Supporting Information

Electrochemically promoted selenocyclization for the synthesis of organoselenyl isoxazoles

Nan Sun,* Zhi Qiao, Jiamin, Li, Jiazhi, Gu, Liqun Jin, and Xinquan Hu*

College of Chemical Engineering, Zhejiang University of Technology, Hangzhou 310032, Hangzhou 310032, People's Republic of China

E-mail: sunnan@zjut.edu.cn and xinquan@zjut.edu.cn

Table of Contents

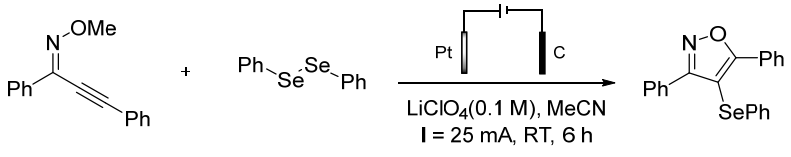
1. General Remarks

The ^{1}H, ^{13}C, ^{77}Se and ^{19}F NMR analyses were performed on a Bruker AVANCE III 500 MHz spectrometer in deuterated solvents. ^{1}H, and ^{13}C NMR spectra were recorded with tetramethylsilane (TMS) as internal standard. The ^{77}Se NMR spectra were recorded with diphenyl diselenide (δ = 461 ppm) as external standard. The ^{19}F NMR spectra were recorded with $CFCl_3$ (δ = 0 ppm) as external standard. Low-resolution mass analyses were performed on a Thermo Scientific TRACE ISQ GC-MS instrument in EI mode (70 eV). High resolution mass analyses were performed on an Agilent 6545 LC/Q-TOF mass spectrometer in positive ESI mode. Melting points (uncorrected) were determined on a BUCHI M-565 apparatus. Gas chromatography (GC) analyses were performed on a Shimadzu GC-2010 Plus instrument with FID detector using an Agilent J&W DB-5 capillary column (30 m x 0.32 mm (i.d.), 0.25 μm).

Electrochemical reactions were conducted using a GWINSTEK GPD-3303S DC power supply in constant current mode using undivided cell equipped with a platinum plate (10 mm x 8 mm x 1 mm) as the anode and a graphite rod (φ = 6 mm) as the cathode under air. Cyclic voltammograms were recorded on a Chenhua CHI 660E Instruments.

Reagents and solvents were purchased as reagent grade and were directly used without further purification. The reactants 2-alkyn-1-one *o*-methyloximes **1** and diorganyl diselenides **2** were prepared according to literature methods.[1-4] Flash column chromatography were performed on silica gel (200-300 mesh) with petroleum ether/ethyl acetate as eluents.

2. Gram-Scale Synthesis



1a **2a** **3aa**

To a 100 mL of undivided three-necked bottle were charged with (*Z*)-1,3-diphenylprop-2-yn-1-one *O*-methyl oxime **1a** (1.18 g, 5.0 mmol), diphenyl diselenide **2a** (1.56 g, 5.0 mmol), $LiClO_4$ (1.06 g, 10.0 mmol) and 100 mL of CH_3CN. The bottle was equipped with a platinum plate (10 mm x 8 mm x 0.1 mm) as the anode and a graphite rod (Φ = 6 mm) as the cathode. The reaction mixture was stirred and electrolyzed at a constant current of 25 mA at room temperature for 6 h until complete consumption of **1a** as monitored by GC analysis. Then, the reaction mixture was concentrated under reduced pressure to remove solvent CH_3CN. The resulted solid was re-dissolved in 30 mL of EtOAc, followed by successively washing with water twice, drying over anhydrous Na_2SO_4 and concentrating under a vacuum. The residual was purified by column chromatography on silica gel with a 75 : 1 mixture of petroleum ether and ethyl acetate as eluent. Finally, 1.80 g of **3aa** was obtained as white solid in 96% yield.

3. Mechanism Studies

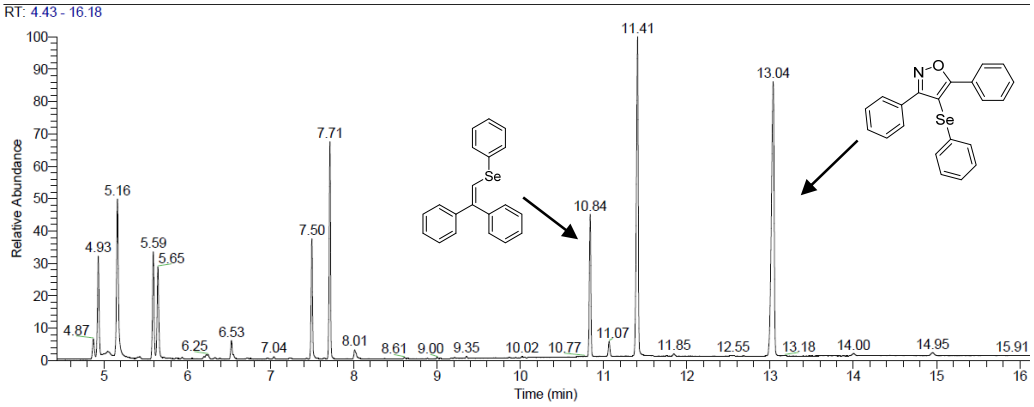


Figure S1 GC-MS chromatogram of the reaction mixture of (*Z*)-1,3-diphenylprop-2-yn-1-one *O*-methyloxime (**1a**), diphenyl diselenide (**2a**) in the presence of 1,1-diphenylethylene

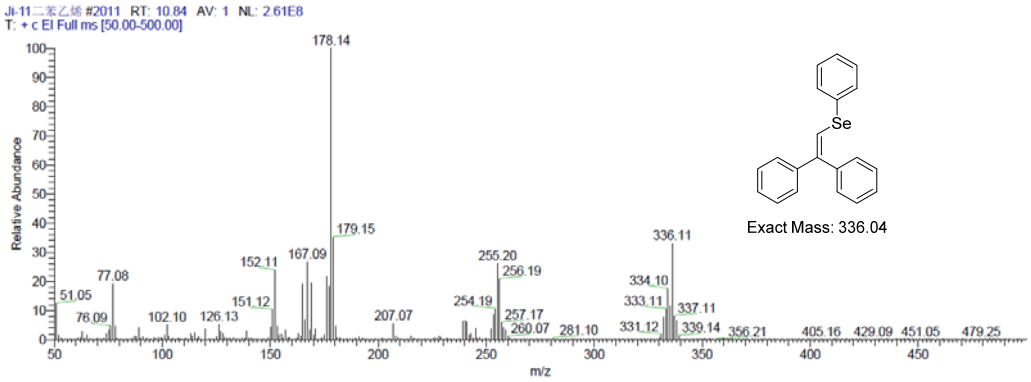


Figure S2 MS spectrum of the peak at 10.84 min

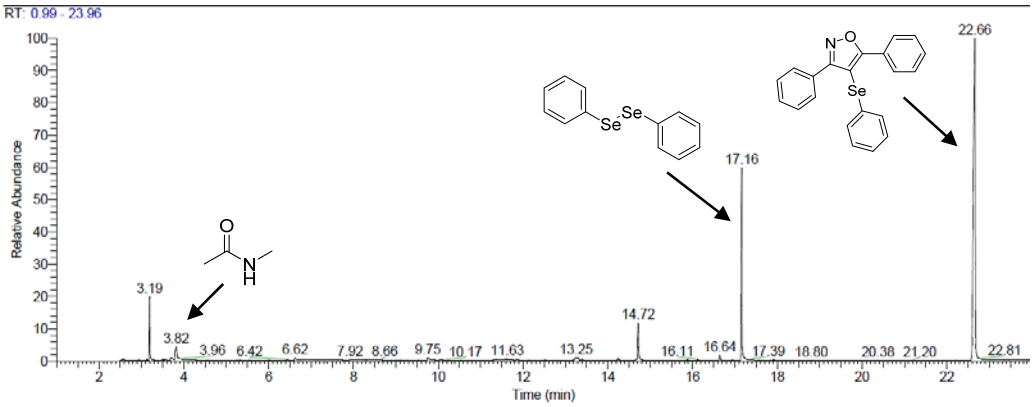


Figure S3 GC-MS chromatogram of the reaction mixture of (*Z*)-1,3-diphenylprop-2-yn-1-one *O*-methyloxime (**1a**) and diphenyl diselenide (**2a**)

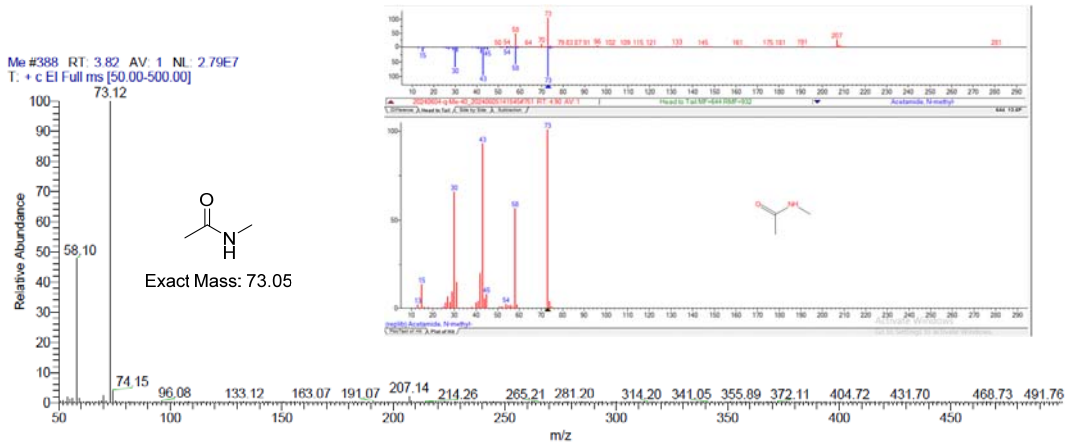


Figure S4 MS spectrum of the peak at 3.82 min

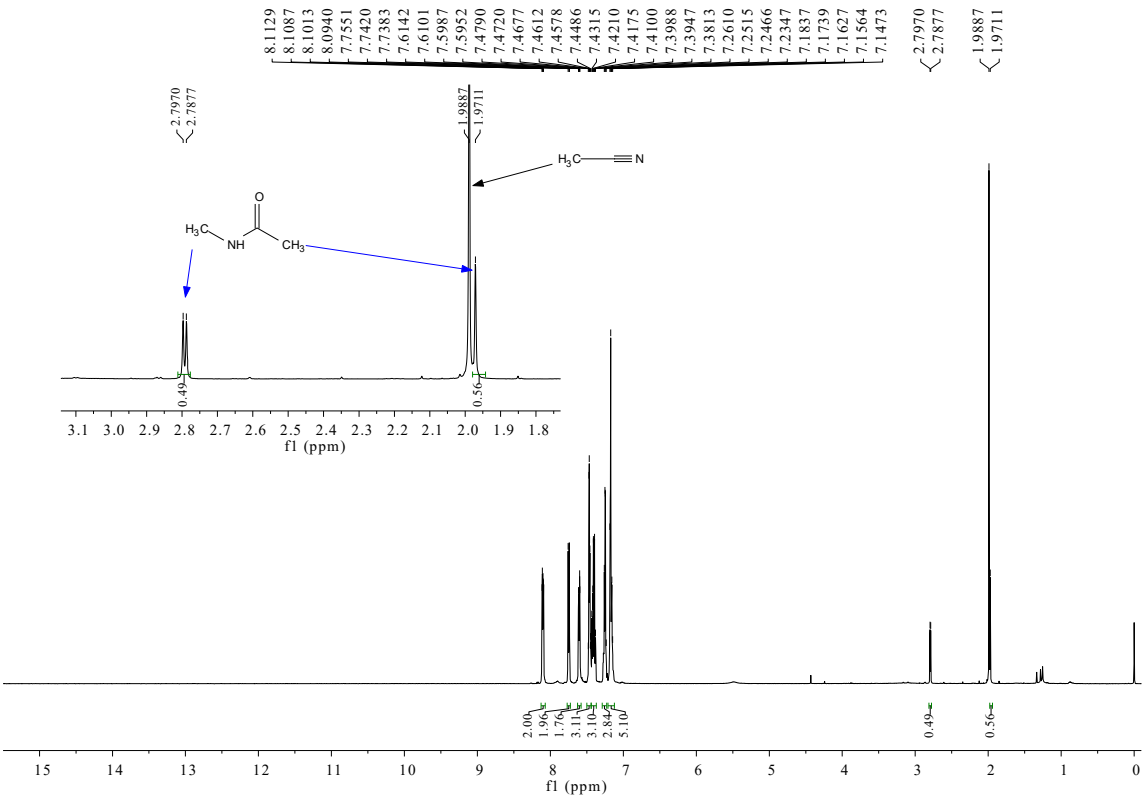


Figure S5 ^{1}H NMR spectrum of reaction mixture of (*Z*)-1,3-diphenylprop-2-yn-1-one *O*-methyloxime (**1a**) and diphenyl diselenide (**2a**) after removing most of the CH_3CN solvent

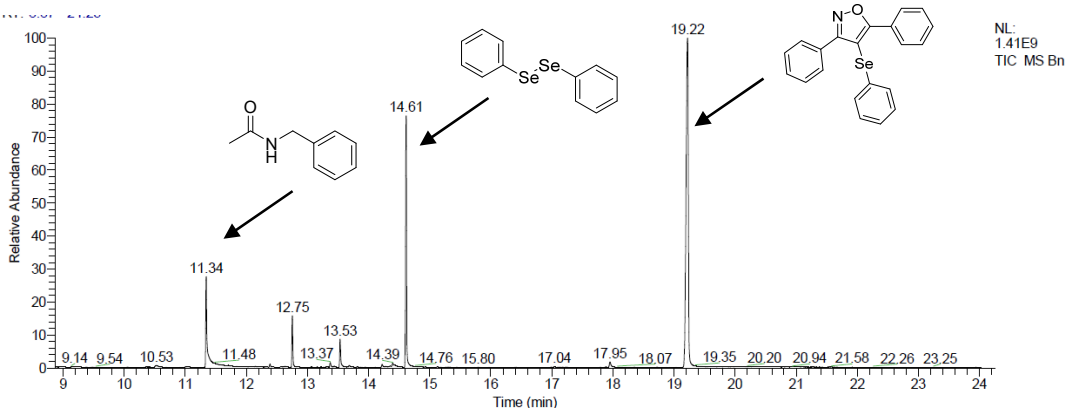


Figure S6 GC-MS chromatogram of the reaction mixture of (*Z*)-1,3-diphenylprop-2-yn-1-one *O*-benzyloxime (**4**) and diphenyl diselenide (**2a**)

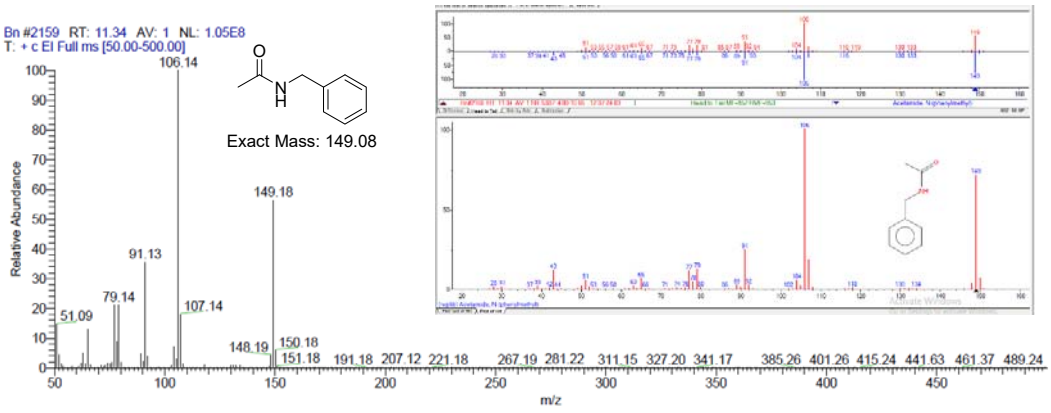


Figure S7 MS spectrum of the peak at 11.34 min

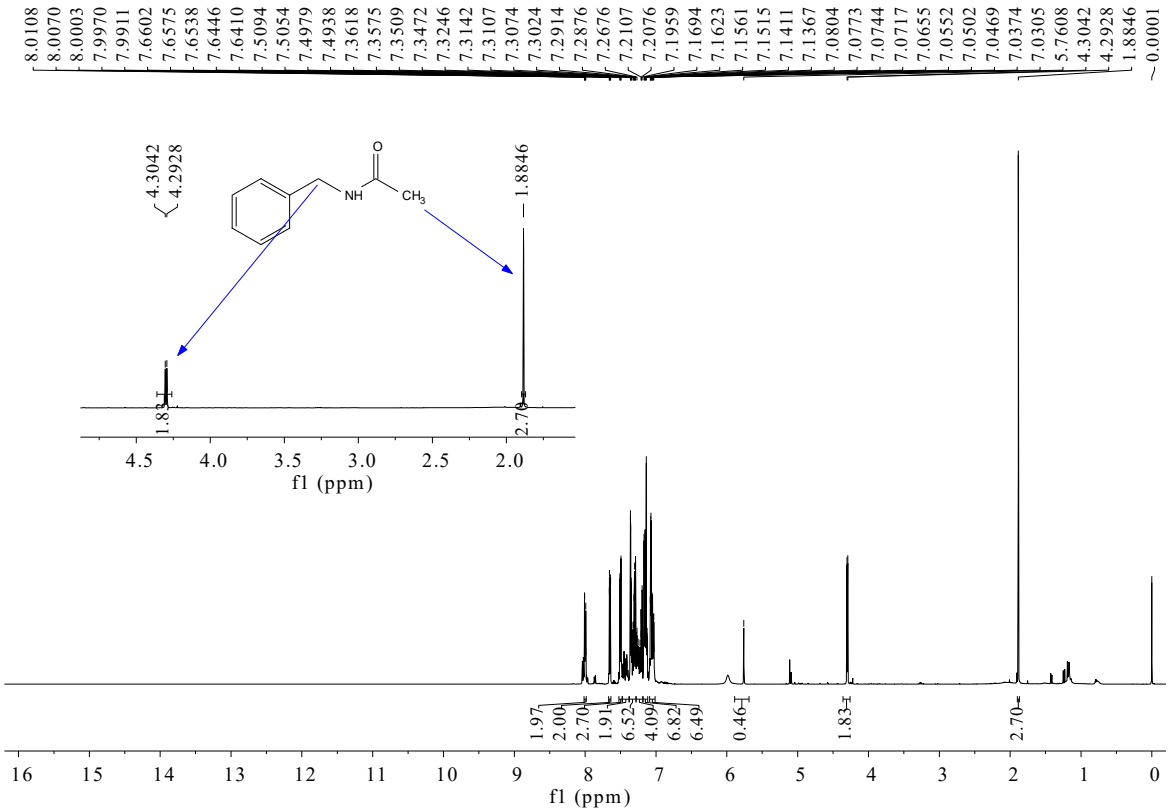
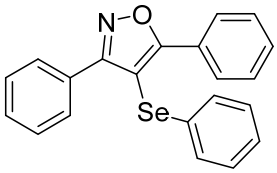


Figure S8 ^{1}H NMR spectrum of reaction mixture of (*Z*)-1,3-diphenylprop-2-yn-1-one *O*-benzyloxime (**4**) and diphenyl diselenide (**2a**) after removing the CH_3CN solvent

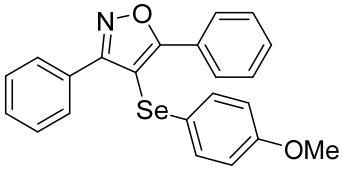
4. Characterization Data for all Synthesized 4-Organoselenyl Isoxazoles

Note: The synthesized 4-organoselenyl isoxazoles, including **3ad, 3ag, 3ai, 3aj, 3ak, 3am, 3ap, 3aq, 3at, 3ga, 3la, 3ma, 3pa, 3qa, 3ra, 3ta** and **6**, in this paper are new compounds.



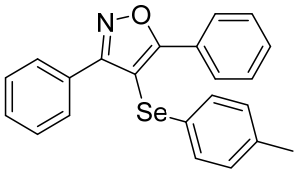
3aa

3,5-Diphenyl-4-(phenylselenyl)isoxazole (3aa) (CAS No.: 870127-46-1): Flash column chromatography eluent (petroleum ether/ethyl acetate = 75:1); Yield: 99% (187 mg); White solid; M.p.: 102-104 ℃ (lit.[5] M.p.: 100-102 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.06-7.94 (m, 2H), 7.65 (d, J = 7.3 Hz, 2H), 7.36-7.31 (m, 3H), 7.31-7.23 (m, 3H), 7.11-7.00 (m, 5H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.53, 165.86, 131.69, 130.88, 129.93, 129.62, 128.92, 128.84, 128.78, 128.48, 127.95, 127.45, 126.68, 96.44 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 236.77 ppm. GC-MS (EI) m/z: 377 (M^+, 6%), 375 (3), 105 (100).



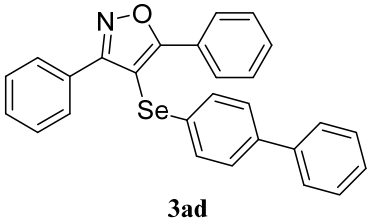
3ab

4-((4-Methoxyphenyl)selenyl)-3,5-diphenylisoxazole (3ab) (CAS No.: 2487302-10-1): Flash column chromatography eluent (petroleum ether/ethyl acetate = 30:1); Yield: 94% (190 mg); White solid; M.p.: 104-106 ℃ (lit.[5] M.p.: 102-104 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.20-8.11 (m, 2H), 7.83-7.70 (m, 2H), 7.53-7.40 (m, 6H), 7.14-7.07 (m, 2H), 6.74-6.67 (m, 2H), 3.72 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 170.76, 164.65, 157.93, 130.49, 129.65, 128.74, 127.91, 127.83, 127.64, 127.33, 126.89, 126.48, 120.15, 114.13, 96.73, 54.20 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 222.57 ppm. GC-MS (EI) m/z: 407 (M^+, 16%), 405 (8), 105 (100).



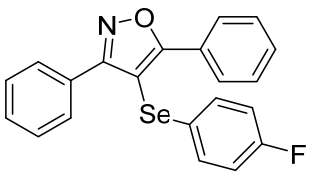
3ac

3,5-Diphenyl-4-(p-tolylselenyl)isoxazole (3ac) (CAS No.: 1422017-97-7): Flash column chromatography eluent (petroleum ether/ethyl acetate = 75:1); Yield: 98% (191 mg); White solid; M.p.: 108-110 ℃ (lit.[6] M.p.: 112-113 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.22-8.16 (m, 2H), 7.89-7.82 (m, 2H), 7.52-7.43 (m, 6H), 7.17-7.11 (m, 2H), 7.03 (d, J = 8.0 Hz, 2H), 2.28 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.38, 165.86, 136.66, 130.83, 130.44, 129.91, 129.10, 128.99, 128.93, 128.78, 128.48, 127.98, 127.87, 127.56, 96.82, 21.04 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 229.46 ppm. GC-MS (EI) m/z: 391 (M^+, 14%), 389 (7), 105 (100).



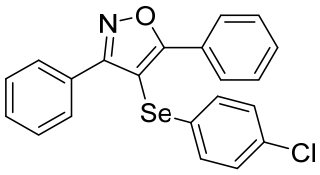
3ad

4-((1,1'-Biphenyl)-4-ylselenyl)-3,5-diphenylisoxazole (3ad): Flash column chromatography eluent (petroleum ether/ethyl acetate = 30:1); Yield: 89% (202 mg); Yellow solid; M.p.: 133-135 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.07-8.02 (m, 2H), 7.72-7.67 (m, 2H), 7.43-7.37 (m, 5H), 7.36-7.29 (m, 7H), 7.25-7.21 (m, 1H), 7.18-7.13 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.54, 165.84, 140.16, 139.66, 130.87, 130.63, 129.92, 129.24, 128.91, 128.87, 128.77, 128.47, 128.19, 127.95, 127.51, 127.43, 126.85, 96.36 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 234.26 ppm. HRMS (+ESI) m/z: [$M+H^+$] calculated for $C_{27}H_{20}NOSe^+$ 454.0705, found 454.0705.



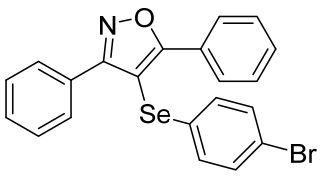
3ae

4-((4-Fluorophenyl)selenyl)-3,5-diphenylisoxazole (3ae) (CAS No.: 1422017-94-4): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 96% (189 mg); White solid; M.p.: 125-127 ℃ (lit.[6] M.p.: 114-115 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.14-8.09 (m, 2H), 7.79-7.71 (m, 2H), 7.52-7.40 (m, 6H), 7.13 (m, 7.16-7.09, 2H), 6.90-6.83 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 171.03, 164.52, 160.95 (d, J_{CF} = 246.3 Hz)，130.30 (d, J_{CF} = 8.0 Hz), 129.80, 128.85, 127.78, 127.69, 127.62, 127.38, 126.82, 126.26, 124.61 (d, J_{CF} = 3.5 Hz), 115.59 (d, J_{CF} = 21.9 Hz), 96.03 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 233.01 ppm; ^{19}F NMR ($CDCl_3$, 470 MHz) δ = -114.93 ppm. GC-MS (EI) m/z: 395 (M^+, 12%), 393 (6), 123 (100).



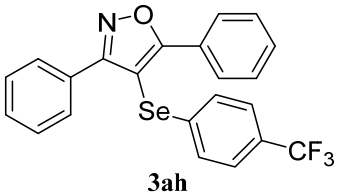
3af

4-((4-Chlorophenyl)selenyl)-3,5-diphenylisoxazole (3af) (CAS No.: 1422017-95-5): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 99% (203 mg); White solid; M.p.: 134-138 ℃ (lit.[5] M.p.: 132-134 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.16-8.10 (m, 2H), 7.83-7.77 (m, 2H), 7.51-7.41 (m, 6H), 7.17-7.10 (m, 4H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.54, 165.65, 132.87, 131.02, 130.24, 130.05, 129.79, 129.72, 128.86, 128.66, 128.56, 127.92, 127.29, 96.26 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 238.22 ppm. GC-MS (EI) m/z: 411 (M^+, 3%), 309 (2), 105 (100).



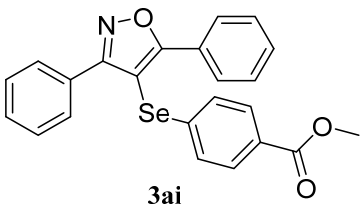
3ag

4-((4-Bromophenyl)selenyl)-3,5-diphenylisoxazole (3ag): Flash column chromatography eluent (petroleum ether/ethyl acetate = 20:1); Yield: 91% (208 mg); White solid; M.p.: 144-147 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.11-8.06 (m, 2H), 7.77-7.71 (m, 2H), 7.51-7.40 (m, 6H), 7.31-7.27 (m, 2H), 7.06-7.01 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.58, 165.64, 132.58, 130.99, 130.50, 130.44, 130.00, 128.81, 128.60, 128.51, 127.89, 127.25, 120.77, 96.07 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 238.79 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{21}H_{15}BrNOSe^+$ 455.9497, found 455.9493.



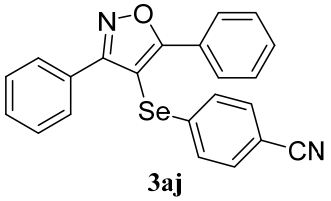
3ah

3,5-Diphenyl-4-((4-(trifluoromethyl)phenyl)selenyl)isoxazole (3ah) (CAS No.: 2487302-13-4): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 83% (185 mg); White solid; M.p.: 152-154 ℃ (lit.[7] M.p.: 101-103 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.11-8.05 (m, 2H), 7.79-7.73 (m, 2H), 7.51-7.40 (m, 8H), 7.29 (d, J = 8.2 Hz, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.94, 165.65, 136.92, 131.11, 130.08, 128.85, 128.82 (q, J_{CF} = 32.5 Hz), 128.74, 128.55, 128.47, 128.42, 127.85, 127.13, 126.29 (q, J_{CF} = 3.7 Hz), 123.99 (q, J_{CF} = 271.7 Hz), 95.29 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 250.00 ppm; ^{19}F NMR (470 MHz, $CDCl_3$) δ = -62.55 ppm. GC-MS (EI) m/z: 445 (M^+, 8%), 443 (4), 105 (100).



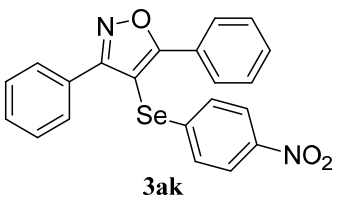
3ai

Methyl 4-((3,5-diphenylisoxazol-4-yl)selenyl)benzoate (3ai): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 87% (190 mg); White solid; M.p.: 161-163 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 7.99-7.93 (m, 2H), 7.77-7.72 (m, 2H), 7.66-7.60 (m, 2H), 7.38-7.27 (m, 6H), 7.16-7.12 (m, 2H), 3.76 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.96, 166.56, 165.70, 138.67, 131.05, 130.52, 130.04, 128.82, 128.73, 128.51, 128.38, 127.85, 127.15, 95.28, 52.13 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 252.41 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{23}H_{18}NO_3Se^+$ 436.0447, found 436.0432.



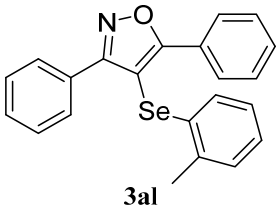
3aj

4-((3,5-Diphenylisoxazol-4-yl)selenyl)benzonitrile (3aj): Flash column chromatography eluent (petroleum ether/ethyl acetate = 10:1); Yield: 95% (191 mg); White solid; M.p.: 159-162 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.07-8.03 (m, 1H), 7.76-7.71 (m, 1H), 7.52-7.40 (m, 4H), 7.29-7.26 (m, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 173.09, 165.50, 139.15, 132.78, 131.24, 130.18, 128.91, 128.65, 128.60, 128.52, 128.28, 127.81, 126.94, 118.45, 110.12, 94.81 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 262.13 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{22}H_{15}N_2OSe^+$ 403.0345, found 403.0347.



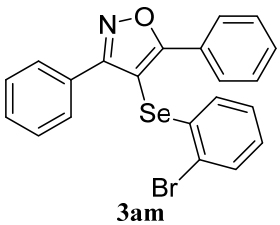
3ak

4-((3,5-Diphenylisoxazol-4-yl)selenyl)benzonitrile (3ak): Flash column chromatography eluent (petroleum ether/ethyl acetate = 10:1); Yield: 70% (147 mg); White solid; M.p.: 159-161 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.07-7.98 (m, 1H), 7.75-7.69 (m, 1H), 7.52-7.37 (m, 4H), 7.34-7.28 (m, 1H) ppm; ^{13}C NMR ($CDCl_3$, 126 MHz): δ = 172.20, 164.47, 145.53, 140.76, 130.29, 129.21, 127.91, 127.60, 127.18, 126.78, 125.85, 123.37, 93.67 ppm; ^{77}Se NMR ($CDCl_3$, 95 MHz): δ = 265.03 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{21}H_{15}N_2O_3Se^+$ 423.0243, found 423.0240.



3al

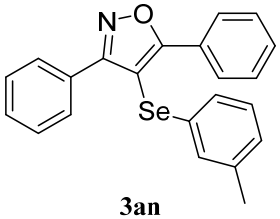
3,5-Diphenyl-4-(o-tolylselenyl)isoxazole (3al) (CAS No.: 1422017-98-8): Flash column chromatography eluent (petroleum ether/ethyl acetate = 75:1); Yield: 96% (188 mg); Yellow solid; M.p.: 130-132 ℃ (lit.[6] M.p.: 89-90 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.14-8.08 (m, 2H), 7.82-7.75 (m, 2H), 7.50-7.39 (m, 6H), 7.17 (d, J = 7.6 Hz, 1H), 7.15-7.08 (m, 1H), 7.05-6.99 (m, 2H), 2.38 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 171.75, 164.93, 135.47, 131.32, 129.73, 129.31, 128.78, 127.68, 127.66, 127.63, 127.33, 126.77, 126.30, 126.09, 125.34, 94.73, 20.15 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 208.91 ppm. GC-MS (EI) m/z: 391 (M^+, 10%), 389 (6), 105 (100).



3am

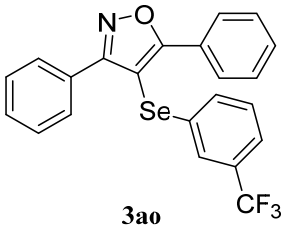
4-((2-Bromophenyl)selenyl)-3,5-diphenylisoxazole (3am): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 87% (198 mg); White solid; M.p.: 149-151 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.11-8.04 (m, 2H), 7.83-7.76 (m, 2H), 7.54-7.41 (m, 7H), 7.12 (td, J = 7.6, 1.4

Hz, 1H), 7.05 (td, J = 7.6, 1.7 Hz, 1H), 6.95 (dd, J = 7.9, 1.6 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 173.32, 165.81, 134.70, 133.06, 131.07, 130.09, 128.87, 128.69, 128.60, 128.49, 128.34, 127.88, 127.59, 127.16, 122.27, 96.25 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 265.40 ppm. HRMS (+ESI) m/z: [M + H$^+$] calculated for $C_{21}H_{15}BrNOSe^+$ 455.9497, found 455.9470.



3an

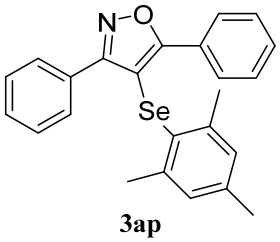
3,5-Diphenyl-4-(m-tolylselenyl)isoxazole (3an) (CAS No.: 2487302-15-6): Flash column chromatography eluent (petroleum ether/ethyl acetate = 75:1); Yield: 99% (193 mg); White solid; M.p.: 99-101 ℃ (lit.[7] M.p.: 99-100 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.19-8.13 (m, 2H), 7.86-7.80 (m, 2H), 7.52-7.41 (m, 6H), 7.10 (t, J = 7.6 Hz, 1H) , 7.07-6.97 (m, 3H), 2.25 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.48, 165.90, 139.45, 131.45, 130.84, 129.90, 129.42, 129.40, 128.95, 128.88, 128.77, 128.47, 127.98, 127.60, 127.52, 125.87, 96.49, 21.40 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 270.62 ppm. GC-MS (EI) m/z: 391 (M$^+$, 13%), 389 (7), 105 (100).



3ao

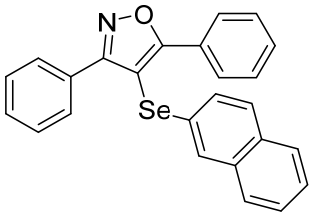
3,5-Diphenyl-4-((3-(trifluoromethyl)phenyl)selenyl)isoxazole (3ao) (CAS No.: 1422017-96-6): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 83% (184 mg); White solid; M.p.: 90-93 ℃ (lit.[6] M.p.: 70-71 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.10-8.07 (m, 2H), 7.76-7.72 (m, 2H), 7.51-7.47 (m, 3H), 7.45-7.37 (m, 5H), 7.30 (d, J = 8.0 Hz, 1H), 7.25 (t, J = 7.8 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 171.48, 164.52, 131.46, 131.13, 130.57(q, J_{CF} = 32.7 Hz), 129.97,

128.97, 128.78, 127.76, 127.69, 127.45, 127.41, 126.83, 126.09, 124.73 (q, J_{CF} = 3.8 Hz), 122.50 (q, J_{CF} = 3.4 Hz), 122.44 (q, J_{CF} = 272.8 Hz), 94.91 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 248.49 ppm; ^{19}F NMR ($CDCl_3$, 470 MHz) δ = -62.82 ppm. GC-MS (EI) m/z: 445 (M^+, 5%), 443 (3), 105 (100).



3ap

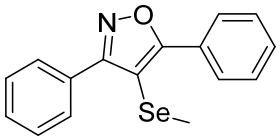
4-(Mesitylselenyl)-3,5-diphenylisoxazole (3ap): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 93% (195 mg); Yellow solid; M.p.: 50-53 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.05-7.98 (m, 2H), 7.52-7.44 (m, 5H), 7.43-7.39 (m, 1H), 7.37-7.32 (m, 2H), 6.66 (s, 2H), 2.16 (s, 3H), 2.08 (s, 6H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 169.74, 166.13, 141.59, 138.06, 130.28, 129.34, 129.23, 128.85, 128.81, 128.52, 128.09, 127.92, 127.41, 99.88, 23.70, 20.79 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 149.85 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{24}H_{22}NOSe^+$ 420.0862, found 420.0855.



3aq

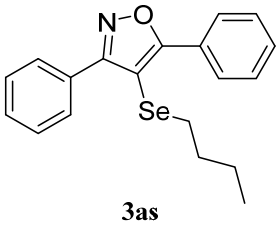
4-(Naphthalen-2-ylselenyl)-3,5-diphenylisoxazole (3aq): Flash column chromatography eluent (petroleum ether/ethyl acetate = 20:1); Yield: 88% (187 mg); White solid; M.p.: 148-150 °C. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.21-8.16 (m, 2H), 7.87-7.82 (m, 2H), 7.80-7.76 (m, 1H), 7.71 (d, J = 8.6 Hz, 1H), 7.69-7.63 (m, 2H), 7.50-7.40 (m, 8H), 7.34 (dd, J = 8.5, 1.9 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 171.68, 164.86, 133.04, 130.90, 129.79, 128.83, 128.16, 128.02, 127.77, 127.67, 127.62, 127.37, 126.82, 126.71, 126.28, 126.01, 125.84, 125.62, 125.21, 124.85, 95.03 ppm; ^{77}Se NMR ($CDCl_3$,

95.5 MHz): δ = 240.25 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{25}H_{18}NOSe^+$ 428.0549, found 428.0550.



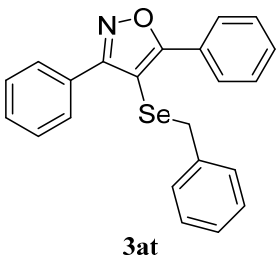
3ar

4-(Methylselenyl)-3,5-diphenylisoxazole (3ar) (CAS No.: 2487302-18-9): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 97% (152 mg); White solid; M.p.: 95-97 ℃ (lit.[7] M.p.: 95-97 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.24 8.21-8.15 (m, 2H), 7.99-7.94 (m, 2H), 7.56-7.49 (m, 6H), 1.95 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 169.14, 163.87, 129.36, 128.75, 128.18, 127.63, 127.48, 126.78, 126.71, 96.81, 8.21 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 36.96 ppm. GC-MS (EI) m/z: 315 (M^+, 31%), 313 (16), 105 (100).



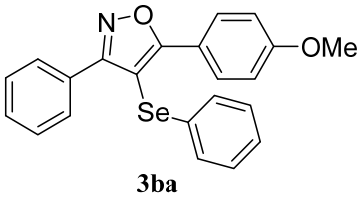
3as

4-(Butylselenyl)-3,5-diphenylisoxazole (3as) (CAS No.: 1422017-99-9): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 97% (172 mg); Yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.20 (dd, J = 8.1, 1.5 Hz, 2H), 7.95 (dd, J = 6.6, 3.0 Hz, 2H), 7.55-7.48 (m, 6H), 2.47 (t, J = 7.3 Hz, 2H), 1.35 (p, J = 7.3 Hz, 2H), 1.15 (h, J = 7.4 Hz, 2H), 0.68 (t, J = 7.3 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 170.66, 165.36, 130.40, 129.78, 129.34, 128.89, 128.64, 128.49, 127.97, 127.88, 96.94, 31.57, 29.08, 22.44, 13.33 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 109.27 ppm. GC-MS (EI) m/z: 357 (M^+, 20%), 355 (10), 105 (100).



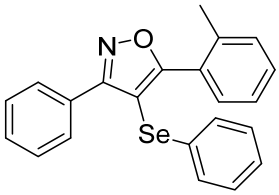
3at

4-(Benzylselenyl)-3,5-diphenylisoxazole (3at): Flash column chromatography eluent (petroleum ether/ethyl acetate = 30:1); Yield: 92% (179 mg); Yellow solid; M.p.: 79-81 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.03-7.99 (m, 2H), 7.88-7.85 (m, 2H), 7.53-7.49 (m, 3H), 7.48-7.45 (m, 3H), 7.11-7.07 (m, 3H), 6.89-6.85 (m, 2H), 3.67 (s, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 170.36, 164.25, 136.10, 129.29, 128.69, 128.01, 127.79, 127.65, 127.40, 127.35, 127.23, 126.73, 126.57, 125.99, 95.62, 31.19 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 198.59 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{22}H_{18}NOSe^+$ 392.0549, found 392.0564.



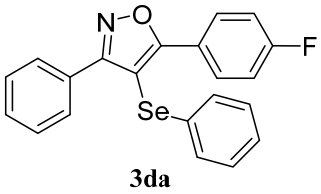
3ba

5-(4-Methoxyphenyl)-3-phenyl-4-(phenylselenyl)isoxazole (3ba) (CAS No.: 1422018-04-9): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 95% (193 mg); White solid; M.p.: 150-152 ℃ (lit.[5] M.p.: 132-134 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.16-8.06 (m, 2H), 7.82-7.70 (m, 2H), 7.47-7.38 (m, 3H), 7.24-7.11 (m, 5H), 7.01-6.95 (m, 2H), 3.84 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.48, 165.86, 161.57, 131.89, 129.82, 129.59, 129.51, 128.88, 128.60, 128.41, 126.54, 120.03, 114.19, 94.76, 55.39 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 235.95 ppm. GC-MS (EI) m/z: 407 (M^+, 8%), 405 (4), 135 (100).



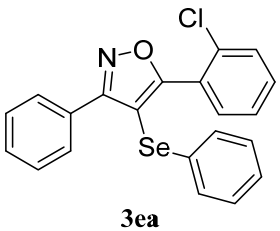
3ca

3-Phenyl-4-(phenylselenyl)-5-(o-tolyl)isoxazole (3ca) (CAS No.: 1422018-02-7): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 97% (190 mg); Yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 7.84-7.74 (m, 2H), 7.30-7.20 (m, 5H), 7.14 (d, J = 7.6 Hz, 1H), 7.08 (t, J = 7.5 Hz, 1H), 6.98 (s, 5H), 2.21 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 175.59, 164.32, 138.21, 131.56, 130.77, 130.71, 130.54, 130.00, 129.63, 129.39, 128.90, 128.67, 128.58, 127.17, 126.78, 125.69, 99.45, 20.34 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 234.66 ppm. GC-MS (EI) m/z: 391 (M^+, 14%), 389 (7), 119 (100).



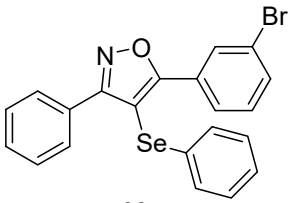
3da

5-(4-Fluorophenyl)-3-phenyl-4-(phenylselenyl)isoxazole (3da) (CAS No.: 2487302-29-2): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 98% (193 mg); White solid; M.p.: 93-95 ℃ (lit.[7] M.p.: 112-113 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.17-8.12 (m, 2H), 7.80-7.75 (m, 2H), 7.48-7.40 (m, 3H), 7.21-7.13 (m, 7H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 171.49, 165.86, 164.14 (d, J_{CF} = 253.1 Hz), 131.41, 130.10 (d, J_{CF} = 8.2 Hz), 129.95, 129.64, 128.84 (d, J_{CF} = 5.9 Hz), 128.67, 128.46, 126.77, 123.72 (d, J_{CF} = 3.4 Hz), 116.07, 115.89, 96.25 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 235.78 ppm; ^{19}F NMR ($CDCl_3$, 470 MHz) δ = -108.16 ppm. GC-MS (EI) m/z: 395 (M^+, 9%), 393 (4), 123 (100).



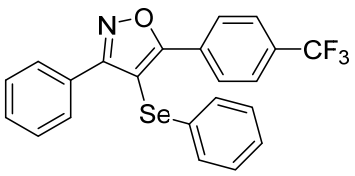
3ea

5-(2-Chlorophenyl)-3-phenyl-4-(phenylselenyl)isoxazole (3ea) (CAS No.: 2487302-28-1): Flash column chromatography eluent (petroleum ether/ethyl acetate = 30:1); Yield: 87% (179 mg); Yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 7.99-7.93 (m, 2H), 7.55-7.51 (m, 1H), 7.50-7.41 (m, 5H), 7.34 (td, J = 7.5, 0.9 Hz, 1H), 7.20-7.12 (m, 5H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.61, 164.15, 134.31, 132.03, 131.95, 130.97, 130.22, 130.06, 129.89, 129.36, 128.63, 128.58, 127.08, 126.89, 126.71, 101.04 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 236.40 ppm. GC-MS (EI) m/z: 411 (M^+, 12%), 409 (6), 139 (100).



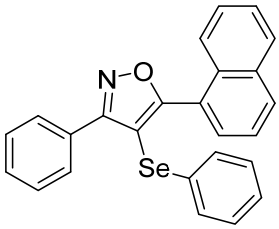
3fa

5-(3-Bromophenyl)-3-phenyl-4-(phenylselenyl)isoxazole (3fa) (CAS No.: 2734166-09-5): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 84% (191 mg); Yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.31-8.26 (m, 1H), 8.09-8.05 (m, 1H), 7.67-7.60 (m, 2H), 7.62-7.58 (m, 1H), 7.48-7.40 (m, 3H), 7.12 (t, J = 7.9 Hz, 1H), 7.05-6.97 (m, 5H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 170.51, 165.82, 133.71, 131.16, 130.80, 130.30, 130.05, 129.68, 129.31, 129.24, 128.94, 128.60, 128.53, 127.00, 126.41, 122.82, 97.78 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 236.30 ppm. GC-MS (EI) m/z: 455 (M^+, 10%), 453 (5), 169 (100).



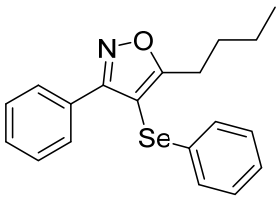
3ga

3-Phenyl-4-(phenylselenyl)-5-(4-(trifluoromethyl)phenyl)isoxazole (3ga): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 86% (192 mg); Yellow solid; M.p.: 106-110 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.26 (d, J = 8.2 Hz, 2H), 7.79-7.76 (m, 2H), 7.74 (d, J = 8.3 Hz, 2H), 7.48-7.40 (m, 3H), 7.23-7.14 (m, 5H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 170.67, 165.97, 132.24 (q, J_{CF} = 32.9 Hz), 131.03, 130.62, 130.07, 129.70, 129.03, 128.88, 128.51, 128.43, 128.21, 126.97, 125.72 (q, J_{CF} = 4.0 Hz), 123.74 (q, J_{CF} = 272.7 Hz), 98.16 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 236.54 ppm; ^{19}F NMR ($CDCl_3$, 470 MHz) δ = -62.94 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{22}H_{15}F_3NOSe^+$ 446.0266, found 446.0256.



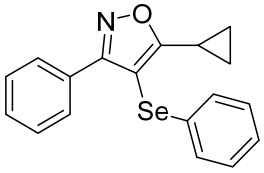
3ha

5-(Naphthalen-1-yl)-3-phenyl-4-(phenylselenyl)isoxazole (3ha) (CAS No.: 1422018-06-1): Flash column chromatography eluent (petroleum ether/ethyl acetate = 30:1); Yield: 97% (207 mg); White solid; M.p.: 114-116 ℃ (lit.[6] M.p.: 106-107 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.03-7.96 (m, 3H), 7.96-7.91 (m, 2H), 7.64 (dd, J = 7.1, 0.9 Hz, 1H), 7.59-7.50 (m, 3H), 7.50-7.44 (m, 3H), 7.14-7.04 (m, 5H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 174.63, 164.56, 133.59, 131.63, 131.28, 131.24, 130.00, 129.84, 129.27, 128.88, 128.72, 128.57, 128.50, 127.24, 126.74, 126.50, 125.43, 124.92, 124.85, 100.77 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 238.98 ppm. GC-MS (EI) m/z: 427 (M^+, 7%), 425 (3), 127 (100).



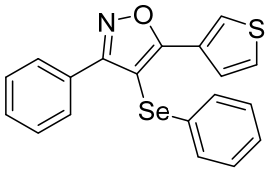
3ia

5-Butyl-3-phenyl-4-(phenylselenyl)isoxazole (3ia) (CAS No.: 1422018-07-2): Flash column chromatography eluent (petroleum ether/ethyl acetate = 75:1); Yield: 95% (170 mg); Yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 7.89-7.83 (m, 2H), 7.46-7.37 (m, 3H), 7.24-7.14 (m, 5H), 2.97 (t, J = 7.55 Hz, 2H), 1.76-1.69 (m, 2H), 1.39 (dq, J = 14.7, 7.4 Hz, 2H), 0.93 (t, J = 7.3 Hz, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 178.97, 163.87, 131.87, 129.85, 129.46, 128.99, 128.87, 128.50, 126.56, 97.23, 29.70, 26.45, 22.33, 13.70 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 215.89 ppm. GC-MS (EI) m/z: 357 (M^+, 36%), 355 (18), 169 (100).



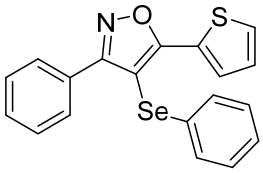
3ja

5-Cyclopropyl-3-phenyl-4-(phenylselenyl)isoxazole (3ja) (CAS No.: 2487302-31-6): Flash column chromatography eluent (petroleum ether/ethyl acetate = 75:1); Yield: 88% (150 mg); Yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 7.82-7.70 (m, 2H), 7.40-7.33 (m, 3H), 7.22-7.12 (m, 5H), 2.41-2.32 (m, 1H), 1.27-1.23 (m, 2H), 1.09-1.04 (m, 2H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 178.85, 164.23, 131.95, 129.85, 129.48, 128.86, 128.57, 128.48, 126.52, 96.37, 9.13, 8.82 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 211.14 ppm. GC-MS (EI) m/z: 341 (M^+, 16%), 339(8), 69 (100).



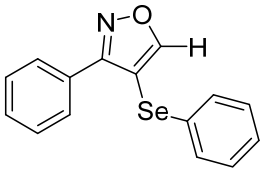
3ka

3-Phenyl-4-(phenylselenyl)-5-(thiophen-3-yl)isoxazole (3ka) (CAS No.:2734166-10-8): Flash column chromatography eluent (petroleum ether/ethyl acetate = 75:1); Yield: 91% (174 mg); White solid; M.p.: 105-107 ℃ (lit.[6] M.p.: 104-106 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.31 (dd, J = 3.0, 1.1 Hz, 1H), 7.88 (dd, J = 5.1, 1.2 Hz, 1H), 7.83-7.80 (m, 2H), 7.46-7.39 (m, 4H), 7.25-7.17 (m, 5H) ppm; ^{13}C NMR ($CDCl_3$, 126 MHz): δ = 169.27, 165.54, 131.36, 129.94, 129.68, 128.88, 128.74, 128.71, 128.48, 128.30, 127.37, 126.72, 126.46, 126.35, 95.52 ppm; ^{77}Se NMR ($CDCl_3$, 95 MHz): δ = 230.27 ppm. GC-MS(EI) m/z: 383 (M^+, 7%), 381 (4), 111 (100).



3la

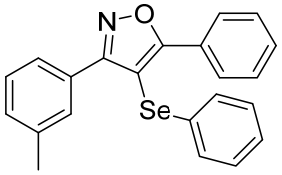
3-Phenyl-4-(phenylselenyl)-5-(thiophen-2-yl)isoxazole (3la): Flash column chromatography eluent (petroleum ether/ethyl acetate = 75:1); Yield: 96% (183 mg); Yellow solid; M.p.: 97-99 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 7.96 (dd, J = 3.7, 0.9 Hz, 1H), 7.84-7.78 (m, 2H), 7.51 (dd, J = 5.0, 1.1 Hz, 1H), 7.47-7.40 (m, 3H), 7.25-7.14 (m, 6H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 168.73, 165.42, 131.21, 130.04, 129.99, 129.59, 129.45, 128.97, 128.81, 128.49, 128.30, 127.66, 126.79, 95.52 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 227.89 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{19}H_{14}NOSSe^+$ 383.9956, found 383.9969.



3ma

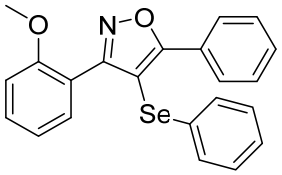
3-Phenyl-4-(phenylselenyl)isoxazole (3ma): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 25% (38 mg); Yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.55 (s, 1H), 7.86-7.82 (m, 2H), 7.47-7.39 (m, 3H), 7.32-7.27 (m, 2H), 7.25-7.20 (m, 3H) ppm; ^{13}C NMR ($CDCl_3$,

125 MHz): δ = 164.01, 162.67, 130.68, 130.34, 130.04, 129.53, 128.59, 128.46, 128.13, 127.22, 101.48 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 224.62 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{15}H_{12}NOSe^+$ 302.0079, found 302.0087.



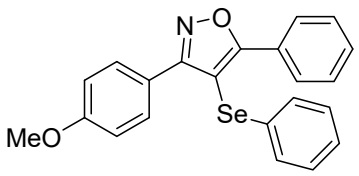
3na

5-Phenyl-4-(phenylselenyl)-3-(m-tolyl)isoxazole (3na) (CAS No.: 2487302-24-7): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 95% (186 mg); Yellow oil. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.23-8.17 (m, 2H), 7.67-7.62 (m, 2H), 7.53-7.49 (m, 3H), 7.35 (t, J = 7.6 Hz, 1H), 7.32-7.27 (m, 3H), 7.26-7.18 (m, 3H), 2.40 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.46, 166.03, 138.12, 131.86, 130.88, 130.70, 129.64, 128.97, 128.80, 128.70, 128.39, 127.96, 127.53, 126.71, 126.03, 96.61, 21.48 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 238.12 ppm. GC-MS (EI) m/z: 391 (M^+, 20%), 389(11), 105 (100).



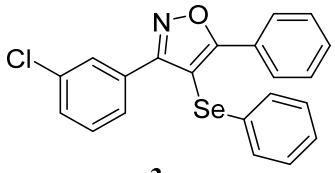
3oa

3-(2-Methoxyphenyl)-5-phenyl-4-(phenylselenyl)isoxazole (3oa) (CAS No.: 2734166-11-9): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 87% (176 mg); White solid; M.p.: 127-129 ℃ (lit.[5] M.p.: 124-126 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.24-8.18 (m, 2H), 7.48-7.43 (m, 4H), 7.40 (dd, J = 7.4, 1.8 Hz, 1H), 7.22-7.13 (m, 5H), 7.06-7.02 (m, 1H), 6.97 (d, J = 8.3 Hz, 1H), 3.62 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 169.48, 164.81, 156.46, 130.38, 130.27, 130.12, 129.46, 128.21, 128.04, 127.57, 126.47, 126.38, 125.27, 119.30, 116.82, 109.80, 98.08, 54.04 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 243.56 ppm. GC-MS (EI) m/z: 407 (M^+, 8%), 405 (4), 105 (100).



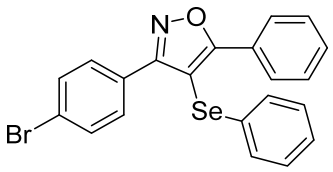
3pa

3-(4-Methoxyphenyl)-5-phenyl-4-(phenylselenyl)isoxazole (3pa): Flash column chromatography eluent (petroleum ether/ethyl acetate = 25:1); Yield: 89% (181 mg); White solid; M.p.: 158-160 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.16 -8.05 (m, 2H), 7.81-7.71 (m, 2H), 7.50-7.44 (m, 3H), 7.25-7.12 (m, 5H), 6.96-6.91 (m, 2H), 3.82 (s, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.50, 165.29, 160.94, 131.80, 130.76, 130.21, 129.59, 128.70, 128.67, 127.93, 127.52, 126.58, 121.14, 113.93, 96.17, 55.29 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 237.07 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{22}H_{18}NO_2Se^+$ 408.0498, found 408.0487.



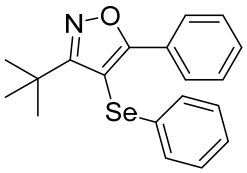
3qa

3-(3-Chlorophenyl)-5-phenyl-4-(phenylselenyl)isoxazole (3qa): Flash column chromatography eluent (petroleum ether/ethyl acetate = 20:1); Yield: 90% (186 mg); White solid; M.p.: 69-72 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.17-8.09 (m, 2H), 7.78 (t, J = 1.8 Hz, 1H), 7.66 (dt, J = 7.5, 1.1 Hz, 1H), 7.52-7.46 (m, 3H), 7.44-7.38 (m, 1H), 7.33 (t, J = 7.8 Hz, 1H), 7.23-7.14 (m, 5H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.62, 164.56, 134.34, 131.18, 130.96, 130.49, 129.92, 129.68, 129.63, 129.20, 128.97, 128.79, 127.92, 127.25, 126.98, 126.91, 96.58 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 236.15 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{21}H_{15}ClNOSe^+$ 412.0002, found 412.0008.



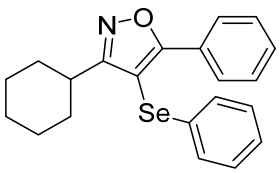
3ra

3-(4-Bromophenyl)-5-phenyl-4-(phenylselenyl)isoxazole (3ra): Flash column chromatography eluent (petroleum ether/ethyl acetate = 20:1); Yield: 86% (197 mg); White solid; M.p.: 129-132 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.16-8.10 (m, 2H), 7.71-7.66 (m, 2H), 7.57-7.52 (m, 2H), 7.50-7.45 (m, 3H), 7.23-7.15 (m, 5H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.81, 164.83, 131.73, 131.40, 131.00, 130.40, 129.71, 128.82, 127.93, 127.72, 127.25, 126.84, 124.56, 96.21 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 235.97 ppm. HRMS (+ESI) m/z: [M + H^+] calculated for $C_{21}H_{15}BrNOSe^+$ 455.9497, found 455.9489.



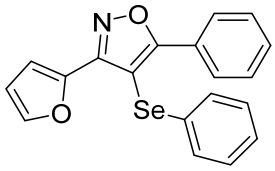
3sa

3-(Tert-butyl)-5-phenyl-4-(phenylselenyl)isoxazole (3sa) (CAS No.: 2734166-14-2): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 93% (166 mg); White solid; M.p.: 90-93 ℃ (lit.[5] M.p.: 82-84 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.02-7.96 (m, 2H), 7.44-7.37 (m, 3H), 7.23-7.18 (m, 2H), 7.18-7.13 (m, 3H), 1.48 (s, 9H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 173.03, 172.56, 132.61, 130.55, 129.52, 128.58, 127.96, 127.75, 127.59, 126.21, 94.61, 33.96, 28.78 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 238.73 ppm. GC-MS (EI) m/z: 357 (M^+, 14%), 355 (7), 105 (100).



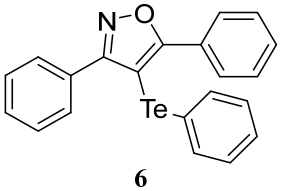
3ta

3-Cyclohexyl-5-phenyl-4-(phenylselenyl)isoxazole (3ta): Flash column chromatography eluent (petroleum ether/ethyl acetate = 75:1); Yield: 97% (185 mg); Yellow solid; M.p.: 78-81 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.10-8.04 (m, 2H), 7.45-7.39 (m, 3H), 7.23-7.15 (m, 5H), 2.80 (tt, J = 11.9, 3.4 Hz, 1H), 1.90 (d, J = 13.9 Hz, 2H), 1.84-1.76 (m, 2H), 1.73-1.57 (m, 3H), 1.37-1.25 (m, 3H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 171.09, 170.87, 131.57, 130.51, 129.50, 128.70, 128.64, 127.66, 127.58, 126.55, 96.61, 36.29, 31.57, 26.28, 25.90 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 216.74 ppm. HRMS (+ESI) m/z: [M + H$^+$] calculated for $C_{21}H_{22}NOSe^+$ 384.0862, found 384.0884.



3ua

3-(Furan-2-yl)-5-phenyl-4-(phenylselenyl)isoxazole (3ua) (CAS No.: 2734166-13-1): Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 88% (161 mg); Yellow solid; M.p.: 104-107 ℃ (lit.[5] M.p.: 104-106 ℃). ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.13-8.09 (m, 2H), 7.60-7.56 (m, 1H), 7.51-7.45 (m, 3H), 7.31 (d, J = 3.5 Hz, 1H), 7.28-7.18 (m, 5H), 6.48 (dd, J = 3.6, 1.8 Hz, 1H) ppm; ^{13}C NMR ($CDCl_3$, 125 MHz): δ = 172.64, 157.31, 144.13, 143.27, 131.03, 130.98, 129.68, 128.73, 128.62, 128.05, 127.02, 126.74, 113.26, 111.50, 94.32 ppm; ^{77}Se NMR ($CDCl_3$, 95.5 MHz): δ = 236.51 ppm. GC-MS (EI) m/z: 367 (M$^+$, 18%), 365 (8), 105 (100).



6

3,5-Diphenyl-4-(phenyltellenyl)isoxazole (6): Eluent: Flash column chromatography eluent (petroleum ether/ethyl acetate = 50:1); Yield: 46% (98 mg); Yellow solid; M.p.: 99-101 ℃. ^{1}H NMR ($CDCl_3$, 500 MHz): δ = 8.07-7.99 (m, 2H), 7.72-7.66 (m, 2H), 7.51-7.38 (m, 6H), 7.37-7.30 (m, 2H), 7.22-7.15 (m,

1H), 7.11 (t, J = 7.5 Hz, 2H) ppm; ^{13}C NMR ($CDCl_3$, 126 MHz): δ = 174.72, 167.96, 134.97, 130.68, 129.76, 129.71, 129.66, 129.29, 128.70, 128.58, 128.33, 128.06, 127.62, 115.32, 79.23 ppm. HRMS (+ESI) m/z: [M+H$^+$] calculated for $C_{21}H_{16}NOTe^+$ 428.0289, found 428.0273.

5. References:

[1] J. P. Waldo and R. C. Larock, *J. Org. Chem.* 2007, **72**, 9643-9647.

[2] D. Singh, A. M. Deobald, L. R. S. Camargo, G. Tabarelli, O. E. D. Rodrigues and A. L. Braga, *Org. Lett.* 2010, **12**, 3288-3291.

[3] T. Leng, G. Wu, Y. B. Zhou, W. X. Gao, J. C. Ding, X. B. Huang, M. C. Liu and H. Y. Wu, *Adv. Synth. Catal.* 2018, **360**, 4336-4340.

[4] J. Wang, X. X. Lu, R. P. Yang, B. B. Zhang, Z. H. Xiang, J. C. Li, L. Liu, S. Chao and X. Shang, *Org. Lett.* 2023, **25**, 8489-8494.

[5] Z. Yu, D. Zhang, X. Li, B. Zhang, Z. Yang, Y. Qian and Y. Du, *Asian J. Org. Chem.*, 2021, **10**, 3015-3019.

[6] A. Speranca, B. Godoi and G. Zeni, *J. Org. Chem.*, 2013, **78**, 1630-1637.

[7] J. Wu, Y. F. Yang, X. B. Huang, W. X. Gao, Y. B. Zhou, M. C. Liu and H. Y. Wu, *ACS Omega*, 2020, **5**, 23358-23363.

6. ^{1}H, ^{13}C, ^{77}Se and ^{19}F NMR spectra of products

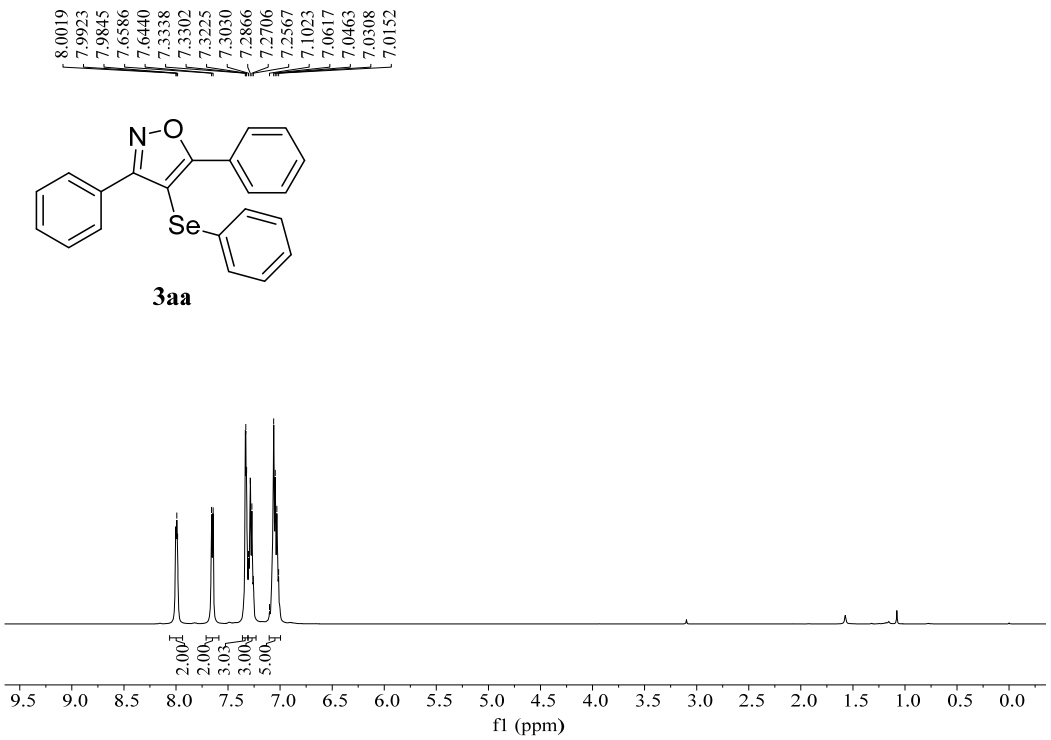


Figure S9 ^{1}H NMR (500 MHz) spectrum of **3aa** in $CDCl_3$

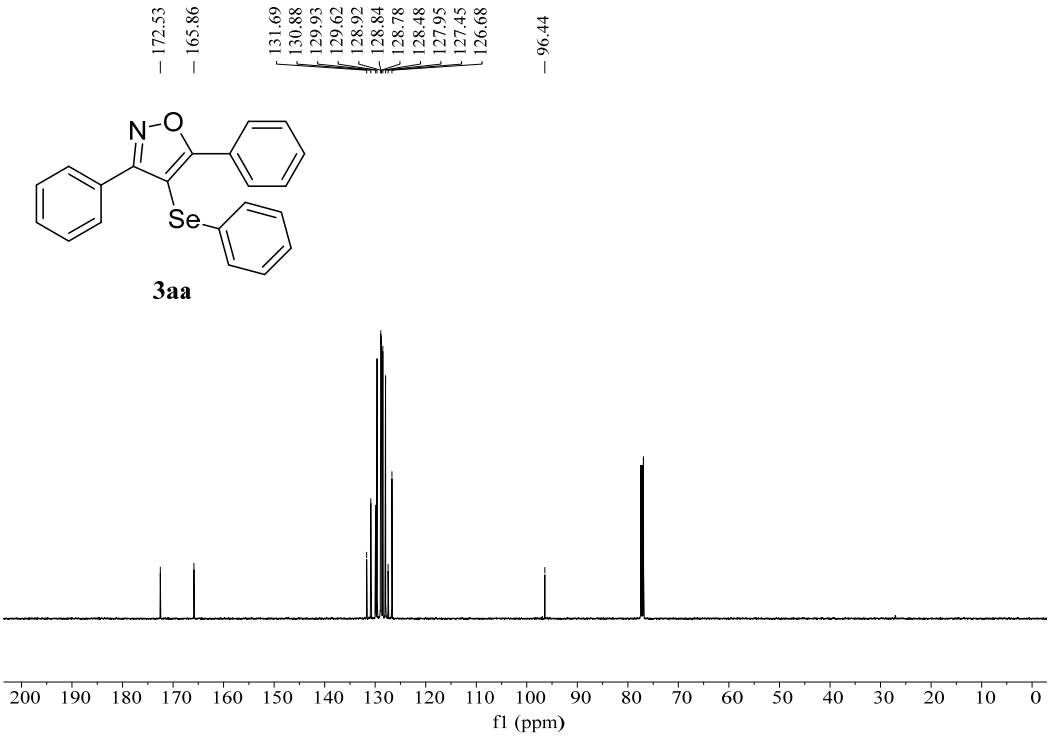


Figure S10 ^{13}C NMR (125 MHz) spectrum of **3aa** in $CDCl_3$

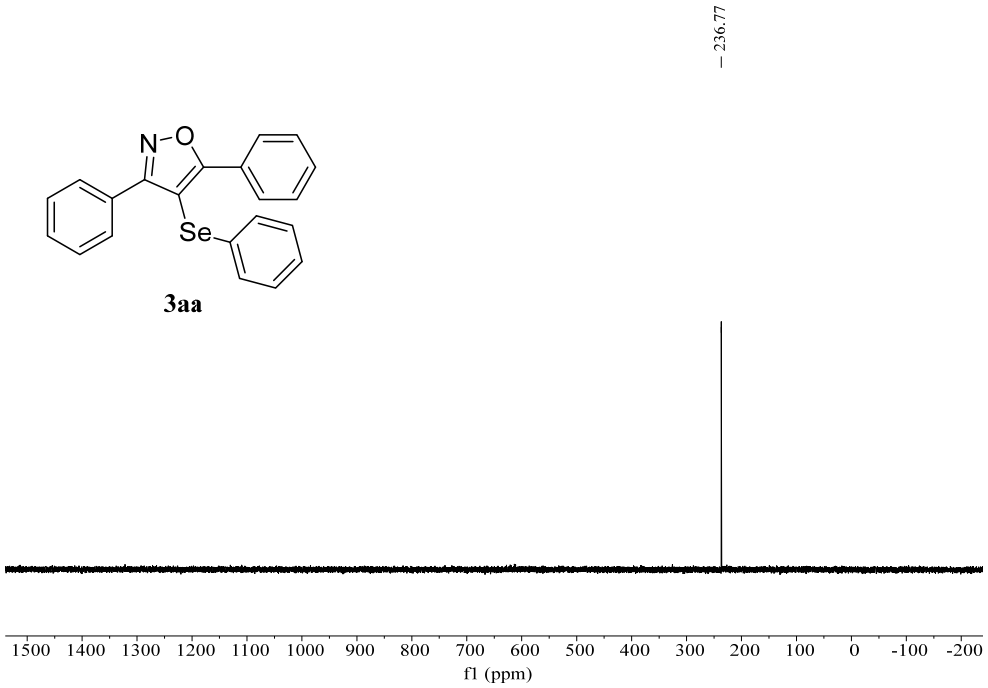


Figure S11 ^{77}Se NMR (95.5 MHz) spectrum of **3aa** in $CDCl_3$

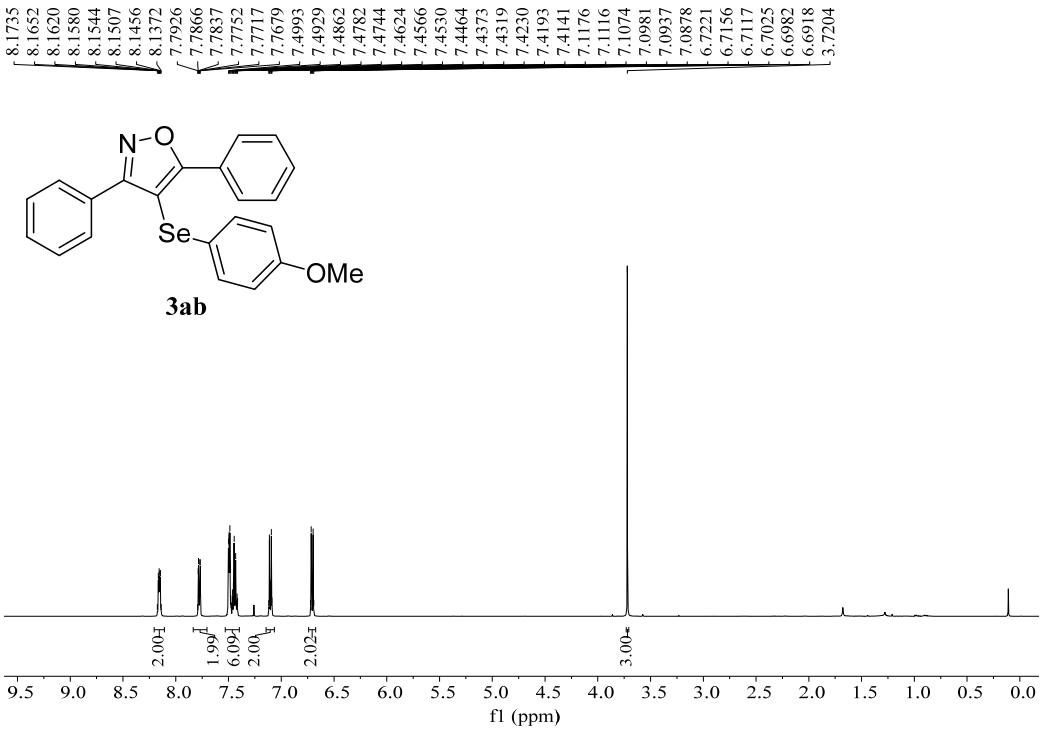


Figure S12 ^{1}H NMR (500 MHz) spectrum of **3ab** in $CDCl_3$

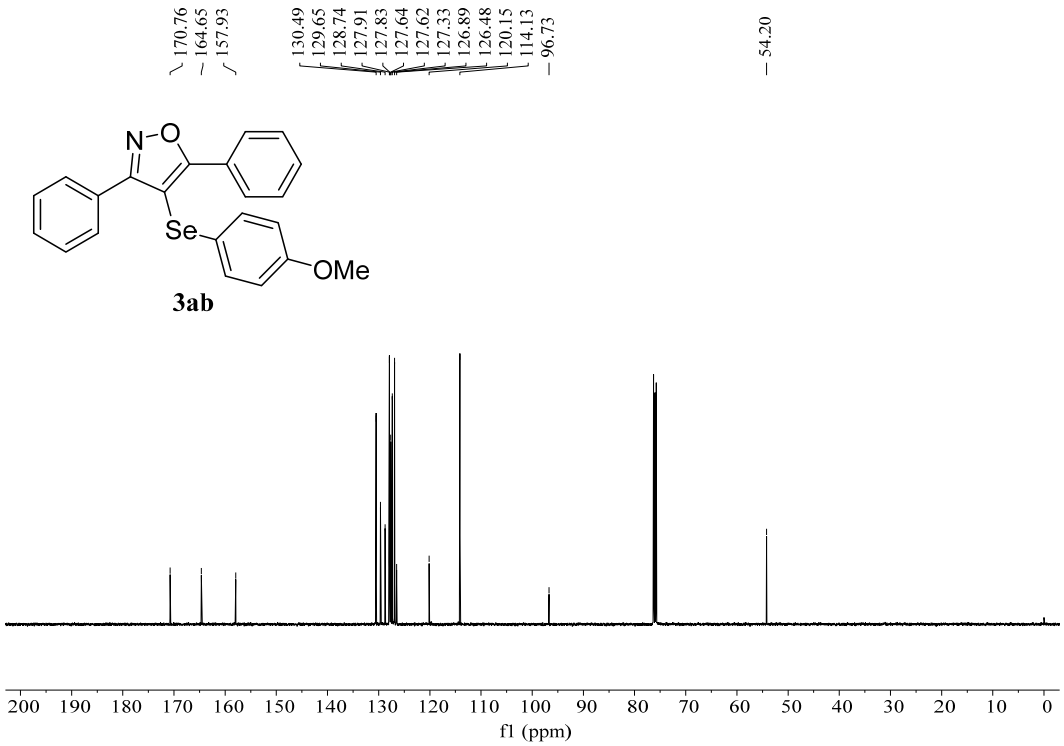


Figure S13 ^{13}C NMR (125 MHz) spectrum of **3ab** in $CDCl_3$

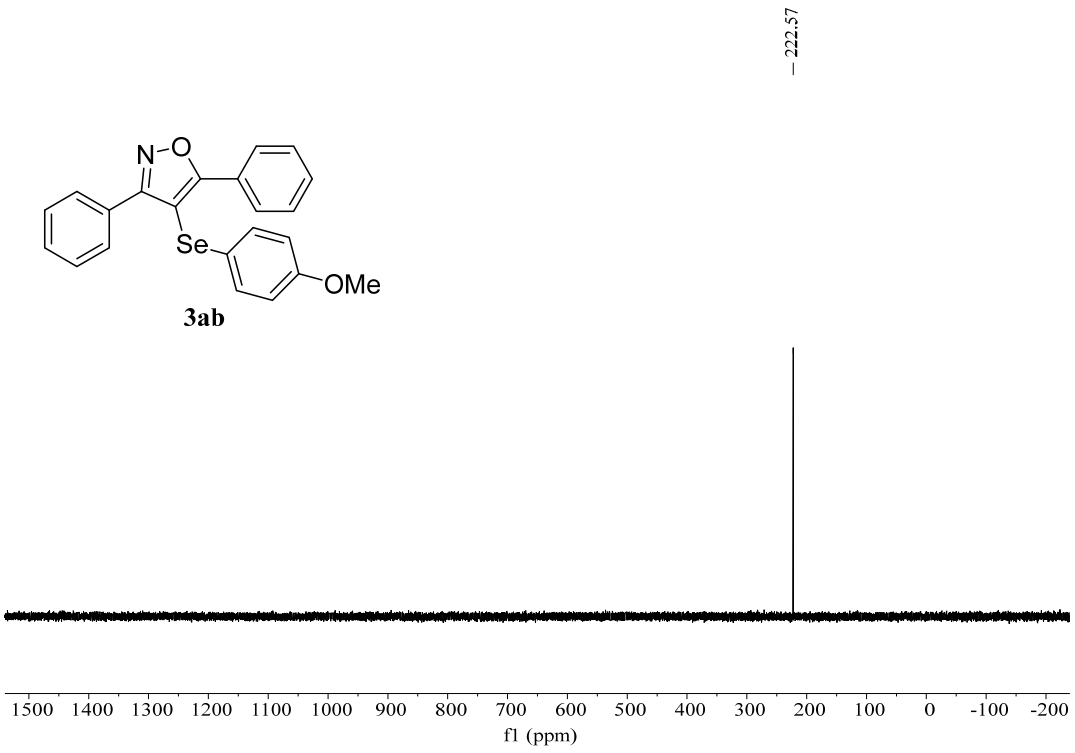


Figure S14 ^{77}Se NMR (95.5 MHz) spectrum of **3ab** in $CDCl_3$

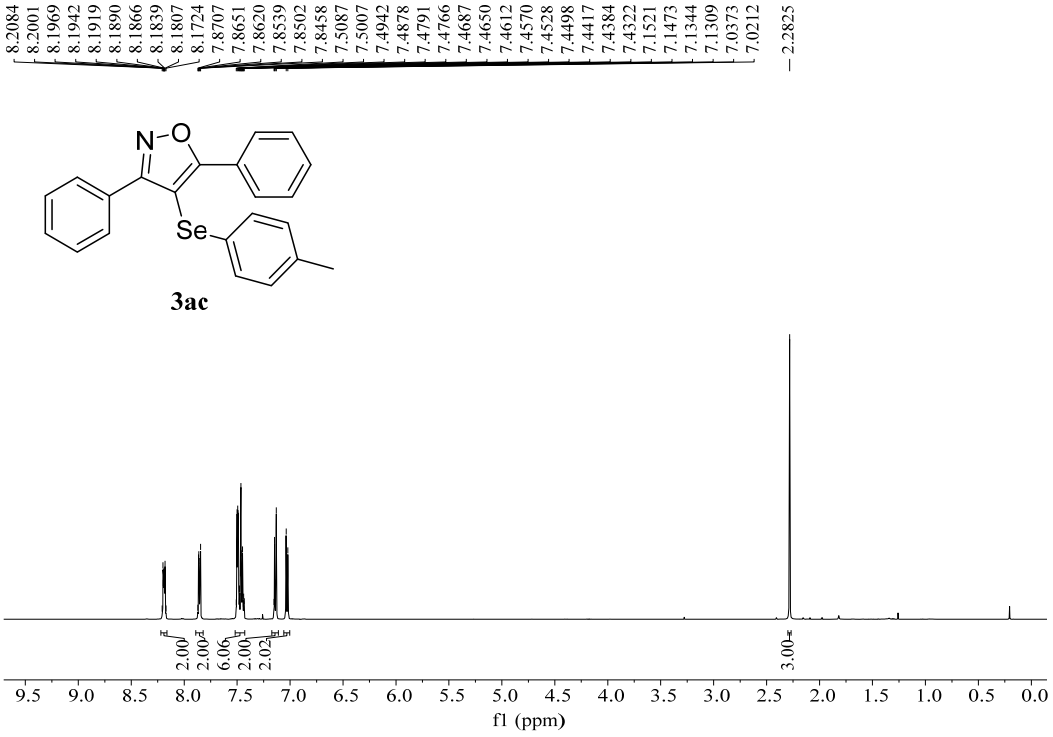


Figure S15 ^{1}H NMR (500 MHz) spectrum of **3ac** in $CDCl_3$

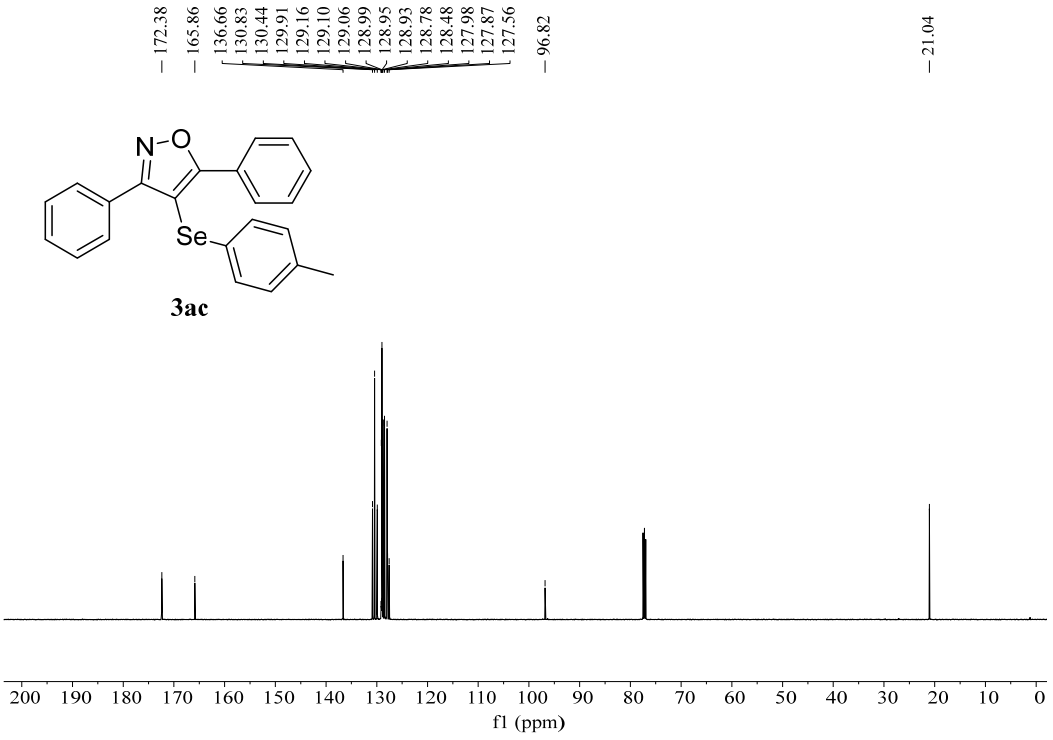


Figure S16 ^{13}C NMR (125 MHz) spectrum of **3ac** in $CDCl_3$

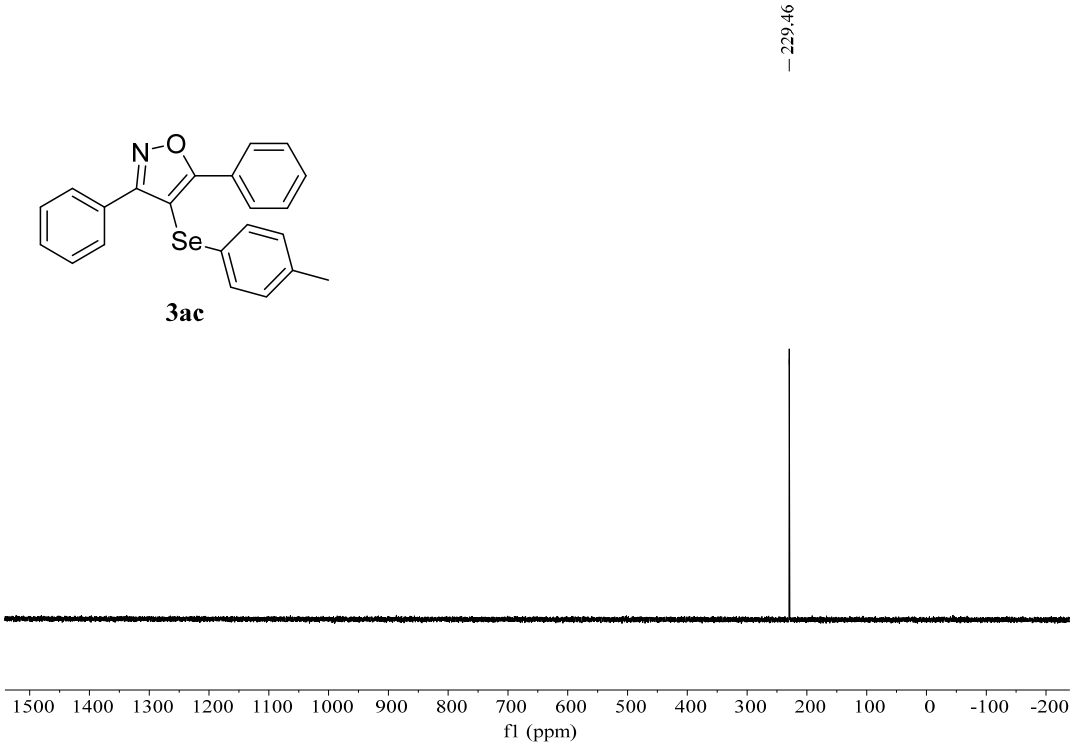


Figure S17 ^{77}Se NMR (95.5 MHz) spectrum of **3ac** in $CDCl_3$

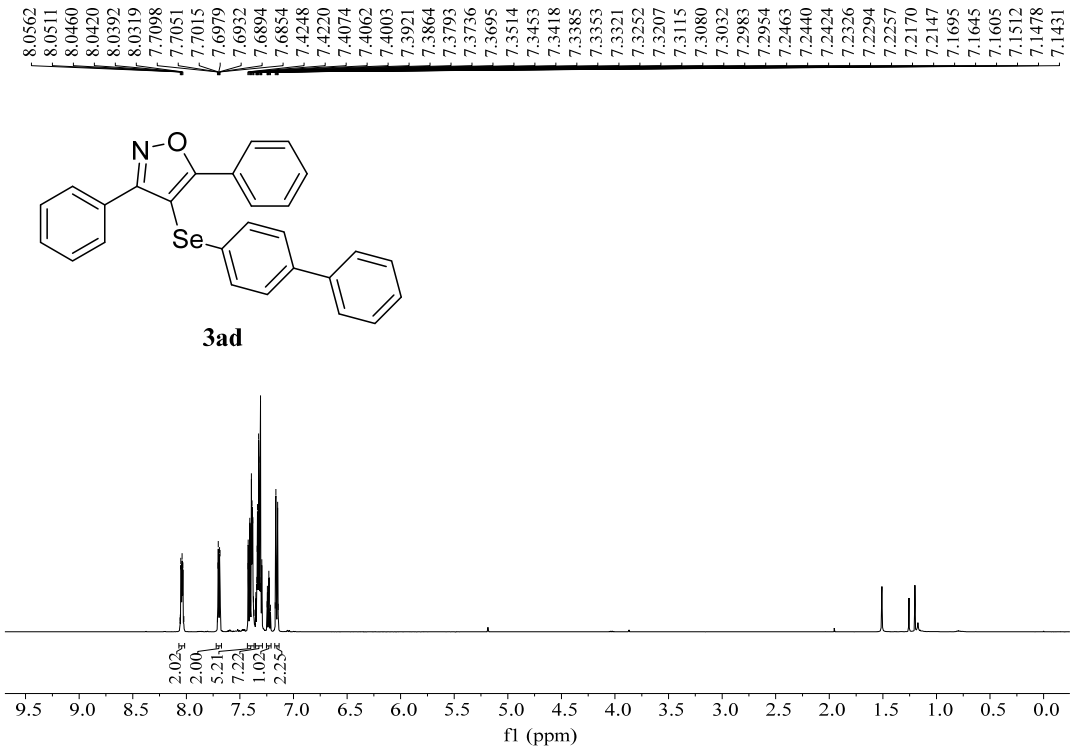


Figure S18 ^{1}H NMR (500 MHz) spectrum of **3ad** in $CDCl_3$

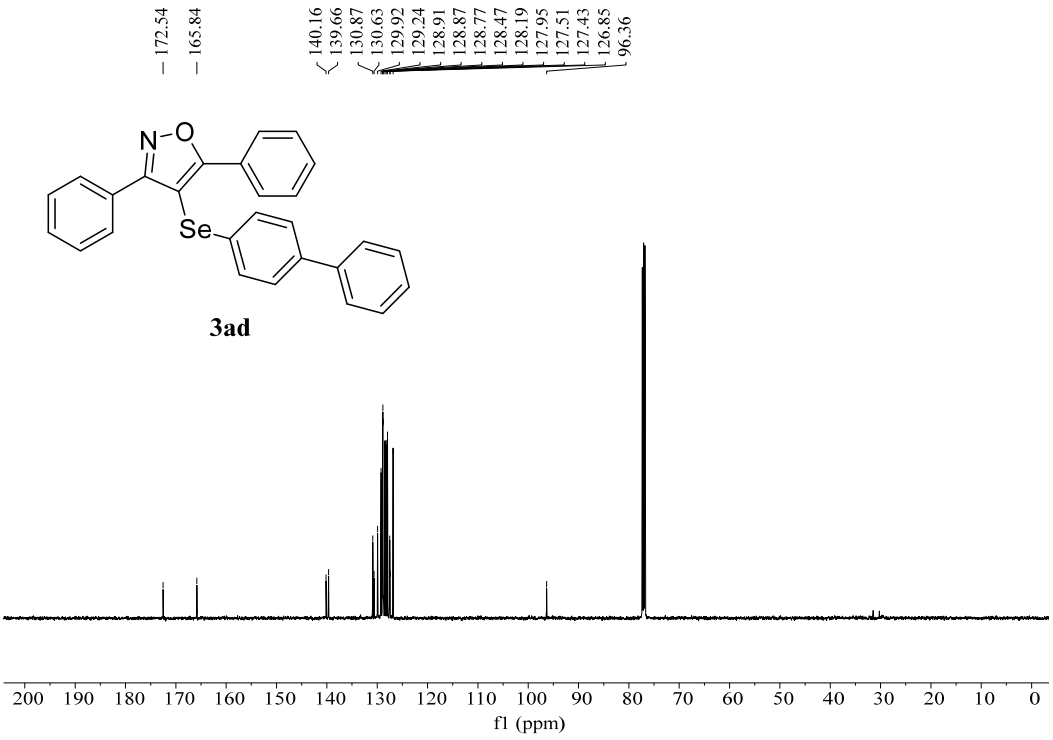


Figure S19 ^{13}C NMR (125 MHz) spectrum of **3ad** in $CDCl_3$

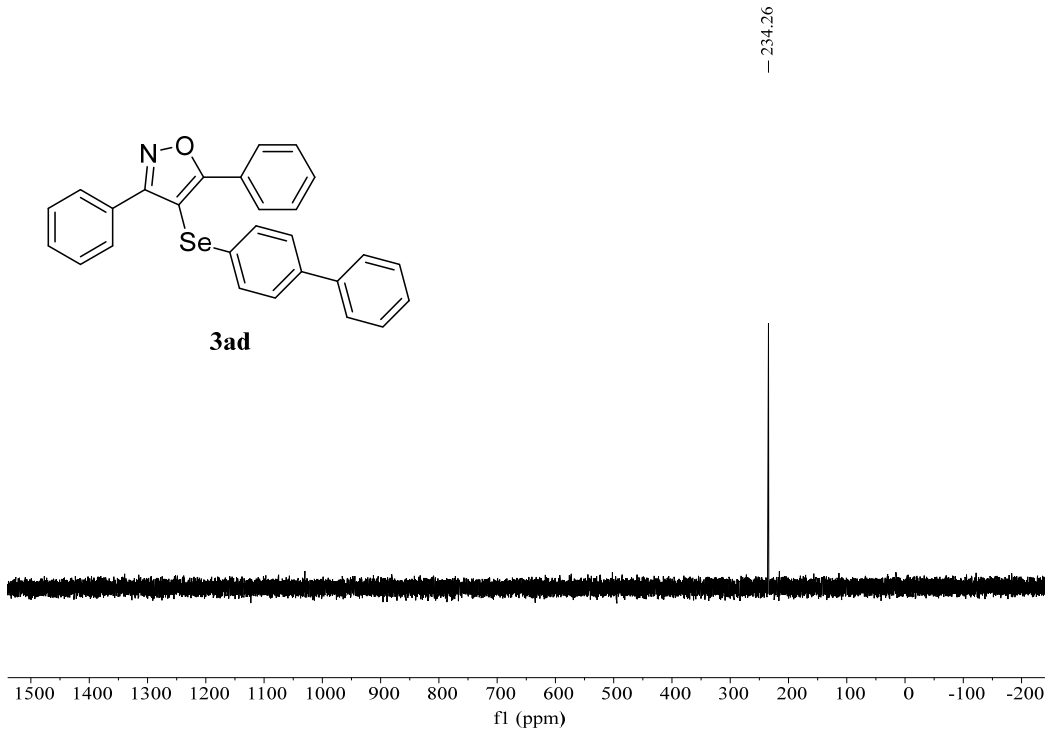


Figure S20 ^{77}Se NMR (95.5 MHz) spectrum of **3ad** in $CDCl_3$

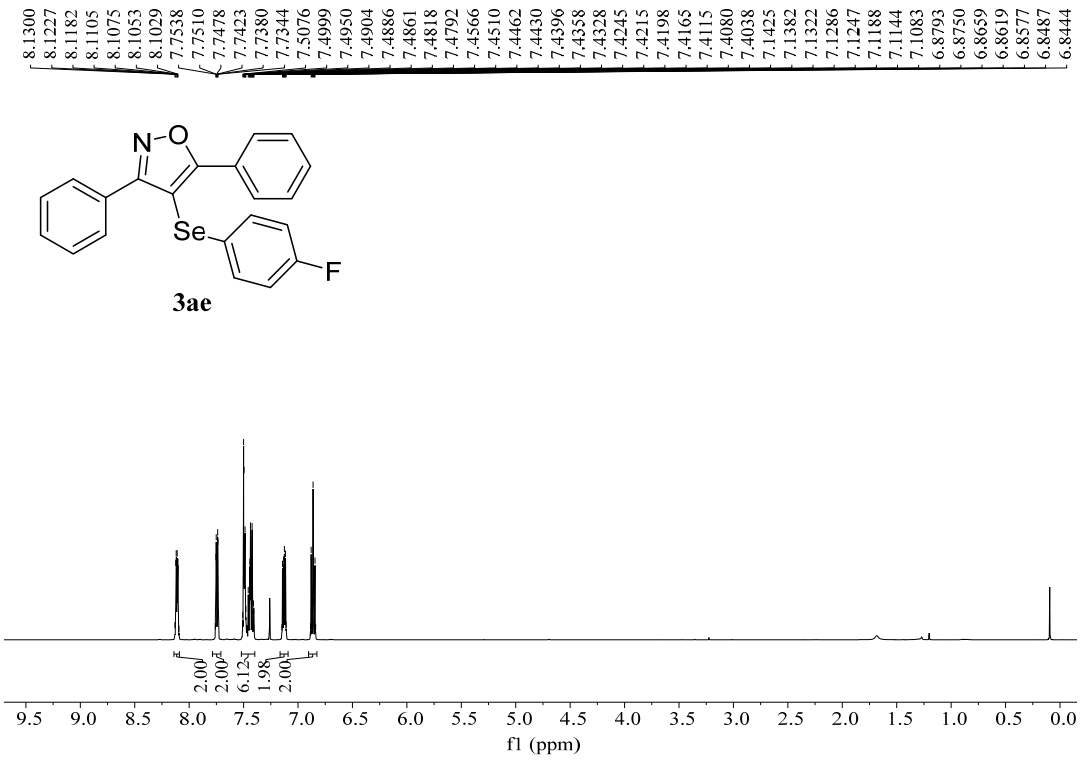


Figure S21 ^{1}H NMR (500 MHz) spectrum of **3ae** in $CDCl_3$

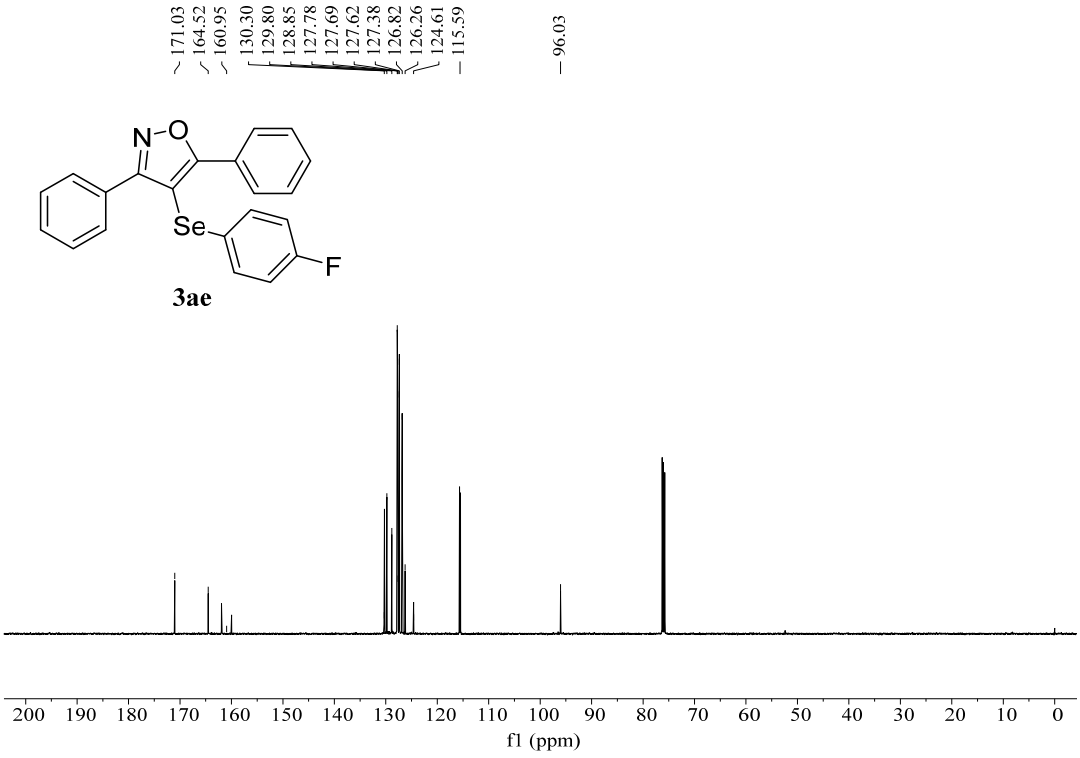


Figure S22 ^{13}C NMR (125 MHz) spectrum of **3ae** in $CDCl_3$

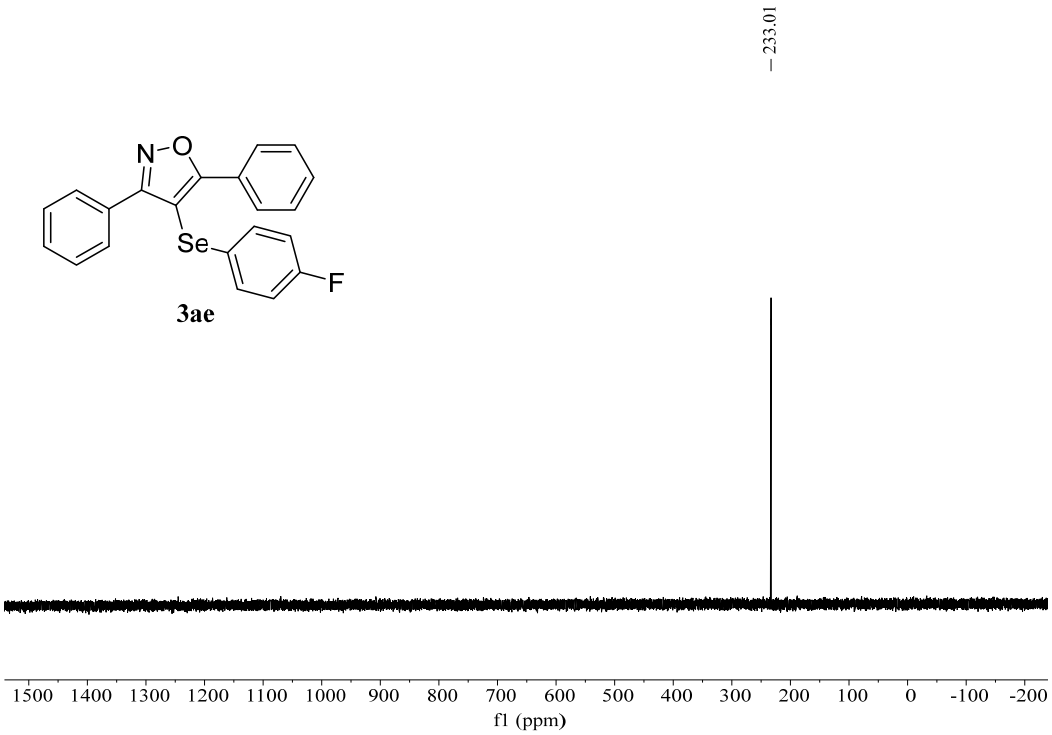


Figure S23 ^{77}Se NMR (95.5 MHz) spectrum of **3ae** in $CDCl_3$

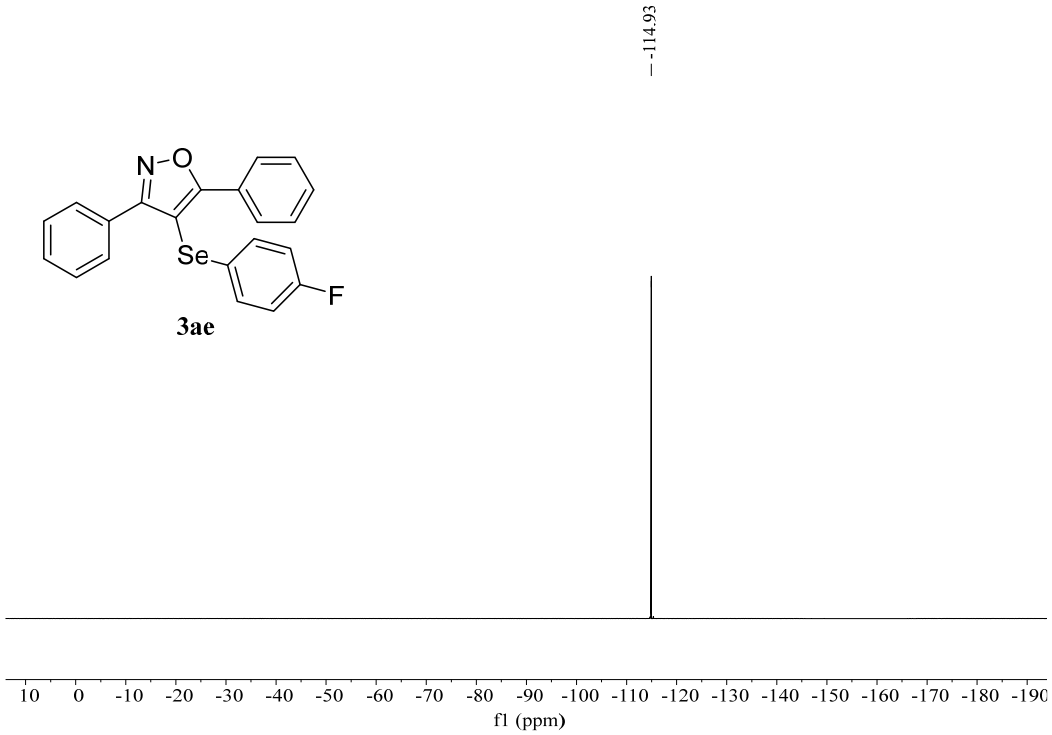


Figure S24 ^{19}F NMR (470 MHz) spectrum of **3ae** in $CDCl_3$

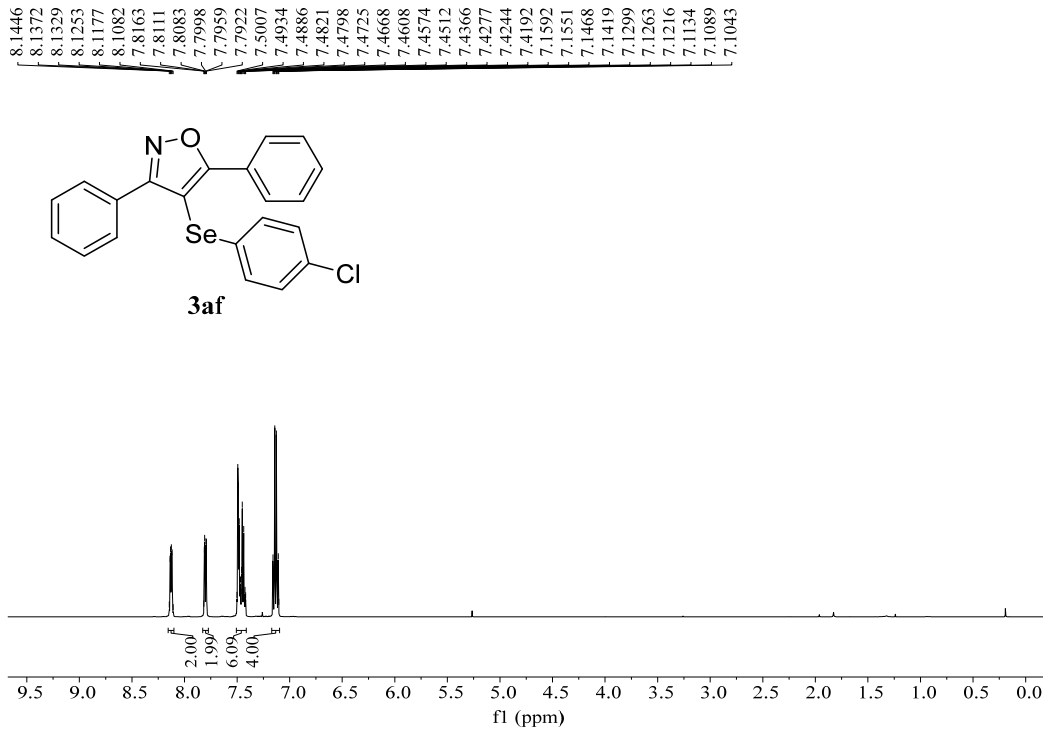


Figure S25 ^{1}H NMR (500 MHz) spectrum of **3af** in $CDCl_3$

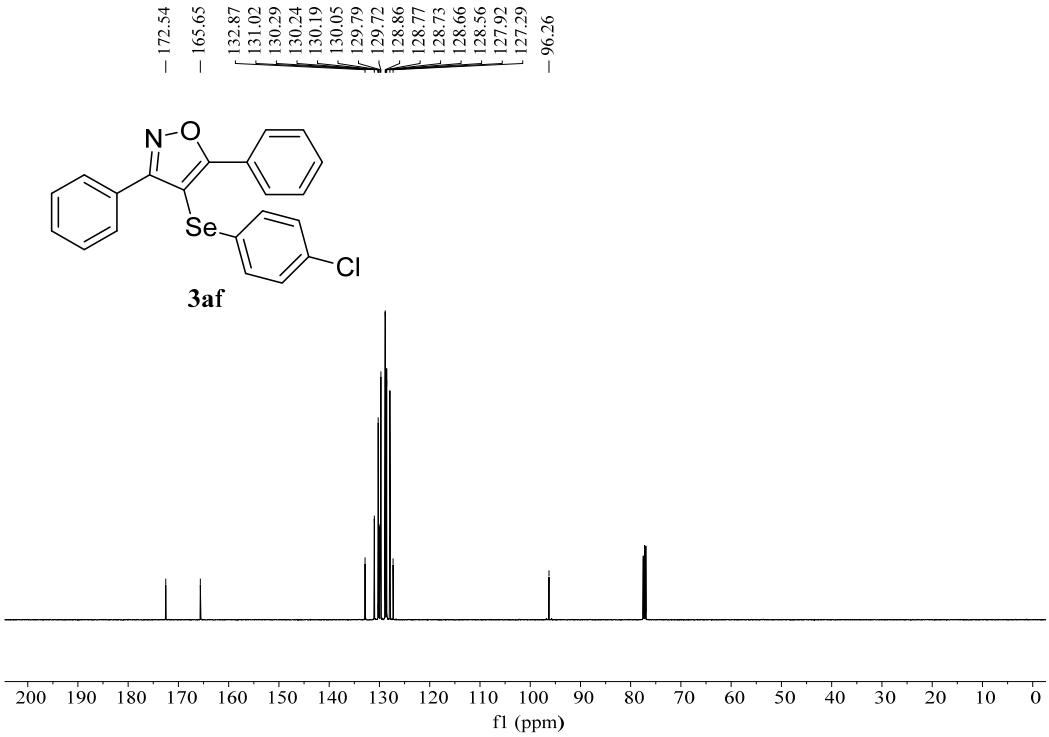


Figure S26 ^{13}C NMR (125 MHz) spectrum of **3af** in $CDCl_3$

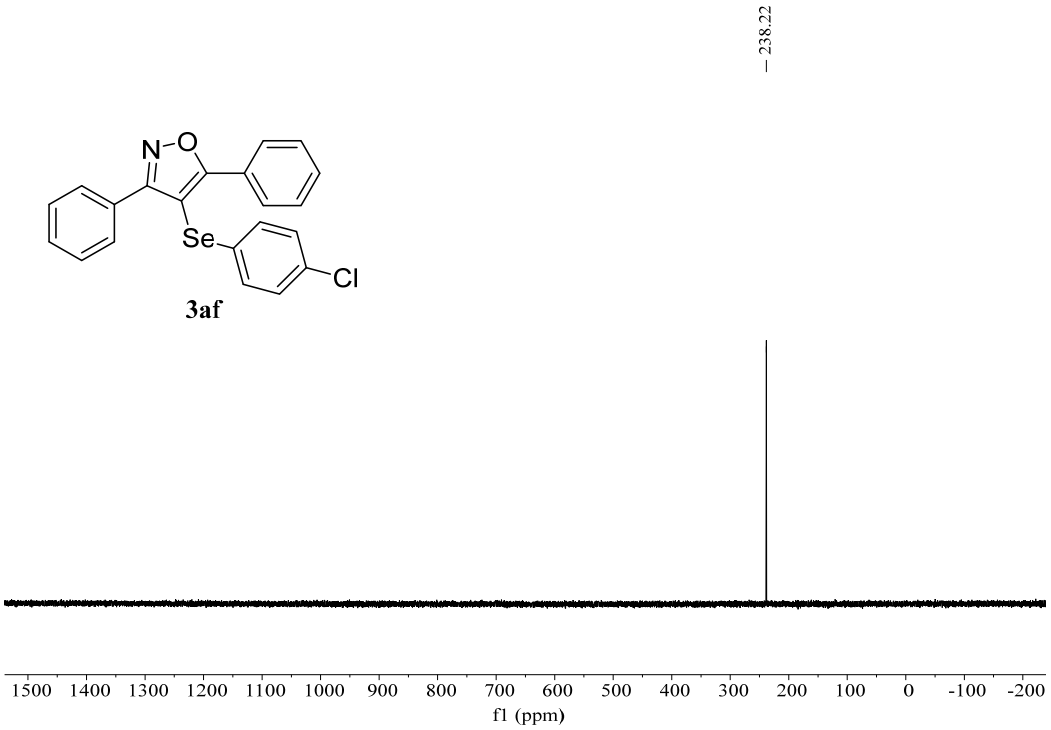


Figure S27 ^{77}Se NMR (95.5 MHz) spectrum of **3af** in $CDCl_3$

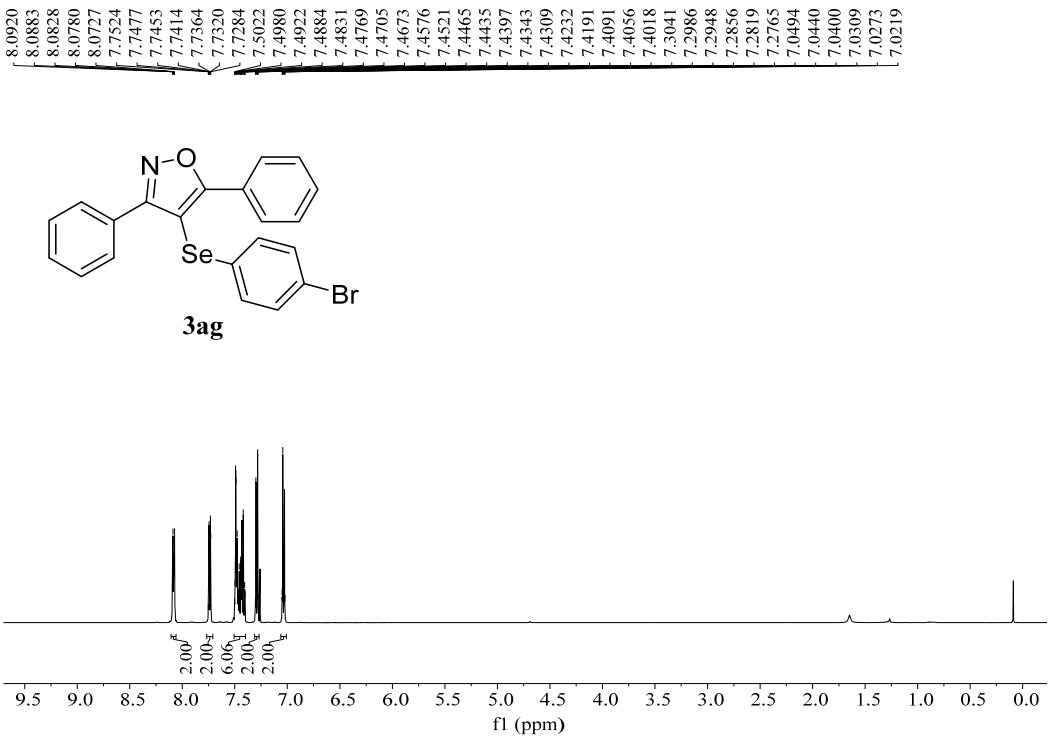


Figure S28 ^{1}H NMR (500 MHz) spectrum of **3ag** in $CDCl_3$

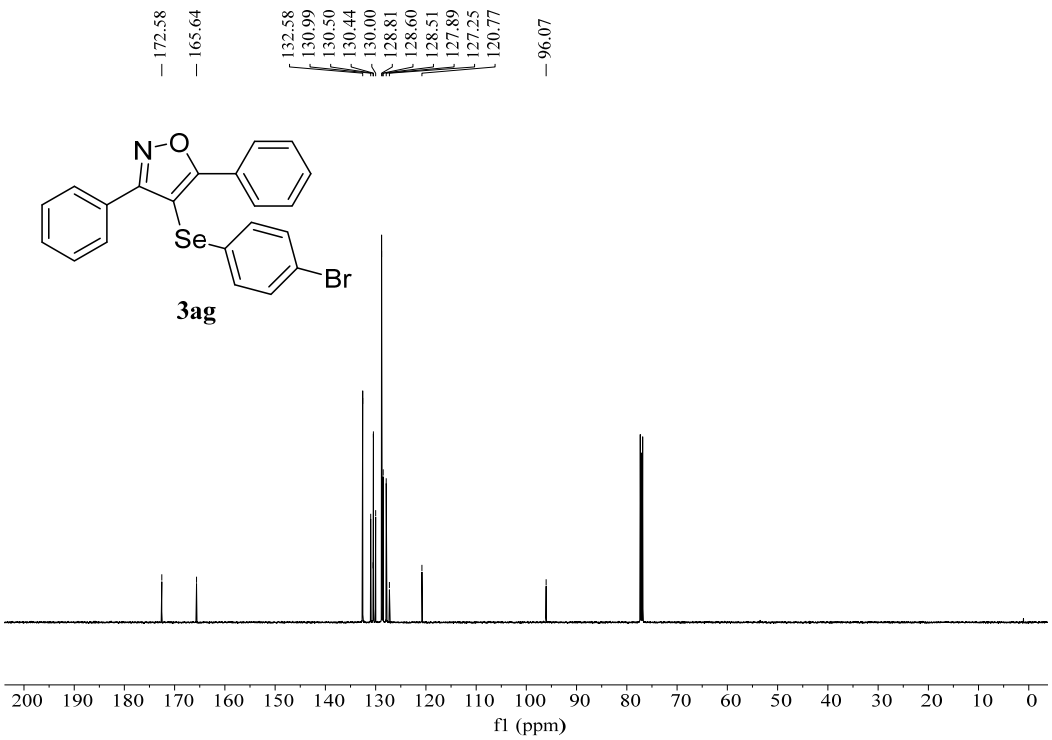


Figure S29 ^{13}C NMR (125 MHz) spectrum of **3ag** in $CDCl_3$

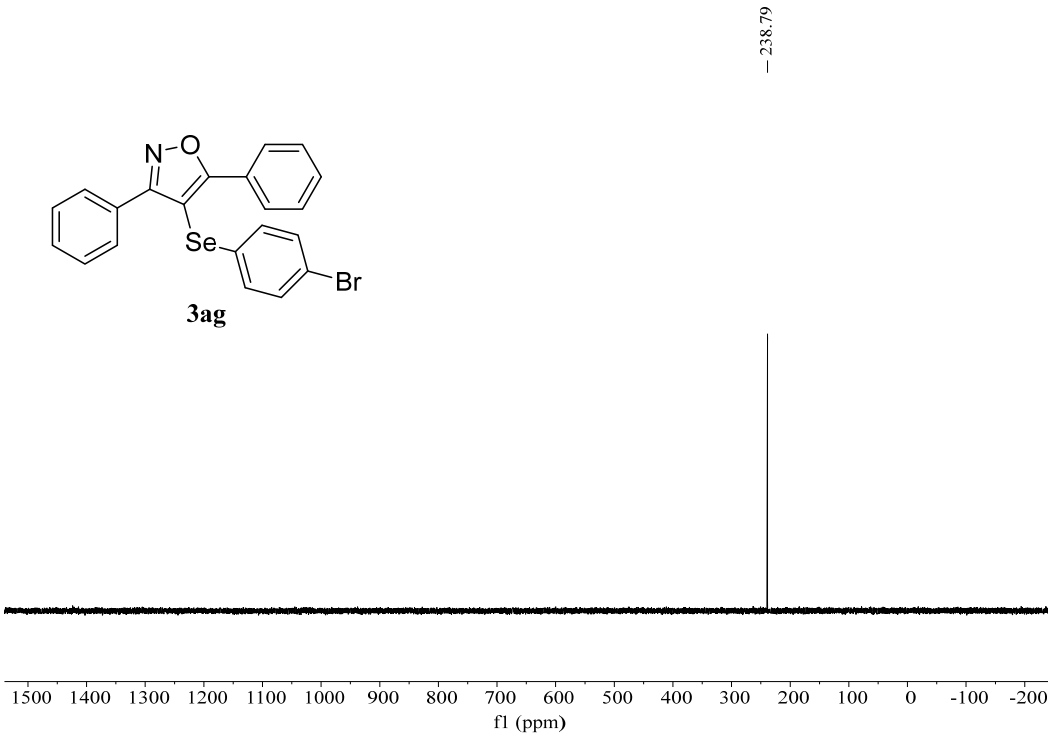


Figure S30 ^{77}Se NMR (95.5 MHz) spectrum of **3ag** in $CDCl_3$

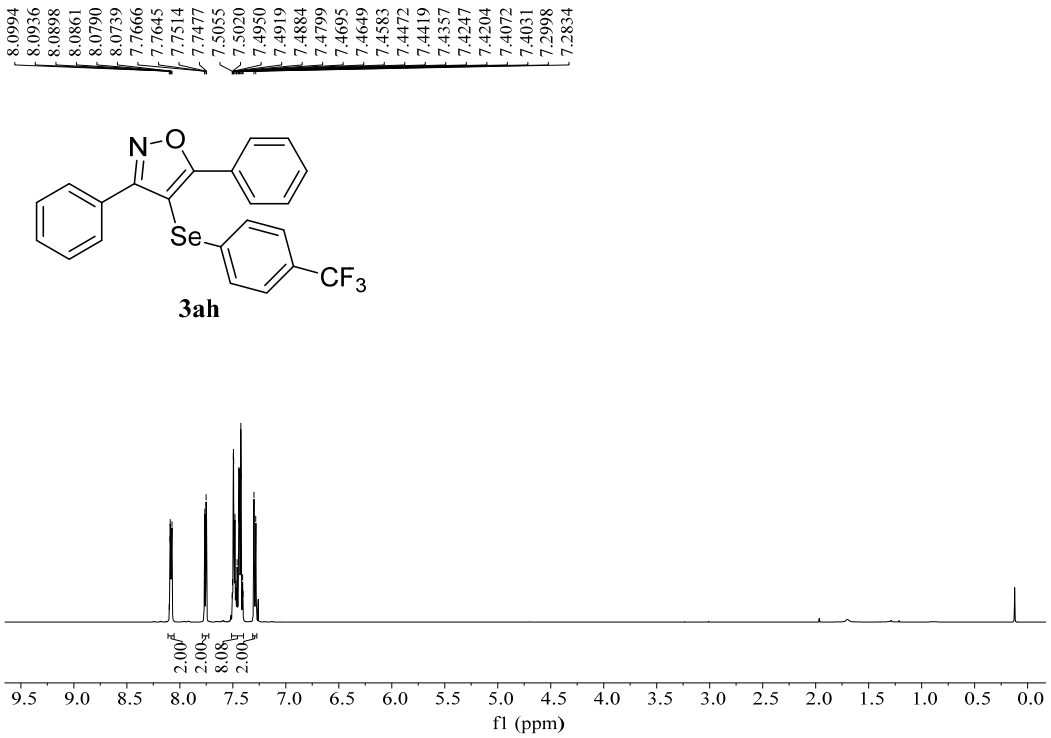


Figure S31 ^{1}H NMR (500 MHz) spectrum of **3ah** in $CDCl_3$

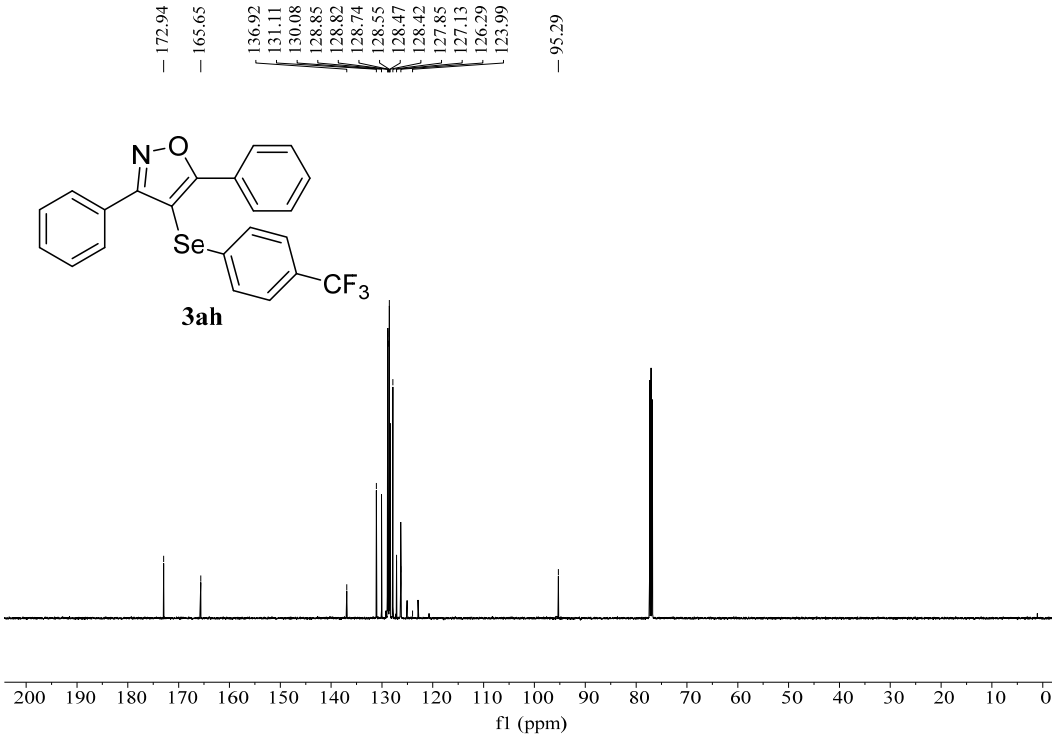


Figure S32 ^{13}C NMR (125 MHz) spectrum of **3ah** in $CDCl_3$

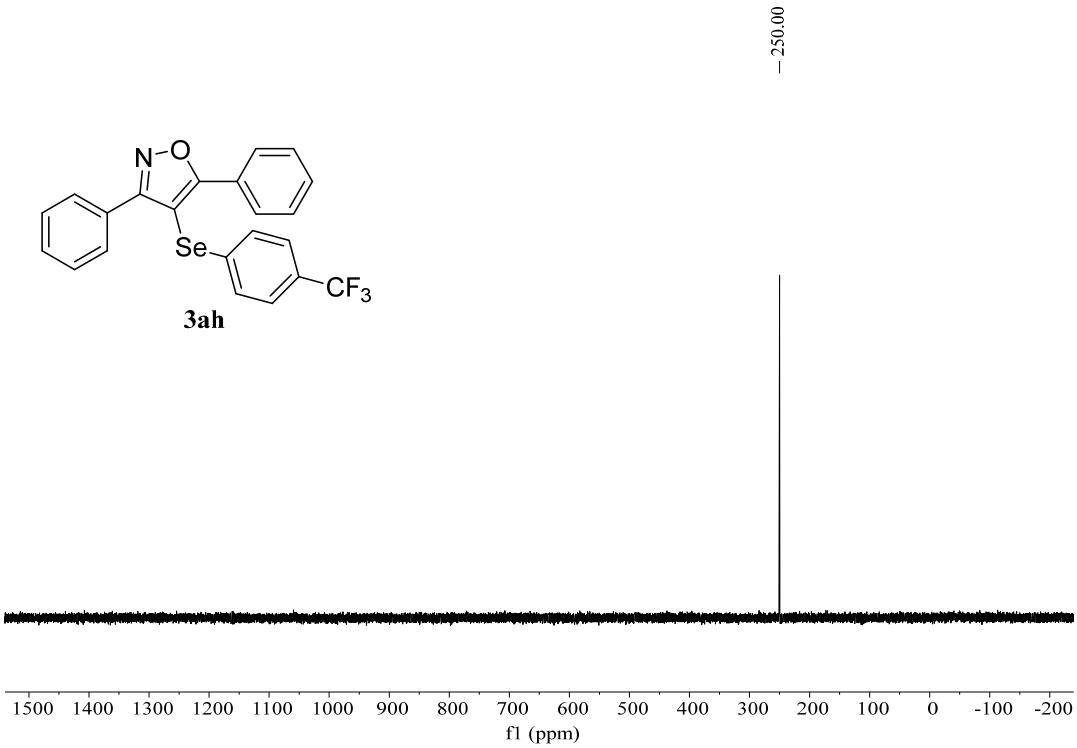


Figure S33 ^{77}Se NMR (95.5 MHz) spectrum of **3ah** in $CDCl_3$

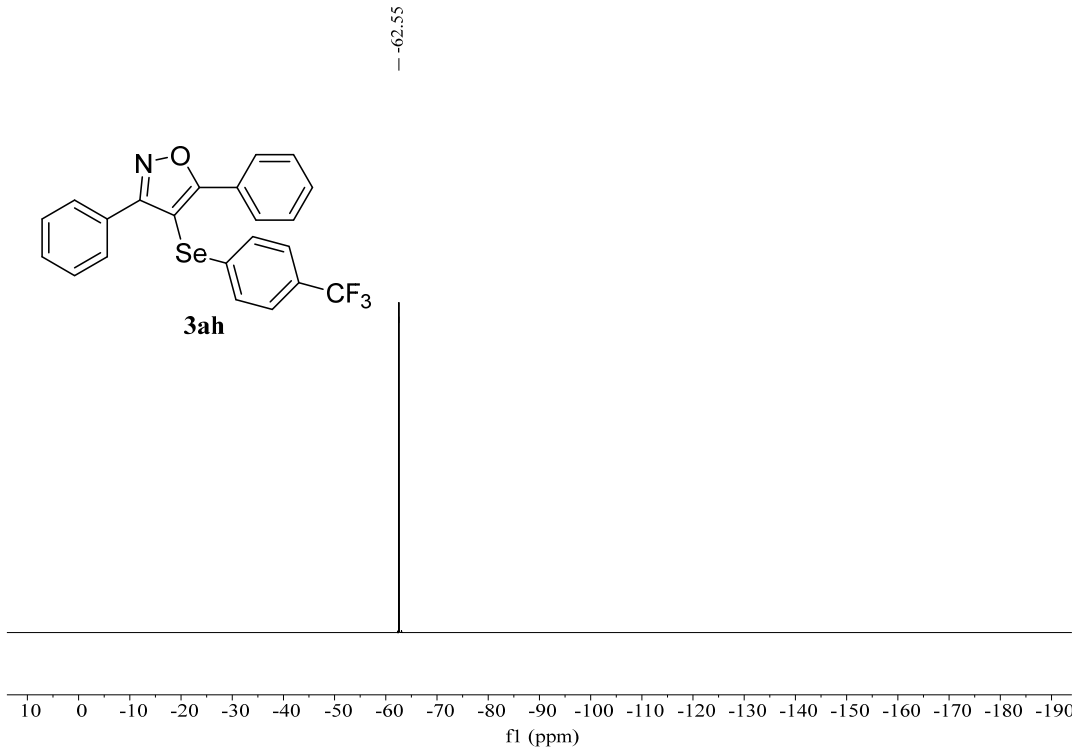


Figure S34 ^{19}F NMR (470 MHz) spectrum of **3ah** in $CDCl_3$

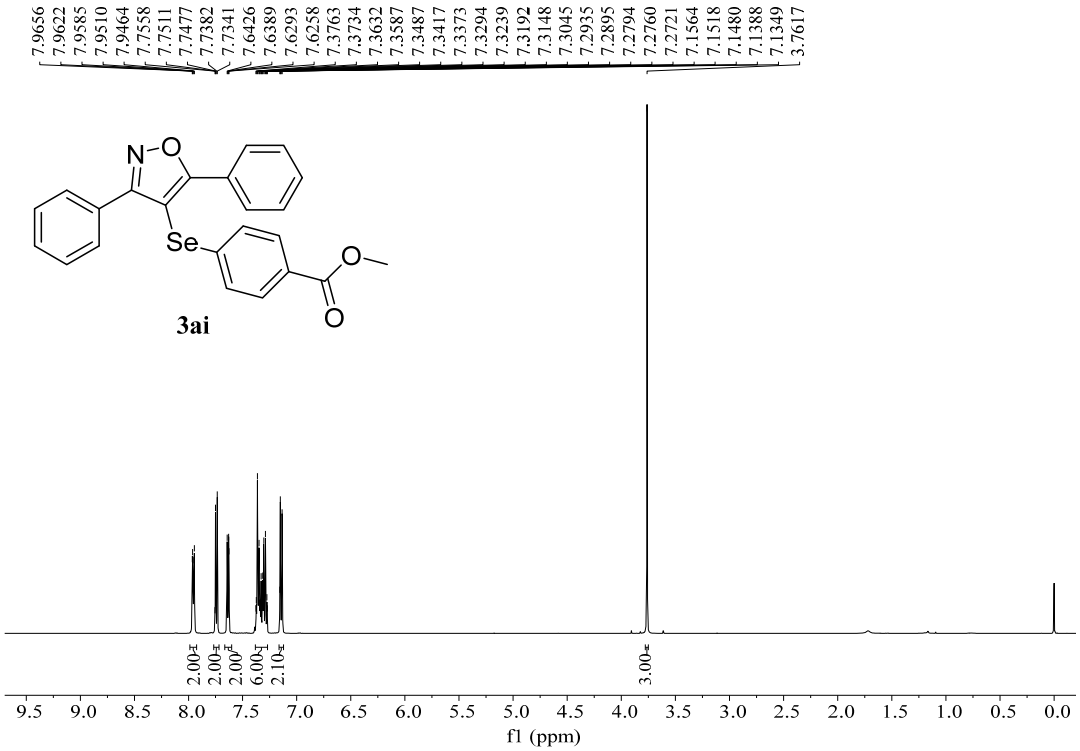


Figure S35 ^{1}H NMR (500 MHz) spectrum of **3ai** in $CDCl_3$

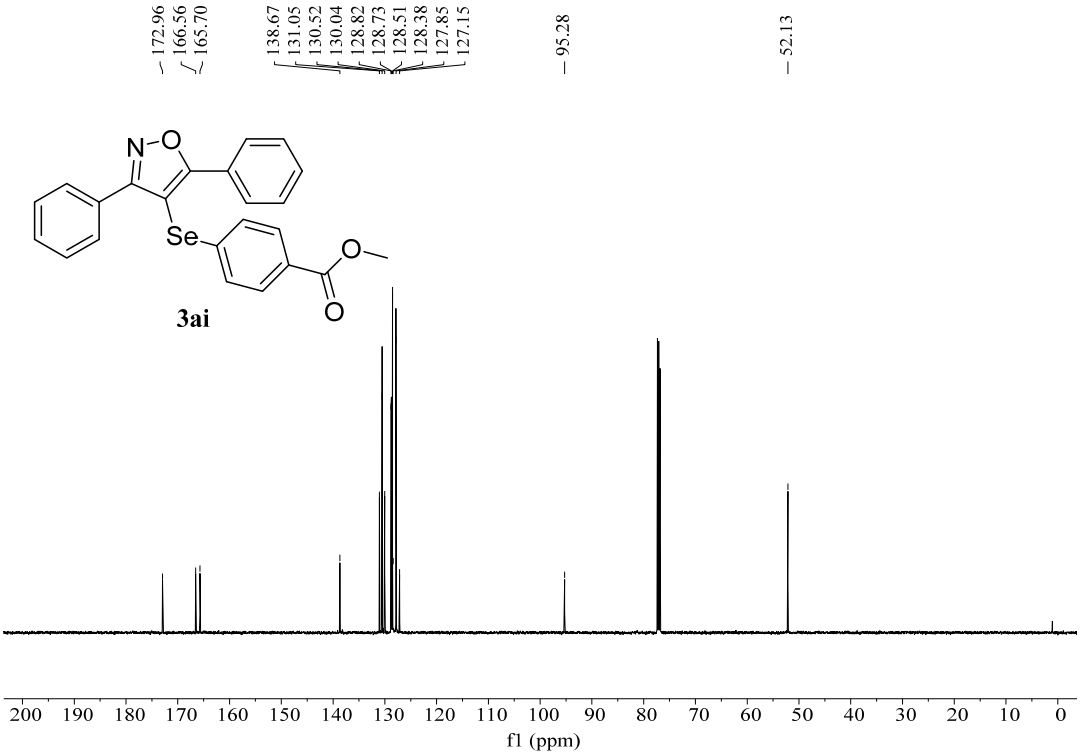


Figure S36 ^{13}C NMR (125 MHz) spectrum of **3ai** in $CDCl_3$

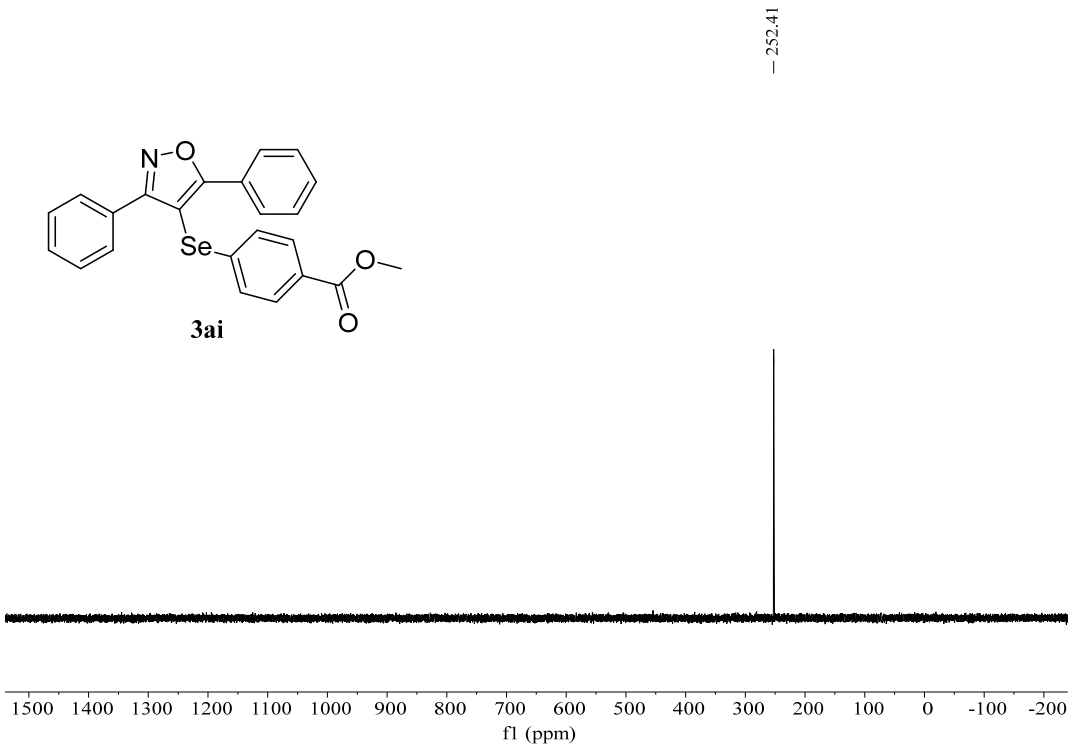


Figure S37 ^{77}Se NMR (95.5 MHz) spectrum of **3ai** in $CDCl_3$

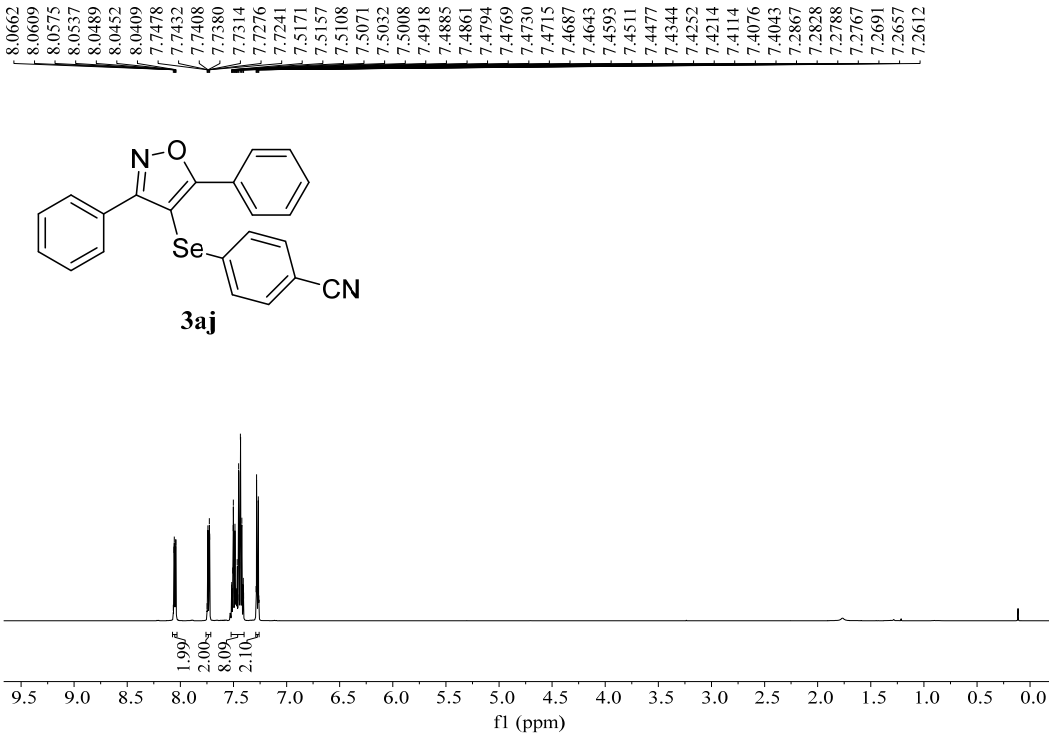


Figure S38 ^{1}H NMR (500 MHz) spectrum of **3aj** in $CDCl_3$

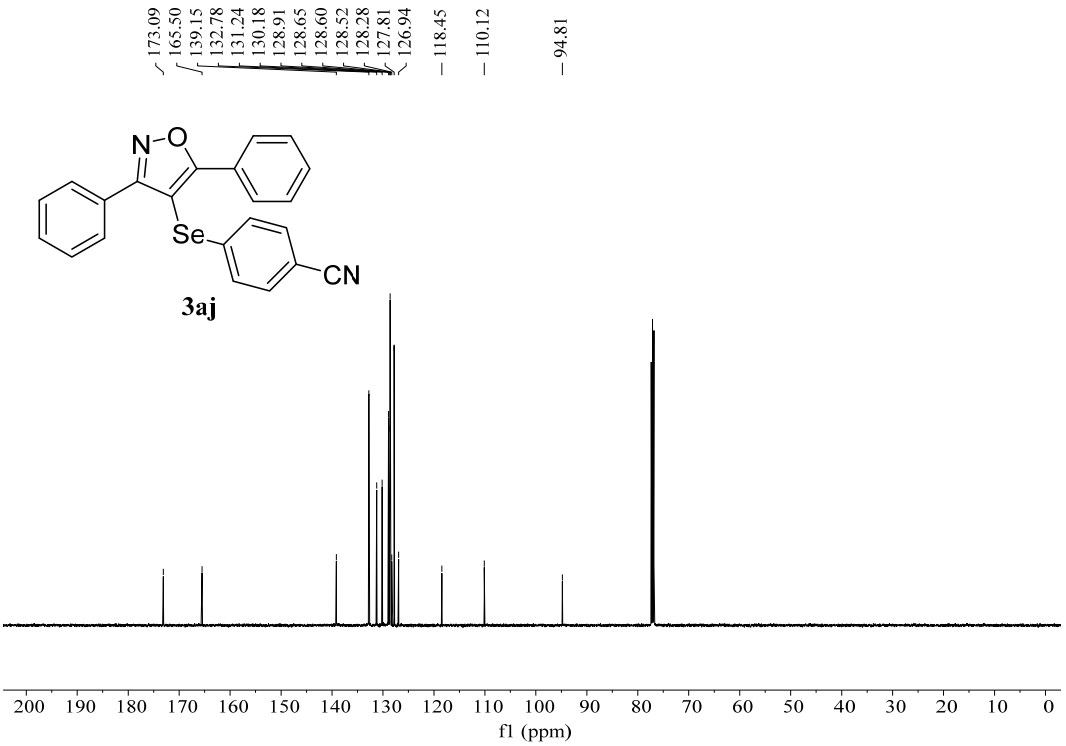


Figure S39 ^{13}C NMR (125 MHz) spectrum of **3aj** in $CDCl_3$

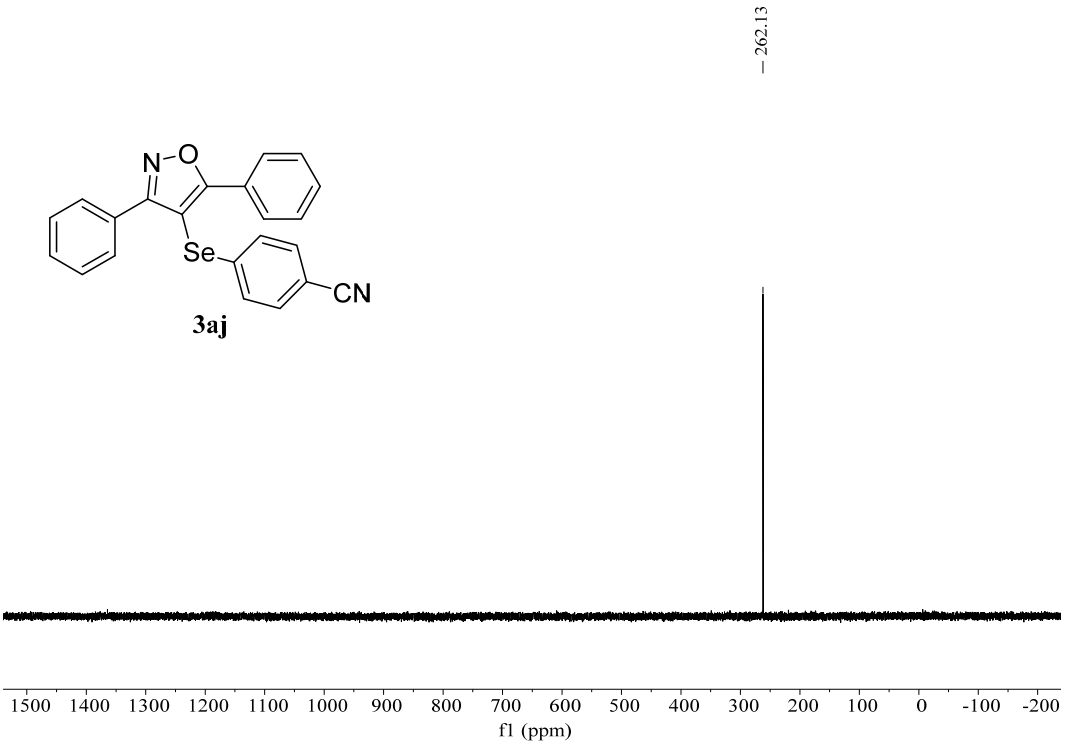


Figure S40 ^{77}Se NMR (95.5 MHz) spectrum of **3aj** in $CDCl_3$

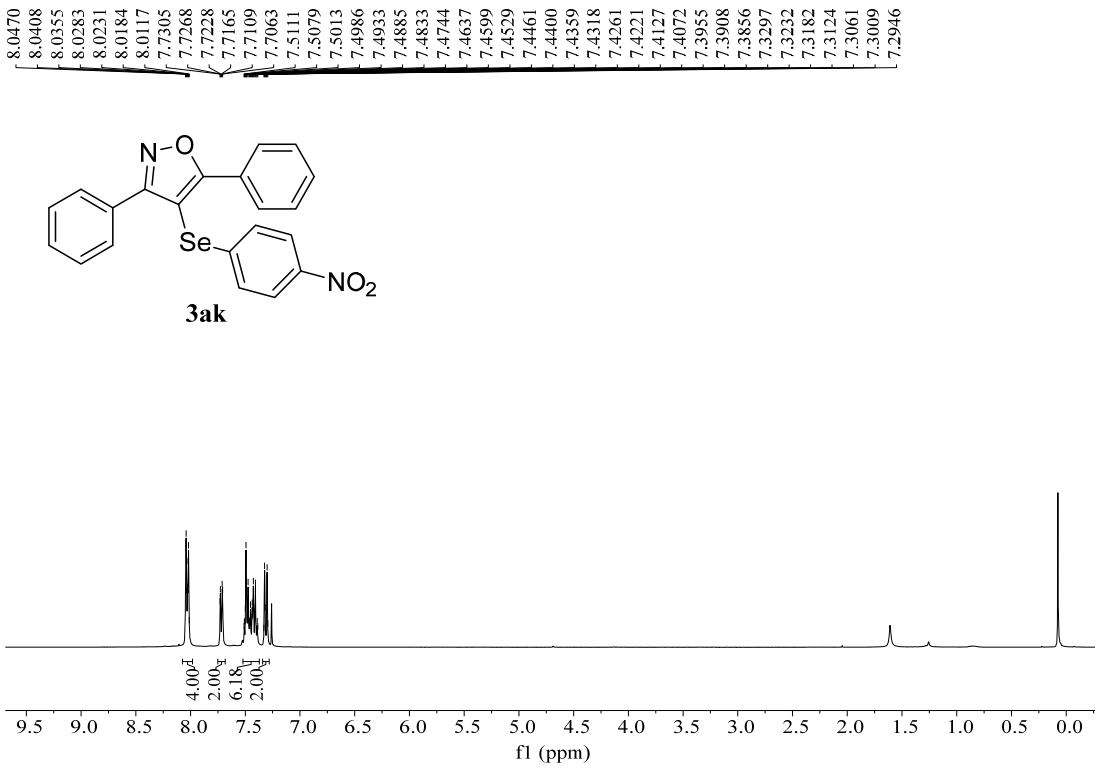


Figure S41 ^{1}H NMR (500 MHz) spectrum of **3ak** in $CDCl_3$

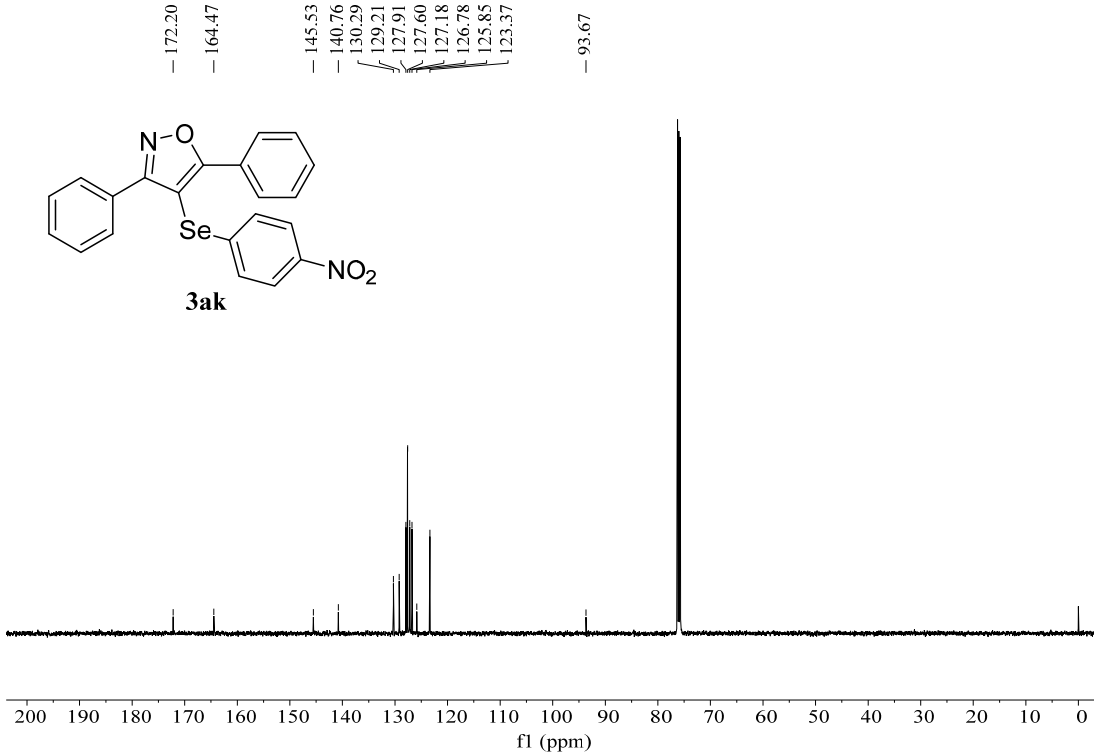


Figure S42 ^{13}C NMR (125 MHz) spectrum of **3ak** in $CDCl_3$

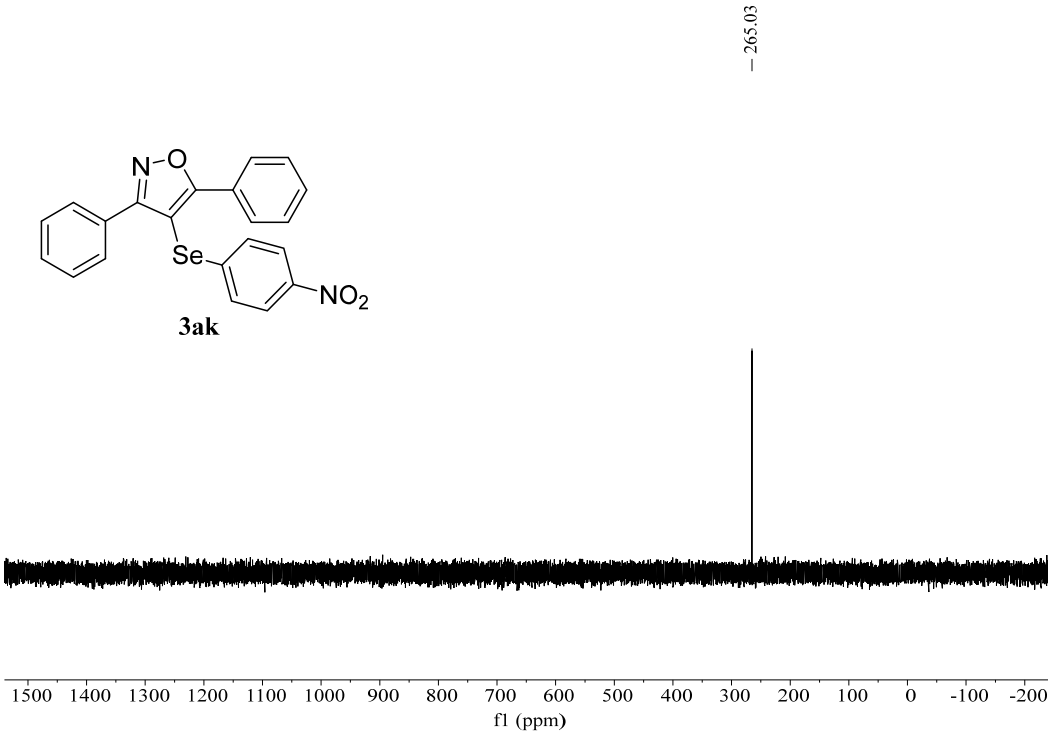


Figure S43 ^{77}Se NMR (95.5 MHz) spectrum of **3ak** in $CDCl_3$

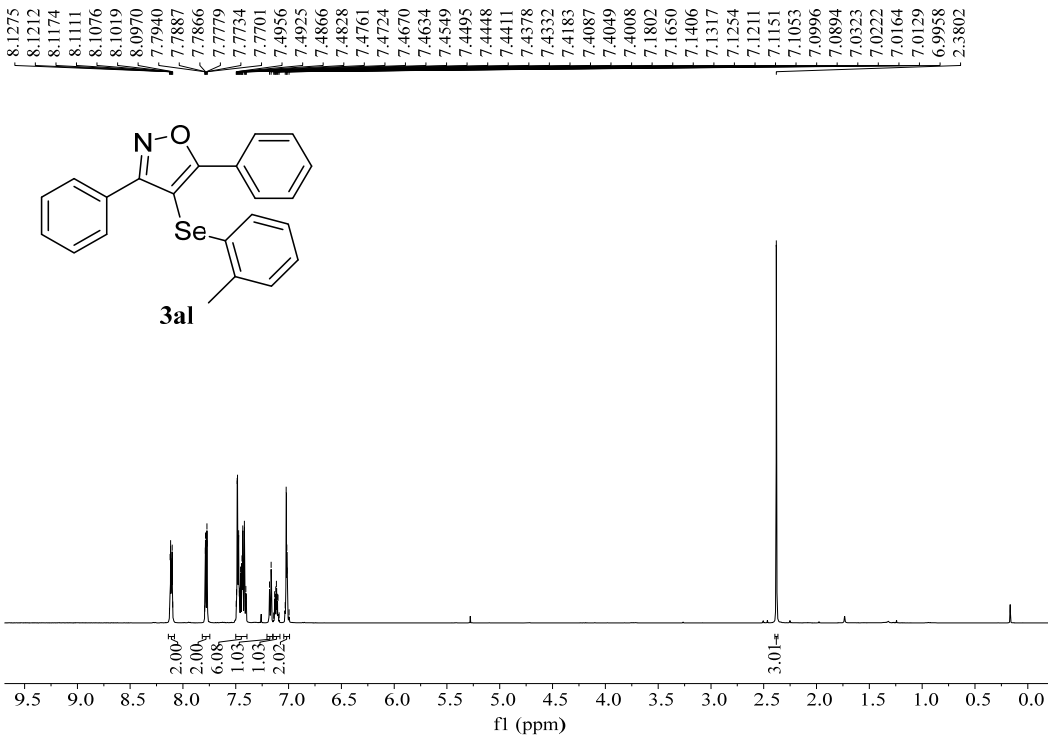


Figure S44 ^{1}H NMR (500 MHz) spectrum of **3al** in $CDCl_3$

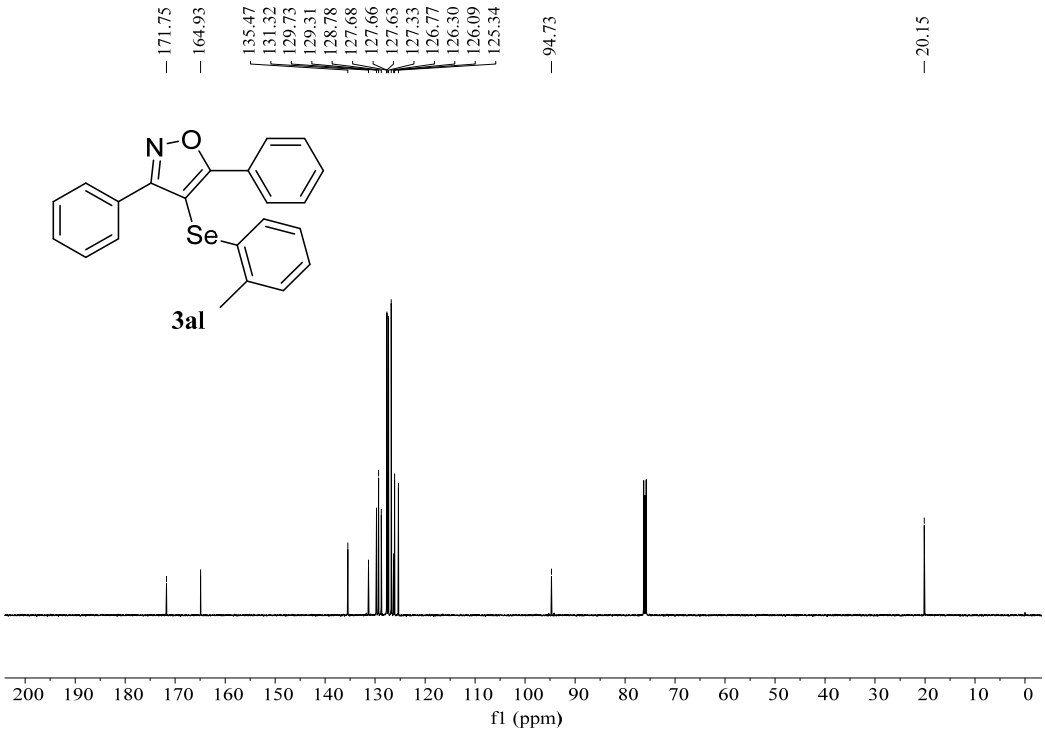


Figure S45 ^{13}C NMR (125 MHz) spectrum of **3al** in $CDCl_3$

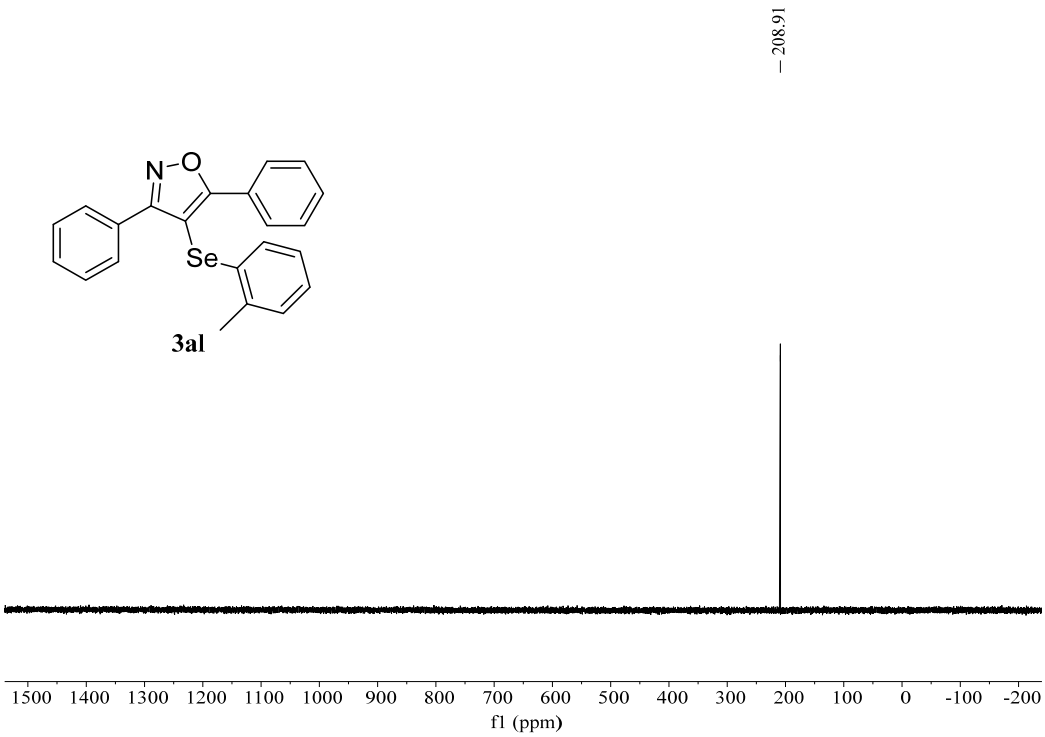


Figure S46 ^{77}Se NMR (95.5 MHz) spectrum of **3al** in $CDCl_3$

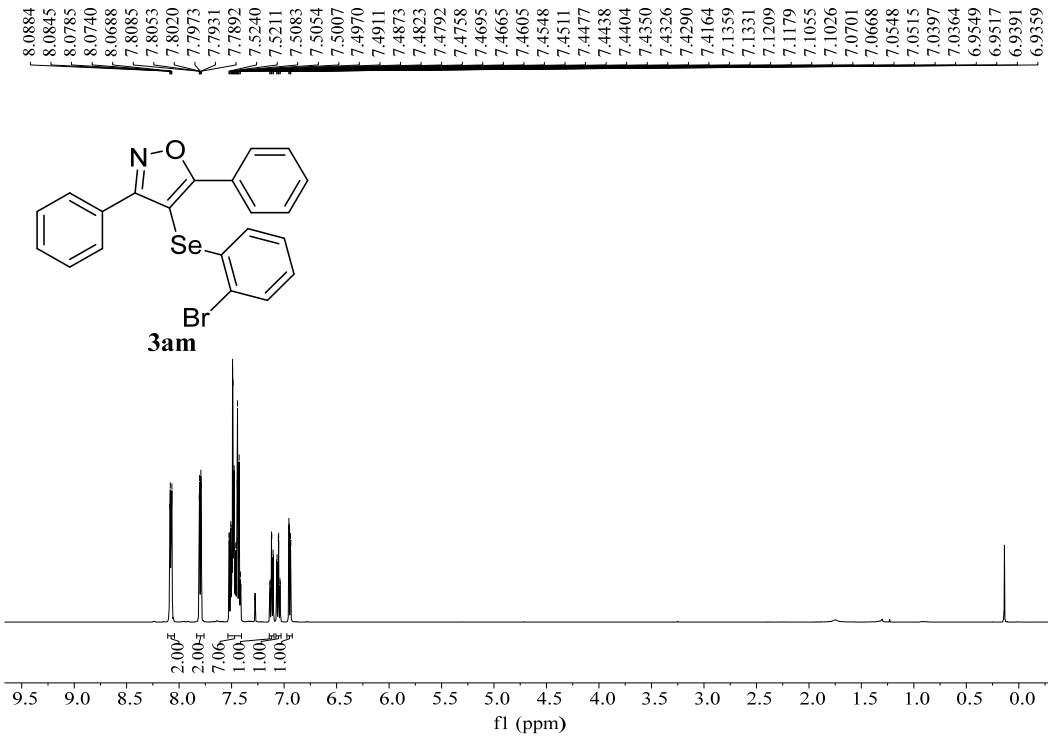


Figure S47 ^{1}H NMR (500 MHz) spectrum of **3am** in $CDCl_3$

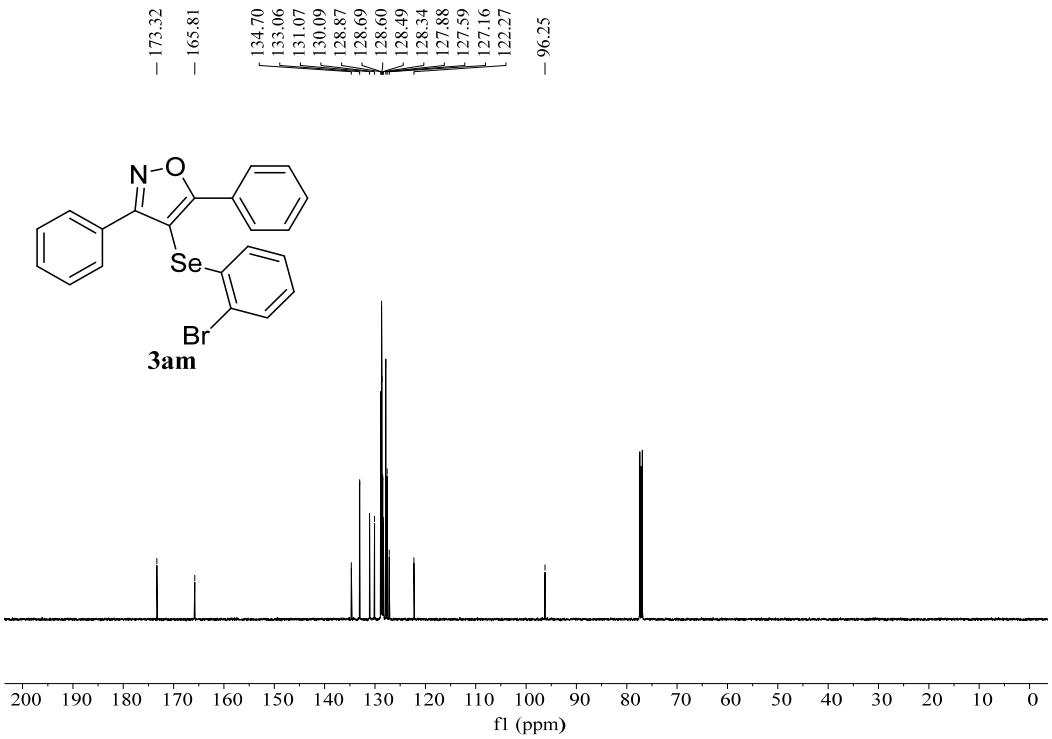


Figure S48 ^{13}C NMR (125 MHz) spectrum of **3am** in $CDCl_3$

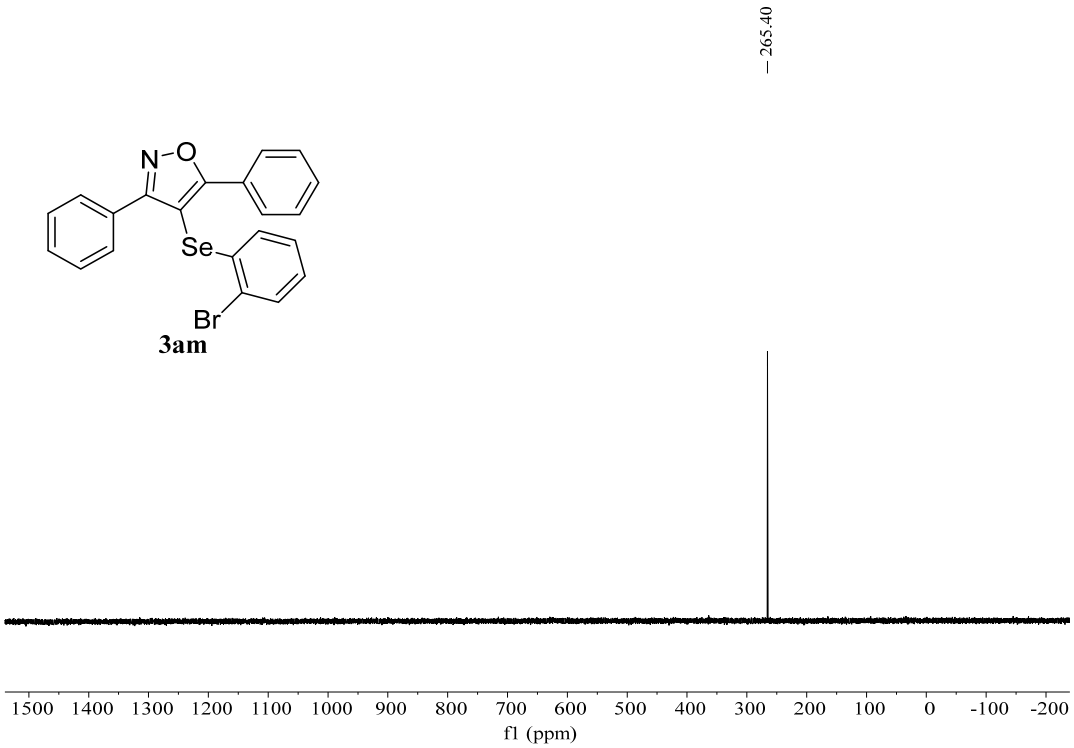


Figure S49 ^{77}Se NMR (95.5 MHz) spectrum of **3am** in $CDCl_3$

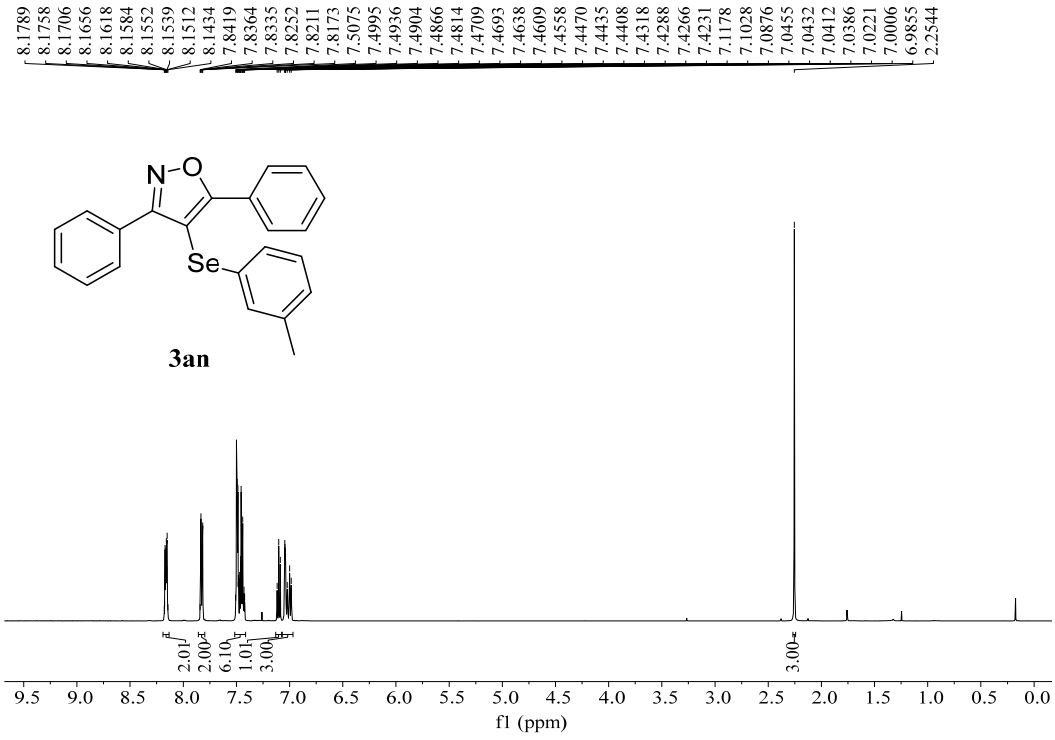


Figure S50 ^{1}H NMR (500 MHz) spectrum of **3an** in $CDCl_3$

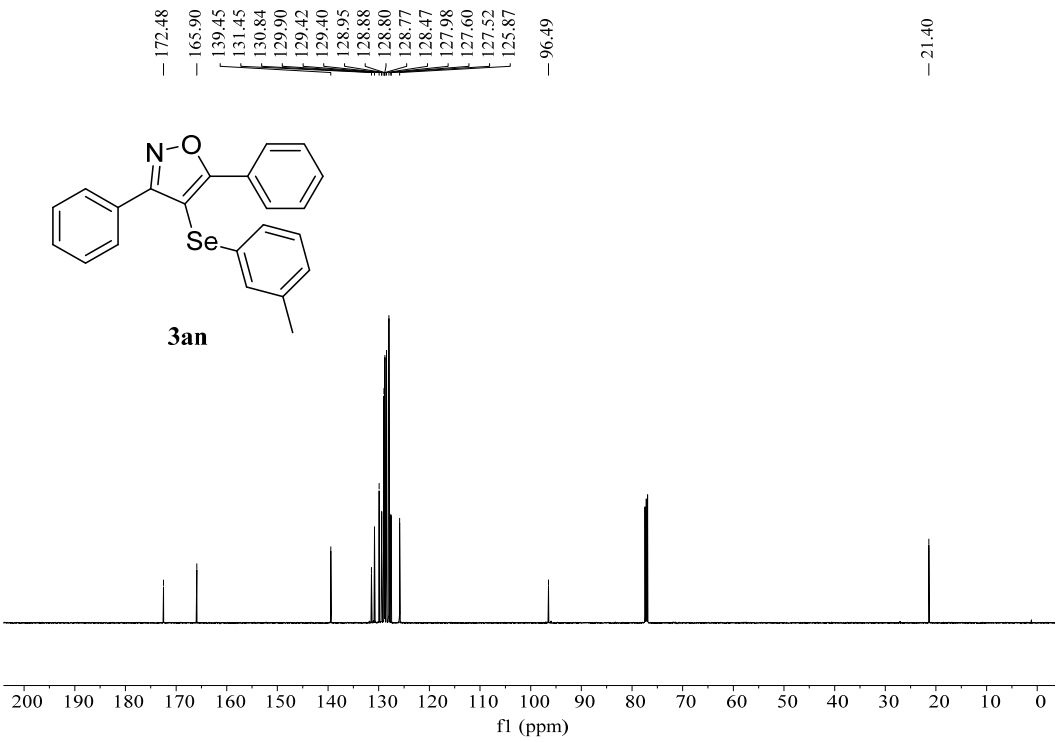


Figure S51 ^{13}C NMR (125 MHz) spectrum of **3an** in $CDCl_3$

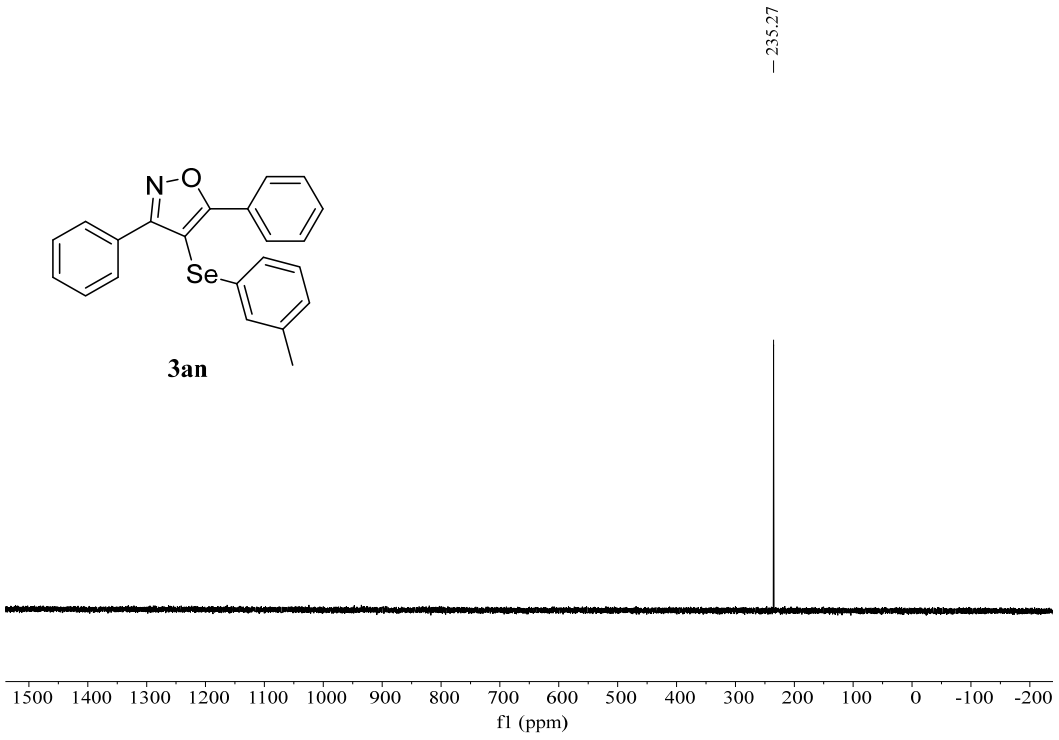


Figure S52 ^{77}Se NMR (95.5 MHz) spectrum of **3an** in $CDCl_3$

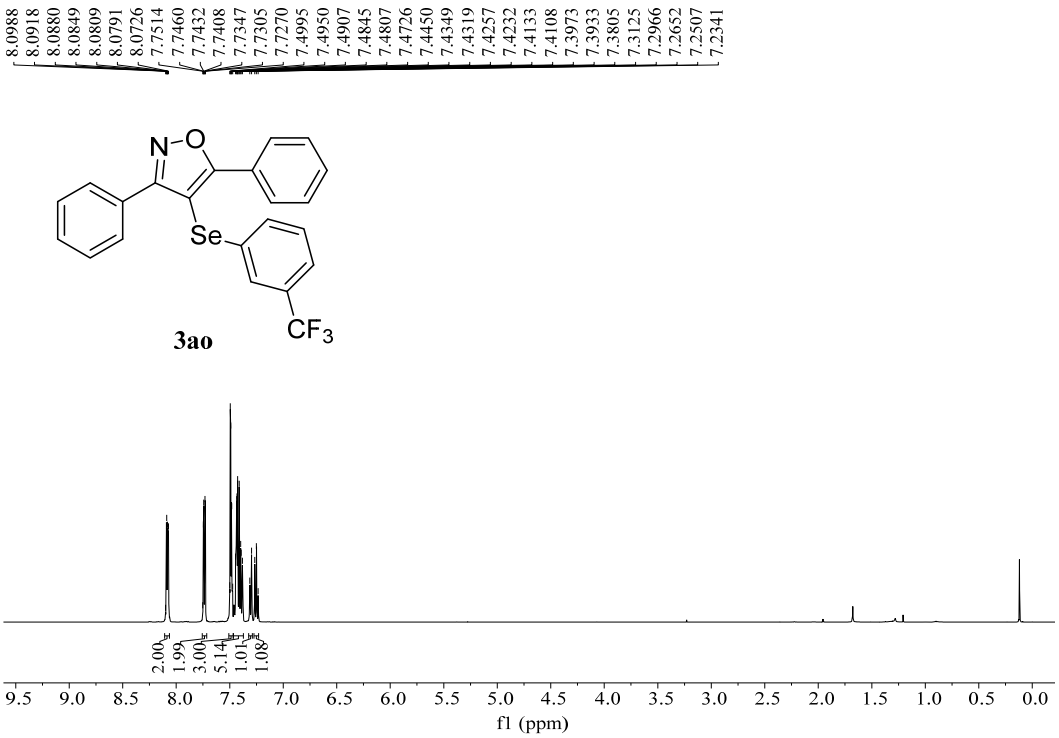


Figure S53 ^{1}H NMR (500 MHz) spectrum of **3ao** in $CDCl_3$

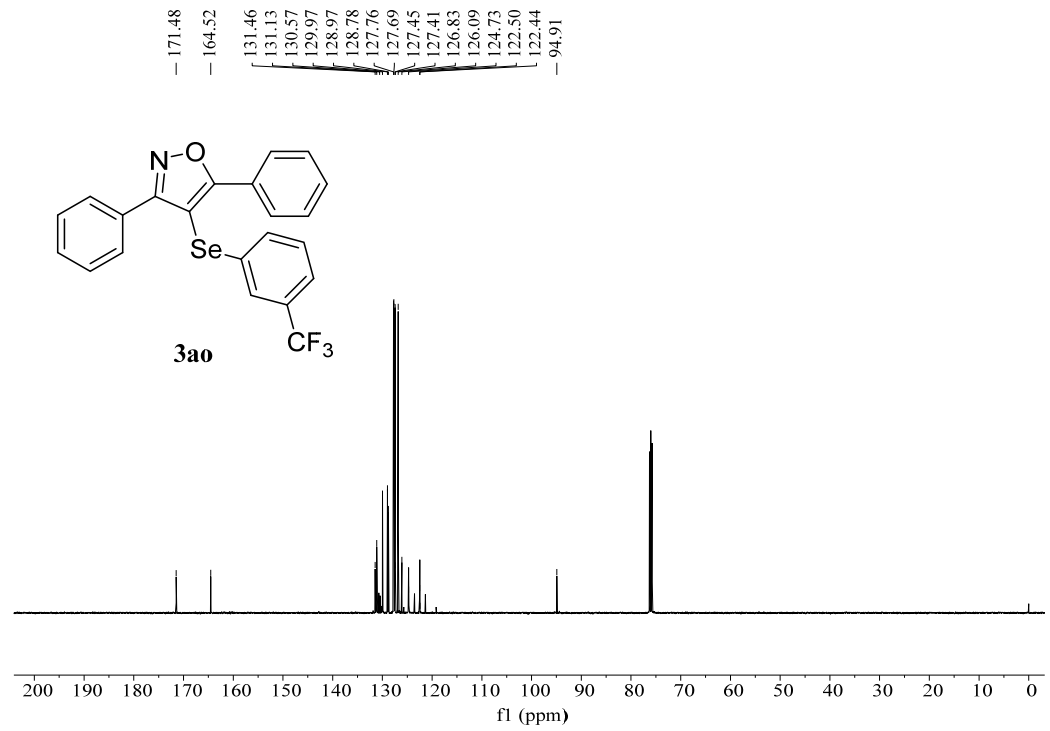


Figure S54 ^{13}C NMR (125 MHz) spectrum of **3ao** in $CDCl_3$

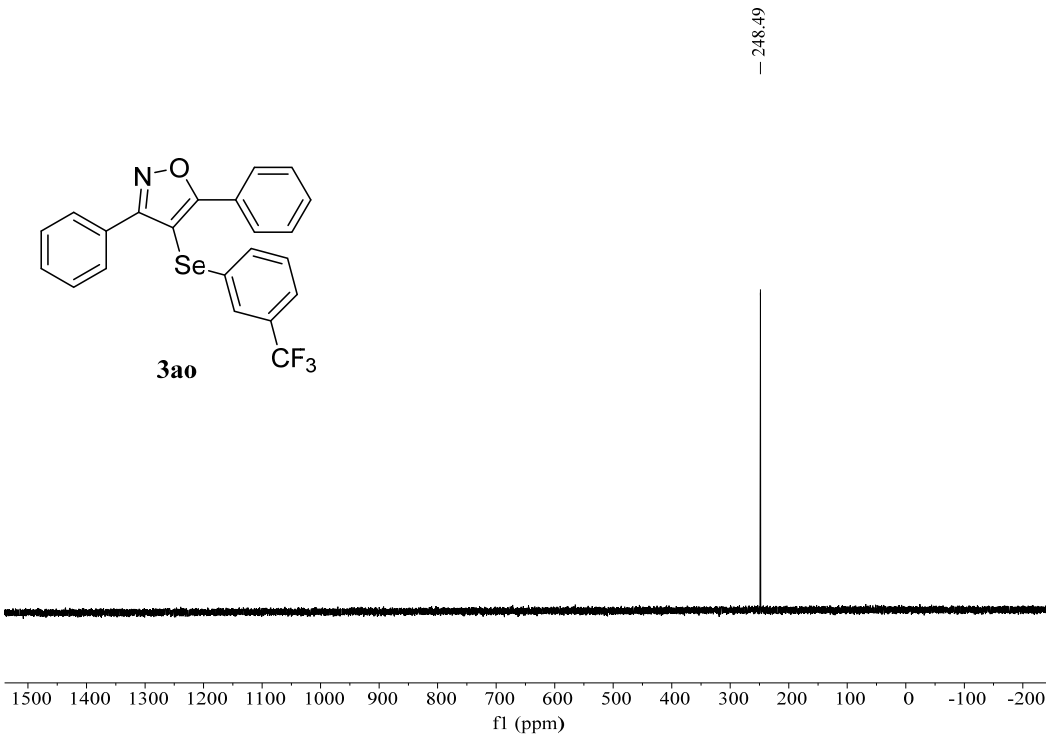


Figure S55 ^{77}Se NMR (95.5 MHz) spectrum of **3ao** in $CDCl_3$

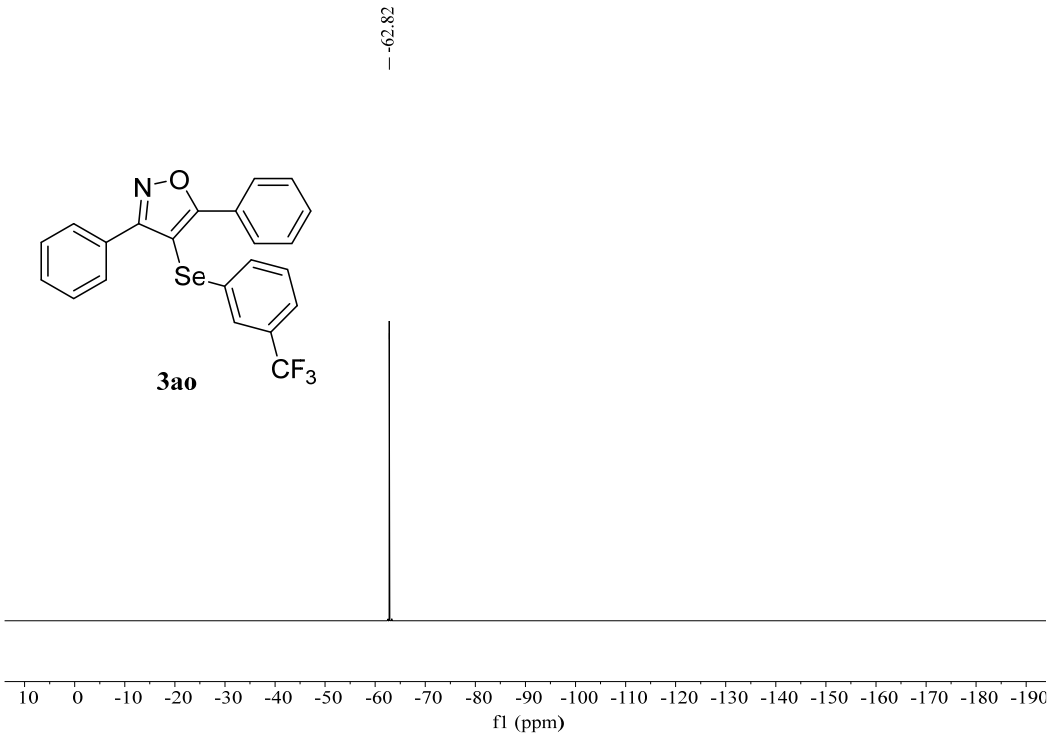


Figure S56 ^{19}F NMR (470 MHz) spectrum of **3ao** in $CDCl_3$

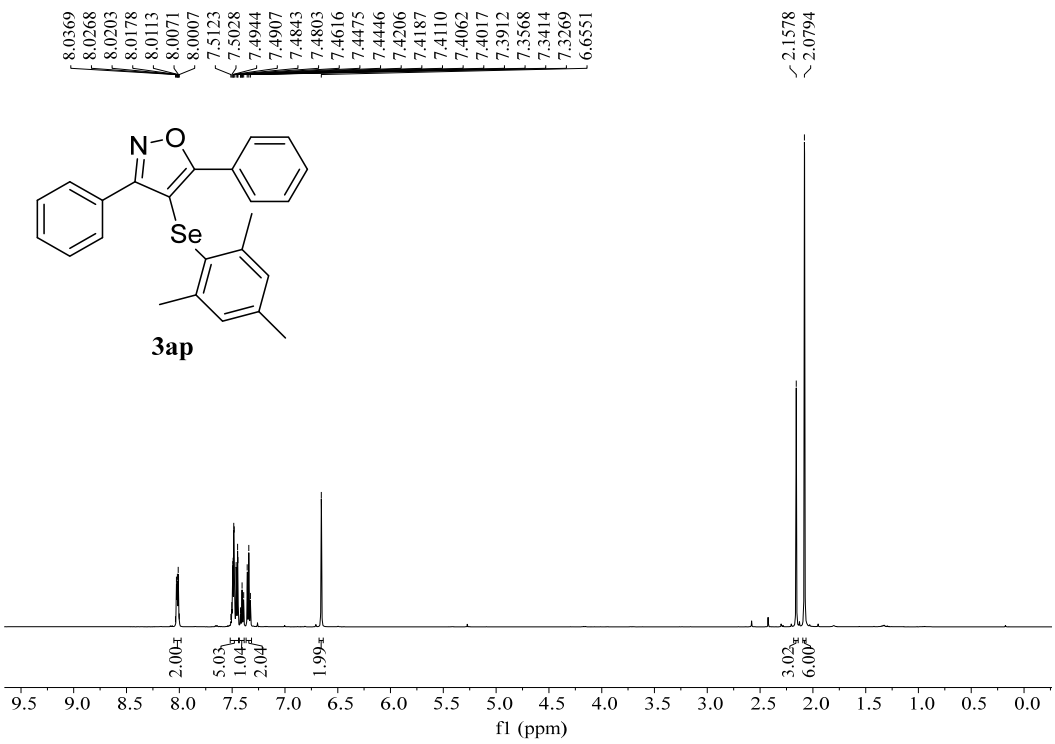


Figure S57 ^{1}H NMR (500 MHz) spectrum of **3ap** in $CDCl_3$

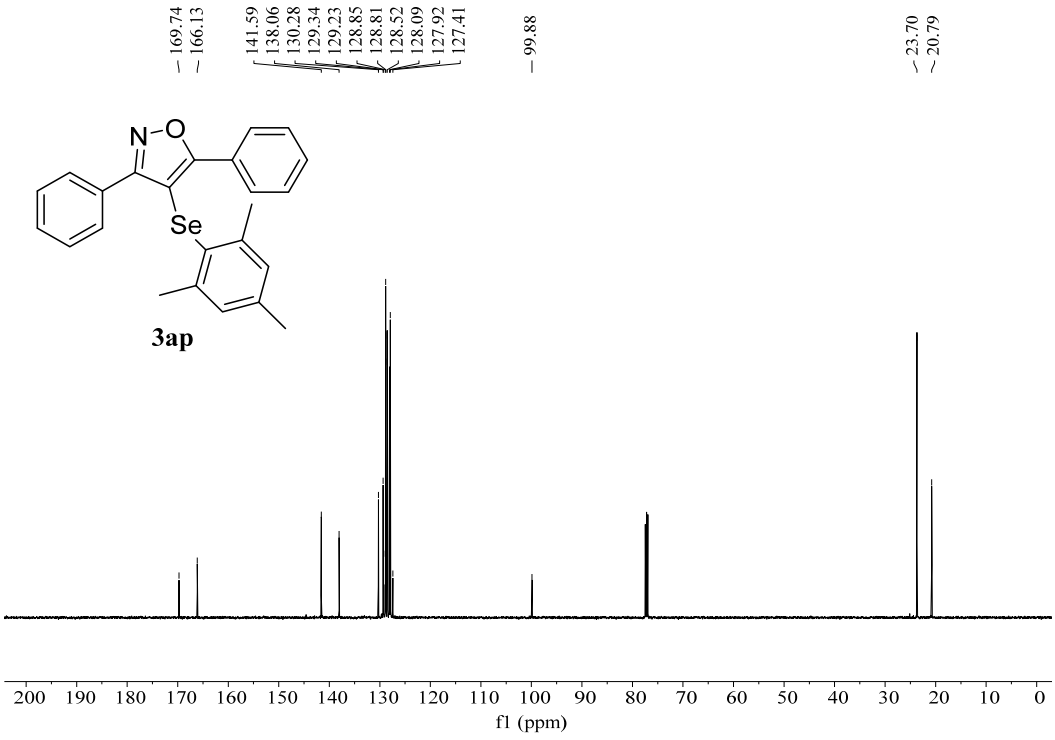


Figure S58 ^{13}C NMR (125 MHz) spectrum of **3ap** in $CDCl_3$

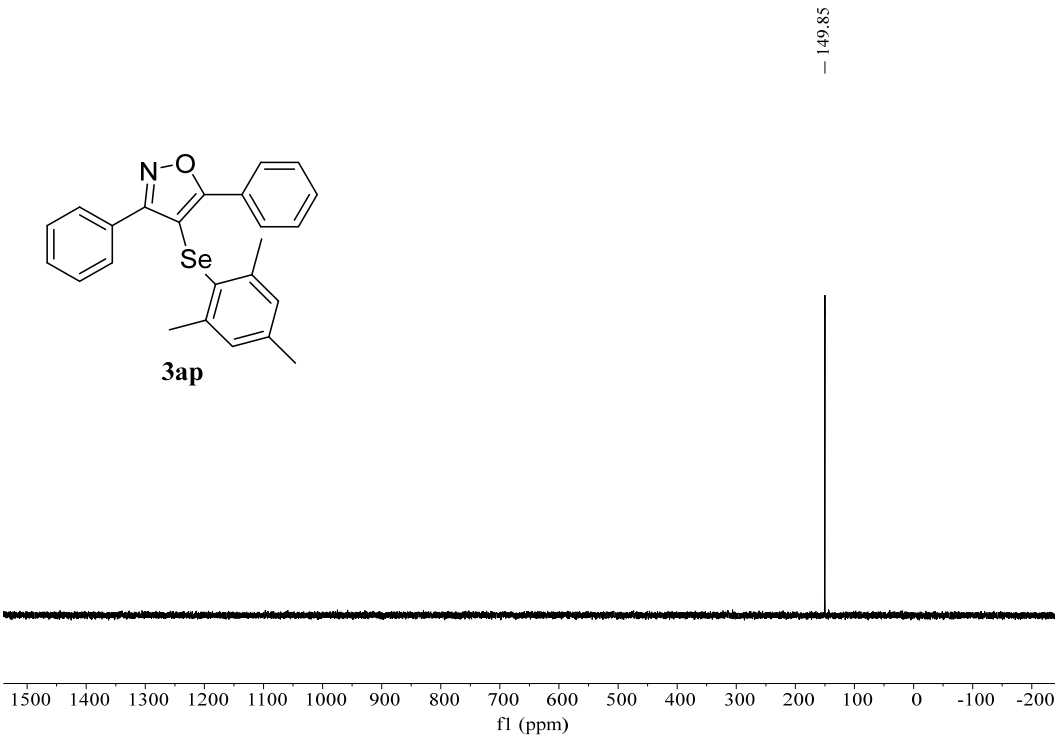


Figure S59 ^{77}Se NMR (95.5 MHz) spectrum of **3ap** in $CDCl_3$

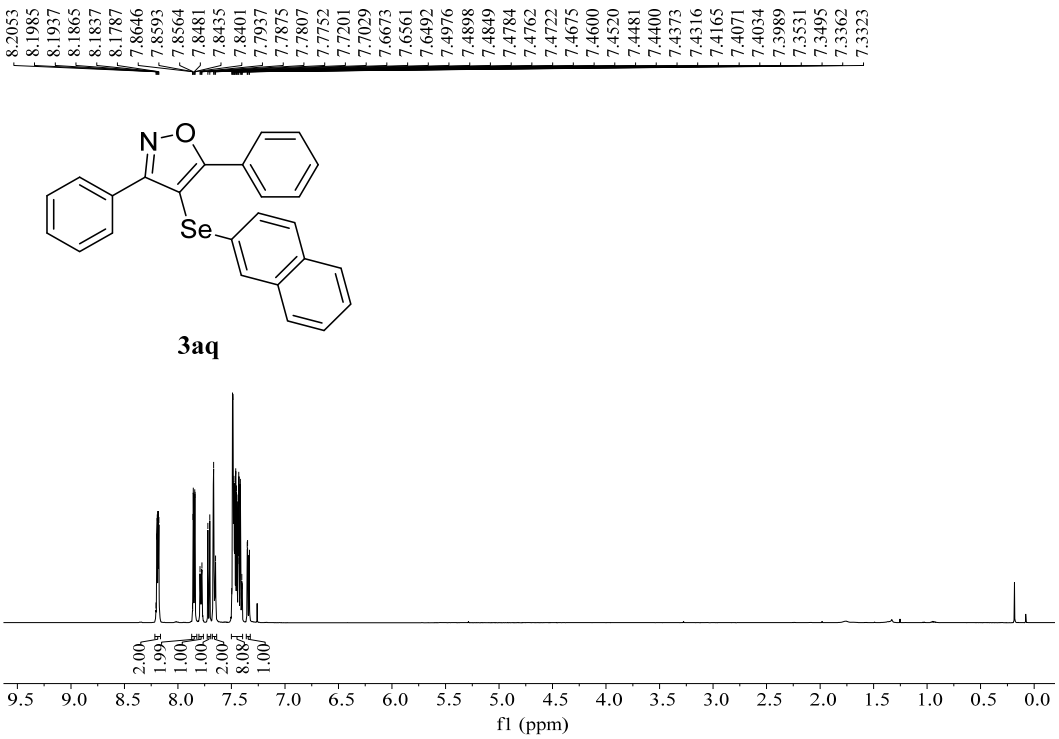


Figure S60 ^{1}H NMR (500 MHz) spectrum of **3aq** in $CDCl_3$

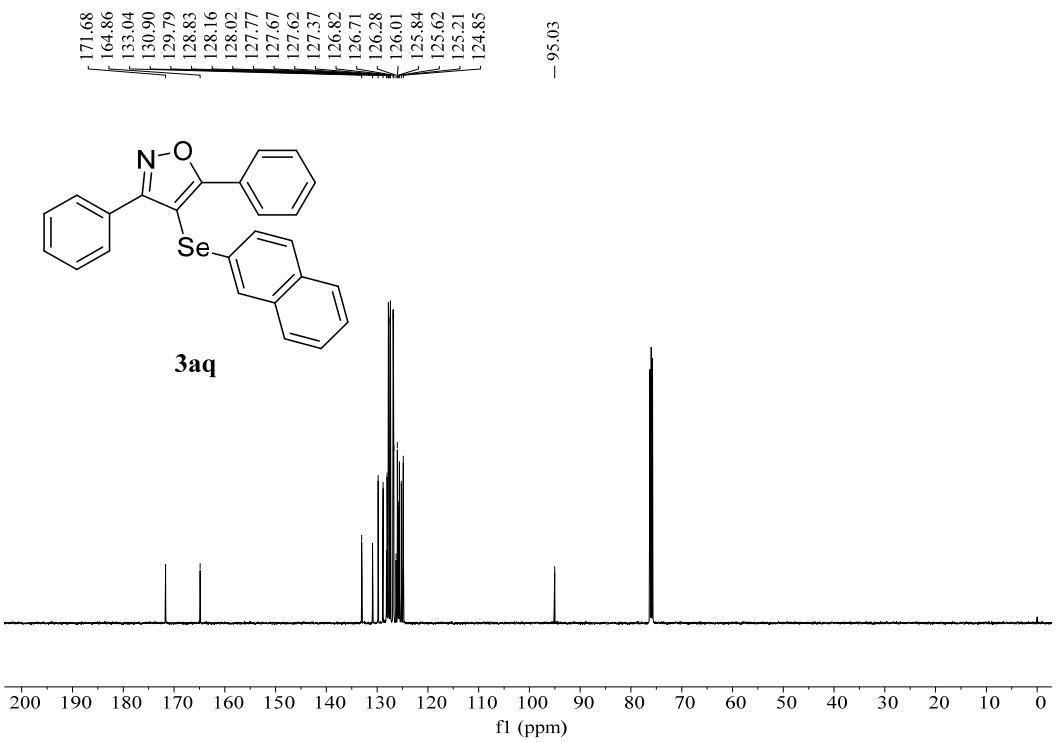


Figure S61 ^{13}C NMR (125 MHz) spectrum of **3aq** in $CDCl_3$

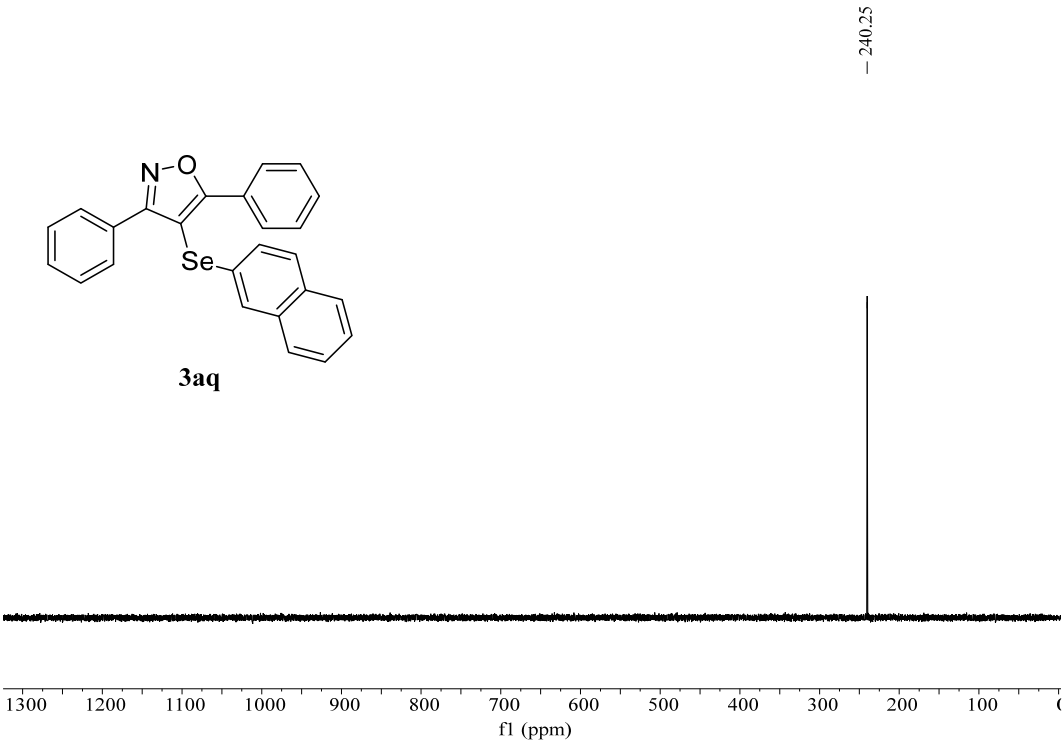


Figure S62 ^{77}Se NMR (95.5 MHz) spectrum of **3aq** in $CDCl_3$

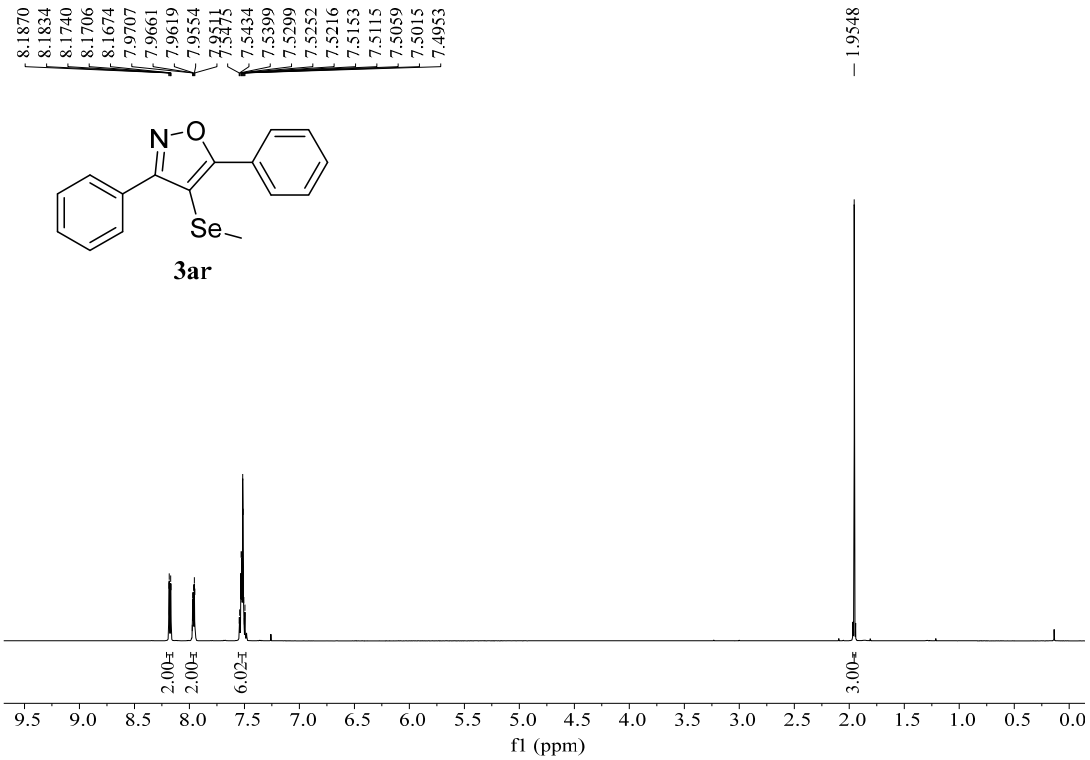


Figure S63 ^{1}H NMR (500 MHz) spectrum of **3ar** in $CDCl_3$

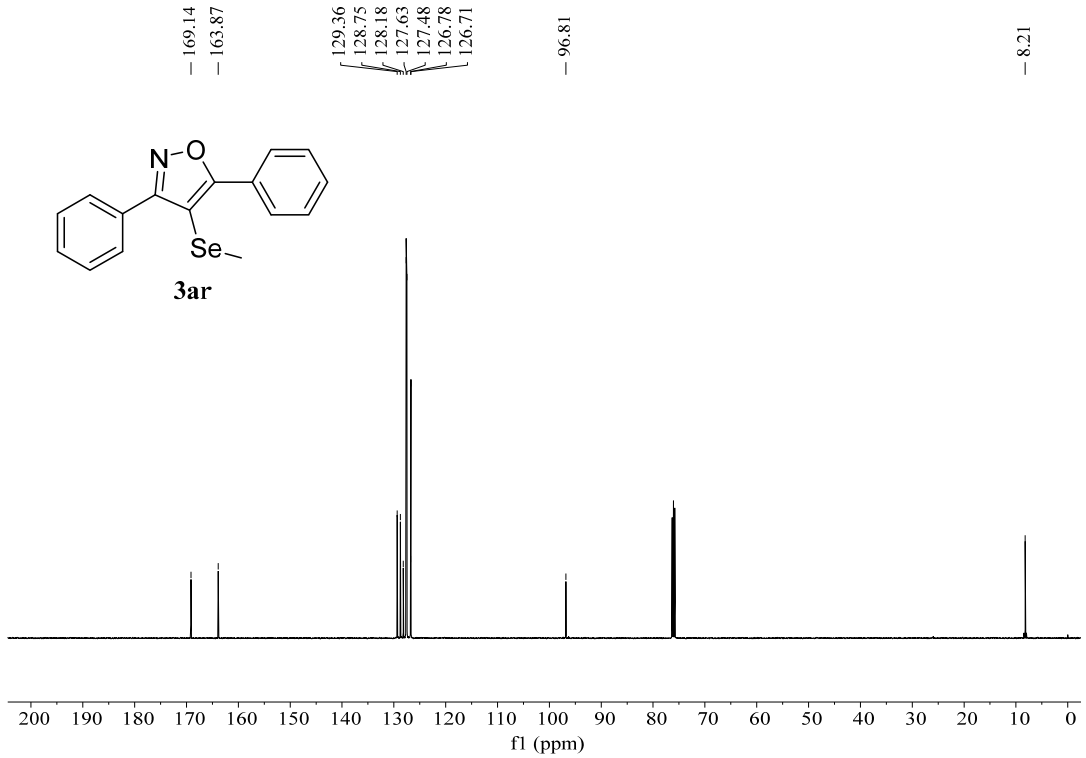


Figure S64 ^{13}C NMR (125 MHz) spectrum of **3ar** in $CDCl_3$

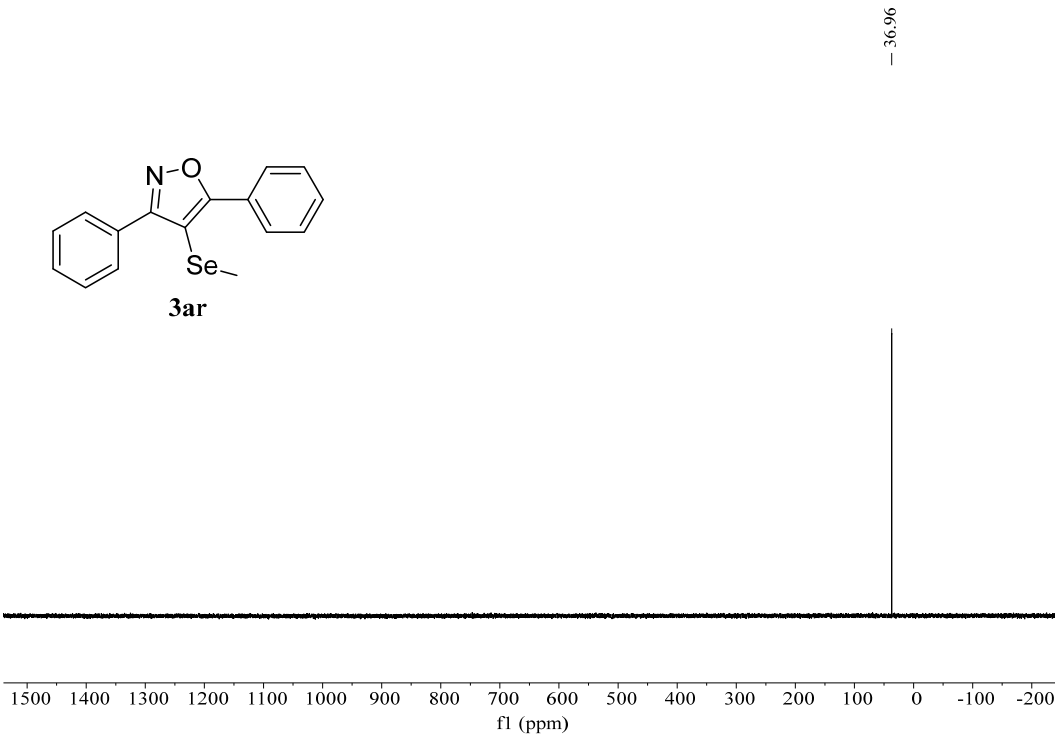


Figure S65 ^{77}Se NMR (95.5 MHz) spectrum of **3ar** in $CDCl_3$

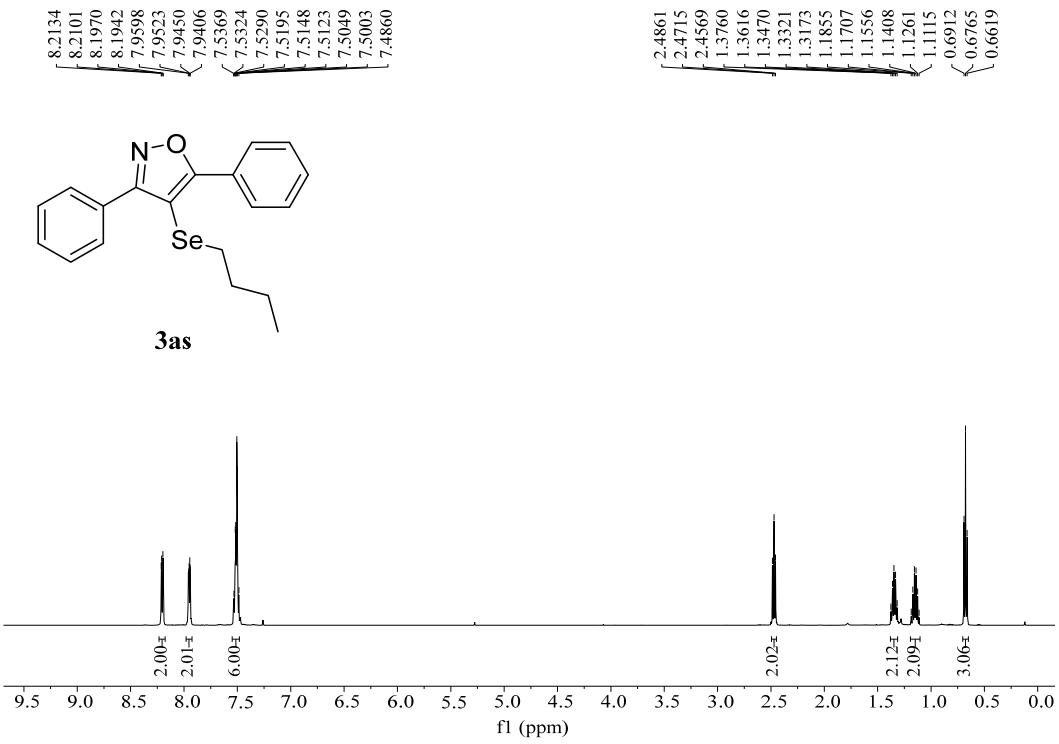


Figure S66 ^{1}H NMR (500 MHz) spectrum of **3as** in $CDCl_3$

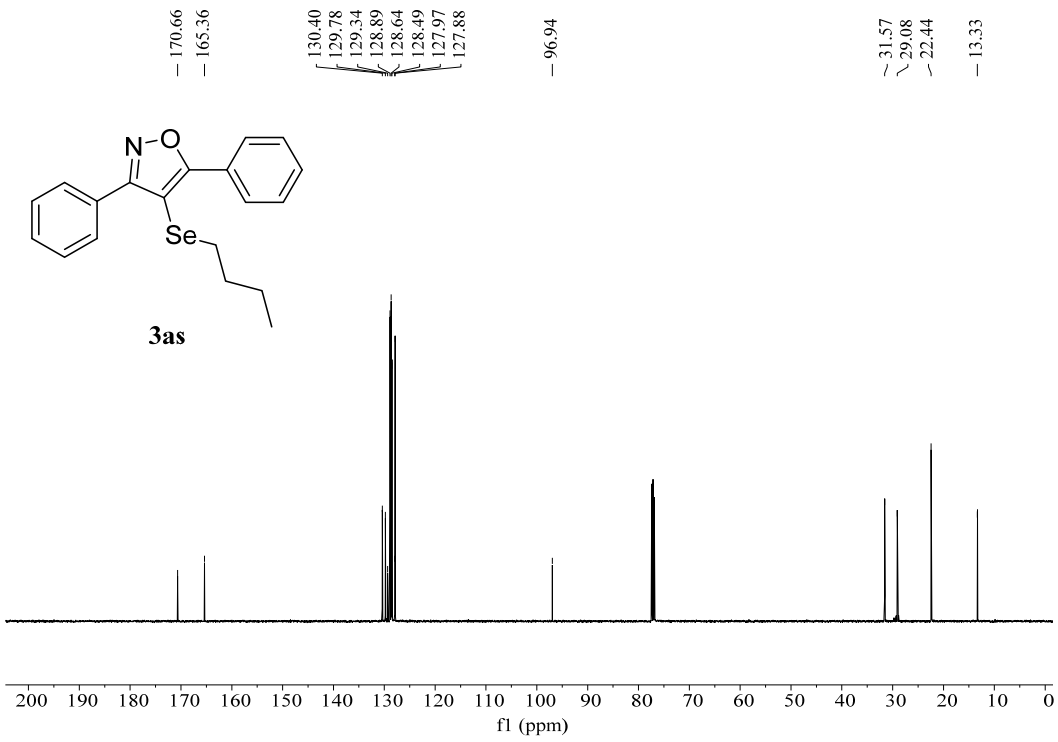


Figure S67 ^{13}C NMR (125 MHz) spectrum of **3as** in $CDCl_3$

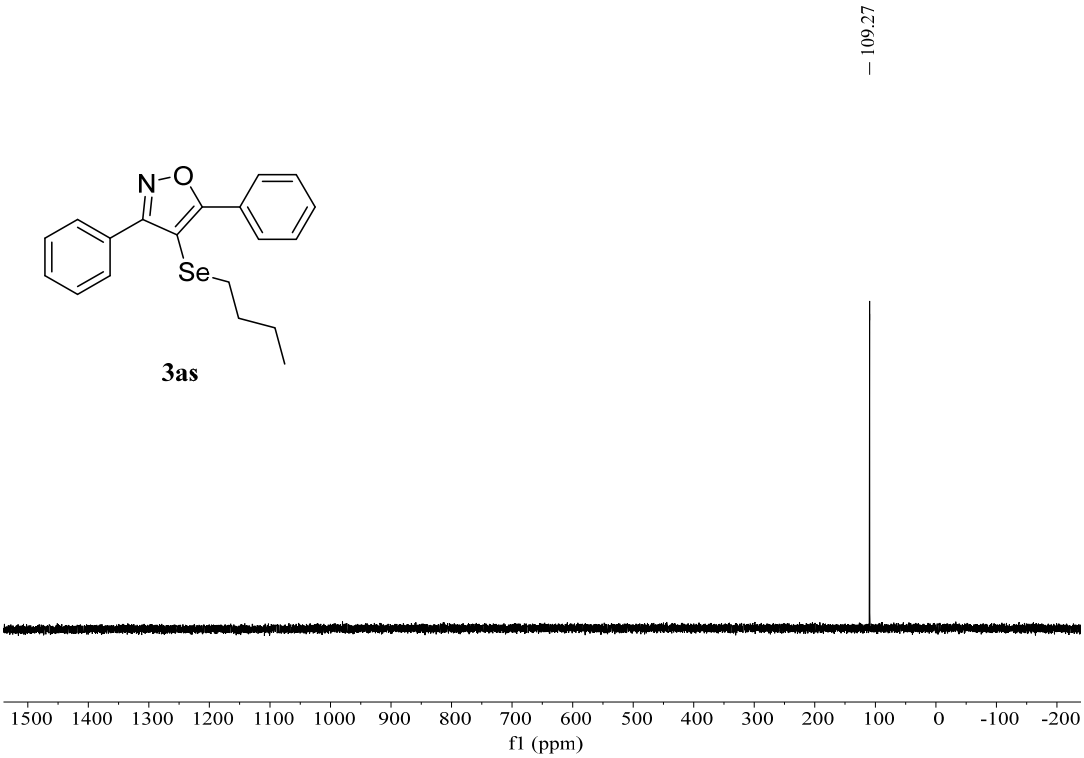


Figure S68 ^{77}Se NMR (95.5 MHz) spectrum of **3as** in $CDCl_3$

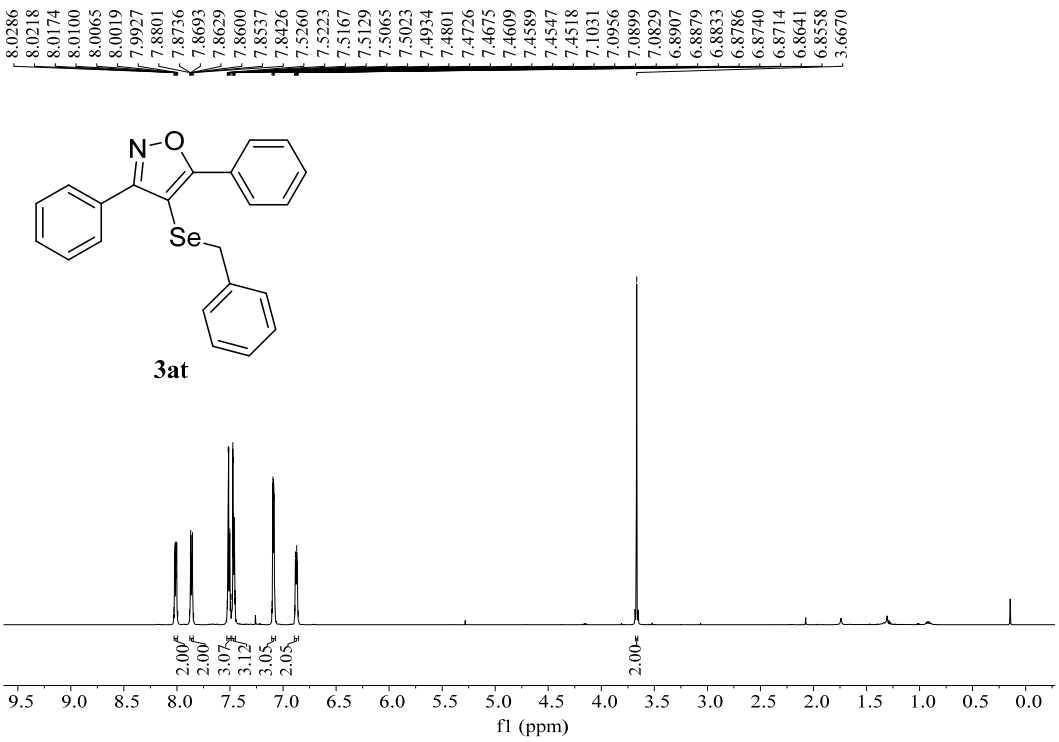


Figure S69 ^{1}H NMR (500 MHz) spectrum of **3at** in $CDCl_3$

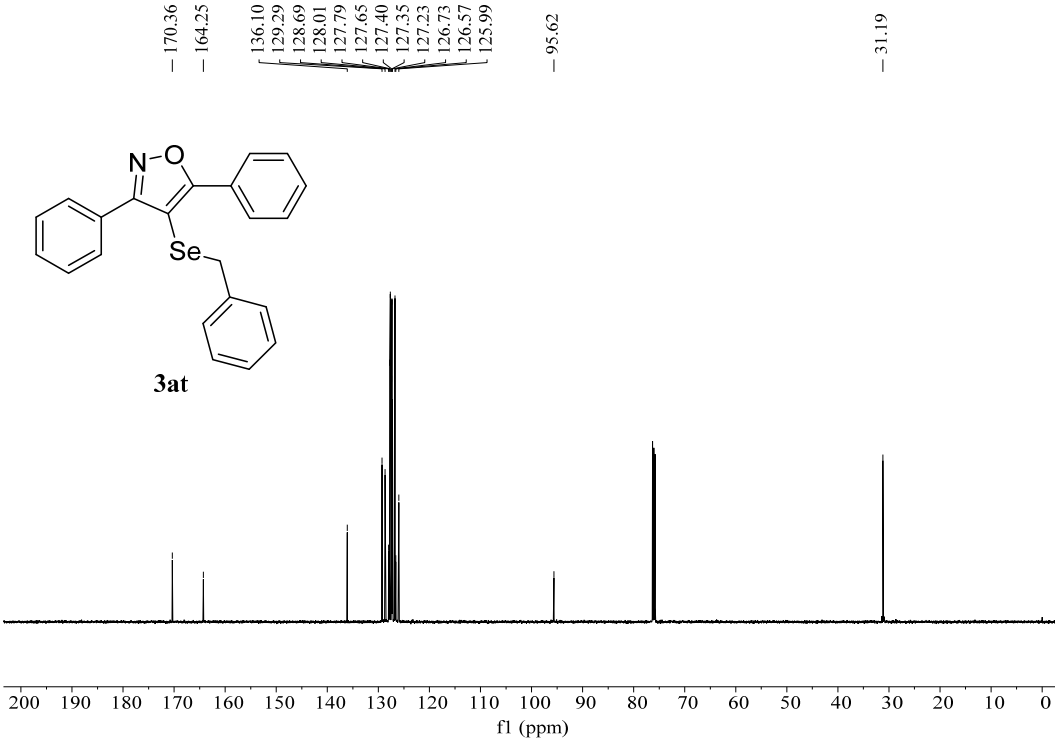


Figure S70 ^{13}C NMR (125 MHz) spectrum of **3at** in $CDCl_3$

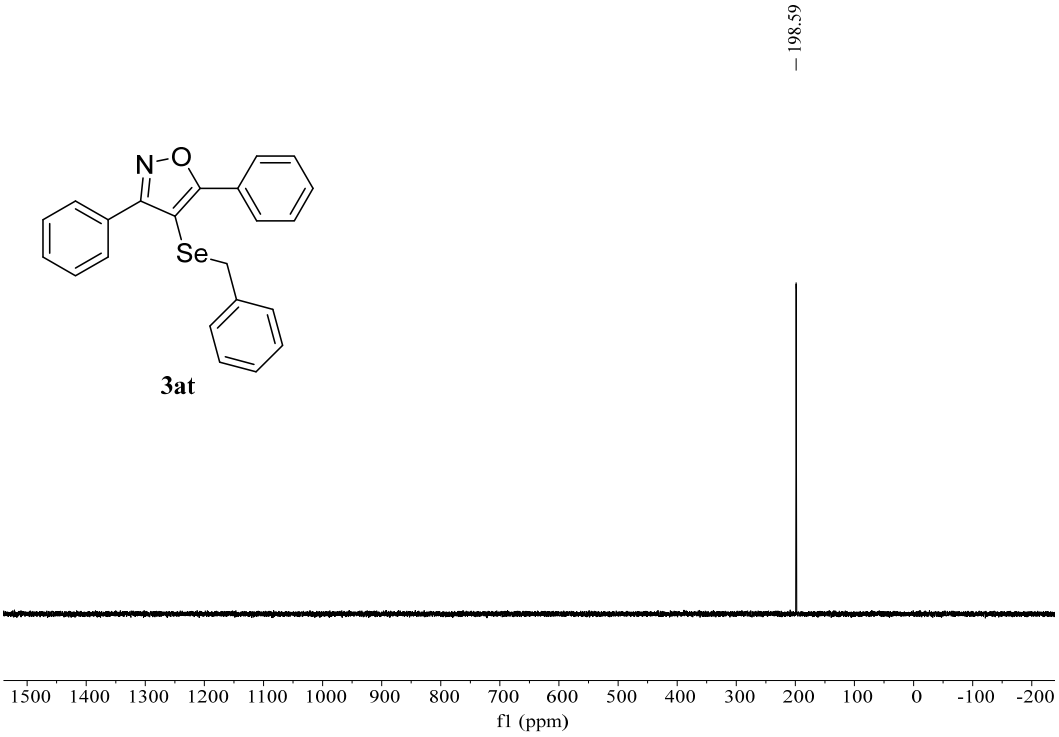


Figure S71 ^{77}Se NMR (95.5 MHz) spectrum of **3at** in $CDCl_3$

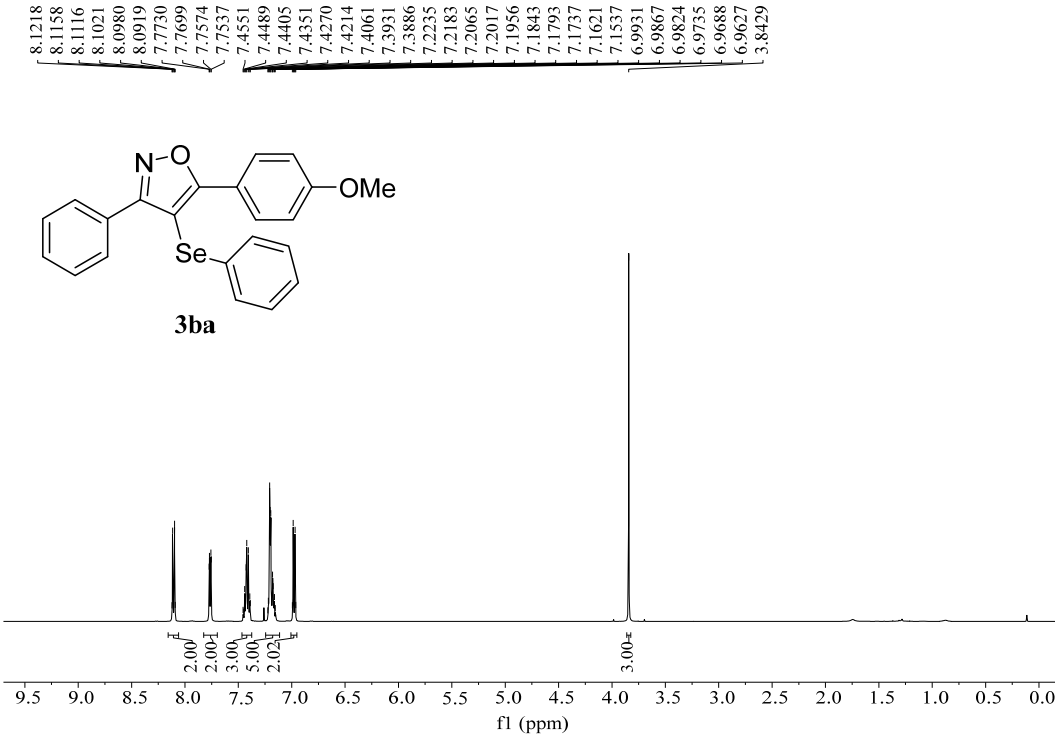


Figure S72 ^{1}H NMR (500 MHz) spectrum of **3ba** in $CDCl_3$

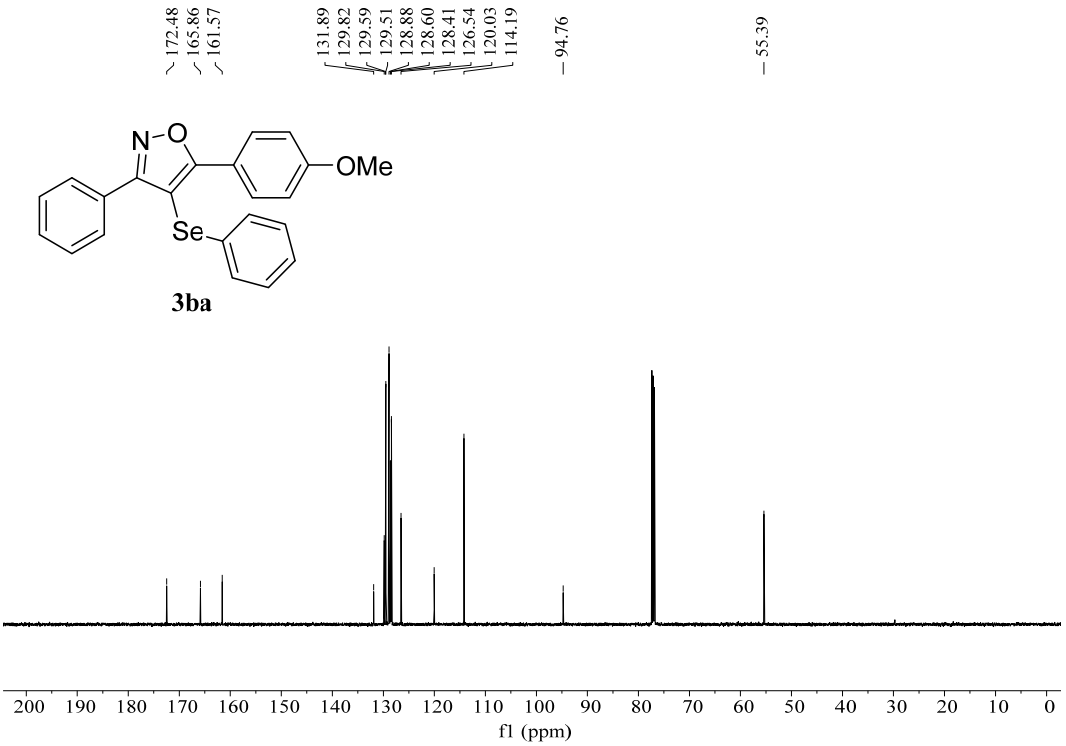


Figure S73 ^{13}C NMR (125 MHz) spectrum of **3ba** in $CDCl_3$

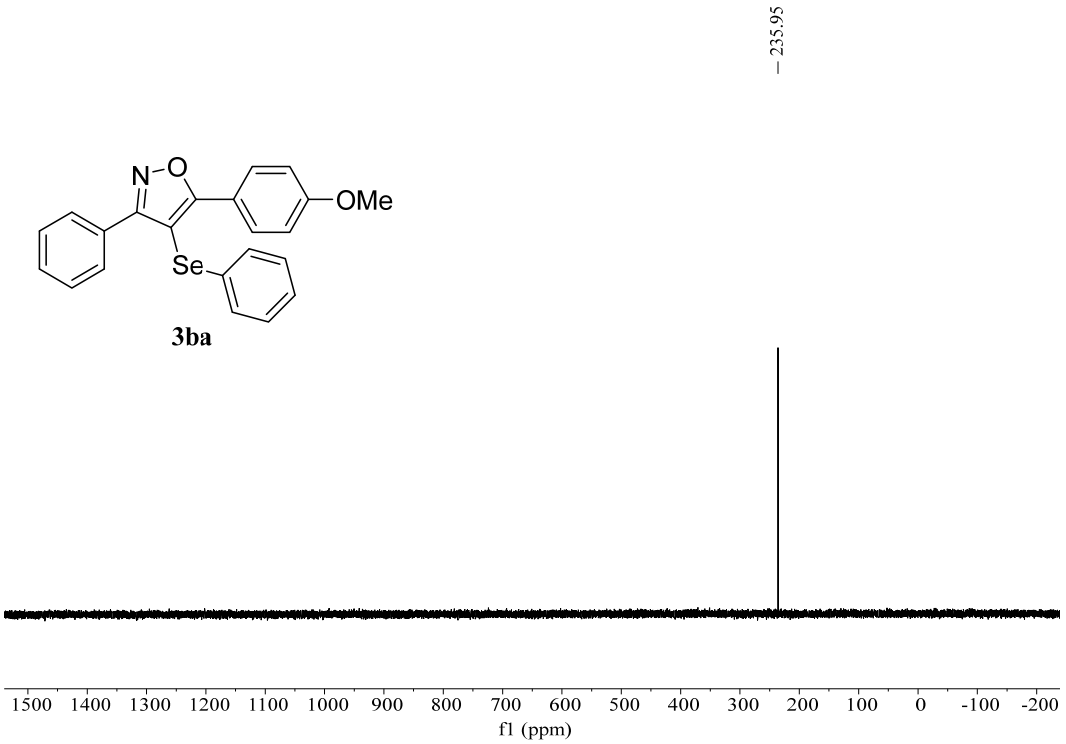


Figure S74 ^{77}Se NMR (95.5 MHz) spectrum of **3ba** in $CDCl_3$

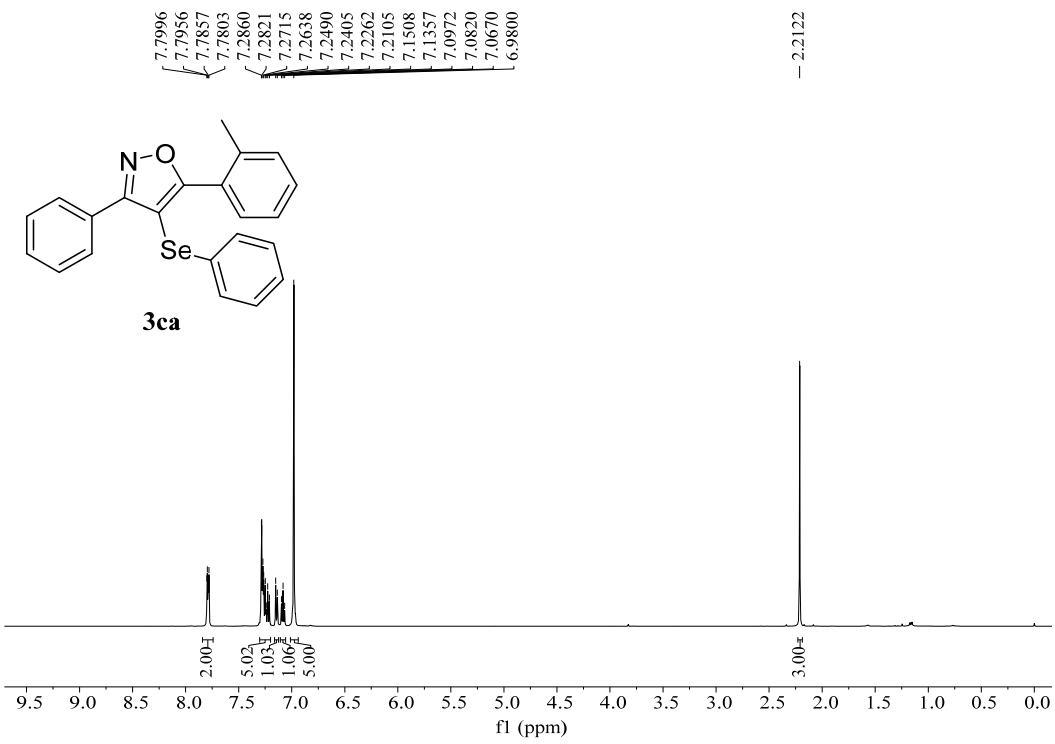


Figure S75 ^{1}H NMR (500 MHz) spectrum of **3ca** in $CDCl_3$

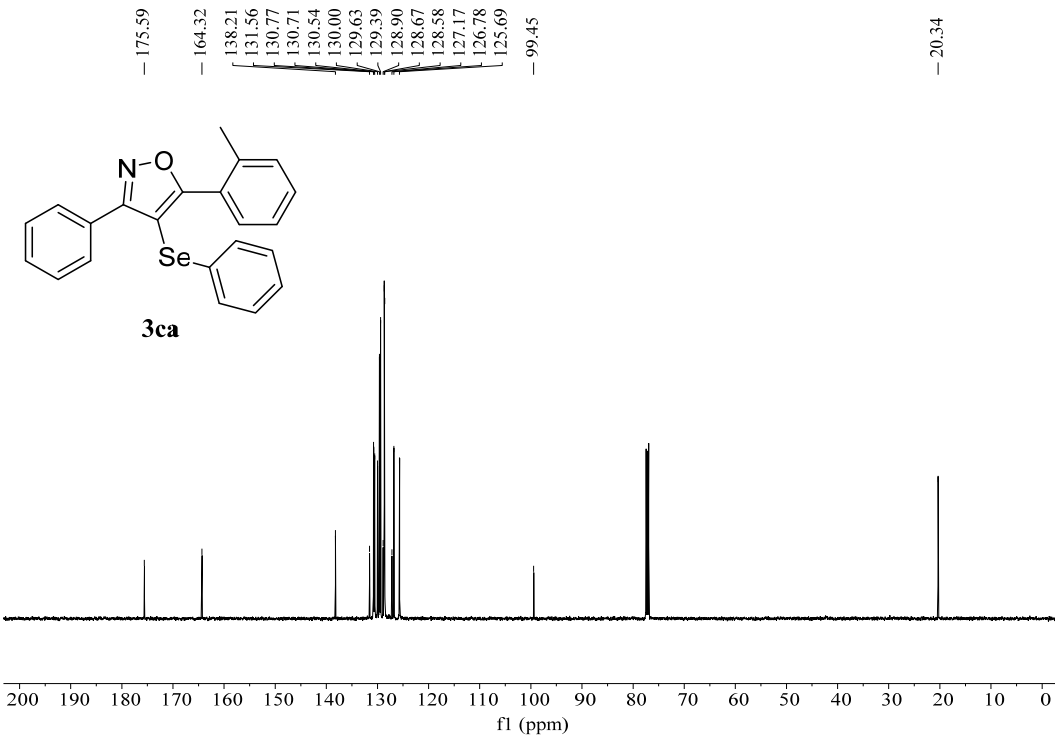


Figure S76 ^{13}C NMR (125 MHz) spectrum of **3ca** in $CDCl_3$

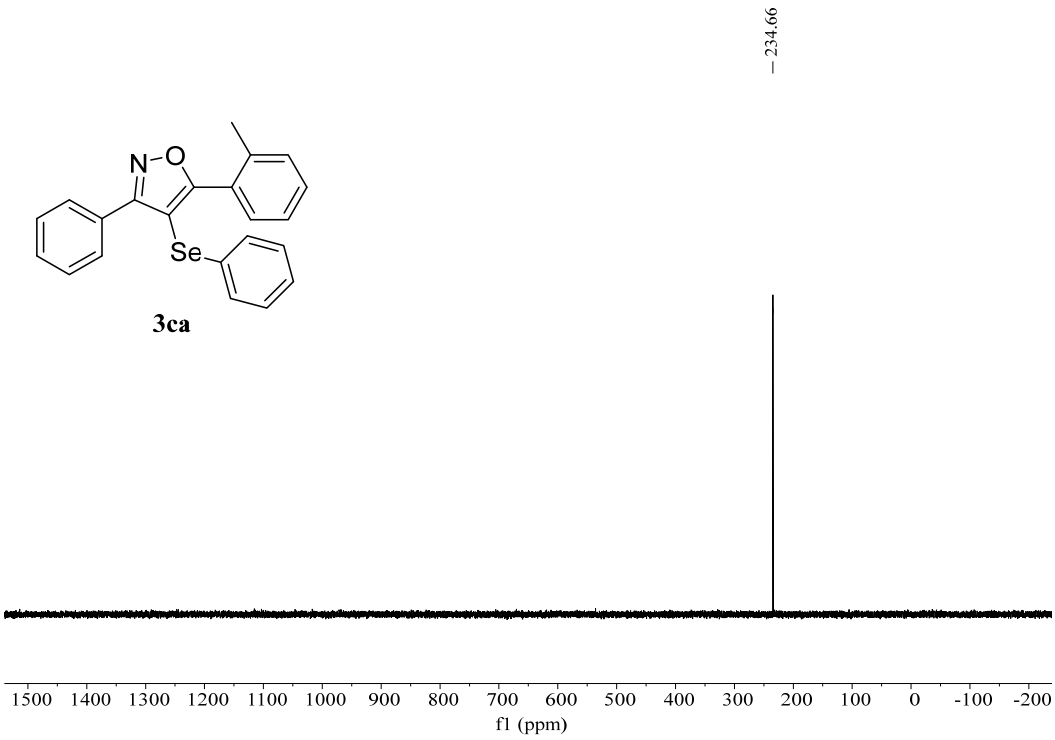


Figure S77 ^{77}Se NMR (95.5 MHz) spectrum of **3ca** in $CDCl_3$

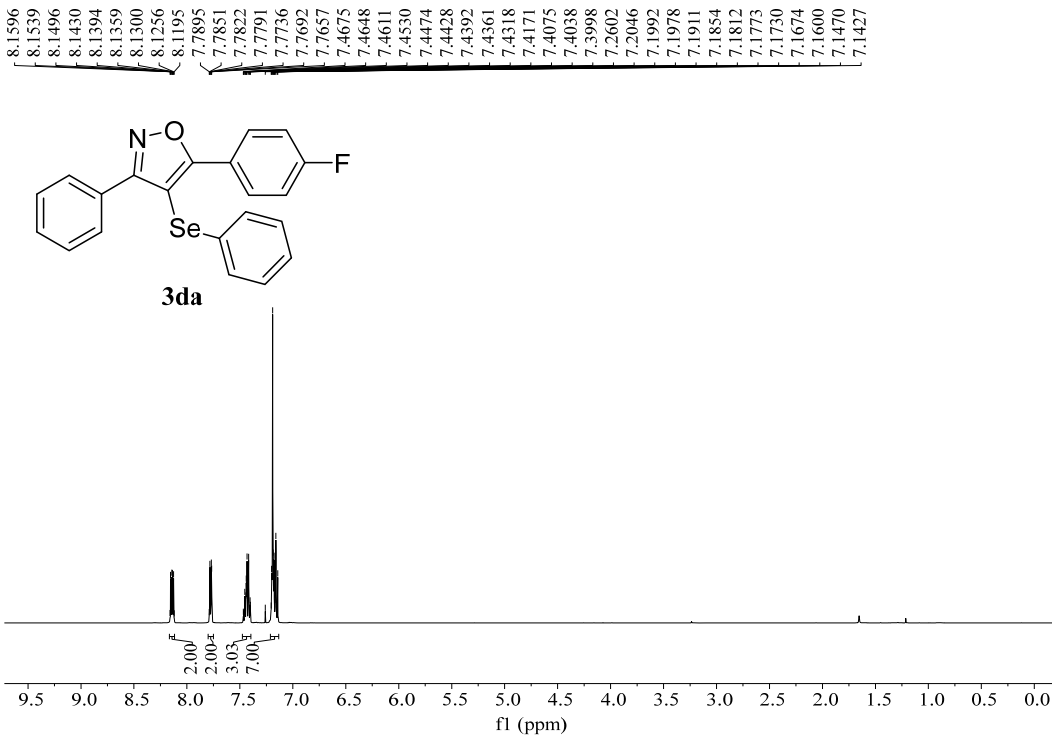


Figure S78 ^{1}H NMR (500 MHz) spectrum of **3da** in $CDCl_3$

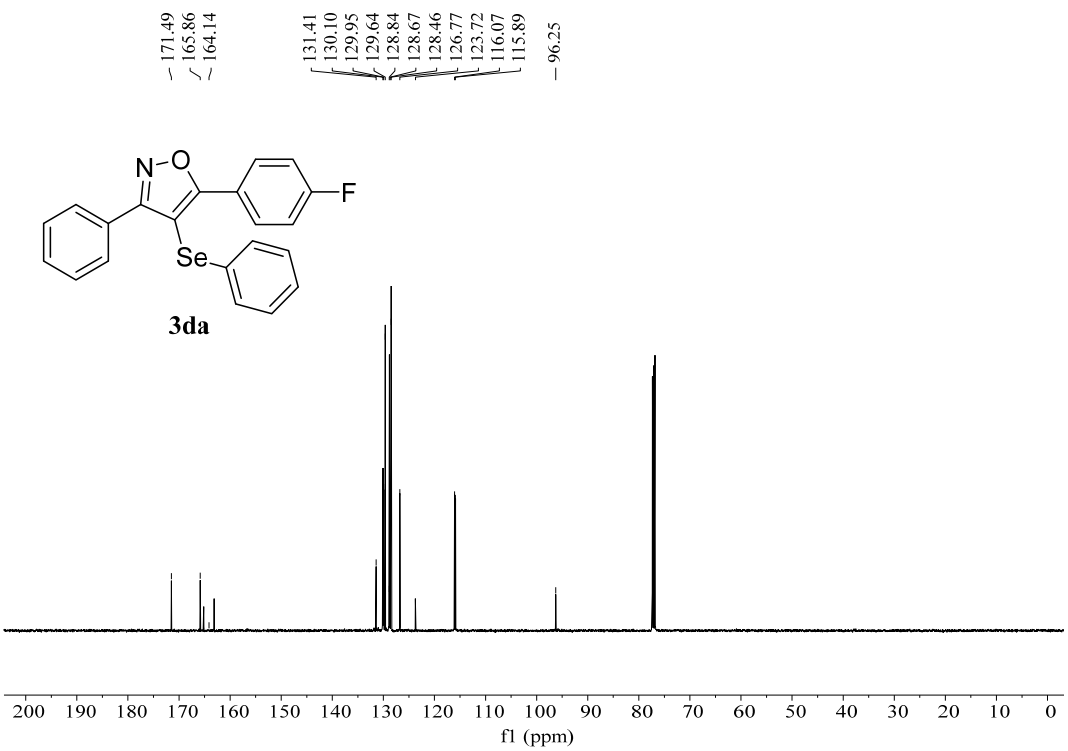


Figure S79 ^{13}C NMR (125 MHz) spectrum of **3da** in $CDCl_3$

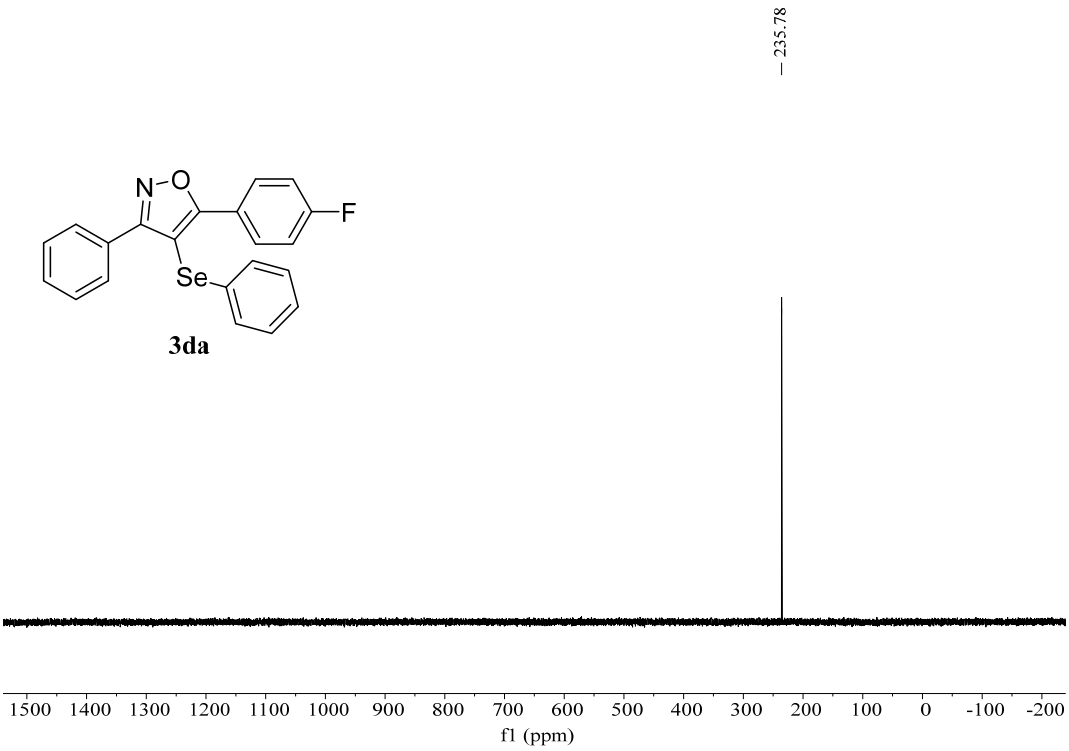


Figure S80 ^{77}Se NMR (95.5 MHz) spectrum of **3da** in $CDCl_3$

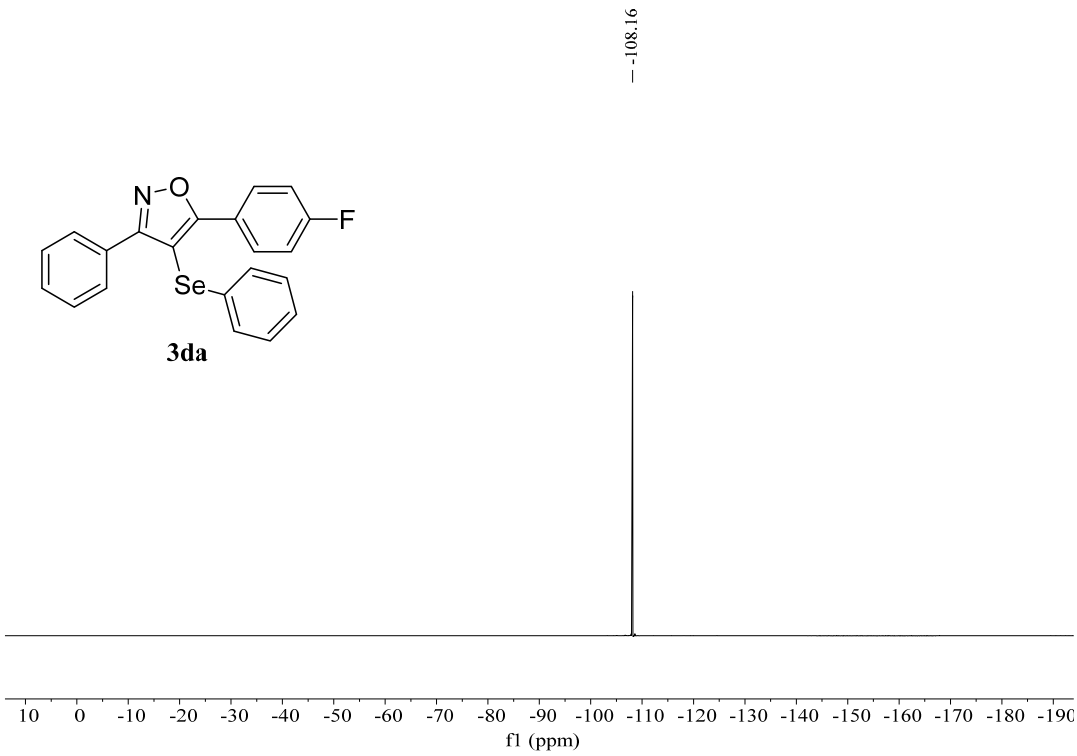


Figure S81 ^{19}F NMR (470 MHz) spectrum of **3da** in $CDCl_3$

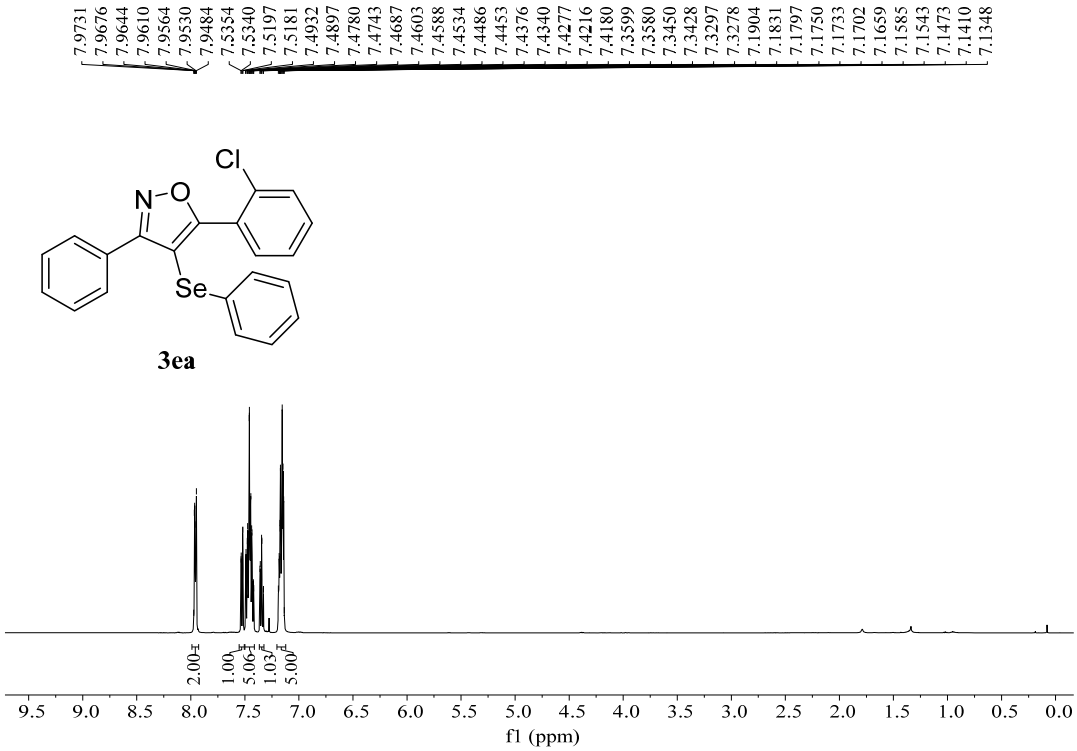


Figure S82 ^{1}H NMR (500 MHz) spectrum of **3ea** in $CDCl_3$

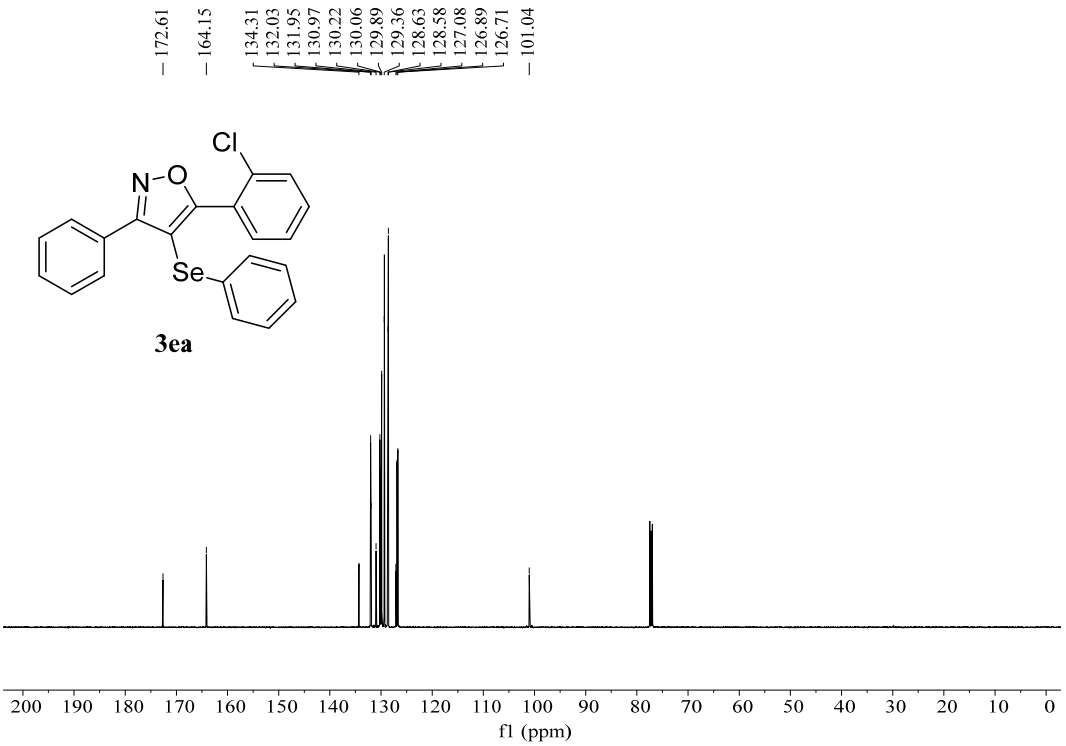


Figure S83 ^{13}C NMR (125 MHz) spectrum of **3ea** in $CDCl_3$

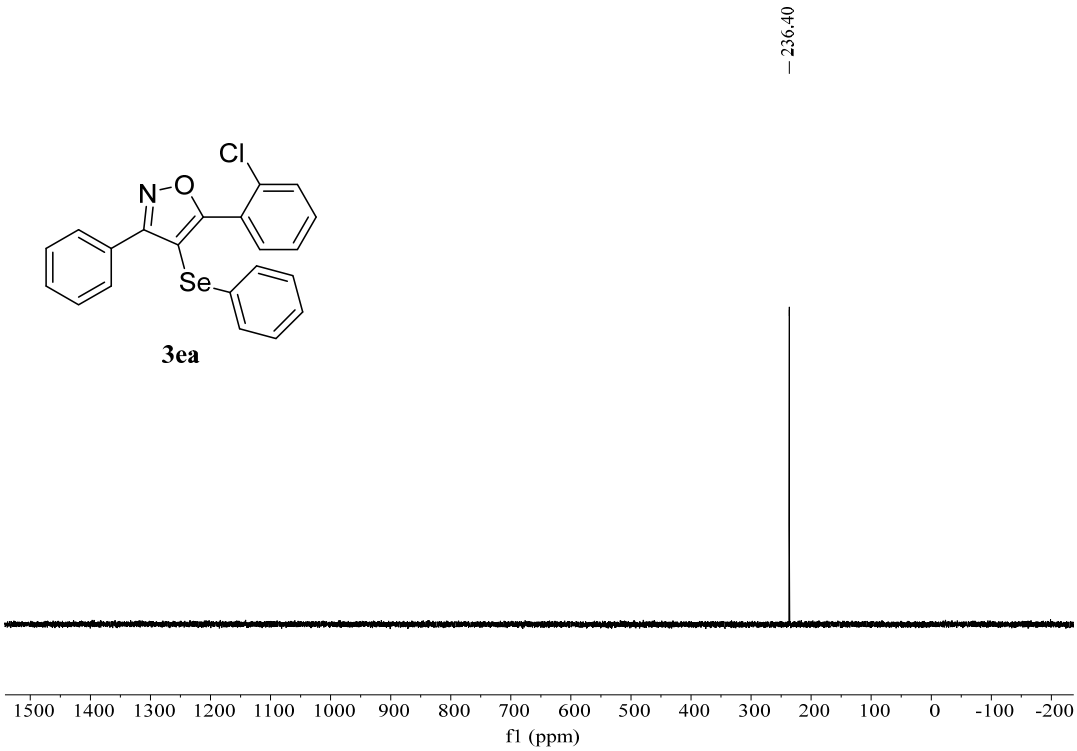


Figure S84 ^{77}Se NMR (95.5 MHz) spectrum of **3ea** in $CDCl_3$

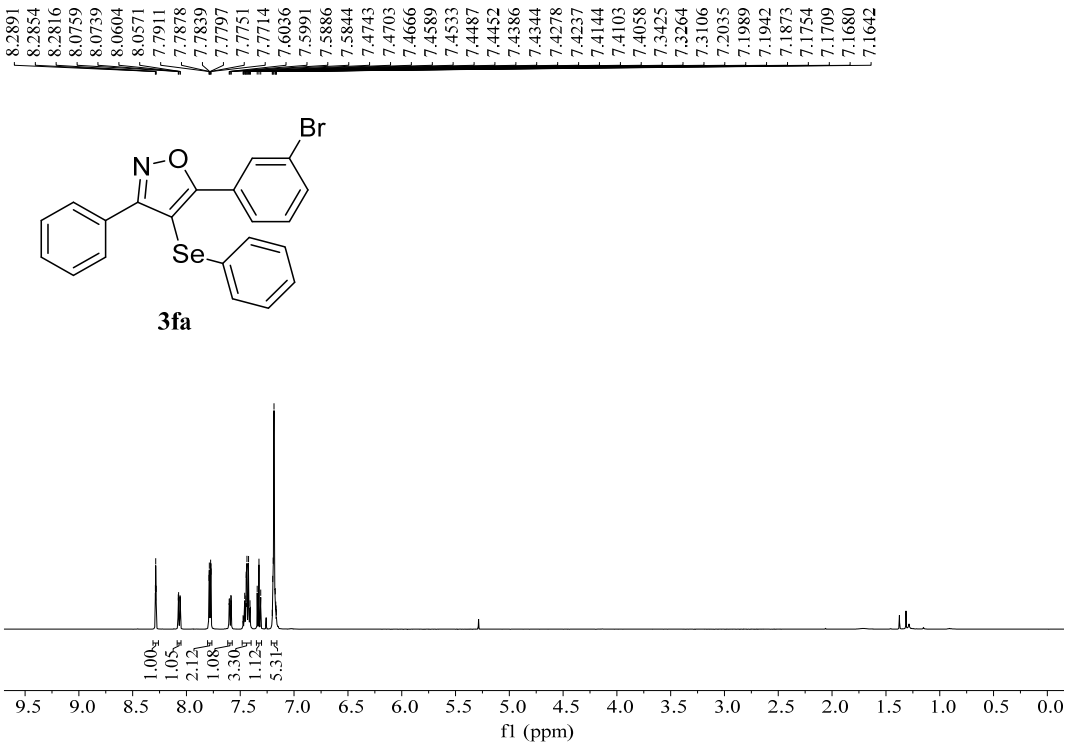


Figure S85 ^{1}H NMR (500 MHz) spectrum of **3fa** in $CDCl_3$

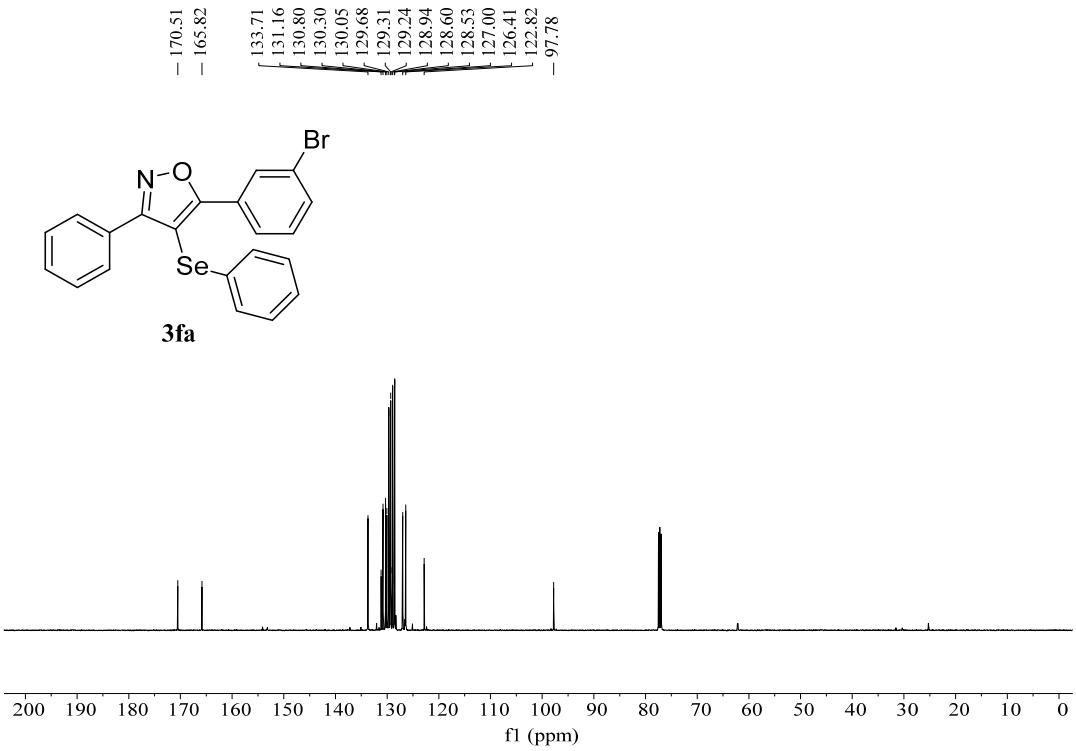


Figure S86 ^{13}C NMR (125 MHz) spectrum of **3fa** in $CDCl_3$

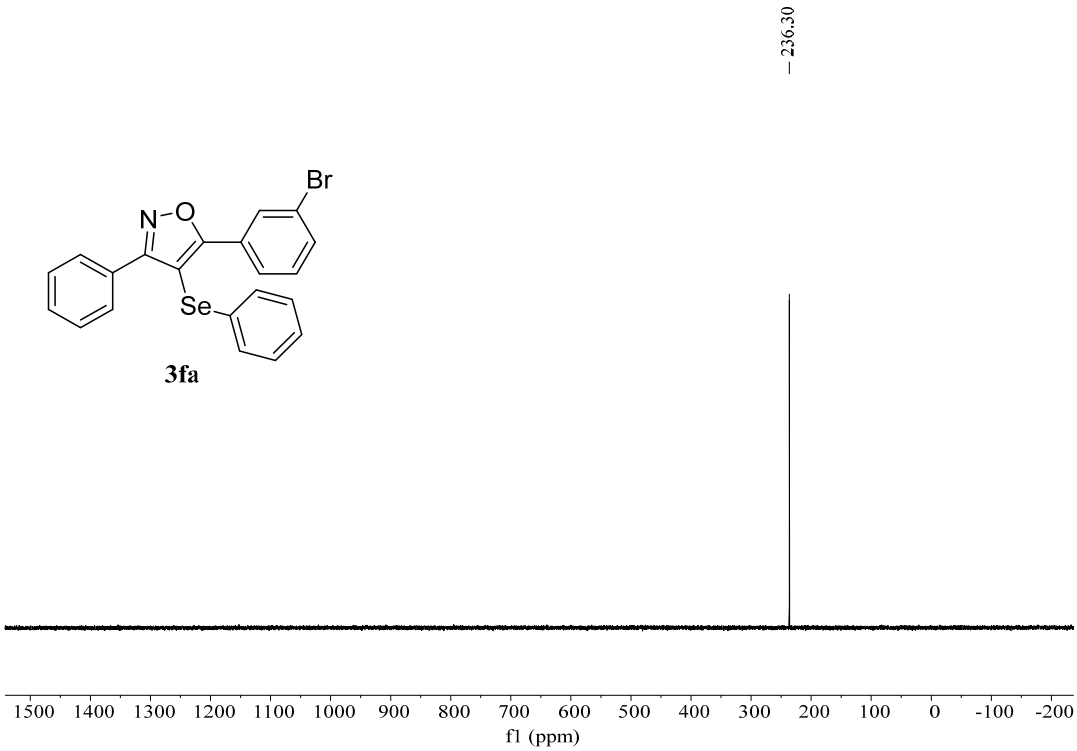


Figure S87 ^{77}Se NMR (95.5 MHz) spectrum of **3fa** in $CDCl_3$

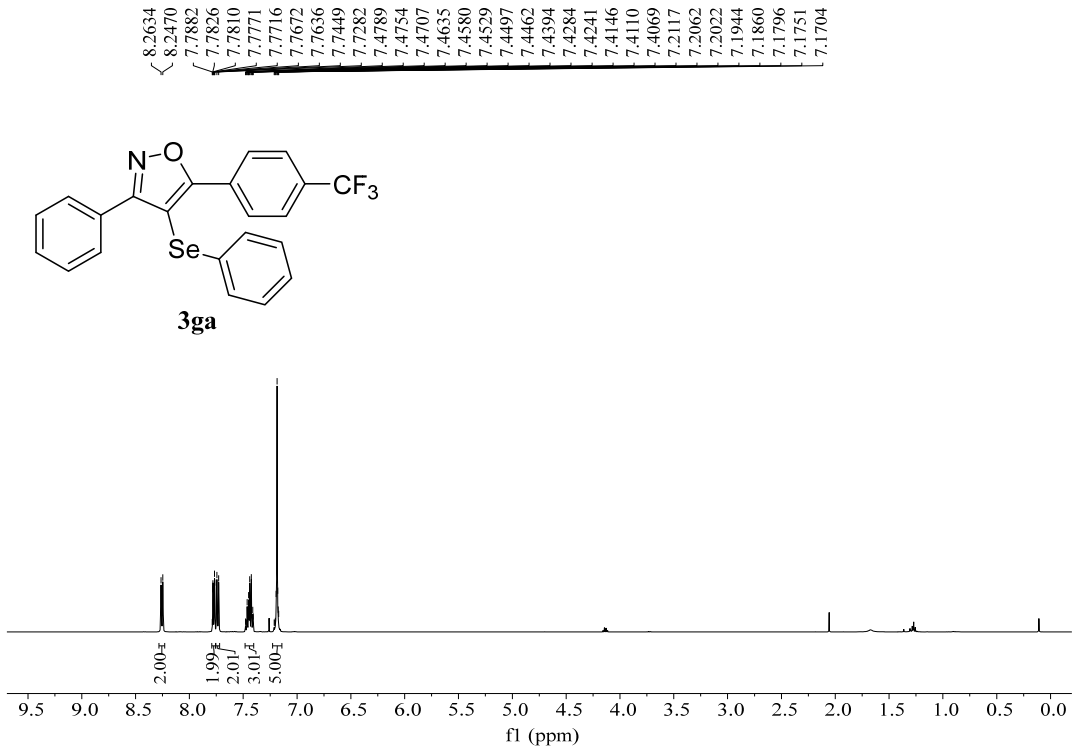


Figure S88 ^{1}H NMR (500 MHz) spectrum of **3ga** in $CDCl_3$

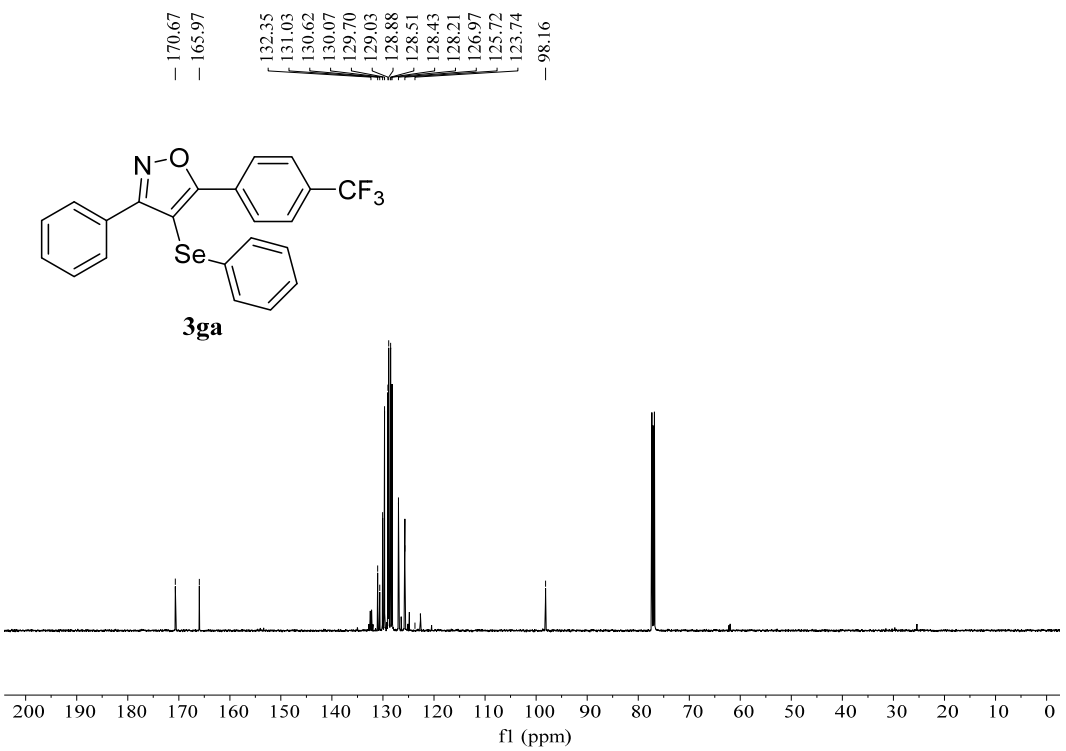


Figure S89 ^{13}C NMR (125 MHz) spectrum of **3ga** in $CDCl_3$

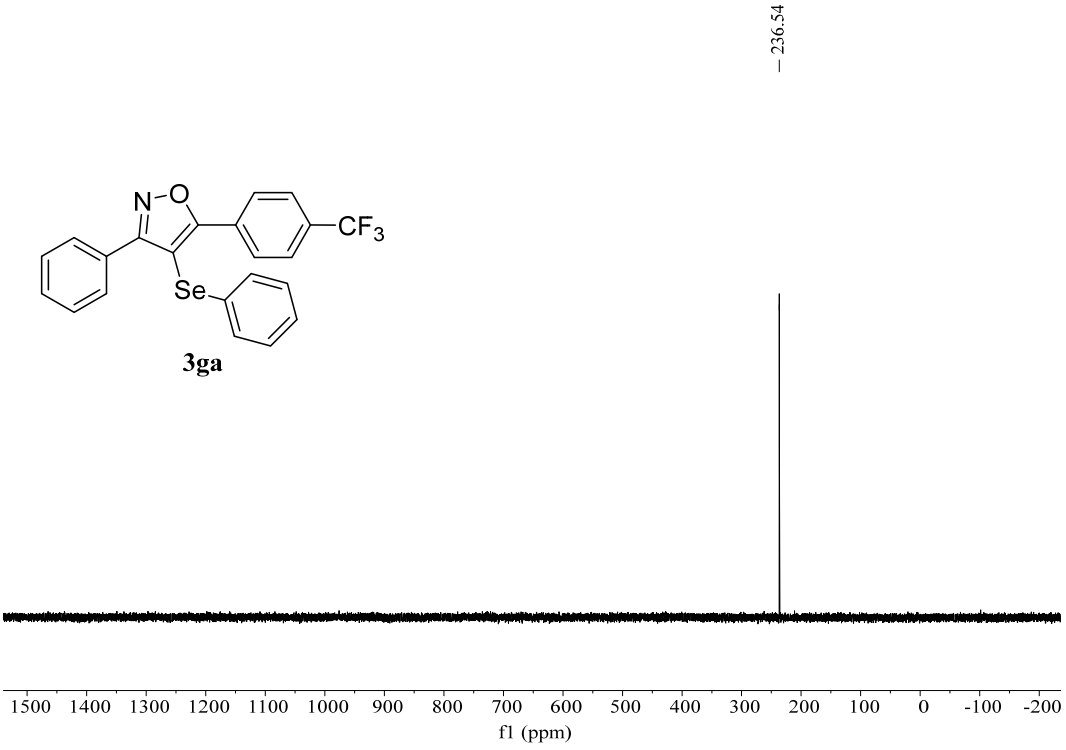


Figure S90 ^{77}Se NMR (95.5 MHz) spectrum of **3ga** in $CDCl_3$

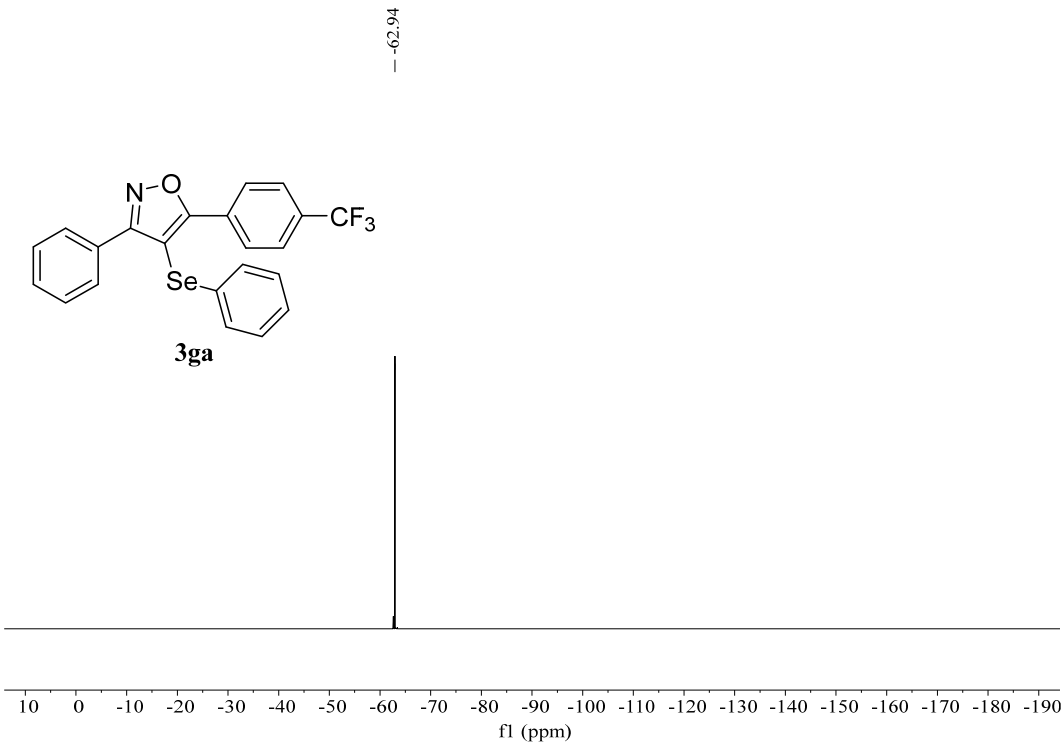


Figure S91 ^{19}F NMR (470 MHz) spectrum of **3ga** in $CDCl_3$

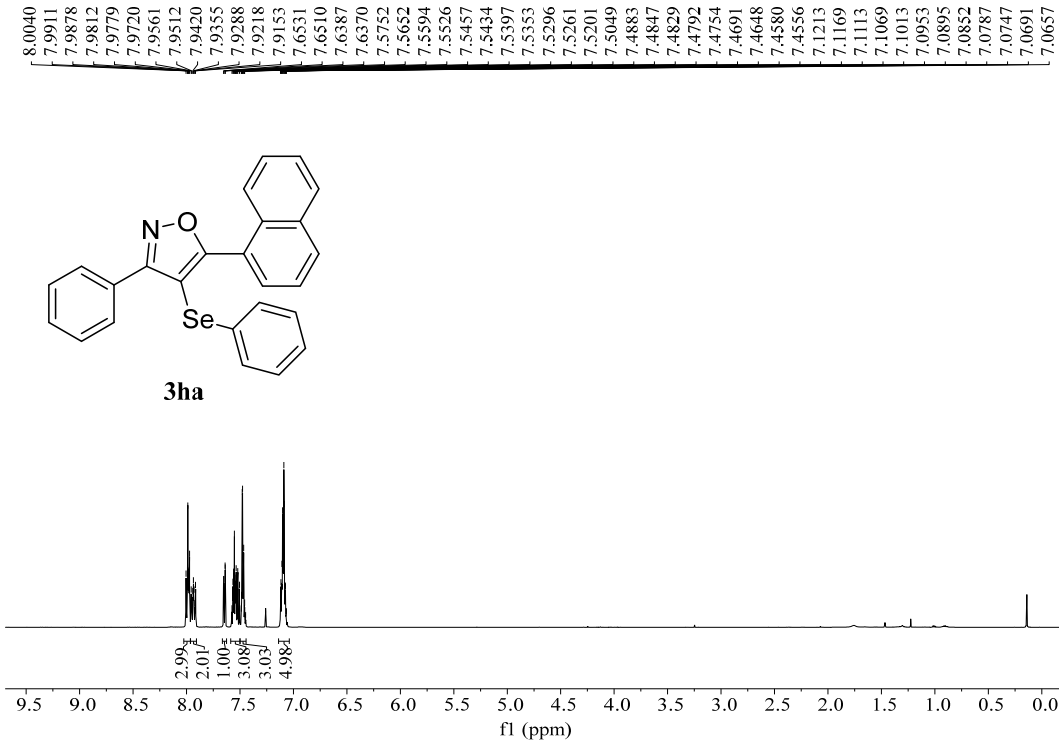


Figure S92 ^{1}H NMR (500 MHz) spectrum of **3ha** in $CDCl_3$

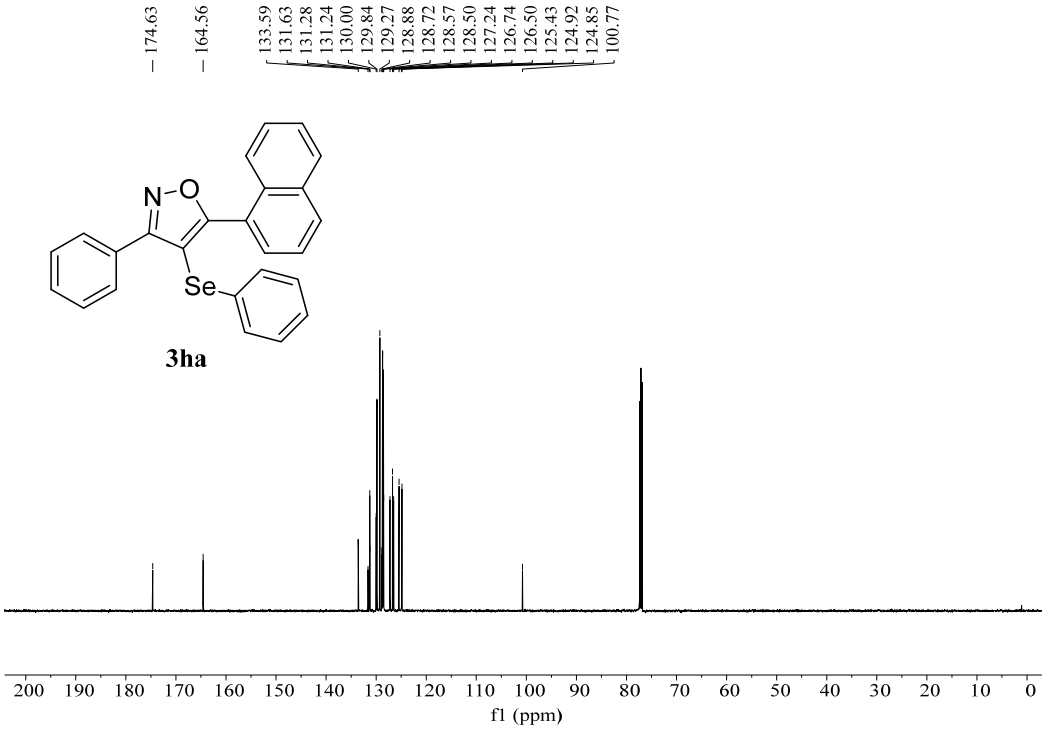


Figure S93 ^{13}C NMR (125 MHz) spectrum of **3ha** in $CDCl_3$

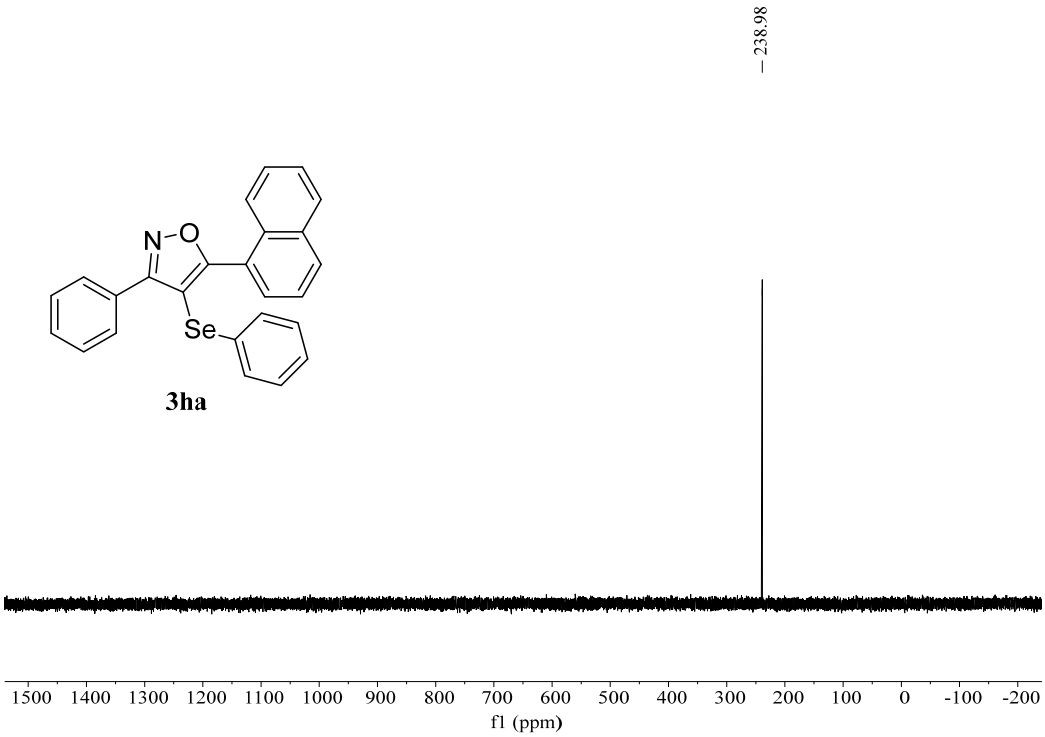


Figure S94 ^{77}Se NMR (95.5 MHz) spectrum of **3ha** in $CDCl_3$

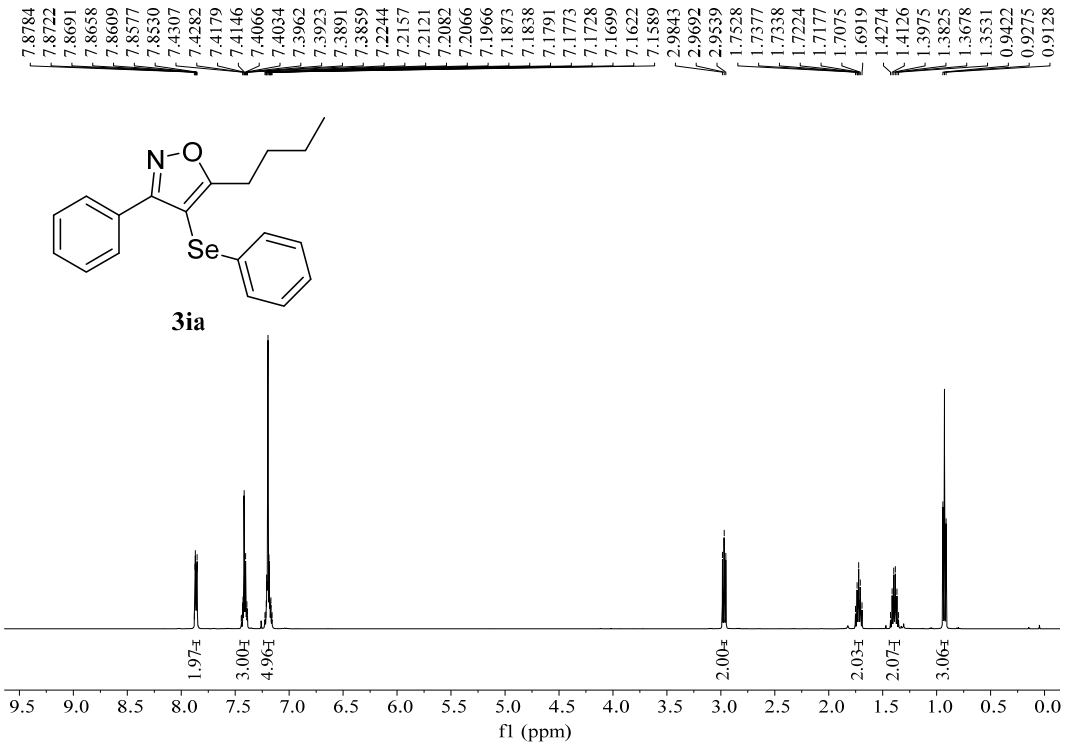


Figure S95 ^{1}H NMR (500 MHz) spectrum of **3ia** in $CDCl_3$

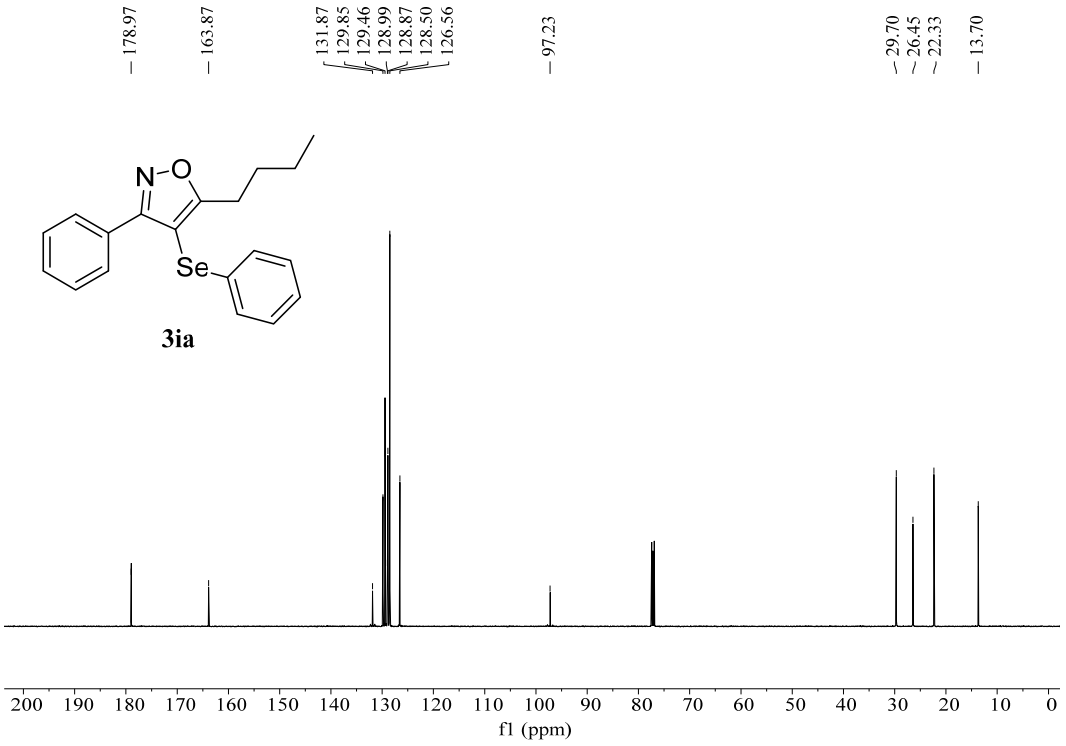


Figure S96 ^{13}C NMR (125 MHz) spectrum of **3ia** in $CDCl_3$

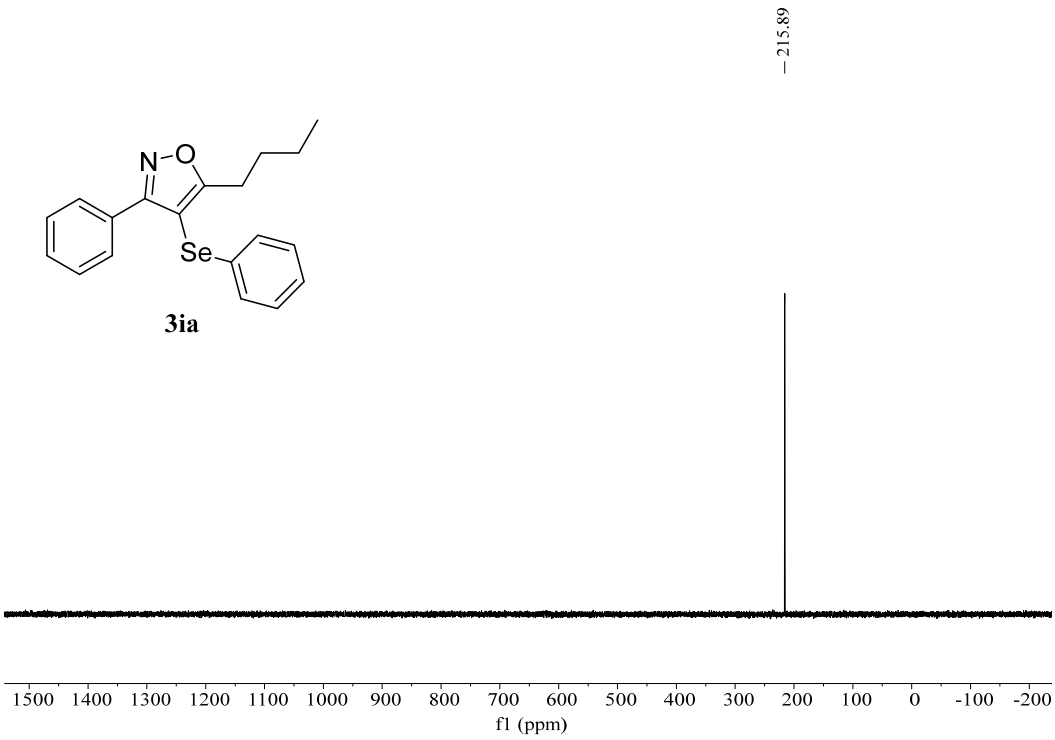


Figure S97 ^{77}Se NMR (95.5 MHz) spectrum of **3ia** in $CDCl_3$

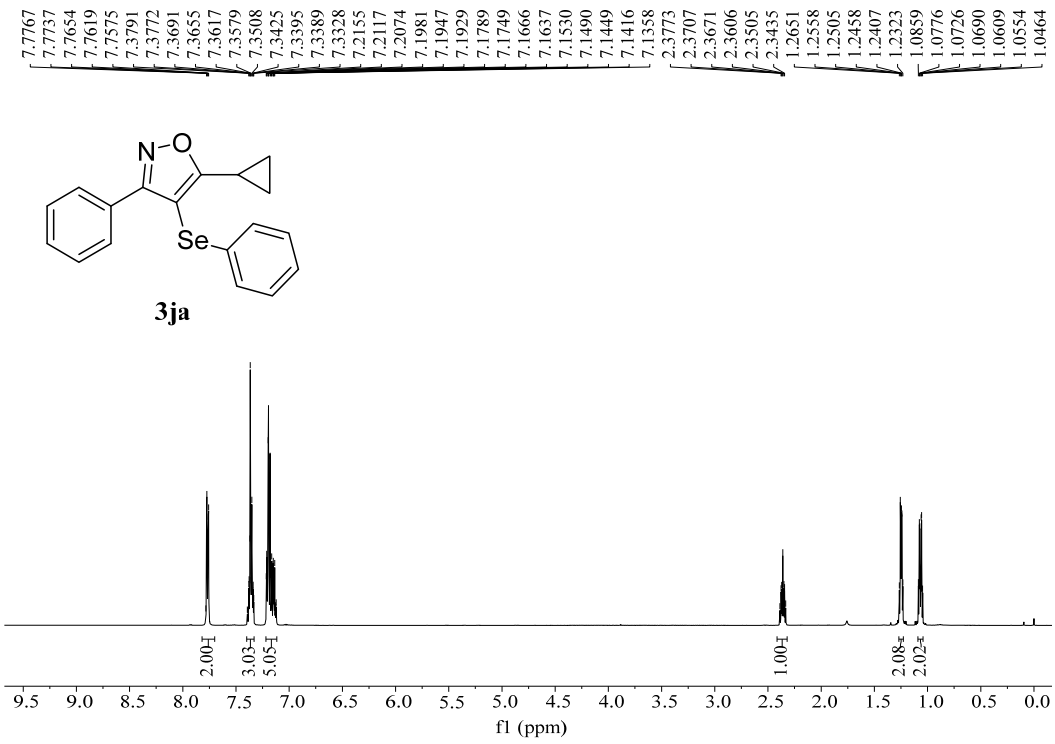


Figure S98 ^{1}H NMR (500 MHz) spectrum of **3ja** in $CDCl_3$

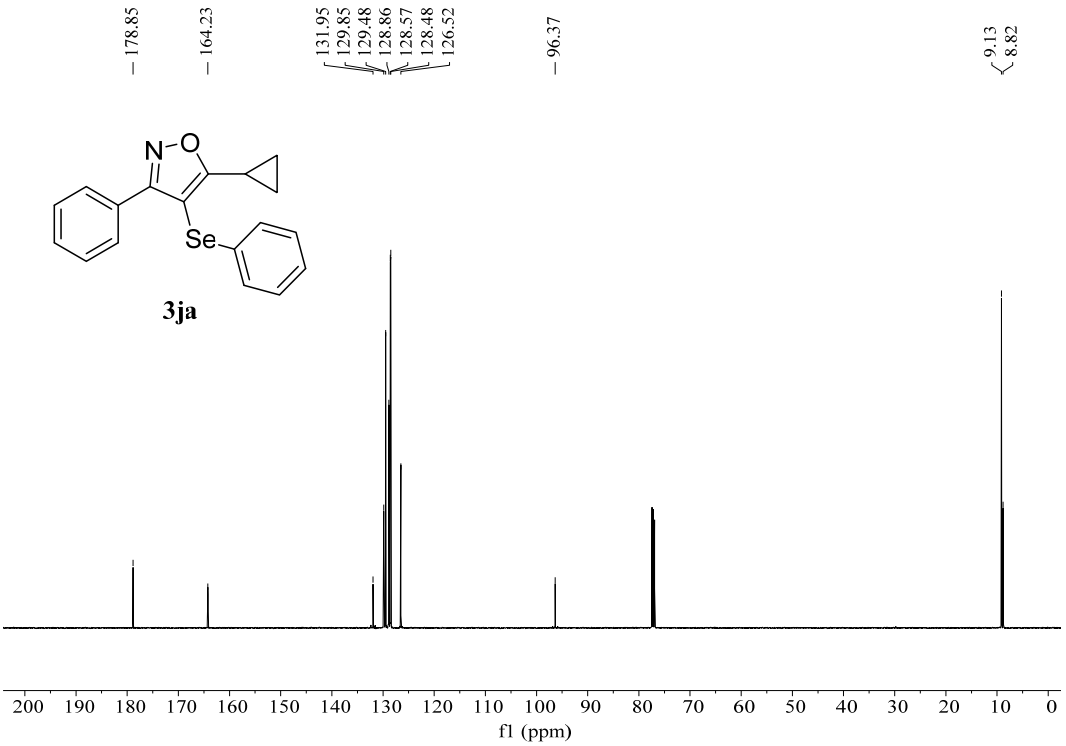


Figure S99 ^{13}C NMR (125 MHz) spectrum of **3ja** in $CDCl_3$

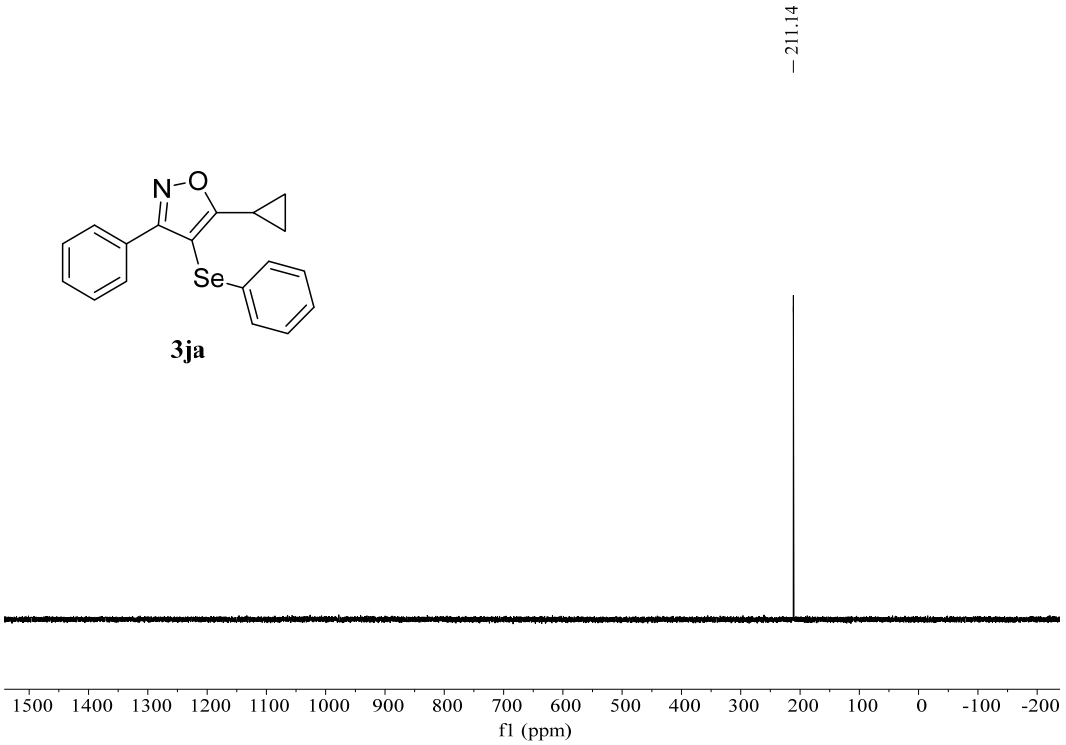


Figure S100 ^{77}Se NMR (95.5 MHz) spectrum of **3ja** in $CDCl_3$

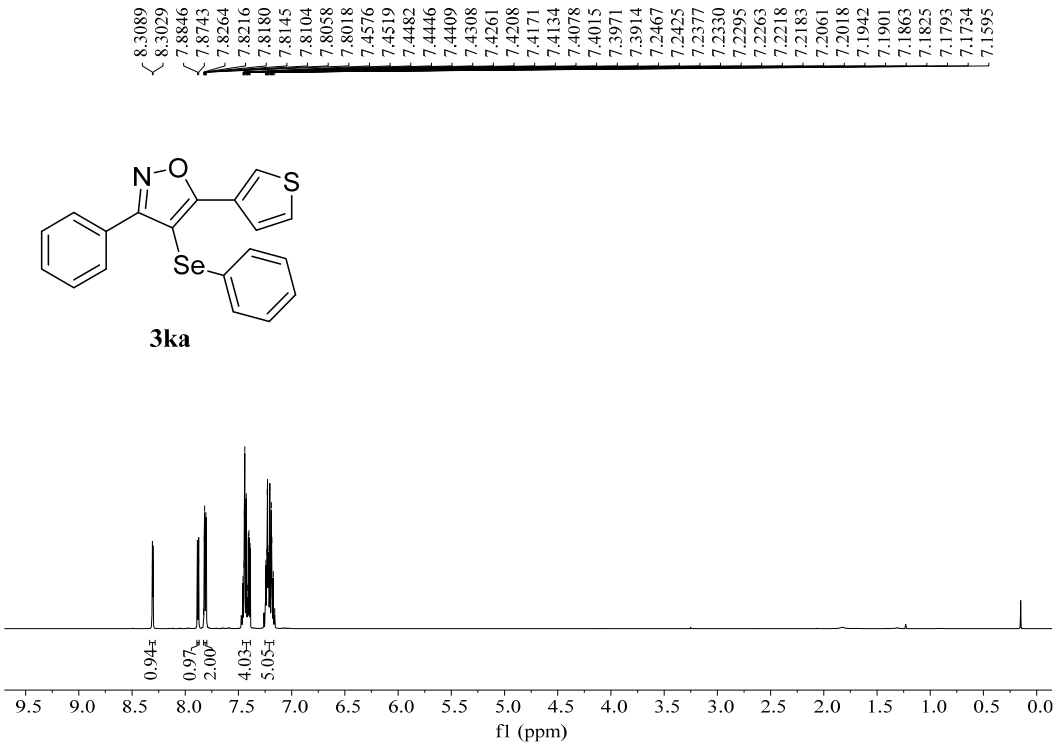


Figure S101 ^{1}H NMR (500 MHz) spectrum of **3ka** in $CDCl_3$

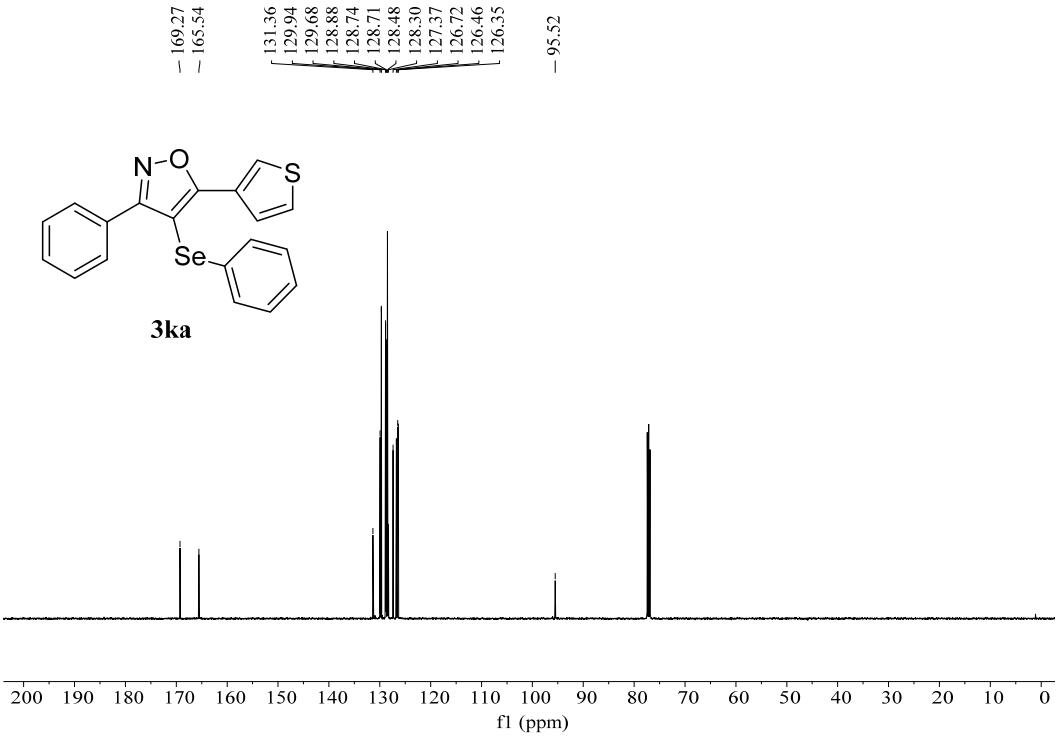


Figure S102 ^{13}C NMR (125 MHz) spectrum of **3ka** in $CDCl_3$

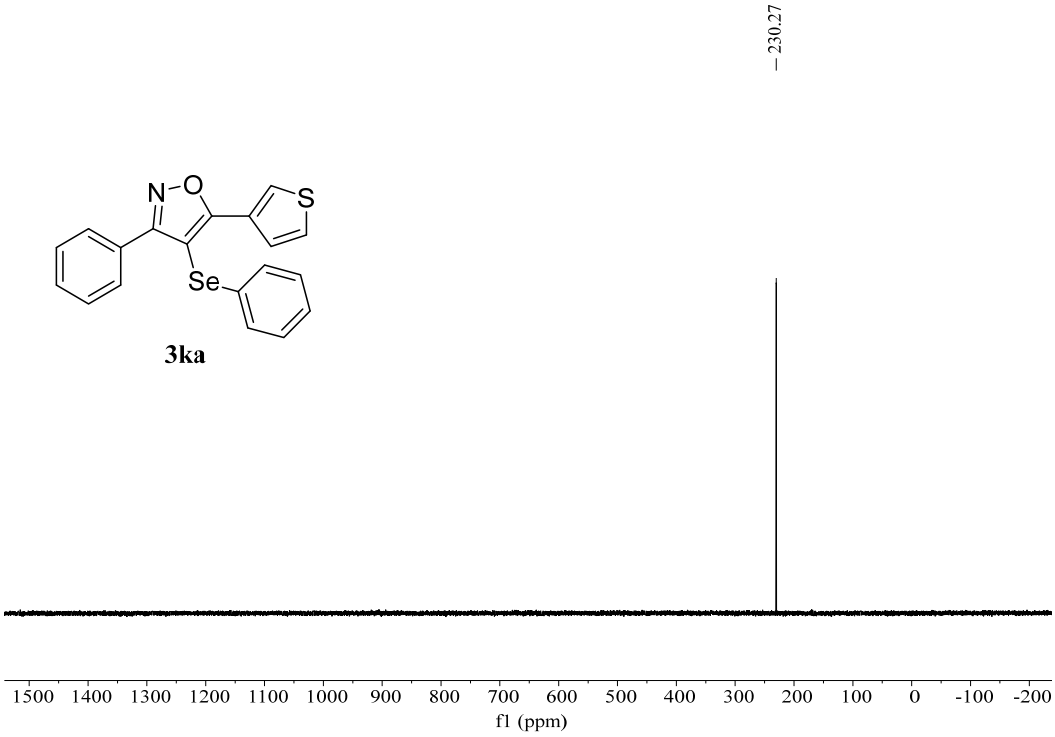


Figure S103 ^{77}Se NMR (95.5 MHz) spectrum of **3ka** in $CDCl_3$

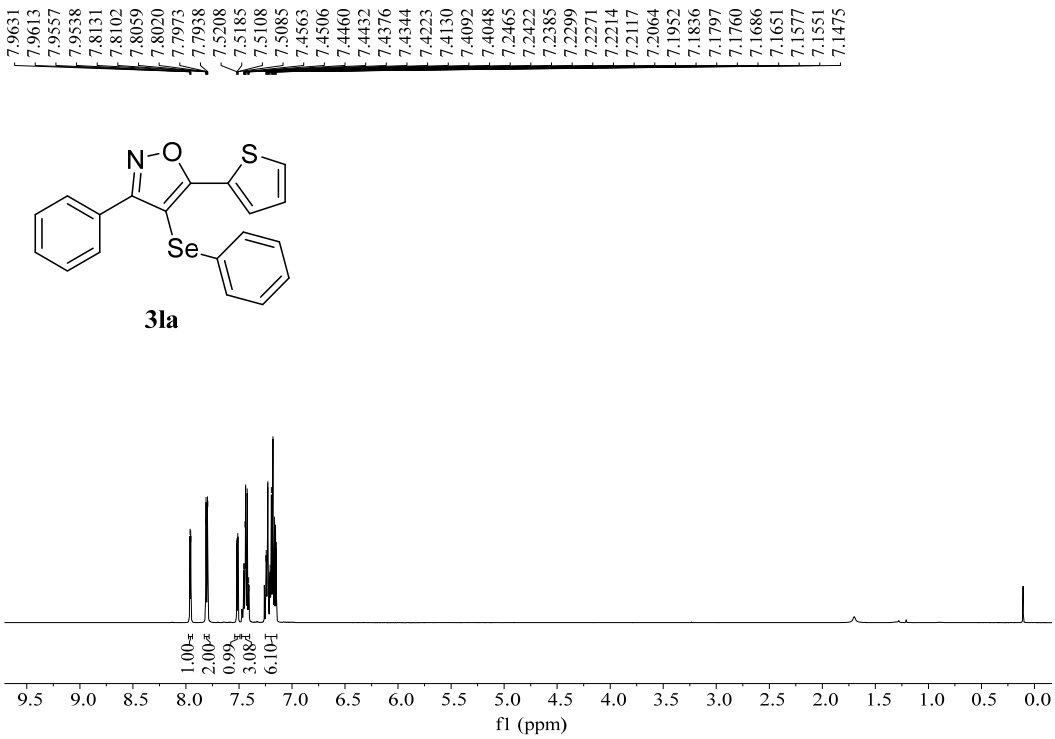


Figure S104 ^{1}H NMR (500 MHz) spectrum of **3la** in $CDCl_3$

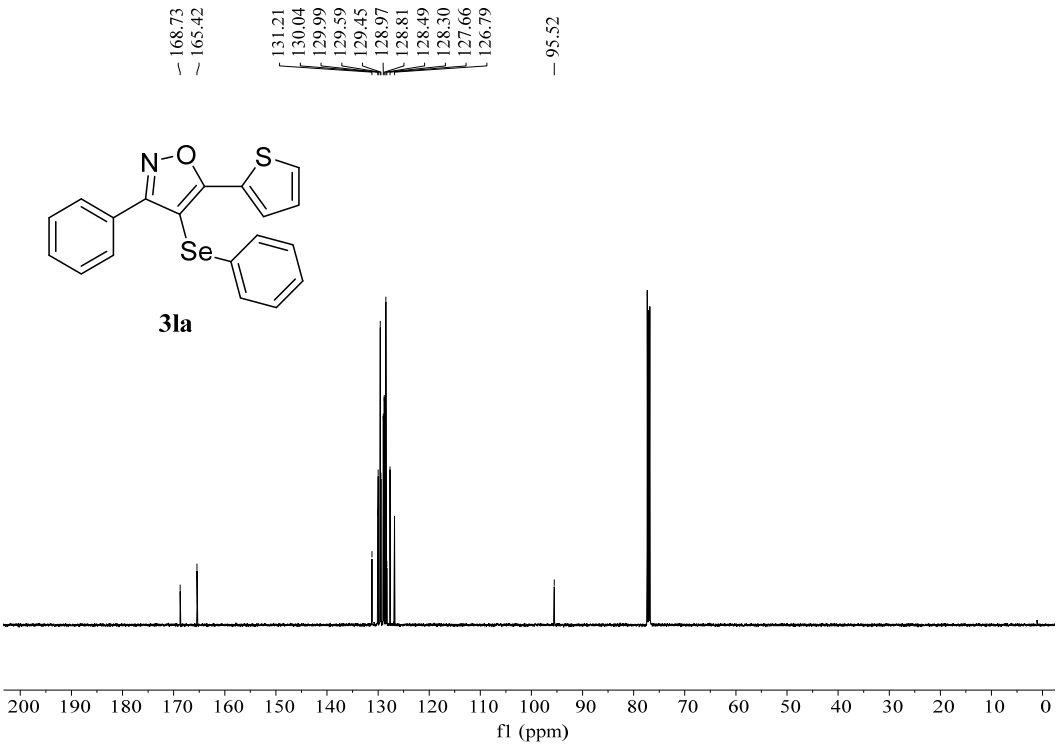


Figure S105 ^{13}C NMR (125 MHz) spectrum of **3la** in $CDCl_3$

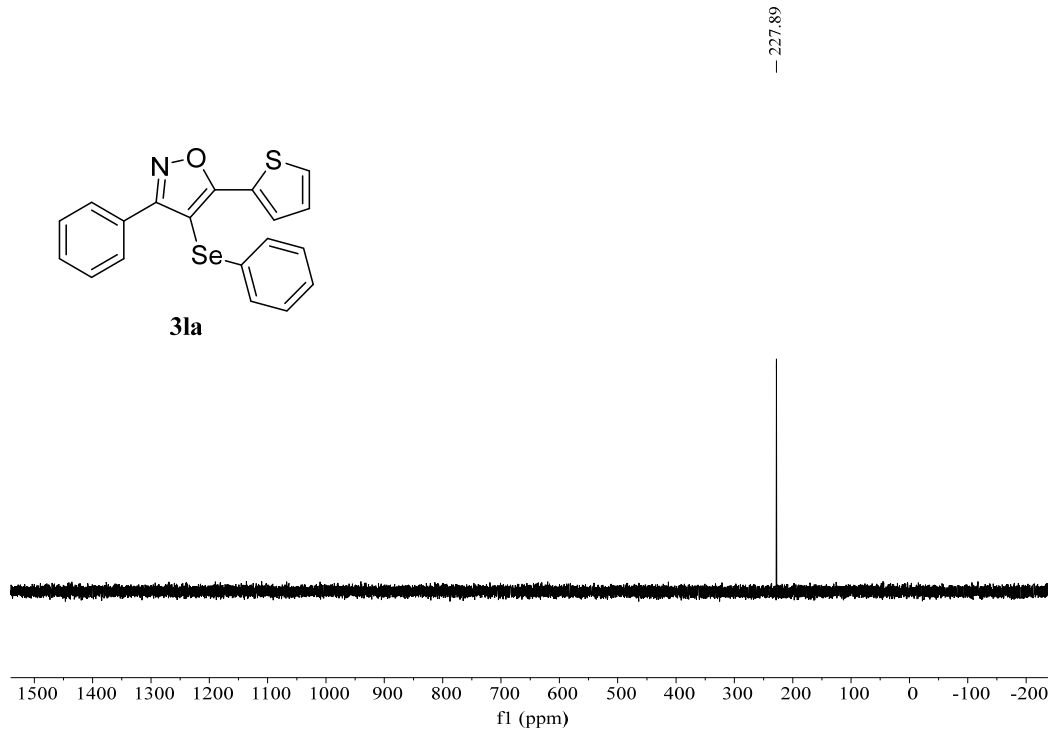


Figure S106 ^{77}Se NMR (95.5 MHz) spectrum of **3la** in $CDCl_3$

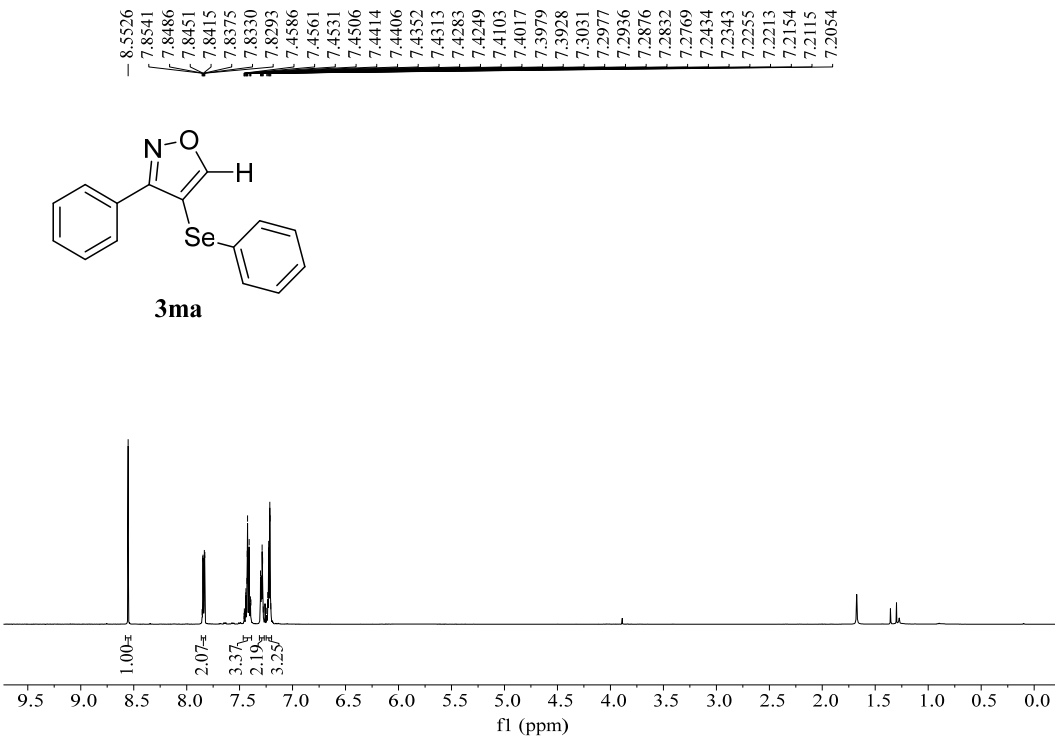


Figure S107 ^{1}H NMR (500 MHz) spectrum of **3ma** in $CDCl_3$

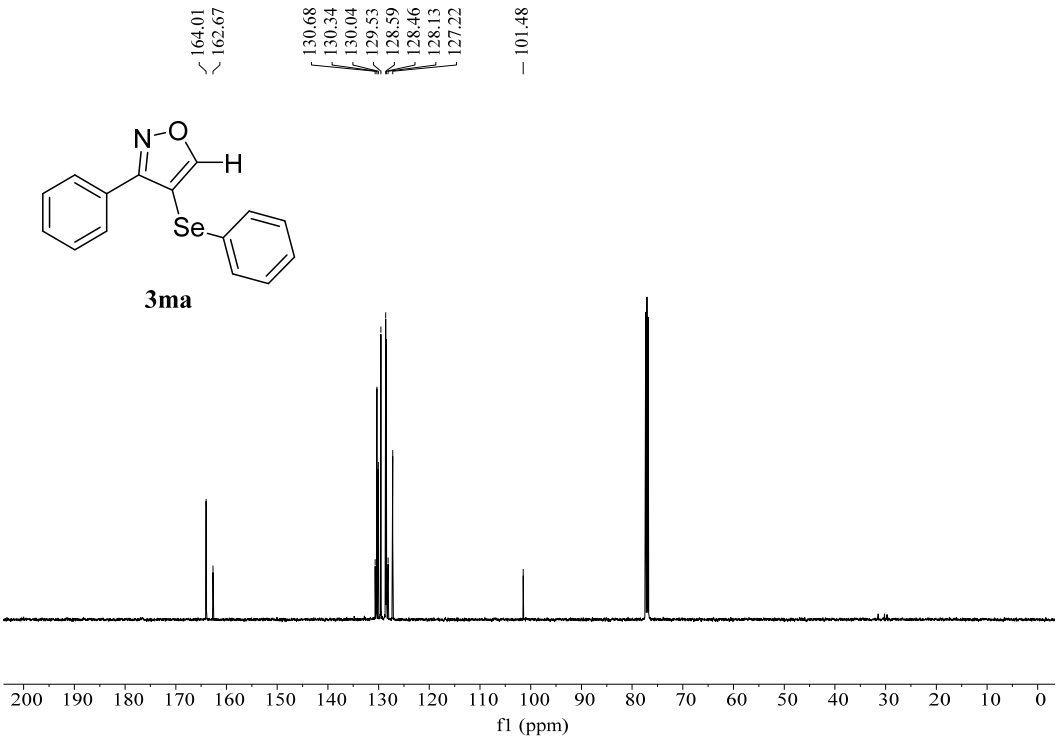


Figure S108 ^{13}C NMR (125 MHz) spectrum of **3ma** in $CDCl_3$

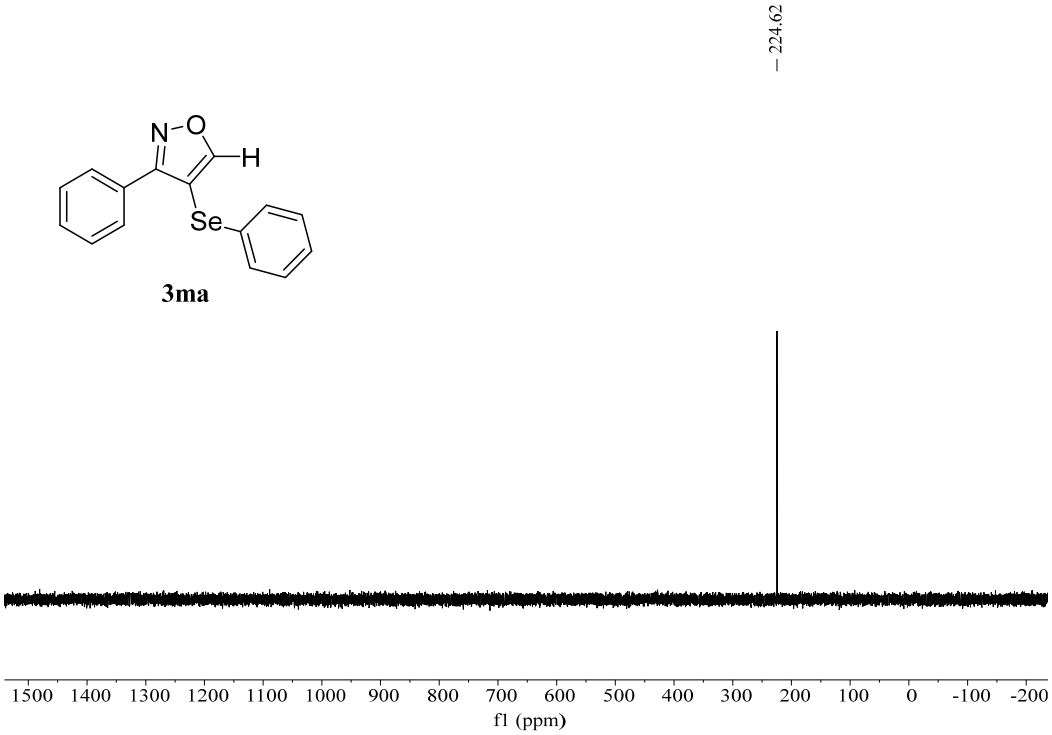


Figure S109 ^{77}Se NMR (95.5 MHz) spectrum of **3ma** in $CDCl_3$

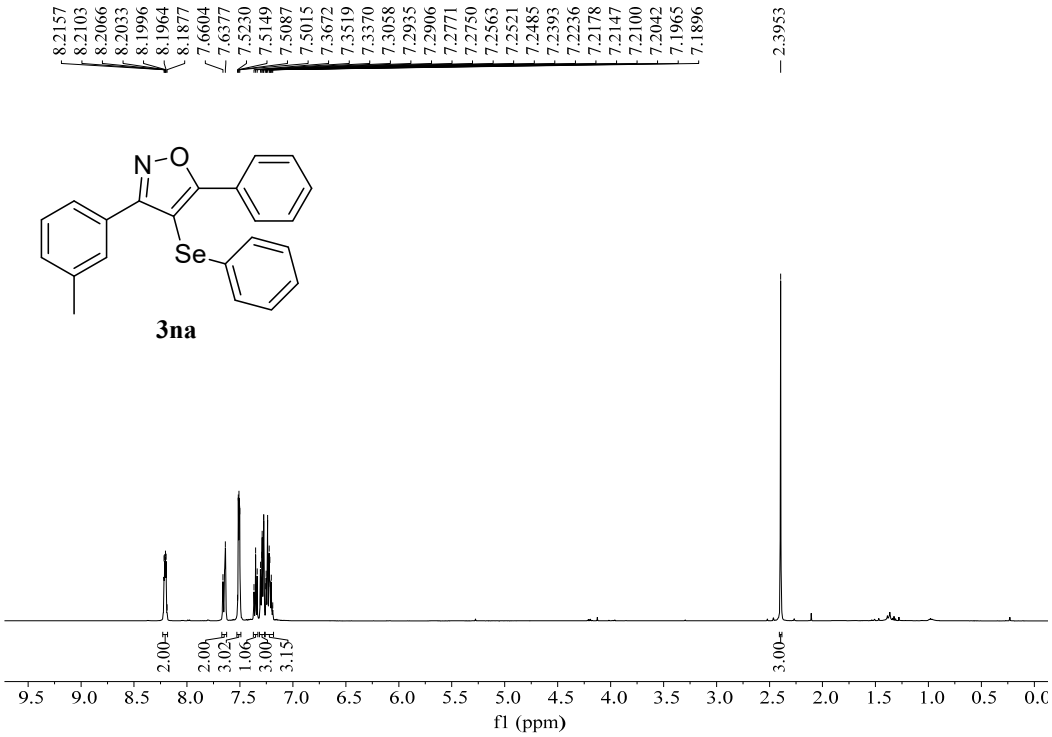


Figure S110 ^{1}H NMR (500 MHz) spectrum of **3na** in $CDCl_3$

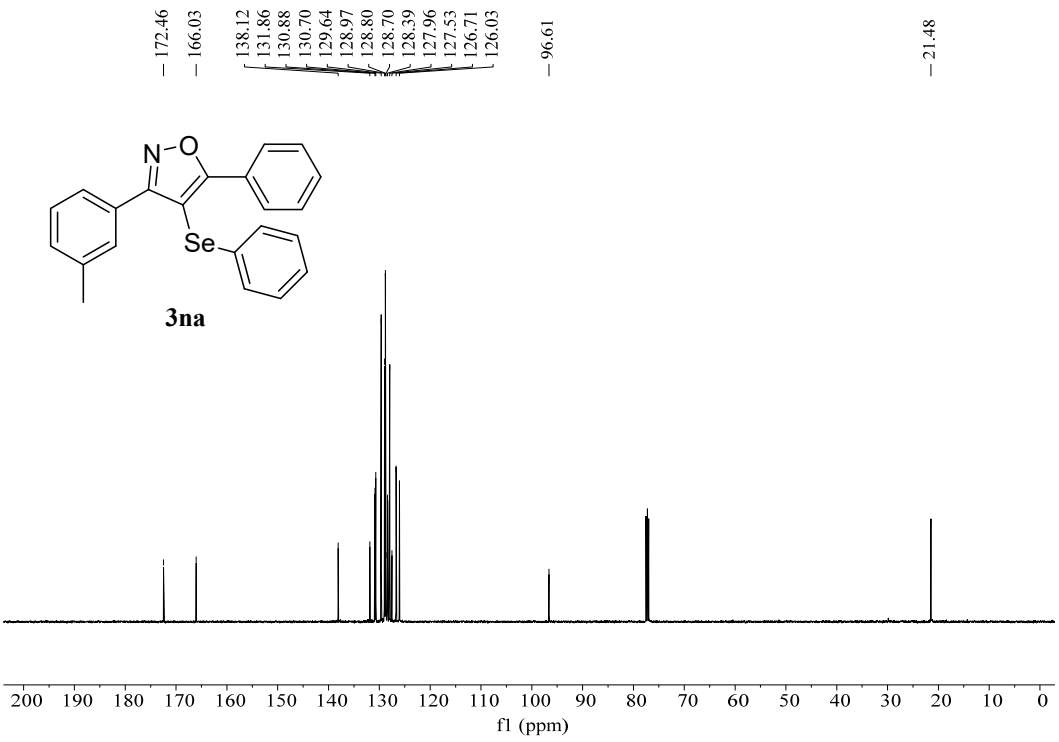


Figure S111 ^{13}C NMR (125 MHz) spectrum of **3na** in $CDCl_3$

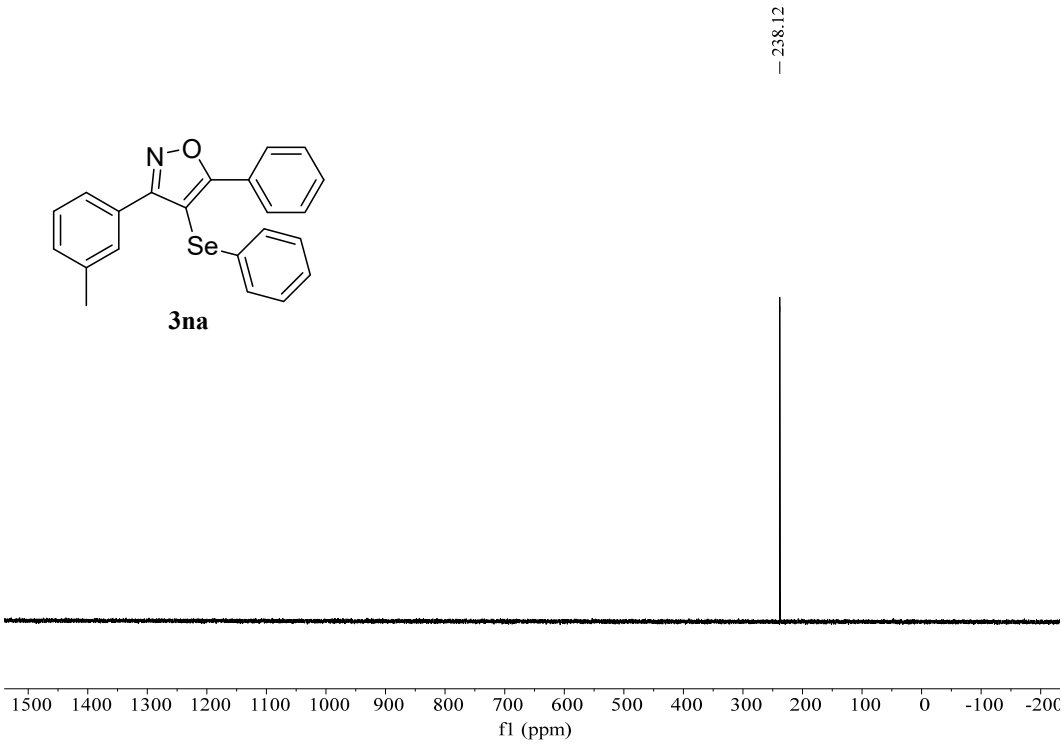


Figure S112 ^{77}Se NMR (95.5 MHz) spectrum of **3na** in $CDCl_3$

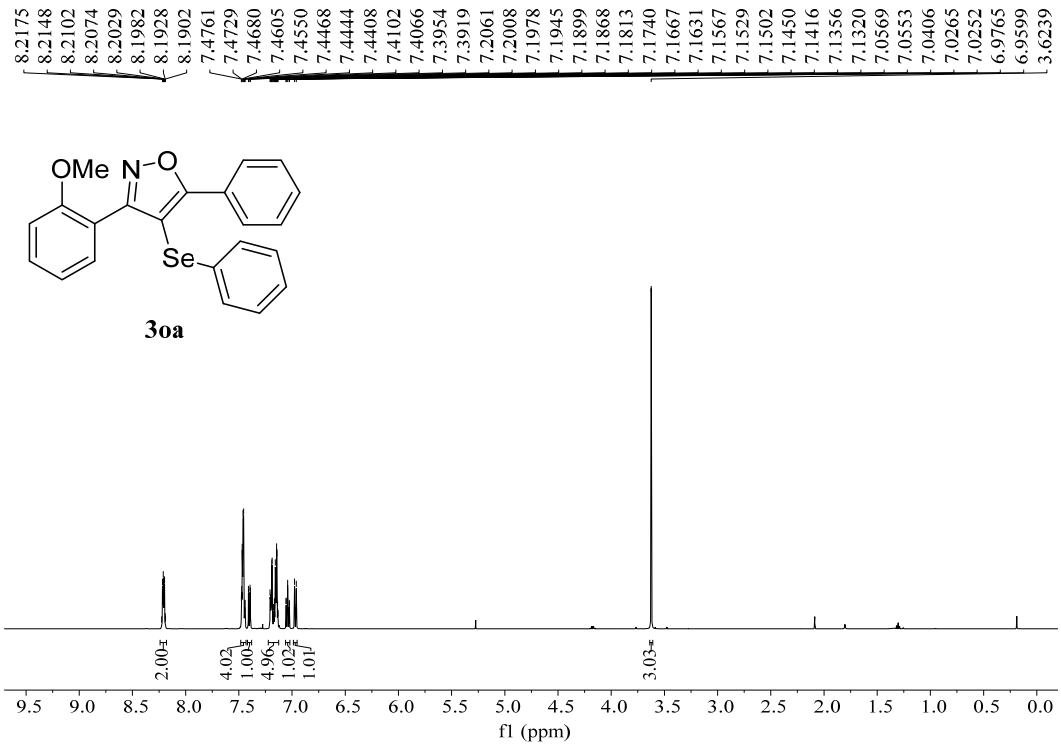


Figure S113 ^{1}H NMR (500 MHz) spectrum of **3oa** in $CDCl_3$

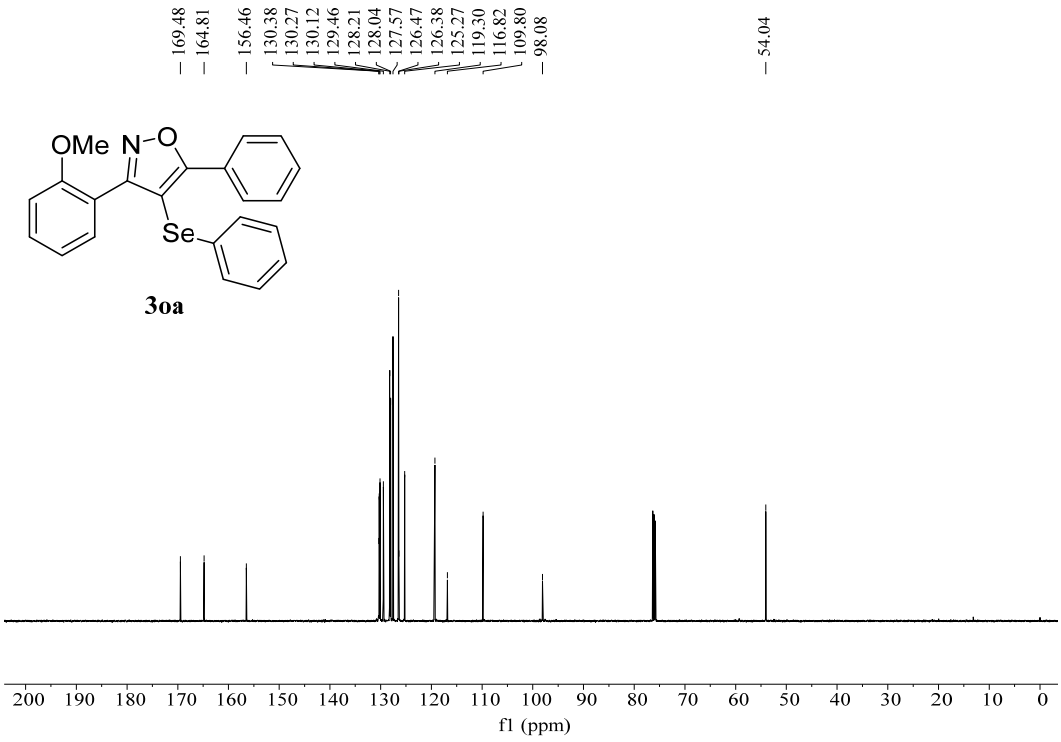


Figure S114 ^{13}C NMR (125 MHz) spectrum of **3oa** in $CDCl_3$

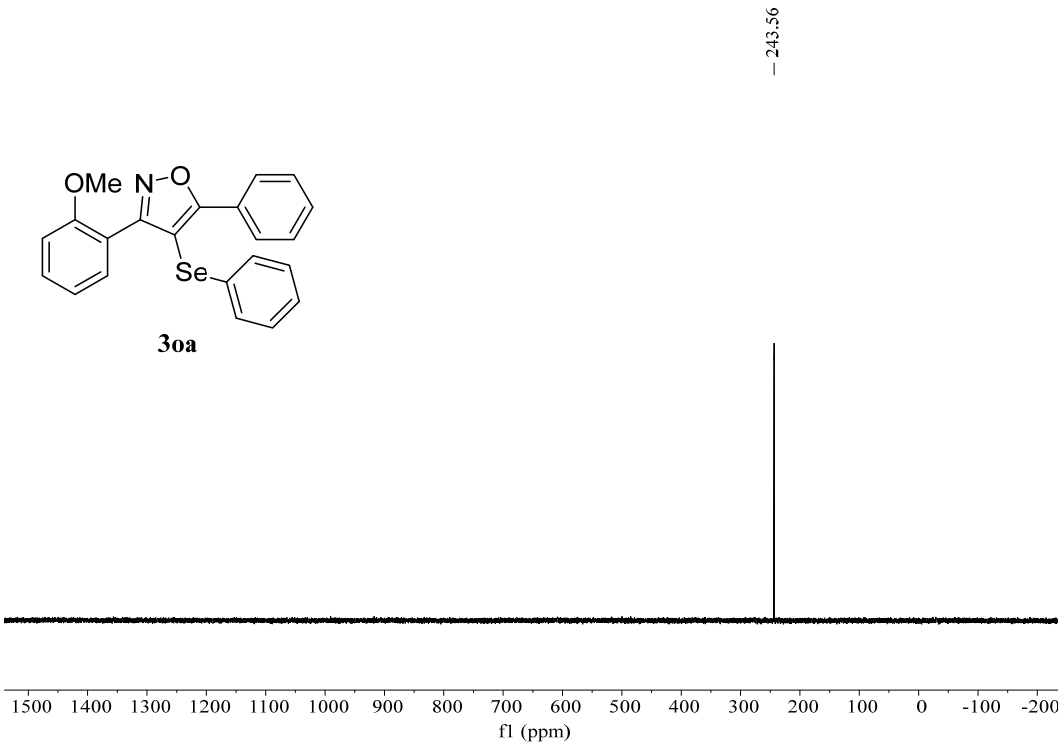


Figure S115 ^{77}Se NMR (95.5 MHz) spectrum of **3oa** in $CDCl_3$

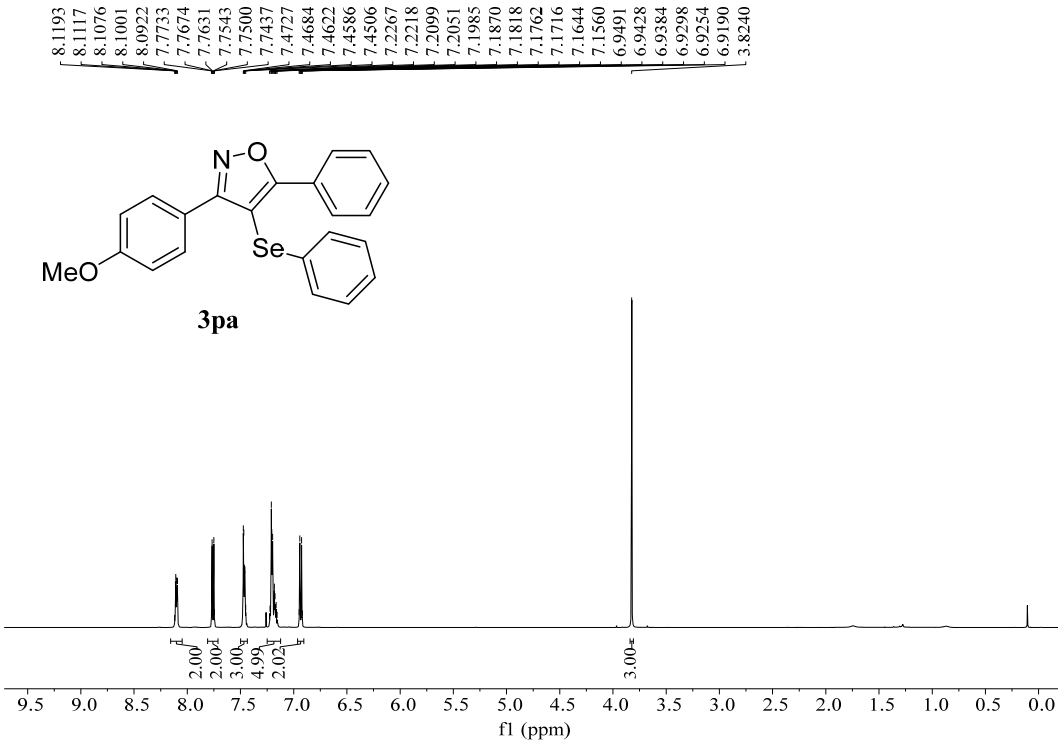


Figure S116 ^{1}H NMR (500 MHz) spectrum of **3pa** in $CDCl_3$

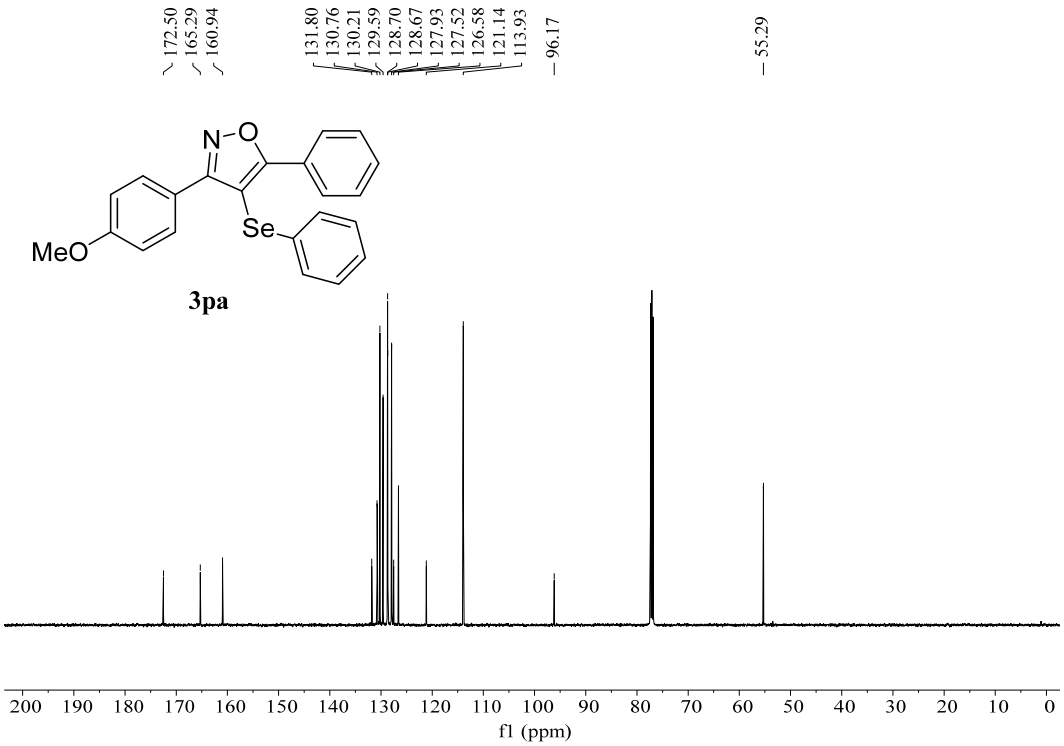


Figure S117 ^{13}C NMR (125 MHz) spectrum of **3pa** in $CDCl_3$

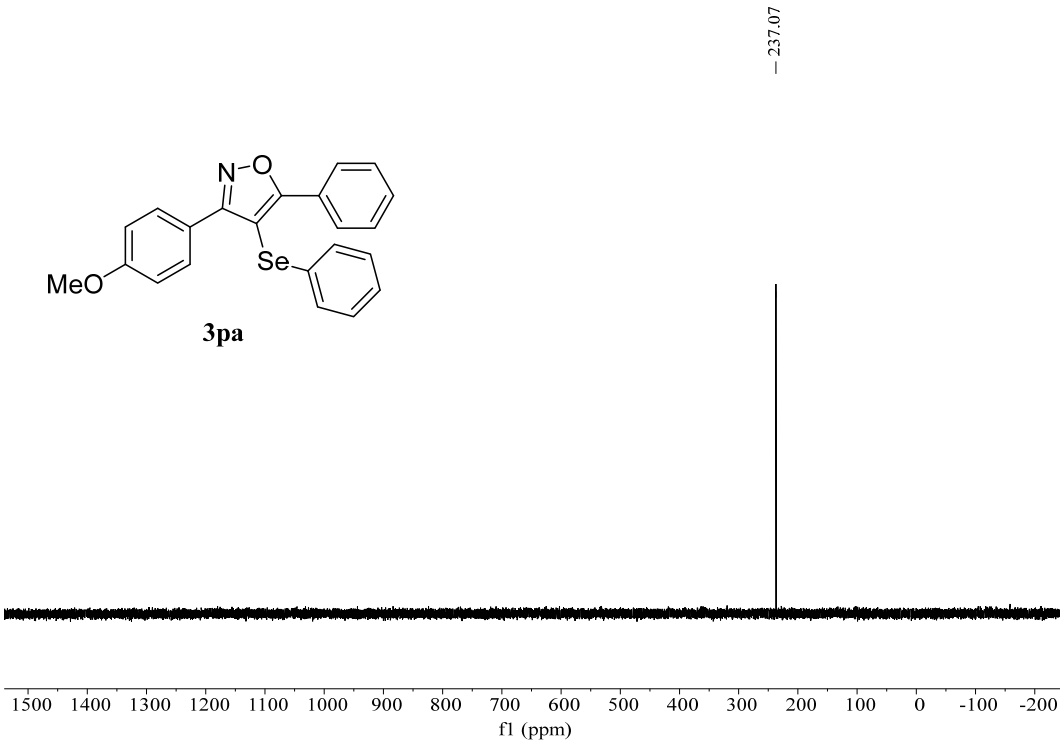


Figure S118 ^{77}Se NMR (95.5 MHz) spectrum of **3pa** in $CDCl_3$

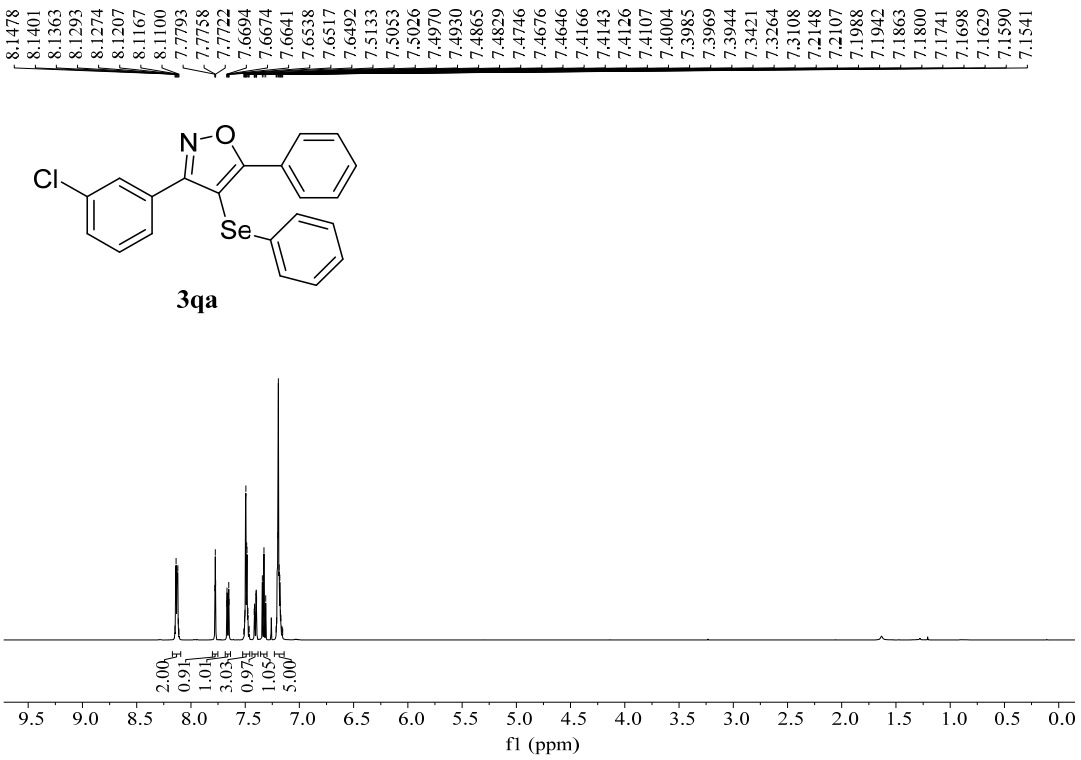


Figure S119 ^{1}H NMR (500 MHz) spectrum of **3qa** in $CDCl_3$

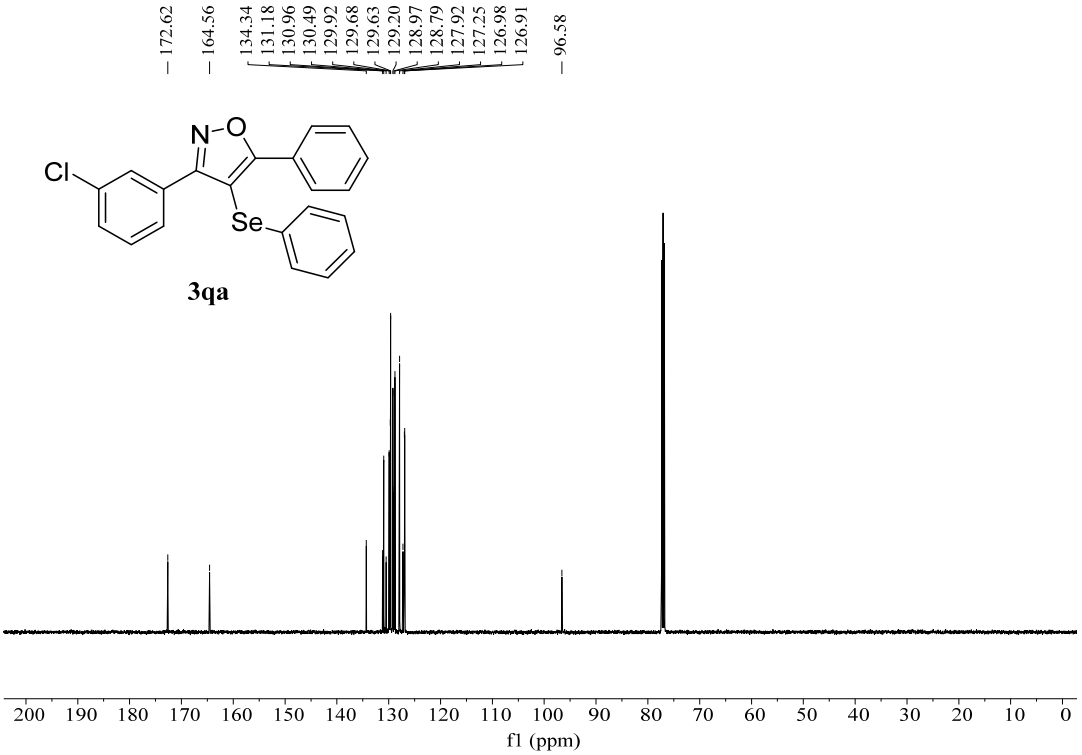


Figure S120 ^{13}C NMR (125 MHz) spectrum of **3qa** in $CDCl_3$

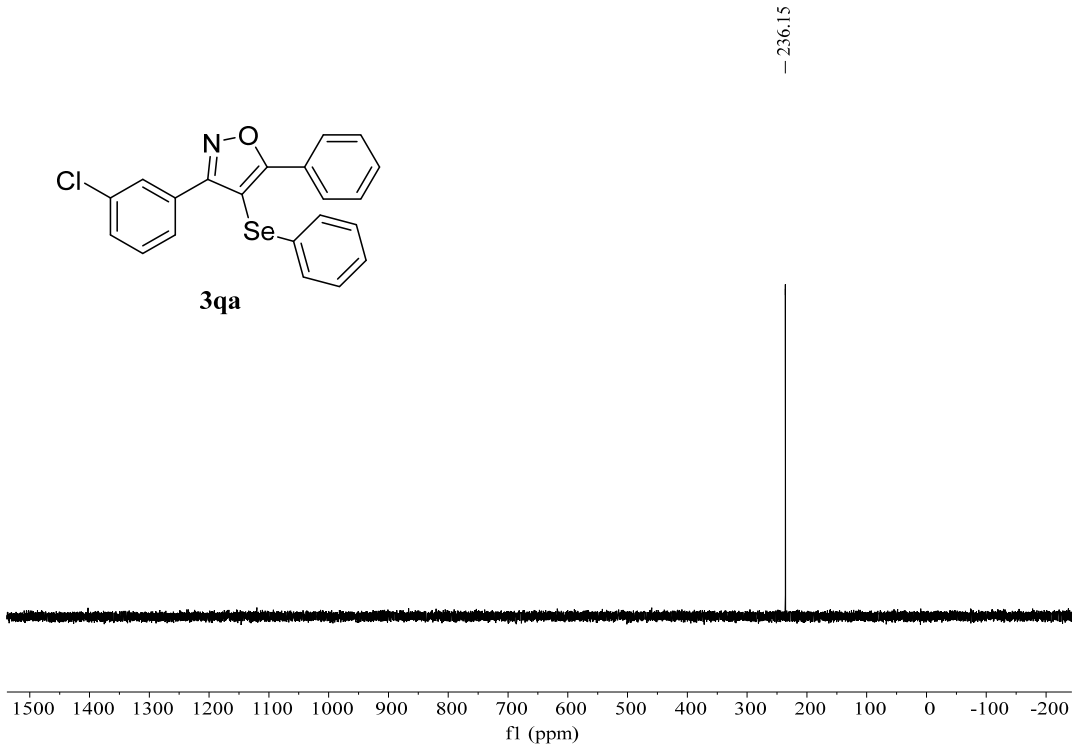


Figure S121 ^{77}Se NMR (95.5 MHz) spectrum of **3qa** in $CDCl_3$

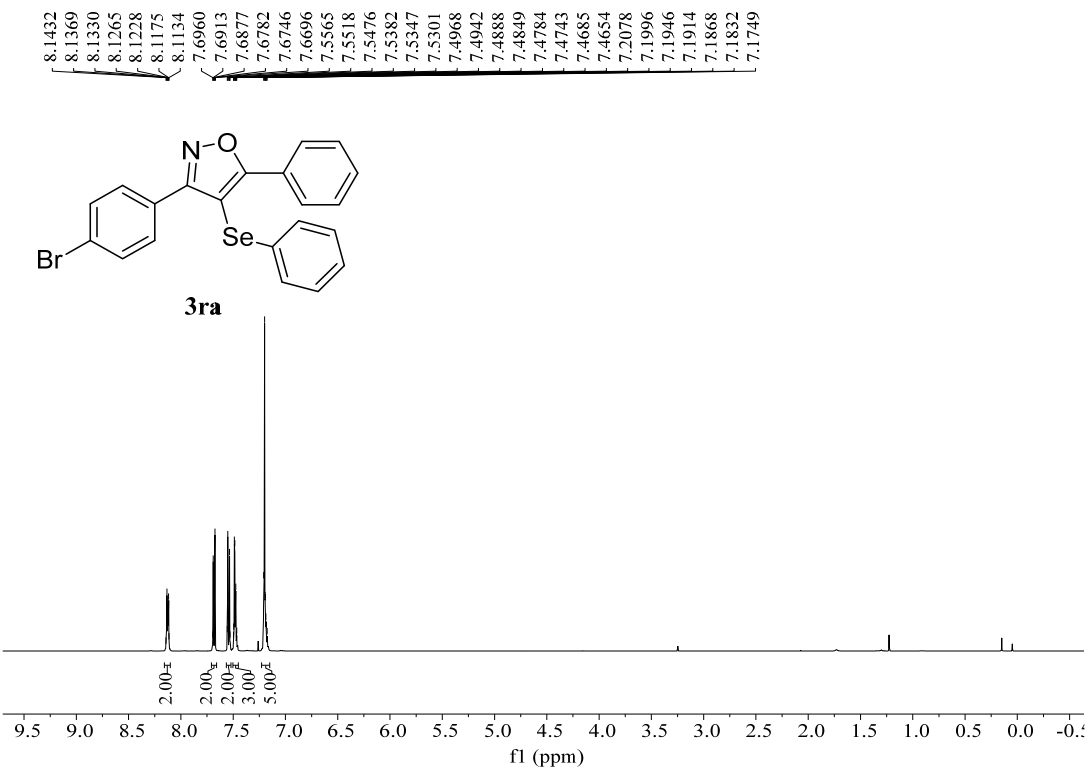


Figure S122 ^{1}H NMR (500 MHz) spectrum of **3ra** in $CDCl_3$

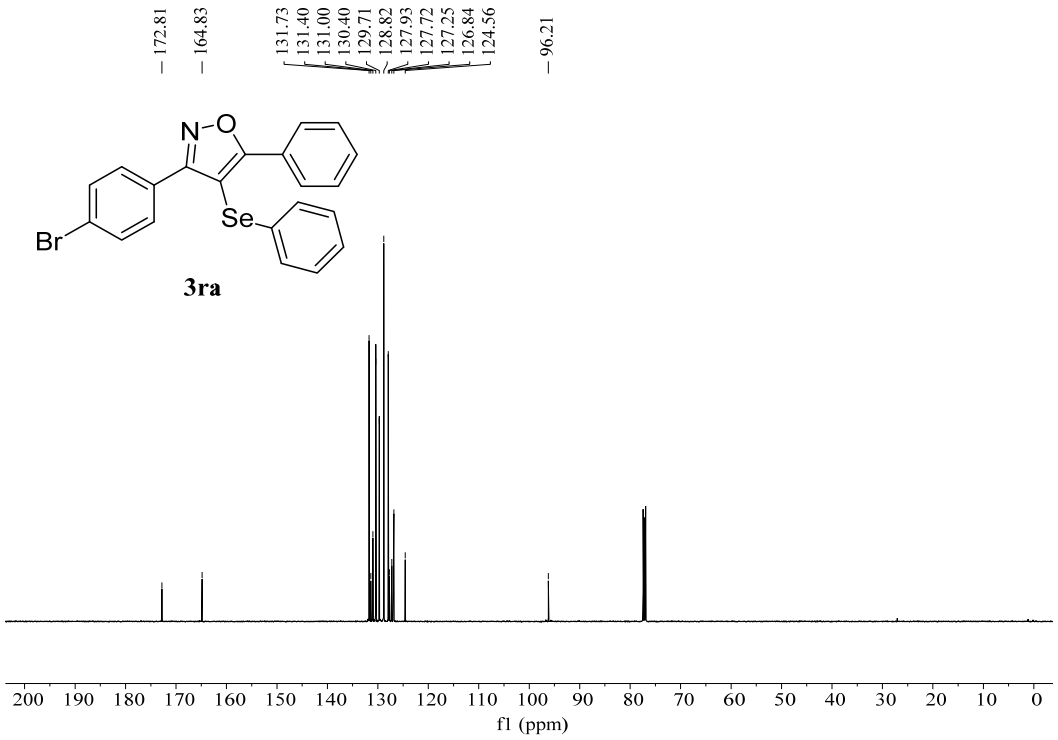


Figure S123 ^{13}C NMR (125 MHz) spectrum of **3ra** in $CDCl_3$

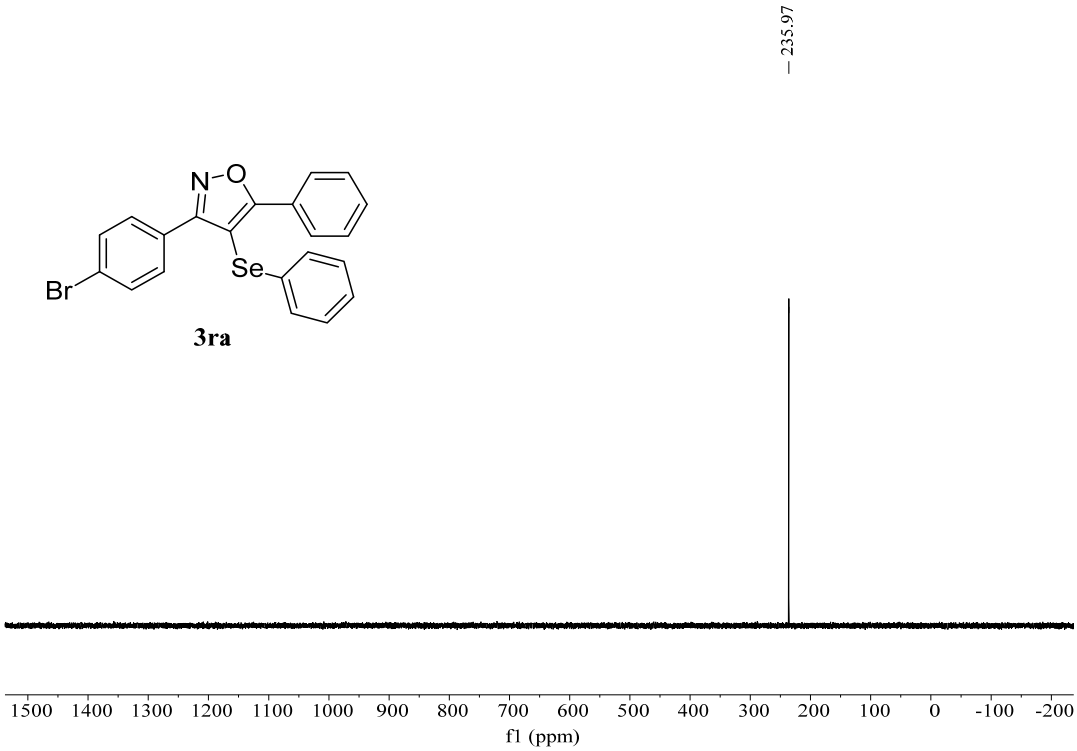


Figure S124 ^{77}Se NMR (95.5 MHz) spectrum of **3ra** in $CDCl_3$

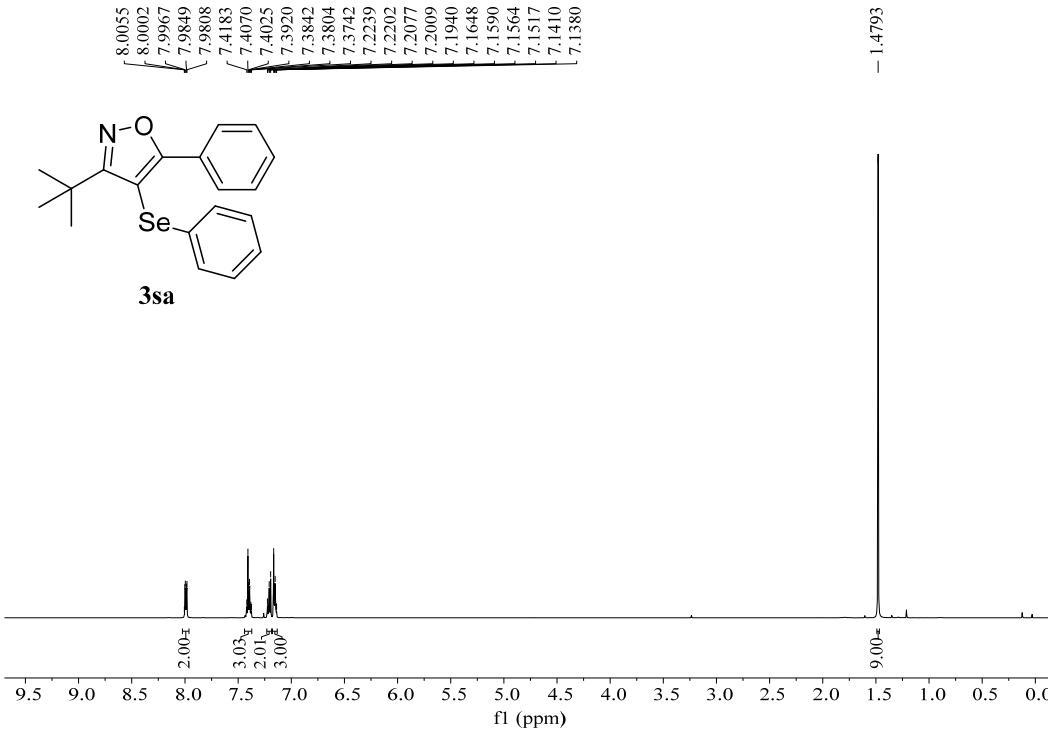


Figure S125 ^{1}H NMR (500 MHz) spectrum of **3sa** in $CDCl_3$

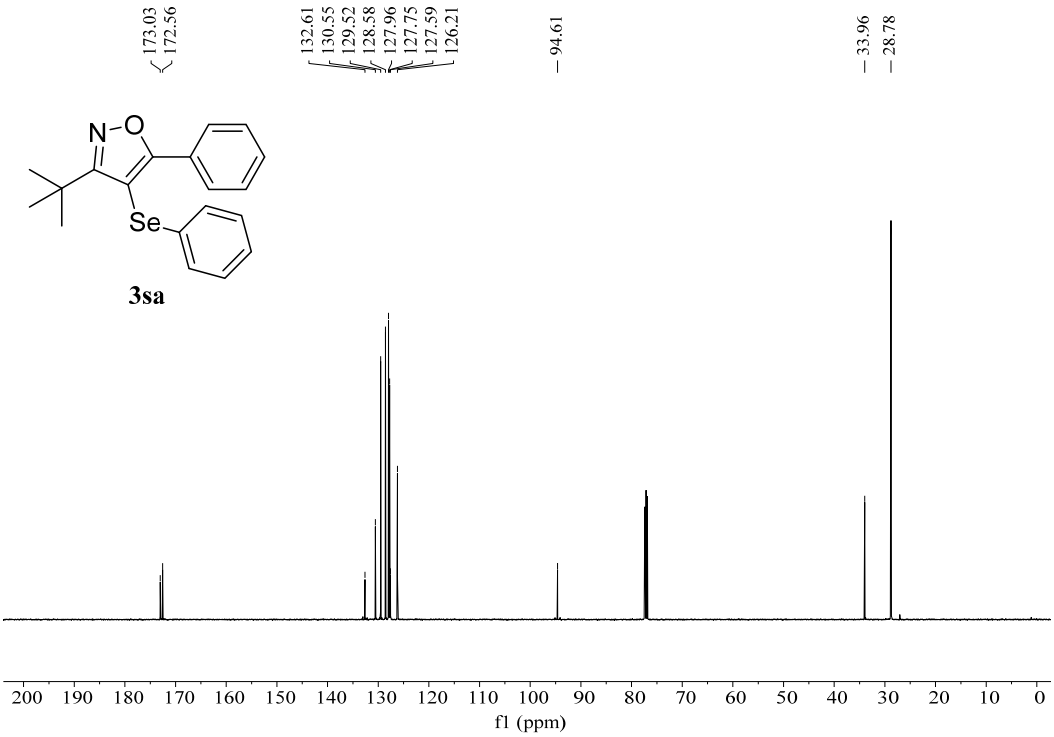


Figure S126 ^{13}C NMR (125 MHz) spectrum of **3sa** in $CDCl_3$

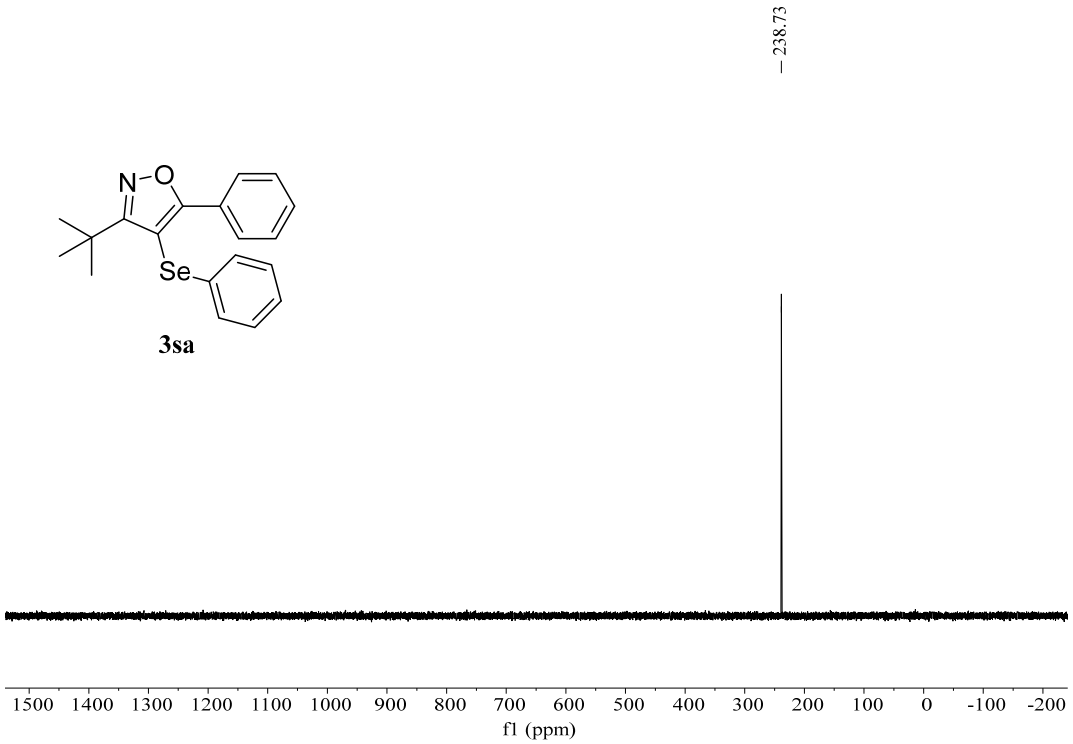


Figure S127 ^{77}Se NMR (95.5 MHz) spectrum of **3sa** in $CDCl_3$

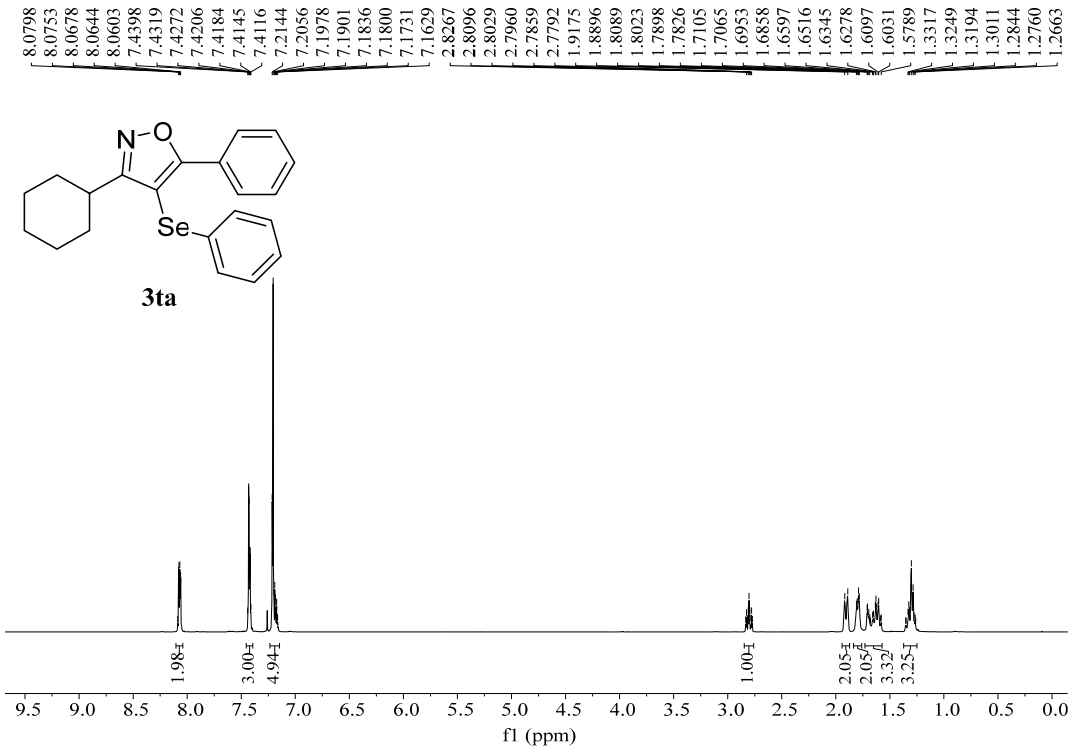


Figure S128 ^{1}H NMR (500 MHz) spectrum of **3ta** in $CDCl_3$

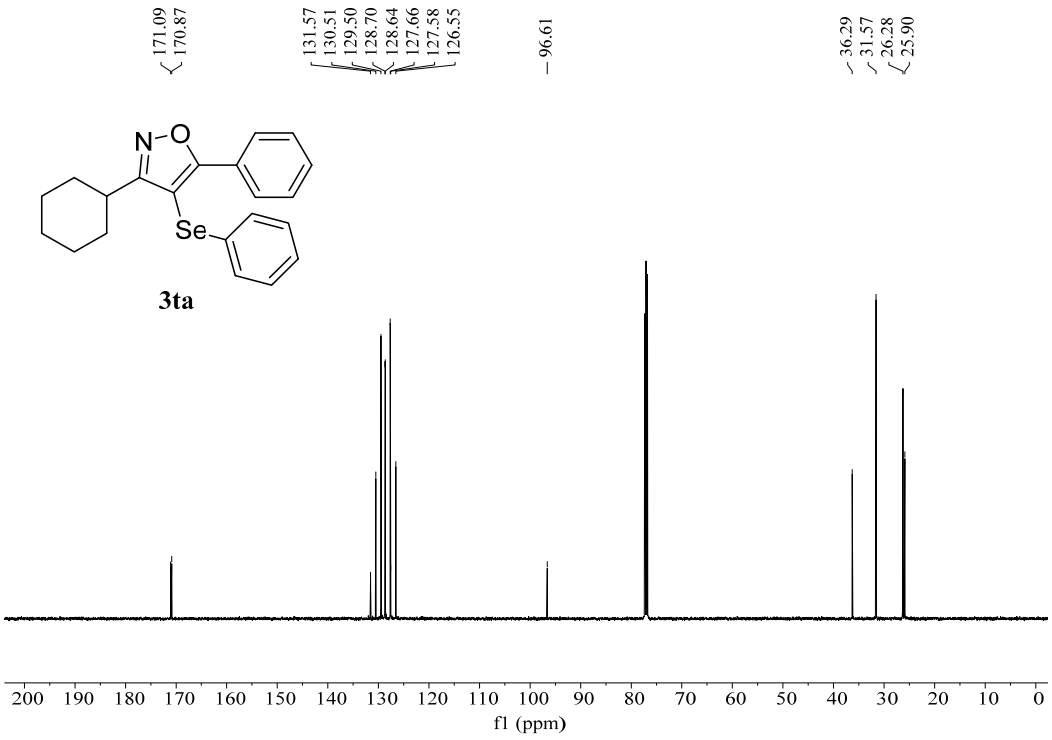


Figure S129 ^{13}C NMR (125 MHz) spectrum of **3ta** in $CDCl_3$

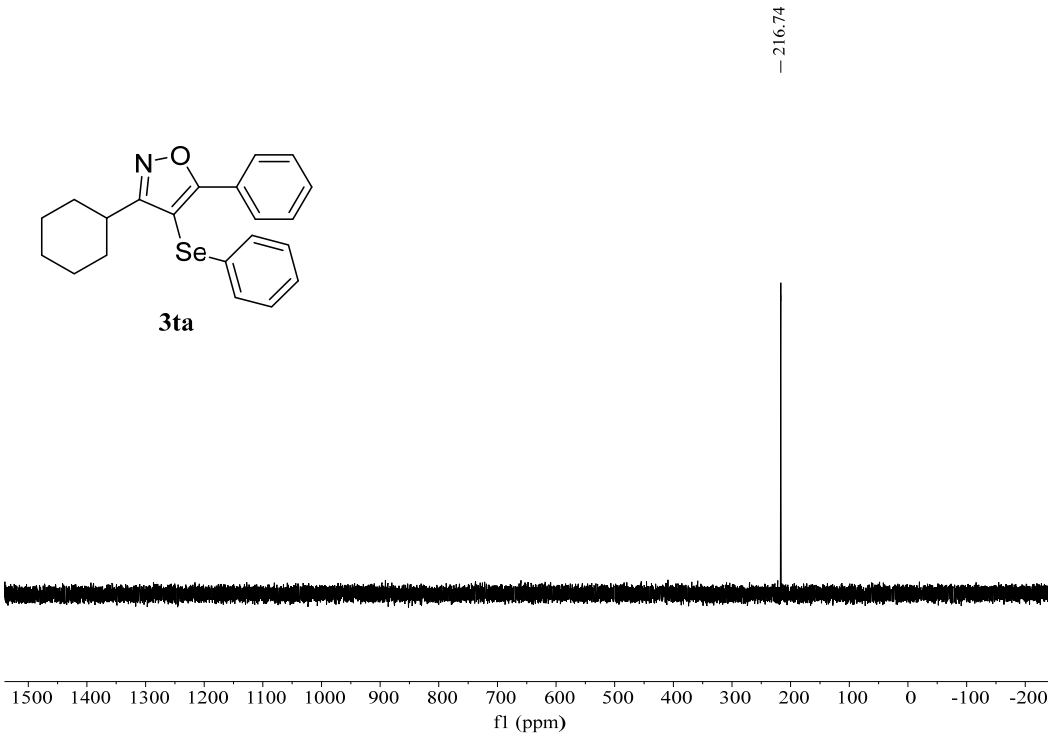


Figure S130 ^{77}Se NMR (95.5 MHz) spectrum of **3ta** in $CDCl_3$

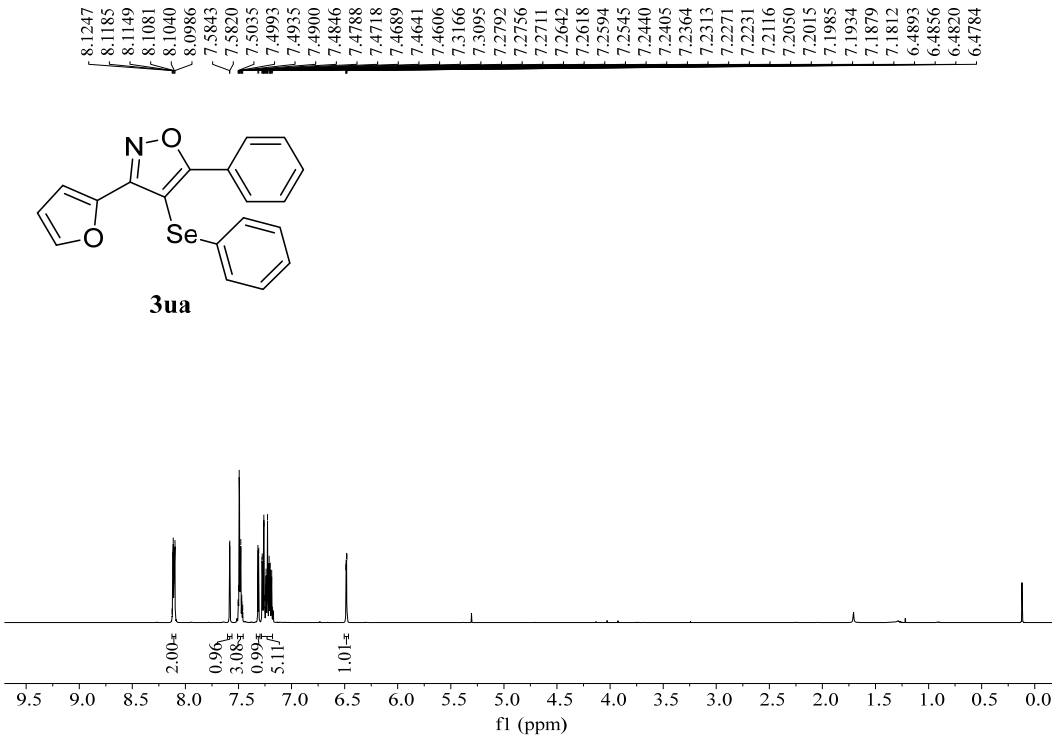


Figure S131 ^{1}H NMR (500 MHz) spectrum of **3ua** in $CDCl_3$

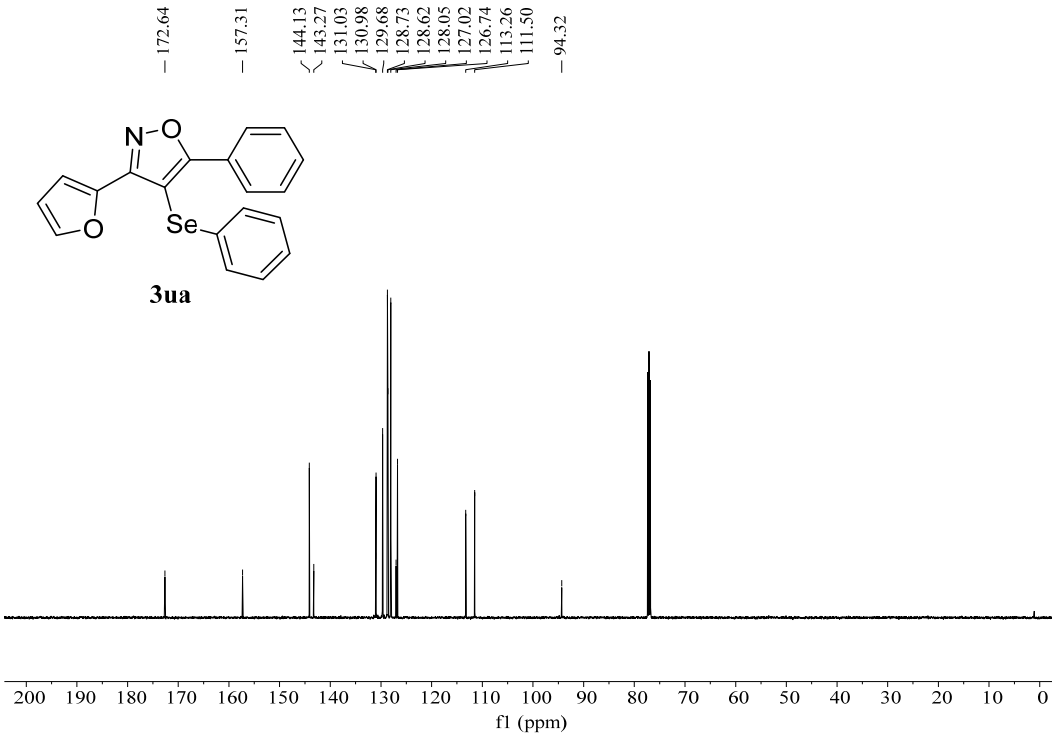


Figure S132 ^{13}C NMR (125 MHz) spectrum of **3ua** in $CDCl_3$

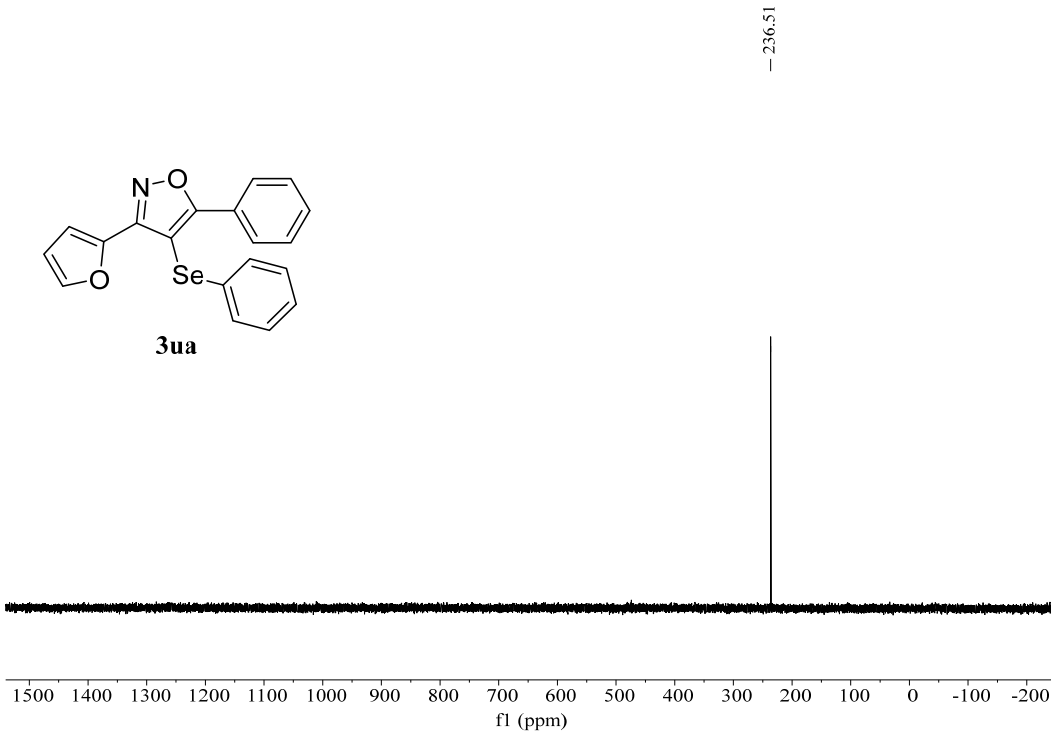


Figure S133 ^{77}Se NMR (95.5 MHz) spectrum of **3ua** in $CDCl_3$

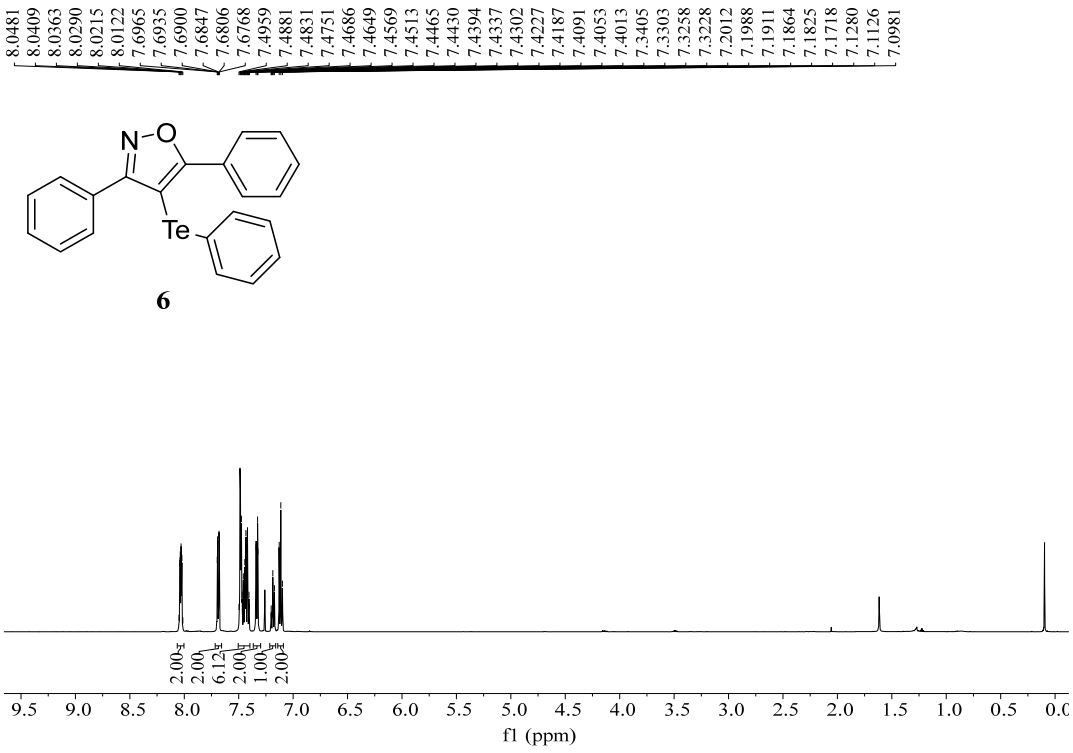


Figure S134 ^{1}H NMR (500 MHz) spectrum of **6** in $CDCl_3$

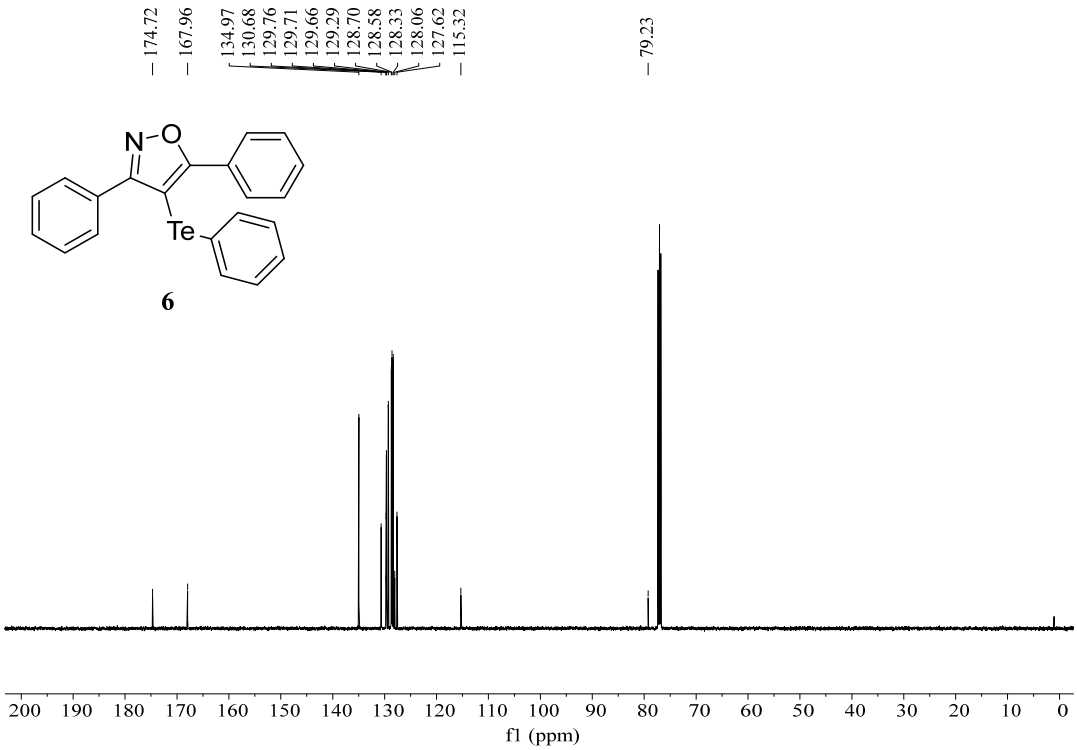


Figure S135 ^{13}C NMR (125 MHz) spectrum of **6** in $CDCl_3$