

## Supplementary Information

### **Green-Light-Induced selective hydroselenation of olefins with diselenides**

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## 1. General information

All reagents and solvents were purchased from TCI, Sigma-Aldrich, Alfa Aesar, Acros and Energy Chemical. All commercial reagents were used as supplied unless otherwise stated. Organic solutions were concentrated by rotary evaporation below 45 °C. All reactions were monitored by TLC, GC-MS. Analytical thin-layer chromatography was performed using Merck Kieselgel 60 F254 0.20 mm pre-coated glass-backed silica gel plates. Visualization of the chromatogram was performed by UV absorbance ( $\lambda_{\text{max}} = 254 \text{ nm}$ ) and/or by staining with aqueous potassium permanganate. Flash column chromatography was performed using silica gel (EM 60 F254 300 - 400 mesh) with the appropriate solvent system.

Proton nuclear magnetic resonance ( $^1\text{H}$  NMR) spectra were recorded on a Bruker DPX 400 (400 MHz) or Avance 500 (500 MHz) spectrometer. Chemical shifts ( $\delta$ ) are recorded in parts per million (ppm) and are quoted to the nearest 0.01 ppm relative to the residual solvent protons ( $\text{CDCl}_3 = 7.26 \text{ ppm}$ ). Coupling constants (J) are quoted in Hertz (Hz), and data reported as follows: Chemical shift (multiplicity, coupling constant, number of protons). Coupling constants were reported to the nearest 0.1 Hz and multiplicity reported according to the following: s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, m = multiplet, br = broad, with associated combinations e.g. dd = doublet of doublets

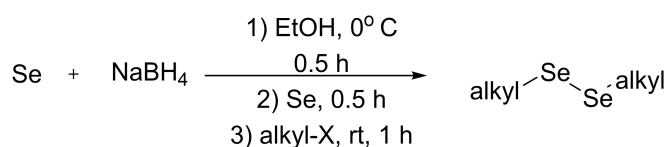
Carbon nuclear magnetic resonance ( $^{13}\text{C}$  NMR) spectra were recorded on a Bruker AVANCE 500 (125 MHz) spectrometer. Chemical shifts ( $\delta$ ) are recorded in parts per million (ppm) and are quoted to the nearest 0.1 ppm relative to the residual solvent protons ( $\text{CDCl}_3 = 77.2 \text{ ppm}$ ). High-resolution mass spectra were recorded on a micrOTOF-Q II 10410 mass spectrometer.

Unless otherwise noted, all reagents and solvents were obtained commercially and used without further purification.

## 2. Substrate preparation

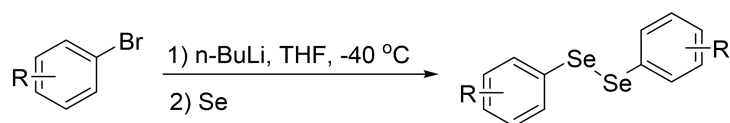


**General procedure I<sup>[1]</sup>:** Phenylboric acid (5 mmol), selenium powder (15 mmol), and silver nitrate (5 mmol) were added to DMSO solution (15 mL) and the reaction mixture was stirred in a heating mantle preheated to  $120 \text{ }^\circ\text{C}$  for 12 h. Then the reaction mixture was cooled to ambient temperature, and evaporated under reduced pressure. The residue was purified by column chromatography to give the desired diselenides.

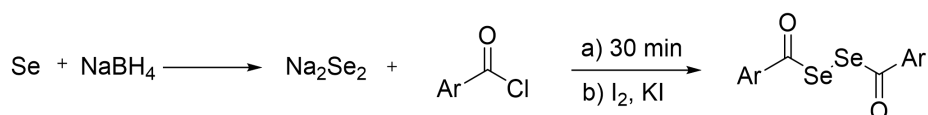


**General procedure II<sup>[2]</sup>:** Selenium (2 mmol) was added to a stirred solution of sodium borohydride (5 mmol) in ethanol (5 mL) at  $0 \text{ }^\circ\text{C}$ . Stirring was continued for 30 min at this temperature. Then, selenium (2 mmol) was added to the reaction mixture and stirred for 30 min at

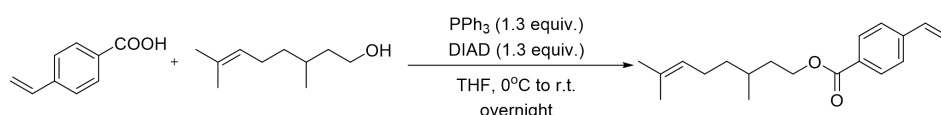
0 °C. Finally, iodides (7 mmol) was added over a period of 5 min. After stirring for a further hour at room temperature, the reaction mixture was extracted with hexane and washed with water, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated in vacuo.



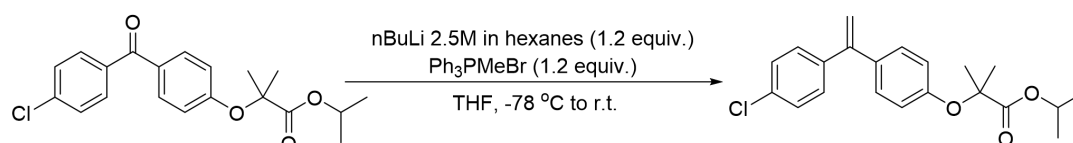
**General procedure III**<sup>[3]</sup>: n-Butyl lithium (8 mmol) was added to the THF (15 mL) solution of bromobenzene (2 mmol) at - 40 °C. Stirring was continued for 2 hours at this temperature. Then, selenium (8 mmol) was added to the reaction mixture and stirred for 2 hours at - 40 °C. After stirring for a further hour at room temperature, water was added to the reaction. the reaction was concentrated, and the residue was purified by silica gel column chromatography separation to give the desired diselenides.



**General procedure IV**<sup>[4]</sup>: Selenium (2 mmol) was added to a stirred solution of sodium borohydride (2.4 mmol) in ethanol (3 mL) at 0 °C. Stirring was continued for 30 min at this temperature. Then, aryl chloride (2 mmol) was added to the reaction mixture and stirred for 30 min at 0 °C. Stirring was continued for 30 min at this temperature. Finally, a solution of iodine (1 mmol) and potassium iodide (0.4 mmol) was added to the reaction mixture and stirred for 30 min at 0 °C. After reaction finished, the organic phase was washed with 1 % NaHCO<sub>3</sub> solution. The reaction mixture was then concentrated, and purified by silica gel column chromatography separation to give the desired diselenides.



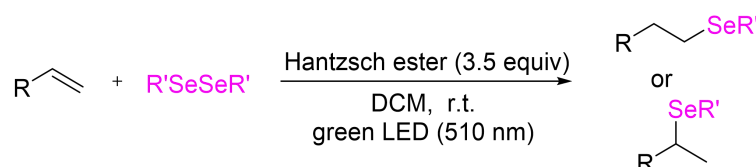
**General procedure V**<sup>[5]</sup>: A dried two-neck round flask were charged with Citronellol (0.47 g, 3.0 mmol, 1.0 equiv.), 4-vinylbenzoic acid (0.58 g, 3.9 mmol, 1.3 equiv.), and PPh<sub>3</sub> (1.02 g, 3.9 mmol, 1.3 equiv.). Dried THF (10 mL) was added to this flask under N<sub>2</sub> atmosphere at 0 °C. Then, DIAD (0.79 g, 3.9 mmol, 1.3 equiv.) was added dropwise and the resulting mixture was warmed to room temperature (14 °C). After the completion of the reaction, the resulting mixture was diluted with 20 mL of H<sub>2</sub>O and extracted with EA (30 mL × 3). The combined organic phase was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtrated, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel to afford the desired product.



**General procedure VI**<sup>[6]</sup>: A round bottom flask equipped with a stir bar was charged with methyltriphenylphosphonium bromide (1.20 equiv.) and placed under a nitrogen atmosphere. THF

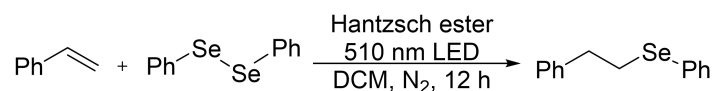
(0.3M) was added, and the reaction was cooled to  $-78\text{ }^{\circ}\text{C}$  prior to addition of nBuLi (1.20 equiv, 2.5M in hexanes) dropwise over 5 minutes. The cooling bath was then removed, and the reaction was stirred for 1 hour. The reaction was then again cooled to  $-78\text{ }^{\circ}\text{C}$ , and carbonyl substrate (1.0 equiv., 0.8M in THF) was added dropwise at this temperature. After addition, the cooling bath was removed, and reaction stirred 16 hours at room temperature.  $\text{NH}_4\text{Cl}$  (sat. aq., 20 mL) was then added and the mixture transferred to a separatory funnel. Ether (20 mL) was added, and the layers separated. The aqueous layer extracted 2x with ether (25 mL). The combined organic extracts were washed with brine (50 mL), dried ( $\text{Na}_2\text{SO}_4$ ), filtered, and concentrated in vacuo. The resulting residue was purified via flash column chromatography ( $\text{SiO}_2$ ) to yield the desired alkene product.

### 3. General procedure for hydroselenation of olefins with diselenides



Diselenide (0.2 mmol), olefin (0.5 mmol) and Hantzsch ester (0.7 mmol) were placed in a 10.0 mL quartz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (4.0 mL) was added. The quartz test tube was sealed and placed approximately 3 cm from a 10W blue LED ( $\lambda=510\text{ nm}$ ). This mixture was then stirred and irradiated at room temperature. The reaction mixture was evaporated and purified directly by column chromatography to afford the product.

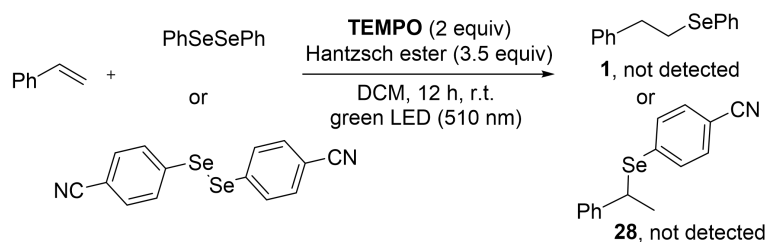
### 4. Gram-Scale synthesis



Styrene (12.5 mmol), diselenide (5 mmol), Hantzsch ester (17.5 mmol) were placed in a 30 mL quartz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (15 mL) was added. The quartz test tube was sealed and placed approximately 3 cm from a 10W green LED ( $\lambda = 510\text{ nm}$ ). This mixture was then stirred and irradiated for 12 h at room temperature. The reaction mixture was evaporated and purified directly by column chromatography to afford the product (94%, 2.46 g).

## 5. Mechanistic studies

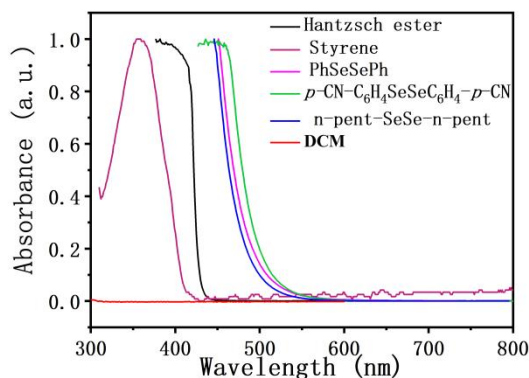
### 5.1 Radical inhibition experiments



Styrene (0.5 mmol), diselenide (0.2 mmol), Hantzsch ester (0.7 mmol), TEMPO (0.4 mmol) were placed in a 10 mL quartz test tube equipped with a magnetic stir bar. After back-filled with nitrogen (this process was repeated three times), DCM (2 mL) was added. The quartz test tube was sealed and placed approximately 3 cm from a 10 W green LED ( $\lambda=510$  nm). This mixture was then stirred and irradiated for 12 h at room temperature. No target products generation were detected by GC-MS.

### 5.2 Ultraviolet absorption and fluorescence quenching experiments

UV/Vis absorption spectra were recorded on a SHIMADZU UV-2600 spectrophotometer.  $1 \times 10^{-1}$  mol/L<sup>-1</sup> Hantzsch ester, Styrene, PhSeSePh, *p*-CN-C<sub>6</sub>H<sub>4</sub>SeSeC<sub>6</sub>H<sub>4</sub>-*p*-CN and "PeSeSe"Pe solutions using DCM as the solvent were prepared. The samples were measured in Surui® fluorescence quartz cuvettes (chamber volume = 3.500 mL, H × W × D = 48 mm × 12.5 mm × 12.5 mm, path length = 10 mm).



**Fig. S1** UV-Vis absorption spectrum

The fluorescence spectrum is recorded on the burker F-7000. Solution with Hantzsch ester and different concentrations Styrene were prepared. The samples were measured in Surui® fluorescence quartz cuvettes (chamber volume = 3.500 mL, H × W × D = 48 mm × 12.5 mm × 12.5 mm, path length = 10 mm). The following parameters were set: data interval = 0.5 nm, scan-speed = 500 nm/min, excitation wavelength  $\lambda_{ex}$  = 330 nm.

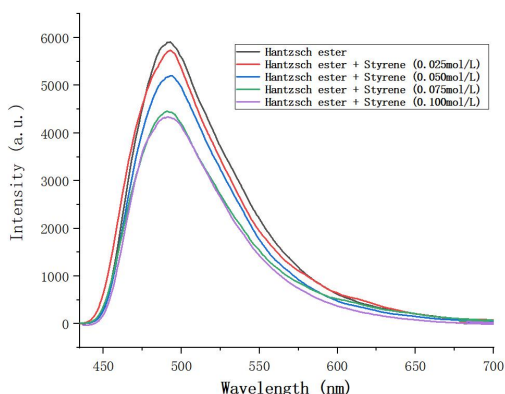


Fig. S2 Fluorescence spectrum

### 5.3 Light on/off experiment

To study the necessity of continuous irradiation with visible light for the progress of the reaction, the reaction proceeding was monitored by GC-MS before and after light irradiation and dark periods. The control experiments shown below with successive intervals of irradiation and dark periods did result in interruption of the reaction progress in the absence of light, demonstrating that light is a necessary component for the reaction after triggering the reaction in the first part.

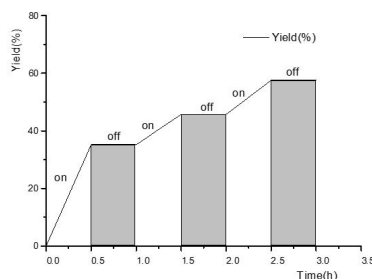
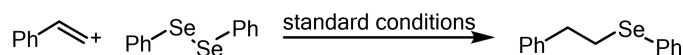
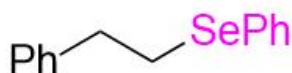
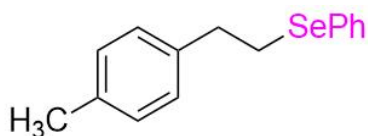


Fig. S3 Light on-off experiment

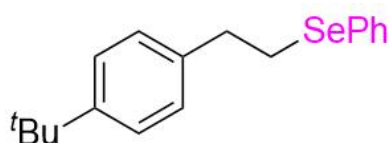
## 6. Characterization of products in details



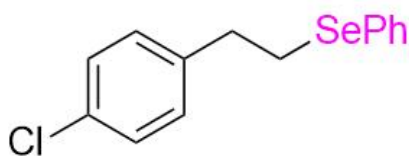
**phenethyl(phenyl)selane (1)**<sup>[7]</sup>: yellow liquid (51.9 mg, 99% yield); EtOAc/PE = 1/50, v/v. <sup>1</sup>H NMR (500 MHz, Chloroform-*d*) δ 7.46 (d, *J* = 10.0 Hz, 2H), 7.24–7.14 (m, 6H), 7.12–7.10 (m, 2H), 3.08 (t, *J* = 10.0 Hz, 2H), 2.94 (t, *J* = 10.0 Hz, 2H); <sup>13</sup>C NMR (125 MHz, Chloroform-*d*) δ 141.2, 132.8, 130.6, 129.3, 128.7, 128.6, 127.0, 126.6, 36.8, 28.9. HR-MS (ESI) *m/z*: Calculated for C<sub>15</sub>H<sub>17</sub>Se<sup>+</sup> [M + H]<sup>+</sup>: 263.0333. Found: 263.0331.



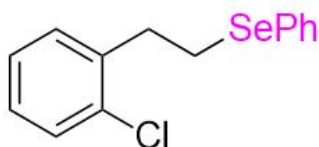
**(4-methylphenethyl)(phenyl)selane (2)**: yellow liquid (54.7 mg, 98% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.49 (d,  $J = 8.0$  Hz, 2H), 7.26–7.19 (m, 3H), 7.09–7.04 (m, 4H), 3.11 (t,  $J = 8.0$  Hz, 2H), 2.94 (t,  $J = 8.0$  Hz, 2H), 2.30 (s, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  138.0, 136.0, 132.6, 130.3, 129.2, 129.1, 128.3, 126.8, 36.2, 28.9, 21.1. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{15}\text{H}_{17}\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 277.0490. Found: 277.0493.



**(4-(tert-butyl)phenethyl)(phenyl)selane (3)**: yellow liquid (63.6 mg, 99% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.46 (d,  $J = 8.0$  Hz, 2H), 7.28 (d,  $J = 8.0$  Hz, 2H), 7.22–7.17 (m, 3H), 7.09–7.07 (m, 2H), 3.10 (t,  $J = 8.0$  Hz, 2H), 2.94 (t,  $J = 8.0$  Hz, 2H), 1.28 (s, 9H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  149.3, 138.2, 132.7, 130.6, 129.3, 128.3, 126.9, 125.6, 36.4, 34.6, 31.6, 28.9. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{18}\text{H}_{23}\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 319.0959. Found: 319.0966.

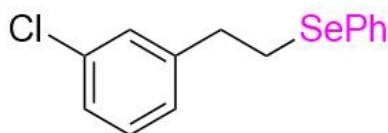


**(4-chlorophenethyl)(phenyl)selane (4)**<sup>[8]</sup>: yellow liquid (59.2 mg, 99% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.50–7.48 (m, 2H), 7.29–7.23 (m, 5H), 7.09 (d,  $J = 5.0$  Hz, 2H), 3.11 (t,  $J = 5.0$  Hz, 2H), 2.96 (t,  $J = 5.0$  Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  139.3, 132.7, 132.1, 129.9, 129.8, 129.1, 128.6, 127.0, 35.8, 28.5. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{14}\text{H}_{14}\text{ClSe}^+$   $[\text{M} + \text{H}]^+$ : 296.9944. Found: 296.9939.

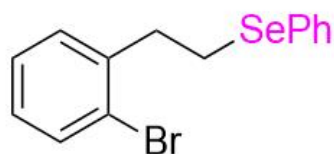


**(2-chlorophenethyl)(phenyl)selane (5)**: yellow liquid (46.2 mg, 78% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.44–7.43 (m, 2H), 7.24–7.22 (m, 1H), 7.20–7.13 (m, 3H), 7.09–7.04 (m, 3H), 3.05 (t,  $J = 5.0$  Hz, 2H), 3.01 (t,  $J = 5.0$  Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  138.5, 133.9, 132.7, 130.7, 130.0, 129.6, 129.1, 128.0, 127.0, 126.9, 34.7, 26.6. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{14}\text{H}_{14}\text{ClSe}^+$   $[\text{M} + \text{H}]^+$ : 296.9944. Found: 296.9949.

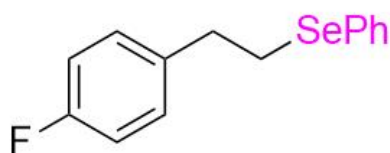




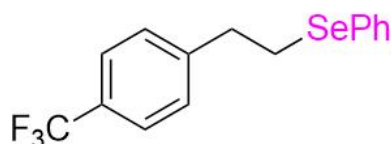
**(3-chlorophenethyl)(phenyl)selane (6):** yellow liquid (37.9 mg, 64% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.42–7.40 (m, 2H), 7.19–7.14 (m, 3H), 7.12–7.06 (m, 3H), 6.95 (d,  $J = 4.0$  Hz, 1H), 3.02 (t,  $J = 8.0$  Hz, 2H), 2.86 (t,  $J = 8.0$  Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  143.0, 134.3, 132.9, 130.0, 129.8, 129.2, 128.6, 127.1, 126.7, 126.6, 36.34, 28.3. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{14}\text{H}_{14}\text{ClSe}^+$   $[\text{M} + \text{H}]^+$ : 296.9944. Found: 296.9939.



**(2-bromophenethyl)(phenyl)selane (7):** yellow liquid (50.3 mg, 74% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.54–7.49 (m, 3H), 7.28–7.18 (m, 5H), 7.07–7.04 (m, 1H), 3.14 (t,  $J = 8.0$  Hz, 2H), 3.10 (t,  $J = 8.0$  Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  140.3, 133.0, 132.8, 130.7, 130.0, 129.1, 128.2, 127.6, 127.0, 124.2, 37.2, 26.8.  $^{19}\text{F NMR}$  (471 MHz, Chloroform-*d*)  $\delta$  (ppm) -116.7. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{14}\text{H}_{14}\text{BrSe}^+$   $[\text{M} + \text{H}]^+$ : 340.9439. Found: 340.9435.

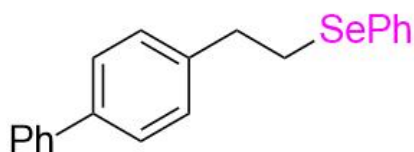


**(4-fluorophenethyl)(phenyl)selane (8):** yellow liquid (55.4 mg, 98% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.49 (d,  $J = 8.0$  Hz, 2H), 7.27–7.23 (m, 3H), 7.13–7.10 (m, 2H), 6.98–6.94 (m, 2H), 3.11 (t,  $J = 8.0$  Hz, 2H), 2.96 (t,  $J = 8.0$  Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  161.6 (d,  $J = 243.8$  Hz), 136.6, 132.7, 130.0, 129.8 (d,  $J = 7.5$  Hz), 129.1, 127.0, 115.3 (d,  $J = 21.3$  Hz), 35.8, 25.9.  $^{19}\text{F NMR}$  (471 MHz, Chloroform-*d*)  $\delta$  -116.7. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{14}\text{H}_{14}\text{FSe}^+$   $[\text{M} + \text{H}]^+$ : 281.0239. Found: 281.0238.

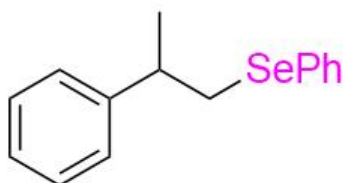


**phenyl(4-(trifluoromethyl)phenethyl)selane (9):** yellow liquid (57.9 mg, 88% yield); EtOAc/PE = 1/10, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.53 (d,  $J = 8.0$  Hz, 2H), 7.50–7.48 (m, 2H), 7.28–7.25 (m, 5H), 3.14 (t,  $J = 8.0$  Hz, 2H), 3.04 (t,  $J = 8.0$  Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  144.9, 132.9, 129.8, 129.2, 128.82 (q,  $J = 32.5$  Hz), 128.8, 127.2, 125.4 (q,  $J = 3.75$  Hz), 123.2 (q,  $J = 271.25$  Hz), 36.4, 28.2.  $^{19}\text{F NMR}$  (471 MHz, Chloroform-*d*)  $\delta$  144.9.

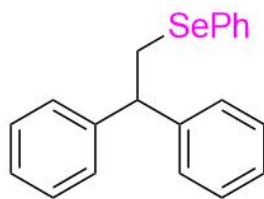
HR-MS (ESI)  $m/z$ : Calculated for  $C_{15}H_{14}F_3Se^+$   $[M + H]^+$ : 331.0207. Found: 331.0200.



**(2-([1,1'-biphenyl]-4-yl)ethyl)(phenyl)selane (10)**: yellow liquid (66.9 mg, 99% yield); EtOAc/PE = 1/20, v/v.  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.47 (d,  $J = 8.0$  Hz, 2H), 7.41 (d,  $J = 8.0$  Hz, 4H), 7.33–7.30 (m, 2H), 7.24–7.21 (m, 1H), 7.18–7.13 (m, 5H), 3.07 (t,  $J = 8.0$  Hz, 2H), 2.93 (t,  $J = 8.0$  Hz, 2H);  $^{13}C$  NMR (125 MHz, Chloroform-*d*)  $\delta$  141.0, 140.1, 139.4, 132.7, 130.3, 129.2, 128.9, 128.8, 127.3, 127.2, 127.1, 127.0, 36.3, 28.8. HR-MS (ESI)  $m/z$ : Calculated for  $C_{20}H_{19}Se^+$   $[M + H]^+$ : 339.0646 Found: 339.0641.



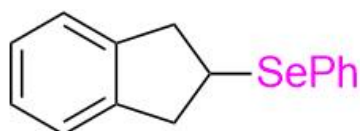
**phenyl(2-phenylpropyl)selane (11)** <sup>[9]</sup>: yellow liquid (54.7 mg, 99% yield); EtOAc/PE = 1/20, v/v.  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$ , 7.45 (d,  $J = 8.0$  Hz, 2H), 7.31–7.28 (m, 2H), 7.25–7.18 (m, 6H), 3.23–3.19 (m, 1H), 3.10–2.98 (m, 2H), 1.39 (d,  $J = 8.0$  Hz, 3H);  $^{13}C$  NMR (125 MHz, Chloroform-*d*)  $\delta$  145.9, 132.5, 130.8, 129.0, 128.5, 126.9, 126.7, 126.6, 40.3, 36.7, 21.8. HR-MS (ESI)  $m/z$ : Calculated for  $C_{15}H_{17}Se^+$   $[M + H]^+$ : 277.0490. Found: 277.0495.



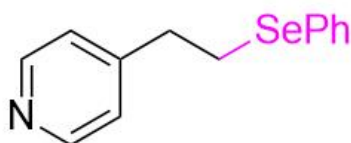
**(2,2-diphenylethyl)(phenyl)selane (12)** <sup>[7]</sup>: yellow solid (67.4 mg, 98% yield); EtOAc/PE = 1/10, v/v. m.p. 123.6–136.5 °C.  $^1H$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.46–7.43 (m, 2H), 7.30–7.27 (m, 4H), 7.24–7.19 (m, 9H), 4.26 (t,  $J = 8.0$  Hz, 1H), 3.58 (d,  $J = 8.0$  Hz, 2H);  $^{13}C$  NMR (125 MHz, Chloroform-*d*)  $\delta$  143.6, 132.8, 130.7, 129.1, 128.6, 127.8, 126.9, 126.7, 51.6, 33.8. HR-MS (ESI)  $m/z$ : Calculated for  $C_{20}H_{19}Se^+$   $[M + H]^+$ : 339.0646. Found: 339.0641.



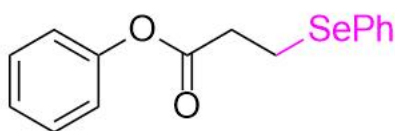
**Isopropyl 2-(4-(1-(4-chlorophenyl)-2-(phenylselanyl)ethyl)phenoxy)-2-methylpropanoate (13)**: orange liquid (73.3 mg, 71% yield); EtOAc/PE = 1/5, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.44–7.42 (m, 2H), 7.25–7.22 (m, 5H), 7.12 (d,  $J = 5.0$  Hz, 2H), 7.03 (d,  $J = 10.0$  Hz, 2H), 6.76 (d,  $J = 10.0$  Hz, 2H), 5.08–5.03 (m, 1H), 4.15 (t,  $J = 10.0$  Hz, 1H), 3.52–3.44 (m, 2H), 1.56 (s, 6H), 1.20 (d,  $J = 5.0$  Hz, 6H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  173.7, 154.4, 142.1, 136.6, 132.9, 132.4, 130.5, 129.2, 129.1, 128.6, 128.3, 127.0, 119.0, 79.1, 68.9, 50.2, 33.8, 25.4, 21.6. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{27}\text{H}_{30}\text{ClO}_3\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 517.1043 Found: 517.1033.



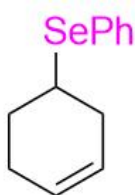
**(2,3-dihydro-1H-inden-2-yl)(phenyl)selane (14)** <sup>[8]</sup>: yellow liquid (32.9 mg, 60% yield); EtOAc/PE = 1/10, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.57–7.52 (m, 2H), 7.28–7.25 (m, 3H), 7.20–7.14 (m, 4H), 4.15–4.10 (m, 1H), 3.41–3.36 (m, 2H), 3.09–3.04 (m, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  142.0, 134.0, 130.1, 129.1, 127.3, 126.6, 124.4, 40.8, 40.3. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{15}\text{H}_{15}\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 275.0333 Found: 275.0336.



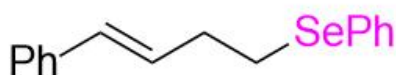
**4-(2-(phenylselanyl)ethyl)pyridine (15)**: yellow liquid (52.4 mg, 99% yield); EtOAc/PE = 1/2, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  8.54 (d,  $J = 8.0$  Hz, 2H), 7.55–7.52 (m, 2H), 7.31–7.30 (m, 3H), 7.13–7.12 (m, 2H), 3.16 (t,  $J = 8.0$  Hz, 2H), 3.01 (t,  $J = 8.0$  Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  149.9, 149.6, 133.0, 129.6, 129.2, 127.3, 123.8, 35.8, 27.2. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{13}\text{H}_{14}\text{NSe}^+$   $[\text{M} + \text{H}]^+$ : 264.0286 Found: 264.0281.



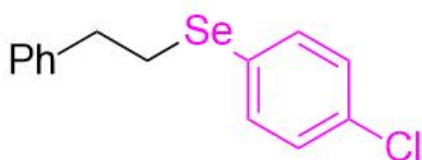
**phenyl 3-(phenylselanyl)propanoate (16):** orange liquid (37.9 mg, 62% yield); EtOAc/PE = 1/10, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.64–7.61 (m, 2H), 7.44–7.40 (m, 2H), 7.35–7.33 (m, 3H), 7.29–7.25 (m, 1H), 7.14–7.12 (m, 2H), 3.26 (t,  $J$  = 8.0 Hz, 2H), 3.02 (t,  $J$  = 8.0 Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  170.7, 150.6, 133.6, 129.5, 129.3, 129.1, 127.5, 125.9, 121.5, 35.5, 21.8. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{15}\text{H}_{15}\text{O}_2\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 307.0232 Found: 307.0229.



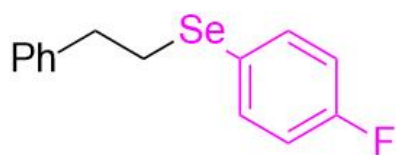
**4-((1-(*p*-tolyl)ethyl)selanyl)benzonitrile (17)**<sup>[10]</sup>: orange liquid (57.4 mg, 99% yield); EtOAc/PE = 1/50, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.57–7.55 (m, 2H), 7.27–7.24 (m, 3H), 5.70–5.59 (m, 2H), 3.52–3.46 (m, 1H), 2.47 (d,  $J$  = 15.0 Hz, 1H), 2.25–2.15 (m, 2H), 2.08 (d,  $J$  = 15.0 Hz, 2H), 1.80–1.72 (m, 1H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  134.7, 129.1, 128.9, 127.4, 126.7, 125.8, 39.1, 32.6, 29.7, 25.4. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{12}\text{H}_{15}\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 239.0333 Found: 239.0338.



**(*E*)-phenyl(4-phenylbut-3-en-1-yl)selane (18):** yellow liquid (44.9 mg, 78% yield); EtOAc/PE = 1/10, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.52–7.50 (m, 2H), 7.32–7.18 (m, 8H), 6.41 (d,  $J$  = 15.0 Hz, 1H), 6.23–6.17 (m, 1H), 3.02 (t,  $J$  = 15.0 Hz, 2H), 2.61 (q,  $J$  = 15.0 Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  137.4, 132.8, 131.3, 130.2, 129.1, 129.0, 128.5, 127.2, 126.9, 126.1, 33.8, 27.2. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{16}\text{H}_{17}\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 289.0490 Found: 289.0495.



**(4-chlorophenyl)(phenethyl)selane (19)**<sup>[11]</sup>: yellow liquid (36.1 mg, 61% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.41 (d,  $J$  = 8.0 Hz, 2H), 7.30–7.27 (m, 2H), 7.24–7.20 (m, 3H), 7.16 (d,  $J$  = 8.0 Hz, 2H), 3.12 (t,  $J$  = 8.0 Hz, 2H), 2.97 (t,  $J$  = 8.0 Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  140.7, 134.1, 133.1, 129.2, 128.6, 128.5, 128.4, 126.5, 36.5, 29.2. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{14}\text{H}_{14}\text{ClSe}^+$   $[\text{M} + \text{H}]^+$ : 296.9944. Found: 296.9942.



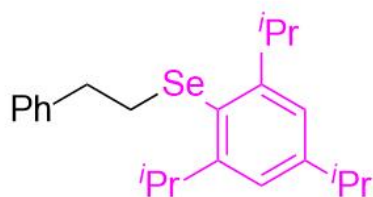
**(4-fluorophenyl)(phenethyl)selane (20)** <sup>[11]</sup>: yellow liquid (36.8 mg, 66% yield); EtOAc/PE = 1/20, v/v. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$ , 7.47–7.44 (m, 2H), 7.27–7.24 (m, 2H), 7.20–7.17 (m, 1H), 7.13 (d,  $J = 4.0$  Hz, 2H), 6.95–6.92 (m, 2H), 3.07 (t,  $J = 4.0$  Hz, 2H), 2.93 (t,  $J = 4.0$  Hz, 2H); <sup>13</sup>C NMR (125 MHz, Chloroform-*d*)  $\delta$  162.4(d,  $J = 250.0$  Hz), 140.9, 135.5(d,  $J = 7.5$  Hz), 128.6, 128.5, 126.5, 124.6, 116.3(d,  $J = 22.5$  Hz), 36.7, 29.7. <sup>19</sup>F NMR (471 MHz, Chloroform-*d*)  $\delta$  -114.6. HR-MS (ESI)  $m/z$ : Calculated for C<sub>14</sub>H<sub>14</sub>FSe<sup>+</sup> [M + H]<sup>+</sup>: 281.0239. Found: 281.0234.



**(2,6-dimethylphenyl)(phenethyl)selane (21)**: yellow liquid (57.9 mg, 99% yield); EtOAc/PE = 1/20, v/v. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.28–7.22 (m, 2H), 7.18–7.15 (m, 1H), 7.11–7.07 (m, 5H), 2.91 (t,  $J = 4.0$  Hz, 2H), 2.86 (t,  $J = 4.0$  Hz, 2H), 2.55 (s, 6H); <sup>13</sup>C NMR (125 MHz, Chloroform-*d*)  $\delta$  143.1, 141.3, 131.2, 128.5, 128.3, 127.6, 126.4, 125.8, 37.0, 28.5, 24.8. HR-MS (ESI)  $m/z$ : Calculated for C<sub>16</sub>H<sub>19</sub>Se<sup>+</sup> [M + H]<sup>+</sup>: 291.0646. Found: 291.0647.

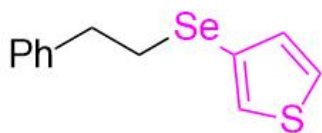


**mesityl(phenethyl)selane (22)**: yellow liquid (60.0 mg, 99% yield); EtOAc/PE = 1/50, v/v. <sup>1</sup>H NMR (400 MHz, Chloroform-*d*)  $\delta$  7.26–7.23 (m, 2H), 7.18–7.16 (m, 1H), 7.12–7.10 (m, 2H), 6.92 (s, 2H), 2.88 (t,  $J = 8.0$  Hz, 2H), 2.85 (t,  $J = 8.0$  Hz, 2H), 2.52 (s, 6H), 2.26 (s, 3H); <sup>13</sup>C NMR (125 MHz, Chloroform-*d*)  $\delta$  143.2, 141.4, 138.1, 128.6, 128.5, 128.3, 127.7, 126.3, 37.0, 28.5, 24.6, 21.0. HR-MS (ESI)  $m/z$ : Calculated for C<sub>17</sub>H<sub>21</sub>Se<sup>+</sup> [M + H]<sup>+</sup>: 305.0803 Found: 305.0810.

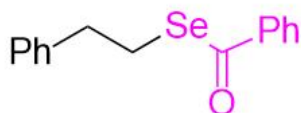


**phenethyl(2,4,6-triisopropylphenyl)selane (23)**: yellow liquid (32.6 mg, 42% yield); EtOAc/PE

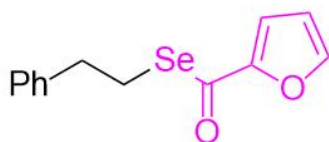
= 1/10, v/v.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.32–7.29 (m, 2H), 7.25–7.23 (m, 1H), 7.18–7.10 (m, 2H), 6.92 (s, 2H), 3.95 (t,  $J = 8.0$ , 2H), 2.96 (t, 6H), 2.93–2.87 (m, 3H), 1.27 (d,  $J = 8.0$  Hz, 18H);  $^{13}\text{C}$  NMR (125 MHz, Chloroform-*d*)  $\delta$  153.1, 149.5, 141.4, 128.5, 128.3, 127.2, 126.3, 121.7, 36.8, 34.3, 34.2, 30.6, 24.6, 24.0. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{23}\text{H}_{33}\text{Se}^+$  [ $\text{M} + \text{H}$ ] $^+$ : 389.1742 Found: 389.1750.



**Se-phenethyl benzoselenoate (24)**: orange liquid (36.5 mg, 68% yield); EtOAc/PE = 1/10, v/v.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.31–7.25 (m, 4H), 7.22–7.20 (m, 1H), 7.16 (d,  $J = 8.0$  Hz, 2H), 7.10 (d,  $J = 4.0$  Hz, 1H), 3.06 (t,  $J = 4.0$  Hz, 2H), 2.96 (t,  $J = 4.0$  Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz, Chloroform-*d*)  $\delta$  140.9, 132.2, 128.5, 128.4, 127.1, 126.4, 126.3, 122.8, 36.8, 29.6. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{12}\text{H}_{13}\text{SSe}^+$  [ $\text{M} + \text{H}$ ] $^+$ : 268.9898 Found: 268.9902.



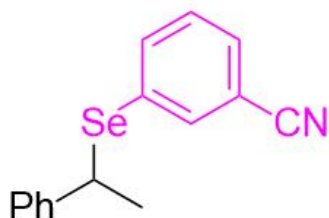
**Se-phenethyl benzoselenoate (25)**: red liquid (31.3 mg, 54% yield); EtOAc/PE = 1/10, v/v.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  8.00–7.98 (m, 2H), 7.67–7.62 (m, 1H), 7.54–7.50 (m, 2H), 7.42–7.30 (m, 5H), 3.42 (t,  $J = 8.0$  Hz, 2H), 3.14 (t,  $J = 8.0$  Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz, Chloroform-*d*)  $\delta$  194.8, 141.1, 139.1, 133.6, 128.8, 128.6, 128.5, 127.2, 126.5, 36.9, 26.9. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{15}\text{H}_{15}\text{OSe}^+$  [ $\text{M} + \text{H}$ ] $^+$ : 291.0283 Found: 291.0286.



**Se-phenethyl furan-2-carboselenoate (26)**: red liquid (25.8 mg, 46% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H}$  NMR (400 MHz, Chloroform-*d*)  $\delta$  7.58 (d,  $J = 4.0$  Hz, 1H), 7.32–7.29 (m, 2H), 7.26–7.21 (m, 3H), 7.17 (d,  $J = 4.0$  Hz, 1H), 3.30 (t,  $J = 4.0$  Hz, 2H), 3.04 (t,  $J = 4.0$  Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz, Chloroform-*d*)  $\delta$  182.1, 159.6, 152.5, 146.4, 141.0, 128.5, 126.5, 114.7, 112.6, 36.9, 25.6. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{13}\text{H}_{13}\text{O}_2\text{Se}^+$  [ $\text{M} + \text{H}$ ] $^+$ : 281.0075 Found: 281.0069.



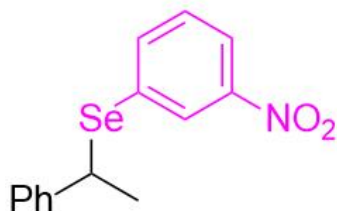
**4-((1-phenylethyl)selanyl)benzonitrile (28)**: yellow liquid (56.8 mg, 99% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.44–7.42 (m, 4H), 7.28–7.27 (m, 4H), 7.23–7.20 (m, 1H), 4.59 (q,  $J = 5.0$  Hz, 1H), 1.79 (d,  $J = 5.0$  Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  142.6, 137.7, 134.0, 132.1, 128.6, 127.5, 127.3, 118.7, 110.8, 42.8, 22.4. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{15}\text{H}_{14}\text{NSe}^+$   $[\text{M} + \text{H}]^+$ : 288.0268 Found: 288.0264.



**3-((1-phenylethyl)selanyl)benzonitrile (29)**: yellow liquid (36.2 mg, 63% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.60–7.57 (m, 2H), 7.52–7.51 (m, 1H), 7.29–7.19 (m, 6H), 4.51 (q,  $J = 5.0$  Hz, 1H), 1.76 (d,  $J = 5.0$  Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  142.7, 139.6, 138.4, 131.3, 131.2, 129.3, 128.5, 127.4, 127.3, 118.2, 112.9, 43.2, 22.0. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{15}\text{H}_{14}\text{NSe}^+$   $[\text{M} + \text{H}]^+$ : 288.0286 Found: 288.0291.



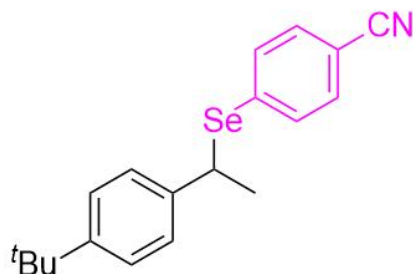
**(1-phenylethyl)(4-(trifluoromethyl)phenyl)selane (30)**<sup>[12]</sup>: yellow liquid (54.1 mg, 82% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.59–7.55 (m, 2H), 7.50 (d,  $J = 5.0$  Hz, 1H), 7.32–7.29 (m, 1H), 7.26–7.23 (m, 2H), 7.21–7.17 (m, 3H), 4.50 (q,  $J = 5.0$  Hz, 1H), 1.77 (d,  $J = 5.0$  Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  142.9, 138.6, 132.0, 130.8 (q,  $J = 32.5$  Hz), 129.0, 128.5 (q,  $J = 308.75$  Hz), 128.4, 127.3, 124.5 (q,  $J = 3.75$  Hz), 42.9, 22.0.  $^{19}\text{F NMR}$  (471 MHz, Chloroform-*d*)  $\delta$  -62.77 (s, 3F). HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{15}\text{H}_{14}\text{F}_3\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 331.0207 Found: 331.0204.



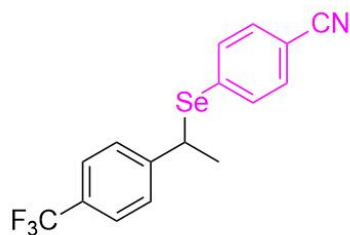
**(3-nitrophenyl)(1-phenylethyl)selane (31)**: red liquid (46.1 mg, 75% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  8.21 (s, 1H), 8.09 (d,  $J = 5.0$  Hz, 1H), 7.65 (d,  $J = 10.0$  Hz, 1H), 7.36–7.33 (m, 1H), 7.26–7.23 (m, 4H), 7.21–7.18 (m, 1H), 4.59–4.55 (m, 1H), 1.79 (d,  $J = 10.0$  Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  142.6, 140.8, 131.8, 129.5, 129.3, 128.6, 128.5, 127.5, 127.3, 122.5, 43.2, 22.1. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{14}\text{H}_{14}\text{NO}_2\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 308.0184 Found: 308.0182.



**4-((1-(p-tolyl)ethyl)selanyl)benzonitrile (32)**: orange liquid (57.4 mg, 99% yield); EtOAc/PE = 1/50, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.47–7.43 (m, 4H), 7.18 (d,  $J = 5.0$  Hz, 2H), 7.08 (d,  $J = 10.0$  Hz, 2H), 4.58 (q,  $J = 10.0$  Hz, 1H), 2.31 (s, 3H), 1.77 (d,  $J = 10.0$  Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  139.6, 138.1, 137.2, 133.8, 132.1, 129.3, 127.2, 118.8, 110.6, 42.6, 22.6, 21.1. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{16}\text{H}_{16}\text{NSe}^+$   $[\text{M} + \text{H}]^+$ : 302.0442 Found: 302.0448.



**4-((1-(4-(tert-butyl)phenyl)ethyl)selanyl)benzonitrile(33)**: yellow liquid (67.2 mg, 99% yield); EtOAc/PE = 1/10, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.47–7.43 (m, 4H), 7.30–7.29 (m, 2H), 7.21 (d,  $J = 10.0$  Hz, 2H), 4.60 (q,  $J = 5.0$  Hz, 1H), 1.79 (d,  $J = 5.0$  Hz, 3H), 1.30 (s, 9H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  150.5, 139.3, 138.2, 133.8, 132.0, 127.0, 125.5, 118.8, 110.6, 42.5, 34.6, 31.3, 22.4. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{19}\text{H}_{21}\text{NSe}^+$   $[\text{M} + \text{H}]^+$ : 343.0839 Found: 343.0831.

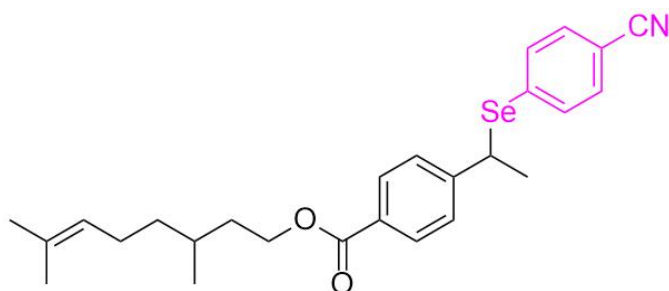


**4-((1-(4-(trifluoromethyl)phenyl)ethyl)selanyl)benzonitrile (34)**: yellow liquid (52.5 mg, 74% yield); EtOAc/PE = 1/20, v/v. m.p. 111.5–113.0 °C.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.53 (d,  $J = 5.0$  Hz, 2H), 7.48–7.45 (m, 4H), 7.37 (d,  $J = 10.0$  Hz, 2H), 4.60 (q,  $J = 5.0$  Hz, 1H), 1.80 (d, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  146.9, 136.7, 134.3, 132.2, 129.55 (q,  $J = 31.25$  Hz), 127.6, 125.55 (q,  $J = 3.75$  Hz), 123.99 (q,  $J = 271.25$  Hz), 118.5, 111.3, 42.0, 22.1.  $^{19}\text{F NMR}$  (471 MHz, Chloroform-*d*)  $\delta$  -62.50(s, 3F). HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{16}\text{H}_{13}\text{F}_3\text{NSe}^+$   $[\text{M} + \text{H}]^+$ : 356.0160 Found: 356.0154.

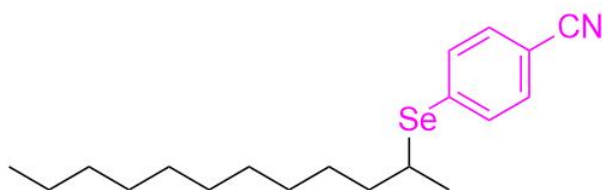




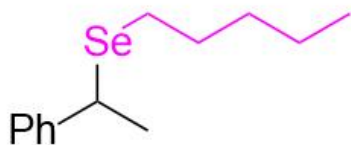
**4-((1,2-diphenylethyl)selanyl)benzonitrile (35):** yellow liquid (24.7 mg, 34% yield); EtOAc/PE = 1/10, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.41–7.36 (m, 4H), 7.26–7.11 (m, 10H), 4.60 (t,  $J$  = 10.0 Hz, 1H), 3.35 (d,  $J$  = 10.0 Hz, 2H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  140.1, 138.6, 137.6, 133.9, 132.0, 129.0, 128.7, 128.5, 128.4, 128.0, 127.5, 126.7, 126.5, 50.3, 42.9. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{21}\text{H}_{18}\text{NSe}^+$   $[\text{M} + \text{H}]^+$ : 364.0599 Found: 364.0591.



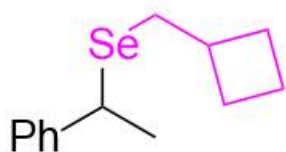
**3,7-dimethyloct-6-en-1-yl 4-((1-(4-cyanophenyl)ethyl)selanyl)ethylbenzoate (36):** yellow liquid (78.8 mg, 84% yield); EtOAc/PE = 1/5, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.93 (d,  $J$  = 10.0 Hz, 2H), 7.47–7.43 (m, 4H), 7.32 (d,  $J$  = 5.0 Hz, 2H), 5.11–5.08 (m, 1H), 4.61–4.57 (m, 1H), 4.37–4.31 (m, 2H), 2.07–1.94 (m, 2H), 1.84–1.77 (m, 4H), 1.67–1.62 (m, 4H), 1.60–1.57 (m, 4H), 1.43–1.37 (m, 1H), 1.27–1.20 (m, 1H), 0.97 (d,  $J$  = 5.0 Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  166.2, 147.9, 136.9, 134.3, 132.2, 131.4, 129.9, 129.6, 127.2, 124.6, 118.5, 111.2, 63.6, 42.3, 37.0, 35.5, 29.6, 25.7, 25.4, 22.0, 19.5, 17.7. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{26}\text{H}_{32}\text{NO}_2\text{Se}^+$   $[\text{M} + \text{H}]^+$ : 470.1593 Found: 470.1588.



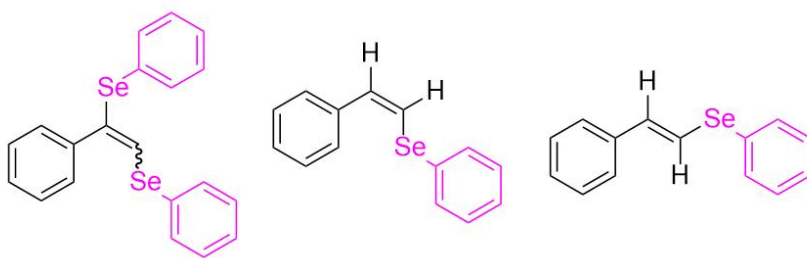
**4-(dodecan-2-ylselanyl)benzonitrile (37):** yellow liquid (45.6 mg, 65% yield); EtOAc/PE = 1/50, v/v.  $^1\text{H NMR}$  (500 MHz, Chloroform-*d*)  $\delta$  7.56–7.53 (m, 2H), 7.51–7.48 (m, 2H), 3.49–3.42 (m, 1H), 1.70–1.59 (m, 2H), 1.47 (d,  $J$  = 5.0 Hz, 3H), 1.26 (s, 16H), 0.88 (t,  $J$  = 5.0 Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  138.5, 132.8, 132.1, 118.8, 109.9, 39.3, 37.4, 31.9, 29.6, 29.5, 29.4, 29.3, 29.2, 27.8, 22.7, 22.0, 14.1. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{19}\text{H}_{30}\text{NSe}^+$   $[\text{M} + \text{H}]^+$ : 352.1538 Found: 352.1531.



**pentyl(1-phenylethyl)selane (38)**: yellow liquid (31.7 mg, 62% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.37–7.29 (m, 4H), 7.25–7.21 (m, 1H), 4.22–4.17 (m, 1H), 2.43 (t,  $J = 4.0$  Hz, 2H), 1.76 (d,  $J = 4.0$  Hz, 3H), 1.59–1.54 (m, 3H), 1.36–1.32 (m, 2H), 1.30 (d,  $J = 4.0$  Hz, 1H), 0.88 (t,  $J = 4.0$  Hz, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  144.7, 128.4, 127.2, 126.8, 37.0, 32.5, 24.2, 23.1, 22.7, 22.0, 13.6. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{13}\text{H}_{21}\text{Se}^+$  [ $\text{M} + \text{H}$ ] $^+$ : 257.0803 Found: 257.0795.



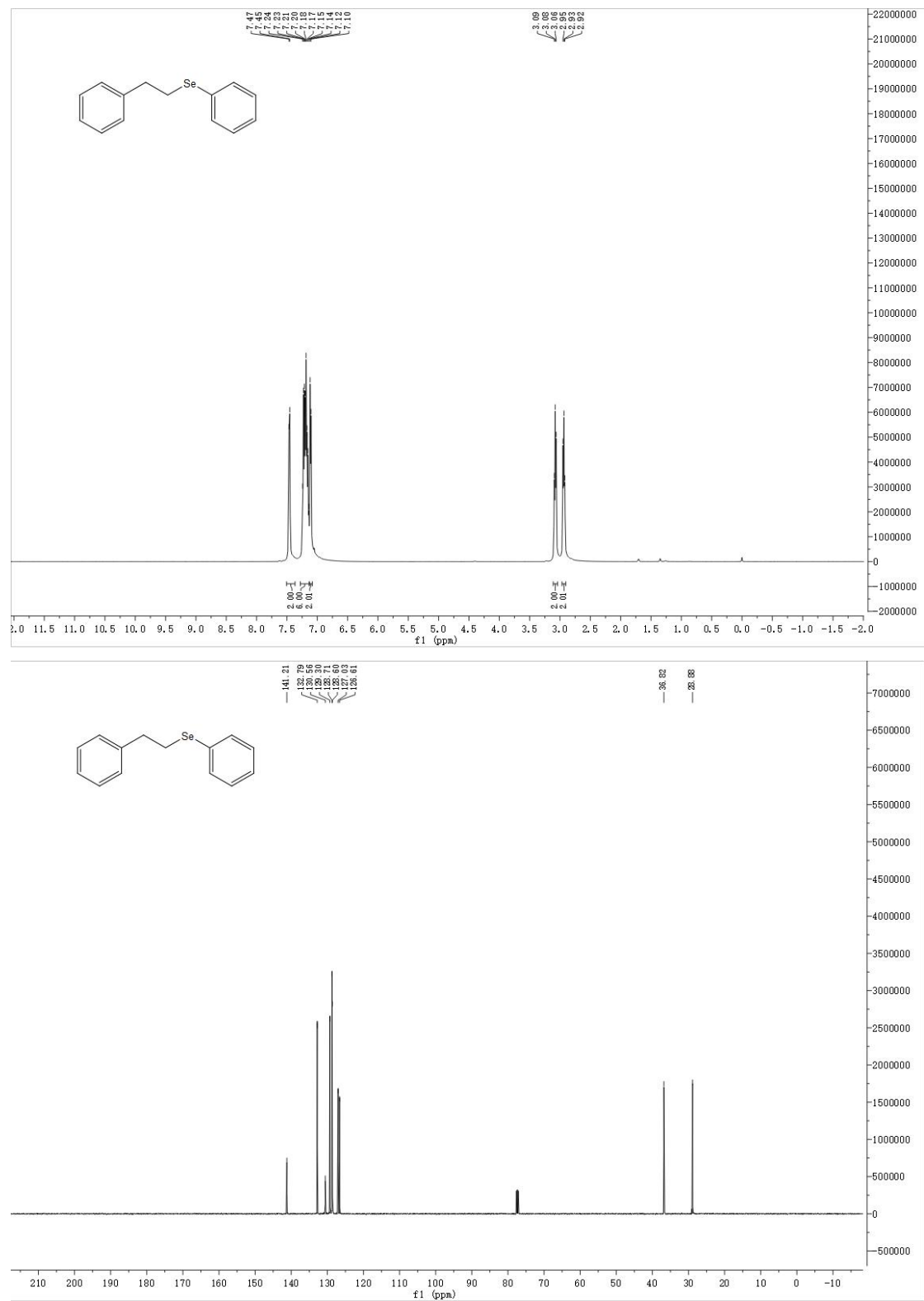
**(cyclobutylmethyl)(1-phenylethyl)selane (39)**: yellow liquid (38.1 mg, 71% yield); EtOAc/PE = 1/20, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.33–7.26 (m, 4H), 7.21–7.19 (m, 1H), 4.14 (q,  $J = 8.0$  Hz, 1H), 2.52–2.47 (m, 2H), 2.45–2.33 (m, 1H), 2.07–2.01 (m, 2H), 1.82–1.74 (m, 1H), 1.71 (d,  $J = 8.0$  Hz, 3H), 1.63–1.56 (m, 3H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  144.8, 128.4, 127.2, 126.7, 36.9, 36.0, 31.5, 28.9, 22.8, 17.6. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{14}\text{H}_{21}\text{Se}^+$  [ $\text{M} + \text{H}$ ] $^+$ : 255.0646 Found: 255.0641.



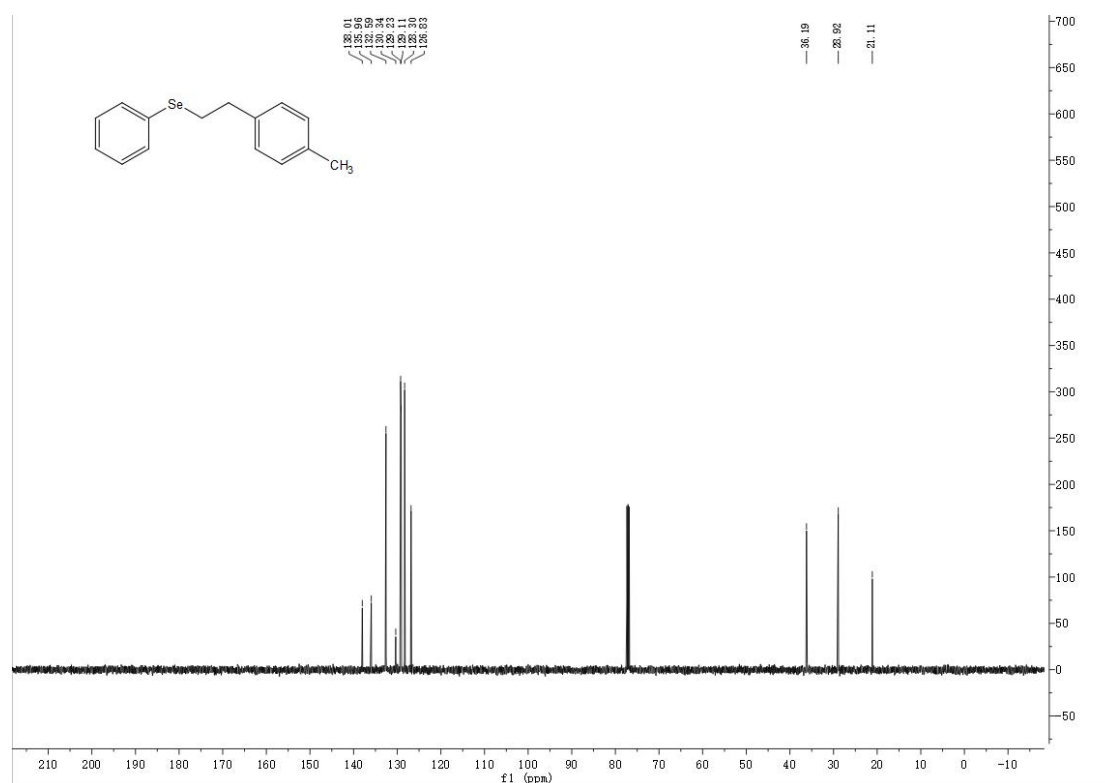
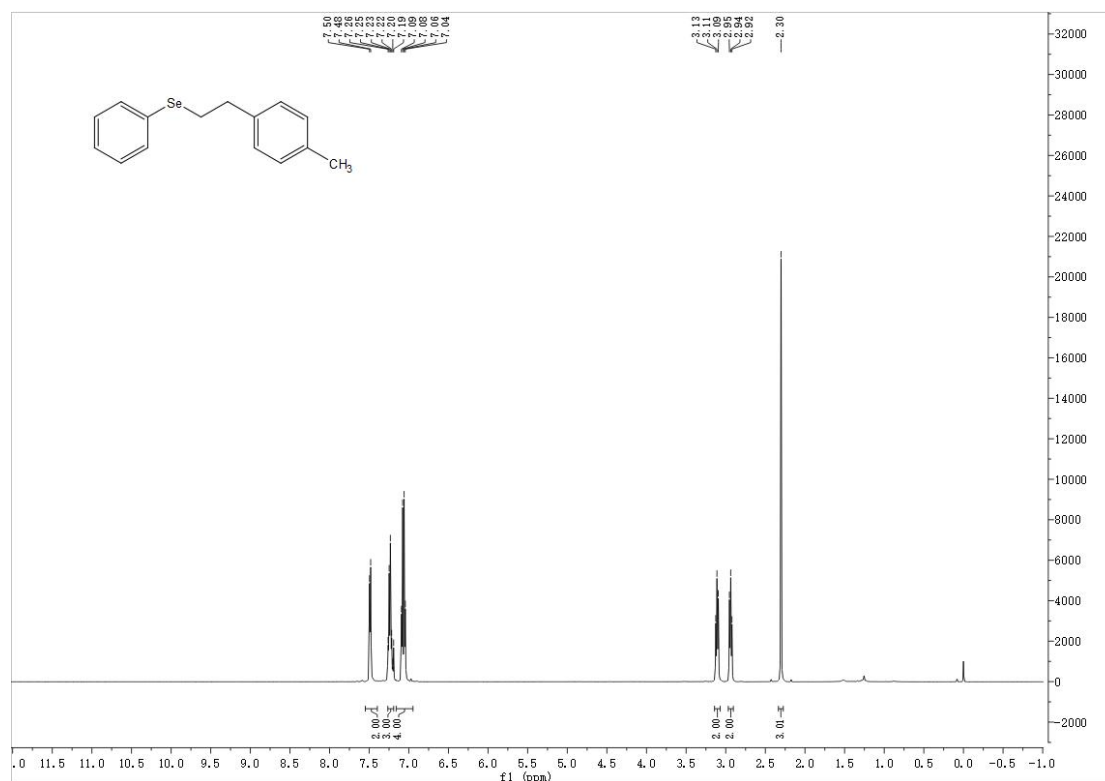
**(1-phenylethene-1,2-diyl)bis(phenylselane) (40)**, **(*Z*)-phenyl(styryl)selane (41)**, **(*E*)-phenyl(styryl)selane (42)**: yellow liquid (40.6 mg, 78% total yield, 40:41:42 = 4:5:1); EtOAc/PE = 1/10, v/v.  $^1\text{H NMR}$  (400 MHz, Chloroform-*d*)  $\delta$  7.66–7.64 (m, 2H), 7.62–7.60 (m, 0.4H), 7.49–7.43 (m, 4H), 7.38–7.29 (m, 5.8H), 7.24 (d,  $J = 16.0$  Hz, 0.2H), 7.04 (d,  $J = 8.0$  Hz, 1H), 6.94 (d,  $J = 16.0$  Hz, 0.2H), 6.85 (d,  $J = 8.0$  Hz, 1H);  $^{13}\text{C NMR}$  (125 MHz, Chloroform-*d*)  $\delta$  137.3, 135.2, 132.8, 132.6, 131.7, 130.1, 129.4, 128.7, 128.4, 128.3, 127.7, 127.5, 127.3, 126.1, 124.0, 119.5. HR-MS (ESI)  $m/z$ : Calculated for  $\text{C}_{20}\text{H}_{17}\text{Se}_2^+$  [ $\text{M} + \text{H}$ ] $^+$ : 416.9656, Found: 416.9641; Calculated for  $\text{C}_{14}\text{H}_{13}\text{Se}^+$  [ $\text{M} + \text{H}$ ] $^+$ : 261.0177, Found: 261.0173 and 261.0175.

## 7. $^1\text{H}$ , $^{13}\text{C}$ and $^{19}\text{F}$ NMR spectra of products

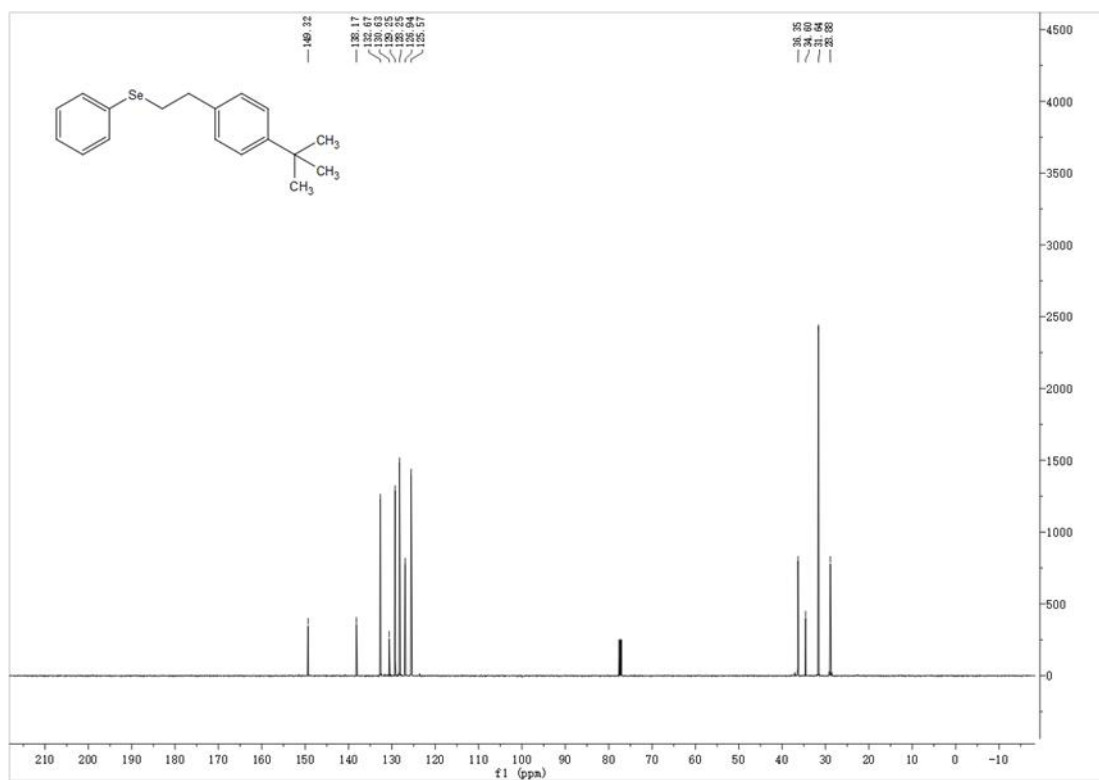
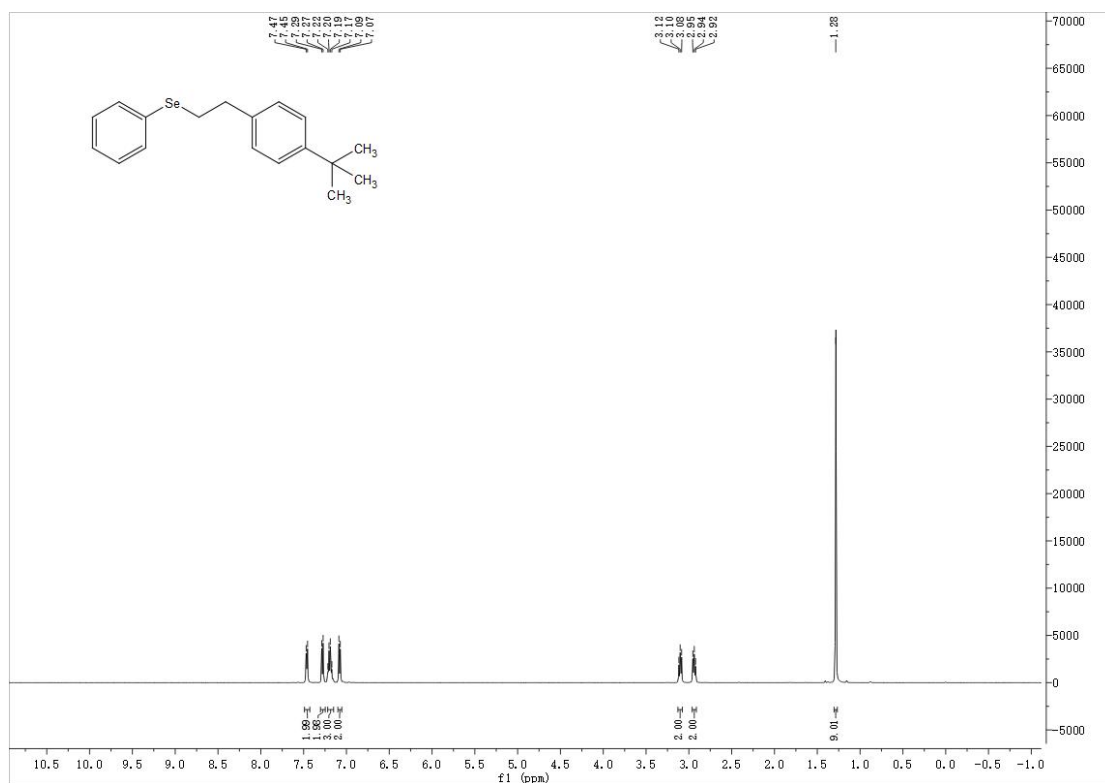
### Compound (1)



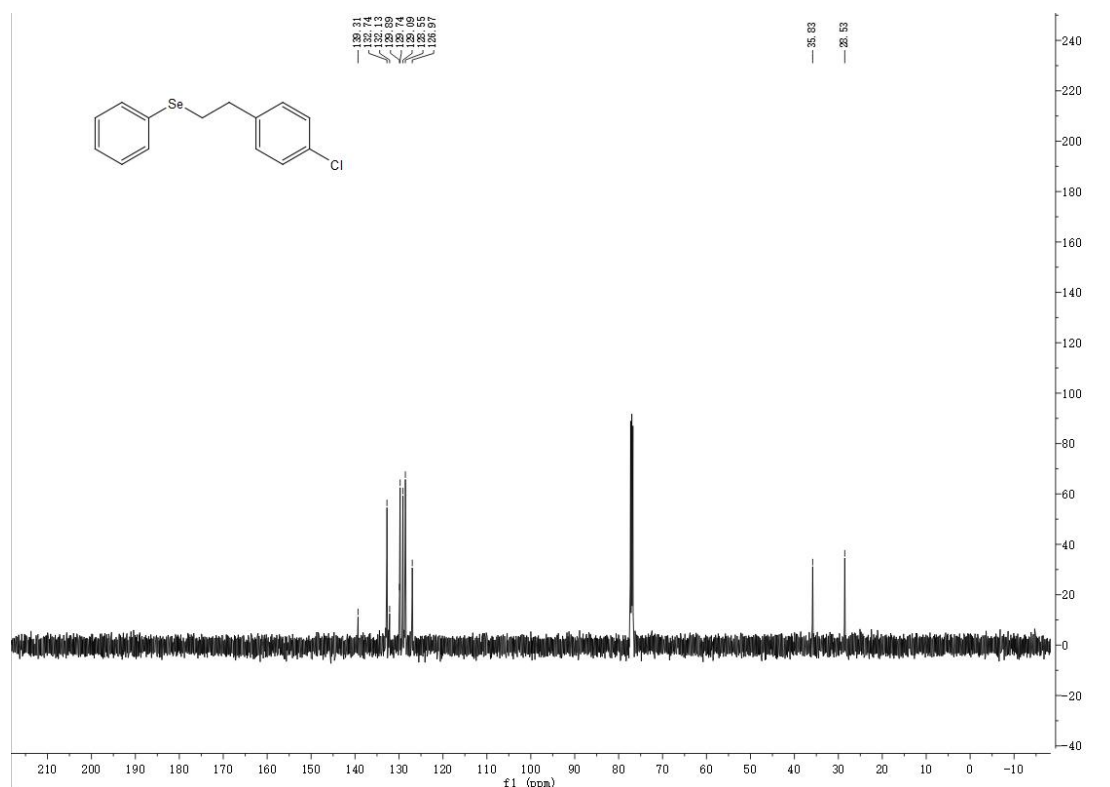
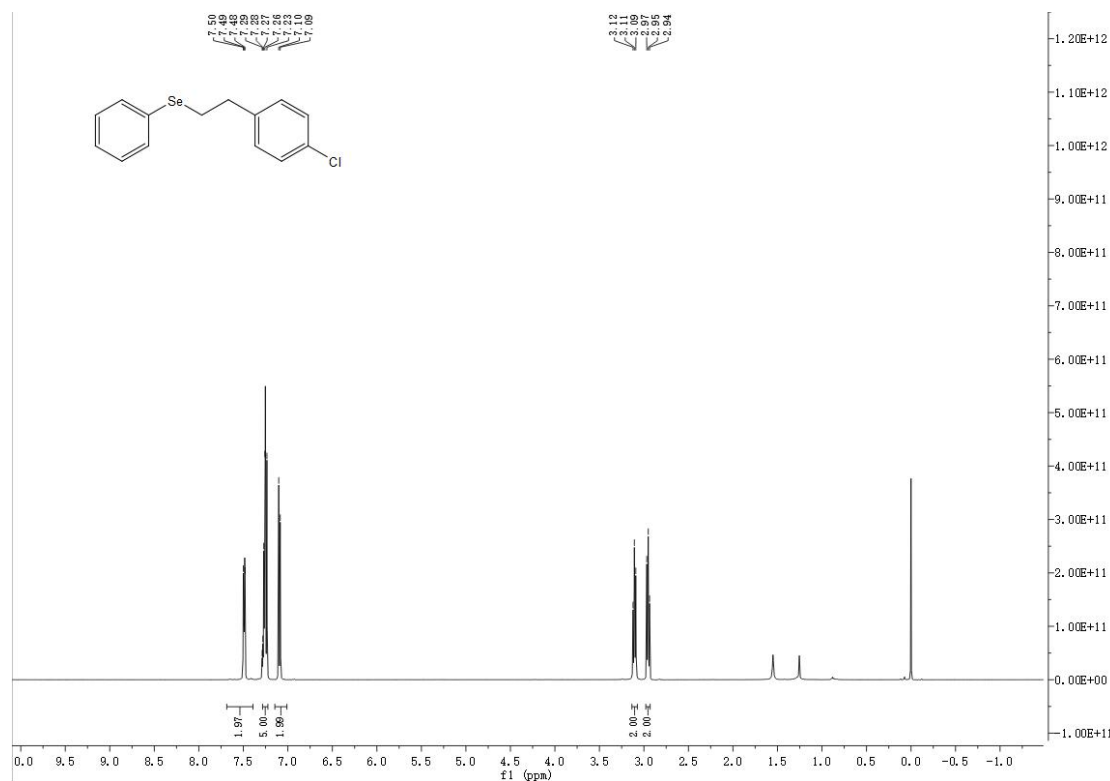
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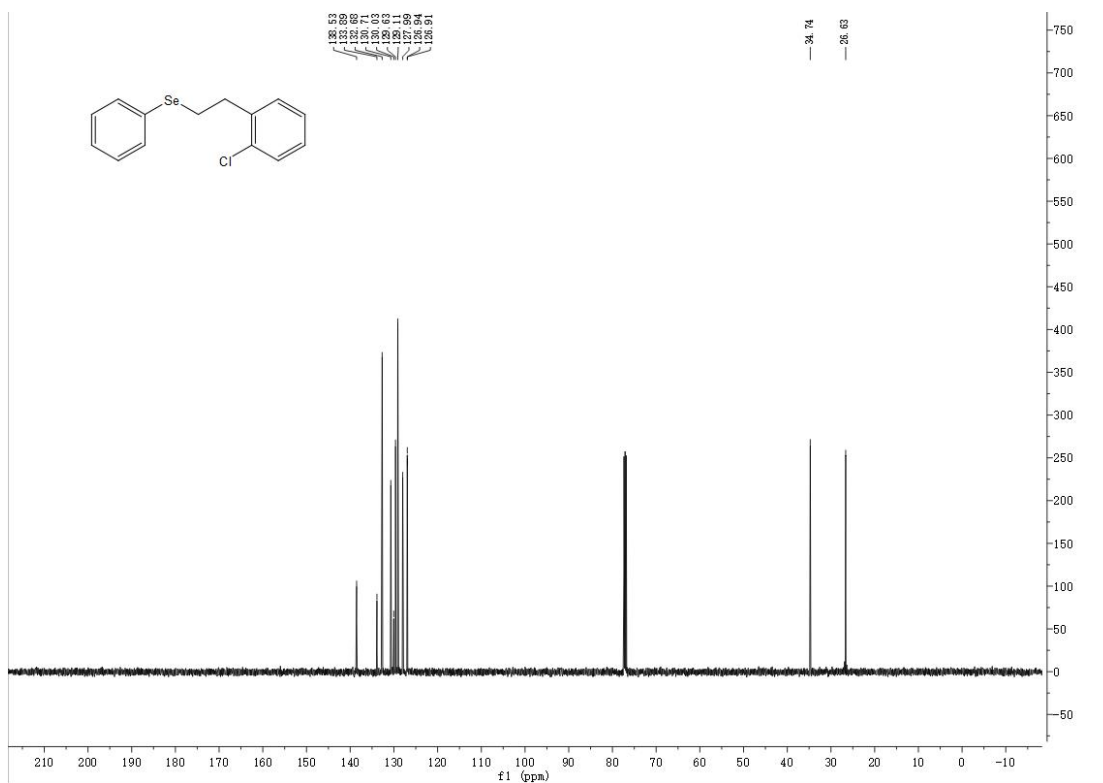
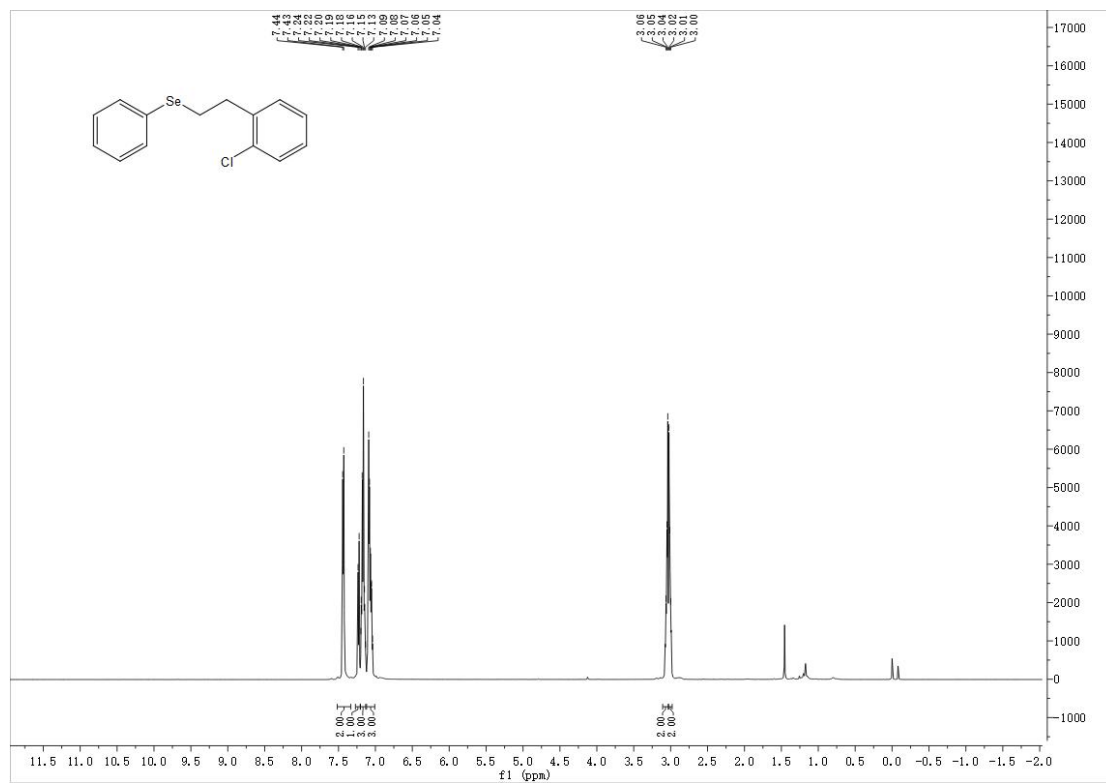
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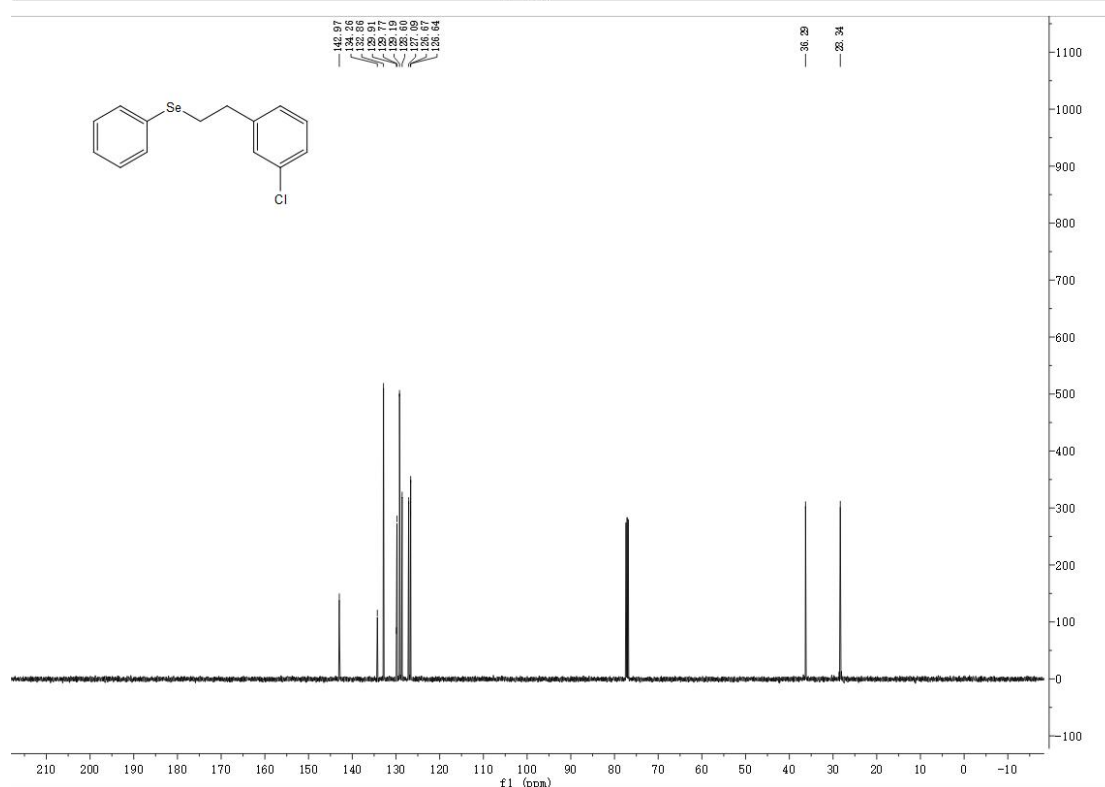
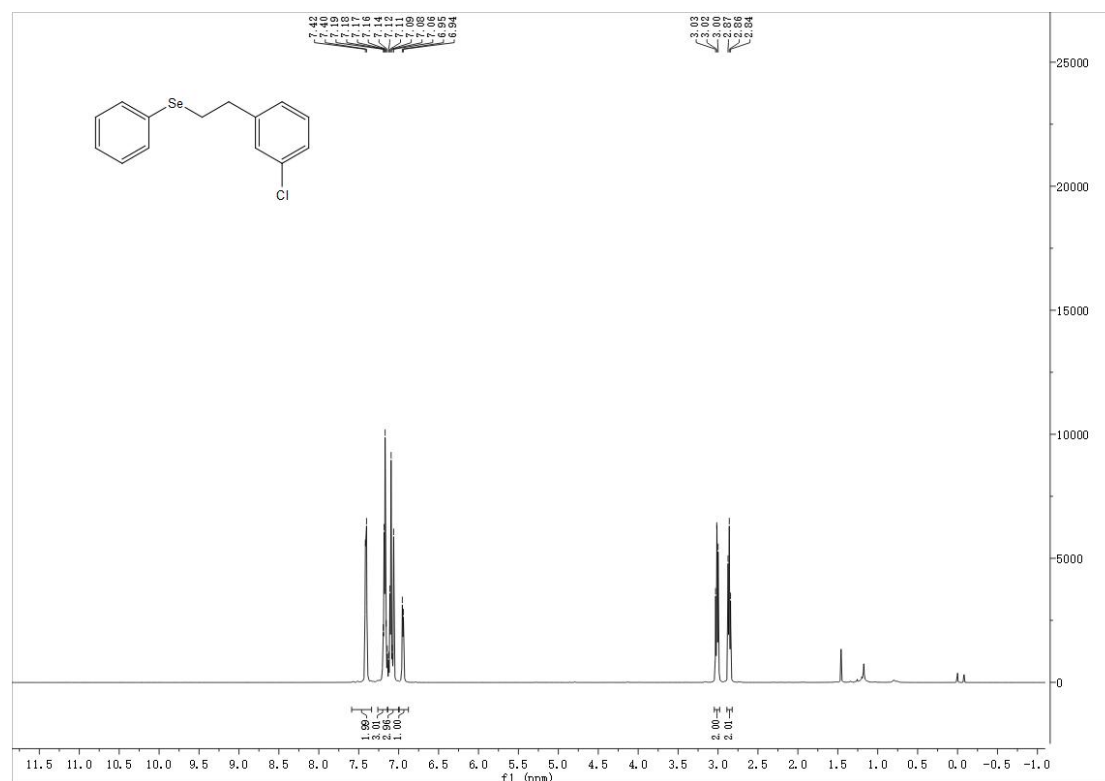
# Compound (4)



### Compound (5)

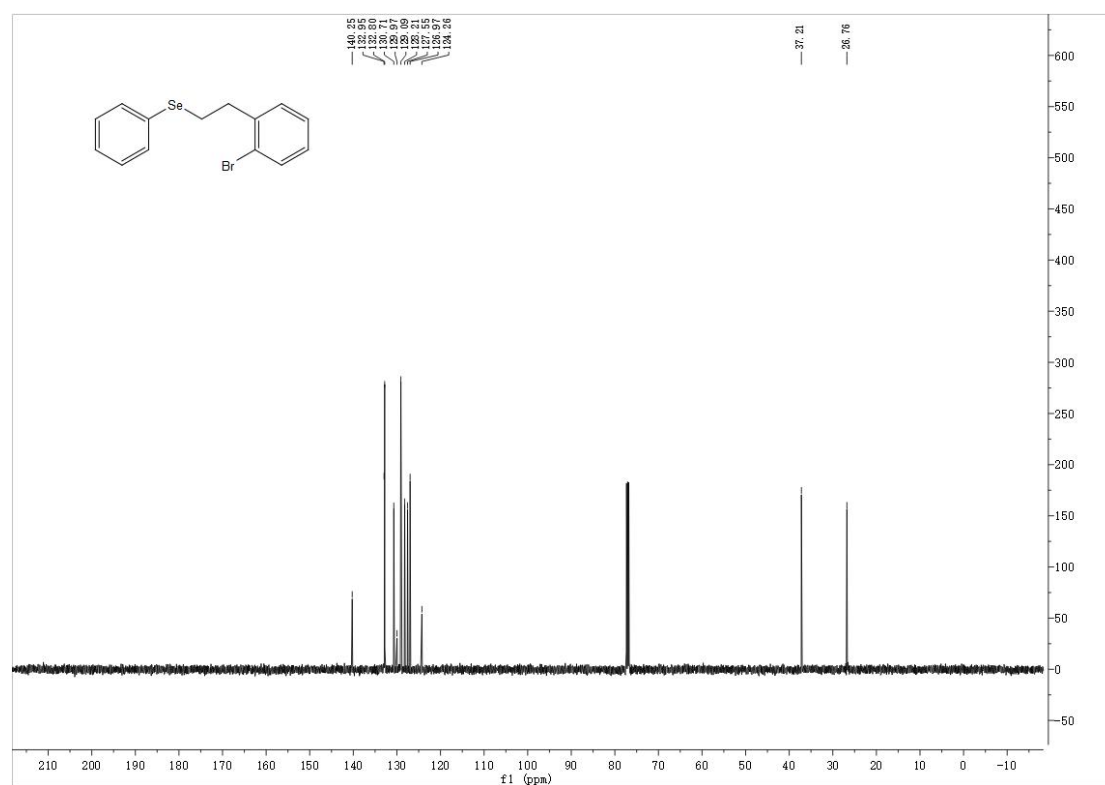
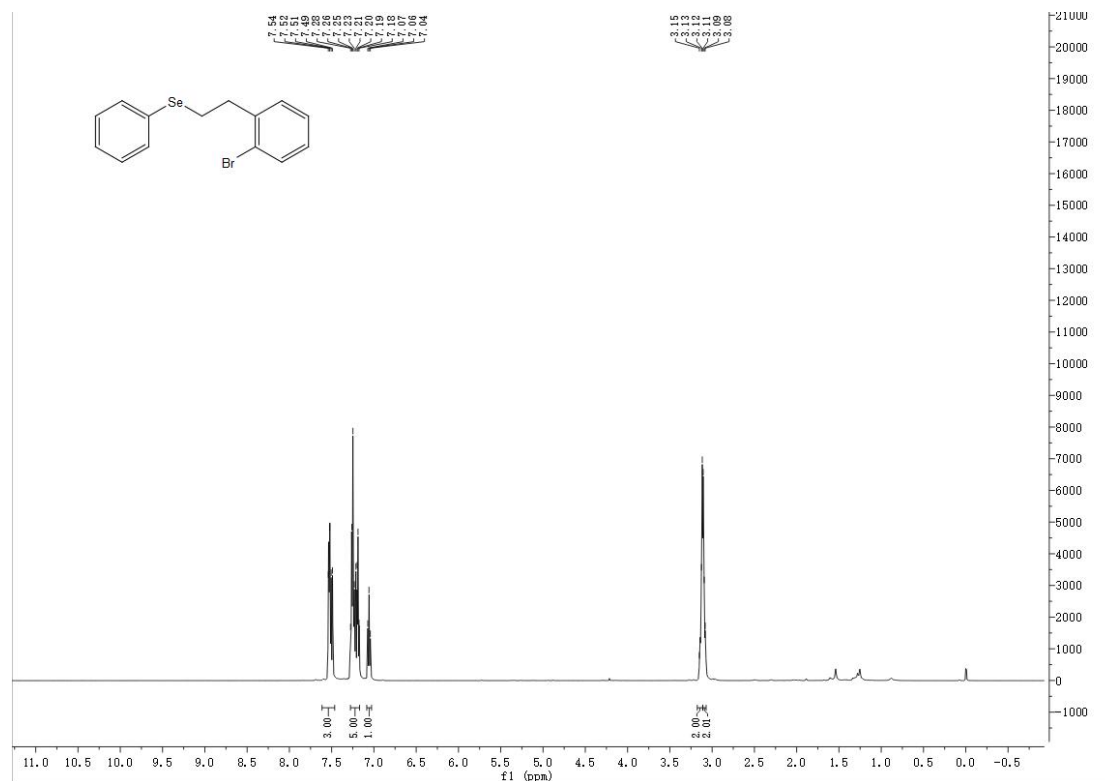


**Compound (6)**

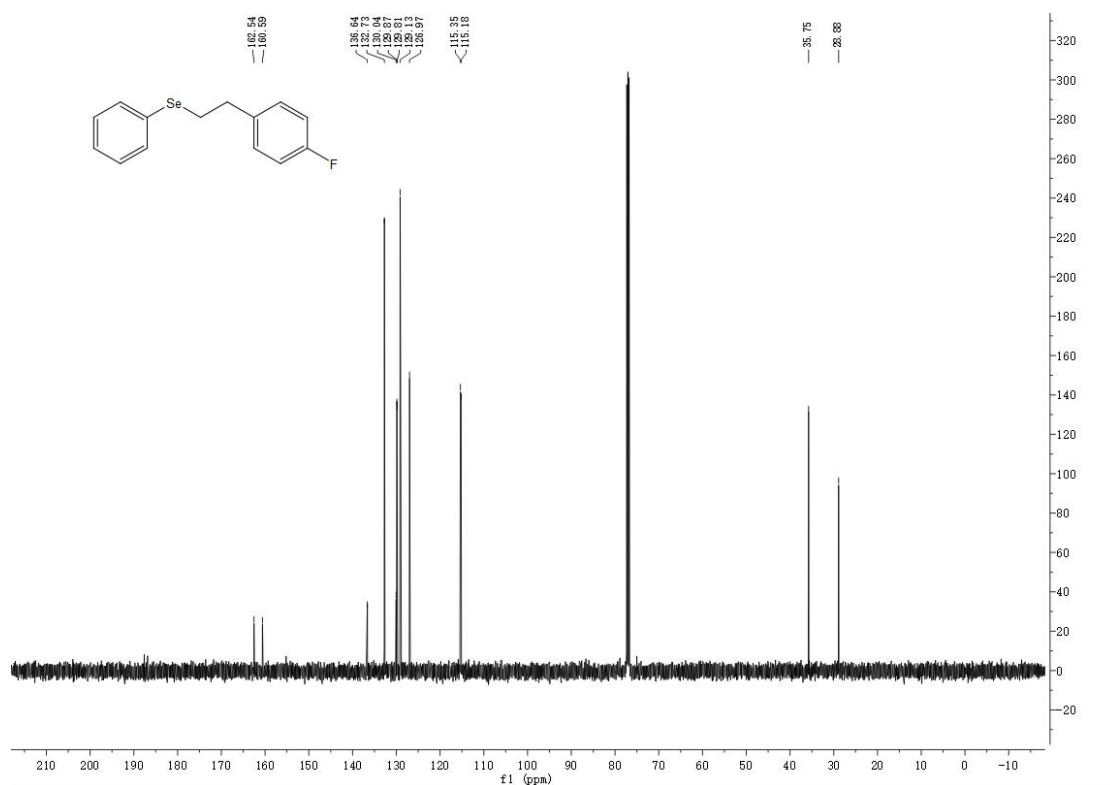
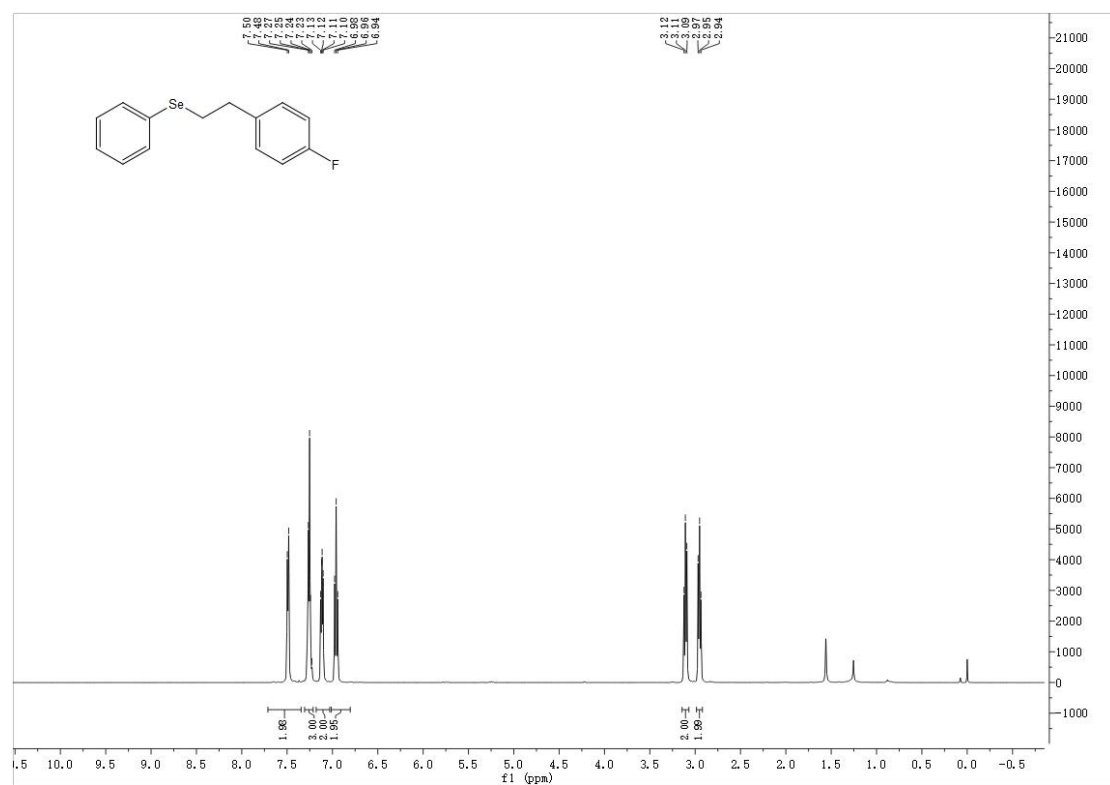


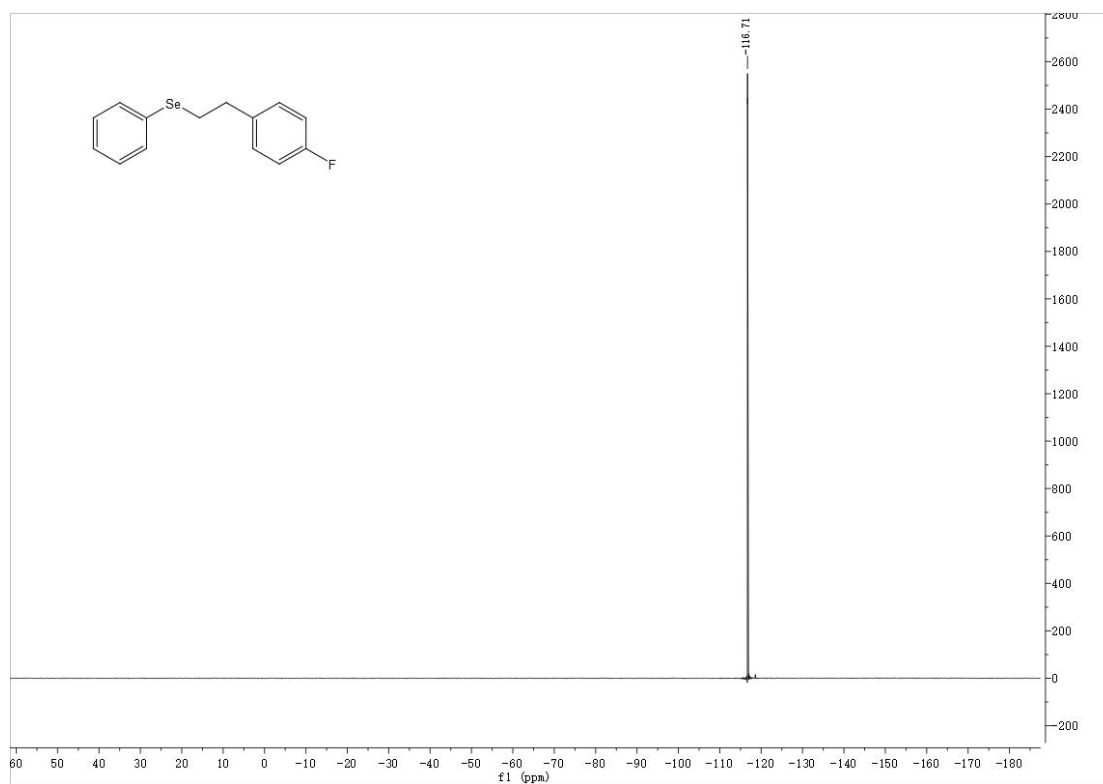


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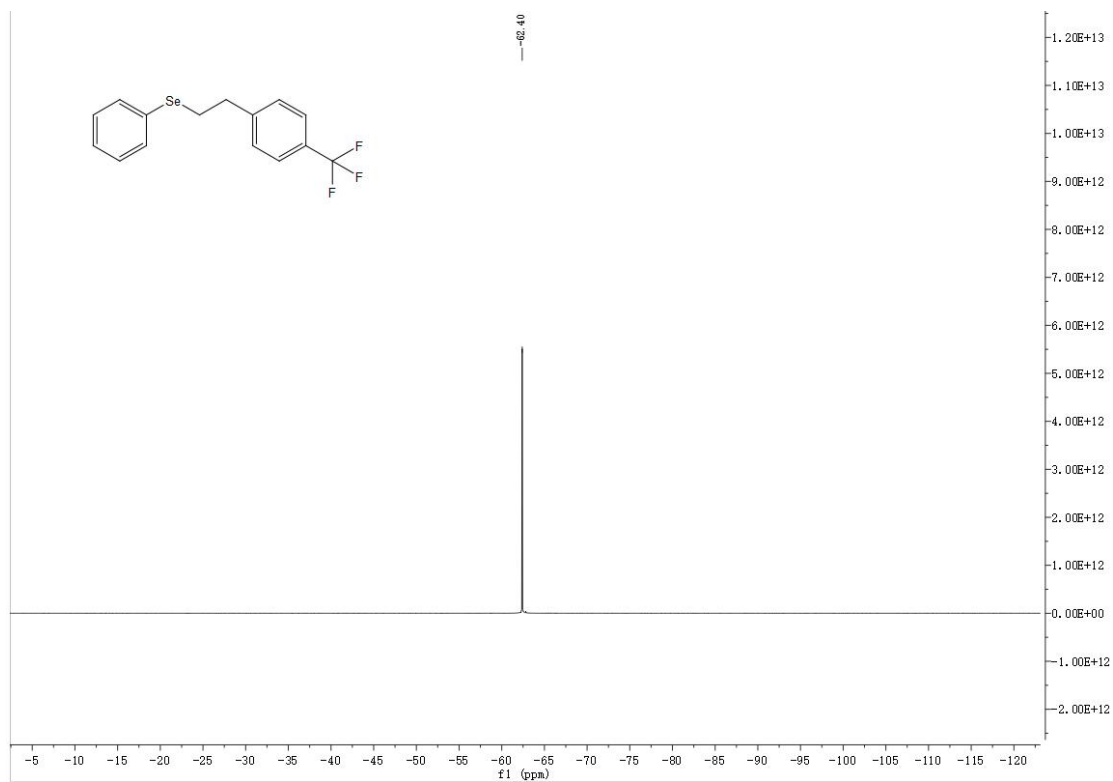


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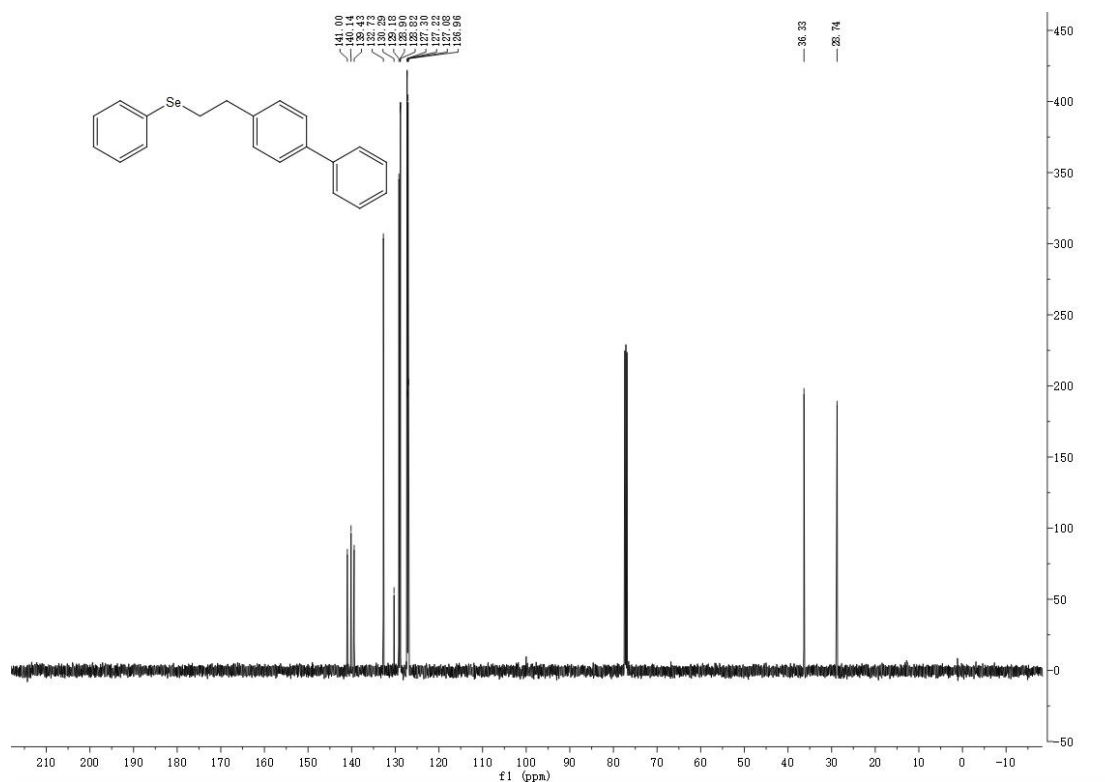
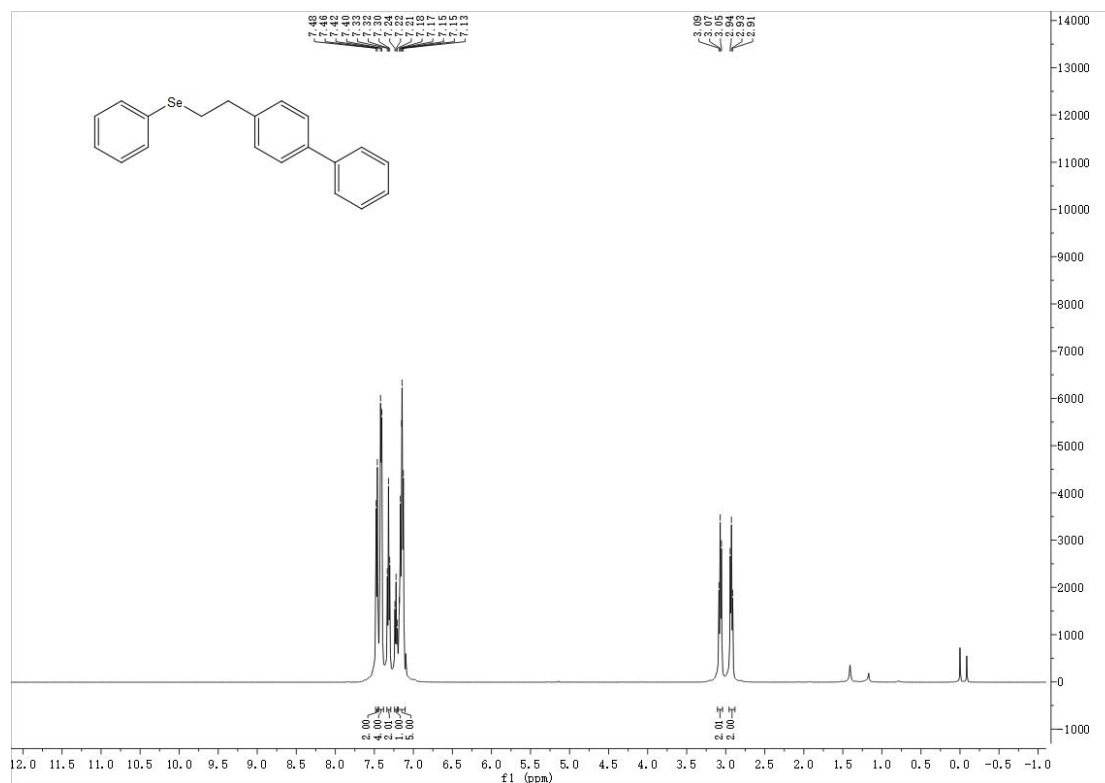




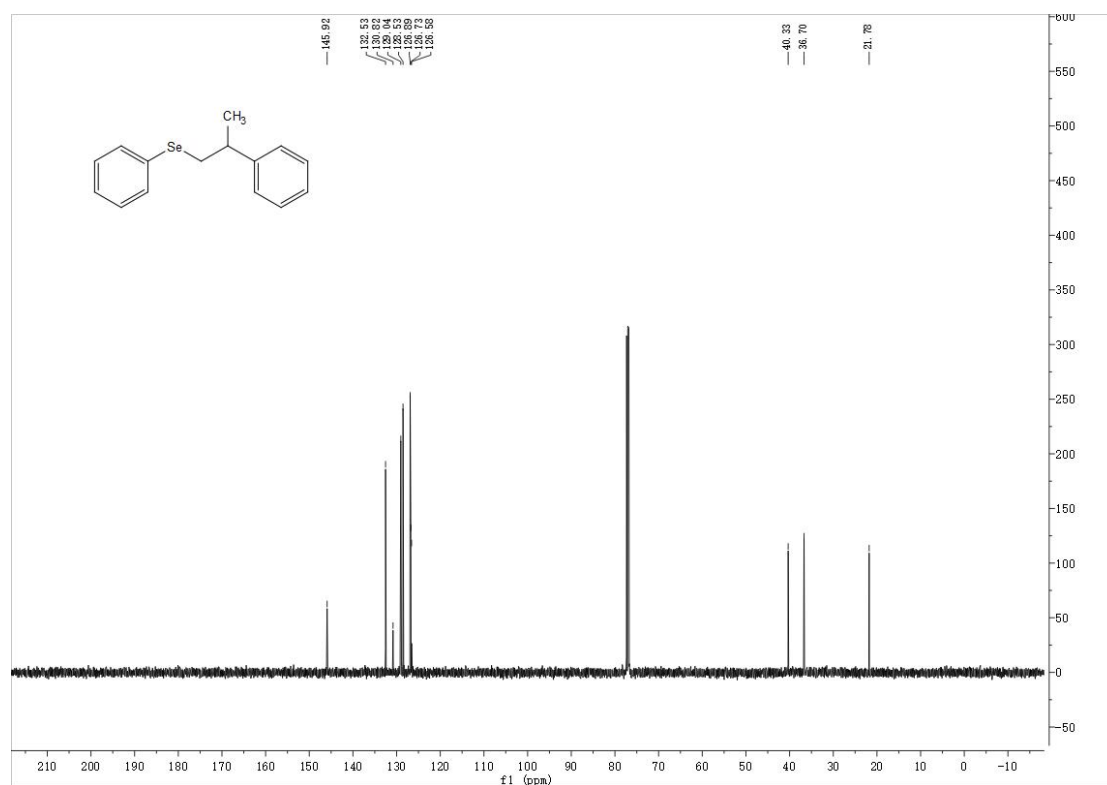
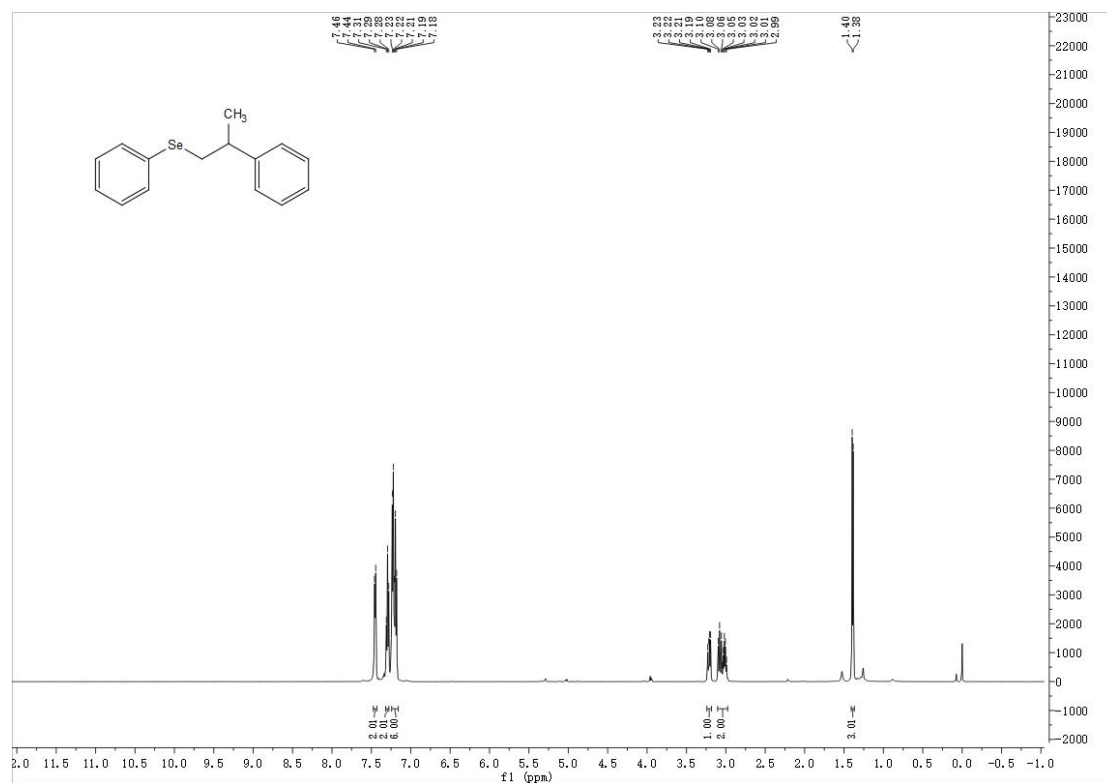




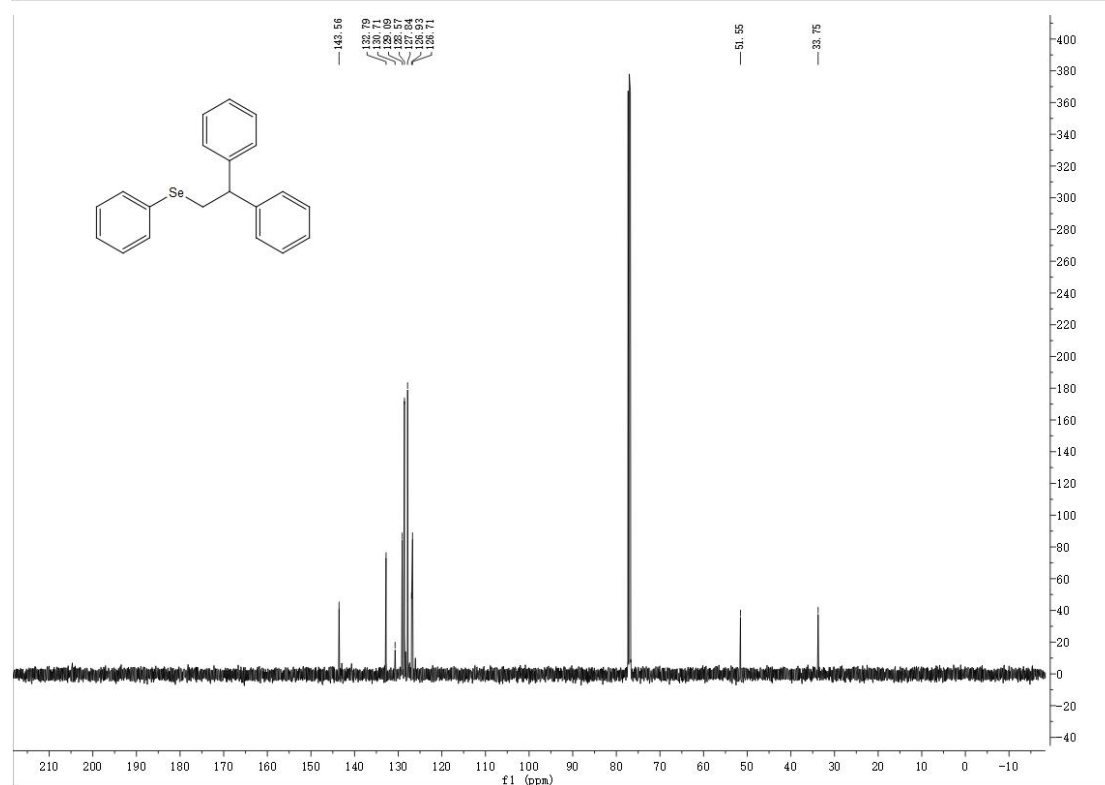
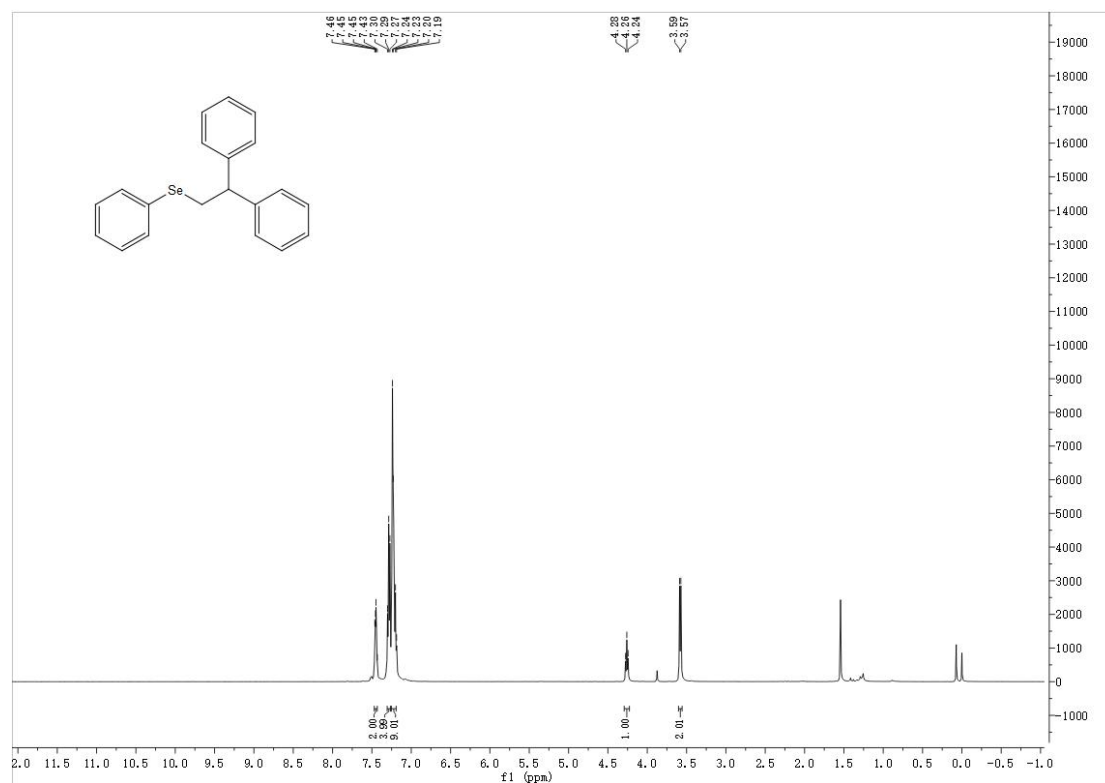
# Compound (10)



**Compound (11)**

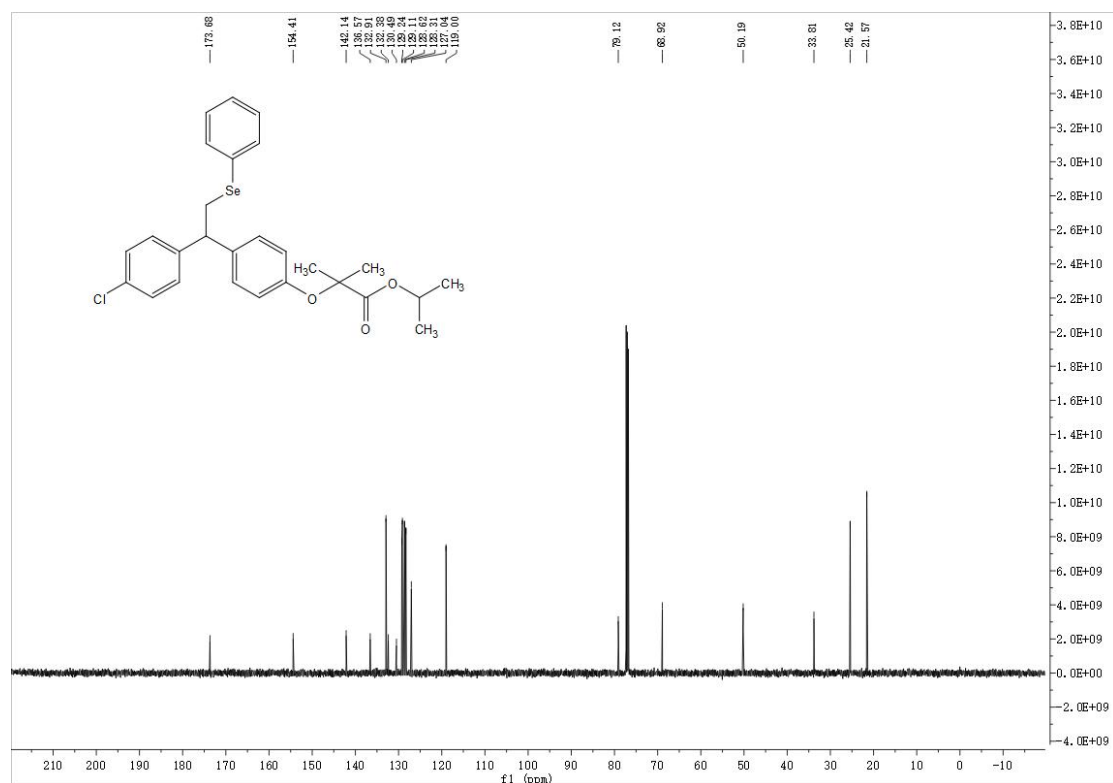
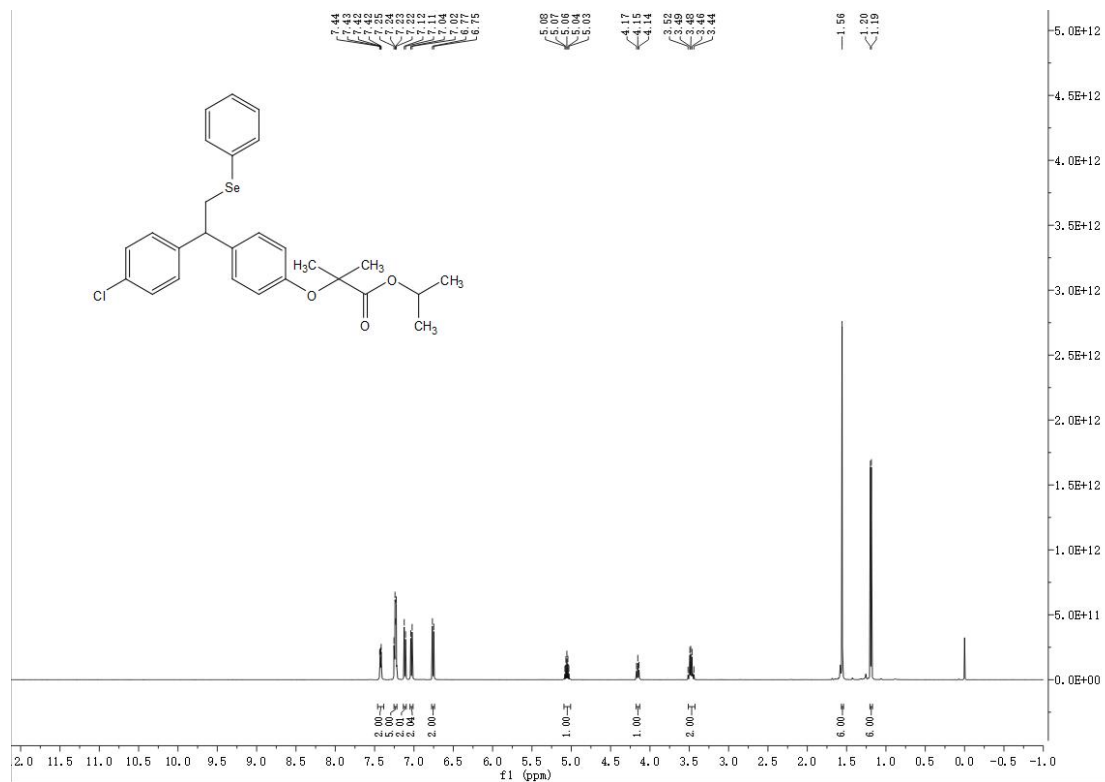


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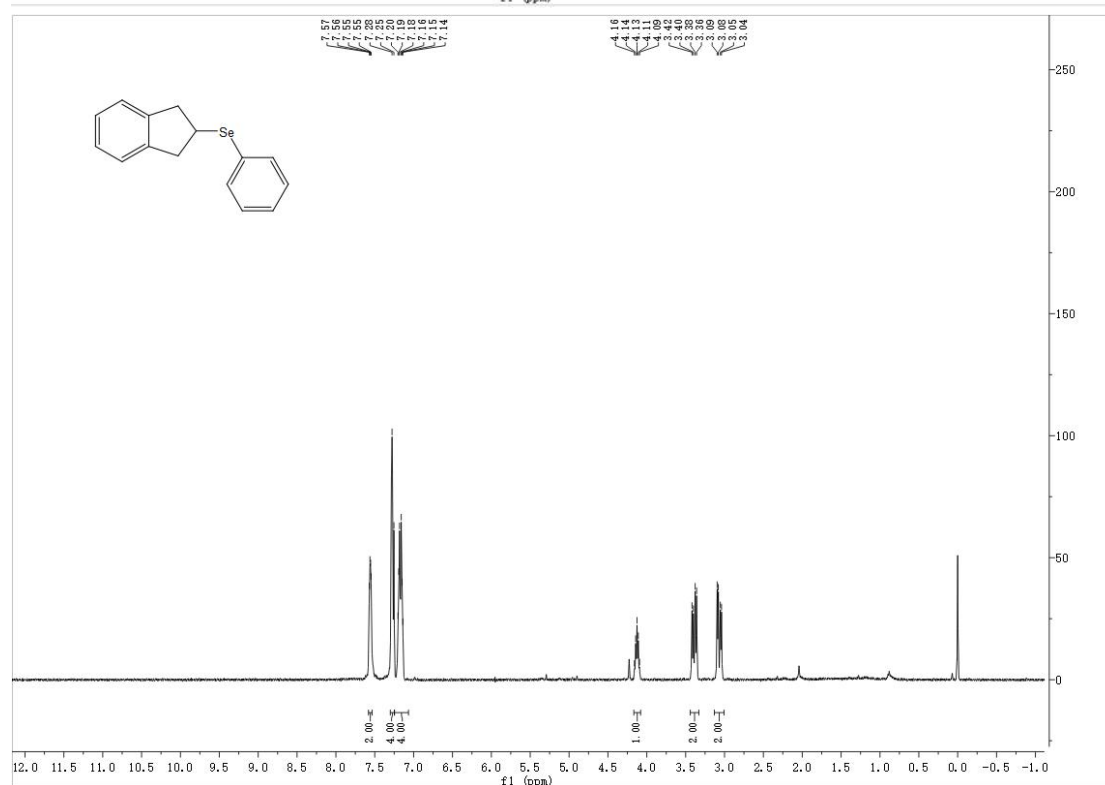
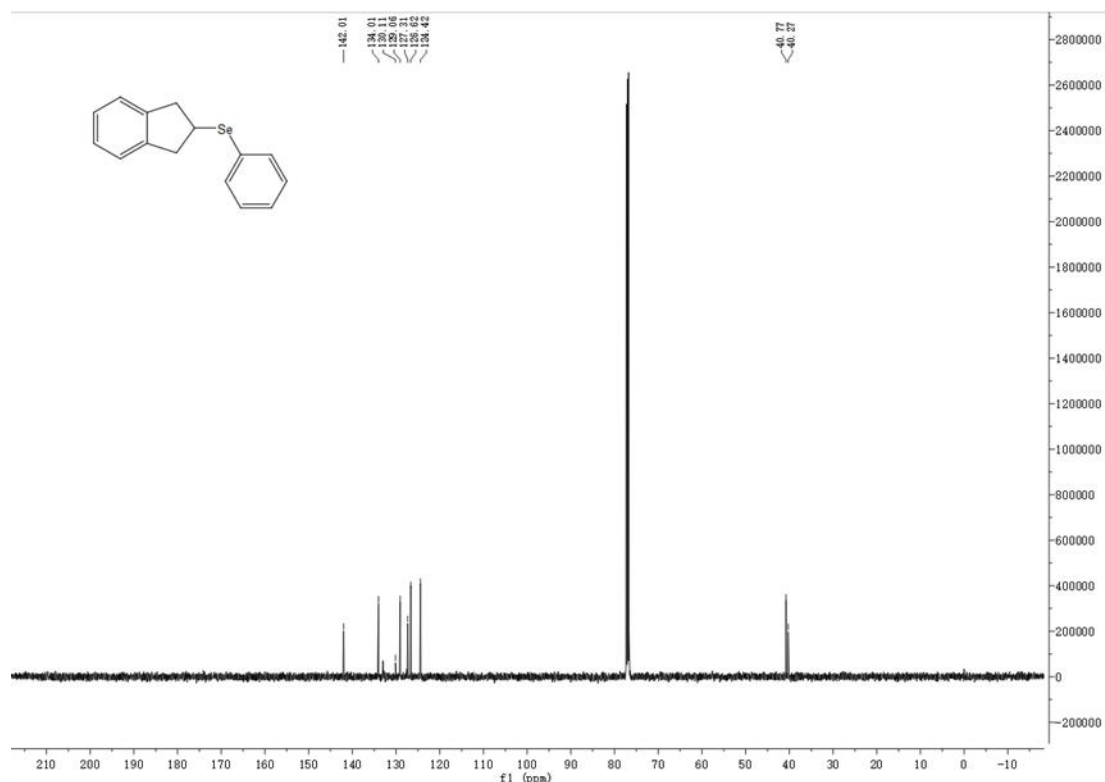




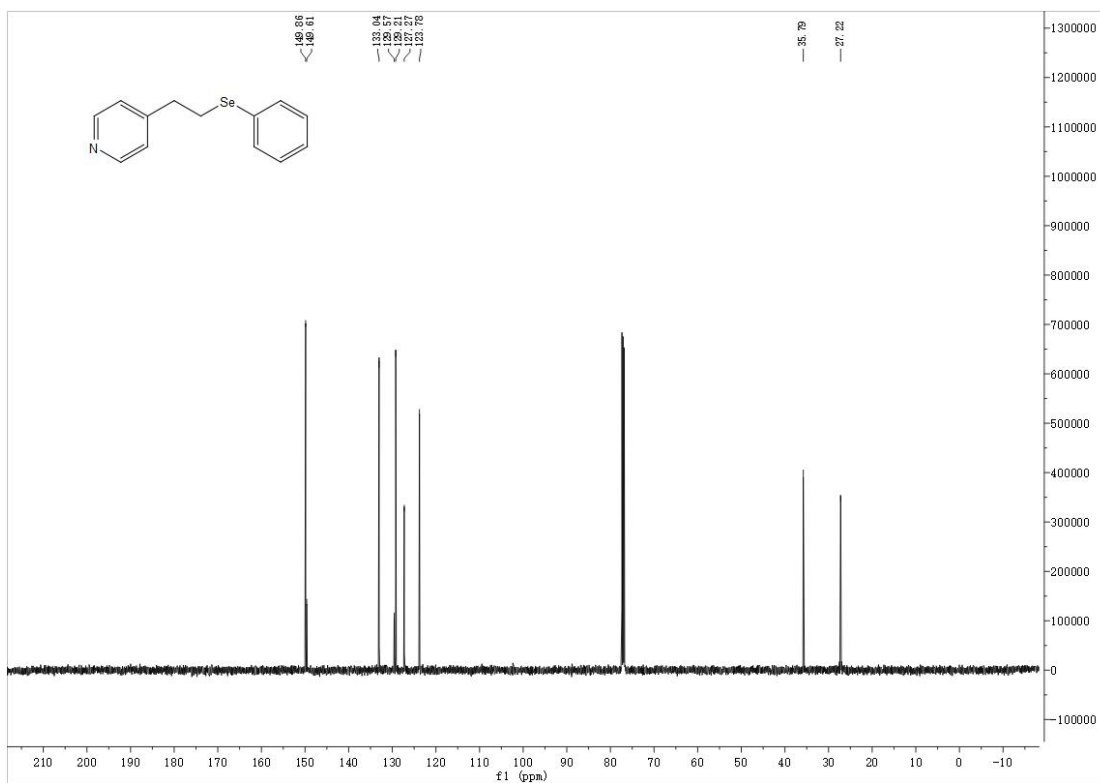
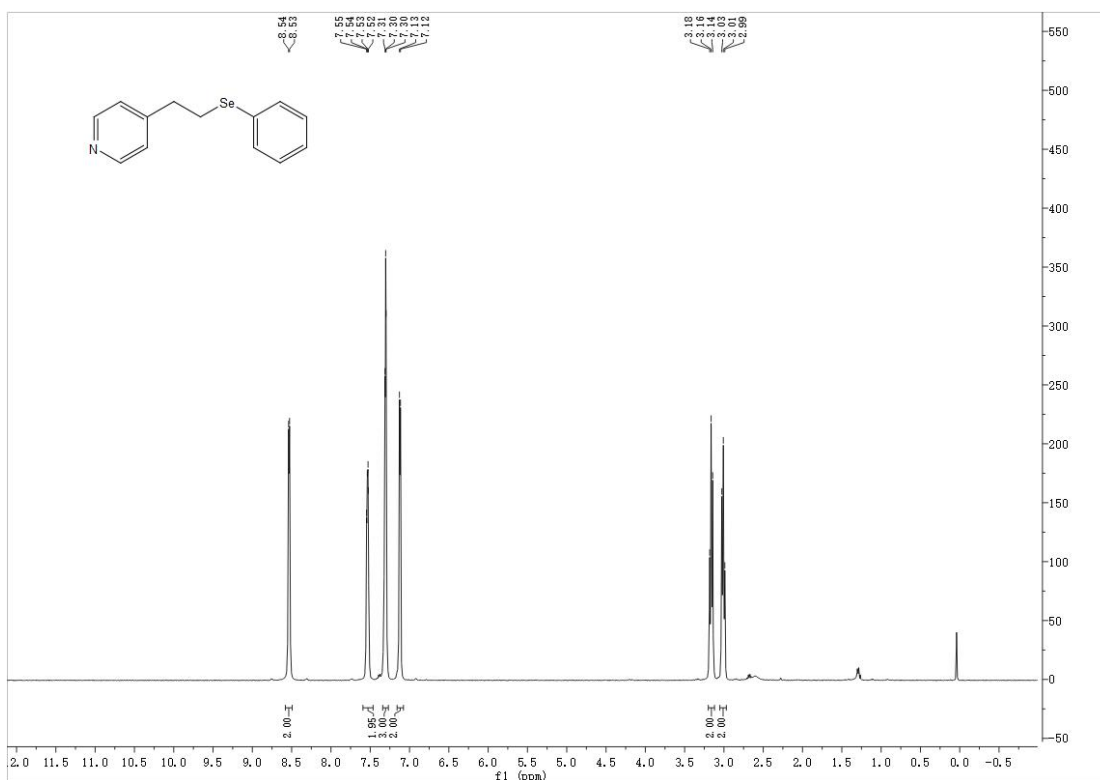
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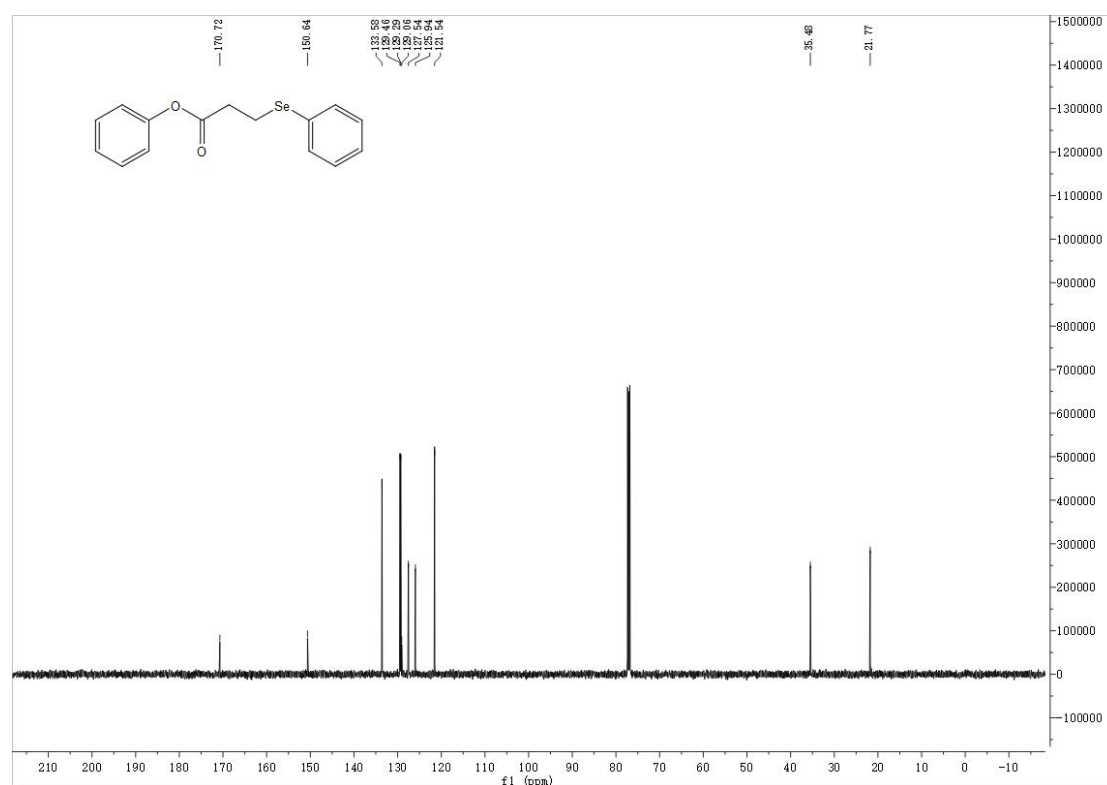
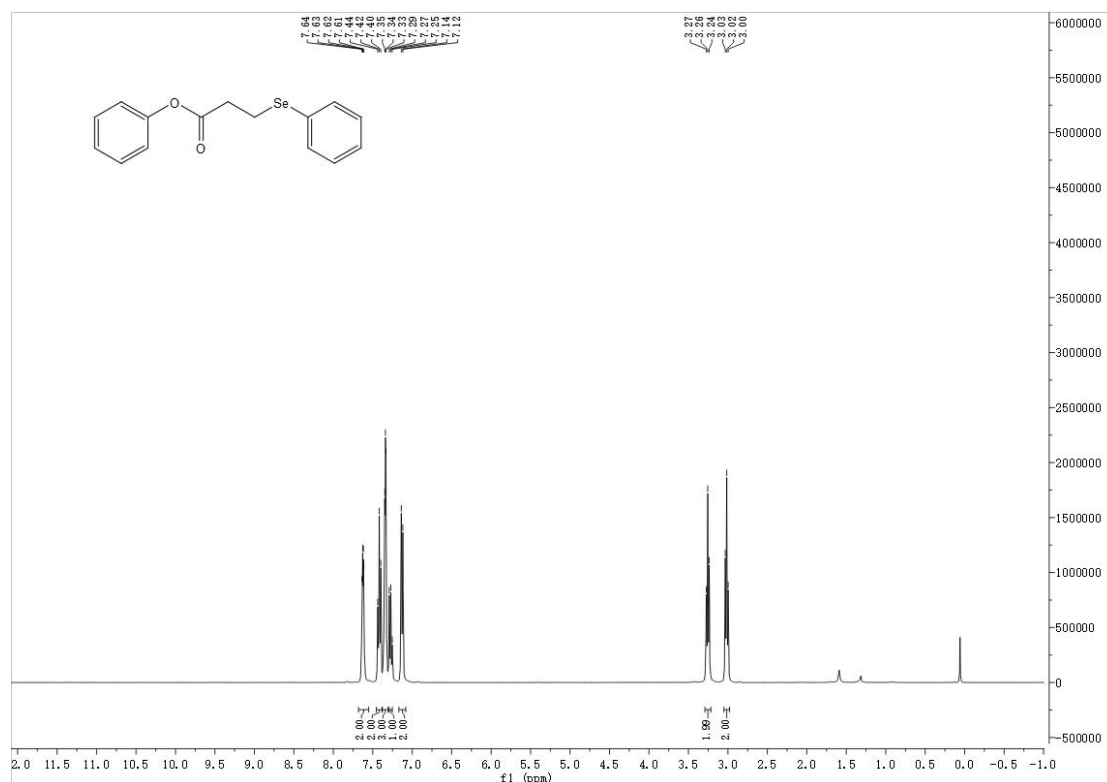
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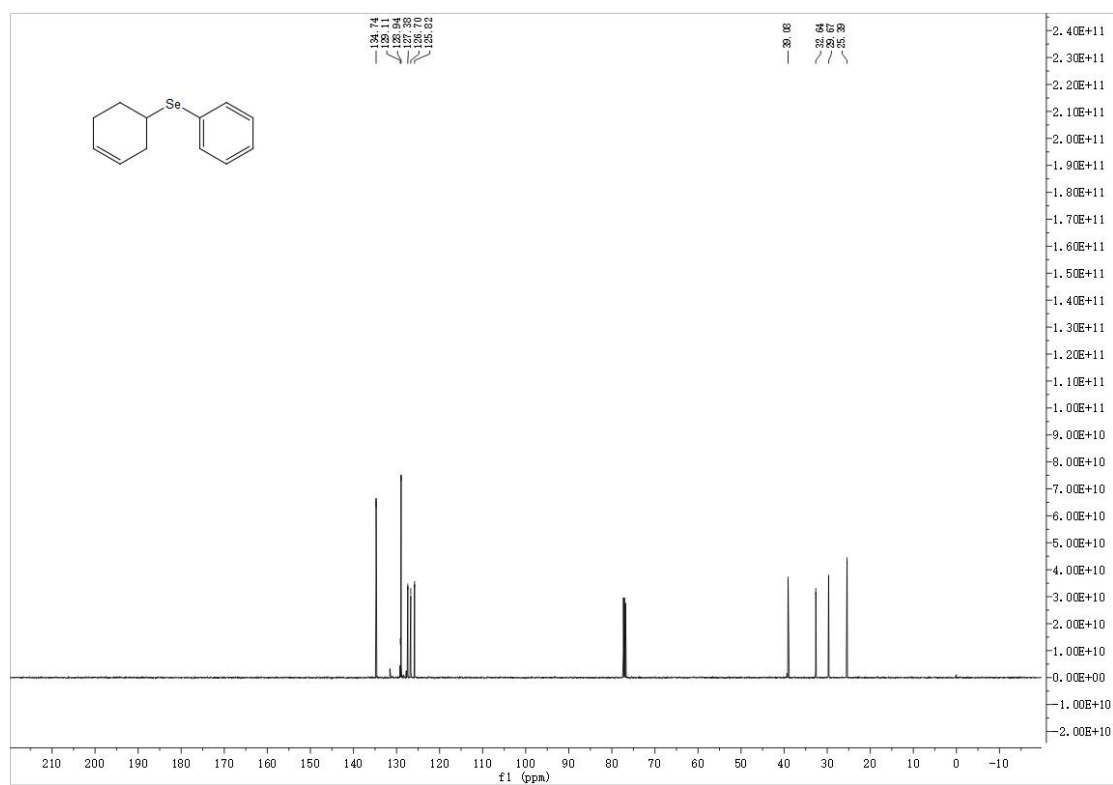
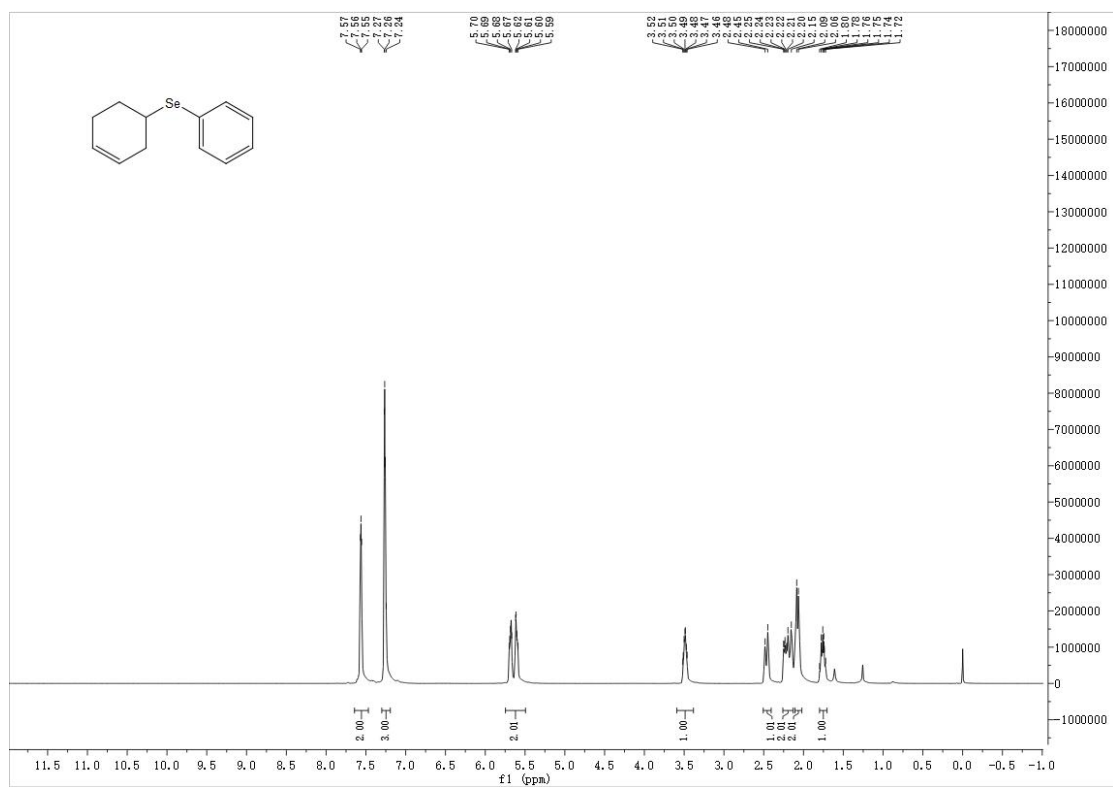
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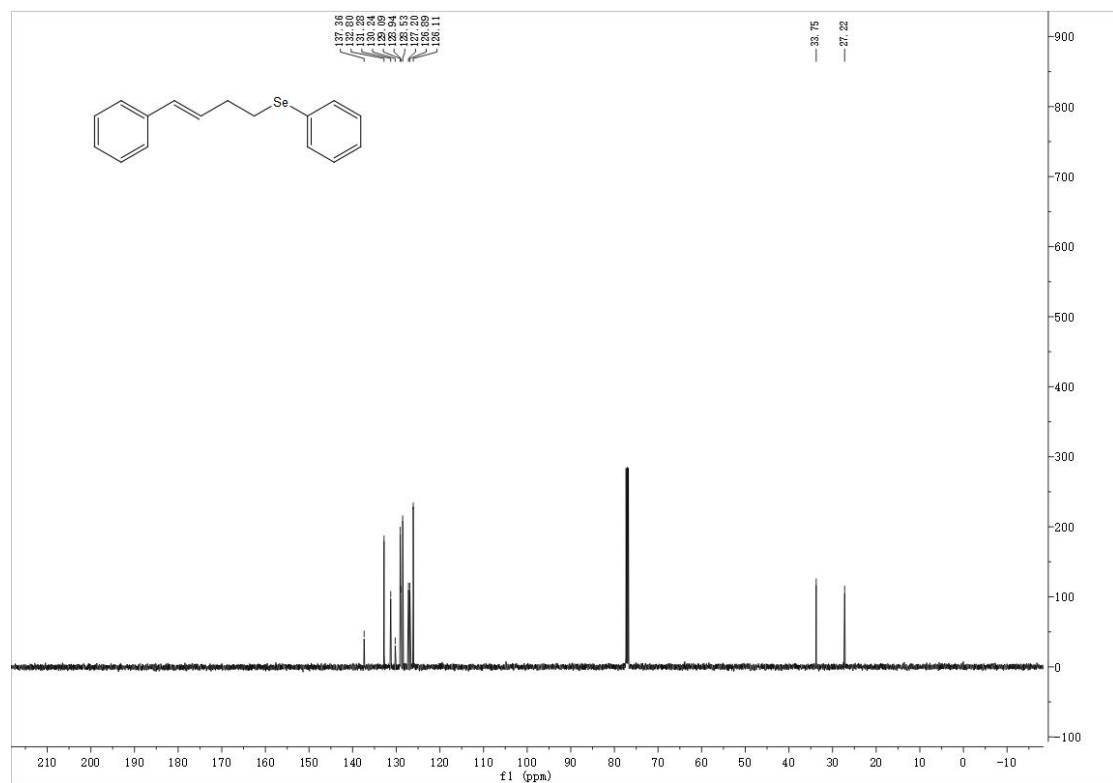
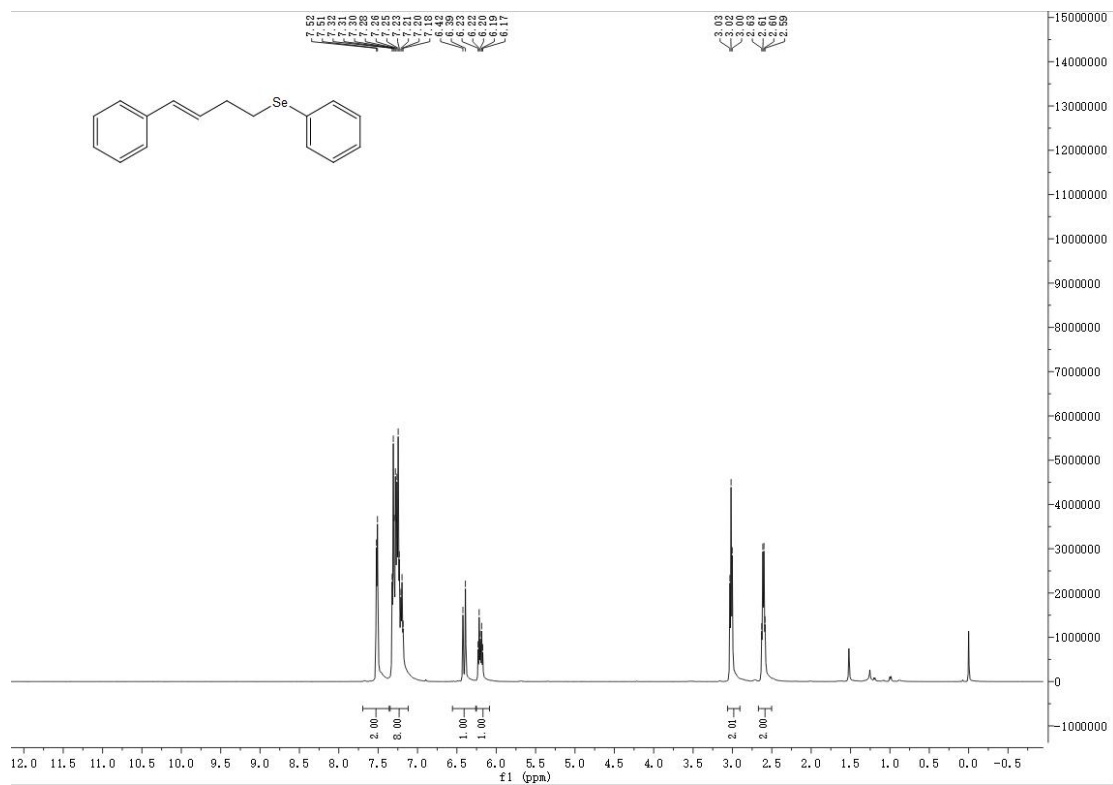
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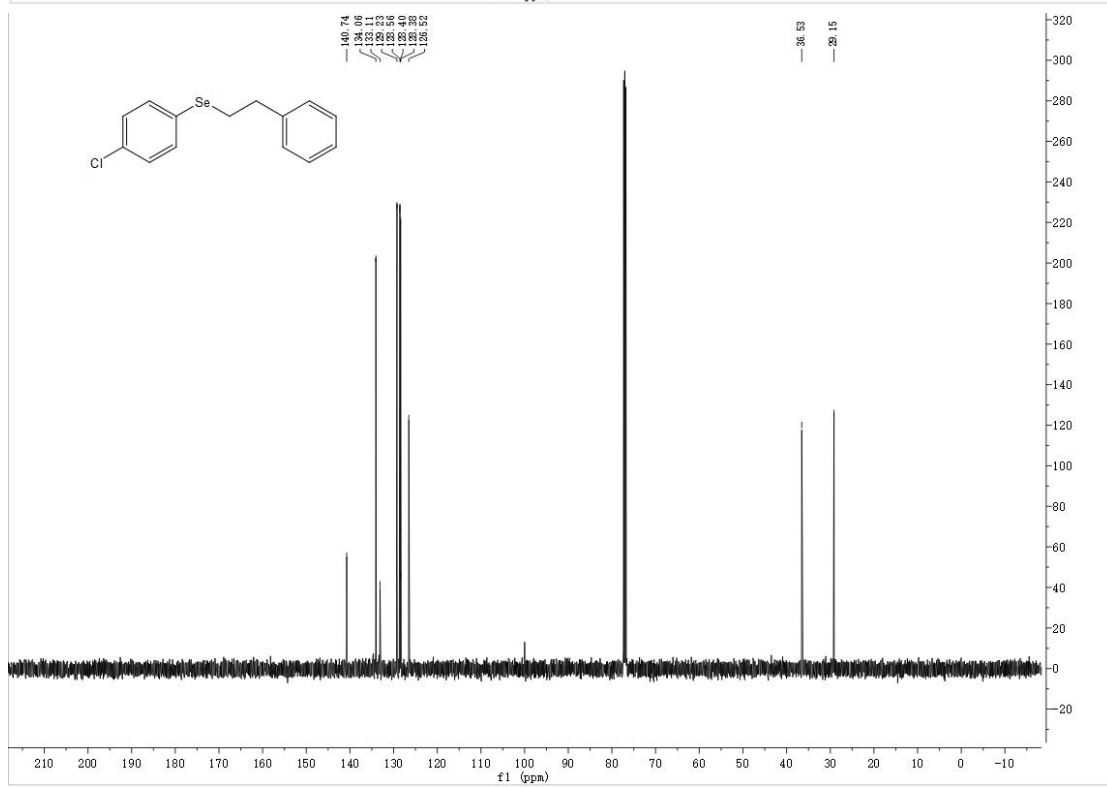
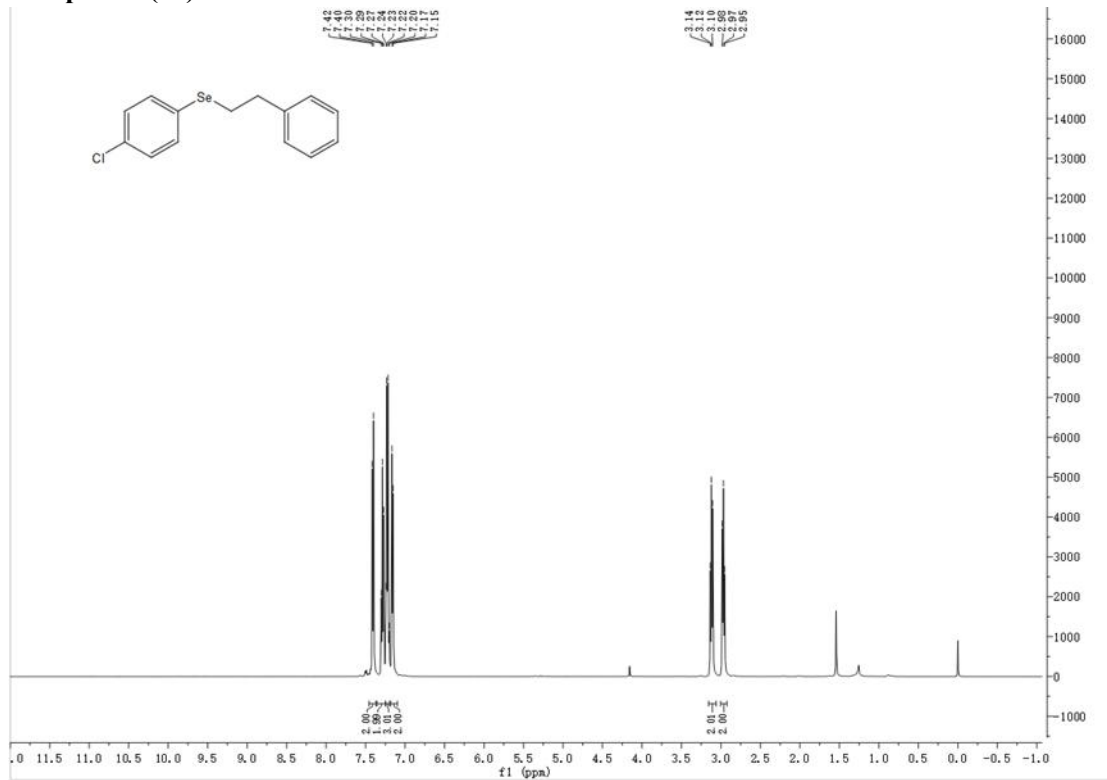
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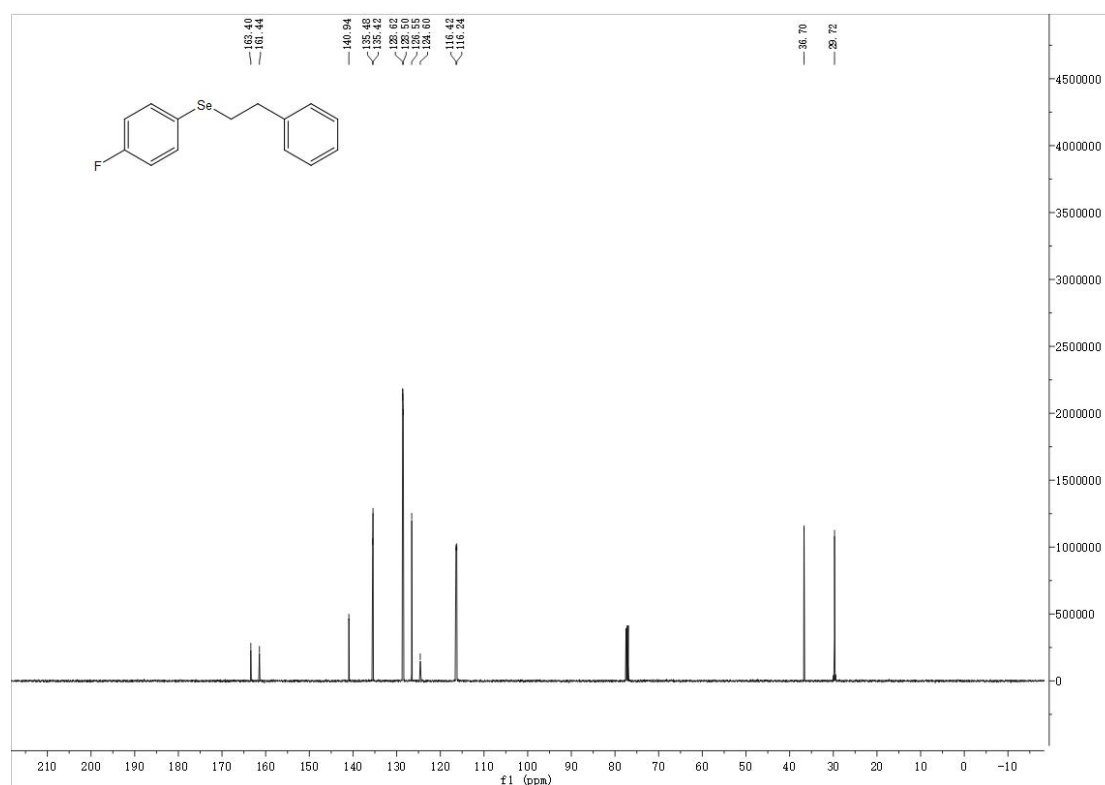
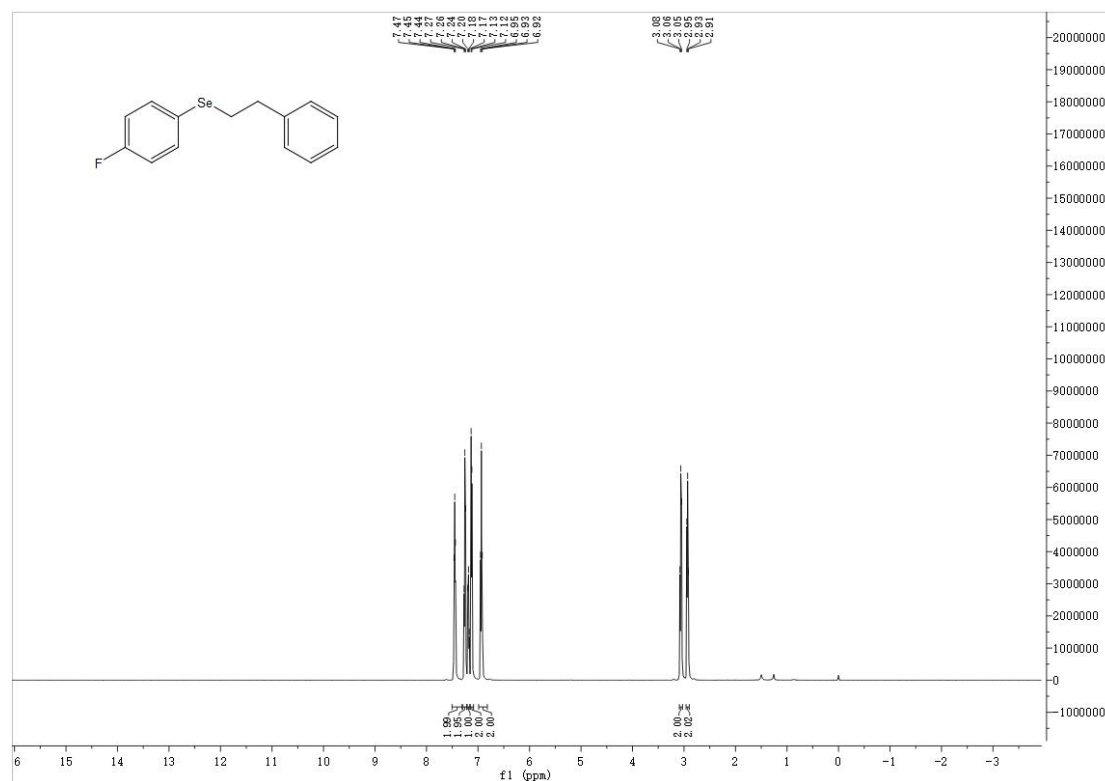
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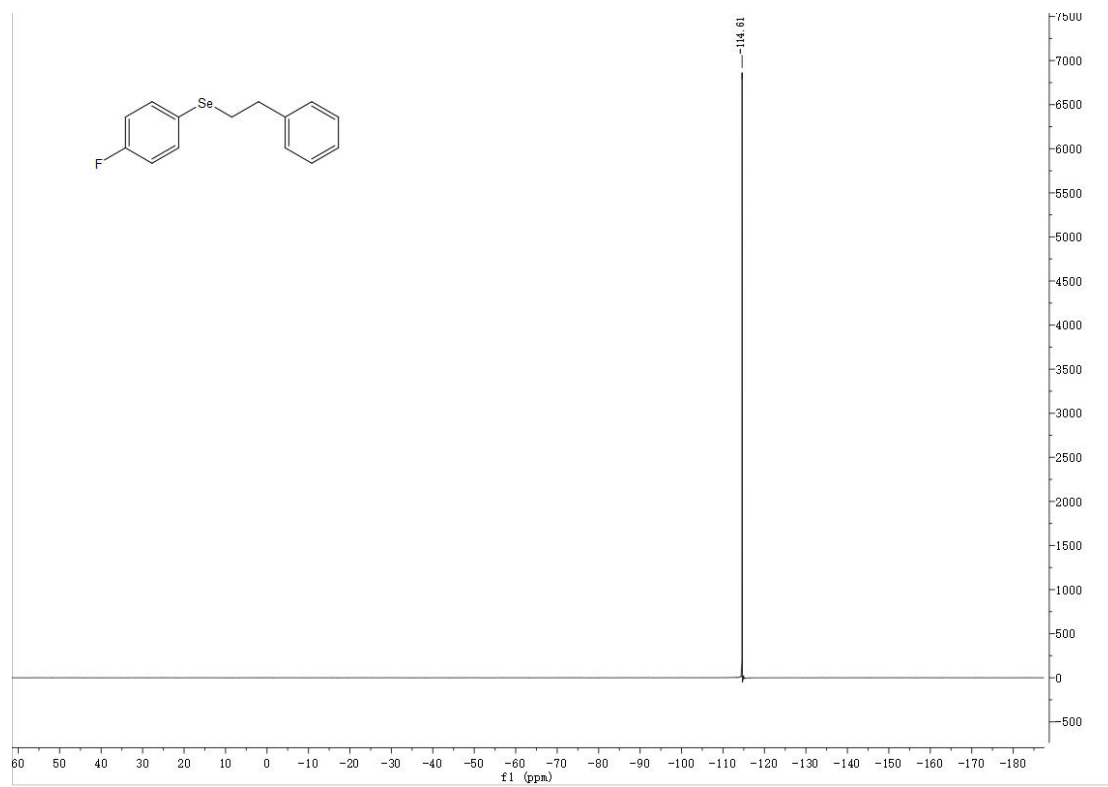
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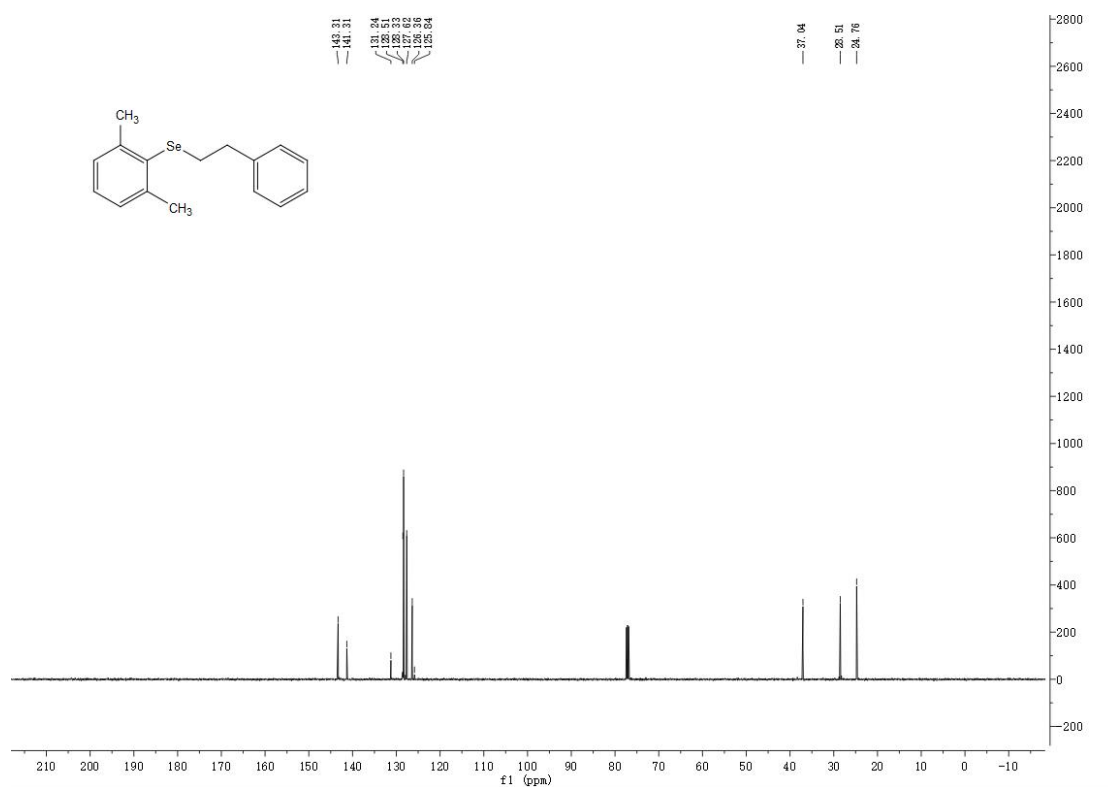
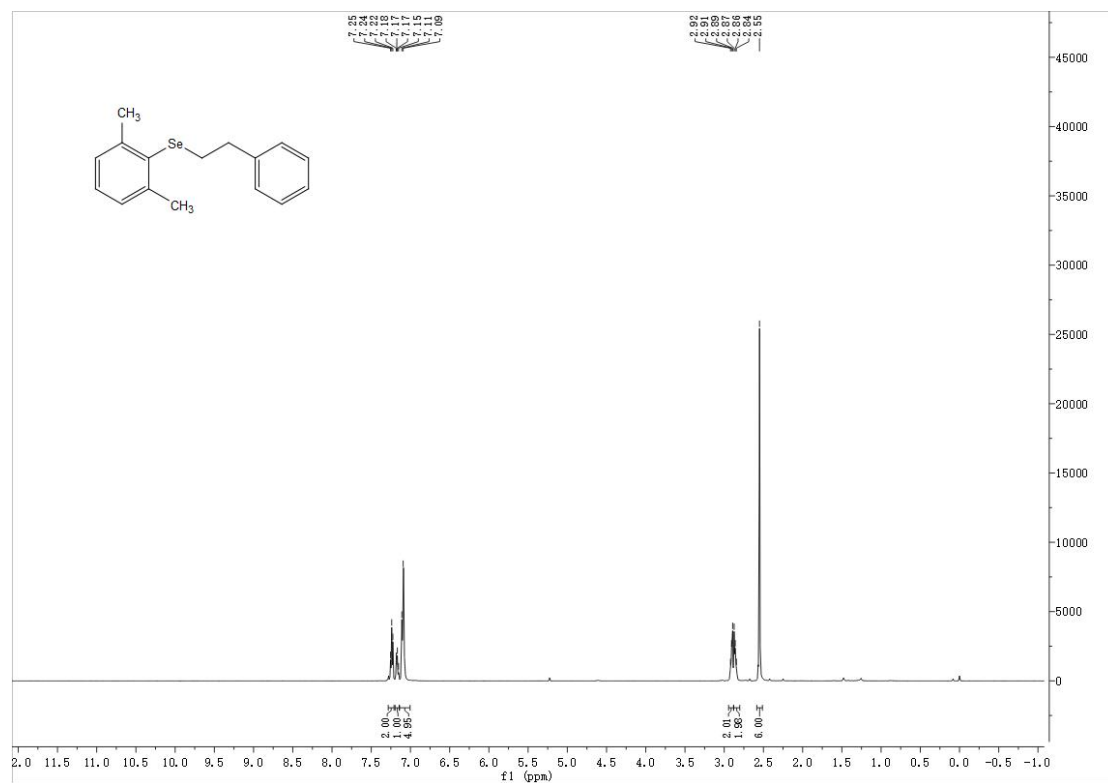
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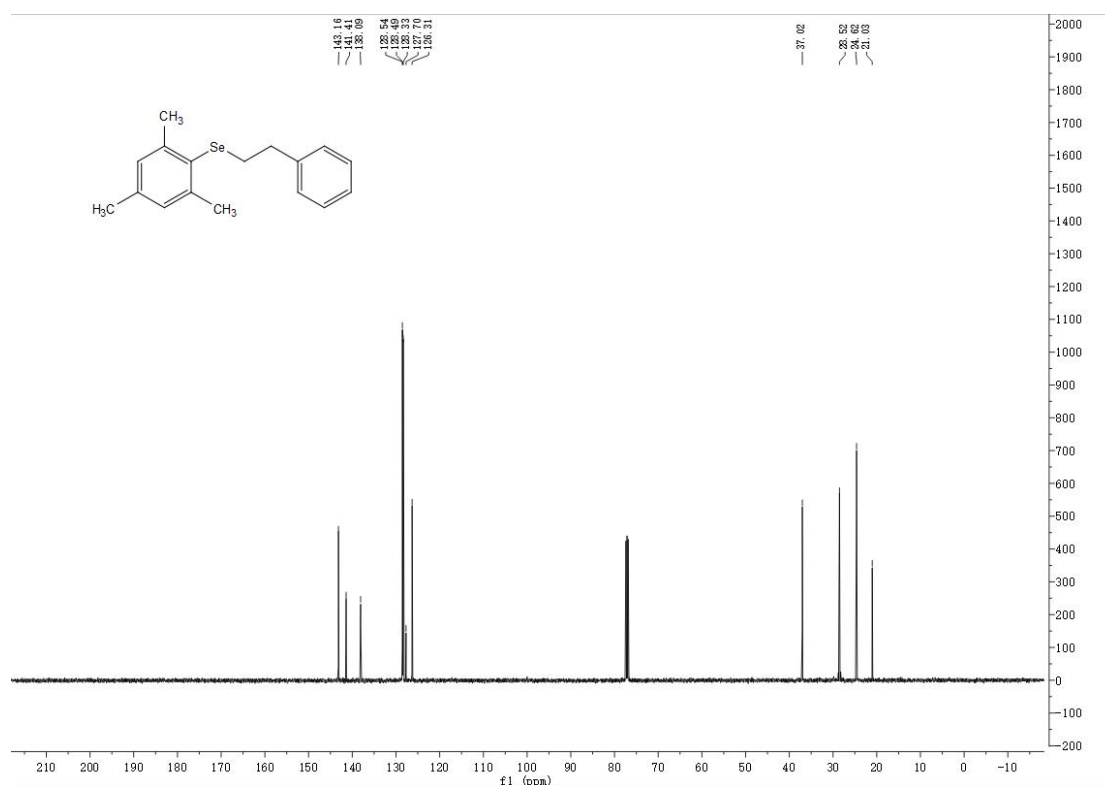
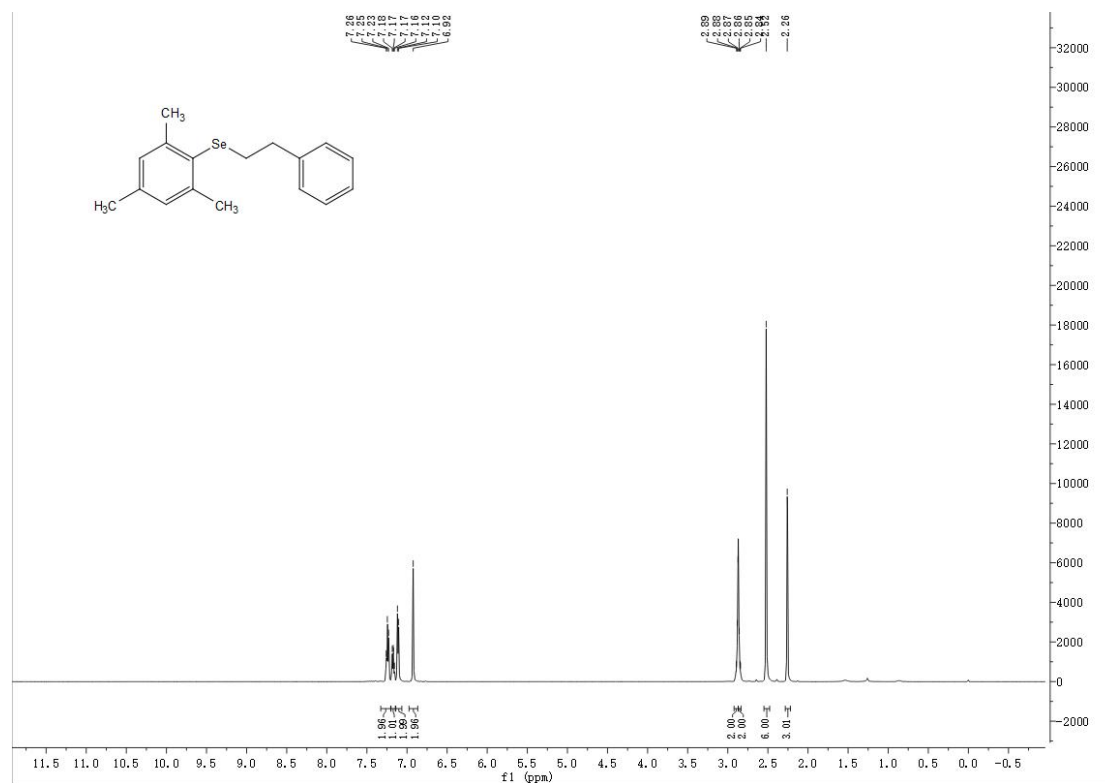




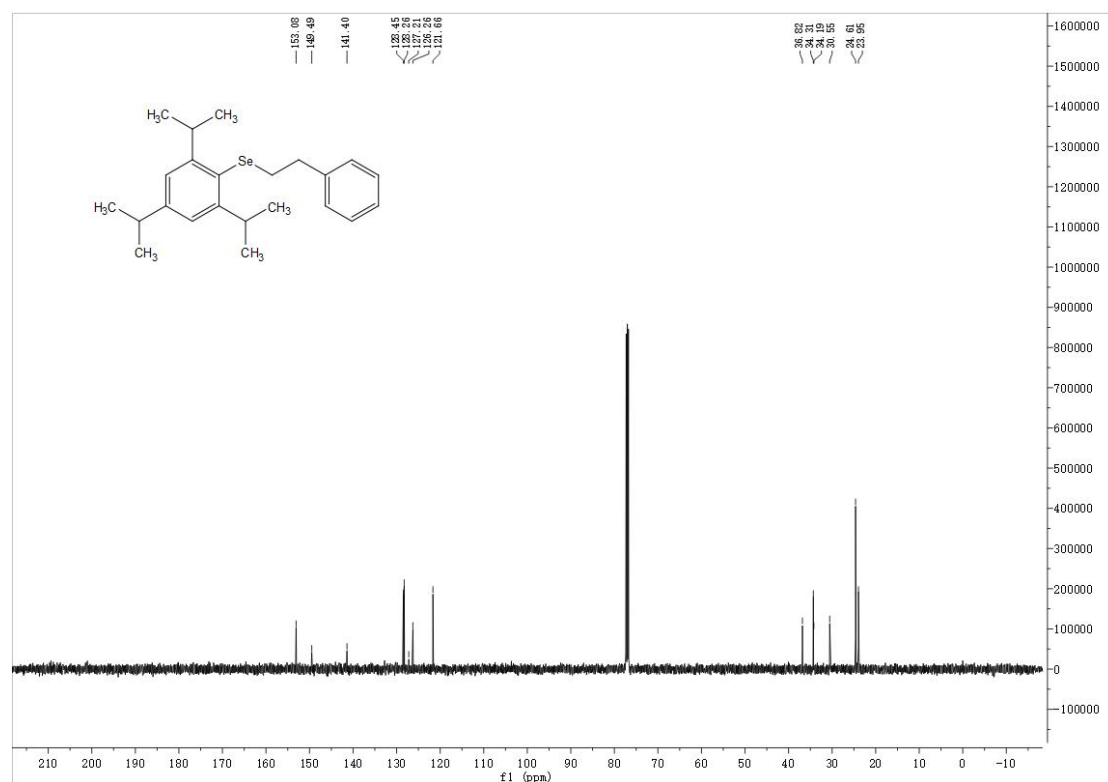
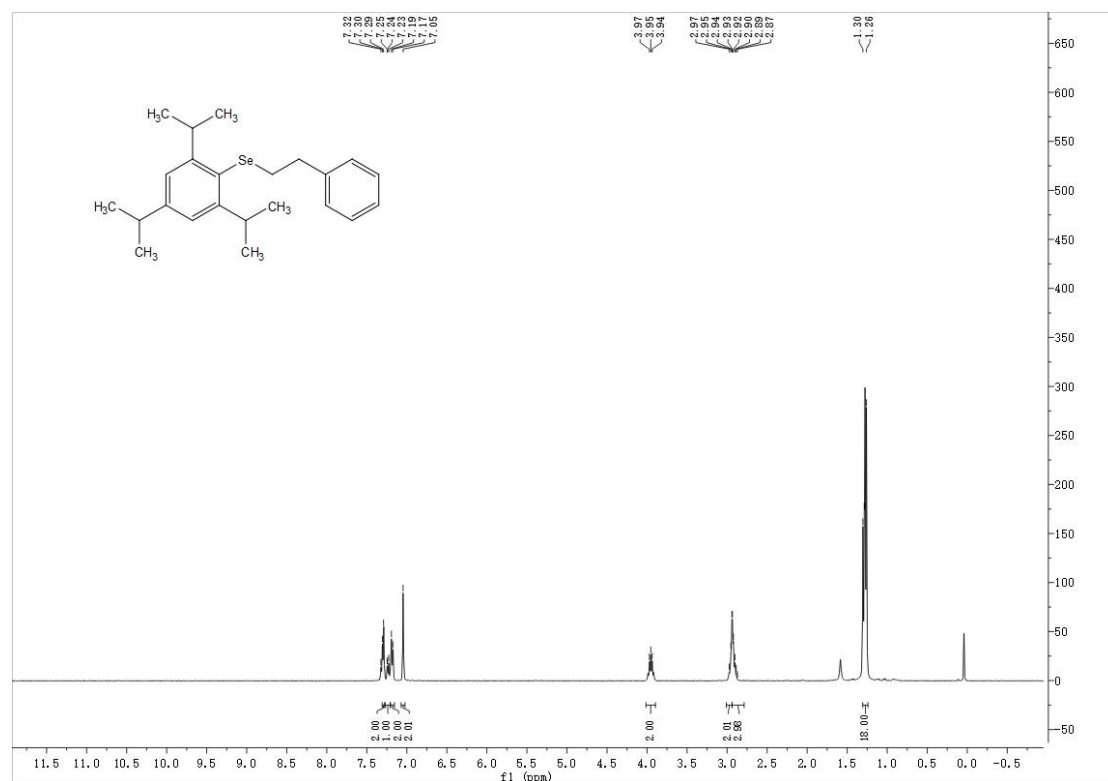
# Compound (21)



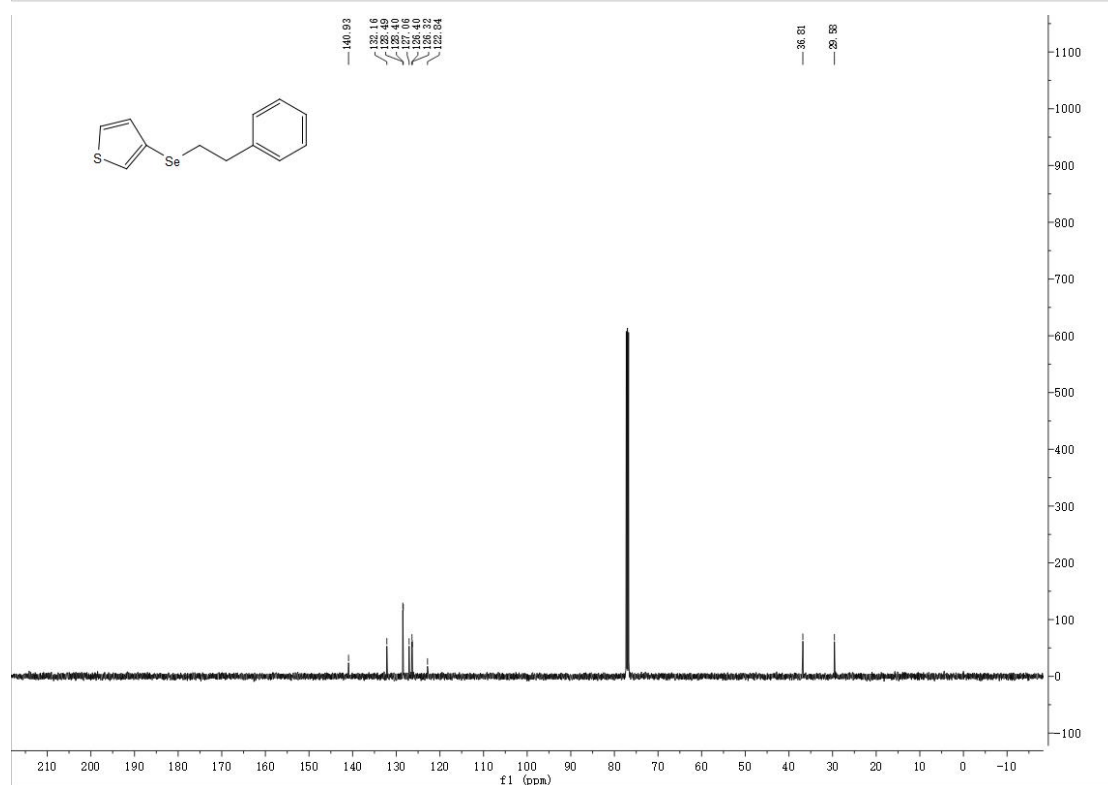
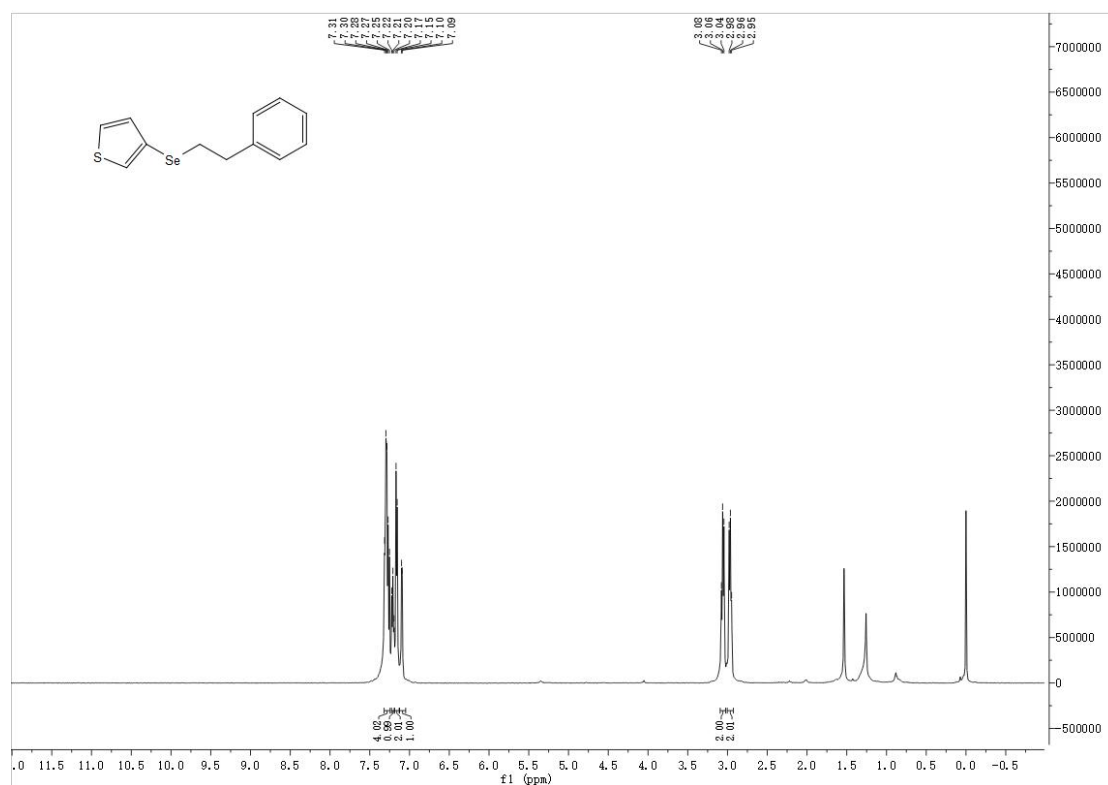
# Compound (22)



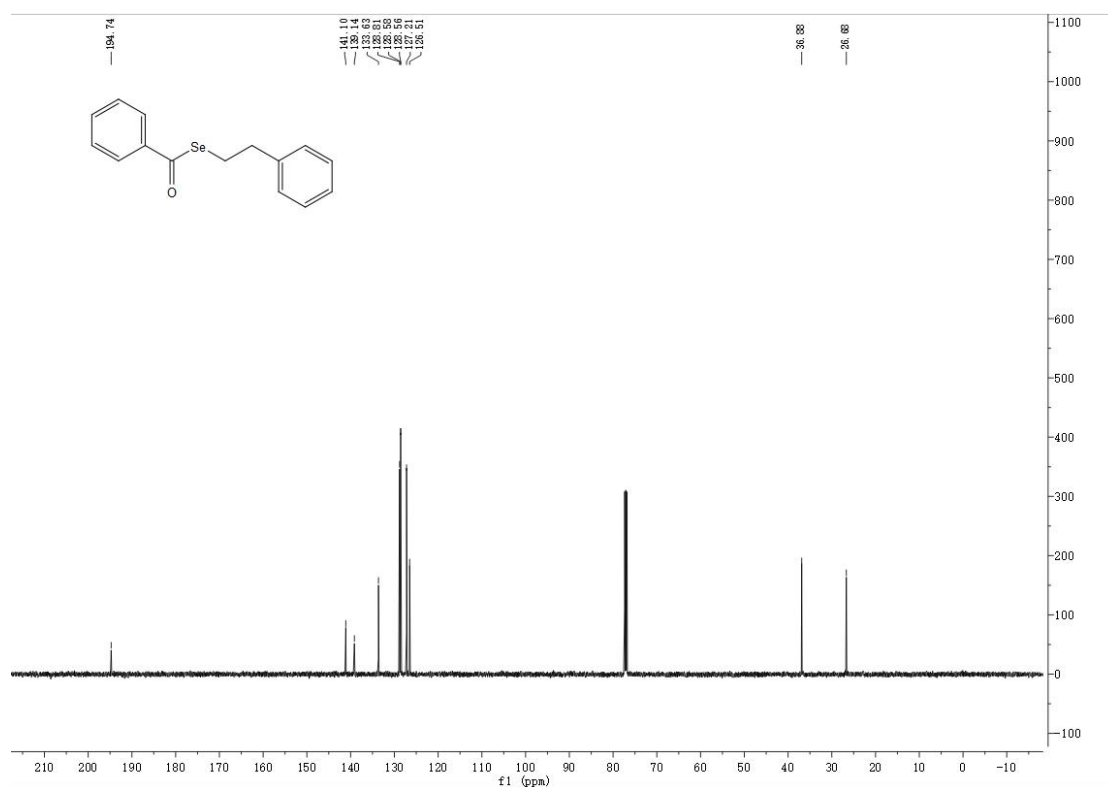
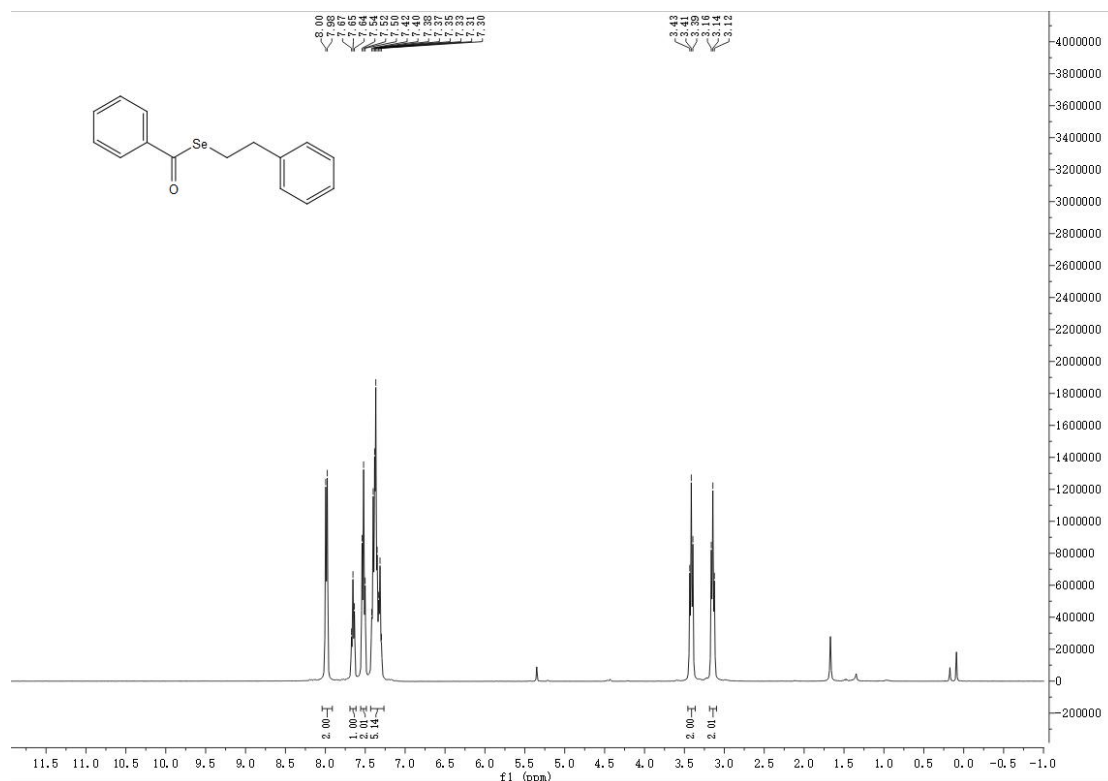
# Compound (23)



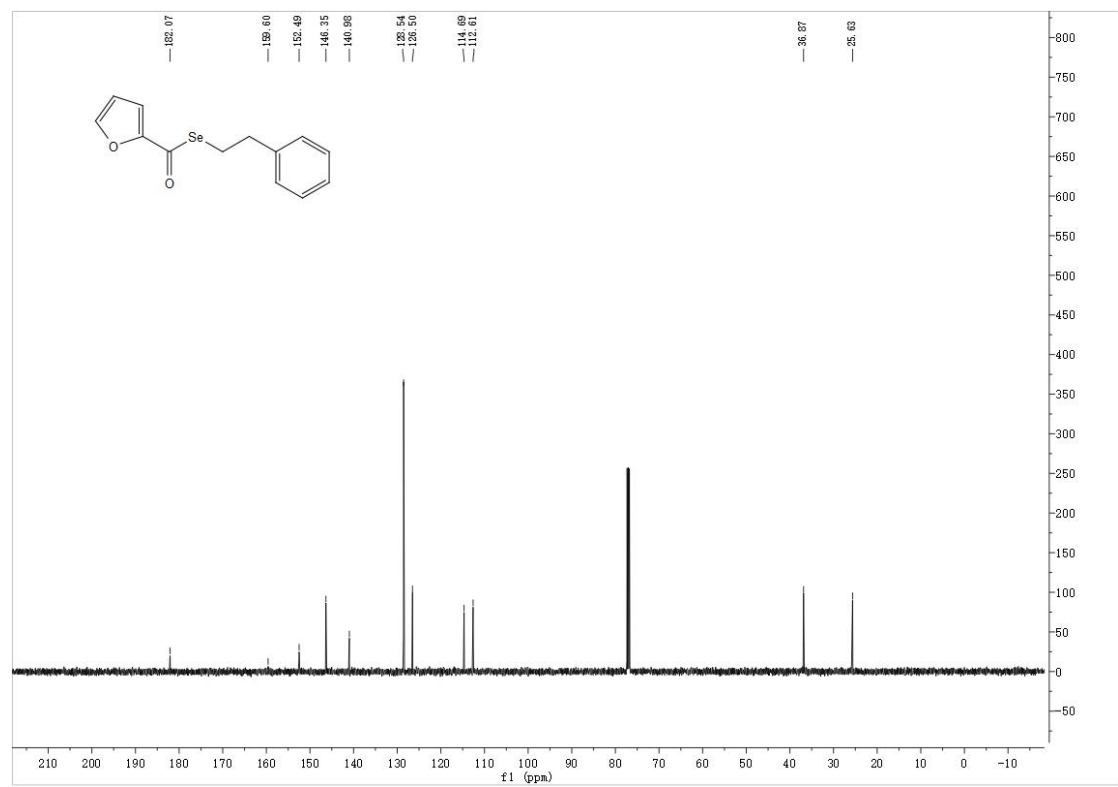
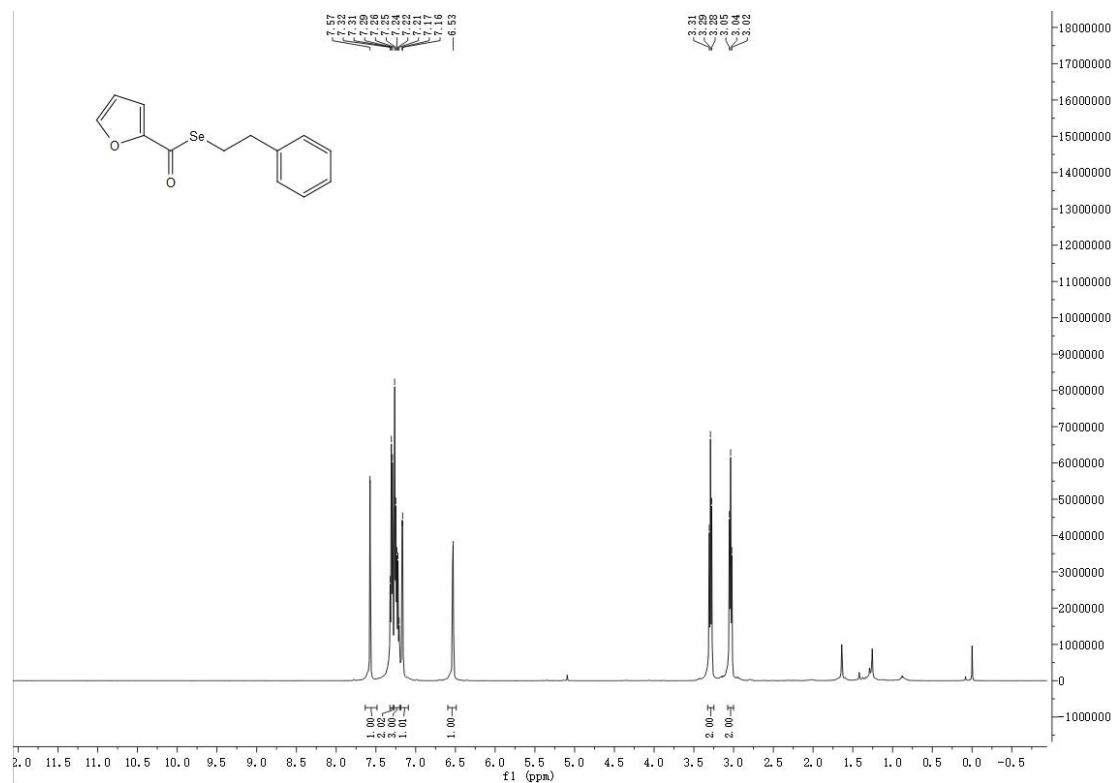
# Compound (24)



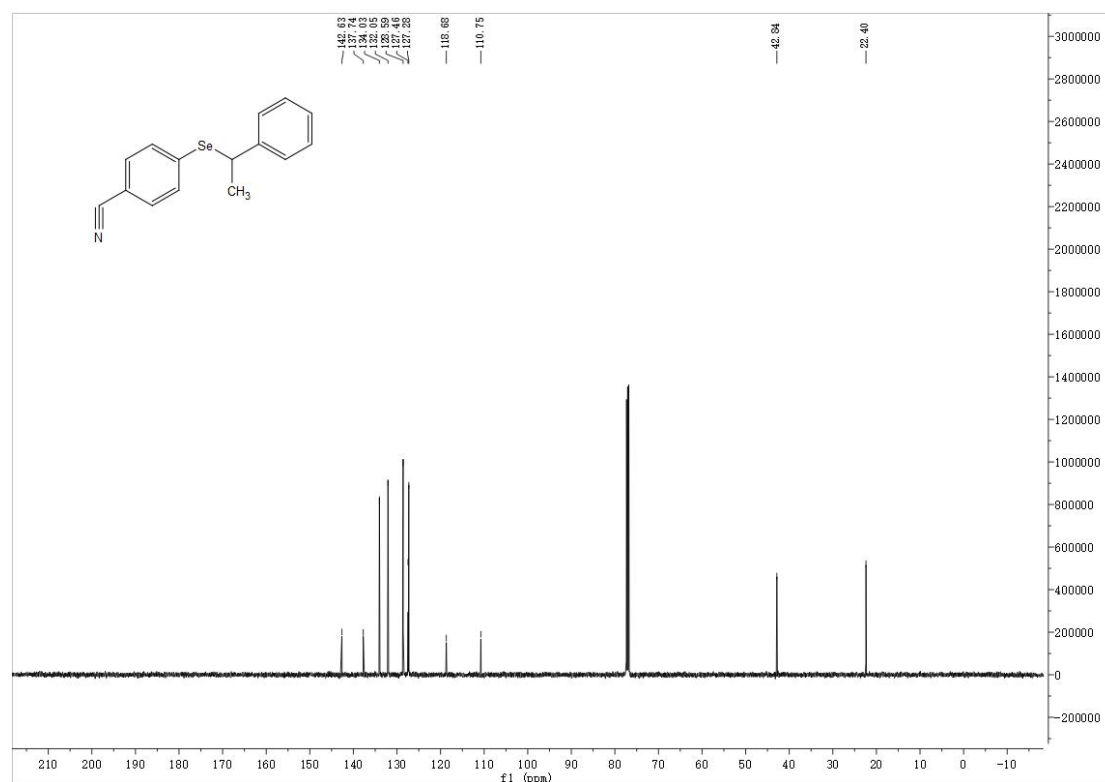
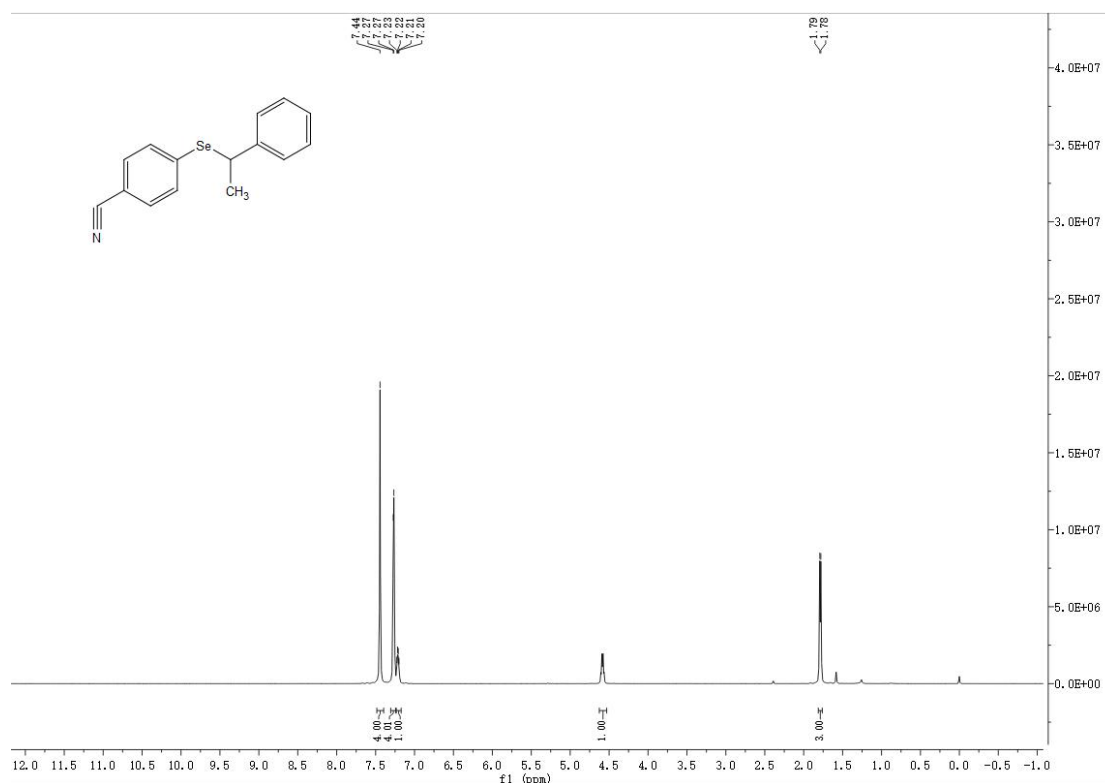
# Compound (25)



# Compound (26)

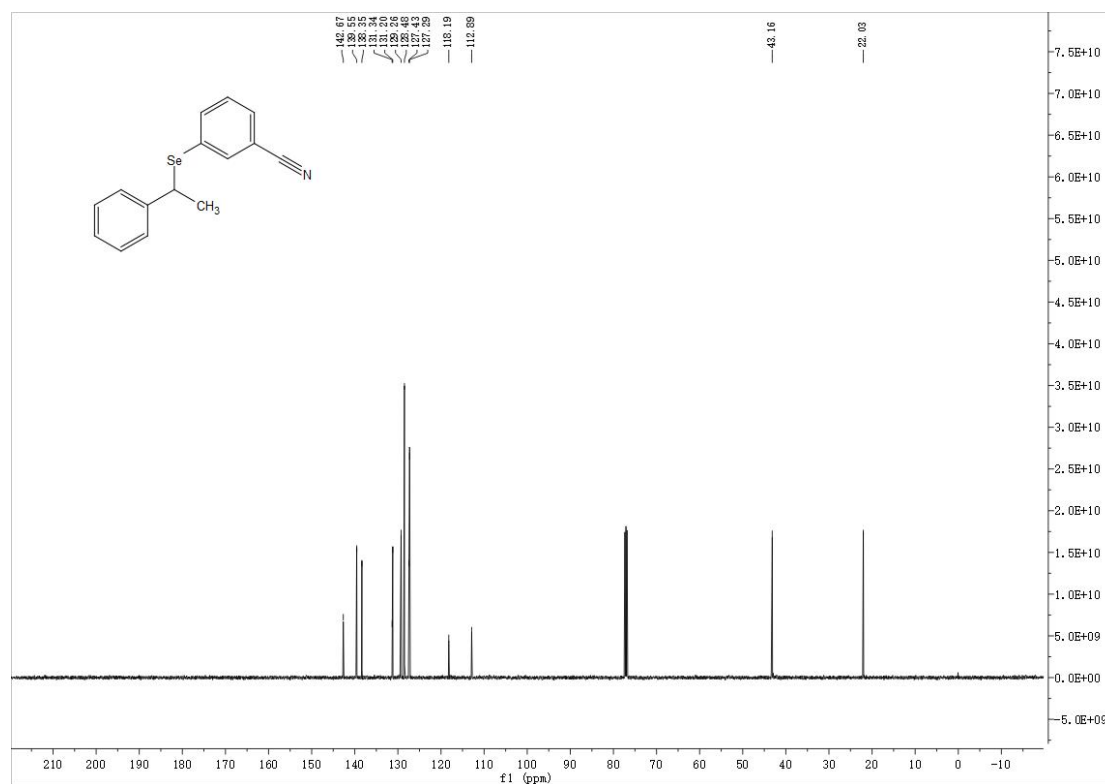
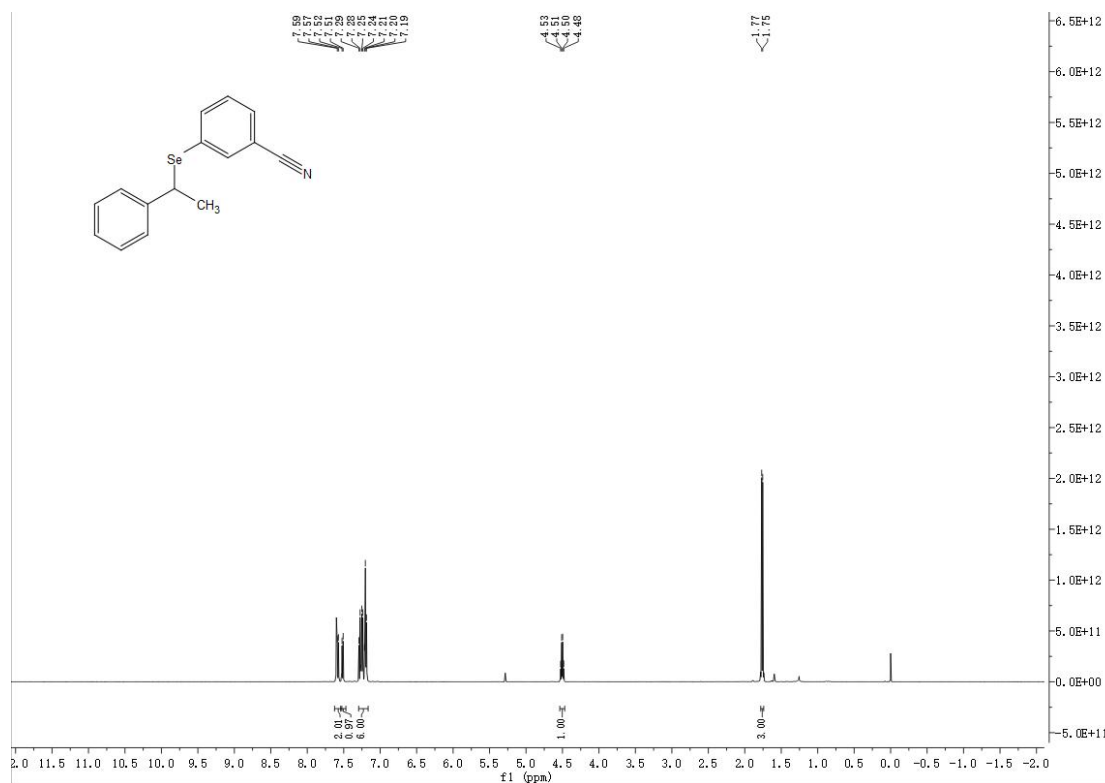


# Compound (28)

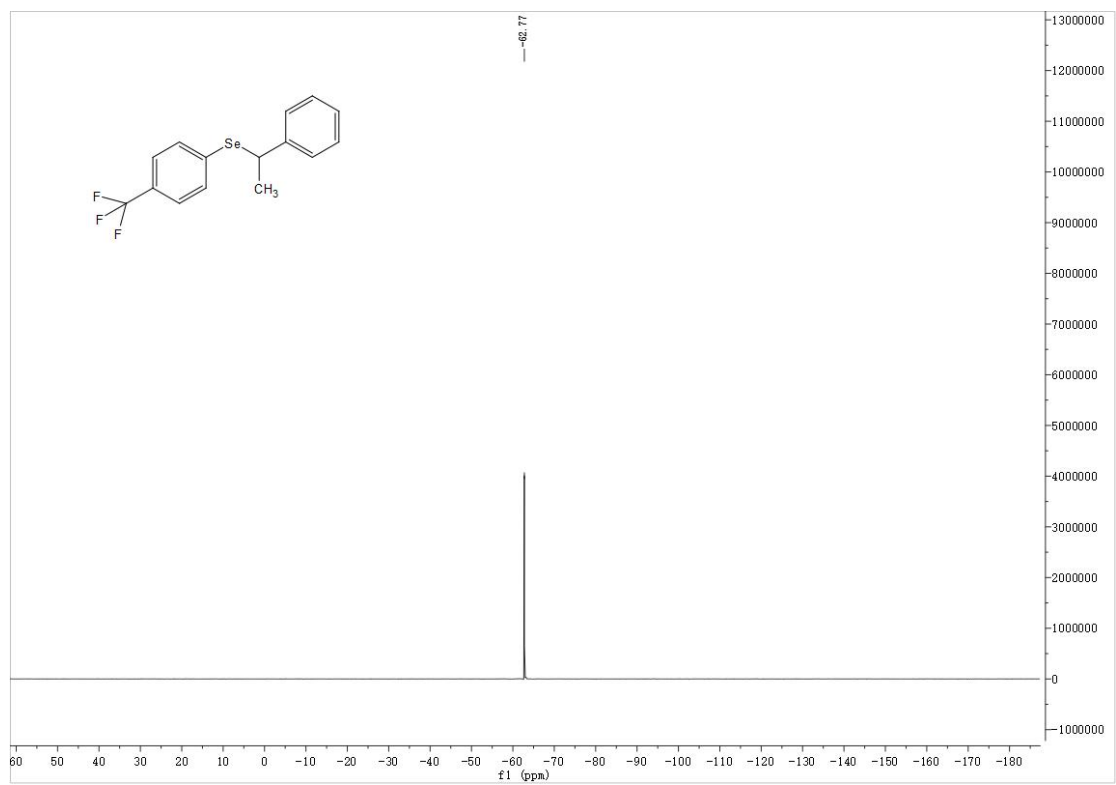




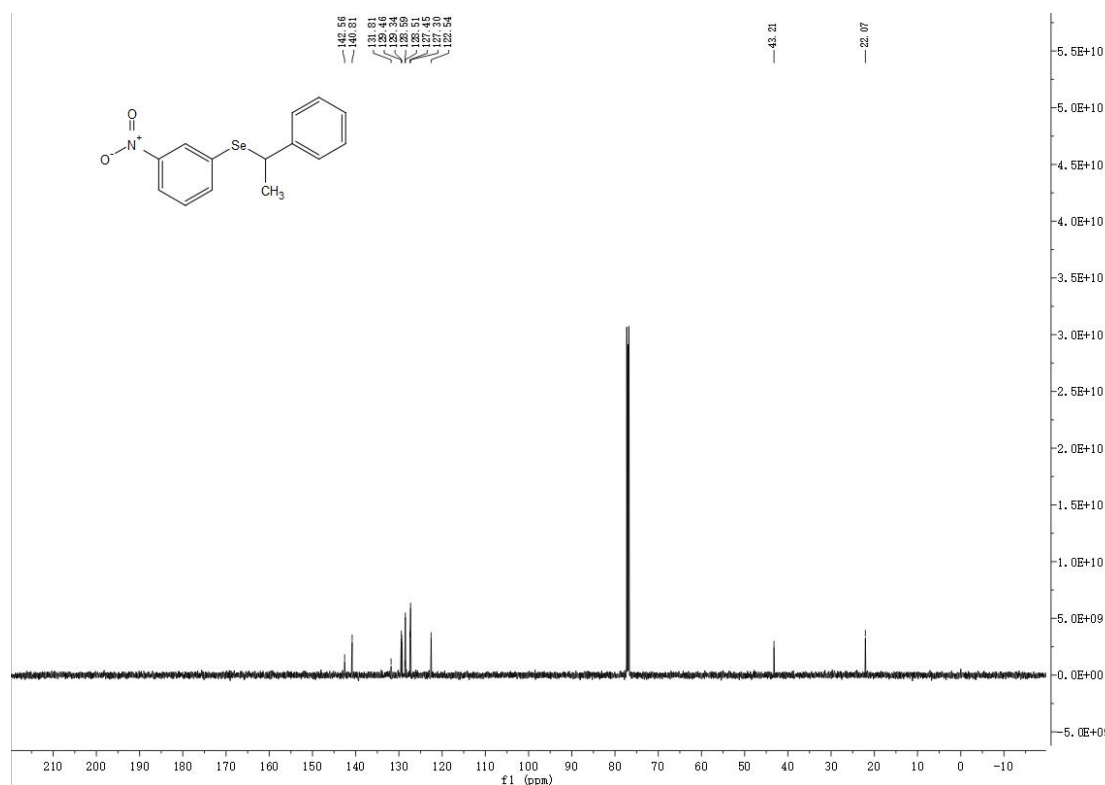
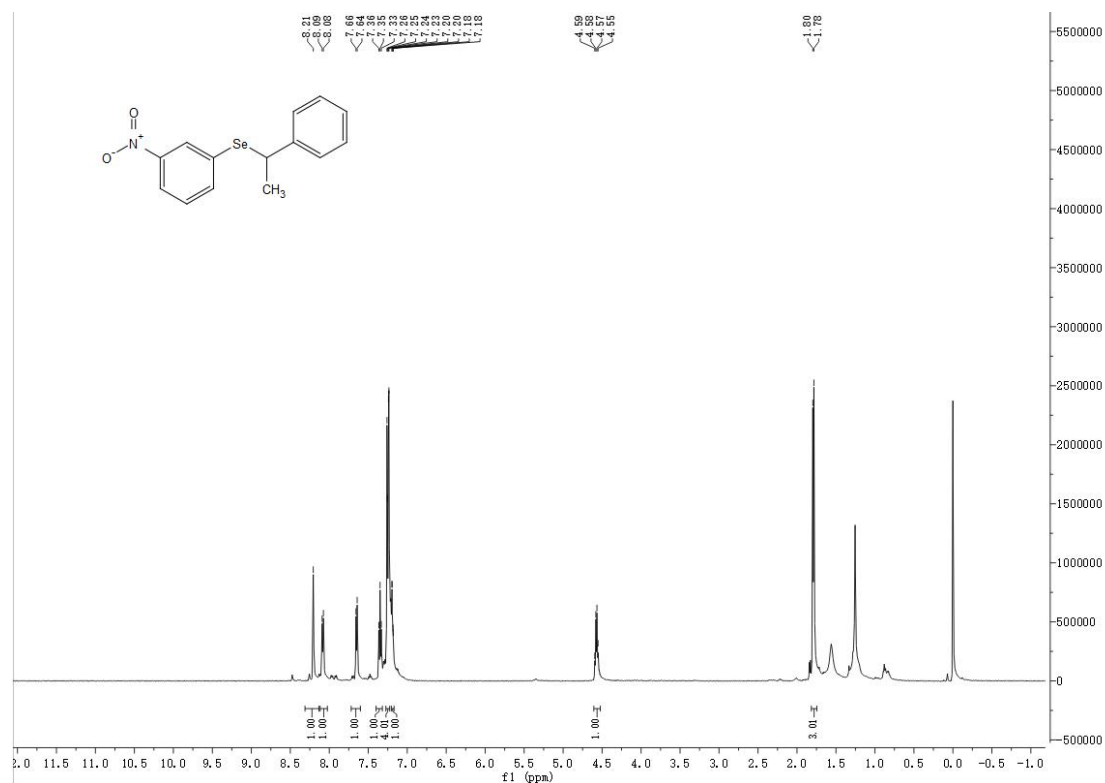
**Compound (29)**



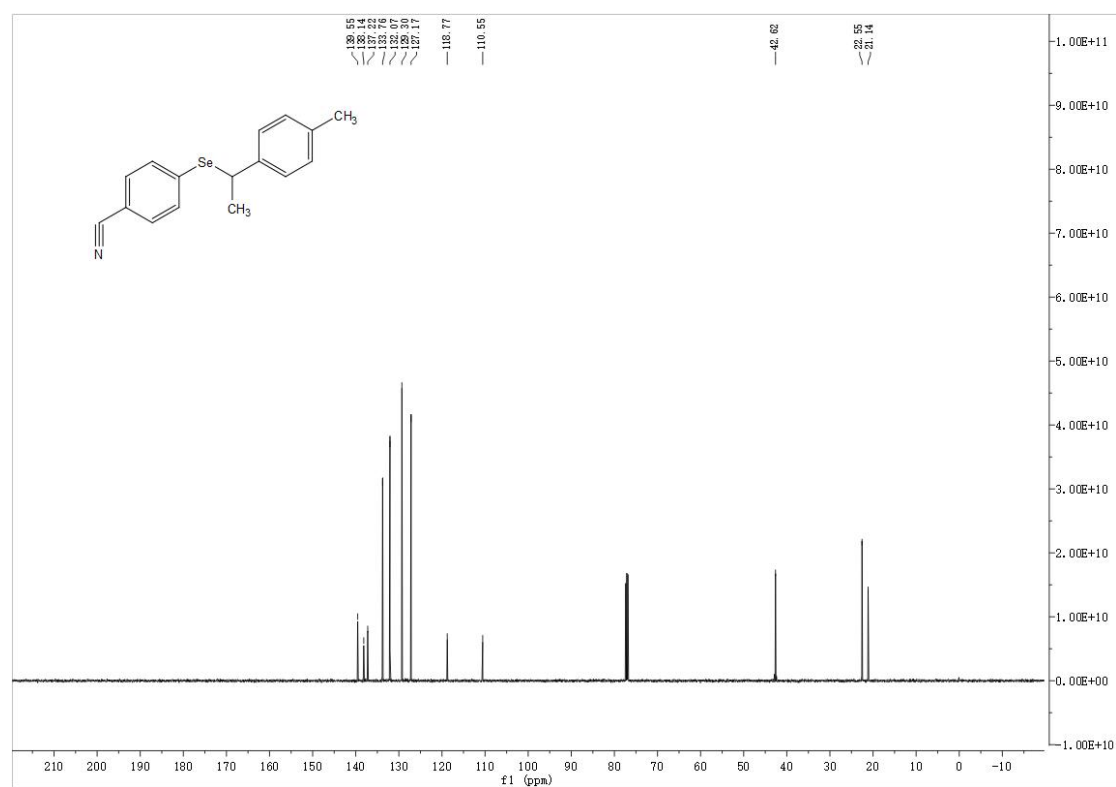
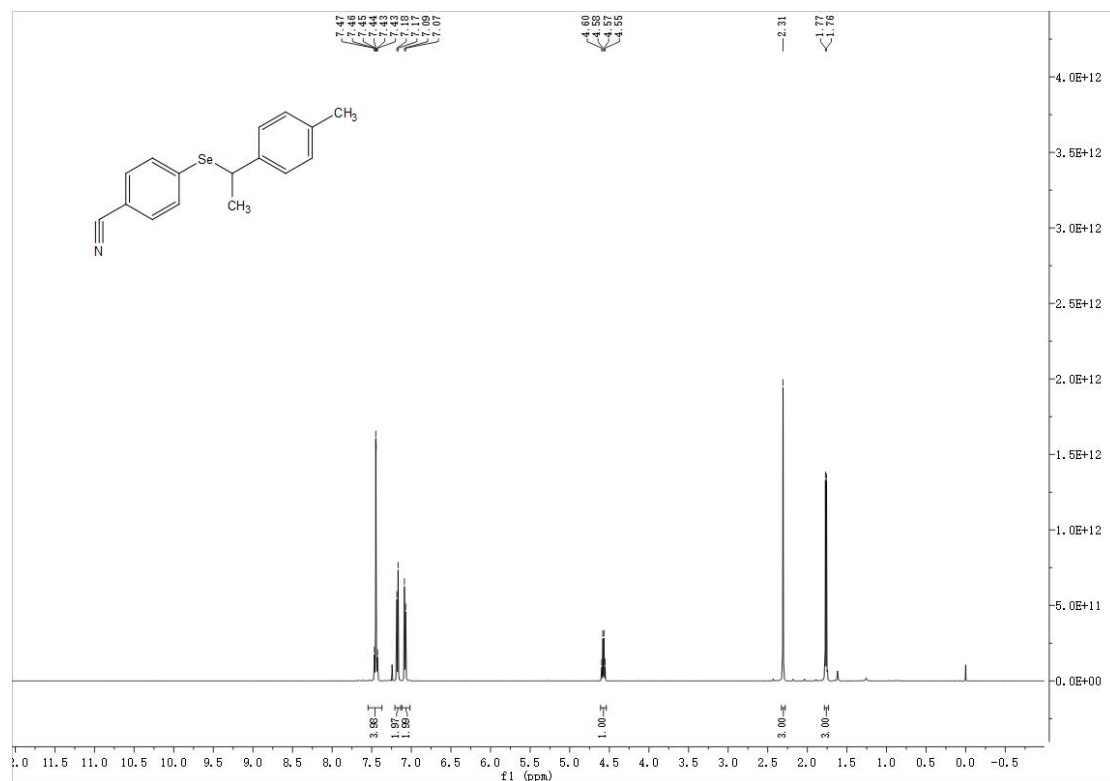




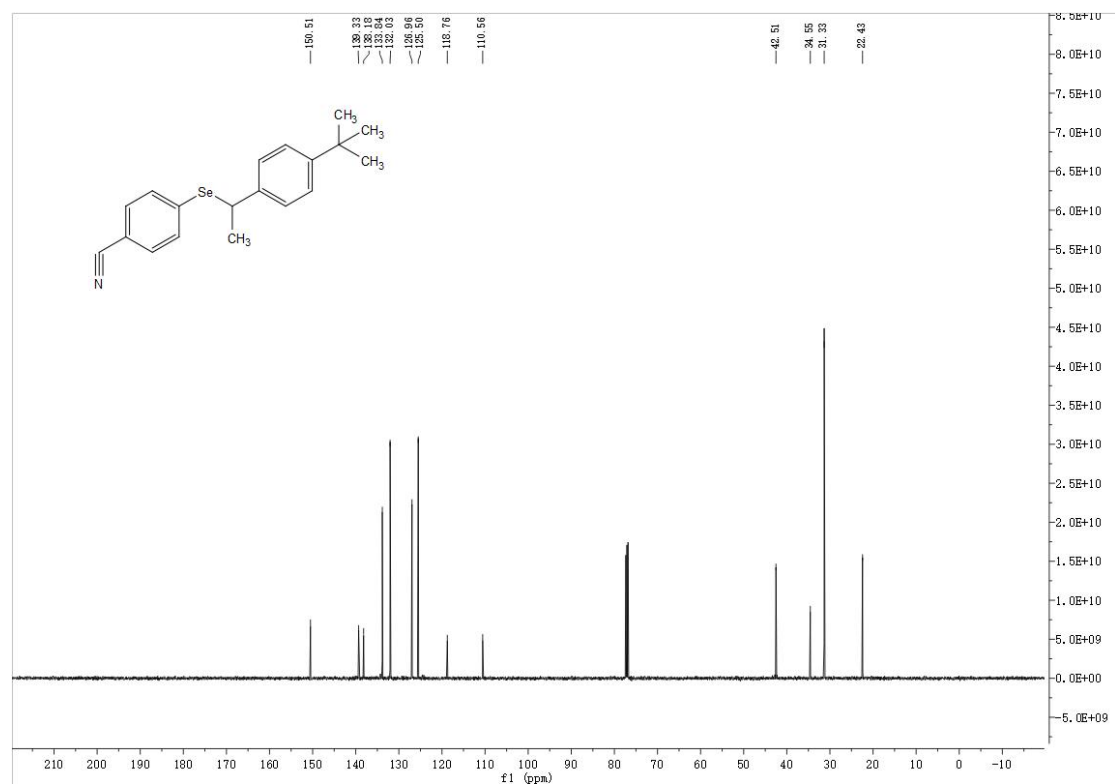
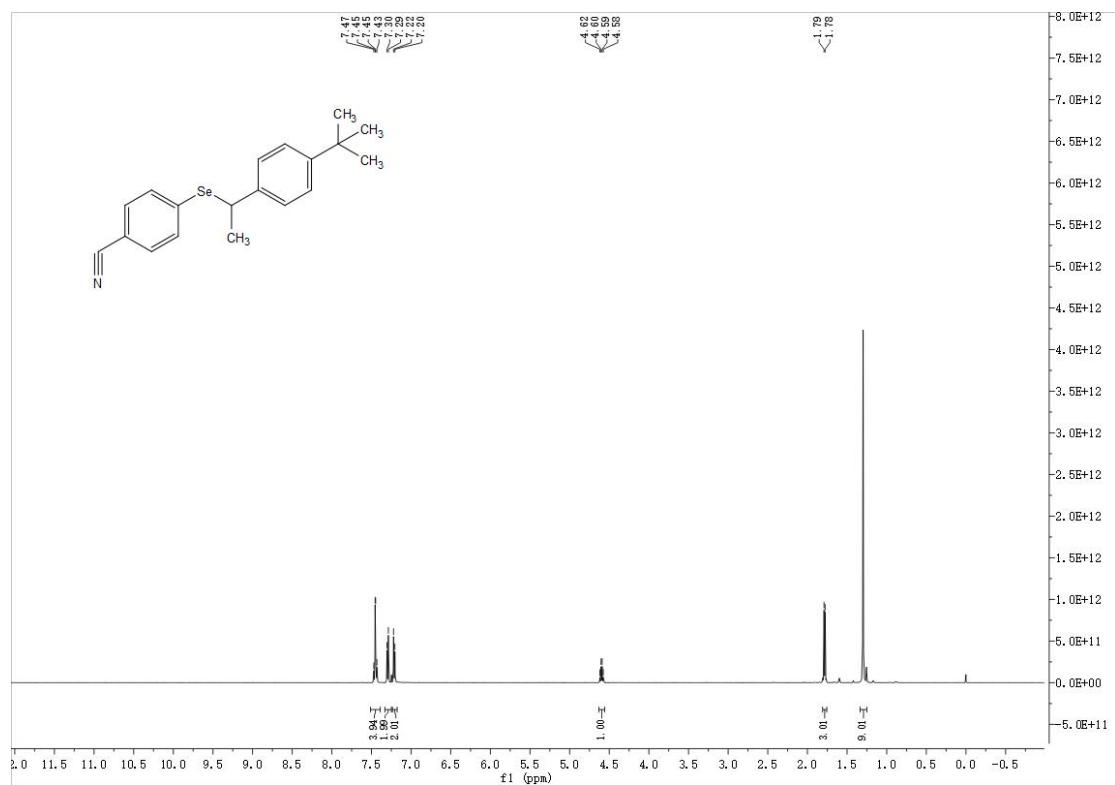
### Compound (31)



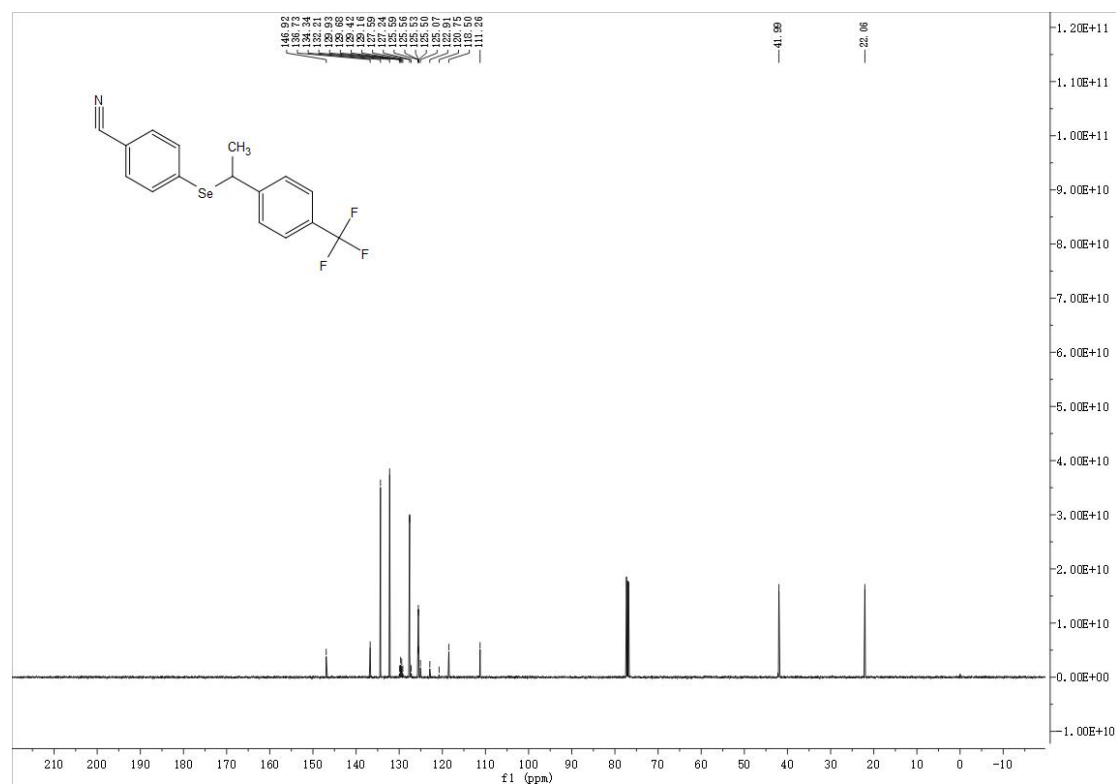
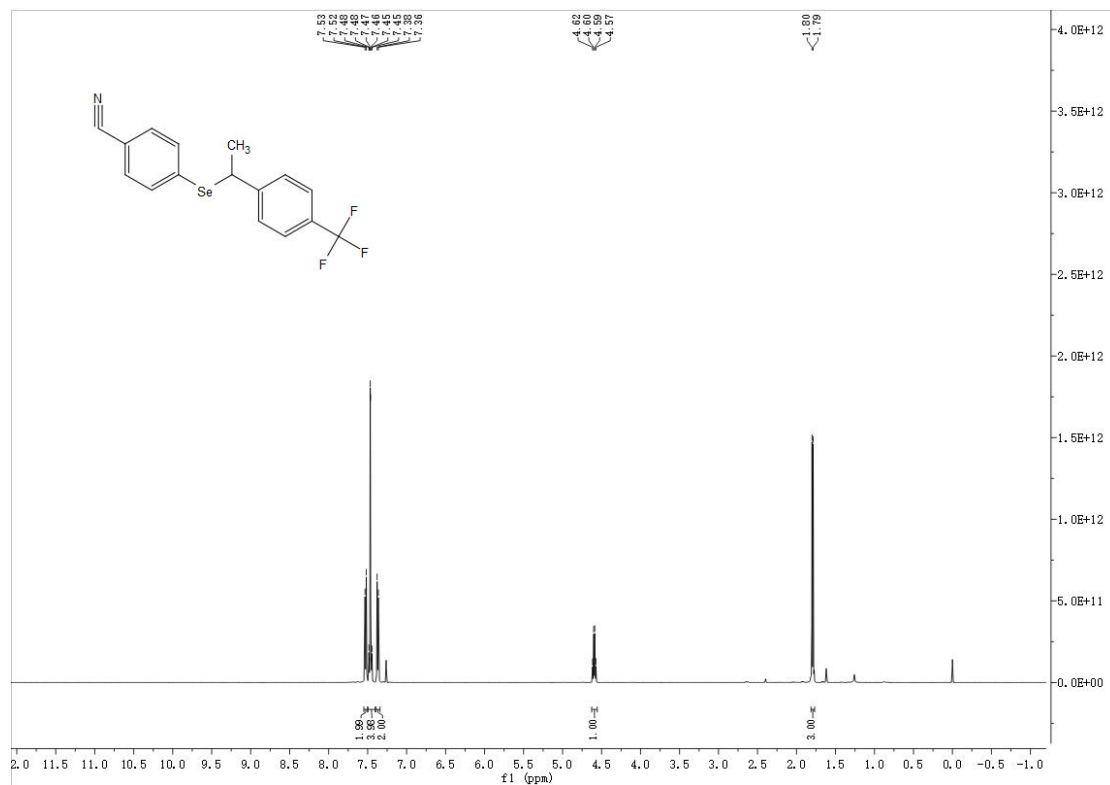
**Compound (32)**

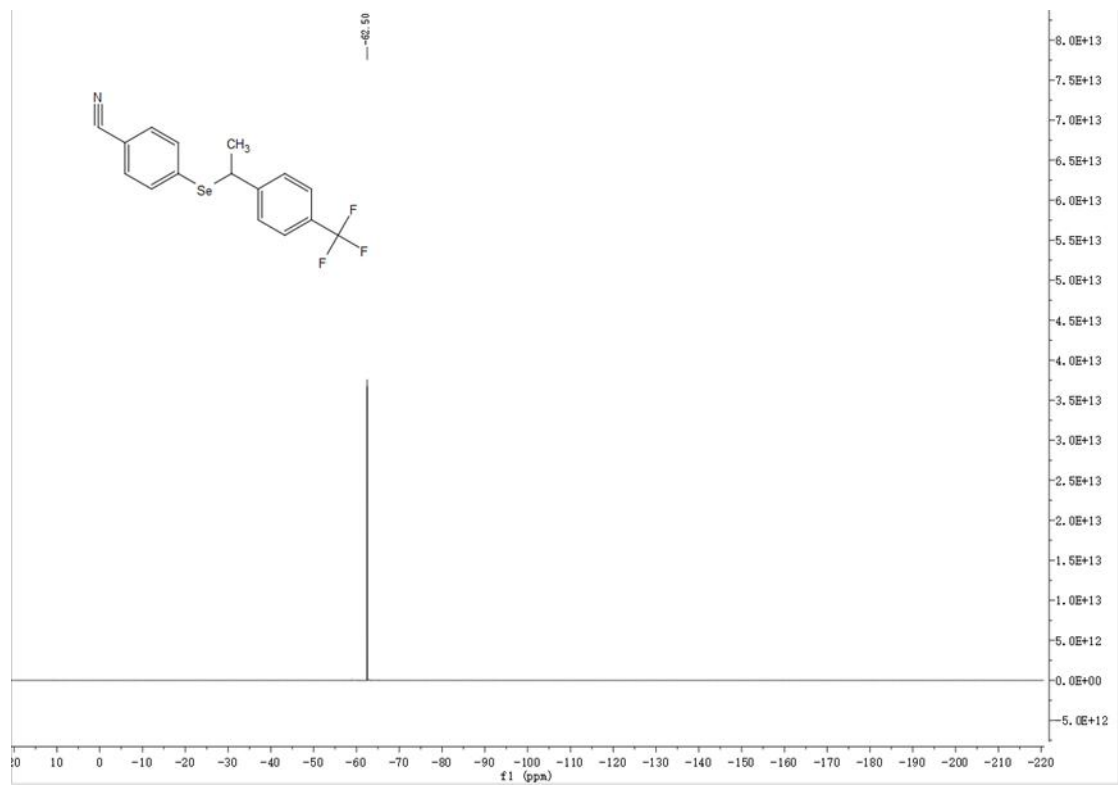


### Compound (33)



### Compound (34)

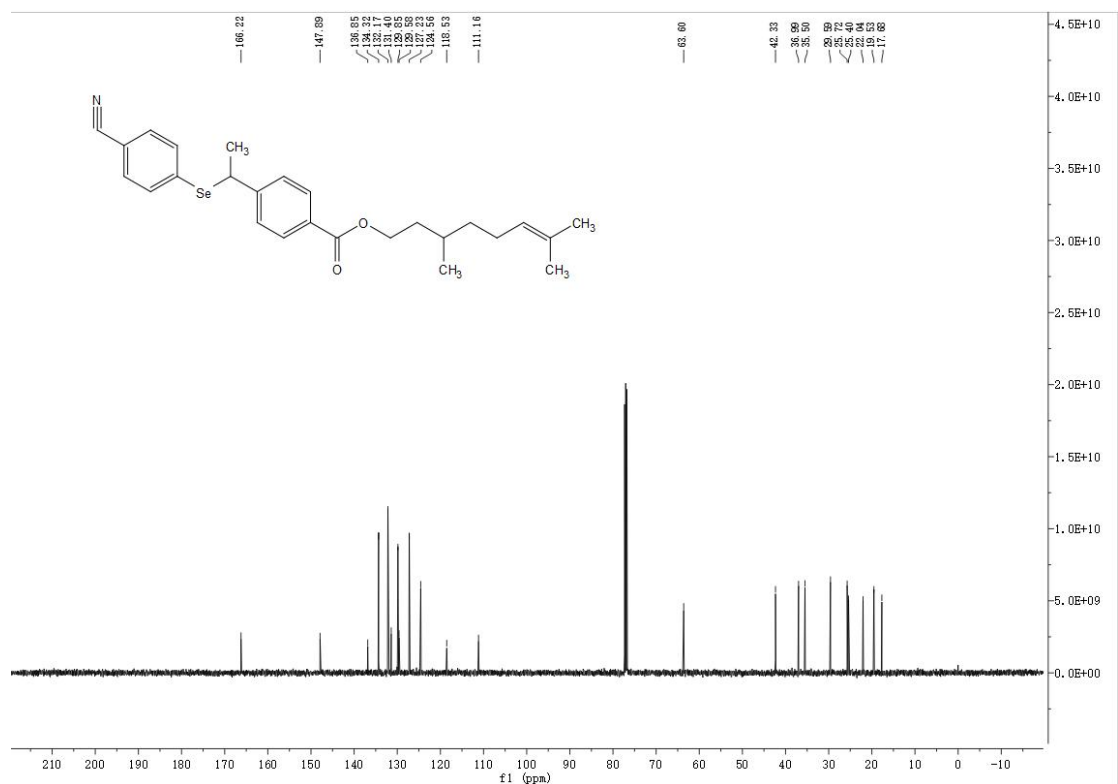
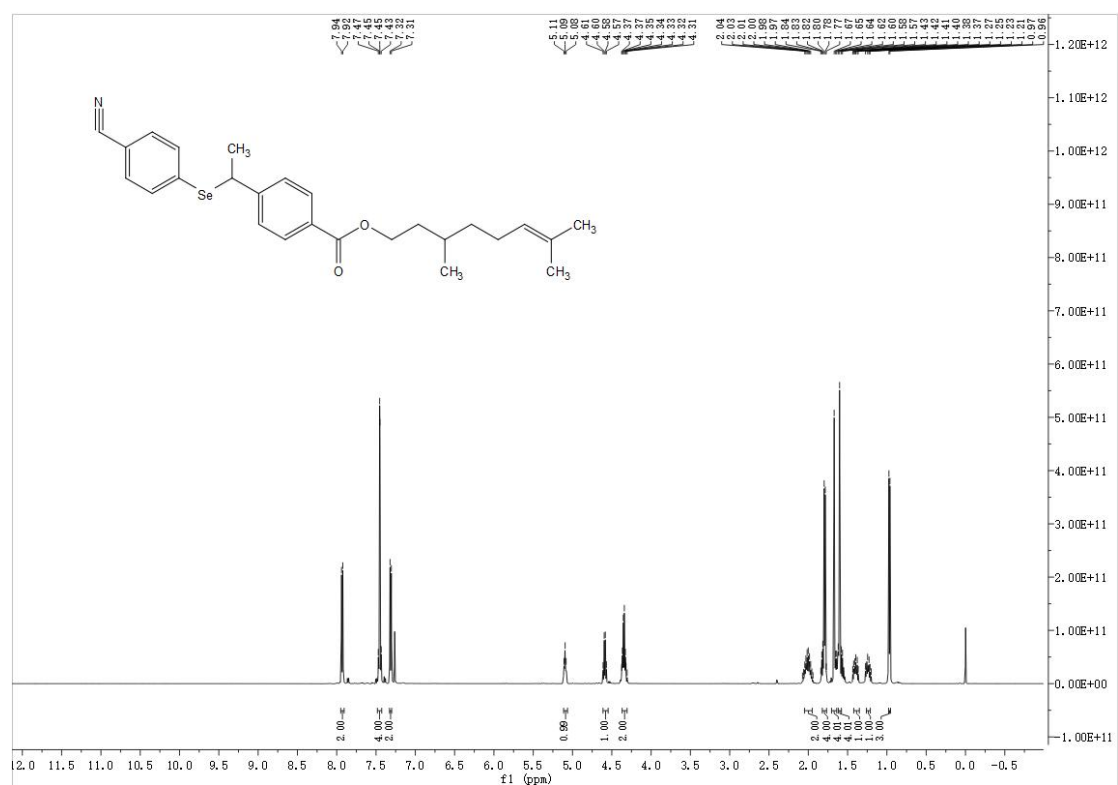




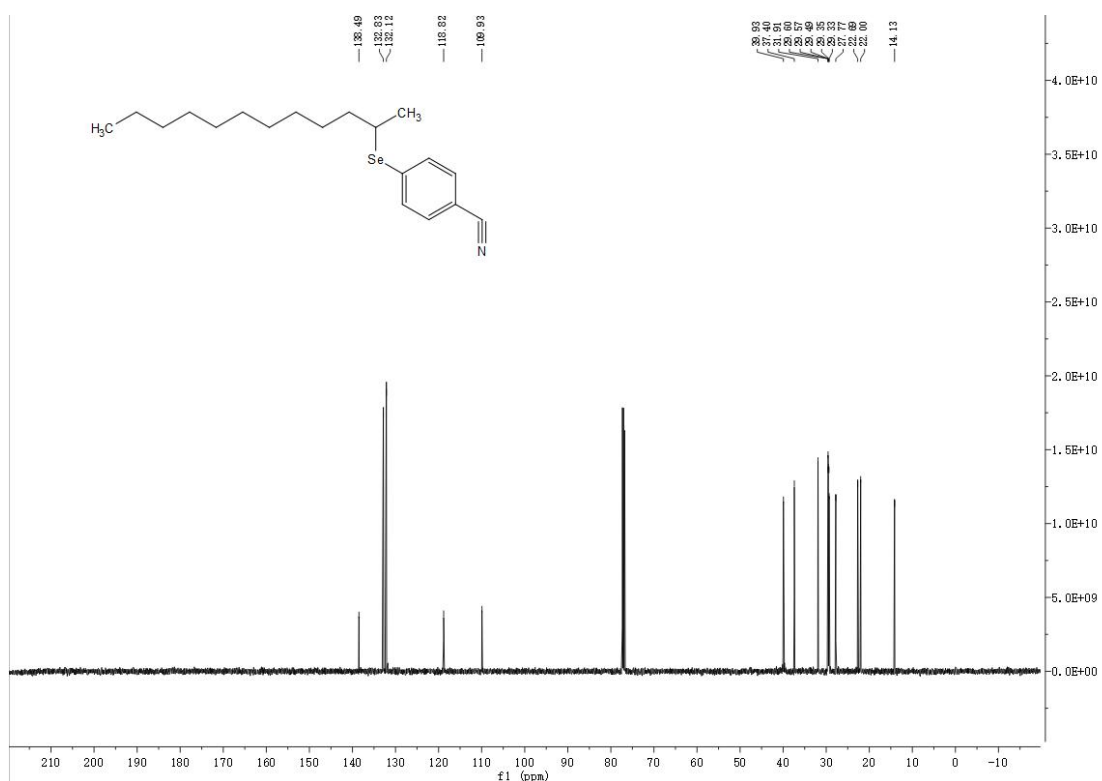
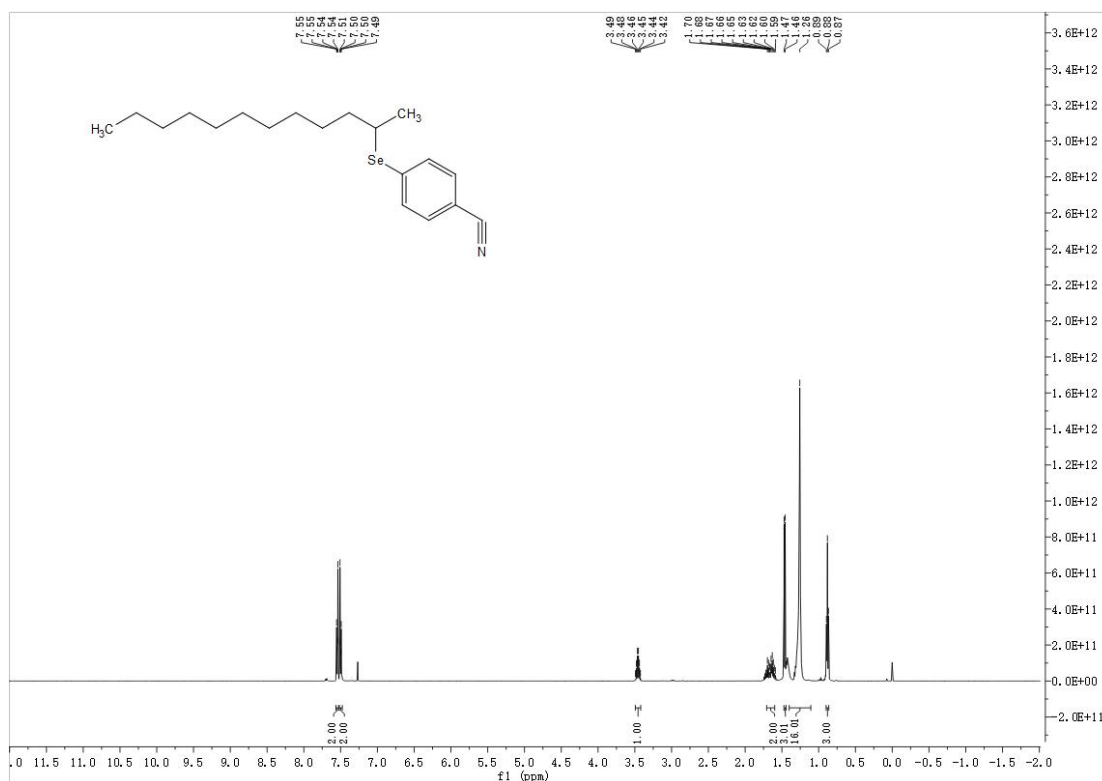




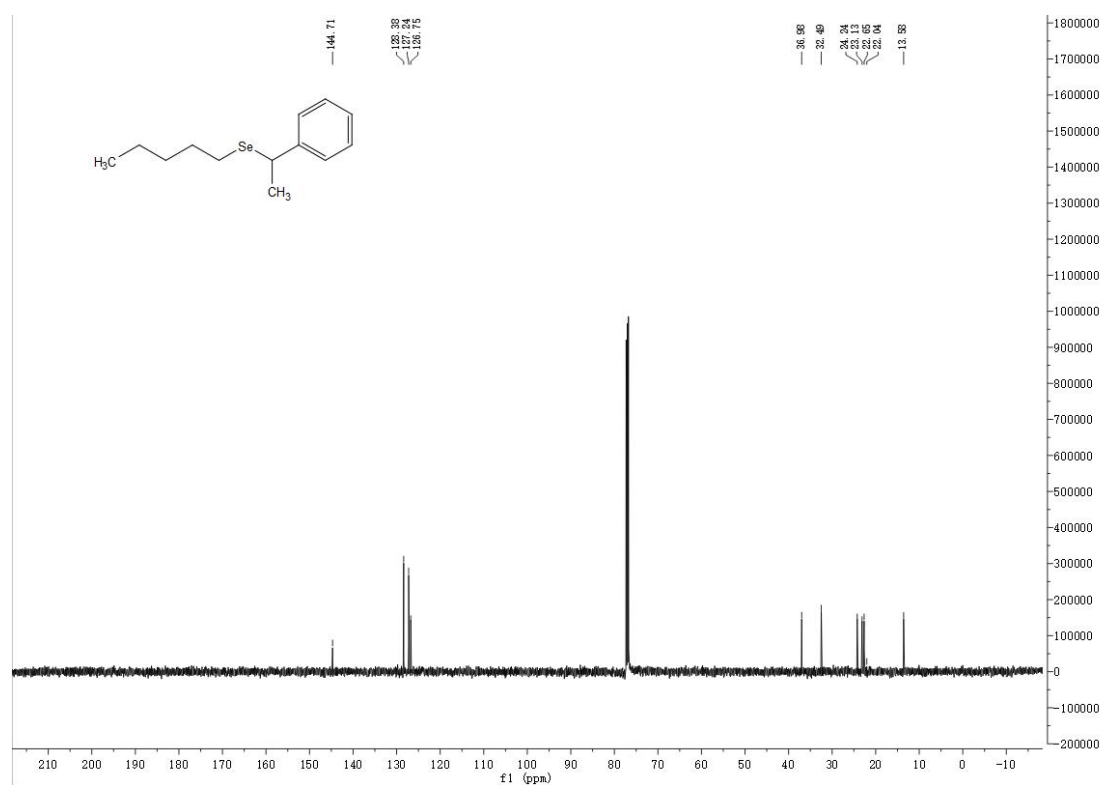
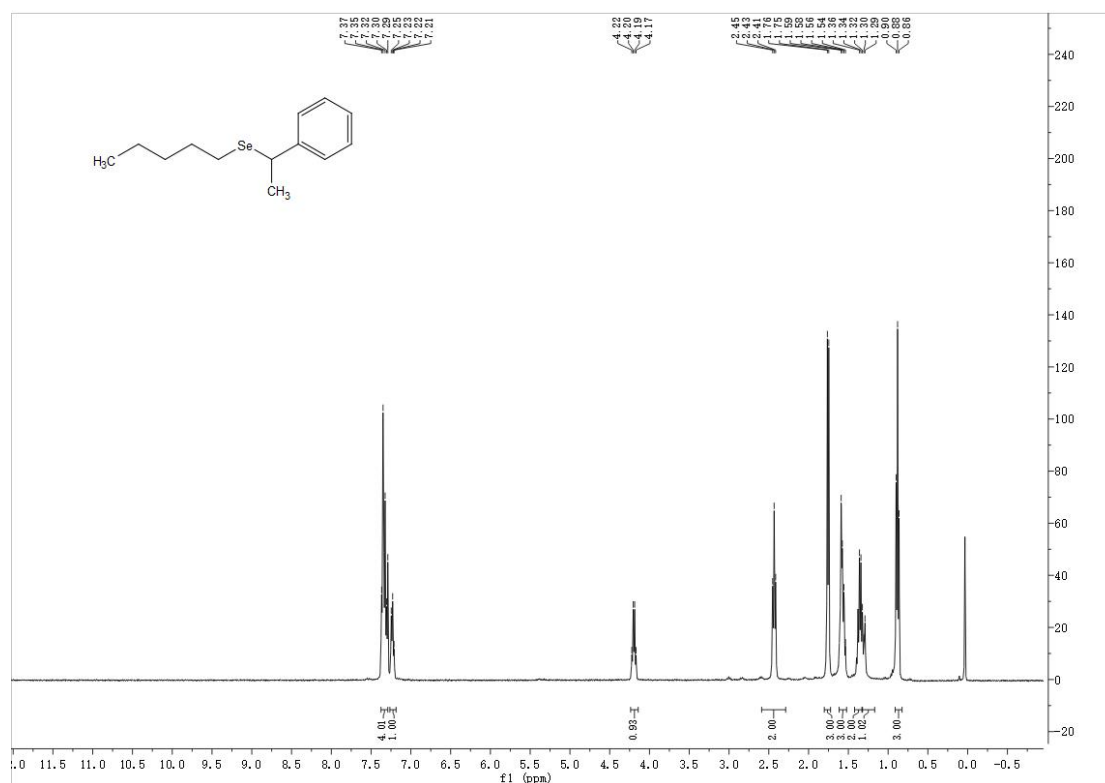
**Compound (36)**



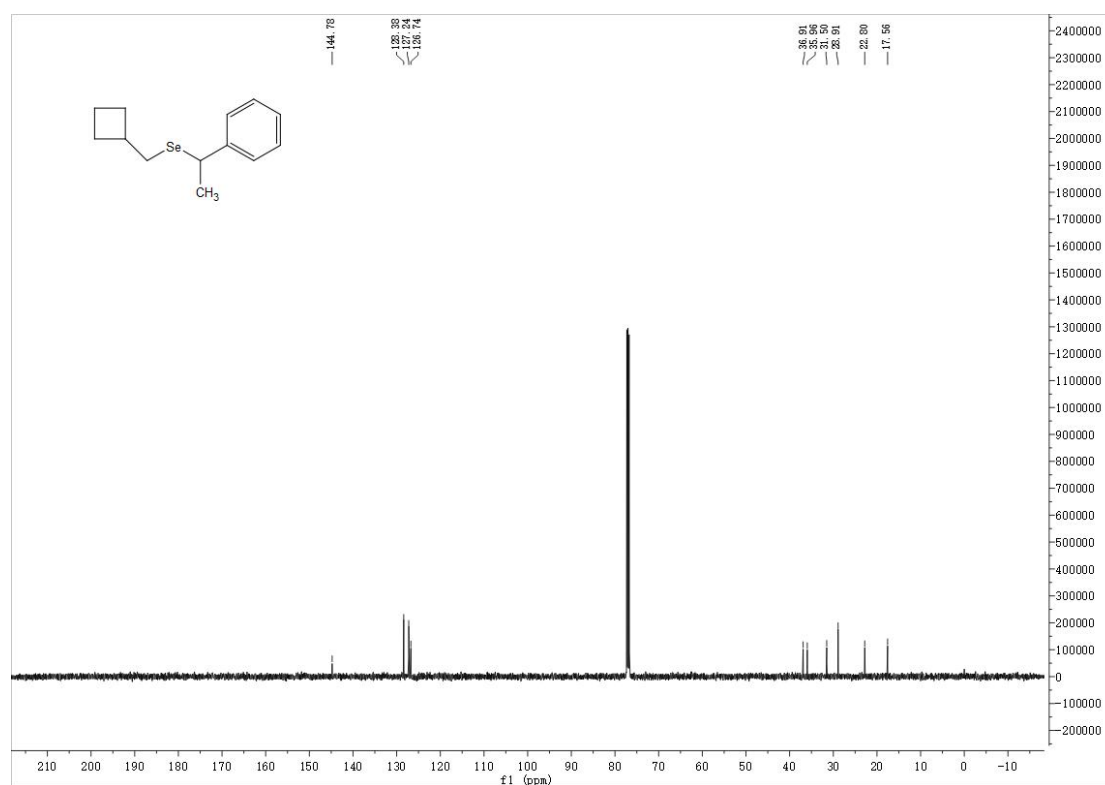
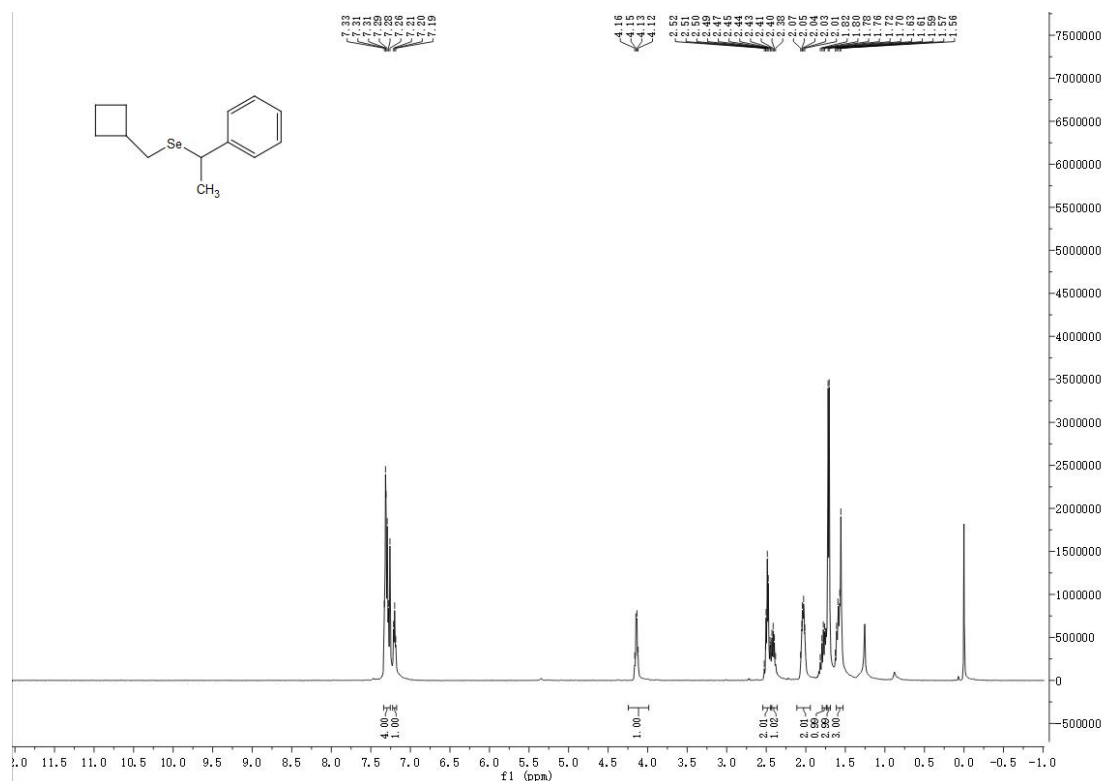
**Compound (37)**



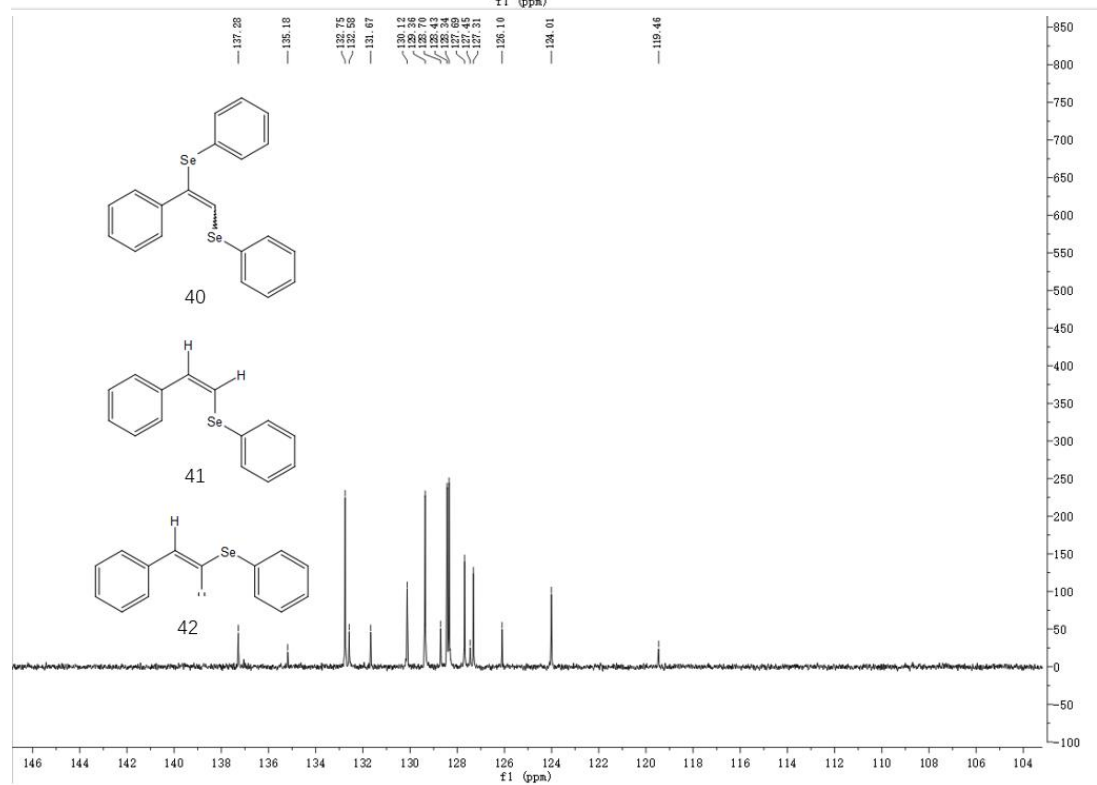
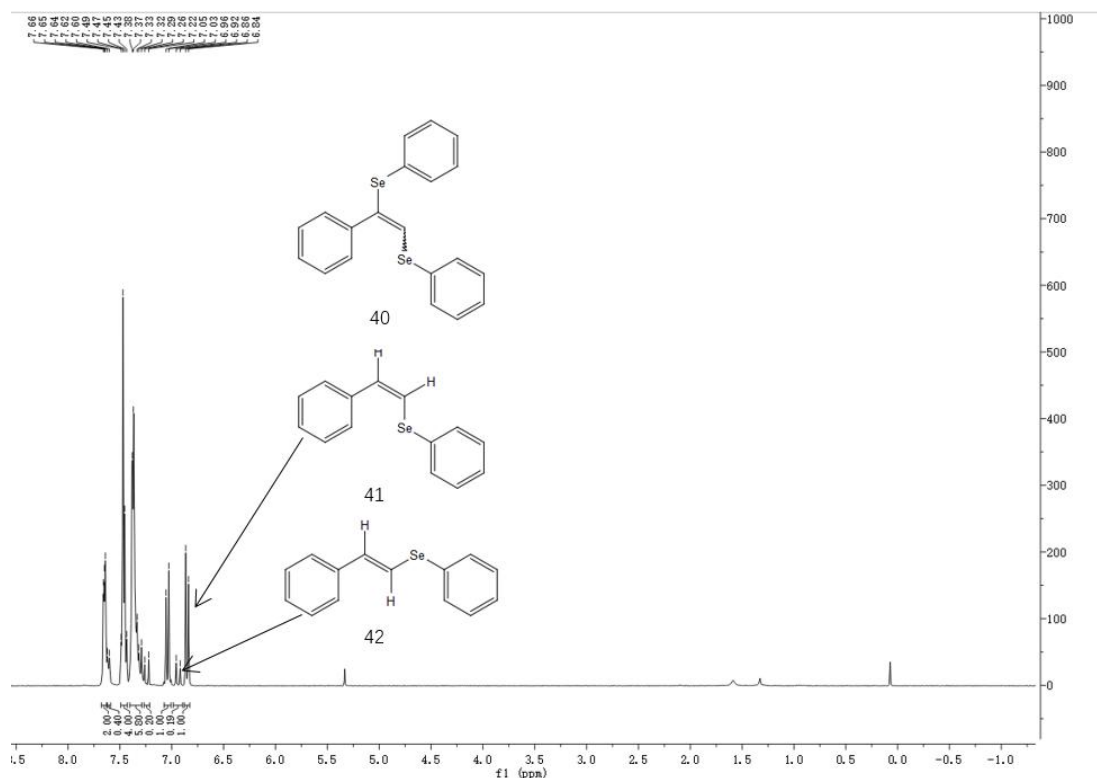
**Compound (38)**



# Compound (39)



**Compounds (40+41+42)**



## 8. References

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