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# Supporting Information

## One-Pot Four-Component Assembly for Diselenocarbamates

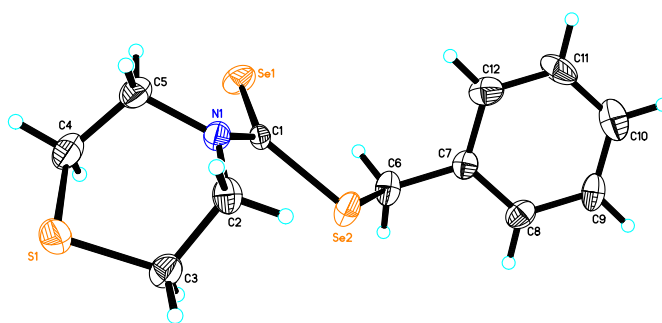
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Crystal of **3n** (CCDC-2062541)

<b>Table 1 Crystal data and structure refinement for 3n.</b>	
Identification code	<b>3n</b>
Empirical formula	C <sub>12</sub> H <sub>15</sub> NSSe <sub>2</sub>
Formula weight	363.23
Temperature/K	296(2)
Crystal system	monoclinic
Space group	C2/c
a/Å	23.773(14)
b/Å	6.507(4)
c/Å	18.085(11)
α/°	90
β/°	105.479(11)
γ/°	90
Volume/Å <sup>3</sup>	2696(3)
Z	8
ρ <sub>calc</sub> /cm <sup>3</sup>	1.790
μ/mm <sup>-1</sup>	5.614
F(000)	1424.0
Crystal size/mm <sup>3</sup>	0.200 × 0.180 × 0.100
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	4.674 to 50.09
Index ranges	-23 ≤ h ≤ 28, -7 ≤ k ≤ 7, -21 ≤ l ≤ 9
Reflections collected	3865
Independent reflections	2306 [R <sub>int</sub> = 0.0315, R <sub>sigma</sub> = 0.0603]

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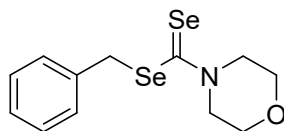
Data/restraints/parameters	2306/0/145
Goodness-of-fit on $F^2$	1.065
Final R indexes [ $I \geq 2\sigma(I)$ ]	$R_1 = 0.0621$ , $wR_2 = 0.1861$
Final R indexes [all data]	$R_1 = 0.0780$ , $wR_2 = 0.2229$
Largest diff. peak/hole / $e \text{ \AA}^{-3}$	0.93/-1.12

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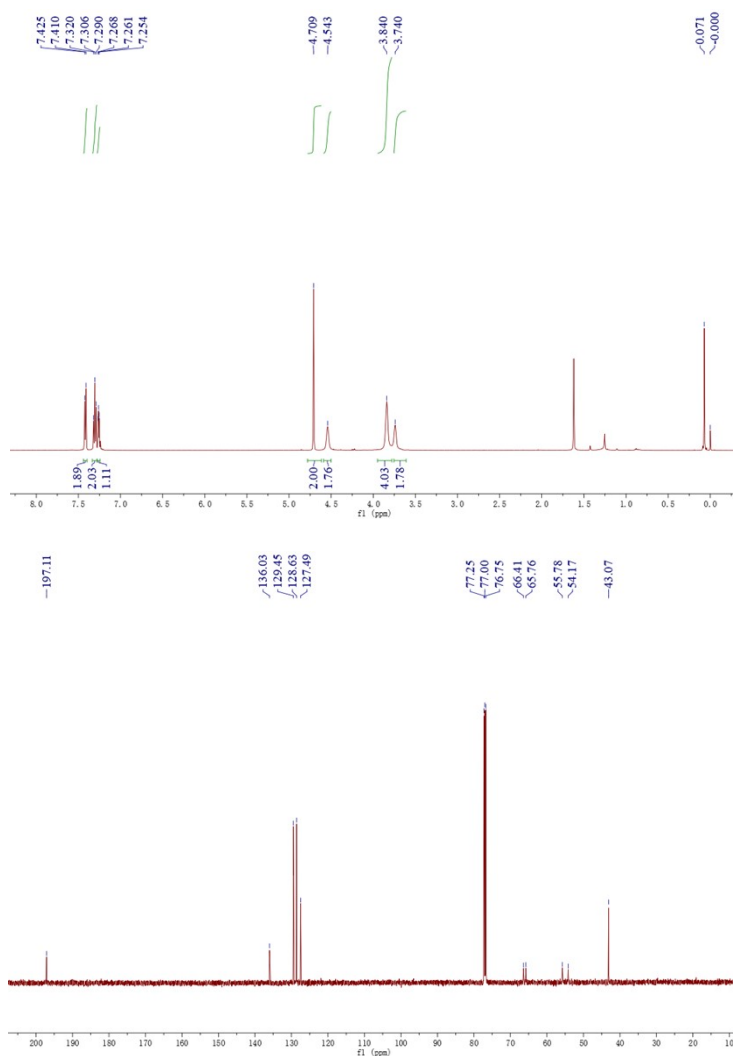
**General remarks.** Melting points are uncorrected. NMR spectra were recorded at 500 or 400 (for  $^1\text{H}$  NMR) or 125 MHz (for  $^{13}\text{C}$  NMR), respectively.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra recorded in  $\text{CDCl}_3$  solutions were referenced to TMS (0.00 ppm) and the residual solvent peak (77.0 ppm), respectively.  $J$ -values are in Hz. All amines were distilled prior to using. Other commercially obtained reagents were used without further purification. Organic solvents used were dried by standard methods. The mass analyzer type for the high resolution mass spectra is Q-TOF. Flash column chromatography was performed on silica gel.

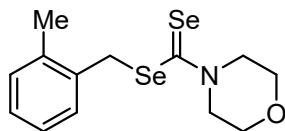
***General procedure for the synthesis of diselenocarbamates***

Under air atmosphere,  $t\text{BuOK}$  (1.8 mmol, 6.0 equiv), Se (0.9 mmol, 3.0 equiv), diselane **1** (0.36 mmol, 1.2 equiv), NMP (1.0 mL), amine **2** (0.3 mmol) and chloroform (3.0 mmol, 10.0 equiv) were successively added into a Schlenk reaction tube. Then the mixture was stirred at 50 °C for 12 h. After the reaction was finished, saturated aq.  $\text{NH}_4\text{Cl}$  was added and the mixture was extracted with ethyl acetate. The organic layer was washed by brine and dried over anhydrous  $\text{Na}_2\text{SO}_4$ . Then the organic solvent was evaporated under vacuum and the residue was purified by flash column chromatography on silica gel to give the pure products **3**.

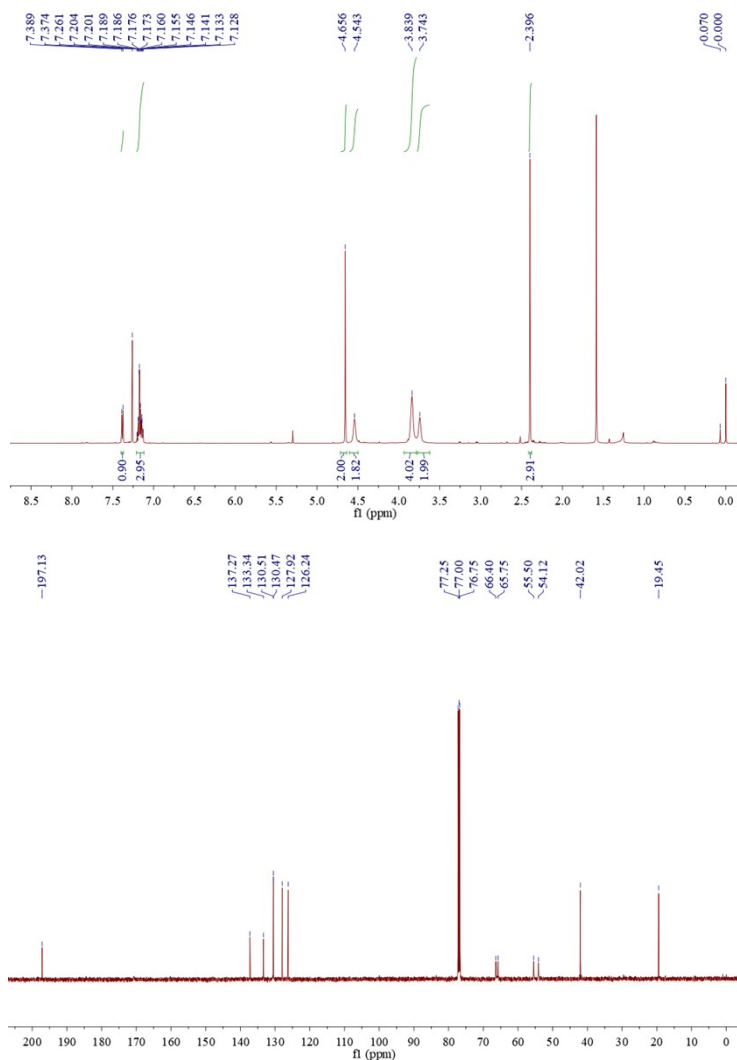


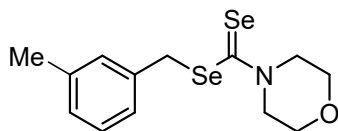
Compound **3a**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (85.4 mg, 82%). m.p. 66.0-66.2 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.42 (d, *J* = 7.5 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.26 (t, *J* = 3.5 Hz, 1H), 4.71 (s, 2H), 4.54 (br, 2H), 3.84 (br, 4H), 3.74 (br, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 197.1, 136.0, 129.5, 128.6, 127.5, 66.4, 65.8, 55.8, 54.2, 43.1. IR (neat) 2953, 2854, 1495, 1407, 1237, 1111, 1028, 960, 850, 760, 698 cm<sup>-1</sup>. MS (ESI): 350 [M+H<sup>+</sup>]. HRMS (ESI) calcd for C<sub>12</sub>H<sub>16</sub>NOSe<sub>2</sub> [M+H<sup>+</sup>] 349.9559; found 349.9551.



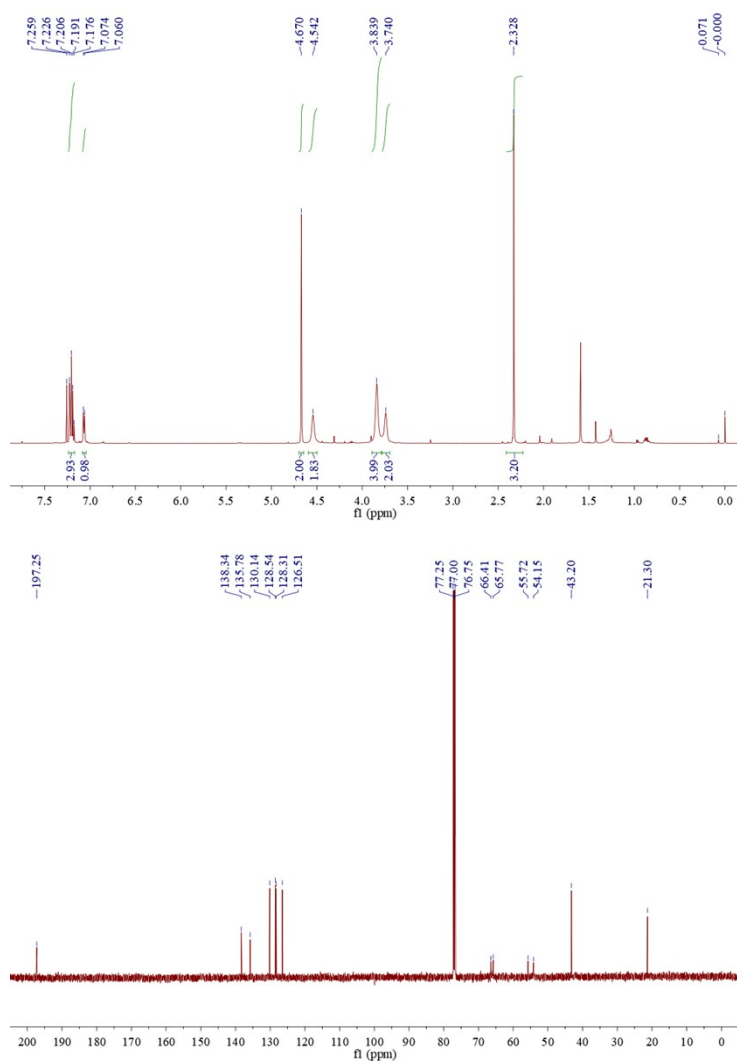


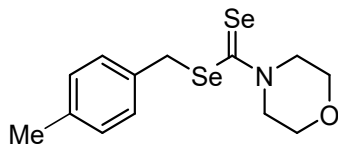
Compound **3b**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (98.6 mg, 91%). m.p. 70.7-71.3 °C.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.38 (d,  $J = 7.5$  Hz, 1H), 7.20-7.13 (m, 3H), 4.66 (s, 2H), 4.54 (br, 2H), 3.84 (br, 4H), 3.74 (br, 2H), 2.40 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  197.1, 137.3, 133.3, 130.51, 130.47, 127.9, 126.2, 66.4, 65.8, 55.5, 54.1, 42.0, 19.5. IR (neat) 2965, 2859, 1460, 1414, 1263, 1211, 1107, 1017, 938, 805, 767  $\text{cm}^{-1}$ . MS (ESI): 364  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_{13}\text{H}_{18}\text{NOSe}_2$   $[\text{M}+\text{H}^+]$  363.9716; found 363.9725.



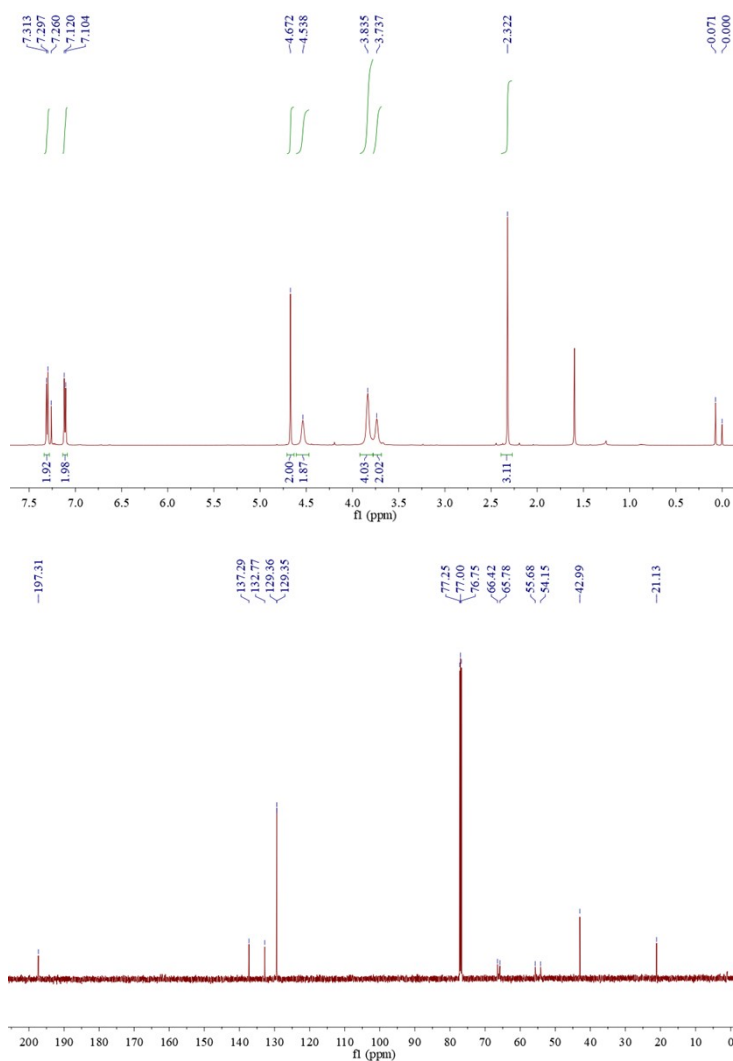


Compound **3c**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as brown liquid (92.1 mg, 85%).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.23-7.18 (m, 3H), 7.07 (d,  $J = 7.0$  Hz, 1H), 4.67 (s, 2H), 4.54 (br, 2H), 3.84 (br, 4H), 3.74 (br, 2H), 2.33 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  197.3, 138.3, 135.8, 130.1, 128.5, 128.3, 126.5, 66.4, 65.8, 55.7, 54.2, 43.2, 21.3. IR (neat) 2962, 2855, 1602, 1472, 1423, 1262, 1234, 1105, 1020, 936, 842, 790, 698  $\text{cm}^{-1}$ . MS (ESI): 364  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_{13}\text{H}_{18}\text{NOSe}_2$   $[\text{M}+\text{H}^+]$  363.9716; found 363.9720.

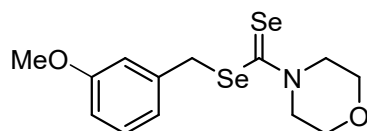




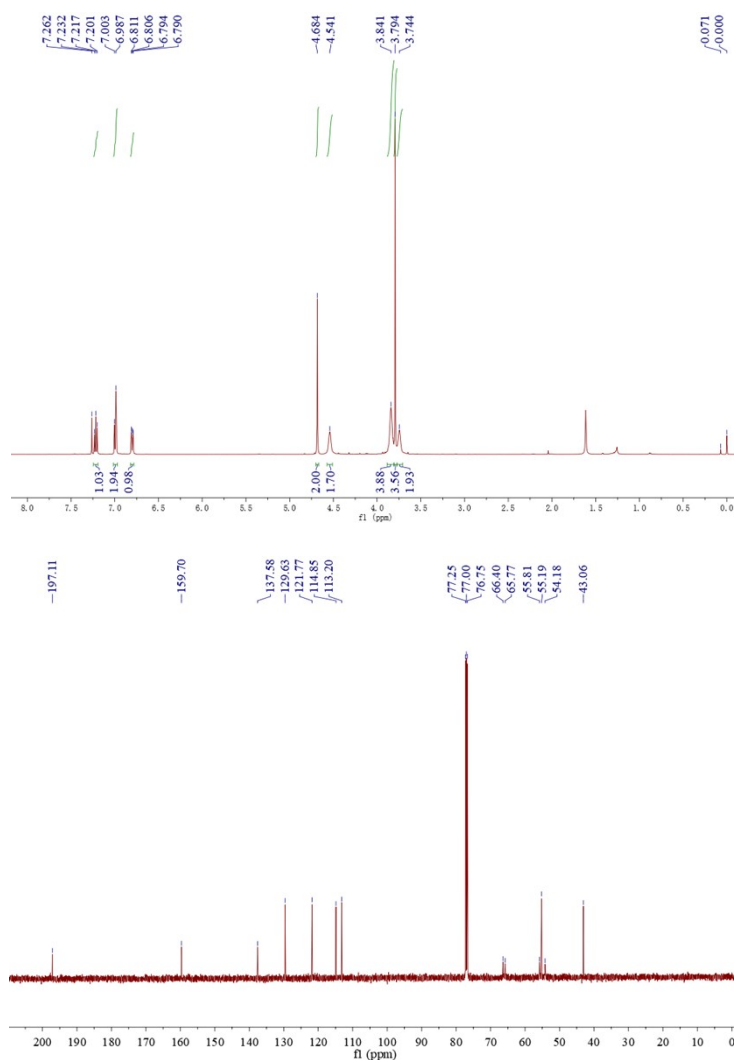
Compound **3d**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as brown liquid (96.4 mg, 89%). m.p. 75.6-76.5 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.31 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 4.67 (s, 2H), 4.54 (br, 2H), 3.84 (br, 4H), 3.74 (br, 2H), 2.32 (s, 3H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 197.3, 137.3, 132.8, 129.36, 129.35, 66.4, 65.8, 55.7, 54.2, 43.0, 21.1. IR (neat) 2961, 2854, 1515, 1414, 1263, 1237, 1111, 1020, 955, 811, 715 cm<sup>-1</sup>. MS (ESI): 364 [M+H<sup>+</sup>]. HRMS (ESI) calcd for C<sub>13</sub>H<sub>18</sub>NOSe<sub>2</sub> [M+H<sup>+</sup>] 363.9716; found 363.9705.

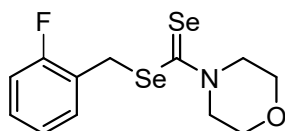




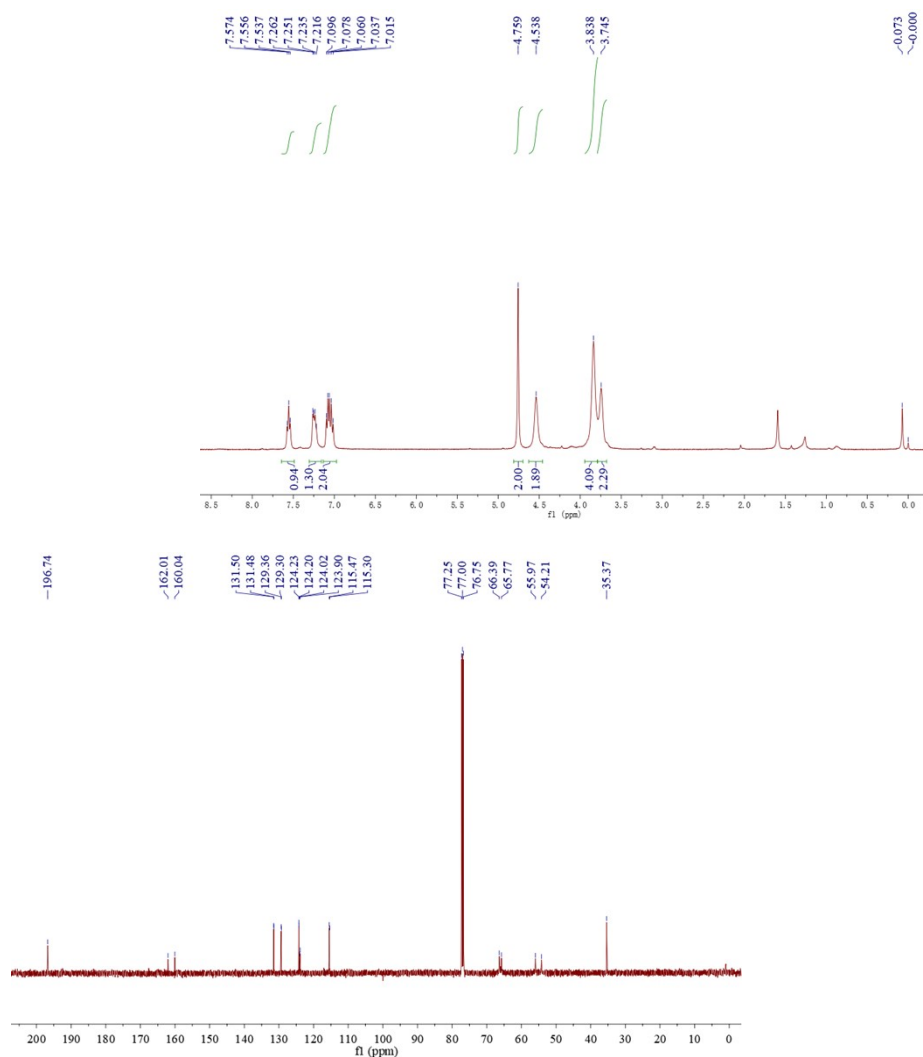


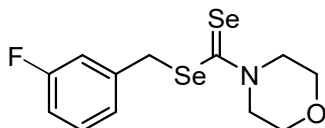
Compound **3e**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as orange liquid (95.1 mg, 84%).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.22 (t,  $J = 8.0$  Hz, 1H), 6.99 (d,  $J = 8.0$  Hz, 2H), 6.8 (dd,  $J = 8.0, 2.5$  Hz, 1H), 4.68 (s, 2H), 4.54 (br, 2H), 3.84 (br, 4H), 3.79 (s, 3H), 3.74 (br, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  197.1, 159.7, 137.6, 129.6, 121.8, 114.9, 113.2, 66.4, 65.8, 55.8, 55.2, 54.2, 43.1. IR (neat) 2982, 2852, 1596, 1462, 1416, 1296, 1261, 1225, 1150, 1107, 1021, 941, 849, 780, 697  $\text{cm}^{-1}$ . MS (ESI): 380  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_{13}\text{H}_{18}\text{NO}_2\text{Se}_2$   $[\text{M}+\text{H}^+]$  379.9665; found 379.9659.



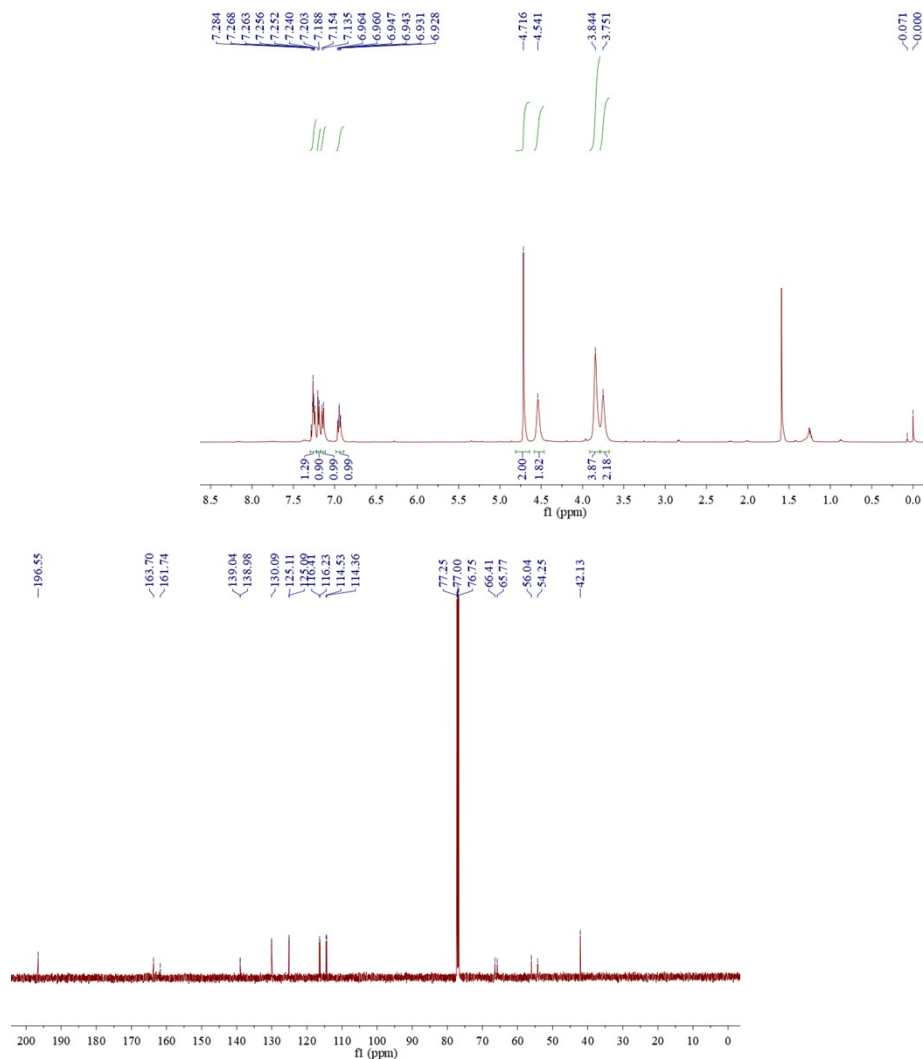


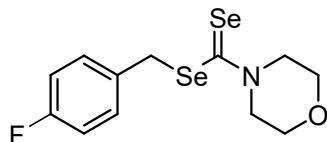
Compound **3f**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as orange solid (90.9 mg, 83%). m.p. 57.4-58.5 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 7.56 (t, *J* = 7.2 Hz, 1H), 7.26-7.22 (m, 1H), 7.10-7.02 (m, 2H), 4.76 (s, 2H), 4.54 (br, 2H), 3.84 (br, 4H), 3.75 (br, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 196.7, 161.0 (d, *J*<sub>C-F</sub> = 246.25 Hz), 131.5 (d, *J*<sub>C-F</sub> = 2.5 Hz), 129.3 (d, *J*<sub>C-F</sub> = 7.5 Hz), 124.2 (d, *J*<sub>C-F</sub> = 3.75 Hz), 124.0 (d, *J*<sub>C-F</sub> = 15.0 Hz), 115.4 (d, *J*<sub>C-F</sub> = 21.25 Hz), 66.4, 65.8, 56.0, 54.2, 35.4. <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>) δ -115.96. IR (neat) 2974, 2913, 2858, 1698, 1585, 1488, 1421, 1259, 1223, 1111, 1025, 954, 848, 756 cm<sup>-1</sup>. MS (ESI): 390 [M+Na<sup>+</sup>]. HRMS (ESI) calcd for C<sub>12</sub>H<sub>14</sub>FNNaOSe<sub>2</sub> [M+Na<sup>+</sup>] 389.9284; found 389.9293.



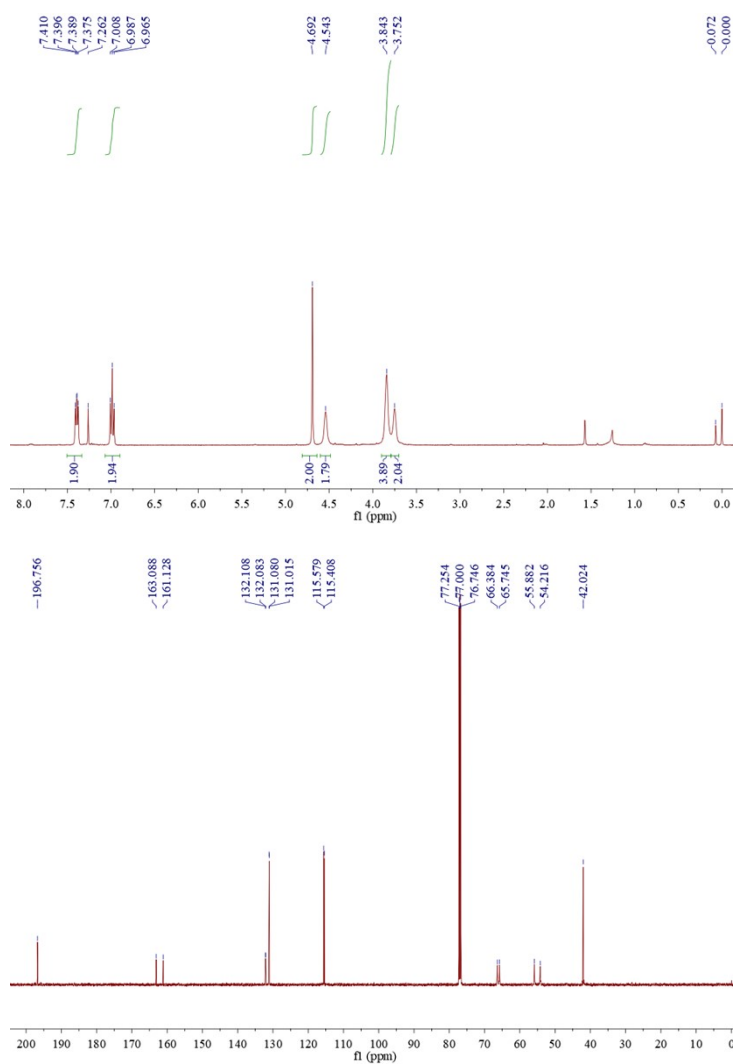


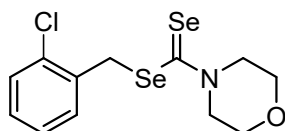
Compound **3g**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as orange liquid (90.9 mg, 83%).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.28-7.24 (m, 1H), 7.20 (d,  $J = 7.5$  Hz, 1H), 7.14 (d,  $J = 9.5$  Hz, 1H), 6.95 (td,  $J = 7.5, 1.5$  Hz, 1H), 4.72 (s, 2H), 4.54 (br, 2H), 3.84 (br, 4H), 3.75 (br, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.6, 162.7 (d,  $J_{\text{C-F}} = 245.0$  Hz), 139.0 (d,  $J_{\text{C-F}} = 7.5$  Hz), 130.1 (d,  $J_{\text{C-F}} = 8.75$  Hz), 125.1 (d,  $J_{\text{C-F}} = 2.5$  Hz), 116.3 (d,  $J_{\text{C-F}} = 22.5$  Hz), 114.4 (d,  $J_{\text{C-F}} = 21.25$  Hz), 66.4, 65.8, 56.0, 54.3, 42.1.  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -112.76. IR (neat) 2900, 2855, 1585, 1470, 1423, 1259, 1229, 1104, 1021, 934, 871, 790, 688  $\text{cm}^{-1}$ . MS (ESI): 390  $[\text{M}+\text{Na}^+]$ . HRMS (ESI) calcd for  $\text{C}_{12}\text{H}_{14}\text{FNNaOSe}_2$   $[\text{M}+\text{Na}^+]$  389.9284; found 389.9278.



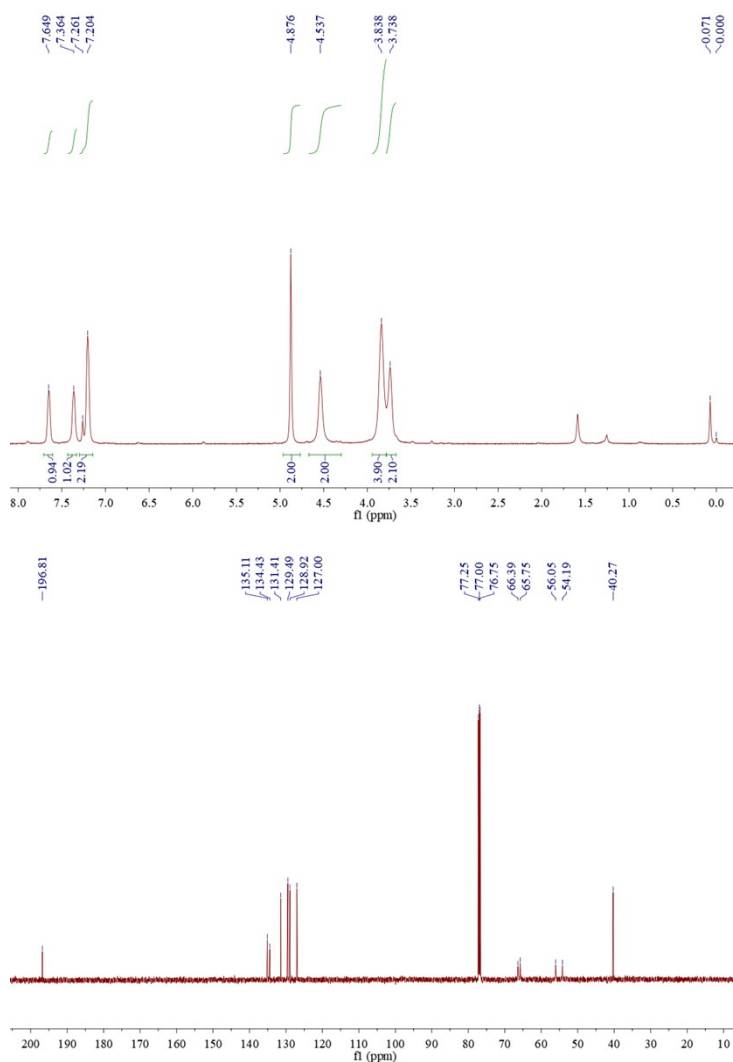


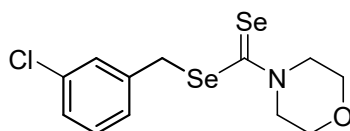
Compound **3h**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as orange solid (88.7 mg, 81%). m.p. 71.4-71.5 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.39 (dd,  $J = 8.4, 5.6$  Hz, 2H), 6.99 (t,  $J = 8.4$  Hz, 2H), 4.69 (s, 2H), 4.54 (br, 2H), 3.84 (br, 2H), 3.75 (br, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.8, 162.1 (d,  $J_{\text{C-F}} = 245.0$  Hz), 132.1 (d,  $J_{\text{C-F}} = 3.125$  Hz), 131.0 (d,  $J_{\text{C-F}} = 8.125$  Hz), 115.5 (d,  $J_{\text{C-F}} = 21.375$  Hz), 66.4, 65.7, 55.9, 54.2, 42.0.  $^{19}\text{F}$  NMR (470 MHz,  $\text{CDCl}_3$ )  $\delta$  -114.54. IR (neat) 2868, 1600, 1509, 1417, 1266, 1220, 1118, 1025, 961, 821, 749  $\text{cm}^{-1}$ . MS (ESI): 390  $[\text{M}+\text{Na}^+]$ . HRMS (ESI) calcd for  $\text{C}_{12}\text{H}_{14}\text{FNNaOSe}_2$   $[\text{M}+\text{Na}^+]$  389.9284; found 389.9274.



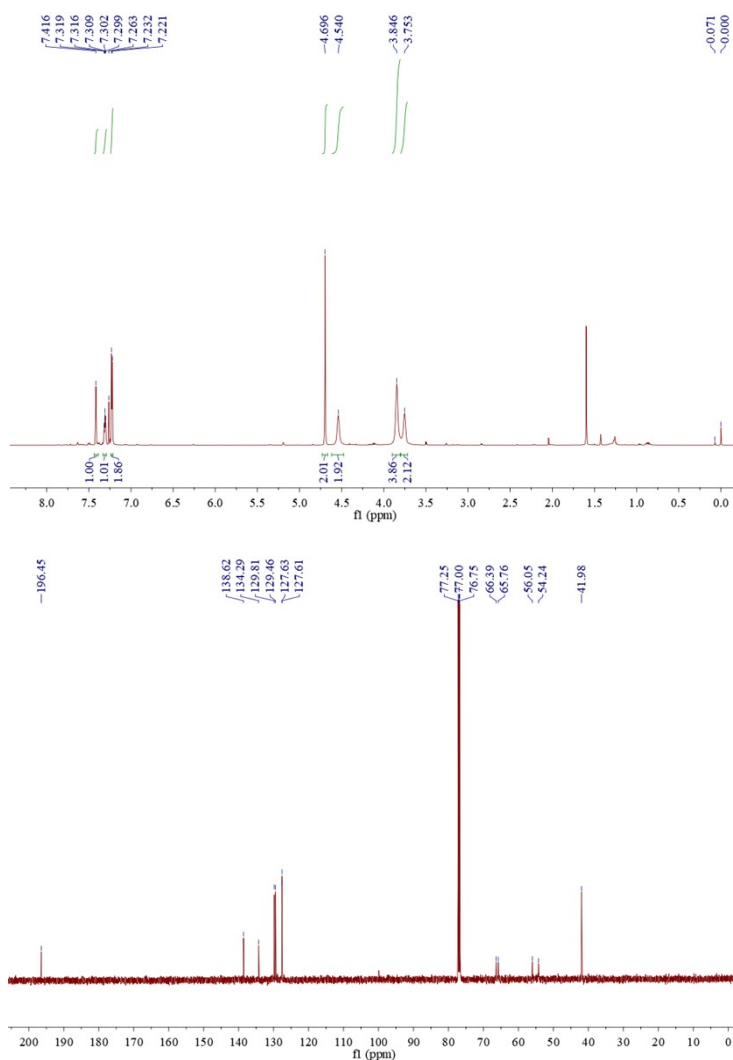


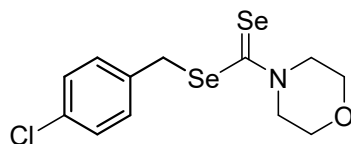
Compound **3i**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (99.6 mg, 87%). m.p. 76.7-78.2 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 7.65 (br, 1H), 7.36 (br, 1H), 7.20 (br, 2H), 4.88 (s, 2H), 4.54 (br, 2H), 3.84 (br, 2H), 3.74 (br, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 196.8, 135.1, 134.4, 131.4, 129.5, 128.9, 127.0, 66.4, 65.8, 56.1, 54.2, 40.3. IR (neat) 2967, 2923, 2859, 1415, 1303, 1261, 1216, 1109, 1019, 950, 806, 748, 678 cm<sup>-1</sup>. MS (ESI): 406 [M+Na<sup>+</sup>]. HRMS (ESI) calcd for C<sub>12</sub>H<sub>14</sub>ClNNaOSe<sub>2</sub> [M+Na<sup>+</sup>] 405.8984; found 405.8984.



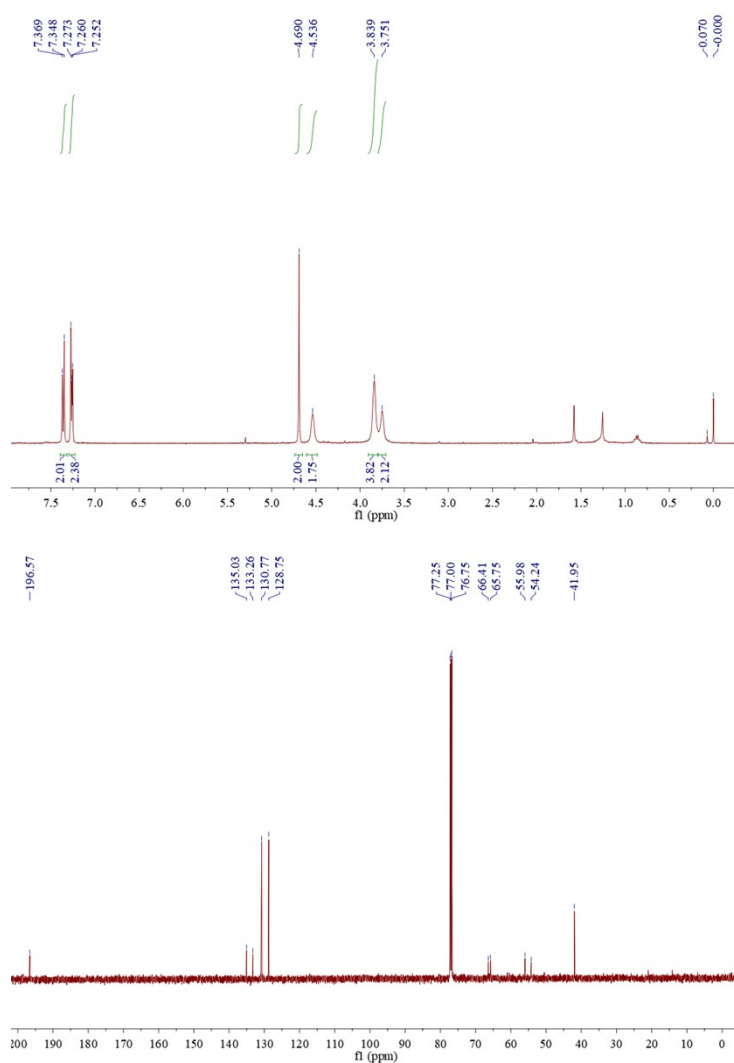


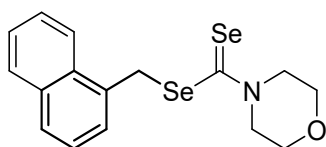
Compound **3j**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow liquid (92.7 mg, 81%).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.42 (s, 1H), 7.32-7.30 (m, 1H), 7.23 (d,  $J = 5.5$  Hz, 2H), 4.70 (s, 2H), 4.54 (br, 2H), 3.85 (br, 2H), 3.75 (br, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.5, 138.6, 134.3, 129.8, 129.5, 127.63, 127.61, 66.4, 65.8, 56.1, 54.2, 42.0. IR (neat) 2961, 2856, 1594, 1570, 1471, 1417, 1299, 1262, 1215, 1102, 1020, 933, 865, 796, 678  $\text{cm}^{-1}$ . MS (ESI): 406  $[\text{M}+\text{Na}^+]$ . HRMS (ESI) calcd for  $\text{C}_{12}\text{H}_{14}\text{ClNNaOSe}_2$   $[\text{M}+\text{Na}^+]$  405.8984; found 405.8986.



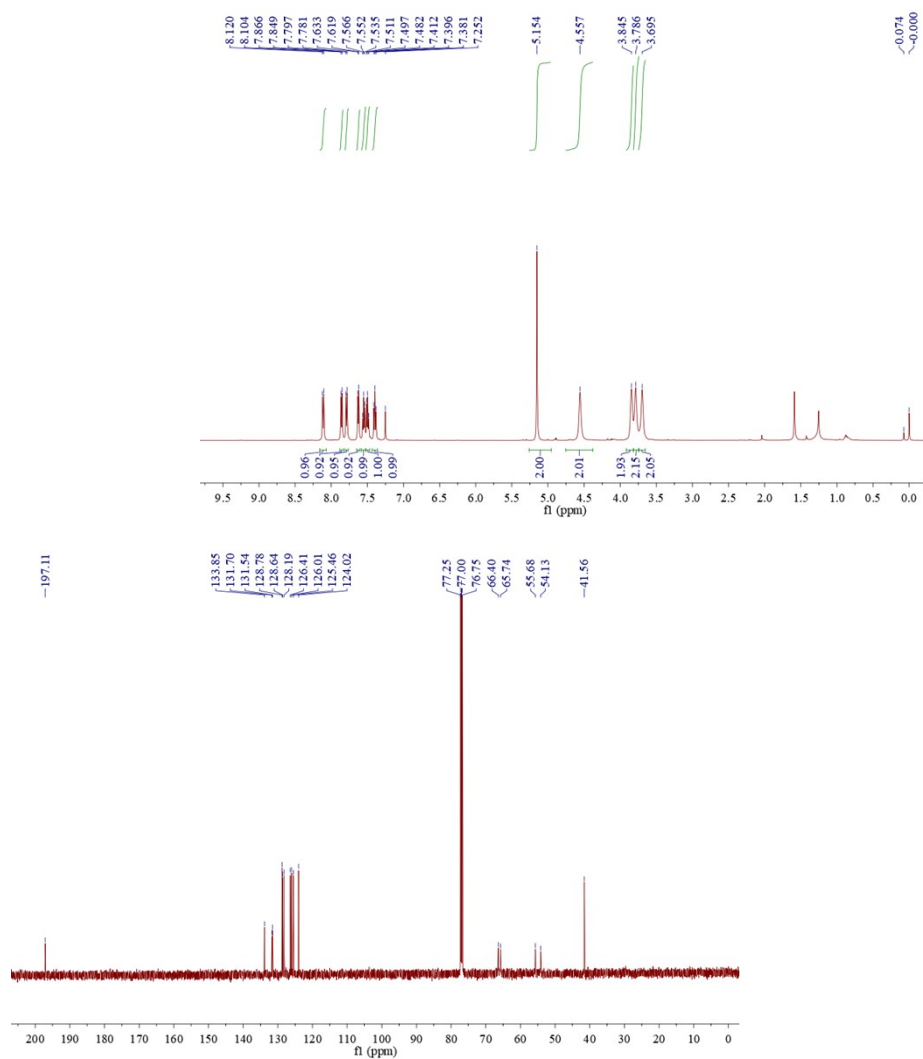


Compound **3k**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (98.5 mg, 86%). m.p. 91.7-92.2 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.36 (d,  $J = 8.4$  Hz, 2H), 7.26 (d,  $J = 8.4$  Hz, 2H), 4.69 (s, 2H), 4.54 (br, 2H), 3.84 (br, 4H), 3.75 (br, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.6, 135.0, 133.3, 130.8, 128.8, 66.4, 65.8, 56.0, 54.2, 42.0. IR (neat) 2956, 2855, 1471, 1423, 1298, 1257, 1225, 1107, 1021, 934, 829, 722  $\text{cm}^{-1}$ . MS (ESI): 406  $[\text{M}+\text{Na}^+]$ . HRMS (ESI) calcd for  $\text{C}_{12}\text{H}_{14}\text{ClNNaOSe}_2$   $[\text{M}+\text{Na}^+]$  405.8984; found 405.8975.

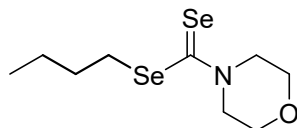




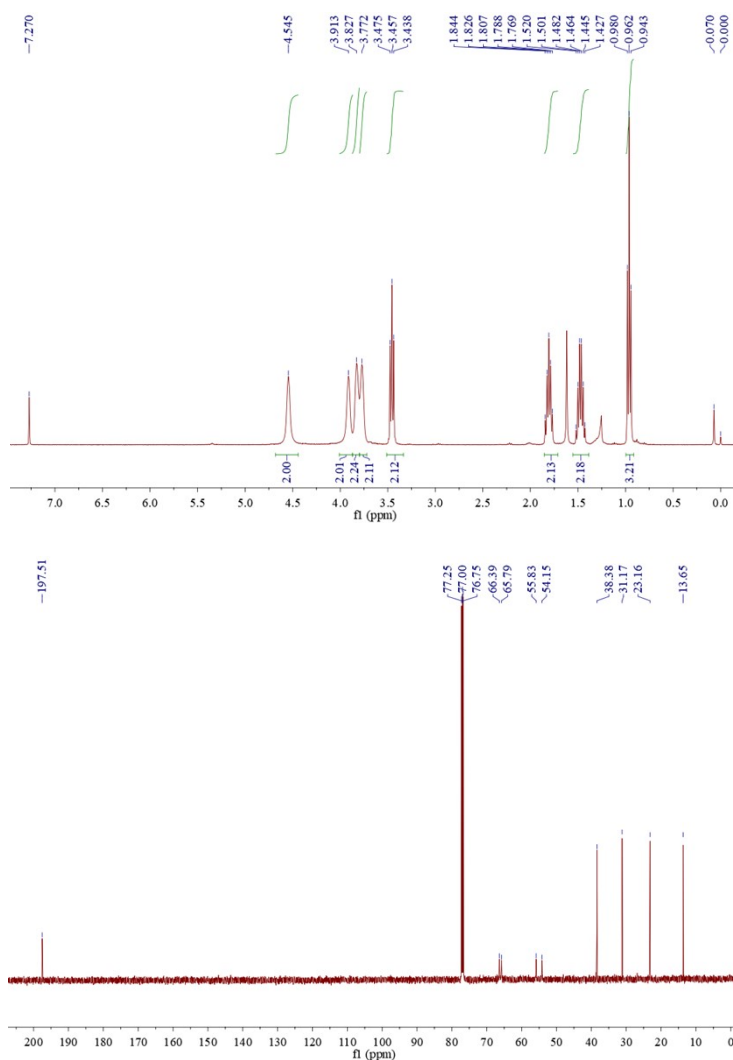
Compound **3I**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (101.3 mg, 85%). m.p. 108.5-110.2 °C.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  8.11 (d,  $J = 8.0$  Hz, 1H), 7.86 (d,  $J = 8.5$  Hz, 1H), 7.79 (d,  $J = 8.0$  Hz, 1H), 7.63 (d,  $J = 7.0$  Hz, 1H), 7.55 (t,  $J = 7.5$  Hz, 1H), 7.50 (t,  $J = 7.5$  Hz, 1H), 7.40 (t,  $J = 8.0$  Hz, 1H), 5.15 (s, 2H), 4.56 (br, 2H), 3.85 (br, 2H), 3.79 (br, 2H), 3.70 (br, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  197.1, 133.9, 131.7, 131.5, 128.8, 128.6, 128.2, 126.4, 126.0, 125.5, 124.0, 66.4, 65.7, 55.7, 54.1, 41.6. IR (neat) 2965, 2855, 1510, 1462, 1416, 1266, 1222, 1111, 1021, 948, 846, 778  $\text{cm}^{-1}$ . MS (ESI): 400  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_{16}\text{H}_{18}\text{NOSe}_2$   $[\text{M}+\text{H}^+]$  399.9716; found 399.9704.

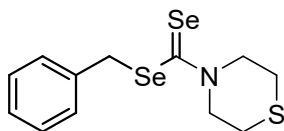




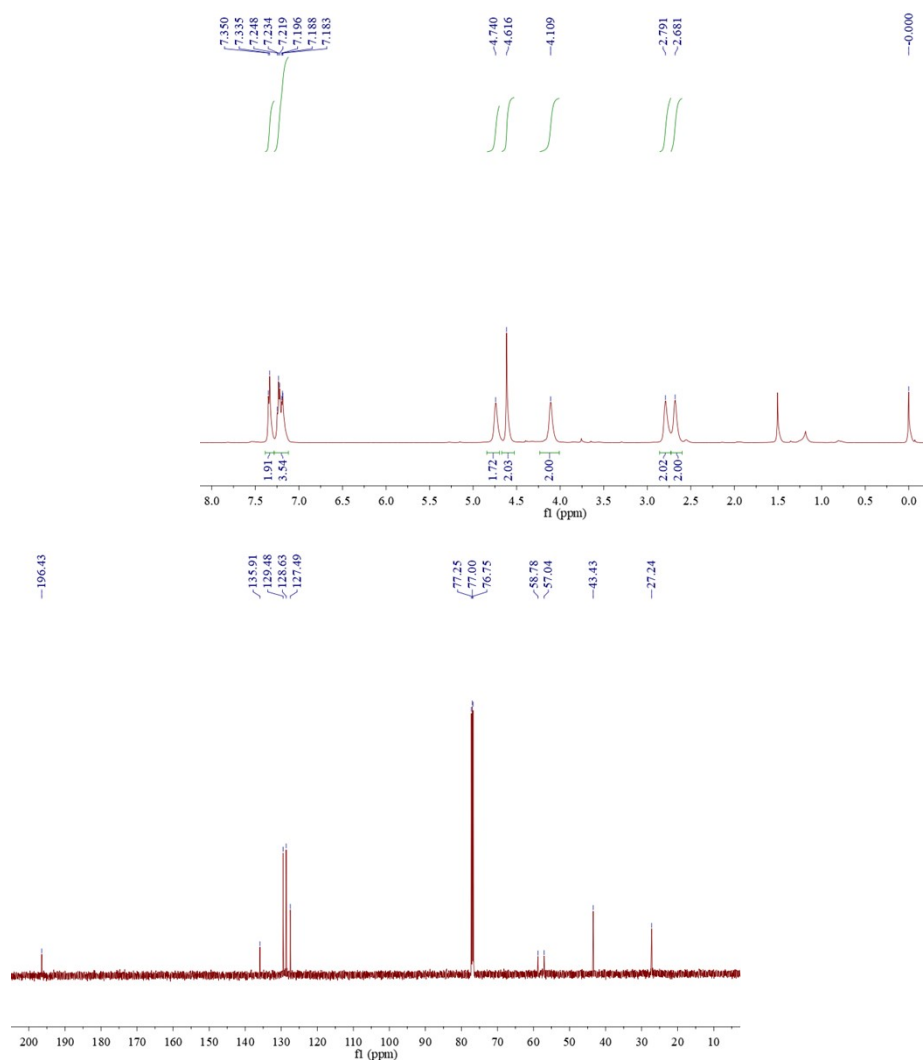


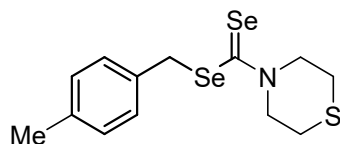
Compound **3m**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as brown liquid (83.6 mg, 89%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  4.54 (br, 2H), 3.91 (br, 2H), 3.83 (br, 2H), 3.77 (br, 2H), 3.46 (t,  $J = 7.6$  Hz, 2H), 1.81 (pent,  $J = 7.6$  Hz, 2H), 1.52-1.43 (m, 2H), 0.96 (t,  $J = 7.2$  Hz, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  197.5, 66.4, 65.8, 55.8, 54.2, 38.4, 31.2, 23.2, 13.7. IR (neat) 2957, 2923, 2853, 1459, 1413, 1262, 1224, 1109, 1019, 941, 843, 797  $\text{cm}^{-1}$ . MS (ESI): 316  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_9\text{H}_{18}\text{NOSe}_2$   $[\text{M}+\text{H}^+]$  315.9715; found 315.9701.



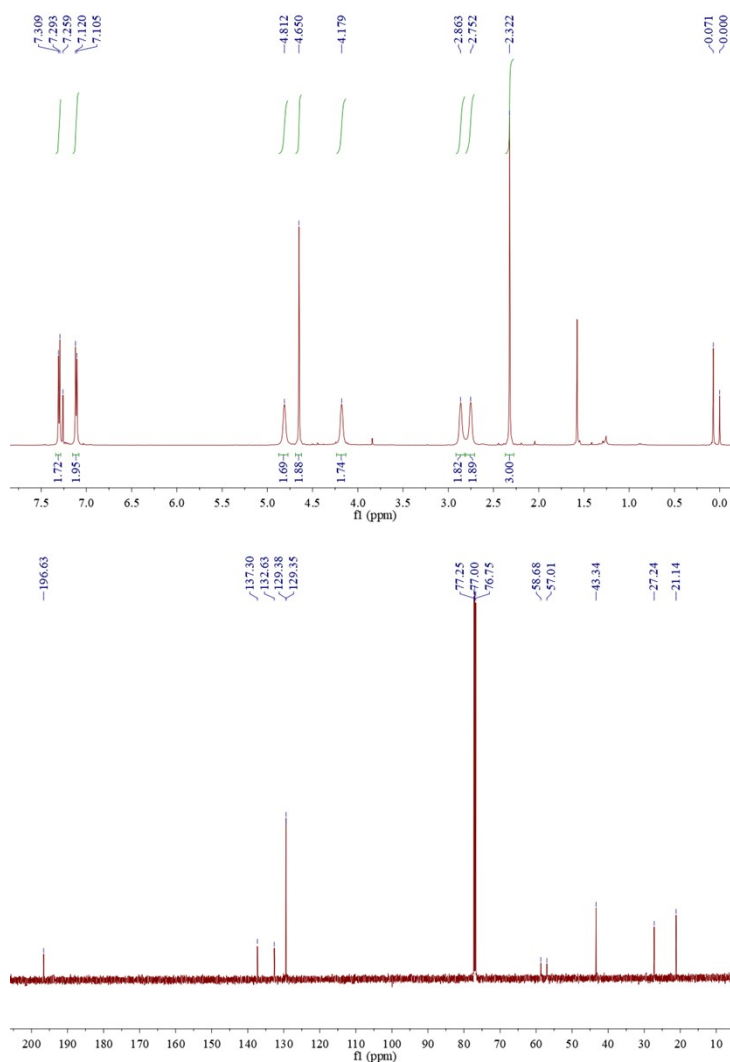


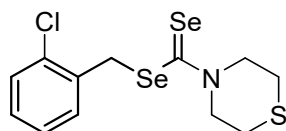
Compound **3n**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (89.3 mg, 82%). m.p. 118.9-120.7 °C.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.34 (d,  $J = 7.5$  Hz, 2H), 7.25-7.18 (m, 3H), 4.74 (br, 2H), 4.62 (s, 2H), 4.11 (br, 2H), 2.79 (br, 2H), 2.68 (br, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.4, 135.9, 129.5, 128.6, 127.5, 58.8, 57.0, 43.4, 27.2. IR (neat) 2961, 2895, 1469, 1411, 1346, 1274, 1220, 1120, 1021, 937, 867, 756, 696  $\text{cm}^{-1}$ . MS (ESI): 388  $[\text{M}+\text{Na}^+]$ . HRMS (ESI) calcd for  $\text{C}_{12}\text{H}_{15}\text{NNaSSe}_2$   $[\text{M}+\text{Na}^+]$  387.9149; found 387.9157.



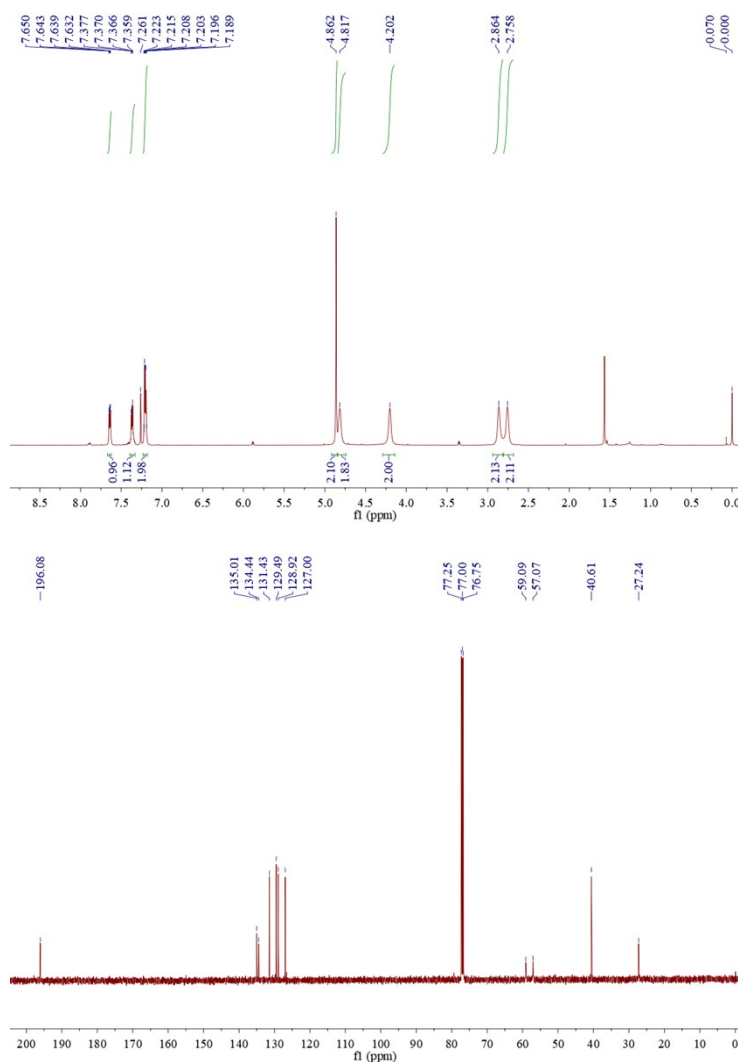


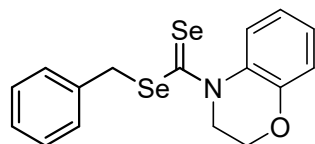
Compound **30**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (90.6 mg, 80%). m.p. 110.5-111.3 °C.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.30 (d,  $J = 8.0$  Hz, 2H), 7.11 (d,  $J = 8.0$  Hz, 2H), 4.81 (br, 2H), 4.65 (s, 2H), 4.18 (br, 2H), 2.86 (br, 2H), 2.75 (br, 2H), 2.32 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.6, 137.3, 132.6, 129.38, 129.35, 58.7, 57.0, 43.3, 27.2, 21.1. IR (neat) 2957, 2904, 1510, 1468, 1410, 1274, 1222, 1189, 1123, 1021, 942, 809, 717  $\text{cm}^{-1}$ . MS (ESI): 380  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_{13}\text{H}_{18}\text{NSSe}_2$   $[\text{M}+\text{H}^+]$  379.9486; found 379.9474.



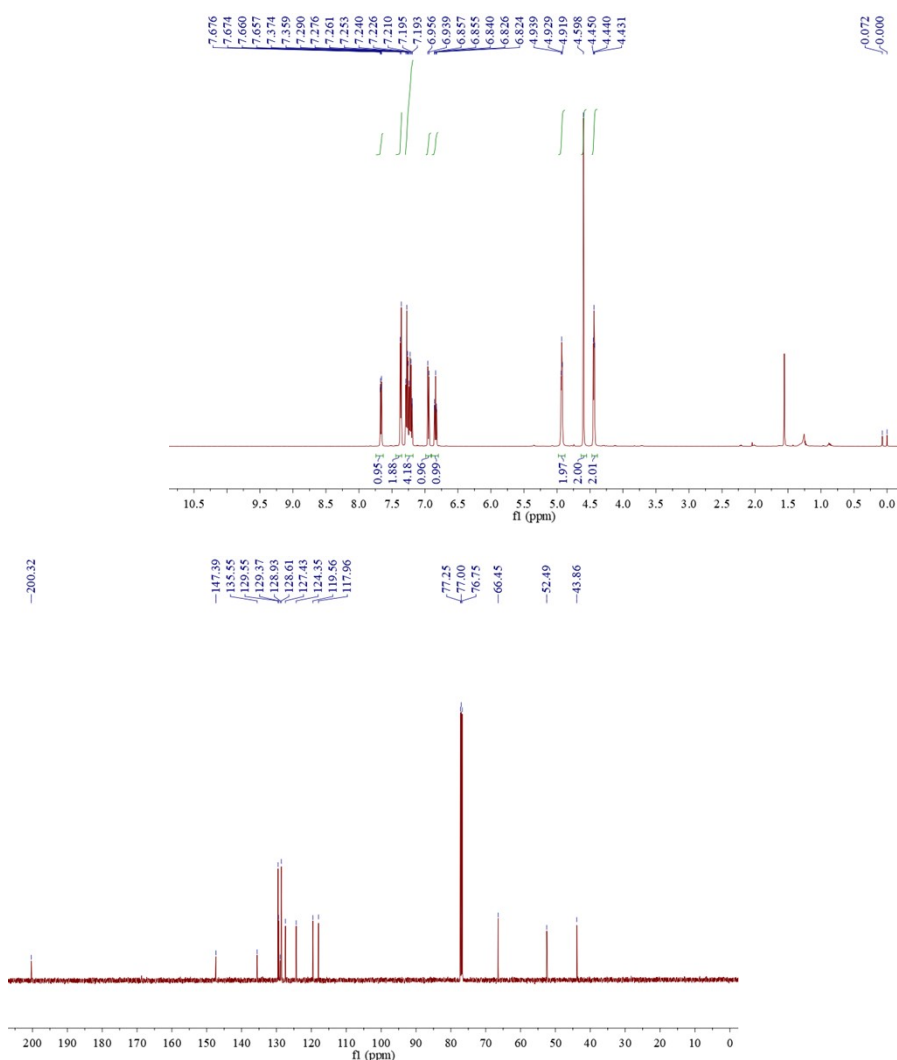


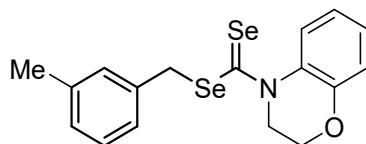
Compound **3p**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (106.2 mg, 89%). m.p. 88.6-90.4 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.64 (dd, *J* = 5.5, 3.5 Hz, 1H), 7.37 (dd, *J* = 5.5, 3.5 Hz, 1H), 7.22-7.19 (m, 2H), 4.86 (s, 2H), 4.82 (br, 4H), 4.20 (br, 2H), 2.86 (br, 2H), 2.76 (br, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 196.1, 135.0, 134.4, 131.4, 129.5, 128.9, 127.0, 59.1, 57.1, 40.6, 27.2. IR (neat) 2953, 2911, 1467, 1416, 1348, 1278, 1218, 1189, 1126, 1031, 944, 863, 758, 679 cm<sup>-1</sup>. MS: 422 [M+Na<sup>+</sup>]. HRMS (ESI) calcd for C<sub>12</sub>H<sub>14</sub>ClNNaSSe<sub>2</sub> [M+Na<sup>+</sup>] 421.8755; found 421.8771.



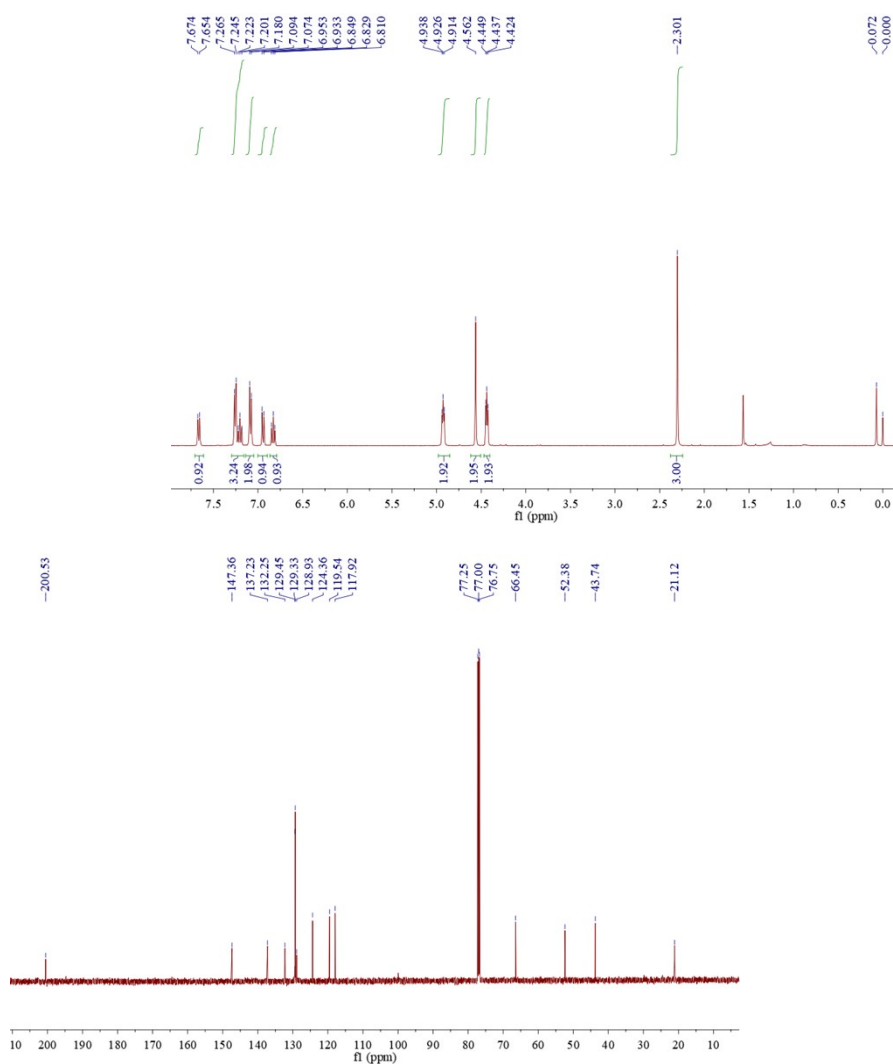


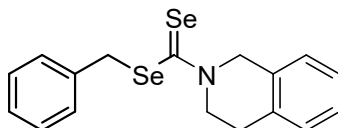
Compound **3q**: purification on silica gel (petroleum ether/ethyl acetate = 40:1) afforded the compound as yellow solid (100.8 mg, 85%). m.p. 115.9-116.0 °C.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.67 (dd,  $J = 8.0, 1.5$  Hz, 1H), 7.37 (d,  $J = 7.5$  Hz, 2H), 7.29-7.19 (m, 4H), 6.95 (d,  $J = 8.5$  Hz, 1H), 6.84 (td,  $J = 8.0, 1.0$  Hz, 1H), 4.93 (t,  $J = 5.0$  Hz, 2H), 4.60 (s, 2H), 4.44 (t,  $J = 5.0$  Hz, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  200.3, 147.4, 135.6, 129.6, 129.4, 128.9, 128.6, 127.4, 124.4, 119.6, 118.0, 66.5, 52.5, 43.9. IR (neat) 2982, 2935, 1579, 1490, 1440, 1370, 1256, 1209, 1047, 955, 822, 751  $\text{cm}^{-1}$ . MS (ESI): 398  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_{16}\text{H}_{16}\text{NOSe}_2$   $[\text{M}+\text{H}^+]$  397.9560; found 397.9546.



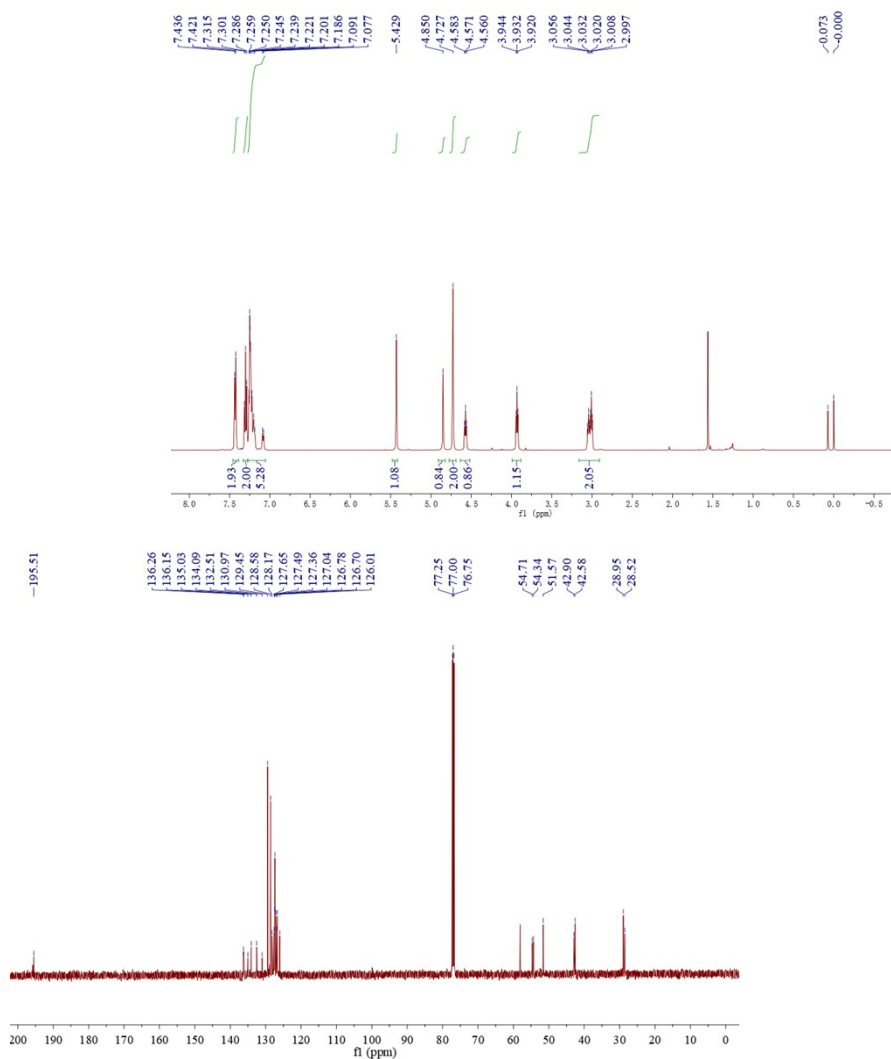


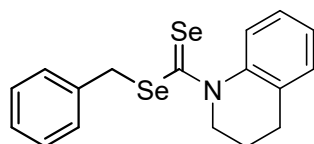
Compound **3r**: purification on silica gel (petroleum ether/ethyl acetate = 40:1) afforded the compound as yellow solid (92.1 mg, 75%). m.p. 110.9-111.0 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 7.66 (d, *J* = 8.0 Hz, 1H), 7.27-7.18 (m, 3H), 7.08 (d, *J* = 8.0 Hz, 2H), 6.94 (d, *J* = 8.0 Hz, 1H), 6.83 (t, *J* = 8.0 Hz, 1H), 4.93 (t, *J* = 4.8 Hz, 2H), 4.56 (s, 2H), 4.44 (t, *J* = 4.8 Hz, 2H), 2.30 (s, 3H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 200.5, 147.4, 137.2, 132.3, 129.5, 129.3, 128.9, 124.4, 119.5, 117.9, 66.5, 52.4, 43.7, 21.1. IR (neat) 2942, 1603, 1491, 1439, 1371, 1257, 1213, 1051, 958, 817, 753 cm<sup>-1</sup>. MS (ESI): 412 [M+H<sup>+</sup>]. HRMS (ESI) calcd for C<sub>17</sub>H<sub>18</sub>NOSe<sub>2</sub> [M+H<sup>+</sup>] 411.9716; found 411.9724.



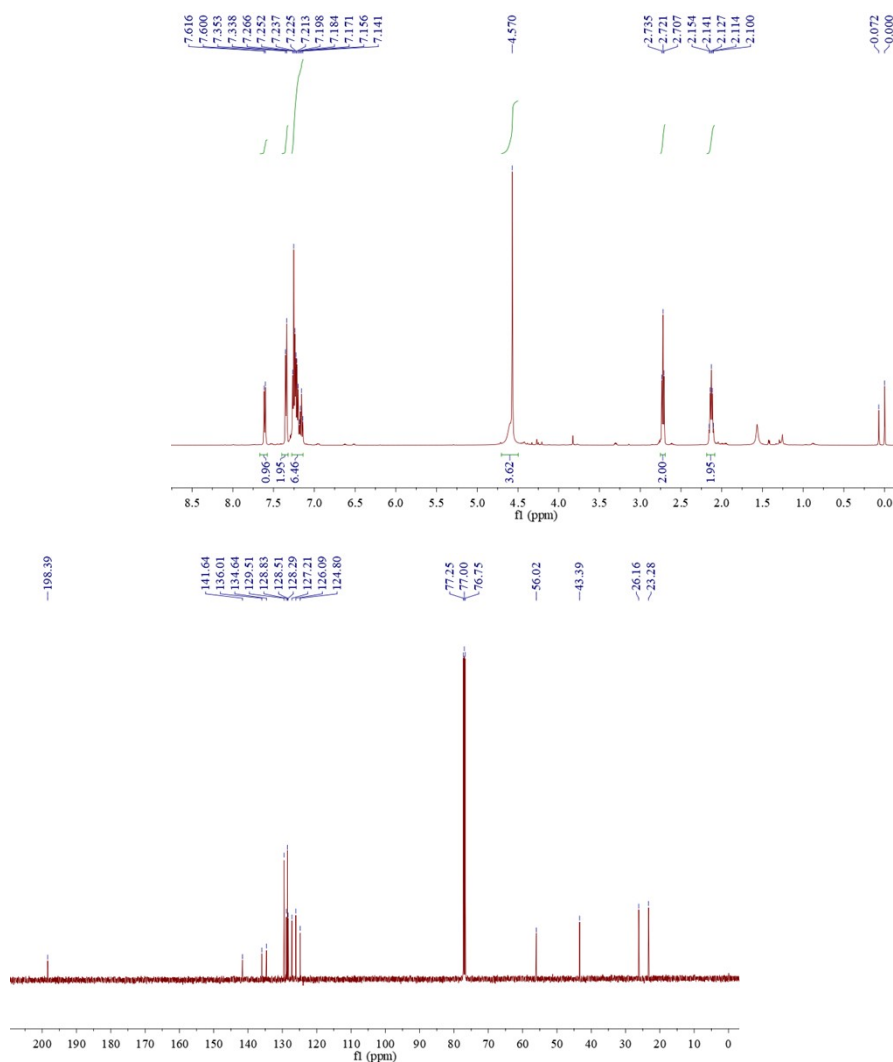


Compound **3s**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (95.6 mg, 81%). m.p. 94.9-95.3 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.43 (d, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.26-7.18 (m, 5H), 5.43 (s, 1H), 4.85 (s, 1H), 4.73 (s, 2H), 4.57 (t, *J* = 6.0 Hz, 1H), 3.93 (t, *J* = 6.0 Hz, 1H), 3.06-3.00 (m, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 195.5, 136.3, 136.2, 135.0, 134.1, 132.5, 131.0, 129.5, 128.6, 128.2, 127.7, 127.5, 127.4, 127.0, 126.8, 126.7, 126.0, 54.7, 54.3, 51.6, 42.9, 42.6, 29.0, 28.5. IR (neat) 2918, 1494, 1414, 1281, 1230, 1145, 1108, 1039, 921, 883, 748, 690 cm<sup>-1</sup>. MS (ESI): 418 [M+H<sup>+</sup>]. HRMS (ESI) calcd for C<sub>17</sub>H<sub>17</sub>NNaSe<sub>2</sub> [M+H<sup>+</sup>] 417.9587; found 417.9585.

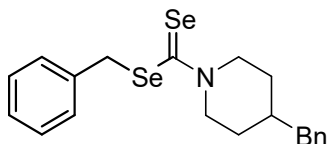




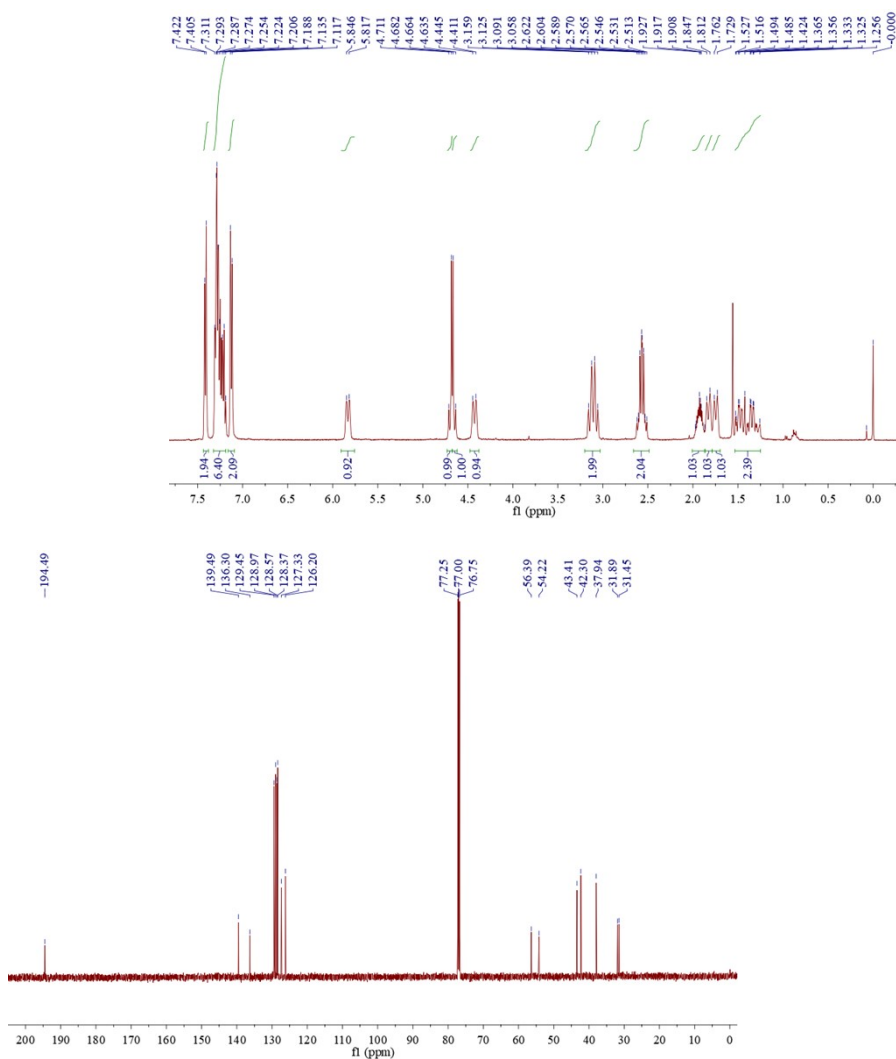
Compound **3t**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (74.3 mg, 63%). m.p. 91.2-93.0 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.61 (d, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 7.5 Hz, 2H), 7.27-7.14 (m, 6H), 4.57 (s, 4H), 2.72 (t, *J* = 7.0 Hz, 2H), 2.13 (pent, *J* = 7.0 Hz, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 198.4, 141.6, 136.0, 134.6, 129.5, 128.8, 128.5, 128.3, 127.2, 126.1, 124.8, 56.0, 43.4, 26.2, 23.3. IR (neat) 2950, 2931, 1486, 1449, 1377, 1255, 1169, 1108, 1062, 1027, 929, 825, 758, 696 cm<sup>-1</sup>. MS (ESI): 418 [M+Na<sup>+</sup>]. HRMS (ESI) calcd for C<sub>17</sub>H<sub>17</sub>NNaSe<sub>2</sub> [M+Na<sup>+</sup>] 417.9587; found 417.9583.

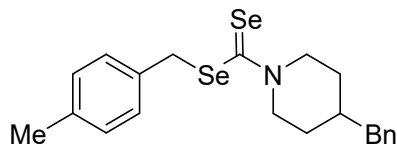




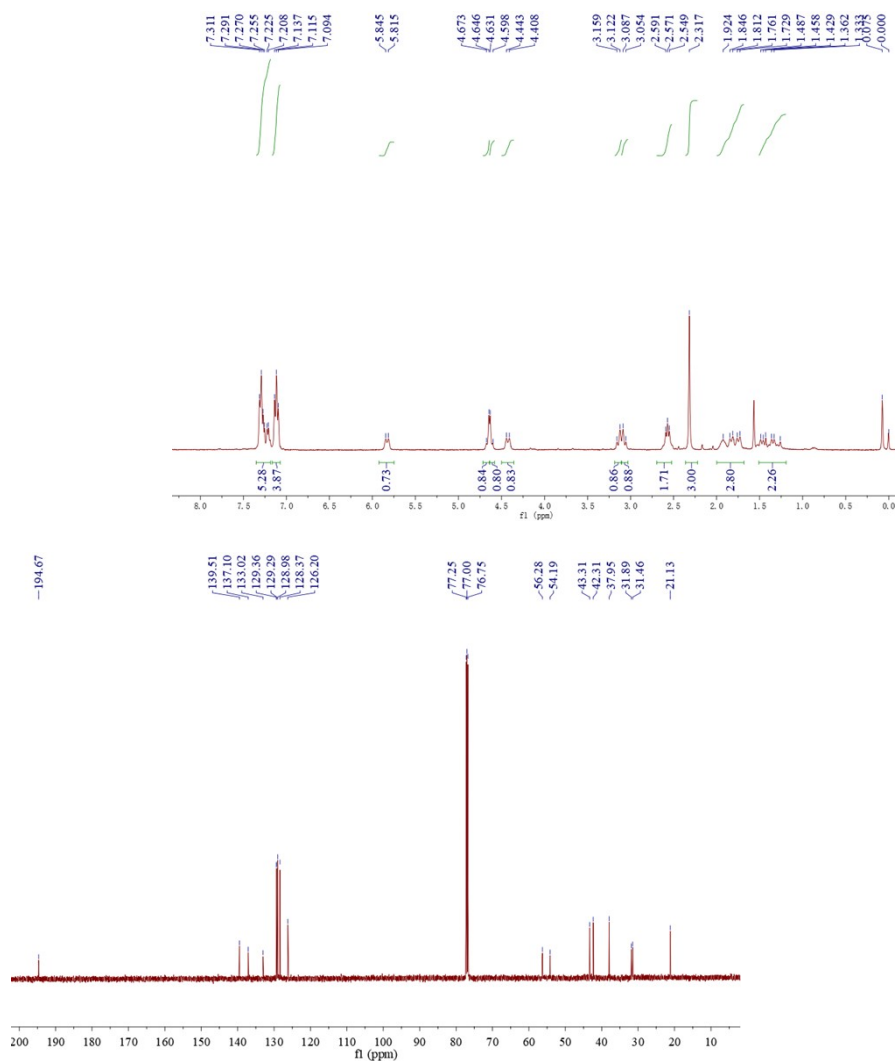


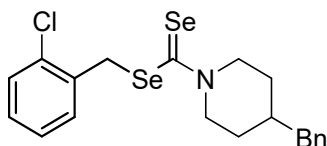
Compound **3u**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (86.2 mg, 66%). m.p. 84.3-84.5 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 7.41 (d, *J* = 6.8 Hz, 2H), 7.31-7.19 (m, 6H), 7.13 (d, *J* = 7.2 Hz, 2H), 5.83 (d, *J* = 11.6 Hz, 1H), 4.70 (d, *J* = 11.6 Hz, 1H), 4.65 (d, *J* = 11.6 Hz, 1H), 4.43 (d, *J* = 13.6 Hz, 1H), 3.11 (q, *J* = 13.6 Hz, 2H), 2.62-2.51 (m, 2H), 1.97-1.87 (m, 1H), 1.83 (d, *J* = 14.0 Hz, 1H), 1.75 (d, *J* = 13.2 Hz, 1H), 1.52-1.26 (m, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 194.5, 139.5, 136.3, 129.5, 129.0, 128.6, 128.4, 127.3, 126.2, 56.4, 54.2, 43.4, 42.3, 37.9, 31.9, 31.5. IR (neat) 2914, 2844, 1482, 1429, 1355, 1253, 1229, 1142, 1053, 964, 866, 809, 744, 694 cm<sup>-1</sup>. MS (ESI): 460 [M+Na<sup>+</sup>]. HRMS (ESI) calcd for C<sub>20</sub>H<sub>23</sub>NNaSe<sub>2</sub> [M+Na<sup>+</sup>] 460.0057; found 460.0047.



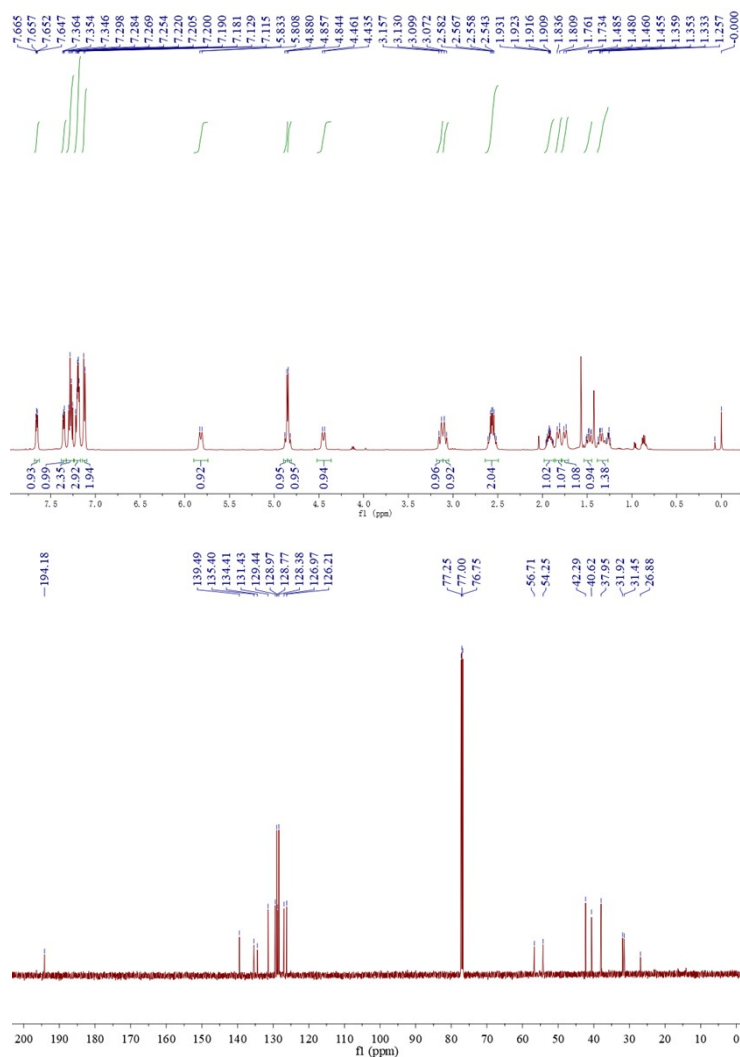


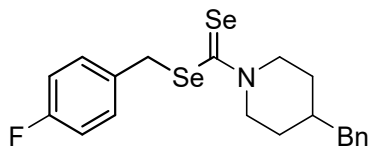
Compound **3v**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (87.6 mg, 65%). m.p. 84.8-85.3 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 7.31-7.21 (m, 5H), 7.12 (t, *J* = 8.8 Hz, 4H), 5.83 (d, *J* = 12.0 Hz, 1H), 4.66 (d, *J* = 10.8 Hz, 1H), 4.61 (d, *J* = 13.2 Hz, 1H), 4.43 (d, *J* = 14.0 Hz, 1H), 3.14 (d, *J* = 14.8 Hz, 1H), 3.07 (d, *J* = 13.2 Hz, 1H), 2.57 (t, *J* = 8.0 Hz, 2H), 2.32 (s, 3H), 1.92-1.73 (m, 3H), 1.49-1.26 (m, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 194.7, 139.5, 137.1, 133.0, 129.4, 129.3, 129.0, 128.4, 126.2, 56.3, 54.2, 43.3, 42.3, 38.0, 31.9, 31.5, 21.1. IR (neat) 2901, 1514, 1475, 1429, 1353, 1250, 1070, 963, 813, 744, 679 cm<sup>-1</sup>. MS (ESI): 452 [M+H<sup>+</sup>]. HRMS (ESI) calcd for C<sub>21</sub>H<sub>26</sub>NSe<sub>2</sub> [M+H<sup>+</sup>] 452.0394; found 452.0401.



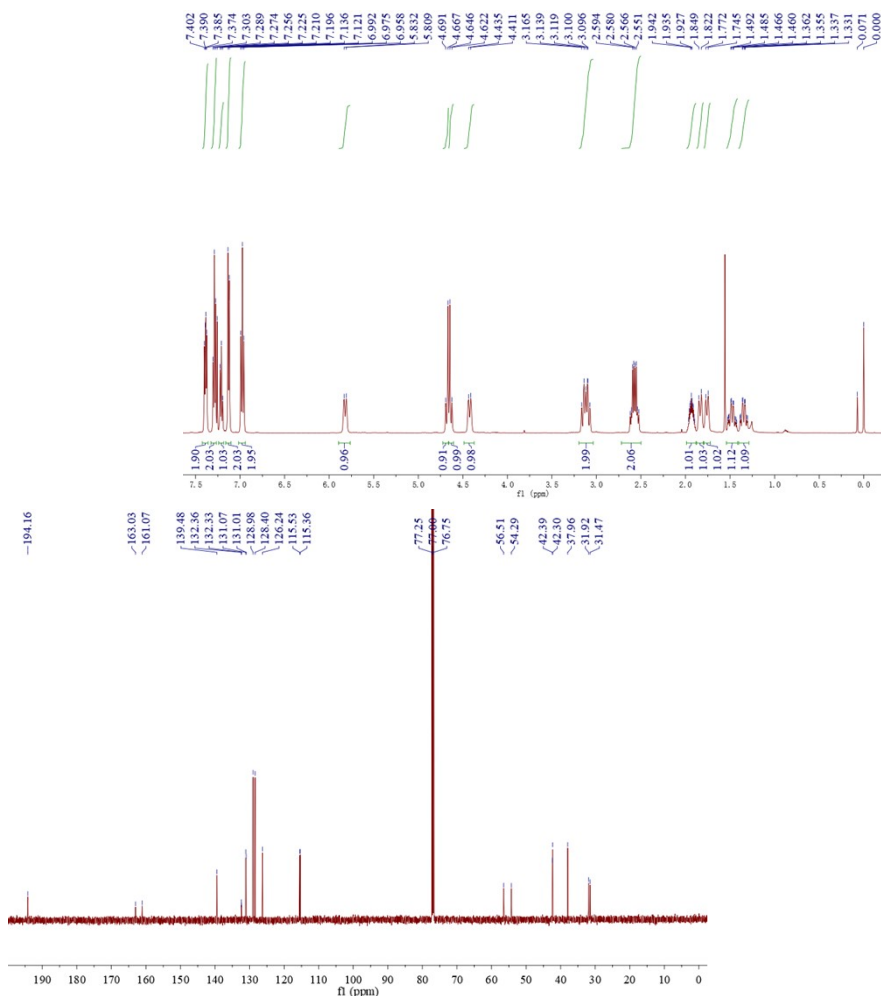


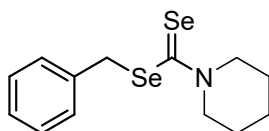
Compound **3w**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow liquid (98.7 mg, 70%).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.67-7.65 (m, 1H), 7.36-7.35 (m, 1H), 7.28 (t,  $J = 7.5$  Hz, 2H), 7.22-7.18 (m, 3H), 7.12 (d,  $J = 7.0$  Hz, 2H), 5.82 (d,  $J = 12.5$  Hz, 1H), 4.87 (d,  $J = 12.0$  Hz, 1H), 4.83 (d,  $J = 12.0$  Hz, 1H), 4.45 (d,  $J = 13.0$  Hz, 1H), 3.14 (d,  $J = 13.5$  Hz, 1H), 3.09 (d,  $J = 13.5$  Hz, 1H), 2.61-2.52 (m, 2H), 1.96-1.88 (m, 1H), 1.82 (d,  $J = 13.5$  Hz, 1H), 1.75 (d,  $J = 13.5$  Hz, 1H), 1.51-1.46 (m, 1H), 1.38-1.24 (m, 1H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  194.2, 139.5, 135.4, 134.4, 131.4, 129.4, 129.0, 128.8, 128.4, 127.0, 126.2, 56.7, 54.3, 42.3, 40.6, 38.0, 31.9, 31.5, 26.9. IR (neat) 2914, 2874, 1473, 1427, 1356, 1226, 1177, 1051, 961, 856, 749, 699  $\text{cm}^{-1}$ . MS (ESI): 472  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_{20}\text{H}_{23}\text{ClINSe}_2$   $[\text{M}+\text{H}^+]$  471.9843; found 471.9849.



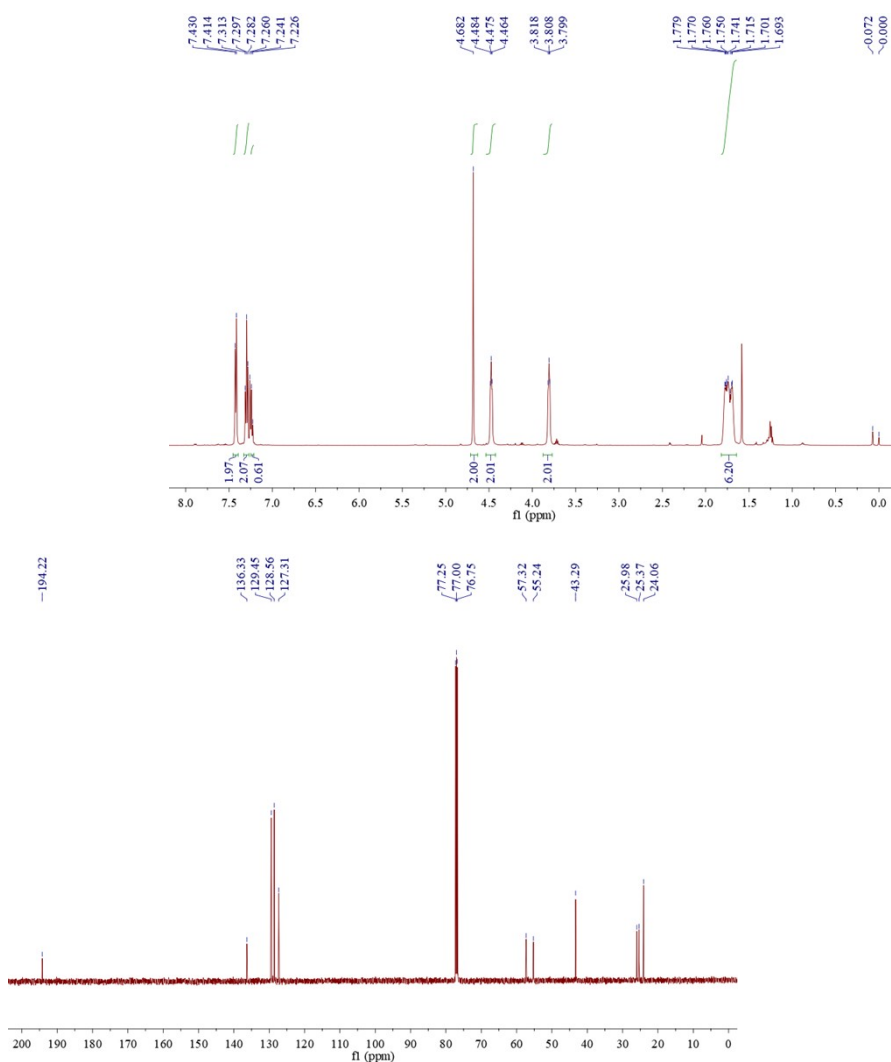


Compound **3x**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as white solid (102.0 mg, 75%). m.p. 96.8-97.2 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.39 (dd, *J* = 8.0, 5.5 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.13 (d, *J* = 7.5 Hz, 2H), 6.98 (t, *J* = 8.5 Hz, 2H), 5.82 (d, *J* = 11.5 Hz, 1H), 4.68 (d, *J* = 12.0 Hz, 1H), 4.63 (d, *J* = 12.0 Hz, 1H), 4.42 (d, *J* = 12.0 Hz, 1H), 3.17-3.07 (m, 2H), 2.62-2.53 (m, 2H), 1.97-1.90 (m, 1H), 1.84 (d, *J* = 13.5 Hz, 1H), 1.76 (d, *J* = 13.5 Hz, 1H), 1.53-1.43 (m, 1H), 1.39-1.31 (m, 1H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 194.2, 162.1 (d, *J*<sub>C-F</sub> = 245.0 Hz), 139.5, 132.3 (d, *J*<sub>C-F</sub> = 3.75 Hz), 131.0 (d, *J*<sub>C-F</sub> = 7.5 Hz), 129.0, 128.4, 126.2, 115.4 (d, *J*<sub>C-F</sub> = 21.25 Hz), 56.5, 54.3, 42.4, 42.3, 38.0, 31.9, 31.5. <sup>19</sup>F NMR (470 MHz, CDCl<sub>3</sub>) δ -114.84. IR (neat) 2928, 2859, 1597, 1483, 1435, 1355, 1219, 1147, 1086, 960, 837, 746, 697 cm<sup>-1</sup>. MS (ESI): 456 [M+H<sup>+</sup>]. HRMS (ESI) calcd for C<sub>20</sub>H<sub>23</sub>FNSe<sub>2</sub> [M+H<sup>+</sup>] 456.0143; found 456.0147.

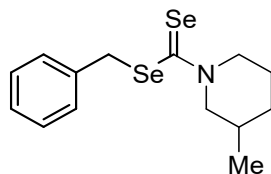




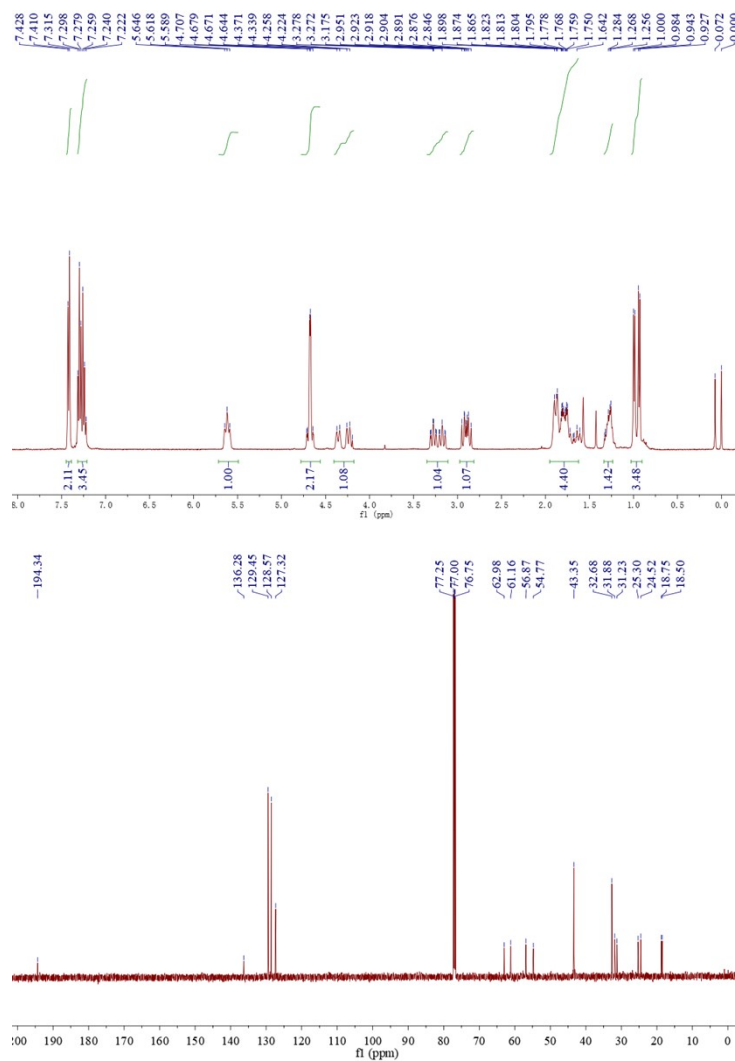
Compound **3y**<sup>1</sup>: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (63.2 mg, 61%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 7.5 Hz, 2H), 7.23 (d, *J* = 7.5 Hz, 1H), 4.68 (s, 2H), 4.48 (t, *J* = 4.5 Hz, 2H), 3.81 (t, *J* = 4.5 Hz, 2H), 1.78-1.68 (m, 6H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 194.2, 136.3, 129.5, 128.6, 127.3, 57.3, 55.2, 43.3, 26.0, 25.4, 24.1.

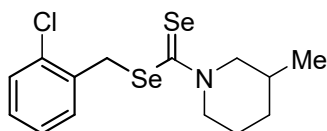


(1) Pan, X.-Q.; Zhu, J.; Zou, J.-P.; Zhang, Z.-B.; Cheng, Z.-P.; Zhou, N.-C.; Zhang, W.; Zhu, X.-L. *Org. Lett.* **2012**, *14*, 6170-6173.

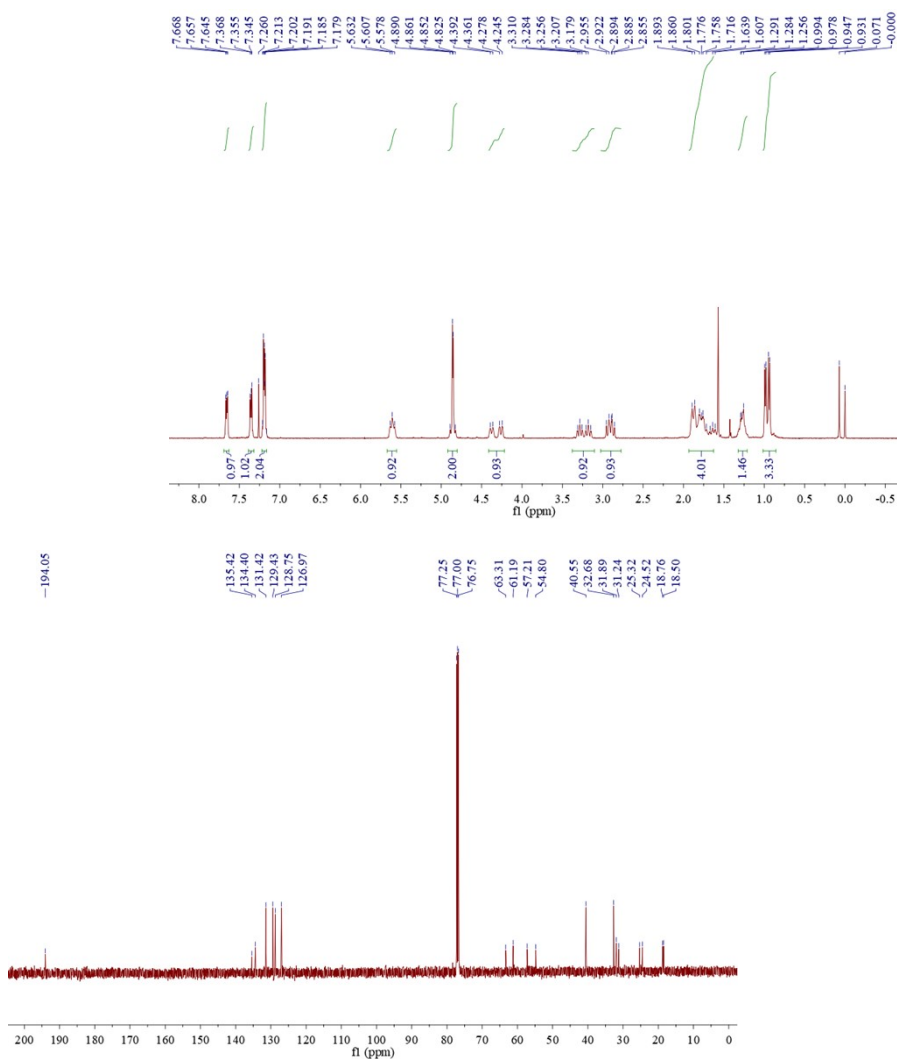


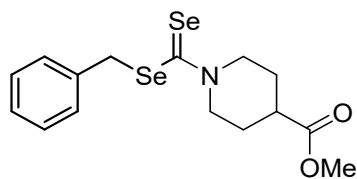
Compound **3z**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as brown solid (71.1 mg, 66%). m.p. 61.2-61.5 °C. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, TMS) δ 7.42 (d, *J* = 7.2 Hz, 2H), 7.32-7.22 (m, 3H), 5.62 (t, *J* = 11.2 Hz, 1H), 4.71-4.64 (m, 2H), 4.37-4.20 (m, 1H), 3.31-3.14 (m, 1H), 2.95-2.85 (m, 1H), 1.90-1.61 (m, 4H), 1.33-1.26 (m, 1H), 0.96 (dd, *J* = 22.8, 6.4 Hz, 3H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 194.3, 136.3, 129.5, 128.6, 127.3, 63.0, 61.2, 56.9, 54.8, 43.4, 32.7, 31.9, 31.2, 25.3, 24.5, 18.8, 18.5. IR (neat) 2933, 2847, 1478, 1427, 1282, 1229, 1102, 965, 874, 810, 755, 692 cm<sup>-1</sup>. MS (ESI): 362 [M+H<sup>+</sup>]. HRMS (ESI) calcd for C<sub>14</sub>H<sub>20</sub>NSe<sub>2</sub> [M+H<sup>+</sup>] 361.9923; found 361.9913.



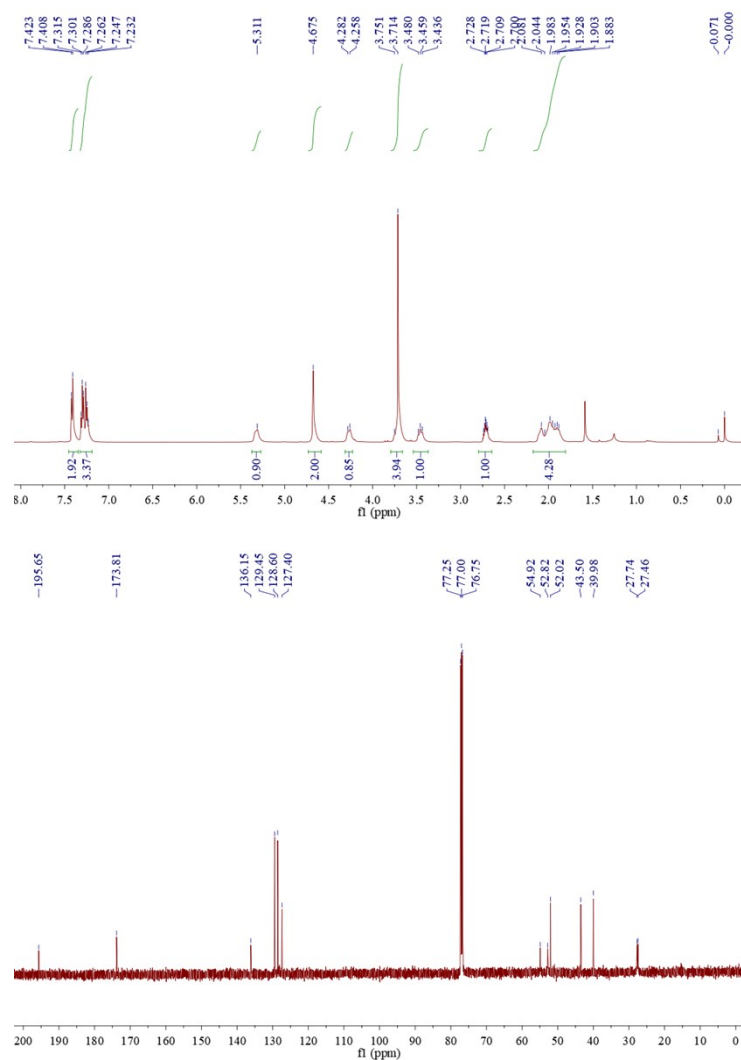


Compound **3aa**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as brown liquid (83.9 mg, 71%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.67-7.65 (m, 1H), 7.37-7.25 (m, 1H), 7.21-7.16 (m, 2H), 5.61 (t,  $J = 10.8$  Hz, 1H), 4.86 (dd,  $J = 15.2, 11.2$  Hz, 2H), 4.39-4.25 (m, 1H), 3.31-3.15 (m, 1H), 2.96-2.86 (m, 1H), 1.89-1.64 (m, 4H), 1.29-1.26 (m, 1H), 0.96 (dd,  $J = 18.8, 6.4$  Hz, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  194.1, 135.4, 134.4, 131.4, 129.4, 128.8, 127.0, 63.3, 61.2, 57.2, 54.8, 40.6, 32.7, 31.9, 31.2, 25.3, 24.5, 18.8, 18.5. IR (neat) 2926, 2850, 1471, 1425, 1227, 1137, 1084, 1033, 964, 902, 847, 752, 680  $\text{cm}^{-1}$ . HRMS (ESI): 418  $[\text{M}+\text{Na}^+]$ . HRMS (ESI) calcd for  $\text{C}_{14}\text{H}_{18}\text{ClNNaSe}_2$   $[\text{M}+\text{Na}^+]$  417.9348; found 417.9354.

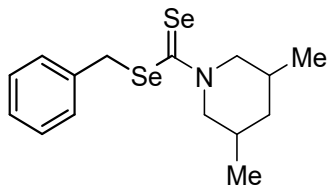




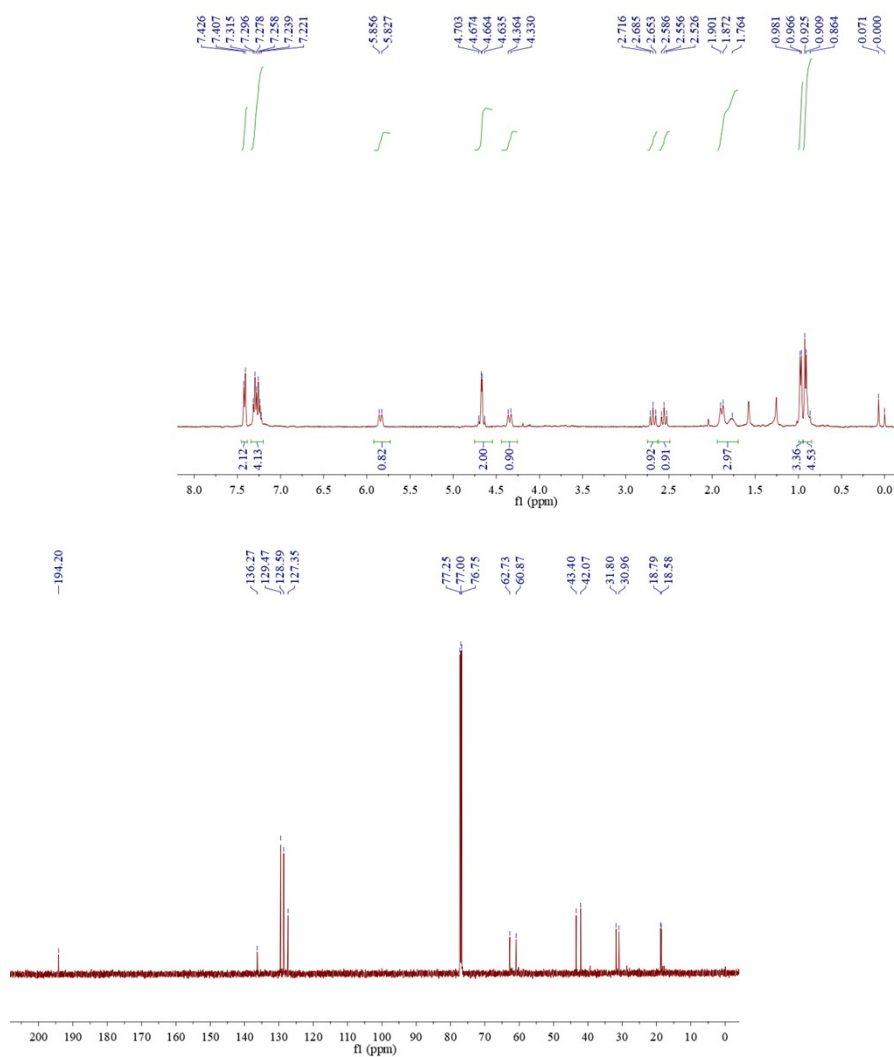
Compound **3ab**: purification on silica gel (petroleum ether/ethyl acetate = 15:1) afforded the compound as yellow liquid (53.2 mg, 44%).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.42 (d,  $J = 7.5$  Hz, 2H), 7.32-7.23 (m, 3H), 5.31 (s, 1H), 4.68 (s, 2H), 4.28-4.26 (m, 1H), 3.75-3.71 (m, 4H), 3.48-3.44 (m, 1H), 2.74-2.69 (m, 1H), 2.08-1.88 (m, 4H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  195.7, 173.8, 136.2, 129.5, 128.6, 127.4, 54.9, 52.8, 52.0, 43.5, 40.0, 27.7, 27.5. IR (neat) 2950, 2923, 2851, 1720, 1423, 1307, 1255, 1199, 1175, 1027, 970, 865, 757, 695  $\text{cm}^{-1}$ . MS (ESI): 428  $[\text{M}+\text{Na}^+]$ . HRMS (ESI) calcd for  $\text{C}_{15}\text{H}_{19}\text{NNaO}_2\text{Se}_2$   $[\text{M}+\text{Na}^+]$  427.9641; found 427.9648.

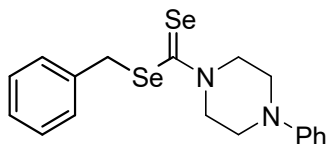




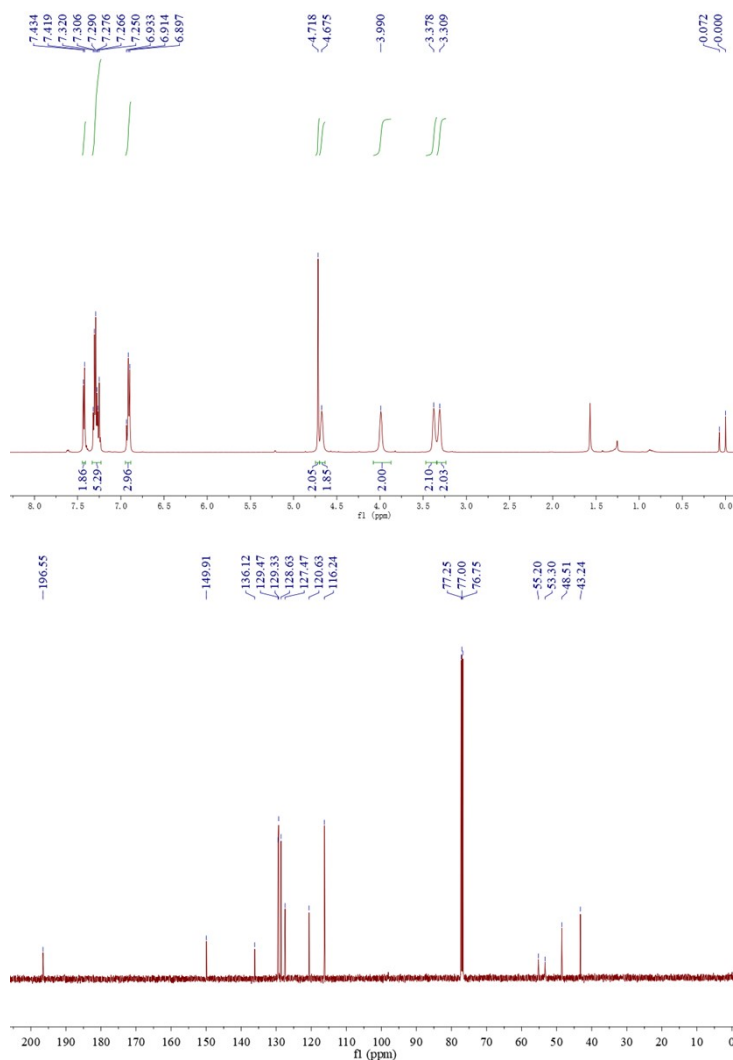


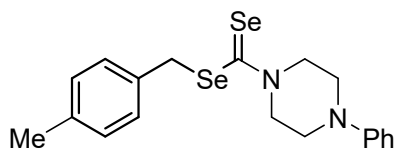
Compound **3ac**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow liquid (66.1 mg, 59%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.42 (d,  $J = 7.6$  Hz, 2H), 7.32-7.22 (m, 3H), 5.84 (d,  $J = 11.6$  Hz, 1H), 4.69 (d,  $J = 11.6$  Hz, 1H), 4.65 (d,  $J = 11.6$  Hz, 1H), 4.35 (d,  $J = 13.6$  Hz, 1H), 2.69 (t,  $J = 12.6$  Hz, 1H), 2.56 (t,  $J = 12.0$  Hz, 1H), 1.90-1.76 (m, 3H), 0.97 (d,  $J = 6.0$  Hz, 3H), 0.93-0.86 (m, 4H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  194.2, 136.3, 129.5, 128.6, 127.4, 62.7, 60.9, 43.4, 42.1, 31.8, 31.0, 18.8, 18.6. IR (neat) 2953, 2907, 2869, 1473, 1425, 1282, 1232, 1101, 965, 885, 830, 757, 695  $\text{cm}^{-1}$ . MS (ESI): 398  $[\text{M}+\text{Na}^+]$ . HRMS (ESI) calcd for  $\text{C}_{15}\text{H}_{21}\text{NNaSe}_2$   $[\text{M}+\text{Na}^+]$  397.9899; found 397.9885.



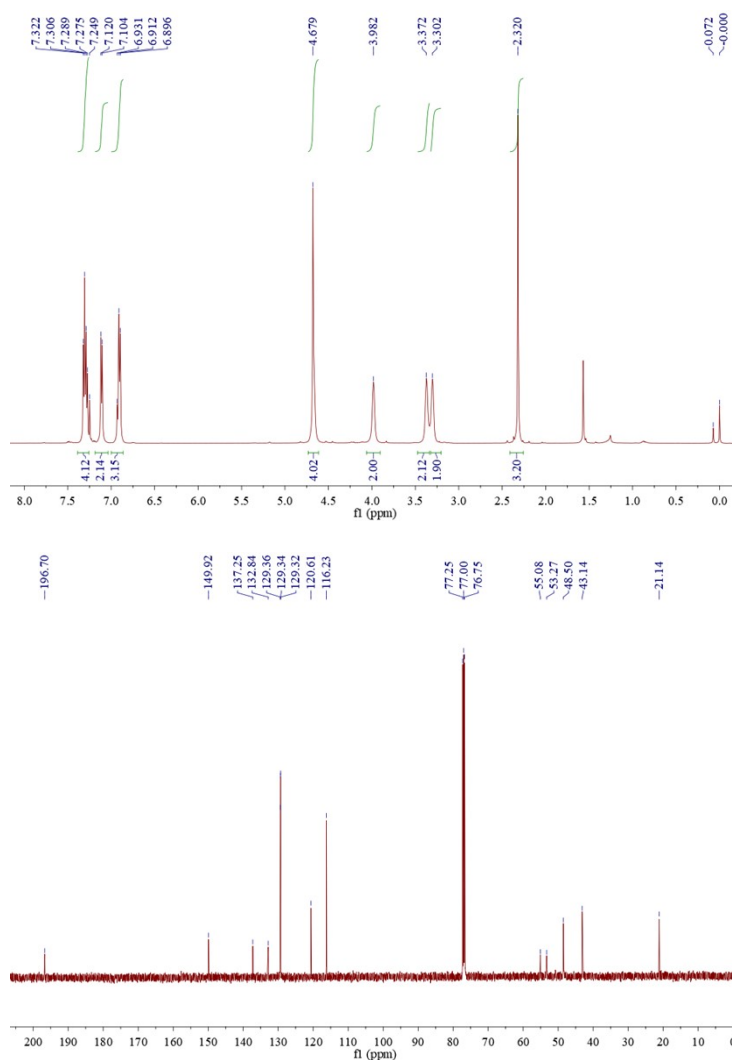


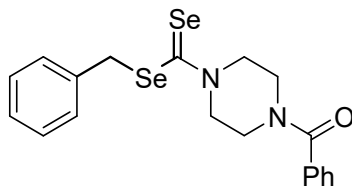
Compound **3ad**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (86.1 mg, 68%). m.p. 71.6-72.0 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.43 (d, *J* = 7.5 Hz, 2H), 7.32-7.25 (m, 5H), 6.93-6.90 (m, 3H), 4.72 (s, 2H), 4.68 (br, 2H), 3.99 (br, 2H), 3.38 (br, 2H), 3.31 (br, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 196.6, 149.9, 136.1, 129.5, 129.3, 128.6, 127.5, 120.6, 116.2, 55.2, 53.3, 48.5, 43.2. IR (neat) 2914, 2819, 1738, 1599, 1492, 1410, 1333, 1219, 1142, 1011, 926, 856, 756, 690 cm<sup>-1</sup>. MS (ESI): 425 [M+H<sup>+</sup>]. HRMS (ESI) calcd for C<sub>18</sub>H<sub>21</sub>N<sub>2</sub>Se<sub>2</sub> [M+H<sup>+</sup>] 425.0033; found 425.0035.



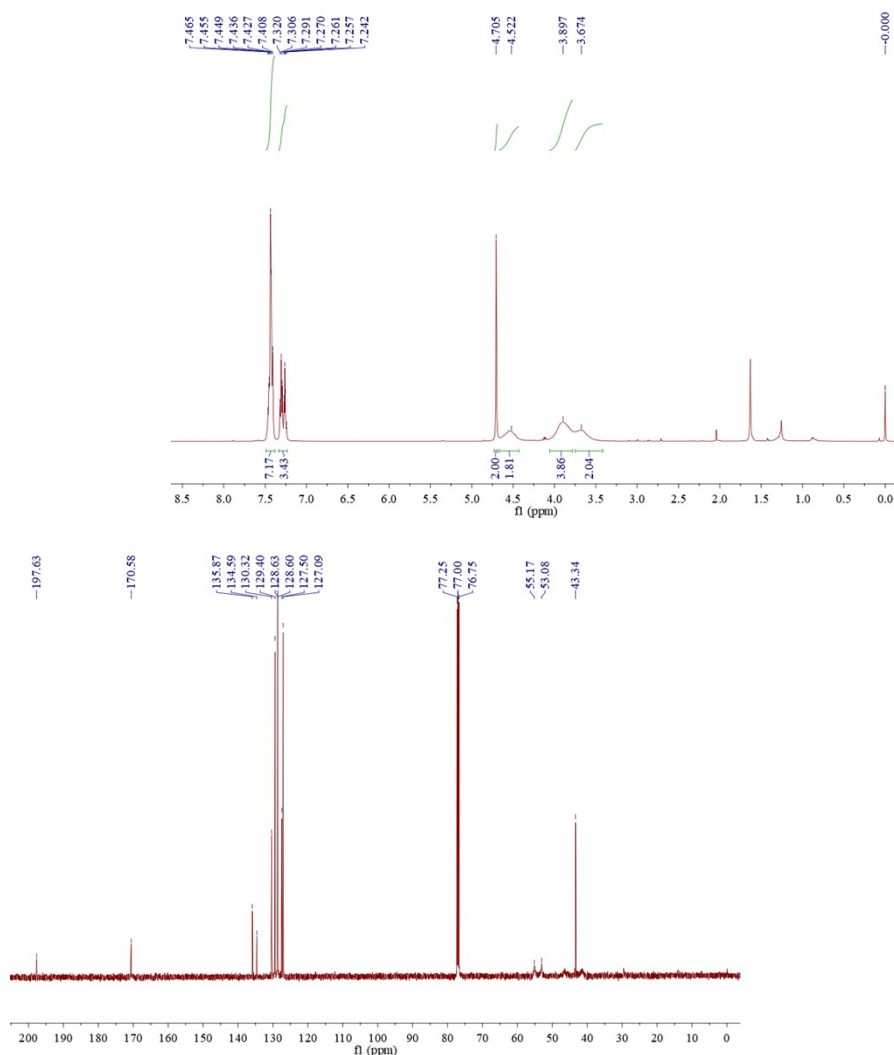


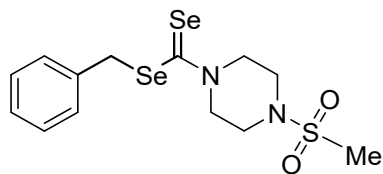
Compound **3ae**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (94.2 mg, 72%). m.p. 102.9-103.1 °C.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.32-7.28 (m, 4H), 7.11 (d,  $J = 8.0$  Hz, 2H), 6.93-6.90 (m, 3H), 4.68 (br, 4H), 3.98 (br, 2H), 3.37 (br, 2H), 3.30 (br, 2H), 2.32 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.7, 149.9, 137.3, 132.8, 129.4, 129.34, 129.32, 120.6, 116.2, 55.1, 53.3, 48.5, 43.1, 21.1. IR (neat) 2919, 2815, 1601, 1469, 1421, 1375, 1266, 1213, 1134, 1008, 915, 861, 816, 755, 689  $\text{cm}^{-1}$ . MS (ESI): 439  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_{19}\text{H}_{22}\text{N}_2\text{Se}_2$   $[\text{M}+\text{H}^+]$  439.0189; found 439.0175.



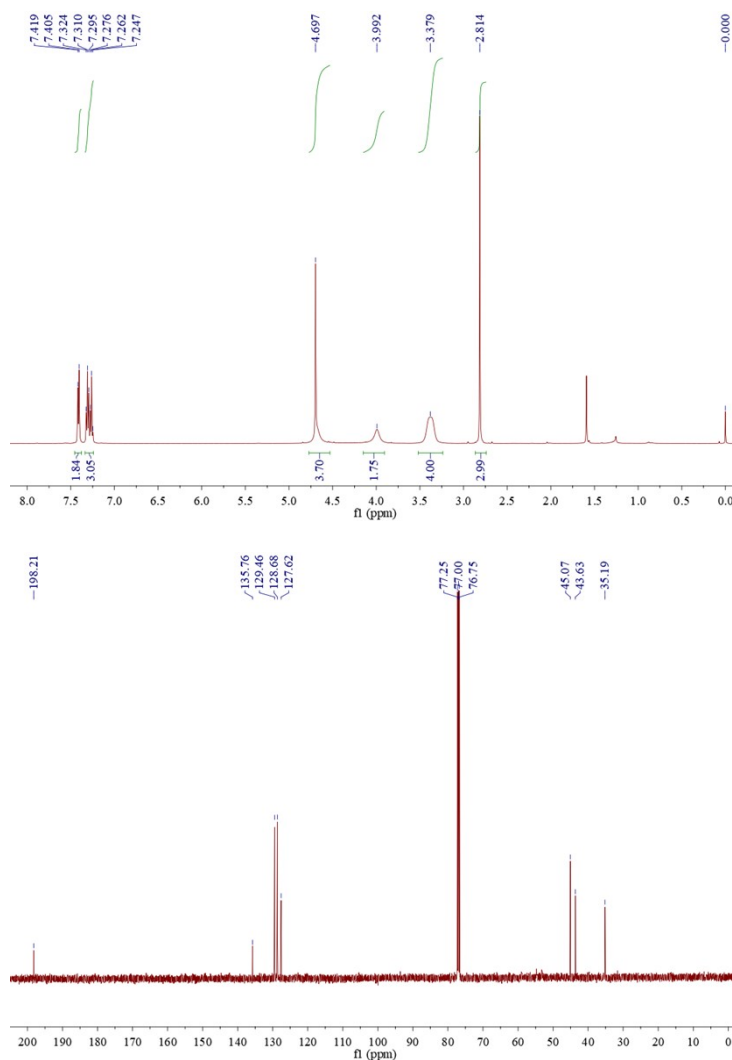


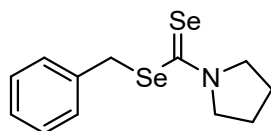
Compound **3af**: purification on silica gel (petroleum ether/ethyl acetate = 5:1) afforded the compound as yellow solid (72.9 mg, 54%). m.p. 137.0-137.5 °C.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.47-7.41 (m, 7H), 7.32-7.24 (m, 3H), 4.71 (s, 2H), 4.52 (br, 2H), 3.90 (br, 4H), 3.67 (br, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  197.6, 170.6, 135.9, 134.6, 130.3, 129.4, 128.63, 128.60, 127.5, 127.1, 55.2, 53.1, 43.4. IR (neat) 2922, 2861, 1623, 1418, 1280, 1218, 1150, 1005, 870, 759, 693  $\text{cm}^{-1}$ . MS (ESI): 475  $[\text{M}+\text{Na}^+]$ . HRMS (ESI) calcd for  $\text{C}_{19}\text{H}_{20}\text{N}_2\text{NaOSe}_2$   $[\text{M}+\text{Na}^+]$  474.9802; found 474.9799.



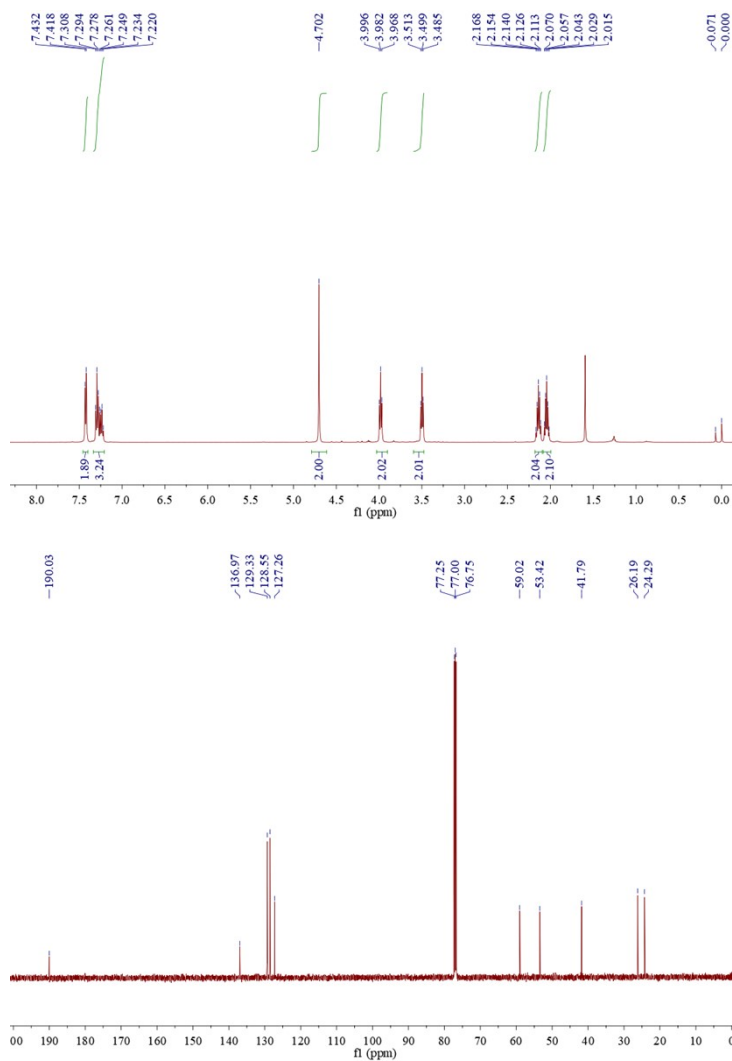


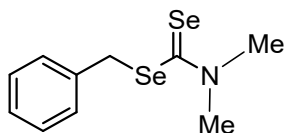
Compound **3ag**: purification on silica gel (petroleum ether/ethyl acetate = 5:1) afforded the compound as yellow solid (94.2 mg, 74%). m.p. 145.5-146.0 °C.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.41 (d,  $J = 7.0$  Hz, 2H), 7.32-7.25 (m, 3H), 4.70 (s, 4H), 3.99 (br, 2H), 3.38 (br, 4H), 2.81 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  198.2, 135.8, 129.5, 128.7, 127.6, 45.1, 43.6, 35.2. IR (neat) 2924, 2861, 1739, 1449, 1418, 1329, 1269, 1220, 1150, 1021, 952, 864, 781, 692  $\text{cm}^{-1}$ . MS (ESI): 427  $[\text{M}+\text{H}^+]$ . HRMS (ESI) calcd for  $\text{C}_{13}\text{H}_{19}\text{N}_2\text{O}_2\text{SSe}_2$   $[\text{M}+\text{H}^+]$  426.9493; found 426.9486.



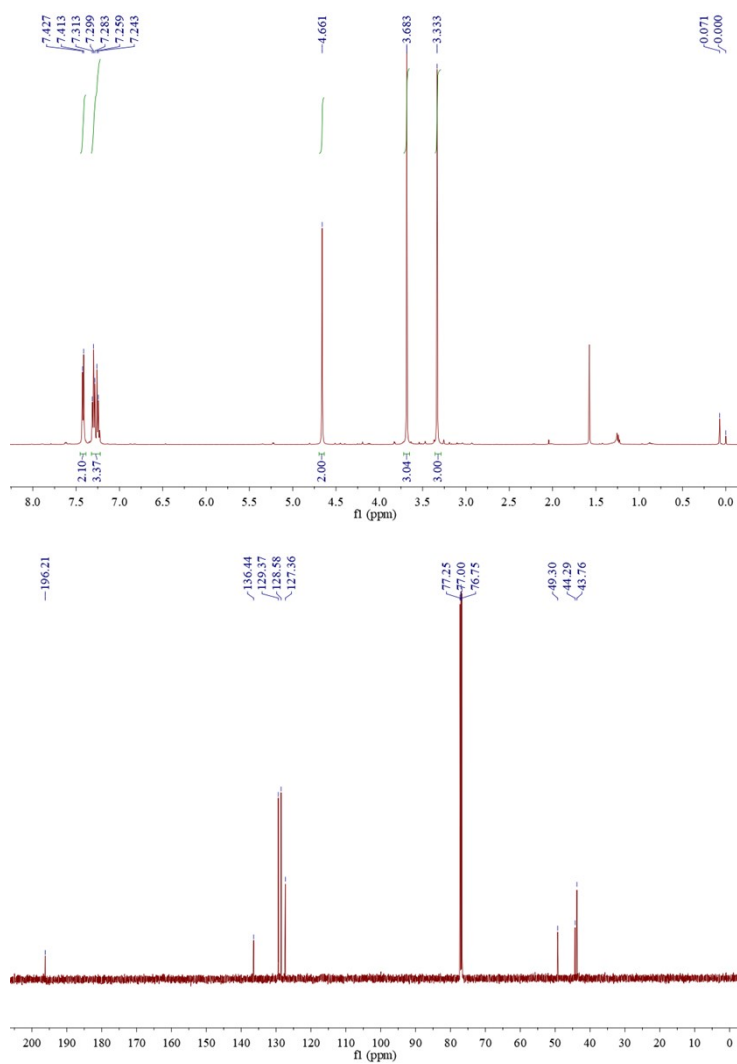


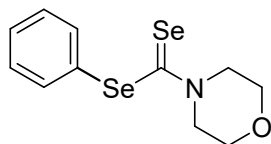
Compound **3ah**<sup>1</sup>: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as brown liquid (40.7 mg, 41%). <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.43 (d, *J* = 7.0 Hz, 2H), 7.31-7.22 (m, 3H), 4.70 (s, 2H), 3.98 (t, *J* = 7.0 Hz, 2H), 3.50 (t, *J* = 7.0 Hz, 2H), 2.14 (pent, *J* = 7.0 Hz, 2H), 2.04 (pent, *J* = 7.0 Hz, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 190.0, 137.0, 129.3, 128.6, 127.3, 59.0, 53.4, 41.8, 26.2, 24.3.





Compound **3ai**: purification on silica gel (petroleum ether/ethyl acetate = 20:1) afforded the compound as yellow solid (40.3 mg, 44%).  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ , TMS)  $\delta$  7.42 (d,  $J = 7.0$  Hz, 2H), 7.31-7.24 (m, 3H), 4.66 (s, 2H), 3.68 (s, 3H), 3.33 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  196.2, 136.4, 129.4, 128.6, 127.4, 49.3, 44.3, 43.8.





Compound **3aj**: purification on silica gel (petroleum ether/ethyl acetate =10:1) afforded the compound as brown solid (58.0 mg, 58%). m.p. 168.2-170.2 °C. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>, TMS) δ 7.59 (d, *J* = 7.5 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 4.51 (s, 2H), 3.94 (br, 2H), 3.84 (br, 2H), 3.81 (br, 2H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 197.8, 137.8, 131.6, 130.0, 129.5, 66.4, 65.9, 56.2, 54.9. IR (neat) 2963, 2919, 2845, 1458, 1417, 1264, 1225, 1109, 1019, 945, 845, 769, 744, 685 cm<sup>-1</sup>. MS (ESI): 358 [M+Na<sup>+</sup>]. HRMS (ESI) calcd for C<sub>11</sub>H<sub>13</sub>NNaOSe<sub>2</sub> [M+Na<sup>+</sup>] 357.9222; found 357.9231.

