Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information for

Acid-Catalyzed Oxidative Cleavage of S–S and Se–Se Bonds with DEAD: Efficient Access to Sulfides and Selenides

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1. General considerations

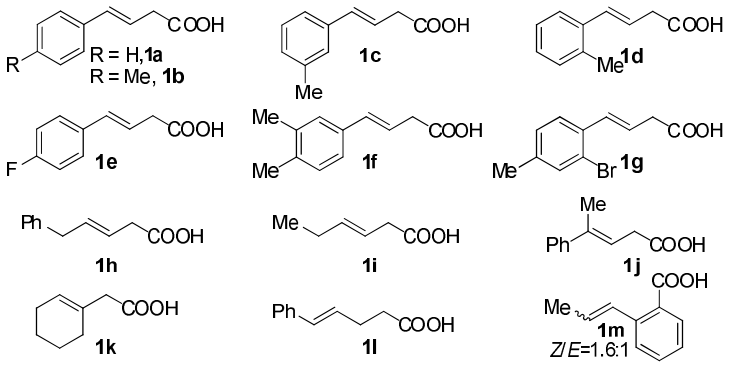
Unless otherwise noted, commercial reagents were purchased from Adamas, Alfa Aesar, Aladdin, Accela, *J&K*, Macklin, or TCI, and used without further purification. All the reactions were carried out without special care. Flash column chromatography was carried out on 200-300 mesh silica gels (Qingdao, China).

^{1}H, ^{19}F and $^{13}C\{^{1}H\}$ NMR spectra were recorded on an AVANCE III 400MHz spectrometer at ambient temperature. ^{1}H NMR spectra are referred to the TMS signal and ^{13}C NMR spectra are referred to the residual solvent signal. Data for ^{1}H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant (Hz), integration. Data for $^{13}C\{^{1}H\}$ NMR and ^{19}F NMR are reported as follows: chemical shift (δ ppm), multiplicity (d = doublet, q = quartet), coupling constant (Hz).

High resolution mass spectra of novel compounds were recorded on LTQ Orbitrap Elite LC/MS (ESI) at analytical center of Sun Yat-Sen University, and Thermo Fisher Scientific LTQ FT Ultra at National Center for Organic Mass Spectrometry in Shanghai Institute of Organic Chemistry, Chinese Academic of Sciences.

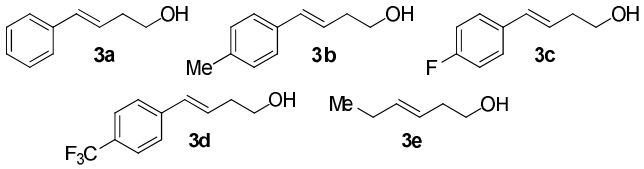
2. Experimental procedures and characterization data

2.1 Preparation of (*E*)-4-phenylbut-3-enoic acid analogues



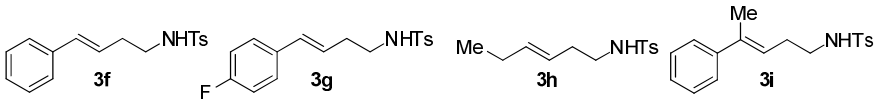
Substrates **1b**,[1] **1c**,[1] **1d**,[1] **1e**,[1] **1f**,[1] **1g**,[1] **1h**,[2] **1j**,[3] **1l**,[4] and **1m**[5] were prepared according to the literature methods. All the above prepared compounds are known and were identified by comparison of their NMR data with those reported in the literature. Substrates **1a**, **1i**, **1k** were commercial available.

2.2 Preparation of (*E*)-4-phenylbut-3-en-1-ol analogues



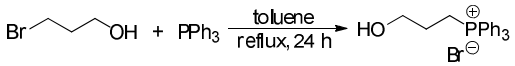
Substrates **3a**,[6] **3b**,[6] **3c**,[6] and **3d**[6] were prepared according to the literature methods. All the above prepared compounds are known and were identified by comparison of their NMR data with those reported in the literature. Substrate **3e** was commercial available.

2.3 Preparation of (*E*)-4-methyl-*N*-(4-phenylbut-3-en-1-yl)benzenesulfonamide analogues

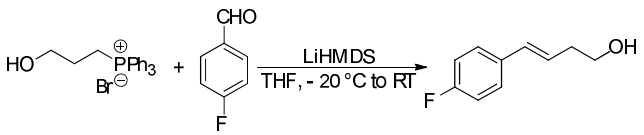


Substrates **3f**,[7] **3h**,[8] and **3i**[9] are known and were prepared according to the typical procedure for the synthesis of **3g**.

Typical procedure for the synthesis of substrate **3g**:[6,10]

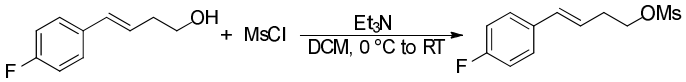


To an oven-dried 100 mL Schlenk flask equipped with a stir bar and Graham condenser was added PPh_3 (10.49 g, 40.0 mmol). The flask was then evacuated and back-filled with nitrogen for three times. Next, 60 mL anhydrous toluene and 3-bromo-l-propanol (5.40 mL, 60.0 mmol) were added via syringe. The homogeneous mixture was stirred at reflux for 24 h. The resulting colorless solution was cooled to room temperature and then 30 mL Et_2O was added. After keeping at -20 ºC for 4 h, the formed precipitate was filtered, washed with Et_2O (20 mL x 3), and dried *in vacuo* to afford the triphenylphosphonium bromide (14.97 g, 93%).

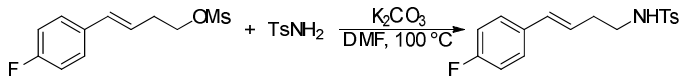


To an oven-dried 50 mL Schlenk flask equipped with a stir bar was added triphenylphosphonium bromide (2.89 g, 7.2 mmol). The flask was then evacuated and

back-filled with nitrogen for three times. Then 20 mL anhydrous THF was added via syringe and the suspension was cooled to -20 ºC. To the colorless suspension was added LiHMDS (1.0 M in THF, 15 mL, 15.0 mmol) dropwise via syringe over 5 min. The resulting orange solution was stirred at -20 ºC for 30 min and became homogeneous gradually. Then, a solution of 4-fluorobenzaldehyde (0.64 mL, 6.0 mmol) in 2 mL anhydrous THF was added via syringe at -20 ºC. After the resulting mixture was allowed to warm up to room temperature for 15 h, saturated aqueous NH_4Cl solution was added. The organic phase was extracted with ethyl acetate (10 mL x 3). The combined organic layers were dried over Na_2SO_4, filtered and then concentrated under reduced pressure. The residue was purified by silica gel column chromatography (eluent: PE/EA = 5/1, v/v) to afford (*E*)-4-(4-fluorophenyl)but-3-en-1-ol (0.61 g, 61%) as a colorless oil .

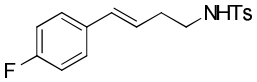


To an oven-dried 25 mL Schlenk flask equipped with a stir bar was added (*E*)-4-(4-fluorophenyl)but-3-en-1-ol (0.61 g, 3.7 mmol). The flask was then evacuated and back-filled with nitrogen for three times. Anhydrous DCM (10 mL) was added via syringe and the solution was cooled to 0 ºC. Then, triethylamine (1.53 mL, 11.0 mmol) and MsCl (0.43 mL, 5.5 mmol) were added dropwise via syringe. The mixture was warmed up to 25 °C and stirred overnight. The reaction was quenched with water and extracted with DCM (15 mL x 3). The combined organic extracts were dried over anhydrous Na_2SO_4, filtered and concentrated. The residue was used for the next step without further purification.



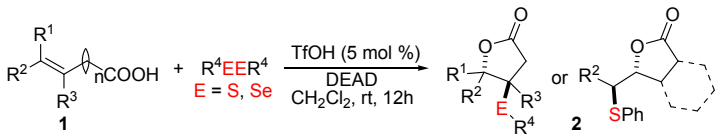
To an oven-dried 25 mL Schlenk flask equipped with a stir bar was added (*E*)-4-(4-fluorophenyl)but-3-en-1-yl methanesulfonate (0.81 g, 3.3 mmol) and $TsNH_2$ (0.85 g, 4.94 mmol) in 5 mL DMF. The flask was evacuated and back-filled with nitrogen for three times. K_2CO_3 (1.14 g, 8.2 mmol) was added to the resulting solution. The mixture was heated to 100 °C and stirred overnight. After the reaction was cooled to room temperature, water was added and the mixture was extracted with Et_2O (20

mL x 3). The combined organic extracts were washed with water (15 mL x 3) and saturated brine, dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (eluent: toluene/EA = 50/1 to 25/1, v/v) to afford **3g** (1.22 g, 62%) as a white solid.

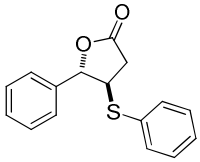


(*E*)-*N*-(4-(4-fluorophenyl)but-3-en-1-yl)-4-methylbenzenesulfonamide (3g): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.63 (m, 2H), 7.25 (dd, *J* = 8.9, 3.3 Hz, 2H), 7.23 – 7.18 (m, 2H), 7.03 – 6.78 (m, 2H), 6.30 (d, *J* = 15.9 Hz, 1H), 5.90 (dt, *J* = 15.8, 7.1 Hz, 1H), 5.17 – 4.97 (m, 1H), 3.06 (p, *J* = 6.5 Hz, 2H), 2.38 (d, *J* = 12.2 Hz, 3H), 2.34 (qd, *J* = 6.8, 1.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.23 (d, *J* = 246.5 Hz), 143.53, 136.97, 133.16 (t, *J* = 2.5 Hz), 131.91, 131.88, 129.81, 127.69 (d, *J* = 8.0 Hz), 125.53, 115.45 (d, *J* = 21.6 Hz), 42.67, 33.04, 21.61. ^{19}F NMR (377 MHz, $CDCl_3$) δ -114.83. HR-ESI-MS m/z calcd. for $C_{17}H_{19}O_2NFS$ $[M+H]^+$: 320.1115, found: 320.1110.

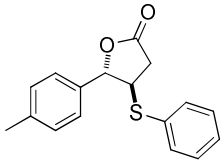
2.4 Synthesis of 4-(phenylthio)dihydrofuran-2(*3H*)-one analogues (2a-2q)



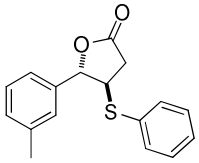
Typical procedure for the synthesis of 2a: To an oven-dried 20 mL vial equipped with a stir bar were added **1a** (25.2 mg, 0.15 mmol, 1.0 equiv), diphenyl disufide (19.8 mg, 0.09 mmol, 0.6 equiv), DCM (3.0 mL), DEAD (14.4 μL, 0.09 mmol, 0.6 equiv) and TfOH (100 μL 0.075 M solution in DCM, 0.0075 mmol, 5 mol %) successively. The solution was stirred at room temperature for 12 h and then concentrated under reduced pressure. The residue was directly purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford the desired product **2a** (40.1 mg, 99%) as a colorless oil.



5-Phenyl-4-(phenylthio)dihydrofuran-2(*3H*)-one (2a):[11] Prepared by the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2a** (40.1 mg, 99%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 6.98 (m, 10H), 5.31 (d, *J* = 5.8 Hz, 1H), 3.80 (dd, *J* = 13.9, 7.0 Hz, 1H), 2.99 (dd, *J* = 18.0, 8.2 Hz, 1H), 2.62 (dd, *J* = 18.0, 7.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.25, 137.40, 133.37, 133.30, 131.80, 129.44, 128.87, 128.83, 128.57, 125.50, 85.17, 76.84, 49.91, 35.45. HR-ESI-MS m/z calcd. for $C_{16}H_{15}O_2S$ $[M+H]^+$: 271.0787; found: 271.0786.

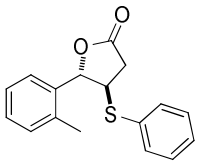


4-(Phenylthio)-5-(*p*-tolyl)dihydrofuran-2(*3H*)-one (2b):[11] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2b** (40.7 mg, 95%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 12.0 Hz, 2H), 7.32 (d, *J* = 2.4 Hz, 3H), 7.20 – 7.11 (m, 4H), 5.30 (d, *J* = 5.8 Hz, 1H), 3.79 (dd, *J* = 13.9, 7.0 Hz, 1H), 3.01 (dd, *J* = 18.0, 8.1 Hz, 1H), 2.63 (dd, *J* = 18.0, 7.0 Hz, 1H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.43, 138.90, 134.45, 133.51, 131.91, 129.58, 129.51, 128.63, 125.58, 85.30, 50.01, 35.65, 21.28. HR-ESI-MS m/z calcd. for $C_{17}H_{17}O_2S$ $[M+H]^+$: 285.0943; found: 285.0940.

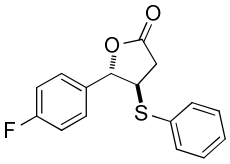


4-(Phenylthio)-5-(*m*-tolyl)dihydrofuran-2(*3H*)-one (2c):[11] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2c** (42.1 mg, 99%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.37 (m, 2H), 7.32 (d, *J* = 3.1 Hz, 3H), 7.28 – 7.19 (m, 1H), 7.19 – 7.10 (m, 1H), 7.04 (d, *J* = 5.8 Hz, 2H), 5.31 (d, *J* =

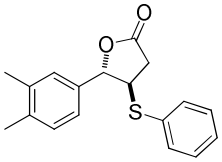
5.6 Hz, 1H), 3.92 – 3.65 (m, 1H), 3.01 (dd, *J* = 18.0, 8.2 Hz, 1H), 2.80 – 2.42 (m, 1H), 2.32 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.46, 138.73, 137.49, 133.52, 131.95, 129.68, 129.51, 128.81, 128.66, 126.13, 122.62, 85.42, 49.96, 35.52, 21.53. HR-ESI-MS m/z calcd. for $C_{17}H_{17}O_2S$ $[M+H]^+$: 285.0943; found: 285.0941.



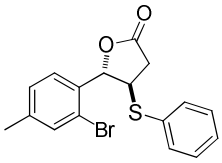
4-(Phenylthio)-5-(*o*-tolyl)dihydrofuran-2(3*H*)-one (2d): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2d** (38.9 mg, 91%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.35 (m, 2H), 7.30 (dd, *J* = 9.2, 6.0 Hz, 3H), 7.26 – 7.19 (m, 3H), 7.19 – 7.09 (m, 1H), 5.62 (d, *J* = 4.1 Hz, 1H), 3.92 – 3.68 (m, 1H), 3.00 (dd, *J* = 18.0, 7.8 Hz, 1H), 2.63 (dd, *J* = 18.0, 4.9 Hz, 1H), 2.24 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.94, 135.66, 135.26, 133.51, 132.08, 131.14, 129.50, 128.82, 128.72, 126.54, 124.62, 82.81, 49.16, 35.42, 19.21. HR-ESI-MS m/z calcd. for $C_{17}H_{17}O_2S$ $[M+H]^+$: 285.0943; found: 285.0940.



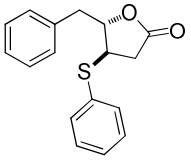
5-(4-Fluorophenyl)-4-(phenylthio)dihydrofuran-2(*3H*)-one (2e): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2e** (41.4 mg, 96%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.18 (m, 7H), 7.04 (t, *J* = 8.5 Hz, 2H), 5.29 (d, *J* = 6.4 Hz, 1H), 3.76 (dd, *J* = 14.8, 7.7 Hz, 1H), 3.02 (dd, *J* = 17.9, 8.2 Hz, 1H), 2.66 (dd, *J* = 17.9, 7.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.96, 163.05 (d, *J* = 248.2 Hz), 133.67, 133.26 (d, *J* = 3.1 Hz), 131.67, 129.62, 128.87, 127.64 (d, *J* = 8.3 Hz), 115.96 (d, *J* = 21.9 Hz), 84.88, 50.23, 35.80. ^{19}F NMR (377 MHz, $CDCl_3$) δ -112.52. HR-ESI-MS m/z calcd. for $C_{16}H_{14}O_2FS$ $[M+H]^+$: 289.0693; found: 289.0691.



5-(3,4-Dimethylphenyl)-4-(phenylthio)dihydrofuran-2(*3H*)-one (2f): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2f** (38.7 mg, 86%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.36 (m, 2H), 7.36 – 7.28 (m, 3H), 7.11 (d, *J* = 7.8 Hz, 1H), 6.97 (d, *J* = 7.0 Hz, 2H), 5.28 (d, *J* = 5.7 Hz, 1H), 3.81 (dd, *J* = 13.9, 6.8 Hz, 1H), 3.02 (dd, *J* = 18.0, 8.2 Hz, 1H), 2.63 (dd, *J* = 18.0, 6.8 Hz, 1H), 2.25 (s, 3H) 2.23 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 174.58, 137.55, 137.30, 134.89, 133.53, 131.99, 130.10, 129.50, 128.62, 126.77, 123.04, 85.41, 49.94, 35.62, 19.95, 19.64. HR-ESI-MS m/z calcd. for $C_{18}H_{19}O_2S$ $[M+H]^+$: 299.1100; found: 299.1094.

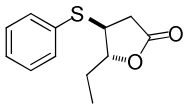


5-(2-Bromo-4-methylphenyl)-4-(phenylthio)dihydrofuran-2(*3H*)-one (2g): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2g** (40.7 mg, 74%) as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.43 (m, 2H), 7.41 (s, 1H), 7.36 – 7.28 (m, 3H), 7.18 – 7.08 (m, 2H), 5.69 (d, *J* = 3.4 Hz, 1H), 3.92 (dt, *J* = 7.5, 3.8 Hz, 1H), 2.90 (dd, *J* = 18.0, 7.6 Hz, 1H), 2.73 – 2.47 (m, 1H), 2.32 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.04, 140.84, 134.09, 134.07, 133.66, 131.89, 129.43, 128.79, 128.66, 126.59, 121.63, 84.25, 49.03, 34.94, 20.86. HR-ESI-MS m/z calcd. for $C_{17}H_{16}O_2BrS$ $[M+H]^+$: 363.0048; found: 363.0041.

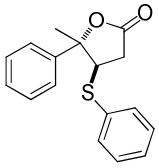


5-Benzyl-4-(phenylthio)dihydrofuran-2(*3H*)-one (2h): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2h** (32.7 mg, 76%) as a colorless oil. ^{1}H NMR

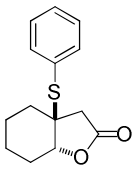
(400 MHz, $CDCl_3$) δ 7.44 – 7.20 (m, 8H), 7.13 (d, *J* = 6.6 Hz, 2H), 4.59 (dd, *J* = 11.0, 5.6 Hz, 1H), 3.60 (dd, *J* = 14.1, 7.1 Hz, 1H), 3.05 (dd, *J* = 14.4, 4.8 Hz, 1H), 2.92 (dd, J = 14.4, 6.2 Hz, 1H), 2.71 (dd, *J* = 18.0, 8.3 Hz, 1H), 2.47 (dd, *J* = 18.0, 7.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.24, 135.31, 133.44, 131.81, 129.71, 129.56, 128.82, 128.64, 127.26, 84.89, 45.26, 39.50, 35.69. HR-ESI-MS m/z calcd. for $C_{17}H_{17}O_2S$ $[M+H]^+$: 285.0943; found: 285.0944.



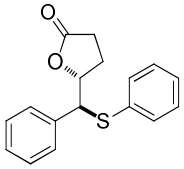
5-Ethyl-4-(phenylthio)dihydrofuran-2(*3H*)-one (2i):[12] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2i** (33.1 mg, 99%) as a colorless oil. Known compound. ^1H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 3.9 Hz, 2H), 7.35 (d, *J* = 5.0 Hz, 3H), 4.28 (dd, *J* = 12.0, 6.2 Hz, 1H), 3.57 (dd, *J* = 14.4, 7.4 Hz, 1H), 2.93 (dd, *J* = 18.0, 8.3 Hz, 1H), 2.57 (dd, *J* = 18.0, 7.4 Hz, 1H), 1.78 (tt, *J* = 14.4, 7.3 Hz, 1H), 1.65 (dp, *J* = 15.0, 7.4 Hz, 1H), 0.99 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.48, 133.40, 132.13, 129.54, 128.63, 86.33, 46.30, 36.21, 27.23, 9.78. HR-ESI-MS m/z calcd. for $C_{12}H_{15}O_2S$ $[M+H]^+$: 223.0787; found: 223.0786.



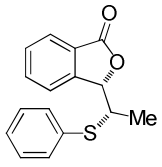
5-Methyl-5-phenyl-4-(phenylthio)dihydrofuran-2(*3H*)-one (2j): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2j** (29.4 mg, 69%) as a colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 7.3 Hz, 2H), 7.40 – 7.32 (m, 4H), 7.27 (t, *J* = 6.3 Hz, 4H), 4.04 (t, *J* = 7.5 Hz, 1H), 2.90 (dd, *J* = 17.8, 7.8 Hz, 1H), 2.72 (dd, *J* = 17.8, 7.2 Hz, 1H), 1.82 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.86, 143.41, 133.28, 132.23, 129.49, 128.80, 128.21, 128.08, 124.31, 88.74, 54.10, 37.41, 24.47. HR-ESI-MS m/z calcd. for $C_{17}H_{17}O_2S$ $[M+H]^+$: 285.0943; found: 285.0939.



3a-(Phenylthio)hexahydrobenzofuran-2(*3H*)-one (2k):[13] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2k** (28.4 mg, 76%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.47 (m, 2H), 7.46 – 7.34 (m, 3H), 4.31 (t, *J* = 3.6 Hz, 1H), 2.70 (d, *J* = 16.7 Hz, 1H), 2.43 (d, *J* = 16.7 Hz, 1H), 2.02 (ddd, *J* = 9.2, 7.1, 3.1 Hz, 2H), 1.79 (dd, *J* = 16.3, 11.9 Hz, 1H), 1.74 – 1.62 (m, 2H), 1.59 – 1.44 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.74, 137.49, 129.99, 129.63, 129.48, 81.05, 51.66, 44.42, 32.56, 25.19, 20.92, 19.69. HR-ESI-MS m/z calcd. for $C_{14}H_{17}O_2S$ $[M+H]^+$: 249.0943; found: 249.0940.

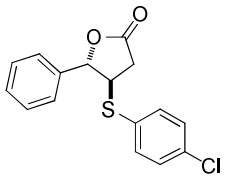


5-(Phenyl(phenylthio)methyl)dihydrofuran-2(*3H*)-one (2l):[14] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2l** (42.5 mg, 99%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.26 (m, 7H), 7.27 – 7.19 (m, 3H), 4.89 (q, *J* = 6.3 Hz, 1H), 4.27 (d, *J* = 5.7 Hz, 1H), 2.51 – 2.31 (m, 2H), 2.30 – 2.17 (m, 1H), 2.17 – 2.01 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.62, 137.46, 133.69, 133.10, 129.13, 128.92, 128.78, 128.13, 128.00, 81.49, 58.30, 28.58, 26.04. HR-ESI-MS m/z calcd. for $C_{17}H_{17}O_2S$ $[M+H]^+$: 285.0943; found: 285.0939.

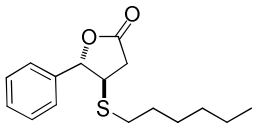


3-(1-(Phenylthio)ethyl)isobenzofuran-1(*3H*)-one (2m): Substrate **1m** (*Z*/*E*=1.6:1) was used. Prepared based on the typical procedure and the crude NMR of reaction mixture showed that diastereomeric products were formed in 82% yield with 2.3:1 dr. The mixture was purified by flash silica gel column chromatography (eluent: PE/EA =

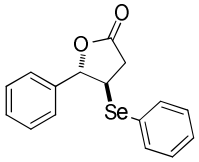
50/1 to 25/1, v/v) to afford major product **2m** (24.2 mg, 59%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 7.6 Hz, 1H), 7.83 (d, *J* = 7.7 Hz, 1H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.46 – 7.41 (m, 2H), 7.35 – 7.23 (m, 3H), 5.56 (d, *J* = 3.1 Hz, 1H), 3.92 (qd, *J* = 7.0, 3.2 Hz, 1H), 1.15 (t, *J* = 32.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.34, 146.78, 133.79, 132.24, 129.60, 129.39, 127.86, 127.24, 125.74, 123.62, 81.71, 45.75, 14.89. HR-ESI-MS m/z calcd. for $C_{16}H_{15}O_2S$ $[M]^+$: 271.0787; found: 271.0784.



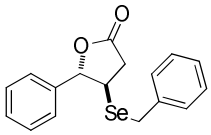
4-((4-Chlorophenyl)thio)-5-phenyldihydrofuran-2(*3H*)-one (2n): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2n** (36.0 mg, 78%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 5.0 Hz, 3H), 7.32 – 7.20 (m, 6H), 5.31 (d, *J* = 5.8 Hz, 1H), 3.79 (dd, *J* = 14.0, 7.0 Hz, 1H), 3.02 (dd, *J* = 17.9, 8.1 Hz, 1H), 2.63 (dd, *J* = 17.9, 7.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.01, 137.27, 135.00, 134.58, 130.51, 129.70, 129.11, 129.01, 125.62, 85.40, 50.26, 35.56. HR-ESI-MS m/z calcd. for $C_{16}H_{14}O_2ClS$ $[M+H]^+$: 305.0397; found: 305.0392.



4-(Hexylthio)-5-phenyldihydrofuran-2(*3H*)-one (2o): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2o** (36.7 mg, 87%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.25 (m, 5H), 5.25 (d, *J* = 7.3 Hz, 1H), 3.42 (dd, *J* = 16.4, 8.2 Hz, 1H), 3.04 (dd, *J* = 17.7, 8.2 Hz, 1H), 2.64 (dt, *J* = 21.1, 10.6 Hz, 1H), 2.47 – 2.38 (m, 2H), 1.53 – 1.38 (m, 2H), 1.35 – 1.12 (m, 6H), 0.86 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.44, 137.62, 129.12, 128.91, 125.99, 86.88, 47.27, 36.99, 31.85, 31.32, 29.49, 28.42, 22.53, 14.07. HR-ESI-MS m/z calcd. for $C_{16}H_{23}O_2S$ $[M+H]^+$: 279.1413; found: 279.1406.



5-Phenyl-4-(phenylselanyl)dihydrofuran-2(*3H*)-one (2p):[15a] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 25/1, v/v) to afford **2p** (46.9 mg, 99%) as a yellow oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.5 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 4H), 7.28 (dd, *J* = 16.8, 9.5 Hz, 4H), 5.37 (d, *J* = 6.8 Hz, 1H), 3.86 – 3.61 (m, 1H), 3.02 (dd, *J* = 18.0, 8.3 Hz, 1H), 2.66 (dd, *J* = 18.0, 8.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.63, 137.37, 136.18, 129.66, 129.15, 128.99, 128.87, 126.12, 125.84, 86.25, 42.32, 36.06. HR-ESI-MS m/z calcd. for $C_{16}H_{15}O_2Se$ $[M+H]^+$: 319.0231; found: 319.0226.

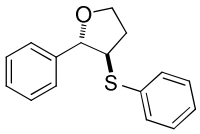


4-(Benzylselanyl)-5-phenyldihydrofuran-2(*3H*)-one (2q): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1 to 30/1, v/v) to afford **2q** (49.3 mg, 99%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (dd, *J* = 12.0, 9.2 Hz, 5H), 7.29 – 7.16 (m, 3H), 7.04 (d, *J* = 7.1 Hz, 2H), 5.34 (d, *J* = 8.3 Hz, 1H), 3.66 (q, *J* = 12.0 Hz, 2H), 3.29 (dd, *J* = 18.0, 8.8 Hz, 1H), 2.84 (dd, *J* = 17.8, 8.3 Hz, 1H), 2.60 (dd, *J* = 17.8, 10.3 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.59, 137.83, 137.38, 129.24, 128.93, 128.89, 128.77, 127.35, 126.26, 87.71, 38.50, 37.19, 28.12. HR-ESI-MS m/z calcd. for $C_{17}H_{17}O_2Se$ $[M+H]^+$: 333.0388; found: 333.0382.

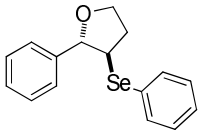
2.5 Synthesis of 3-(phenylthio)tetrahydrofuran analogues(4a-4d)

Typical procedure for the synthesis of 4a: To an oven-dried 20 mL vial equipped with a stir bar were added **3a** (22.2 mg, 0.15 mmol, 1.0 equiv), diphenyl disufide

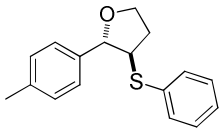
(19.8 mg, 0.09 mmol, 0.6 equiv), DCM (3.0 mL), DEAD (14.4 μL, 0.09 mmol, 0.6 equiv) and TfOH (100 μL 0.15 M solution in DCM, 0.015 mmol, 10 mol %) successively. The solution was stirred at room temperature for 12 h and then concentrated under reduced pressure. The residue was directly purified by flash silica gel column chromatography (eluent: PE/EA = 100/1 to 50/1, v/v) to afford the desired product **4a** (34.0 mg, 88%) as a colorless oil.



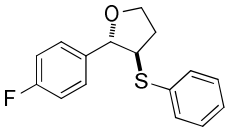
2-Phenyl-3-(phenylthio)tetrahydrofuran (4a): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 100/1 to 50/1, v/v) to afford **4a** (34.0 mg, 88%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.28 (m, 6H), 7.22 (dt, *J* = 14.5, 6.4 Hz, 4H), 4.77 (d, *J* = 5.7 Hz, 1H), 4.25 – 4.16 (m, 1H), 4.08 (q, *J* = 7.8 Hz, 1H), 3.61 (dd, *J* = 12.7, 5.7 Hz, 1H), 2.45 (dq, *J* = 15.4, 7.7 Hz, 1H), 2.07 (td, *J* = 12.0, 5.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.41, 134.87, 131.61, 129.08, 128.49, 127.83, 127.13, 125.91, 85.73, 68.00, 53.71, 33.9. HR-ESI-MS m/z calcd. for $C_{16}H_{17}OS$ $[M+H]^+$: 257.0994; found: 257.0994.



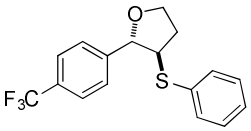
2-Phenyl-3-(phenylselanyl)tetrahydrofuran (4a'):[15b] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 50/1, v/v) to afford **4a'** (33.6 mg, 74%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (t, *J* = 8.7 Hz, 2H), 7.35 – 7.16 (m, 8H), 4.83 (t, *J* = 5.7 Hz, 1H), 4.15 (td, *J* = 7.9, 5.8 Hz, 1H), 4.03 (q, *J* = 7.7 Hz, 1H), 3.61 – 3.51 (m, 1H), 2.46 (dq, *J* = 14.8, 7.4 Hz, 1H), 2.18 – 2.05 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.40, 134.85, 129.20, 128.78, 128.47, 127.90, 127.81, 126.05, 86.23, 68.06, 47.78, 34.31.



3-(Phenylthio)-2-(*p*-tolyl)tetrahydrofuran (4b): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 100/1 to 50/1, v/v) to afford **4b** (25.1 mg, 62%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 7.3 Hz, 2H), 7.29 – 7.16 (m, 5H), 7.11 (d, *J* = 7.8 Hz, 2H), 4.74 (d, *J* = 5.8 Hz, 1H), 4.19 (td, *J* = 8.0, 4.7 Hz, 1H), 4.06 (dd, *J* = 15.6, 7.8 Hz, 1H), 3.59 (dd, *J* = 13.0, 5.6 Hz, 1H), 2.43 (dt, *J* = 15.4, 7.7 Hz, 1H), 2.32 (s, 3H), 2.06 (td, *J* = 12.0, 4.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.38, 137.51, 134.98, 131.56, 129.18, 129.06, 127.06, 125.88, 85.59, 67.92, 53.66, 34.01, 21.28. HR-ESI-MS m/z calcd. for $C_{17}H_{19}OS$ $[M+H]^+$: 271.1151; found: 271.1149.

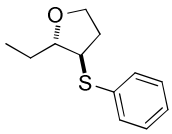


2-(4-Fluorophenyl)-3-(phenylthio)tetrahydrofuran (4c): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 100/1 to 50/1, v/v) to afford **4c** (38.2 mg, 93%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, *J* = 7.2 Hz, 2H), 7.23 (dd, *J* = 16.5, 9.3 Hz, 5H), 6.98 (t, *J* = 7.8 Hz, 2H), 4.71 (d, *J* = 5.8 Hz, 1H), 4.18 (dd, *J* = 12.9, 6.2 Hz, 1H), 4.13 – 4.00 (m, 1H), 3.54 (dd, *J* = 12.0, 5.9 Hz, 1H), 2.62 – 2.23 (m, 1H), 2.08 (td, *J* = 10.7, 5.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.44 (d, *J* = 245.7 Hz), 136.99 (d, *J* = 3.0 Hz), 134.59, 131.73, 129.10, 127.67 (d, *J* = 8.1 Hz), 127.27, 115.32 (d, *J* = 21.5 Hz), 85.33, 67.92, 53.73, 34.03. ^{19}F NMR (377 MHz, $CDCl_3$) δ -114.87. HR-ESI-MS m/z calcd. for $C_{16}H_{16}OFS$ $[M+H]^+$: 275.0900; found: 275.0901.



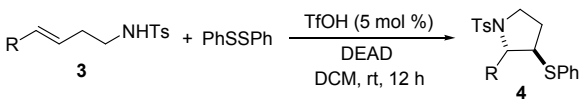
3-(Phenylthio)-2-(4-(trifluoromethyl)phenyl)tetrahydrofuran (4d): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 100/1 to 50/1, v/v) to afford **4d** (48.6 mg, 98%) as a colorless oil. ^{1}H

NMR (400 MHz, $CDCl_3$) δ 7.54 (d, J = 8.1 Hz, 2H), 7.40 (d, J = 8.0 Hz, 2H), 7.34 (d, J = 6.6 Hz, 2H), 7.29 – 7.18 (m, 3H), 4.80 (d, J = 6.0 Hz, 1H), 4.26 – 4.16 (m, 1H), 4.10 (q, J = 7.7 Hz, 1H), 3.55 (dd, J = 13.2, 5.9 Hz, 1H), 2.44 (dq, J = 15.2, 7.6 Hz, 1H), 2.09 (td, J = 12.4, 5.3 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 145.49, 134.30, 131.98, 129.98 (q, J = 32.3 Hz), 129.19, 127.51, 126.19, 125.41 (q, J = 3.7 Hz), 124.24 (q, J = 272.1 Hz), 85.21, 68.18, 53.93, 33.93. ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.53. HRMS (DART POS) m/z calcd. for $C_{17}H_{16}F_3OS$ $[M+H]^+$:325.0868, Found: 325.0865.



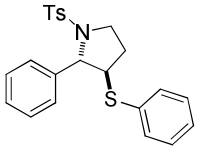
2-Ethyl-3-(phenylthio)tetrahydrofuran (4e): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 100/1 to 50/1, v/v) to afford **4e** (29.4 mg, 94%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.37 (m, 2H), 7.30 (dd, J = 10.2, 4.8 Hz, 2H), 7.27 – 7.20 (m, 1H), 3.93 (td, J = 8.1, 4.7 Hz, 1H), 3.85 (dd, J = 15.7, 7.7 Hz, 1H), 3.75 – 3.53 (m, 1H), 3.46 – 3.22 (m, 1H), 2.34 (dq, J = 12.9, 7.8 Hz, 1H), 1.96 (ddt, J = 12.3, 7.1, 4.9 Hz, 1H), 1.69 – 1.57 (m, 1H), 1.51 (dt, J = 21.3, 7.3 Hz, 1H), 0.95 (t, J = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 135.42, 131.37, 129.10, 127.00, 85.58, 66.86, 49.73, 34.45, 27.54, 10.42. HRMS (DART POS) m/z calcd. for $C_{12}H_{17}OS$ $[M+H]^+$: 209.0995, Found: 209.0994.

2.6 Synthesis of 3-(phenylthio)-1-tosylpyrrolidine analogues (4f-4i)

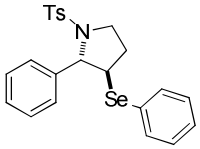


Typical procedure for the synthesis of 4f: To an oven-dried 20 mL vial equipped with a stir bar were added **3f** (45.2 mg, 0.15 mmol, 1.0 equiv), diphenyl disufide (19.8 mg, 0.09 mmol, 0.6 equiv), DCM (3.0 mL), DEAD (14.4 μL, 0.09 mmol, 0.6 equiv) and TfOH (100 μL 0.075 M solution in DCM, 0.0075 mmol, 5 mol %) successively. The solution was stirred at room temperature for 12 h and then concentrated under

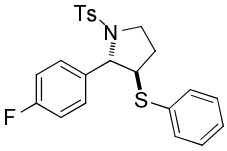
reduced pressure. The residue was directly purified by flash silica gel column chromatography (eluent: PE/EA = 30/1 to 15/1, v/v) to afford the desired product **4f** (58.4 mg, 95%) as a white solid.



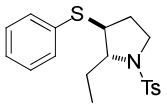
2-Phenyl-3-(phenylthio)-1-tosylpyrrolidine (4f):[16] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 30/1 to 15/1, v/v) to afford **6a** (58.4 mg, 95%) as a white solid. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.8 Hz, 2H), 7.41 – 7.01 (m, 12H), 4.64 (s, 1H), 3.78 (t, *J* = 7.9 Hz, 1H), 3.60 (dd, *J* = 17.9, 7.8 Hz, 2H), 2.45 (s, 3H), 2.36 – 2.21 (m, 1H), 1.83 – 1.70 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.51, 141.68, 134.56, 133.58, 132.30, 129.58, 129.13, 128.51, 127.86, 127.71, 127.52, 126.04, 68.75, 55.55, 47.93, 29.26, 21.65. HR-ESI-MS m/z calcd. for $C_{23}H_{24}O_2NS_2$ $[M+H]^+$: 410.1243; found: 410.1239.



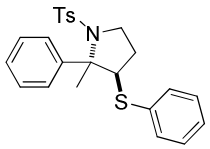
2-Phenyl-3-(phenylselanyl)-1-tosylpyrrolidine (**4f'**): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 30/1 to 15/1, v/v) to afford **4f'** (65.6 mg, 96%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.2 Hz, 2H), 7.42 – 7.11 (m, 12H), 4.68 (d, *J* = 2.5 Hz, 1H), 3.76 (ddd, J = 10.5, 7.5, 3.1 Hz, 1H), 3.69 – 3.53 (m, 2H), 2.44 (s, 3H), 2.38 – 2.23 (m, 1H), 1.77 (ddt, *J* = 13.3, 6.5, 3.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.56, 142.01, 135.01, 134.53, 129.63, 129.30, 128.51, 128.30, 128.25, 127.85, 127.51, 126.11, 69.35, 49.69, 48.34, 30.19, 21.69. HR-ESI-MS m/z calcd. for $C_{23}H_{24}O_2NSSe[M+H]^+$: 458.0687; found: 458.0689.



2-(4-Fluorophenyl)-3-(phenylthio)-1-tosylpyrrolidine (4g): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 30/1 to 15/1, v/v) to afford **4g** (55.6 mg, 86%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.25 (t, *J* = 4.5 Hz, 3H), 7.15 (dt, *J* = 7.2, 5.1 Hz, 4H), 6.95 (t, *J* = 8.5 Hz, 2H), 4.57 (s, 1H), 3.86 – 3.69 (m, 1H), 3.59 (dt, *J* = 16.3, 8.3 Hz, 1H), 3.54 – 3.48 (m, 1H), 2.44 (s, 3H), 2.26 (dt, *J* = 13.7, 7.6 Hz, 1H), 1.86 – 1.61 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.17 (d, *J* = 245.9 Hz), 143.71, 137.51 (d, *J* = 2.9 Hz), 134.40, 133.40, 132.50, 129.66, 129.21, 127.89, 127.82, 127.74, 115.39 (d, *J* = 21.6 Hz), 68.24, 55.73, 48.01, 29.39, 21.68. ^{19}F NMR (377 MHz, $CDCl_3$) δ -115.14. HR-ESI-MS m/z calcd. for $C_{23}H_{23}O_2NFS_2$ $[M+H]^+$: 428.1148; found: 428.1142.



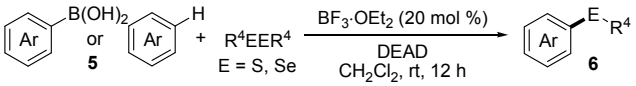
2-Ethyl-3-(phenylthio)-1-tosylpyrrolidine (4h): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 30/1 to 15/1, v/v) to afford **4h** (49.6 mg, 91%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.30 – 7.21 (m, 3H), 7.15 (dd, *J* = 12.5, 11.2 Hz, 2H), 3.66 – 3.47 (m, 2H), 3.49 – 3.25 (m, 2H), 2.47 (s, 3H), 2.34 – 2.12 (m, 1H), 2.01 – 1.82 (m, 1H), 1.65 (m, 2H), 0.88 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.43, 134.13, 133.92, 131.91, 129.58, 129.00, 127.93, 127.43, 67.27, 49.81, 47.46, 30.09, 29.45, 21.66, 10.24. HR-ESI-MS m/z calcd. for $C_{19}H_{24}O_2NS_2$ $[M+H]^+$: 362.1243; found: 362.1237.



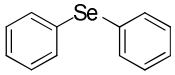
2-Methyl-2-phenyl-3-(phenylthio)-1-tosylpyrrolidine (4i): Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent:

PE/EA = 30/1 to 15/1, v/v) to afford **4i** (41.8 mg, 65%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (t, *J* = 7.3 Hz, 2H), 7.36 (t, *J* = 10.0 Hz, 2H), 7.31 – 7.17 (m, 5H), 7.15 – 7.08 (m, 3H), 7.04 (dd, *J* = 6.1, 2.7 Hz, 2H), 3.78 (td, *J* = 8.6, 5.5 Hz, 1H), 3.74 – 3.68 (m, 1H), 3.61 (dt, *J* = 12.3, 6.1 Hz, 1H), 2.41 (s, 3H), 2.23 – 2.10 (m, 1H), 2.02 – 1.85 (m, 1H), 1.92 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.97, 142.92, 138.31, 134.48, 131.89, 129.38, 129.00, 128.24, 127.27, 127.20, 126.22,125.91, 72.40, 62.60, 47.41, 29.95, 21.96, 21.62. HR-ESI-MS m/z calcd. for $C_{24}H_{26}O_2NS_2$ $[M+H]^+$: 424.1399; found: 424.1394.

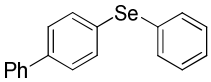
2.7 Synthesis of diaryl selenides (6a-6p)



Typical procedure for the synthesis of 6a: To an oven-dried 20 mL vial equipped with a stir bar were added phenylboronic acid (18.6 mg, 0.15 mmol, 1.0 equiv), diphenyl diselenide (28.2 mg, 0.09 mmol, 0.6 equiv), DCM (3.0 mL), DEAD (14.4 μL, 0.09 mmol, 0.6 equiv) and $BF_3{\cdot}Et_2O$ (3.8 μL, 0.03 mmol, 20 mol %) successively. The solution was stirred at room temperature for 12 h and then concentrated under reduced pressure. The residue was directly purified by flash silica gel column chromatography (eluent: PE) to afford the desired product **6a** (32.9 mg, 94%) as a yellow oil.

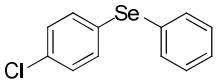


Diphenylselane (6a):[17] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6a** (32.9 mg, 94%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.42 (m, 4H), 7.32 – 7.21 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 133.11, 131.26, 129.46, 127.46.

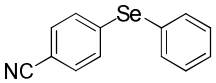


[1,1'-Biphenyl]-4-yl(phenyl)selane (6b):[18] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6b** (38.5 mg, 83%) as a white solid. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d,

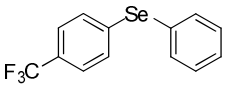
J = 7.3 Hz, 2H), 7.54 – 7.46 (m, 6H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.33 (t, *J* = 7.3 Hz, 1H), 7.31 – 7.24 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 140.49, 140.41, 133.40, 133.21, 131.18, 130.36, 129.52, 128.96, 128.13, 127.62, 127.55, 127.10.



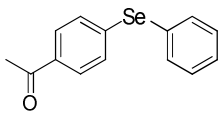
(4-Chlorophenyl)(phenyl)selane (6c):[17] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6c** (40.1 mg, 97%) as a white solid. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.43 (m, 2H), 7.39 – 7.33 (m, 2H), 7.28 (dd, *J* = 3.9, 2.4 Hz, 3H), 7.25 – 7.19 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 134.24, 133.63, 133.31, 130.77, 129.69, 129.60, 127.80.



4-(Phenylselanyl)benzonitrile (6d):[18] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6d** (38.1 mg, 98%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (dd, *J* = 7.9, 1.5 Hz, 2H), 7.48 – 7.35 (m, 5H), 7.33 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.05, 135.77, 132.49, 130.33, 130.06, 129.25, 127.60, 118.89, 109.73.

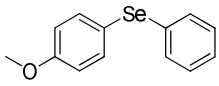


Phenyl(4-(trifluoromethyl)phenyl)selane (6e):[18] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6e** (44.2 mg, 98%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, *J* = 7.5, 1.8 Hz, 2H), 7.44 (q, *J* = 8.6 Hz, 4H), 7.38 – 7.31 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.93, 134.99, 131.14, 129.87, 129.06, 128.85, 128.68, 126.04 (q, *J* = 3.6 Hz), 124.24 (d, *J* = 271.9 Hz). ^{19}F NMR (377 MHz, $CDCl_3$) δ -62.61.

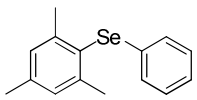


1-(4-(Phenylselanyl)phenyl)ethanone (6f):[18] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 100/1, v/v) to afford **6f** (34.4 mg, 83%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.5 Hz, 2H), 7.62 – 7.55 (m, 2H), 7.43 – 7.31 (m,

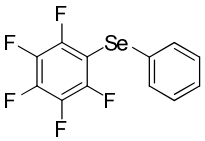
5H), 2.55 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.45, 140.43, 135.28, 135.24, 130.41, 129.86, 129.04, 128.75, 128.58, 26.61.



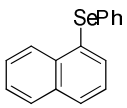
(4-Methoxyphenyl)(phenyl)selane (6g):[17] Prepared based on the typical procedure using *4-MeOC$_6$H$_4$B(OH)$_2$* (22.8 mg, 0.15 mmol, 1.0 equiv) and *Ph$_2$Se$_2$* (28.1 mg, 0.09 mmol, 0.6 equiv) and purified by flash silica gel column chromatography (eluent: PE/EA = 100/1, v/v) to afford **6g** (36.9 mg, 93%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.46 (m, 2H), 7.36 – 7.29 (m, 2H), 7.24 – 7.15 (m, 3H), 6.89 – 6.80 (m, 2H), 3.80 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.91, 136.67, 133.33, 131.02, 129.28, 126.58, 120.06, 115.26, 55.43.



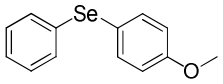
Mesityl(phenyl)selane (6h):[18] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6h** (39.4 mg, 95%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 – 7.04 (m, 5H), 6.99 (s, 2H), 2.43 (s, 6H), 2.30 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 143.78, 139.22, 133.60, 129.25, 128.99, 128.56, 126.89, 125.48, 24.41, 21.21.



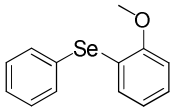
(Perfluorophenyl)(phenyl)selane (6i):[19] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6i** (37.3 mg, 77%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, J = 6.6 Hz, 2H), 7.33 – 7.23 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.69 – 147.30 (m), 146.33 – 145.67 (m), 143.79 – 142.45 (m), 141.61 – 140.06 (m), 139.37 – 138.57 (m), 136.79 – 136.21 (m), 133.58, 129.66, 128.68, 103.98 – 103.28 (m). ^{19}F NMR (377 MHz, $CDCl_3$) δ -126.05 (dd, J = 26.4, 9.5 Hz, 2F), -151.61 (t, J = 22.4 Hz, 1F), -159.91 – -160.37 (m, 2F).



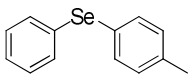
Naphthalen-1-yl(phenyl)selane (6j):[20] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6j** (36.3 mg, 85%) as a yellow oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 – 8.26 (m, 1H), 7.83 (dd, *J* = 4.8, 3.1 Hz, 2H), 7.76 (dd, *J* = 7.1, 0.9 Hz, 1H), 7.54 – 7.45 (m, 2H), 7.35 (m, *J* = 7.7, 5.7, 5.3 Hz, 3H), 7.25 – 7.11 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 134.26, 134.21, 133.96, 131.83, 131.81, 129.50, 129.42, 129.31, 128.70, 127.77, 127.06, 126.92, 126.49, 126.14.



(4-Methoxyphenyl)(phenyl)selane (6g):[17] Prepared based on the typical procedure using *PhB(OH)$_2$* (18.3 mg, 0.15 mmol, 1.0 equiv) and *(4-MeOC$_6$H$_4$)$_2$Se$_2$* (33.5 mg, 0.09 mmol, 0.6 equiv) and purified by flash silica gel column chromatography (eluent: PE/EA = 100/1, v/v) to afford **6g** (34.0 mg, 86%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.7 Hz, 2H), 7.36 – 7.29 (m, 3H), 7.25 – 7.14 (m, 2H), 6.86 (t, *J* = 8.0 Hz, 2H), 3.80 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.90, 136.66, 134.70, 133.32, 131.02, 129.27, 126.57, 115.26, 55.42.

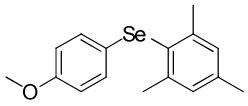


(2-Methoxyphenyl)(phenyl)selane (6k):[21] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE/EA = 200/1, v/v) to afford **6k** (22.9 mg, 58%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.52 (m, 2H), 7.38 – 7.29 (m, 3H), 7.22 – 7.14 (m, 1H), 6.95 (dd, *J* = 7.7, 1.4 Hz, 1H), 6.85 (d, *J* = 8.1 Hz, 1H), 6.79 (dd, *J* = 10.9, 4.1 Hz, 1H), 3.89 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.72, 135.58, 130.92, 129.58, 128.38, 128.24, 127.83, 122.04, 121.75, 110.51, 56.00.

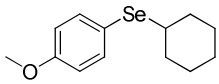


Phenyl(p-tolyl)selane (6l):[17] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6l** (34.9 mg, 94%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (dd, *J* = 12.3, 10.3 Hz, 4H), 7.28 – 7.19 (m, 3H), 7.08 (t, *J* = 10.3 Hz, 2H), 2.33 (s, 3H). ^{13}C NMR

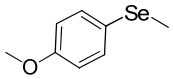
(101 MHz, $CDCl_3$) δ 137.80, 134.02, 132.23, 132.19, 130.33, 129.35, 127.00, 126.92, 21.29.



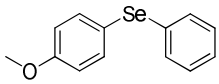
Mesityl(4-methoxyphenyl)selane (6m):[21] Prepared based on the typical procedure and purified by preparative TLC (eluent: PE) to afford **6m** (27.2 mg, 59%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.06 (d, 2H), 6.96 (s, 2H), 6.73 (d, J = 5.8 Hz, 2H), 3.76 (s, 3H), 2.44 (s, 6H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.23, 143.42, 138.87, 130.85, 128.91, 127.99, 123.37, 115.06, 55.38, 24.46, 21.16.



Cyclohexyl(4-methoxyphenyl)selane (6n):[22] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6n** (25.5 mg, 71%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, J = 8.6 Hz, 2H), 6.81 (d, J = 8.6 Hz, 2H), 3.79 (s, 3H), 3.10 (tt, J = 10.8, 3.7 Hz, 1H), 1.99 (dd, J = 21.5, 10.9 Hz, 2H), 1.80 – 1.64 (m, 2H), 1.43 (dt, J = 13.0, 10.4 Hz, 2H), 1.34 – 1.14 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.59, 137.51, 119.12, 114.59, 55.35, 43.68, 34.31, 27.04, 25.87.

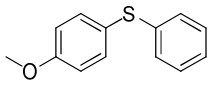


(4-Methoxyphenyl)(methyl)selane (6o):[23] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford **6o** (21.5 mg, 83%) as a yellow oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, 2H), 6.82 (d, 2H), 3.79 (s, 3H), 2.30 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 158.94, 133.58, 121.65, 114.93, 55.43, 8.84.



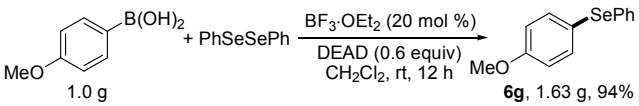
(4-Methoxyphenyl)(phenyl)selane (6g):[17] Prepared based on the typical procedure using anisole and PhSeSePh, and purified by preparative thin layer chromatography to afford **6g** (23.5 mg, 59%) as a colorless oil. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, 2H), 7.37 – 7.29 (m, 2H), 7.24 – 7.15 (m, 3H), 6.85 (d, 2H), 3.80 (s,

3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.91, 136.66, 133.32, 131.03, 129.27, 126.58, 120.07, 115.26, 55.43.



(4-Methoxyphenyl)(phenyl)sulfane (6p):[24] Prepared based on the typical procedure and purified by flash silica gel column chromatography (eluent: PE) to afford as a colorless oil. When 4-methoxyphenylboronic acid reacted with PhSSPh, **6p** (18.7 mg, 57%) was obtained. When anisole reacted with PhSSPh, **6p** (23.3 mg, 72%) was obtained. Known compound. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, 2H), 7.27 – 7.19 (m, 2H), 7.19 – 7.10 (m, 3H), 6.89 (d, 2H), 3.81 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.95, 138.72, 135.48, 129.05, 128.34, 125.88, 124.45, 115.11, 55.49.

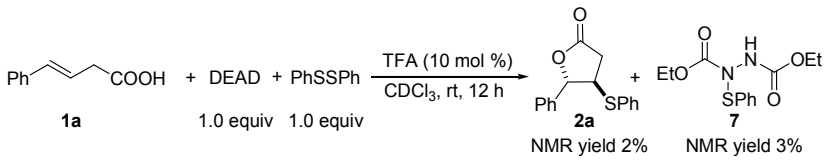
3. Procedure of the gram-scale reaction



To an oven-dried 50 mL vial equipped with a stir bar were added 4-methoxyphenylboronic acid (1.00 g, 6.6 mmol, 1.0 equiv), diphenyl diselenide (1.23 g, 3.9 mmol, 0.6 equiv), DCM (15.0 mL), DEAD (634 μL, 3.9 mmol, 0.6 equiv) and $BF_3 \cdot Et_2O$ (172 μL, 1.3 mmol, 20 mol %) successively. The solution was stirred at room temperature for 12 h and then concentrated under reduced pressure. The residue was directly purified by flash silica gel column chromatography (eluent: PE/EA = 100/1, v/v) to afford the desired product **6g** (1.63 g, 94%) as a colorless oil.

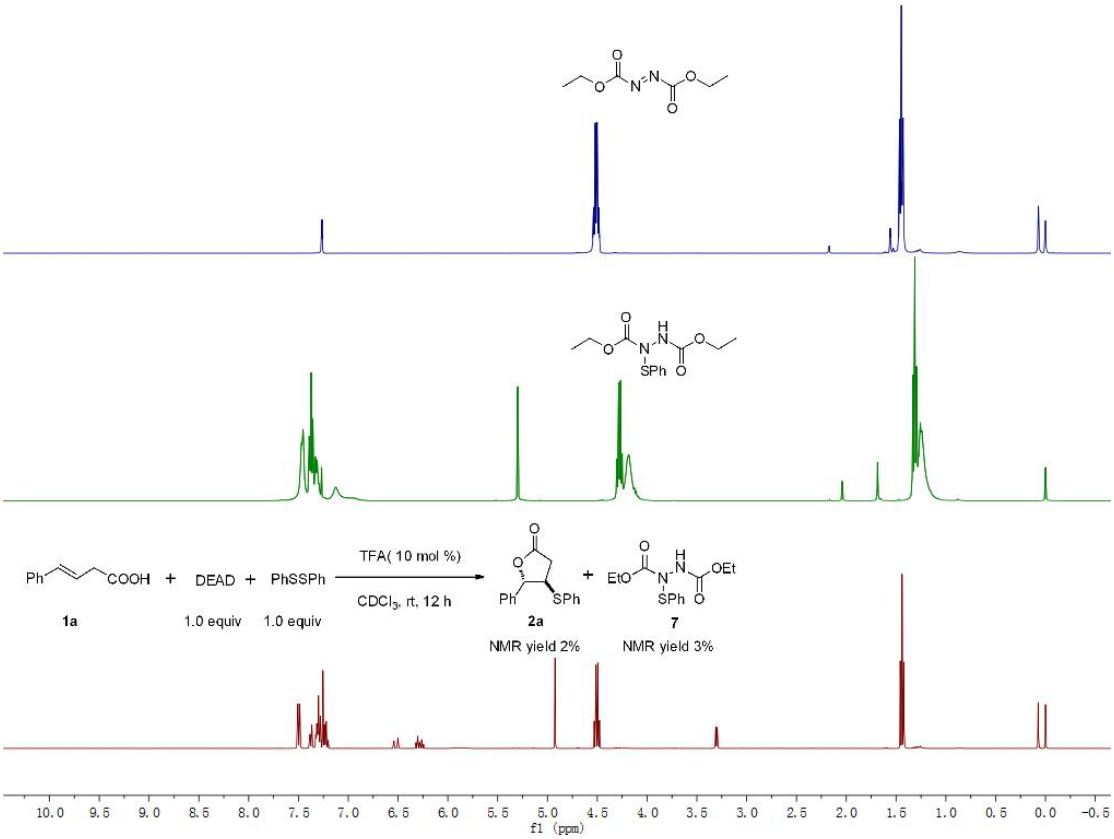
4. Mechanistic Studies

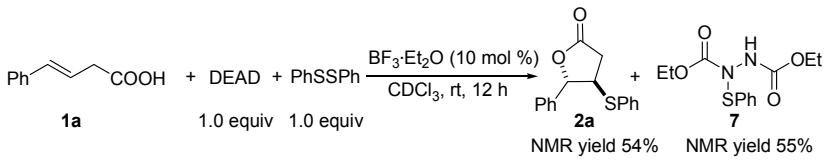
4.1 Oxidative Cleavage of S-S Bond



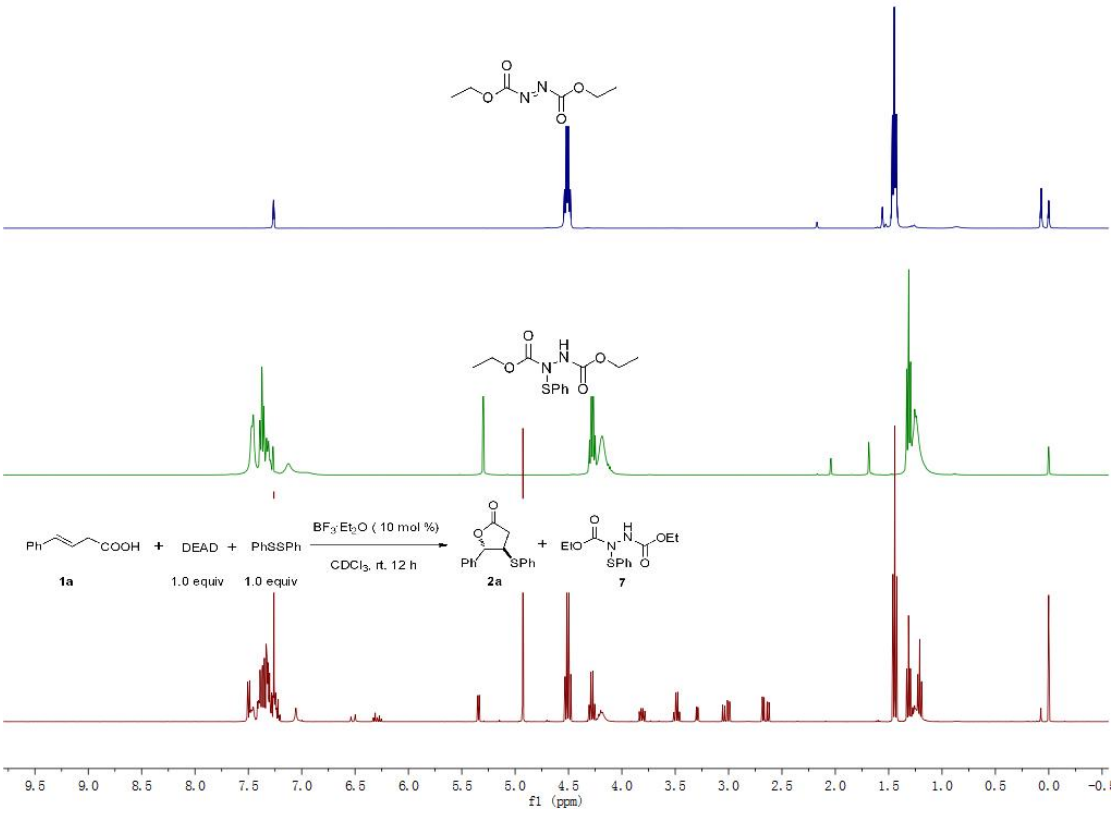
To a solution of **1a** (8.4 mg, 0.05 mmol, 1.0 equiv), PhSSPh (11.1 mg, 0.05 mmol, 1.0 equiv) and DEAD (7.9 μL, 0.05 mmol, 1.0 equiv) in $CDCl_3$ (1.0 mL) was added TFA

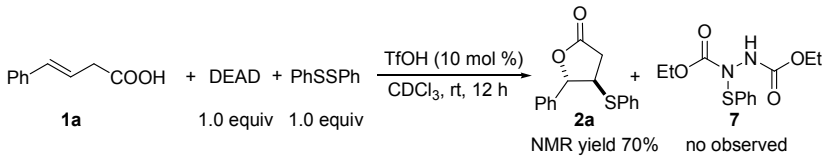
(100 μL 0.05 M solution in $CDCl_3$, 0.005 mmol, 10 mol %). The solution was stirred at room temperature for 12 h. The yields of **2a** and compound **7** were determined by ^1H NMR (400 MHz, $CDCl_3$) with CH_2Br_2 as the internal standard.



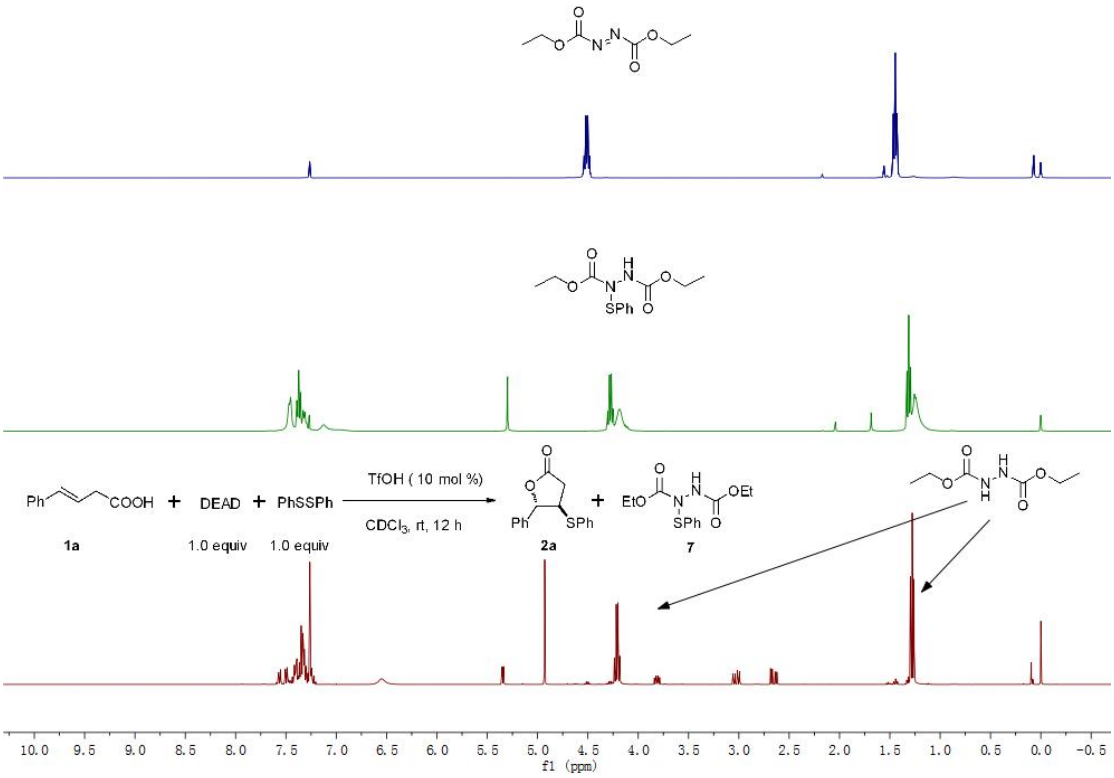


To a solution of **1a** (8.4 mg, 0.05 mmol, 1.0 equiv), PhSSPh (11.1 mg, 0.05 mmol, 1.0 equiv) and DEAD (7.9 μL, 0.05 mmol, 1.0 equiv) in $CDCl_3$ (1.0 mL) was added $BF_3{\cdot}Et_2O$ (100 μL 0.05 M solution in $CDCl_3$, 0.005 mmol, 10 mol %). The solution was stirred at room temperature for 12 h. The yields of **2a** and compound **7** were determined by ^1H NMR (400 MHz, $CDCl_3$) with CH_2Br_2 as the internal standard.

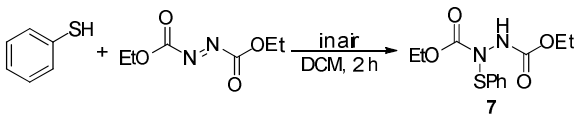




To a solution of **1a** (8.4 mg, 0.05 mmol, 1.0 equiv), PhSSPh (11.1 mg, 0.05 mmol, 1.0 equiv) and DEAD (7.9 μL, 0.05 mmol, 1.0 equiv) in $CDCl_3$ (1.0 mL) was added TfOH (100 μL 0.05 M solution in $CDCl_3$, 0.005 mmol, 10 mol %). The solution was stirred at room temperature for 12 h. The yields of **2a** and compound **7** were determined by ^1H NMR (400 MHz, $CDCl_3$) with CH_2Br_2 as the internal standard.

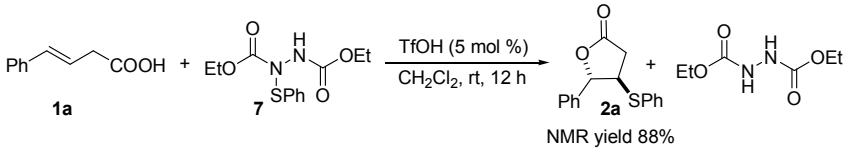


4.2 Synthesis of Compound 7



Compound **7** was prepared according to the literature:[25] To a solution of thiol (511 μL, 5.0 mmol, 1.0 equiv) in DCM (10.0 mL) was added DEAD (803 μL, 5.0 mmol, 1.0 equiv). The solution was stirred in air at room temperature. Two hours later, the solution was washed with aqueous NaOH (1 mol/L) and brine, and concentrated under reduced pressure. The residue was purified by flash silica gel column chromatography (eluent: PE/EA = 10/1, v/v) to afford compound **7**.

4.3 Reaction of Compound 7 with 1a



To a solution of **1a** (8.4 mg, 0.05 mmol, 1.0 equiv) and compound **7** (14.2 mg, 0.05

mmol, 1.0 equiv) in DCM (1.0 mL) was added TfOH (100 μL 0.025 M solution in DCM, 0.0025 mmol, 5 mol %). The resulting solution was stirred at room temperature for 12 h, after which the solvent was removed under reduced pressure. The yield of **2a** was determined by 400 MHz NMR with CH_2Br_2 as the internal standard.

5. References

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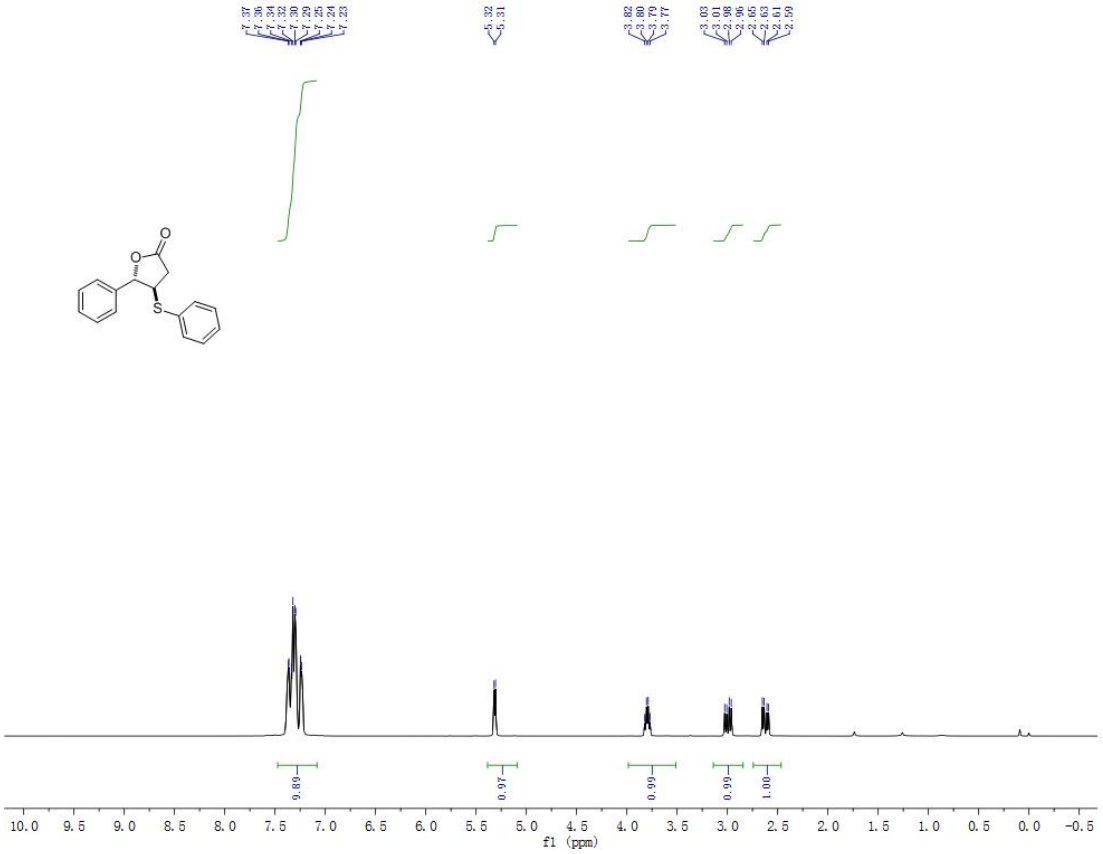
6. NMR spectra for products

^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2a**



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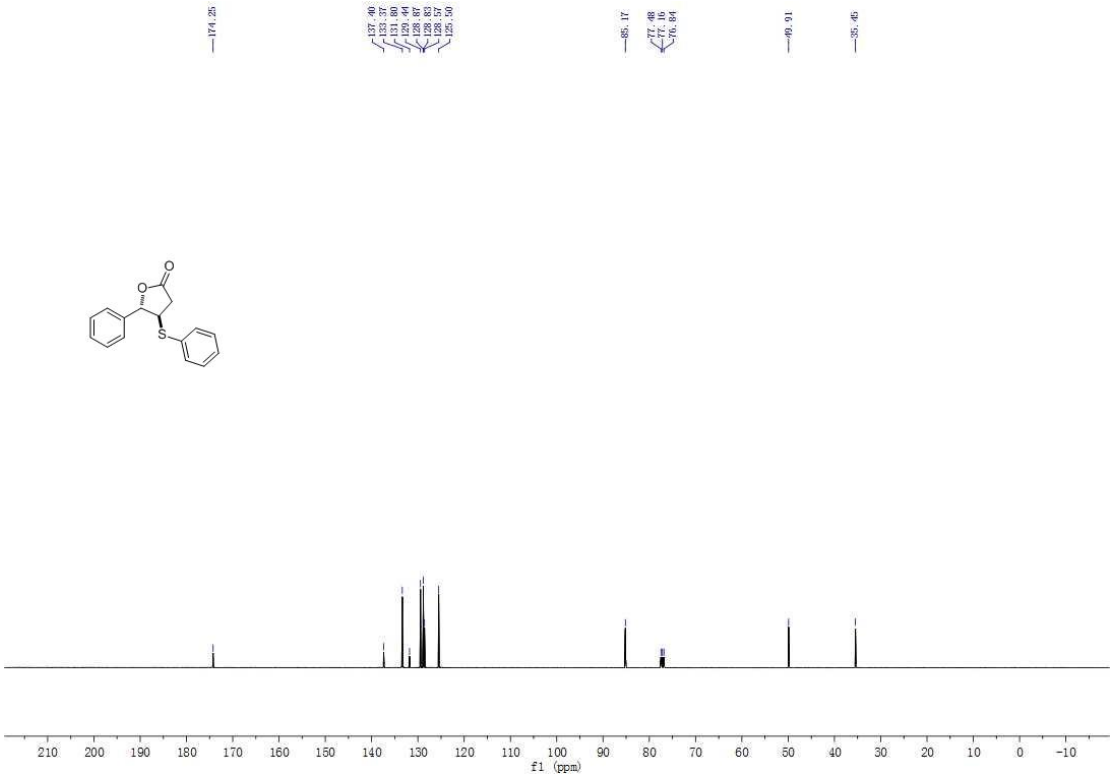
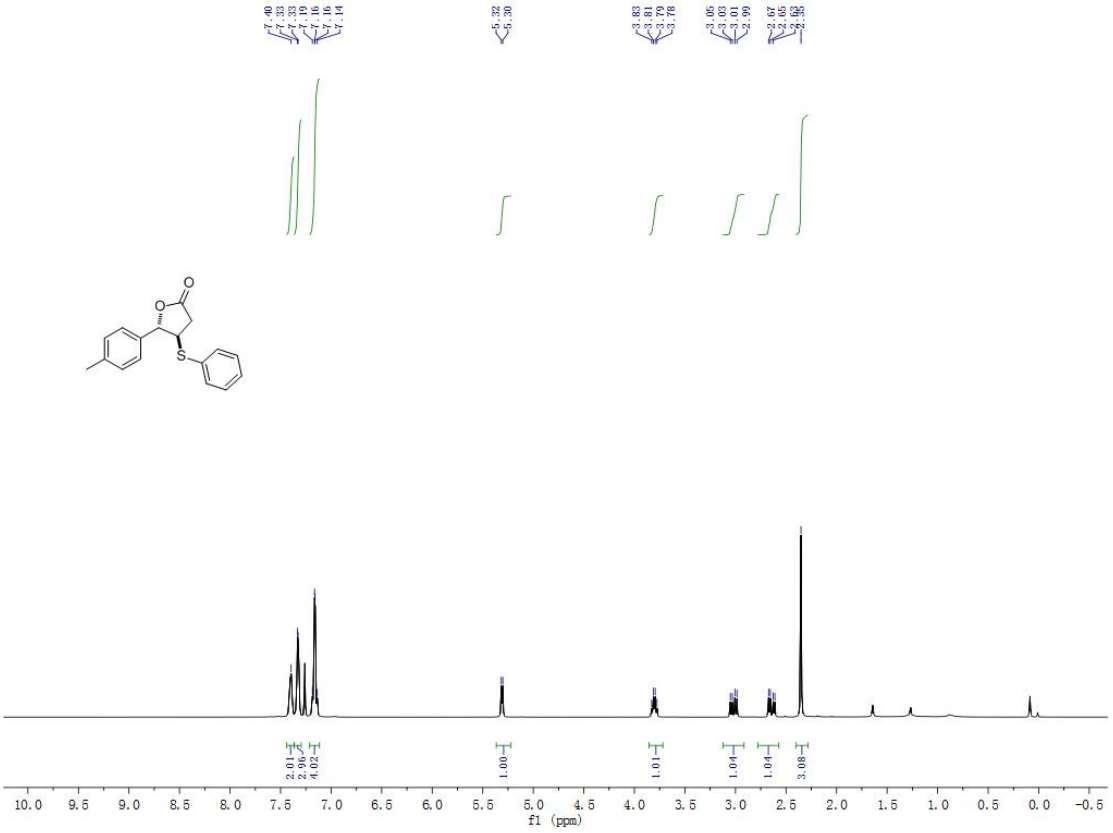
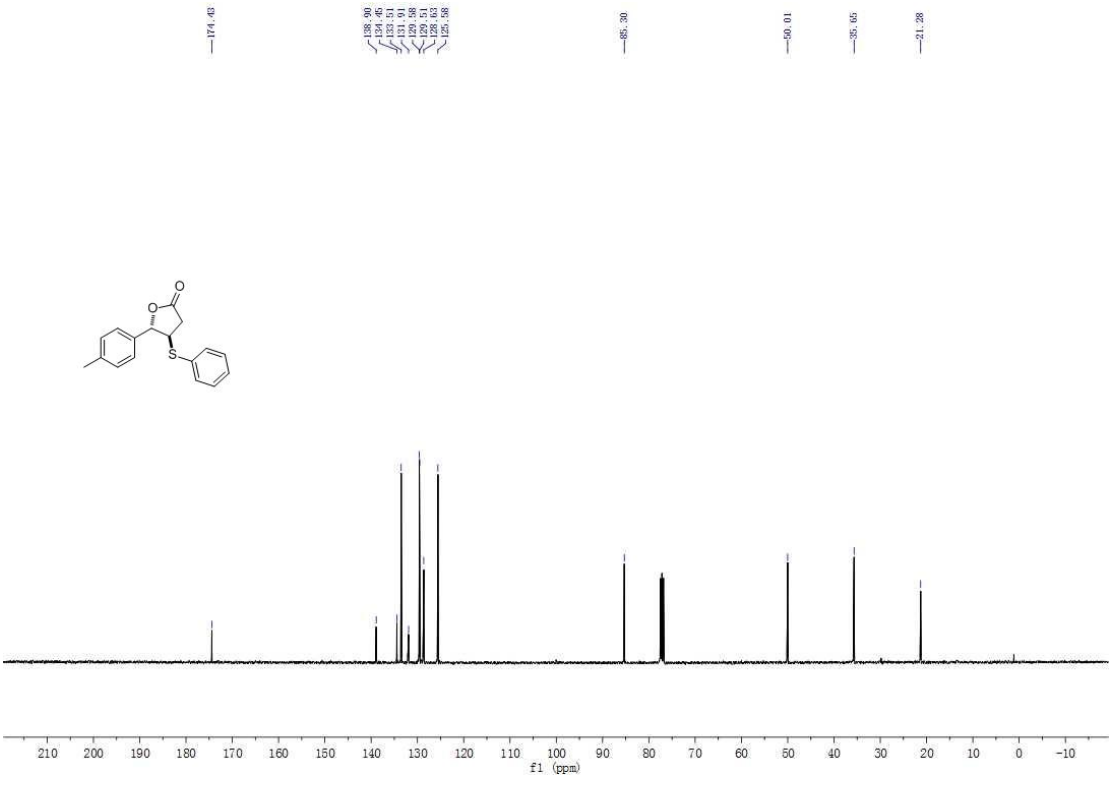


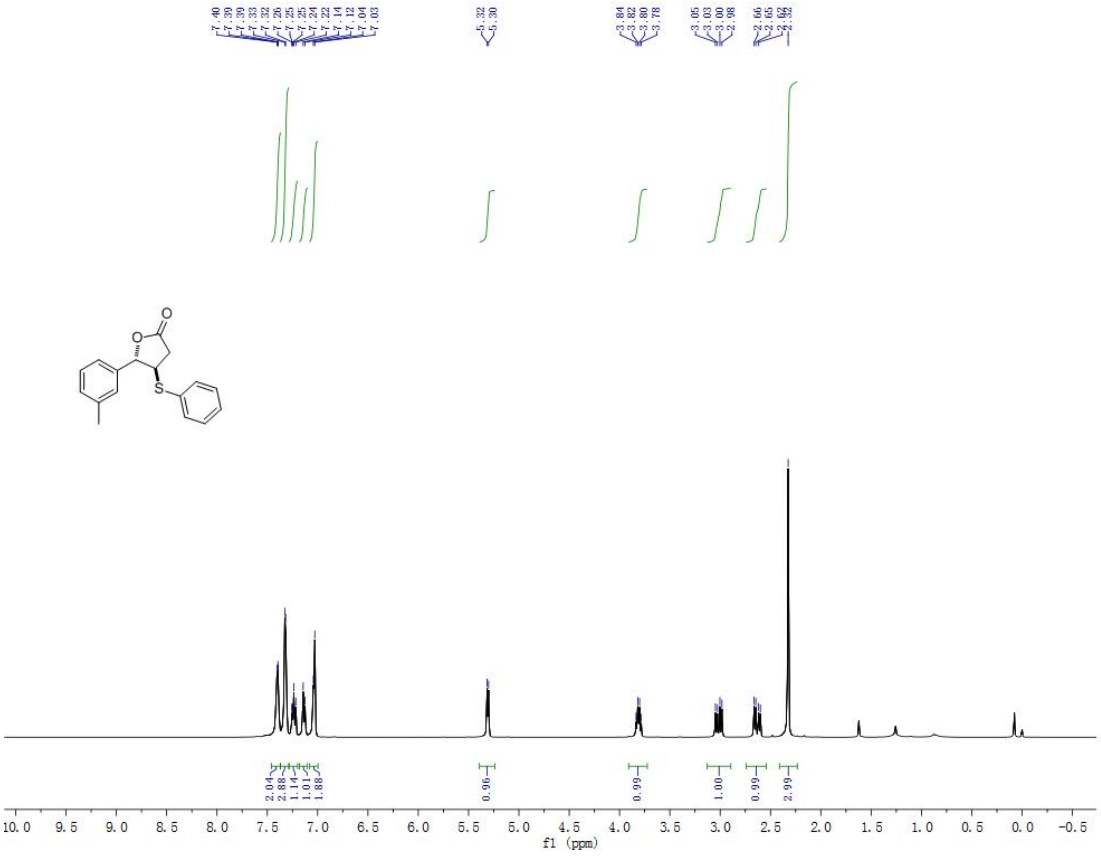
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2b**



^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2b**



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2c**



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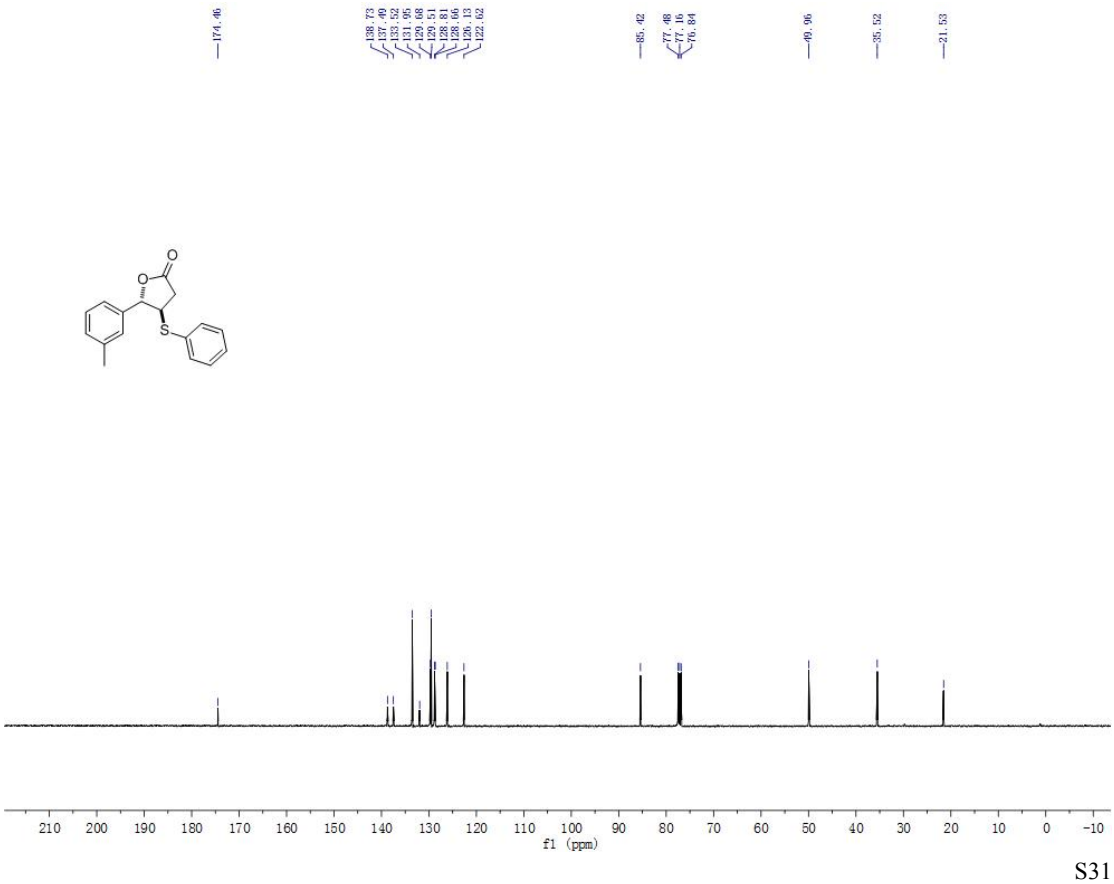
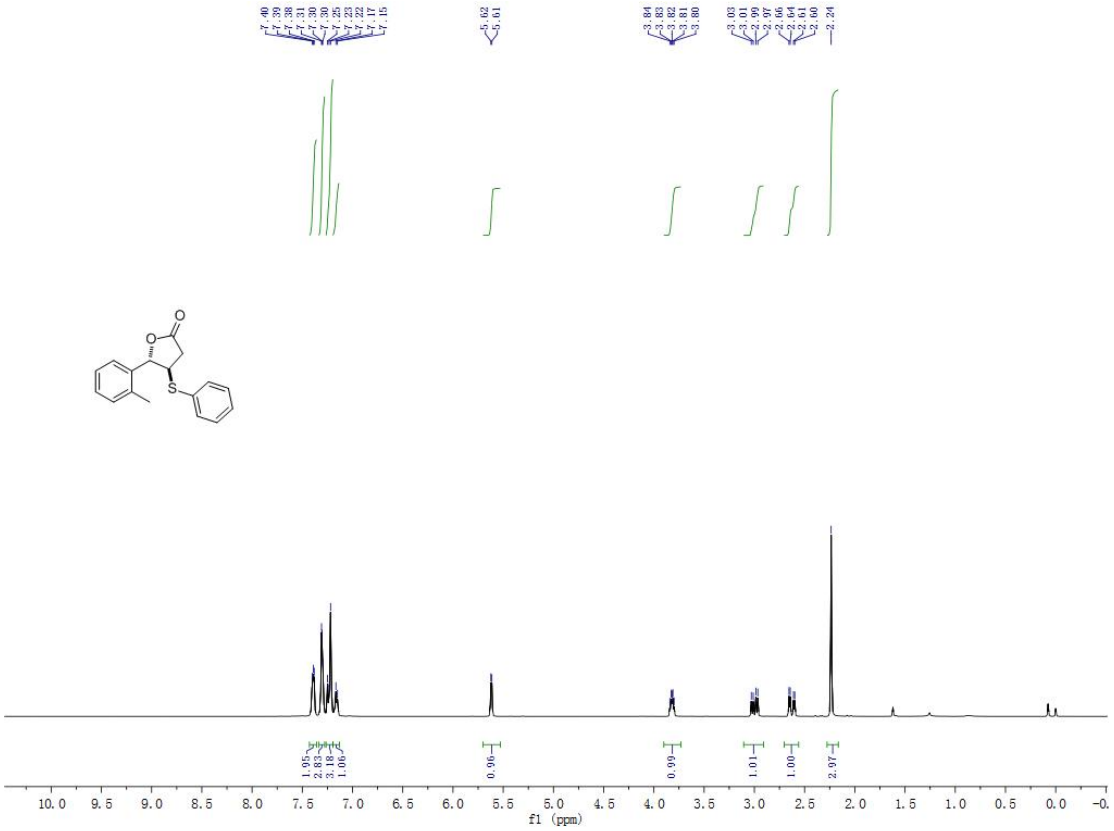
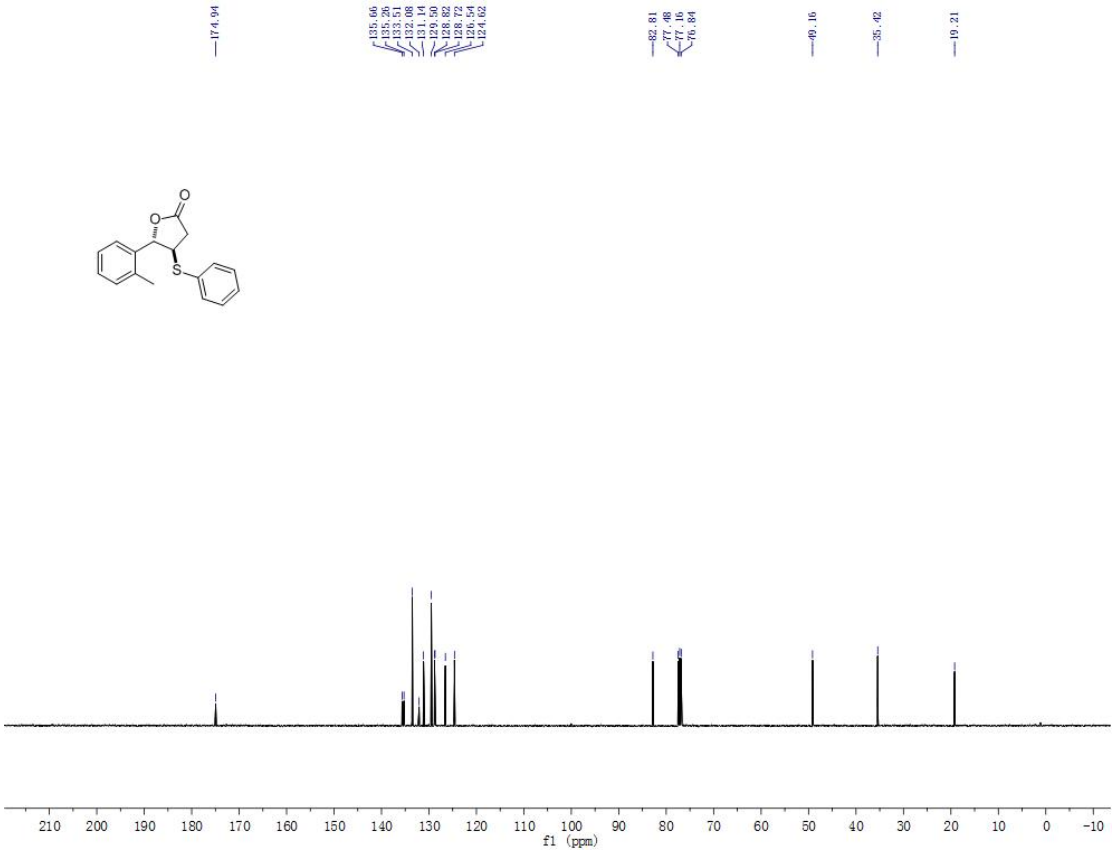


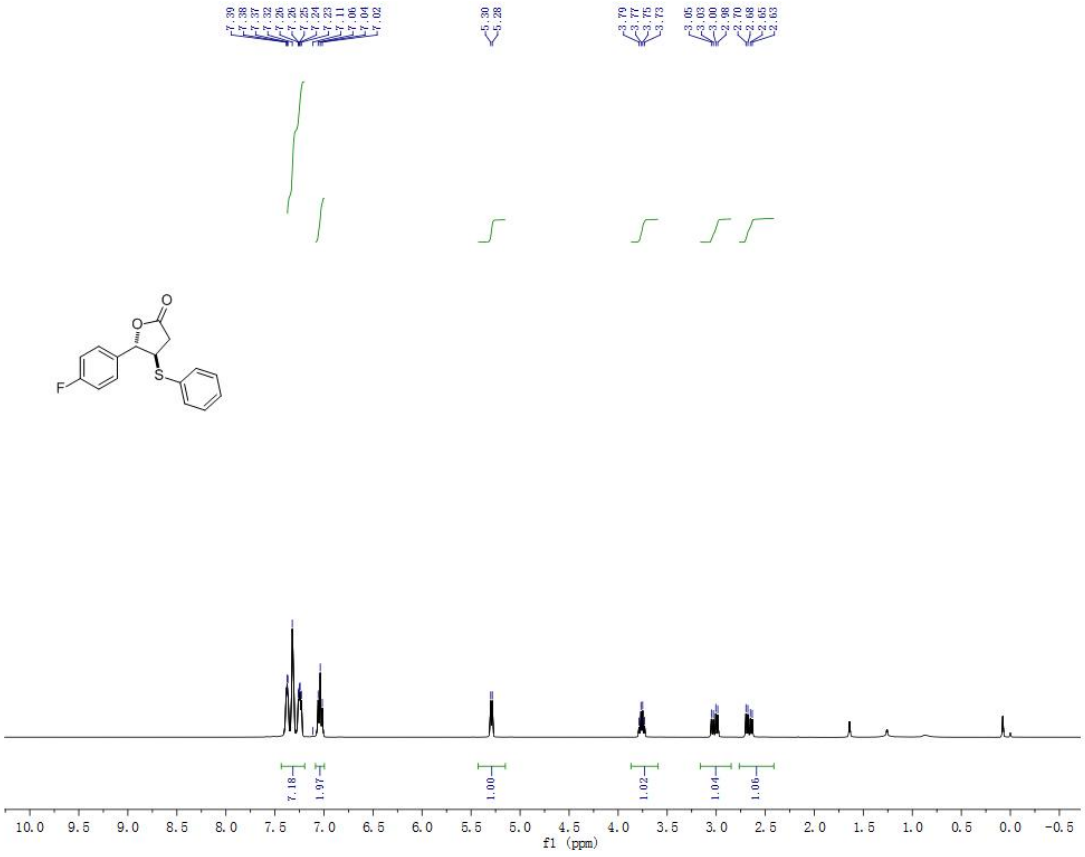
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2d**



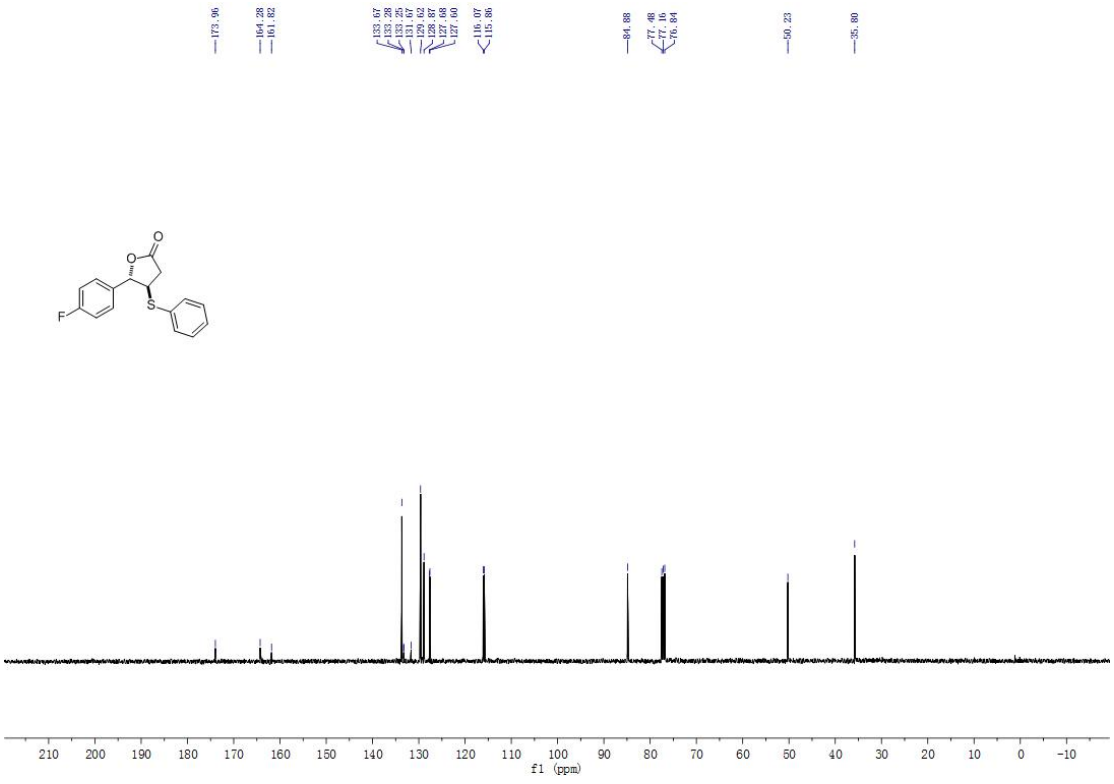
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2d**



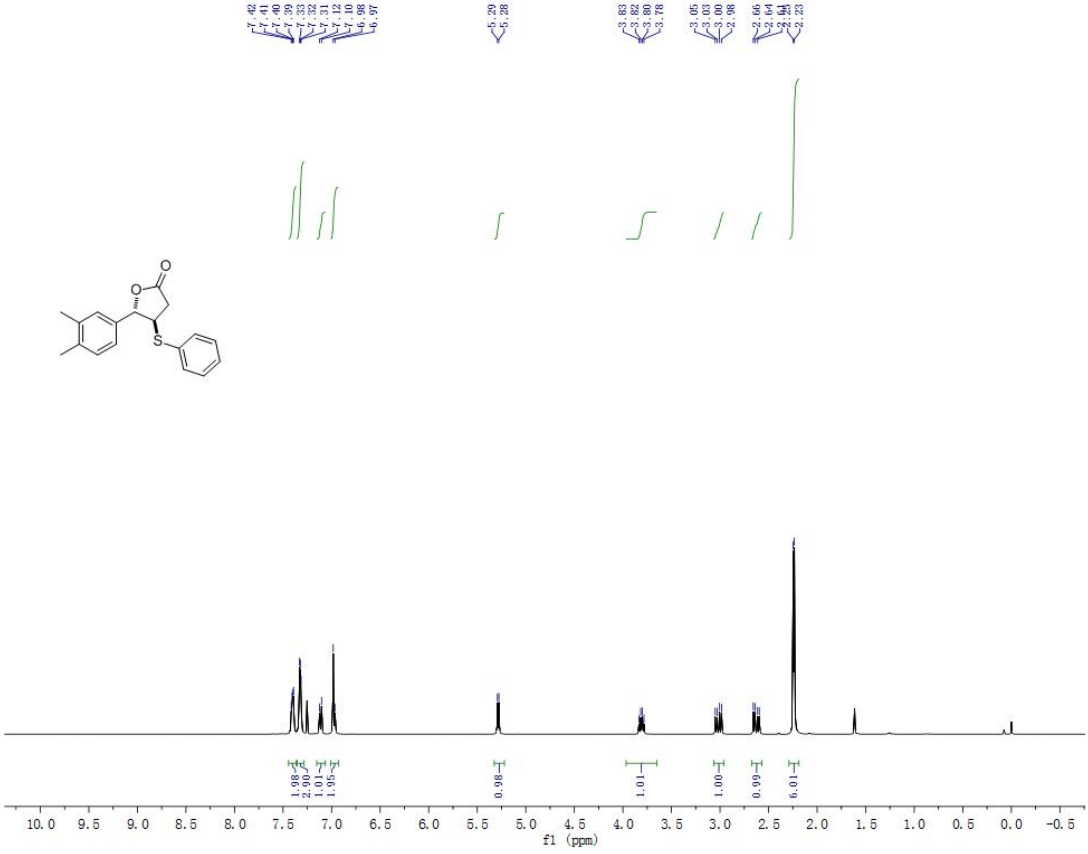
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2e**



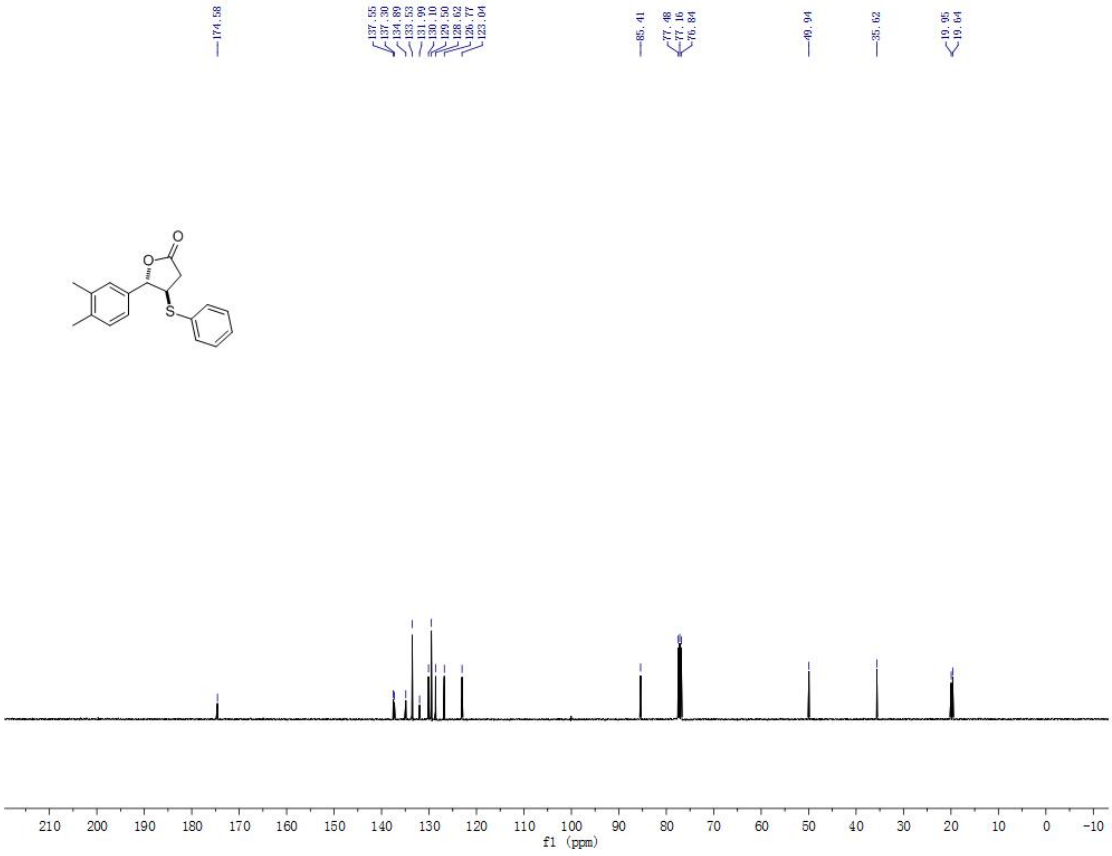
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2e**



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2f**



^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2f**



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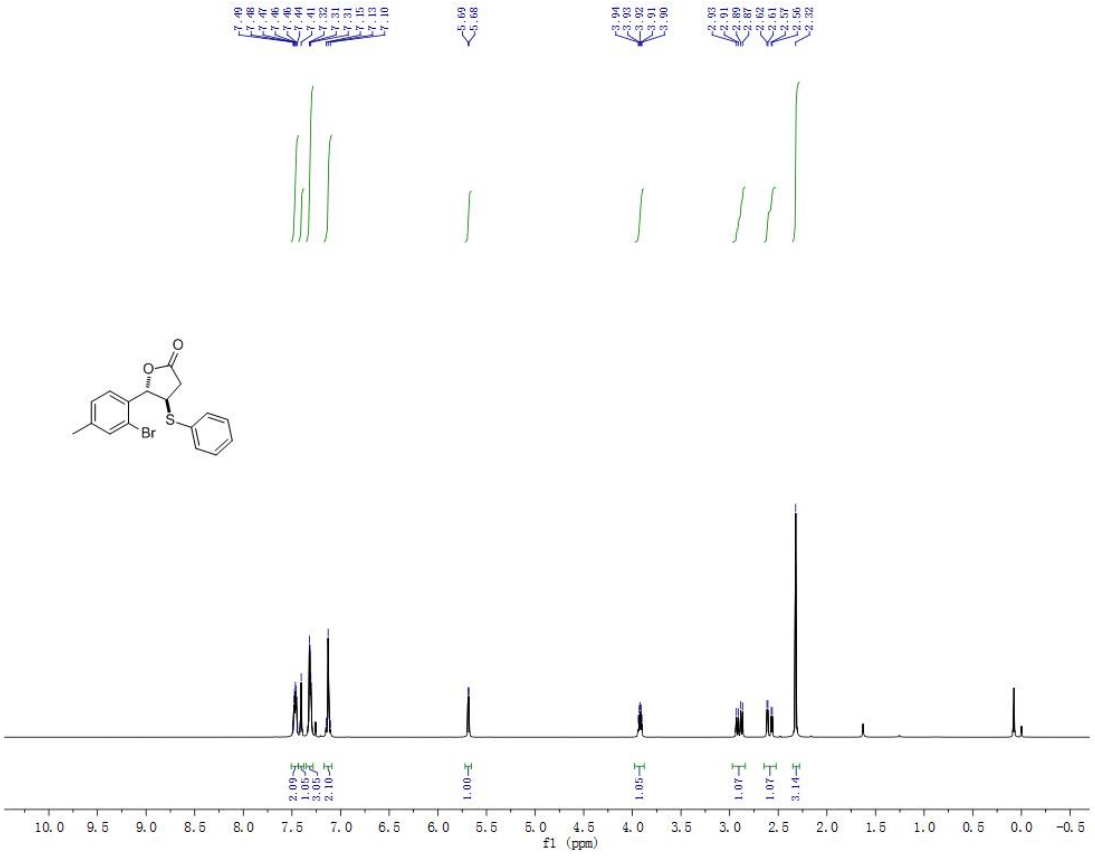

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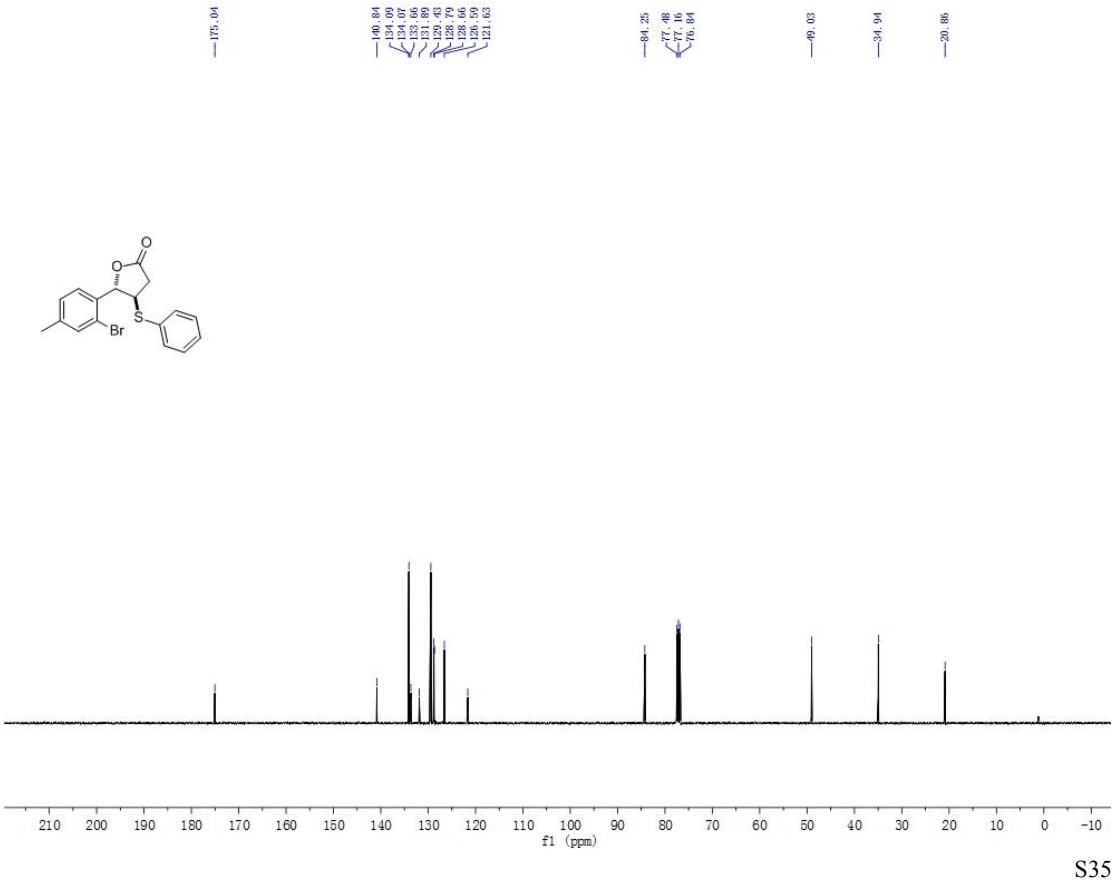

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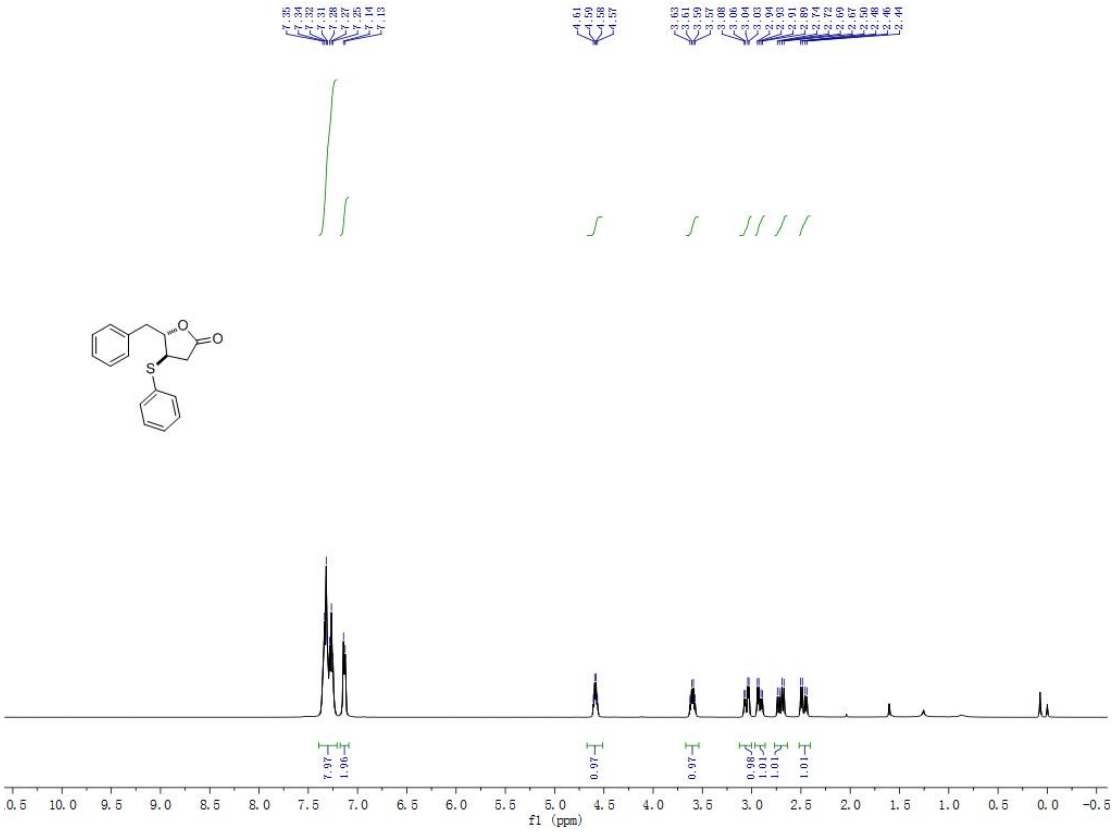

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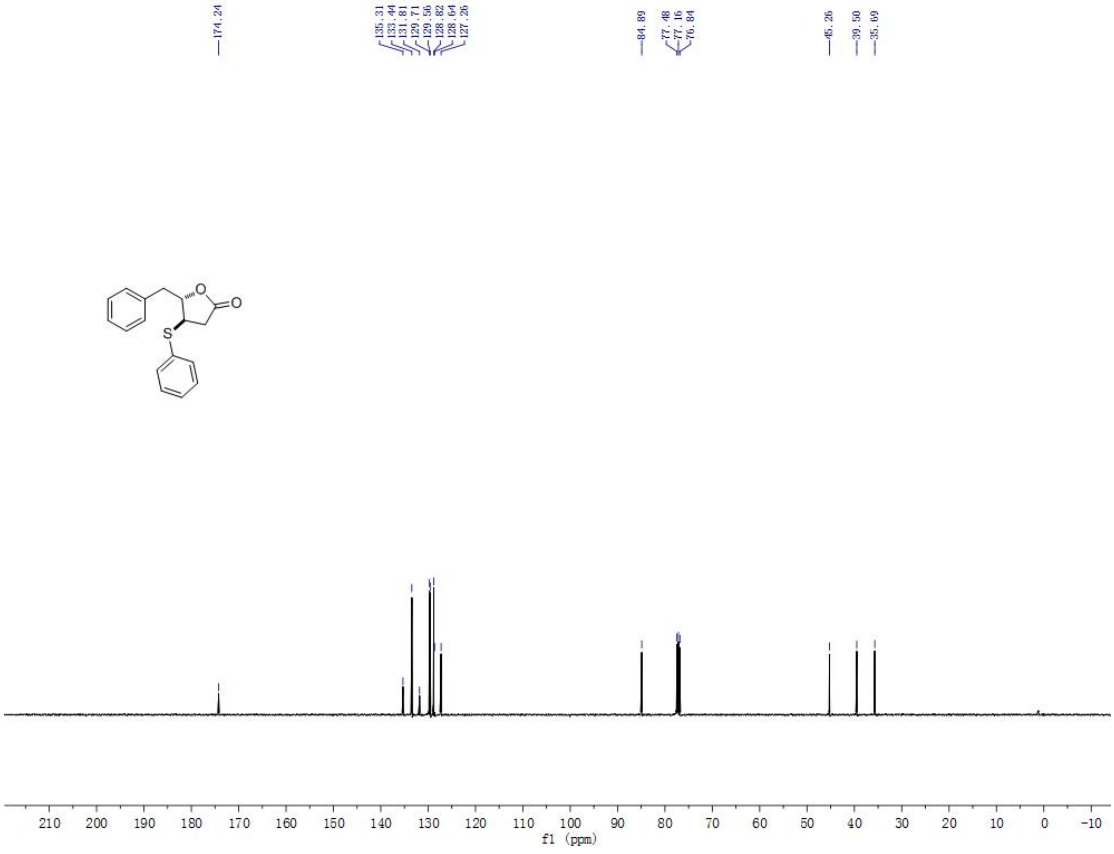
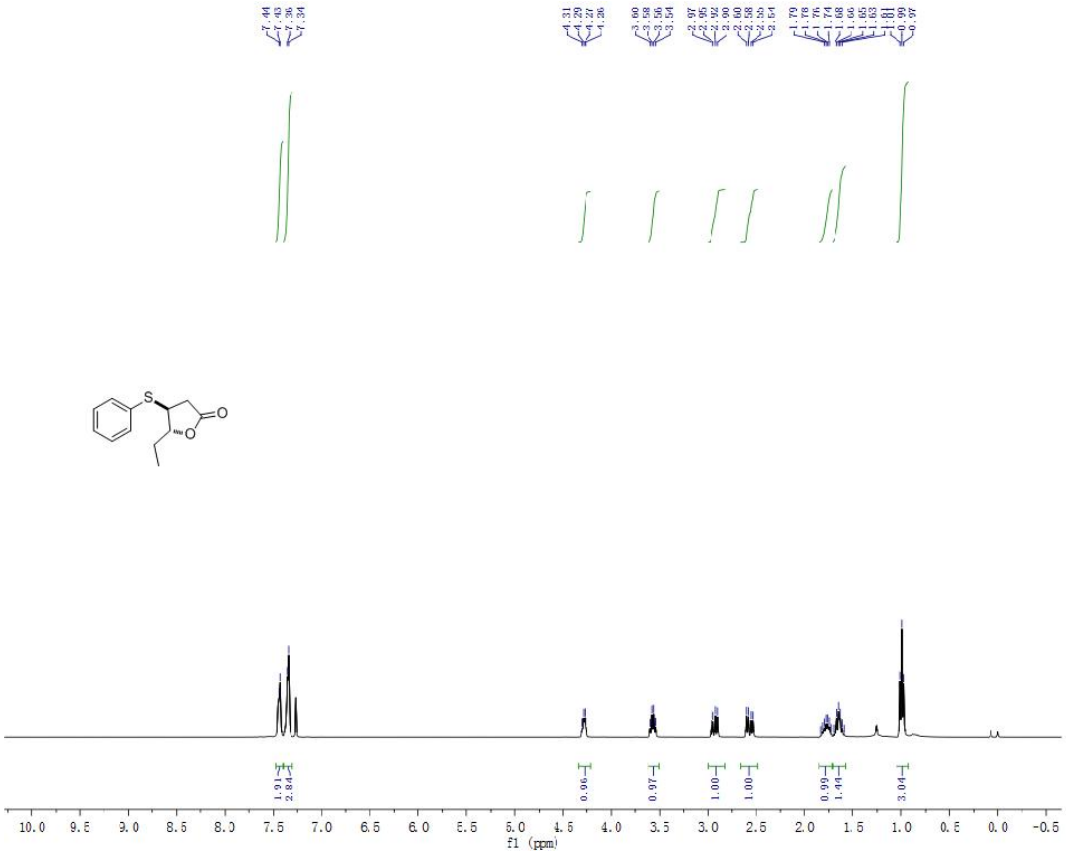
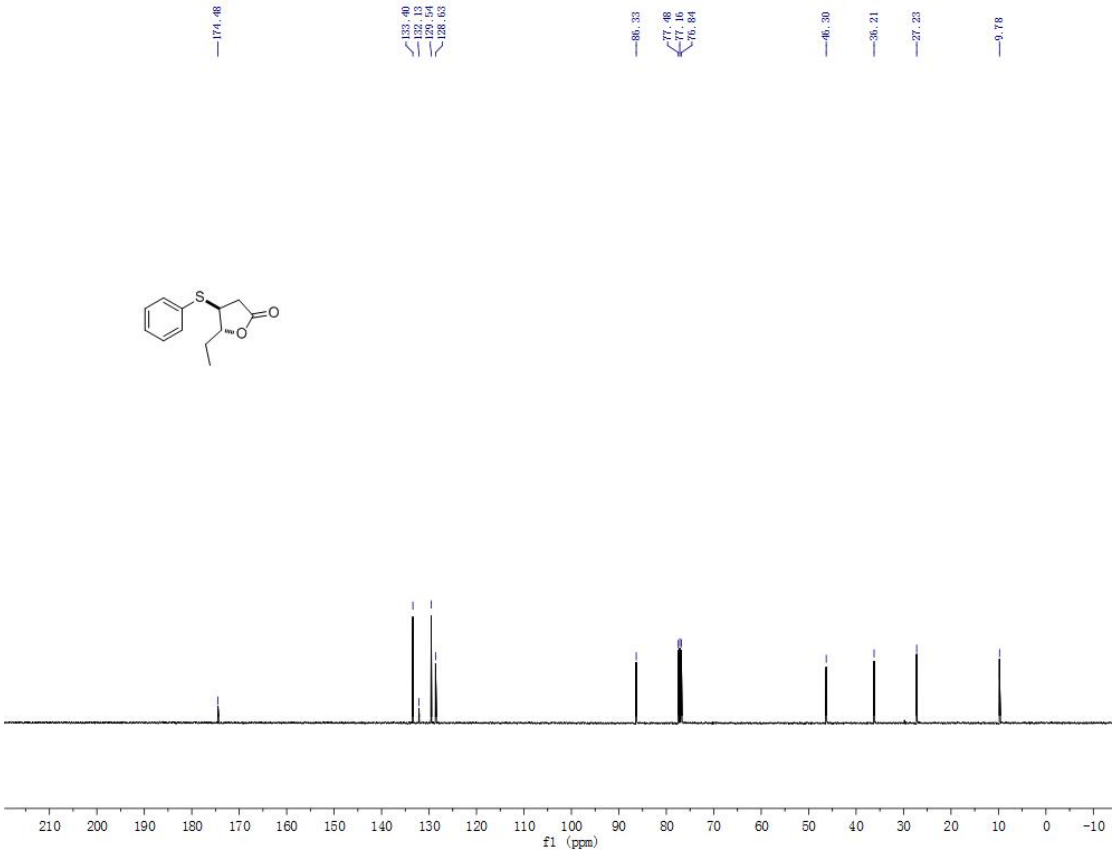


^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2i**



^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2i**



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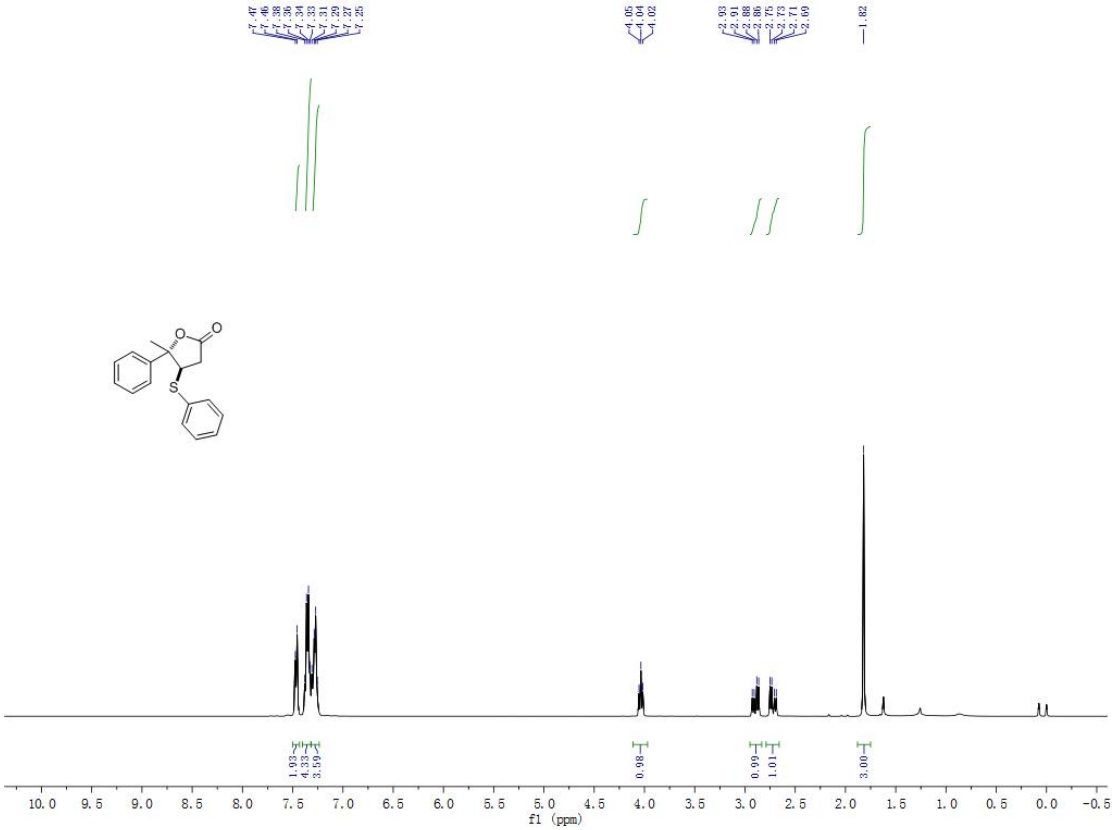
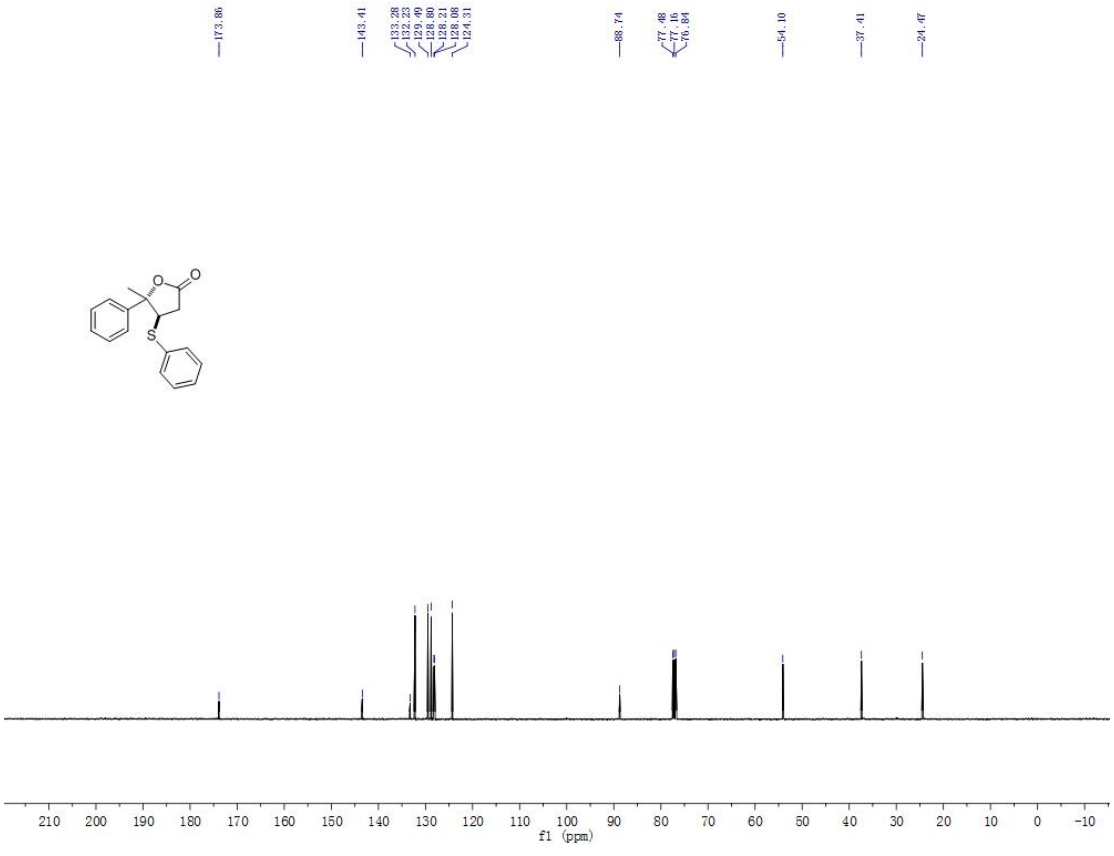
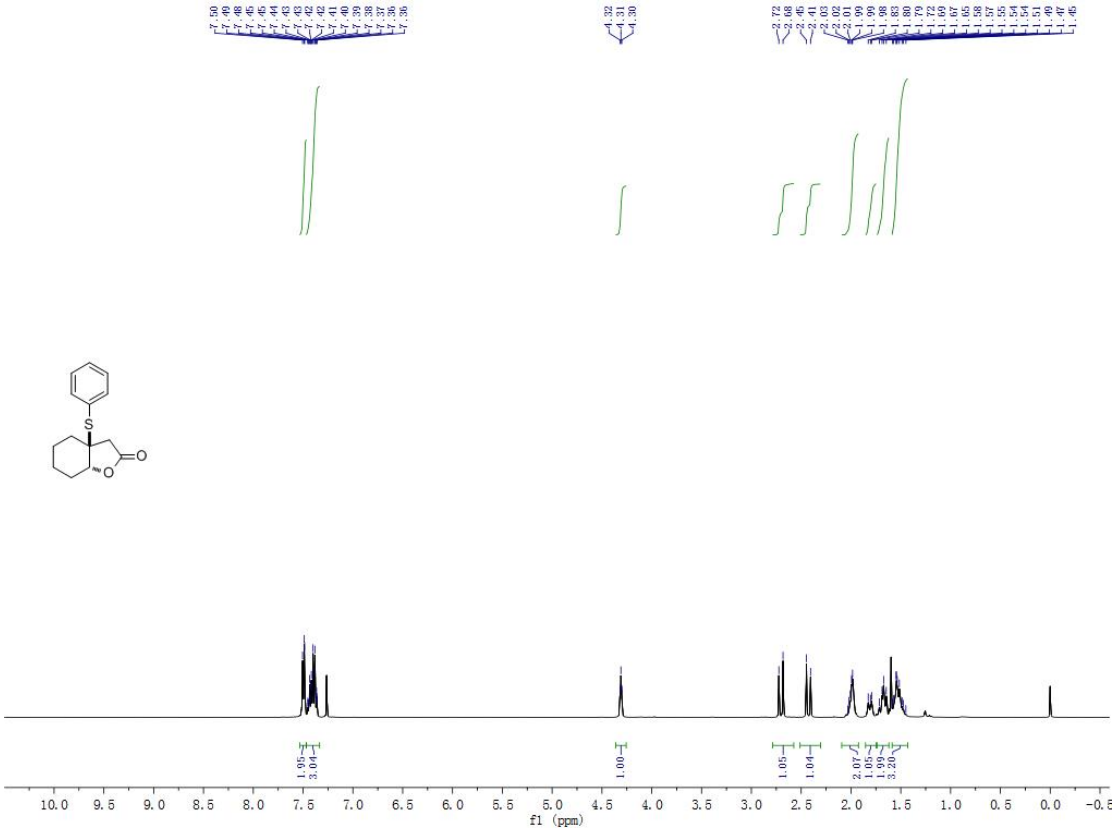


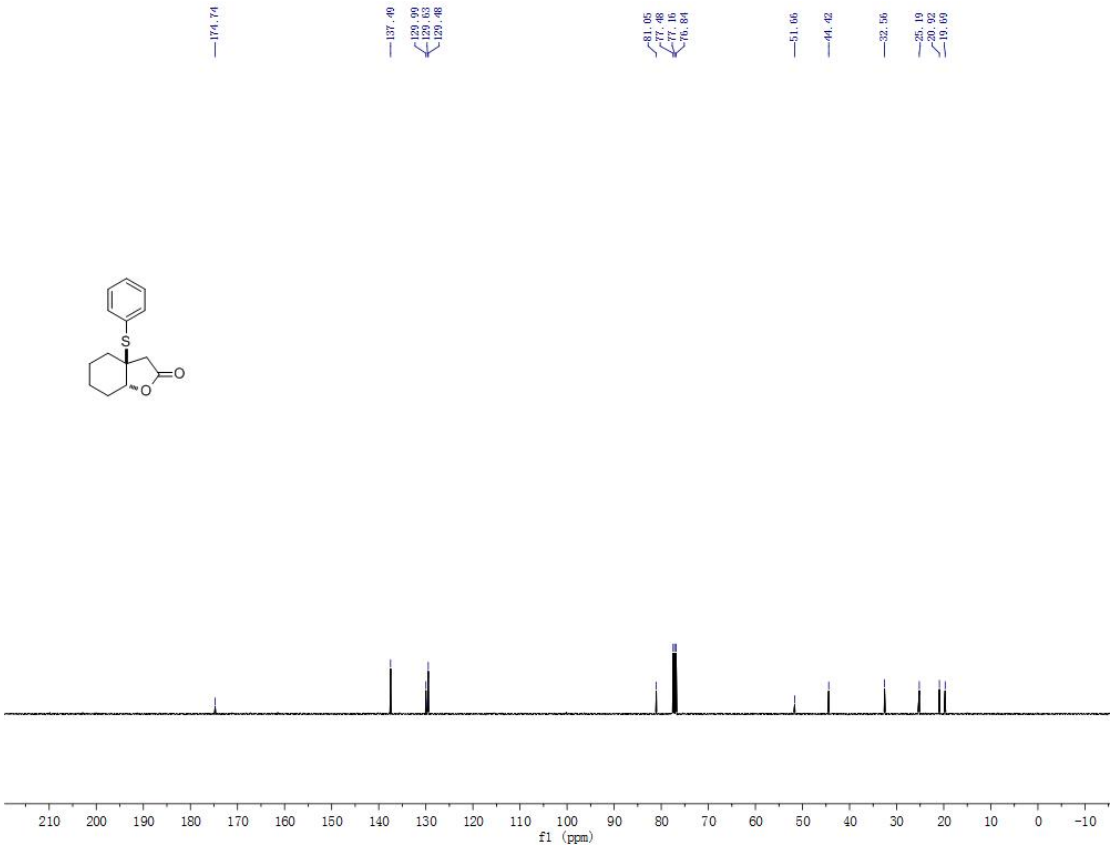
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2j**



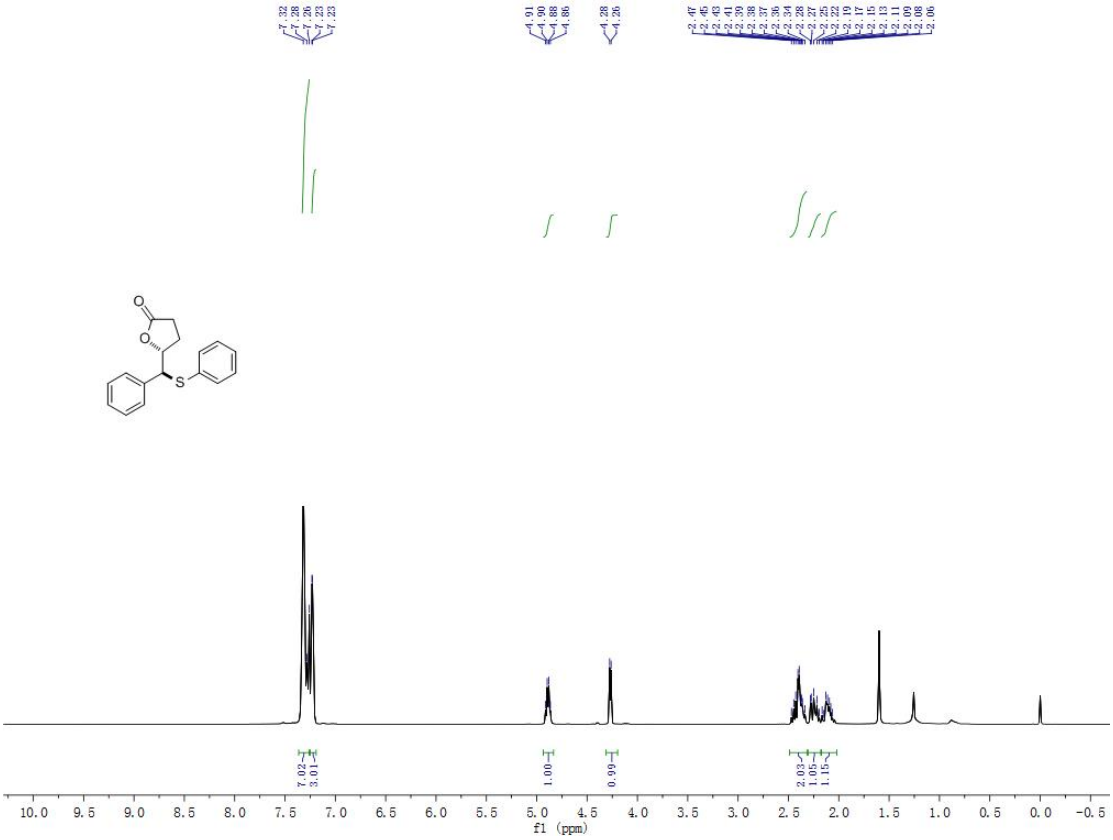
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2k**



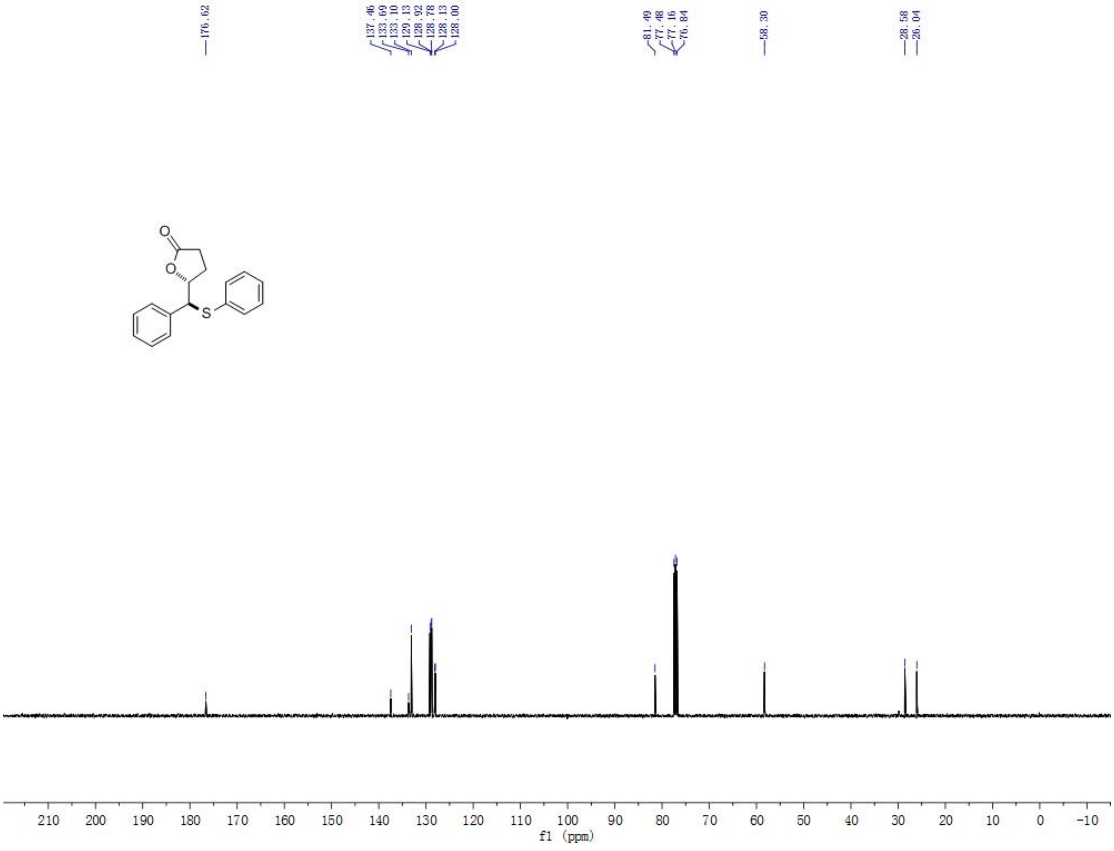
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2k**



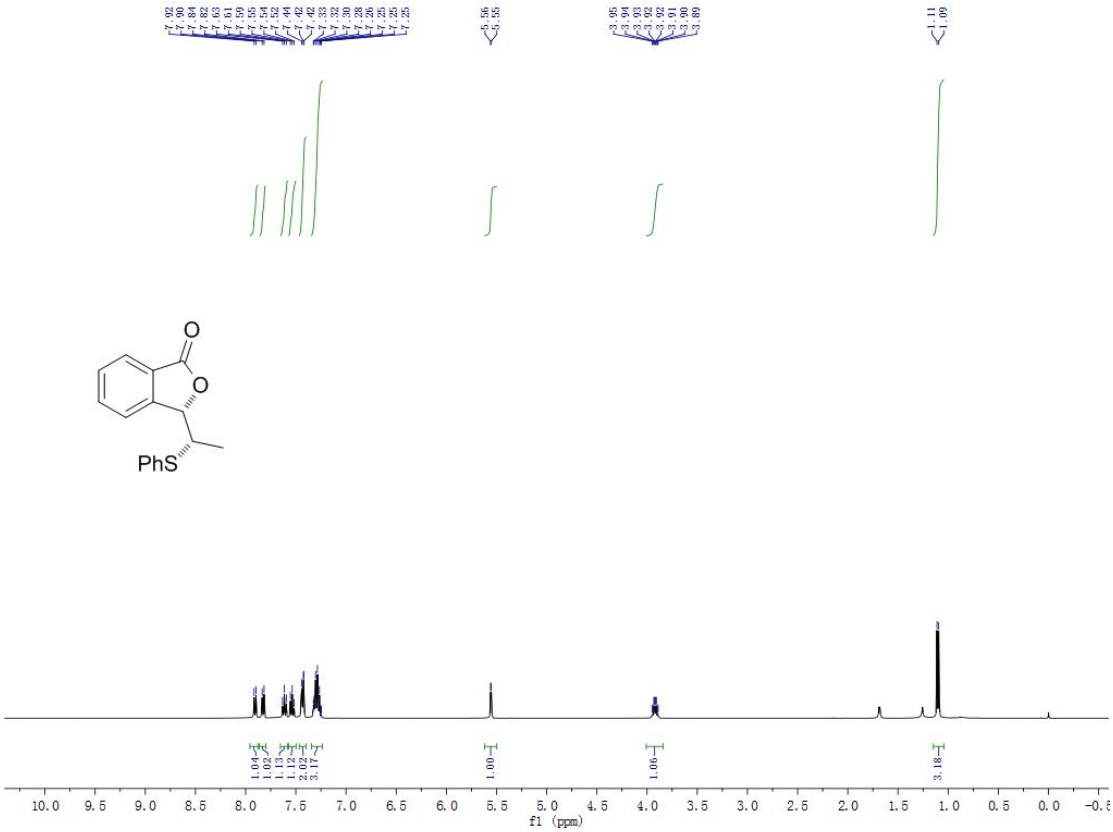
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2l**



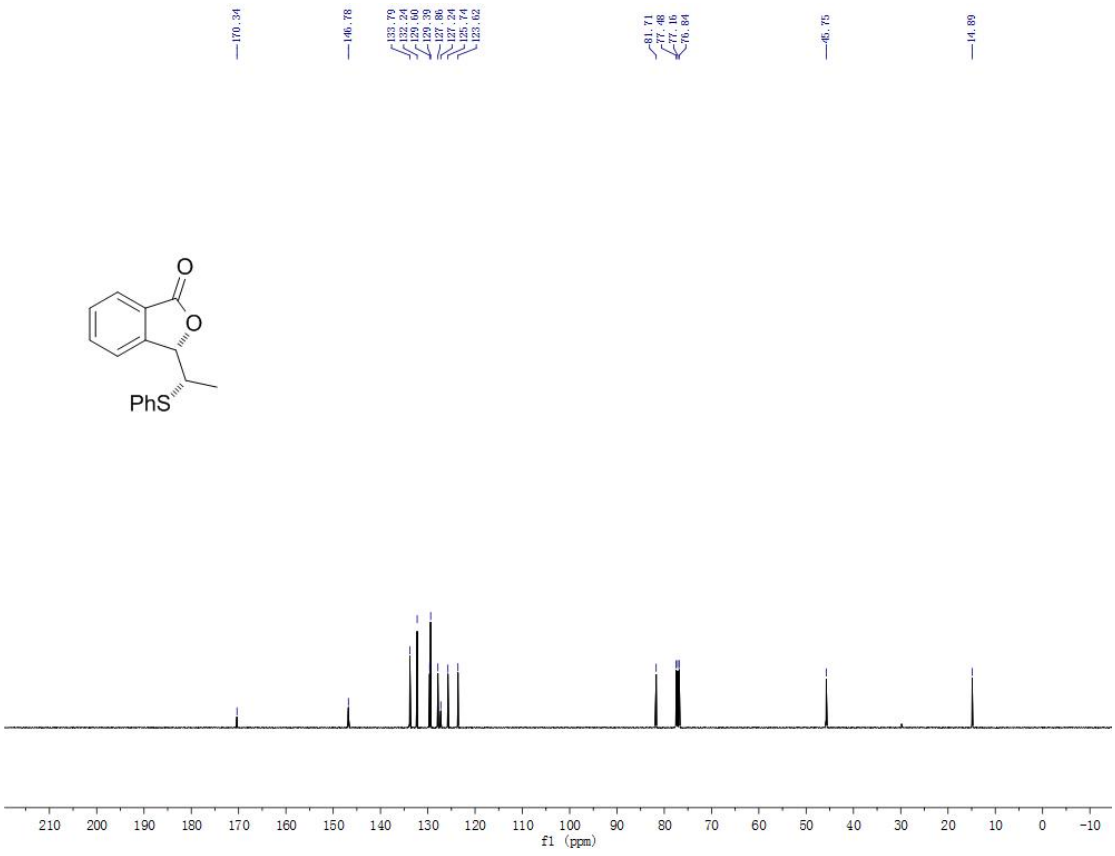
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2l**



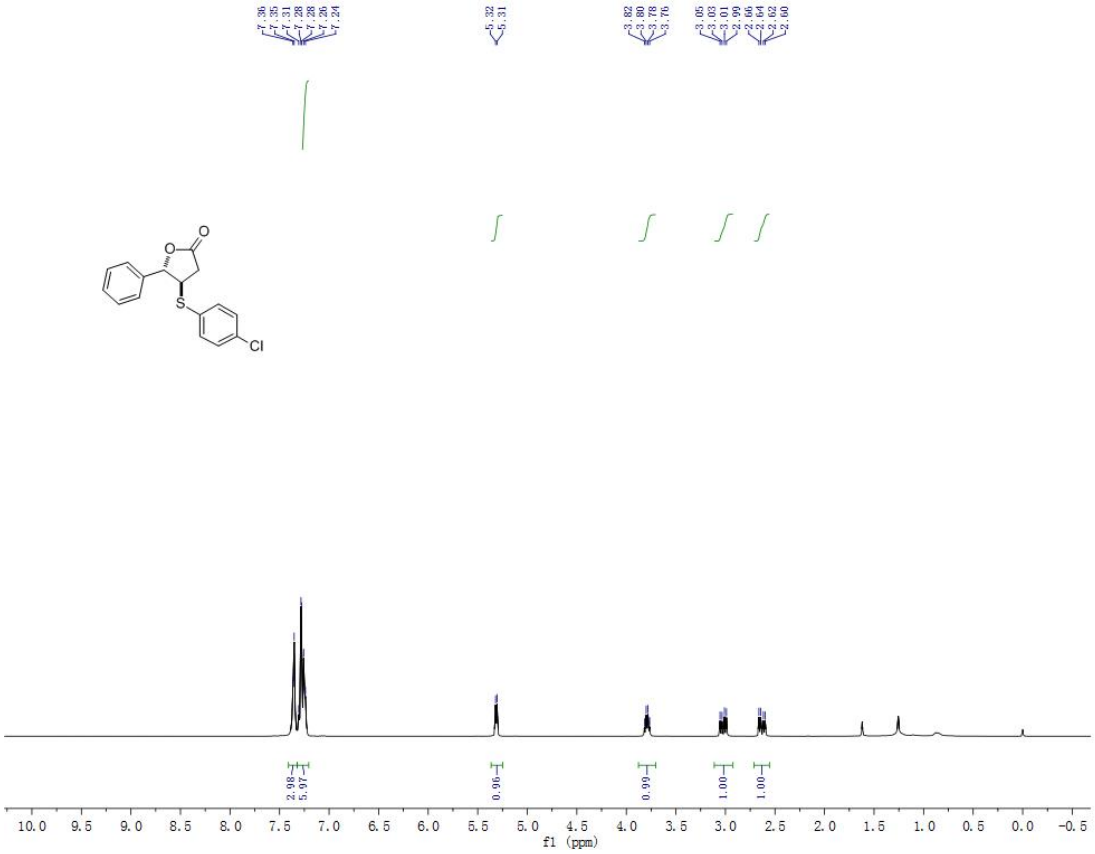
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2m**



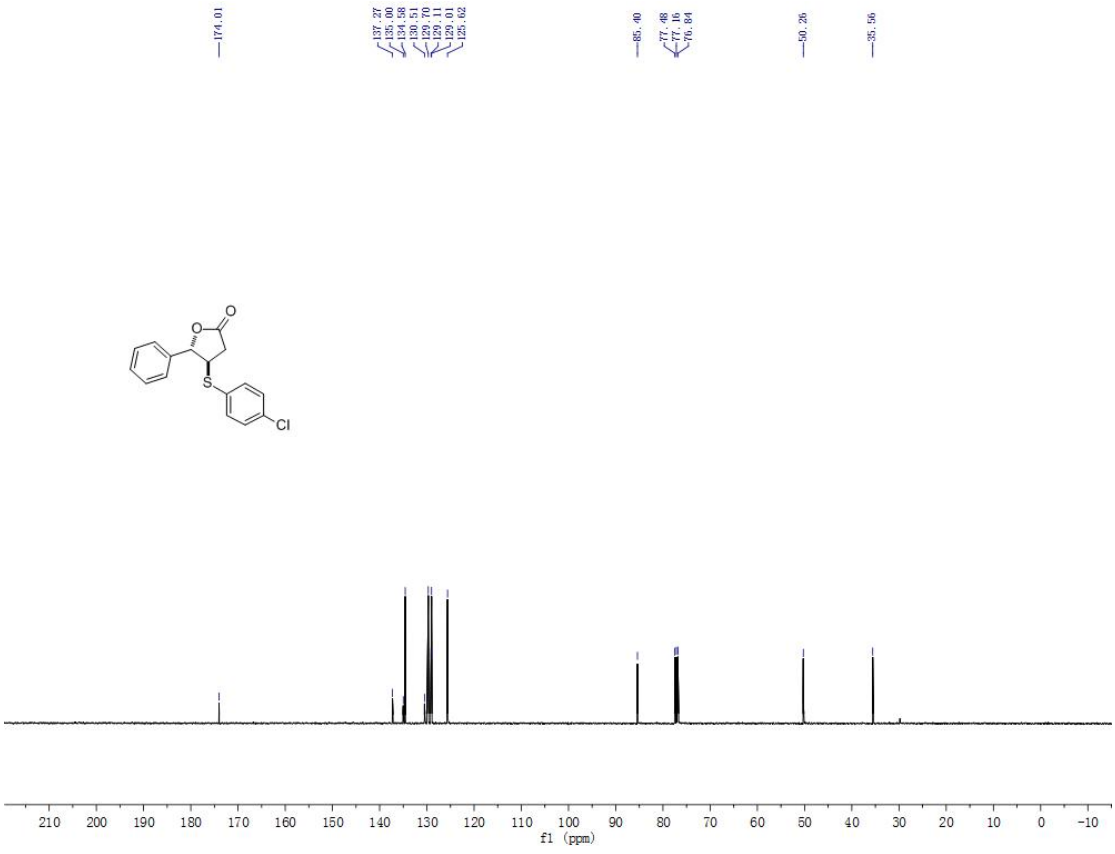
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2m**



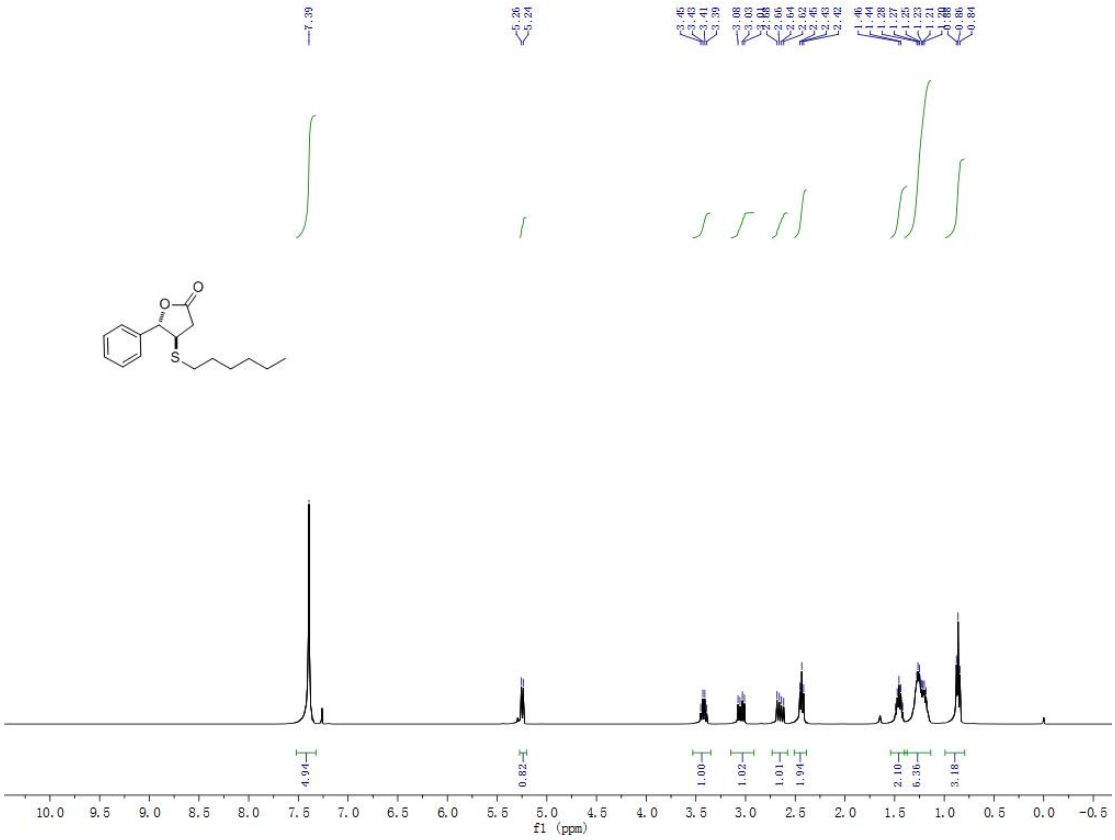
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2n**



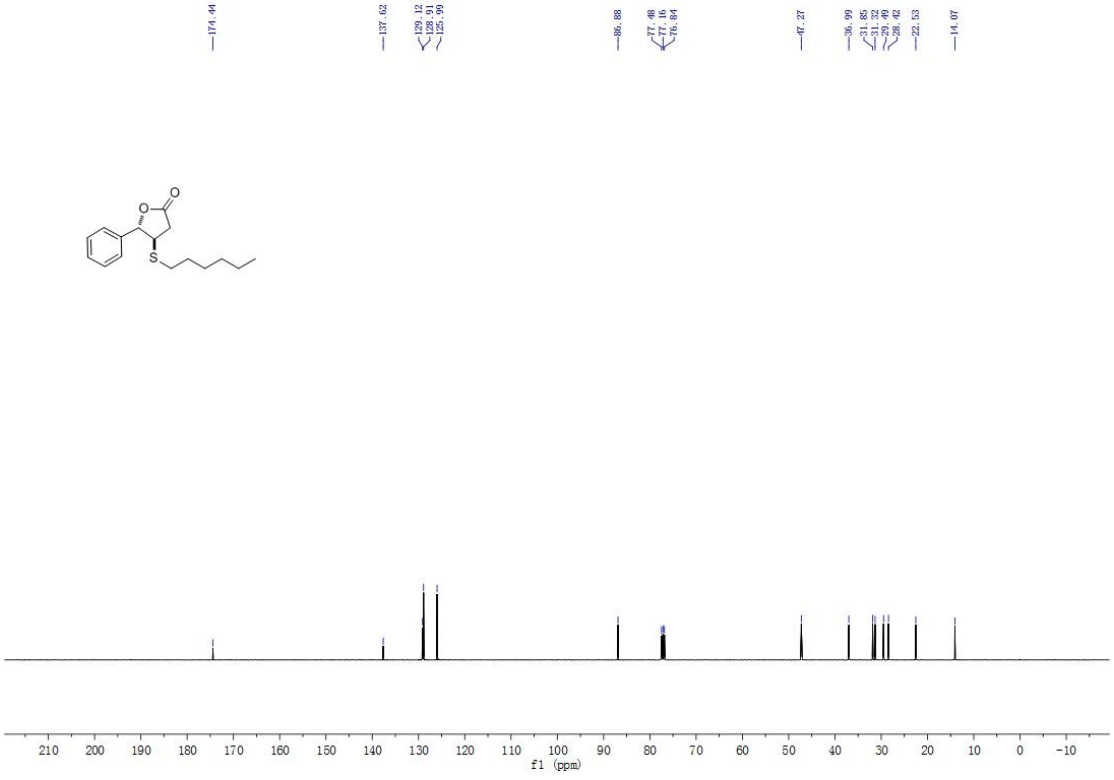
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2n**



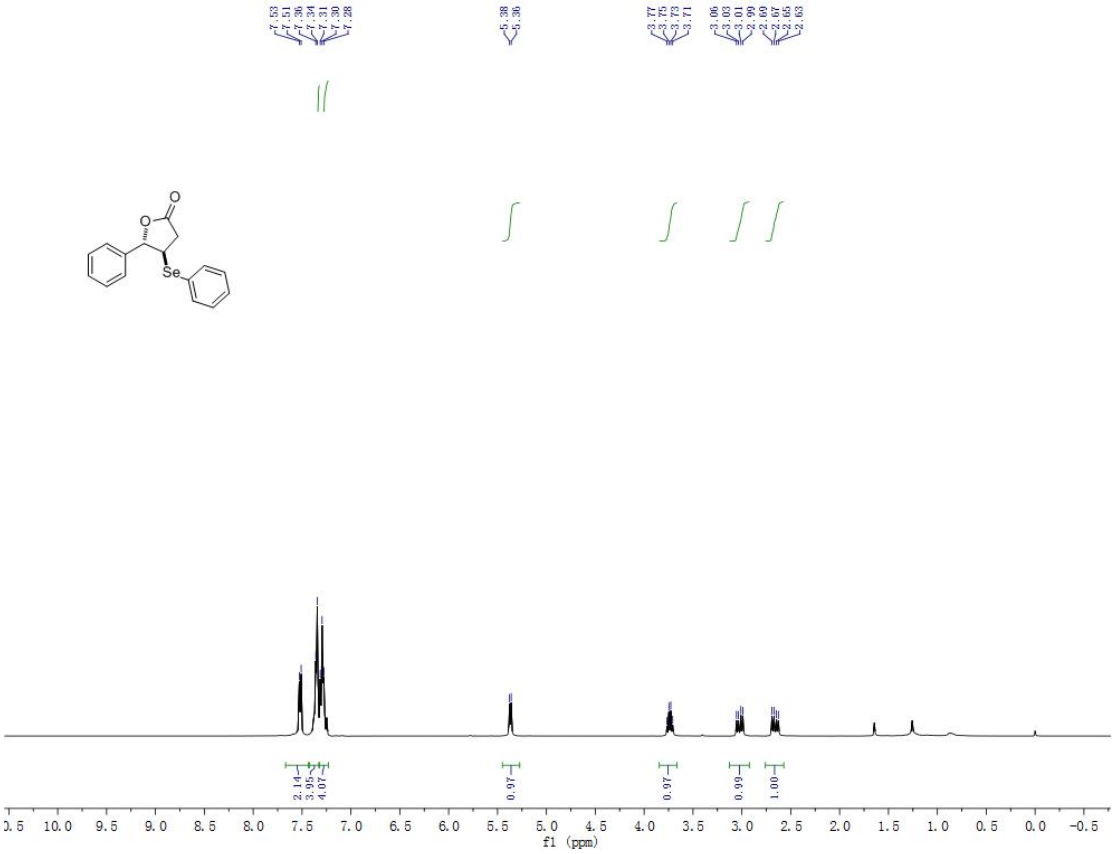
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2o**



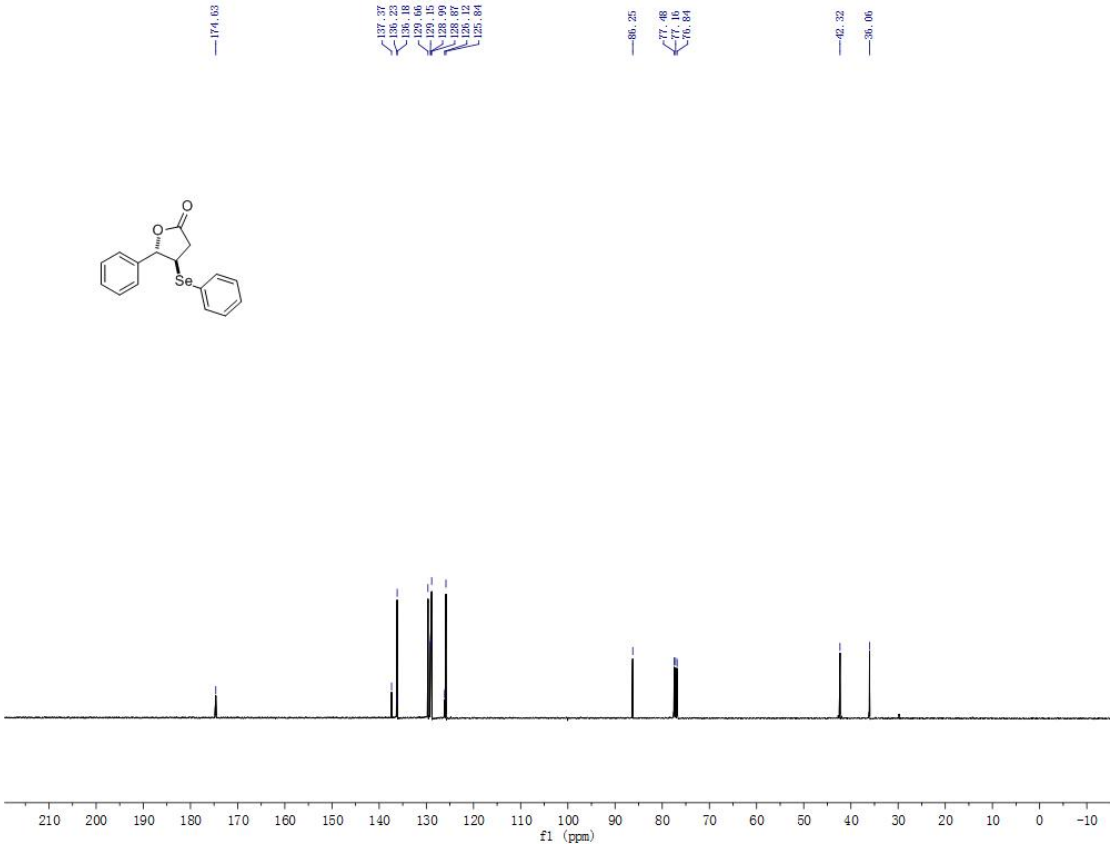
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2o**



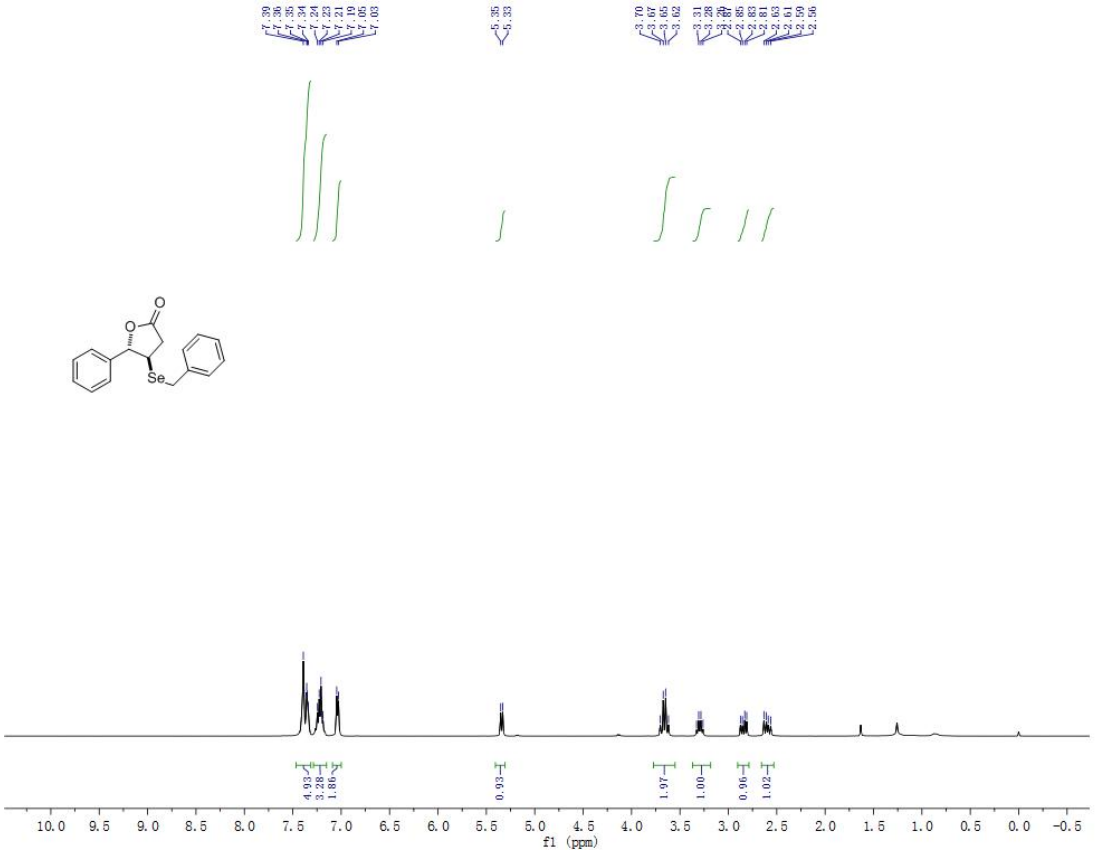
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2p**



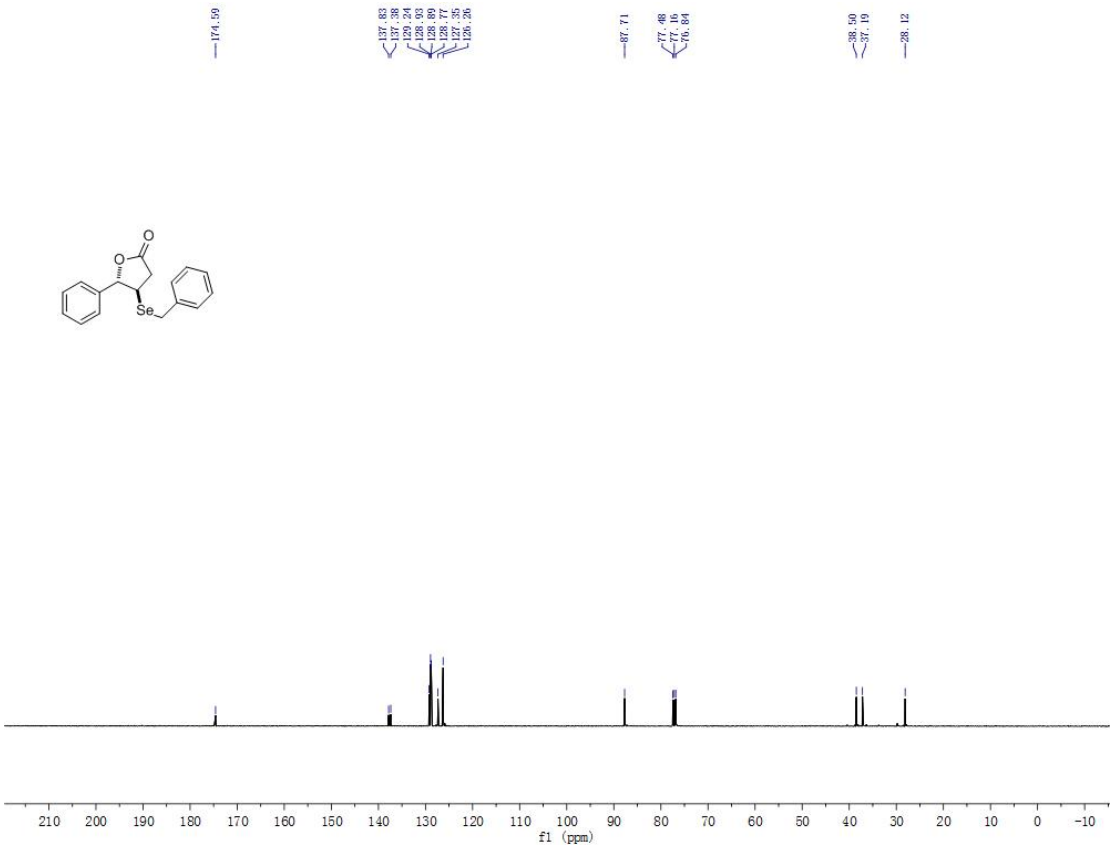
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2p**



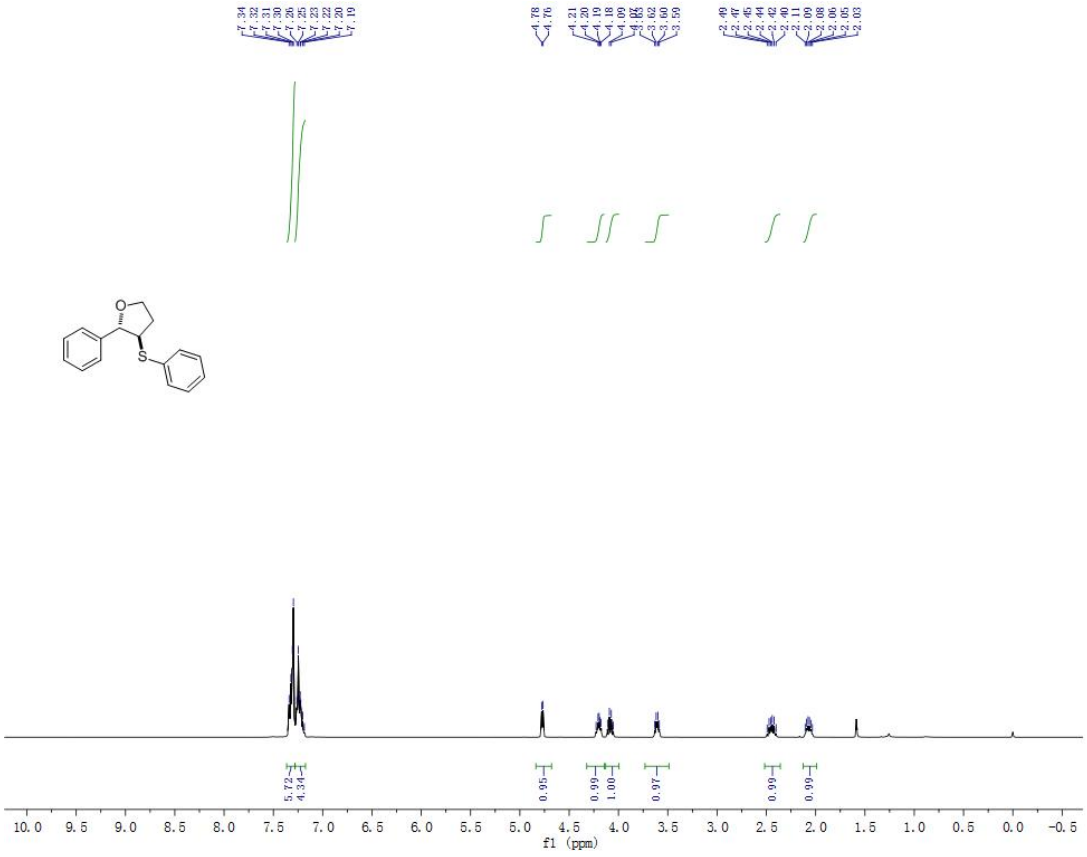
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **2q**



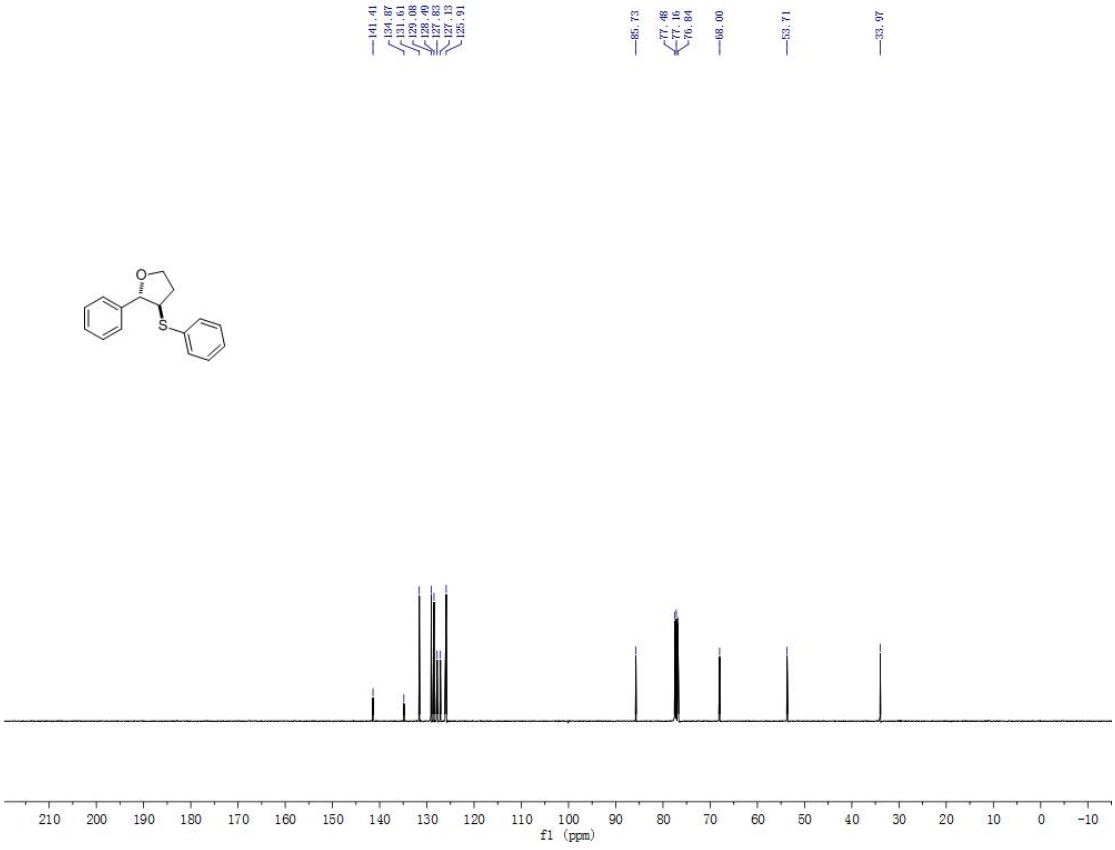
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **2q**



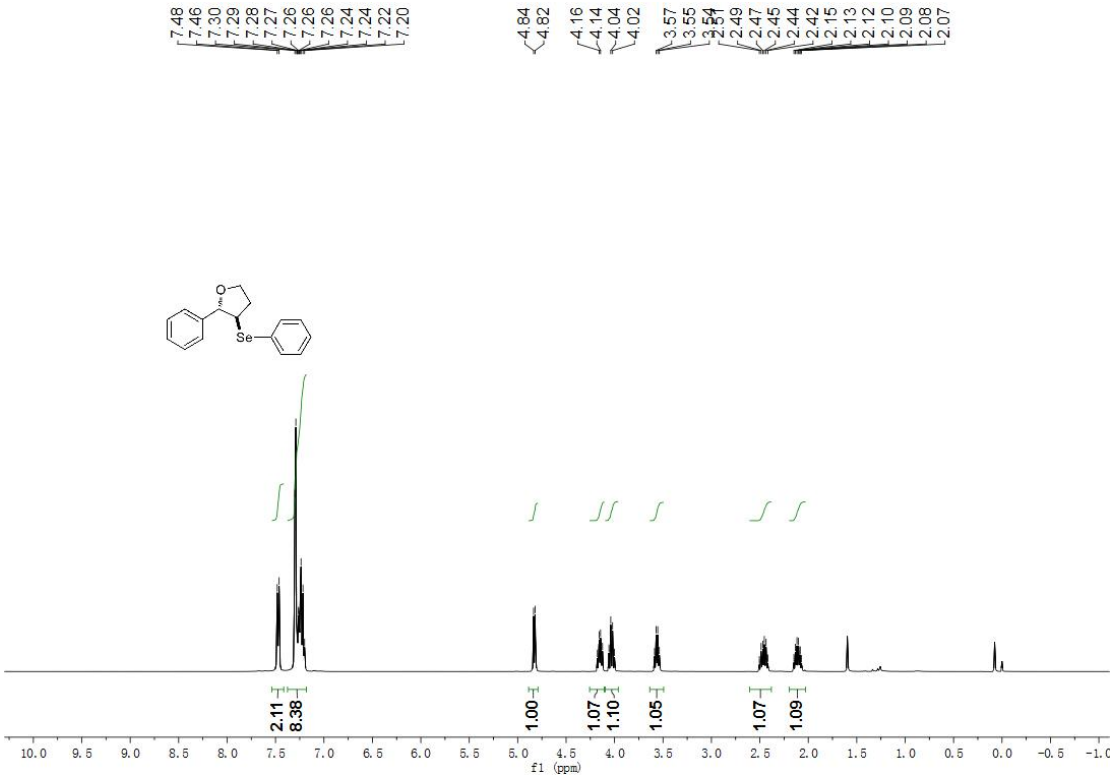
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4a**



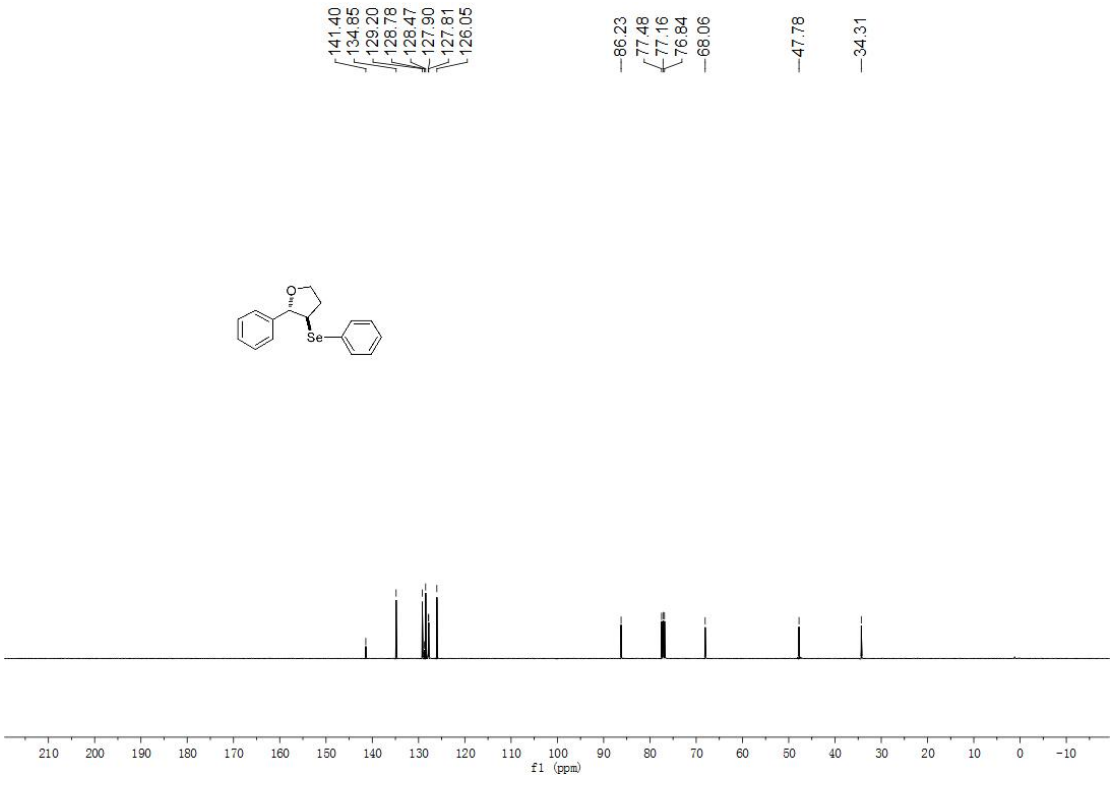
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4a**



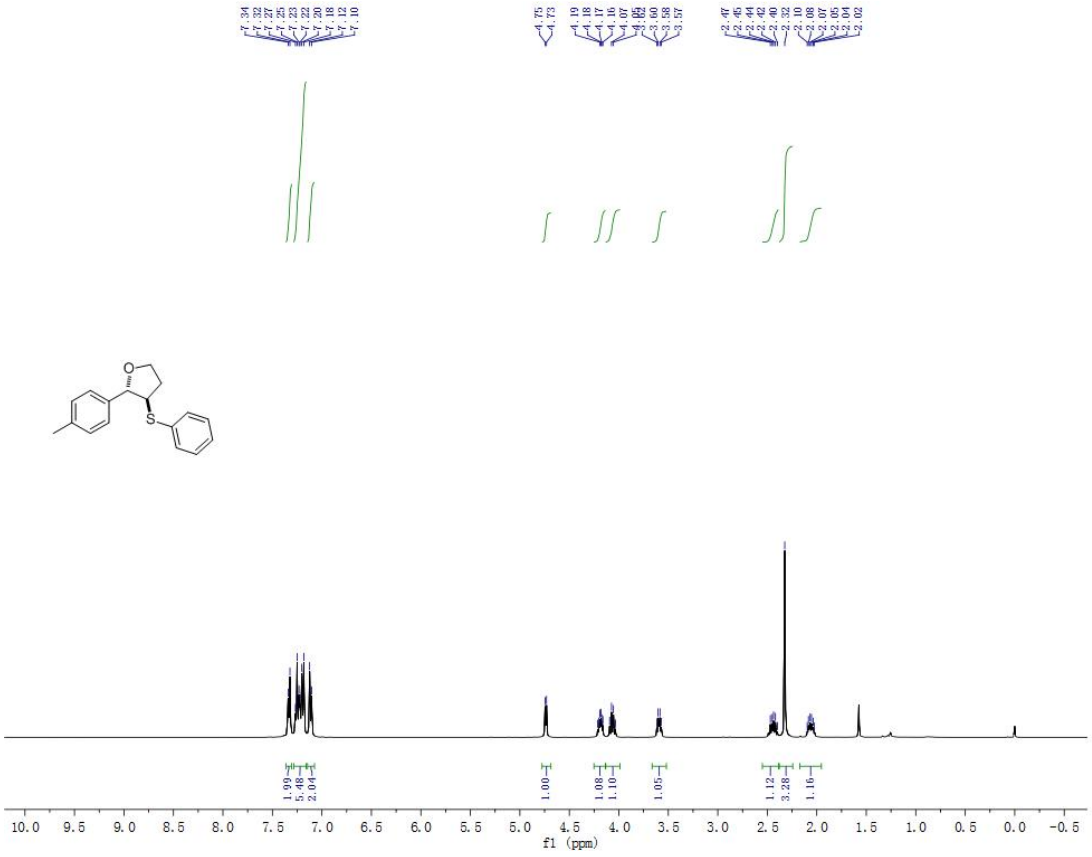
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4a'**



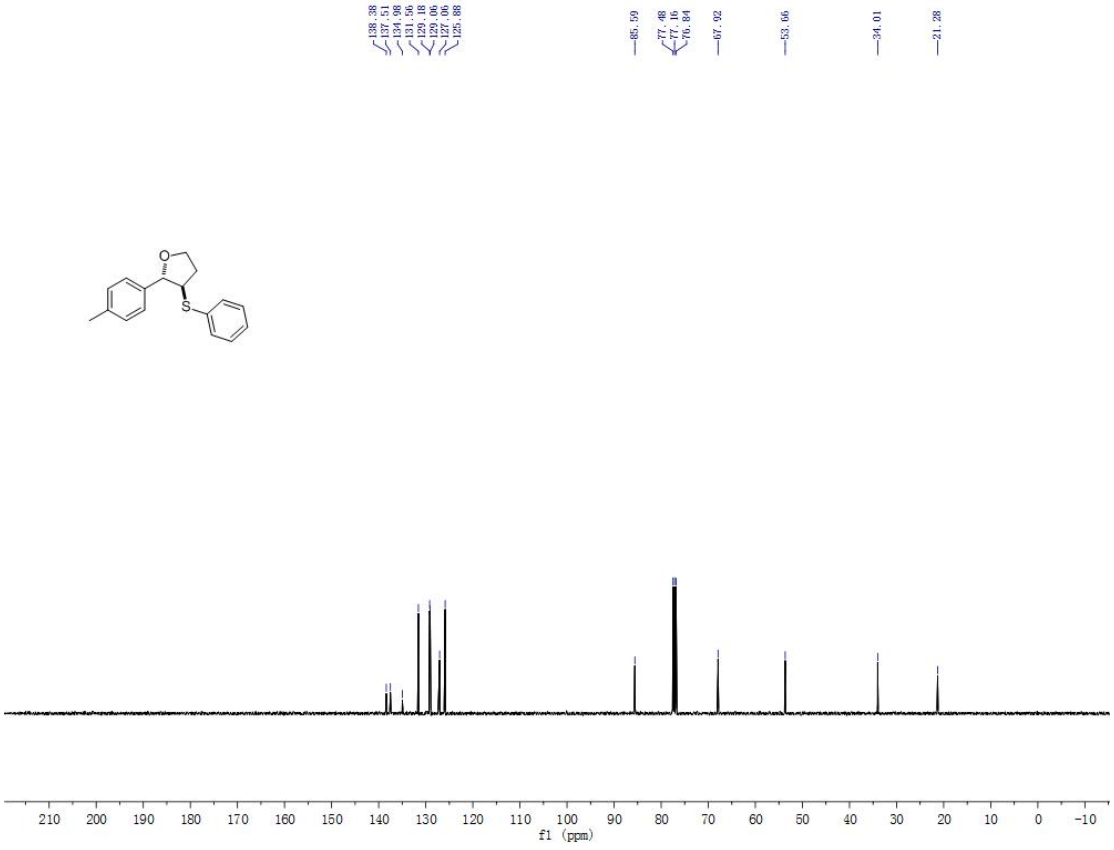
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4a'**



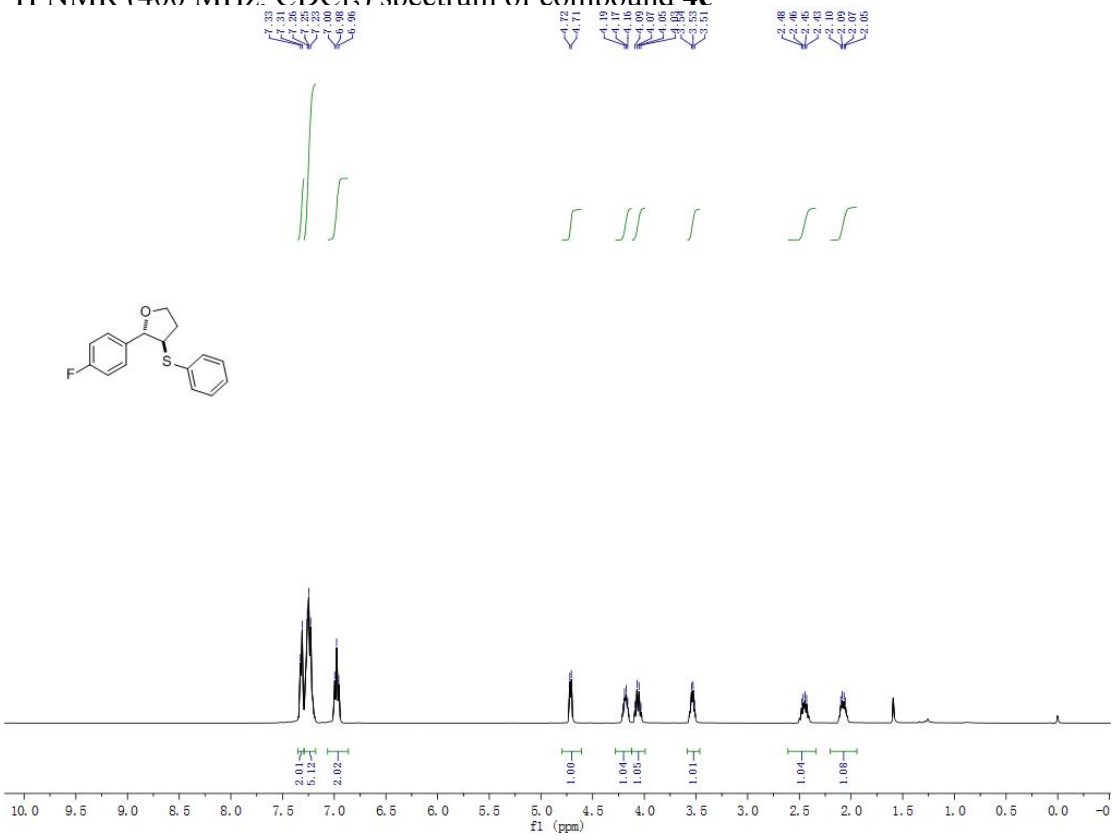
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4b**



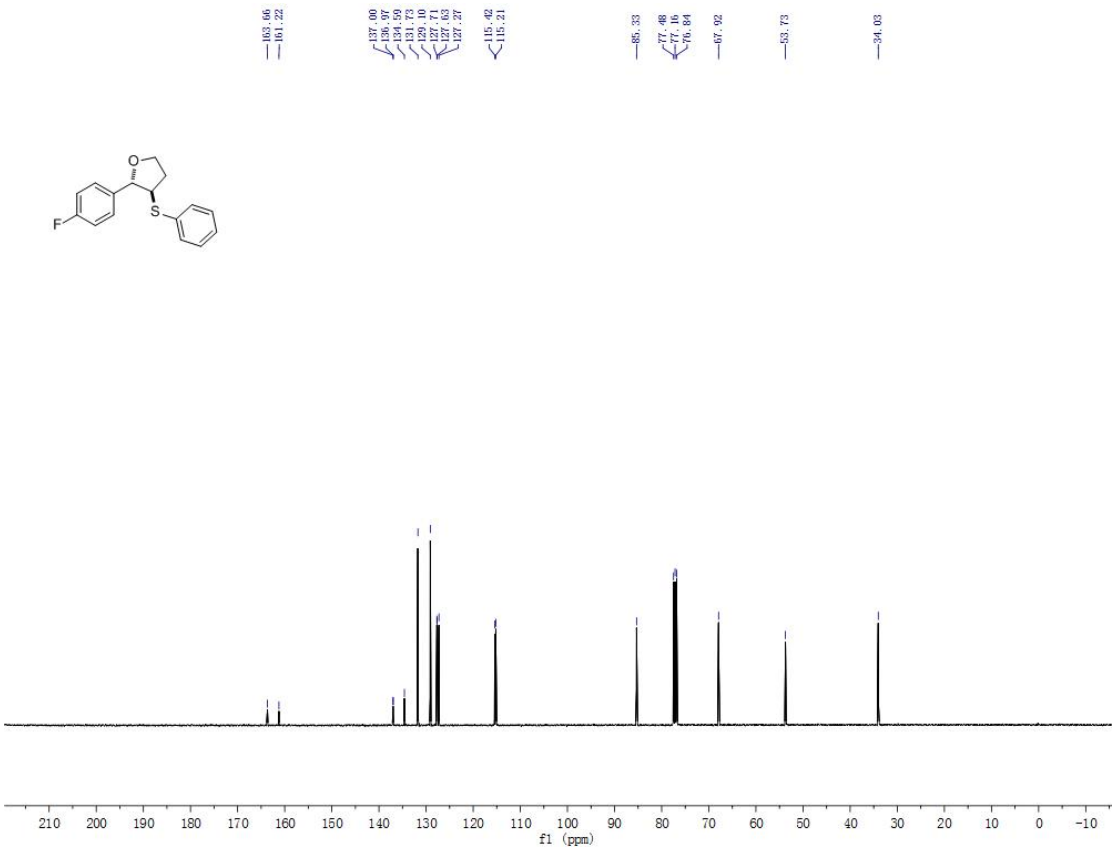
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4b**



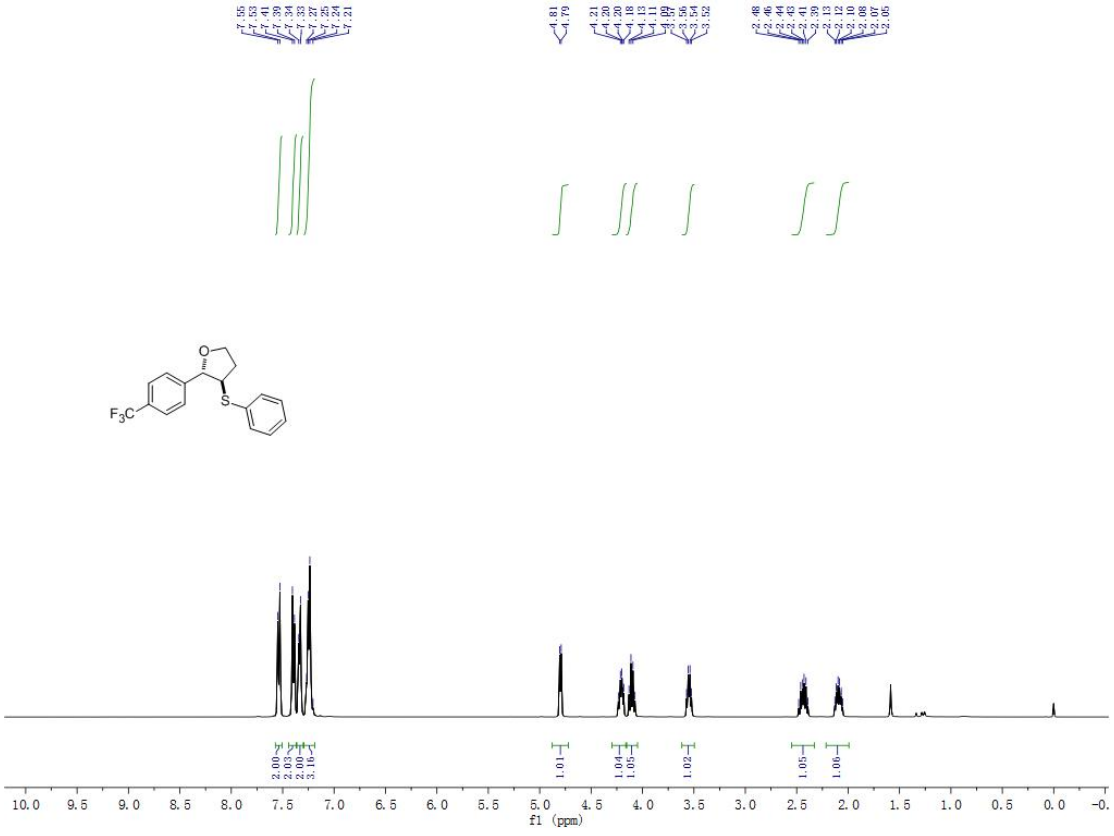
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4c**



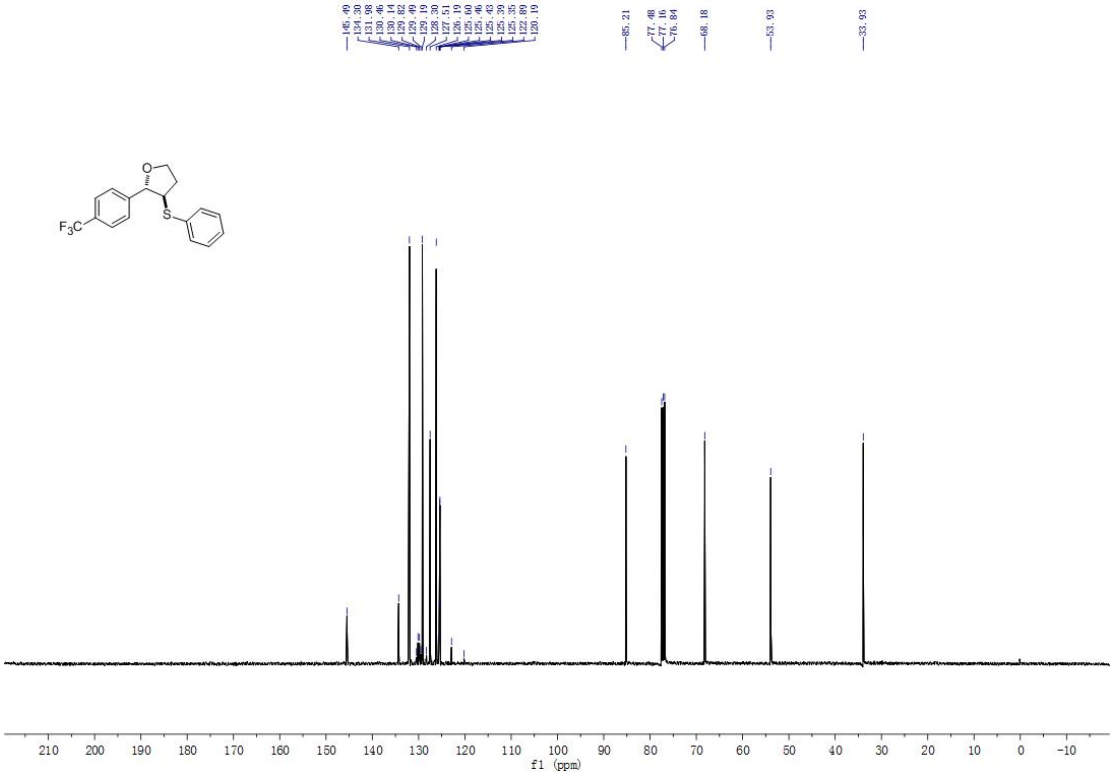
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4c**



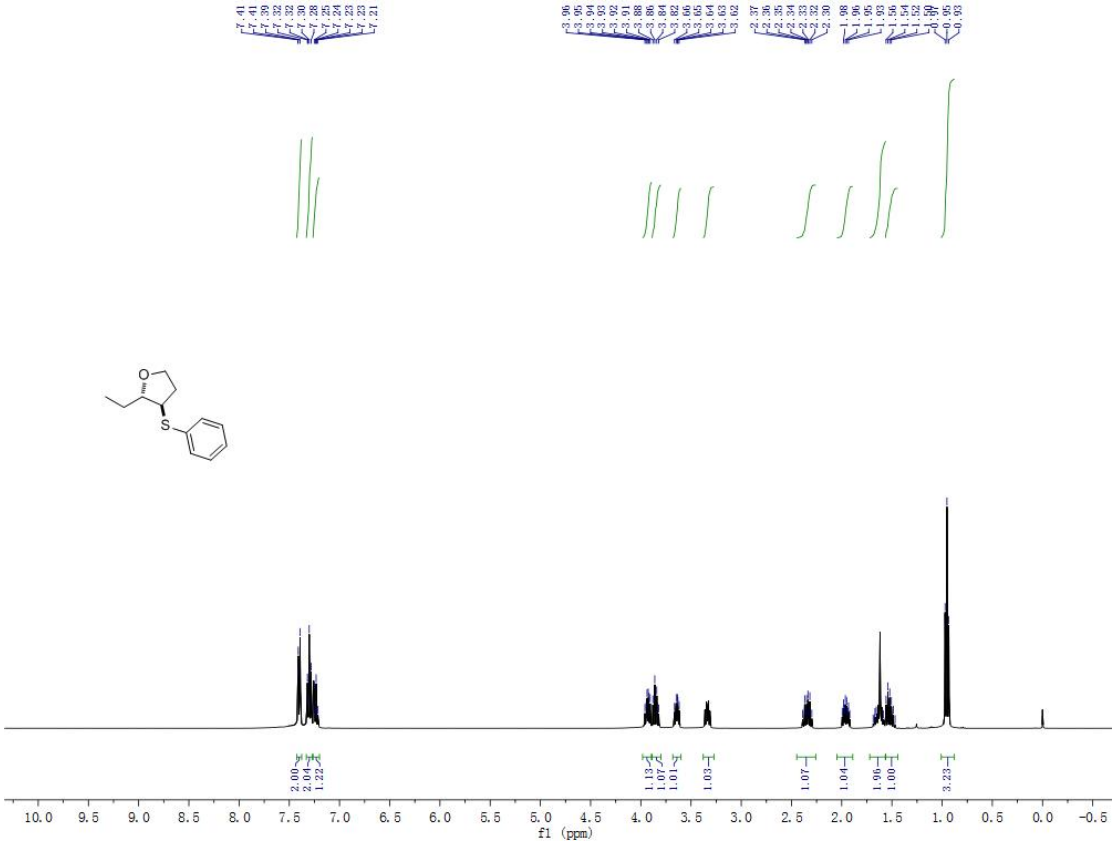
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4d**



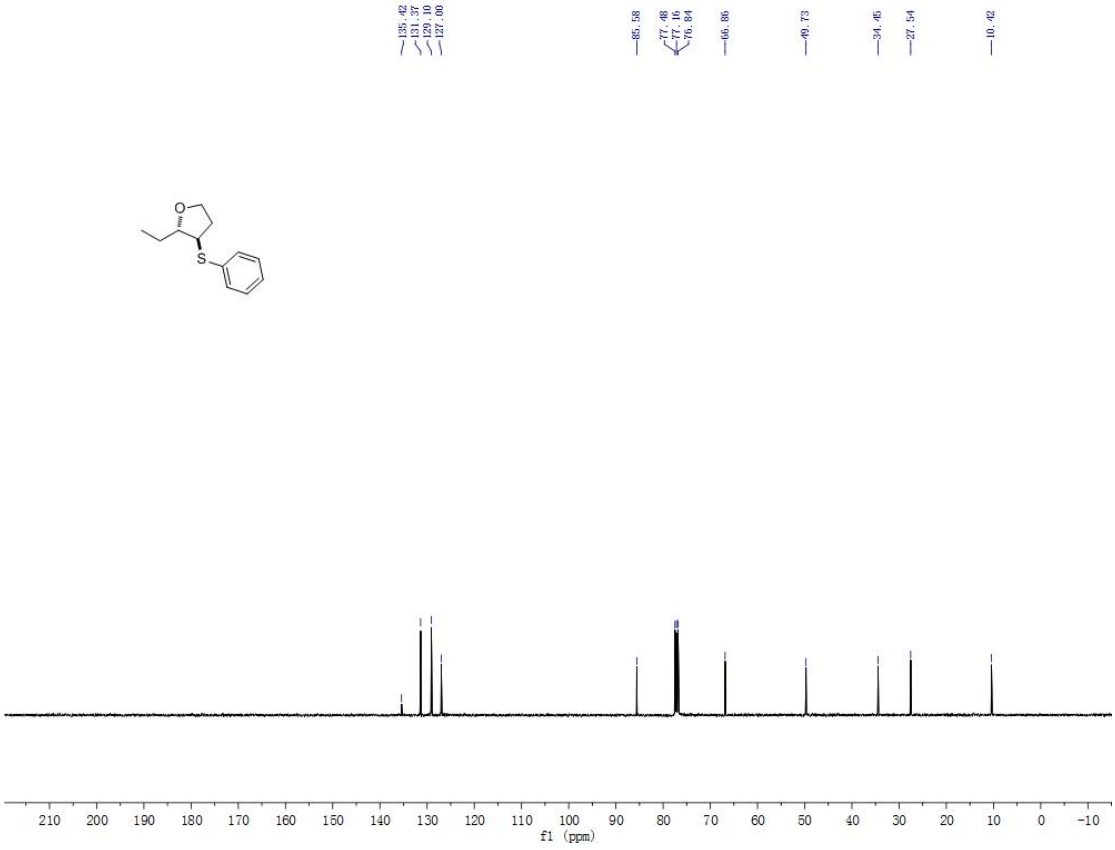
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4d**



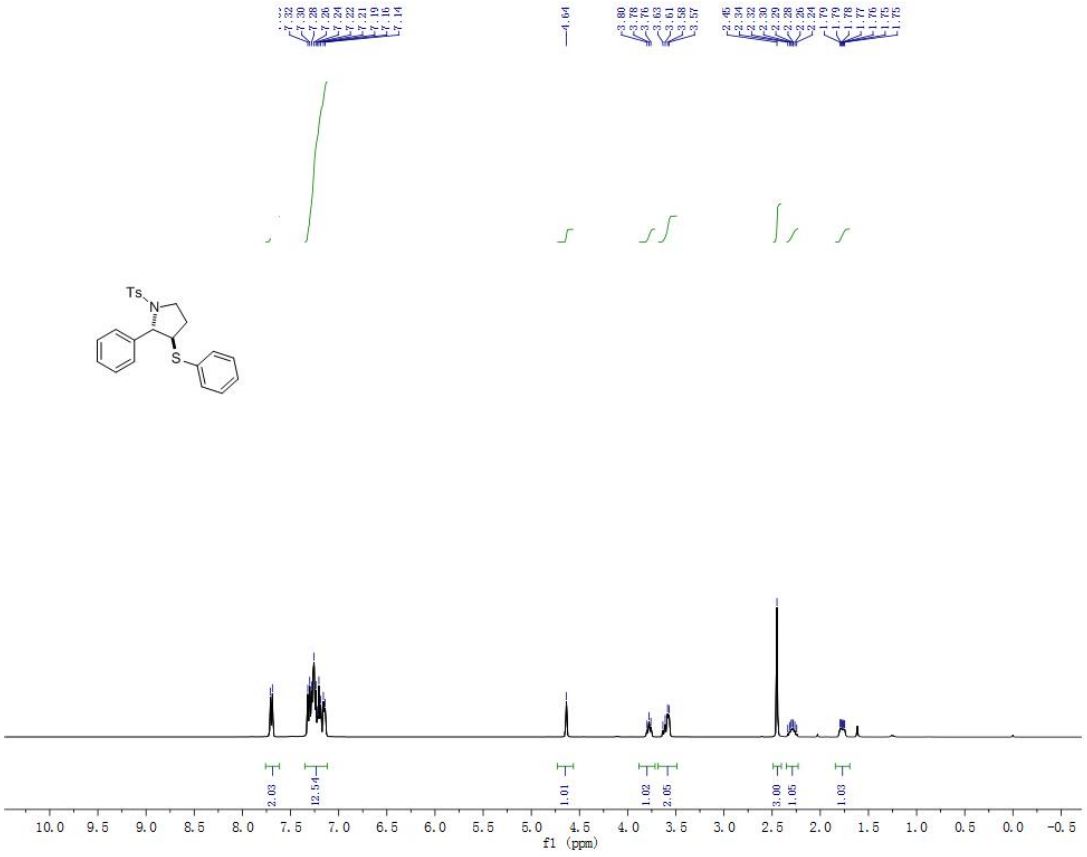
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4e**



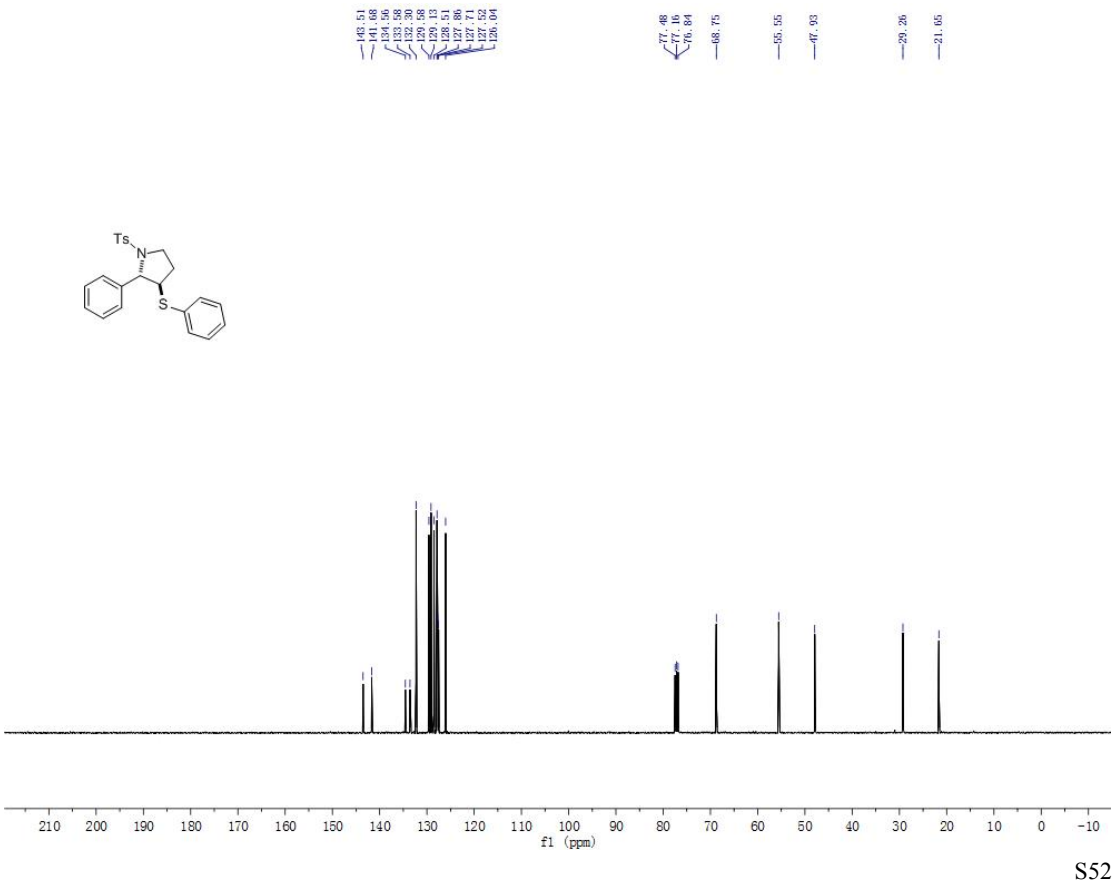
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4e**



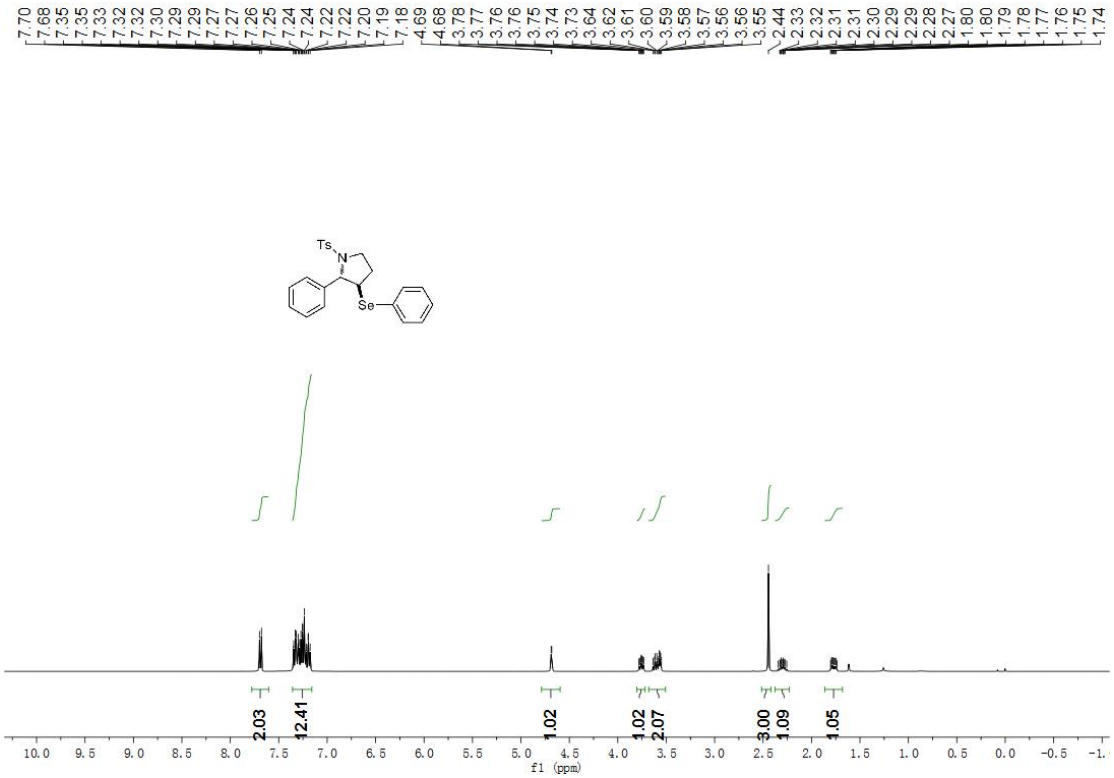
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4f**



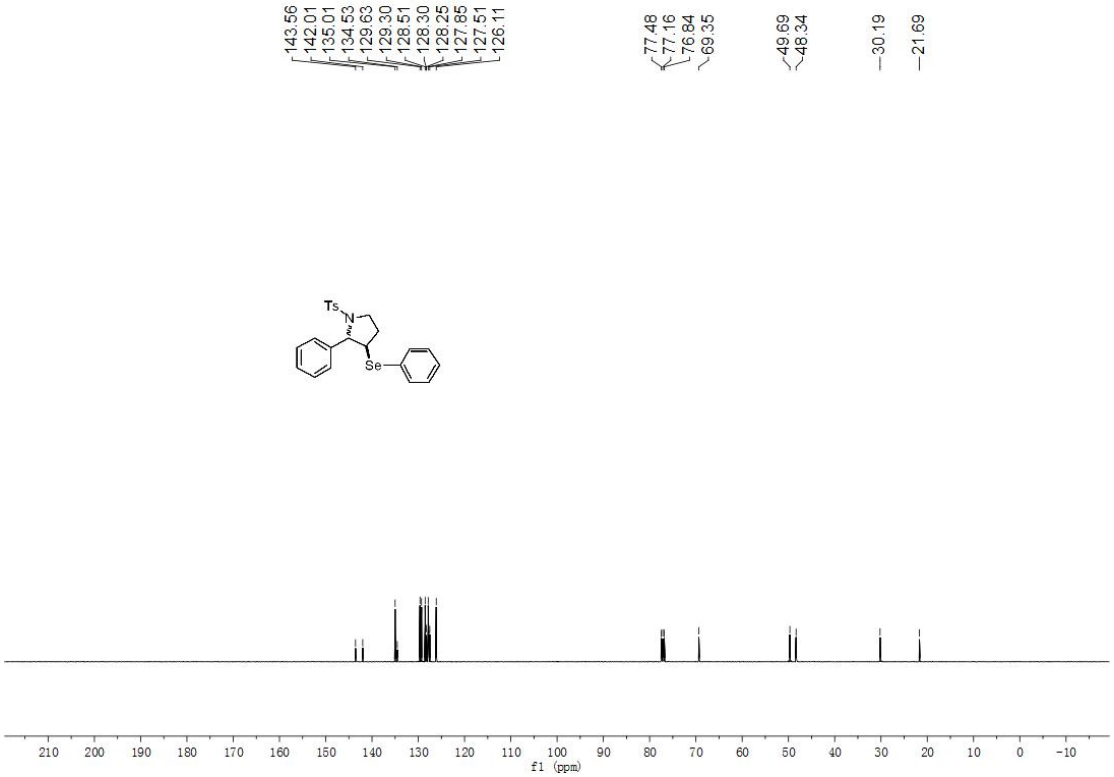
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4f**



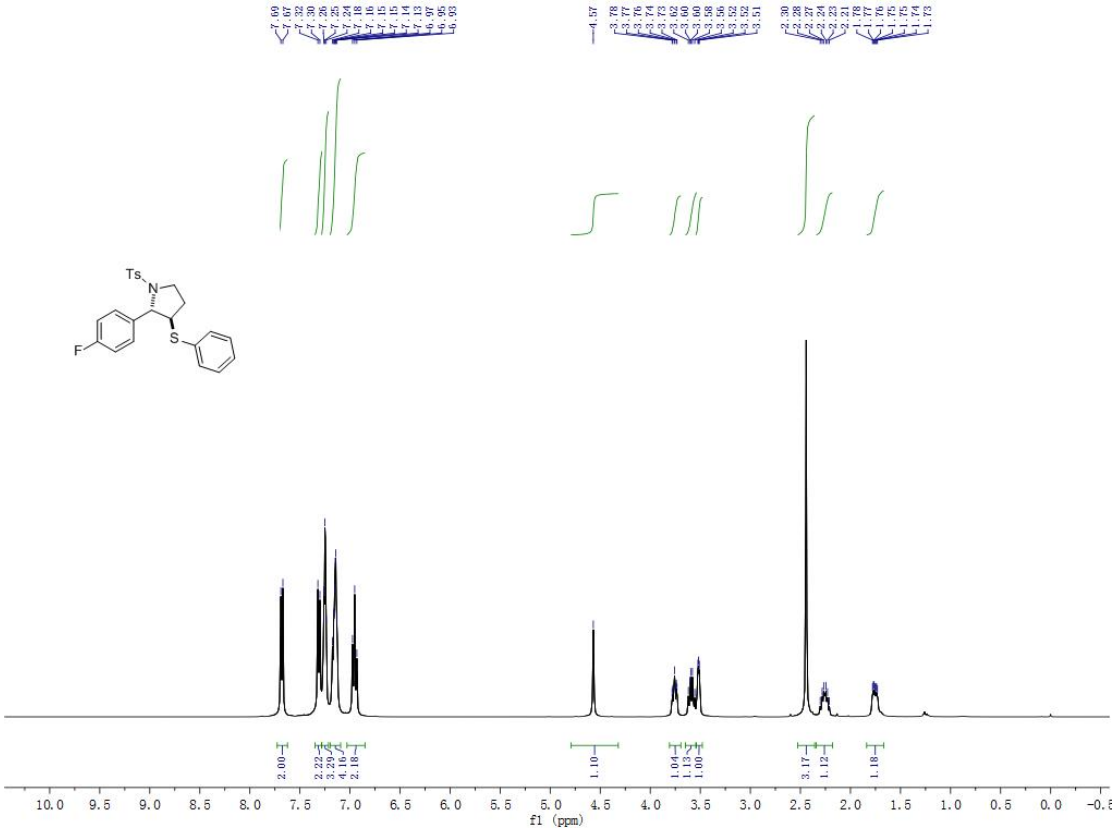
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4f'**



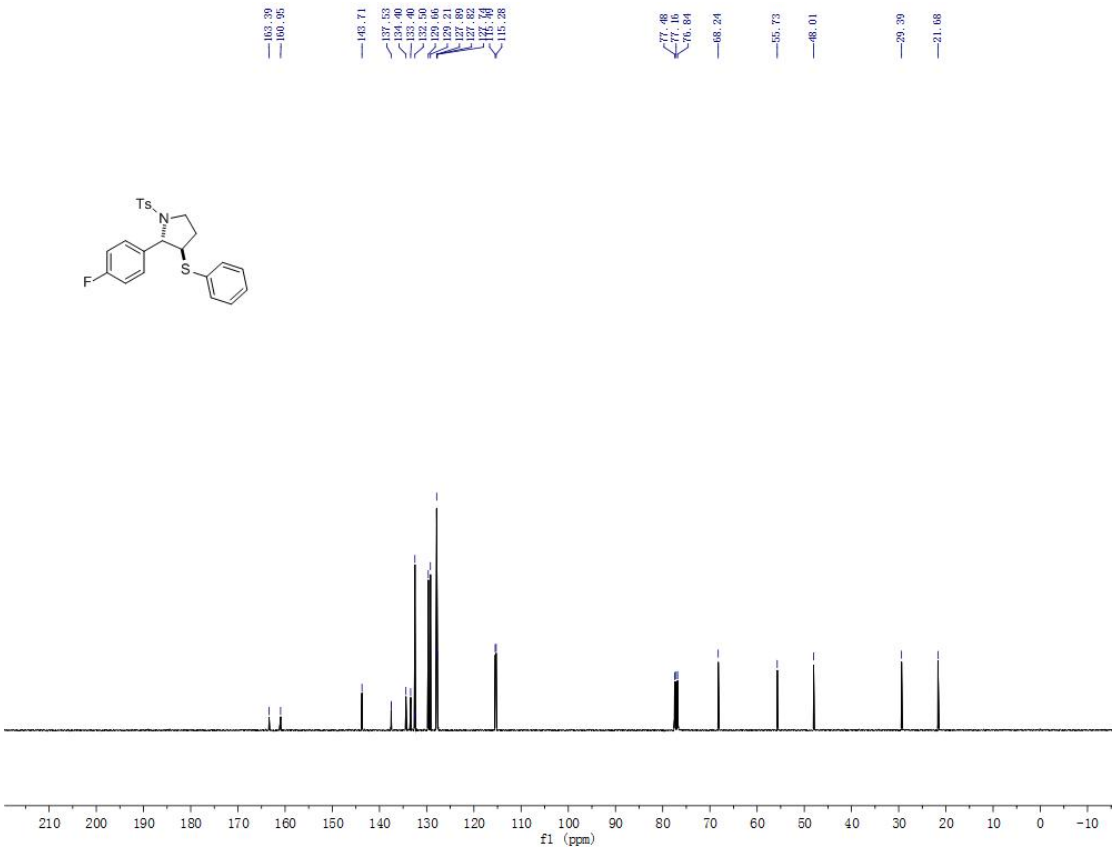
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4f'**



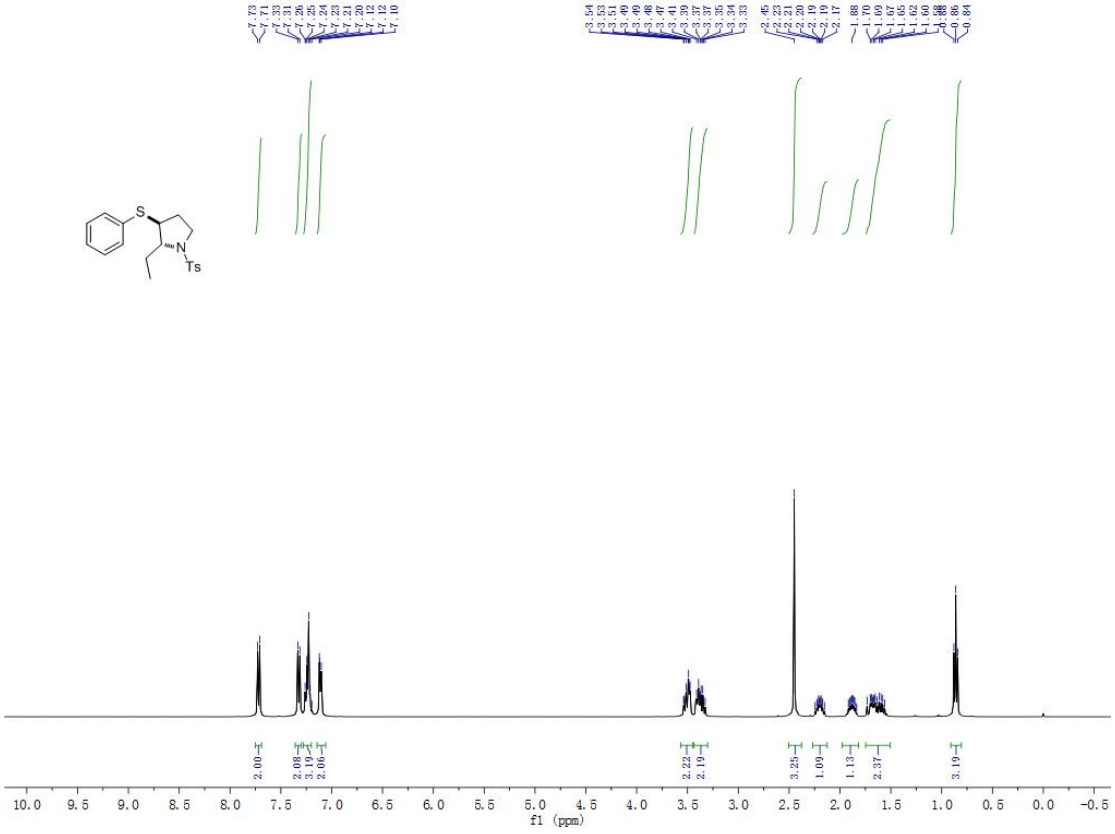
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4g**



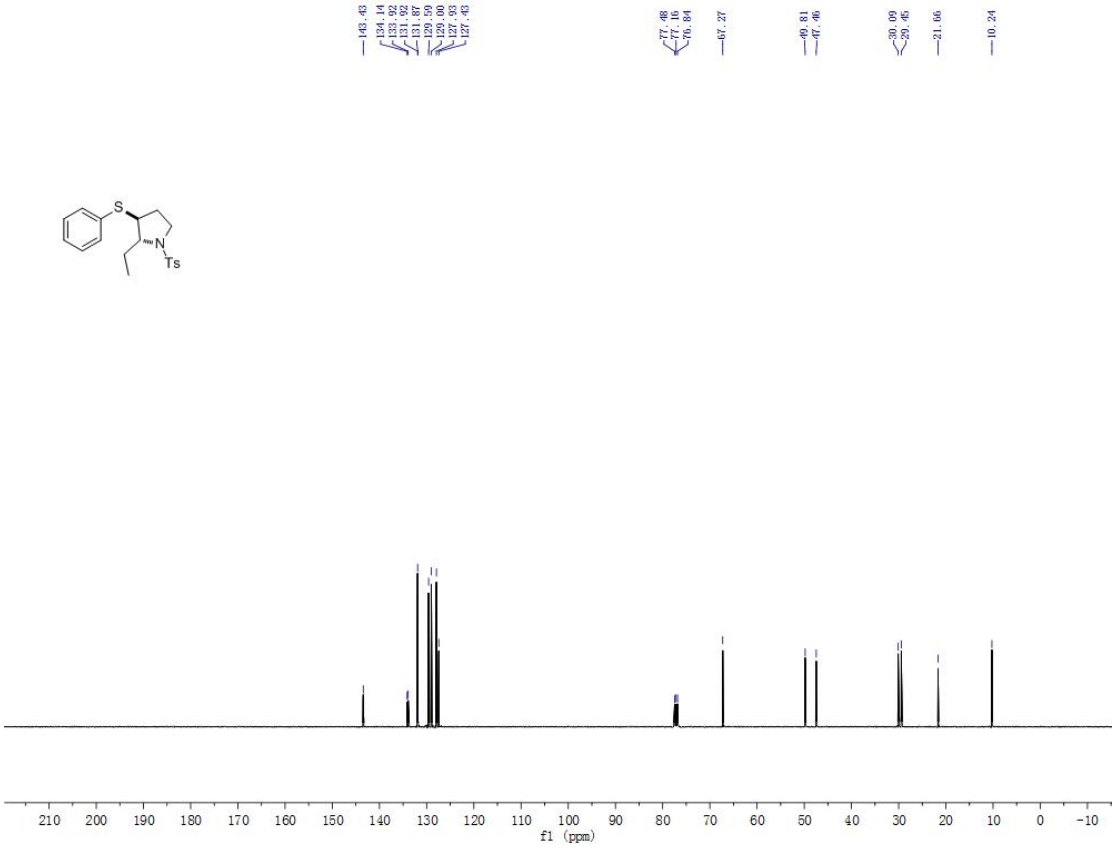
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4g**



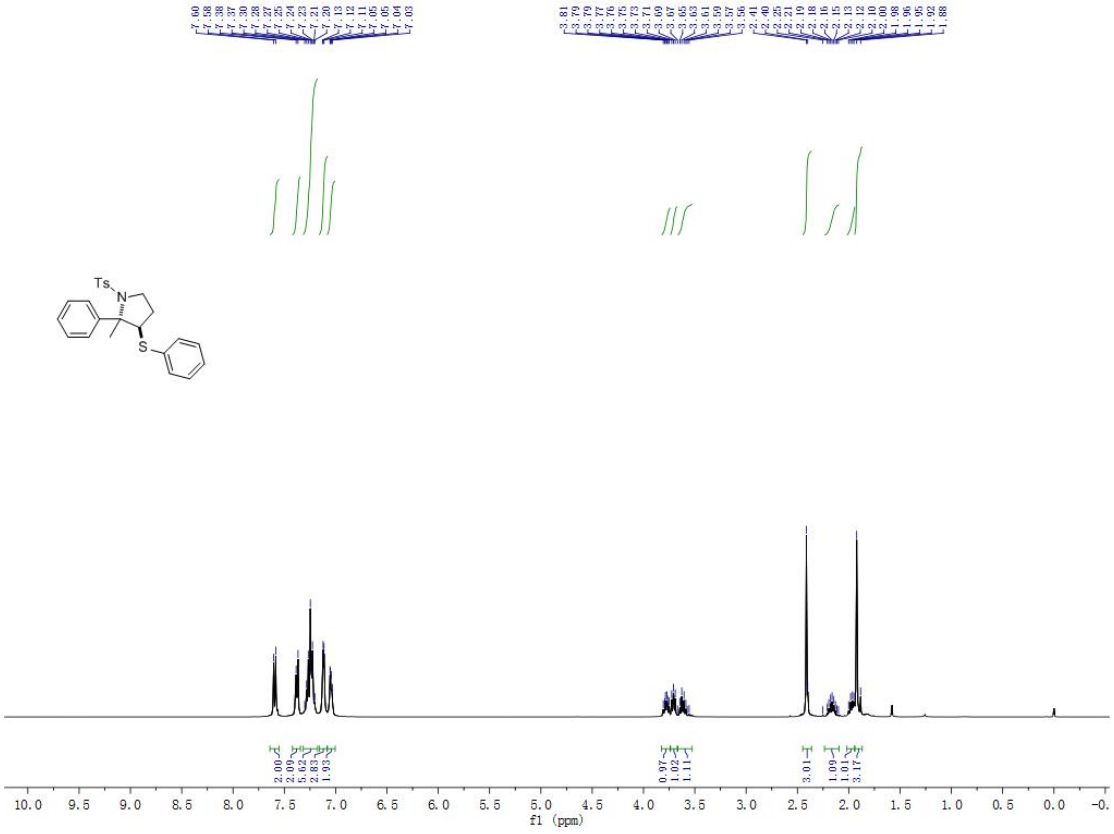
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4h**



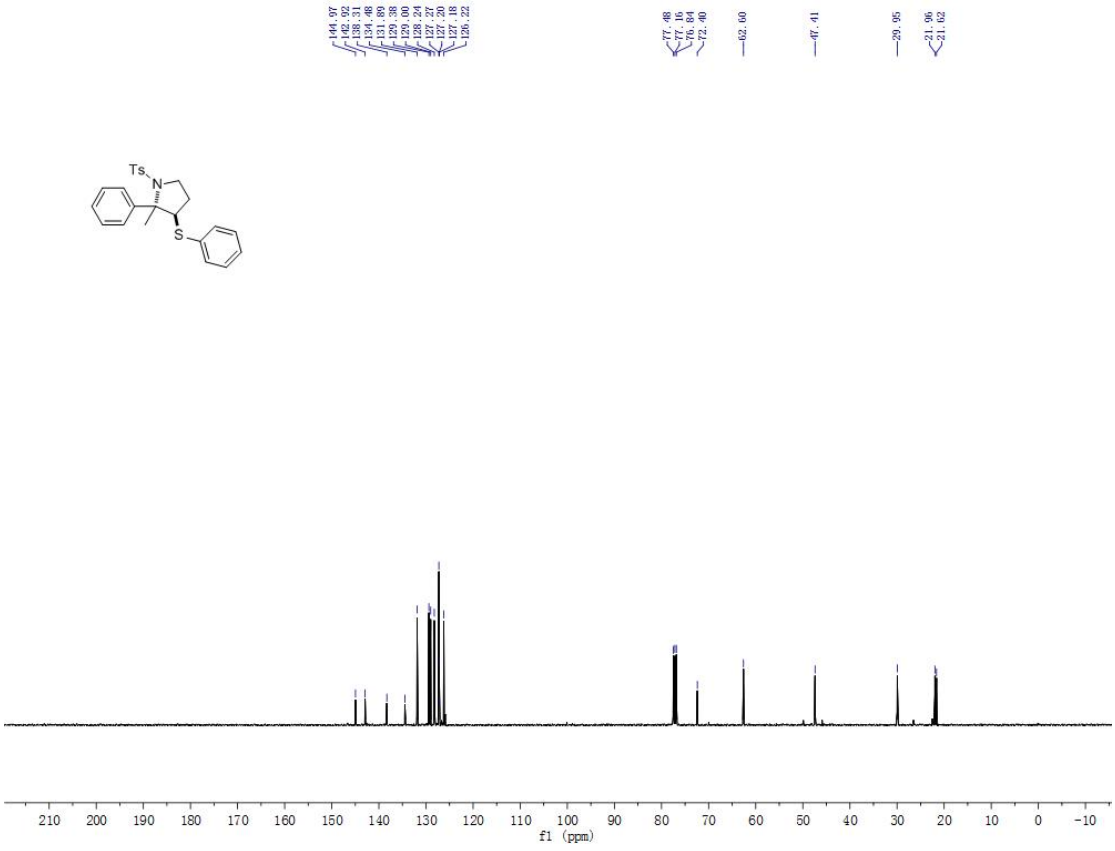
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4h**



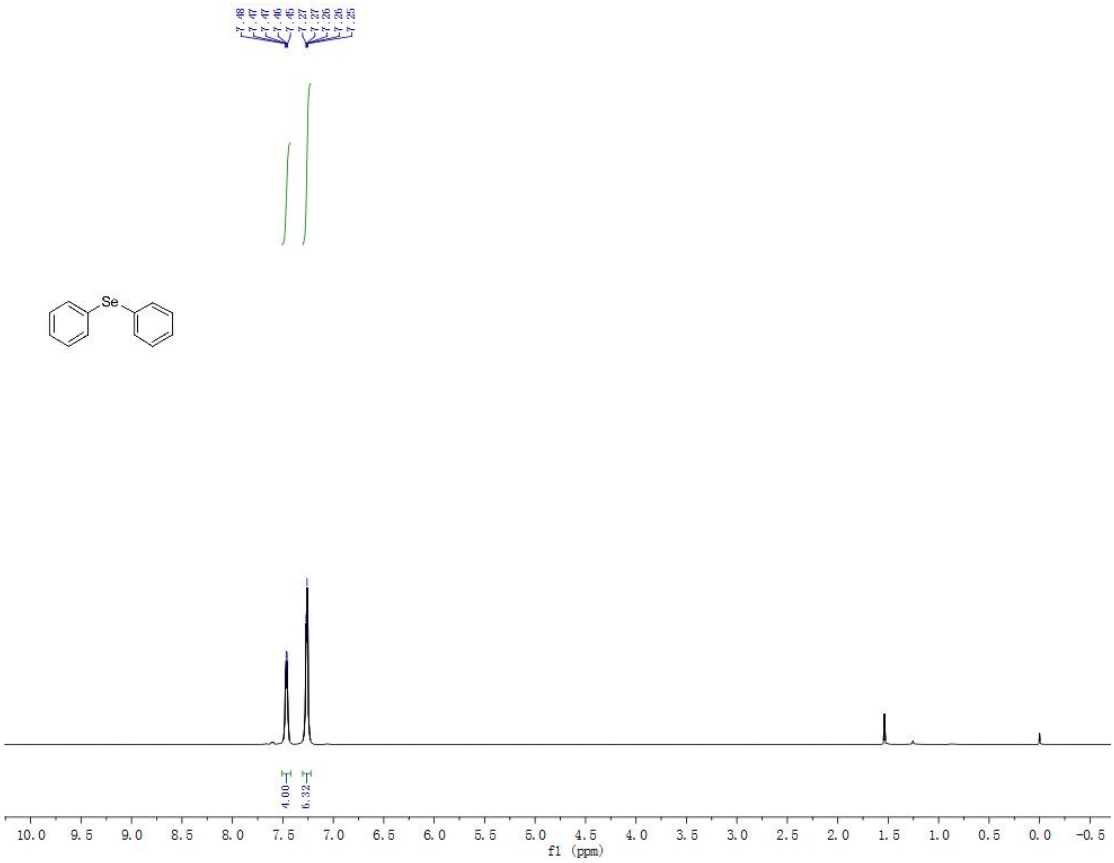
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **4i**



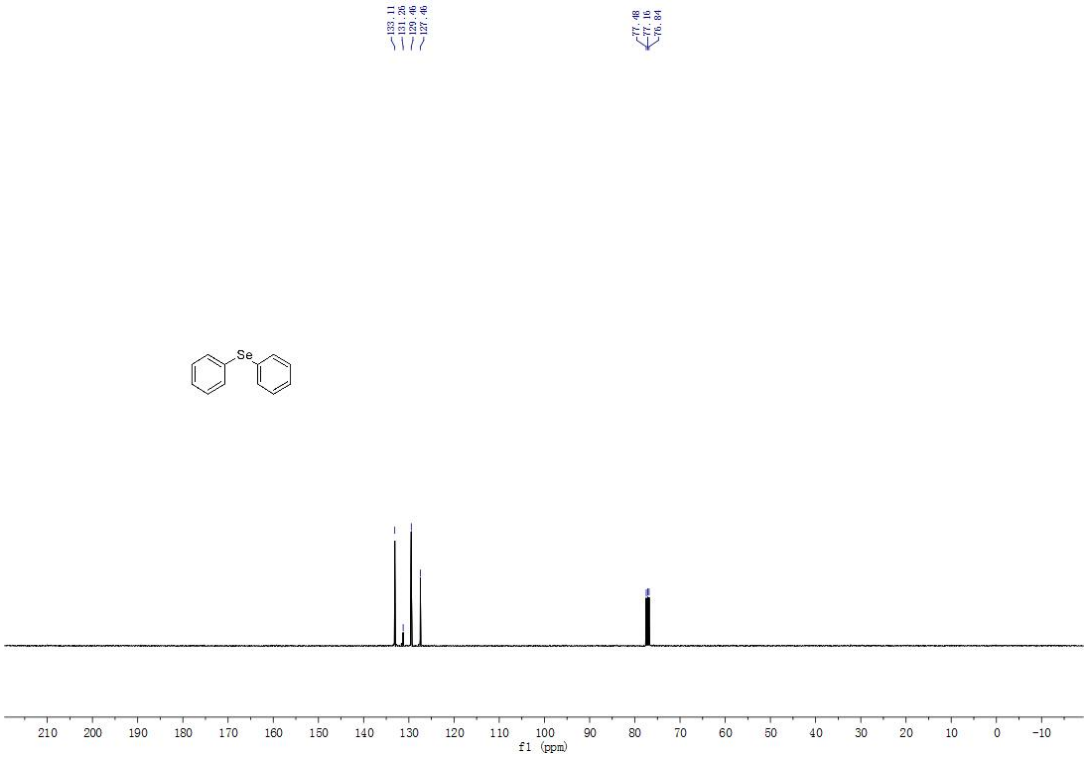
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **4i**



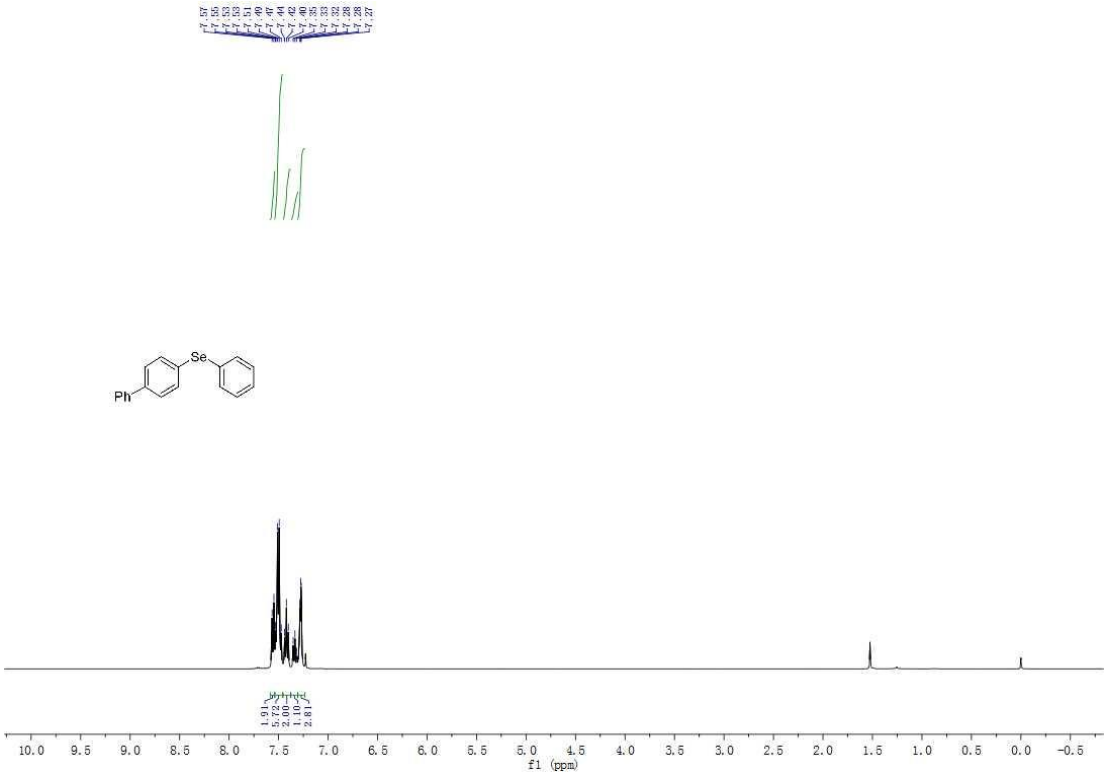
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6a**



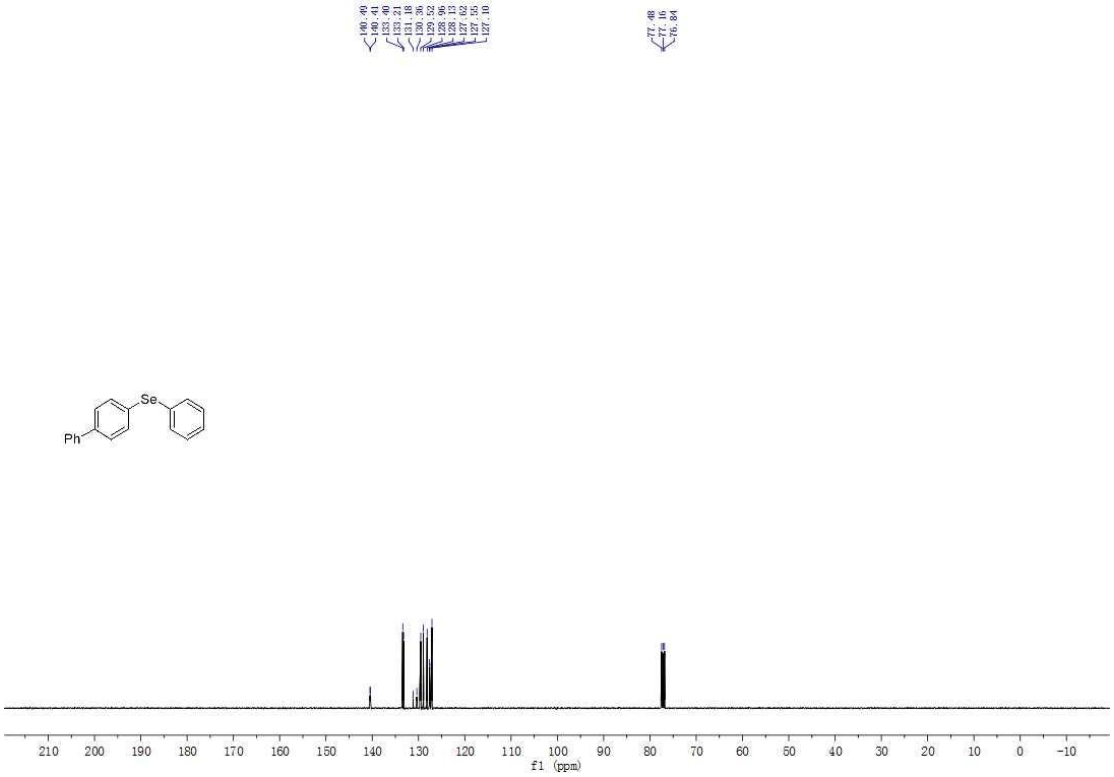
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6a**



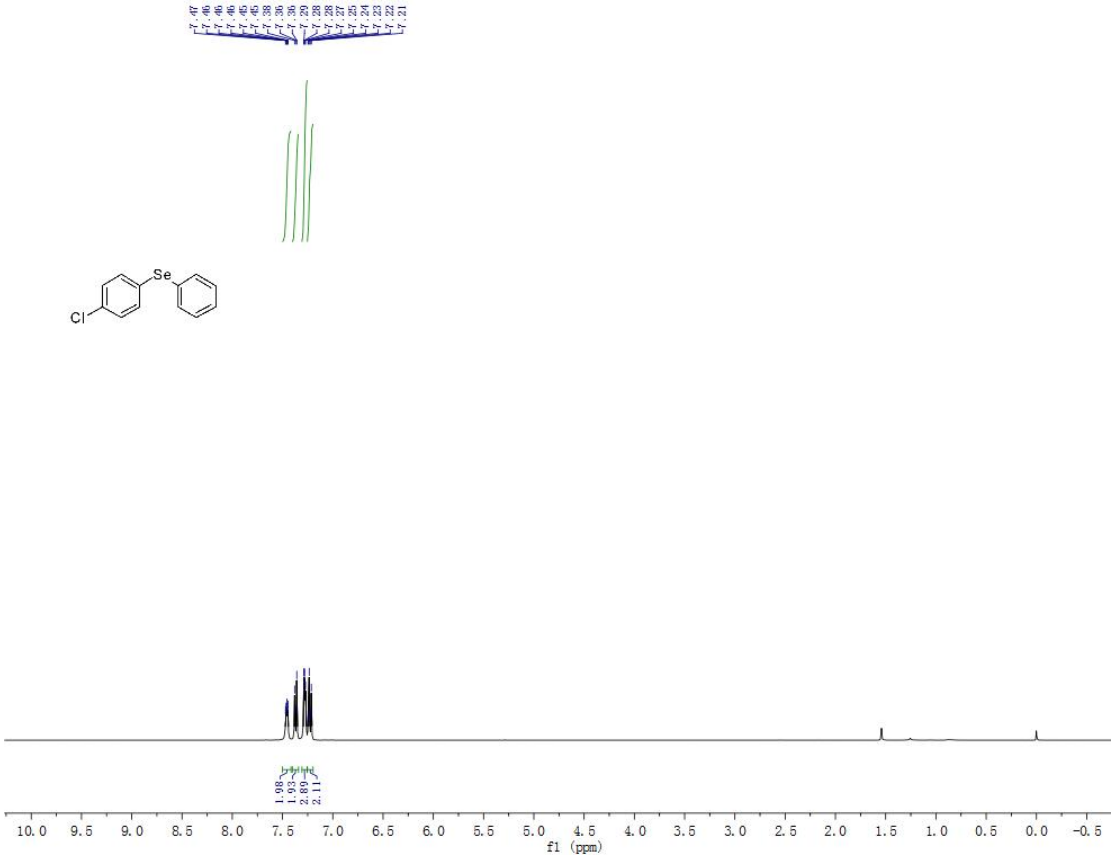
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6b**



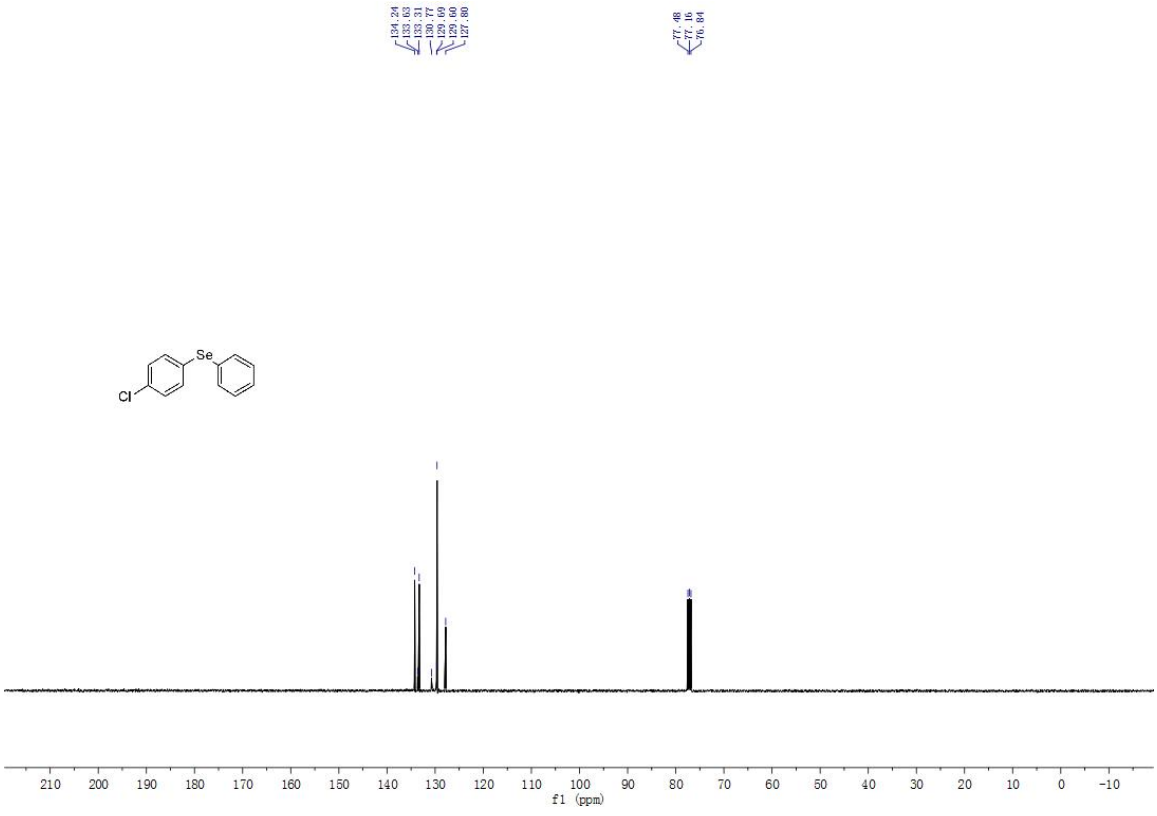
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6b**



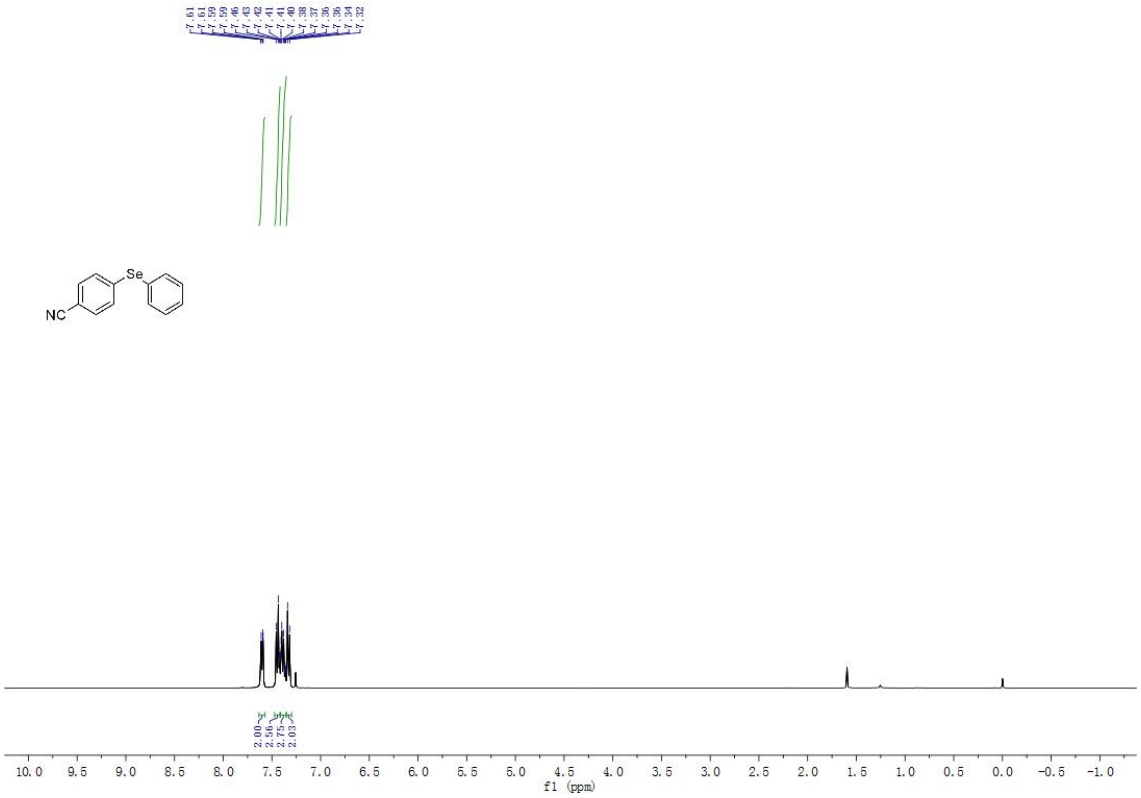
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6c**



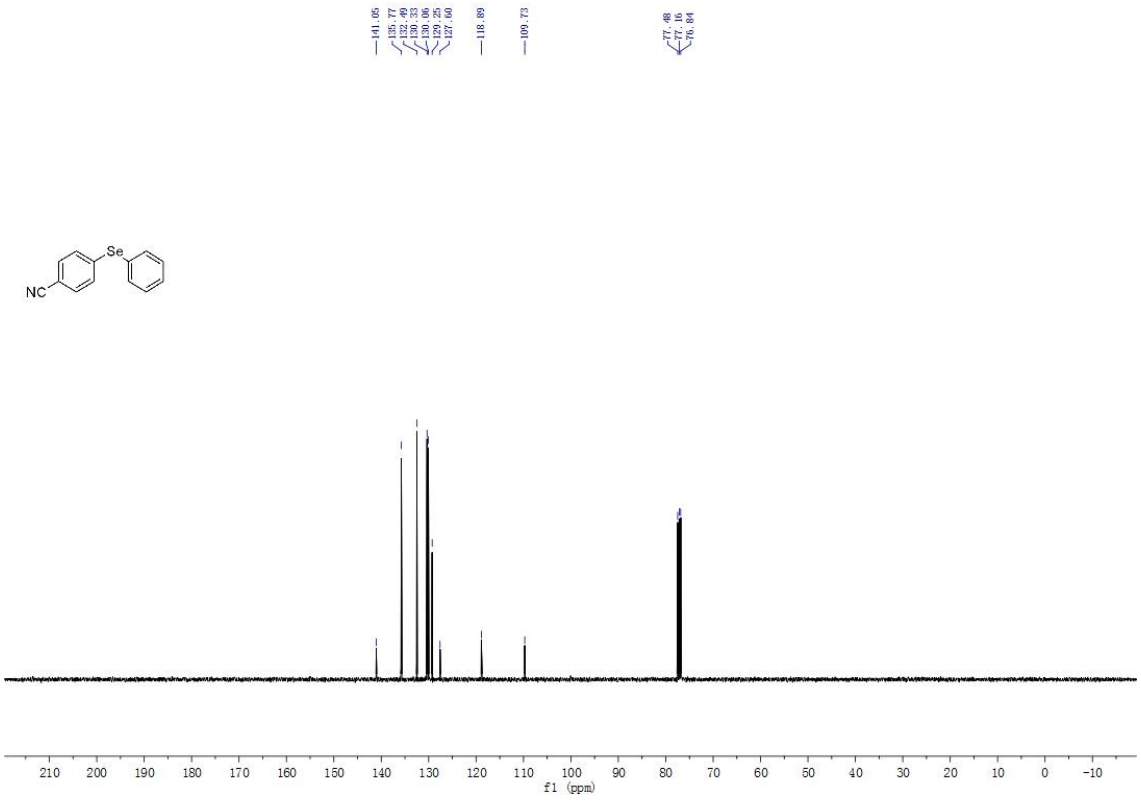
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6c**



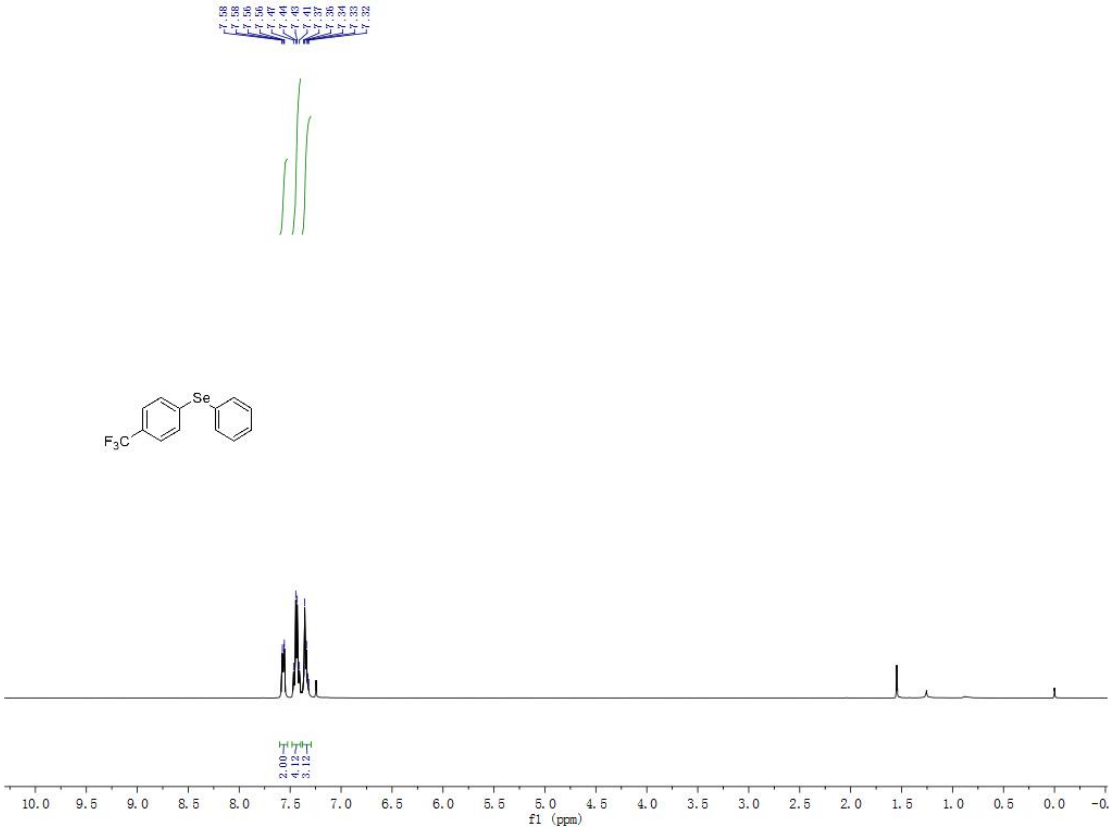
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound**6d**



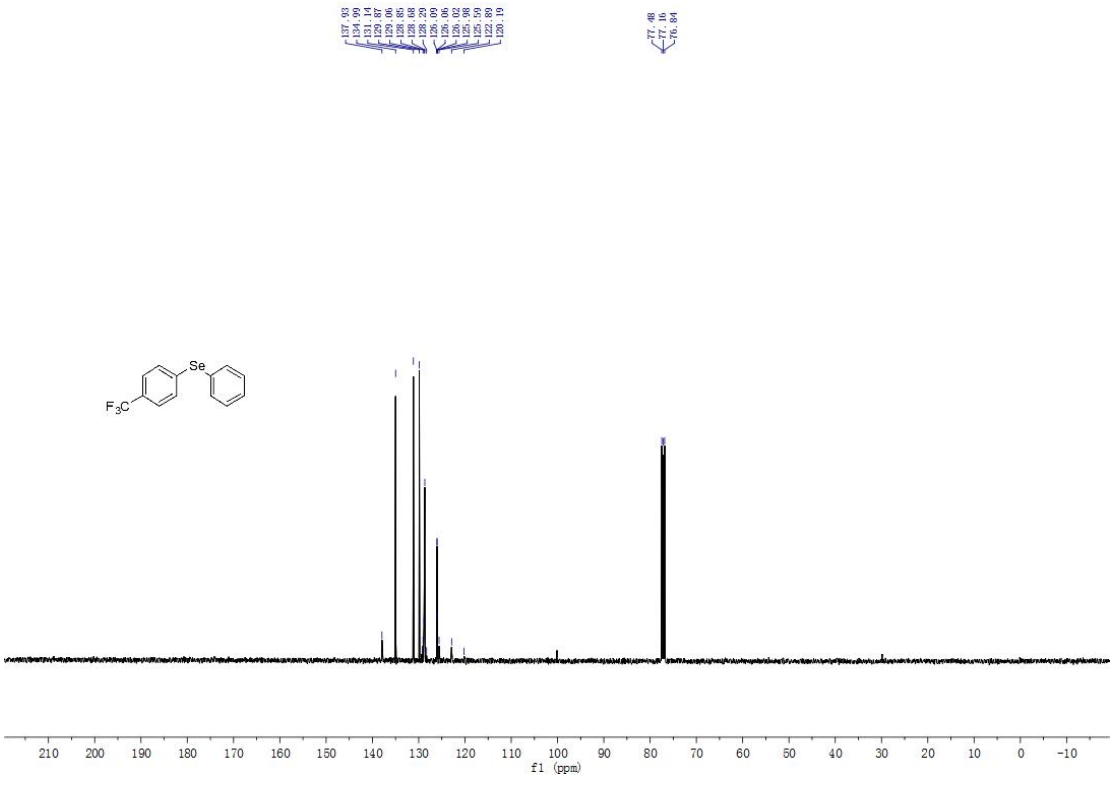
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6d**



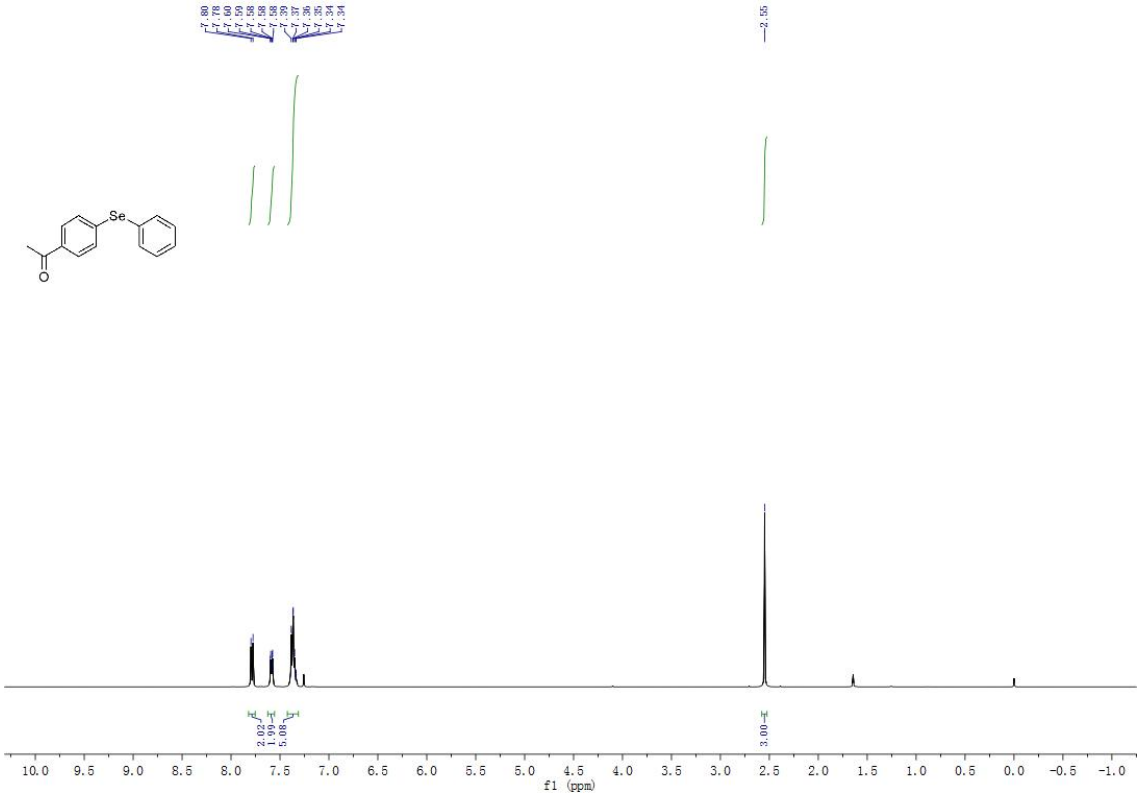
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6e**



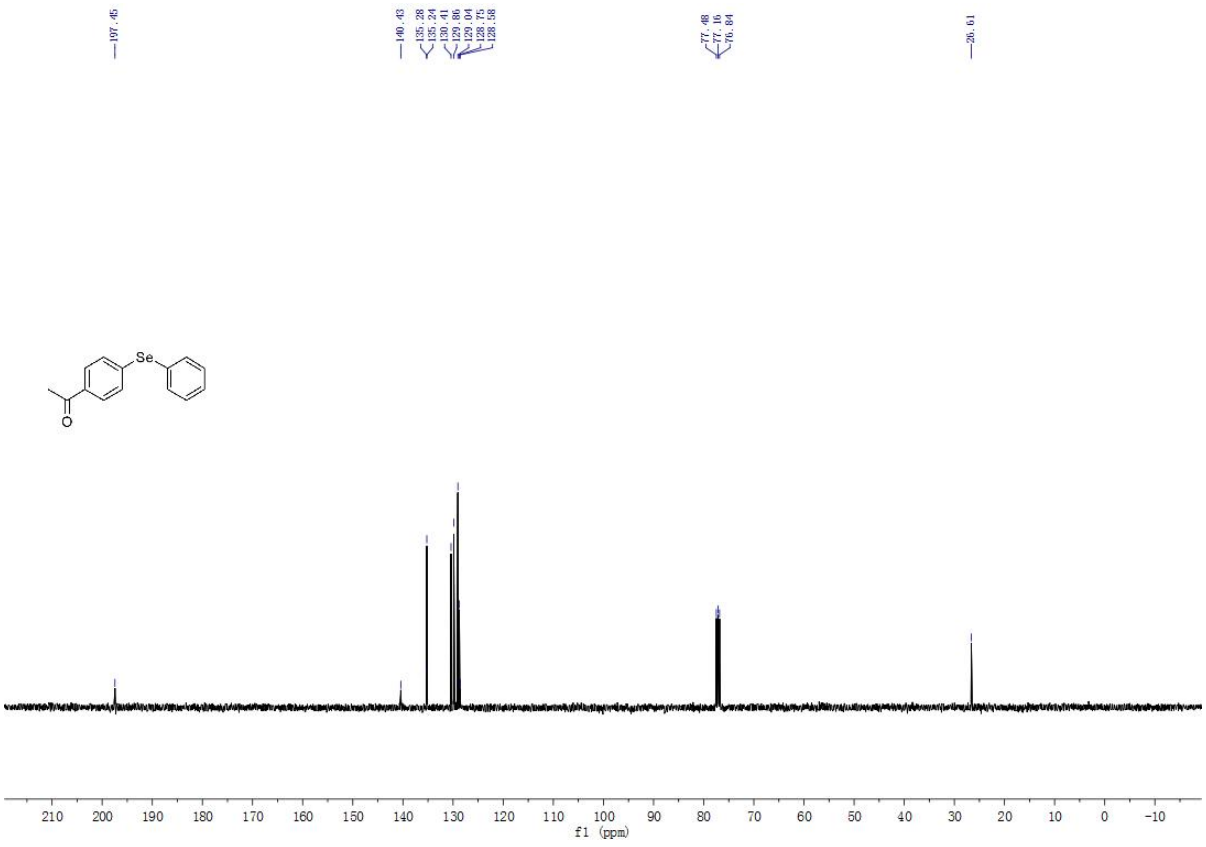
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6e**



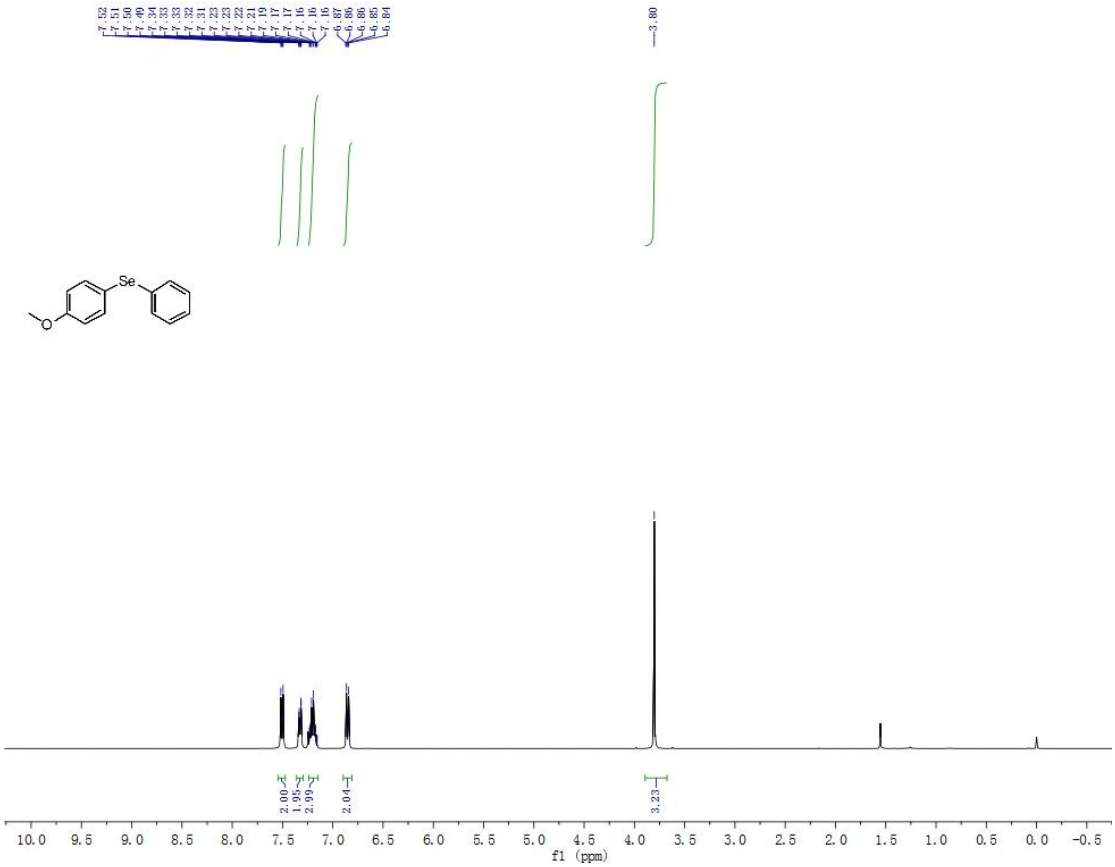
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6f**



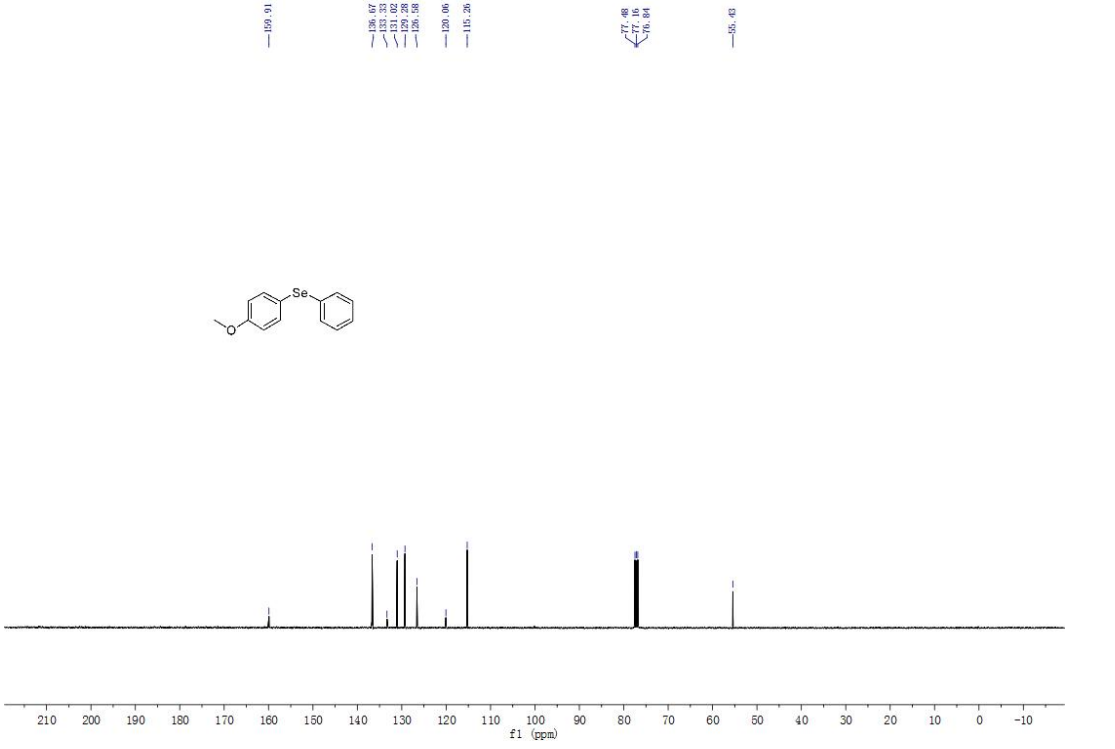
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6f**



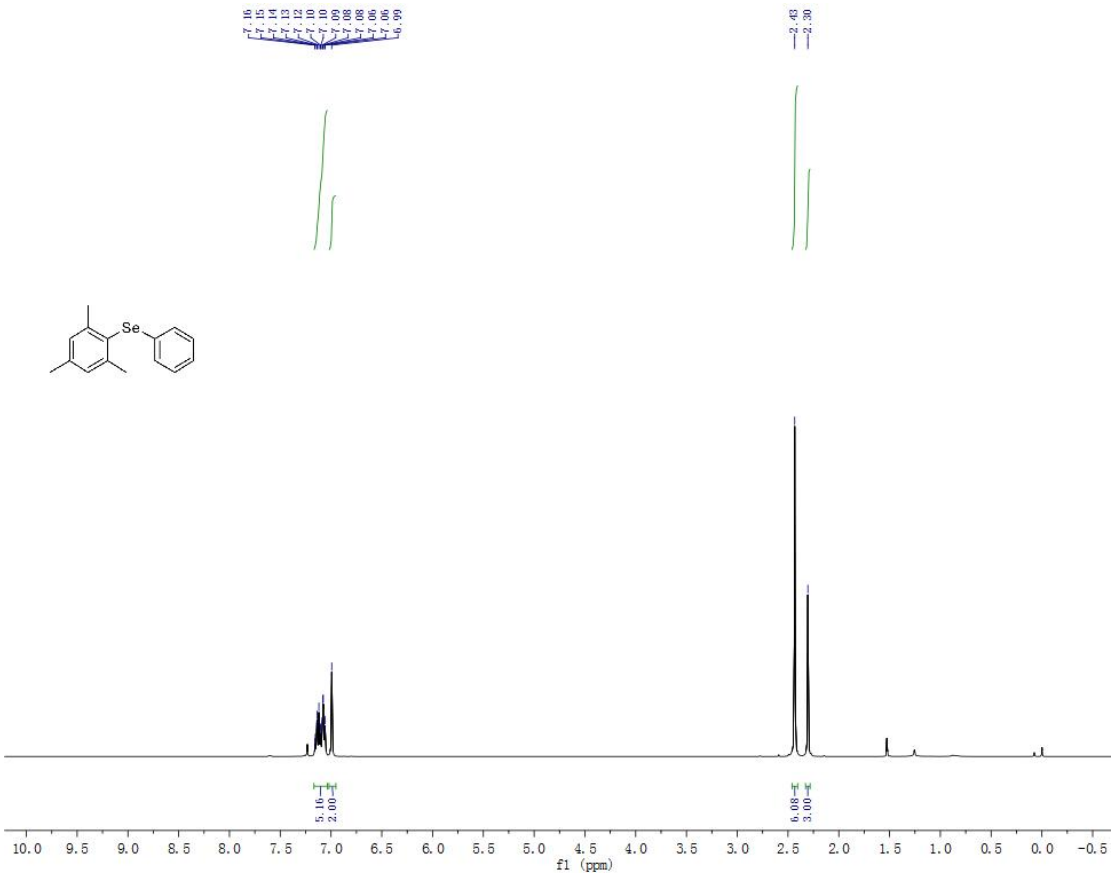
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6g**



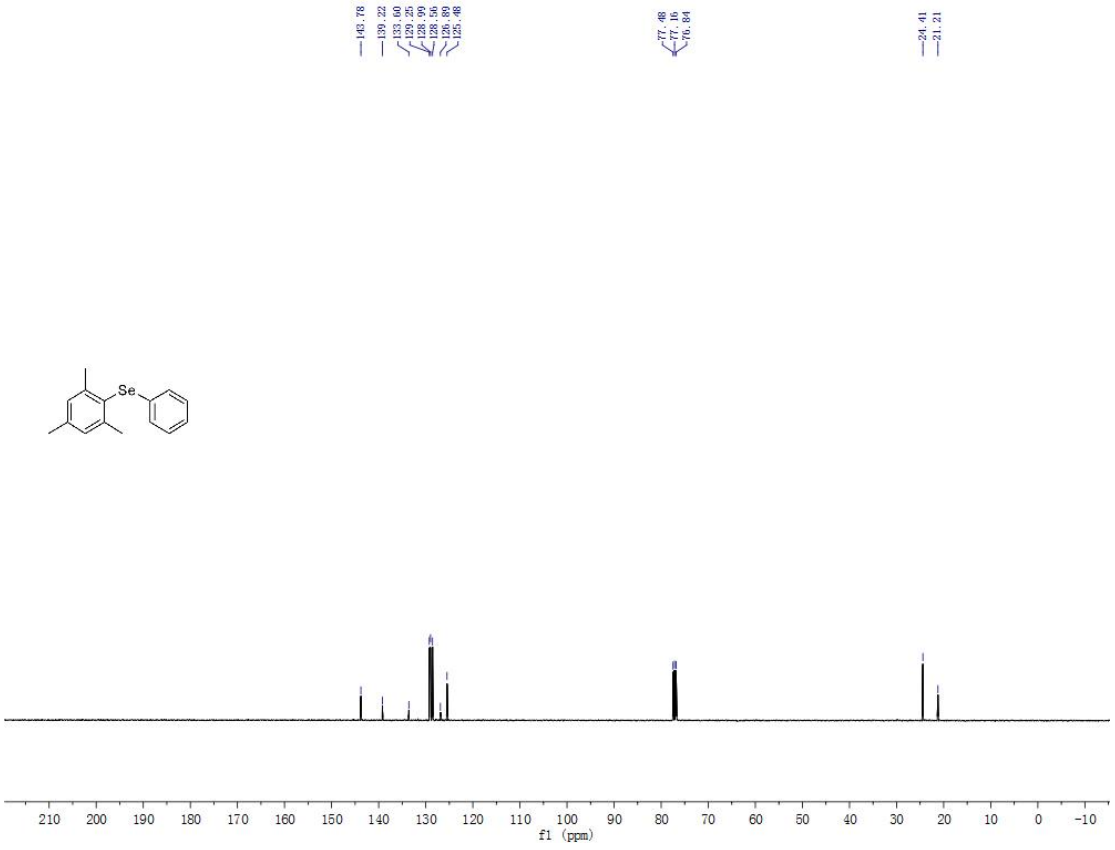
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6g**



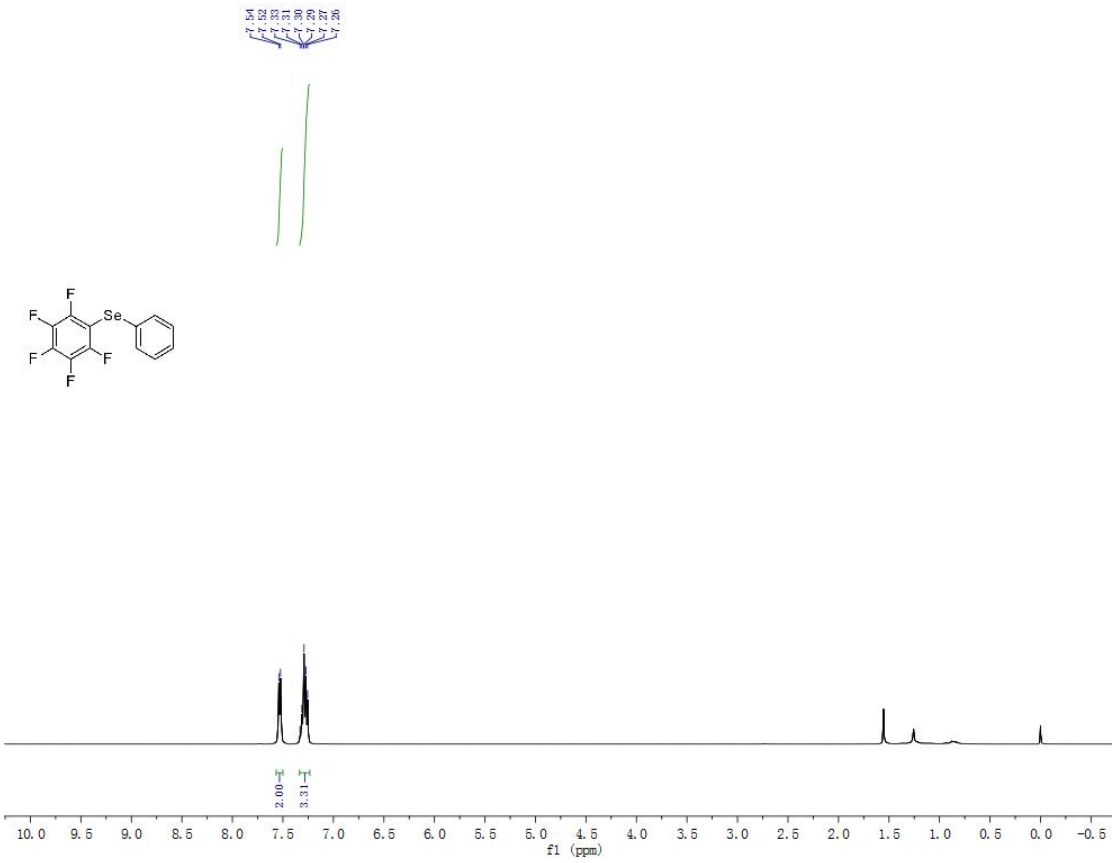
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6h**



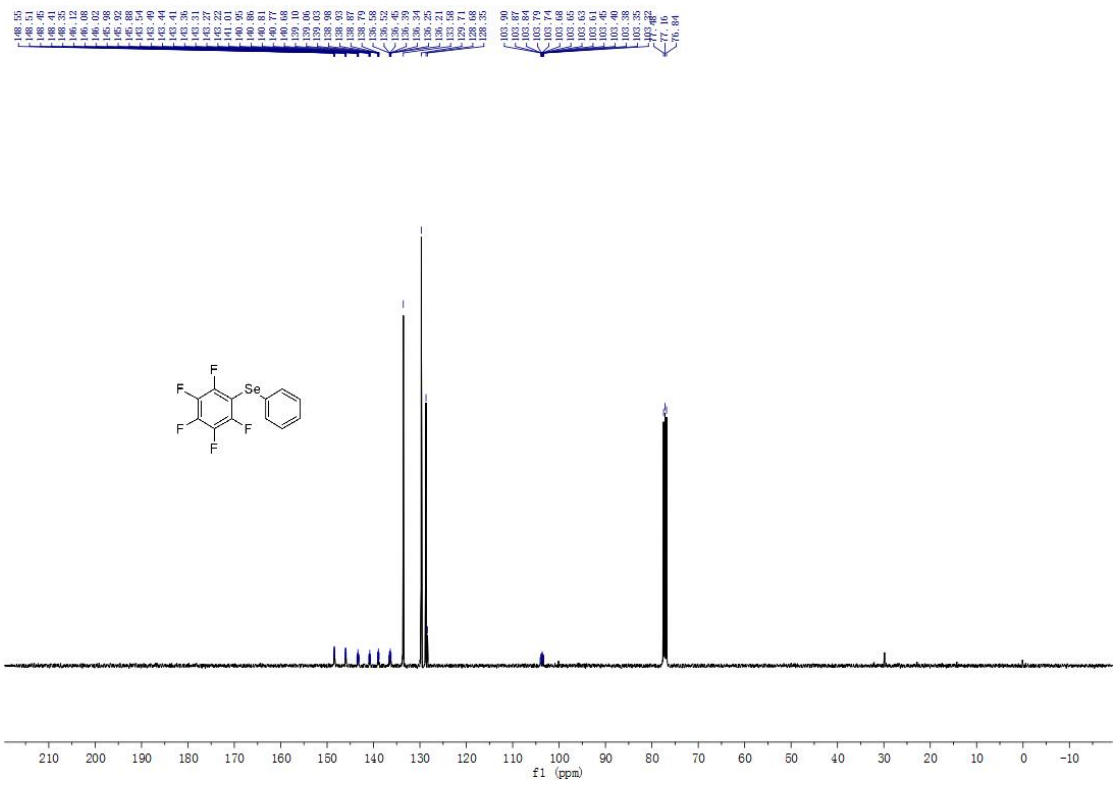
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6h**



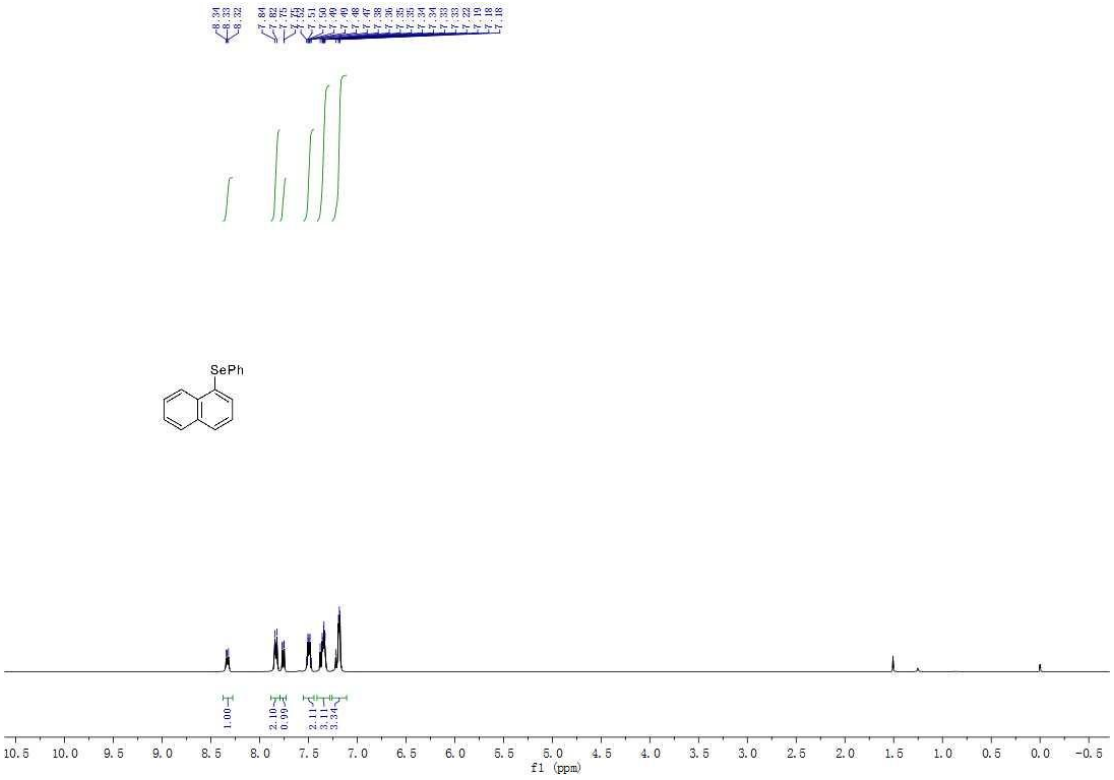
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6i**



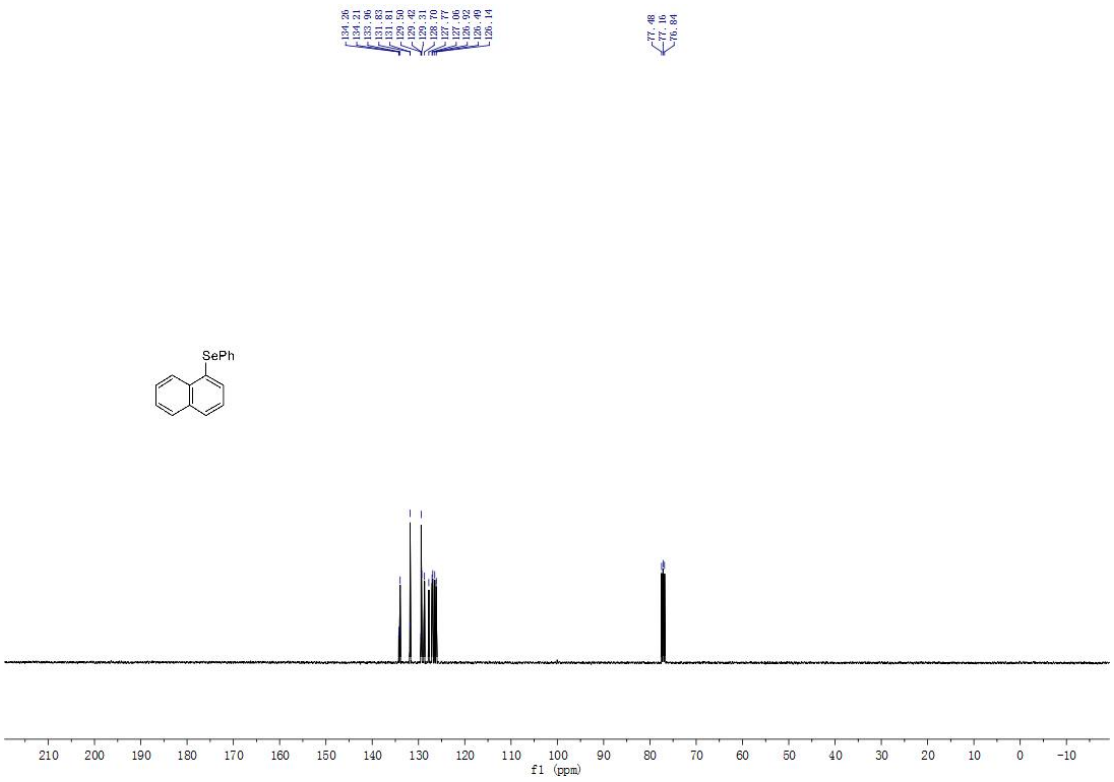
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6i**



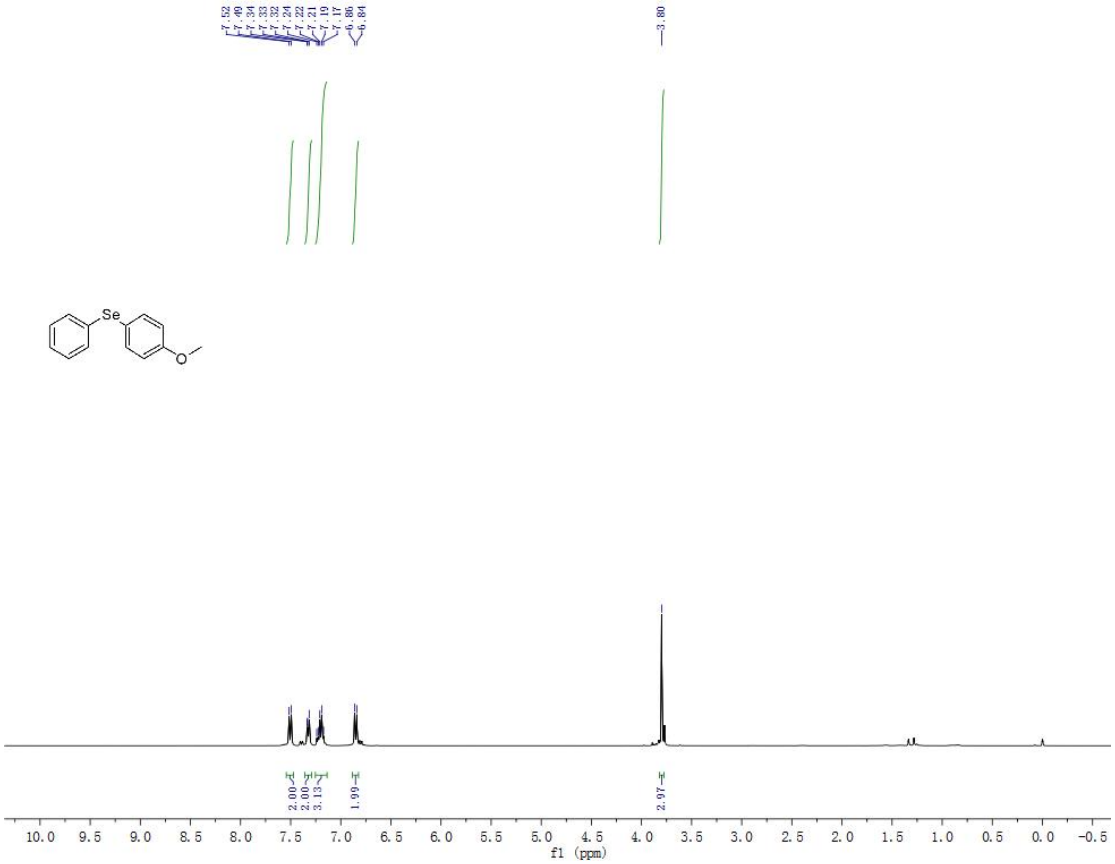
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6j**



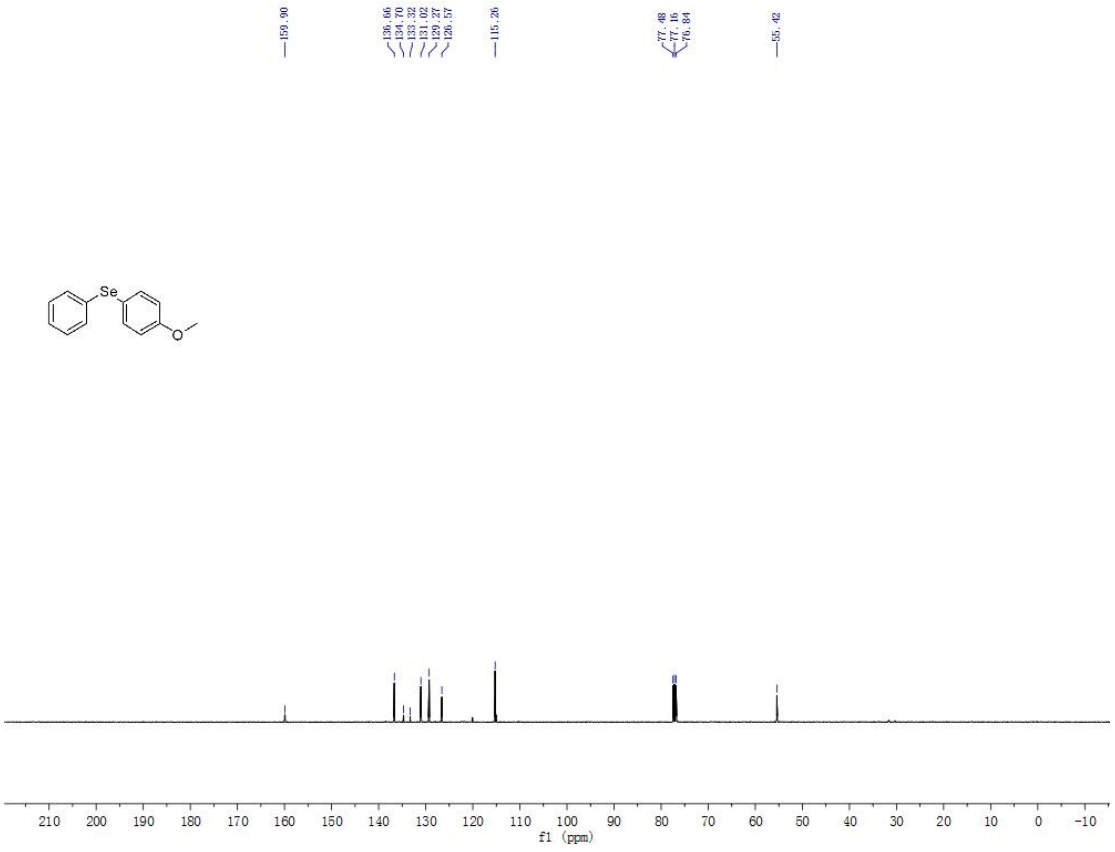
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6j**



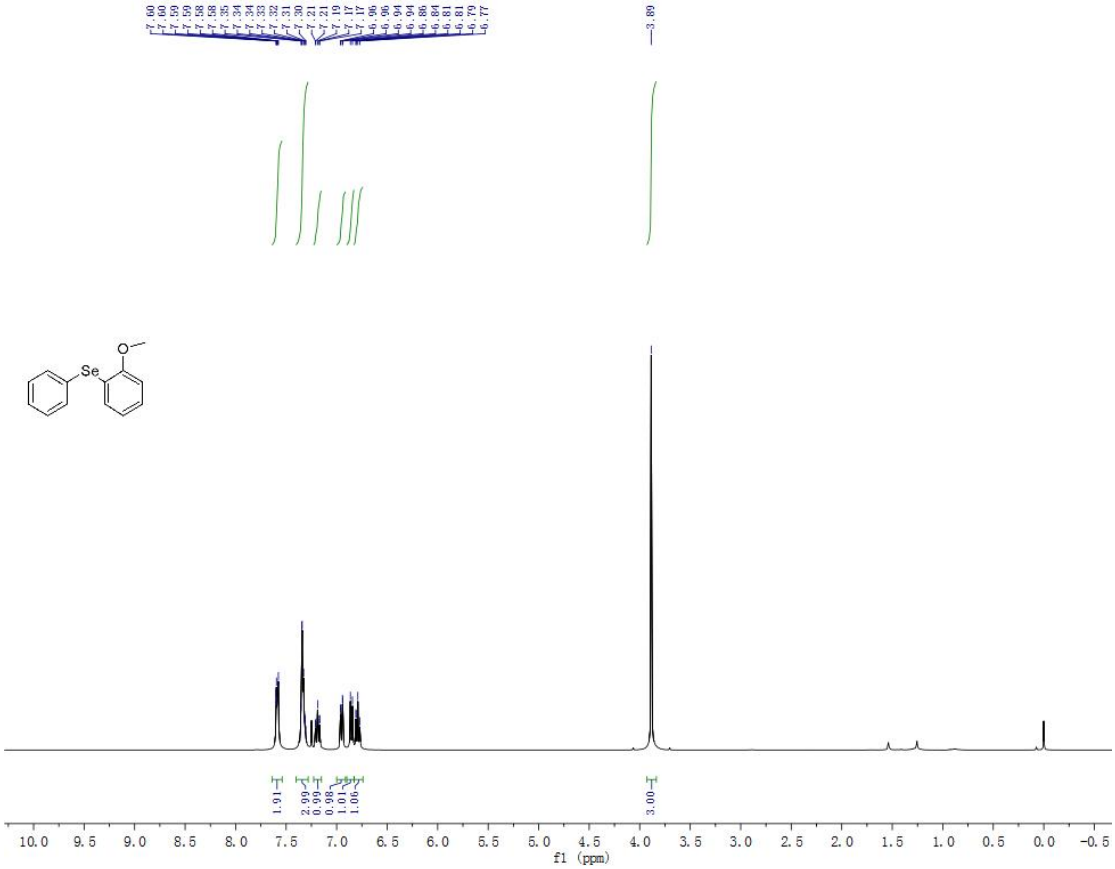
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6g**



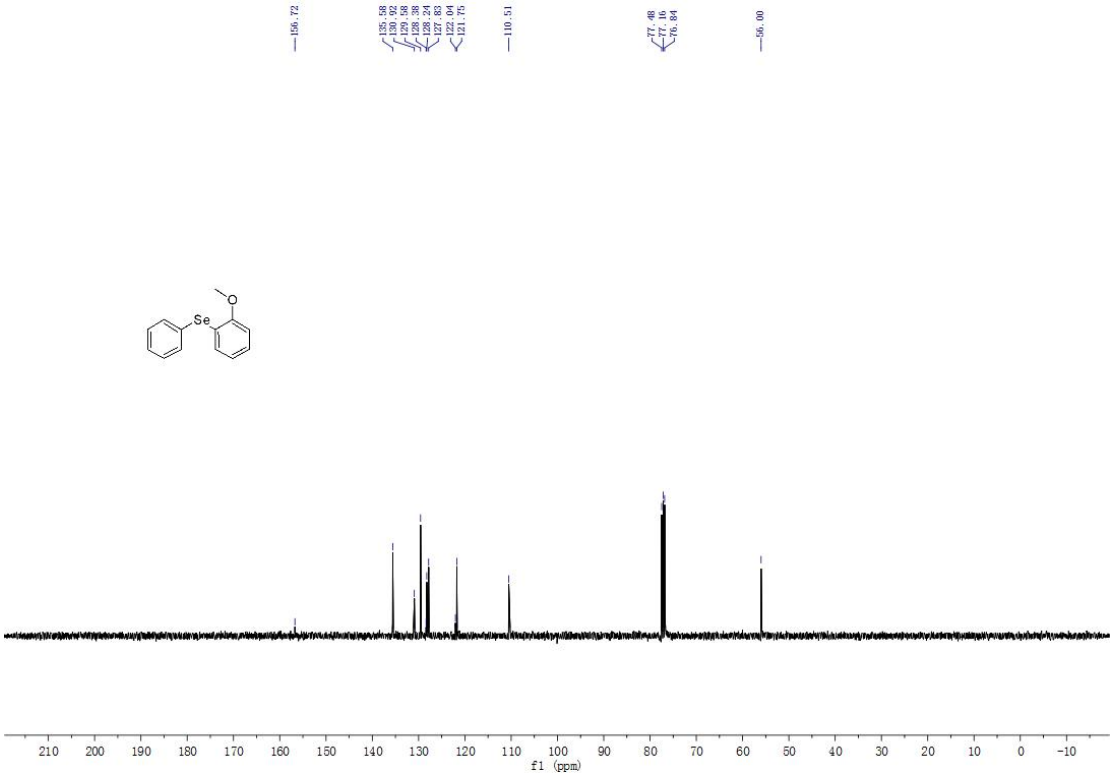
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6g**



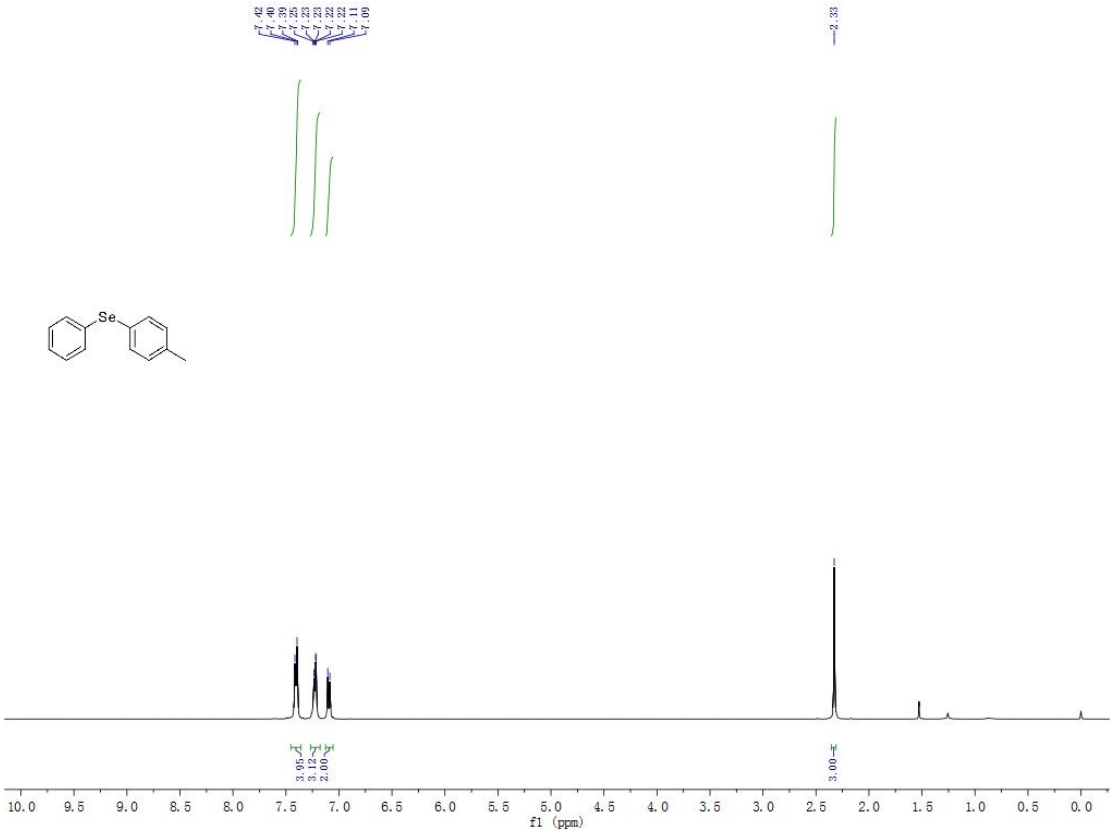
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6k**



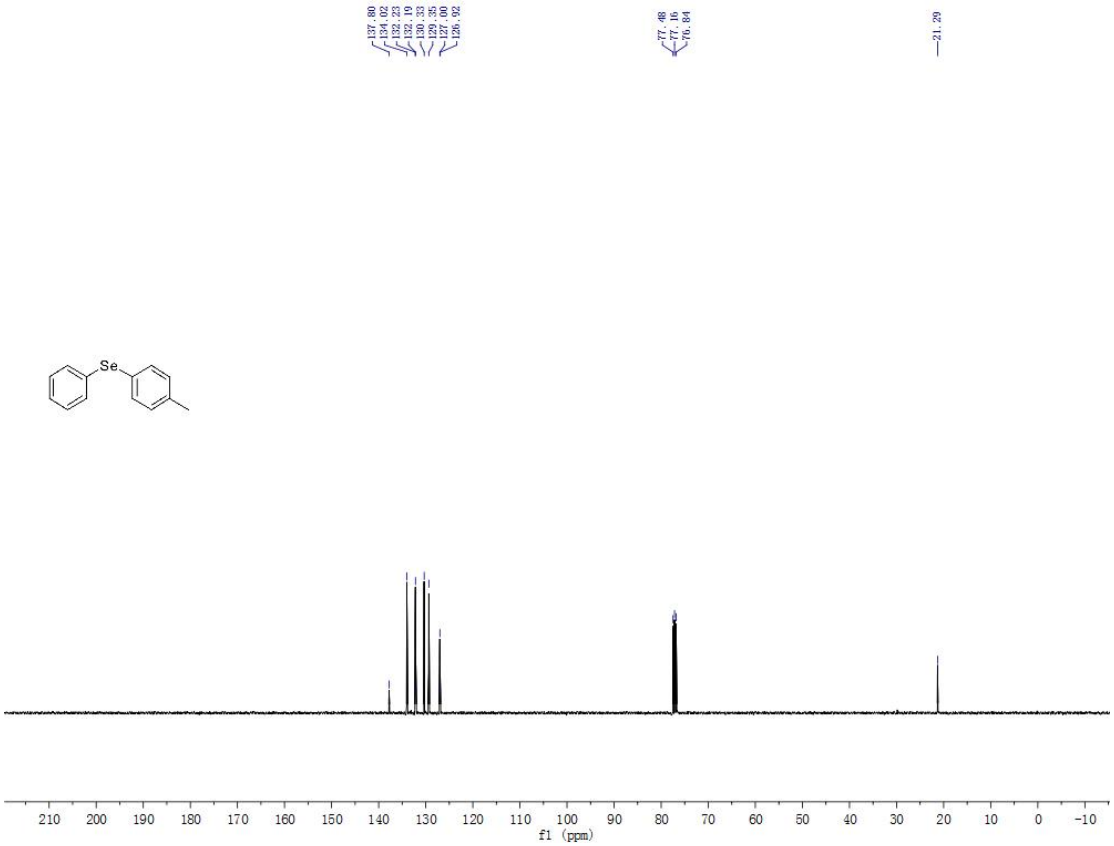
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6k**



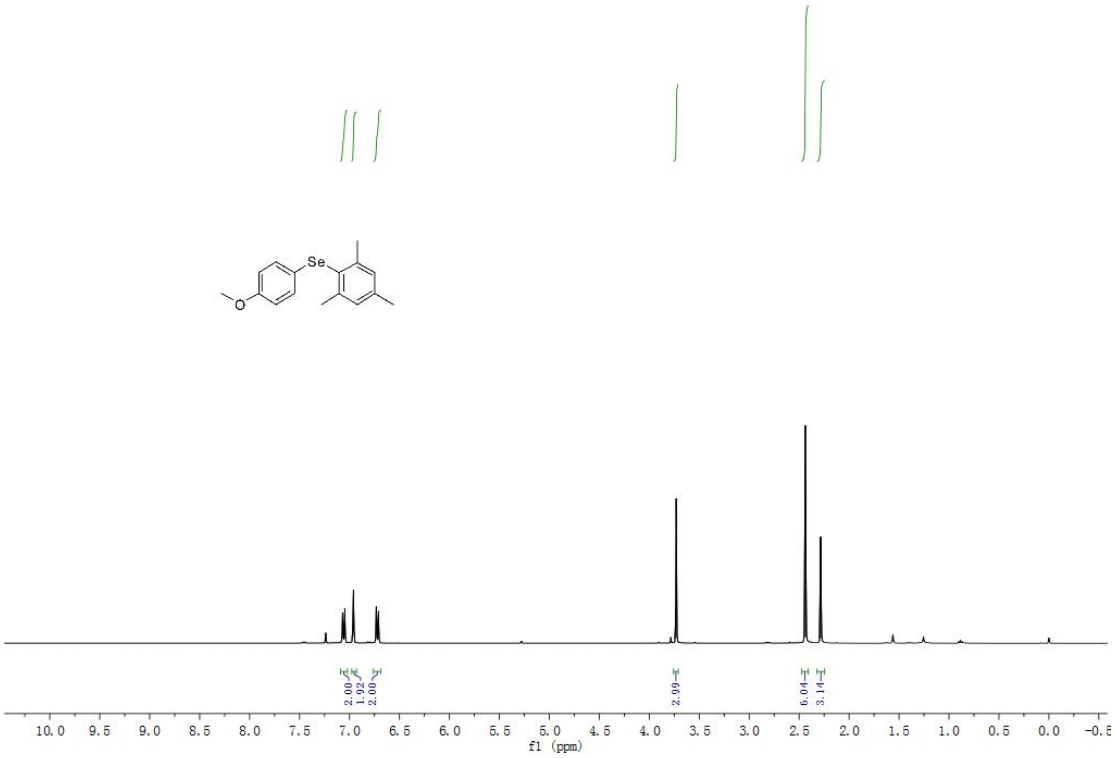
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6l**



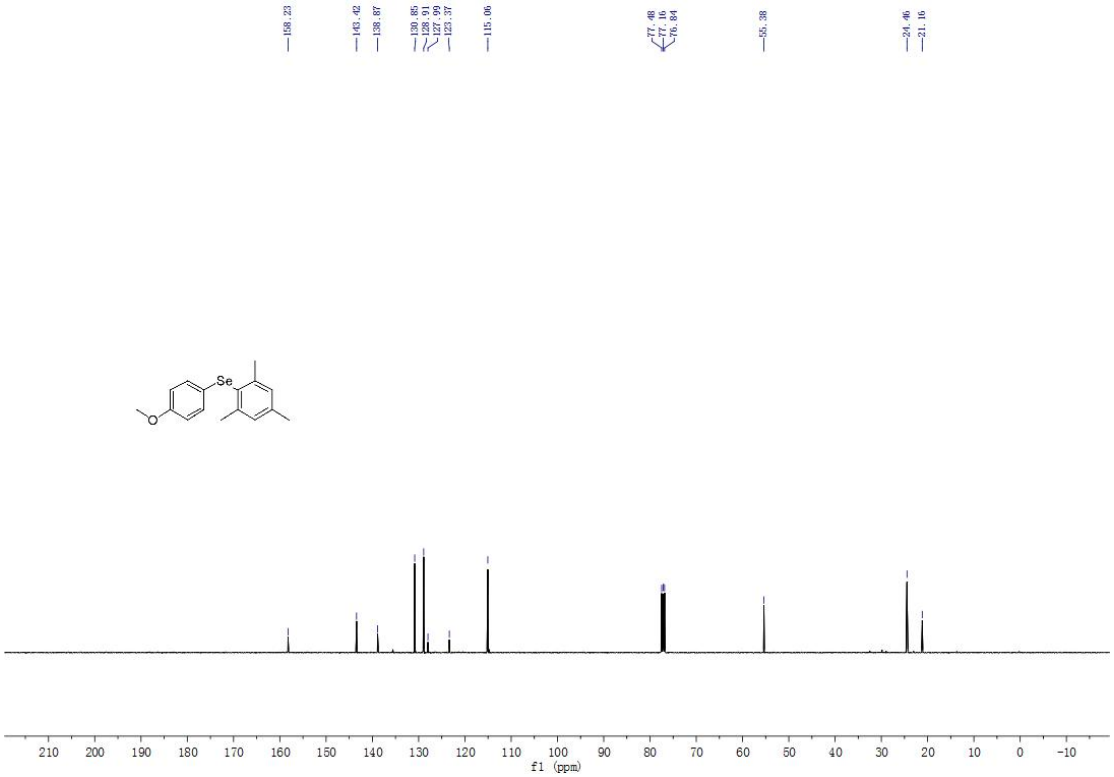
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6l**



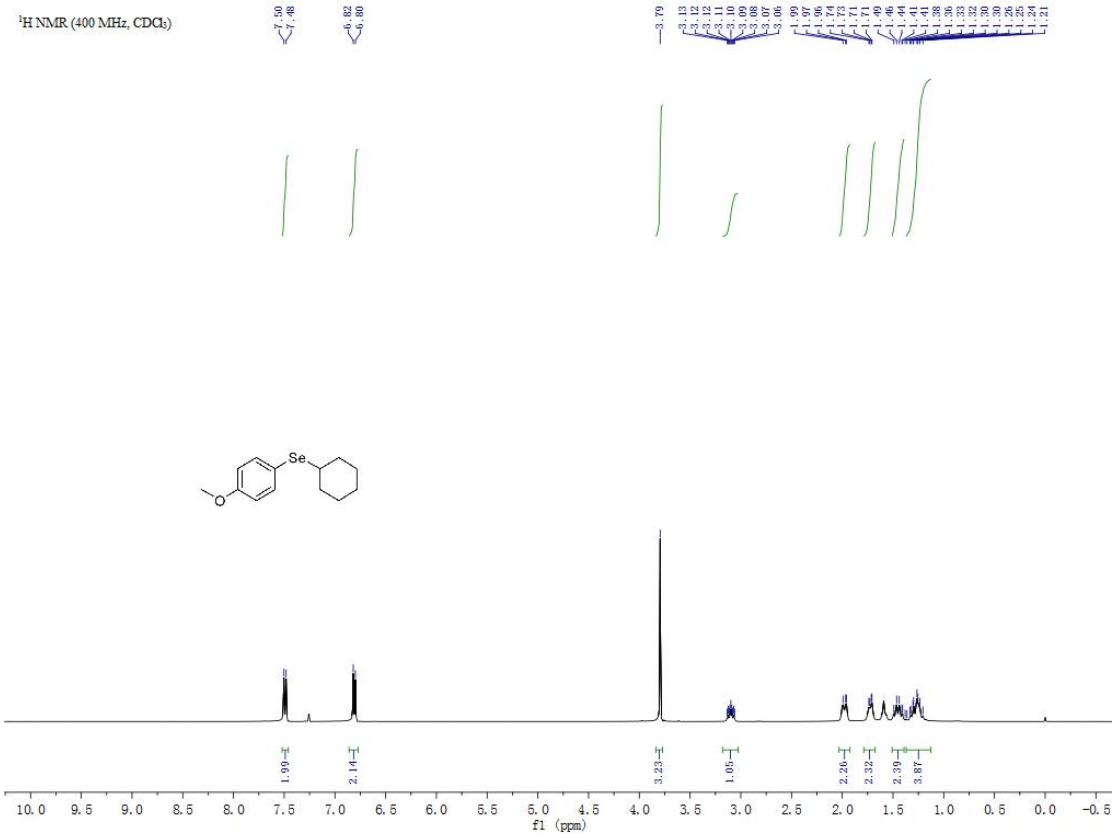
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6m**



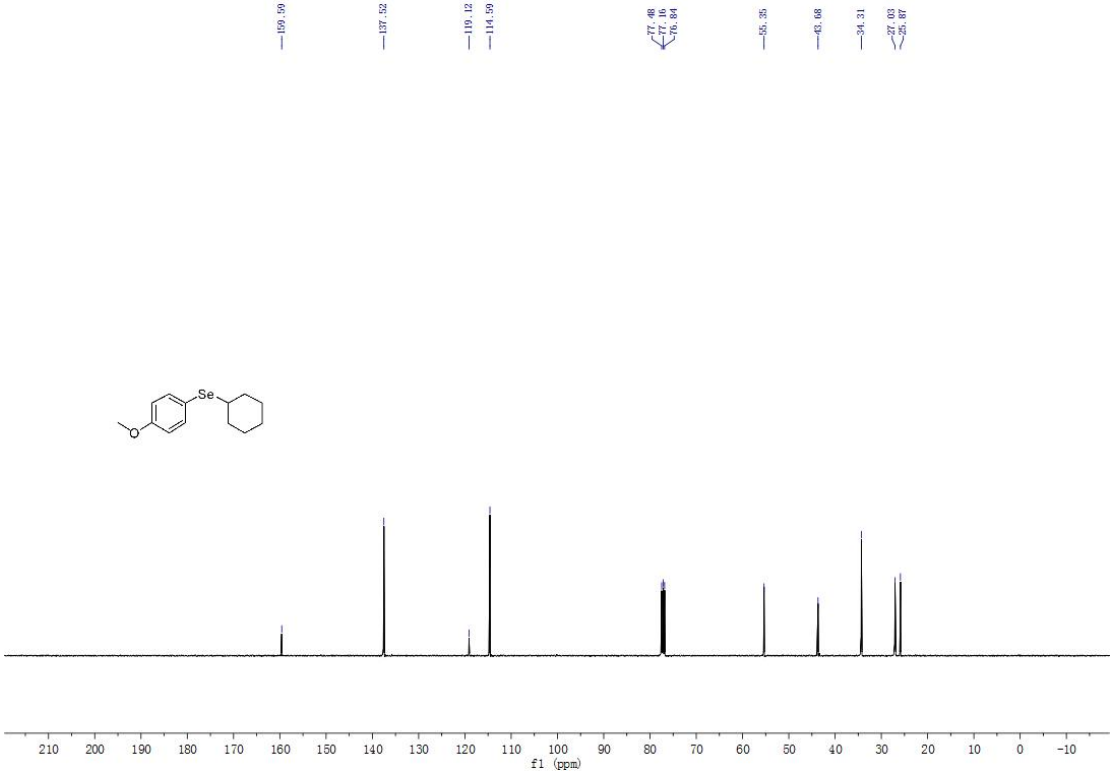
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6m**



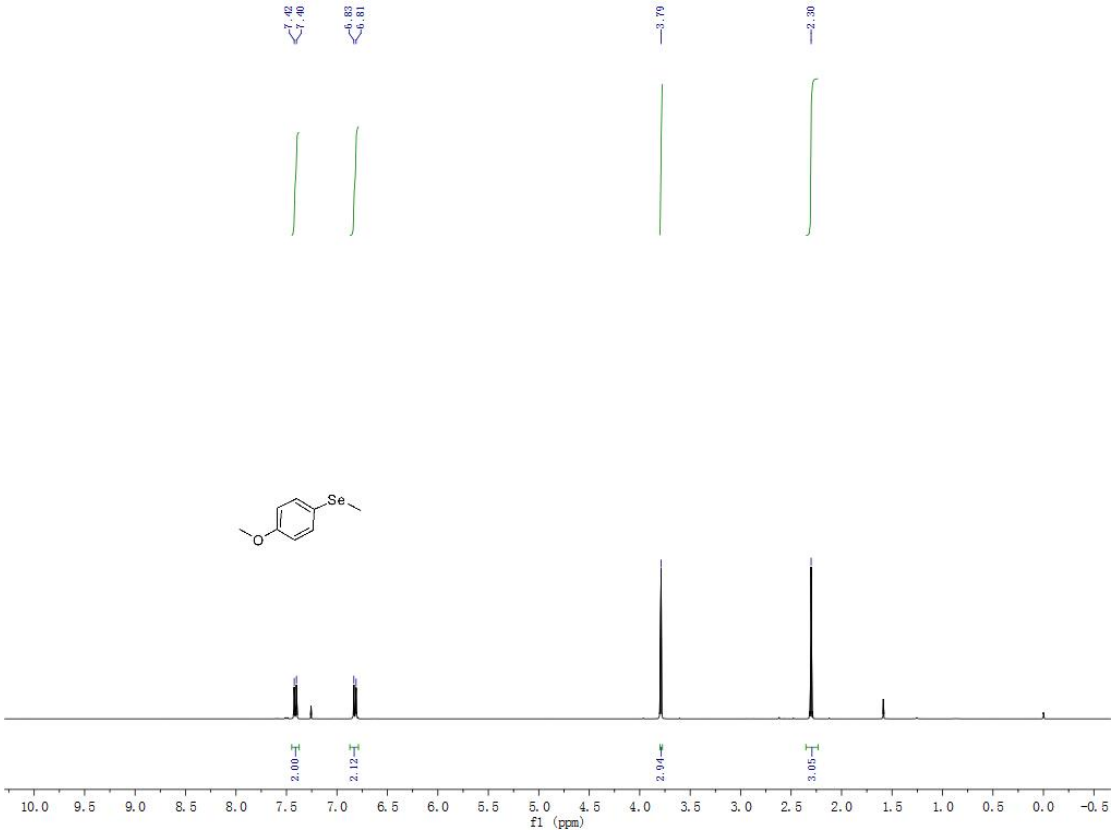
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6n**



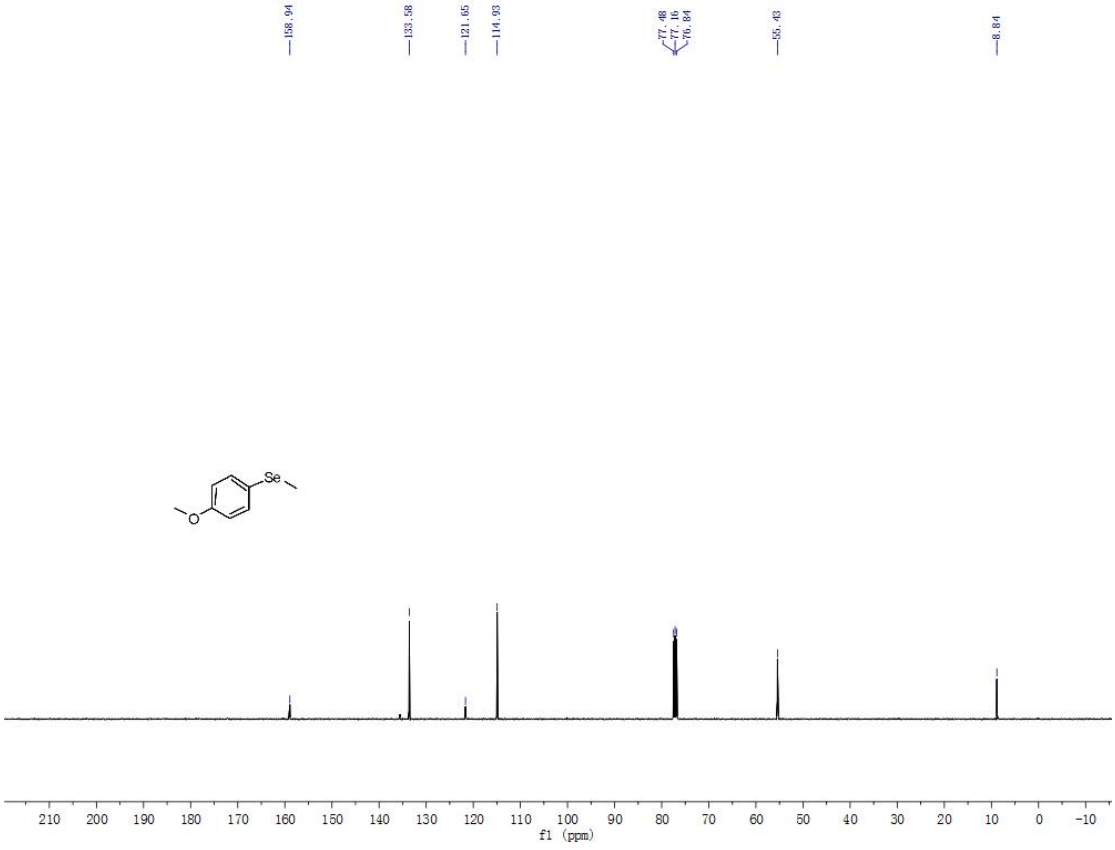
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6n**



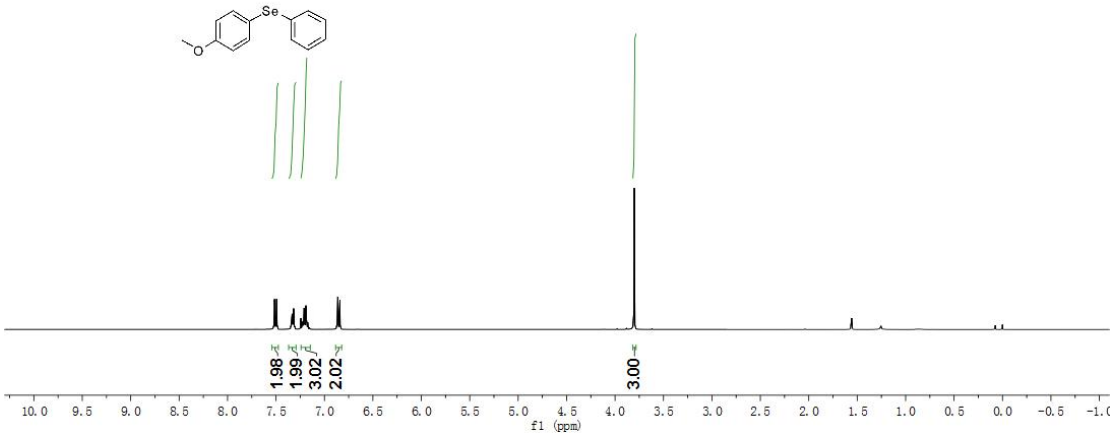
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6o**



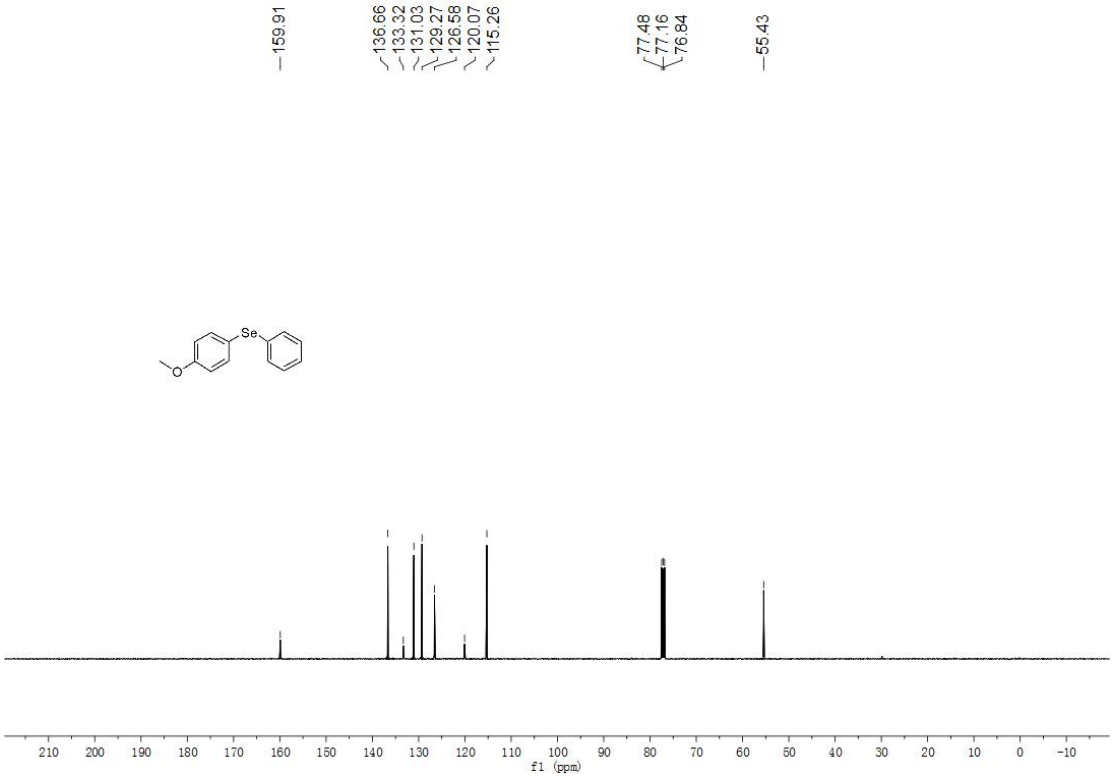
^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6o**



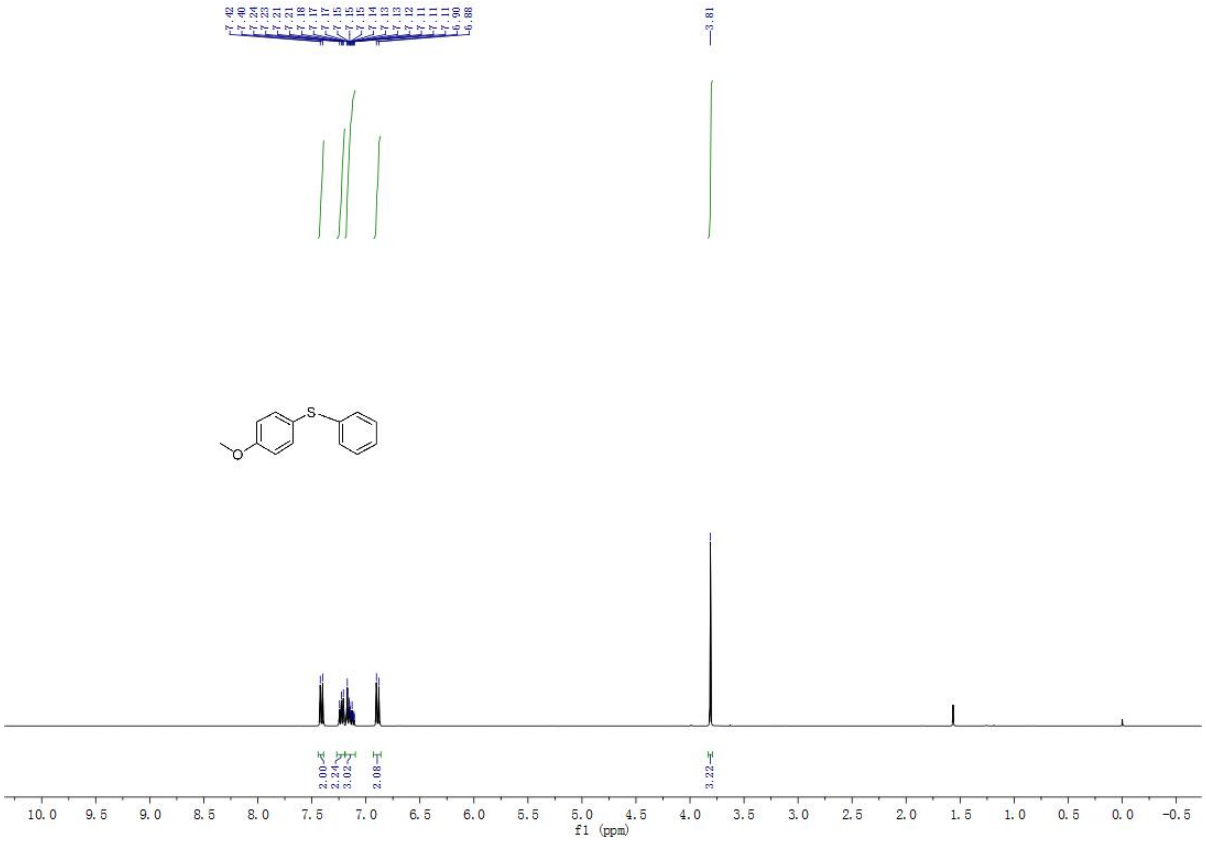
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6g**



^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6g**



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound **6p**



^{13}C NMR (101 MHz, $CDCl_3$) spectrum of compound **6p**