Electronic Supplementary Material (ESI) for ChemComm.

SUPPORTING INFORMATION

Transition Metal-Free Coupling Reaction of Benzylic Trimethylammonium Salts with Di(hetero)aryl Disulfides and Diselenides

Fuhai Li,[a] Dan Wang,[a] Hongyi Chen,[a] Ze He,[a] Lihong Zhou,[b] and Qingle Zeng*[a]

[a] *State Key Laboratory of Geohazard Prevention and Geoenvironment Protection, College of Materials, Chemistry & Chemical Engineering, Chengdu University of Technology, Chengdu 610059, China.*

[b] *College of Environment and Ecology, Chengdu University of Technology, Chengdu 610059, China.*

E-mail: qinglezeng@hotmail.com; qlzeng@cdut.edu.cn

Contents

General Information

All coupling reactions were carried out under Argon atmosphere in dried test tube sealed with rubber sleeve stopper. The test tube used was dried in an electric oven at 110 °C. Chemicals were purchased from Aladdin, Adamas, Aldrich, Alfa Aesar, and Kelong Chemical Co. and used as received, unless other mentioned. Petroleum ether refers to the fraction boiling in the 60-90 °C range. Unless otherwise stated, there is no further purification from the commercial supplier's products. ^{1}H NMR spectra were determined on a Bruker Avance III 400 MHz instrument. ^{1}H NMR data are reported in δ units (ppm), and were measured relative to the signals for residual chloroform (7.26 ppm), EtOAc (4.12 ppm, 2.04 ppm, 1.25 ppm) or acetone (2.05 ppm) in the deuterated solvent, unless otherwise stated. ^{13}C NMR spectra are reported in (ppm) relative to deuterated chloroform (77.2 ppm) unless otherwise stated, and all were obtained with ^{1}H decoupling. Mass spectral data of the products were collected by GC–MS analysis with a QP-2010 SE. All chiral HPLC analyses were performed on a LC3000 I type high performance liquid chromatograph (Beijing Gangchen Technology Co., Ltd.) with Daicel Chiralcel OD-H, Chiralcel OJ-H, Chiralpak AD-H and Chiralpak AS-H chiral columns (4.6 mm × 250 mm × 5 μm) with n-hexane/isopropanol as mobile phase, and the UV detection was monitored at 254 nm. Optical rotations were measured on a Autopol IV polarimeter with a sodium lamp at λ = 589 nm and reported as $[\alpha]^{T}_{D}$ (*c* =g/100 mL, solvent).

General Prcedure for the Synthesis of Quaternary Ammonium Triflates

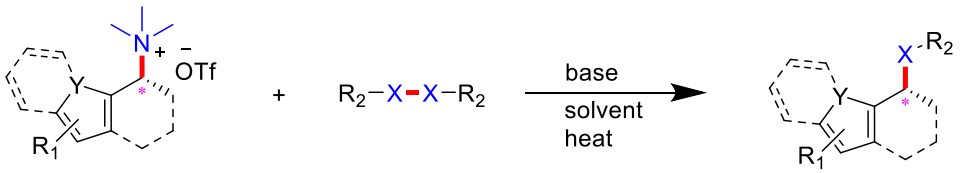
In a dry 50 mL round bottom flask was added 5.0 mmol tertiary amine and 30 mL pre-frozen ethyl ether. Then, under vigorous stirring, 5.5 mmol methyl triflate was added dropwise to the flask. The resulting white solid was filtered and washed twice with ether (2 × 20 mL). After vacuum drying, the corresponding quaternary ammonium salt was obtained.

General Prcedure for the Synthesis of Diselenides

Diselenides were prepared according to literature methods.[1] To a stirred solution of selenium powder (10.0 mmol) and aryl iodine (4.0 mmol) in DMSO (10.0 mL) was added CuO nanoparticles (10.0 mol %) followed by KOH (2.5 equiv) under nitrogen atmosphere at 90 °C. The progress of the reaction was monitored by TLC. After the reaction was complete, it was quenched with saturated brine. The mixture was extracted with n-hexane for three times. The combined organic layer was washed with

water for three times, dried over anhydrous $MgSO_4$. The dried solution was condensed on a rotary evaporator to give pure diaryl diselenide.

General Procedure for Coupling of Trimethylammonium Triflates with Disulfides and Diselenides

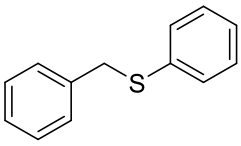


Y= S, CH=CH, CH=N
X= S, Se
R_2= Aryl, Heteroaryl

To an oven-dried 25 mL test tube with standard ground joint equipped with a stir bar were added benzylic Trimethylammonium triflate (1.0 mmol), disulfide or diselenide (1.2 mmol), Cs_2CO_3 (2.0 mmol), MeCN (5.0 mL). After the test tube was sealed with a rubber sleeve stopper, the air in the tube was pumped out with a vacuum pump and then injected with argon (repeat for three cycles). The mixture was stirred at 80 °C for 12 h. After cooled to room temperature, the reaction mixture was quenched by the addition of saturated NaCl solution (10 mL). The reaction mixture was extracted with ethyl acetate (15 mL × 3). The combined organic phase was dried over $MgSO_4$, filtered and concentrated in vacuum on a rotary evaporator. The resulting residue was purified by silica gel flash chromatography, eluting with petroleum ether/EtOAc to afford the corresponding products as a colorless/yellowish oil or white/yellowish solid.

Characterization Data and HPLC Traces of Products 3, 5, 6, 7.

Benzyl(phenyl)sulfane (3aa-S).[2]



3aa-S

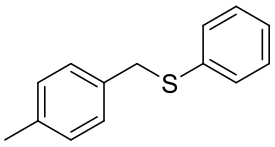
yellowish solid, 178.3 mg, 89% yield.

Melting point: 37 - 39 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.24 – 7.17 (m, 6H), 7.17 – 7.10 (m, 3H), 7.10 – 7.04 (m, 1H), 4.01 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 137.5, 136.4, 129.9, 128.9, 128.9, 128.5, 127.2, 126.4, 39.1.

(4-methylbenzyl)(phenyl)sulfane (3ba-S).[3]



3ba-S

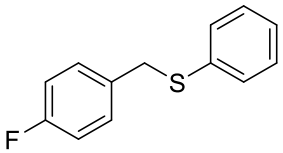
White solid, 192.9 mg, 90% yield.

Melting point: 64 - 66 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.28 – 7.20 (m, 2H), 7.20 – 7.14 (m, 2H), 7.14 – 7.06 (m, 3H), 7.01 (d, J = 7.9 Hz, 2H), 4.01 (s, 2H), 2.24 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 136.9, 136.7, 134.3, 129.6, 129.2, 128.8, 128.7, 126.2, 38.7, 21.1.

(4-fluorobenzyl)(phenyl)sulfane (3ca-S).[4]



3ca-S

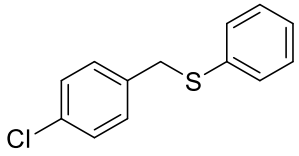
White solid, 192.1 mg, 88% yield.

Melting point: 58 - 60 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.30 – 7.05 (m, 7H), 6.96 – 6.75 (m, 2H), 3.99 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 162.0 (d, *J* = 245.5 Hz), 135.9, 133.3 (d, *J* = 3.3 Hz), 130.4 (d, *J* = 8.1 Hz), 130.2, 128.9, 126.6, 115.3 (d, *J* = 21.6 Hz), 38.5.

(4-chlorobenzyl)(phenyl)sulfane (3da-S).[5]



3da-S

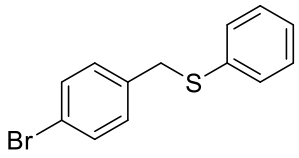
White solid, 208.9 mg, 89% yield.

Melting point: 67 - 69 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.27 – 7.03 (m, 9H), 3.97 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 136.2, 135.7, 133.0, 130.3, 130.1, 128.9, 128.6, 126.7, 38.6.

(4-bromobenzyl)(phenyl)sulfane (3ea-S).[4]



3ea-S

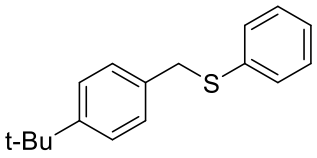
White solid, 245.7 mg, 88% yield.

Melting point: 72 - 74 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.35 – 7.26 (m, 2H), 7.24 – 7.08 (m, 5H), 7.08 – 7.00 (m, 2H), 3.95 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 136.7, 135.7, 131.6, 130.5, 130.3, 129.0, 126.7, 121.1, 38.6.

(4-(tert-butyl)benzyl)(phenyl)sulfane (3fa-S).[6]



3fa-S

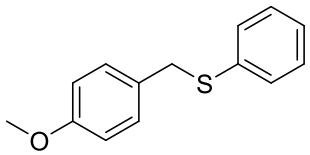
White solid, 235.9 mg, 92% yield.

Melting point: 44 - 46 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.28 – 7.21 (m, 4H), 7.17 (t, J = 7.6 Hz, 4H), 7.13 – 7.04 (m, 1H), 4.03 (s, 2H), 1.22 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-d) δ 150.2, 136.9, 134.3, 129.4, 128.9, 128.5, 126.1, 125.5, 38.5, 34.5, 31.4.

(4-methoxybenzyl)(phenyl)sulfane (3ga-S).[7]



3ga-S

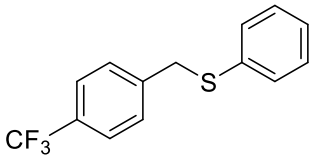
White solid, 216.5mg, 94% yield.

Melting point: 78 - 80 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.32 – 6.96 (m, 7H), 6.84 – 6.67 (m, 2H), 3.99 (s, 2H), 3.69 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 158.8, 136.6, 130.0, 129.8, 129.4, 128.8, 126.3, 113.9, 55.3, 38.5.

Phenyl(4-(trifluoromethyl)benzyl)sulfane (3ha-S).[6]



3ha-S

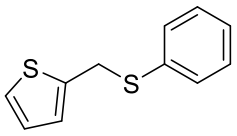
White solid, 228.1mg, 85% yield.

Melting point: 63 - 65 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.44 (d, J = 8.1 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 7.24 – 7.08 (m, 5H), 4.04 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 141.9, 135.4, 130.4, 129.4 (d, J = 32.6 Hz), 129.1, 129.0, 126.9, 125.4 (q, J = 3.8 Hz), 122.8, 38.8.

2-((phenylthio)methyl)thiophene (3ia-S).[8]



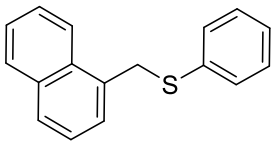
3ia-S

Yellowish oil, 187.8 mg, 91% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.30 – 7.22 (m, 2H), 7.22 – 7.04 (m, 4H), 6.79 (d, *J* = 4.3 Hz, 2H), 4.22 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 140.9, 135.7, 130.4, 128.9, 126.8, 126.7, 126.3, 125.0, 33.8.

(naphthalen-1-ylmethyl)(phenyl)sulfane (3ja-S).[6]



3ja-S

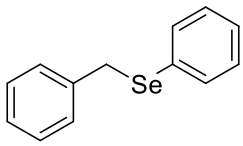
Yellowish solid, 225.3 mg, 90% yield.

Melting point: 69 - 71 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.04 (dq, *J* = 8.8, 1.0 Hz, 1H), 7.84 – 7.71 (m, 1H), 7.71 – 7.57 (m, 1H), 7.42 (dddd, *J* = 19.7, 8.1, 6.8, 1.4 Hz, 2H), 7.28 – 7.20 (m, 4H), 7.16 (ddt, *J* = 8.1, 6.5, 0.8 Hz, 2H), 7.12 – 7.07 (m, 1H), 4.45 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 136.7, 134.0, 132.8, 131.5, 130.2, 128.9, 128.9, 128.3, 127.4, 126.6, 126.3, 125.9, 125.3, 124.0, 37.2.

Benzyl(phenyl)selane (3aa-Se).[5]



3aa-Se

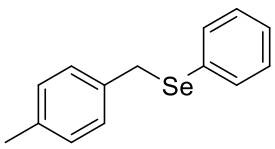
Colorless oil, 222.5 mg, 90% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.46 – 7.42 (m, 2H), 7.26 – 7.17 (m, 8H), 4.10 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 138.7, 133.6, 130.5, 129.0, 128.9, 128.5, 127.3, 126.9, 32.3.

GCMS (EI): *m*/*z* (%) = 248.00(10) [M]$^+$, 156.95(5), 91.05(100).

(4-methylbenzyl)(phenyl)selane (3ba-Se).[5]



3ba-Se

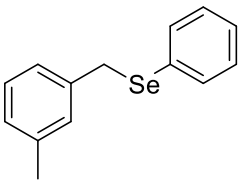
Colorless oil, 237.7 mg, 91% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.35 (dddd, J = 6.7, 4.5, 3.6, 1.4 Hz, 2H), 7.15 – 7.09 (m, 3H), 7.00 (d, J = 8.1 Hz, 2H), 6.94 (d, J = 7.9 Hz, 2H), 3.97 (s, 2H), 2.19 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 136.6, 135.6, 133.4, 130.9, 129.3, 129.1, 128.8, 127.3, 32.1, 21.3.

GCMS (EI): *m*/*z* (%) = 262.05(10) [M]$^+$, 156.95(5), 105.05(100), 91.05(5).

(3-methylbenzyl)(phenyl)selane (3ca-Se).[5]



3ca-Se

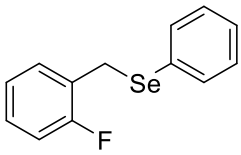
Yellowish oil, 235.1 mg, 90% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.59 – 7.49 (m, 2H), 7.37 – 7.28 (m, 3H), 7.25 – 7.15 (m, 1H), 7.09 (d, J = 7.4 Hz, 3H), 4.15 (s, 2H), 2.36 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 138.5, 138.1, 133.5, 130.7, 129.7, 129.0, 128.4, 127.7, 127.3, 126.0, 32.3, 21.4.

GCMS (EI): *m*/*z* (%) = 262.05 (10) [M]$^+$, 156.95(5), 105.05 (100), 91.05 (5).

(2-fluorobenzyl)(phenyl)selane (3da-Se).



3da-Se

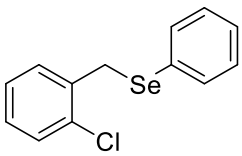
Colorless oil, 230.7 mg, 87% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.39 – 7.34 (m, 2H), 7.17 – 7.10 (m, 3H), 7.09 – 7.02 (m, 1H), 6.96 (td, J = 7.6, 1.8 Hz, 1H), 6.92 – 6.84 (m, 2H), 3.99 (d, J = 1.2 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 160.7 (d, J = 247.2 Hz), 134.2, 130.9 (d, J = 3.8 Hz), 129.8, 129.0, 128.7 (d, J = 8.1 Hz), 127.7, 126.2 (d, J = 14.7 Hz), 124.0 (d, J = 3.7 Hz), 115.5 (d, J = 21.6 Hz), 24.8 (d, J = 3.0 Hz).

GCMS (EI): *m*/*z* (%) = 265.95 (10) [M]$^+$, 156.95 (5), 109.05 (100), 83.00 (10).

(2-chlorobenzyl)(phenyl)selane (3ea-Se).[9]



3ea-Se

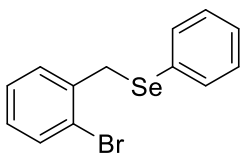
Colorless oil, 247.8 mg, 88% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.47 (ddd, J = 8.1, 4.1, 1.8 Hz, 2H), 7.34 (dq, J = 7.8, 1.4 Hz, 1H), 7.29 – 7.20 (m, 3H), 7.16 – 6.99 (m, 3H), 4.17 (d, J = 2.8 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 136.7, 134.5, 133.9, 130.7, 129.8, 129.7, 129.0, 128.3, 127.7, 126.7, 30.1.

GCMS (EI): *m*/*z* (%) = 281.95 (10) [M]$^+$, 125.05 (100), 99.05 (5), 89.05 (15).

(2-bromobenzyl)(phenyl)selane (3fa-Se).[8]



3fa-Se

Colorless oil, 280.4 mg, 86% yield.

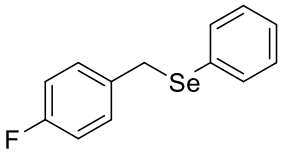
^{1}H NMR (400 MHz, Chloroform-d) δ 7.61 – 7.42 (m, 3H), 7.26 (dq, J = 11.7, 7.5, 5.6 Hz, 3H), 7.17 – 6.96 (m, 3H), 4.22 – 4.16 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 138.3, 134.5, 133.1, 130.7, 129.8,

129.0, 128.5, 127.7, 127.3, 124.4, 33.0.

GCMS (EI): *m*/*z* (%) = 327.95 (20), 325.95 (25) $[M]^+$, 168.95 (100), 90.05 (50).

(4-fluorobenzyl)(phenyl)selane (3ga-Se).[8]



3ga-Se

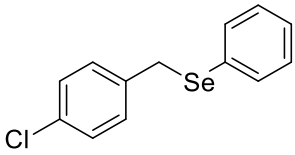
Colorless oil, 233.376 mg, 88% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.43 – 7.39 (m, 2H), 7.25 – 7.20 (m, 3H), 7.14 – 7.08 (m, 2H), 6.93 – 6.86 (m, 2H), 4.04 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 161.8(d, J = 245.4 Hz), 134.5 (d, J = 3.3 Hz), 133.9, 130.4 (d, J = 8.1 Hz), 130.0, 129.1, 127.6, 115.3 (d, J = 21.4 Hz), 31.4.

GCMS (EI): *m*/*z* (%) = 266.00 (5) $[M]^+$, 156.95 (5), 109.05 (100), 83.05 (10).

(4-chlorobenzyl)(phenyl)selane (3ha-Se).[5]



3ha-Se

White solid, 250.7 mg, 89% yield.

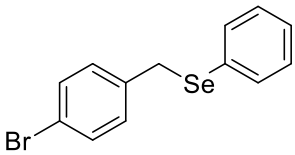
Melting point : 63 - 65 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.44 – 7.39 (m, 2H), 7.28 – 7.21 (m, 3H), 7.20 – 7.15 (m, 2H), 7.10 – 7.04 (m, 2H), 4.02 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 137.4, 133.9, 132.6, 130.2, 129.9, 129.1, 128.6, 127.6, 31.5.

GCMS (EI): *m*/*z* (%) = 291.95 (10) $[M]^+$, 207.00 (10), 157.00 (5), 125.05 (100), 99.00 (5), 89.05 (15).

(4-bromobenzyl)(phenyl)selane (3ia-Se).[8]



3ia-Se

White solid, 280.5 mg, 86% yield.

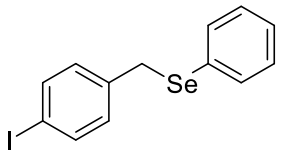
Melting point : 60 - 62 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.43 – 7.39 (m, 2H), 7.35 – 7.31 (m, 2H), 7.27 – 7.21 (m, 3H), 7.03 – 7.00 (m, 2H), 4.00 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 137.9, 133.9, 131.5, 130.5, 129.8, 129.1, 127.6, 120.7, 31.5.

GCMS (EI): *m*/*z* (%) = 325.90 (20) $[M]^+$, 207.00 (40), 170.90 (100), 109.00 (5), 90.05 (40).

(4-iodobenzyl)(phenyl)selane (3ja-Se).



3ja-Se

Yellowish solid, 268.63 mg, 72% yield.

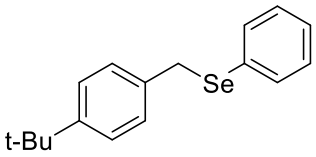
Melting point : 50 - 52 °C;

^{1}H NMR (400 MHz, Chloroform-d) δ 7.51 – 7.45 (m, 2H), 7.38 – 7.31 (m, 2H), 7.21 – 7.16 (m, 3H), 6.86 – 6.81 (m, 2H), 3.93 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 138.6, 137.5, 133.9, 130.8, 129.8, 129.1, 127.6, 92.1, 31.6.

GCMS (EI): *m*/*z* (%) = 373.90 (10) [M]$^+$, 216.95 (100), 156.95 (5), 90.05 (40).

(4-(tert-butyl)benzyl)(phenyl)selane (3ka-Se).



3ka-Se

Yellowish solid, 273.0 mg, 90% yield.

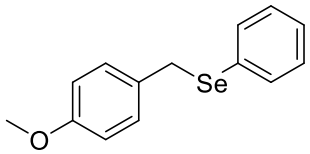
Melting point: 38 - 40 °C

^{1}H NMR (400 MHz, Chloroform-d) δ 7.36 (ddt, J = 3.9, 3.0, 1.6 Hz, 2H), 7.19 – 7.15 (m, 2H), 7.12 (tt, J = 3.5, 2.1 Hz, 3H), 7.09 – 7.04 (m, 2H), 4.00 (s, 2H), 1.19 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-d) δ 149.9, 135.5, 133.3, 131.0, 129.1, 128.6, 127.2, 125.5, 34.6, 31.9, 31.5.

GCMS (EI): *m*/*z* (%) = 304.05 (5) [M]$^+$, 156.95 (5), 147.10 (100), 132.10 (15), 117.05 (20), 91.05 (10).

(4-methoxybenzyl)(phenyl)selane (3la-Se).[7]



3la-Se

White solid, 255.0 mg, 92% yield.

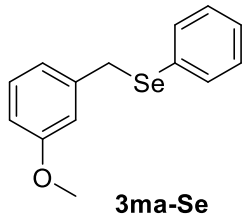
Melting point: 67 - 69 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.43 – 7.29 (m, 2H), 7.23 – 7.11 (m, 3H), 7.11 – 6.99 (m, 2H), 6.75 – 6.65 (m, 2H), 4.01 (s, 2H), 3.70 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 158.5, 133.5, 130.6, 130.6, 130.0, 129.0, 127.2, 113.9, 55.3, 31.8.

GCMS (EI): *m*/*z* (%) = 278.05 (20) [M]$^+$, 207.00 (30), 157.00 (25), 121.05 (100), 91.05 (50).

(3-methoxybenzyl)(phenyl)selane (3ma-Se).[10]



3ma-Se

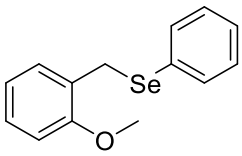
Yellowish oil, 246.7 mg, 89% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.56 – 7.49 (m, 2H), 7.31 (dd, J = 5.0, 1.9 Hz, 3H), 7.22 (t, J = 7.8 Hz, 1H), 6.87 (dt, J = 7.6, 1.3 Hz, 1H), 6.83 – 6.78 (m, 2H), 4.14 (s, 2H), 3.78 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 159.6, 140.2, 133.7, 130.5, 129.5, 129.1, 127.4, 121.3, 114.2, 112.8, 55.2, 32.3.

GCMS (EI): *m*/*z* (%) = 278.00 (20) $[M]^+$, 156.95 (5), 121.05 (100), 91.05 (30).

(2-methoxybenzyl)(phenyl)selane (3na-Se).[10]



3na-Se

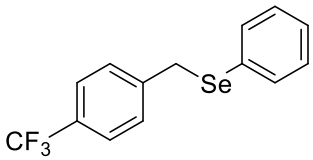
Colorless oil, 249.5 mg, 90% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.42 – 7.31 (m, 2H), 7.16 – 7.00 (m, 4H), 6.92 (dd, J = 7.7, 1.7 Hz, 1H), 6.76 – 6.62 (m, 2H), 4.02 (s, 2H), 3.65 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 157.2, 133.8, 131.1, 130.2, 128.9, 128.4, 127.4, 127.2, 120.4, 110.6, 55.5, 27.0.

GCMS (EI): *m*/*z* (%) = 277.95 (10) $[M]^+$, 207.00 (5), 156.90 (5), 121.05 (100), 91.05 (60).

Phenyl(4-(trifluoromethyl)benzyl)selane (3oa-Se).



3oa-Se

White solid, 264.8 mg, 84% yield.

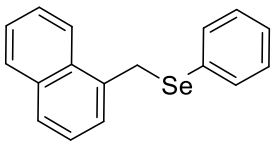
Melting point: 52 – 54 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.44 – 7.28 (m, 4H), 7.22 – 7.06 (m, 5H), 3.99 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 143.1, 134.1, 129.5, 129.2, 129.1, 127.8, 125.6, 125.4 (q, J = 3.8 Hz), 122.8, 31.6.

GCMS (EI): *m*/*z* (%) = 316.00 (20) $[M]^+$, 159.05 (100), 109.05 (20), 89.05 (5).

(naphthalen-1-ylmethyl)(phenyl)selane (3pa-Se).[7]



3pa-Se

White solid, 261.6 mg, 88% yield.

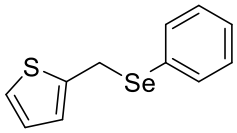
Melting point: 33 – 35 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.98 (dd, J = 8.4, 1.4 Hz, 1H), 7.71 (dd, J = 8.0, 1.5 Hz, 1H), 7.59 (dt, J = 8.2, 1.1 Hz, 1H), 7.45 – 7.27 (m, 4H), 7.23 – 6.98 (m, 5H), 4.41 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 134.2, 134.1, 133.9, 131.3, 130.9, 129.1, 128.9, 128.0, 127.5, 127.2, 126.1, 125.9, 125.3, 124.1, 30.2.

GCMS (EI): *m*/*z* (%) = 298.00 (10) [M]$^+$, 156.95 (5), 141.15 (100), 115.05 (50), 89.05 (5).

2-((phenylselanyl)methyl)thiophene (3qa-Se).[8]



3qa-Se

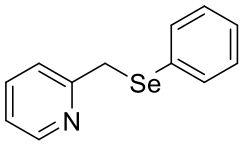
Colorless oil, 227.9 mg, 90% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.61 – 7.47 (m, 2H), 7.35 – 7.28 (m, 3H), 7.21 (dd, J = 5.1, 1.3 Hz, 1H), 6.91 (dd, J = 5.1, 3.5 Hz, 1H), 6.87 – 6.82 (m, 1H), 4.37 (d, J = 0.8 Hz, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 141.9, 133.8, 130.3, 129.1, 127.6, 126.8, 126.3, 124.8, 25.8.

GCMS (EI): *m*/*z* (%) = 253.95 (10) [M]$^+$, 156.95 (10), 97.05 (100).

2-((phenylselanyl)methyl)pyridine (3ra-Se).[11]



3ra-Se

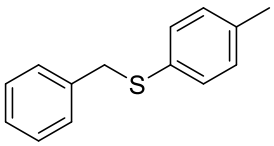
Brown oil, 181.9 mg, 73% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.46 – 8.32 (m, 1H), 7.48 – 7.34 (m, 3H), 7.17 – 7.06 (m, 3H), 7.06 – 6.87 (m, 2H), 4.13 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 158.7, 149.4, 136.4, 133.6, 129.9, 129.0, 127.4, 123.0, 121.7, 33.8.

GCMS (EI): *m*/*z* (%) = 248.95 (25) [M]$^+$, 233.95 (5), 168.05 (100), 92.05 (50).

Benzyl(p-tolyl)sulfane (3ab-S).[2]



3ab-S

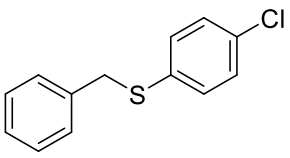
White solid, 180.3 mg, 84% yield.

Melting point: 39 – 40 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.23 – 7.08 (m, 7H), 7.02 – 6.91 (m, 2H), 3.96 (s, 2H), 2.20 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 137.9, 136.6, 132.6, 130.8, 129.7, 128.9, 128.5, 127.1, 39.8, 21.1.

Benzyl(4-chlorophenyl)sulfane (3ac-S).[2]



3ac-S

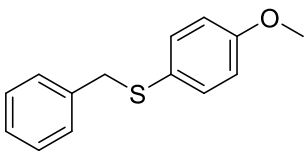
White solid, 211.2 mg, 90% yield.

Melting point: 42 – 43 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.31 (qd, *J* = 6.7, 1.2 Hz, 5H), 7.25 (s, 4H), 4.12 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 137.2, 134.7, 132.5, 131.4, 129.0, 128.8, 128.6, 127.4, 39.3.

Benzyl(4-methoxyphenyl)sulfane (3ad-S).[2]



3ad-S

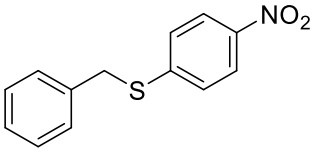
White solid, 195.5 mg, 85% yield.

Melting point: 38 – 40 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.28 – 7.03 (m, 7H), 6.75 – 6.62 (m, 2H), 3.91 (s, 2H), 3.69 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.2, 138.2, 134.1, 128.9, 128.4, 127.0, 126.1, 114.4, 55.3, 41.2.

Benzyl(4-nitrophenyl)sulfane (3ae-S).[2]



3ae-S

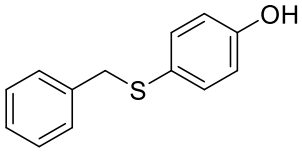
Yellowish solid, 200.9 mg, 82% yield.

Melting point: 111 – 113 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.13 – 7.88 (m, 2H), 7.37 – 7.10 (m, 7H), 4.16 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 147.3, 145.3, 135.5, 128.9, 128.7, 127.8, 126.7, 123.9, 37.0.

4-(benzylthio)phenol (3af-S).[12]



3af-S

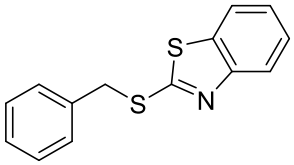
Yellowish solid, 185.7 mg, 86% yield.

Melting point: 111 – 113 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.24 – 7.05 (m, 7H), 6.67 – 6.52 (m, 2H), 3.90 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 155.3, 138.1, 134.4, 128.9, 128.4, 127.0, 126.1, 116.0, 41.3.

2-(benzylthio)benzo[d]thiazole (3ag-S).[13]



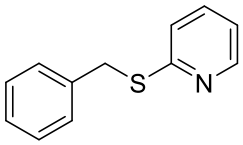
3ag-S

Colorless oil, 215.8mg, 84% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.82 (dt, *J* = 8.1, 0.9 Hz, 1H), 7.69 – 7.63 (m, 1H), 7.41 – 7.30 (m, 3H), 7.27 – 7.13 (m, 4H), 4.52 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 166.5, 153.2, 136.2, 135.4, 129.2, 128.7, 127.8, 126.1, 124.3, 121.6, 121.0, 37.8.

2-(benzylthio)pyridine (3ah-S).[14]



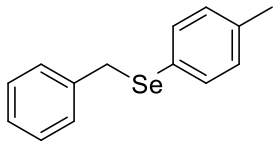
3ah-S

Yellowish oil, 180.9 mg, 90% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.55 – 8.36 (m, 1H), 7.51 – 7.43 (m, 3H), 7.39 – 7.31 (m, 2H), 7.31 – 7.24 (m, 1H), 7.19 (dt, *J* = 8.0, 1.0 Hz, 1H), 7.01 (ddd, *J* = 7.5, 5.0, 1.1 Hz, 1H), 4.50 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 158.9, 149.5, 138.1, 136.0, 129.0, 128.6, 127.2, 122.1, 119.6, 34.5.

Benzyl(p-tolyl)selane (3ab-Se).[15]



3ab-Se

Yellowish solid, 237.5 mg, 91% yield.

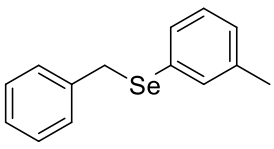
Melting point: 34 – 35 °C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.33 (dt, J = 8.0, 1.8 Hz, 2H), 7.24 – 7.13 (m, 5H), 7.03 (dd, J = 8.0, 2.2 Hz, 2H), 4.06 – 4.01 (m, 2H), 2.30 (d, J = 2.2 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-d): δ 139.0, 137.4, 134.1, 129.9, 128.9, 128.5, 126.9, 126.7, 32.6, 21.2.

GCMS (EI): m/z (%) = 262.05 (10) $[M]^+$, 206.95 (5), 170.95 (5), 91.05 (100).

Benzyl(m-tolyl)selane (3ac-Se).



3ac-Se

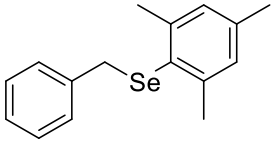
Colorless oil, 234.9 mg, 90% yield.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.19 – 7.07 (m, 7H), 7.04 (dd, J = 8.7, 7.1 Hz, 1H), 6.96 (d, J = 7.7 Hz, 1H), 4.01 (s, 2H), 2.20 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 138.8, 138.8, 134.2, 130.5, 130.4, 129.0, 128.9, 128.5, 128.2, 127.0, 32.2, 21.4.

GCMS (EI): m/z (%) = 262.05 (15) $[M]^+$, 207.00 (10), 182.10 (10), 167.00 (5), 91.10 (100).

Benzyl(mesityl)selane (3ad-Se).



3ad-Se

White solid, 268.8 mg, 93% yield.

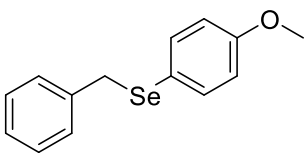
Melting point: 43 – 45 °C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.16 – 7.01 (m, 3H), 6.99 – 6.88 (m, 2H), 6.81 (s, 2H), 3.71 (s, 2H), 2.29 (s, 6H), 2.18 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d): δ 143.6, 139.6, 138.4, 128.6, 128.4, 128.3, 127.8, 126.6, 31.5, 24.3, 21.1.

GCMS (EI): *m*/*z* (%) = 290.05 (10) $[M]^{+}$, 119.10(10), 91.05 (100).

Benzyl(4-methoxyphenyl)selane (3ae-Se).[8]



3ae-Se

White solid, 252.1 mg, 91% yield.

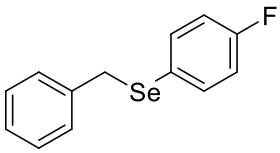
Melting point: 43-45°C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.38 – 7.31 (m, 2H), 7.25 – 7.14 (m, 3H), 7.14 – 7.09 (m, 2H), 6.80 – 6.73 (m, 2H), 3.99 (s, 2H), 3.78 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d): δ 159.6, 139.1, 136.6, 128.9, 128.4, 126.7, 120.0, 114.6, 55.3, 33.2.

GCMS (EI): m/z (%) = 278.00 (20) $[M]^{+}$, 186.90(5), 91.05 (100).

Benzyl(4-fluorophenyl)selane (3af-Se).[16]



3af-Se

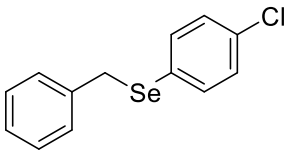
Colorless oil, 233.2 mg, 88% yield.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.51 – 7.40 (m, 2H), 7.34 – 7.24 (m, 3H), 7.23 – 7.16 (m, 2H), 7.04 – 6.90 (m, 2H), 4.10 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d): δ 162.7 (d, J = 247.4 Hz), 138.7, 136.6 (d, J = 8.0 Hz), 128.9, 128.5, 127.0, 124.4 (d, J = 3.5 Hz), 116.2 (d, J = 21.5 Hz), 33.0.

GCMS (EI): m/z (%) = 266.00 (10) $[M]^{+}$, 174.95 (5), 91.05 (100).

Benzyl(4-chlorophenyl)selane (3ag-Se).[15]



3ag-Se

White solid, 250.6 mg, 89% yield.

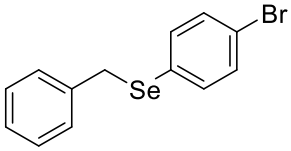
Melting point: 54 - 56 °C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.31 – 7.22 (m, 2H), 7.18 – 7.06 (m, 7H), 3.98 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d): δ 138.3, 135.2, 133.7, 129.1, 128.9, 128.5, 128.4, 127.0, 32.6.

GCMS (EI): m/z (%) = 281.95 (10) $[M]^+$, 190.85 (5), 155.95 (5), 91.05 (100).

Benzyl(4-bromophenyl)selane (3ah-Se).



3ah-Se

White solid, 286.9 mg, 88% yield.

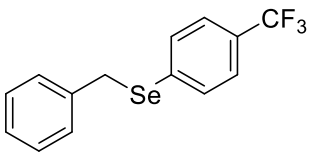
Melting point: 68 - 70 °C

^{1}H NMR (400 MHz, Chloroform-d): δ 7.37 – 7.32 (m, 2H), 7.30 – 7.15 (m, 7H), 4.07 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d): δ 138.3, 135.3, 132.1, 129.1, 128.9, 128.6, 127.1, 121.8, 32.5.

GCMS (EI): m/z (%) = 325.90 (20) $[M]^+$, 234.85 (5), 155.95 (10), 90.95 (100).

Benzyl(4-(trifluoromethyl)phenyl)selane (3ai-Se).[8]



3ai-Se

White solid, 277.2 mg, 88% yield.

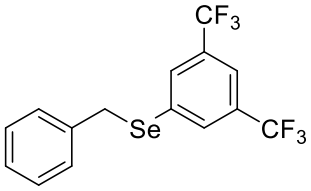
Melting point: 63 - 65 °C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.40 (q, J = 8.4 Hz, 4H), 7.23 – 7.12 (m, 5H), 4.09 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 137.65, 135.97, 132.38, 129.22, 128.91, 128.64, 127.25, 125.68 (q, J = 3.8 Hz), 122.77, 31.79.

GCMS (EI): m/z (%) = 316.05 (5) $[M]^+$, 224.95 (5), 145.05 (5), 91.05 (100)..

Benzyl(3,5-bis(trifluoromethyl)phenyl)selane (3aj-Se).[17]



3aj-Se

White solid, 337.0 mg, 88% yield.

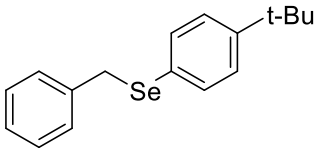
Melting point: 70 - 72 °C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.67 (d, J = 1.6 Hz, 2H), 7.63 – 7.59 (m, 1H), 7.17 – 7.06 (m, 5H), 4.07 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d) δ 136.9, 133.4 – 133.0 (m), 132.9, 131.8 (d, J = 33.5 Hz), 128.8 (d, J = 30.4 Hz), 127.5, 124.3, 121.6, 121.2 – 120.6 (m), 32.4.

GCMS (EI): m/z (%) = 384.00 (10) [M]$^+$, 292.95 (5), 213.00 (5), 90.95 (100).

Benzyl(4-(tert-butyl)phenyl)selane (3ak-Se).[16]



3ak-Se

White solid, 272.7 mg, 90% yield.

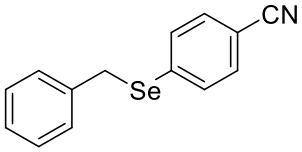
Melting point: 45 - 47 °C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.46 – 7.33 (m, 2H), 7.31 – 7.13 (m, 7H), 4.08 (s, 2H), 1.30 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-d): δ 150.6, 138.8, 133.5, 128.9, 128.5, 127.0, 126.8, 126.1, 34.6, 32.4, 31.3.

GCMS (EI): m/z (%) = 304.10 (20) [M]$^+$, 197.95 (5), 169.95 (5), 117.05 (5), 91.15 (100).

4-(benzylselanyl)benzonitrile (3al-Se).



3al-Se

White solid, 231.2 mg, 85% yield.

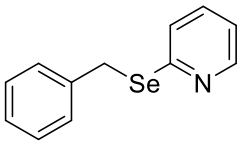
Melting point: 72 - 74 °C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.38 (s, 5H), 7.22 – 7.13 (m, 4H), 4.11 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d): δ 138.7, 137.0, 132.2, 131.8, 128.9, 128.7, 127.4, 118.8, 110.1, 31.5.

GCMS (EI): m/z (%) = 272.95 (5) [M]$^+$,181.90 (5), 91.05 (100).

2-(benzylselanyl)pyridine (3am-Se).[14]



3am-Se

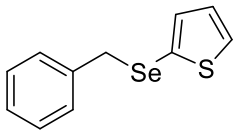
Yellowish oil, 210.8 mg, 85% yield.

^{1}H NMR (400 MHz, Chloroform-d): δ 8.40 (ddd, J = 4.9, 2.0, 1.0 Hz, 1H), 7.38 – 7.26 (m, 3H), 7.22 – 7.05 (m, 5H), 6.93 (dddd, J = 12.3, 7.4, 4.9, 1.1 Hz, 1H), 4.37 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d): δ 155.6, 150.1, 139.1, 136.1, 129.1, 128.6, 126.9, 125.5, 120.6, 29.3.

GCMS (EI): m/z (%) = 249.00 (10) [M]$^+$, 168.10(70), 91.15 (100).

2-(benzylselanyl)thiophene (3an-Se).[8]



3an-Se

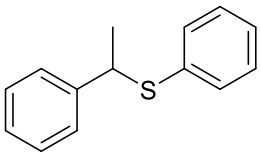
Colorless oil, 227.7 mg, 90% yield.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.35 (dd, J = 5.3, 1.2 Hz, 1H), 7.27 – 7.16 (m, 3H), 7.13 – 7.08 (m, 2H), 7.00 (dd, J = 3.5, 1.2 Hz, 1H), 6.92 (dd, J = 5.3, 3.5 Hz, 1H), 3.98 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-d): δ 138.5, 136.4, 131.3, 128.9, 128.4, 128.0, 127.0, 123.5, 35.6.

GCMS (EI): m/z (%) = 253.95 (15) $[M]^+$, 162.90 (5), 118.95 (5), 91.15 (100).

Phenyl(1-phenylethyl)sulfane (5aa).[18]



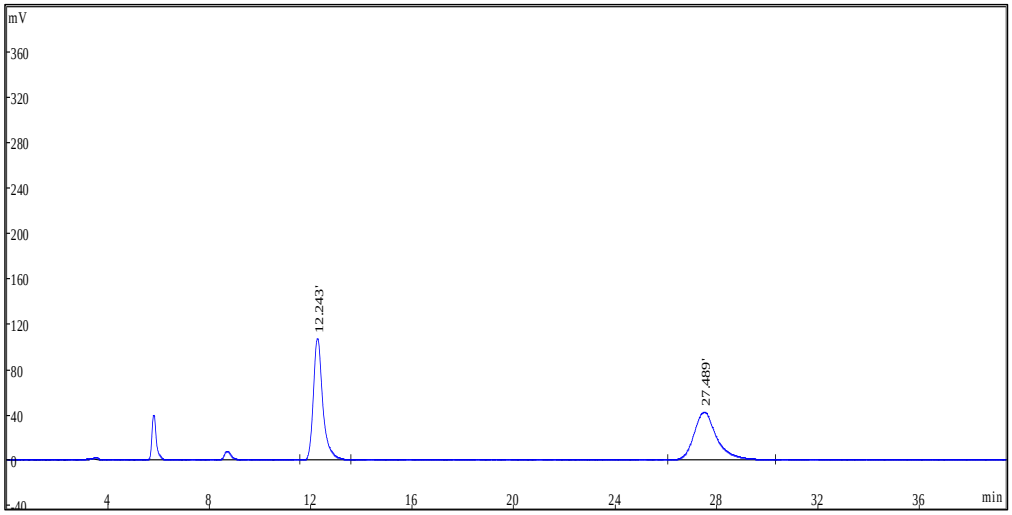
rac-5aa

Yellowish oil, 188.3 mg, 88% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.25 – 7.16 (m, 6H), 7.13 (dtd, *J* = 7.2, 5.6, 5.1, 1.8 Hz, 4H), 4.26 (q, *J* = 7.0 Hz, 1H), 1.55 (d, *J* = 7.0 Hz, 3H).

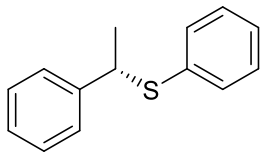
^{13}C NMR (101 MHz, Chloroform-d) δ 143.3, 135.2, 132.5, 129.6, 128.7, 128.4, 127.3, 127.2, 48.0, 22.4.

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol =90/10; flow rate = 1.0 mL/min; t_R(major) =12.24 min, t_R(major) = 27.49 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	12.243	50.16	2592999	106879	13.42	0
2	27.489	49.84	2576227	42092	0.00	1.245
Total		100	5169226	148971		

(S)-phenyl(1-phenylethyl)sulfane (5aa).

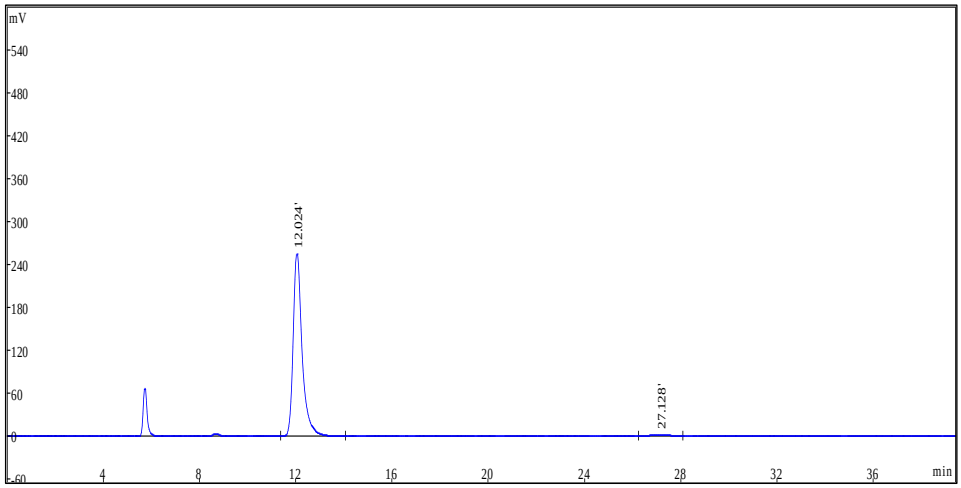


(S)-5aa

Yellowish oil, 188.9 mg, 88% yield, 97% ee.

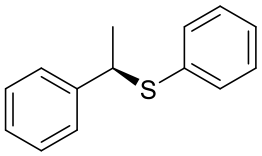
$[\alpha]^{26}_D$ = -38.5 (c= 1.14, EtOAc).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol =90/10; flow rate = 1.0 mL/min; t_R(major) =12.02 min, t_R(minor) = 27.13 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	12.024	98.57	6177029	254660	15.38	-0.000
2	27.128	1.427	89445	1804	0.00	1.256
Total		100	6266474	256464		

(R)-phenyl(1-phenylethyl)sulfane (5aa).

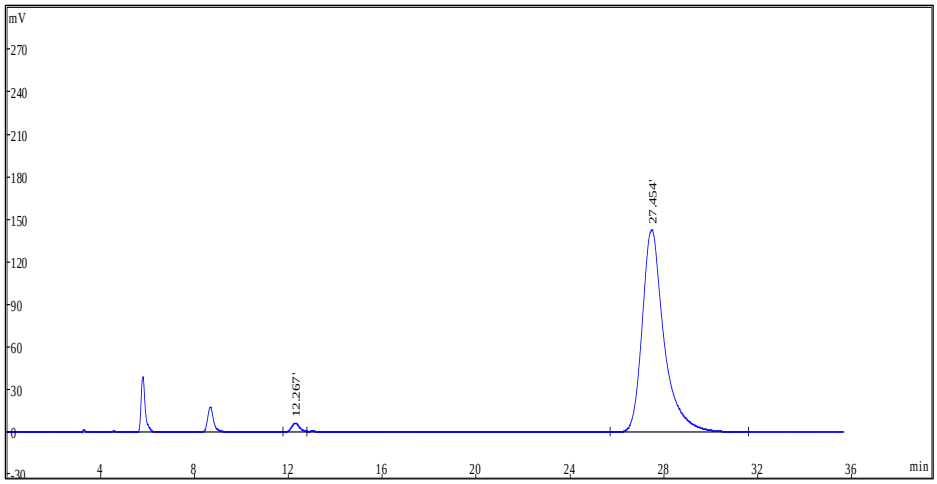


(R)-5aa

Yellowish oil, 190.5 mg, 89% yield, 96% ee.

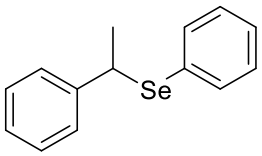
$[\alpha]^{26}_{D}$= 41. 0 (c= 1.10, EtOAc).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol =90/10; flow rate = 1.0 mL/min; t_R(minor) =12.27 min, t_R(major) = 27.45 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	12.267	1.722	156690	6538	13.17	0
2	27.454	98.28	8943601	142519	0.00	1.238
Total		100	9100291	149057		

Phenyl(1-phenylethyl)selane (5ab).[18]



rac-5ab

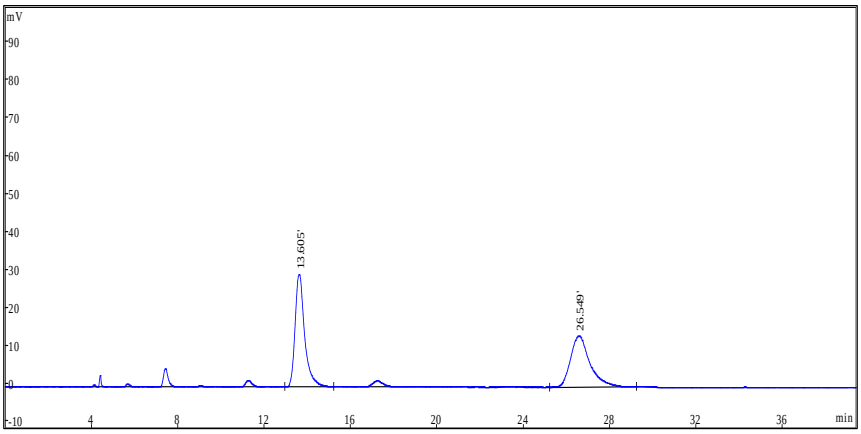
Colorless oil, 219.0 mg, 84% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.45 – 7.41 (m, 2H), 7.29 – 7.14 (m, 9H), 4.45 (q, J = 7.1 Hz, 1H), 1.74 (d, J = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 143.6, 135.5, 129.9, 128.8, 128.3, 127.8, 127.3, 127.0, 42.5, 22.2.

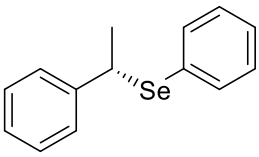
GCMS (EI): m/z (%) = 262.00(5) $[M]^{+}$, 157.00(5), 105.10 (100).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol =99/1; flow rate = 1.0 mL/min; t_R(major) = 13.60 min, t_R(major) = 26.55 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	13.605	50.42	872384	29766	10.55	0
2	26.549	49.58	857801	13622	0.00	0.951
Total		100	1730185	43388		

(S)-phenyl(1-phenylethyl)selane (5ab).

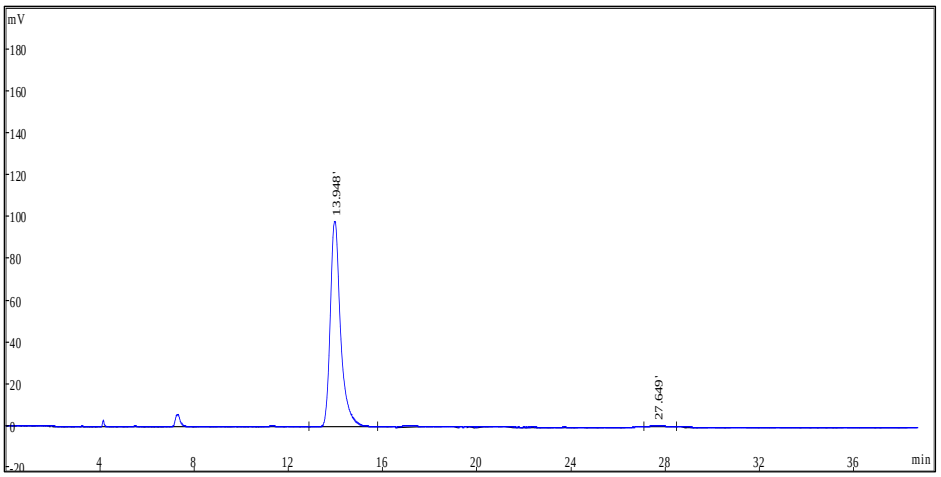


Colorless oil, 221.8 mg, 85% yield, 98% ee.

$[\alpha]^{20}_{D}$ = -40.2 (c=1.02, EtOAc)

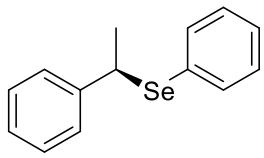
HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol =99/1; flow rate = 1.0 mL/min; t_R(major) = 13.95 min, t_R(minor) = 27.65 min.

(S)-5ab



Rank	Time	Conc.	Area	Height	Resolut.	k
1	13.948	99.19	2870440	98528	15.12	0.000
2	27.649	0.8105	23454	601	0.00	0.982
Total		100	2893894	99129		

(R)-phenyl(1-phenylethyl)selane (5ab).

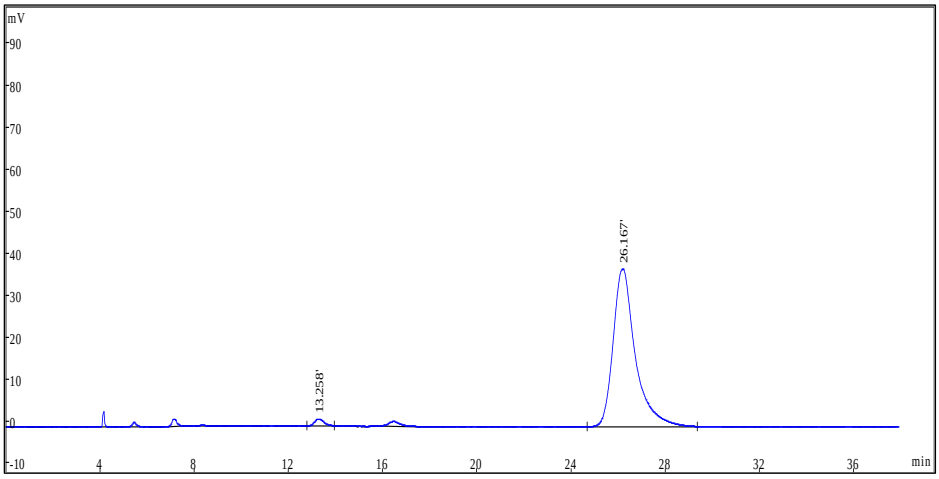


(R)-5ab

Colorless oil, 220.0 mg, 84% yield, 96% ee.

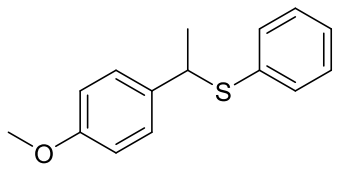
$[\alpha]^{20}_{D}$ = 45.3 (c=1.31, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol =99/1; flow rate = 1.0 mL/min; t_R(minor) = 13.26 min, t_R(major) = 26.17 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	13.258	1.867	45416	1692	10.74	0
2	26.167	98.13	2387633	37563	0.00	0.974
Total		100	2433049	39255		

(1-(4-methoxyphenyl)ethyl)(phenyl)sulfane (5ba).[19]



rac-5ba

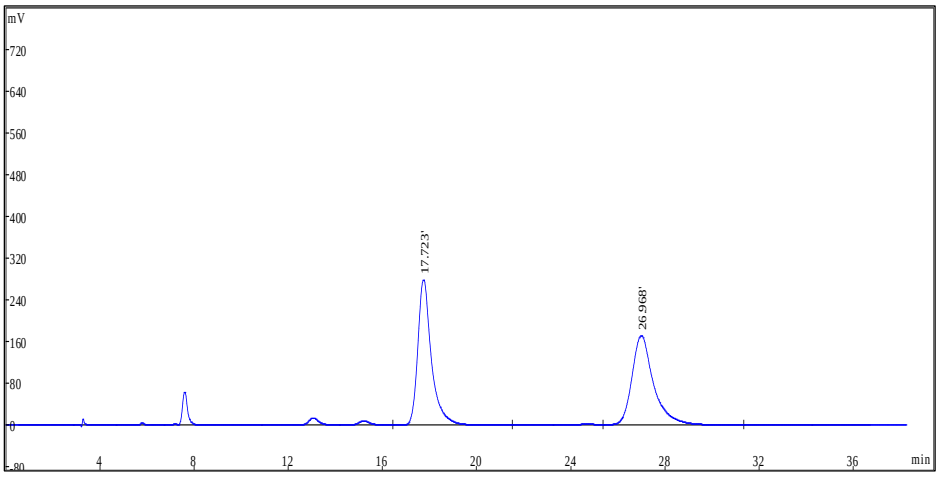
White solid, 226.9 mg, 93% yield.

Melting point : 48 – 50 °C

^{1}H NMR (400 MHz, Chloroform-d) δ 7.27 – 7.18 (m, 2H), 7.17 – 7.04 (m, 5H), 6.81 – 6.62 (m, 2H), 4.24 (q, *J* = 7.0 Hz, 1H), 3.68 (s, 3H), 1.52 (d, *J* = 7.0 Hz, 3H).

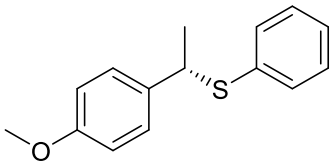
^{13}C NMR (101 MHz, Chloroform-d) δ 158.7, 135.3, 135.3, 132.5, 128.7, 128.4, 127.1, 113.8, 55.3, 47.4, 22.5.

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 90/10; flow rate = 1.0 mL/min; t_R(major) = 17.723 min, t_R(major) = 26.97 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	17.723	49.87	10741586	278221	6.84	0
2	26.968	50.13	10799102	171387	0.00	0.522
Total		100	21540688	449608		

(S)-(1-(4-methoxyphenyl)ethyl)(phenyl)sulfane (**5ba**).



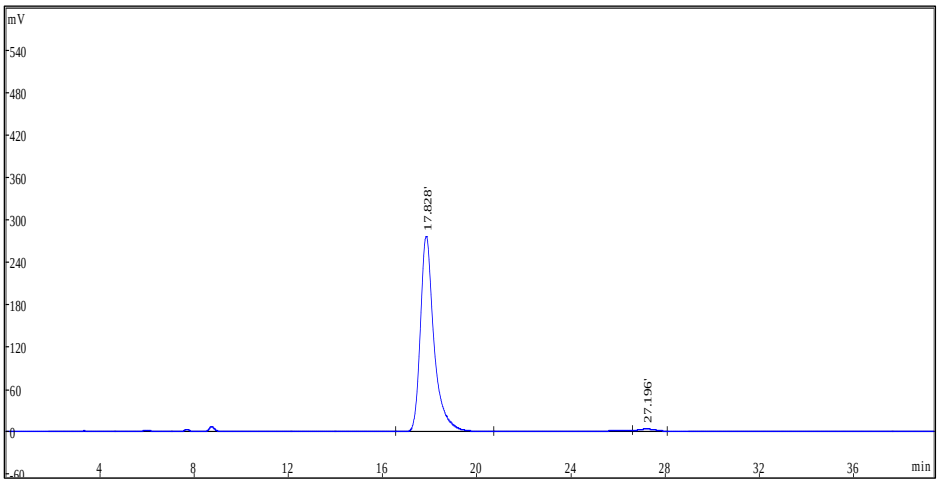
(S)-5ba

White solid, 226.0 mg, 93% yield, 96% ee.

Melting point : 48 – 50 °C

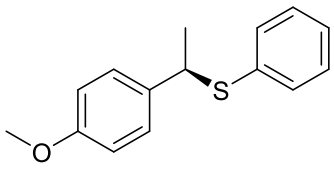
$[\alpha]^{25}_{D}$ = -174.1 (c= 1.11, EtOAc).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 90/10; flow rate = 1.0 mL/min; t_R(major) = 17.83 min, t_R(minor) = 27.20 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	17.828	98.39	10658736	276395	8.06	0.000
2	27.196	1.612	174648	3577	0.00	0.526
Total		100	10833384	279972		

(R)-(1-(4-methoxyphenyl)ethyl)(phenyl)sulfane (5ba).



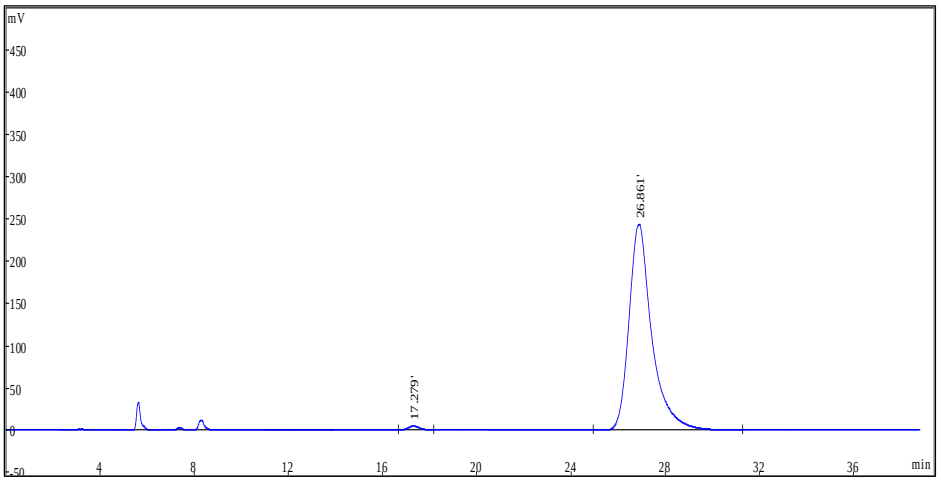
(R)-5ba

White solid, 226.5 mg, 93% yield, 98% ee.

Melting point : 48 – 50 °C

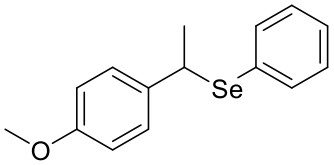
$[\alpha]^{25}_{D}$ = 186.2 (c= 1.05, EtOAc).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 90/10; flow rate = 1.0 mL/min; t_R(minor) = 17.28 min, t_R(major) = 26.86 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	17.279	0.9876	158690	4676	7.26	-0.000
2	26.861	99.01	15909238	243470	0.00	0.555
Total		100	16067928	248146		

(1-(4-methoxyphenyl)ethyl)(phenyl)selane (5bb).[20]



rac-5bb

Yellowish solid, 262.0 mg, 90% yield.

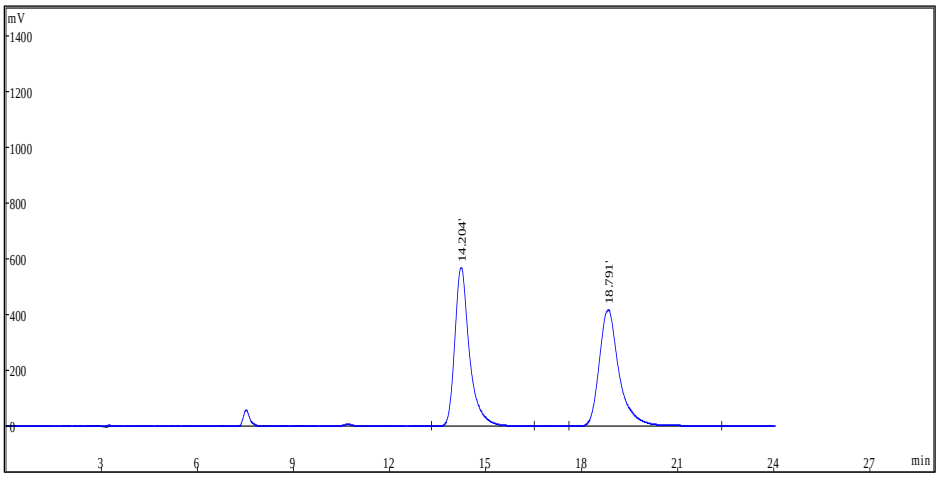
Melting point: 55 - 58 °C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.46 – 7.40 (m, 2H), 7.27 – 7.12 (m, 5H), 6.81 – 6.72 (m, 2H), 4.44 (qd, J = 7.1, 1.9 Hz, 1H), 3.74 (s, 3H), 1.71 (dt, J = 7.1, 1.3 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 158.5, 135.7, 135.5, 130.2, 128.9, 128.4, 127.8, 113.7, 55.3, 42.1, 22.5.

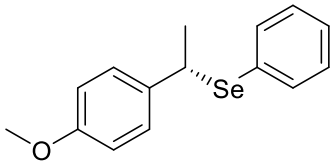
GCMS (EI): m/z (%) = 292.05(5) $[M]^+$, 156.95(5), 135.05(100), 105.10(5), 91.05(5).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 90/10; flow rate = 1.0 mL/min; t_R(minor) = 14.20 min, t_R(major) = 18.79 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	14.204	49.75	17418770	568144	4.73	0
2	18.791	50.25	17596530	416787	0.00	0.323
Total		100	35015300	984931		

(S)-(1-(4-methoxyphenyl)ethyl)(phenyl)selane (5bb).

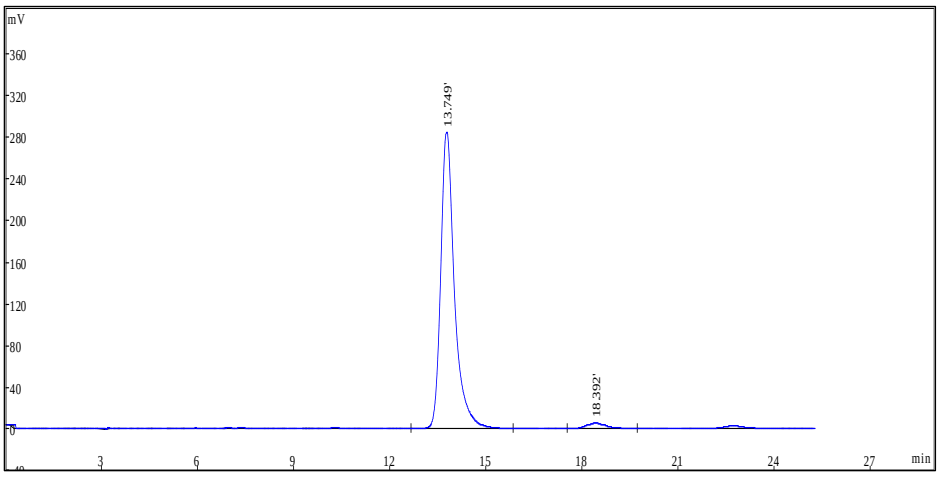


(S)-5bb

Yellowish solid,261.8 mg, 90% yield, 94% ee.

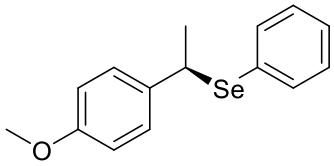
$[\alpha]^{20}_D$ = -167.5 (c=1.20, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 90/10; flow rate = 1.0 mL/min; t_R(major) = 13.75 min, t_R(major) = 18.39 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	13.749	97.06	8194127	285094	5.05	0
2	18.392	2.937	247909	6141	0.00	0.338
Total		100	8442036	291235		

(R)-(1-(4-methoxyphenyl)ethyl)(phenyl)selane (5bb).

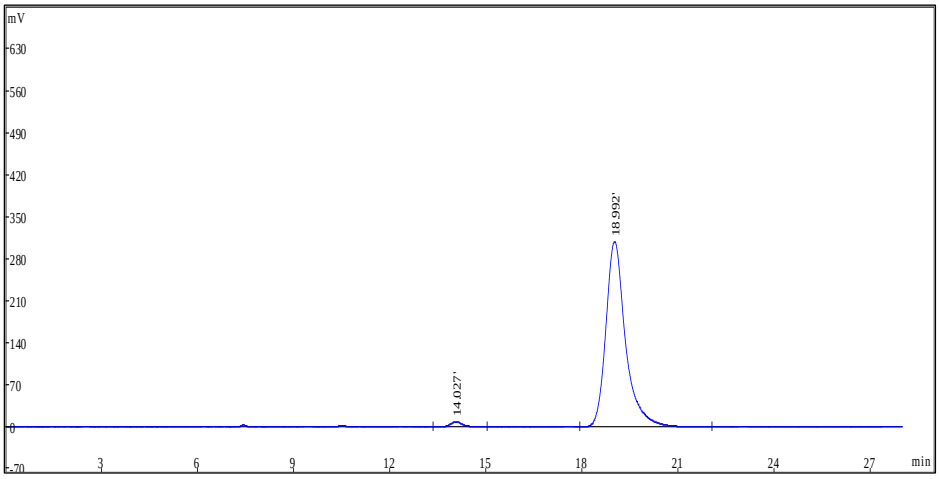


(R)-5bb

Yellowish solid, 261.4 mg, 90% yield, 96% ee.

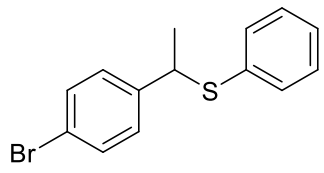
$[\alpha]^{20}_{D}$ = 187.86 (c=1.12, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 90/10; flow rate = 1.0 mL/min; t_R(minor) = 14.03 min, t_R(major) = 18.99 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	14.027	1.821	237618	8782	5.44	0
2	18.992	98.18	12808557	307720	0.00	0.354
Total		100	13046175	316502		

(1-(4-bromophenyl)ethyl)(phenyl)sulfane (5ca).[18]



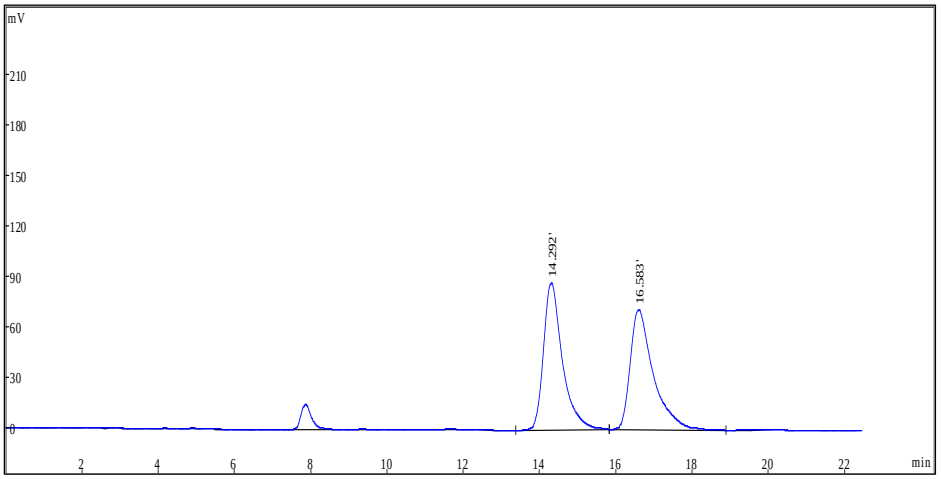
rac-5ca

Colorless oil, 263.7 mg, 90% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.36 – 7.24 (m, 2H), 7.22 – 7.10 (m, 5H), 7.09 – 7.02 (m, 2H), 4.19 (q, *J* = 7.0 Hz, 1H), 1.51 (d, *J* = 7.0 Hz, 3H).

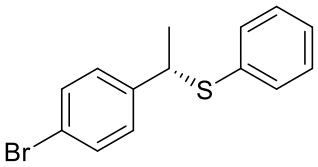
^{13}C NMR (101 MHz, Chloroform-d) δ 142.4, 134.6, 132.7, 131.5, 129.0, 128.8, 127.4, 120.8, 47.5, 22.2.

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99.5/0.5; flow rate = 1.2 mL/min; t_R(major) = 14.29 min, t_R(major) = 16.58 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	14.292	49.95	2964381	87569	2.28	0.000
2	16.583	50.05	2970521	71500	0.00	0.160
Total		100	5934902	159069		

(S)-(1-(4-bromophenyl)ethyl)(phenyl)sulfane (5ca).

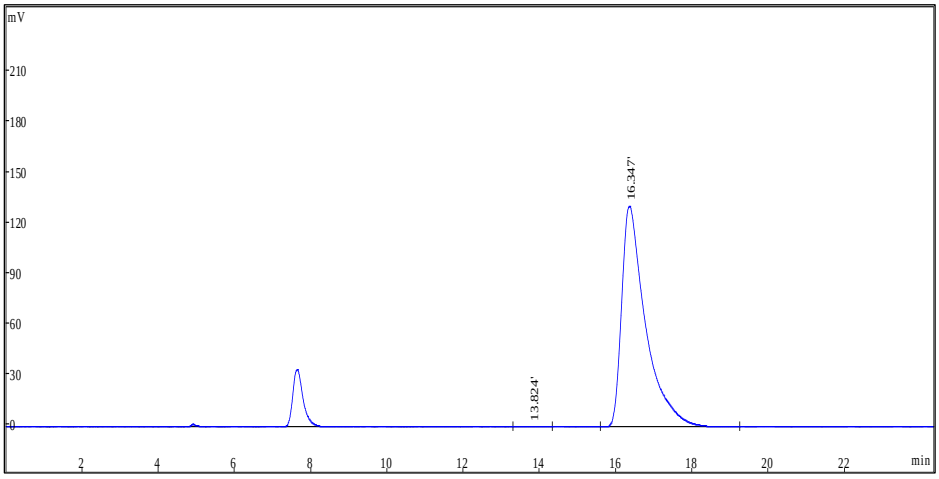


(S)-5ca

Colorless oil, 263.1 mg, 90% yield, 99%ee.

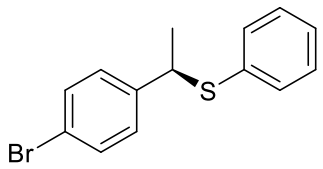
$[\alpha]^{26}_D$ = -187.6 (c= 1.06, EtOAc).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99.5/0.5; flow rate = 1.2 mL/min; t_R(minor) = 13.82 min, t_R(major) = 16.35 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	13.824	0.2536	14308	458	2.56	-0.000
2	16.347	99.75	5627102	131444	0.00	0.182
Total		100	5641410	131902		

(R)-(1-(4-bromophenyl)ethyl)(phenyl)sulfane (5ca).

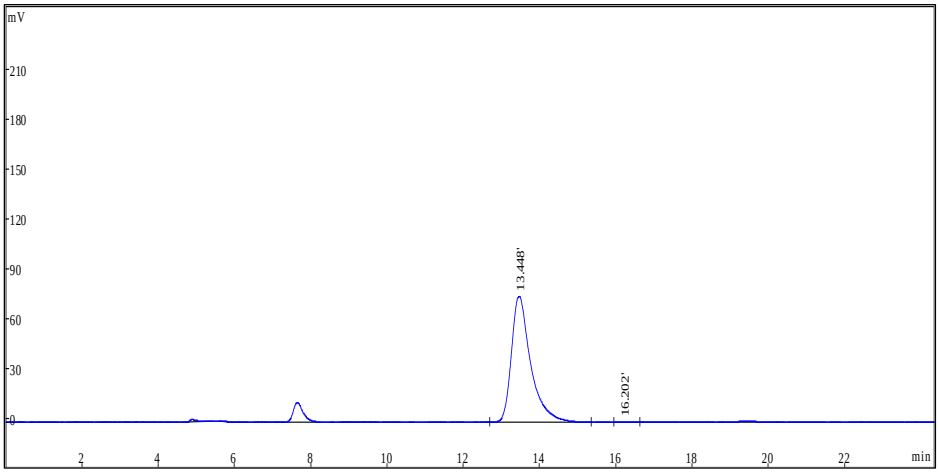


(R)-5ca

Colorless oil, 264.0 mg, 90% yield, 99% ee.

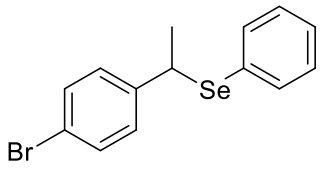
$[\alpha]^{26}_{D}$= 201.4 (c= 1.12, EtOAc).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99.5/0.5; flow rate = 1.2 mL/min; t_R(major) = 13.45 min, t_R(minor) = 16.20 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	13.448	99.86	2591174	75573	3.89	0
2	16.202	0.1433	3720	196	0.00	0.205
Total		100	2594894	75769		

(1-(4-bromophenyl)ethyl)(phenyl)selane (5cb).



rac-5cb

White solid, 299.2 mg, 88% yield.

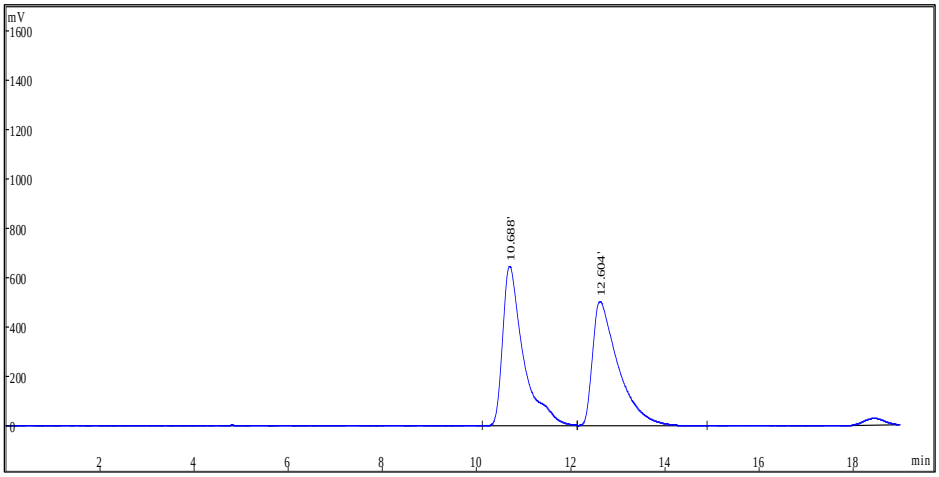
Melting point: 50 - 52 °C.

^{1}H NMR (400 MHz, Chloroform-d): δ 7.36 – 7.31 (m, 2H), 7.30 – 7.25 (m, 2H), 7.24 – 7.11 (m, 4H), 7.05 – 6.91 (m, 2H), 4.31 (q, J = 7.1 Hz, 1H), 1.64 (d, J = 7.1 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-d): δ 142.8, 135.7, 131.4, 129.3, 128.9, 128.9, 128.1, 120.5, 41.6, 22.0.

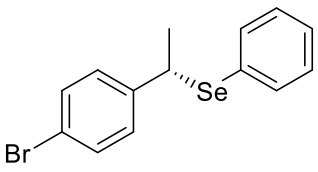
GCMS (EI): m/z (%) = 339.90(5) $[M]^+$, 182.95(90), 156.95(5), 104.05(100).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99.5/0.5; flow rate = 1.2 mL/min; t_R(major) = 10.67 min, t_R(major) = 12.60 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	10.688	50.92	19166816	644803	2.17	0
2	12.604	49.08	18472102	501964	0.00	0.179
Total		100	37638918	1146767		

(S)-(1-(4-bromophenyl)ethyl)(phenyl)selane (5cb).

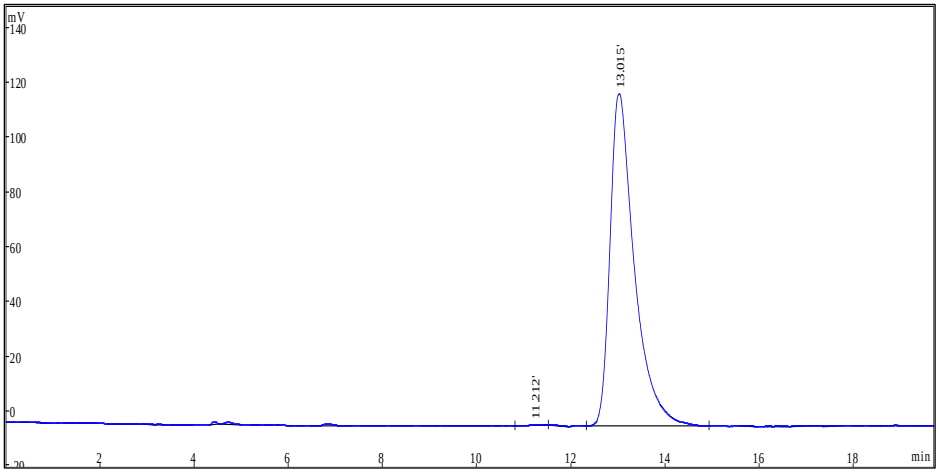


(S)-5cb

White solid, 299.5 mg, 88% yield, 99% ee.

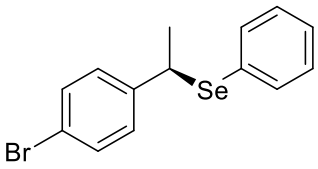
$[\alpha]^{20}_{D}$ = -223.4 (c=0.86, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99.5/0.5; flow rate = 1.2 mL/min; t_R(minor) = 11.21 min, t_R(major) = 13.02 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	11.212	0.06761	2903	194	2.70	0.000
2	13.015	99.93	4290590	121577	0.00	0.161
Total		100	4293493	121771		

(R)-(1-(4-bromophenyl)ethyl)(phenyl)selane (5cb).

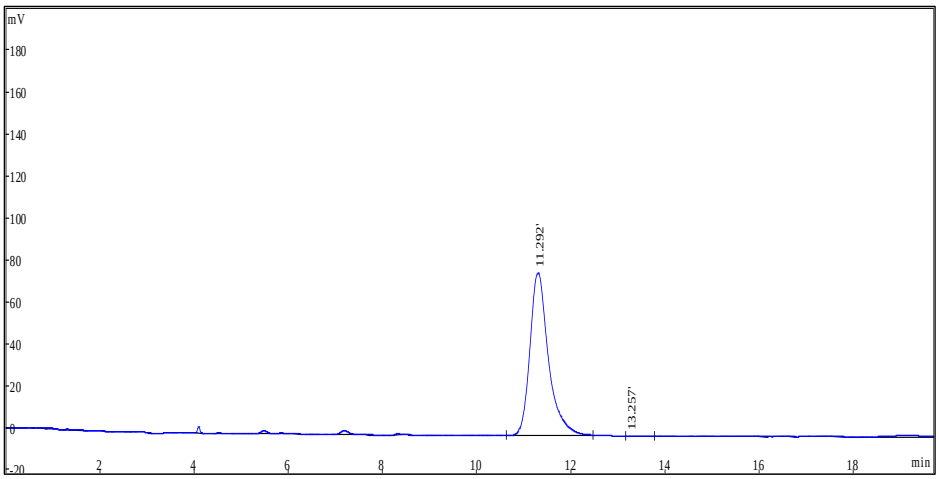


(R)-5cb

White solid, 303.1 mg, 89% yield, 99% ee.

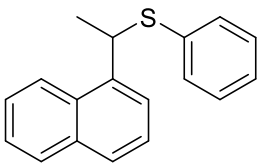
$[\alpha]^{20}_{D}$ = 191.4 (c=1.22, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99.5/0.5; flow rate = 1.2 mL/min; t_R(mjaor) = 11.29 min, t_R(minor) = 13.26 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	11.292	99.89	2073582	77706	2.74	0.000
2	13.257	0.1073	2228	82	0.00	0.174
Total		100	2075810	77788		

(1-(naphthalen-1-yl)ethyl)(phenyl)sulfane (5da).[21]



rac-5da

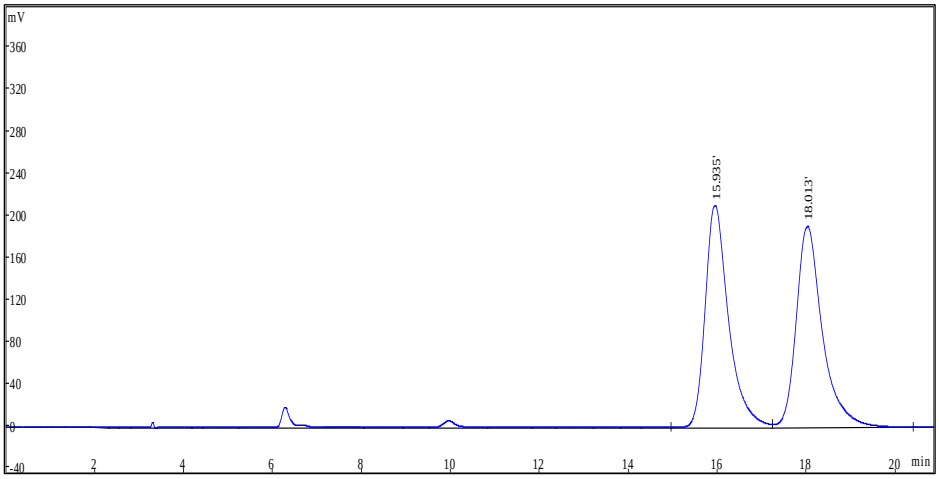
Yellowish solid, 229.7 mg, 87% yield.

Melting point : 75 – 77 °C.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.13 (d, *J* = 8.5 Hz, 1H), 7.81 – 7.70 (m, 1H), 7.64 (dt, *J* = 8.2, 1.1 Hz, 1H), 7.51 – 7.34 (m, 3H), 7.30 (dd, *J* = 8.2, 7.2 Hz, 1H), 7.25 – 7.17 (m, 2H), 7.14 – 7.04 (m, 3H), 5.07 (q, *J* = 6.9 Hz, 1H), 1.68 (d, *J* = 6.9 Hz, 3H).

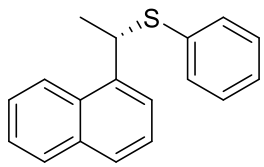
^{13}C NMR (101 MHz, Chloroform-d) δ 138.3, 135.3, 134.0, 132.2, 130.9, 129.1, 128.8, 128.0, 127.1, 126.2, 125.7, 125.5, 124.4, 123.2, 43.0, 22.2.

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 95/5; flow rate = 1.0 mL/min; t_R(major) = 15.94 min, t_R(major) = 18.01 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	15.935	49.46	7696114	211876	2.02	0
2	18.013	50.54	7864464	191771	0.00	0.130
Total		100	15560578	403647		

(S)-(1-(naphthalen-1-yl)ethyl)(phenyl)sulfane (5da).

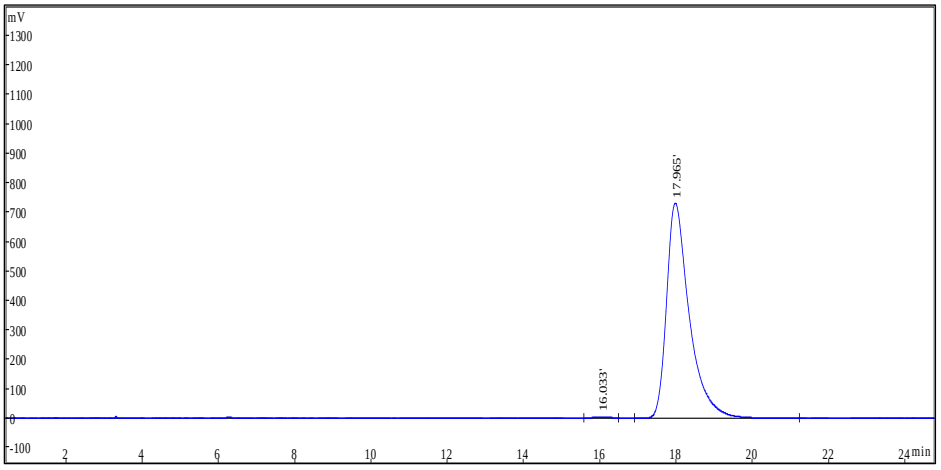


(S)-5da

Yellowish solid, 232.3 mg, 88% yield, 99% ee.

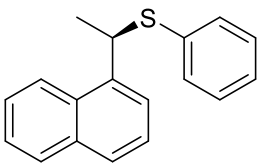
$[\alpha]^{26}_D$ = -72.4 (c= 1.03, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 95/5; flow rate = 1.0 mL/min; t_R(minor) = 16.03 min, t_R(major) = 17.96 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	16.033	0.4534	135410	4954	2.13	0
2	17.965	99.55	29732387	730240	0.00	0.120
Total		100	29867797	735194		

(R)-(1-(naphthalen-1-yl)ethyl)(phenyl)sulfane (5da).

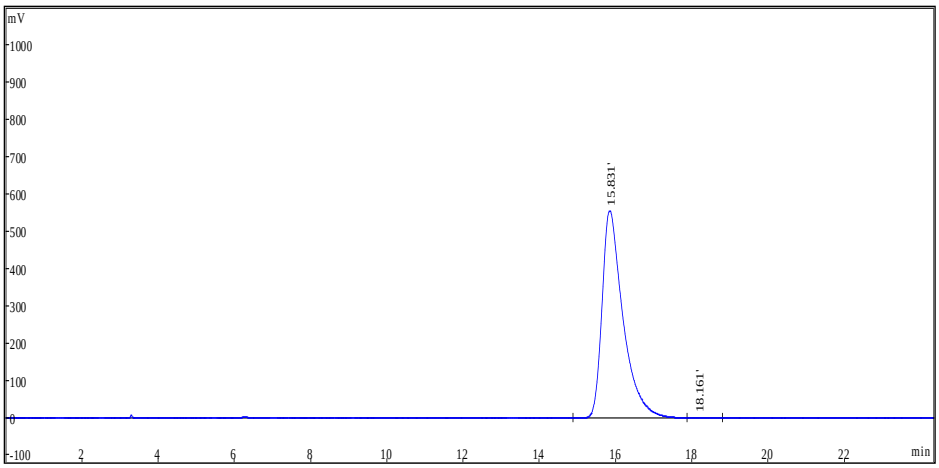


Yellowish solid, 230.0 mg, 87% yield, 99% ee.

$[\alpha]^{26}_{D}$ = 86.5 (c= 0.96, EtOAc).

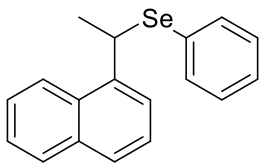
HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 95/5; flow rate = 1.0 mL/min; t_R(major) = 15.83 min, t_R(minor) = 18.16 min.

(R)-5da



Rank	Time	Conc.	Area	Height	Resolut.	k
1	15.831	99.82	20775923	554615	4.68	0.000
2	18.161	0.1829	38070	955	0.00	0.147
Total		100	20813993	555570		

(1-(naphthalen-1-yl)ethyl)(phenyl)selane (5db).



rac-5db

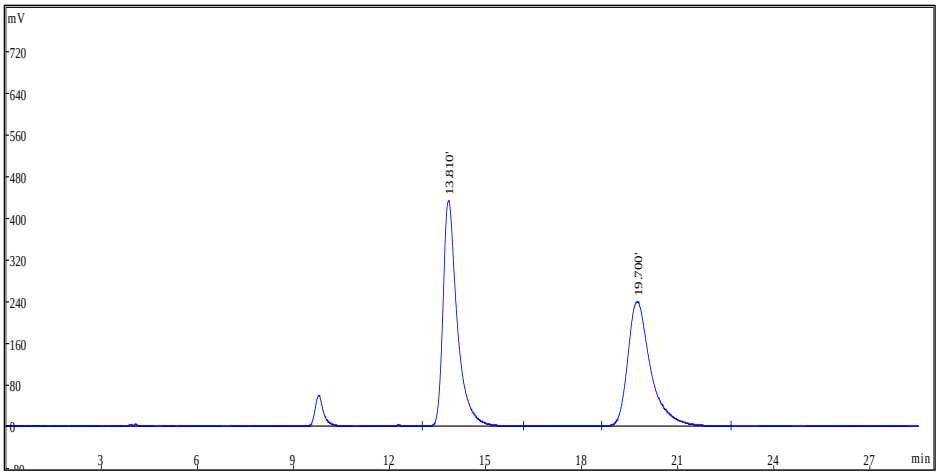
Colorless oil, 270.6 mg, 87% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.11 (d, J = 8.5 Hz, 1H), 7.73 (dd, J = 8.0, 1.5 Hz, 1H), 7.61 (dt, J = 7.9, 1.1 Hz, 1H), 7.46 – 7.21 (m, 6H), 7.19 – 7.00 (m, 3H), 5.10 (q, J = 7.0 Hz, 1H), 1.81 (d, J = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 138.8, 135.7, 134.0, 130.7, 129.9, 129.0, 128.9, 128.0, 127.8, 126.1, 125.7, 125.4, 124.1, 123.4, 37.4, 22.5.

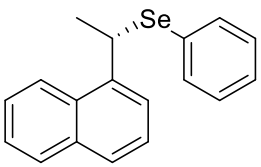
GCMS (EI): m/z (%) = 312.00(5) $[M]^{+}$, 207.00(5), 155.10(100), 141.10(10), 115.05(10).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 95/5; flow rate = 0.8 mL/min; t_R(major) = 13.81 min, t_R(major) = 19.70 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	13.810	52.51	12730189	431052	5.69	0
2	19.700	47.49	11511959	237980	0.00	0.427
Total		100	24242148	669032		

(S)-(1-(naphthalen-1-yl)ethyl)(phenyl)selane (5db).

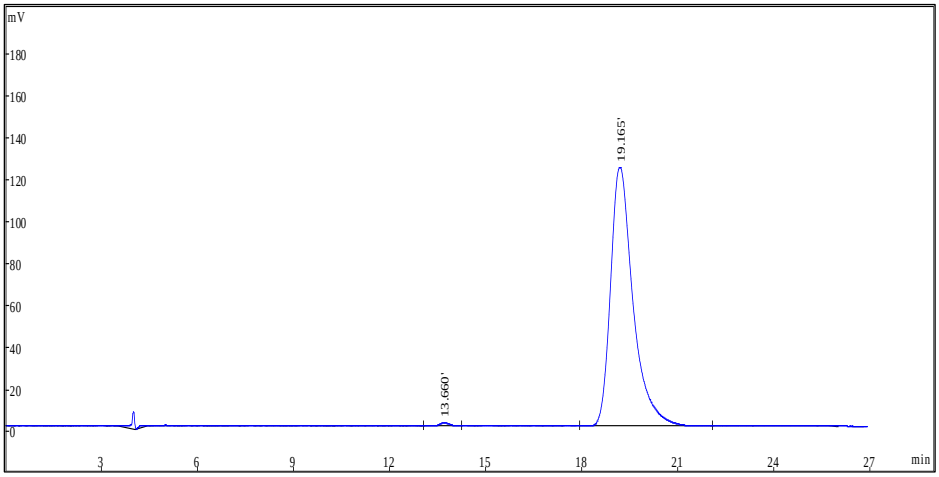


(S)-5db

Colorless oil, 273.8mg, 88% yield, 98% ee.

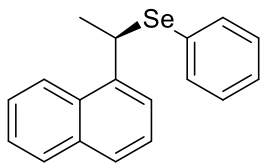
$[\alpha]^{20}_D$ = -71.6 (c=0.96, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 95/5; flow rate = 0.8 mL/min; t_R(minor) = 13.66 min, t_R(major) = 19.16 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	13.660	0.7502	44791	1828	5.72	0
2	19.165	99.25	5925446	123639	0.00	0.403
Total		100	5970237	125467		

(R)-(1-(naphthalen-1-yl)ethyl)(phenyl)selane (5db).

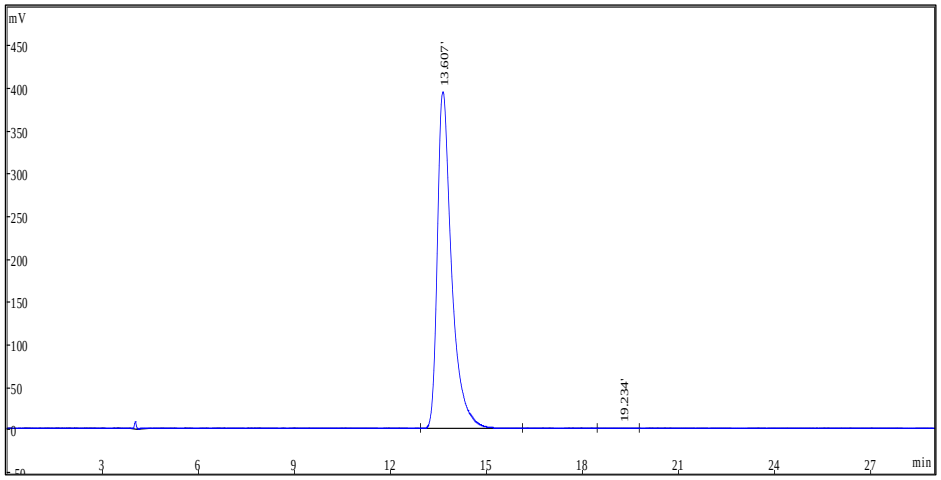


(R)-5db

Colorless oil, 270.1 mg, 87% yield, 99% ee.

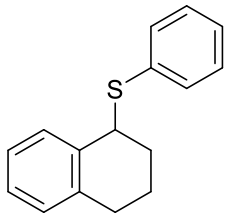
$[\alpha]^{20}_{D}$ = 63.8 (c=1.02, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 95/5; flow rate = 0.8 mL/min; t_R(major) = 13.61 min, t_R(minor) = 19.23 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	13.607	99.89	11747952	393634	6.33	0
2	19.234	0.1125	13234	358	0.00	0.414
Total		100	11761186	393992		

Phenyl(1,2,3,4-tetrahydronaphthalen-1-yl)sulfane (5ea).[22]



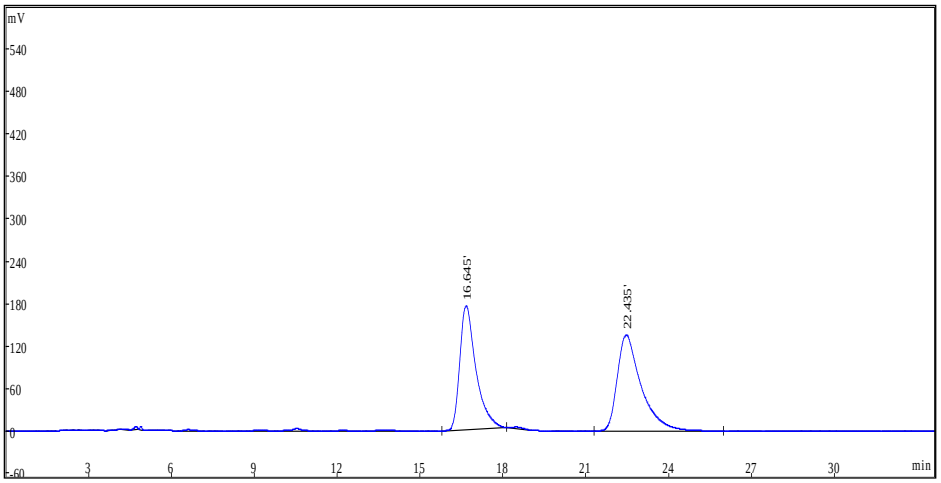
rac-5ea

Colorless oil, 221.1 mg, 92% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.57 – 7.51 (m, 2H), 7.49 (ddt, *J* = 8.4, 5.7, 2.8 Hz, 1H), 7.43 – 7.34 (m, 2H), 7.34 – 7.27 (m, 1H), 7.25 – 7.19 (m, 2H), 7.16 (dt, *J* = 5.6, 3.2 Hz, 1H), 4.65 (t, *J* = 4.1 Hz, 1H), 3.00 – 2.71 (m, 2H), 2.39 – 2.20 (m, 1H), 2.18 – 1.95 (m, 2H), 1.83 (dddd, *J* = 15.3, 5.7, 3.9, 2.4 Hz, 1H).

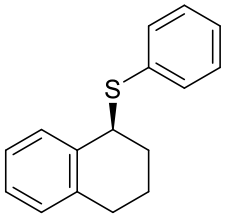
^{13}C NMR (101 MHz, Chloroform-d) δ 137.7, 136.2, 135.6, 131.9, 130.6, 129.3, 129.0, 127.1, 127.0, 125.8, 47.7, 29.2, 28.6, 18.7.

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99/1; flow rate = 0.8 mL/min; t_R(major) = 16.64 min, t_R(major) = 22.44 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	16.645	46.48	6890121	174535	4.44	0
2	22.435	53.52	7934023	135521	0.00	0.348
Total		100	14824144	310056		

(S)-phenyl(1,2,3,4-tetrahydronaphthalen-1-yl)sulfane (5ea).

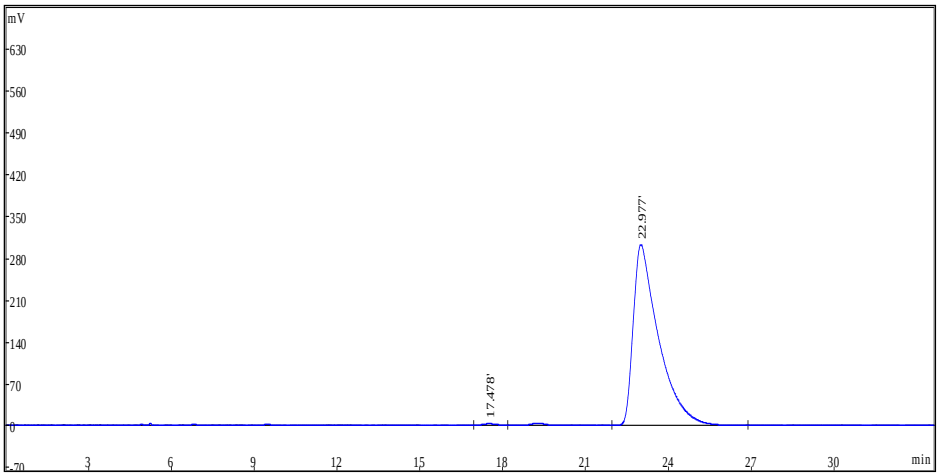


(S)-5ea

Colorless oil, 220.8 mg, 92% yield, 99% ee.

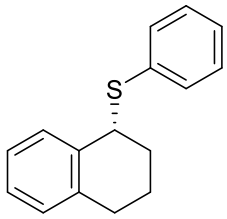
$[\alpha]^{25}_D$ = 52.1 (c= 1.08, EtOAc).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99/1; flow rate = 0.8 mL/min; t_R(minor) = 17.48 min, t_R(major) = 22.98 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	17.478	0.4927	93708	2865	4.34	0.000
2	22.977	99.51	18926448	302033	0.00	0.315
Total		100	19020156	304898		

(R)-phenyl(1,2,3,4-tetrahydronaphthalen-1-yl)sulfane (5ea).

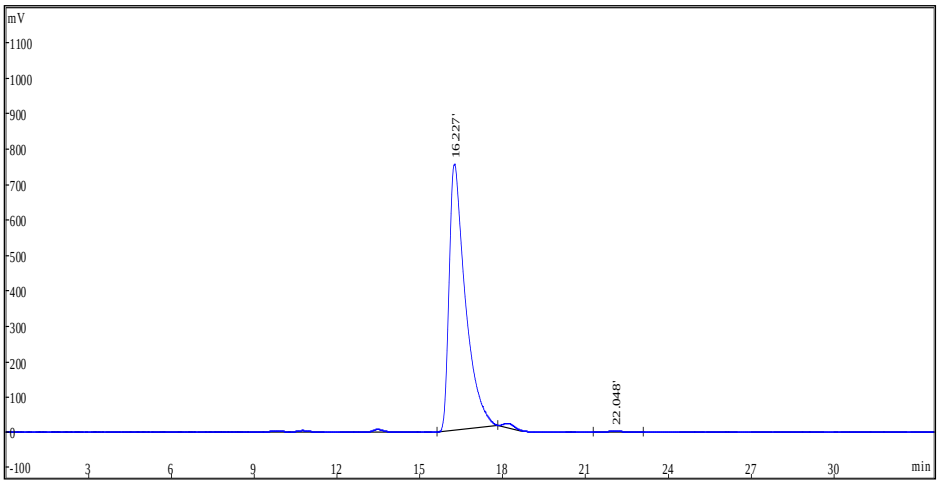


(R)-5ea

Colorless oil, 220.3 mg, 92% yield, 98% ee.

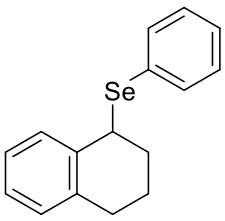
$[\alpha]^{25}_D$ = -54.9 (c= 0.82, EtOAc).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99/1; flow rate = 0.8 mL/min; t_R(major) = 17.23 min, t_R(minor) = 22.05 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	16.227	99.43	29665645	750270	5.07	0.000
2	22.048	0.5679	169419	3618	0.00	0.359
Total		100	29835064	753888		

Phenyl(1,2,3,4-tetrahydronaphthalen-1-yl)selane (5eb).[23]



rac-5eb

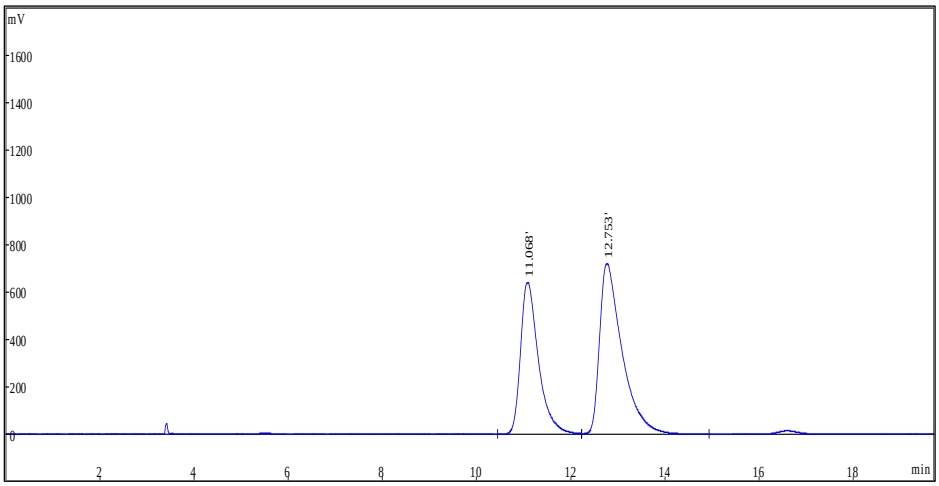
Colorless oil, 255.4 mg 89% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.58 (qd, J = 5.3, 4.7, 2.8 Hz, 2H), 7.33 (dq, J = 5.2, 2.6 Hz, 1H), 7.26 (dq, J = 4.1, 2.0 Hz, 3H), 7.09 (dq, J = 5.0, 2.7 Hz, 2H), 7.03 (q, J = 3.1 Hz, 1H), 4.76 (q, J = 3.9 Hz, 1H), 2.89 – 2.65 (m, 2H), 2.24 (ddt, J = 14.2, 9.4, 5.8, 2.8 Hz, 1H), 2.10 – 1.91 (m, 2H), 1.85 – 1.66 (m, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 137.2, 136.4, 134.8, 131.2, 130.6, 129.4, 129.2, 127.7, 126.9, 125.7, 44.6, 29.2, 29.1, 19.2.

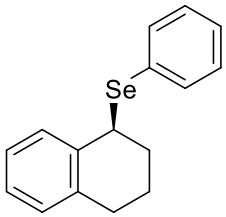
GCMS (EI): m/z (%) = 288.05(5) $[M]^{+}$, 156.95(5), 131.05(100), 141.10(10), 115.05(15), 91.05(25).

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99/1; flow rate = 1.0 mL/min; t_R(major) = 11.07 min, t_R(major) = 12.75 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	11.068	41.47	16405899	639713	2.19	-0.000
2	12.753	58.53	23159045	717083	0.00	0.152
Total		100	39564944	1356796		

(S)-phenyl(1,2,3,4-tetrahydronaphthalen-1-yl)selane (5eb).

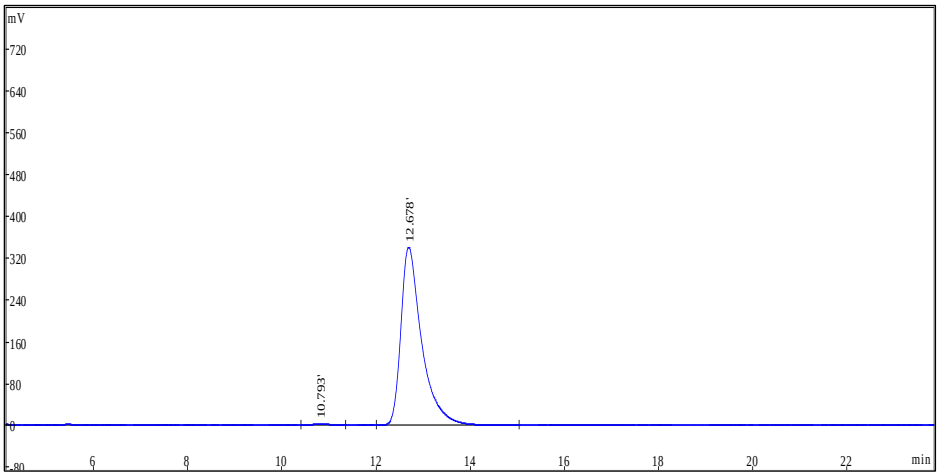


(S)-5eb

Colorless oil, 256.1 mg 89% yield. 98%ee.

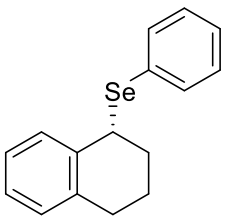
$[\alpha]^{20}_{D}$ = 33.7 (c=1.55, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99/1; flow rate = 1.0 mL/min; t_R(minor) = 11.79 min, t_R(major) = 12.68 min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	10.793	0.6458	66396	2749	2.62	0
2	12.678	99.35	10214974	340156	0.00	0.175
Total		100	10281370	342905		

(R)-phenyl(1,2,3,4-tetrahydronaphthalen-1-yl)selane (5eb).

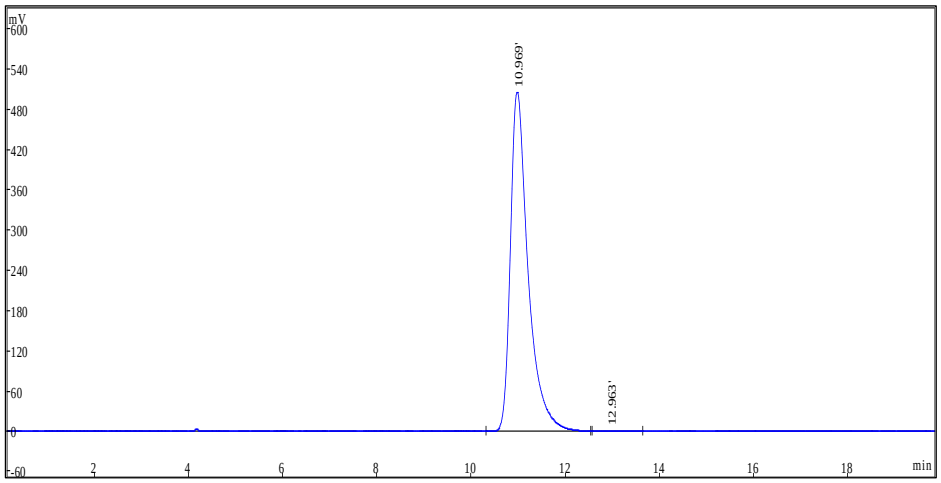


(R)-5eb

Colorless oil, 255.5 mg 89% yield, 99%ee.

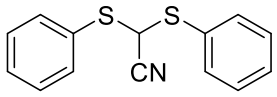
$[\alpha]^{20}_{D}$ = -33.8 (c=1.53, EtOAc)

HPLC: Chiralcel OJ-H column; λ = 254 nm; hexane/isopropanol = 99/1; flow rate = 1.0 mL/min; t_R(major) = 10.97 min, t_R(minor) = 12.96min.



Rank	Time	Conc.	Area	Height	Resolut.	k
1	10.969	99.8	12803898	505629	2.93	0
2	12.963	0.1976	25345	978	0.00	0.182
Total		100	12829243	506607		

2,2-bis(phenylthio)acetonitrile (6).[24]



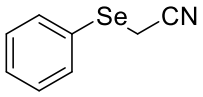
6

Yellowish oil, 92.6 mg, 36% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.62 – 7.52 (m, 4H), 7.38 – 7.29 (m, 6H), 4.74 (s, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 135.1, 130.2, 130.1, 129.5, 116.0, 42.3.

2-(phenylselanyl)acetonitrile (7).[25]



7

Yellowish oil, 101.9 mg, 52% yield.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.66 – 7.52 (m, 2H), 7.32 – 7.21 (m, 3H), 3.25 (s, 2H).

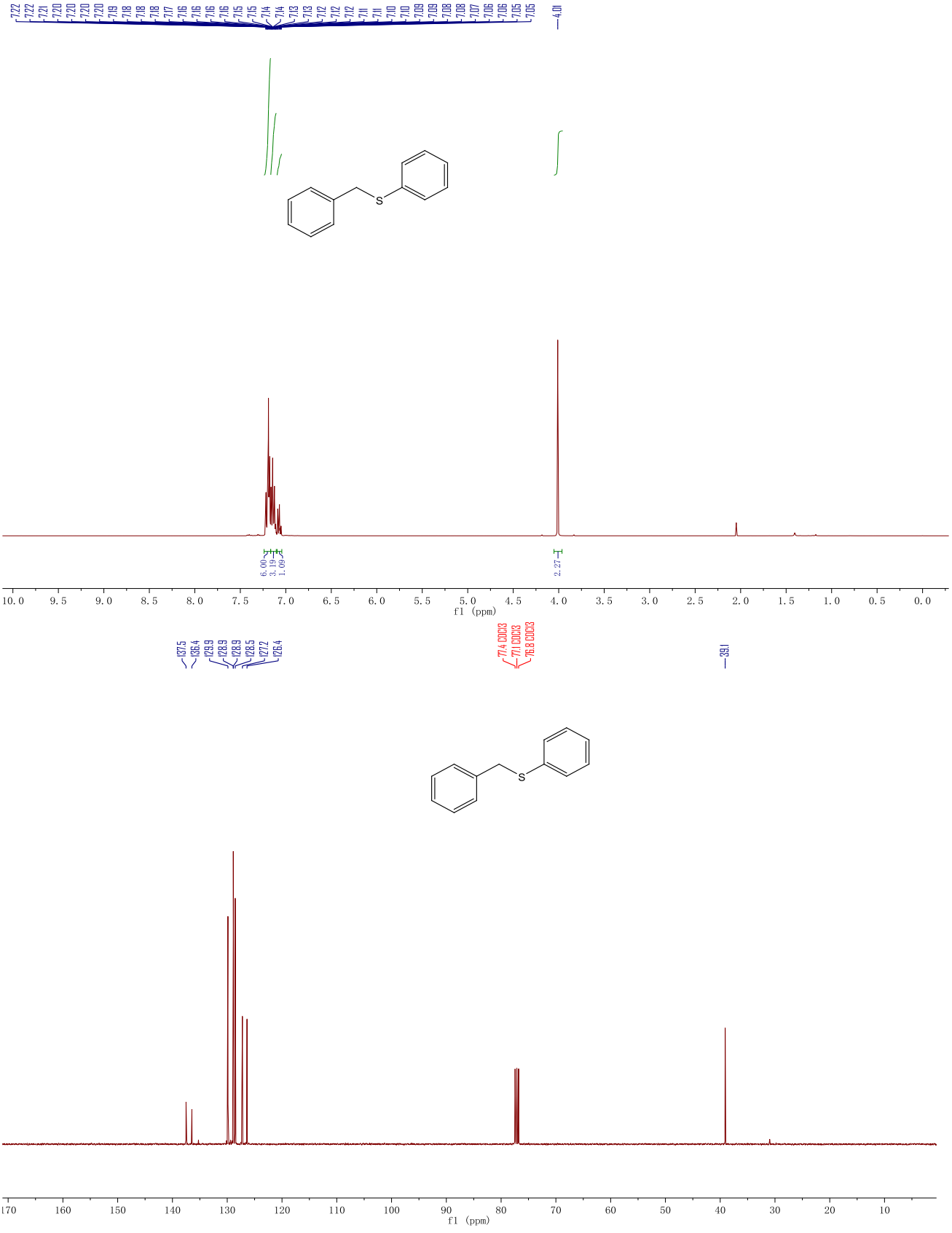
^{13}C NMR (101 MHz, Chloroform-d) δ 134.8, 129.7, 129.3, 127.2, 117.6, 8.1.

References

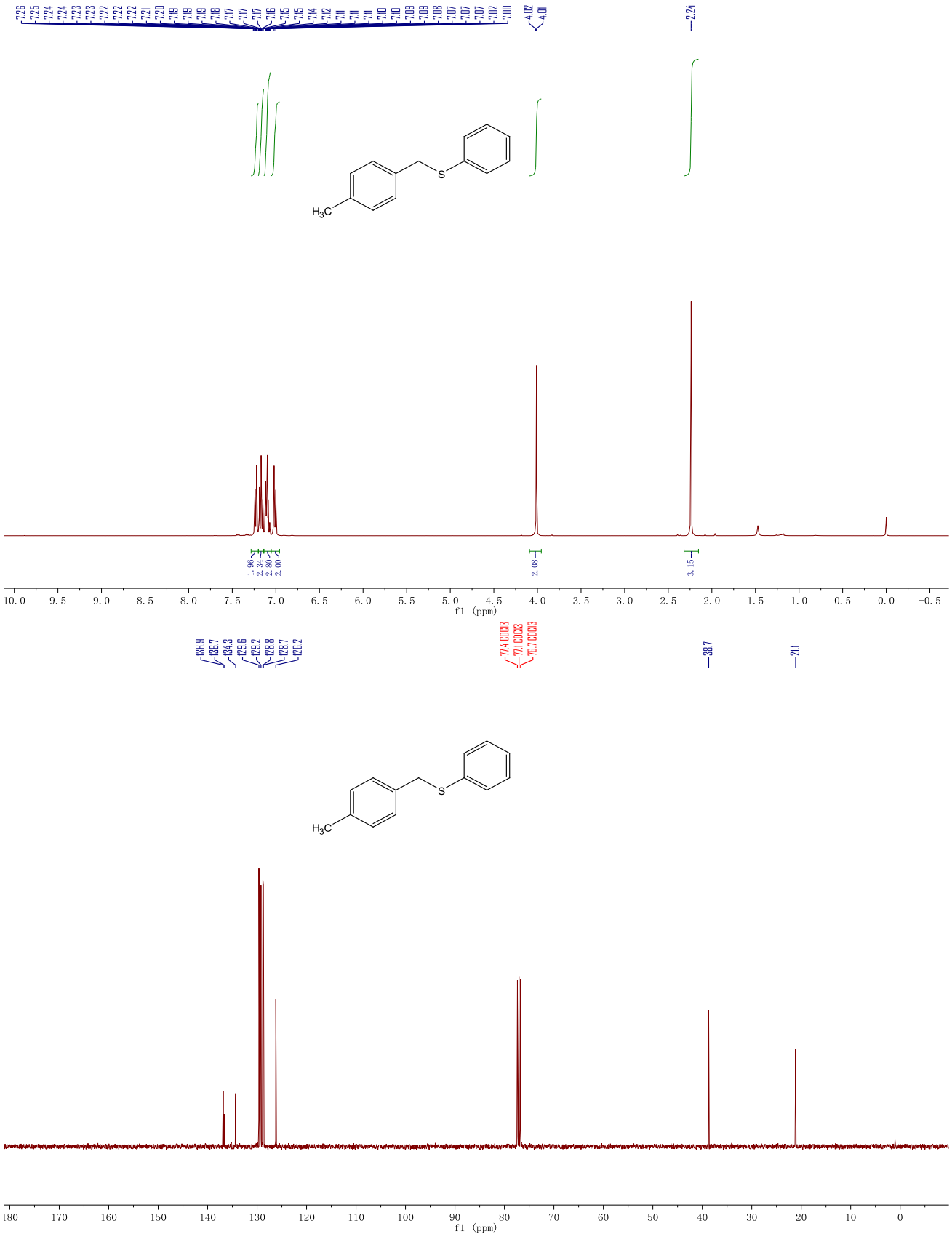
1. D. Singh, A. M. Deobald, L. R. Camargo, G. Tabarelli, O. E. Rodrigues and A. L. Braga, *Org. Lett.* 2010, **12**, 3288.
2. L. Wang, W.-Y. Zhou, S. C. Chen, M. Y. He and Q. Chen, *Adv. Synth. Catal.* 2012, **354**, 839.
3. S. Narayanaperumal, E. E. Alberto, K. Gul, C. Y. Kawasoko, L. Dornelles, O. E. D. Rodrigues and A. L. Braga, *Tetrahedron* 2011, **67**, 4723.
4. Y. Zhang, Y. Li, X. Zhang and X. Jiang, *Chem. Commun.* 2015, **51**, 941.
5. K. Gul, S. Narayanaperumal, L. Dornelles, O. E. D. Rodrigues and A. L. Braga, *Tetrahedron Lett.* 2011, **52**, 3592.
6. T. Liu, R. Qiu, L. Zhu, S. F. Yin, C. T. Au and N. Kambe, *Chem. Asian. J.* 2018, **13**, 3833.
7. B. C. Ranu, T. Mandal, *J. Org. Chem.* 2004, **69**, 5793.
8. F.-H. Cui, J. Chen, S.-X. Su, Y.-l. Xu, H.-S. Wang and Y.-M. Pan, *Adv. Synth. Catal.* 2017, **359**, 3950.
9. P. G. Gassman, T. Miura and A. Mossman, *J. Org. Chem.* 1982, **47**, 954.
10. B. C. Ranu and T. Mandal, *Tetrahedron Lett.* 2006, **47**, 5677.
11. S. F. Lim, B. L. Harris, P. Blanc and J. M. White, *J. Org. Chem.* 2011, **76**, 1673.
12. Y. Li, J. Pu and X. Jiang, *Org. Lett.* 2014, **16**, 2692.
13. S. B. Zhang, X. Liu, M. Y. Gao and Z. B. Dong, *J. Org. Chem.* 2018, **83**, 14933.
14. X. Ma, Q. Liu, X. Jia, C. Su and Q. Xu, *RSC Adv.* 2016, **6**, 56930.
15. S. Narayanaperumal, E. E. Alberto, F. M. de Andrade, E. J. Lenardao, P. S. Taube and A. L. Braga, *Org Biomol Chem* 2009, **7**, 4647.
16. H. Wang, S. Chen, G. Liu, H. Guan, D. Zhong, J. Cai, Z. Zheng, J. Mao and P. J. Walsh, *Organometallics* 2018, **37**, 4086.
17. V. Nascimento, E. E. Alberto, D. W. Tondo, D. Dambrowski, M. R. Detty, F. Nome and A. L. Braga, *J. Am. Chem. Soc.* 2012, **134**, 138.
18. Y. Gui and S. K. Tian, *Org. Lett.* 2017, **19**, 1554.
19. L. D. S. Yadav, Garima and R. Kapoor, *Synth. Commun.* 2010, **41**, 100.
20. N. Toshiki, N. Yutaka and S. Noboru, *Chem. Lett.* 2003, **32**, 918.
21. S. Chand, A. K. Pandey, R. Singh, S. Kumar and K. N. Singh, *Chem. Asian. J.* 2019, **14**, 4712.
22. K. Kuciński and G. Hreczycho, *Eur. J. Org. Chem.* 2017, **2017**, 5572.
23. H. Abe, A. Yamasaki and T. Harayama, *Chem. Pharm. Bull.* 1998, **46**, 1311.
24. H. Ishibashi, M. Okada, K. Sato, M. Ikeda, K. Ishiyama and Y. Tamura, *Chem. Pharm. Bull.* 1985, **33**, 90.
25. M. A. Arrica and T. Wirth, *Eur. J. Org. Chem.* 2005, **2005**, 395.

Copies of ^{1}H and ^{13}C NMR Spectra for Compounds 3, 5, 6, 7

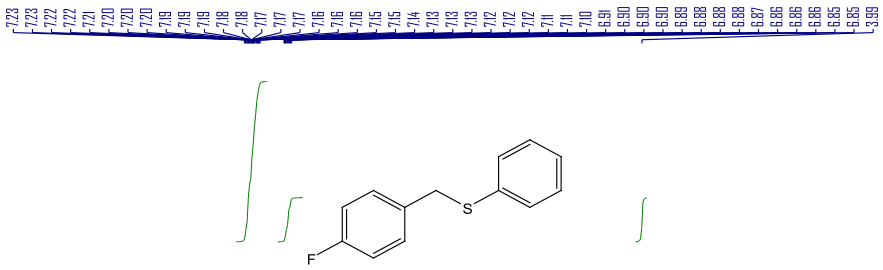
Product 3aa-S

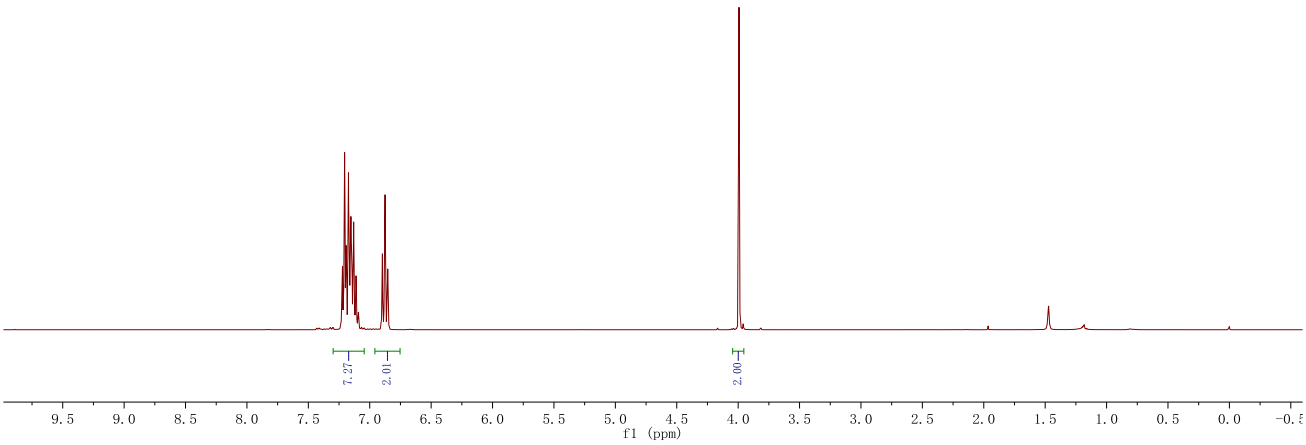


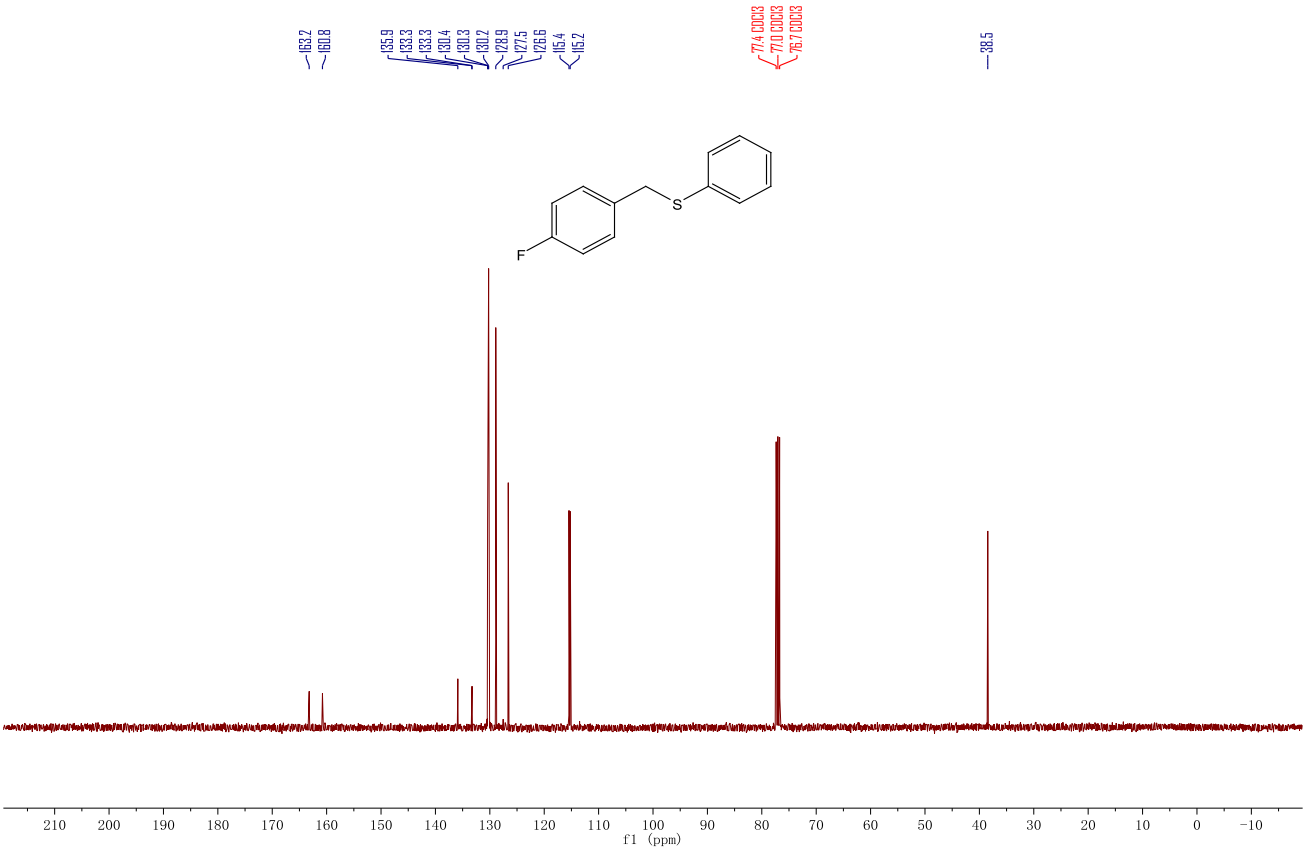
Product 3ba-S



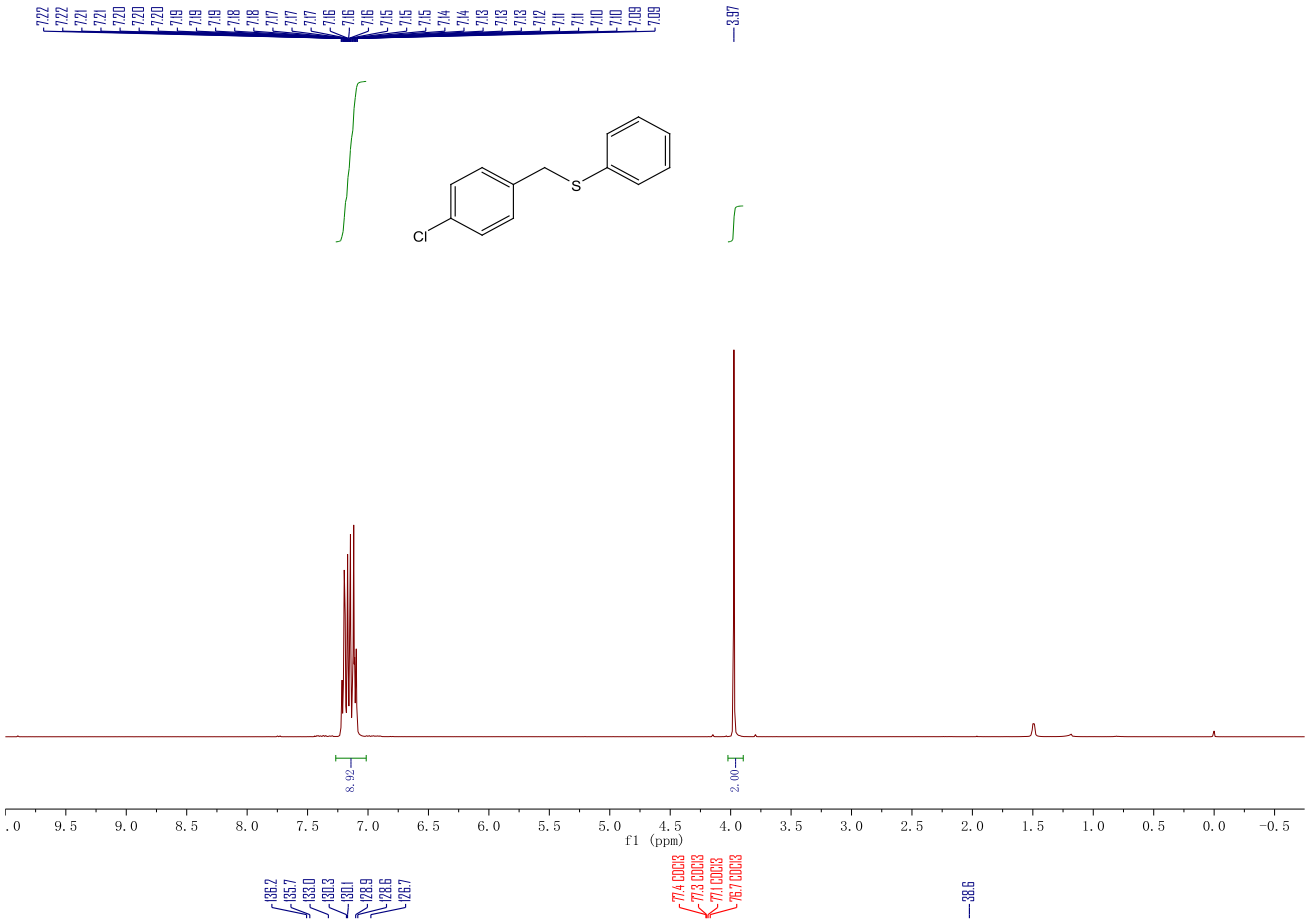
Product 3ca-S

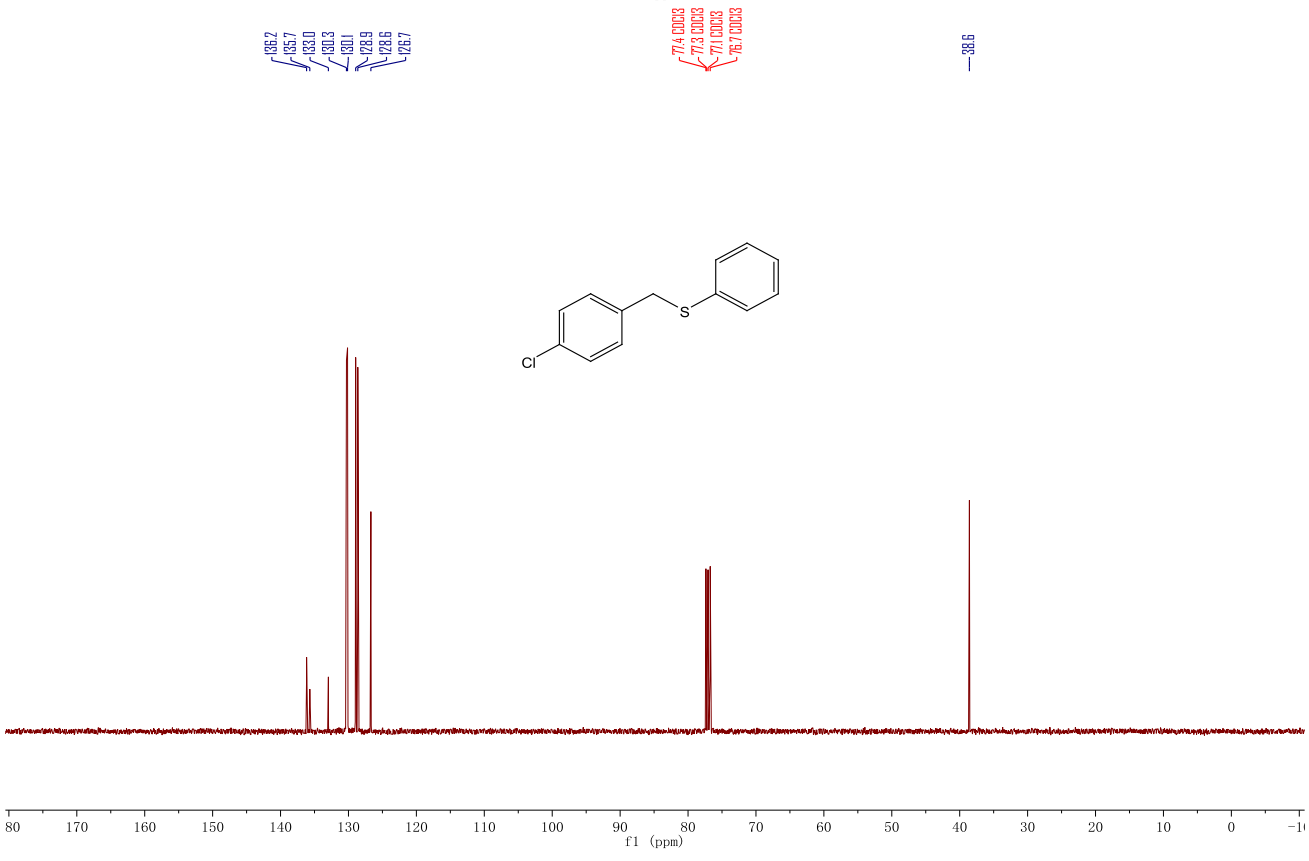




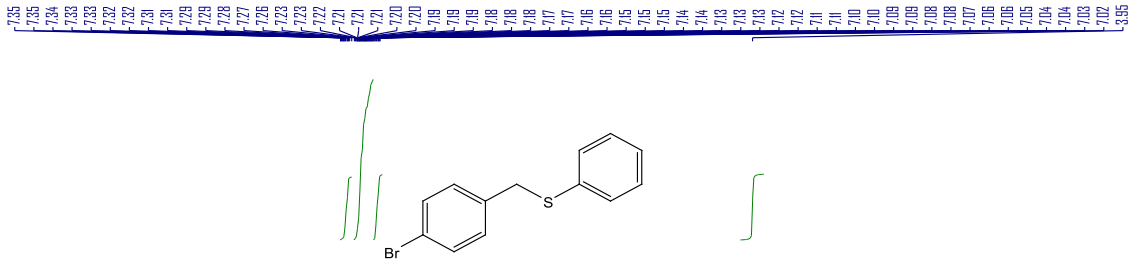


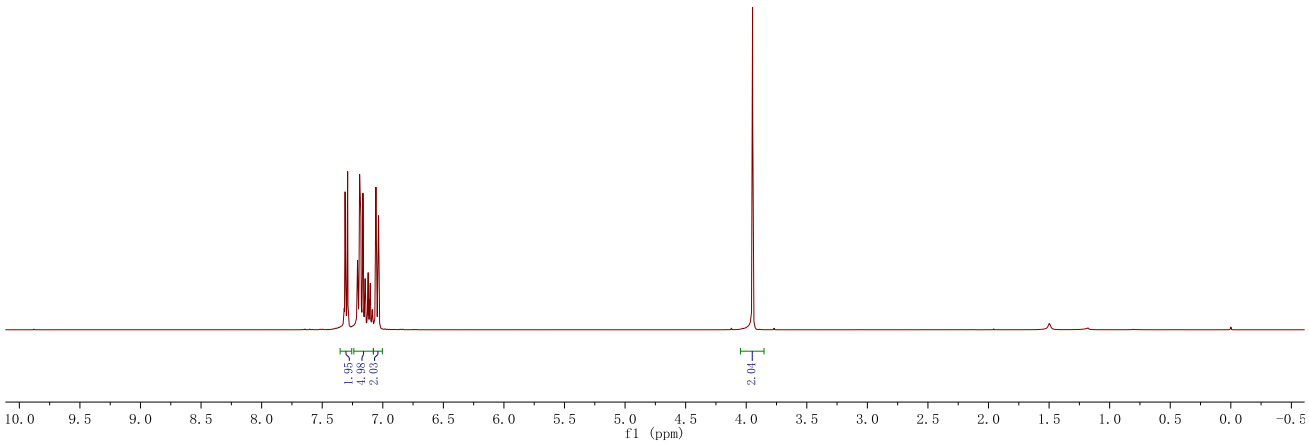
Product 3da-S

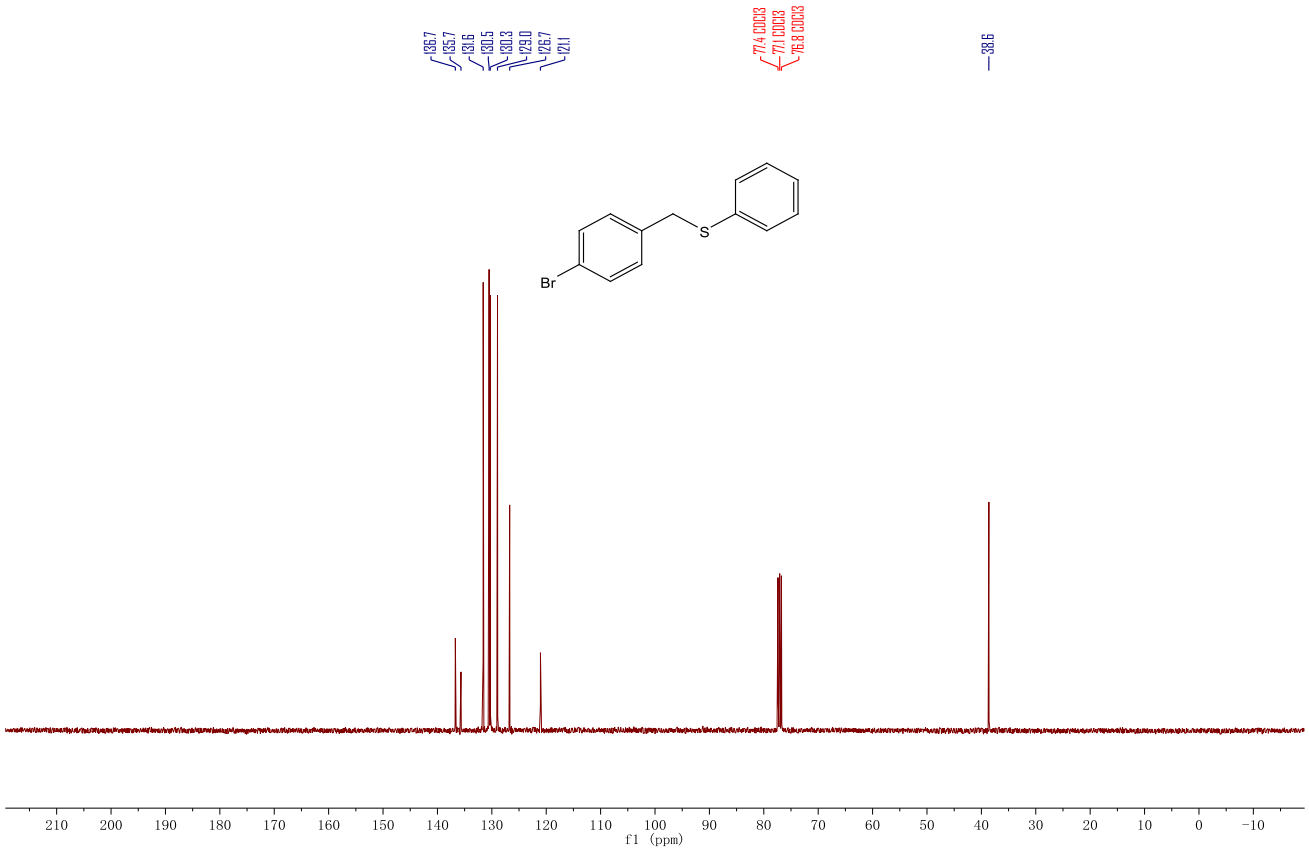




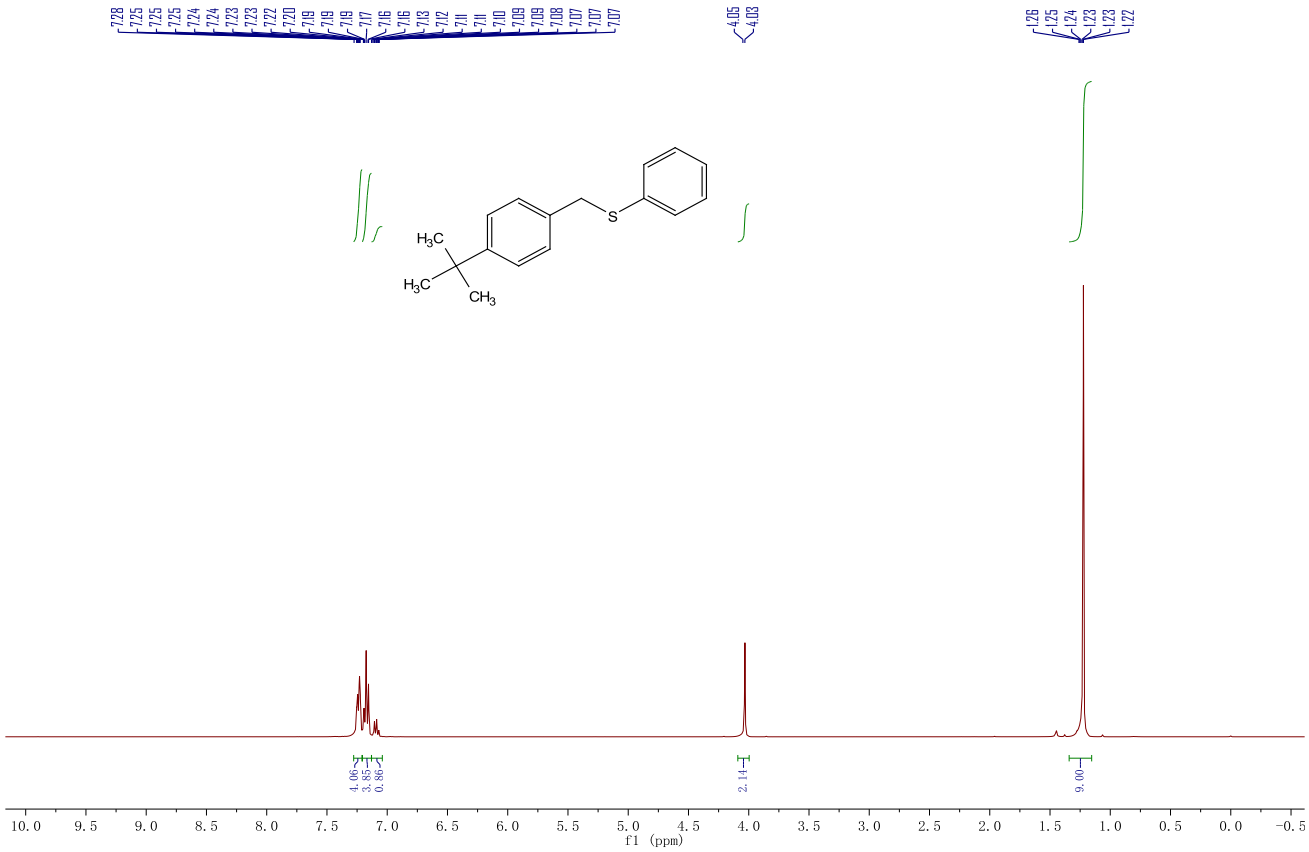
Product 3ea-S

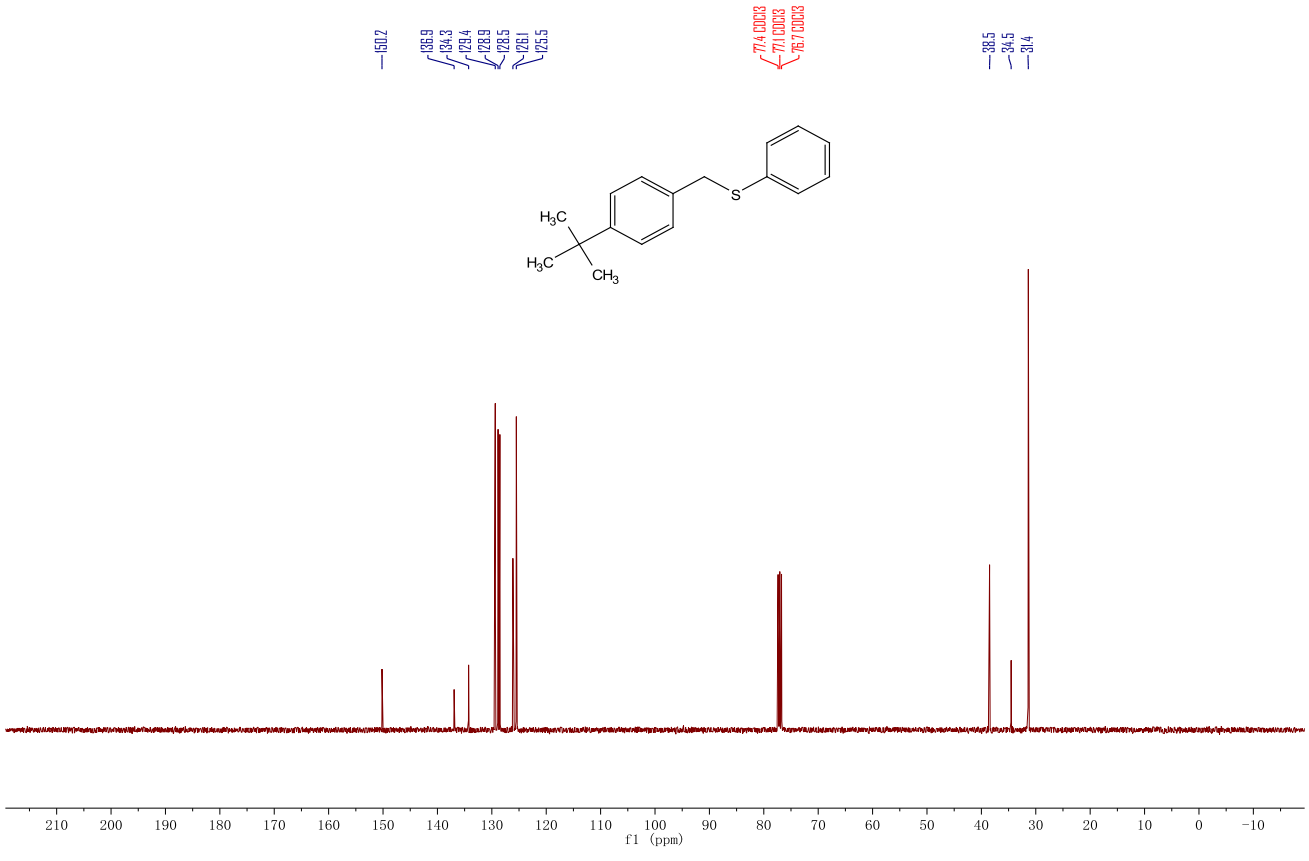




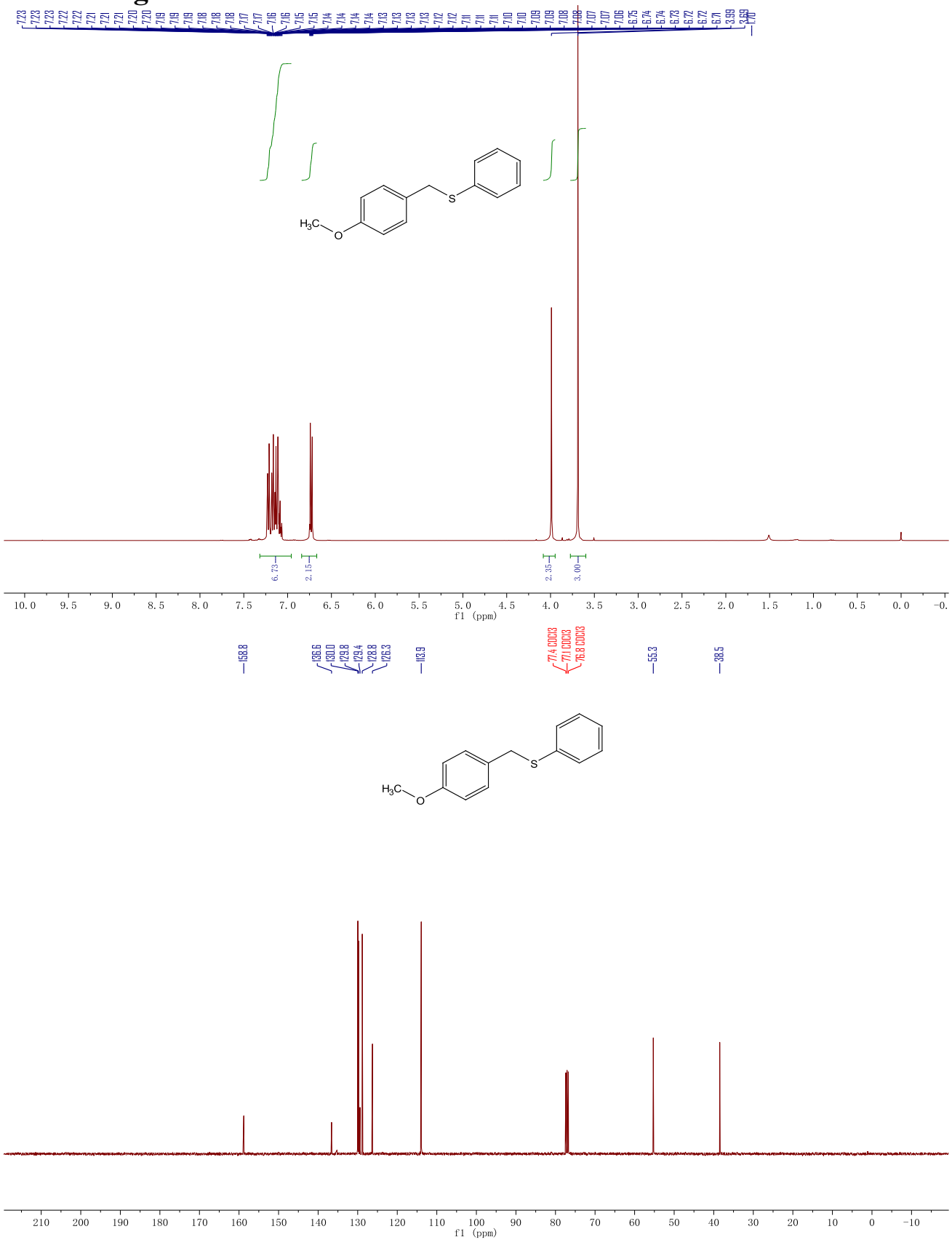


Product 3fa-S

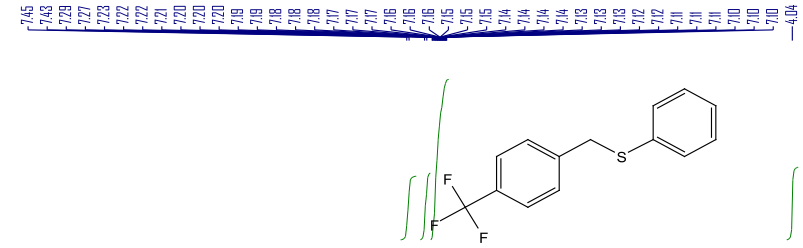


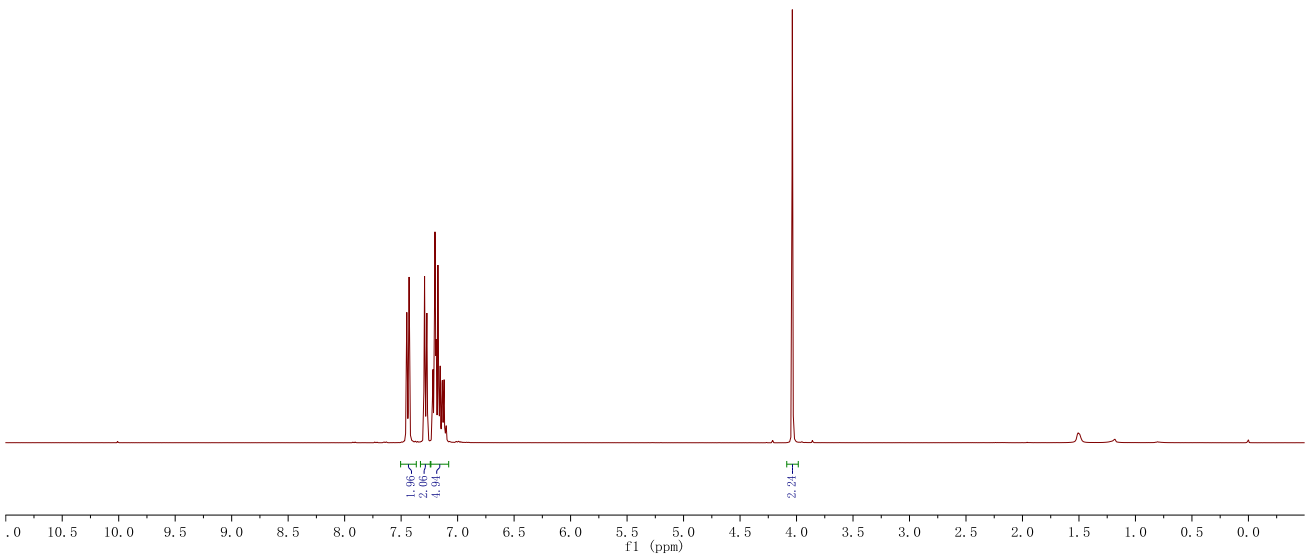


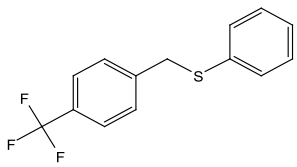
Product 3ga-S



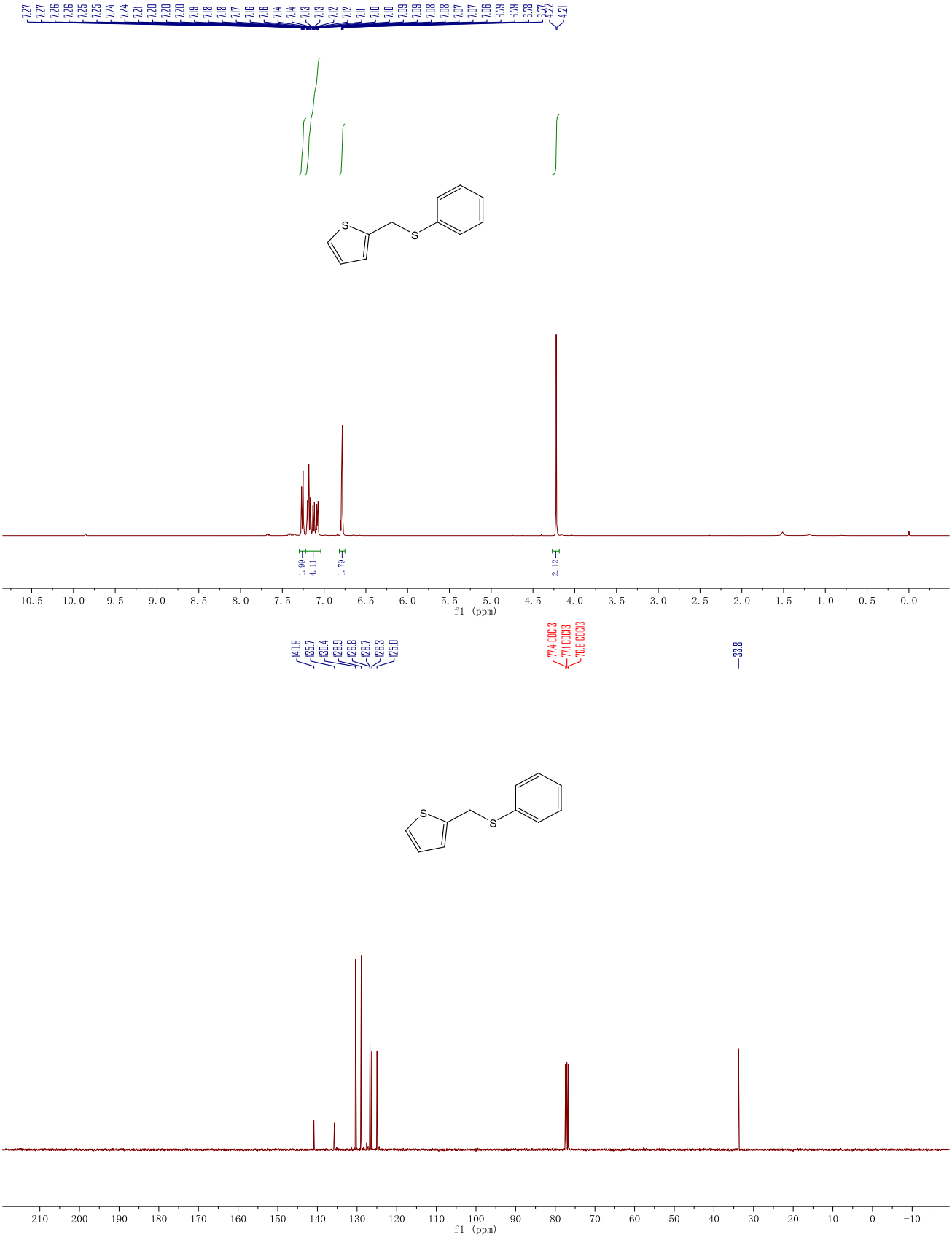
Product 3ha-S



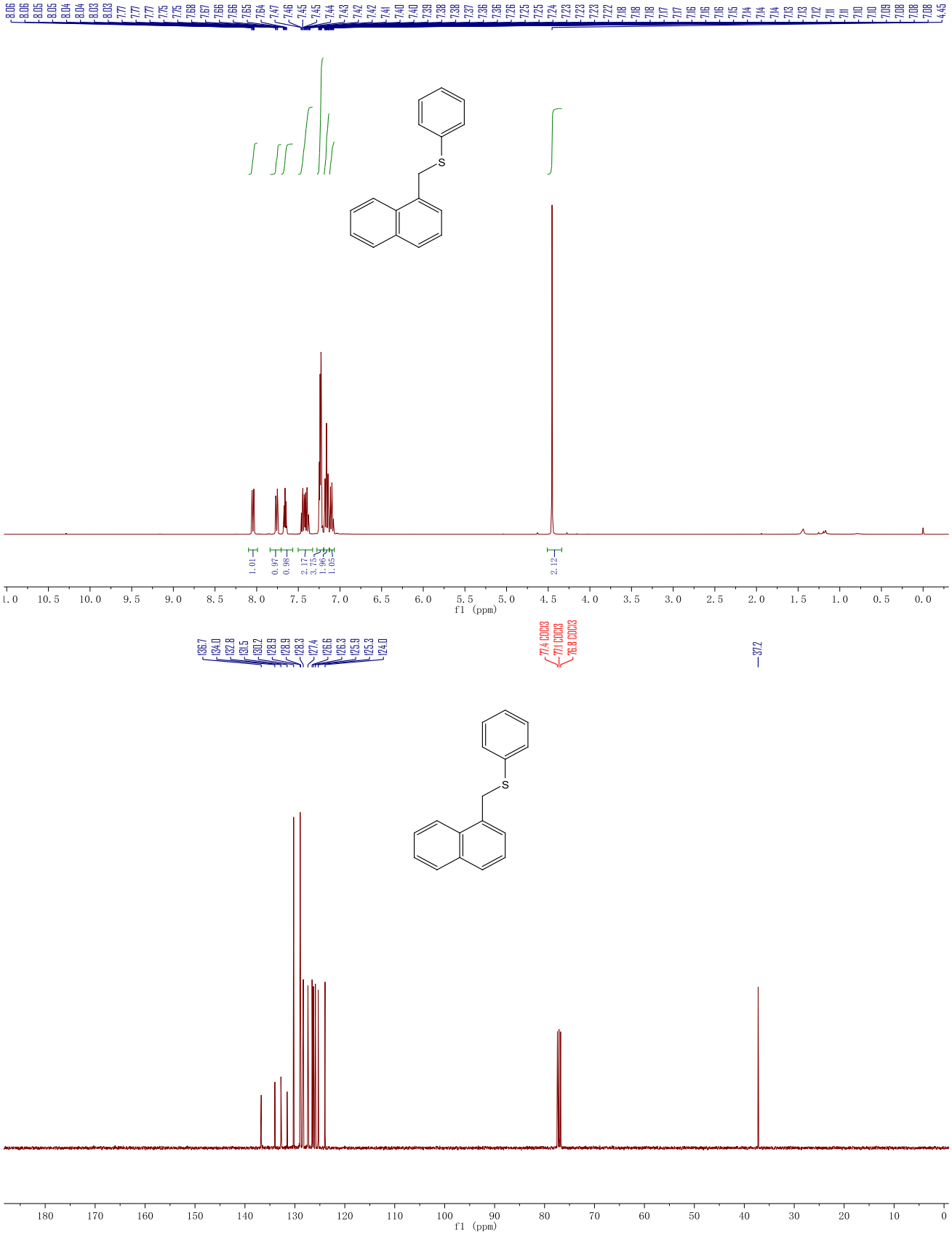




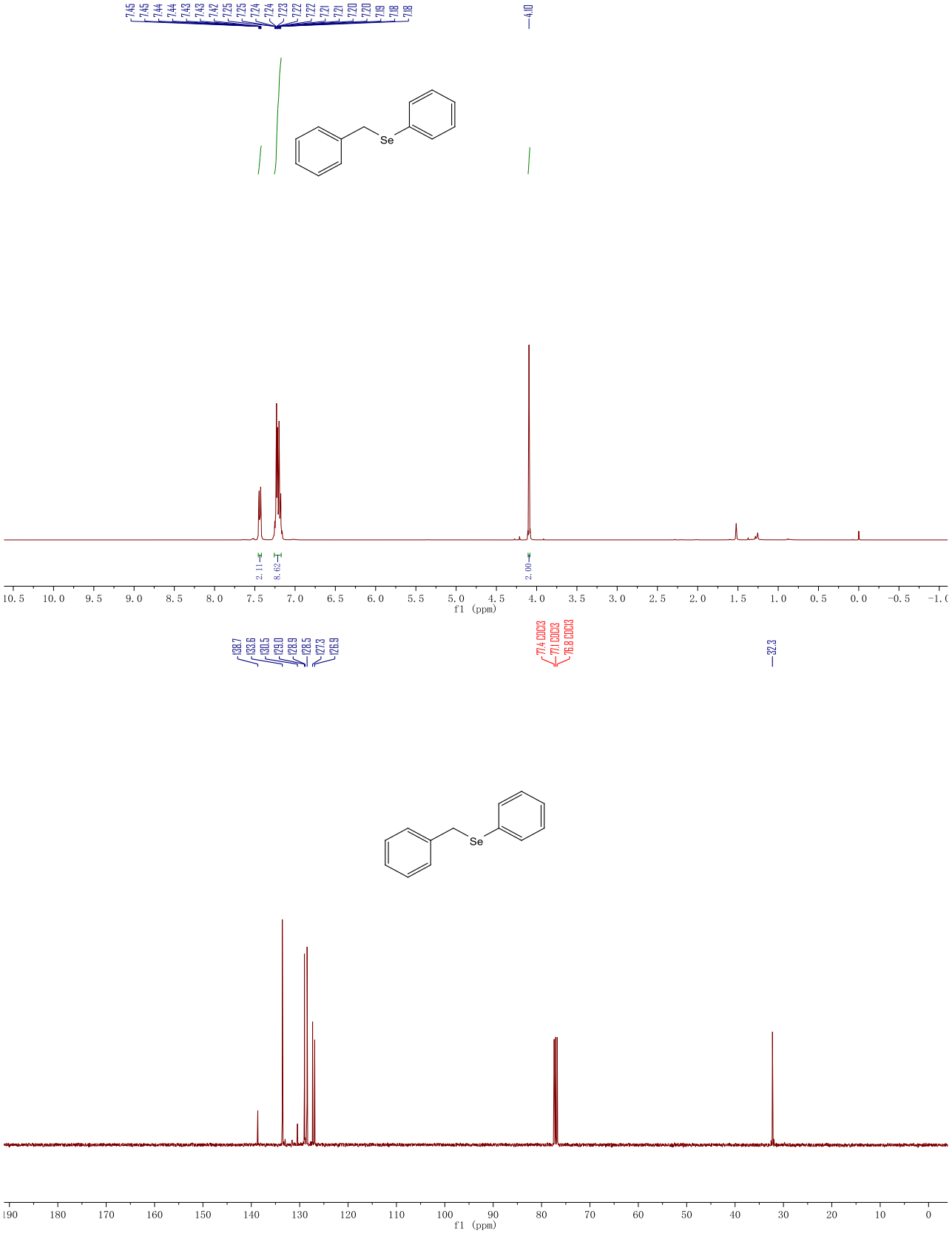
Product 3ia-S



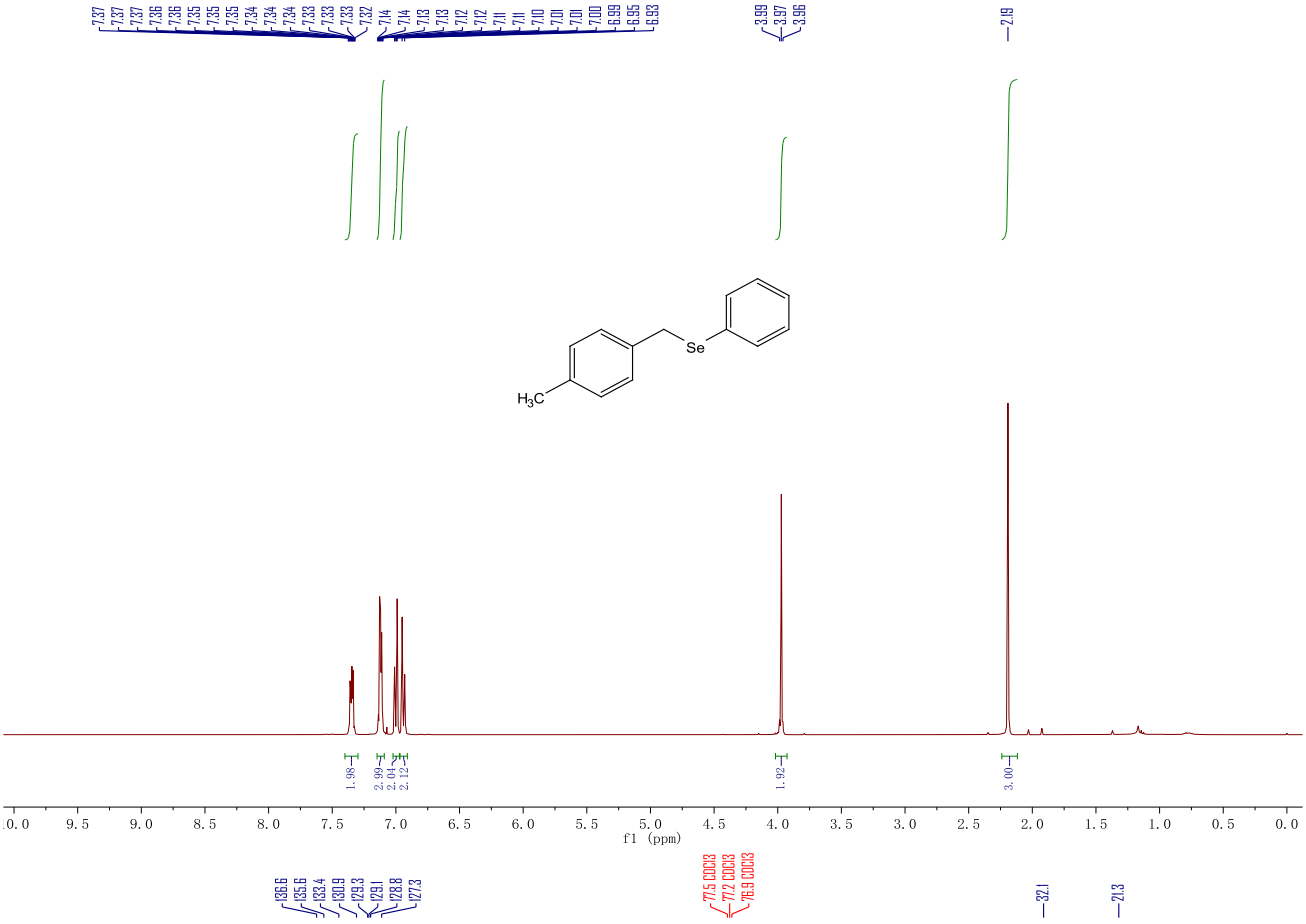
Product 3ja-S

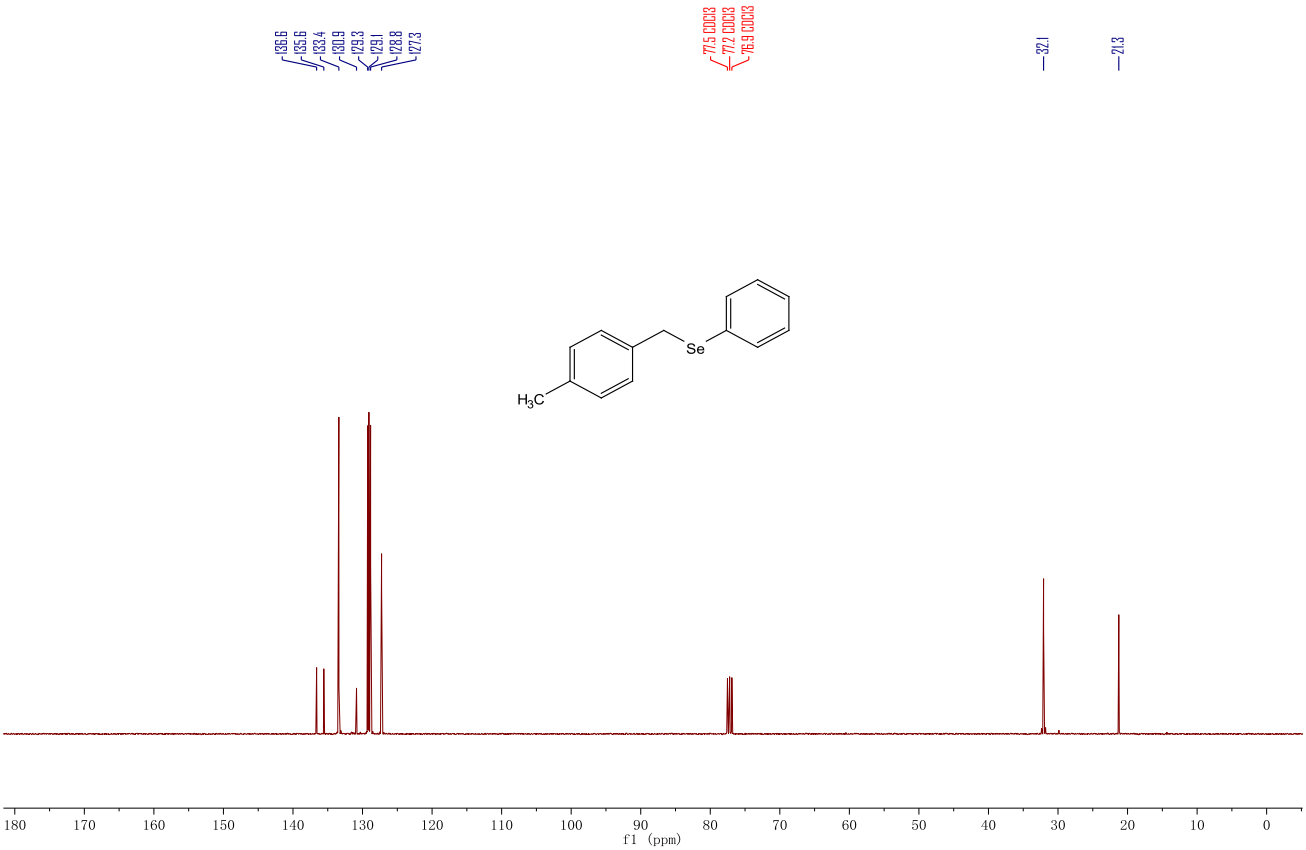


Product 3aa-Se

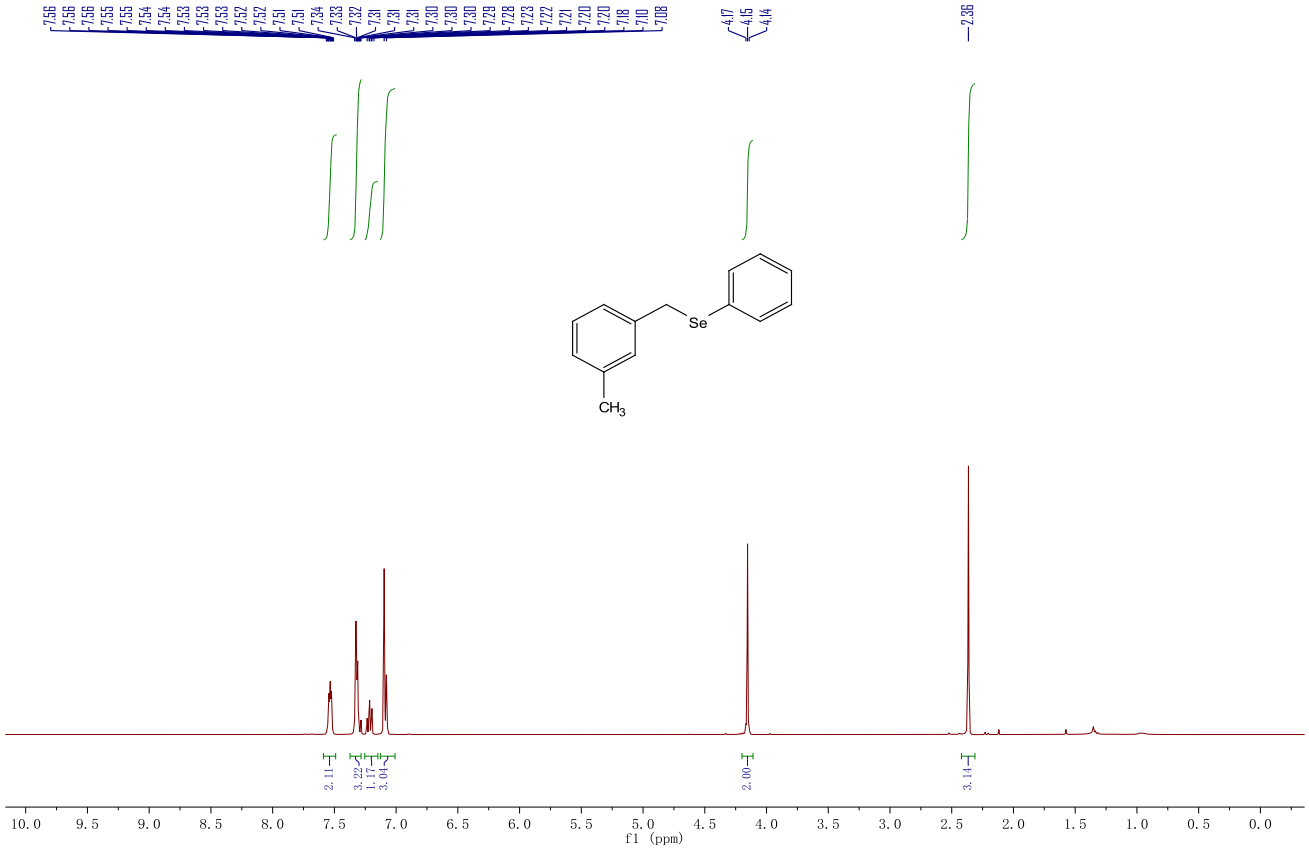


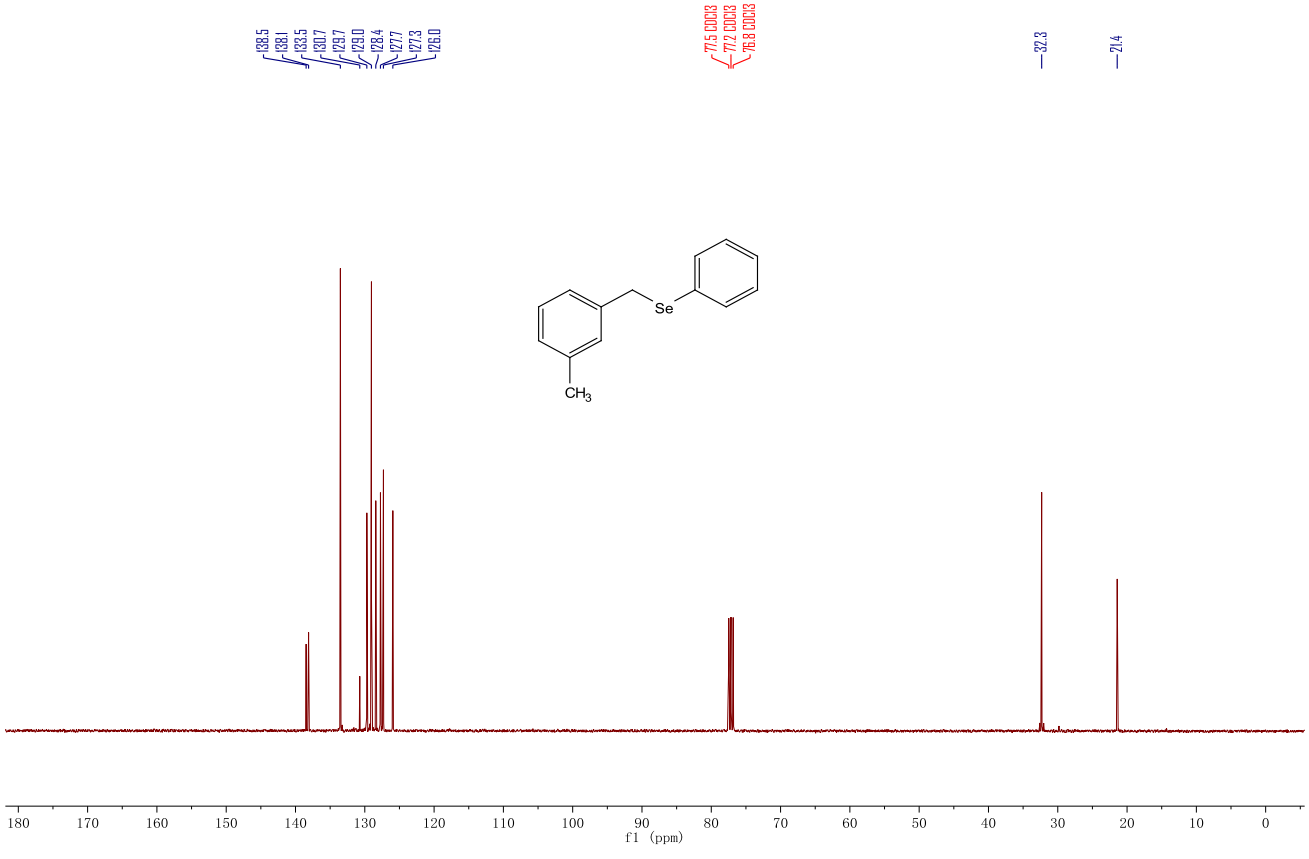
Product 3ba-Se



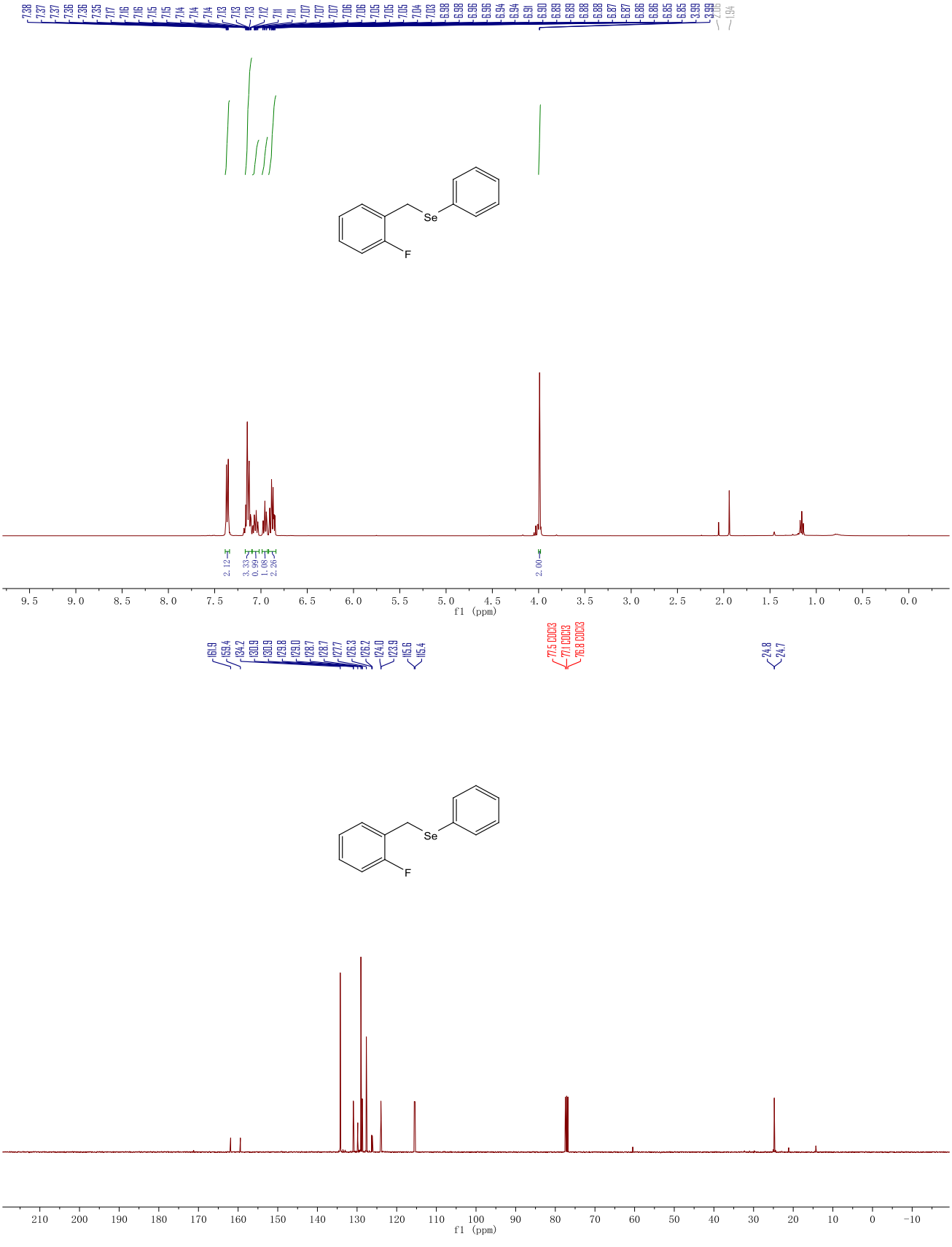


Product 3ca-Se

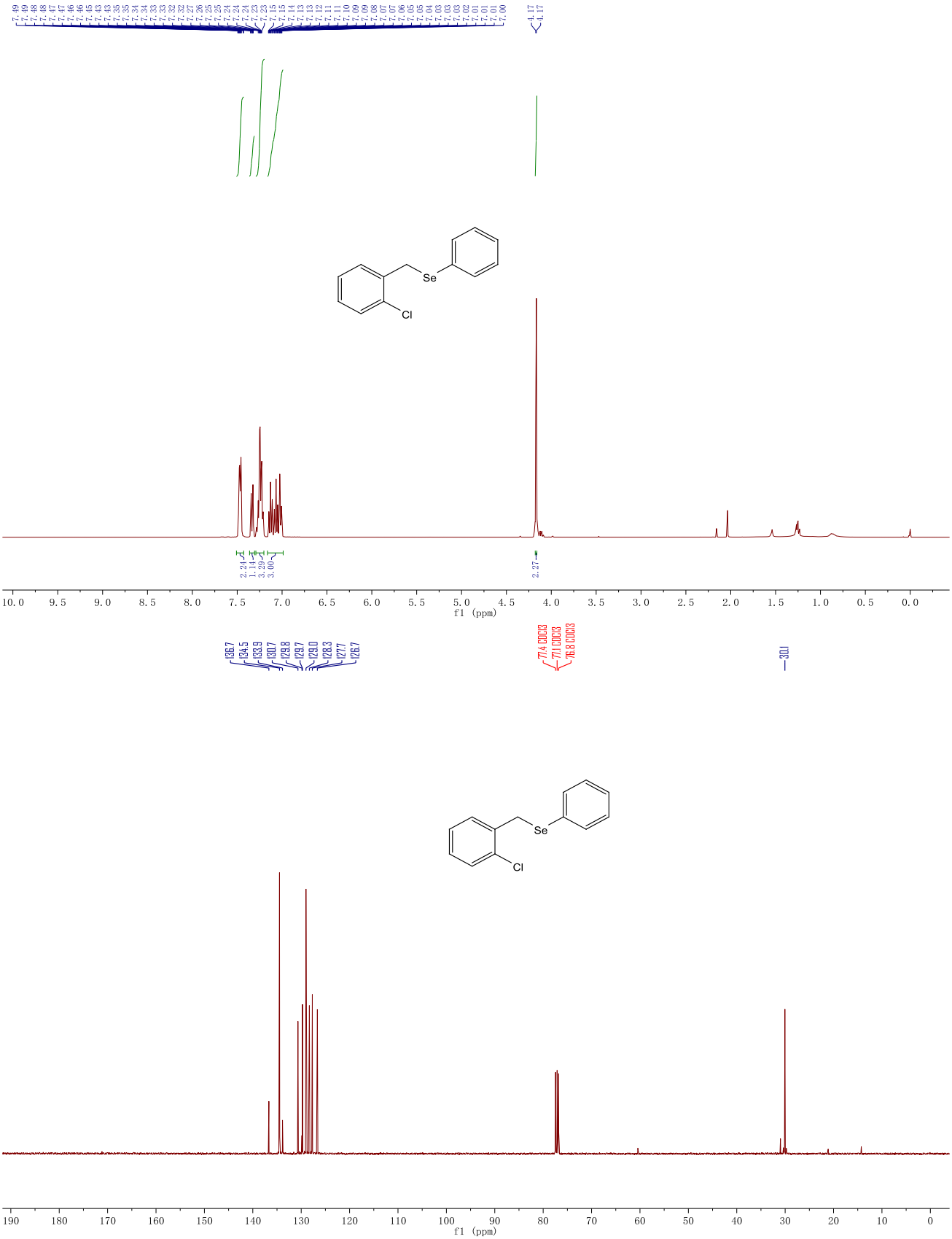




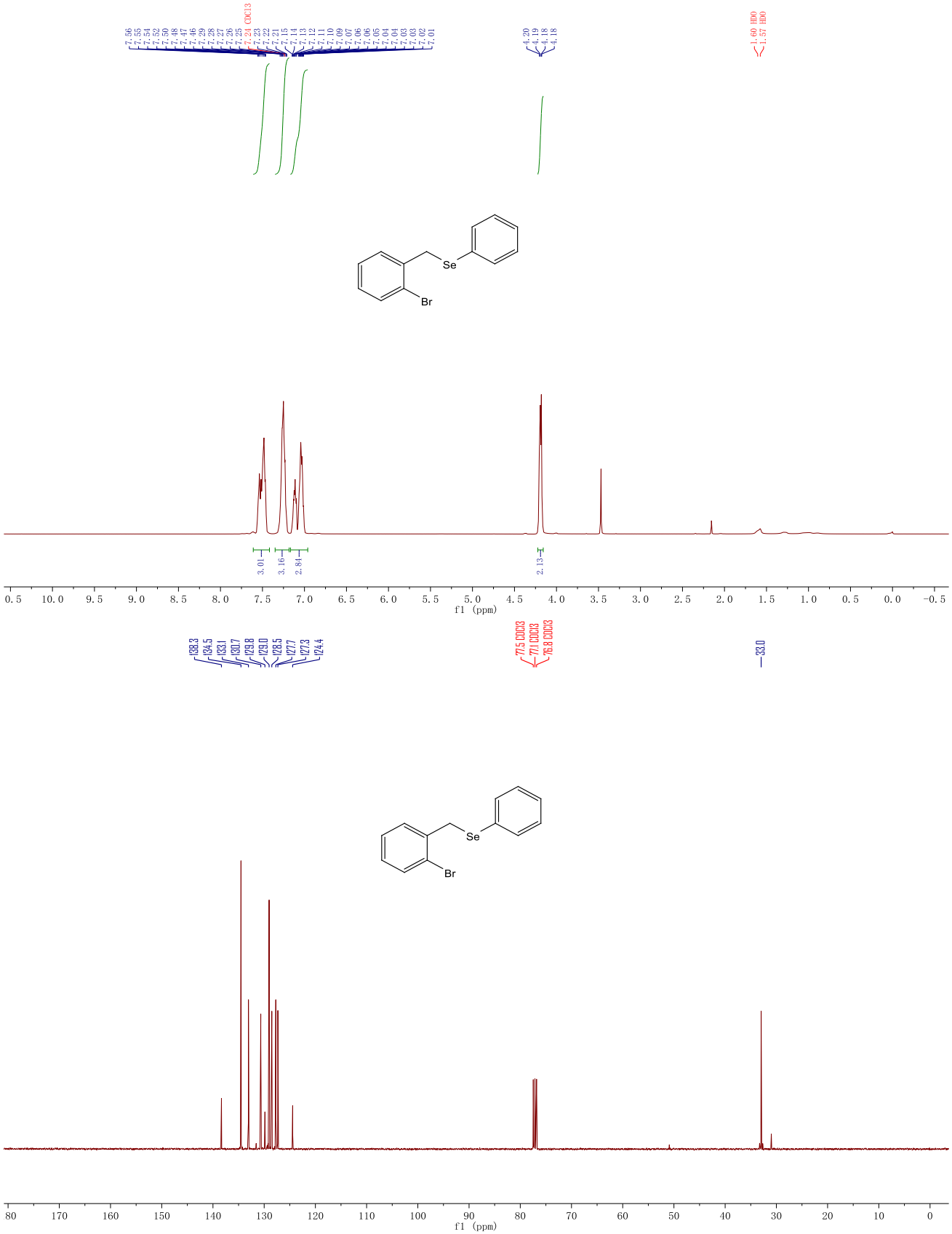
Product 3da-Se



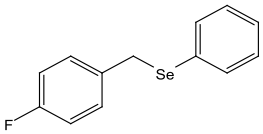
Product 3ea-Se

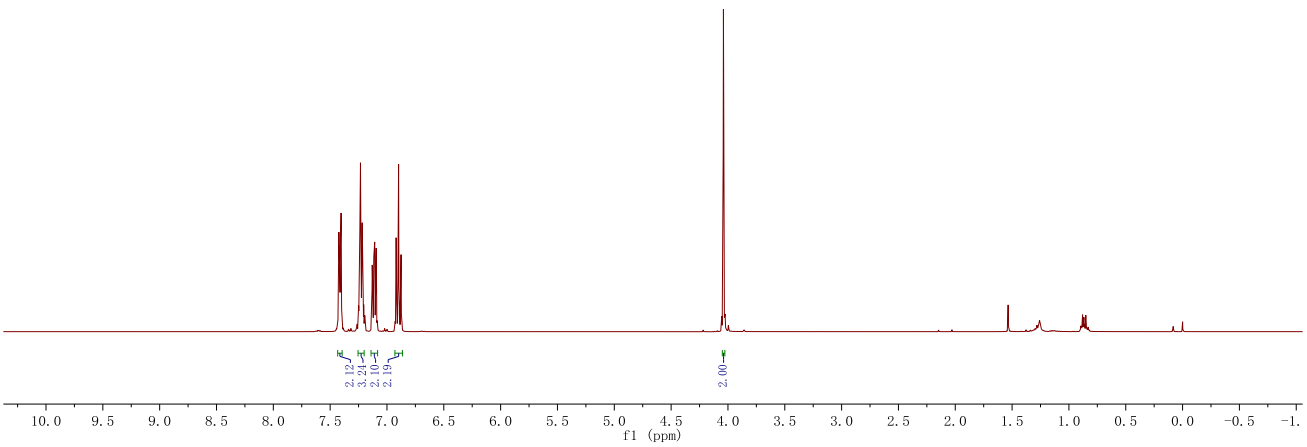


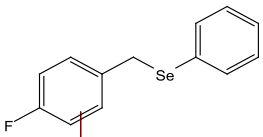
Product 3fa-Se

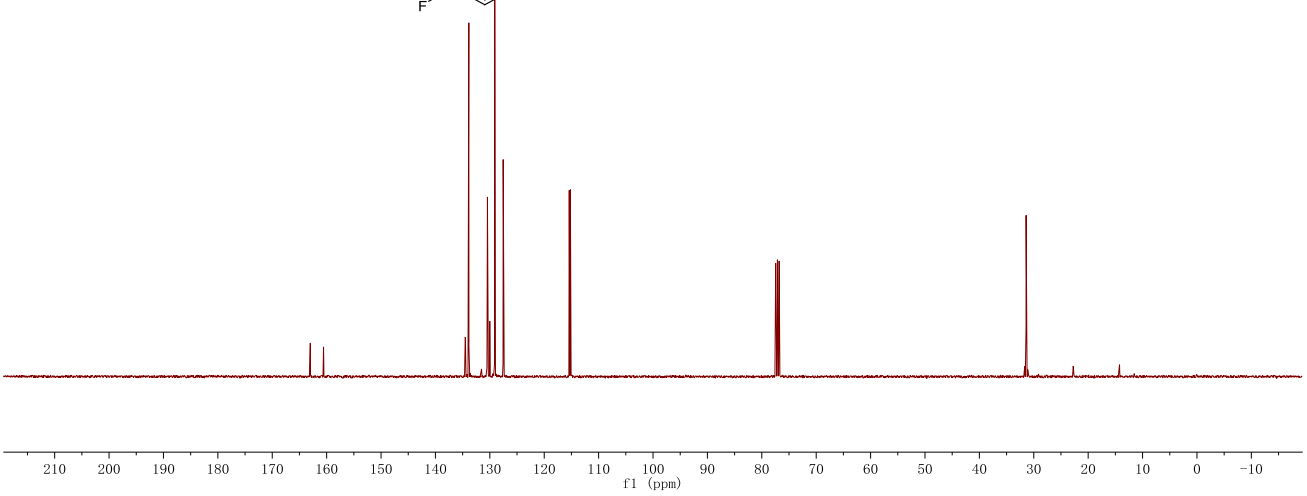


Product 3ga-Se

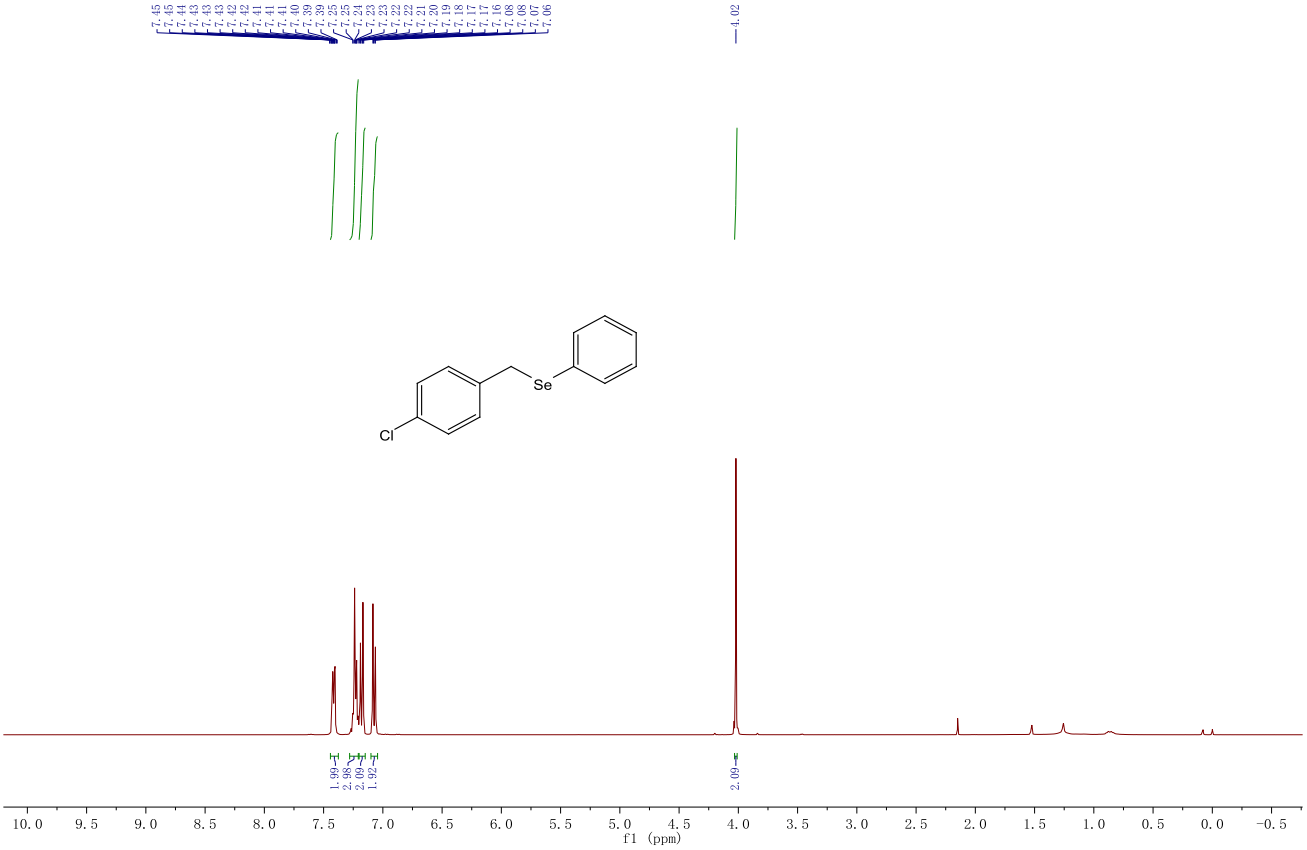


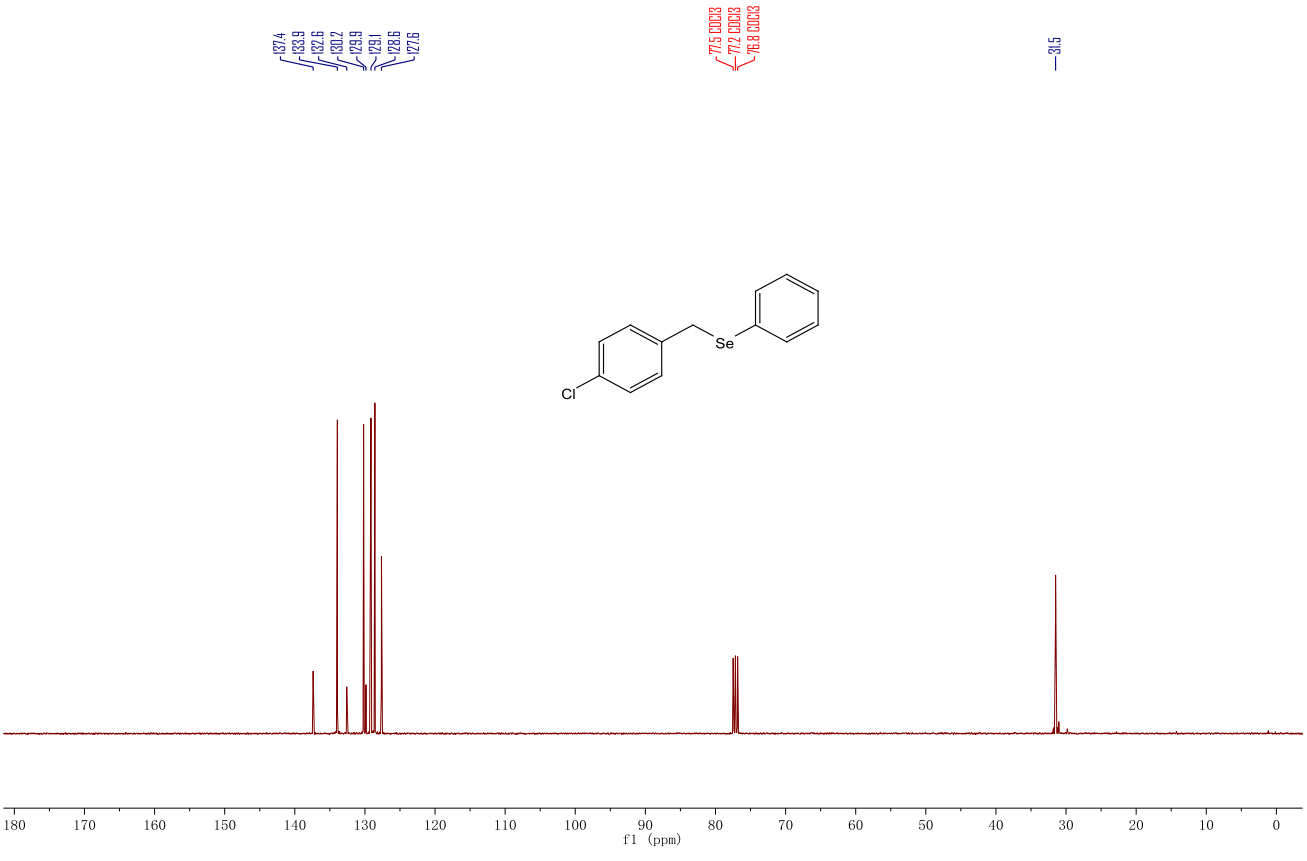




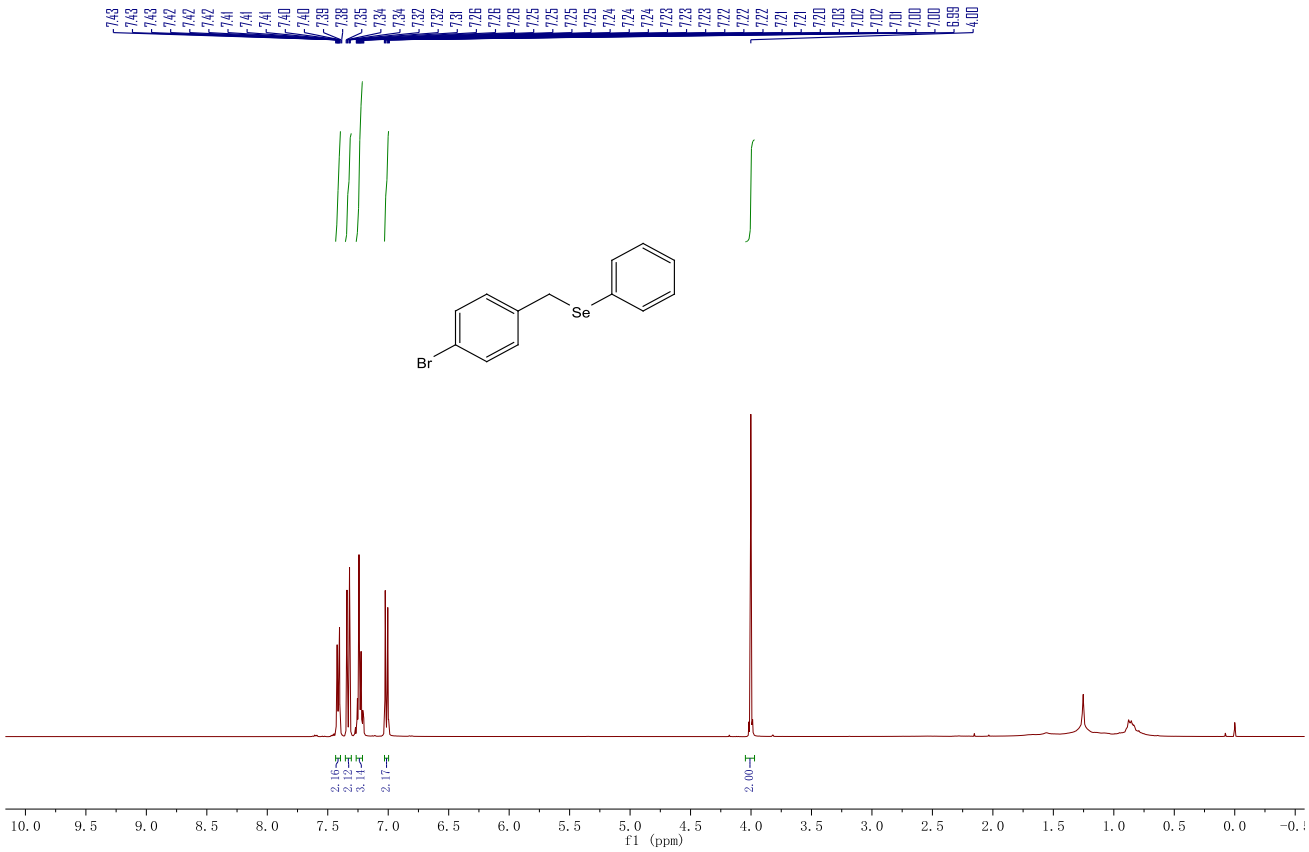


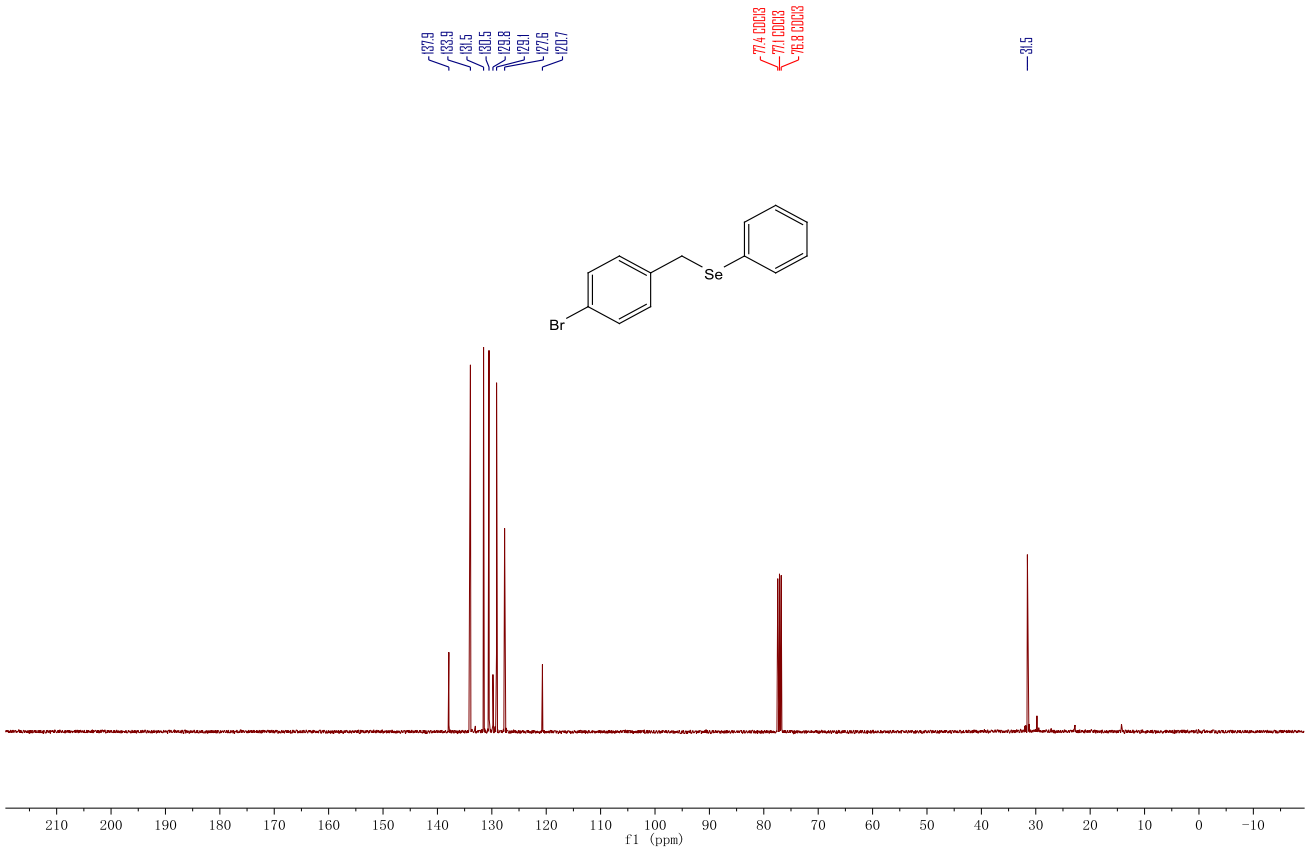
Product 3ha-Se



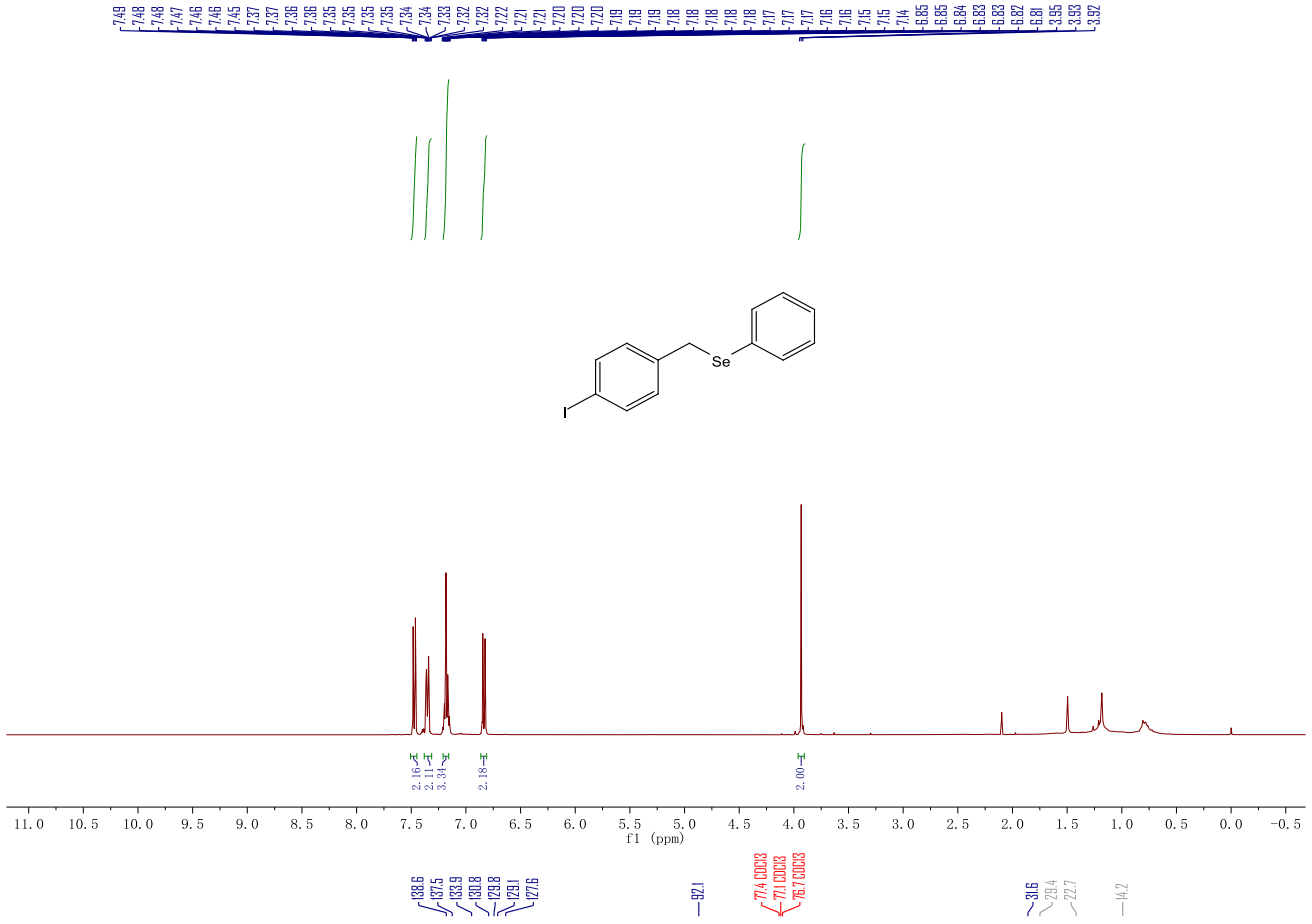


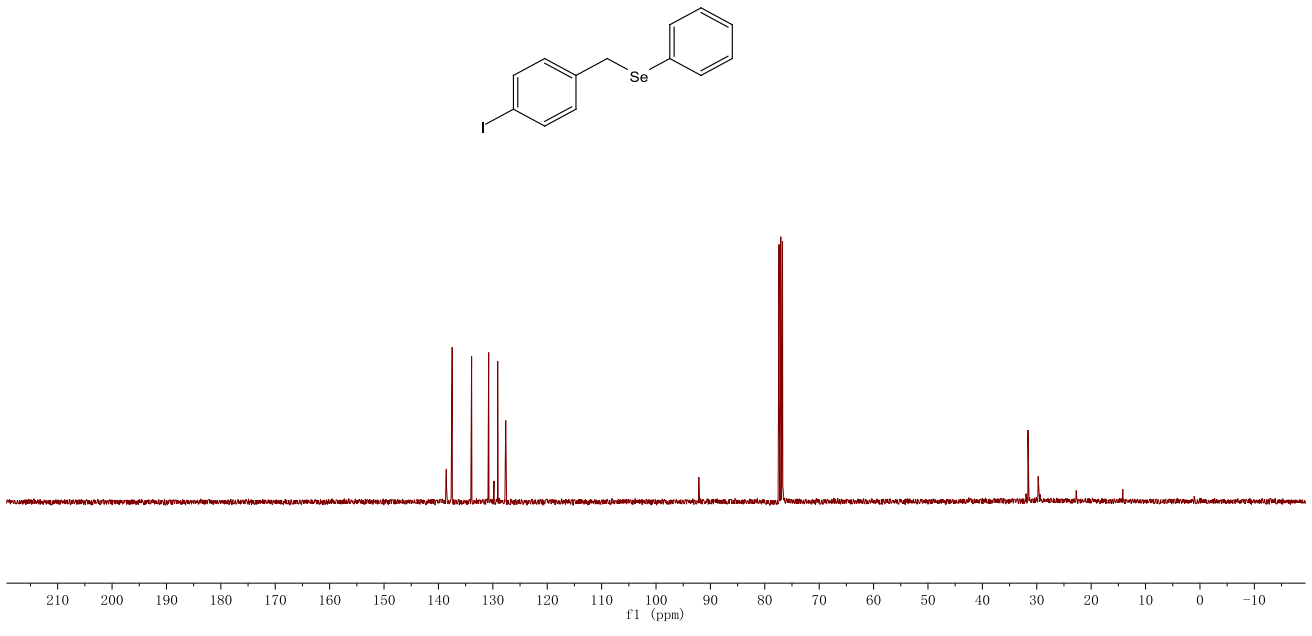
Product 3ia-Se



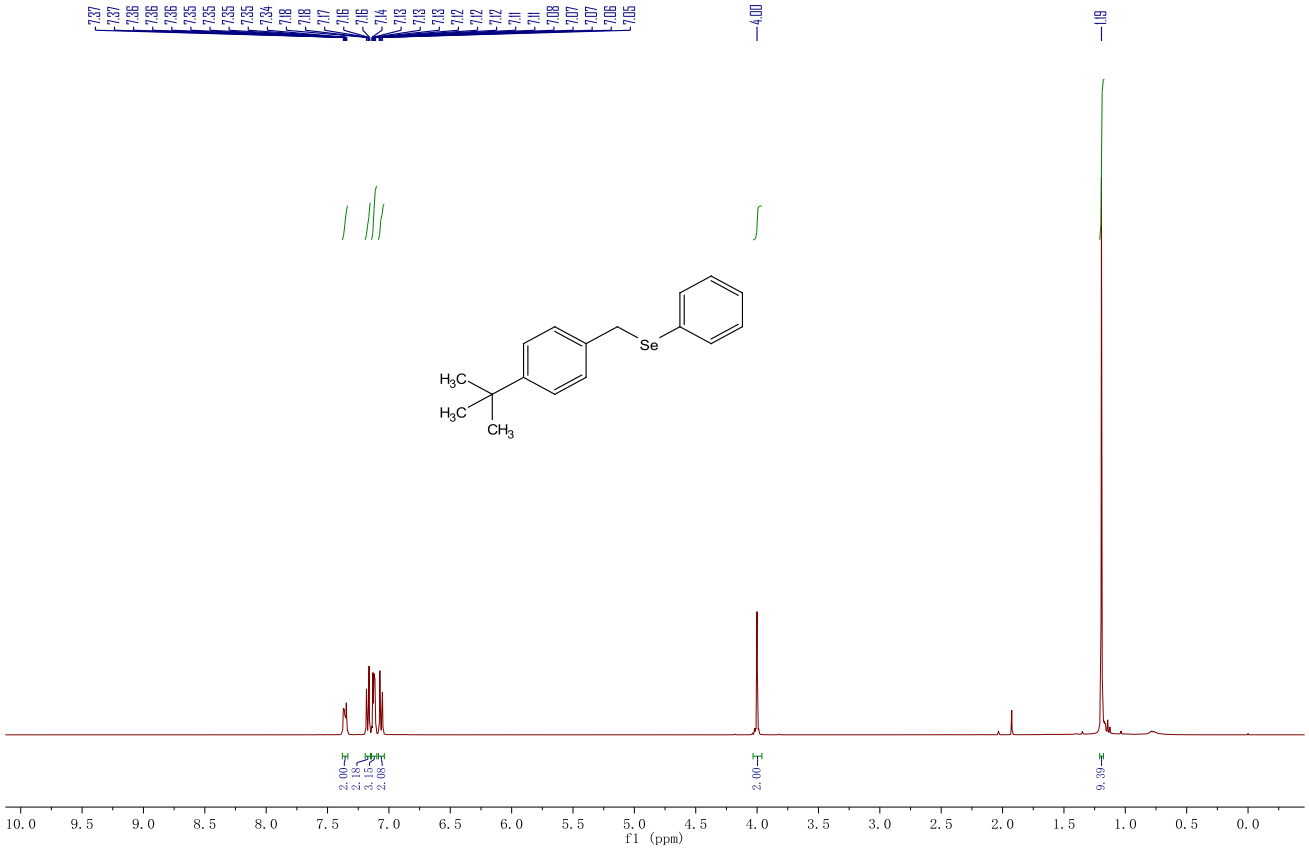


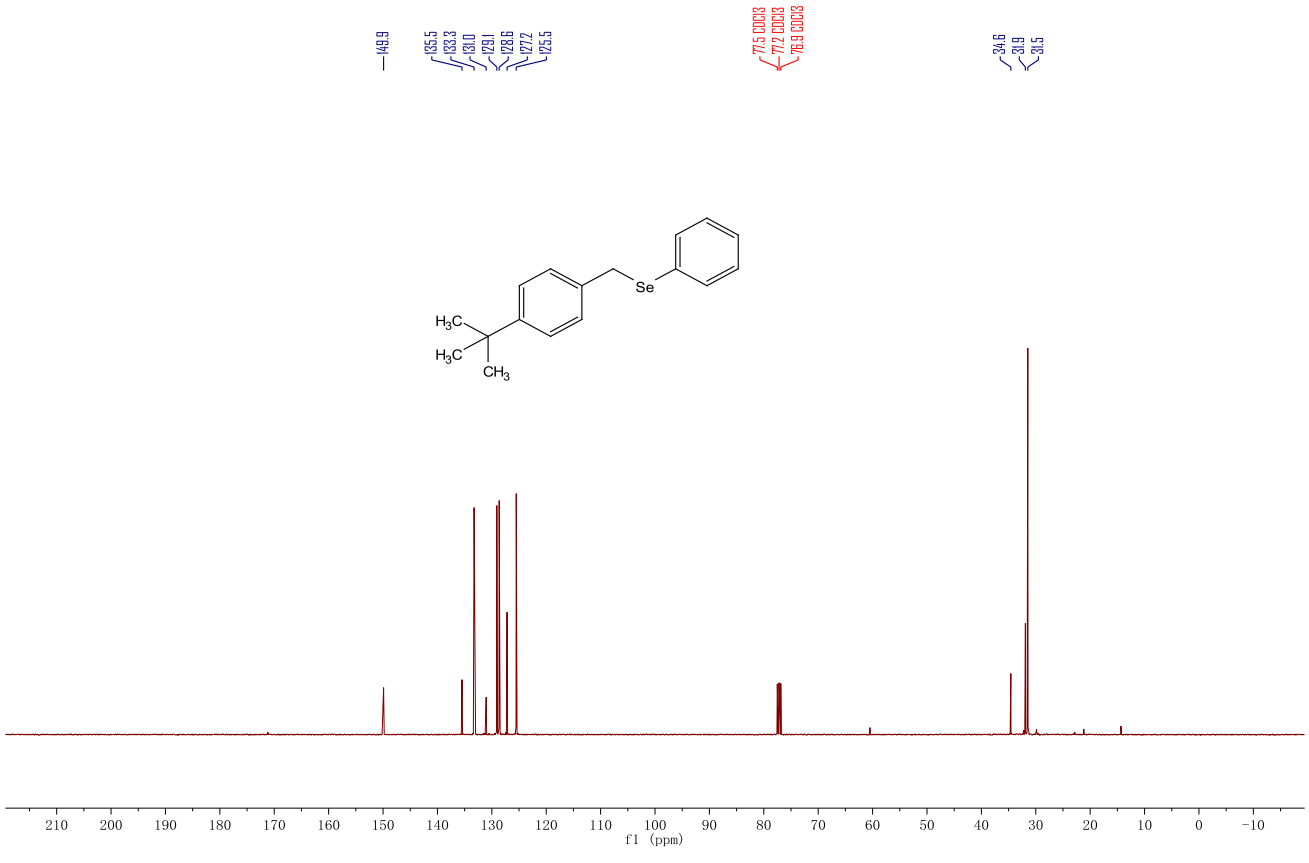
Product 3ja-Se



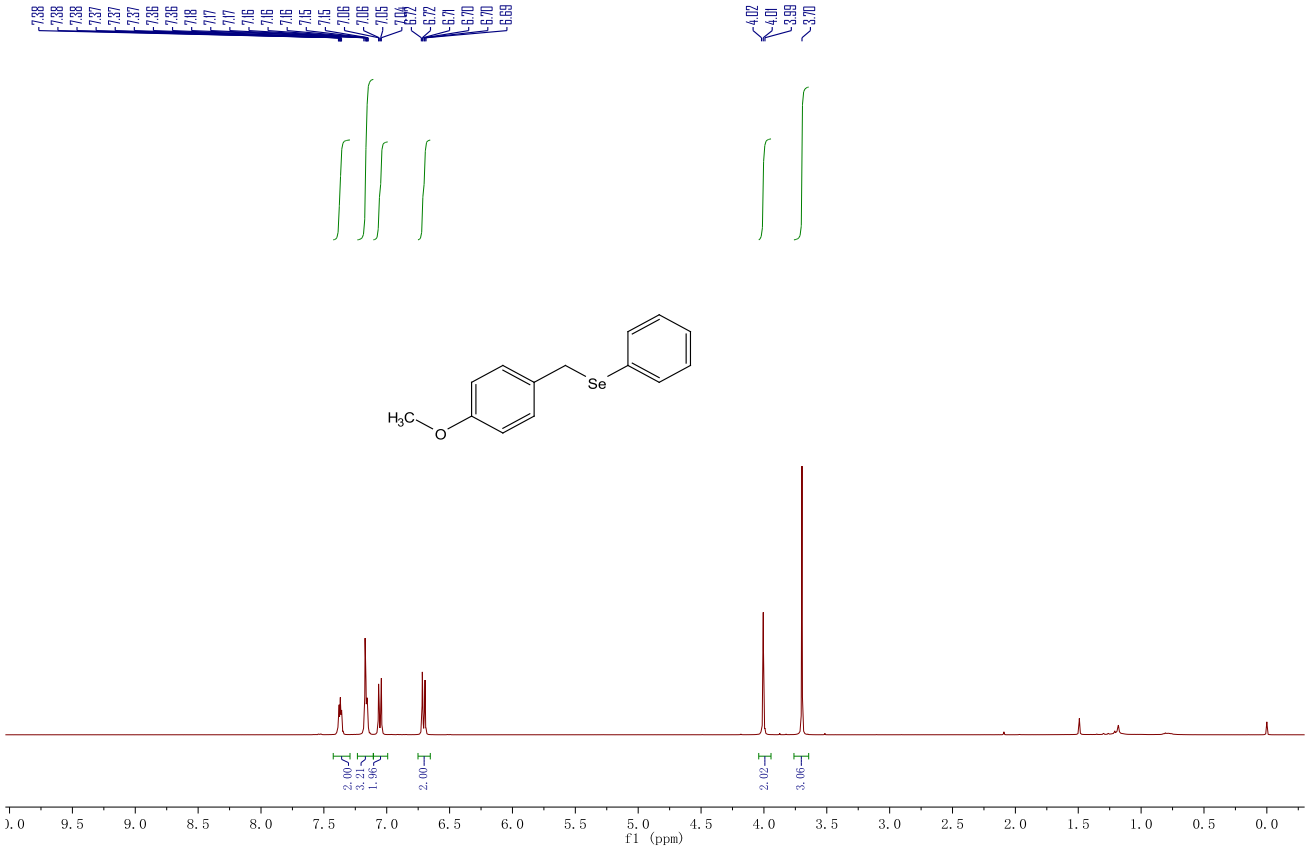


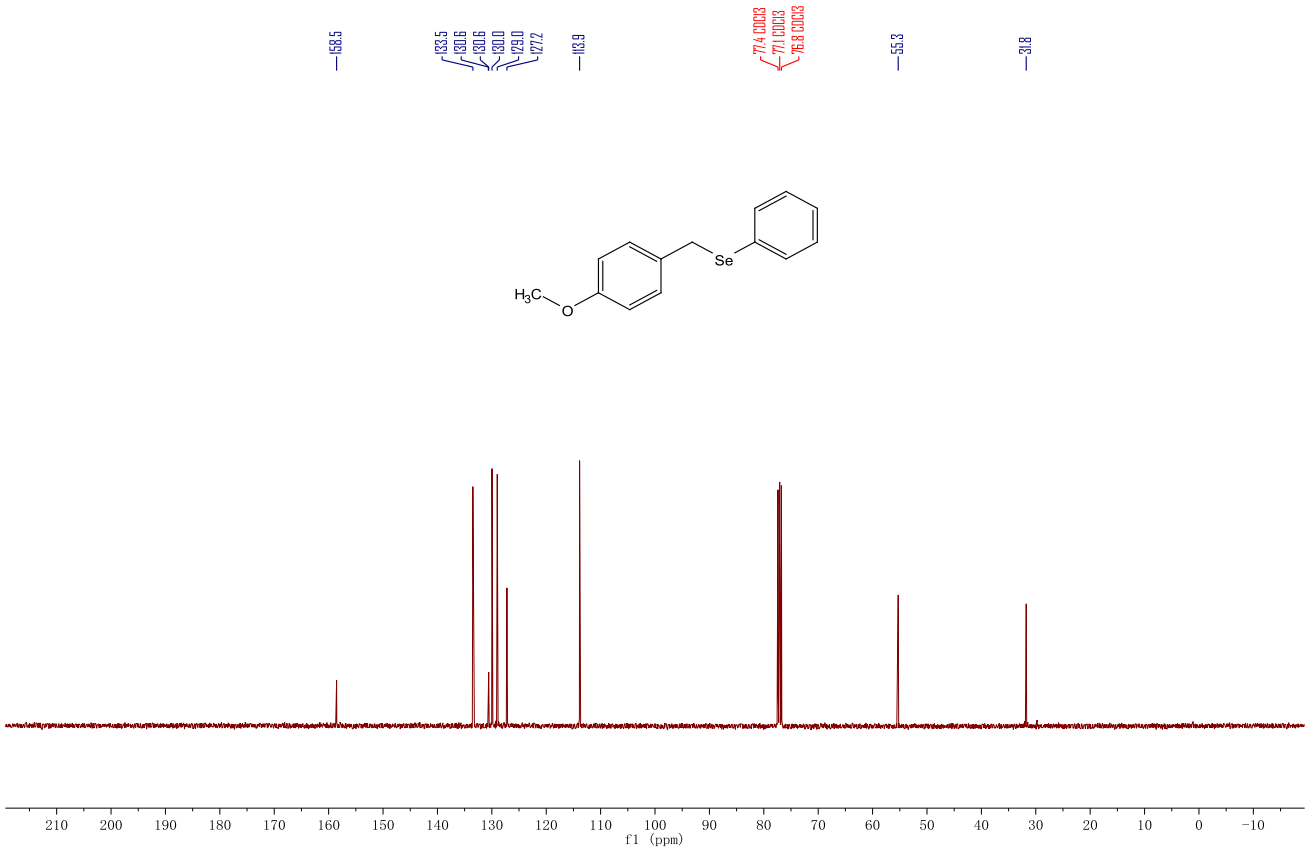
Product 3ka-Se



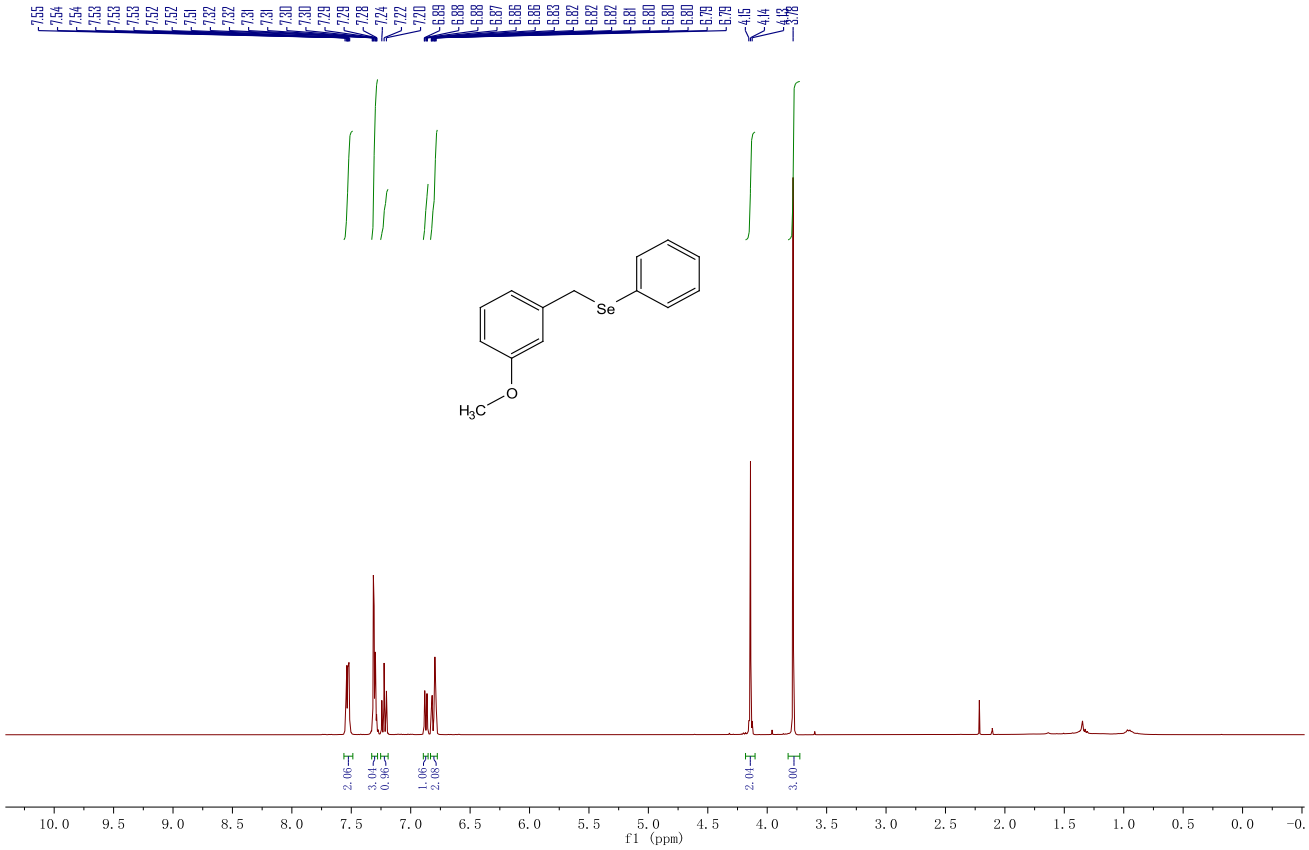


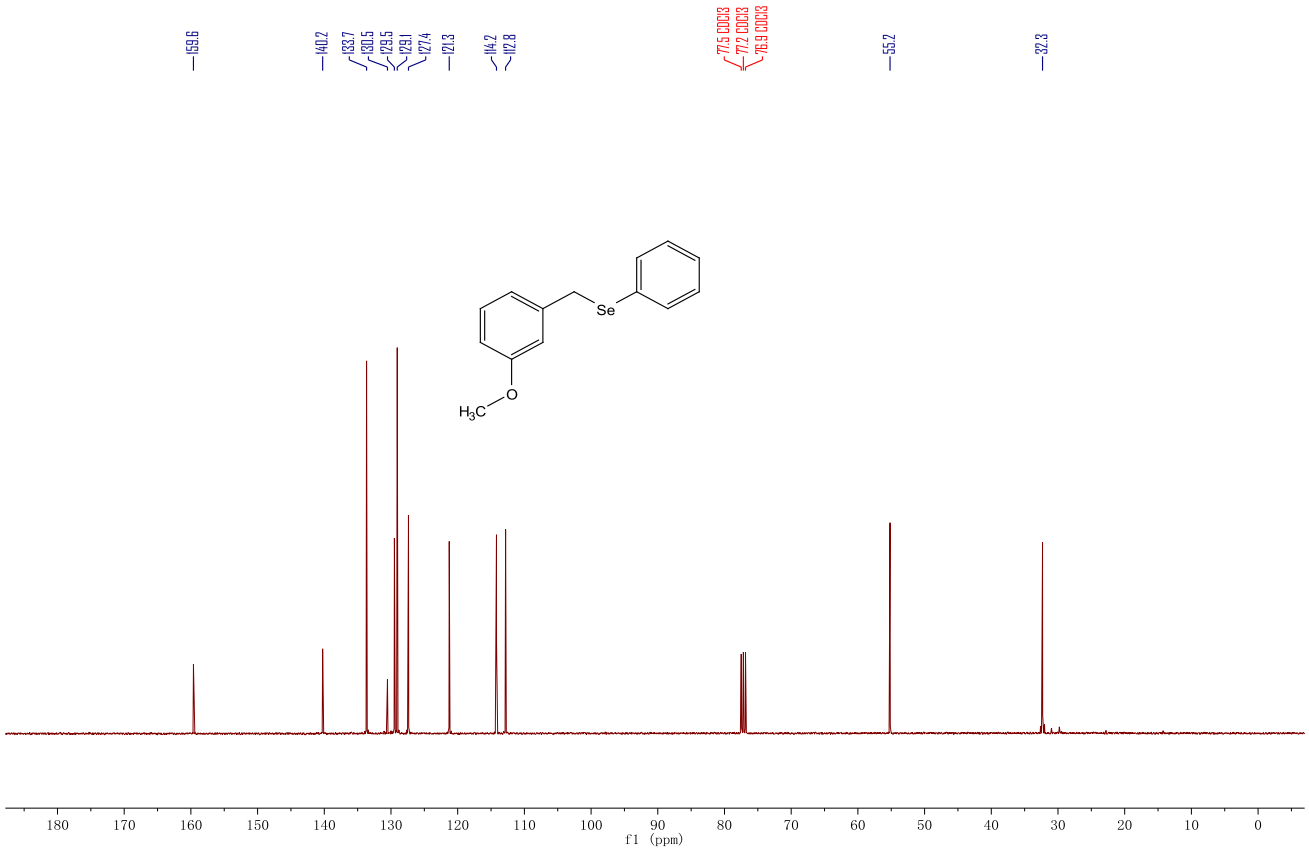
Product 3la-Se



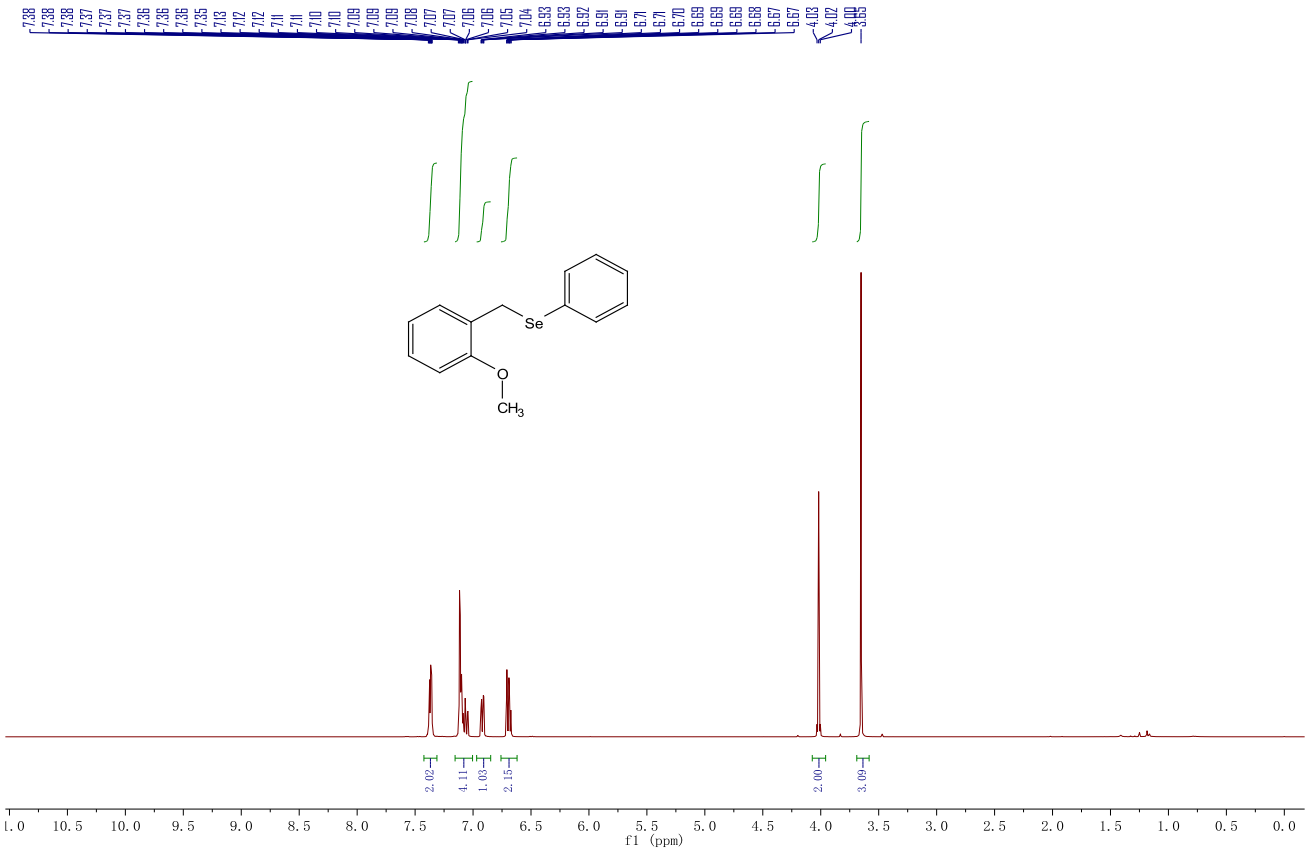


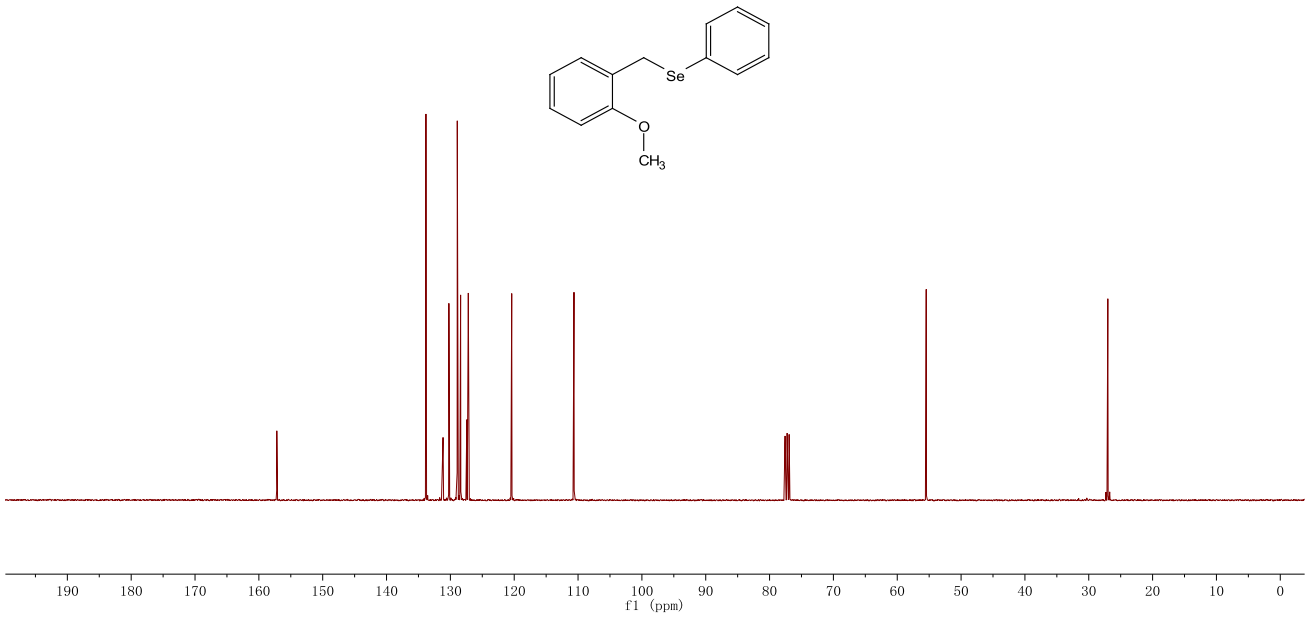
Product 3ma-Se



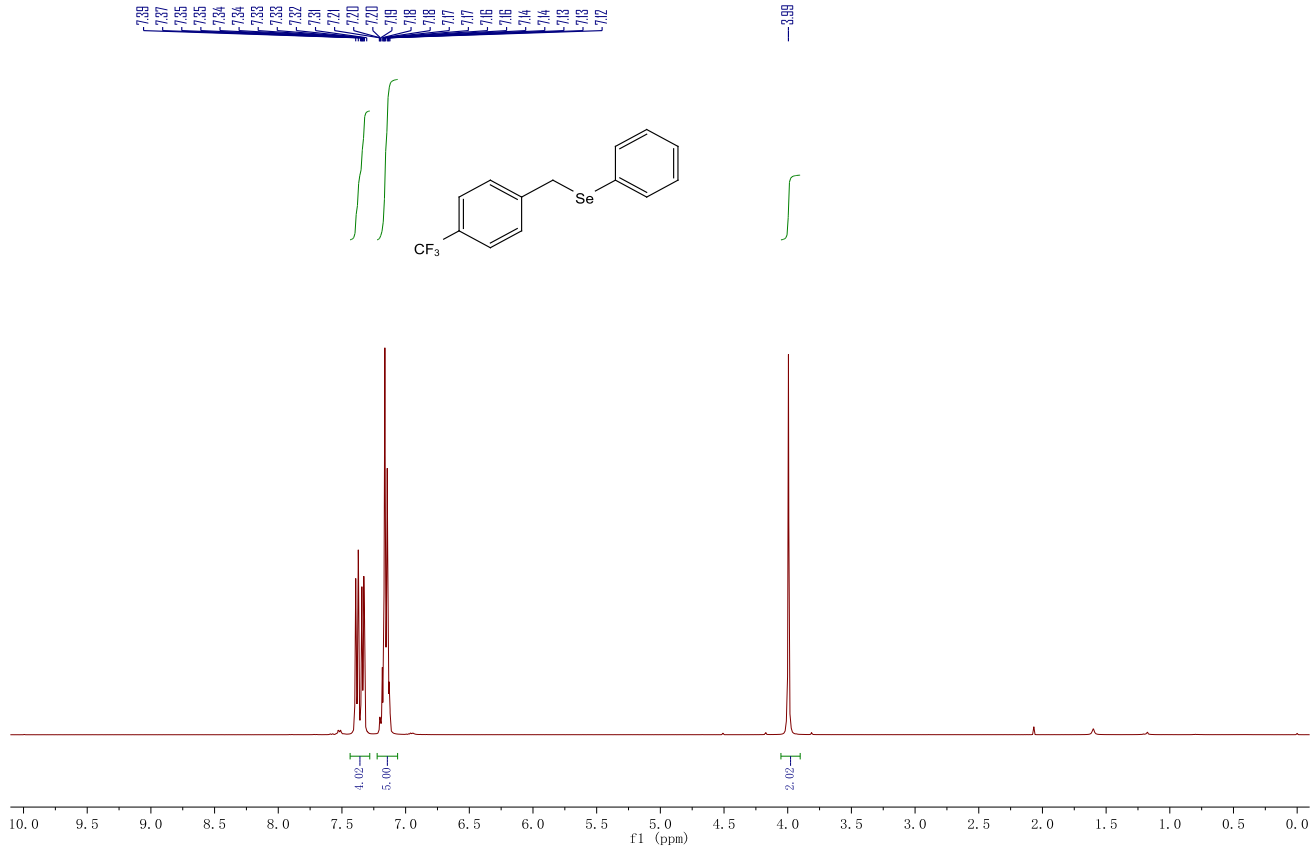


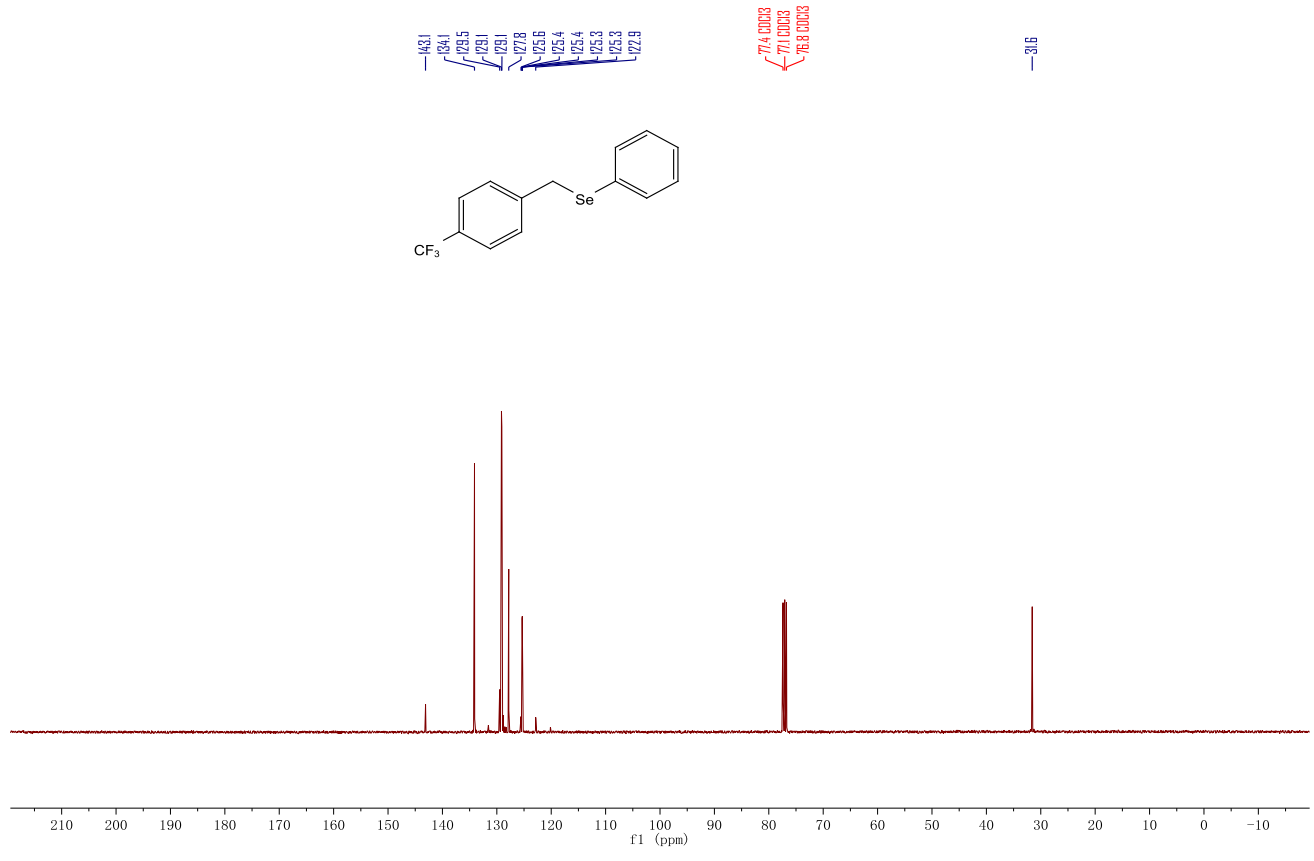
Product 3na-Se



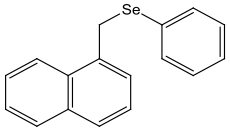


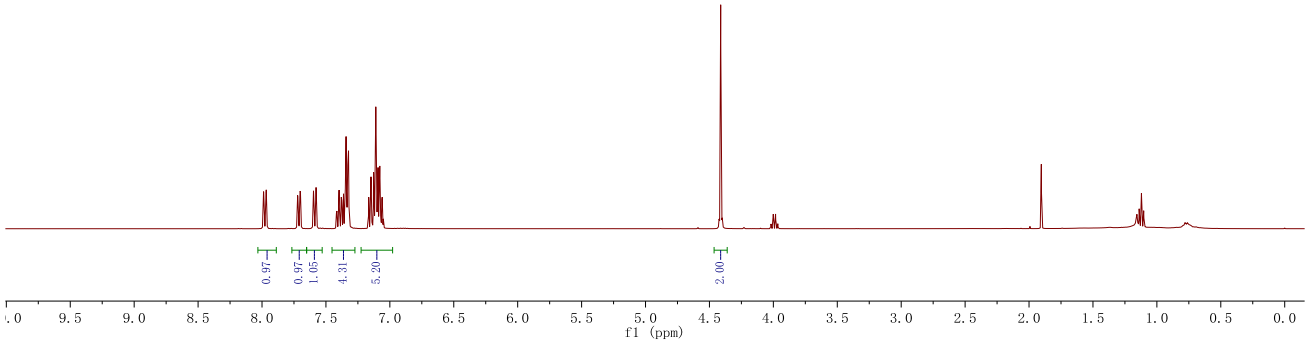
Product 3oa-Se

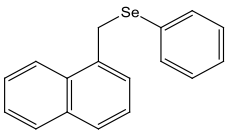




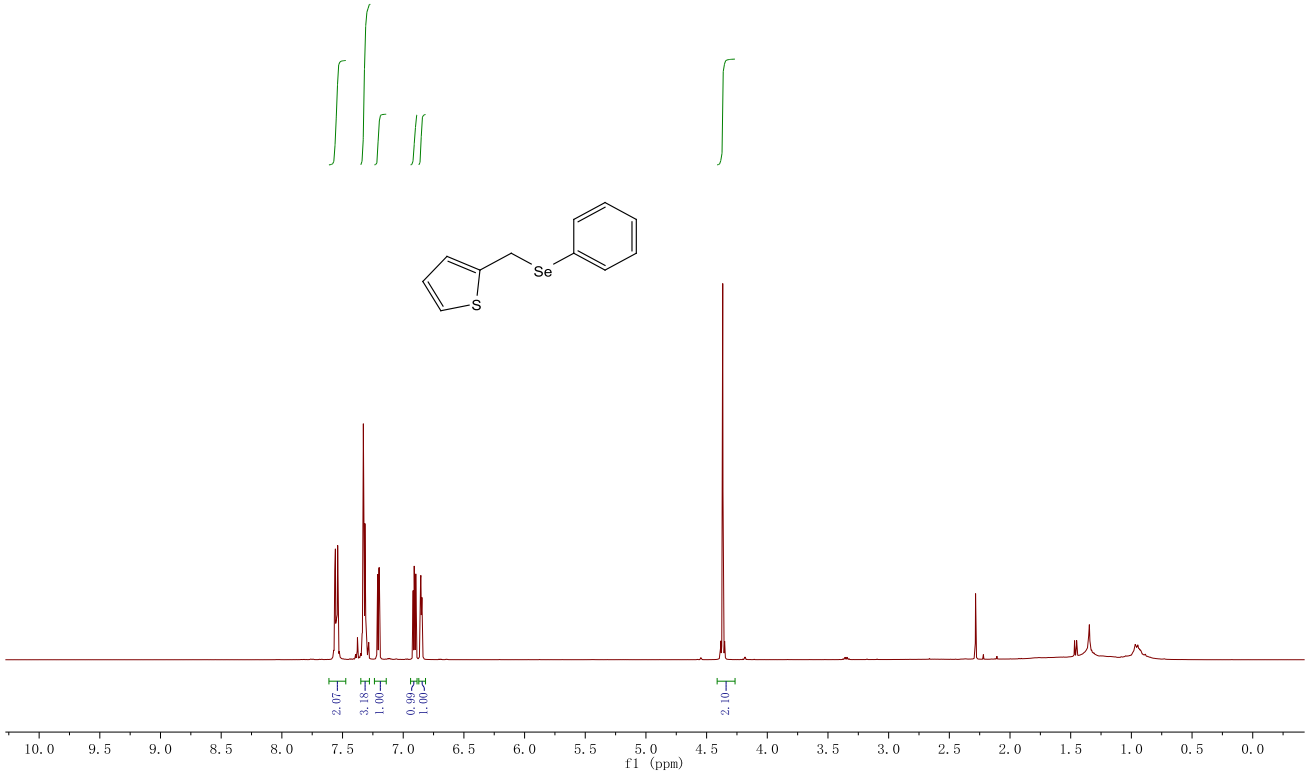
Product 3pa-Se

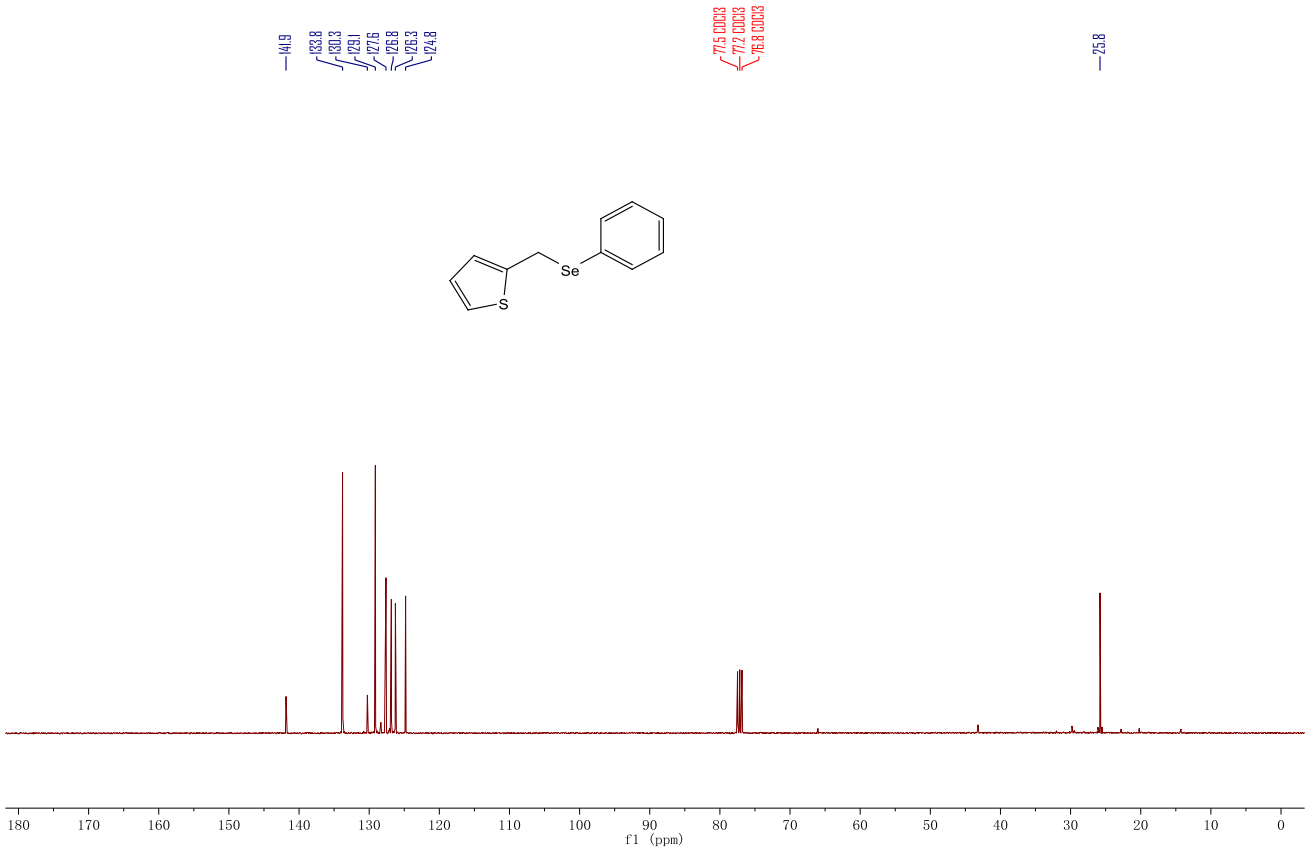




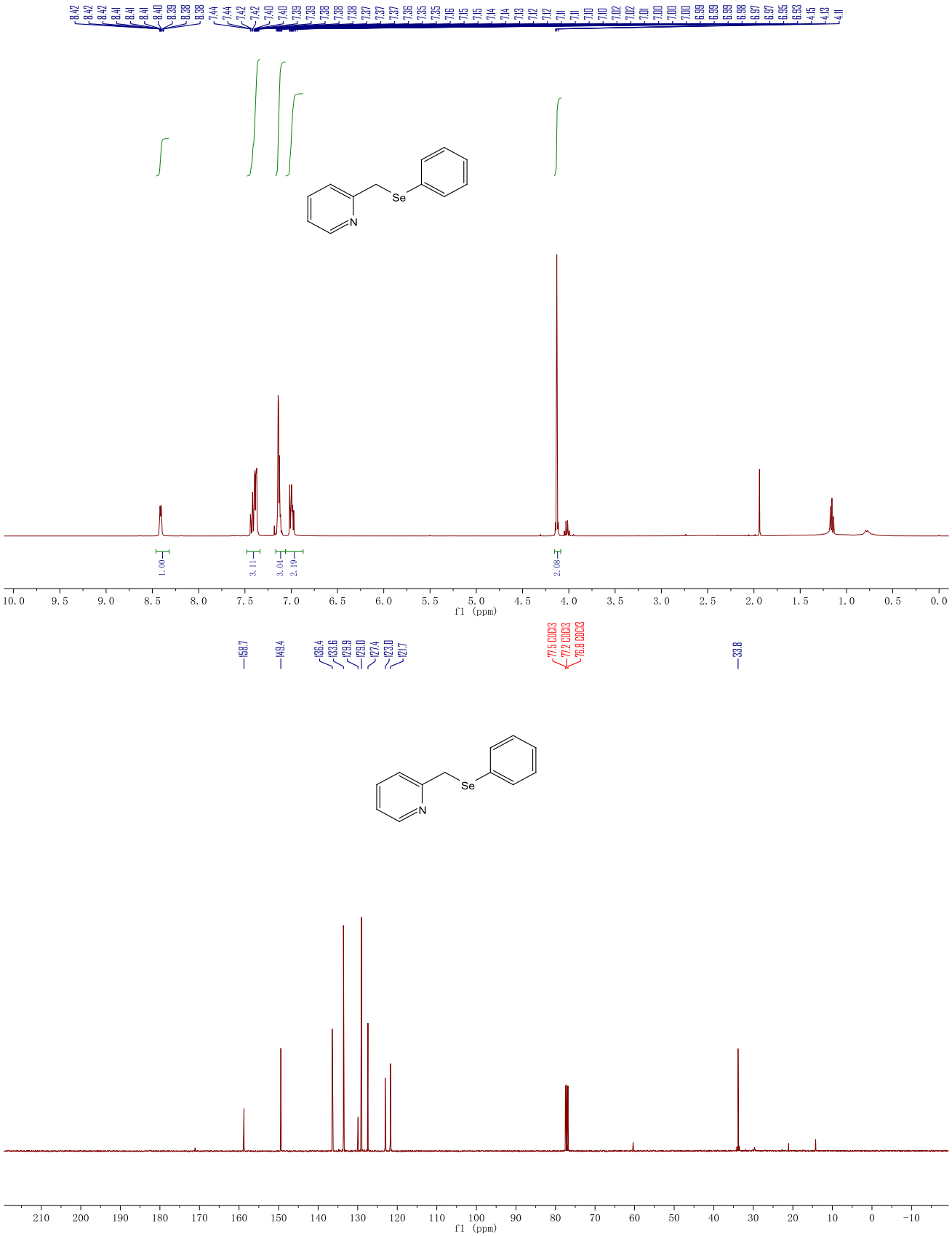


Product 3qa-Se

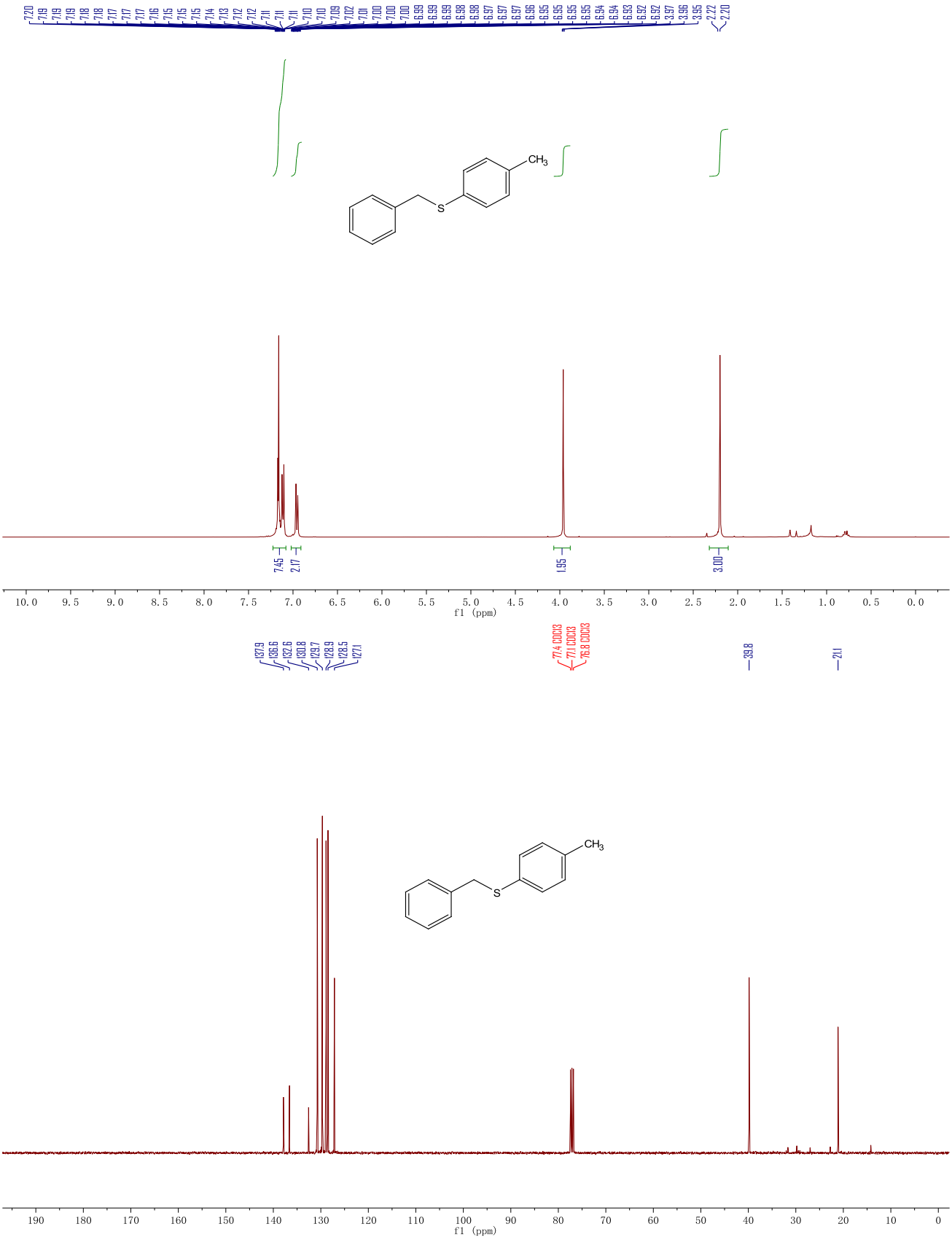




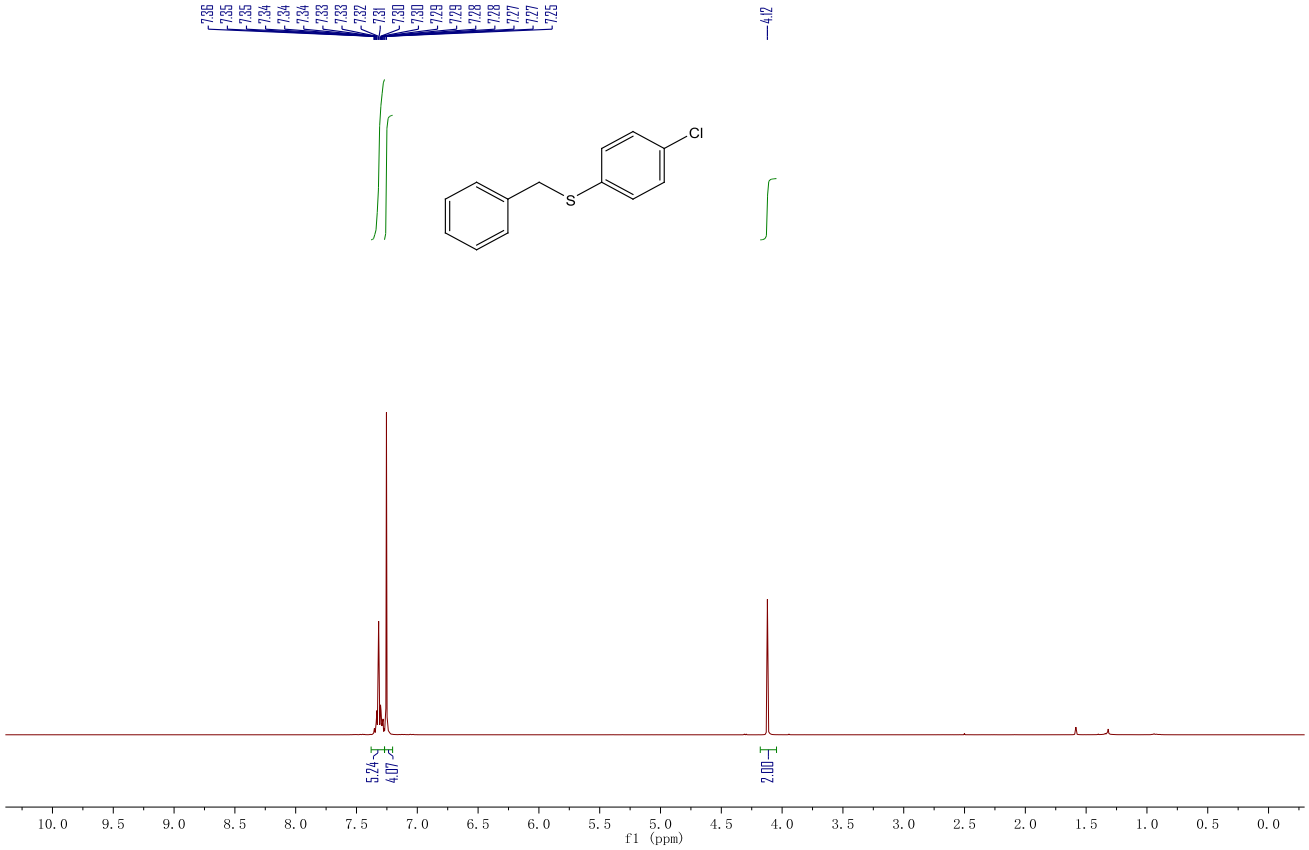
Product 3ra-Se

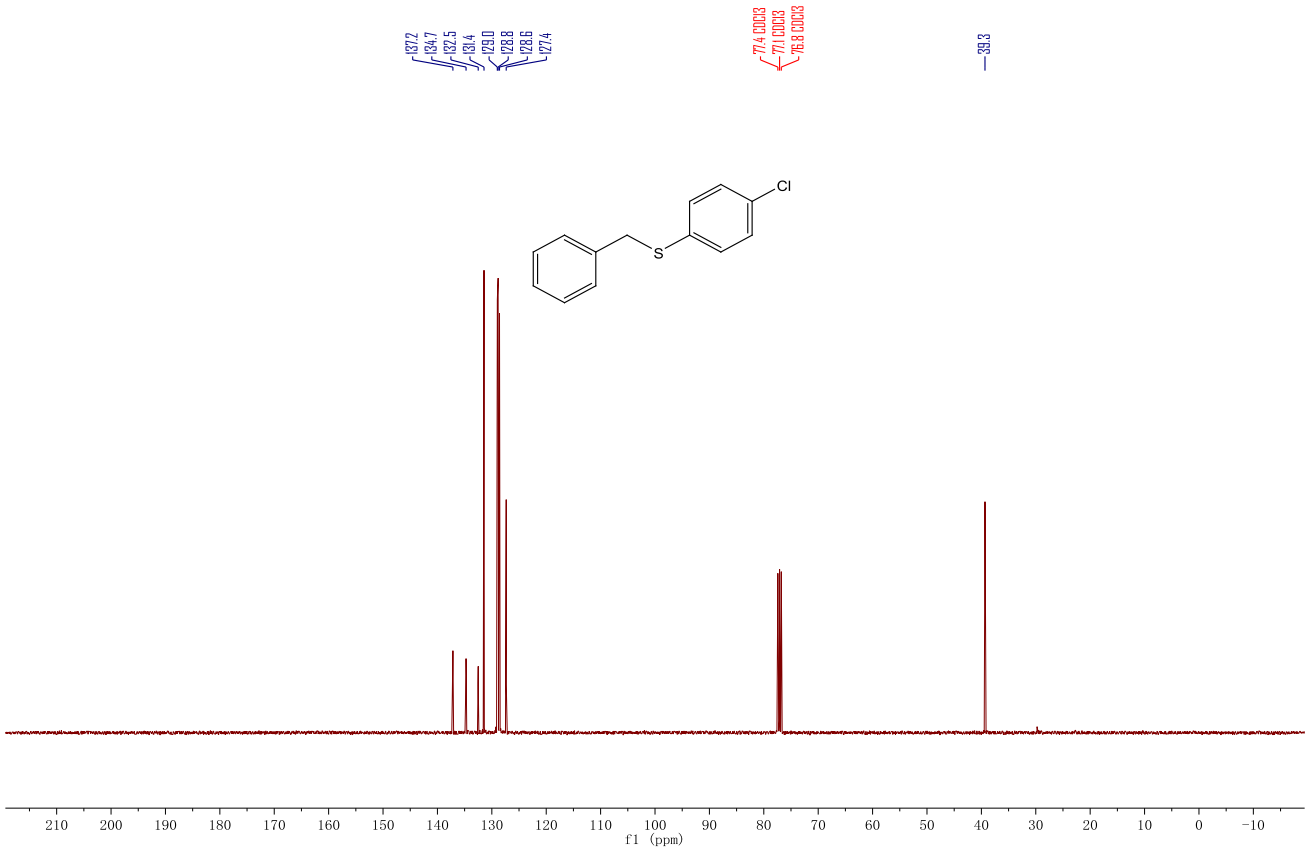


Product 3ab-S

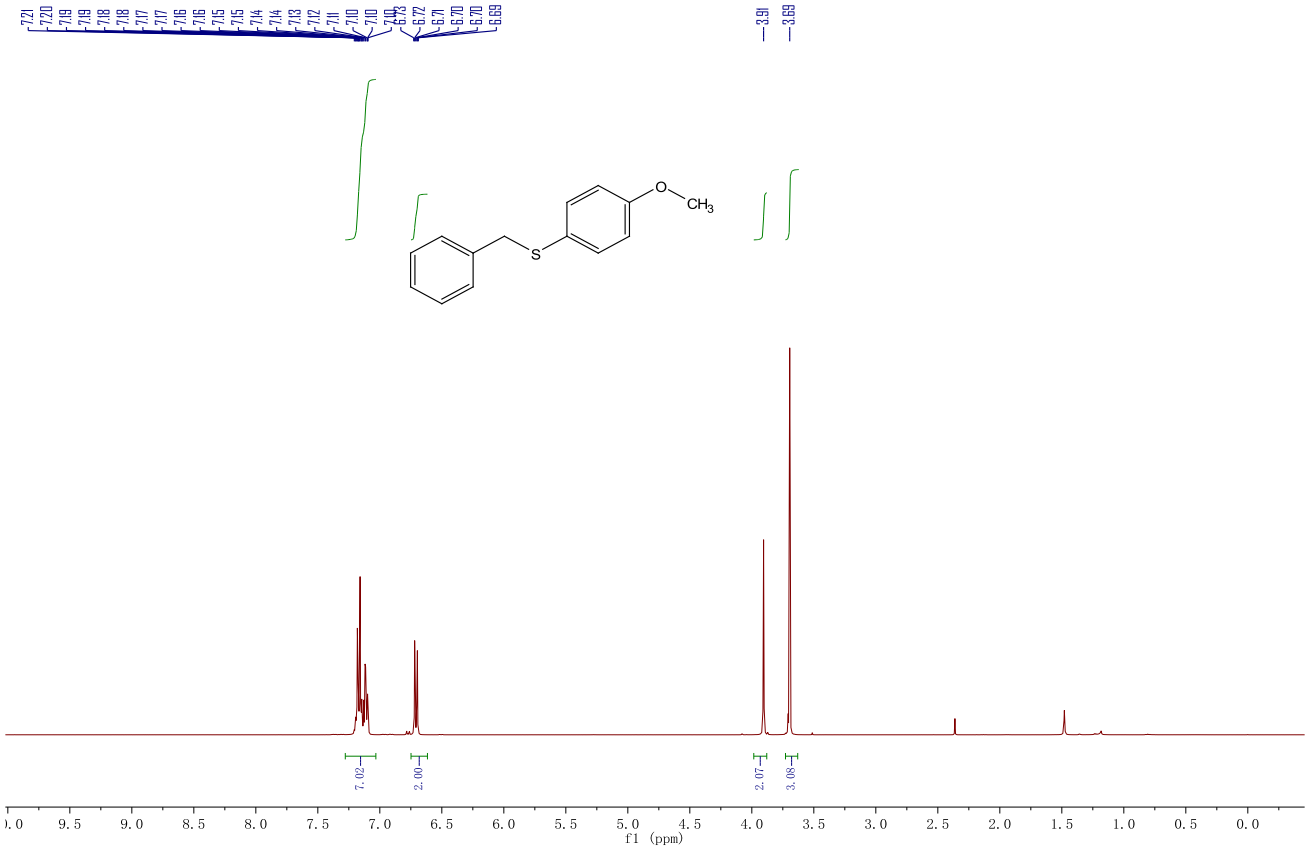


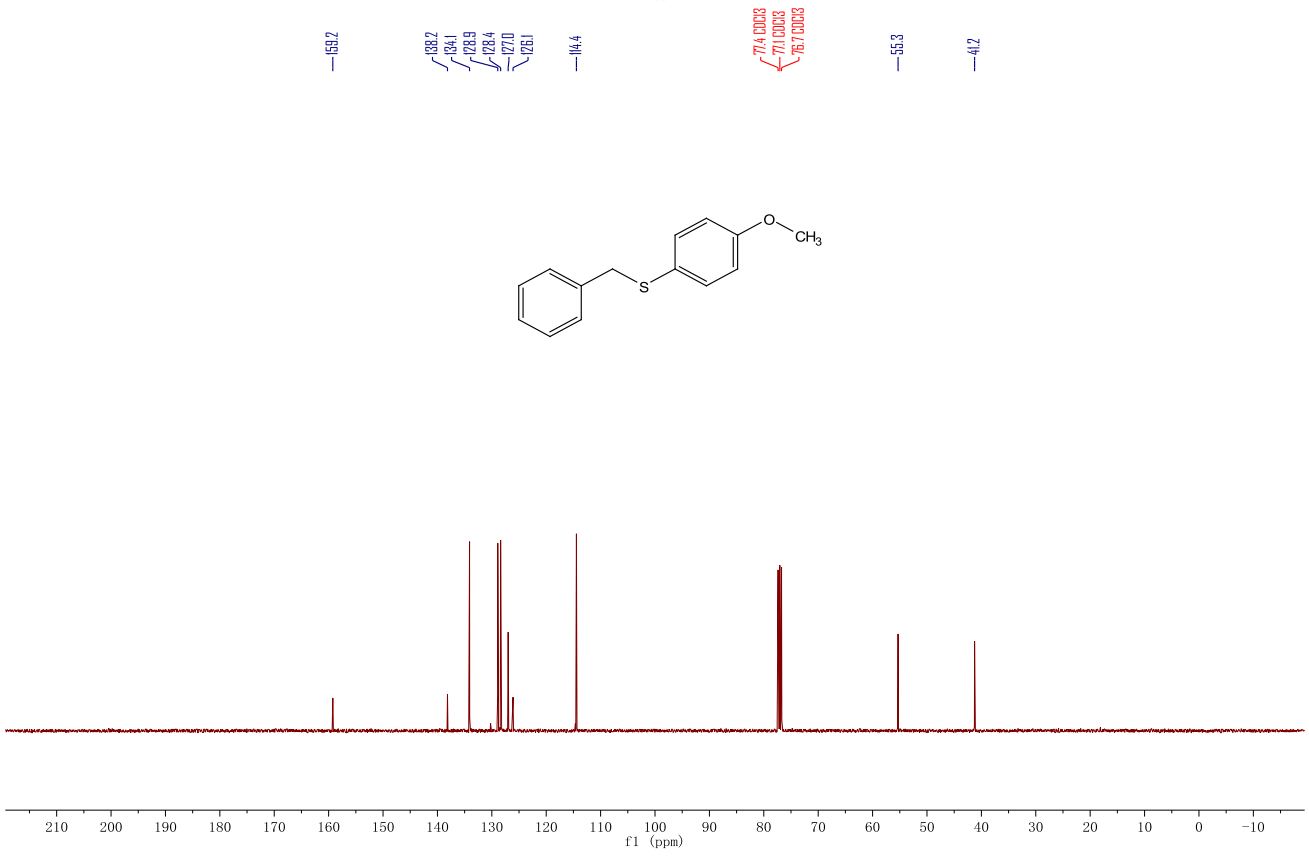
Product 3ac-S



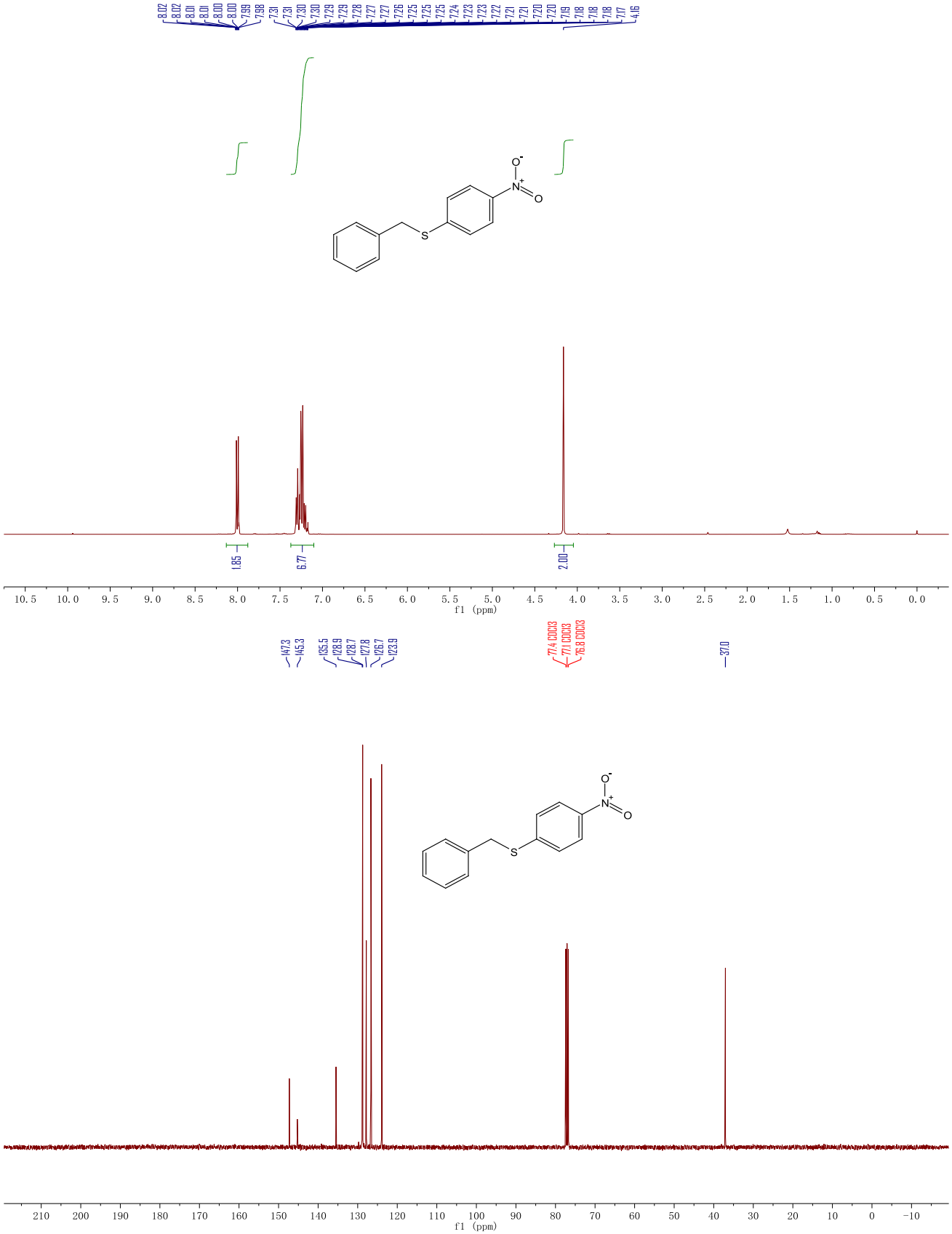


Product 3ad-S

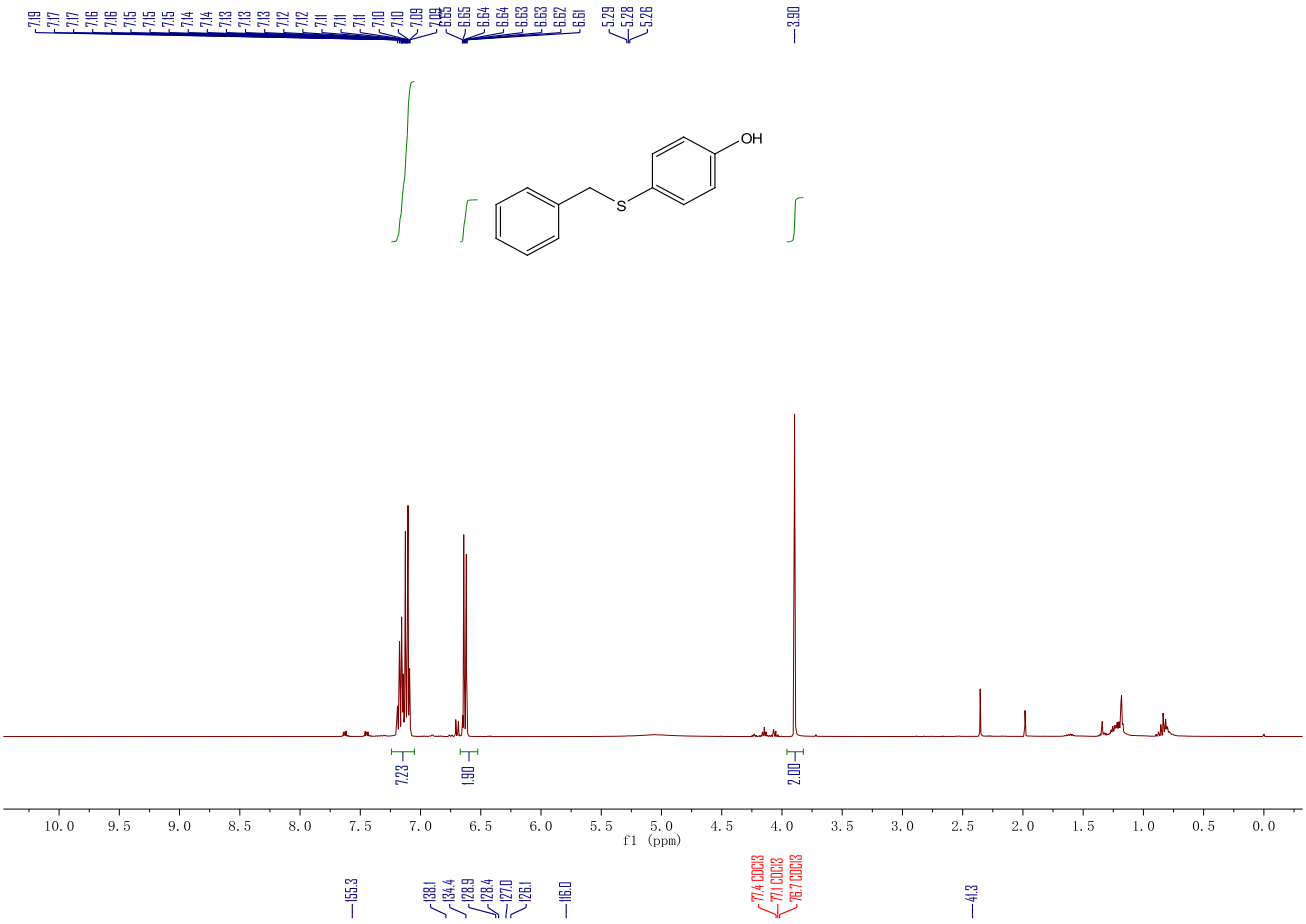


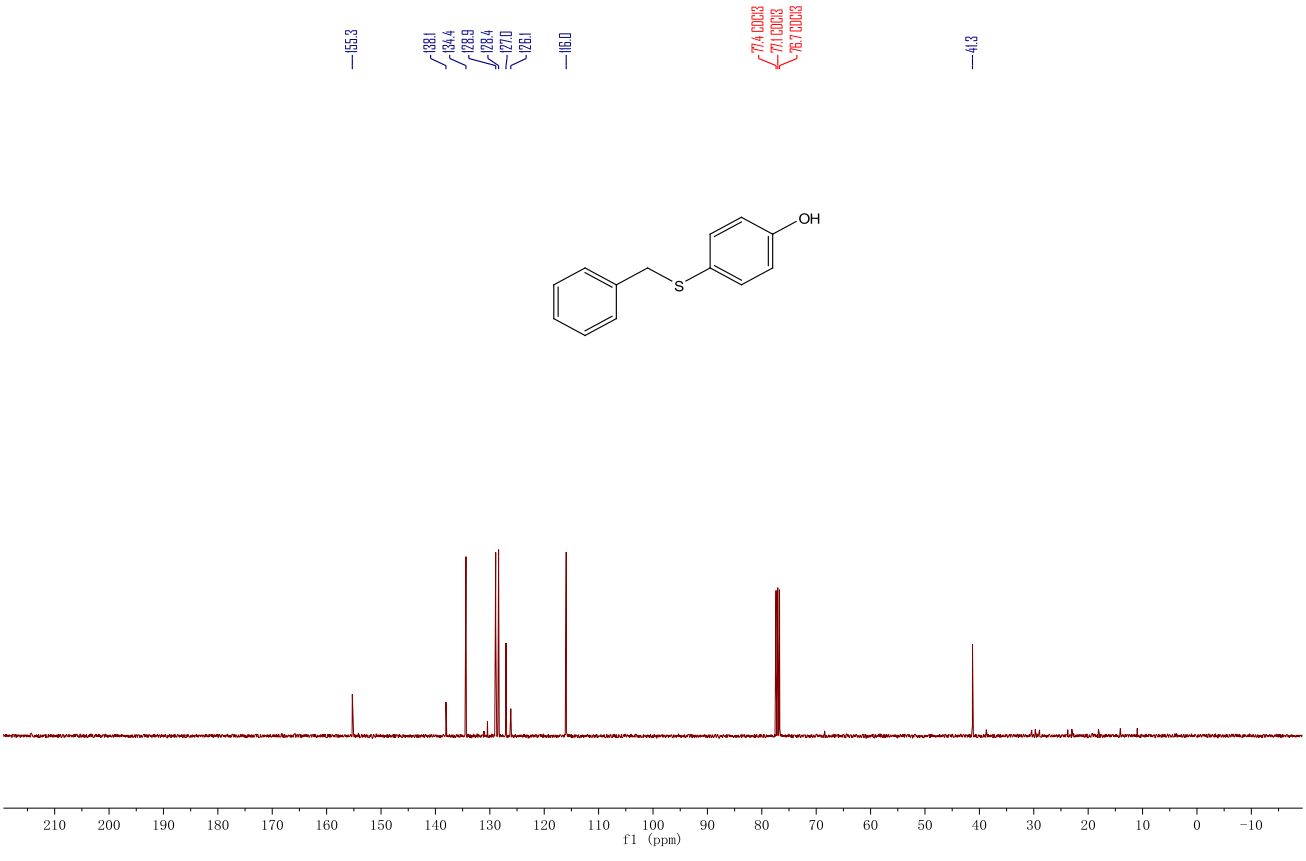


Product 3ae-S

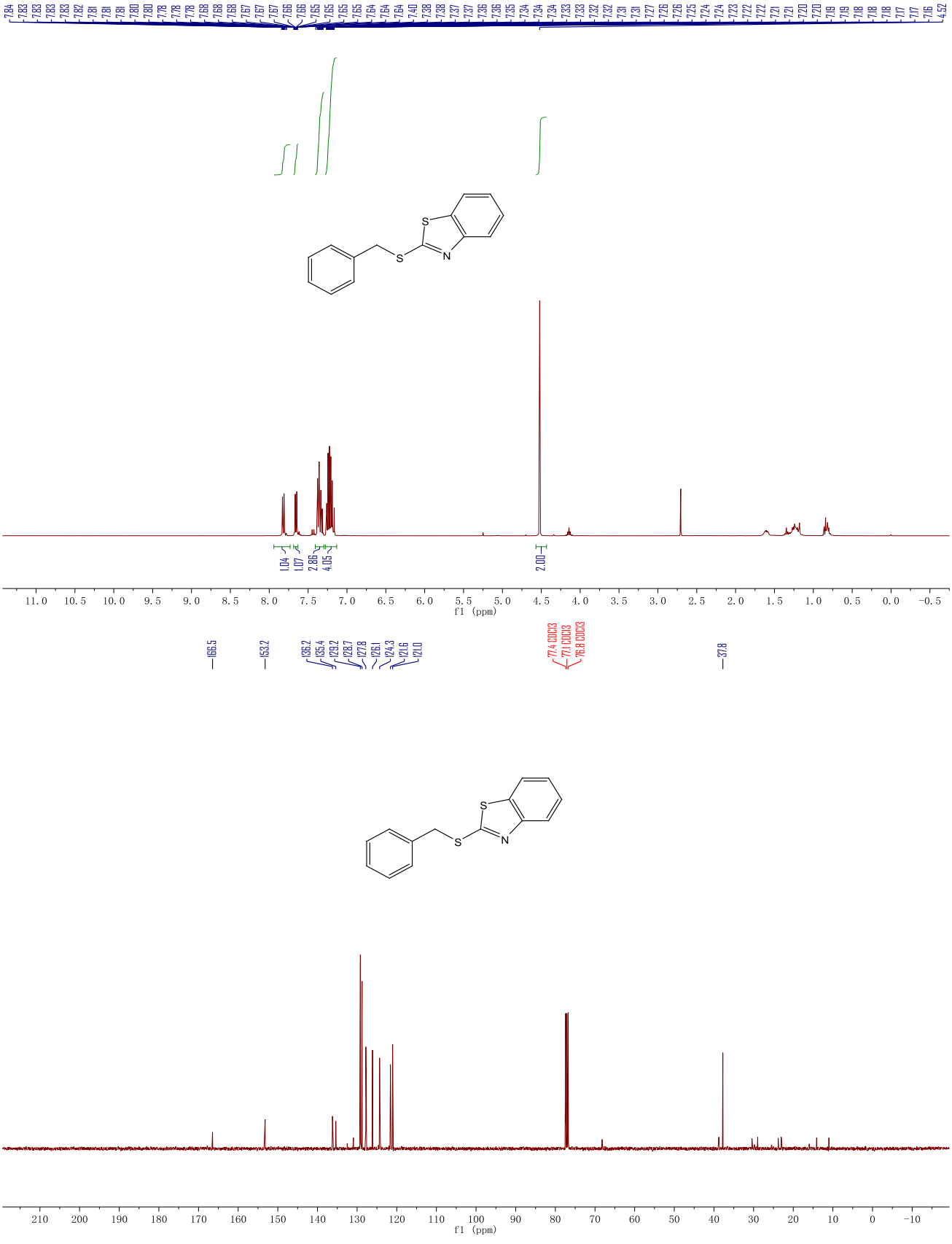


Product 3af-S

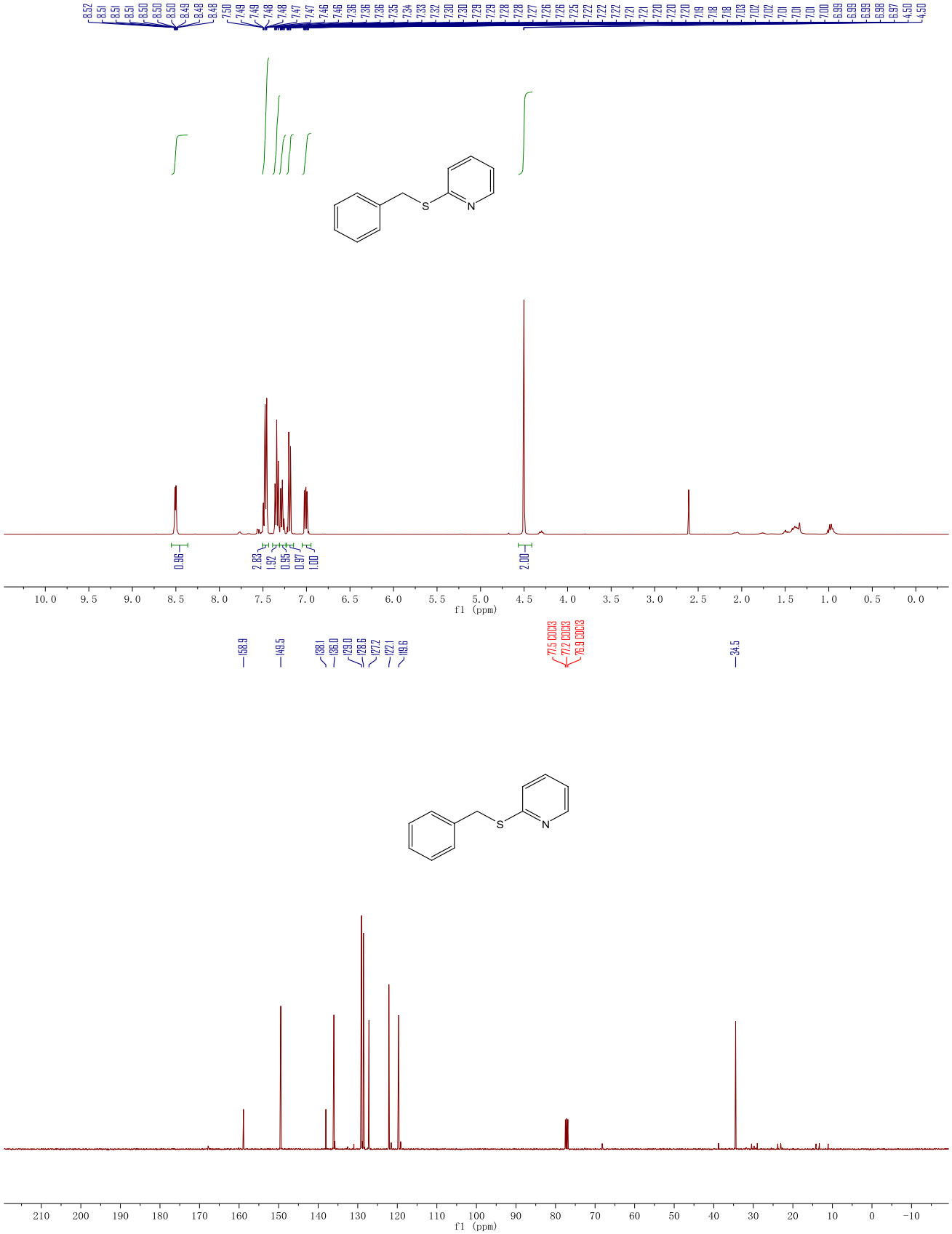




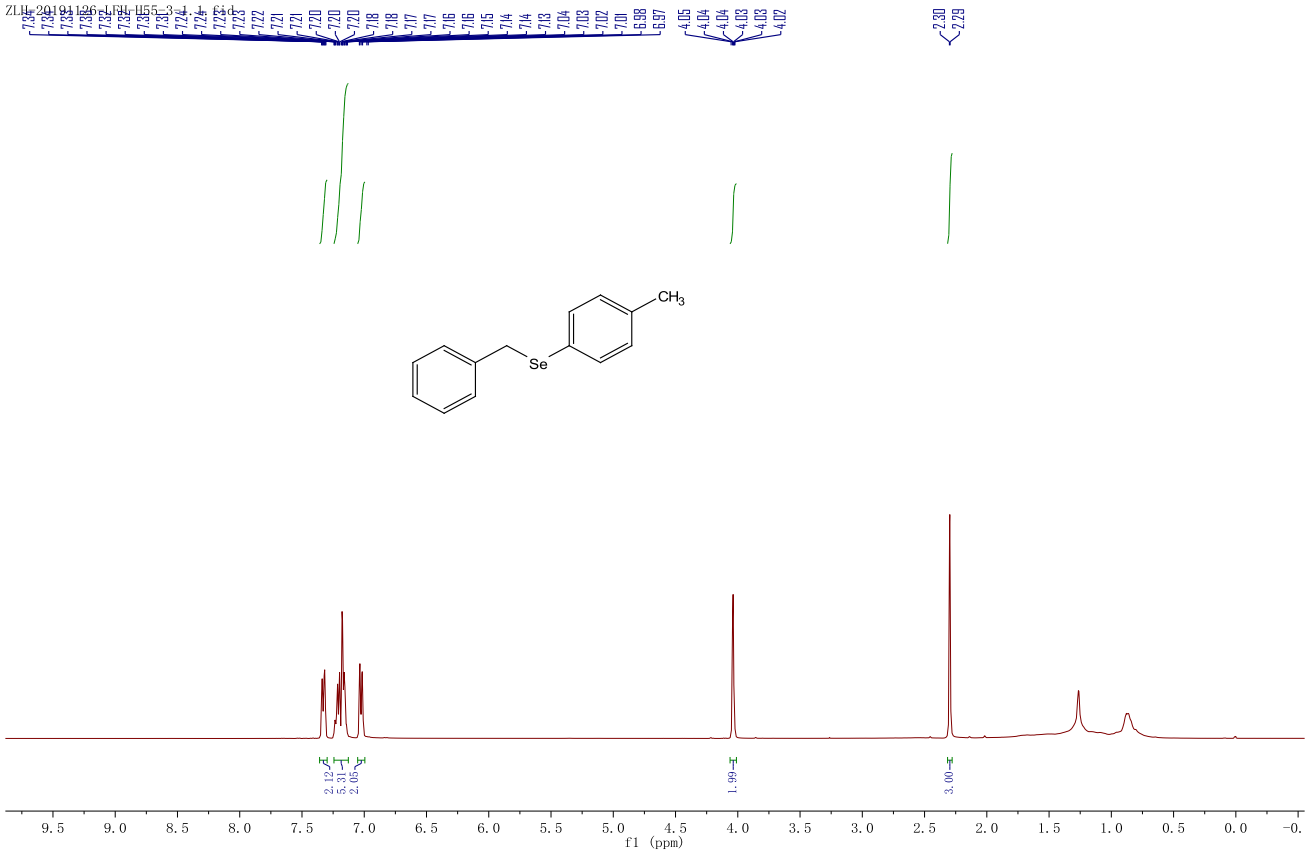
Product 3ag-S



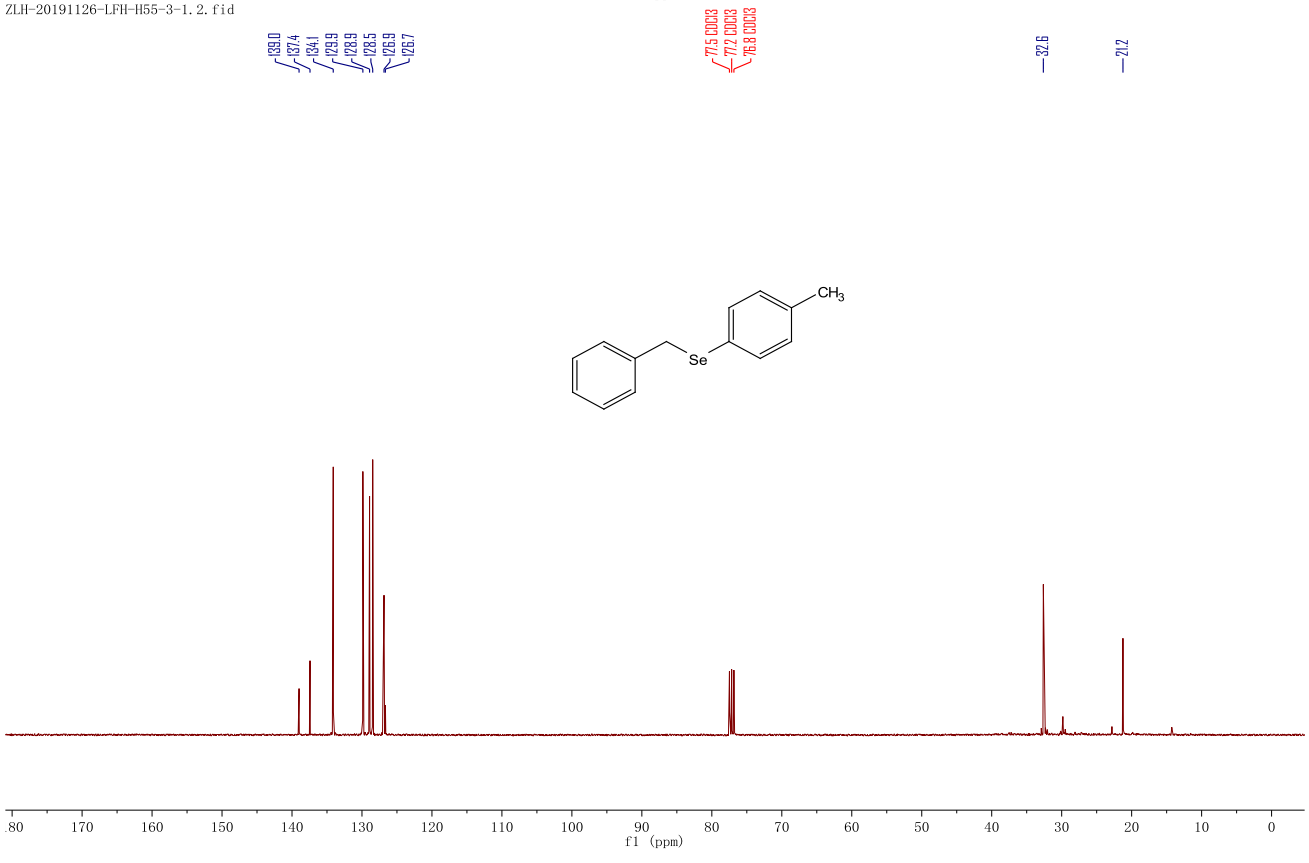
Product 3ah-S



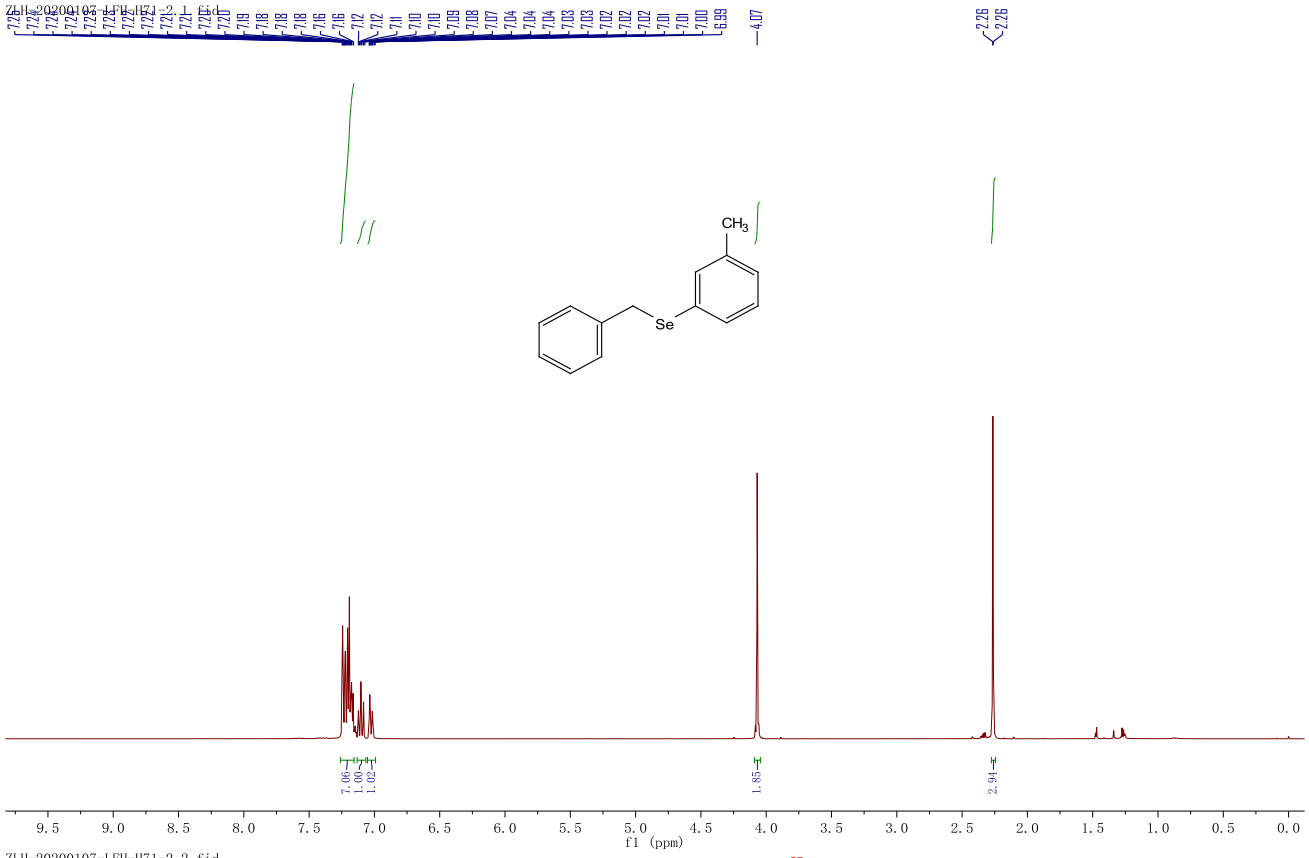
Product 3ab-Se



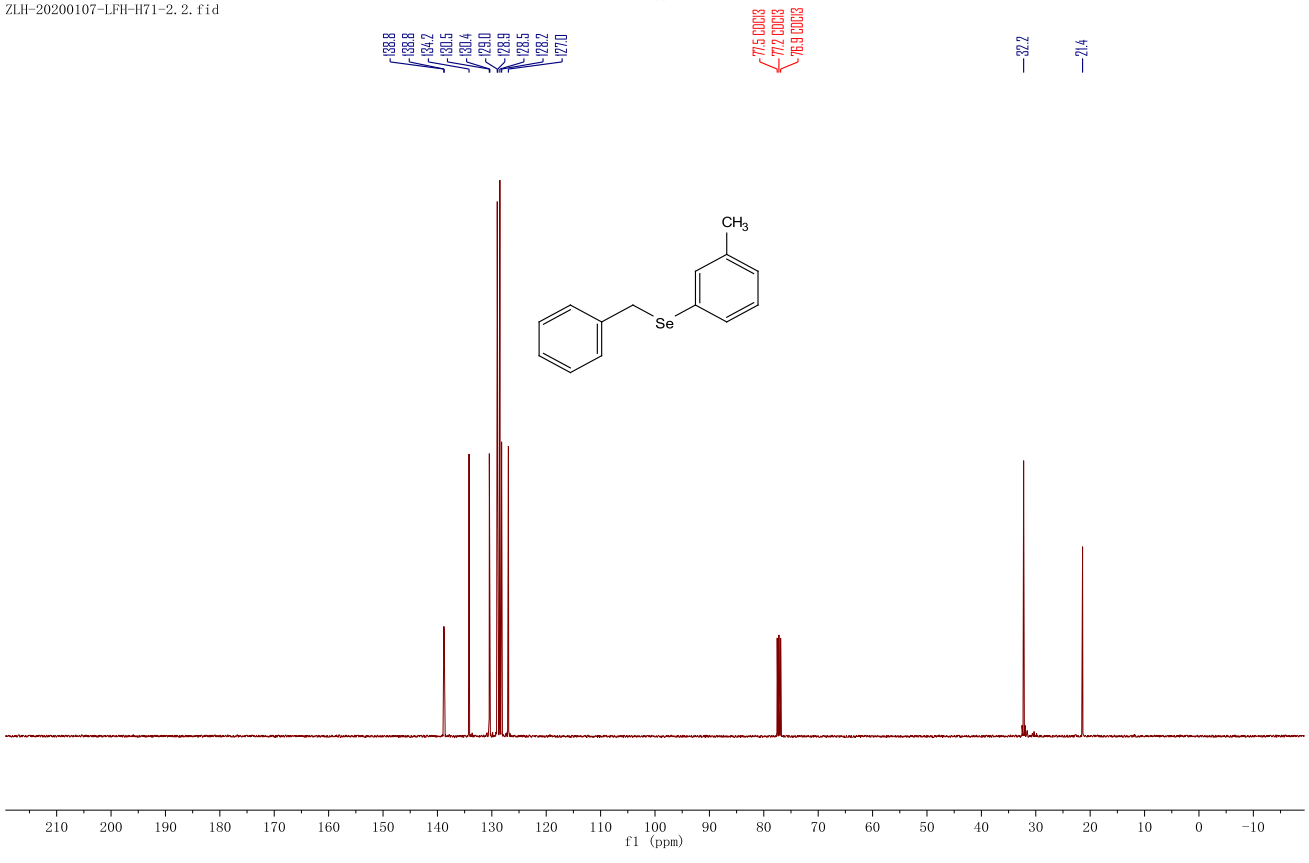
ZLH-20191126-LFH-H55-3-1.2.fid



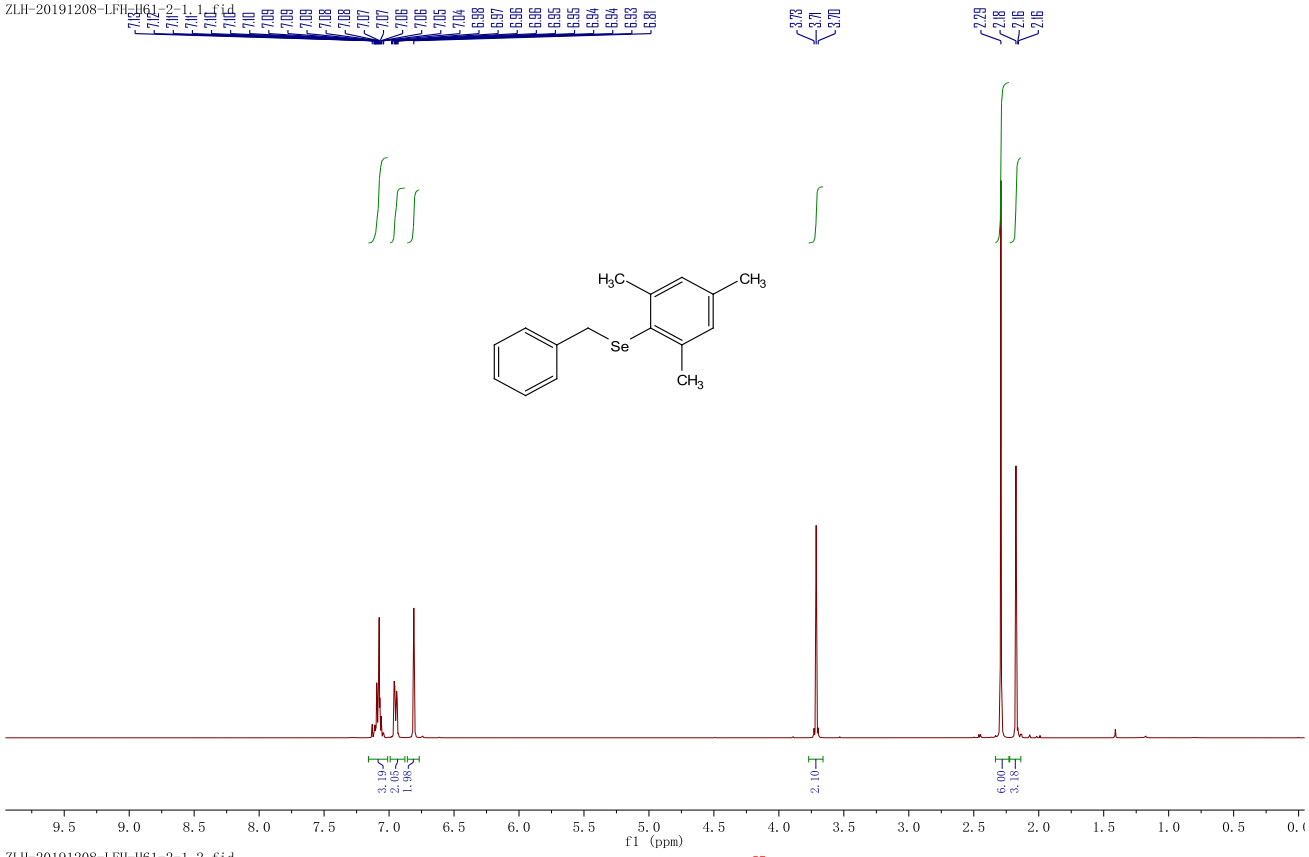
Product 3ac-Se

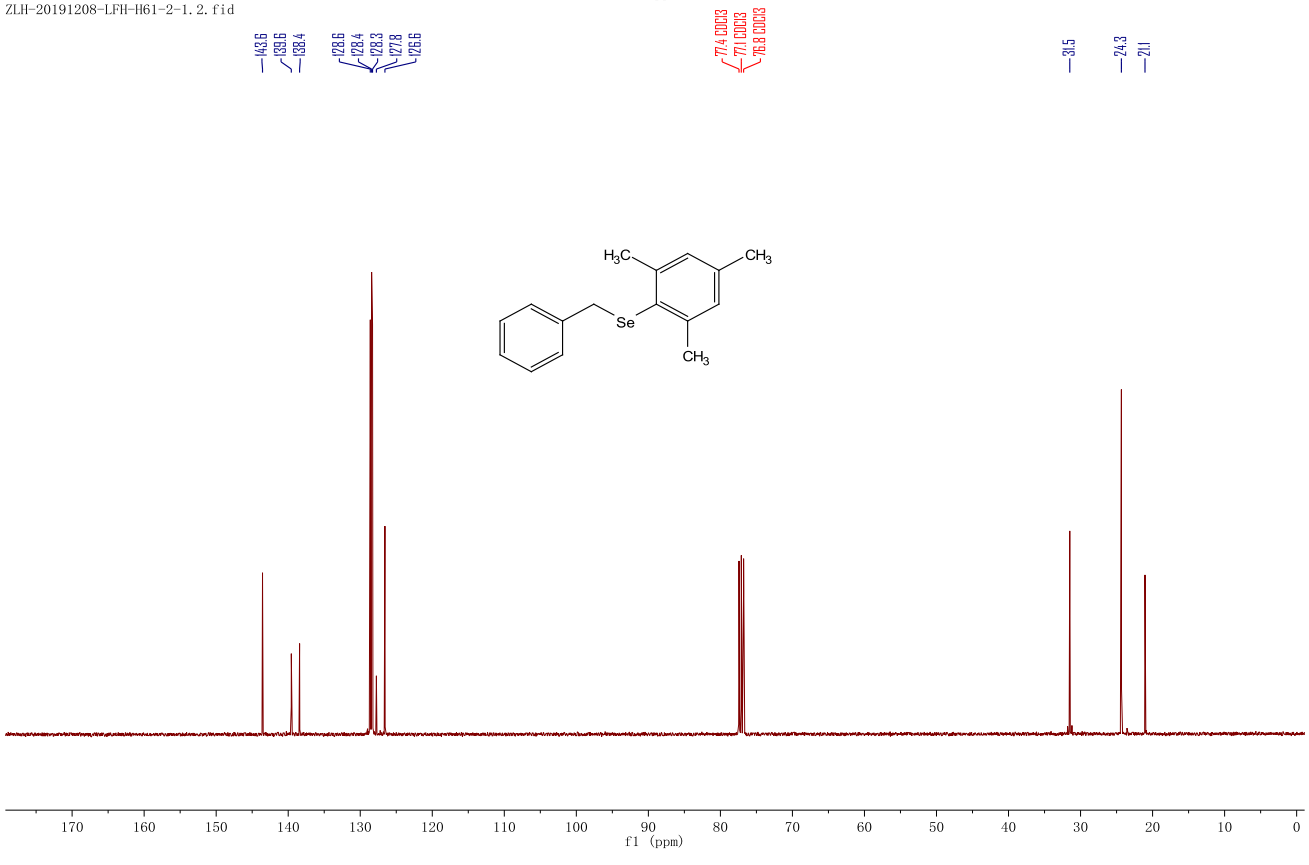


ZLH-20200107-LFH-H71-2.2.fid

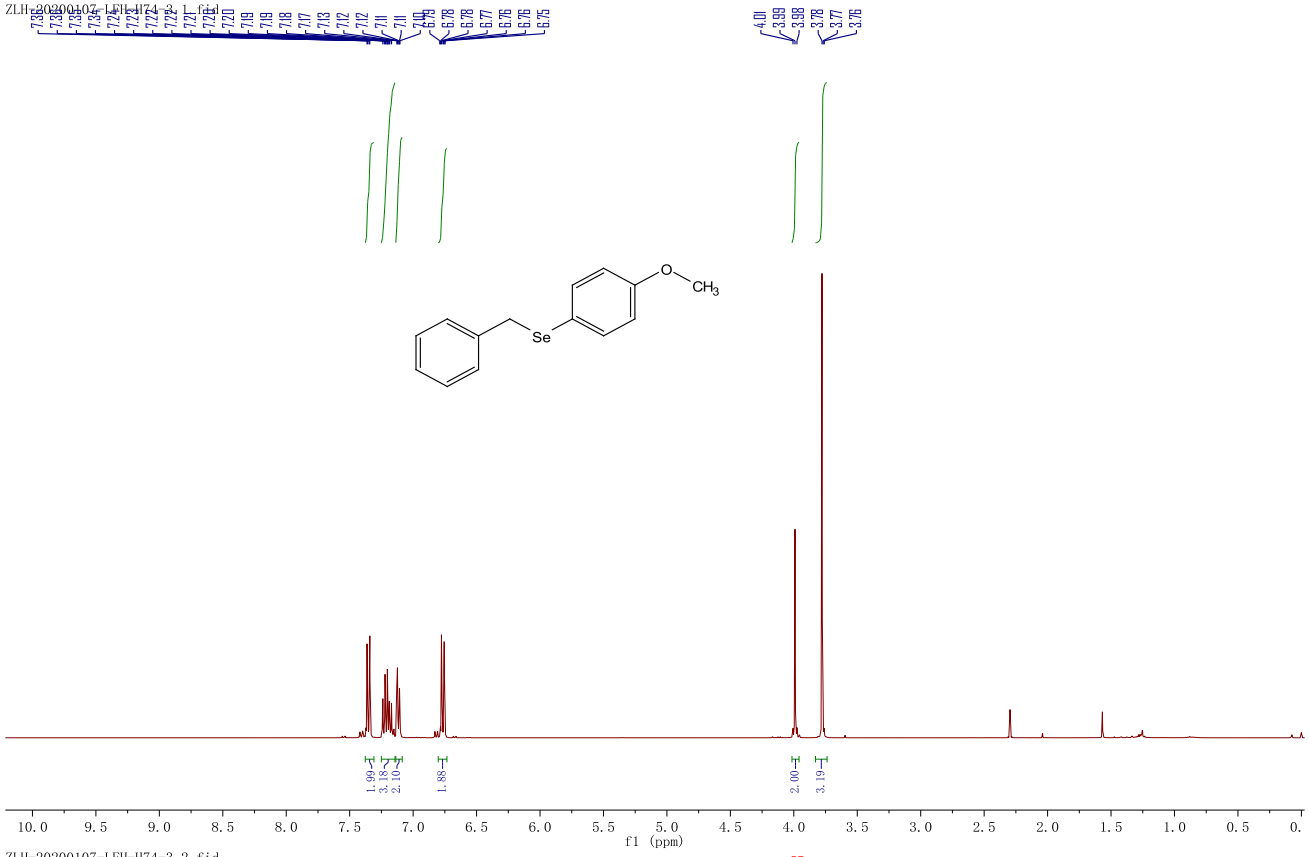


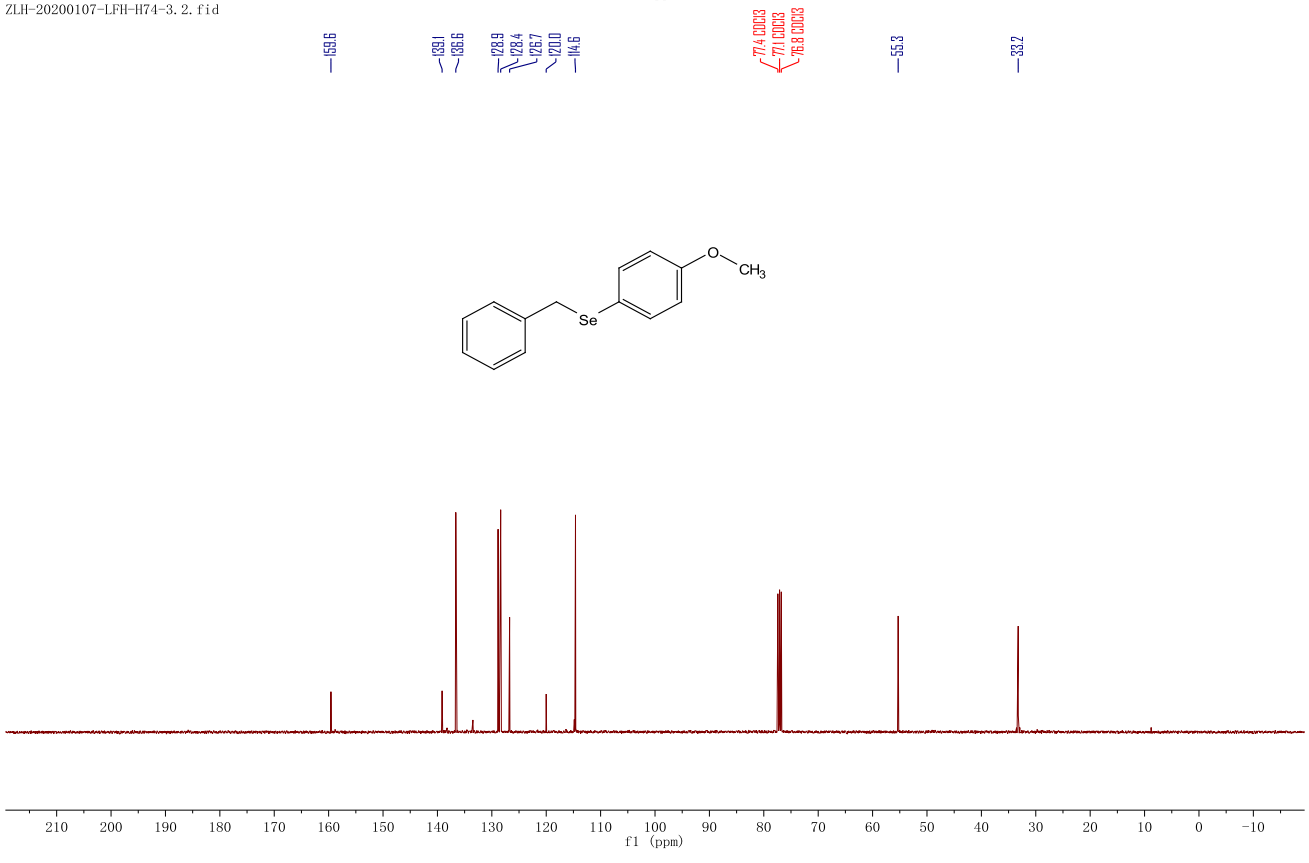
Product 3ad-Se



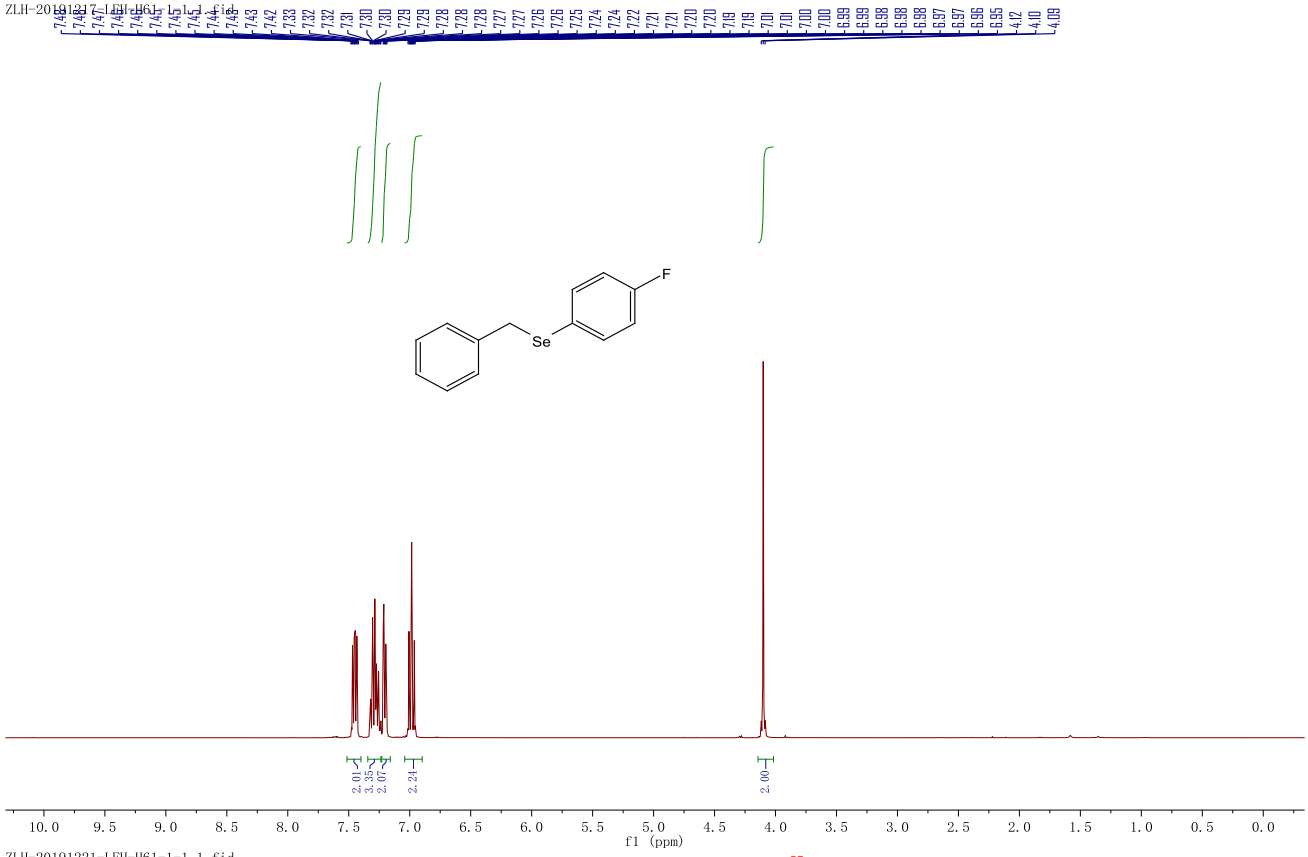


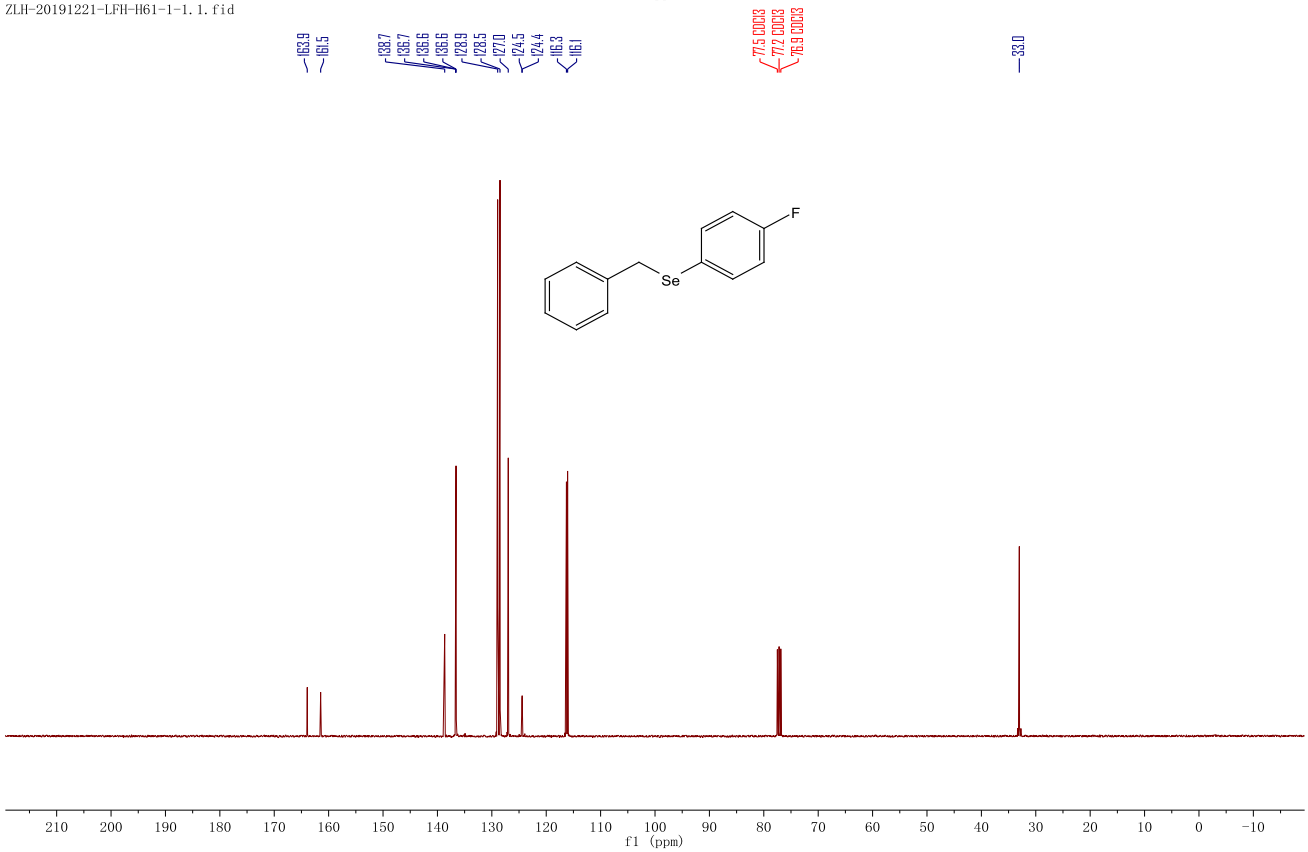
Product 3ae-Se



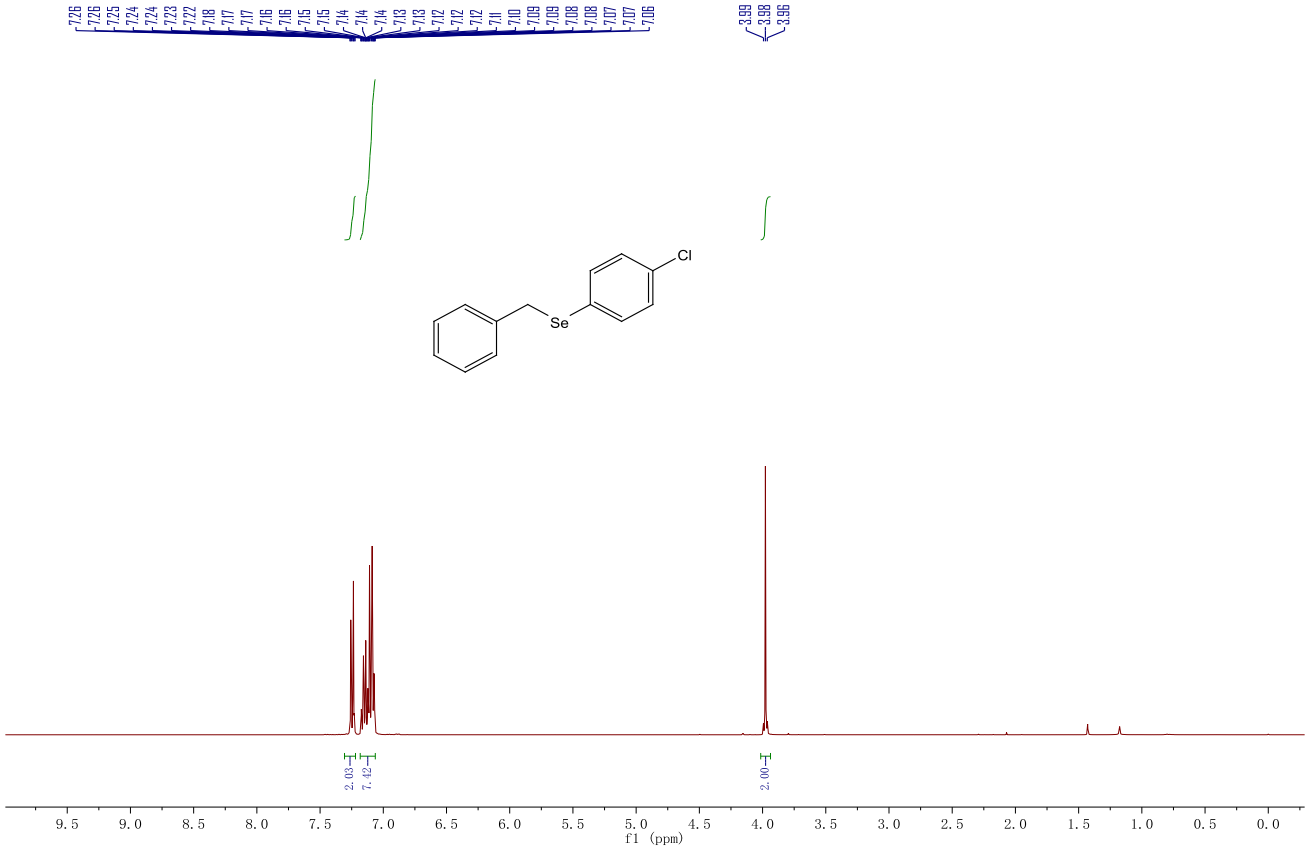


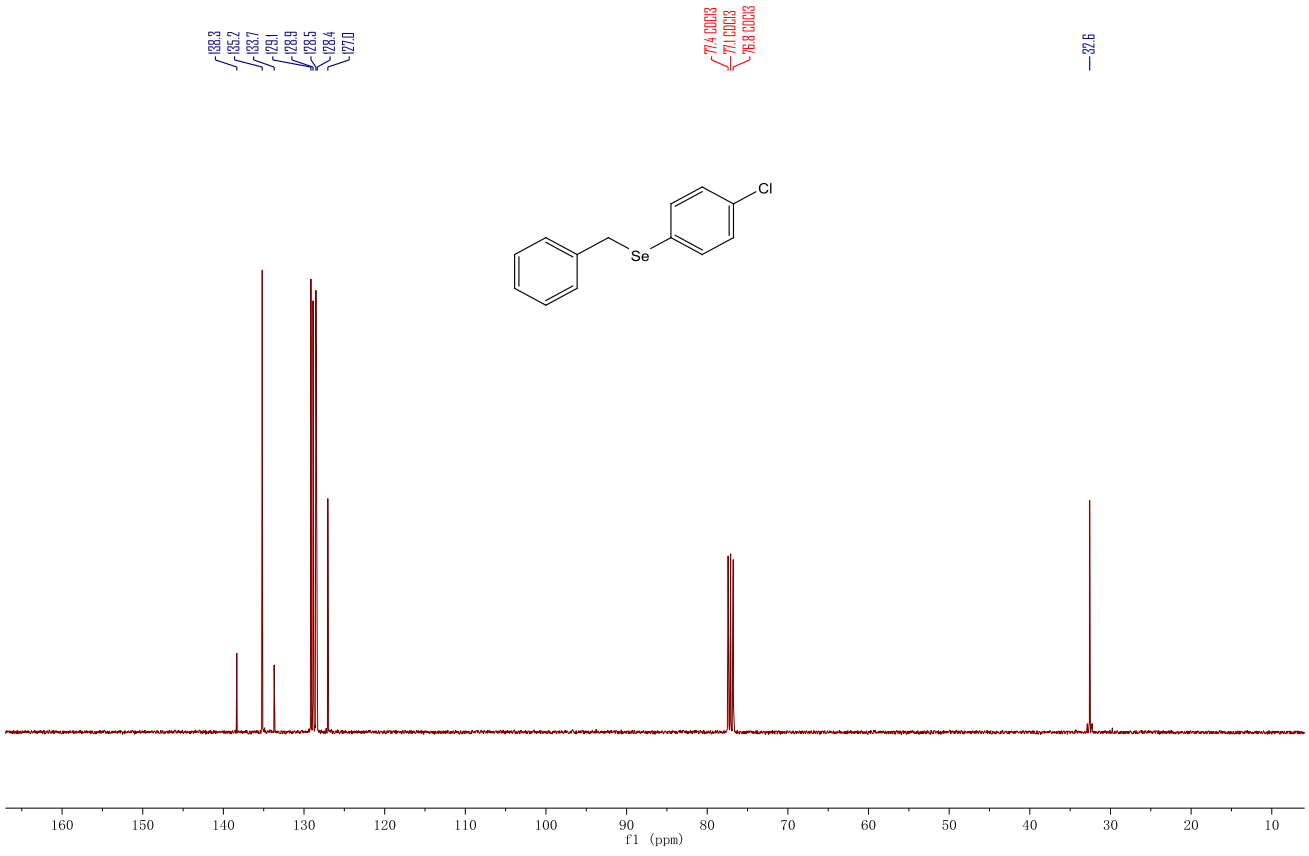
Product 3af-Se



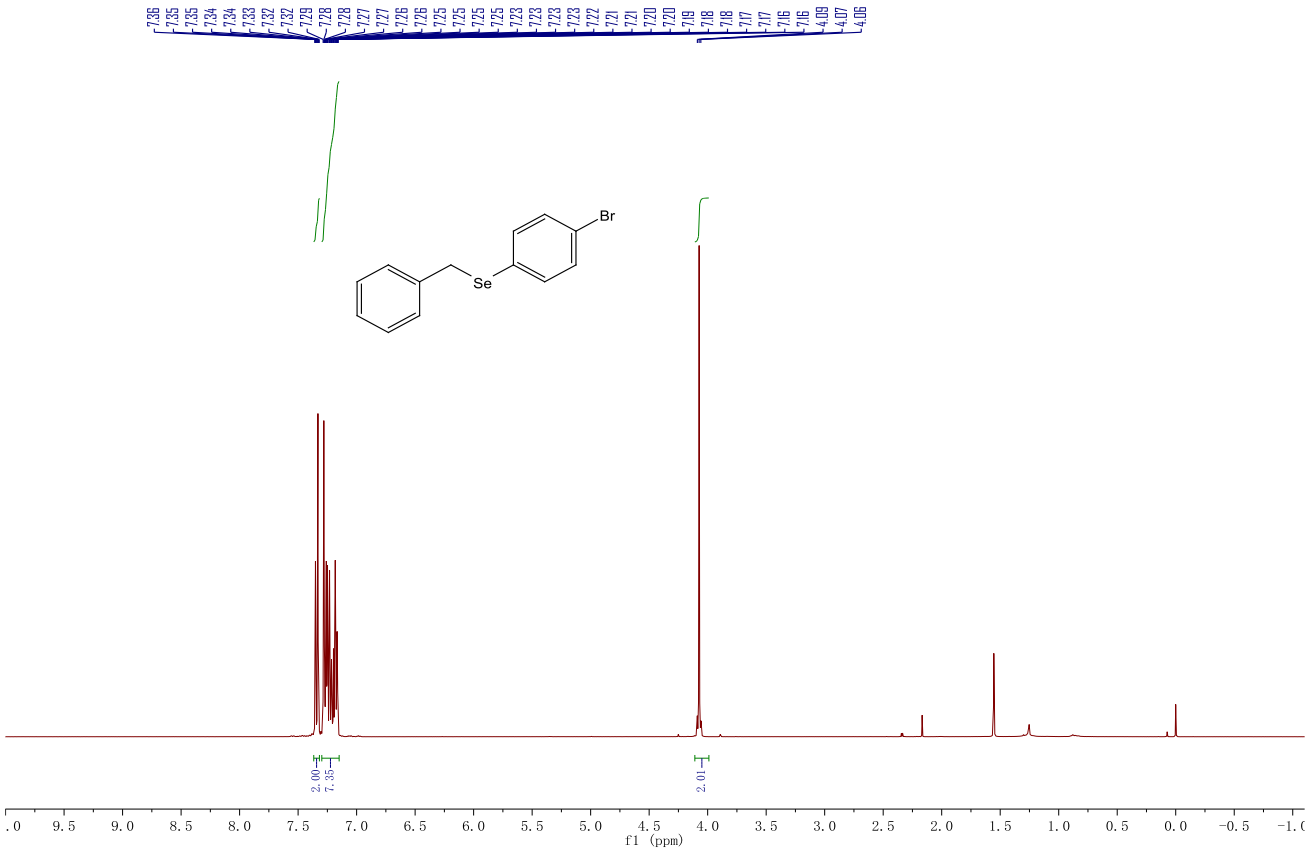


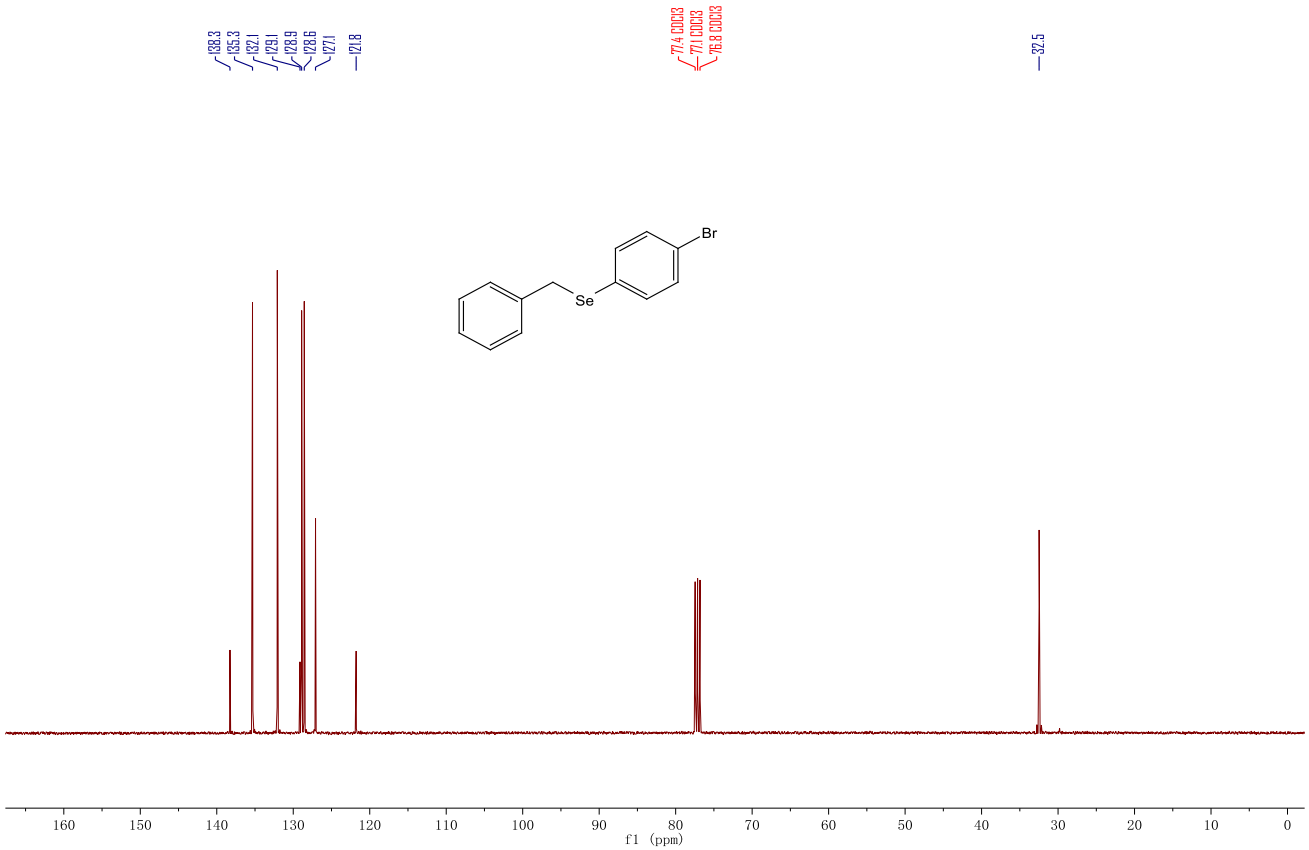
Product 3ag-Se



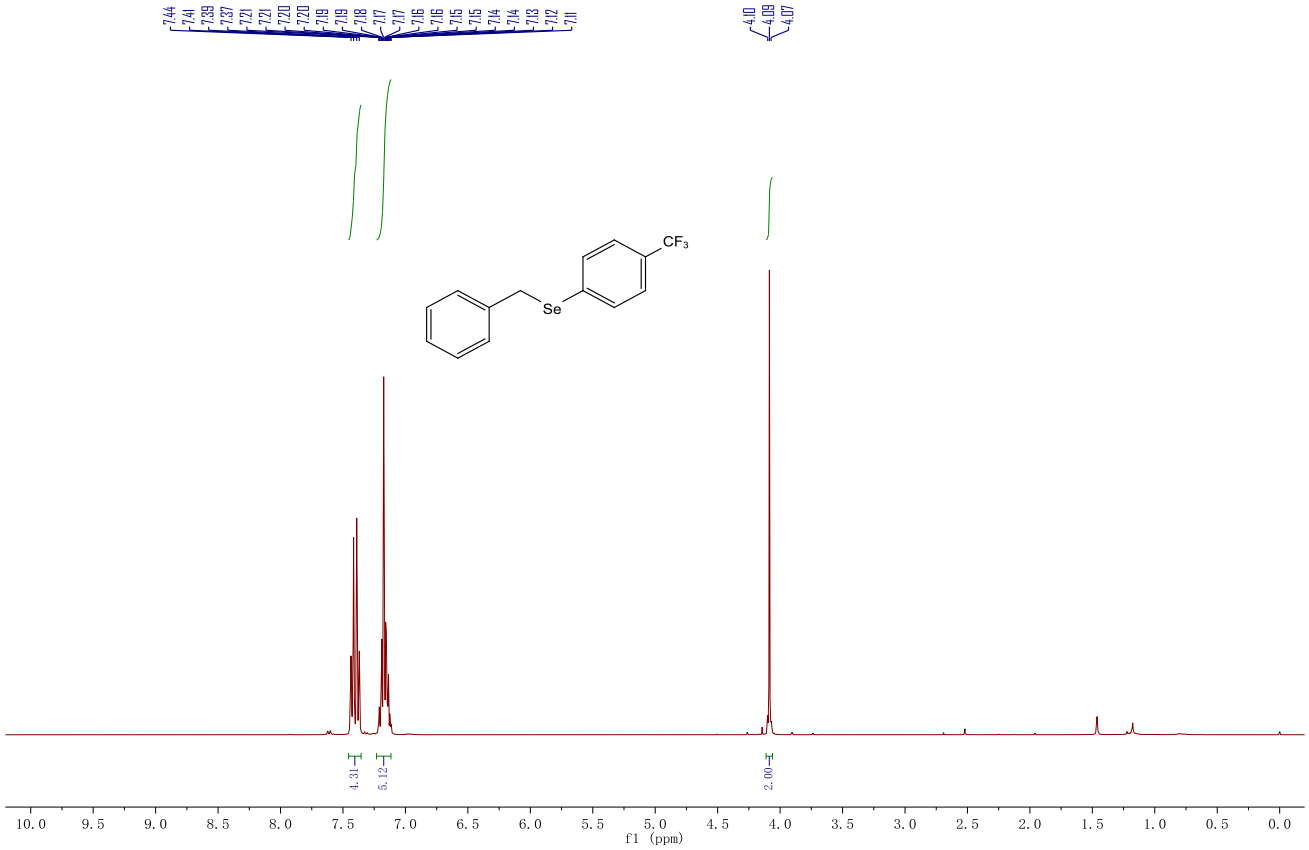


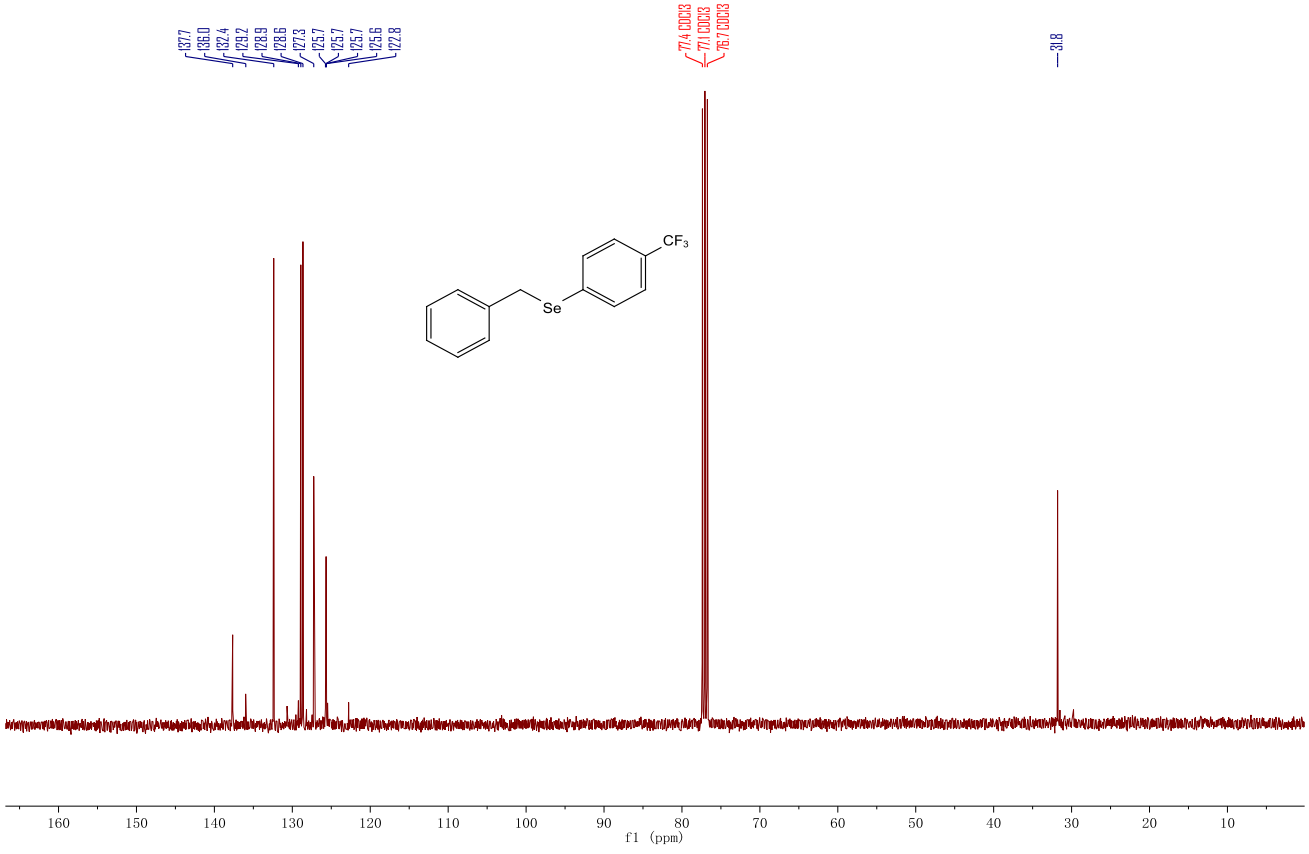
Product 3ah-Se



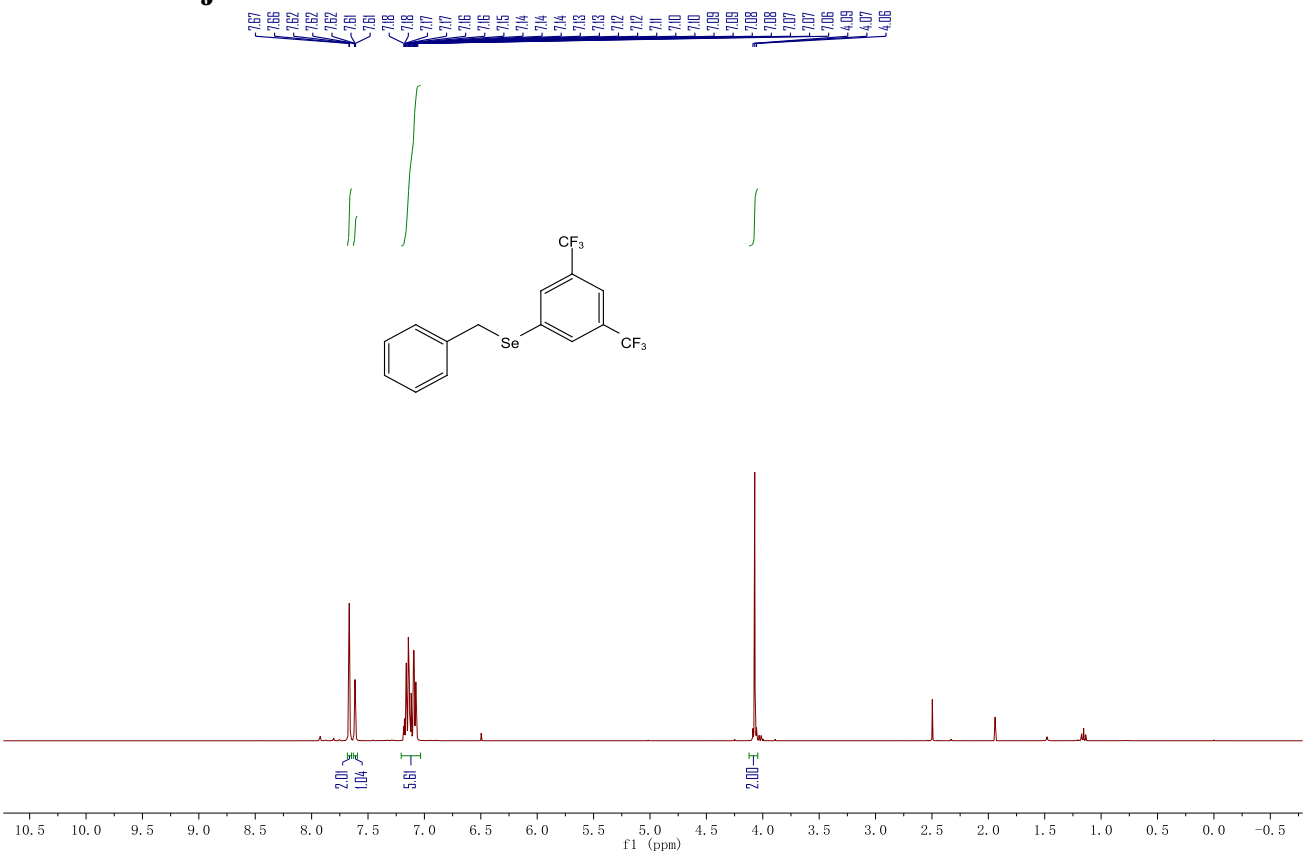


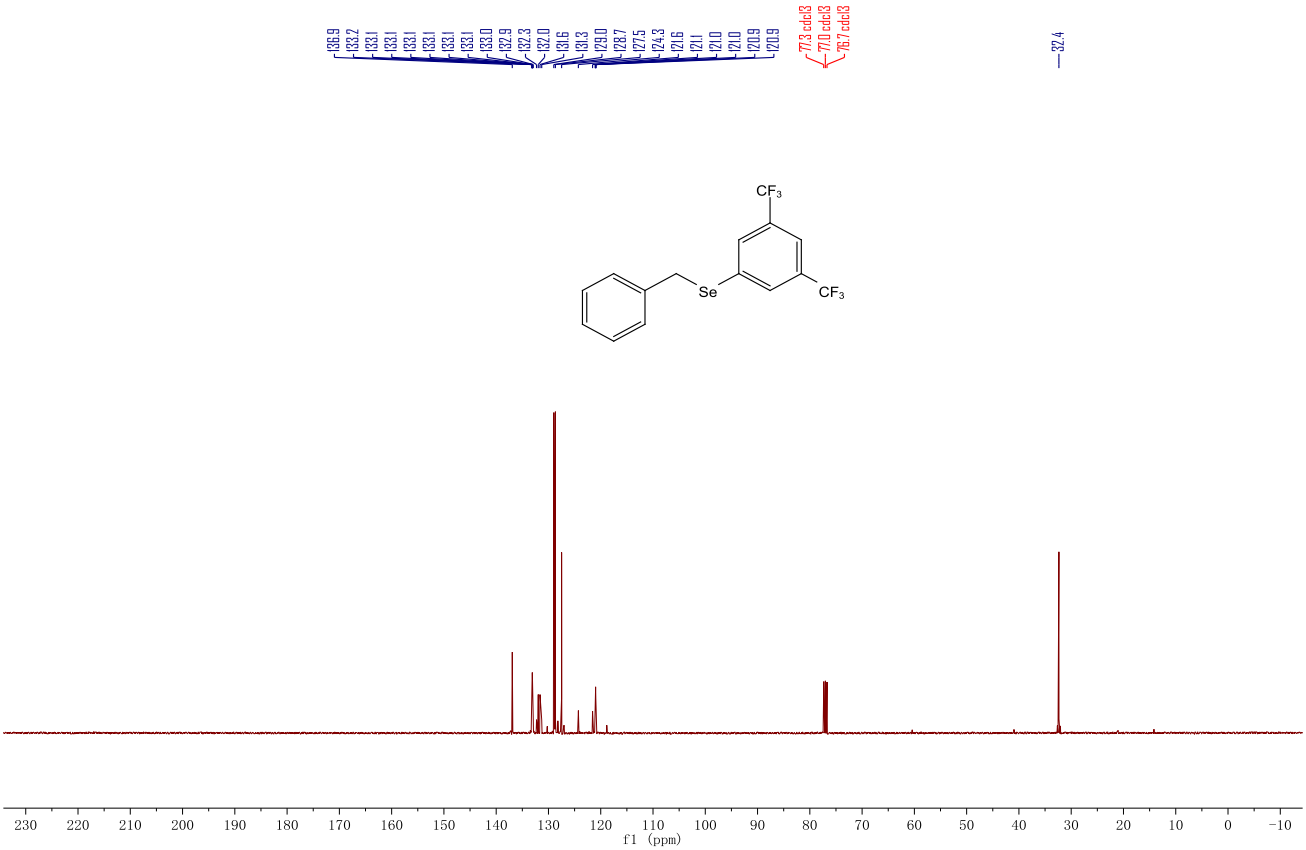
Product 3ai-Se



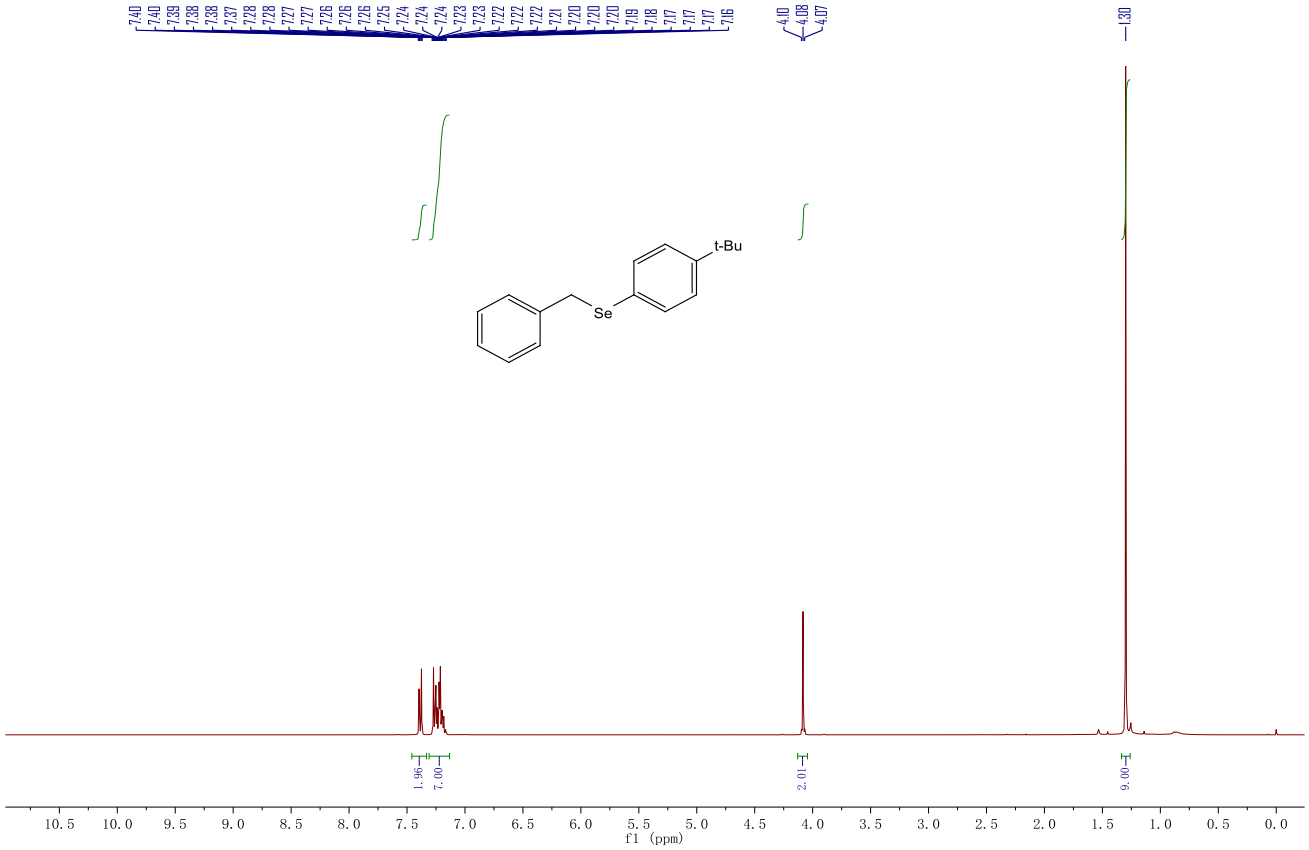


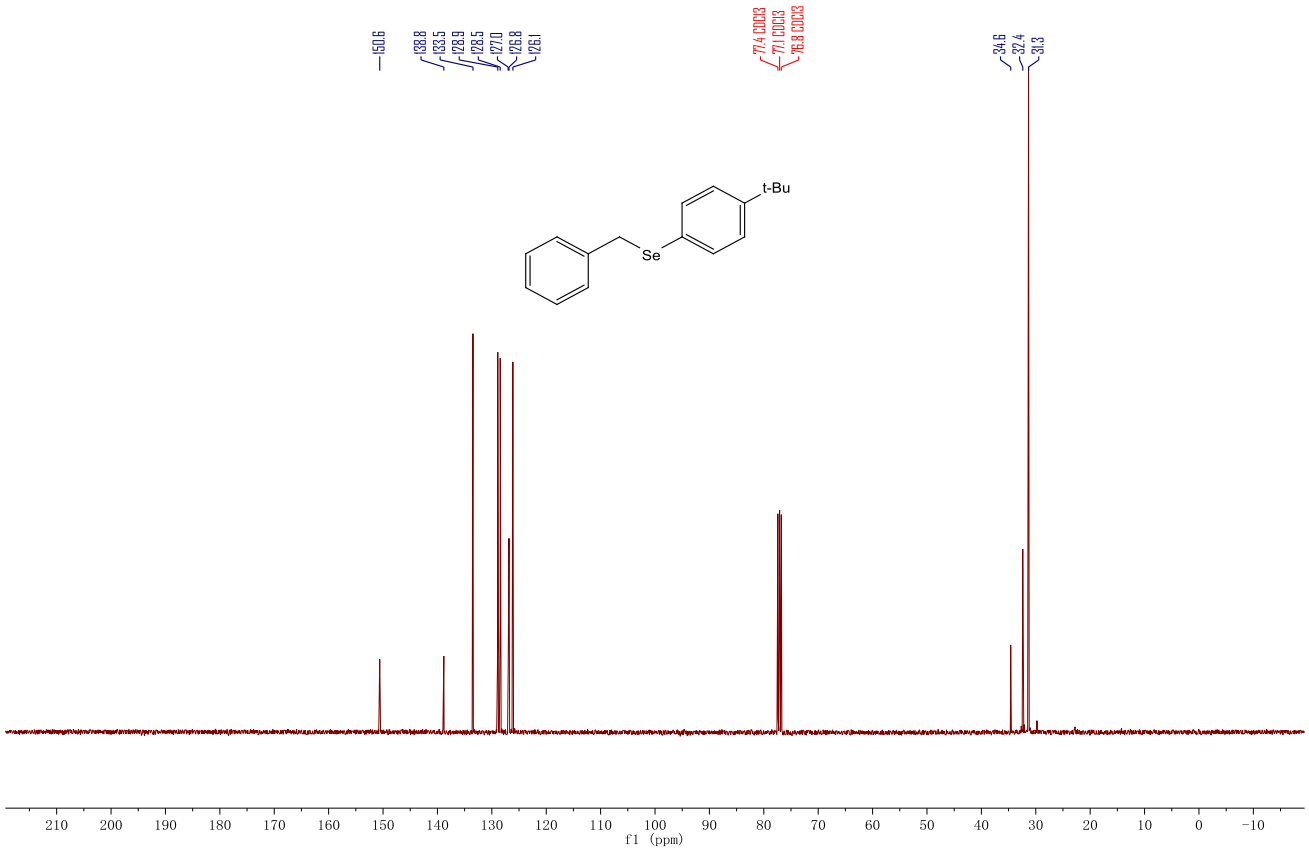
Product 3aj-Se



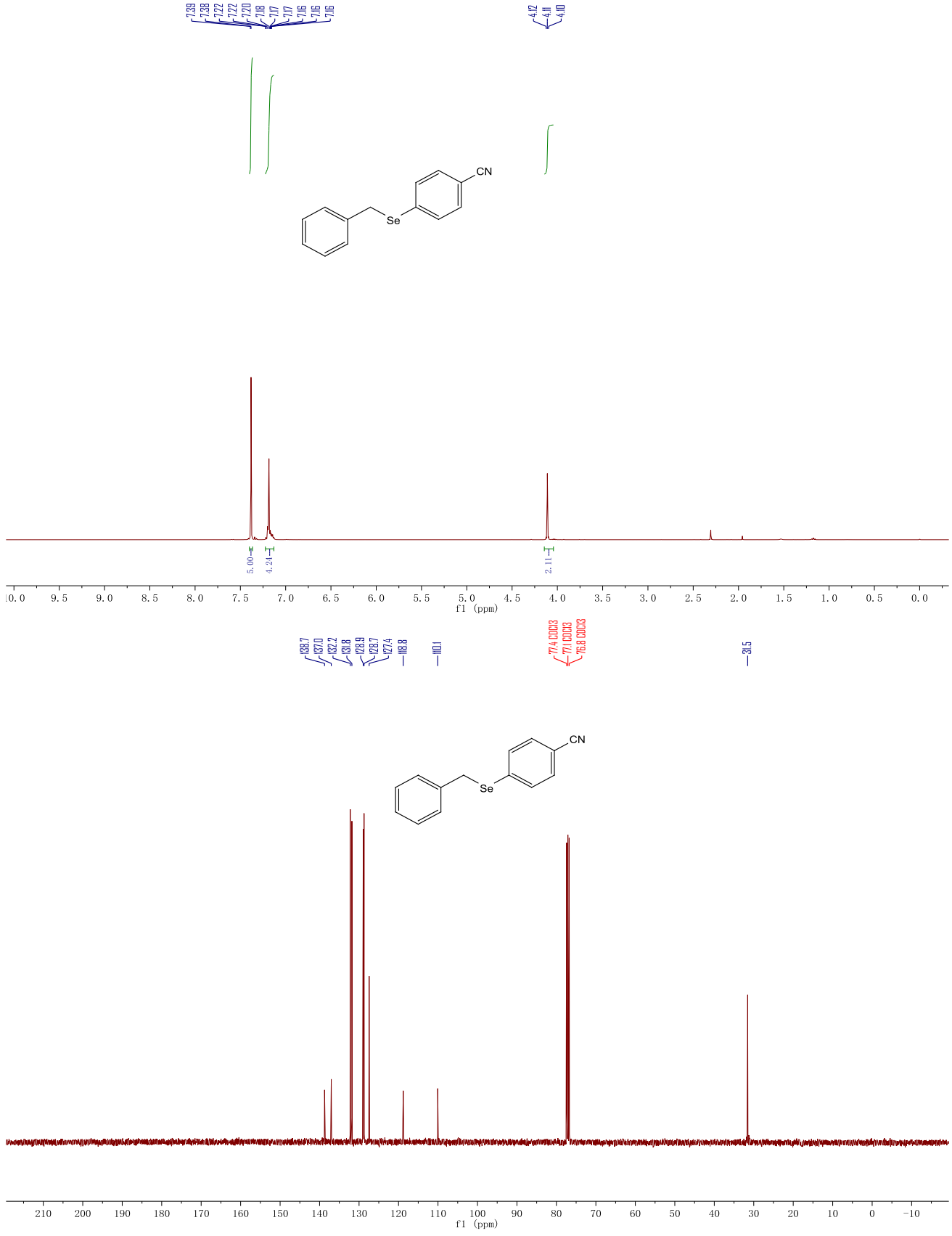


Product 3ak-Se

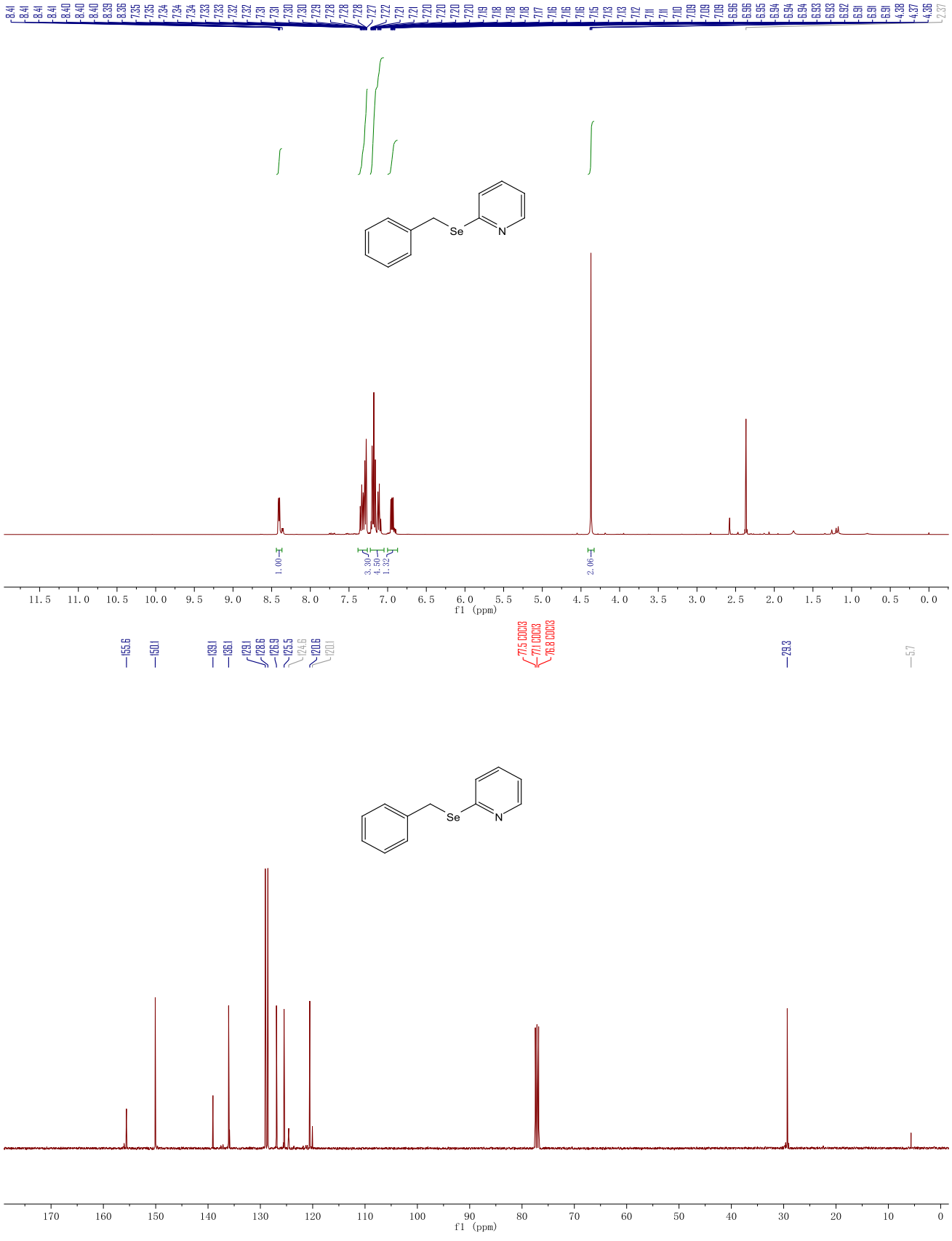




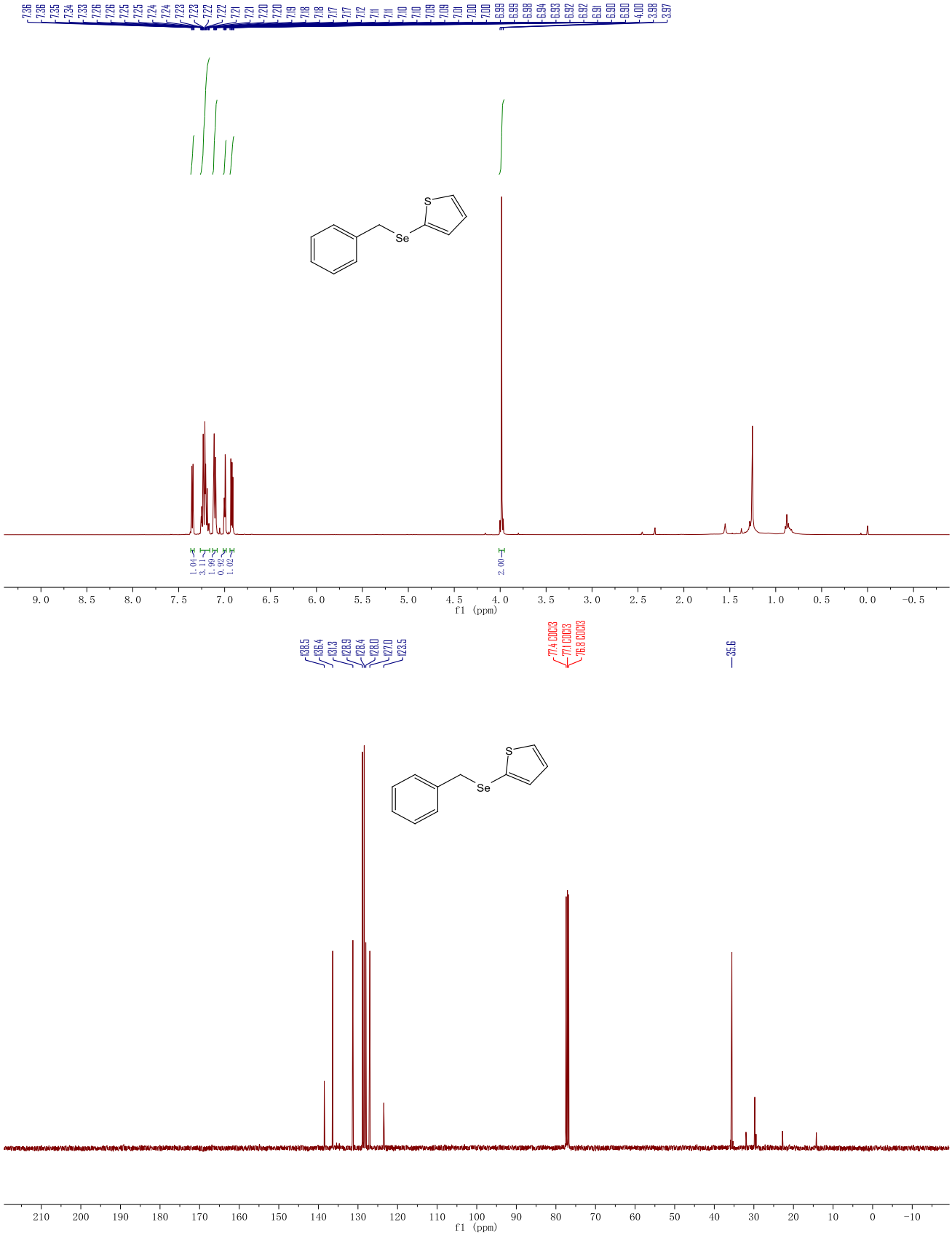
Product 3al-Se



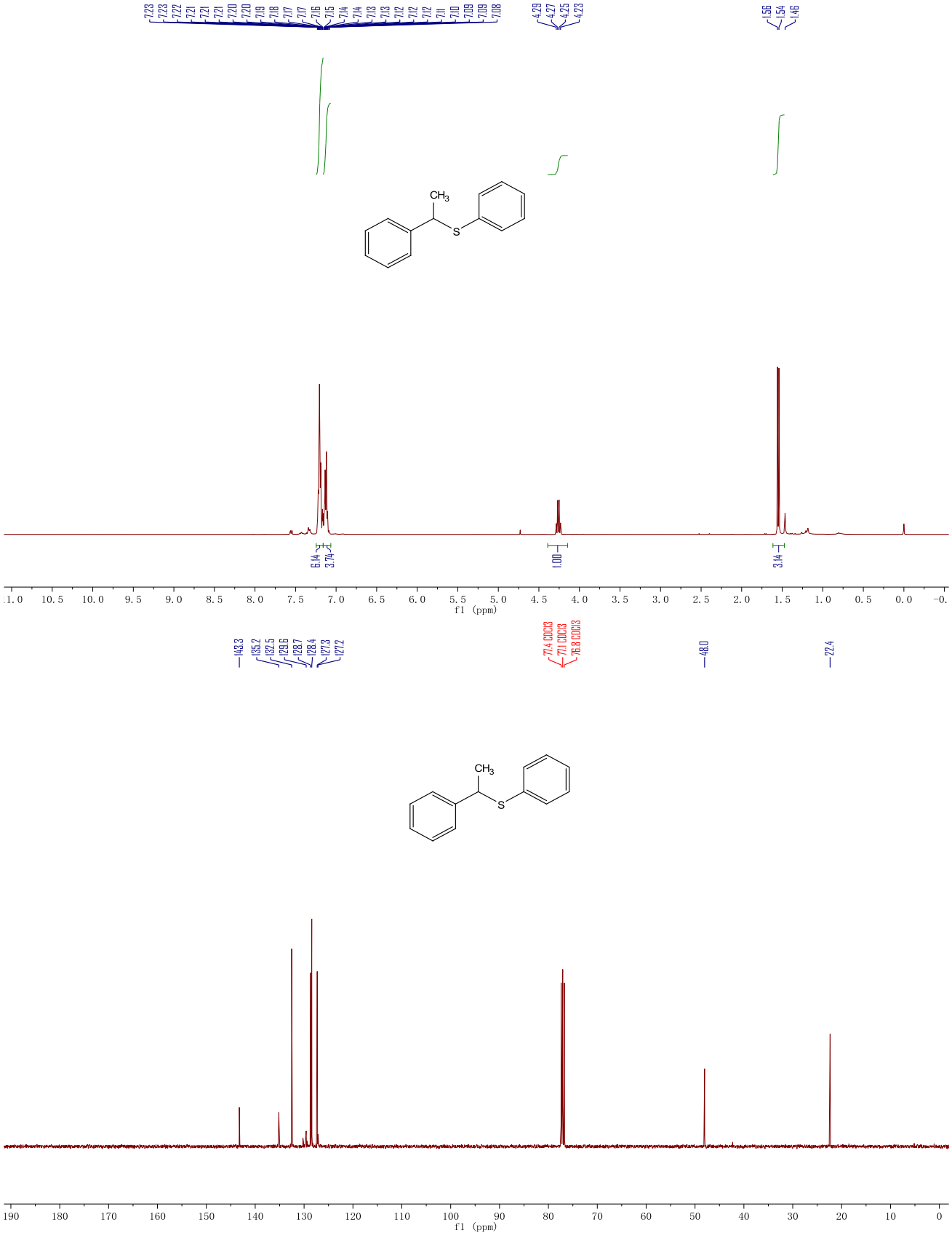
Product 3am-Se



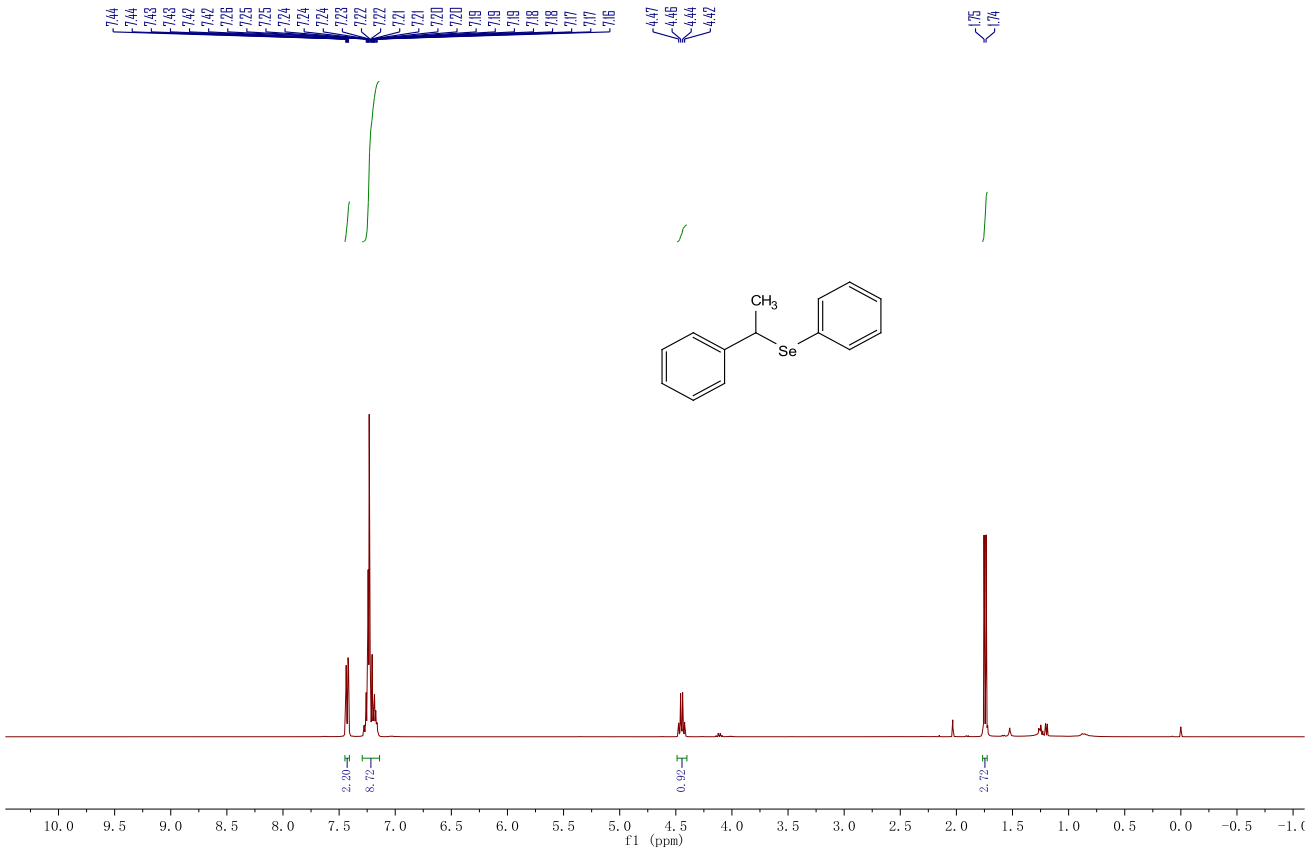
Product 3an-Se

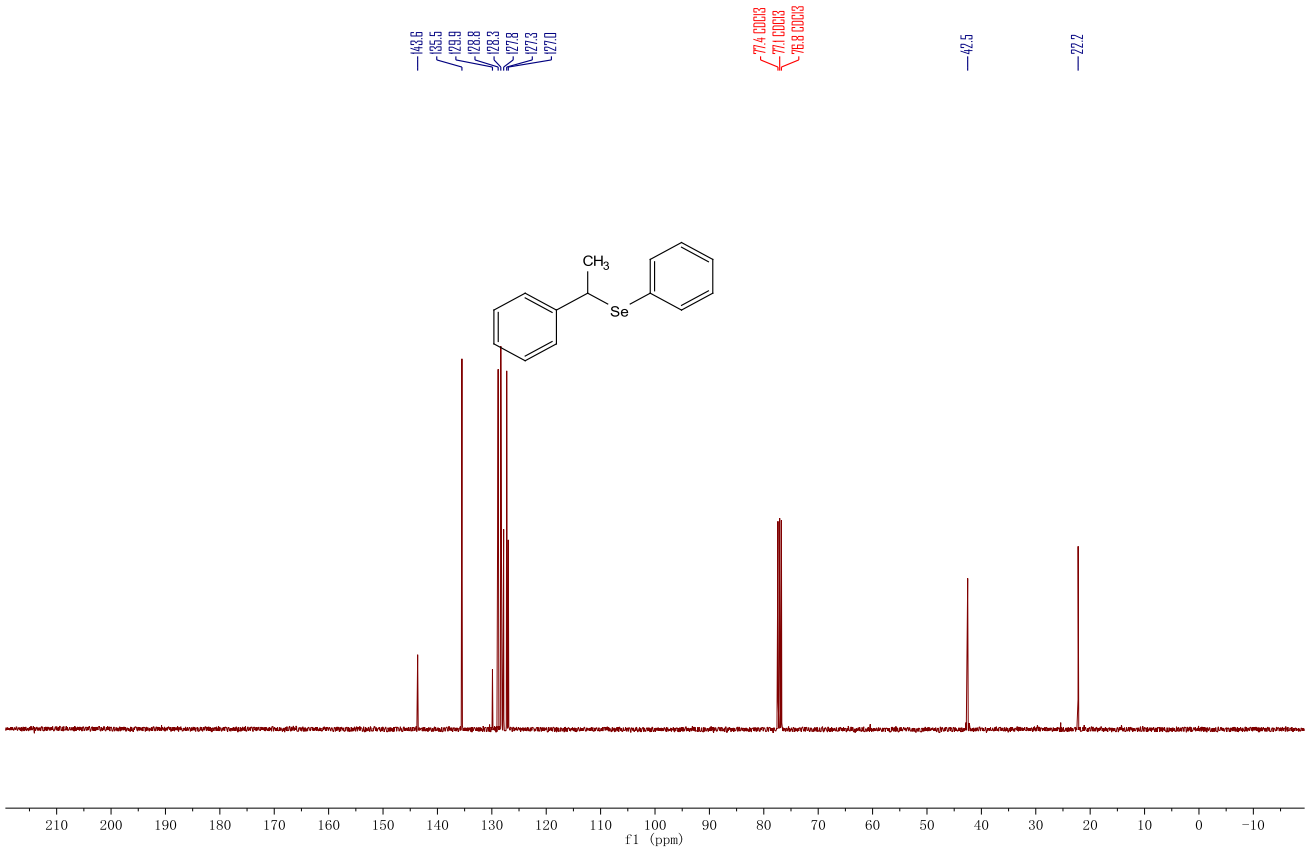


Product 5aa

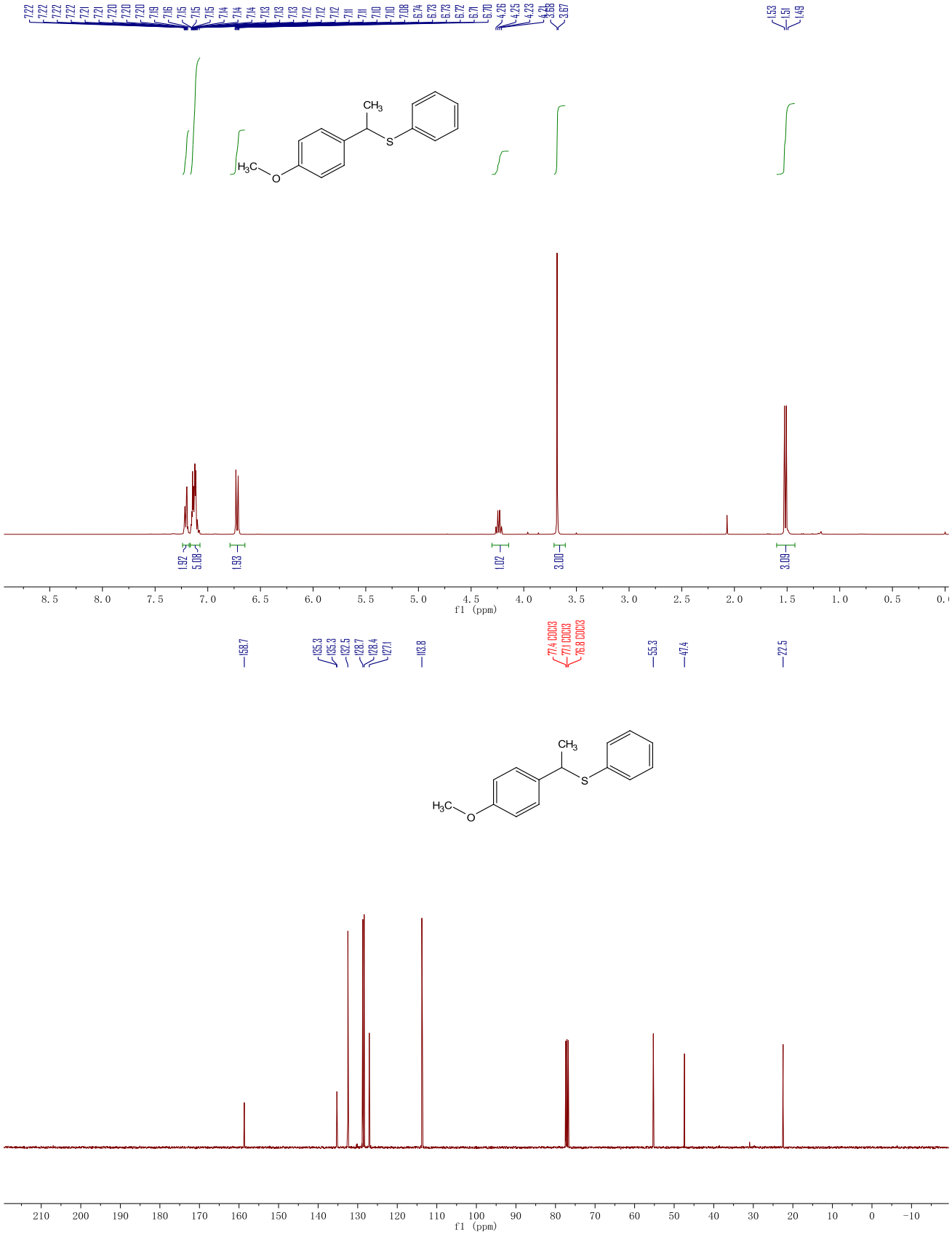

7.23
7.23
7.22
7.21
7.21
7.21
7.20
7.20
7.19
7.18
7.17
7.17
7.16
7.15
7.14
7.14
7.13
7.13
7.12
7.12
7.12
7.11
7.10
7.09
7.09
7.08
4.29
4.27
4.25
4.23
1.56
1.54
1.46
CH3
S
6.14
3.74
1.00
3.14
f1 (ppm)
143.3
135.2
132.5
129.6
128.7
128.4
127.3
127.2
77.4 CDCl3
77.1 CDCl3
76.8 CDCl3
48.0
22.4
CH3
S
f1 (ppm)

Product 5ab

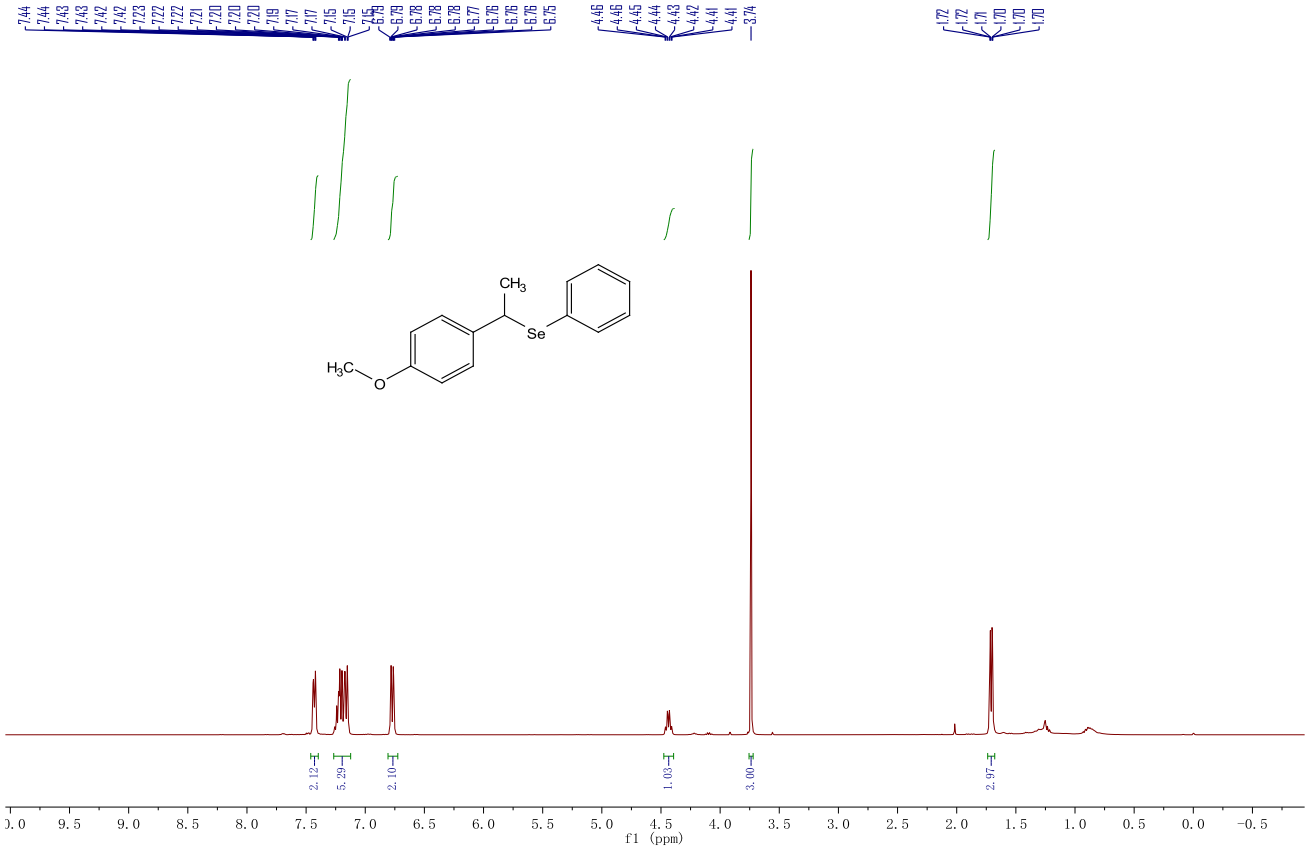

7.44
7.44
7.43
7.43
7.42
7.42
7.26
7.25
7.25
7.24
7.24
7.24
7.24
7.23
7.22
7.22
7.21
7.21
7.20
7.20
7.19
7.19
7.19
7.18
7.18
7.17
7.17
7.16
4.47
4.46
4.44
4.42
1.75
1.74
CH3
Se
2.20
8.72
0.92
2.72
f1 (ppm)

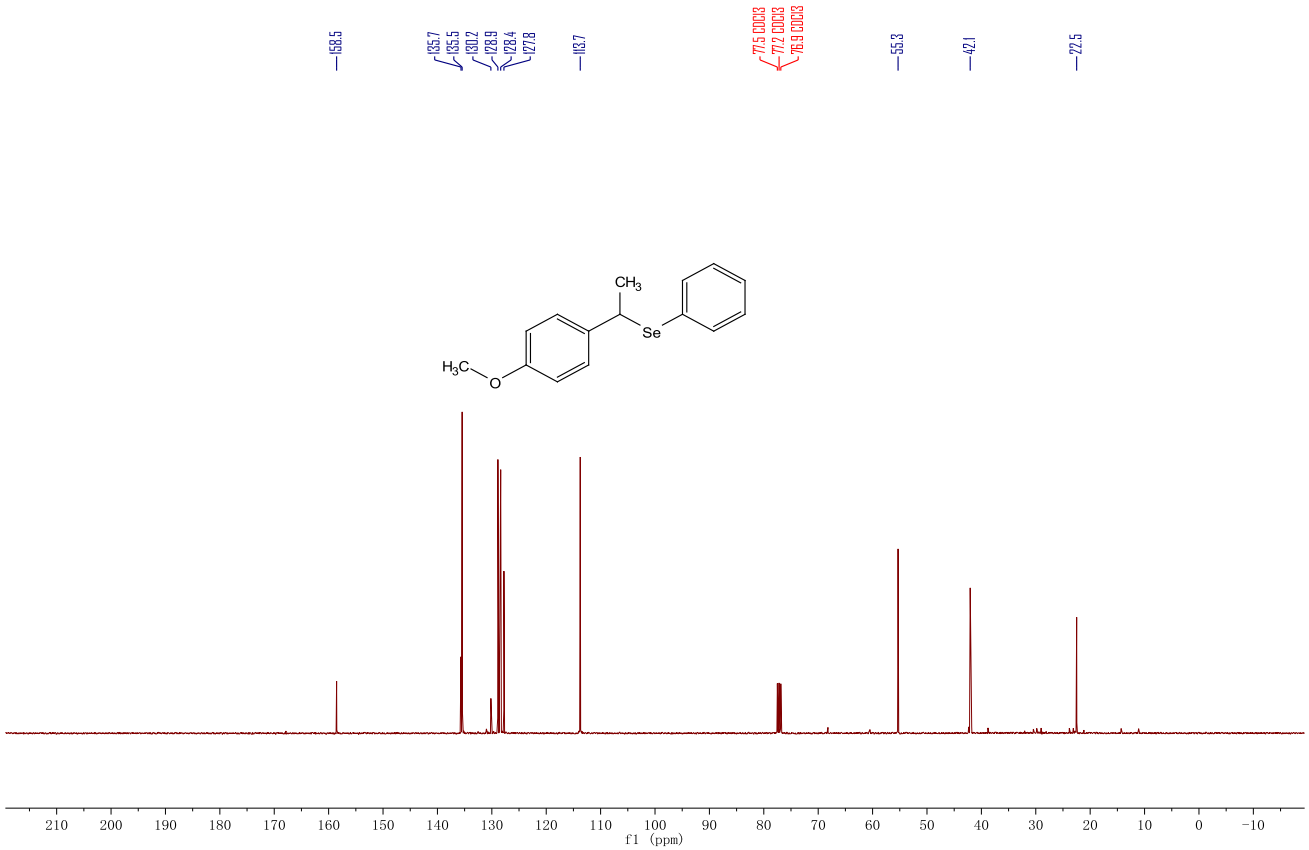

143.6
135.5
129.9
128.8
128.3
127.8
127.3
127.0
77.4 CDCl3
77.1 CDCl3
76.8 CDCl3
42.5
22.2
CH3
Se
f1 (ppm)

Product 5ba

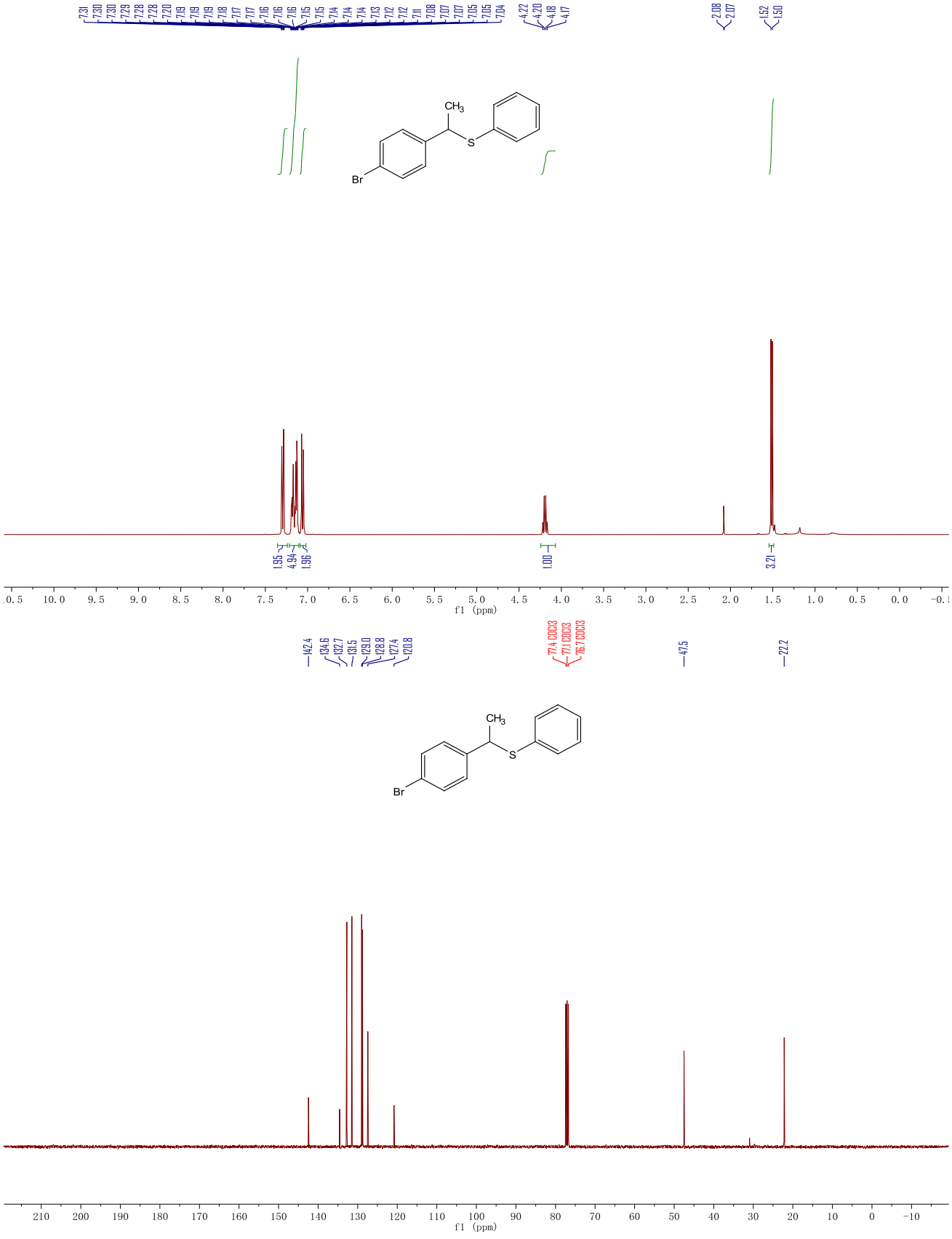

7.22
7.22
7.22
7.22
7.21
7.21
7.20
7.20
7.20
7.19
7.16
7.15
7.15
7.15
7.14
7.14
7.14
7.13
7.13
7.13
7.12
7.12
7.12
7.11
7.11
7.10
7.10
7.08
6.74
6.73
6.73
6.72
6.71
6.70
4.26
4.25
4.23
3.68
3.67
1.53
1.51
1.49
CH3
S
H3C
O
1.92
5.08
1.93
1.02
3.00
3.09
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
158.7
135.3
135.3
132.5
128.7
128.4
127.1
113.8
77.4 CDCl3
77.1 CDCl3
76.8 CDCl3
55.3
47.4
22.5
CH3
S
H3C
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

Product 5bb

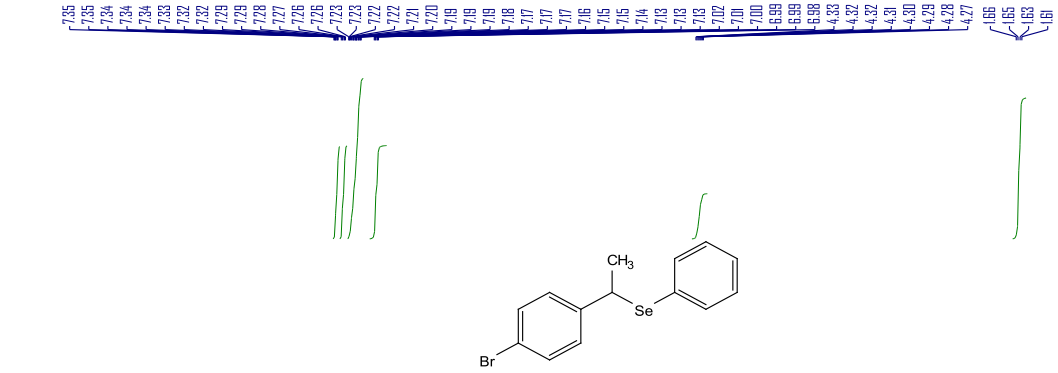

7.44
7.44
7.43
7.43
7.42
7.42
7.23
7.22
7.22
7.21
7.20
7.20
7.20
7.19
7.17
7.17
7.15
7.15
6.79
6.78
6.78
6.78
6.77
6.76
6.76
6.76
6.75
4.46
4.46
4.45
4.44
4.43
4.42
4.41
4.41
3.74
1.72
1.72
1.71
1.70
1.70
1.70
CH3
Se
H3C
O
2.12
5.29
2.10
1.03
3.00
2.97
f1 (ppm)


158.5
135.7
135.5
130.2
128.9
128.4
127.8
113.7
77.5 CDCl3
77.2 CDCl3
76.9 CDCl3
55.3
42.1
22.5
CH3
Se
H3C
O
f1 (ppm)

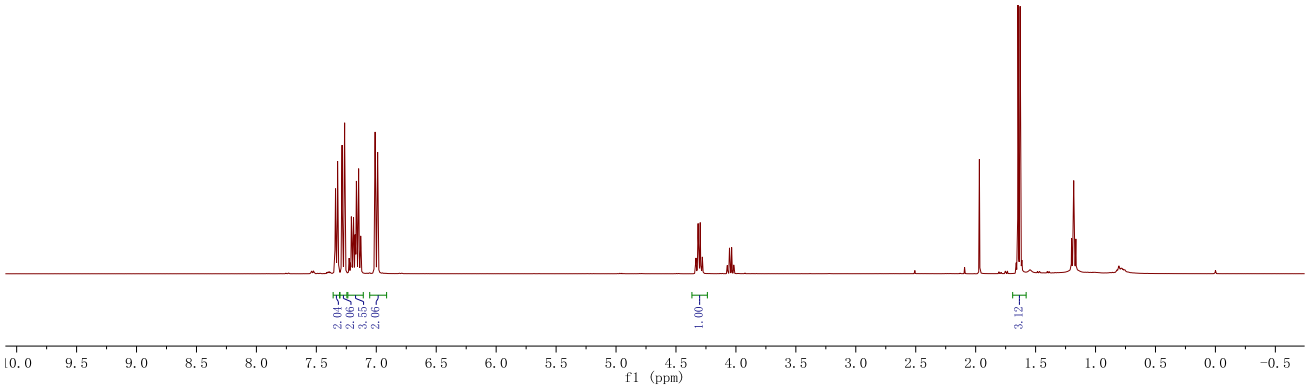
Product 5ca


7.31
7.30
7.30
7.29
7.28
7.28
7.20
7.19
7.19
7.19
7.18
7.17
7.17
7.16
7.16
7.16
7.15
7.15
7.14
7.14
7.14
7.13
7.12
7.12
7.11
7.08
7.07
7.07
7.05
7.05
7.04
4.22
4.20
4.18
4.17
2.08
2.07
1.52
1.50
CH3
S
Br
1.95
4.94
1.96
1.00
3.21
f1 (ppm)
142.4
134.6
132.7
131.5
129.0
128.8
127.4
120.8
77.4 CDCl3
77.1 CDCl3
76.7 CDCl3
47.5
22.2
CH3
S
Br
f1 (ppm)

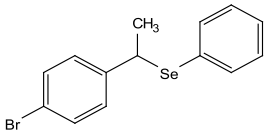
Product 5cb

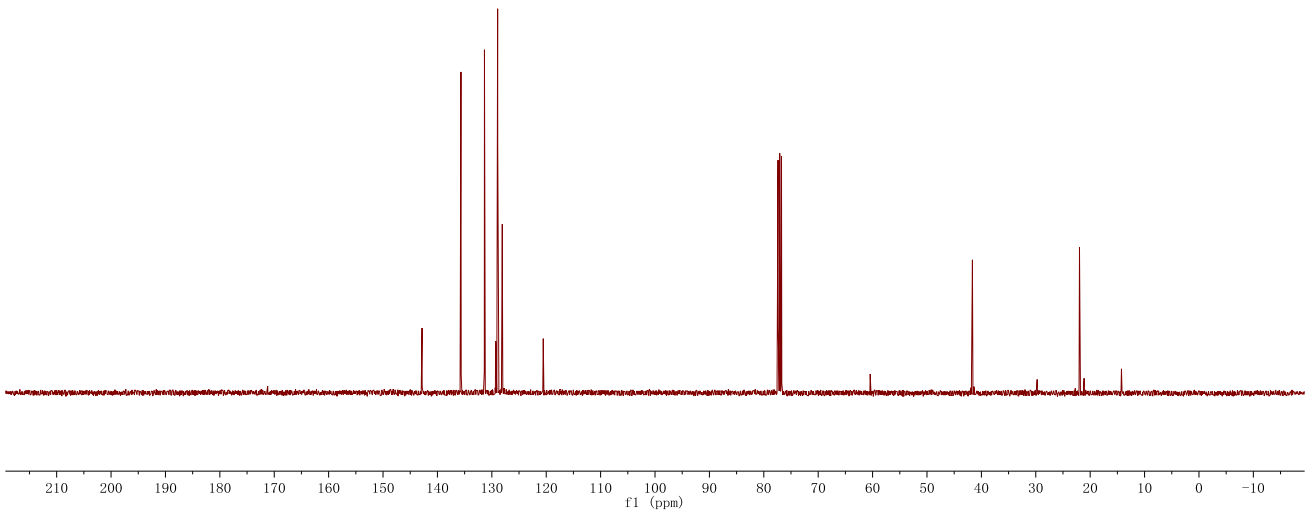

7.35
7.35
7.34
7.34
7.34
7.33
7.32
7.32
7.29
7.29
7.28
7.27
7.26
7.26
7.23
7.23
7.22
7.22
7.21
7.20
7.19
7.19
7.19
7.18
7.17
7.17
7.17
7.16
7.15
7.15
7.14
7.13
7.13
7.13
7.02
7.01
7.00
6.99
6.99
6.98
4.33
4.32
4.32
4.31
4.30
4.29
4.28
4.27
1.66
1.65
1.63
1.61

CH3
Se
Br

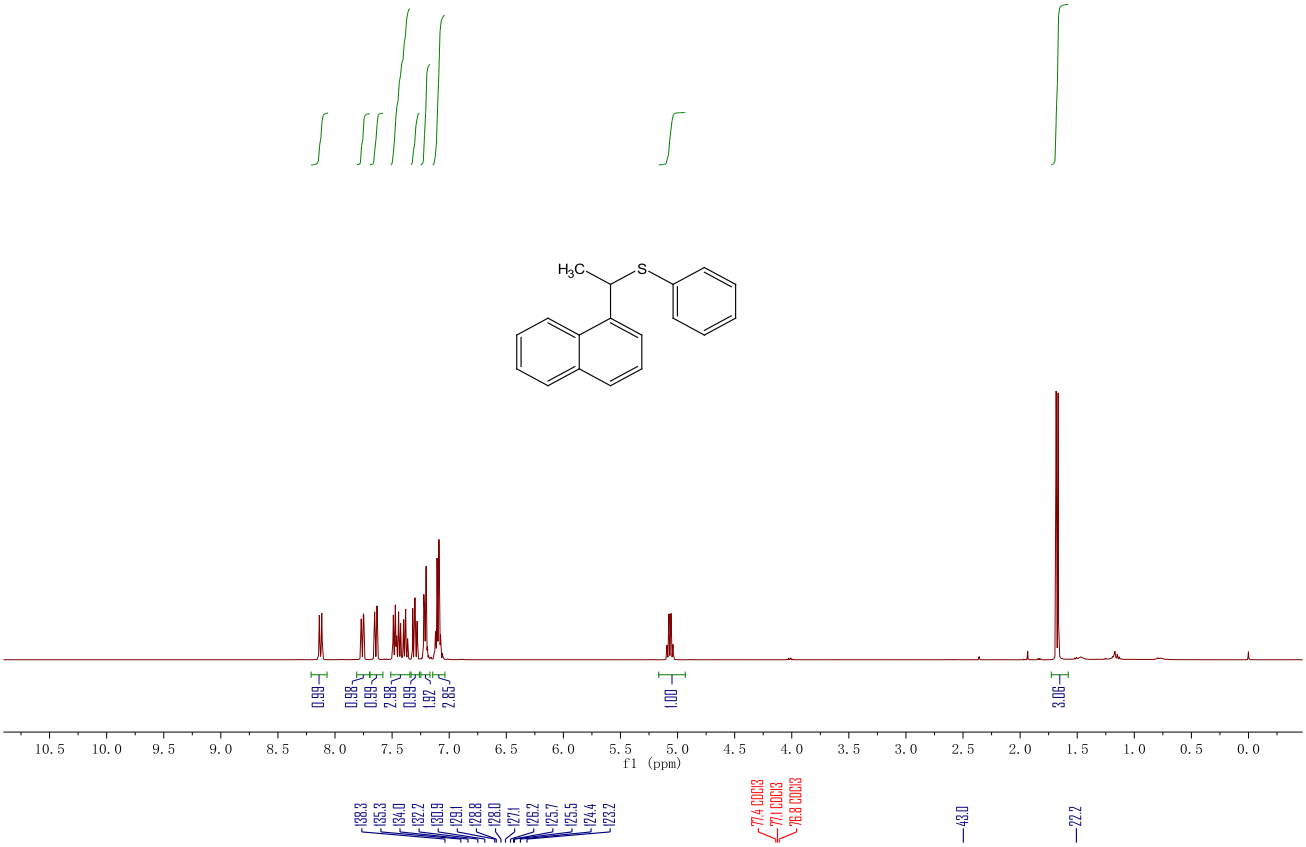

2.04
2.06
3.55
2.06
1.00
3.12
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)


142.8
135.7
131.4
129.3
128.9
128.9
128.1
120.5
77.4 CDCl3
77.1 CDCl3
76.7 CDCl3
41.6
22.0

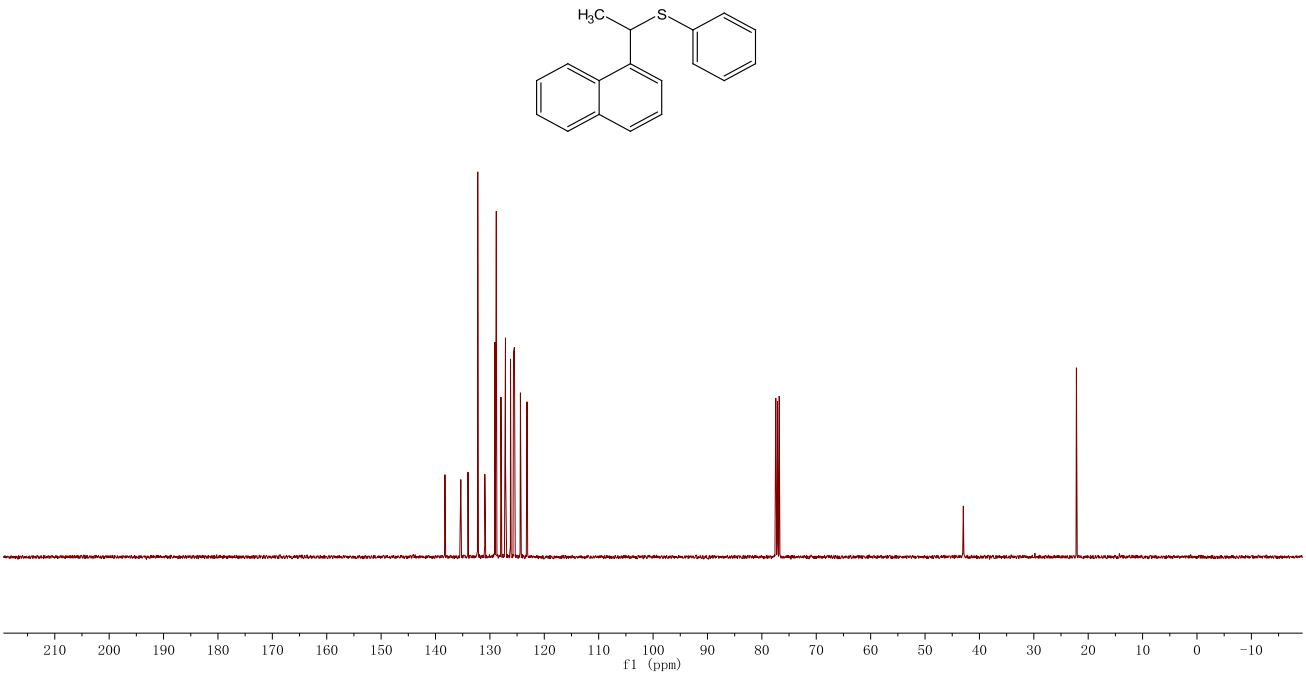

CH3
Se
Br


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

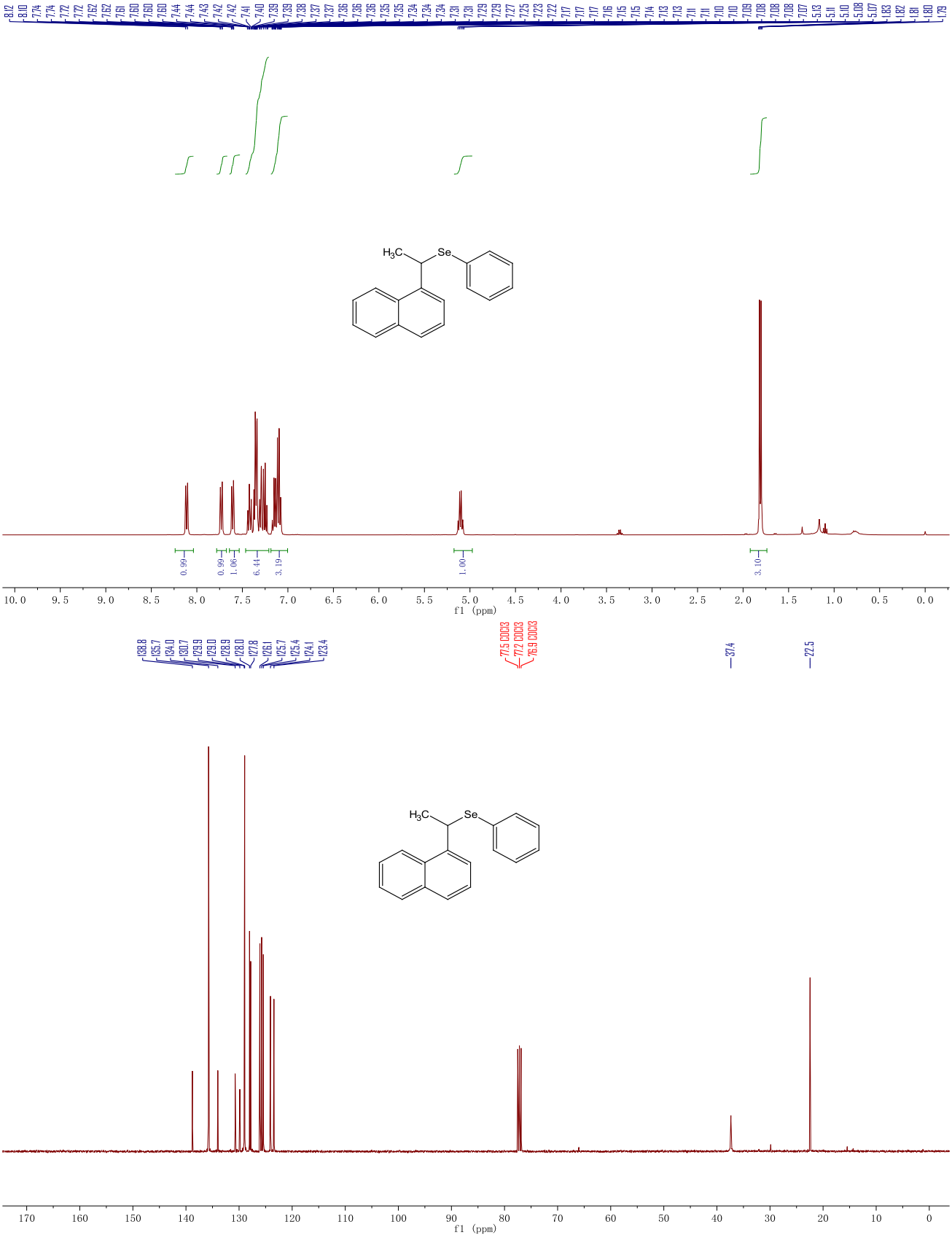
Product 5da


H3C
S
0.99
0.98
0.99
2.08
0.99
1.92
2.85
1.00
3.06
f1 (ppm)

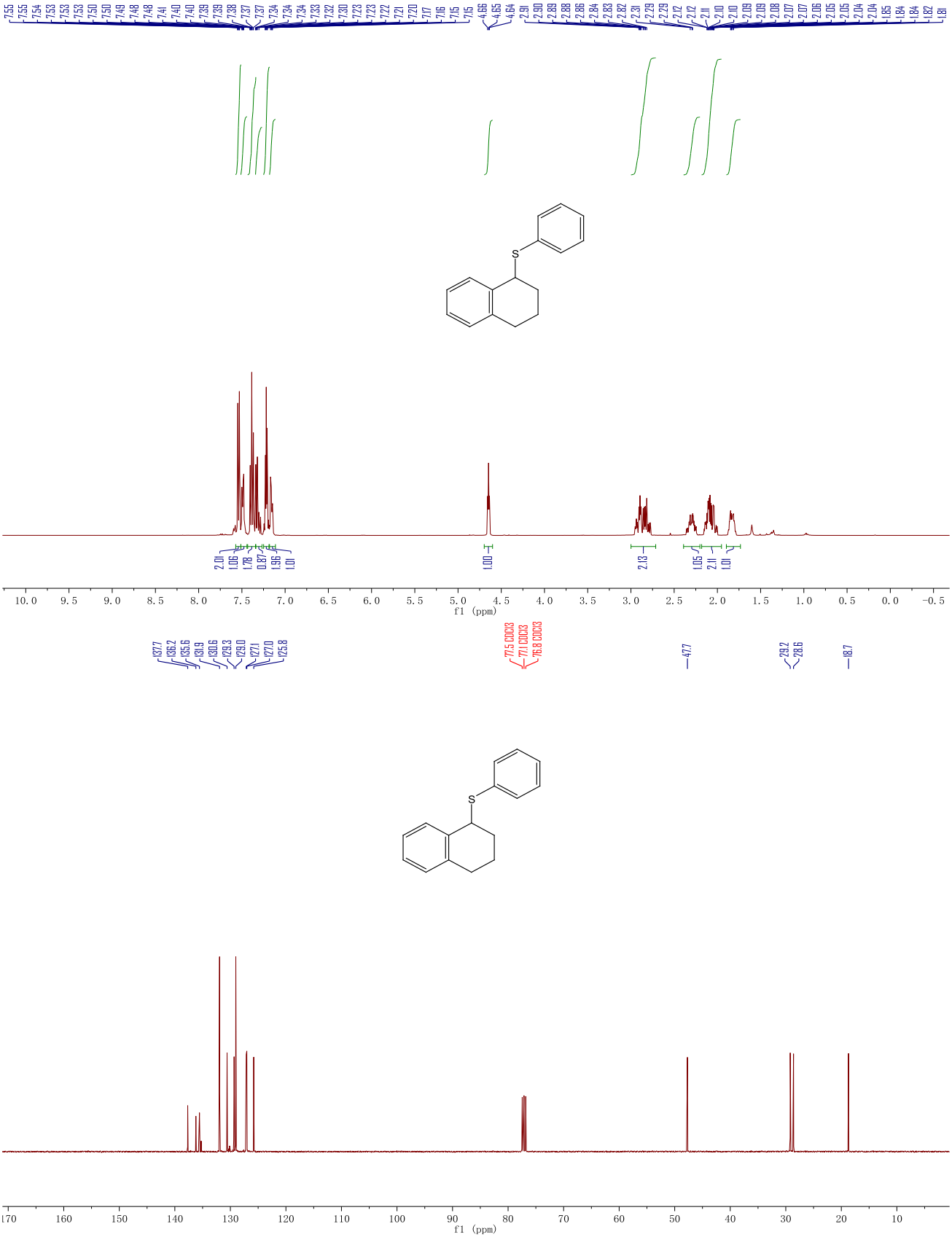
138.3
135.3
134.0
132.2
130.9
129.1
128.8
128.0
127.1
126.2
125.7
125.5
124.4
123.2
77.4 CDCl3
77.1 CDCl3
76.8 CDCl3
43.0
22.2


H3C
S
f1 (ppm)

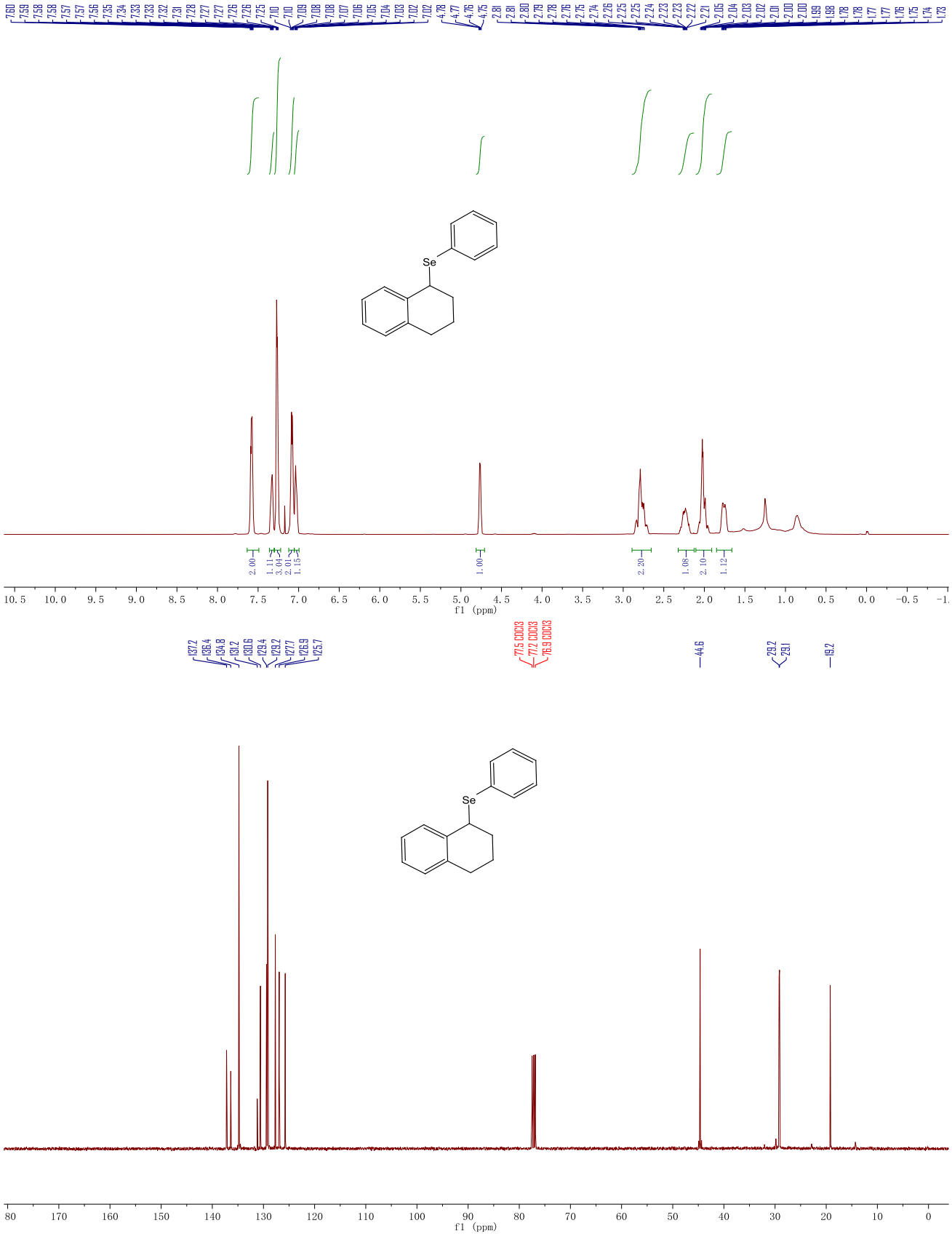
Product 5db



Product 5ea

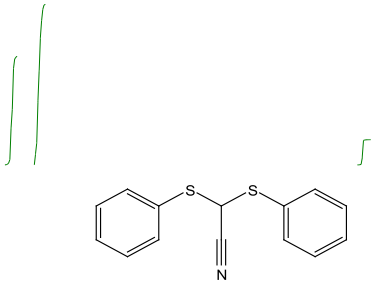

7.55 7.55 7.54 7.53 7.53 7.53 7.50 7.50 7.49 7.48 7.48 7.41 7.40 7.40 7.39 7.39 7.38 7.37 7.37 7.34 7.34 7.34 7.33 7.32 7.30 7.23 7.23 7.22 7.21 7.20 7.17 7.16 7.15 7.15 4.66 4.65 4.64 2.91 2.90 2.89 2.88 2.86 2.84 2.83 2.82 2.31 2.29 2.29 2.12 2.12 2.11 2.10 2.10 2.09 2.09 2.08 2.08 2.07 2.07 2.06 2.05 2.05 2.04 2.04 1.85 1.84 1.84 1.82 1.81
S
2.01 1.06 1.78 0.87 1.96 1.01 1.00 2.13 1.05 2.11 1.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)
137.7 135.2 135.6 131.9 130.6 129.3 129.0 127.1 127.0 125.8
77.5 CDCl3 77.1 CDCl3 76.8 CDCl3
47.7
29.2 28.6
18.7
S
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

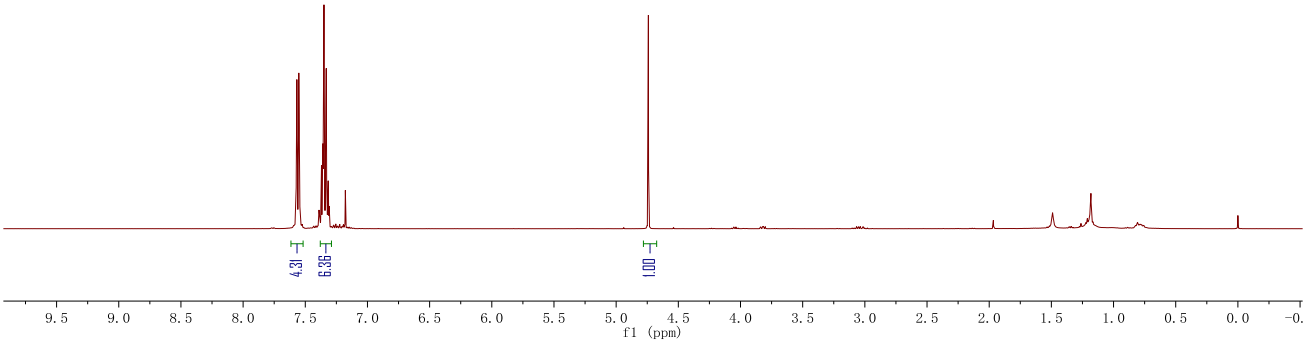
Product 5eb



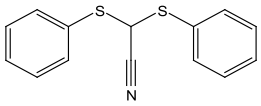
Compound 6

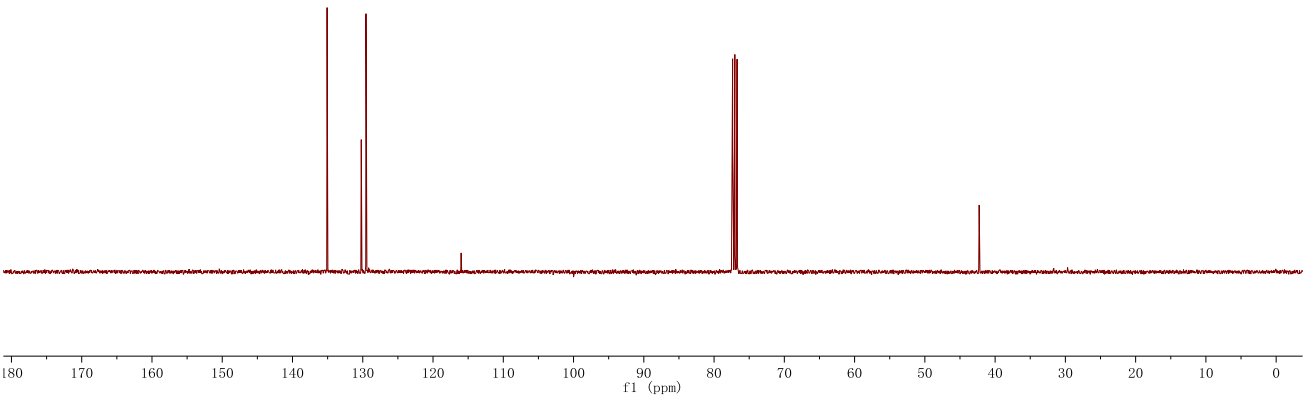

7.59
7.59
7.59
7.58
7.57
7.57
7.56
7.56
7.56
7.55
7.55
7.54
7.54
7.54
7.53
7.53
7.53
7.39
7.39
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.35
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.32
7.32
7.31
7.31
4.74
4.74


S
S
N

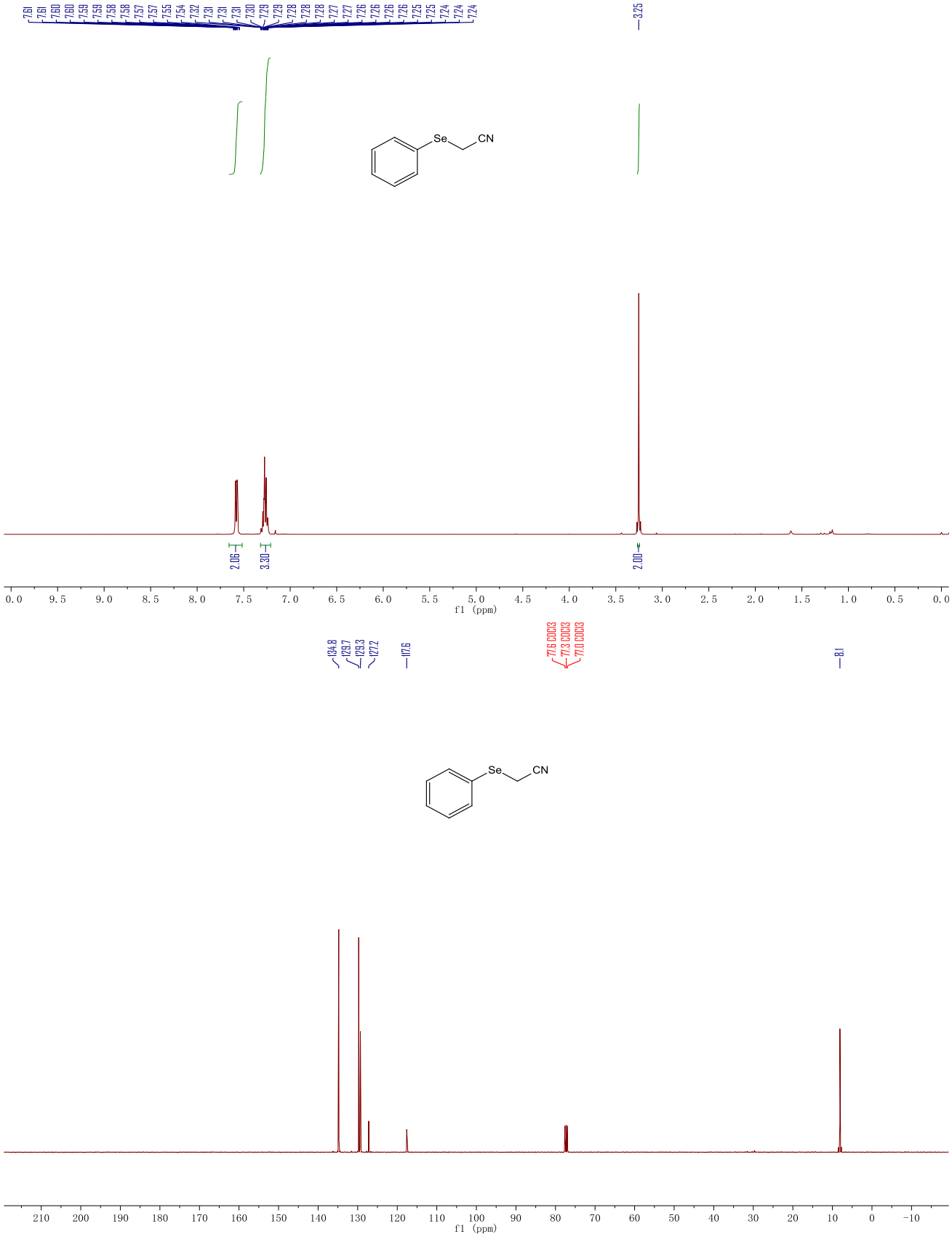

4.31
6.36
1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


135.1
130.2
130.1
129.5
116.0
77.4 CDCl3
77.0 CDCl3
76.7 CDCl3
42.3


S
S
N


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Compound 7


7.61
7.61
7.60
7.60
7.60
7.59
7.59
7.58
7.58
7.57
7.57
7.55
7.54
7.32
7.31
7.31
7.31
7.30
7.29
7.29
7.28
7.28
7.28
7.28
7.27
7.27
7.26
7.26
7.26
7.26
7.26
7.25
7.25
7.24
7.24
7.24
3.25
Se
CN
2.06
3.30
2.00
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
134.8
129.7
129.3
127.2
117.6
77.6 CDCl3
77.3 CDCl3
77.0 CDCl3
8.1
Se
CN
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)