. Mp: 86−88 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.86−7.83 (2H, m), 7.58−7.54 (1H, m), 7.50−7.45 (2H, m), 7.26−7.21 (3H, m), 7.18−7.15 (2H, m), 5.04 (1H, t, $J$ = 6.0 Hz), 4.13 (2H, d, $J$ = 6.4 Hz). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz): 139.8, 136.1, 132.6, 129.1, 128.6, 127.9, 127.8, 127.0, 47.4. HRMS (EI, $m/z$): calcd for $C_{13}H_{13}NO_2S$ [M]$^+$ 247.0667, found 247.0669. FTIR (neat): 3284, 3064, 1447, 1326, 1160 cm$^{-1}$.

*N-Butylbenzenesulfonamide (**6f**).*[28] The representative experimental procedures were applied to *S*-phenyl benzenesulfonothiolate (62.6 mg, 0.25 mmol) with butylamine (50 μL, 0.50 mmol) to obtain **6f** as a colorless liquid in 66% yield. $^1$H NMR ($CDCl_3$, 400 MHz): 7.92−7.87 (2H, m), 7.60−7.56 (1H, m), 7.54−7.49 (2H, m), 4.74 (1H, bs), 2.97−2.92 (2H, m), 1.47−1.40 (2H, m), 1.33−1.24 (2H, m), 0.84 (3H, t, $J$ = 7.2 Hz). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz): 139.8, 132.6, 129.1, 127.0, 43.1, 31.8, 19.9, 13.8. HRMS (EI, $m/z$): calcd for $C_{10}H_{15}NO_2S$ [M]$^+$, 213.0824, found 213.0821. FTIR (neat): 3284, 2960, 2873, 1447, 1325, 1161 cm$^{-1}$.

*4-((4-Fluorophenyl)sulfonyl)morpholine (**7f**).*[27] The representative experimental procedure was applied to *S*-4-fluorophenyl 4-fluorobenzenesulfonothioate (71.6 mg, 0.25 mmol) with morpholine (44 μL, 0.50 mmol) to obtain **7f** as a white solid in 89% yield. Mp: 103−105 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.80−7.76 (2H, m), 7.28−7.22 (2H, m), 3.75 (4H, t, $J$ = 4.8 Hz), 3.00 (4H, t, $J$ = 4.8 Hz). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz): 166.5 (d, $J$ = 253 Hz), 131.2 (d, $J$ = 3.8 Hz), 130.5 (d, $J$ = 9.1 Hz), 116.5 (d, $J$ = 22 Hz), 66.1, 46.1. HRMS (EI, $m/z$): calcd for $C_{10}H_{12}FNO_3S$ [M]$^+$ 245.0522, found 245.0523. FTIR (neat): 2962, 2858, 1347, 1158 cm$^{-1}$.

*4-((4-Chlorophenyl)sulfonyl)morpholine (**8f**).*[27] The representative experimental procedure was applied to *S*-4-chlorophenyl 4-chlorobenzenesulfonothioate (79.8 mg, 0.25 mmol) with morpholine (44 μL, 0.50 mmol) to obtain **8f** as a white solid in 86% yield. Mp: 149−151 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.71−7.69 (2H, m), 7.55−7.53 (2H, m), 3.74 (4H, t, $J$ = 4.8 Hz), 3.00 (4H, t, $J$ = 4.8 Hz). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz): 139.6, 133.5, 129.4, 129.2, 66.1, 46.1. HRMS (EI, $m/z$): calcd for $C_{10}H_{12}ClNO_3S$ [M]$^+$ 261.0226, found 261.0229. FTIR (neat): 2978, 2866, 1453, 1350, 1163 cm$^{-1}$.

*4-Tosylmorpholine (**9f**).*[27] The representative experimental procedure was applied to *S*-*p*-tolyl 4-methylbenzenesulfonothioate (69.6 mg, 0.25 mmol) with morpholine (44 μL, 0.50 mmol) to obtain **9f** as a white solid in 91% yield. Mp: 149−151 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.64 (2H, d, $J$ = 8.0 Hz), 7.35 (2H, d, $J$ = 8.0 Hz), 3.73 (4H, t, $J$ = 4.8 Hz), 2.98 (4H, t, $J$ = 4.8 Hz), 2.44 (3H, s). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz) $\delta$: 143.8, 131.8, 129.7, 127.8, 66.1, 46.1, 21.7. HRMS (EI, $m/z$): calcd for $C_{11}H_{15}NO_3S$ [M]$^+$ 241.0773, found 241.0775. FTIR (neat): 2972, 2851, 1452, 1346, 1165 cm$^{-1}$.

*4-((4-Methoxyphenyl)sulfonyl)morpholine (**10f**).*[27] The representative experimental procedure was applied to S-4-methoxyphenyl 4-methoxybenzenesulfonothiolate (77.6 mg, 0.25 mmol) with morpholine (44 μL, 0.50 mmol) to obtain **10f** as a white solid in 95% yield. Mp: 112−113 °C. $^1$H NMR ($CDCl_3$, 400 MHz): 7.70 (2H, d, $J$ = 9.2 Hz), 7.02 (2H, d, $J$ = 8.8 Hz), 3.88 (3H, s), 3.73 (4H, t, $J$ = 4.8 Hz), 2.97 (4H, t, $J$ = 4.8 Hz). $^{13}$C{$^1$H}NMR ($CDCl_3$, 100 MHz): 163.0, 129.9, 126.3, 114.2, 66.1, 55.8, 46.1. HRMS (EI, $m/z$): calcd for $C_{11}H_{15}NO_4S$ [M]$^+$ 257.0722, found 257.0719. FTIR (neat): 2974, 2852, 1454, 1347, 1162 cm$^{-1}$.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.joc.6b03016.

Spectroscopic data for products (PDF)

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### Notes

The authors declare no competing financial interest.

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