

SUPPORTING INFORMATION

Blue-Light-Promoted Radical Sulfoximido-Chalcogenization of Aliphatic and Aromatic Alkenes

Hongyi Chen,^a Li Chen,^a Ze He,^a and Qingle Zeng^{a,*}

[†] State Key Laboratory of Geohazard Prevention and Geoenvironment Protection,
Chengdu University of Technology, College of Materials, Chemistry & Chemical
Engineering, Chengdu 610059, China
qinglezeng@hotmail.com, qlzeng@cdut.edu.cn

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General information

All radical sulfoximido-chalcogenization of aliphatic and aromatic alkenes reactions were carried out under air atmosphere in dried glassware and monitored by thin-layer chromatography (TLC). The glassware used was dried in an electric oven at 120 °C. Chemicals were purchased from Aladdin, Adamas, Aldrich, Alfa Aesar, and Kelong Chemical Co. and used as received. 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. The light irradiation was performed by 30 W blue LED. Photochemical reaction instrument (Phchem II) is produced by Beijing newbit technology co., LTD. ¹H NMR spectra were determined on a Bruker Avance III 400 MHz instrument or on an Agilent Technologies 400 MHz instrument. ¹H NMR data are reported in δ units (ppm), and were measured relative to the signals for residual chloroform (7.26 ppm), DMSO (2.50 ppm) or acetone (2.05 ppm) in the deuterated solvent, unless otherwise stated. ¹³C NMR spectra are reported in (ppm) relative to deuteriochloroform (77.2 ppm), DMSO-d₆ (39.5 ppm) or acetone-d₆ (206.7 ppm for C=O) unless otherwise stated, and all were obtained with ¹H decoupling. High-resolution mass spectra are recorded on an LCMS-IT-TOF instrument.

General procedure of synthesis of NH-sulfoximines

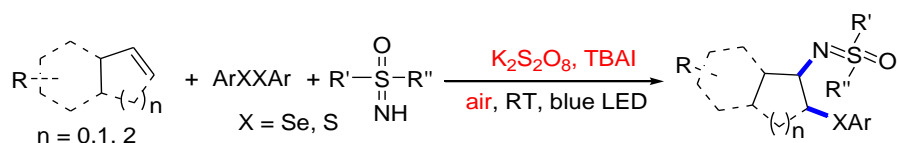
In a dried schlenk tube, sulfide (0.5 mmol), (diacetoxyiodo)benzene (1.25 mmol, 2.5 equiv) and ammonium carbamate (1.0 mmol, 2.0 equiv) were added to a flask equipped with a stir bar. MeOH (1 mL, 0.5 M) was added and the reaction was stirred at 25 °C for 3-10 h. The reaction was then quenched by the addition of saturated sodium bicarbonate (5 mL), the aqueous phase was extracted with ethyl acetate (10 mL \times 3). The combined organic phase was dried over anhydrous MgSO₄, filtered and concentrated. The resulting residual was purified by flash silica gel column chromatography using mixture of petroleum ether and ethyl acetate as eluent to afford the NH-sulfoximines.

General procedure of synthesis of diaryl diselenide

Diselenides were prepared according to literature methods. To a stirred solution of selenium powder (10 mmol) and aryl iodide (4 mmol) in DMSO (10 mL) was added CuO nanoparticles (10 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 Sulfoximido-Chalcogenization of Aliphatic and Aromatic Alkenes



To an oven-dried 25 mL test tube with standard ground joint equipped with a stir bar were added alkenes (1.2 mmol), disulfide or diselenide (0.5 mmol), NH-sulfoximines (1.2 mmol), TBAI (0.5 mol%), $\text{K}_2\text{S}_2\text{O}_8$ (1.5 mol%) and EtOAc (3.0 mL), at room temperature under an air atmosphere irradiated by a 30 W blue LED for 20 hours. After

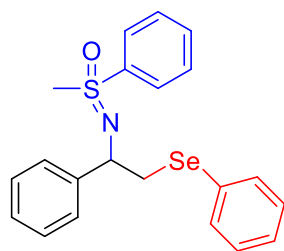
completion of the reaction as indicated by TLC, the reaction was then quenched by the addition of saturated $\text{Na}_2\text{S}_2\text{O}_3$ solution (5 mL). The color of the reaction system changed from reddish brown to yellow. The reaction mixture was extracted with ethyl acetate (15 mL \times 3). 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 (5:1-2:1) to afford the desired products as a yellow or colorless oil.



Fig. S1. Reaction Setup

Characterization Data of New Compounds

Methyl(phenyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanone (4a)



Yellow oil, 377.7 mg, 91% yield. dr=1:1.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 7.95 – 7.90 (m, 2H), 7.65 – 7.60 (m, 2H), 7.58 – 7.51 (m, 1H), 7.50 – 7.39 (m, 3H), 7.34 – 7.07 (m, 22H), 4.28 (q, $J = 6.9$ Hz, 2H), 3.43 (dd, $J = 11.8, 6.7$ Hz, 1H), 3.28 (ddd, $J = 14.4, 11.9,$

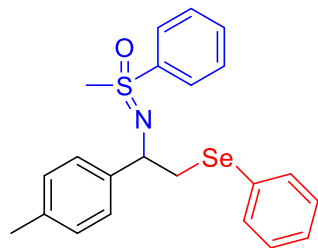
7.8 Hz, 2H), 3.11 (dd, $J = 11.9, 6.0$ Hz, 1H), 3.05 (s, 3H), 2.97 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 145.05 and 143.94, 139.76 and 139.43, 132.96 and 132.76, 132.07 and 132.01, 131.26 and 131.14, 129.30 and 129.06, 128.92 and 128.89, 128.67 and 128.57, 128.34 and 128.16, 127.22 and 127.08, 127.04 and 126.77, 126.41 and 126.32, 58.92 and 58.78, 45.19 and 45.04, 38.78 and 38.42.

IR (neat, cm^{-1}): 3057, 3026, 2927, 2359, 1578, 1477, 1445, 1233, 1131, 1084, 1070, 1022, 978, 741, 690.

HRMS (ESI) m/z $[M + \text{Na}]^+$ Calcd for $\text{C}_{21}\text{H}_{21}\text{NNaOSSe}^+$ 438.0401, found 438.0382.

Methyl(phenyl)((2-(phenylselanyl)-1-(p-tolyl)ethyl)imino)- λ^6 -sulfanone (4b)



Yellow oil, 386.1 mg, 90% yield. dr=1:1.1.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.02 (ddd, $J = 8.6, 3.2, 1.3$ Hz, 2H), 7.76 – 7.71 (m, 2H), 7.66 – 7.60 (m, 1H), 7.59 – 7.50 (m, 3H), 7.44 – 7.36 (m, 6H), 7.28 (d, $J = 1.8$ Hz, 1H), 7.22 – 7.16 (m, 8H), 7.14 –

7.11 (m, 2H), 7.02 (ddt, $J = 15.0, 8.0, 4.3$ Hz, 3H), 4.35 (tdd, $J = 5.7, 3.7, 1.9$ Hz, 2H), 3.51 (ddd, $J = 11.7, 6.6, 1.6$ Hz, 1H), 3.37 (dddd, $J = 11.8, 9.5, 7.8, 2.3$ Hz, 2H), 3.20 (ddd, $J = 11.8, 6.1, 1.3$ Hz, 1H), 3.12 (d, $J = 3.8$ Hz, 3H), 3.05 (d, $J = 3.2$ Hz, 3H), 2.35 –

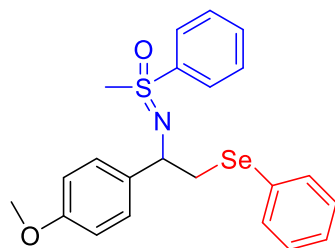
2.26 (m, 6H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 144.92 and 143.70, 142.10 and 140.96, 137.79 and 136.72, 132.93 and 132.74, 131.98 and 131.93, 129.29 and 129.23, 129.07 and 129.01, 128.90 and 128.87, 128.63 and 128.60, 126.89 and 126.62, 126.34 and 126.25, 124.15 and 123.86, 58.91 and 58.69, 45.19 and 45.02, 38.76 and 38.36, 21.59 and 21.22.

IR (neat, cm^{-1}): 3054, 3018, 2924, 2855, 1578, 1477, 1445, 1232, 1131, 1084, 1021, 978, 786, 740, 690.

HRMS (ESI) m/z $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{22}\text{H}_{23}\text{NNaOSSe}^+$ 452.0558, found 452.0535.

((1-(4-methoxyphenyl)-2-(phenylselanyl)ethyl)imino)(methyl)(phenyl)- λ^6 -sulfanone (4c)



Yellow oil, 441.4 mg, 97% yield. dr=1:1.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 7.93 – 7.89 (m, 2H), 7.65 – 7.61 (m, 2H), 7.56 – 7.41 (m, 5H), 7.34 – 7.25 (m, 6H), 7.22 – 7.17 (m, 3H), 7.11 – 7.02 (m, 8H), 6.77 – 6.73 (m, 2H), 6.68 – 6.63 (m, 2H), 4.25

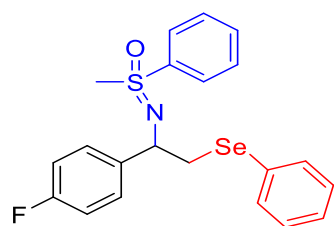
(ddd, $J = 7.9, 6.4, 4.3$ Hz, 2H), 3.70 (d, $J = 6.4$ Hz, 6H), 3.40 (dd, $J = 11.7, 6.5$ Hz, 1H), 3.30 – 3.21 (m, 2H), 3.09 (dd, $J = 11.8, 6.2$ Hz, 1H), 3.01 (s, 3H), 2.95 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 158.67 and 158.60, 139.85 and 139.51, 137.21 and 136.18, 132.90 and 132.71, 132.04 and 131.99, 131.31 and 131.18, 129.28 and 129.05, 128.89 and 128.86, 128.64 and 128.59, 128.05 and 127.79, 126.35 and 126.27, 113.70 and 113.51, 58.37 and 58.25, 55.24 and 55.21, 45.20 and 45.03, 38.87 and 38.53.

IR (neat, cm^{-1}): 3056, 2998, 2928, 2833, 1609, 1578, 1509, 1477, 1444, 1243, 1174, 1130, 1023, 979, 829, 740, 690.

HRMS (ESI) m/z $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{22}\text{H}_{23}\text{NNaO}_2\text{SSe}^+$ 468.0507, found 468.0507.

((1-(4-fluorophenyl)-2-(phenylselanyl)ethyl)imino)(methyl)(phenyl)- λ^6 -sulfanone

(4d)

Yellow oil, 385.4 mg, 89% yield. dr=1:1.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.02 – 7.97 (m, 2H), 7.73 – 7.67 (m, 2H), 7.67 – 7.61 (m, 1H), 7.60 – 7.50 (m, 3H), 7.44 – 7.30 (m, 8H), 7.21 – 7.15 (m, 8H), 7.01 – 6.95 (m, 2H), 6.92 – 6.85 (m, 2H), 4.35 (ddd, $J = 16.0, 7.7, 6.4$ Hz, 2H), 3.49 (dd, $J = 11.9, 6.4$ Hz, 1H), 3.37 – 3.28 (m, 2H), 3.19 – 3.11 (m, 4H), 3.07 (s, 3H).

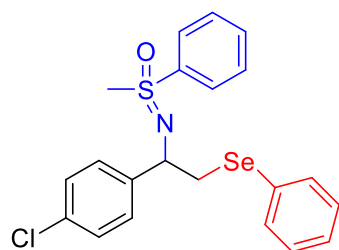
^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 163.08 (d, $J = 236.5$ Hz) and 160.65 (d, $J = 235.4$ Hz), 140.77 (d, $J = 3.0$ Hz) and 139.73 (d, $J = 3.1$ Hz), 133.01 and 132.81, 132.16 and 132.09, 131.03 and 130.88, 129.34 and 129.09, 128.92 and 128.89, 128.66 and 128.61, 128.53 and 128.47, 128.35 (d, $J = 8.5$ Hz) and 128.27 (d, $J = 8.5$ Hz), 126.49 and 126.41, 115.05 (d, $J = 21.4$ Hz) and 114.83 (d, $J = 21.4$ Hz), 58.28 and 58.08, 45.29 and 45.02, 38.77 and 38.48.

^{19}F NMR (376 MHz, Chloroform-d) δ -115.70, -115.76.

IR (neat, cm^{-1}): 3062, 3024, 3002, 2925, 2861, 1601, 1578, 1506, 1476, 1443, 1435, 1230, 1120, 1078, 1022, 981, 828, 737, 688.

HRMS (ESI) m/z $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{21}\text{H}_{20}\text{FNNaOSSe}^+$ 456.0307, found 456.0309.

((1-(4-chlorophenyl)-2-(phenylselanyl)ethyl)imino)(methyl)(phenyl)- λ^6 -sulfanone

(4e)

Yellow oil, 385.4 mg, 87% yield. dr=1:1.3.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.02 – 7.96 (m, 2H), 7.72 – 7.60 (m, 3H), 7.60 – 7.50 (m, 3H), 7.47 – 7.22 (m, 11H), 7.21 – 7.11 (m, 9H), 4.32 (ddd, $J = 18.3, 7.7, 6.3$ Hz, 2H), 3.47 (dd, $J = 11.9, 6.2$ Hz, 1H), 3.31 (ddd, $J = 14.2, 11.9, 7.7$ Hz, 2H), 3.19 – 3.10 (m, 4H), 3.07 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 143.55 and 142.43, 139.66 and 139.22, 133.05 and 132.86, 132.73 and 132.63, 132.19 and 132.12,

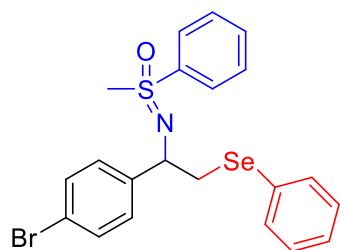
130.94 and 130.78, 129.37 and 129.13, 128.94 and 128.90, 128.68 and 128.66, 128.46 and 128.41, 128.20 and 128.19, 126.54 and 126.44, 58.34 and 58.17, 45.29 and 45.02, 38.57 and 38.28.

IR (neat, cm^{-1}): 3055, 3024, 3002, 2924, 2859, 1576, 1487, 1476, 1443, 1435, 1404, 1320, 1230, 1122, 1083, 983, 882, 818, 788, 736, 687.

HRMS (ESI) m/z $[M + H]^+$ Calcd for $\text{C}_{21}\text{H}_{21}\text{ClNOSse}^+$ 450.0192, found 450.0193.

((1-(4-bromophenyl)-2-(phenylselanyl)ethyl)imino)(methyl)(phenyl)- λ^6 -sulfanone

(4f)



Yellow oil, 468.3 mg, 95% yield. dr=1:1.3.

^1H NMR (400 MHz, Chloroform- d , mixture of two isomers) δ 7.92 – 7.86 (m, 2H), 7.62 – 7.52 (m, 3H), 7.51 – 7.42 (m, 3H), 7.35 – 7.18 (m, 10H), 7.15 (d, $J = 2.0$ Hz, 1H), 7.14 (d, $J = 2.0$ Hz, 1H), 7.09 (dt, $J = 5.3, 2.5$ Hz, 6H), 7.00 (d, $J =$

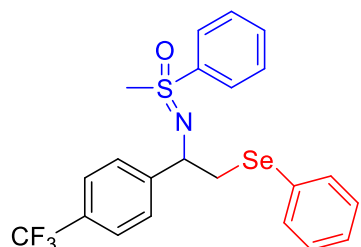
2.0 Hz, 1H), 6.98 (d, $J = 2.0$ Hz, 1H), 4.21 (ddd, $J = 18.3, 7.7, 6.2$ Hz, 2H), 3.37 (dd, $J = 11.9, 6.2$ Hz, 1H), 3.21 (ddd, $J = 13.8, 12.0, 7.6$ Hz, 2H), 3.08 – 3.00 (m, 4H), 2.98 (s, 3H).

^{13}C NMR (101 MHz, Chloroform- d , mixture of two isomers) δ 144.05 and 142.91, 139.63 and 139.20, 133.06 and 132.87, 132.21 and 132.13, 131.35 and 131.13, 130.91 and 130.74, 129.37 and 129.14, 128.94 and 128.90, 128.84 and 128.67, 128.58 and 128.47, 126.54 and 126.45, 120.93 and 120.81, 58.40 and 58.23, 45.29 and 45.01, 38.49 and 38.19.

IR (neat, cm^{-1}): 3067, 3025, 2925, 2858, 1575, 1476, 1443, 1434, 1402, 1321, 1229, 1122, 1067, 1005, 985, 881, 816, 789, 737, 692.

HRMS (ESI) m/z $[M + H]^+$ Calcd for $\text{C}_{21}\text{H}_{21}\text{BrNOSse}^+$ 493.9687, found 493.9696.

Methyl(phenyl)((2-(phenylselanyl)-1-(4-(trifluoromethyl)phenyl)ethyl)imino)- λ^6 -sulfanone (4g)



Yellow oil, 410.6 mg, 85% yield. dr=1:1.6.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.02 – 7.98 (m, 2H), 7.66 (tt, J = 8.3, 1.3 Hz, 3H), 7.61 – 7.29 (m, 17H), 7.18 (ddq, J = 5.3, 3.7, 2.0 Hz, 6H), 4.45 – 4.34 (m, 2H), 3.50 (dd, J = 12.1, 6.2 Hz, 1H), 3.33 (ddd, J = 12.1, 8.4, 7.6 Hz, 2H), 3.20 – 3.07 (m, 7H).

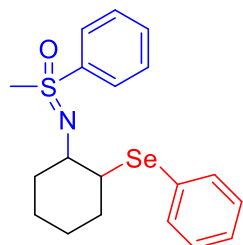
^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 148.97 and 147.75, 139.53 and 139.11, 133.14 and 132.91, 132.32 and 132.21, 130.74 and 130.55, 129.41 and 129.30, 129.14 and 129.07, 128.95 and 128.91, 128.70 and 128.37, 127.49 (d, J = 6.4 Hz) and 127.20 (d, J = 6.4 Hz), 126.64 and 126.56, 125.22 (q, J = 3.9 Hz) and 124.98 (q, J = 3.7 Hz), 58.62 and 58.47, 45.32 and 45.00, 38.38 and 38.00.

^{19}F NMR (376 MHz, Chloroform-d) δ -62.30, -62.36.

IR (neat, cm^{-1}): 3074, 3026, 2927, 2860, 1613, 1576, 1477, 1445, 1323, 1226, 1159, 1122, 1119, 1107, 1066, 1013, 986, 831, 739, 688.

HRMS (ESI) m/z $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{22}\text{H}_{21}\text{F}_3\text{NOSse}^+$ 484.0456, found 484.0454.

Methyl(phenyl)((2-(phenylselanyl)cyclohexyl)imino)- λ^6 -sulfanone (4h)



Yellow oil, 345.9 mg, 88% yield. dr=1:1.4.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.01 – 7.94 (m, 2H), 7.94 – 7.85 (m, 2H), 7.59 – 7.40 (m, 10H), 7.21 – 7.10 (m, 6H), 3.42 (ddd, J = 10.4, 9.0, 3.9 Hz, 1H), 3.34 – 3.23 (m, 2H), 3.04 (s, 3H), 2.99 (s, 3H), 2.89 (td, J = 9.7, 4.1 Hz, 1H), 2.09 –

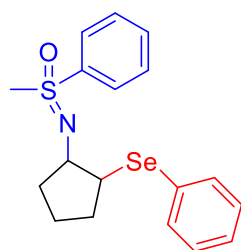
1.81 (m, 4H), 1.56 – 1.07 (m, 12H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 141.46 and 139.14, 134.59 and 134.46, 132.80 and 132.73, 130.21 and 130.11, 129.27 and 129.13, 129.12 and 128.81, 128.70 and 128.08, 126.89 and 126.87, 59.66 and 58.51, 51.18 and 51.08, 45.85 and 43.55, 37.38 and 36.43, 32.84 and 32.62, 26.26 and 26.06, 24.34 and 24.31.

IR (neat, cm^{-1}): 3055, 2927, 2853, 1577, 1476, 1444, 1234, 1143, 1085, 1067, 1022, 996, 969, 782, 741, 690.

HRMS (ESI) m/z $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{19}\text{H}_{24}\text{NOSse}^+$ 394.0738, found 394.0742.

Methyl(phenyl)((2-(phenylselanyl)cyclopentyl)imino)- λ^6 -sulfanone (4i)



Yellow oil, 307.0 mg, 81% yield. dr=1:1.4.

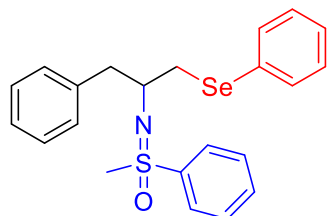
^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 7.89 – 7.84 (m, 2H), 7.80 – 7.70 (m, 2H), 7.56 – 7.38 (m, 10H), 7.15 – 7.10 (m, 6H), 3.61 (dt, $J = 7.9, 5.1$ Hz, 1H), 3.51 – 3.41 (m, 2H), 3.23 (q, $J = 6.7$ Hz, 1H), 2.98 (s, 3H), 2.96 (s, 3H), 2.28 – 2.06 (m, 2H), 1.96 – 1.81 (m, 2H), 1.71 – 1.46 (m, 8H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 140.21 and 139.12, 133.67 and 133.51, 132.89 and 132.77, 130.63 and 130.60, 129.33 and 129.24, 129.20 and 129.12, 128.78 and 128.50, 126.78 and 126.69, 62.26 and 61.98, 50.71 and 50.37, 45.51 and 44.64, 35.50 and 34.39, 32.05 and 31.52, 22.87 and 22.66.

IR (neat, cm^{-1}): 3056, 2956, 2866, 1578, 1477, 1445, 1235, 1137, 1085, 1069, 1022, 981, 785, 741, 690.

HRMS (ESI) m/z $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{18}\text{H}_{22}\text{NOSSe}^+$ 380.0582, found 380.0580.

Methyl(phenyl)((1-phenyl-3-(phenylselanyl)propan-2-yl)imino)- λ^6 -sulfanone (4j)



Yellow oil, 201.7 mg, 47% yield. dr=1:1.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 7.96 – 7.89 (m, 3H), 7.65 – 7.50 (m, 6H), 7.49 – 7.42 (m, 4H), 7.30 – 7.18 (m, 17H), 3.64 – 3.54 (m, 2H), 3.44 (dd, $J = 13.9, 6.9$ Hz, 1H), 3.35 – 3.27 (m, 3H), 3.18 – 3.06 (m, 8H), 2.99 (dd, $J = 13.6, 6.5$ Hz, 2H).

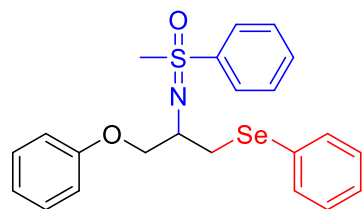
^{13}C NMR (101 MHz, Chloroform-d) δ 140.00 and 139.89, 139.76 and 139.25, 134.26 and 133.99, 132.88 and 132.86, 131.82 and 131.80, 130.02 and 129.99, 129.41 and 129.40, 129.34 and 129.26, 128.87 and 128.64, 128.53 and 128.22, 127.09 and 126.98, 126.24 and 126.22, 49.17 and 48.86, 47.85 and 47.82, 45.18 and 44.79, 39.22 and 39.12.

IR (neat, cm^{-1}): 3058, 3024, 2924, 2850, 1578, 1495, 1477, 1452, 1312, 1231, 1137,

1085, 1022, 977, 742, 690.

HRMS (ESI) m/z $[M + H]^+$ Calcd for $C_{22}H_{24}NOSse^+$ 430.0738, found 430.0734.

Methyl((1-phenoxy-3-(phenylselanyl)propan-2-yl)imino)(phenyl)- λ^6 -sulfanone (4k)



Yellow oil, 338.2 mg, 76% yield. dr=1:1.

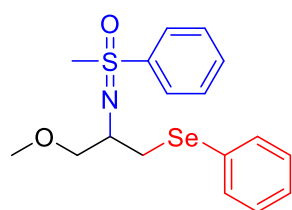
1H NMR (400 MHz, Chloroform- d , mixture of two isomers) δ 7.74 (ddd, $J = 8.4, 6.7, 1.3$ Hz, 4H), 7.52 – 7.32 (m, 10H), 7.19 – 7.10 (m, 10H), 6.84 (dddd, $J = 10.1, 6.3, 3.2, 2.0$ Hz, 2H), 6.78 – 6.68 (m, 4H), 4.37 – 4.28 (m, 3H), 4.14 (dd, $J = 9.6, 4.5$ Hz, 1H), 3.61 – 3.50 (m, 2H), 3.41 (ddd, $J = 15.8, 12.6, 5.2$ Hz, 2H), 3.32 – 3.22 (m, 2H), 2.98 (d, $J = 0.9$ Hz, 6H).

^{13}C NMR (101 MHz, Chloroform- d , mixture of two isomers) δ 158.66 and 158.58, 139.12 and 138.74, 134.22 and 134.04, 133.00 and 132.95, 131.83 and 131.80, 129.47 and 129.44, 129.42 and 129.41, 129.09 and 129.08, 128.75 and 128.63, 127.43 and 127.36, 120.77 and 120.76, 114.65 and 114.61, 67.98 and 67.94, 46.09 and 45.86, 45.38 and 45.11, 45.01 and 44.83.

IR (neat, cm^{-1}): 3058, 2925, 2851, 1598, 1585, 1496, 1477, 1445, 1239, 1141, 1085, 980, 785, 742, 690.

HRMS (ESI) m/z $[M + H]^+$ Calcd for $C_{22}H_{24}NO_2Sse^+$ 446.0687, found 446.0690.

((1-methoxy-3-(phenylselanyl)propan-2-yl)imino)(methyl)(phenyl)- λ^6 -sulfanone (4l)



Yellow oil, 245.2 mg, 64% yield. dr=1:1.

1H NMR (400 MHz, Chloroform- d , mixture of two isomers) δ 7.79 (dq, $J = 7.0, 1.2$ Hz, 4H), 7.53 – 7.40 (m, 10H), 7.15 – 7.08 (m, 6H), 3.70 (dd, $J = 10.9, 5.8$ Hz, 3H), 3.60 (dd, $J = 9.8, 4.7$ Hz, 1H), 3.41 – 3.35 (m, 2H), 3.28 – 3.21 (m, 7H), 3.20 – 3.07 (m, 3H), 2.98 (d, $J = 5.2$ Hz, 6H).

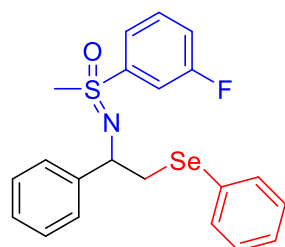
^{13}C NMR (101 MHz, Chloroform- d , mixture of two isomers) δ 139.45 and 139.06, 133.95 and 133.66, 132.96 and 132.94, 129.68 and 129.62, 129.42 and 129.41, 129.29 and 129.24, 128.68 and 128.58, 127.12 and 127.02, 72.96 and 72.83, 58.85

and 58.77, 46.70 and 46.67, 45.61 and 45.60, 45.01 and 44.79.

IR (neat, cm^{-1}): 3056, 2983, 2924, 2888, 1578, 1477, 1445, 1239, 1139, 1087, 1022, 979, 785, 742, 691.

HRMS (ESI) m/z $[M + H]^+$ Calcd for $\text{C}_{17}\text{H}_{22}\text{NO}_2\text{SSe}^+$ 384.0531, found 384.0533.

(3-fluorophenyl)(methyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanon (4m)



Yellow oil, 342.1 mg, 79% yield. dr=1:1.

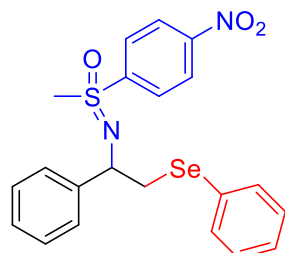
^1H NMR (400 MHz, Chloroform- d , mixture of two isomers) δ 7.80 (ddd, $J = 7.8, 1.7, 1.0$ Hz, 1H), 7.75 (ddd, $J = 8.1, 2.6, 1.7$ Hz, 1H), 7.58 – 7.53 (m, 1H), 7.53 – 7.49 (m, 1H), 7.48 – 7.28 (m, 13H), 7.27 – 7.19 (m, 11H), 4.42 – 4.33 (m, 2H), 3.51 (dd, $J = 11.8, 6.8$ Hz, 1H), 3.42 – 3.30 (m, 2H), 3.20 (dd, $J = 12.0, 5.7$ Hz, 1H), 3.12 (s, 3H), 3.05 (s, 3H).

^{13}C NMR (101 MHz, Chloroform- d , mixture of two isomers) δ 163.79 (d, $J = 250.6$ Hz) and 161.28 (d, $J = 250.5$ Hz), 144.80 and 143.59, 142.27 (d, $J = 7.3$ Hz) and 141.88 (d, $J = 7.2$ Hz), 132.25 and 132.08, 131.05 (d, $J = 8.0$ Hz) and 130.95 (d, $J = 8.0$ Hz), 130.70 and 130.63, 128.93 and 128.89, 128.37 and 128.18, 127.30 and 127.22, 126.99 and 126.70, 126.50 and 126.44, 124.37 and 124.12, 120.17 (d, $J = 22.1$ Hz) and 119.88 (d, $J = 22.1$ Hz), 116.09 (d, $J = 25.4$ Hz) and 115.86 (d, $J = 25.4$ Hz), 59.13 and 58.72, 45.21 and 44.97, 38.69 and 38.24.

IR (neat, cm^{-1}): 3060, 3027, 2926, 2854, 1592, 1477, 1435, 1238, 1129, 1081, 1022, 981, 877, 737, 701, 678, 600.

HRMS (ESI) m/z $[M + H]^+$ Calcd for $\text{C}_{21}\text{H}_{21}\text{FNOSSe}^+$ 434.0488, found 434.0497.

Methyl(4-nitrophenyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanonone (4n)



Yellow oil, 381.8 mg, 83% yield. dr=1:1.

^1H NMR (400 MHz, Chloroform- d , mixture of two isomers) δ 8.35 – 8.30 (m, 2H), 8.19 – 8.12 (m, 4H), 7.83 – 7.79 (m, 2H), 7.48 – 7.42 (m, 2H), 7.41 – 7.29 (m, 6H), 7.29 – 7.13 (m, 12H),

4.41 (t, J = 7.1 Hz, 1H), 4.31 (dd, J = 8.4, 5.4 Hz, 1H), 3.50 (dd, J = 11.9, 7.2 Hz, 1H), 3.38 – 3.28 (m, 2H), 3.20 (dd, J = 12.2, 5.3 Hz, 1H), 3.15 (s, 3H), 3.10 (s, 3H).

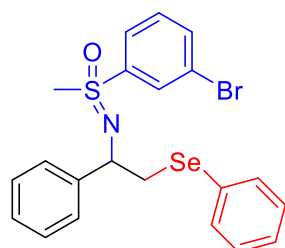
¹³C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 150.29 and 150.00, 146.35 and 145.60, 144.57 and 143.32, 132.30 and 131.89, 130.96 and 130.89, 130.02 and 129.71, 129.07 and 129.02, 128.48 and 128.29, 127.45 and 127.39, 127.04 and 126.69, 126.63 and 126.61, 124.36 and 124.00, 59.20 and 58.66, 44.98 and 44.68, 38.48 and 38.19.

IR (neat, cm⁻¹): 3095, 3056, 3032, 2926, 2860, 2360, 1603, 1578, 1524, 1480, 1437, 1346, 1228, 1129, 1072, 1022, 985, 854, 739, 719, 696.

HRMS (ESI) m/z [M + Na]⁺ Calcd for C₂₁H₂₀N₂NaO₃SSe⁺ 483.0252, found 483.0249.

(3-bromophenyl)(methyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)-λ⁶-sulfanone

(4o)



Yellow oil, 428.9 mg, 87% yield. dr=1:1.1.

¹H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.19 (t, J = 1.8 Hz, 1H), 7.95 (ddd, J = 7.8, 1.8, 1.0 Hz, 1H), 7.81 (t, J = 1.8 Hz, 1H), 7.75 (ddd, J = 7.9, 2.0, 1.0 Hz, 1H), 7.60 (dddd, J = 7.9, 5.9, 1.9, 1.0 Hz, 2H), 7.47 – 7.35 (m, 7H),

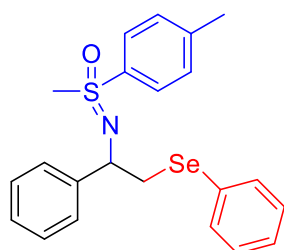
7.35 – 7.16 (m, 15H), 4.41 – 4.33 (m, 2H), 3.51 (dd, J = 11.8, 6.9 Hz, 1H), 3.42 – 3.30 (m, 2H), 3.19 (dd, J = 12.0, 5.6 Hz, 1H), 3.11 (s, 3H), 3.05 (s, 3H).

¹³C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 144.88 and 143.54, 141.92 and 141.59, 136.04 and 135.74, 132.23 and 132.08, 131.68 and 131.58, 131.19 and 131.01, 130.86 and 130.55, 129.03 and 129.00, 128.42 and 128.29, 127.36 and 127.33, 127.31 and 127.05, 126.99 and 126.76, 126.57 and 126.52, 123.38 and 123.05, 59.20 and 58.72, 45.27 and 45.03, 38.74 and 38.19.

IR (neat, cm⁻¹): 3057, 3025, 2926, 2854, 1577, 1568, 1477, 1453, 1402, 1235, 1132, 1071, 1022, 979, 777, 735, 700, 677.

HRMS (ESI) m/z [M + Na]⁺ Calcd for C₂₁H₂₀BrNNaOSSe⁺ 515.9506, found 515.9506.

Methyl((1-phenyl-2-(phenylselanyl)ethyl)imino)(p-tolyl)- λ^6 -sulfanone (4p)



Yellow oil, 356.1 mg, 83% yield. dr=1:1.2.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 7.91 – 7.86 (m, 2H), 7.63 – 7.58 (m, 2H), 7.44 – 7.27 (m, 11H), 7.27 – 7.14 (m, 13H), 4.37 (dt, J = 8.1, 6.5 Hz, 2H), 3.51 (dd, J = 11.8, 6.5 Hz, 1H), 3.43 – 3.32 (m, 2H), 3.20 (dd, J = 11.8, 6.1

Hz, 1H), 3.10 (s, 3H), 3.04 (s, 3H), 2.47 (s, 3H), 2.40 (s, 3H).

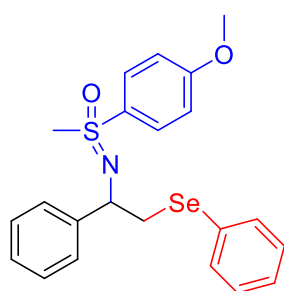
^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 145.21 and 144.14, 143.76 and 143.62, 136.64 and 136.29, 131.99 and 131.94, 131.38 and 131.25, 129.99 and 129.77, 128.94 and 128.91, 128.73 and 128.68, 128.34 and 128.18, 127.18 and 127.11, 127.07 and 126.84, 126.38 and 126.29, 58.89 and 58.83, 45.24 and 45.17, 38.80 and 38.51, 21.64 and 21.56.

IR (neat, cm^{-1}): 3057, 3026, 2926, 2859, 1596, 1578, 1491, 1478, 1451, 1436, 1232, 1131, 1084, 1022, 978, 817, 737, 700, 669, 624.

HRMS (ESI) m/z $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{22}\text{H}_{23}\text{NNaOSSe}^+$ 452.0558, found 452.0557.

(4-methoxyphenyl)(methyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanone

(4q)



Yellow oil, 396.1 mg, 89% yield. dr=1:1.3.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 7.97 – 7.90 (m, 2H), 7.66 – 7.61 (m, 2H), 7.40 (tdd, J = 7.8, 2.9, 1.6 Hz, 6H), 7.34 – 7.16 (m, 14H), 7.04 – 6.98 (m, 2H), 6.89 – 6.83 (m, 2H), 4.42 – 4.34 (m, 2H), 3.90 (s, 3H), 3.85 (s, 3H),

3.51 (dd, J = 11.7, 6.5 Hz, 1H), 3.37 (ddd, J = 12.5, 11.8, 7.8 Hz, 2H), 3.21 (dd, J = 11.9, 6.0 Hz, 1H), 3.10 (s, 3H), 3.04 (s, 3H).

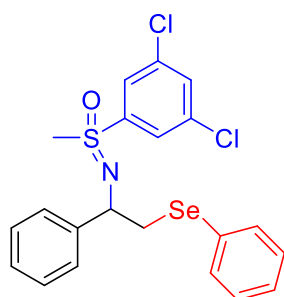
^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 163.21 and 163.09, 145.20 and 144.14, 131.99 and 131.94, 131.35 and 131.20, 130.90 and 130.84, 130.75 and 130.47, 128.91 and 128.86, 128.32 and 128.17, 127.15 and 127.07, 127.05 and 126.76, 126.36 and 126.25, 114.49 and 114.27, 58.84 and 58.76, 55.69

and 55.64, 45.51 and 45.38, 38.79 and 38.45.

IR (neat, cm^{-1}): 3058, 3025, 2927, 2838, 1593, 1577, 1495, 1477, 1310, 1258, 1230, 1128, 1087, 1023, 979, 835, 803, 736, 700, 669.

HRMS (ESI) m/z $[M + H]^+$ Calcd for $\text{C}_{22}\text{H}_{24}\text{NO}_2\text{SSe}^+$ 446.0687, found 446.0693.

(3,5-dichlorophenyl)(methyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanone (4r)



Yellow oil, 444.3 mg, 92% yield. dr=1:1.1.

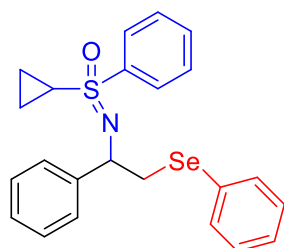
^1H NMR (400 MHz, Chloroform- d , mixture of two isomers) δ 7.92 (d, $J = 1.8$ Hz, 2H), 7.59 (t, $J = 1.9$ Hz, 1H), 7.50 – 7.40 (m, 7H), 7.38 – 7.17 (m, 16H), 4.40 – 4.31 (m, 2H), 3.56 – 3.46 (m, 1H), 3.42 – 3.29 (m, 2H), 3.21 – 3.13 (m, 1H), 3.10 (s, 3H), 3.05 (s, 3H).

^{13}C NMR (101 MHz, Chloroform- d , mixture of two isomers) δ 144.68 and 143.28, 143.15 and 142.99, 136.14 and 135.75, 132.94 and 132.52, 132.37 and 132.11, 131.52 and 131.09, 129.04 and 129.01, 128.45 and 128.31, 127.54 and 127.40, 127.24 and 127.06, 126.91 and 126.72, 126.65 and 126.61, 59.50 and 58.67, 45.24 and 44.91, 38.64 and 37.90.

IR (neat, cm^{-1}): 3070, 3026, 2927, 2851, 1567, 1477, 1436, 1416, 1242, 1141, 1022, 980, 869, 799, 736, 700, 668.

HRMS (ESI) m/z $[M + \text{Na}]^+$ Calcd for $\text{C}_{21}\text{H}_{19}\text{Cl}_2\text{NNaOSSe}^+$ 505.9622, found 505.9622.

Cyclopropyl(phenyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanone (4s)



Yellow oil, 401.4 mg, 91% yield. dr=1:1.

^1H NMR (400 MHz, Chloroform- d , mixture of two isomers) δ 8.02 – 7.96 (m, 2H), 7.77 – 7.72 (m, 2H), 7.66 – 7.48 (m, 5H), 7.42 (dddd, $J = 9.8, 7.4, 5.5, 1.6$ Hz, 8H), 7.34 – 7.19 (m, 13H), 4.58 – 4.46 (m, 2H), 3.51 (dd, $J = 11.7, 7.2$ Hz, 1H), 3.45 – 3.32 (m, 2H), 3.23 (dd, $J = 11.8, 6.1$ Hz, 1H), 2.55 (tt, $J = 7.7, 4.9$ Hz, 1H), 2.34 (tt, $J =$

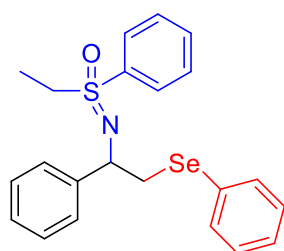
7.9, 4.7 Hz, 1H), 1.47 (tqd, J = 10.5, 4.9, 3.3 Hz, 2H), 1.15 – 1.01 (m, 3H), 1.00 – 0.91 (m, 1H), 0.85 – 0.73 (m, 2H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 145.53 and 144.45, 140.19 and 139.78, 132.64 and 132.52, 132.04 and 131.96, 131.52 and 131.38, 129.13 and 128.95, 128.94 and 128.93, 128.67 and 128.66, 128.25 and 128.16, 127.11 and 127.04, 126.96 and 126.82, 126.38 and 126.30, 58.59 and 58.50, 39.18 and 39.01, 32.92 and 32.42, 6.55 and 6.48, 5.27 and 4.89.

IR (neat, cm^{-1}): 3058, 3026, 2926, 2853, 1578, 1477, 1445, 1238, 1132, 1070, 1022, 885, 735, 691.

HRMS (ESI) m/z $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{23}\text{H}_{23}\text{NNaOSSe}^+$ 464.0558, found 464.0558.

Ethyl(phenyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanone (4t)



Yellow oil, 377.6 mg, 88% yield. dr=1:1.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.01 – 7.96 (m, 2H), 7.67 – 7.48 (m, 7H), 7.39 (tdd, J = 9.1, 6.7, 4.0 Hz, 8H), 7.33 – 7.17 (m, 13H), 4.45 – 4.38 (m, 2H), 3.53 (dd, J = 11.7, 6.3 Hz, 1H), 3.38 (ddd, J = 20.6, 11.8, 7.8 Hz, 2H),

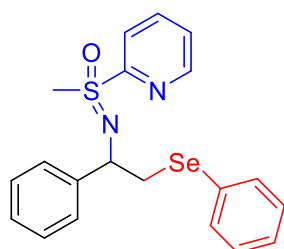
3.30 – 3.12 (m, 5H), 1.25 (t, J = 7.4 Hz, 3H), 1.16 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 145.39 and 144.17, 137.85 and 137.36, 132.87 and 132.66, 132.04 and 131.94, 131.54 and 131.42, 129.55 and 129.37, 129.18 and 128.92, 128.90 and 128.82, 128.57 and 128.23, 128.09 and 127.75, 127.09 and 126.75, 126.34 and 126.21, 58.74 and 58.41, 51.04 and 51.03, 38.99 and 38.63, 7.65 and 7.50.

IR (neat, cm^{-1}): 3058, 3026, 2977, 2934, 2872, 1578, 1478, 1445, 1239, 1219, 1130, 1085, 1053, 1022, 731, 691.

HRMS (ESI) m/z $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{22}\text{H}_{23}\text{NNaOSSe}^+$ 452.0558, found 452.0558.

Methyl((1-phenyl-2-(phenylselanyl)ethyl)imino)(pyridin-2-yl)- λ^6 -sulfanone (4u)



Yellow oil, 353.6 mg, 85% yield. dr=1:1.

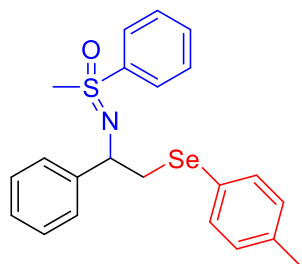
^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.73 (ddd, $J = 4.7, 1.8, 0.9$ Hz, 1H), 8.48 (ddd, $J = 4.7, 1.7, 0.9$ Hz, 1H), 8.10 (dt, $J = 7.9, 1.1$ Hz, 1H), 7.89 (td, $J = 7.7, 1.7$ Hz, 1H), 7.70 (dt, $J = 7.9, 1.1$ Hz, 1H), 7.62 (td, $J = 7.7, 1.8$ Hz, 1H), 7.52 – 7.45 (m, 3H), 7.44 – 7.38 (m, 2H), 7.34 – 7.13 (m, 12H), 7.12 – 7.01 (m, 5H), 4.54 (t, $J = 7.2$ Hz, 1H), 4.47 (t, $J = 7.0$ Hz, 1H), 3.49 (dd, $J = 11.7, 7.4$ Hz, 1H), 3.34 – 3.24 (m, 5H), 3.24 – 3.15 (m, 4H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 158.40 and 158.22, 150.00 and 149.63, 144.50 and 142.97, 137.83 and 137.30, 132.40 and 132.21, 131.18 and 131.15, 128.90 and 128.84, 128.36 and 127.89, 127.27 and 127.09, 126.97 and 126.84, 126.60 and 126.45, 126.38 and 125.89, 122.78 and 122.11, 58.57 and 58.49, 41.26 and 41.21, 38.35 and 38.18.

IR (neat, cm^{-1}): 3055, 3027, 2928, 1577, 1477, 1452, 1436, 1424, 1232, 1142, 1112, 1073, 1023, 989, 760, 737, 700.

HRMS (ESI) m/z $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{20}\text{H}_{20}\text{N}_2\text{NaOSse}^+$ 439.0354, found 439.0349.

Methyl(phenyl)((1-phenyl-2-(p-tolylselanyl)ethyl)imino)- λ^6 -sulfanone (4v)



Yellow oil, 381.9 mg, 89% yield. dr=1:1.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.04 – 7.99 (m, 2H), 7.74 – 7.69 (m, 2H), 7.66 – 7.60 (m, 1H), 7.59 – 7.49 (m, 3H), 7.42 – 7.35 (m, 4H), 7.34 – 7.19 (m, 12H), 7.00 (dd, $J = 7.9, 5.8$ Hz, 4H), 4.36 (dt, $J = 8.3, 6.6$ Hz, 2H), 3.47 (dd, $J = 11.7, 6.7$ Hz, 1H), 3.33 (ddd, $J = 15.4, 11.8, 7.7$ Hz, 2H), 3.19 – 3.08 (m, 4H), 3.05 (s, 3H), 2.31 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 145.10 and 144.00, 139.83 and 139.52, 136.41 and 136.29, 132.89 and 132.69, 132.61 and 132.59, 129.72 and 129.69, 129.27 and 129.01, 128.68 and 128.55, 128.30 and 128.13, 127.26 and 127.15, 127.09 and 127.04, 127.02 and 126.79, 58.96 and 58.80, 45.19 and 45.00, 39.17 and 38.79, 21.06.

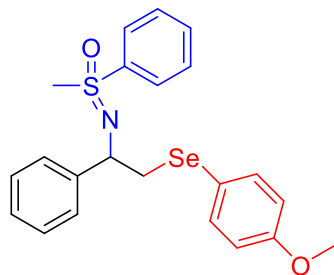
IR (neat, cm^{-1}): 3060, 3025, 2924, 2861, 1489, 1445, 1234, 1131, 1085, 1069, 1015,

978, 803, 744, 700, 689.

HRMS (ESI) m/z $[M + Na]^+$ Calcd for $C_{22}H_{23}NNaOSSe^+$ 452.0558, found 452.0566.

((2-((4-methoxyphenyl)selanyl)-1-phenylethyl)imino)(methyl)(phenyl)- λ^6 -sulfanone

(4w)



Yellow oil, 378.3 mg, 85% yield. dr=1:1.

1H NMR (400 MHz, Chloroform-d, mixture of two isomers)

δ 7.94 – 7.89 (m, 2H), 7.63 – 7.58 (m, 2H), 7.57 – 7.51 (m, 1H), 7.50 – 7.38 (m, 3H), 7.32 – 7.17 (m, 11H), 7.16 – 7.09 (m, 5H), 6.68 – 6.60 (m, 4H), 4.24 (td, $J = 6.8, 6.1, 1.8$ Hz,

2H), 3.69 (s, 6H), 3.33 (dd, $J = 11.8, 6.7$ Hz, 1H), 3.19 (ddd, $J = 15.8, 11.8, 7.6$ Hz, 2H), 3.05 – 2.99 (m, 4H), 2.94 (s, 3H).

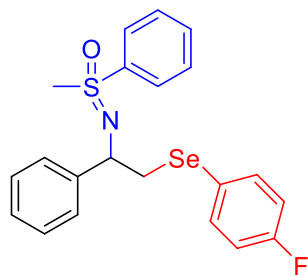
^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 158.94 and 158.89, 145.07 and 143.97, 139.79 and 139.60, 135.04 and 134.99, 132.89 and 132.67, 129.26 and 129.00, 128.64 and 128.54, 128.29 and 128.11, 127.12 and 127.06, 126.99 and 126.81, 120.91 and 120.79, 114.63 and 114.60, 58.95 and 58.82, 55.27, 45.17 and 45.07, 40.01 and 39.61.

IR (neat, cm^{-1}): 3059, 3025, 2928, 2835, 1591, 1490, 1445, 1284, 1245, 1175, 1131, 1027, 978, 824, 744, 701, 689.

HRMS (ESI) m/z $[M + Na]^+$ Calcd for $C_{22}H_{23}NNaO_2SSe^+$ 468.0507, found 468.0506.

((2-((4-fluorophenyl)selanyl)-1-phenylethyl)imino)(methyl)(phenyl)- λ^6 -sulfanone

(4x)



Yellow oil, 359.4 mg, 83% yield. dr=1:1.

1H NMR (400 MHz, Chloroform-d, mixture of two isomers)

δ 8.02 – 7.98 (m, 2H), 7.71 – 7.62 (m, 3H), 7.60 – 7.49 (m, 3H), 7.41 – 7.28 (m, 10H), 7.27 – 7.16 (m, 6H), 6.87 (ddt, $J = 8.8, 6.6, 2.6$ Hz, 4H), 4.34 (ddd, $J = 14.2, 7.7, 6.3$ Hz, 2H),

3.46 (dd, $J = 11.8, 6.3$ Hz, 1H), 3.31 (ddd, $J = 13.1, 11.9, 7.7$ Hz, 2H), 3.19 – 3.11 (m,

4H), 3.03 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 163.23 (d, $J = 248.2$ Hz) and 160.79 (d, $J = 248.3$ Hz), 144.87 and 143.75, 139.57 and 139.52, 134.84 (d, $J = 7.8$ Hz) and 134.74 (d, $J = 7.9$ Hz), 132.99 and 132.79, 129.31 and 129.07, 128.57 and 128.56, 128.36 and 128.16, 127.25 and 127.10, 127.05 and 126.80, 125.44 (d, $J = 3.4$ Hz) and 125.26 (d, $J = 3.4$ Hz), 116.08 (d, $J = 21.4$ Hz) and 115.87 (d, $J = 21.5$ Hz), 58.85 and 58.77, 45.17 and 45.09, 39.76 and 39.38.

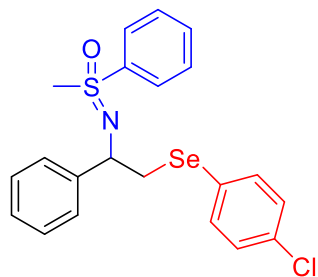
^{19}F NMR (376 MHz, Chloroform-d) δ -115.39, -115.61.

IR (neat, cm^{-1}): 3061, 3027, 2927, 2853, 1583, 1487, 1446, 1229, 1131, 1085, 1068, 979, 826, 744, 701, 690.

HRMS (ESI) m/z $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{21}\text{H}_{20}\text{FNNaOSSe}^+$ 456.0307, found 456.0307.

((2-((4-chlorophenyl)selenanyl)-1-phenylethyl)imino)(methyl)(phenyl)- λ^6 -sulfanone

(4y)



Yellow oil, 390.6 mg, 87% yield. dr=1:1.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.02 – 7.98 (m, 2H), 7.71 – 7.62 (m, 3H), 7.59 – 7.50 (m, 3H), 7.42 – 7.35 (m, 4H), 7.34 – 7.18 (m, 12H), 7.17 – 7.10 (m, 4H), 4.35 (ddd, $J = 16.9, 7.7, 6.2$ Hz, 2H), 3.48 (dd, $J =$

11.8, 6.3 Hz, 1H), 3.39 – 3.28 (m, 2H), 3.19 (dd, $J = 11.9, 6.1$ Hz, 1H), 3.12 (s, 3H), 3.03 (s, 3H).

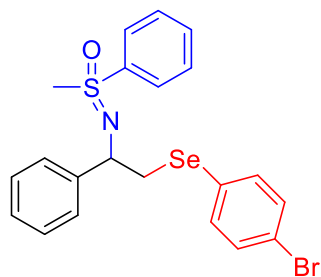
^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 144.83 and 143.72, 139.51 and 139.45, 133.62 and 133.54, 133.03 and 132.85, 132.53 and 132.42, 129.46 and 129.33, 129.29 and 129.12, 129.00 and 128.95, 128.57 and 128.54, 128.40 and 128.20, 127.30 and 127.17, 127.06 and 126.81, 58.78 and 58.72, 45.16 and 45.09, 39.19 and 38.81.

IR (neat, cm^{-1}): 3060, 3026, 2927, 2853, 1474, 1445, 1234, 1132, 1089, 1010, 979, 813, 744, 701, 689.

HRMS (ESI) m/z $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{21}\text{H}_{20}\text{ClNNaOSSe}^+$ 472.0012, found 472.0011.

((2-((4-bromophenyl)selenanyl)-1-phenylethyl)imino)(methyl)(phenyl)-λ⁶-sulfanone

(4z)



Yellow oil, 458.5 mg, 93% yield. dr=1:1.

¹H NMR (400 MHz, Chloroform-d, mixture of two isomers)

δ 8.01 – 7.97 (m, 2H), 7.71 – 7.62 (m, 3H), 7.60 – 7.50 (m, 3H), 7.43 – 7.35 (m, 4H), 7.34 – 7.19 (m, 16H), 4.34 (ddd, J = 16.4, 7.7, 6.2 Hz, 2H), 3.48 (dd, J = 11.8, 6.3 Hz, 1H), 3.33

(td, J = 12.1, 7.7 Hz, 2H), 3.19 (dd, J = 11.9, 6.1 Hz, 1H), 3.12 (s, 3H), 3.03 (s, 3H).

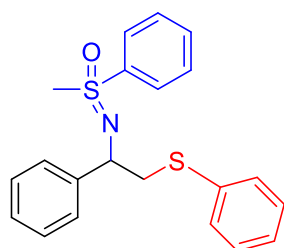
¹³C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 144.85 and 143.74,

139.51 and 139.45, 133.82 and 133.74, 133.05 and 132.87, 131.91 and 131.87, 130.27 and 130.10, 129.36 and 129.14, 128.56 and 128.53, 128.41 and 128.23, 127.31 and 127.19, 127.08 and 126.83, 120.53 and 120.43, 58.76 and 58.71, 45.14 and 45.08, 39.09 and 38.73.

IR (neat, cm⁻¹): 3059, 3026, 2927, 1617, 1468, 1445, 1380, 1232, 1131, 1073, 1005, 978, 808, 743, 701, 689.

HRMS (ESI) m/z [M + Na]⁺ Calcd for C₂₁H₂₀BrNNaOSSe⁺ 515.9506, found 515.9503.

Methyl(phenyl)((1-phenyl-2-(phenylthio)ethyl)imino)-λ⁶-sulfanone (4ab)



Colourless oil, 218.0 mg, 71% yield. dr=1:1.

¹H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ

7.95 – 7.90 (m, 2H), 7.65 – 7.59 (m, 2H), 7.57 – 7.51 (m, 1H), 7.50 – 7.39 (m, 3H), 7.34 – 7.25 (m, 8H), 7.24 – 7.07 (m, 14H), 4.28 (dt, J = 8.0, 6.5 Hz, 2H), 3.43 (dd, J = 11.8, 6.7 Hz, 1H),

3.28 (ddd, J = 14.2, 11.8, 7.7 Hz, 2H), 3.11 (dd, J = 11.9, 6.1 Hz, 1H), 3.04 (s, 3H), 2.96 (s, 3H).

¹³C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 144.98 and 143.81,

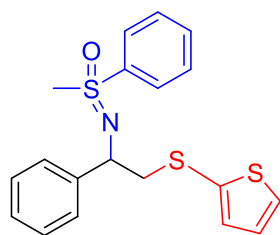
139.63 and 139.34, 132.99 and 132.80, 132.09 and 132.01, 131.23 and 131.09, 129.31 and 129.07, 128.91 and 128.88, 128.68 and 128.57, 128.35 and 128.16,

127.23 and 127.10, 127.04 and 126.76, 126.41 and 126.33, 58.91 and 58.76, 45.18 and 45.01, 38.72 and 38.32.

IR (neat, cm^{-1}): 3059, 3026, 2924, 2854, 1682, 1636, 1581, 1479, 1446, 1405, 1314, 1280, 1228, 1134, 1087, 1025, 981, 742, 690.

HRMS (ESI) m/z $[M + H]^+$ Calcd for $\text{C}_{21}\text{H}_{22}\text{NOS}_2^+$ 368.1137, found 368.1145.

Methyl(phenyl)((1-phenyl-2-(thiophen-2-ylthio)ethyl)imino)- λ^6 -sulfanone (4ac)



Colourless oil, 223.8 mg, 60% yield. dr=1:1.

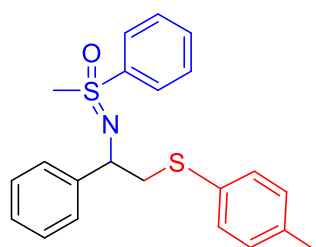
^1H NMR (400 MHz, Chloroform- d , mixture of two isomers) δ 7.99 – 7.89 (m, 2H), 7.64 – 7.57 (m, 2H), 7.58 – 7.51 (m, 1H), 7.51 – 7.45 (m, 2H), 7.44 – 7.38 (m, 1H), 7.32 – 7.25 (m, 4H), 7.24 – 7.09 (m, 10H), 6.91 (dd, $J = 3.5, 1.3$ Hz, 1H), 6.85 – 6.77 (m, 3H), 4.22 (q, $J = 6.8$ Hz, 2H), 3.29 (dd, $J = 12.9, 6.9$ Hz, 1H), 3.21 – 3.09 (m, 2H), 3.04 (s, 3H), 3.01 – 2.94 (m, 4H).

^{13}C NMR (101 MHz, Chloroform- d , mixture of two isomers) δ 144.49 and 143.39, 139.81 and 139.45, 135.47 and 135.29, 132.99 and 132.83, 132.72 and 132.65, 129.34 and 129.03, 128.74 and 128.63, 128.57 and 128.52, 128.35 and 128.18, 127.37 and 127.36, 127.21 and 127.20, 127.09 and 126.92, 58.40 and 58.12, 49.55 and 48.98, 45.25 and 45.04.

IR (neat, cm^{-1}): 3061, 3026, 2924, 2854, 2360, 1490, 1445, 1405, 1315, 1235, 1134, 1085, 981, 845, 744, 699.

HRMS (ESI) m/z $[M + H]^+$ Calcd for $\text{C}_{19}\text{H}_{19}\text{NNaOS}_3^+$ 396.0521, found 396.0506.

Methyl(phenyl)((1-phenyl-2-(p-tolylthio)ethyl)imino)- λ^6 -sulfanone (4ad)



Colourless oil, 251.5 mg, 66% yield. dr=1:1.2.

^1H NMR (400 MHz, Chloroform- d , mixture of two isomers) δ 8.05 – 8.00 (m, 2H), 7.75 – 7.71 (m, 2H), 7.66 – 7.60 (m, 1H), 7.58 – 7.49 (m, 3H), 7.39 (ddd, $J = 7.5, 4.3, 2.2$ Hz, 4H), 7.34 – 7.20 (m, 10H), 7.17 – 7.12 (m, 2H), 7.07 – 7.01 (m,

4H), 4.30 (ddd, J = 7.7, 6.3, 4.2 Hz, 2H), 3.51 (dd, J = 12.9, 6.4 Hz, 1H), 3.32 (ddd, J = 22.1, 13.0, 7.6 Hz, 2H), 3.18 (dd, J = 13.0, 6.1 Hz, 1H), 3.12 (s, 3H), 3.05 (s, 3H), 2.31 (s, 6H).

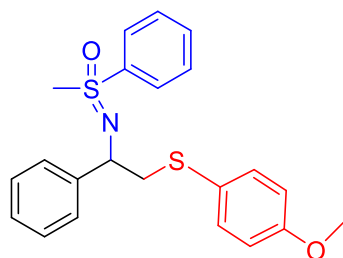
^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 144.76 and 143.64, 139.78 and 139.45, 135.58 and 135.45, 133.26 and 133.17, 132.92 and 132.74, 129.57 and 129.55, 129.39 and 129.36, 129.29 and 129.05, 128.73 and 128.58, 128.33 and 128.15, 127.20 and 127.18, 127.07 and 126.88, 58.28 and 58.15, 45.24 and 45.03, 44.74 and 44.25, 21.01.

IR (neat, cm^{-1}): 3060, 3026, 2921, 2855, 1492, 1454, 1405, 1315, 1236, 1133, 1088, 1017, 981, 805, 743, 699, 689.

HRMS (ESI) m/z $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{22}\text{H}_{24}\text{NOS}_2^+$ 382.1294, found 382.1288.

((2-((4-methoxyphenyl)thio)-1-phenylethyl)imino)(methyl)(phenyl)- λ^6 -sulfanone

(4ae)



Colourless oil, 250.2 mg, 63% yield. dr=1:2.6.

^1H NMR (400 MHz, Chloroform-d, mixture of two isomers) δ 8.03 – 7.99 (m, 1H), 7.72 – 7.67 (m, 3H), 7.64 – 7.47 (m, 3H), 7.41 – 7.35 (m, 4H), 7.33 – 7.17 (m, 13H), 6.81 – 6.74 (m, 4H), 4.26 (ddd, J = 7.5, 6.3, 3.4 Hz, 2H),

3.79 (s, 6H), 3.44 (dd, J = 12.9, 6.4 Hz, 1H), 3.27 (ddd, J = 22.6, 13.0, 7.6 Hz, 2H), 3.08 (d, J = 29.8 Hz, 7H).

^{13}C NMR (101 MHz, Chloroform-d, mixture of two isomers) δ 158.51 and 158.45, 144.73 and 143.63, 139.80 and 139.60, 132.89 and 132.68, 132.45 and 132.38, 129.26 and 129.00, 128.68 and 128.55, 128.29 and 128.11, 127.22 and 127.19, 127.14 and 127.12, 127.01 and 126.91, 114.47 and 114.43, 58.36 and 58.25, 55.32, 46.20 and 45.69, 45.20 and 45.10.

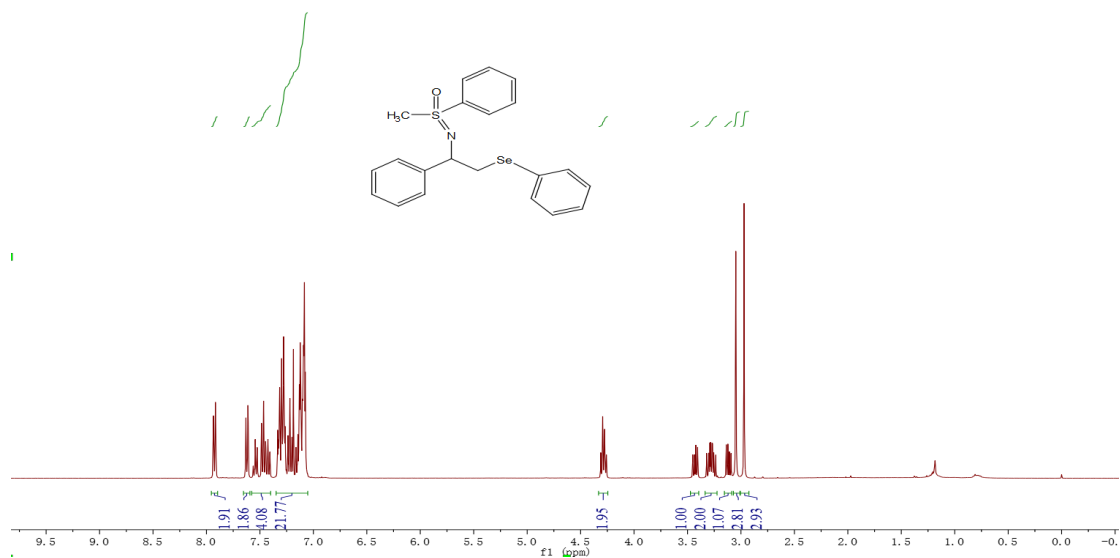
IR (neat, cm^{-1}): 3060, 3026, 2924, 2851, 1592, 1494, 1445, 1284, 1243, 1132, 1029, 981, 827, 744, 699.

HRMS (ESI) m/z $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{22}\text{H}_{24}\text{NO}_2\text{S}_2^+$ 398.1243, found 398.1238.

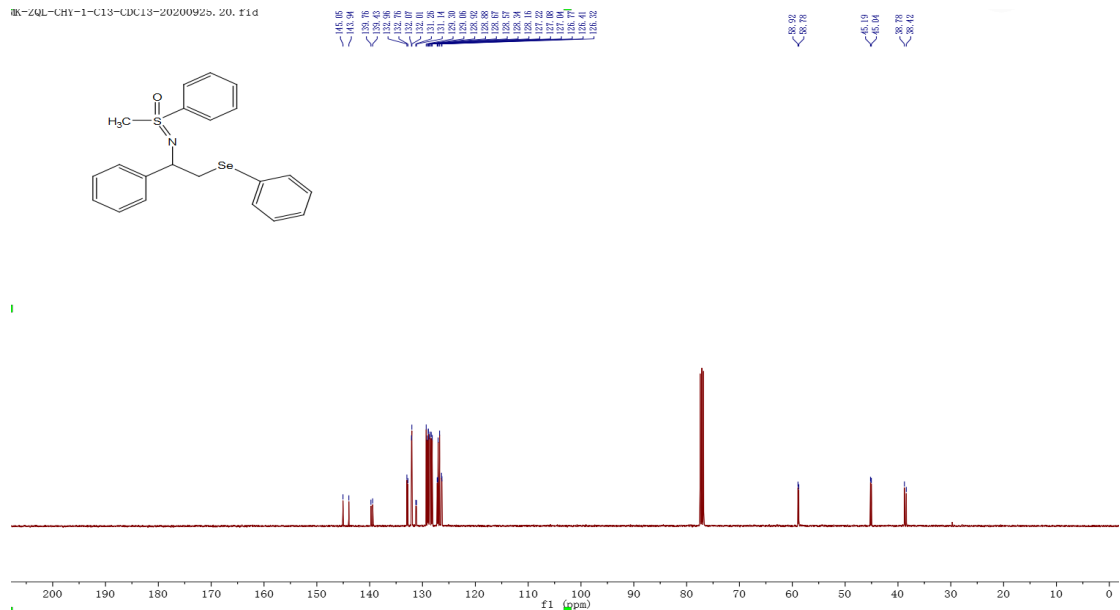
Copies of ¹H and ¹³C NMR Spectra for Compounds 4a-4ac

Methyl(phenyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)-λ⁶-sulfanone (4a)

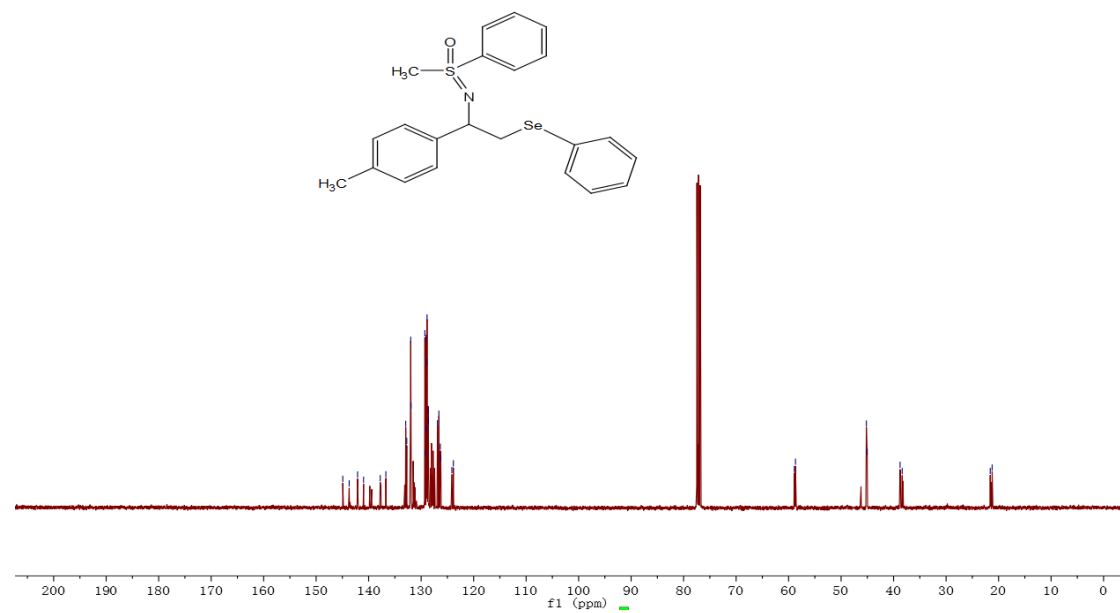
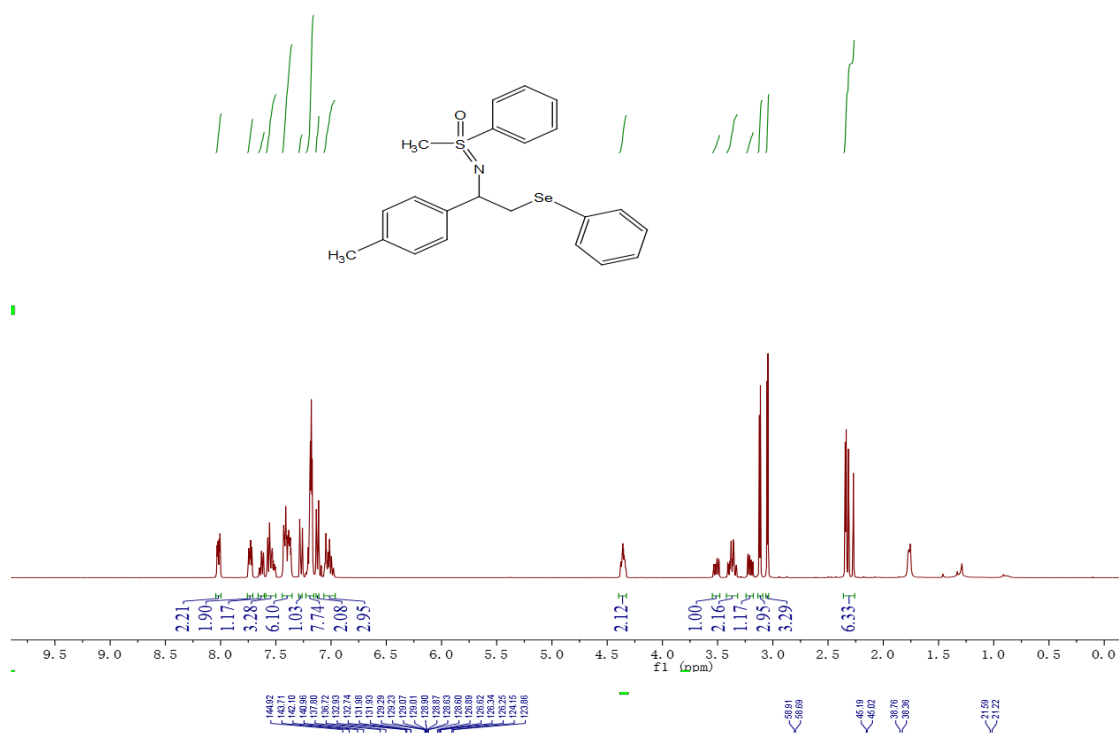
FLH-20200111-CHY-1.1.fid



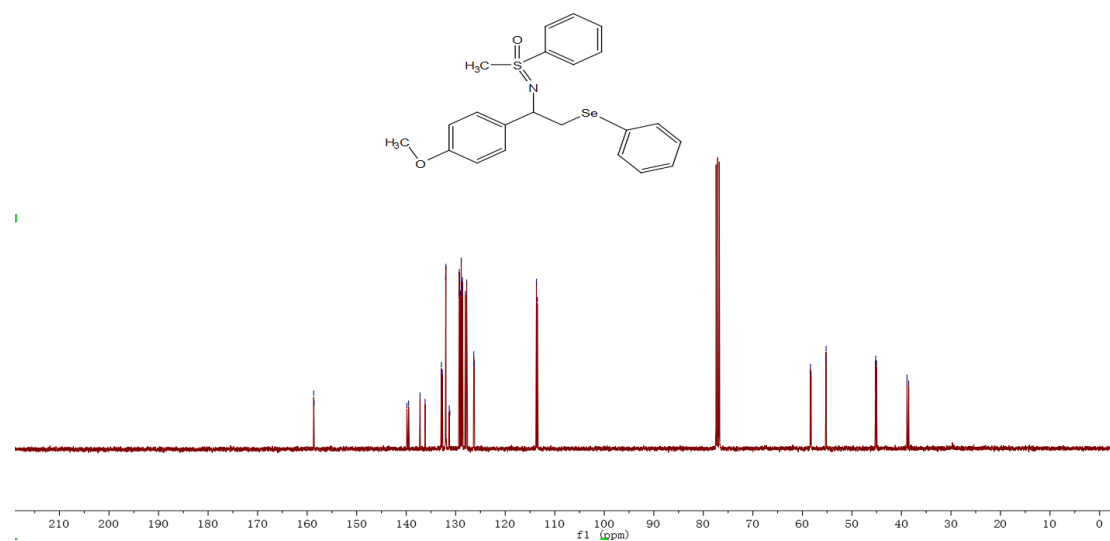
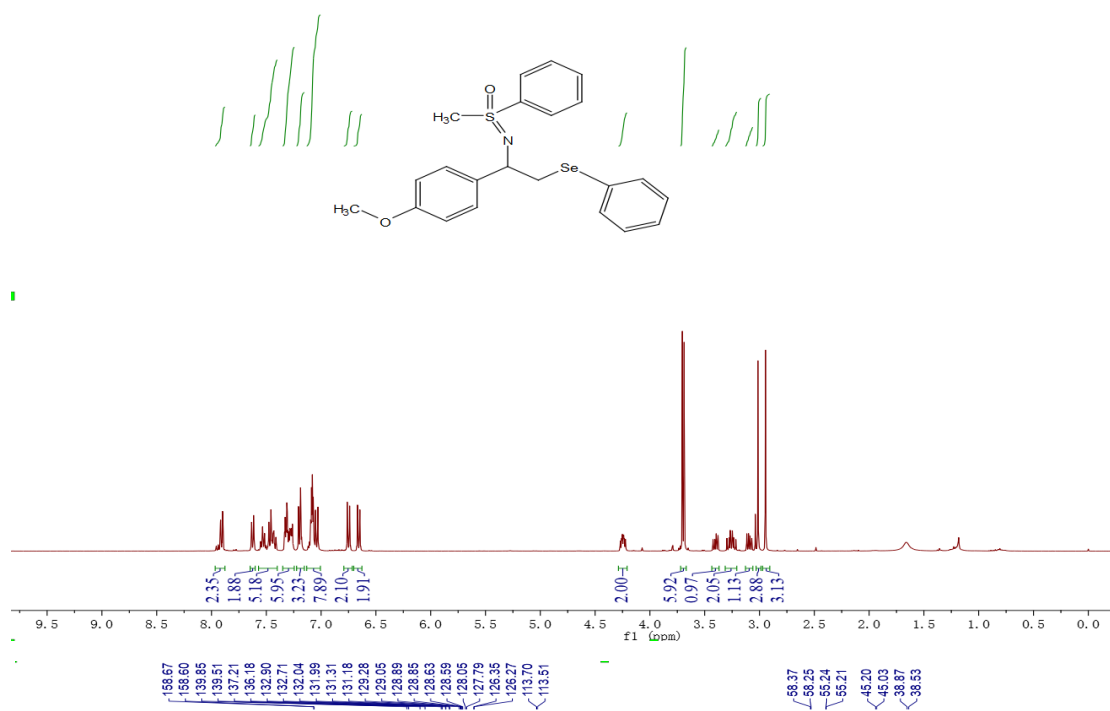
UK-24L-CHY-1-C13-CDCl3-20200925.20.fid



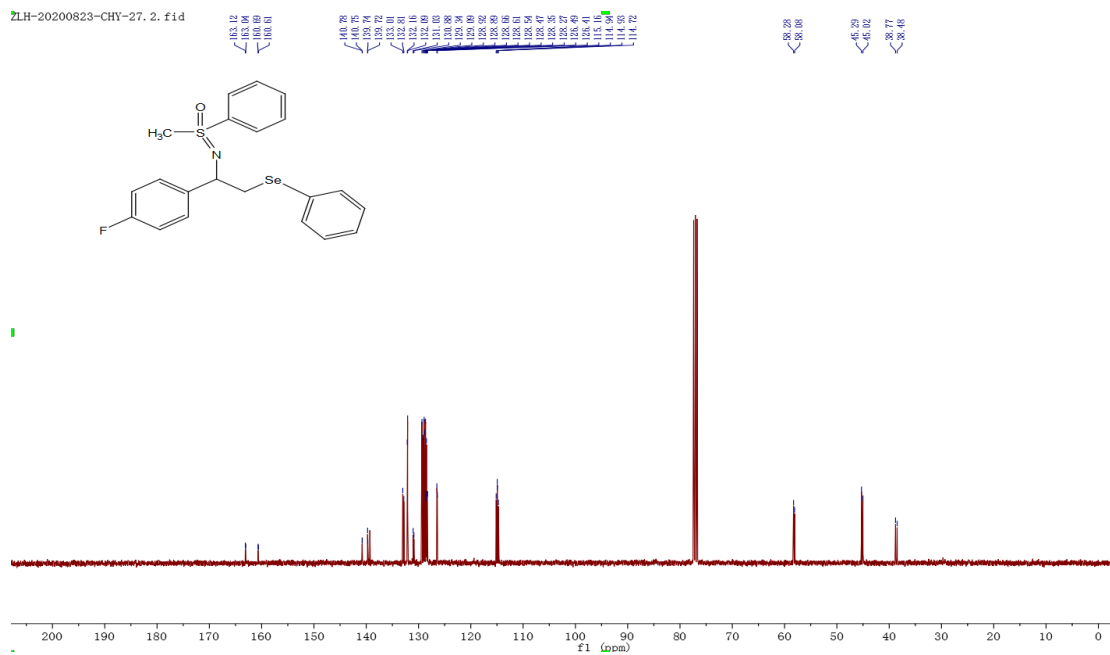
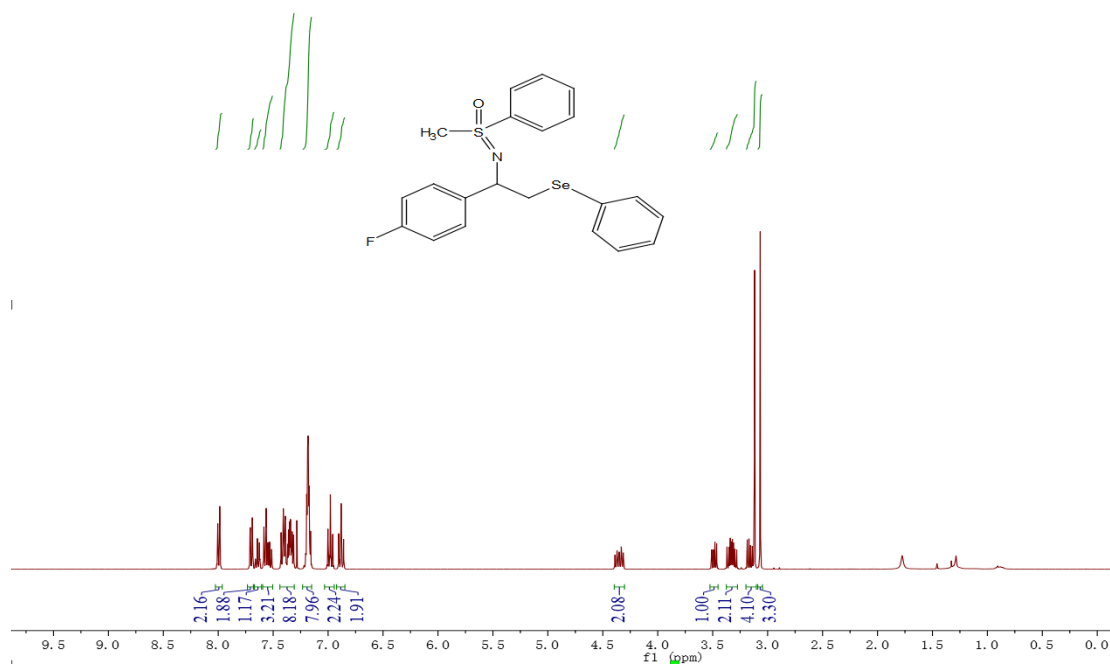
Methyl(phenyl)((2-(phenylselanyl)-1-(p-tolyl)ethyl)imino)-λ⁶-sulfanone (4b)

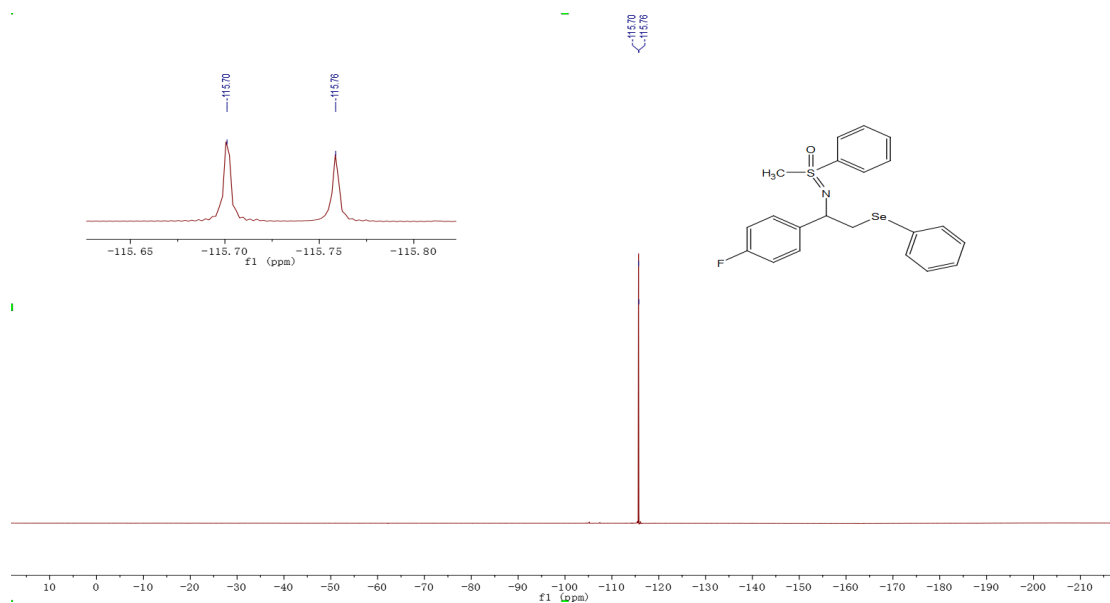


((1-(4-methoxyphenyl)-2-(phenylselanyl)ethyl)imino)(methyl)(phenyl)-I6-sulfanone (4c)



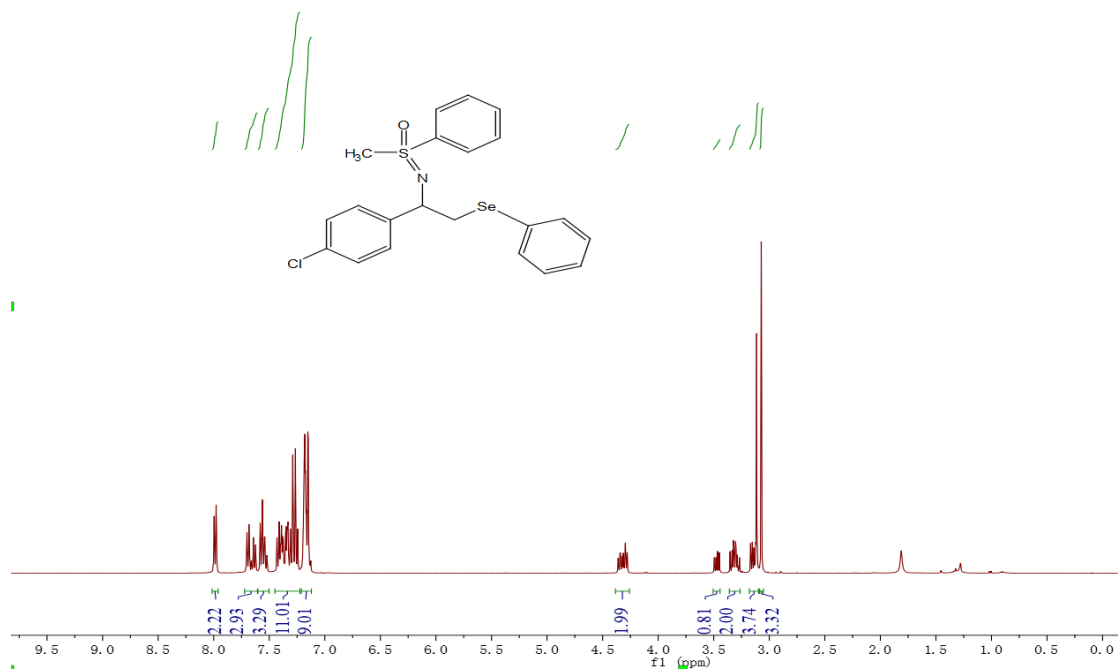
((1-(4-fluorophenyl)-2-(phenylselanyl)ethyl)imino)(methyl)(phenyl)-16-sulfanone
(4d)



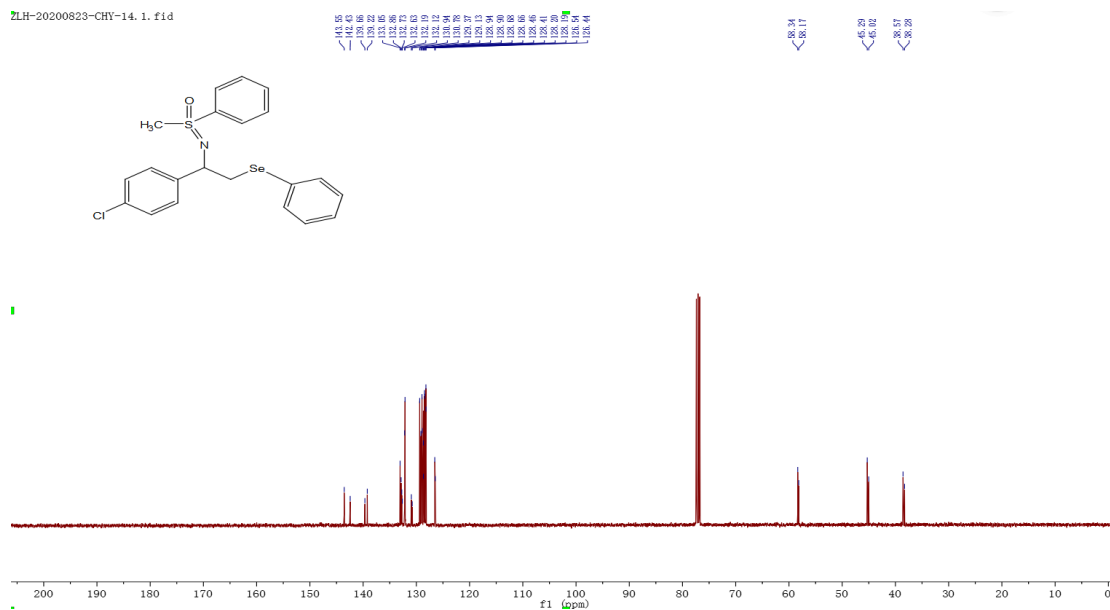


((1-(4-chlorophenyl)-2-(phenylselanyl)ethyl)imino)(methyl)(phenyl)-λ⁶-sulfanone (4e)

MS-QIL-CHY-14-H1-CDC13-20200804. 20. fid

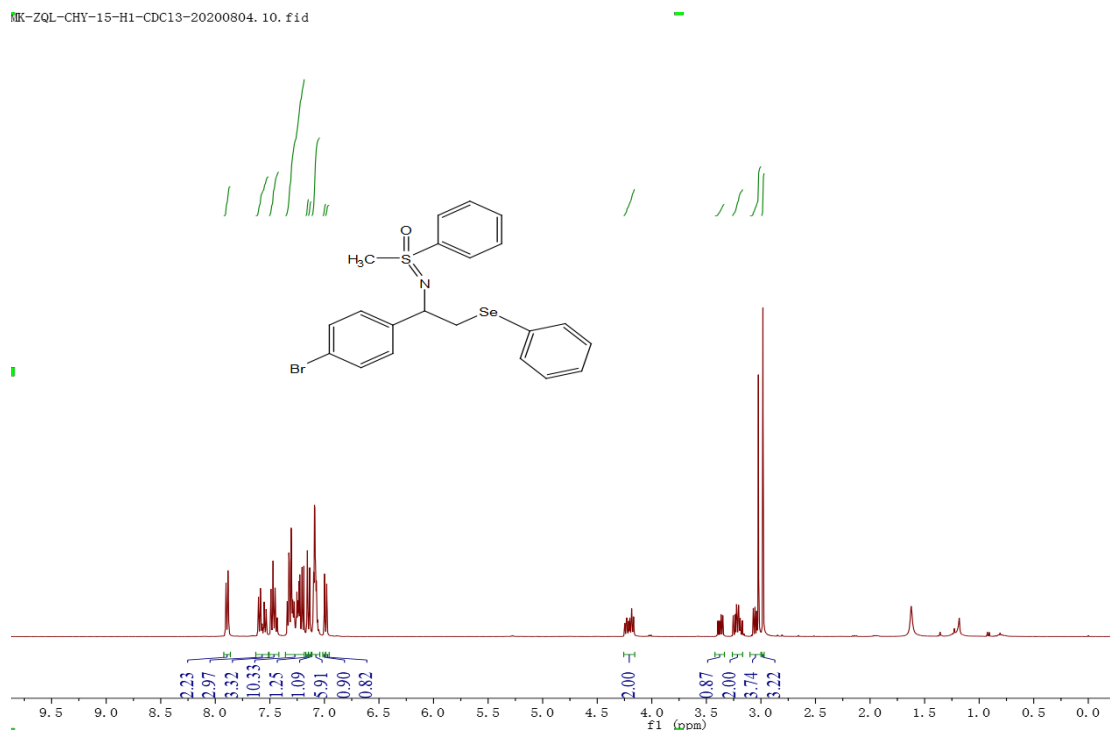


ZLH-20200823-CHY-14. 1. f1d

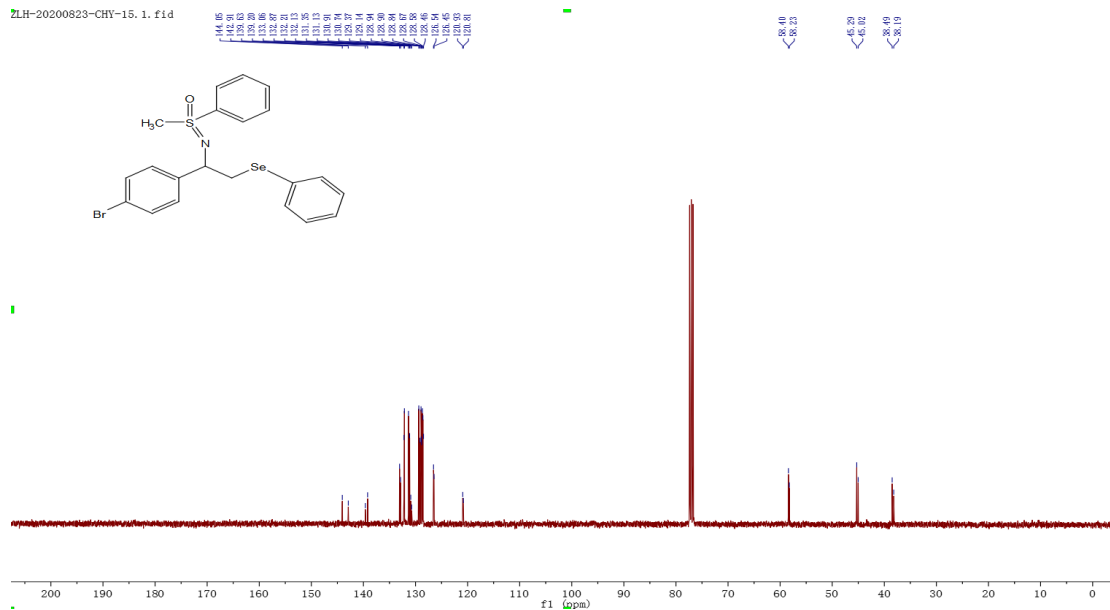


((1-(4-bromophenyl)-2-(phenylselanyl)ethyl)imino)(methyl)(phenyl)-λ⁶-sulfanone (4f)

MR-ZQL-CHY-15-H1-CDC13-20200804. 10. f1d

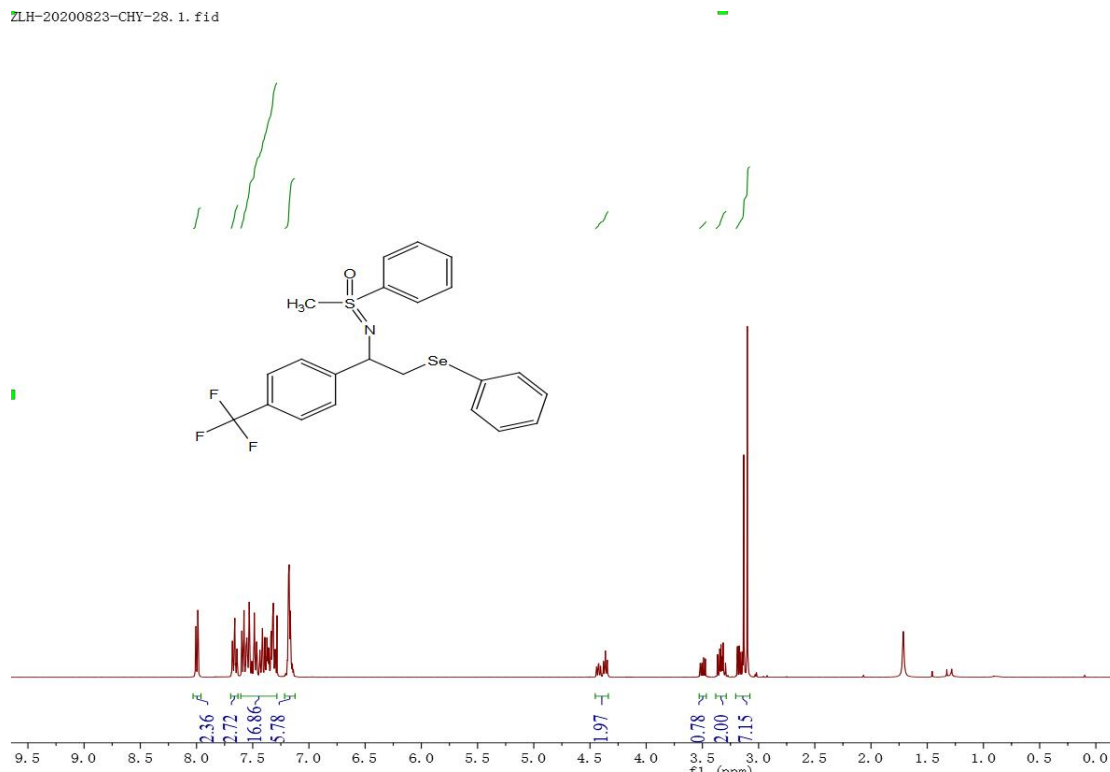


ZLH-20200823-CHY-15. 1. fid

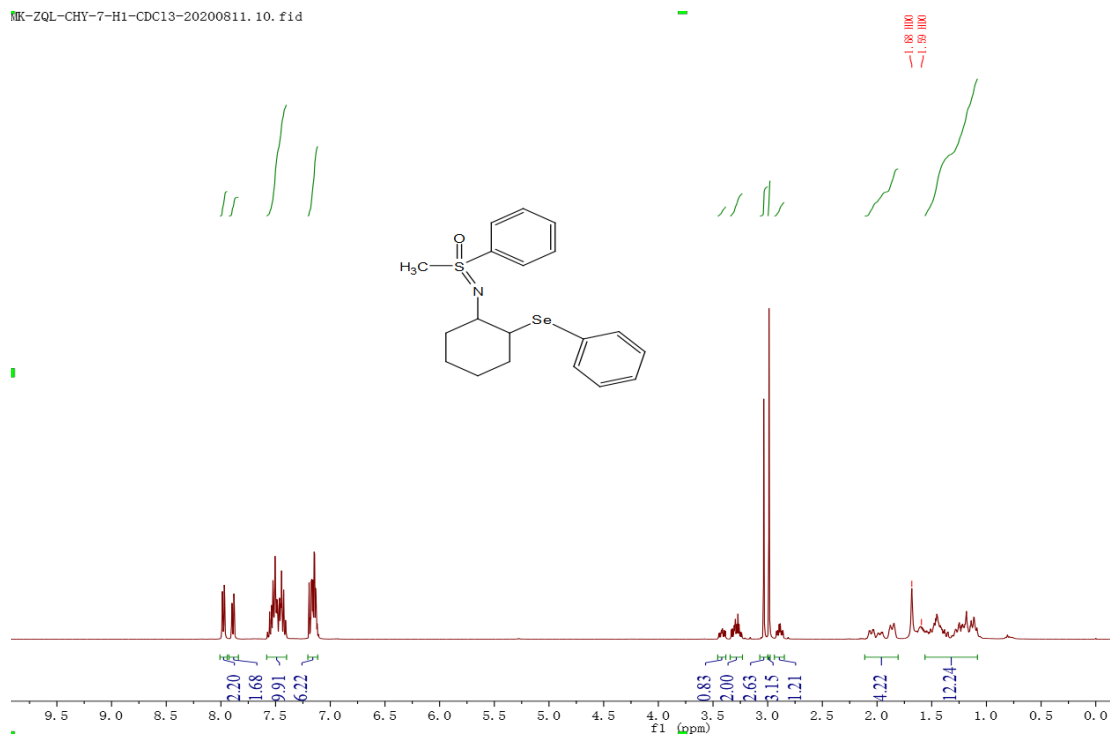


Methyl(phenyl)((2-(phenylselanyl)-1-(4-(trifluoromethyl)phenyl)ethyl)imino)-λ⁶-sulfanone (4g)

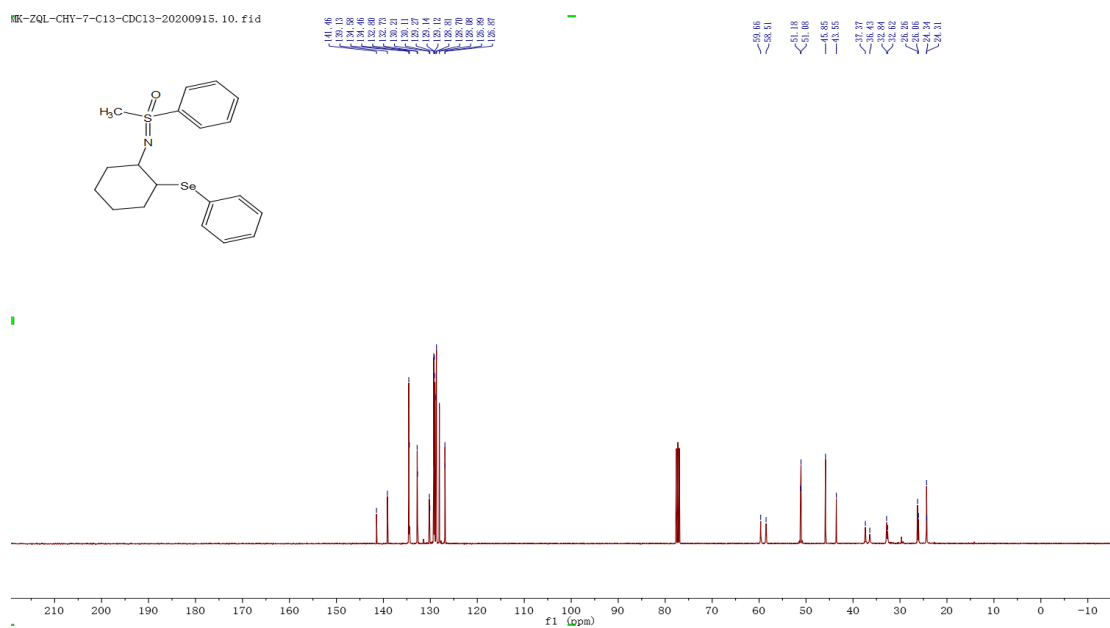
ZLH-20200823-CHY-28. 1. fid



MK-ZQL-CHY-7-H1-CDC13-20200811. 10. f1d

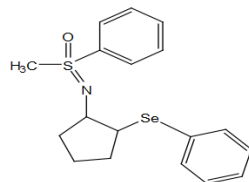
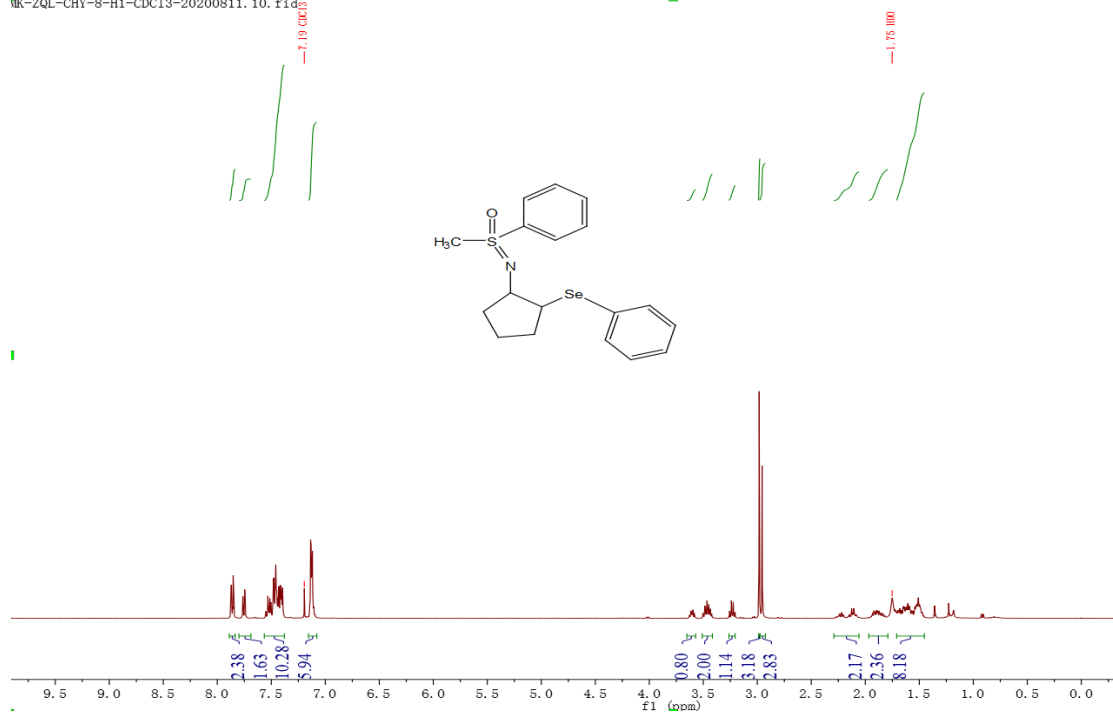


MK-ZQL-CHY-7-C13-CDC13-20200915. 10. f1d

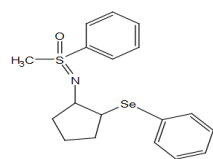
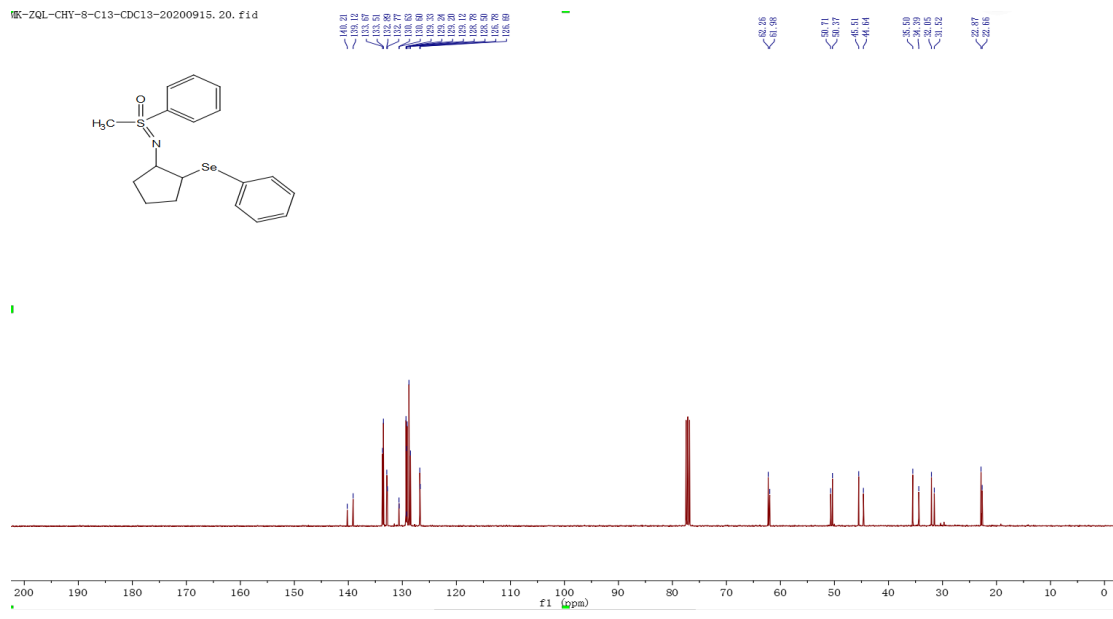


Methyl(phenyl)((2-(phenylselanyl)cyclopentyl)imino)-λ⁶-sulfanone (4i)

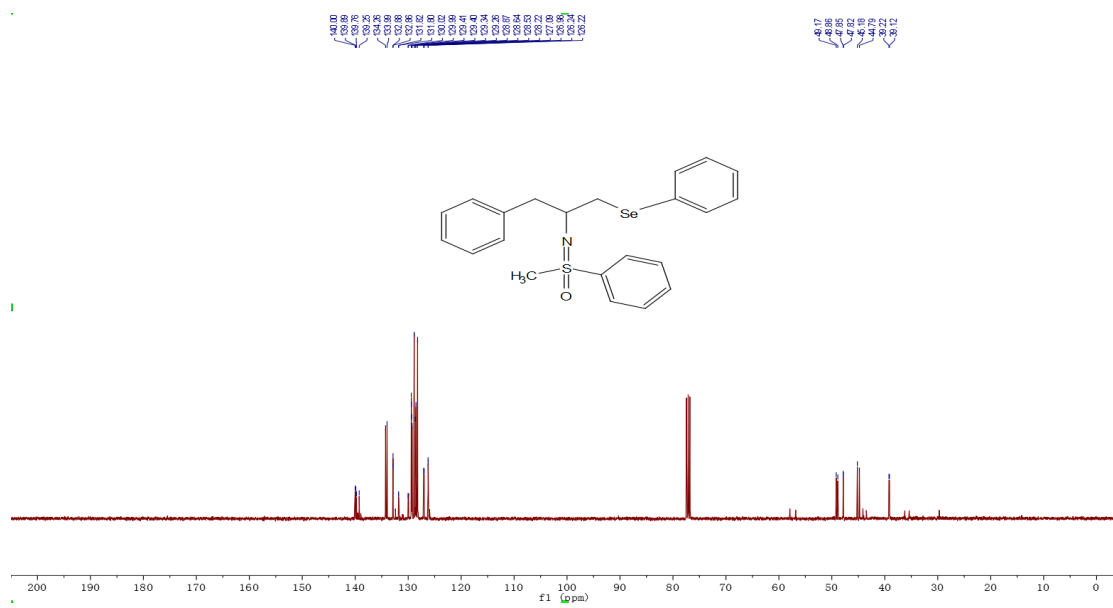
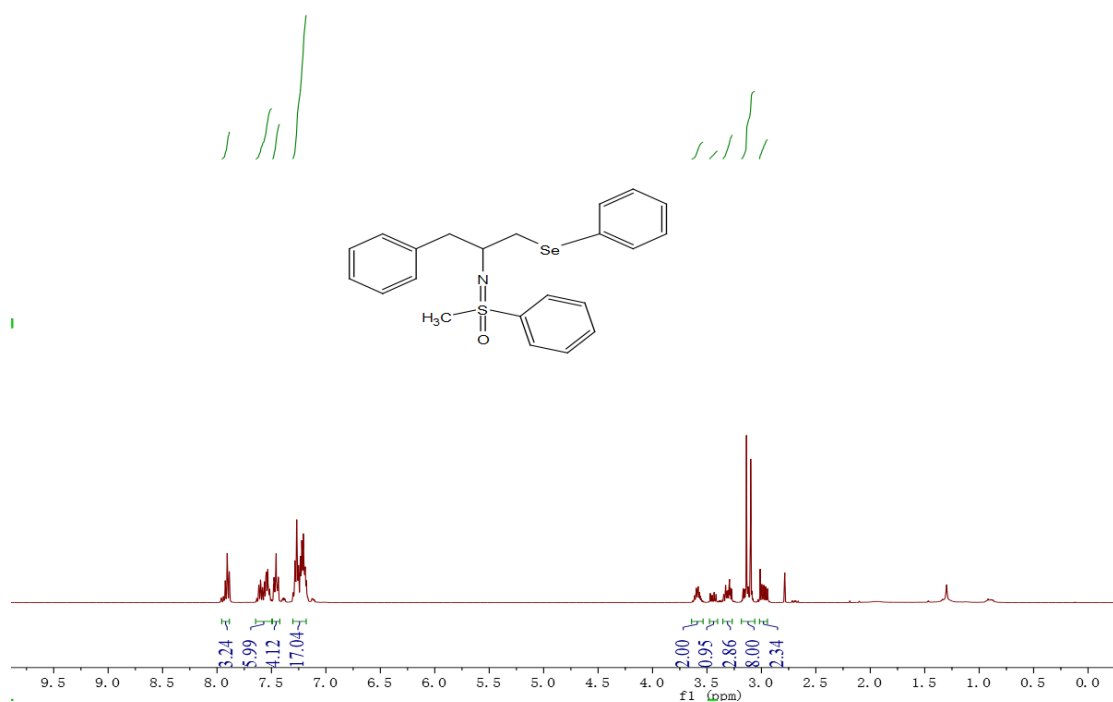
MS-ZQL-CHY-8-H1-CDC13-20200811.10.fid



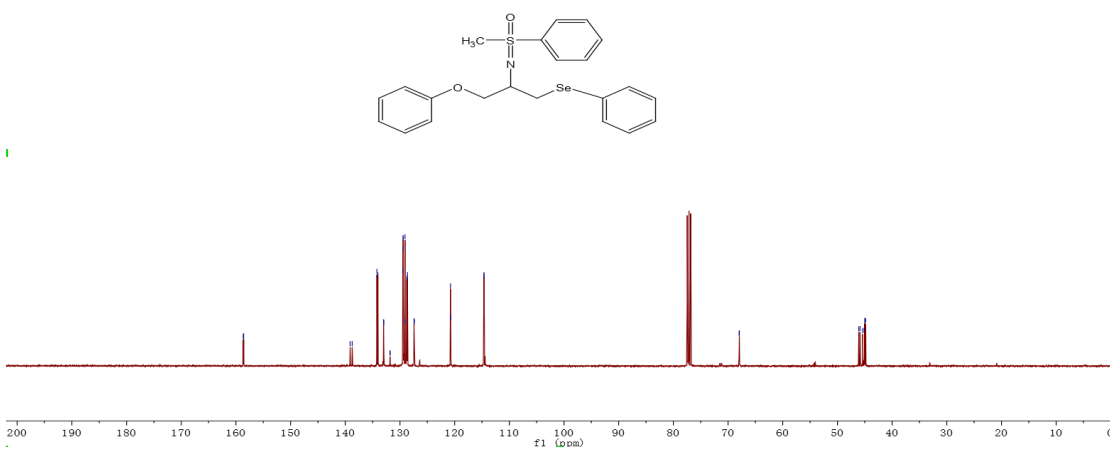
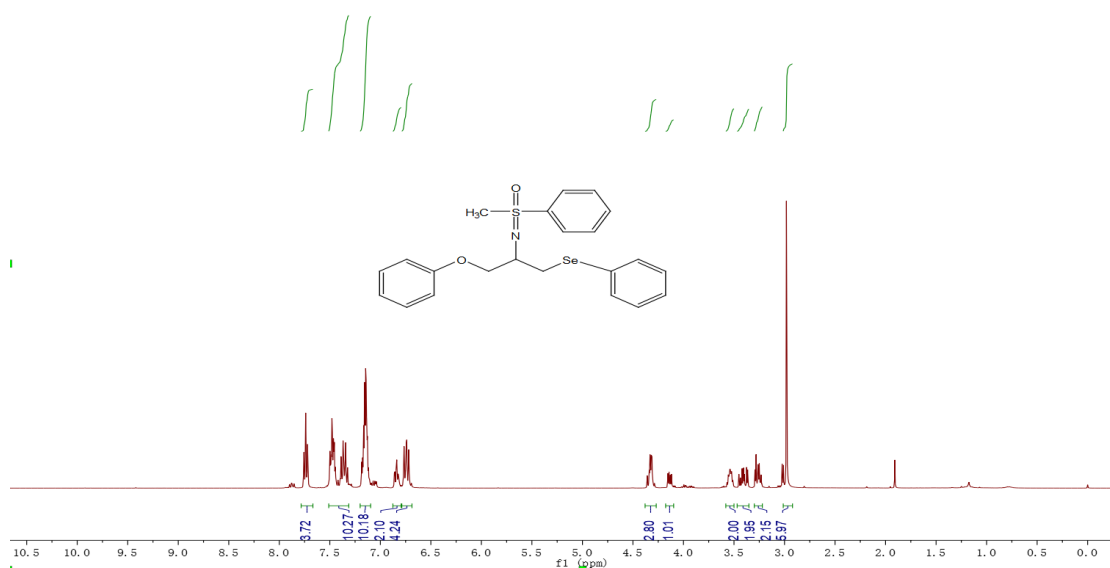
MS-ZQL-CHY-8-C13-CDC13-20200915.20.fid



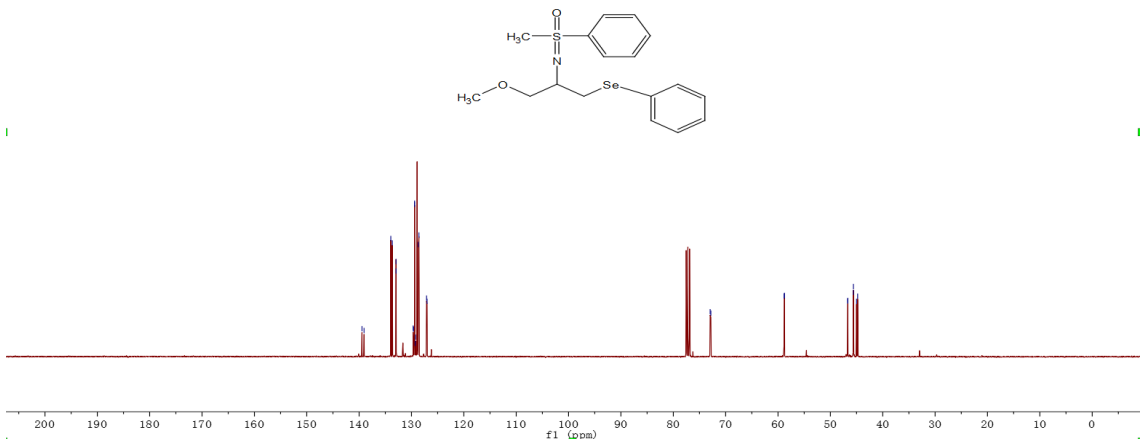
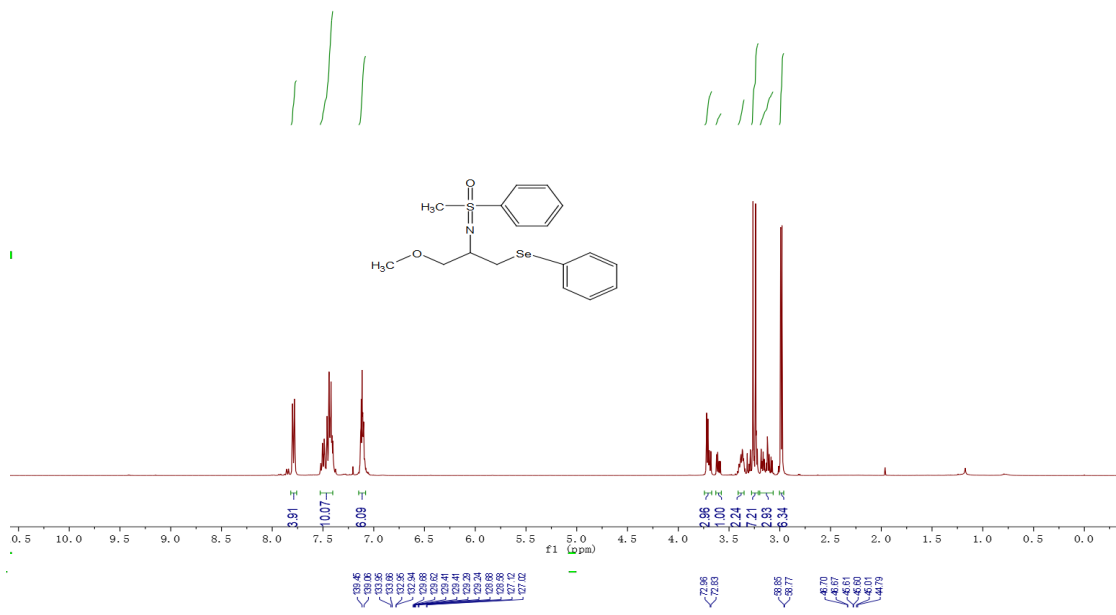
Methyl(phenyl)((1-phenyl-3-(phenylselanyl)propan-2-yl)imino)-λ⁶-sulfanone (4j)



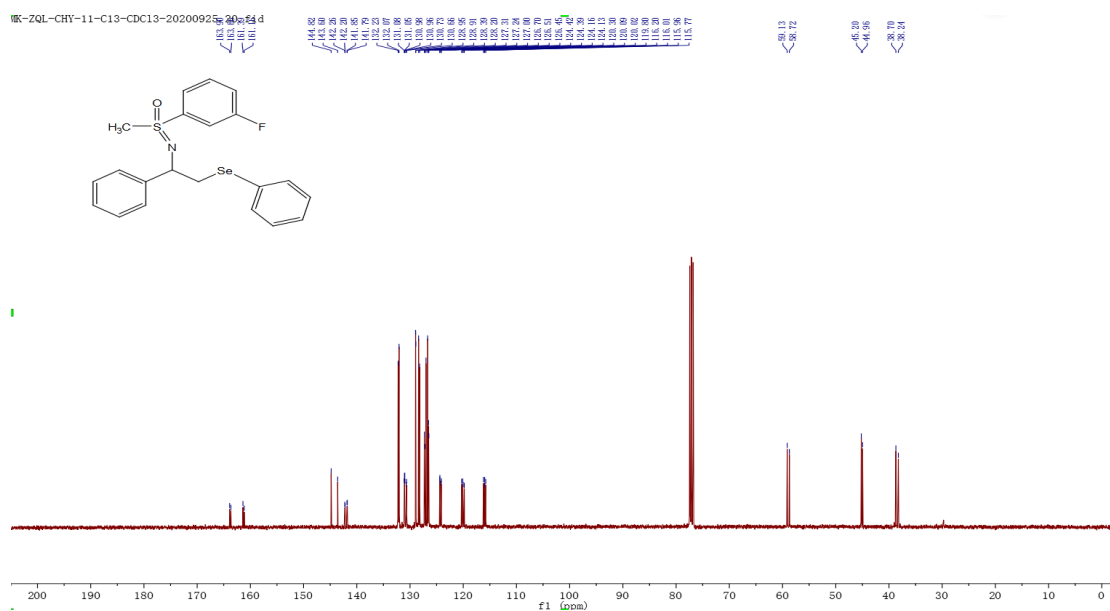
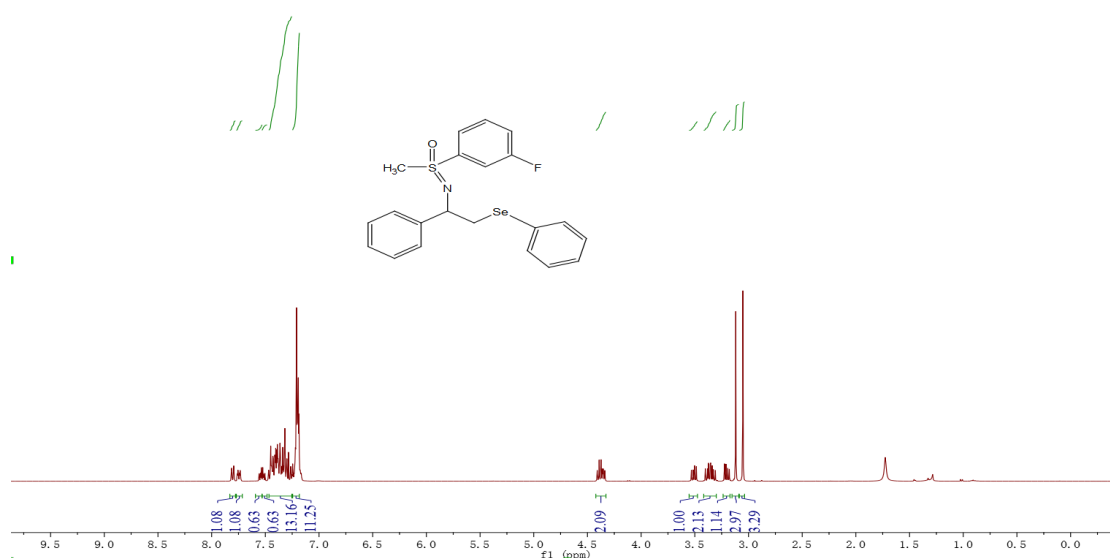
Methyl((1-phenoxy-3-(phenylselanyl)propan-2-yl)imino)(phenyl)- λ⁶-sulfanone (4k)



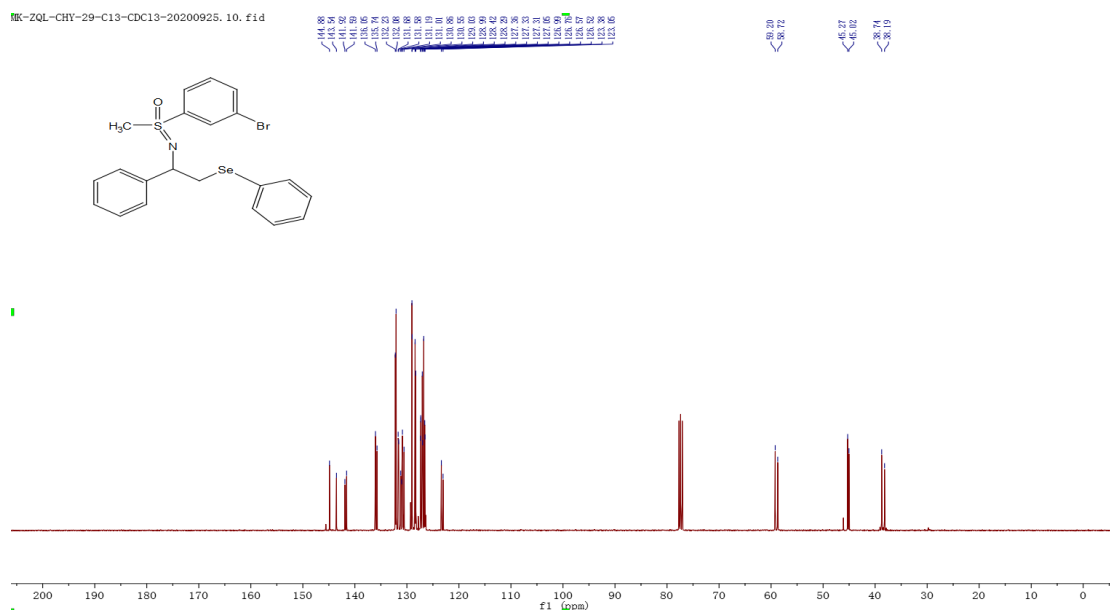
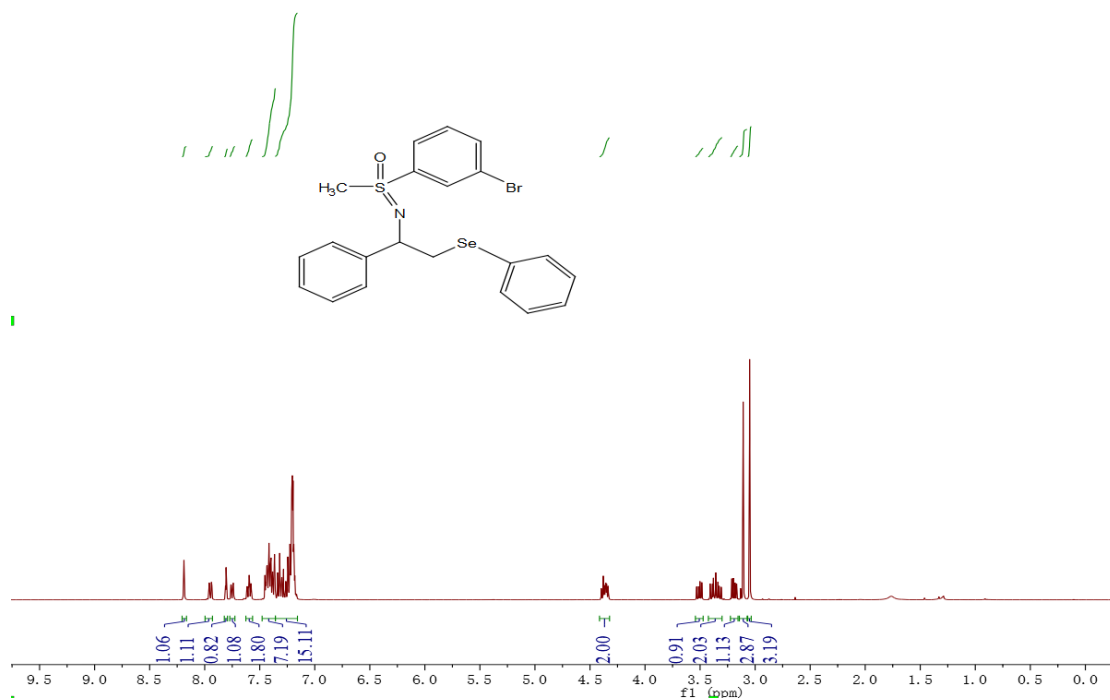
((1-methoxy-3-(phenylselanyl)propan-2-yl)imino)(methyl)(phenyl)-λ⁶-sulfanone (4I)



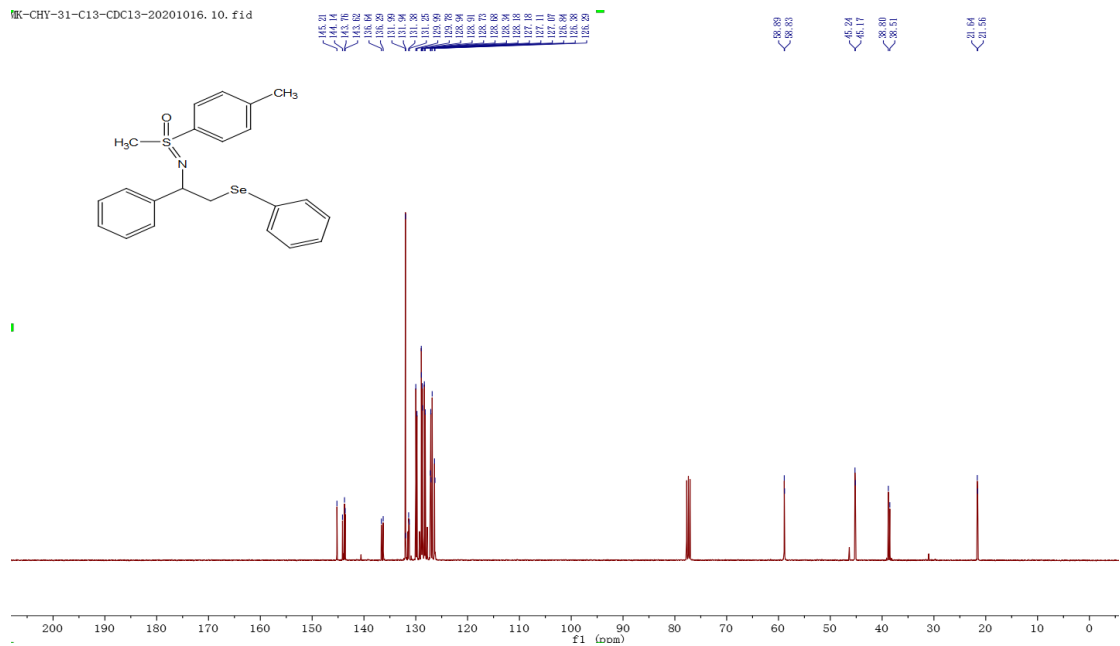
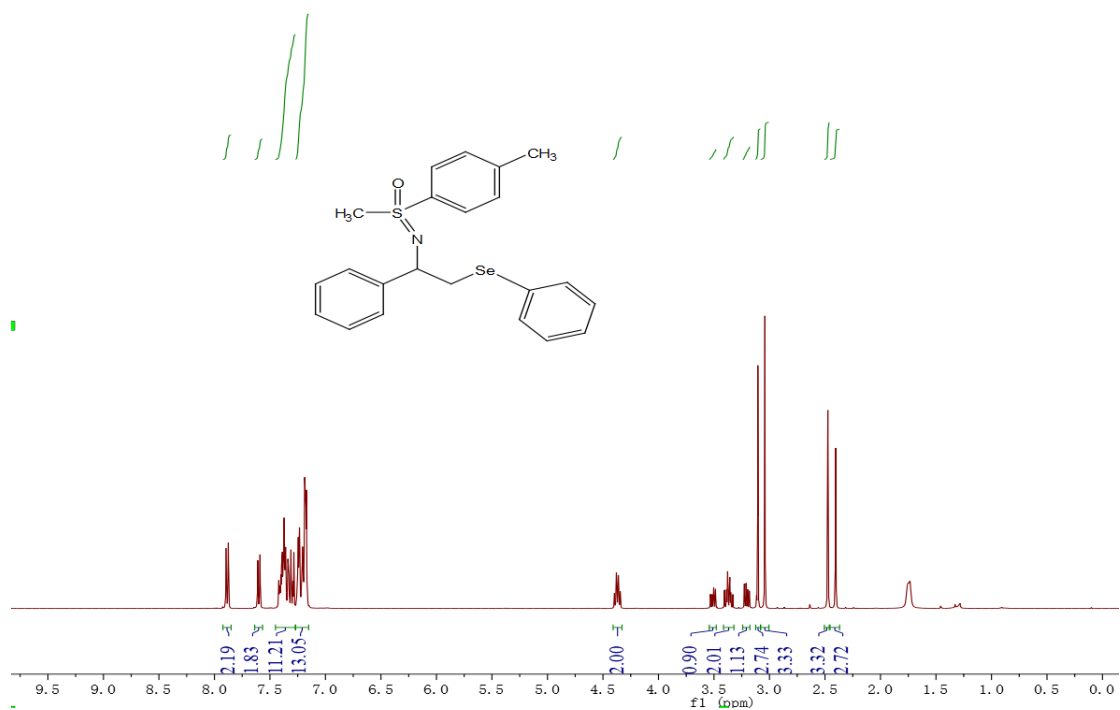
(3-fluorophenyl)(methyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanon (4m)



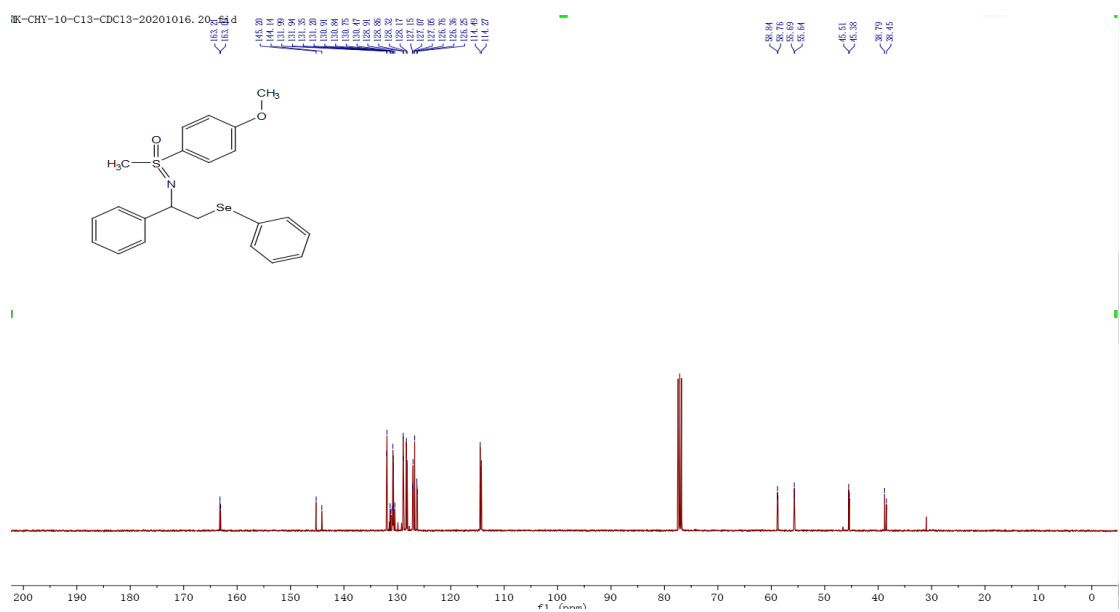
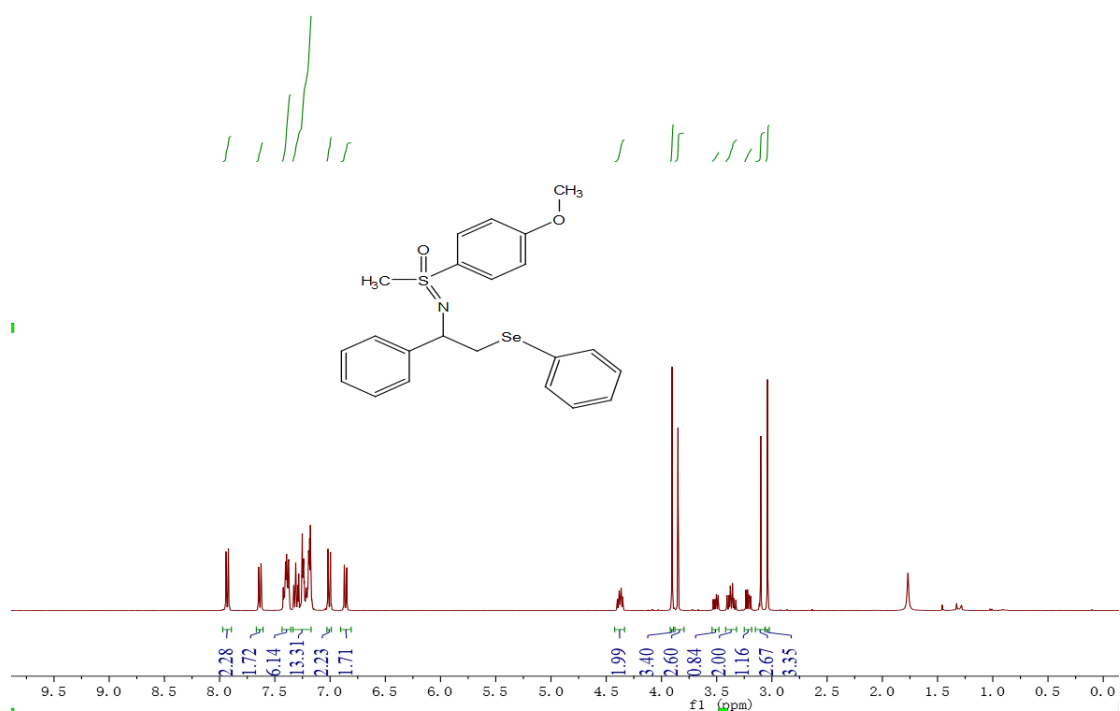
Methyl(4-nitrophenyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanone (4n)



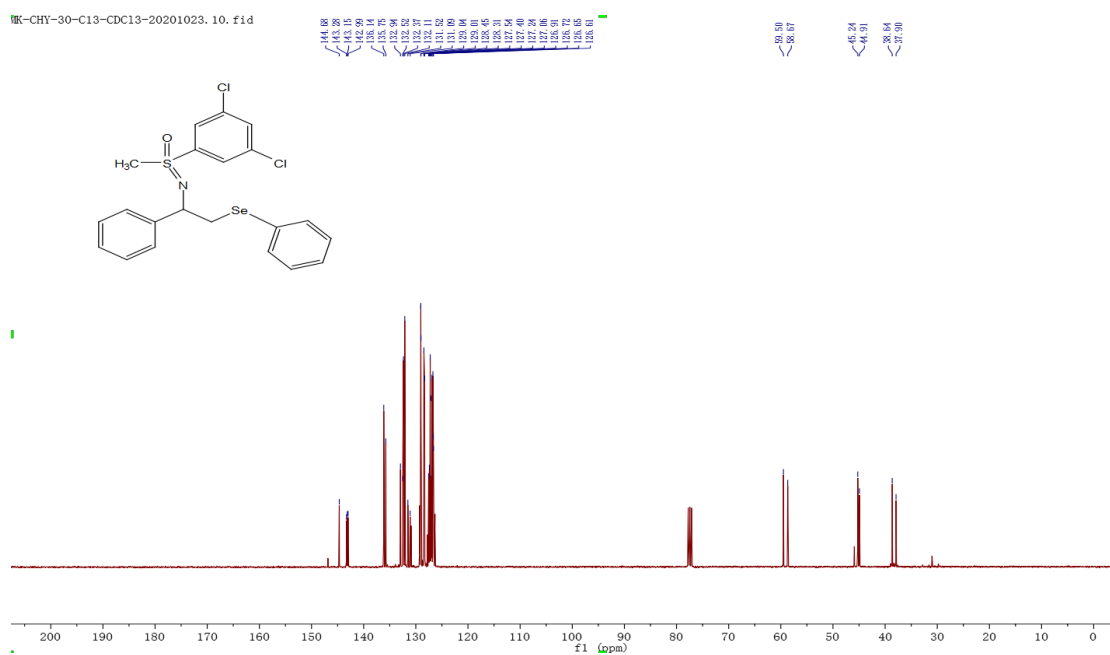
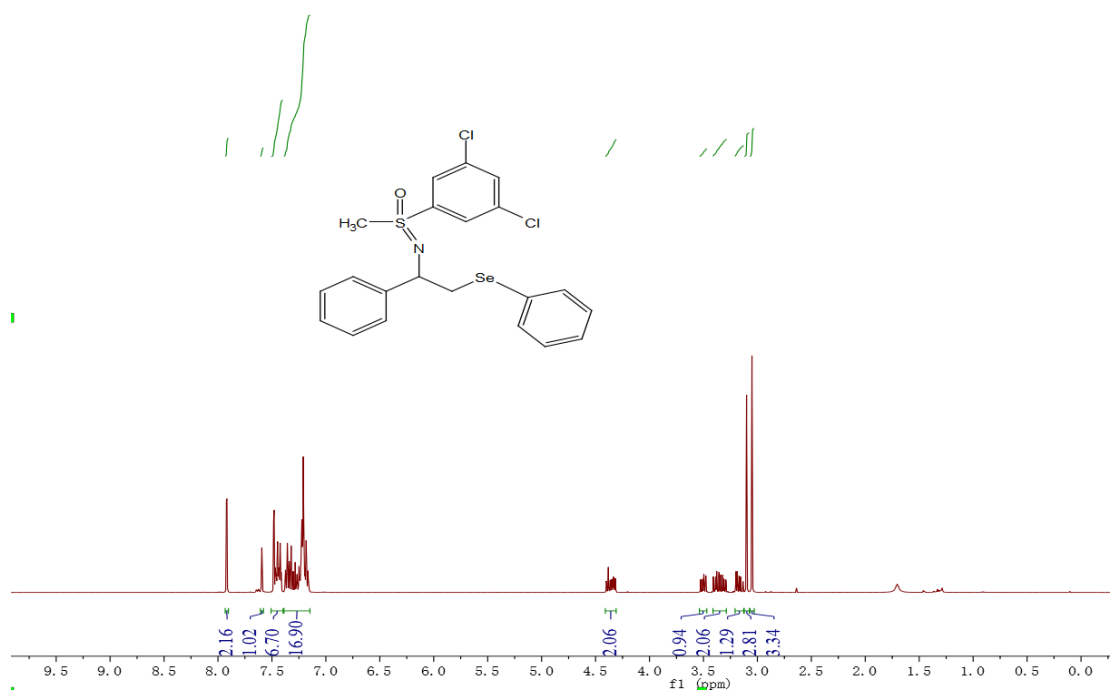
Methyl((1-phenyl-2-(phenylselanyl)ethyl)imino)(p-tolyl)-λ⁶-sulfanone (4p)



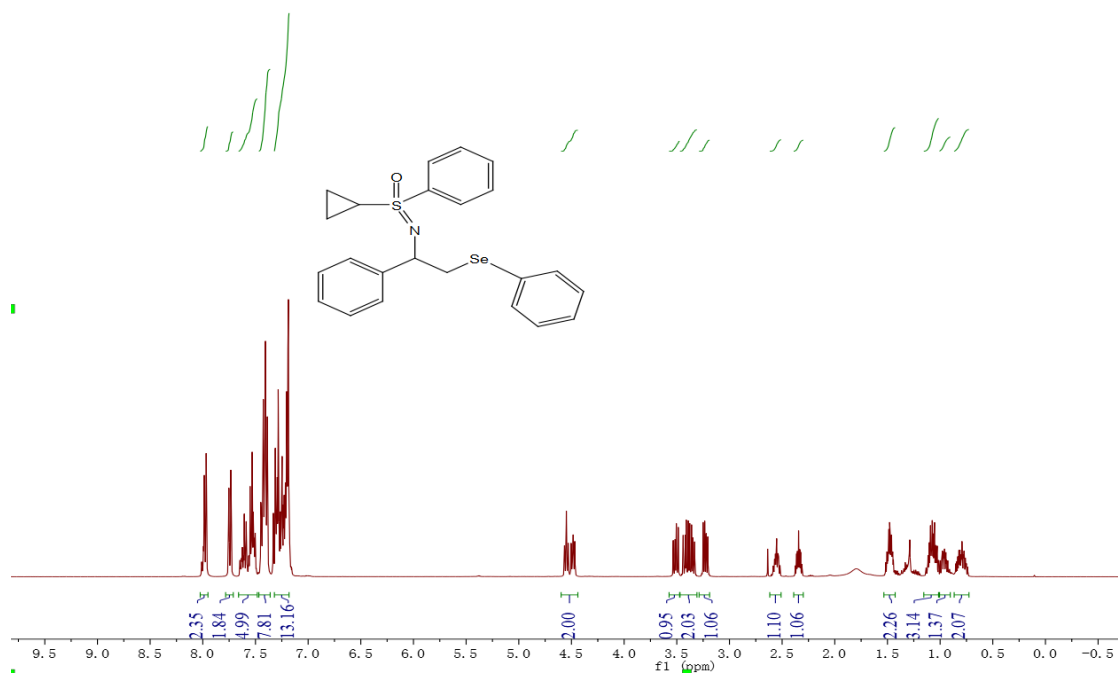
(4-methoxyphenyl)(methyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)-λ⁶-sulfanone (4q)



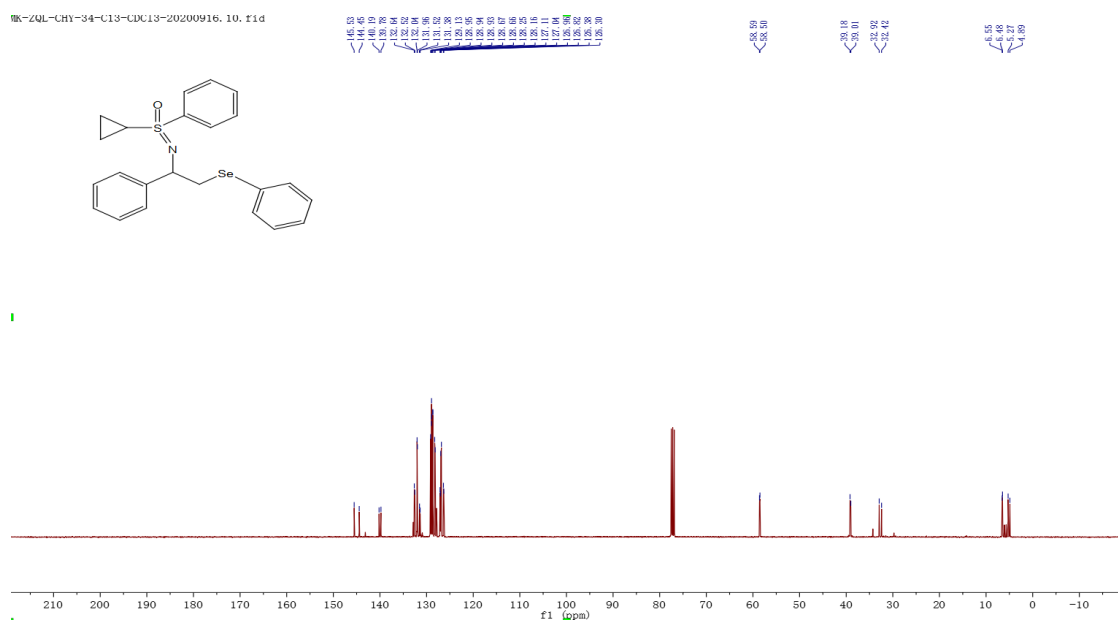
(3,5-dichlorophenyl)(methyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)-λ⁶-sulfanone (4r)



Cyclopropyl(phenyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanone (4s)

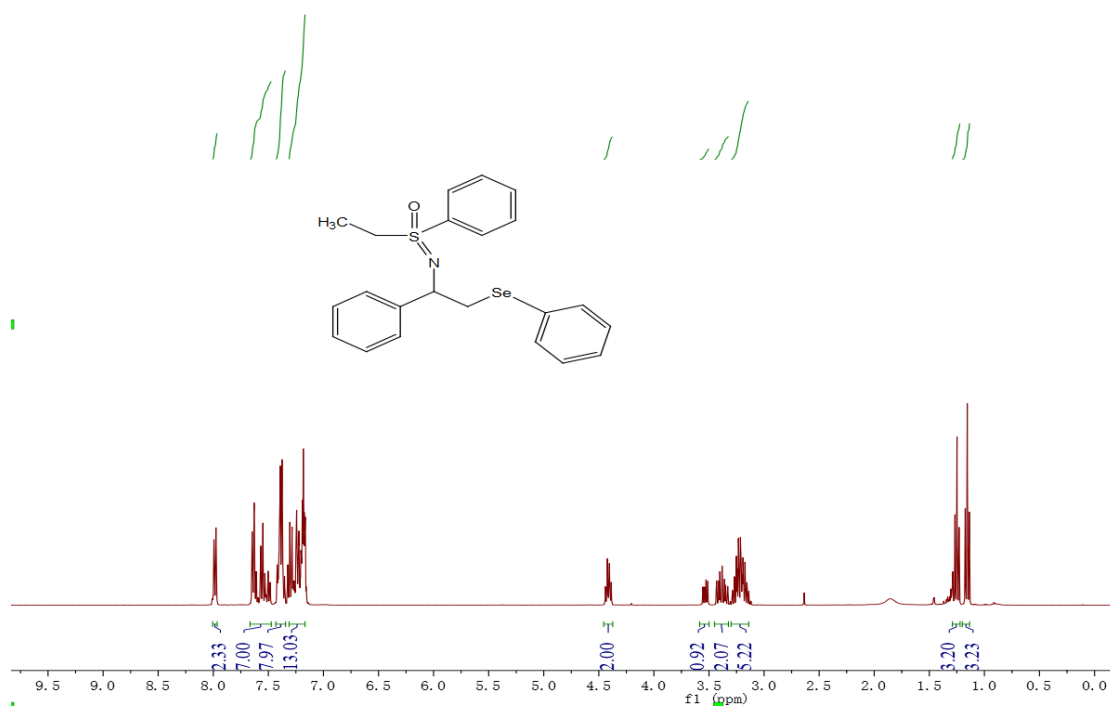


MR-ZQL-CHY-34-C13-CDC13-20200916. 10. F1d

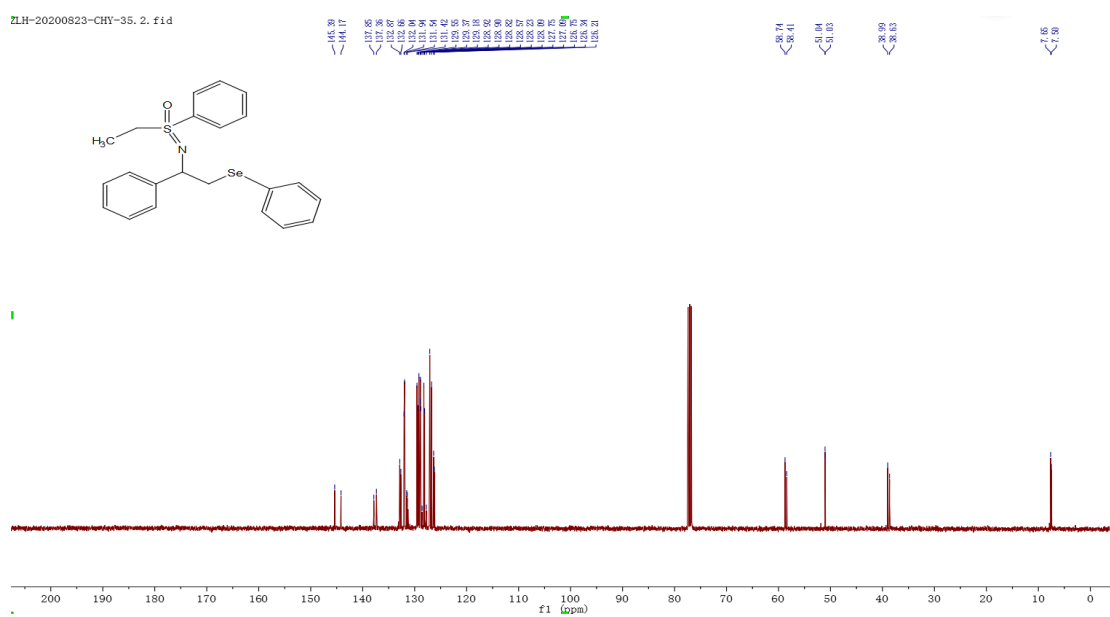


Ethyl(phenyl)((1-phenyl-2-(phenylselanyl)ethyl)imino)- λ^6 -sulfanone (4t)

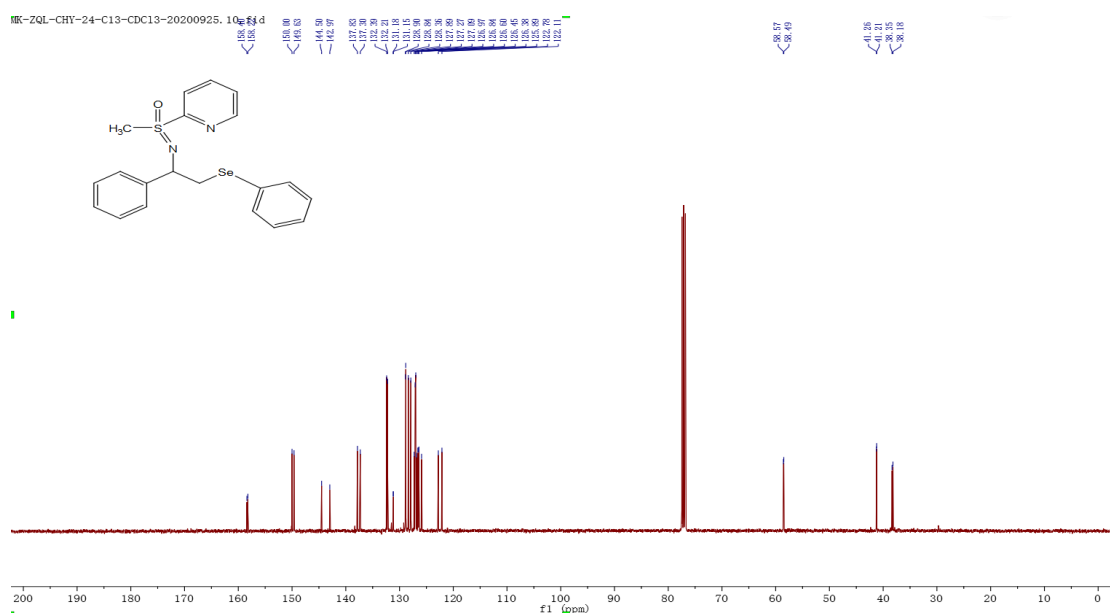
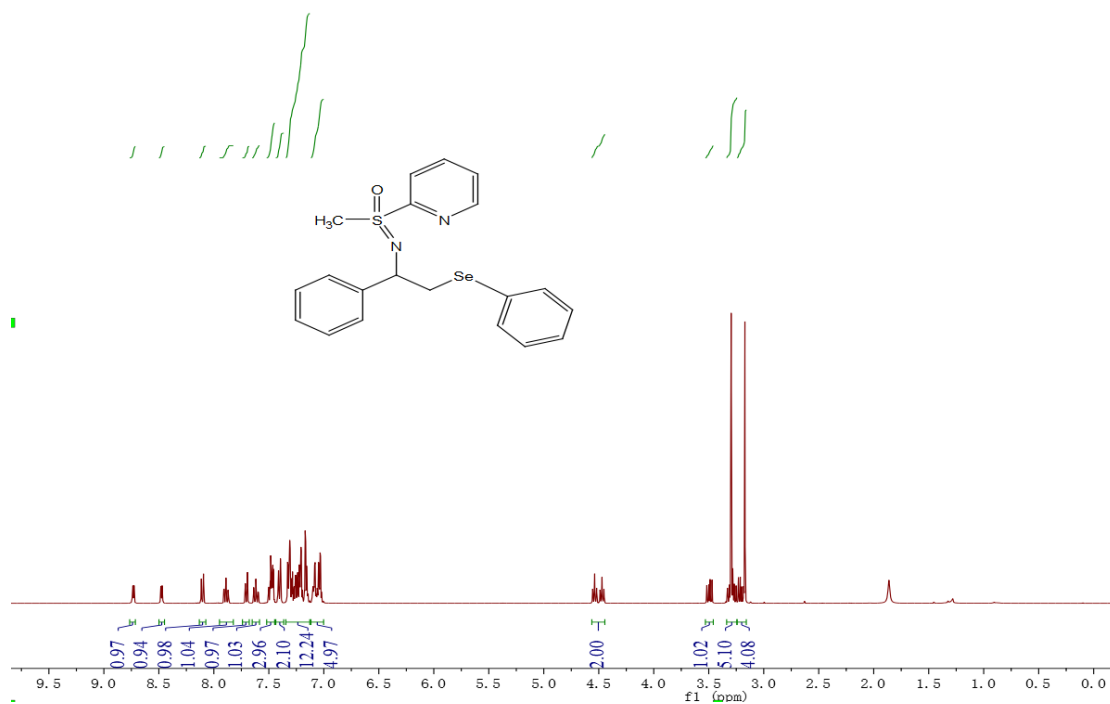
ZLH-20200823-CHY-35. 1. f1d



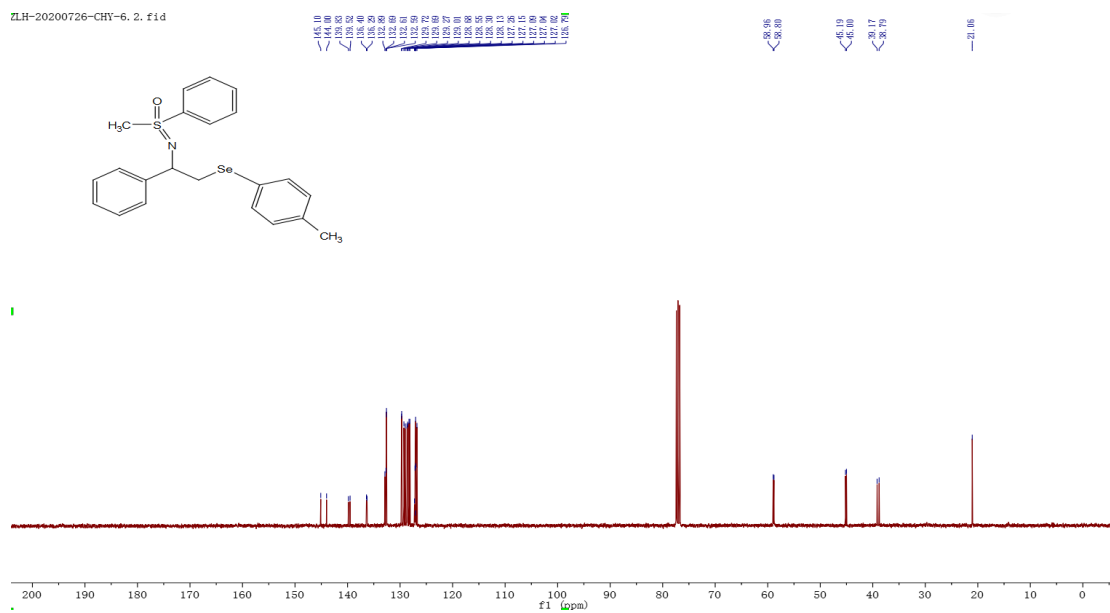
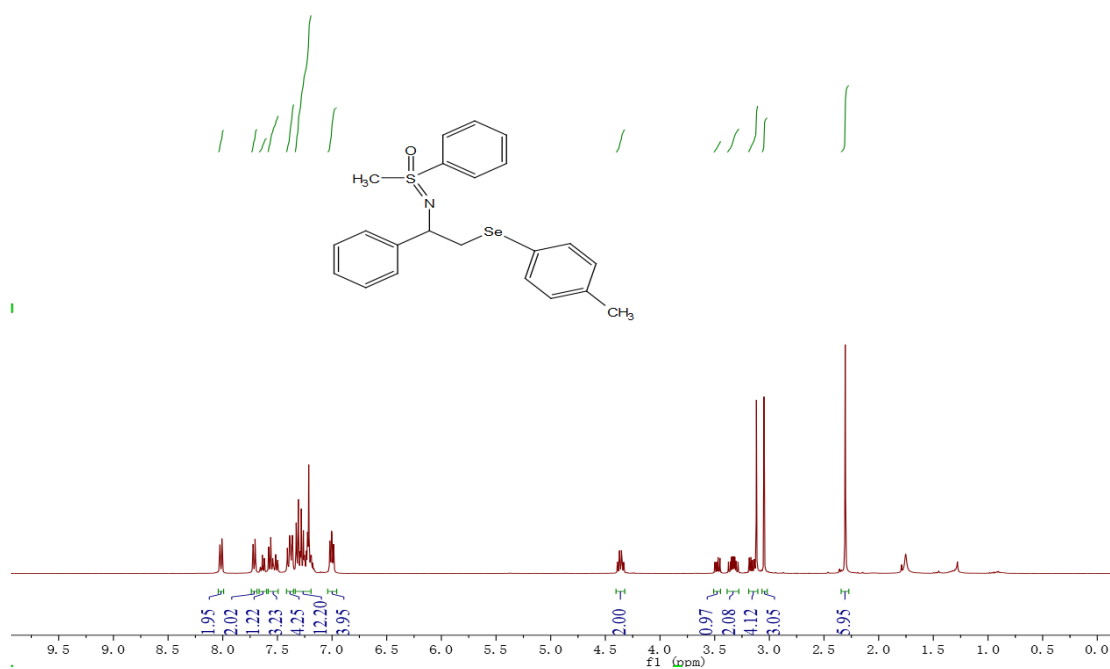
ZLH-20200823-CHY-35. 2. f1d



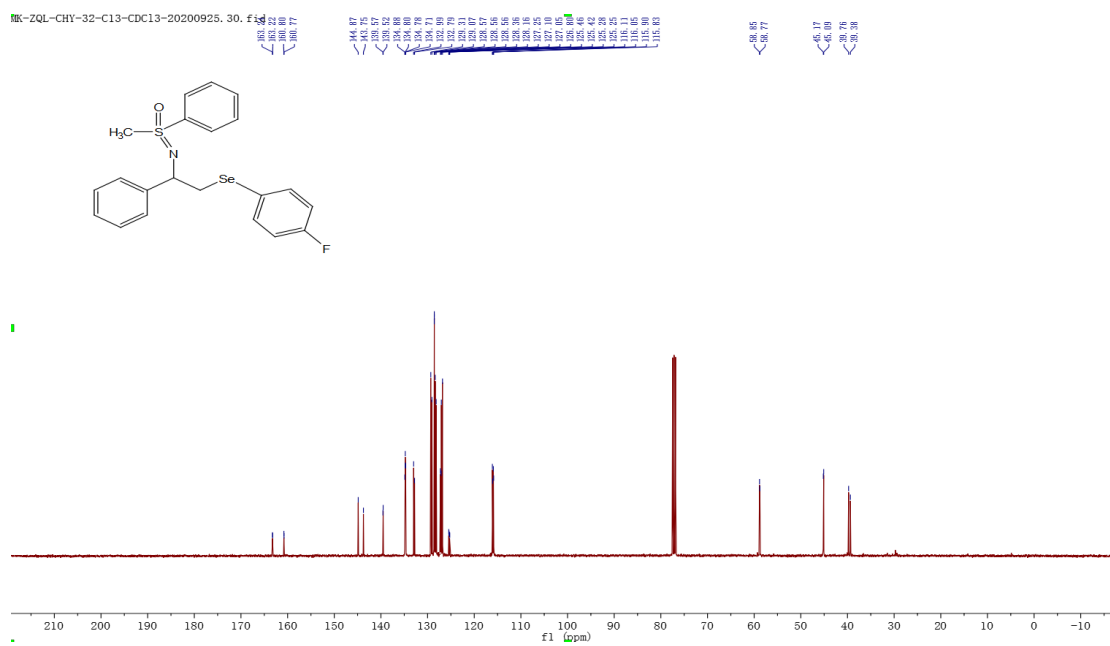
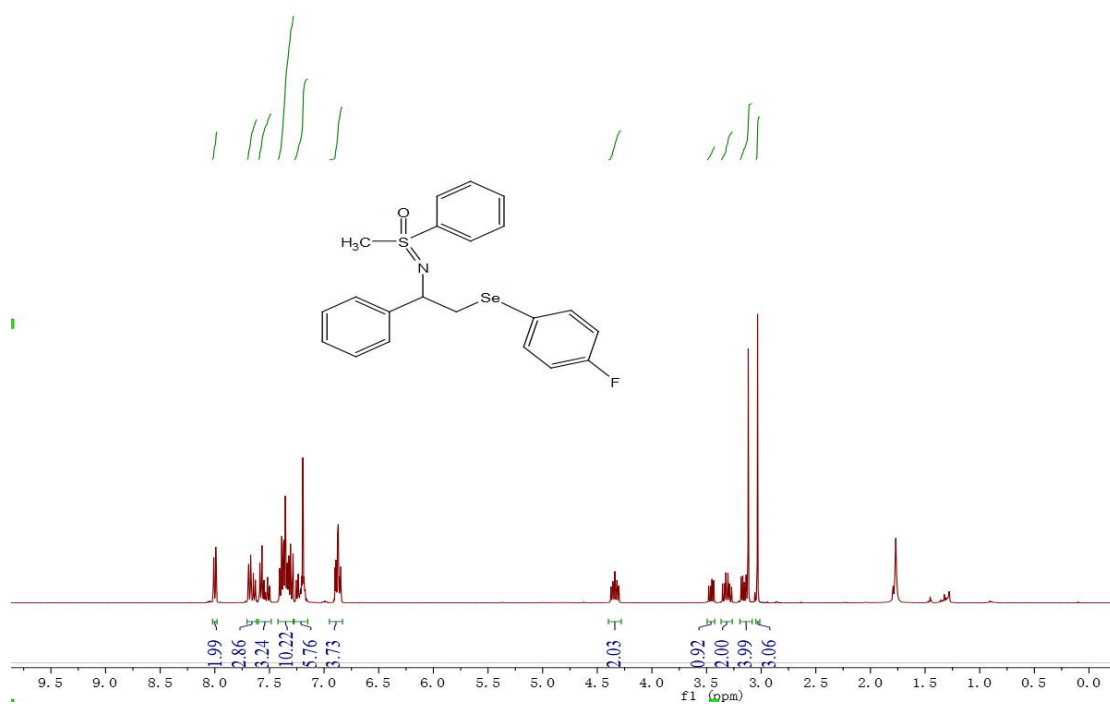
Methyl((1-phenyl-2-(phenylselanyl)ethyl)imino)(pyridin-2-yl)-λ⁶-sulfanone (4u)

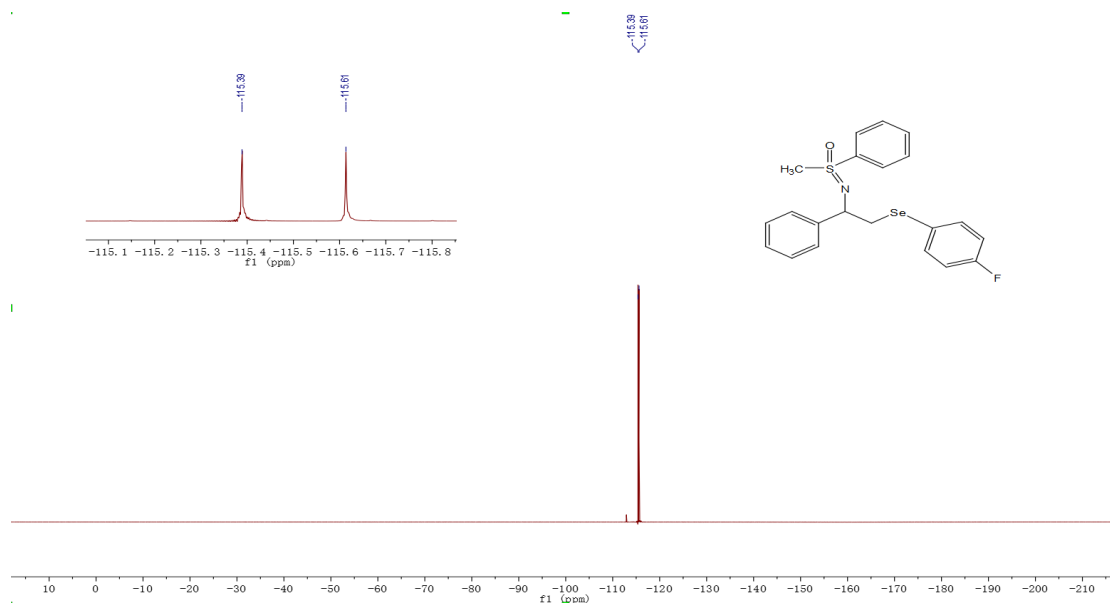


Methyl(phenyl)((1-phenyl-2-(p-tolylselanyl)ethyl)imino)-λ⁶-sulfanone (4v)



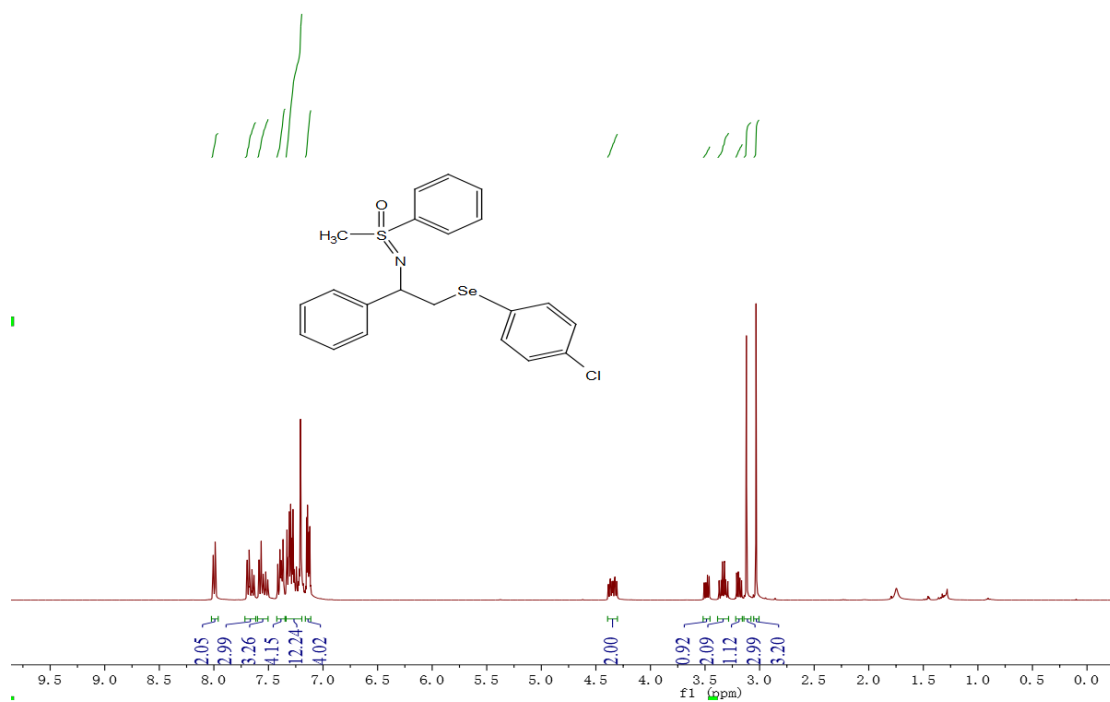
((2-((4-methoxyphenyl)selenanyl)-1-phenylethyl)imino)(methyl)(phenyl)-λ⁶-sulfanone (4w)



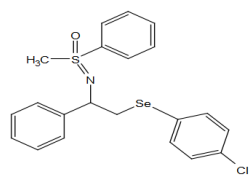


((2-((4-chlorophenyl)selenanyl)-1-phenylethyl)imino)(methyl)(phenyl)-λ⁶-sulfanone
(4y)

ZLH-20200823-CHY-33.1.fid



HK-ZQL-CHY-33-C13-CDC13-20200925. 10. f1.d



143.82
139.91
139.81
135.82
133.94
133.84
132.83
132.73
132.63
129.46
129.33
129.23
129.12
129.02
128.91
128.81
128.71
127.39
127.29
127.19
126.81
126.81

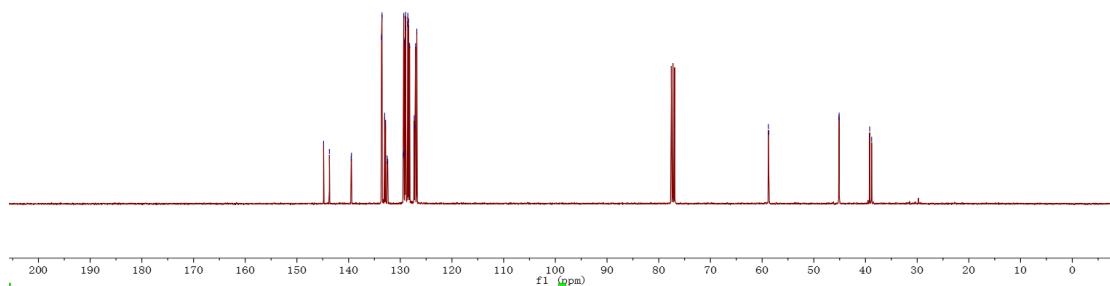
38.78

45.15

45.09

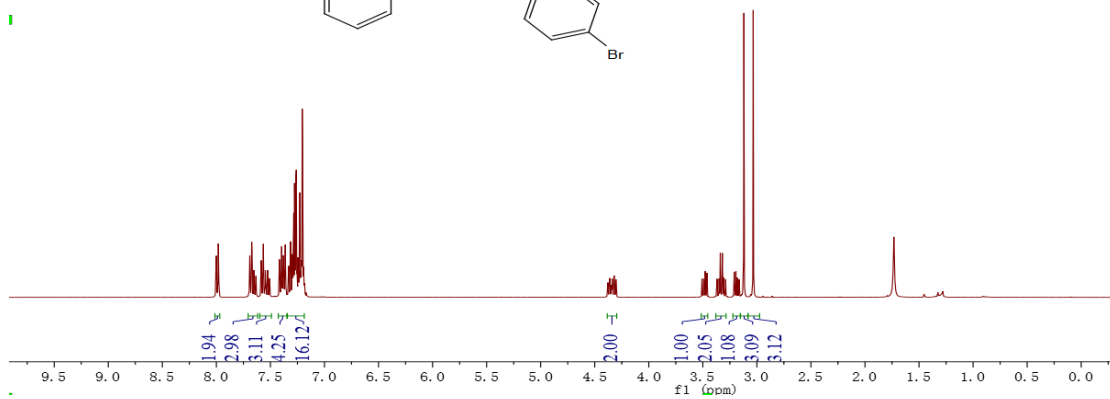
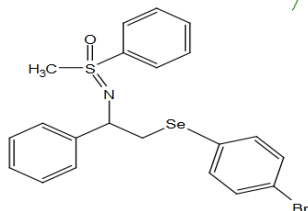
39.19

38.81

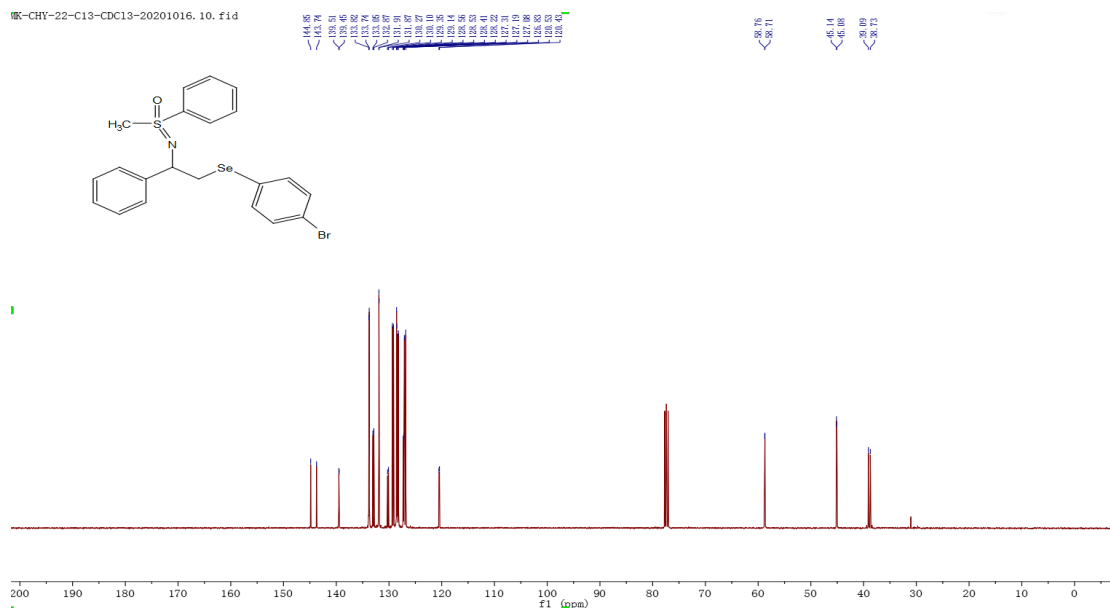


((2-((4-bromophenyl)selenyl)-1-phenylethyl)imino)(methyl)(phenyl)-λ⁶-sulfanone (4z)

ZLH-20200823-CHY-22. 1. f1.d

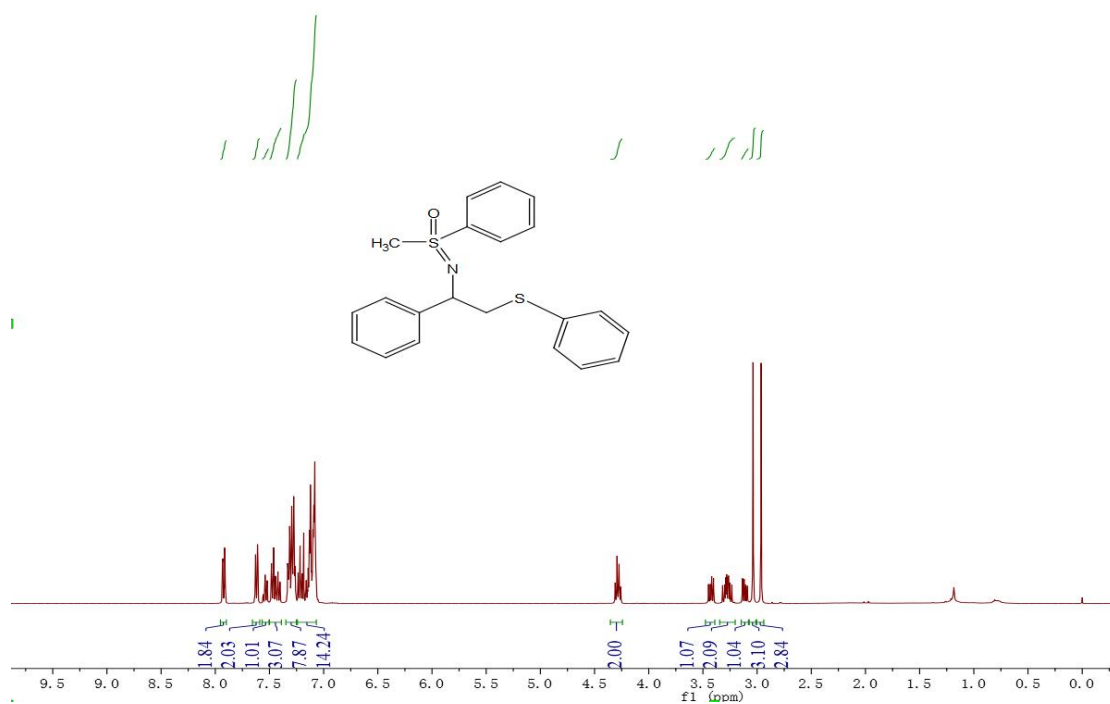


HK-CHY-22-C13-CDC13-20201016. 10. fid

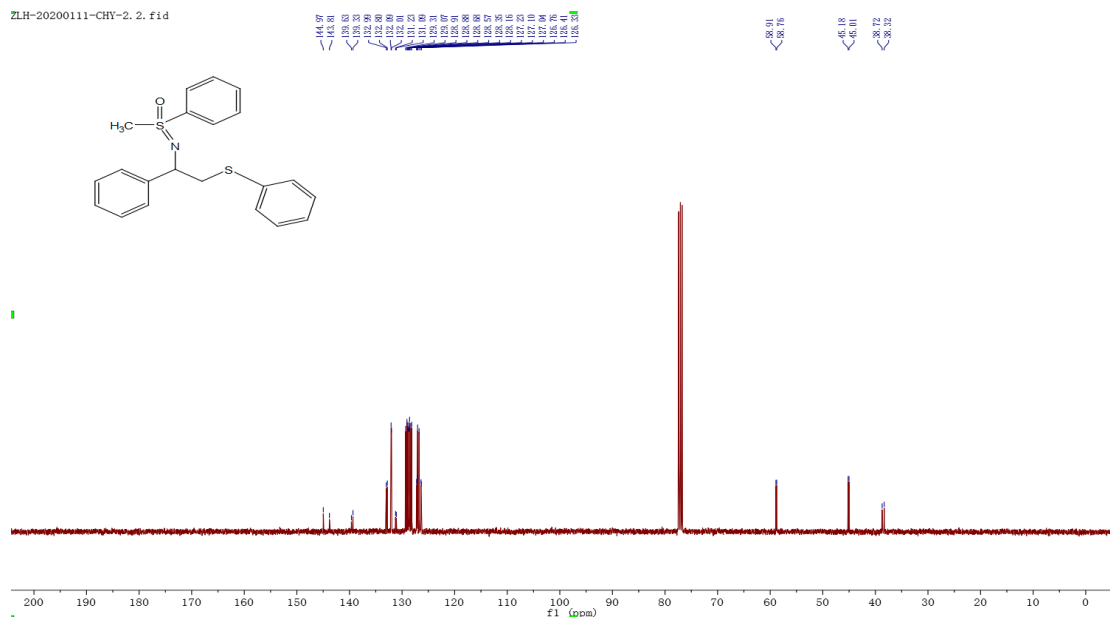


Methyl(phenyl)((1-phenyl-2-(phenylthio)ethyl)imino)- λ^6 -sulfanone (4ab)

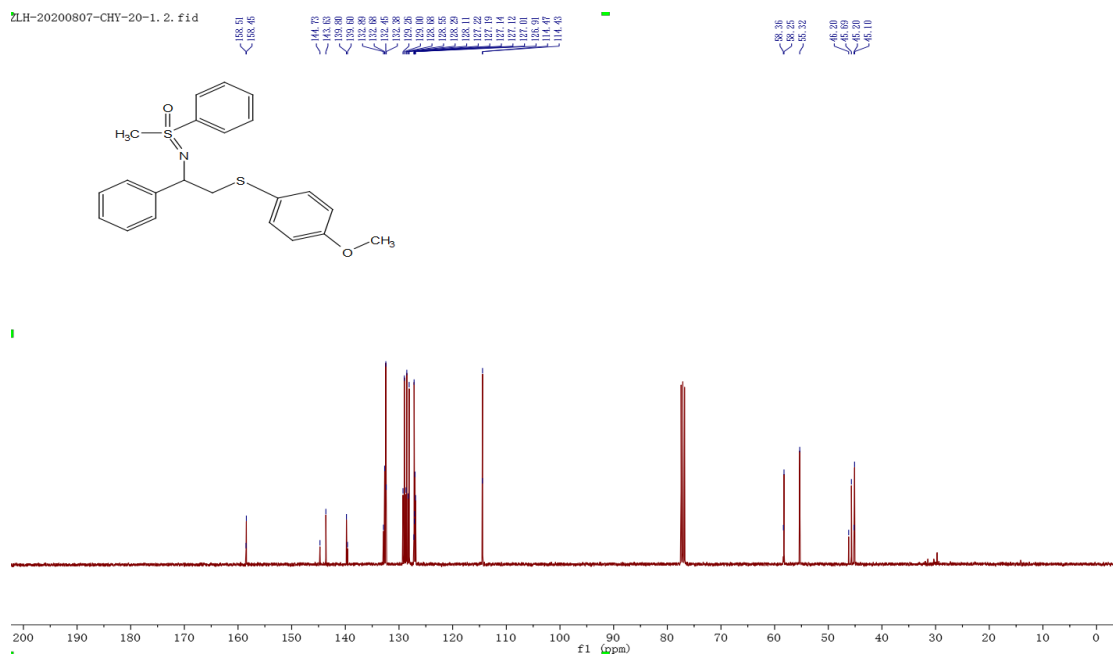
ZLH-20200111-CHY-2. 1. fid



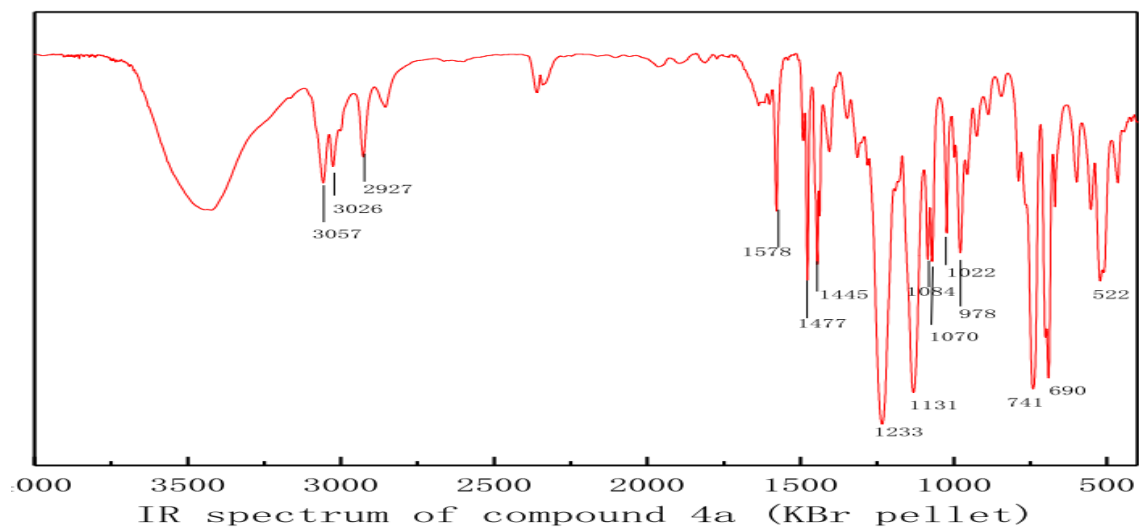
ZLH-20200111-CHY-2.2.fid

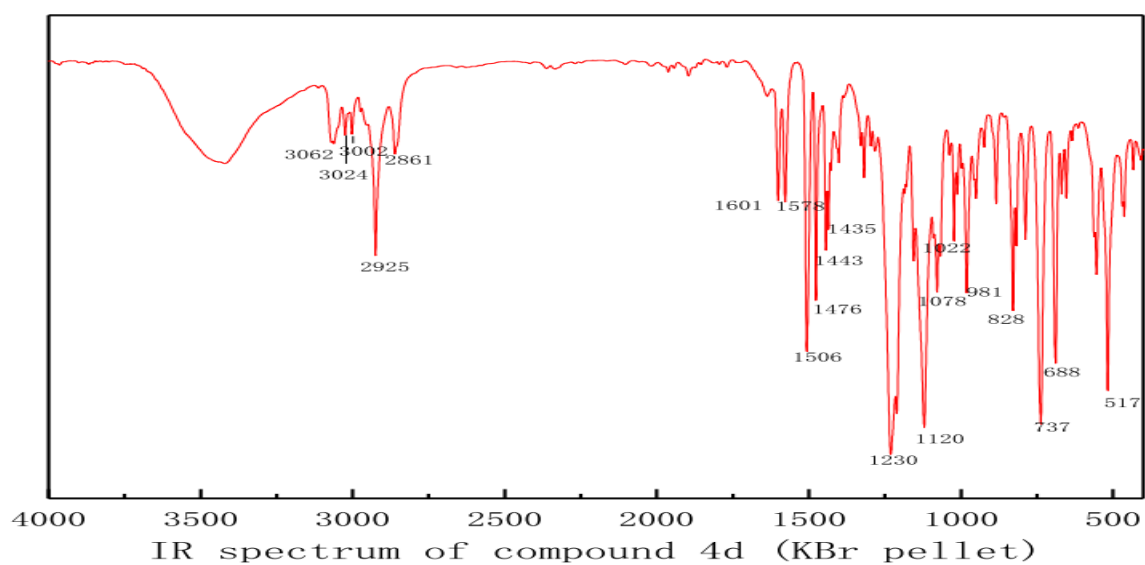
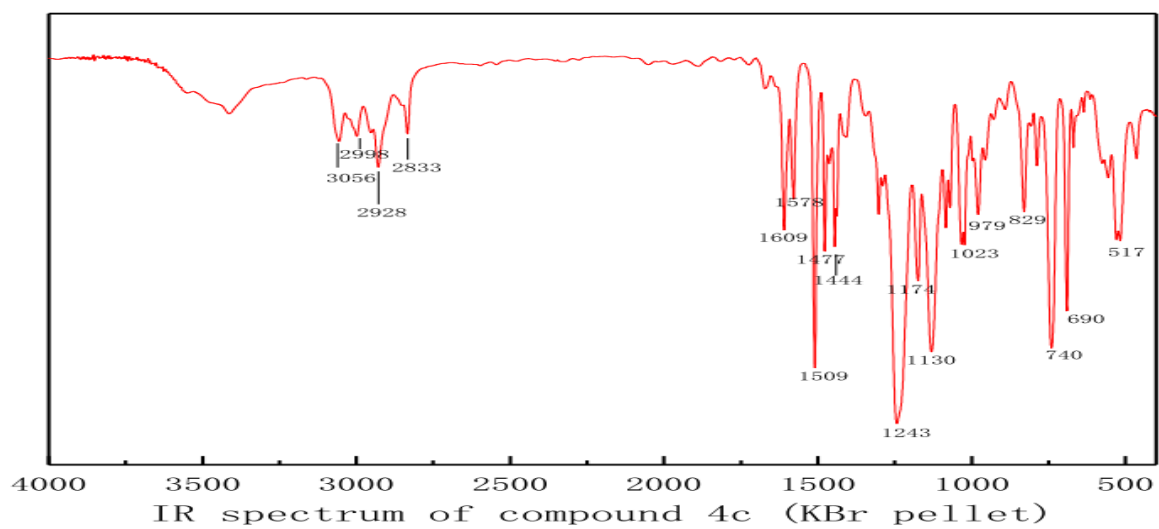
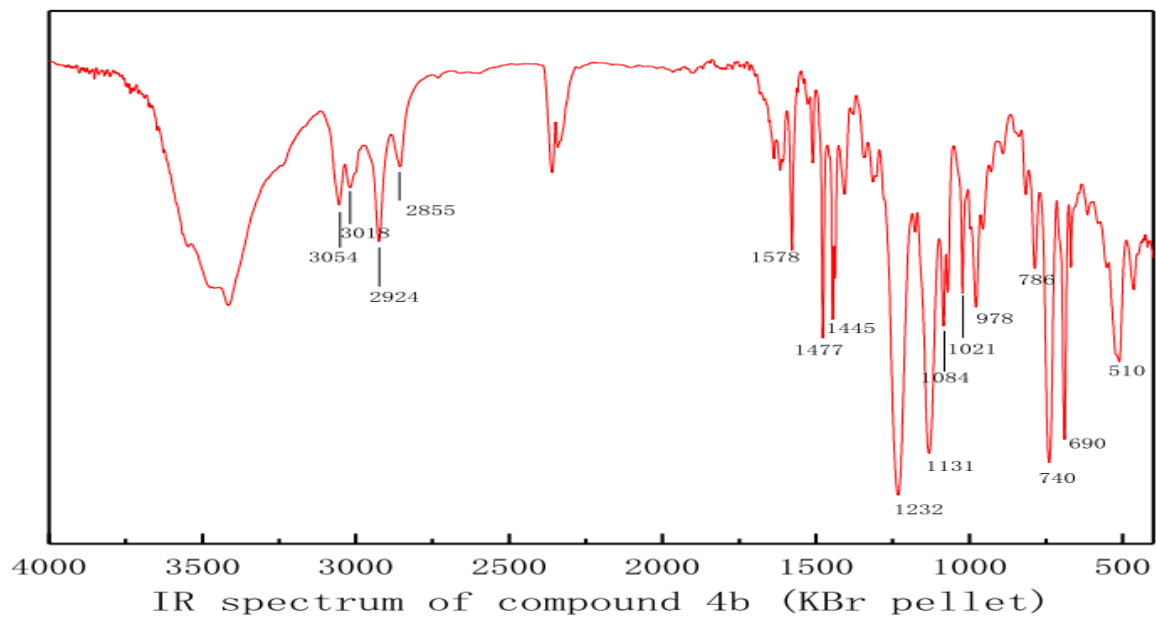


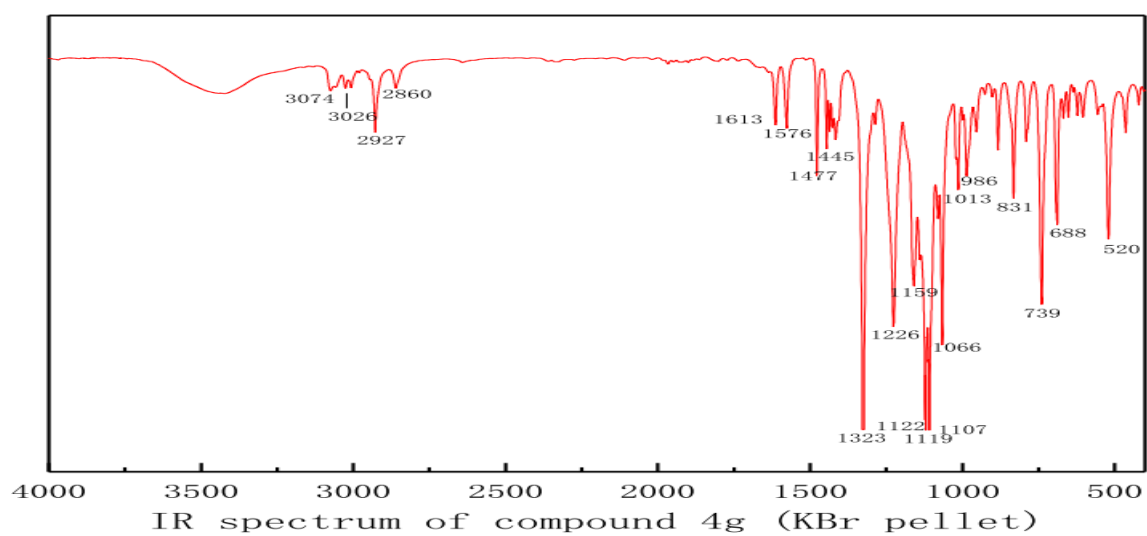
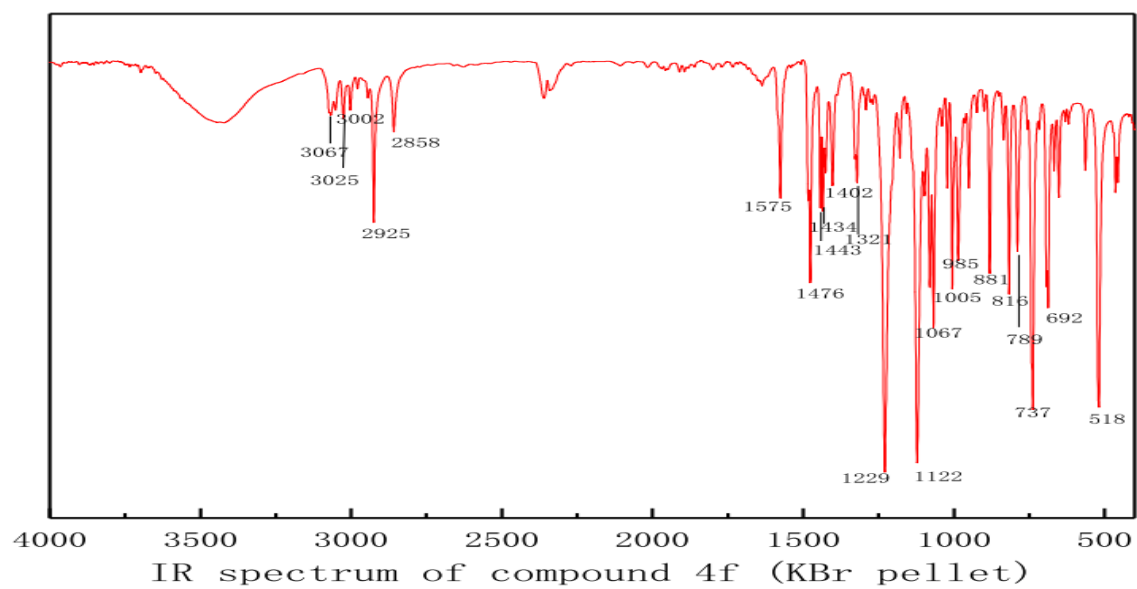
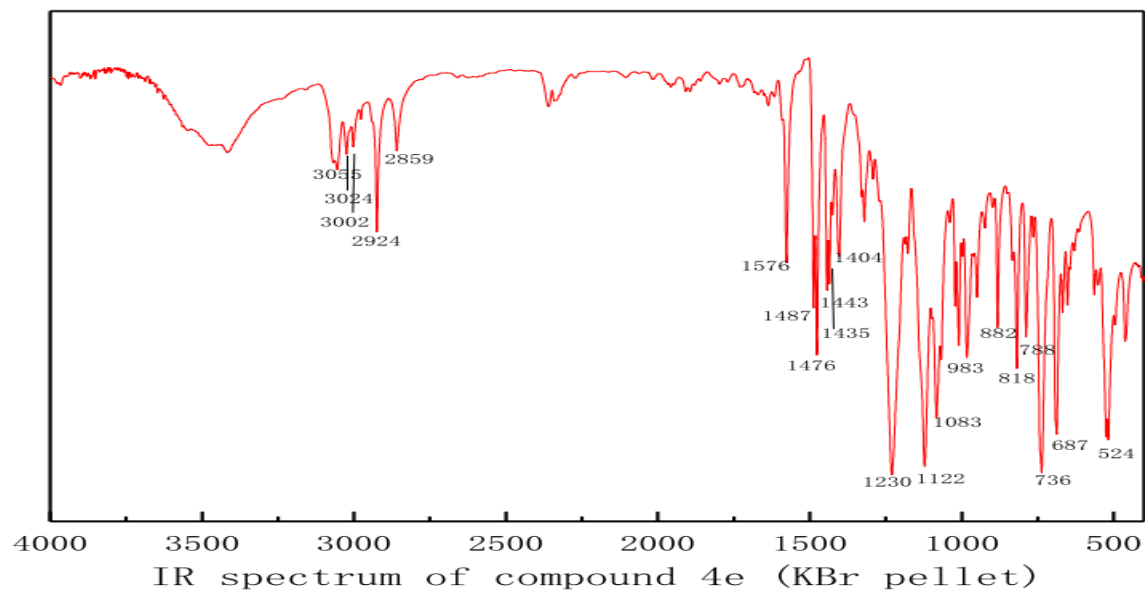
ZLH-20200807-CHY-20-1. 2. fid

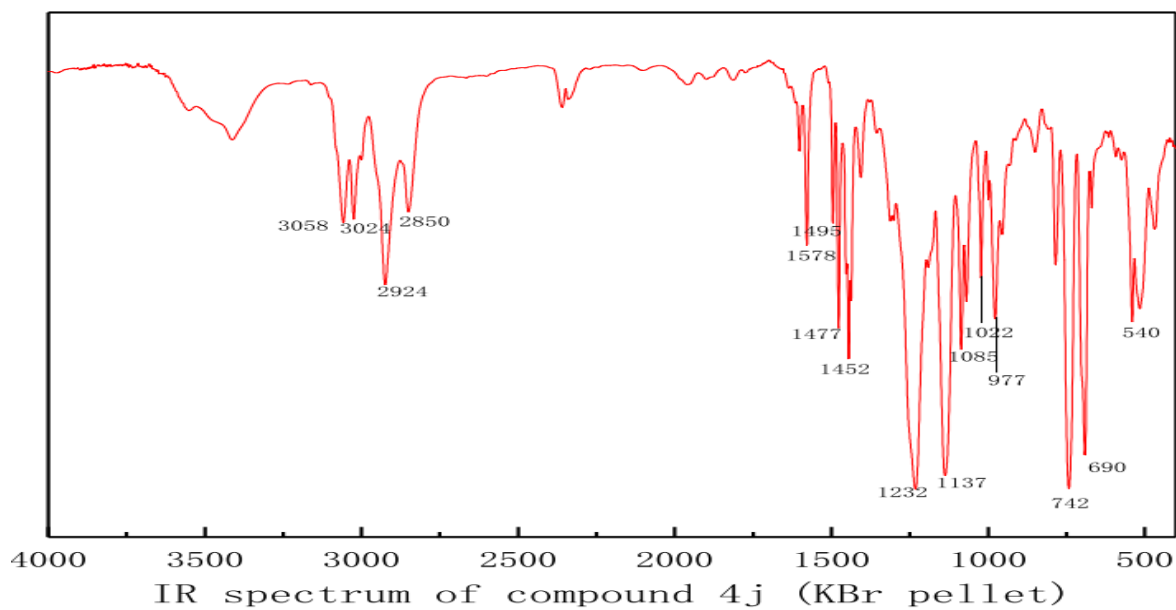
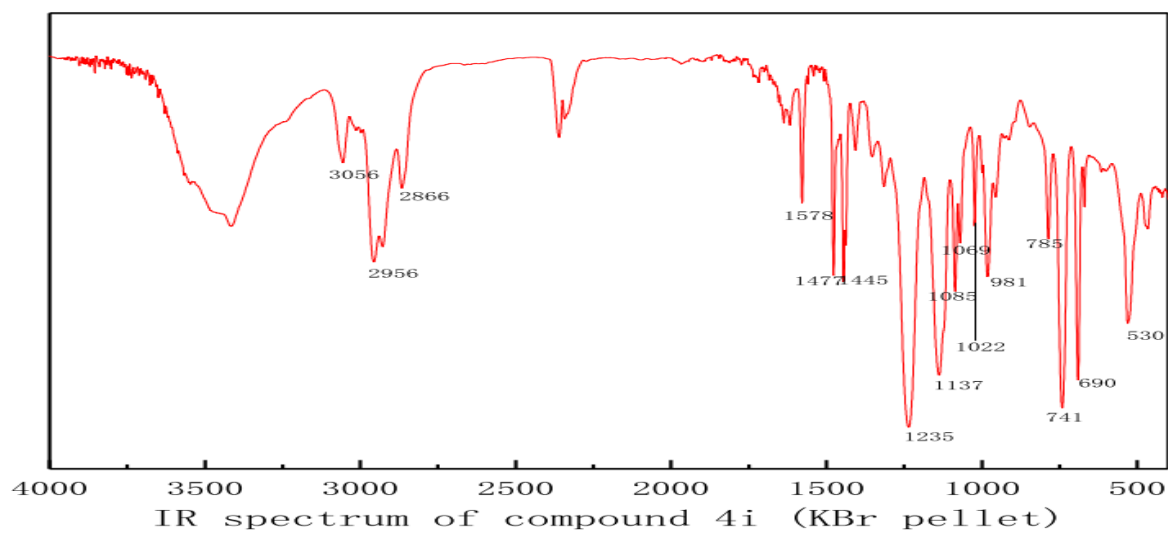
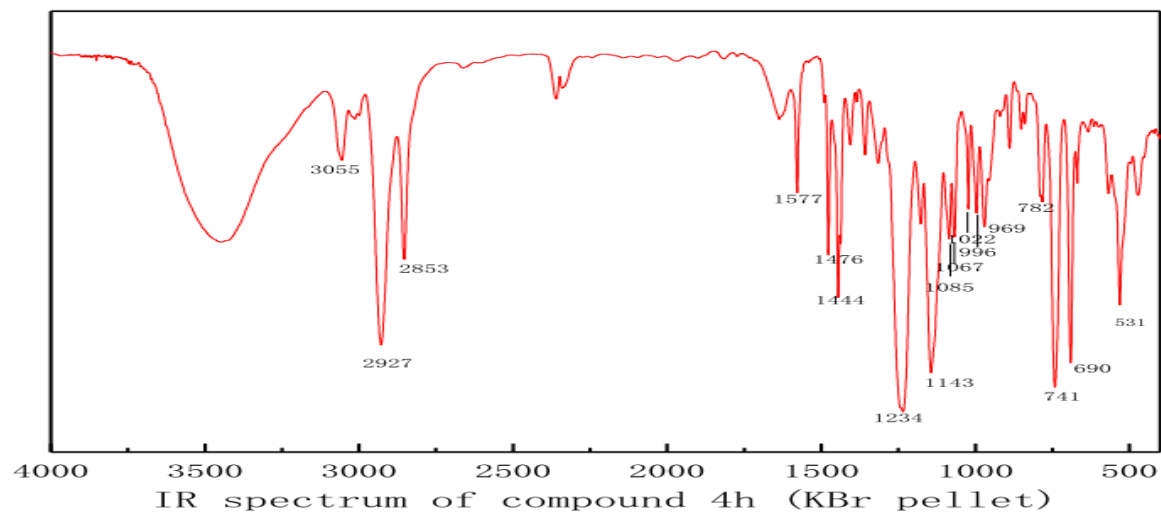


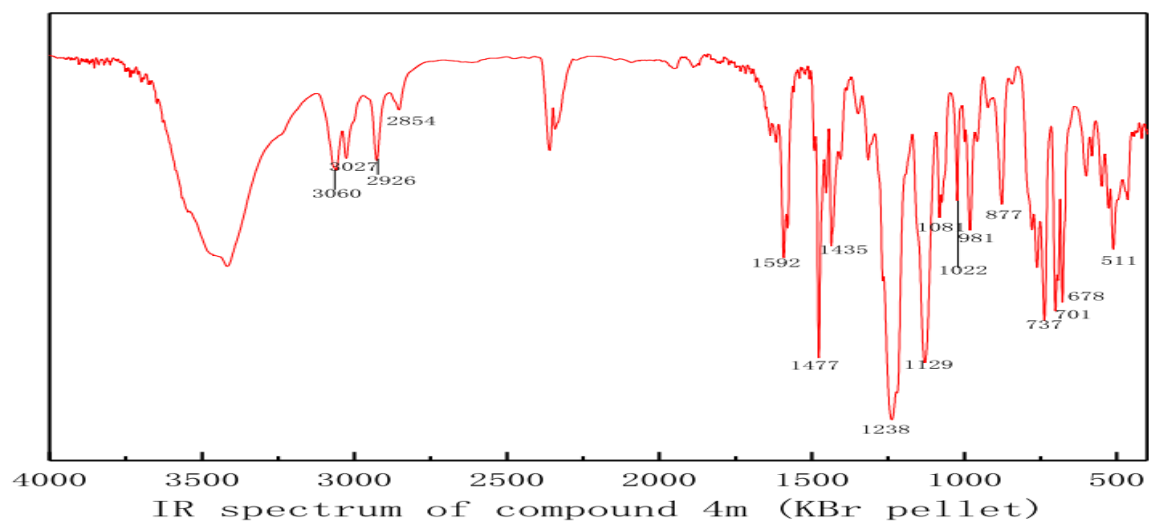
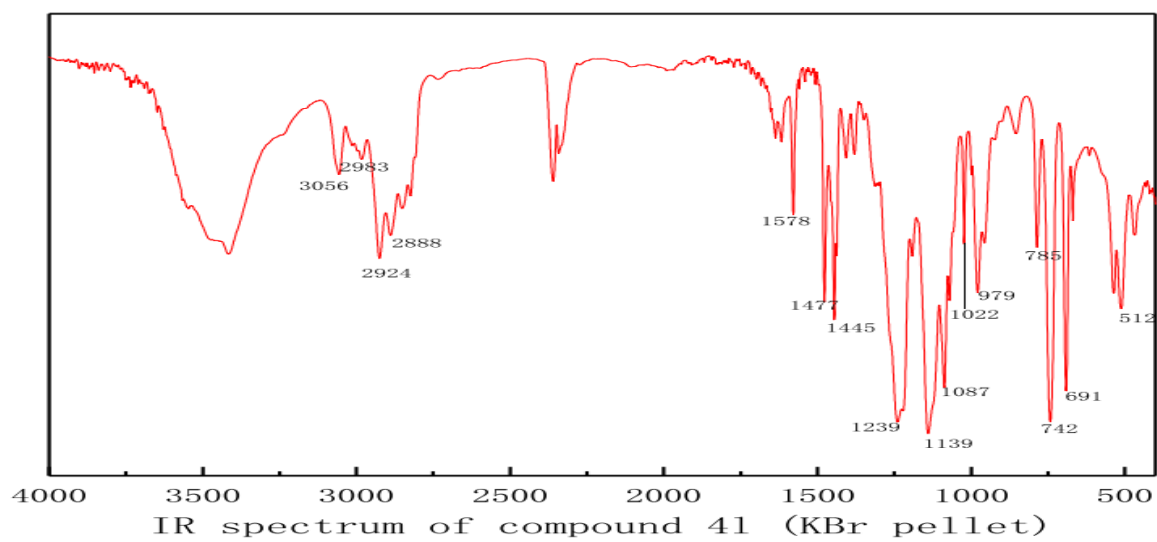
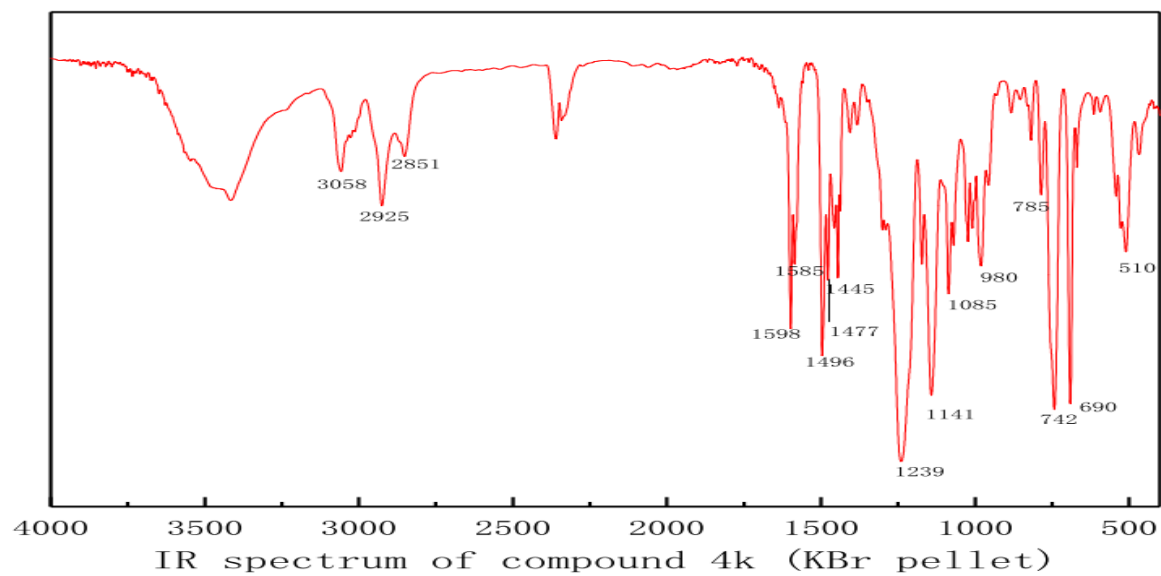
IR spectra

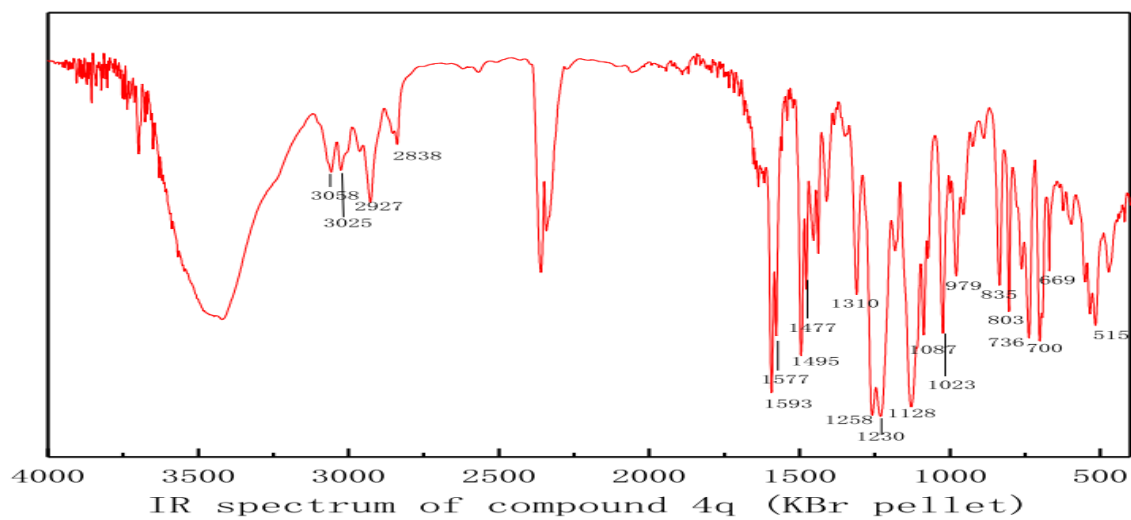
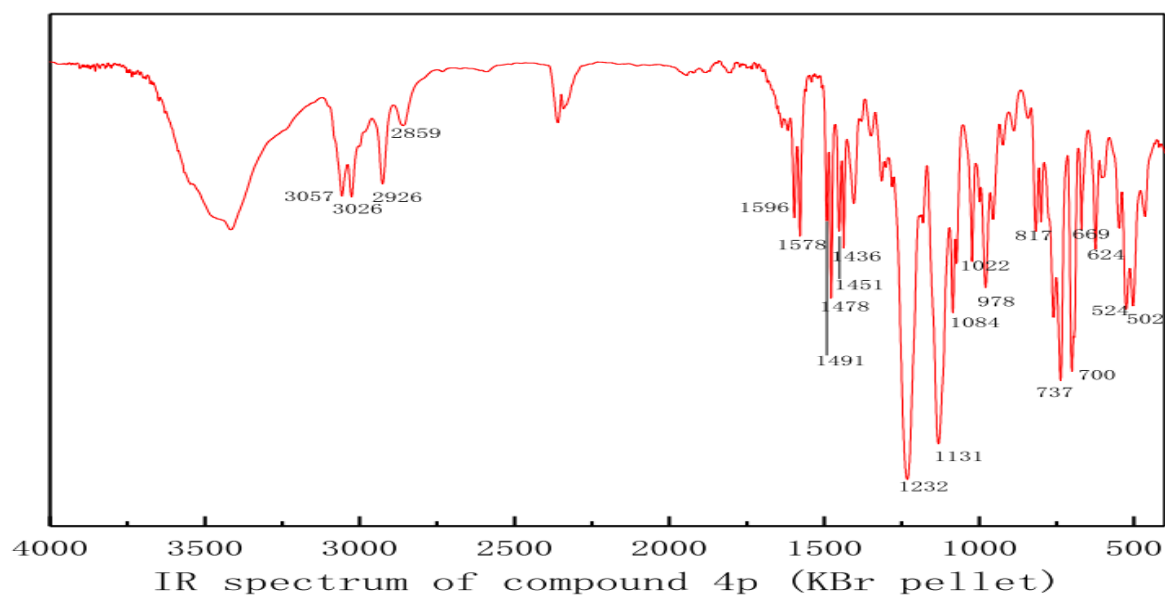
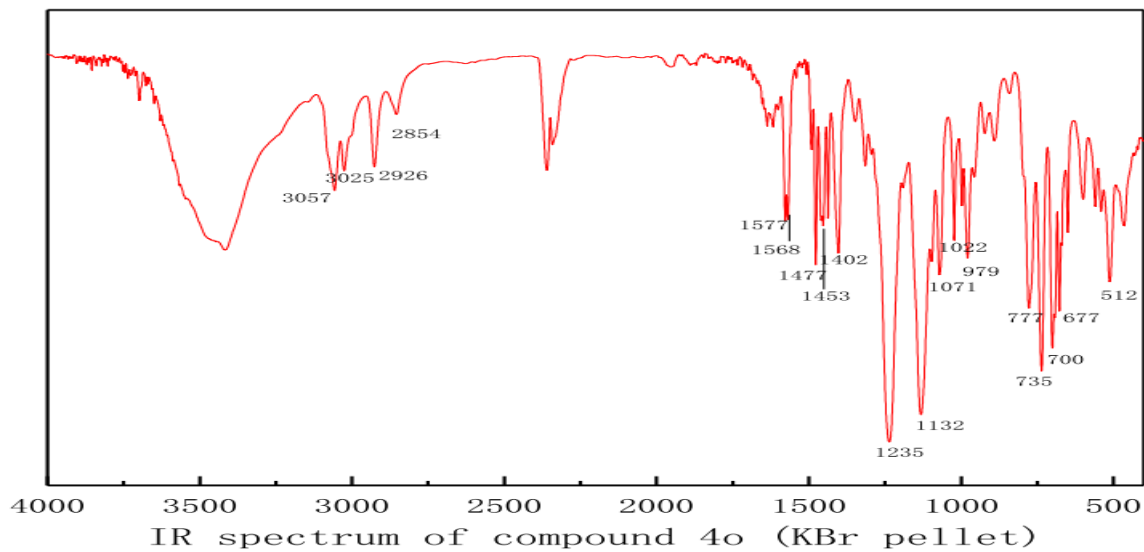


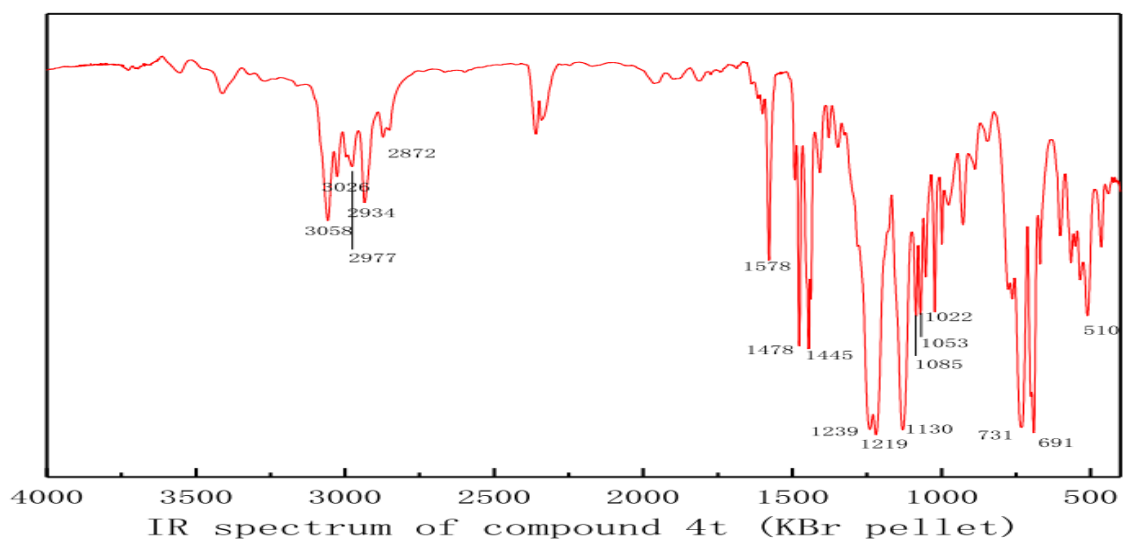
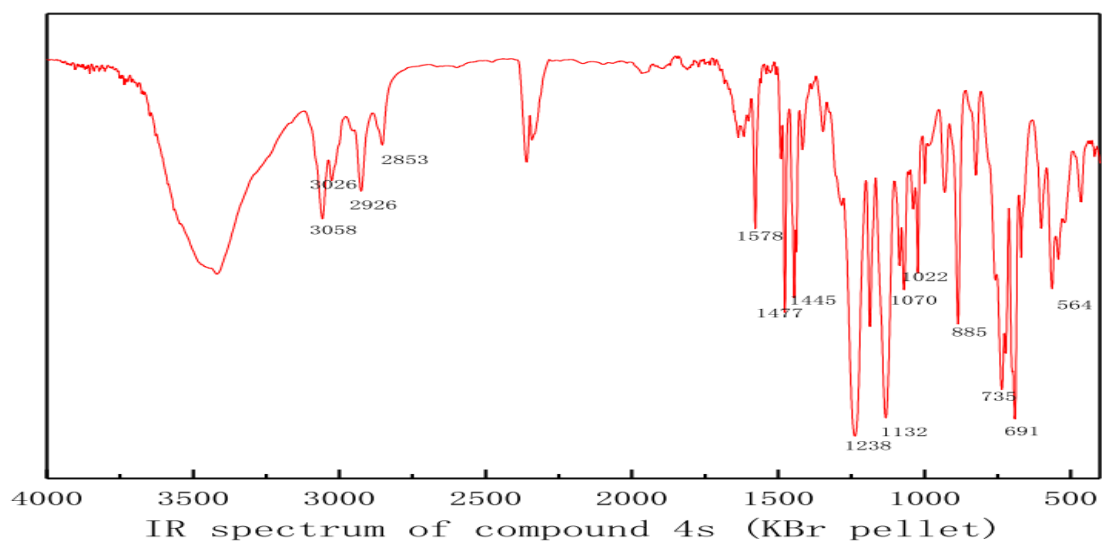
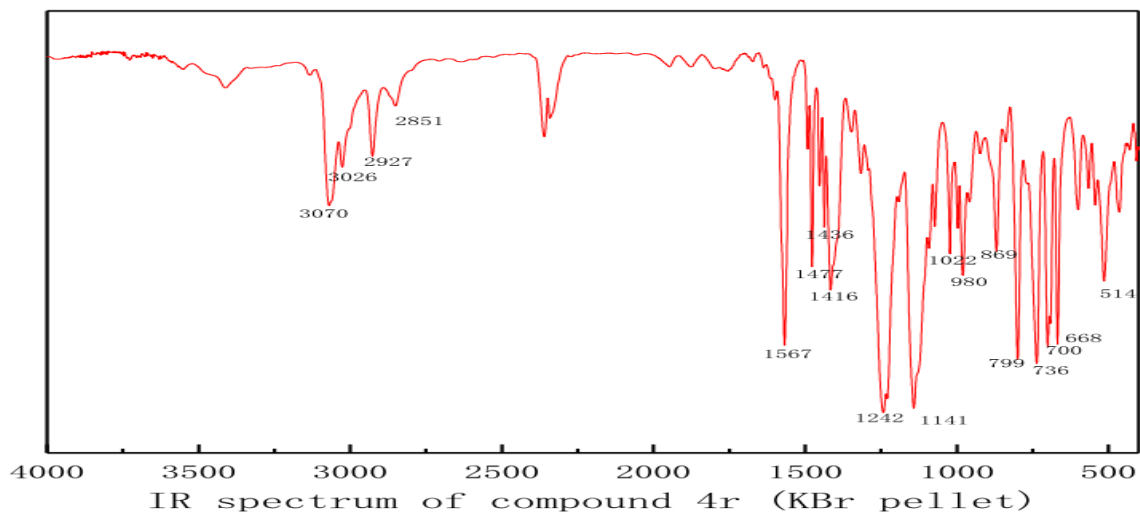


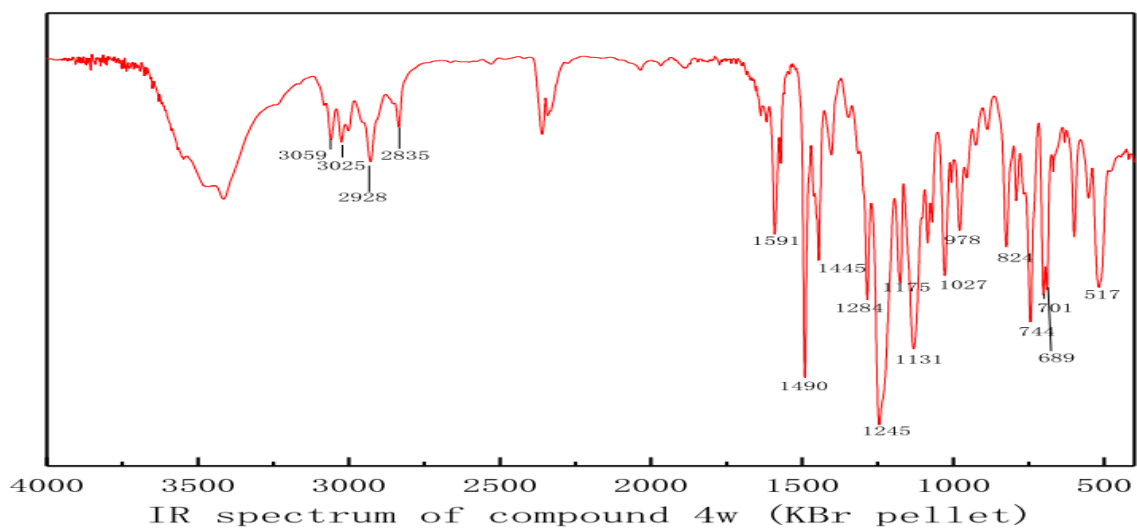
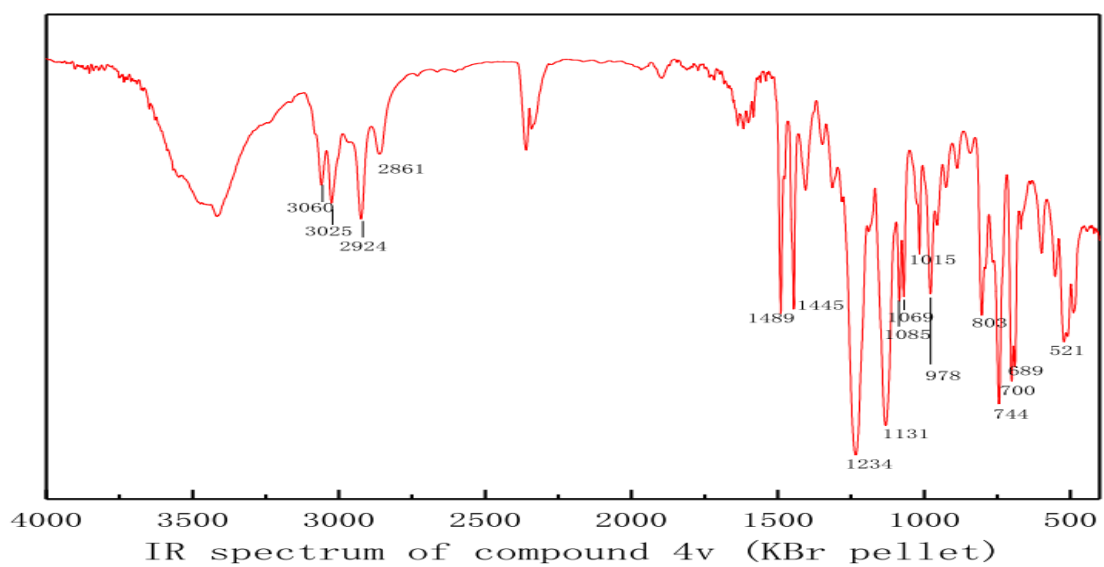
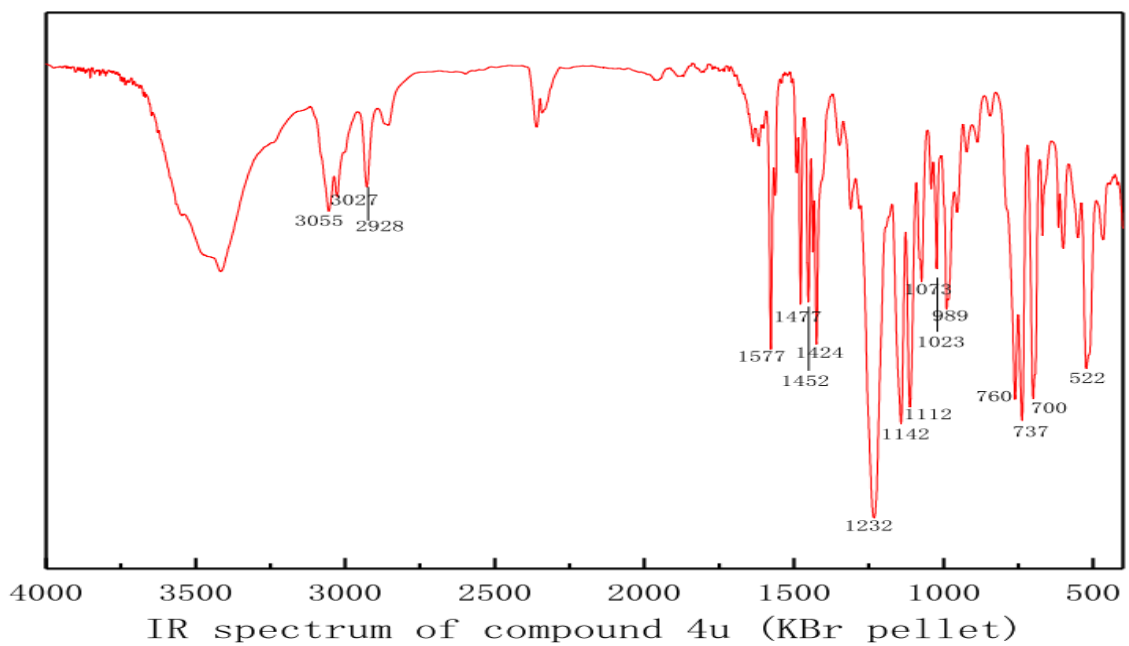


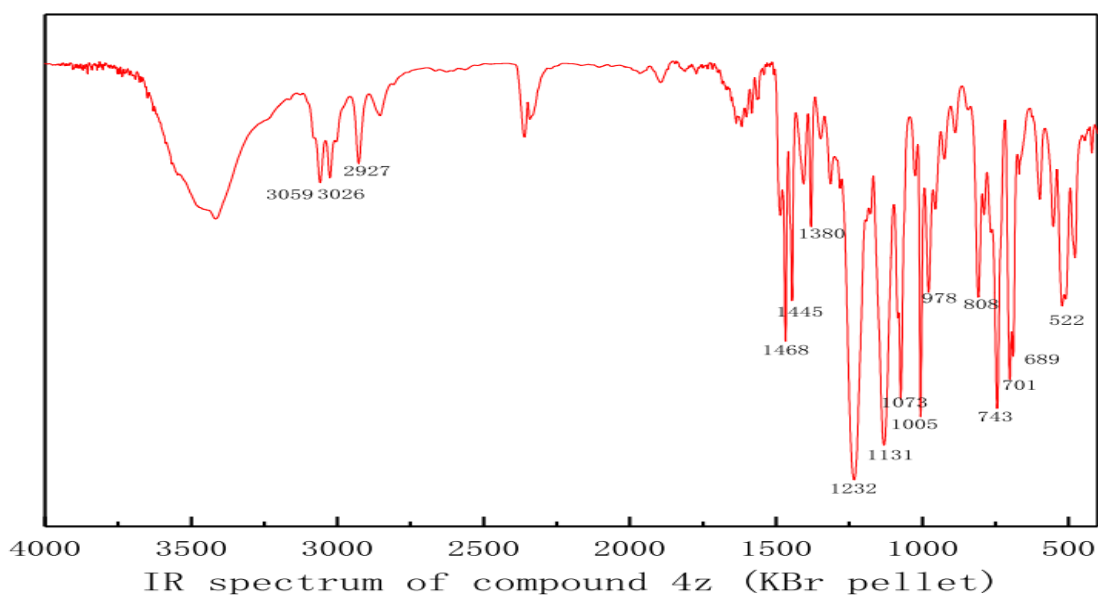
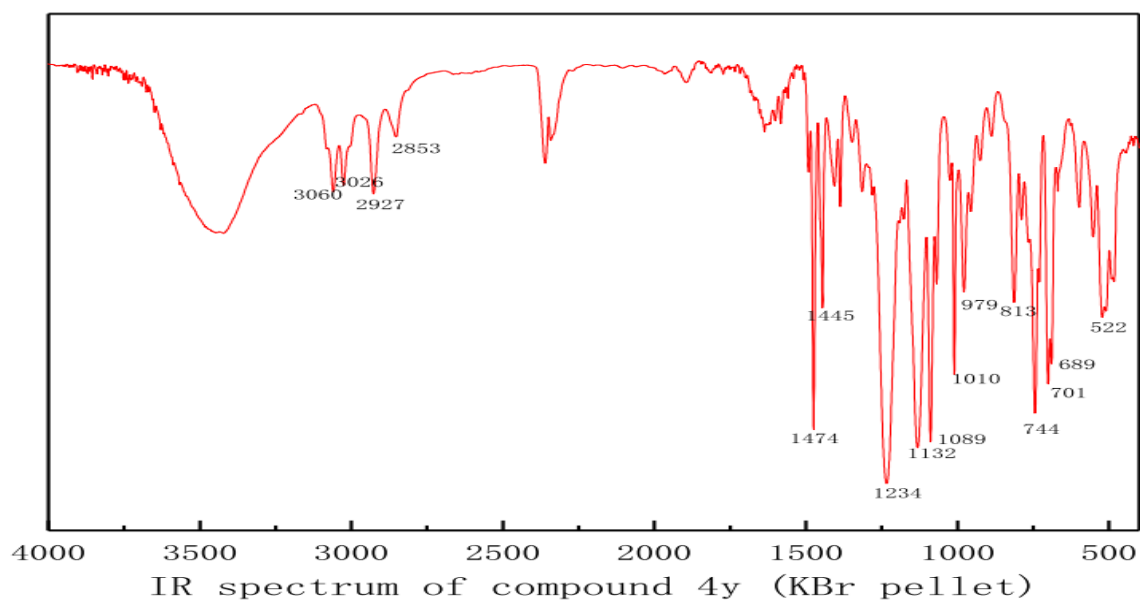
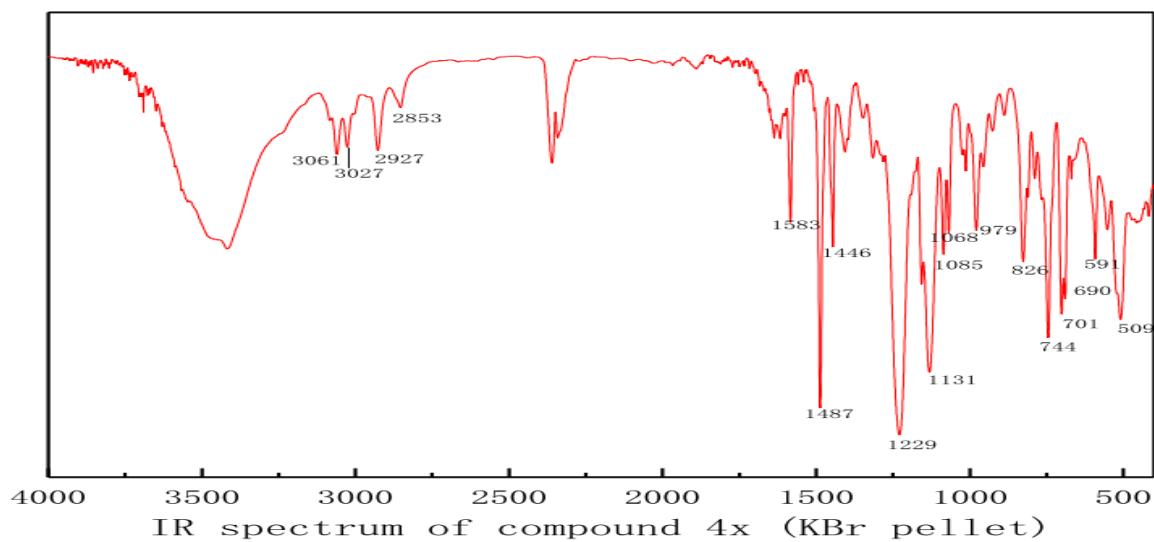


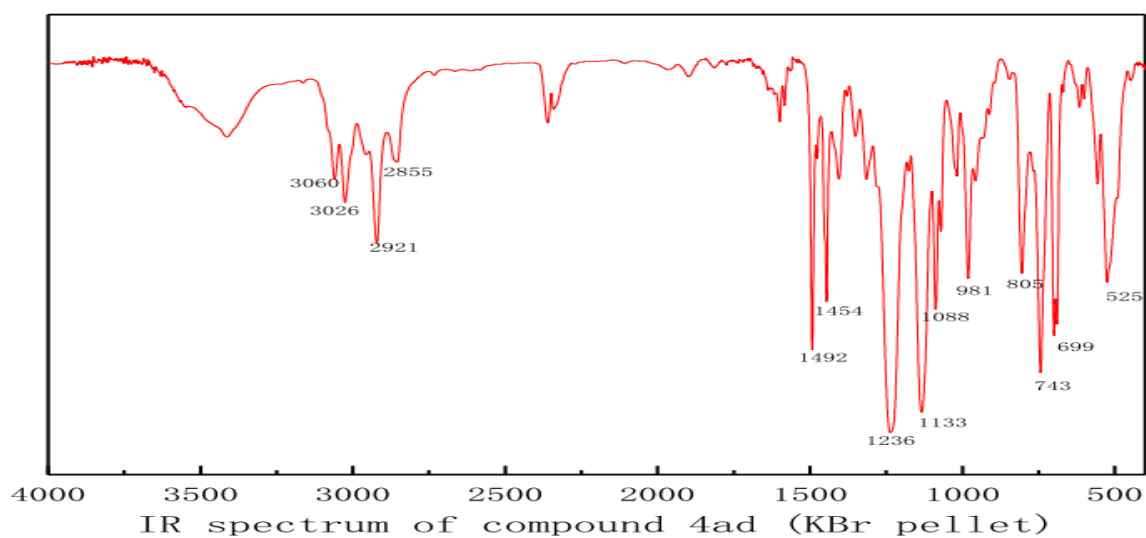
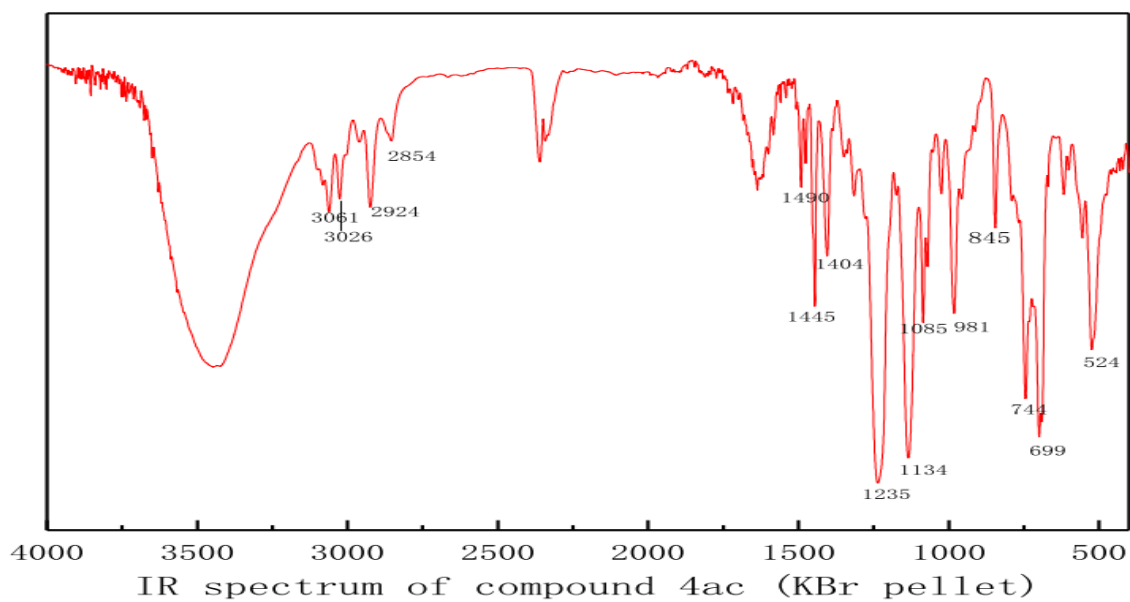
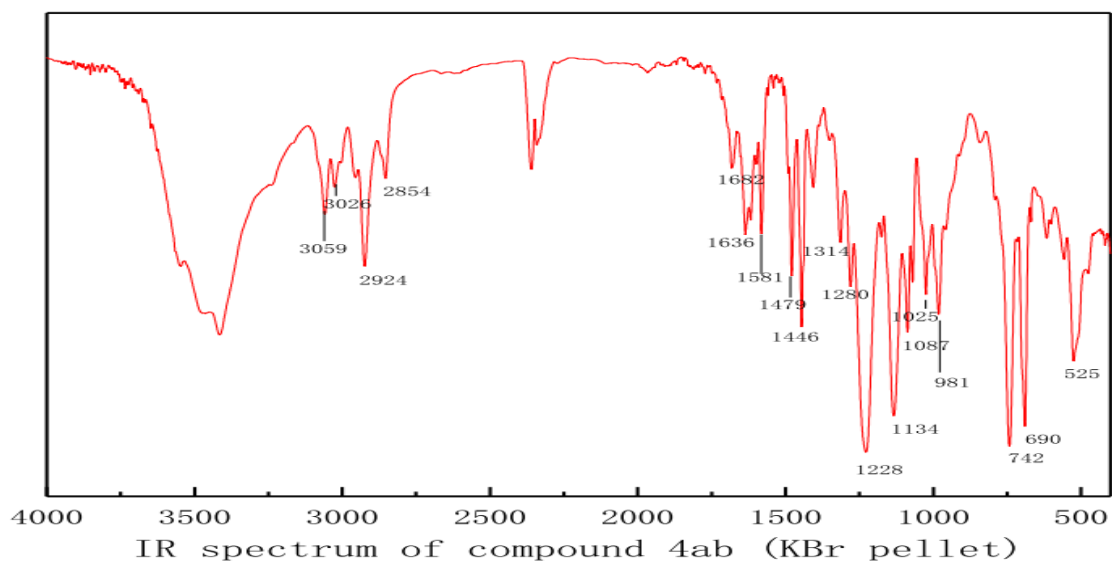


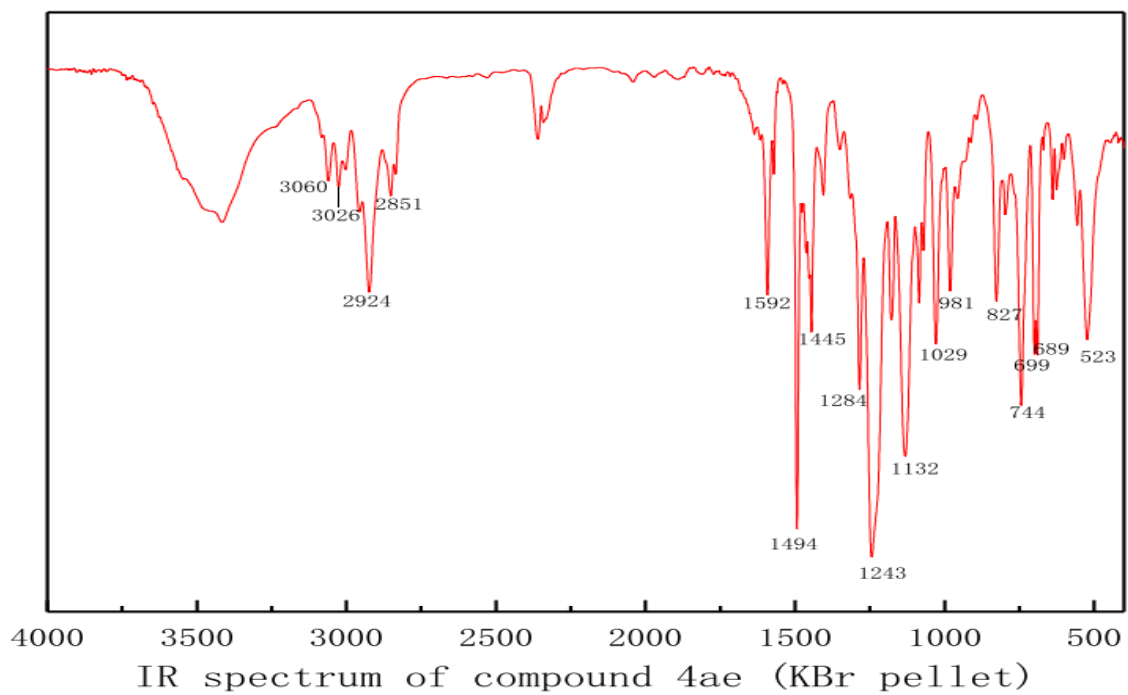






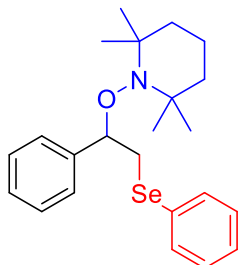






HRMS of radical-trapped TEMPO adduct

2,2,6,6-tetramethyl-1-(1-phenyl-2-(phenylselanyl)ethoxy)piperidine (8)



HRMS (ESI) m/z $[M + H]^+$ Calcd for $C_{23}H_{32}NOSe^+$ 418.1644, found 418.1641.

D:\WD\Data\20201015\ZQL-CHY-1

10/15/20 10:40:18

ZQL-CHY-1 #32-61 RT: 0.15-0.28 AV: 30 NL: 8.51E6
T: FTMS + p ESI Full ms [100.0000-1000.0000]

