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Forging C–S(Se) Bonds by Nickel-catalyzed Decarbonylation of Carboxylic Acid and Cleavage of Aryl Dichalcogenides

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A nickel-catalyzed decarbonylation of carboxylic acids cross-coupling protocol has been developed for the straightforward C–S(Se) bond formation. This reaction is promoted by a commercially-available, user-friendly, inexpensive, air and moisture-stable nickel precatalyst. Various carboxylic acids and a wide range of aryl dichalcogenide substrates were tolerated in this process which afforded products in good to excellent yields. In addition, the present reaction can be conducted on gram scale in good yield.

Introduction

Aryl chalcogen-containing compounds are of much importance and continued interest because of their essential applications in biologically active molecules,[1] pharmaceuticals[2] and organic materials[3] (Figure 1). Examples include Vortioxetine (an antidepressant drug), Chloropromazine (a dopamine antagonist),[2a,b] Thymitaq (an anticancer)[2d] and organic electronic material triselenasumanene.[3b] Therefore, methods for effectively constructing C–S(Se) bonds continue to attract attention. The common synthetic methods for the synthesis of aryl sulfides use transition metal-catalyzed cross-coupling of aryl halides or pseudohalides with thiols.[4] However, dimerization of thiols or selenols is a major obstacle for C–S(Se) bond formation.[5]

In the last few decades, chemists are more and more interested in nickel catalysis. This is because it has the properties of easier oxidative addition,[6a] abundance and economic advantages.[6b] In particular, the nickel-catalyzed decarbonylation reaction has been extensively explored by various research groups.[7,8] For instance (Scheme 1), in 2018, Szostak's research group reported the use of air and moisture-stable nickel precatalysts to convert thioesters into thioethers by decarbonylation.[9] In the same year, Rueping's research group disclosed a new and selective cross-coupling reaction of esters and amides with mercaptans as coupling partners.[10] In 2019, our laboratory reported that under the catalysis of a stable nickel precatalyst, anhydride and thiophenol established C–S bonds through decarbonylation or decarbonylation followed by decarboxylation.[11] Most recently, Sanford et al. described the development of a nickel-catalyzed decarbonylative reaction for the synthesis of fluoroalkyl thioethers from the corresponding thioesters.[12] Many additional reports about nickel-catalyzed cross-electrophilic coupling reactions have been reported.[13] In order for metal catalysis to be widely used in industrial research, it is still urgent to find robust, bench-stable Ni(II) precatalysts.

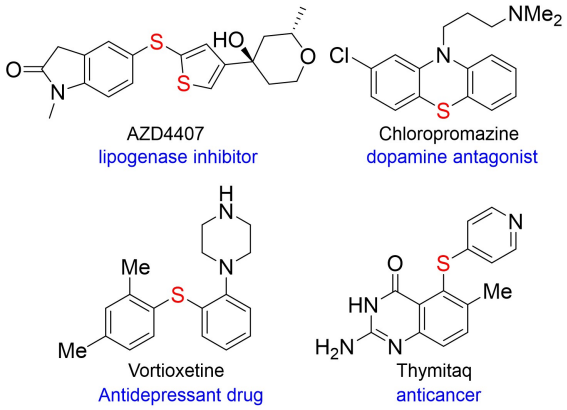

Figure 1. Examples of pharmaceutically-relevant thioethers

Carboxylic acids are one of the most attractive chemical components in organic synthesis and one of the most common organic molecules in nature.[14] The development of thioethers through cross-coupling of readily accessible carboxylic acids would greatly expand the range of electrophiles, given that: a) carboxylic acids are inexpensive and readily available; b) carboxylic acids are derived from a different pool of precursors than aryl halides and pseudohalides; and c) carboxylic acids can be used directly as starting materials for the reaction, or they can be converted into carboxylic acid derivatives under mild reaction conditions to participate in the reaction.[15]

Herein, we report the nickel-catalyzed decarbonylation cross-coupling reaction of carboxylic acids and aryl dichalcogenides (Scheme 1c).

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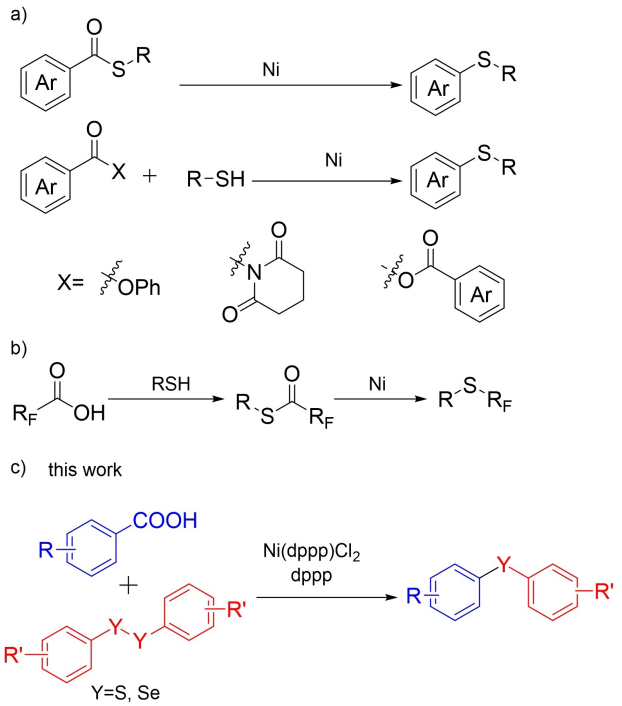


Scheme 1. Metal-catalyzed decarbonylation coupling reaction

Results and Discussion

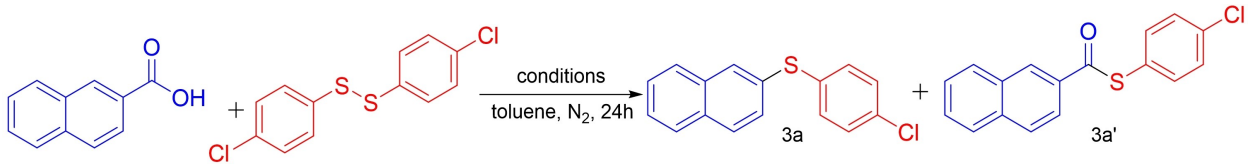
At the outset, 2-naphthoic acid (1 equiv.) and 1,2-bis(4-chlorophenyl)disulfane (1.2 equiv.) were chosen as coupling partners in the presence of a nickel/ligand catalytic system with the use of additives (Table 1). We first screened several catalysts using toluene as the solvent. With $NiBr_2$(DME) (10 mol%) as the catalyst, different phosphorus ligands were screened (entry 1–3). Although there is no target thioether formed, part of the thioester is formed under the action of the bidentate phosphine ligand. Switching bases to $NaHCO_3$ or K_2CO_3 resulted in no decarbonylation products (entry 4–5). To our delight, changing the metal species to Zn provided 15% yield of **3a** (entry 6), but replacing Zn with Mg led to no desired product (entry 7). We next turned our attention to nickel precatalysts and found that the more stable and less expensive nickel precatalysts Ni(dppp)Cl_2 can provide the thioether product with a yield of 25%. Other air-stable nickel sources such as $NiCl_2$, Ni(OAc)·$4H_2O$ and $NiBr_2$, resulted in reduced yield compared to the Ni(dppp)Cl_2 (entry 8–11). When adding pivalic anhydride in the presence of a base, the target compound is not obtained (entry 12). Further optimizations were performed by screening various anhydrides. The results showed that the use of Piv_2O increased the yield to 55% (entry 13–14). When the amount of Piv_2O was increased to 1.5 equiv., the yield of the target product was increased to 62% (entry 15). Encouraged by the results of this reaction conditions, and inspired by the fact that only thioesters were generated by the addition of Mn, we tried to add Zn and Mn at the same time. Interestingly, by adding Zn and Mn at the same time, the yield of the desired product was increased to 71% (entry 16). Therefore, we screened the ratio of Zn and Mn and found that when Mn:Zn = 2:1.5, the yield increased slightly to 75% (entry 17–20). Notably, increasing the dppp loading to 40 mol% afforded the product **3a** in 80% yield (entry 21). When no metal was added or only Mn was added in the reaction system, there was no or little decarbonylation product, but thioester was formed (entry 22–23). When only Zn was added, **3a** was obtained with 45% yield (entry 24). In the absence of Piv_2O, only 34% yield of the expected product was formed (entry 25). This suggests that part of the carboxylic acid is obtained through the formation of thioester intermediates, and then decarbonylates to the target product. As anticipated, control experiments revealed that the transformation did not proceed without the nickel catalyst or in the absence of supporting ligand (entry 26–27).

Encouraged by the initial results, we used 2-naphthoic acid as the coupling partner to explore the flexibility of various symmetric disulfides (Table 2). The disulfides with both electron-rich or electron-poor groups can be converted into aryl sulfides smoothly. Aryl disulfides with an electron-donating group or an electron withdrawing group on the benzene ring furnished the expected coupling products **3a**–**3i** in 76–92% yields. In addition, steric hindrance doesn't have much influence on the reaction, and the target compounds with good to excellent yields can be achieved (**3e**, **3h**). Unsubstituted aryl sulfide can also provide cross-coupling product in high yield (**3j**). Very encouragingly, aliphatic disulfide compounds were also workable, providing desired cross-coupling compounds in moderate yields (**3k**, **3l**).

We subsequently turned our attention to a series of carboxylic acids to determine the scope of our method (Table 3). As shown, the scope of the reaction is very broad and tolerates the coupling of electron-neutral, electron-withdrawing and electron-rich substrates. phenyl, naphthyl, halogen, hydrocarbyl, methoxy, cyano were all well tolerated in this reaction (**4a**–**4r**, **4u**). Acetyl group and ester group also performed well (**4s**, **4t**). This reaction is also workable to cinnamic acid, with an 83% yield obtained (**4v**). To our delight, most heterocycles are suitable for this reaction. Except for the product 4x, most of the heterocycles can give moderate to good decarbonylation cross-coupling compounds (**4w**, **4y**–**4ad**). In addition, steric hindrance has a slight impact on yield (**4e**, **4f**). To apply our discovery to medicinal chemistry and pharmaceutical research, we carried out direct derivatization of a drug. Adapalene (Retinoic acid drug) was cross-coupled with diphenyl disulfide to obtain compound 4ae with a yield of 76% (**4ae**). Despite the broad scope in the aforementioned examples, however, aliphatic carboxylic acids are not suitable for this reaction (**4af**).

To further demonstrate the potential of this method, we investigated reactions between carboxylic acids and diselenides (Table 4). When diselenide reacts with different naphthoic acid, a moderate to good yield can be obtained (**5a**–**5c**). Benzoic acids with different substituent can also provide cross-coupling products in moderate yields (**5e**–**5h**). For example, 4-phenylbenzoic acid can provide the target product with a yield of 40% (**5d**).

Table 1. Optimization of the reaction conditions.[a,b]

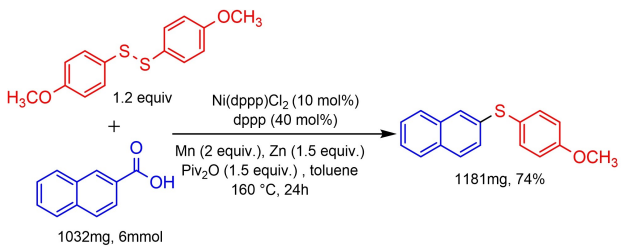


Entry	Catalyst	Ligand	Metal	Additive	Base	Yield 3a	Yield 3a'
1	$NiBr_2$(DME)	PPh_3	Mn	/	Na_2CO_3	none	/
2	$NiBr_2$(DME)	dppb	Mn	/	Na_2CO_3	none	48%
3	$NiBr_2$(DME)	dppp	Mn	/	Na_2CO_3	none	49%
4	$NiBr_2$(DME)	dppp	Mn	/	$NaHCO_3$	none	20%
5	$NiBr_2$(DME)	dppp	Mn	/	K_2CO_3	none	/
6	$NiBr_2$(DME)	dppp	Zn	/	Na_2CO_3	15%	/
7	$NiBr_2$(DME)	dppp	Mg	/	Na_2CO_3	none	/
8	$NiCl_2$	dppp	Zn	/	Na_2CO_3	none	/
9	Ni(dppp)Cl_2	dppp	Zn	/	Na_2CO_3	25%	/
10	Ni(OAc)·$4H_2O$	dppp	Zn	/	Na_2CO_3	trace	/
11	$NiBr_2$	dppp	Zn	/	Na_2CO_3	20%	/
12[c]	Ni(dppp)Cl_2	dppp	Zn	Piv_2O	Na_2CO_3	none	/
13[c]	Ni(dppp)Cl_2	dppp	Zn	Piv_2O	/	55%	/
14[c]	Ni(dppp)Cl_2	dppp	Zn	$CH_3(CO)_2O$	/	34%	/
15[d]	Ni(dppp)Cl_2	dppp	Zn	Piv_2O	/	62%	/
16[d]	Ni(dppp)Cl_2	dppp	Mn:Zn = 1.5:1.5	Piv_2O	/	71%	/
17[d]	Ni(dppp)Cl_2	dppp	Mn:Zn = 2:1.5	Piv_2O	/	75%	/
18[d]	Ni(dppp)Cl_2	dppp	Mn:Zn = 2.5:1.5	Piv_2O	/	74%	/
19[d]	Ni(dppp)Cl_2	dppp	Mn:Zn = 2:2.5	Piv_2O	/	71%	/
20[d]	Ni(dppp)Cl_2	dppp	Mn:Zn = 2:2	Piv_2O	/	59%	/
21[d,e]	Ni(dppp)Cl_2	dppp	Mn:Zn = 2:1.5	Piv_2O	/	80%	/
22[d,e]	Ni(dppp)Cl_2	dppp	/	Piv_2O	/	none	84%
23[d,e,f]	Ni(dppp)Cl_2	dppp	Mn	Piv_2O	/	trace	49%
24[d,e,g]	Ni(dppp)Cl_2	dppp	Zn	Piv_2O	/	45%	/
25[e]	Ni(dppp)Cl_2	dppp	Mn:Zn = 2:1.5	/	/	34%	/
26[d,e]	/	dppp	Mn:Zn = 2:1.5	Piv_2O	/	none	/
27[d,e]	Ni(dppp)Cl_2	/	Mn:Zn = 2:1.5	Piv_2O	/	none	/

[a] Reaction conditions: 2-naphthoic acid (0.5 mmol), 1,2-bis(4-chlorophenyl)disulfane (0.6 mmol), catalyst (10 mol%), ligand (20 mol%), metal (1.25 mmol), base (0.5 mmol) and toluene (2 mL), N_2, 160 °C, 24 h. [b] Isolated yields. [c] Anhydride (0.5 mmol). [d] Piv_2O (0.75 mmol). [e] dppp (40 mol%) [f] Mn (1 mmol). [g] Zn (0.75 mmol)

To prove the practicality of this method, we next performed a gram-scale reaction of 2-naphthoic acid with 1,2-bis(4-methoxyphenyl)disulfane. When the model reaction was performed on a 6.0 mmol scale, the yield of the desired product **3f** was obtained in 74% (Scheme 2), thus demonstrating the scalability of this strategy.

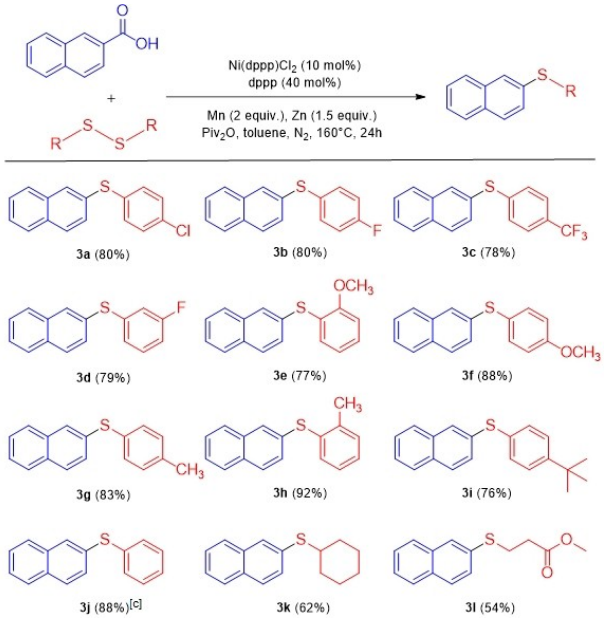
With the completion of the substrate expansion, we performed some mechanism control experiments (Scheme 3). It is reported in the literature that the combination of the carboxylic acid and pivalic anhydride was effective for the generation of the active mixed anhydride intermediate.[16] Therefore, we synthesized the mixed anhydride according to the relevant literature,[17] and under standard conditions, the desired product was isolated with a yield of 70% (Scheme 3a). In addition, in the absence of nickel source and pivalic anhydride, we have separated a small amount of thioester (Scheme 3b).



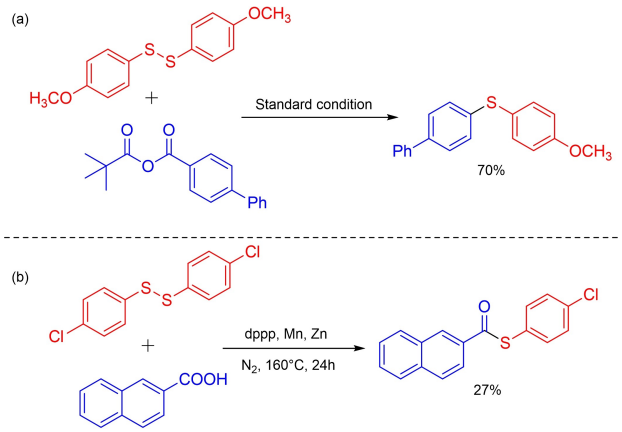
Scheme 2. Large-scale synthesis.

Based on the previous literature reports,[10,18] a plausible mechanism of the Ni(II)-catalyzed decarbonylation cross-coupling reaction of aryl dichalcogenides and carboxylic acids is shown in Scheme 4. We speculate that there are two reaction pathways. First, the carboxylic acid was activated in situ by Piv_2O to produce a mixing anhydride A, followed by oxidative addition with the Ni(0) give an intermediate B. At the same time, disulfide compound generated aryl thiolate ion under the action of reducing reagent.[19] Intermediate B was attacked by aryl thiolate ion to obtain compound E. The resulting E was further decarbonylated to generate intermediate F, which is then reductively eliminated to obtain the desired product. In addition, building the C–S bond may pass through path B. Carboxylic acid was attacked by aryl thiolate ion to form thioester. The C (acyl)–S bond of thioester D is oxidatively

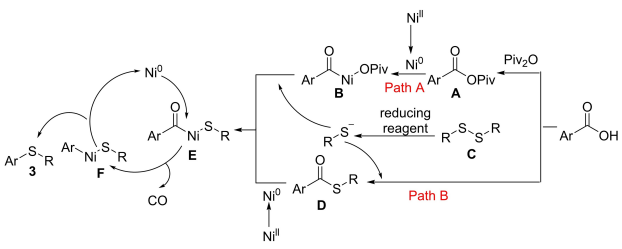
Table 2. Substrate scope of disulfides.[a,b]



[a] Reaction conditions: 2-naphthoic acid (0.5 mmol), disulfide (0.6 mmol), Ni(dppp)Cl_2 (10 mol%), dppp (40 mol%), Piv_2O (1.5 equiv.), Mn (1 mmol), Zn (0.75 mmol) and toluene (2 mL), 160 °C, 24 h. [b] Isolated yields. [c] dppp (20 mol%).



Scheme 3. Mechanistic experiments



Scheme 4. Proposed reaction mechanism.

added with Ni(0) to obtain acyl nickel (II) intermediate E. Intermediate E has undergone decarbonylation and reductive elimination to obtain target product and active Ni(0) to complete the catalytic cycle.

Conclusion

In conclusion, we have developed a nickel-catalyzed decarbonylative cross-coupling reaction of carboxylic acids and aryl dichalcogenides. This method can be applied practically with a wide range of substrates and good tolerance of functional groups, compatible with ketone, ester, cyanide and alkene. In this reaction carboxylic acid was used directly as the source of aryl group to realize one-pot construction of C–S(Se) bonds. By using this method, we can not only directly use low-odor and low-sensitivity aryl dichalcogenides to construct C–S(Se) bonds, but also make the reaction on gram scale with good yield.

Experimental Section

General Information

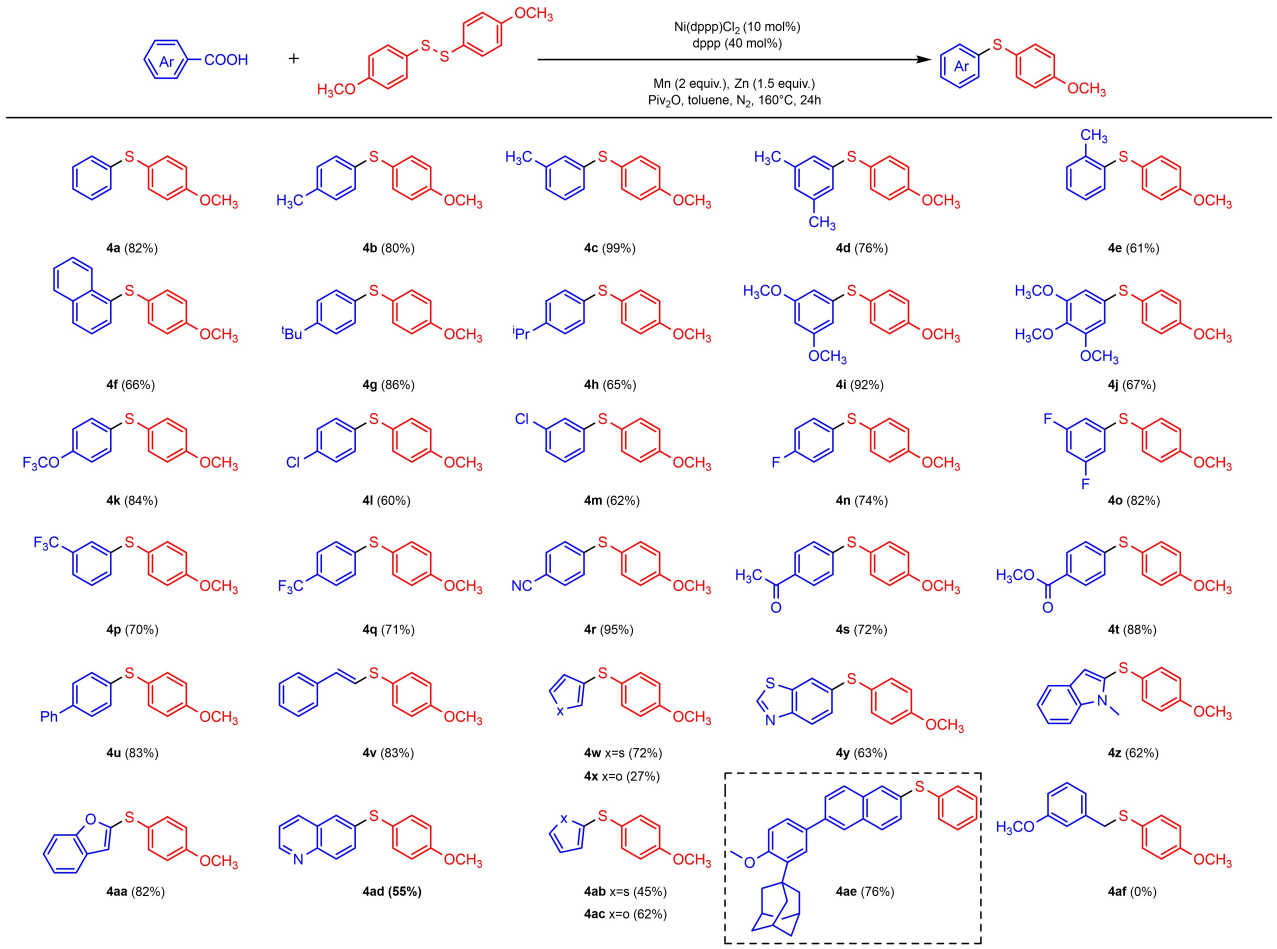
Reactants and reagents were purchased from commercial suppliers. All solvents were dried and freshly distilled. TLC was performed on silica HSGF254 plates. Melting points were determined with a digital melting-point apparatus. ^1H and ^{13}C NMR spectra were obtained from a solution in deuterated chloroform ($CDCl_3$) with tetramethylsilane as an internal standard using a 400/101 MHz ($^1H/^{13}C$) spectrometer. Chemical shifts (δ) are given in ppm and coupling constants (J) in Hz. Multiplicities are reported as follows: singlet (s), doublet (d), doublet of doublets (dd), triplet (t), quartet (q), and multiplet (m). Column chromatography was performed on silica gel 300–400 mesh. The structures of known compounds were further corroborated by comparing their ^1H, ^{13}C NMR data with those of literature. High-resolution mass spectra (HRMS) analyses were carried out on an electron impact ion source (EI) apparatus using time-of-flflight (TOF) mass spectrometry.

General procedure for synthesis of substrates. K_2CO_3 (0.25 mmol, 34.5 mg) was added to a 25 mL tube with a magnetic bar. MeCN (10 mL) and aryl thiols (20 mmol) were added and then the mixture was stirred at 30 °C under air and monitored by TLC. The solution was diluted with ethyl acetate (10 mL), and evaporated under vacuum. The crude reaction mixture was purified by column chromatograhy on silica gel (petroleum ether/ethyl acetate) to afford product 2.[20]

General Procedure for the Synthesis of Aryl Sulfide (3a–3l and 4a–4ae). Carboxylic acid (0.5 mol), disulfide (0.6 mmol), Ni(dppp)Cl_2 (0.05 mmol, 27 mg), dppp (0.2 mmol, 83 mg), Piv_2O (0.75 mmol, 140 mg), Mn (1 mmol, 55 mg) and Zn (0.75 mmol, 49 mg) were successively added into a 15 mL sealed tube, using anhydrous toluene (2 mL) as the solvent. The mixture was stirred in a 160 °C oil bath under nitrogen for 24 h. Upon completion of the reaction as indicated by TLC, the mixture was diluted with EtOAc and then filtered through a pad of celite. The solvent was removed under vacuum. The residue was purified on a silica column (petroleum ether/ethyl acetate) to give the pure target product.

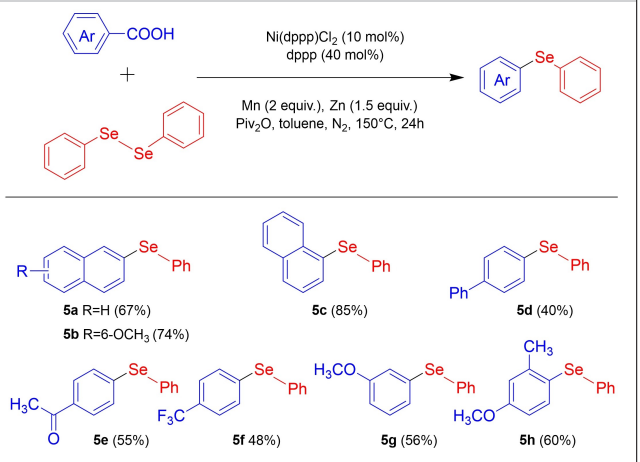
General Procedure for the Synthesis of Aryl Selenide (5a–5h). Carboxylic acid (0.5 mol), diselenide (0.6 mmol), Ni(dppp)Cl_2 (0.05 mmol, 27 mg), dppp (0.2 mmol, 83 mg), Piv_2O (0.75 mmol, 140 mg), Mn (1 mmol, 55 mg) and Zn (0.75 mmol, 49 mg) were successively added into a 15 mL sealed tube, using anhydrous

Table 3. Substrate scope of carboxylic acids.[a,b]



[a] Reaction conditions: carboxylic acid (0.5 mmol), 1,2-bis(4-methoxyphenyl)disulfane (0.6 mmol), Ni(dppp)Cl_2 (10 mol%), dppp (40 mol%), Piv_2O (1.5 equiv.), Mn (1 mmol), Zn (0.75 mmol) and toluene (2 mL), 160 °C, 24 h. [b] Isolated yields.

Table 4. Substrate scope of diselenides.[a,b]



[a] Reaction conditions: carboxylic acid (0.5 mmol), diselenide (0.6 mmol), Ni(dppp)Cl_2 (10 mol%), dppp (40 mol%), Piv_2O (1.5 equiv.), Mn (1 mmol), Zn (0.75 mmol) and toluene (2 mL), 150 °C, 24 h. [b] Isolated yields.

toluene (2 mL) as the solvent. The mixture was stirred in a 150 °C oil bath under nitrogen for 24 h. Upon completion of the reaction as indicated by TLC, the mixture was diluted with EtOAc and then filtered through a pad of celite. The solvent was removed under vacuum. The residue was purified on a silica column (petroleum ether/ethyl acetate) to give the pure target product.

1,2-di-o-tolyldisulfane (2a)[21] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55–7.46 (m, 2H), 7.18–7.07 (m, 6H), 2.41 (s, 6H).

1,2-bis(2-methoxyphenyl)disulfane (2b)[20] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, J = 7.6 Hz, 2H), 7.18 (t, J = 7.6 Hz, 2H), 6.90 (t, J = 7.4 Hz, 2H), 6.85 (d, J = 8.0 Hz, 2H), 3.89 (s, 6H).

1,2-bis(3-fluorophenyl)disulfane (2c)[21] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.24 (m, 4H), 7.22 (dd, J = 7.0, 1.9 Hz, 2H), 6.96–6.89 (m, 2H).

1,2-Bis(4-fluorophenyl)disulfane (2d)[20] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (dd, J = 8.2, 5.3 Hz, 4H), 6.99 (t, J = 8.5 Hz, 4H).

1,2-Bis(4-(trifluoromethyl)phenyl)disulfane (2e)[20] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61–7.54 (m, 8H).

1,2-Bis(4-(tert-butyl)phenyl)disulfane (2f)[21] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, J = 8.2 Hz, 4H), 7.32 (d, J = 8.3 Hz, 4H), 1.29 (s, 18H).

1,2-Bis(4-methoxyphenyl)disulfane (2 g)[20] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, J = 8.4 Hz, 4H), 6.82 (d, J = 8.4 Hz, 4H), 3.77 (s, 6H).

(4-Chlorophenyl)(naphthalen-2-yl)sulfane (3 a)[22] The title compound was isolated by column chromatography (petroleum ether) as a white solid (108 mg, 80% yield). mp 101.5–103.0 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (s, 1H), 7.83–7.71 (m, 3H), 7.52–7.45 (m, 2H), 7.38 (d, J = 8.5 Hz, 1H), 7.30–7.25 (m, 4H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 134.7 (s), 133.7 (s), 133.0 (s), 132.4 (s), 132.3 (s), 131.9 (s), 130.3 (s), 129.3 (s), 129.1 (s), 128.7 (s), 127.7 (s), 127.4 (s), 126.7 (s), 126.4 (s).

(4-Fluorophenyl)(naphthalen-2-yl)sulfane (3 b)[22] The title compound was isolated by column chromatography (petroleum ether) as a white solid (102 mg, 80% yield). mp 70.2–71.7 °C.^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, J = 8.0 Hz, 1H), 7.77–7.67 (m, 3H), 7.51–7.43 (m, 2H), 7.43–7.37 (m, 2H), 7.34 (d, J = 8.5 Hz, 1H), 7.03 (t, J = 8.5 Hz, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 162.4 (d, $J_{C\text{-}F}$ = 246 Hz), 134.0 (s), 133.9 (s), 133.8 (s), 133.7 (s), 132.1 (s), 130.3 (d, $J_{C\text{-}F}$ = 3.4 Hz), 128.9 (s), 128.6 (s), 127.8 (d, $J_{C\text{-}F}$ = 10.6 Hz), 127.3 (s), 126.7 (s), 126.1 (s), 116.4 (d, $J_{C\text{-}F}$ = 21.9 Hz).

Naphthalen-2-yl(4-(trifluoromethyl)phenyl)sulfane (3 c)[23] The title compound was isolated by column chromatography (petroleum ether) as a white solid (118 mg, 78% yield). mp 105.4–107.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.89–7.83 (m, 2H), 7.83–7.77 (m, 1H), 7.59–7.51 (m, 2H), 7.51–7.43 (m, 3H), 7.30 (d, J = 8.1 Hz, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 142.8 (d, J = 1.2 Hz), 133.8 (s), 133.0 (s), 132.9 (s), 130.2 (s), 129.6 (s), 129.4 (s), 128.3 (s), 127.8 (s), 127.7 (s), 127.0 (s), 126.9 (s), 125.8 (q, $J_{C\text{-}F}$ = 3.7 Hz), 122.7 (q, $J_{C\text{-}F}$ = 272.9 Hz).

(3-Fluorophenyl)(naphthalen-2-yl)sulfane (3 d) The title compound was isolated by column chromatography (petroleum ether) as a white solid (100 mg, 79% yield). mp 60.5–61.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.87–7.75 (m, 3H), 7.54–7.48 (m, 2H), 7.44 (dd, J = 8.6, 1.7 Hz, 1H), 7.22 (dd, J = 8.1, 6.1 Hz, 1H), 7.07 (dd, J = 7.8, 0.7 Hz, 1H), 6.97 (dt, J = 9.3, 1.8 Hz, 1H), 6.89 (td, J = 8.4, 2.5 Hz, 1H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 163.0 (d, $J_{C\text{-}F}$ = 249.4 Hz), 139.2 (d, $J_{C\text{-}F}$ = 7.7 Hz), 133.8 (s), 132.7 (s), 131.8 (s), 131.0 (s), 130.3 (d, $J_{C\text{-}F}$ = 8.5 Hz), 129.6 (s), 129.2 (s), 127.7 (d, $J_{C\text{-}F}$ = 17.9 Hz), 126.7 (d, $J_{C\text{-}F}$ = 6.2 Hz), 125.1 (d, $J_{C\text{-}F}$ = 3.0 Hz), 116.4 (d, $J_{C\text{-}F}$ = 23.4 Hz), 113.5 (d, $J_{C\text{-}F}$ = 21.4 Hz). HRMS (EI) m/z calcd for $C_{16}H_{11}FS$ $[M]^+$: 254.0566, found: 254.0566

(2-methoxyphenyl)(naphthalen-2-yl)sulfane (3 e)[11] The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/300) as a colorless oil (102 mg, 77% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (s, 1H), 7.82–7.68 (m, 3H), 7.52–7.43 (m, 2H), 7.41 (d, J = 8.6 Hz, 1H), 7.26 (d, J = 8.1 Hz, 1H), 7.11 (d, J = 7.4 Hz, 1H), 6.92 (d, J = 8.1 Hz, 1H), 6.86 (t, J = 7.3 Hz, 1H), 3.87 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 157.32 (s), 133.83 (s), 132.3 (s), 131.9 (s), 131.7 (s), 130.2 (s), 129.0 (s), 128.7 (s), 128.4 (s), 127.7 (s), 127.4 (s), 126.4 (s), 126.1 (s), 124.0 (s), 121.3 (s), 110.9 (s), 55.9 (s).

(4-methoxyphenyl)(naphthalen-2-yl)sulfane (3 f)[22] The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/300) as a white solid (117 mg, 88% yield). mp 67.7–69.0 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.73–7.64 (m, 2H), 7.59 (s, 1H), 7.49–7.38 (m, 4H), 7.29 (d, J = 8.4 Hz, 1H), 6.91 (d, J = 8.6 Hz, 2H), 3.83 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.8 (s), 135.9 (s), 135.2 (s), 133.7 (s), 131.7 (s), 128.5 (s), 127.7 (s), 127.1 (s), 126.7 (s), 126.5 (s), 126.4 (s), 125.6 (s), 124.4 (s), 115.0 (s), 55.5 (s).

Naphthalen-2-yl(p–tolyl)sulfane (3 g)[22] The title compound was isolated by column chromatography (petroleum ether) as a white solid (104 mg, 83% yield). mp 68.7–70.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 7.5 Hz, 1H), 7.75–7.66 (m, 3H), 7.49–7.40 (m, 2H), 7.34 (t, J = 8.6 Hz, 3H), 7.14 (d, J = 7.7 Hz, 2H), 2.35 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 137.6 (s), 134.3 (s), 133.7 (s), 132.1 (s), 132.0 (s), 131.4 (s), 130.1 (s), 128.7 (s), 128.4 (s), 127.9 (s), 127.7 (s), 127.3 (s), 126.5 (s), 125.9 (s), 21.1 (s).

Naphthalen-2-yl(o–tolyl)sulfane (3 h)[11] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (115 mg, 92% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 7.5 Hz, 1H), 7.73 (d, J = 8.6 Hz, 1H), 7.70–7.61 (m, 2H), 7.48–7.39 (m, 2H), 7.36–7.27 (m, 2H), 7.27–7.19 (m, 2H), 7.15 (t, J = 7.3 Hz, 1H), 2.40 (s, 3H).^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 139.9 (s), 133.8 (d, J = 9.2 Hz), 133.5 (s), 132.9 (s), 131.9 (s), 130.6 (s), 128.7 (s), 128.1 (s), 127.9 (s), 127.69 (s), 127.66 (s), 127.2 (s), 126.7 (s), 126.5 (s), 125.8 (s), 20.6 (s).

(4-(Tert-butyl)phenyl)(naphthalen-2-yl)sulfane (3 i)[22] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (111 mg, 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81–7.66 (m, 4H), 7.49–7.40 (m, 2H), 7.38 (d, J = 8.6 Hz, 1H), 7.33 (s, 4H), 1.31 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 150.5 (s), 133.8 (s), 133.8 (s), 132.1 (s), 131.7 (s), 131.3 (s), 128.9 (s), 128.7 (s), 128.3 (s), 127.7 (s), 127.3 (s), 126.4 (s), 126.0 (s), 34.5 (s), 31.2 (s).

Naphthalen-2-yl(phenyl)sulfane (3 j)[22] The title compound was isolated by column chromatography (petroleum ether) as a white solid (104 mg, 88% yield). mp 55.4–57.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (s, 1H), 7.82–7.70 (m, 3H), 7.53–7.43 (m, 2H), 7.43–7.35 (m, 3H), 7.35–7.26 (m, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 135.8 (s), 133.8 (s), 133.0 (s), 132.3 (s), 130.9 (s), 129.9 (s), 129.2 (s), 128.8 (s), 128.7 (s), 127.7 (s), 127.4 (s), 127.0 (s), 126.6 (s), 126.2 (s).

Cyclohexyl(naphthalen-2-yl)sulfane (3 k)[24] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (75 mg, 62% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (s, 1H), 7.82–7.69 (m, 3H), 7.53–7.38 (m, 3H), 3.29–3.16 (m, 1H), 2.02 (d, J = 12.5 Hz, 2H), 1.85–1.72 (m, 2H), 1.68–1.58 (m, 1H), 1.48–1.27 (m, 5H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 133.6 (s), 132.6 (s), 132.0 (s), 130.1 (s), 129.6 (s), 128.2 (s), 127.6 (s), 127.2 (s), 126.4 (s), 125.8 (s), 46.5 (s), 33.34 (s), 26.0 (s), 25.8 (s).

Methyl 3-(naphthalen-2-ylthio)propanoate (3 l) The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/40) as a colorless oil (66 mg, 54% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83–7.71 (m, 4H), 7.53–7.41 (m, 3H), 3.68 (s, 3H), 3.27 (t, J = 7.3 Hz, 2H), 2.68 (t, J = 7.3 Hz, 2H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 172.2 (s), 133.7 (s), 132.6 (s), 132.0 (s), 128.6 (s), 128.1 (s), 127.8 (s), 127.7 (s), 127.2 (s), 126.6 (s), 125.9 (s), 51.8 (s), 34.2 (s), 28.9 (s). HRMS (EI) m/z calcd for $C_{14}H_{14}O_2S$ $[M]^+$: 246.0715, found: 246.0721

(4-Methoxyphenyl)(phenyl)sulfane (4 a)[25] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (89 mg, 82% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, J = 8.5 Hz, 2H), 7.25–7.19 (m, 2H), 7.19–7.10 (m, 3H), 6.89 (d, J = 8.6 Hz, 2H), 3.81 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.8 (s), 138.6 (s), 135.3 (s), 128.9 (s), 128.2 (s), 125.7 (s), 124.3 (s), 115.0 (s), 55.3 (s).

(4-Methoxyphenyl)(p-tolyl)sulfane (4 b)[25] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (92 mg, 80% yield). ^{1}H NMR (400 MHz,$CDCl_3$) δ 7.36 (d, J = 8.5 Hz, 2H), 7.13 (d, J = 8.0 Hz, 2H), 7.06 (d, J = 7.9 Hz, 2H), 6.86 (d, J = 8.5 Hz, 2H), 3.80 (s, 3H), 2.30 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.4 (s), 136.1 (s), 134.3 (s), 134.3 (s), 129.7 (s), 129.3 (s), 125.6 (s), 114.8 (s), 55.3 (s), 21.0 (s).

(4-Methoxyphenyl)(m-tolyl)sulfane (4 c)[26] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (114 mg, 99% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.40 (m, 1H), 7.40–7.37 (m, 1H), 7.15–7.09 (m, 1H), 7.01 (s, 1H), 6.98–6.93 (m, 2H), 6.91–6.89 (m, 1H), 6.88–6.85 (m, 1H), 3.81 (s, 3H), 2.27 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.7 (s), 138.7 (s), 138.1 (s),

135.1 (s), 128.9 (s), 128.8 (s), 126.7 (s), 125.5 (s), 124.6 (s), 114.9 (s), 55.3 (s), 21.3 (s).

(3,5-Dimethylphenyl)(4-methoxyphenyl)sulfane (4d)[25] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (93 mg, 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, J = 8.0 Hz, 2H), 6.88 (d, J = 8.0 Hz, 2H), 6.79 (d, J = 13.3 Hz, 3H), 3.81 (s, 3H), 2.22 (s, 6H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.6 (s), 138.6 (s), 137.7 (s), 135.0 (s), 127.8 (s), 126.2 (s), 124.8 (s), 114.8 (s), 55.3 (s), 21.2 (s).

(4-Methoxyphenyl)(o–tolyl)sulfane (4e)[26] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (70 mg, 61% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, J = 8.3 Hz, 2H), 7.17 (d, J = 6.9 Hz, 1H), 7.12–7.02 (m, 2H), 6.97 (d, J = 7.2 Hz, 1H), 6.89 (d, J = 8.3 Hz, 2H), 3.81 (s, 3H), 2.38 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.5 (s), 137.03 (s), 136.99 (s), 134.5 (s), 130.2 (s), 129.0 (s), 126.4 (s), 126.1 (s), 124.4 (s), 115.0 (s), 55.3 (s), 20.3 (s).

(4-Methoxyphenyl)(naphthalen-1-yl)sulfane (4f)[27] The title compound was isolated by column chromatography (petroleum ether) as a white solid (88 mg, 66% yield). mp 105.3-106.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (d, J = 7.6 Hz, 1H), 7.84 (d, J = 7.4 Hz, 1H), 7.72 (d, J = 6.6 Hz, 1H), 7.57–7.45 (m, 2H), 7.33 (d, J = 7.0 Hz, 4H), 6.86 (d, J = 8.2 Hz, 2H), 3.78 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.3 (s), 134.6 (s), 133.9 (s), 133.8 (s), 132.2 (s), 128.5 (s), 128.4 (s), 127.4 (s), 126.5 (s), 126.3 (s), 125.7 (s), 125.1 (s), 124.9 (s), 114.98 (s).

(4-(Tert-butyl)phenyl)(4-methoxyphenyl)sulfane (4g)[27] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (117 mg, 86% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, J = 7.9 Hz, 2H), 7.28–7.23 (m, 2H), 7.13 (d, J = 7.6 Hz, 2H), 6.88 (d, J = 7.8 Hz, 2H), 3.80 (s, 3H), 1.28 (s, 9H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.6 (s), 149.2 (s), 134.8 (s), 134.6 (s), 128.5 (s), 126.0 (s), 125.1 (s), 114.9 (s), 55.3 (s), 34.4 (s), 31.3 (s).

(4-Isopropylphenyl)(4-methoxyphenyl)sulfane (4h)[28] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (84 mg, 65% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, J = 8.6 Hz, 2H), 7.20–7.03 (m, 4H), 6.87 (d, J = 8.6 Hz, 2H), 3.80 (s, 3H), 2.91–2.79 (m, 1H), 1.21 (d, J = 6.9 Hz, 6H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.5 (s), 147.0 (s), 134.8 (s), 134.6 (s), 129.0 (s), 127.1 (s), 125.3 (s), 114.8 (s), 55.3 (s), 33.6 (s), 23.9 (s).

(3,5-Dimethoxyphenyl)(4-methoxyphenyl)sulfane (4i) The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/40) as a yellow oil (127 mg, 92% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, J = 8.3 Hz, 2H), 6.90 (d, J = 8.3 Hz, 2H), 6.28 (s, 2H), 6.23 (s, 1H), 3.82 (s, 3H), 3.71 (s, 6H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.9 (s), 160.0 (s), 141.0 (s), 135.9 (s), 123.3 (s), 115.0 (s), 105.6 (s), 97.9 (s), 55.31 (s), 55.27 (s). HRMS (EI) m/z calcd for $C_{15}H_{16}O_3S$ $[M]^+$: 276.0820, found: 276.0824

(4-Methoxyphenyl)(3,4,5-trimethoxyphenyl)sulfane (4j)[29] The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/20) as a white solid (103 mg, 67% yield). mp 122.2–123.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, J = 8.3 Hz, 2H), 6.89 (d, J = 8.3 Hz, 2H), 6.47 (s, 2H), 3.82 (d, J = 2.8 Hz, 6H), 3.76 (s, 6H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.6 (s), 153.5 (s), 136.7 (s), 134.4 (s), 132.7 (s), 125.1(s), 114.9 (s), 106.5 (s), 60.9 (s), 56.1 (s), 55.4 (s).

(4-Methoxyphenyl)(4-(trifluoromethoxy)phenyl)sulfane (4k)[27] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (126 mg, 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 8.2 Hz, 2H), 7.14 (d, J = 8.4 Hz, 2H), 7.07 (d, J = 8.2 Hz, 2H), 6.91 (d, J = 8.2 Hz, 2H), 3.81 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.2 (s), 147.2 (d, $J_{C\text{-}F}$ = 1.6 Hz), 137.8 (s), 135.8 (s), 132.1 (q, $J_{C\text{-}F}$ = 234.9 Hz), 129.0 (s), 123.4 (s), 121.6 (s),119.1 (s) 115.2 (s), 55.4 (s).

(4-Chlorophenyl)(4-methoxyphenyl)sulfane (4l)[27] The title compound was isolated by column chromatography (petroleum ether) as a white solid (75 mg, 60% yield). mp 64.7–65.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, J = 8.2 Hz, 2H), 7.19 (d, J = 8.1 Hz, 2H), 7.07 (d, J = 8.1 Hz, 2H), 6.90 (d, J = 8.2 Hz, 2H), 3.83 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.0 (s), 137.3 (s), 135.5 (s), 131.6 (s), 129.3 (s), 129.0 (s), 123.7 (s), 115.1 (s), 55.4 (s).

(3-Chlorophenyl)(4-methoxyphenyl)sulfane (4m)[28] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (77 mg, 62% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, J = 8.3 Hz, 2H), 7.14 (t, J = 7.7 Hz, 1H), 7.07 (d, J = 9.3 Hz, 2H), 7.00 (d, J = 7.5 Hz, 1H), 6.93 (d, J = 8.3 Hz, 2H), 3.84 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.3 (s), 141.3 (s), 136.1 (s), 134.8 (s), 129.8 (s), 127.0 (s), 125.6 (s), 125.4 (s), 122.6 (s), 115.2 (s), 55.4 (s).

(4-Fluorophenyl)(4-methoxyphenyl)sulfane (4n)[27] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (86 mg, 74% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, J = 8.4 Hz, 2H), 7.23–7.13 (m, 2H), 6.95 (t, J = 8.5 Hz, 2H), 6.88 (d, J = 8.4 Hz, 2H), 3.81 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 162.8 (d, $J_{C\text{-}F}$ = 246.8 Hz), 159.6 (s), 134.5 (s), 133.1 (d, $J_{C\text{-}F}$ = 3.3 Hz), 131.0 (d, $J_{C\text{-}F}$ = 8.0 Hz), 125.2 (s), 116.2 (d, $J_{C\text{-}F}$ = 22.0 Hz), 115.0 (s), 55.4 (s).

(3,5-Difluorophenyl)(4-methoxyphenyl)sulfane (4o)[30] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (103 mg, 82% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, J = 8.2 Hz, 2H), 6.95 (d, J = 8.2 Hz, 2H), 6.60–6.47 (m, 3H), 3.85 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 164.3 (d, $J_{C\text{-}F}$ = 13.2 Hz), 161.8 (d, $J_{C\text{-}F}$ = 13.2 Hz), 160.8 (s), 144.0 (t, $J_{C\text{-}F}$ = 9.6 Hz), 136.9 (s), 132.0 (s), 121.1 (s), 115.4 (s), 114.9 (s), 109.2 (d, $J_{C\text{-}F}$ = 7.6 Hz), 109.0 (d, $J_{C\text{-}F}$ = 7.6 Hz), 100.6 (t, $J_{C\text{-}F}$ = 25.8 Hz), 55.4 (s).

(4-Methoxyphenyl)(3-(trifluoromethyl)phenyl)sulfane (4p)[25] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (100 mg, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 8.3 Hz, 2H), 7.39–7.28 (m, 3H), 7.24 (d, J = 8.8 Hz, 1H), 6.94 (d, J = 8.3 Hz, 2H), 3.84 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.4 (s), 140.8 (s), 136.2 (s), 131.6 (d, JC-F = 32.5 Hz), 130.9 (d, $J_{C\text{-}F}$ = 32.4 Hz), 130.3 (d, $J_{C\text{-}F}$ = 1.1 Hz), 129.2 (s), 123.8 (q, $J_{C\text{-}F}$ = 3.9 Hz), 122.4 (s), 122.3 (s), 122.1 (q, $J_{C\text{-}F}$ = 3.8 Hz), 115.3 (s), 55.4 (s).

(4-Methoxyphenyl)(4-(trifluoromethyl)phenyl)sulfane (4q)[11] The title compound was isolated by column chromatography (petroleum ether) as a white solid (101 mg, 71% yield). mp 83.5-85.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (dd, J = 13.6, 8.1 Hz, 4H), 7.13 (d, J = 7.9 Hz, 2H), 6.95 (d, J = 7.8 Hz, 2H), 3.85 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.6 (s), 144.8 (s), 136.7 (s), 126.4 (s), 125.6 (q, $J_{C\text{-}F}$ = 3.8 Hz), 122.8 (q, $J_{C\text{-}F}$ = 272.7 Hz), 121.6 (s), 115.4 (s), 55.4 (s).

4-((4-Methoxyphenyl)thio)benzonitrile (4r)[27] The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/30) as a white solid (115 mg, 95% yield). mp 102.5-103.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (t, J = 9.3 Hz, 4H), 7.07 (d, J = 8.3 Hz, 2H), 6.97 (d, J = 8.5 Hz, 2H), 3.86 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.9 (s), 147.3 (s), 137.1 (s), 132.2 (s), 126.0 (s), 120.3 (s), 118.9 (s), 115.5 (s), 108.0 (s), 55.4 (s).

1-(4-((4-Methoxyphenyl)thio)phenyl)ethan-1-one (4s)[26] The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/30) as a yellow oil (93 mg, 72% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, J = 8.0 Hz, 2H), 7.48 (d, J = 8.0 Hz, 2H), 7.09 (d, J = 8.0 Hz, 2H), 6.96 (d, J = 8.0 Hz, 2H), 3.85 (s, 3H), 2.53 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 197.1 (s), 160.6 (s), 146.9 (s), 136.8 (s), 133.8 (s), 128.8 (s), 125.8 (s), 121.3 (s), 115.3 (s), 55.4 (s), 26.4 (s).

Methyl 4-((4-methoxyphenyl)thio)benzoate (4t)[31] The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/30) as a yellow solid (120 mg, 88% yield). mp 92.5-93.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 8.1 Hz, 2H), 7.47 (d, J = 8.3 Hz, 2H), 7.08 (d, J = 8.1 Hz, 2H), 6.95 (d, J = 8.3 Hz, 2H), 3.87 (s, 3H), 3.85 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 166.8 (s), 160.6 (s), 146.4 (s), 136.7 (s), 129.9 (s), 126.7 (s), 125.8 (s), 121.6 (s), 115.3 (s), 55.4 (s), 52.0 (s).

[1,1′-biphenyl]-4-yl(4-methoxyphenyl)sulfane (4u)[26] The title compound was isolated by column chromatography (petroleum ether) as a white solid (121 mg, 83% yield). mp 96.5–97.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, J = 7.5 Hz, 2H), 7.48–7.39 (m, 6H), 7.32 (t, J = 7.2 Hz, 1H), 7.23 (d, J = 8.3 Hz, 2H), 6.92 (d, J = 8.5 Hz, 2H), 3.83 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.9 (s), 140.4 (s), 138.7 (s), 137.8 (s), 135.4 (s), 128.8 (s), 128.5 (s), 127.6 (s), 127.2 (s), 126.8 (s), 124.2 (s), 115.0 (s), 55.4 (s).

(E)-(4-Methoxyphenyl)(styryl)sulfane (4v)[32] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (100 mg, 83% yield). 1H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, J = 8.5 Hz, 2H), 7.28 (d, J = 4.1 Hz, 4H), 7.23–7.15 (m, 1H), 6.90 (d, J = 8.5 Hz, 2H), 6.82 (d, J = 15.4 Hz, 1H), 6.50 (d, J = 15.4 Hz, 1H), 3.82 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.5 (s), 136.7 (s), 133.5 (s), 128.9 (s), 128.6 (s), 127.2 (s), 125.8 (s), 125.7 (s), 124.5 (s), 114.9 (s), 55.4 (s).

3-((4-Methoxyphenyl)thio)thiophene (4w)[31] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (80 mg, 72% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35–7.28 (m, 3H), 7.12 (d, J = 1.6 Hz, 1H), 6.94 (d, J = 4.6 Hz, 1H), 6.84 (d, J = 8.6 Hz, 2H), 3.79 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.1 (s), 132.7 (s), 132.4 (s), 129.7 (s), 126.4 (s), 126.3 (s), 124.3 (s), 114.8 (s), 55.3 (s).

3-((4-Methoxyphenyl)thio)furan (4x)[33] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (28 mg, 27% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.44 (s, 1H), 7.24 (m, 2H), 6.82 (d, J = 8.6 Hz, 2H), 6.37 (s, 1H), 3.78 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.7 (s), 144.5 (s), 143.9 (s), 131.0 (s), 126.9 (s), 116.4 (s), 114.7 (s), 113.9 (s), 55.4 (s).

6-((4-Methoxyphenyl)thio)benzo[d]thiazole (4y) The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/20) as a yellow solid (86 mg, 63% yield). mp 81.5-82.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.90 (s, 1H), 7.98 (d, J = 8.6 Hz, 1H), 7.66 (s, 1H), 7.46 (d, J = 8.1 Hz, 2H), 7.35 (d, J = 8.5 Hz, 1H), 6.93 (d, J = 8.1 Hz, 2H), 3.84 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.1 (s), 153.4 (s), 151.5 (s), 136.8 (s), 135.6 (s), 134.8 (s), 126.7 (s), 123.9 (s), 123.7 (s), 120.6 (s), 115.2 (s), 55.4 (s). HRMS (EI) m/z calcd for $C_{14}H_{11}NOS_2$ [M]$^+$: 273.0282, found: 273.0281

2-((4-Methoxyphenyl)thio)-1-methyl-1H-indole (4z)[34] The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/60) as a yellow solid (86 mg, 62% yield). mp 64.5–65.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, J = 7.9 Hz, 1H), 7.32–7.25 (m, 2H), 7.12 (t, J = 8.1 Hz, 3H), 6.86 (s, 1H), 6.78 (d, J = 8.6 Hz, 2H), 3.75 (s, 3H), 3.68 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.5 (s), 138.5 (s), 129.7 (s), 129.1 (s), 127.2 (s), 126.8 (s), 122.6 (s), 120.7 (s), 119.8 (s), 114.8 (s), 110.5 (s), 109.7 (s), 55.3 (s), 29.9 (s).

2-((4-Methoxyphenyl)thio)benzofuran (4aa) The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (105 mg, 82% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, J = 7.7 Hz, 1H), 7.41 (d, J = 8.3 Hz, 3H), 7.28–7.23 (m, 1H), 7.19 (t, J = 7.4 Hz, 1H), 6.87–6.84 (m, 2H), 6.84–6.81 (m, 1H), 3.77 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.6 (s), 156.5 (s), 150.1 (s), 132.9 (s), 128.5 (s), 124.7 (s), 123.5 (s), 122.9 (s), 120.6 (s), 114.9 (s), 111.8 (s), 111.2 (s), 55.3 (s). HRMS (EI) m/z calcd for $C_{15}H_{12}O_2S$ [M]$^+$: 256.0558, found: 256.0557

2-((4-Methoxyphenyl)thio)thiophene (4ab)[35] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (50 mg, 45% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, J = 5.1 Hz, 1H), 7.28 (d, J = 8.6 Hz, 2H), 7.20 (d, J = 2.6 Hz, 1H), 7.02–6.96 (m, 1H), 6.82 (d, J = 8.6 Hz, 2H), 3.77 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.9 (s), 134.3 (s), 133.9 (s), 131.1 (s), 123.0 (s), 128.3 (s), 127.6 (s), 114.7 (s), 55.3 (s).

2-((4-Methoxyphenyl)thio)furan (4ac) The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (64 mg, 62% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (s, 1H), 7.30–7.24 (m, 2H), 6.81 (d, J = 8.6 Hz, 2H), 6.64 (d, J = 2.7 Hz, 1H), 6.41 (s, 1H), 3.76 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 159.0 (s), 145.9 (s), 144.9 (s), 131.1 (s), 125.9 (s), 117.7 (s), 114.7 (s), 111.7 (s), 55.3 (s). HRMS (EI) m/z calcd for $C_{11}H_{10}O_2S$ [M]$^+$: 206.0402, found: 206.0405

6-((4-Methoxyphenyl)thio)quinoline (4ad)[36] The title compound was isolated by column chromatography (petroleum ether) as a colorless oil (73 mg, 55% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (d, J = 1.8 Hz, 1H), 7.96 (d, J = 8.4 Hz, 2H), 7.53–7.43 (m, 4H), 7.36–7.29 (m, 1H), 6.95 (d, J = 8.2 Hz, 2H), 3.84 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 160.2 (s), 149.7 (s), 146.6 (s), 137.8 (s), 135.9 (s), 135.0 (s), 129.8 (s), 129.5 (s), 128.6 (s), 124.8 (s), 123.1 (s), 121.5 (s), 115.2 (s), 55.3 (s).

(5-((3r,5r,7r)-Adamantan-1-yl)-6-(4-methoxyphenyl) naphthalen-2-yl) (phenyl)sulfane (4ae) The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/120) as a white solid (193 mg, 76% yield). mp 157.8-158.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.86 (s, 1H), 7.82–7.75 (m, 2H), 7.73 (d, J = 8.2 Hz, 1H), 7.57 (s, 1H), 7.51 (d, J = 8.1 Hz, 1H), 7.42 (d, J = 8.4 Hz, 1H), 7.37 (d, J = 6.9 Hz, 2H), 7.31 (t, J = 6.6 Hz, 2H), 7.26–7.24 (m, 1H), 6.98 (d, J = 8.1 Hz, 1H), 3.89 (s, 3H), 2.18 (s, 6H), 2.10 (s, 3H), 1.80 (s, 6H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.7 (s), 139.4 (s), 138.9 (s), 136.2 (s), 132.9 (s), 132.7 (s), 132.6 (s), 132.3 (s), 130.7 (s), 130.0 (s), 129.3 (s), 129.2 (s), 129.0 (s), 127.8 (s), 126.9 (s), 126.4 (s), 125.9 (s), 125.6 (s), 124.8 (s), 112.1 (s), 55.2 (s), 40.6 (s), 37.2 (s), 37.1 (s), 29.1 (s). HRMS (EI) m/z calcd for $C_{33}H_{32}OS$ [M]$^+$: 476.2174, found: 476.2186

Naphthalen-2-yl(phenyl)selane (5a)[37] The title compound was isolated by column chromatography (petroleum ether) as a yellow solid (95 mg, 67% yield). mp 51.2–52.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.84–7.77 (m, 1H), 7.73 (d, J = 7.8 Hz, 2H), 7.50–7.43 (m, 4H), 7.31–7.22 (m, 4H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 134.0 (s), 132.9 (s), 132.0 (s), 131.2 (s), 130.5 (s), 129.4 (s), 128.8 (s), 128.4 (s), 127.8 (s), 127.4 (s), 127.3 (s), 126.5 (s), 126.21 (s).

(6-Methoxynaphthalen-2-yl)(phenyl)selane (5b) The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/120) as a white solid (116 mg, 74% yield). mp 106.3–107.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (s, 1H), 7.68–7.62 (m, 2H), 7.52 (d, J = 8.4 Hz, 1H), 7.46–7.39 (m, 2H), 7.26–7.23 (m, 3H), 7.15 (d, J = 9.0 Hz, 1H), 7.10 (s, 1H), 3.91 (s, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 158.1 (s), 133.8 (s), 133.0 (s), 132.1 (s), 132.0 (s), 131.8 (s), 129.5 (s), 129.3 (s), 129.0 (s), 127.7 (s), 126.9 (s), 124.8 (s), 119.3 (s), 105.73 (s), 55.3 (s). HRMS (EI) m/z calcd for $C_{17}H_{14}OSe$ [M]$^+$: 314.0210, found: 314.0217

Naphthalen-1-yl(phenyl)selane (5c)[38] The title compound was isolated by column chromatography (petroleum ether) as a yellow oil (120 mg, 85% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38–8.29 (m, 1H), 7.85 (d, J = 7.7 Hz, 2H), 7.77 (d, J = 6.9 Hz, 1H), 7.56–7.46 (m, 2H), 7.42–7.31 (m, 3H), 7.23–7.15 (m, 3H). ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 134.1 (s), 134.1 (s), 133.8 (s), 131.68 (s), 131.66 (s), 129.4 (s),

129.3 (s), 129.2 (s), 128.6 (s), 127.6 (s), 126.9 (s), 126.8 (s), 126.3 (s), 126.0 (s).

[1,1′-Biphenyl]-4-yl(4-methoxyphenyl)selane (5 d)[39] The title compound was isolated by column chromatography (petroleum ether) as a yellow solid (68 mg, 40% yield). mp 69.3–70.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, J = 7.5 Hz, 2H), 7.54–7.46 (m, 6H), 7.43 (t, J = 7.4 Hz, 2H), 7.38–7.31 (m, 1H), 7.31–7.26 (m, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 140.4 (s), 140.3 (s), 133.3 (s), 133.1 (s), 131.0 (s), 130.2 (s), 129.4 (s), 128.8 (s), 128.0 (s), 127.5 (s), 127.4 (s), 126.9 (s).

1-(4-(Phenylselanyl)phenyl)ethan-1-one (5 e)[38] The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/30) as a yellow solid (71 mg, 55% yield). mp 59.0–60.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, J = 7.9 Hz, 2H), 7.58 (d, J = 6.1 Hz, 2H), 7.44–7.29 (m, 5H), 2.54 (s, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 197.2 (s), 140.2 (s), 135.1 (s), 135.0 (s), 130.2 (s), 129.7 (s), 128.8 (s), 128.5 (s), 128.4 (s), 26.4 (s).

Phenyl(4-(trifluoromethyl)phenyl)selane (5 f)[40] The title compound was isolated by column chromatography (petroleum ether) as a yellow oil (72 mg, 48% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58–7.54 (m, 2H), 7.48–7.39 (m, 4H), 7.39–7.29 (m, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 137.8 (s), 134.8 (s), 133.0 (s), 131.0 (s), 129.7 (s), 128.7 (s), 128.5 (s), 125.9 (q, $J_{C\text{-}F}$ = 3.8 Hz), 122.7 (q, $J_{C\text{-}F}$ = 273.0 Hz).

(3-Methoxyphenyl)(phenyl)selane (5 g)[41] The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/300) as a yellow oil (74 mg, 56% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (s, 2H), 7.36–7.08 (m, 4H), 7.08–6.91 (m, 2H), 6.79 (d, J = 7.9 Hz, 1H), 3.74 (s, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 160.0 (s), 133.2 (s), 132.2 (s), 130.7 (s), 130.0 (s), 129.3 (s), 127.4 (s), 124.9 (s), 118.0 (s), 113.1 (s), 55.2 (s).

(4-Methoxy-2-methylphenyl)(phenyl)selane (5 h) The title compound was isolated by column chromatography (eluent: EtOAc/petroleum ether = 1/300) as a colorless oil (83 mg, 60% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, J = 8.3 Hz, 1H), 7.21 (dd, J = 18.5, 9.2 Hz, 5H), 6.85 (s, 1H), 6.69 (d, J = 8.2 Hz, 1H), 3.80 (s, 3H), 2.39 (s, 3H). $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 160.2 (s), 143.3 (s), 137.8 (s), 132.8 (s), 130.2 (s), 129.1 (s), 126.1 (s), 120.6 (s), 116.1 (s), 112.2 (s), 55.2 (s), 23.1 (s). HRMS (EI) m/z calcd for $C_{14}H_{14}OSe$ $[M]^+$: 278.0210, found: 278.0209

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Conflict of Interest

The authors declare no conflict of interest.

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