

Supporting Information

Visible-light-mediated selenylation/cyclization reactions of diselenide with acrylimide derivatives: Synthesis of selenosubstituted pyrrolidine-2,5-diones

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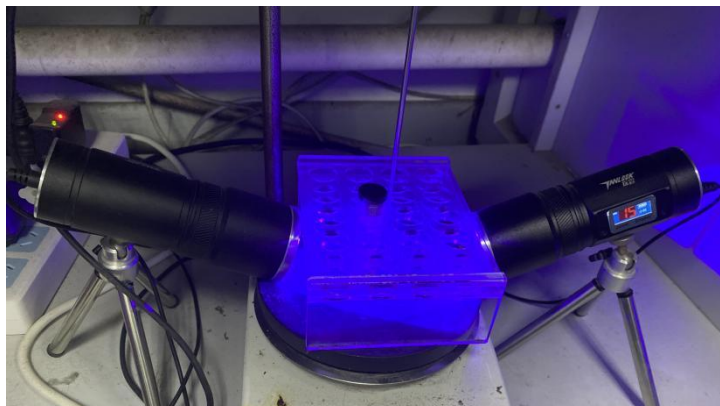
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1. General Information

Unless otherwise stated, all commercially available compounds are used as specified without further purification. The solvents used for chromatographic analysis were of analytical grade and did not require further purification. Toluene was purchased from Jiangsu Qiang Sheng Functional Chemical Co. Analytical thin-layer chromatography (TLC) was performed on silica gel and observed by UV light irradiation. Column chromatography was performed using 300-400 mesh silica gel. $^1\text{H-NMR}$ and $^{13}\text{C-NMR}$ were recorded in CDCl_3 on a BRUKER 400 MHz spectrometer. Chemical shifts (δ) are reported with reference to the internal tetramethylsilane standard or the CDCl_3 residual peak (δ 7.26) for $^1\text{H NMR}$. $^{13}\text{C NMR}$ chemical shifts are reported with respect to CDCl_3 (δ 77.16). Data are reported in the following order: chemical shifts (δ) in ppm; multiples are expressed as s (singlet), bs (broad singlet), d (doublet), t (triplet), and m (multiplet); and coupling constants (J) are in hertz (Hz). IR spectra were recorded on a BRUKER VERTEX 70 spectrophotometer and are reported in absorption frequencies (cm^{-1}). HRMS spectra were obtained by using a GCT Premier TOF-MS with a CI source or a BRUKER microTOF-Q III instrument with an ESI source.

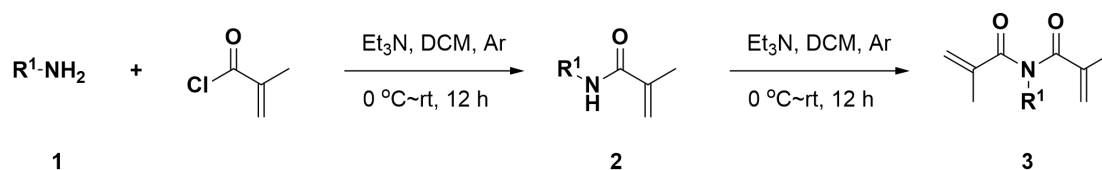
2. Light Sources and Photochemical Reactions Setup



LED bulbs were purchased from TanLu. LED bulbs purchased from TanLu were used in our studies (model number: YL170618-23653-DSS (SZY) 0707 purple LED bulb). The light sources were placed about 10 cm away from the reaction vessel during the reactions.

3. General procedures

3.1 Preparation of starting materials 3

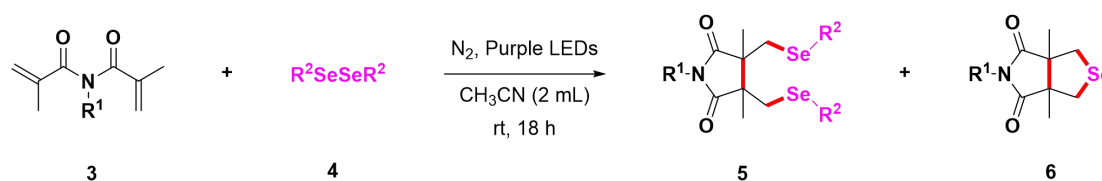


To a mixture of **1** (1.0 equiv.) and Et_3N (2 equiv.) in DCM (20 mL) were added methacryloyl chloride (1.2 equiv.) under an argon atmosphere. After stirring at $0\text{ }^\circ\text{C}$ for 30 minute and room temperature overnight. The mixture was extracted with DCM, washed with brine, and dried over Na_2SO_4 . The concentrated residue was purified by column chromatography over silica gel using petroleum ether/ethyl acetate 20:1 to get **2**. Next, under an argon atmosphere, methacryloyl chloride (1.2 equiv.) was dropwise added to a mixture of **2** (1.0 equiv.), and Et_3N (2 equiv.) in DCM (20 mL) at $0\text{ }^\circ\text{C}$. After stirring at $0\text{ }^\circ\text{C}$ for 30 minute and room temperature overnight. The mixture was extracted with DCM, washed with brine, and dried over Na_2SO_4 . The concentrated residue was purified by column chromatography over silica gel using petroleum ether/ethyl acetate 20:1 to get **3**.

3.2 Preparation of starting materials 4

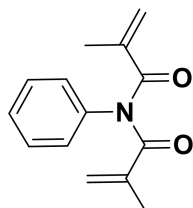
To a mixture of Se (1.0 equiv.) and NaOH (1.5 equiv.) in DMF (20 mL) were added hydrazinium hydroxide solution (1.0 equiv.) under an argon atmosphere. The resulting mixture was stirred for 2 h at $90\text{ }^\circ\text{C}$ during which it turned a reddish-brown. The alkyl halide (1.5 equiv.) was then added, and stirring was continued for 2 h at room temperature until reaction completion. The mixture was extracted with DCM, washed with brine, and dried over Na_2SO_4 . The concentrated residue was purified by column chromatography over silica gel using petroleum ether/ethyl acetate 50:1 to get **4**.

3.3 General Procedure and Product Characterization



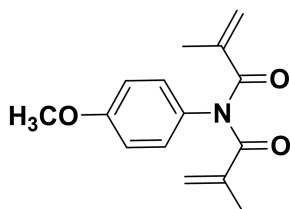
In glovebox, an oven-dried screw-capped 8 mL vial equipped with a magnetic stir bar was charged with **3** (0.2 mmol, 1 equiv.), **4** (0.2 mmol, 1 equiv.) and acetonitrile (2.0 mL) was added via syringe. The reaction mixture was stirred at room temperature for 18 h with a 40 W Purple LEDs lamp. After 18 h, the reaction solution was purified by flash column chromatography without extraction to obtain pure product **5**, **6**.

4. Spectroscopic Data of Compounds



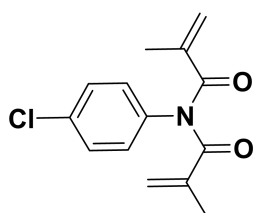
N-methacryloyl-*N*-phenylmethacrylamide (3a)

White solid; Yield 84% (1.9245 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 66.2 - 68.1 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.28 (m, J = 8.5, 6.9 Hz, 2H), 7.22 - 7.17 (m, 1H), 6.99 (m, J = 7.5, 1.8 Hz, 2H), 5.43 (s, 2H), 5.35 (d, J = 1.7 Hz, 2H), 1.81 (s, J = 1.3 Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 174.5, 143.5, 139.1, 129.5, 127.9, 127.3, 121.7, 19.0; **IR (neat)**: ν = 1659, 1345, 1145, 945, 751, 513 cm⁻¹; **HRMS** (ESI): calcd. for C₁₄H₁₅NO₂ [M+Na]: 252.0995, found: 252.0998.



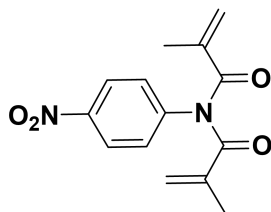
N-methacryloyl-*N*-(4-methoxyphenyl) methacrylamide (3b)

Pale yellow solid; Yield 74% (1.9174 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 63.8 - 65.7 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.06 - 7.01 (m, 2H), 6.94 - 6.89 (m, 2H), 5.53 (s, 2H), 5.45 (d, J = 1.7 Hz, 2H), 3.79 (s, 3H), 1.93 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 174.6, 159.1, 143.5, 131.6, 128.5, 121.4, 114.8, 55.5, 19.0; **IR (neat)**: ν = 1658, 1510, 1240, 1171, 1032, 825 cm⁻¹; **HRMS** (ESI): calcd. for C₁₅H₁₇NO₃Na [M+Na]⁺: 282.1101, found: 282.1092.



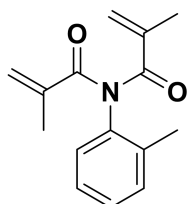
N-(4-chlorophenyl)-*N*-methacryloyl methacrylamide (3c)

Brown solid; Yield 54% (1.4200 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 61.4 - 63.2 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.41 - 7.34 (m, 2H), 7.09 - 7.03 (m, 2H), 5.53 (d, J = 1.2 Hz, 2H), 5.49 (d, J = 1.6 Hz, 2H), 1.93 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 174.2, 143.5, 137.5, 133.7, 129.7, 128.6, 121.9, 18.9; **IR (neat)**: ν = 1662, 1623, 1488, 1296, 1171, 1087, 934, 819 cm⁻¹; **HRMS** (ESI): calcd. for C₁₄H₁₄ClNO₂Na [M+Na]⁺: 286.0605, found: 286.0610.



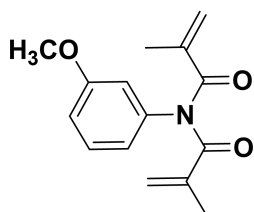
***N*-methacryloyl-*N*-(4-nitrophenyl)methacrylamide (3d)**

Yellow solid; Yield 73% (2.0010 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 107.2 - 109.1 °C; **¹H NMR** (400 MHz, CDCl₃) δ 8.30 - 8.25 (m, 2H), 7.31 - 7.27 (m, 2H), 5.57 (s, 4H), 1.96 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 173.9, 146.5, 144.9, 143.6, 127.7, 124.8, 122.6, 18.9; **IR (neat)**: ν = 2923, 1704, 1372, 1160, 763, 525 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₄H₁₄N₂O₄Na [M+Na]⁺: 297.0846, found: 297.0841.



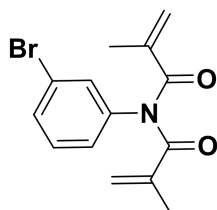
***N*-methacryloyl-*N*-(*o*-tolyl)methacrylamide (3e)**

Brown solid; Yield 64% (1.5560 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 55.9 - 57.5 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.35 - 7.26 (m, 3H), 7.04 (d, *J* = 7.0 Hz, 1H), 5.62 (s, 2H), 5.51 (d, *J* = 1.7 Hz, 2H), 2.30 (s, 3H), 2.00 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 174.1, 143.4, 137.8, 135.8, 131.4, 128.6, 127.9, 127.1, 121.1, 19.1, 18.0; **IR (neat)**: ν = 1662, 1624, 1166, 927, 753, 606 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₅H₁₇NO₂Na [M+Na]⁺: 266.1151, found: 266.1148.



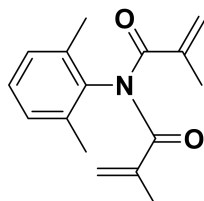
***N*-methacryloyl-*N*-(3-methoxyphenyl)methacrylamide (3f)**

White solid; Yield 44% (1.1401 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 42.3 - 43.9 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.31 (t, *J* = 8.1 Hz, 1H), 6.88 (m, *J* = 8.4, 2.5, 0.9 Hz, 1H), 6.70 (m, *J* = 7.8, 2.0, 0.9 Hz, 1H), 6.65 (t, *J* = 2.2 Hz, 1H), 5.57 (s, 2H), 5.48 (d, *J* = 1.6 Hz, 2H), 3.79 (s, 3H), 1.94 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 174.4, 160.4, 143.4, 140.1, 130.1, 121.7, 119.5, 113.6, 113.2, 55.4, 19.0; **IR (neat)**: ν = 2922, 1666, 1602, 1292, 1217, 1169, 1040, 779 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₅H₁₇NO₃Na [M+Na]⁺: 282.1101, found: 282.1091.



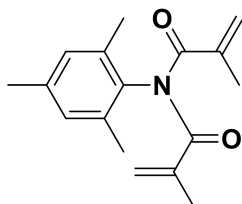
N-(3-bromophenyl)-*N*-methacryloylmethacrylamide (3g)

Yellow solid; Yield 83% (2.5483 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 52.2 - 54.8 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.46 (m, *J* = 8.0, 1.9, 1.0 Hz, 1H), 7.32 - 7.25 (m, 2H), 7.07 (m, *J* = 8.0, 2.1, 1.0 Hz, 1H), 5.55 (d, *J* = 1.1 Hz, 2H), 5.51 (d, *J* = 1.5 Hz, 2H), 1.94 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 174.1, 143.5, 140.2, 131.0, 130.6, 130.5, 126.1, 122.7, 122.0, 18.9; **IR (neat)**: ν = 1700, 1667, 1297, 1165, 934, 781, 679 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₄H₁₄BrNO₂Na [M+Na]: 330.0100, found: 330.0094.



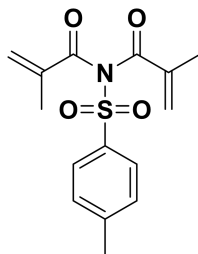
N-(2,6-dimethylphenyl)-*N*-methacryloylmethacrylamide (3h)

White solid; Yield 51% (1.3114 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 68.2 - 70.1 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.18 (m, *J* = 8.4, 6.5 Hz, 1H), 7.10 (d, *J* = 7.5 Hz, 2H), 5.56 (s, 2H), 5.44 (d, *J* = 1.6 Hz, 2H), 2.23 (s, 6H), 1.96 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 173.5, 143.2, 136.9, 136.2, 129.1, 128.8, 120.5, 19.4, 18.7; **IR (neat)**: ν = 1656, 1626, 1296, 1177, 774, 607 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₆H₁₉NO₂Na [M+Na]⁺: 280.1308, found: 280.1309.



N-mesityl-*N*-methacryloylmethacrylamide (3i)

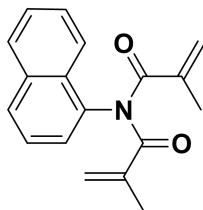
Yellow solid; Yield 87% (2.3591 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 79.8 - 81.3 °C; **¹H NMR** (400 MHz, CDCl₃) δ 6.92 (s, 2H), 5.55 (s, 2H), 5.43 (d, *J* = 1.6 Hz, 2H), 2.29 (s, 3H), 2.18 (s, 6H), 1.96 (s, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 173.7, 143.3, 138.5, 135.8, 134.2, 129.9, 120.3, 21.0, 19.5, 18.6; **IR (neat)**: ν = 2923, 1658, 1507, 1173, 929, 852 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₇H₂₁NO₂Na [M+Na]⁺: 294.1465, found: 294.1455.



N-methacryloyl-*N*-tosylmethacrylamide (3j)

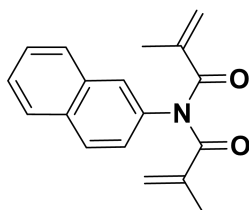
White solid; Yield 64% (1.9654 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 10/1); **Mp**: 61.9 - 63.1 °C; **¹H NMR** (400 MHz, CDCl₃) δ 7.92 (d, *J* = 1.9 Hz, 1H), 7.90 (d, *J* = 2.0 Hz, 1H), 7.30 - 7.26 (m, 2H), 5.60 (d, *J* = 1.6 Hz, 2H), 5.57 (d, *J* = 1.0 Hz, 2H), 2.38 (s, 3H), 1.82 (d, *J* = 0.6 Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 168.1, 144.5, 141.0, 134.5, 128.5, 128.2,

125.3, 20.7, 17.7; **IR (neat)**: $\nu = 3292, 1691, 1363, 1168, 1082, 2358, 569 \text{ cm}^{-1}$; **HRMS (ESI)**: calcd. for $\text{C}_{15}\text{H}_{17}\text{NO}_4\text{S} [\text{M}+\text{Na}]^+$: 330.0770, found: 330.0769.



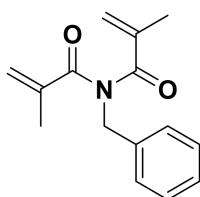
***N*-methacryloyl-*N*-(naphthalen-1-yl)methacrylamide (3k)**

Brown solid; Yield 87% (2.4284 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 92.0 - 94.1 °C; **¹H NMR** (400 MHz, CDCl_3) δ 7.94 - 7.80 (m, 3H), 7.62 - 7.43 (m, 3H), 7.24 (m, $J = 7.2, 1.1 \text{ Hz}$, 1H), 5.78 (s, 2H), 5.49 (d, $J = 1.6 \text{ Hz}$, 2H), 1.99 (s, 6H); **¹³C NMR** (100 MHz, CDCl_3) δ 174.7, 142.6, 135.7, 134.7, 130.2, 129.2, 128.8, 127.4, 126.5, 125.9, 125.5, 122.0, 121.4, 19.1; **IR (neat)**: $\nu = 1659, 1623, 1344, 1296, 1166, 795, 772 \text{ cm}^{-1}$; **HRMS (ESI)**: calcd. for $\text{C}_{18}\text{H}_{17}\text{NO}_2\text{Na} [\text{M}+\text{Na}]^+$: 302.1151, found: 302.1158.



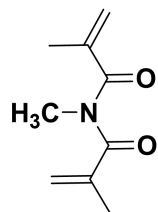
***N*-methacryloyl-*N*-(naphthalen-2-yl)methacrylamide (3l)**

White solid; Yield 48% (1.3400 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 102.3 - 104.1 °C; **¹H NMR** (400 MHz, CDCl_3) δ 7.92 - 7.83 (m, 2H), 7.83 - 7.78 (m, 1H), 7.56 (d, $J = 2.2 \text{ Hz}$, 1H), 7.53 - 7.47 (m, 2H), 7.24 (m, $J = 8.7, 2.2 \text{ Hz}$, 1H), 5.65 (s, 2H), 5.51 (d, $J = 1.6 \text{ Hz}$, 2H), 1.97 (s, 6H); **¹³C NMR** (100 MHz, CDCl_3) δ 174.1, 143.4, 137.8, 135.8, 131.4, 128.6, 127.9, 127.1, 121.1, 19.1, 18.0; **IR (neat)**: $\nu = 1664, 1626, 1179, 933, 817, 750, 477 \text{ cm}^{-1}$; **HRMS (ESI)**: calcd. for $\text{C}_{18}\text{H}_{17}\text{NO}_2\text{Na} [\text{M}+\text{Na}]^+$: 302.1151, found: 302.1142.



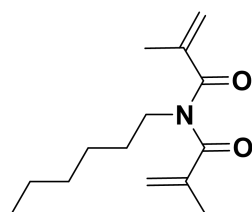
***N*-benzyl-*N*-methacryloylmethacrylamide (3m)**

Pale yellow liquid; Yield 80% (1.9450 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **¹H NMR** (400 MHz, CDCl_3) δ 7.34 (d, $J = 6.8 \text{ Hz}$, 2H), 7.25 - 7.15 (m, 3H), 5.18 (s, 2H), 4.96 (s, 2H), 4.84 (s, 2H), 1.77 (s, 6H); **¹³C NMR** (100 MHz, CDCl_3) δ 174.6, 144.7, 137.0, 129.0, 128.5, 127.7, 120.0, 48.4, 19.1; **IR (neat)**: $\nu = 1737, 1657, 1626, 1342, 1226, 1146, 930, 700 \text{ cm}^{-1}$; **HRMS (ESI)**: calcd. for $\text{C}_{15}\text{H}_{17}\text{NO}_2 [\text{M}+\text{Na}]^+$: 266.1151, found: 266.1147.



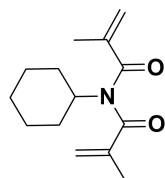
N-methacryloyl-*N*-methylmethacrylamide (**3n**)

Pale yellow solid; Yield 51% (0.8522 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 66.2 - 68.3 °C; **¹H NMR** (400 MHz, CDCl₃) δ 5.28 (d, *J* = 4.5 Hz, 2H), 5.10 (d, *J* = 7.0 Hz, 2H), 3.21 (d, *J* = 8.0 Hz, 3H), 1.84 (d, *J* = 5.5 Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 175.2, 144.3, 119.8, 32.0, 19.1; **IR (neat)**: ν = 1702, 1653, 1546, 1158, 1073, 938 cm⁻¹; **HRMS** (ESI): calcd. for C₉H₁₃NO₂ [M]: 167.0946, found: 167.0948.



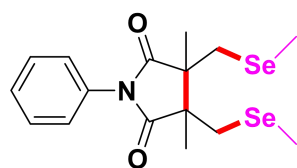
N-hexyl-*N*-methacryloylmethacrylamide (**3o**)

Colorless oil; Yield 48% (1.1384 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **¹H NMR** (400 MHz, CDCl₃) δ 5.28 (d, *J* = 1.6 Hz, 2H), 5.11 (s, 2H), 3.75 - 3.68 (m, 2H), 1.85 (s, 6H), 1.63 - 1.53 (m, 2H), 1.32 - 1.23 (m, 6H), 0.88 - 0.82 (m, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ 174.9, 144.9, 119.6, 45.2, 31.4, 28.3, 26.6, 22.5, 19.1, 14.0; **IR (neat)**: ν = 2927, 1656, 1627, 1344, 1105, 924 cm⁻¹; **HRMS** (ESI): calcd. for C₁₄H₂₃NO₂Na [M+Na]: 260.1621, found: 260.1614.



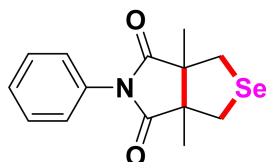
N-cyclohexyl-*N*-methacryloylmethacrylamide (**3p**)

Yellow solid; Yield 61% (1.4345 g, 10 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 58.6 - 60.2 °C; **¹H NMR** (400 MHz, CDCl₃) δ 5.31 (d, *J* = 1.7 Hz, 2H), 5.16 (s, 2H), 4.28 (m, *J* = 12.2, 3.7 Hz, 1H), 2.04 - 1.94 (m, 2H), 1.82 (s, 6H), 1.80 - 1.59 (m, 5H), 1.38 - 1.27 (m, 2H), 1.21 - 1.13 (m, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 174.9, 145.9, 120.5, 56.4, 29.9, 26.2, 25.3, 19.0. **IR (neat)**: ν = 2930, 1701, 1652, 1178, 1129, 930 cm⁻¹; **HRMS** (ESI): calcd. for C₁₄H₂₁NO₂Na [M+Na]: 258.1465, found: 258.1462.



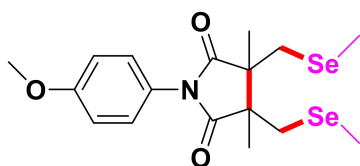
3,4-dimethyl-3,4-bis((methylselanyl)methyl)-1-phenylpyrrolidine-2,5-dione (**5a**)

Yellow solid; Yield 67% (0.1123 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 54.6 - 56.8 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.44 (m, *J* = 3.9, 1.5 Hz, 4H), 7.39 - 7.33 (m, 1H), 2.89 (d, *J* = 12.8 Hz, 2H), 2.80 (d, *J* = 12.8 Hz, 2H), 1.91 (d, *J* = 1.5 Hz, 6H), 1.40 (d, *J* = 1.6 Hz, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.4, 132.2, 129.1, 128.4, 126.4, 49.8, 34.8, 18.7, 7.4. **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 55.0; **IR (neat)**: ν = 2922, 1704, 1492, 1371, 1133, 761, 692 cm⁻¹; **HRMS** (ESI): calcd. for C₁₆H₂₁NO₂Se₂Na [M+Na]: 441.9795, found: 441.9802.



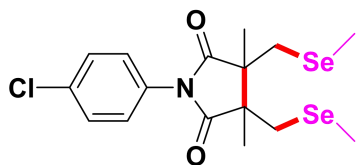
3a,6a-dimethyl-5-phenyltetrahydro-4H-selenopheno[3,4-c]pyrrole-4,6(5H)-dione (6a)

White solid; Yield 28% (0.0346 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 10/1); **Mp**: 99.8 - 101.7 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.42 (m, *J* = 8.3, 6.6 Hz, 2H), 7.38 - 7.33 (m, 1H), 7.28 - 7.23 (m, 2H), 3.45 (d, *J* = 11.6 Hz, 2H), 2.93 (d, *J* = 11.6 Hz, 2H), 1.36 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 180.3, 132.0, 129.1, 128.8, 126.5, 57.5, 36.2, 20.1. **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 155.6; **IR (neat)**: ν = 1708, 1494, 1370, 1179, 1077, 964, 692 cm⁻¹; **HRMS** (ESI): calcd. for C₁₄H₁₅NO₂Se [M]: 309.0268, found: 309.0269.



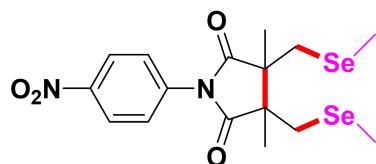
(4-methoxyphenyl)-3,4-dimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5b1)

Brown solid; Yield 47% (0.0845 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 70.3-72.0 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 9.0 Hz, 2H), 6.98 - 6.93 (m, 2H), 3.80 (s, 3H), 2.89 (d, *J* = 12.8 Hz, 2H), 2.80 (d, *J* = 12.8 Hz, 2H), 1.92 (s, 6H), 1.40 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.6, 159.4, 127.6, 124.9, 114.4, 55.5, 49.8, 34.8, 18.7, 7.3; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 54.9; **IR (neat)**: ν = 2924, 1703, 1512, 1247, 1023 cm⁻¹; **HRMS** (ESI): calcd. for C₁₇H₂₃NO₃Se₂Na [M+Na]: 471.9901, found: 471.9905.



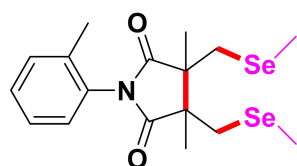
(4-chlorophenyl)-3,4-dimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5c1)

Pale yellow solid; Yield 51% (0.0919 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 103.7-105.0 °C. **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.42 (s, 4H), 2.89 (d, *J* = 12.8 Hz, 2H), 2.79 (d, *J* = 12.8 Hz, 2H), 1.90 (s, 6H), 1.40 (s, 6H). **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.1, 134.1, 130.6, 129.3, 127.6, 49.8, 34.9, 18.6, 7.3. **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 55.1; **IR (neat)**: ν = 2923, 2852, 1708, 1489, 1378 cm⁻¹; **HRMS** (ESI) : calcd. for C₁₆H₂₀ClNO₂Se₂ [M]: 452.9513, found: 452.9508.



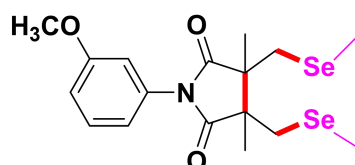
3,4-dimethyl-3,4-bis((methylselanyl)methyl)-1-(4-nitrophenyl)pyrrolidine-2,5-dione (5d1)

Yellow solid; Yield 35% (0.0647 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 103.8 -105.0 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 8.34 - 8.30 (m, 2H), 7.76 - 7.70 (m, 2H), 2.91 (d, *J* = 12.8 Hz, 2H), 2.80 (d, *J* = 12.8 Hz, 2H), 1.88 (s, 6H), 1.42 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 178.6, 146.9, 137.6, 126.8, 124.3, 50.0, 34.9, 18.4, 7.3; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 55.2; **IR (neat)**: ν = 2922, 1706, 1523, 1344 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₆H₂₀N₂O₄Se₂ [M]: 463.9754, found: 463.9756.



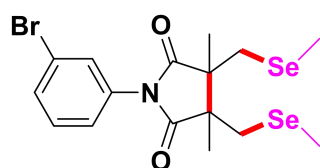
3,4-dimethyl-3,4-bis((methylselanyl)methyl)-1-(o-tolyl)pyrrolidine-2,5-dione (5e1)

Yellow solid; Yield 51% (0.0877 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 71.6 - 73.8 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.33 - 7.27 (m, 3H), 7.22 (m, *J* = 7.4, 1.3 Hz, 1H), 3.02 (m, *J* = 19.9, 12.8 Hz, 2H), 2.88 (t, *J* = 13.0 Hz, 2H), 2.24 (s, 3H), 2.06 (s, 3H), 2.03 (s, 3H), 1.52 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.6, 179.5, 135.8, 131.2, 131.1, 129.3, 128.0, 126.8, 51.1, 50.8, 33.2, 33.1, 20.5, 19.7, 18.0, 7.8; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 60.9, 50.4; **IR (neat)**: ν = 2923, 1704, 1372, 1160 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₇H₂₃NO₂Se₂Na [M+Na]: 455.9951, found: 455.9959.



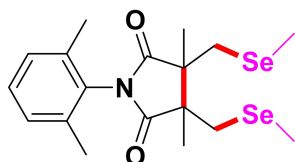
1-(3-methoxyphenyl)-3,4-dimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5f1)

Yellow solid; Yield 64% (0.1155 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 80.1 - 82.8 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.35 (t, *J* = 8.1 Hz, 1H), 7.05 - 6.98 (m, 2H), 6.91 (m, *J* = 8.4, 2.6, 1.0 Hz, 1H), 3.79 (s, 3H), 2.90 (d, *J* = 12.9 Hz, 2H), 2.80 (d, *J* = 12.8 Hz, 2H), 1.93 (s, 6H), 1.40 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.3, 160.0, 133.2, 129.7, 118.7, 114.3, 112.2, 55.4, 49.8, 34.8, 18.7, 7.4; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 54.9; **IR (neat)**: ν = 2957, 2921, 2361, 1705, 1254, 1028, 781 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₇H₂₃NO₃Se₂Na [M+Na]: 471.9901, found: 471.9895.



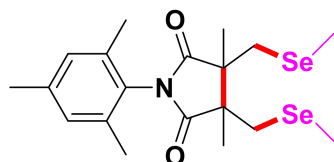
1-(3-bromophenyl)-3,4-dimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5g1)

Yellow solid; Yield 20% (0.0401 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 61.9 - 63.8 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.64 (s, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.43 (d, *J* = 8.1 Hz, 1H), 7.34 (t, *J* = 8.0 Hz, 1H), 2.90 (d, *J* = 12.9 Hz, 2H), 2.80 (d, *J* = 12.7 Hz, 2H), 1.92 (s, 6H), 1.41 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.0, 133.3, 131.5, 130.3, 129.4, 125.1, 122.4, 49.9, 34.8, 18.6, 7.4; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 55.0; **IR (neat)**: ν = 2923, 2361, 1703, 1366, 1162 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₆H₂₀BrNO₂Se₂ [M]: 496.9008, found: 496.8997.



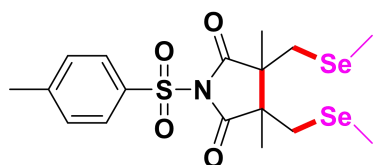
1-(2,6-dimethylphenyl)-3,4-dimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5h1)

Pale yellow solid; Yield 60% (0.1073 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1). **Mp**: 120.8 - 123.0 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.21 (m, *J* = 8.3, 6.8 Hz, 1H), 7.11 (d, *J* = 7.6 Hz, 2H), 3.11 (d, *J* = 12.8 Hz, 2H), 2.91 (d, *J* = 12.8 Hz, 2H), 2.13 (s, 6H), 2.10 (s, 6H), 1.62 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.5, 135.9, 129.9, 129.5, 128.7, 51.7, 30.9, 22.5, 18.0, 8.0; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 56.3. **IR (neat)**: ν = 2922, 2361, 2198, 1706, 1161 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₈H₂₅NO₂Se₂Na [M+Na]: 470.0108, found: 470.0101.



1-mesityl-3,4-dimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5i1)

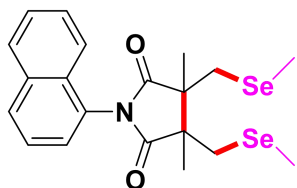
Yellow oil; Yield 57% (0.1053 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **¹H NMR** (400 MHz, Chloroform-*d*) δ 6.93 (s, 2H), 3.11 (d, *J* = 12.8 Hz, 2H), 2.90 (d, *J* = 12.8 Hz, 2H), 2.28 (s, 3H), 2.10 (s, 6H), 2.09 (s, 6H), 1.61 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.7, 139.3, 135.5, 129.5, 127.2, 51.6, 30.9, 22.5, 21.1, 17.9, 8.0; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 56.0; **IR (neat)**: ν = 2923, 1705, 1365, 1144 cm⁻¹; **HRMS (ESI)**: calcd. for C₁₉H₂₇NO₂Se₂Na [M+Na]: 484.0264, found: 484.0257.



3,4-dimethyl-3,4-bis((methylselanyl)methyl)-1-tosylpyrrolidine-2,5-dione (5j1)

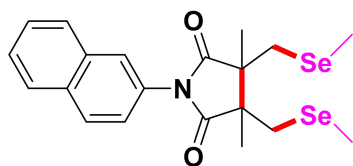
Yellow solid; Yield 42% (0.0835 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 10/1); **Mp**: 94.4 - 96.1 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.4 Hz, 2H), 7.28 (d, *J* = 8.3 Hz, 2H), 2.67 (d, *J* = 13.0 Hz, 2H), 2.58 (d, *J* = 13.0 Hz, 2H), 2.37 (s, 3H),

1.74 (s, 6H), 1.20 (s, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 176.3, 175.3, 146.3, 134.8, 129.8, 129.7, 129.0, 128.5, 58.4, 51.3, 35.6, 34.0, 21.9, 21.8, 19.9, 18.0, 7.4; ^{77}Se NMR (76 MHz, Chloroform-*d*) δ 55.1; **IR** (neat): ν = 2922, 2361, 1731, 1378, 1174, 565 cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{17}\text{H}_{23}\text{NO}_4\text{SSe}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 519.9570, found: 519.9573.



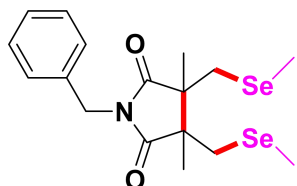
3,4-dimethyl-3,4-bis((methylselanyl)methyl)-1-(naphthalen-1-yl)pyrrolidine-2,5-dione (5k1)

Pale yellow solid; Yield 43% (0.0814 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 108.2 - 110.0 $^{\circ}\text{C}$; ^1H NMR (400 MHz, Chloroform-*d*) δ 7.93 (m, J = 12.0, 5.3, 3.1 Hz, 3H), 7.57 - 7.50 (m, 4H), 3.07 (m, J = 13.0, 1.9 Hz, 2H), 2.97 (d, J = 13.0 Hz, 1H), 2.89 (d, J = 12.9 Hz, 1H), 2.01 (s, 3H), 1.98 (s, 3H), 1.59 (s, 3H), 1.54 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 180.1, 180.0, 134.4, 129.8, 129.7, 129.1, 128.5, 126.9, 126.5, 126.2, 125.3, 122.9, 50.8, 50.7, 34.1, 33.8, 20.2, 20.0, 8.0, 7.9; ^{77}Se NMR (76 MHz, Chloroform-*d*) δ 62.1, 50.6; **IR** (neat): ν = 2922, 2361, 1702, 769 cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{20}\text{H}_{23}\text{NO}_2\text{Se}_2\text{Na}$ $[\text{M}+\text{Na}]^+$: 491.9951, found: 491.9958.



3,4-dimethyl-3,4-bis((methylselanyl)methyl)-1-(naphthalen-2-yl)pyrrolidine-2,5-dione (5l1)

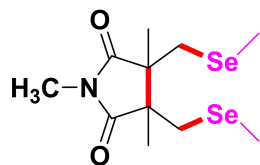
Yellow oil; Yield 39% (0.0731 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); ^1H NMR (400 MHz, Chloroform-*d*) δ 7.97 - 7.91 (m, 2H), 7.87 (q, J = 4.7 Hz, 2H), 7.58 (d, J = 8.7 Hz, 1H), 7.51 (m, J = 6.5, 3.1 Hz, 2H), 2.95 (d, J = 12.8 Hz, 2H), 2.86 (d, J = 13.3 Hz, 2H), 1.94 (s, 6H), 1.46 (s, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 179.5, 133.2, 132.8, 129.7, 128.9, 128.3, 127.8, 126.7, 126.5, 125.4, 124.0, 49.9, 34.8, 18.7, 7.4; ^{77}Se NMR (76 MHz, Chloroform-*d*) δ 55.5. **IR** (neat): ν = 2926, 2926, 1705, 1373, 812, 743 cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{20}\text{H}_{23}\text{NO}_2\text{Se}_2$ $[\text{M}]$: 469.0059, found: 469.0053.



1-benzyl-3,4-dimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5m1)

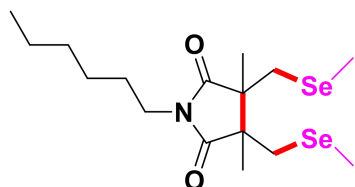
Brown oil; Yield 50% (0.0866 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1). ^1H NMR (400 MHz, Chloroform-*d*) δ 7.47 - 7.44 (m, 2H), 7.33 - 7.27 (m, 3H), 4.74 (d, J = 14.0 Hz, 1H), 4.65 (d, J = 13.9 Hz, 1H), 2.81 (d, J = 12.8 Hz, 2H), 2.73 (d, J = 12.8 Hz, 2H), 1.82 (s, 6H), 1.35 (s, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 180.3, 135.6, 129.3, 128.5, 127.9, 50.4, 42.6, 33.8, 19.0, 7.2; ^{77}Se NMR (76 MHz, Chloroform-*d*) δ 56.2; **IR** (neat): ν = 2976,

2927, 2927, 1695, 1343, 697 cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{17}\text{H}_{23}\text{NO}_2\text{Se}_2\text{Na}[\text{M}+\text{Na}]$: 455.9951, found: 455.9964.



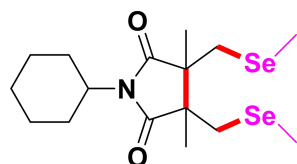
1,3,4-trimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5n1)

Yellow oil; Yield 68% (0.0960 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **^1H NMR** (400 MHz, Chloroform-*d*) δ 3.01 (s, 3H), 2.79 (d, $J = 12.7$ Hz, 2H), 2.70 (d, $J = 12.8$ Hz, 2H), 1.94 (s, 6H), 1.31 (s, 6H); **^{13}C NMR** (100 MHz, Chloroform-*d*) δ 181.1, 180.6, 57.5, 50.2, 35.7, 34.2, 25.3, 24.6, 20.0, 18.7, 7.1; **^{77}Se NMR** (76 MHz, Chloroform-*d*) δ 153.9, 57.3; **IR (neat)**: $\nu = 2928, 1693, 1433, 1308, 1055$ cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{11}\text{H}_{19}\text{NO}_2\text{Se}_2\text{Na}[\text{M}+\text{Na}]$: 379.9638, found: 379.9637.



1-hexyl-3,4-dimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5o1)

Yellow oil; Yield 42% (0.0717 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **^1H NMR** (400 MHz, Chloroform-*d*) δ 3.49 - 3.37 (m, 2H), 2.77 (d, $J = 12.7$ Hz, 2H), 2.67 (d, $J = 12.7$ Hz, 2H), 1.93 (s, 6H), 1.57 - 1.49 (m, 2H), 1.30 (s, 6H), 1.25 - 1.21 (m, 6H), 0.82 (d, $J = 5.9$ Hz, 3H); **^{13}C NMR** (100 MHz, Chloroform-*d*) δ 180.5, 50.3, 39.0, 33.5, 31.3, 27.6, 26.6, 22.5, 19.3, 14.0, 7.4; **^{77}Se NMR** (76 MHz, Chloroform-*d*) δ 55.1; **IR (neat)**: $\nu = 2926, 2857, 1694, 1399$ cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{16}\text{H}_{29}\text{NO}_2\text{Se}_2\text{Na}[\text{M}+\text{Na}]^+$: 450.0421, found: 450.0419.



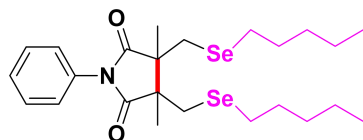
1-cyclohexyl-3,4-dimethyl-3,4-bis((methylselanyl)methyl)pyrrolidine-2,5-dione (5p1)

Yellow oil; Yield 49% (0.0832 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **^1H NMR** (400 MHz, Chloroform-*d*) δ 3.94 (m, $J = 12.3, 8.5, 3.8$ Hz, 1H), 2.76 (d, $J = 12.5$ Hz, 2H), 2.68 (d, $J = 12.6$ Hz, 2H), 2.11 (m, $J = 18.8, 12.3, 3.5$ Hz, 2H), 1.96 (s, 6H), 1.80 - 1.75 (m, 2H), 1.63 (q, $J = 3.6, 3.1$ Hz, 2H), 1.30 (s, 6H), 1.25 (d, $J = 3.0$ Hz, 1H), 1.21 (d, $J = 3.4$ Hz, 2H), 1.19 - 1.13 (m, 1H); **^{13}C NMR** (100 MHz, Chloroform-*d*) δ 180.4, 56.7, 52.2, 49.9, 35.9, 33.7, 29.1, 28.8, 25.9, 25.8, 25.1, 20.0, 19.2, 7.5; **^{77}Se NMR** (76 MHz, Chloroform-*d*) δ 52.9; **IR (neat)**: $\nu = 2925, 1691, 1366, 1180, 1122$ cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{16}\text{H}_{27}\text{NO}_2\text{Se}_2\text{Na}[\text{M}+\text{Na}]$: 448.0264, found: 448.0254.



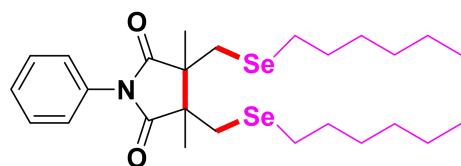
3,4-dimethyl-1-phenyl-3,4-bis((propylselanyl)methyl)pyrrolidine-2,5-dione (5b2)

Yellow solid; Yield 42% (0.0801 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 51.7 - 54.0 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.44 (q, J = 3.8, 3.1 Hz, 4H), 7.39 - 7.32 (m, 1H), 2.93 - 2.78 (m, 4H), 2.47 (m, J = 7.4, 3.7 Hz, 4H), 1.59 (m, J = 7.3, 1.5 Hz, 4H), 1.43 (s, 6H), 0.90 (t, J = 7.3 Hz, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.5, 132.2, 128.9, 128.4, 126.5, 50.0, 32.8, 29.3, 23.5, 18.9, 14.3; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 130.5; **IR (neat)** : ν = 2927, 2361, 1706, 1371, 691 cm⁻¹; **HRMS** (ESI): calcd. for C₂₀H₂₉NO₂Se₂Na [M+Na]⁺: 498.0421, found: 498.0431.



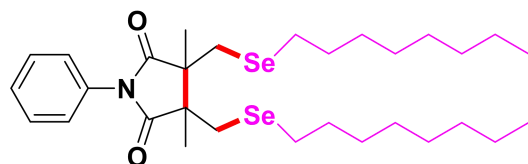
3,4-dimethyl-3,4-bis((pentylselanyl)methyl)-1-phenylpyrrolidine-2,5-dione (5c2)

Yellow oil; Yield 24% (0.0508 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.45 (d, J = 3.6 Hz, 4H), 7.37 (m, J = 7.0, 4.6, 3.1 Hz, 1H), 2.89 (d, J = 12.6 Hz, 2H), 2.82 (d, J = 12.6 Hz, 2H), 2.48 (m, J = 8.3, 5.8, 2.9 Hz, 4H), 1.60 - 1.53 (m, 4H), 1.44 (s, 6H), 1.27 - 1.23 (m, 8H), 0.84 (m, J = 5.7, 4.6, 2.8 Hz, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.5, 132.2, 128.9, 128.3, 126.5, 50.0, 32.9, 31.9, 29.9, 27.2, 22.1, 18.9, 13.9; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 134.2; **IR (neat)**: ν = 2925, 1708, 1370, 760, 692 cm⁻¹; **HRMS** (ESI): calcd. for C₂₄H₃₇NO₂Se₂H [M+H]⁺: 532.1227, found: 532.1225.



3,4-bis((hexylselanyl)methyl)-3,4-dimethyl-1-phenylpyrrolidine-2,5-dione (5d2)

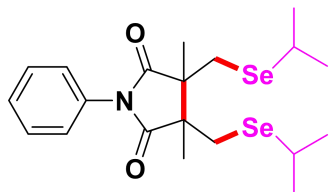
Yellow oil; Yield 26% (0.0584 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.45 (d, J = 5.0 Hz, 4H), 7.40 - 7.33 (m, 1H), 2.92 - 2.78 (m, 4H), 2.54 - 2.42 (m, 4H), 1.60 - 1.49 (m, 4H), 1.43 (s, 6H), 1.23 (m, J = 17.0, 14.1, 9.3, 5.4 Hz, 12H), 0.85 (t, J = 6.9 Hz, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.5, 132.2, 128.9, 128.3, 126.5, 50.0, 32.9, 31.3, 30.2, 29.4, 27.3, 22.5, 18.9, 14.0; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 134.1; **IR (neat)**: ν = 2924, 1708, 1370, 760, 691 cm⁻¹; **HRMS** (ESI): calcd. for C₂₆H₄₁NO₂Se₂[M]: 559.1468, found: 559.1472.



3,4-dimethyl-3,4-bis((octylselanyl)methyl)-1-phenylpyrrolidine-2,5-dione (5e2)

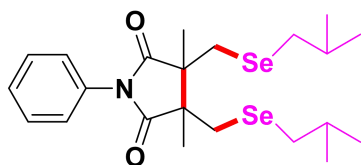
Yellow oil; Yield 35% (0.0870 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.44 (d, J = 4.4 Hz, 4H), 7.40 - 7.33 (m, 1H), 2.92 - 2.77 (m, 4H), 2.48 (m, J = 7.4, 2.5 Hz, 4H), 1.55 (m, J = 7.0, 1.5 Hz, 4H), 1.43 (s, 6H), 1.30 - 1.18 (m, 20H), 0.86 (t, J = 6.9 Hz, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.5, 132.2,

128.9, 128.3, 126.5, 50.0, 32.9, 31.8, 30.2, 29.7, 29.1, 29.0, 27.3, 22.6, 18.9, 14.1; ^{77}Se NMR (76 MHz, Chloroform-*d*) δ 134.0; **IR** (neat): $\nu = 2923, 1709, 1377, 692\text{ cm}^{-1}$; **HRMS** (ESI): calcd. for $\text{C}_{30}\text{H}_{49}\text{NO}_2\text{Se}_2\text{H}$ [M+H]: 616.2166, found: 616.2174.



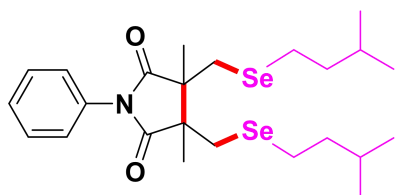
3,4-bis((isopropylselanyl)methyl)-3,4-dimethyl-1-phenylpyrrolidine-2,5-dione (5f2)

Yellow solid; Yield 39% (0.0741 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 62.6 - 64.1 °C; ^1H NMR (400 MHz, Chloroform-*d*) δ 7.46 - 7.40 (m, 4H), 7.36 (m, $J = 6.5, 3.1, 1.8\text{ Hz}$, 1H), 2.99 (m, $J = 13.6, 6.8\text{ Hz}$, 2H), 2.91 - 2.79 (m, 4H), 1.46 (s, 6H), 1.36 (d, $J = 6.8\text{ Hz}$, 6H), 1.27 (d, $J = 6.9\text{ Hz}$, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 179.5, 132.2, 128.9, 128.3, 126.5, 50.1, 31.7, 31.3, 24.4, 24.0, 19.1; ^{77}Se NMR (76 MHz, Chloroform-*d*) δ 259.5; **IR** (neat): $\nu = 2923, 2361, 1707, 1671, 1369, 1151\text{ cm}^{-1}$; **HRMS** (ESI): calcd. for $\text{C}_{20}\text{H}_{29}\text{NO}_2\text{Se}_2\text{Na}$ [M+Na]: 498.0421, found: 498.0416.



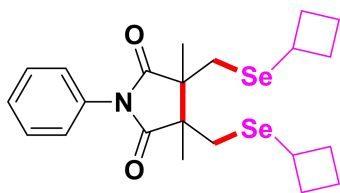
3,4-bis((isobutylselanyl)methyl)-3,4-dimethyl-1-phenylpyrrolidine-2,5-dione (5g2)

Yellow solid; Yield 62% (0.1240 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 48.3 - 50.5 °C; ^1H NMR (400 MHz, Chloroform-*d*) δ 7.44 (d, $J = 4.4\text{ Hz}$, 4H), 7.39 - 7.34 (m, 1H), 2.88 (d, $J = 12.5\text{ Hz}$, 2H), 2.80 (d, $J = 12.5\text{ Hz}$, 2H), 2.42 (d, $J = 5.4\text{ Hz}$, 4H), 1.76 (m, $J = 6.7\text{ Hz}$, 2H), 1.44 (s, 6H), 0.92 (s, 6H), 0.90 (s, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 179.4, 132.2, 128.9, 128.3, 126.5, 50.1, 37.3, 33.6, 29.2, 22.6, 22.5, 18.9; ^{77}Se NMR (76 MHz, Chloroform-*d*) δ 109.7; **IR** (neat): $\nu = 2958, 1705, 1023, 796\text{ cm}^{-1}$; **HRMS** (ESI): calcd. for $\text{C}_{20}\text{H}_{29}\text{NO}_2\text{Se}_2\text{Na}$ [M+Na]: 526.0734, found: 526.0734.



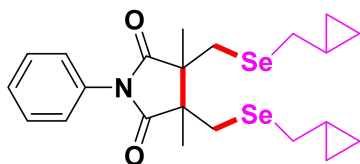
3,4-bis((isopentylselanyl)methyl)-3,4-dimethyl-1-phenylpyrrolidine-2,5-dione (5i2)

Yellow oli; Yield 29% (0.0610 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); ^1H NMR (400 MHz, Chloroform-*d*) δ 7.45 (d, $J = 4.3\text{ Hz}$, 4H), 7.39 - 7.33 (m, 1H), 2.85 (q, $J = 12.6\text{ Hz}$, 4H), 2.49 (m, $J = 8.8, 7.1, 2.1\text{ Hz}$, 4H), 1.54 (m, $J = 13.0, 6.5\text{ Hz}$, 2H), 1.48 - 1.45 (m, 2H), 1.44 (s, 6H), 1.41 (d, $J = 2.4\text{ Hz}$, 1H), 1.26 (d, $J = 9.4\text{ Hz}$, 1H), 0.82 (s, 6H), 0.80 (s, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 179.4, 132.2, 128.9, 128.3, 126.4, 50.0, 39.1, 32.8, 28.2, 25.1, 22.1, 18.9; ^{77}Se NMR (76 MHz, Chloroform-*d*) δ 135.6; **IR** (neat): $\nu = 2954, 2361, 1706, 1380, 762, 693\text{ cm}^{-1}$; **HRMS** (ESI): calcd. for $\text{C}_{24}\text{H}_{37}\text{NO}_2\text{Se}_2\text{H}$ [M+H] $^+$: 532.1227, found: 532.1223.



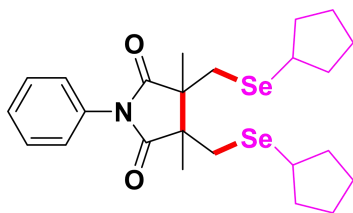
3,4-bis((cyclobutylselanyl)methyl)-3,4-dimethyl-1-phenylpyrrolidine-2,5-dione (5j2)

Yellow solid; Yield 40% (0.0804 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 77.2- 80.0 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.47 - 7.34 (m, 5H), 3.54 (q, *J* = 7.9 Hz, 2H), 2.86 - 2.73 (m, 4H), 2.41 - 2.32 (m, 2H), 2.27 - 2.15 (m, 2H), 2.09 - 1.91 (m, 6H), 1.89 - 1.78 (m, 2H), 1.41 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.5, 132.2, 128.9, 128.4, 126.6, 49.9, 34.5, 32.0, 31.0, 19.7, 19.0; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 226.3; **IR (neat)**: ν = 2963, 2931, 2361, 1703, 1026, 799 cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{22}\text{H}_{29}\text{NO}_2\text{Se}_2\text{Na}$ [M+Na]: 522.0421, found: 522.0419.



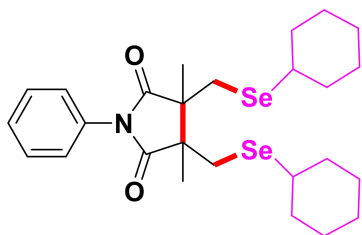
3,4-bis((cyclopropylmethyl)selanyl)methyl)-3,4-dimethyl-1-phenylpyrrolidine-2,5-dione (5k2)

Yellow solid; Yield 52% (0.1035 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 64.6 - 66.5 °C; **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.44 (d, *J* = 4.3 Hz, 4H), 7.38 - 7.34 (m, 1H), 2.92 (s, 4H), 2.46 - 2.40 (m, 4H), 1.43 (s, 6H), 0.94 (m, *J* = 7.6, 4.5 Hz, 2H), 0.54 - 0.49 (m, 4H), 0.16 - 0.08 (m, 4H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.5, 132.2, 128.9, 128.4, 126.5, 50.0, 32.4, 32.4, 19.0, 11.6, 6.8, 6.5; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 146.5; **IR (neat)**: ν = 2925, 2361, 1705, 1171, 761 cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{22}\text{H}_{29}\text{NO}_2\text{Se}_2\text{Na}$ [M+Na]: 522.0421, found: 522.0417.



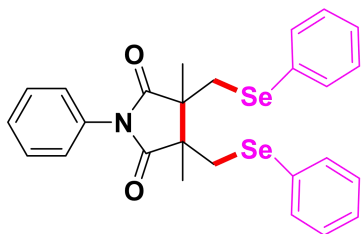
3,4-bis((cyclopentylselanyl)methyl)-3,4-dimethyl-1-phenylpyrrolidine-2,5-dione (5l2)

White solid; Yield 49% (0.1027 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **Mp**: 76.3 - 78.6 °C. **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.44 (d, *J* = 3.5 Hz, 4H), 7.39 - 7.33 (m, 1H), 3.13 (p, *J* = 6.8 Hz, 2H), 2.93 - 2.78 (m, 4H), 2.00 (m, *J* = 12.3, 7.1, 6.2, 3.4 Hz, 2H), 1.93 - 1.83 (m, 2H), 1.83 - 1.46 (m, 12H), 1.44 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 179.6, 132.2, 128.9, 128.3, 126.6, 50.0, 40.0, 34.5, 33.7, 32.5, 24.9, 24.7, 19.0; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 221.6; **IR (neat)**: ν = 2927, 1703, 1024, 800, 761 cm^{-1} ; **HRMS** (ESI): calcd. for $\text{C}_{24}\text{H}_{33}\text{NO}_2\text{Se}_2\text{Na}$ [M+Na]: 550.0734, found: 550.0732.



3,4-bis((cyclohexylselanyl)methyl)-3,4-dimethyl-1-phenylpyrrolidine-2,5-dione (5m2)

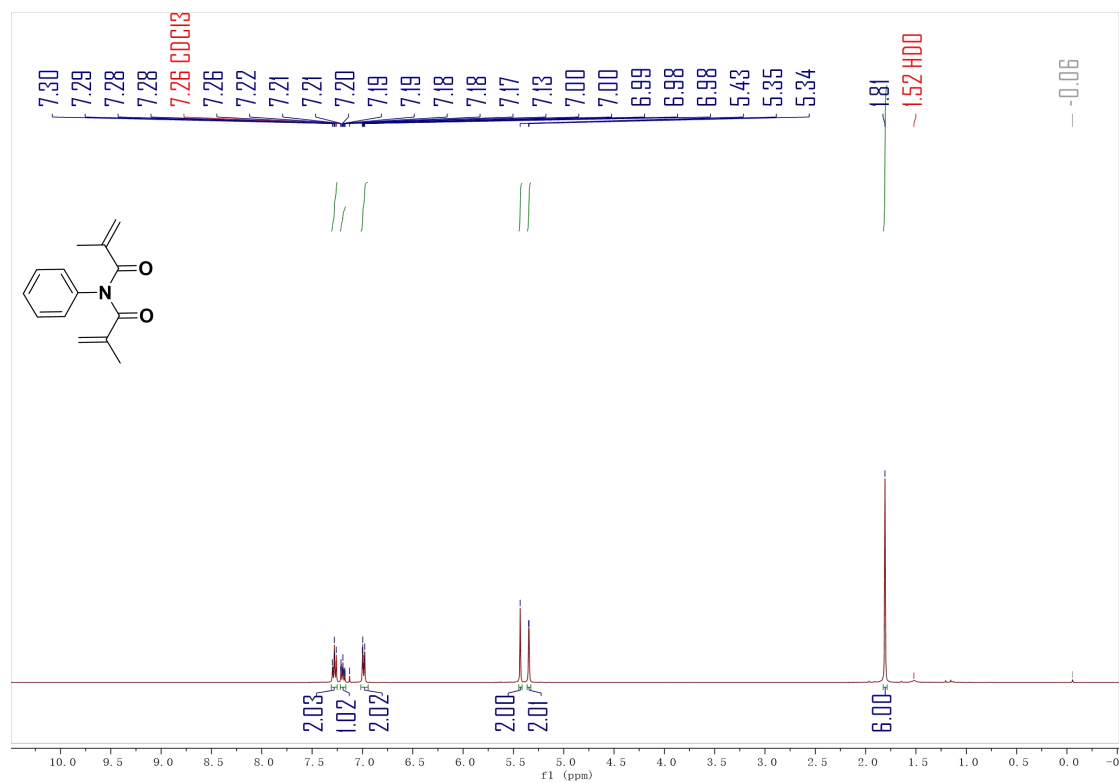
Yellow oil; Yield 32% (0.0701 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **¹H NMR** (400 MHz, CDCl₃) δ 7.44 (d, J = 5.8 Hz, 4H), 7.36 (m, J = 8.3, 4.3, 2.5 Hz, 1H), 2.85 (s, 4H), 2.80 (m, J = 10.6, 3.6 Hz, 2H), 1.98 (d, J = 14.5 Hz, 2H), 1.90 (d, J = 12.2 Hz, 2H), 1.70 - 1.62 (m, 4H), 1.58 - 1.53 (m, 2H), 1.46 (s, 6H), 1.43 - 1.34 (m, 4H), 1.27 - 1.21 (m, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 179.5, 132.2, 128.8, 128.3, 126.5, 50.2, 41.3, 34.3, 34.1, 30.9, 26.6, 25.7, 19.0; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 231.4; **IR (neat)**: ν = 2924, 2849, 1707, 1368, 1136, 759, 691 cm⁻¹; **HRMS** (ESI): calcd. for C₂₆H₃₇NO₂Se₂ [M]: 555.1155, found: 555.1158.



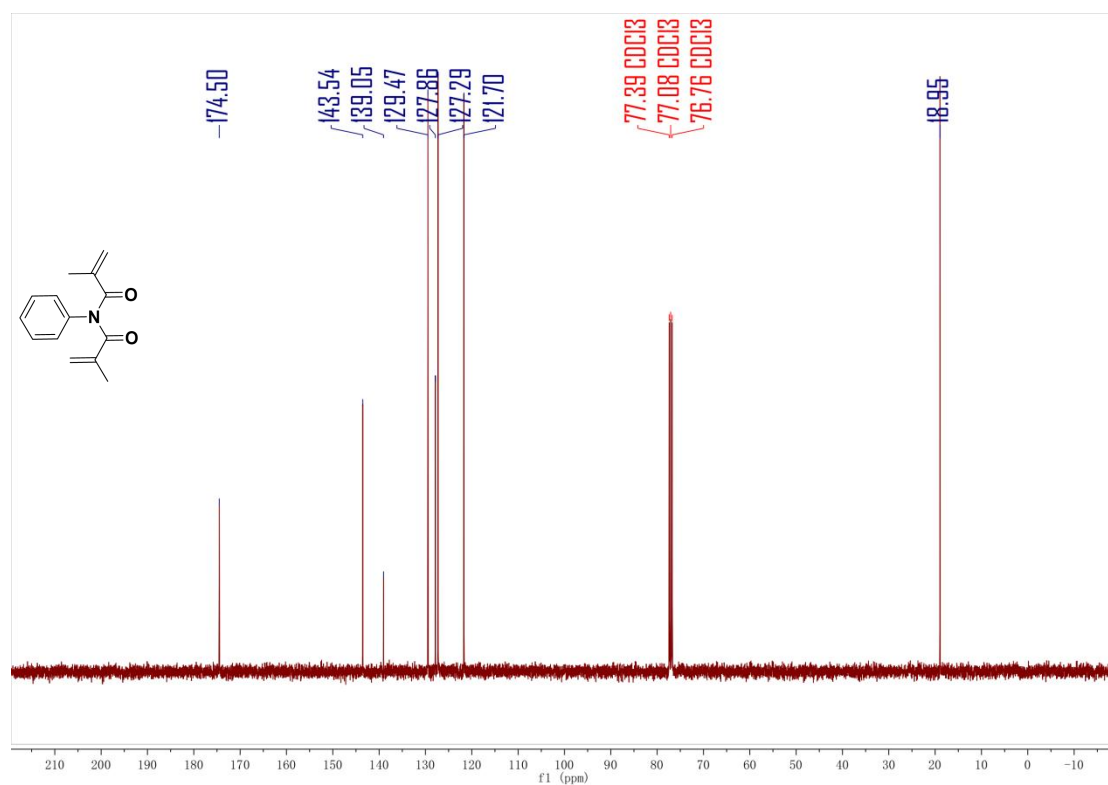
3,4-dimethyl-1-phenyl-3,4-bis((phenylselanyl)methyl)pyrrolidine-2,5-dione (5n2)

Yellow oil; Yield 62% (0.1341 g, 0.4 mmol scale), and purified by flash column chromatography on silica gel (PE /EA = 20/1); **¹H NMR** (400 MHz, Chloroform-*d*) δ 7.49 - 7.42 (m, 6H), 7.40 - 7.35 (m, 1H), 7.28 - 7.22 (m, 9H), 3.53 (d, J = 12.3 Hz, 2H), 3.42 (d, J = 12.3 Hz, 2H), 1.56 (s, 6H); **¹³C NMR** (100 MHz, Chloroform-*d*) δ 178.9, 133.3, 131.8, 130.5, 129.4, 129.0, 128.5, 127.6, 126.4, 51.1, 36.3, 19.0; **⁷⁷Se NMR** (76 MHz, Chloroform-*d*) δ 244.6; **IR (neat)**: ν = 2923, 1707, 1367, 736, 689 cm⁻¹; **HRMS** (ESI): calcd. for C₂₆H₂₅NO₂Se₂ [M]: 543.0216, found: 543.0211.

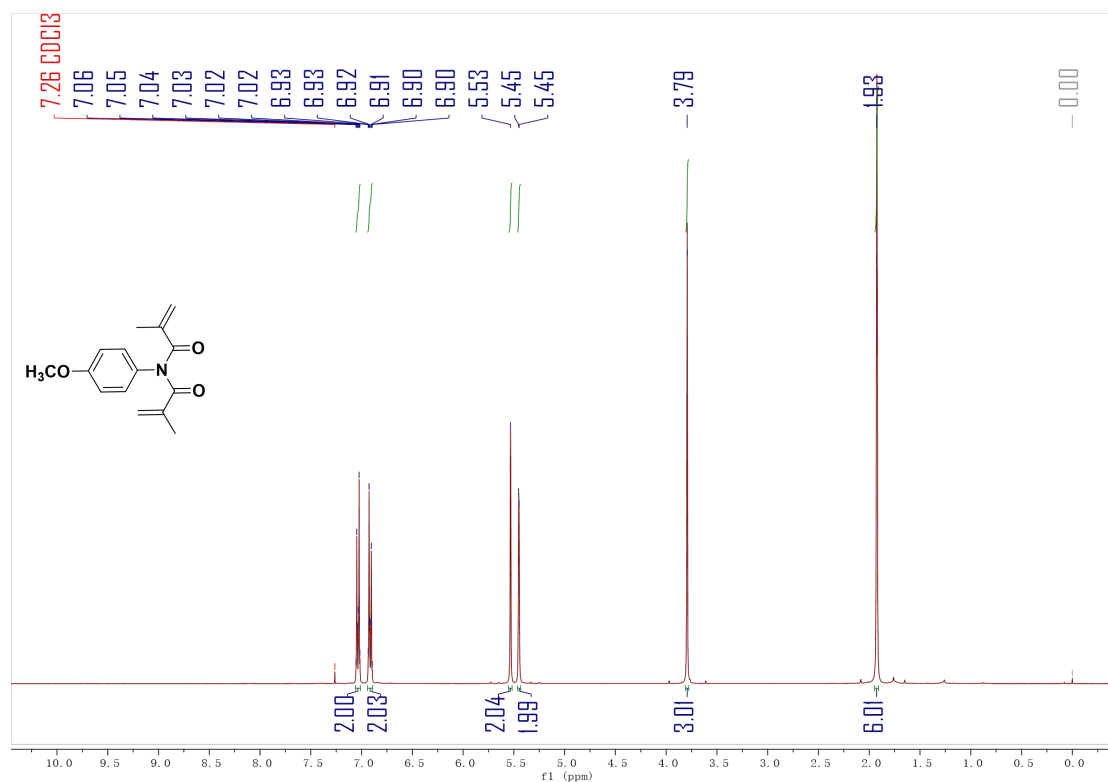
5. Copies of ^1H , ^{13}C , ^{77}Se and Spectra for Compounds



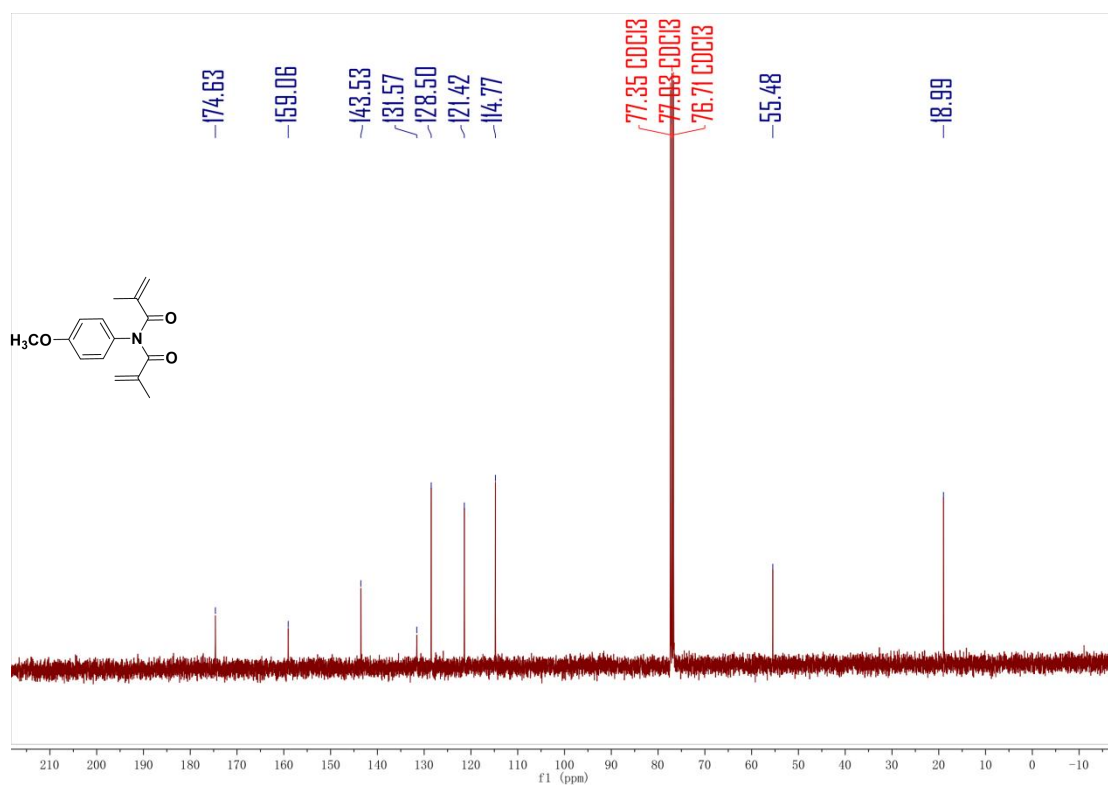
^1H NMR Spectra of 3a (400 MHz, CDCl_3)



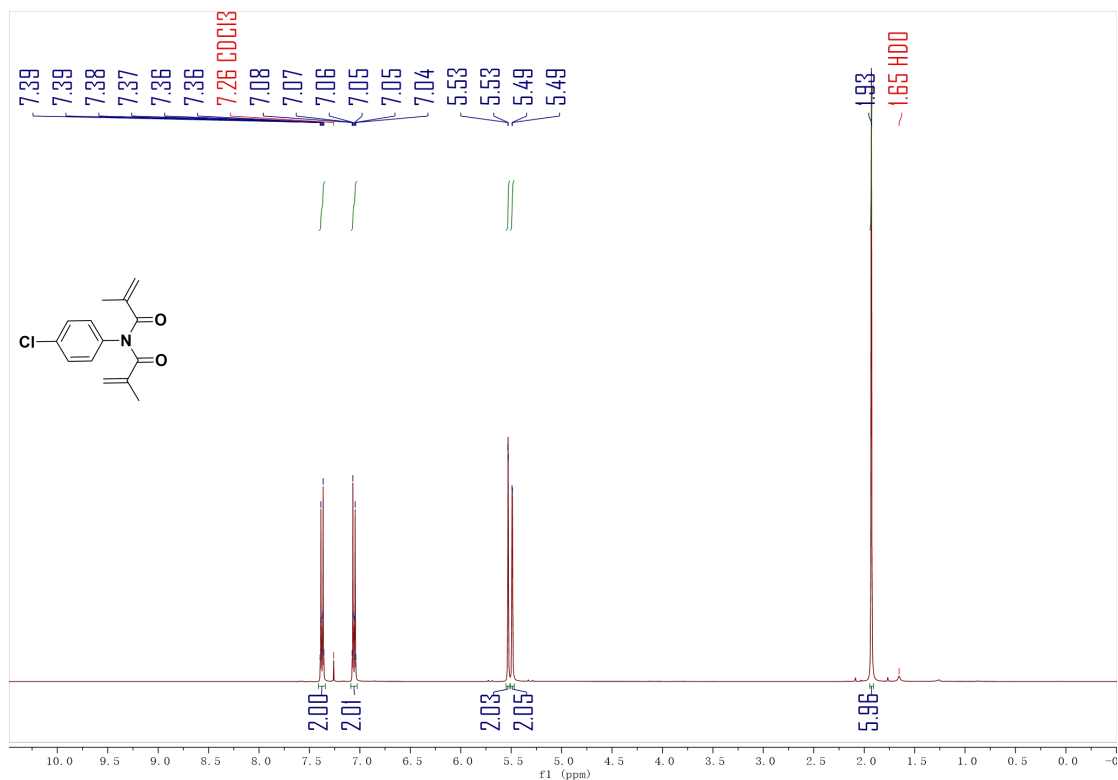
^{13}C NMR Spectra of 3a (100 MHz, CDCl_3)



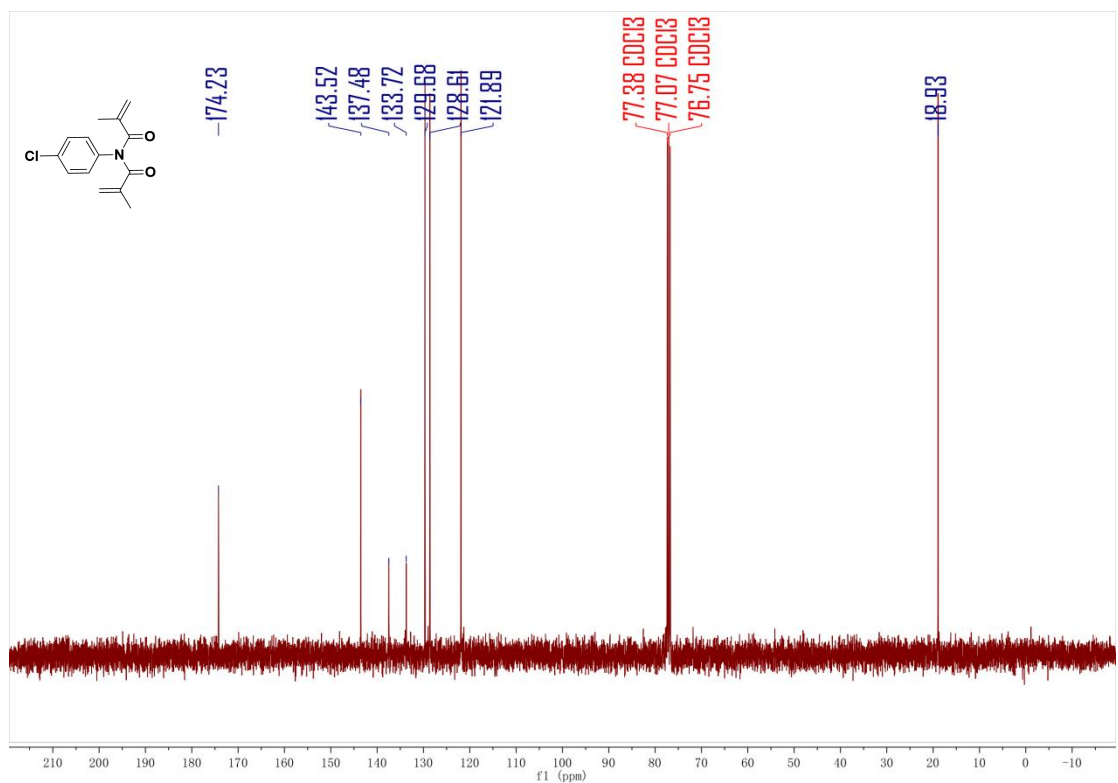
¹H NMR Spectra of 3b (400 MHz, CDCl₃)



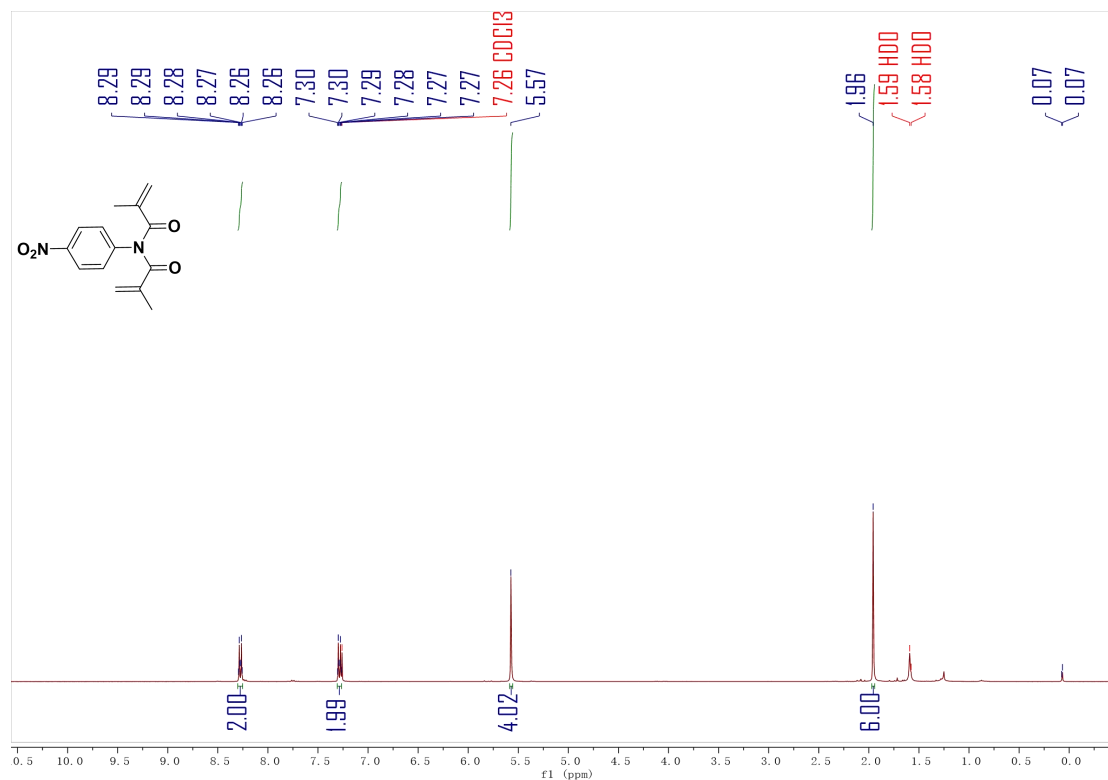
¹³C NMR Spectra of 3b (100 MHz, CDCl₃)



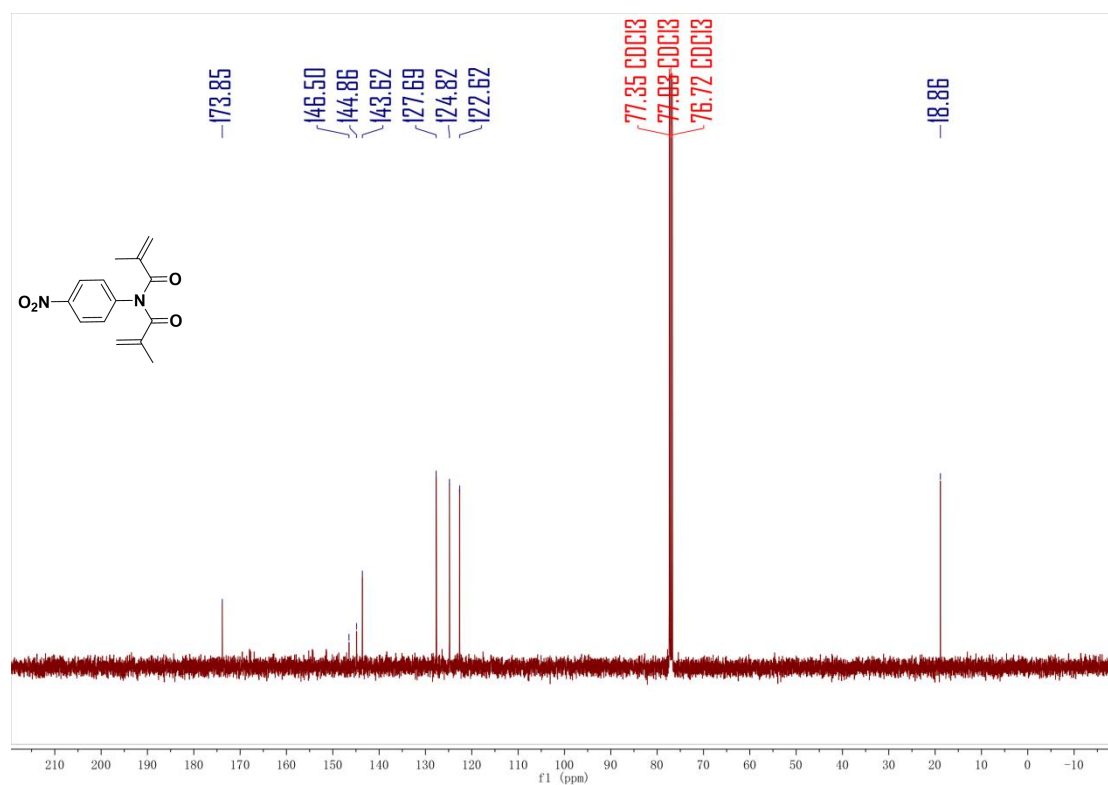
¹H NMR Spectra of 3c (400 MHz, CDCl₃)



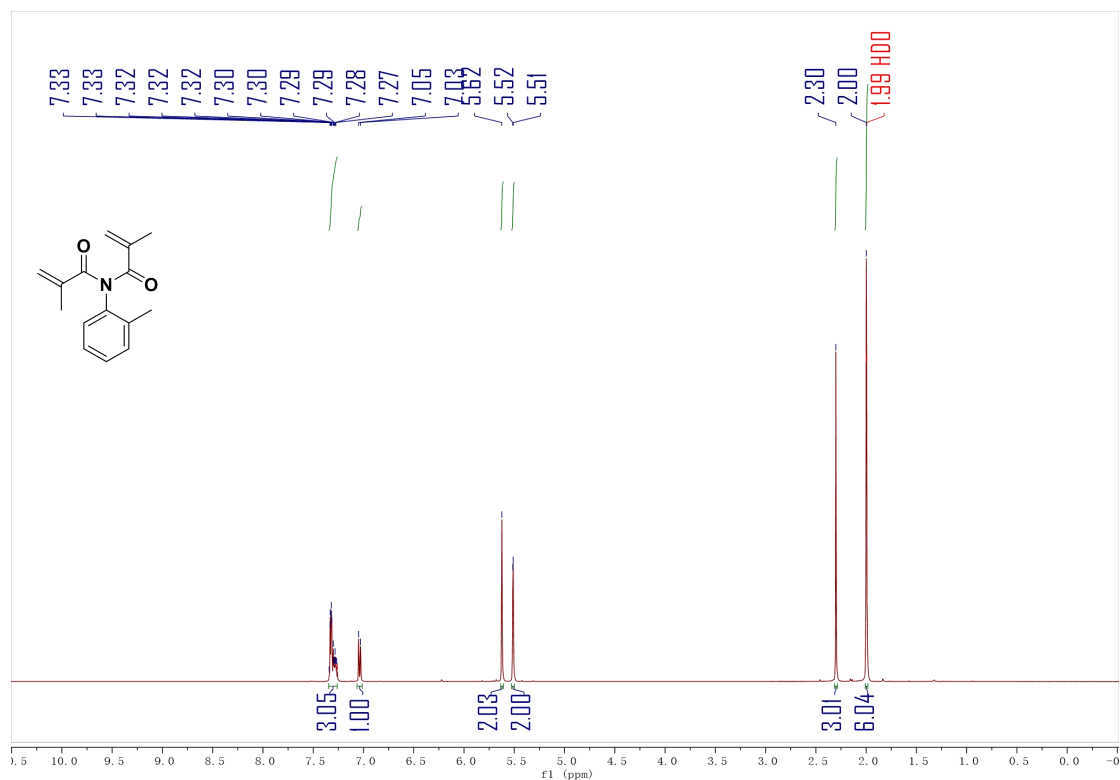
¹³C NMR Spectra of 3c (100 MHz, CDCl₃)



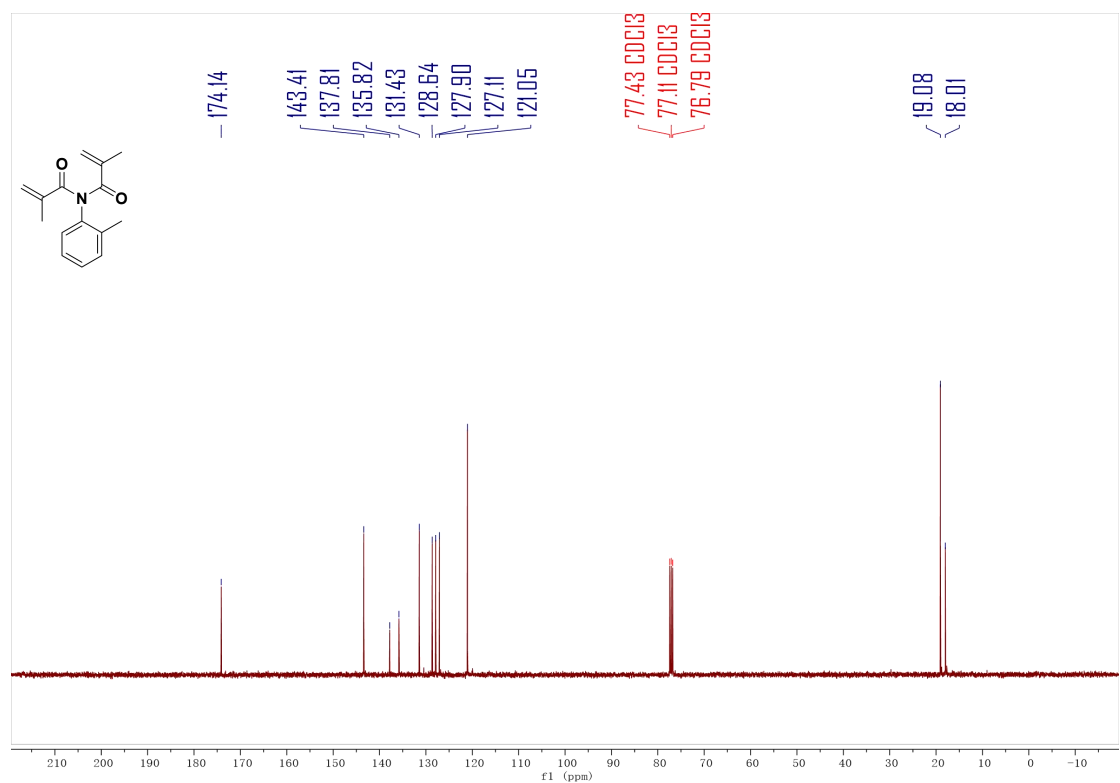
¹H NMR Spectra of 3d (400 MHz, CDCl₃)



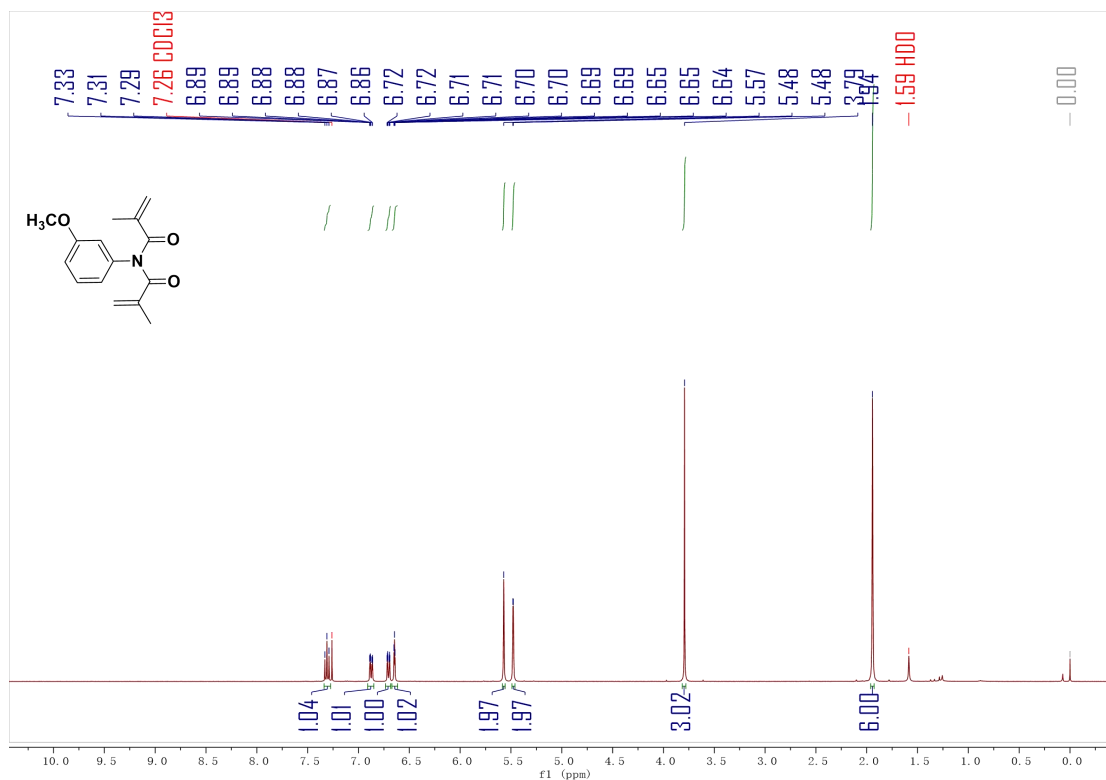
¹³C NMR Spectra of 3d (100 MHz, CDCl₃)



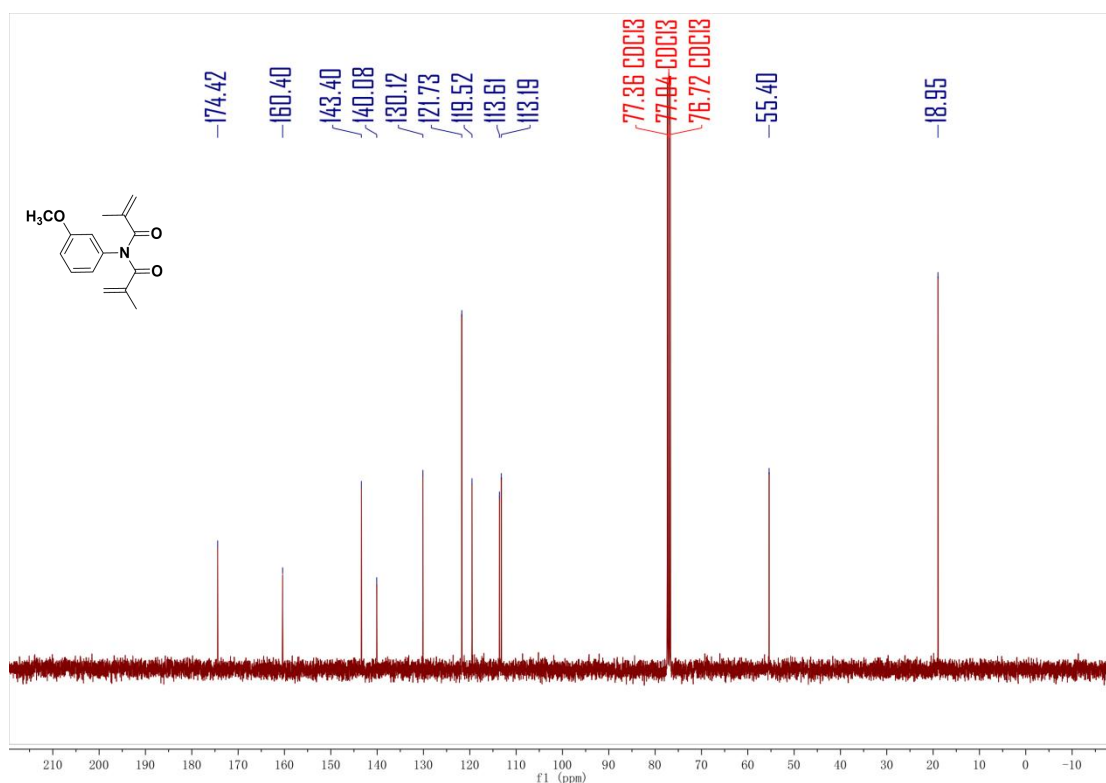
¹H NMR Spectra of 3e (400 MHz, CDCl₃)



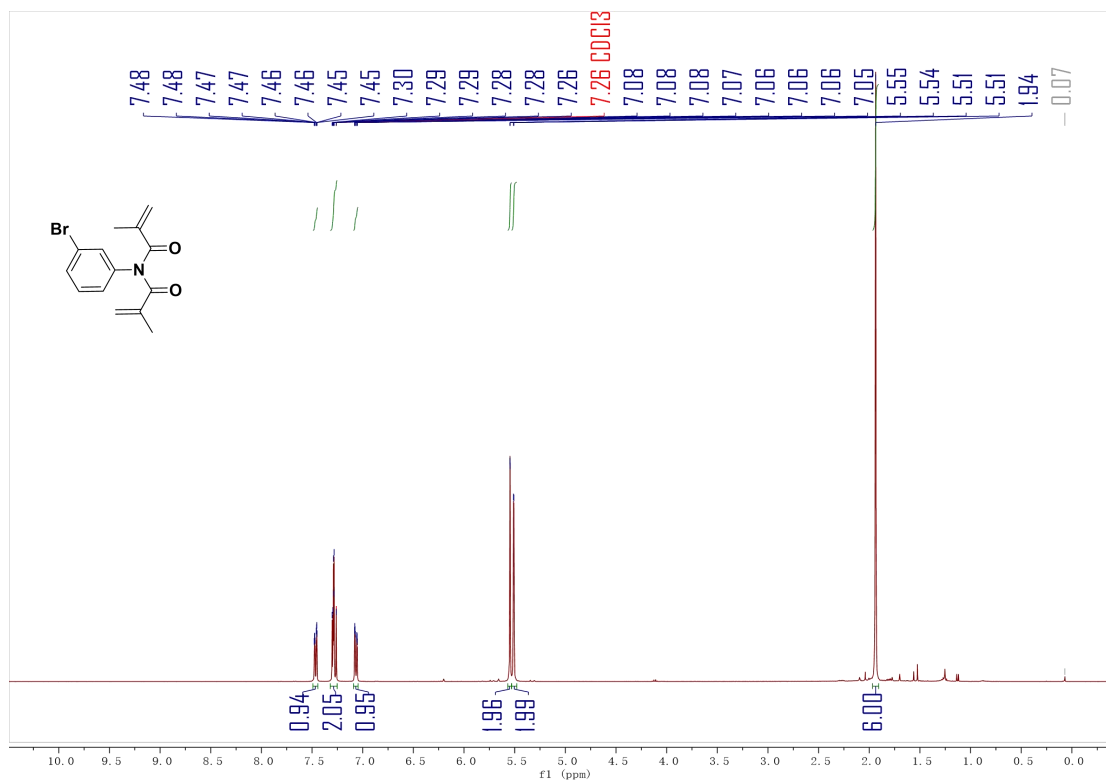
¹³C NMR Spectra of 3e (100 MHz, CDCl₃)



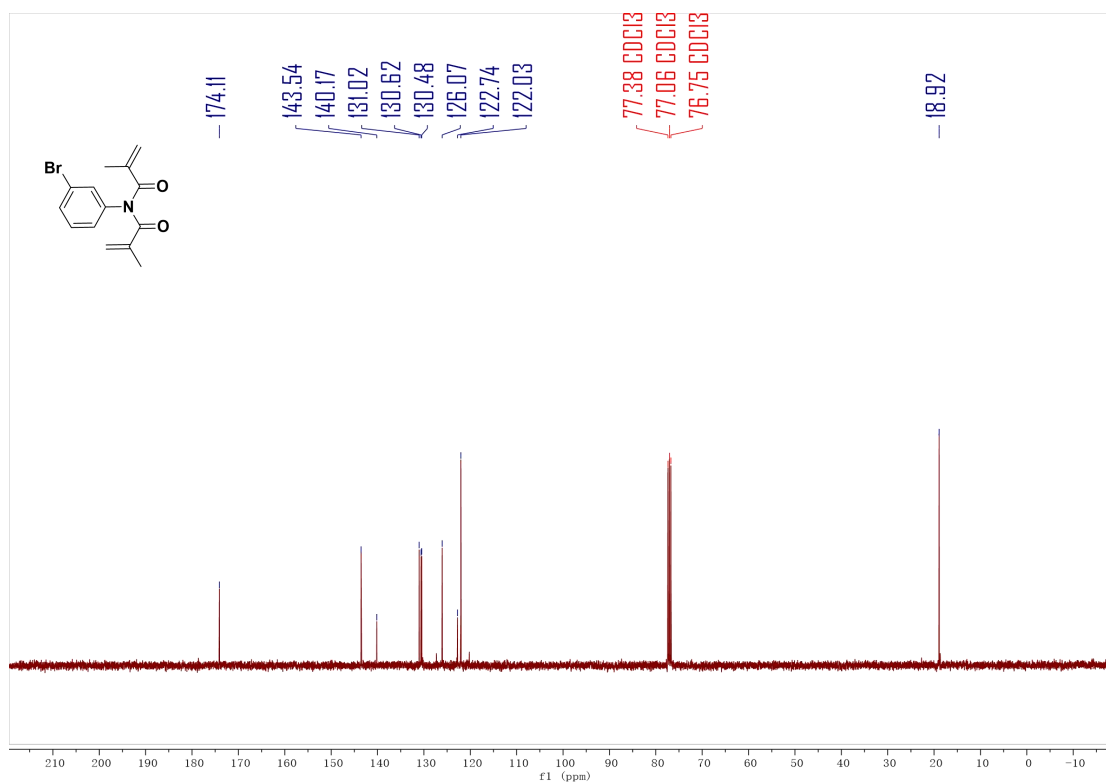
¹H NMR Spectra of 3f (400 MHz, CDCl₃)



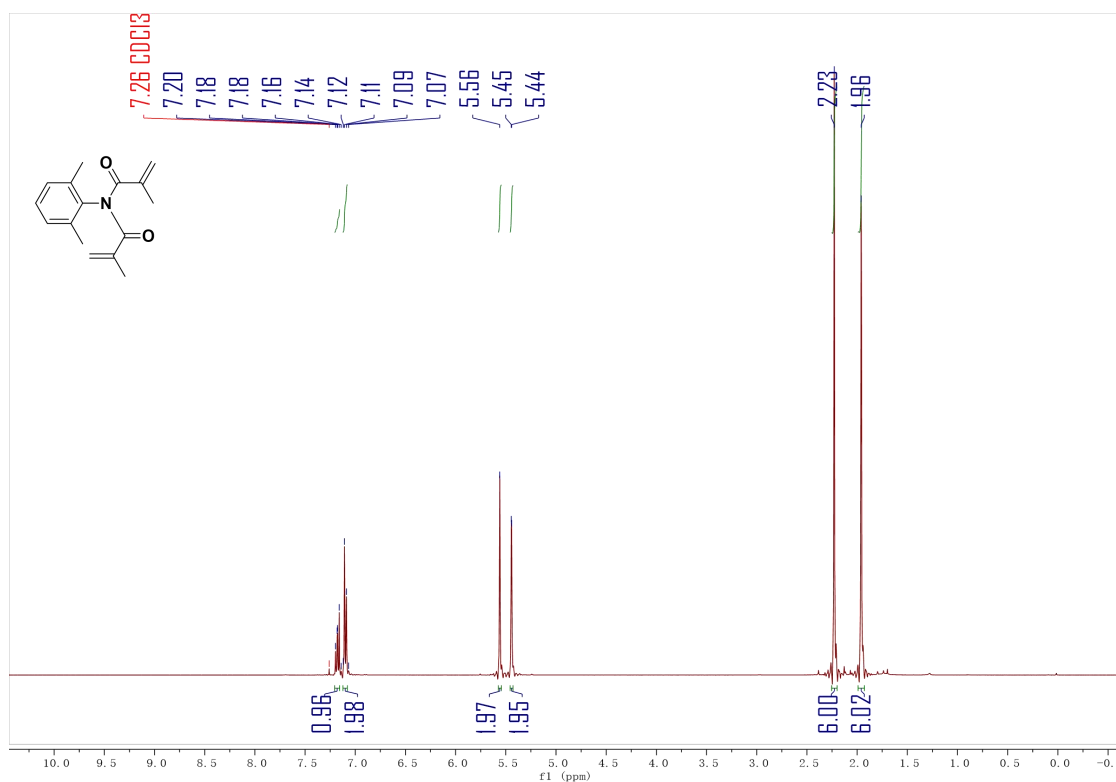
¹³C NMR Spectra of 3f (100 MHz, CDCl₃)



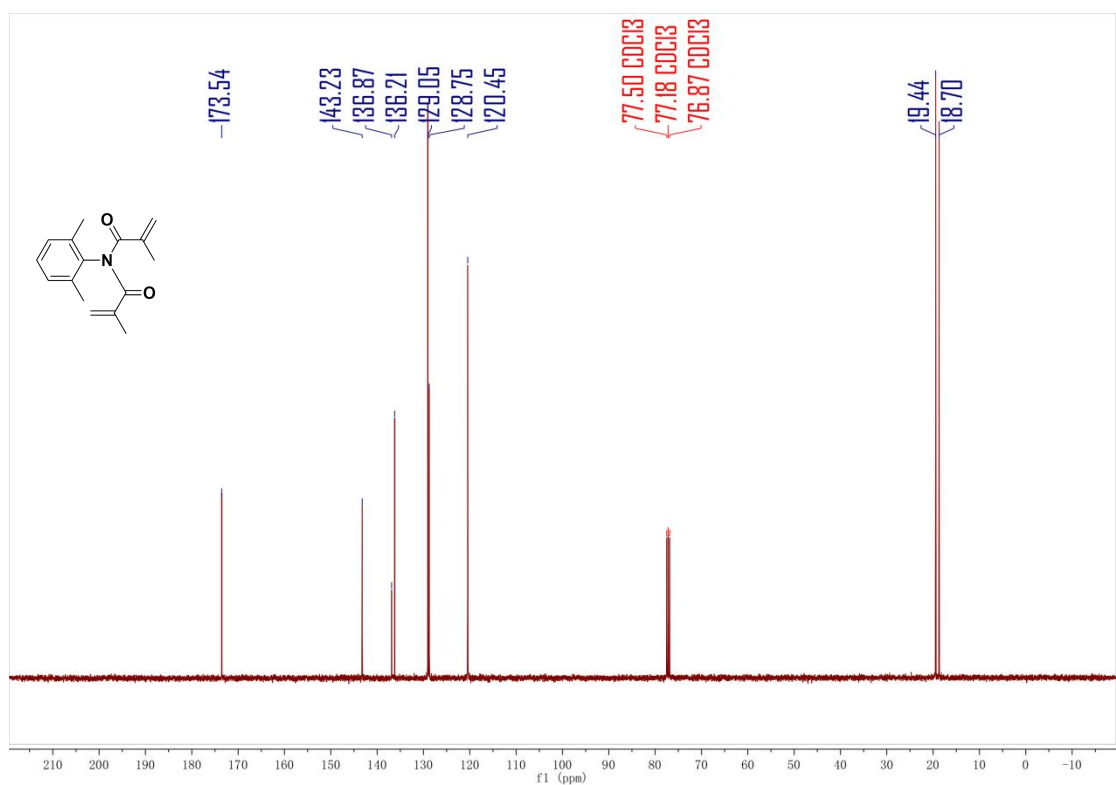
¹H NMR Spectra of 3g (400 MHz, CDCl₃)



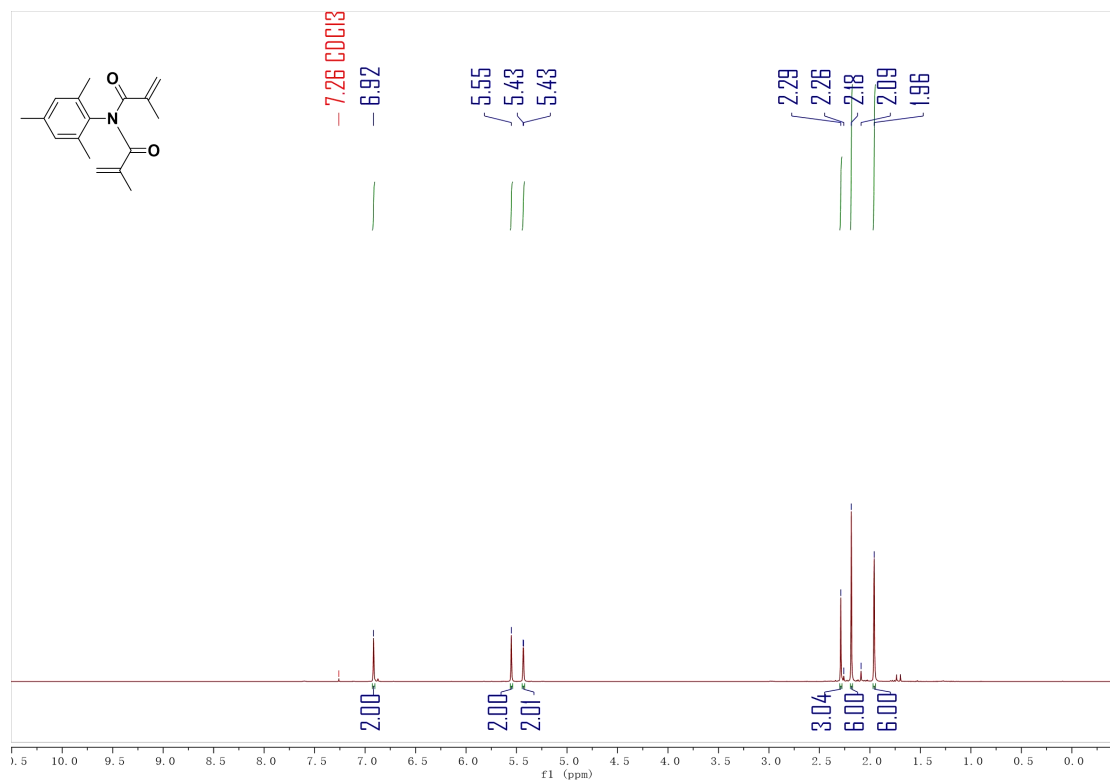
¹³C NMR Spectra of 3g (100 MHz, CDCl₃)



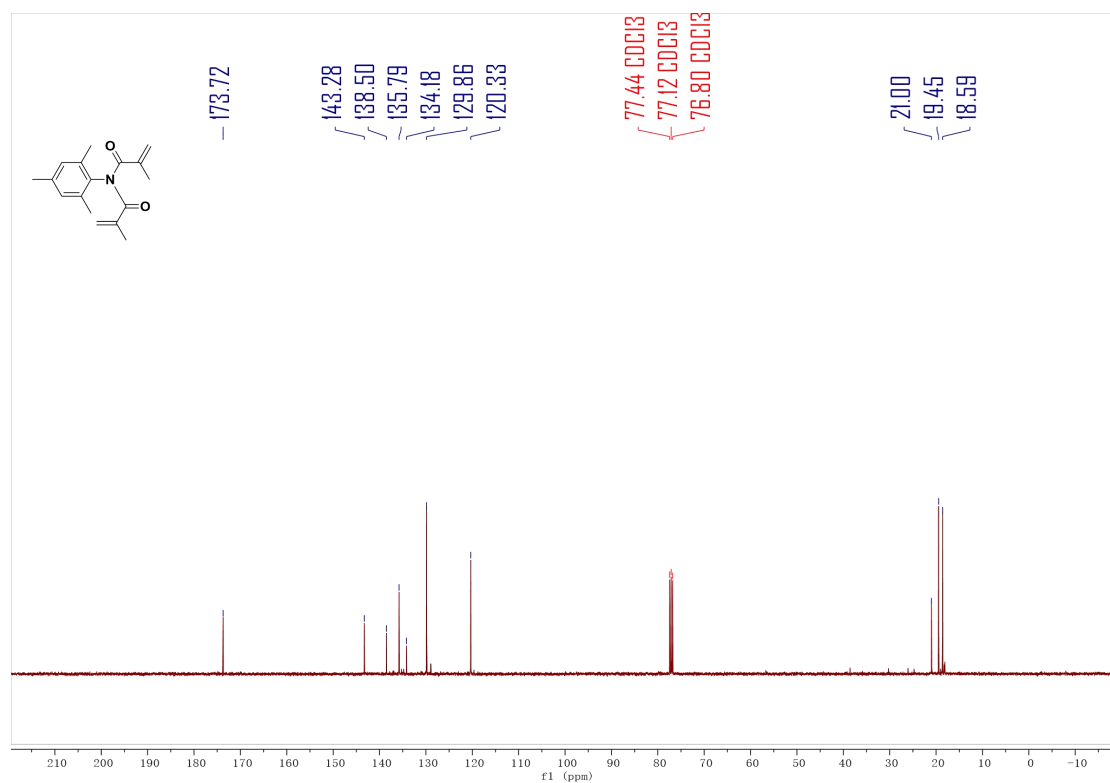
¹H NMR Spectra of 3h (400 MHz, CDCl₃)



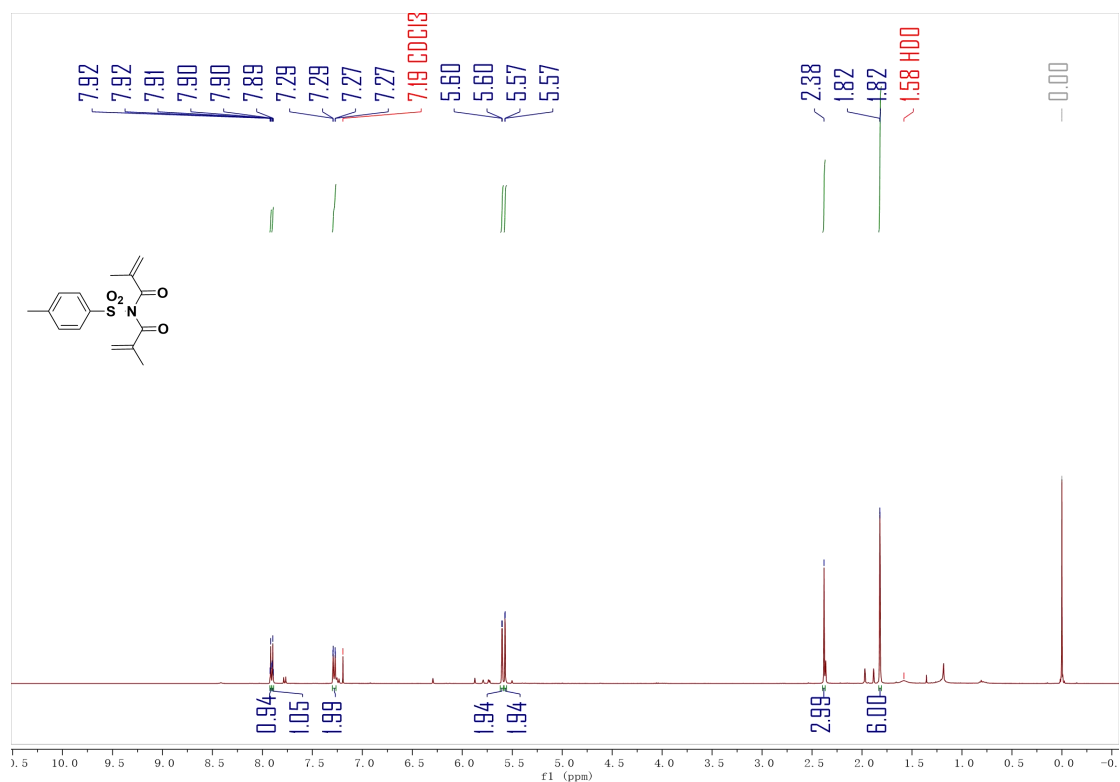
¹³C NMR Spectra of 3h (100 MHz, CDCl₃)



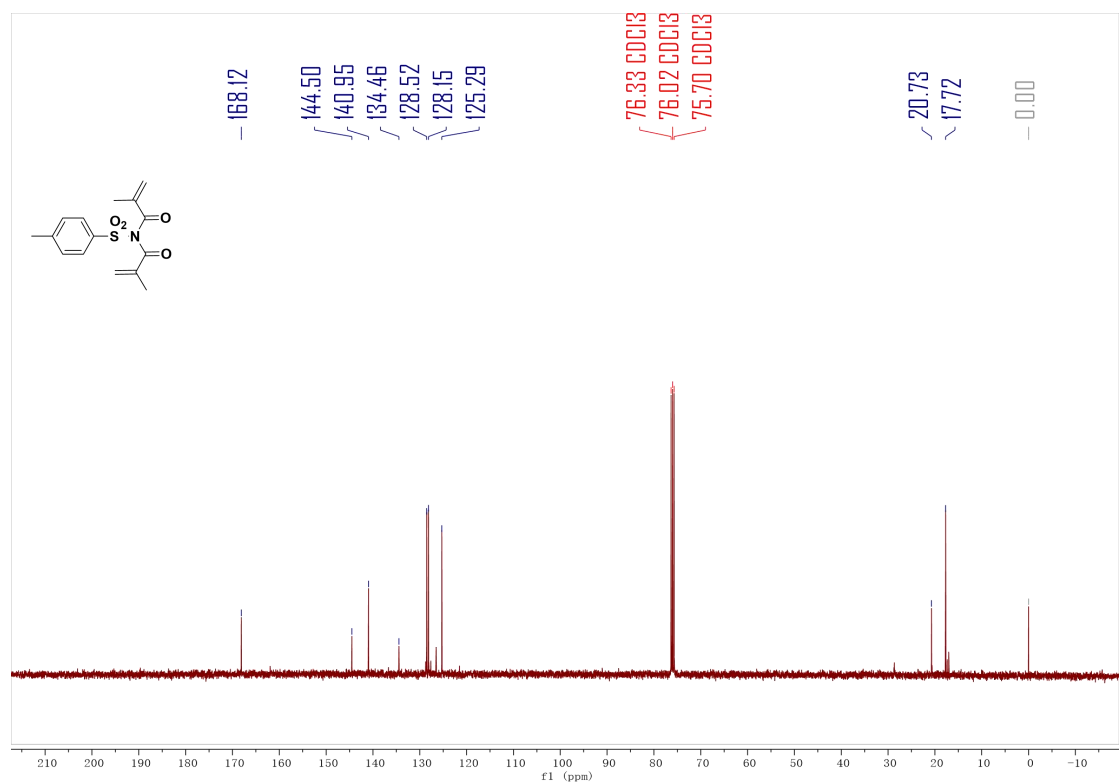
¹H NMR Spectra of 3i (400 MHz, CDCl₃)



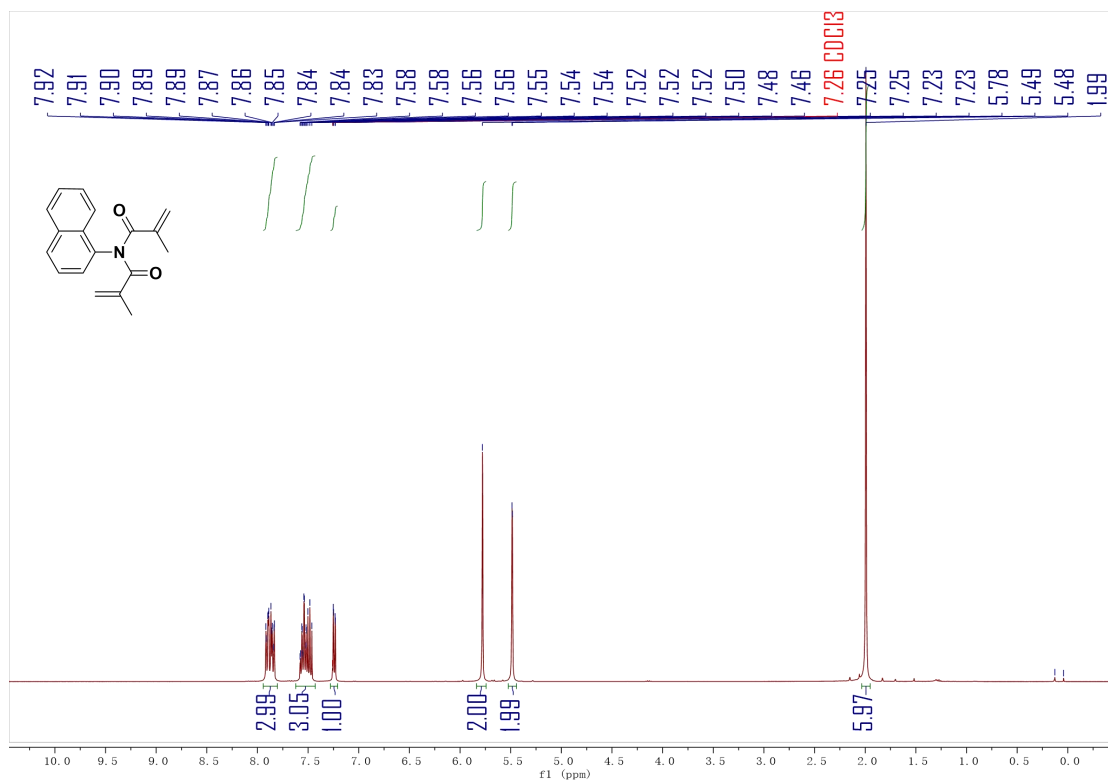
¹³C NMR Spectra of 3i (100 MHz, CDCl₃)



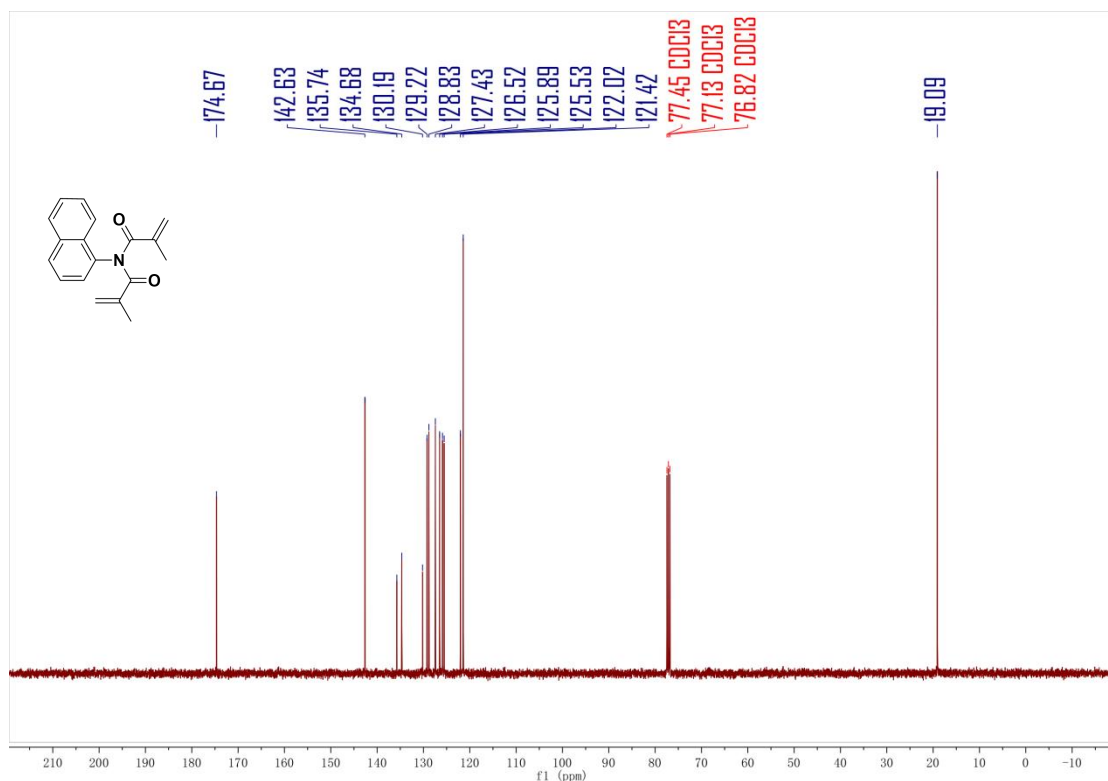
¹H NMR Spectra of 3j (400 MHz, CDCl₃)



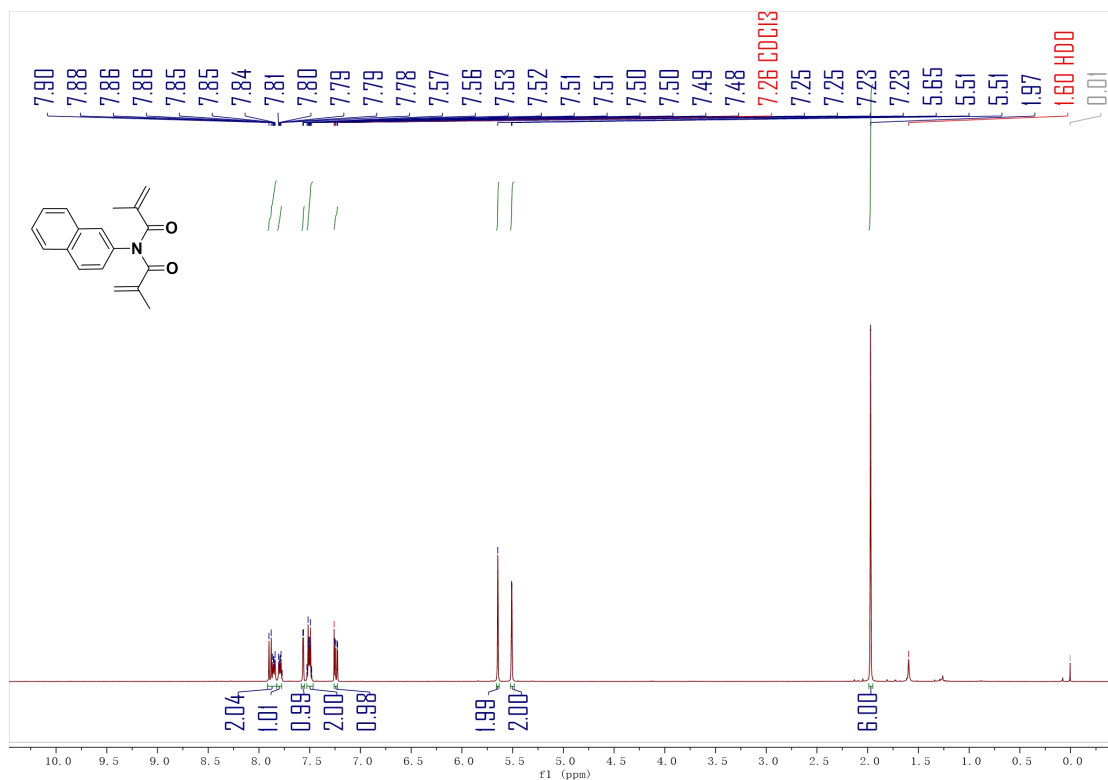
¹³C NMR Spectra of 3j (100 MHz, CDCl₃)



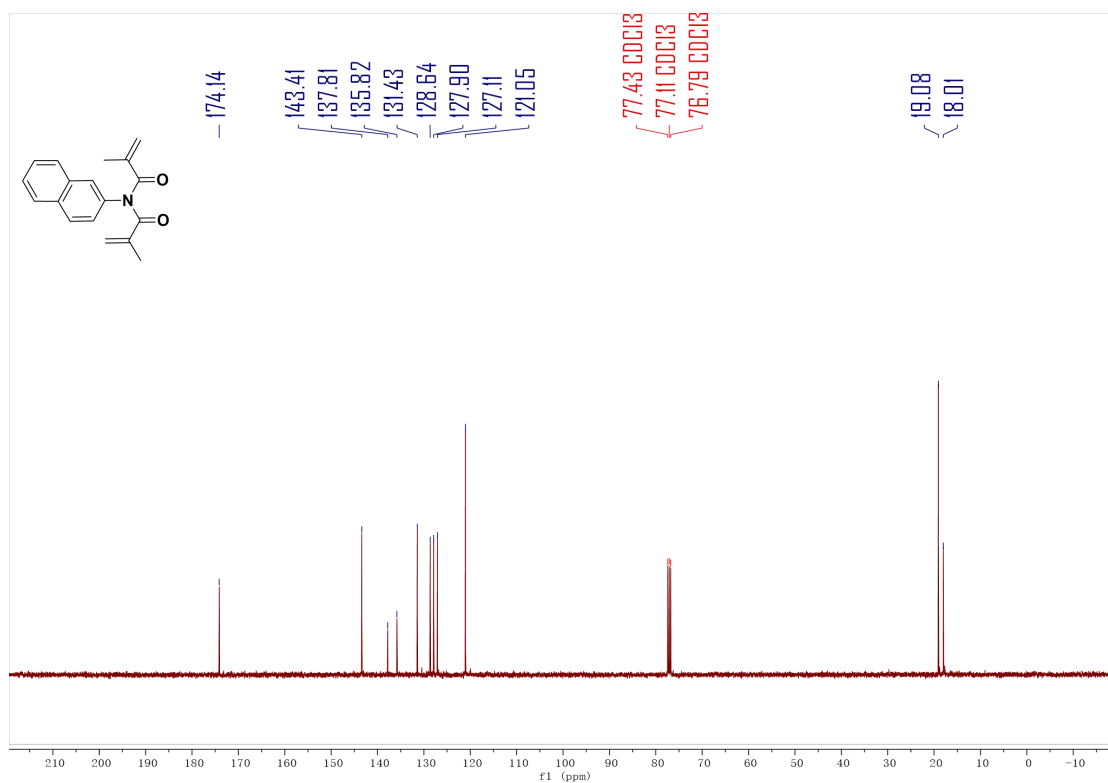
¹H NMR Spectra of 3k (400 MHz, CDCl₃)



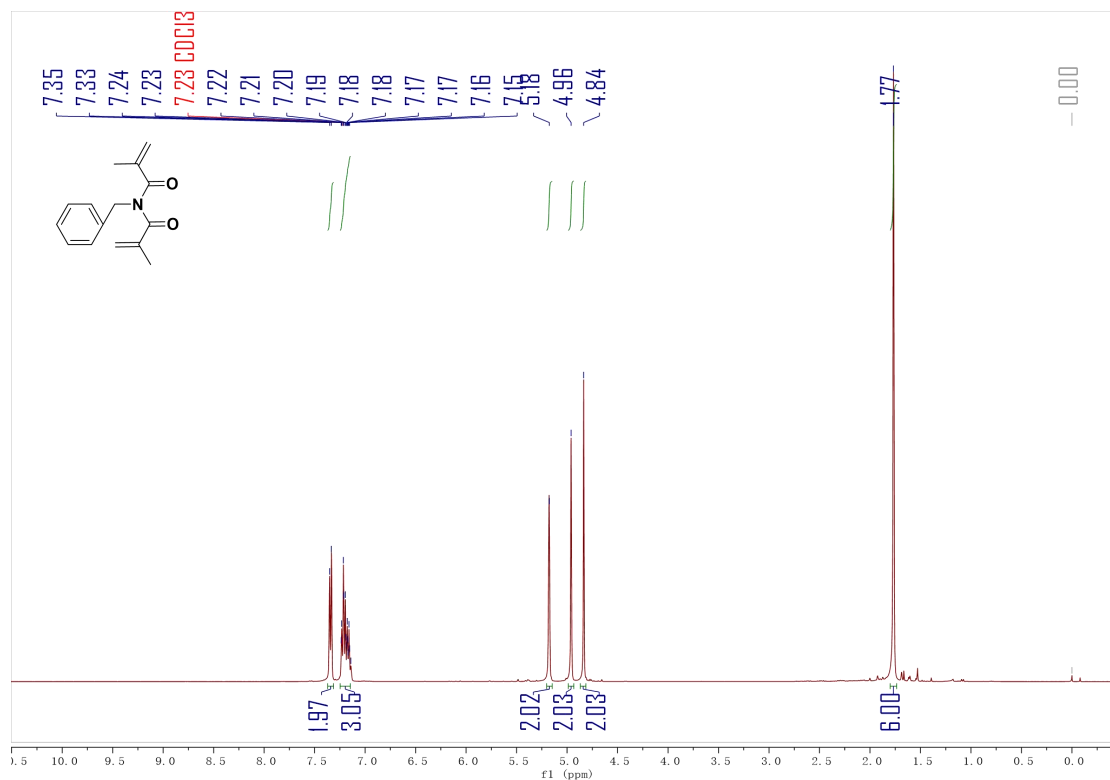
¹³C NMR Spectra of 3k (100 MHz, CDCl₃)



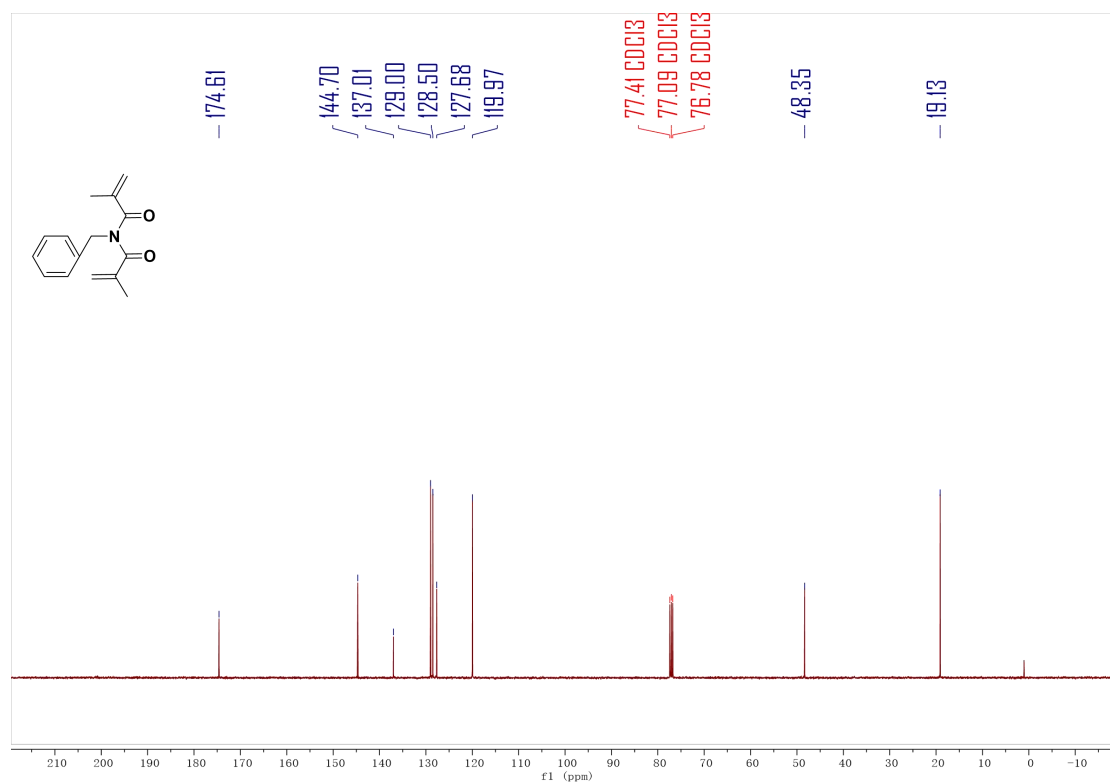
¹H NMR Spectra of 3l (400 MHz, CDCl₃)



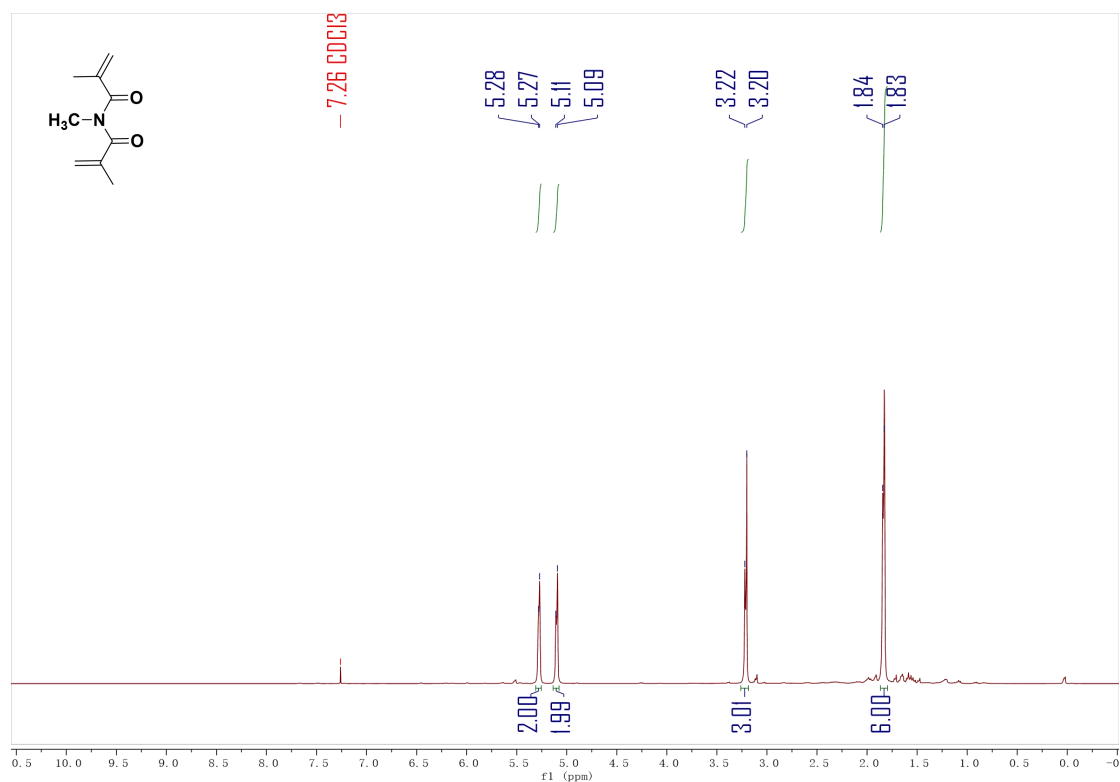
¹³C NMR Spectra of 3l (100 MHz, CDCl₃)



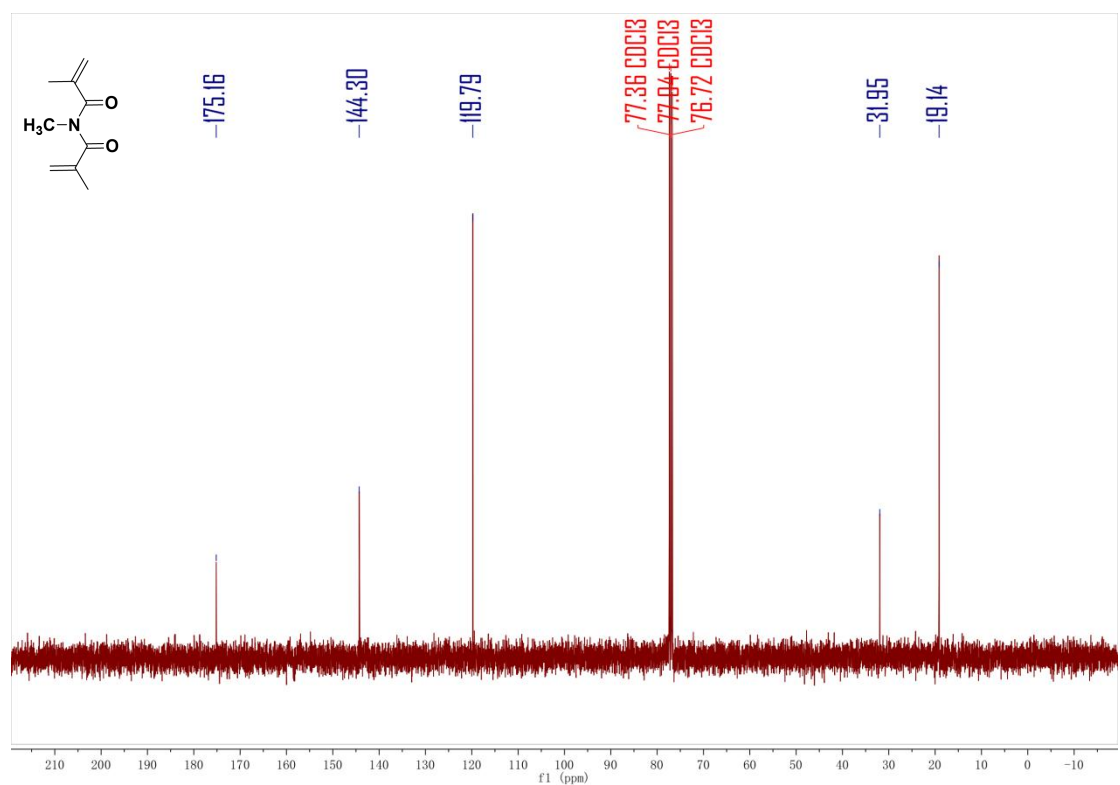
¹H NMR Spectra of 3m (400 MHz, CDCl₃)



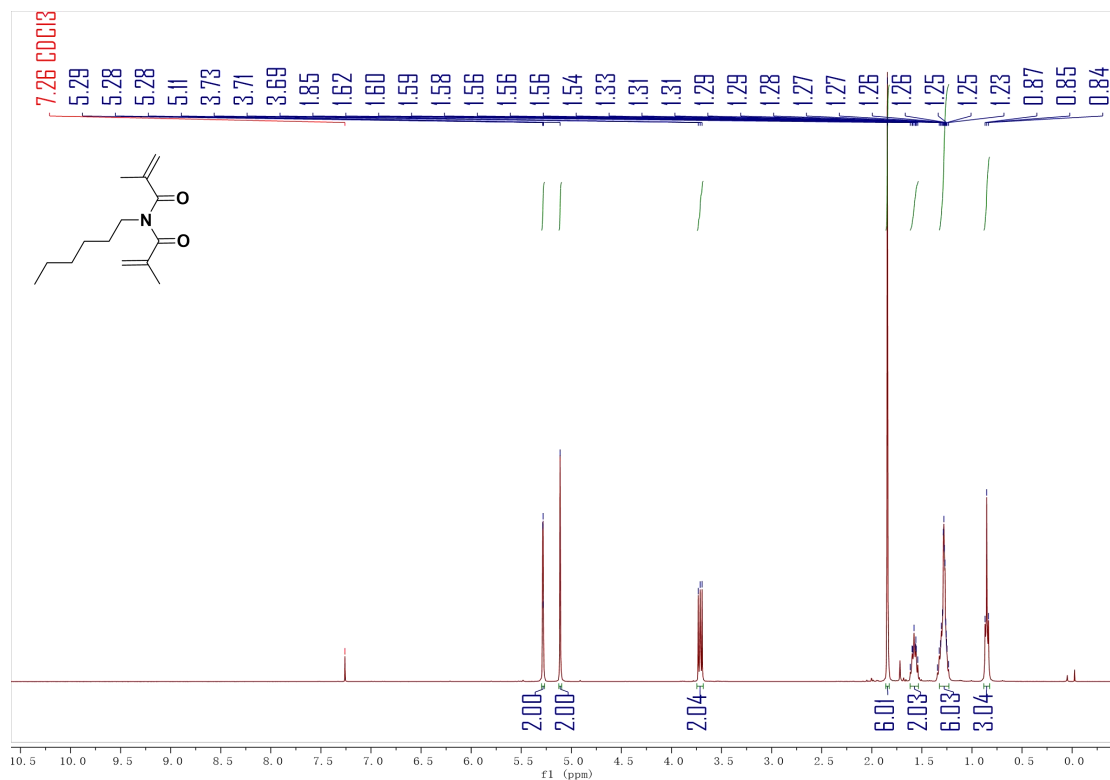
¹³C NMR Spectra of 3m (100 MHz, CDCl₃)



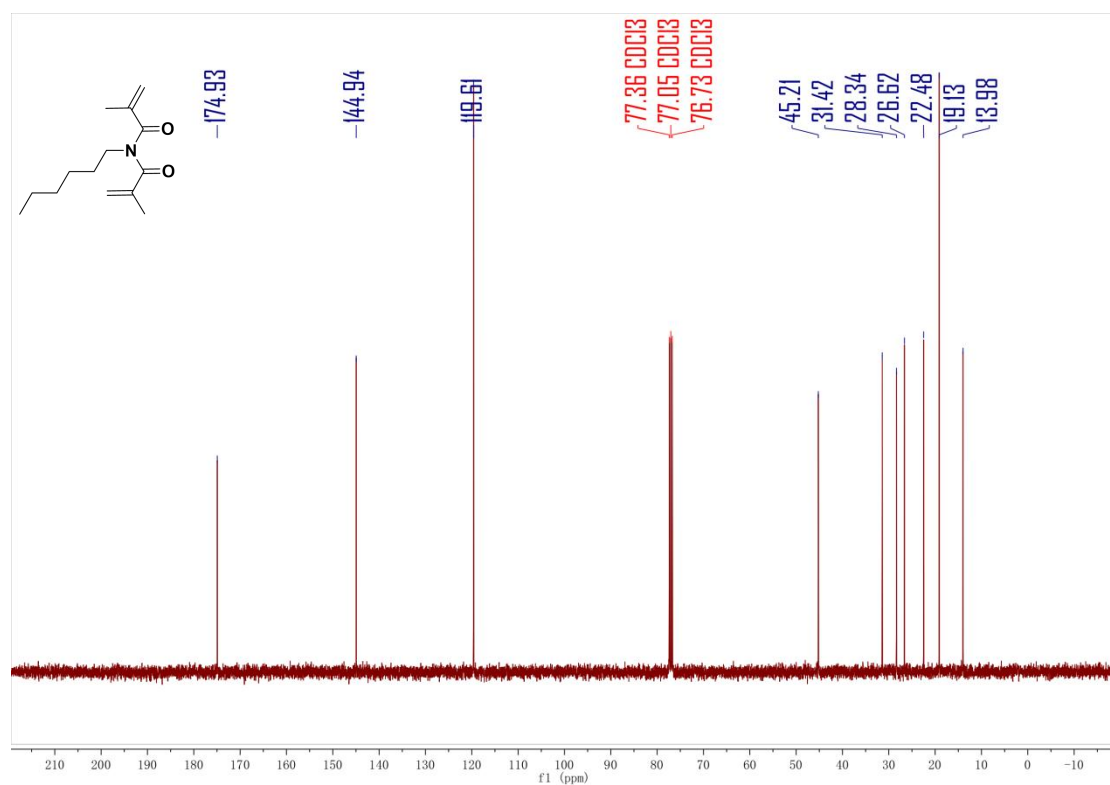
¹H NMR Spectra of 3n (400 MHz, CDCl₃)



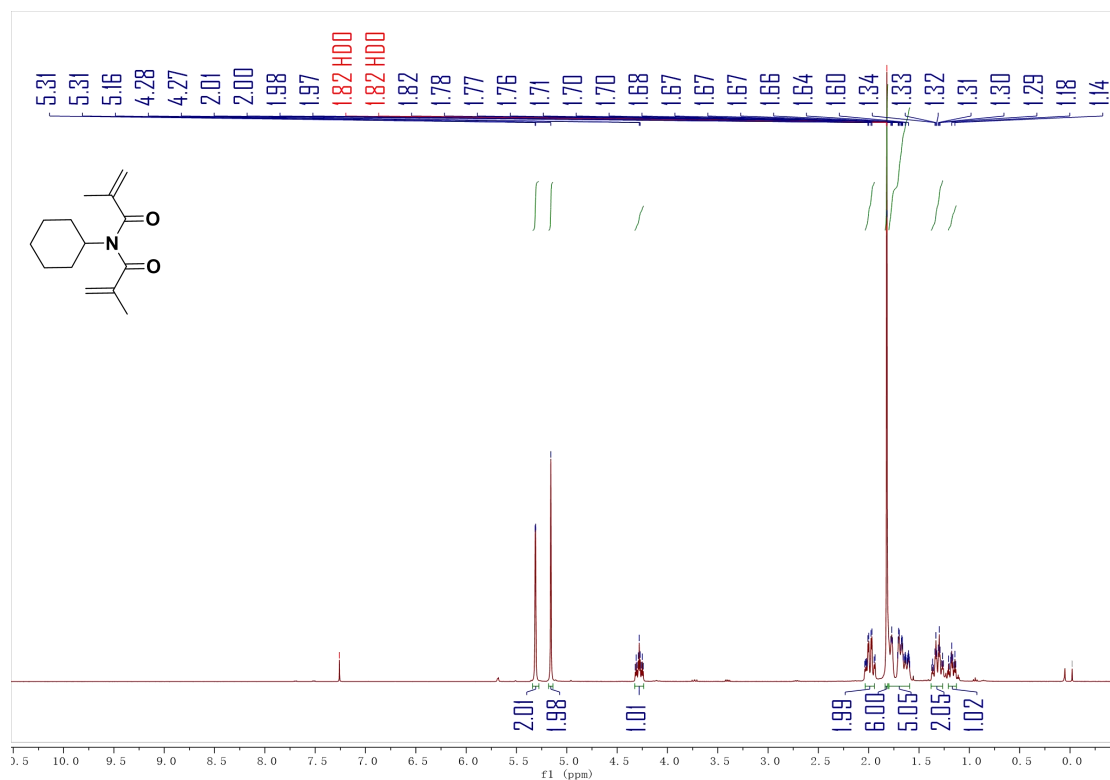
¹³C NMR Spectra of 3n (100 MHz, CDCl₃)



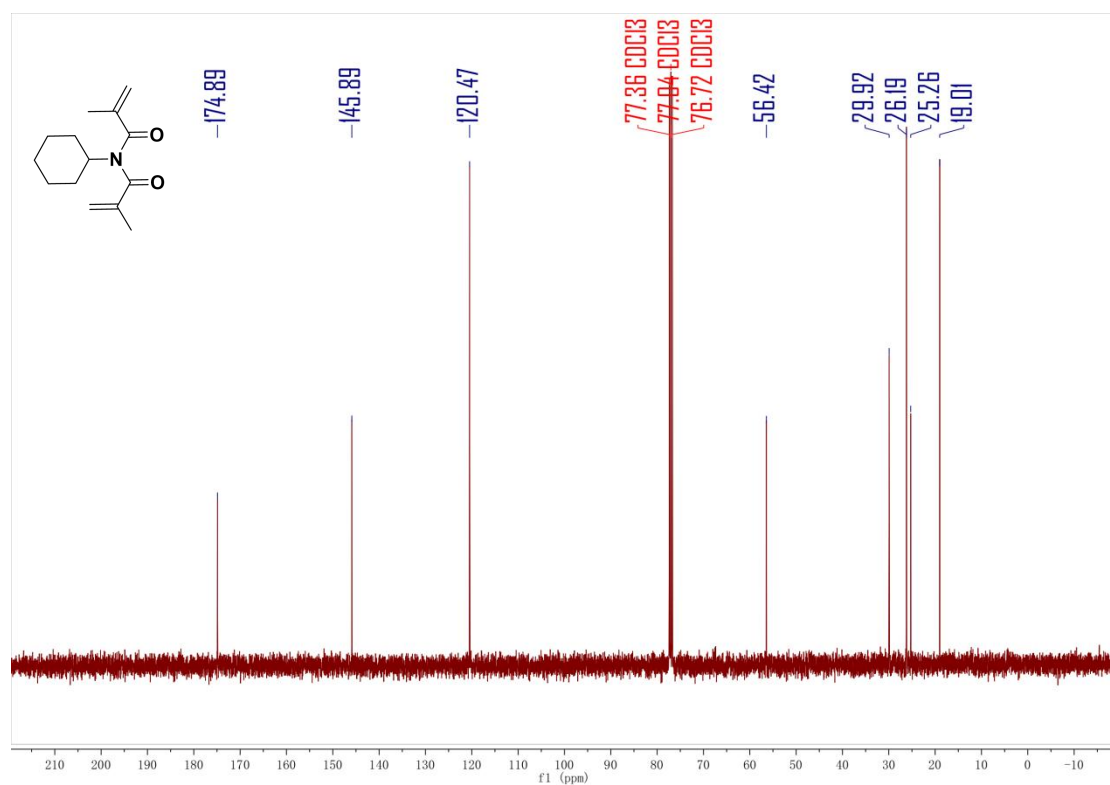
¹H NMR Spectra of 3o (400 MHz, CDCl₃)



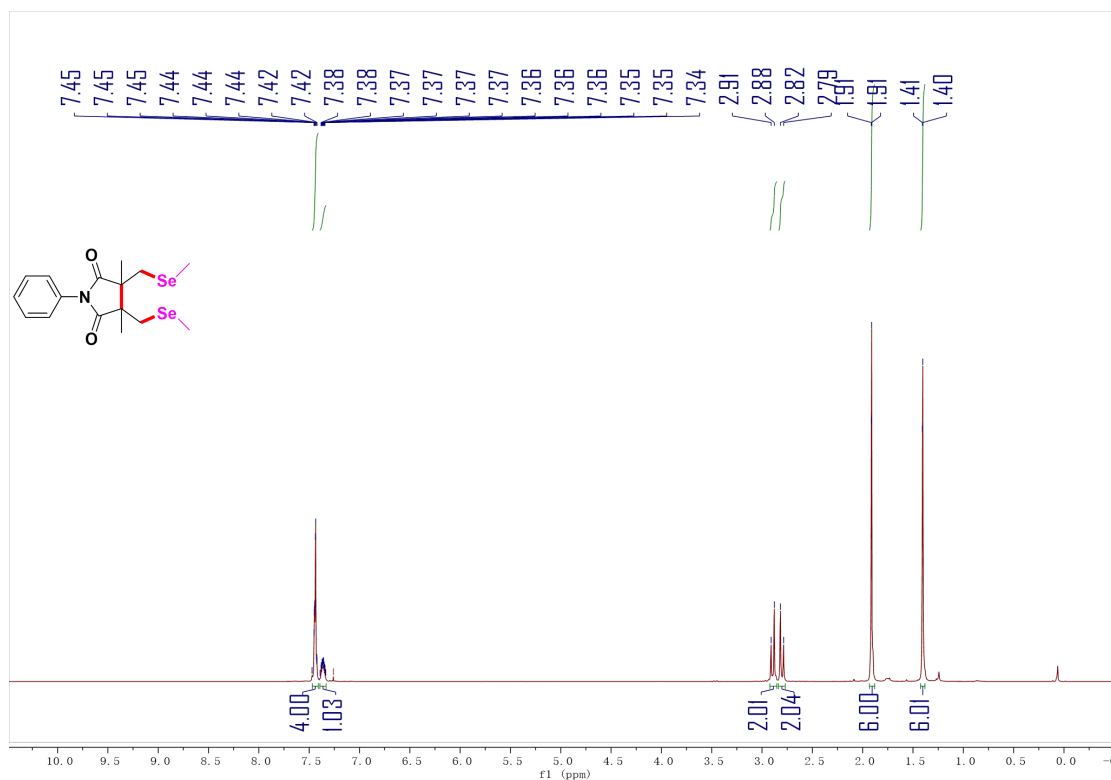
¹³C NMR Spectra of 3o (100 MHz, CDCl₃)



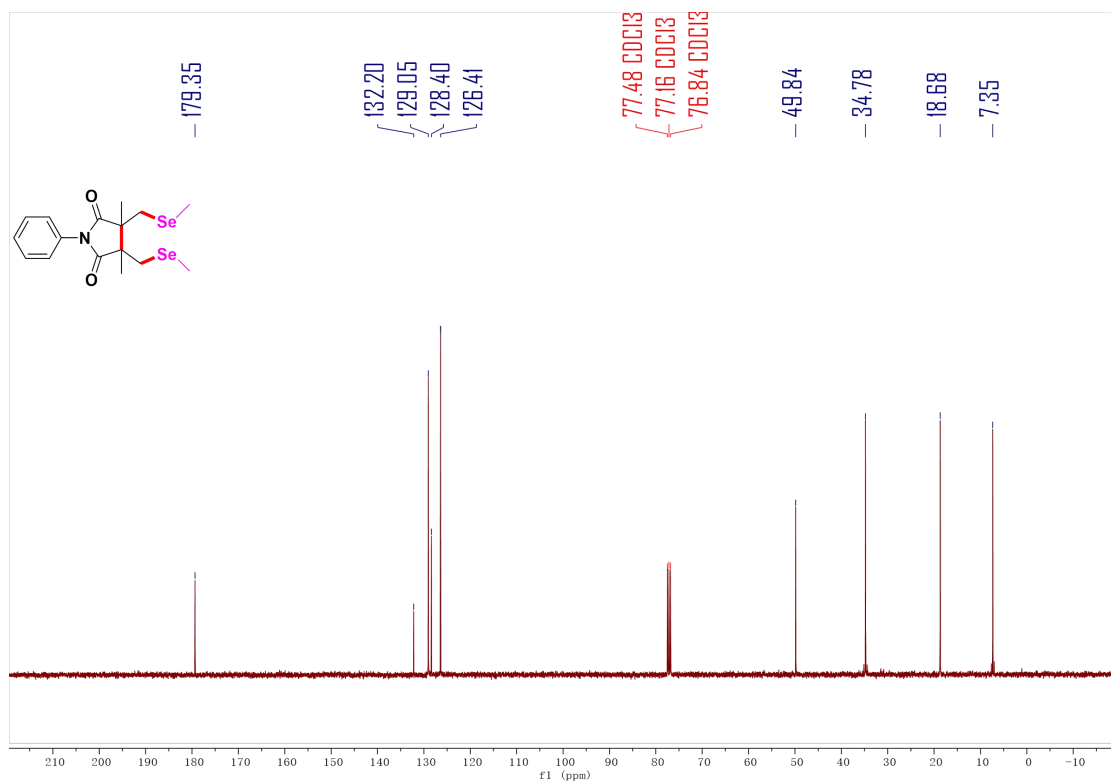
¹H NMR Spectra of 3p (400 MHz, CDCl₃)



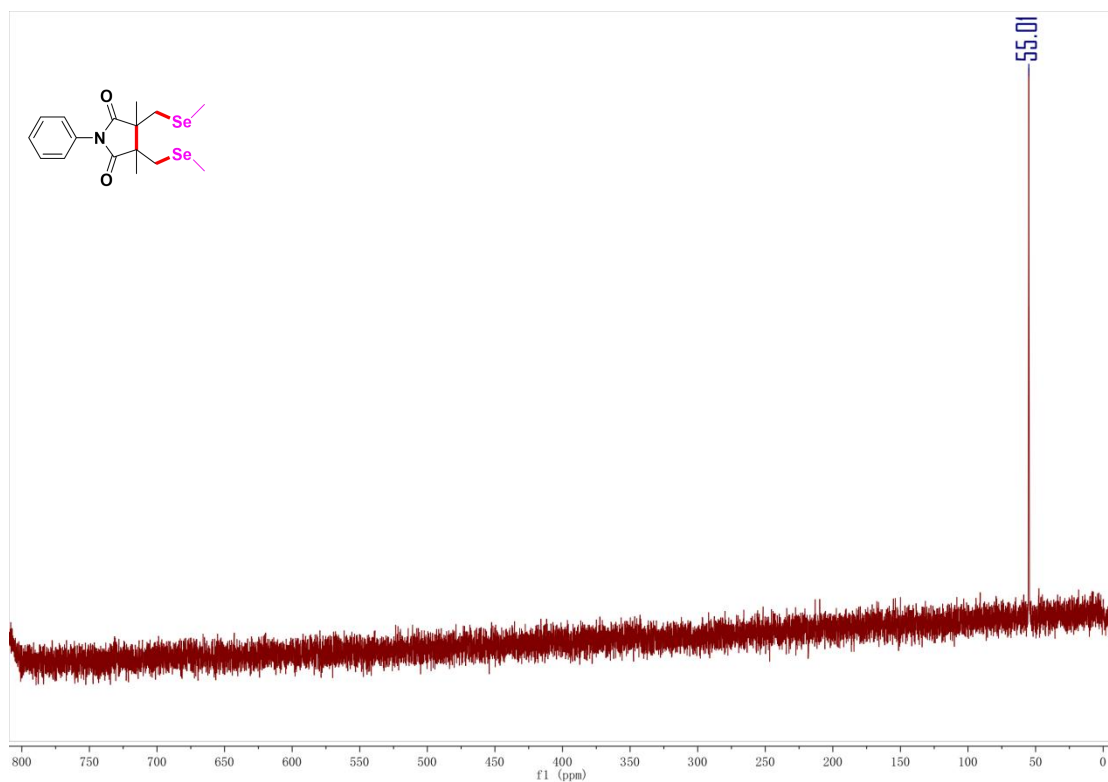
¹³C NMR Spectra of 3p (100 MHz, CDCl₃)



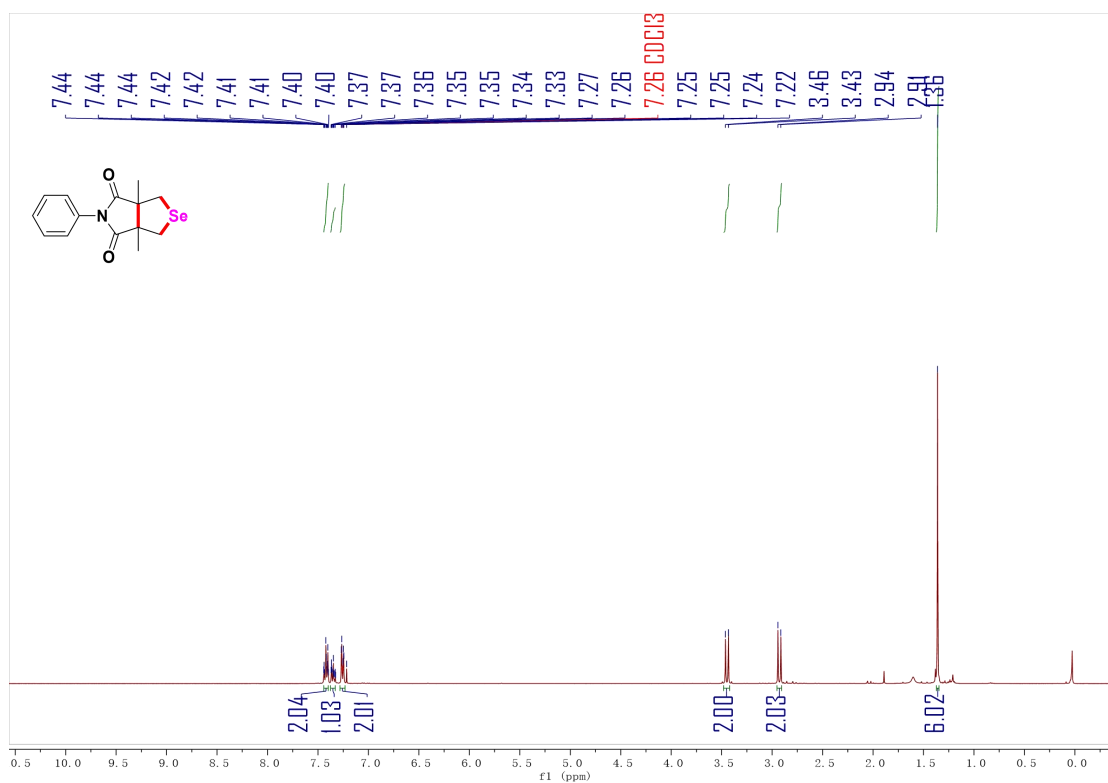
¹H NMR Spectra of 5a (400 MHz, CDCl₃)



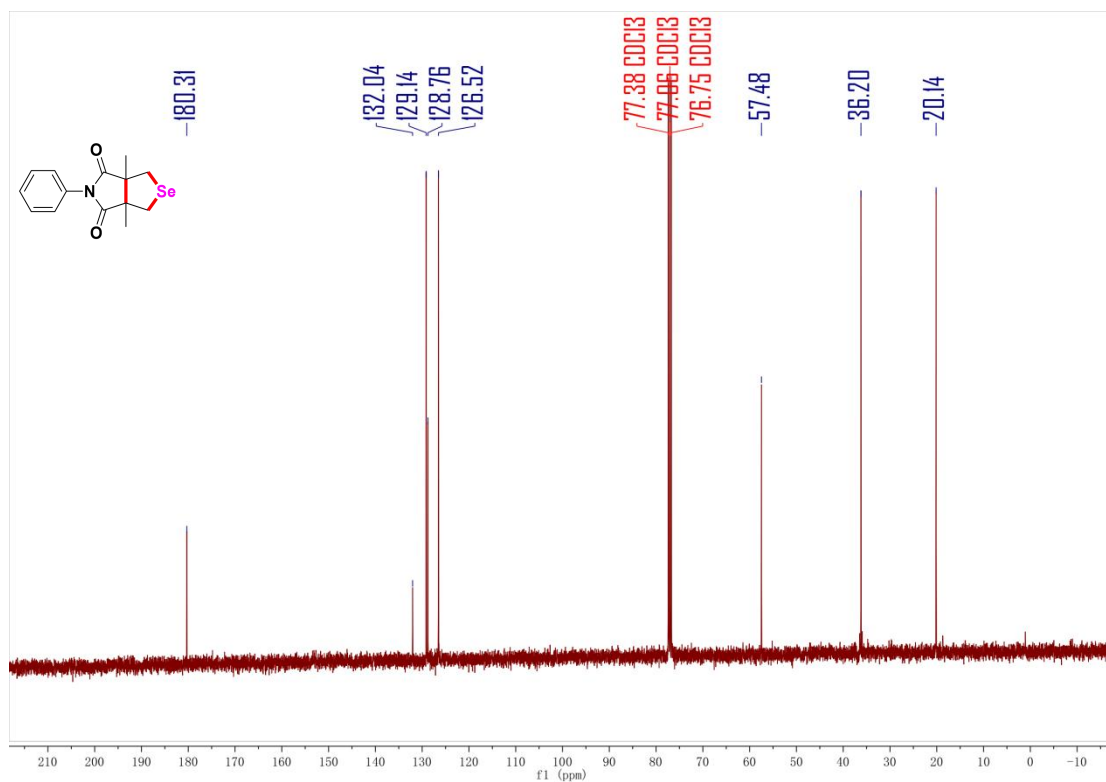
¹³C NMR Spectra of 5a (100 MHz, CDCl₃)



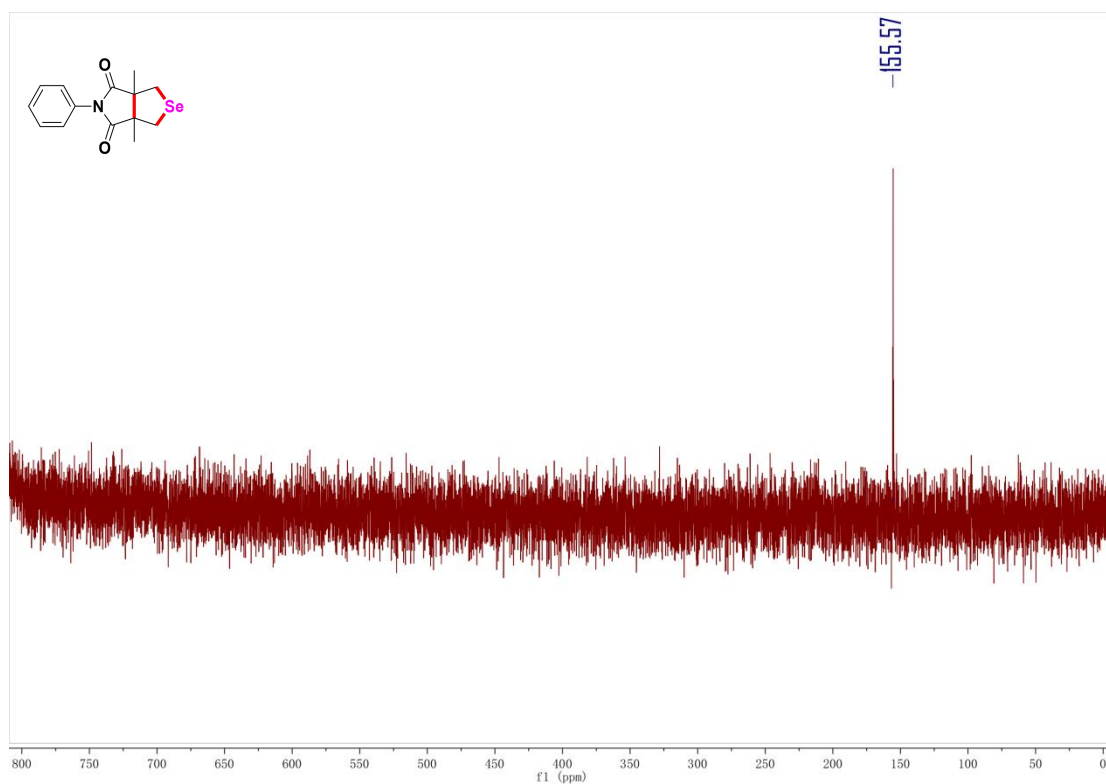
⁷⁷Se NMR Spectra of 5a (76 MHz, CDCl₃)



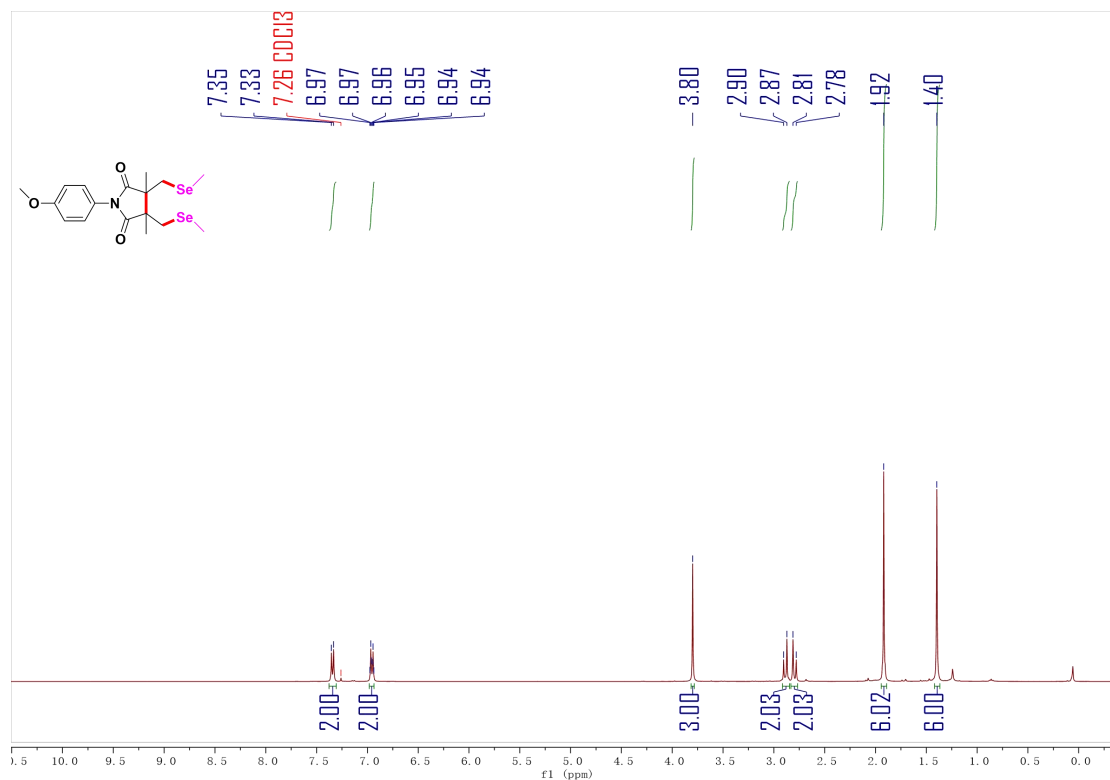
¹H NMR Spectra of 6a (400 MHz, CDCl₃)



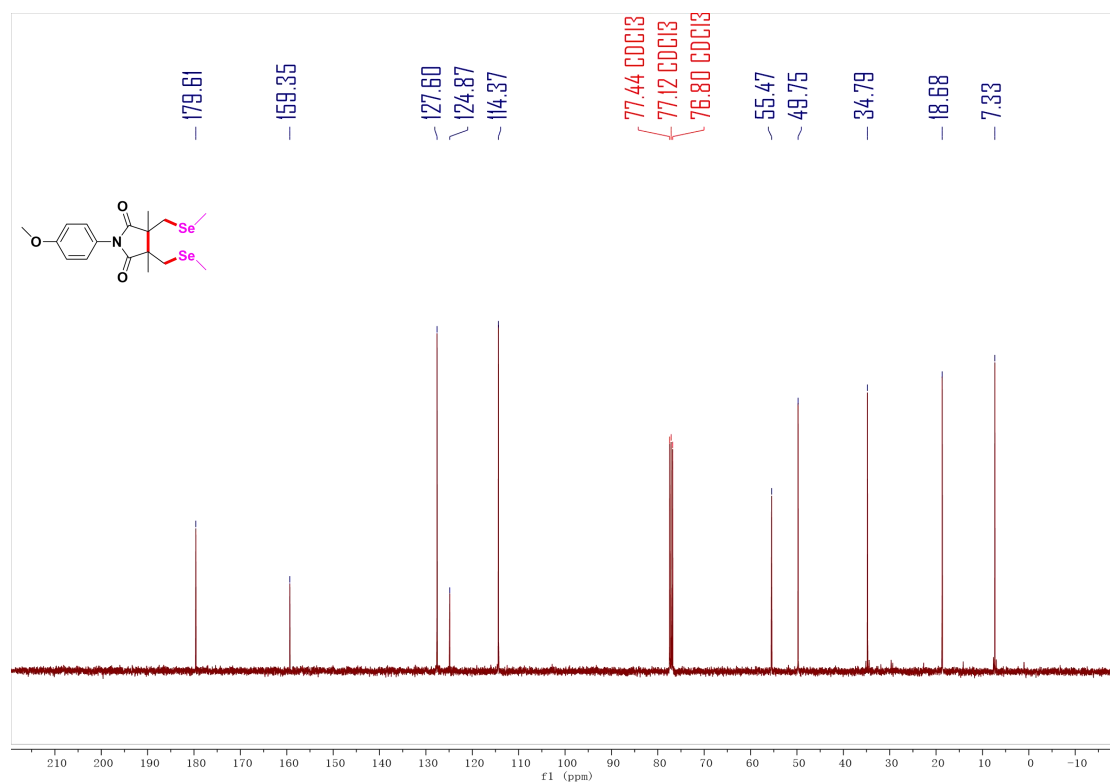
¹³C NMR Spectra of 6a (100 MHz, CDCl₃)



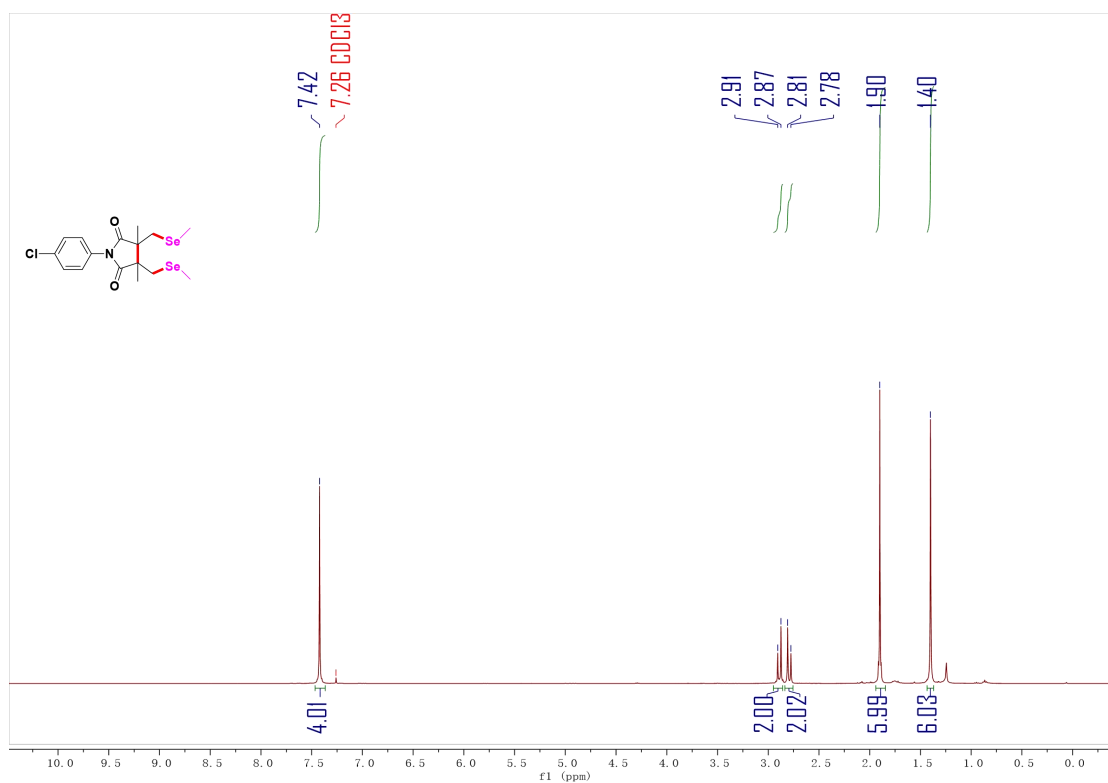
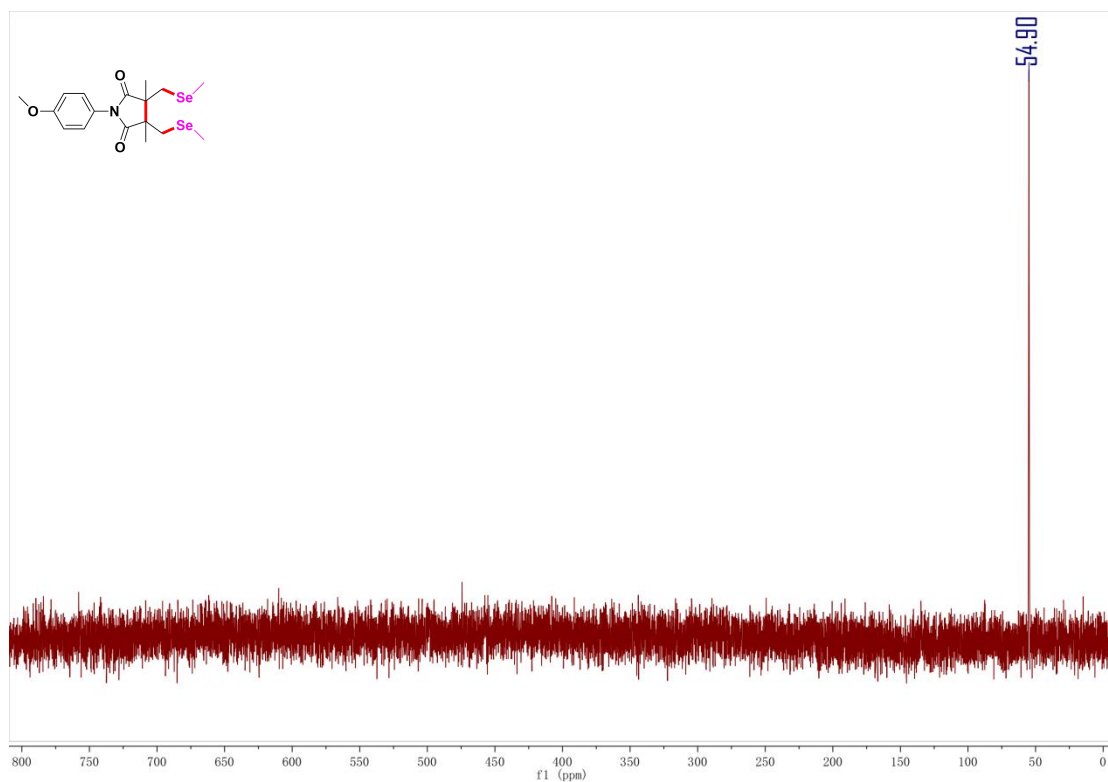
⁷⁷Se NMR Spectra of 6a (76 MHz, CDCl₃)

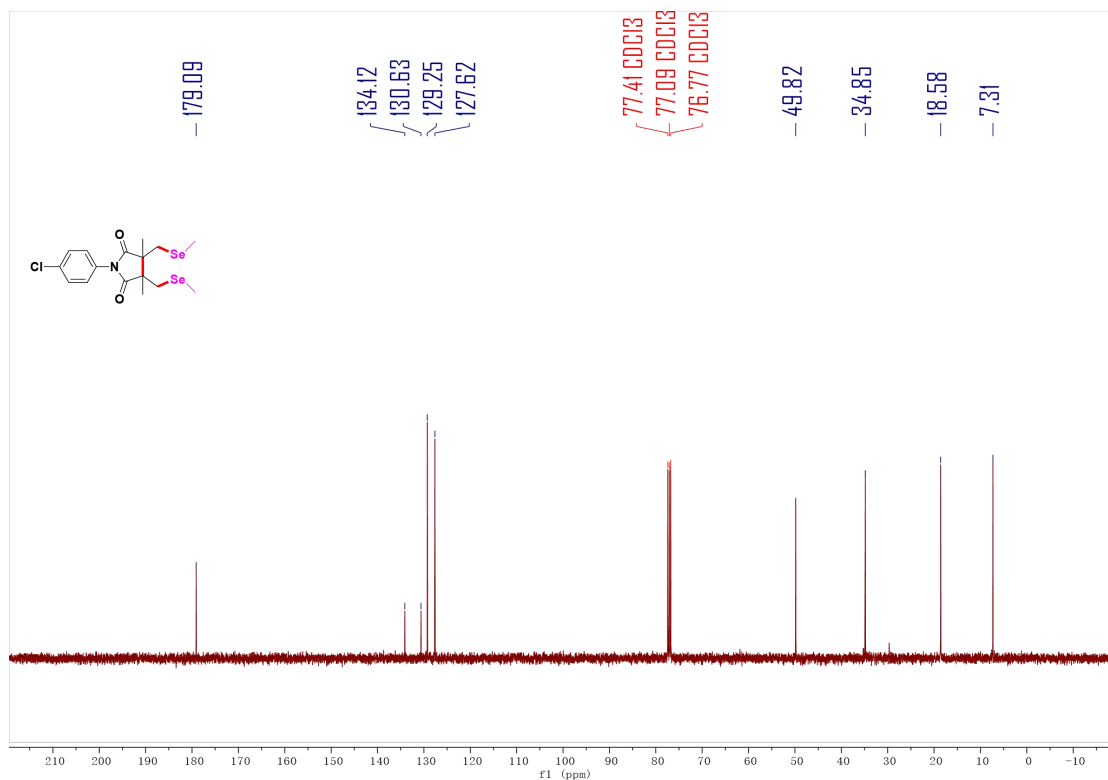


¹H NMR Spectra of 5b1 (400 MHz, CDCl₃)

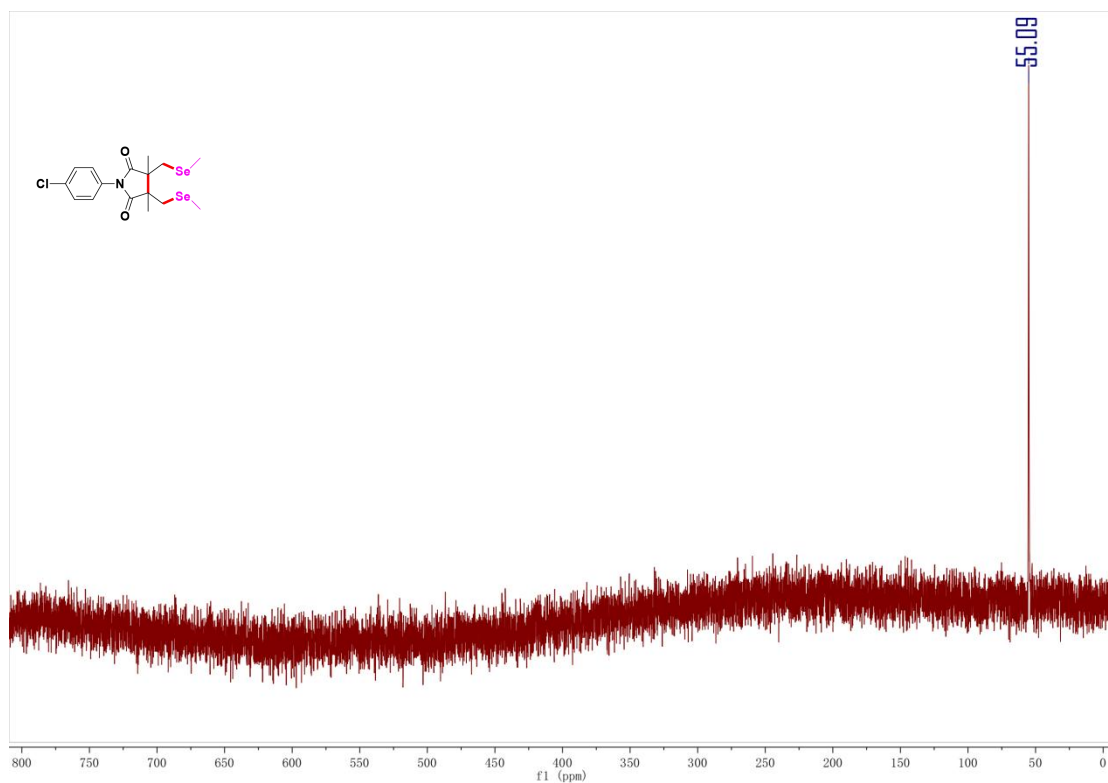


¹³C NMR Spectra of 5b1 (100 MHz, CDCl₃)

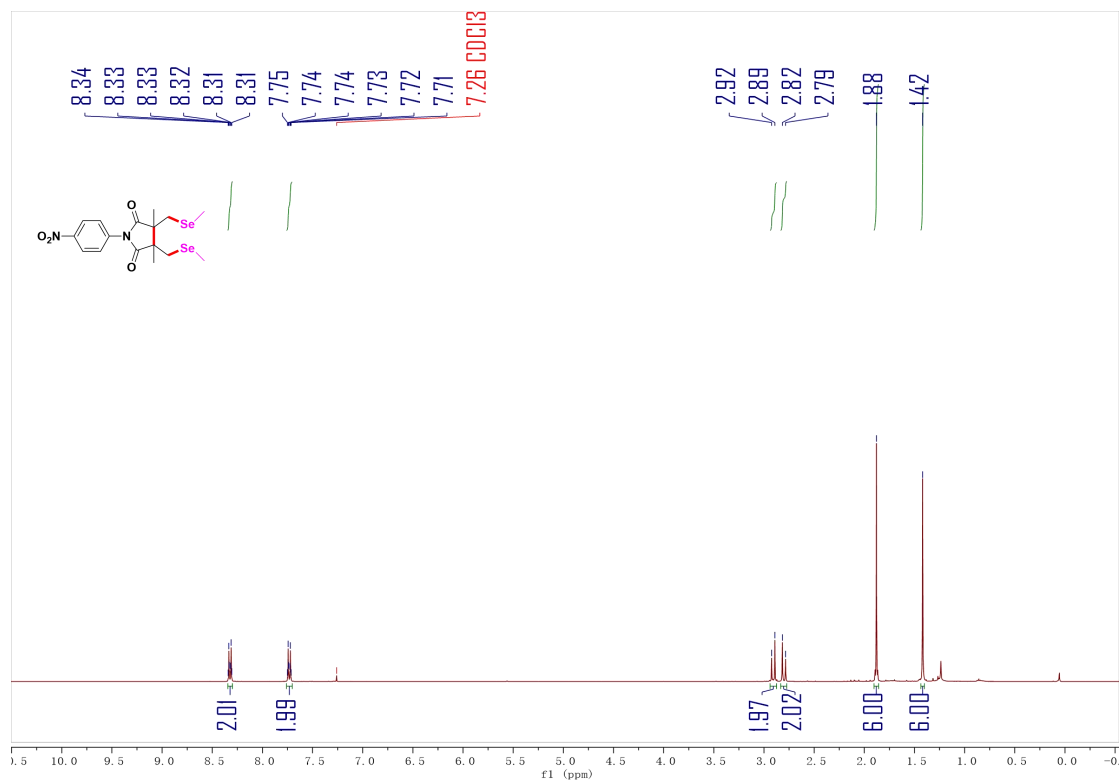




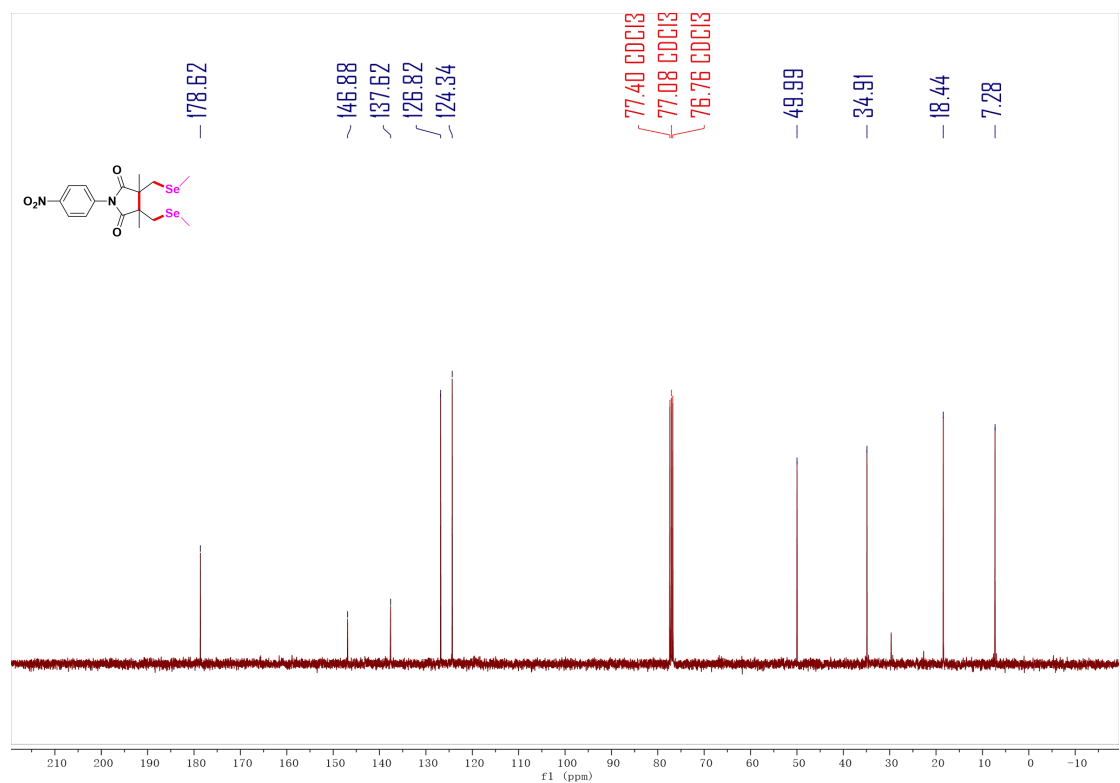
¹³C NMR Spectra of 5c1 (100 MHz, CDCl₃)



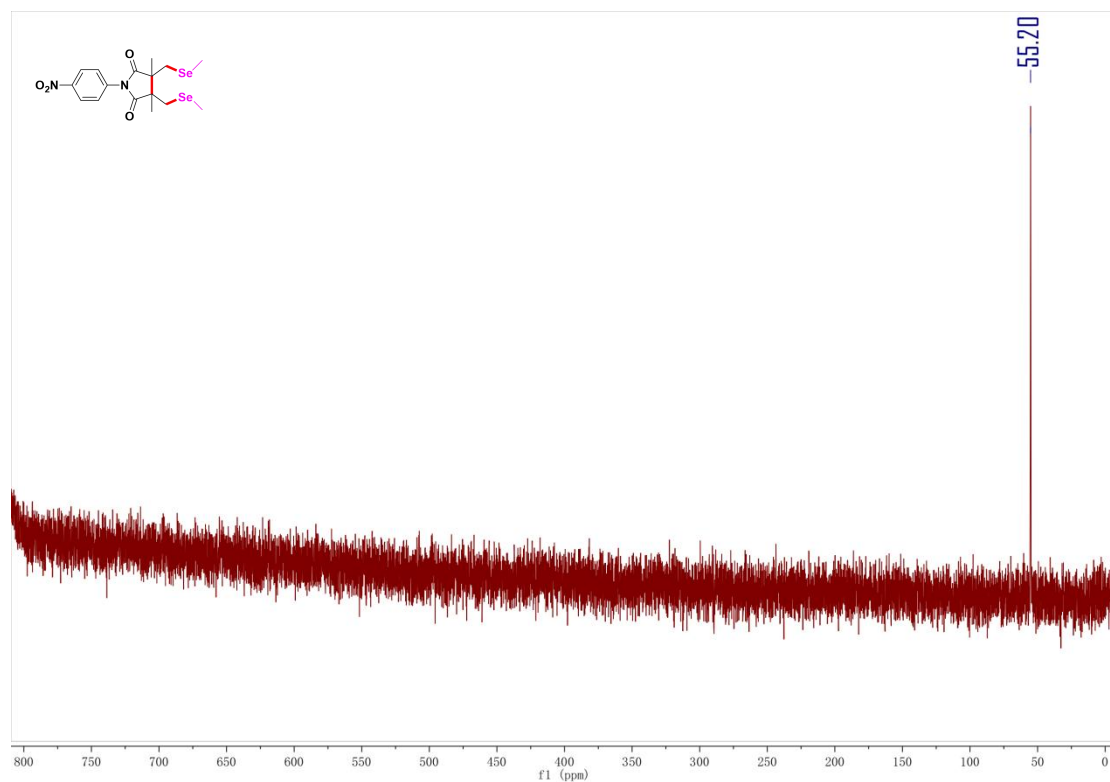
⁷⁷Se NMR Spectra of 5c1 (76 MHz, CDCl₃)



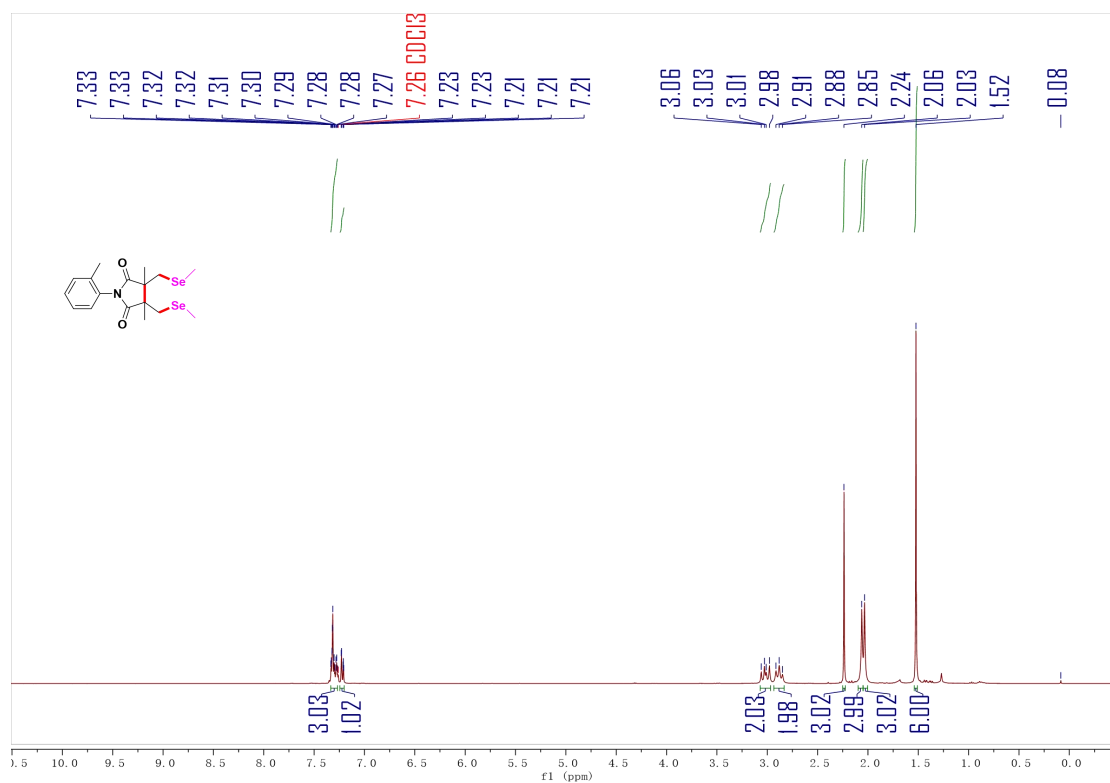
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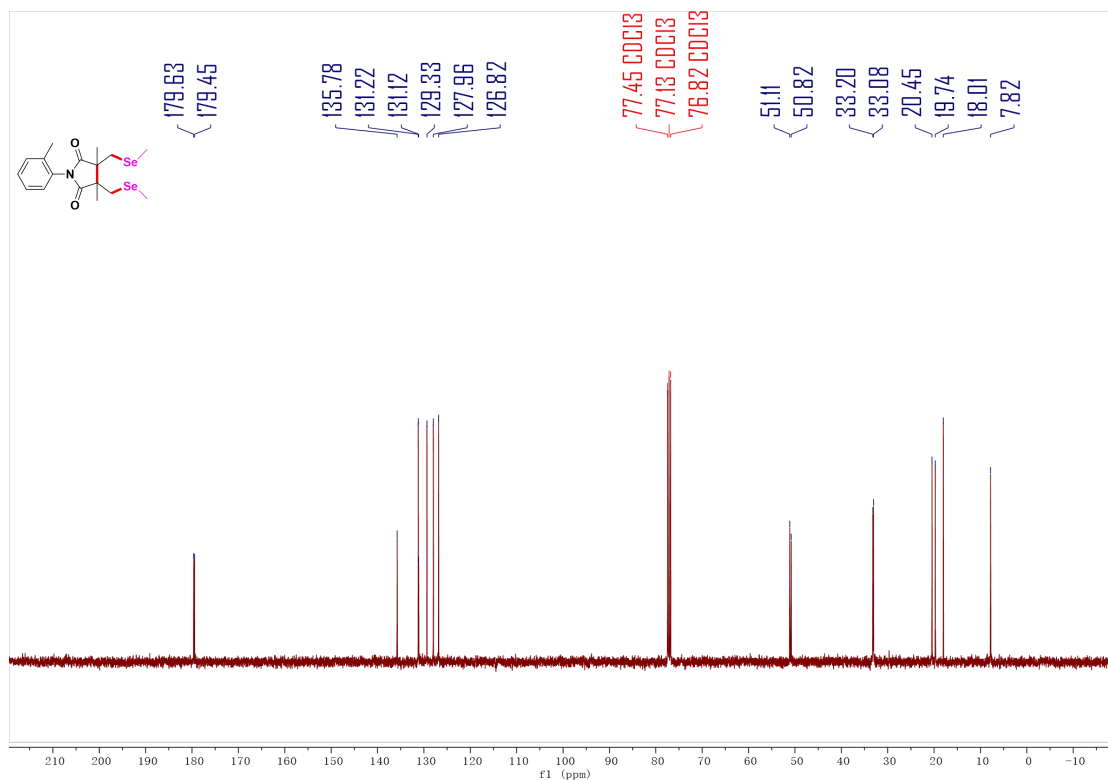
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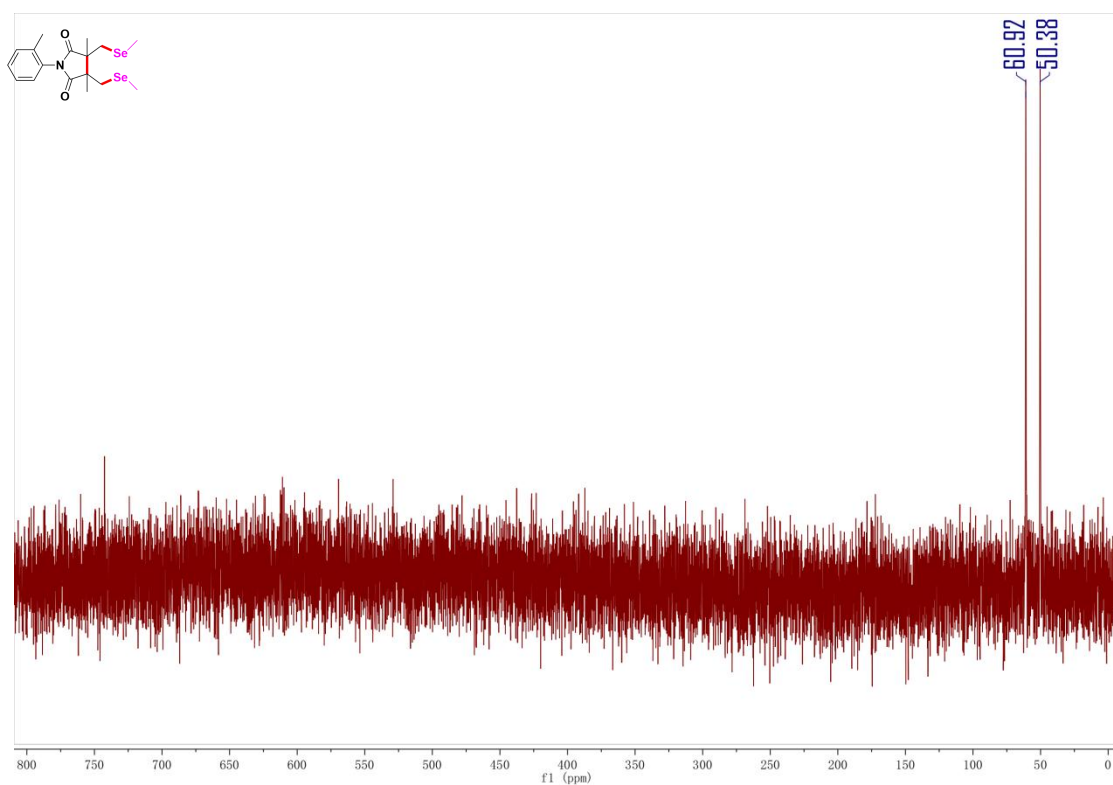
^{77}Se NMR Spectra of 5d1 (76 MHz, CDCl_3)



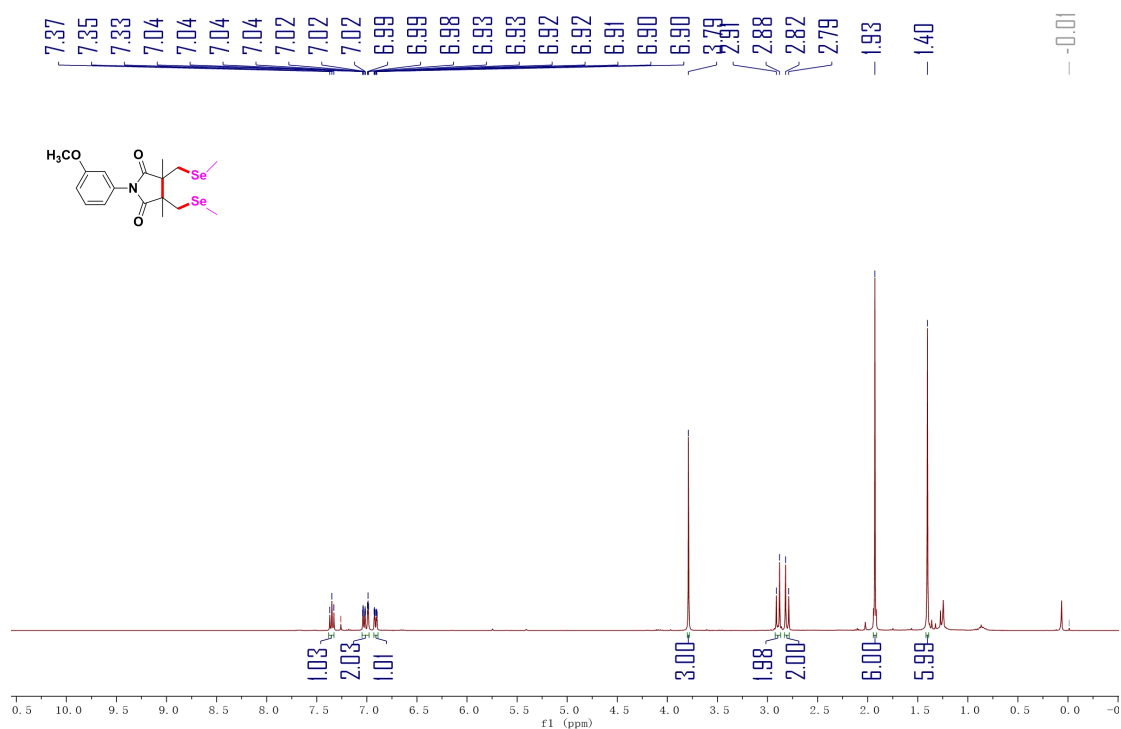
^1H NMR Spectra of 5e1 (400 MHz, CDCl_3)



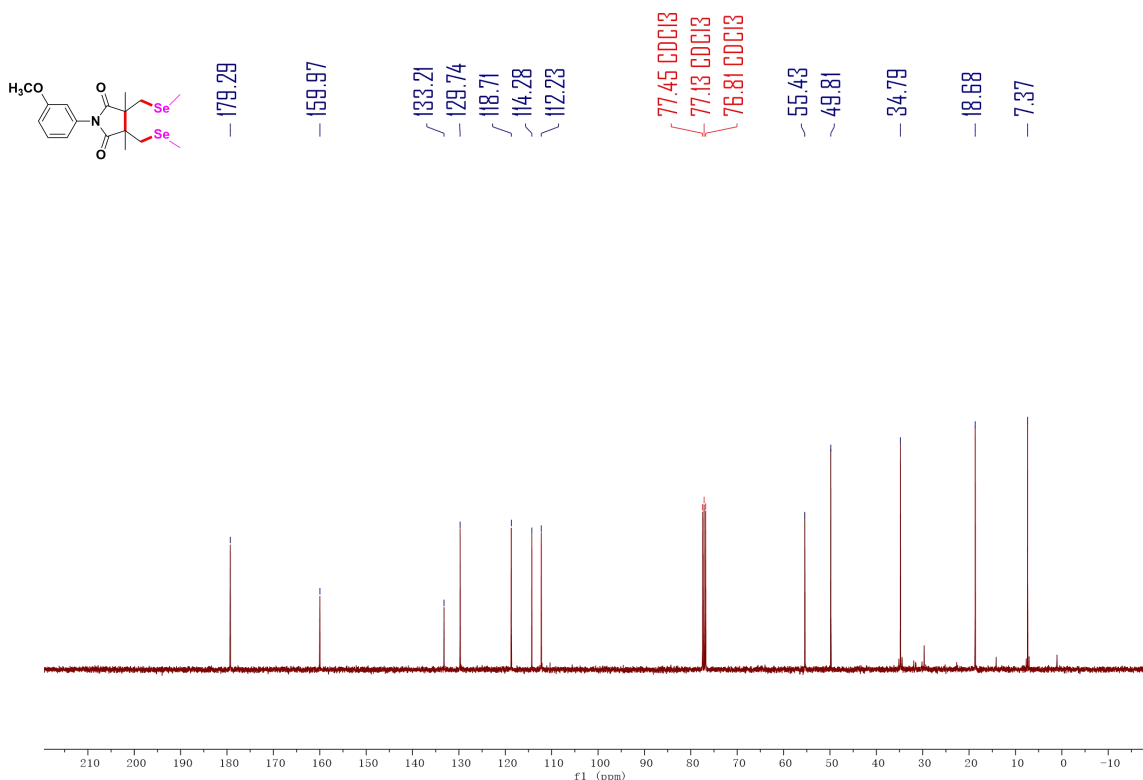
¹³C NMR Spectra of 5e1 (100 MHz, CDCl₃)



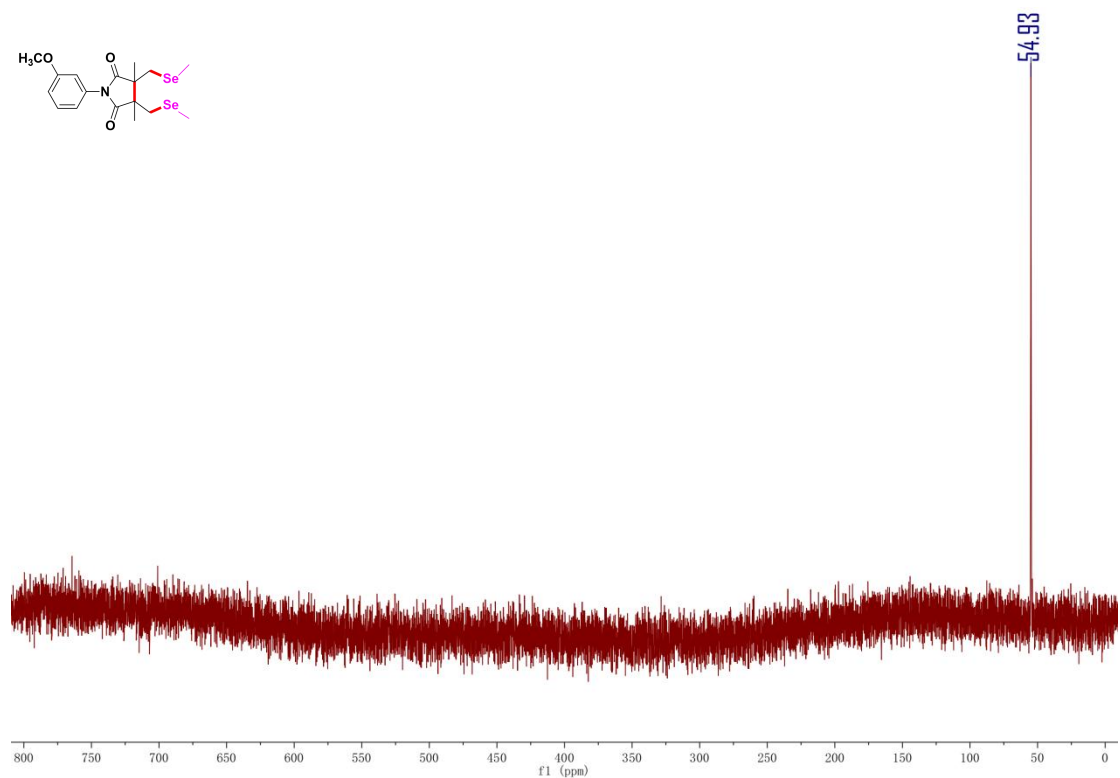
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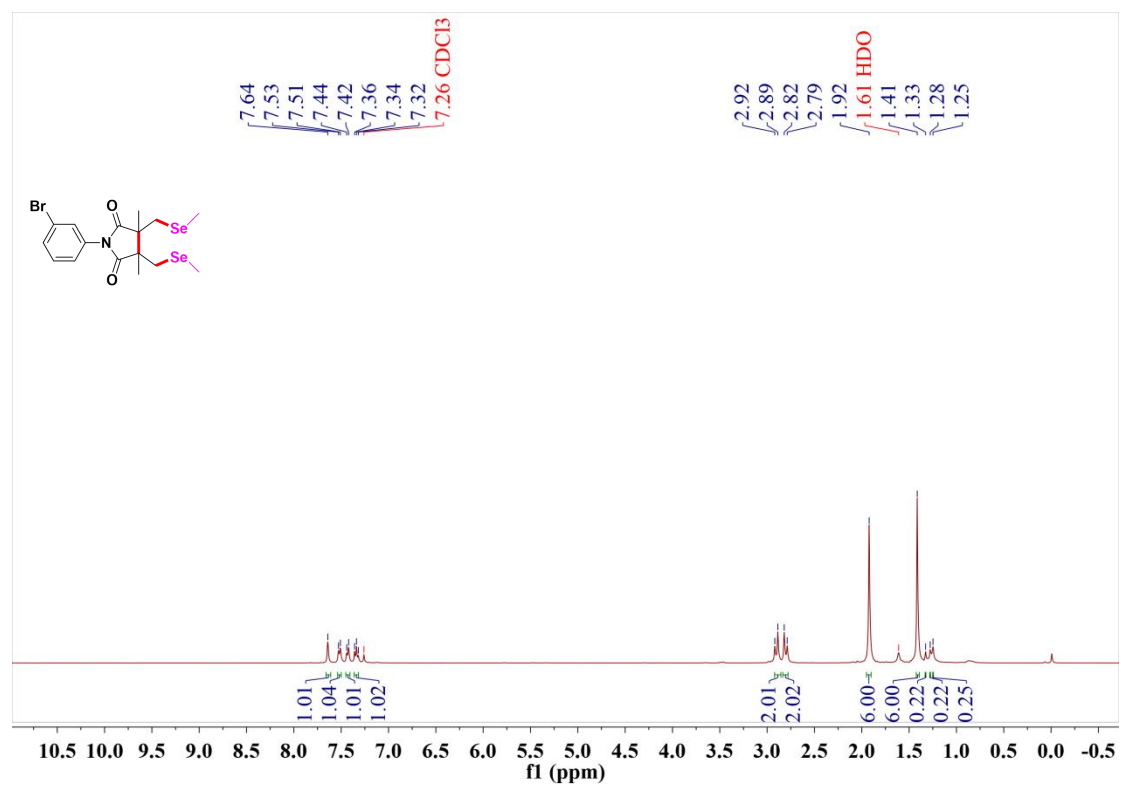
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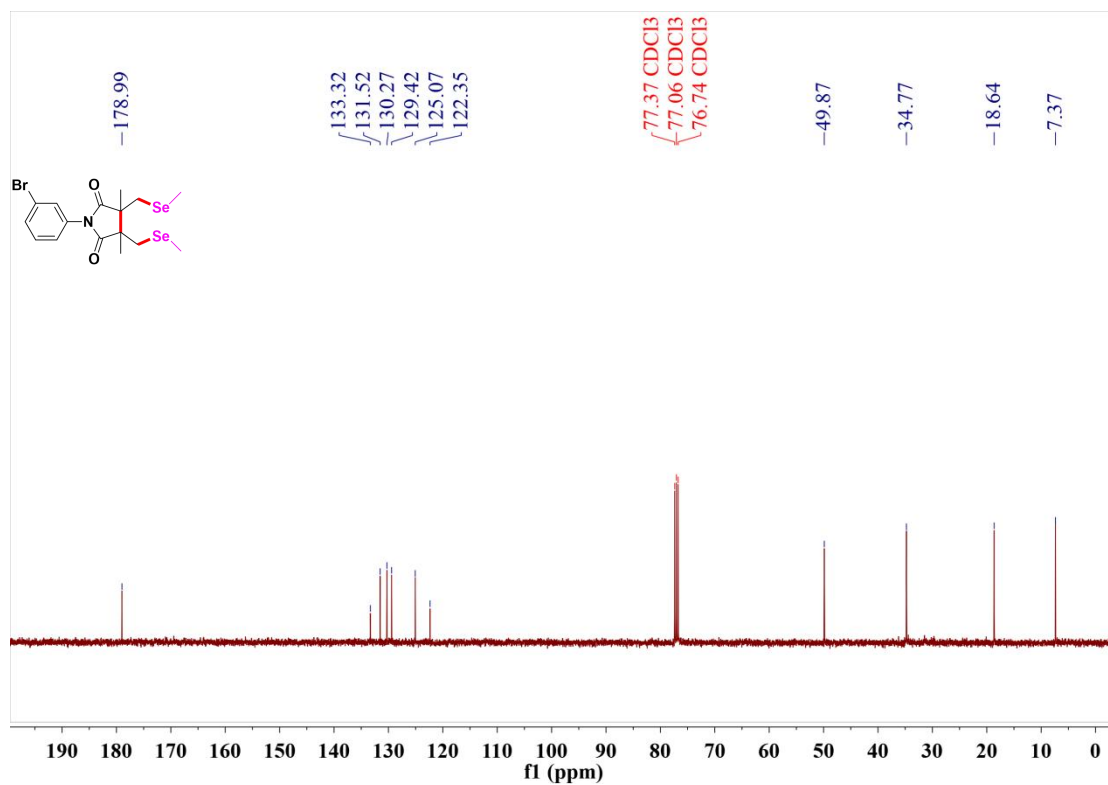
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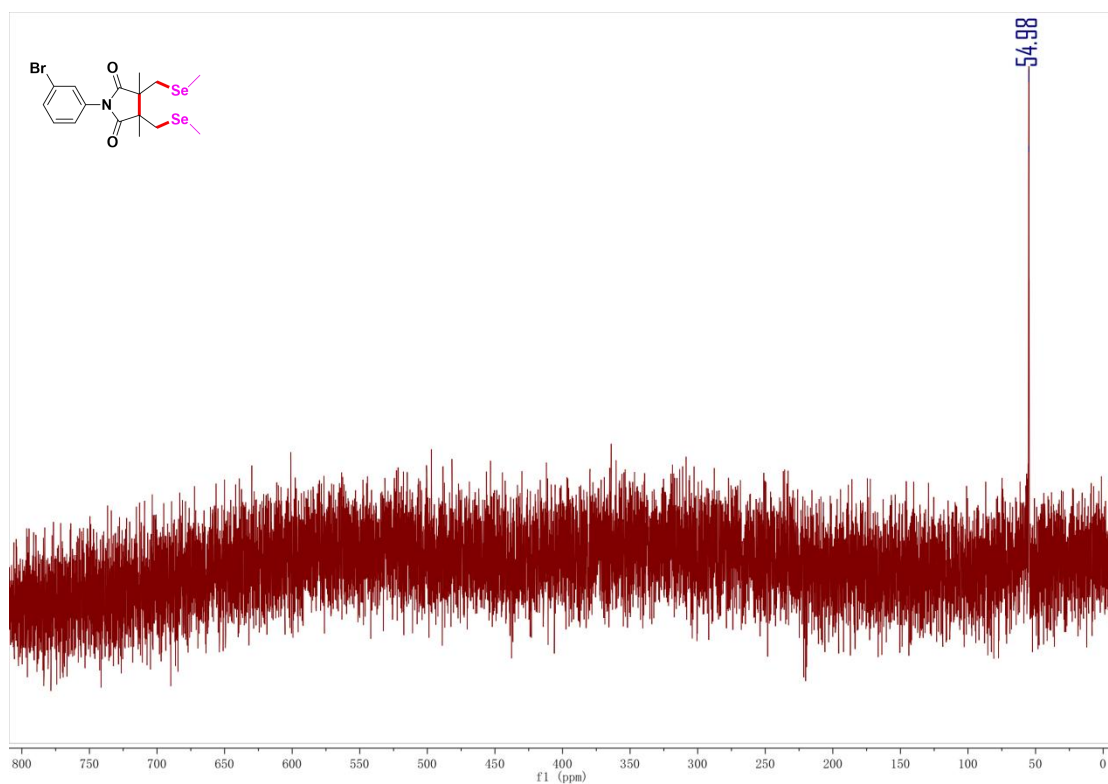
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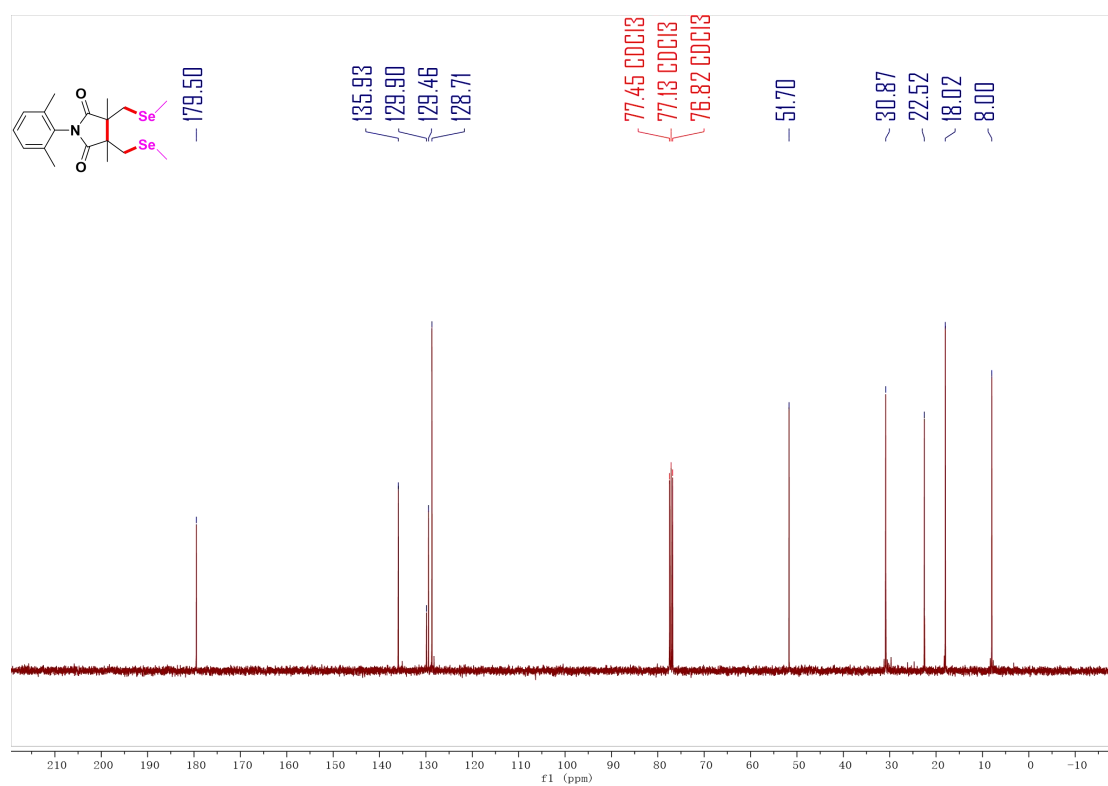
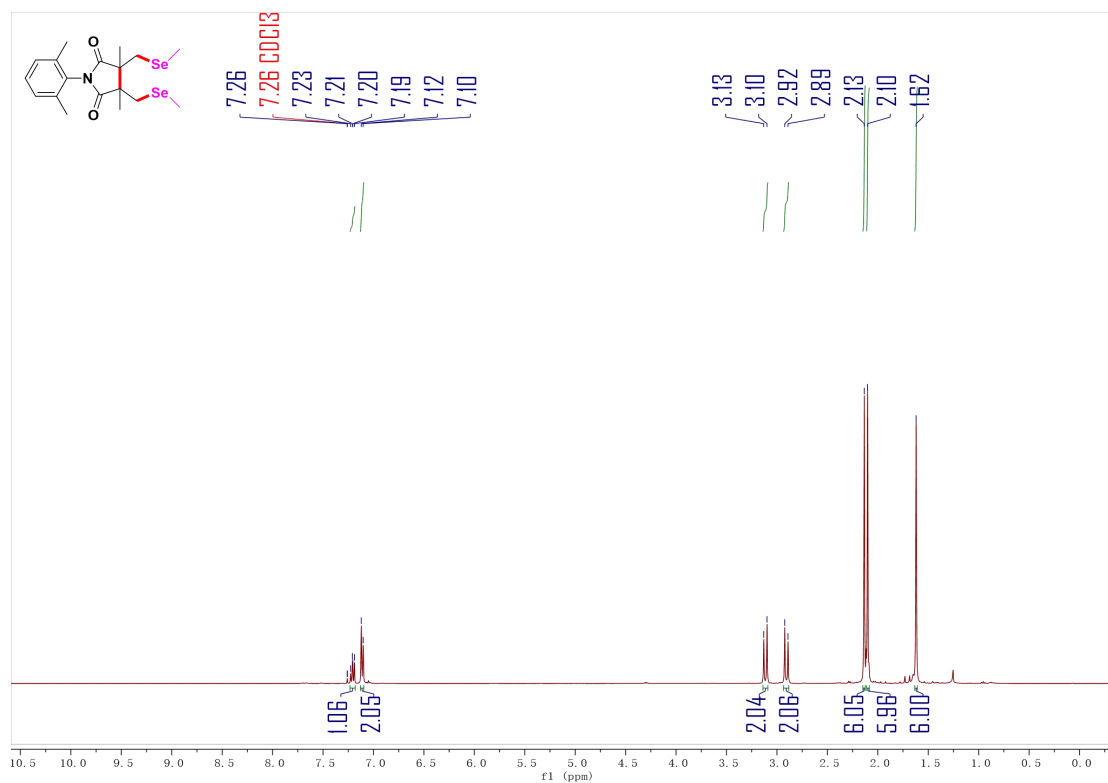
¹H NMR Spectra of 5g1 (400 MHz, CDCl₃)

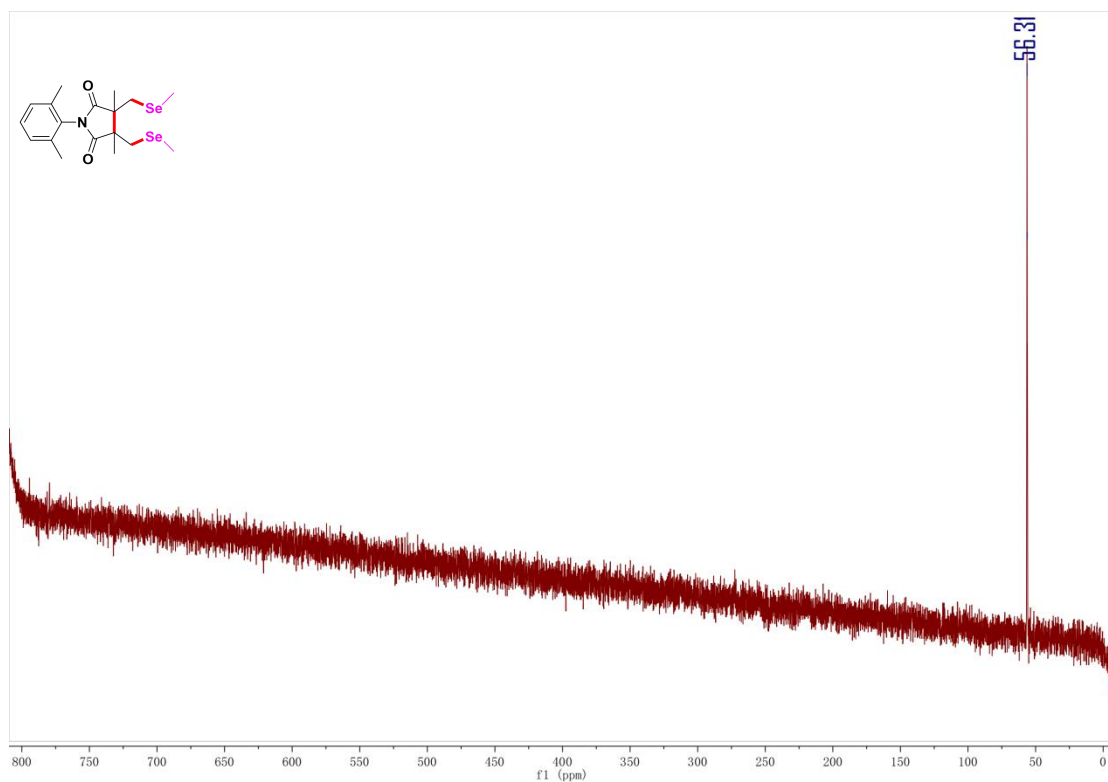


^{13}C NMR Spectra of 5g1 (100 MHz, CDCl₃)

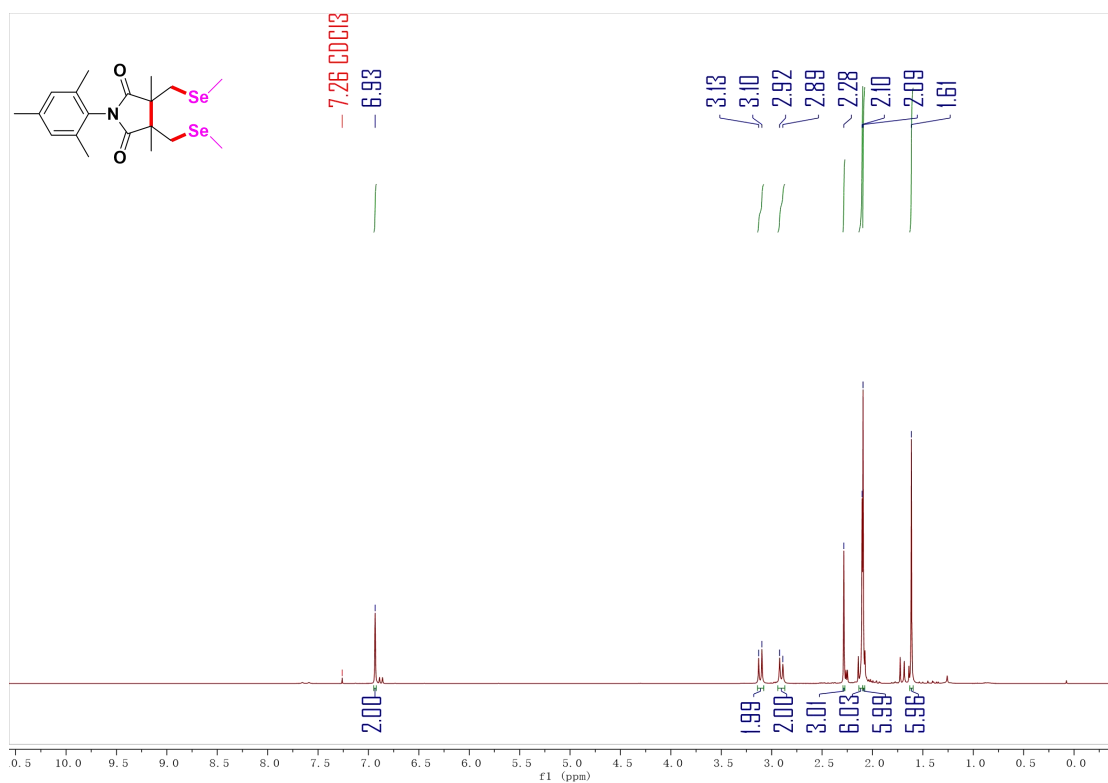


^{77}Se NMR Spectra of 5g1 (76 MHz, CDCl₃)

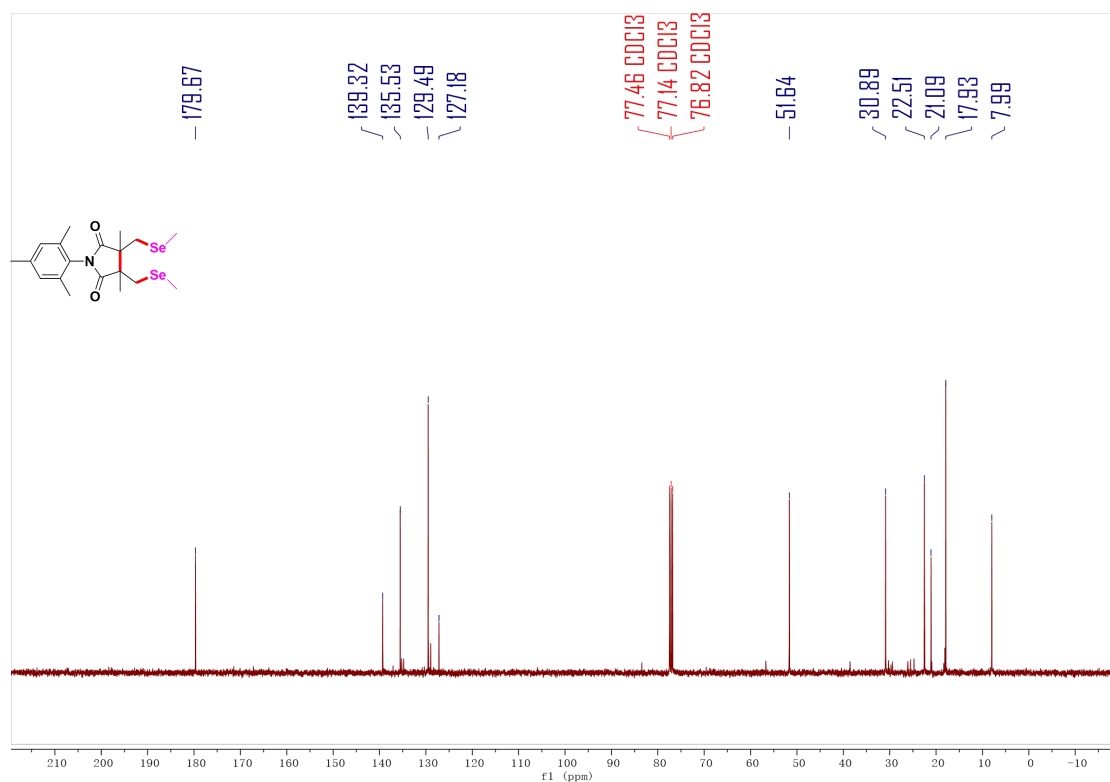




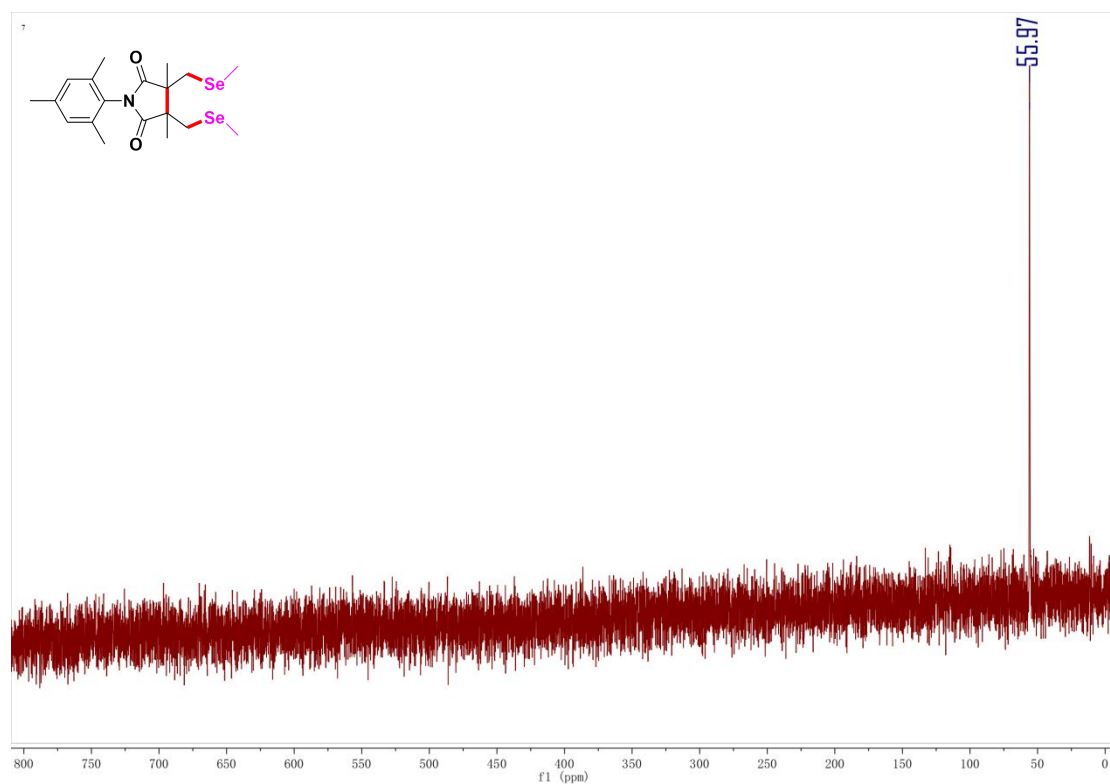
^{77}Se NMR Spectra of **5h1** (76 MHz, CDCl_3)



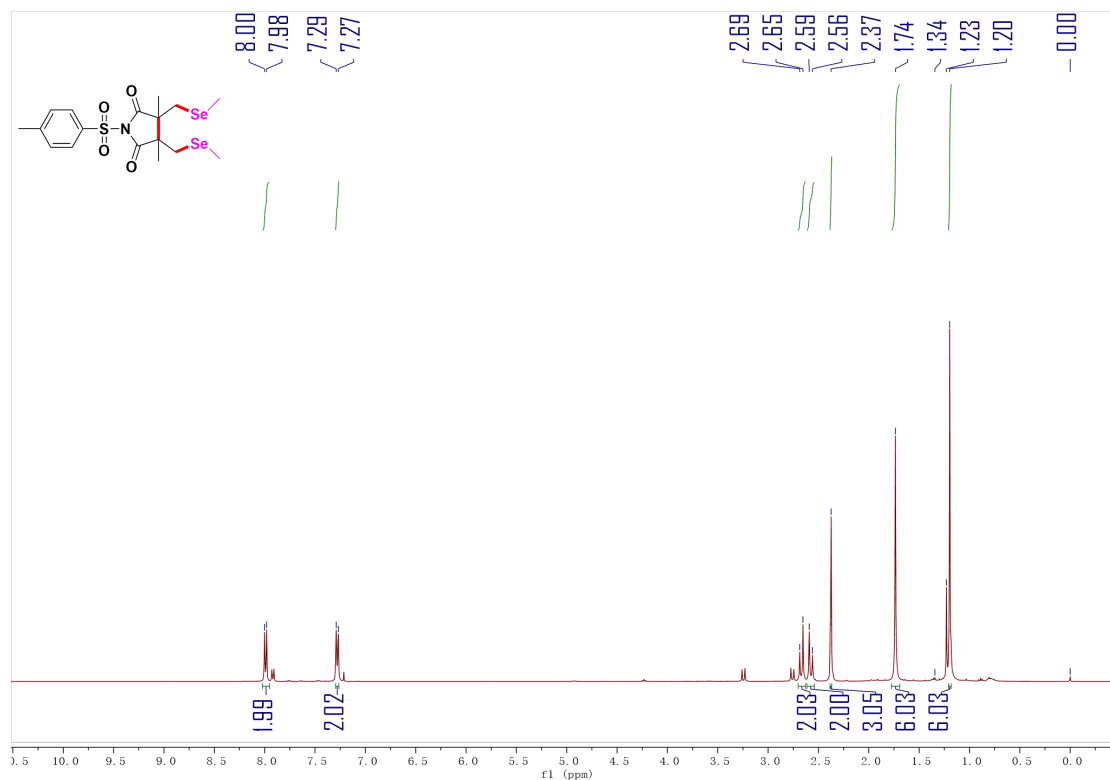
^1H NMR Spectra of **5i1** (400 MHz, CDCl_3)



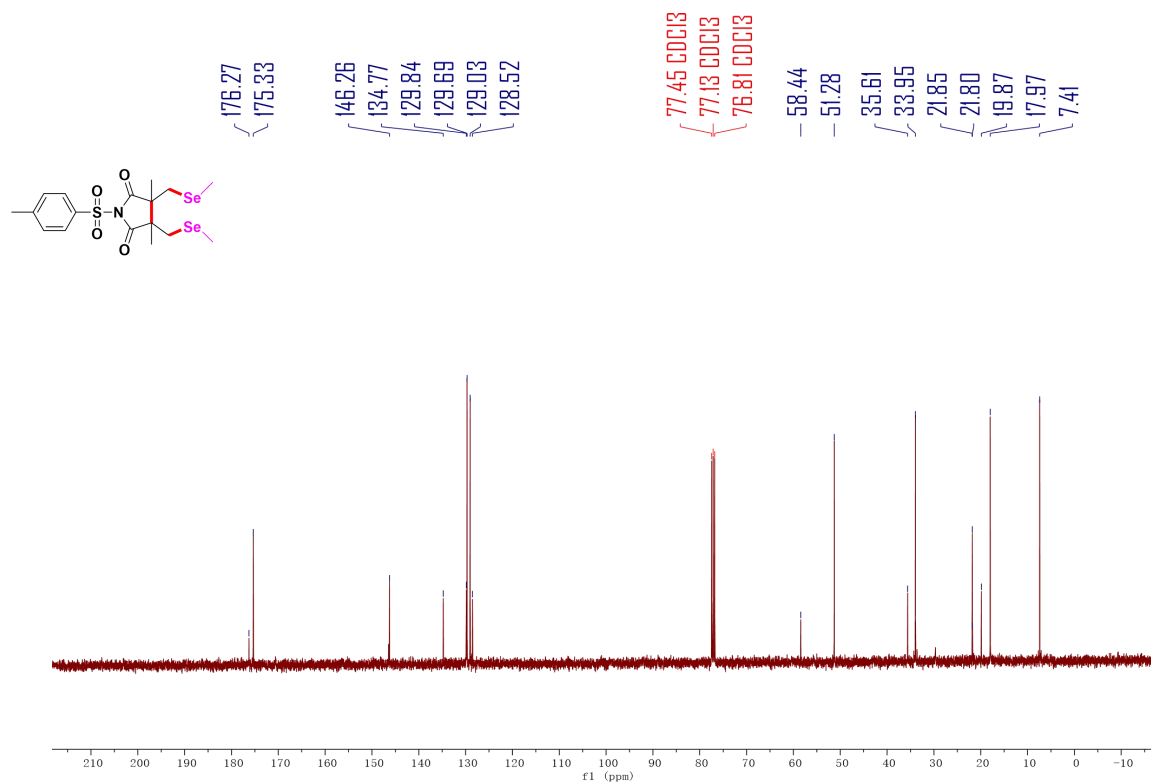
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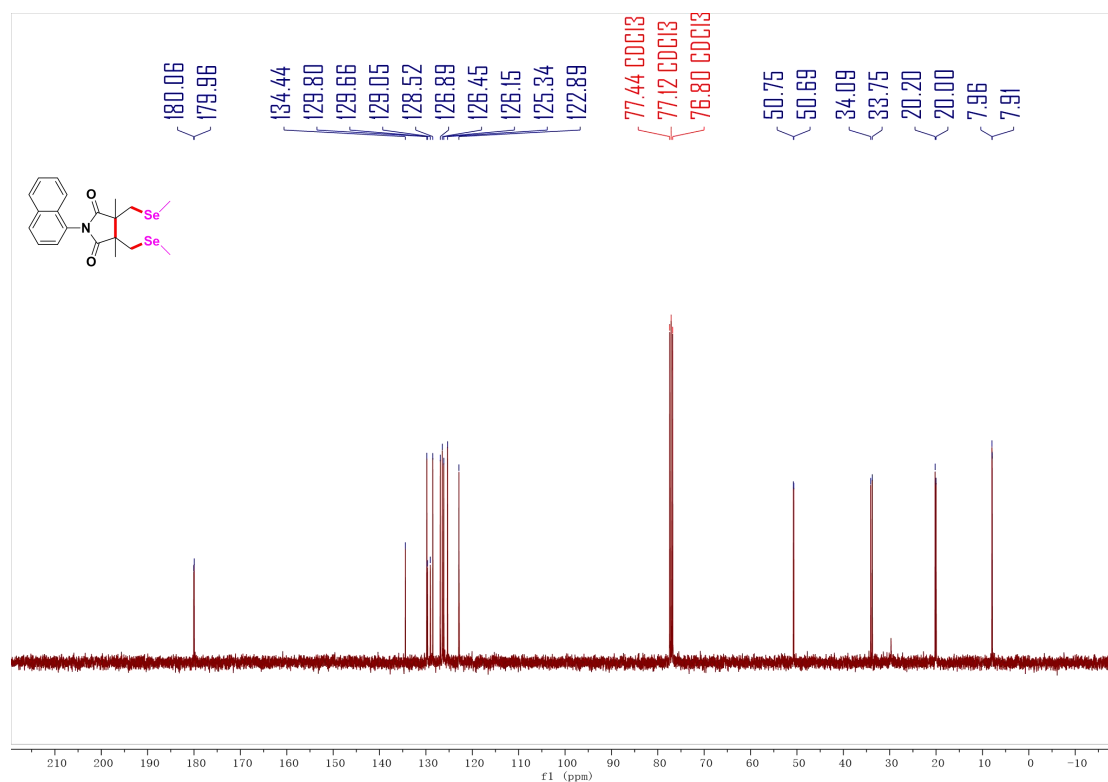
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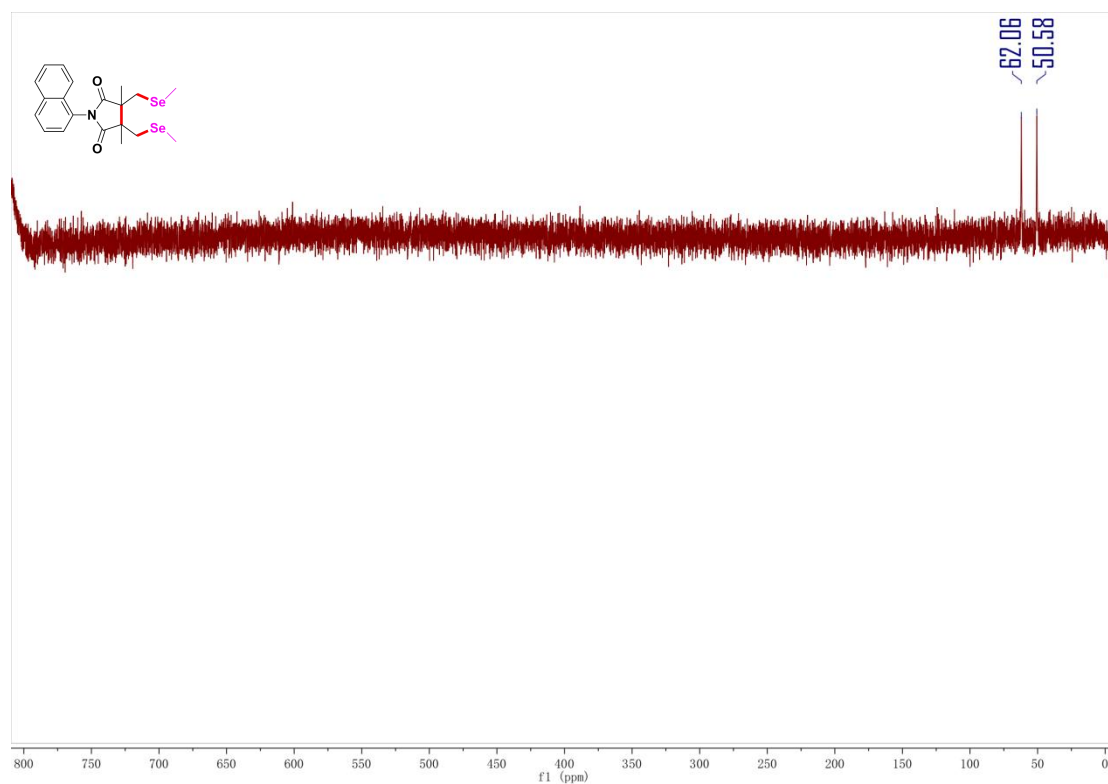
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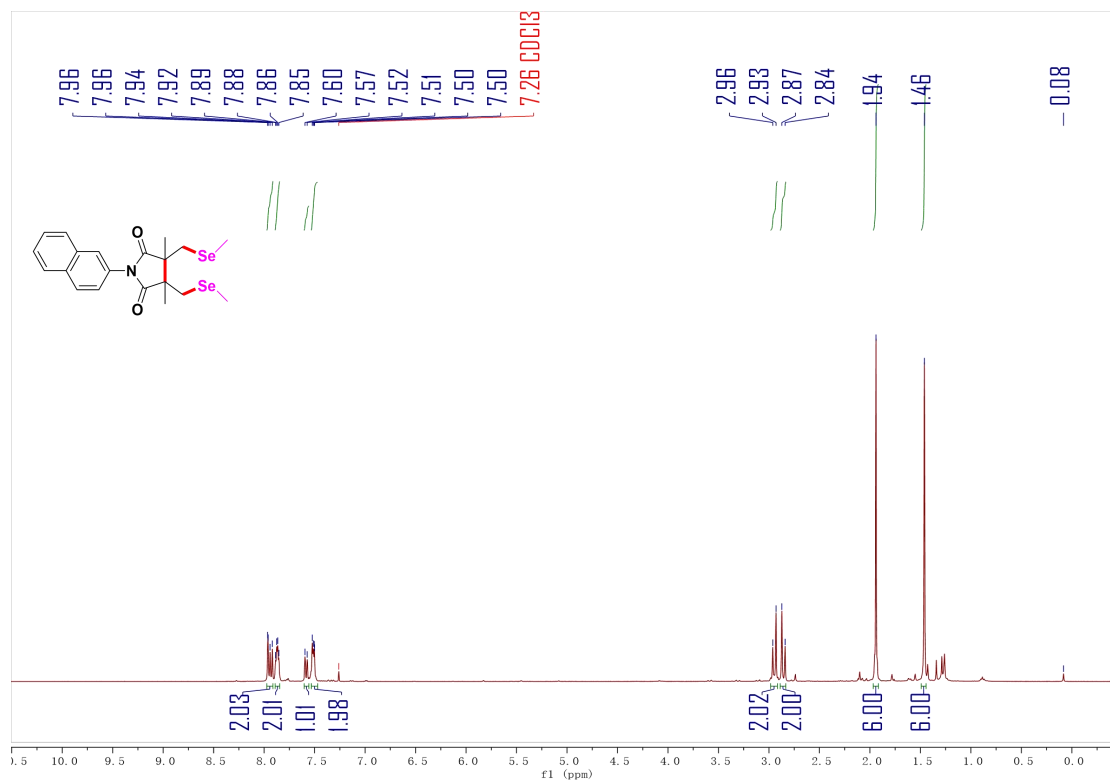
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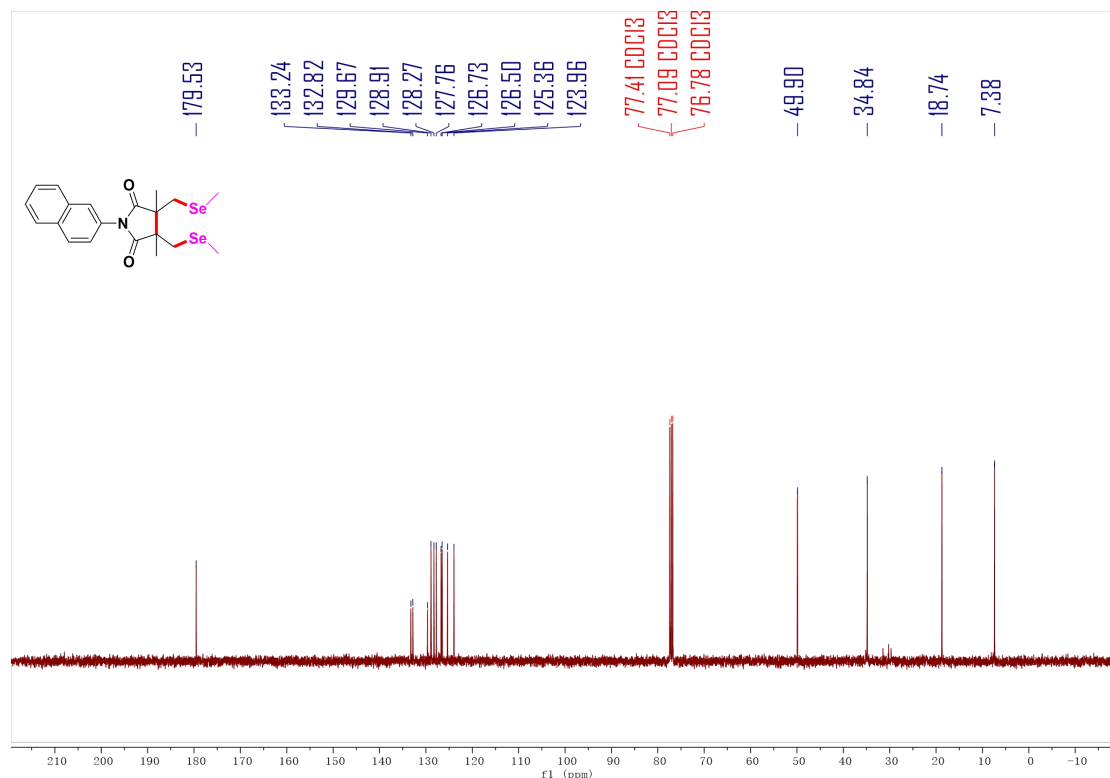
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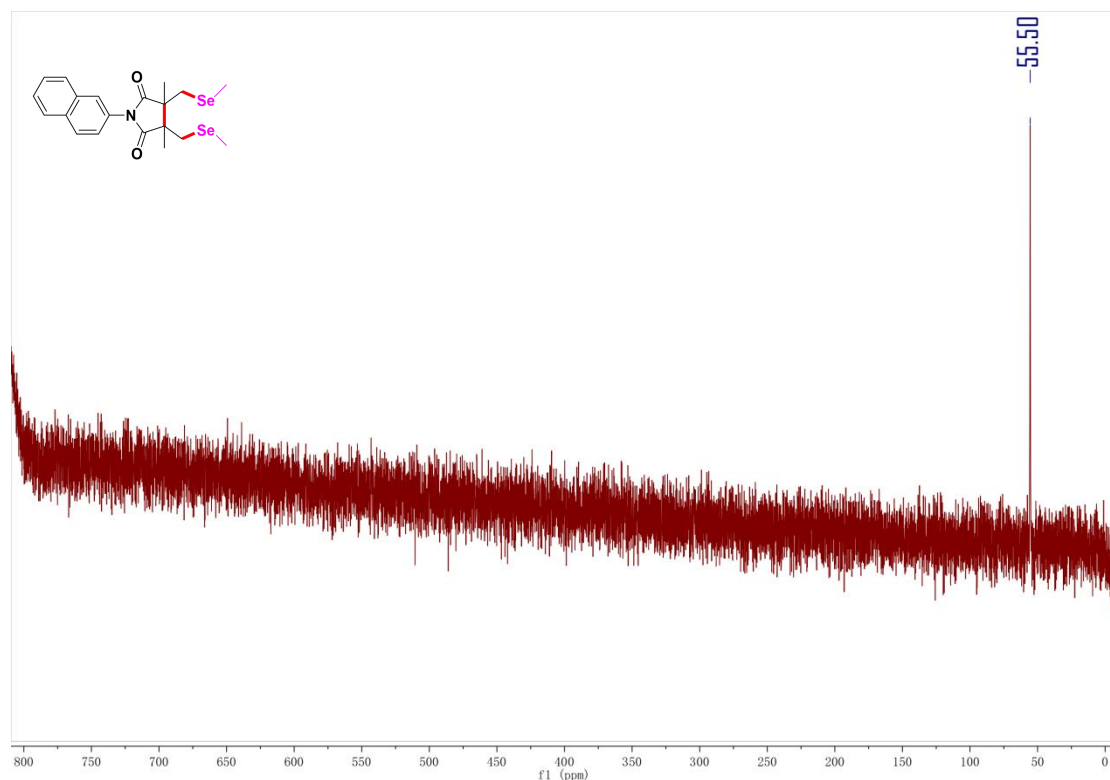
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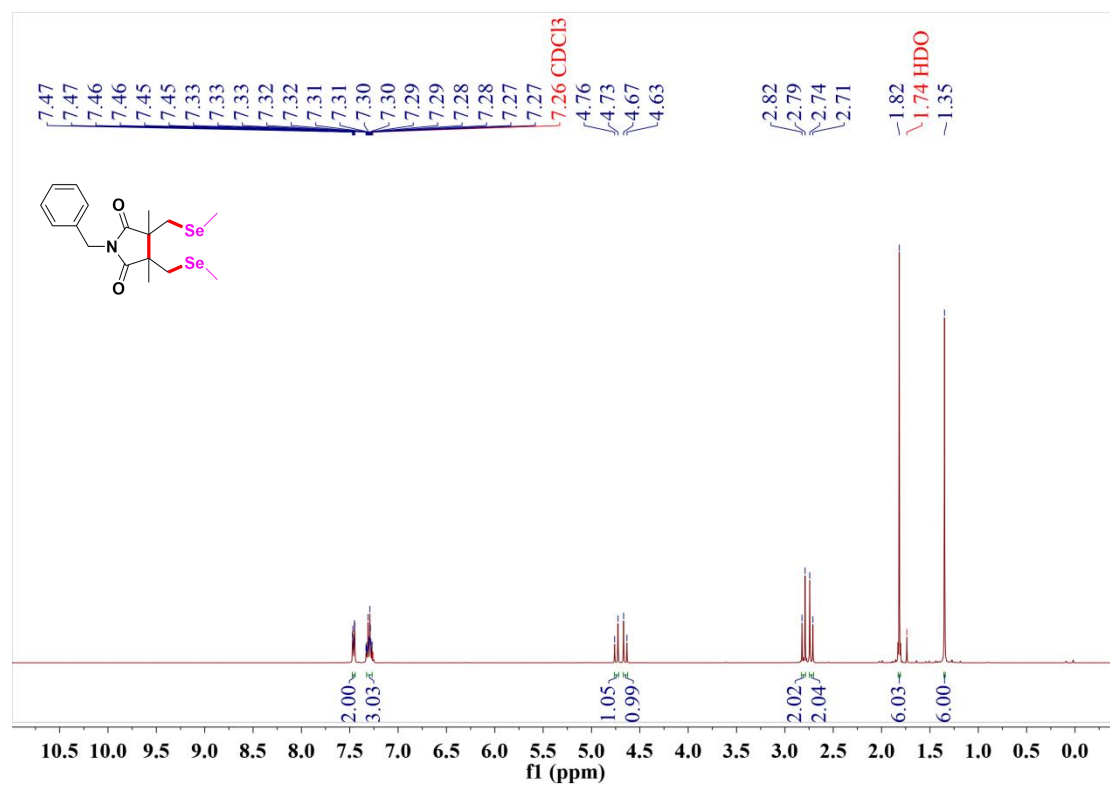
¹H NMR Spectra of 511 (400 MHz, CDCl₃)



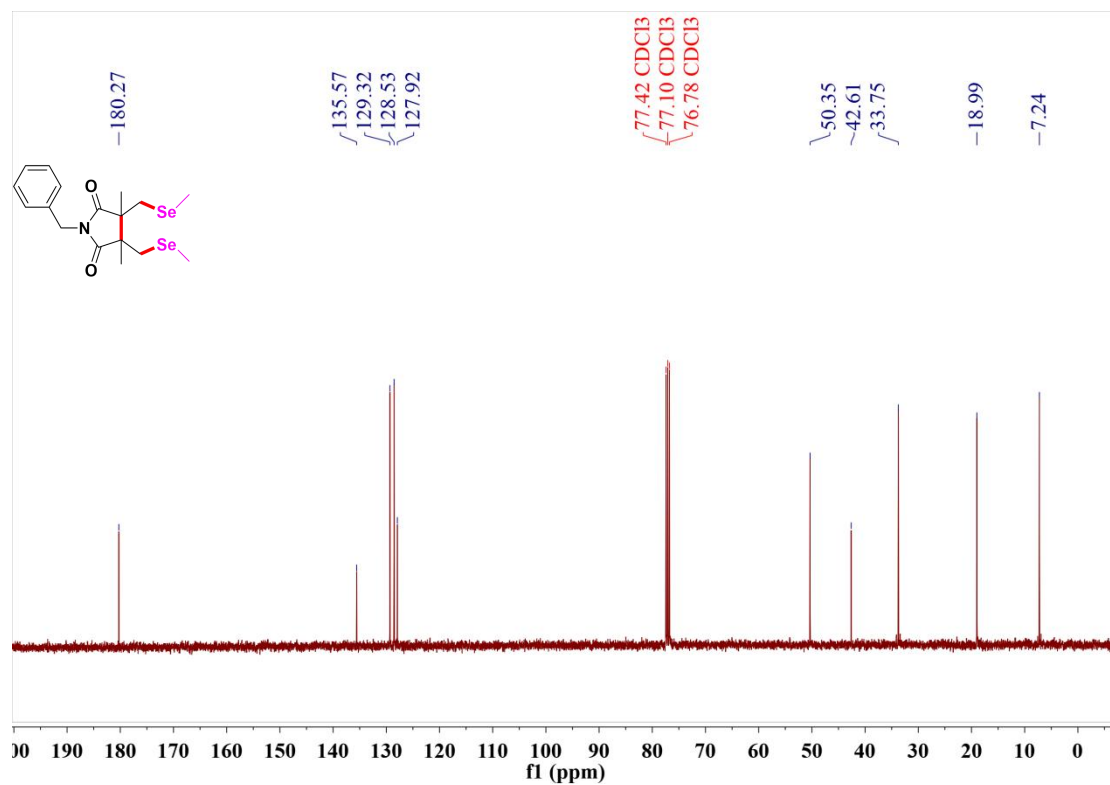
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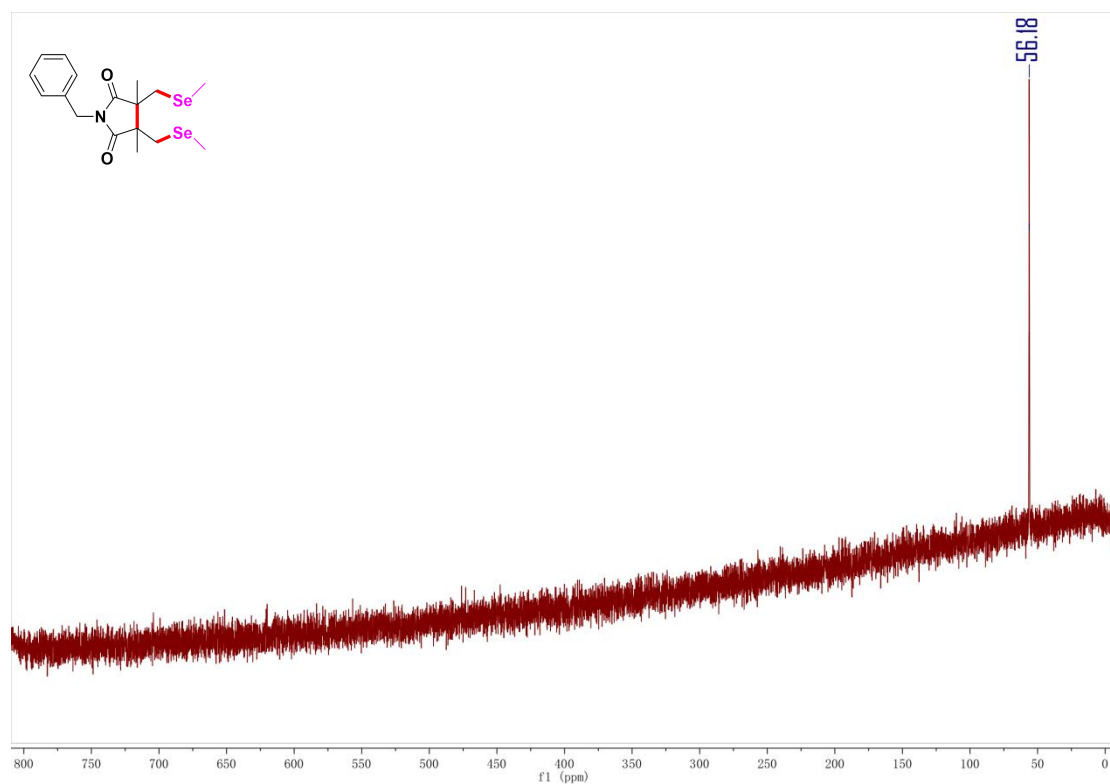
⁷⁷Se NMR Spectra of 51l (76 MHz, CDCl₃)



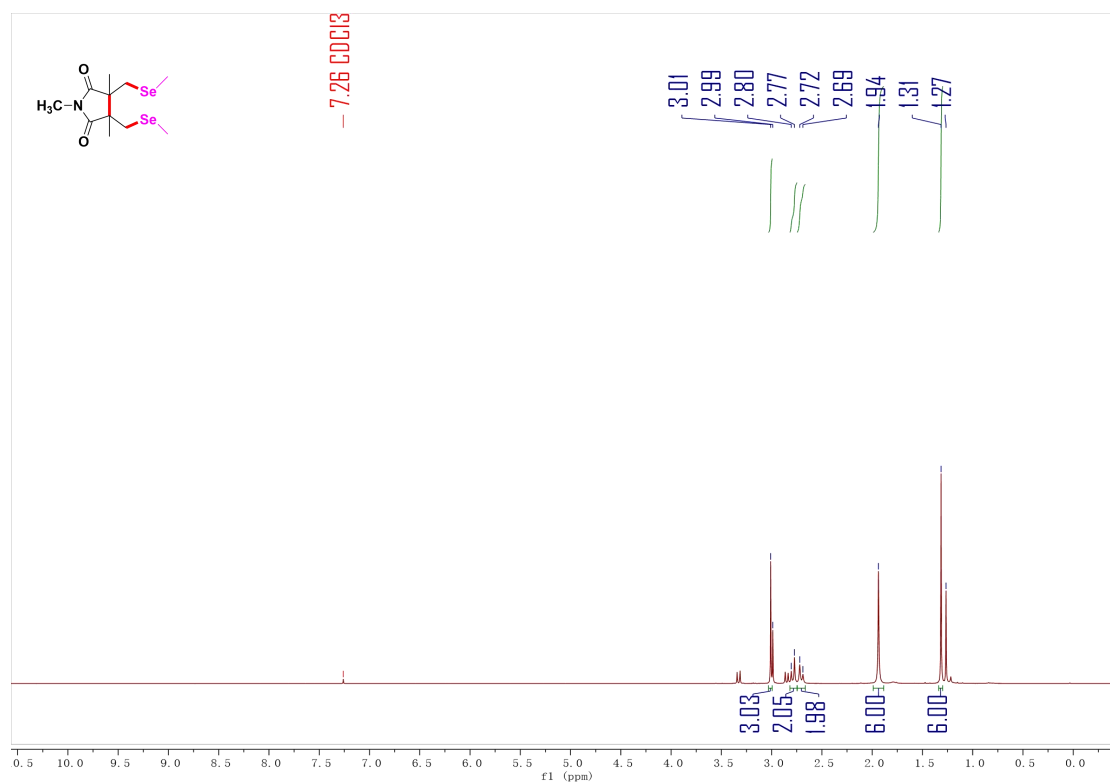
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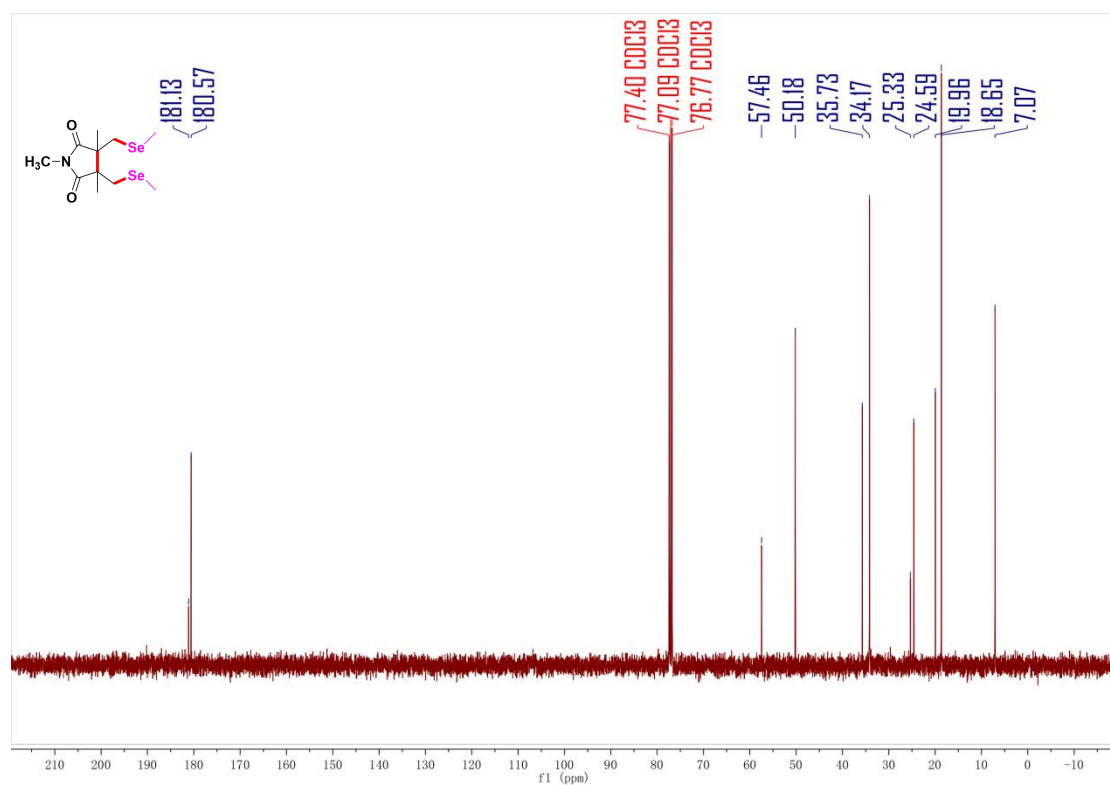
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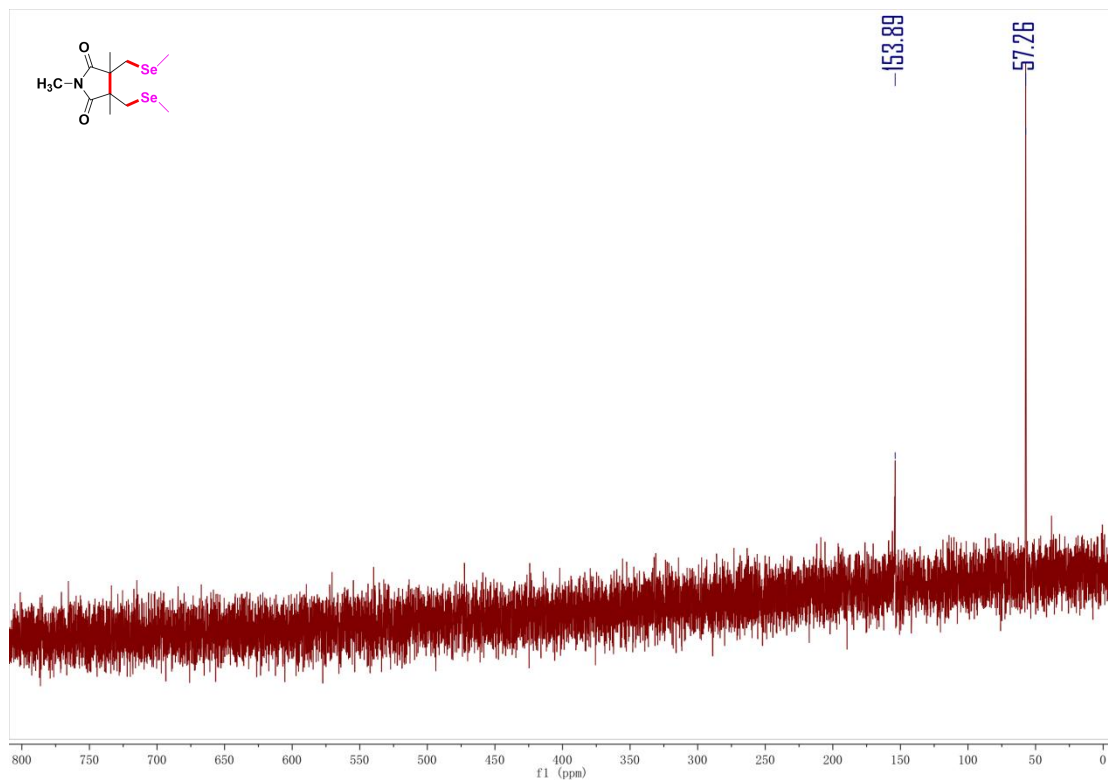
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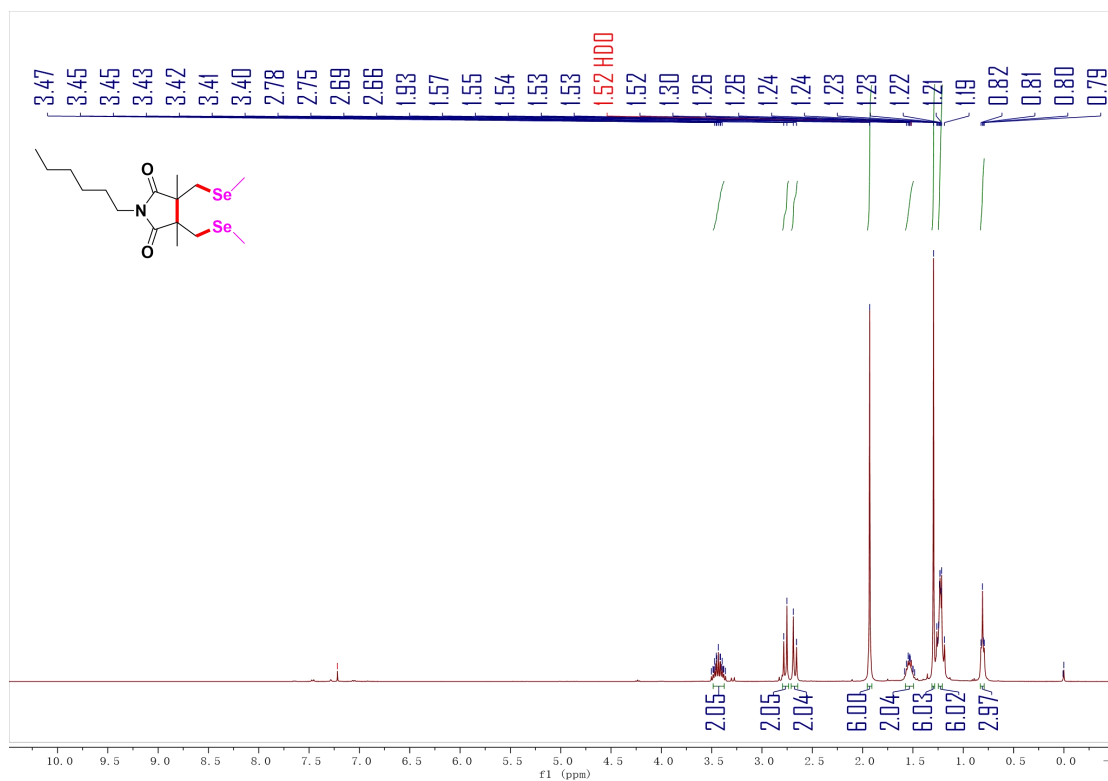
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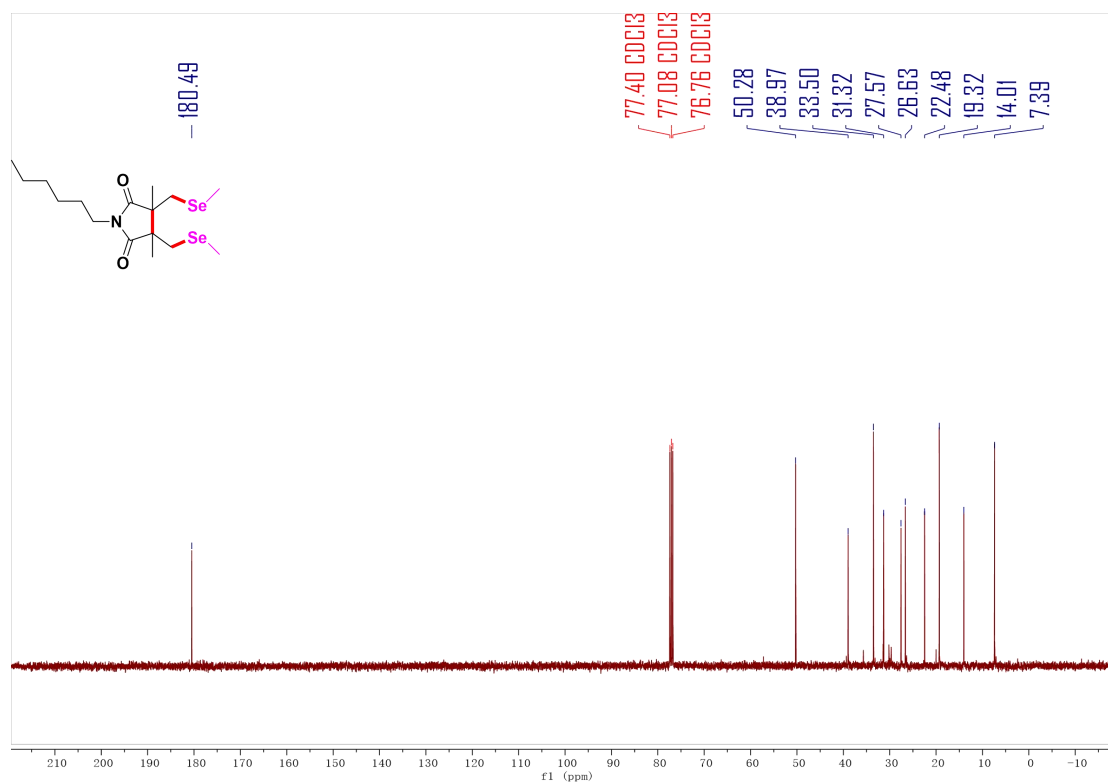
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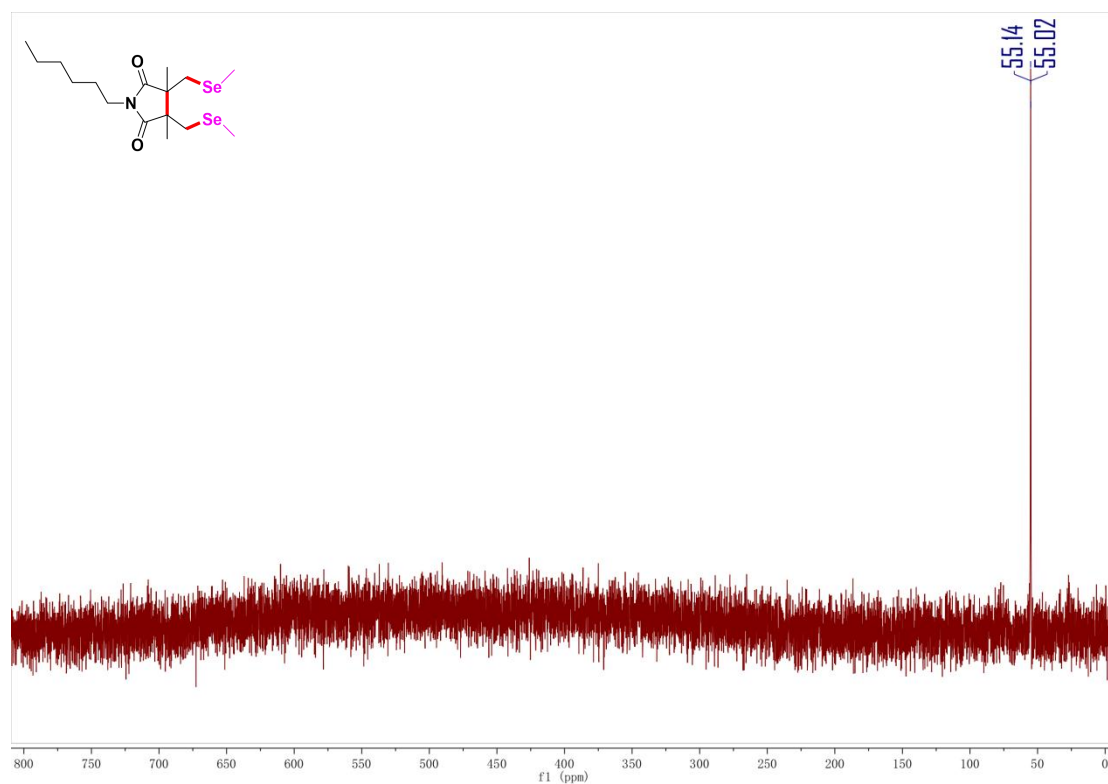
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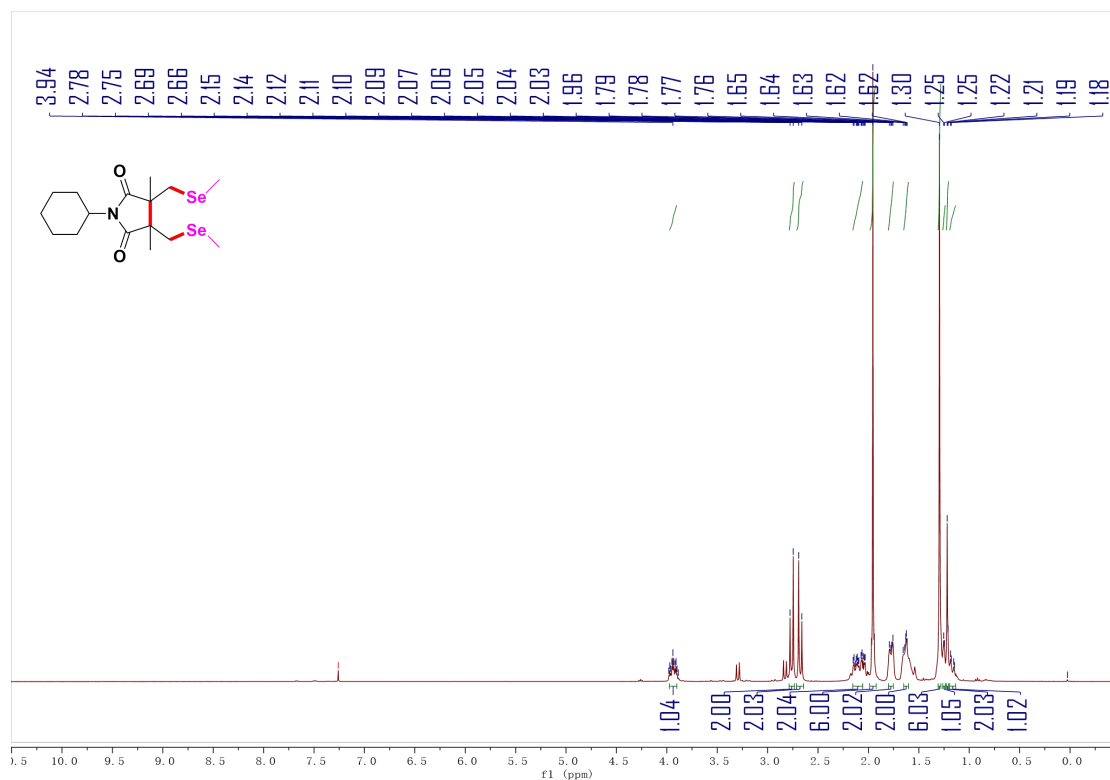
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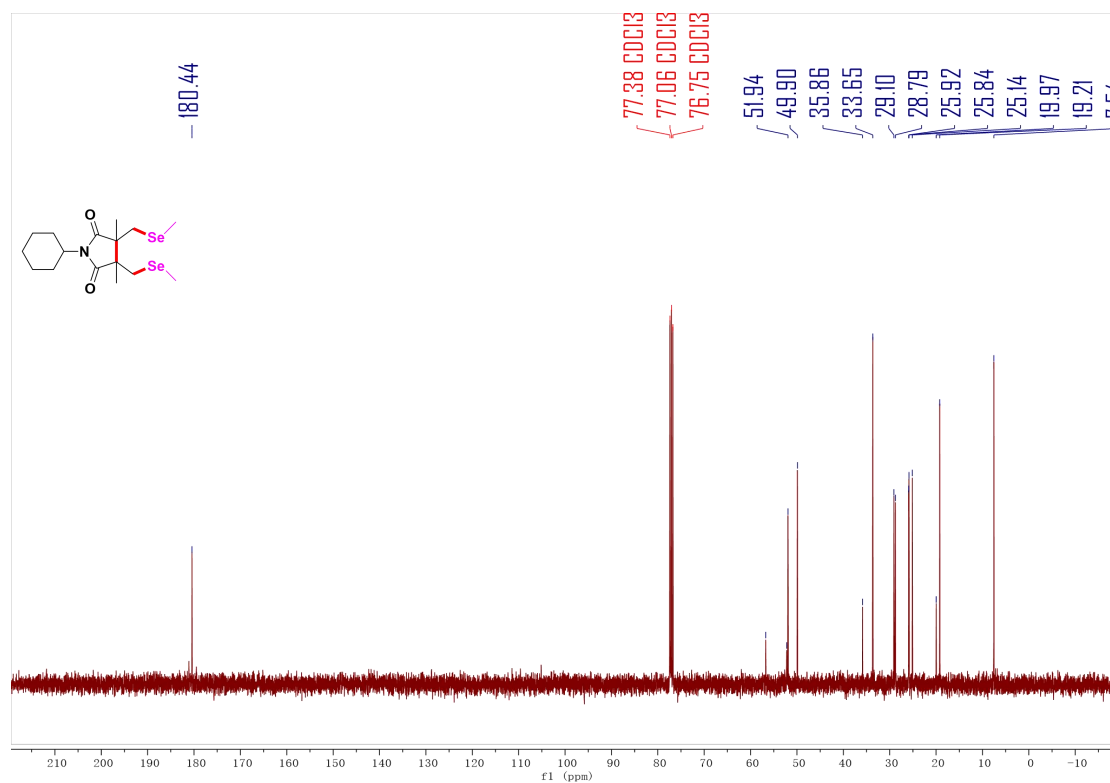
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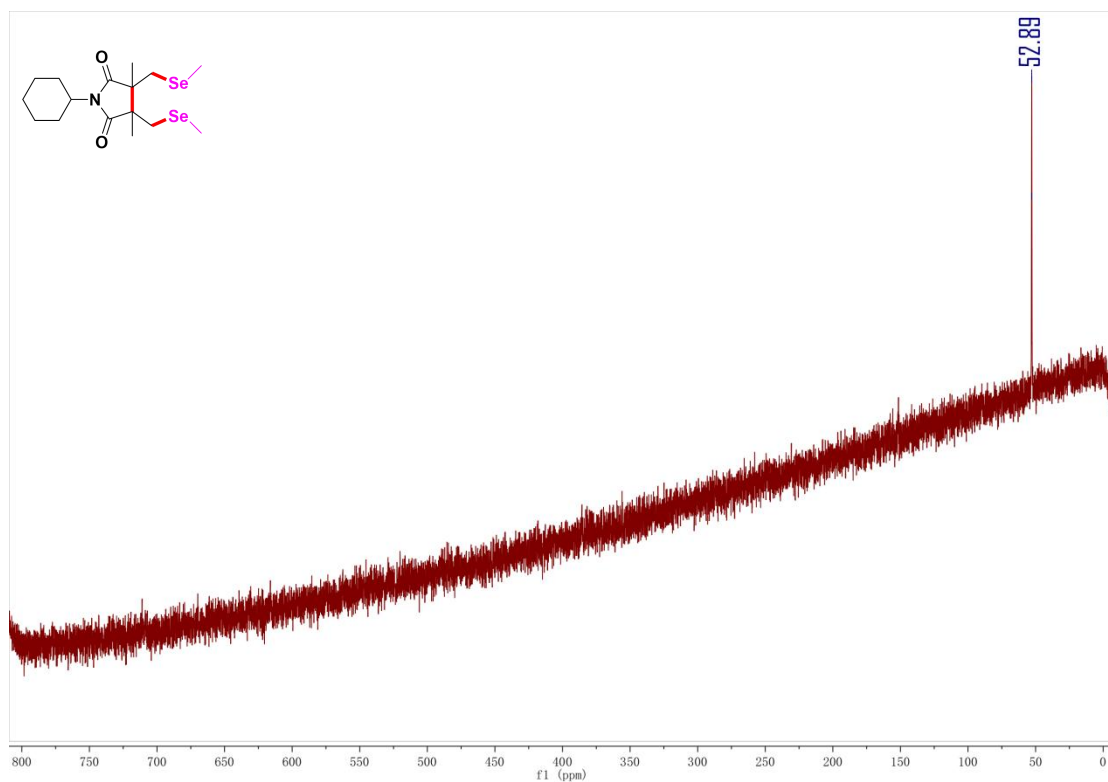
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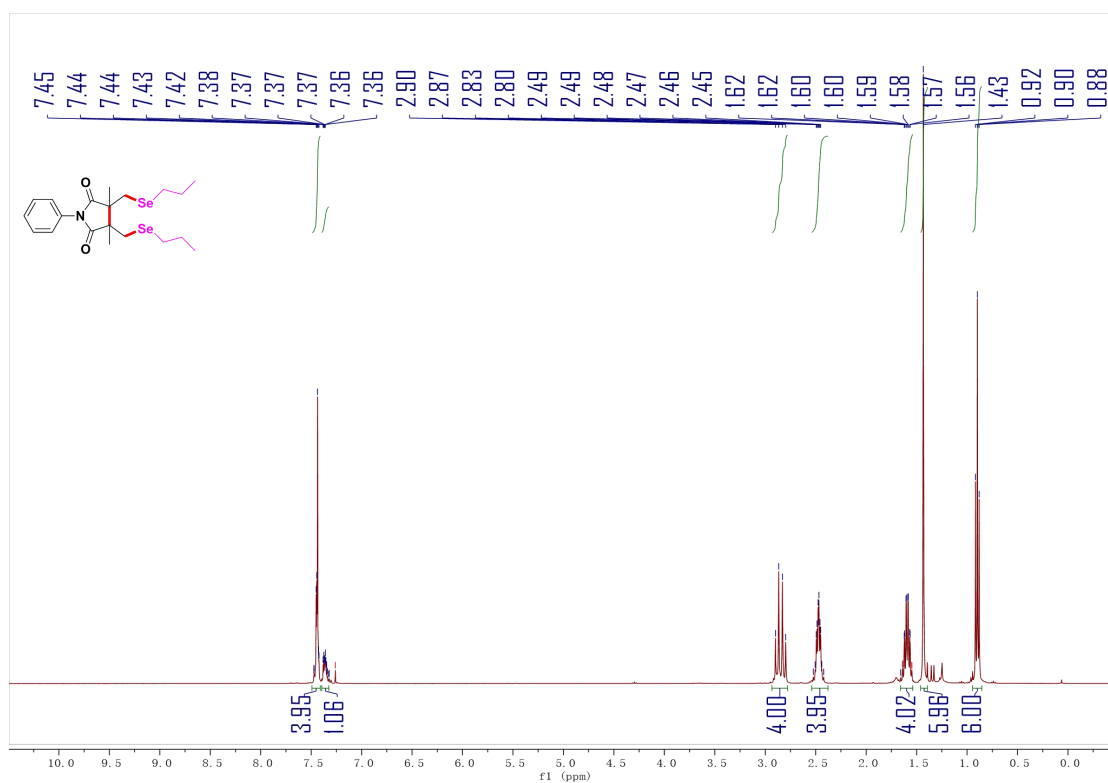
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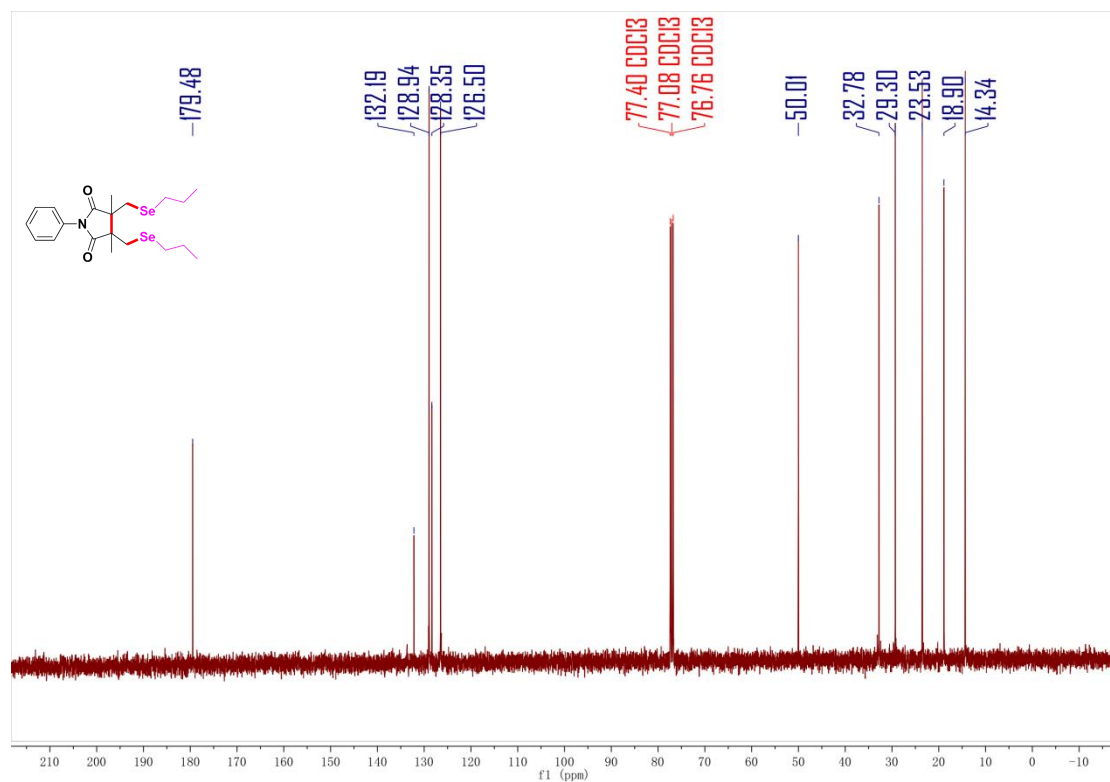
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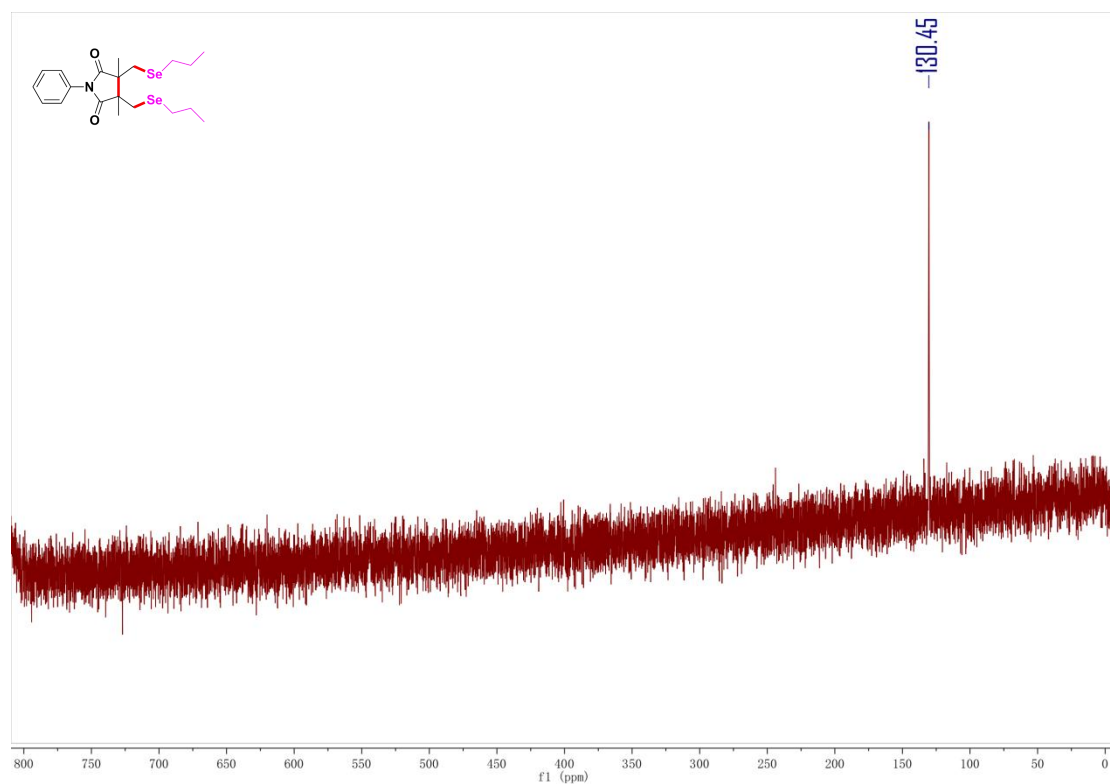
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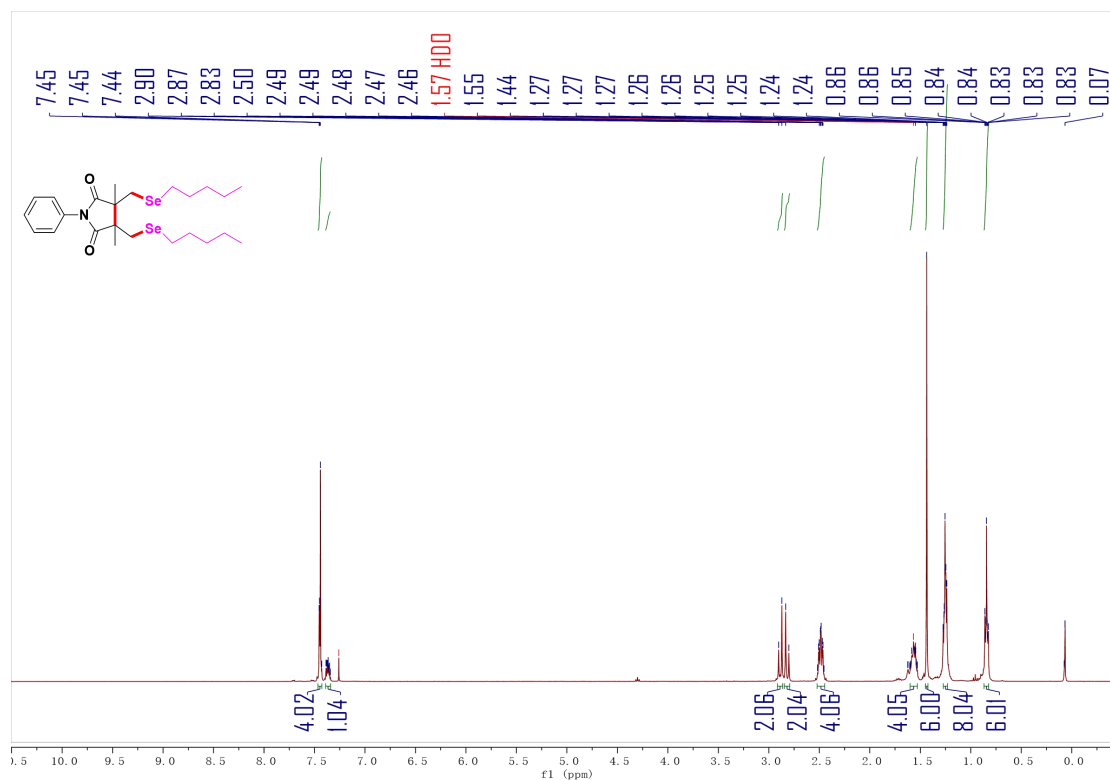
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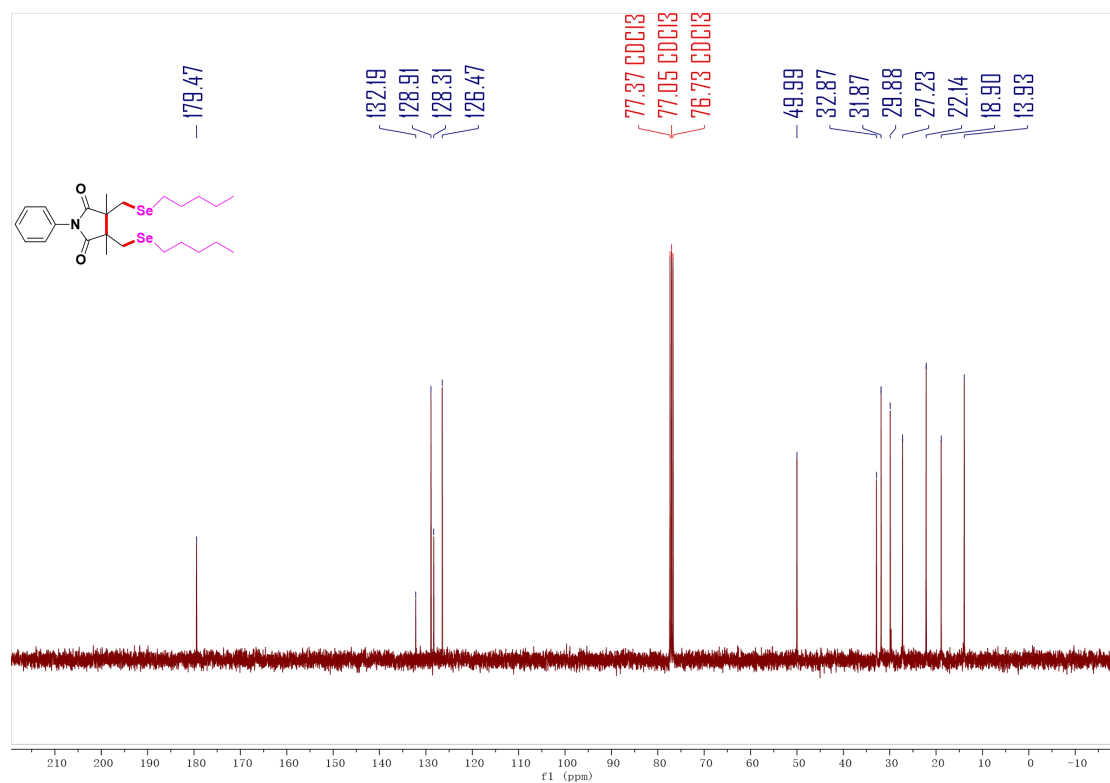
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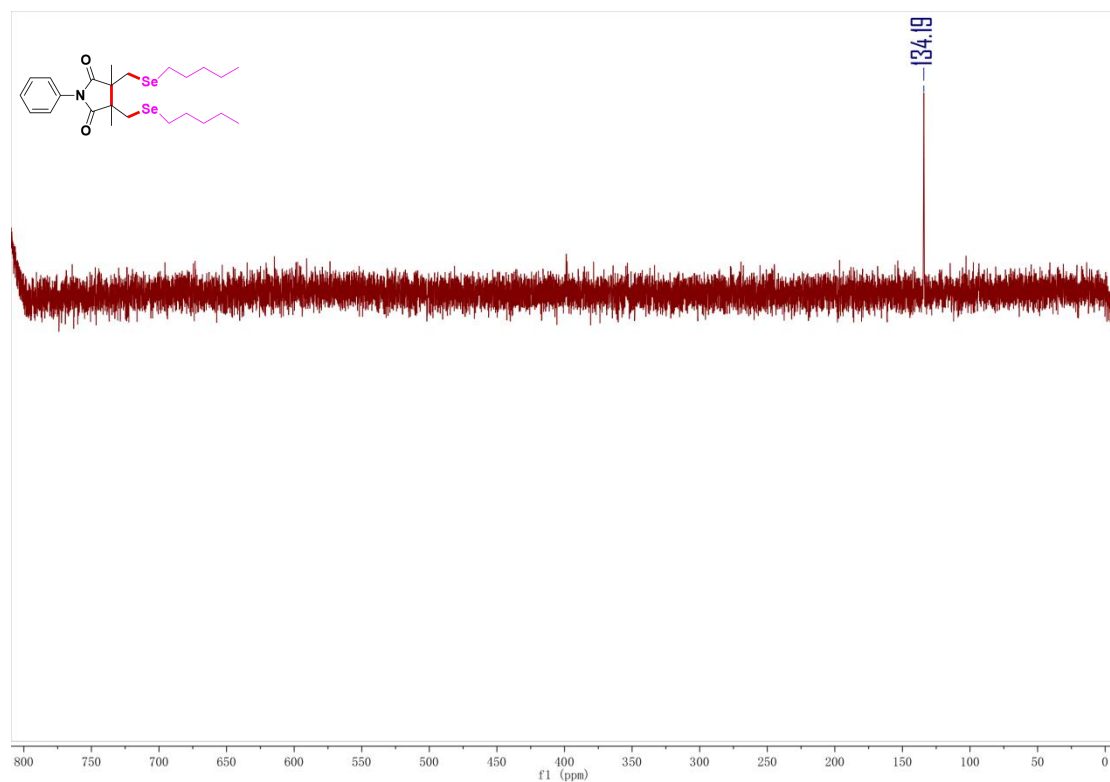
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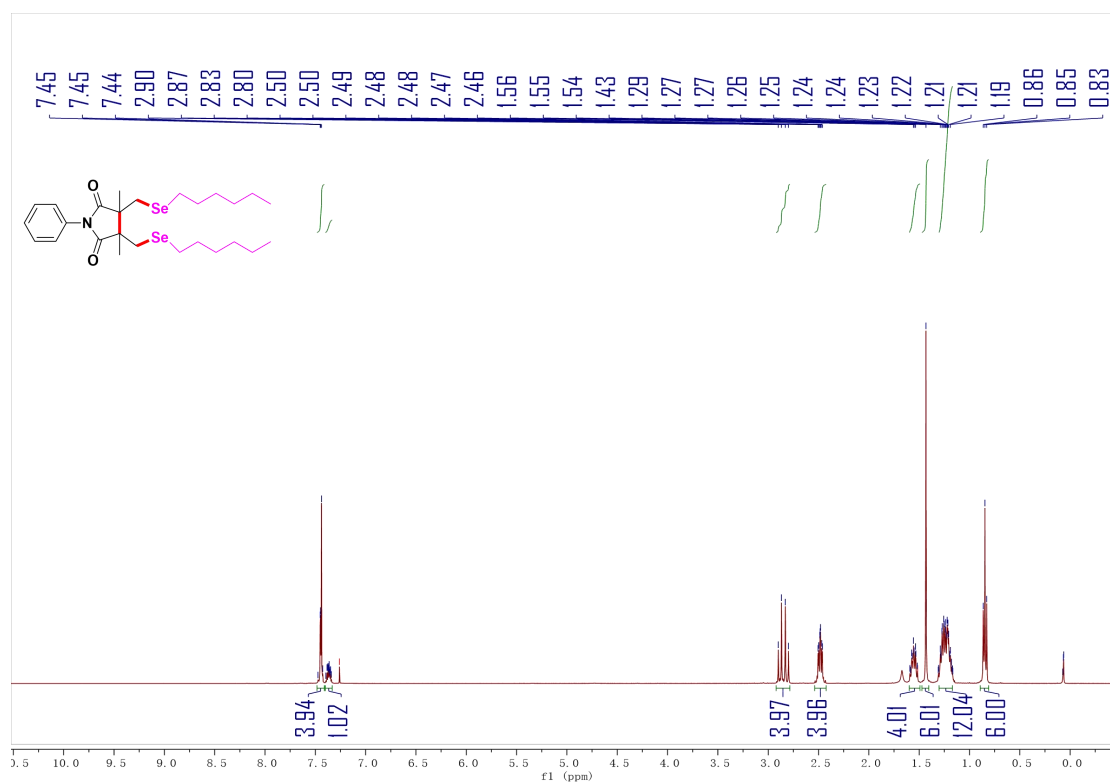
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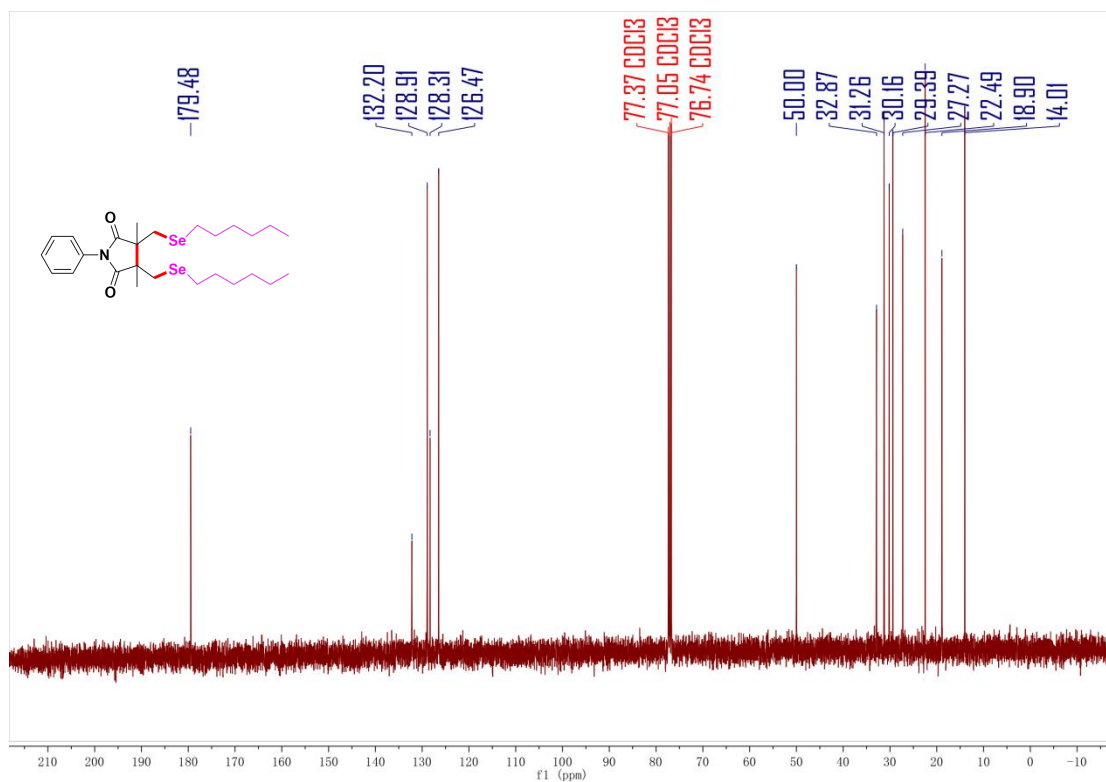
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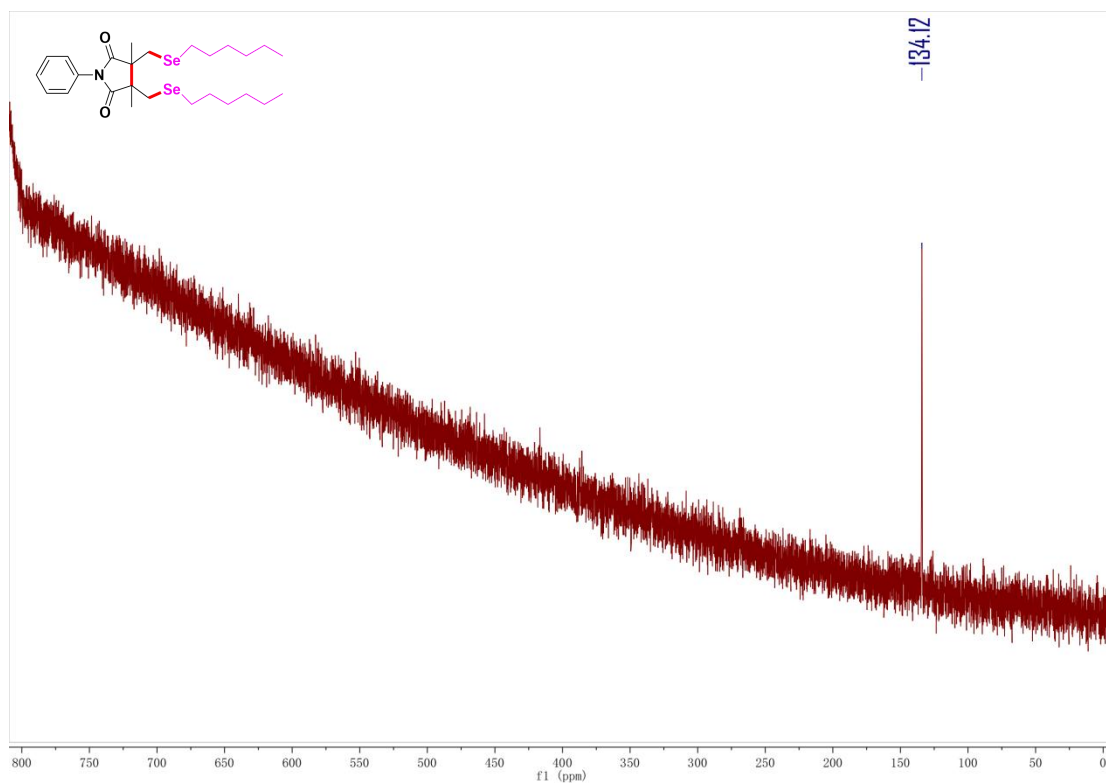
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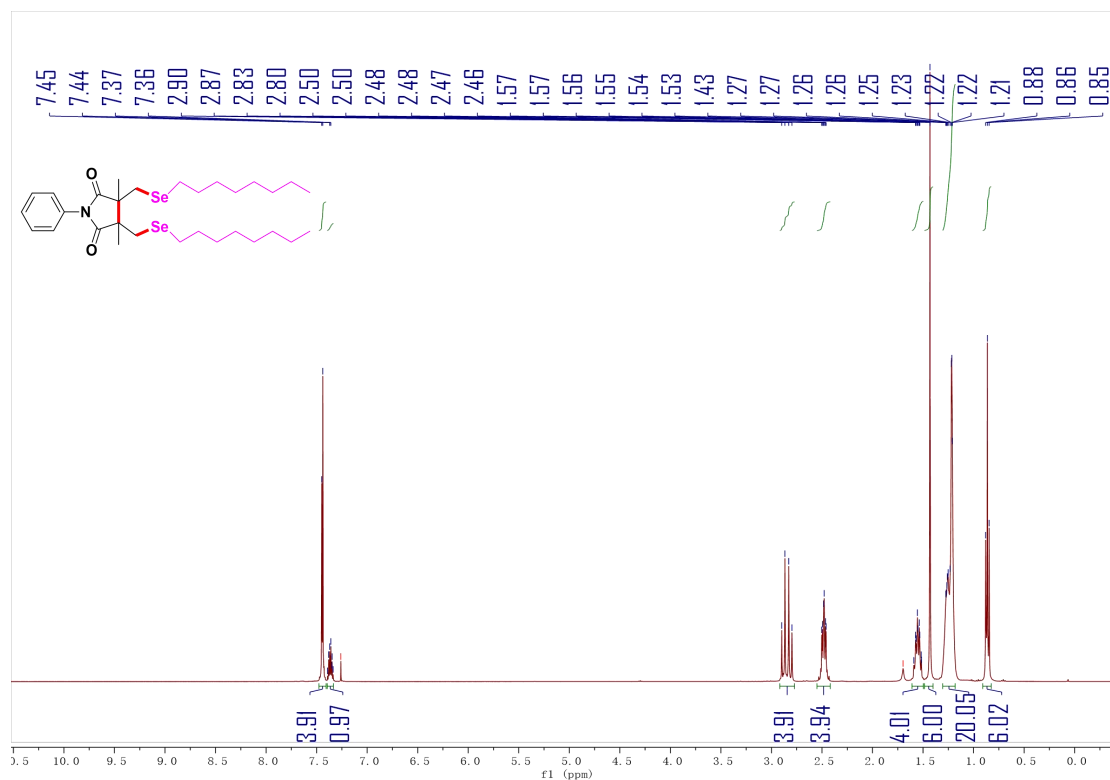
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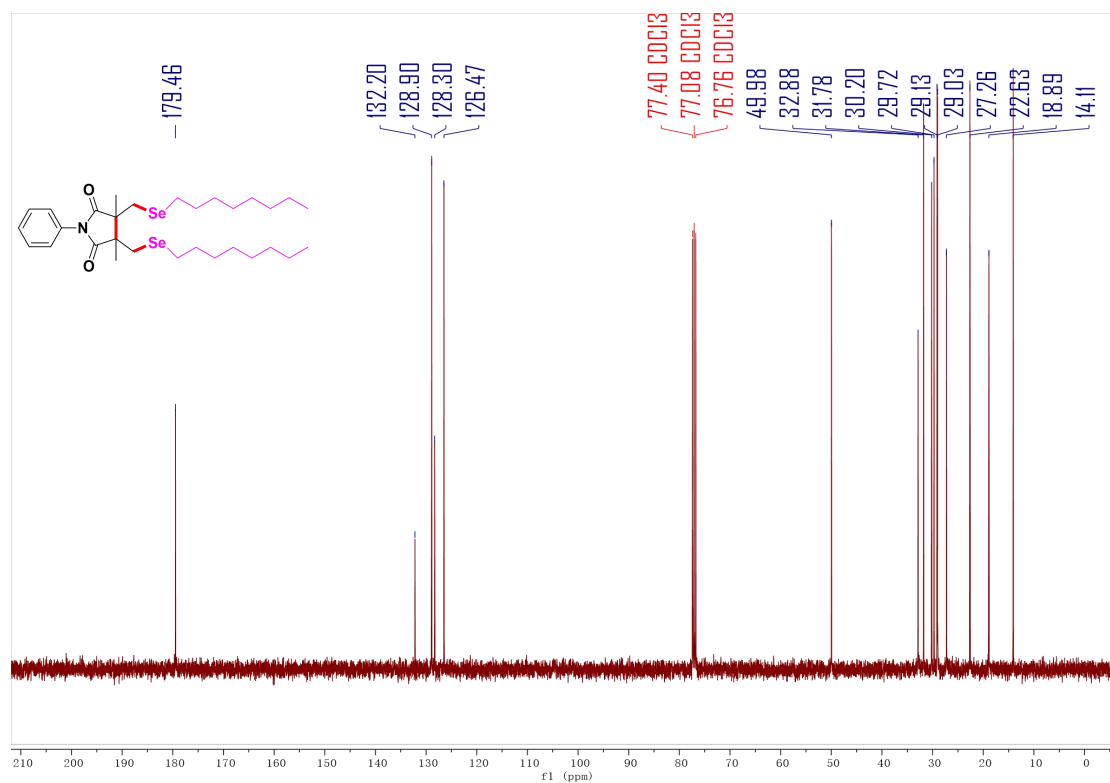
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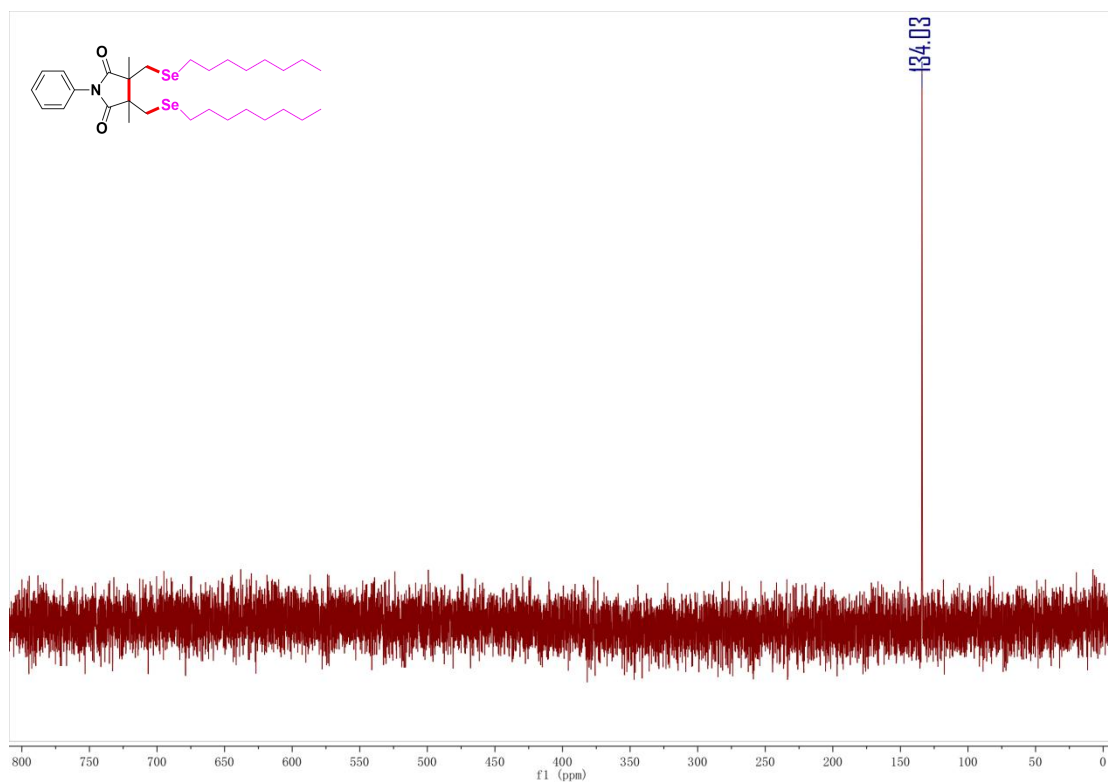
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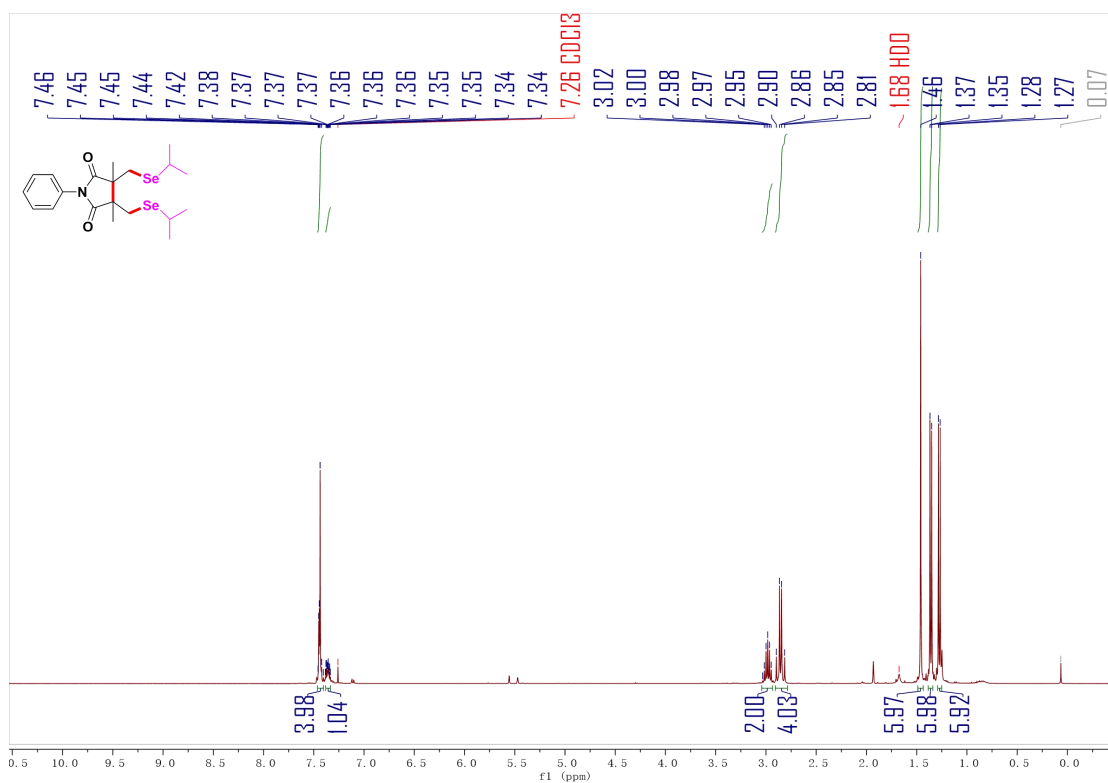
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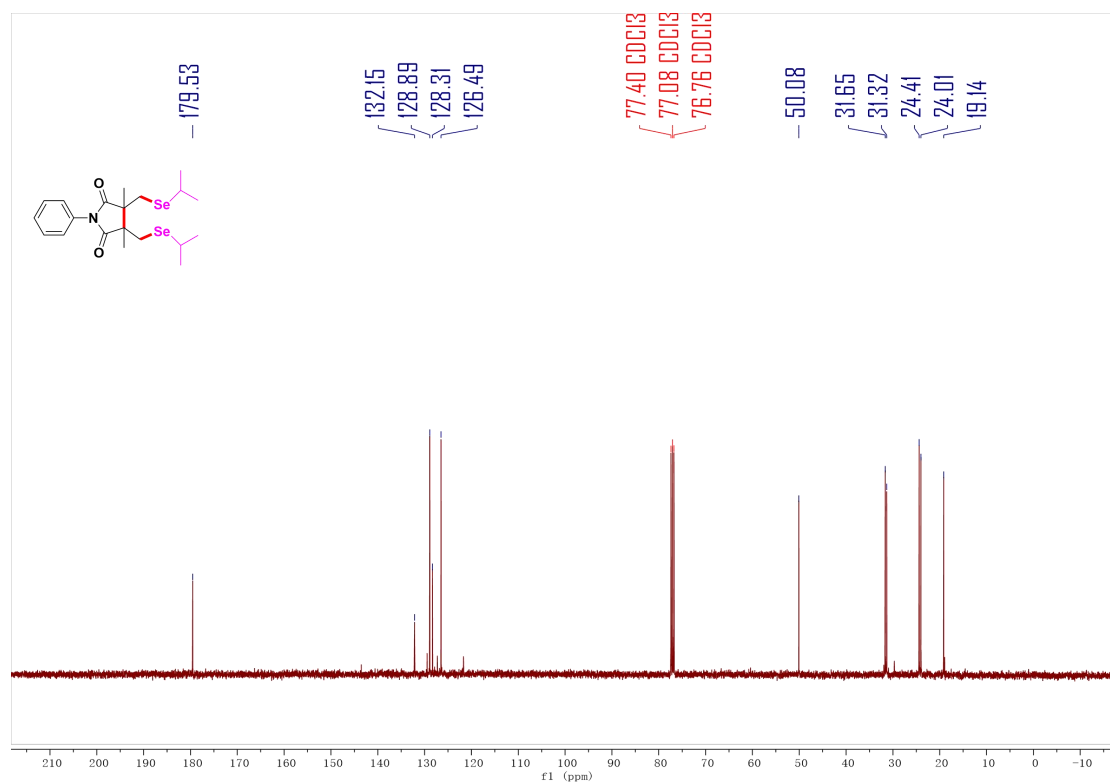
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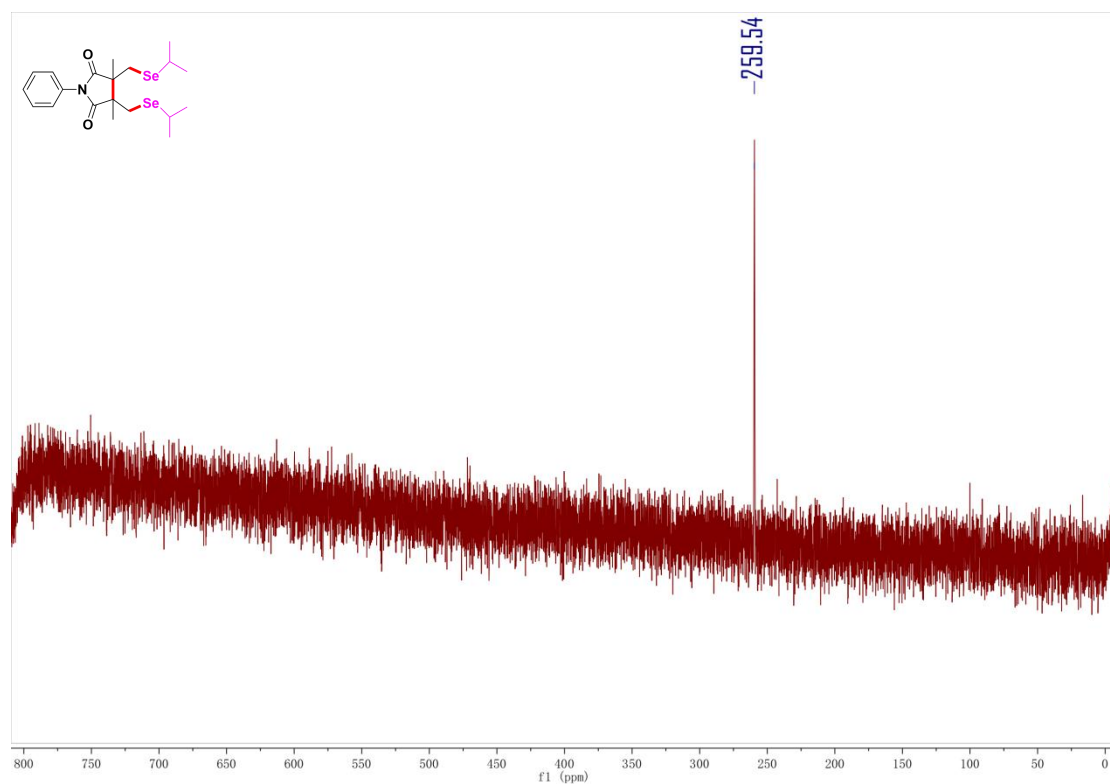
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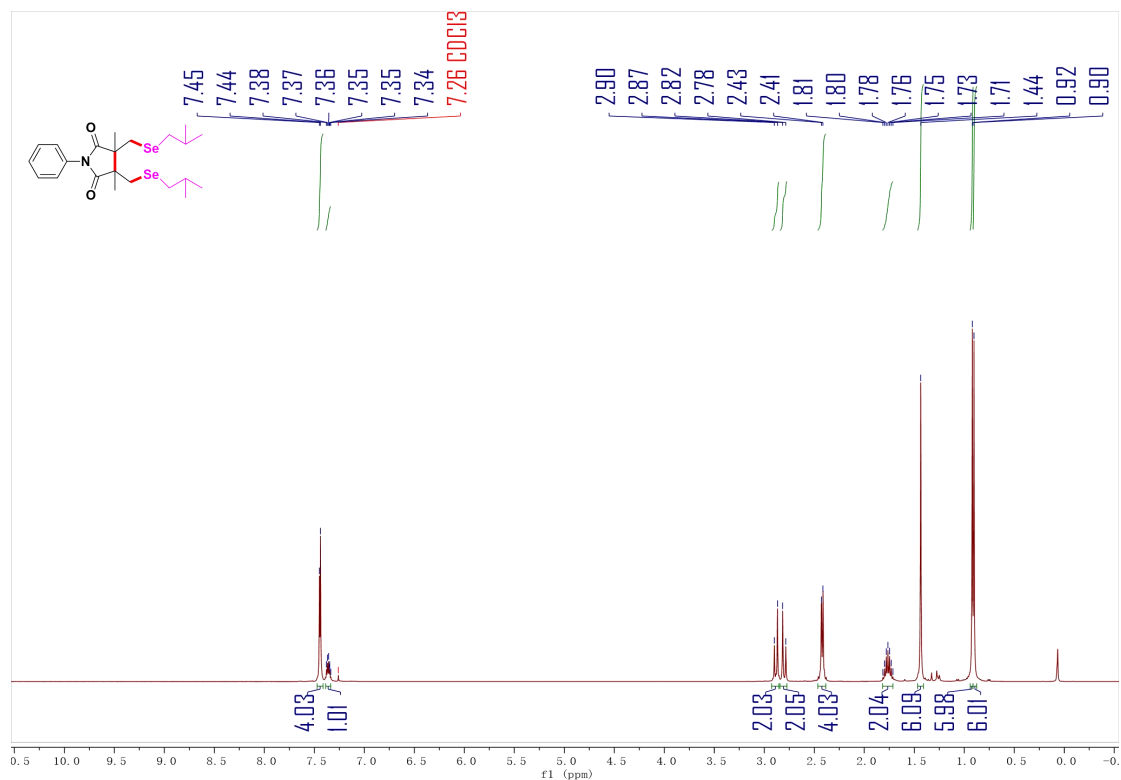
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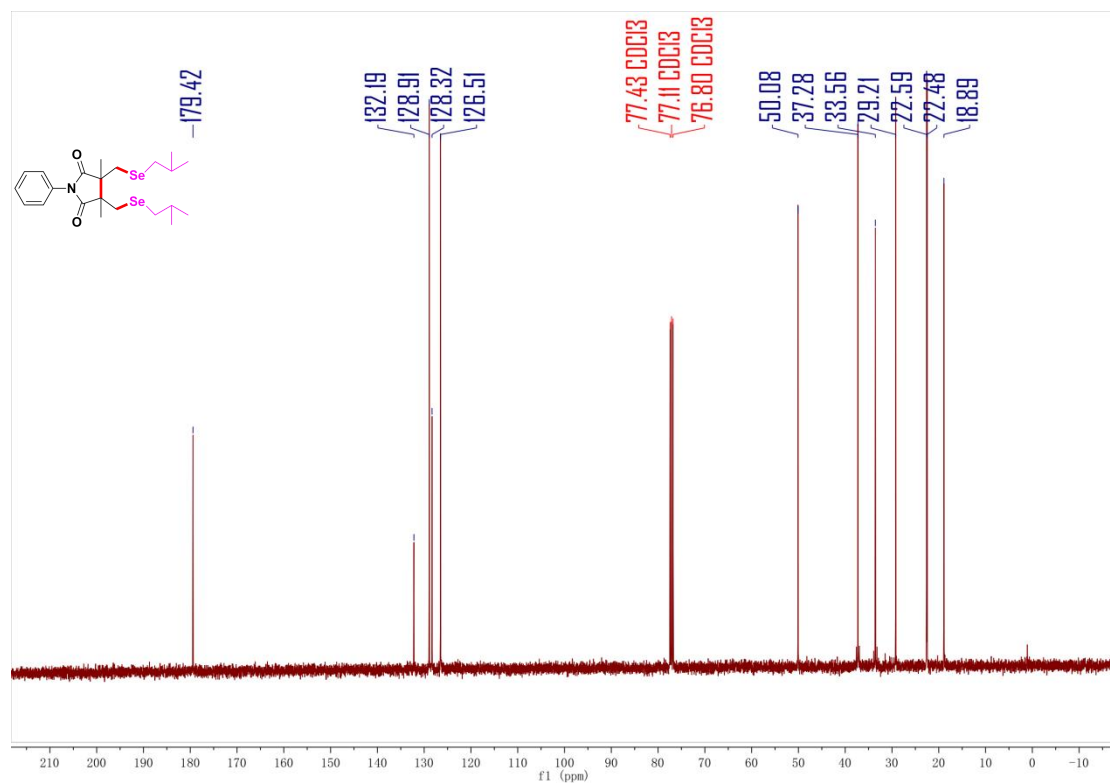
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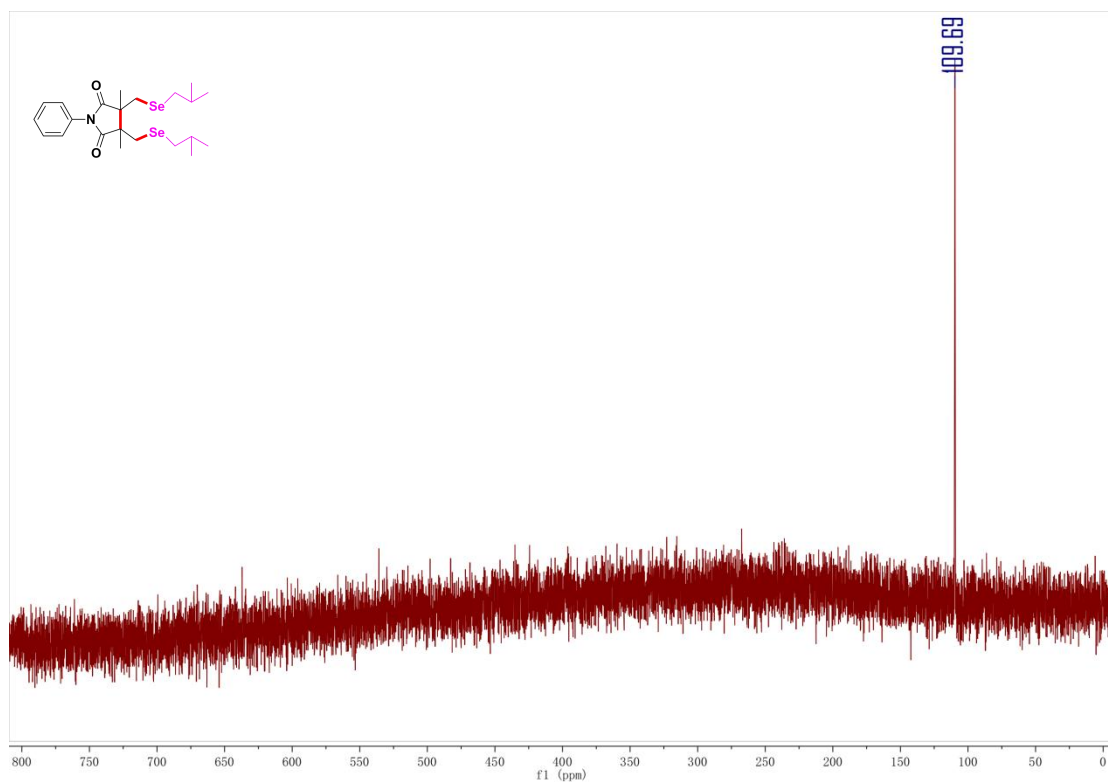
⁷⁷Se NMR Spectra of 5f2 (76 MHz, CDCl₃)



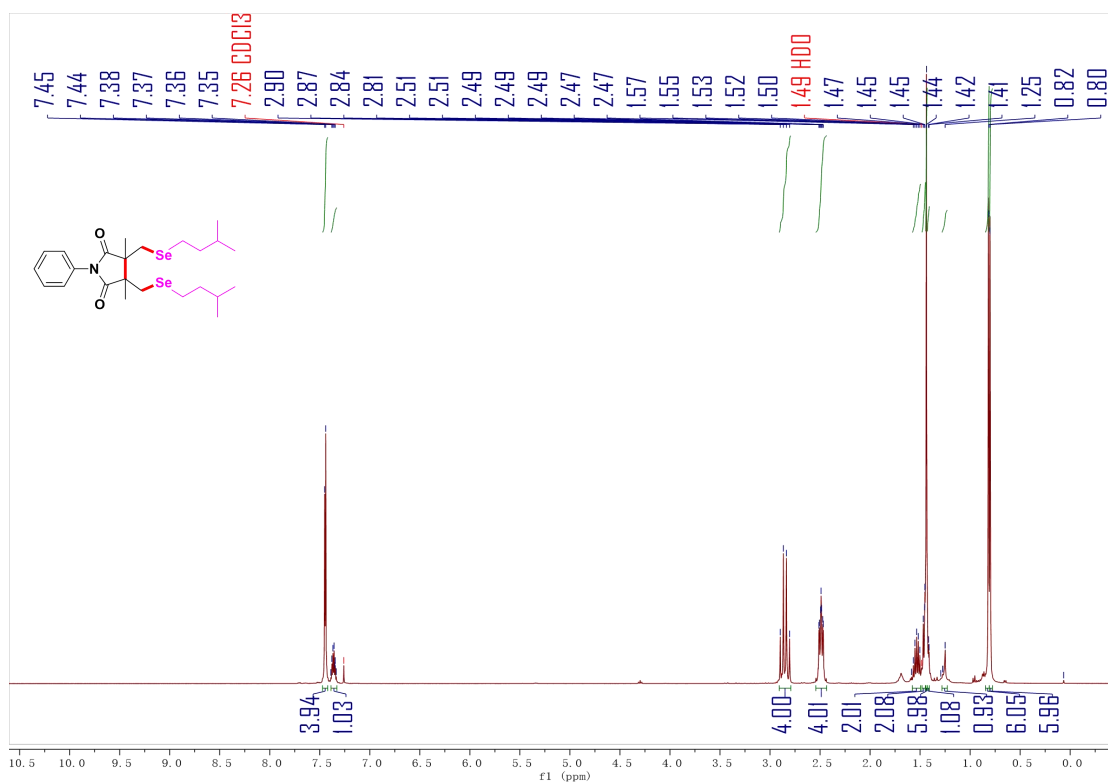
¹H NMR Spectra of 5g2 (400 MHz, CDCl₃)



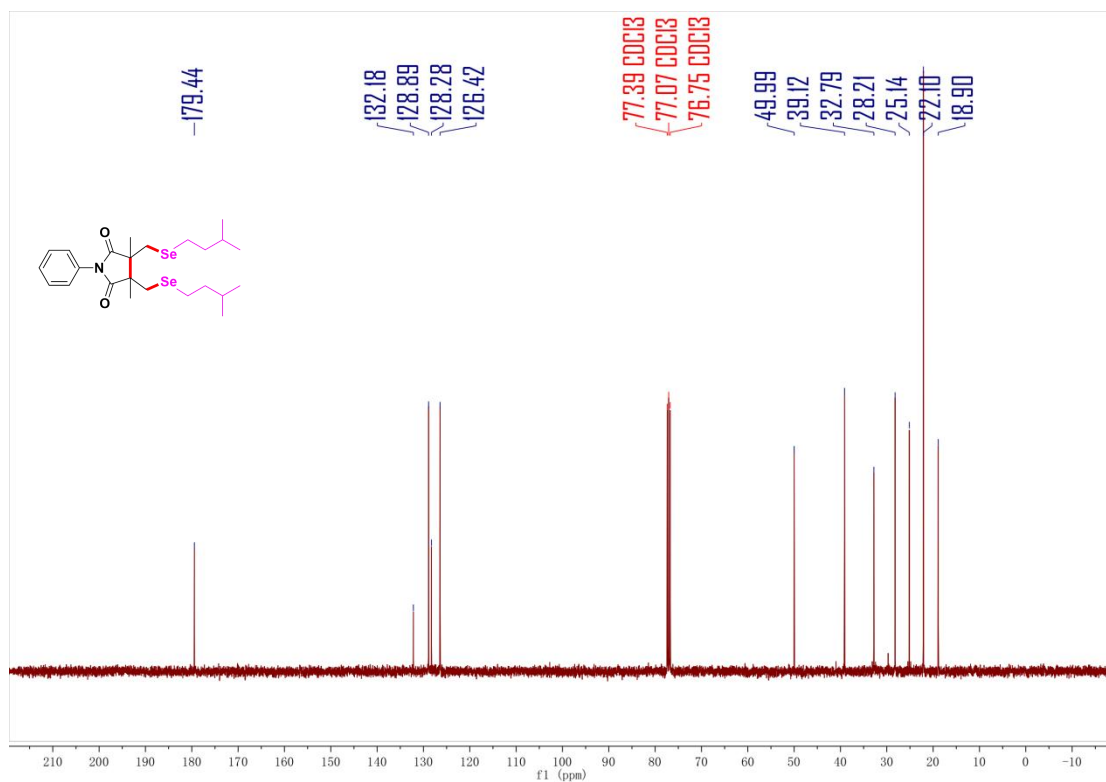
¹³C NMR Spectra of 5g2 (100 MHz, CDCl₃)



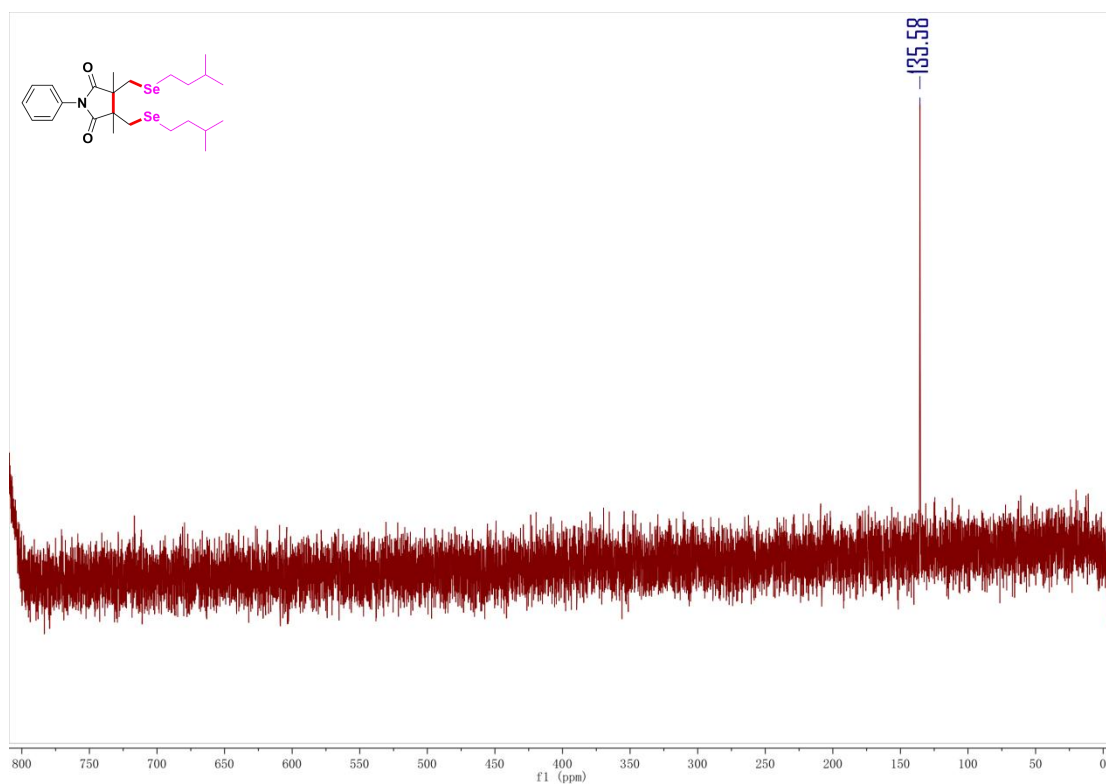
⁷⁷Se NMR Spectra of 5g2 (76 MHz, CDCl₃)



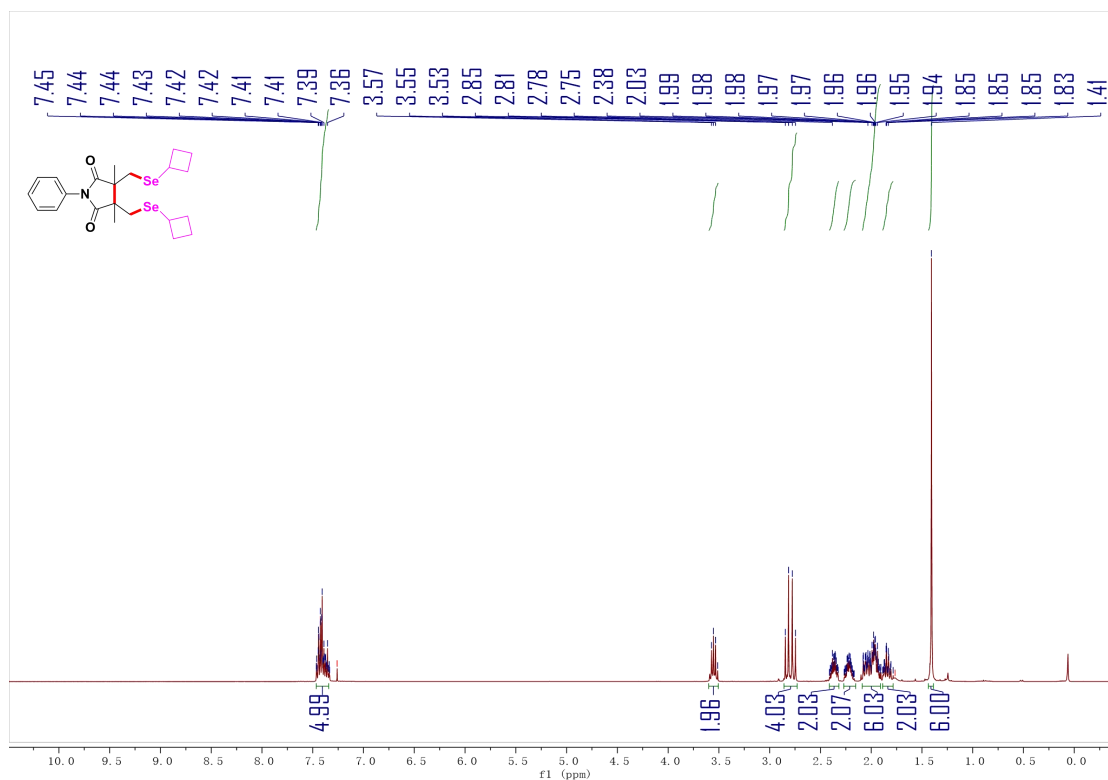
¹H NMR Spectra of 5i2 (400 MHz, CDCl₃)



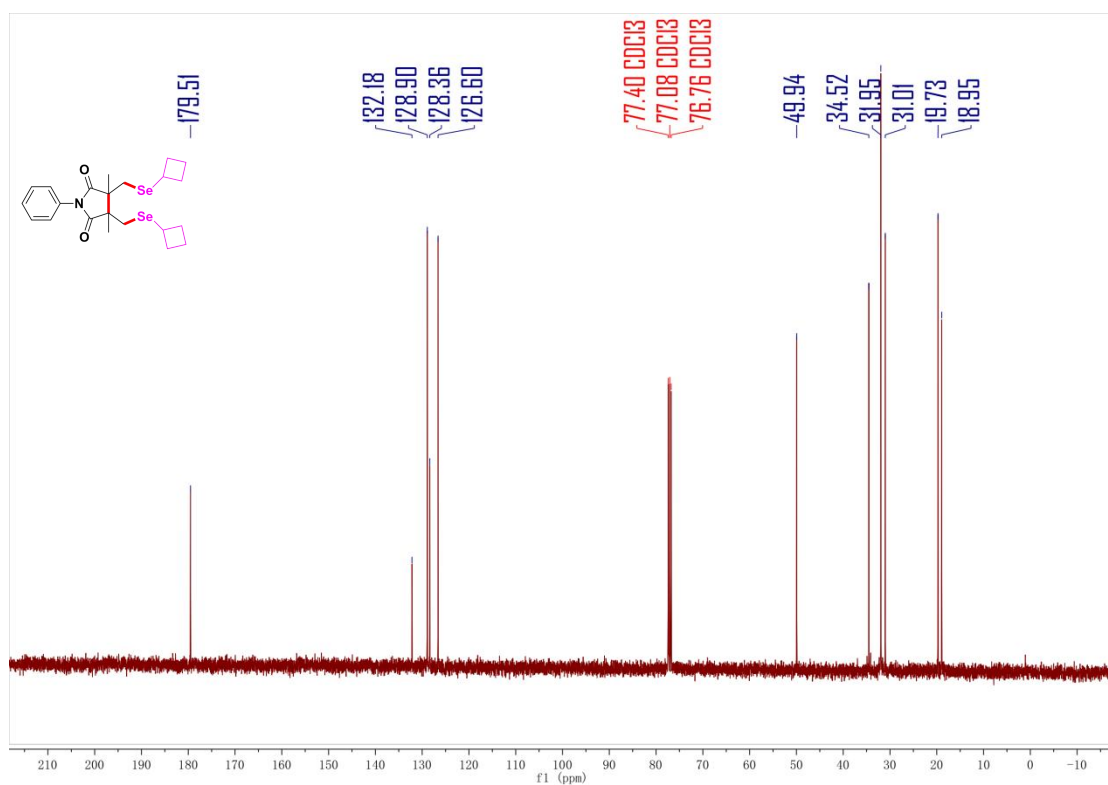
¹³C NMR Spectra of 5i2 (100 MHz, CDCl₃)



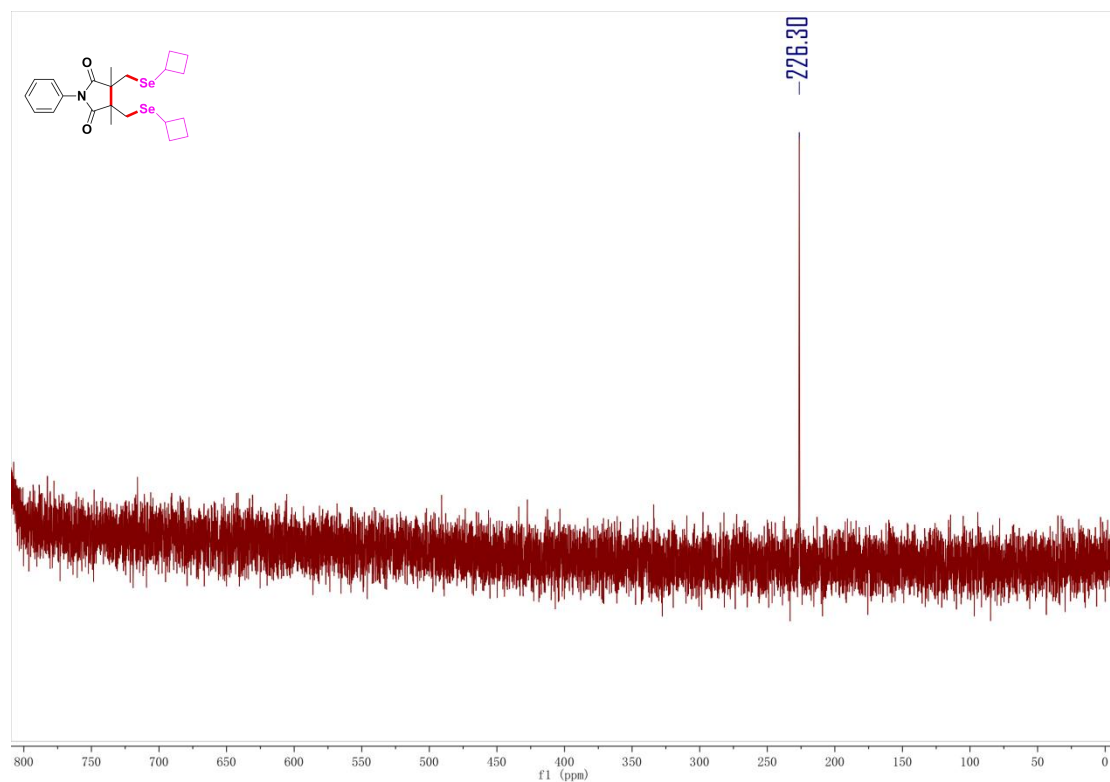
⁷⁷Se NMR Spectra of 5i2 (76 MHz, CDCl₃)



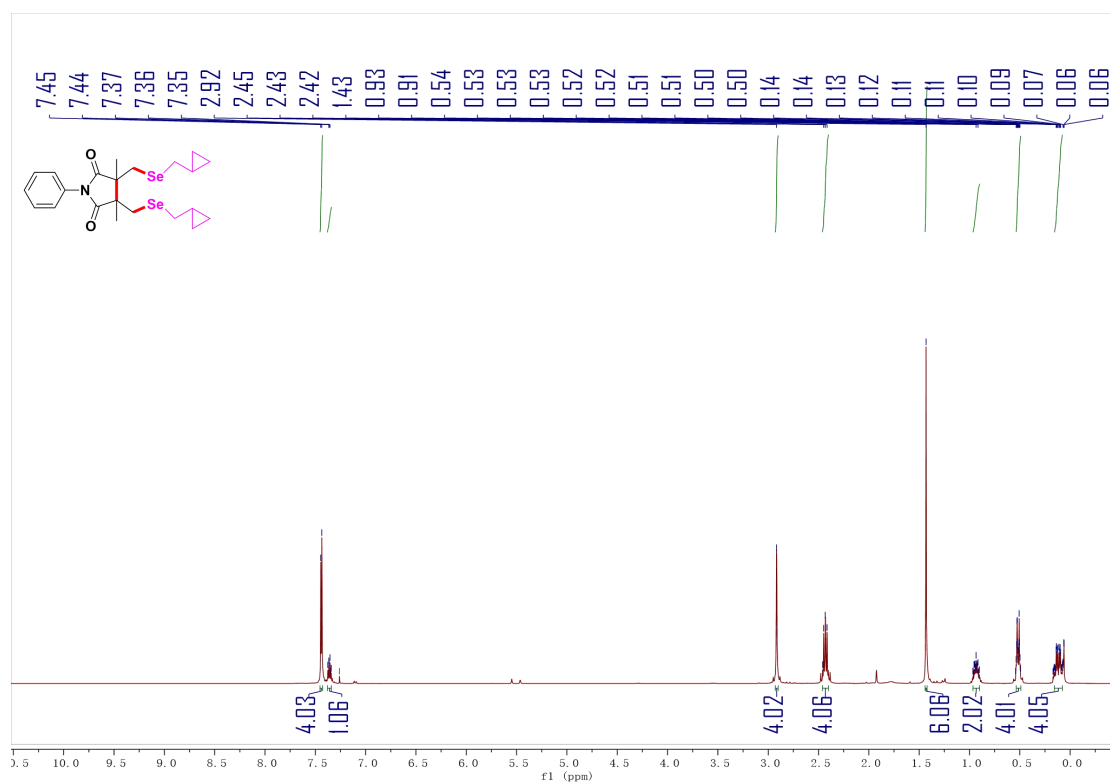
¹H NMR Spectra of 5j2 (400 MHz, CDCl₃)



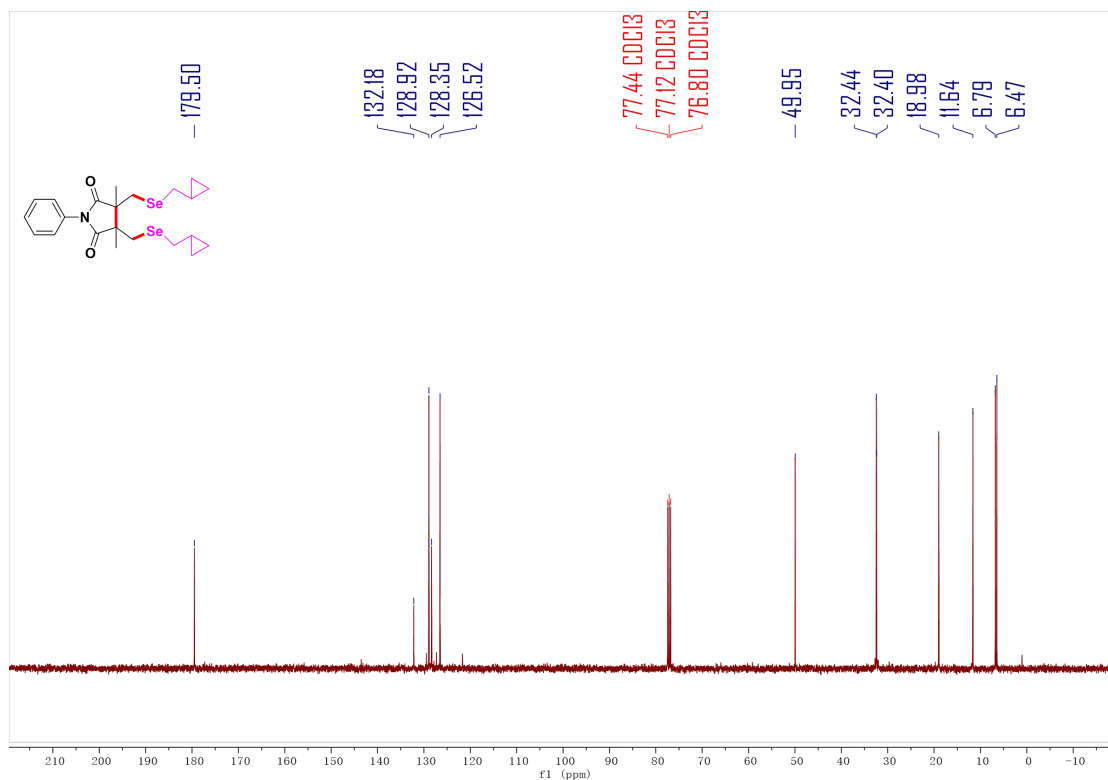
¹³C NMR Spectra of 5j2 (100 MHz, CDCl₃)



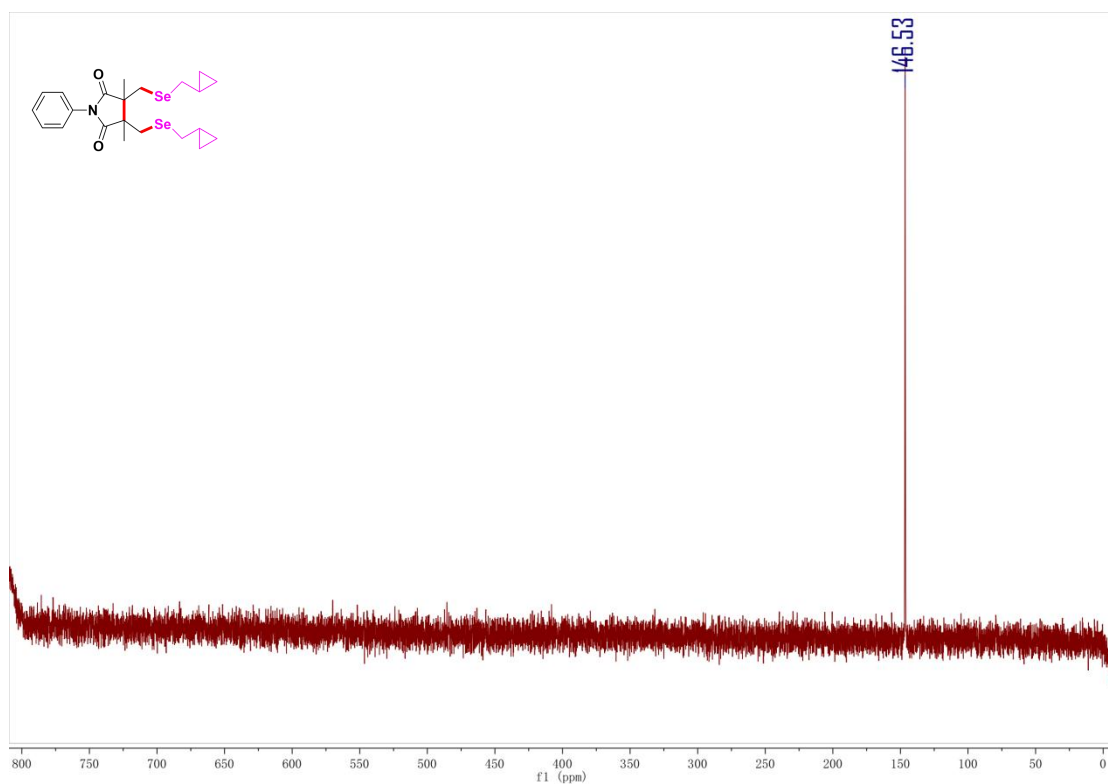
⁷⁷Se NMR Spectra of 5j2 (76 MHz, CDCl₃)



¹H NMR Spectra of 5k2 (400 MHz, CDCl₃)



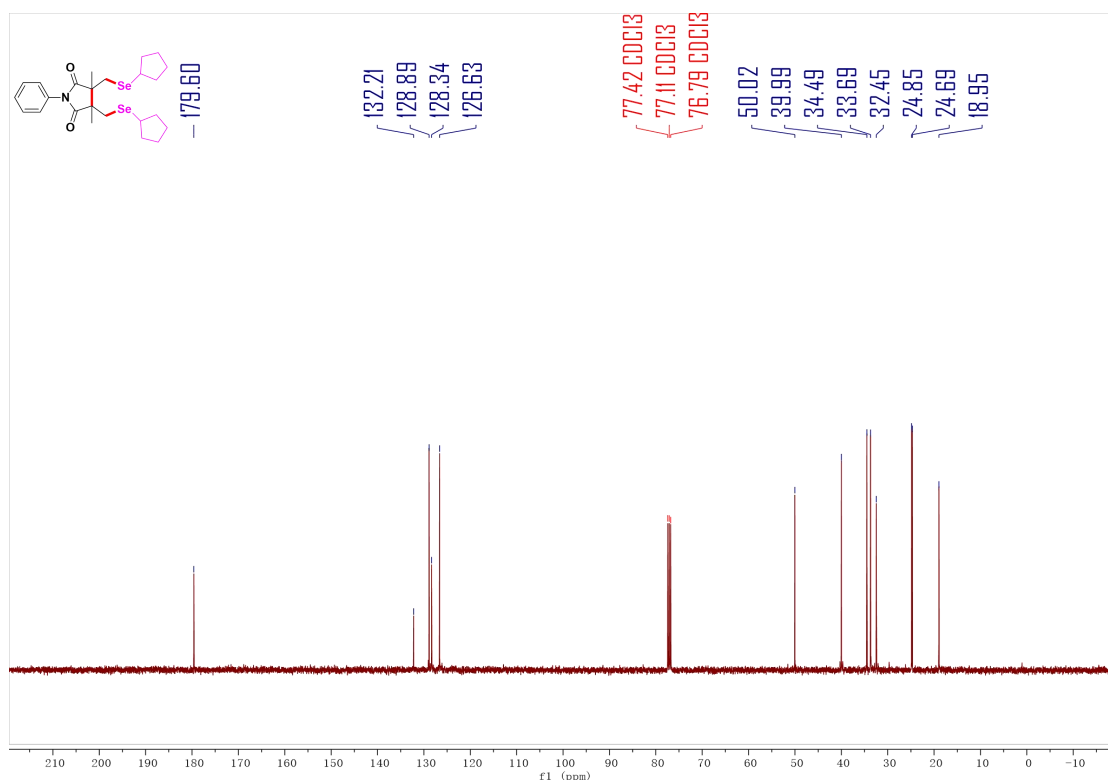
¹³C NMR Spectra of 5k2 (100 MHz, CDCl₃)



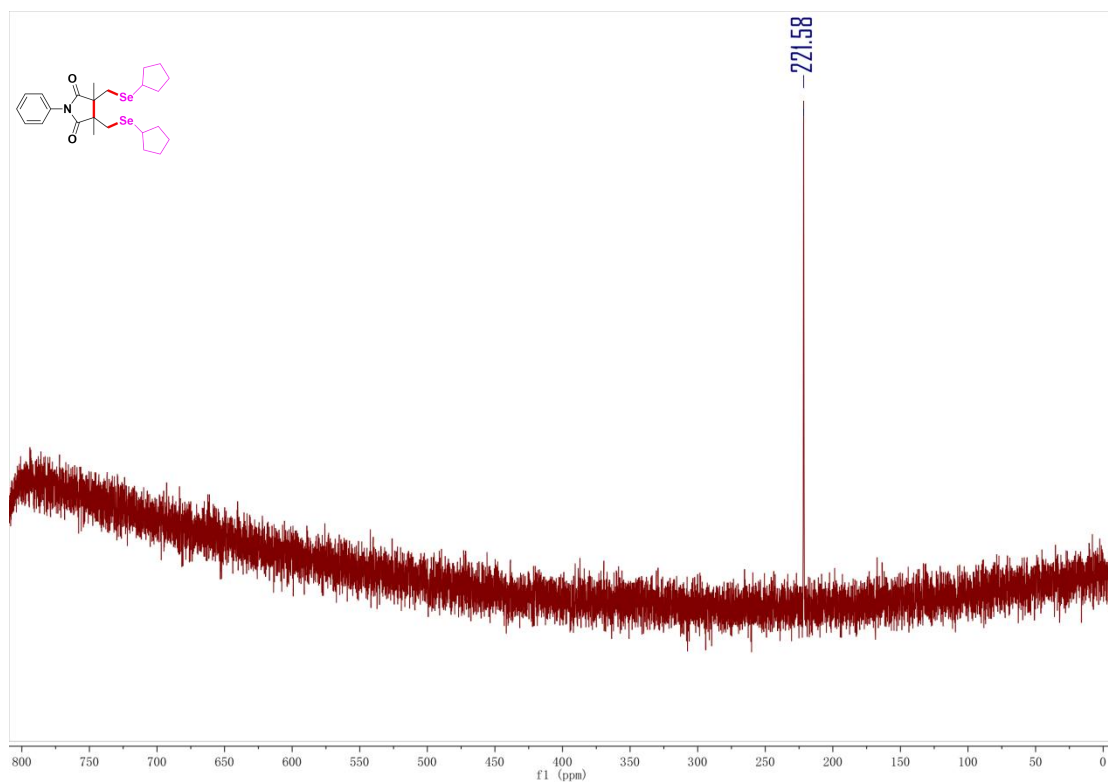
⁷⁷Se NMR Spectra of 5k2 (76 MHz, CDCl₃)



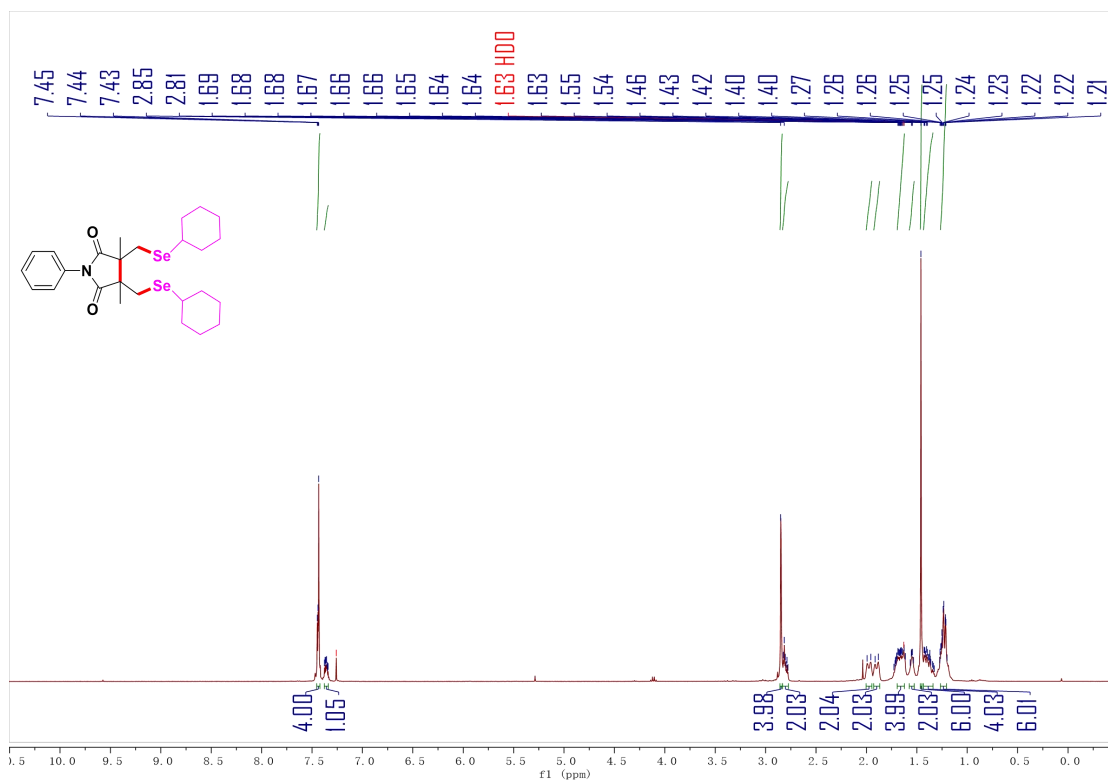
¹H NMR Spectra of 512 (400 MHz, CDCl₃)



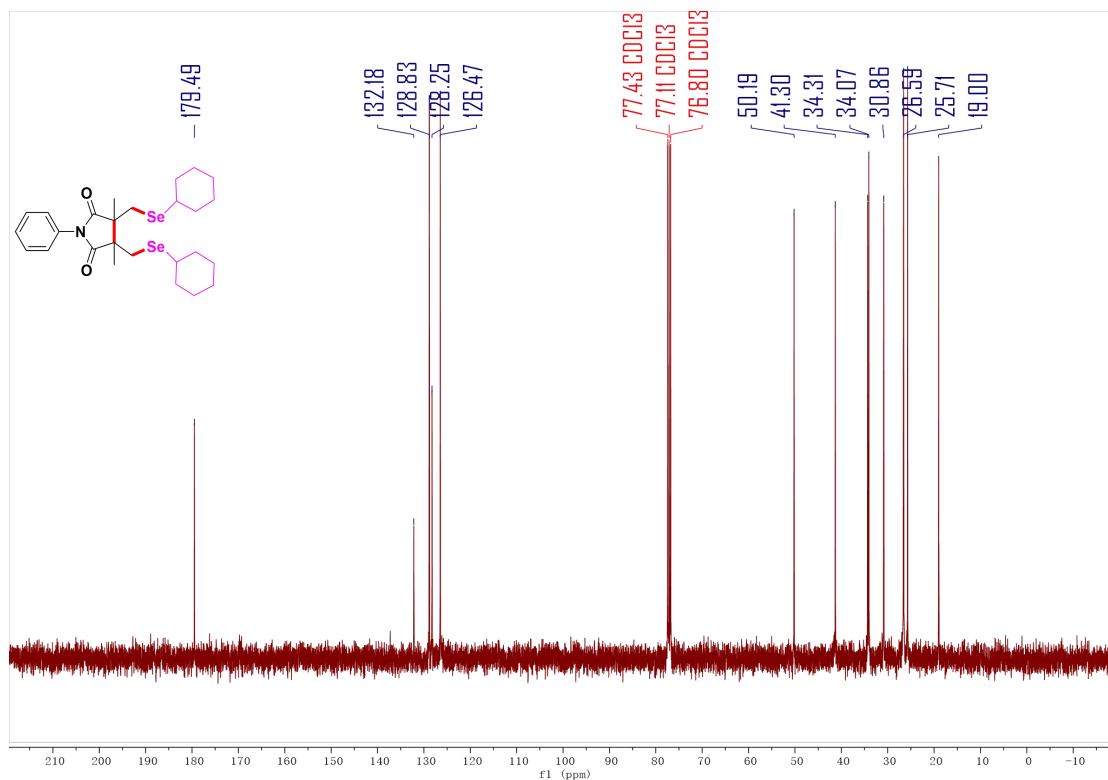
¹³C NMR Spectra of 512 (100 MHz, CDCl₃)



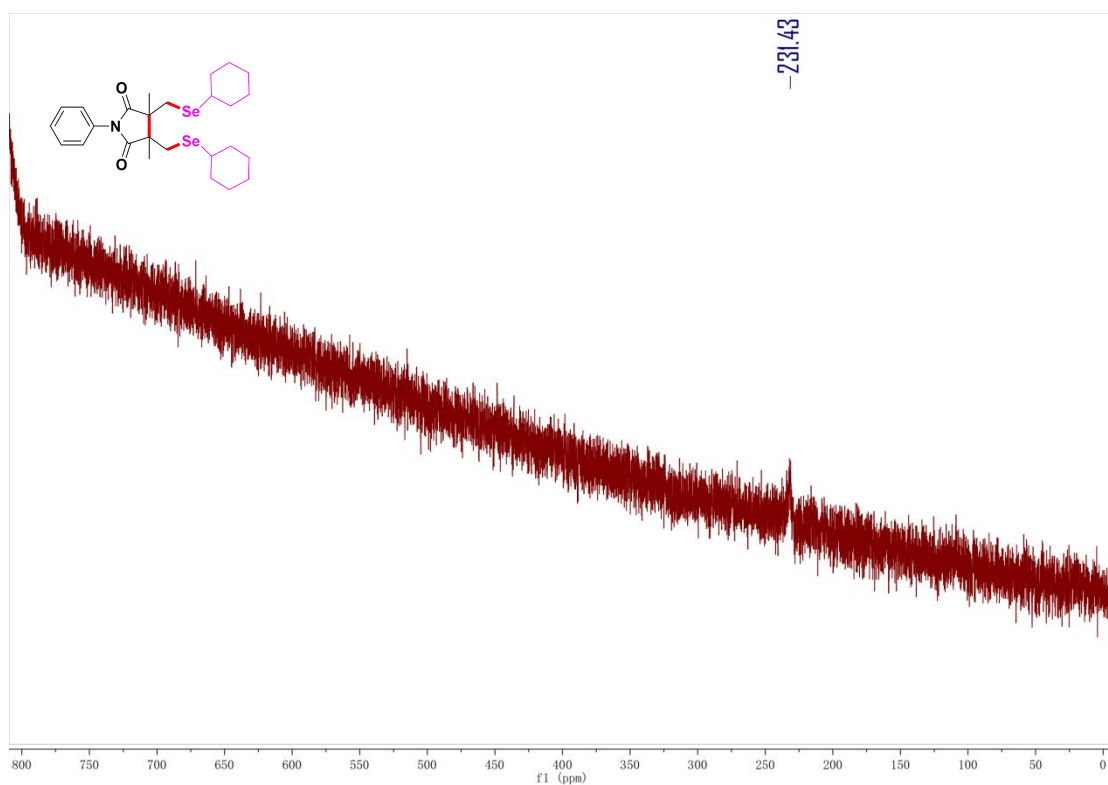
⁷⁷Se NMR Spectra of 5l2 (76 MHz, CDCl₃)



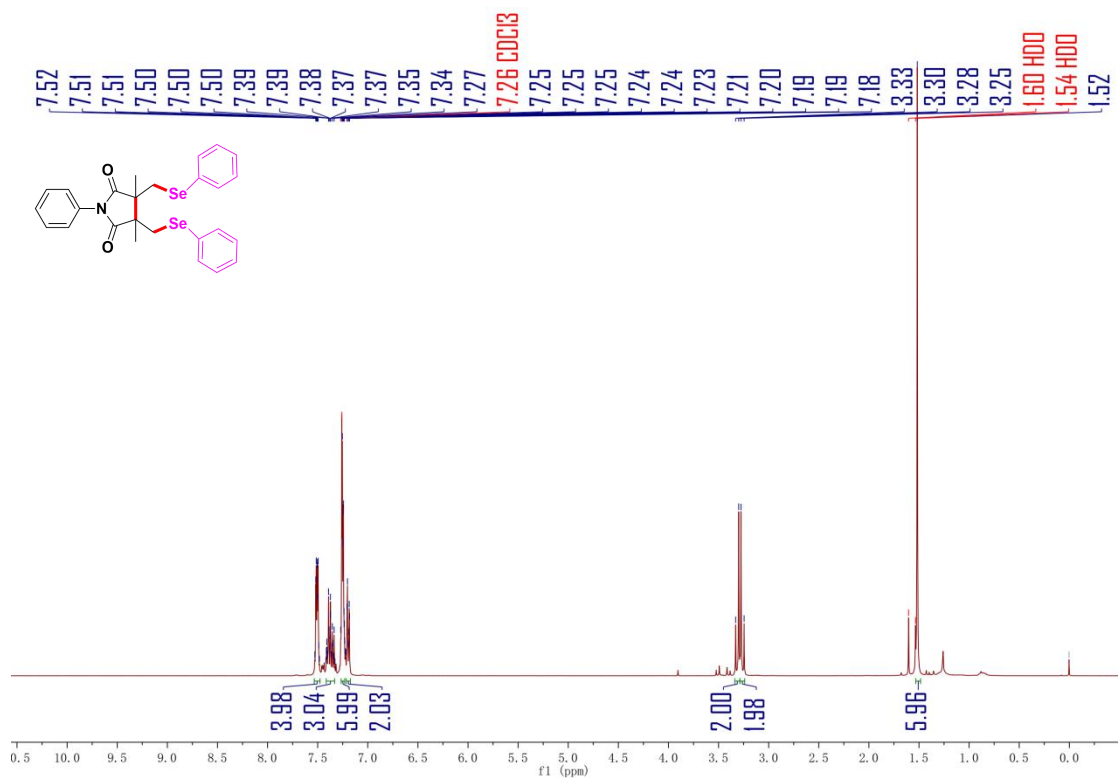
¹H NMR Spectra of 5m2 (400 MHz, CDCl₃)



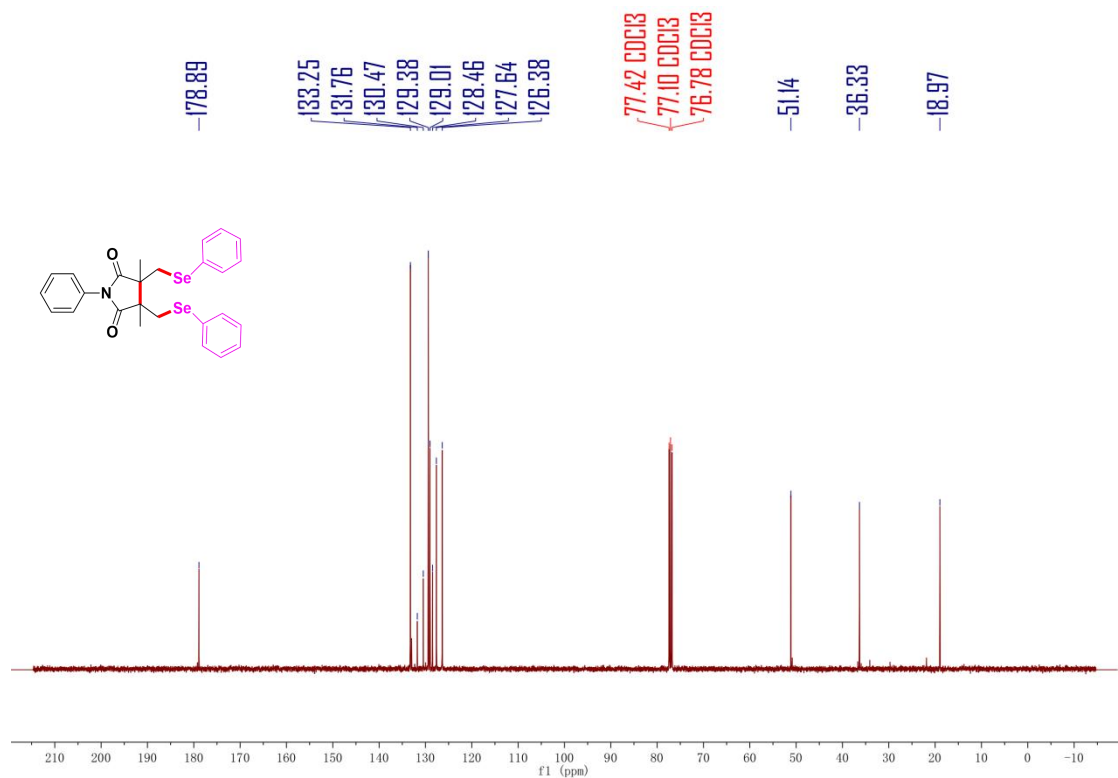
¹³C NMR Spectra of 5m2 (100 MHz, CDCl₃)



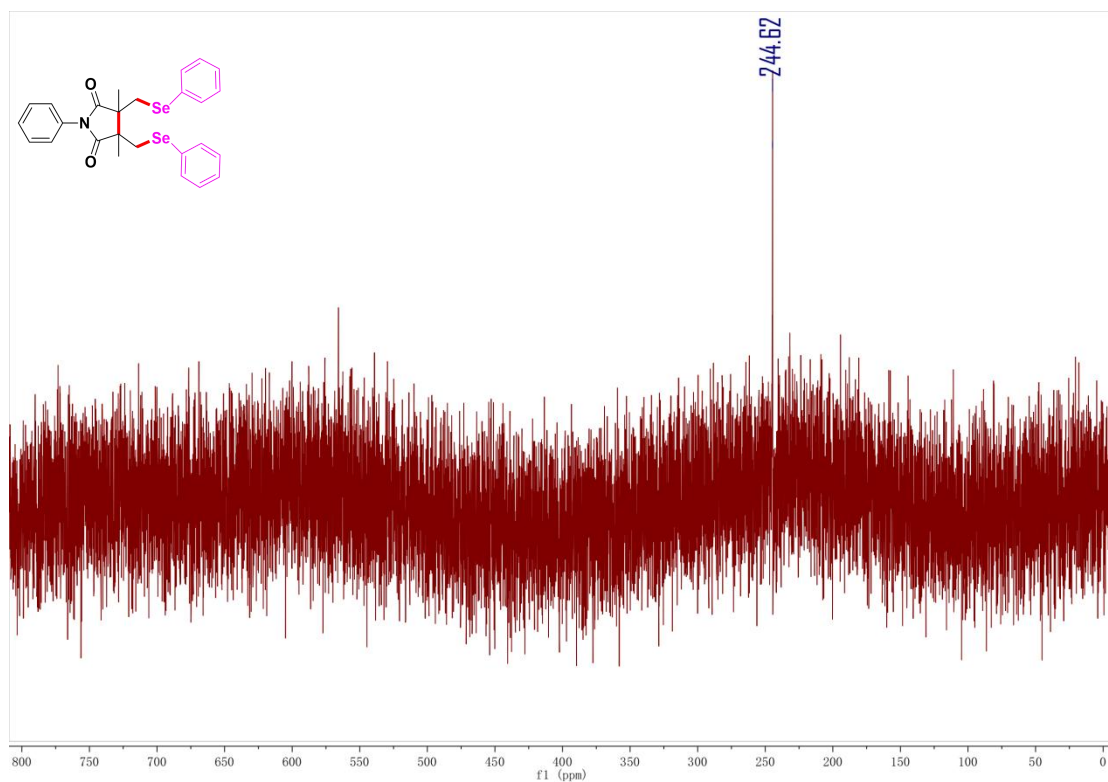
⁷⁷Se NMR Spectra of 5m2 (76 MHz, CDCl₃)



¹H NMR Spectra of 5n2 (400 MHz, CDCl₃)



¹³C NMR Spectra of 5n2 (100 MHz, CDCl₃)



^{77}Se NMR Spectra of **5n2** (76 MHz, CDCl_3)