Supporting Information

Additive-Free Four-Component Radical Selenosulfonylation of Alkenes

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(A) General information

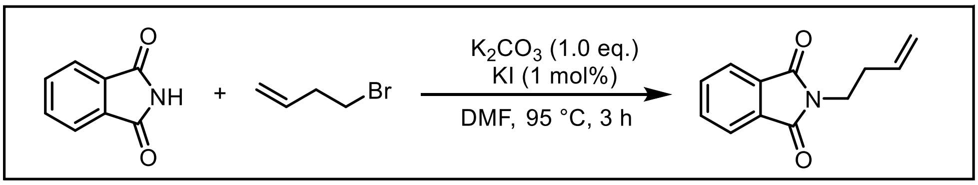
All chemicals were acquired from commercial sources and were employed as received unless otherwise mentioned. The reaction was monitored by TLC with silica gel plates, and the visualization was displayed under UV light (254 nm). ^{1}H NMR, ^{13}C NMR, and ^{19}F NMR spectra were recorded on a Bruker 400 (400, 101, and 376 MHz) or Bruker 500 (500, 126, and 471 MHz) advance spectrometer at room temperature in $CDCl_3$ (solvent signals, δ 7.26 and 77.0 ppm) using TMS as an internal standard. HRMS spectra were measured on an electrospray ionization quadrupole time-of-flight (ESI-Q-TOF) mass spectrometer or Thermo fisher Q-Exactive with apci source. GC-MS spectra were analyzed by Agilent 5977B GC/MSD. *X*-Ray diffractions of single crystal was carried out at 100(2) K on a Bruker D8 VENTURE diffractometer using Mo-Kα radiation (λ = 0.71073 Å). Compound **5p**/**5v** (20.0 mg) was dissolved in ethyl acetate (1.0 mL), and then petroleum ether (2.0 mL) was added to the mixture. The mixture was evaporated slowly at room temperature to obtain crystal **5p**/**5v**.

(B) Typical experimental procedures

(1) General procedure for preparation of alkenes 1

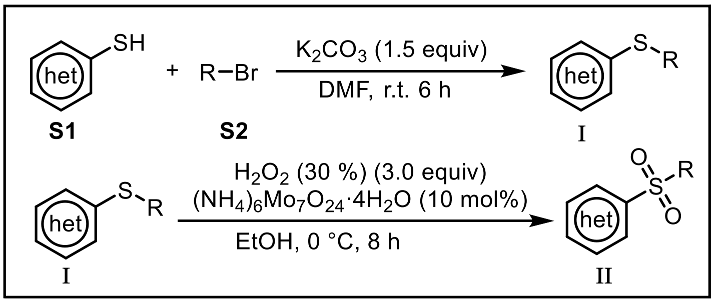
Some of the common alkenes were purchased from commercial sources and used as received. Some of the alkenes, including **1e**, **1f**, **1o, 1p**, **1v** were prepared accordingly following the reported literature.

1.1 General procedure for preparation of alkenes **1e**:[1]



A round-bottomed flask was filled with isoindole-1,3-dione (5.0 mmol, 735.7 mg, 1.0 equiv), potassium carbonate (5.0 mmol, 691.1 mg, 1.0 equiv), potassium iodide (0.05 mmol, 8.3 mg, 1 mol%), 3-buten-1-ylbromide (5.0 mmol, 675.0 mg, 1.0 equiv), and *N*,*N*-dimethylformamide (DMF) (20 mL). The flask was equipped with a reflux condenser, and stirred at 95 °C in oil baths for 3 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. When the reaction was finished, ethyl acetate (EtOAc) (30 mL) was added to the reaction mixture and washed with brine (3 × 20 mL). After extraction, the organic layer was dried over anhydrous Na_2SO_4, filtered and the solvent was evaporated under reduced pressure to afford the desired product.

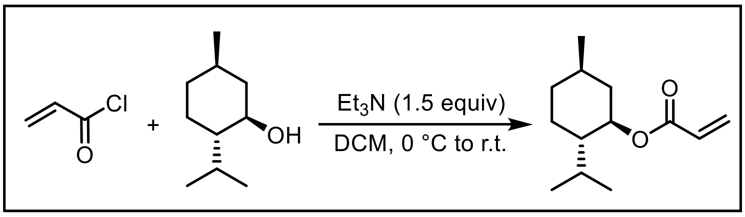
1.2 General procedure for preparation of alkenes **1f** and **1v**:[2]



A round-bottomed flask was filled with compound **S1** (5.0 mmol), compound **S2** (7.5 mmol, 1.5 equiv), K_2CO_3 (7.5 mmol, 1036.6 mg, 1.5 equiv), and 30 mL DMF. Subsequently, the mixture was stirred at room temperature for 6 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. When the reaction was finished, EtOAc (30 mL) was added to the reaction mixture and washed with brine (3 × 20 mL). After extraction, the organic layer was dried over anhydrous Na_2SO_4, filtered and the solvent was evaporated under reduced pressure to afford the desired product **I**.

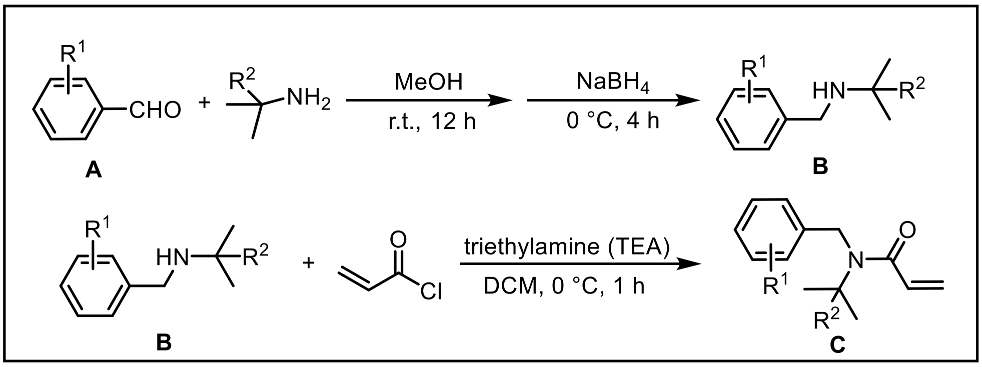
The product **I** was dissolved (5.0 mmol, 1 equiv) in EtOH (30 mL), $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ (0.5 mmol, 617.9 mg, 10 mol%), and 30% H_2O_2 (15.0 mmol) was added at 0 ℃. The resulting mixture was stirred at room temperature for 8 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. When the reaction was finished, EtOAc (30 mL) was added to the reaction mixture and washed with brine (3 × 20 mL). The organic layer was separated, dried over Na_2SO_4, evaporated under reduced pressure. Finally, the residue was purified by column chromatography to afford the pure diselenides **II**.

1.3 General procedure for preparation of alkenes **1o**:[3]



A round-bottomed flask was filled with the *L*-Menthol (3.0 mmol, 468.8 mg, 1.0 equiv), Et_3N (4.5 mmol, 455.4 mg, 1.5 equiv), and dry DCM (10 mL) at 0 °C. Then, acryloyl chloride (3.3 mmol, 298.7 mg, 1.1 equiv) was slowly added to the solution. After the addition was complete, the ice-water cooling bath was removed, and the resulting suspension was stirred vigorously at room temperature overnight. Upon completion, the solvent was evaporated under reduced pressure, and the crude product was purified by column chromatography on silica gel to afford the desired product **1o** as a colorless oil.

1.4 General procedure for preparation of alkenes **1p**:[4]



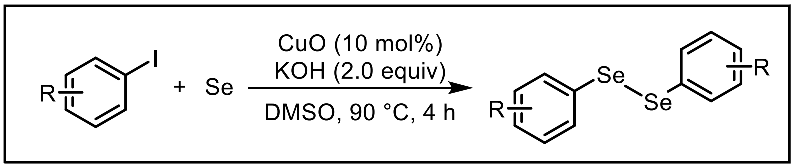
Step 1:

A round-bottomed flask was filled with benzaldehyde derivatives **A** (5.0 mmol, 1.0 equiv), amine (20.0 mmol, 4.0 equiv), and MeOH (50 mL). Subsequently, the mixture was stirred at room temperature for 12 h. After cooling the reaction solution to 0 °C, $NaBH_4$ (7.5 mmol, 283.7 mg, 1.5 equiv) was gradually added in portions, and then this reaction mixture was stirred at room temperature for an additional 4 h. When the reaction was finished, EtOAc (30 mL) was added to the reaction mixture and washed with brine (3 × 20 mL). After extraction, the organic layer was dried over anhydrous Na_2SO_4, filtered, and the solvent was evaporated under reduced pressure to obtain a residue containing **B**, which was directly employed in the next step without further purification.

Step 2:

The crude product **B** was dissolved in dichloromethane (DCM) (100 mL). Then TEA (7.5 mmol, 758.9 mg, 1.5 equiv) was added and stirred at 0 °C for 10 min. Subsequently, acryloyl chloride (5.0 mmol, 452.5 mg, 1.0 equiv) was added dropwise to the reaction solution and continuously stirred for an additional 1 h. Upon completion, the reaction mixture was quenched with saturated aqueous ammonium chloride and extracted with CH_2Cl_2 three times. The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4, filtered, concentrated, and purified by column chromatography on silica gel (15:1 petroleum ether:ethyl acetate) to give the product **C**.

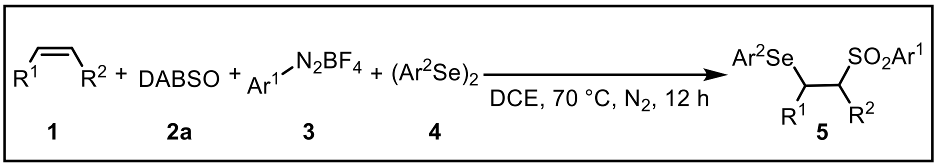
(2) **General procedure for the synthesis of substrates 4.**[5]



A round-bottomed flask was filled with Se metal (2.2 mmol, 173.7 mg, 2.2 equiv), halides (1.0 mmol, 1.0 equiv), copper oxide (0.1 mmol, 8.0 mg, 10.0 mol%), potassium hydroxide (2.0 mmol, 112.2 mg, 2.0 equiv), and dry dimethyl sulfoxide (DMSO, 20 mL). Then the mixture was stirred under nitrogen atmosphere at 90 ℃ in oil baths for 4 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. When the reaction was finished, EtOAc (30 mL) was added to the reaction mixture and washed with brine (3 × 20 mL). The organic layer was separated, dried over Na_2SO_4, evaporated under reduced pressure. Finally, the residue was purified by column chromatography on silica gel to afford the pure diselenides.

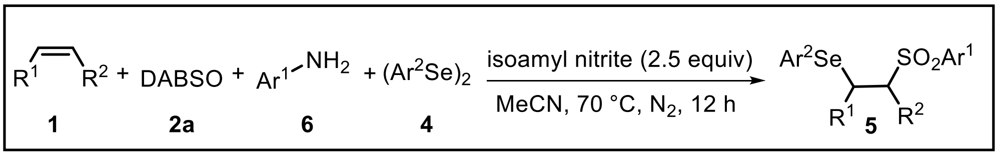
(3) General procedure for synthesis of compounds 5.

3.1 Aryldiazonium tetrafluoroborate as the aryl source.



To the sealed tube were added alkenes (**1**, 0.2 mmol, 1.0 equiv), DABSO (**2a**, 0.4 mmol, 96.2 mg, 2.0 equiv), aryldiazonium tetrafluoroborate (**3**, 2.0 equiv), diselenide (**4**, 1.5 equiv), and dichloroethane (DCE, 2.0 mL). Then the reaction mixture was stirred at 70 ℃ in oil baths under nitrogen atmosphere for 12 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. Upon completion of the reaction, the solution was concentrated under reduced pressure, and the mixture was purified by flash column chromatography on silica gel (hexane/ethyl acetate = 6:1~3:1) to afford the desired products **5**.

3.2 Arylamines as the aryl source.

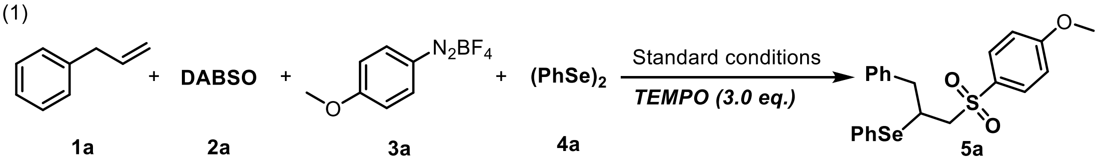


To the sealed tube were added alkenes (**1**, 0.2 mmol, 1.0 equiv), DABSO (**2a**, 0.4 mmol, 96.2 mg, 2.0 equiv), arylamines (**6**, 2.0 equiv), diselenide (**4**, 1.5 equiv), isoamyl nitrite (2.5 equiv), and MeCN (2.0 mL). Then the reaction mixture was stirred at 70 ℃ in oil baths under nitrogen atmosphere for 12 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. Upon completion of the reaction, the solution was concentrated under reduced pressure, and the mixture was purified by flash column chromatography on silica gel (hexane/ethyl acetate = 6:1~3:1) to afford the desired products **5**.

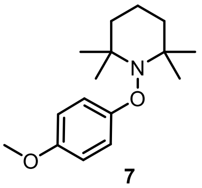
(C) Mechanistic investigation.

(1) Radical scavenging experiments

(1)

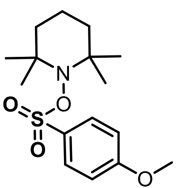


Detected by HRMS



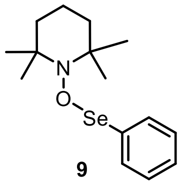
7

$[M+Na]^+$ = 286.1778
found 286.1777



8

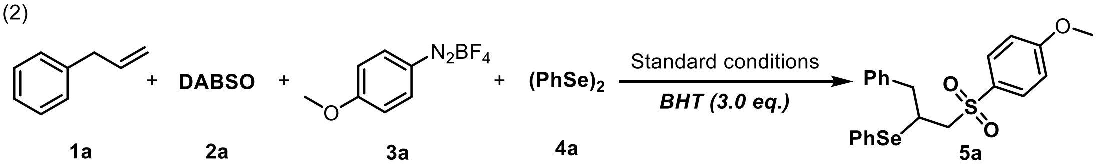
$[M+Na]^+$ = 350.1397
found 350.1384



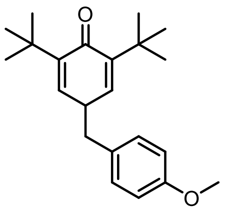
9

$[M+Na]^+$ = 336.0837
found 336.0840

(2)

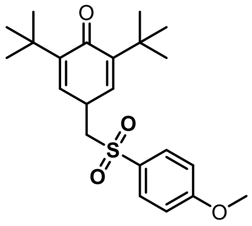


Detected by HRMS



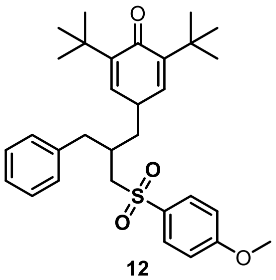
10

$[M+Na]^+$ = 349.2138
found 349.2136



11

$[M+Na]^+$ = 413.1757
found 413.1755

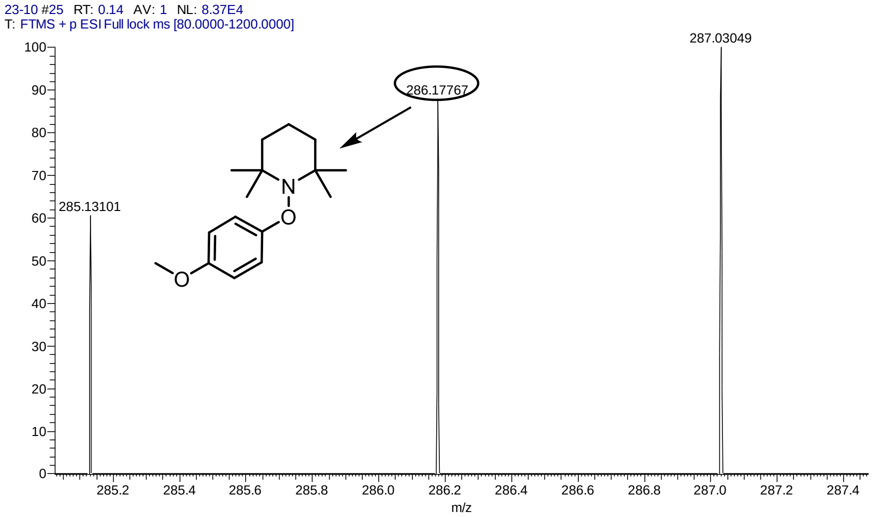


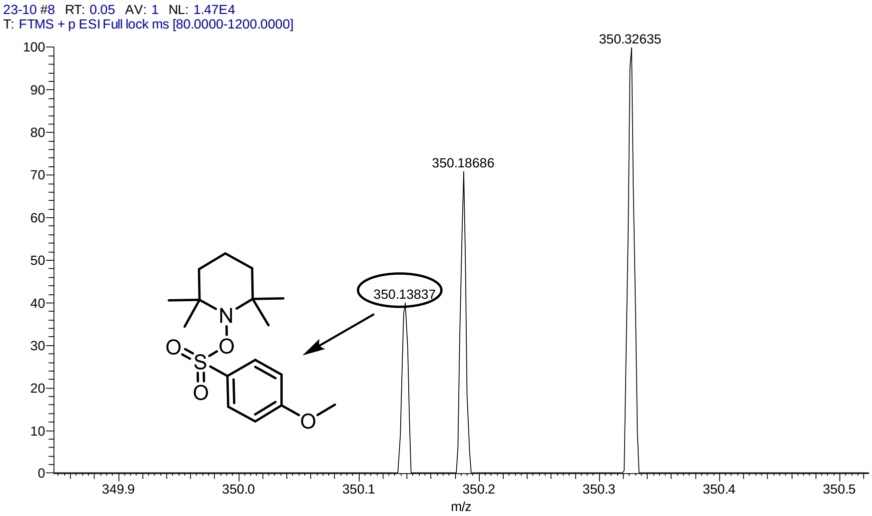
12

$[M+Na]^+$ = 531.2540
found 531.2537

Reaction (1): To the sealed tube were added allylbenzene (**1a**, 23.7 mg, 0.2 mmol), DABSO (**2a**, 96.2 mg, 2.0 equiv), 4-methoxybenzenediazonium tetrafluoroborate (**3a**, 88.8 mg, 2.0 equiv), diphenyl diselenide (**4a,** 93.7 mg, 1.5 equiv), TEMPO (93.8 mg, 3.0 equiv), and DCE (2.0 mL). Then the tube was stirred at 70 °C in oil baths under nitrogen atmosphere for 12 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was completed, radical trapping adduct **7**, **8** and **9** were observed through HRMS analysis of the reaction solution, shown in **Figure S1-S3.**

Reaction (2): To the sealed tube were added allylbenzene (**1a**, 23.7 mg, 0.2 mmol), DABSO (**2a**, 96.2 mg, 2.0 equiv), 4-methoxybenzenediazonium tetrafluoroborate (**3a**, 88.8 mg, 2.0 equiv), diphenyl diselenide (**4a,** 93.7 mg, 1.5 equiv), BHT (132.2 mg, 3.0 equiv), and DCE (2.0 mL). Then the tube was stirred at 70 ℃ in oil baths under nitrogen atmosphere for 12 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was completed, radical trapping adduct **10**, **11** and **12** were observed through HRMS analysis of the reaction solution, shown in **Figure S4-S6.**





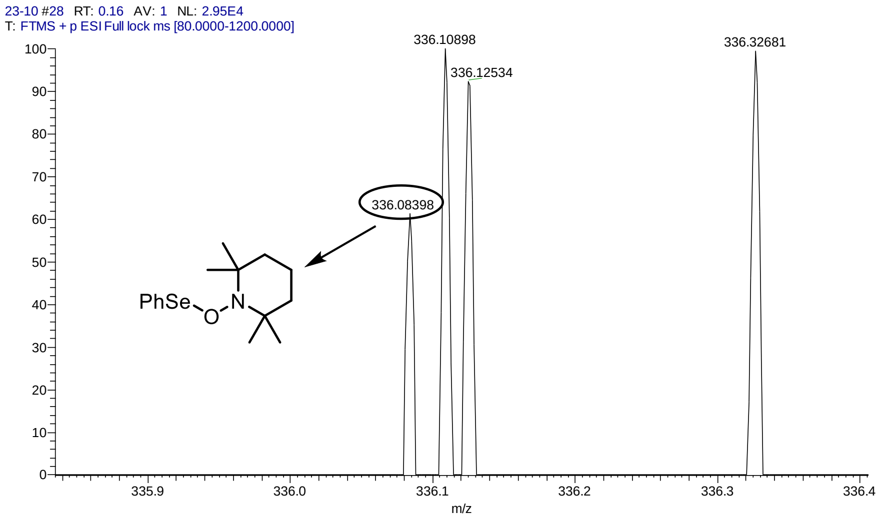
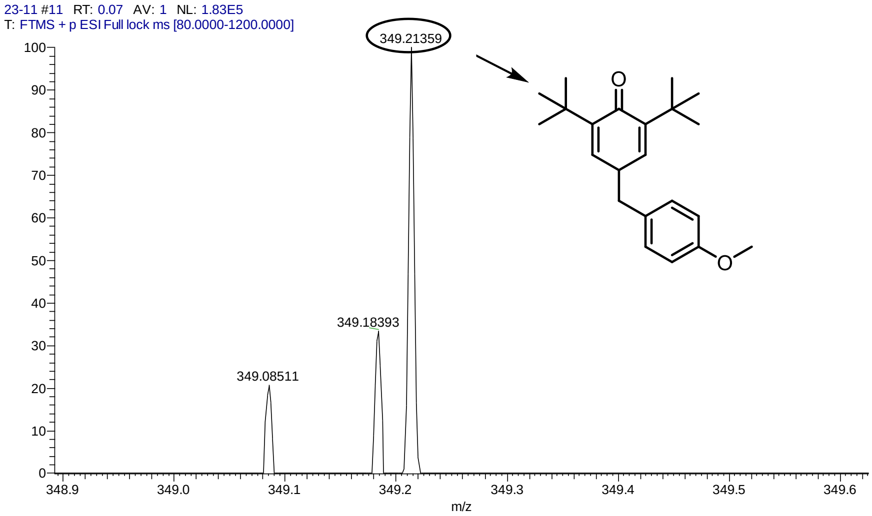
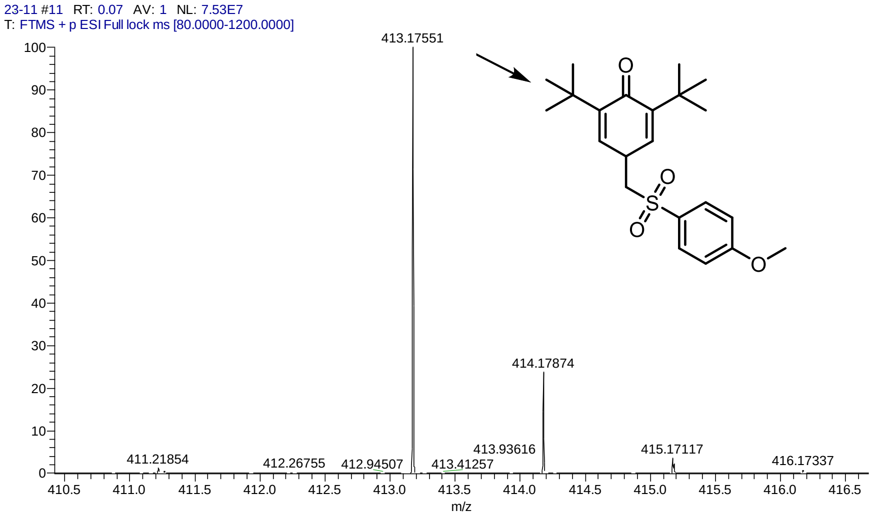


Figure S1-S3. HRMS spectra of **7**, **8** and **9**. The reaction mixture was analyzed by Thermo Scientific LTQ Orbitrap XL.





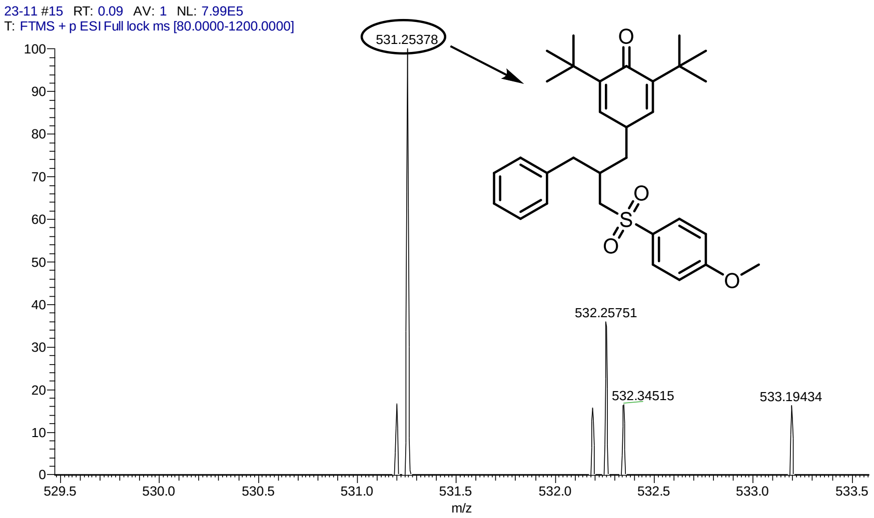
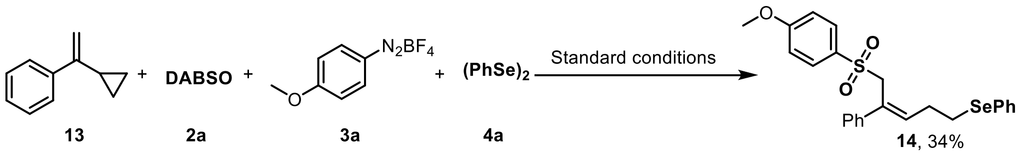


Figure S4-S6. HRMS spectra of **10**, **11** and **12**. The reaction mixture was analyzed by Thermo Scientific LTQ Orbitrap XL.

(2) Radical-clock experiment



To the sealed tube were added (1-cyclopropylvinyl)benzene (**13**, 29.2 mg, 0.2 mmol), DABSO (**2a**, 96.2 mg, 2.0 equiv), 4-methoxybenzenediazonium tetrafluoroborate (**3a**, 88.8 mg, 2.0 equiv), diphenyl diselenide (**4a**, 93.7 mg, 1.5 equiv), and DCE (2.0 mL). Then the tube was stirred at 70 °C in oil baths under nitrogen atmosphere for 12 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was completed, the solution was concentrated under reduced pressure, and the resulting residue was purified by silica gel column chromatography to obtain **14**. Simultaneously, **14** was observed through HRMS analysis of the reaction solution , shown in **Figure S7.**

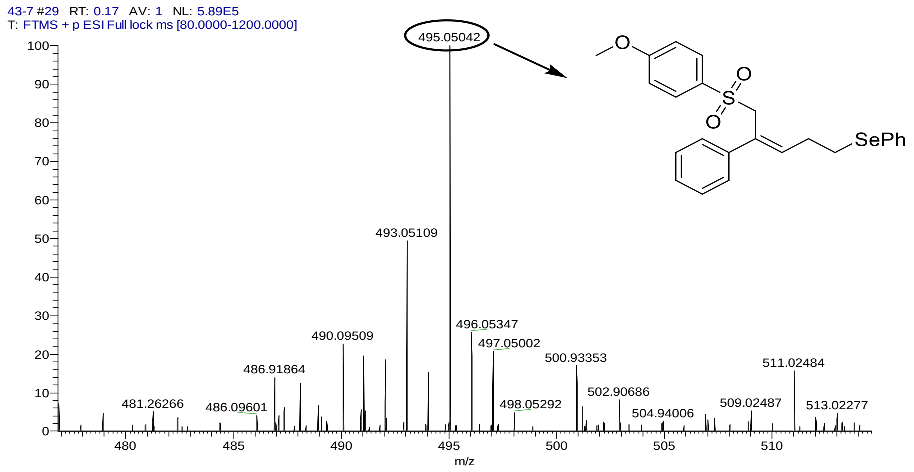
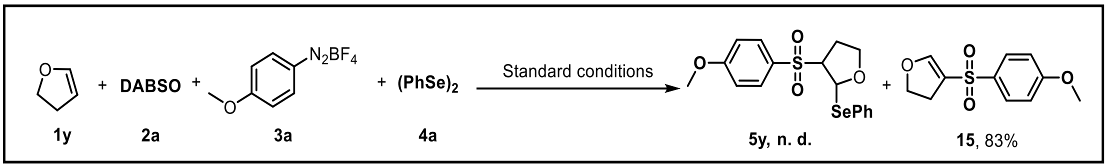


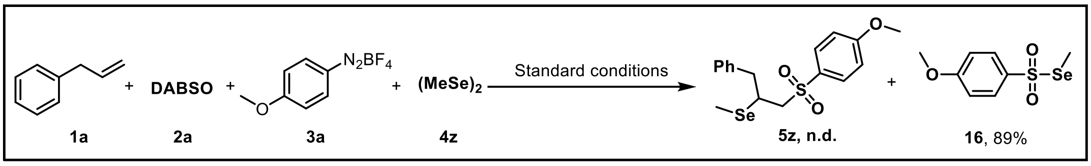
Figure S7. HRMS spectra of **14**. The reaction mixture was analyzed by Thermo Scientific LTQ Orbitrap XL.

（3) Selenosulfonylation of 2,3-dihydrofuran



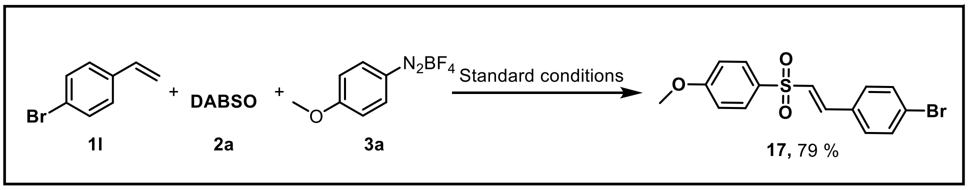
To the sealed tube were added 2,3-dihydrofuran (**1y**, 14.2 mg, 0.2 mmol), DABSO (**2a**, 96.2 mg, 2.0 equiv), 4-methoxybenzenediazonium tetrafluoroborate (**3a**, 88.8 mg, 2.0 equiv), diphenyl diselenide (**4a**, 93.7 mg, 1.5 equiv), and DCE (2.0 mL). Then the tube was stirred at 70 °C in oil baths under nitrogen atmosphere for 12 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the solution was concentrated under reduced pressure, and the resulting residue was purified by silica gel column chromatography to obtain the byprouct **15**.

(4) Dimethyl diselenide as the selenium radical source



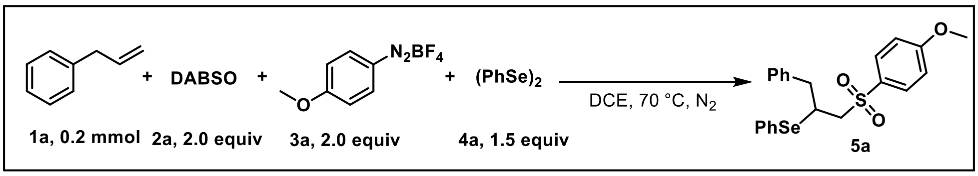
To the sealed tube were added allylbenzene (**1a**, 23.7 mg, 0.2 mmol), DABSO (**2a**, 96.2 mg, 2.0 equiv), 4-methoxybenzenediazonium tetrafluoroborate (**3a**, 88.8 mg, 2.0 equiv), dimethyl diselenide (**4z,** 56.4 mg, 1.5 equiv), and DCE (2.0 mL). Then the tube was stirred at 70 °C in oil baths under nitrogen atmosphere for 12 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the solution was concentrated under reduced pressure, and the resulting residue was purified by silica gel column chromatography to obtain the **16**. This result illustrates the aryl radicals in conjunction with DABSO to form sulfonyl radical exhibited a superior generation rate and reactivity compared to selenyl radical.

(5) Sulfonylation of 4-bromostyrene



To the sealed tube were added 4-bromostyrene (**1l**, 23.7 mg, 0.2 mmol), DABSO (**2a**, 96.2 mg, 2.0 equiv), 4-methoxybenzenediazonium tetrafluoroborate (**3a**, 88.8 mg, 2.0 equiv), and DCE (2.0 mL). Then the tube was stirred at 70 °C in oil baths under nitrogen atmosphere for 12 h, until complete the consumption of starting material as monitored by TLC and/or GC-MS analysis. After the reaction was finished, the solution was concentrated under reduced pressure, then the resulting residue was purified by silica gel column chromatography to obtain the **17**.

(D) Sensitivity assessments



Standard Conditions: n = 0.2 mmol, c = 0.1 M, V = 2.0 mL, in degassed, T = 70 °C.

Stock Solution: n = 2.0 mmol, c = 0.111 M, V = 18.0 mL, **1a**: 236.4 mg, **2a**: 961.2 mg, **3a**: 887.8 mg, **4a**: 936.8 mg, DCE: 18.0 mL.

Stock Solution ‘big scale’ : n = 6.02 mmol, c = 0.1 M, V = 60.2 mL, **1a**: 711.5 mg, **2a**: 2893.2 mg, **3a**: 2672.3 mg, **4a**: 2818.6 mg, DCE: 60.2 mL.

The method is on account of parameter variations in positive and negative direction of reference standard reaction conditions. Only one parameter is consciously changed in each experiment, while maintaining the others at the standard level. The yield of the product is determined by GC-FID analysis. The calibration of the product of the reaction was carried out using the single point calibration technique for gas chromatography (GC) analysis. The preparation of the experiments and the results of the sensitivity assessment are displayed in Tables S1 and S2.

Table S1. Preparation of sensitivity assessment of reaction.

Number	**Experiment**	**Preparation**
1	High c	1.8 mL stock sol.
2	Low c	1.8 mL stock sol. + 0.4 mL DCE
3	High H_2O	1.8 mL stock sol. + 0.2 mL DCE + 10 µL H_2O
4	High O_2	1.8 mL stock sol. + 0.2 mL DCE + air
5	Low O_2	1.8 mL stock sol. + 0.2 mL DCE + degassed
6	Low T	1.8 mL stock sol. + 0.2 mL DCE, T = 60 °C
7	High T	1.8 mL stock sol. + 0.2 mL DCE, T = 90 °C
8	Control	1.8 mL stock sol. + 0.2 mL DCE
9	Big Scale	40.0 mL stock solution ‘big scale’

Table S2. Results of sensitivity assessment of reaction.

Number	Experiment	Yield / %	Deviation / %
1	High *c*	82	+5
2	Low *c*	73	-7
3	High H_2O	65	-17
4	High O_2	14	-82
5	Low O_2	78	0
6	High *T*	59	-25
7	Low *T*	73	-7
8	Control	78	0
9	Big Scale	71	-9

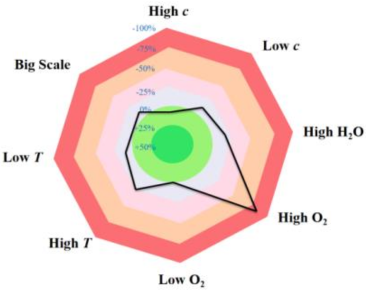
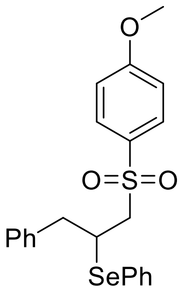
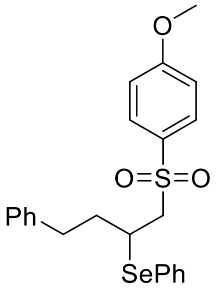


Figure 8. Radar diagram of the sensitivity for this reaction.

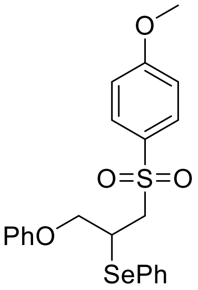
(E) Analytical data



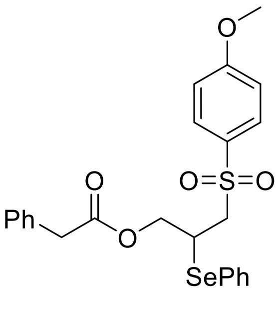
(1-((4-Methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)(phenyl)selane (5a). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1 v/v). Yellow oil (69.6 mg, 78% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.69-7.66 (m, 2H), 7.31 (t, J = 7.5 Hz, 2H), 7.28-7.24 (m, 6H), 7.17 (t, J = 7.5 Hz, 2H), 6.95-6.92 (m ,2H), 3.87 (s, 3H), 3.70-3.64 (m, 1H), 3.49-3.43 (m, 2H), 3.36-3.33 (m, 1H), 3.08-3.03 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 163.7, 137.8, 134.7, 130.4, 130.0, 129.4, 129.2, 128.4, 128.0, 127.9, 126.9, 114.4, 60.4, 55.6, 39.7, 38.1; HRMS *m/z* (ESI) calcd for $C_{22}H_{23}O_3SSe$ ([M+H]$^+$) 447.0528, found 447.0524.



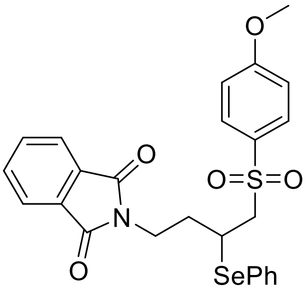
(1-((4-Methoxyphenyl)sulfonyl)-4-phenylbutan-2-yl)(phenyl) selane (5b). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1 v/v). Yellow oil (68.1 mg, 74% yield); ^{1}H NMR (400 MHz, CDCl$_3$) δ: 7.30 (d, J = 8.8 Hz, 2H), 7.23 (d, J = 7.2 Hz, 2H), 7.19-7.15 (m, 6H), 7.07 (t, J = 7.6 Hz, 2H), 6.72 (d, J = 8.8 Hz, 2H), 3.77 (s, 3H), 3.31 (t, J = 5.2 Hz, 2H), 3.17-3.11 (m, 1H), 2.89-2.83 (m, 1H), 2.75-2.67 (m, 1H), 2.51-2.42 (m, 1H), 1.88-1.78 (m, 1H); ^{13}C NMR (101 MHz, CDCl$_3$) δ: 163.5, 140.7, 134.8, 130.0, 129.8, 129.2, 128.8, 128.4, 128.0, 127.3, 126.0, 114.3, 61.9, 55.6, 36.5, 34.7, 33.3; HRMS *m/z* (ESI) calcd for $C_{23}H_{25}O_3SSe$ ($[M+H]^+$) 461.0684, found 461.0686.



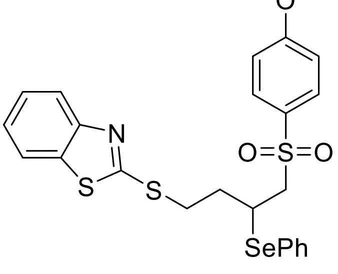
(1-((4-Methoxyphenyl)sulfonyl)-3-phenoxypropan-2-yl)(phenyl) selane (5c). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1 v/v). Yellow solid (59.1 mg, 64% yield); ^{1}H NMR (400 MHz, CDCl$_3$) δ: 7.64 (d, J = 8.4 Hz, 2H), 7.38 (d, J = 7.6 Hz, 2H), 7.22-7.15 (m, 5H), 6.89 (t, J = 7.2 Hz, 1H), 6.83 (d, J = 8.4 Hz, 2H), 6.75 (d, J = 8.4 Hz, 2H), 4.35-4.32 (m, 1H), 4.22-4.19 (m, 1H), 3.77 (s, 3H), 3.73 (d, J = 12.8 Hz, 2H), 3.36 (d, J = 12.0 Hz, 1H); ^{13}C NMR (101 MHz, CDCl$_3$) δ: 163.8, 158.0, 134.9, 130.5, 130.0, 129.4 (2), 128.3, 127.5, 121.3, 114.7, 114.5, 68.6, 58.1, 55.6, 35.9; HRMS *m/z* (ESI) calcd for $C_{22}H_{23}O_4SSe$ ($[M+H]^+$) 463.0477, found 463.0475.



3-((4-Methoxyphenyl)sulfonyl)-2-(phenylselanyl)propyl 2-phenylacetate (5d). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1 v/v). Yellow solid (69.6 mg, 69% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.61 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 7.6 Hz, 2H), 7.25-7.14 (m, 8H), 6.86 (d, J = 8.0 Hz, 2H), 4.35 (d, J = 3.2 Hz, 2H), 3.78 (s, 3H), 3.58 (t, J = 4.4 Hz, 1H), 3.54 (s, 2H), 3.41-3.35 (m, 1H), 3.27-3.22 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 170.8, 163.8, 135.1, 133.5, 130.2, 130.1, 129.3 (2), 128.5, 127.1, 126.7, 114.5, 65.2, 58.1, 55.6, 41.0, 34.7; HRMS *m/z* (ESI) calcd for $C_{24}H_{25}O_5SSe$ ([M+H]$^+$) 505.0582, found 505.0583.

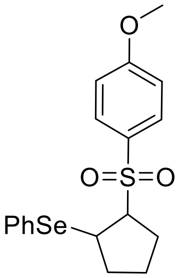


2-(4-((4-Methoxyphenyl)sulfonyl)-3-(phenylselanyl)butyl)isoindoline-1,3-dione (5e). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (3:1 v/v). Yellow oil (75.1 mg, 71% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.88-7.86 (m, 2H), 7.74-7.73 (m, 2H), 7.61-7.59 (m, 2H), 7.45-7.43 (m, 2H), 7.31 (t, J = 7.5 Hz, 1H), 7.23 (t, J = 7.5 Hz, 2H), 6.88-6.86 (m, 2H), 3.98-3.89 (m, 2H), 3.86 (s, 3H), 3.48-3.45 (m, 1H), 3.39-3.34 (m, 1H), 3.30-3.26 (m, 1H), 2.54-2.49 (m, 1H), 1.97-1.92 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 168.2, 163.6, 135.7, 133.9, 132.1, 130.1, 130.0, 129.3, 128.5, 126.4, 123.2, 114.4, 61.7, 55.6, 36.5, 34.6, 32.0; HRMS *m/z* (ESI) calcd for $C_{25}H_{24}NO_5SSe$ ([M+H]$^+$) 530.0535, found 530.0539.

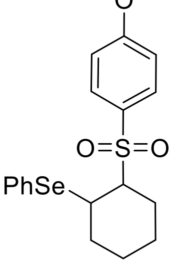


2-((4-((4-Methoxyphenyl)sulfonyl)-3-(phenylselanyl)butyl)thio)benzo[*d*]thiazole (5f). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (3:1, v/v). Yellow oil (80.2

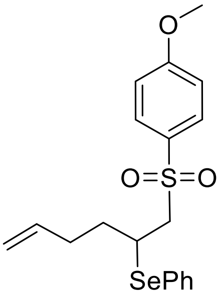
mg, 73% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.89-7.87 (m, 1H), 7.78-7.76 (m, 1H), 7.65-7.61 (m, 2H), 7.44-7.38 (m, 3H), 7.32-7.27 (m, 2H), 7.21-7.18 (m, 2H), 6.85-6.81 (m, 2H), 3.80 (s, 3H), 3.73-3.66 (m, 1H), 3.55-3.38 (m, 4H), 2.75-2.66 (m, 1H), 2.10-2.00 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 166.1, 163.6, 153.1, 135.3, 135.1, 130.1, 129.9, 129.3, 128.4, 126.5, 126.0, 124.2, 121.5, 120.9, 114.4, 61.8, 55.6, 36.2, 32.9, 31.5; HRMS *m/z* (ESI) calcd for $C_{24}H_{24}NO_3S_3Se$ ($[M+H]^+$) 550.0078, found 550.0076.



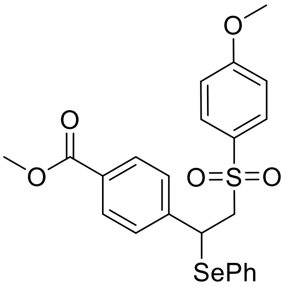
(2-((4-Methoxyphenyl)sulfonyl)cyclopentyl)(phenyl)selane (5g). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1 v/v). Yellow oil (52.3 mg, 66% yield, d.r. > 20:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.60 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 7.2 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 2H), 3.87 (t, *J* = 3.6 Hz, 1H), 3.80 (s, 3H), 3.43-3.39 (m, 1H), 2.22-2.16 (m, 2H), 2.07-2.02 (m, 1H), 1.85-1.72 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.6, 134.7, 130.5, 129.5, 129.1, 128.6, 127.9, 114.3, 70.7, 55.6, 41.4, 33.8, 25.9, 24.8; HRMS *m/z* (ESI) calcd for $C_{18}H_{21}O_3SSe$ ($[M+H]^+$) 397.0371, found 397.0375.



(2-((4-Methoxyphenyl)sulfonyl)cyclohexyl)(phenyl)selane (5h). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1, v/v). Yellow oil (51.7 mg, 63% yield, d.r. > 20:1); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.65-7.63 (m, 2H), 7.29-7.27 (m, 2H), 7.25-7.23 (m, 1H), 7.16 (t, *J* = 7.5 Hz, 2H), 6.94-6.91 (m, 2H), 3.88 (s, 3H), 3.87 (d, *J* = 3.0 Hz, 1H), 3.12-3.09 (m, 1H), 2.47-2.42 (m, 1H), 2.22-2.16 (m, 1H), 2.13-2.09 (m, 1H), 1.95-1.85 (m, 2H), 1.61-1.54 (m, 3H); ^{13}C NMR (126 MHz, CDCl$_3$) *δ*: 163.5, 134.0, 130.5, 129.4, 129.2, 128.9, 127.6, 114.3, 64.1, 55.6, 38.8, 28.4, 22.4, 21.6, 21.4; HRMS *m/z* (ESI) calcd for $C_{19}H_{23}O_3SSe$ ([M+H]$^+$) 411.0528, found 411.0522.

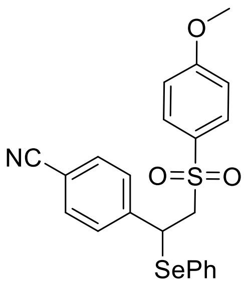


(1-((4-Methoxyphenyl)sulfonyl)hex-5-en-2-yl)(phenyl)selane (5i). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1 v/v). Yellow oil (50.0 mg, 61% yield); ^{1}H NMR (500 MHz, CDCl$_3$) *δ*: 7.68-7.65 (m, 2H), 7.35-7.33 (m, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.20 (t, *J* = 7.5 Hz, 2H), 6.96-6.93 (m, 2H), 5.82-5.74 (m, 1H), 5.09-5.01 (m, 2H), 3.89 (s, 3H), 3.46-3.37 (m, 3H), 2.39-2.33 (m, 1H), 2.27-2.20 (m, 2H), 1.72-1.67 (m, 1H); ^{13}C NMR (126 MHz, CDCl$_3$) *δ*: 163.7, 137.1, 135.0, 130.6, 130.1, 129.3, 128.1, 127.4, 115.8, 114.4, 62.0, 55.7, 36.8, 32.6, 31.6; HRMS *m/z* (ESI) calcd for $C_{19}H_{23}O_3SSe$ ([M+H]$^+$) 411.0528, found 411.0522.

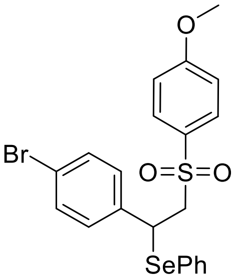


Methyl-4-(2-((4-methoxyphenyl)sulfonyl)-1-(phenylselanyl)ethyl)benzoate (5j). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (3:1, v/v). White solid (74.5 mg, 76% yield); ^{1}H NMR (400 MHz, CDCl$_3$) *δ*: 7.66 (d, *J* = 8.0 Hz, 2H), 7.35-7.29 (m, 4H), 7.24 (d, *J* = 6.8 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 2H), 6.95 (d, *J* = 8.0 Hz, 2H), 6.63 (d, *J* = 8.8 Hz, 2H), 4.63-4.59 (m, 1H), 3.91-3.84 (m, 1H), 3.81 (s, 3H), 3.69 (s, 3H), 3.62-3.58 (m, 1H); ^{13}C NMR

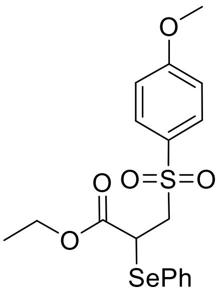
(101 MHz, $CDCl_3$) δ: 166.4, 163.5, 143.1, 135.7, 130.2, 130.0, 129.5, 129.4, 129.0 (2), 127.6 (2), 114.1, 60.8, 55.5, 52.1, 39.4; HRMS *m/z* (ESI) calcd for $C_{23}H_{23}O_5SSe$ ($[M+H]^+$) 491.0426, found 491.0427.



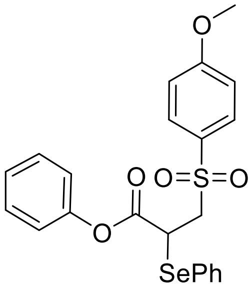
4-(2-((4-Methoxyphenyl)sulfonyl)-1-(phenylselanyl)ethyl)benzonitrile (5k). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (3:1, v/v). Pale yellow solid (65.8 mg, 72% yield); ^1H NMR (500 MHz, $CDCl_3$) δ: 7.45-7.42 (m, 2H), 7.37-7.33 (m, 5H), 7.27-7.24 (m, 2H), 7.06 (d, J = 8.0 Hz, 2H), 6.78-6.76 (m, 2H), 4.68-4.65 (m, 1H), 3.91-3.87 (m, 1H), 3.83 (s, 3H), 3.70-3.66 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 163.7, 143.7, 135.9, 131.9, 130.1, 130.0, 129.4, 129.2, 128.3, 127.0, 118.3, 114.2, 111.0, 60.5, 55.7, 39.1; HRMS *m/z* (ESI) calcd for $C_{22}H_{20}NO_3SSe$ ($[M+H]^+$) 458.0324, found 458.0323.



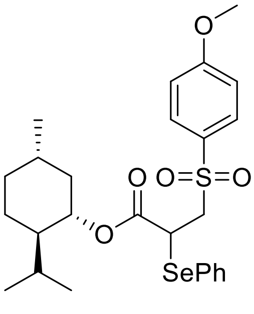
(1-(4-Bromophenyl)-2-((4-methoxyphenyl)sulfonyl)ethyl)(phenyl)selane (5l). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1, v/v). Pale yellow solid (71.3 mg, 70% yield); ^1H NMR (500 MHz, $CDCl_3$) δ: 7.41-7.39 (m, 2H), 7.37-7.35 (m, 2H), 7.33-7.31 (m, 1H), 7.27-7.24 (m, 2H), 7.17-7.16 (m, 2H), 6.83 (d, J = 8.5 Hz, 2H), 6.72 (d, J = 9.0 Hz, 2H), 4.65-4.62 (m, 1H), 3.93-3.87 (m, 1H), 3.82 (s, 3H), 3.64-3.61 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 163.4, 136.8, 135.6, 131.3, 130.3, 129.9, 129.4, 129.2, 128.9, 127.8, 121.2, 114.0, 61.0, 55.6, 39.2; HRMS *m/z* (ESI) calcd for $C_{21}H_{20}BrO_3SSe$ ($[M+H]^+$) 510.9476, found 510.9473.



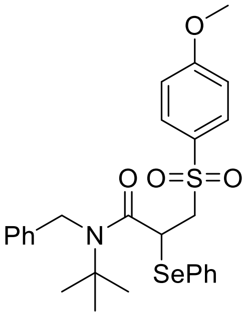
Ethyl-3-((4-methoxyphenyl)sulfonyl)-2-(phenylselanyl)propanoate (5m). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1, v/v). Yellow oil (54.8 mg, 64% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.68 (d, J = 8.8 Hz, 2H), 7.43 (d, J = 7.2 Hz, 2H), 7.30 (t, J = 7.6 Hz, 1H), 7.21 (t, J = 7.6 Hz, 2H), 6.90 (d, J = 8.4 Hz, 2H), 3.89 (t, J = 7.2 Hz, 2H), 3.85 (s, 1H), 3.79 (s, 3H), 3.74 (d, J = 11.6 Hz, 1H), 3.37-3.33 (m, 1H), 1.08 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 170.1, 163.9, 136.1, 130.5, 129.9, 129.4, 129.3, 125.8, 114.4, 61.6, 58.6, 55.7, 34.4, 13.8; HRMS m/z (ESI) calcd for $C_{18}H_{21}O_5SSe$ ([M+H]$^+$) 429.0269, found 429.0265.



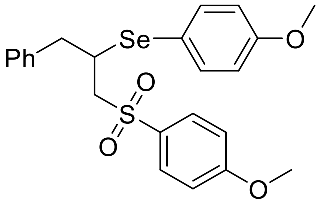
Phenyl-3-((4-methoxyphenyl)sulfonyl)-2-(phenylselanyl)propanoate (5n). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1, v/v). Yellow oil (63.8 mg, 67% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.82-7.79 (m, 2H), 7.57 (d, J = 7.0 Hz, 2H), 7.40-7.28 (m, 5H), 7.21 (t, J = 7.5 Hz, 1H), 6.98 (t, J = 8.5 Hz, 4H), 4.15-4.12 (m, 1H), 3.87 (t, J = 7.0 Hz, 1H), 3.83 (s, 3H), 3.54-3.51 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 169.0, 164.0, 150.4, 136.3, 130.4, 129.8, 129.6, 129.4, 129.2, 126.0, 125.4, 121.1, 114.6, 58.6, 55.6, 34.1; HRMS m/z (ESI) calcd for $C_{22}H_{21}O_5SSe$ ([M+H]$^+$) 477.0269, found 477.0266.



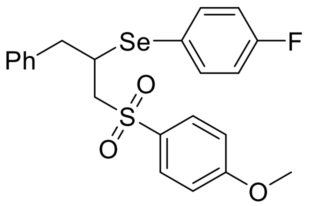
(1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl 3-((4-methoxyphenyl)sulfonyl)-2-(phenylselanyl)propanoate (5o). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1, v/v). Yellow oil (78.7 mg, 73% yield, d.r. = 1:1); ^{1}H NMR (400 MHz, CDCl$_3$) δ: 7.76-7.73 (m, 2H), 7.49-7.46 (m, 2H), 7.37-7.33 (m, 1H), 7.30-7.25 (m, 2H), 6.99-6.95 (m, 2H), 4.69-4.59 (m, 1H), 3.93-3.89 (m, 1.5H), 3.86 (d, *J* = 2.4 Hz, 3H), 3.83 (t, *J* = 2.4 Hz, 0.5H), 3.44-3.38 (m, 1H), 2.10-1.92 (m, 2H), 1.87-1.84 (m, 1H), 1.67 (d, *J* = 11.6 Hz, 2H), 1.45-1.35 (m, 2H), 1.09-0.95 (m, 2H), 0.92 (d, *J* = 6.0 Hz, 3H), 0.89 (d, *J* = 5.2 Hz, 3H), 0.78 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR(101 MHz, CDCl$_3$) δ: 170.1, 169.7, 163.8 (2), 135.6, 135.5, 130.3, 130.2 (2), 129.9, 129.2 (3), 129.1, 126.2, 125.8, 114.4 (2), 75.8 (2), 58.6, 58.3, 55.6, 46.9, 46.7, 40.4, 39.9, 34.7, 34.0, 33.9, 31.2 (2), 25.7, 25.4, 23.1, 22.9, 21.9 (2), 20.8, 20.7, 16.1, 15.9; HRMS *m/z* (ESI) calcd for $C_{26}H_{35}O_5SSe$ ([M+H]$^+$) 539.1365, found 539.1361.



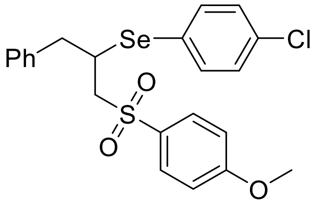
***N*-Benzyl-*N*-(*tert*-butyl)-3-((4-methoxyphenyl)sulfonyl)-2-(phenylselanyl)propanami (5p)**. The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1, v/v). Yellow soild (65.4 mg, 60% yield); ^{1}H NMR (500 MHz, CDCl$_3$) δ: 7.59 (d, *J* = 8.5 Hz, 2H), 7.42-7.39 (m, 4H), 7.32-7.29 (m, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.20 (d, *J* = 7.5 Hz, 2H), 7.15 (t, *J* = 8.0 Hz, 2H), 6.87-6.86 (m, 2H), 4.92-4.81 (m, 2H), 4.36-4.31 (m, 1H), 4.02-3.99 (m, 1H), 3.84 (s, 3H), 3.31-3.28 (m, 1H), 1.45 (s, 9H); ^{13}C NMR (126 MHz, CDCl$_3$) δ: 170.1, 163.6, 138.9, 135.1, 131.1, 130.0, 129.3, 128.8 (2), 127.1, 126.9, 126.1, 114.3, 60.2, 58.6, 55.6, 49.3, 36.5, 28.3; HRMS *m/z* (ESI) calcd for $C_{27}H_{32}NO_4SSe$ ([M+H]$^+$) 546.1212, found 546.1218.



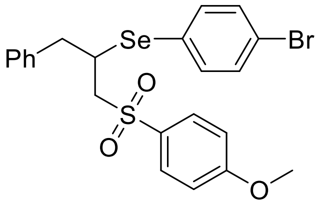
(4-Methoxyphenyl)(1-((4-methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)selane (5q). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1, v/v). Yellow oil (53.3 mg, 56% yield); ^{1}H NMR (400 MHz, CDCl$_3$) δ: 7.58 (d, J = 8.8 Hz, 2H), 7.25 (t, J = 7.2 Hz, 2H), 7.17 (t, J = 7.6 Hz, 5H), 6.86 (d, J = 8.4 Hz, 2H), 6.64 (d, J = 8.4 Hz, 2H), 3.82 (s, 3H), 3.73 (s, 3H), 3.45-3.39 (m, 1H), 3.34 (t, J = 12.0 Hz, 2H), 3.29-3.25 (m, 1H), 2.96-2.91 (m, 1H); ^{13}C NMR (101 MHz, CDCl$_3$) δ: 163.7, 160.0, 138.1, 137.4, 130.5, 130.0, 129.4, 128.4, 126.8, 117.6, 114.9, 114.4, 60.4, 55.7, 55.2, 39.8, 38.5; HRMS *m/z* (ESI) calcd for $C_{23}H_{25}O_4SSe$ ([M+H]$^+$) 477.0633, found 477.0638.



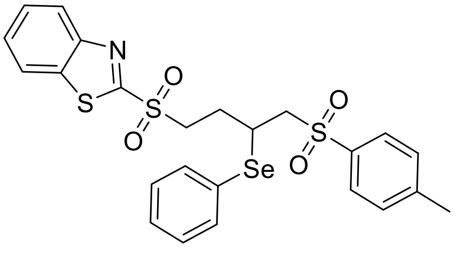
(4-Fluorophenyl)(1-((4-methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)selane (5r). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1, v/v). Yellow oil (75.2 mg, 81% yield); ^{1}H NMR (500 MHz, CDCl$_3$) δ: 7.68-7.65 (m, 2H), 7.32 (t, J = 7.0 Hz, 2H), 7.27-7.23 (m, 5H), 6.96-6.93 (m, 2H), 6.87 (t, J = 8.5 Hz, 2H), 3.89 (s, 3H), 3.59-3.55 (m, 1H), 3.48-3.43 (m, 2H), 3.33-3.29 (m, 1H), 3.05-3.01 (m, 1H); ^{13}C NMR (126 MHz, CDCl$_3$) δ: 163.8 (d, $J_{C\text{-}F}$ = 7.8 Hz), 161.8, 137.8, 137.3 (d, $J_{C\text{-}F}$ = 8.2 Hz), 130.3, 130.0, 129.4, 128.5, 126.9, 122.4 (d, $J_{C\text{-}F}$ = 3.4 Hz), 116.4 (d, $J_{C\text{-}F}$ = 21.5 Hz), 114.4, 60.4, 55.7, 39.9, 39.0; ^{19}F NMR (471 MHz, CDCl$_3$) δ: -112.6; HRMS *m/z* (ESI) calcd for $C_{22}H_{22}FO_3SSe$ ([M+H]$^+$) 465.0433, found 465.0430.



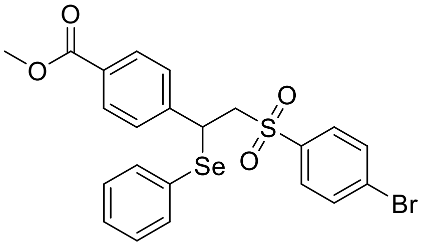
(4-Chlorophenyl)(1-((4-methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)selane (5s). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1, v/v). Yellow oil (79.7 mg, 83% yield); ^{1}H NMR (400 MHz, CDCl$_3$) δ: 7.60 (d, J = 8.4 Hz, 2H), 7.25-7.16 (m, 5H), 7.11 (d, J = 8.0 Hz, 2H), 7.05 (d, J = 8.0 Hz, 2H), 6.88 (d, J = 8.4 Hz, 2H), 3.82 (s, 3H), 3.57-3.52 (m, 1H), 3.43-3.37 (m, 2H), 3.25-3.21 (m, 1H), 3.00-2.94 (m, 1H); ^{13}C NMR (101 MHz, CDCl$_3$) δ: 163.8, 137.7, 136.1, 134.4, 130.3, 130.0, 129.4(2), 128.5, 127.0, 126.1, 114.5, 60.4, 55.7, 39.9, 38.9; HRMS m/z (ESI) calcd for $C_{22}H_{22}ClO_3SSe$ ([M+H]$^+$) 481.0138, found 481.0134.



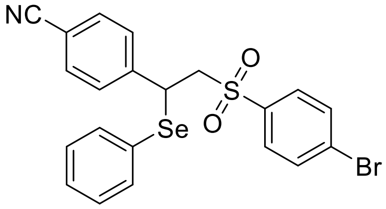
(4-Bromophenyl)(1-((4-methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)selane (5t). The product was purified by silica gel columnchromatography with petroleum ether/ethyl acetate (5:1, v/v). Yellow oil (83.8 mg, 80% yield); ^{1}H NMR (500 MHz, CDCl$_3$) δ: 7.69-7.66 (m, 2H), 7.32 (t, J = 7.5 Hz, 2H), 7.27-7.24 (m, 5H), 7.12-7.09 (m, 2H), 6.97-6.94 (m, 2H), 3.89 (s, 3H), 3.65-3.60 (m, 1H), 3.50-3.45 (m, 2H), 3.31-3.28 (m, 1H), 3.07-3.02 (m, 1H); ^{13}C NMR (126 MHz, CDCl$_3$) δ: 163.8, 137.6, 136.2, 132.3, 130.2, 130.0, 129.3, 128.5, 127.0, 126.8, 122.5, 114.4, 60.4, 55.7, 39.8, 38.8; HRMS m/z (ESI) calcd for $C_{22}H_{22}BrO_3SSe$ ([M+H]$^+$) 524.9633, found 524.9639.



2-((3-(Phenylselanyl)-4-tosylbutyl)sulfonyl)benzo[*d*]thiazole (5v). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1, v/v). Yellow solid (74.6 mg, 66% yield); ^{1}H NMR (500 MHz, CDCl$_3$) δ: 8.25 (d, J = 8.0 Hz, 1H), 8.03 (d, J = 8.0 Hz, 1H), 7.68-7.60 (m, 4H), 7.34-7.26 (m, 5H), 7.13 (t, J = 8.0 Hz, 2H), 3.97-3.91 (m, 1H), 3.79-3.73 (m, 1H), 3.55-3.47 (m, 2H), 3.26-3.21 (m, 1H), 2.74-2.67 (m, 1H), 2.44 (s, 3H), 2.10-2.02 (m, 1H); ^{13}C NMR (126 MHz, CDCl$_3$) δ: 165.3, 152.6, 145.1, 136.8, 135.8, 135.7, 130.0, 129.4, 128.9, 128.1, 128.0, 127.7, 125.5, 122.4, 61.5, 53.0, 35.1, 26.6, 21.6; HRMS *m/z* (ESI) calcd for $C_{24}H_{24}NO_4S_3Se$ ([M+H]$^+$) 566.0027, found 566.0023.

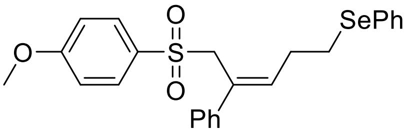


Methyl-4-(2-((4-bromophenyl)sulfonyl)-1-(phenylselanyl)ethyl)benzoate (5w). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1, v/v). White solid (74.2 mg, 69% yield); ^{1}H NMR (400 MHz, CDCl$_3$) δ: 7.67 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.0 Hz, 4H), 7.25 (d, J = 7.6 Hz, 3H), 7.17 (t, J = 7.2 Hz, 2H), 6.95 (d, J = 8.0 Hz, 2H), 4.59 (d, J = 11.2 Hz, 1H), 3.91 (t, J = 13.2 Hz, 1H), 3.83 (s, 3H), 3.61 (d, J = 14.8 Hz, 1H); ^{13}C NMR (101 MHz, CDCl$_3$) δ: 166.2, 142.7, 137.8, 135.7, 132.1, 129.6, 129.4, 129.3(2), 129.1, 128.8, 127.5, 127.4, 60.8, 52.1, 39.1; HRMS *m/z* (ESI) calcd for $C_{22}H_{20}BrO_4SSe$ ([M+H]$^+$) 538.9425, found 538.9421.

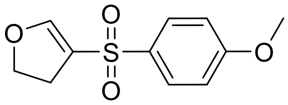


4-(2-((4-Bromophenyl)sulfonyl)-1-(phenylselanyl)ethyl)benzonitrile (5x). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1, v/v). White solid (65.6 mg, 65% yield); ^{1}H NMR (400 MHz,

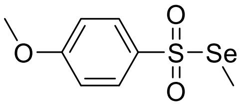
$CDCl_3$) δ: 7.41 (d, J = 8.8 Hz, 2H), 7.33 (t, J = 4.0 Hz, 4H), 7.27 (t, J = 6.0 Hz, 3H), 7.18 (d, J = 7.6 Hz, 2H), 7.00 (t, J = 8.4 Hz, 2H), 4.58-4.55 (m, 1H), 3.89-3.83 (m, 1H), 3.63-3.58 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 143.3, 137.6, 135.9, 132.7, 132.3, 132.0, 129.5, 129.3, 128.2, 126.9, 118.1, 111.1, 60.3, 38.7; HRMS *m/z* (ESI) calcd for $C_{21}H_{17}BrNO_2SSe$ ([M+H]$^+$) 505.9323, found 505.9327.



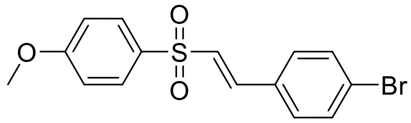
(5-((4-Methoxyphenyl)sulfonyl)-4-phenylpent-3-en-1-yl)(phenyl)selane (14). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1, v/v). Yellow oil (32.1 mg, 34% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.51 (d, J = 8.4 Hz, 2H), 7.45-7.41 (m, 2H), 7.20 (t, J = 4.0 Hz, 3H), 7.13-7.07 (m, 5H), 6.73 (d, J = 8.4 Hz, 2H), 5.93 (t, J = 7.6 Hz, 1H), 4.17 (s, 2H), 3.74 (s, 3H), 2.85 (t, J = 7.2 Hz, 2H), 2.44-2.39 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.6, 140.6, 136.3, 132.8, 130.5, 130.4, 129.7, 129.4, 129.1, 128.2, 127.3, 127.0, 126.4, 114.0, 57.9, 55.6, 29.9, 26.5; HRMS *m/z* (ESI) calcd for $C_{24}H_{24}NaO_3SSe$ ([M+Na]$^+$) 495.0504, found 495.0508.



4-((4-Methoxyphenyl)sulfonyl)-2,3-dihydrofuran (15). The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (3:1, v/v). Yellow oil (39.8 mg, 83% yield); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.83-7.81 (m, 2H), 7.18 (t, J = 2.0 Hz, 1H), 7.01-6.99 (m, 2H), 4.60 (d, J = 10.0 Hz, 2H), 3.88 (s, 3H), 2.81-2.77 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 163.3, 155.9, 132.3, 129.4, 117.9, 114.3, 73.8, 55.6, 28.0; HRMS *m/z* (ESI) calcd for $C_{11}H_{13}O_4S$ ($[M+H]^+$) 241.0529, found 241.0527.



***Se*-Methyl 4-methoxybenzenesulfonoselenoate (16)**. The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (5:1, v/v). Yellow oil (47.3 mg, 89% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75-7.73 (m, 2H), 6.93-6.91 (m, 2H), 3.82 (s, 3H), 2.53 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.5, 138.1, 128.8, 114.1, 55.7, 12.6; HRMS *m/z* (ESI) calcd for $C_8H_{11}O_3SSe$ ($[M+H]^+$) 266.9589, found 266.9586.



(*E*)-1-Bromo-4-(2-((4-methoxyphenyl)sulfonyl)vinyl)benzene (17).[6] The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (4:1, v/v). White solid (55.6 mg, 22% yield, *E/Z* > 20:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.79 (d, J = 8.4 Hz, 2H), 7.51-7.43 (m, 3H), 7.26 (d, J = 8.0 Hz, 2H), 6.94 (d, J = 8.4 Hz, 2H), 6.77 (d, J = 15.6 Hz, 1H), 3.80 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ: 163.6, 139.9, 132.3, 131.8, 131.4, 129.9, 129.8, 128.6, 125.4, 114.6, 55.7.

(F) Reference

(1) Gadde, K.; Mampuys, P.; Guidetti, A.; Ching, H. Y. V.; Herrebout, W. A.; Van Doorslaer, S.; Abbaspour Tehrani, K.; Maes, B. U. W. Thiosulfonylation of Unactivated Alkenes with Visible-Light Organic Photocatalysis. *ACS Catal.* **2020**, *10*, 8765-8779.

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(3) Zeng, G.; Luo, H.; Jiang, K.; Cai, J.; Yin, B. Fully Atom-Economic Access to

Spiro-Cyclic Skeletons through Photoredox-Induced Hydrogen Transfer/Giese Addition/Dearomative Cyclization/Protonation Cascade. *Org. Chem. Front.* **2024**, *11*, 3165-3172.

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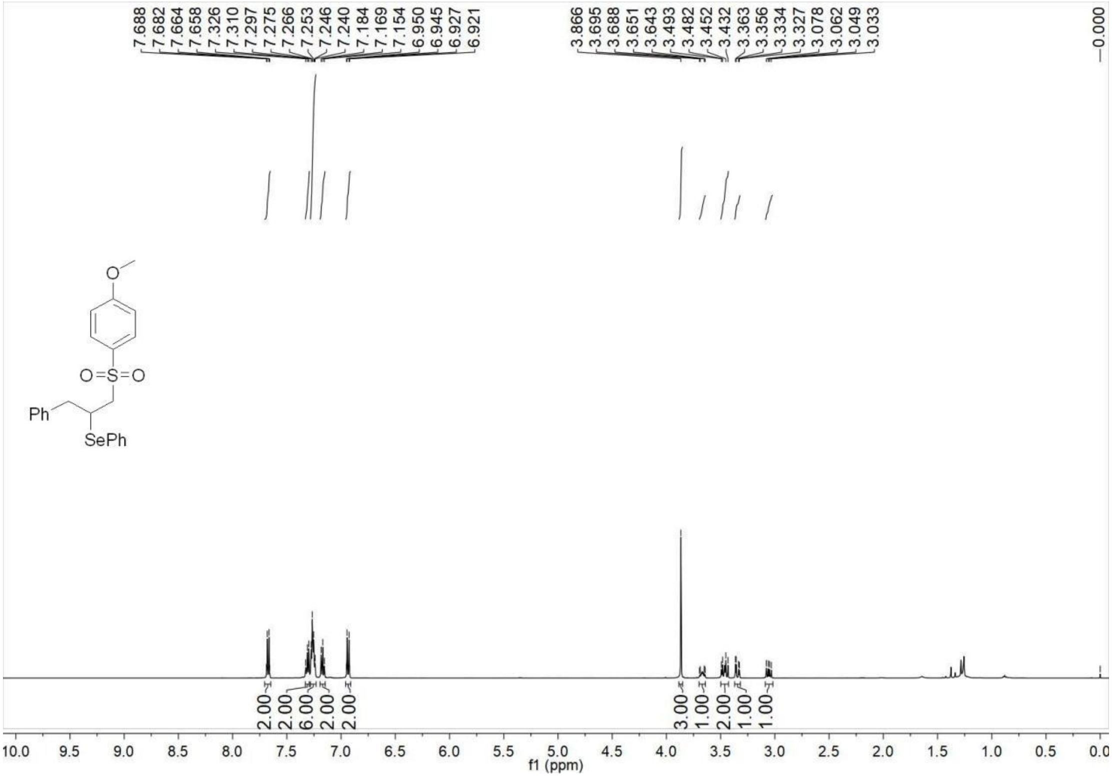
(5) Shu, X. R.; Li, M. H.; Wu, C.; Luo, X. N.; Yang, D. Q.; Yang, M. Q.; Lu, Y. J.; Ge, G. P.; Liu, J.; Wei, W. T. Four-Component Radical 1,2-Selenosulfonylation of Allenes. *Org. Lett.* **2024**, *26*, 5705-5712.

(6) Mei, L.; Shu, X.-R.; Liu, F.-L.; Li, J.-Z.; Zhang, J.-F.; Tang, K.; Wei, W.-T. Multicomponent Hydrosulfonylation of Alkynes for The Synthesis of Vinyl Sulfones. *Green Chem.* **2023**, *25,* 8820-8825.

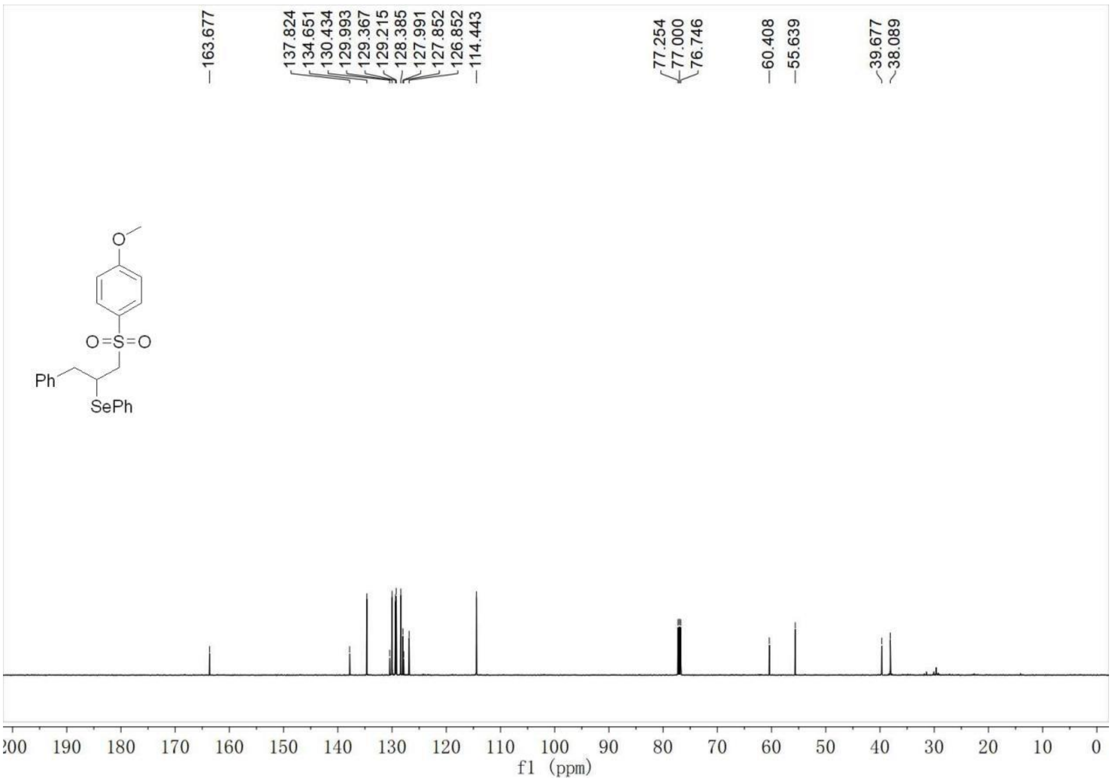
(G) Spectra

(1-((4-Methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)(phenyl)selane (5a)

^{1}H NMR (500 MHz, $CDCl_3$) of **5a**

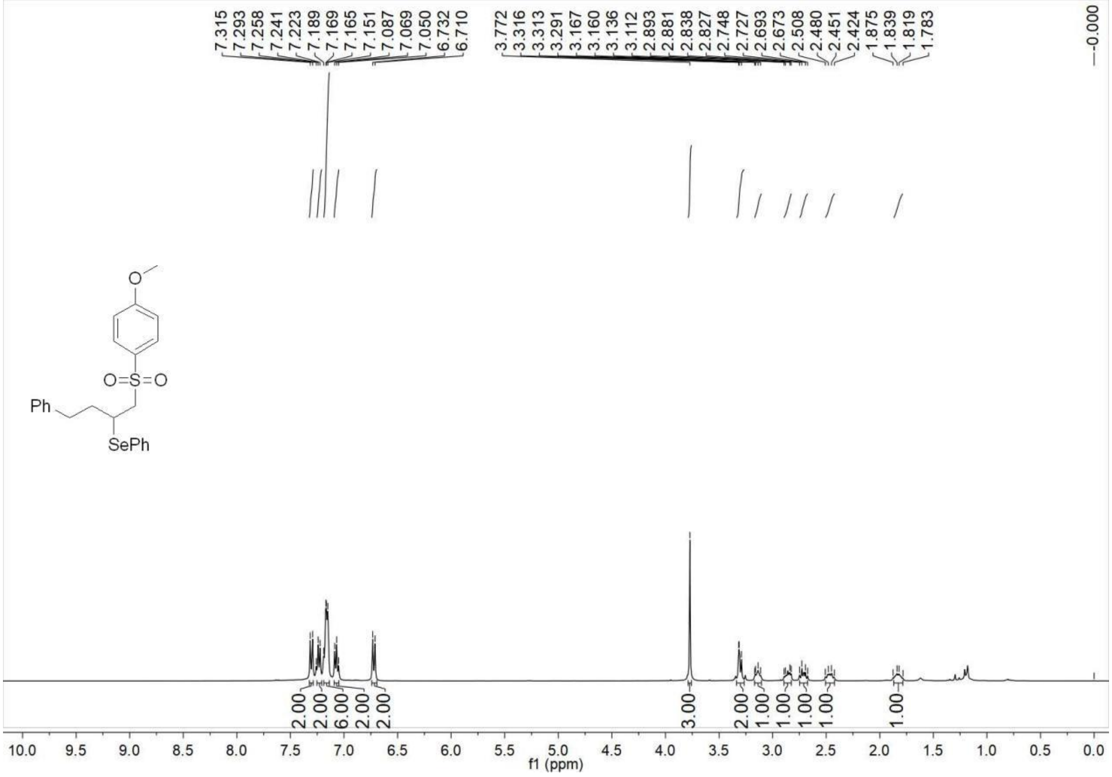


^{13}C NMR (126 MHz, $CDCl_3$) of **5a**

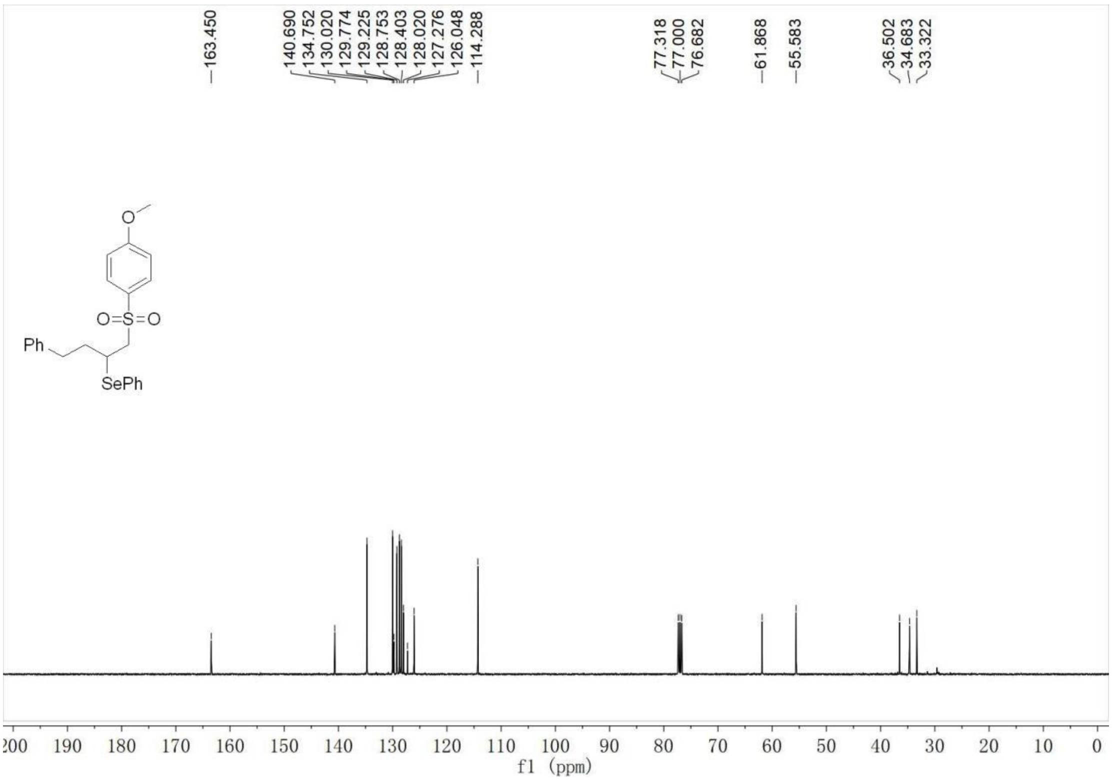


(1-((4-Methoxyphenyl)sulfonyl)-4-phenylbutan-2-yl)(phenyl)selane (5b)

^{1}H NMR (400 MHz, $CDCl_3$) of **5b**

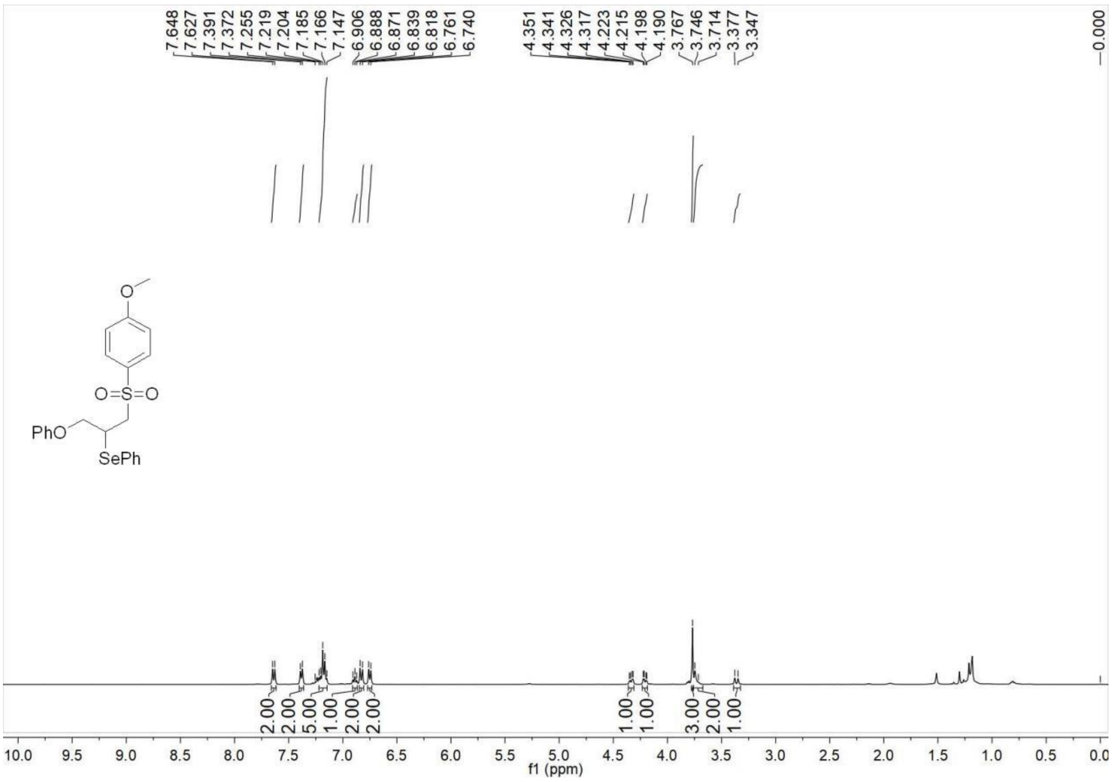


^{13}C NMR (101 MHz, $CDCl_3$) of **5b**

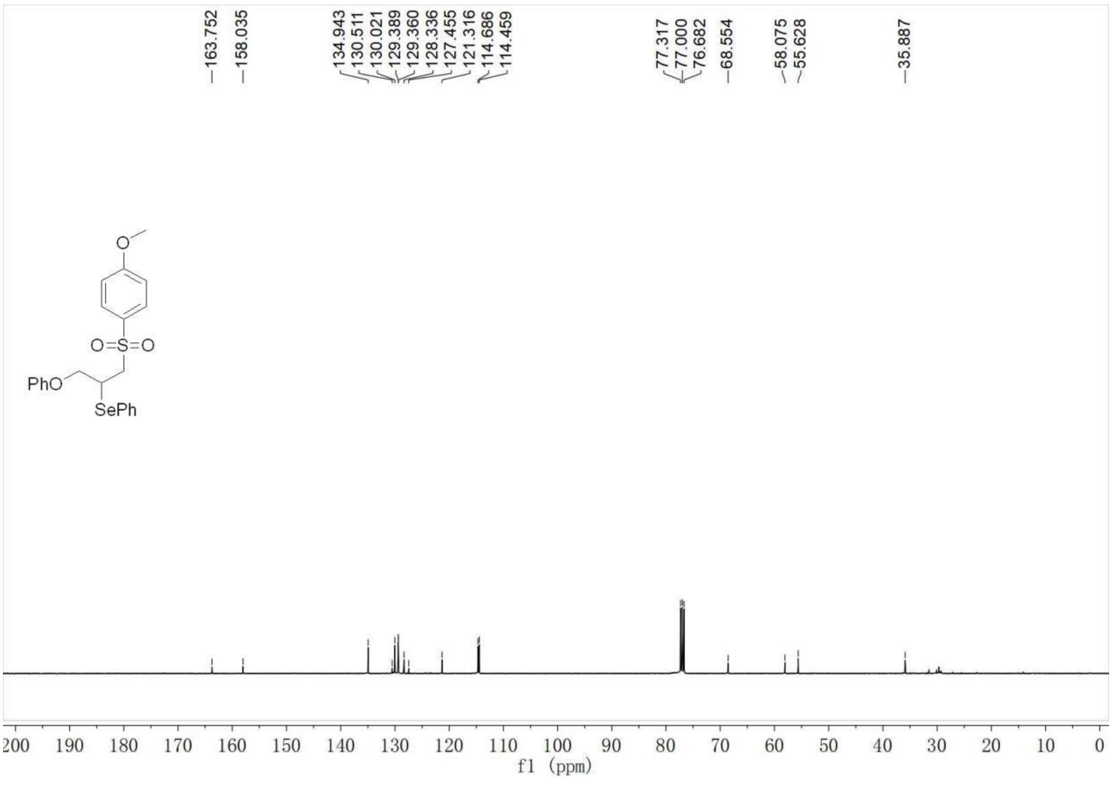


(1-((4-Methoxyphenyl)sulfonyl)-3-phenoxypropan-2-yl)(phenyl)selane (5c)

^{1}H NMR (400 MHz, CDCl$_3$) of **5c**

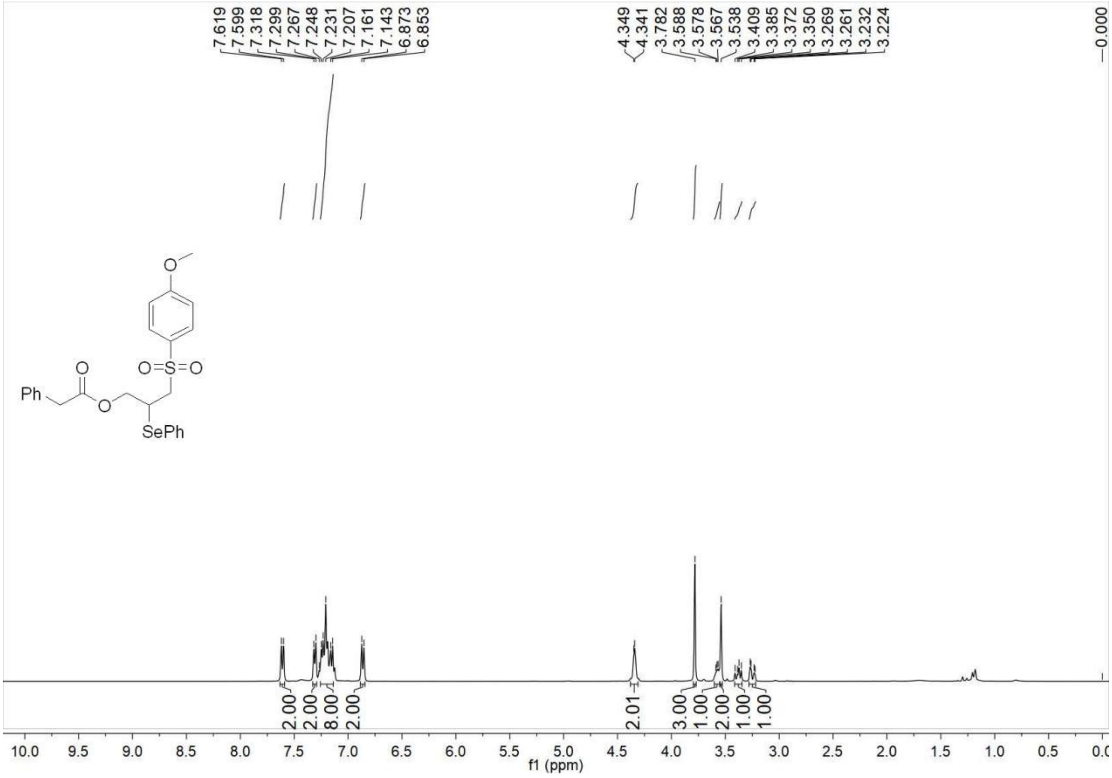


^{13}C NMR (101 MHz, CDCl$_3$) of **5c**

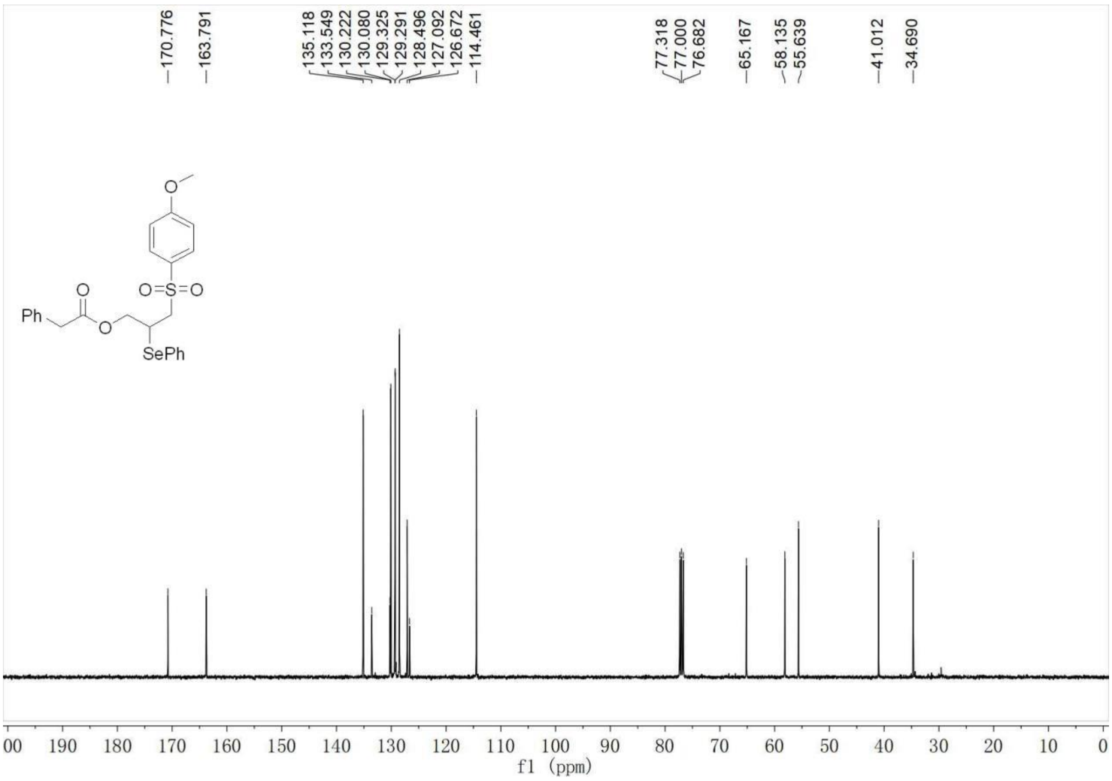


3-((4-Methoxyphenyl)sulfonyl)-2-(phenylselanyl)propyl 2-phenylacetate (5d)

^{1}H NMR (400 MHz, CDCl$_3$) of **5d**

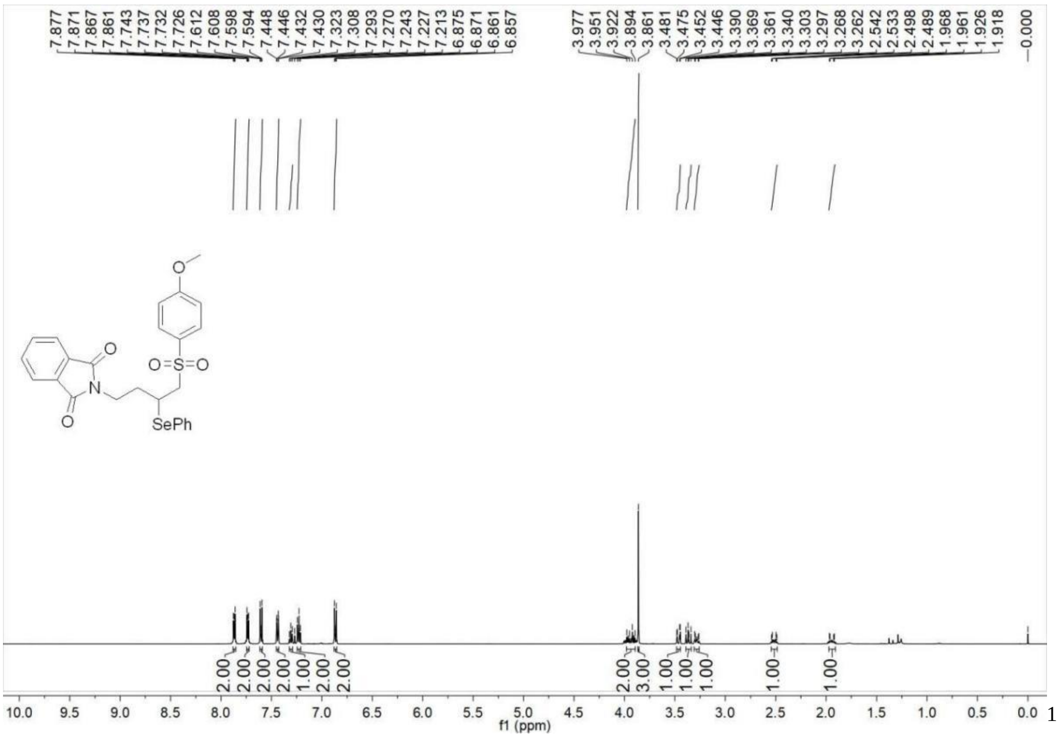


^{13}C NMR (101 MHz, CDCl$_3$) of **5d**



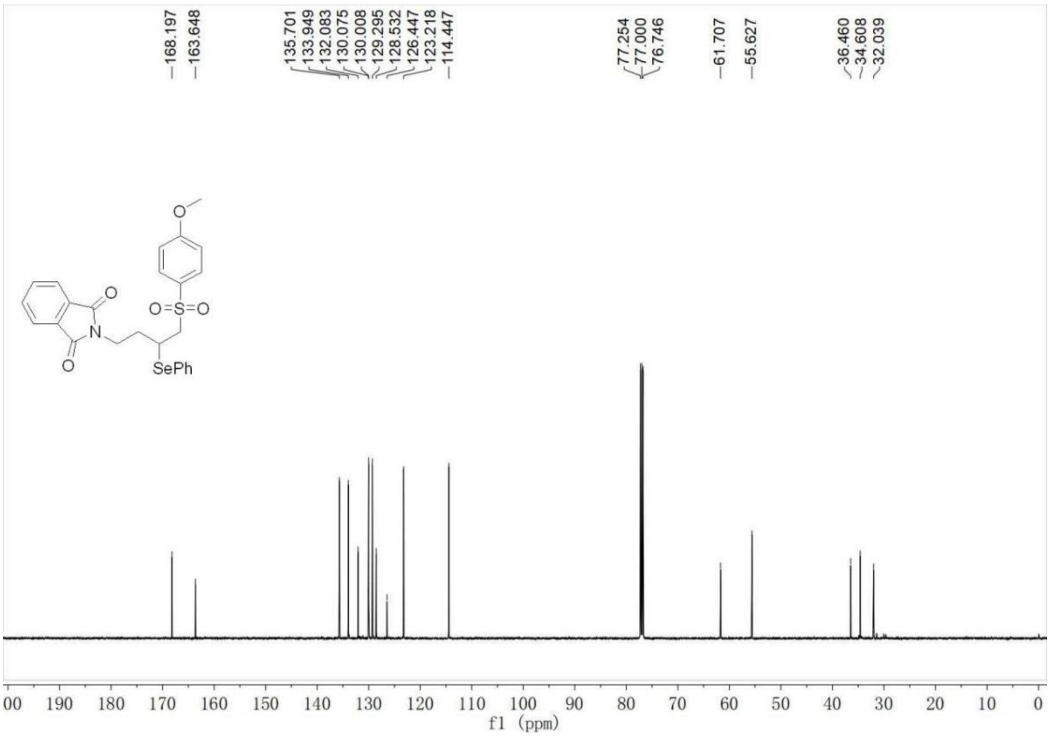
2-(4-((4-Methoxyphenyl)sulfonyl)-3-(phenylselanyl)butyl)isoindoline-1,3-dione (5e)

^{1}H NMR (500 MHz, CDCl$_3$) of **5e**



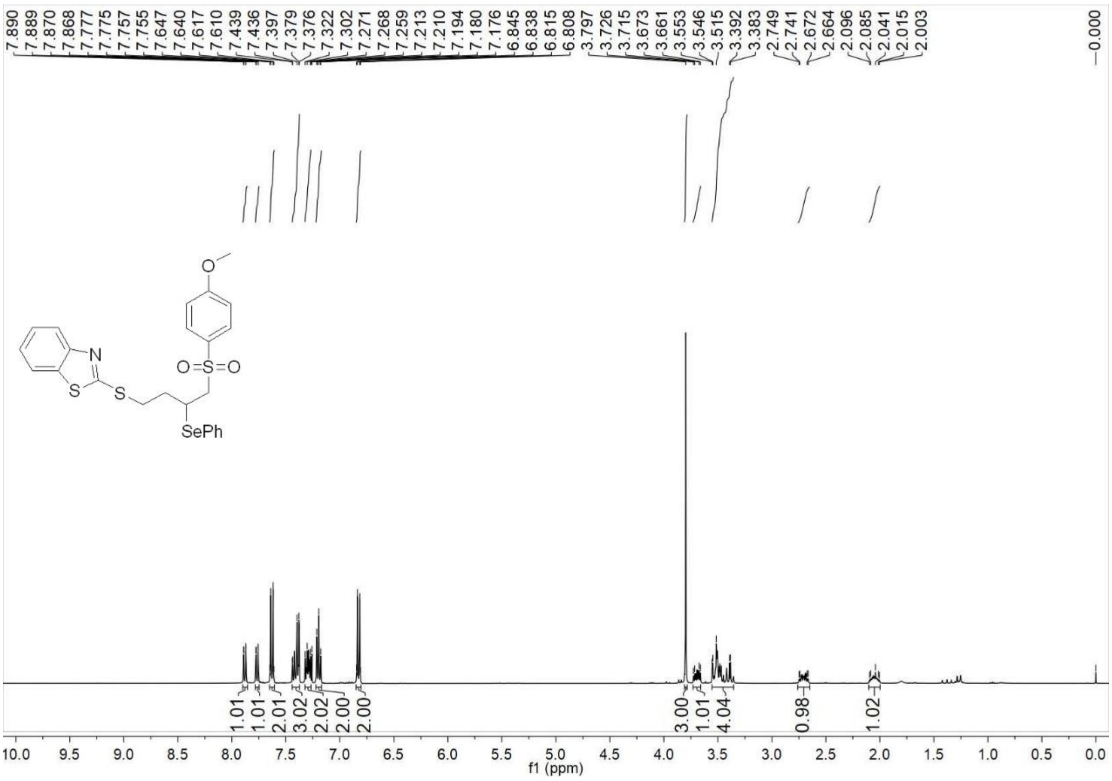
^{13}C

^{13}C NMR (126 MHz, CDCl$_3$) of **5e**

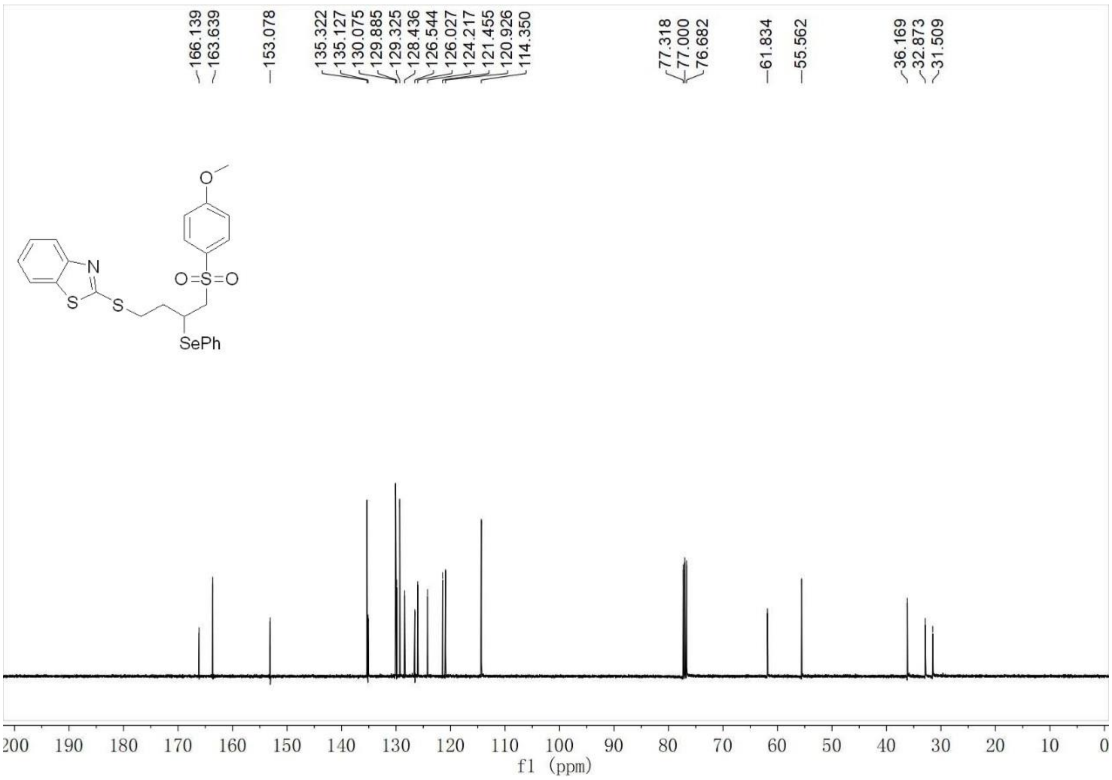


2-((4-((4-Methoxyphenyl)sulfonyl)-3-(phenylselanyl)butyl)thio)benzo[*d*]thiazole (5f)

^{1}H NMR (400 MHz, CDCl$_3$) of **5f**

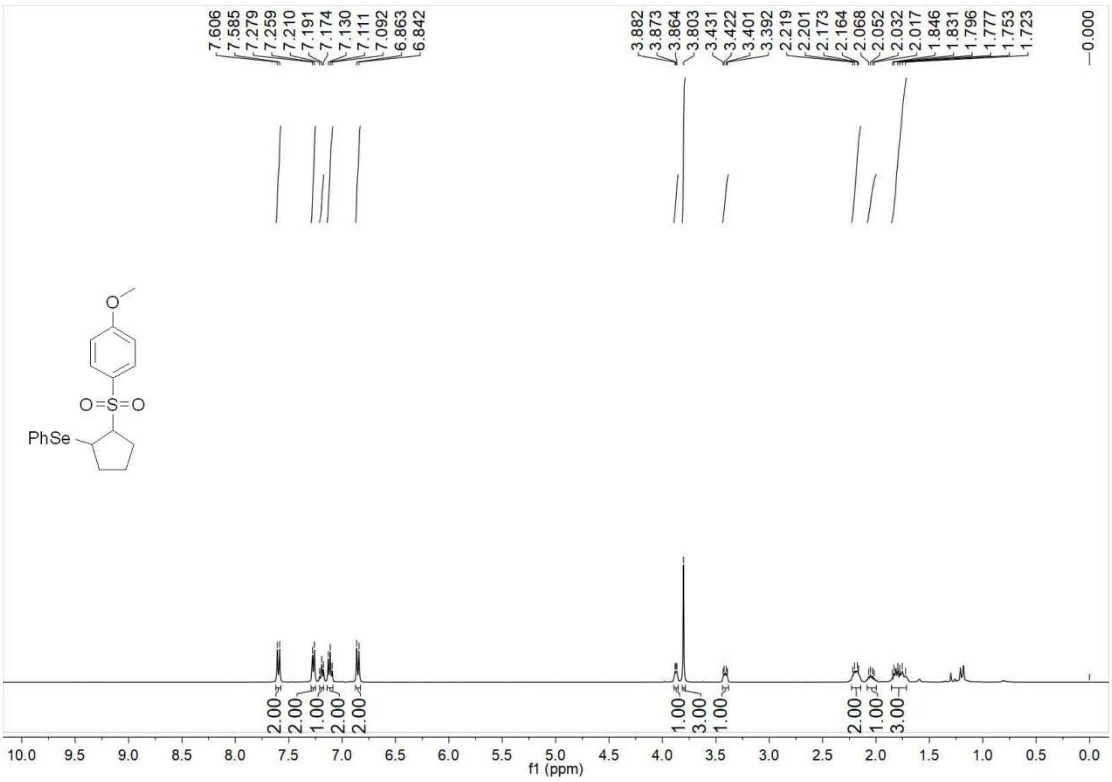


^{13}C NMR (101 MHz, CDCl$_3$) of **5f**

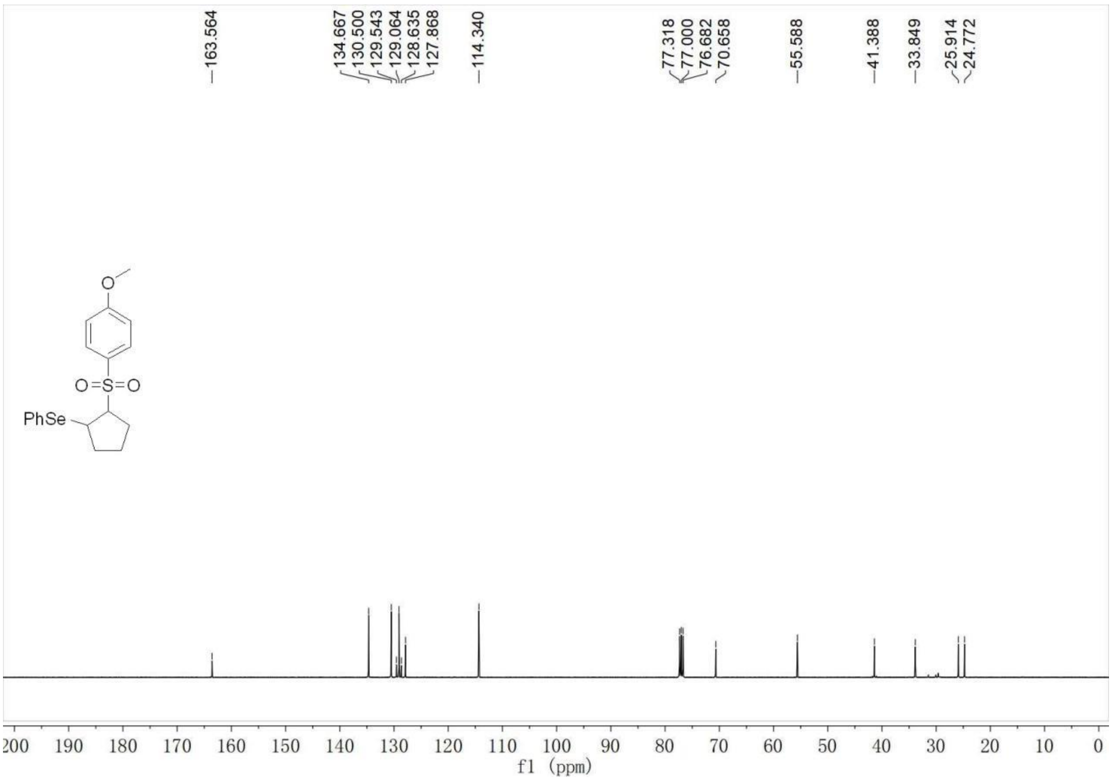


(2-((4-Methoxyphenyl)sulfonyl)cyclopentyl)(phenyl)selane (5g)

^{1}H NMR (400 MHz, CDCl$_3$) of **5g**

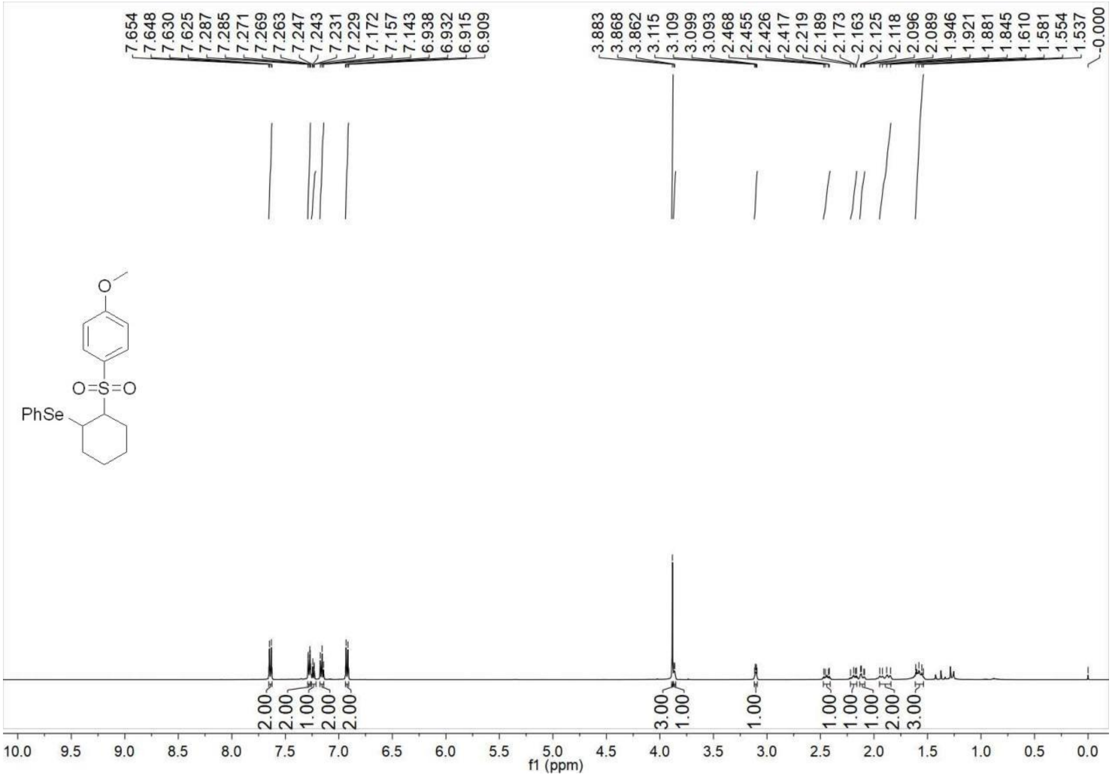


^{13}C NMR (101 MHz, CDCl$_3$) of **5g**

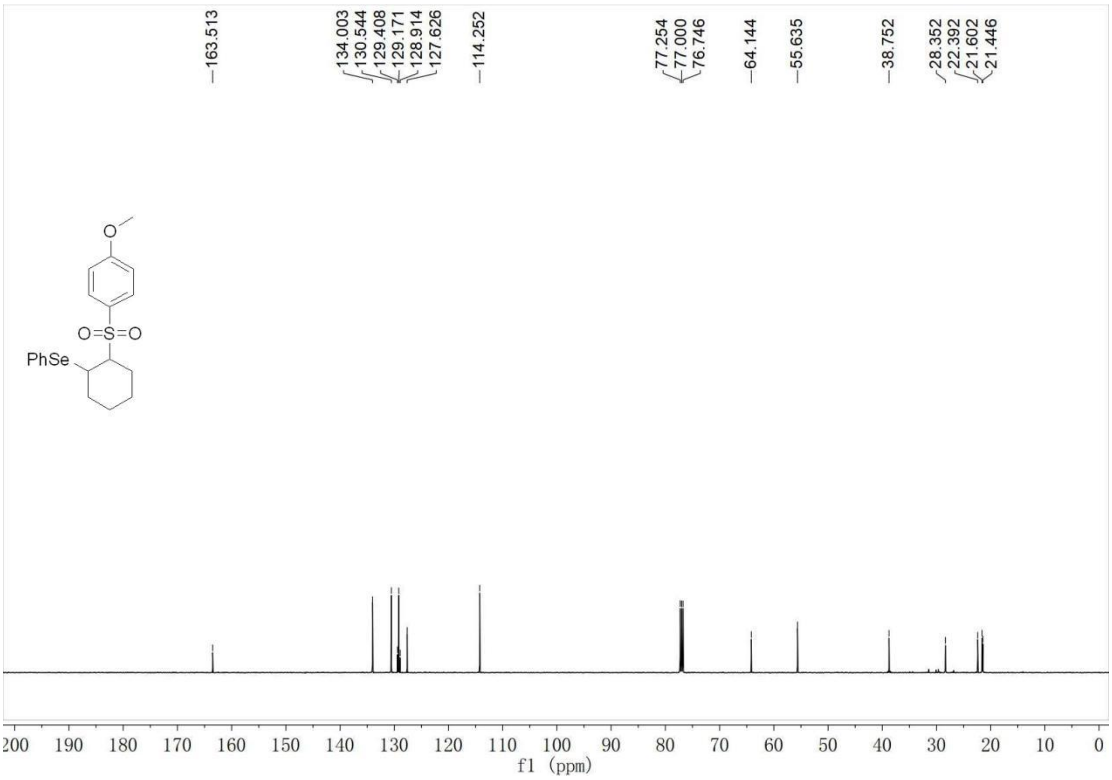


(2-((4-Methoxyphenyl)sulfonyl)cyclohexyl)(phenyl)selane (5h)

^{1}H NMR (500 MHz, CDCl$_3$) of **5h**

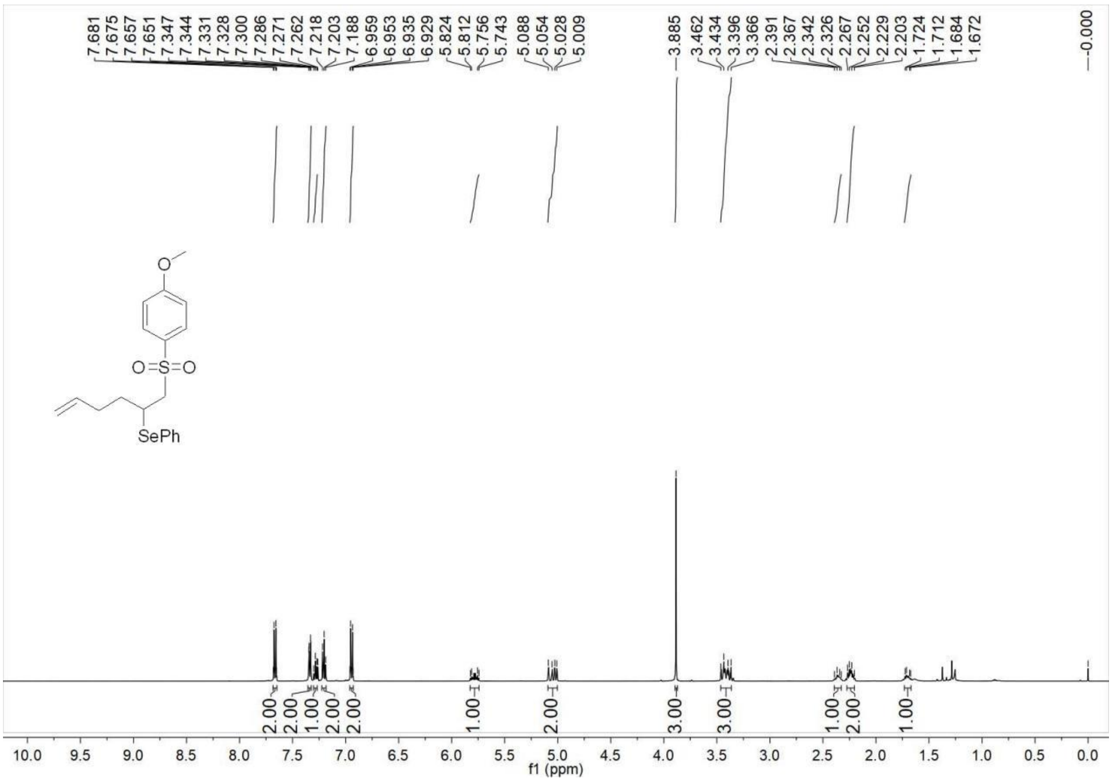


^{13}C NMR (126 MHz, CDCl$_3$) of **5h**

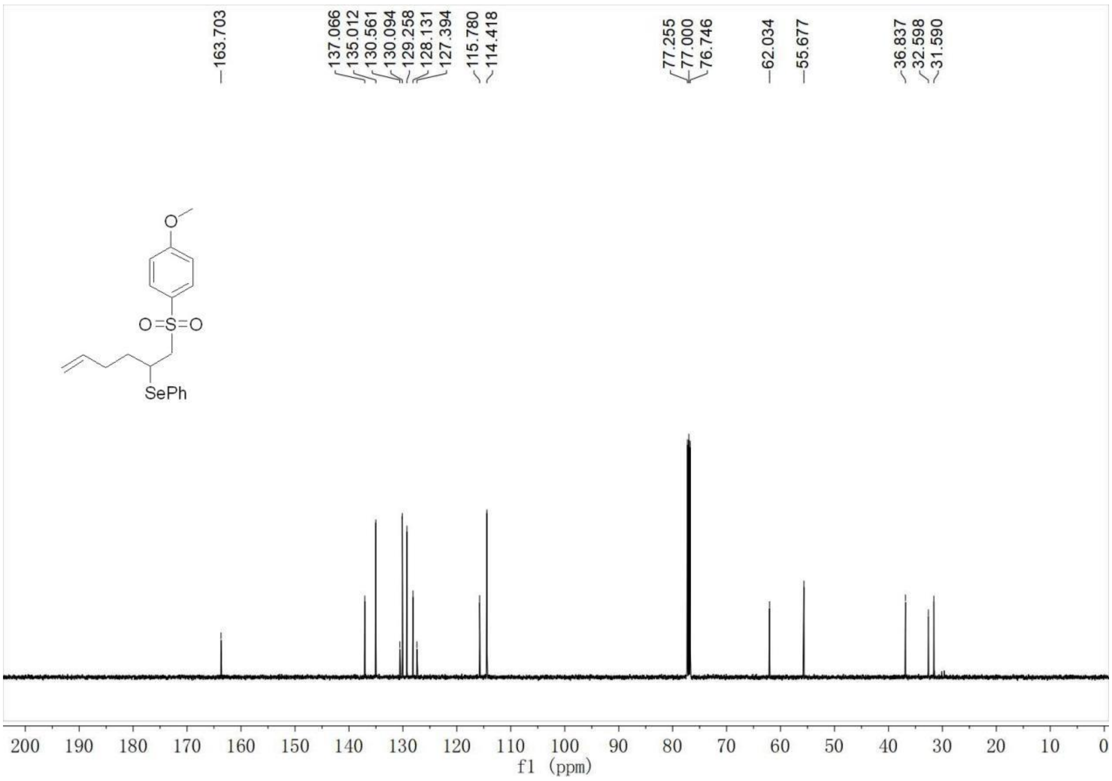


(1-((4-Methoxyphenyl)sulfonyl)hex-5-en-2-yl)(phenyl)selane (5i)

^{1}H NMR (500 MHz, CDCl$_3$) of **5i**

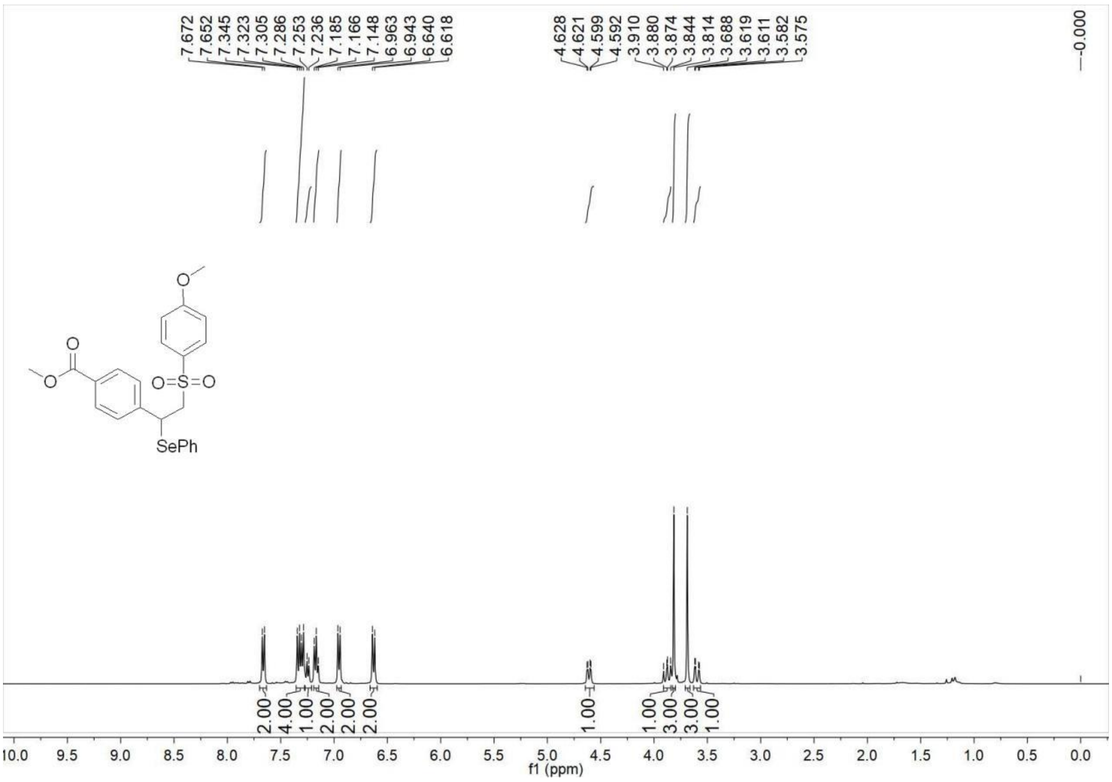


^{13}C NMR (126 MHz, CDCl$_3$) of **5i**

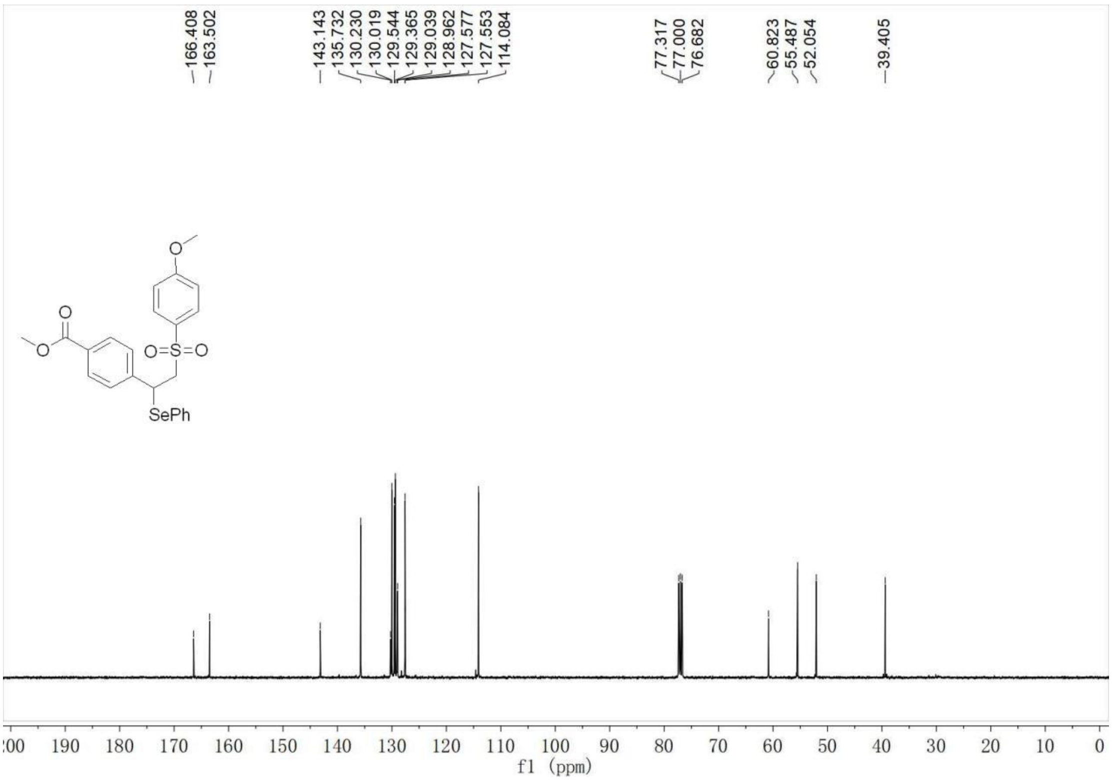


Methyl-4-(2-((4-methoxyphenyl)sulfonyl)-1-(phenylselanyl)ethyl)benzoate (5j)

^{1}H NMR (400 MHz, CDCl$_3$) of **5j**

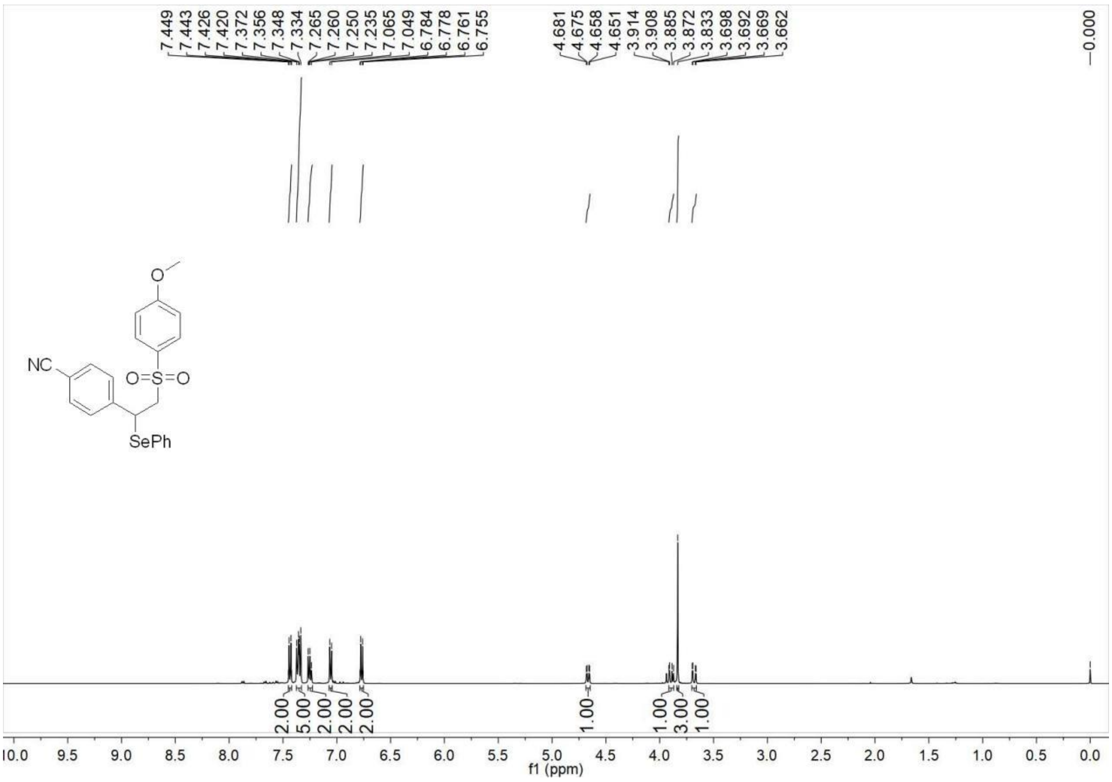


^{13}C NMR (101 MHz, CDCl$_3$) of **5j**

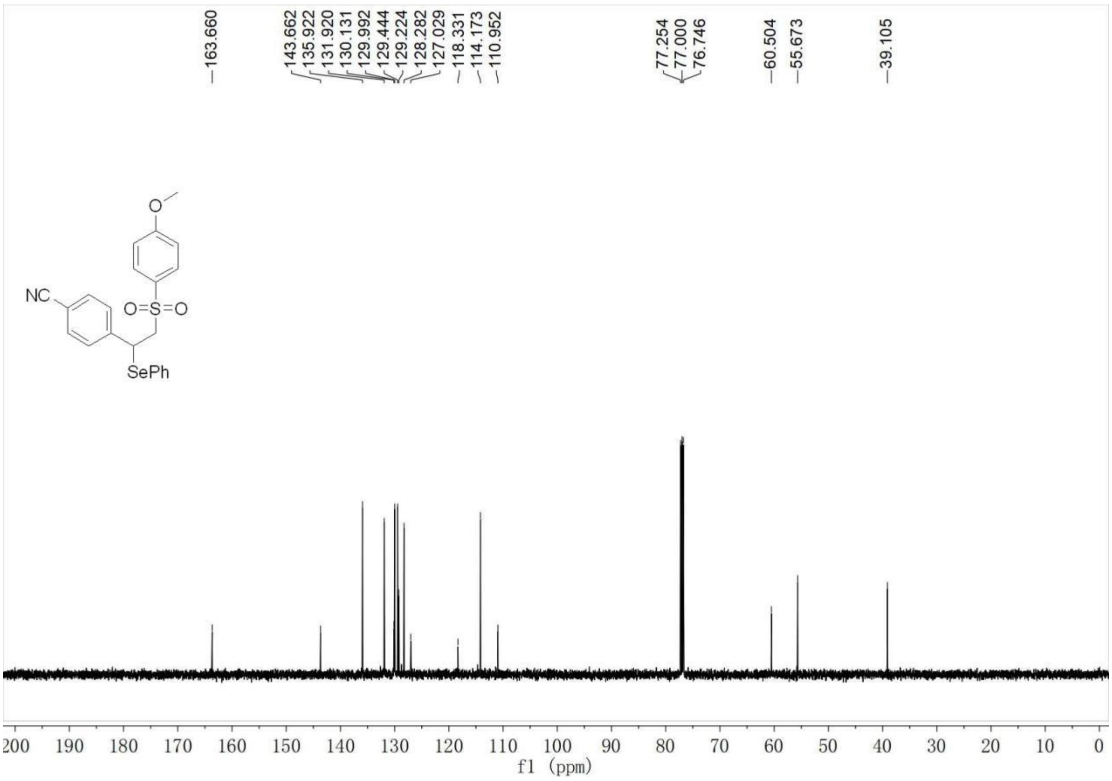


4-(2-((4-Methoxyphenyl)sulfonyl)-1-(phenylselanyl)ethyl)benzonitrile (5k)

^{1}H NMR (500 MHz, CDCl$_3$) of **5k**

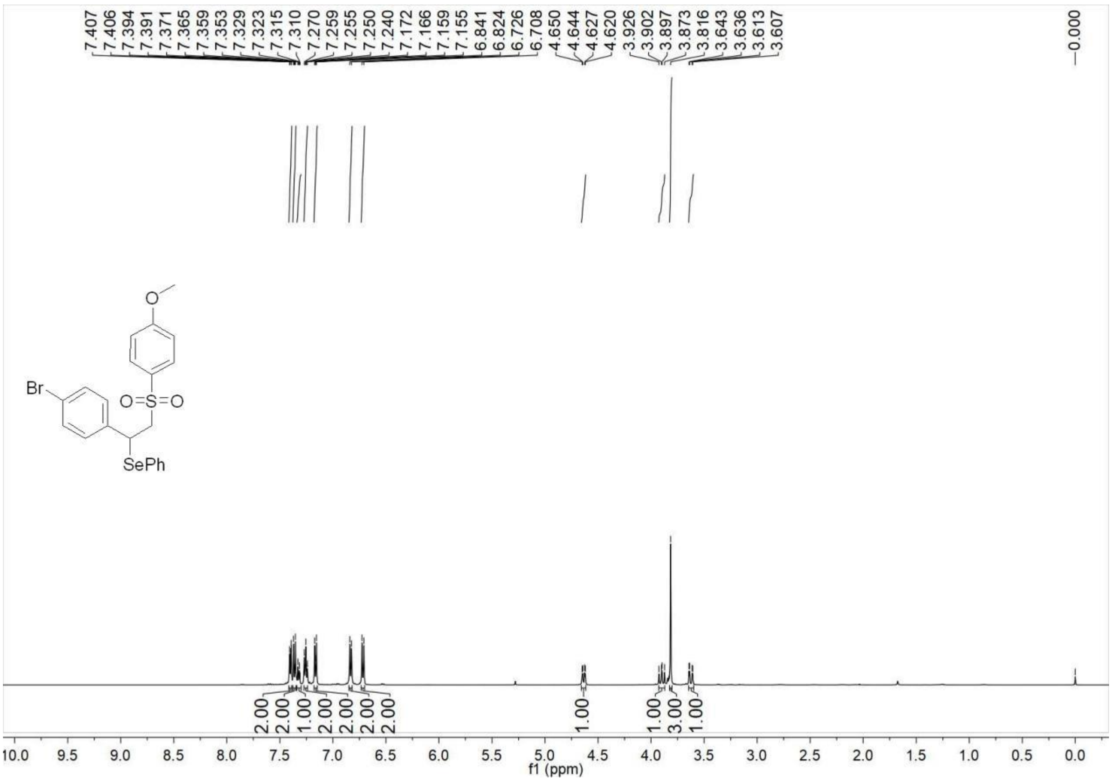


^{13}C NMR (126 MHz, CDCl$_3$) of **5k**

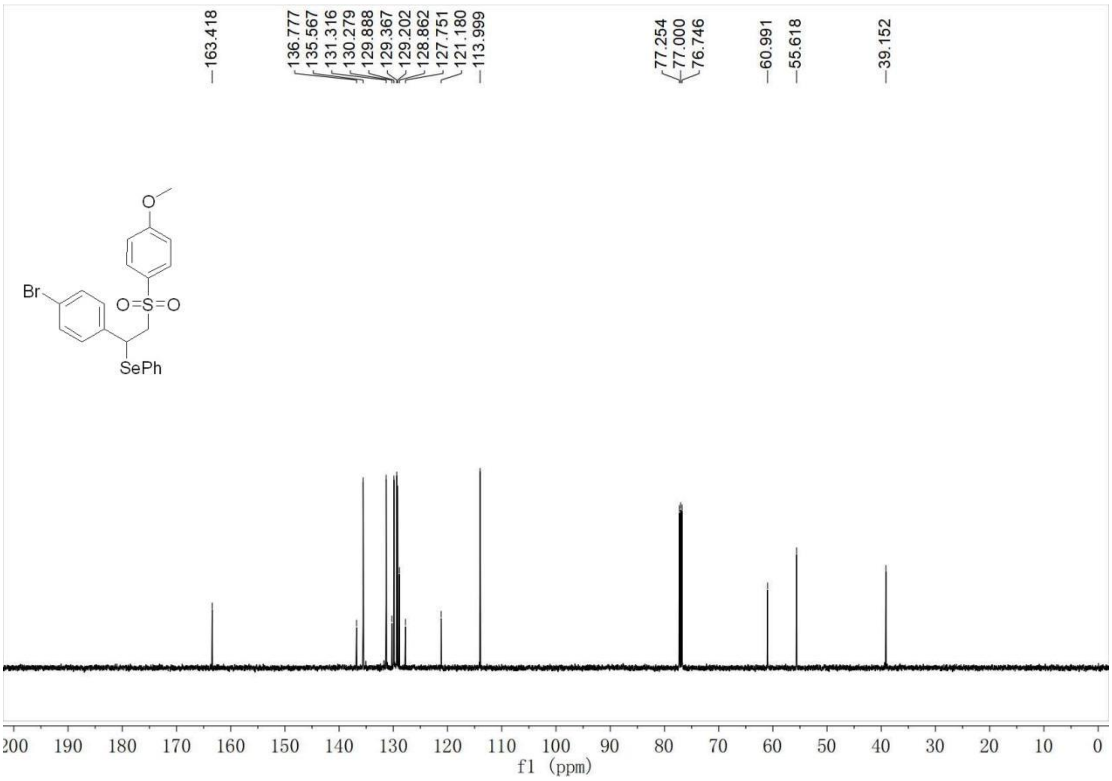


(1-(4-Bromophenyl)-2-((4-methoxyphenyl)sulfonyl)ethyl)(phenyl)selane (5l)

^{1}H NMR (500 MHz, $CDCl_3$) of **5l**

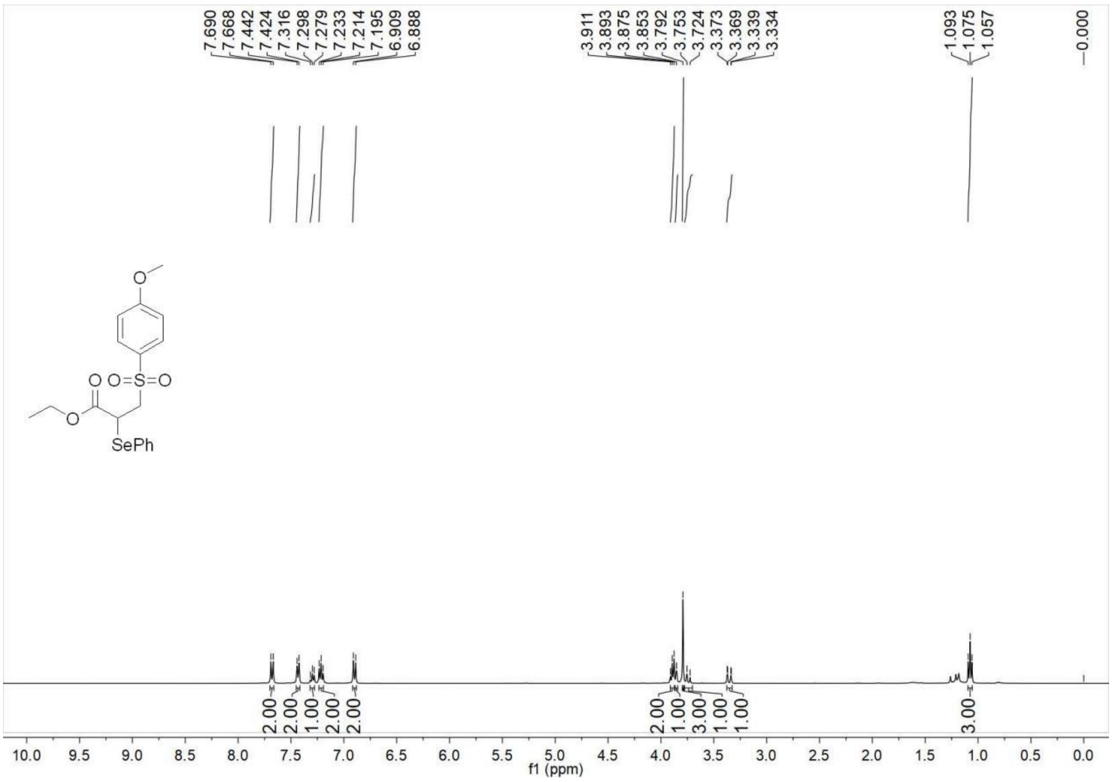


^{13}C NMR (126 MHz, $CDCl_3$) of **5l**

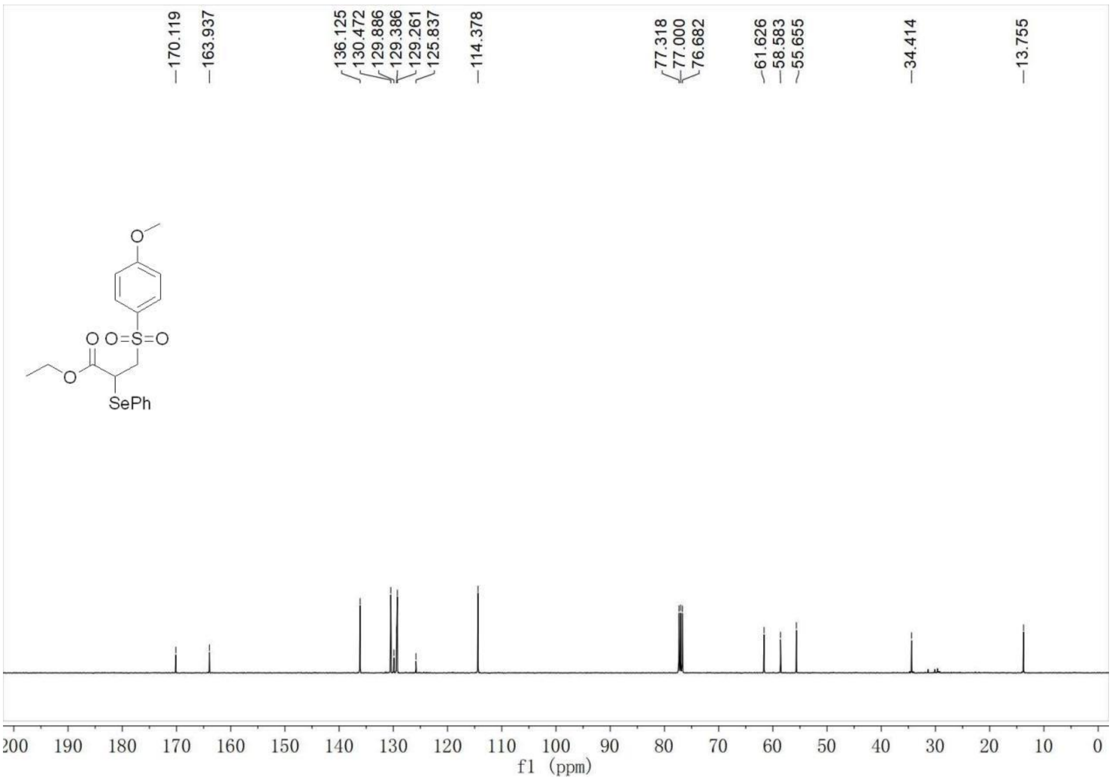


Ethyl-3-((4-methoxyphenyl)sulfonyl)-2-(phenylselanyl)propanoate (5m)

^{1}H NMR (400 MHz, $CDCl_3$) of **5m**

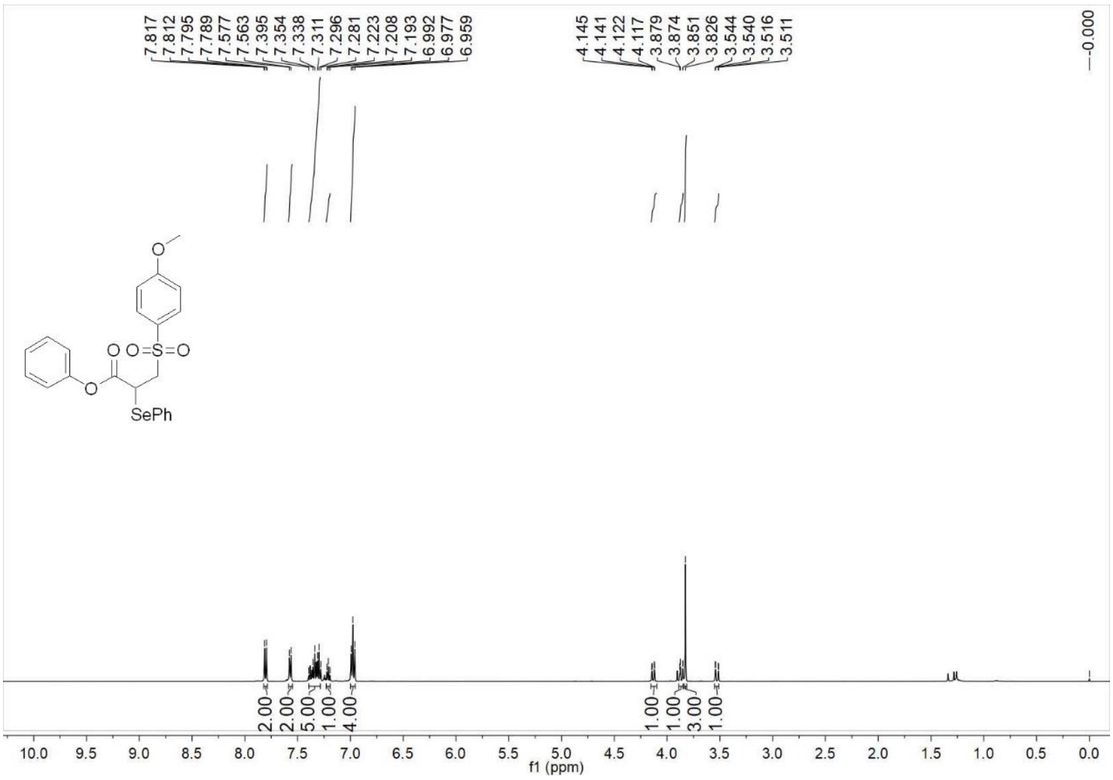


^{13}C NMR (101 MHz, $CDCl_3$) of **5m**

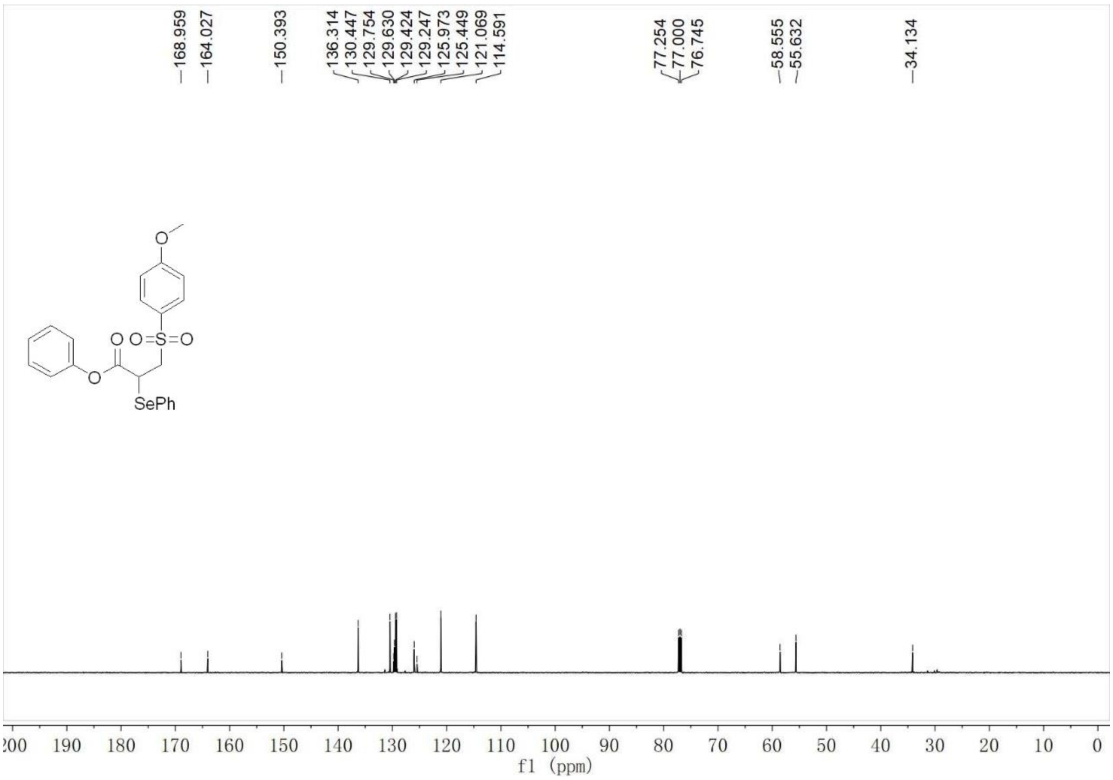


Phenyl-3-((4-methoxyphenyl)sulfonyl)-2-(phenylselanyl)propanoate (5n)

^{1}H NMR (500 MHz, CDCl$_3$) of **5n**

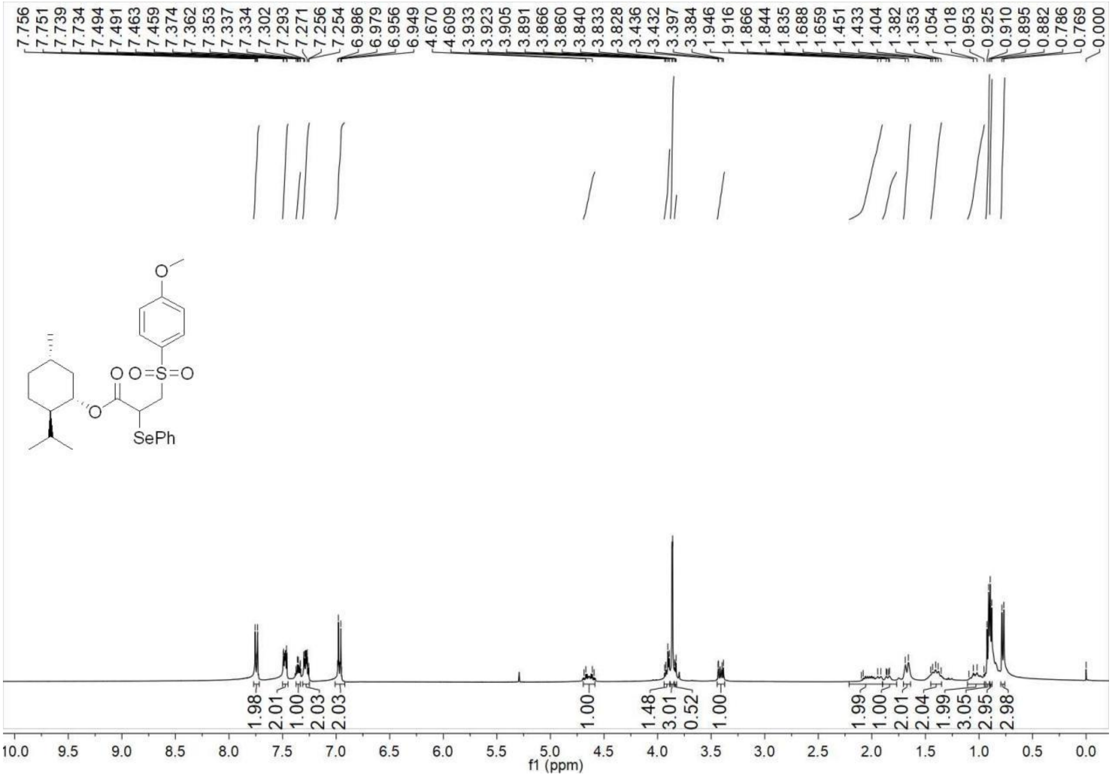


^{13}C NMR (126 MHz, CDCl$_3$) of **5n**

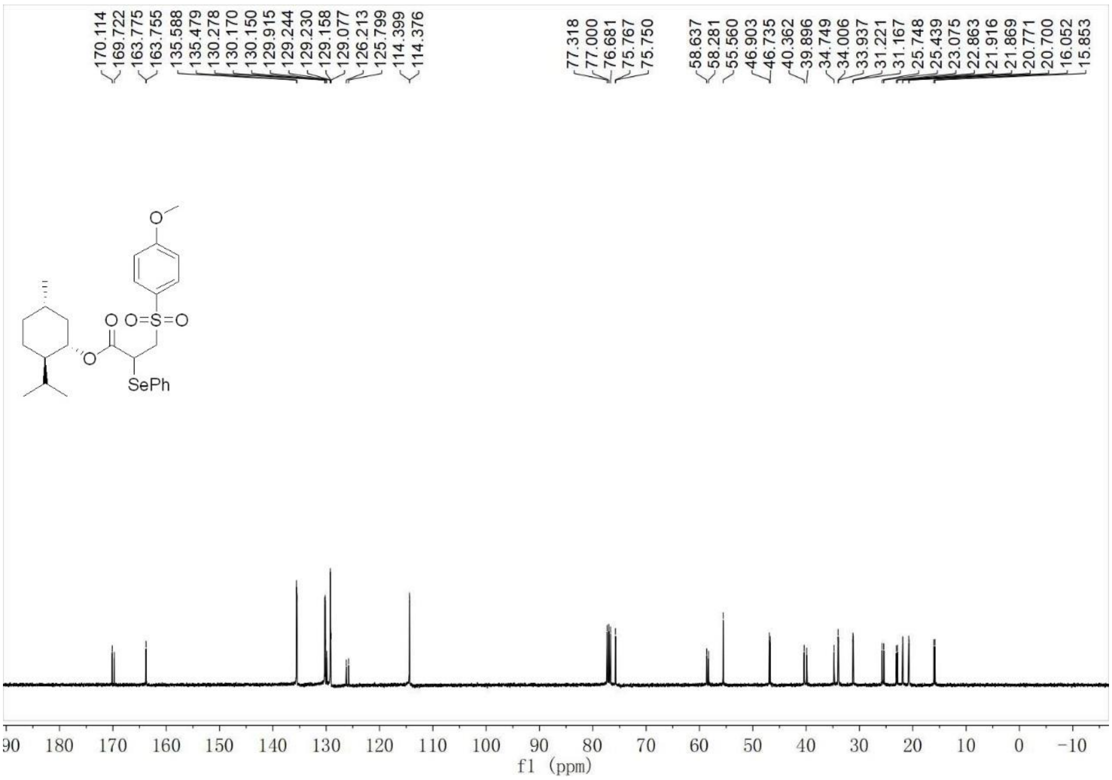


(1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl 3-((4-methoxyphenyl)sulfonyl)-2-(phenylselanyl)propanoate (5o)

^{1}H NMR (400 MHz, $CDCl_3$) of **5o**

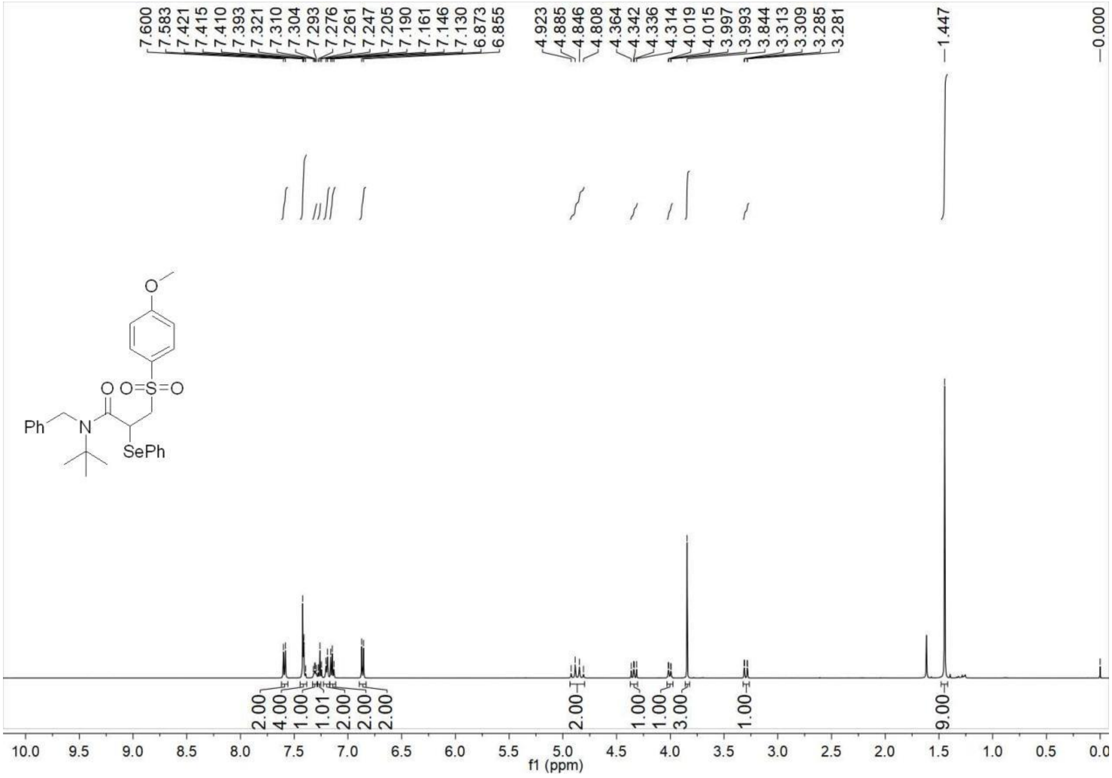


^{13}C NMR (101 MHz, $CDCl_3$) of **5o**

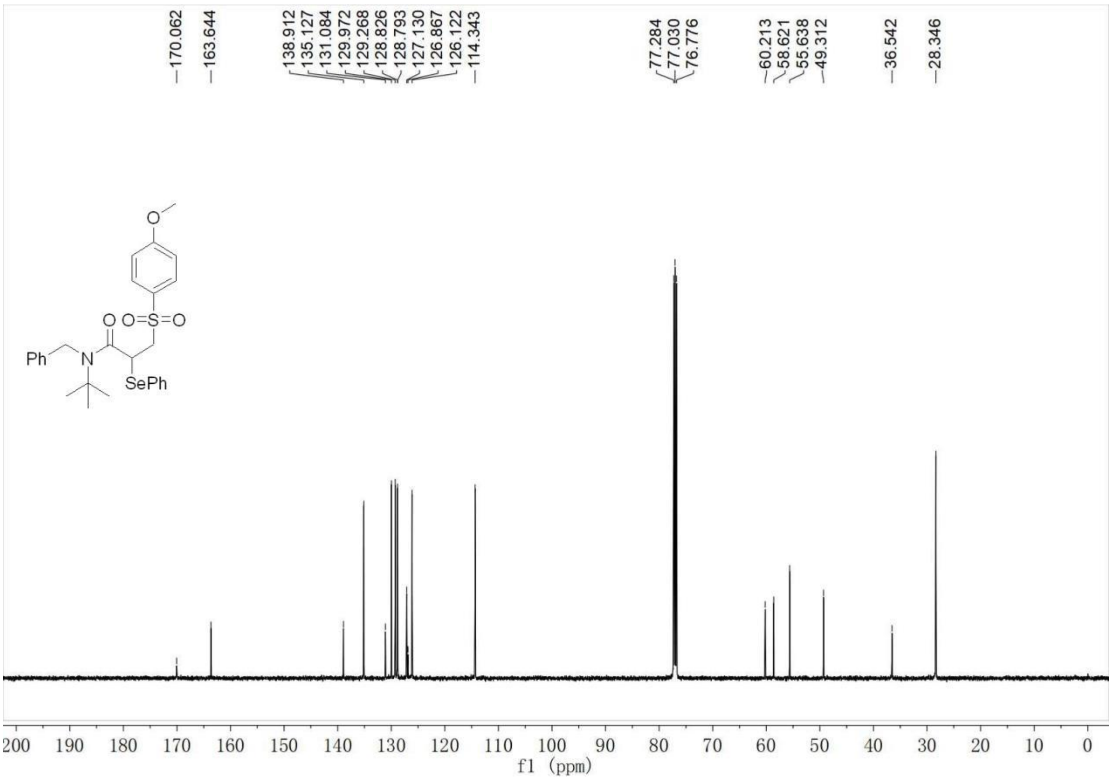


N-Benzyl-*N*-(*tert*-butyl)-3-((4-methoxyphenyl)sulfonyl)-2-(phenylselanyl)propanami (5p)

^{1}H NMR (500 MHz, CDCl$_3$) of **5p**

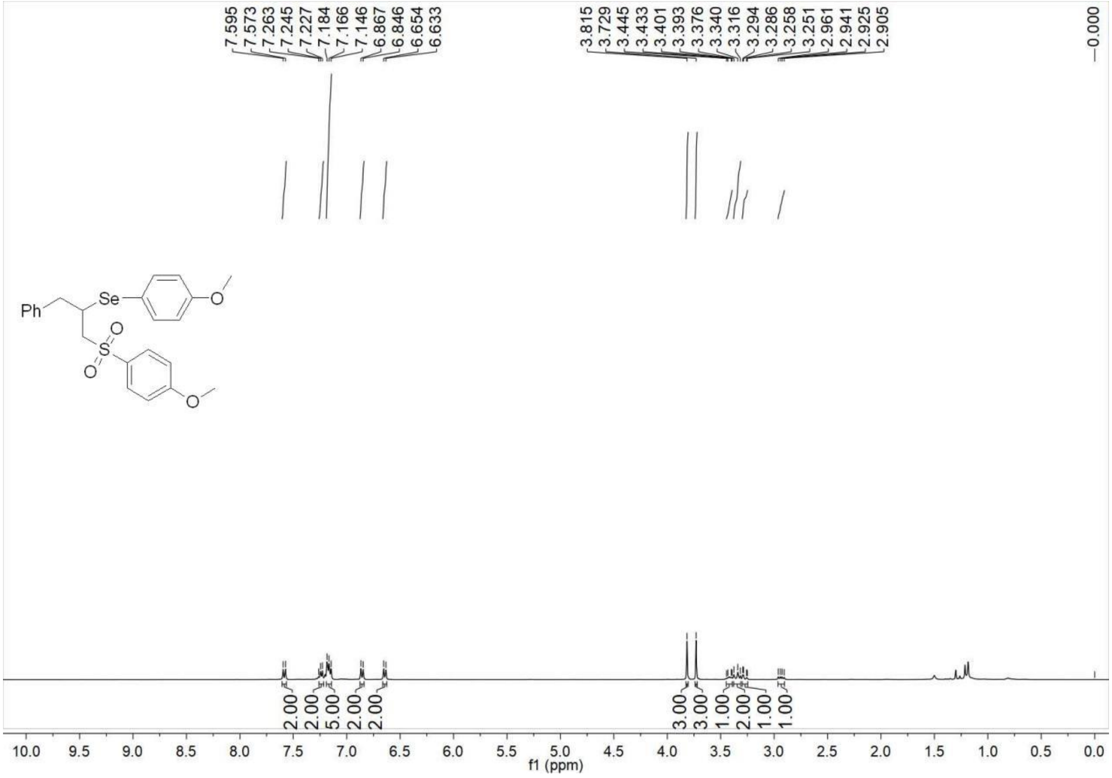


^{13}C NMR (126 MHz, CDCl$_3$) of **5p**

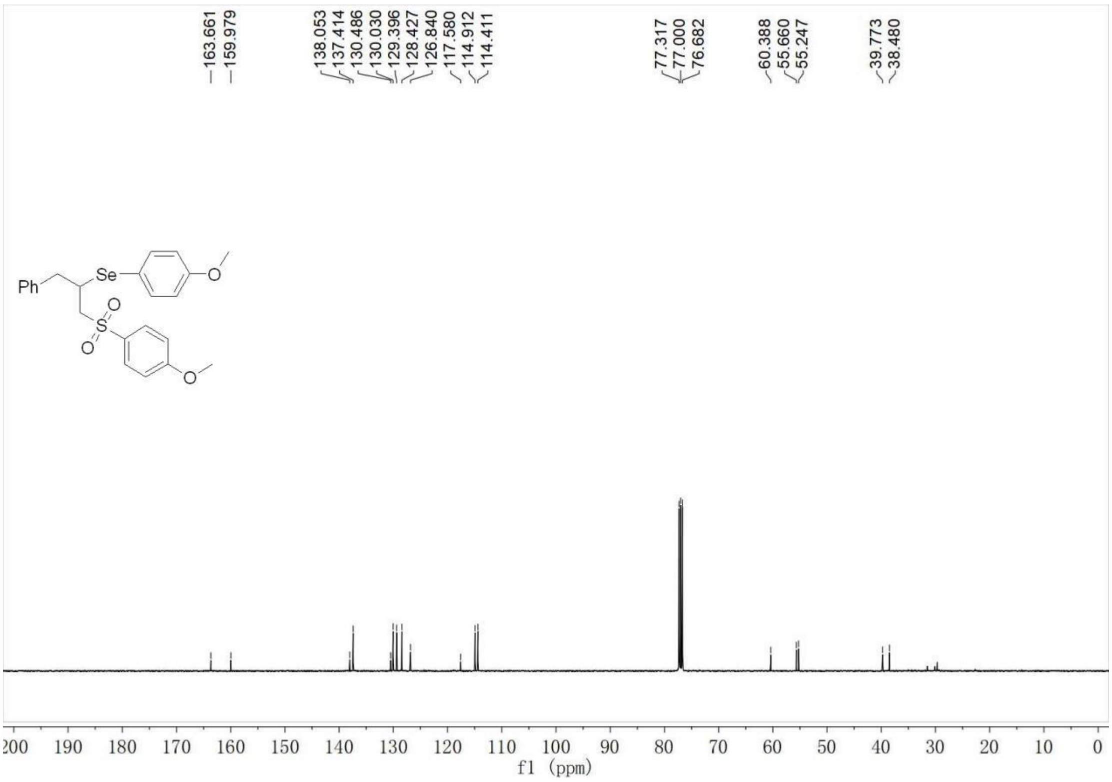


(4-Methoxyphenyl)(1-((4-methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)selane (5q)

^{1}H NMR (400 MHz, CDCl$_3$) of **5q**

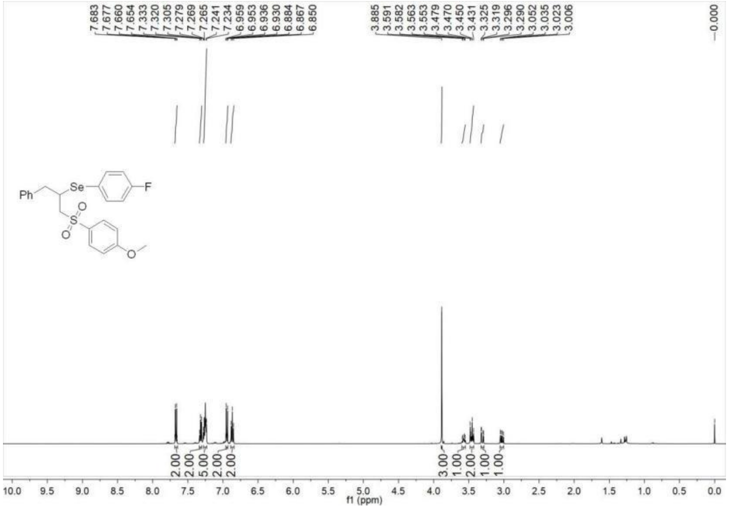


^{13}C NMR (101 MHz, CDCl$_3$) of **5q**

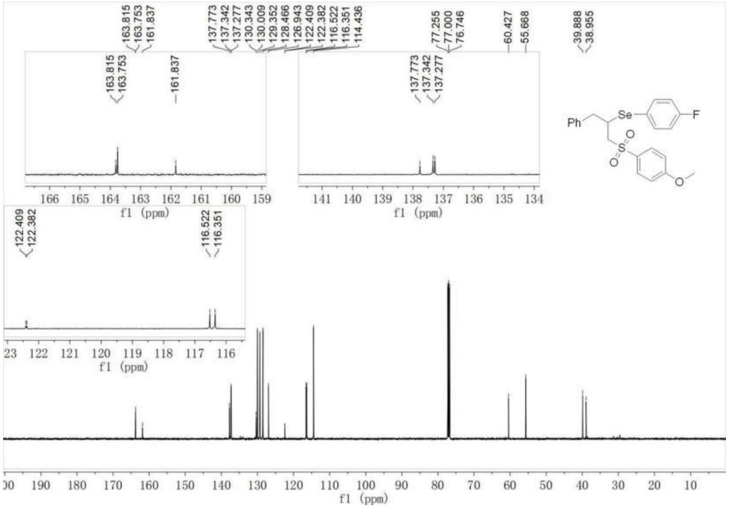


(4-Fluorophenyl)(1-((4-methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)selane (5r)

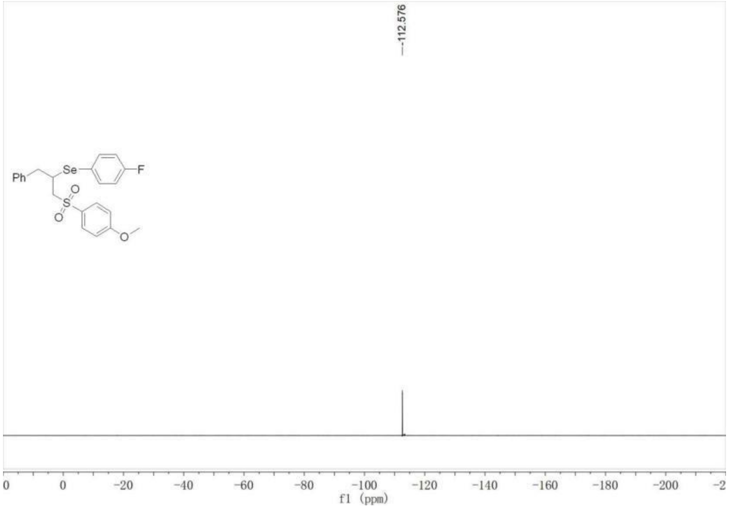
^{1}H NMR (500 MHz, CDCl$_3$) of **5r**



^{13}C NMR (126 MHz, CDCl$_3$) of **5r**

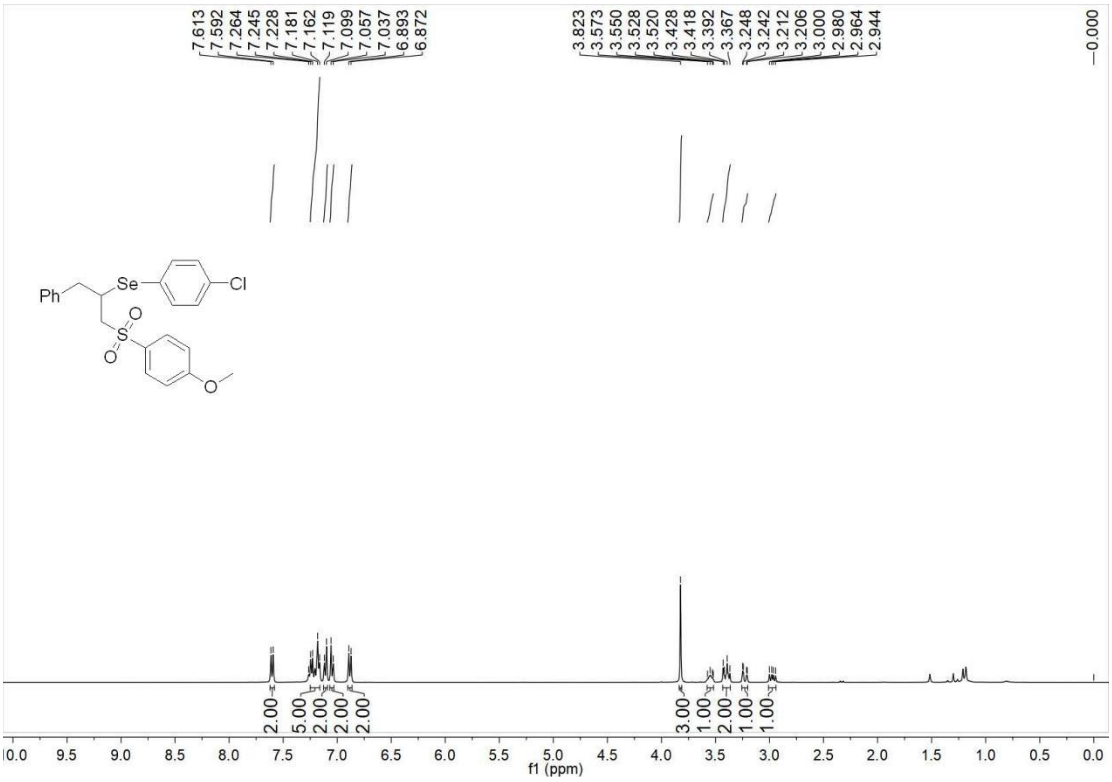


^{19}F NMR (471 MHz, CDCl$_3$) of **5r**

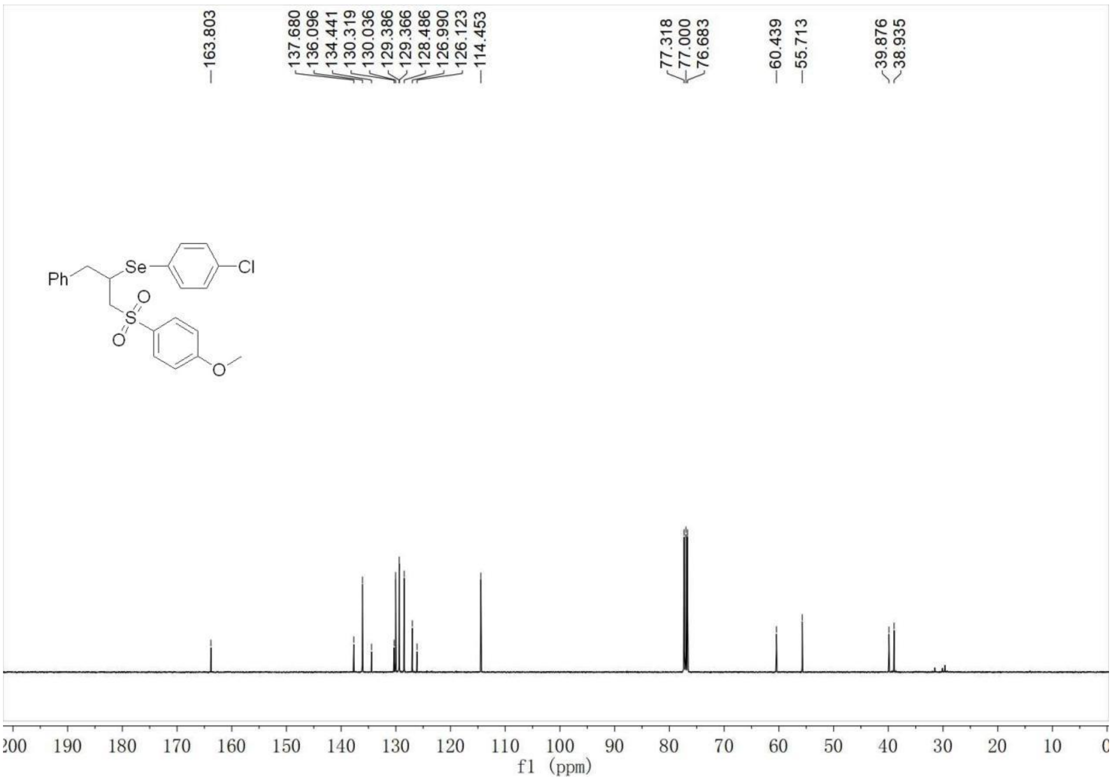


(4-Chlorophenyl)(1-((4-methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)selane (5s)

^{1}H NMR (400 MHz, CDCl$_3$) of **5s**

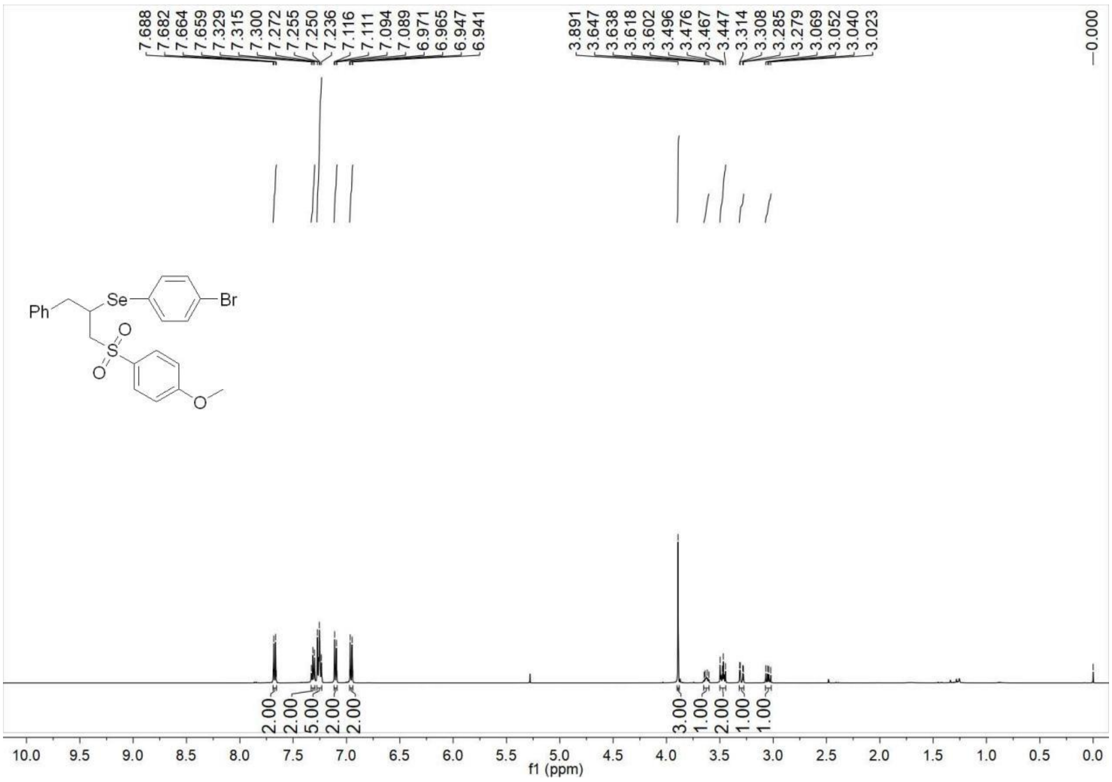


^{13}C NMR (101 MHz, CDCl$_3$) of **5s**

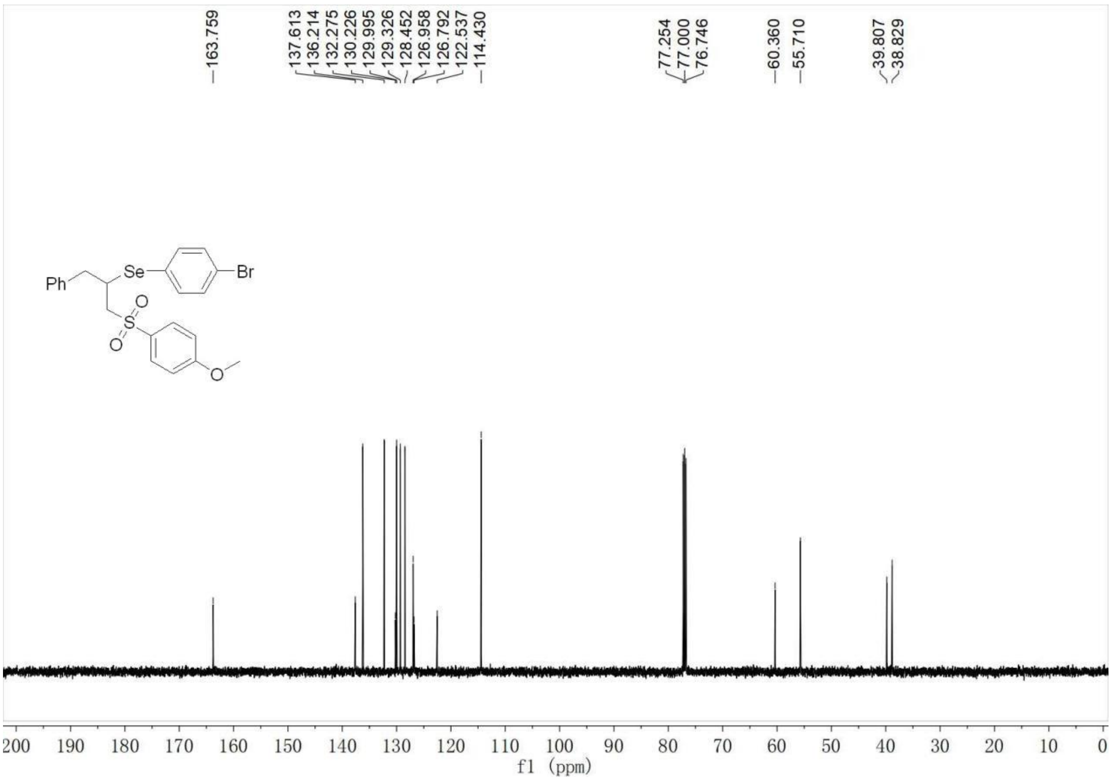


(4-Bromophenyl)(1-((4-methoxyphenyl)sulfonyl)-3-phenylpropan-2-yl)selane (5t)

^{1}H NMR (500 MHz, $CDCl_3$) of **5t**

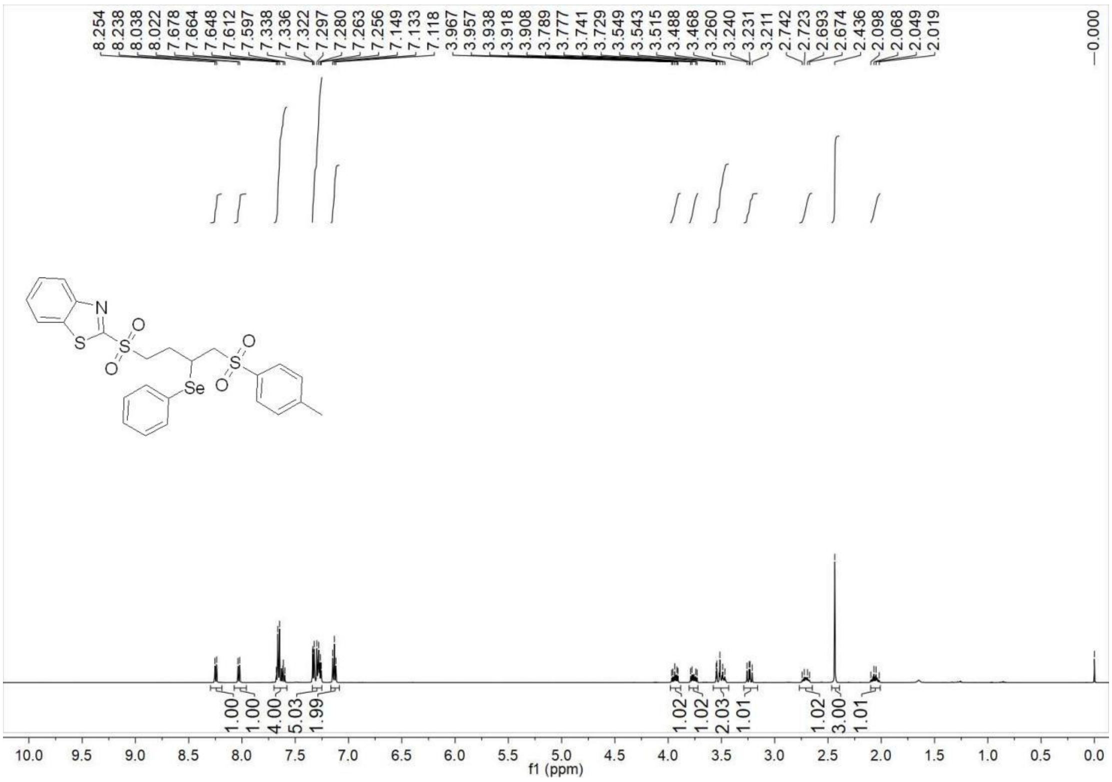


^{13}C NMR (126 MHz, $CDCl_3$) of **5t**

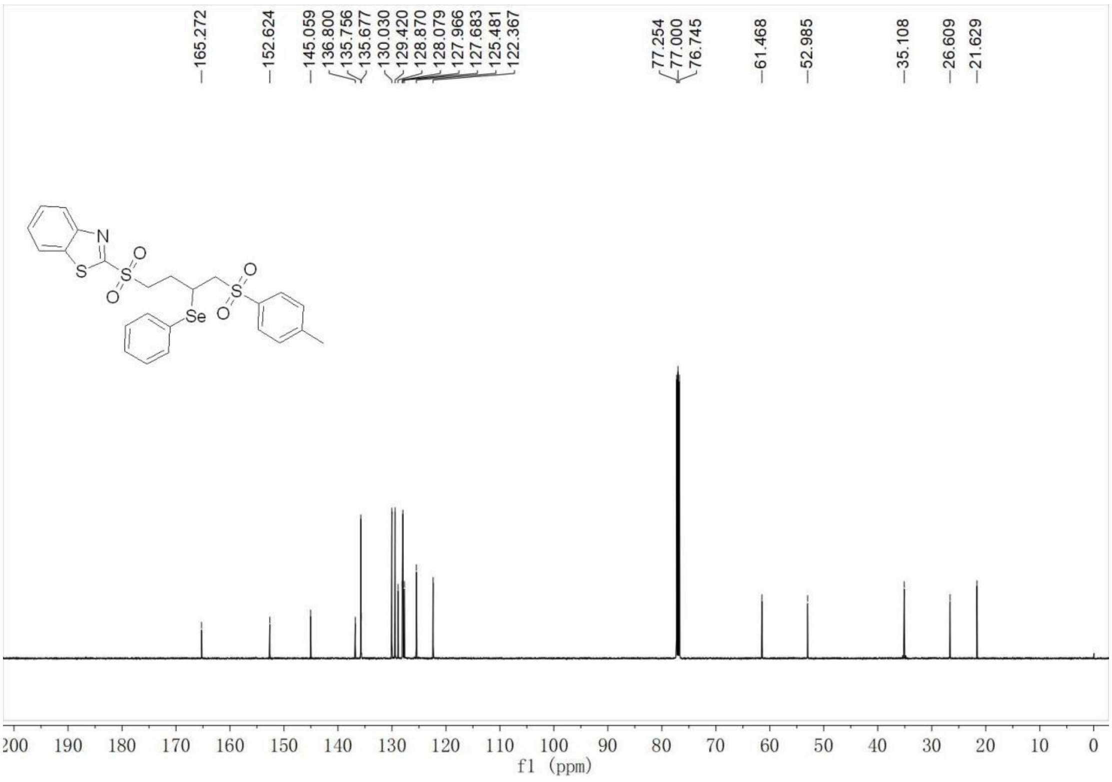


2-((3-(Phenylselanyl)-4-tosylbutyl)sulfonyl)benzo[*d*]thiazole (5v)

^{1}H NMR (500 MHz, CDCl$_3$) of **5v**

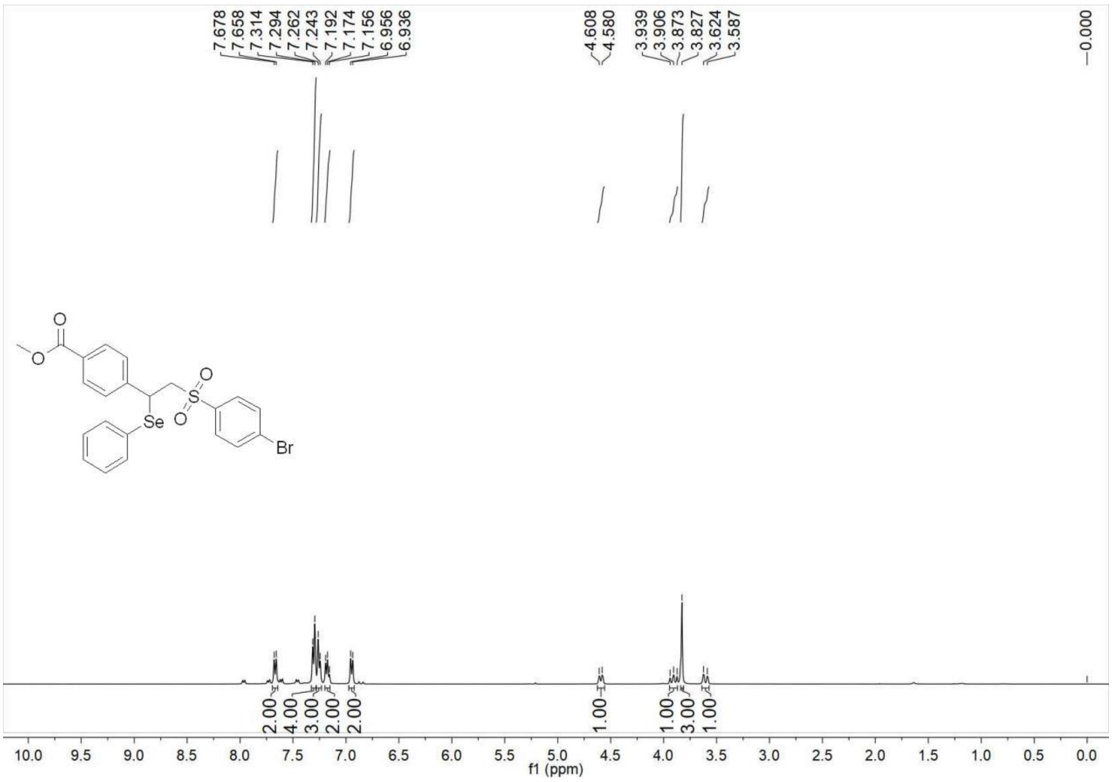


^{13}C NMR (126 MHz, CDCl$_3$) of **5v**

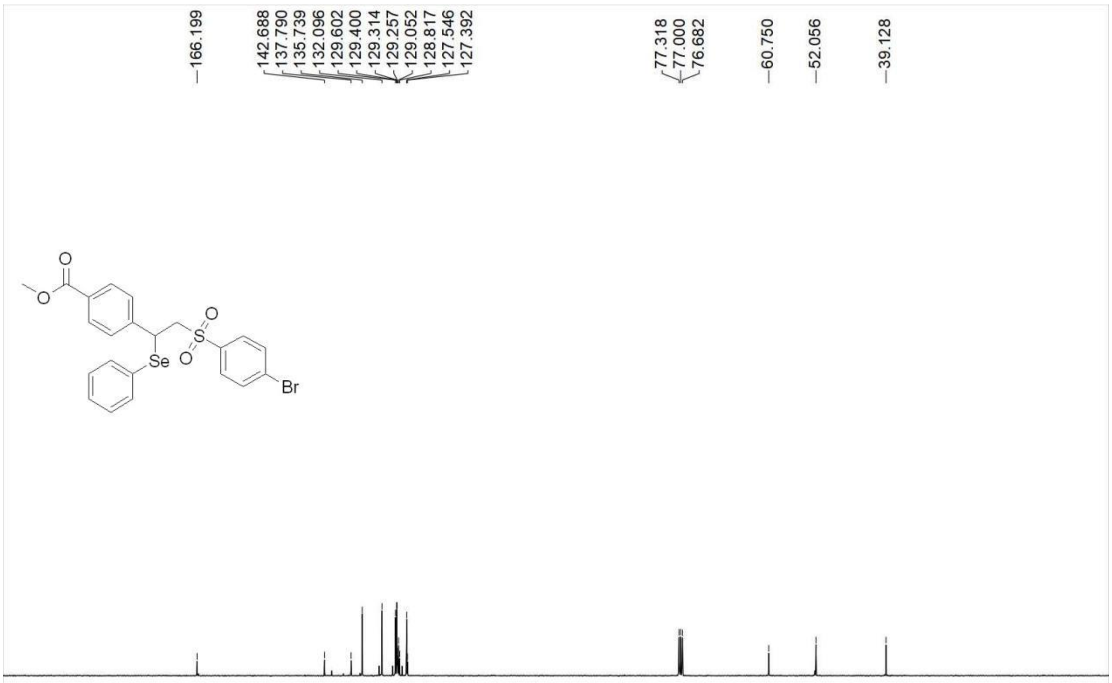


Methyl-4-(2-((4-bromophenyl)sulfonyl)-1-(phenylselanyl)ethyl)benzoate (5w)

^{1}H NMR (400 MHz, $CDCl_3$) of **5w**

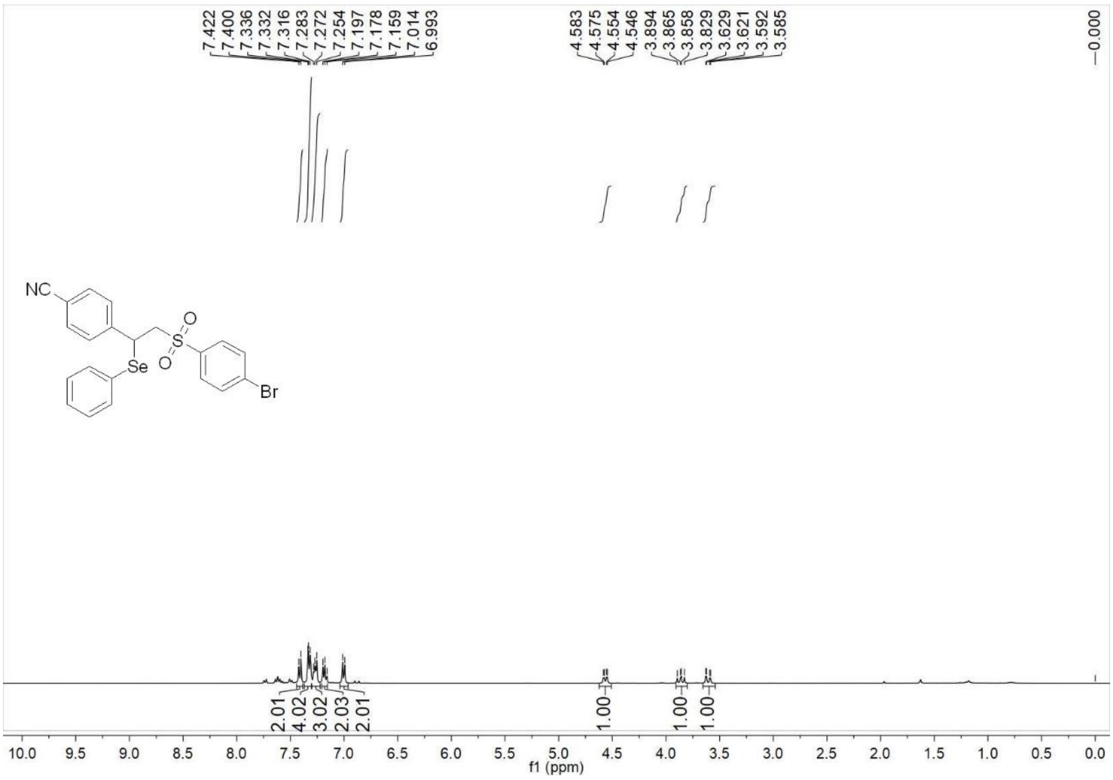


^{13}C NMR (101 MHz, $CDCl_3$) of **5w**

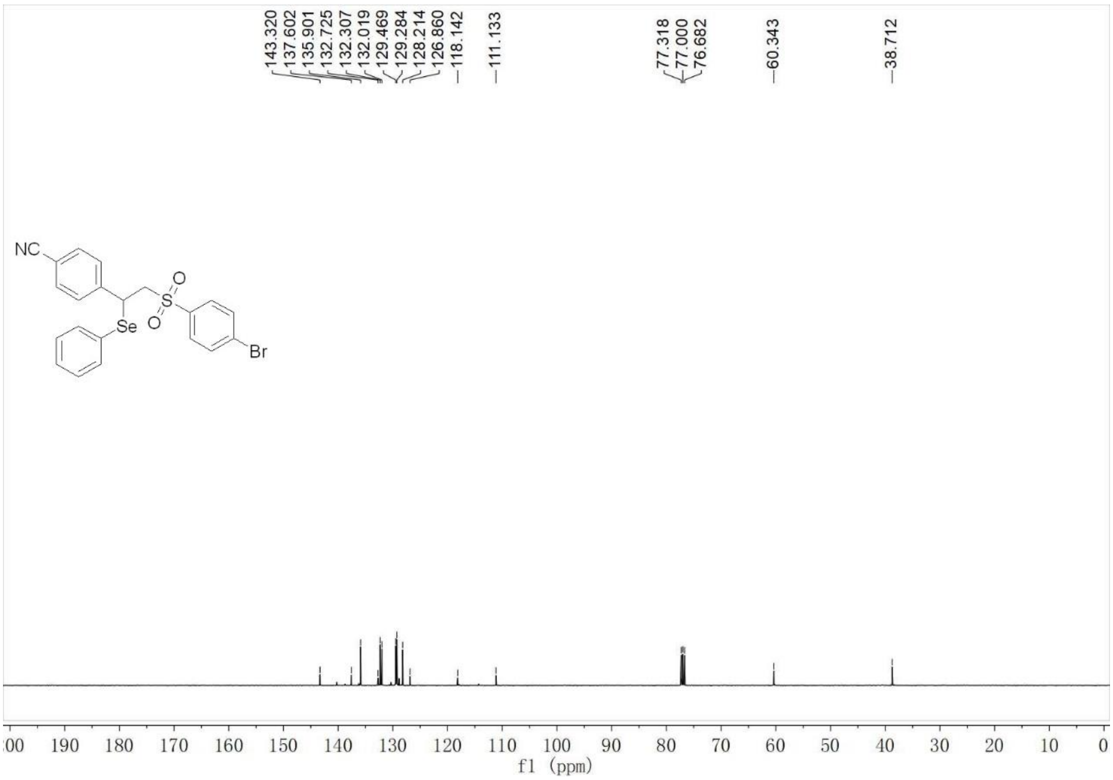


4-(2-((4-Bromophenyl)sulfonyl)-1-(phenylselanyl)ethyl)benzonitrile (5x)

^{1}H NMR (400 MHz, $CDCl_3$) of **5x**

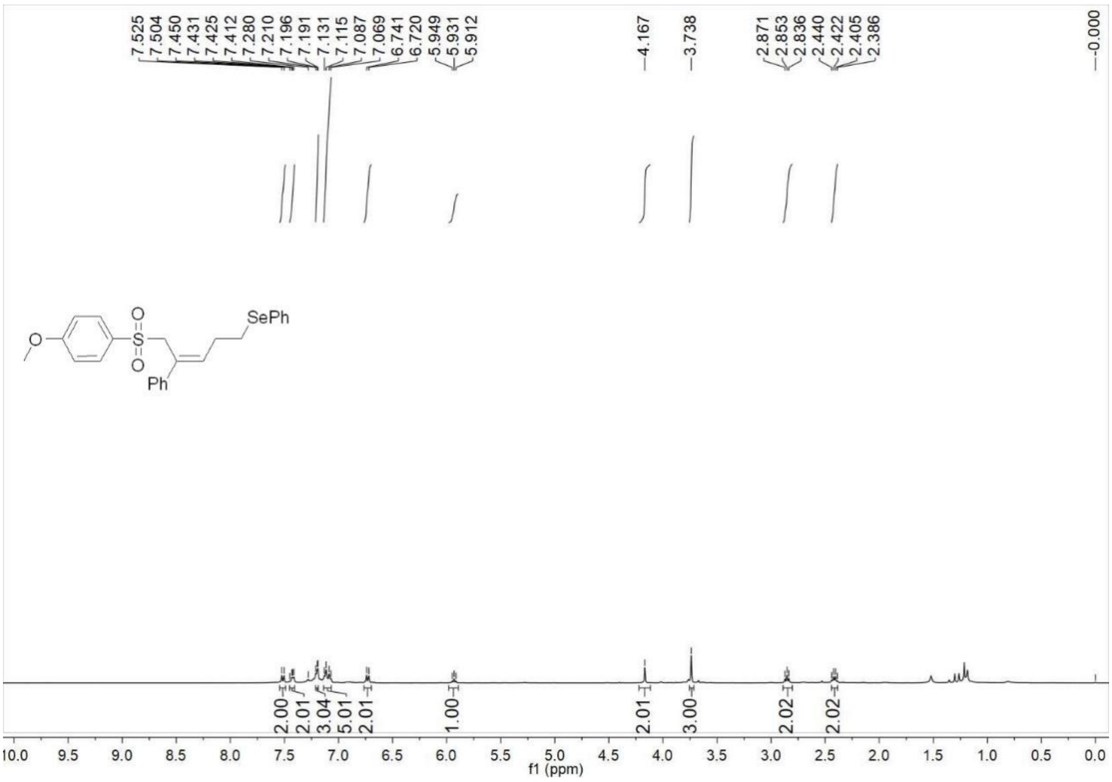


^{13}C NMR (101 MHz, $CDCl_3$) of **5x**

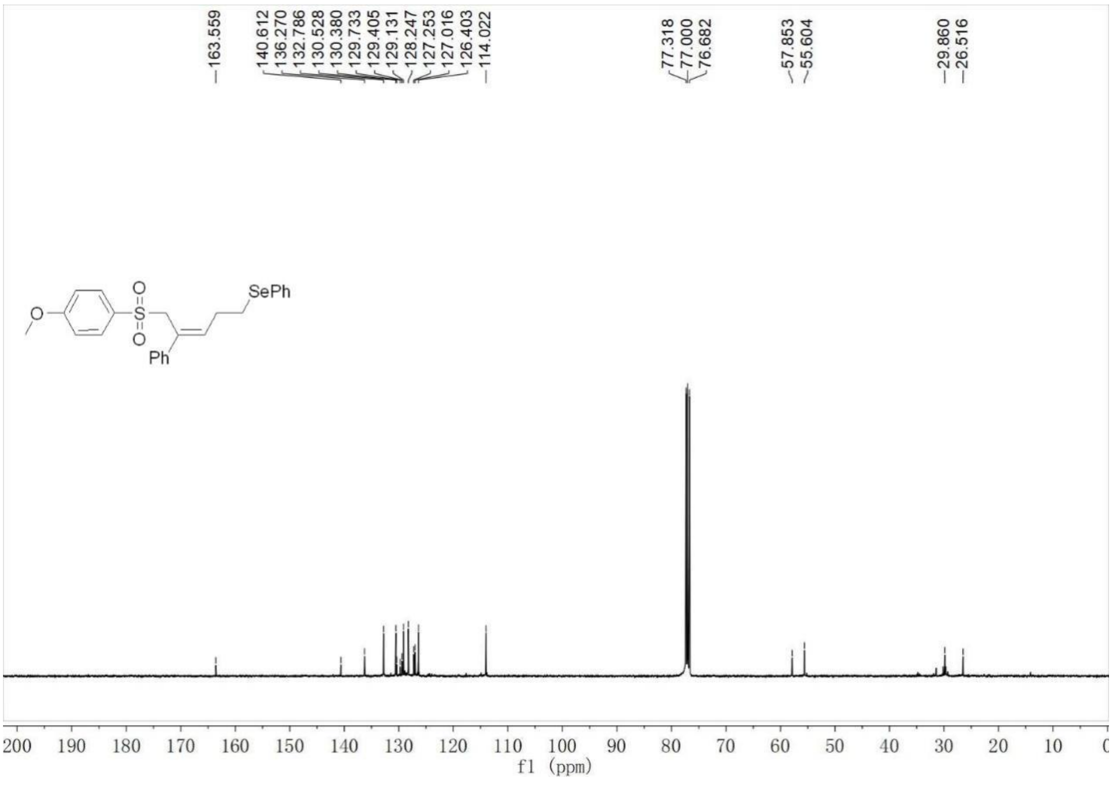


(5-((4-Methoxyphenyl)sulfonyl)-4-phenylpent-3-en-1-yl)(phenyl)selane (14)

^{1}H NMR (400 MHz, $CDCl_3$) of **14**

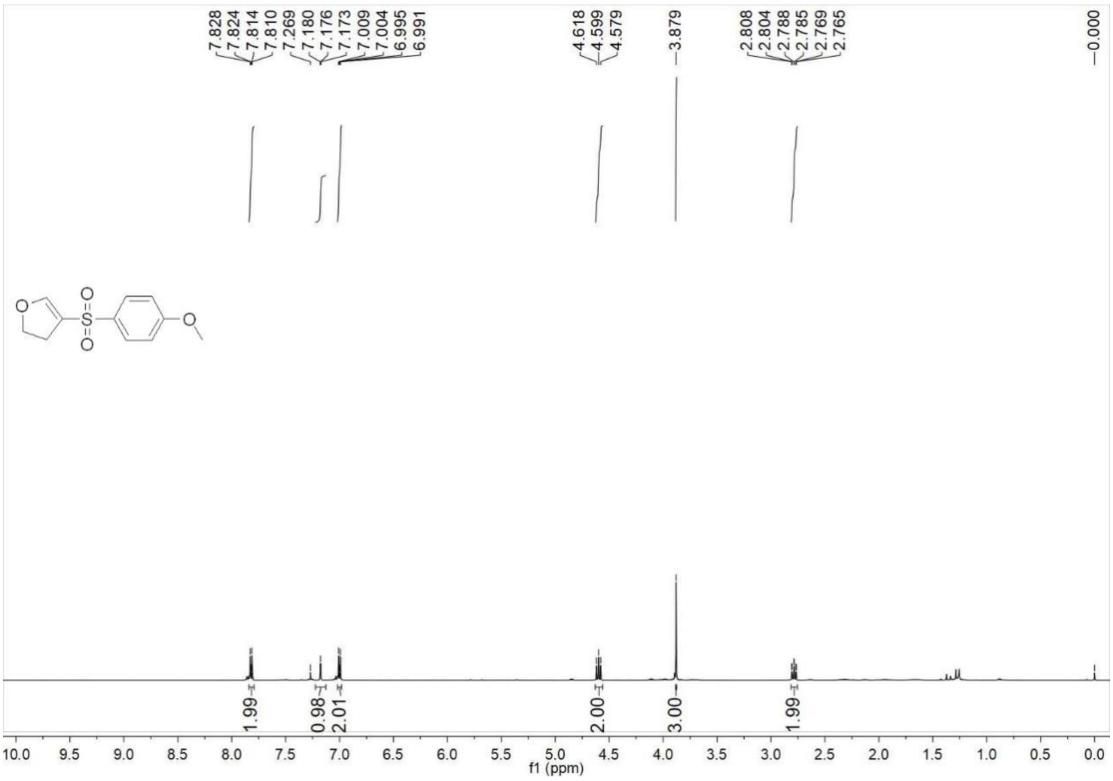


^{13}C NMR (101 MHz, $CDCl_3$) of **14**

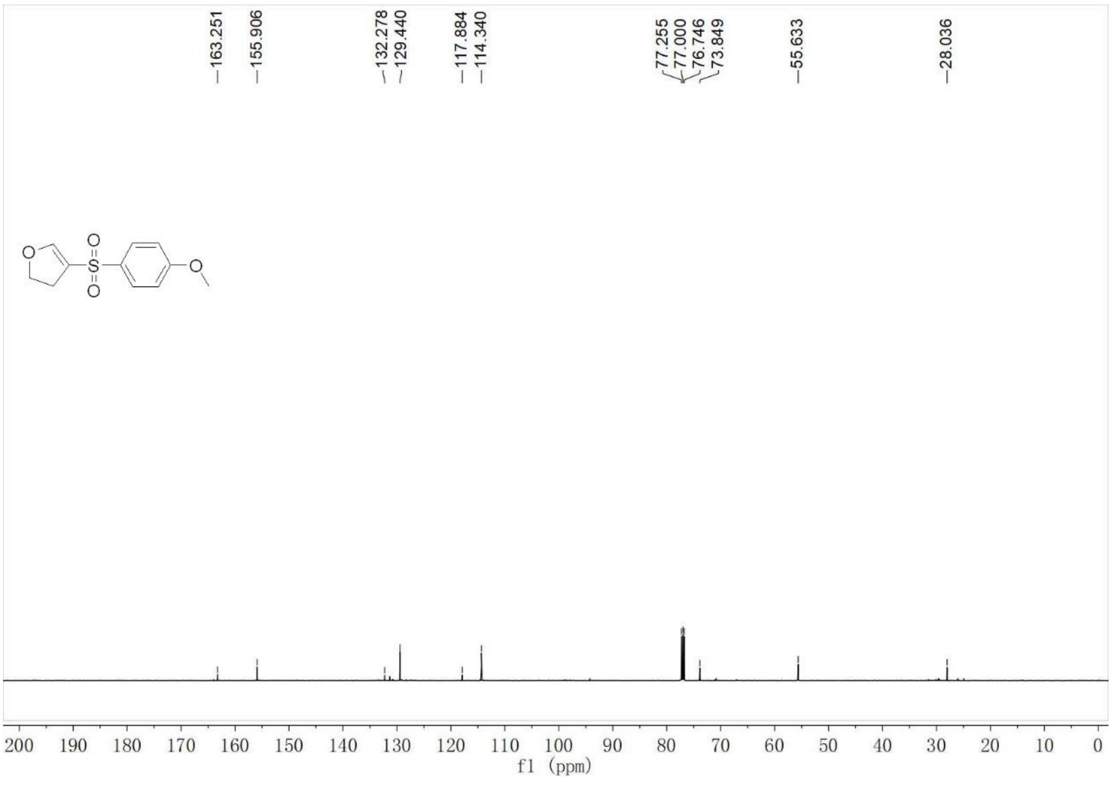


4-((4-Methoxyphenyl)sulfonyl)-2,3-dihydrofuran (15)

^{1}H NMR (500 MHz, CDCl$_3$) of **15**

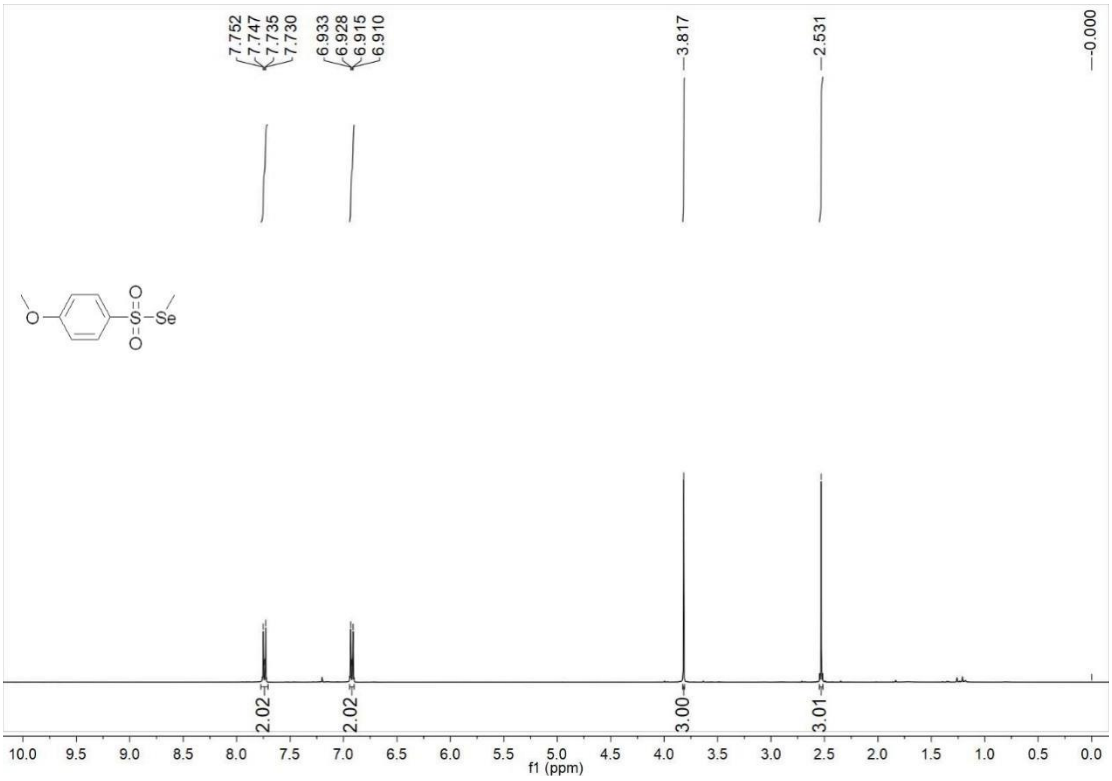


^{13}C NMR (126 MHz, CDCl$_3$) of **15**

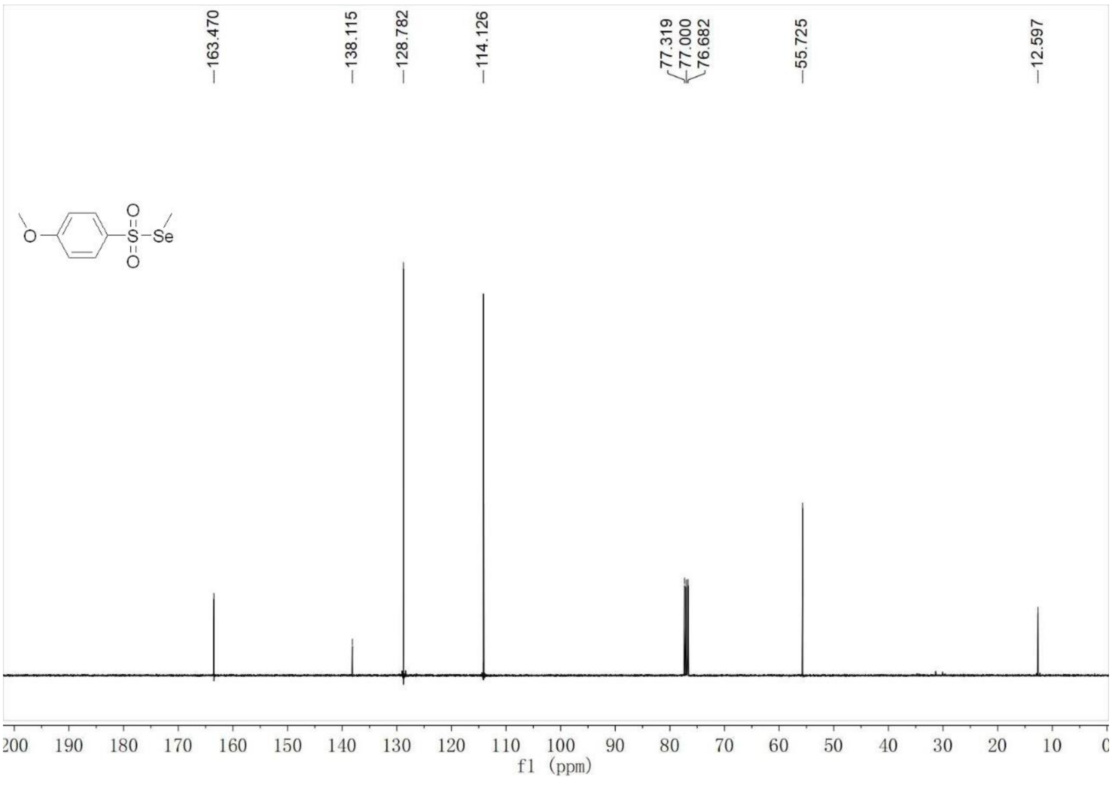


Se-Methyl 4-methoxybenzenesulfonoselenoate (16)

^{1}H NMR (400 MHz, CDCl$_3$) of **16**

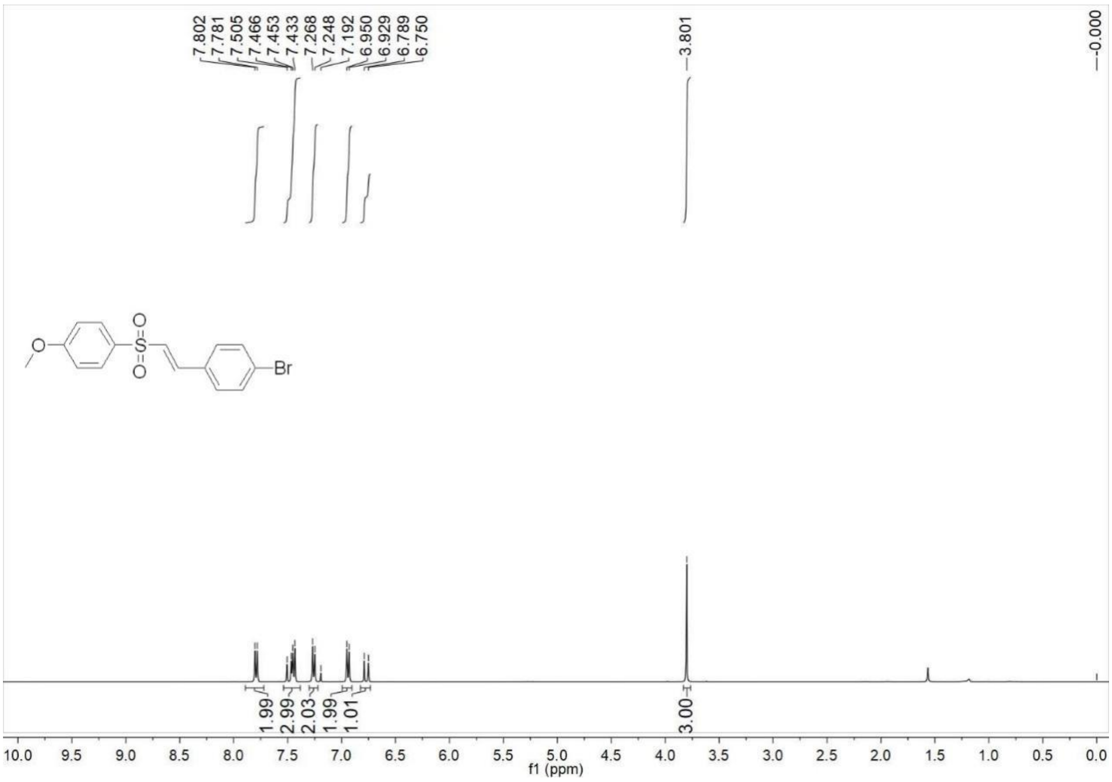


^{13}C NMR (101 MHz, CDCl$_3$) of **16**

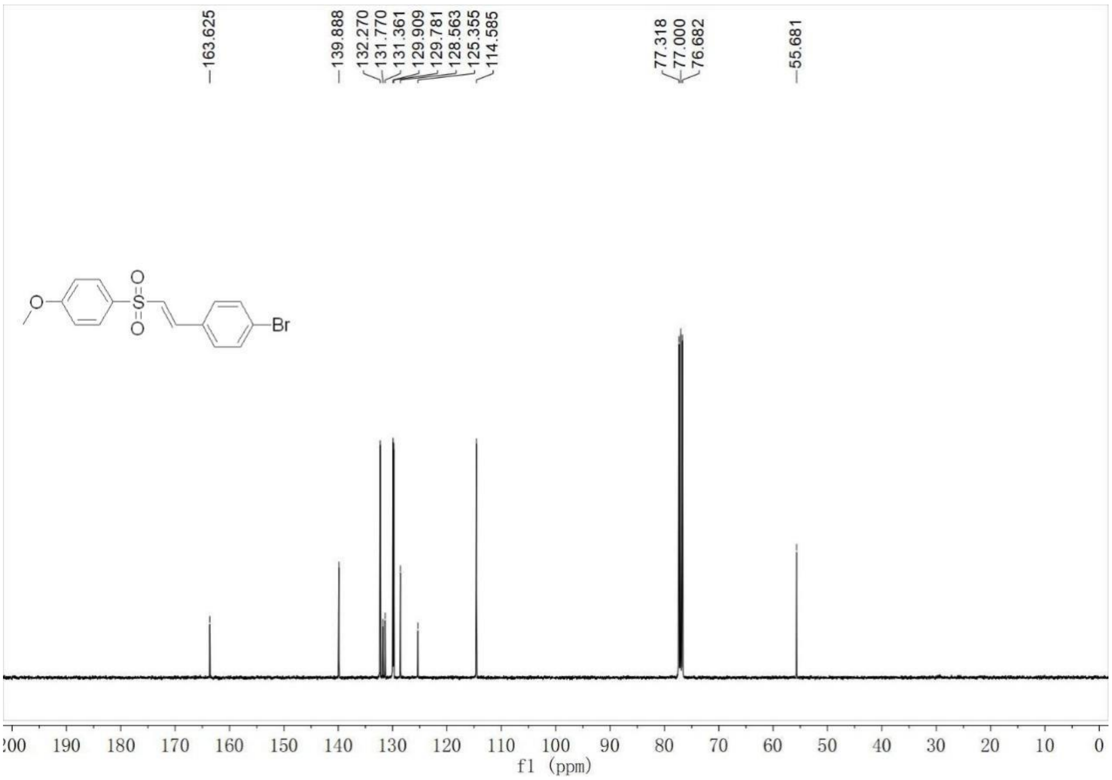


(*E*)-1-Bromo-4-(2-((4-methoxyphenyl)sulfonyl)vinyl)benzene (17)

^{1}H NMR (400 MHz, CDCl$_3$) of **17**



^{13}C NMR (101 MHz, CDCl$_3$) of **17**



(H) The *X*-ray single-crystal diffraction analysis of product 5p and 5v

1.1 The *X*-ray single-crystal diffraction analysis of product 5p

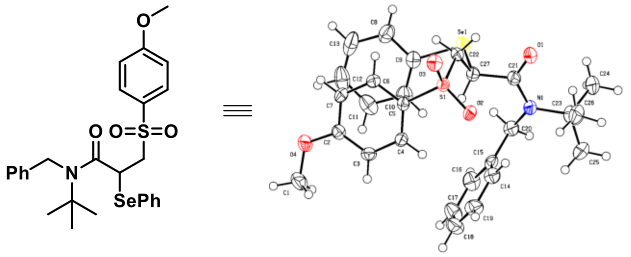


Table 3 Crystal data and structure refinement for **5p.**

Identification code	**5p**
Empirical formula	$C_{27}H_{31}NO_4SSe$
Formula weight	544.55
Temperature/K	173
Crystal system	triclinic
Space group	P-1
a/Å	10.3235(6)
b/Å	10.9307(6)
c/Å	13.9260(8)
α/°	67.930(2)
β/°	71.694(2)
γ/°	66.015(2)
Volume/Å^3	1306.17(13)
Z	2
ρ_{calc}g/cm^3	1.385
μ/mm^{-1}	1.550
F(000)	564.0
Crystal size/mm^3	0.12×0.06×0.04
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.256 to 52.78
Index ranges	$-12\leq h\leq 12$, $-12\leq k\leq 13$, $-16\leq l\leq 17$
Reflections collected	15201
Independent reflections	5338 [R_{int} = 0.0524, R_{sigma} = 0.0607]
Data/restraints/parameters	5338/0/311
Goodness-of-fit on F^2	1.027
Final R indexes [I>=2σ (I)]	R_1= 0.0400, wR_2= 0.0863
Final R indexes [all data]	R_1= 0.0569, wR_2= 0.0940
Largest diff. peak/hole / e Å^{-3}	0.47/-0.49

Table 4. Fractional atomic coordinates (×10^4) and equivalent isotropic displacement parameters (Å^2×10^3) for **5p**

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Se1	5784.0(3)	5860.0(3)	1300.3(2)	35.89(11)
S1	6220.1(6)	2828.1(6)	4457.6(5)	25.19(15)
O2	7291.1(18)	1596.2(18)	4196.2(14)	31.3(4)
O3	6206.5(19)	3102.7(19)	5397.3(14)	33.1(4)
O1	9085(2)	3864(2)	2162.0(15)	37.7(5)
O4	386.6(19)	2810(2)	4930.0(17)	41.8(5)
N1	8759(2)	2639(2)	1298.8(17)	29.7(5)
C27	6652(3)	4067(2)	2324.2(19)	26.9(6)
C21	8284(3)	3521(3)	1906(2)	29.0(6)
C22	6376(3)	4321(3)	3380.2(19)	28.2(6)
C5	4504(3)	2805(2)	4523.8(18)	23.8(5)
C4	4340(3)	1767(3)	4287(2)	27.5(6)
C3	2968(3)	1736(3)	4401(2)	30.0(6)
C2	1772(3)	2764(3)	4744(2)	28.6(6)
C15	7122(3)	1371(3)	1370.7(19)	28.5(6)
C9	3819(3)	5871(3)	1799(2)	31.9(6)
C7	1950(3)	3839(3)	4949(2)	31.9(6)
C6	3303(3)	3861(3)	4840(2)	29.1(6)
C20	7745(3)	2558(3)	801(2)	31.7(6)
C14	7525(3)	330(3)	2264(2)	34.5(6)
C16	6088(3)	1332(3)	947(2)	41.2(7)
C10	3398(3)	4878(3)	1694(2)	41.6(7)
C23	10371(3)	1977(3)	968(2)	39.2(7)
C8	2802(3)	6930(3)	2218(2)	45.4(8)
C19	6934(3)	-746(3)	2719(2)	41.3(7)
C24	11027(3)	3080(3)	197(3)	53.1(9)
C18	5929(3)	-777(3)	2287(3)	47.7(8)
C26	11061(3)	1217(3)	1945(3)	51.9(8)
C1	125(3)	1651(3)	4875(3)	49.1(8)
C11	1965(3)	4943(4)	2013(2)	49.0(8)
C17	5500(3)	263(4)	1406(3)	51.9(8)
C25	10662(4)	890(4)	413(3)	56.6(9)
C12	954(4)	6009(4)	2405(2)	54.6(9)
C13	1359(4)	6994(4)	2509(3)	59.2(10)

Table 5. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **5p**
The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Se1	31.06(17)	28.32(16)	35.23(17)	2.68(11)	-3.72(12)	-9.76(12)
S1	21.3(3)	24.2(3)	26.7(3)	-5.8(2)	-3.7(2)	-6.1(3)
O2	21.7(9)	26.4(9)	39.7(11)	-9.9(8)	-5.7(8)	-1.8(7)
O3	33.4(10)	38.1(11)	29.2(10)	-9.5(8)	-9.5(8)	-10.6(8)
O1	26.9(10)	43.2(12)	45.1(12)	-19.3(9)	2.1(8)	-14.2(9)
O4	23.4(10)	38.6(11)	63.9(14)	-14.3(10)	-12.2(9)	-8.0(9)
N1	26.7(12)	29.5(12)	30.0(12)	-9.6(9)	1.3(9)	-10.5(9)
C27	24.7(14)	23.4(13)	25.8(13)	-2.5(10)	-3.0(10)	-6.9(10)
C21	27.0(14)	25.2(13)	28.2(14)	-2.5(10)	-1.8(11)	-9.3(11)
C22	24.8(14)	25.9(13)	31.9(14)	-7.1(10)	0.1(11)	-11.4(11)
C5	21.4(13)	23.1(12)	21.0(12)	-2.5(9)	-2.9(10)	-6.0(10)
C4	24.3(14)	25.6(13)	28.9(14)	-8.5(10)	-2.4(10)	-6.1(11)
C3	32.8(15)	23.7(13)	33.9(15)	-7.5(11)	-7.1(11)	-9.7(11)
C2	23.8(14)	26.5(13)	31.8(14)	-2.7(10)	-9.1(11)	-7.0(11)
C15	27.9(14)	26.9(13)	24.0(13)	-8.8(10)	1.3(10)	-5.9(11)
C9	30.2(15)	32.0(14)	25.2(13)	-0.6(11)	-6.7(11)	-7.9(12)
C7	24.3(14)	24.2(13)	40.6(16)	-10.5(11)	-5.6(11)	-0.9(11)
C6	28.4(14)	23.0(13)	33.0(14)	-7.9(10)	-3.6(11)	-7.4(11)
C20	37.3(16)	31.1(14)	23.4(13)	-5.9(11)	-2.0(11)	-12.5(12)
C14	38.1(16)	35.0(15)	29.2(14)	-8.8(11)	-5.5(12)	-12.0(13)
C16	37.1(17)	44.1(17)	38.3(17)	-4.2(13)	-14.8(13)	-10.6(14)
C10	37.3(17)	44.6(18)	39.4(17)	-9.3(13)	-9.2(13)	-10.9(14)
C23	26.5(15)	40.8(16)	44.1(17)	-17.8(13)	6.6(12)	-9.9(13)
C8	39.9(18)	36.8(17)	45.2(18)	-10.3(13)	-3.3(14)	-3.7(14)
C19	54(2)	32.2(16)	32.0(16)	-4.0(12)	-3.1(13)	-17.1(14)
C24	40.9(19)	56(2)	56(2)	-23.3(16)	19.6(15)	-24.3(16)
C18	52(2)	45.7(19)	49.4(19)	-12.5(15)	1.3(15)	-29.7(16)
C26	28.2(17)	46.9(19)	69(2)	-18.9(16)	-7.5(15)	0.1(14)
C1	33.1(17)	48.6(19)	71(2)	-14.0(16)	-13.4(15)	-18.9(15)
C11	45(2)	55(2)	46.7(19)	1.0(15)	-15.9(15)	-25.7(17)
C17	40.5(19)	62(2)	60(2)	-13.8(17)	-13.4(16)	-24.1(16)
C25	45(2)	49(2)	73(2)	-36.6(18)	12.2(17)	-12.5(16)
C12	33.6(18)	68(2)	38.8(18)	7.7(16)	-5.5(14)	-15.7(17)
C13	36.5(19)	55(2)	53(2)	-11.5(17)	4.7(15)	3.5(16)

Table 6. Bond Lengths for **5p**

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Se1	C27	1.988(2)	C2	C7	1.399(4)
Se1	C9	1.923(3)	C15	C20	1.520(3)
S1	O2	1.4399(18)	C15	C14	1.375(4)
S1	O3	1.4433(19)	C15	C16	1.393(4)
S1	C22	1.778(2)	C9	C10	1.390(4)
S1	C5	1.755(2)	C9	C8	1.384(4)
O1	C21	1.220(3)	C7	C6	1.365(4)
O4	C2	1.354(3)	C14	C19	1.392(4)
O4	C1	1.431(3)	C16	C17	1.383(4)
N1	C21	1.361(3)	C10	C11	1.384(4)
N1	C20	1.470(3)	C23	C24	1.517(4)
N1	C23	1.515(3)	C23	C26	1.526(4)
C27	C21	1.537(4)	C23	C25	1.534(4)
C27	C22	1.517(4)	C8	C13	1.395(5)
C5	C4	1.377(3)	C19	C18	1.369(4)
C5	C6	1.392(3)	C18	C17	1.371(4)
C4	C3	1.389(4)	C11	C12	1.370(5)
C3	C2	1.386(4)	C12	C13	1.370(5)

Table 7. Bond Angles for **5p**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C9	Se1	C27	96.71(10)	C14	C15	C20	124.4(2)
O2	S1	O3	118.78(11)	C14	C15	C16	118.2(2)
O2	S1	C22	108.94(11)	C16	C15	C20	117.4(2)
O2	S1	C5	108.66(11)	C10	C9	Se1	121.0(2)
O3	S1	C22	107.21(12)	C8	C9	Se1	119.1(2)
O3	S1	C5	108.62(11)	C8	C9	C10	119.9(3)
C5	S1	C22	103.56(11)	C6	C7	C2	120.2(2)
C2	O4	C1	118.4(2)	C7	C6	C5	119.6(2)
C21	N1	C20	120.1(2)	N1	C20	C15	116.7(2)
C21	N1	C23	118.7(2)	C15	C14	C19	120.8(3)
C20	N1	C23	120.1(2)	C17	C16	C15	120.7(3)
C21	C27	Se1	108.89(15)	C11	C10	C9	120.1(3)
C22	C27	Se1	108.90(16)	N1	C23	C24	110.1(2)
C22	C27	C21	110.1(2)	N1	C23	C26	109.5(2)
O1	C21	N1	123.7(2)	N1	C23	C25	110.0(2)
O1	C21	C27	118.5(2)	C24	C23	C26	111.8(3)
N1	C21	C27	117.8(2)	C24	C23	C25	107.9(3)
C27	C22	S1	112.12(17)	C26	C23	C25	107.6(3)
C4	C5	S1	121.39(19)	C9	C8	C13	119.0(3)
C4	C5	C6	120.7(2)	C18	C19	C14	120.3(3)
C6	C5	S1	117.9(2)	C19	C18	C17	119.6(3)
C5	C4	C3	120.0(2)	C12	C11	C10	120.0(3)
C2	C3	C4	119.3(2)	C18	C17	C16	120.3(3)
O4	C2	C3	124.8(2)	C11	C12	C13	120.3(3)
O4	C2	C7	115.1(2)	C12	C13	C8	120.7(3)
C3	C2	C7	120.1(2)				

Table 8. Torsion Angles for **5p**.

A	B	C	D	Angle/˚	A	B	C	D	Angle/˚
Se1	C27	C21	O1	90.7(2)	C2	C7	C6	C5	-0.1(4)
Se1	C27	C21	N1	-91.5(2)	C15	C14	C19	C18	-0.7(4)
Se1	C27	C22	S1	149.42(13)	C15	C16	C17	C18	0.0(5)
Se1	C9	C10	C11	-176.9(2)	C9	C10	C11	C12	1.8(4)
Se1	C9	C8	C13	175.4(2)	C9	C8	C13	C12	1.4(5)
S1	C5	C4	C3	176.55(19)	C6	C5	C4	C3	-2.5(4)
S1	C5	C6	C7	-176.8(2)	C20	N1	C21	O1	-164.2(2)
O2	S1	C22	C27	40.8(2)	C20	N1	C21	C27	18.1(3)
O2	S1	C5	C4	-3.5(2)	C20	N1	C23	C24	99.4(3)
O2	S1	C5	C6	175.65(18)	C20	N1	C23	C26	-137.3(2)
O3	S1	C22	C27	170.52(17)	C20	N1	C23	C25	-19.3(3)
O3	S1	C5	C4	-134.0(2)	C20	C15	C14	C19	-177.9(3)
O3	S1	C5	C6	45.1(2)	C20	C15	C16	C17	178.3(3)
O4	C2	C7	C6	176.8(2)	C14	C15	C20	N1	-5.0(4)
C21	N1	C20	C15	-97.5(3)	C14	C15	C16	C17	-0.9(4)
C21	N1	C23	C24	-68.3(3)	C14	C19	C18	C17	-0.3(5)
C21	N1	C23	C26	54.9(3)	C16	C15	C20	N1	175.9(2)
C21	N1	C23	C25	172.9(2)	C16	C15	C14	C19	1.2(4)
C21	C27	C22	S1	-91.3(2)	C10	C9	C8	C13	-1.3(4)
C22	S1	C5	C4	112.3(2)	C10	C11	C12	C13	-1.7(5)
C22	S1	C5	C6	-68.6(2)	C23	N1	C21	O1	3.6(4)
C22	C27	C21	O1	-28.6(3)	C23	N1	C21	C27	-174.1(2)
C22	C27	C21	N1	149.2(2)	C23	N1	C20	C15	94.9(3)
C5	S1	C22	C27	-74.74(19)	C8	C9	C10	C11	-0.3(4)
C5	C4	C3	C2	0.6(4)	C19	C18	C17	C16	0.6(5)
C4	C5	C6	C7	2.3(4)	C1	O4	C2	C3	6.7(4)
C4	C3	C2	O4	-177.0(2)	C1	O4	C2	C7	-171.8(3)
C4	C3	C2	C7	1.5(4)	C11	C12	C13	C8	0.1(5)
C3	C2	C7	C6	-1.8(4)					

Table 9. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for **5p**.

Atom	*x*	*y*	*z*	U(eq)
H27	6202.44	3367.81	2406.87	32
H22A	5475.56	5117.26	3448.51	34
H22B	7179.44	4575.66	3405.97	34
H4	5166.56	1072.91	4044.66	33
H3	2849.24	1016.93	4244.88	36
H7	1127.41	4556.6	5165.76	38
H6	3424.05	4592.21	4977.51	35
H20A	8246.15	2476.14	82.57	38
H20B	6932.27	3450.38	720.78	38
H14	8214.25	346.44	2573.98	41
H16	5783.16	2046.75	335.86	49
H10	4093.65	4153.76	1403.86	50
H8	3082.46	7602.08	2306.38	54
H19	7230.38	-1462.83	3331.41	50
H24A	10543.87	3559.29	-411.66	80
H24B	12059.06	2633.91	-43.16	80
H24C	10903.37	3759.56	546.11	80
H18	5530.73	-1514.71	2597.38	57
H26A	11006.11	1901.22	2264.43	78
H26B	12075.03	678.14	1742.2	78
H26C	10545.44	583.5	2455.44	78
H1A	382.6	1631.37	4139.94	74
H1B	-899.1	1747.22	5145.58	74
H1C	713.5	780.05	5302.63	74
H11	1682.8	4248.87	1959.49	59
H17	4795.27	250.22	1108.93	62
H25A	10155.95	222.92	866.2	85
H25B	11702.52	391.41	273.87	85
H25C	10315.23	1361.23	-255.98	85
H12	-32.89	6065.82	2604.66	65
H13	649.53	7727.85	2783.1	71

1.2 The *X*-ray single-crystal diffraction analysis of product 5v

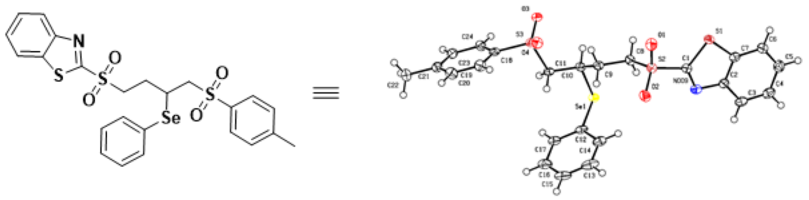


Table 10 Crystal data and structure refinement for **5v**.

Identification code	**5v**
Empirical formula	$C_{24}H_{23}NO_4S_3Se$
Formula weight	564.57
Temperature/K	100
Crystal system	triclinic
Space group	P-1
a/Å	8.3353(5)
b/Å	12.5099(8)
c/Å	12.6251(8)
α/°	114.330(2)
β/°	91.596(2)
γ/°	92.594(2)
Volume/Å^3	1196.75(13)
Z	2
ρcalcg/cm^3	1.567
μ/mm-1	1.863
F(000)	576.0
Crystal size/mm^3	0.15 × 0.08 × 0.05
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.858 to 53.054
Index ranges	$-9 \leq h \leq 10$, $-15 \leq k \leq 15$, $-15 \leq l \leq 15$
Reflections collected	13663
Independent reflections	4938 [R_{int} = 0.0646, R_{sigma} = 0.0750]
Data/restraints/parameters	4938/0/299
Goodness-of-fit on F^2	1.079
Final R indexes [I>=2σ (I)]	R_1 = 0.0475, wR_2 = 0.1079
Final R indexes [all data]	R_1 = 0.0649, wR_2 = 0.1186
Largest diff. peak/hole / e Å^{-3}	0.57/-0.85

Table 11 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for **5v**.

U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	x	y	z	U(eq)
Se1	4961.6(4)	1995.3(3)	7533.3(3)	21.04(13)
S2	-386.5(11)	3083.9(8)	8224.7(8)	21.3(2)
S1	-2187.3(11)	2813.0(8)	10153.4(8)	22.2(2)
S3	5487.3(10)	5751.4(8)	7908.4(7)	19.5(2)
O3	5929(3)	6242(2)	9136(2)	23.8(6)
O4	4012(3)	6093(2)	7536(2)	25.0(6)
O1	-1151(3)	4177(2)	8759(2)	27.5(6)
O2	-570(3)	2433(3)	6985(2)	28.8(6)
N009	-895(4)	1076(3)	8516(2)	21.1(7)
C2	-1617(4)	619(3)	9229(3)	20.9(8)
C1	-1126(4)	2182(3)	8903(3)	19.2(7)
C7	-2374(4)	1435(3)	10177(3)	21.2(8)
C10	4448(4)	3672(3)	8054(3)	19.8(8)
C3	-1637(5)	-562(3)	9062(3)	24.8(8)
C24	8654(4)	6125(3)	7574(3)	23.0(8)
C11	5444(4)	4190(3)	7376(3)	18.2(7)
C18	7068(4)	6059(3)	7157(3)	21.0(8)
C9	2643(4)	3800(3)	7941(3)	20.4(8)
C12	4476(5)	1512(3)	5893(3)	23.0(8)
C19	6719(5)	6208(4)	6149(3)	26.6(9)
C8	1681(4)	3284(3)	8653(3)	21.5(8)
C4	-2417(5)	-896(4)	9840(3)	28.8(9)
C23	9892(5)	6344(3)	6960(3)	24.8(8)
C21	9582(5)	6505(3)	5952(3)	25.7(8)
C6	-3147(5)	1099(4)	10975(3)	27.8(9)
C16	5395(6)	1219(4)	4010(3)	33.1(10)
C17	5707(5)	1569(3)	5203(3)	25.7(8)
C20	7981(5)	6422(4)	5552(3)	29.7(9)
C14	2926(5)	1098(3)	5413(3)	29.7(9)
C5	-3147(5)	-61(4)	10796(3)	29.9(9)
C15	3852(6)	809(4)	3534(3)	37.0(11)
C22	10923(5)	6762(4)	5305(4)	33.2(10)
C13	2626(6)	742(4)	4223(4)	37.7(11)

Table 12 Anisotropic Displacement Parameters (Å^2×10^3) for **5v**.

The Anisotropic displacement factor exponent takes the form:

$-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Se1	23.8(2)	17.1(2)	20.6(2)	5.82(15)	1.84(14)	4.15(15)
S2	20.5(4)	22.8(5)	20.8(4)	9.1(4)	0.8(4)	2.6(4)
S1	25.8(5)	19.0(5)	19.6(4)	5.2(4)	5.4(4)	4.7(4)
S3	20.1(4)	15.9(5)	20.3(4)	4.8(4)	2.5(3)	3.7(4)
O3	26.7(13)	18.8(14)	20.5(13)	2.8(11)	3.1(11)	0.1(11)
O4	19.8(13)	24.8(15)	31.1(14)	11.4(12)	4.2(11)	8.1(11)
O1	25.6(14)	25.3(15)	36.3(15)	16.7(12)	4.2(12)	7.8(12)
O2	26.7(14)	38.9(17)	20.3(13)	12.4(12)	-1.5(11)	-3.2(12)
N009	20.3(15)	20.0(17)	20.9(15)	6.2(13)	-1.4(12)	5.2(13)
C2	17.8(17)	23(2)	19.8(17)	6.9(15)	-2.2(14)	-1.5(15)
C1	17.2(17)	21(2)	18.4(17)	6.9(15)	0.0(14)	3.7(15)
C7	21.6(18)	20(2)	21.2(18)	7.6(15)	-2.2(15)	1.5(15)
C10	23.8(18)	17.5(19)	17.2(17)	6.0(15)	2.0(14)	3.8(15)
C3	27(2)	19(2)	25.9(19)	6.2(16)	-3.0(16)	4.8(16)
C24	23.5(19)	24(2)	22.7(18)	9.8(16)	4.1(15)	4.7(16)
C11	18.1(17)	14.2(18)	19.2(17)	3.4(14)	0.1(14)	5.3(14)
C18	22.9(18)	13.4(18)	21.9(18)	2.1(15)	2.4(15)	4.1(15)
C9	18.8(17)	18.6(19)	22.4(18)	6.6(15)	1.4(14)	4.3(15)
C12	29(2)	13.5(18)	21.0(18)	1.9(15)	-3.3(15)	4.8(15)
C19	22.1(19)	30(2)	28(2)	11.4(17)	-0.2(16)	2.8(17)
C8	17.9(17)	27(2)	19.9(17)	9.7(16)	0.8(14)	2.4(15)
C4	36(2)	20(2)	30(2)	10.3(17)	-5.9(17)	-0.5(17)
C23	24.4(19)	22(2)	25.9(19)	8.4(16)	0.9(16)	0.1(16)
C21	29(2)	19(2)	29(2)	8.6(16)	6.7(16)	1.1(16)
C6	32(2)	24(2)	25.6(19)	8.4(17)	6.5(17)	1.8(17)
C16	49(3)	22(2)	25(2)	5.7(17)	1.0(19)	7.2(19)
C17	33(2)	17(2)	22.7(19)	3.9(16)	-1.4(16)	3.6(16)
C20	32(2)	33(2)	26(2)	14.2(18)	0.5(17)	6.3(18)
C14	29(2)	20(2)	33(2)	3.1(17)	-2.4(17)	5.7(17)
C5	32(2)	32(2)	28(2)	15.5(18)	-1.0(17)	-2.9(18)
C15	62(3)	19(2)	22(2)	0.9(17)	-9(2)	12(2)
C22	35(2)	34(2)	32(2)	14.7(19)	9.4(18)	-3.1(19)
C13	40(2)	25(2)	34(2)	-0.9(18)	-16(2)	8.7(19)

Table 13 Bond Lengths for **5v**.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Se1	C10	1.994(4)	C10	C9	1.529(5)
Se1	C12	1.928(4)	C3	C4	1.382(6)
S2	O1	1.440(3)	C24	C18	1.395(5)
S2	O2	1.436(3)	C24	C23	1.390(5)
S2	C1	1.774(4)	C18	C19	1.384(5)
S2	C8	1.767(4)	C9	C8	1.529(5)
S1	C1	1.736(3)	C12	C17	1.383(5)
S1	C7	1.737(4)	C12	C14	1.394(5)
S3	O3	1.442(3)	C19	C20	1.390(6)
S3	O4	1.448(3)	C4	C5	1.406(6)
S3	C11	1.783(4)	C23	C21	1.388(5)
S3	C18	1.763(4)	C21	C20	1.398(6)
N009	C2	1.388(5)	C21	C22	1.502(5)
N009	C1	1.288(5)	C6	C5	1.373(6)
C2	C7	1.403(5)	C16	C17	1.397(5)
C2	C3	1.403(5)	C16	C15	1.387(6)
C7	C6	1.403(5)	C14	C13	1.391(6)
C10	C11	1.514(5)	C15	C13	1.382(7)

Table 14 Bond Angles for **5v**.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C12	Se1	C10	97.38(15)	C9	C10	Se1	111.9(2)
O1	S2	C1	106.13(16)	C4	C3	C2	118.8(3)
O1	S2	C8	110.40(18)	C23	C24	C18	118.9(3)
O2	S2	O1	119.01(16)	C10	C11	S3	115.0(2)
O2	S2	C1	108.78(17)	C24	C18	S3	119.5(3)
O2	S2	C8	109.07(16)	C19	C18	S3	119.5(3)
C8	S2	C1	102.07(17)	C19	C18	C24	121.0(4)
C1	S1	C7	87.71(18)	C10	C9	C8	111.9(3)
O3	S3	O4	118.26(15)	C17	C12	Se1	118.4(3)
O3	S3	C11	108.00(16)	C17	C12	C14	120.8(4)
O3	S3	C18	109.42(16)	C14	C12	Se1	120.7(3)
O4	S3	C11	109.60(16)	C18	C19	C20	118.8(4)
O4	S3	C18	107.97(17)	C9	C8	S2	111.2(2)
C18	S3	C11	102.44(16)	C3	C4	C5	120.6(4)
C1	N009	C2	109.2(3)	C21	C23	C24	121.5(4)
N009	C2	C7	115.1(3)	C23	C21	C20	118.1(4)
N009	C2	C3	125.2(3)	C23	C21	C22	121.2(4)
C3	C2	C7	119.8(3)	C20	C21	C22	120.6(4)
S1	C1	S2	118.8(2)	C5	C6	C7	117.8(4)
N009	C1	S2	122.9(3)	C15	C16	C17	119.3(4)
N009	C1	S1	118.2(3)	C12	C17	C16	119.8(4)
C2	C7	S1	109.8(3)	C19	C20	C21	121.6(4)
C2	C7	C6	121.4(4)	C13	C14	C12	119.1(4)
C6	C7	S1	128.9(3)	C6	C5	C4	121.6(4)
C11	C10	Se1	107.6(2)	C13	C15	C16	120.8(4)
C11	C10	C9	113.5(3)	C15	C13	C14	120.1(4)

Table 15 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å2×10^3) for **5v**.

Atom	*x*	*y*	*z*	U(eq)
H10	4801.14	4095.46	8895.29	24
H3	-1123.66	-1122.67	8425.65	30
H24	8884.67	6021.27	8265.43	28
H11A	5017.25	3844.76	6556.46	22
H11B	6561.2	3954.56	7381.05	22
H9A	2269.82	3395.09	7111.83	25
H9B	2442.6	4643.13	8208.71	25
H19	5636.52	6164.01	5871.35	32
H8A	1824.71	3818.98	9489.12	26
H8B	2094.8	2518.8	8546.13	26
H4	-2460	-1695.68	9727.9	35
H23	10973.94	6383.86	7237.73	30
H6	-3653.37	1655.08	11618.89	33
H16	6231.36	1261.06	3530.27	40
H17	6761.33	1845.04	5538.26	31
H20	7749.58	6513.55	4853.87	36
H14	2085.15	1060.33	5891.45	36
H5	-3653.87	-305.85	11330.88	36
H15	3634.11	570.63	2723	44
H22A	10926.41	7585.35	5416.92	50
H22B	10763.87	6249.25	4473.33	50
H22C	11952.93	6619.28	5601.24	50
H13	1576.47	451.47	3883.55	45