Electronic Supplementary Material (ESI) for ChemComm.
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An Electrochemical Cascade Process: Synthesis of 3-Selenylindoles From 2-Alkynylanilines with Diselenides

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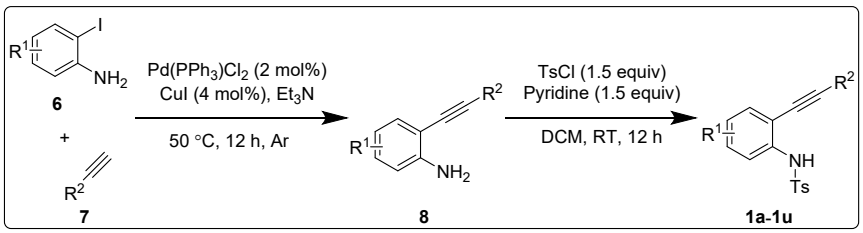
Supporting Information

Experimental:

General methods:

IR spectra were recorded on a Bruker Tensor 37 (FTIR) spectrophotometer. ^{1}H NMR spectra were recorded on Bruker Advance 400 (400 MHz) and 600 (600 MHz) spectrometers at 295 K in $CDCl_3$; chemical shifts (δ ppm) and coupling constants (Hz) are reported in standard fashion concerning either internal standard tetramethylsilane (TMS) (δ_H = 0.00 ppm) or $CDCl_3$ (δ_H = 7.26 ppm). $^{13}C\{^{1}H\}$ NMR spectra were recorded on Bruker Advance 400 (101 MHz) spectrometers at RT in $CDCl_3$. Chemical shifts (δ ppm) are reported relative to $CDCl_3$ [δ = 77.16 ppm (central line of the triplet)]. In the $^{13}C\{^{1}H\}$ NMR, the nature of carbons (C, CH, CH_2, and CH_3) was determined by recording the DEPT-135 spectra and is given in parentheses and noted as s = singlet (for C), d = doublet (for CH), t = triplet (for CH_2) and q = quartet (for CH_3). In the ^{1}H-NMR, the following abbreviations were used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, sept = septet, dd = doublet of doublet, m = multiplet and br. s = broad singlet. The assignment of signals was confirmed by ^{1}H, $^{13}C\{^{1}H\}$ CPD, and DEPT spectra. High-resolution mass spectra (HRMS) were recorded on an Agilent 6538 UHD Q-TOF electron spray ionization (ESI) mode and atmospheric pressure chemical ionization (APCI) modes. Melting points are recorded using Tempo and Mettler FP1 melting point apparatus in capillary tubes. A single crystal of **3aa** and **3ab** was selected and mounted on an Oxford SuperNova, Dual, Cu at zero, Eos diffractometer. The crystal was kept at 298 K during data collection. Using Olex2, the structure was solved with Olex2. Solve structure solution program using direct methods and refined with the olex2. refinement package using Gauss−Newton minimization. Electrolysis reactions were conducted using ElectraSyn 2.0 Package supply purchased from IKA Instruments. Reactions were monitored by TLC on silica gel using a combination of hexane and ethyl acetate as eluents. Solvents were distilled before use; petroleum ether in the boiling range of 60−80 °C was used. Cyclic voltammetry (CV) analysis was performed on ElectraSyn 2.0 Package, using a glassy carbon electrode as the working electrode, a platinum electrode as a counter electrode and Ag/AgCl electrode as a reference electrode. A cyclic voltammogram was recorded at a 100 mV/s scan rate. The starting materials terminal acetylenes, 2-iodo anilines, aryl sulfonyl chlorides, and $LiClO_4$ were purchased from Sigma/TCI/ local sources and used as received. Acme's silica gel (60–120 mesh) was used for column chromatography (approximately 20 g per gram of crude material).

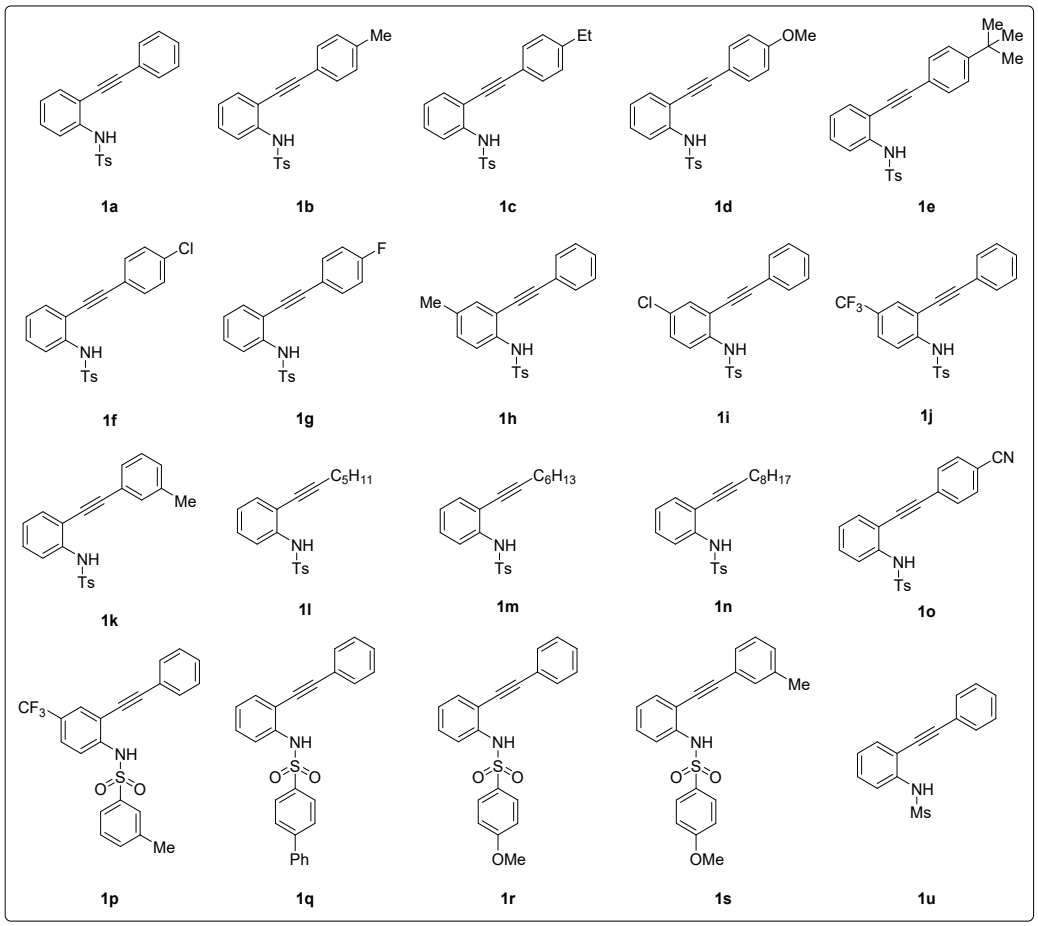
General Procedure - 1 (GP-1) for the Preparation of *N*-Protected 2-Alkynylbenzenamine (1a-1u):



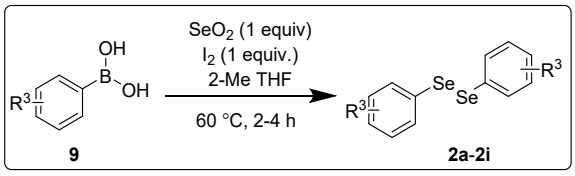
To an oven-dried Schlenk tube equipped with a magnetic stir bar, were added 2-iodoanilines **6** (500 mg, 2.28 mmol), $PdCl_2(PPh_3)_2$ (2 mol %), and CuI (4 mol %) in Et_3N (5 mL) and the mixture was allowed to stir at room temperature for 10 min. Then terminal acetylene **7** was added (280 mg, 1.2 equiv) under an argon atmosphere, and the reaction was performed at room temperature for 12 h. Upon completion, the mixture was poured into an aqueous NH_4Cl solution (40 mL) and extracted with ethyl acetate (3 × 10 mL) and the combined organic phase was washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished **8**.

The above-collected residue **8** (1.0 equiv) in CH_2Cl_2 (10 mL) was taken in an oven-dried round-bottomed flask equipped with a magnetic stir bar, then the TsCl (1.5 equiv) and pyridine (1.5 equiv) were added in it. The resulting mixture was stirred at room temperature for 12 h. The mixture was poured into an aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 95:05) as the eluent furnished the desired product **1a-1u** (78 to 90%), as yellow liquids or yellow solids (Table S1).

Table-S1. The following *N*-substituted 2-(arylethynyl)anilines **1a-1u** are prepared by using the literature reports.[1,2]

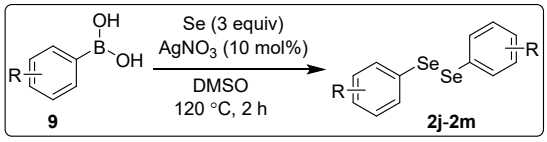


General Procedure - 2 (GP-2) for the Preparation of Diaryl Diselenides (2a-2i):



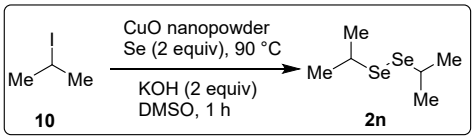
The reaction was carried out in a 25 mL oven-dried round bottom flask equipped with magnetic stir bar and charged with aryl boronic acid **9** (1.0 equiv), selenium dioxide (1.0 equiv), I_2 (1.0 equiv), and 2-Me-THF (5 mL). The resulting reaction mixture was stirred at 60 °C for 2–4 h. The reaction progress was monitored by TLC. After completion of the reaction, iodine was quenched with hypo solution, and it was worked up with ethyl acetate (3 × 10 mL) and the combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 100:00–95:05) as the eluent furnished the desired product **2a-2i** (70 to 80%), as yellow liquids or yellow solids (or red solids) (Table S2).

General Procedure - 3 (GP-3) for the Preparation of Diaryl Diselenides (2j-2m)



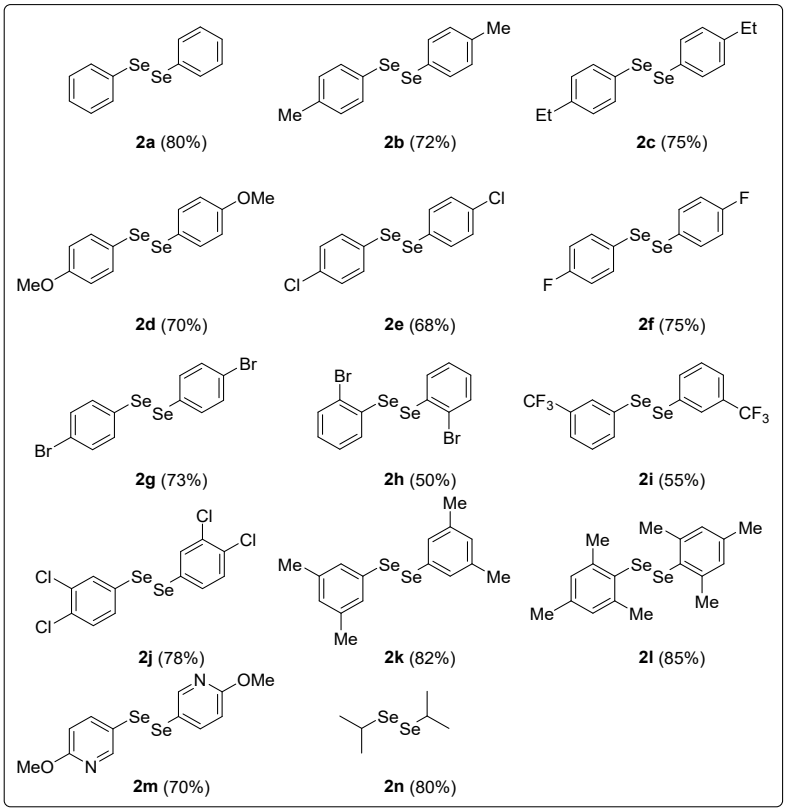
The reaction was carried out in a 25 mL oven-dried round bottom flask equipped with magnetic stir bar and charged with arylboronic acid (1 equiv), selenium (3 equiv), AgNO3 (10 mol%), and DMSO (2.0 mL). The mixture was stirred in a heating mantle preheated to 120 °C for 2 h. The reaction progress was monitored by TLC. After cooling to room temperature, the reaction mixture was diluted with H_2O (10 mL) and extracted with EtOAc (3×10 mL). The combined organic phase was washed with water and brine (30mL), dried over anhydrous Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 100:00–95:05) as the eluent furnished the desired product **2j-2m** (70 to 85%), as yellow liquids or yellow solids (Table S2).

General Procedure - 4 (GP-4) for the Preparation of Diaryl Diselenides (2n)

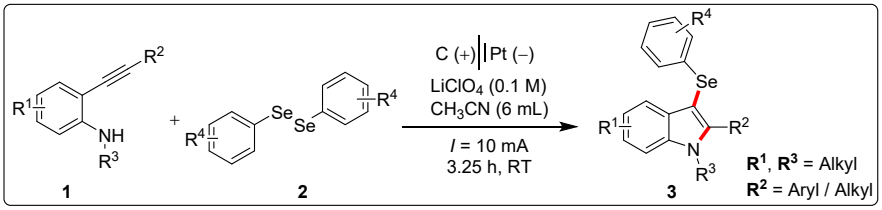


The reaction was carried out in a 25 mL oven-dried round bottom flask equipped with magnetic stir bar and charged with **10** (1.0 equiv), CuO nanopowder (10 mol%), selenium powder (2.0 equiv), KOH (2.0 equiv), and DMSO (2 mL). The resulting reaction mixture was stirred at 90 °C for 1 h. The reaction progress was monitored by TLC. After completion of the reaction, it was worked up with ethyl acetate (3 × 10 mL) and the combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate 100:00) as the eluent furnished the desired product **2n** in 75% of isolated yield, as yellow liquids (Table S2).

Table-S2. The following starting material diaryl diselenide **2a-2n** are prepared by using the literature.[3–5]



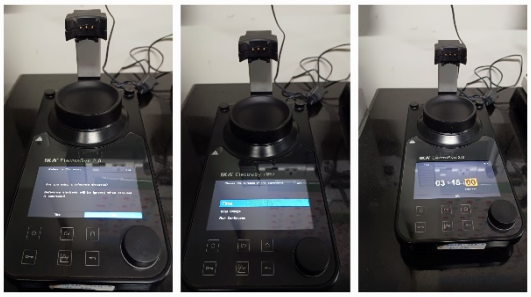
General Procedure for the Preparation of 3-selenylindole 5 (GP-5):



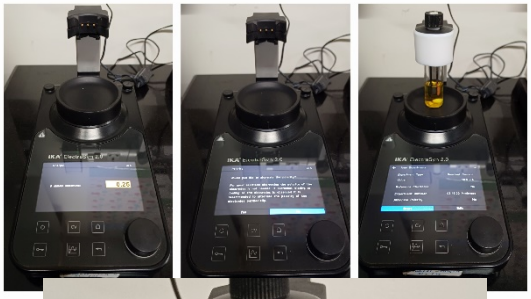
The reaction was carried out with *N*-substituted 2-(arylethynyl)anilines **1** (0.25 mmol), diaryl diselenide **2** (0.25 mmol, 1 equiv), $LiClO_4$ (0.1 M) in 6 mL CH_3CN was added to an oven dried ElectraSyn vial (10 mL) with a magnetic stirring bar. The ElectraSyn vial cap equipped with the anode (Graphite) and cathode (Platinum) was inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 10 mA for 3 h 15 min. After electrolysis, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (2 mL), and the mixture was poured into an aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the desired product 3-selenylindole **3** (70 to 95% yields) as a brown liquids or brown solids.

Photographic guide for electrochemical reaction:

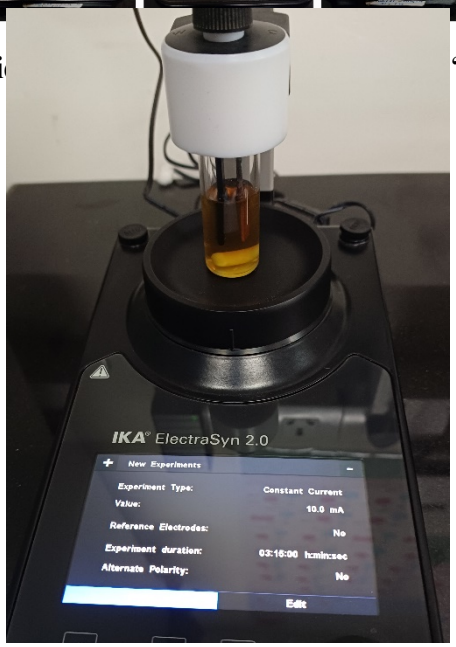
Left: Select New “Experiment” **Middle:** Select “Constant Current” **Right:** Select “10 mA”



Left: Reference electrode chose “No” **Middle:** Select “Time” **Right:** Select “3 h 15 min”



Left: Select “0.25 mmol” **Mi** “No” **Right:** Select “Start the experiment”



‘zoomed’ photo of the experiment.

Cyclic Voltammograms:

The cyclic voltammetry (CV) studies were carried out to further investigate the reaction mechanism, and below Figure 1S shows the cyclic voltammetry (CV) curves with 0.1 M $LiClO_4$ solution in CH_3CN as a background. The voltammogram was obtained at a scan rate of 100 mV/s with Pt wire as a counter electrode, Ag/AgCl as a reference electrode which is submerged in saturated aqueous KCl solution, and Pt disk electrode as a working electrode. Within the scanning window (0 to 2.5 V). An obvious there is no peak for background (Curve 1). The CV of 4-methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **1a** displayed oxidation peak at 1.72 V *vs* Ag/AgCl (curve 2). When testing diphenyl diselenide **2a**, an oxidation peak was seemed at 1.41 V *vs* Ag/AgCl in curve 3. The mixture of 4-methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide (**1a**) and diphenyl diselenide (**2a**) showed two oxidation signals at 1.71 V and 1.40 V *vs* Ag/AgCl (curve 4), respectively.

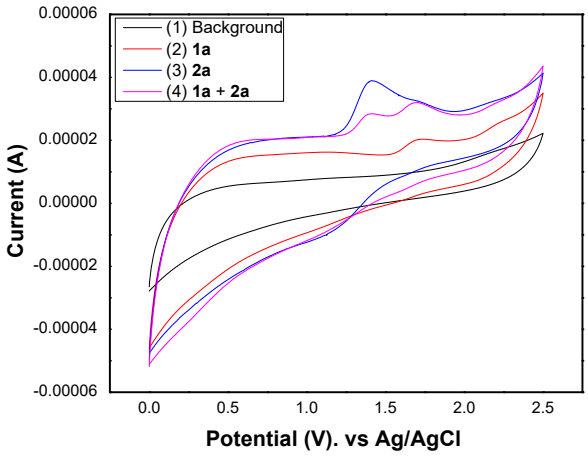


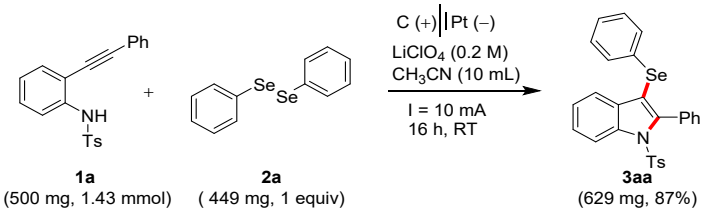
Figure S1. Cyclic voltammograms of reactants and their mixtures in 0.1 M $LiClO_4$ solution in CH_3CN at room temperature: a) Background; b) 4-methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **1a** (0.01 M); c) diphenyl diselenide **2a** (0.01 M); d) 4-methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **1a** (0.01 M) + diphenyl diselenide **2a** (0.01 M); The voltammogram was obtained at a scan rate of 100 mV/s with Pt wire as a counter electrode, Ag/AgCl as a reference electrode, and Pt disk electrode as a working electrode.

Table S3: Optimization table

entry[a]	variation from standard conditions	yield (%)[b]
1	**None**	**90%**
2	C_{gr} (+) and Ni (-)	85%
3	C_{gr} (+) and C_{gr} (-)	81%
4	Pt (+) and Pt (-)	75%
5	$TBAPF_6$	88%
6	$TBABF_4$	75%

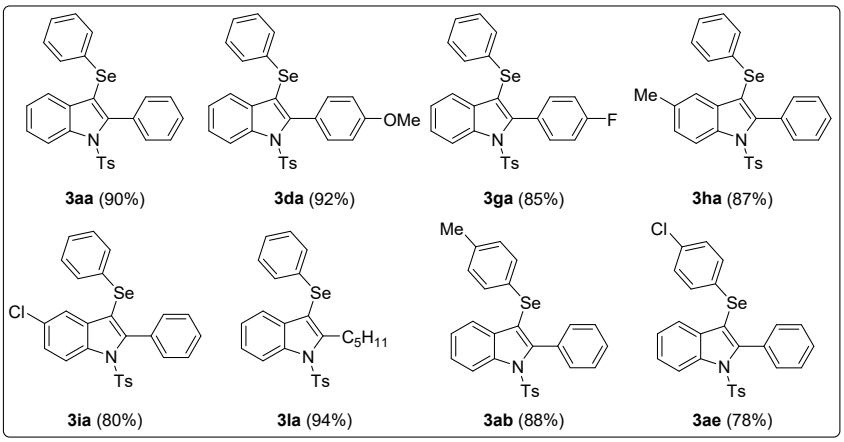
7	LiOTf	60%
8	TBAB	38%
9	TBACl	36%
10	TBAI	30%
11	$^{n}Et_4BF_4$	25%
12	DCE	60%
13	DCM	40%
14	DMF	15%
15	DMA	10%
16	CH_3CN/H_2O (5/1)	30%
17	DMF/H_2O (5/1)	0%
18	I = 5 mA, I = 8 mA, I = 15 mA	70%, 86%, 80%
19	No electricity	0%

Scheme S2: Scale up reaction.



1a (500 mg, 1.43 mmol) **2a** (449 mg, 1 equiv) **3aa** (629 mg, 87%)

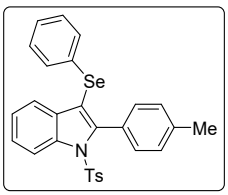
Table-S4. The following 3-selenylindoles (**3aa**, **3da**, **3ga**, **3ha**, **3ia**, **3la**, **3ab**, and **3ae)** are known in the literature as shown in Table-S4.[6]



3aa (90%) **3da** (92%) **3ga** (85%) **3ha** (87%)

3ia (80%) **3la** (94%) **3ab** (88%) **3ae** (78%)

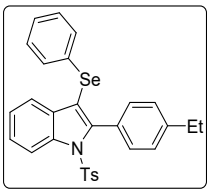
Characterization of compounds:



3-(Phenylselanyl)-2-(*p*-tolyl)-1-tosyl-1*H*-indole (3ba):

GP-5 was carried out with 4-methyl-*N*-(2-(*p*-tolylethynyl)phenyl)benzenesulfonamide **1b** (90 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ba** (122 mg, 95%) as a brown solid , mp = 142–144 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1b**) = 0.25, R_f (**2a**) = 0.95, R_f (**3ba**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3058, 2921, 1443, 1373, 1178, 1084, 1021, 817, 743, 671 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.26 (d, J = 8.4 Hz, 1H), 7.32 (d, J = 7.8 Hz, 1H), 7.27 (t, J = 7.7 Hz, 1H), 7.22 (d, J = 8.2 Hz, 2H), 7.16 – 7.06 (m, 5H), 6.96 – 6.89 (m, 5H), 6.87 – 6.78 (m, 2H), 2.30 (s, 3H), 2.19 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.2, 144.9, 139.2, 137.6, 134.9, 132.3, 131.9, 131.5 (2 × Ar–CH), 129.4 (2 × Ar–CH), 129.1 (2 × Ar–CH), 129.0 (2 × Ar–CH), 128.04, 128.01 (2 × Ar–CH), 126.9 (2 × Ar–CH), 126.0, 125.6, 124.7, 121.2, 116.4, 110.4, 21.65 (2 × CH_3) ppm. **HRMS (ESI)**: *m/z* calcd for $C_{28}H_{23}NNaO_2SSe^+$ $[M+Na]^+$: 540.0507, found: 540.0491.

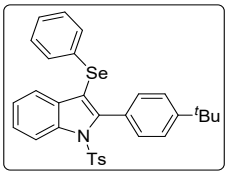


2-(4-ethylphenyl)-3-(phenylselanyl)-1-tosyl-1*H*-indole (3ca):

GP-5 was carried out with *N*-(2-((4-ethylphenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1c** (94 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ca** (116 mg, 87%) as a brown solid, mp = 146–148 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1c**) = 0.25, R_f (**2a**) = 0.95, R_f (**3ca**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3054, 2963, 1443, 1371, 1175, 1081, 1018, 831, 737, 665 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.36 (d, J = 8.4 Hz, 1H), 7.38 (ddd, J = 11.6, 8.4, 4.4 Hz, 2H), 7.33 – 7.27 (m, 2H), 7.27 – 7.17 (m, 5H), 7.08 – 6.97 (m, 5H), 6.92 (dd, J = 7.0, 1.2 Hz, 2H), 2.71 (q, J = 7.6 Hz, 2H), 2.29 (s, 3H), 1.28 (t, J = 7.6 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.3, 145.3, 144.9, 137.6, 134.9, 132.3, 131.9, 131.6 (2 × Ar–CH), 129.4 (2 × Ar–CH), 129.2 (2 × Ar–CH), 129.0 (2 × Ar–CH), 128.2, 126.9 (2 × Ar–CH), 126.7

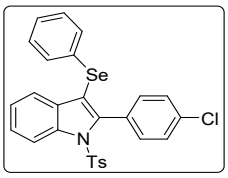
(2 × Ar–CH), 126.0, 125.6, 124.7, 121.2, 116.4, 110.4, 28.8, 21.7, 15.2 ppm. **HRMS (ESI):** *m/z* calcd for $C_{29}H_{25}NNaO_2SSe^+$ $[M+Na]^+$: 554.0663, found: 554.0647.



2-(4-(*tert*-butyl)phenyl)-3-(phenylselanyl)-1-tosyl-1*H*-indole (3ea)

GP-5 was carried out with *N*-(2-((4-(*tert*-butyl)phenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1e** (101 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ea** (117 mg, 84%) as a brown solid, mp = 132–134 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1e**) = 0.25, R_f (**2a**) = 0.95, R_f (**3ea**) = 0.40, UV detection].

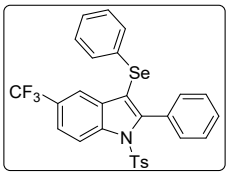
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3059, 2959, 1444, 1372, 1178, 1083, 1020, 829, 743, 670 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.36 (dd, J = 8.4, 0.7 Hz, 1H), 7.46 – 7.34 (m, 4H), 7.30 – 7.19 (m, 5H), 7.10 – 6.97 (m, 5H), 6.93 (d, J = 6.9 Hz, 2H), 2.30 (s, 3H), 1.37 (s, 9H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 152.2, 145.2, 144.8, 137.7, 135.0, 132.3, 131.9, 131.4 (2 × Ar–CH), 129.4 (2 × Ar–CH), 129.3 (2 × Ar–CH), 129.0 (2 × Ar–CH), 127.8, 127.0 (2 × Ar–CH), 126.1, 125.6, 124.7, 124.1 (2 × Ar–CH), 121.2, 116.4, 110.3, 34.9, 31.5 (3 × CH_3), 21.7 ppm. **HRMS (ESI):** *m/z* calcd for $C_{31}H_{30}NO_2SSe^+$ $[M+H]^+$: 560.1157, found: 560.1137.



2-(4-chlorophenyl)-3-(phenylselanyl)-1-tosyl-1*H*-indole (3fa):

GP-5 was carried out with *N*-(2-((4-chlorophenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1f** (95 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3fa** (109 mg, 82%) as a brown solid, mp = 184–186 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1f**) = 0.25, R_f (**2a**) = 0.95, R_f (**3fa**) = 0.40, UV detection].

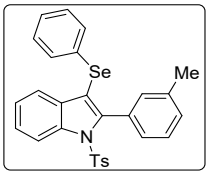
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3059, 2922, 1442, 1373, 1176, 1085, 1016, 825, 741, 668 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.36 (d, J = 8.4 Hz, 1H), 7.48 – 7.39 (m, 2H), 7.38 – 7.33 (m, 2H), 7.32 – 7.22 (m, 5H), 7.12 – 7.00 (m, 5H), 6.94 – 6.87 (m, 2H), 2.33 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.2, 143.6, 137.7, 135.4, 134.9, 132.9 (2 × Ar–CH), 132.2, 131.7, 129.6 (2 × Ar–CH), 129.5, 129.3 (2 × Ar–CH), 129.2 (2 × Ar–CH), 127.6 (2 × Ar–CH), 126.9 (2 × Ar–CH), 126.3, 126.1, 124.9, 121.4, 116.4, 111.2, 21.7 ppm. **HRMS (ESI):** *m/z* calcd for $C_{27}H_{20}ClKNO_2SSe^+$ $[M+K]^+$: 575.9700, found: 575.9664.



2-phenyl-3-(phenylselanyl)-1-tosyl-5-(trifluoromethyl)-1*H*-indole (3ja):

GP-5 was carried out with 4-methyl-*N*-(2-(phenylethynyl)-4-(trifluoromethyl)phenyl)benzenesulfonamide **1j** (104 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ja** (122 mg, 85%) as a brown solid, mp = 120–122 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1j**) = 0.25, R_f (**2a**) = 0.95, R_f (**3ja**) = 0.40, UV detection].

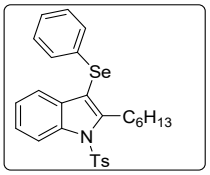
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3062, 2926, 1381, 1333, 1254, 1175, 1122, 692 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.55 (d, J = 8.8 Hz, 1H), 7.83 (s, 1H), 7.70 (d, J = 8.8 Hz, 1H), 7.50 (dd, J = 8.4, 6.3 Hz, 1H), 7.43 (t, J = 7.5 Hz, 2H), 7.37 – 7.29 (m, 4H), 7.19 – 7.05 (m, 5H), 7.01 (dd, J = 8.2, 1.3 Hz, 2H), 2.38 (s, 3H). ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 146.4, 145.6, 139.1, 134.9, 132.1, 131.7 (2 × Ar–CH), 131.2, 130.3, 129.8 (2 × Ar–CH), 129.7 (2 × Ar–CH), 129.6, 129.2 (2 × Ar–CH), 127.3 (2 × Ar–CH), 127.0 (2 × Ar–CH), 126.9 (q, $J_{C–F}$ = 32.3), 126.6, 124.5 (q, $J_{C–F}$ = 273.7 Hz) 122.4 (q, $J_{C–F}$ = 3.4 Hz), 118.8 (q, $J_{C–F}$ = 4.0 Hz), 116.6, 110.3, 21.7 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –61.19 –61.23 (m) ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{20}F_3KNO_2SSe^+$ $[M+K]^+$: 609.9964, found: 609.9962.



3-(phenylselanyl)-2-(*m*-tolyl)-1-tosyl-1*H*-indole (3ka):

GP-5 was carried out with 4-methyl-*N*-(2-(*m*-tolylethynyl)phenyl)benzenesulfonamide **1k** (91 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ka** (116 mg, 90%) as a brown solid, mp = 124–126 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1k**) = 0.25, R_f (**2a**) = 0.95, R_f (**3ka**) = 0.40, UV detection].

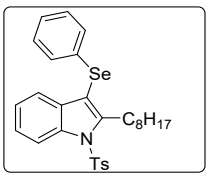
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3053, 2922, 1587, 1443, 1373, 1175, 1087, 1026, 797, 742, 669 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.27 (d, J = 8.4 Hz, 1H), 7.35 (d, J = 7.7 Hz, 1H), 7.28 (t, J = 7.8 Hz, 1H), 7.24 – 7.09 (m, 5H), 7.02 (d, J = 6.8 Hz, 1H), 6.98 – 6.88 (m, 6H), 6.88 – 6.80 (m, 2H), 2.22 (s, 3H), 2.19 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.0, 144.9, 137.6, 136.6, 135.2, 132.4, 132.1, 132.0, 130.8, 130.0, 129.5 (2 × Ar–CH), 129.4 (2 × Ar–CH), 129.0 (2 × Ar–CH), 128.7, 127.1, 127.0 (2 × Ar–CH), 126.1, 125.7, 124.6, 121.2, 116.2, 110.4, 21.6, 21.5 ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{23}NNaO_2SSe^+$ $[M+Na]^+$: 540.0507, found: 540.0495.



2-hexyl-3-(phenylselanyl)-1-tosyl-1*H*-indole (3ma):

GP-5 was carried out with 4-methyl-*N*-(2-(oct-1-yn-1-yl)phenyl)benzenesulfonamide **1m** (89 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ma** (121 mg, 95%) as a brown liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1m**) = 0.35, R_f (**2a**) = 0.95, R_f (**3ma**) = 0.50, UV detection].

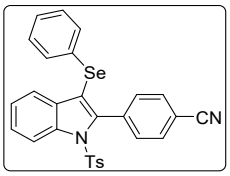
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3060, 2924, 1445, 1370, 1176, 1059, 745, 698 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.26 (d, J = 8.4 Hz, 1H), 7.67 – 7.63 (m, 2H), 7.49 – 7.45 (m, 1H), 7.33 (ddd, J = 8.5, 7.2, 1.3 Hz, 1H), 7.25 – 7.20 (m, 3H), 7.14 – 7.07 (m, 3H), 7.07 – 7.04 (m, 2H), 3.29 (t, J = 8 Hz, 2H), 2.36 (s, 3H), 1.72 – 1.66 (m, 2H), 1.44 – 1.36 (m, 2H), 1.33 – 1.26 (m, 4H), 0.89 (t, J = 7.1 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 147.3, 144.9, 137.1, 135.9, 132.0, 131.9, 129.9 (2 × Ar–CH), 129.1 (2 × Ar–CH), 128.8 (2 × Ar–CH), 126.4 (2 × Ar–CH), 126.0, 124.9, 124.3, 120.6, 115.3, 108.0, 31.5, 31.2, 29.3, 28.7, 22.6, 21.6, 14.2 ppm. **HRMS (ESI):** *m/z* calcd for $C_{27}H_{29}NNaO_2SSe^+$ $[M+Na]^+$: 534.0976, found: 534.0944.



2-octyl-3-(phenylselanyl)-1-tosyl-1*H*-indole (3na):

GP-5 was carried out with *N*-(2-(dec-1-yn-1-yl)phenyl)-4-methylbenzenesulfonamide **1n** (96 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3na** (124 mg, 92%) as a brown liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1n**) = 0.35, R_f (**2a**) = 0.95, R_f (**3na**) = 0.50, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3059, 2923, 1447, 1369, 1172, 1070, 741, 696, cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.12 (d, J = 8.4 Hz, 1H), 7.51 (d, J = 8.1 Hz, 2H), 7.33 (d, J = 7.7 Hz, 1H), 7.19 (t, J = 7.7 Hz, 1H), 7.13 – 7.03 (m, 3H), 7.00 – 6.87 (m, 5H), 3.15 (t, J = 8 Hz, 2H), 2.22 (s, 3H), 1.63 – 1.50 (m, 2H), 1.34 – 1.21 (m, 2H), 1.19 – 1.08 (m, 8H), 0.76 (t, J = 6.7 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 147.3, 144.9, 137.1, 135.9, 132.0, 131.9, 129.9 (2 × Ar–CH), 129.1 (2 × Ar–CH), 128.8 (2 × Ar–CH), 126.4 (2 × Ar–CH), 126.0, 124.9, 124.3, 120.6, 115.3, 108.0, 31.9, 31.3, 29.7, 29.3, 29.2, 28.6, 22.7, 21.6, 14.2 ppm. **HRMS (ESI):** *m/z* calcd for $C_{29}H_{33}NNaO_2SSe^+$ $[M+Na]^+$: 562.1289, found: 562.1256.

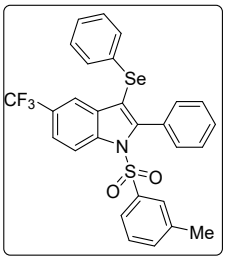


4-(3-(phenylselanyl)-1-tosyl-1*H*-indol-2-yl)benzonitrile (3oa):

GP-5 was carried out with *N*-(2-((4-cyanophenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1o** (93 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3oa** (92 mg, 70%) as a brown solid, mp = 174–176 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1o**) = 0.25, R_f (**2a**) = 0.95, R_f (**3oa**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3060, 2925, 2228, 1597, 1445, 1374, 1176, 1088, 1019, 841, 744, 667 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.36 (d, J = 8.4 Hz, 1H), 7.70 – 7.65 (m, 2H), 7.51 – 7.43 (m, 4H), 7.35 – 7.27 (m, 3H), 7.15 – 7.09 (m, 3H), 7.08 – 7.02 (m, 2H), 6.93 – 6.86 (m, 2H), 2.36 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.5, 142.5, 137.8,

135.9, 134.6, 132.3 (2 × Ar–CH), 132.2, 131.3, 131.0 (2 × Ar–CH), 129.7 (2 × Ar–CH), 129.5 (2 × Ar–CH), 129.3 (2 × Ar–CH), 126.8 (2 × Ar–CH), 126.6, 126.6, 125.2, 121.7, 118.8, 116.5, 112.8, 112.6, 21.8 ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{21}N_2O_2SSe^+$ $[M+H]^+$: 529.0483, found: 529.0432.

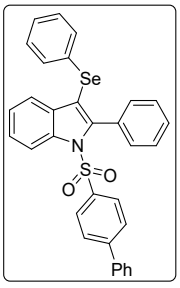


2-phenyl-3-(phenylselanyl)-1-(*m*-tolylsulfonyl)-5-(trifluoromethyl)-1*H*-indole (3pa):

GP-5 was carried out with 3-methyl-*N*-(2-(phenylethynyl)-4-(trifluoromethyl)phenyl)benzenesulfonamide **1p** (104 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3pa** (123 mg, 86%) as a brown solid, mp = 104–106 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1p**) = 0.25, R_f (**2a**) = 0.95, R_f (**3pa**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3062, 2925, 1379, 1331, 1252, 1169, 1116, 732 cm^{-1}.

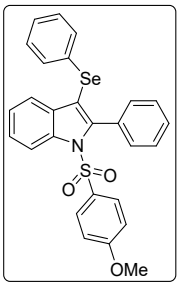
^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.41 (d, J = 8.8 Hz, 1H), 7.70 (s, 1H), 7.57 (d, J = 8.8 Hz, 1H), 7.41 – 7.33 (m, 1H), 7.27 (dd, J = 11.5, 4.2 Hz, 2H), 7.22 (d, J = 6.8 Hz, 1H), 7.16 – 7.04 (m, 5H), 7.01 – 6.95 (m, 3H), 6.87 (dd, J = 8.0, 1.2 Hz, 2H), 2.15 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 146.3, 139.4, 139.1, 137.8, 135.1, 132.0, 131.9 (2 × Ar–CH), 131.2, 130.2, 129.8 (2 × Ar–CH), 129.6, 129.3 (2 × Ar–CH), 129.0, 127.5, 127.3 (2 × Ar–CH), 126.9 (q, J = 33.3 Hz), 126.6, 124.5 (q, J = 272.7 Hz), 124.0, 122.4 (q, J = 3.4 Hz), 118.8 (q, J = 4.1 Hz), 116.6, 110.2, 21.3 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –61.26 (d, J = 8.6 Hz). ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{20}F_3KNO_2SSe^+$ $[M+K]^+$: 609.9964, found: 609.9943.



1-([1,1'-biphenyl]-4-ylsulfonyl)-2-phenyl-3-(phenylselanyl)-1*H*-indole (3qa):

GP-5 was carried out with *N*-(2-(phenylethynyl)phenyl)-[1,1'-biphenyl]-4-sulfonamide **1q** (103 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3qa** (114 mg, 80%) as a brown solid, mp = 122–124 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1q**) = 0.25, R_f (**2a**) = 0.95, R_f (**3qa**) = 0.40, UV detection].

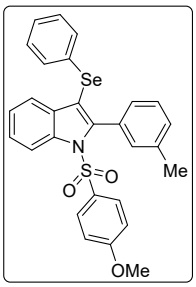
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3055, 2925, 1585, 1444, 1374, 1176, 1085, 1017, 747, 684 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.42 (d, J = 8.4 Hz, 1H), 7.51 – 7.31 (m, 16H), 7.25 (t, J = 7.5 Hz, 1H), 7.04 – 6.85 (m, 5H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 146.7, 144.8, 138.7, 137.6, 136.4, 132.2, 131.7 (2 × Ar–CH), 130.9, 129.34 (2 × Ar–CH), 129.30, 129.1 (3 × Ar–CH, 1 × Ar–C), 128.8, 127.5 (2 × Ar–CH), 127.34 (2 × Ar–CH), 127.30 (3 × Ar–CH), 127.2 (2 × Ar–CH), 126.2, 125.9, 124.8, 121.4, 116.4, 110.9 ppm. **HRMS (ESI):** *m/z* calcd for $C_{32}H_{23}NNaO_2SSe^+$ $[M+Na]^+$: 588.0507, found: 588.0488.



1-((4-methoxyphenyl)sulfonyl)-2-phenyl-3-(phenylselanyl)-1*H*-indole (3ra):

GP-5 was carried out with 4-methoxy-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **1r** (110 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ra** (114 mg, 88%) as a brown solid, mp = 130–132 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1r**) = 0.20, R_f (**2a**) = 0.95, R_f (**3ra**) = 0.30, UV detection].

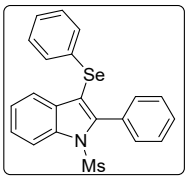
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3061, 2932, 1589, 1374, 1260, 1172, 1088, 1022, 756 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.37 (d, J = 8.4 Hz, 1H), 7.47 – 7.31 (m, 9H), 7.29 – 7.22 (m, 1H), 7.11 – 6.99 (m, 3H), 6.93 (dt, J = 3.6, 2.1 Hz, 2H), 6.77 – 6.69 (m, 2H), 3.76 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 163.8, 145.0, 137.7, 132.3, 131.9, 131.7 (2 × Ar–CH), 131.1, 129.6, 129.3 (2 × Ar–CH), 129.2 (2 × Ar–CH), 129.1 (2 × Ar–CH), 127.2 (2 × Ar–CH), 126.1, 125.8, 124.7, 121.3, 116.4, 114.0 (2 × Ar–CH), 110.6, 55.7 ppm. **HRMS (ESI):** *m/z* calcd for $C_{27}H_{21}NNaO_3SSe^+$ $[M+Na]^+$: 542.0300, found: 542.0286.



1-((4-methoxyphenyl)sulfonyl)-3-(phenylselanyl)-2-(*m*-tolyl)-1*H*-indole (3sa):

GP-5 was carried out with 4-methoxy-*N*-(2-(*m*-tolylethynyl)phenyl)benzenesulfonamide **1s** (94 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3sa** (115 mg, 87%) as a brown solid, mp = 116–118 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1s**) = 0.20, R_f (**2a**) = 0.95, R_f (**3sa**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3055, 2924, 1586, 1370, 1259, 1167, 1086, 1023, 739, 682 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.27 (dd, J = 8.4, 0.6 Hz, 1H), 7.39 – 7.34 (m, 1H), 7.32 – 7.22 (m, 3H), 7.20 – 7.10 (m, 3H), 7.02 (d, J = 7.0 Hz, 1H), 6.99 – 6.90 (m, 4H), 6.89 – 6.81 (m, 2H), 6.65 – 6.59 (m, 2H), 3.64 (s, 3H), 2.23 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 163.8, 145.1, 137.6, 136.6, 132.4, 132.2, 132.1, 130.9, 130.0, 129.6, 129.4 (2 × Ar–CH), 129.2 (2 × Ar–CH), 129.1 (2 × Ar–CH), 128.6, 127.1, 126.1, 125.6, 124.6, 121.2, 116.3, 113.9 (2 × Ar–CH), 110.3, 55.7, 21.5 ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{23}NNaO_3SSe^+$ $[M+Na]^+$: 556.0456, found: 556.0440.

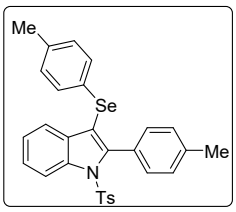


1-(methylsulfonyl)-2-phenyl-3-(phenylselanyl)-1*H*-indole (3ua):

GP-5 was carried out with *N*-(2-(phenylethynyl)phenyl)methanesulfonamide **1u** (68 mg, 0.25 mmol), 1,2-diphenyldiselane **2a** (78 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ua** (91 mg, 85%) as a brown solid, mp = 144–146 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1u**) = 0.25, R_f (**2a**) = 0.95, R_f (**3ua**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3059, 2926, 1444, 1369, 1173, 1104, 1023, 957, 758, 694 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.18 (d, J = 8.4 Hz, 1H), 7.62 (d, J = 7.8 Hz, 1H),

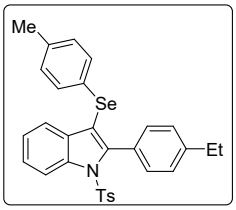
7.51 – 7.40 (m, 6H), 7.38 – 7.32 (m, 1H), 7.21 – 7.11 (m, 5H), 2.91 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 144.3, 136.9, 131.7, 131.40, 131.37 (2 × Ar–CH), 130.9, 130.1 (2 × Ar–CH), 129.5, 129.2 (2 × Ar–CH), 127.6 (2 × Ar–CH), 126.5, 125.9, 124.8, 121.6, 115.3, 110.2, 41.0 ppm. **HRMS (ESI):** *m/z* calcd for $C_{21}H_{17}NNaO_2SSe^+$ $[M+Na]^+$: 450.0037, found: 450.0018.



2-(*p*-tolyl)-3-(*p*-tolylselanyl)-1-tosyl-1*H*-indole (3bb):

GP-5 was carried out with 4-methyl-*N*-(2-(*p*-tolylethynyl)phenyl)benzenesulfonamide **1b** (90 mg, 0.25 mmol), 1,2-di-*p*-tolyldiselane **2b** (85 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3bb** (119 mg, 90%) as a brown solid, mp = 120–122 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1b**) = 0.25, R_f (**2b**) = 0.95, R_f (**3bb**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3029, 2923, 1445, 1376, 1179, 1088, 1020, 813, 752, 665 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.35 (dd, J = 8.4, 0.6 Hz, 1H), 7.45 – 7.34 (m, 2H), 7.31 (dd, J = 8.3, 1.2 Hz, 2H), 7.23 (dt, J = 14.6, 4.0 Hz, 5H), 7.07 (d, J = 8.1 Hz, 2H), 6.84 (d, J = 1.1 Hz, 4H), 2.42 (s, 3H), 2.32 (s, 3H), 2.21 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 144.9, 144.9, 139.2, 137.6, 135.9, 135.0, 132.3, 131.6 (2 × Ar–CH), 129.9 (2 × Ar–CH), 129.6 (2 × Ar–CH), 129.4 (2 × Ar–CH), 128.1, 128.0 (2 × Ar–CH), 127.9, 126.9 (2 × Ar–CH), 125.6, 124.7, 121.2, 116.4, 110.8, 21.7 (2 × CH_3), 21.0 ppm. **HRMS (ESI):** *m/z* calcd for $C_{29}H_{25}KNO_2SSe^+$ $[M+K]^+$: 570.0403, found: 570.0387.

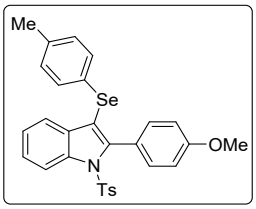


2-(4-ethylphenyl)-3-(*p*-tolylselanyl)-1-tosyl-1*H*-indole (3cb):

GP-5 was carried out with *N*-(2-((4-ethylphenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1c** (94 mg, 0.25 mmol), 1,2-di-*p*-tolyldiselane **2b** (85 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05)

furnished the product **3cb** (116 mg, 85%) as a brown solid, mp = 110–112 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1c**) = 0.25, R_f (**2b**) = 0.95, R_f (**3cb**) = 0.40, UV detection].

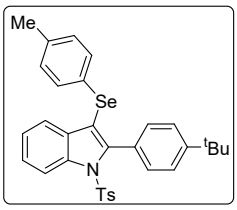
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3029, 2963, 14444, 1372, 1175, 1086, 1017, 807, 744, 663 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.35 (d, J = 8.4 Hz, 1H), 7.42 (dd, J = 7.8, 0.5 Hz, 1H), 7.37 (ddd, J = 8.5, 7.3, 1.3 Hz, 1H), 7.32 – 7.19 (m, 7H), 7.05 (d, J = 8.1 Hz, 2H), 6.84 (s, 4H), 2.72 (q, J = 7.6 Hz, 2H), 2.30 (s, 3H), 2.20 (s, 3H), 1.29 (t, J = 7.6 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.3, 144.9, 144.8, 137.6, 135.9, 135.0, 132.3, 131.7 (2 × Ar–CH), 129.9 (2 × Ar–CH), 129.6 (2 × Ar–CH), 129.4 (2 × Ar–CH), 128.3, 127.9, 126.9 (2 × Ar–CH), 126.7 (2 × Ar–CH), 125.6, 124.6, 121.2, 116.4, 110.8, 28.8, 21.7, 21.0, 15.2 ppm. **HRMS (ESI):** *m/z* calcd for $C_{30}H_{27}NNaO_2SSe^+$ $[M+Na]^+$: 568.0820, found: 568.0806.



2-(4-methoxyphenyl)-3-(*p*-tolylselanyl)-1-tosyl-1*H*-indole (3db):

GP-5 was carried out with *N*-(2-((4-methoxyphenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1d** (95 mg, 0.25 mmol), 1,2-di-*p*-tolyldiselane **2b** (85 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3db** (113 mg, 82%) as a brown solid, mp = 90–92 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1d**) = 0.20, R_f (**2b**) = 0.95, R_f (**3db**) = 0.30, UV detection].

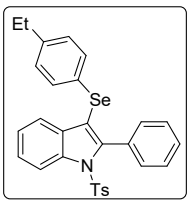
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3043, 2926, 1605, 1494, 1445, 1373, 1248, 1176, 1086, 1024, 825, 749, 665 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.35 (d, J = 8.4 Hz, 1H), 7.45 – 7.35 (m, 2H), 7.31 – 7.22 (m, 5H), 7.07 (d, J = 8.1 Hz, 2H), 6.95 – 6.89 (m, 2H), 6.88 – 6.78 (m, 4H), 3.86 (s, 3H), 2.33 (s, 3H), 2.22 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 160.3, 144.9, 144.8, 137.6, 135.9, 135.1, 133.2 (2 × Ar–CH), 132.4, 129.9 (2 × Ar–CH), 129.5 (2 × Ar–CH), 129.4 (2 × Ar–CH), 128.0, 126.9 (2 × Ar–CH), 125.6, 124.7, 123.2, 121.2, 116.5, 112.7 (2 × Ar–CH), 110.6, 55.3, 21.7, 21.1 ppm. **HRMS (ESI):** *m/z* calcd for $C_{29}H_{25}KNO_3SSe^+$ $[M+K]^+$: 586.0352, found: 586.0356.



2-(4-(*tert*-butyl)phenyl)-3-(*p*-tolylselanyl)-1-tosyl-1*H*-indole (3eb):

GP-5 was carried out with *N*-(2-((4-(*tert*-butyl)phenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1e** (101 mg, 0.25 mmol), 1,2-di-*p*-tolyldiselane **2b** (85 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3eb** (115 mg, 80%) as a brown solid, mp = 105–106 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1e**) = 0.25, R_f (**2b**) = 0.95, R_f (**3eb**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3035, 2959, 1445, 1371, 1176, 1081, 1018, 804, 744, 666 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.26 (d, J = 8.4 Hz, 1H), 7.36 – 7.32 (m, 1H), 7.31 – 7.25 (m, 3H), 7.20 – 7.10 (m, 5H), 6.95 (d, J = 8.0 Hz, 2H), 6.75 (s, 4H), 2.21 (s, 3H), 2.11 (s, 3H), 1.28 (s, 9H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 152.1, 144.9, 144.7, 137.7, 135.9, 135.0, 132.3, 131.5 (2 × Ar–CH), 129.9 (2 × Ar–CH), 129.7 (2 × Ar–CH), 129.4 (2 × Ar–CH), 127.94, 127.91, 127.0 (2 × Ar–CH), 125.5, 124.6, 124.1 (2 × Ar–CH), 121.2, 116.4, 110.7, 34.9, 31.5 (3 × CH_3), 21.7, 21.05 ppm. **HRMS (ESI):** *m/z* calcd for $C_{32}H_{31}NNaO_2SSe^+$ $[M+Na]^+$: 596.1133, found: 596.1115.

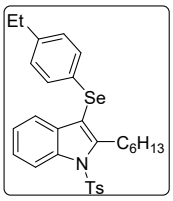


3-((4-ethylphenyl)selanyl)-2-phenyl-1-tosyl-1*H*-indole (3ac):

GP-5 was carried out with 4-methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **1a** (87 mg, 0.25 mmol), 1,2-bis(4-ethylphenyl)diselane **2c** (92 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ac** (113 mg, 85%) as a brown solid, mp = 132–134 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1a**) = 0.25, R_f (**2c**) = 0.95, R_f (**3ac**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3057, 2925, 1446, 1377, 1179, 1088, 761 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.36 (d, J = 8.4 Hz, 1H), 7.48 – 7.36 (m, 5H), 7.36 – 7.29 (m, 4H), 7.28 – 7.23 (m, 1H), 7.08 (d, J = 8.0 Hz, 2H), 6.87 (s, 4H), 2.52 (q, J = 7.6 Hz, 2H), 2.34

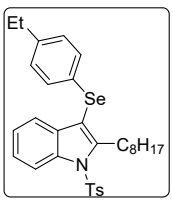
(s, 3H), 1.15 (t, J = 7.6 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 144.9, 144.7, 142.5, 137.6, 135.2, 132.3, 131.8 (2 × Ar–CH), 131.1, 129.8 (2 × Ar–CH), 129.5 (2 × Ar–CH), 129.2, 128.7 (2 × Ar–CH), 128.1, 127.2 (2 × Ar–CH), 127.0 (2 × Ar–CH), 125.7, 124.7, 121.4, 116.4, 111.1, 28.4, 21.7, 15.5 ppm. **HRMS (ESI):** *m/z* calcd for $C_{29}H_{25}NNaO_2SSe^+$ $[M+Na]^+$: 554.0663, found: 554.0639.



3-((4-ethylphenyl)selanyl)-2-hexyl-1-tosyl-1*H*-indole (3mc):

GP-5 was carried out with 4-methyl-*N*-(2-(oct-1-yn-1-yl)phenyl)benzenesulfonamide **1m** (89 mg, 0.25 mmol), 1,2-bis(4-ethylphenyl)diselane **2c** (92 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3mc** (120 mg, 89%) as a brown liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1m**) = 0.35, R_f (**2c**) = 0.95, R_f (**3mc**) = 0.50, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2925, 2860, 1448, 1370, 1176, 1017, 814, 752, 702, 659 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.11 (d, J = 8.4 Hz, 1H), 7.51 (d, J = 8.2 Hz, 2H), 7.35 (d, J = 7.6 Hz, 1H), 7.19 (dt, J = 8.3, 2.6 Hz, 1H), 7.14 – 7.03 (m, 3H), 6.83 (dd, J = 22.0, 8.2 Hz, 4H), 3.16 (t, J = 8 Hz, 2H), 2.43 (q, J = 7.6 Hz, 2H), 2.23 (s, 3H), 1.55 (dt, J = 15.3, 7.7 Hz, 2H), 1.31 – 1.21 (m, 2H), 1.19 – 1.12 (m, 4H), 1.05 (t, J = 7.6 Hz, 3H), 0.76 (t, J = 6.8 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 147.1, 144.9, 142.3, 137.1, 135.9, 132.1, 129.9 (2 × Ar–CH), 129.2 (2 × Ar–CH), 128.8 (2 × Ar–CH), 128.3, 126.4 (2 × Ar–CH), 124.9, 124.2, 120.7, 115.3, 108.4, 31.6, 31.2, 29.4, 28.7, 28.4, 22.6, 21.6, 15.5, 14.2 ppm. **HRMS (ESI):** *m/z* calcd for $C_{29}H_{33}KNO_2SSe^+$ $[M+K]^+$: 578.1029, found: 578.1008.

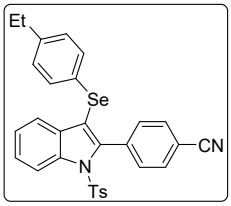


3-((4-ethylphenyl)selanyl)-2-octyl-1-tosyl-1*H*-indole (3nc):

GP-5 was carried out with *N*-(2-(dec-1-yn-1-yl)phenyl)-4-methylbenzenesulfonamide **1n** (96 mg, 0.25 mmol), 1,2-bis(4-ethylphenyl)diselane **2c** (92 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3nc**

(130 mg, 92%) as a brown liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1n**) = 0.35, R_f (**2c**) = 0.95, R_f (**3nc**) = 0.50, UV detection].

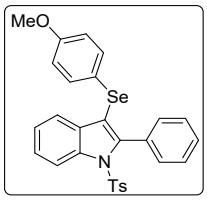
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2923, 2857, 1448, 1369, 1175, 814, 752, 701, 658 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.10 (dd, J = 8.3, 1.0 Hz, 1H), 7.55 – 7.46 (m, 2H), 7.34 (d, J = 7.6 Hz, 1H), 7.18 (t, J = 7.8 Hz, 1H), 7.10 – 7.02 (m, 3H), 6.83 (dt, J = 21.4, 4.6 Hz, 4H), 3.16 (t, J = 8 Hz, 2H), 2.42 (q, J = 7.6 Hz, 2H), 2.21 (s, 3H), 1.63 – 1.49 (m, 2H), 1.33 – 1.21 (m, 2H), 1.18 – 1.10 (m, 8H), 1.05 (t, J = 7.6 Hz, 3H), 0.76 (t, J = 6.1 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 147.1, 144.9, 142.3, 137.1, 135.9, 132.1, 129.9 (2 × Ar–CH), 129.2 (2 × Ar–CH), 128.7 (2 × Ar–CH), 128.3, 126.4 (2 × Ar–CH), 124.9, 124.2, 120.7, 115.3, 108.4, 31.9, 31.3, 29.7, 29.35, 29.29, 28.7, 28.4, 22.7, 21.6, 15.5, 14.2 ppm. **HRMS (ESI):** *m/z* calcd for $C_{31}H_{37}NNaO_2SSe^+$ $[M+Na]^+$: 590.1602, found: 590.1579.



4-(3-((4-ethylphenyl)selanyl)-1-tosyl-1*H*-indol-2-yl)benzonitrile (3oc):

GP-5 was carried out with *N*-(2-((4-cyanophenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1o** (93 mg, 0.25 mmol), 1,2-bis(4-ethylphenyl)diselane **2c** (92 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3oc** (104 mg, 75%) as a brown solid, mp = 136–138 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1o**) = 0.25, R_f (**2c**) = 0.95, R_f (**3oc**) = 0.40, UV detection].

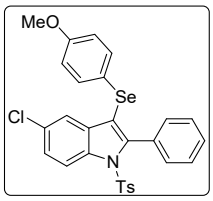
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3055, 2964, 2227, 1444, 1374, 1175, 1084, 1015, 829, 746, 663 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.37 (d, J = 8.4 Hz, 1H), 7.72 – 7.66 (m, 2H), 7.55 – 7.43 (m, 4H), 7.34 – 7.28 (m, 3H), 7.13 (d, J = 8.2 Hz, 2H), 6.87 (dd, J = 25.9, 8.2 Hz, 4H), 2.55 (q, J = 7.6 Hz, 2H), 2.36 (s, 3H), 1.18 (t, J = 7.6 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.3, 142.8, 142.1, 137.7, 135.9, 134.4, 132.3 (2 × Ar–CH), 130.9 (2 × Ar–CH), 129.7 (2 × Ar–CH), 129.6 (2 × Ar–CH), 128.8 (2 × Ar–CH), 127.5, 126.7 (2 × Ar–CH, 1 × Ar–C), 126.5, 125.1, 121.6, 118.7, 116.4, 112.9, 112.6, 28.3, 21.7, 15.4 ppm. **HRMS (ESI):** *m/z* calcd for $C_{30}H_{24}N_2NaO_2SSe^+$ $[M+Na]^+$: 579.0616, found: 579.0593.



3-((4-methoxyphenyl)selanyl)-2-phenyl-1-tosyl-1*H*-indole (3ad):

GP-5 was carried out with 4-methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **1a** (87 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)diselane **2d** (93 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3oc** (115 mg, 86%) as a brown solid, mp = 100–102 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1a**) = 0.25, R_f (**2d**) = 0.80, R_f (**3ad**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3056, 2935, 1589, 1444, 1372, 1241, 1174, 1085, 1023, 817, 755, 663 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.34 (d, J = 8.4 Hz, 1H), 7.49 – 7.35 (m, 5H), 7.34 – 7.22 (m, 5H), 7.06 (d, J = 8.4 Hz, 2H), 6.97 – 6.91 (m, 2H), 6.63 – 6.56 (m, 2H), 3.68 (s, 3H), 2.30 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 158.7, 144.9, 144.1, 137.5, 135.1, 132.24 (2 × Ar–CH), 132.2, 131.8 (2 × Ar–CH), 131.2, 129.5 (2 × Ar–CH), 129.2, 127.2 (2 × Ar–CH), 126.9 (2 × Ar–CH), 125.6, 124.6, 121.3, 121.2, 116.2, 114.8 (2 × Ar–CH), 111.8, 55.3, 21.7 ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{23}NNaO_3SSe^+$ $[M+Na]^+$: 556.0456, found: 556.0439.

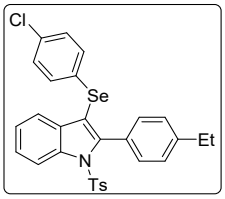


5-chloro-3-((4-methoxyphenyl)selanyl)-2-phenyl-1-tosyl-1*H*-indole (3id):

GP-5 was carried out with *N*-(4-chloro-2-(phenylethynyl)phenyl)-4-methylbenzenesulfonamide **1i** (95 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)diselane **2d** (93 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3id** (116 mg, 82%) as a brown solid, mp = 114–116 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1i**) = 0.25, R_f (**2d**) = 0.80, R_f (**3id**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3062, 2941, 1587, 1443, 1375, 1245, 1176, 1087, 1025, 812, 696 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.31 (d, J = 8.9 Hz, 1H), 7.55 – 7.47 (m, 2H), 7.44 (t, J = 7.3 Hz, 2H), 7.38 – 7.30 (m, 5H), 7.11 (d, J = 8.1 Hz, 2H), 6.97 (d, J = 8.8 Hz, 2H),

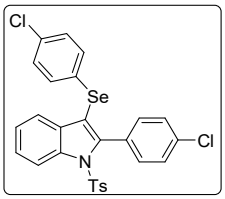
6.68 – 6.61 (m, 2H), 3.72 (s, 3H), 2.35 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 158.9, 145.5, 145.2, 135.8, 134.8, 133.7, 132.4 (2 × Ar–CH), 131.8 (2 × Ar–CH), 130.7, 130.4, 129.6 (2 × Ar–CH), 129.4, 127.3 (2 × Ar–CH), 126.9 (2 × Ar–CH), 125.8, 120.84, 120.82, 117.4, 114.9 (2 × Ar–CH), 110.9, 55.3, 21.7 ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{22}ClNNaO_3SSe^+$ $[M+Na]^+$: 590.0066, found: 590.0049.



3-((4-chlorophenyl)selanyl)-2-(4-ethylphenyl)-1-tosyl-1*H*-indole (3ce):

GP-5 was carried out with *N*-(2-((4-ethylphenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1c** (94 mg, 0.25 mmol), 1,2-bis(4-chlorophenyl)diselane **2e** (95 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ce** (110 mg, 78%) as a brown solid, mp = 110–112 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1c**) = 0.25, R_f (**2e**) = 0.95, R_f (**3ce**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3037, 2962, 1444, 1373, 1178, 1085, 1017, 810, 747, 667 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.29 (d, J = 9.0 Hz, 1H), 7.35 – 7.29 (m, 2H), 7.24 – 7.17 (m, 3H), 7.13 (s, 4H), 7.00 (d, J = 8.2 Hz, 2H), 6.94 – 6.88 (m, 2H), 6.80 – 6.72 (m, 2H), 2.65 (q, J = 7.6 Hz, 2H), 2.25 (s, 3H), 1.22 (t, J = 7.6 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.5, 145.4, 145.0, 137.6, 135.1, 132.1, 131.9, 131.6 (2 × Ar–CH), 130.6 (2 × Ar–CH), 130.2, 129.5 (2 × Ar–CH), 129.2 (2 × Ar–CH), 128.0, 127.0 (2 × Ar–CH), 126.8 (2 × Ar–CH), 125.8, 124.8, 121.0, 116.5, 110.0, 28.8, 21.7, 15.2 ppm. **HRMS (ESI):** *m/z* calcd for $C_{29}H_{24}ClNNaO_2SSe^+$ $[M+Na]^+$: 588.0274, found: 588.0236.

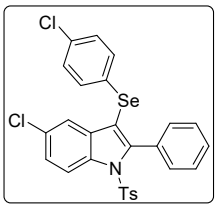


2-(4-chlorophenyl)-3-((4-chlorophenyl)selanyl)-1-tosyl-1*H*-indole (3fe):

GP-5 was carried out with *N*-(2-((4-chlorophenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1f** (95 mg, 0.25 mmol), 1,2-bis(4-chlorophenyl)diselane **2e** (95 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl

acetate, 95:05) furnished the product **3fe** (109 mg, 77%) as a brown solid, mp = 140–142 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1f**) = 0.25, R_f (**2e**) = 0.95, R_f (**3fe**) = 0.40, UV detection].

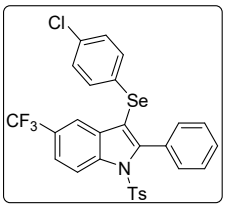
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3059, 2923, 1376, 1179, 1088, 1015, 821, 750, 666 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.29 (d, J = 9.0 Hz, 1H), 7.39 – 7.33 (m, 2H), 7.31 – 7.26 (m, 2H), 7.22 (d, J = 8.3 Hz, 3H), 7.18 – 7.11 (m, 2H), 7.02 (d, J = 8.2 Hz, 2H), 6.96 – 6.88 (m, 2H), 6.79 – 6.70 (m, 2H), 2.27 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.3, 143.7, 137.6, 135.6, 134.9, 132.9 (2 × Ar–CH), 132.4, 131.9, 130.6 (2 × Ar–CH), 129.9, 129.6 (2 × Ar–CH), 129.33 (2 × Ar–CH), 129.31, 127.7 (2 × Ar–CH), 126.9 (2 × Ar–CH), 126.2, 125.0, 121.2, 116.5, 110.8, 21.7 ppm. **HRMS (ESI):** *m/z* calcd for $C_{27}H_{19}Cl_2NNaO_2SSe^+$ $[M+Na]^+$: 593.9571, found: 593.9543.



5-chloro-3-((4-chlorophenyl)selanyl)-2-phenyl-1-tosyl-1*H*-indole (3ie):

GP-5 was carried out with *N*-(4-chloro-2-(phenylethynyl)phenyl)-4-methylbenzenesulfonamide **1i** (95 mg, 0.25 mmol), 1,2-bis(4-chlorophenyl)diselane **2e** (95 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ie** (114 mg, 80%) as a brown solid, mp = 124–126 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1i**) = 0.25, R_f (**2e**) = 0.95, R_f (**3ie**) = 0.40, UV detection].

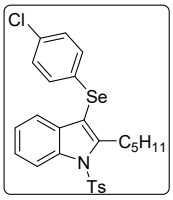
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3058, 2923, 1440, 1376, 1176, 1085, 1013, 810, 741, 692, 664 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.22 (d, J = 8.8 Hz, 1H), 7.41 – 7.35 (m, 2H), 7.33 – 7.27 (m, 3H), 7.22 – 7.15 (m, 4H), 7.02 (d, J = 8.1 Hz, 2H), 6.95 – 6.90 (m, 2H), 6.77 – 6.71 (m, 2H), 2.27 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 146.6, 145.4, 135.9, 134.9, 133.4, 132.5, 131.7 (2 × Ar–CH), 130.8, 130.7 (2 × Ar–CH), 130.3, 129.75, 129.7 (2 × Ar–CH), 129.4 (2 × Ar–CH), 127.4 (2 × Ar–CH), 127.0 (2 × Ar–CH), 126.2, 120.7, 117.6, 109.4, 21.8 ppm. **HRMS (ESI):** *m/z* calcd for $C_{27}H_{19}Cl_2NNaO_2SSe^+$ $[M+Na]^+$: 593.9571, found: 593.9553.



3-((4-chlorophenyl)selanyl)-2-phenyl-1-tosyl-5-(trifluoromethyl)-1*H*-indole (3je):

GP-5 was carried out with 4-methyl-*N*-(2-(phenylethynyl)-4-(trifluoromethyl)phenyl)benzenesulfonamide **1j** (104 mg, 0.25 mmol), 1,2-bis(4-chlorophenyl)diselane **2e** (95 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3je** (114 mg, 75%) as a brown solid, mp = 110–112 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1j**) = 0.25, R_f (**2e**) = 0.95, R_f (**3je**) = 0.40, UV detection].

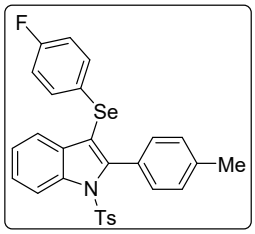
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3061, 2926, 1382, 1335, 1256, 1177, 1125, 815, 697, 663 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.41 (d, J = 8.8 Hz, 1H), 7.75 – 7.65 (m, 1H), 7.58 (dd, J = 8.8, 1.6 Hz, 1H), 7.44 – 7.36 (m, 1H), 7.34 – 7.27 (m, 2H), 7.24 – 7.19 (m, 2H), 7.17 – 7.12 (m, 2H), 7.04 (d, J = 8.1 Hz, 2H), 6.98 – 6.90 (m, 2H), 6.84 – 6.73 (m, 2H), 2.28 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 146.7, 145.7, 139.0, 135.0, 132.7, 131.8, 131.7 (2 × Ar–CH), 131.1 (2 × Ar–CH), 130.2, 129.8 (2 × Ar–CH), 129.7, 129.5, 129.4 (2 × Ar–CH), 127.4 (2 × Ar–CH), 127.1 (q, $J_{C–F}$ = 32.3 Hz), 127.09 (2 × Ar–CH), 124.4 (q, $J_{C–F}$ = 272.7 Hz), 122.6 (q, $J_{C–F}$ = 4.0 Hz), 118.6 (q, $J_{C–F}$ = 4.0 Hz), 116.7, 109.9, 21.8 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –61.3. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{19}ClF_3NO_2SSe^+$ $[M]^+$: 604.9937, found: 604.9961.



3-((4-chlorophenyl)selanyl)-2-pentyl-1-tosyl-1*H*-indole (3le):

GP-5 was carried out with *N*-(2-(hept-1-yn-1-yl)phenyl)-4-methylbenzenesulfonamide **1l** (85 mg, 0.25 mmol), 1,2-bis(4-chlorophenyl)diselane **2e** (95 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3le** (112 mg, 85%) as a brown liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1l**) = 0.35, R_f (**2e**) = 0.95, R_f (**3le**) = 0.50, UV detection].

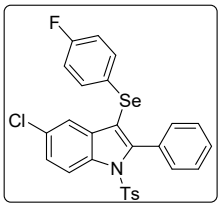
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3058, 2932, 1452, 1371, 1173, 1085, 810, 750, 702, 658 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.13 (d, *J* = 8.4 Hz, 1H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.31 (dd, *J* = 7.7, 0.4 Hz, 1H), 7.26 – 7.18 (m, 1H), 7.16 – 7.06 (m, 3H), 6.97 – 6.90 (m, 2H), 6.87 – 6.79 (m, 2H), 3.14 (t, *J* = 8 Hz, 2H), 2.26 (s, 3H), 1.63 – 1.50 (m, 2H), 1.29 – 1.17 (m, 4H), 0.76 (t, *J* = 7.0 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 147.4, 145.1, 137.1, 135.9, 132.1, 131.7, 130.3, 130.1 (2 × Ar–CH), 129.9 (2 × Ar–CH), 129.2 (2 × Ar–CH), 126.4 (2 × Ar–CH), 125.1, 124.4, 120.5, 115.4, 107.7, 31.8, 31.0, 28.6, 22.4, 21.7, 14.09 ppm. **HRMS (ESI):** *m/z* calcd for $C_{26}H_{27}ClNO_2SSe^+$ $[M+H]^+$: 532.0611, found: 532.0574.



3-((4-fluorophenyl)selanyl)-2-(m-tolyl)-1-tosyl-1*H*-indole (3bf):

GP-5 was carried out with 4-methyl-*N*-(2-(*p*-tolylethynyl)phenyl)benzenesulfonamide **1b** (90 mg, 0.25 mmol), 1,2-bis(4-fluorophenyl)diselane **2f** (87 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3bf** (109 mg, 82%) as a brown solid, mp = 118–120 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1b**) = 0.25, R_f (**2f**) = 0.95, R_f (**3bf**) = 0.40, UV detection].

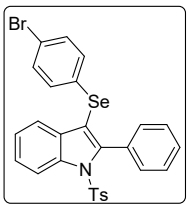
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3034, 2922, 1486, 1443, 1373, 1221, 1174, 1085, 1020, 816, 746, 666 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.36 (d, *J* = 8.4 Hz, 1H), 7.45 – 7.34 (m, 2H), 7.34 – 7.29 (m, 2H), 7.27 – 7.22 (m, 1H), 7.21 (s, 4H), 7.07 (d, *J* = 8.1 Hz, 2H), 6.98 – 6.88 (m, 2H), 6.77 – 6.70 (m, 2H), 2.42 (s, 3H), 2.31 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 161.7 (d, *J* = 246.4 Hz), 144.97, 144.9, 139.3, 137.5, 135.0, 131.9, 131.6 (2 × Ar–CH), 131.58 (d, *J* = 7.4 Hz, 2 × Ar–CH), 129.4 (2 × Ar–CH), 128.1 (2 × Ar–CH), 128.0, 126.9 (2 × Ar–CH), 126.1 (d, *J* = 3.3 Hz), 125.7, 124.7, 121.0, 116.4, 116.2 (d, *J* = 21.7 Hz, 2 × Ar–CH), 116.1, 114.7, 110.7, 21.67, 21.66 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –115.92 (dd, *J* = 11.5, 8.0 Hz). **HRMS (ESI):** *m/z* calcd for $C_{28}H_{23}FNO_2SSe^+$ $[M+H]^+$: 536.0593, found: 536.0581.



5-chloro-3-((4-fluorophenyl)selanyl)-2-phenyl-1-tosyl-1*H*-indole (3if):

GP-5 was carried out with *N*-(4-chloro-2-(phenylethynyl)phenyl)-4-methylbenzenesulfonamide **1i** (95 mg, 0.25 mmol), 1,2-bis(4-fluorophenyl)diselane **2f** (87 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3if** (110 mg, 80%) as a brown solid, mp = 138–140 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1i**) = 0.25, R_f (**2f**) = 0.95, R_f (**3if**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3062, 2925, 1440, 1376, 1225, 1176, 1088, 909, 816, 734, 664 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.33 (d, J = 8.9 Hz, 1H), 7.53 – 7.46 (m, 2H), 7.45 – 7.36 (m, 3H), 7.35 – 7.27 (m, 4H), 7.12 (d, J = 8.1 Hz, 2H), 7.00 – 6.88 (m, 2H), 6.87 – 6.71 (m, 2H), 2.36 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 161.9 (d, J = 246.4 Hz), 146.1, 145.4, 135.8, 134.9, 133.4, 131.9 (d, J = 7.7 Hz, 2 × Ar–CH), 131.7 (2 × Ar–CH), 130.6, 130.4, 129.6 (2 × Ar–CH), 129.5, 127.3 (2 × Ar–CH), 126.9 (2 × Ar–CH), 126.0, 125.6 (d, J = 3.4 Hz), 120.7, 117.5, 116.4 (d, J = 21.7 Hz, 2 × Ar–CH), 110.1, 21.69 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –115.32 (d, J = 6.9 Hz). **HRMS (ESI):** *m/z* calcd for $C_{27}H_{19}ClFKNO_2SSe^+$ $[M+K]^+$: 593.9606, found: 593.9617.

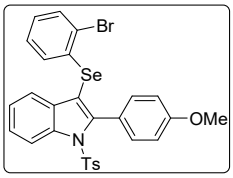


3-((4-bromophenyl)selanyl)-2-phenyl-1-tosyl-1*H*-indole (3ag):

GP-5 was carried out with 4-methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **1a** (87 mg, 0.25 mmol), 1,2-bis(4-bromophenyl)diselane **2g** (118 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3if** (102 mg, 70%) as a brown solid, mp = 142–144 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1a**) = 0.25, R_f (**2g**) = 0.95, R_f (**3ag**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3059, 2924, 1449, 1377, 1179, 1085, 1010, 760, 699, 664 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.31 (dd, J = 6.4, 2.6 Hz, 1H), 7.41 – 7.29 (m, 5H), 7.26 – 7.17 (m, 5H), 7.11 – 7.04 (m, 2H), 7.02 (d, J = 8.0 Hz, 2H), 6.75 – 6.67 (m, 2H), 2.28 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.2, 137.6, 135.1, 132.2 (2 × Ar–CH), 131.9, 131.7 (2 × Ar–CH), 130.94, 130.9 (2 × Ar–CH), 130.8, 129.6 (2 × Ar–CH), 129.5,

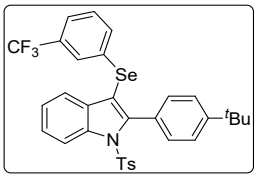
129.4, 127.3 (2 × Ar–CH), 127.0 (2 × Ar–CH), 126.0, 124.9, 121.1, 120.1, 116.5, 110.1, 21.8 ppm. **HRMS (ESI):** *m/z* calcd for $C_{27}H_{20}Br^{79}NO_2SSe^+$ $[M]^+$: 580.9558, found: 580.9588. $C_{27}H_{20}Br^{81}NO_2SSe^+$ $[M]^+$: 582.9537, found: 582.9562.



3-((2-bromophenyl)selanyl)-2-(4-methoxyphenyl)-1-tosyl-1*H*-indole (3dh):

GP-5 was carried out with *N*-(2-((4-methoxyphenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1d** (95 mg, 0.25 mmol), 1,2-bis(2-bromophenyl)diselane **2h** (118 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3dh** (115 mg, 75%) as a brown solid, mp = 190–192 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1d**) = 0.20, R_f (**2h**) = 0.95, R_f (**3dh**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3054, 2924, 1606, 1496, 1441, 1374, 1249, 1176, 1087, 1021, 829, 745, 666 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.33 (d, J = 8.4 Hz, 1H), 7.41 – 7.28 (m, 3H), 7.25 – 7.14 (m, 5H), 7.03 (d, J = 8.0 Hz, 2H), 6.92 – 6.79 (m, 3H), 6.72 (td, J = 7.7, 1.3 Hz, 1H), 6.19 (dd, J = 7.9, 1.5 Hz, 1H), 3.79 (s, 3H), 2.28 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 160.5, 146.2, 145.0, 137.8, 135.2, 135.1, 133.0 (2 × Ar–CH), 132.7, 132.0, 129.5 (2 × Ar–CH), 128.7, 127.7, 127.1 (2 × Ar–CH), 126.9, 125.9, 124.9, 122.7, 122.5, 121.1, 116.7, 112.8 (2 × Ar–CH), 110.0, 55.4, 21.8 ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{22}Br^{79}KNO_3SSe^+$ $[M+K]^+$: 649.9301, found: 649.9277. $C_{28}H_{22}Br^{81}KNO_3SSe^+$ $[M+K]^+$: 651.9280, found: 651.9265.

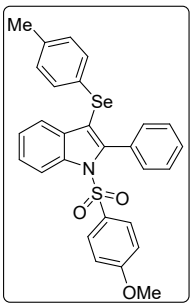


2-(4-(*tert*-butyl)phenyl)-1-tosyl-3-((3-(trifluoromethyl)phenyl)selanyl)-1*H*-indole (3ei):

GP-5 was carried out with *N*-(2-((4-(*tert*-butyl)phenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1e** (101 mg, 0.25 mmol), 1,2-bis(3-(trifluoromethyl)phenyl)diselane **2i** (112 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column

chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ei** (110 mg, 70%) as a brown solid, mp = 118–120 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1e**) = 0.25, R_f (**2i**) = 0.95, R_f (**3ei**) = 0.40, UV detection].

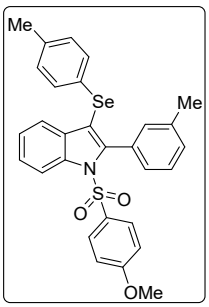
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3058, 2960, 1443, 1375, 1315, 1171, 1121, 1080, 744, 666 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.29 (d, J = 8.4 Hz, 1H), 7.36 – 7.25 (m, 4H), 7.24 – 7.14 (m, 5H), 7.14 – 7.08 (m, 2H), 7.04 – 6.91 (m, 4H), 2.21 (s, 3H), 1.27 (s, 9H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 152.5, 145.6, 145.1, 137.6, 135.2, 133.3, 132.5, 131.7, 131.4 (2 × Ar–CH), 131.3 (q, $J_{C–F}$ = 32.3 Hz), 129.5 (2 × Ar–CH), 129.3, 127.5, 127.0 (2 × Ar–CH), 125.9 (q, $J_{C–F}$ = 3.0 Hz), 125.8, 124.7, 124.2 (2 × Ar–CH), 123.7 (q, $J_{C–F}$ = 272.7 Hz), 122.9 (q, $J_{C–F}$ = 3.8 Hz), 120.9, 116.4, 109.1, 34.9, 31.4 (3 × CH_3), 21.7 ppm. **^{19}F NMR** (565 MHz, $CDCl_3$) δ = –62.80. **HRMS (ESI):** *m/z* calcd for $C_{32}H_{28}F_3NNaO_2SSe^+$ $[M+Na]^+$: 650.0850, found: 650.0828.



1-((4-methoxyphenyl)sulfonyl)-2-phenyl-3-(*p*-tolylselanyl)-1*H*-indole (3rb):

GP-5 was carried out with 4-methoxy-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **1r** (91 mg, 0.25 mmol), 1,2-di-*p*-tolyldiselane **2b** (85 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3rb** (116 mg, 87%) as a brown solid, mp = 130–132 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1r**) = 0.20, R_f (**2b**) = 0.95, R_f (**3rb**) = 0.30, UV detection].

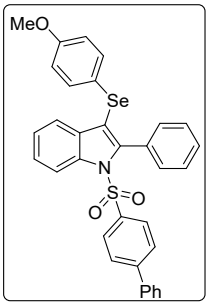
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3061, 2929, 1588, 1491, 1447, 1372, 1258, 1172, 1085, 1021, 759 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.41 (d, J = 8.4 Hz, 1H), 7.53 – 7.35 (m, 9H), 7.32 – 7.26 (m, 1H), 6.93 – 6.86 (m, 4H), 6.80 – 6.74 (m, 2H), 3.79 (s, 3H), 2.25 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 163.8, 144.7, 137.6, 136.0, 132.3, 131.7 (2 × Ar–CH), 131.1, 129.9 (2 × Ar–CH), 129.6 (2 × Ar–CH), 129.5, 129.2 (3 × Ar–CH), 127.9, 127.2 (2 × Ar–CH), 125.7, 124.7, 121.3, 116.4, 113.9 (2 × Ar–CH), 110.9, 55.6, 21.0 ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{23}NNaO_3SSe^+$ $[M+Na]^+$: 556.0456, found: 556.0442.



1-((4-methoxyphenyl)sulfonyl)-2-(*m*-tolyl)-3-(*p*-tolylselanyl)-1*H*-indole (3sb):

GP-5 was carried out with 4-methoxy-*N*-(2-(*m*-tolylethynyl)phenyl)benzenesulfonamide **1s** (94 mg, 0.25 mmol), 1,2-di-*p*-tolyldiselane **2b** (85 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3sb** (123 mg, 90%) as a brown solid, mp = 160–162 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1s**) = 0.20, R_f (**2b**) = 0.95, R_f (**3sb**) = 0.30, UV detection].

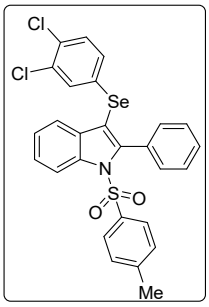
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3022, 2922, 1588, 1370, 1259, 1166, 1084, 1021, 796, 742, 676 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.41 (d, J = 8.4 Hz, 1H), 7.51 (dd, J = 7.8, 0.5 Hz, 1H), 7.45 – 7.38 (m, 3H), 7.34 – 7.27 (m, 3H), 7.17 (d, J = 7.4 Hz, 1H), 7.11 (s, 1H), 6.95 – 6.88 (m, 4H), 6.79 – 6.75 (m, 2H), 3.80 (s, 3H), 2.40 (s, 3H), 2.26 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 163.7, 144.8, 137.6, 136.6, 136.0, 132.5, 132.2, 130.9, 129.95, 129.9 (2 × Ar–CH), 129.8 (2 × Ar–CH), 129.7, 129.2 (2 × Ar–CH), 128.7, 128.0, 127.1, 125.6, 124.5, 121.2, 116.3, 113.9 (2 × Ar–CH), 110.7, 55.6, 21.5, 21.0 ppm. **HRMS (ESI):** *m/z* calcd for $C_{29}H_{25}NNaO_3SSe^+$ $[M+Na]^+$: 570.0613, found: 570.0595.



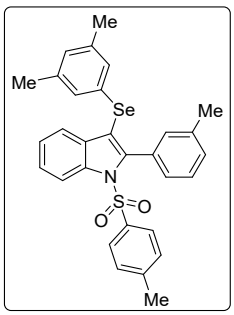
1-([1,1'-biphenyl]-4-ylsulfonyl)-3-((4-methoxyphenyl)selanyl)-2-phenyl-1*H*-indole (3qd):

GP-5 was carried out with *N*-(2-(phenylethynyl)phenyl)-[1,1'-biphenyl]-4-sulfonamide **1q** (103 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)diselane **2d** (93 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the

product **3qd** (120 mg, 80%) as a brown solid, mp = 122–124 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1q**) = 0.25, R_f (**2d**) = 0.80, R_f (**3qd**) = 0.30, UV detection].
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3056, 2942, 1588, 1485, 1444, 1374, 1242, 1174, 1085, 1022, 825, 757, 689 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.47 (dd, J = 8.4, 2.1 Hz, 1H), 7.60 – 7.39 (m, 16H), 7.37 – 7.29 (m, 1H), 6.98 (dd, J = 8.8, 1.6 Hz, 2H), 6.60 (dd, J = 8.7, 1.3 Hz, 2H), 3.61 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 158.7, 146.6, 144.1, 138.8, 137.6, 136.4, 132.4, 132.1 (2 × Ar–CH), 131.9 (2 × Ar–CH), 131.1, 129.3, 129.2 (2 × Ar–CH), 128.9, 127.5 (2 × Ar–CH), 127.32 (4 × Ar–CH), 127.3 (2 × Ar–CH), 125.8, 124.8, 121.4, 121.3, 116.4, 114.9 (2 × Ar–CH), 112.1, 55.1 ppm. **HRMS (ESI):** *m/z* calcd for $C_{33}H_{25}NNaO_3SSe^+$ $[M+Na]^+$: 618.0613, found: 618.0592.



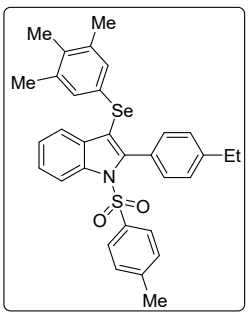
3-((3,4-dichlorophenyl)selanyl)-2-phenyl-1-tosyl-1*H*-indole (3aj):
GP-5 was carried out with 4-methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **1a** (87 mg, 0.25 mmol), 1,2-bis(3,4-dichlorophenyl)diselane **2j** (112 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3aj** (119 mg, 83%) as a white solid, mp = 150–152 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1a**) = 0.25, R_f (**2j**) = 0.80, R_f (**3aj**) = 0.30, UV detection].
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3056, 1446, 1371, 1175, 1084, 1023, 750, 695 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.43 (dd, J = 8.0, 1.2 Hz, 1H), 7.53 – 7.40 (m, 5H), 7.38 – 7.31 (m, 4H), 7.13 (d, J = 8.1 Hz, 2H), 7.10 (d, J = 8.4 Hz, 1H), 7.06 (d, J = 2.0 Hz, 1H), 6.76 (dd, J = 8.4, 2.1 Hz, 1H), 2.35 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 145.3, 145.2, 137.5, 135.1, 133.1, 131.7, 131.6 (2 × Ar–CH), 131.5, 130.7, 130.6, 130.3, 129.6 (2 × Ar–CH), 129.5, 128.6, 127.3 (2 × Ar–CH), 126.9 (2 × Ar–CH), 126.1, 124.9, 120.9, 116.3, 109.4, 21.7 ppm. **HRMS (ESI):** *m/z* calcd for $C_{27}H_{20}Cl_2NO_2SSe^+$ $[M+H]^+$: 571.9752, found: 571.9744.



3-((3,5-dimethylphenyl)selanyl)-2-(*m*-tolyl)-1-tosyl-1*H*-indole (3kk):

GP-5 was carried out with 4-methyl-*N*-(2-(*m*-tolylethynyl)phenyl)benzenesulfonamide **1k** (90 mg, 0.25 mmol), 1,2-bis(3,5-dimethylphenyl)diselane **2k** (92 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3kk** (115 mg, 85%) as a yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1k**) = 0.25, R_f (**2k**) = 0.80, R_f (**3kk**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3034, 1585, 1443, 1371, 1174, 1084, 1029, 798, 740, 666 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.45 (d, J = 8.4 Hz, 1H), 7.56 (d, J = 7.8 Hz, 1H), 7.49 – 7.44 (m, 1H), 7.36 – 7.30 (m, 3H), 7.21 (dd, J = 6.8, 0.5 Hz, 1H), 7.17 – 7.11 (m, 3H), 6.77 (s, 1H), 6.71 (s, 2H), 2.41 (s, 3H), 2.35 (s, 3H), 2.16 (s, 6H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 144.8, 144.7, 138.5 (2 × Ar–C), 137.4, 136.5, 135.3, 132.5, 132.2, 131.3, 130.9, 129.9, 129.4 (2 × Ar–CH), 128.7, 128.2, 127.5 (2 × Ar–CH), 127.0, 126.9 (2 × Ar–CH), 125.6, 124.5, 121.3, 116.0, 110.5, 21.6, 21.5, 21.2 (2 × CH_3) ppm. **HRMS (ESI):** *m/z* calcd for $C_{30}H_{28}NO_2SSe^+$ $[M+H]^+$: 546.1000, found: 546.1009.

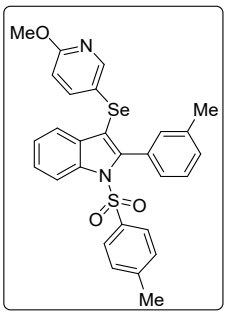


2-(4-ethylphenyl)-1-tosyl-3-((3,4,5-trimethylphenyl)selanyl)-1*H*-indole (3cl):

GP-5 was carried out with *N*-(2-((4-ethylphenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1c** (94 mg, 0.25 mmol), 1,2-dimesityldiselane **2l** (99 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05)

furnished the product **3cl** (143 mg, 82%) as a yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1c**) = 0.25, R_f (**2l**) = 0.80, R_f (**3cl**) = 0.30, UV detection].

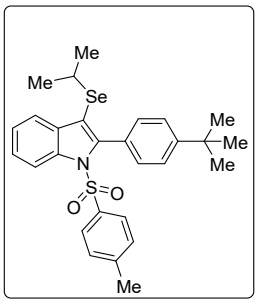
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3052, 1297, 1264, 1176, 1121, 732, 702, 663 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.30 (d, J = 8.4 Hz, 1H), 7.31 – 7.25 (m, 3H), 7.22 – 7.16 (m, 4H), 7.12 – 7.06 (m, 1H), 7.05 – 6.98 (m, 3H), 6.72 (s, 2H), 2.73 (q, J = 7.6 Hz, 2H), 2.26 (s, 3H), 2.17 (s, 3H), 2.09 (s, 6H), 1.31 (t, J = 7.6 Hz, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 144.9, 144.7, 141.8 (2 × Ar–C), 140.8, 137.8, 137.3, 134.9, 132.1, 131.3 (2 × Ar–CH), 129.3 (2 × Ar–CH), 128.9, 128.7 (2 × Ar–CH), 126.9, 126.8 (2 × Ar–CH), 126.7 (2 × Ar–CH), 125.1, 124.2, 120.5, 116.1, 112.4, 28.9, 23.9 (2 × CH_3), 21.6, 20.9, 15.4 ppm. **HRMS (ESI):** *m/z* calcd for $C_{32}H_{32}NO_2SSe^+$ $[M+H]^+$: 574.1313, found: 574.1316.



3-((6-methoxypyridin-3-yl)selanyl)-2-(*m*-tolyl)-1-tosyl-1*H*-indole (3km):

GP-5 was carried out with 4-methyl-*N*-(2-(*m*-tolylethynyl)phenyl)benzenesulfonamide **1k** (90 mg, 0.25 mmol), 1,2-bis(6-methoxypyridin-3-yl)diselane **2m** (94 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 6 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3km** (105 mg, 77%) as a yellow solid, mp = 178–180 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1k**) = 0.25, R_f (**2m**) = 0.60, R_f (**3km**) = 0.27, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2937, 1580, 1471, 1366, 1280, 1175, 1088, 1023, 747, 662 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.35 (d, J = 8.4 Hz, 1H), 7.91 (d, J = 2.3 Hz, 1H), 7.57 – 7.51 (m, 1H), 7.40 (ddd, J = 8.5, 7.3, 1.3 Hz, 1H), 7.35 – 7.27 (m, 5H), 7.23 (dd, J = 8.6, 2.4 Hz, 1H), 7.14 – 7.06 (m, 3H), 7.00 (s, 1H), 6.47 (d, J = 8.6 Hz, 1H), 3.84 (s, 3H), 2.38 (s, 3H), 2.33 (s, 3H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 163.4, 149.3, 145.0, 144.3, 142.3, 137.3, 136.8, 135.4, 132.5, 131.7, 130.9, 130.1, 129.5 (2 × Ar–CH), 128.9, 127.2, 127.0 (2 × Ar–CH), 125.7, 124.5, 120.9, 118.8, 116.1, 111.7, 110.6, 53.5, 21.7, 21.5 ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{25}N_2O_3SSe^+$ $[M+H]^+$: 549.0746, found: 549.0752.



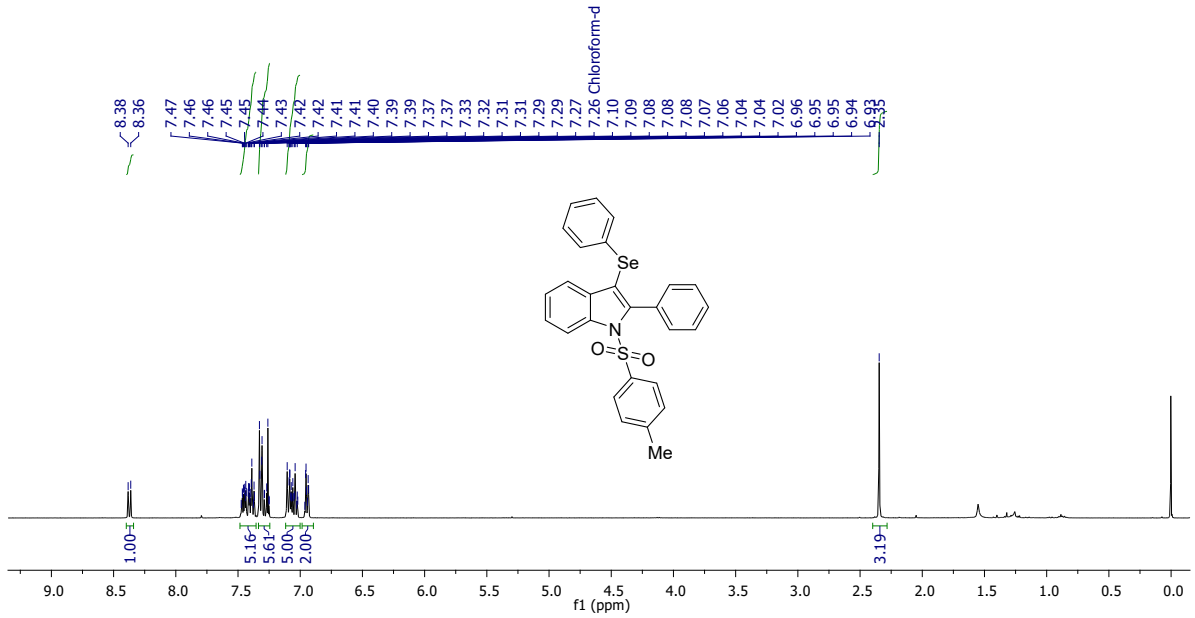
2-(4-(*tert*-butyl)phenyl)-3-(isopropylselanyl)-1-tosyl-1*H*-indole (3en):

GP-5 was carried out with *N*-(2-((4-(*tert*-butyl)phenyl)ethynyl)phenyl)-4-methylbenzenesulfonamide **1e** (101 mg, 0.25 mmol), 1,2-diisopropyldiselane **2n** (61 mg, 0.25 mmol), and $LiClO_4$ (64 mg, 0.1 M) in CH_3CN (6 mL) at 10 mA for 3 h 15 min. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3en** (112 mg, 86%) as a yellow liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1e**) = 0.25, R_f (**2n**) = 0.90, R_f (**3en**) = 0.60, UV detection].

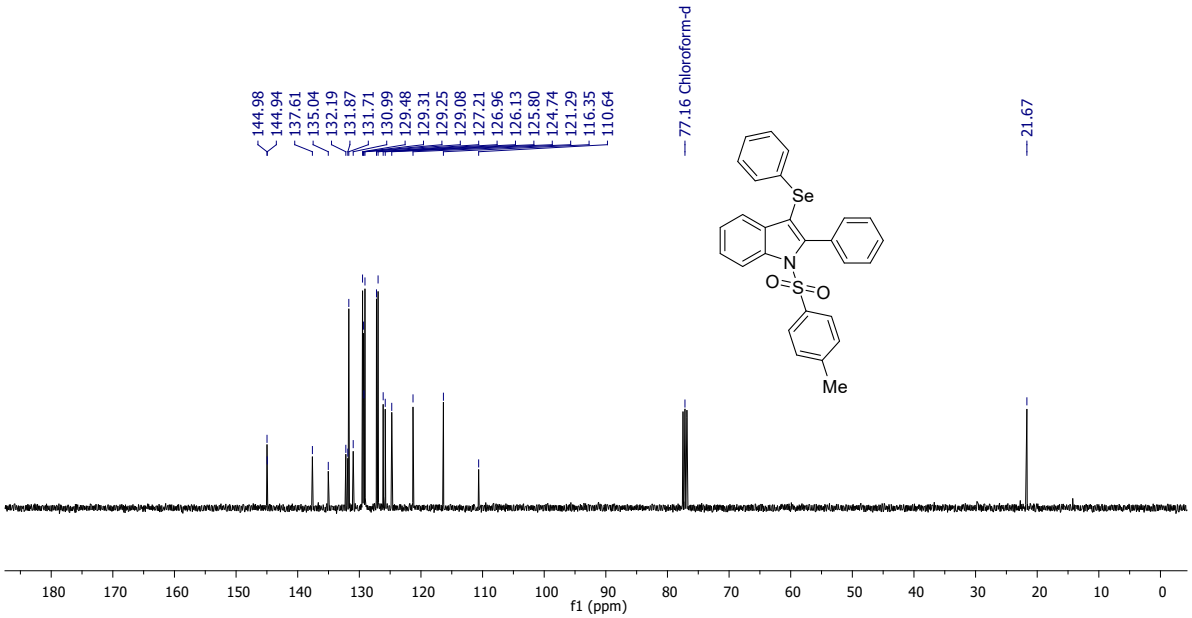
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3001, 2252, 1436, 1377, 1039, 919, 739 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.37 (d, J = 8.2 Hz, 1H), 7.73 – 7.65 (m, 1H), 7.45 – 7.39 (m, 3H), 7.35 (td, J = 7.6, 1.0 Hz, 1H), 7.30 (dd, J = 8.2, 1.6 Hz, 4H), 7.05 (d, J = 8.2 Hz, 2H), 3.21 – 3.08 (m, 1H), 2.30 (s, 3H), 1.43 (s, 9H), 1.13 (d, J = 6.8 Hz, 6H) ppm. **^{13}C{H} NMR (**101 MHz, $CDCl_3$) δ = 151.8, 144.7, 144.5, 137.3, 135.2, 133.2, 131.8 (2 × Ar–CH), 129.3 (2 × Ar–CH), 128.3, 126.9 (2 × Ar–CH), 125.3, 124.3, 123.9 (2 × Ar–CH), 121.3, 116.1, 110.8, 34.8, 33.9, 31.5 (3 × CH_3), 24.2 (2 × CH_3), 21.6 ppm. **HRMS (ESI):** *m/z* calcd for $C_{28}H_{32}NO_2SSe^+$ $[M+H]^+$: 526.1313, found: 526.1318.

NMR Spectra:

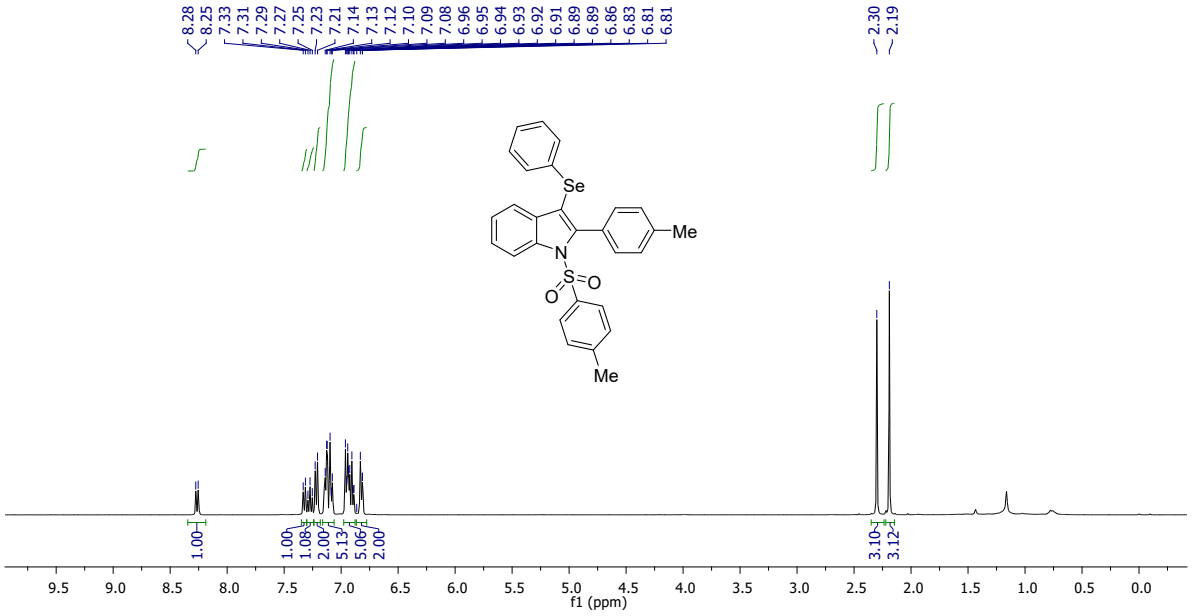
^{1}H-NMR (400 MHz) spectrum of **3aa** in $CDCl_3$



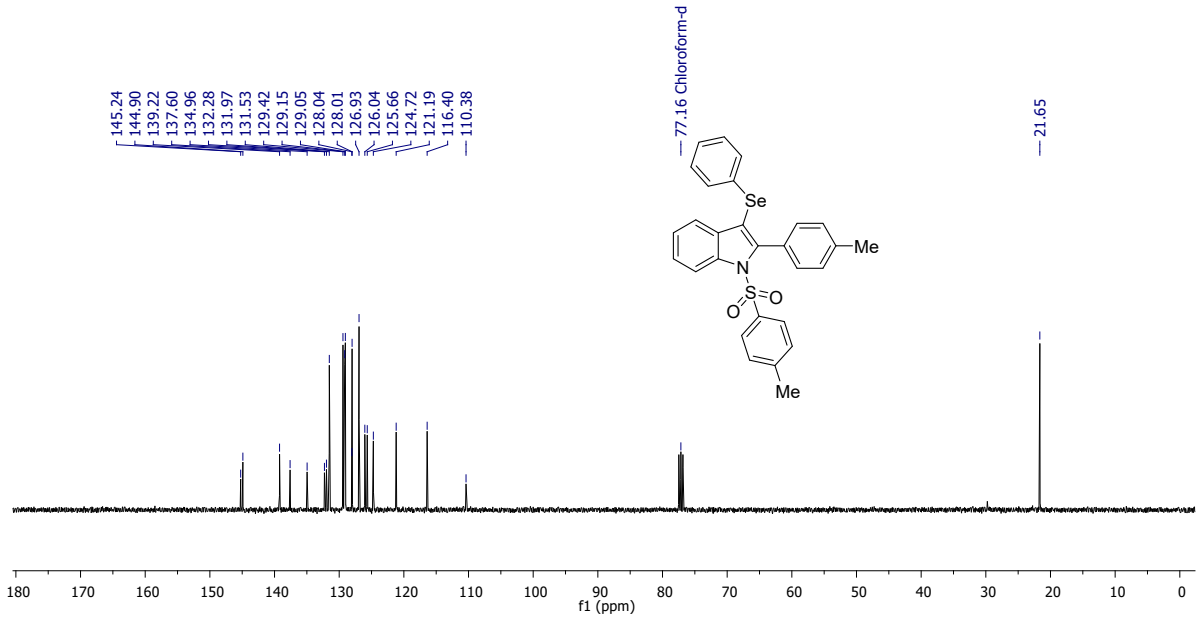
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3aa** in $CDCl_3$



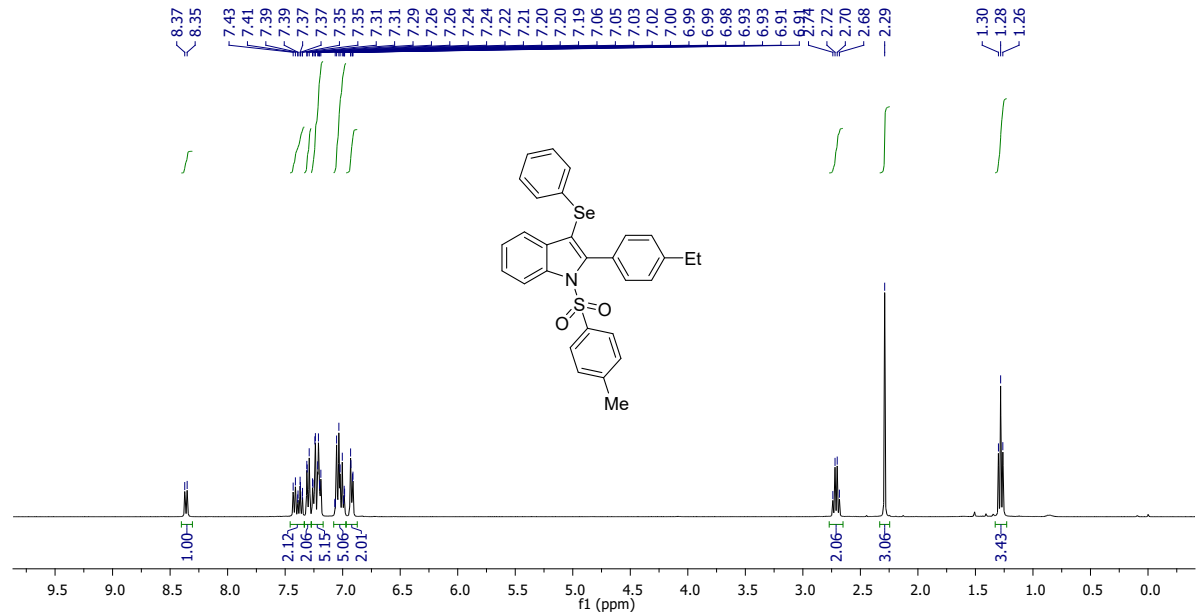
^{1}H-NMR (400 MHz) spectrum of **3ba** in $CDCl_3$



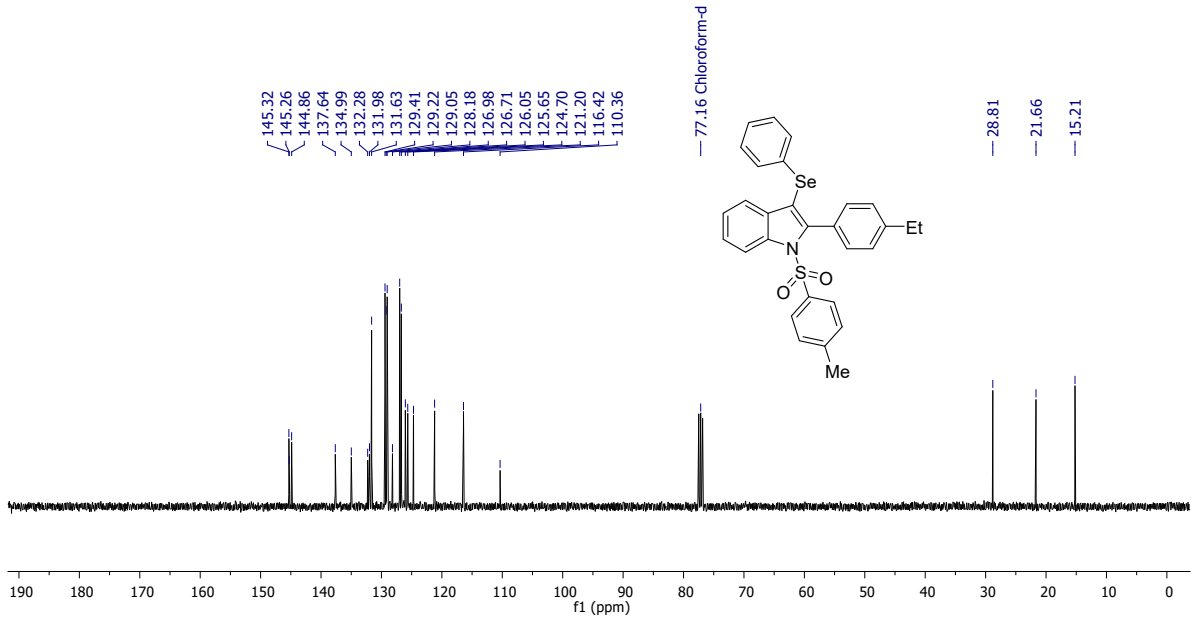
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ba** in $CDCl_3$



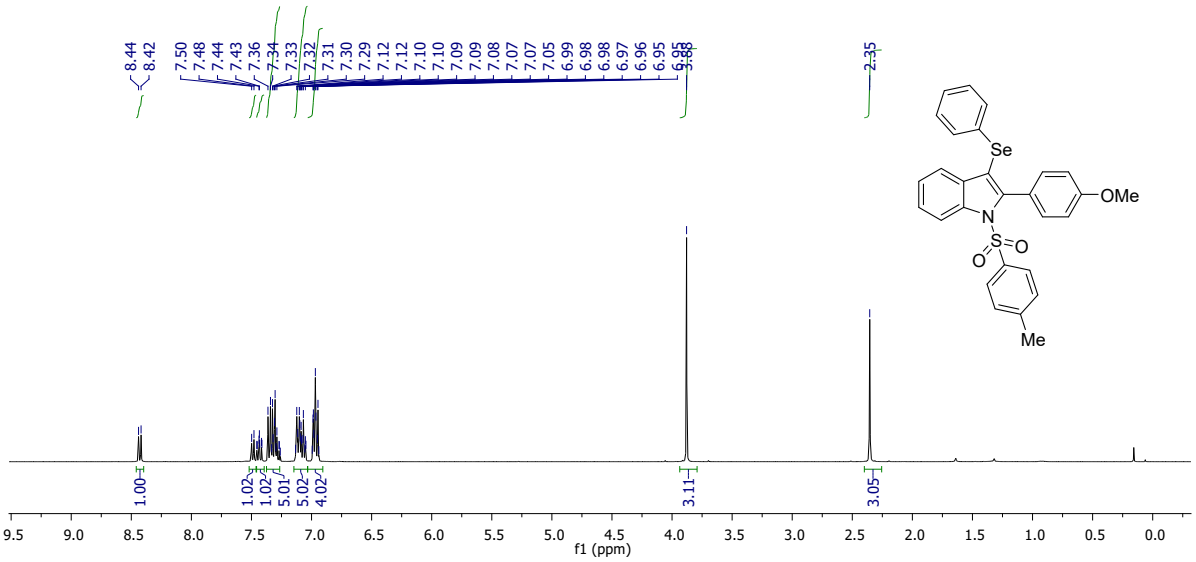
^{1}H-NMR (400 MHz) spectrum of **3ca** in $CDCl_3$



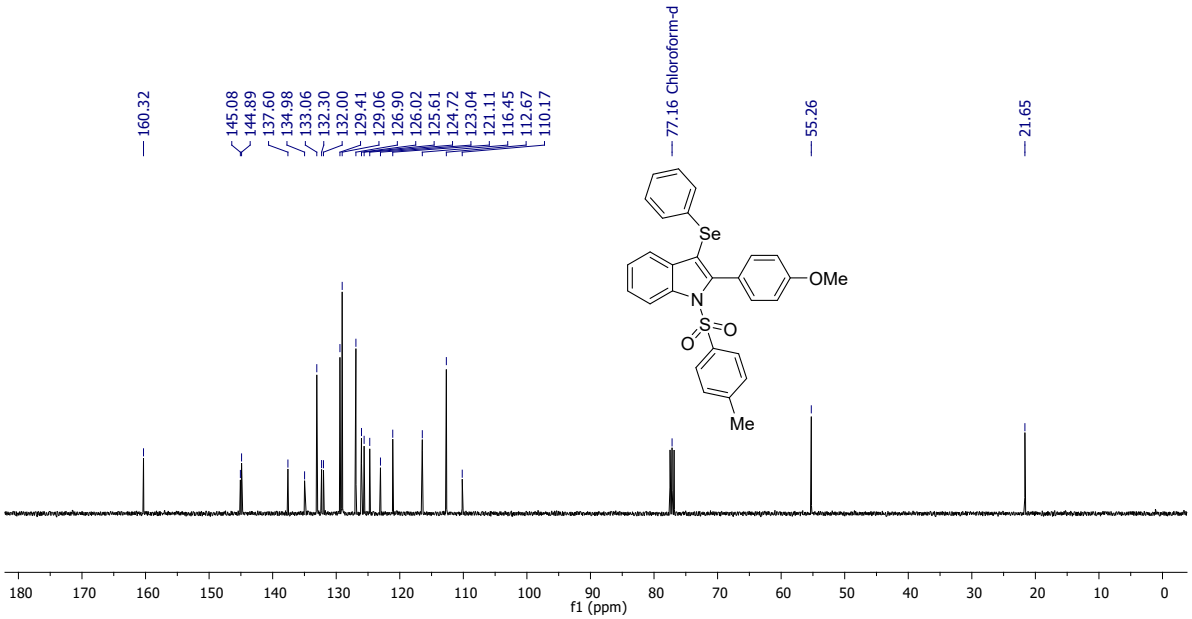
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ca** in $CDCl_3$



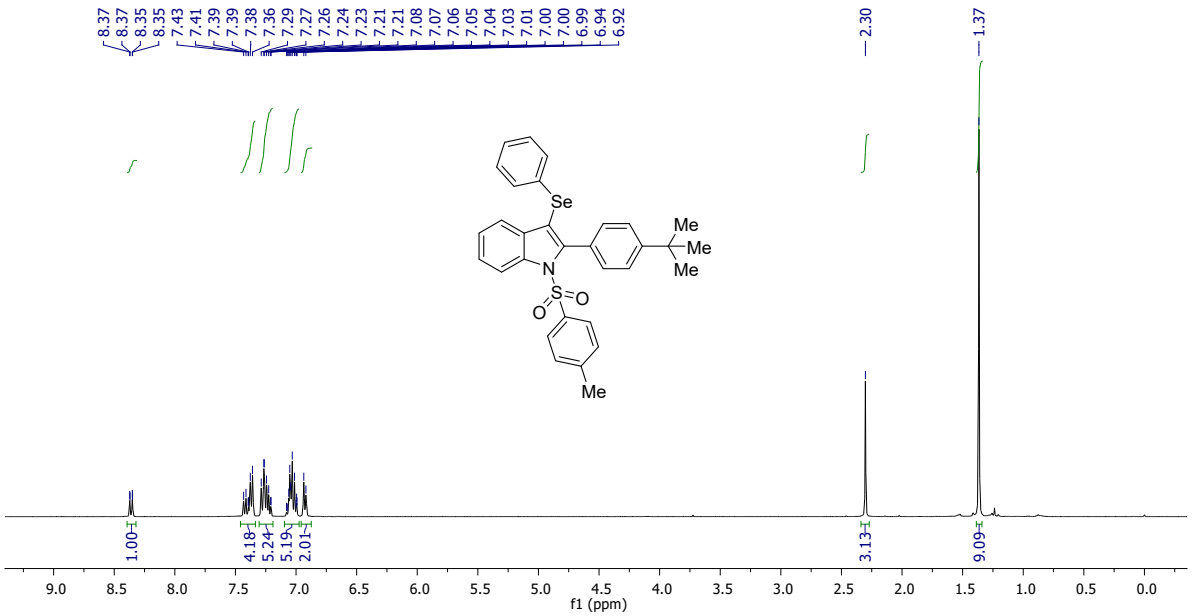
^{1}H-NMR (400 MHz) spectrum of **3da** in $CDCl_3$



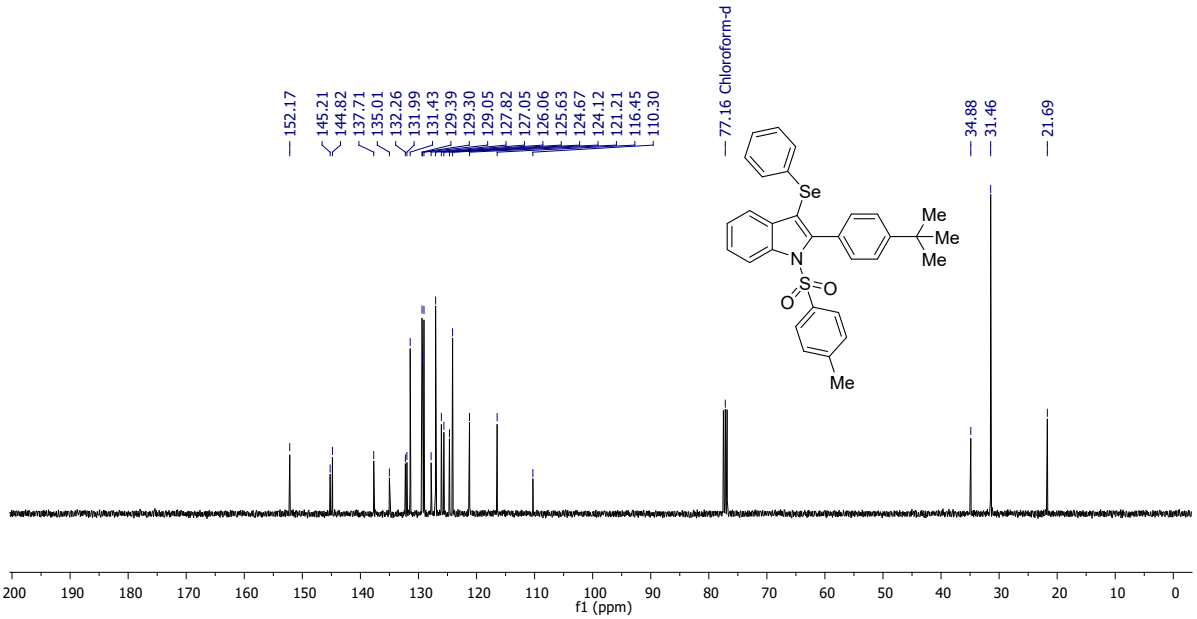
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3da** in $CDCl_3$



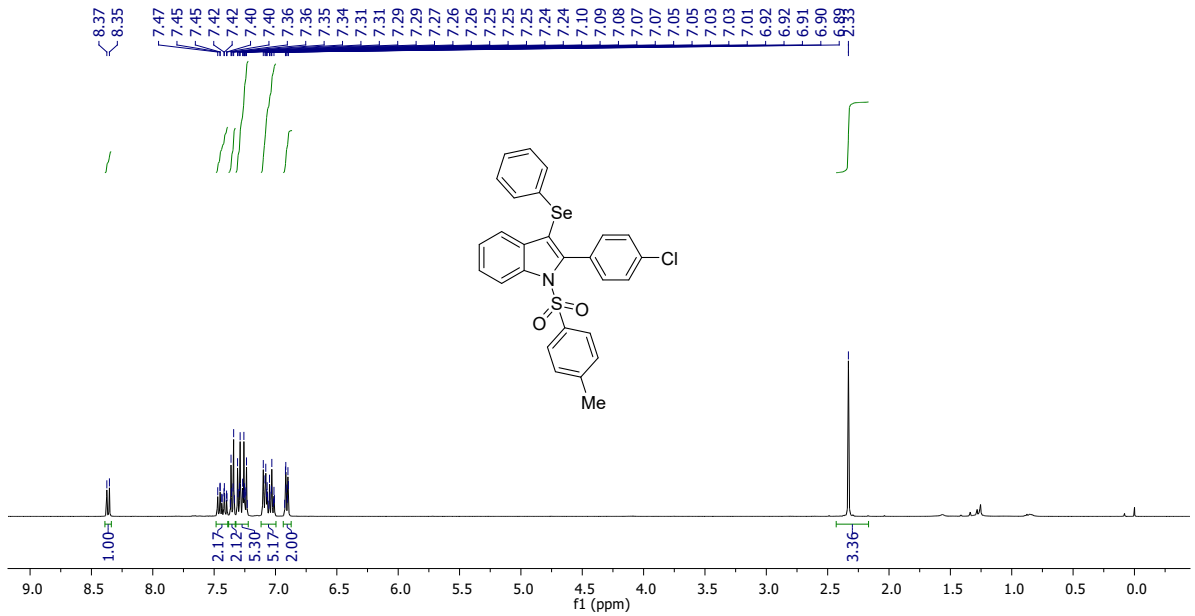
^{1}H-NMR (400 MHz) spectrum of **3ea** in $CDCl_3$



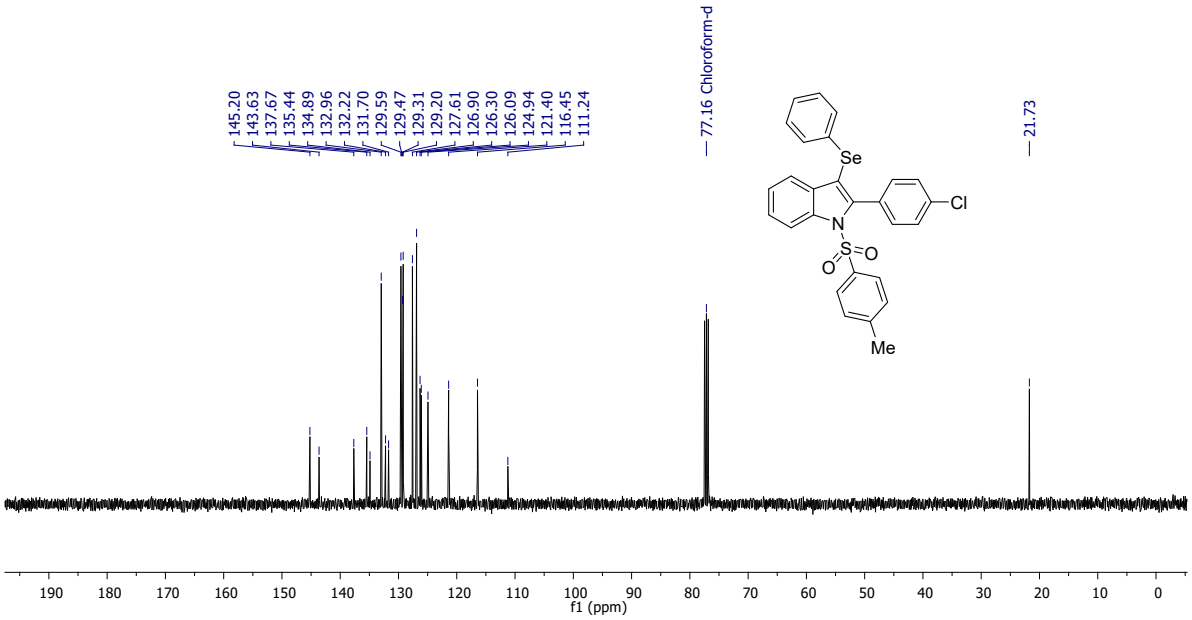
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ea** in $CDCl_3$



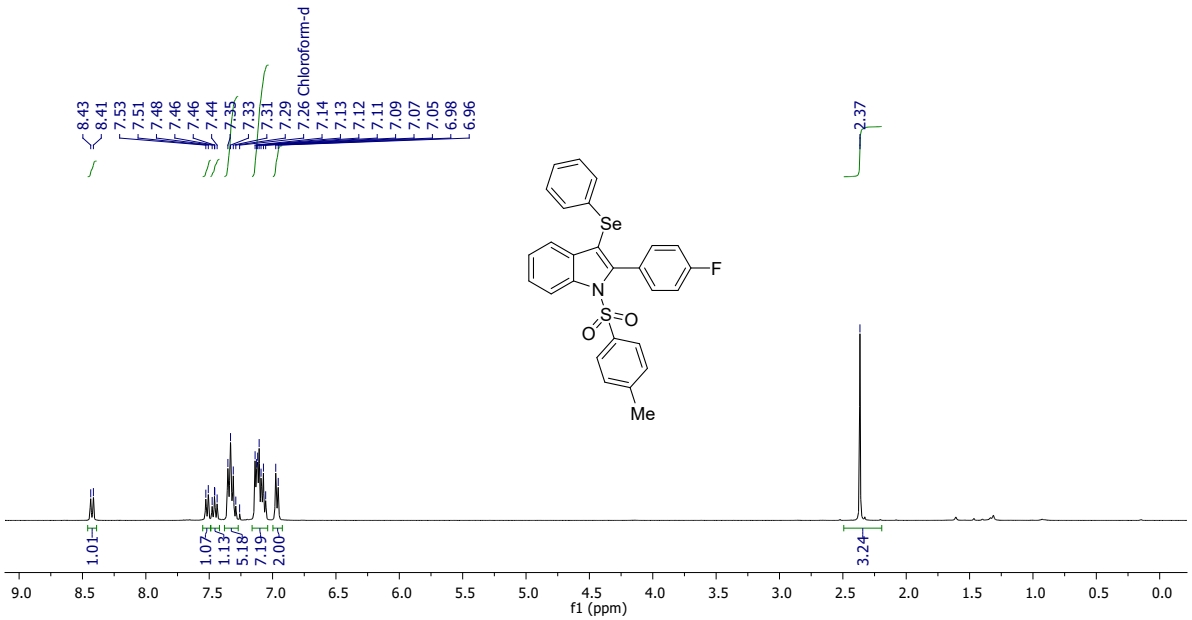
^{1}H-NMR (400 MHz) spectrum of **3fa** in $CDCl_3$



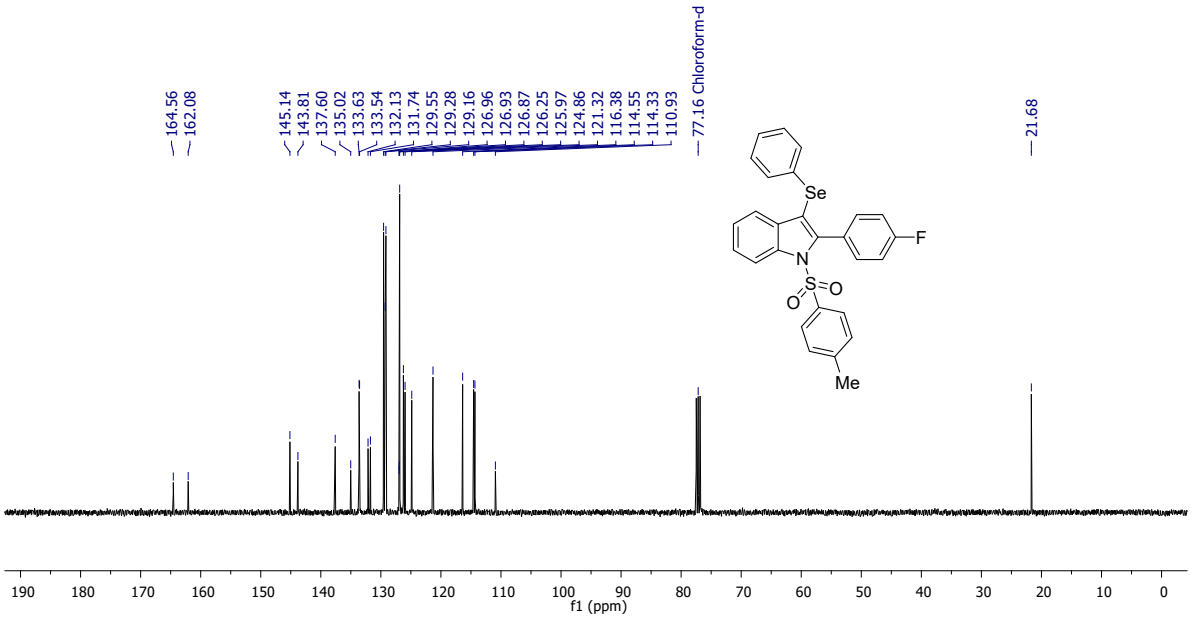
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3fa** in $CDCl_3$



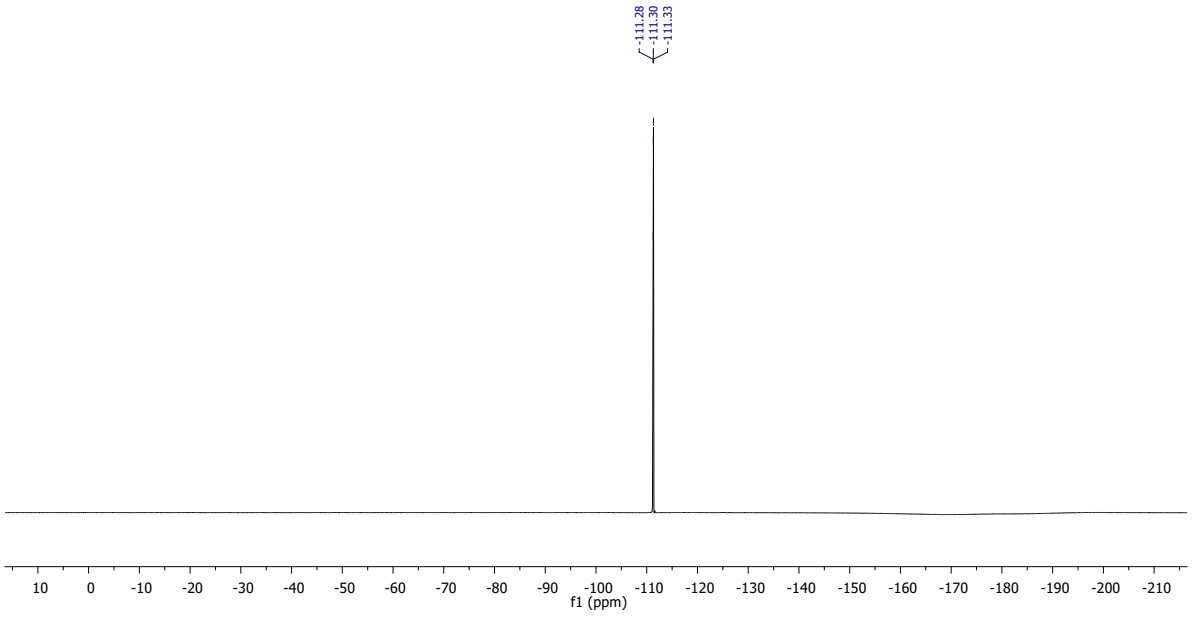
^{1}H-NMR (400 MHz) spectrum of **3ga** in $CDCl_3$



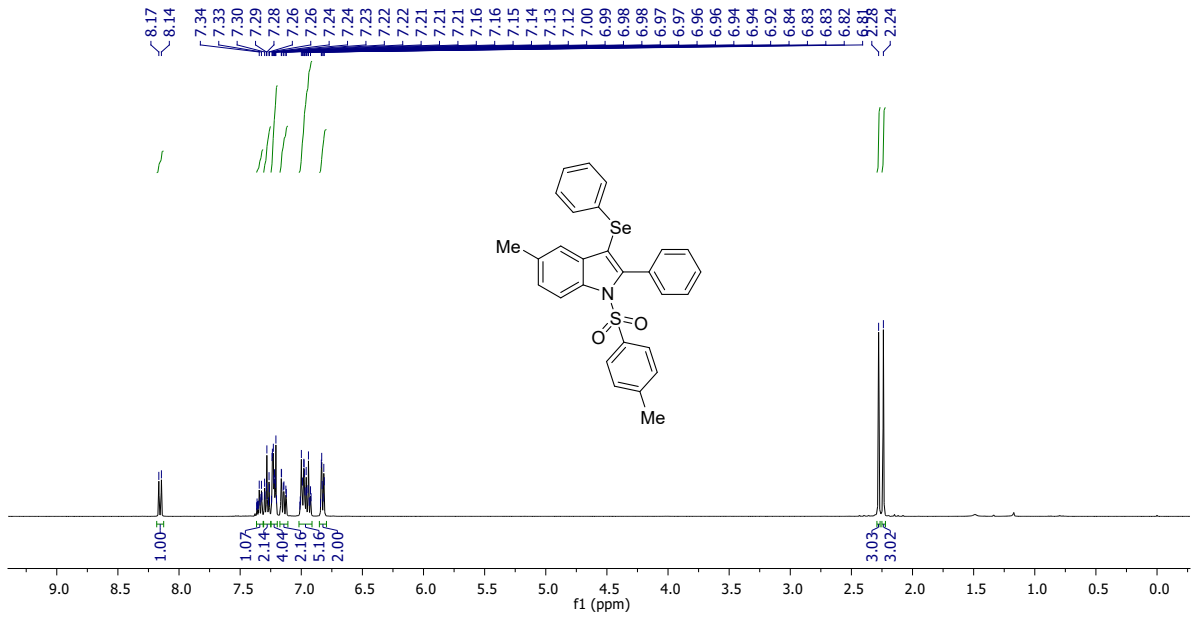
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ga** in $CDCl_3$



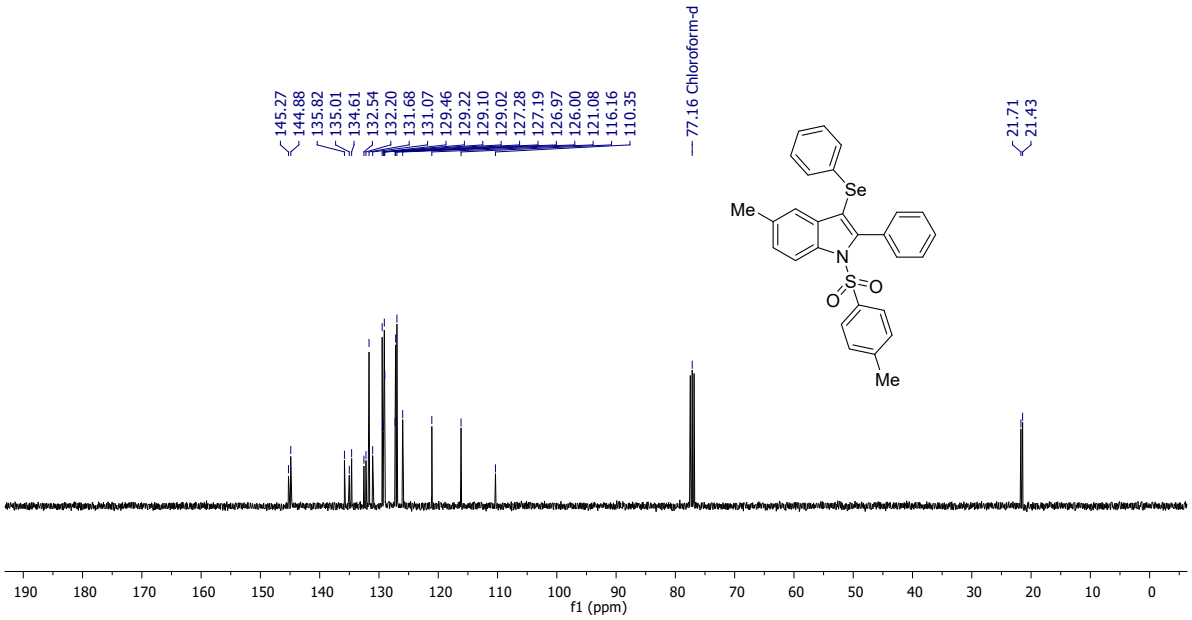
^{19}F NMR (565 MHz) spectrum of **3ga** in $CDCl_3$



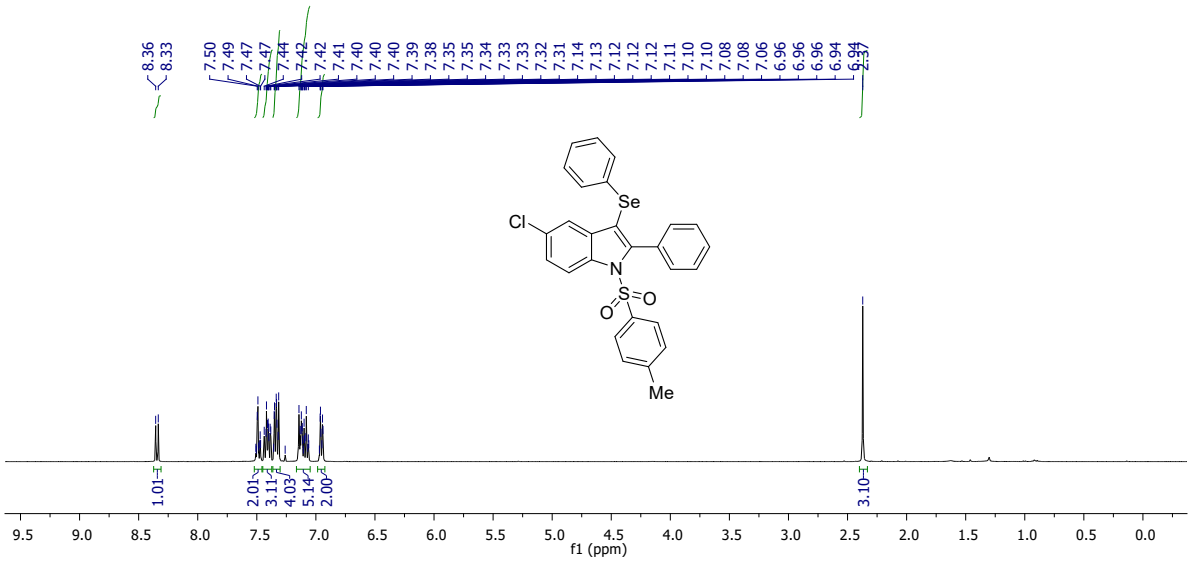
^{1}H-NMR (400 MHz) spectrum of **3ha** in $CDCl_3$



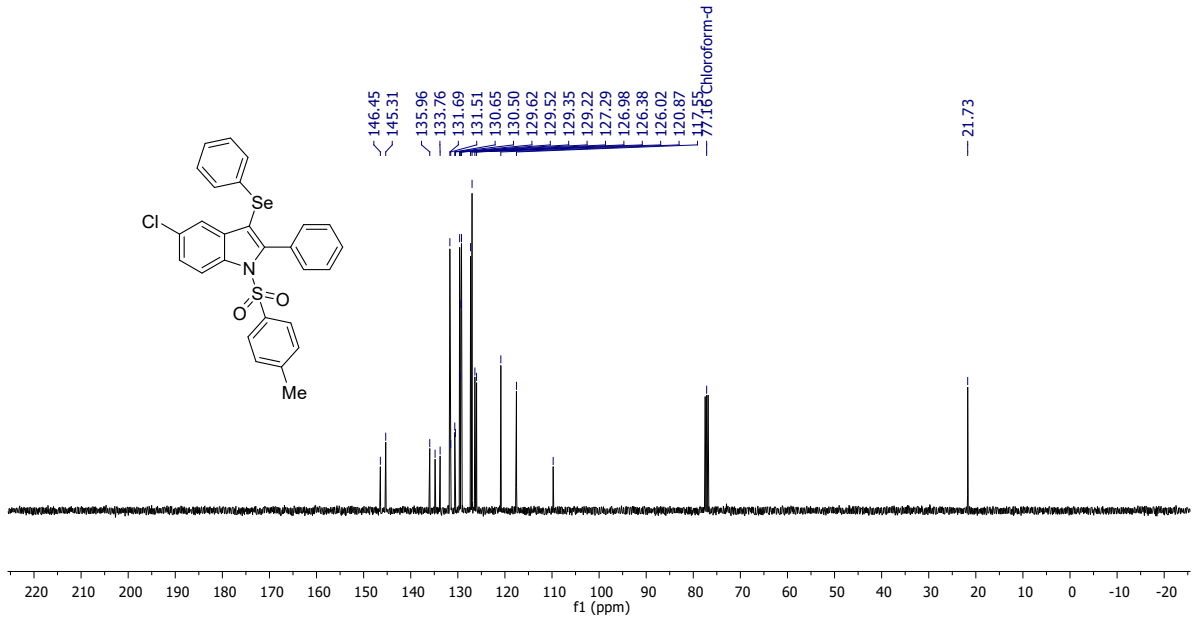
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ha** in $CDCl_3$



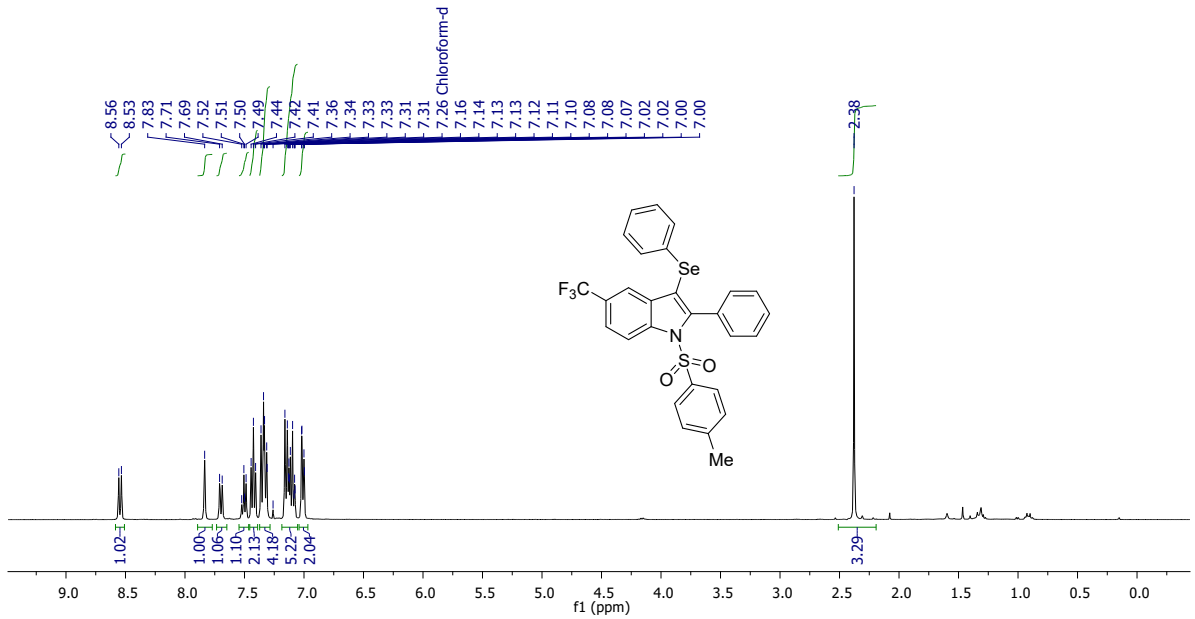
^{1}H-NMR (400 MHz) spectrum of **3ia** in $CDCl_3$



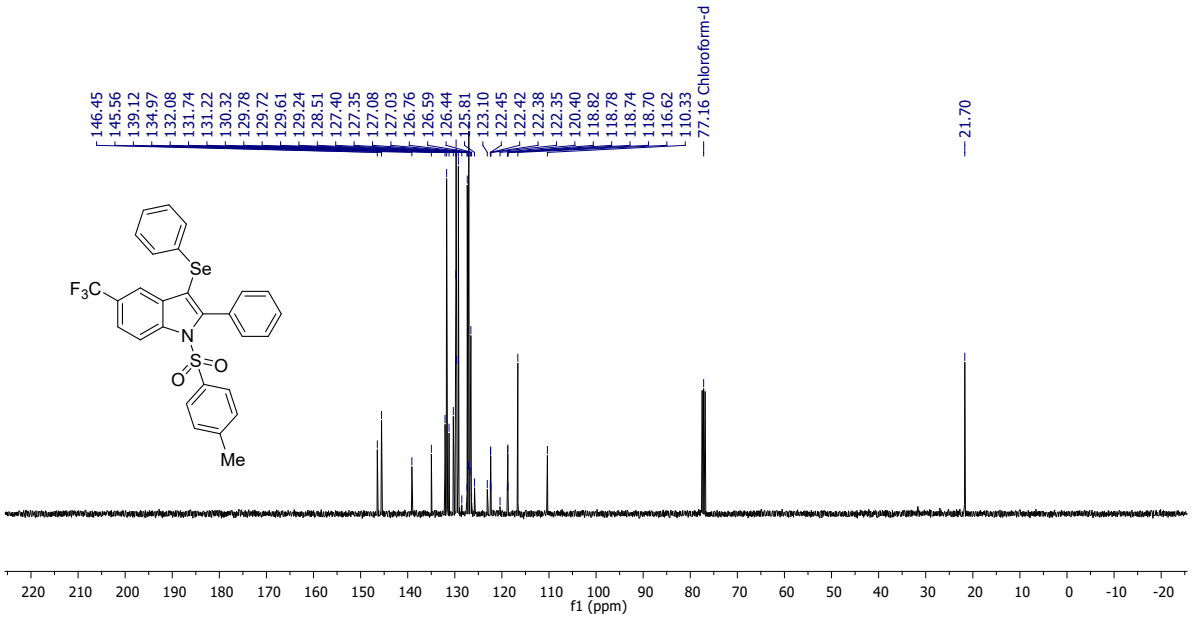
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ia** in $CDCl_3$



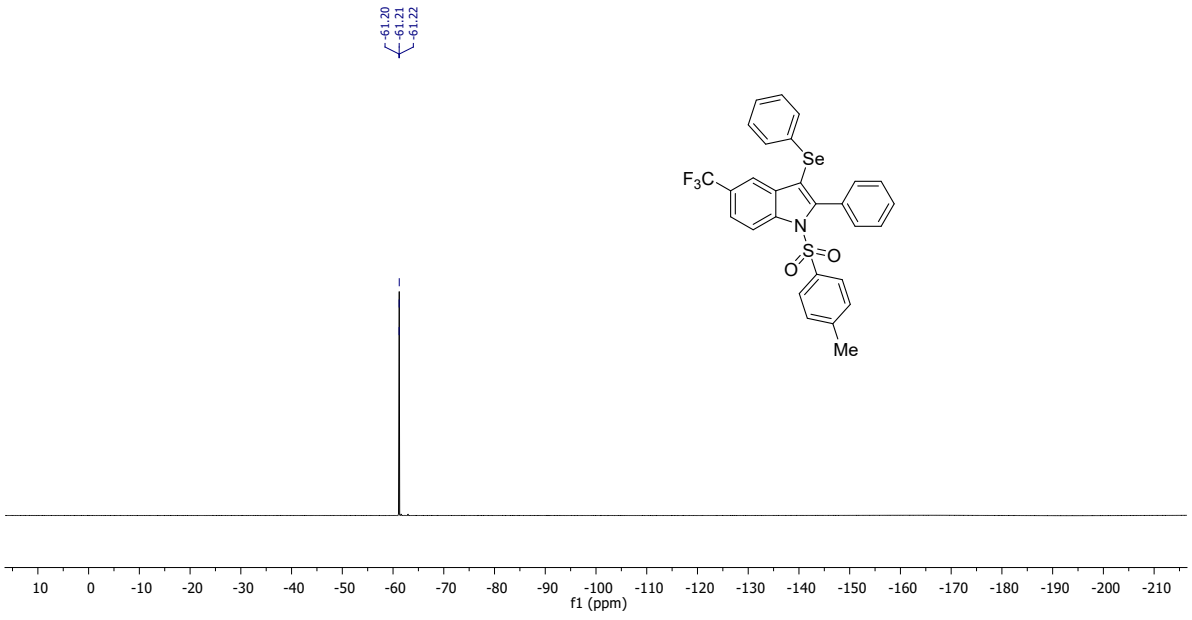
^{1}H-NMR (400 MHz) spectrum of **3ja** in $CDCl_3$



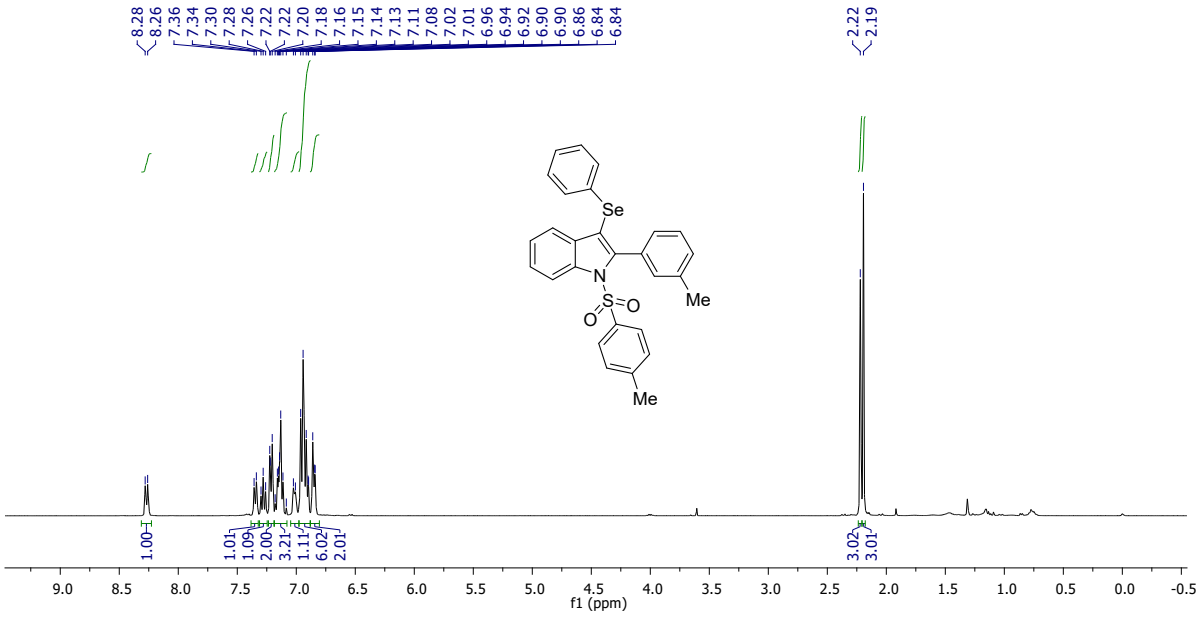
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ja** in $CDCl_3$



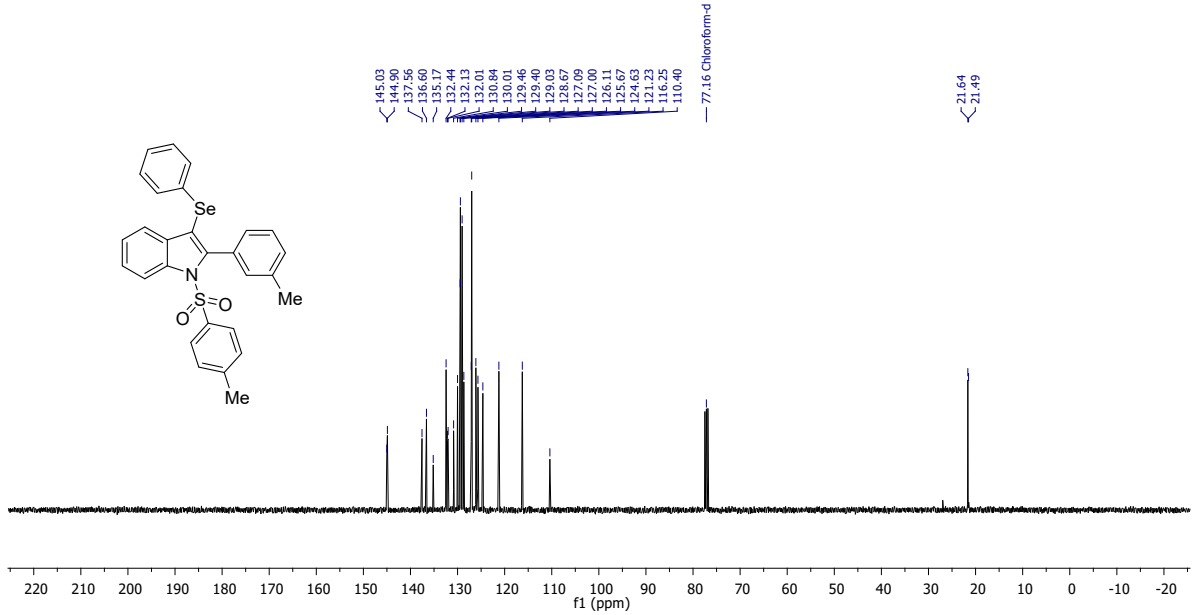
^{19}F NMR (565 MHz) spectrum of **3ja** in $CDCl_3$



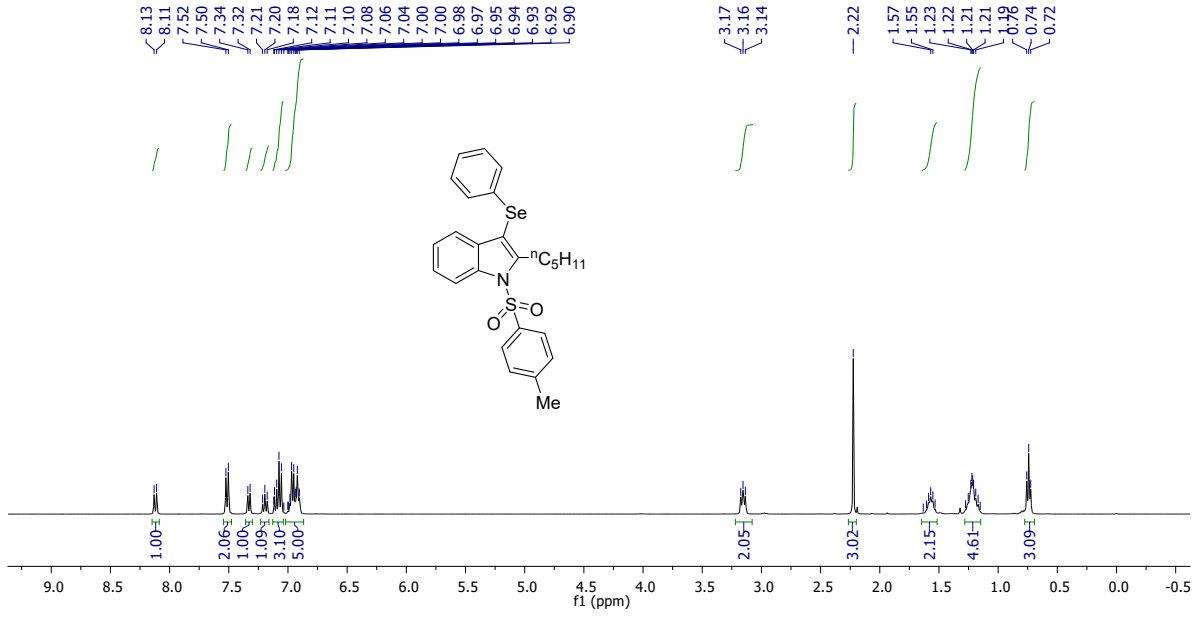
^{1}H-NMR (400 MHz) spectrum of **3ka** in $CDCl_3$



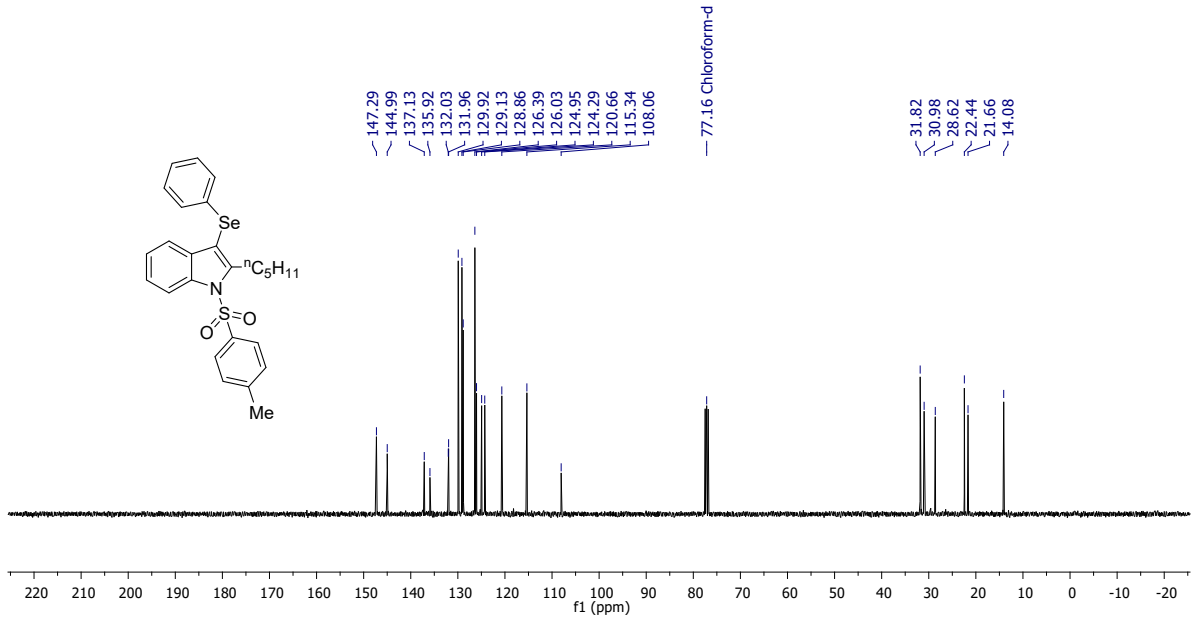
$^{13}C\{^{1}H\}$-NMR (101 MHz) spectrum of **3ka** in $CDCl_3$



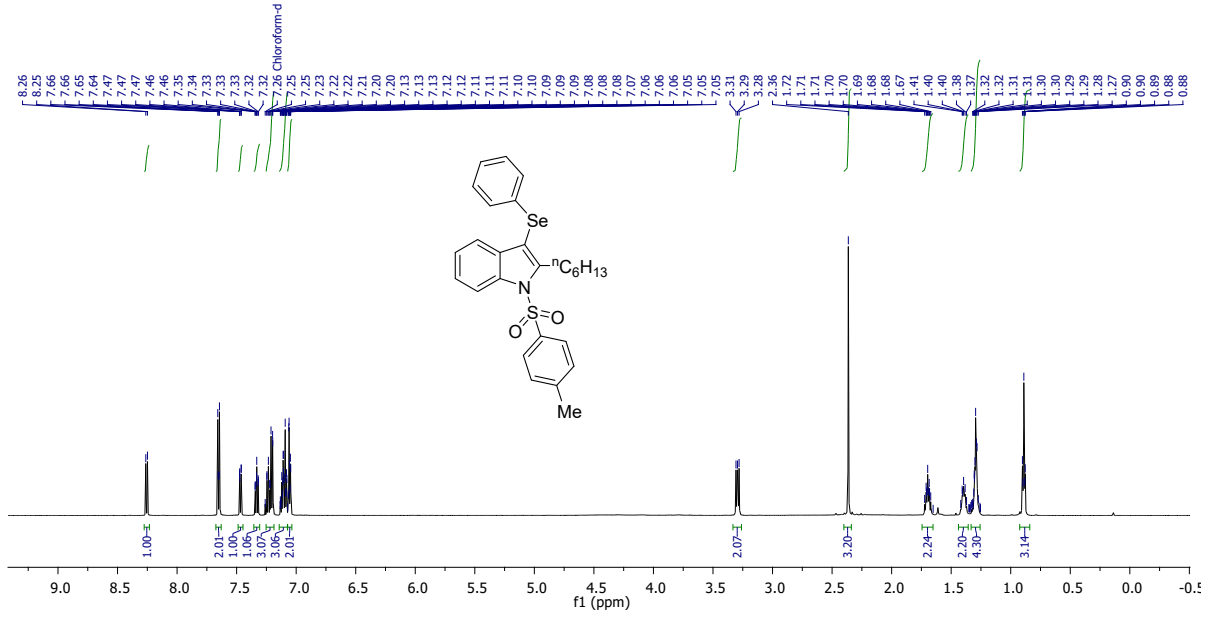
^{1}H-NMR (400 MHz) spectrum of **3la** in $CDCl_3$



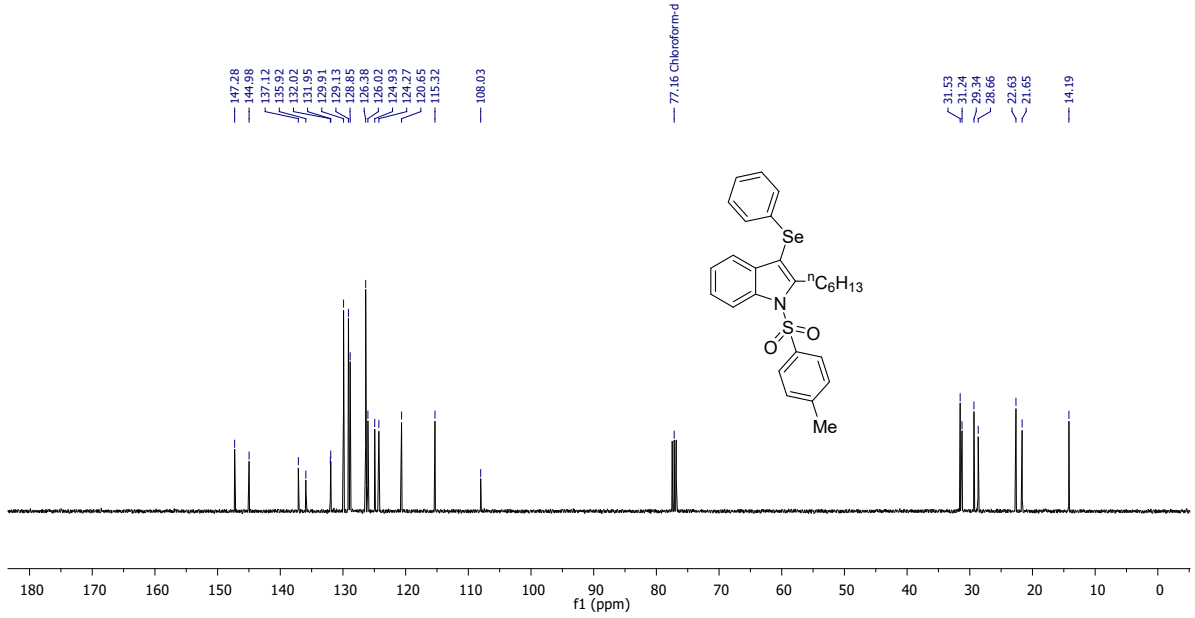
$^{13}C\{^{1}H\}$-NMR (101 MHz) spectrum of **3la** in $CDCl_3$



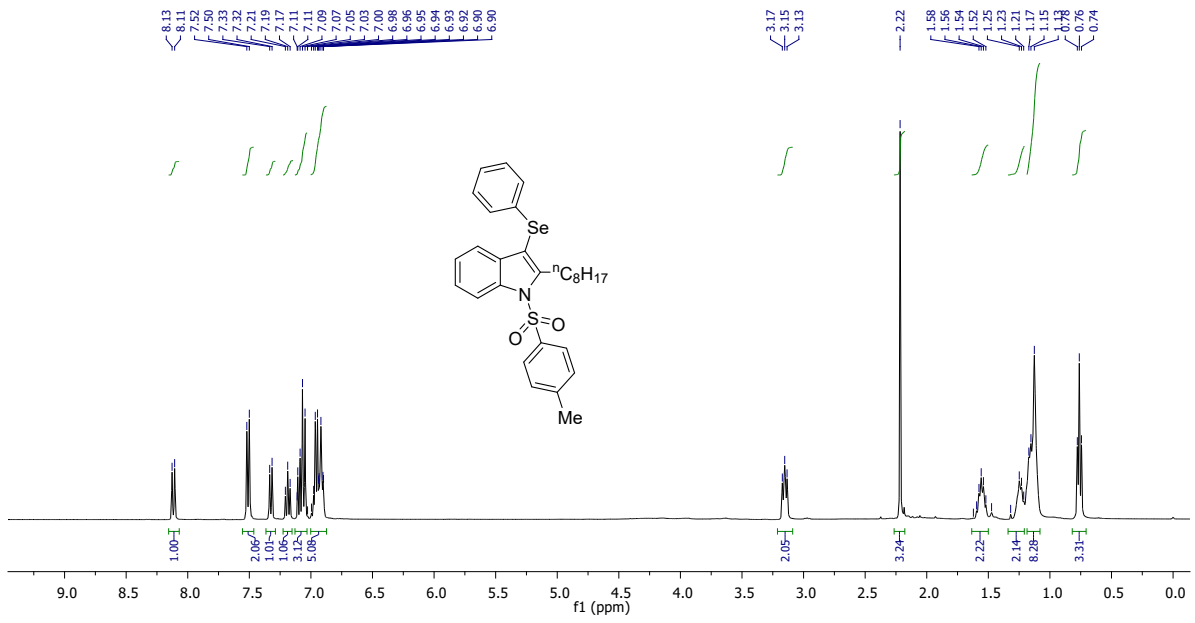
^{1}H-NMR (600 MHz) spectrum of **3ma** in $CDCl_3$



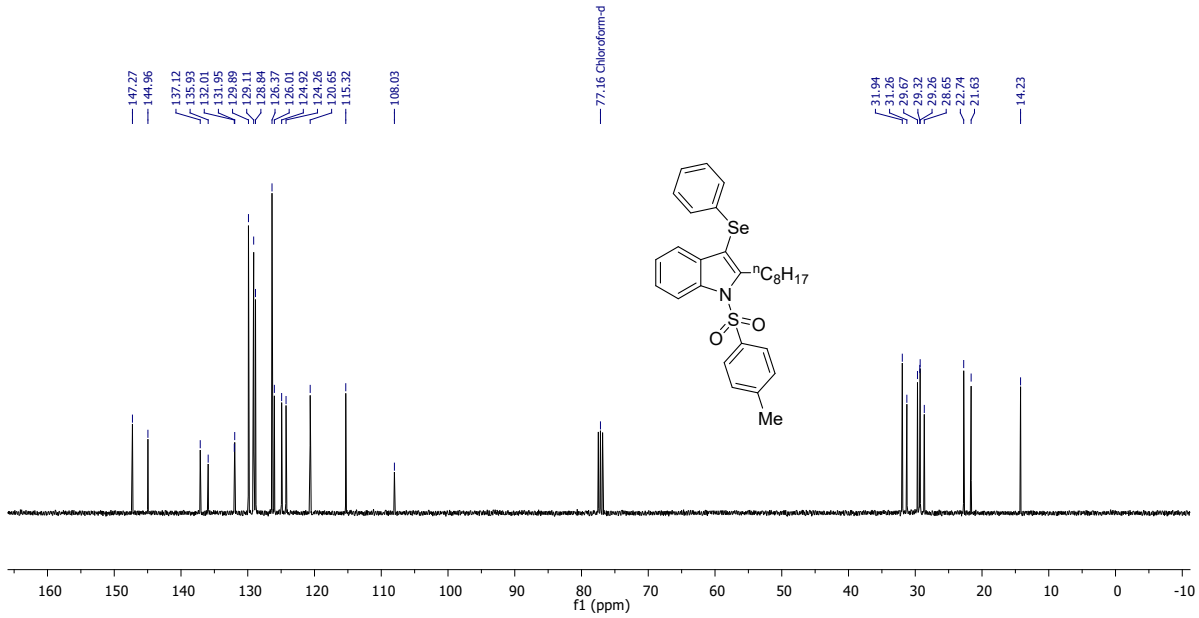
$^{13}C\{^{1}H\}$-NMR (101 MHz) spectrum of **3ma** in $CDCl_3$



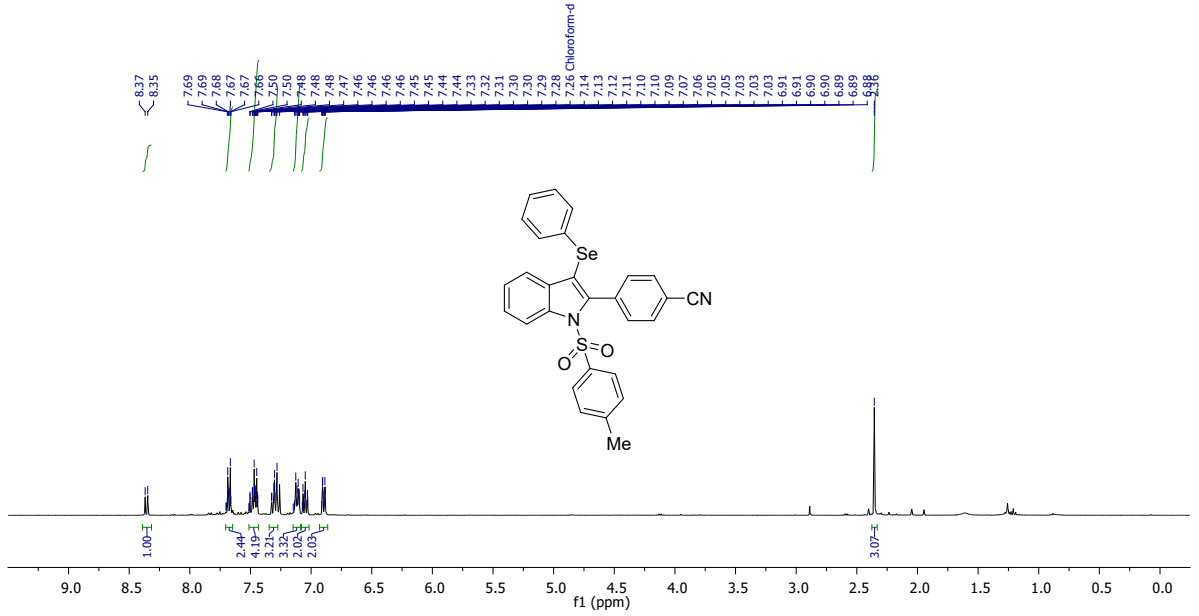
^{1}H-NMR (400 MHz) spectrum of **3na** in $CDCl_3$



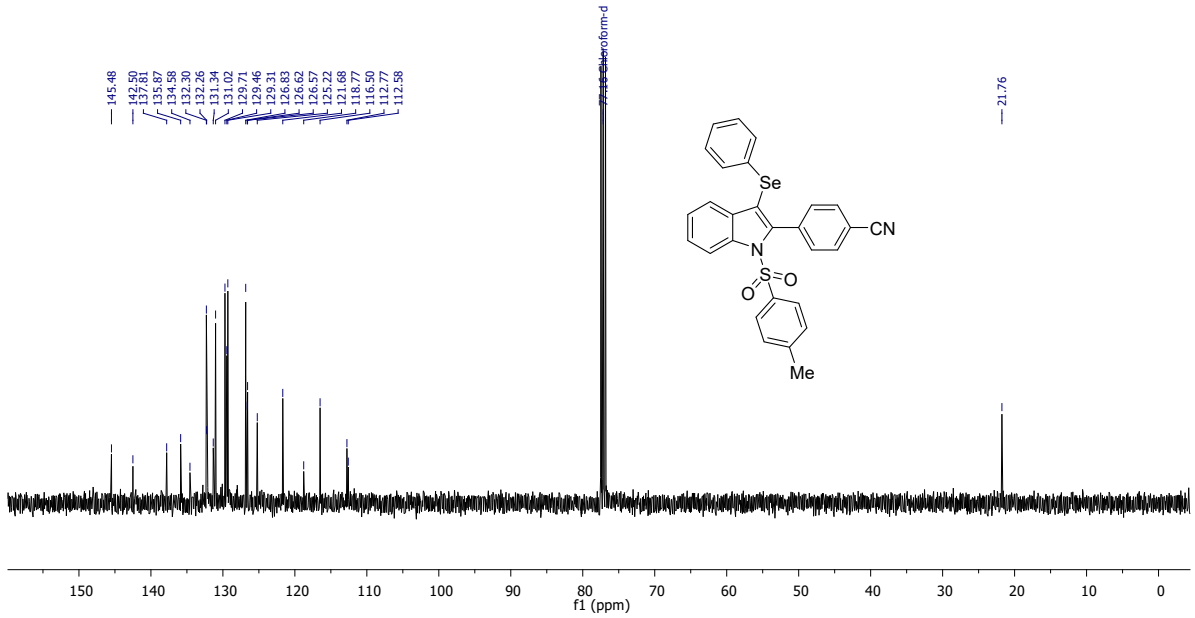
$^{13}C\{^{1}H\}$-NMR (101 MHz) spectrum of **3na** in $CDCl_3$



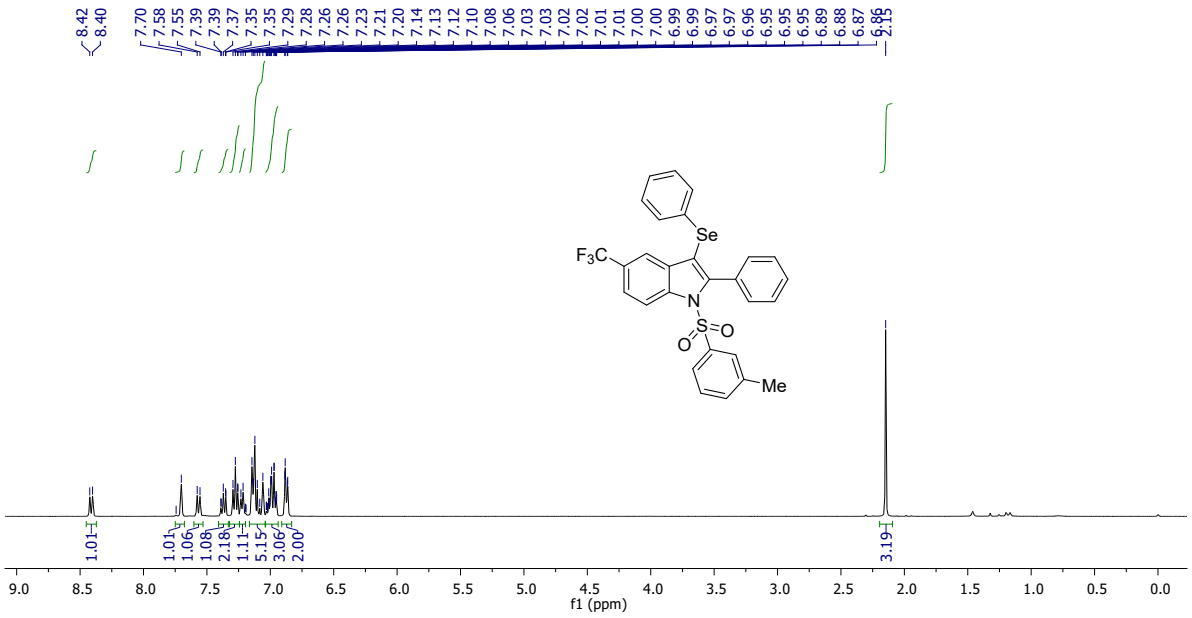
^{1}H-NMR (400 MHz) spectrum of **3oa** in $CDCl_3$



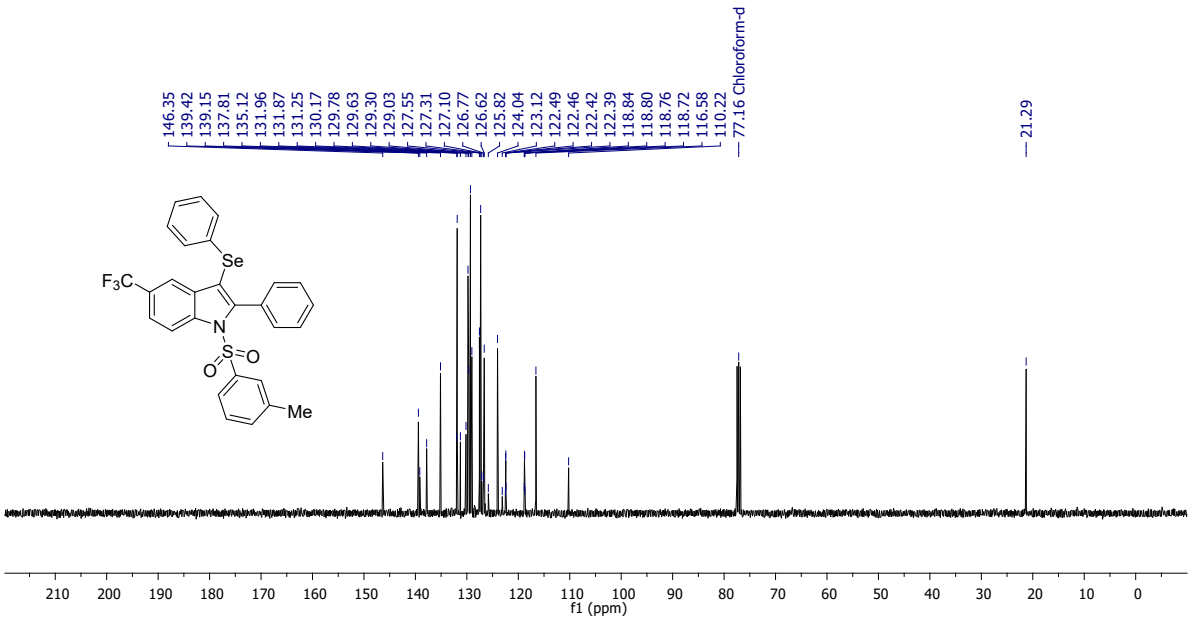
$^{13}C\{^{1}H\}$-NMR (101 MHz) spectrum of **3oa** in $CDCl_3$



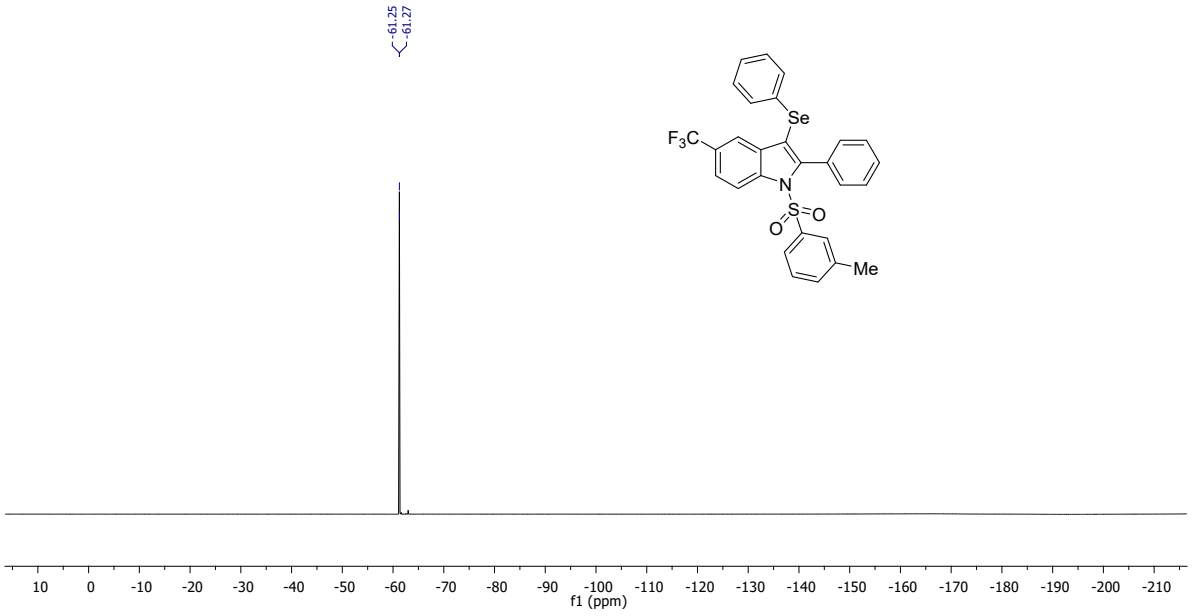
^{1}H-NMR (400 MHz) spectrum of **3pa** in $CDCl_3$



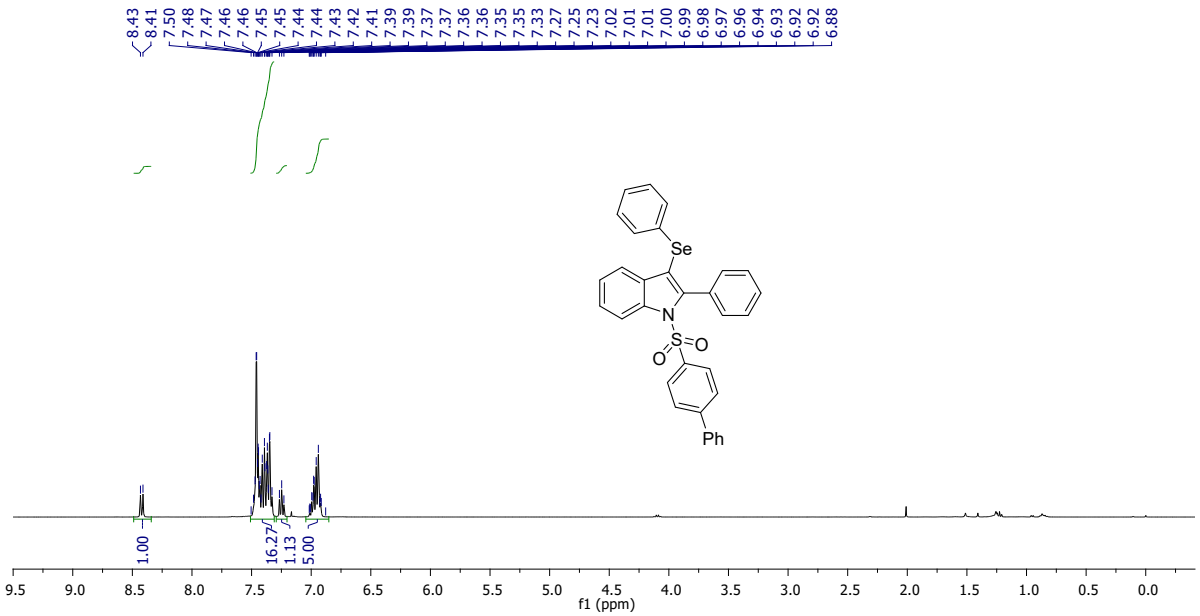
$^{13}C\{^{1}H\}$-NMR (101 MHz) spectrum of **3pa** in $CDCl_3$



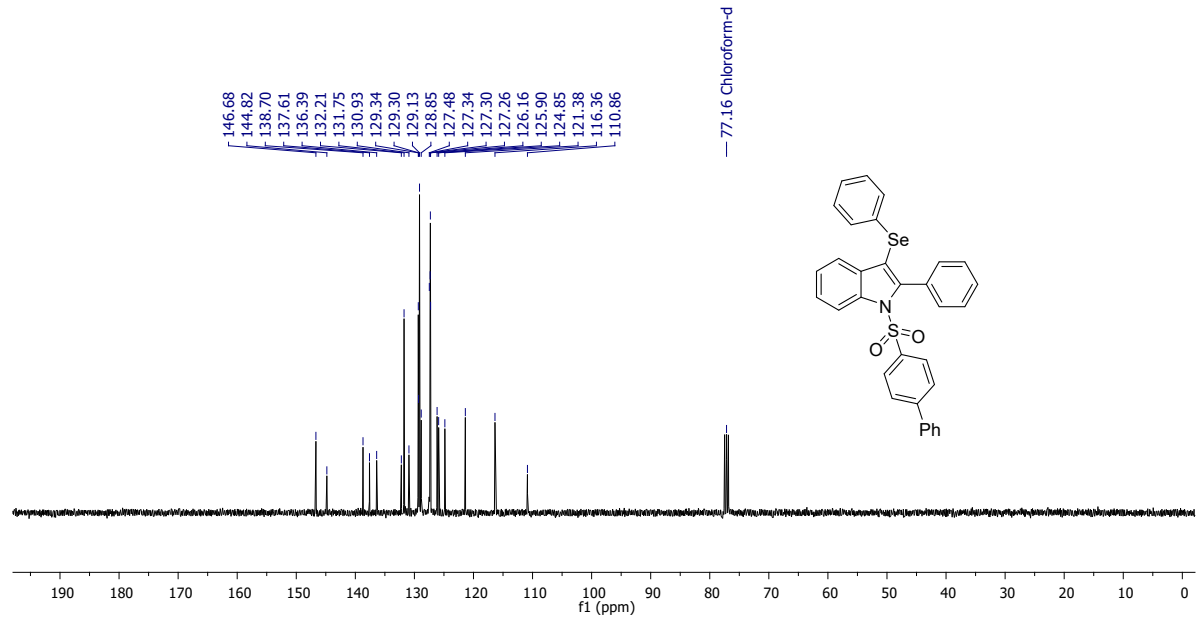
^{19}F NMR (565 MHz) spectrum of **3pa** in $CDCl_3$



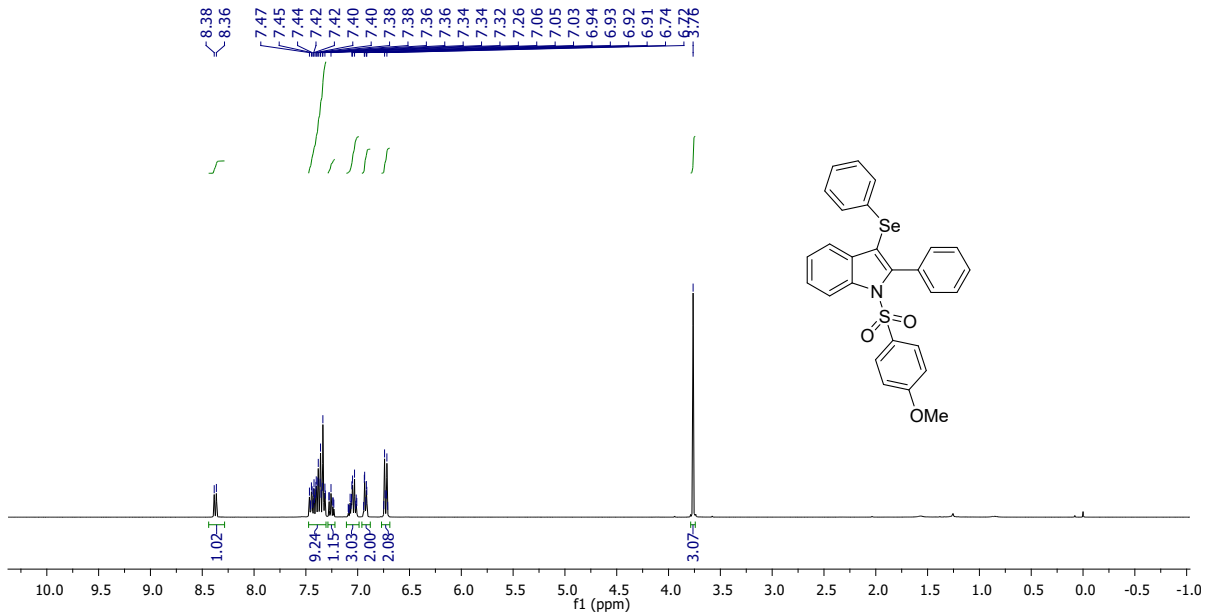
^{1}H-NMR (400 MHz) spectrum of **3qa** in $CDCl_3$



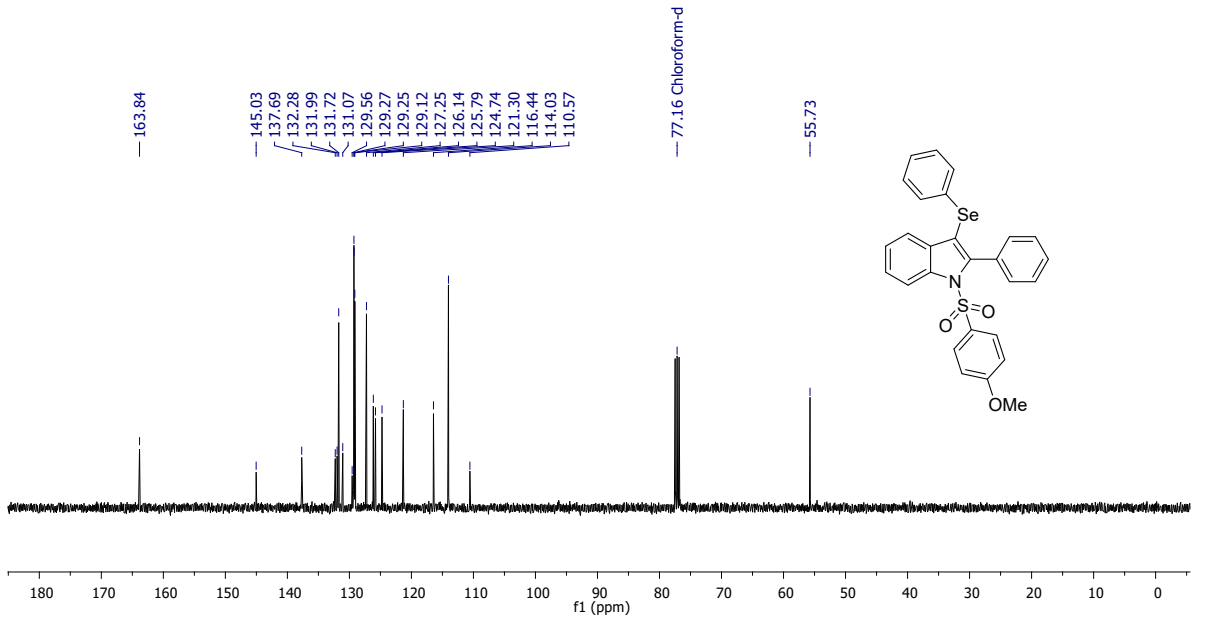
$^{13}C\{^{1}H\}$-NMR (101 MHz) spectrum of **3qa** in $CDCl_3$



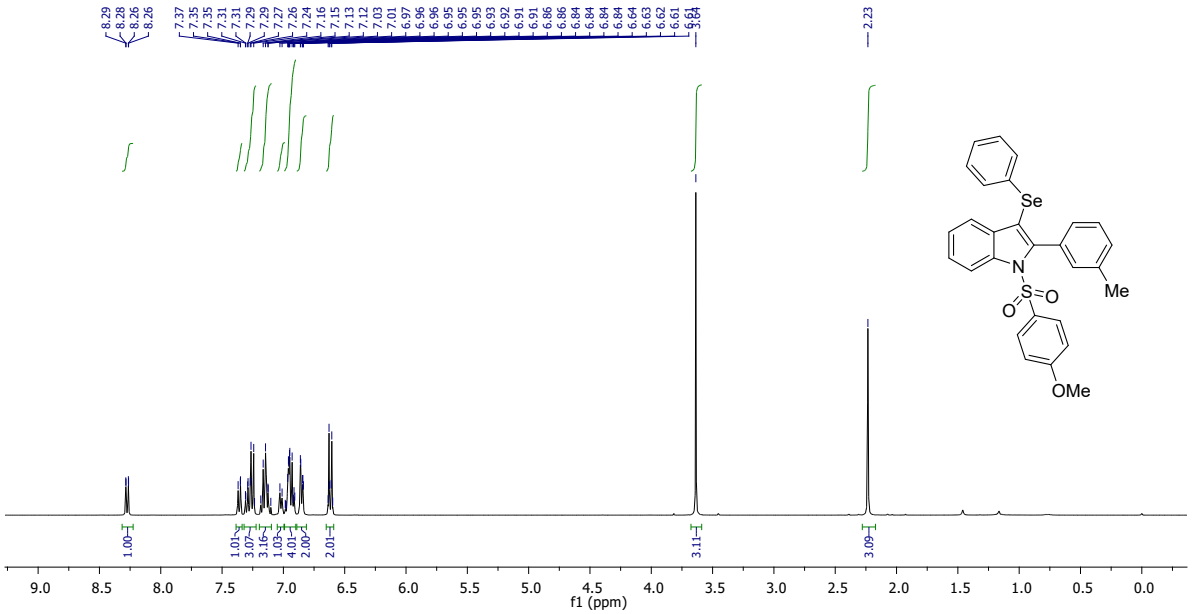
^{1}H-NMR (400 MHz) spectrum of **3ra** in $CDCl_3$



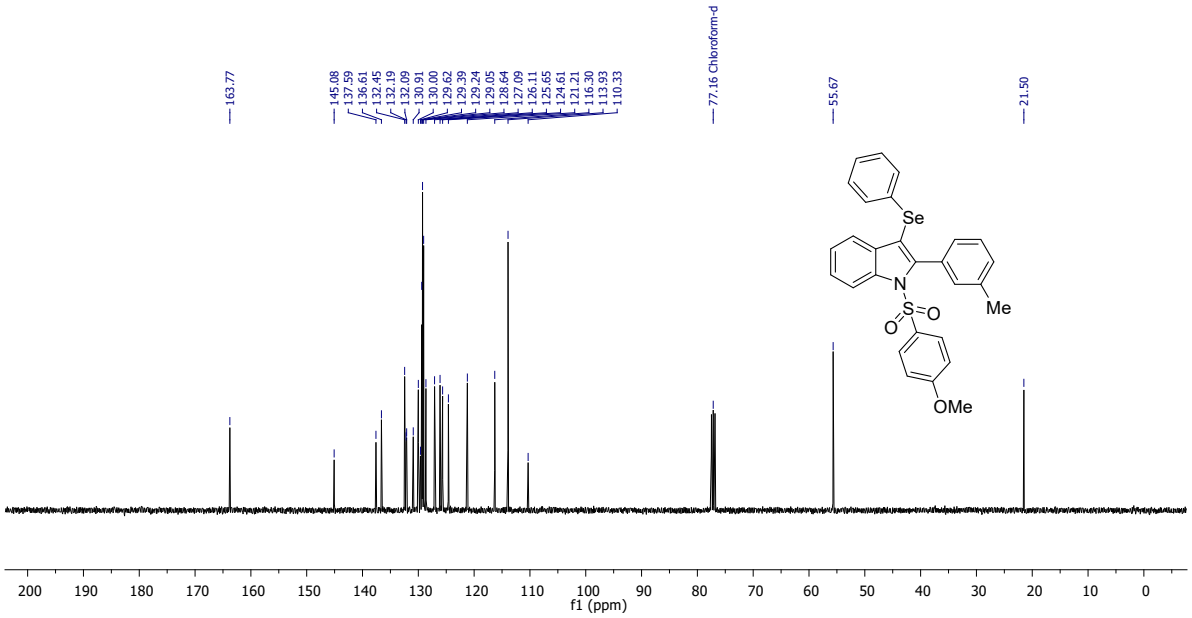
$^{13}C\{^{1}H\}$-NMR (101 MHz) spectrum of **3ra** in $CDCl_3$



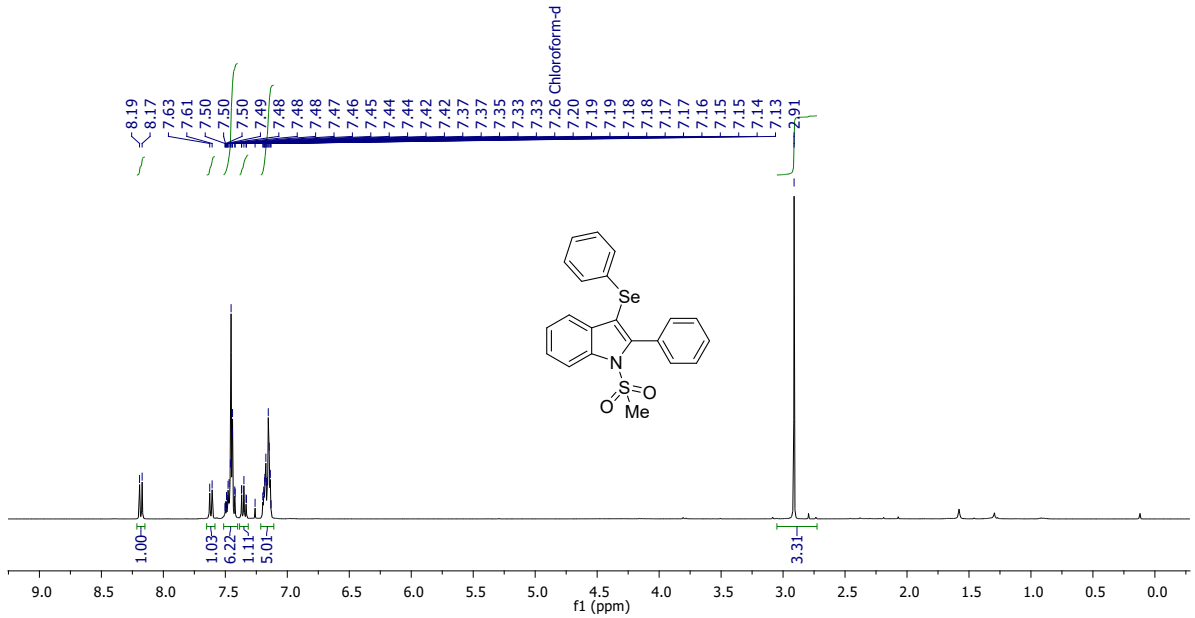
^{1}H-NMR (400 MHz) spectrum of **3sa** in $CDCl_3$



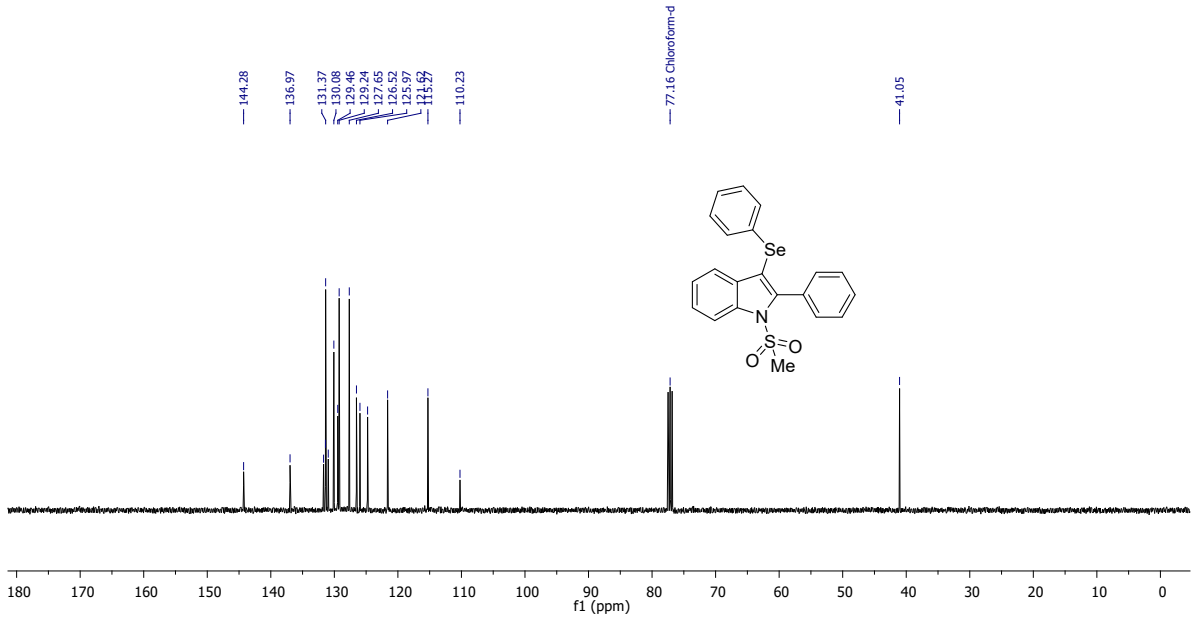
$^{13}C\{^{1}H\}$-NMR (101 MHz) spectrum of **3sa** in $CDCl_3$



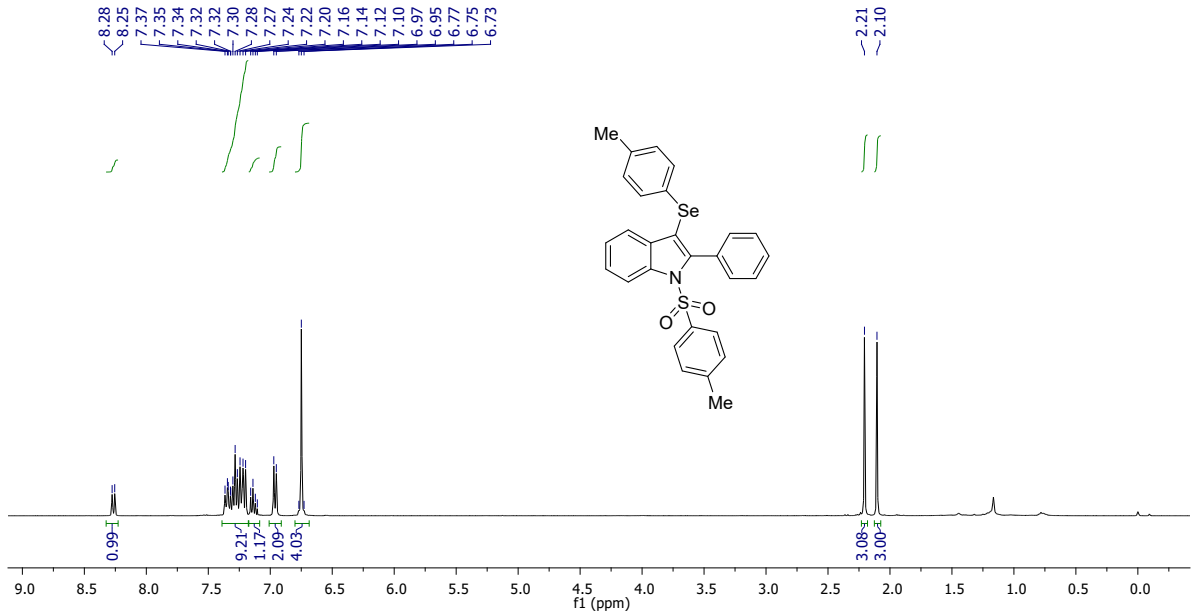
^{1}H-NMR (400 MHz) spectrum of **3ua** in $CDCl_3$



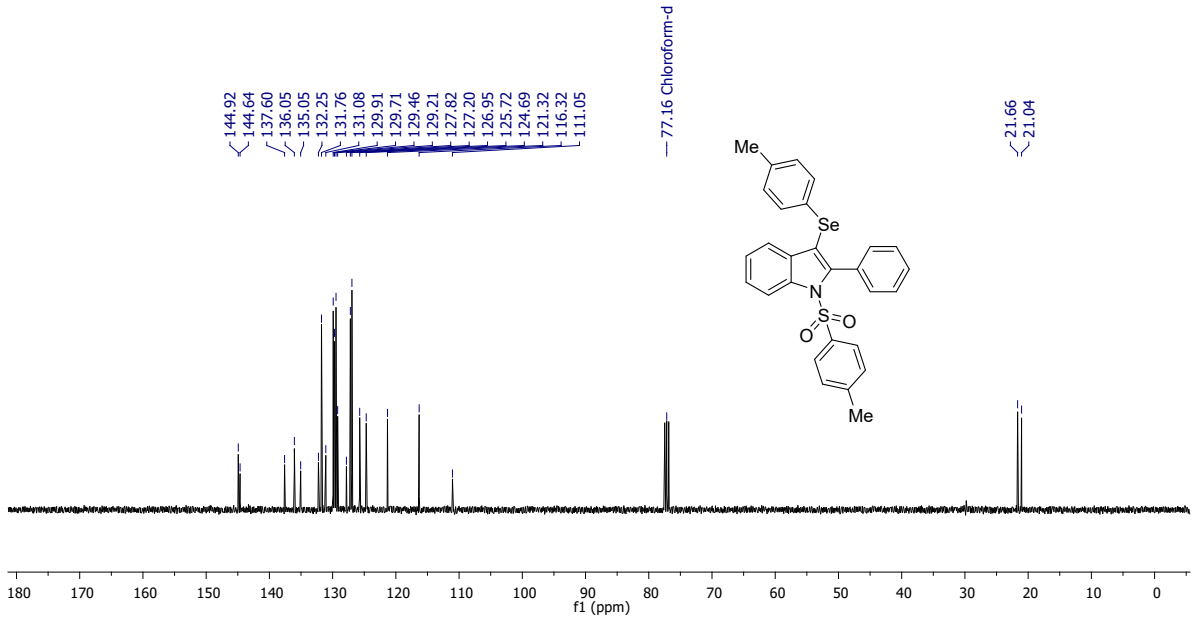
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ua** in $CDCl_3$



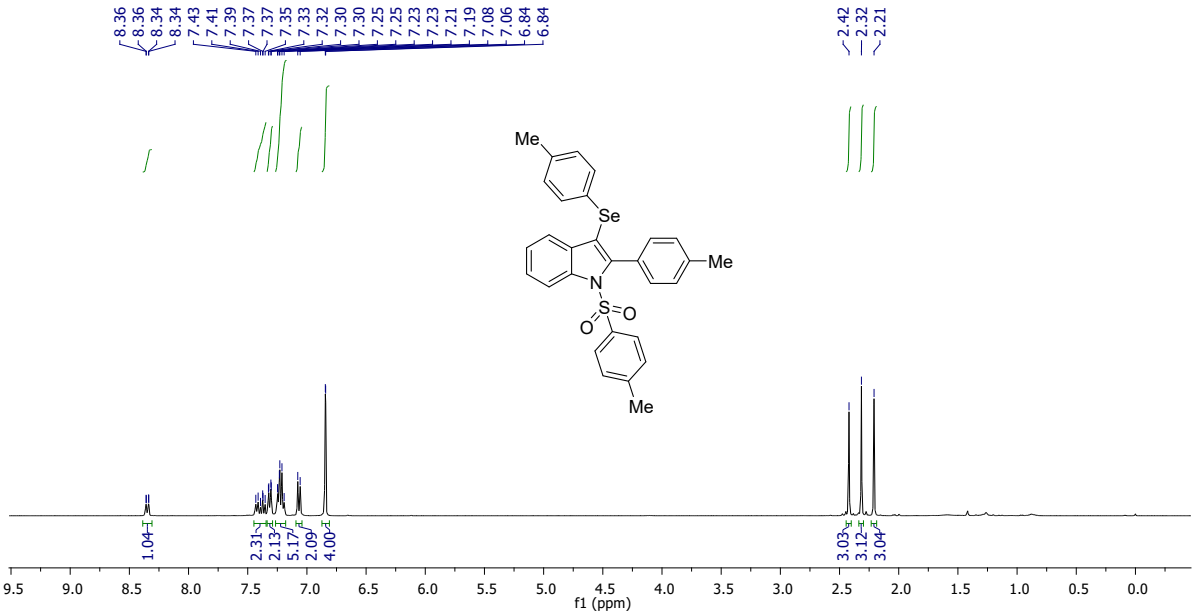
^{1}H-NMR (400 MHz) spectrum of **3ab** in $CDCl_3$



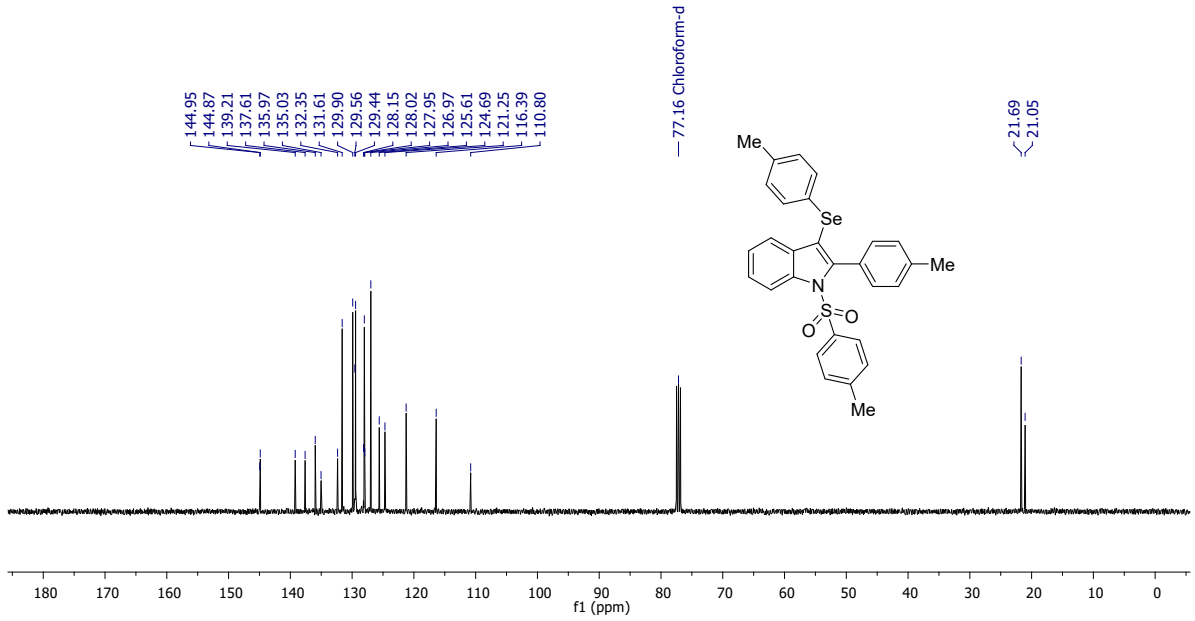
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ab** in $CDCl_3$



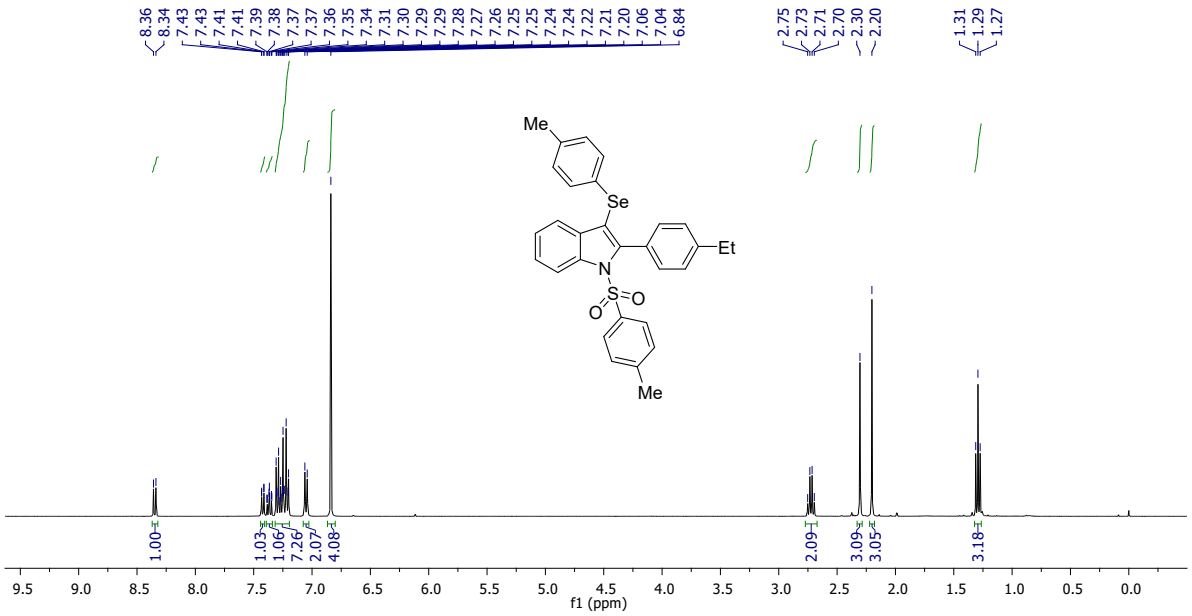
^{1}H-NMR (400 MHz) spectrum of **3bb** in $CDCl_3$



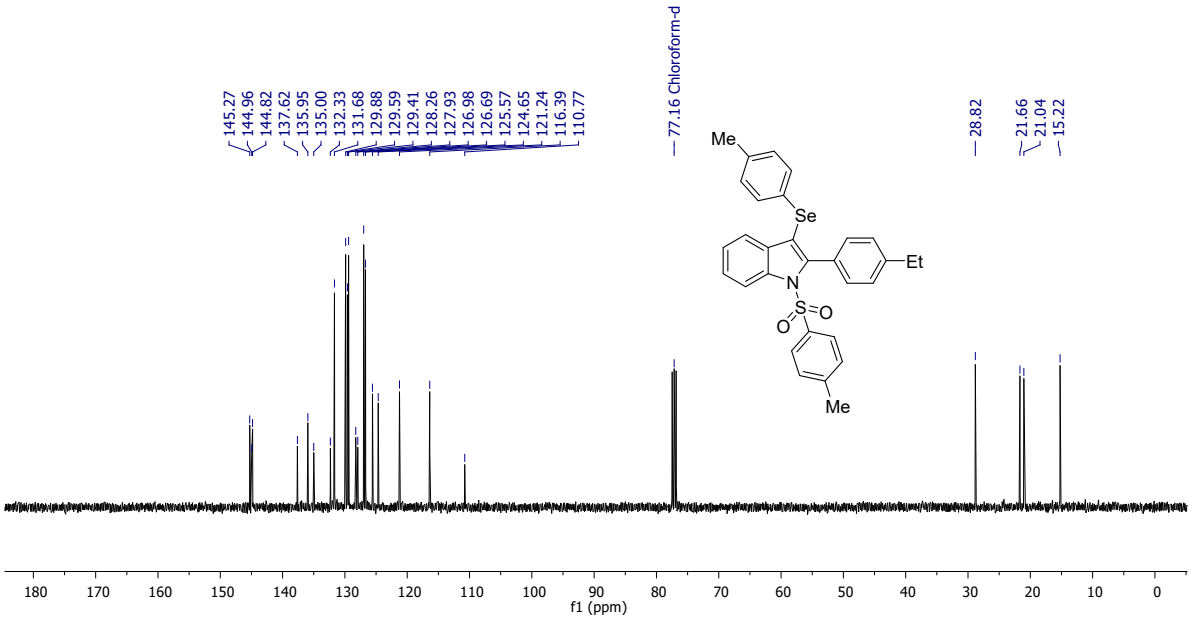
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3bb** in $CDCl_3$



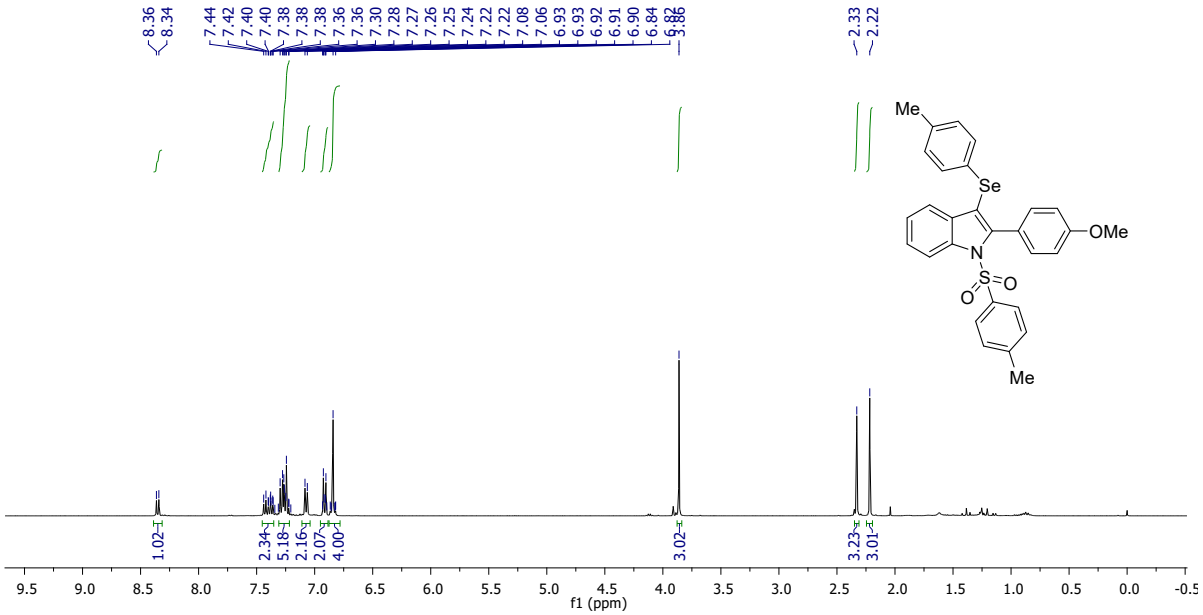
^{1}H-NMR (400 MHz) spectrum of **3cb** in $CDCl_3$



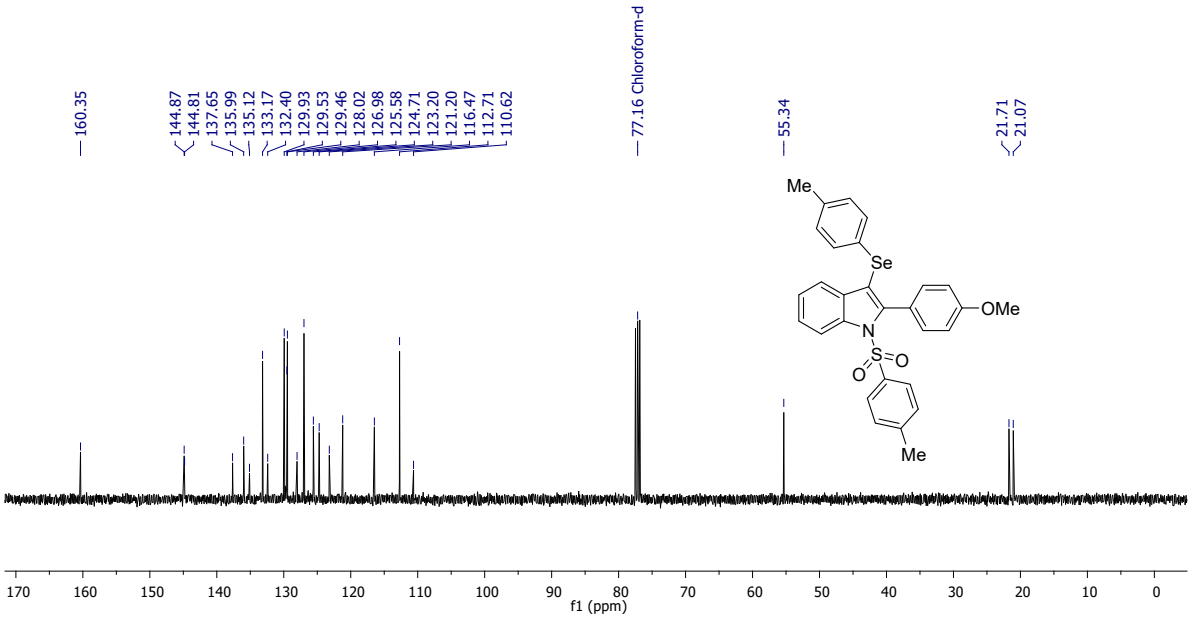
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3cb** in $CDCl_3$



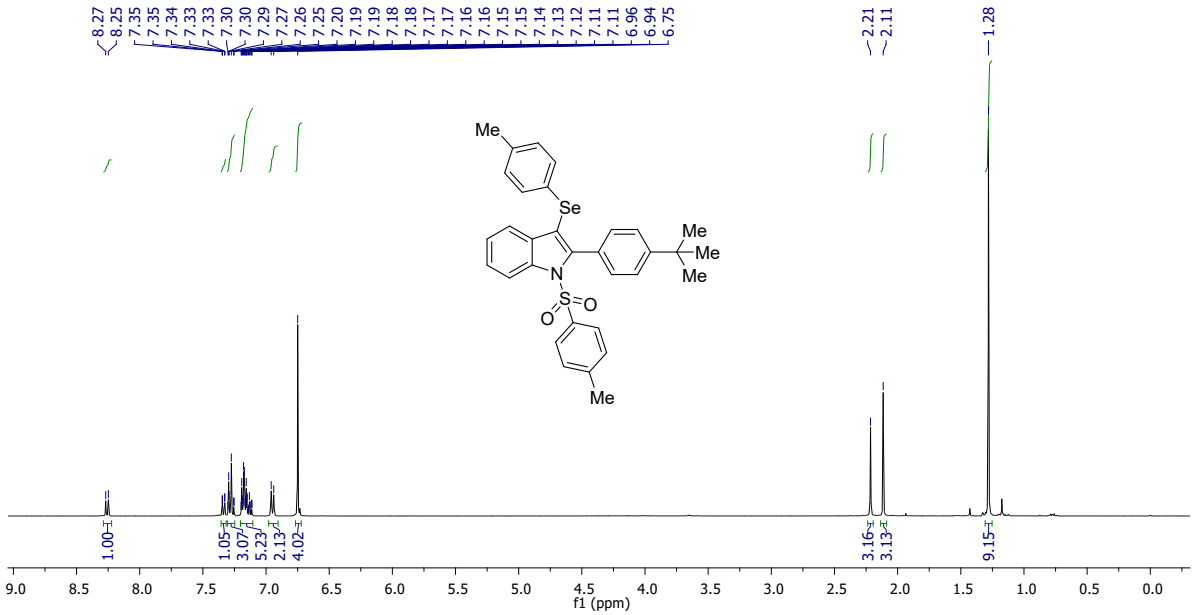
^{1}H-NMR (400 MHz) spectrum of **3db** in $CDCl_3$



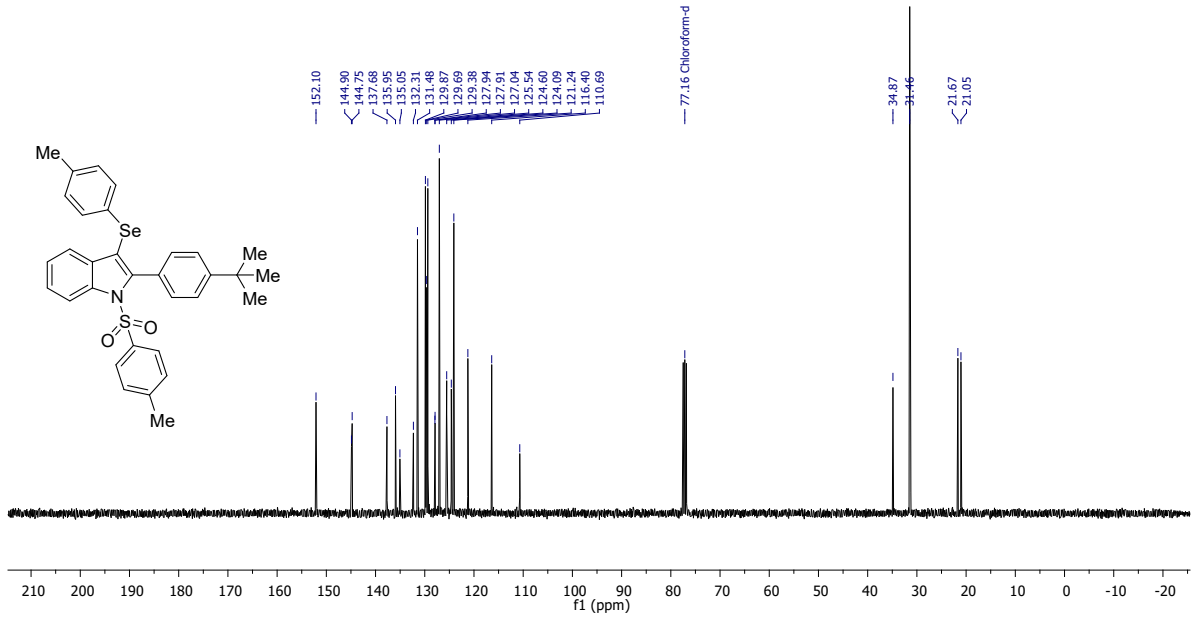
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3db** in $CDCl_3$



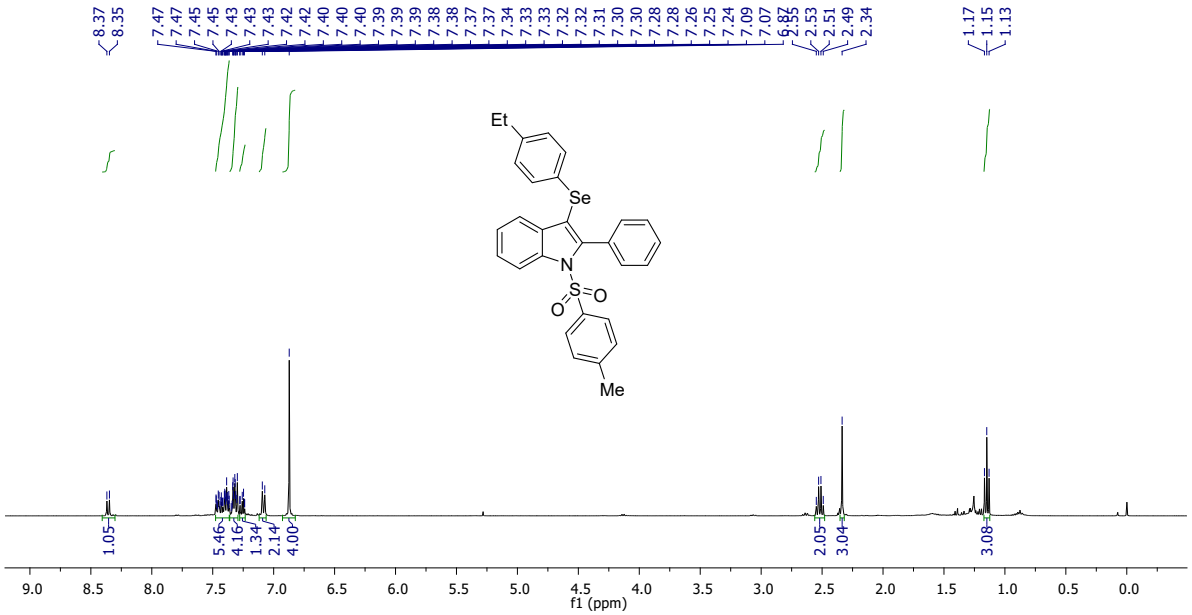
^{1}H-NMR (400 MHz) spectrum of **3eb** in $CDCl_3$



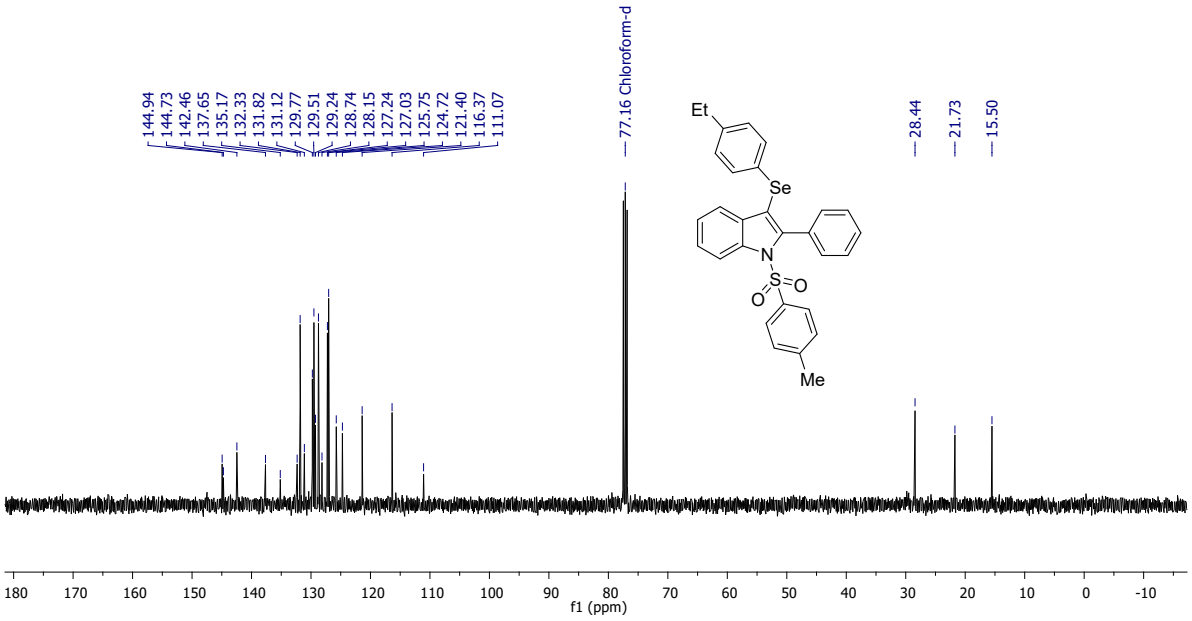
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3eb** in $CDCl_3$



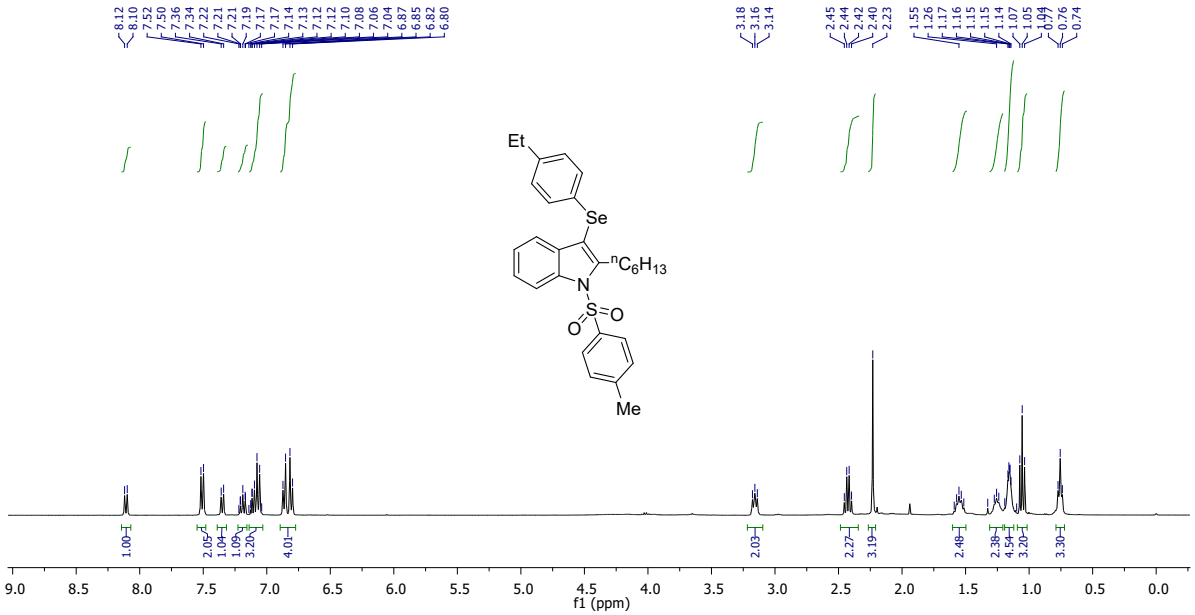
^{1}H-NMR (400 MHz) spectrum of **3ac** in $CDCl_3$



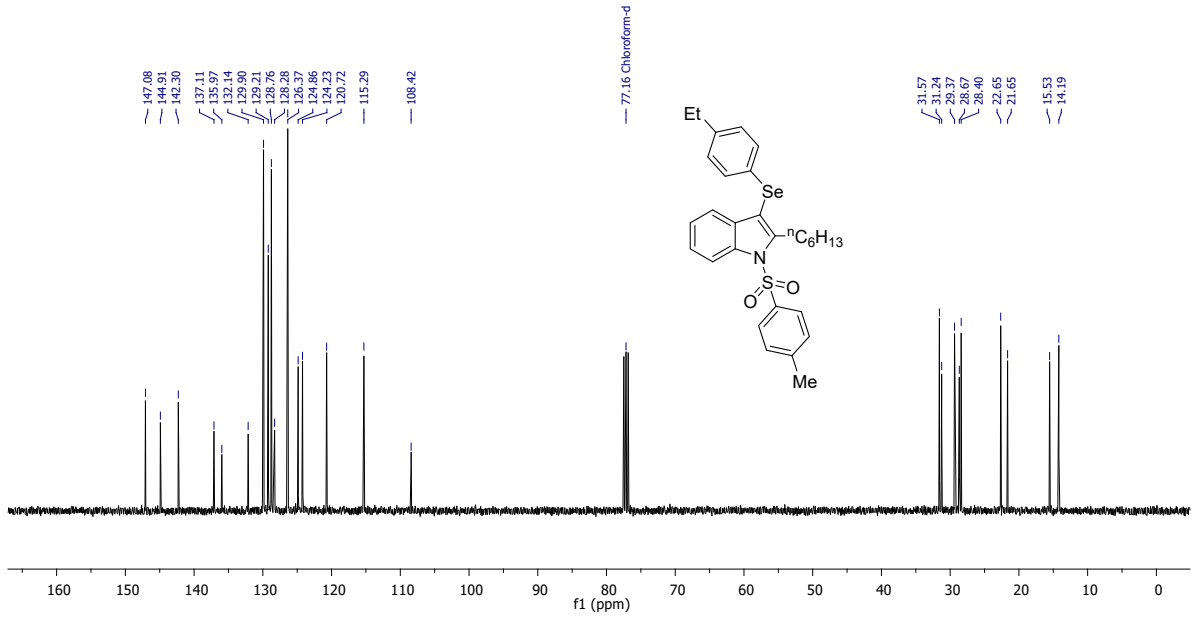
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ac** in $CDCl_3$



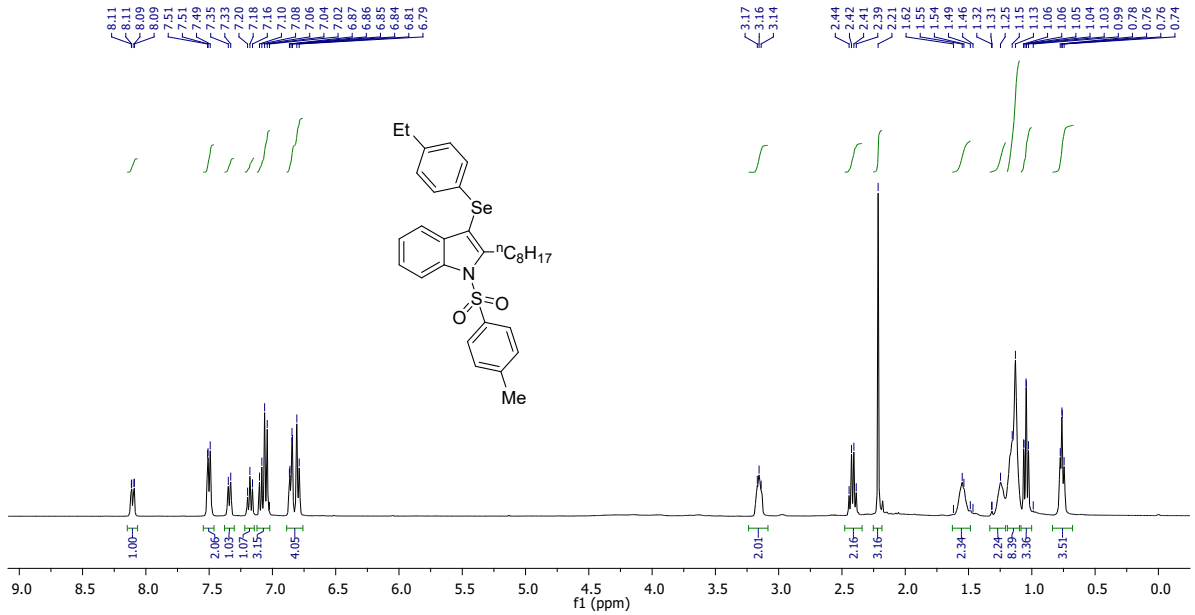
^{1}H-NMR (400 MHz) spectrum of **3mc** in $CDCl_3$



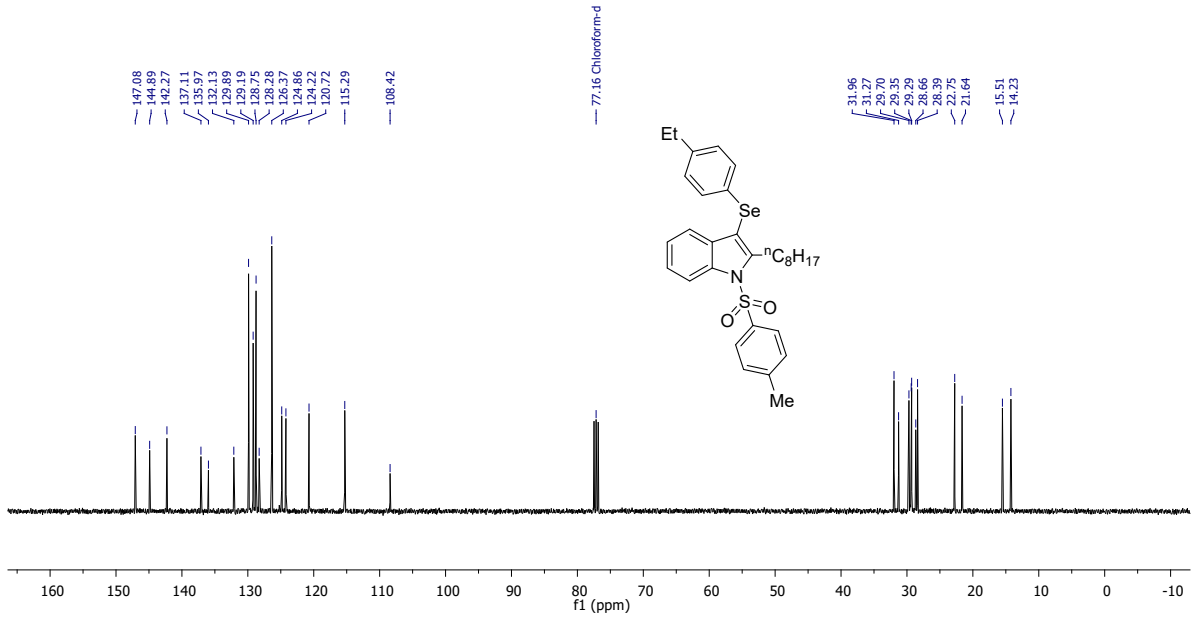
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3mc** in $CDCl_3$



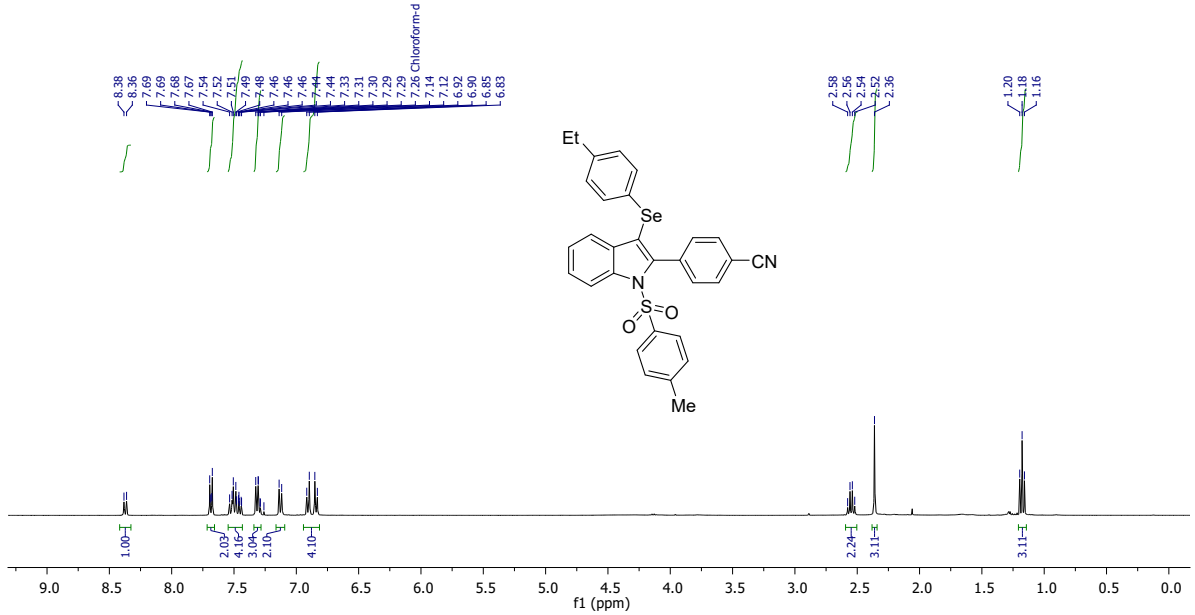
^{1}H-NMR (400 MHz) spectrum of **3nc** in $CDCl_3$



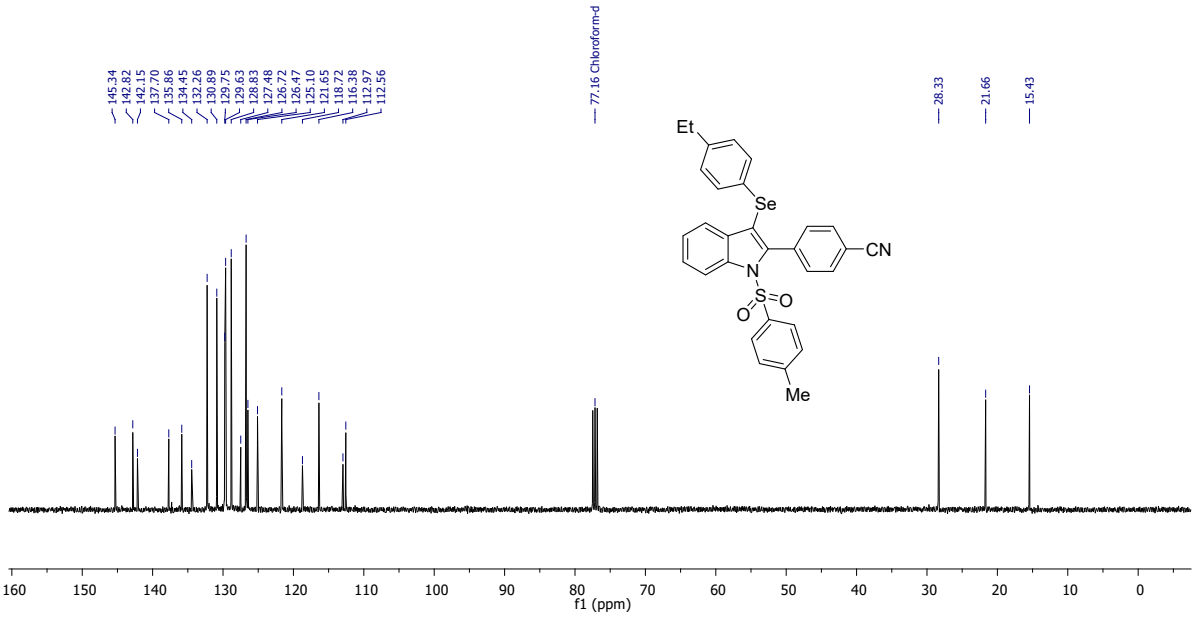
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3nc** in $CDCl_3$



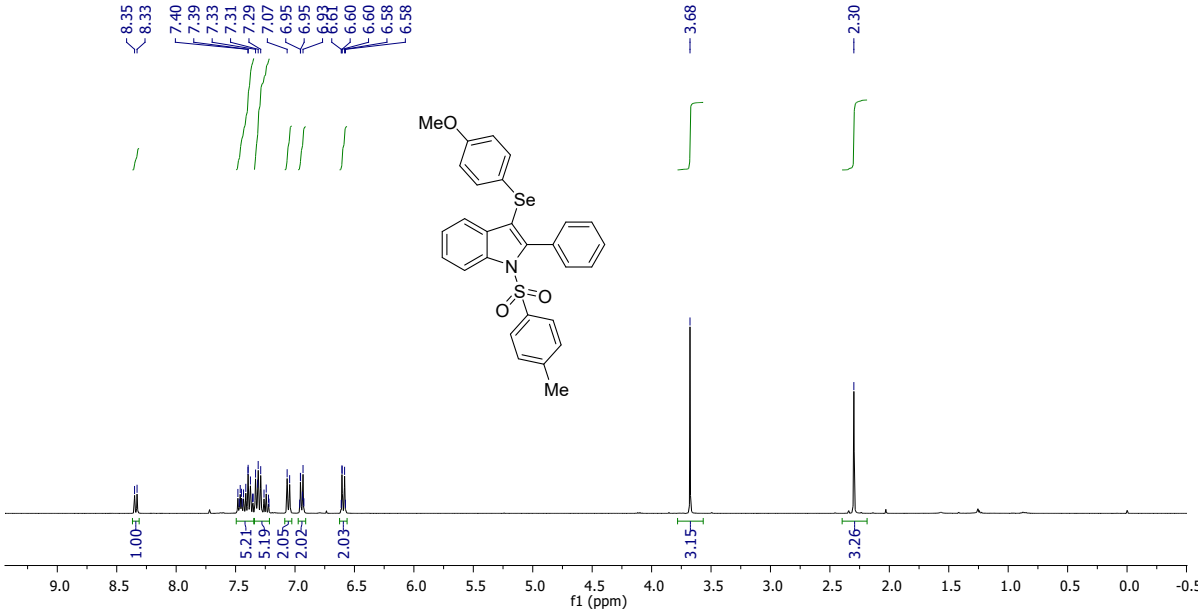
^{1}H-NMR (400 MHz) spectrum of **3oc** in $CDCl_3$



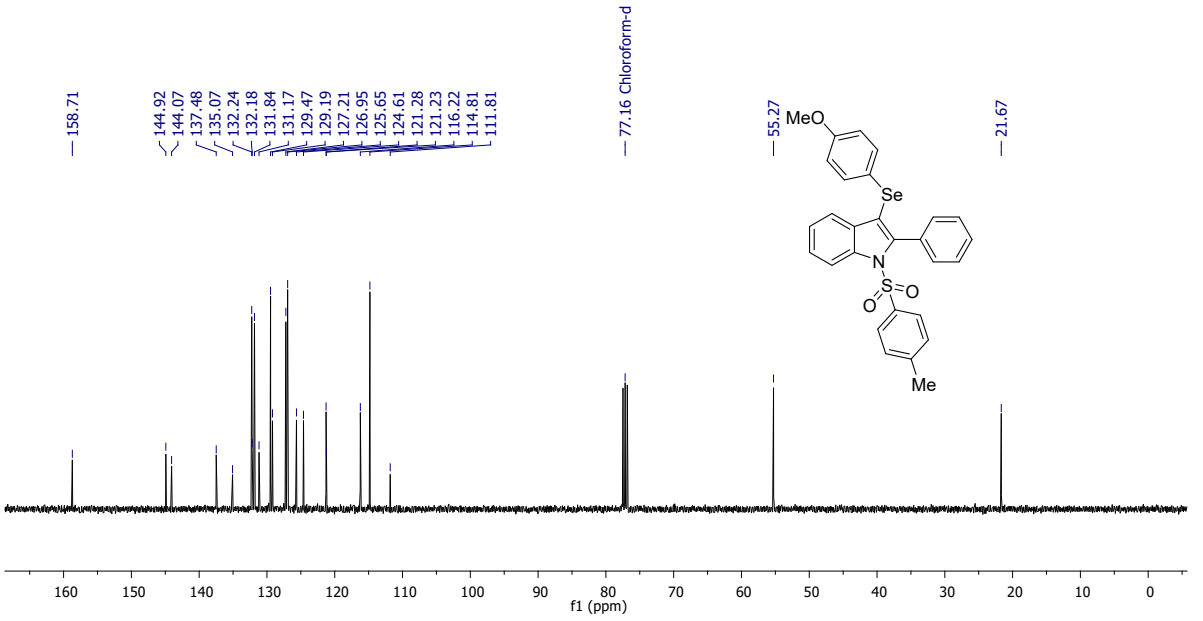
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3oc** in $CDCl_3$



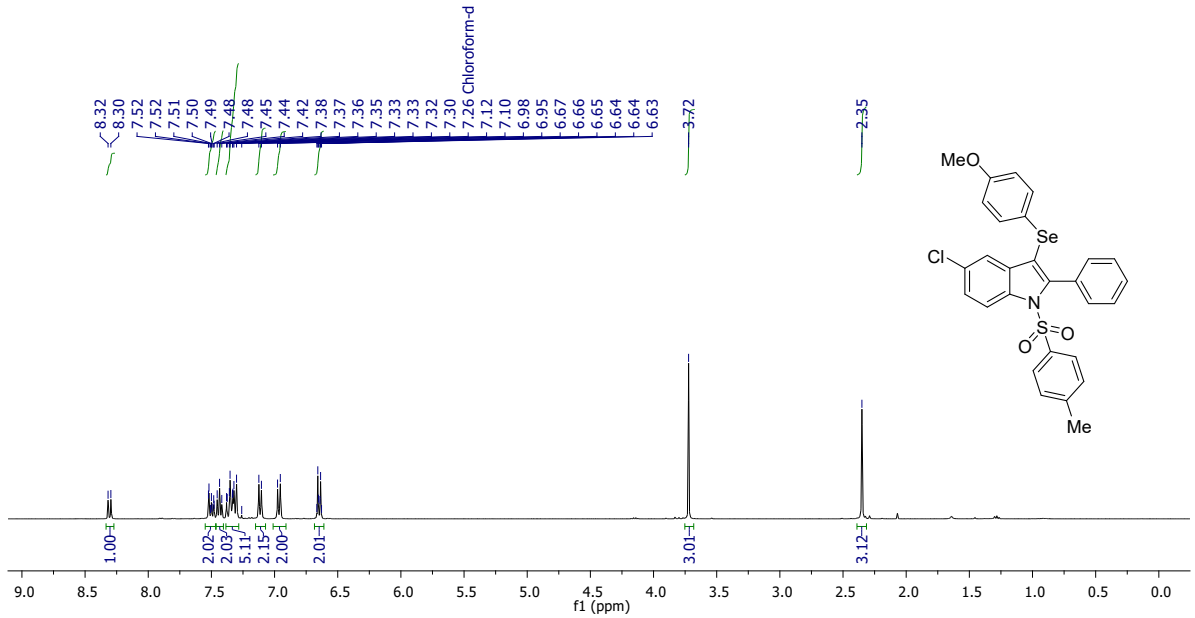
^{1}H-NMR (400 MHz) spectrum of **3ad** in $CDCl_3$



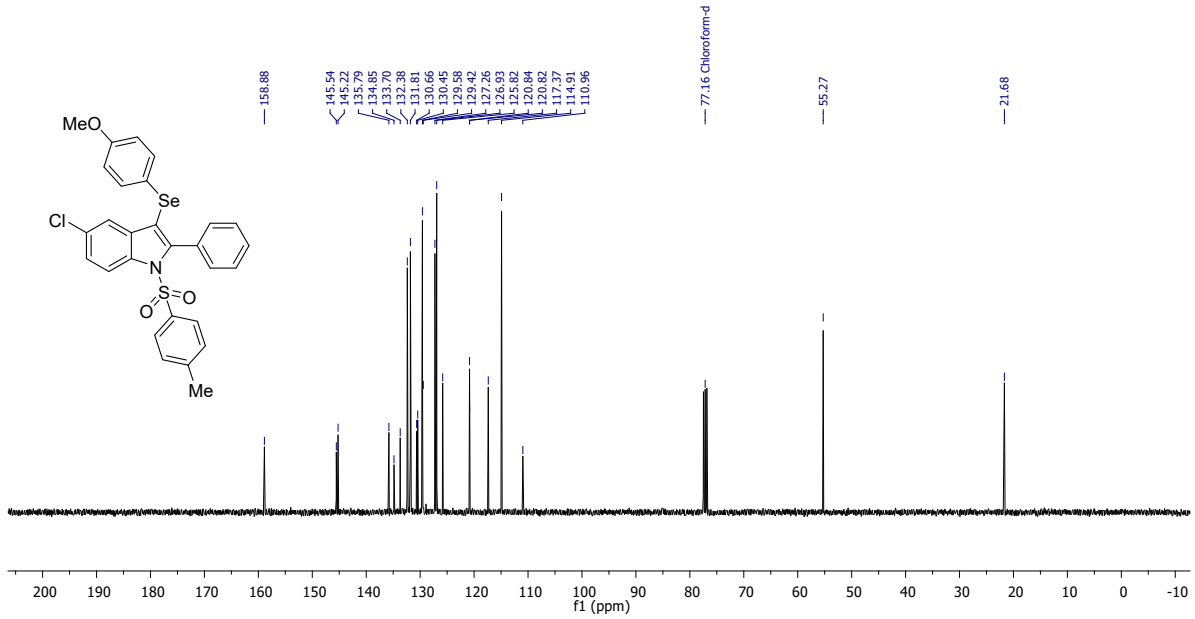
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ad** in $CDCl_3$



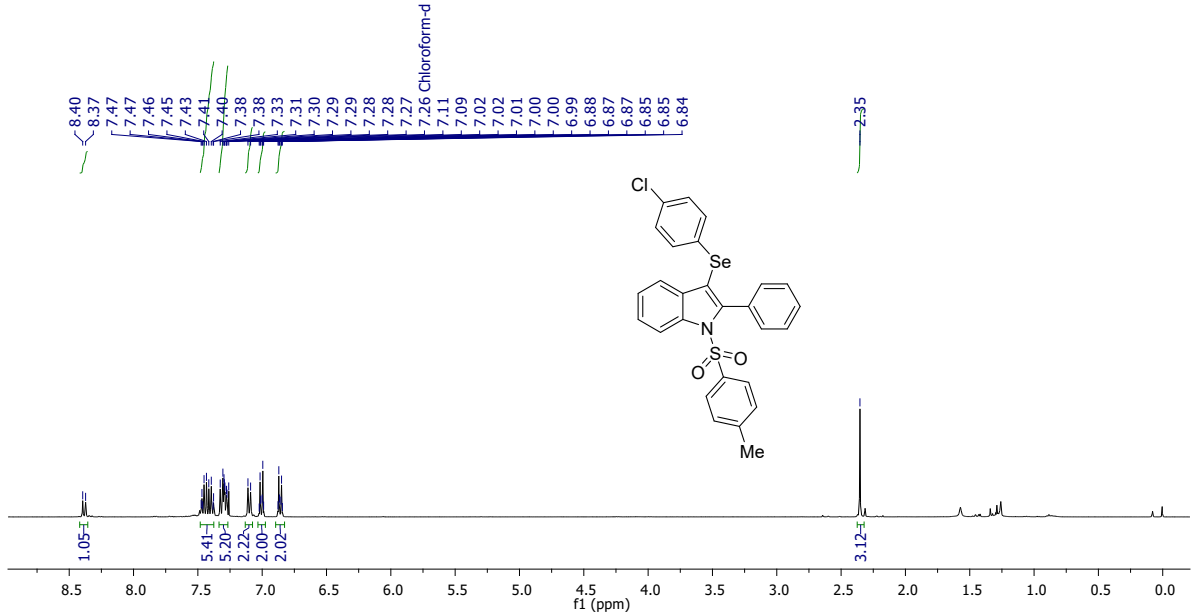
^{1}H-NMR (400 MHz) spectrum of **3id** in $CDCl_3$



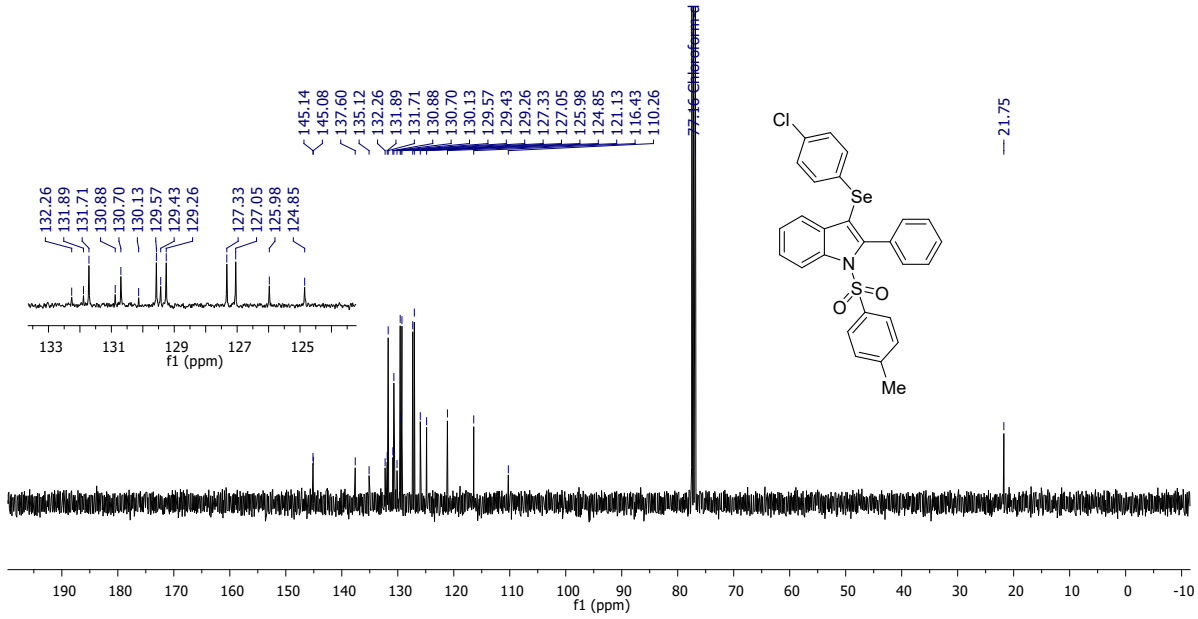
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3id** in $CDCl_3$



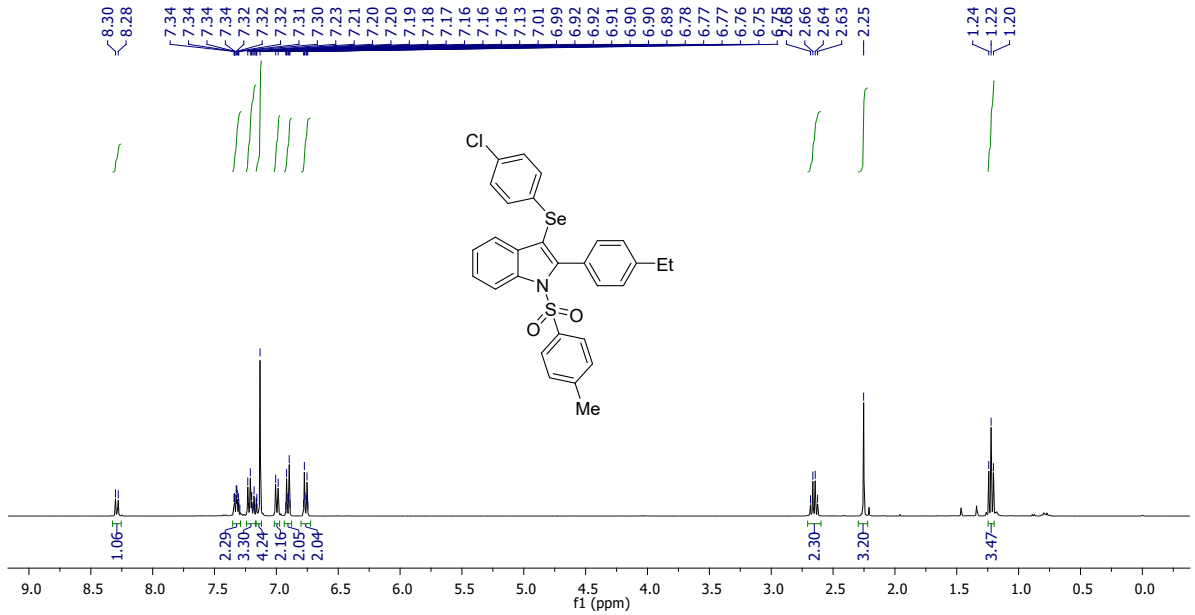
^{1}H-NMR (400 MHz) spectrum of **3ae** in $CDCl_3$



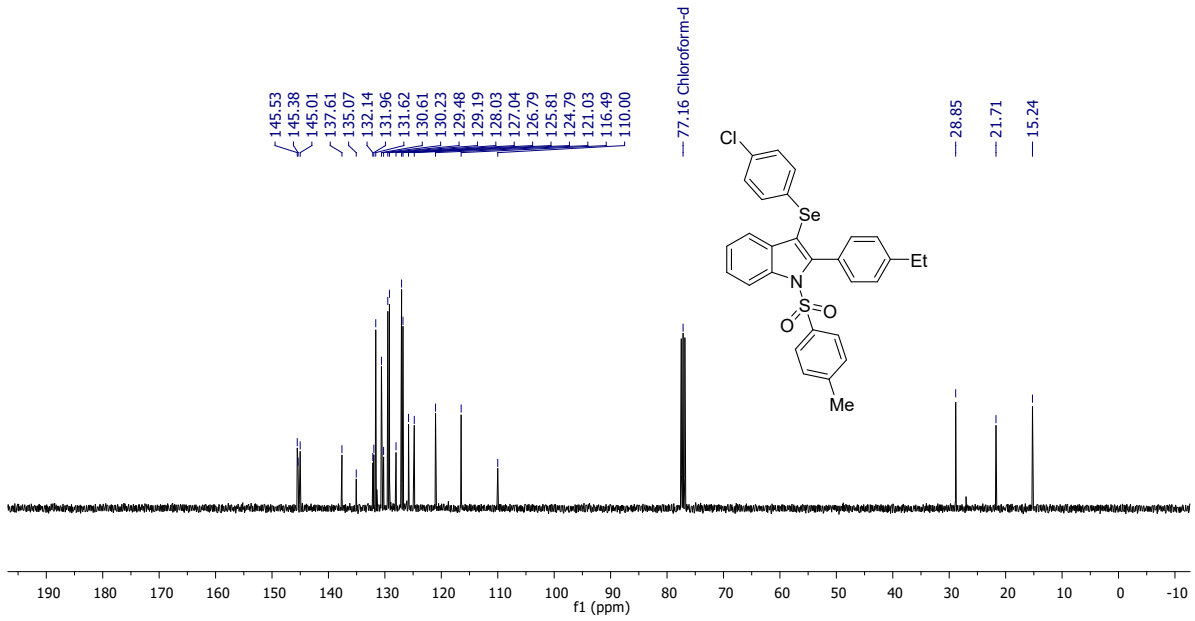
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ae** in $CDCl_3$



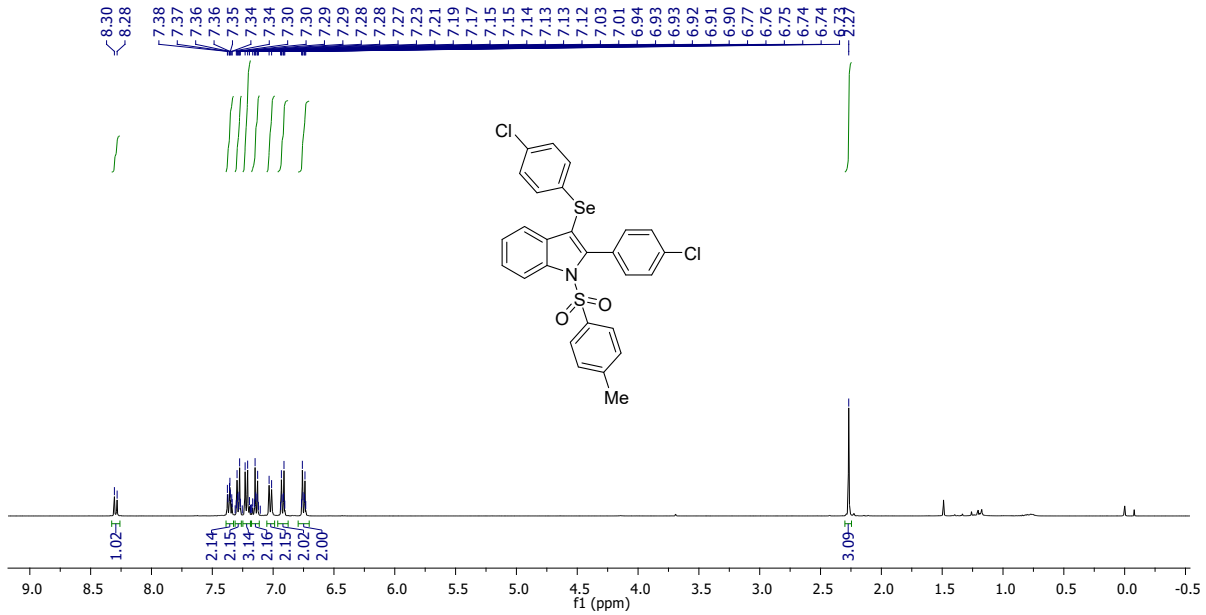
^{1}H-NMR (400 MHz) spectrum of **3ce** in $CDCl_3$



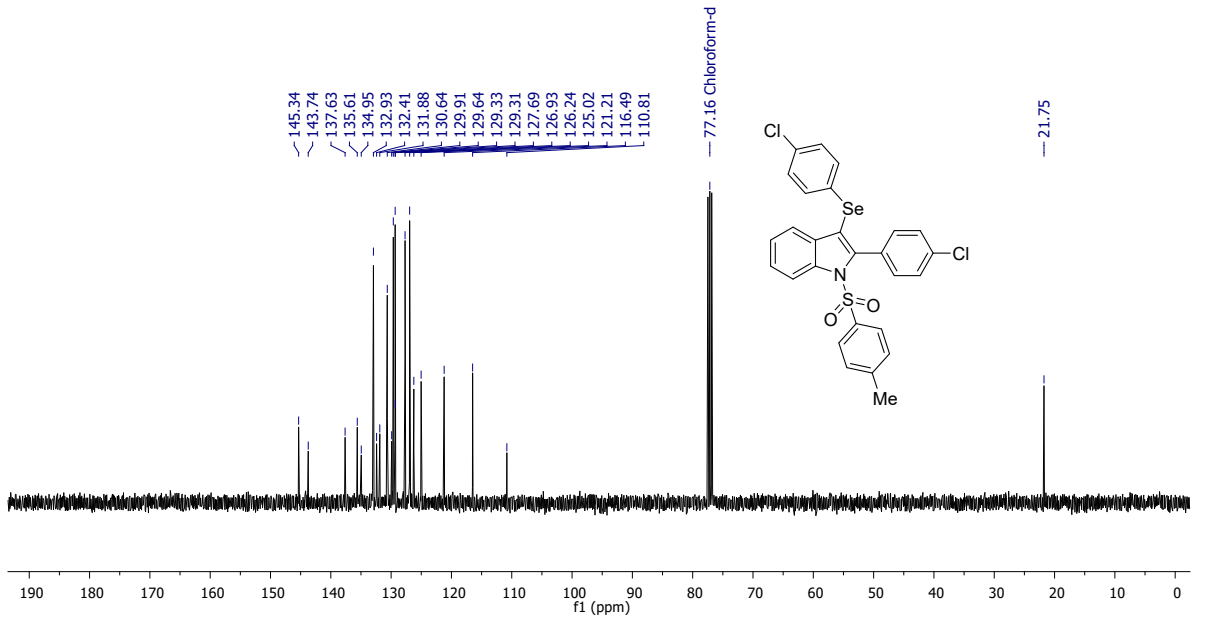
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ce** in $CDCl_3$



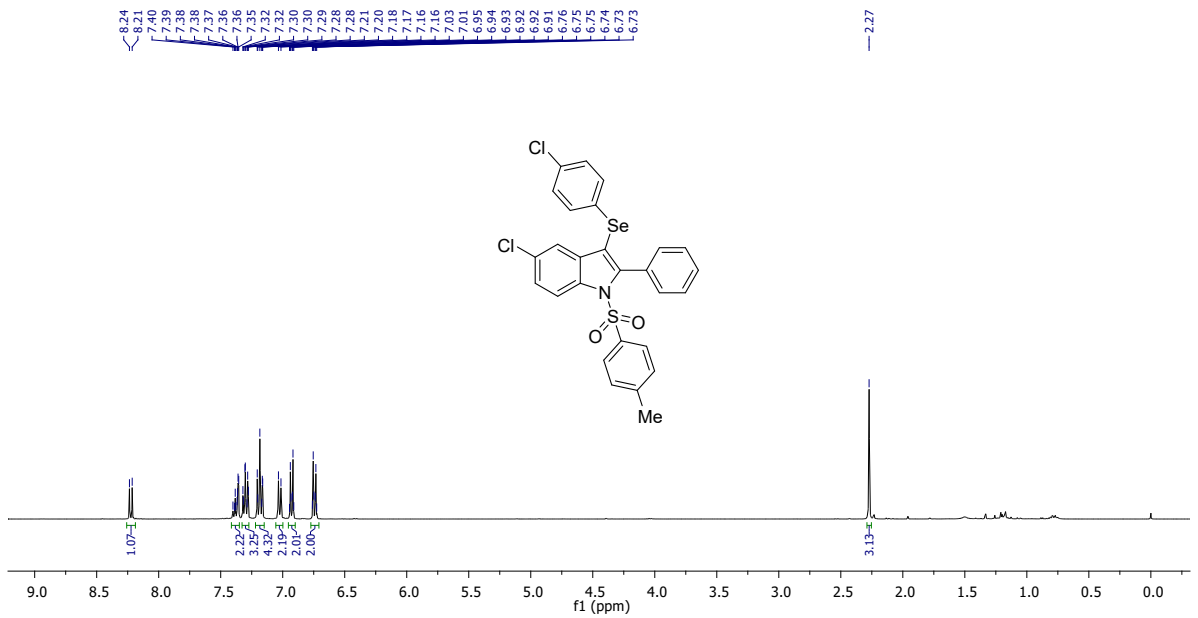
^{1}H-NMR (400 MHz) spectrum of **3fe** in $CDCl_3$



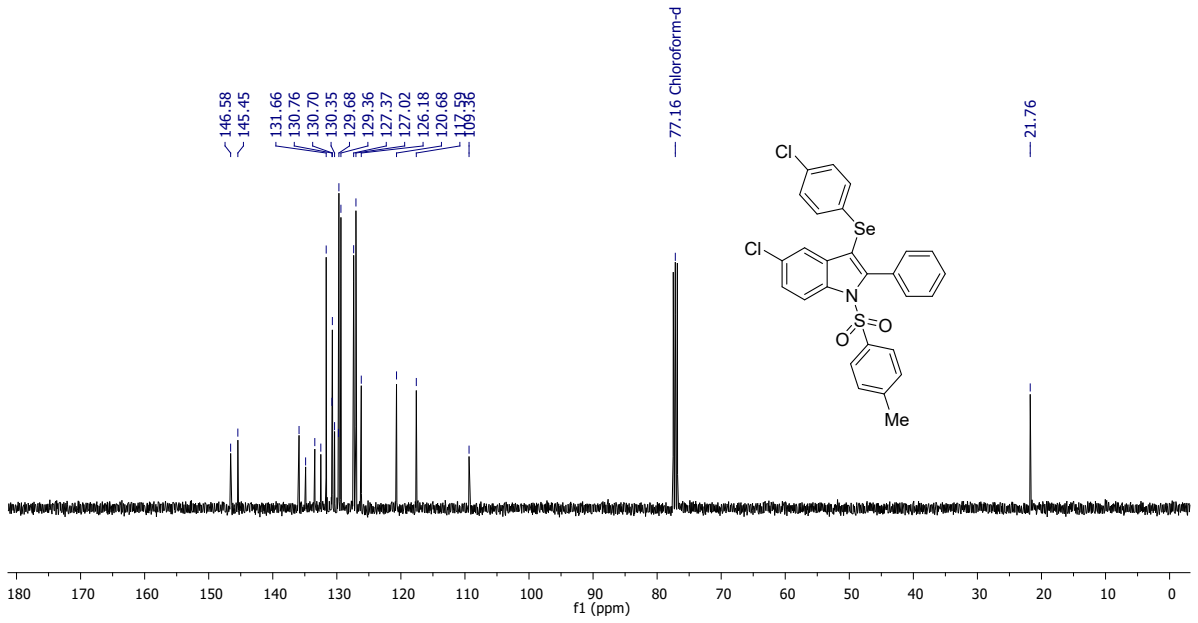
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3fe** in $CDCl_3$



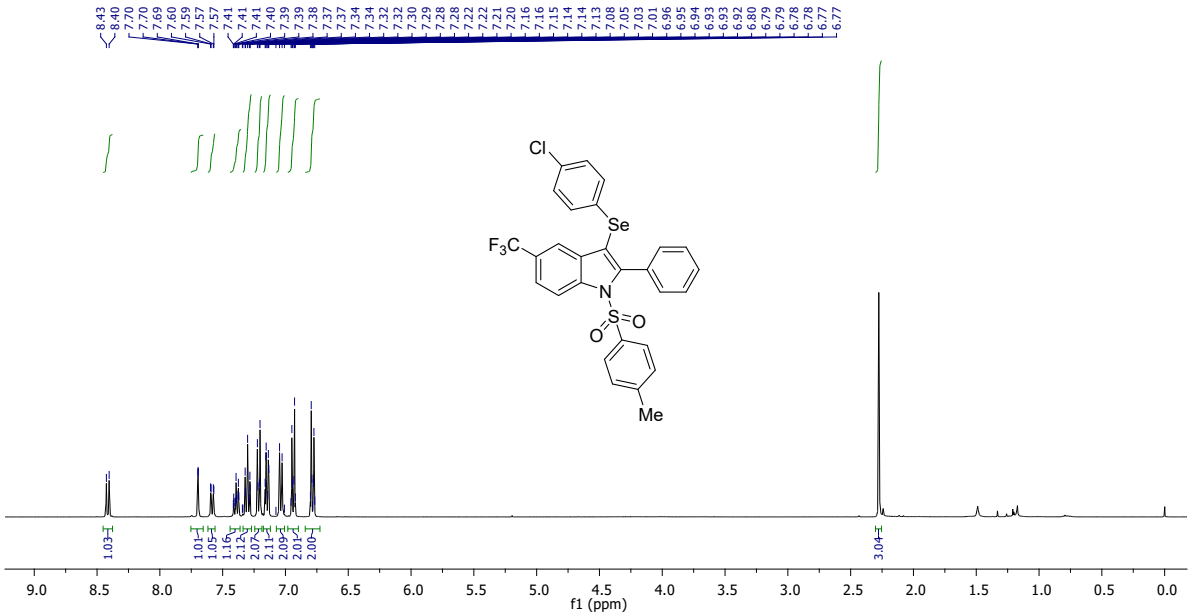
^{1}H-NMR (400 MHz) spectrum of **3ie** in $CDCl_3$



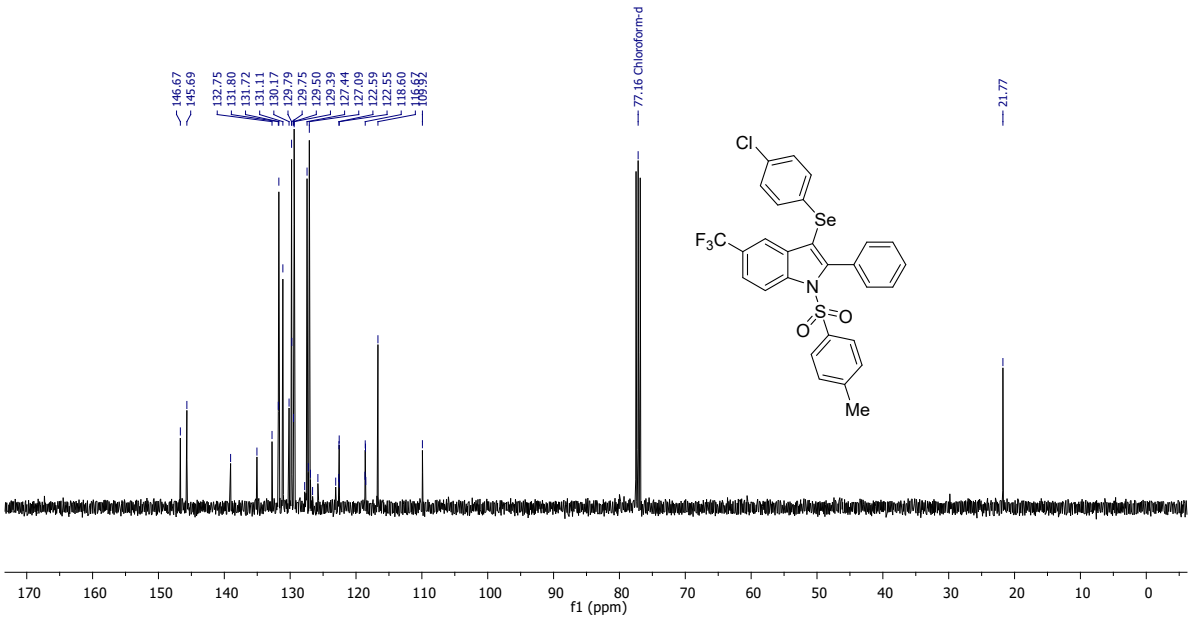
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ie** in $CDCl_3$



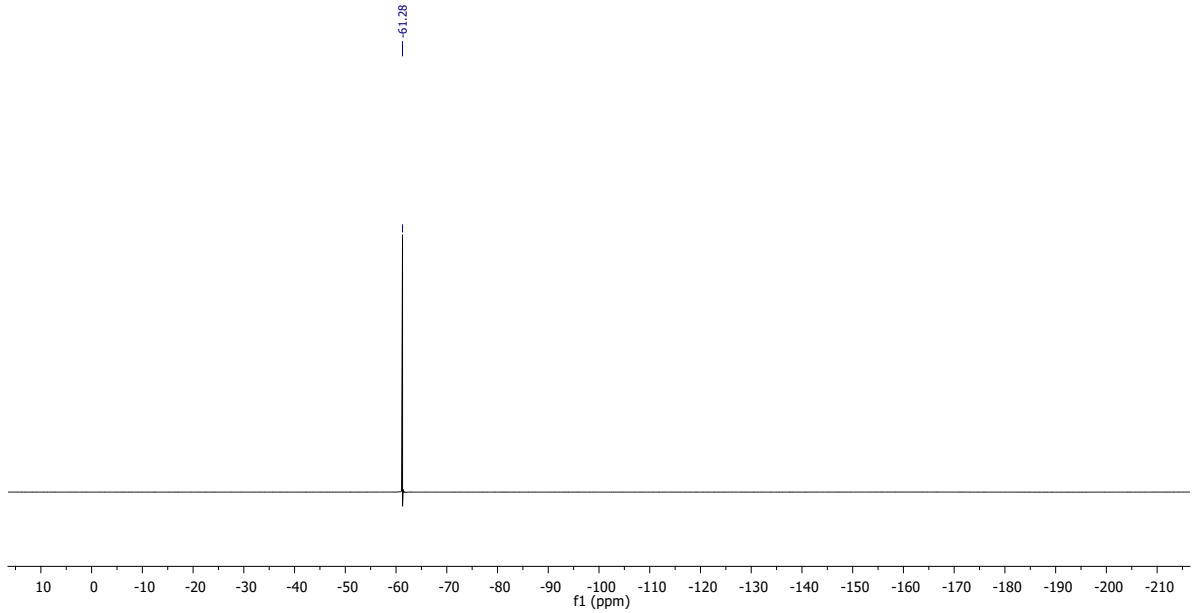
^{1}H-NMR (400 MHz) spectrum of **3je** in $CDCl_3$



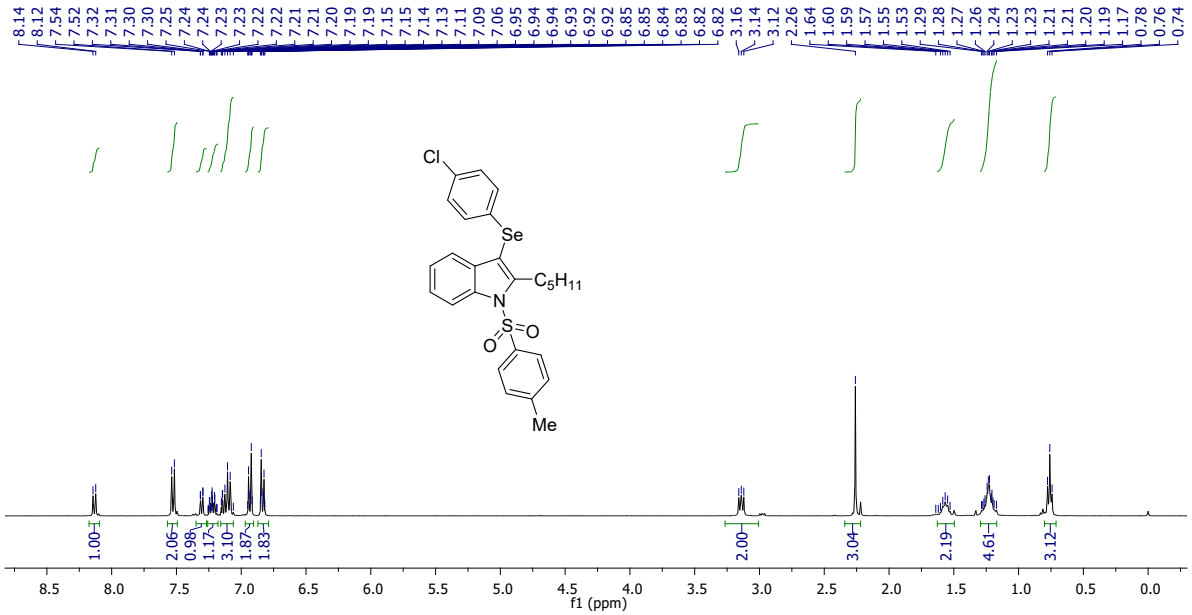
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3je** in $CDCl_3$



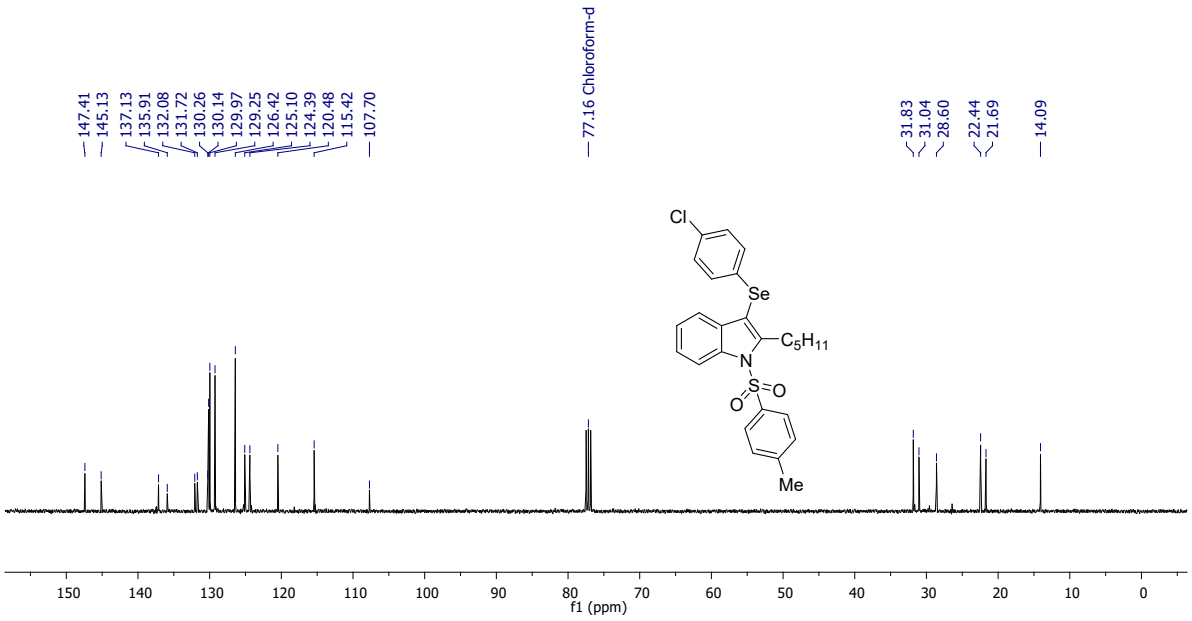
^{19}F NMR (565 MHz) spectrum of **3je** in $CDCl_3$



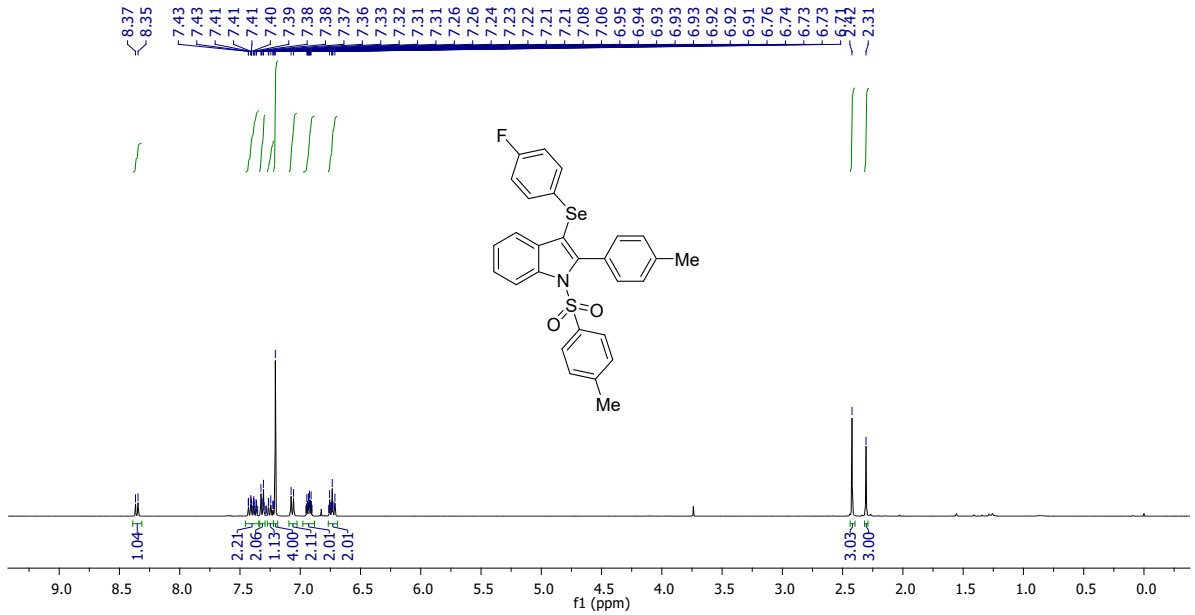
^{1}H-NMR (400 MHz) spectrum of **3le** in $CDCl_3$



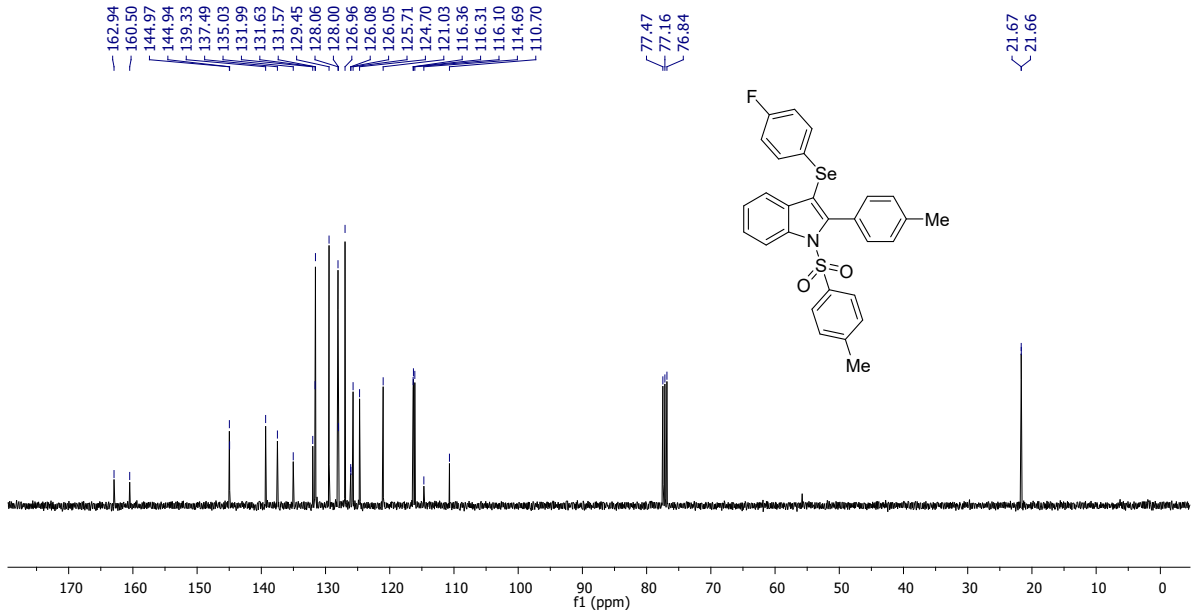
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3le** in $CDCl_3$



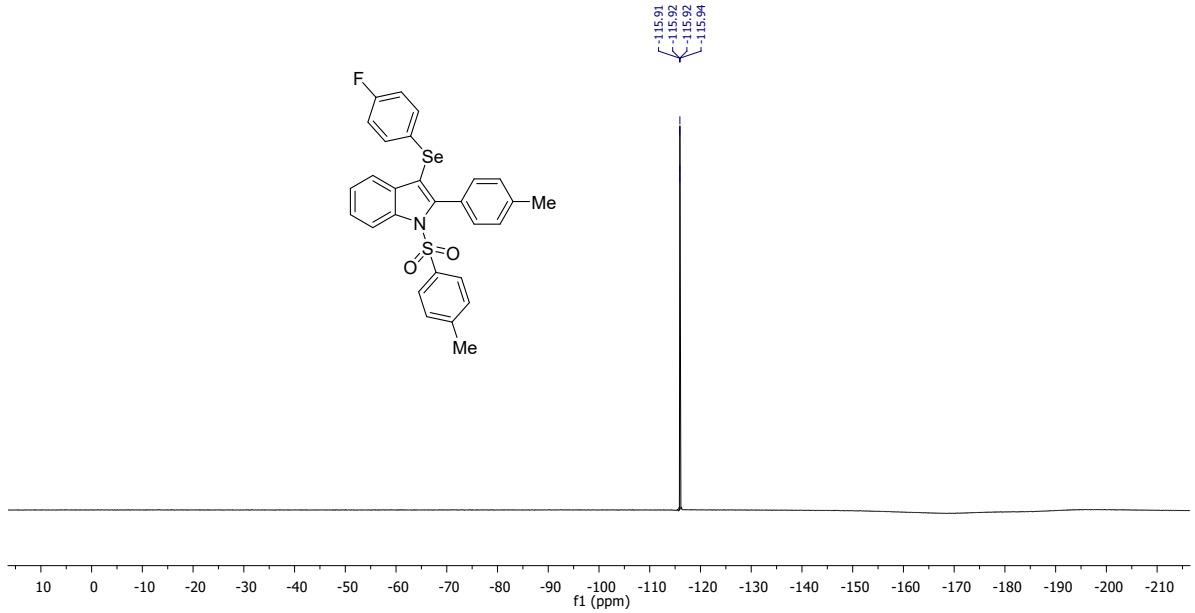
^{1}H-NMR (400 MHz) spectrum of **3bf** in $CDCl_3$



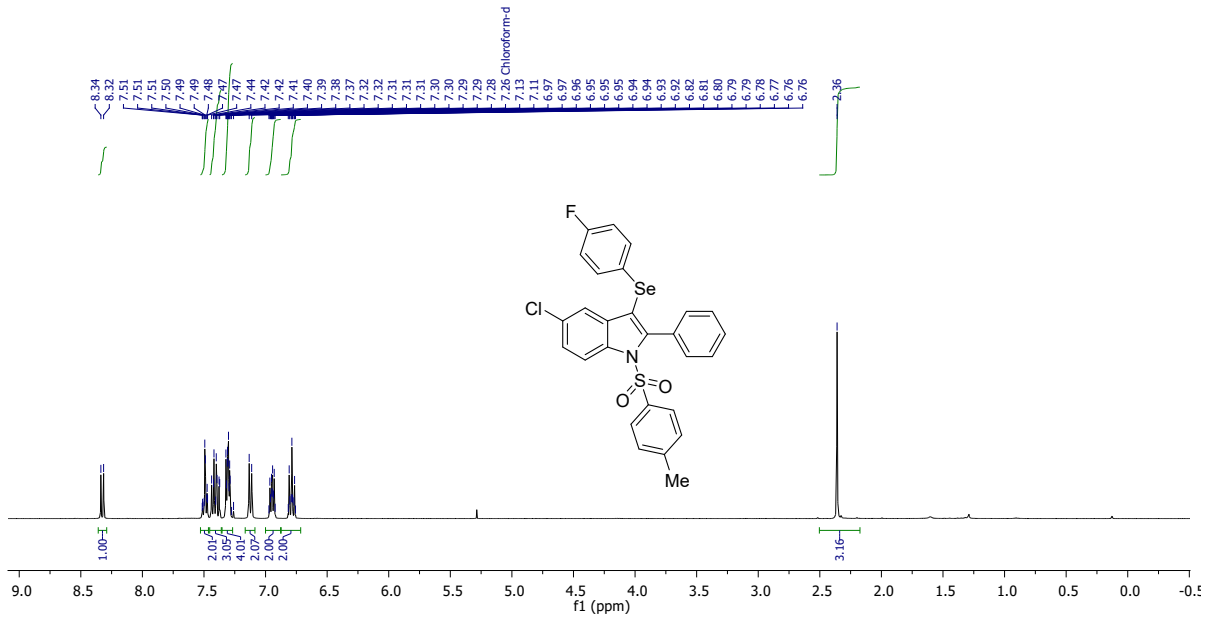
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3bf** in $CDCl_3$



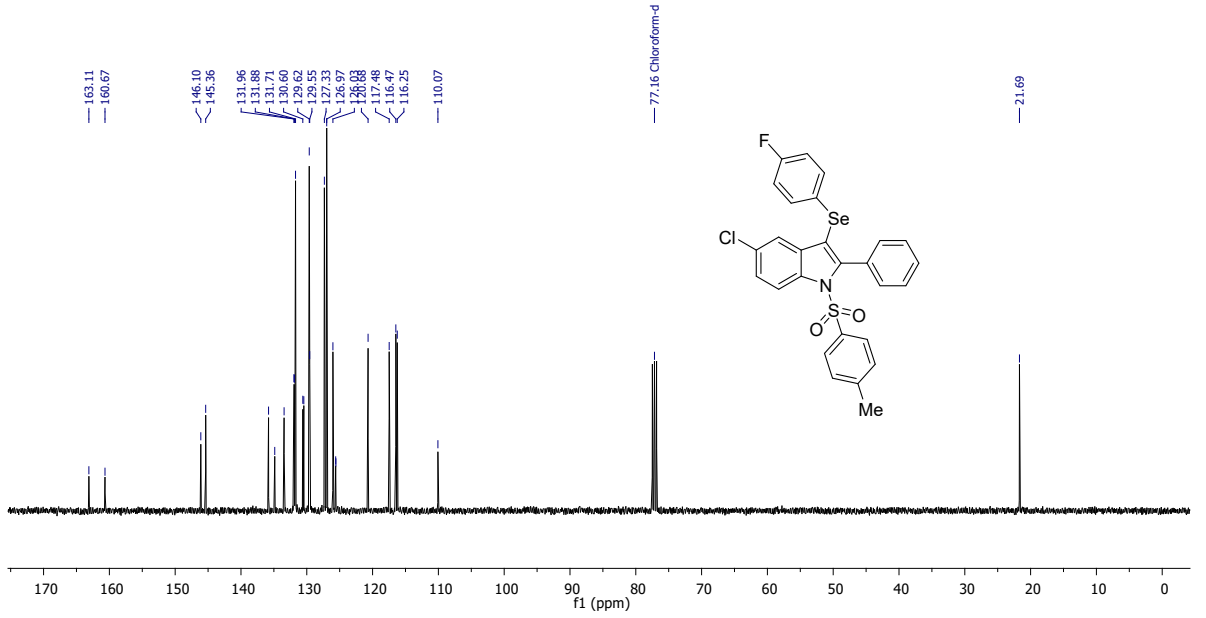
^{19}F NMR (565 MHz) spectrum of **3bf** in $CDCl_3$



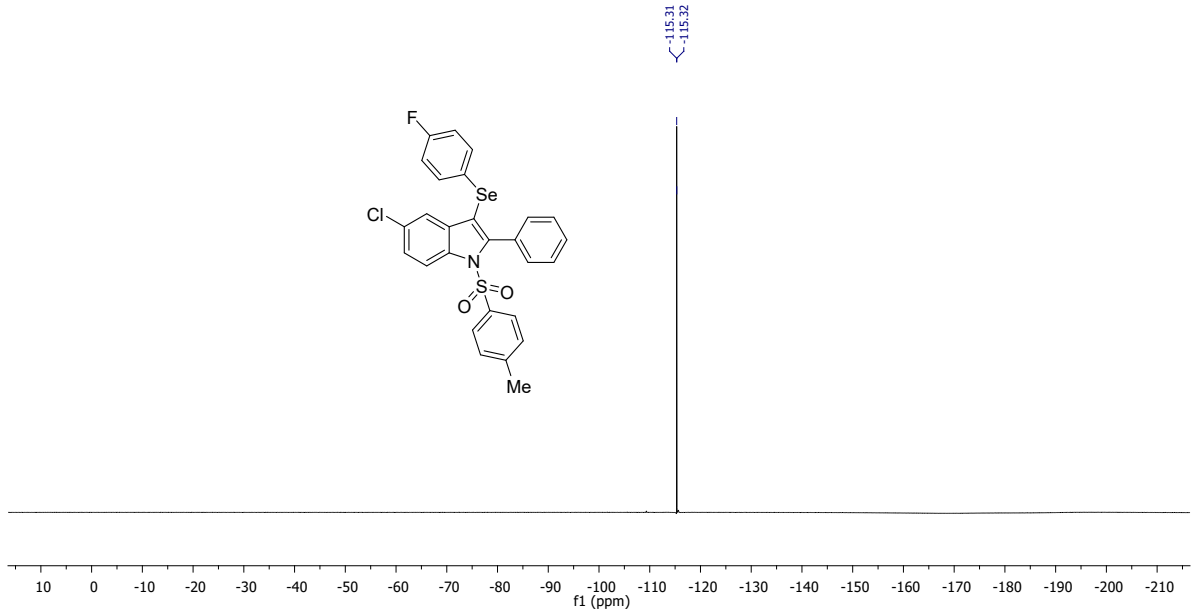
^{1}H-NMR (400 MHz) spectrum of **3if** in $CDCl_3$



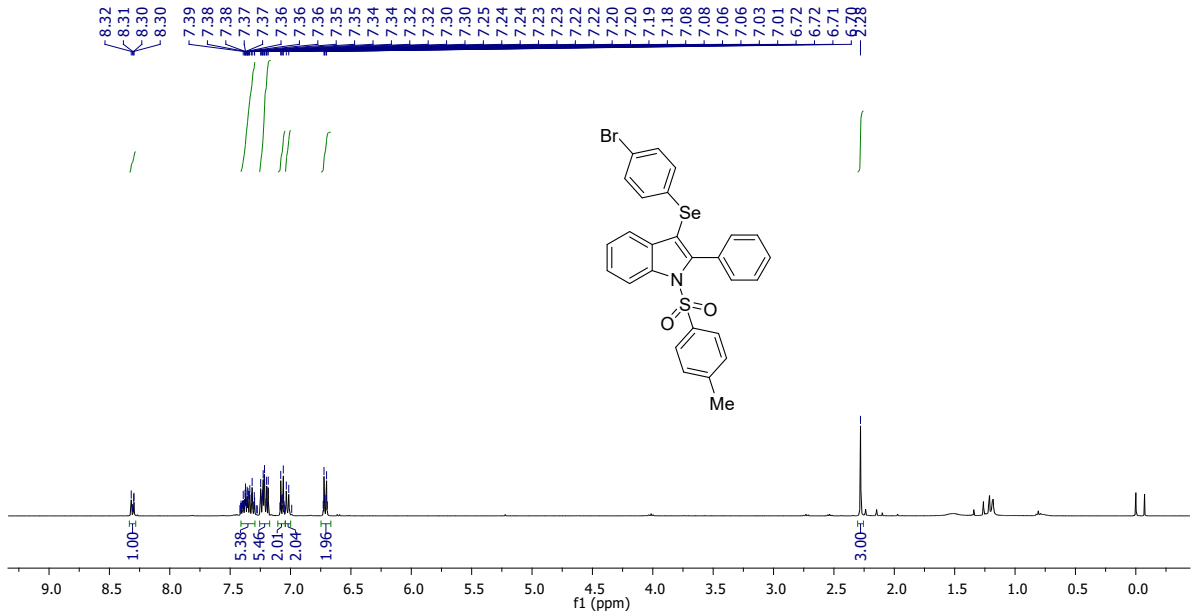
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3if** in $CDCl_3$



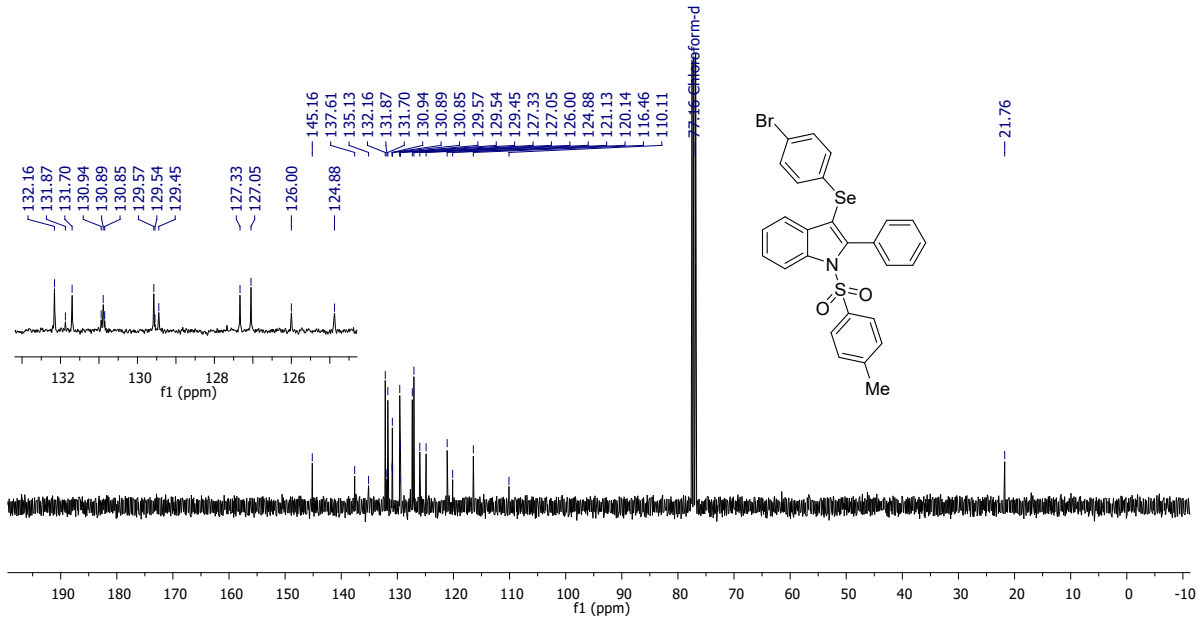
^{19}F NMR (565 MHz) spectrum of **3if** in $CDCl_3$



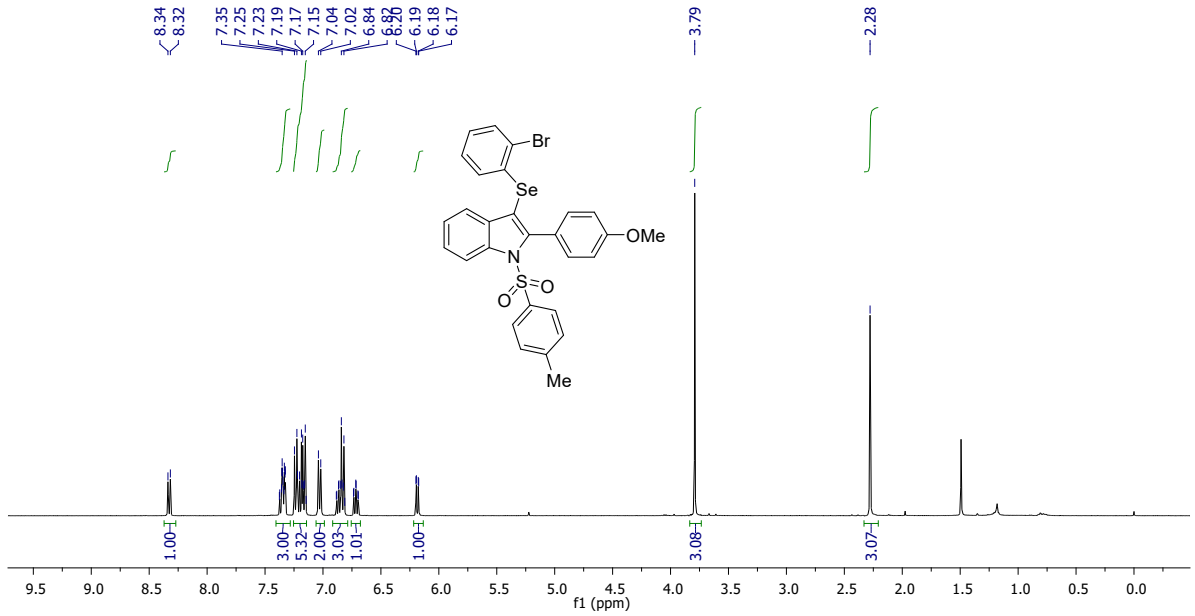
^{1}H-NMR (400 MHz) spectrum of **3ag** in $CDCl_3$



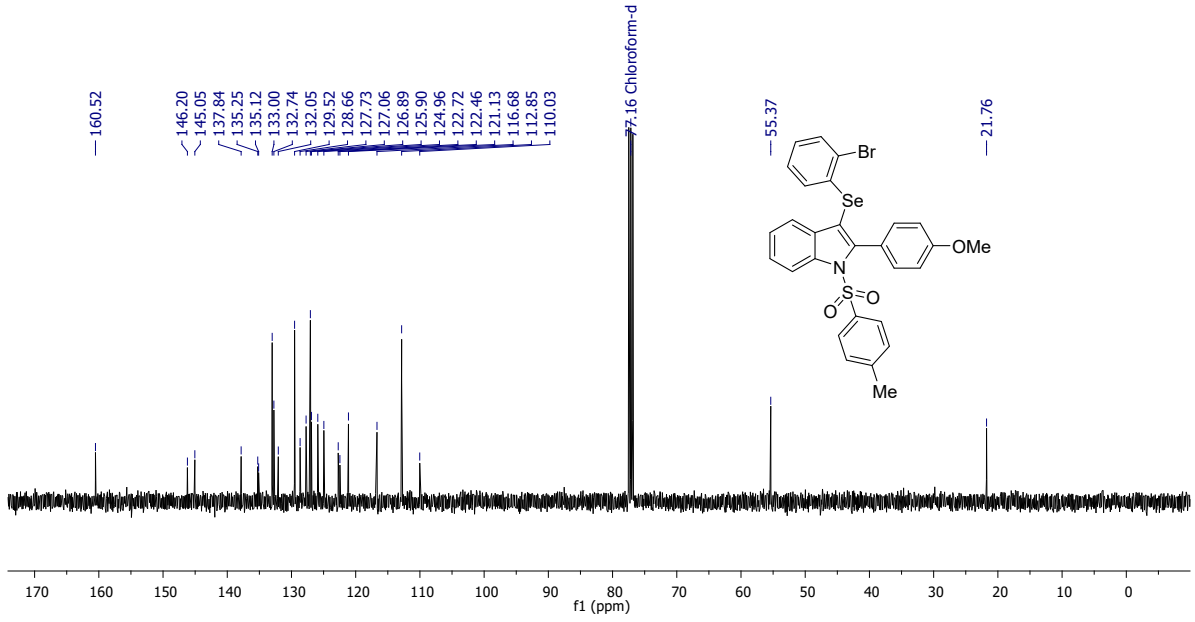
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ag** in $CDCl_3$



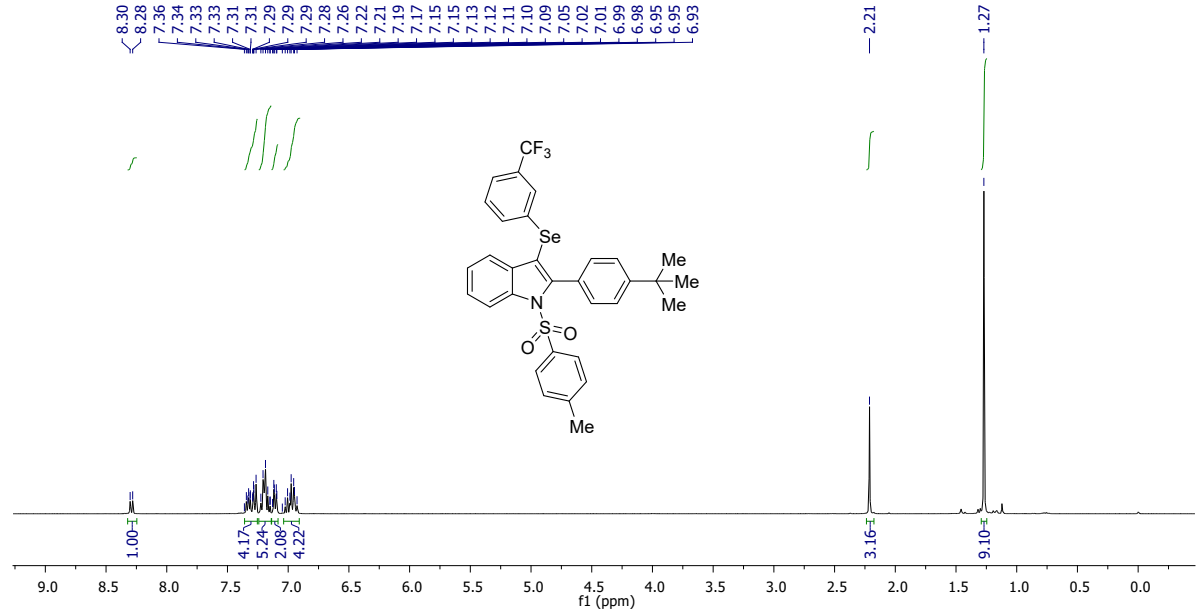
^{1}H-NMR (400 MHz) spectrum of **3dh** in $CDCl_3$



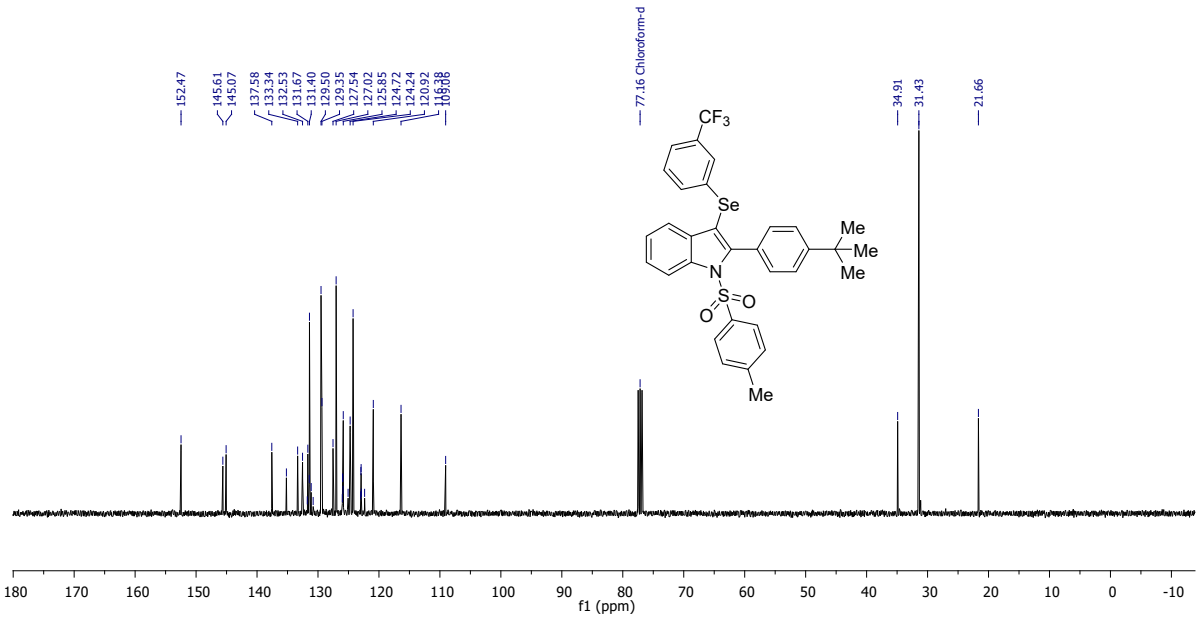
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3dh** in $CDCl_3$



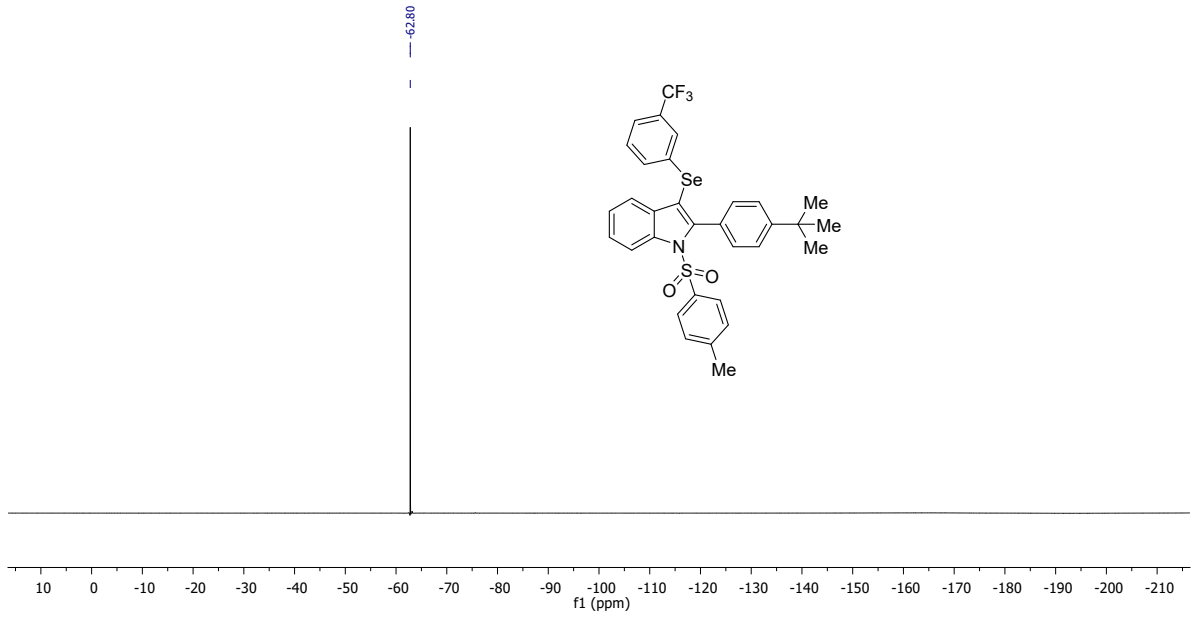
^{1}H-NMR (400 MHz) spectrum of **3ei** in $CDCl_3$



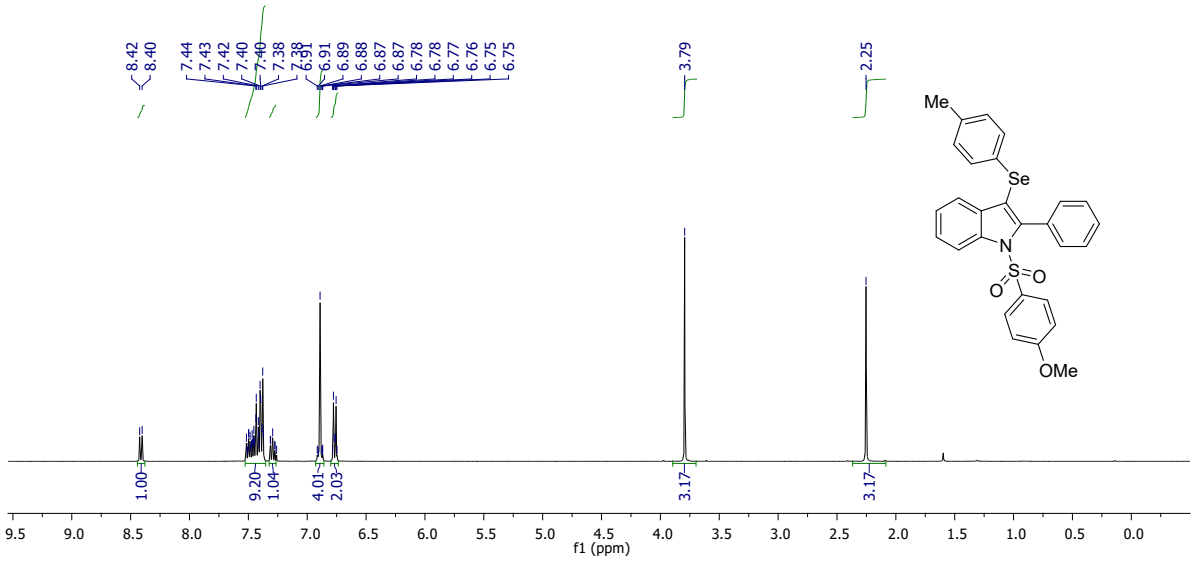
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ei** in $CDCl_3$



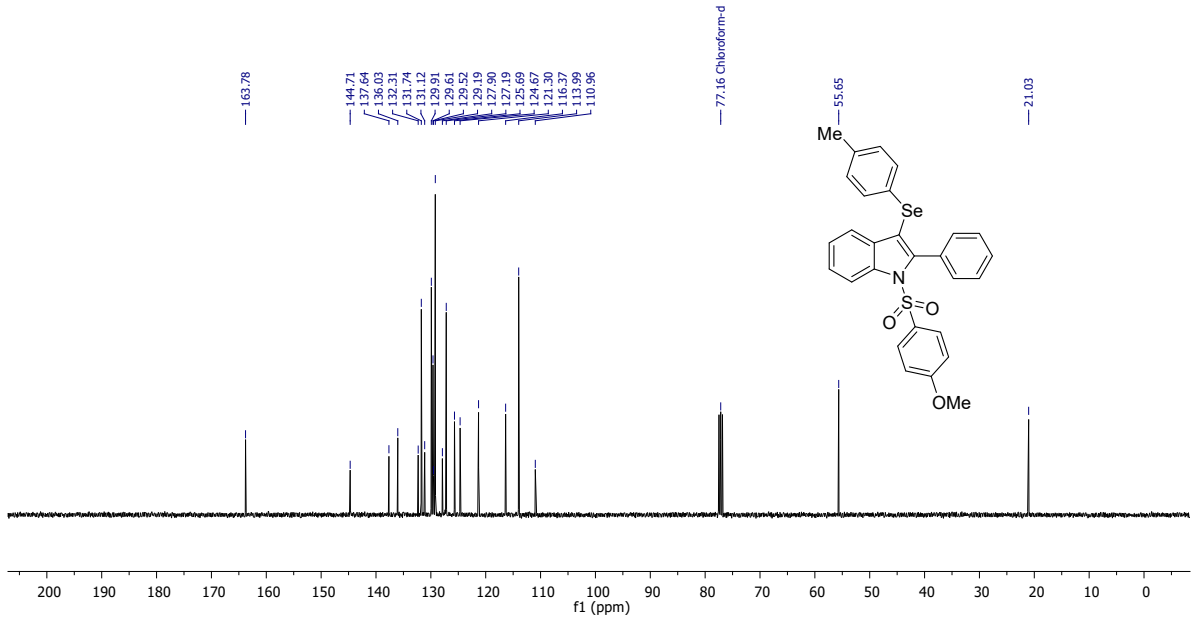
^{19}F NMR (565 MHz) spectrum of **3ei** in $CDCl_3$



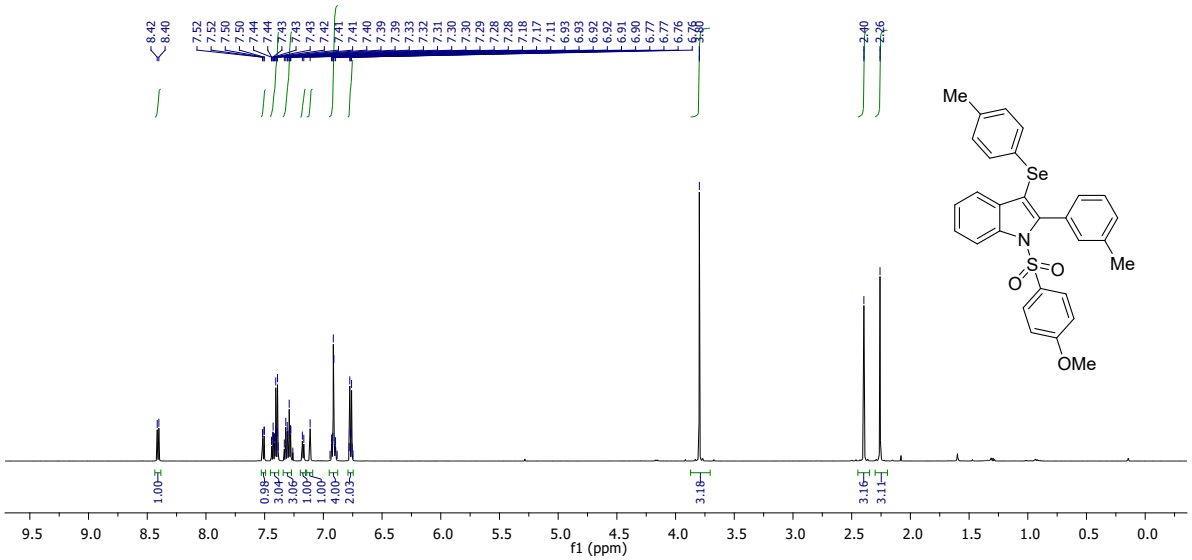
^{1}H-NMR (400 MHz) spectrum of **3rb** in $CDCl_3$



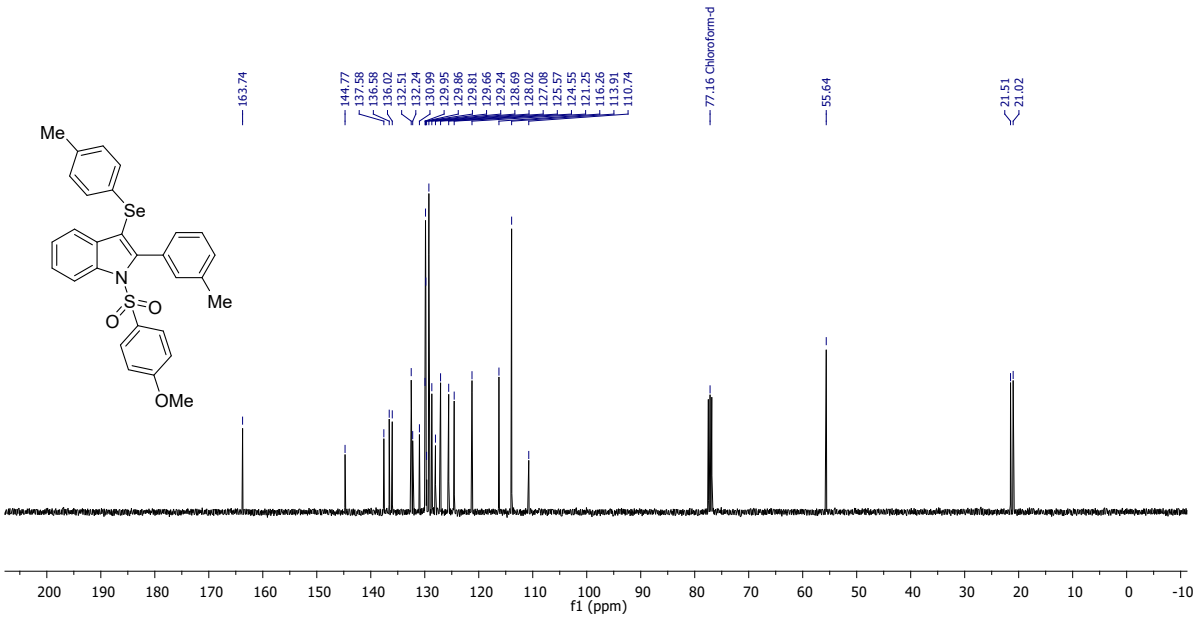
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3rb** in $CDCl_3$



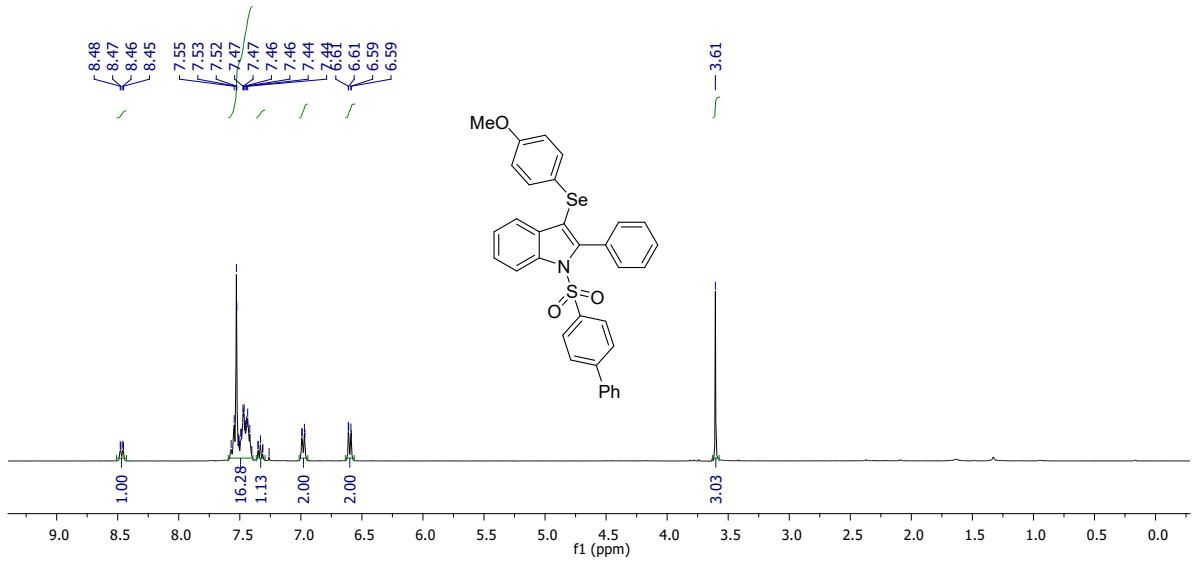
^{1}H-NMR (600 MHz) spectrum of **3sb** in $CDCl_3$



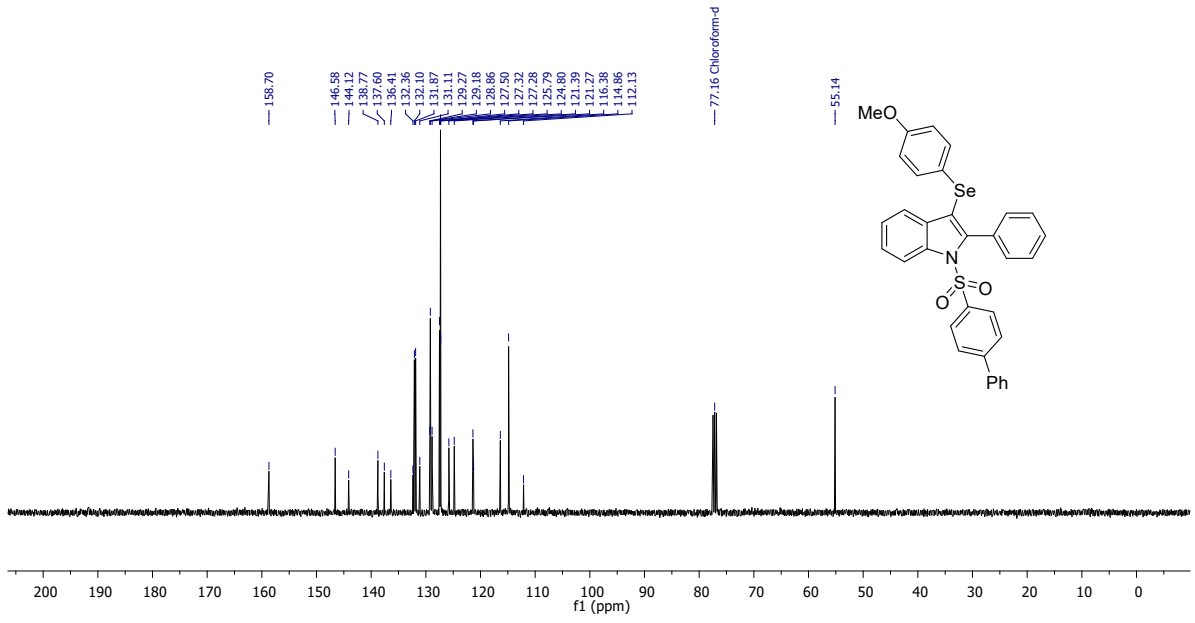
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3sb** in $CDCl_3$



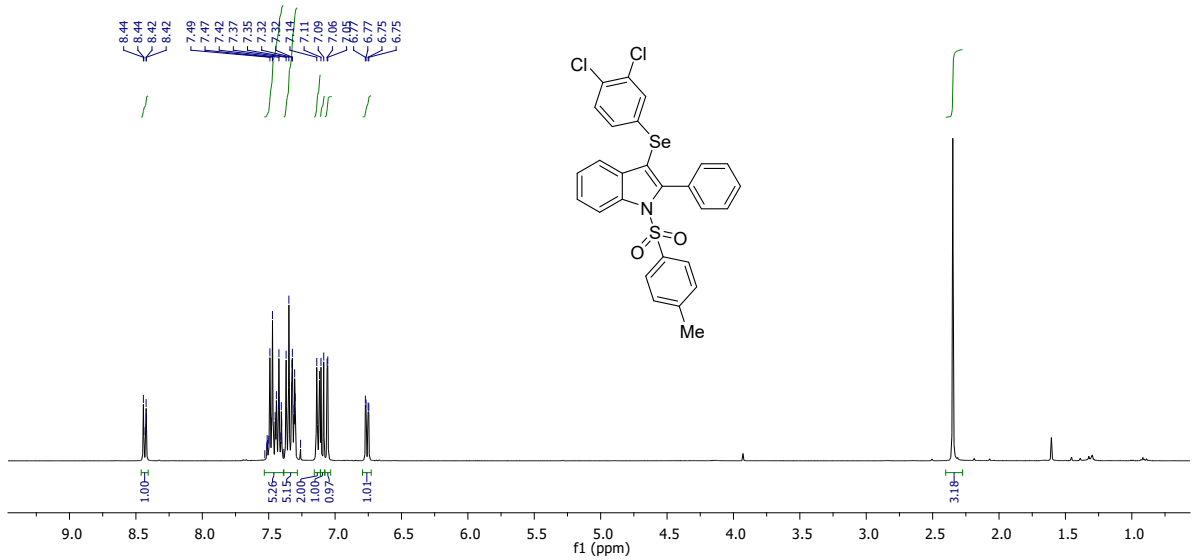
^{1}H-NMR (400 MHz) spectrum of **3qd** in $CDCl_3$



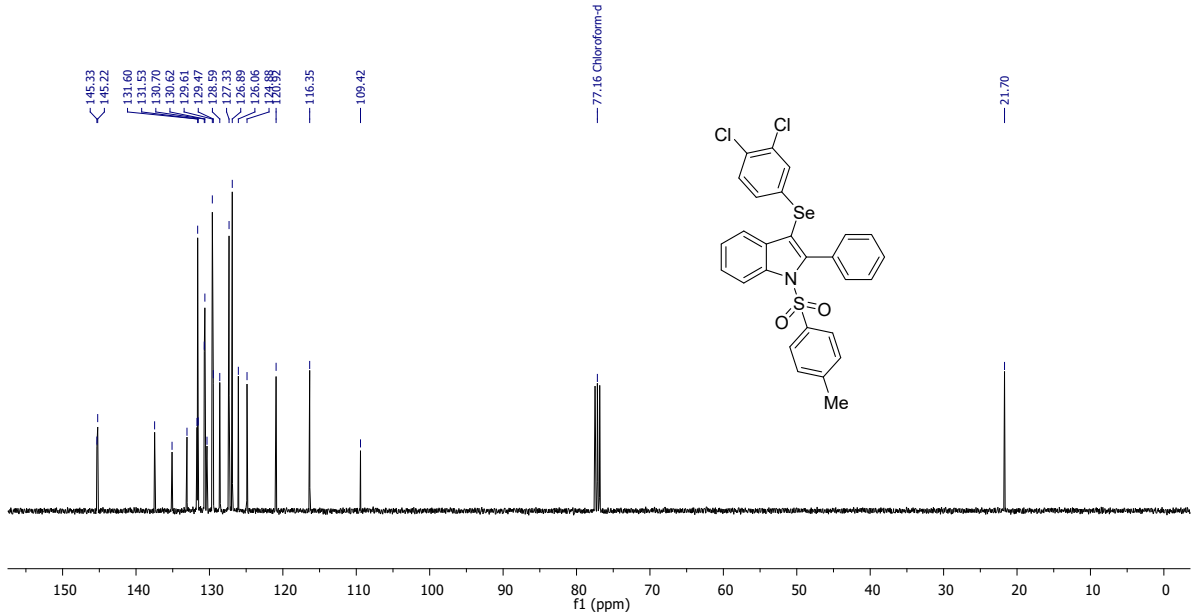
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3qd** in $CDCl_3$



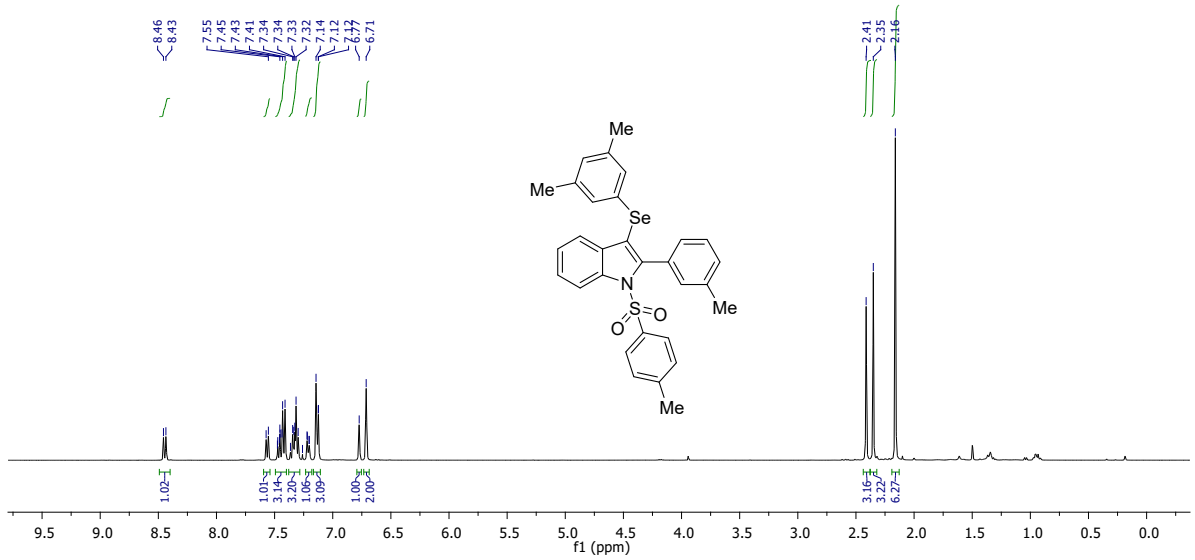
^{1}H-NMR (400 MHz) spectrum of **3aj** in $CDCl_3$



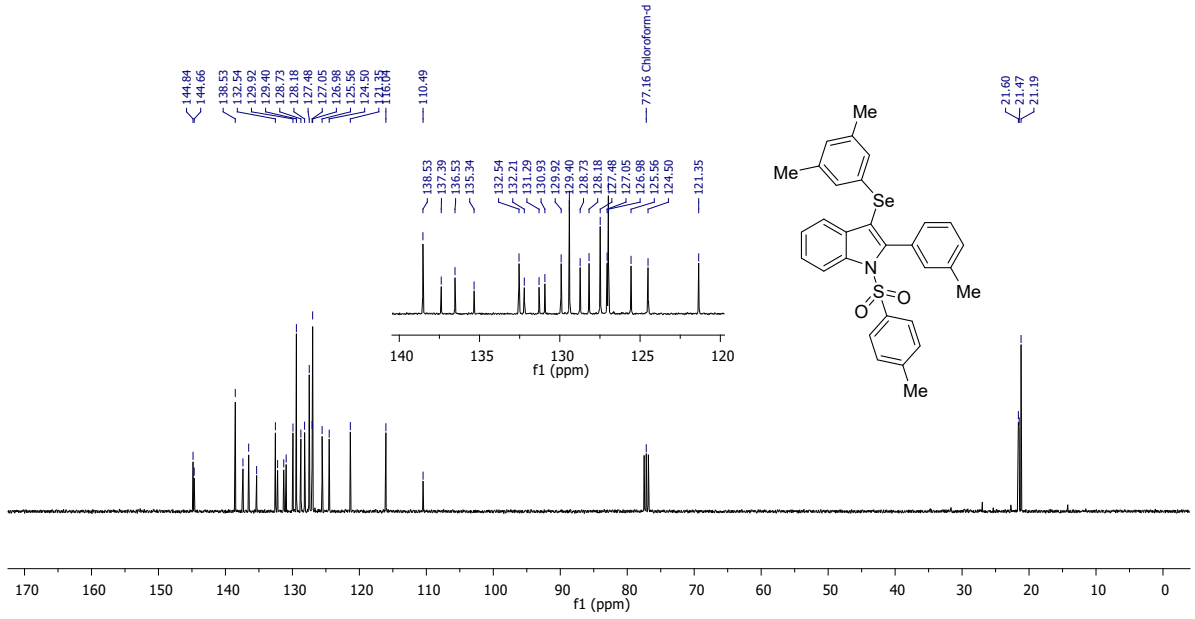
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3aj** in $CDCl_3$



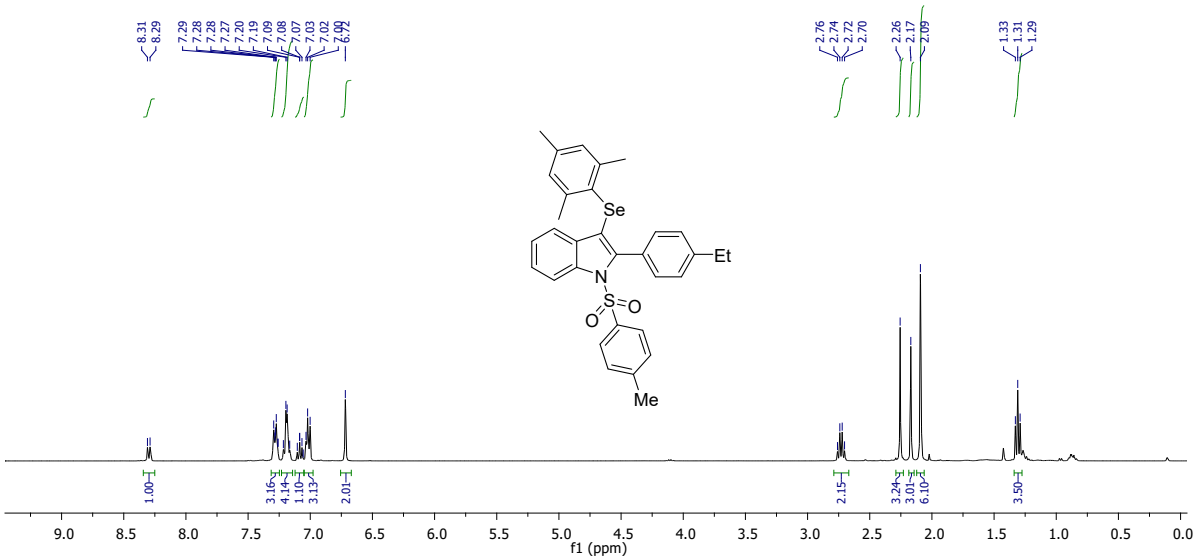
^{1}H-NMR (400 MHz) spectrum of **3kk** in $CDCl_3$



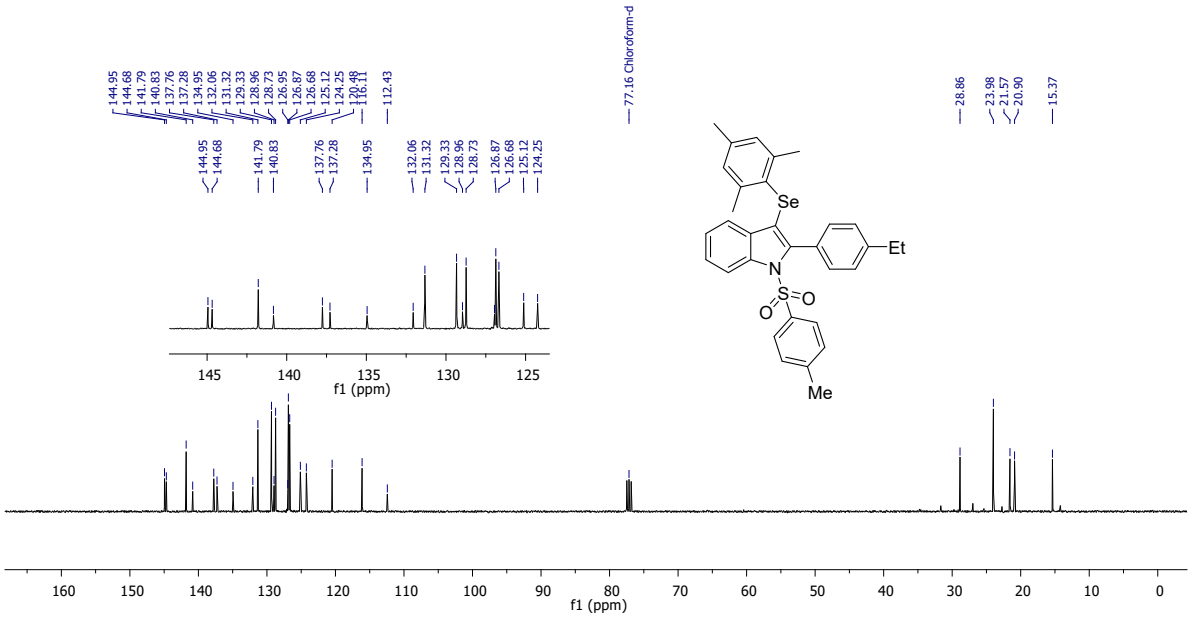
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3kk** in $CDCl_3$



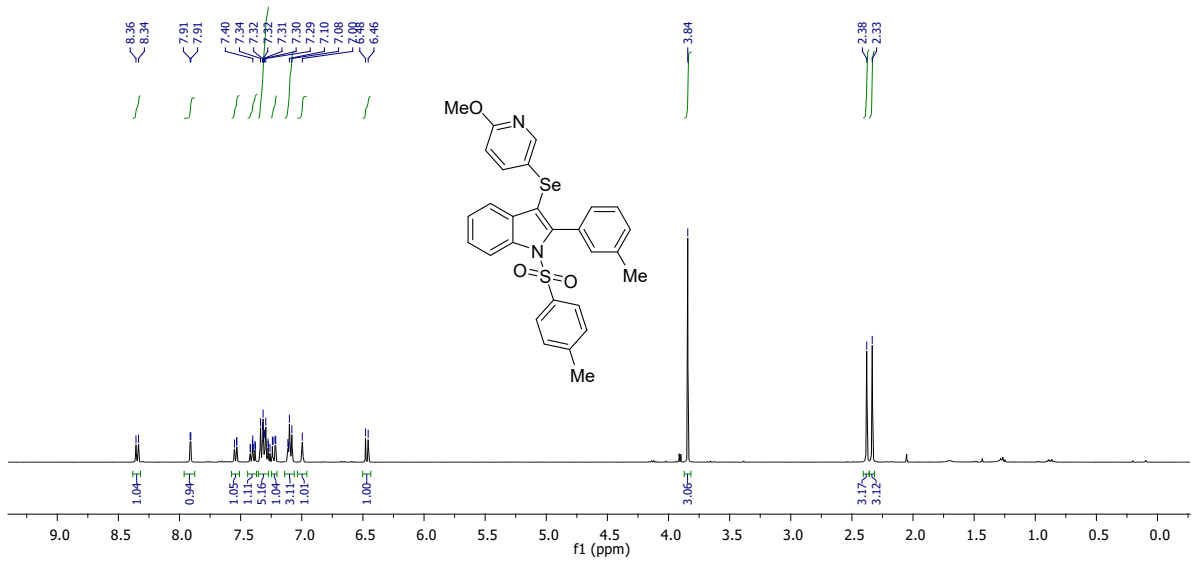
^{1}H-NMR (400 MHz) spectrum of **3cl** in $CDCl_3$



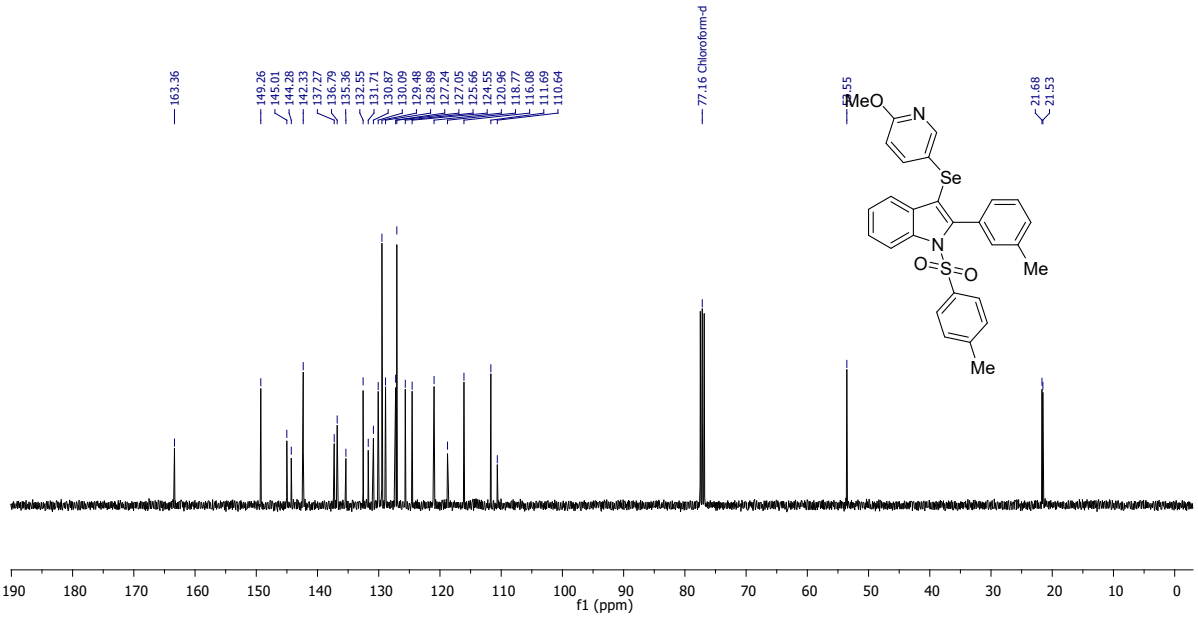
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3cl** in $CDCl_3$



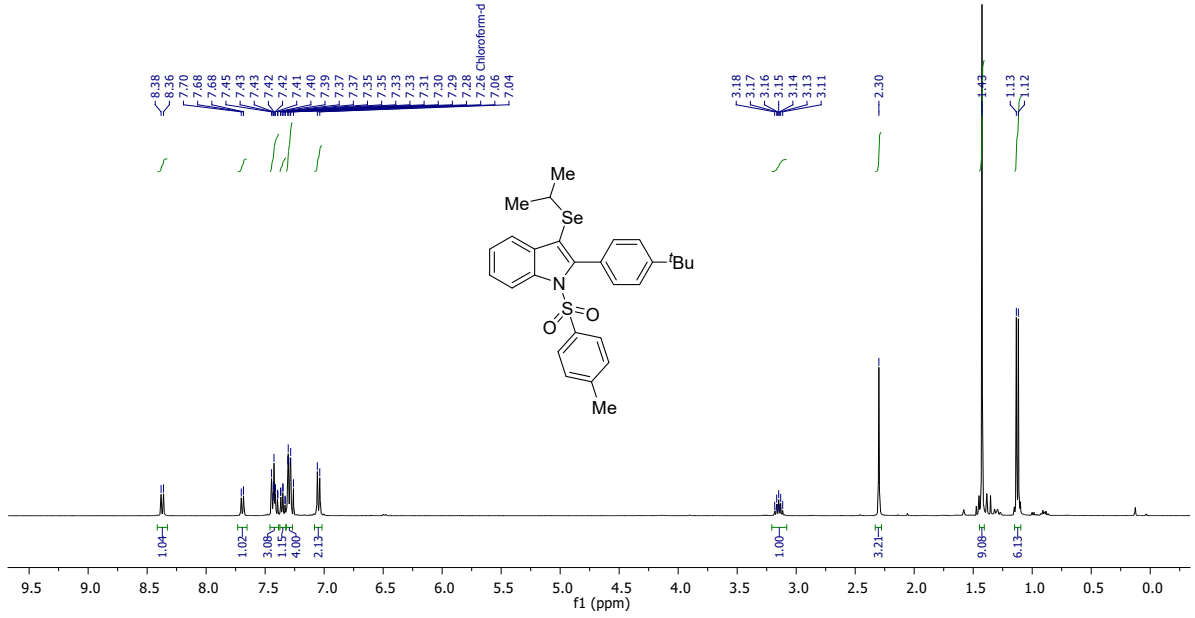
^{1}H-NMR (400 MHz) spectrum of **3km** in $CDCl_3$



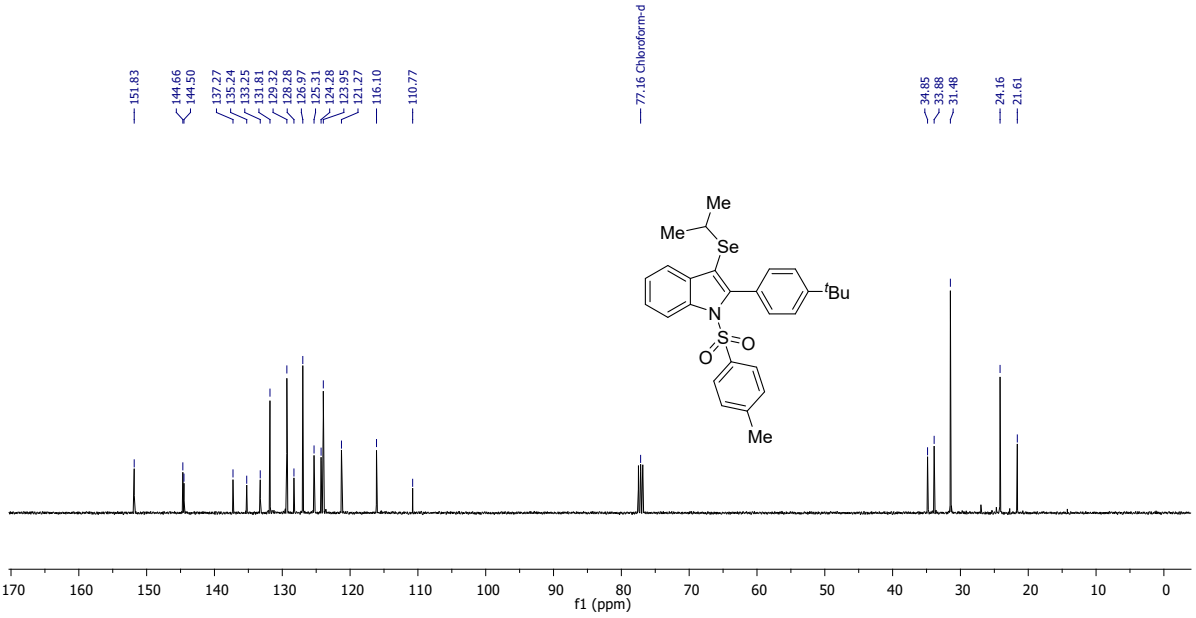
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3km** in $CDCl_3$



^{1}H-NMR (400 MHz) spectrum of **3en** in $CDCl_3$



^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3en** in $CDCl_3$



Crystal structure data:

X-Ray crystal structure of compound **3aa**: Crystal of compound **3aa** were obtained by dissolving the product in CH_2Cl_2 and allowing the solvent to slowly evaporate at room temperature. The crystal structure information for this compound has been deposited at the Cambridge Crystallographic Data Centre. **CCDC No. 2256835** contains the crystal structure information of this compound and can be obtained free of charge *via* http://www.ccdc.cam.ac.uk

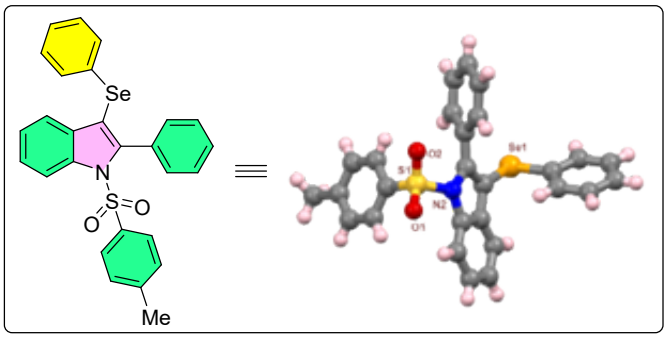


Figure S2: X-ray structure of the product **3aa** with the ellipsoids drawn at the 50% probability level (**CCDC**-2256835).

Table 4S: Crystal data and structure refinement for **3aa** (**CCDC**-2256835)

Table 1 Crystal data and structure refinement for mo_GS_AB_2_537_0m.	
Compound name	3aa
Empirical formula	$C_{27}H_{21}NO_2SSe$
Formula weight	502.47
Temperature/K	299.00
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	5.9597(4)
b/Å	18.6637(15)
c/Å	20.6440(13)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2296.2(3)
Z	4
ρ_{calc}g/cm^3	1.453
μ/mm^{-1}	1.751
F(000)	1024.0
Crystal size/mm^3	0.56 × 0.234 × 0.095
Radiation	MoKα (λ = 0.71073)

2Θ range for data collection/°	4.364 to 54.204	
Index ranges	$-7 \leq h \leq 5, -20 \leq k \leq 23, -26 \leq l \leq 26$	
Reflections collected	19920	
Independent reflections	5060 [R_{int} = 0.0579, R_{sigma} = 0.0543]	
Data/restraints/parameters	5060/0/290	
Goodness-of-fit on F^2	1.019	
Final R indexes [I>=2σ (I)]	R_1 = 0.0350, wR_2 = 0.0808	
Final R indexes [all data]	R_1 = 0.0449, wR_2 = 0.0854	
Largest diff. peak/hole / e $Å^{-3}$	0.22/-0.51	
Flack parameter	0.065(7)	

X-Ray crystal structure of compound **3ab**: Crystal of compound **3ab** were obtained by dissolving the product in CH_2Cl_2 and allowing the solvent to slowly evaporate at room temperature. The crystal structure information for this compound has been deposited at the Cambridge Crystallographic Data Centre. **CCDC No. 2244978** contains the crystal structure information of this compound and can be obtained free of charge *via* http://www.ccdc.cam.ac.uk

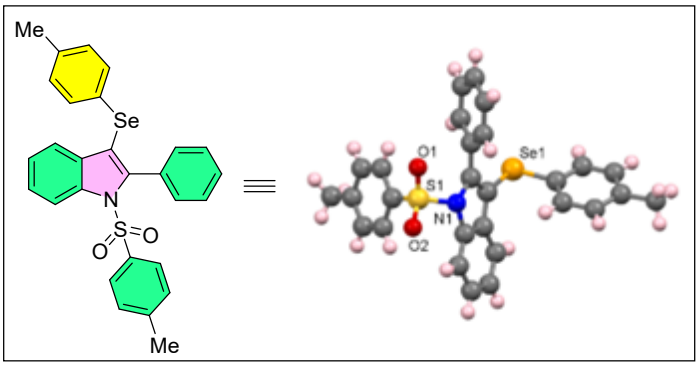


Figure S3: X-ray structure of the product **3ab** with the ellipsoids drawn at the 50% probability level (**CCDC**-2244978).

Table 5S: Crystal data and structure refinement for **3ab** (**CCDC**-2244978).

Table 1 Crystal data and structure refinement for mo_GS_AB_2_560_0m.	
Compound name	3ab
Empirical formula	$C_{28}H_{23}NO_2SSe$
Formula weight	516.49
Temperature/K	299.00
Crystal system	monoclinic
Space group	$P2_1$
a/Å	9.9442(10)
b/Å	11.3228(10)
c/Å	10.8455(10)
α/°	90
β/°	92.163(3)

γ/°	90
Volume/Å^3	1220.3(2)
Z	2
ρ_{calc}g/cm^3	1.406
μ/mm^{-1}	1.649
F(000)	528.0
Crystal size/mm^3	0.35 × 0.123 × 0.096
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.098 to 54.206
Index ranges	-12 ≤ h ≤ 12, -14 ≤ k ≤ 14, -13 ≤ l ≤ 13
Reflections collected	12026
Independent reflections	5161 [R_{int} = 0.0332, R_{sigma} = 0.0583]
Data/restraints/parameters	5161/1/295
Goodness-of-fit on F^2	1.018
Final R indexes [I>=2σ (I)]	R_1 = 0.0299, wR_2 = 0.0655
Final R indexes [all data]	R_1 = 0.0364, wR_2 = 0.0698
Largest diff. peak/hole / e Å^{-3}	0.22/-0.29
Flack parameter	0.042(6)

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