

## ***Supporting Information***

### **Fe<sup>3+</sup>-mediated selective 1,2-Se/S bifunctionalization of olefin**

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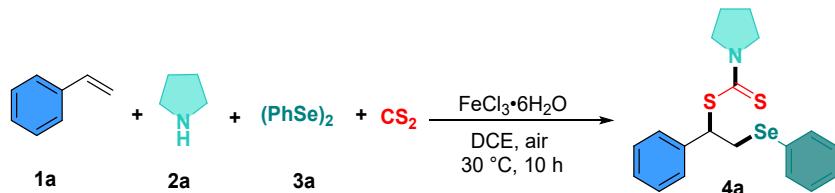
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## 1 General Information

### 1.1 Instruments and reagents

Without special instructions, all major chemicals and solvents were obtained from commercial sources and used without further purification.  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded on a Bruker Avance III-500 spectrometer (Swiss Bruker, Switzerland). Chemical shifts ( $\delta$ ) are reported in ppm, using TMS ( $\delta=0$ ) as an internal standard in  $\text{CDCl}_3$ . The HRMS spectrum was measured by micromass QTOF<sub>2</sub> Quadrupole/Time of Flight Tandem mass spectrometer with electron spray ionization. UV-visible spectroscopy of reaction solution was recorded on a UV-5500PC UV-visible spectrophotometer.

### 1.2 General Method for the Synthesis



The model substrates styrene (**1a**), pyrrolidine (**2a**), diphenyl diselenide (**3a**), Iron (III) chloride hexahydrate ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ), and  $\text{CS}_2$  were added to a dry 10 mL test tube, followed by dichloroethane (DCE) (0.5 mL). Subsequently, the tube was maintained at air atmosphere of 30 °C and agitated for a period of 10 h. The reaction was monitored by thin-layer chromatography (TLC) using a petroleum ether/ethyl acetate solvent system with a ratio of 10:1 (vol/vol). Once the reaction had reached completion, the solvent was concentrated under reduced pressure. The desired product was obtained through purification by silica gel column chromatography, employing the same ratio of petroleum ether to ethyl acetate.

## 2 Additional Optimization of Reaction Conditions

**Table S1** Optimization of the reaction conditions <sup>a</sup>

Entry	Cat.	Solvent	Time (h)	T (°C)	Yield <sup>b</sup> (%)
1	----	DCE (2.0 mL)	24	25	N.D.
2	FeCl <sub>3</sub> ·6H <sub>2</sub> O (0.6 eq.)	DCE (2.0 mL)	12	25	Trace
3	FeCl <sub>3</sub> ·6H <sub>2</sub> O (0.8 eq.)	DCE (2.0 mL)	12	25	50
4	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	ACN (2.0 mL)	12	25	Trace
5	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	THF (2.0 mL)	12	25	Trace
6	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DMF (2.0 mL)	12	25	Trace
7	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (2.0 mL)	12	25	65
8	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.5 eq.)	DCE (2.0 mL)	12	25	68
9	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.1 mL)	6	25	34
10	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.25 mL)	6	25	64
11	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	6	25	81
12	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (1.0 mL)	6	25	77
13	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	6	0	23
14	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	6	22	67
15	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	6	26	81
16	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	6	28	80
17	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	6	30	82
18	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	6	35	74
19	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	6	45	70
20	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	6	60	57
21	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	8	30	88
<b>22</b>	<b>FeCl<sub>3</sub>·6H<sub>2</sub>O (1.0 eq.)</b>	<b>DCE (0.5 mL)</b>	<b>10</b>	<b>30</b>	<b>92</b>
23	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	12	30	84
24	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	14	30	84
25	FeCl <sub>3</sub> ·6H <sub>2</sub> O (1.0 eq.)	DCE (0.5 mL)	18	30	82

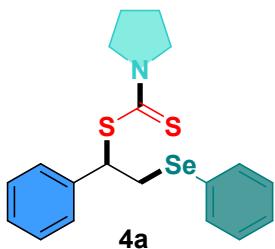
<sup>a</sup>Reaction conditions: **1a** (0.2 mmol), **2a** (0.25 mmol), **3a** (0.05 mmol), CS<sub>2</sub> (0.25 mmol); <sup>b</sup> HPLC yield.

### 3 Characterization of compounds

#### **1-phenyl-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4a)<sup>1</sup>**

Yellow oil (92%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.51 (dd, *J* = 6.4, 2.9 Hz, 2H), 7.31 (dt, *J* = 20.6, 6.1 Hz, 5H), 7.24 – 7.19 (m, 3H), 5.39 (dd, *J* = 10.9, 5.0 Hz, 1H), 3.91 – 3.88 (m, 2H), 3.86 (d, *J* = 5.1 Hz, 1H), 3.58 (dd, *J* = 12.4, 5.9 Hz, 1H), 3.53 – 3.45 (m, 2H), 2.01 (dd, *J* = 11.2, 6.6 Hz, 2H), 1.97 – 1.91 (m, 2H).  
**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.74, 138.86, 133.53, 129.97, 128.92, 128.68, 128.48, 128.01, 127.09, 54.98, 54.90, 50.58, 33.46, 26.08, 24.25.

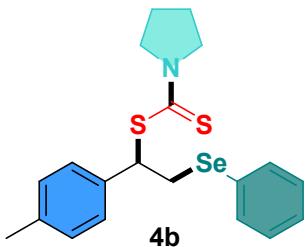


#### **2-(phenylselanyl)-1-(p-tolyl)ethyl pyrrolidine-1-carbodithioate (4b)**

Yellow oil (78%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.55 – 7.49 (m, 2H), 7.25 – 7.20 (m, 5H), 7.12 (d, *J* = 7.9 Hz, 2H), 5.34 (dd, *J* = 11.1, 4.9 Hz, 1H), 3.92 – 3.85 (m, 3H), 3.57 (dt, *J* = 13.6, 7.0 Hz, 1H), 3.52 – 3.45 (m, 2H), 2.33 (s, 3H), 2.01 (dd, *J* = 10.8, 6.7 Hz, 2H), 1.96 – 1.89 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.91, 137.80, 135.71, 133.49, 130.04, 129.42, 128.89, 128.33, 127.02, 54.84, 54.68, 50.56, 33.45, 26.09, 24.25, 21.27.

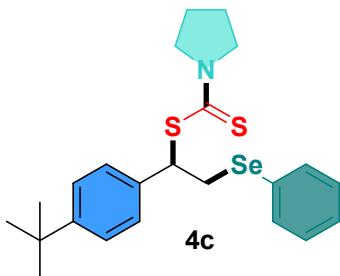


#### **1-(4-(tert-butyl)phenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4c)**

Yellow oil (74%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.48 (dd, *J* = 6.5, 3.0 Hz, 2H), 7.32 – 7.24 (m, 4H), 7.24 – 7.13 (m, 3H), 5.39 (dd, *J* = 10.8, 4.9 Hz, 1H), 3.94 – 3.83 (m, 3H), 3.58 (dt, *J* = 13.4, 6.9 Hz, 1H), 3.55 – 3.45 (m, 2H), 2.01 (dt, *J* = 12.4, 6.5 Hz, 2H), 1.94 (dt, *J* = 10.2, 4.3 Hz, 2H), 1.30 (s, 9H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.99, 150.78, 135.58, 133.53, 130.21, 128.85, 128.07, 127.01, 125.59, 54.88, 54.86, 50.57, 34.59, 33.74, 31.36, 26.08, 24.25.

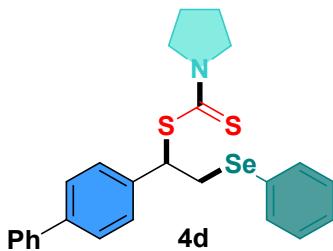


**1-([1,1'-biphenyl]-4-yl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4d)**

Yellow oil (84%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.56 (d, *J* = 7.4 Hz, 2H), 7.51 (d, *J* = 8.2 Hz, 4H), 7.45 – 7.38 (m, 4H), 7.34 (t, *J* = 7.3 Hz, 1H), 7.25 – 7.18 (m, 3H), 5.45 (dd, *J* = 10.9, 4.9 Hz, 1H), 3.90 (dt, *J* = 8.7, 4.9 Hz, 3H), 3.58 (dd, *J* = 12.4, 5.9 Hz, 1H), 3.56 – 3.45 (m, 2H), 2.00 (dt, *J* = 12.6, 6.6 Hz, 2H), 1.97 – 1.90 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.68, 140.79, 140.65, 137.88, 133.64, 129.97, 128.93, 128.80, 127.40, 127.36, 127.12, 54.94, 54.87, 50.62, 33.49, 26.10, 24.27.

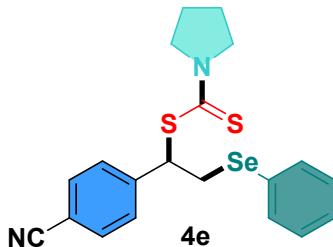


**1-(4-cyanophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4e)**

Yellow oil (82%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.54 (d, *J* = 8.2 Hz, 2H), 7.43 (t, *J* = 8.8 Hz, 4H), 7.26 – 7.18 (m, 3H), 5.46 (dd, *J* = 10.7, 5.2 Hz, 1H), 3.87 (q, *J* = 6.5 Hz, 2H), 3.77 (dd, *J* = 12.4, 5.2 Hz, 1H), 3.59 (dt, *J* = 13.6, 6.9 Hz, 1H), 3.55 – 3.49 (m, 1H), 3.40 (dd, *J* = 12.3, 10.8 Hz, 1H), 2.07 – 2.00 (m, 2H), 1.95 (td, *J* = 6.8, 3.6 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 189.48, 144.94, 133.70, 132.22, 129.37, 129.07, 127.47, 118.68, 111.48, 55.14, 54.63, 50.66, 32.69, 26.07, 24.22.

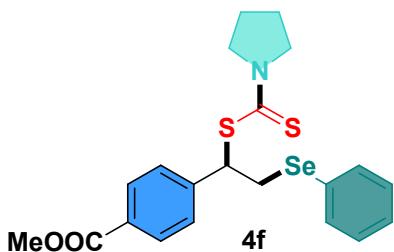


**Methyl-4-(2-(phenylselanyl)-1-((pyrrolidine-1-carbonothioyl)thio)ethyl)benzoate (4f)**

Yellow oil (80%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.48 (dd, *J* = 7.5, 1.9 Hz, 2H), 7.40 (d, *J* = 8.3 Hz, 2H), 7.26 – 7.18 (m, 3H), 5.45 (dd, *J* = 10.8, 5.1 Hz, 1H), 3.91 (s, 3H), 3.88 (dd, *J* = 11.2, 6.2 Hz, 2H), 3.83 (dd, *J* = 12.2, 5.1 Hz, 1H), 3.59 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.51 (dt, *J* = 11.7, 6.8 Hz, 1H), 3.48 – 3.41 (m, 1H), 2.02 (dt, *J* = 12.8, 6.6 Hz, 2H), 1.98 – 1.91 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.07, 166.72, 144.35, 133.65, 129.88, 129.55, 128.98, 128.57, 127.29, 55.02, 54.65, 52.18, 50.61, 32.96, 26.07, 24.23.

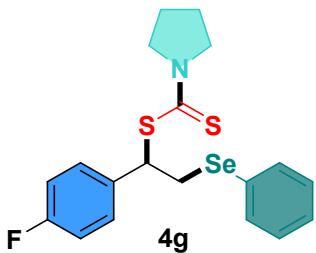


**1-(4-fluorophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4g)**

Yellow oil (78%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.48 (dd, *J* = 6.4, 3.1 Hz, 2H), 7.29 (dd, *J* = 8.6, 5.3 Hz, 2H), 7.21 (dd, *J* = 5.1, 1.8 Hz, 3H), 6.97 (t, *J* = 8.6 Hz, 2H), 5.37 (dd, *J* = 10.9, 5.0 Hz, 1H), 3.92 – 3.80 (m, 3H), 3.58 (dt, *J* = 13.6, 6.9 Hz, 1H), 3.54 – 3.48 (m, 1H), 3.45 – 3.39 (m, 1H), 2.05 – 1.97 (m, 2H), 1.94 (dq, *J* = 7.1, 4.9, 3.2 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.41, 163.23, 133.60, 130.17, 130.11, 129.74, 128.95, 127.20, 115.59, 115.42, 54.93, 54.30, 50.60, 33.58, 26.07, 24.24.

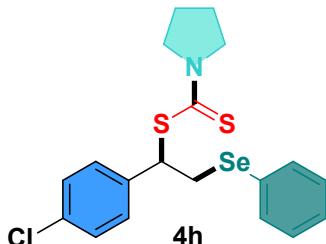


**1-(4-chlorophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4h)**

Yellow oil (94%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.48 (dd, *J* = 7.4, 2.0 Hz, 2H), 7.25 (d, *J* = 3.6 Hz, 4H), 7.24 – 7.14 (m, 3H), 5.36 (dd, *J* = 10.9, 5.0 Hz, 1H), 3.85 (ddd, *J* = 28.6, 11.1, 6.0 Hz, 3H), 3.53 (ddt, *J* = 25.1, 11.5, 5.9 Hz, 2H), 3.44 – 3.36 (m, 1H), 2.04 – 1.99 (m, 2H), 1.95 (q, *J* = 6.6 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.24, 137.62, 133.64, 129.86, 129.64, 128.97, 128.74, 127.24, 54.97, 54.36, 50.61, 33.29, 26.07, 24.24.

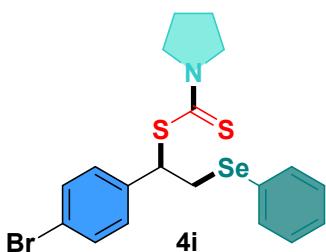


**1-(4-bromophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4i)**

Yellow oil (85%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.47 (d, *J* = 7.7 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.20 (dd, *J* = 12.4, 7.6 Hz, 5H), 5.35 (dd, *J* = 10.9, 5.0 Hz, 1H), 3.92 – 3.84 (m, 2H), 3.81 (dd, *J* = 12.2, 5.0 Hz, 1H), 3.54 (ddt, *J* = 35.3, 11.5, 6.8 Hz, 2H), 3.43 – 3.35 (m, 1H), 2.05 – 1.98 (m, 2H), 1.94 (q, *J* = 7.5 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.19, 138.15, 133.66, 131.68, 130.20, 129.61, 128.98, 127.25, 121.86, 54.98, 54.42, 50.62, 33.22, 26.08, 24.25.

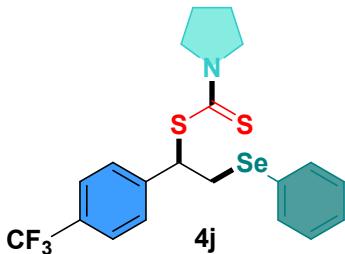


**2-(phenylselanyl)-1-(4-(trifluoromethyl)phenyl)ethyl pyrrolidine-1-carbodithioate (4j)**

Yellow oil (90%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.51 (d, *J* = 8.2 Hz, 2H), 7.43 (t, *J* = 7.7 Hz, 4H), 7.25 – 7.15 (m, 3H), 5.47 (dd, *J* = 10.8, 5.1 Hz, 1H), 3.88 (q, *J* = 6.7 Hz, 2H), 3.81 (dd, *J* = 12.3, 5.1 Hz, 1H), 3.61 – 3.49 (m, 2H), 3.47 – 3.39 (m, 1H), 2.03 (dt, *J* = 12.8, 6.6 Hz, 2H), 1.98 – 1.91 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 189.89, 143.28, 133.73, 129.45, 128.99, 128.93, 127.35, 125.47, 125.44, 55.06, 54.65, 50.63, 33.08, 26.07, 24.23.

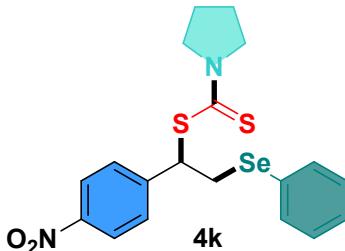


**1-(4-nitrophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4k)**

Yellow oil (78%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 8.10 (d, *J* = 8.7 Hz, 2H), 7.46 (dd, *J* = 15.6, 7.6 Hz, 4H), 7.26 – 7.15 (m, 3H), 5.51 (dd, *J* = 10.7, 5.2 Hz, 1H), 3.88 (q, *J* = 6.5 Hz, 2H), 3.79 (dd, *J* = 12.4, 5.2 Hz, 1H), 3.56 (ddt, *J* = 34.1, 11.6, 6.8 Hz, 2H), 3.46 – 3.37 (m, 1H), 2.04 (dt, *J* = 12.9, 6.6 Hz, 2H), 1.99 – 1.91 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 189.32, 147.21, 147.08, 133.73, 129.51, 129.15, 129.08, 127.52, 123.65, 55.17, 54.32, 50.68, 32.66, 26.07, 24.23.

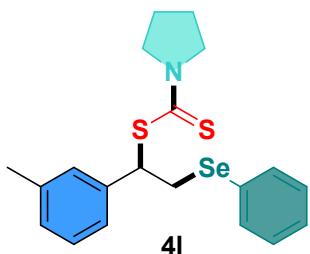


**2-(phenylselanyl)-1-(m-tolyl)ethyl pyrrolidine-1-carbodithioate (4l)**

Yellow oil (65%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.56 – 7.40 (m, 2H), 7.25 – 7.18 (m, 4H), 7.13 (d, *J* = 11.5 Hz, 2H), 7.08 (d, *J* = 7.3 Hz, 1H), 5.36 (dd, *J* = 11.0, 4.9 Hz, 1H), 3.96 (q, *J* = 6.6 Hz, 1H), 3.89 (t, *J* = 6.7 Hz, 2H), 3.61 – 3.55 (m, 1H), 3.56 – 3.45 (m, 2H), 2.31 (s, 3H), 2.04 – 1.98 (m, 2H), 1.97 – 1.88 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.88, 138.61, 138.35, 133.51, 131.50, 129.21, 129.18, 128.86, 128.84, 128.55, 127.04, 125.49, 55.03, 50.56, 33.44, 26.08, 24.25, 21.49.

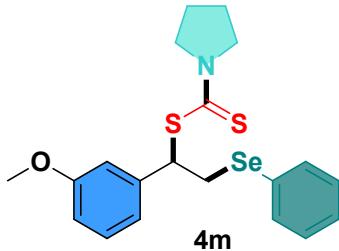


**1-(3-methoxyphenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4m)**

Yellow oil (92%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.52 (dd, *J* = 6.5, 3.0 Hz, 2H), 7.25 – 7.19 (m, 4H), 6.93 (d, *J* = 7.6 Hz, 1H), 6.86 (s, 1H), 6.81 (dd, *J* = 8.2, 2.4 Hz, 1H), 5.36 (dd, *J* = 11.0, 4.9 Hz, 1H), 3.91 – 3.84 (m, 3H), 3.77 (s, 3H), 3.61 – 3.55 (m, 1H), 3.54 – 3.44 (m, 2H), 2.05 – 1.99 (m, 2H), 1.97 – 1.91 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.76, 159.65, 140.32, 133.54, 130.03, 129.69, 128.89, 127.08, 120.70, 114.06, 113.59, 55.25, 55.03, 54.90, 50.58, 33.38, 26.08, 24.25.

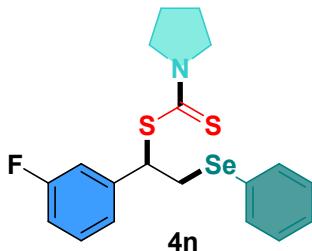


**1-(3-fluorophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4n)**

Yellow oil (90%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.50 (dd, *J* = 6.5, 3.0 Hz, 2H), 7.28 – 7.21 (m, 4H), 7.12 (d, *J* = 7.7 Hz, 1H), 7.03 (d, *J* = 9.7 Hz, 1H), 6.95 (td, *J* = 8.4, 2.3 Hz, 1H), 5.39 (dd, *J* = 10.8, 5.1 Hz, 1H), 3.88 (td, *J* = 6.9, 2.8 Hz, 2H), 3.81 (dd, *J* = 12.2, 5.1 Hz, 1H), 3.55 (ddt, *J* = 33.5, 11.8, 6.9 Hz, 2H), 3.47 – 3.36 (m, 1H), 2.05 – 1.99 (m, 2H), 1.98 – 1.91 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.20, 163.70, 141.63, 133.65, 130.12, 129.67, 128.97, 127.27, 124.26, 115.57, 114.97, 54.99, 54.59, 50.61, 33.23, 26.07, 24.24.

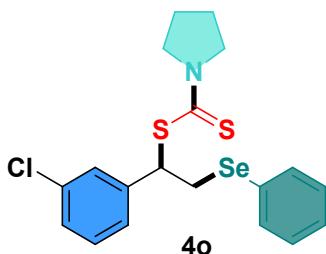


**1-(3-chlorophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4o)**

Yellow oil (91%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.53 – 7.45 (m, 2H), 7.30 (s, 1H), 7.27 – 7.18 (m, 6H), 5.37 (dd, *J* = 10.8, 5.1 Hz, 1H), 3.88 (td, *J* = 6.8, 3.2 Hz, 2H), 3.80 (dd, *J* = 12.2, 5.0 Hz, 1H), 3.54 (ddt, *J* = 25.4, 12.0, 6.0 Hz, 2H), 3.41 (t, *J* = 11.5 Hz, 1H), 2.06 – 1.99 (m, 2H), 1.98 – 1.90 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.12, 141.17, 134.38, 133.68, 129.80, 129.60, 128.98, 128.60, 128.08, 127.32, 126.81, 55.01, 54.59, 50.62, 33.18, 26.08, 24.24.

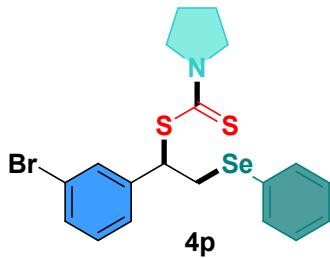


**1-(3-bromophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4p)**

Yellow oil (90%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.48 (dd, *J* = 6.3, 2.8 Hz, 2H), 7.45 (s, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.27 – 7.20 (m, 4H), 7.15 (t, *J* = 7.8 Hz, 1H), 5.36 (dd, *J* = 10.8, 5.1 Hz, 1H), 3.88 (td, *J* = 6.8, 3.2 Hz, 2H), 3.79 (dd, *J* = 12.2, 5.1 Hz, 1H), 3.54 (ddt, *J* = 25.3, 11.9, 6.0 Hz, 2H), 3.44 – 3.35 (m, 1H), 2.02 (q, *J* = 8.2, 6.5 Hz, 2H), 1.95 (q, *J* = 8.8, 7.8 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.09, 141.45, 133.69, 131.49, 131.00, 130.08, 129.58, 128.98, 127.33, 127.28, 122.62, 55.02, 54.57, 50.63, 33.19, 26.08, 24.25.

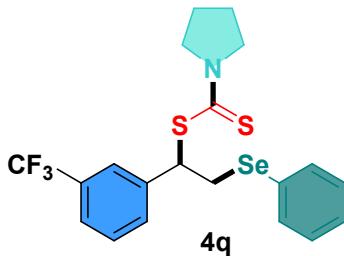


**2-(phenylselanyl)-1-(3-(trifluoromethyl)phenyl)ethyl pyrrolidine-1-carbodithioate (4q)**

Yellow oil (70%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.54 (s, 1H), 7.50 (t, *J* = 8.7 Hz, 2H), 7.45 (dd, *J* = 7.6, 1.8 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 1H), 7.27 – 7.18 (m, 3H), 5.45 (dd, *J* = 10.8, 5.0 Hz, 1H), 3.88 (q, *J* = 6.6 Hz, 2H), 3.83 (dd, *J* = 12.3, 5.0 Hz, 1H), 3.59 (dt, *J* = 13.6, 6.8 Hz, 1H), 3.52 (dt, *J* = 11.7, 6.8 Hz, 1H), 3.46 – 3.38 (m, 1H), 2.05 – 1.99 (m, 2H), 1.98 – 1.91 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 189.93, 140.24, 133.73, 132.07, 131.49, 129.37, 128.98, 127.35, 125.30, 124.66, 55.04, 54.71, 50.64, 33.17, 26.06, 24.23.

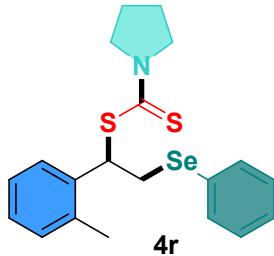


**2-(phenylselanyl)-1-(o-tolyl)ethyl pyrrolidine-1-carbodithioate (4r)**

Yellow oil (89%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.53 (dd, *J* = 6.4, 3.0 Hz, 2H), 7.27 (d, *J* = 7.0 Hz, 1H), 7.24 – 7.19 (m, 3H), 7.19 – 7.12 (m, 3H), 5.56 (dd, *J* = 11.4, 4.9 Hz, 1H), 3.89 (dt, *J* = 9.0, 5.5 Hz, 3H), 3.62 – 3.53 (m, 2H), 3.51 – 3.45 (m, 1H), 2.33 (s, 3H), 2.04 – 1.99 (m, 2H), 1.95 (q, *J* = 6.5 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 191.14, 137.34, 136.22, 133.54, 130.77, 129.95, 128.88, 127.87, 127.34, 127.08, 126.13, 54.90, 50.98, 50.55, 32.53, 26.09, 24.25, 19.88.

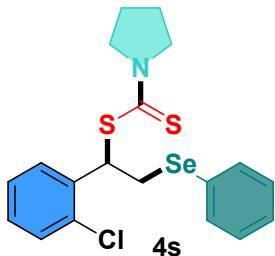


**1-(2-chlorophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4s)**

Yellow oil (80%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.55 – 7.48 (m, 2H), 7.38 – 7.33 (m, 2H), 7.24 – 7.13 (m, 5H), 5.85 (dd, *J* = 10.9, 5.1 Hz, 1H), 3.89 (t, *J* = 7.0 Hz, 2H), 3.85 (dd, *J* = 12.2, 5.1 Hz, 1H), 3.62 – 3.56 (m, 2H), 3.55 – 3.49 (m, 1H), 2.04 – 1.99 (m, 2H), 1.94 (q, *J* = 6.5 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.53, 136.21, 134.53, 133.79, 130.08, 129.91, 129.61, 129.05, 128.89, 127.21, 126.81, 55.02, 52.08, 50.55, 32.11, 26.10, 24.24.

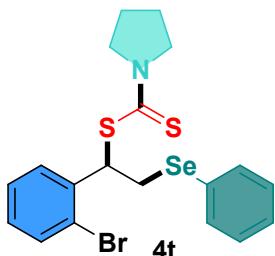


**1-(2-bromophenyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4t)**

Yellow oil (88%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.58 – 7.51 (m, 3H), 7.34 (d, *J* = 7.7 Hz, 1H), 7.26 – 7.19 (m, 4H), 7.14 – 7.09 (m, 1H), 5.82 (dd, *J* = 10.8, 5.1 Hz, 1H), 3.91 – 3.84 (m, 3H), 3.61 – 3.49 (m, 3H), 2.01 (q, *J* = 6.5 Hz, 2H), 1.94 (p, *J* = 6.8 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.39, 137.75, 133.85, 133.42, 129.63, 129.28, 128.88, 127.42, 127.22, 54.97, 54.24, 50.56, 32.24, 26.11, 24.24.

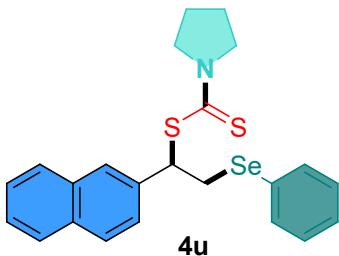


**1-(naphthalen-2-yl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4u)**

Yellow oil (74%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.82 – 7.76 (m, 4H), 7.50 (dd, *J* = 7.0, 2.3 Hz, 2H), 7.47 – 7.41 (m, 3H), 7.25 – 7.15 (m, 3H), 5.58 (dd, *J* = 11.0, 5.0 Hz, 1H), 3.94 (dd, *J* = 12.0, 5.0 Hz, 1H), 3.89 (q, *J* = 6.4 Hz, 2H), 3.64 – 3.55 (m, 2H), 3.49 (dt, *J* = 11.7, 6.9 Hz, 1H), 1.99 (dt, *J* = 12.3, 6.2 Hz, 2H), 1.93 (dt, *J* = 11.5, 6.6 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 190.65, 136.19, 133.57, 133.21, 132.99, 130.00, 128.89, 128.52, 128.07, 127.70, 127.47, 127.11, 126.35, 126.30, 126.19, 55.26, 54.95, 50.61, 33.26, 26.08, 24.25.

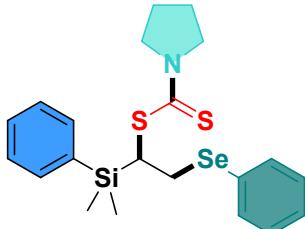


**1-(dimethyl(phenyl)silyl)-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4v)**

Yellow oil (99%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.62 (dd, *J* = 7.4, 1.8 Hz, 2H), 7.47 (dd, *J* = 6.4, 3.1 Hz, 2H), 7.36 (d, *J* = 7.4 Hz, 3H), 7.21 – 7.13 (m, 3H), 4.10 (dd, *J* = 14.0, 4.8 Hz, 1H), 3.99 – 3.86 (m, 1H), 3.81 (sex, *J* = 6.6 Hz, 2H), 3.45 (dd, *J* = 13.9, 10.6 Hz, 1H), 3.35 (dd, *J* = 11.7, 6.6 Hz, 1H), 3.31 – 3.20 (m, 2H), 2.05 – 1.88 (m, 4H), 0.50 (d, *J* = 5.2 Hz, 6H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 192.21, 136.62, 134.18, 132.99, 129.48, 128.71, 127.80, 126.73, 54.67, 50.42, 41.47, 30.38, 25.89, 24.27, -2.98, -3.50.



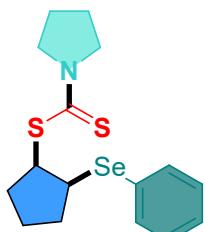
**4v:4v'=90:10, 99%**

**(1*R*,2*S*)-2-(phenylselanyl)cyclopentyl pyrrolidine-1-carbodithioate (4w)**

Yellow oil (96%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.64 – 7.58 (m, 2H), 7.27 (dd, *J* = 3.6, 1.9 Hz, 3H), 4.35 – 4.28 (m, 1H), 3.90 (t, *J* = 6.9 Hz, 2H), 3.69 (q, *J* = 5.9 Hz, 1H), 3.58 (t, *J* = 6.8 Hz, 2H), 2.56 – 2.49 (m, 1H), 2.21 (q, *J* = 8.9, 8.5 Hz, 1H), 2.04 (q, *J* = 6.8 Hz, 2H), 1.96 (q, *J* = 6.8 Hz, 2H), 1.86 – 1.79 (m, 2H), 1.78 – 1.69 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 191.99, 135.12, 128.93, 127.63, 55.95, 54.73, 50.67, 47.28, 33.03, 32.30, 26.12, 24.32, 23.44.



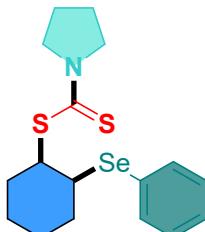
**4w, 96%**

**(1*R*,2*S*)-2-(phenylselanyl)cyclohexyl pyrrolidine-1-carbodithioate (4x)**

Yellow oil (96%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.68 – 7.59 (m, 2H), 7.27 (dt, *J* = 5.0, 2.5 Hz, 3H), 4.39 (q, *J* = 6.8 Hz, 1H), 3.91 (t, *J* = 6.9 Hz, 2H), 3.62 (dt, *J* = 10.5, 5.1 Hz, 3H), 2.41 (dq, *J* = 10.1, 5.9, 5.5 Hz, 1H), 2.04 (q, *J* = 6.8 Hz, 2H), 1.95 (p, *J* = 6.8 Hz, 2H), 1.74 (ddt, *J* = 42.5, 12.8, 5.1 Hz, 4H), 1.57 (p, *J* = 5.6 Hz, 2H), 1.46 – 1.39 (m, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 191.50, 135.43, 128.92, 127.64, 54.85, 50.69, 47.32, 26.04, 24.28.



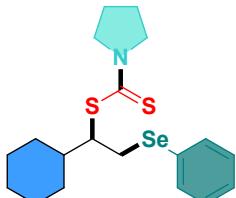
**4x, 96%**

**(R)-1-cyclohexyl-2-(phenylselanyl)ethyl pyrrolidine-1-carbodithioate (4y)**

Yellow oil (96%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.61 (dd, *J* = 6.5, 3.0 Hz, 2H), 7.28 – 7.22 (m, 3H), 3.94 – 3.81 (m, 3H), 3.75 (dd, *J* = 13.9, 7.3 Hz, 1H), 3.59 (dt, *J* = 13.6, 6.9 Hz, 2H), 3.47 – 3.40 (m, 1H), 2.03 (dt, *J* = 13.4, 6.8 Hz, 2H), 1.95 (q, *J* = 6.7 Hz, 2H), 1.89 (d, *J* = 12.1 Hz, 1H), 1.81 – 1.70 (m, 4H), 1.65 (s, 1H), 1.48 (q, *J* = 12.0, 10.6 Hz, 1H), 1.29 – 1.14 (m, 4H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 192.23, 134.64, 128.93, 127.31, 54.97, 53.04, 50.62, 41.59, 41.20, 31.73, 29.81, 26.35, 26.04, 24.30.



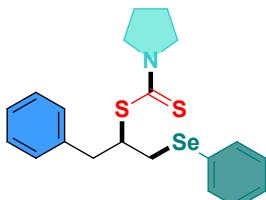
**4y:4y`=87:13, 99%**

**(R)-1-phenyl-3-(phenylselanyl)propan-2-yl pyrrolidine-1-carbodithioate (4z)**

Yellow oil (98%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.55 (dd, *J* = 7.9, 1.5 Hz, 2H), 7.33 – 7.18 (m, 8H), 3.88 (t, *J* = 7.0 Hz, 2H), 3.76 (dt, *J* = 8.6, 5.0 Hz, 2H), 3.70 – 3.42 (m, 3H), 3.20 (dd, *J* = 14.3, 5.1 Hz, 1H), 2.96 (dd, *J* = 14.3, 7.4 Hz, 1H), 2.04 (q, *J* = 6.7 Hz, 2H), 1.95 (m, *J* = 6.6 Hz, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 191.87, 138.92, 135.67, 133.06, 129.38, 129.00, 128.35, 127.91, 126.60, 55.08, 50.71, 45.36, 42.17, 40.64, 26.08, 24.32.



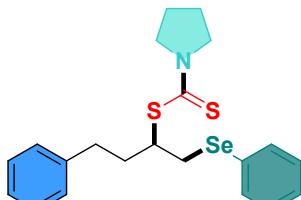
**4z:4z`=86:14, 98%**

**(R)-4-phenyl-1-(phenylselanyl)butan-2-yl pyrrolidine-1-carbodithioate (4aa)**

Yellow oil (99%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.61 (dd, *J* = 7.7, 1.4 Hz, 2H), 7.32 – 7.22 (m, 5H), 7.18 (d, *J* = 6.0 Hz, 3H), 3.89 (t, *J* = 6.9 Hz, 2H), 3.81 (dd, *J* = 13.9, 6.4 Hz, 1H), 3.63 (dq, *J* = 18.4, 6.3, 5.0 Hz, 3H), 3.46 (dt, *J* = 13.9, 7.9 Hz, 1H), 2.97 (ddd, *J* = 14.9, 10.2, 5.2 Hz, 1H), 2.83 – 2.74 (m, 1H), 2.14 (ddd, *J* = 15.8, 10.2, 5.8 Hz, 1H), 2.04 (q, *J* = 6.7 Hz, 2H), 2.01 – 1.85 (m, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 192.02, 141.54, 135.78, 133.37, 129.07, 128.53, 128.40, 128.00, 125.93, 55.12, 50.71, 44.03, 42.76, 35.87, 33.99, 26.07, 24.32.



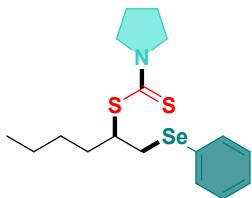
**4aa:4aa`=82:18, 99%**

**(R)-1-(phenylselanyl)hexan-2-yl pyrrolidine-1-carbodithioate (4ab)**

Yellow oil (80%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.61 (dd, *J* = 13.2, 8.6 Hz, 2H), 7.27 (dq, *J* = 13.4, 6.7 Hz, 3H), 3.90 (t, *J* = 6.9 Hz, 2H), 3.74 (dd, *J* = 13.7, 6.5 Hz, 1H), 3.63 (d, *J* = 8.2 Hz, 2H), 3.59 (dd, *J* = 13.4, 7.2 Hz, 1H), 3.46 (dt, *J* = 13.6, 6.7 Hz, 1H), 2.06 (m, 2H), 1.96 (m, 2H), 1.81 (ddt, *J* = 15.6, 10.9, 5.4 Hz, 1H), 1.67 – 1.55 (m, 2H), 1.46 (dq, *J* = 12.8, 6.6, 4.8 Hz, 1H), 1.32 (dt, *J* = 14.7, 7.2 Hz, 2H), 0.88 (dt, *J* = 13.5, 7.0 Hz, 3H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 192.22, 135.68, 133.11, 128.95, 127.82, 55.04, 50.67, 44.63, 42.87, 34.01, 29.88, 26.05, 24.30, 22.47, 14.04.



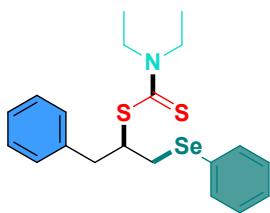
**4ab:4ab`=80:20, 80%**

**(R)-1-phenyl-3-(phenylselanyl)propan-2-yl diethylcarbamodithioate (5a)**

Yellow oil (87%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.61 – 7.50 (m, 2H), 7.31 – 7.21 (m, 8H), 4.05 – 3.92 (m, 2H), 3.89 – 3.65 (m, 4H), 3.63 (q, *J* = 6.6 Hz, 1H), 3.20 (dd, *J* = 14.2, 5.5 Hz, 1H), 2.99 – 2.93 (m, 1H), 1.25 (dq, *J* = 12.6, 6.4, 5.7 Hz, 6H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 194.67, 138.96, 135.68, 133.05, 129.38, 128.99, 128.34, 127.90, 126.59, 49.57, 46.78, 45.32, 42.80, 40.76, 12.59, 11.64.



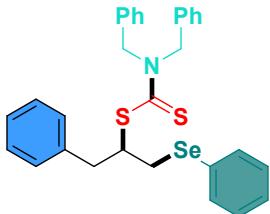
**5a:5a`=85:15, 87%**

**(R)-1-phenyl-3-(phenylselanyl)propan-2-yl dibenzylcarbamodithioate (5b)**

Yellow oil (54%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.55 (d, *J* = 6.9 Hz, 2H), 7.37 – 7.18 (m, 18H), 5.37 (d, *J* = 14.8 Hz, 1H), 5.22 (d, *J* = 14.3 Hz, 1H), 4.90 (q, *J* = 16.8 Hz, 2H), 3.97 – 3.72 (m, 2H), 3.69 (dd, *J* = 12.5, 6.6 Hz, 1H), 3.24 – 3.13 (m, 1H), 2.96 (dd, *J* = 14.2, 7.7 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 198.69, 138.95, 135.78, 133.31, 132.09, 129.50, 129.40, 129.09, 129.03, 128.81, 128.39, 128.29, 127.99, 127.90, 127.80, 127.17, 126.64, 56.28, 54.04, 45.29, 43.64, 40.79.



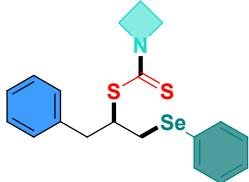
**5b:5b`=86:14, 54%**

**(R)-1-phenyl-3-(phenylselanyl)propan-2-yl azetidine-1-carbodithioate (5c)**

Yellow oil (90%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.57 – 7.50 (m, 2H), 7.25 (ddd, *J* = 24.5, 11.6, 7.4 Hz, 8H), 4.24 (t, *J* = 7.6 Hz, 2H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.75 – 3.62 (m, 2H), 3.62 – 3.56 (m, 1H), 3.18 (dd, *J* = 14.2, 5.5 Hz, 1H), 2.94 (dd, *J* = 14.3, 7.8 Hz, 1H), 2.39 – 2.31 (m, 2H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 193.23, 138.84, 135.66, 129.35, 129.03, 128.36, 127.95, 126.62, 54.65, 53.18, 45.61, 41.58, 40.52, 15.44.



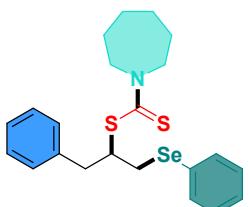
**5c:5c'=80:20, 90%**

**(R)-1-phenyl-3-(phenylselanyl)propan-2-yl azepane-1-carbodithioate (5d)**

Yellow oil (93%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.55 (dd, *J* = 7.8, 1.5 Hz, 2H), 7.26 (ddt, *J* = 19.8, 13.3, 7.8 Hz, 8H), 4.19 (dt, *J* = 12.1, 6.0 Hz, 1H), 4.13 – 4.08 (m, 1H), 3.93 – 3.81 (m, 2H), 3.80 – 3.71 (m, 2H), 3.67 – 3.60 (m, 1H), 3.20 (dd, *J* = 14.4, 5.3 Hz, 1H), 2.96 (dd, *J* = 14.3, 7.6 Hz, 1H), 1.89 – 1.80 (m, 4H), 1.59 – 1.51 (m, 4H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 195.43, 138.98, 135.69, 133.07, 129.40, 128.98, 128.34, 127.90, 126.59, 55.73, 52.89, 45.40, 42.71, 40.74, 27.41, 26.68, 26.59, 26.28.



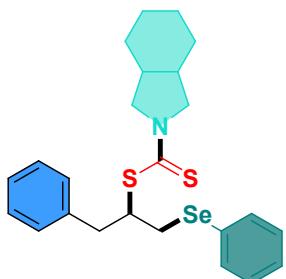
**5d:5d'=85:15, 93%**

**(R)-1-phenyl-3-(phenylselanyl)propan-2-yl octahydro-2H-isoindole-2-carbodithioate (5e)**

Yellow oil (93%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.55 (d, *J* = 7.5 Hz, 2H), 7.30 – 7.21 (m, 8H), 3.91 – 3.83 (m, 1H), 3.83 – 3.78 (m, 1H), 3.78 – 3.70 (m, 2H), 3.67 – 3.59 (m, 2H), 3.55 – 3.50 (m, 1H), 3.20 (dd, *J* = 14.0, 5.1 Hz, 1H), 2.96 (dd, *J* = 14.9, 7.8 Hz, 1H), 2.37 (dt, *J* = 12.6, 6.2 Hz, 1H), 2.28 (dt, *J* = 13.2, 6.6 Hz, 1H), 1.64 – 1.58 (m, 2H), 1.54 – 1.48 (m, 2H), 1.41 (dt, *J* = 16.0, 8.5 Hz, 4H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 192.66, 138.93, 135.71, 129.39, 128.99, 128.34, 127.91, 126.59, 58.88, 54.73, 45.48, 42.23, 40.68, 37.67, 35.85, 25.76, 25.64, 22.61, 22.45.



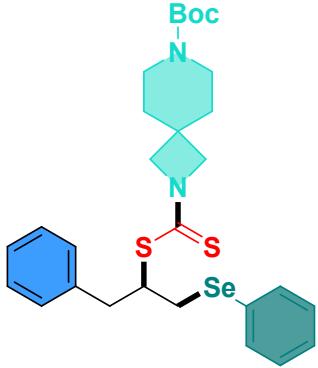
**5e:5e'=88:12, 93%**

**Tert-butyl-2-(((1-phenyl-3-(phenylselanyl)propan-2-yl)thio)carbonothioyl)-2,7-diazaspiro[3.5] nonane-7-carboxylate (5f)**

Yellow oil (68%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.56 – 7.50 (m, 2H), 7.25 (m, 8H), 3.94 (d, *J* = 11.5 Hz, 2H), 3.86 (d, *J* = 4.9 Hz, 1H), 3.78 (d, *J* = 3.2 Hz, 1H), 3.73 – 3.66 (m, 1H), 3.63 – 3.57 (m, 1H), 3.42 – 3.31 (m, 4H), 3.26 – 3.07 (m, 2H), 2.95 (dd, *J* = 14.3, 7.6 Hz, 1H), 1.77 – 1.69 (m, 4H), 1.46 (s, 9H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 194.41, 154.66, 138.77, 135.69, 133.17, 129.33, 129.04, 128.38, 128.00, 126.65, 79.94, 63.84, 62.43, 53.07, 45.51, 41.81, 40.60, 34.95, 34.29, 32.49, 28.44.



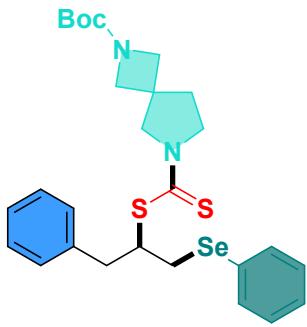
**5f:5f'=61:39, 68%**

**Tert-butyl-6-(((1-phenyl-3-(phenylselanyl)propan-2-yl)thio)carbonothioyl)-2,6-diazaspiro[3.4] octane-2-carboxylate (5g)**

Yellow oil (70%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.59 – 7.47 (m, 2H), 7.37 – 7.17 (m, 8H), 4.03 (s, 1H), 4.02 – 3.88 (m, 3H), 3.84 (td, *J* = 10.0, 8.7, 4.1 Hz, 3H), 3.80 (s, 1H), 3.71 (dq, *J* = 18.0, 6.7, 5.4 Hz, 2H), 3.63 (dd, *J* = 16.7, 10.6 Hz, 1H), 3.17 (dd, *J* = 13.9, 4.6 Hz, 1H), 2.96 (dd, *J* = 14.1, 7.4 Hz, 1H), 2.22 (dt, *J* = 20.0, 7.0 Hz, 1H), 2.13 (dt, *J* = 13.0, 7.1 Hz, 1H), 1.44 (s, 9H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 193.03, 156.12, 138.80, 135.72, 135.68, 129.34, 129.03, 128.38, 128.00, 126.65, 79.95, 63.28, 59.29, 53.27, 49.18, 45.14, 40.72, 38.27, 36.28, 34.61, 28.38.



**5g:5g'=67:33, 70%**

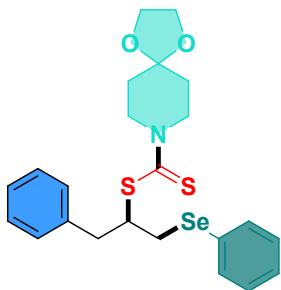
**1-phenyl-3-(phenylselanyl)propan-2-yl 1,4-dioxa-8-azaspiro[4.5]decane-8-carbodithioate(5h)**

Yellow oil (96%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.55 (d, *J* = 7.9 Hz, 2H), 7.31 – 7.19 (m, 8H), 4.38 (d, *J* = 33.3 Hz, 2H), 3.97 (t, *J* = 2.3 Hz, 6H), 3.74 (q, *J* = 8.2, 7.2 Hz, 2H), 3.66 – 3.60 (m, 1H), 3.18 (dd, *J* = 14.0, 5.6 Hz, 1H), 2.96 (dd, *J* = 14.1, 7.3 Hz, 1H), 1.77 (s, 4H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 195.65, 138.86, 135.74, 129.38, 129.02, 128.37, 127.99, 126.63,

106.60, 64.66, 47.84, 45.22, 43.11, 40.75, 35.12.



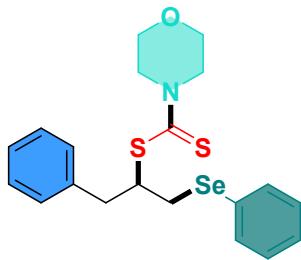
**5h:5h` = 84:16, 96%**

**(R)-1-phenyl-3-(phenylselanyl)propan-2-yl morpholine-4-carbodithioate (5i)**

Yellow oil (70%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.53 (dd, *J* = 18.0, 5.5 Hz, 2H), 7.32 – 7.19 (m, 8H), 4.29 (s, 2H), 3.95 (s, 2H), 3.80 – 3.62 (m, 7H), 3.20 – 3.14 (m, 1H), 2.97 (dd, *J* = 14.3, 7.1 Hz, 1H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 196.71, 138.79, 135.74, 129.35, 129.04, 128.39, 128.03, 126.68, 66.44, 53.66, 45.08, 42.62, 40.79, 39.34, 32.25.



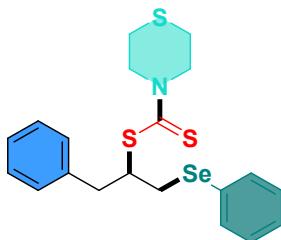
**5i:5i` = 74:26, 70%**

**(R)-1-phenyl-3-(phenylselanyl)propan-2-yl thiomorpholine-4-carbodithioate (5j)**

Yellow oil (56%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.59 – 7.48 (m, 2H), 7.33 – 7.17 (m, 8H), 4.75 – 4.04 (m, 5H), 3.76 – 3.71 (m, 1H), 3.69 – 3.60 (m, 1H), 3.20 – 3.14 (m, 1H), 2.98 (dd, *J* = 14.3, 7.3 Hz, 1H), 2.70 (d, *J* = 6.3 Hz, 4H).

**<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 196.01, 138.79, 135.70, 133.14, 129.35, 129.04, 128.39, 128.01, 126.67, 53.90, 45.07, 42.82, 40.82, 39.36, 32.24, 27.26.



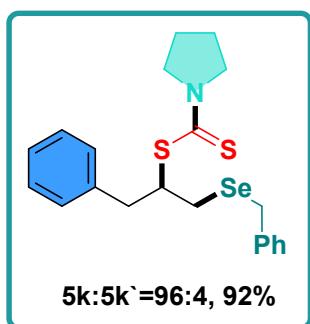
**5j:5j` = 80:20, 56%**

**(R)-1-(benzylselanyl)-3-phenylpropan-2-yl pyrrolidine-1-carbodithioate (5k)**

Yellow oil (92%);

**<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.24 (dq, *J* = 12.6, 6.3, 5.7 Hz, 7H), 7.16 (d, *J* = 7.0 Hz, 3H), 3.95 – 3.91 (m, 3H), 3.83 (q, *J* = 11.7 Hz, 2H), 3.65 (dt, *J* = 11.7, 5.8 Hz, 2H), 3.57 (dd, *J* = 13.8, 8.4 Hz, 1H), 3.43 (ddd, *J* = 13.7, 8.3, 5.4 Hz, 1H), 3.19 (dd, *J* = 14.1, 5.1 Hz, 1H), 2.91 (dd, *J* = 14.1, 8.3 Hz, 1H), 2.07 (m, 2H), 1.98 (m, 2H).

**$^{13}\text{C}$  NMR** (126 MHz,  $\text{CDCl}_3$ )  $\delta$  192.07, 139.34, 138.84, 129.42, 129.03, 128.43, 128.28, 126.59, 126.54, 55.14, 50.72, 42.53, 41.44, 40.94, 27.70, 26.08, 24.33.

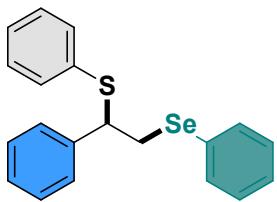


**(R)-phenyl(1-phenyl-2-(phenylselanyl)ethyl)sulfane (6a)**

Yellow oil (91%);

**$^1\text{H}$  NMR** (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 (d,  $J = 5.3$  Hz, 4H), 7.24 (d,  $J = 11.4$  Hz, 6H), 7.20 (d,  $J = 7.6$  Hz, 2H), 7.16 (t,  $J = 6.5$  Hz, 3H), 4.25 (dd,  $J = 10.6, 4.3$  Hz, 1H), 3.49 (dd,  $J = 12.8, 5.6$  Hz, 1H), 3.39 – 3.30 (m, 1H).

**$^{13}\text{C}$  NMR** (126 MHz,  $\text{CDCl}_3$ )  $\delta$  139.60, 135.47, 134.16, 132.93, 129.98, 129.00, 128.99, 128.59, 128.13, 127.85, 127.70, 126.46, 52.36, 39.65.

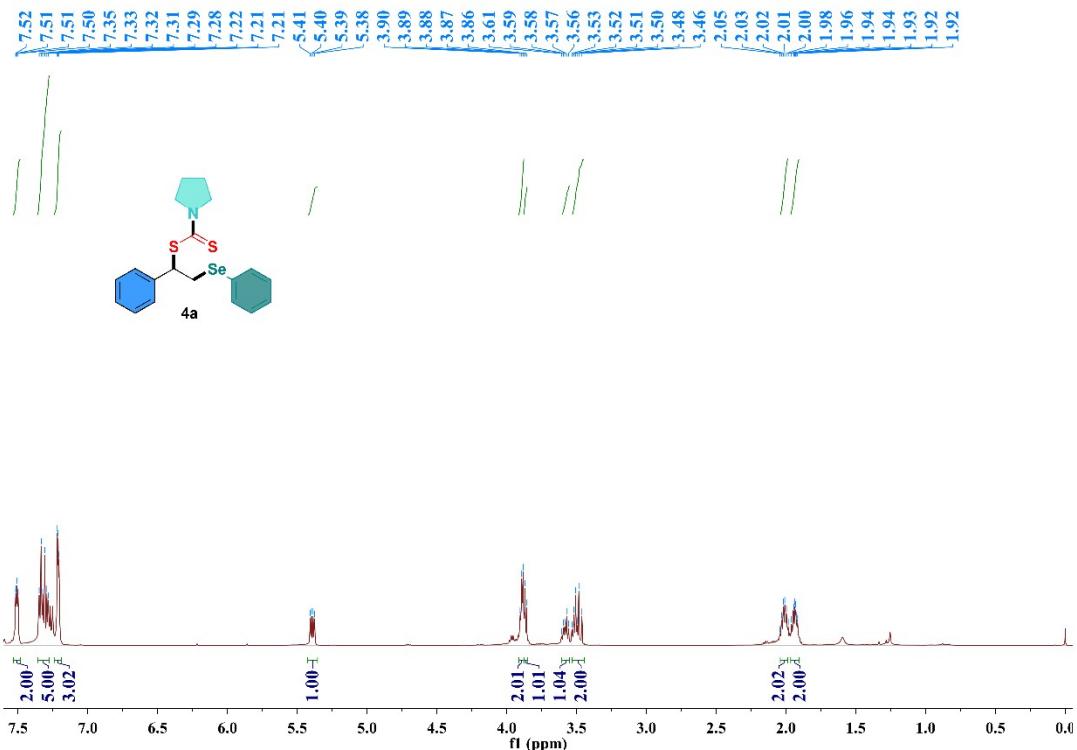


**References:**

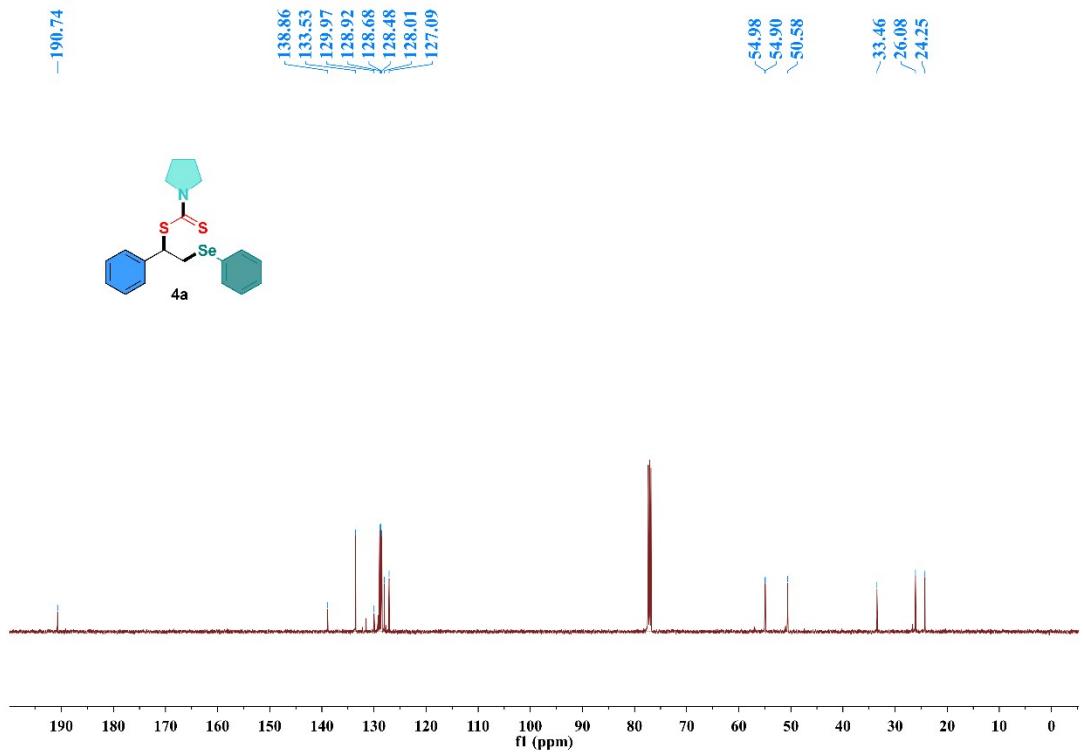
1. Zhou, Y.; Zhang, J.-Q.; Ren, H.; Dong, Z.-B., Electrochemical Difunctionalization of Alkenes: Simultaneous Construction of C–Se and C–S Bonds. *J. Org. Chem.* **2023**, 88 (9), 5321–5328.

#### 4 $^1\text{H}$ -NMR and $^{13}\text{C}$ -NMR spectra for target products

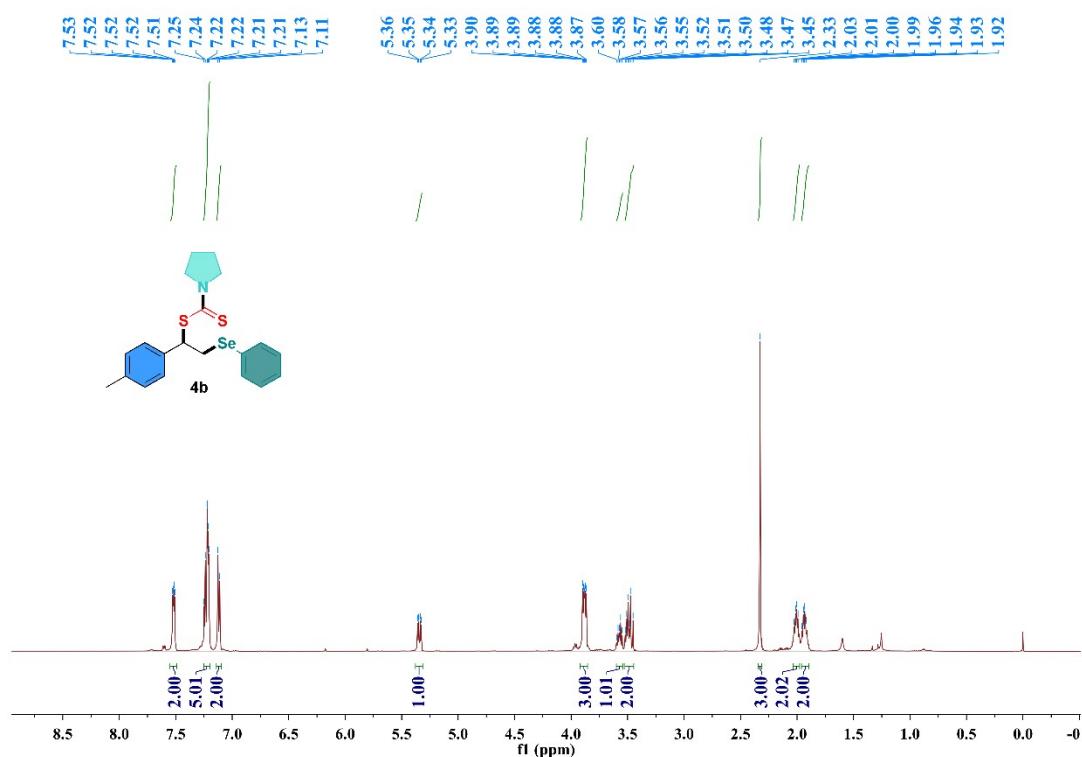
$^1\text{H}$  NMR spectra of compound 4a



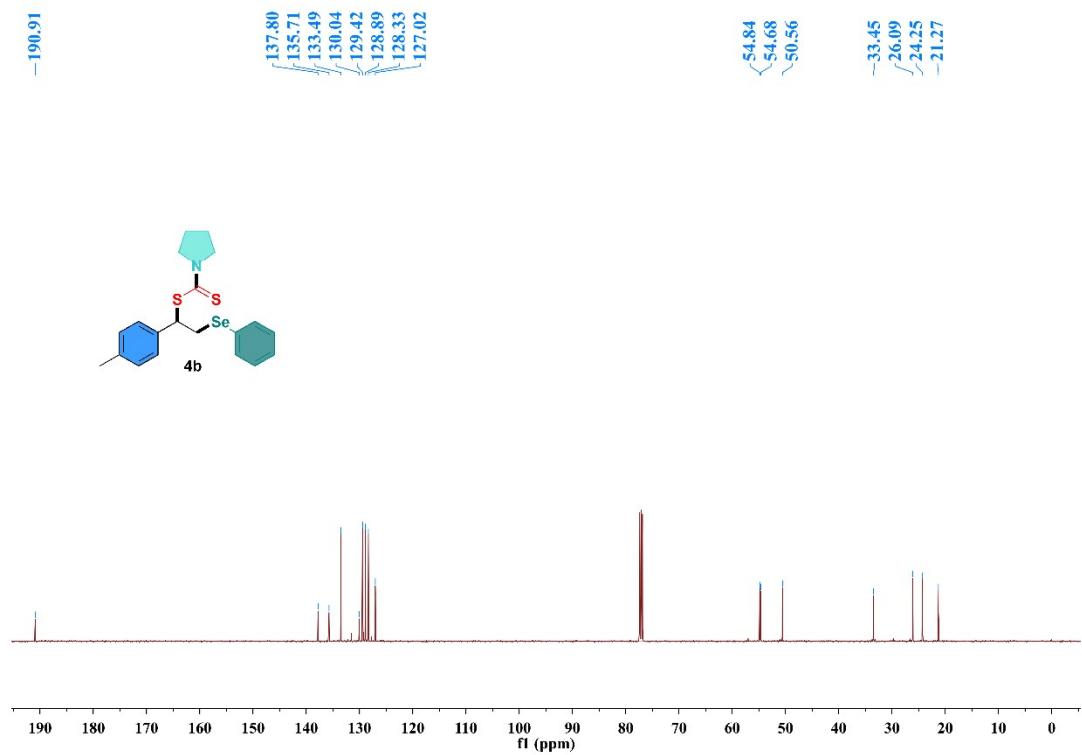
$^{13}\text{C}$  NMR spectra of compound 4a



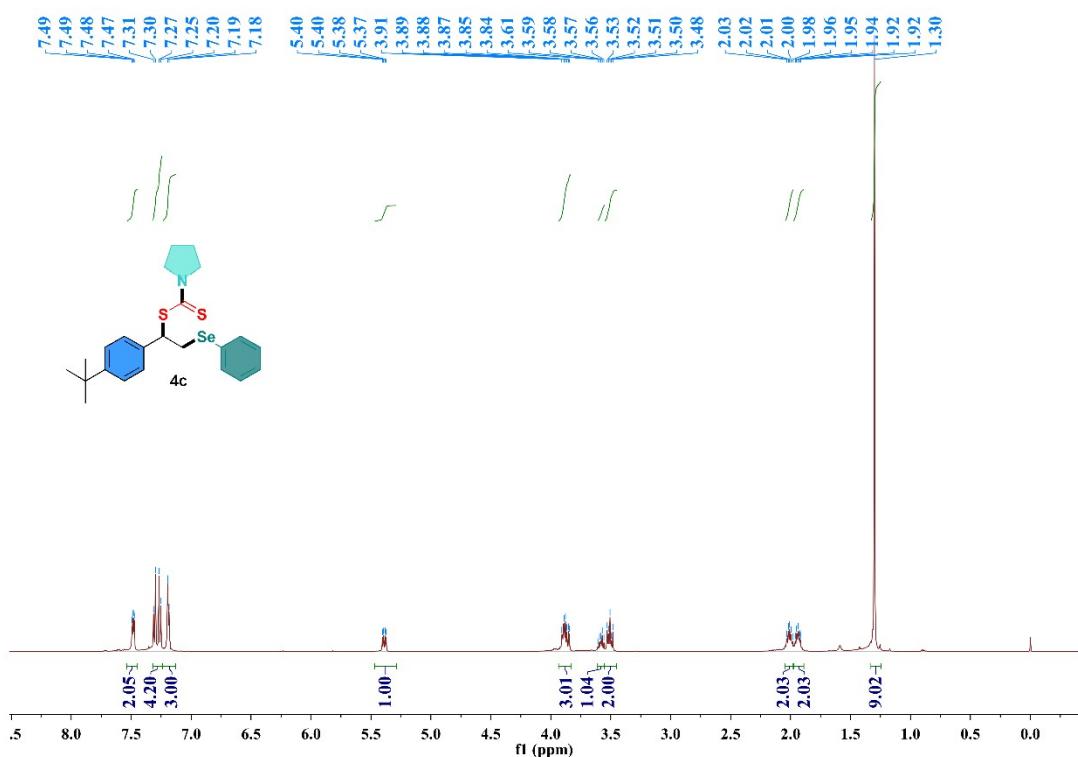
<sup>1</sup>H NMR spectra of compound **4b**



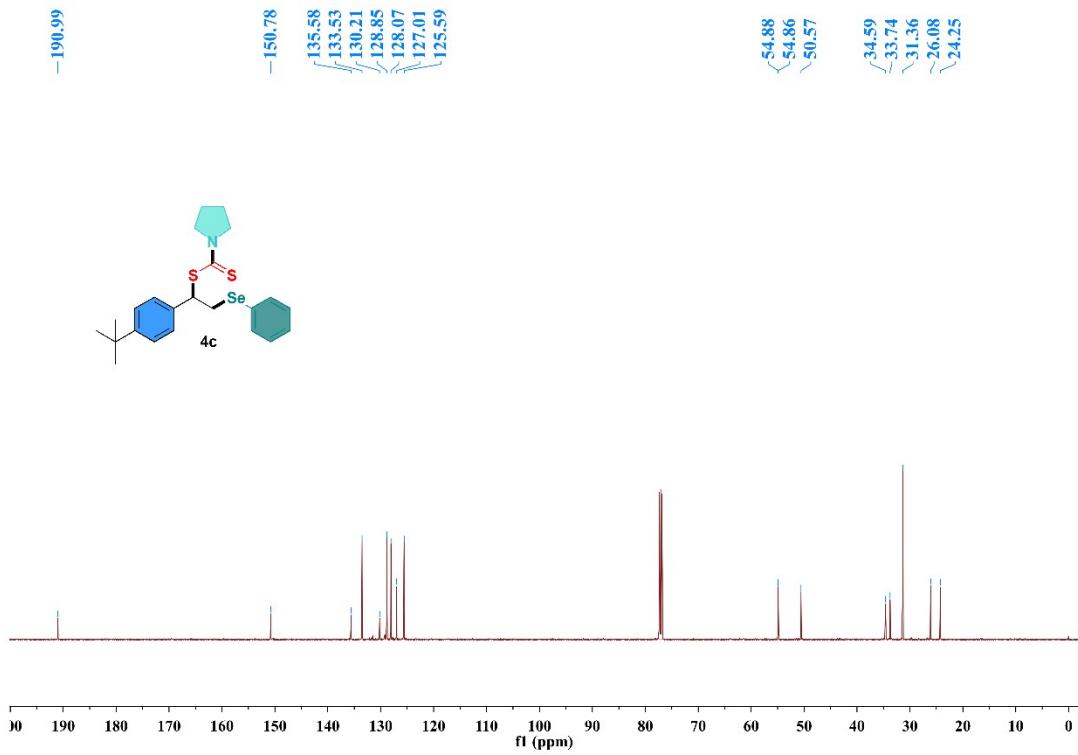
<sup>13</sup>C NMR spectra of compound **4b**



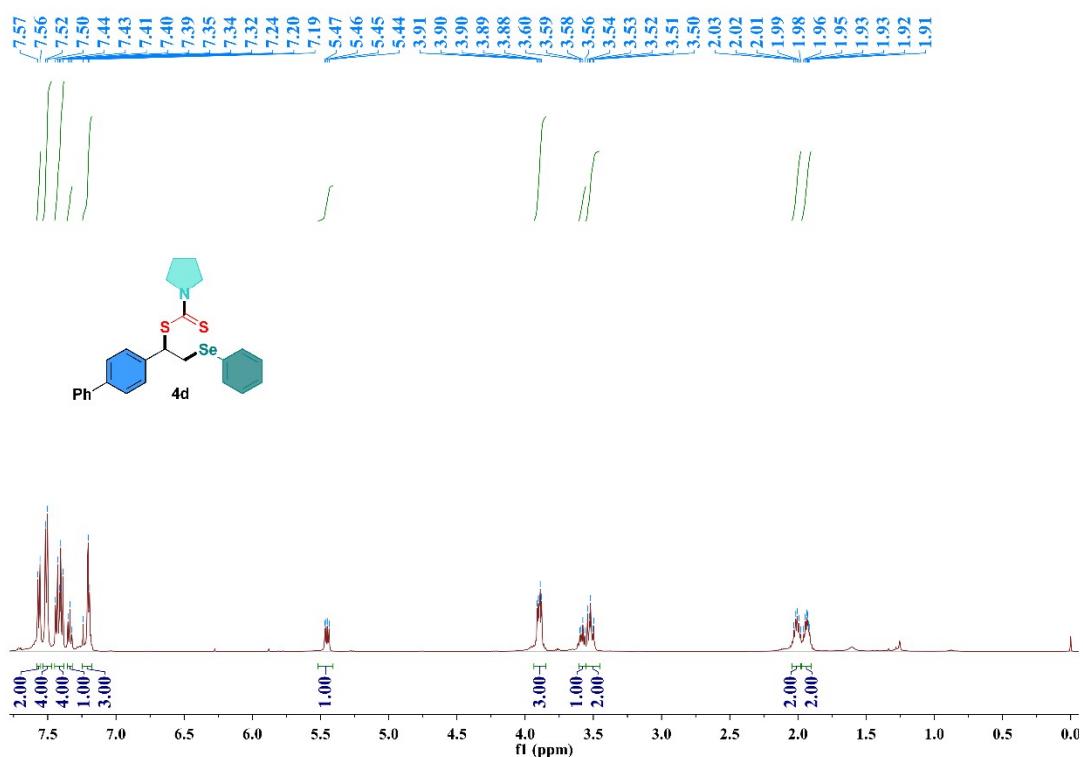
<sup>1</sup>H NMR spectra of compound **4c**



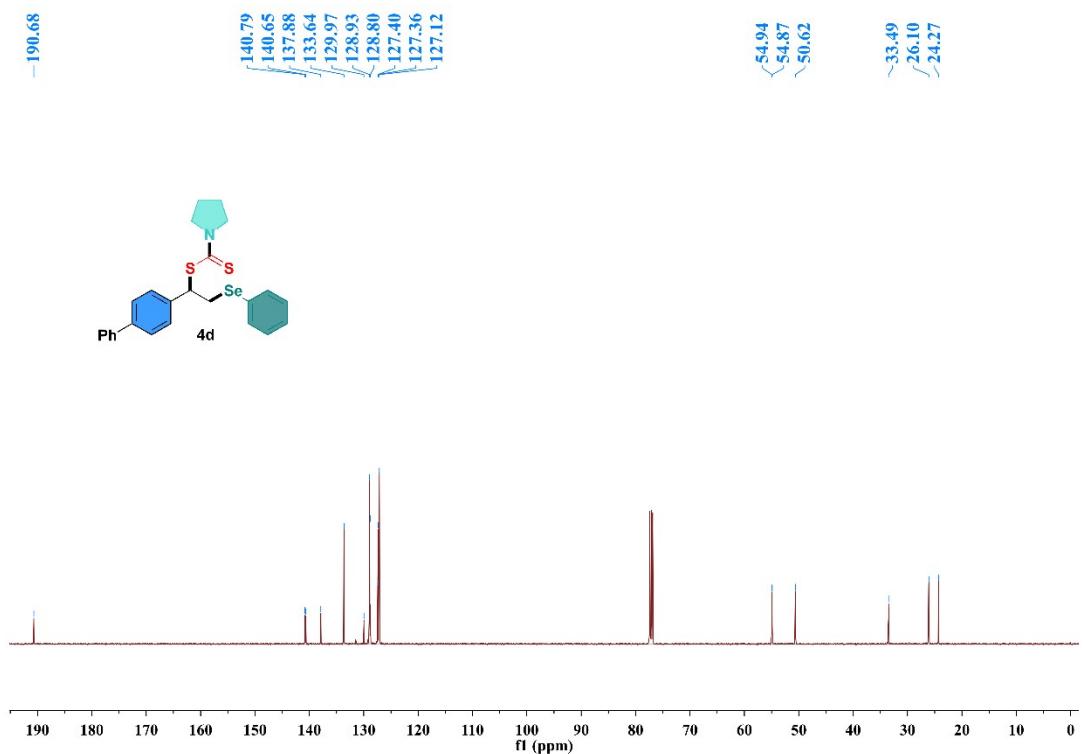
### <sup>13</sup>C NMR spectra of compound 4c



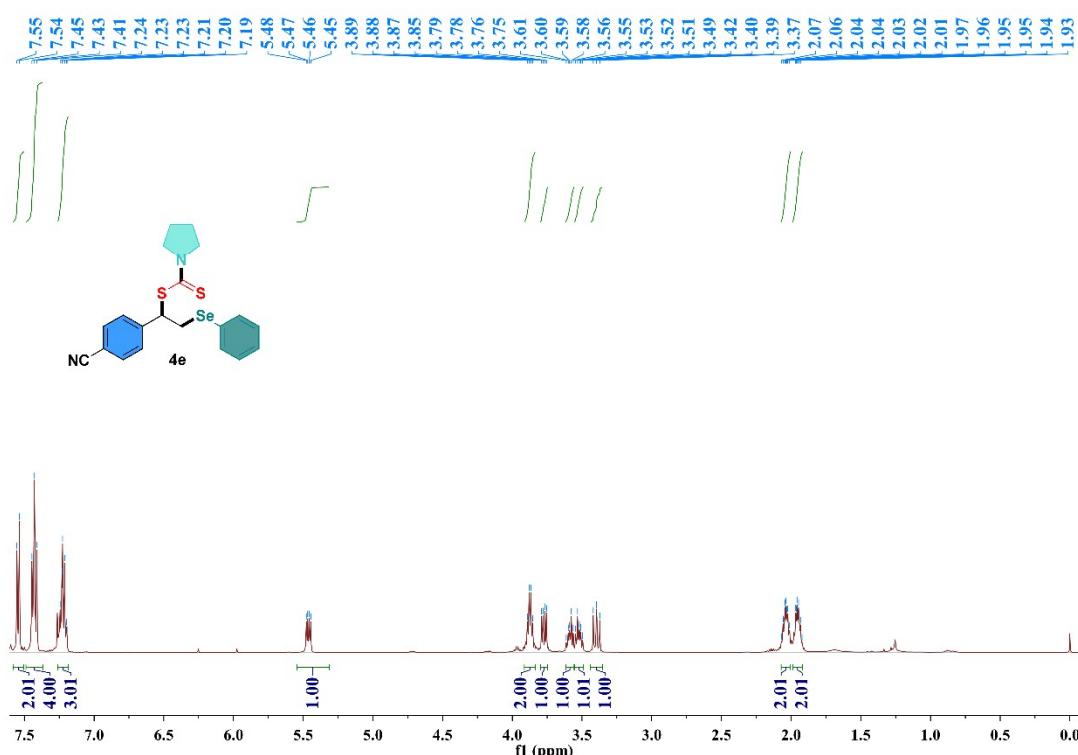
<sup>1</sup>H NMR spectra of compound **4d**



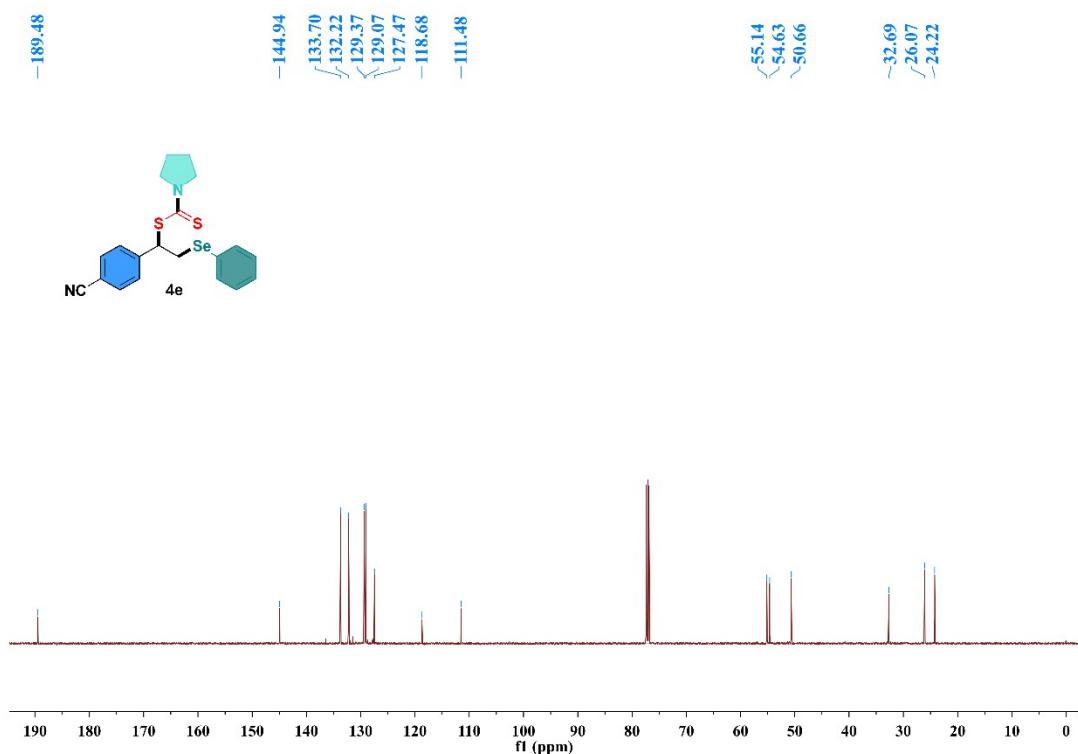
<sup>13</sup>C NMR spectra of compound **4d**



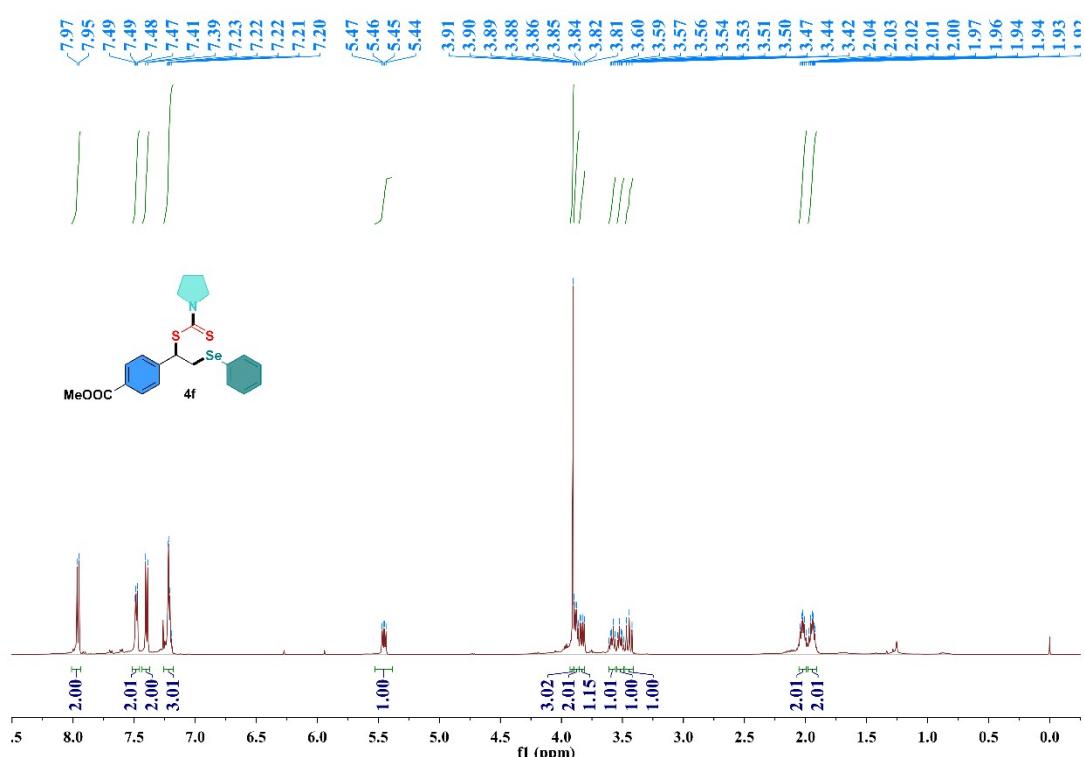
<sup>1</sup>H NMR spectra of compound **4e**



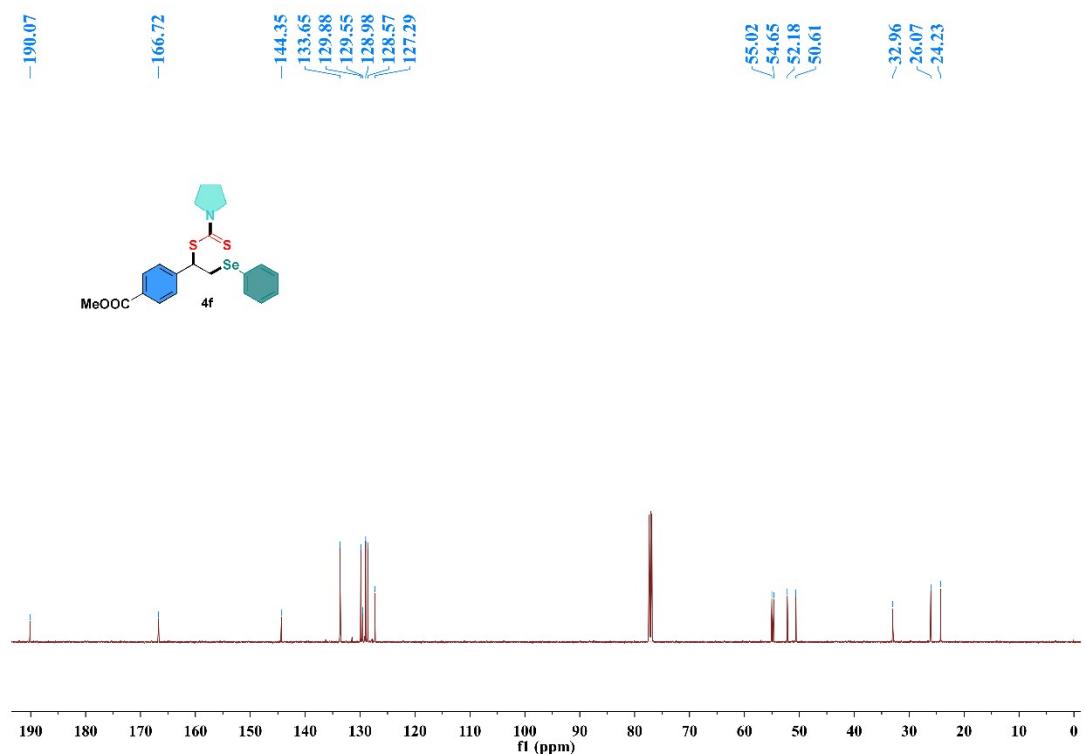
<sup>13</sup>C NMR spectra of compound **4e**



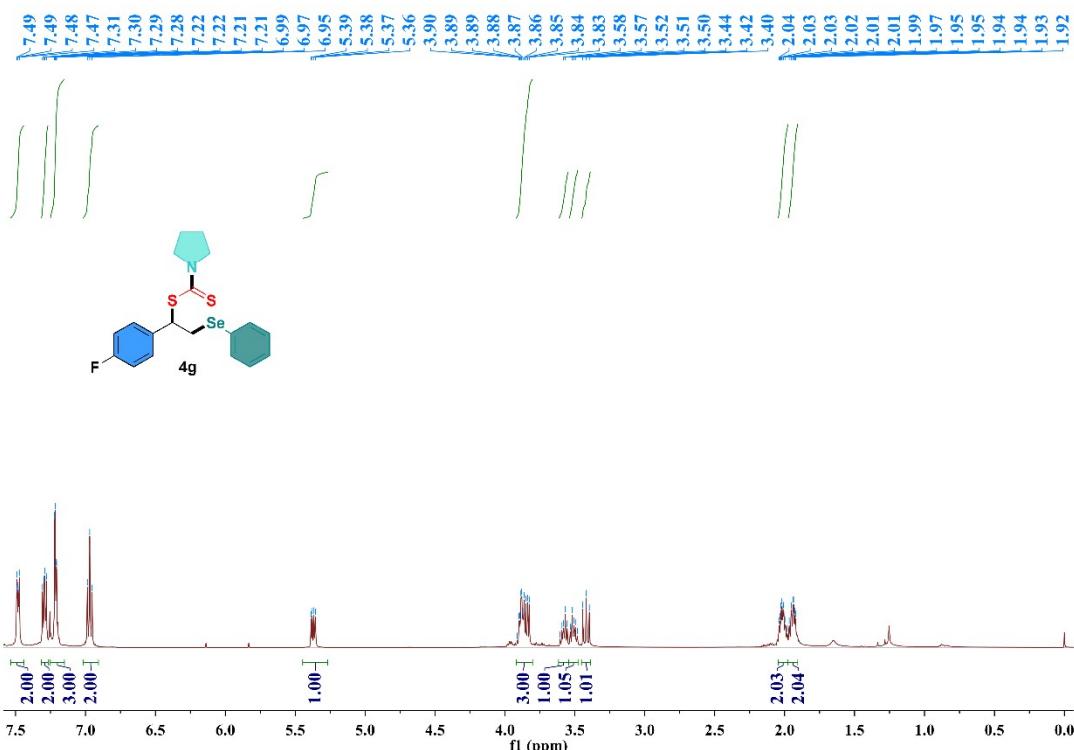
<sup>1</sup>H NMR spectra of compound **4f**



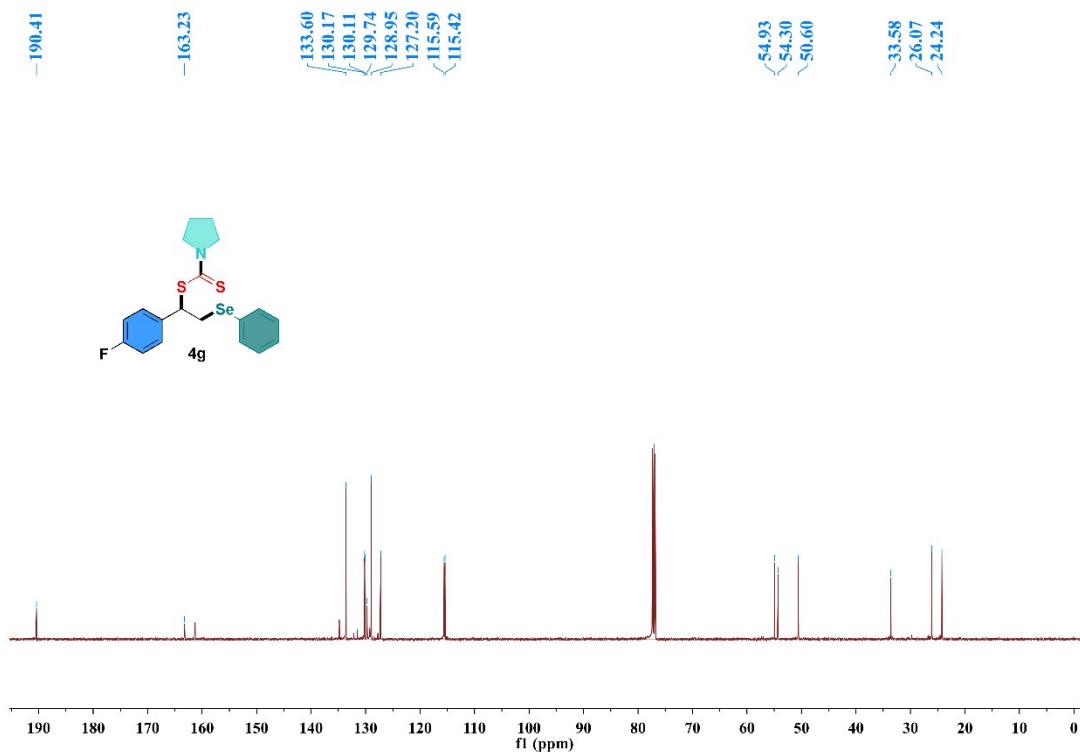
<sup>13</sup>C NMR spectra of compound **4f**



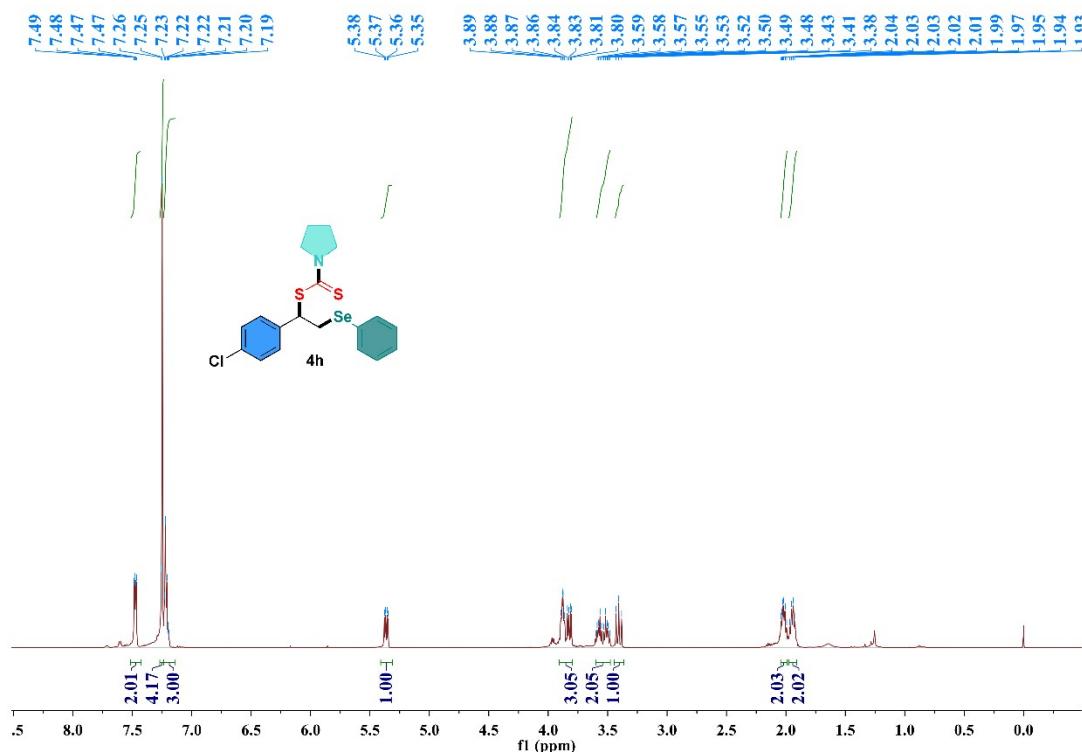
<sup>1</sup>H NMR spectra of compound 4g



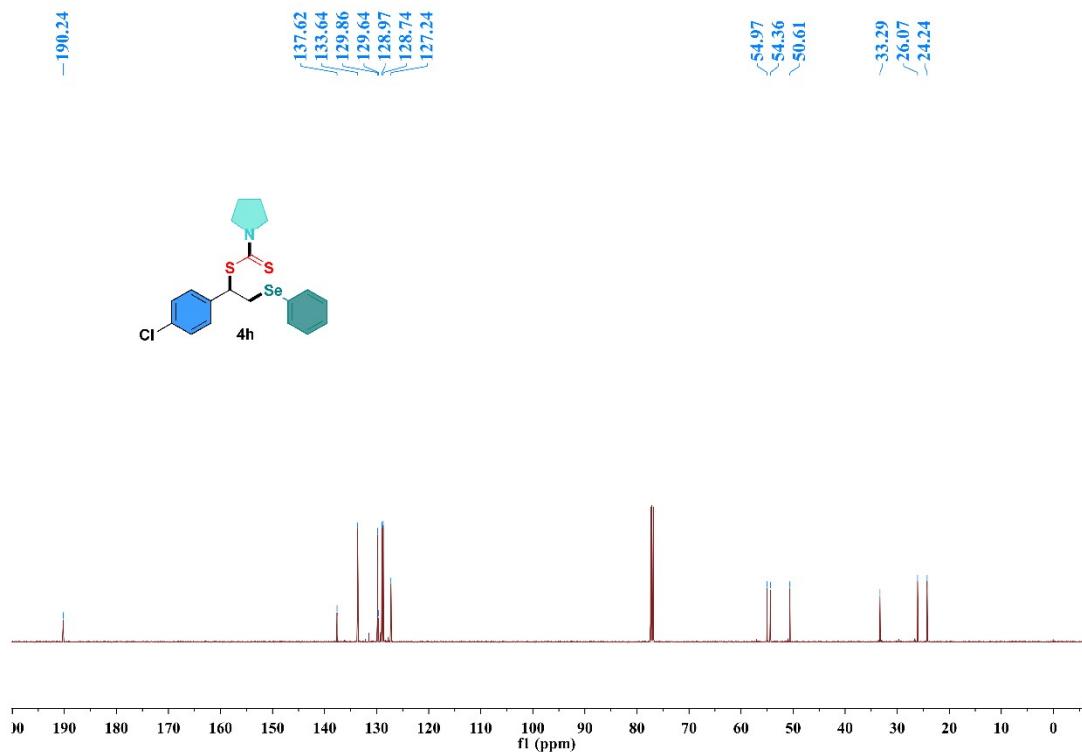
<sup>13</sup>C NMR spectra of compound 4g



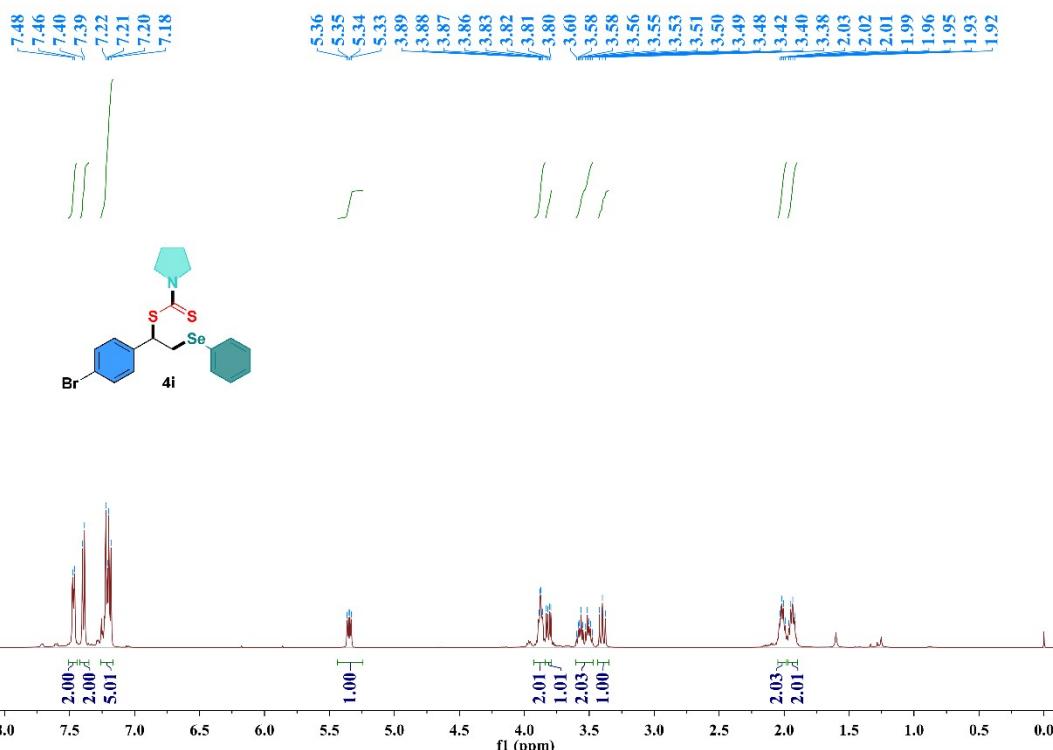
<sup>1</sup>H NMR spectra of compound **4h**



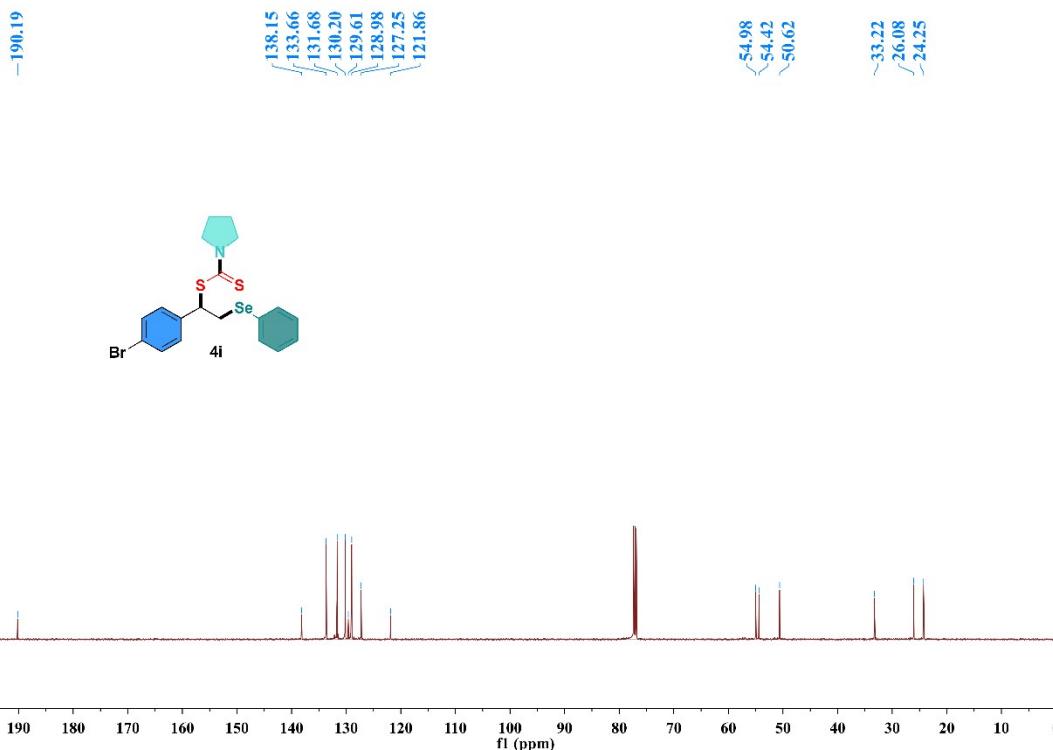
<sup>13</sup>C NMR spectra of compound **4h**



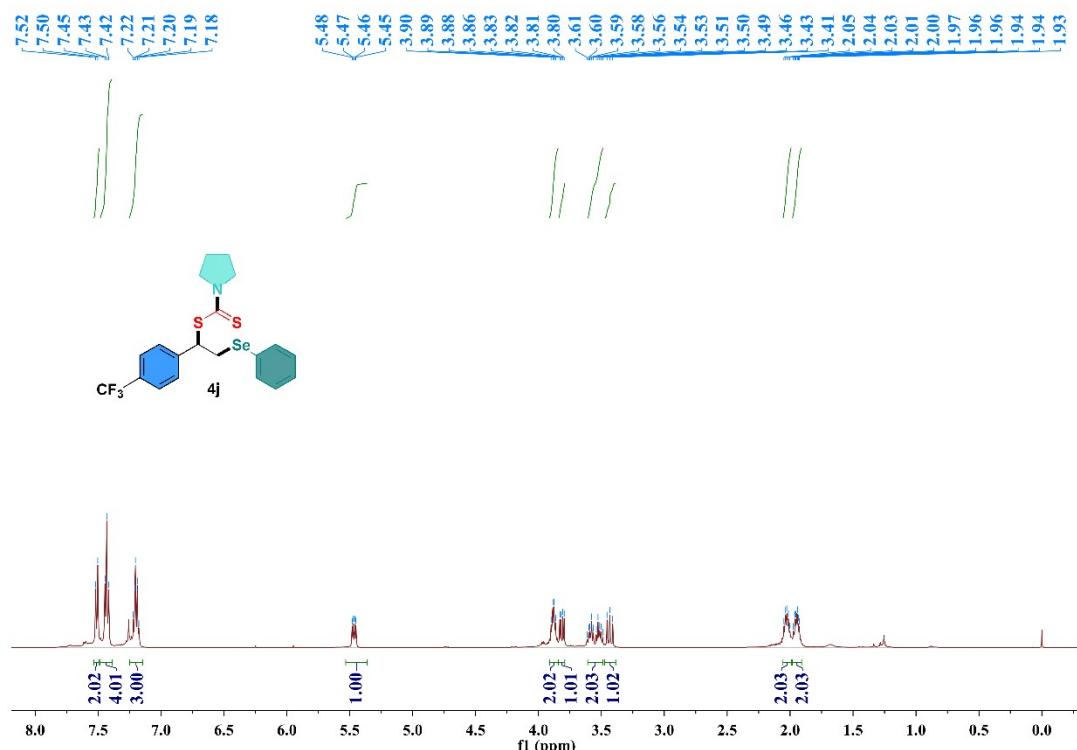
<sup>1</sup>H NMR spectra of compound **4i**



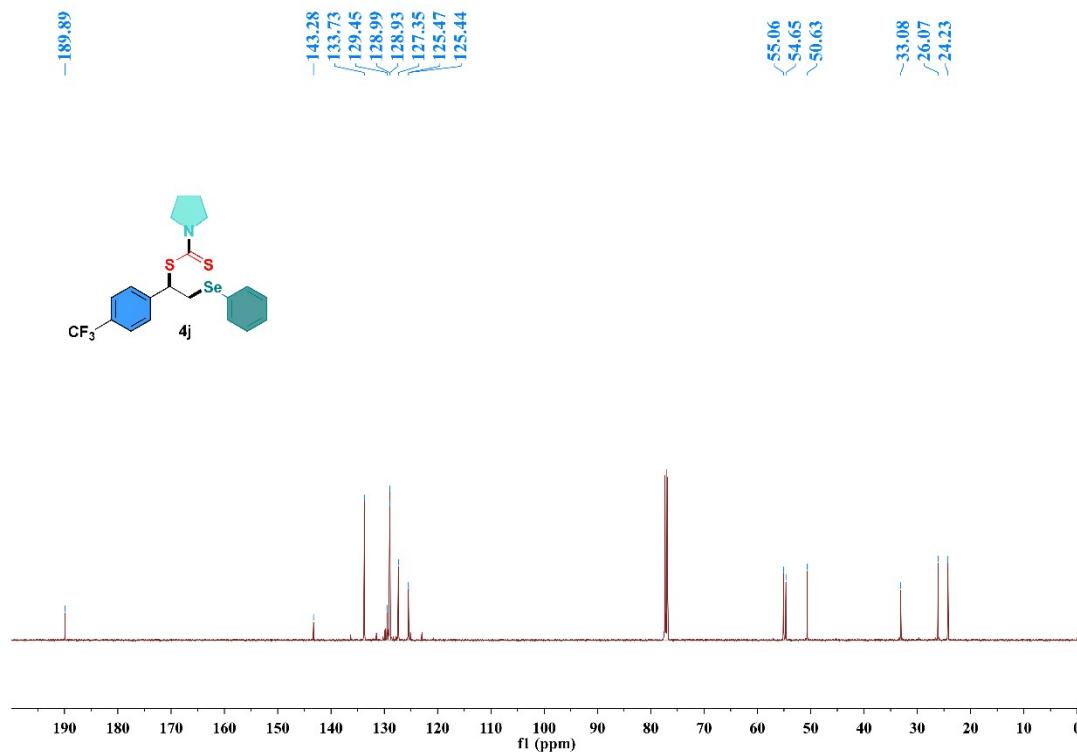
<sup>13</sup>C NMR spectra of compound **4i**



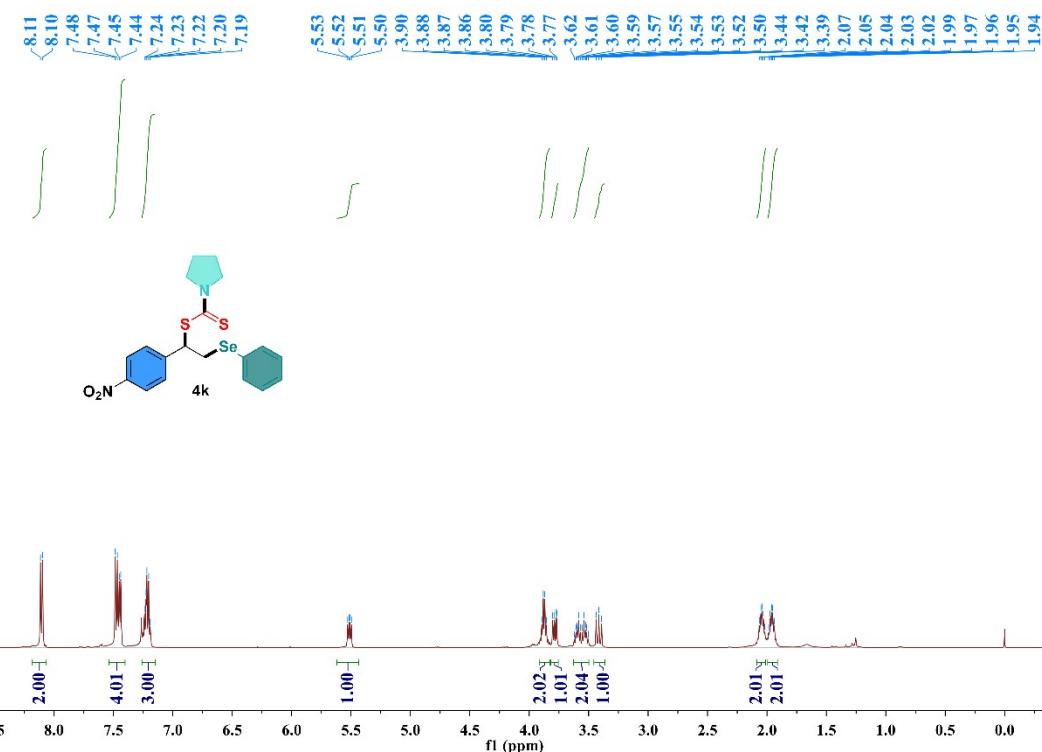
<sup>1</sup>H NMR spectra of compound **4j**



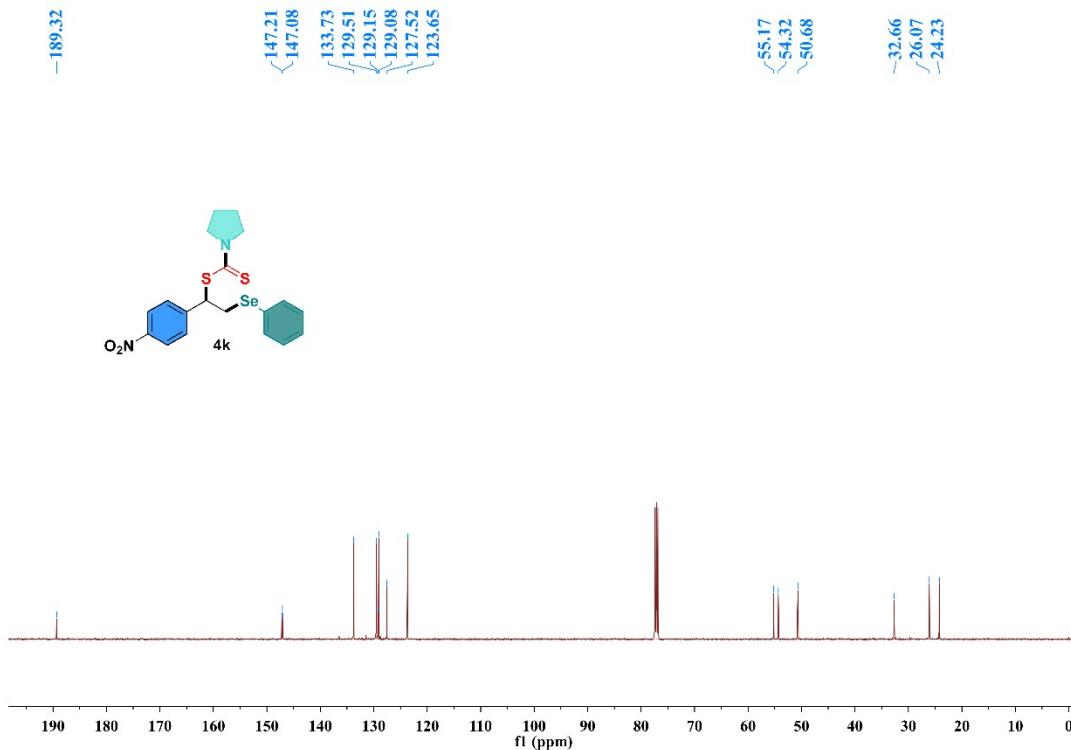
<sup>13</sup>C NMR spectra of compound **4j**



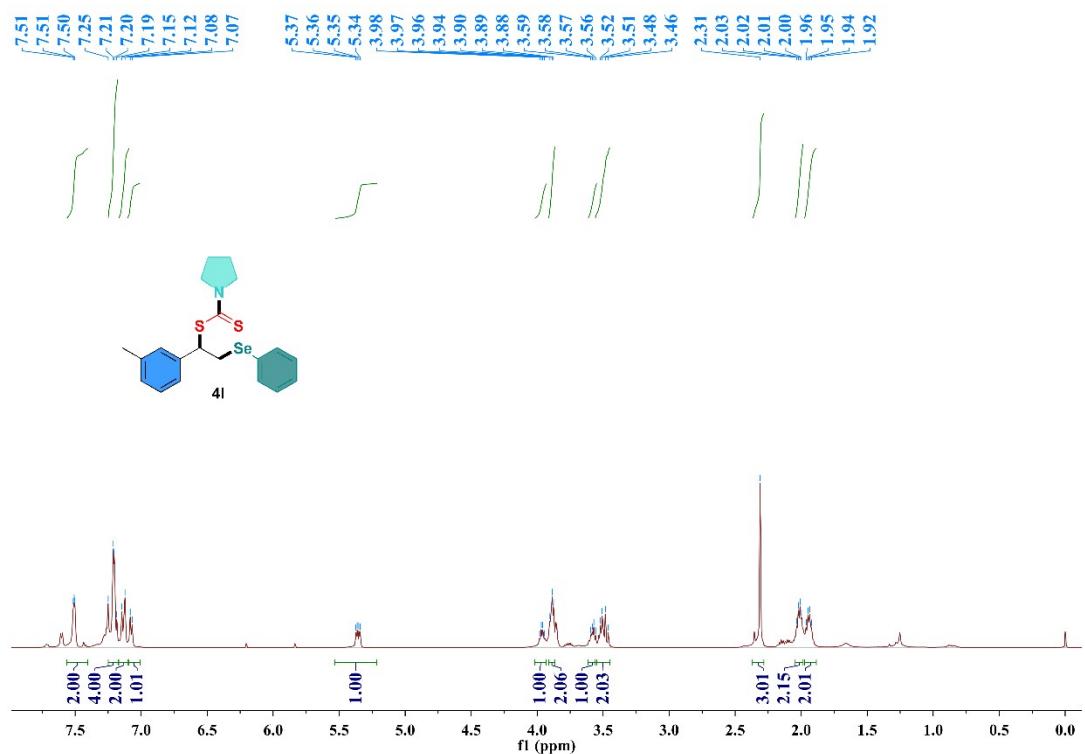
<sup>1</sup>H NMR spectra of compound **4k**



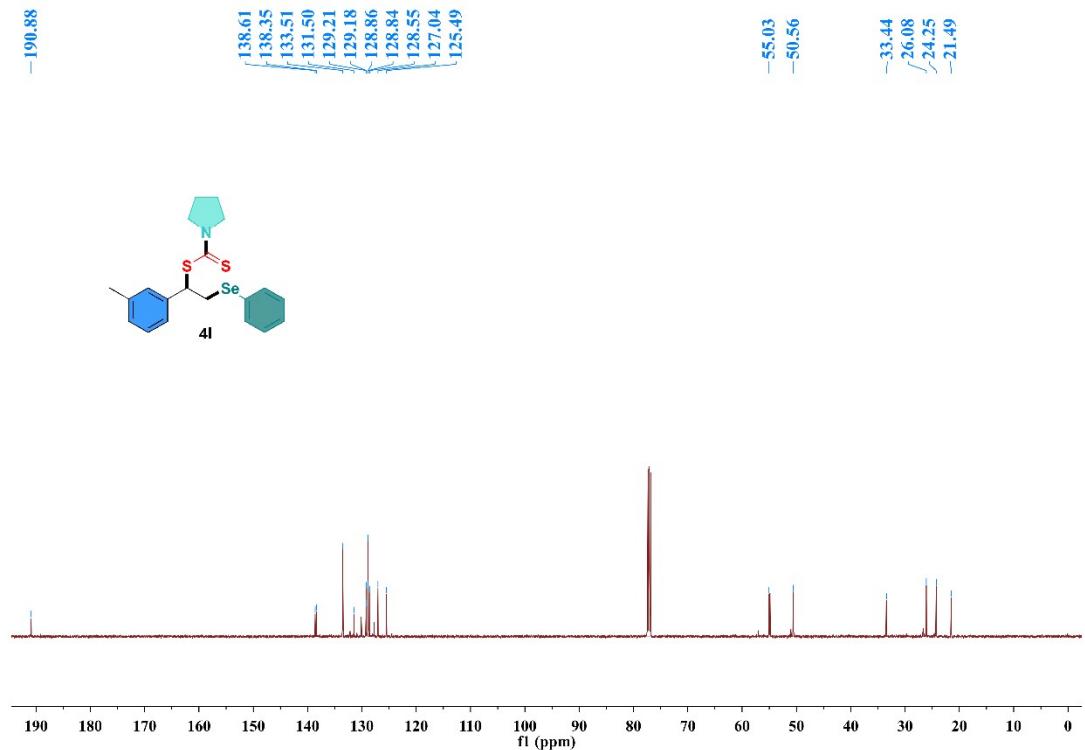
### <sup>13</sup>C NMR spectra of compound 4k



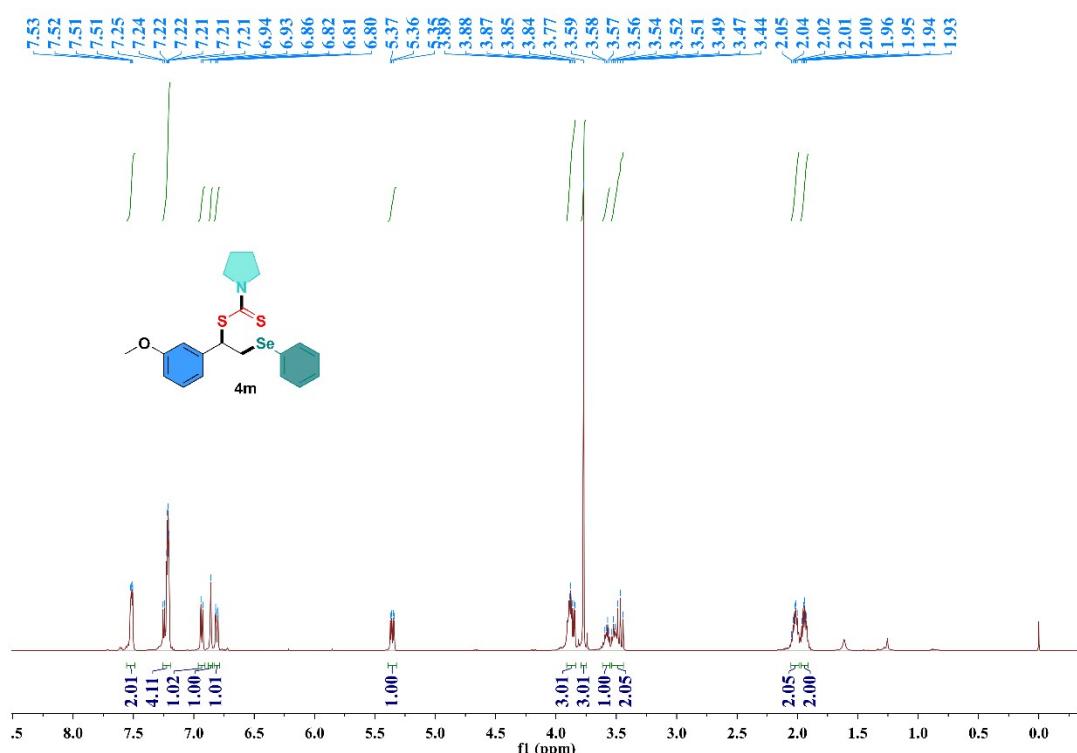
<sup>1</sup>H NMR spectra of compound **4l**



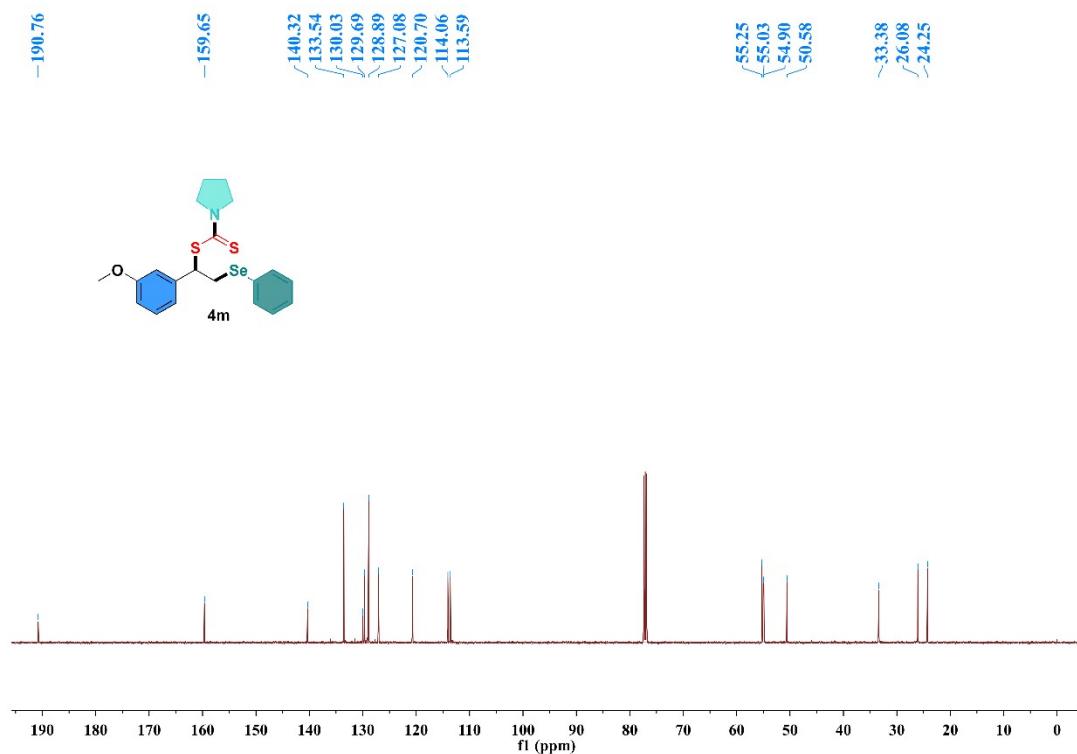
### <sup>13</sup>C NMR spectra of compound 4l



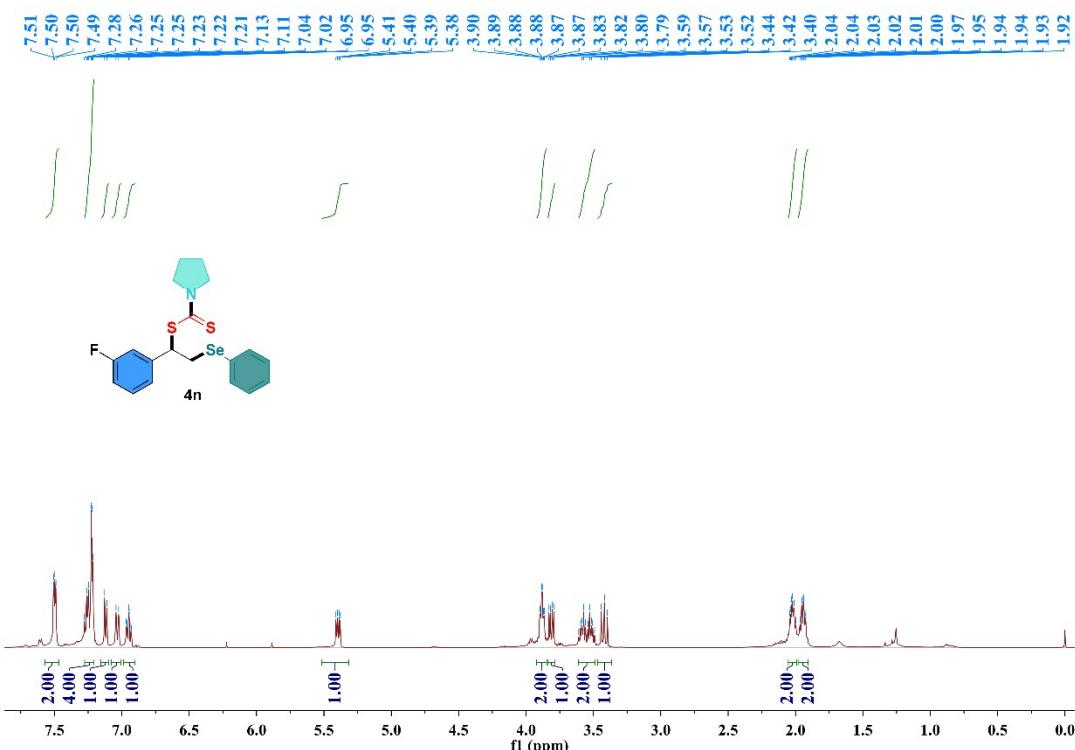
<sup>1</sup>H NMR spectra of compound **4m**



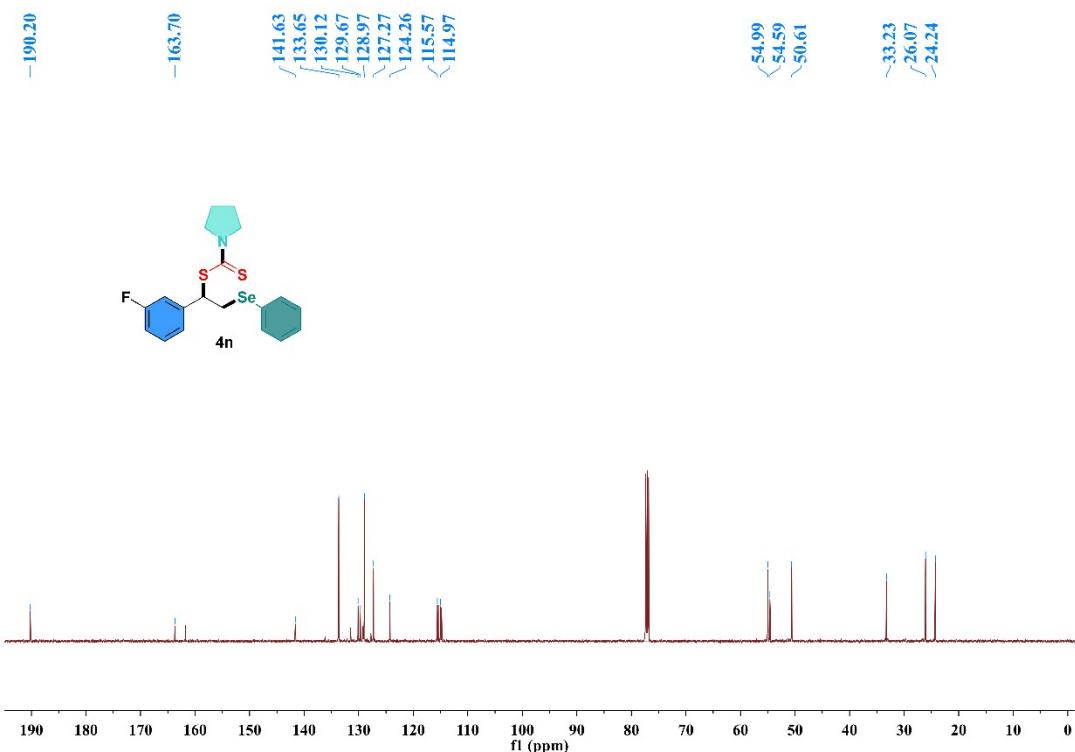
<sup>13</sup>C NMR spectra of compound **4m**



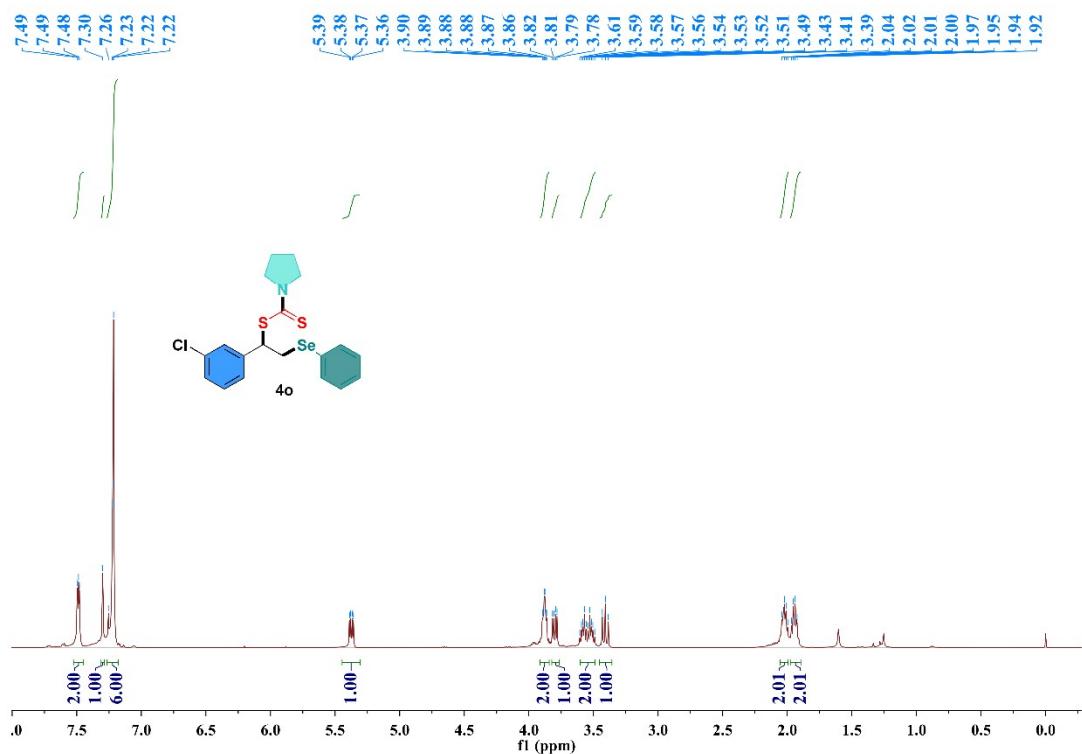
<sup>1</sup>H NMR spectra of compound **4n**



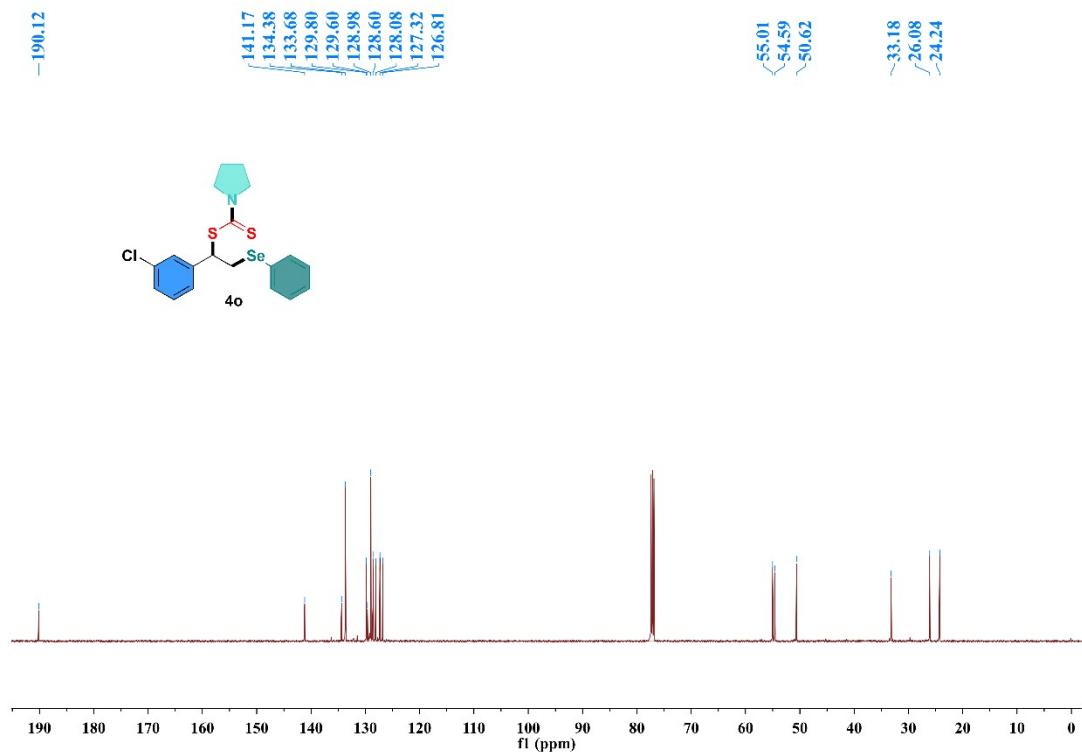
<sup>13</sup>C NMR spectra of compound **4n**



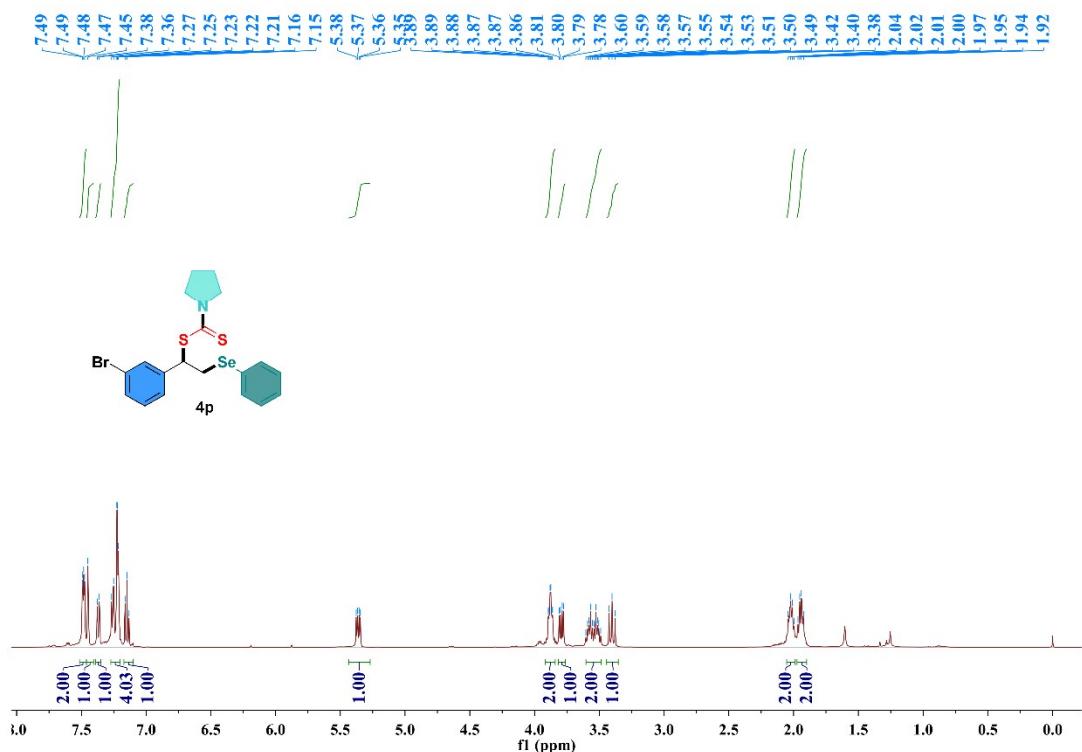
<sup>1</sup>H NMR spectra of compound **4o**



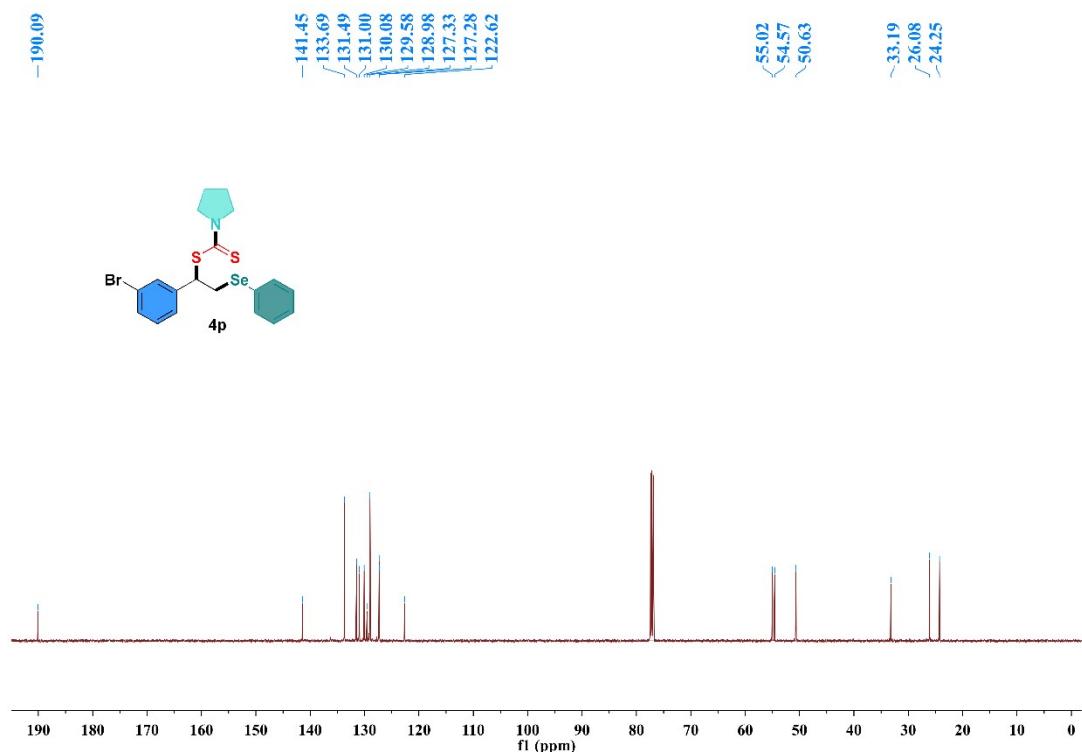
<sup>13</sup>C NMR spectra of compound **4o**



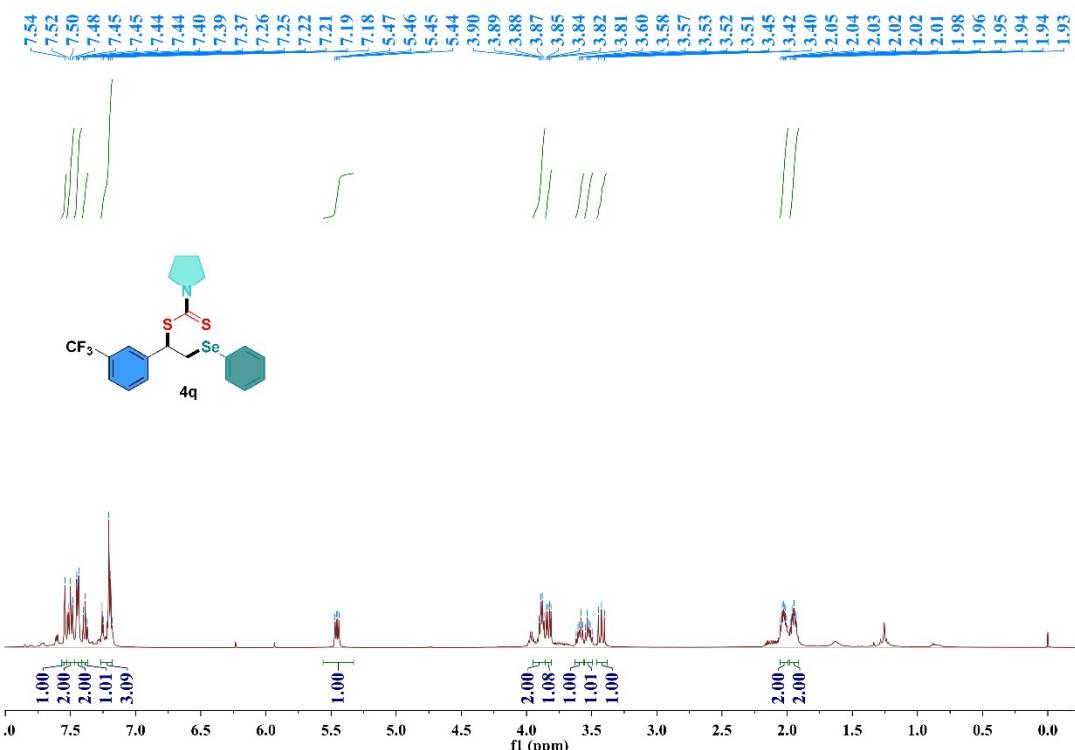
<sup>1</sup>H NMR spectra of compound **4p**



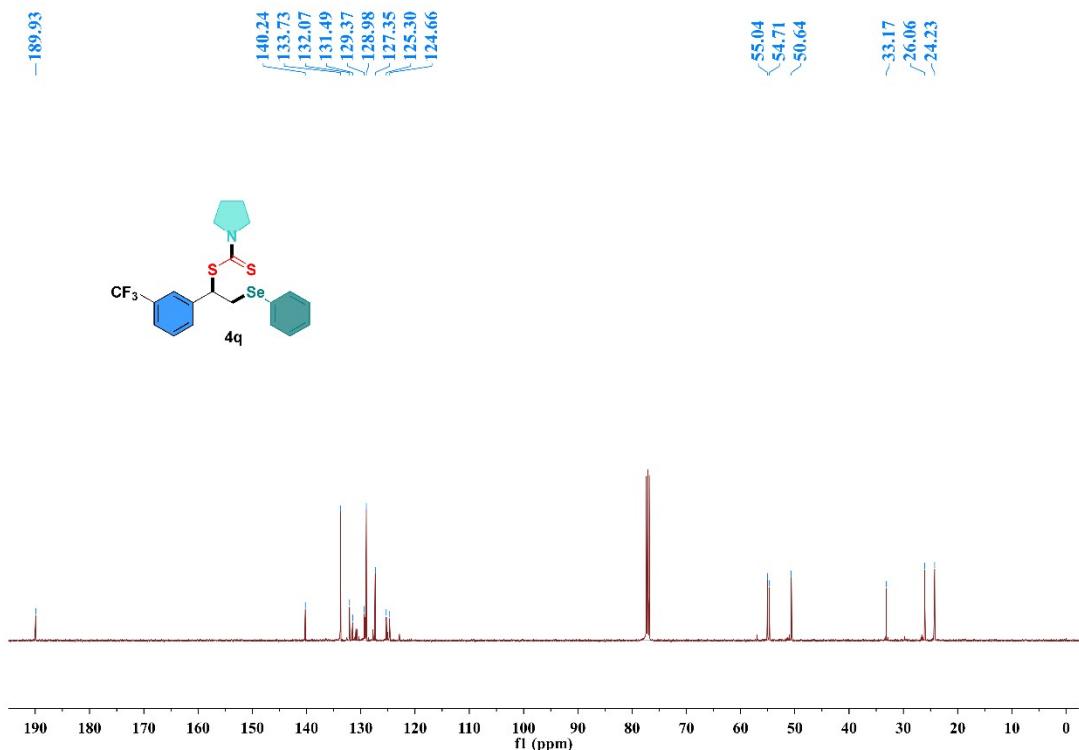
<sup>13</sup>C NMR spectra of compound **4p**



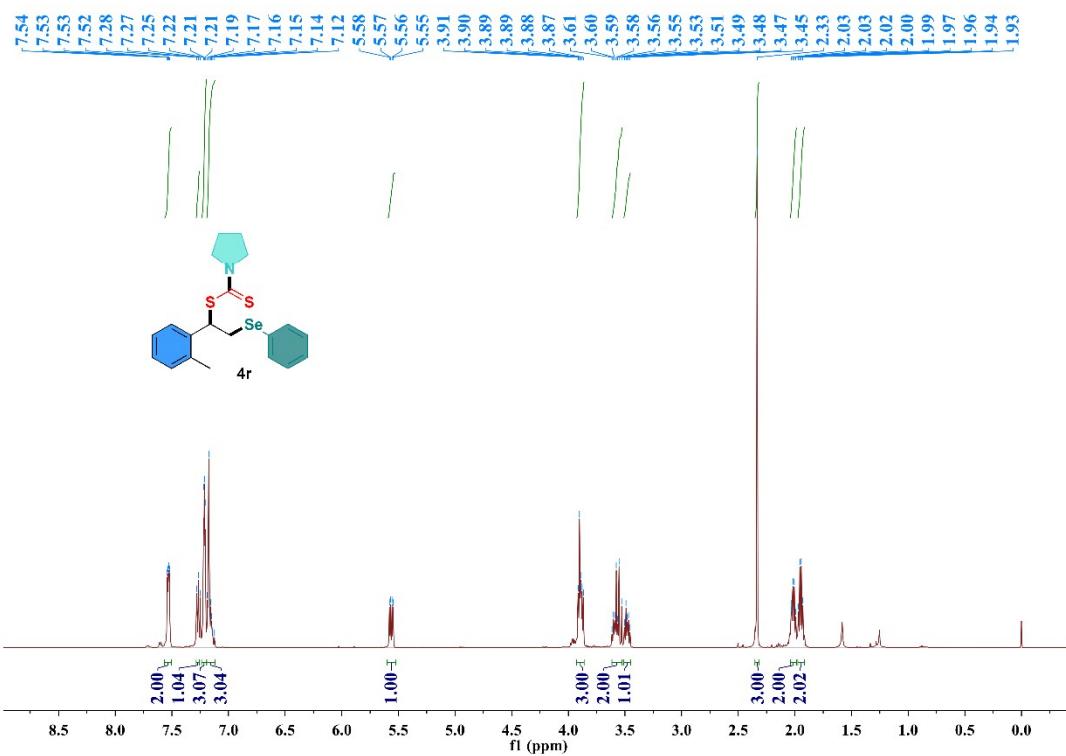
<sup>1</sup>H NMR spectra of compound **4q**



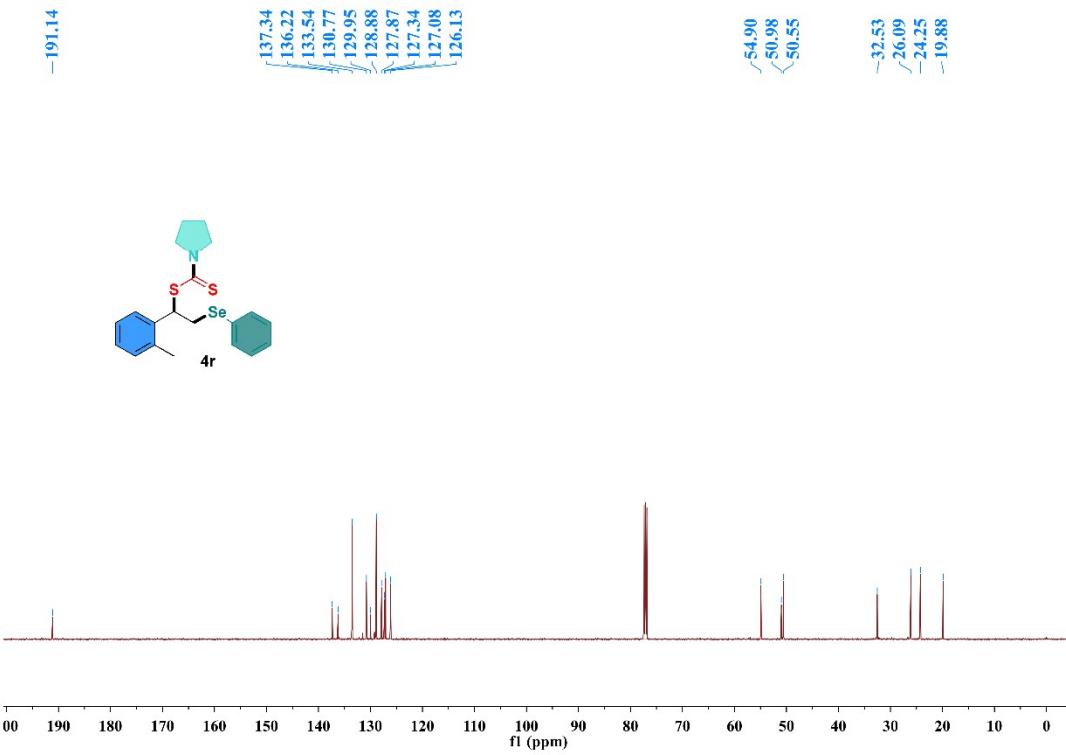
<sup>13</sup>C NMR spectra of compound **4q**



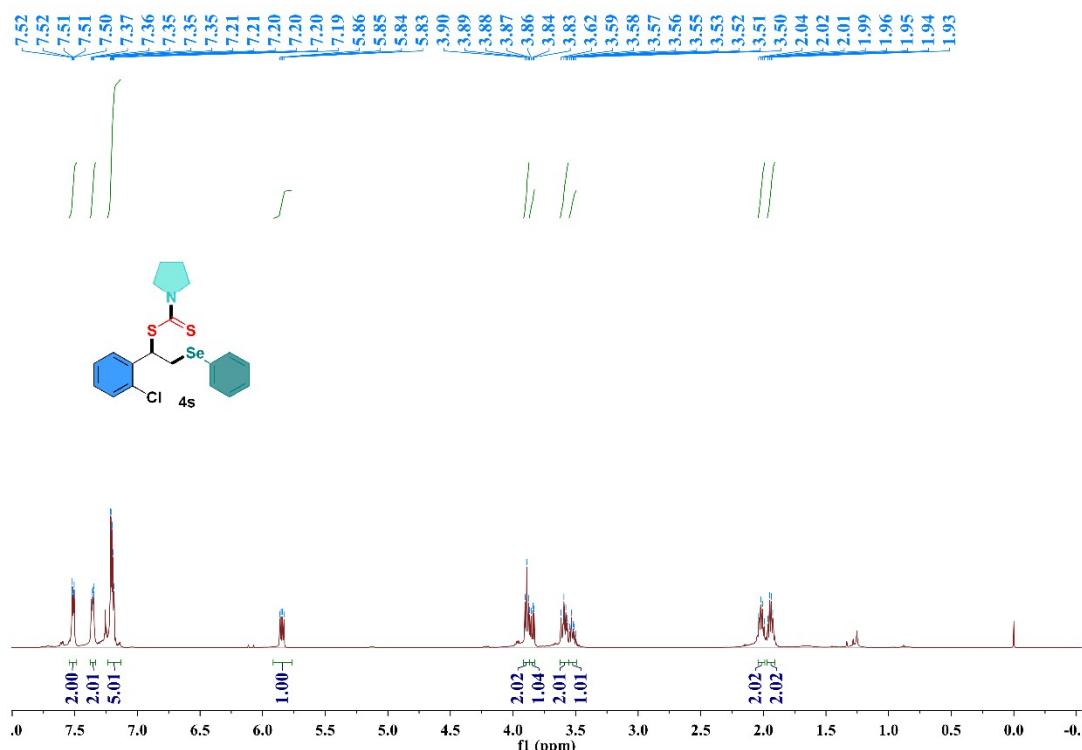
<sup>1</sup>H NMR spectra of compound **4r**



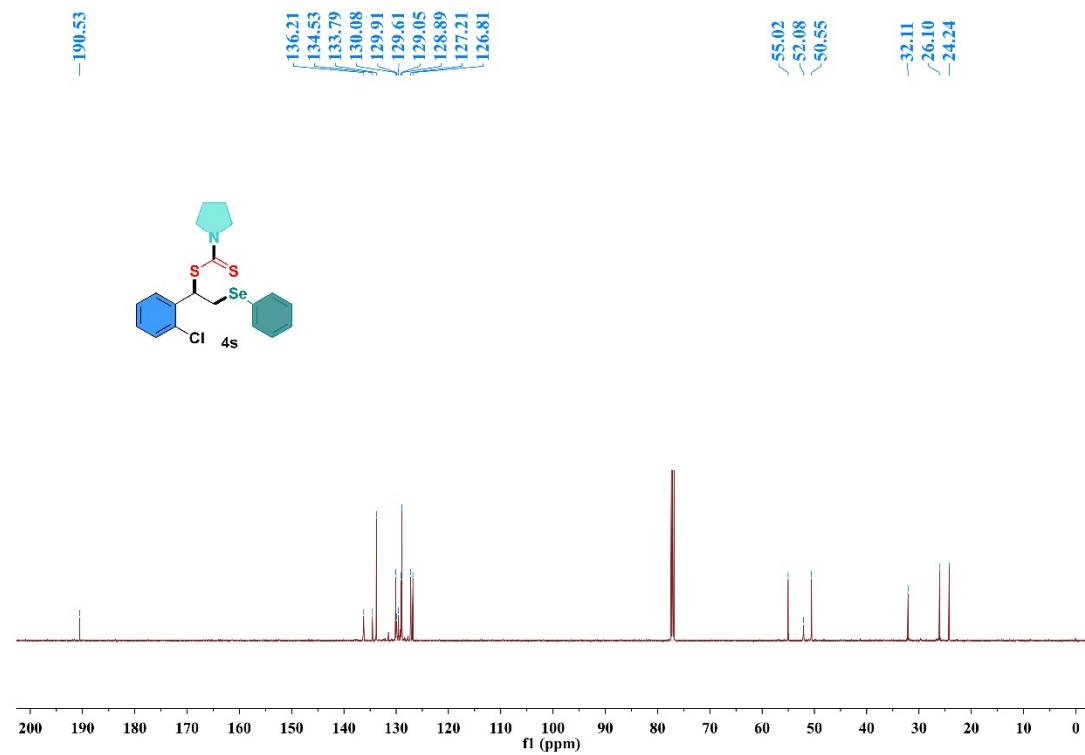
### <sup>13</sup>C NMR spectra of compound 4r



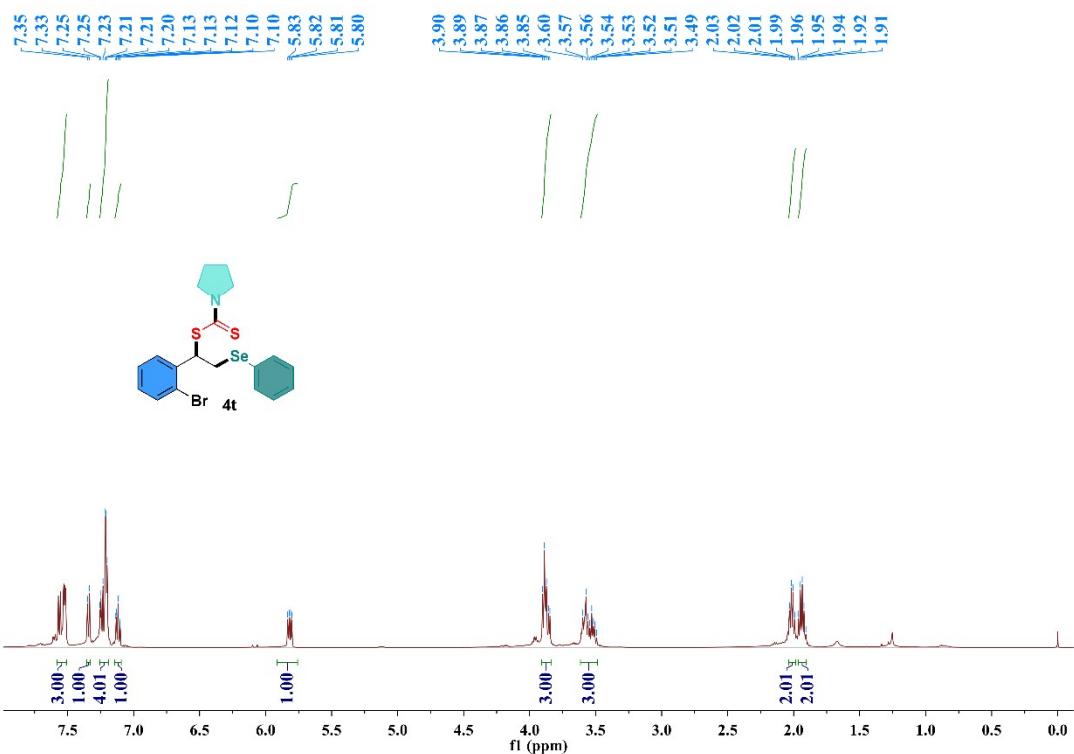
<sup>1</sup>H NMR spectra of compound **4s**



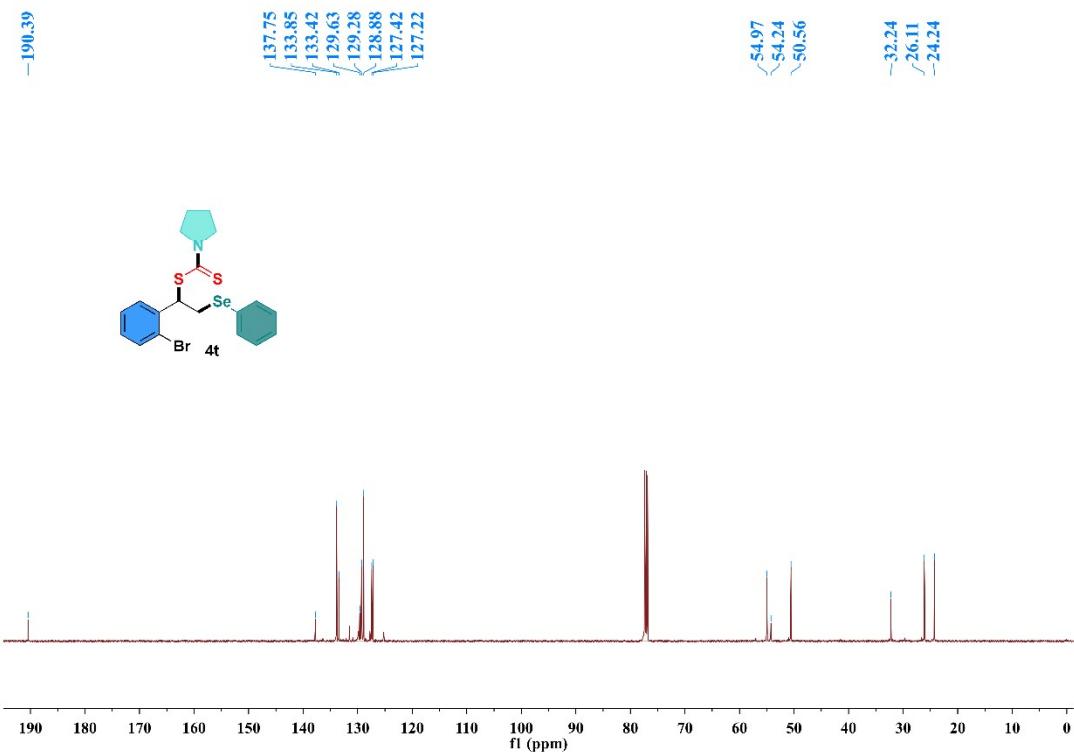
<sup>13</sup>C NMR spectra of compound **4s**



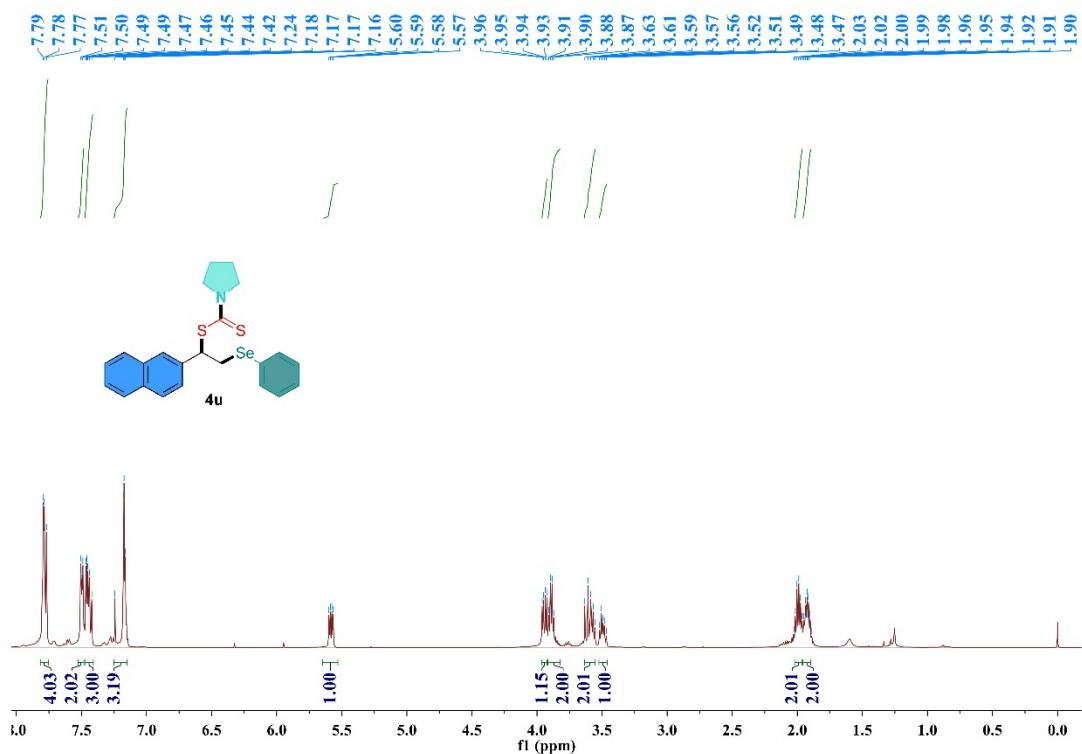
<sup>1</sup>H NMR spectra of compound 4t



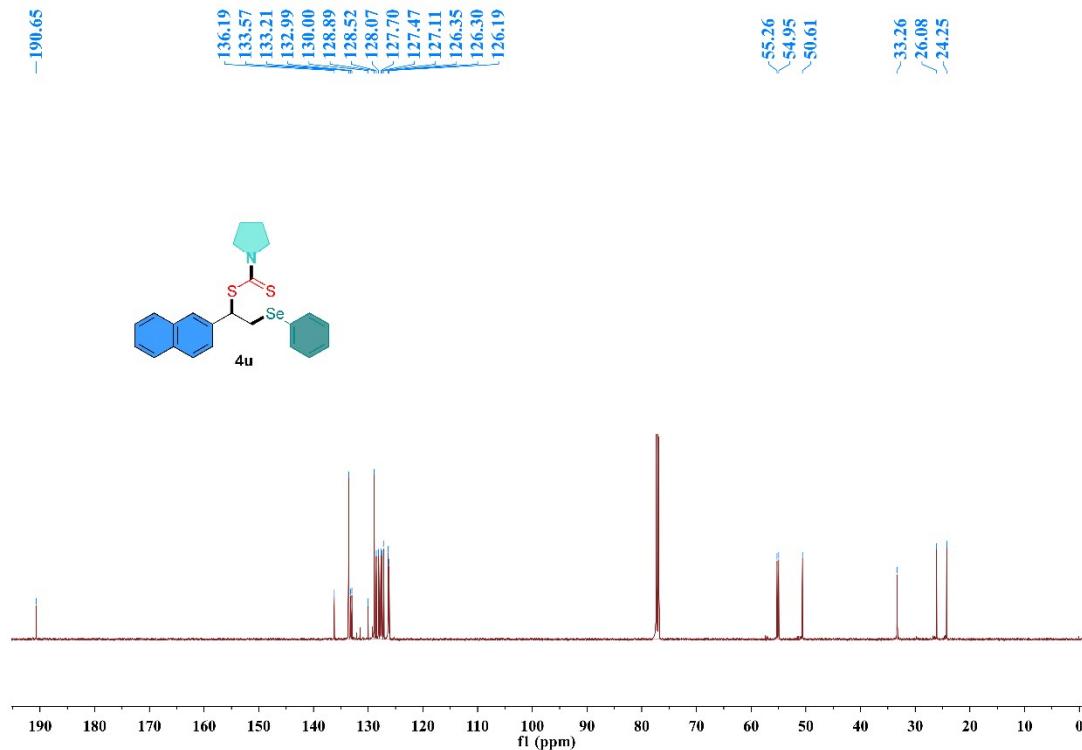
<sup>13</sup>C NMR spectra of compound 4t



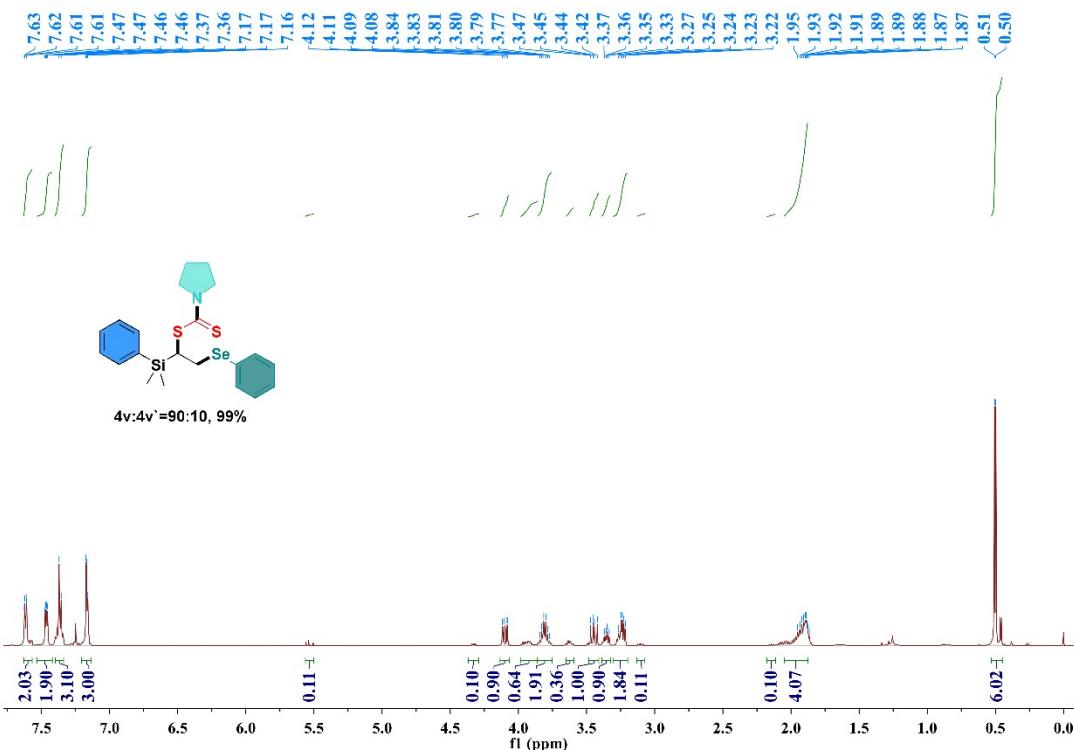
<sup>1</sup>H NMR spectra of compound **4u**



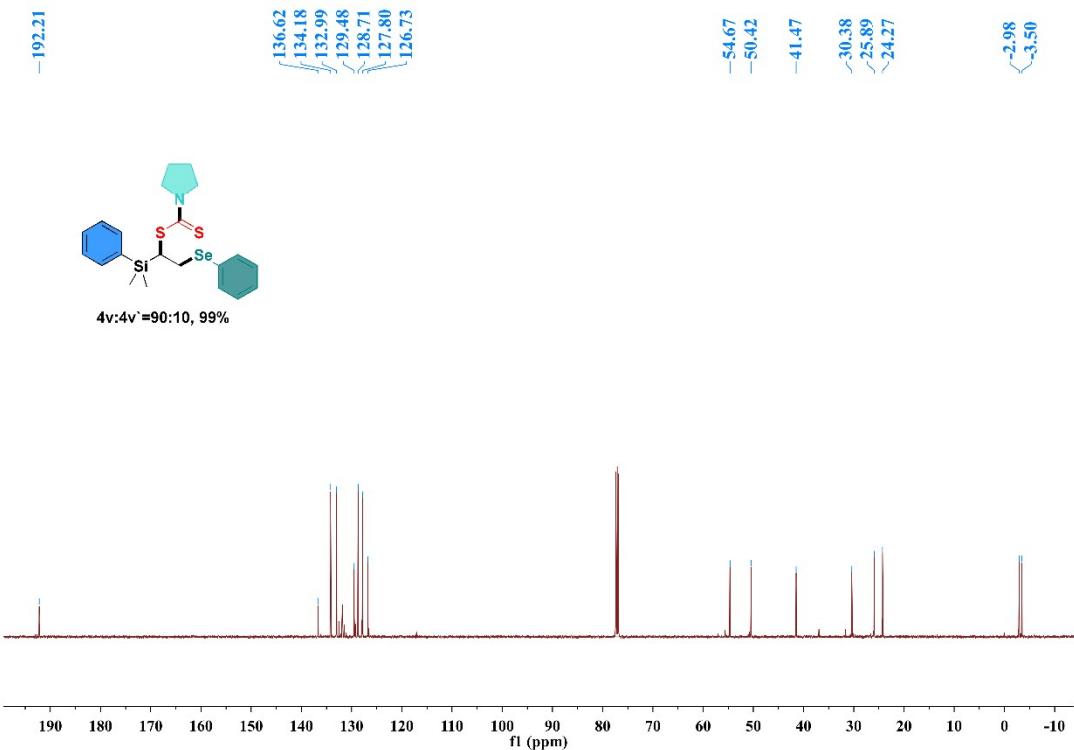
<sup>13</sup>C NMR spectra of compound **4u**



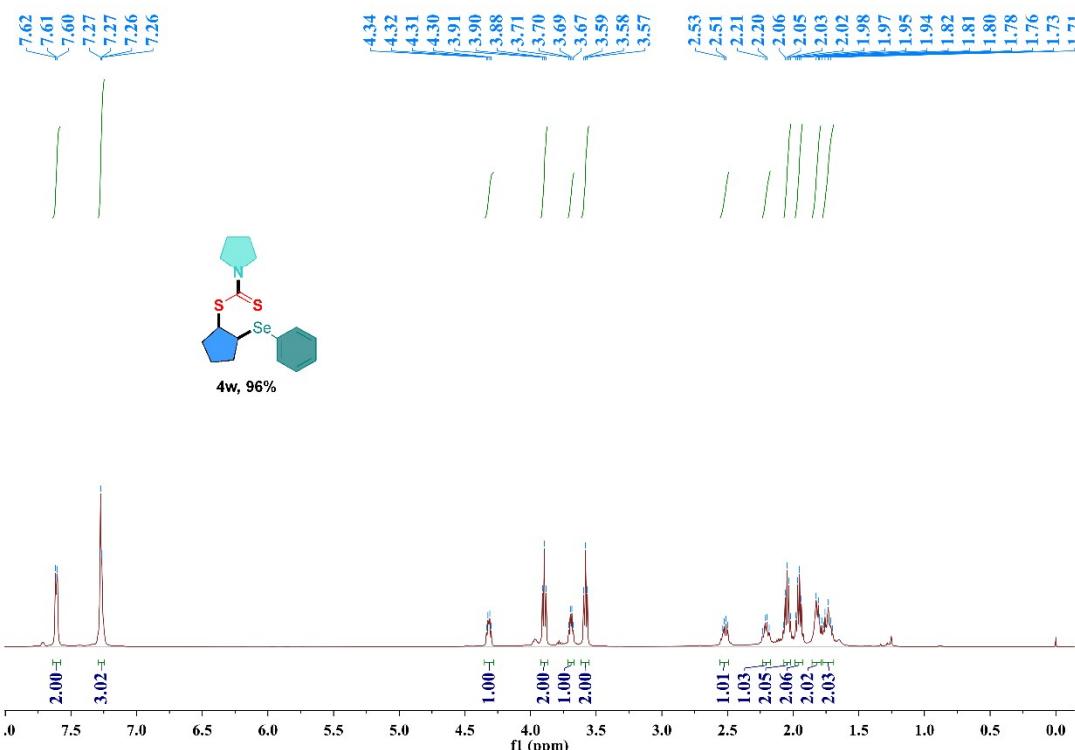
<sup>1</sup>H NMR spectra of compound **4v**



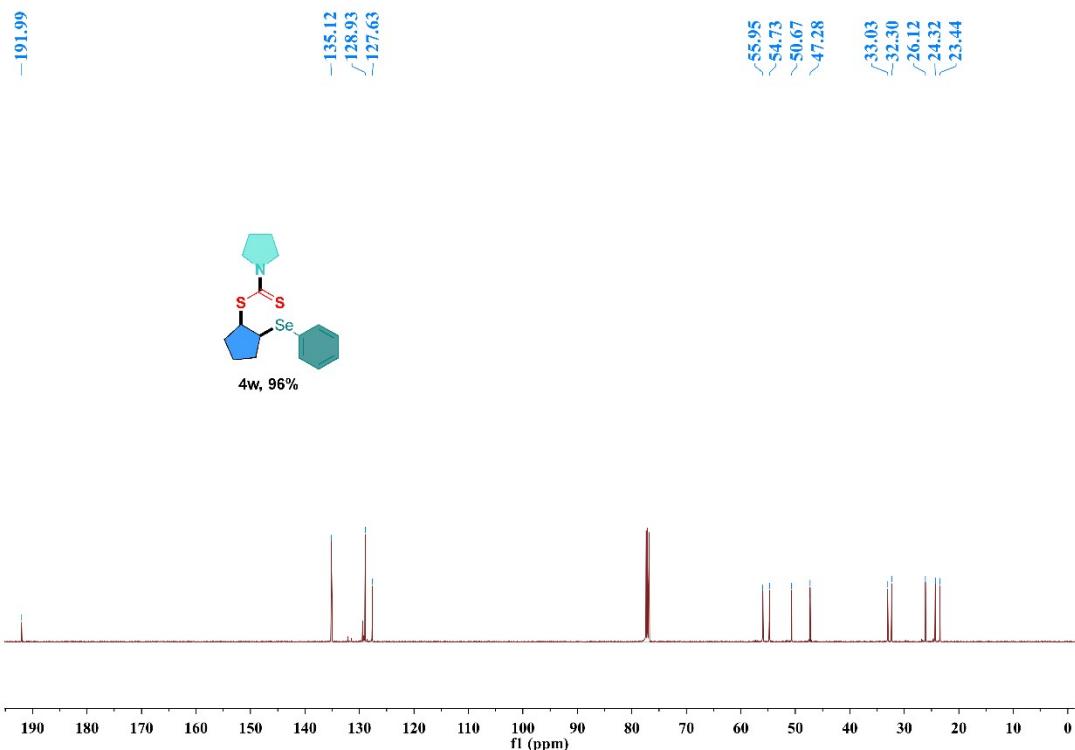
<sup>13</sup>C NMR spectra of compound **4v**



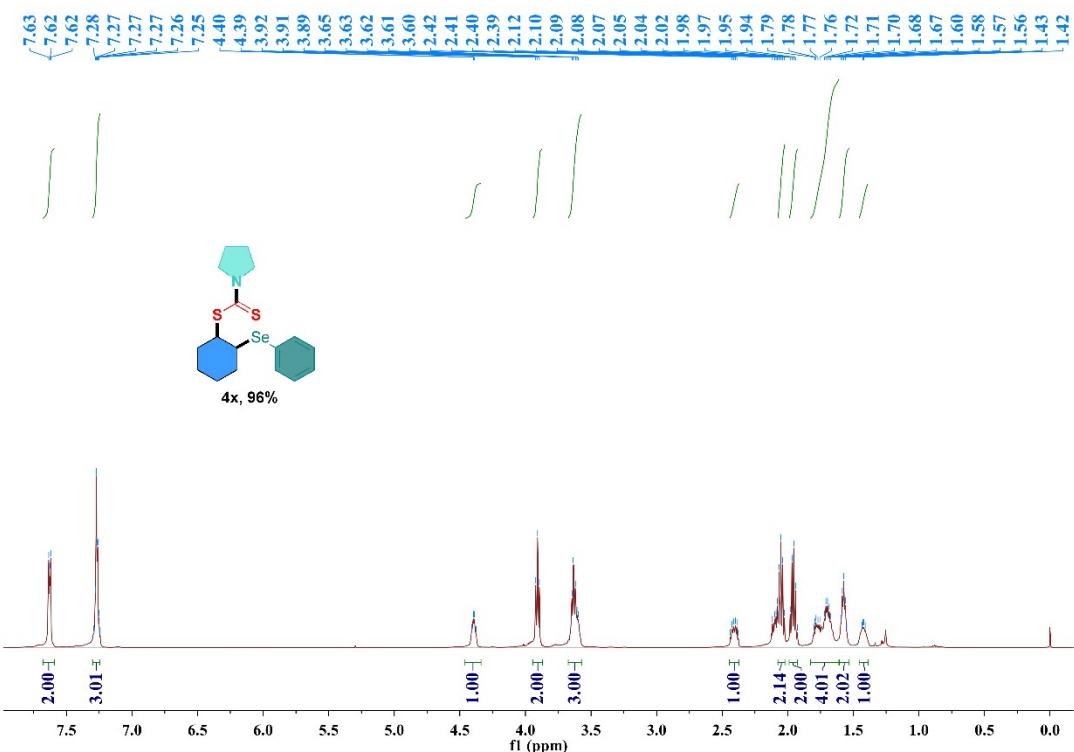
<sup>1</sup>H NMR spectra of compound 4w



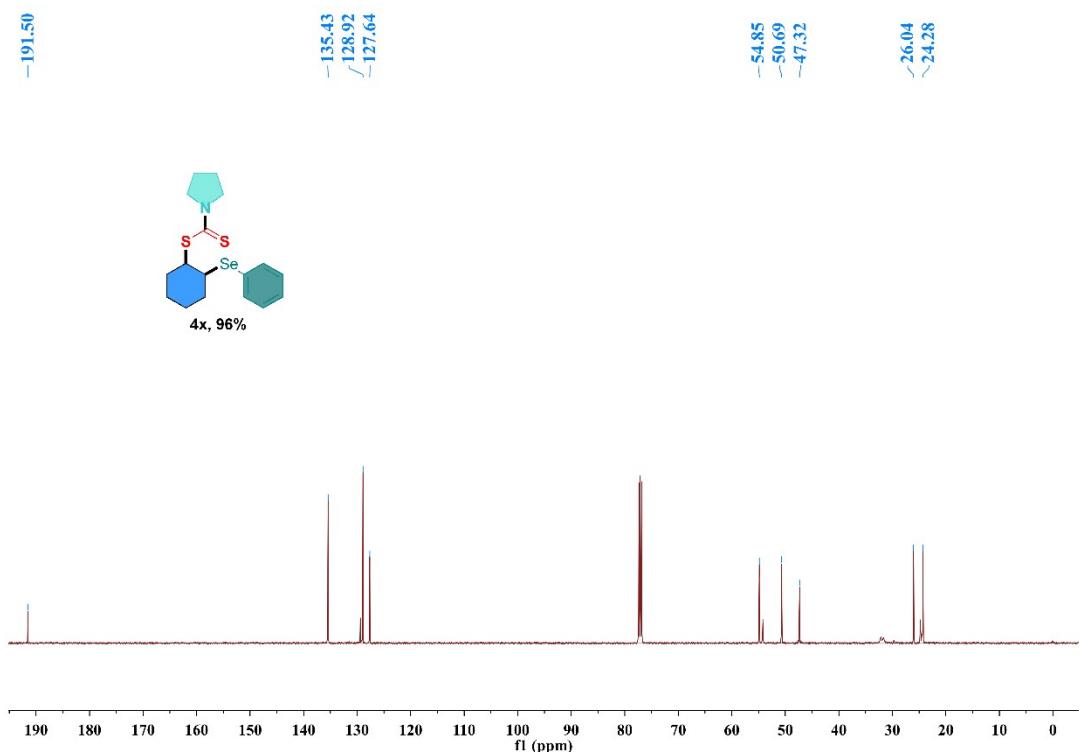
<sup>13</sup>C NMR spectra of compound 4w



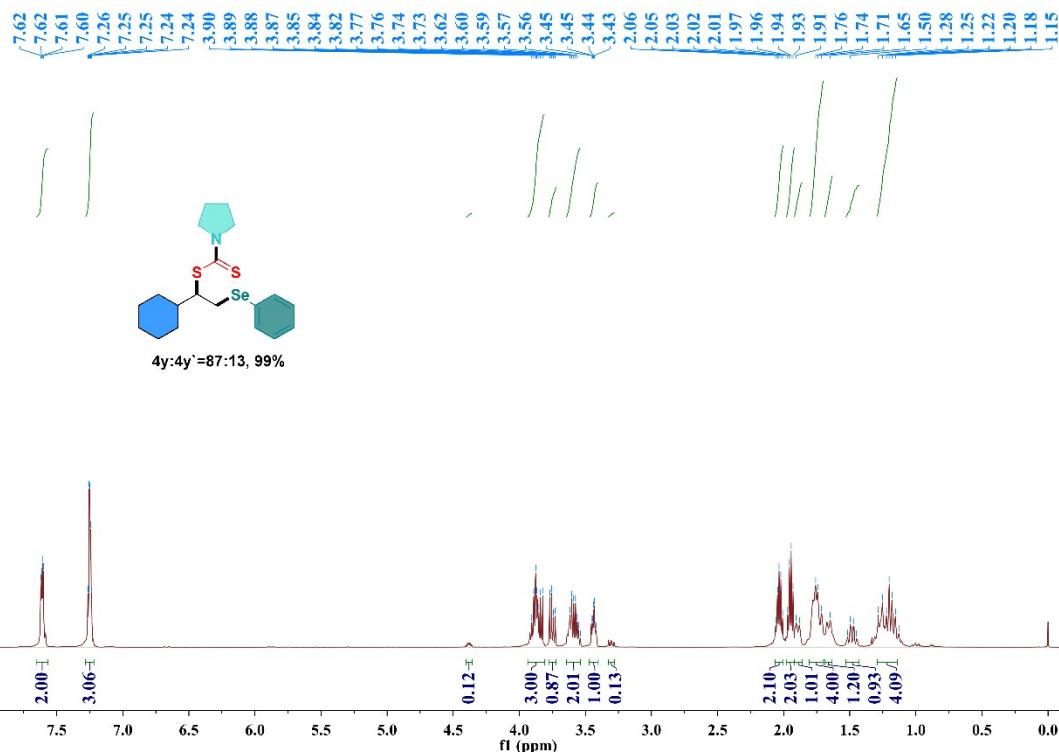
<sup>1</sup>H NMR spectra of compound **4x**



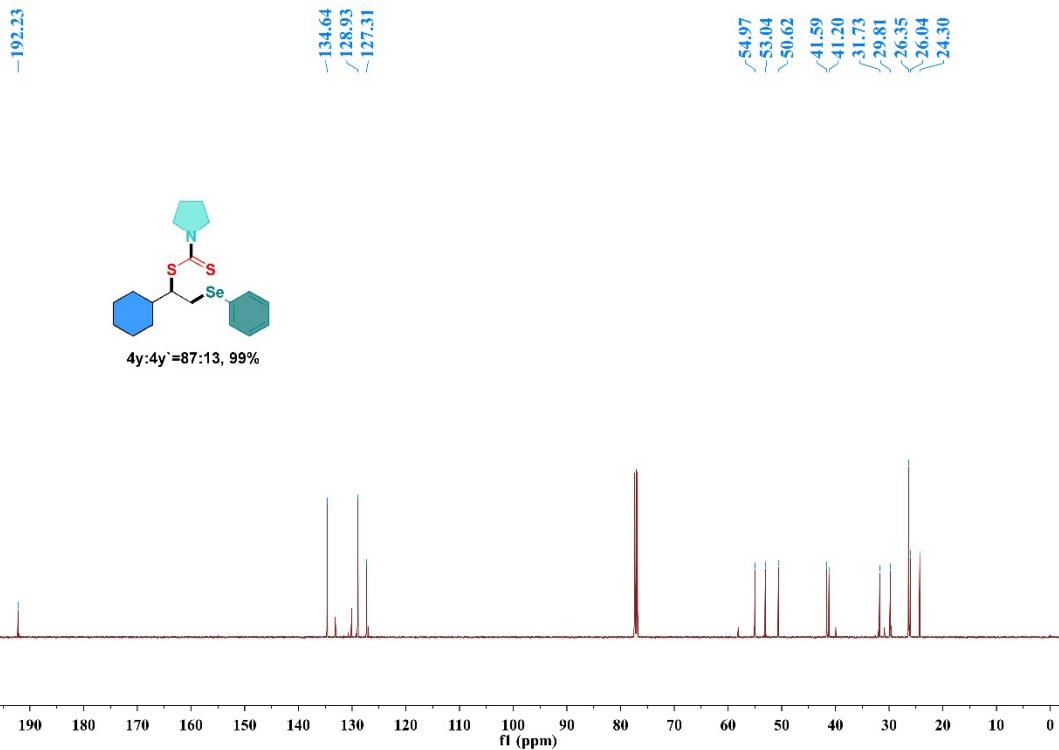
<sup>13</sup>C NMR spectra of compound **4x**



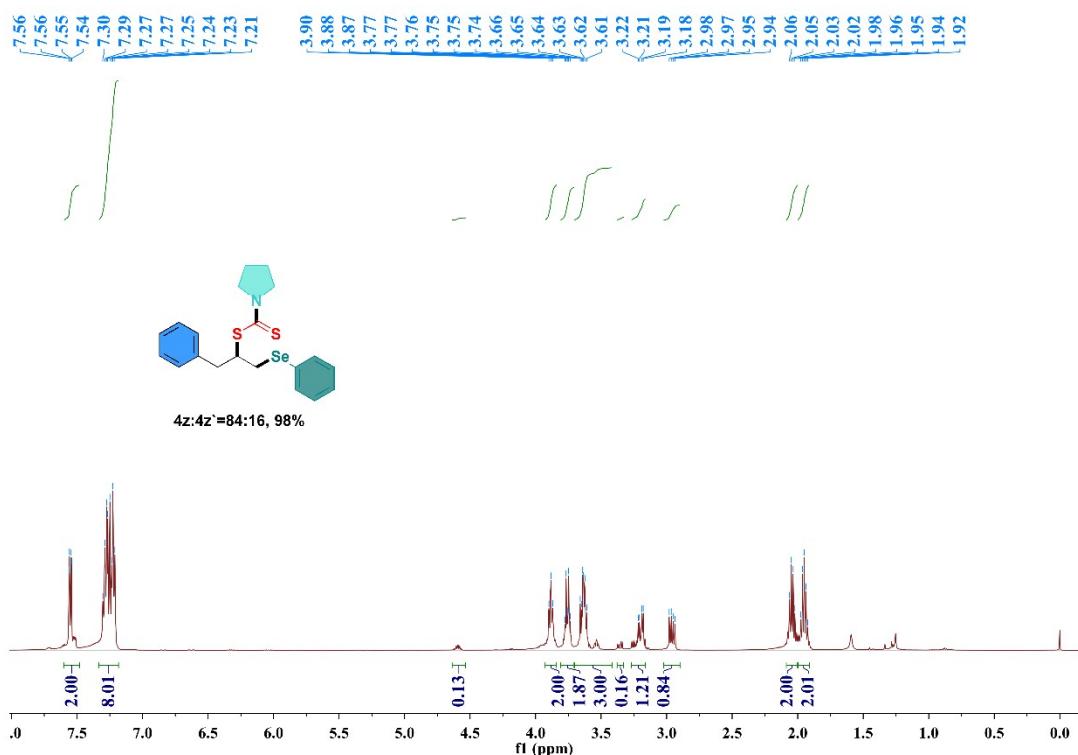
<sup>1</sup>H NMR spectra of compound 4y



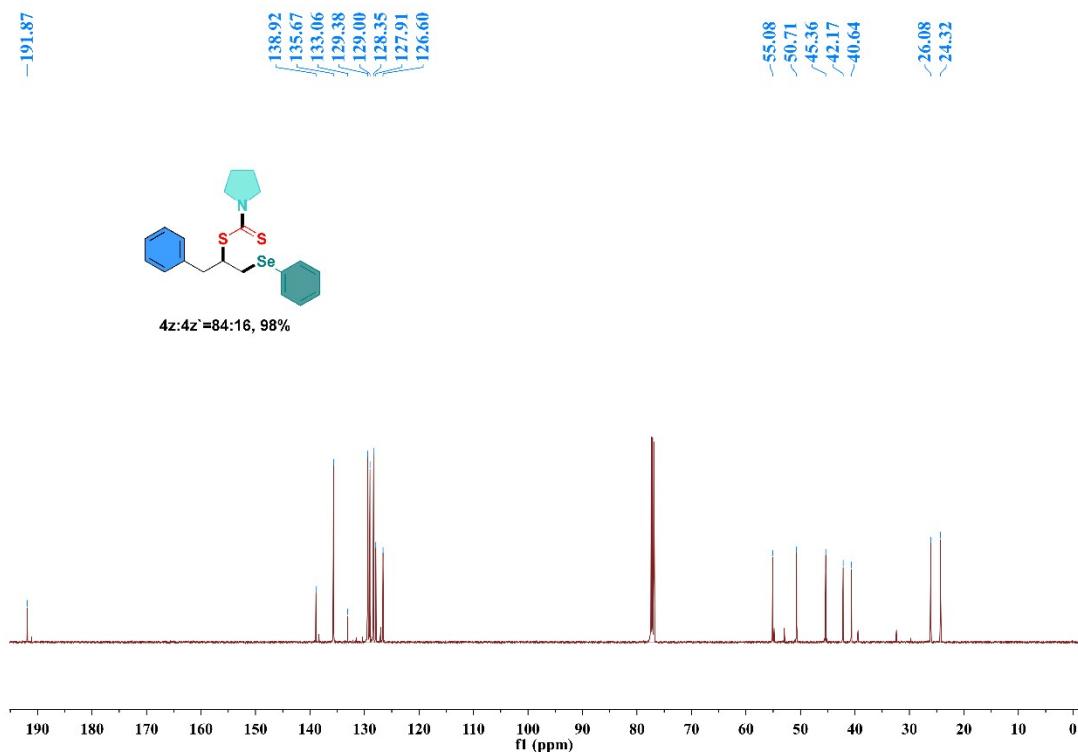
<sup>13</sup>C NMR spectra of compound 4y



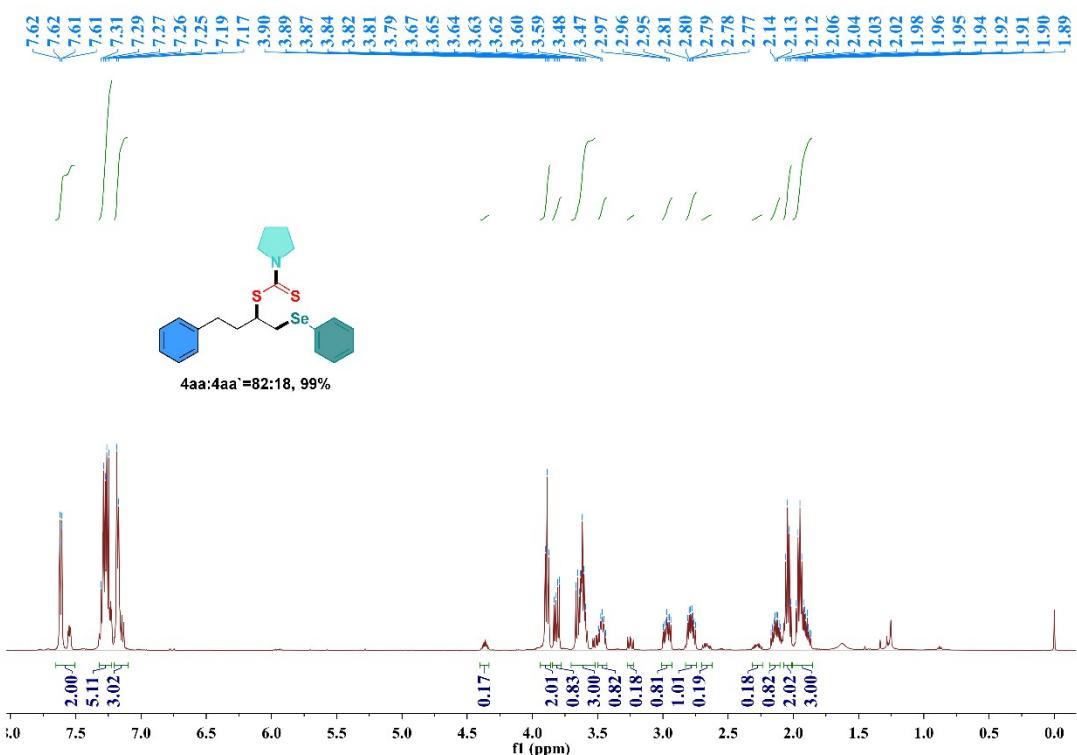
<sup>1</sup>H NMR spectra of compound **4z**



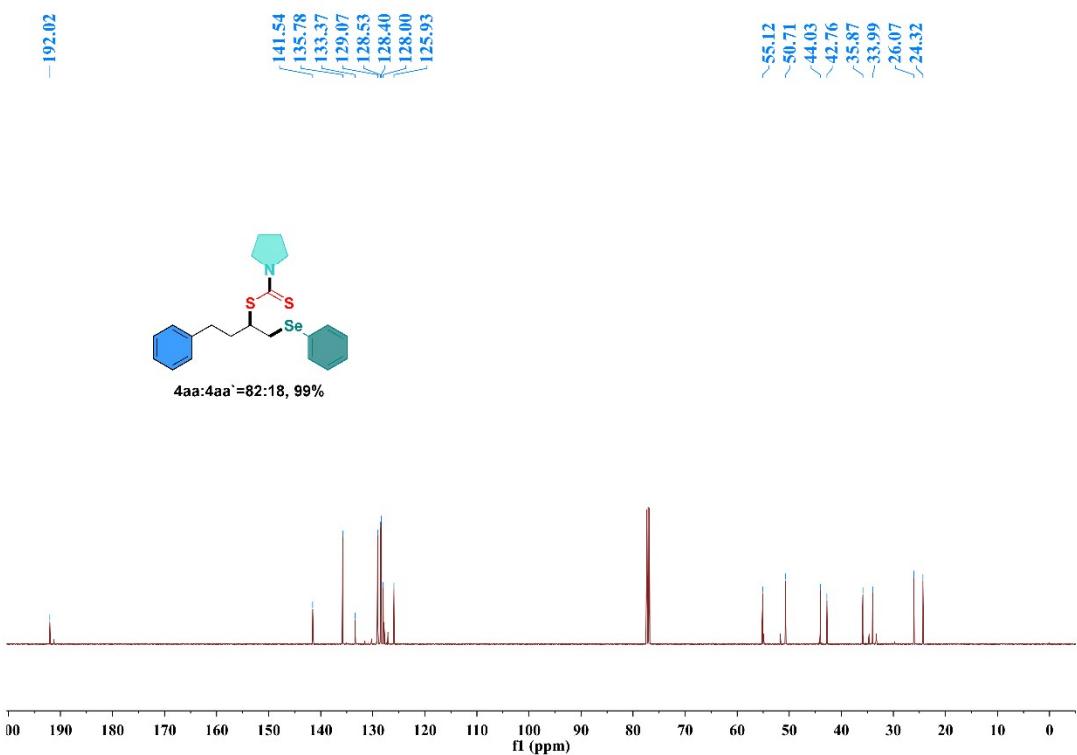
<sup>13</sup>C NMR spectra of compound **4z**



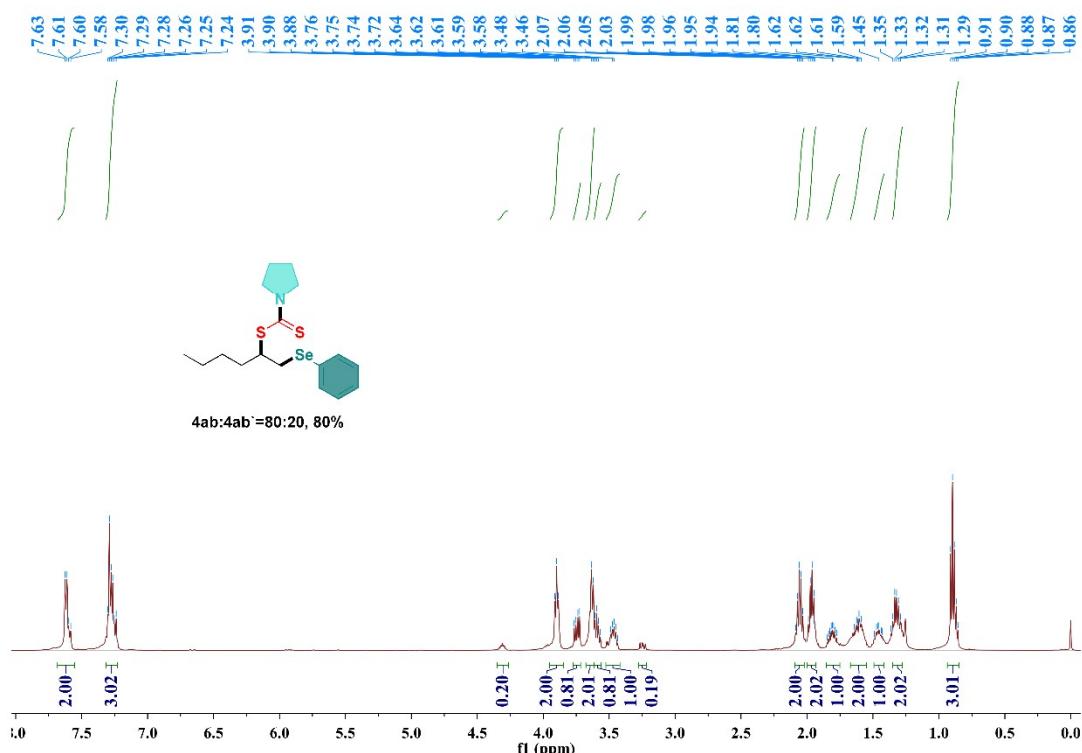
<sup>1</sup>H NMR spectra of compound 4aa



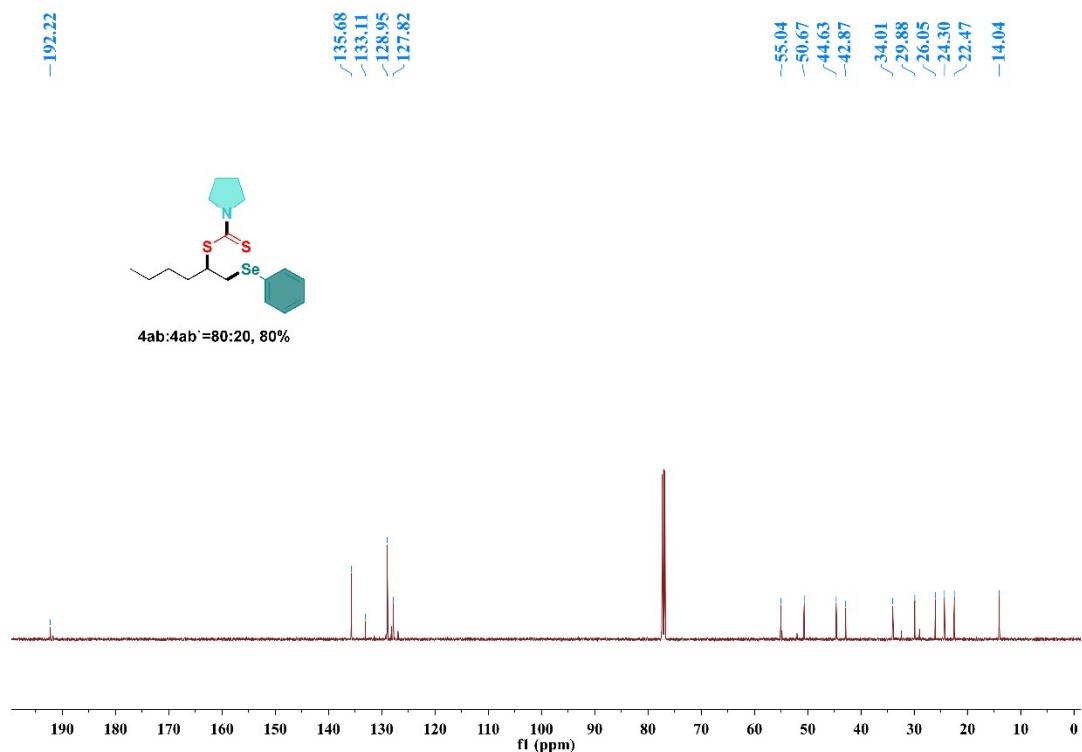
<sup>13</sup>C NMR spectra of compound 4aa



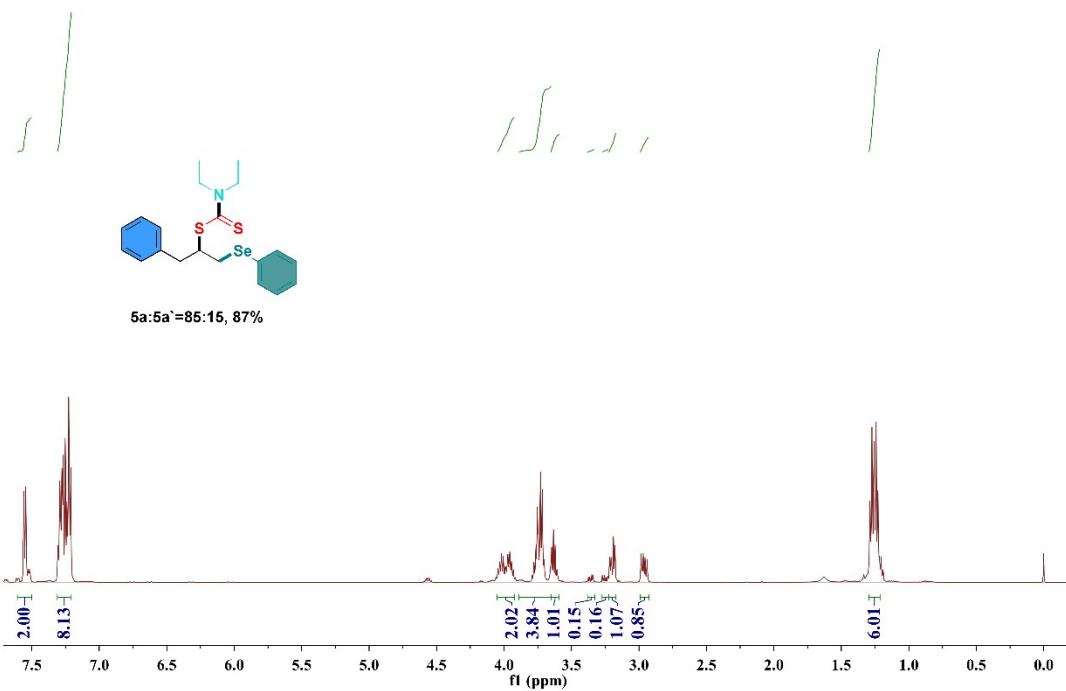
<sup>1</sup>H NMR spectra of compound **4ab**



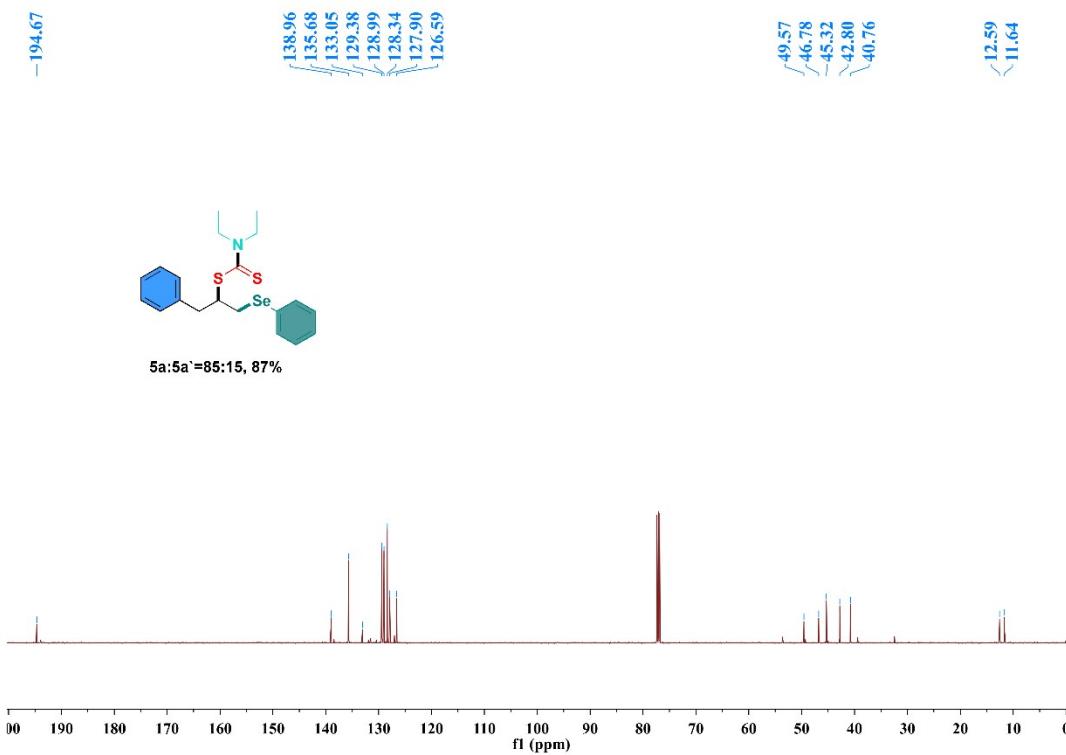
<sup>13</sup>C NMR spectra of compound **4ab**



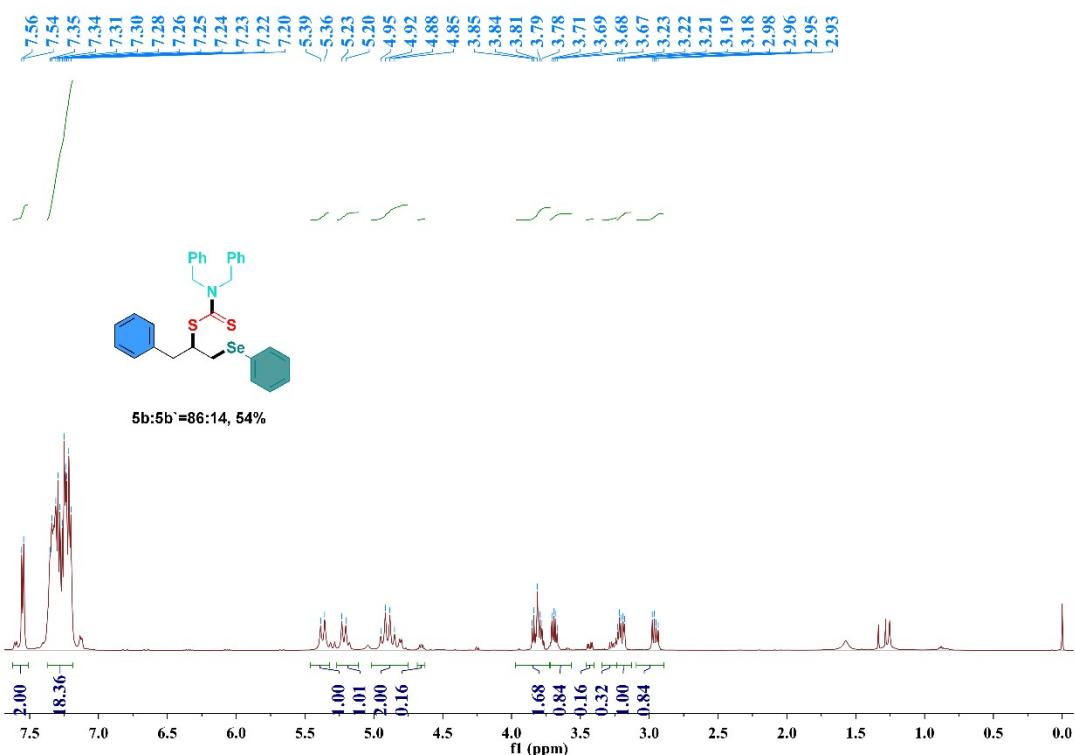
<sup>1</sup>H NMR spectra of compound **5a**



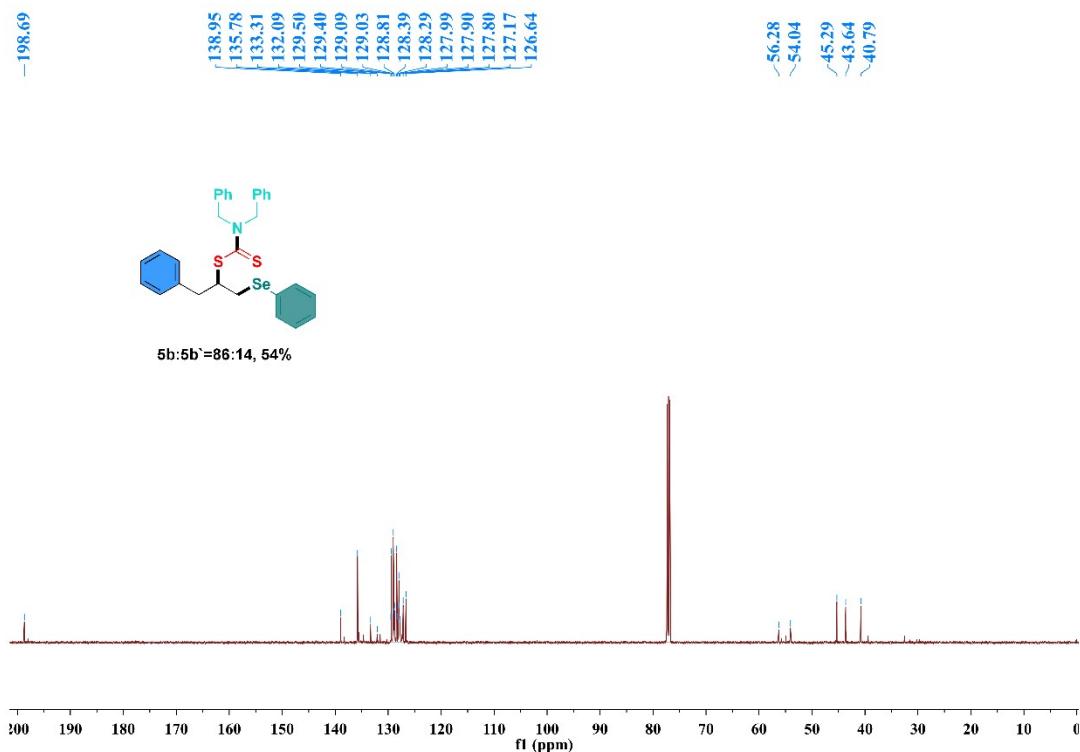
<sup>13</sup>C NMR spectra of compound **5a**



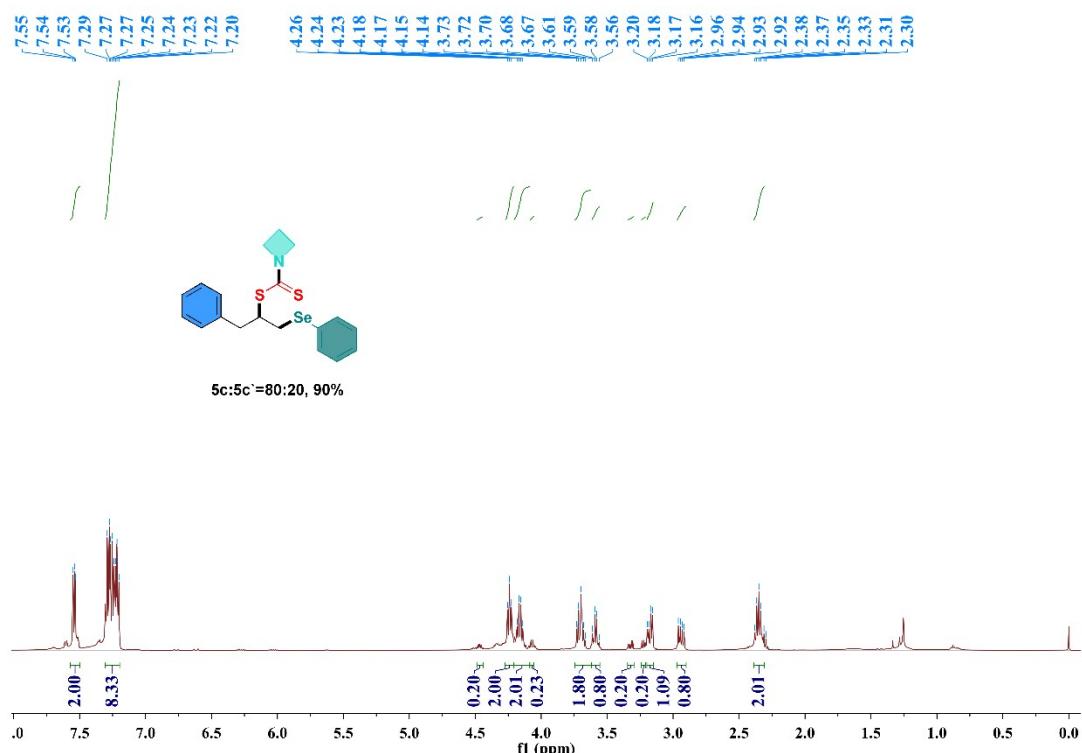
<sup>1</sup>H NMR spectra of compound **5b**



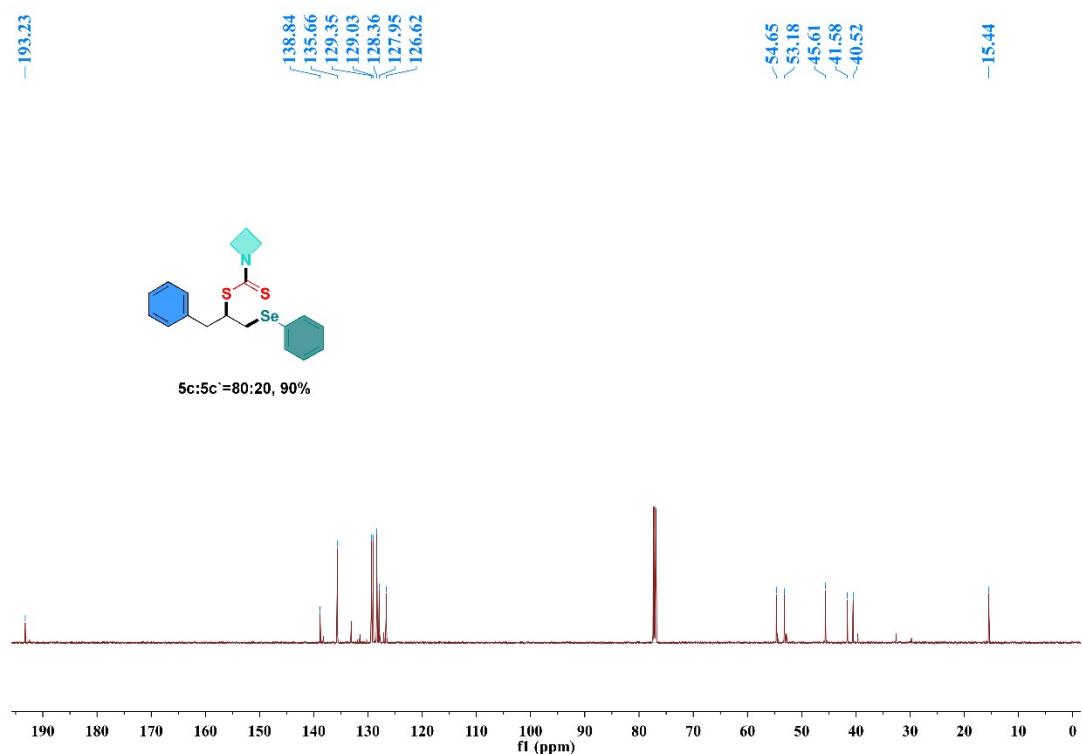
<sup>13</sup>C NMR spectra of compound **5b**



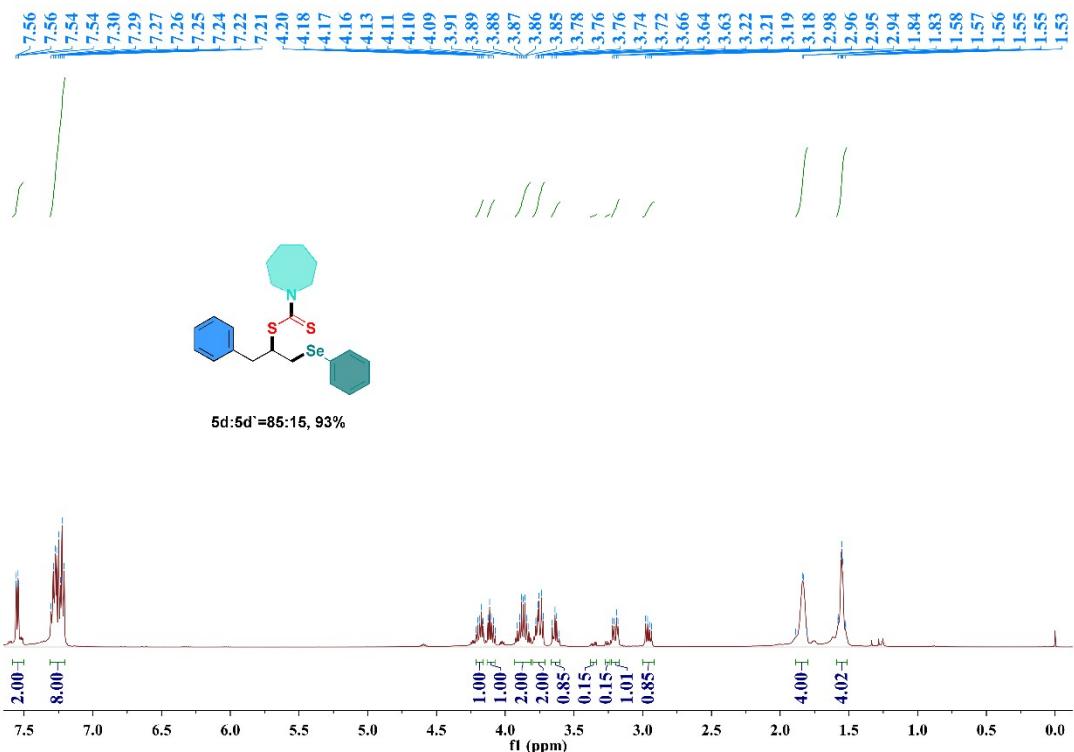
<sup>1</sup>H NMR spectra of compound **5c**



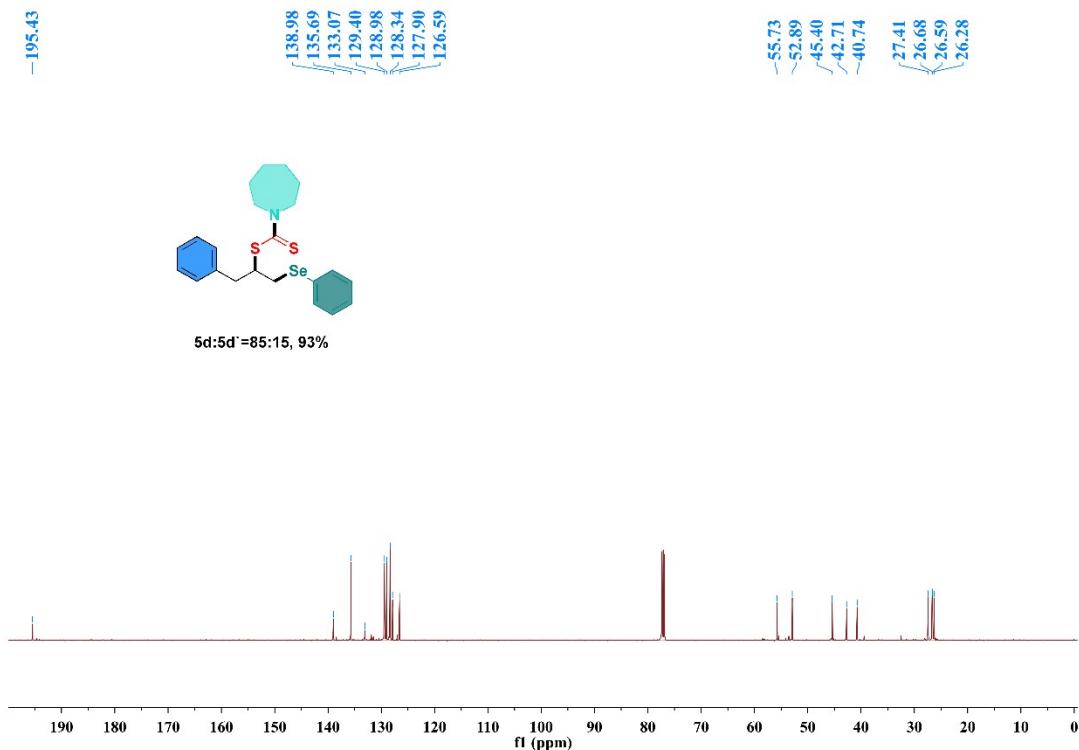
<sup>13</sup>C NMR spectra of compound **5c**



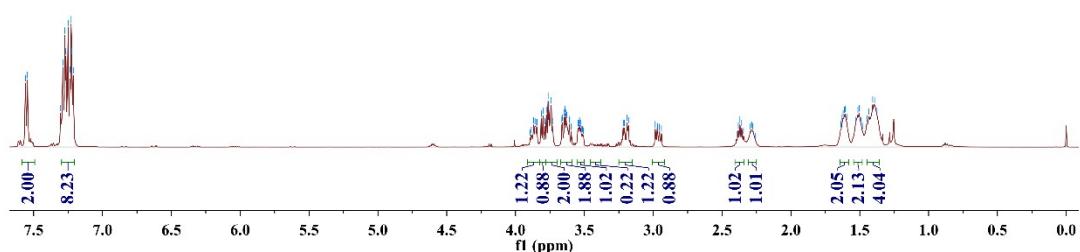
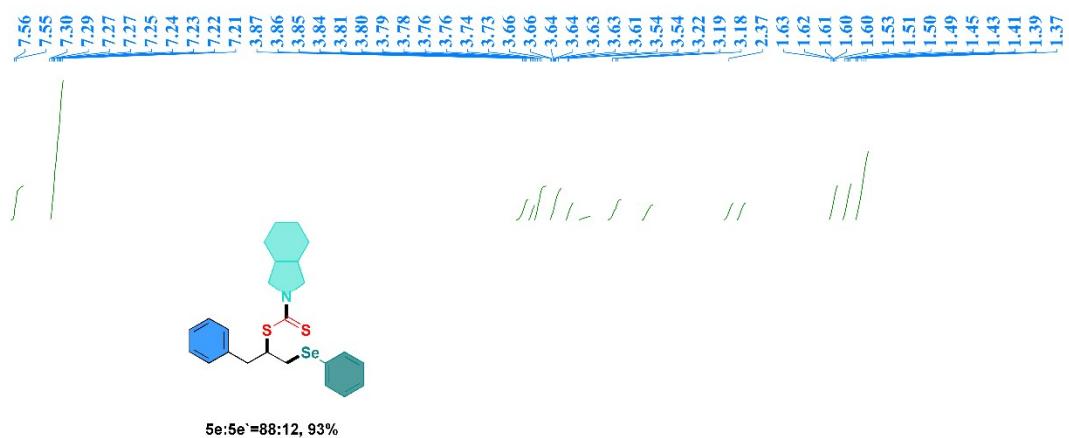
<sup>1</sup>H NMR spectra of compound **5d**



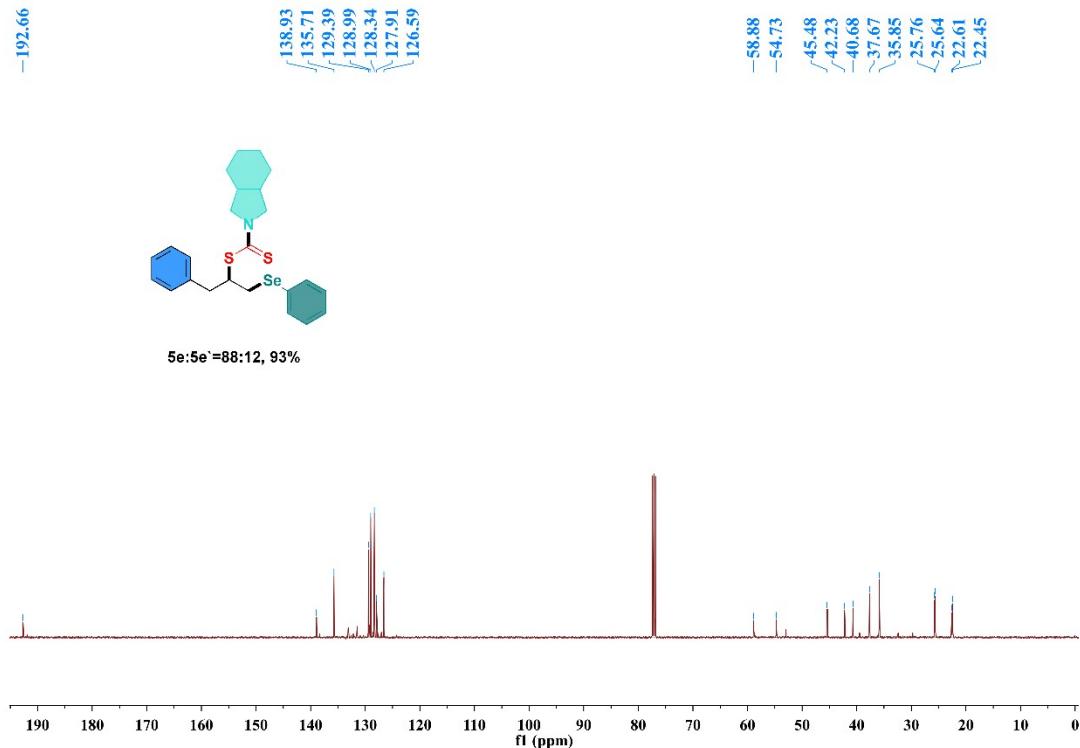
<sup>15</sup>C NMR spectra of compound **5d**



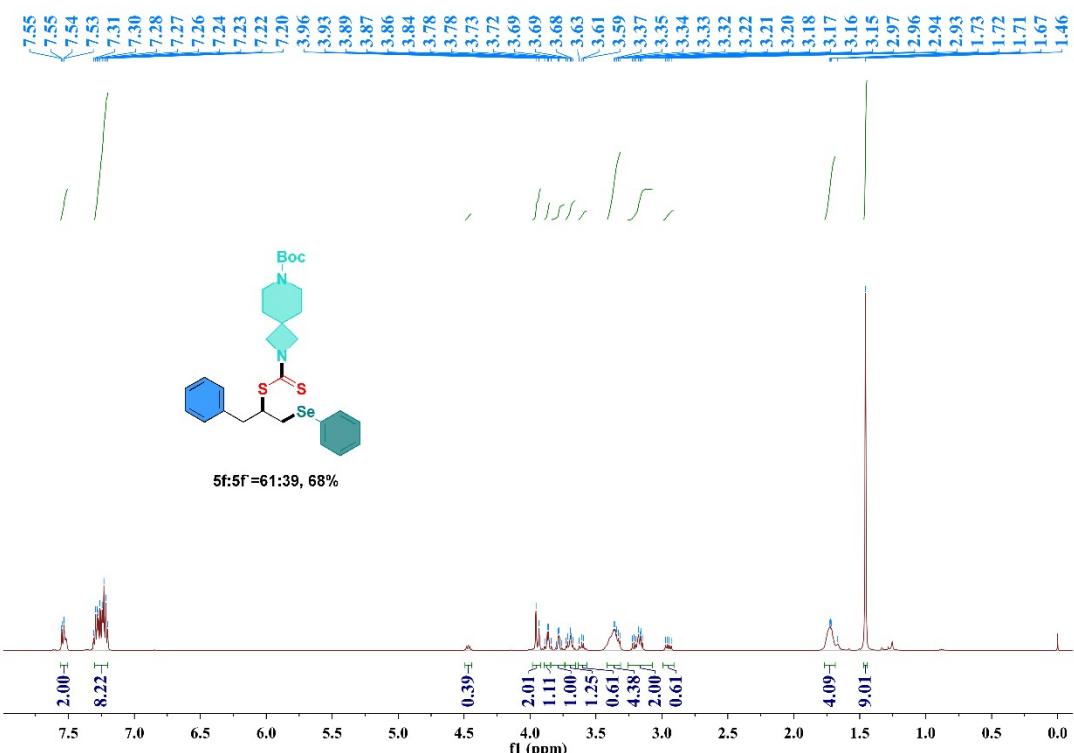
<sup>1</sup>H NMR spectra of compound **5e**



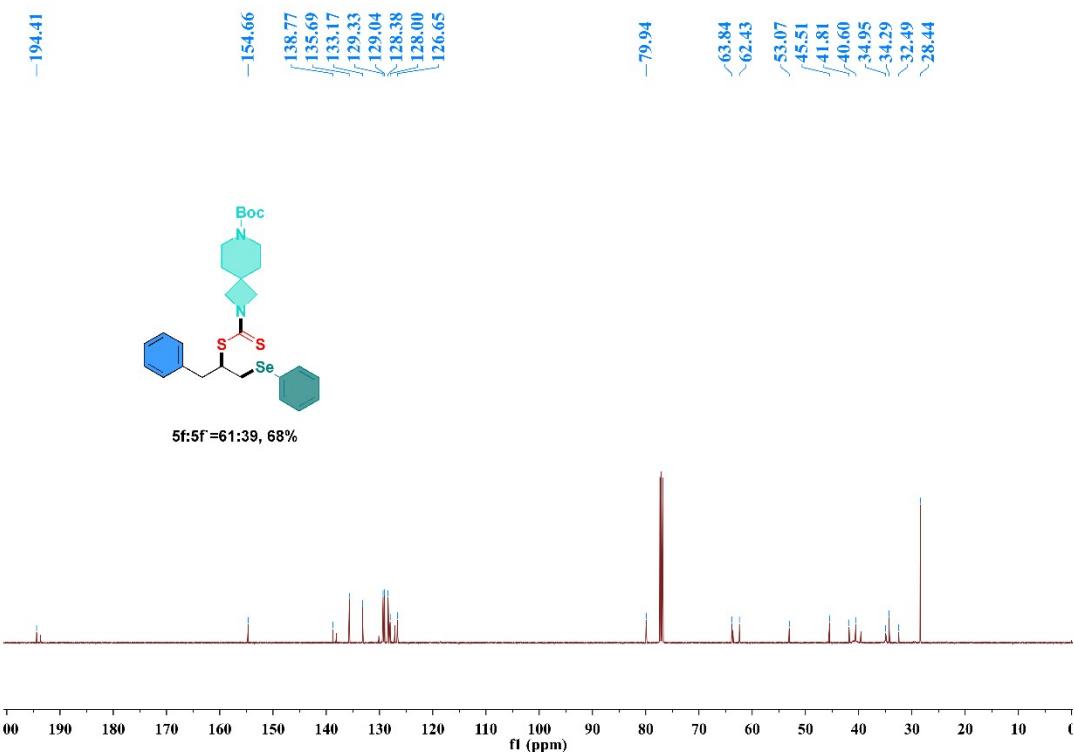
### <sup>13</sup>C NMR spectra of compound **5e**



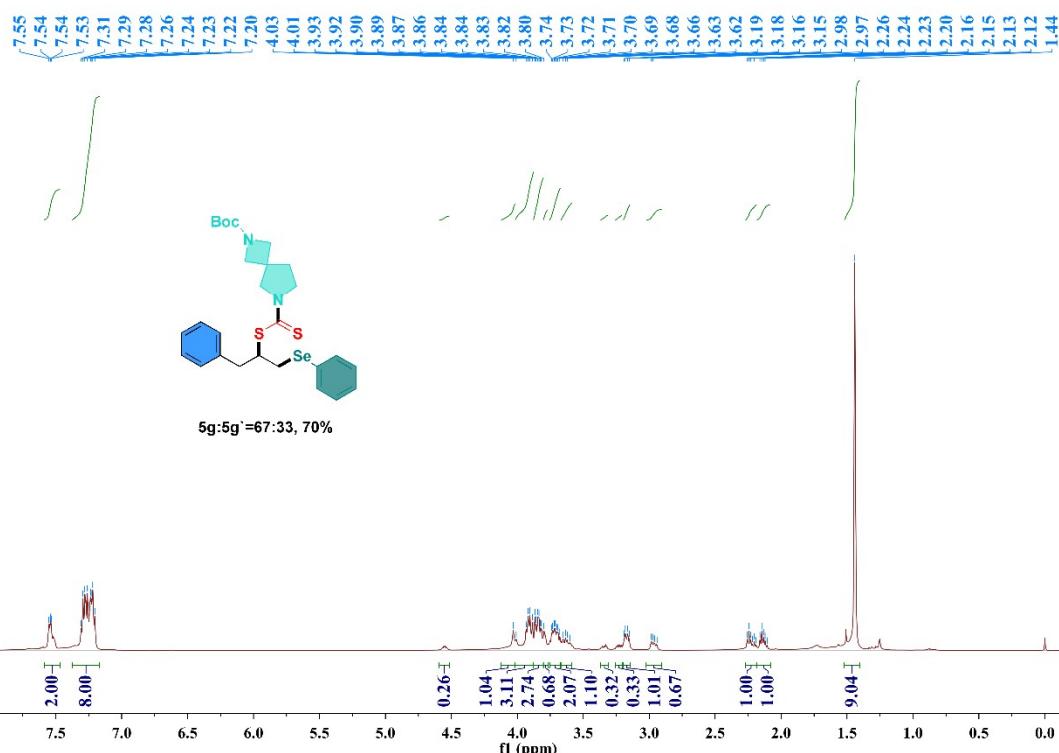
<sup>1</sup>H NMR spectra of compound **5f**



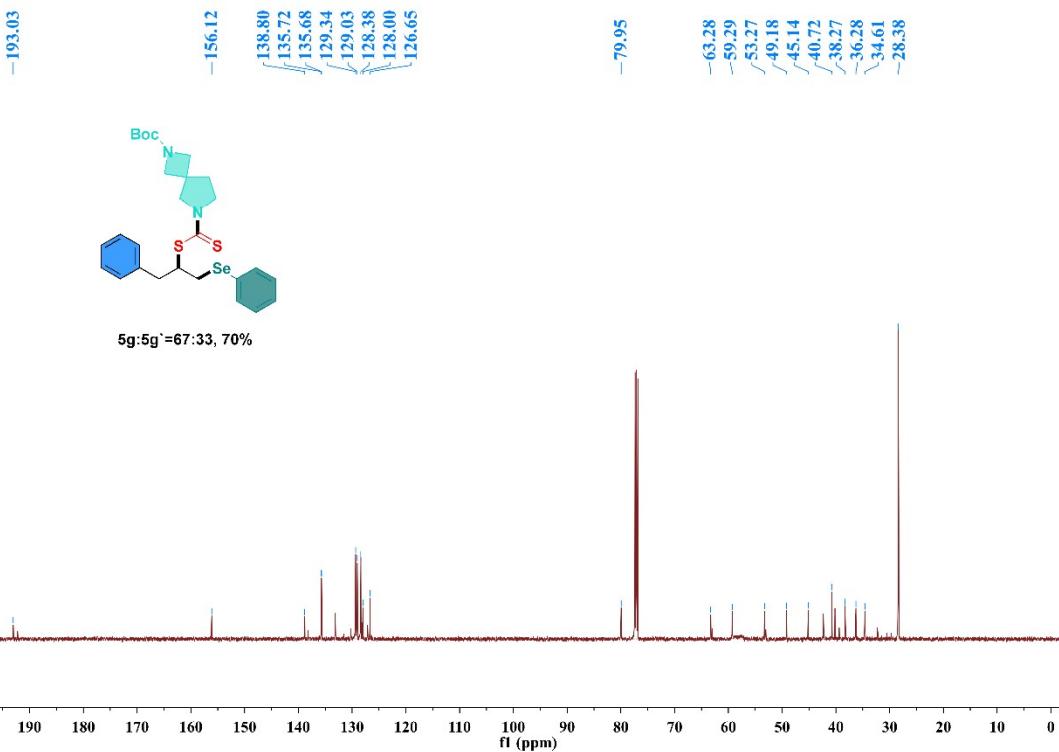
<sup>13</sup>C NMR spectra of compound **5f**



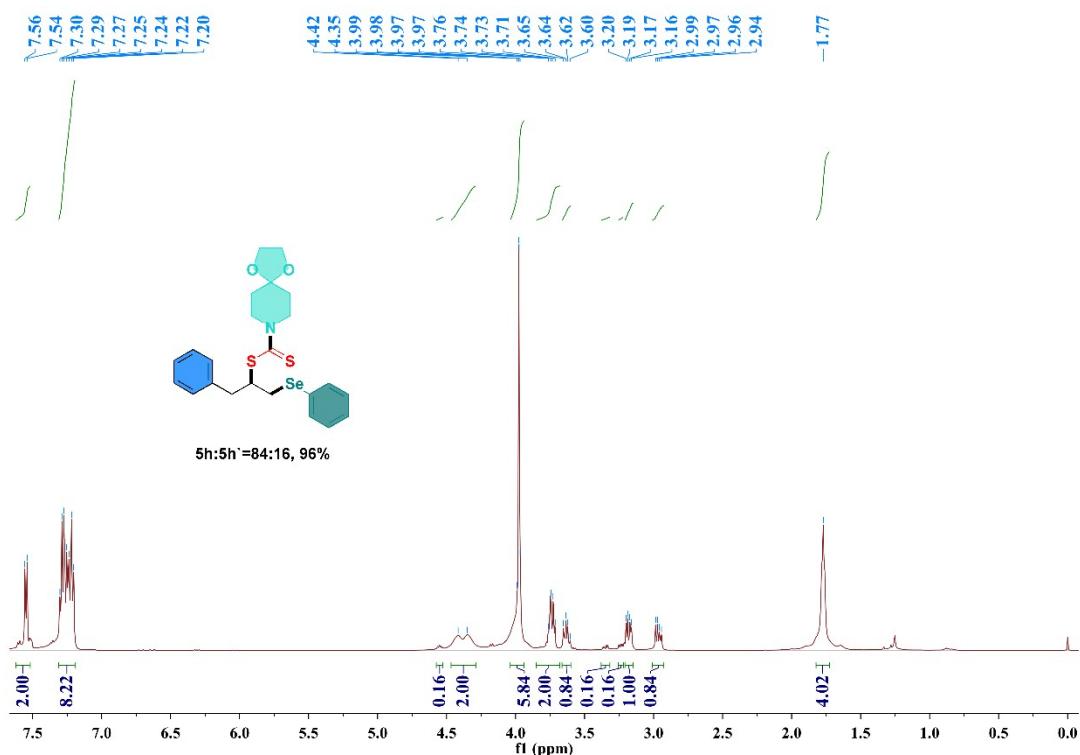
<sup>1</sup>H NMR spectra of compound **5g**



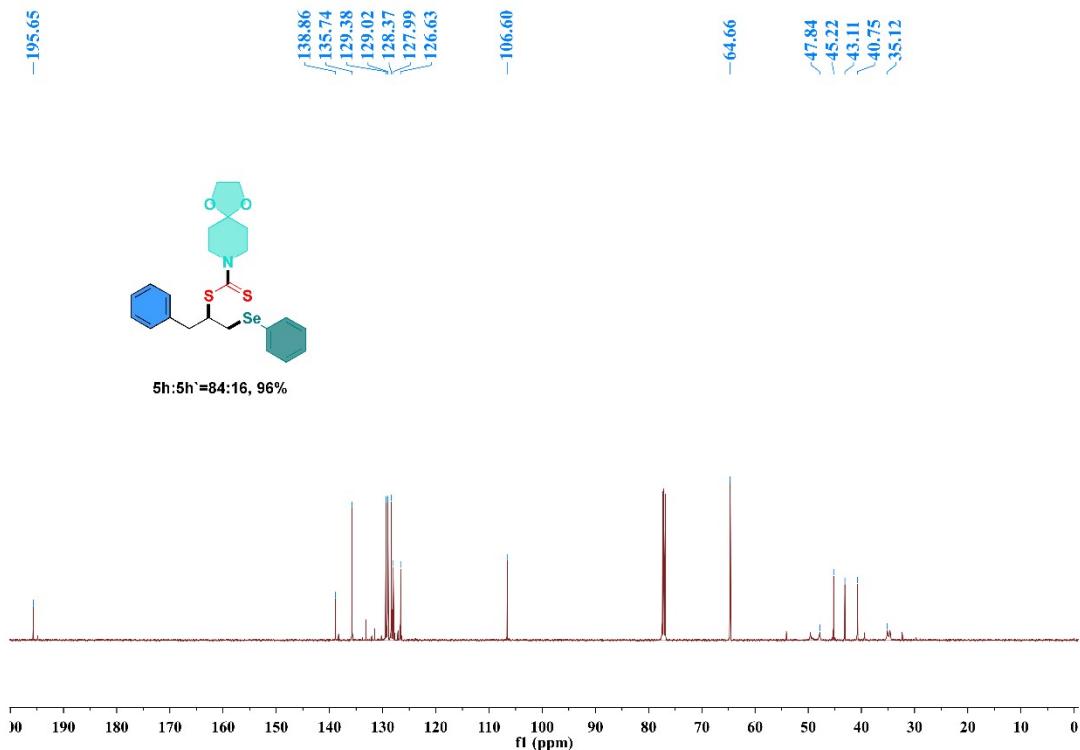
<sup>13</sup>C NMR spectra of compound **5g**



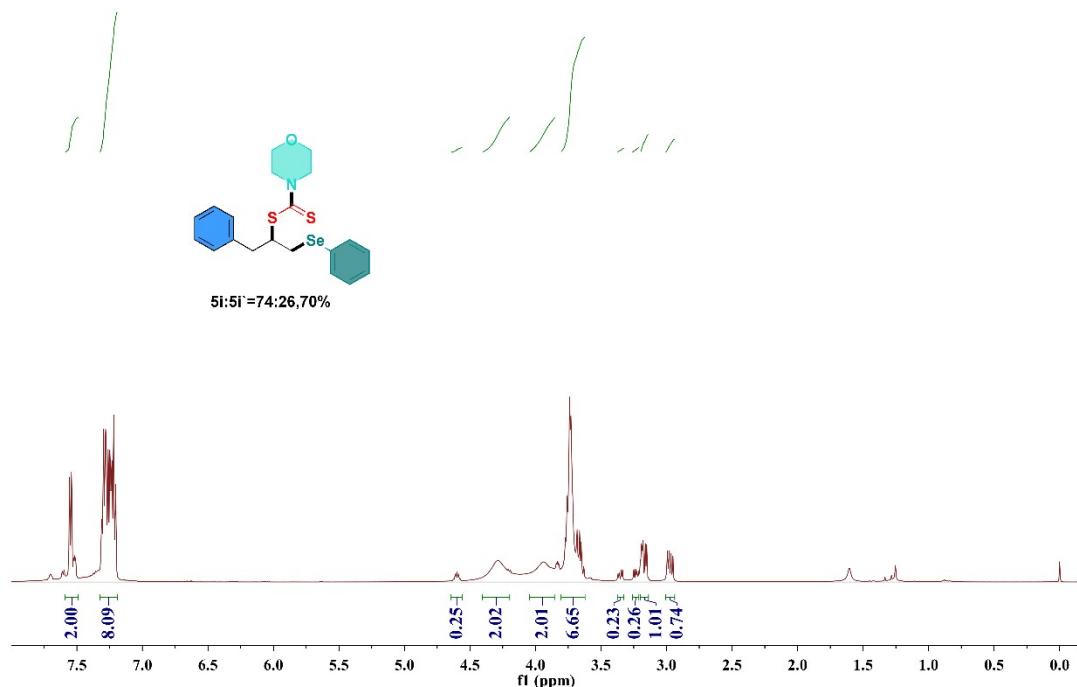
<sup>1</sup>H NMR spectra of compound **5h**



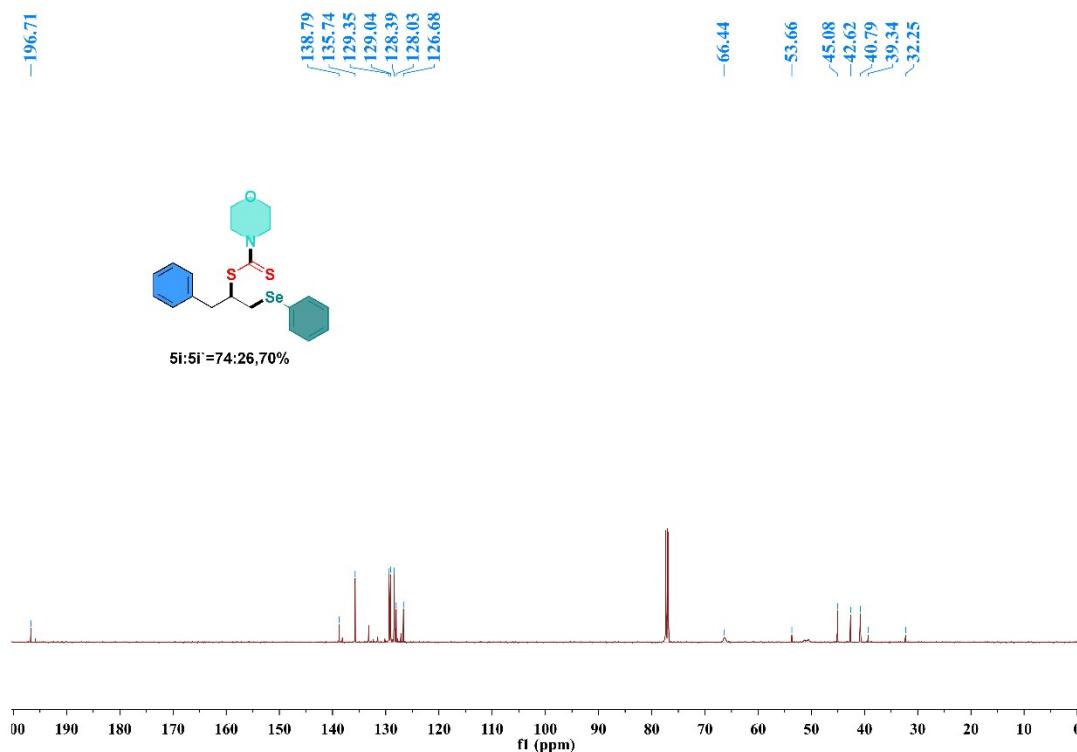
<sup>13</sup>C NMR spectra of compound **5h**



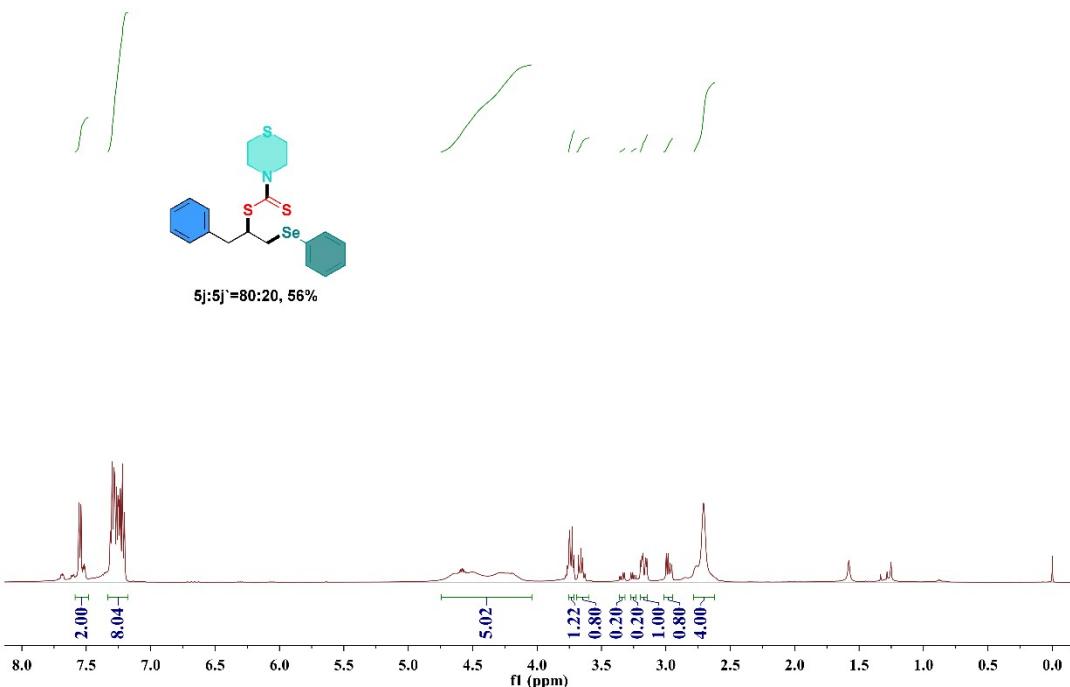
<sup>1</sup>H NMR spectra of compound **5i**



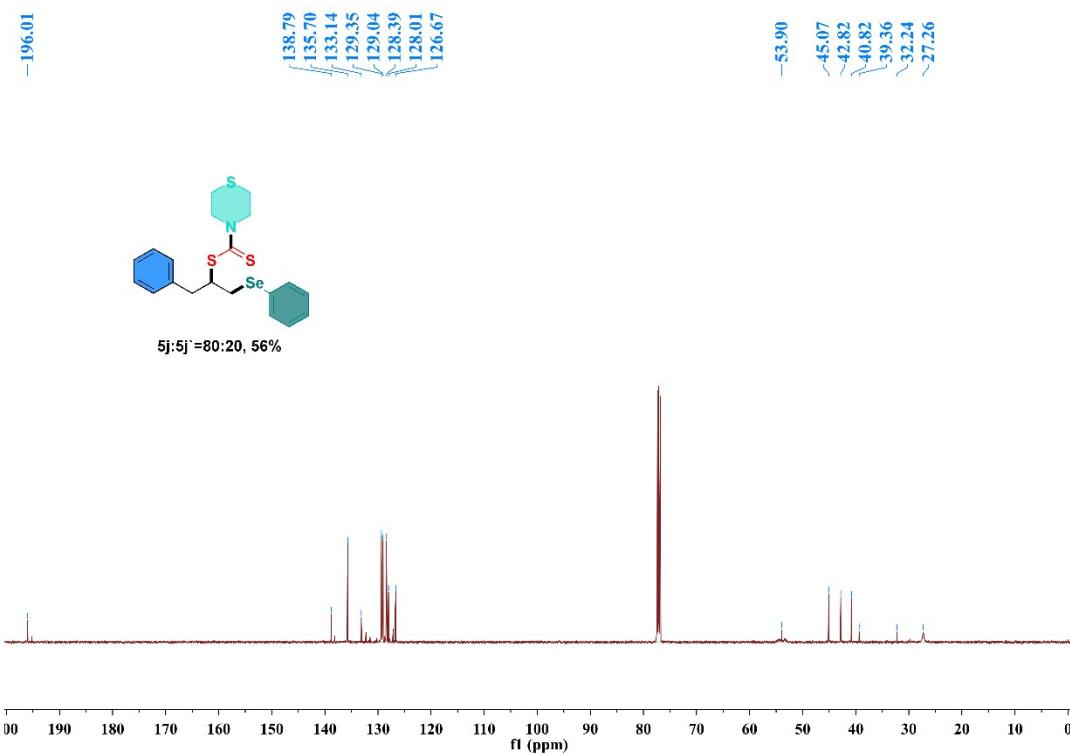
<sup>13</sup>C NMR spectra of compound **5i**



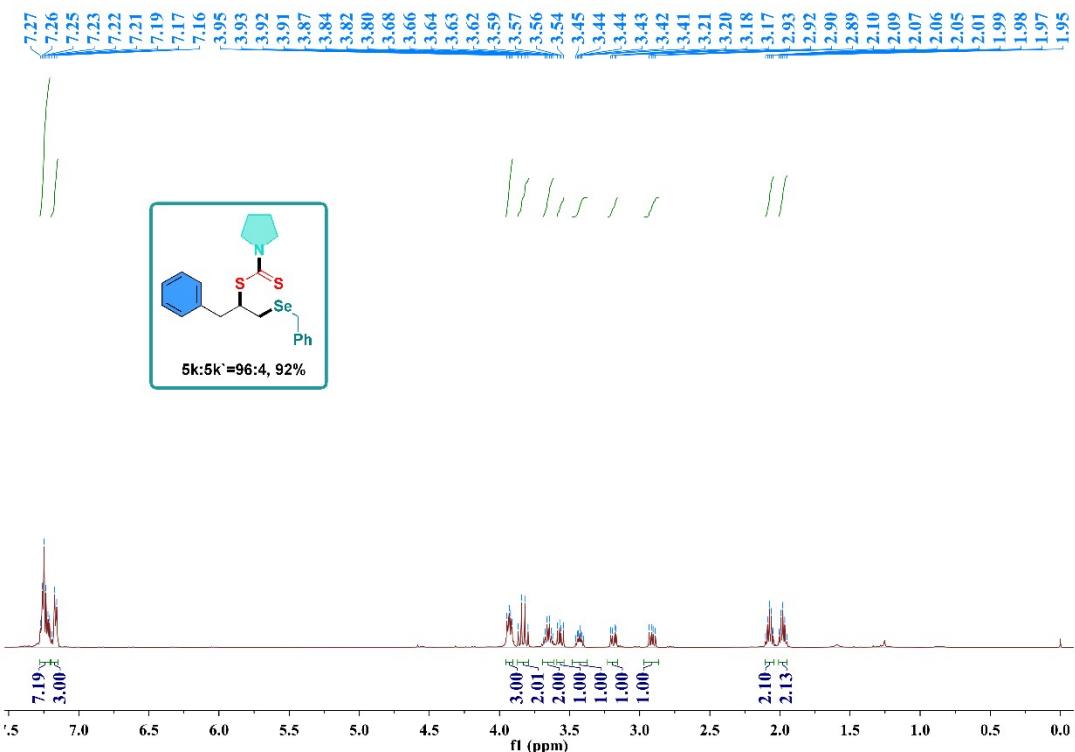
<sup>1</sup>H NMR spectra of compound **5j**



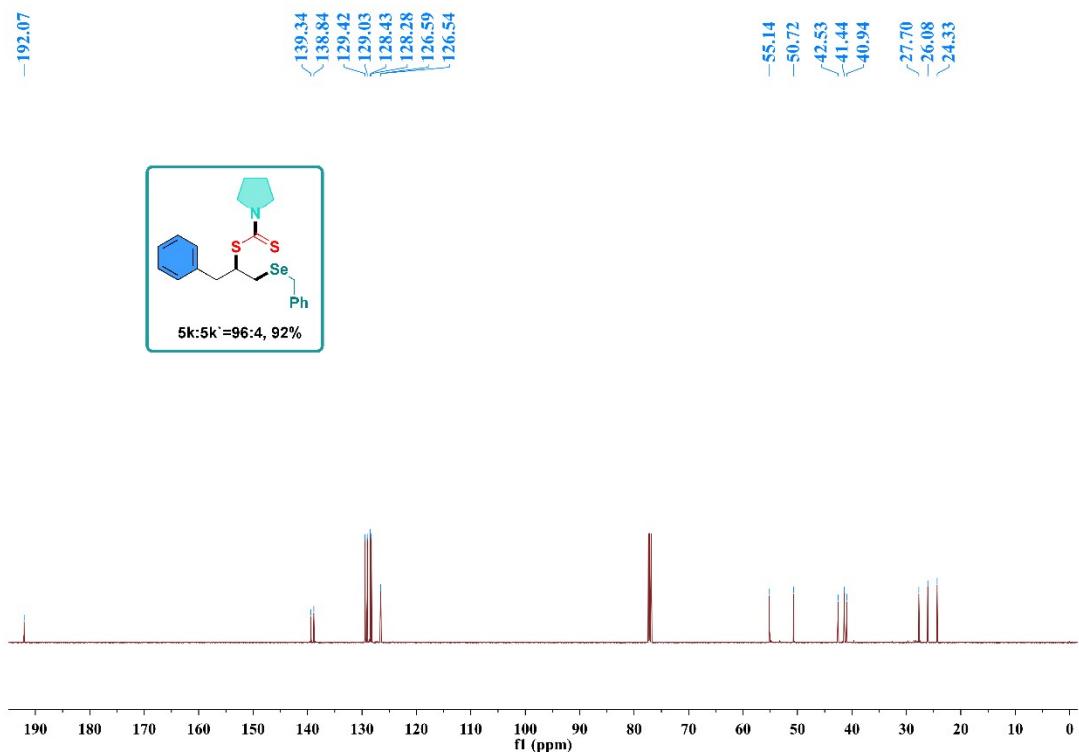
<sup>13</sup>C NMR spectra of compound **5j**



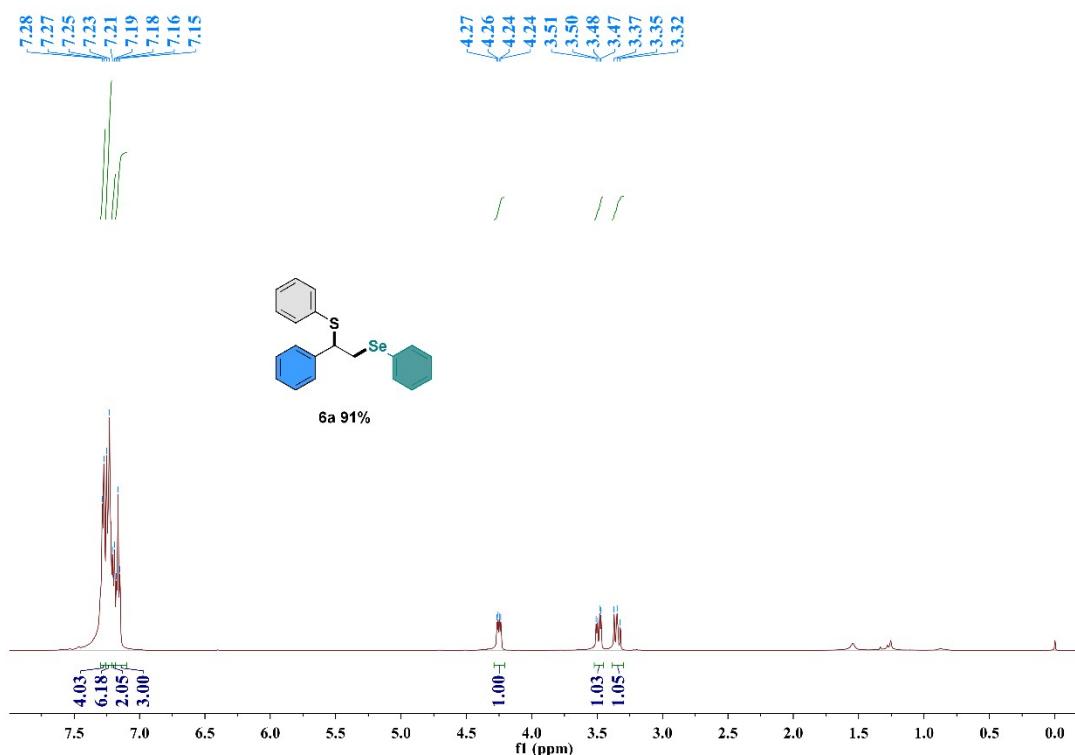
<sup>1</sup>H NMR spectra of compound **5k**



<sup>13</sup>C NMR spectra of compound **5k**



<sup>1</sup>H NMR spectra of compound 6a



<sup>13</sup>C NMR spectra of compound 6a

