Supporting Information

Metal-free iodine-promoted strategy for the synthesis of C2, C3-bissulfenylated indoles

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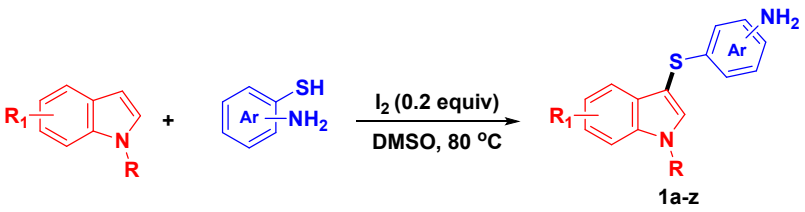
General information

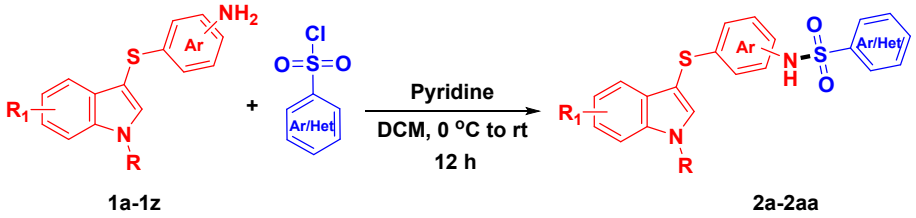
Commercially available chemicals were purchased from Alfa Aesar, Avra, Finar, and Sigma-Aldrich and used as received. All the starting materials were synthesized according to the reported procedures. Thin-layer chromatography (TLC) was performed using Merck silica gel 60 F_{254} precoated plates (0.25 mm) and visualized by a UV lamp for reaction monitoring. Silica gel for column chromatography (particle size 100–200 mesh) was purchased from Finar Limited India. ^{1}H and $^{13}C\{^{1}H\}$ NMR (nuclear magnetic resonance) spectra were recorded on a Bruker 400 MHz instrument. Chemical shifts were recorded in parts per million (ppm) relative to tetramethylsilane (δ 0.00), chloroform (7.26 ppm). ^{1}H NMR splitting patterns are designated as br s (broad singlet), s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets), m (multiplet), *etc*. $^{13}C\{^{1}H\}$ NMR spectral values were reported relative to $CDCl_3$ (77.16 ppm). High-resolution mass spectra were obtained on Agilent Q–Tof 6230 mass. The X-ray crystal-structure determinations of **3a** were obtained on Bruker Kappa Apex II**.**

General procedure for the preparation of 2-((1-alkyl-1*H*-indol-3-yl)thio)anilines (1a-1z)[1]



In a 100 mL round-bottom flask, 1-ethyl-7-methyl-1*H*-indole (250 mg, 1.72 mmol, 1.0 equiv.), 2-aminobenzenethiol (237 mg, 1.9 mmol, 1.1 equiv.), and iodine (87.4 mg, 0.34 mmol, 0.2 equiv.) were combined with 10 mL of DMSO. The mixture was stirred at 80 °C in an oil bath, and the reaction progress was monitored by thin-layer chromatography (TLC). Once the reaction was complete, the mixture was cooled to room temperature, washed with saturated sodium thiosulfate solution, and extracted with ethyl acetate (3 × 25 mL). The combined organic extracts were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography using a petroleum ether/ethyl acetate mixture (8.5:1.5, v/v) as the eluent, yielding the pure compound **1a**. The same procedure was followed to synthesize compounds **1b-1z**.

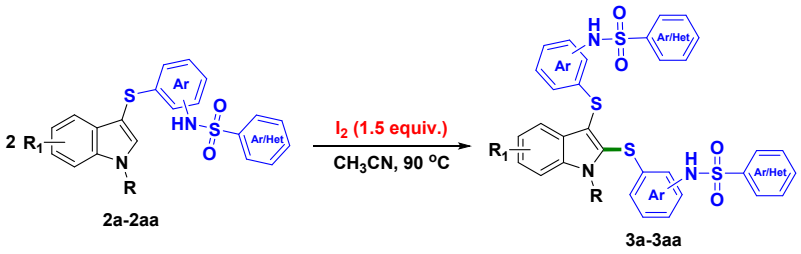
General procedure for the preparation of *N*-(2-((1-alkyl-1*H*-indol-3-yl)thio)phenyl)benzenesulfonamides (2a-2aa)[2]



1a-1z 2a-2aa

To a solution of 2-((1-ethyl-1*H*-indol-3-yl)thio)aniline **1a** (200 mg, 0.71 mmol, 1.0 equiv.) in 10 mL of dichloromethane, cooled to 0 °C, benzenesulfonyl chloride (157.9 mg, 0.89 mmol, 1.2 eq.) and pyridine (77.4 mmL, 0.97 mmol, 1.3 eq.) were added. The mixture was then allowed to warm to room temperature and stirred, until TLC confirmed complete consumption of the starting material. Then the reaction was quenched with 1N hydrochloric acid and extracted with CH_2Cl_2 (3 × 25 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography using a petroleum ether/ethyl acetate mixture (8:2, v/v) as the eluent, yielding pure compound **2a.** The same method was applied to prepare compounds **2b-2aa**.

General procedure for the preparation of compounds 3a-3aa (3a as an example)

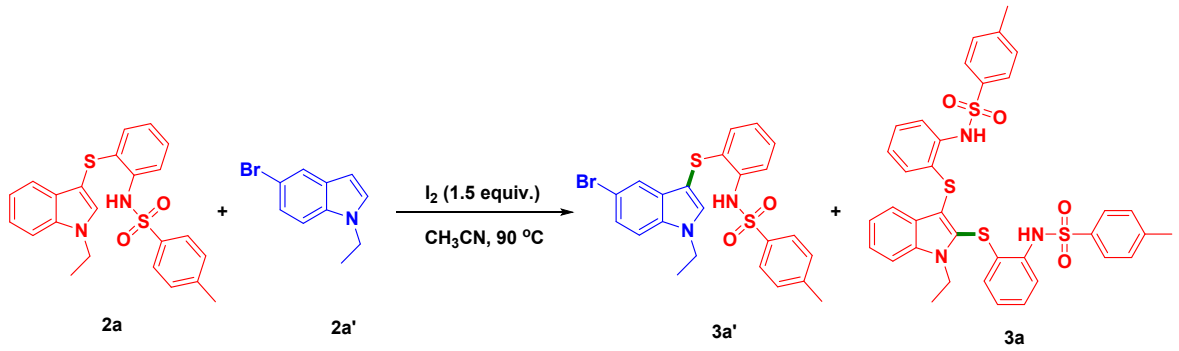


2a-2aa 3a-3aa

An oven-dried sealed tube was loaded with *N*-(2-((1-ethyl-1*H*-indol-3-yl)thio)phenyl)benzenesulfonamide **2a** (200 mg, 0.48 mmol, 1.0 equiv.), iodine (90.1 mg, 0.35 mmol, 1.5 equiv.), and 4 mL of acetonitrile. The mixture was stirred at 90 °C, and the reaction progress was monitored by TLC. Upon completion, the reaction mixture was washed with a sodium thiosulfate solution and extracted with ethyl acetate (3 × 20 mL). The combined organic layers were dried over anhydrous sodium sulphate and concentrated using a rotary evaporator. The resulting residue was purified by silica gel column chromatography with petroleum ether/ethyl acetate (8.5:1.5, v/v) as the eluent, yielding pure product **3a** in 131 mg, 79%. All the other compounds **3b-3aa** were synthesized using the same procedure.

Control experiments:

Experiment for the confirmation of aryl hypoiodothioite formation



The experiment was carried out using **2a** (30 mg, 0.071 mmol, 1.0 equiv.) and a C3-unsubstituted indole **2a′** (16 mg, 0.071 mmol, 1.0 equiv.) under the standard reaction conditions. The corresponding C3-sulfenylated indole **3a′** was obtained as the major product, along with a trace amount of a symmetrical bis-sulfenylated byproduct **3a**. The formation of the C3-sulfenylated product supports the in-situ generation of an electrophilic sulfenyl species, most plausibly an aryl hypoiodothioite. This species undergoes an electrophilic substitution reaction at the C3 position of the indole **2a′**, leading to C–S bond formation. The minor formation of the bissulfenylated product **3a** likely arises from a side pathway involving further C–S bond formation at the C2 position *via* sulfenyl species interaction.

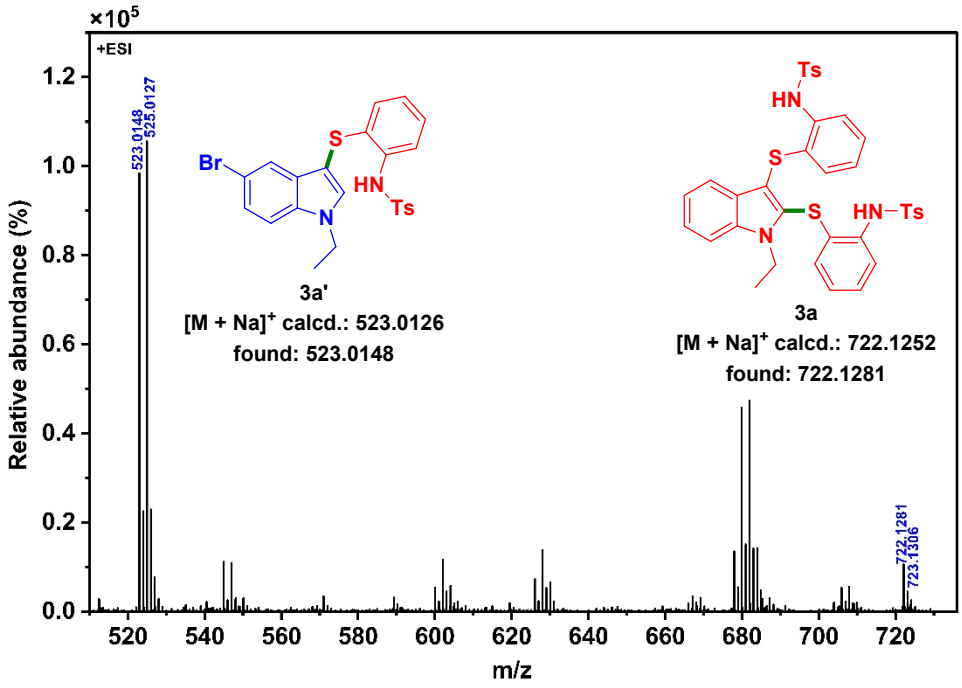


Figure S1. HRMS for the detection of **3a′** and **3a**

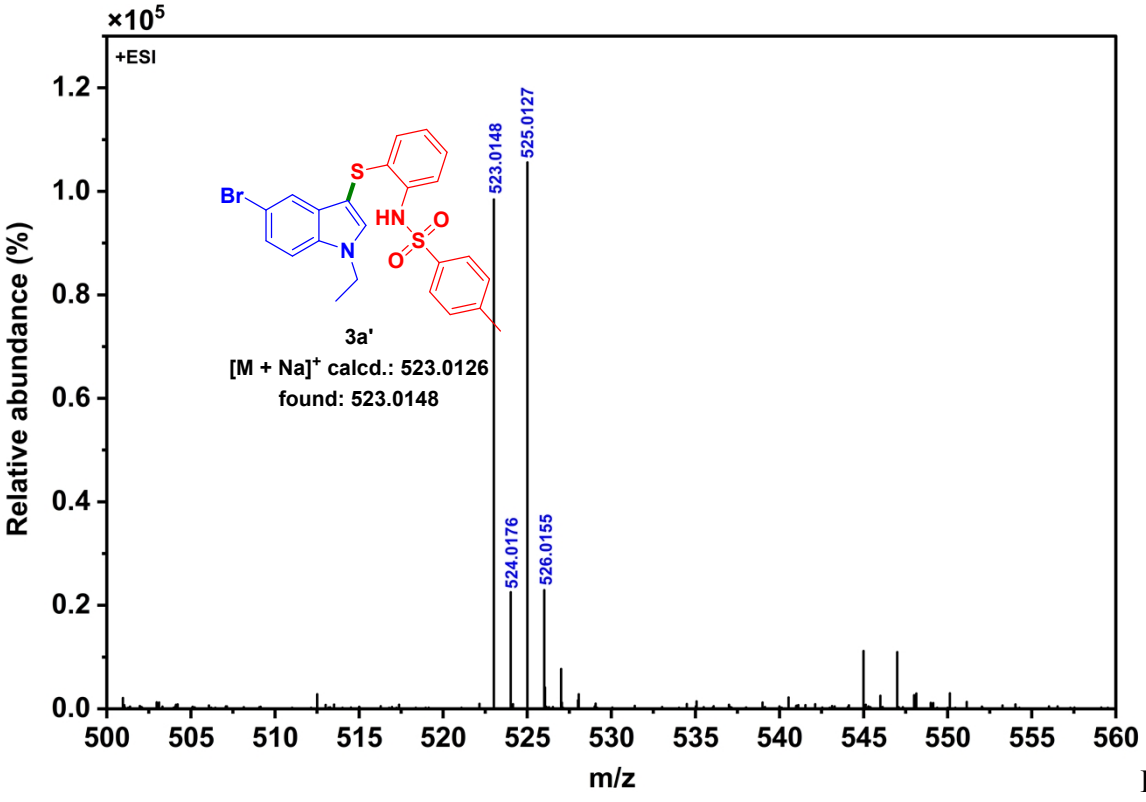


Figure S2. Expanded HRMS spectrum showing the signals corresponding to **3a′**

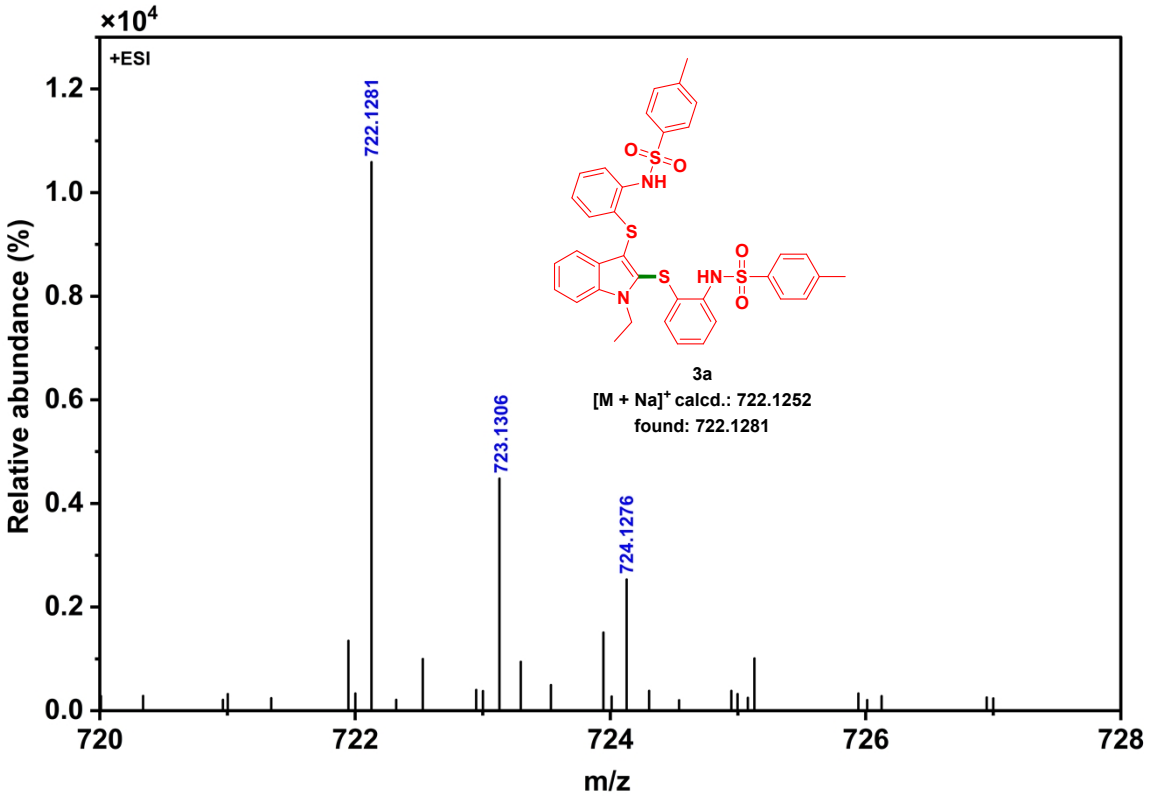
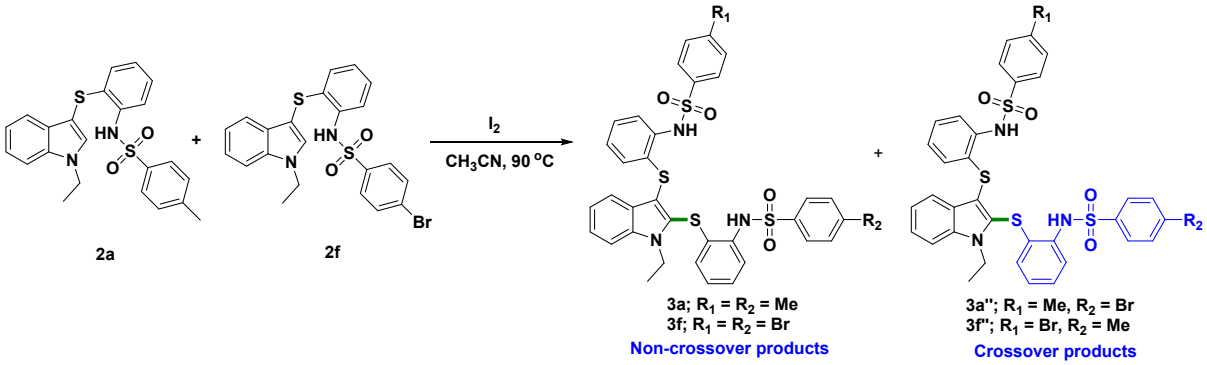


Figure S3. Expanded HRMS spectrum showing the signals corresponding to **3a**

Crossover experiment



The crossover experiment was carried out using two different C3-phenylthio indoles **2a** (30 mg, 0.071 mmol, 1.0 equiv.) and **2f** (34.6 mg, 0.071 mmol, 1.0 equiv.) under the standard conditions. In addition to the expected symmetrical or non-crossover C2, C3-bissulfenylated products **3a** and **3f**, the formation of crossover C2, C3-bissulfenylated products **3a′′** and **3f′′** was also observed in the HRMS (ESI). The formation of these mixed products indicates that the sulfenyl group is transferred through a common intermediate. These findings support the in-situ generation of a reactive electrophilic sulfenyl species, most likely an aryl hypoiodothioite (ArS-I), which can react with other indole substrates under the reaction conditions. HRMS for the non-crossover **3a, 3f** and crossover products **3a", 3f"** are given below.

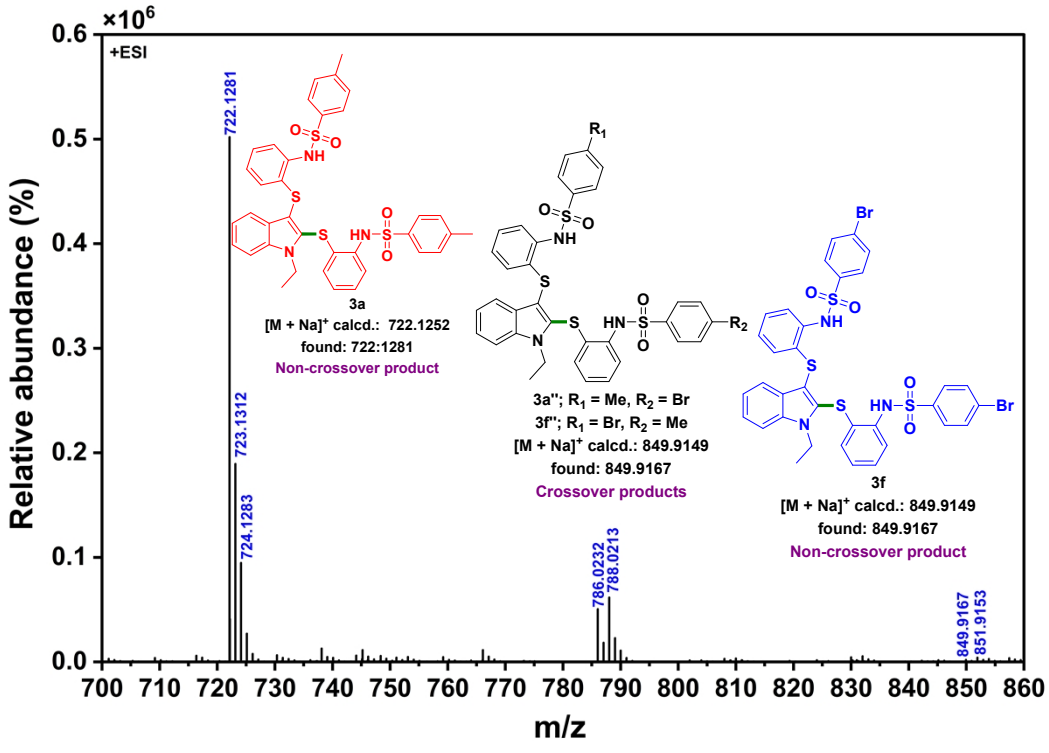


Figure S4. HRMS for the detection of non-crossover and crossover products

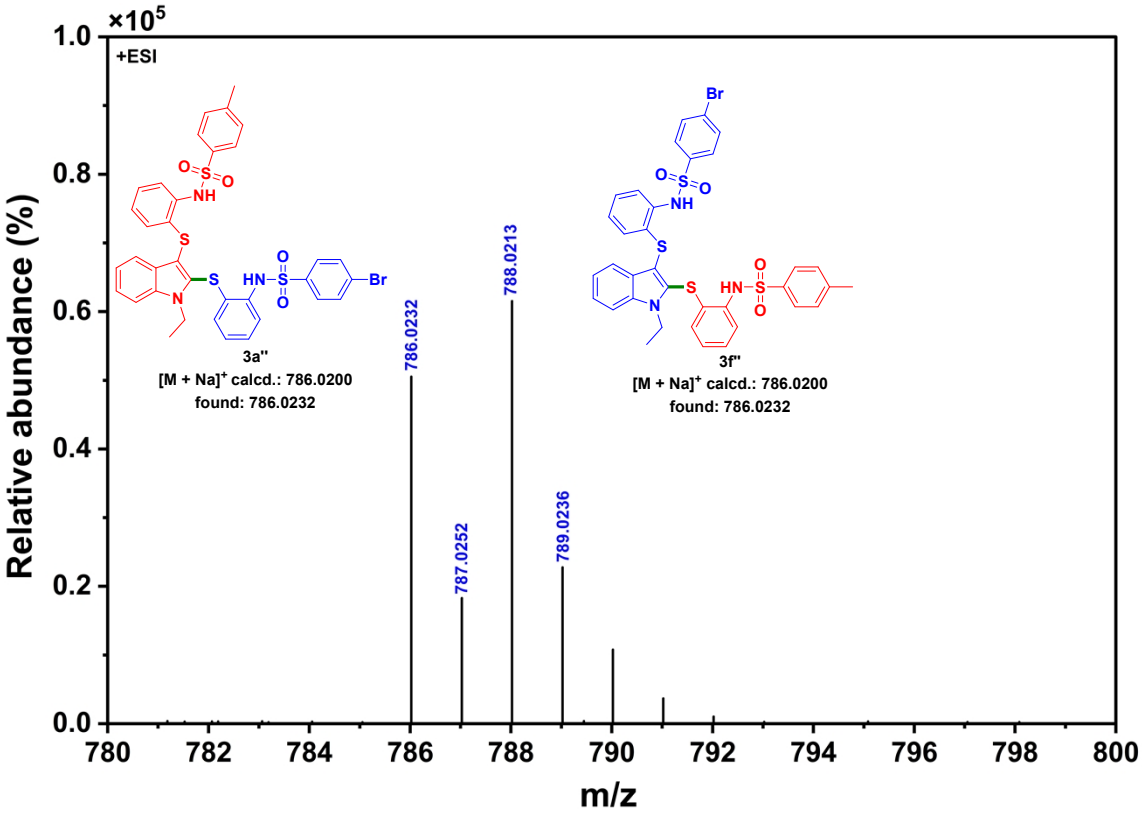
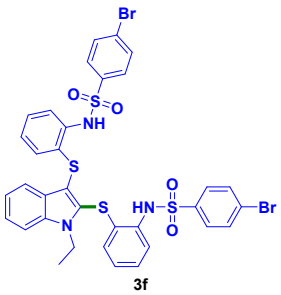


Figure S5. Expanded HRMS spectrum for the detection of **3a"** and **3f"**



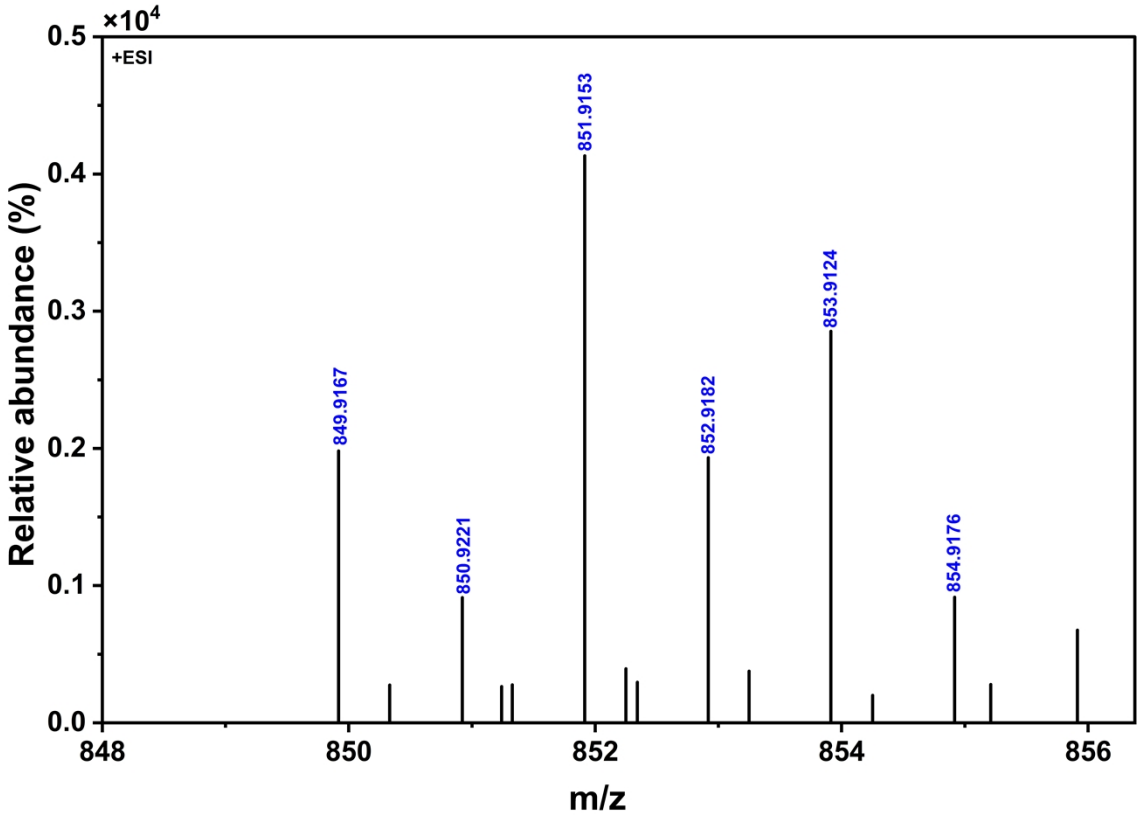
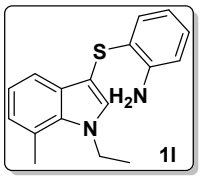


Figure S6. Expanded HRMS spectrum for the detection of **3f**

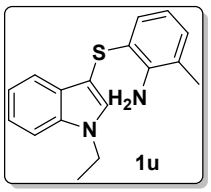
Spectroscopic and physical data

2-((1-Ethyl-7-methyl-1*H*-indol-3-yl)thio)aniline (1l)



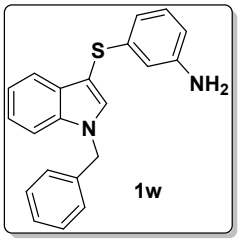
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Violet liquid; Yield: 370 mg, 69%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.2 Hz, 1H), 7.31 (s, 1H), 7.18 (d, *J* = 7.5 Hz, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 7.06 – 7.01 (m, 2H), 6.71 (dd, *J* = 7.9, 1.3 Hz, 1H), 6.70 – 6.61 (m, 1H), 4.41 (q, *J* = 7.2 Hz, 2H), 4.28 (br s, 2H), 2.77 (s, 3H), 1.49 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 145.3, 135.3, 133.6, 131.3, 130.8, 127.6, 125.4, 121.4, 121.3, 120.5, 118.9, 117.8, 115.3, 102.1, 43.8, 19.8, 17.8; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{17}H_{19}N_2S$: 283.1269; found: 283.1274.

2-((1-Ethyl-1*H*-indol-3-yl)thio)-6-methylaniline (1u)



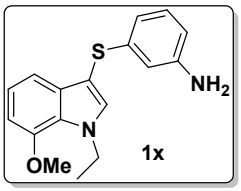
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Brown liquid; Yield: 331 mg, 68%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.49 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.37 (dd, *J* = 8.3, 0.9 Hz, 1H), 7.26 – 7.15 (m, 1H), 7.11 – 7.05 (m, 2H), 7.03 – 6.98 (m, 1H), 6.78 (dd, *J* = 8.1, 1.0 Hz, 1H), 5.06 (br s, 2H), 4.17 (q, *J* = 7.3 Hz, 2H), 2.62 (s, 3H), 1.46 (t, *J* = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.8, 137.0, 136.6, 136.1, 133.3, 129.6, 127.3, 126.7, 122.9, 122.5, 120.5, 119.7, 109.9, 100.3, 41.4, 21.6, 15.4; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{17}H_{19}N_2S$: 283.1269; found: 283.1268.

3-((1-Benzyl-1*H*-indol-3-yl)thio)aniline (1w)



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); White solid; Yield: 298 mg, 75%; m.p.: 131-133 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 – 7.69 (m, 1H), 7.42 – 7.34 (m, 2H), 7.34 – 7.28 (m, 3H), 7.24 (dd, *J* = 6.9, 1.3 Hz, 1H), 7.21 (dd, *J* = 7.6, 1.4 Hz, 2H), 7.19 – 7.15 (m, 2H), 7.07 – 6.99 (m, 1H), 6.71 (dd, *J* = 7.9, 1.3 Hz, 1H), 6.68 – 6.59 (m, 1H), 5.33 (br s, 2H), 3.44 (s, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 145.5, 137.0, 136.8, 133.0, 131.7, 129.7, 128.9, 127.9 (2C), 126.9, 122.7, 121.0, 120.5, 119.8, 118.9, 115.3, 110.2, 103.0, 50.4; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{21}H_{19}N_2S$: 331.1269; found: 331.1268.

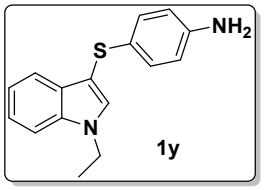
3-((1-Ethyl-7-methoxy-1*H*-indol-3-yl)thio)aniline (1x)



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Brown solid; Yield: 271 mg, 64%; m.p.: 164-166 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, *J* = 8.4, 1.0 Hz, 1H), 7.52 (s, 1H), 7.49 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.39 – 7.16 (m, 2H), 6.72 (d, *J* = 2.8 Hz, 1H), 6.66 (d, *J* = 8.6 Hz, 1H), 6.60 (dd, *J* = 8.6, 2.8 Hz, 1H), 4.21 (q, *J* = 7.3 Hz, 2H), 3.62

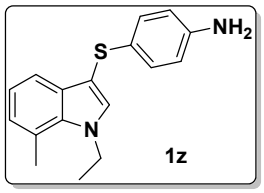
(s, 3H), 3.17 (br s, 2H), 1.50 (t, J = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 154.9, 145.3, 132.5, 131.7, 131.1, 130.5, 127.7, 121.7, 119.0, 115.4, 112.9, 110.7, 101.2, 101.0, 55.9, 41.6, 15.5; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{17}H_{19}N_2OS$: 299.1218; found: 299.1211.

4-((1-Ethyl-1*H*-indol-3-yl)thio)aniline (1y)



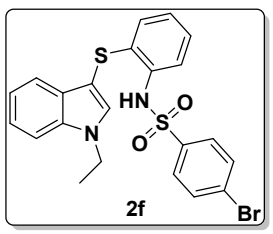
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); White solid; Yield: 390 mg, 84%; m.p.: 135-137 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 7.8 Hz, 1H), 7.42 – 7.38 (m, 2H), 7.34 – 7.26 (m, 1H), 7.22 – 7.13 (m, 1H), 7.08 (d, J = 8.6 Hz, 2H), 6.59 (d, J = 8.6 Hz, 2H), 4.21 (q, J = 7.3 Hz, 2H), 3.73 (br s, 2H), 1.52 (t, J = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 143.3, 136.5, 132.5, 129.9, 128.9, 128.0, 122.3, 120.2, 119.9, 116.2, 109.7, 102.9, 41.3, 15.4; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{16}H_{17}N_2S$: 269.1112; found: 269.1108.

4-((1-Ethyl-7-methyl-1*H*-indol-3-yl)thio)aniline (1z)



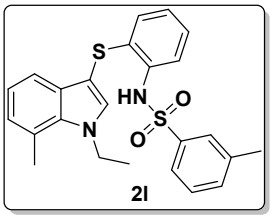
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Violet liquid; Yield: 370 mg, 69%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (dd, J = 7.7, 1.7 Hz, 1H), 7.38 – 7.27 (m, 1H), 7.26 (s, 1H), 7.11 – 7.02 (m, 1H), 6.99 (dd, J = 7.7, 1.3 Hz, 1H), 6.99 – 6.91 (m, 2H), 6.74 (dd, J = 8.1, 1.3 Hz, 1H), 4.68 (br s, 2H), 4.38 (q, J = 7.2 Hz, 2H), 2.70 (s, 3H), 1.43 (t, J = 7.2 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 136.7, 136.1, 134.5, 133.2, 130.1, 129.5, 126.1, 122.4, 120.4, 119.9, 109.8, 101.2, 41.4, 20.9, 15.4; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{17}H_{19}N_2S$: 283.1269; found: 283.1261.

4-Bromo-*N*-(2-((1-ethyl-1*H*-indol-3-yl)thio)phenyl)benzenesulfonamide (2f)



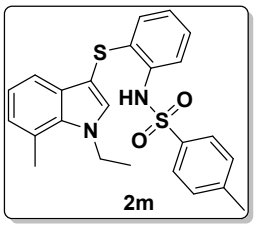
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Brown solid; Yield: 256 mg, 71%; m.p.: 113-115 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.55 (m, 2H), 7.57 – 7.47 (m, 3H), 7.43 – 7.39 (m, 2H), 7.34 – 7.28 (m, 3H), 7.20 – 7.11 (m, 2H), 7.08 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.00 – 6.91 (m, 1H), 4.20 (q, *J* = 7.3 Hz, 2H), 1.51 (t, *J* = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 138.2, 136.5, 134.4, 132.5, 132.1, 130.4, 129.9, 129.2, 128.9, 128.1, 127.5, 125.9, 122.8, 122.4, 120.8, 119.3, 110.0, 99.5, 41.5, 15.4; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{22}H_{20}BrN_2O_2S_2$: 487.0150; found: 487.0130.

***N*-(2-((1-Ethyl-7-methyl-1*H*-indol-3-yl)thio)phenyl)-3-methylbenzenesulfonamide (2l)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Biege solid; Yield: 330 mg, 85%; m.p.: 136-138 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (s, 1H), 7.62 – 7.51 (m, 2H), 7.49 (s, 1H), 7.37 (d, *J* = 7.7 Hz, 1H), 7.33 – 7.22 (m, 2H), 7.23 – 7.15 (m, 1H), 7.16 – 7.06 (m, 2H), 7.04 – 6.95 (m, 2H), 6.90 (td, *J* = 7.6, 1.4 Hz, 1H), 4.40 (q, *J* = 7.2 Hz, 2H), 2.74 (s, 3H), 2.36 (s, 3H), 1.48 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 139.2, 139.1, 135.3, 135.1, 134.1, 133.8, 130.7, 130.4, 129.0, 128.8, 127.8, 127.5, 125.7, 125.4, 124.5, 121.6, 121.4, 120.8, 117.4, 100.3, 43.9, 21.3, 19.7, 17.7; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{24}H_{25}N_2O_2S_2$: 437.1357; found: 437.1362.

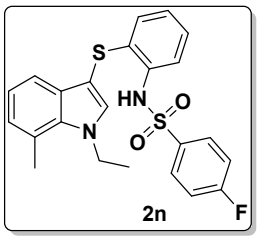
***N*-(2-((1-Ethyl-7-methyl-1*H*-indol-3-yl)thio)phenyl)-4-methylbenzenesulfonamide (2m)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Biege solid; Yield: 341 mg, 88%; m.p.: 128-130 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.61 (m, 2H), 7.59 – 7.52 (m, 1H), 7.49 – 7.43 (m, 1H), 7.25 (d, *J* = 1.4 Hz, 1H), 7.20 (d, *J* = 8.1 Hz, 2H), 7.19 – 7.14 (m, 1H), 7.16 – 7.05 (m, 2H), 7.04 – 6.94 (m, 2H), 6.90 (td, *J* = 7.5, 1.4 Hz, 1H), 4.40 (q, *J* = 7.2 Hz, 2H), 2.75 (s, 3H), 2.41 (s, 3H), 1.48 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.8, 136.3, 135.3, 135.1, 134.1, 130.6, 130.3, 129.5, 129.0, 127.4 (2C),

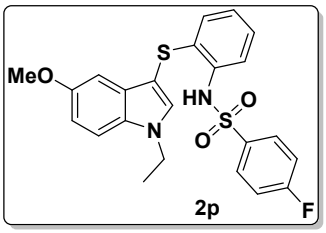
125.7, 125.3, 121.6, 121.4, 120.7, 117.4, 100.2, 43.9, 21.6, 19.7, 17.7; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{24}H_{25}N_2O_2S_2$: 437.1357; found: 437.1361.

***N*-(2-((1-Ethyl-7-methyl-1*H*-indol-3-yl)thio)phenyl)-4-fluorobenzenesulfonamide (2n)**



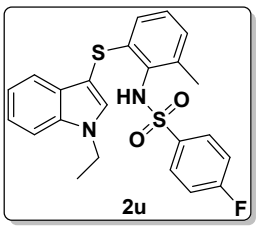
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pale brown solid; Yield: 310 mg, 80%; m.p.: 134-136 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 – 7.67 (m, 2H), 7.57 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.41 (s, 1H), 7.23 (s, 1H), 7.17 – 7.13 (m, 2H), 7.11 (dd, *J* = 10.4, 1.6 Hz, 1H), 7.09 – 7.04 (m, 2H), 7.04 – 6.99 (m, 2H), 6.94 (td, *J* = 7.6, 1.4 Hz, 1H), 4.41 (q, *J* = 7.2 Hz, 2H), 2.75 (s, 3H), 1.48 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 165.3 (d, *J* = 255.1 Hz), 135.3, 135.2 (d, *J* = 3.1 Hz), 134.6, 134.0, 130.5, 130.3, 130.1 (d, *J* = 9.4 Hz), 129.6, 127.5, 125.8 (2C), 122.3, 121.5, 120.9, 117.4, 116.1 (d, *J* = 22.7 Hz), 99.9, 43.9, 19.7, 17.7; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{23}H_{22}FN_2O_2S_2$: 441.1107; found: 441.1100.

***N*-(2-((1-Ethyl-5-methoxy-1*H*-indol-3-yl)thio)phenyl)-4-fluorobenzenesulfonamide (2p)**



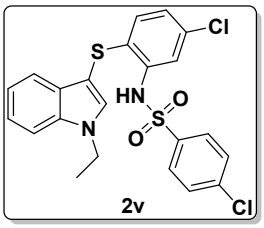
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pale brown solid; Yield: 327 mg, 85%; m.p.: 110-113 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.66 (m, 2H), 7.54 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.42 (s, 1H), 7.28 (t, *J* = 4.4 Hz, 2H), 7.19 – 7.09 (m, 2H), 7.09 – 7.00 (m, 2H), 7.00 – 6.90 (m, 2H), 6.85 (d, *J* = 2.5 Hz, 1H), 4.15 (q, *J* = 7.3 Hz, 2H), 3.81 (s, 3H), 1.49 (t, *J* = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 165.3 (d, *J* = 254.9 Hz), 155.1, 135.2 (d, *J* = 3.3 Hz), 134.7, 132.8, 131.6, 130.6, 130.1 (d, *J* = 9.4 Hz), 130.0, 129.8, 127.6, 125.8, 122.1, 116.0 (d, *J* = 22.5 Hz), 113.2, 110.9, 100.6, 98.8, 55.8, 41.6, 15.4; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{23}H_{22}FN_2O_3S_2$: 457.1056; found: 457.1061.

***N*-(2-((1-Ethyl-1*H*-indol-3-yl)thio)-6-methylphenyl)-4-fluorobenzenesulfonamide (2u)**



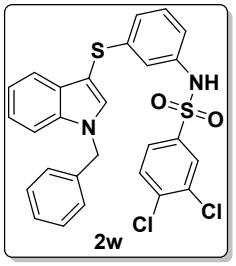
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pink solid; Yield: 226 mg, 72%; m.p.: 99-101 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.60 (m, 1H), 7.56 – 7.49 (m, 1H), 7.42 (d, *J* = 8.3 Hz, 1H), 7.37 (s, 1H), 7.36 – 7.25 (m, 1H), 7.19 – 7.11 (m, 1H), 7.03 – 6.92 (m, 4H), 6.92 (d, *J* = 9.0 Hz, 2H), 6.80 (s, 1H), 4.23 (q, *J* = 7.3 Hz, 2H), 2.39 (s, 3H), 1.53 (t, *J* = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.5 (d, *J* = 253.5 Hz), 147.2 (d, *J* = 8.4 Hz), 137.2, 136.6, 133.3, 132.8, 130.7, 129.7, 126.7, 125.2 (d, *J* = 3.3 Hz), 123.7 (d, *J* = 13.2 Hz), 122.5, 122.4, 120.5, 119.7, 117.2 (d, *J* = 20.9 Hz), 109.9, 100.2, 41.4, 21.6, 15.4; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{23}H_{22}FN_2O_2S_2$: 441.1107; found: 441.1104.

4-Chloro-*N*-(5-chloro-2-((1-ethyl-1*H*-indol-3-yl)thio)phenyl)benzenesulfonamide (2v)



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pink solid; Yield: 241 mg, 76%; m.p.: 113-115 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.79 – 7.69 (m, 3H), 7.50 – 7.42 (m, 4H), 7.36 (d, *J* = 8.9 Hz, 2H), 7.34 – 7.22 (m, 2H), 7.05 – 6.97 (m, 1H), 4.25 (q, *J* = 7.3 Hz, 2H), 1.58 (t, *J* = 7.3 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, CDCl3) δ 141.0, 139.2, 136.8, 133.7, 132.8, 131.3, 129.7, 128.6, 127.7, 126.4, 126.3, 125.0, 122.7, 120.7, 119.6, 119.6, 110.0, 99.5, 41.4, 15.3; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{22}H_{19}Cl_2N_2O_2S_2$: 477.0265; found: 477.0256.

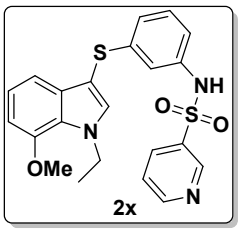
***N*-(3-((1-Benzyl-1*H*-indol-3-yl)thio)phenyl)-3,4-dichlorobenzenesulfonamide (2w)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pink solid; Yield: 230 mg, 70%; m.p.: 98-100 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, *J* = 2.2 Hz, 1H),

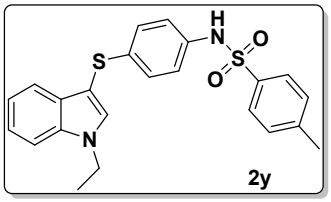
7.97 (s, 1H), 7.48 (s, 1H), 7.47 – 7.44 (m, 2H), 7.40 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.38 – 7.31 (m, 5H), 7.29 – 7.20 (m, 1H), 7.20 – 7.13 (m, 4H), 7.14 – 7.06 (m, 1H), 6.93 (td, *J* = 7.6, 1.3 Hz, 1H), 5.34 (s, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 138.6, 137.1, 136.4, 134.1, 134.0, 133.8, 133.2, 133.0, 131.4, 130.9, 130.5, 129.4, 129.3, 129.0, 128.1, 127.5, 127.0, 125.7, 123.0, 121.1, 120.9, 119.4, 110.4, 101.0, 50.5; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{27}H_{21}Cl_2N_2O_2S_2$: 539.0421; found: 539.0416.

***N*-(3-((1-Ethyl-7-methoxy-1*H*-indol-3-yl)thio)phenyl)pyridine-3-sulfonamide (2x)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8:2, v/v); Violet solid; Yield: 234 mg, 79%; m.p.: 121-123 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.87 (d, *J* = 2.4 Hz, 1H), 8.67 (dd, *J* = 4.8, 1.7 Hz, 1H), 7.87 – 7.81 (m, 1H), 7.50 – 7.46 (m, 1H), 7.39 (s, 1H), 7.24 – 7.16 (m, 2H), 7.12 – 7.04 (m, 2H), 6.99 – 6.93 (m, 1H), 6.91 – 6.81 (m, 2H), 6.72 (d, *J* = 2.6 Hz, 1H), 4.05 (q, *J* = 7.3 Hz, 2H), 3.72 (s, 3H), 1.39 (t, *J* = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 155.1, 153.4, 148.2, 135.9, 134.9, 133.7, 132.9, 131.6, 130.7, 130.1, 130.0, 127.5, 126.4, 123.5, 123.1, 113.2, 110.9, 100.6, 98.2, 55.8, 41.6, 15.4; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{22}H_{22}N_3O_3S_2$: 440.1103; found: 440.1125.

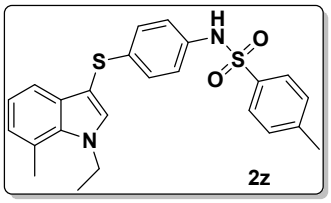
***N*-(4-((1-Ethyl-1*H*-indol-3-yl)thio)phenyl)-4-methylbenzenesulfonamide (2y)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); White solid; Yield: 272 mg, 86%; m.p.: 110-112 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.58 (m, 2H), 7.55 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.45 – 7.38 (m, 2H), 7.33 – 7.27 (m, 1H), 7.24 – 7.19 (m, 2H), 7.19 – 7.14 (m, 1H), 7.01 – 6.95 (m, 2H), 6.89 – 6.84 (m, 2H), 6.61 (s, 1H), 4.23 (q, *J* = 7.3 Hz, 2H), 2.39 (s, 3H), 1.53 (t, *J* = 7.3 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 143.8,

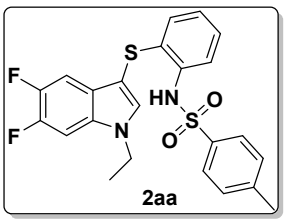
137.0, 136.6, 136.1, 133.3 (2C), 129.7, 129.6, 127.3, 126.7, 122.9, 122.5, 120.5, 119.7, 109.9, 100.3, 41.4, 21.6, 15.4; HRMS (ESI): m/z $[M + Na]^+$ calcd. for $C_{23}H_{22}N_2NaO_2S_2$: 445.1020; found: 445.1002.

_N_-(4-((1-Ethyl-7-methyl-1_H_-indol-3-yl)thio)phenyl)-4-methylbenzenesulfonamide (2z)



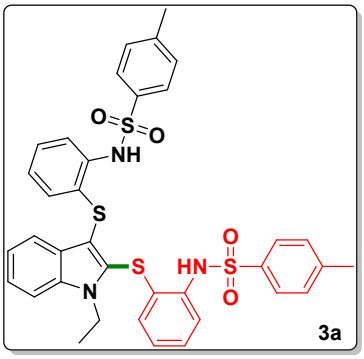
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); White solid; Yield: 226 mg, 73%; m.p.: 136-138 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, J = 8.3 Hz, 2H), 7.53 (dd, J = 8.1, 1.4 Hz, 1H), 7.43 (s, 1H), 7.23 (s, 1H), 7.20 – 7.16 (m, 2H), 7.16 – 7.08 (m, 2H), 7.10 – 7.04 (m, 1H), 6.96 (d, J = 5.5 Hz, 2H), 6.88 (td, J = 7.5, 1.4 Hz, 1H), 4.38 (q, J = 7.2 Hz, 2H), 2.72 (s, 3H), 2.39 (s, 3H), 1.46 (t, J = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.8, 136.3, 135.3, 135.2, 134.1, 130.6, 130.3, 129.5, 129.0, 127.4, 125.7, 125.3, 121.6, 121.4, 120.7, 117.4, 100.2, 43.9, 21.6, 19.7, 17.7; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{24}H_{25}N_2O_2S_2$: 437.1357; found: 437.1351.

_N_-(2-((1-Ethyl-5,6-difluoro-1_H_-indol-3-yl)thio)phenyl)-4-methylbenzenesulfonamide (2aa)



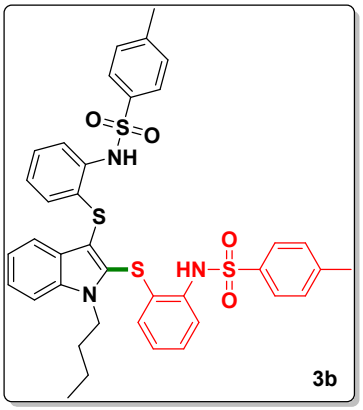
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pink liquid; Yield: 220 mg, 73%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 – 7.57 (m, 2H), 7.45 (dd, J = 8.1, 1.4 Hz, 1H), 7.30 (s, 1H), 7.11 (s, 1H), 7.06 – 7.00 (m, 2H), 6.99 – 6.93 (m, 2H), 6.92 – 6.88 (m, 2H), 6.83 (td, J = 7.4, 1.4 Hz, 1H), 4.29 (q, J = 7.2 Hz, 2H), 2.63 (s, 3H), 1.37 (t, J = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 165.3 (d, J = 254.9 Hz), 135.3, 135.2 (d, J = 3.3 Hz), 134.6, 134.0, 130.2, 130.1 (d, J = 9.5 Hz), 130.1, 129.6, 127.5, 125.8 (d, J = 2.9 Hz), 122.3, 121.5, 120.8, 117.4, 116.2, 116.0, 99.9, 43.9, 19.7, 17.7; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{23}H_{21}F_2N_2O_2S_2$: 459.1013; found: 459.1006.

_N,N'_-(((1-Ethyl-1_H_-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-methylbenzenesulfonamide) (3a)



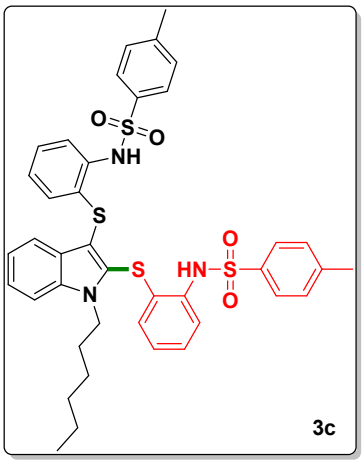
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8:2, v/v); Pale brown solid; Yield: 131 mg, 79%; m.p.: 166-168 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.70 (s, 1H), 7.63 – 7.59 (m, 2H), 7.58 – 7.52 (m, 4H), 7.49 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.33 – 7.28 (m, 2H), 7.22 – 7.13 (m, 4H), 7.09 (td, *J* = 7.7, 1.6 Hz, 1H), 7.05 – 7.00 (m, 3H), 6.87 (dd, *J* = 7.8, 1.4 Hz, 1H), 6.84 – 6.78 (m, 1H), 6.74 (dd, *J* = 7.9, 1.6 Hz, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 2.32 (s, 3H), 2.29 (s, 3H), 0.92 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.8, 143.7, 137.4, 136.5, 136.4, 136.1, 136.0, 132.1, 131.5, 131.0, 129.6, 129.5, 129.2, 128.9, 128.4, 127.9, 127.4, 127.3, 126.5, 125.9, 125.3, 124.3, 123.2, 121.9, 121.5, 119.9, 110.4, 110.3, 39.8, 21.5, 21.4, 14.8; HRMS (ESI): m/z [M + H]$^{+}$ calcd. for $C_{36}H_{34}N_3O_4S_4$: 700.1432; found: 700.1423.

_N,N'_-(((1-Butyl-1_H_-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-methylbenzenesulfonamide) (3b)



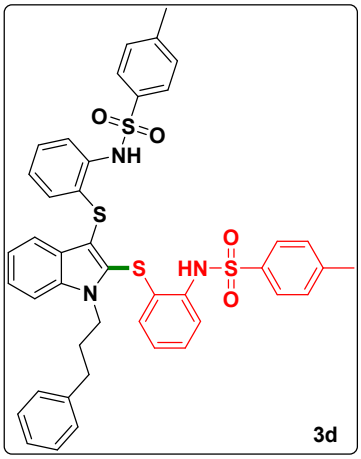
Purified by column chromatography eluting with petroleum ether/ ethyl acetate = 8.5:1.5 (v/v); Pale brown solid; Yield: 124 mg, 77%; m.p.: 185-187 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.72 (s, 1H), 7.63 (d, *J* = 8.3 Hz, 2H), 7.56 – 7.49 (m, 5H), 7.38 – 7.28 (m, 2H), 7.22 – 7.15 (m, 4H), 7.12 – 7.07 (m, 1H), 7.00 – 6.93 (m, 3H), 6.88 – 6.79 (m, 3H), 4.11 (t, *J* = 7.3 Hz, 2H), 2.32 (s, 3H), 2.28 (s, 3H), 1.66 – 1.63 (m, 2H), 1.21 – 1.18 (m, 2H), 0.79 (t, *J* = 6.5 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.8 (2C), 137.7, 136.3, 136.2, 135.9, 132.1, 131.7, 131.5, 129.6, 129.5, 129.1, 128.2, 128.1, 127.4, 127.3, 126.0, 125.6, 125.3, 124.2, 122.6, 122.2, 121.5, 119.8, 110.5, 110.0, 45.0, 31.9, 21.5, 21.5, 20.1, 13.7; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{38}H_{38}N_3O_4S_4$: 728.1745; found: 728.1728.

***N,N'*-(((1-Hexyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-methylbenzenesulfonamide) (3c)**



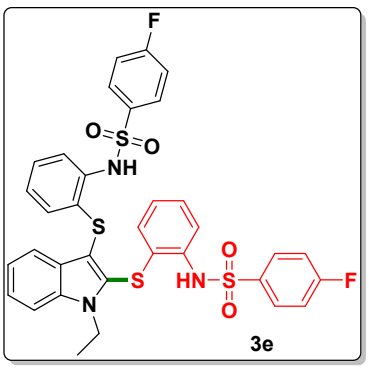
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Brown solid; Yield: 120 mg, 76%; m.p.: 178-180 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (s, 1H), 7.74 (s, 1H), 7.65 (d, *J* = 8.3 Hz, 2H), 7.60 – 7.50 (m, 5H), 7.37 – 7.29 (m, 2H), 7.25 – 7.16 (m, 4H), 7.12 (td, *J* = 7.7, 1.6 Hz, 1H), 7.00 (d, *J* = 8.1 Hz, 2H), 6.96 (dd, *J* = 7.9, 1.5 Hz, 1H), 6.91 – 6.86 (m, 2H), 6.83 (td, *J* = 7.6, 1.3 Hz, 1H), 4.13 (t, *J* = 7.1 Hz, 2H), 2.34 (s, 3H), 2.30 (s, 3H), 1.28 – 1.10 (m, 8H), 0.87 (t, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.8, 143.7, 137.7, 136.4, 136.3, 136.2, 135.8, 132.1, 131.7, 131.6, 129.6, 129.5, 129.2, 129.1, 128.3, 128.2, 127.5, 127.3, 125.7, 125.6, 125.4, 124.3, 122.4, 122.3, 121.5, 119.8, 110.5, 109.9, 45.2, 31.4, 29.8, 26.4, 22.4, 21.5, 21.5, 14.0; HRMS (ESI) (m/z) [M + H]$^+$ calcd. for $C_{40}H_{42}N_3O_4S_4$: 756.2058; found: 756.2056.

***N,N'*-(((1-(3-Phenylpropyl)-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-methylbenzenesulfonamide) (3d)**



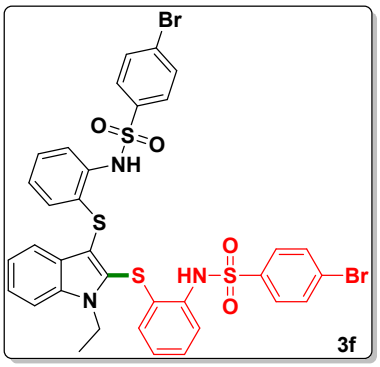
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8:2, v/v); Light yellow solid; Yield: 111 mg, 72%; m.p.: 140-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.58 (s, 1H), 7.58 – 7.52 (m, 2H), 7.53 – 7.42 (m, 3H), 7.43 – 7.36 (m, 2H), 7.24 – 7.18 (m, 2H), 7.18 – 7.10 (m, 3H), 7.13 – 7.03 (m, 4H), 7.07 – 6.99 (m, 1H), 6.95 – 6.88 (m, 2H), 6.88 (dd, *J* = 7.9, 1.5 Hz, 1H), 6.80 (dd, *J* = 7.9, 1.3 Hz, 1H), 6.80 – 6.70 (m, 4H), 4.05 (t, *J* = 7.8 Hz, 2H), 2.39 (t, *J* = 7.8 Hz, 2H), 2.22 (s, 3H), 2.11 (s, 3H), 1.45 – 1.33 (m, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.8, 143.7, 140.5, 137.7, 136.7, 136.3, 136.1, 135.8, 131.9 (2C), 131.6, 129.6, 129.4, 129.3, 129.1, 128.5, 128.3, 128.2 (3C), 127.4, 127.2, 126.3, 125.6, 125.4, 125.3, 124.3, 122.3, 121.6, 119.9, 110.4, 110.2, 44.6, 32.9, 31.1, 21.5, 21.4; HRMS (ESI) (m/z) [M + H]$^{+}$ calcd. for $C_{43}H_{40}N_3O_4S_4$: 790.1902; found: 790.1894.

***N, N'*-(((1-Ethyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-fluorobenzenesulfonamide) (3e)**



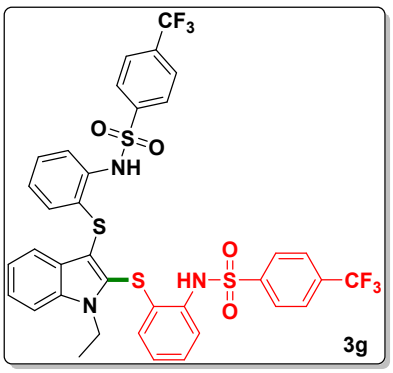
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); brown liquid; Yield: 122 mg, 74%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.80 (s, 1H), 7.77 – 7.72 (m, 2H), 7.71 – 7.66 (m, 2H), 7.60 (dt, *J* = 8.0, 1.0 Hz, 1H), 7.57 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.51 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.39 – 7.34 (m, 2H), 7.28 – 7.21 (m, 2H), 7.15 (ddd, *J* = 8.2, 7.4, 1.6 Hz, 1H), 7.10 – 7.03 (m, 3H), 6.98 – 6.87 (m, 4H), 6.82 (dd, *J* = 7.9, 1.5 Hz, 1H), 4.19 (q, *J* = 7.2 Hz, 2H), 0.96 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 165.3 (d, *J* = 253 Hz), 165.2 (d, *J* = 253 Hz), 137.4, 135.5 (d, *J* = 10 Hz), 135.3 (d, *J* = 3 Hz), 135.2 (d, *J* = 3 Hz), 132.1, 131.2, 131.0, 130.2, 130.06 (d, *J* = 10 Hz), 129.1, 129.0, 128.5 (2C), 127.0, 126.4, 125.8, 124.6, 123.8, 122.6, 121.7, 119.8, 116.4, 116.26 (d, *J* = 22 Hz), 116.09 (d, *J* = 22 Hz), 110.5, 110.1, 39.9, 14.9; HRMS (ESI) (m/z) [M + H]$^+$ calcd. for $C_{34}H_{28}F_2N_3O_4S_4$: 708.0931; found: 708.0928.

***N,N'*-(((1-Ethyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-bromobenzenesulfonamide) (3f)**



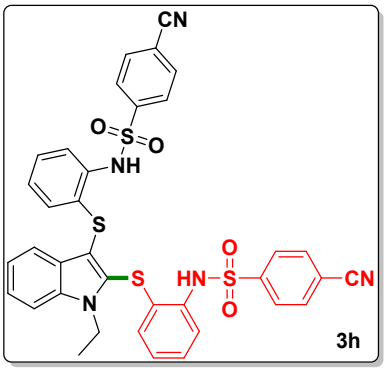
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8.5:1.5 (v/v); Brown solid; Yield: 124 mg, 73%; m.p.: 185-187 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.71 – 7.62 (m, 4H), 7.60 – 7.56 (m, 1H), 7.51 – 7.46 (m, 1H), 7.45 – 7.39 (m, 2H), 7.39 – 7.33 (m, 1H), 7.20 – 7.13 (m, 3H), 7.10 – 7.01 (m, 2H), 6.90 – 6.82 (m, 3H), 6.65 – 6.59 (m, 2H), 6.55 (dd, *J* = 7.9, 1.7 Hz, 1H), 6.18 (dd, *J* = 8.0, 1.5 Hz, 1H), 3.92 (q, *J* = 7.2 Hz, 2H), 0.84 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 138.3, 138.2, 137.5, 135.6, 135.3, 132.3, 132.1, 131.7, 131.3, 131.1, 129.3, 129.1, 128.9, 128.8, 128.7, 128.5, 128.4, 128.2, 128.0, 126.5, 126.4, 126.0, 124.6, 123.6, 122.9, 121.8, 119.8, 110.5, 39.9, 14.8; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{34}H_{28}Br_2N_3O_4S_4$: 849.9149; found: 849.9125.

***N,N'*-(((1-Ethyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-(trifluoromethyl)benzenesulfonamide) (3g)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8:2, v/v); Biege solid; Yield: 122 mg, 72%; m.p.: 152-154 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (s, 1H), 7.94 (s, 1H), 7.89 (d, *J* = 7.8 Hz, 2H), 7.73 (d, *J* = 8.1 Hz, 2H), 7.68 (d, *J* = 8.2 Hz, 2H), 7.64 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.59 – 7.52 (m, 2H), 7.40 (d, *J* = 8.6 Hz, 2H), 7.38 – 7.35 (m, 1H), 7.33 – 7.29 (m, 2H), 7.27 – 7.24 (m, 1H), 7.22 – 7.14 (m, 1H), 7.02 – 6.97 (m, 2H), 6.96 – 6.91 (m, 2H), 4.15 (q, *J* = 7.2 Hz, 2H), 0.83 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 142.8, 142.6, 137.4, 135.6, 134.7, 134.6, 134.4, 131.8, 131.3, 131.2, 129.6, 129.2, 129.1, 128.4, 128.0 (2C), 127.7 (2C), 126.4, 126.3, 126.2 (q, *J* = 3.3 Hz), 126.0, 125.9 (q, *J* = 3 Hz), 124.5 (q, *J* = 273 Hz), 124.7, 123.3, 121.9, 119.7, 110.5, 109.1, 39.9, 14.6; HRMS (ESI) (m/z) [M + H]$^+$ calcd. for $C_{36}H_{28}F_6N_3O_4S_4$: 808.0867; found: 808.0863.

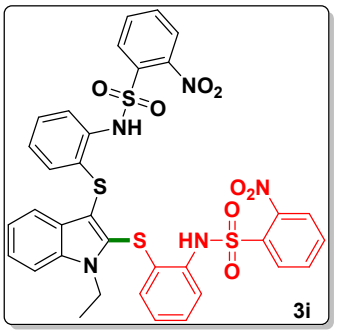
***N,N'*-(((1-Ethyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-cyanobenzenesulfonamide) (3h)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8:2, v/v); Pale pink solid; Yield: 117 mg, 75%; m.p.: 107-109 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (s, 2H), 7.85 (d, *J* = 8.8 Hz, 2H), 7.73 – 7.65 (m, 4H), 7.63 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.59 – 7.52 (m, 2H), 7.46 – 7.38 (m, 3H), 7.35 – 7.29 (m, 3H), 7.20 (ddd, *J* = 8.1, 5.8, 3.2 Hz, 1H), 7.07 – 7.00 (m, 2H), 6.99 – 6.94 (m, 2H), 4.20 (q, *J* = 7.2 Hz, 2H), 0.90 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.3, 143.1, 137.4, 135.6, 134.4, 132.8, 132.5, 132.1, 131.2, 131.1, 129.9, 129.5, 129.0, 128.5, 128.0, 127.6, 126.8, 126.7, 126.0, 125.0, 123.9, 123.4, 122.1, 119.7,

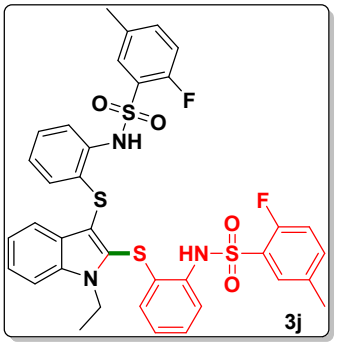
117.2, 117.0, 116.7, 116.5, 110.5, 109.0, 40.0, 14.8; HRMS (ESI) (m/z) $[M + Na]^+$ calcd. for $C_{36}H_{27}N_5NaO_4S_4$: 744.0844; found: 744.0853.

***N,N′*-(((1-Ethyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(2-nitrobenzenesulfonamide) (3i)**



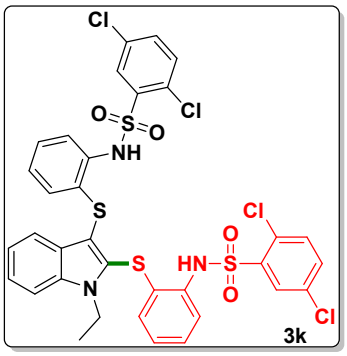
Purified by column chromatography (eluent: petroleum ether/EtOAc = 7.5:2.5, v/v); Light yellow solid; Yield: 114 mg, 68%; m.p.: 128-130 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (s, 1H), 7.97 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.95 (s, 1H), 7.93 (dd, *J* = 3.2, 1.4 Hz, 1H), 7.91 (dd, *J* = 3.4, 1.5 Hz, 1H), 7.87 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.76 (td, *J* = 7.7, 1.6 Hz, 1H), 7.70 (tdd, *J* = 7.6, 3.6, 1.5 Hz, 2H), 7.65 (td, *J* = 7.6, 1.4 Hz, 1H), 7.47 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.43 (dd, *J* = 8.1, 1.2 Hz, 2H), 7.37 (dt, *J* = 8.3, 1.1 Hz, 1H), 7.34 – 7.30 (m, 1H), 7.18 – 7.10 (m, 3H), 6.92– 6.87 (m, 2H), 6.84 (dd, *J* = 7.9, 1.5 Hz, 1H), 6.46 (dd, *J* = 8.0, 1.4 Hz, 1H), 4.20 (q, *J* = 7.2 Hz, 2H), 1.13 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 147.9, 137.3, 134.3, 134.0, 133.9, 133.6, 133.4, 133.3 (2C), 133.0, 132.8, 132.0, 131.6, 131.1, 131.0, 130.9, 130.6, 129.4, 129.1, 127.8, 127.6, 127.4, 126.8, 126.3, 125.7, 125.6, 125.0, 124.3, 121.4, 119.9, 111.1, 110.5, 39.9, 15.1; HRMS (ESI) (m/z) $[M + Na]^+$ calcd. for $C_{34}H_{27}N_5NaO_8S_4$: 784.0640; found: 784.0637.

***N,N′*-(((1-Ethyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(2-fluoro-5-methylbenzenesulfonamide) (3j)**



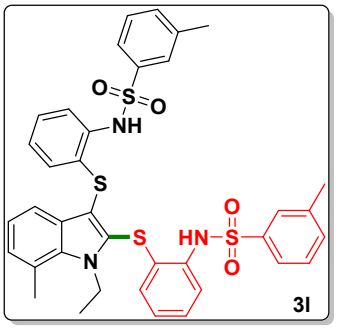
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pale brown solid; Yield: 117 mg, 70%; m.p.: 144-146 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (s, 1H), 7.75 (s, 1H), 7.63 – 7.56 (m, 2H), 7.51 (dt, *J* = 8.0, 1.0 Hz, 1H), 7.34 – 7.28 (m, 2H), 7.29 – 7.17 (m, 4H), 7.15 – 7.06 (m, 1H), 7.02 (dd, *J* = 7.4, 1.5 Hz, 1H), 7.00 – 6.95 (m, 3H), 6.95 – 6.90 (m, 1H), 6.78 – 6.69 (m, 2H), 6.53 (dd, *J* = 8.0, 1.5 Hz, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 2.25 (s, 3H), 2.24 (s, 3H), 1.04 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.5 (d, *J* = 253 Hz), 156.0 (d, *J* = 253 Hz), 137.3, 135.9 (d, *J* = 2.8 Hz), 135.8 (d, *J* = 2.8 Hz), 135.6, 134.5, 134.3 (d, *J* = 3.9 Hz), 134.2 (d, *J* = 4.0 Hz), 132.3, 131.7, 130.6, 130.5, 130.0, 129.2, 128.4, 128.2, 128.1, 127.4, 127.3, 127.0, 126.9, 126.4, 125.3, 124.3, 123.7, 121.3 (d, *J* = 6.3 Hz), 120.0, 117.0 (d, *J* = 5.1 Hz), 116.8 (d, *J* = 4.9 Hz), 111.4, 110.5, 39.9, 20.6 (2C), 15.1; HRMS (ESI) (m/z) $[M + H]^+$ calcd. for $C_{36}H_{32}F_2N_3O_4S_4$: 736.1244; found: 736.1245.

***N,N'*-(((1-Ethyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(2,5-dichlorobenzenesulfonamide) (3k)**



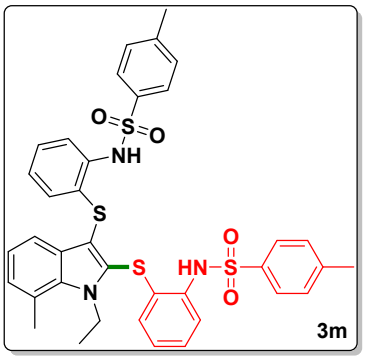
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8:2, v/v); Biege solid; Yield: 118 mg, 70%; m.p.: 181-183 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (s, 1H), 8.09 (s, 1H), 8.07 (ddd, *J* = 2.9, 2.1, 0.7 Hz, 2H), 7.69 (dt, *J* = 7.9, 1.0 Hz, 1H), 7.51 – 7.45 (m, 2H), 7.43 – 7.41 (m, 2H), 7.41 – 7.39 (m, 1H), 7.38 – 7.29 (m, 3H), 7.25 (ddd, *J* = 8.0, 6.8, 1.2 Hz, 1H), 7.15 – 7.04 (m, 3H), 6.89 – 6.80 (m, 2H), 6.53 (dd, *J* = 8.0, 1.4 Hz, 1H), 4.26 (q, *J* = 7.2 Hz, 2H), 1.20 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 139.2, 138.4, 137.4, 135.2, 134.0, 133.9, 133.4, 133.2, 133.1, 133.0 (2C), 132.8, 131.5, 131.3, 131.1, 130.6, 130.6, 130.3, 129.3 (2C), 128.5, 128.1, 128.0, 127.2, 125.6, 125.1, 124.4, 121.5, 121.1, 120.0, 111.7, 110.6, 40.0, 15.3; HRMS (ESI) (m/z) $[M + H]^+$ calcd. for $C_{34}H_{26}Cl_2N_3O_4S_4$: 807.9560; found: 807.9544.

***N,N'*-(((1-Ethyl-7-methyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(3-methylbenzenesulfonamide) (3l)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8:2, v/v); Pale brown solid; Yield: 121 mg, 74%; m.p.: 170-172 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.75 (s, 1H), 7.65 (s, 1H), 7.60 (s, 1H), 7.60 – 7.43 (m, 5H), 7.36 – 7.29 (m, 2H), 7.28 – 7.20 (m, 2H), 7.21 – 7.12 (m, 1H), 7.15 – 7.05 (m, 3H), 7.04 (dd, *J* = 7.9, 1.5 Hz, 1H), 6.85 (dtd, *J* = 21.5, 7.7, 1.4 Hz, 2H), 6.63 (dd, *J* = 8.0, 1.5 Hz, 1H), 4.41 (q, *J* = 7.1 Hz, 2H), 2.72 (s, 3H), 2.31 (s, 3H), 2.30 (s, 3H), 1.07 (t, *J* = 7.0 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 139.6, 139.2, 139.1, 139.0, 136.5, 136.1, 134.9, 133.8, 133.7, 132.3, 131.8, 130.3, 129.6, 128.8 (2C), 128.6, 128.4, 128.3, 127.9, 127.7, 127.6, 127.5, 126.4, 125.3, 124.5, 124.4, 124.3, 122.0, 121.6, 121.5, 118.0, 111.9, 41.4, 21.3 (2C), 20.0, 17.5; HRMS (ESI) (m/z) $[M + H]^+$ calcd. for $C_{37}H_{36}N_3O_4S_4$: 714.1589; found: 714.1598.

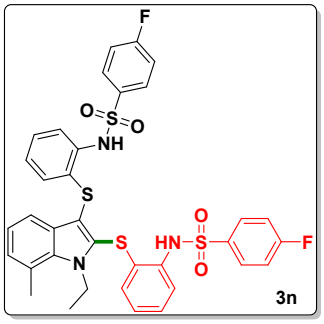
***N,N'*-(((1-Ethyl-7-methyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-methylbenzenesulfonamide) (3m)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pale brown solid; Yield: 117 mg, 72%; m.p.: 166-169 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.69 (s, 1H), 7.69 – 7.58 (m, 4H), 7.55 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.49 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.48 – 7.43 (m, 1H), 7.22 – 7.14 (m, 3H), 7.14 – 7.04 (m, 5H), 7.01 (dd, *J* = 7.9, 1.5 Hz, 1H), 6.88 (td, *J* = 7.6, 1.4 Hz, 1H), 6.82 (td, *J* = 7.6, 1.4 Hz, 1H), 6.70 (dd, *J* = 7.9, 1.5 Hz, 1H), 4.39 (q, *J* = 7.1 Hz, 2H), 2.72 (s, 3H), 2.35 (s, 3H), 2.33 (s, 3H), 0.99 (t, *J* = 7.1 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.8, 143.7, 136.6, 136.5, 136.3, 136.1, 135.5, 132.1, 131.9, 130.3, 130.2, 129.6, 129.5, 128.6, 128.3, 127.8, 127.6, 127.5, 127.4, 127.3, 126.1, 125.2,

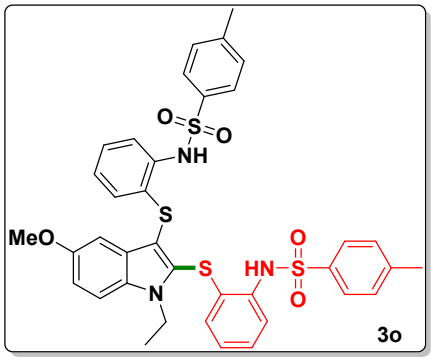
123.6, 121.9, 121.5, 121.4, 118.0, 111.6, 41.4, 21.5, 21.5, 19.9, 17.4; HRMS (ESI) (m/z) [M + H]$^+$ calcd. for $C_{37}H_{36}N_3O_4S_4$: 714.1589; found: 714.1588.

***N,N'*-(((1-Ethyl-7-methyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-fluorobenzenesulfonamide) (3n)**



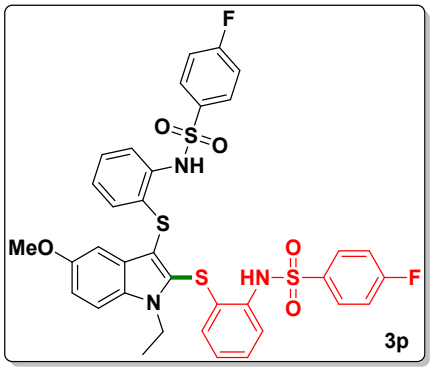
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pale brown solid; Yield: 120 mg, 73%; m.p.: 91-93 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 1H), 7.79 – 7.66 (m, 5H), 7.56 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.53 – 7.43 (m, 2H), 7.26 – 7.17 (m, 1H), 7.17 – 7.09 (m, 3H), 7.08 – 7.02 (m, 3H), 7.01 – 6.90 (m, 3H), 6.88 (td, *J* = 7.6, 1.4 Hz, 1H), 6.73 (dd, *J* = 7.9, 1.5 Hz, 1H), 4.39 (q, *J* = 7.1 Hz, 2H), 2.72 (s, 3H), 1.01 (t, *J* = 7.1 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 165.3 (d, *J* = 254.9 Hz), 165.2 (d, *J* = 253.6 Hz), 136.6, 135.7, 135.5 (d, *J* = 3.0 Hz), 135.2 (d, *J* = 3.0 Hz), 134.9, 132.2, 131.5, 130.2, 130.1 (2C),130.0, 128.7, 128.5, 128.2, 128.1, 127.8, 126.6, 125.8, 124.4, 122.5, 121.7, 121.6, 118.0, 116.3 (d, *J* = 12.5 Hz), 116.1 (d, *J* = 12.5 Hz), 111.4, 41.4, 19.9, 17.4; HRMS (ESI) (m/z) [M + Na]$^+$ calcd. for $C_{35}H_{30}F_2N_3NaO_4S_4$: 744.0907; found: 744.0913.

***N,N'*-(((1-Ethyl-5-methoxy-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-methylbenzenesulfonamide) (3o)**



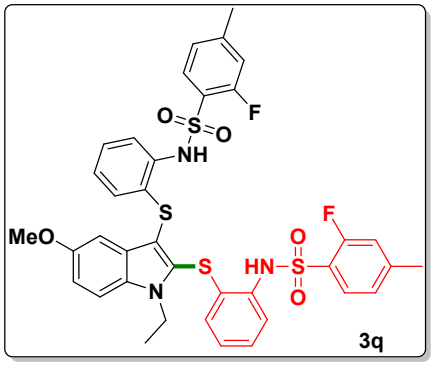
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8:2, v/v); Red liquid; Yield: 121 mg, 75%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (s, 1H), 7.71 (s, 1H), 7.62 (d, *J* = 8.3 Hz, 2H), 7.61 – 7.54 (m, 3H), 7.50 (dd, *J* = 8.1, 1.4 Hz, 1H), 7.22 (d, *J* = 8.9 Hz, 1H), 7.19 – 7.15 (m, 3H), 7.13 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.09 (dd, *J* = 6.6, 1.9 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 7.00 (dd, *J* = 9.0, 2.5 Hz, 1H), 6.90 – 6.79 (m, 2H), 6.74 (dd, *J* = 7.9, 1.5 Hz, 1H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.89 (s, 3H), 2.32 (s, 6H), 0.89 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 155.6, 143.8, 143.7, 136.4, 136.3, 135.9, 132.5, 132.4, 131.1, 131.0, 129.8, 129.7, 129.6, 129.5, 128.9, 128.5, 127.6, 127.4, 127.3, 126.4, 125.8, 125.1, 123.0, 121.4, 115.3, 111.3, 109.6, 100.5, 55.8, 39.9, 21.5 (2C), 14.9; HRMS (ESI) (m/z) [M + H]$^+$ calcd. for $C_{37}H_{36}N_3O_5S_4$: 730.1538; found: 730.1532.

***N,N'*-(((1-Ethyl-5-methoxy-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-fluorobenzenesulfonamide) (3p)**



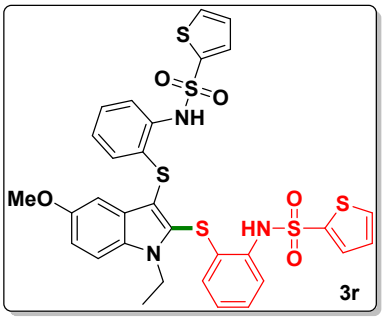
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pale brown solid; Yield: 119 mg, 74%; m.p.: 133-135 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.82 (s, 1H), 7.80 – 7.71 (m, 2H), 7.72 – 7.63 (m, 2H), 7.56 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.50 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.27 – 7.18 (m, 2H), 7.19 – 7.11 (m, 1H), 7.10 – 6.98 (m, 5H), 6.98 – 6.86 (m, 4H), 6.82 (dd, *J* = 7.9, 1.5 Hz, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.89 (s, 3H), 0.93 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 166.5 (d, *J* = 253 Hz), 164.0 (d, *J* = 253 Hz), 155.7, 135.6 (d, *J* = 5.9 Hz), 135.3 (d, *J* = 3.2 Hz), 135.2, 132.5, 131.9, 131.2, 130.9, 130.2, 130.1, 130.0, 129.8, 129.1, 128.7, 128.4, 126.7, 126.3, 125.8, 123.5, 122.6, 116.2 (d, *J* = 22.6 Hz), 116.1(d, *J* = 22.6 Hz), 115.6, 111.5, 109.0, 100.3, 55.8, 40.0, 15.0; HRMS (ESI) (m/z) [M + H]$^+$ calcd. for $C_{35}H_{30}F_2N_3O_5S_4$: 738.1036; found: 738.1038.

***N,N'*-(((1-Ethyl-5-methoxy-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(2-fluoro-4-methylbenzenesulfonamide) (3q)**



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8:2, v/v); Pale brown solid; Yield: 110 mg, 68%; m.p.: 88-90 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 7.83 (s, 1H), 7.78 – 7.71 (m, 2H), 7.44 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.40 (dd, *J* = 8.2, 1.3 Hz, 1H), 7.25 (d, *J* = 9.2 Hz, 1H), 7.15 – 7.04 (m, 4H), 7.01 – 6.98 (m, 3H), 6.97 – 6.92 (m, 2H), 6.85 – 6.79 (m, 2H), 6.65 (dd, *J* = 7.9, 1.5 Hz, 1H), 4.20 (q, *J* = 7.2 Hz, 2H), 3.87 (s, 3H), 2.38 (s, 3H), 2.36 (s, 3H), 1.09 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.9 (d, *J* = 254 Hz), 158.8 (d, *J* = 254 Hz), 155.4, 147.3, 147.2 (2C), 147.1, 135.9, 134.8, 132.6, 132.4, 131.3, 130.5, 130.4, 130.3, 129.8, 128.3 (d, *J* = 2.9 Hz), 127.6 (d, *J* = 2.9 Hz), 126.1, 125.0 (d, *J* = 3.5 Hz), 124.9 (d, *J* = 3.0 Hz), 124.5, 123.0, 120.7, 117.8, 117.7, 117.5 (2C), 115.3, 111.4, 110.4, 100.6, 55.8, 40.0, 21.5 (2C), 15.1; HRMS (ESI) (m/z) $[M + H]^+$ calcd. for $C_{37}H_{34}F_2N_3O_5S_4$: 766.1349; found: 766.1345.

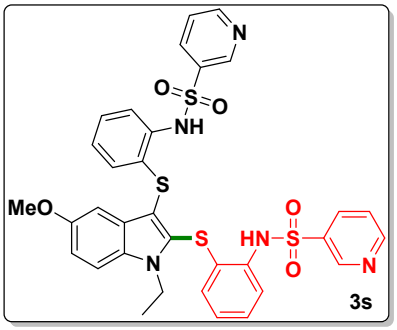
_N_-(2-((1-Ethyl-5-methoxy-2-((2-(thiophene-2-sulfonamido)phenyl)thio)-1_H_-indol-3-yl)thio)phenyl)thiophene-2-sulfonamide (3r)



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Pale brown solid; Yield: 106 mg, 66%; m.p.: 158-160 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.78 (s, 1H), 7.60 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.56 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.55 – 7.48 (m, 2H), 7.48 (dd, *J* = 3.8, 1.3 Hz, 1H), 7.44 (dd, *J* = 3.8, 1.4 Hz, 1H), 7.25 – 7.22 (m, 2H), 7.17 (td, *J* = 7.7, 1.6 Hz, 1H), 7.13 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.09 (d, *J* = 2.4 Hz, 1H), 7.02 – 6.97 (m, 2H), 6.95 – 6.90 (m, 3H), 6.74 (dd, *J* = 7.9, 1.5 Hz, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 3.88 (s, 3H), 1.02 (t, *J* = 7.1 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 155.6, 139.7, 139.6, 135.7,

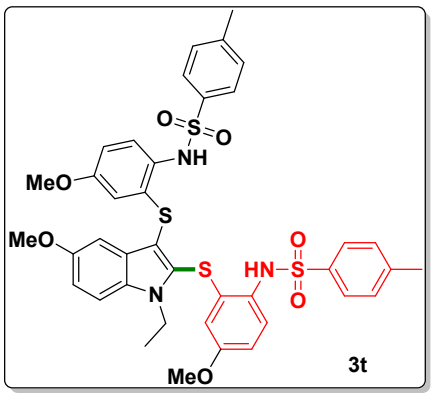
135.2, 132.9 (2C), 132.6 (2C), 132.5, 132.4, 131.1, 130.8, 129.8, 128.8, 128.7, 128.5, 127.6, 127.4 (2C), 126.5, 125.8, 123.9, 122.4, 115.4, 111.4, 109.7, 100.5, 55.8, 40.0, 15.1; HRMS (ESI) (m/z) $[M + H]^+$ calcd. for $C_{31}H_{28}N_3O_5S_6$: 714.0353; found: 714.0349

_N,N'_-(((1-ethyl-5-methoxy-1_H_-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(pyridine-3-sulfonamide) (3s)



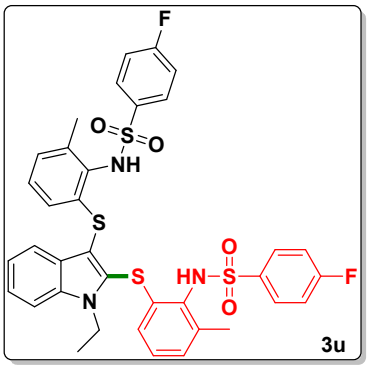
Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Biege solid; Yield: 103 mg, 64%; m.p.: 175-177 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.11 (s, 1H), 8.94 (ddd, J = 5.1, 2.3, 0.8 Hz, 2H), 8.77 (dd, J = 4.9, 1.6 Hz, 1H), 8.70 (dd, J = 4.8, 1.6 Hz, 1H), 8.03 – 7.97 (m, 2H), 7.94 (s, 1H), 7.45 (dt, J = 8.1, 1.6 Hz, 2H), 7.31 (ddd, J = 8.1, 4.9, 0.8 Hz, 1H), 7.26 – 7.22 (m, 1H), 7.21 (d, J = 9.2 Hz, 1H), 7.09 (ddd, J = 8.0, 7.4, 1.5 Hz, 1H), 7.01 (ddd, J = 8.1, 7.4, 1.5 Hz, 1H), 6.94 (dd, J = 9.0, 2.5 Hz, 1H), 6.90 – 6.84 (m, 2H), 6.82 – 6.76 (m, 1H), 6.63 (dd, J = 7.9, 1.5 Hz, 1H), 6.36 (dd, J = 8.0, 1.4 Hz, 1H), 3.99 (q, J = 7.2 Hz, 2H), 3.76 (s, 3H), 0.99 (t, J = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 155.7, 153.2, 153.0, 148.0, 147.8, 136.9, 136.3, 135.7, 135.1, 133.0, 132.9, 132.6, 130.9, 130.8, 130.4, 130.1, 129.0, 128.2, 127.9, 127.5, 127.2, 126.6, 126.2, 123.8, 123.8, 123.6, 115.8, 111.7, 108.0, 100.4, 55.8, 40.1, 15.4; HRMS (ESI) (m/z) $[M + H]^+$ calcd. for $C_{33}H_{30}N_5O_5S_4$: 704.1130; found: 704.1128.

_N,N'_-(((1-Ethyl-5-methoxy-1_H_-indole-2,3-diyl)bis(sulfanediyl))bis(4-methoxy-2,1-phenylene))bis(4-methylbenzenesulfonamide) (3t)



Purified by column chromatography (eluent: petroleum ether/EtOAc = 8.5:1.5, v/v); Brown liquid; Yield: 118 mg, 73%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (dd, *J* = 8.4, 2.6 Hz, 4H), 7.55 (s, 1H), 7.36 – 7.31 (m, 3H), 7.26 – 7.13 (m, 5H), 7.03 – 6.96 (m, 2H), 6.68 – 6.58 (m, 2H), 6.46 (d, *J* = 2.9 Hz, 1H), 6.09 (d, *J* = 2.8 Hz, 1H), 4.14 (q, *J* = 7.1 Hz, 2H), 3.87 (s, 3H), 3.57 (s, 3H), 3.54 (s, 3H), 2.37 (s, 3H), 2.35 (s, 3H), 1.06 (t, *J* = 7.1 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.1, 157.3, 155.5, 143.7, 143.6, 136.7, 136.4, 132.5, 131.9, 131.7, 131.4, 130.0, 129.5, 129.5, 128.3, 127.5 (3C), 127.4, 127.3, 125.5, 116.6, 115.2, 115.1, 113.0, 112.9, 111.4, 109.6, 100.7, 55.8, 55.3, 55.2, 39.9, 21.6 (2C), 15.3; HRMS (ESI) (m/z) $[M + H]^+$ calcd. for $C_{39}H_{40}N_3O_7S_4$: 790.1749; found: 790.1748.

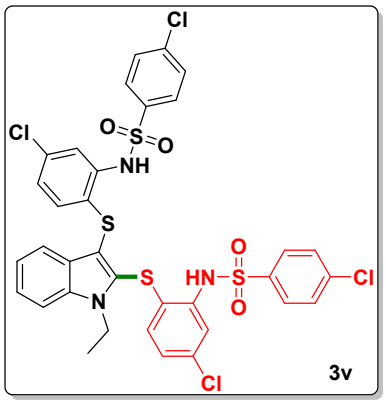
_N,N′_-(((1-Ethyl-1_H_-indole-2,3-diyl)bis(sulfanediyl))bis(6-methyl-2,1-phenylene))bis(4-fluorobenzenesulfonamide) (3u)



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8.5:1.5 (v/v); Pale brown solid; Yield: 118 mg, 71%; m.p.: 153-155 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.59 (s, 1H), 7.45 – 7.37 (m, 2H), 7.36 – 7.31 (m, 1H), 7.16 – 7.10 (m, 2H), 7.09 – 6.99 (m, 4H), 6.95 – 6.89 (m, 1H), 6.86 – 6.71 (m, 5H), 6.59 – 6.52 (m, 2H), 6.34 (dd, *J* = 7.9, 1.5 Hz, 1H), 3.97 (q, *J* = 7.2 Hz, 2H), 2.07 (s, 3H), 2.06 (s, 3H), 0.86 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 157.3 (d, *J* = 253 Hz), 157.2 (d, *J* = 252 Hz), 137.4, 135.9 (d, *J* = 10.0 Hz), 135.6, 134.5, 134.4 (d, *J* = 4.0 Hz), 134.3 (d, *J* = 4.0 Hz), 132.3, 131.8, 130.6 (d, *J* = 11.0 Hz), 130.0, 129.2, 128.4, 128.2, 128.1, 127.3 (d, *J* = 13.6 Hz), 126.9 (d, *J* = 13.2 Hz),

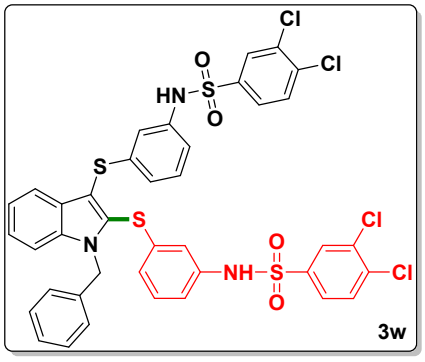
126.4, 125.3, 124.3, 123.7, 121.4 (d, J = 2.9 Hz), 120.0, 116.9 (d, J = 21 Hz), 116.8 (d, J = 21 Hz), 111.4, 110.5, 39.9, 20.6 (2C), 15.1; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{36}H_{32}F_2N_3O_4S_4$: 736.1244; found: 736.1241.

_N,N'_-(((1-Ethyl-1_H_-indole-2,3-diyl)bis(sulfanediyl))bis(4-chloro-2,1-phenylene))bis(4-chlorobenzenesulfonamide) (3v)



Purified by column chromatography eluting with petroleum ether/EtOAc = 8.5:1.5 (v/v); Brown solid; Yield: 124 mg, 76%; m.p.: 153-155 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.91 (m, 2H), 7.60 – 7.56 (m, 1H), 7.46 – 7.40 (m, 5H), 7.35 (s, 1H), 7.21 – 7.17 (m, 1H), 7.11 (s, 1H), 7.02 (d, J = 3.7 Hz, 1H), 6.92 (d, J = 3.9 Hz, 6H), 6.90 – 6.86 (m, 2H), 4.31 (q, J = 7.1 Hz, 2H), 1.10 (t, J = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 137.6, 137.4, 137.3, 136.7, 134.1 (2C), 133.7, 133.6 (2C), 133.2, 132.7, 132.6, 132.3, 131.7, 131.6, 129.5 (2C), 129.3, 129.2, 128.9, 127.6, 124.3, 123.0 (2C), 121.2, 120.3, 110.8, 110.5, 39.8, 15.0; HRMS (ESI): m/z [M + H]$^+$ calcd. for $C_{34}H_{26}Cl_4N_3O_4S_4$: 807.9560; found: 807.9568.

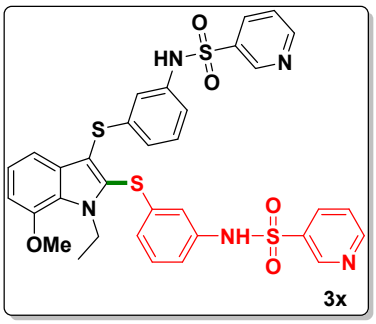
_N,N'_-(((1-Benzyl-1_H_-indole-2,3-diyl)bis(sulfanediyl))bis(3,1-phenylene))bis(3,4-dichlorobenzenesulfonamide) (3w)



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8.5:1.5 (v/v); Pale brown solid; Yield: 122 mg, 76%; m.p.: 185-187 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.52 (s, 1H), 8.11 (s, 1H), 8.09 – 8.05 (m, 1H), 8.02 (d, J = 2.6 Hz, 1H), 7.68 – 7.64 (m, 1H), 7.43 (s, 2H), 7.34 – 7.30 (m, 3H), 7.29 (d, J = 2.7 Hz, 2H), 7.25 – 7.21 (m, 4H), 7.10 – 6.99 (m,

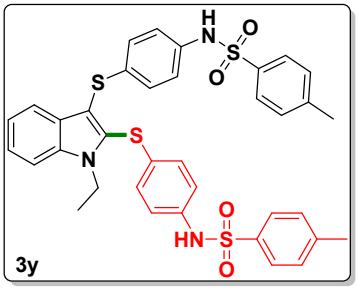
4H), 6.93 – 6.89 (m, 2H), 6.87 – 6.82 (m, 1H), 6.75 – 6.69 (m, 1H), 6.47 – 6.42 (m, 1H), 5.44 (s, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 138.8, 138.5, 138.1, 136.3, 134.9, 133.9, 133.8, 133.5, 133.2, 133.1, 132.9, 132.9, 132.6, 132.3, 131.3, 131.1, 130.5, 130.4, 129.8, 129.3, 129.2, 128.7, 128.4, 128.3, 128.1, 127.6, 126.9, 126.3, 125.7, 124.8, 124.7, 121.8, 121.4, 119.9, 112.2, 111.2, 48.4; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{39}H_{28}Cl_4N_3O_4S_4$: 869.9717; found: 869.9723.

_N,N'_-(((1-Ethyl-7-methoxy-1_H_-indole-2,3-diyl)bis(sulfanediyl))bis(3,1-phenylene))bis(pyridine-3-sulfonamide) (3x)



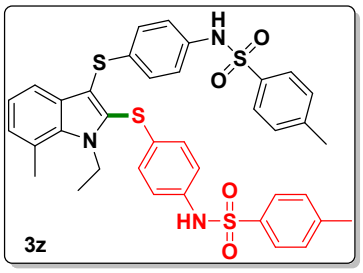
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7.5:2.5 (v/v); Brown solid; Yield: 97 mg, 61%; m.p.: 153-155 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.27 (s, 1H), 9.04 (d, J = 7.1 Hz, 2H), 8.82 (dd, J = 19.7, 4.2 Hz, 2H), 8.16 (d, J = 8.2 Hz, 2H), 8.08 (s, 1H), 7.52 – 7.43 (m, 3H), 7.41 – 7.37 (m, 1H), 7.28 (d, J = 8.9 Hz, 1H), 7.17 – 7.12 (m, 1H), 7.11 – 7.06 (m, 1H), 7.01 (dd, J = 9.0, 2.5 Hz, 1H), 6.97 – 6.92 (m, 1H), 6.91 – 6.85 (m, 2H), 6.70 (dd, J = 7.9, 1.6 Hz, 1H), 6.46 – 6.41 (m, 1H), 4.09 (q, J = 7.2 Hz, 2H), 3.82 (s, 3H), 1.08 (t, J = 7.2 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 155.7, 152.3, 151.7, 147.3, 146.7, 137.3, 136.9, 136.9, 135.9, 132.8, 132.6, 132.7, 131.5, 131.2, 130.6, 130.0, 128.9, 128.2, 127.9, 127.7, 127.2, 126.9, 126.4, 124.4, 124.3, 124.0, 115.8, 111.8, 107.8, 100.5, 55.9, 40.1, 15.4; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{33}H_{30}N_5O_5S_4$: 704.1130; found: 704.1124.

_N,N'_-(((1-Ethyl-1_H_-indole-2,3-diyl)bis(sulfanediyl))bis(4,1-phenylene))bis(4-methylbenzenesulfonamide) (3y)



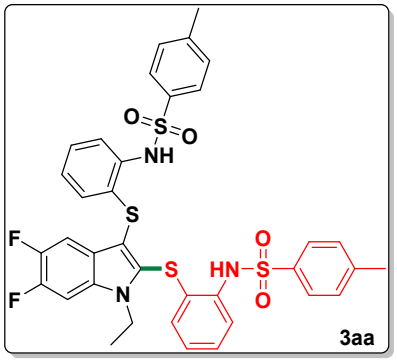
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8.5:1.5 (v/v); Brown liquid; Yield: 130 mg, 81%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (s, 1H), 7.62 (dd, *J* = 8.3, 1.3 Hz, 4H), 7.45 – 7.41 (m, 1H), 7.40 – 7.32 (m, 1H), 7.28 – 7.16 (m, 5H), 6.95 (s, 1H), 6.86 (dd, *J* = 8.9, 2.5 Hz, 4H), 6.80 (d, *J* = 8.8 Hz, 2H), 6.78 – 6.71 (m, 3H), 4.37 (q, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 2.38 (s, 3H), 1.21 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.9, 143.8, 137.2, 136.1, 136.0, 135.4, 135.2, 133.8, 133.7, 132.1, 129.7 (2C), 129.5, 129.0, 127.9, 127.3 (2C), 124.1, 122.8, 122.5, 121.1, 120.3, 111.0, 110.4, 39.7, 21.5 (2C), 15.2; HRMS (ESI): m/z [M + H]$^{+}$ calcd. for $C_{36}H_{34}N_3O_4S_4$: 700.1432; found: 700.1422.

***N,N′*-(((1-Ethyl-7-methyl-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(4,1-phenylene))bis(4-methylbenzenesulfonamide) (3z)**



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8:2 (v/v); Pale brown solid; Yield: 124 mg, 76%; m.p.: 153-155 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (s, 1H), 7.59 (s, 1H), 7.56 – 7.50 (m, 3H), 7.45 (d, *J* = 8.6 Hz, 1H), 7.40 (d, *J* = 8.1 Hz, 1H), 7.37 – 7.32 (m, 1H), 7.19 (d, *J* = 1.2 Hz, 1H), 7.11 – 7.05 (m, 3H), 7.04 – 6.95 (m, 5H), 6.91 (d, *J* = 7.9 Hz, 1H), 6.81 – 6.75 (m, 1H), 6.75 – 6.69 (m, 1H), 6.60 (d, *J* = 7.9 Hz, 1H), 4.30 (q, *J* = 7.1 Hz, 2H), 2.62 (s, 3H), 2.25 (s, 3H), 2.24 (s, 3H), 0.90 (t, *J* = 7.1 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 143.9, 143.8, 136.7, 136.6, 136.4, 136.2, 135.6, 132.2, 132.0, 130.4, 130.3, 129.7, 129.6, 128.7, 128.4, 127.9, 127.7, 127.6, 127.5, 127.5, 126.2, 125.4, 123.8, 122.0, 121.7, 121.6, 118.2, 111.7, 41.5, 21.7, 21.6, 20.0, 17.5; HRMS (ESI): m/z [M + H]$^{+}$ calcd. for $C_{37}H_{36}N_3O_4S_4$: 714.1589; found: 714.1561.

***N,N′*-(((1-Ethyl-5,6-difluoro-1*H*-indole-2,3-diyl)bis(sulfanediyl))bis(2,1-phenylene))bis(4-methylbenzenesulfonamide) (3aa)**



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7.5:2.5 (v/v); Brown solid; Yield: 102 mg, 64%; m.p.: 153-155 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (t, *J* = 7.8 Hz, 2H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.37 (d, *J* = 8.4 Hz, 1H), 7.34 – 7.29 (m, 1H), 7.17 – 7.12 (m, 1H), 6.99 – 6.92 (m, 4H), 6.91 – 6.86 (m, 6H), 6.84 (d, *J* = 8.5 Hz, 3H), 6.74 (s, 1H), 4.27 (q, *J* = 7.2 Hz, 2H), 2.37 (s, 3H), 2.36 (s, 3H), 1.07 (t, *J* = 7.2 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 158.5 (d, *J* = 252.2 Hz), 158.0 (d, *J* = 252.2 Hz), 147.4, 147.3 (d, *J* = 4.6 Hz), 147.2, 137.3, 135.7, 134.6, 133.3, 133.2, 132.6, 130.7 (d, *J* = 3.7 Hz), 129.2, 128.9 (2C), 127.7 (2C), 125.2, 124.1, 123.7 (d, *J* = 13 Hz), 123.5 (d, *J* = 14 Hz), 122.4, 122.2, 121.1, 120.3, 117.3 (d, *J* = 20 Hz), 110.9, 110.4, 39.7, 21.6 (2C), 15.0; HRMS (ESI): m/z $[M + H]^+$ calcd. for $C_{36}H_{32}F_2N_3NaO_4S_4$: 758.1063; found: 758.1093.

References:

1) S. S. Marupalli, M. Arockiaraj, G. Singh and V. Rajeshkumar, *J. Org. Chem.,* 2023, **88**, 12783–12791.
2) G. Singh, S. S. Marupalli, M. Arockiaraj and V. Rajeshkumar, *J. Org. Chem.,* 2024, **89**, 5861–5870.

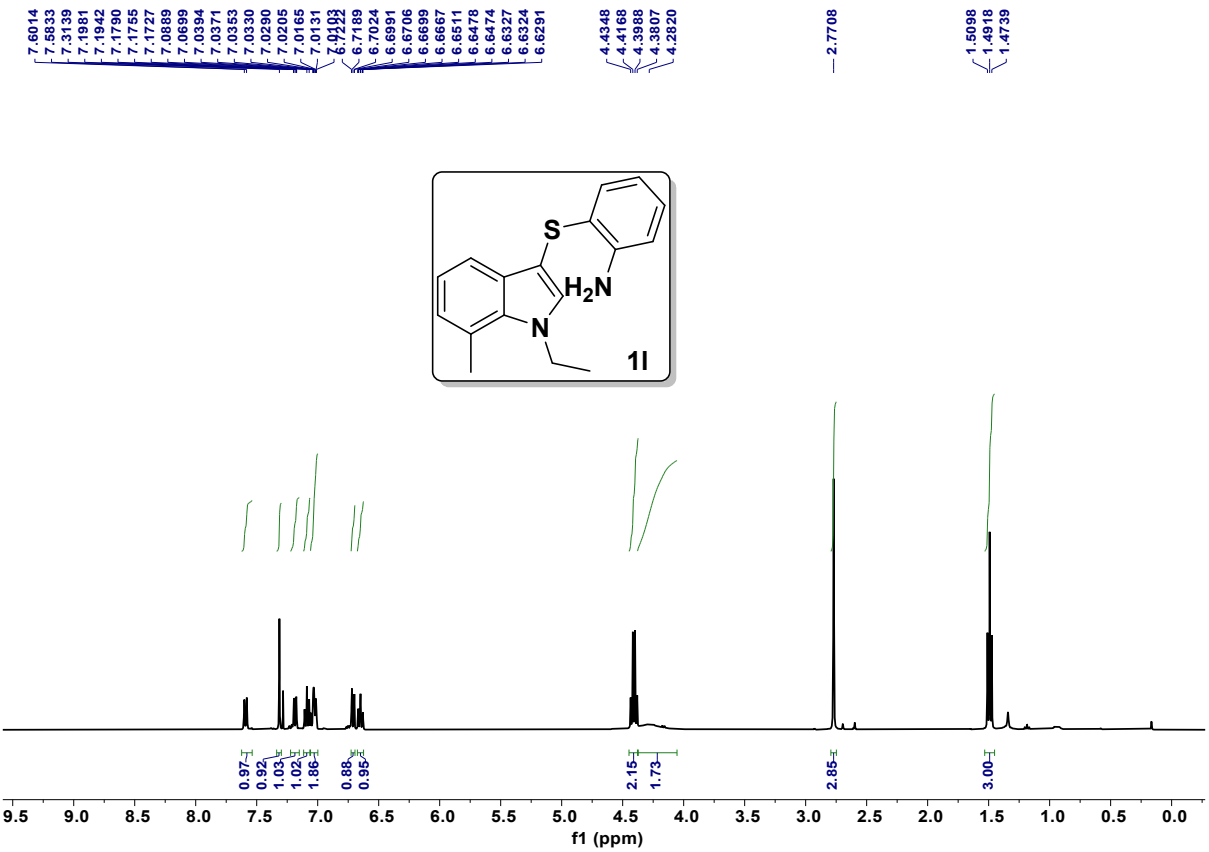


Figure S7. ^{1}H NMR of compound **1l** (400 MHz, $CDCl_3$)

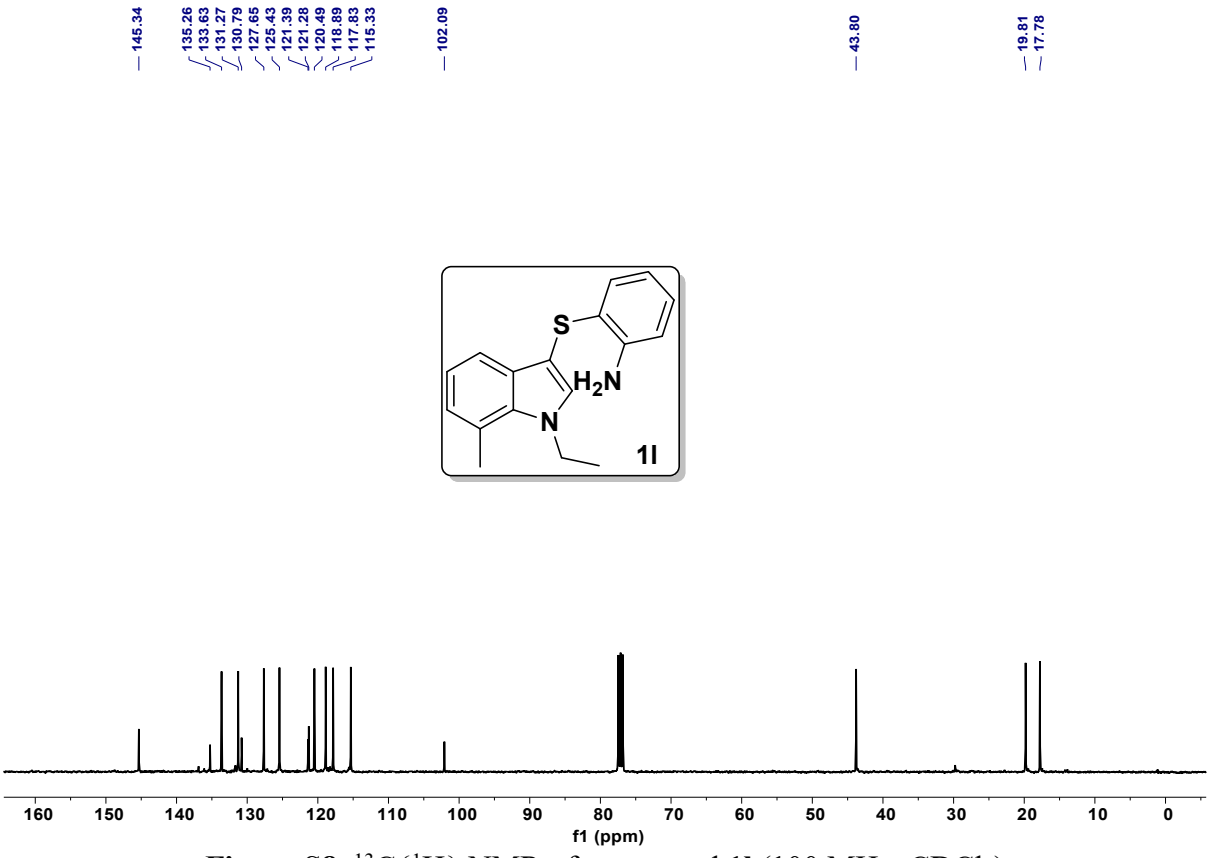


Figure S8. $^{13}C\{^{1}H\}$ NMR of compound **1l** (100 MHz, $CDCl_3$)

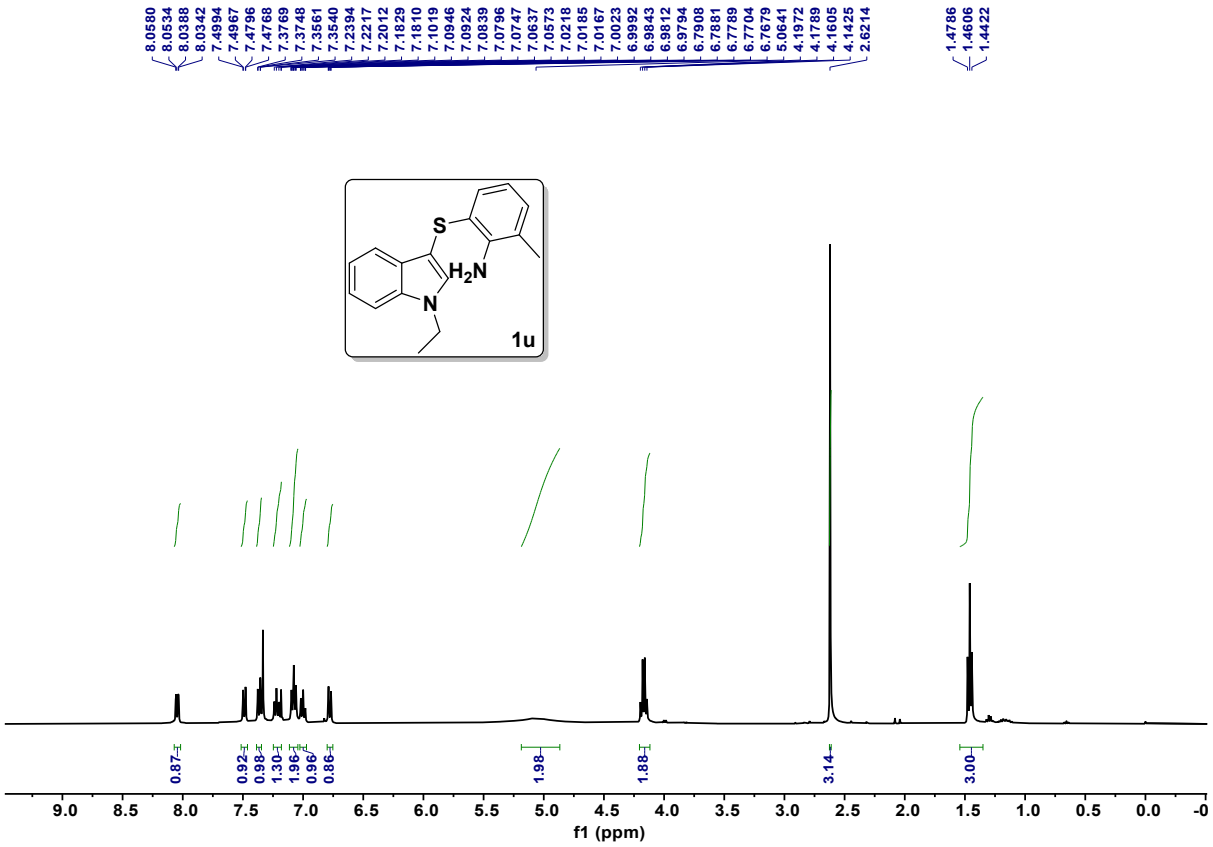


Figure S9. ^{1}H NMR of compound **1u** (400 MHz, $CDCl_3$)

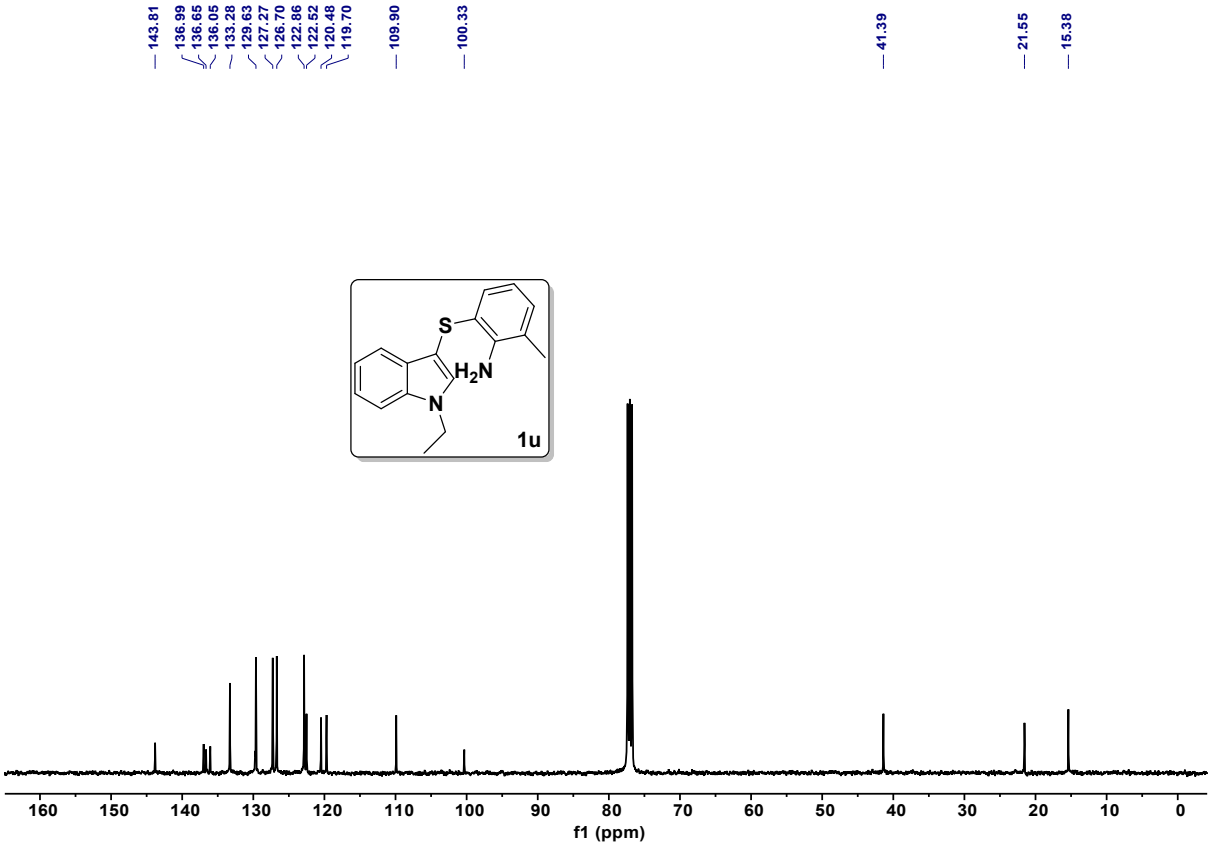


Figure S10. $^{13}C\{^{1}H\}$ NMR of compound **1u** (100 MHz, $CDCl_3$)

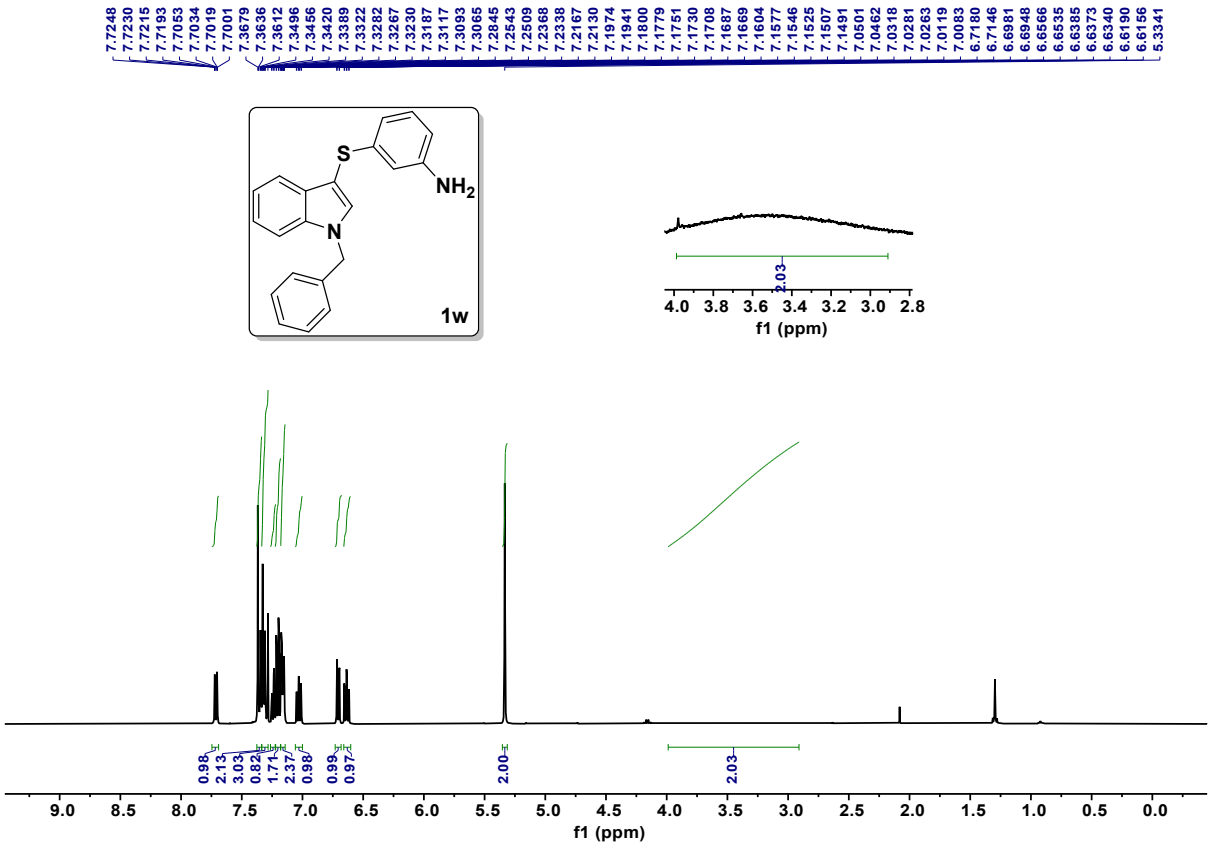


Figure S11. ^{1}H NMR of compound **1w** (400 MHz, $CDCl_3$)

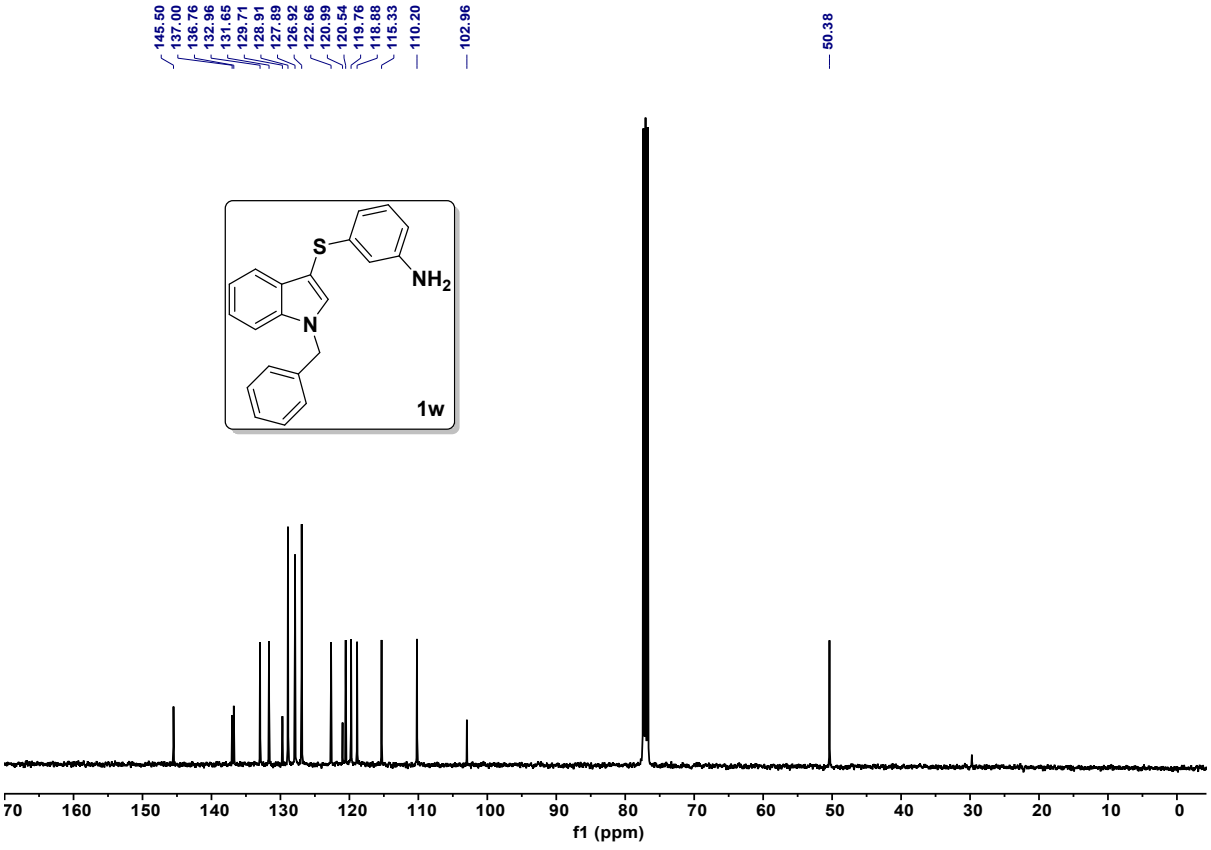


Figure S12. $^{13}C\{^{1}H\}$ NMR of compound **1w** (100 MHz, $CDCl_3$)

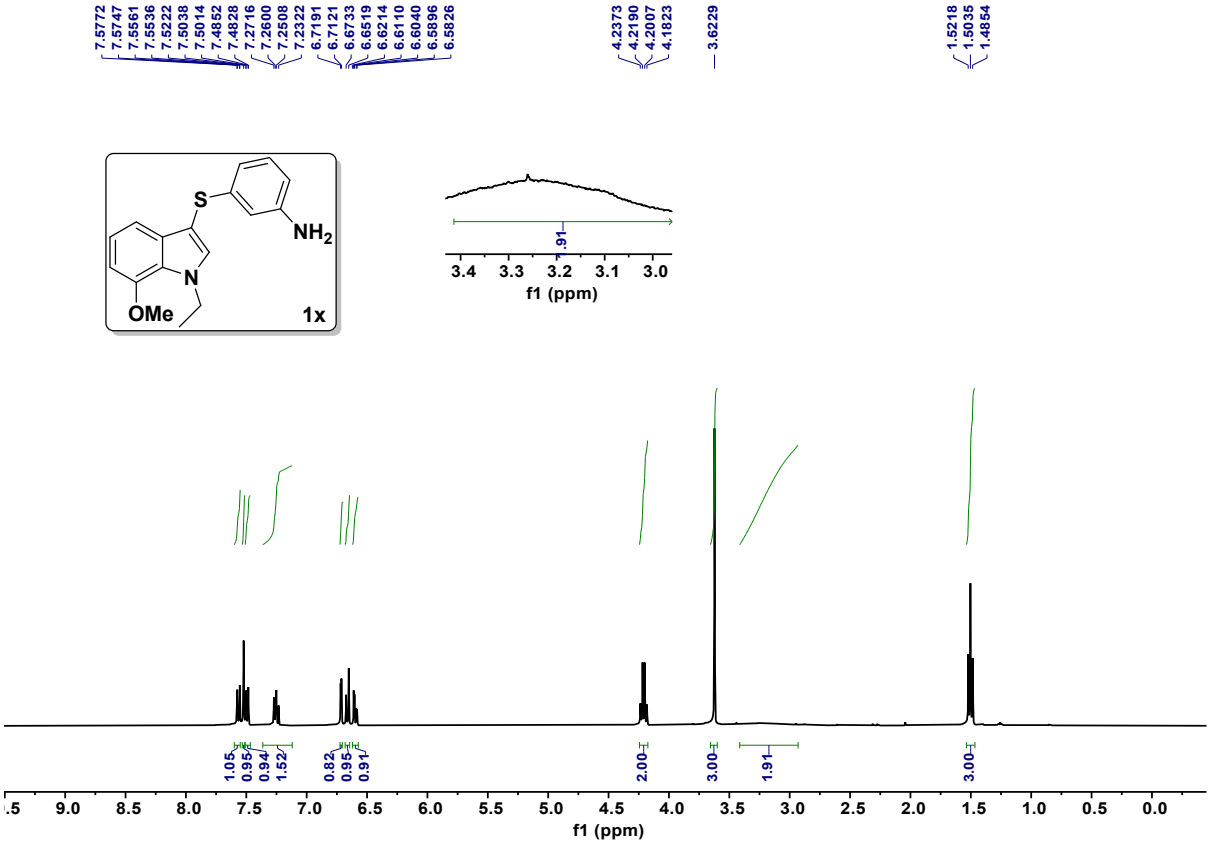


Figure S13. ^{1}H NMR of compound **1x** (400 MHz, $CDCl_3$)

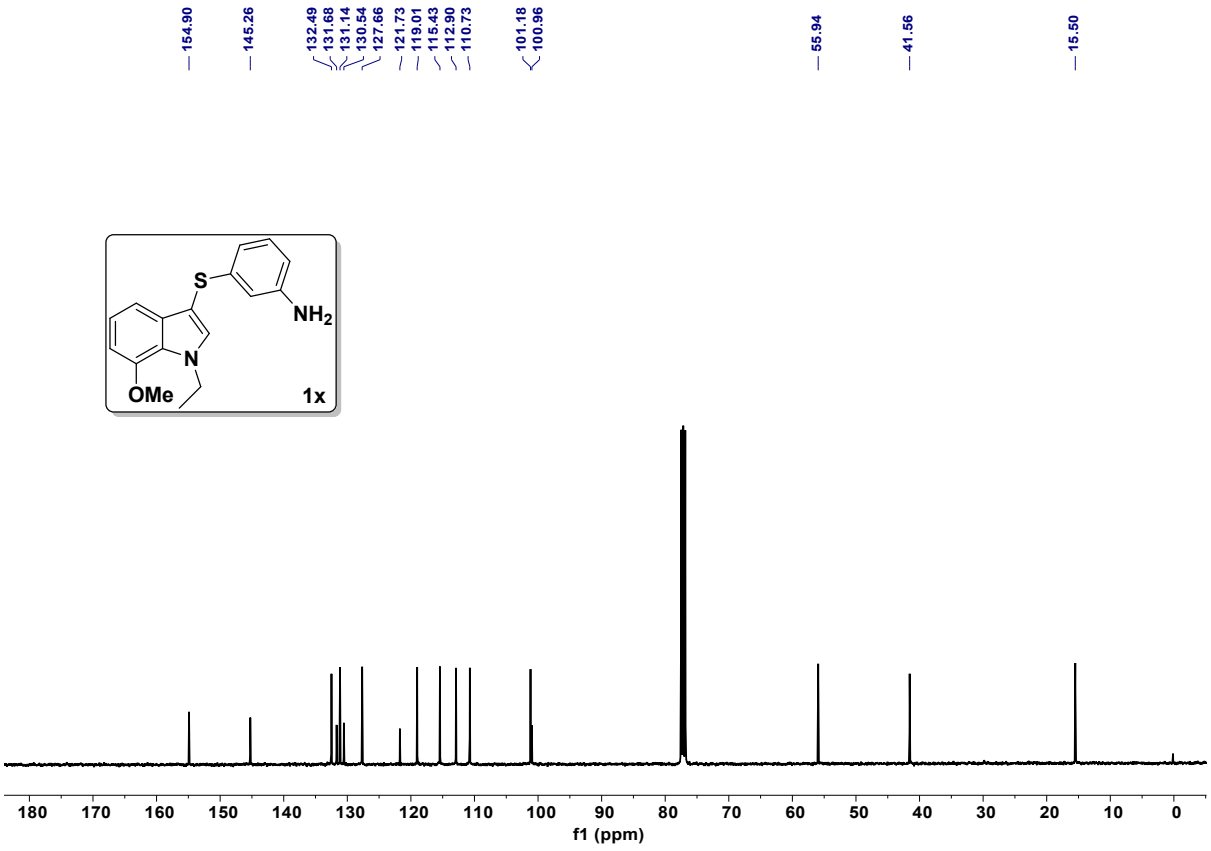


Figure S14. $^{13}C\{^{1}H\}$ NMR of compound **1x** (100 MHz, $CDCl_3$)

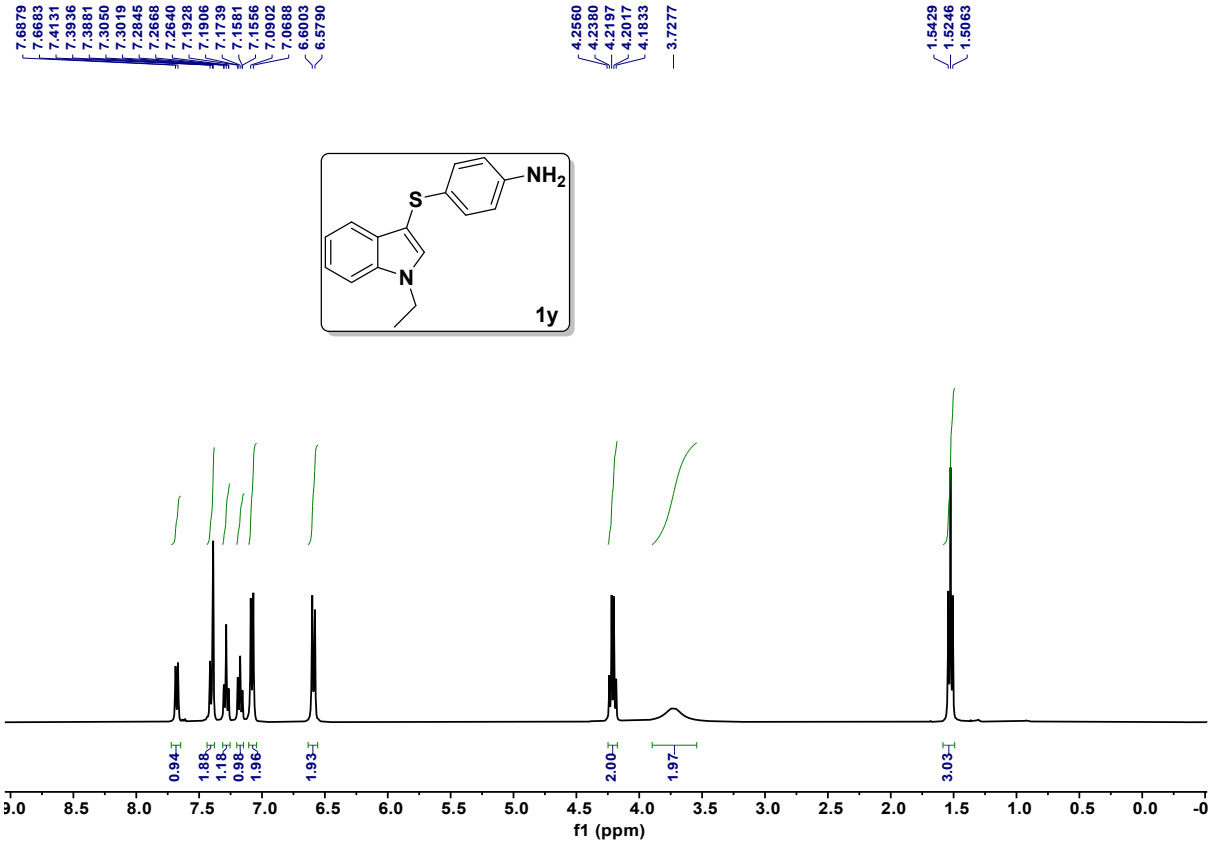


Figure S15. ^{1}H NMR of compound **1y** (400 MHz, $CDCl_3$)

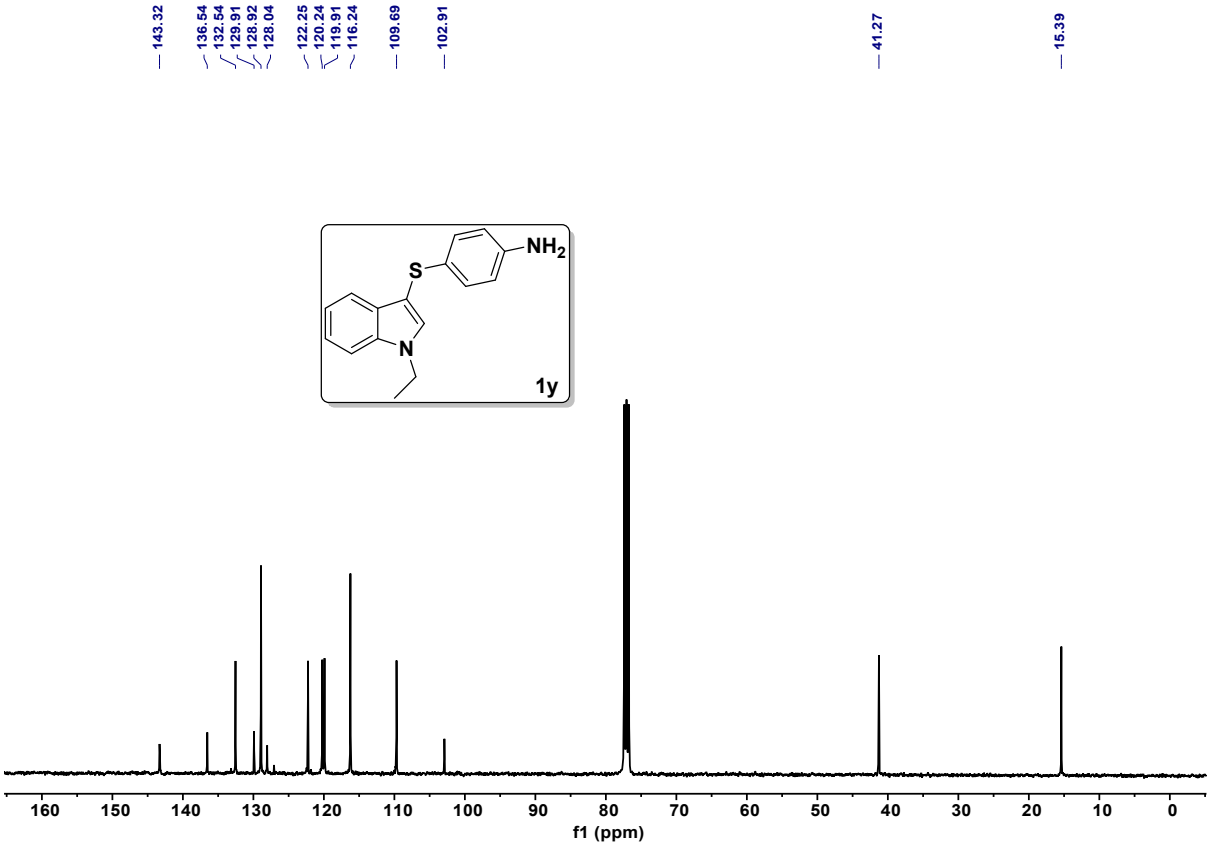


Figure S16. $^{13}C\{^{1}H\}$ NMR of compound **1y** (100 MHz, $CDCl_3$)

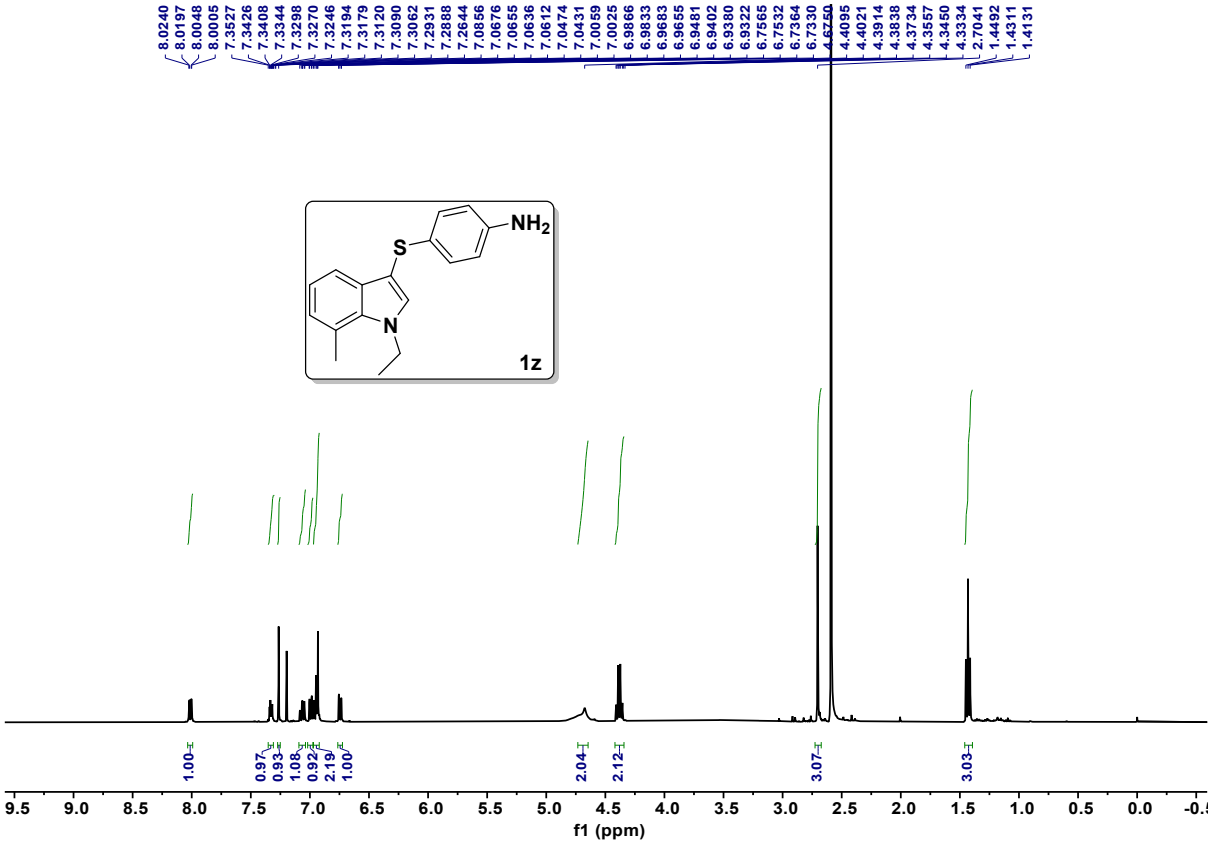


Figure S17. ^{1}H NMR of compound **1z** (400 MHz, $CDCl_3$)

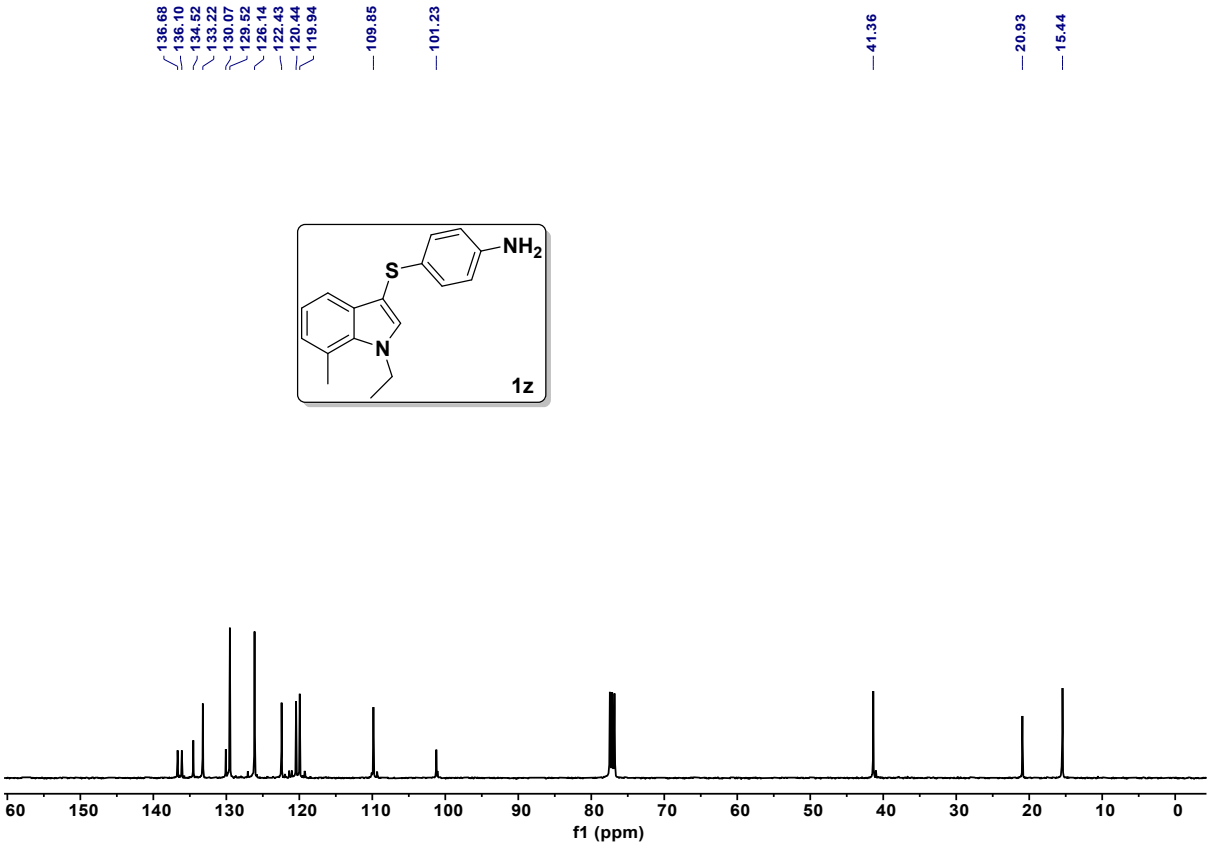


Figure S18. $^{13}C\{^{1}H\}$ NMR of compound **1z** (100 MHz, $CDCl_3$)

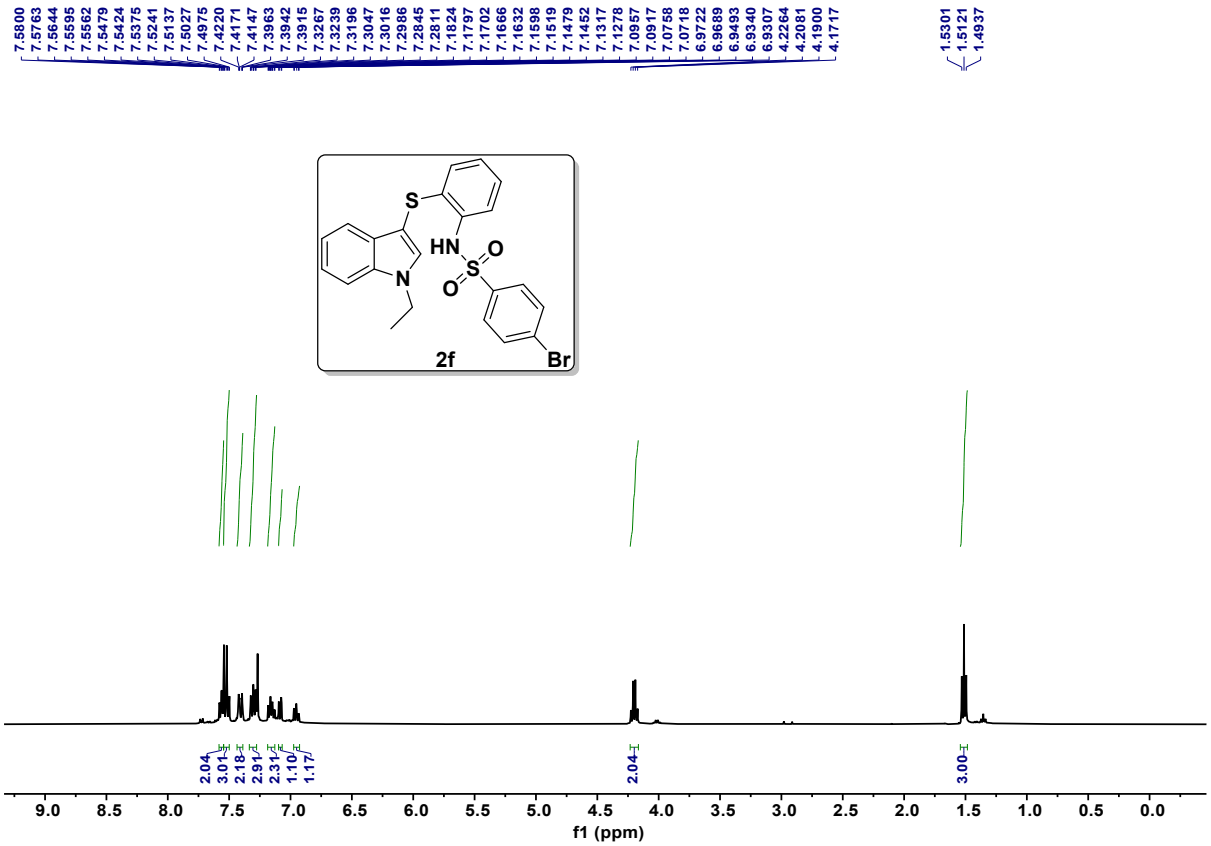


Figure S19. ^{1}H NMR of compound **2f** (400 MHz, $CDCl_3$)

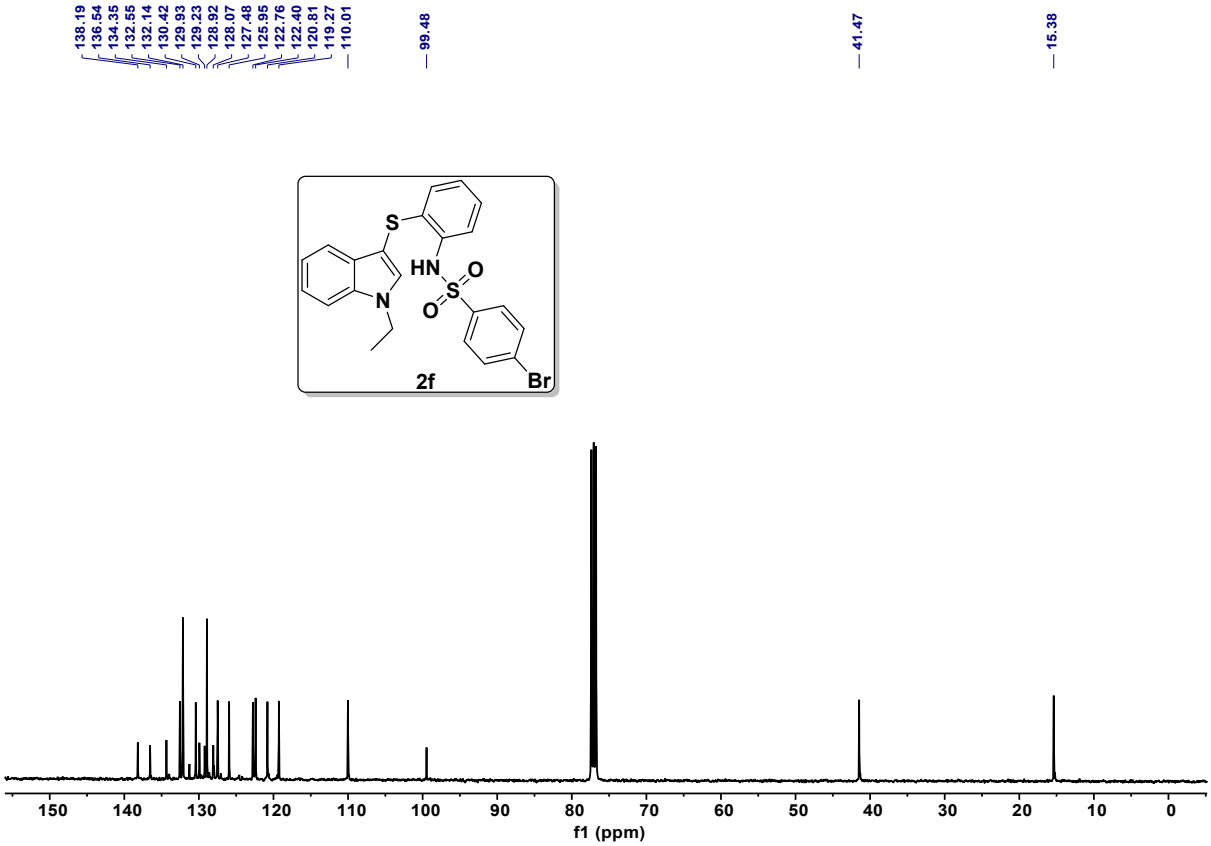


Figure S20. $^{13}C\{^{1}H\}$ NMR of compound **2f** (100 MHz, $CDCl_3$)

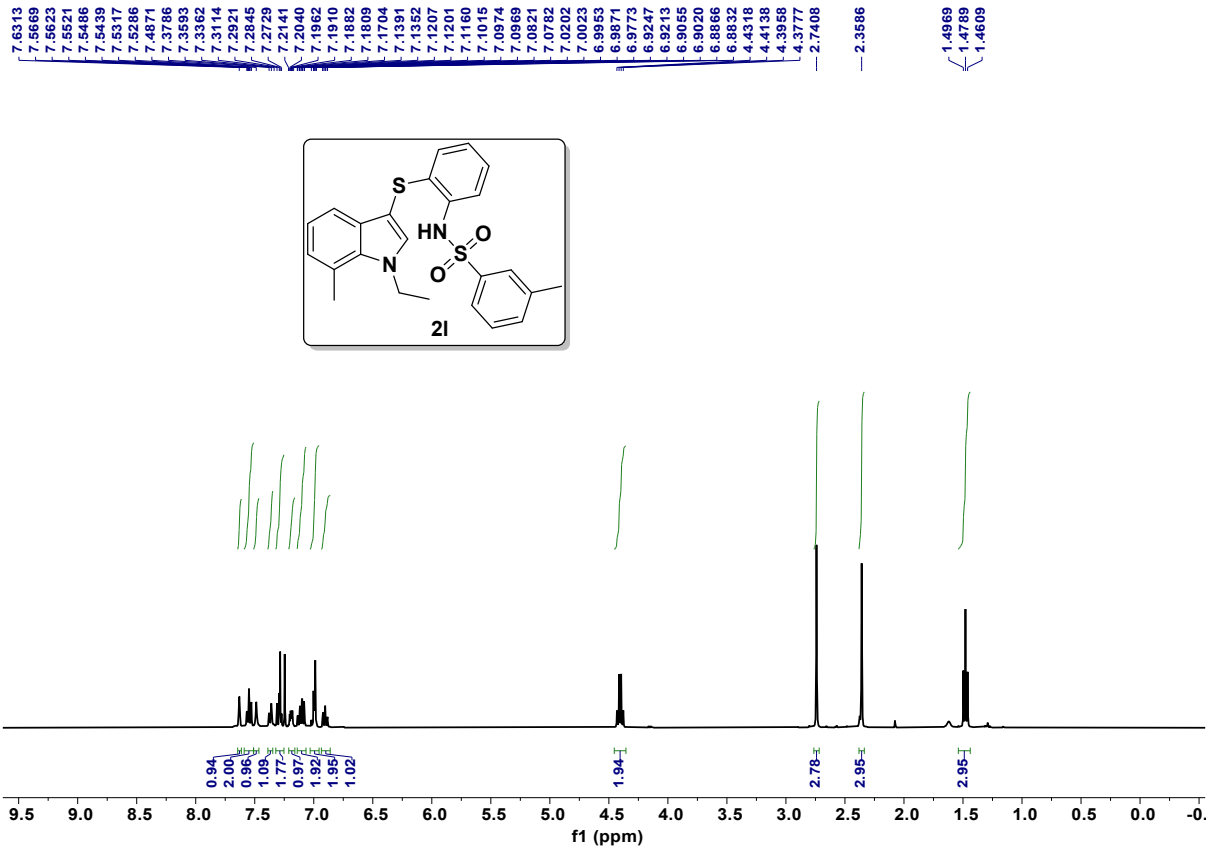


Figure S21. ^{1}H NMR of compound **2l** (400 MHz, $CDCl_3$)

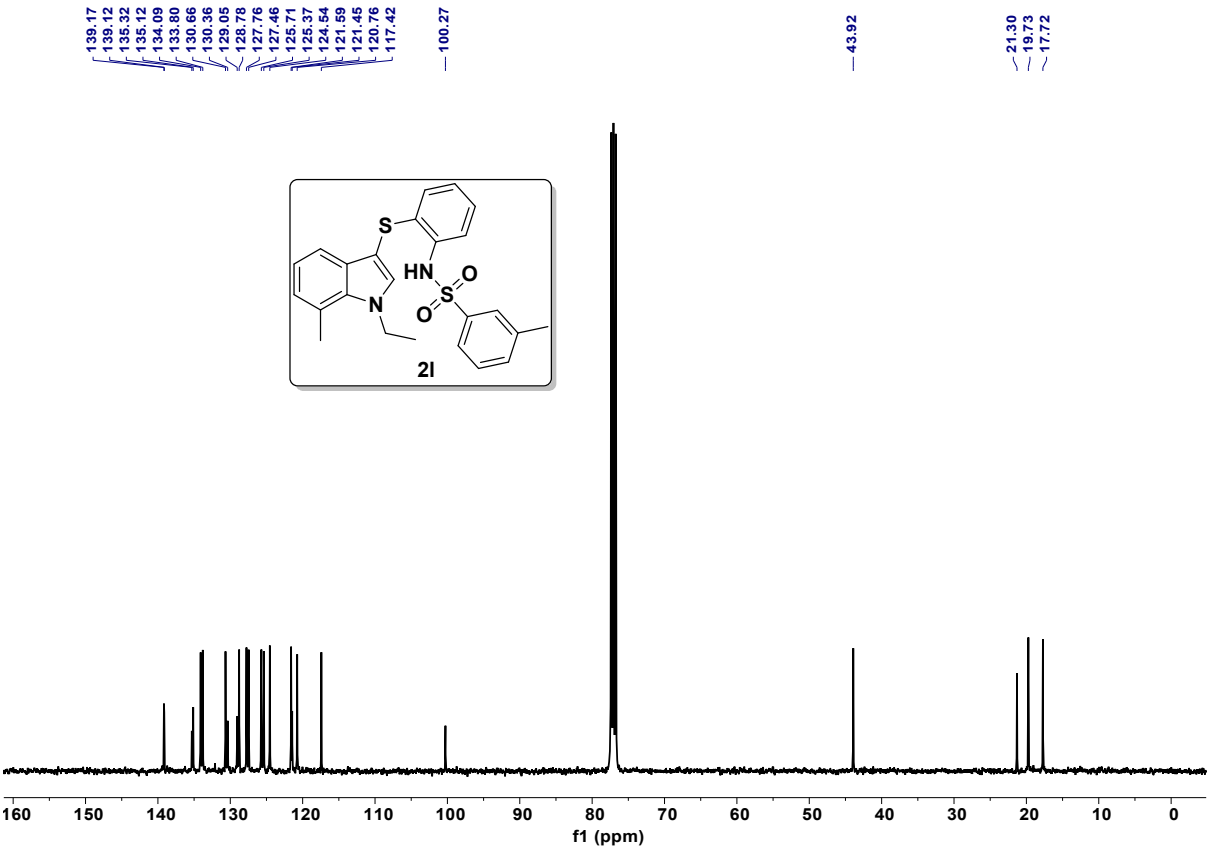


Figure S22. $^{13}C\{^{1}H\}$ NMR of compound **2l** (100 MHz, $CDCl_3$)

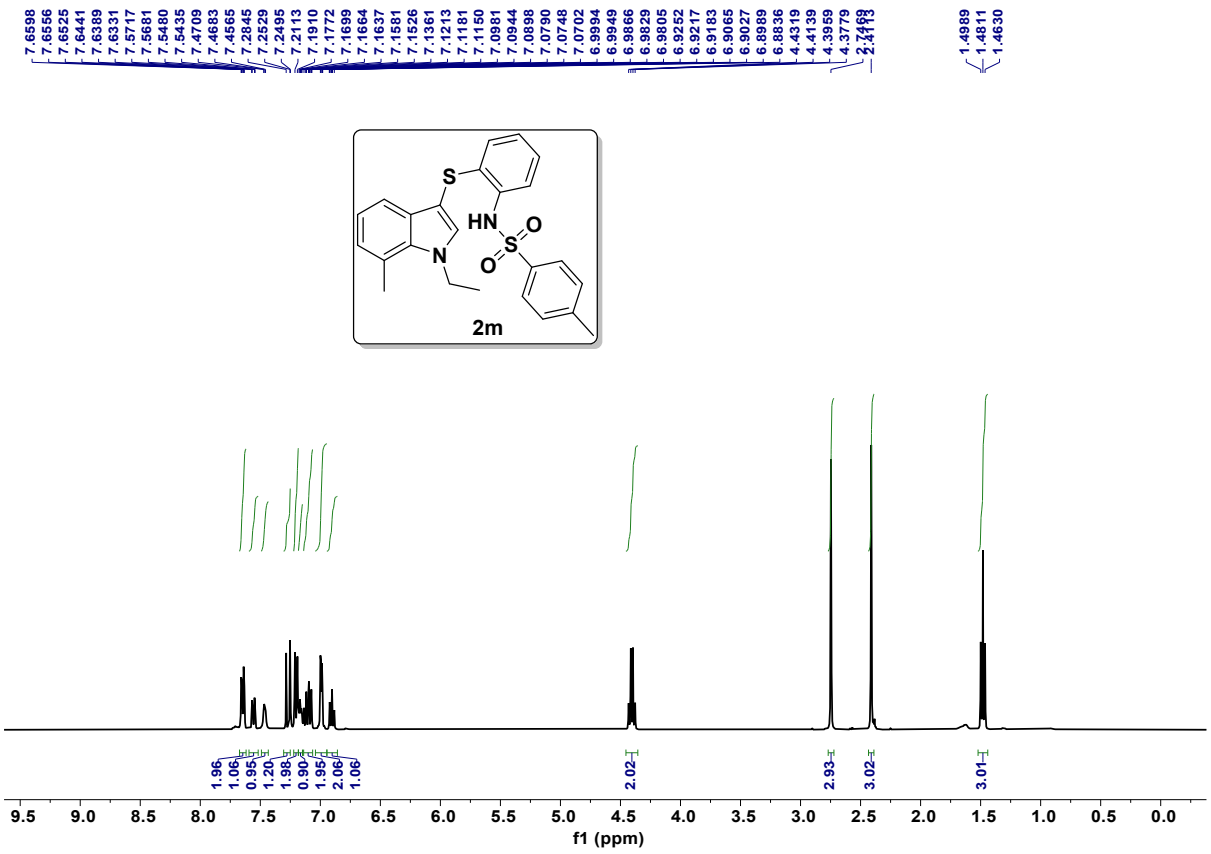


Figure S23. ^{1}H NMR of compound **2m** (400 MHz, $CDCl_3$)

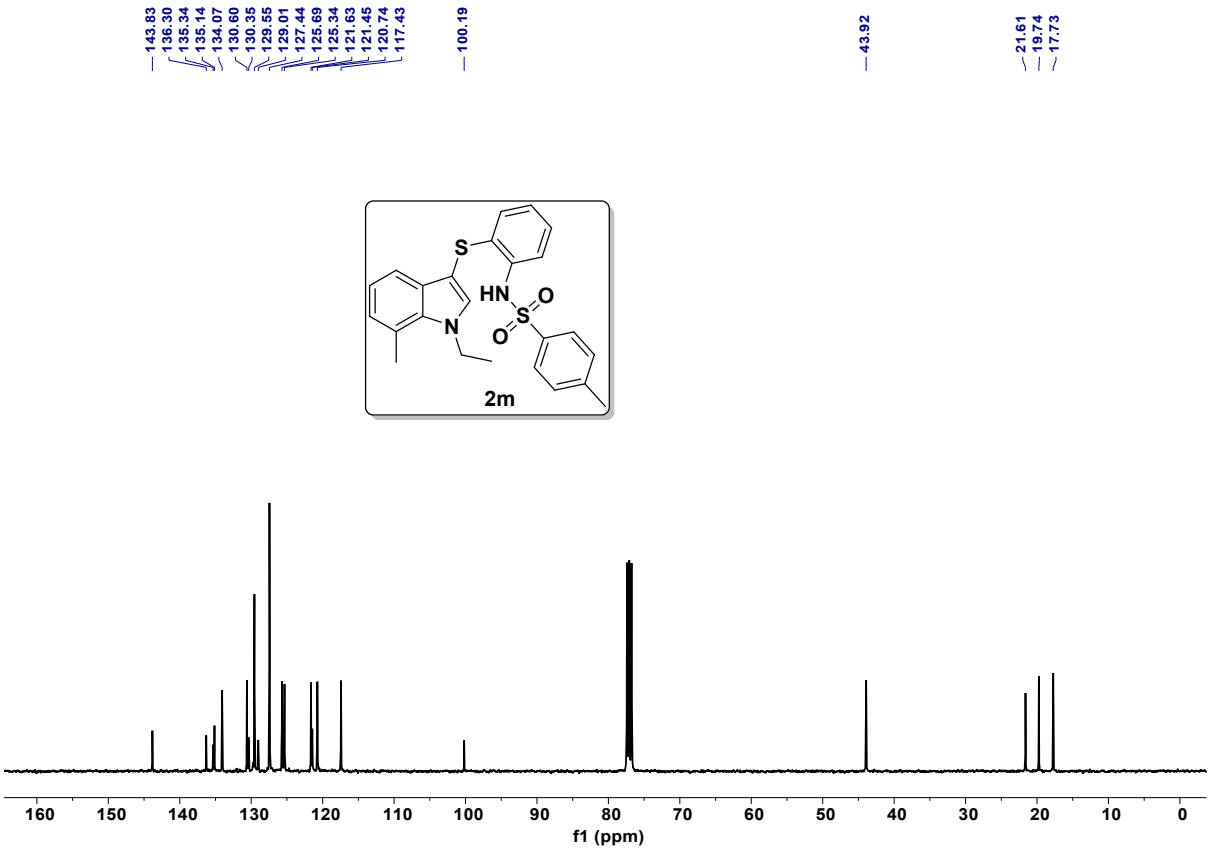


Figure S24. $^{13}C\{^{1}H\}$ NMR of compound **2m** (100 MHz, $CDCl_3$)

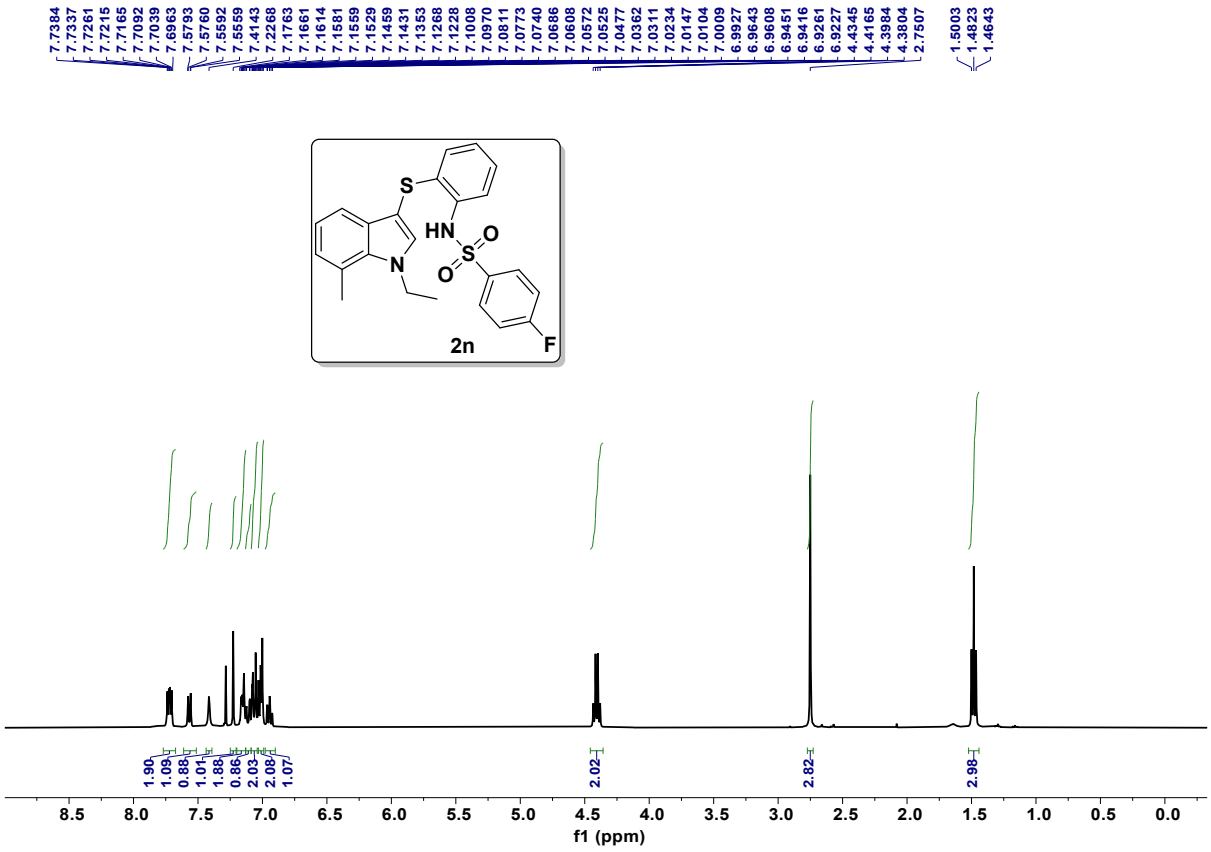


Figure S25. ^{1}H NMR of compound **2n** (400 MHz, $CDCl_3$)

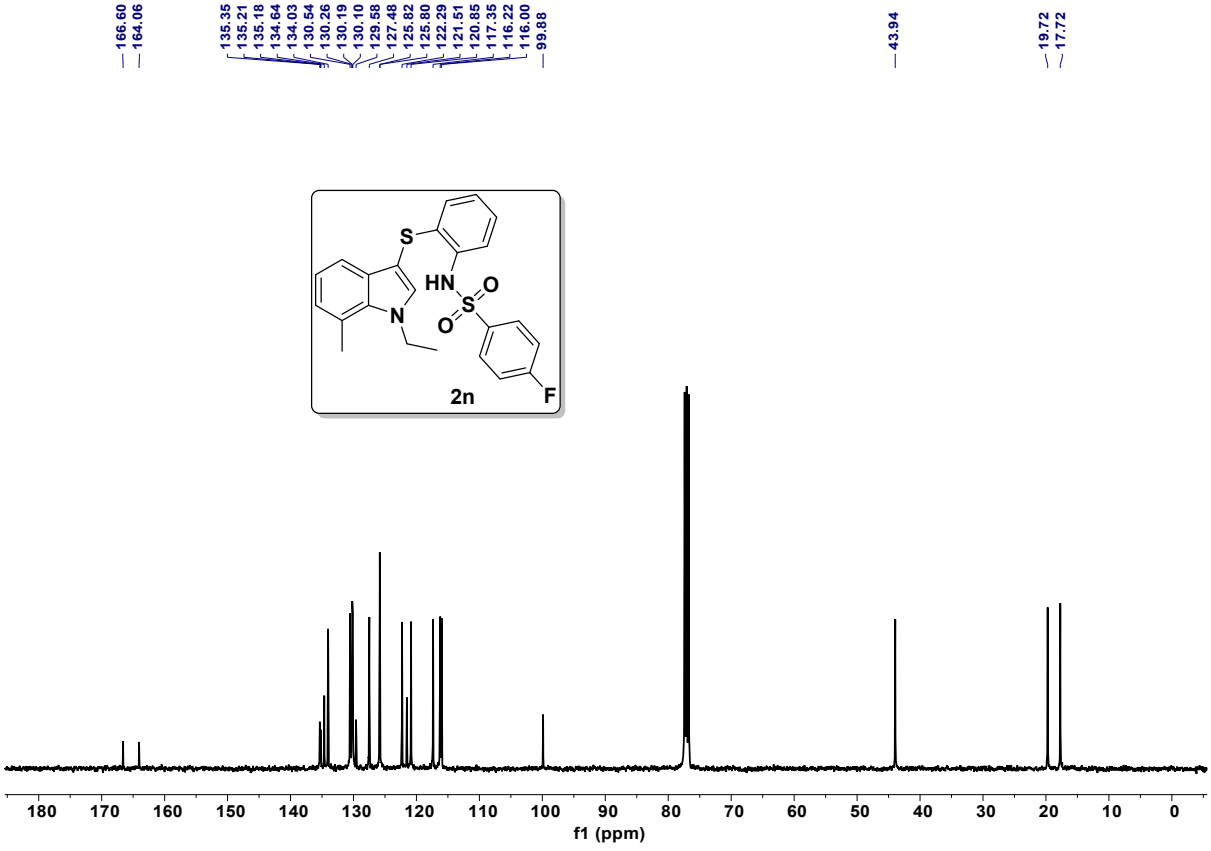


Figure S26. $^{13}C\{^{1}H\}$ NMR of compound **2n** (100 MHz, $CDCl_3$)

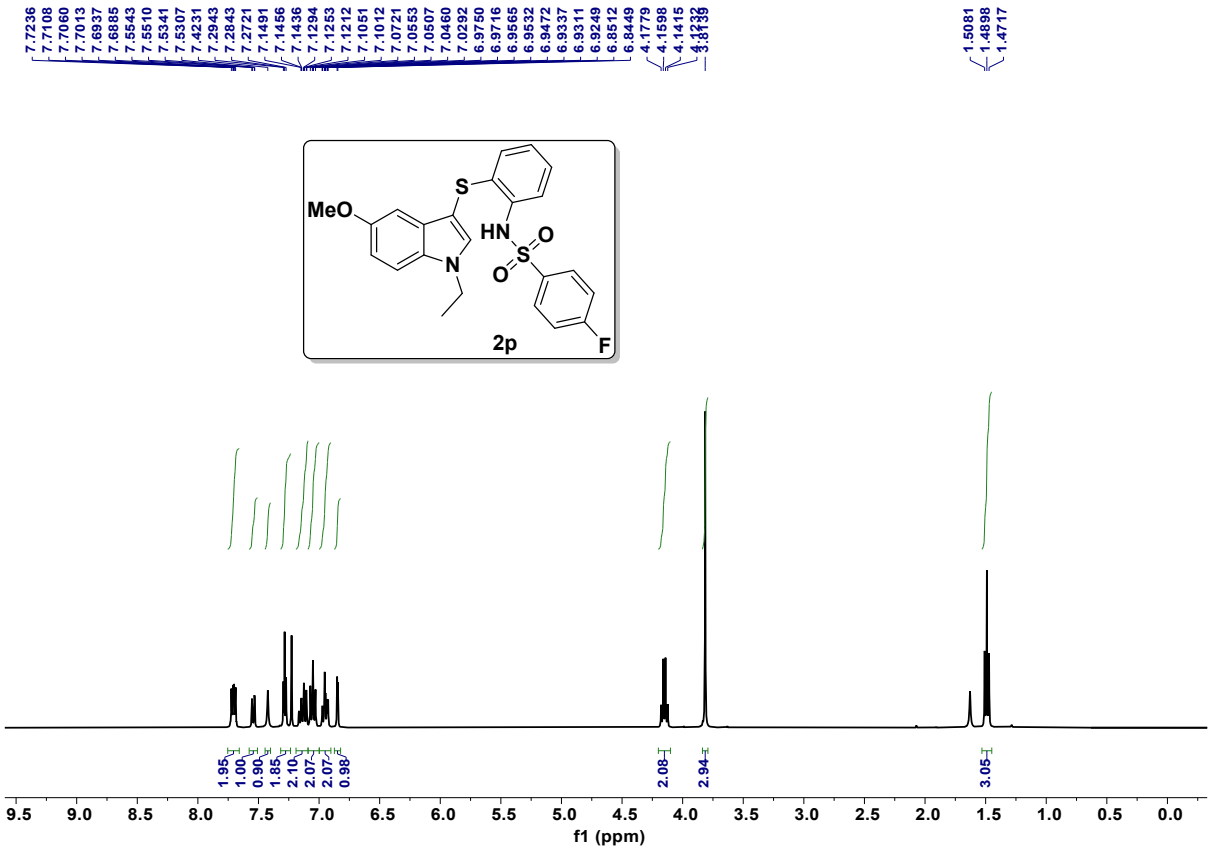


Figure S27. ^{1}H NMR of compound **2p** (400 MHz, $CDCl_3$)

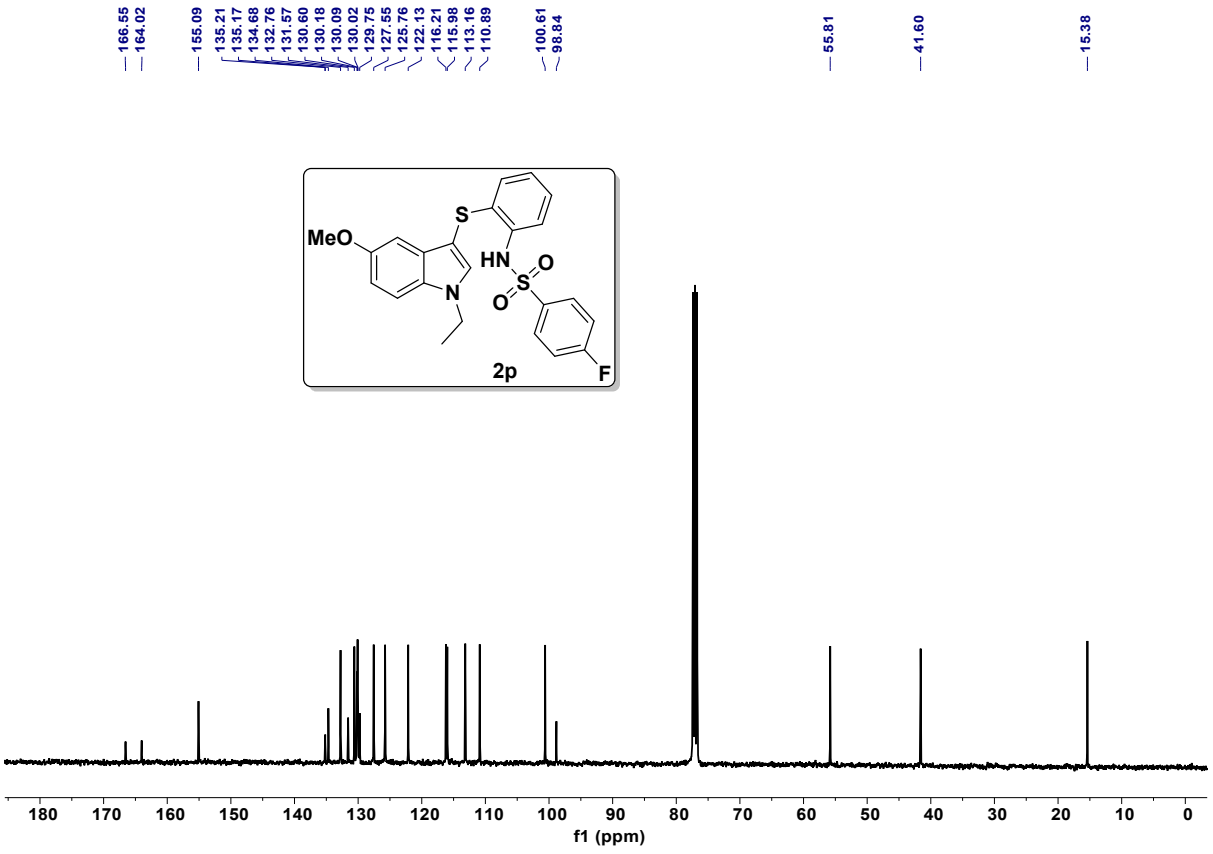


Figure S28. $^{13}C\{^{1}H\}$ NMR of compound **2p** (100 MHz, $CDCl_3$)

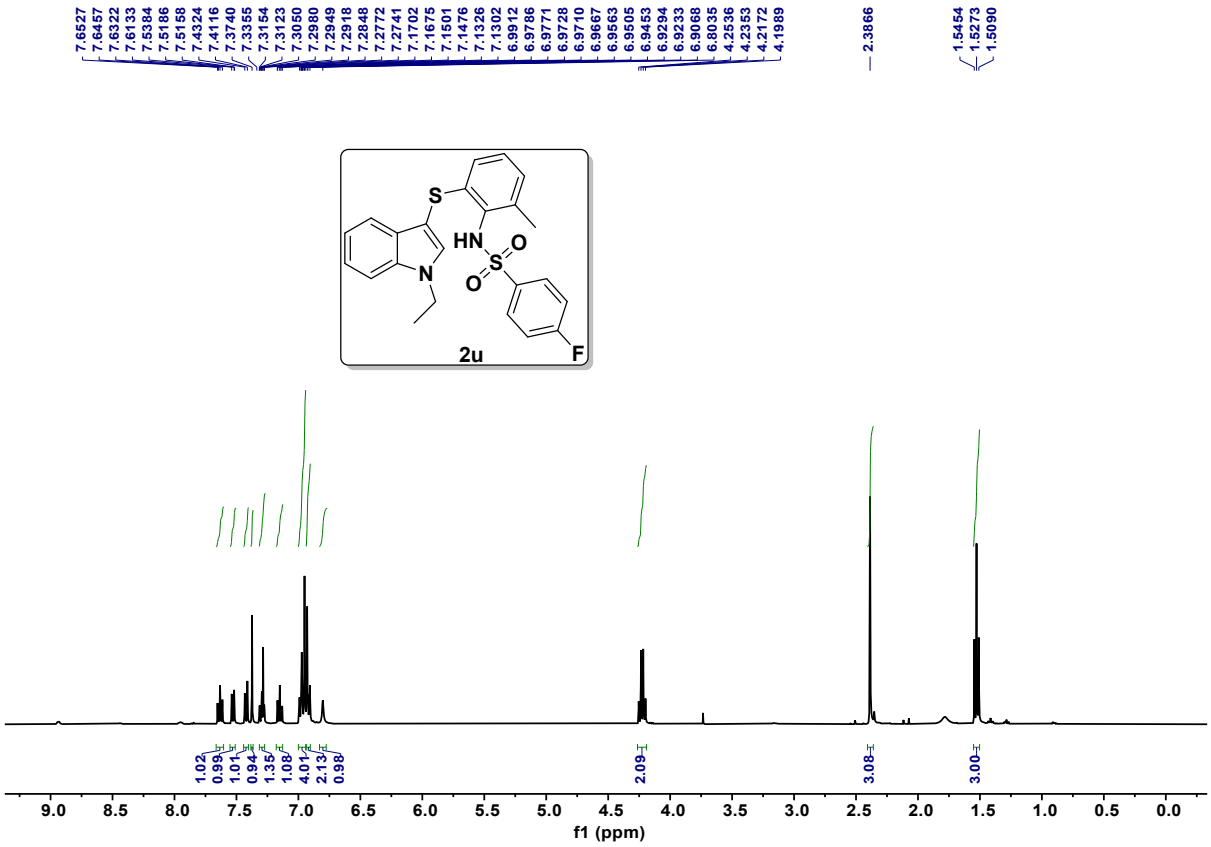


Figure S29. ^{1}H NMR of compound **2u** (400 MHz, $CDCl_3$)

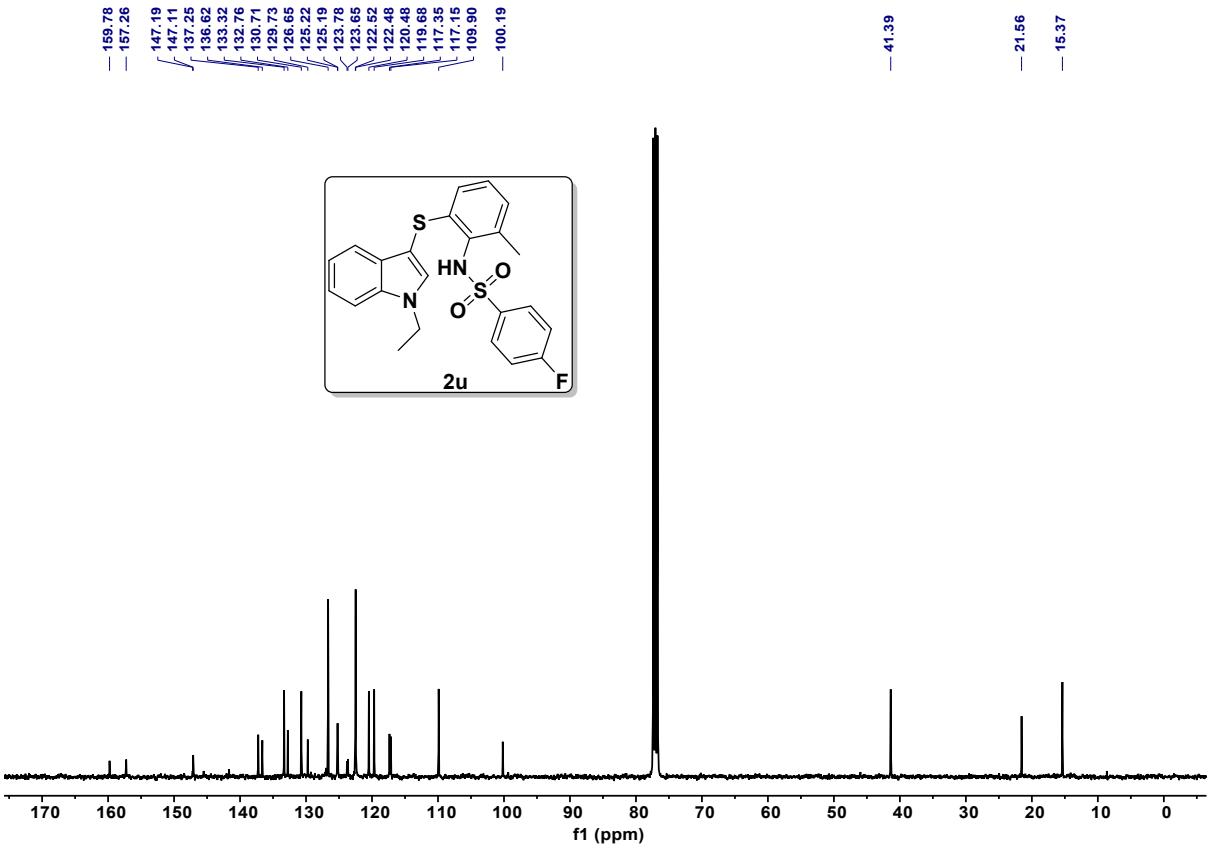


Figure S30. $^{13}C\{^{1}H\}$ NMR of compound **2u** (100 MHz, $CDCl_3$)

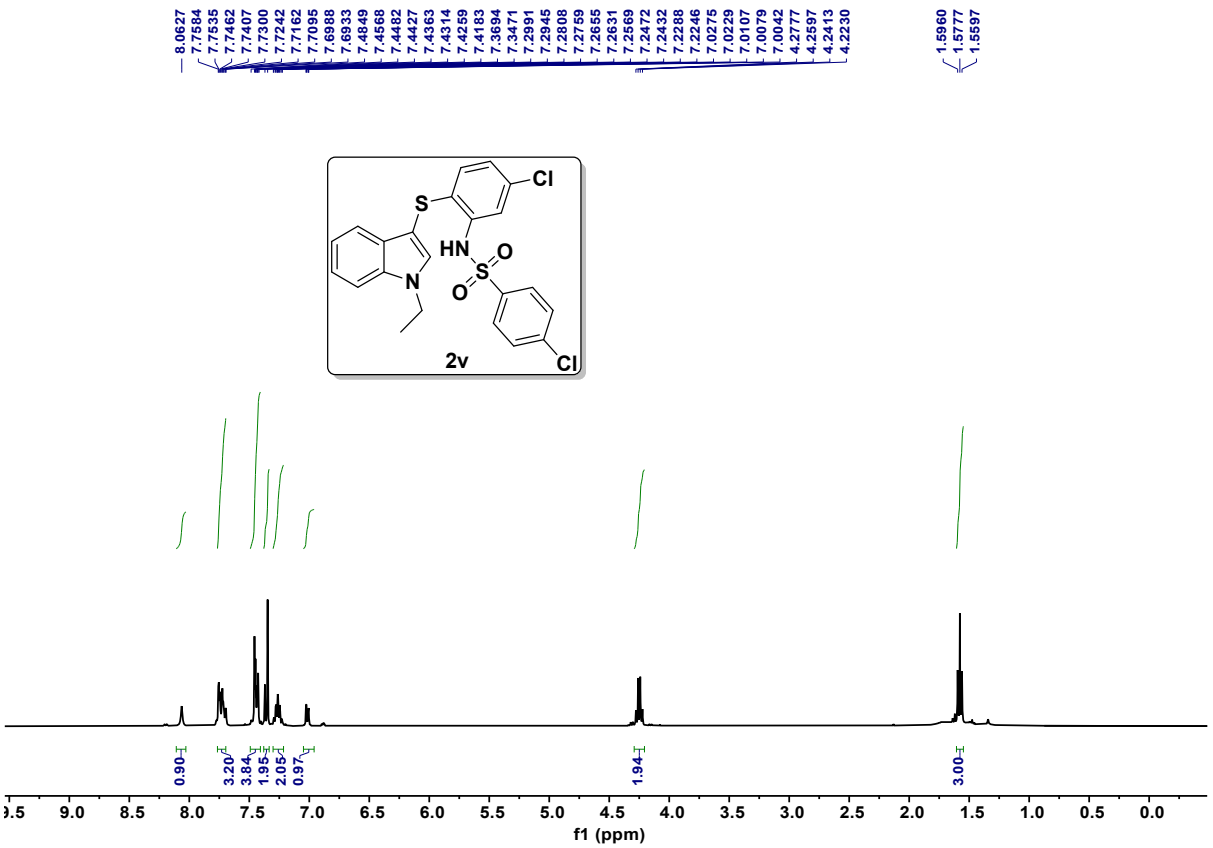


Figure S31. ^{1}H NMR of compound **2v** (400 MHz, $CDCl_3$)

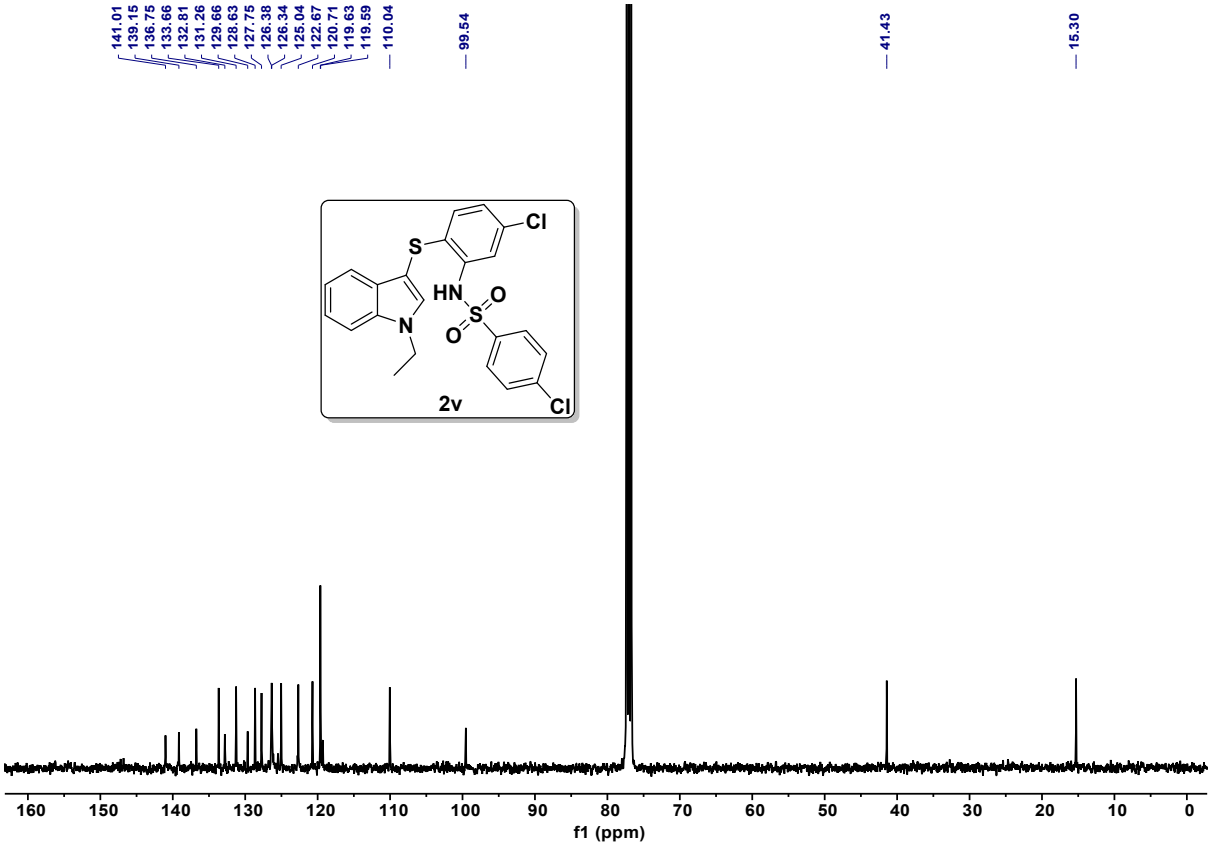


Figure S32. $^{13}C\{^{1}H\}$ NMR of compound **2v** (100 MHz, $CDCl_3$)

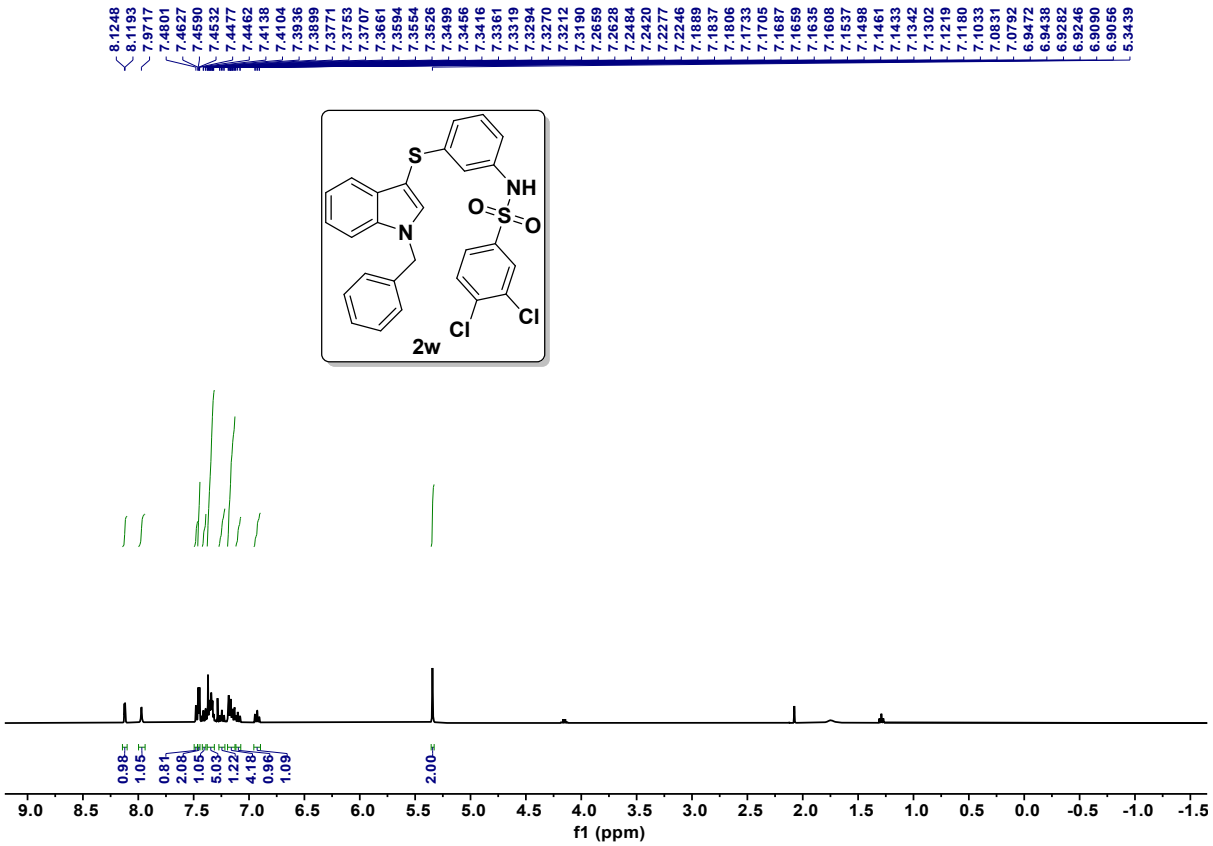


Figure S33. ^{1}H NMR of compound **2w** (400 MHz, $CDCl_3$)

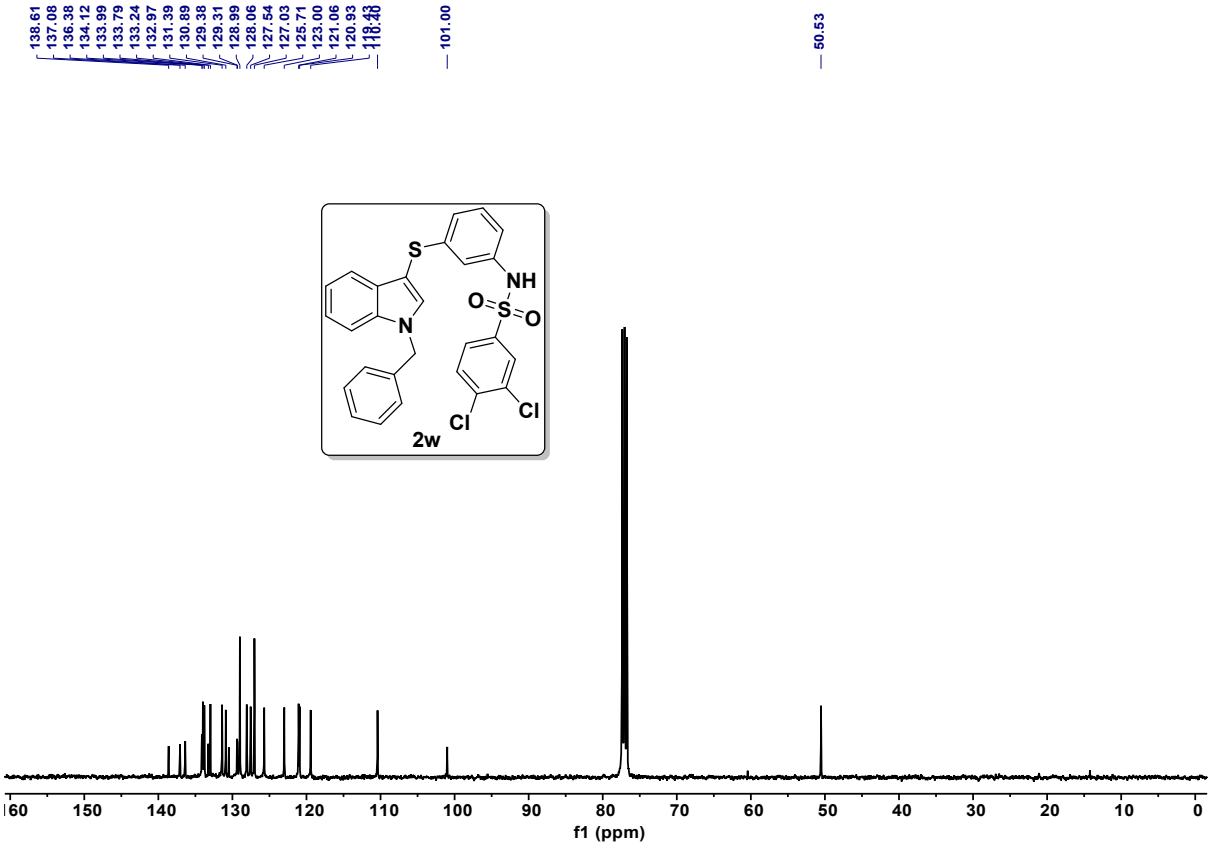


Figure S34. $^{13}C\{^{1}H\}$ NMR of compound **2w** (100 MHz, $CDCl_3$)

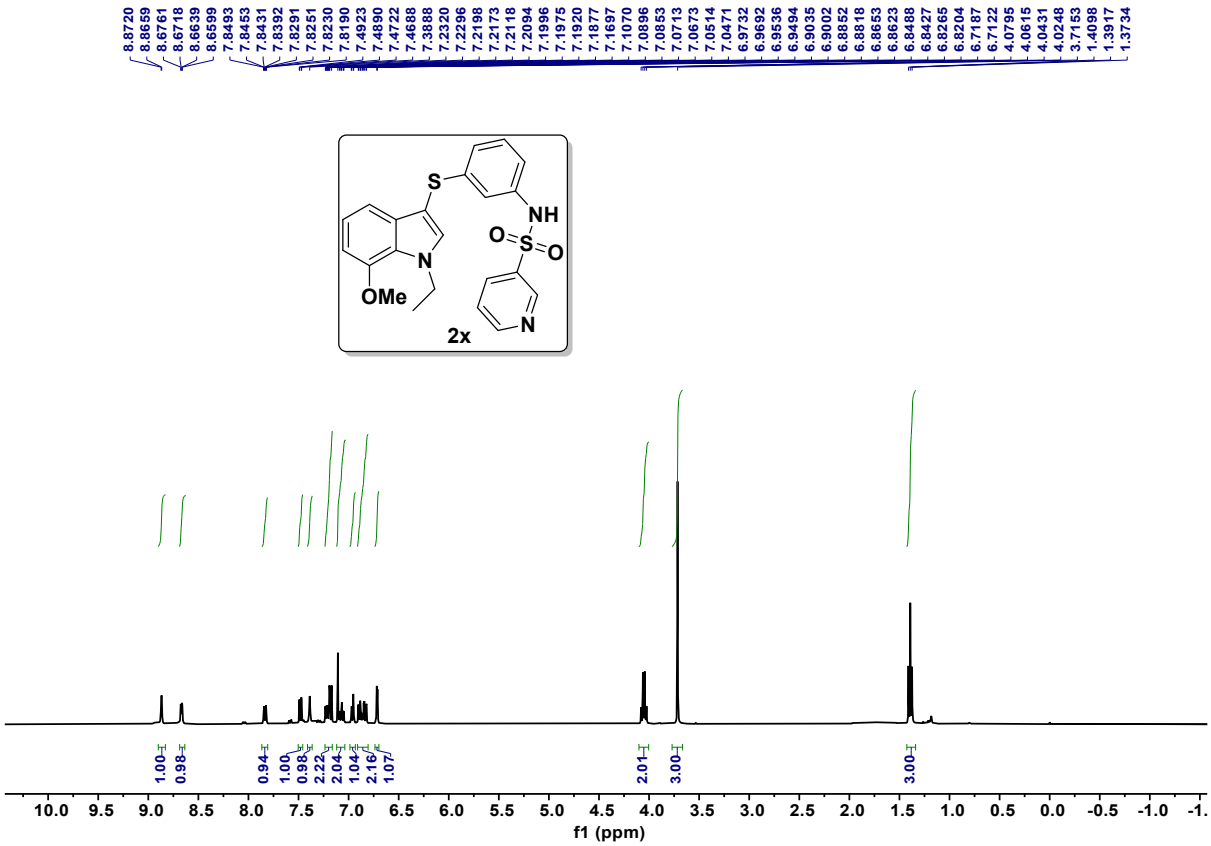


Figure S35. ^{1}H NMR of compound **2x** (400 MHz, $CDCl_3$)

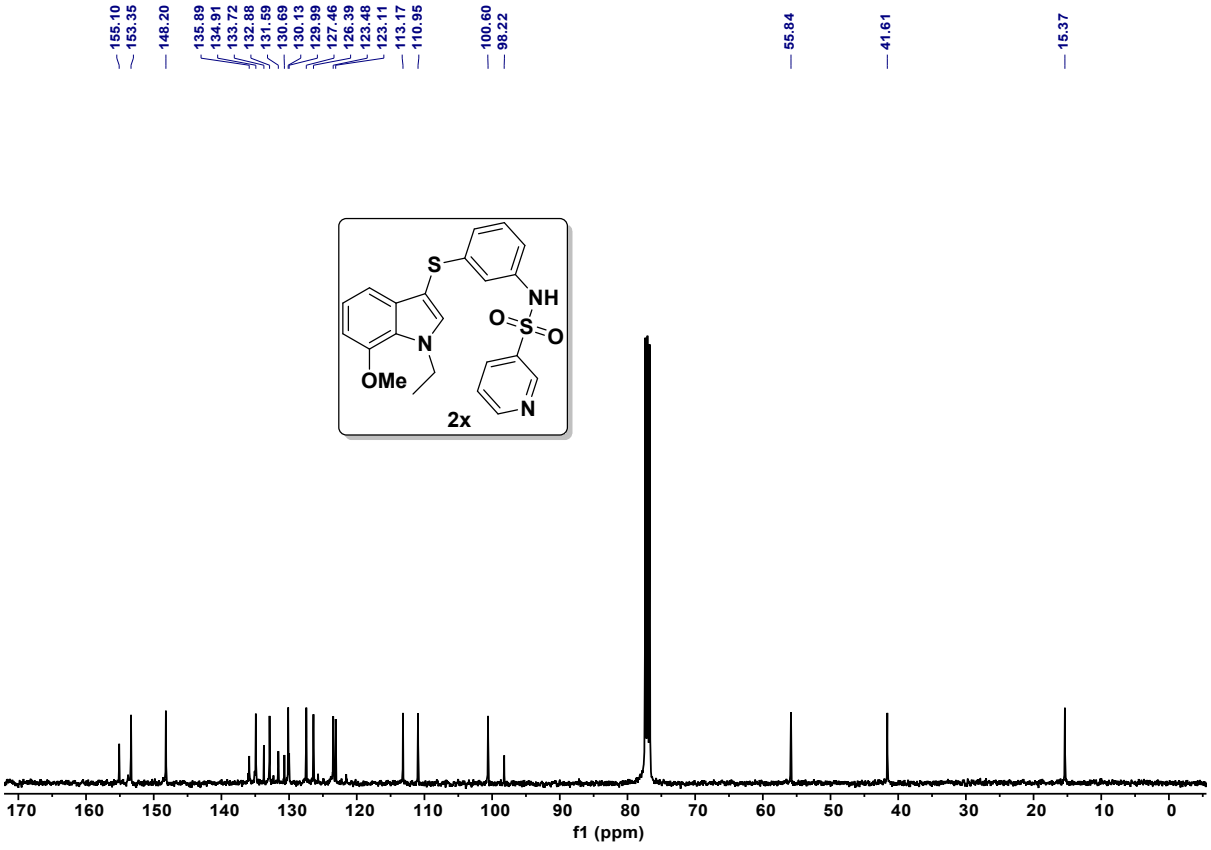


Figure S36. $^{13}C\{^{1}H\}$ NMR of compound **2x** (100 MHz, $CDCl_3$)

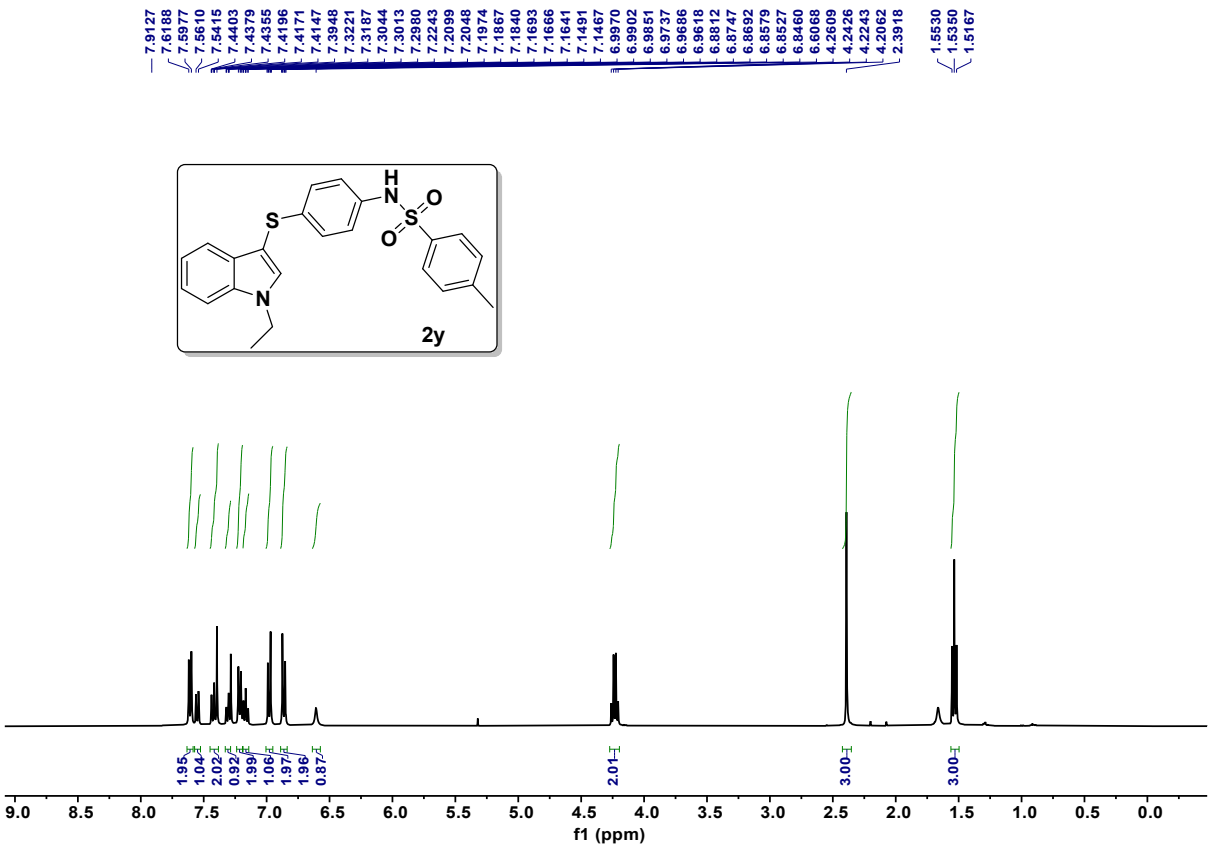


Figure S37. ^{1}H NMR of compound **2y** (400 MHz, $CDCl_3$)

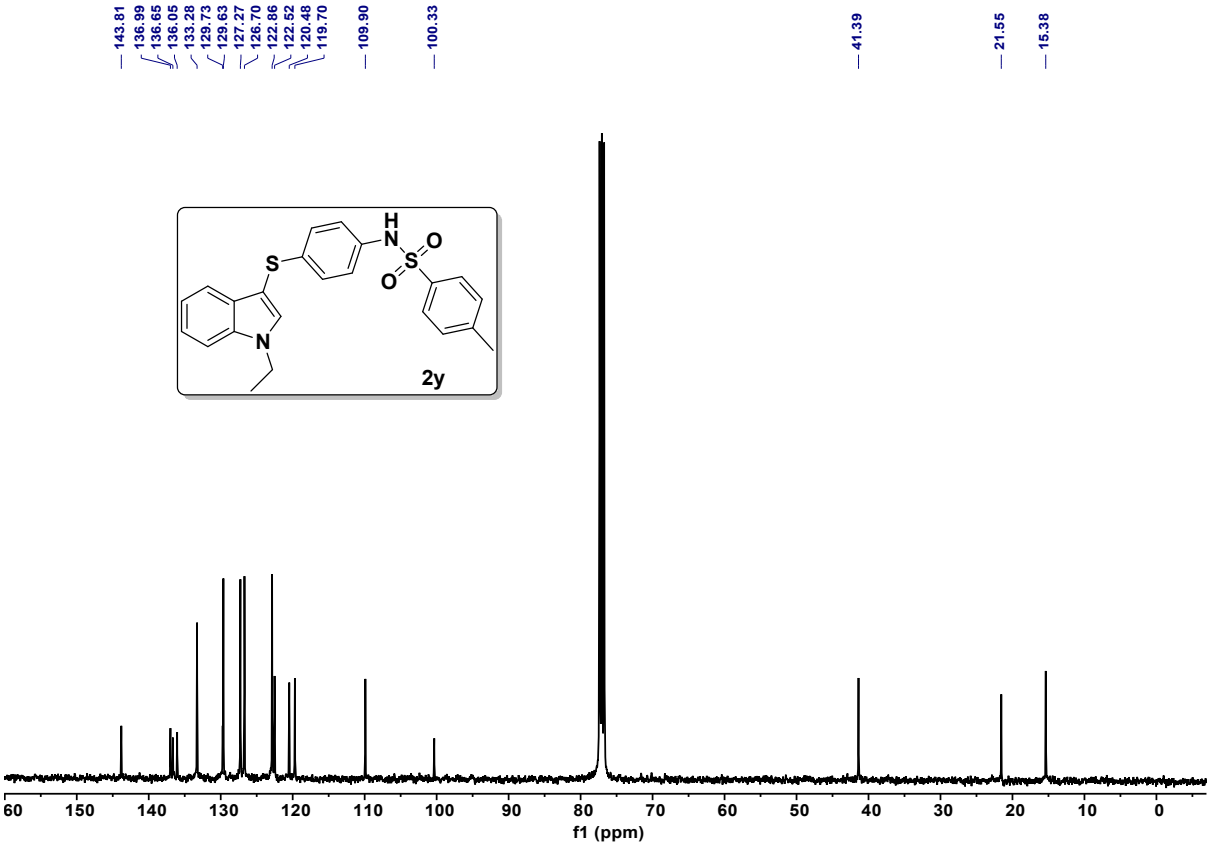


Figure S38. $^{13}C\{^{1}H\}$ NMR of compound **2y** (100 MHz, $CDCl_3$)

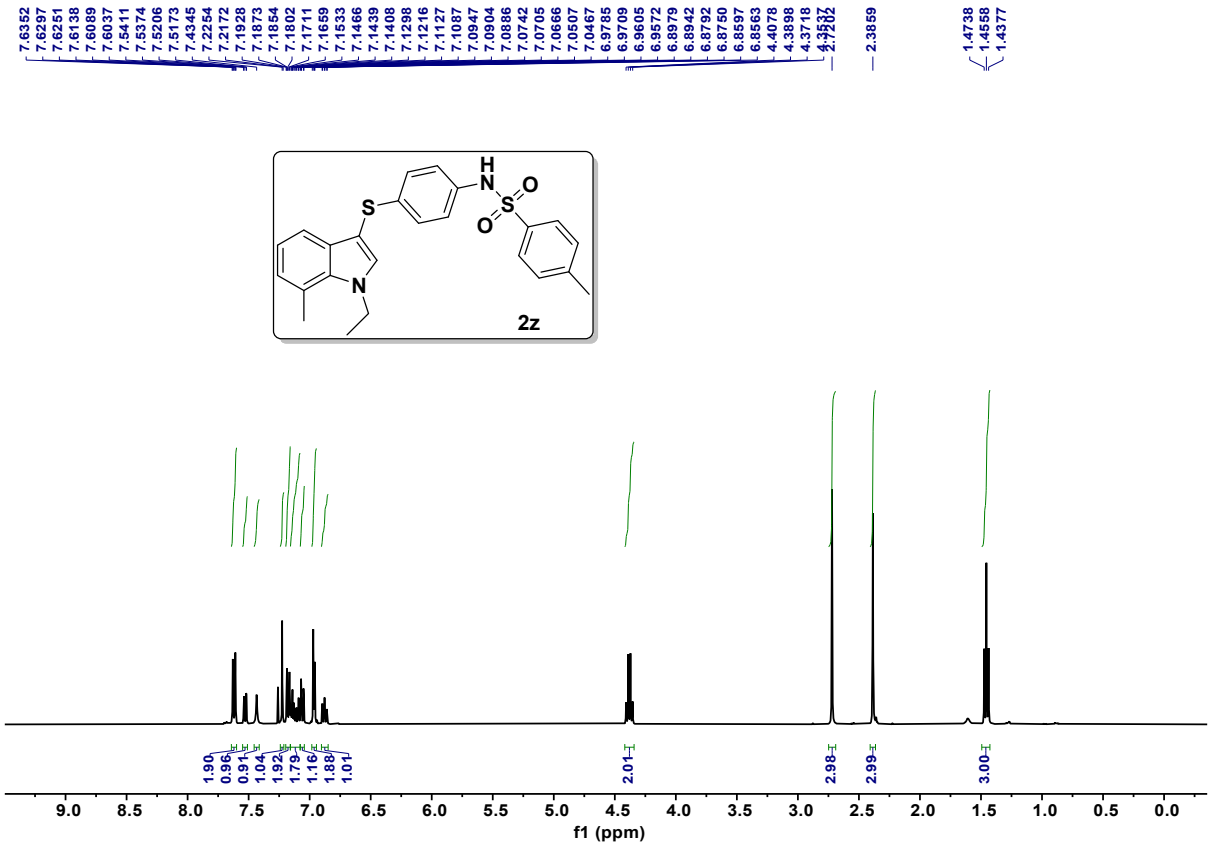


Figure S39. ^{1}H NMR of compound **2z** (400 MHz, $CDCl_3$)

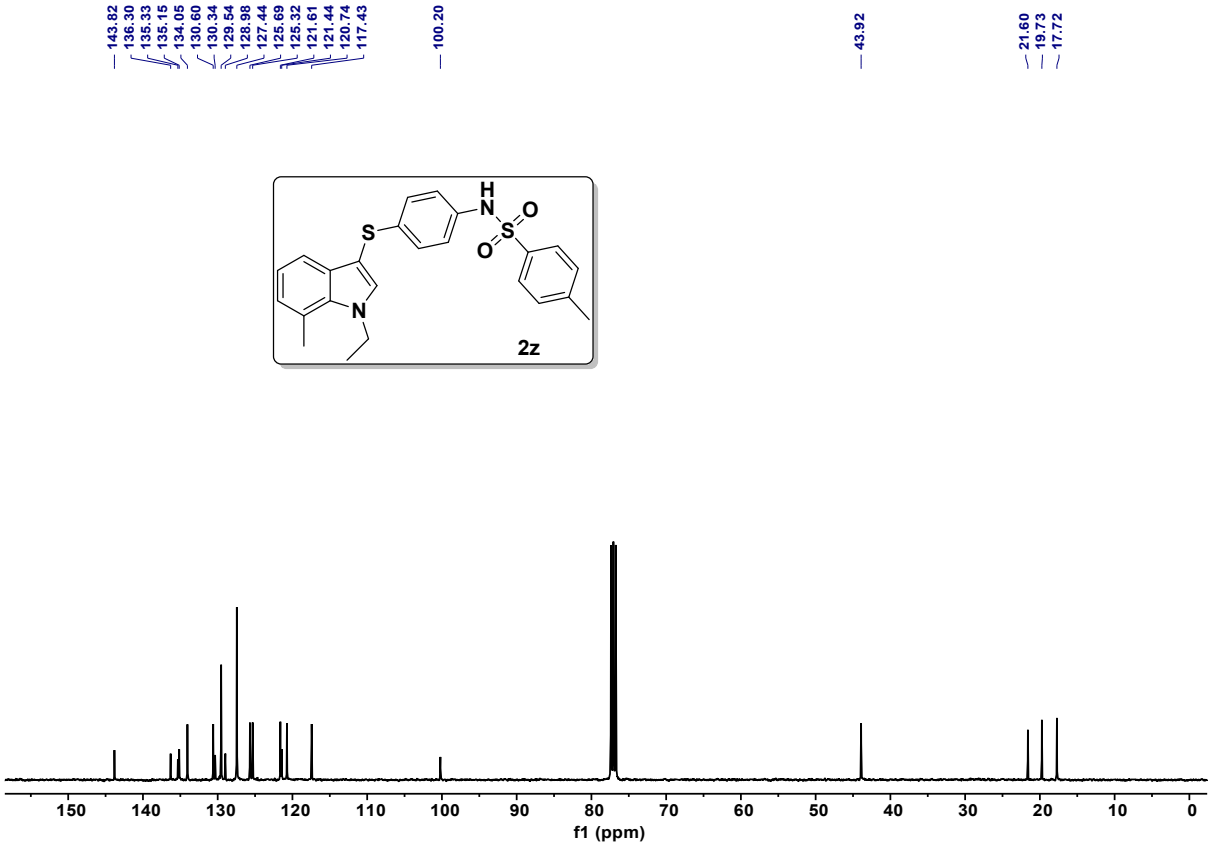


Figure S40. $^{13}C\{^{1}H\}$ NMR of compound **2z** (100 MHz, $CDCl_3$)

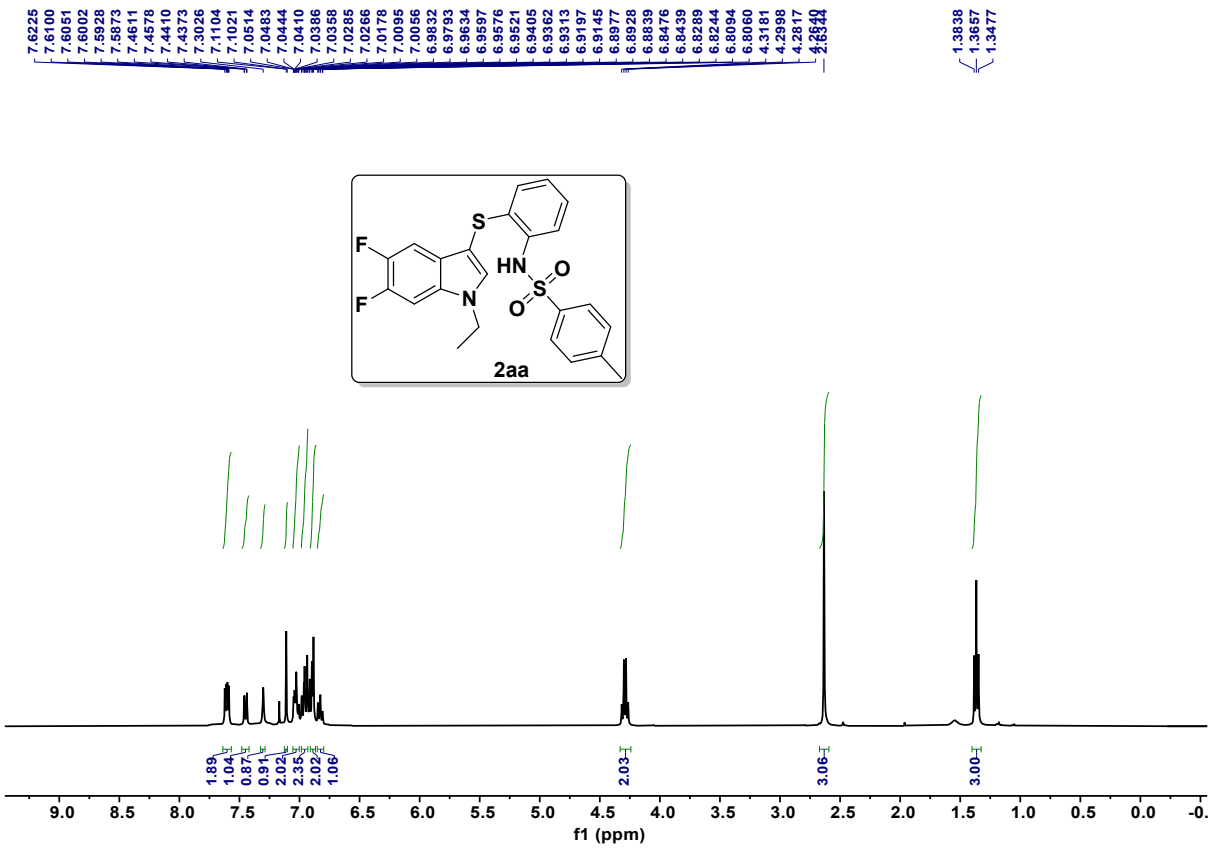


Figure S41. ^{1}H NMR of compound **2aa** (400 MHz, $CDCl_3$)

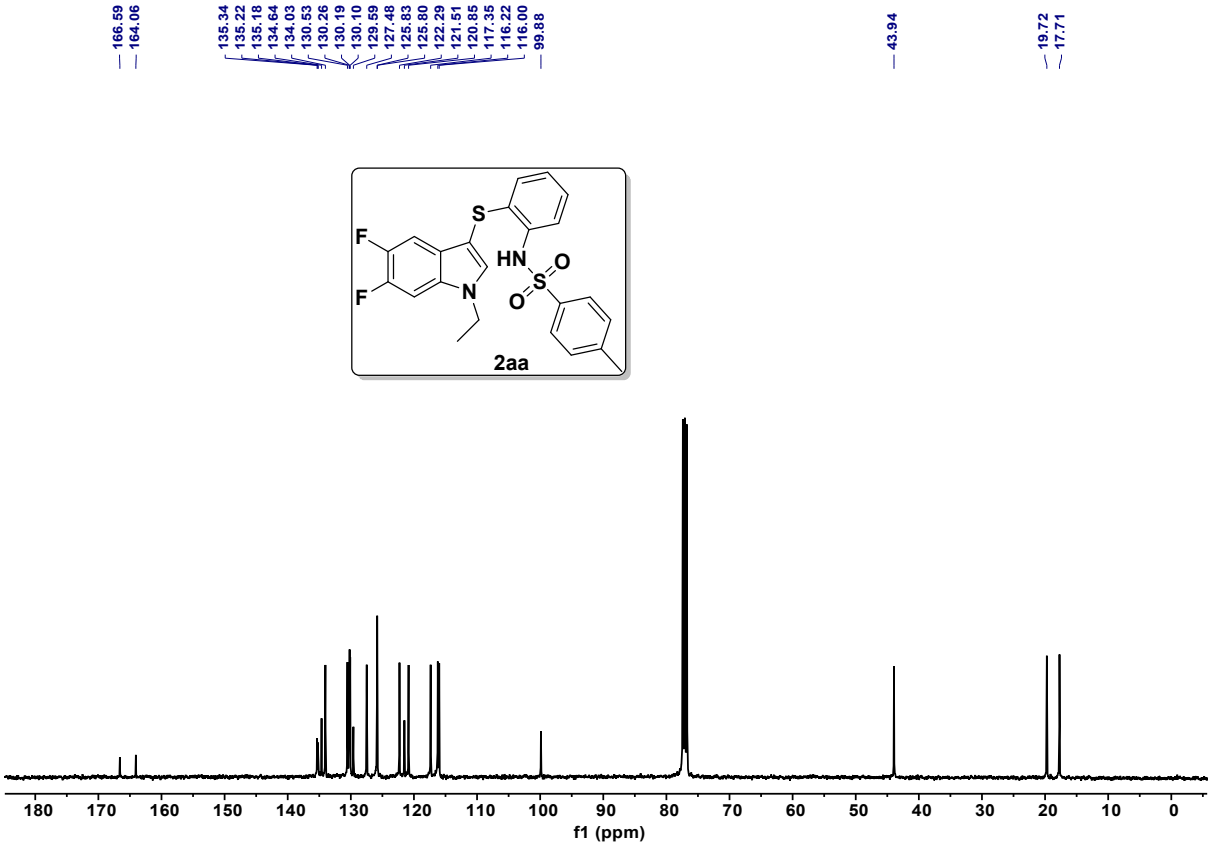


Figure S42. ^{13}C{^{1}H} NMR of compound **2aa** (100 MHz, $CDCl_3$)

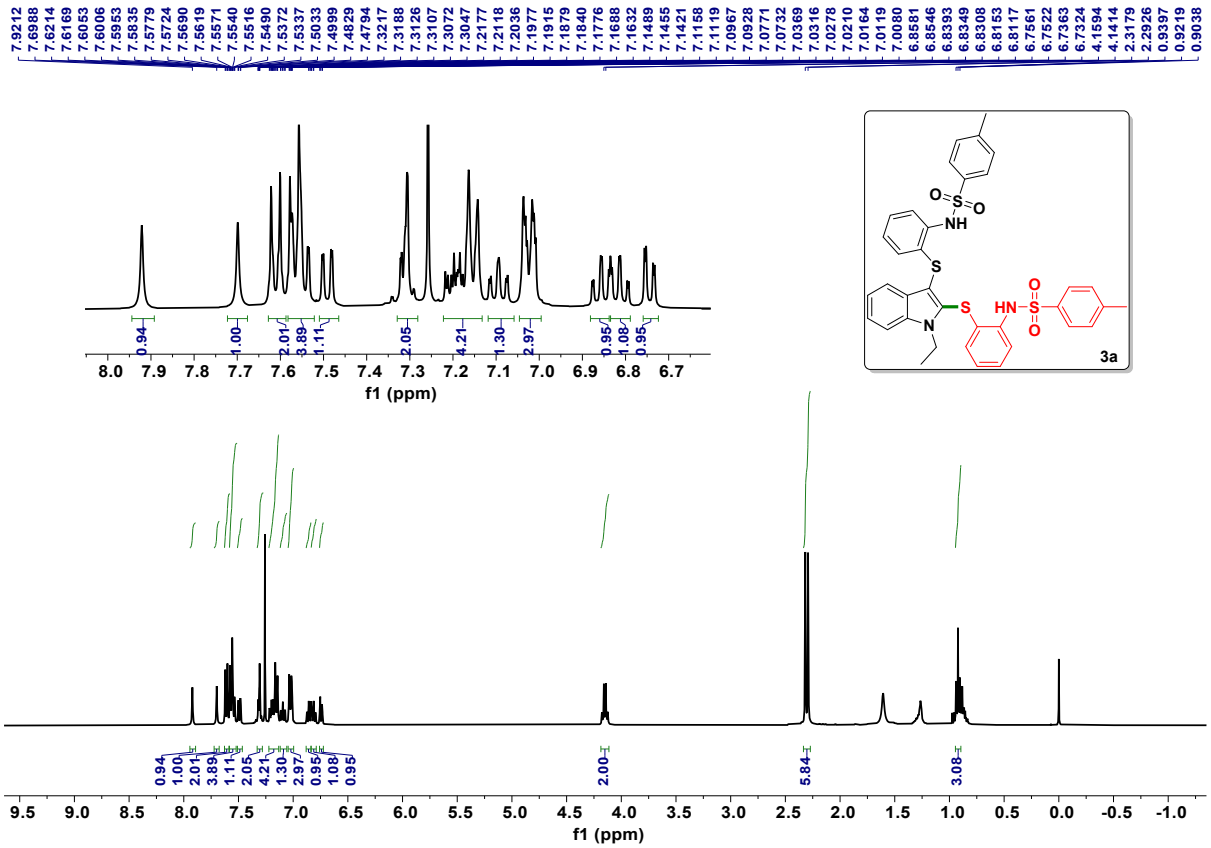


Figure S43. ^{1}H NMR of compound **3a** (400 MHz, $CDCl_3$)

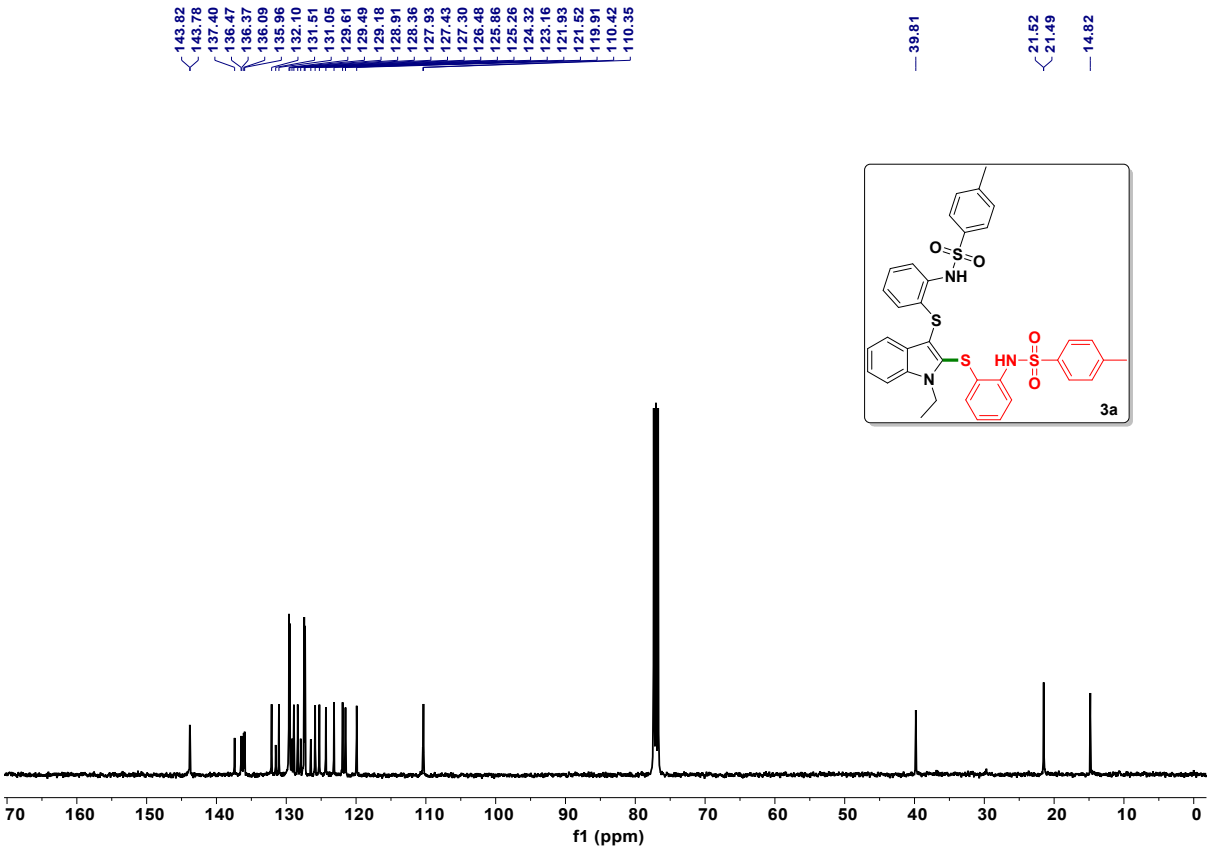


Figure S44. $^{13}C\{^{1}H\}$ NMR of compound **3a** (100 MHz, $CDCl_3$)

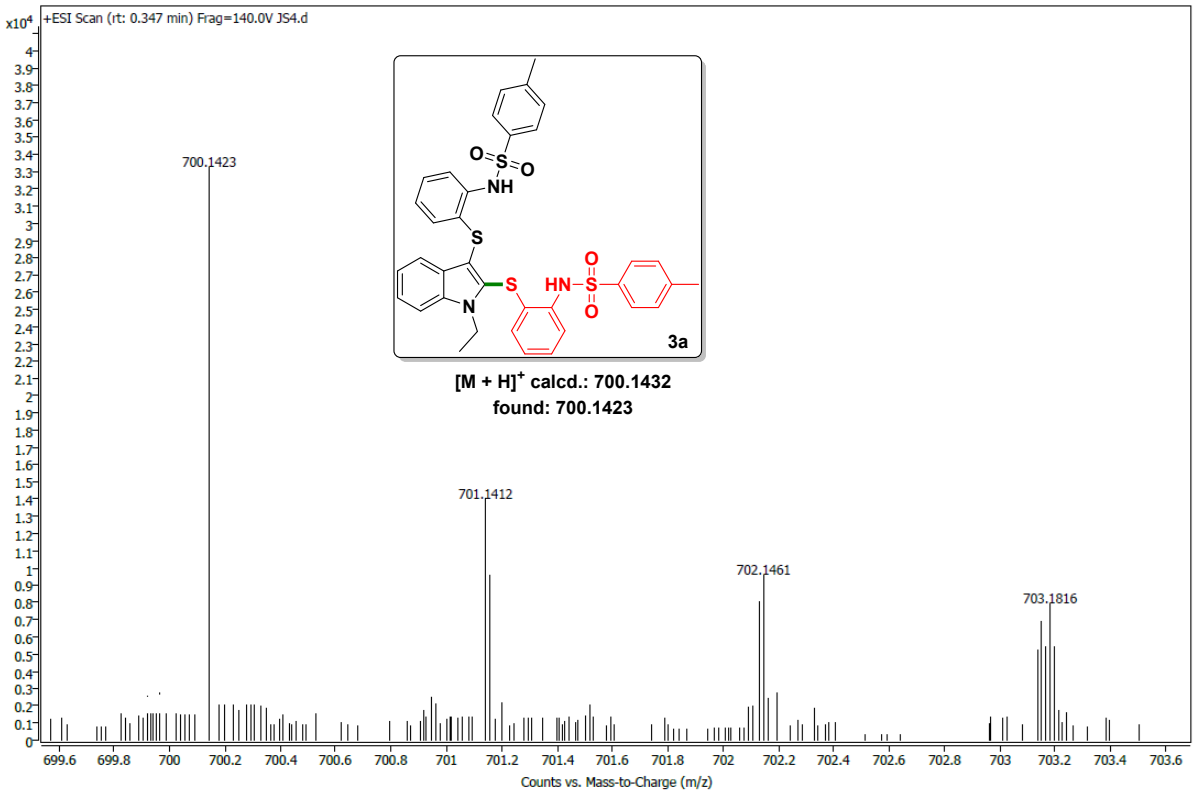


Figure S45. HRMS of compound **3a**

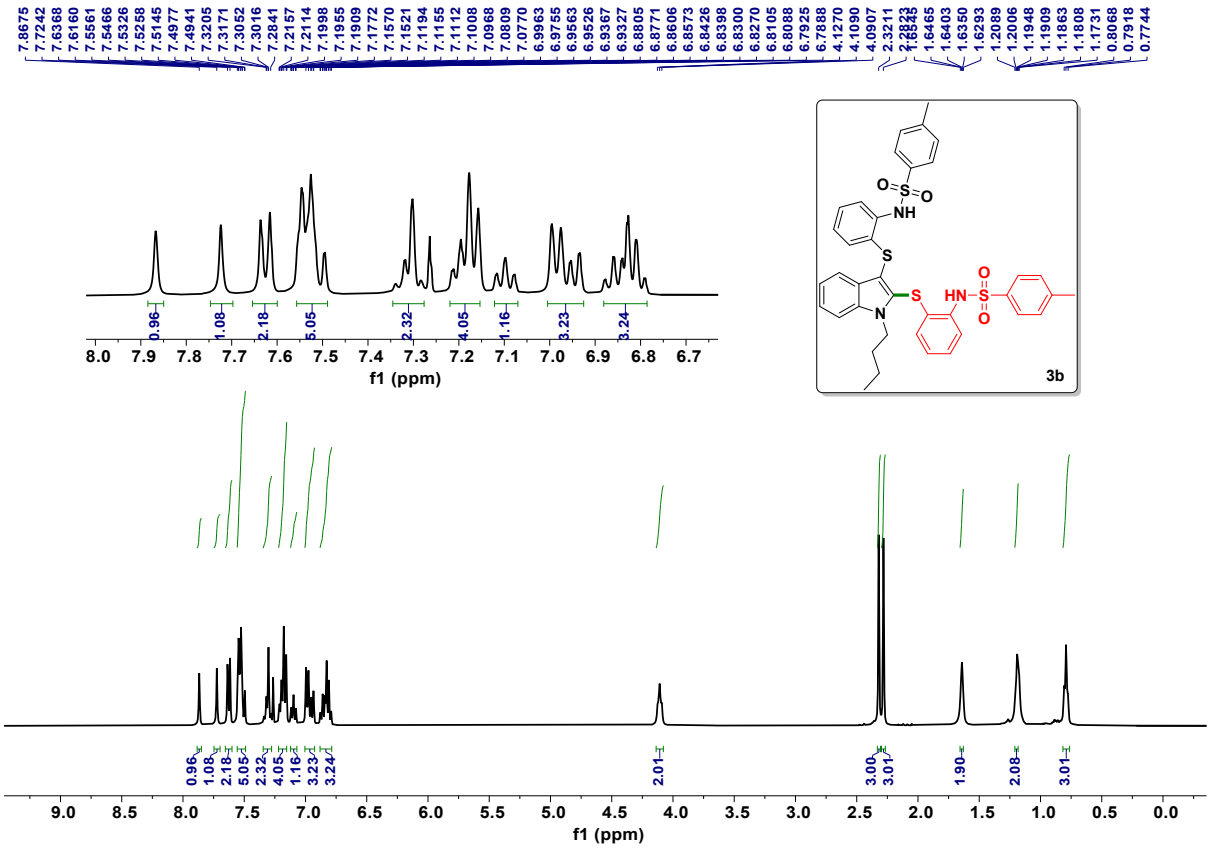


Figure S46. ^{1}H NMR of compound **3b** (400 MHz, $CDCl_3$)

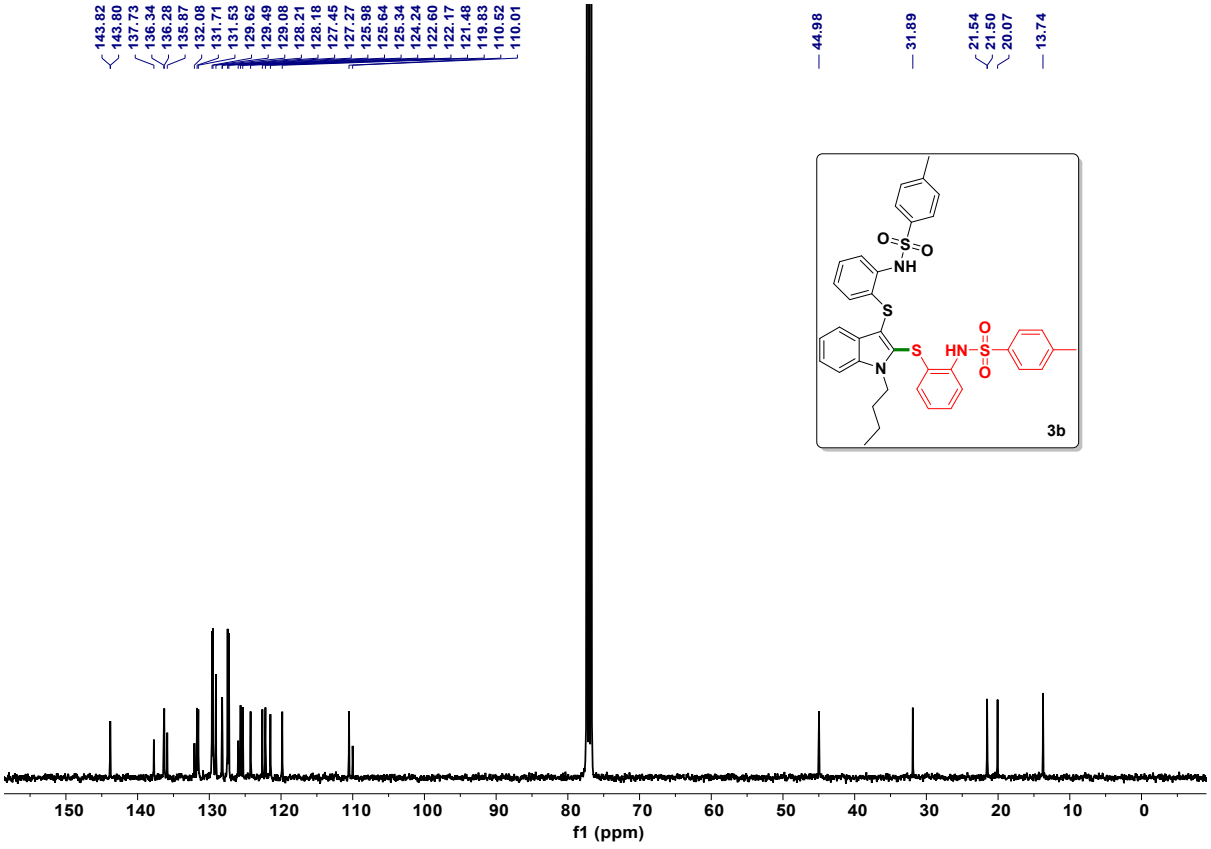


Figure S47. $^{13}C\{^{1}H\}$ NMR of compound **3b** (100 MHz, $CDCl_3$)

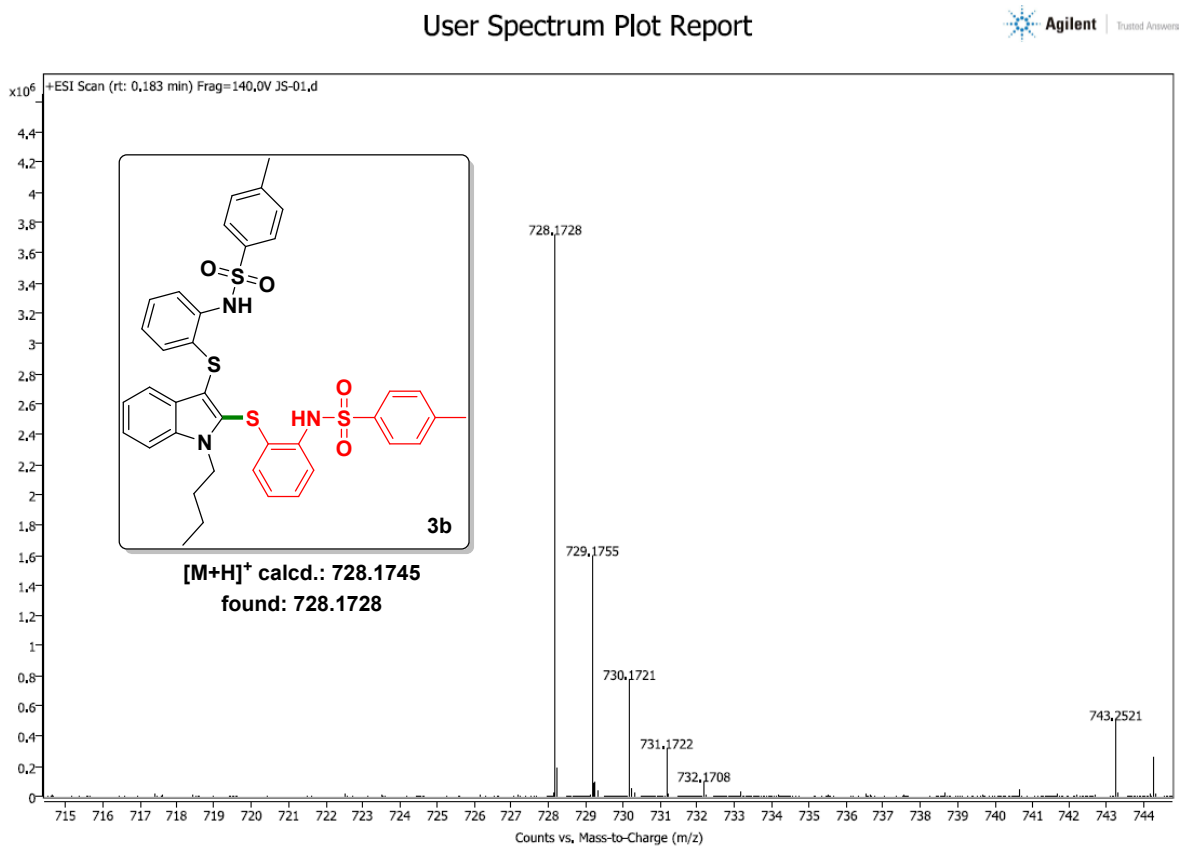


Figure S48. HRMS of compound **3b**

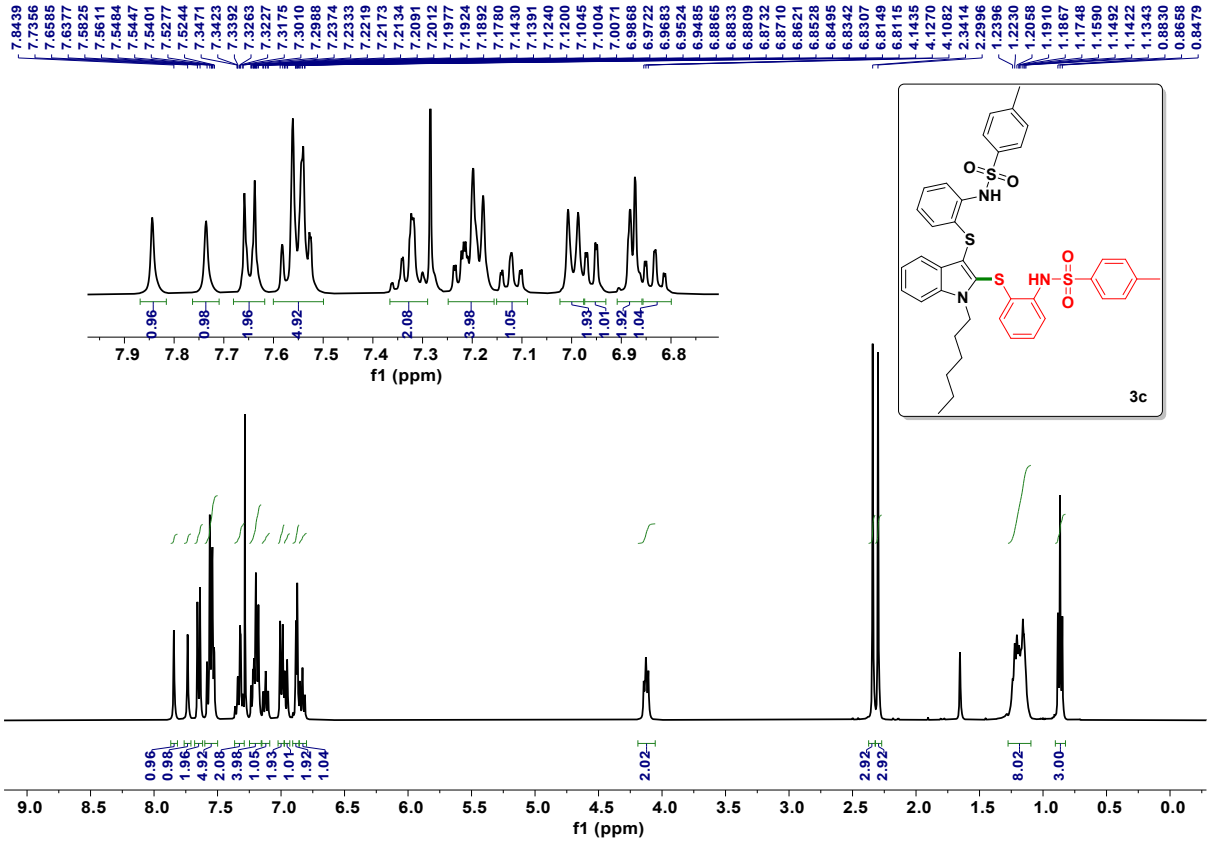


Figure S49. ^{1}H NMR of compound **3c** (400 MHz, $CDCl_3$)

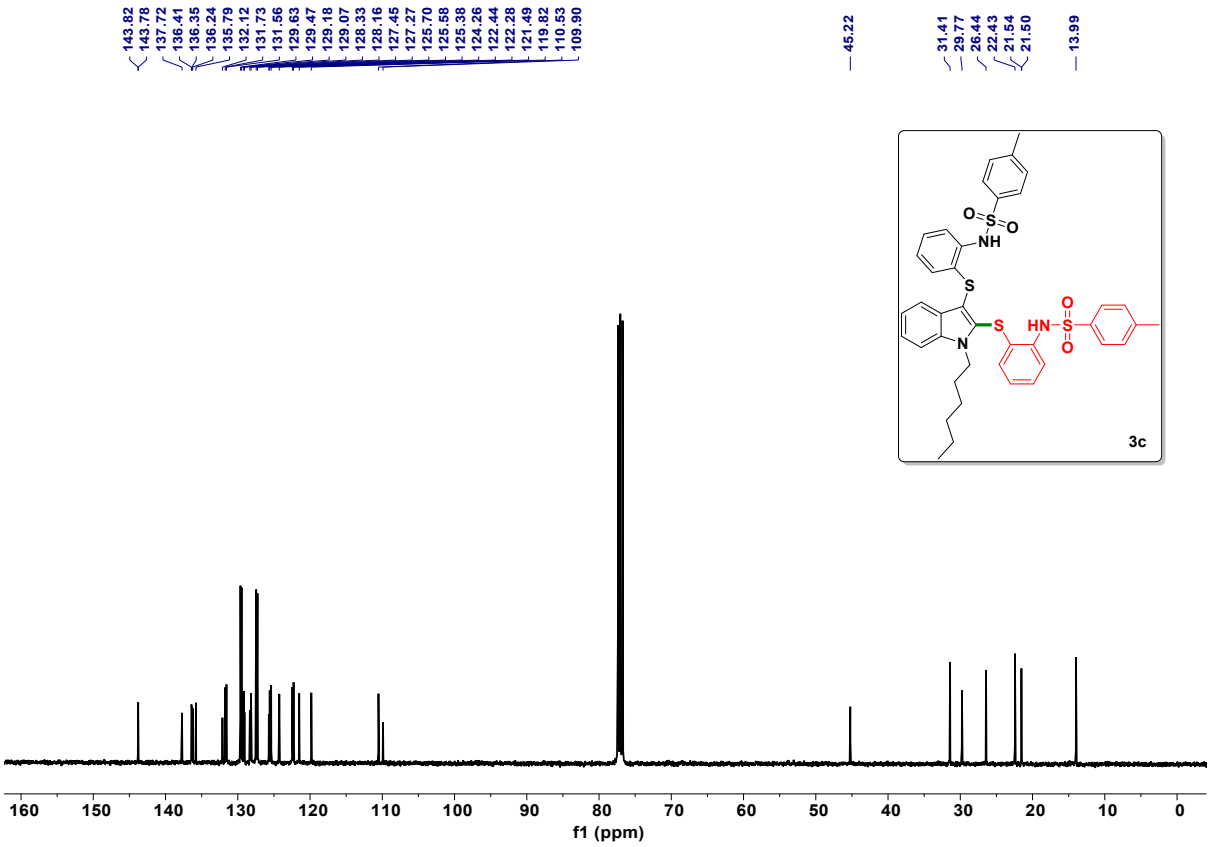


Figure S50. $^{13}C\{^{1}H\}$ NMR of compound **3c** (100 MHz, $CDCl_3$)

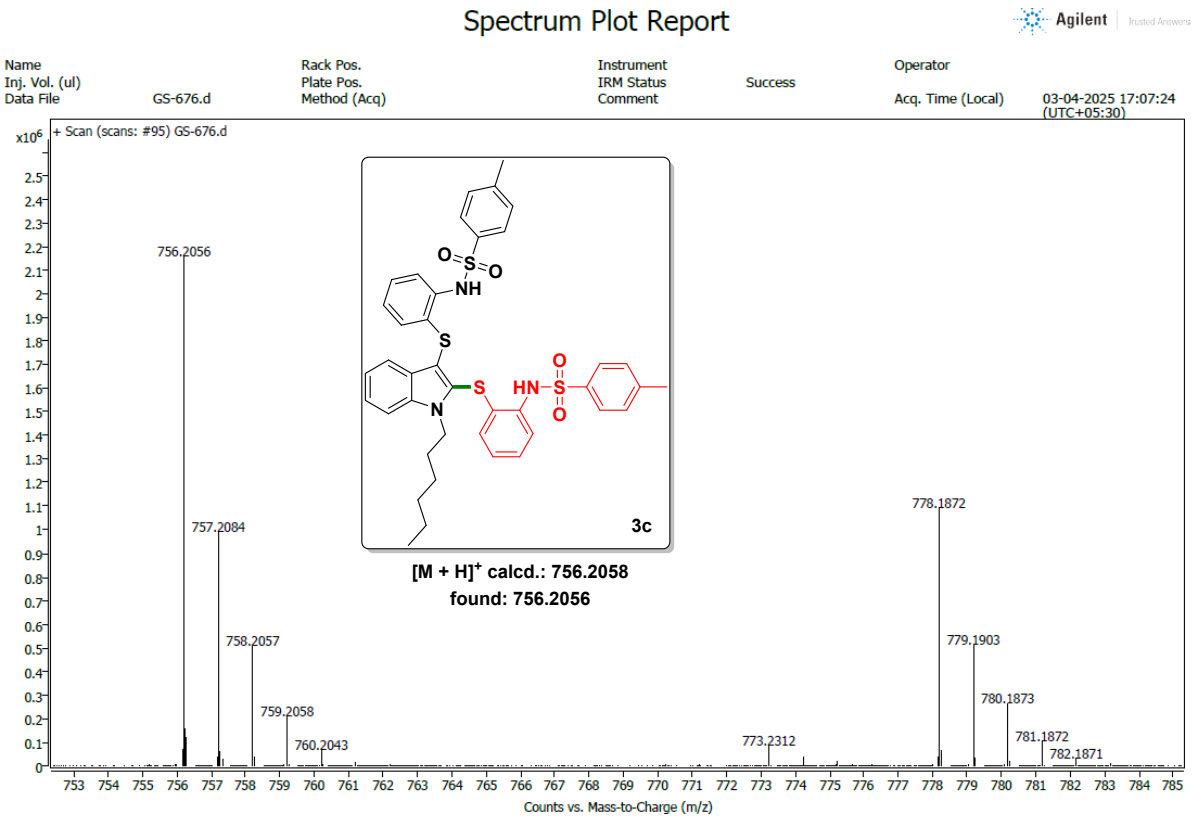


Figure S51. HRMS of compound **3c**

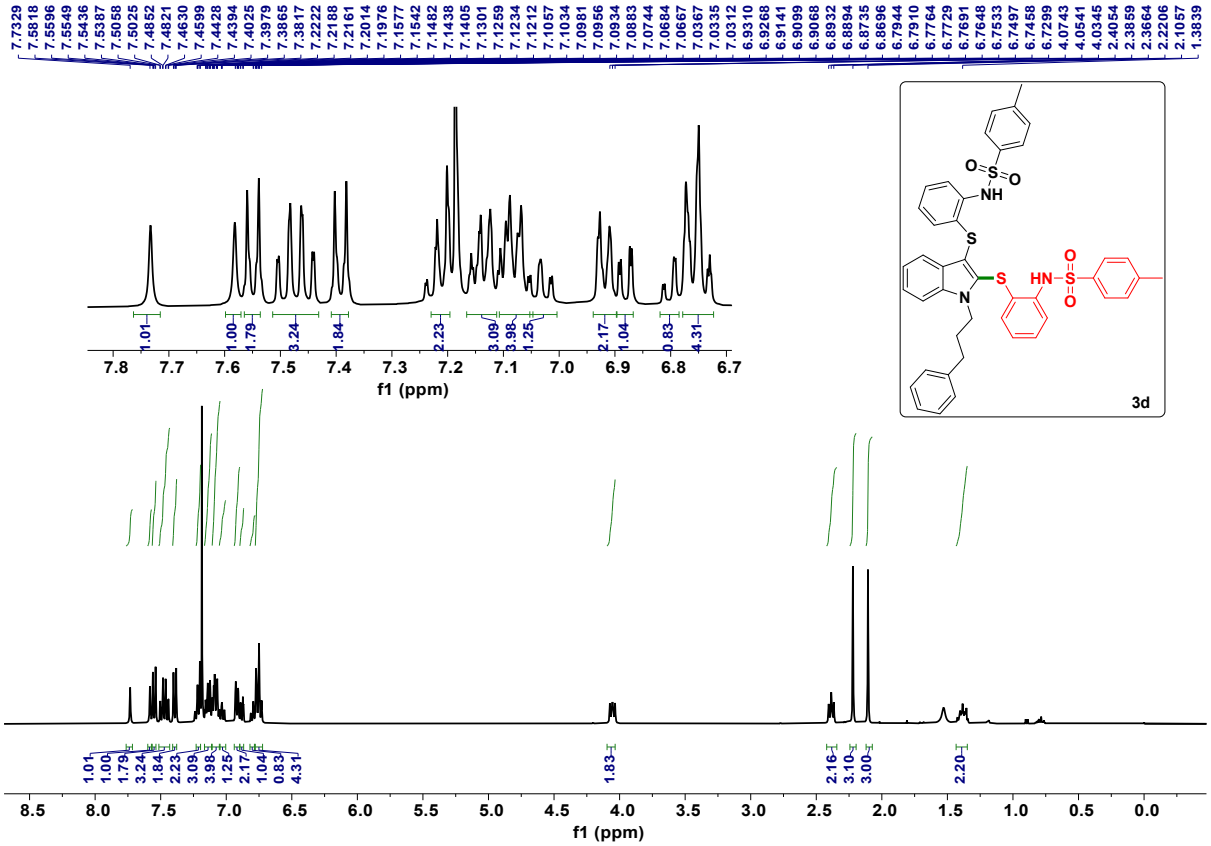


Figure S52. ^{1}H NMR of compound **3d** (400 MHz, $CDCl_3$)

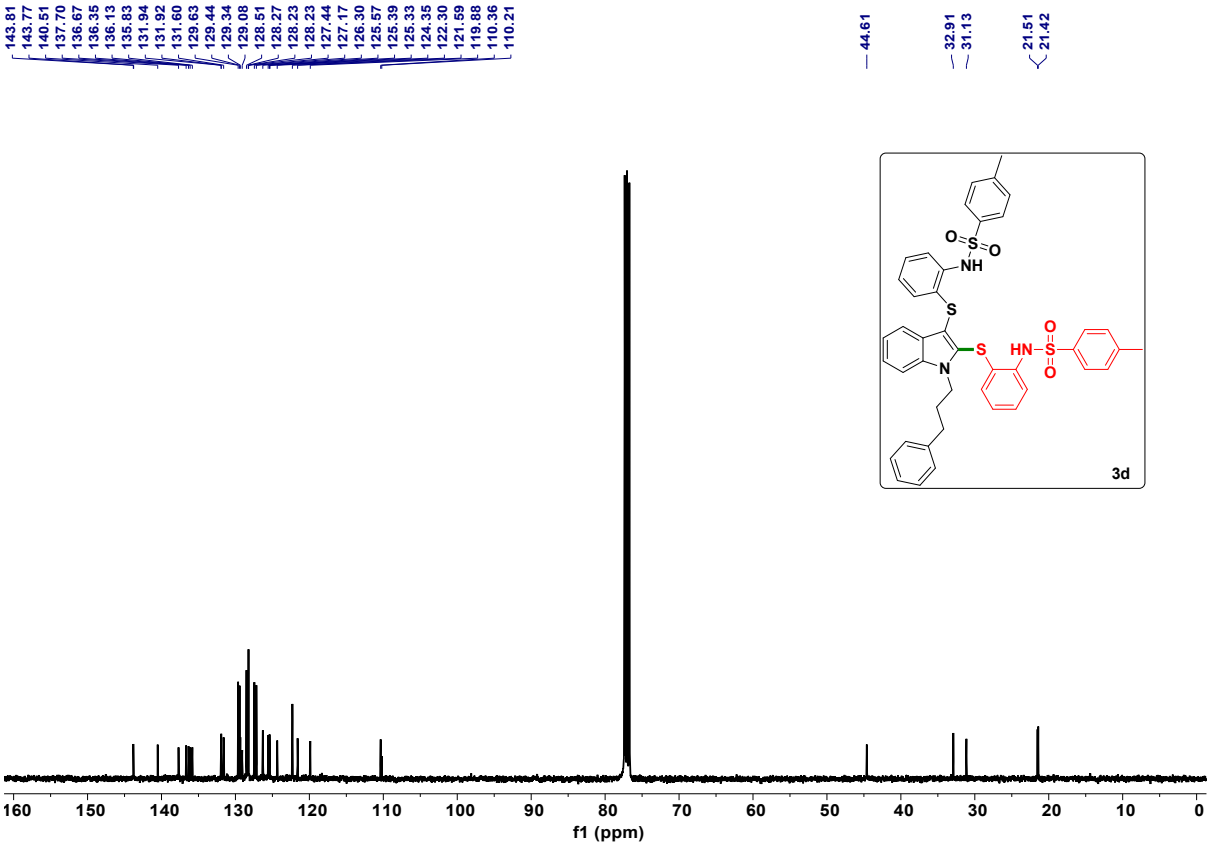


Figure S53. $^{13}C\{^{1}H\}$ NMR of compound **3d** (100 MHz, $CDCl_3$)

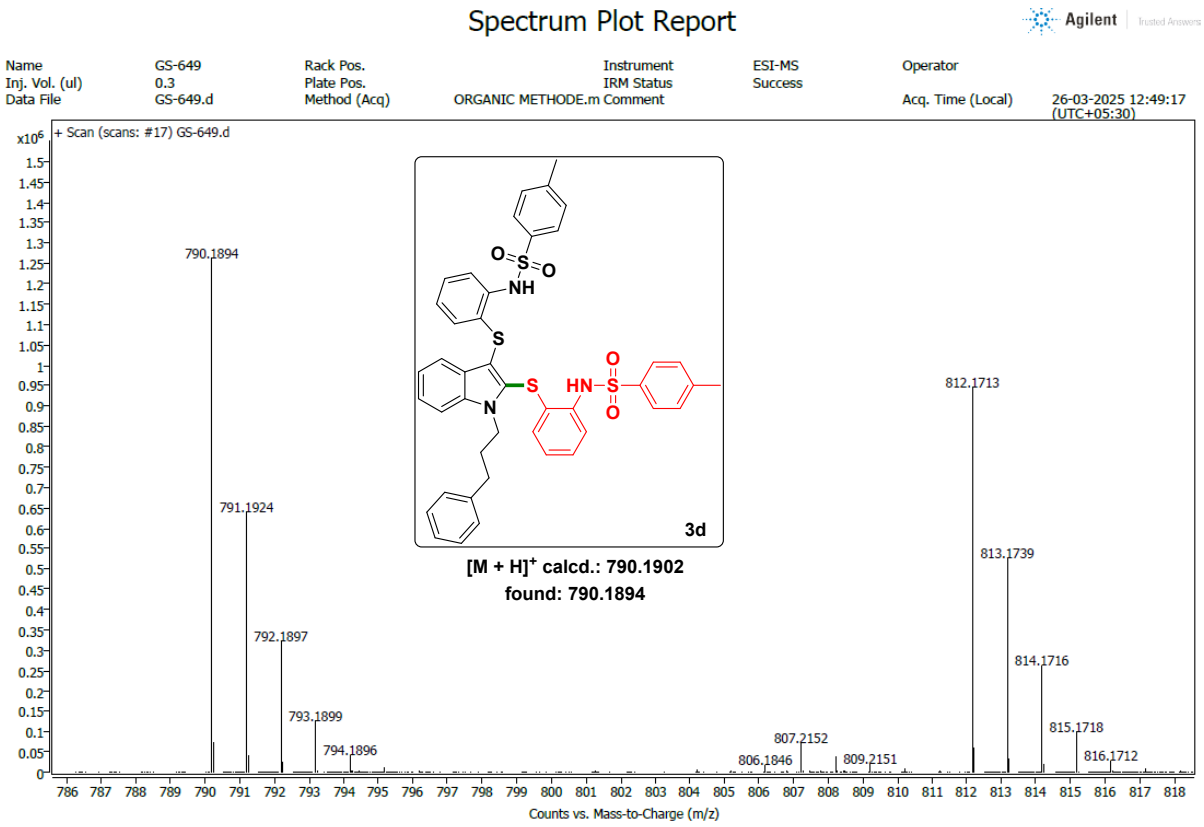


Figure S54. HRMS of compound **3d**

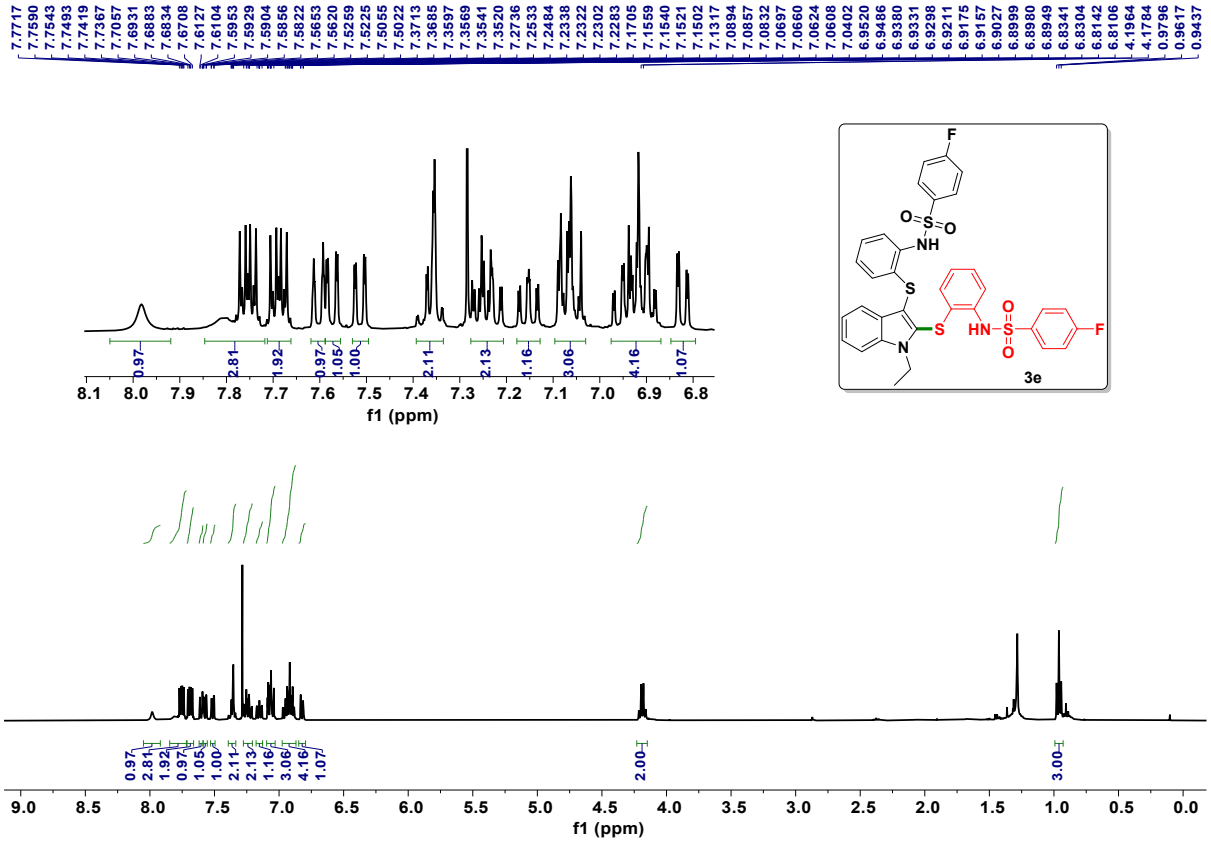


Figure S55. ^{1}H NMR of compound **3e** (400 MHz, $CDCl_3$)

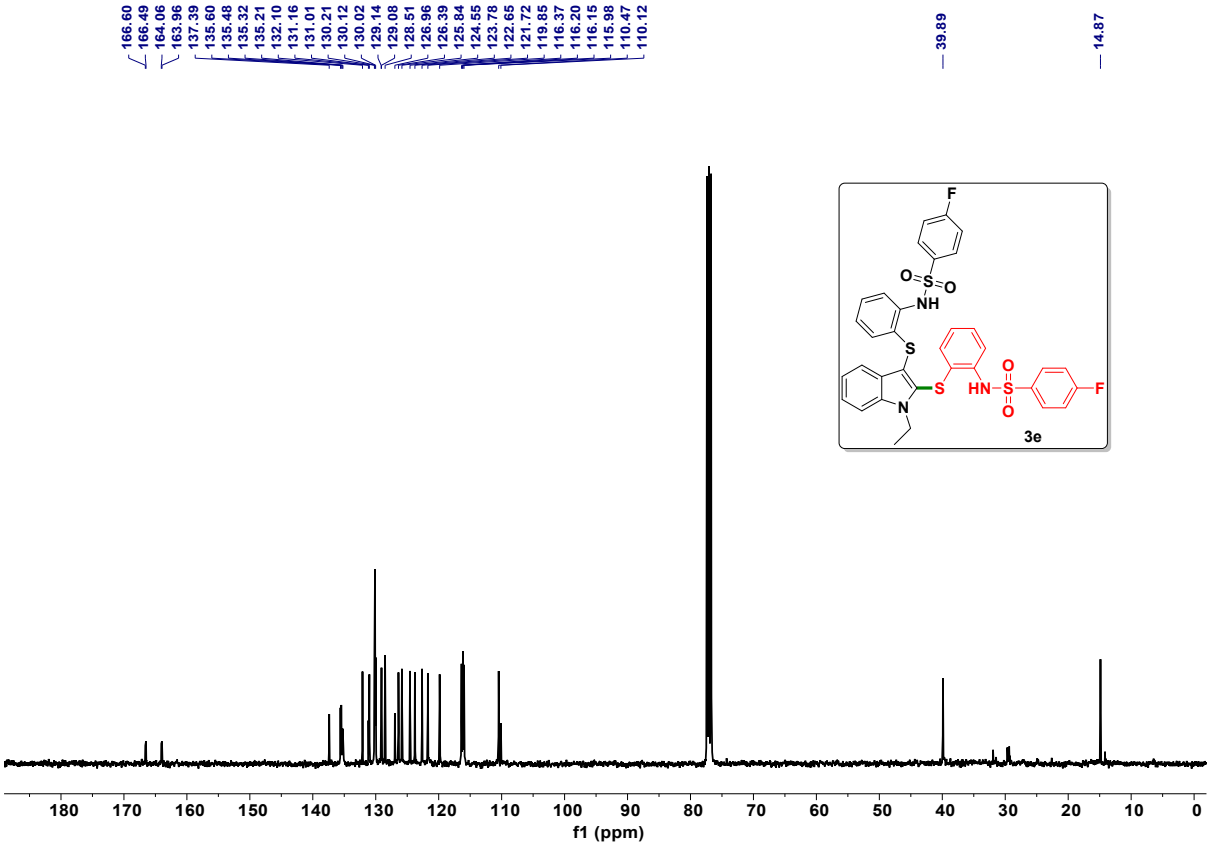


Figure S56. $^{13}C\{^{1}H\}$ NMR of compound **3e** (100 MHz, $CDCl_3$)

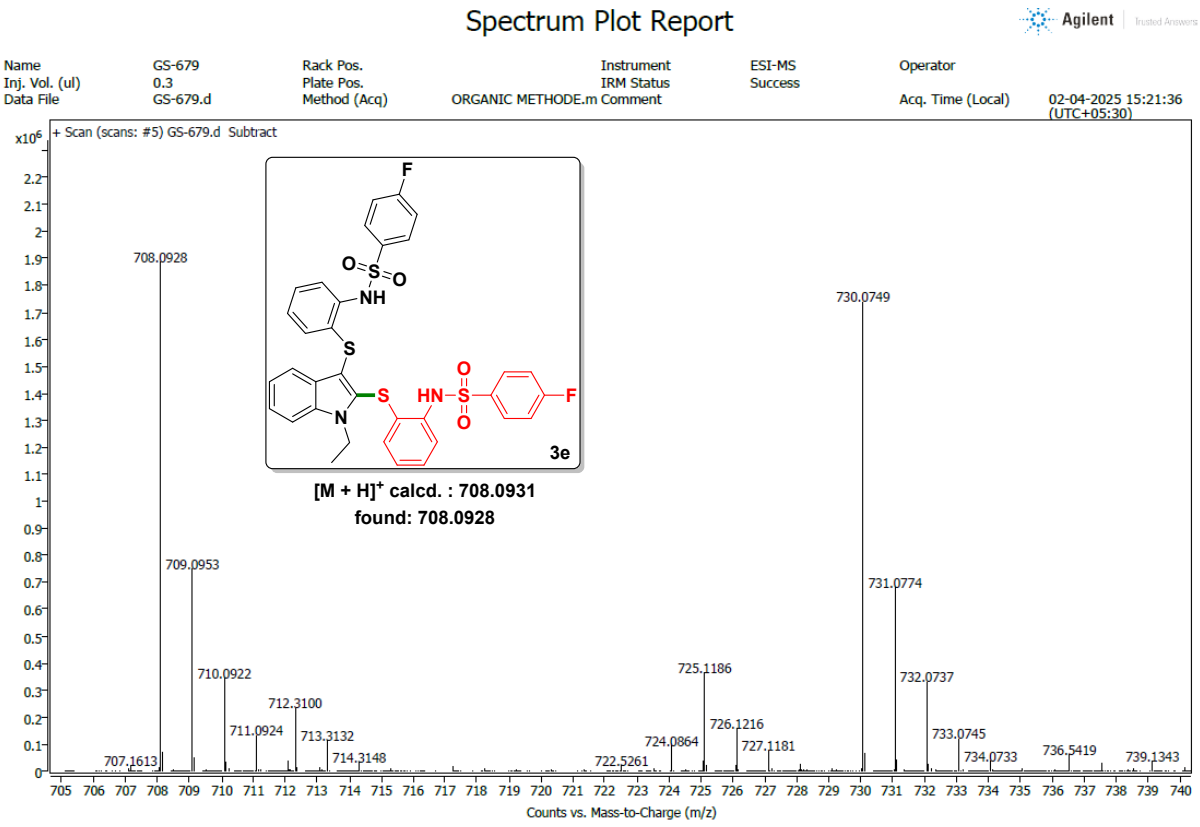


Figure S57. HRMS of compound **3e**

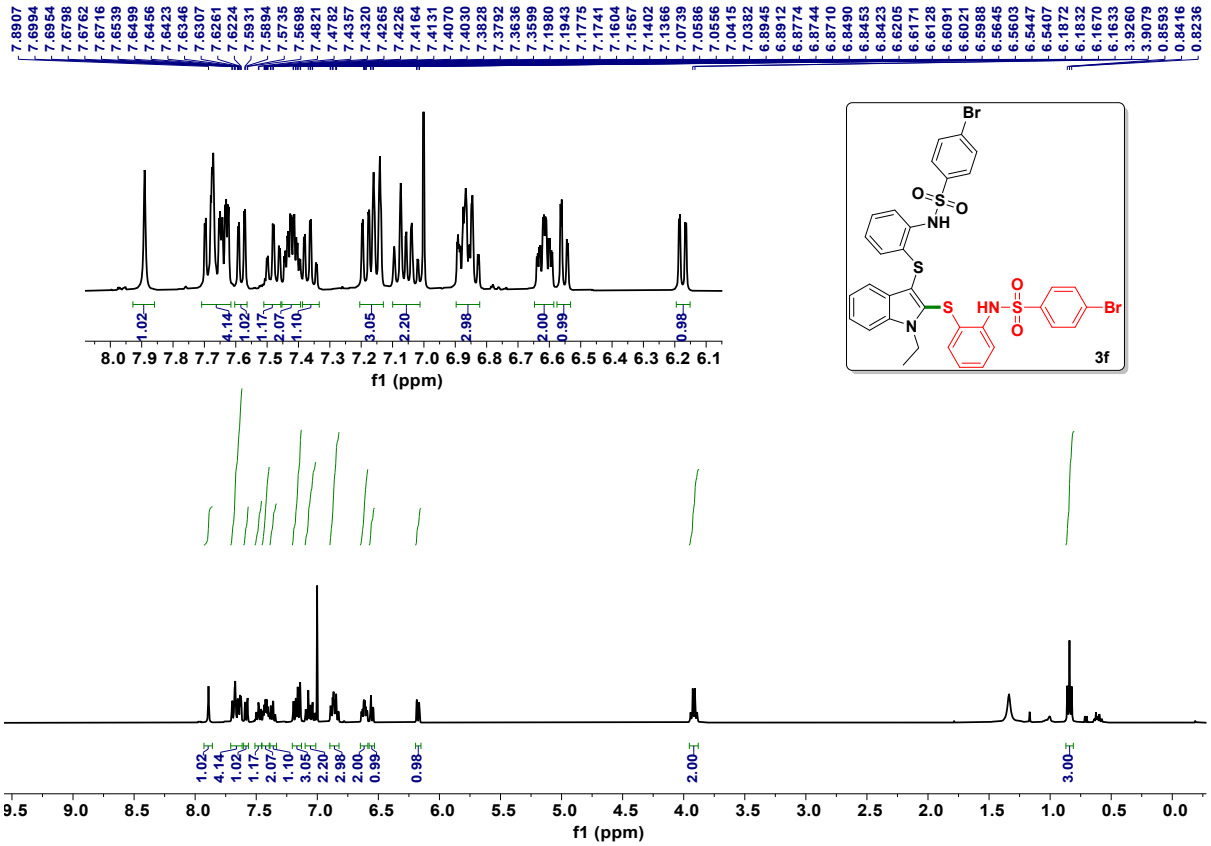


Figure S58. ^{1}H NMR of compound **3f** (400 MHz, $CDCl_3$)

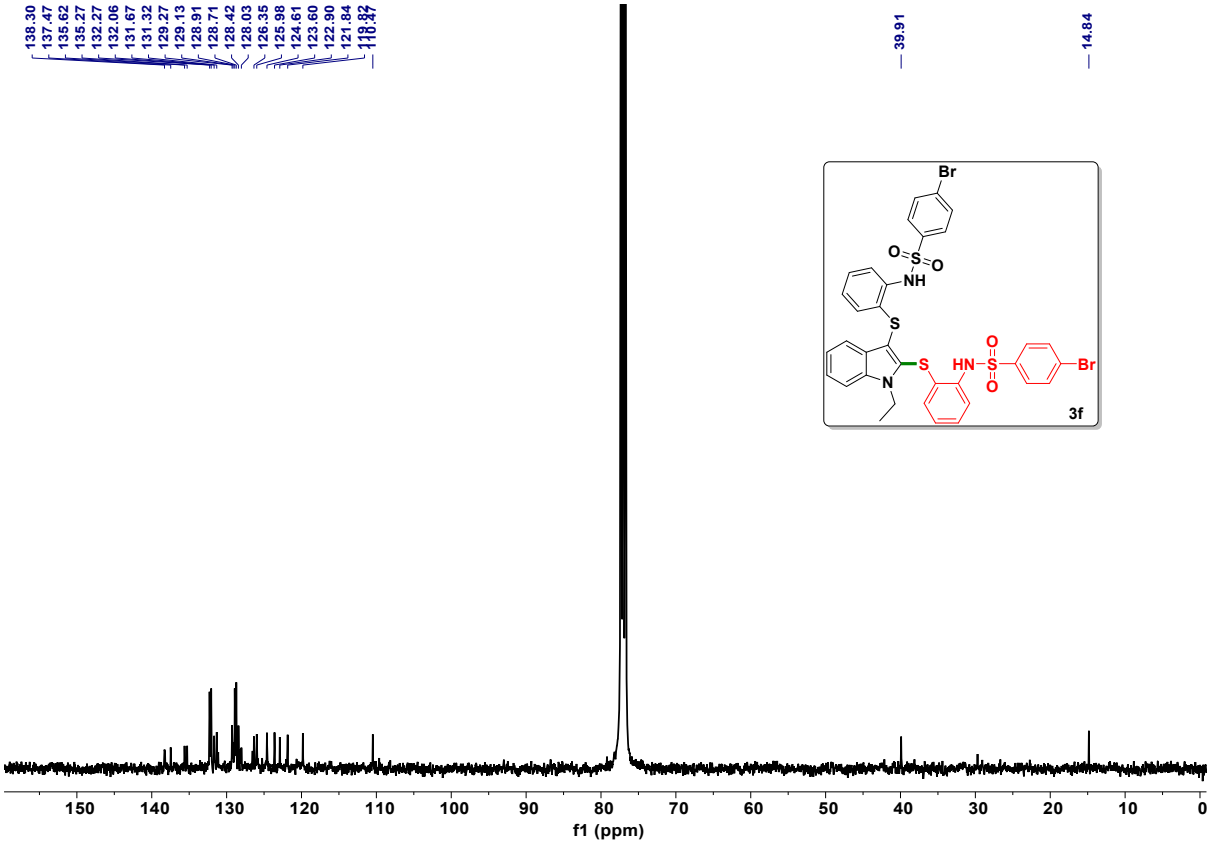


Figure S59. $^{13}C\{^{1}H\}$ NMR of compound **3f** (100 MHz, $CDCl_3$)

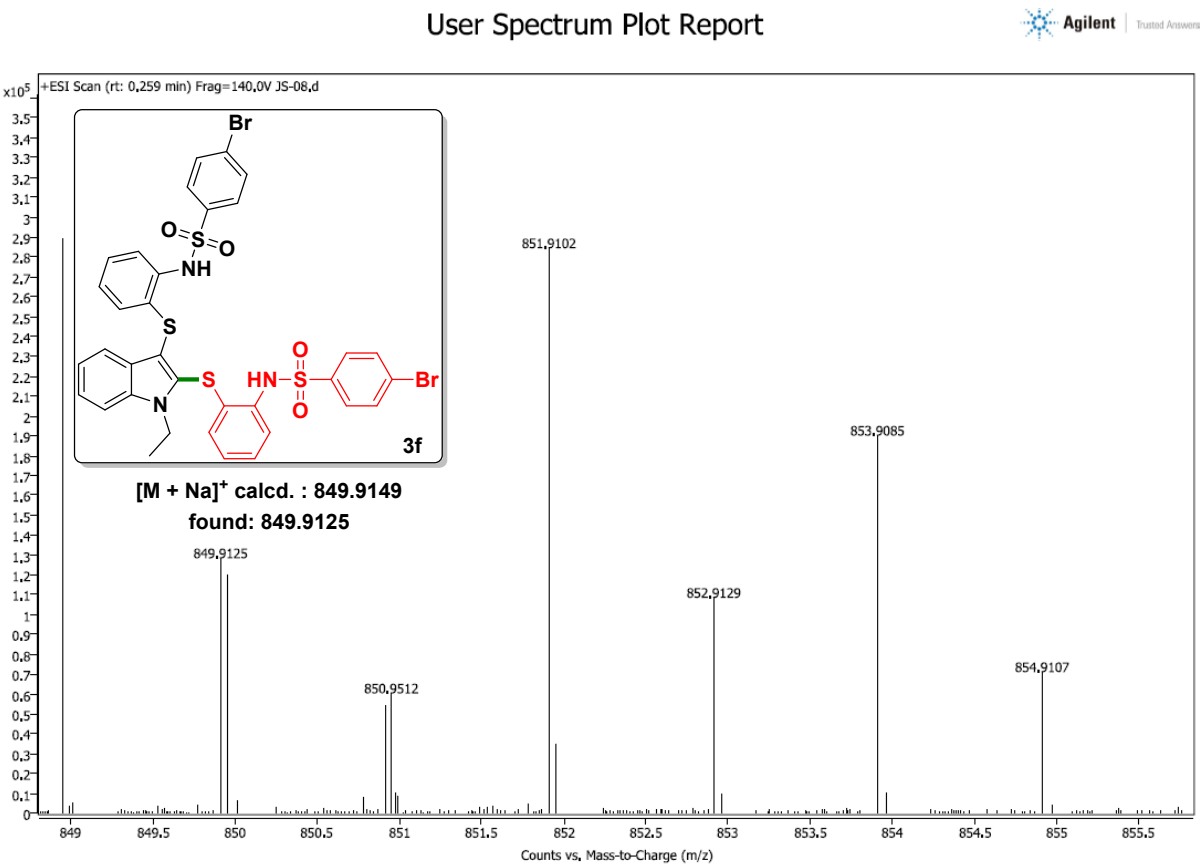


Figure S60. HRMS of compound **3f**

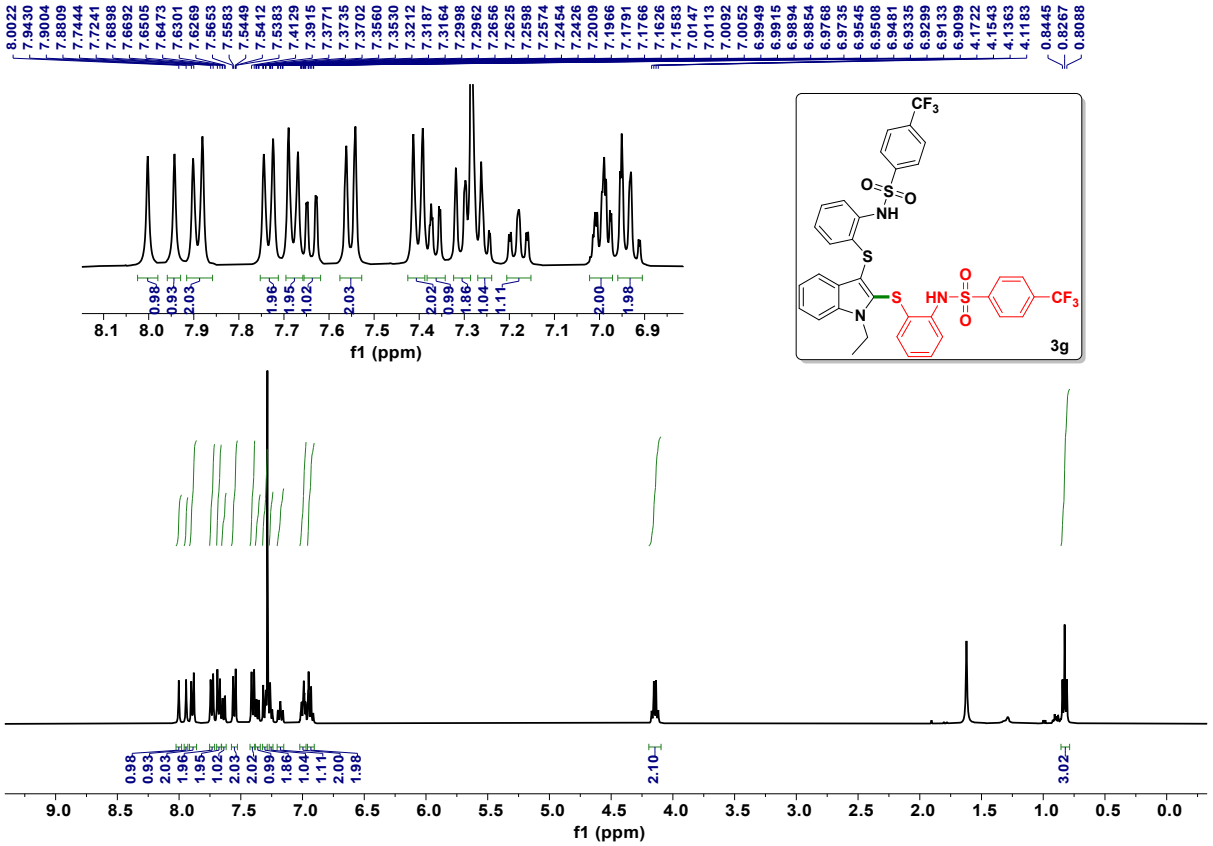


Figure S61. ^{1}H NMR of compound **3g** (400 MHz, $CDCl_3$)

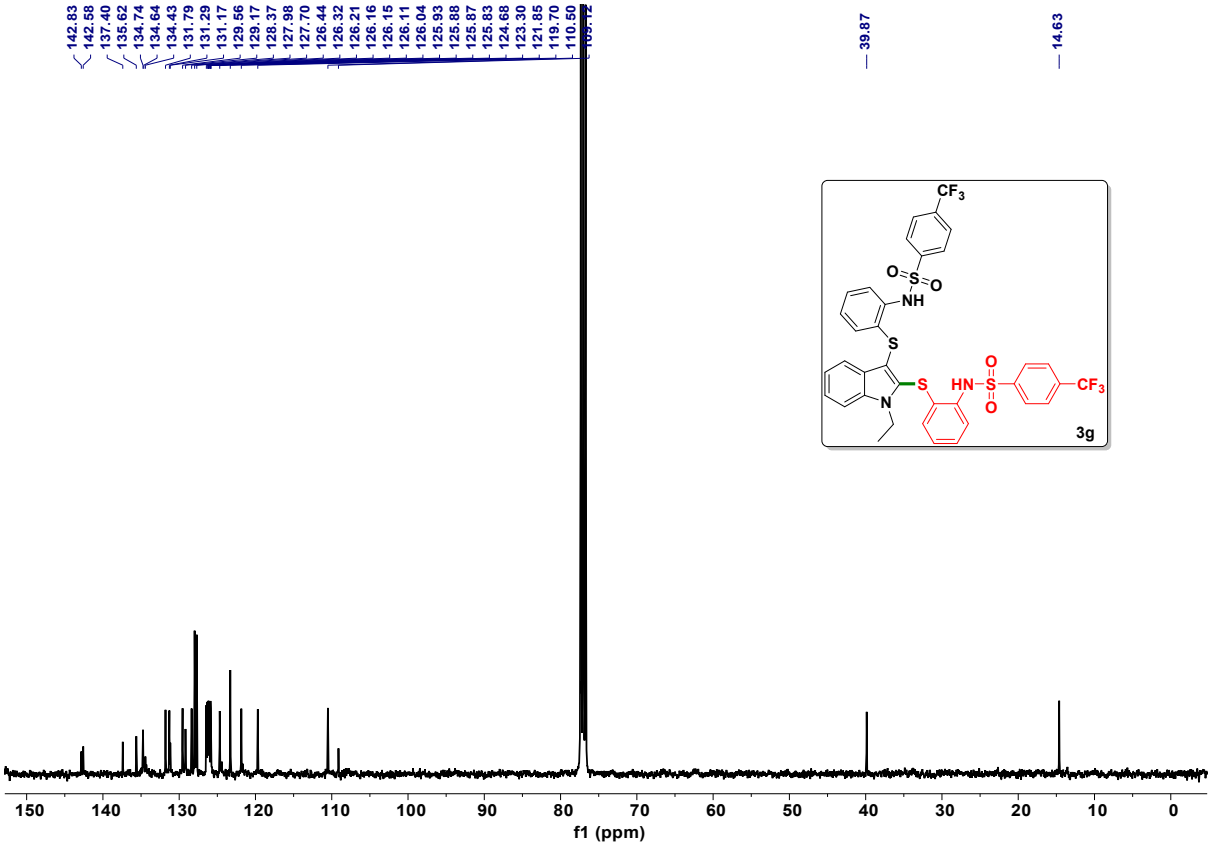


Figure S62. $^{13}C\{^{1}H\}$ NMR of compound **3g** (100 MHz, $CDCl_3$)

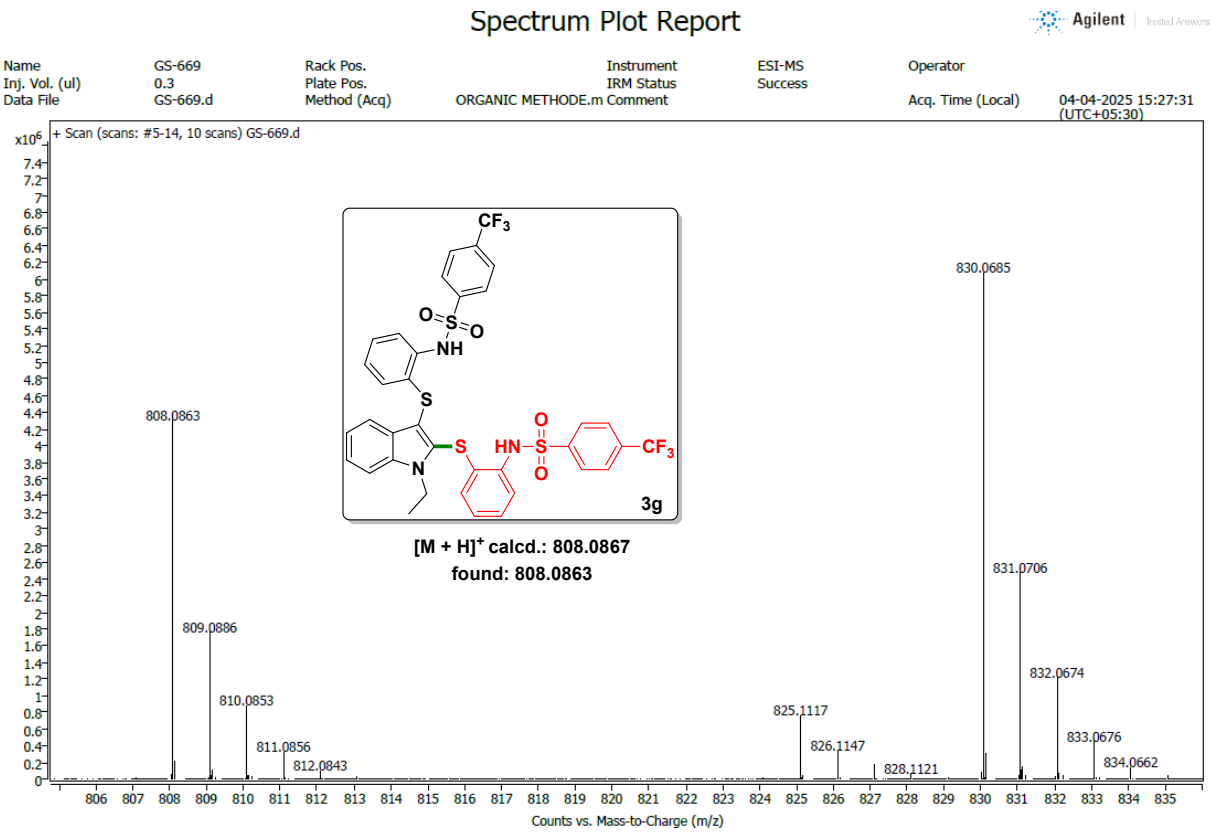


Figure S63. HRMS of compound **3g**

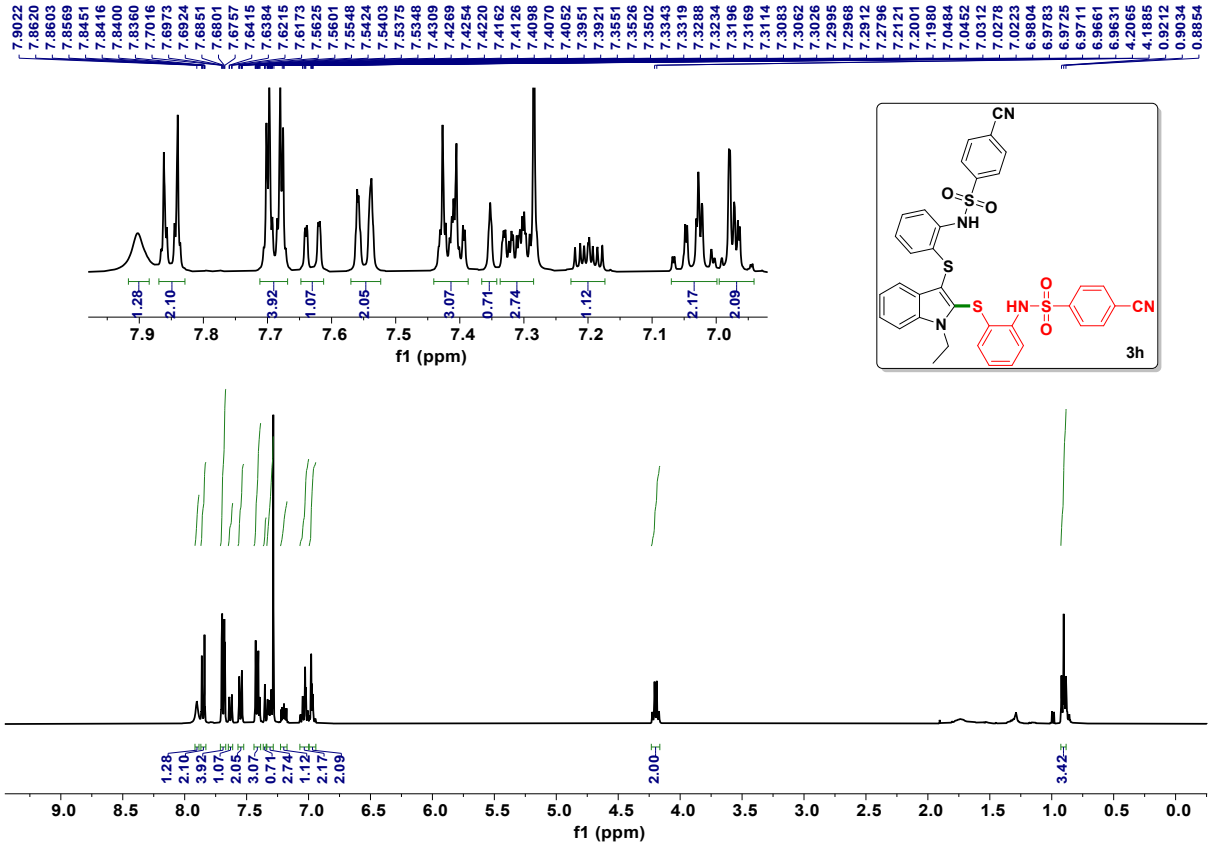


Figure S64. ^{1}H NMR of compound **3h** (400 MHz, $CDCl_3$)

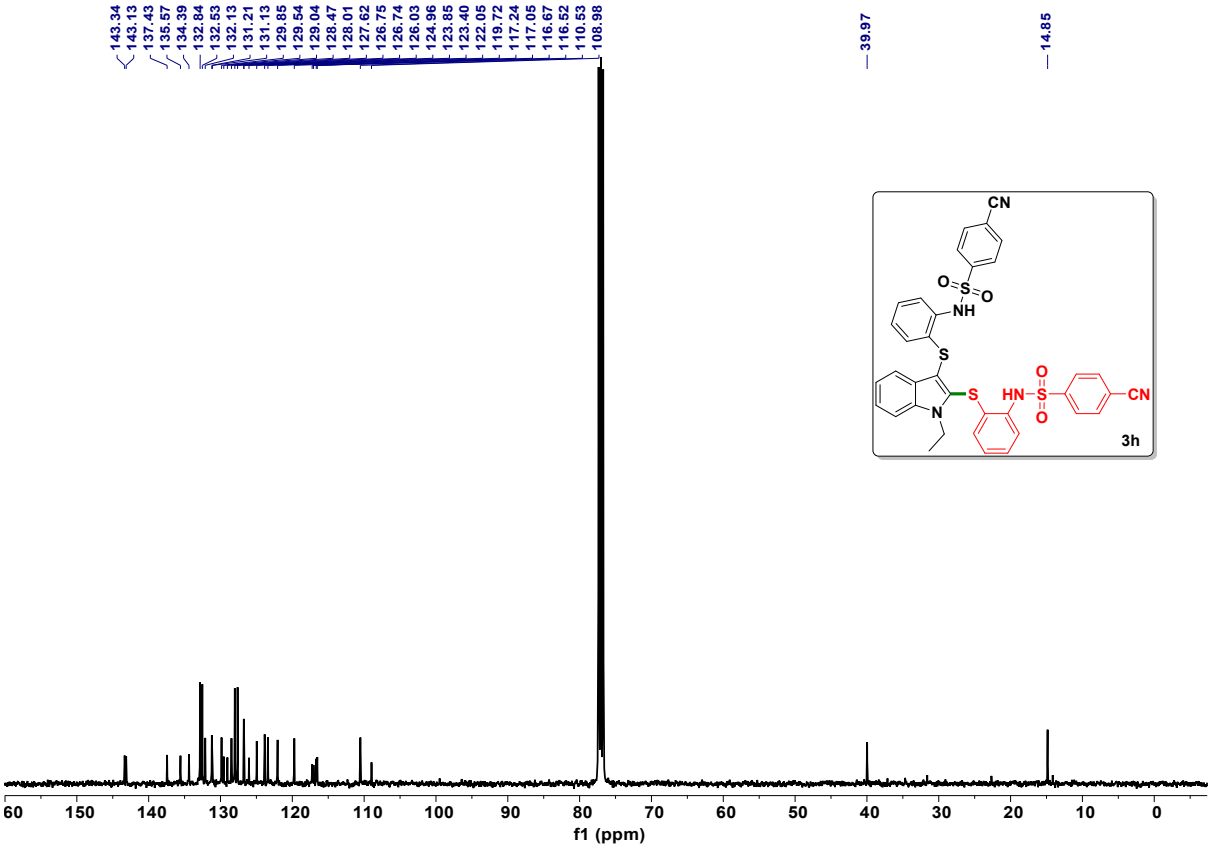


Figure S65. $^{13}C\{^{1}H\}$ NMR of compound **3h** (100 MHz, $CDCl_3$)

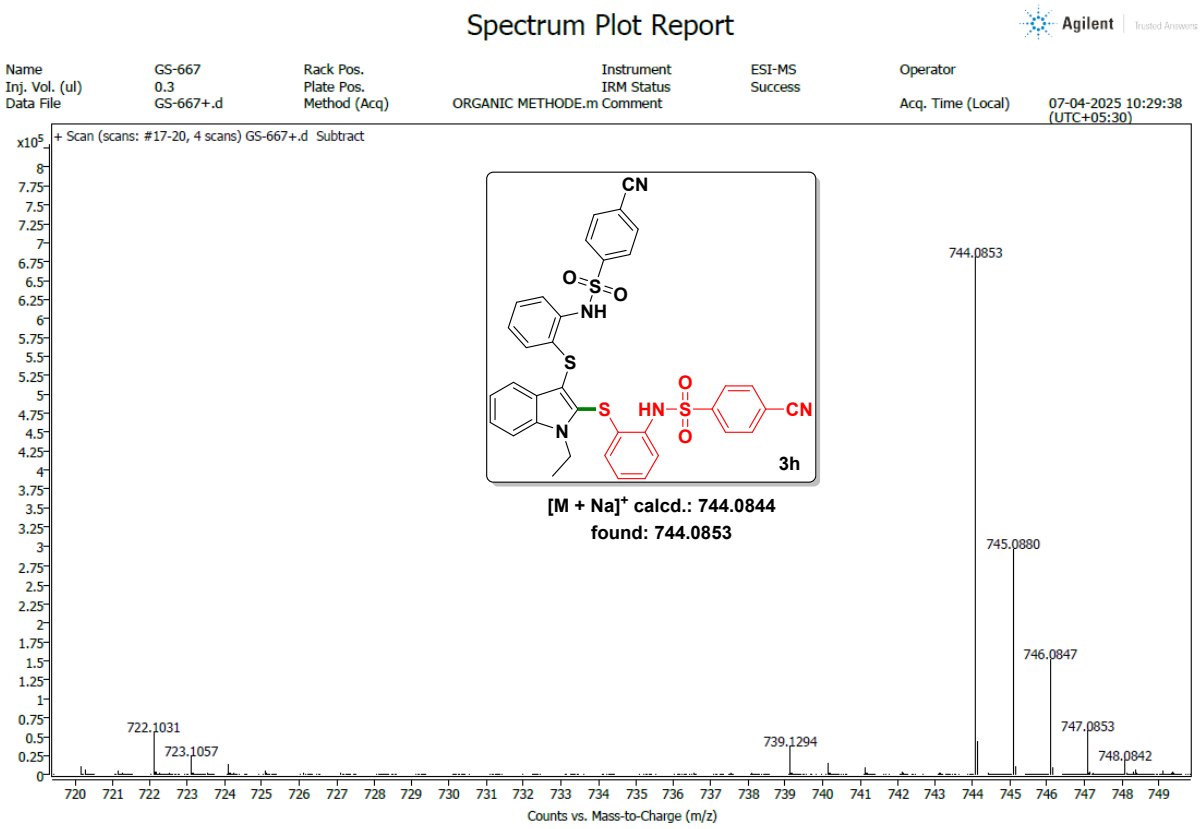


Figure S66. HRMS of compound **3h**

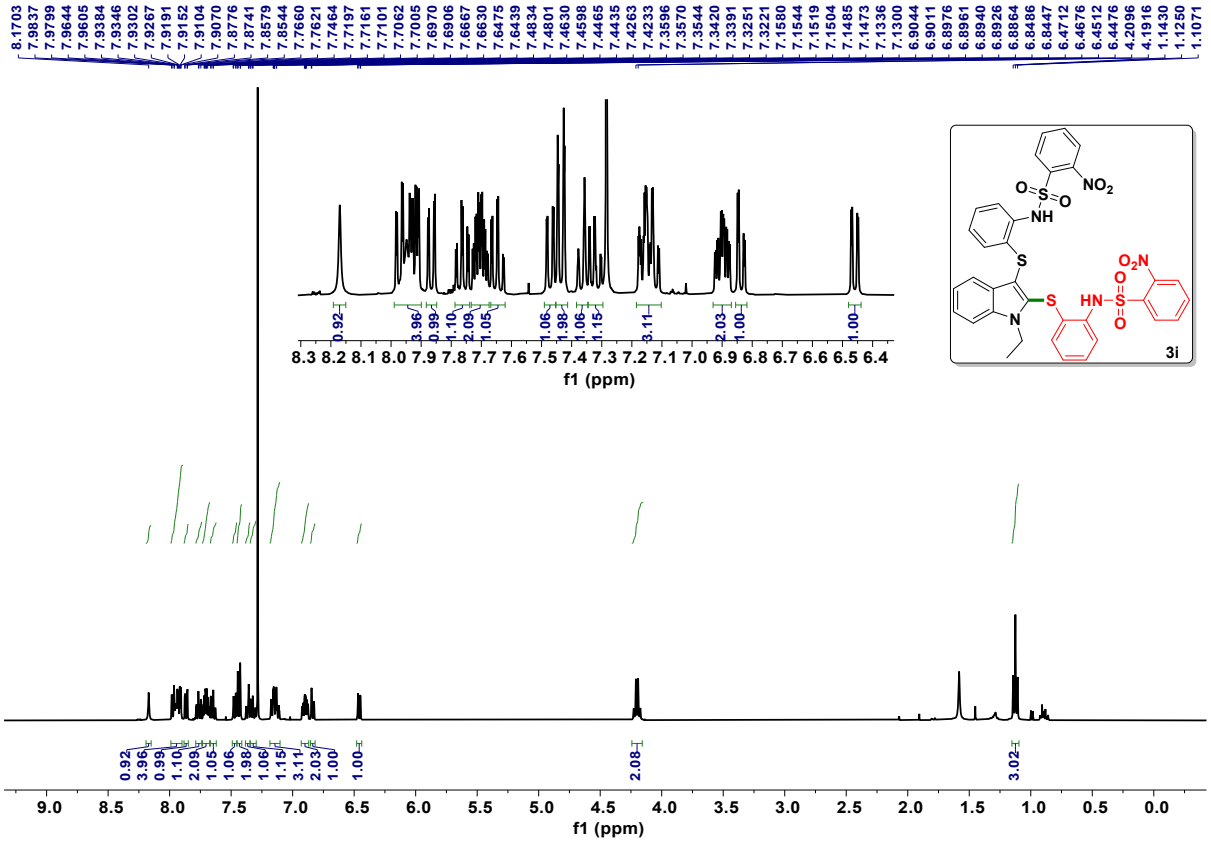


Figure S67. ^{1}H NMR of compound **3i** (400 MHz, $CDCl_3$)

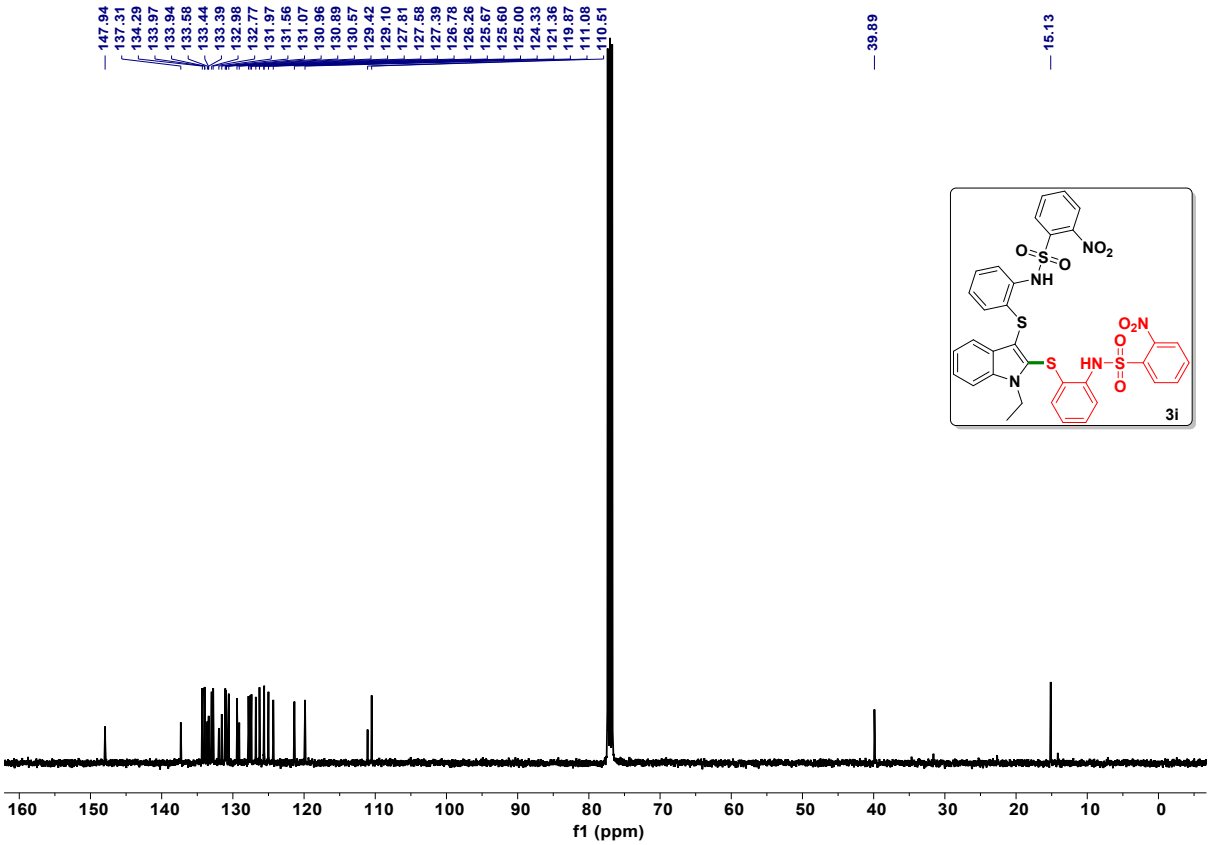


Figure S68. $^{13}C\{^{1}H\}$ NMR of compound **3i** (100 MHz, $CDCl_3$)

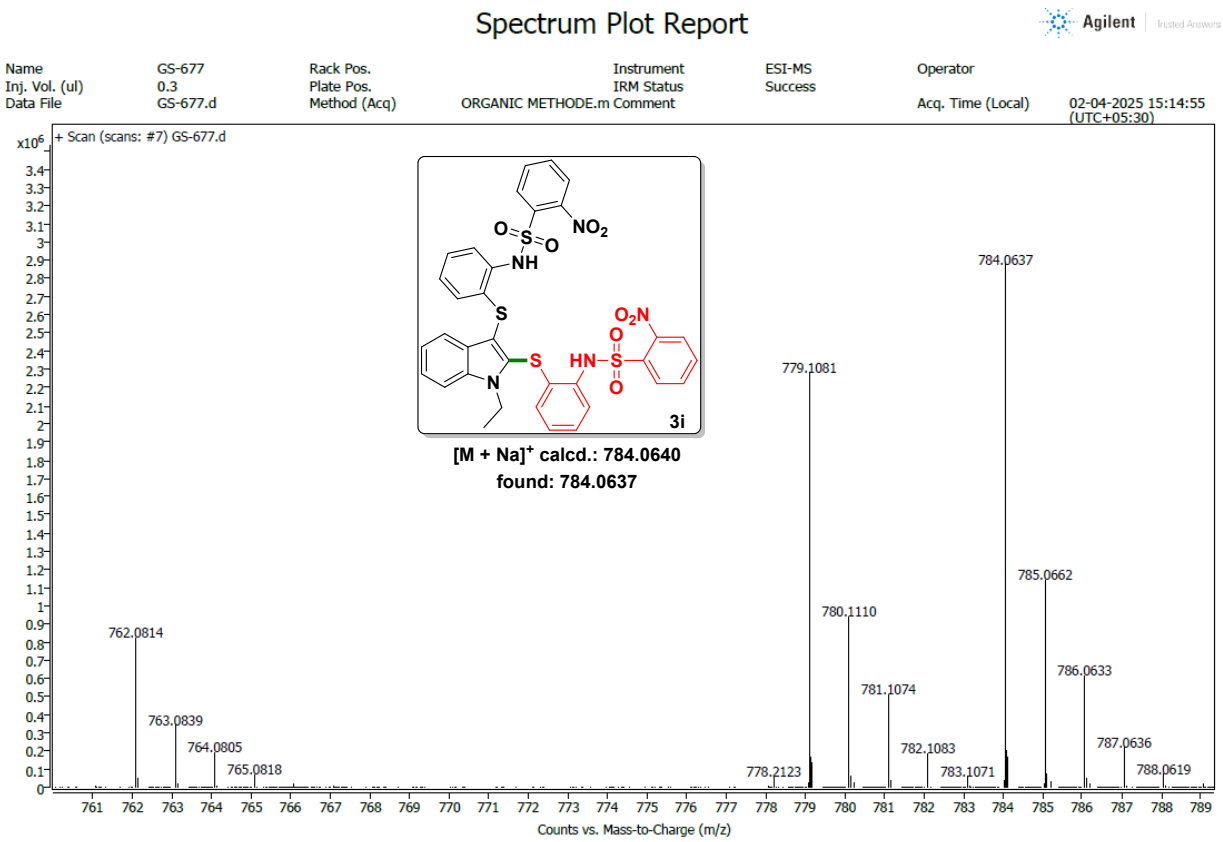


Figure S69. HRMS of compound **3i**

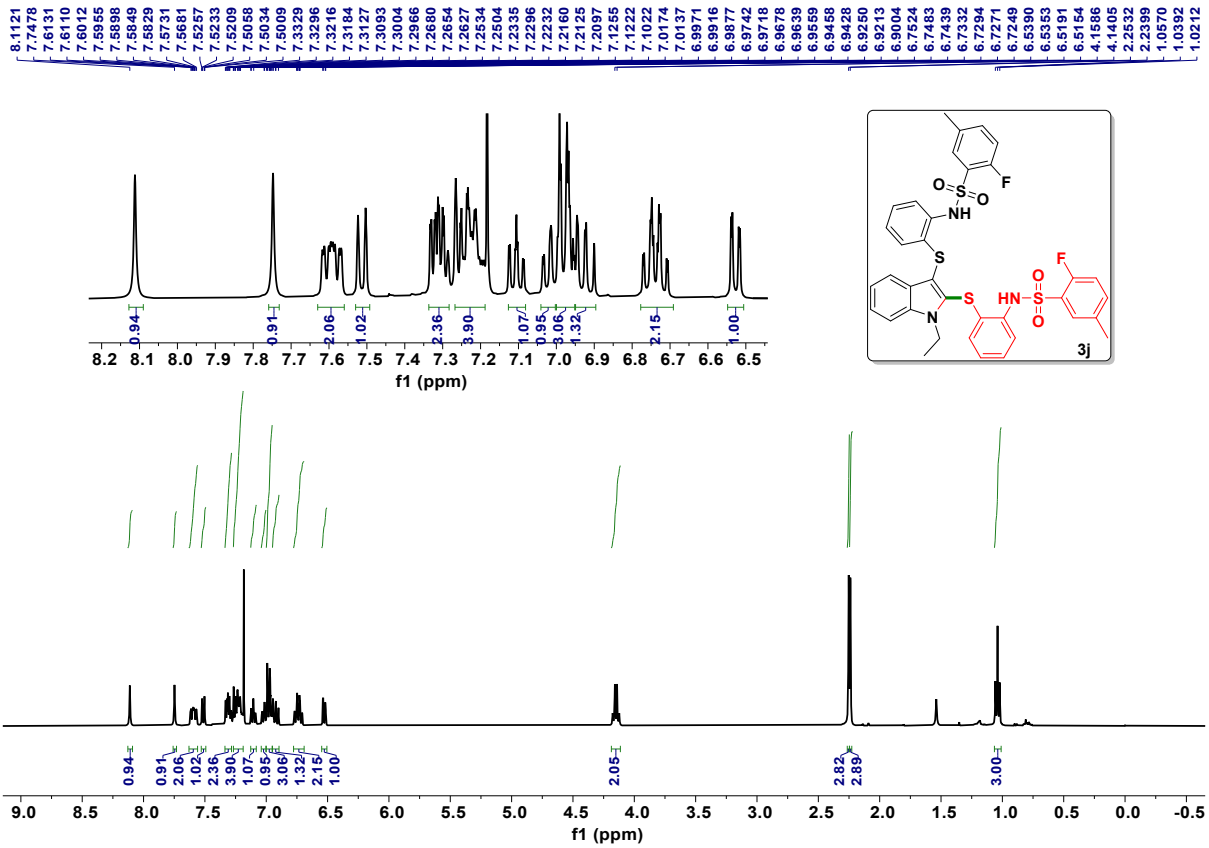


Figure S70. ^{1}H NMR of compound **3j** (400 MHz, $CDCl_3$)

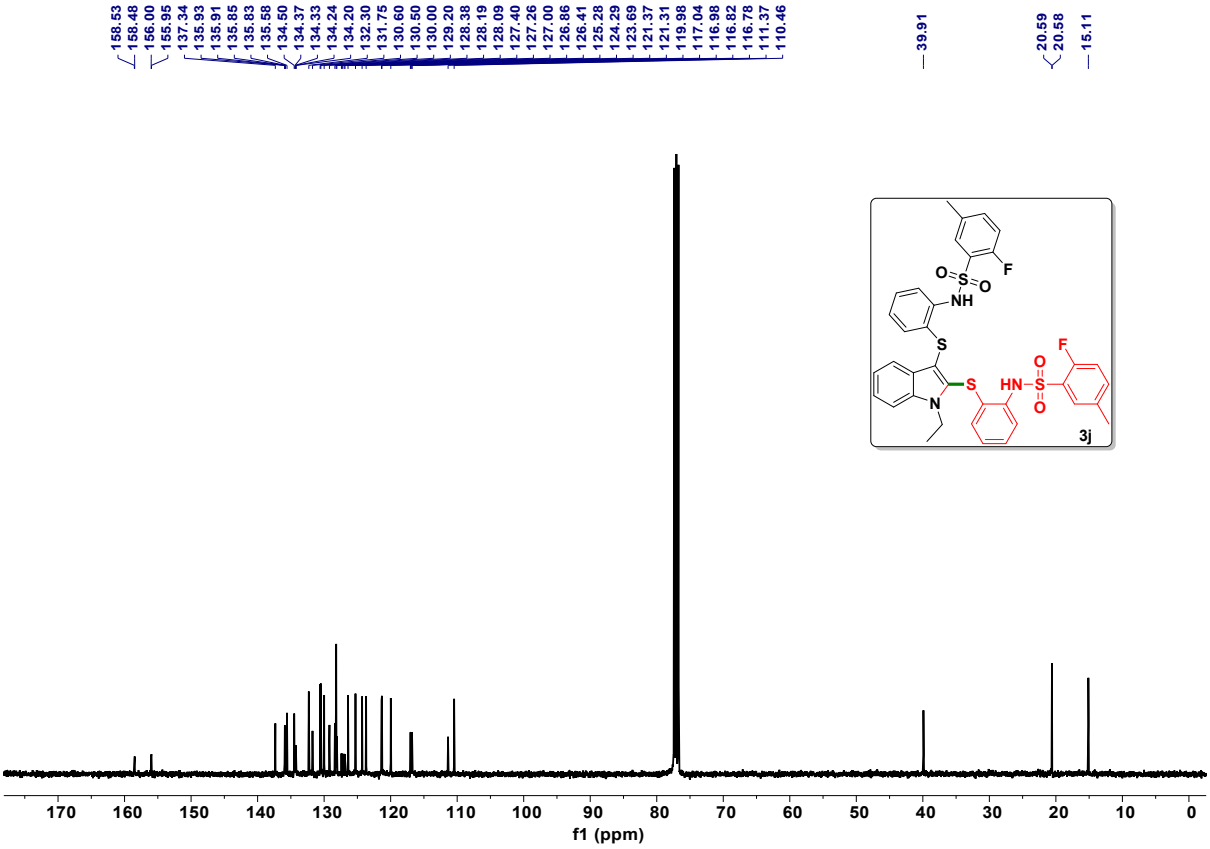


Figure S71. $^{13}C\{^{1}H\}$ NMR of compound **3j** (100 MHz, $CDCl_3$)

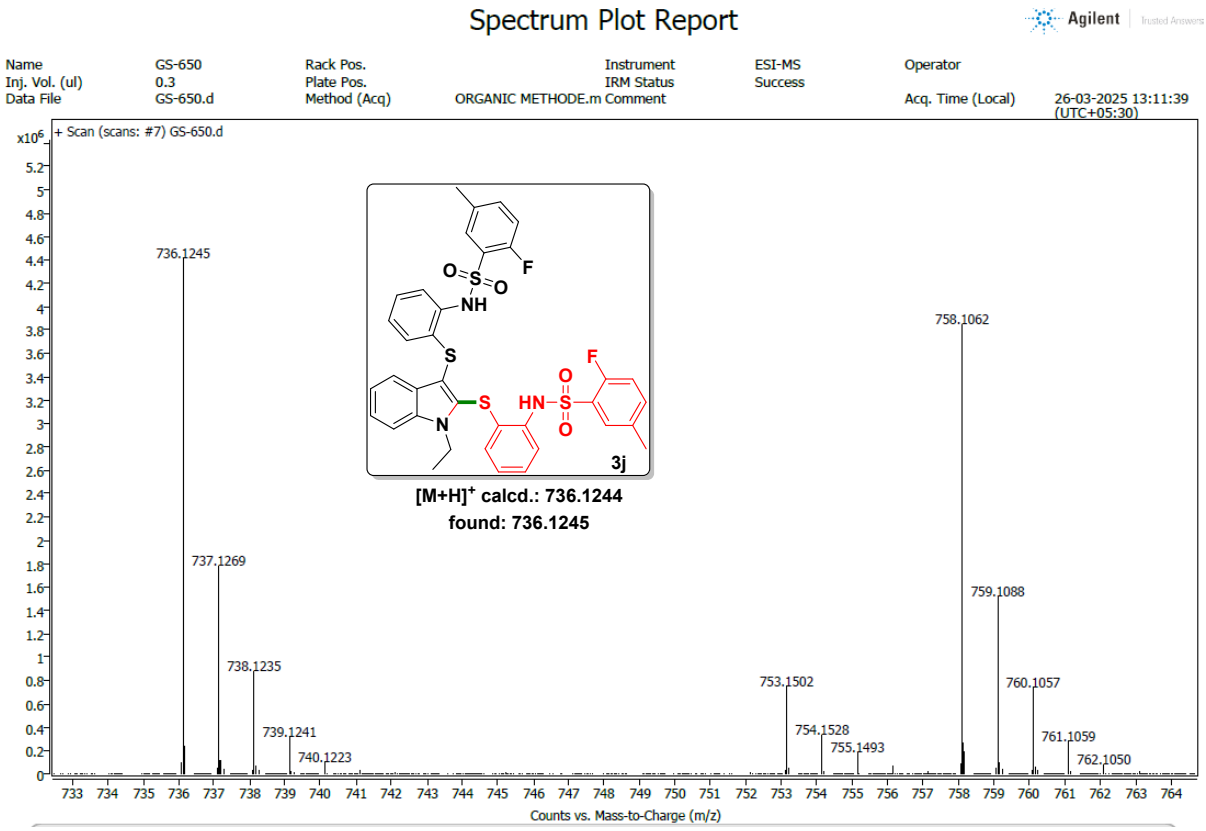


Figure S72. HRMS of compound **3j**

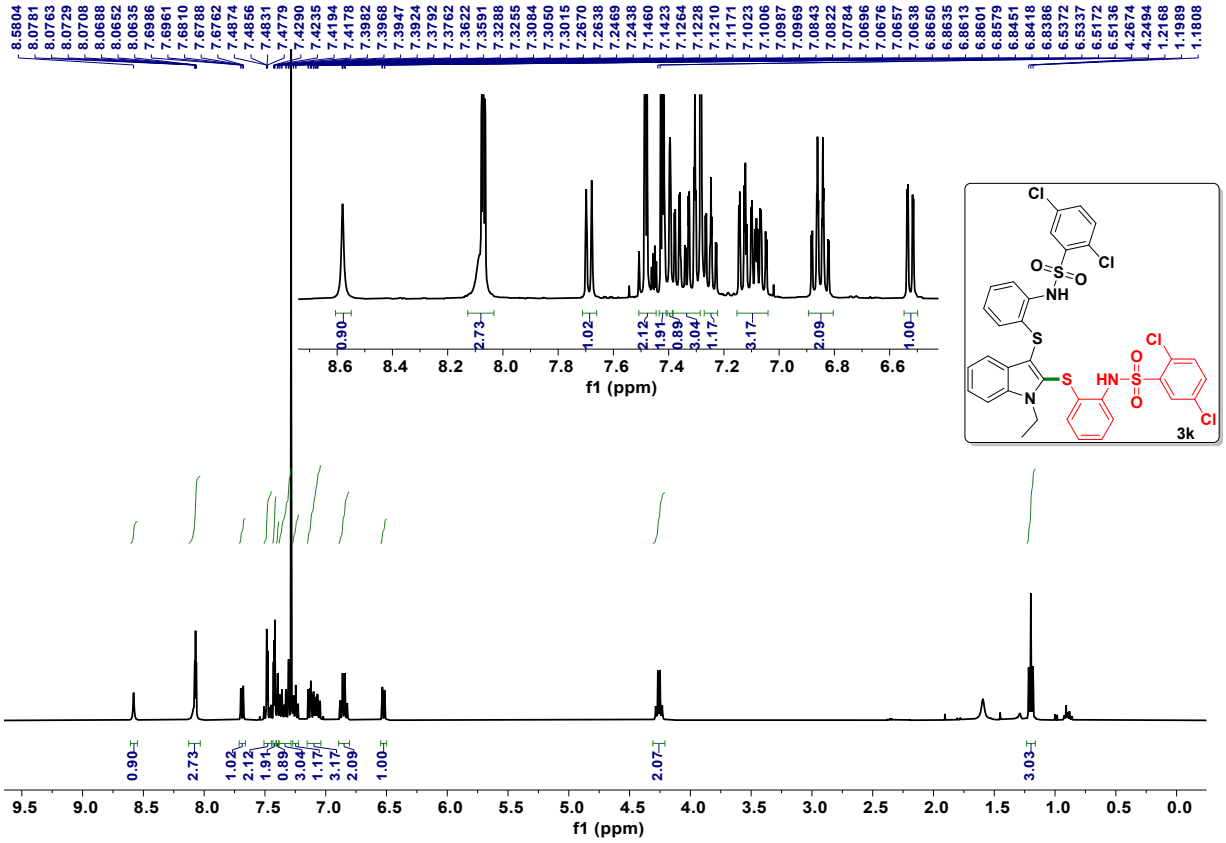


Figure S73. ^{1}H NMR of compound **3k** (400 MHz, $CDCl_3$)

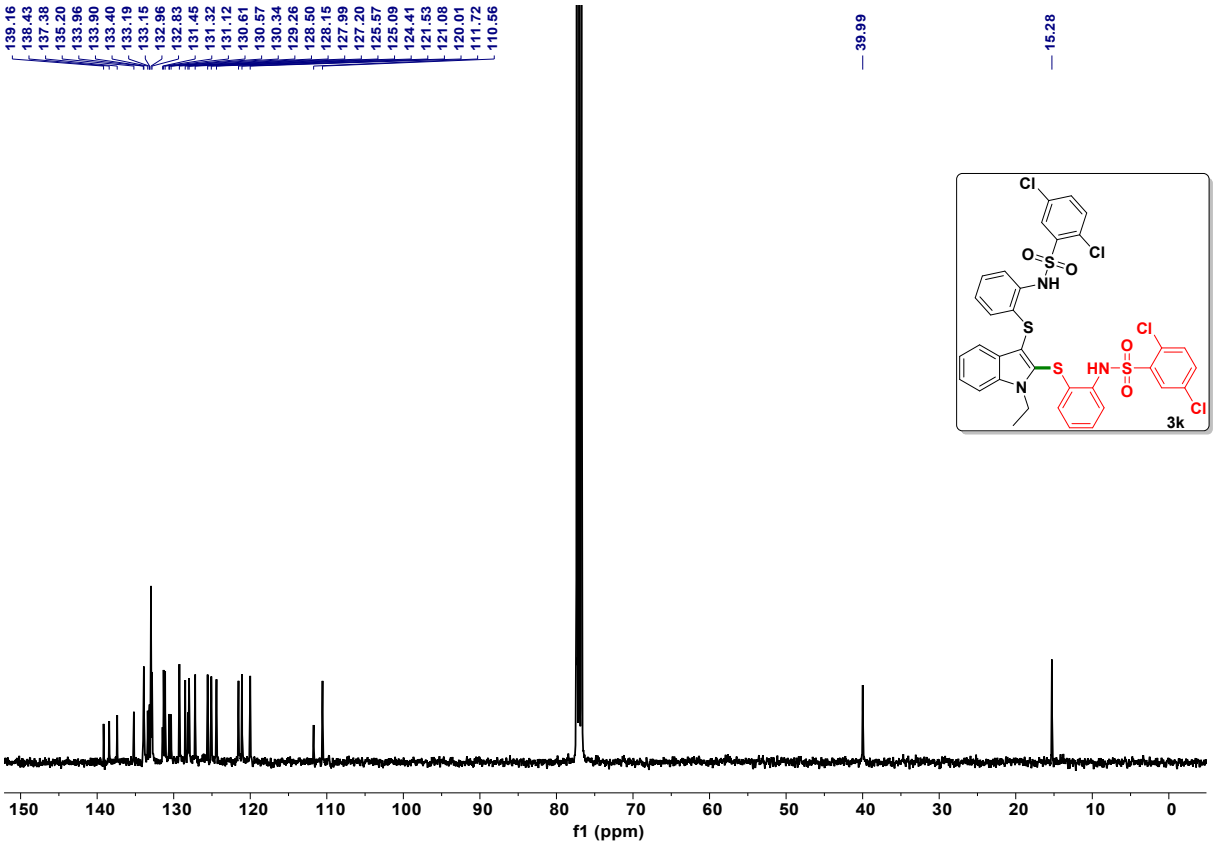


Figure S74. $^{13}C\{^{1}H\}$ NMR of compound **3k** (100 MHz, $CDCl_3$)

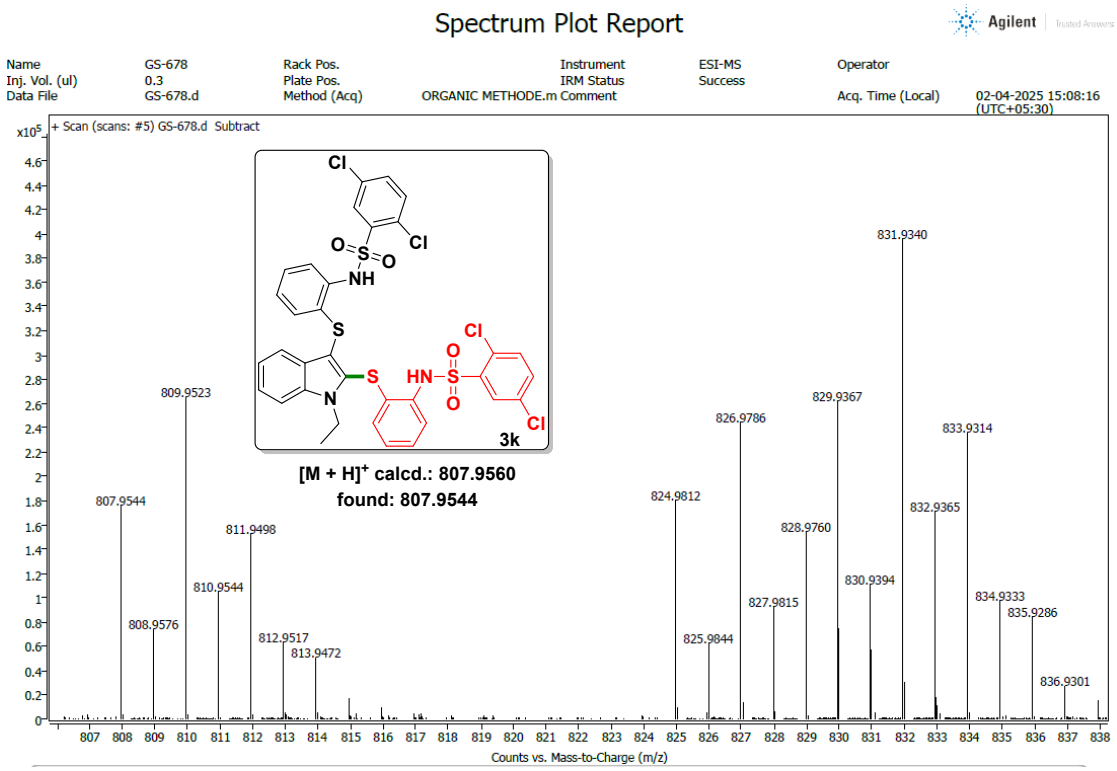


Figure S75. HRMS of compound **3k**

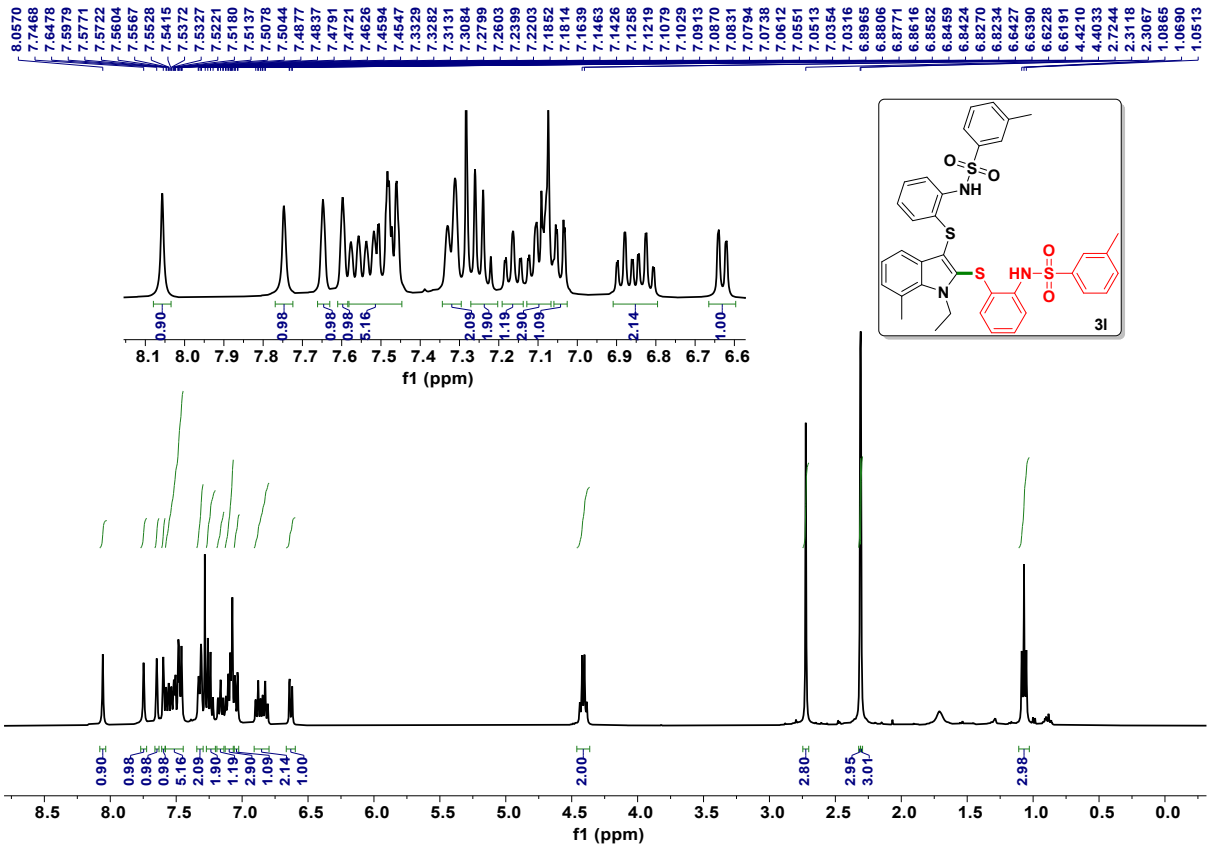


Figure S76. ^{1}H NMR of compound **3l** (400 MHz, $CDCl_3$)

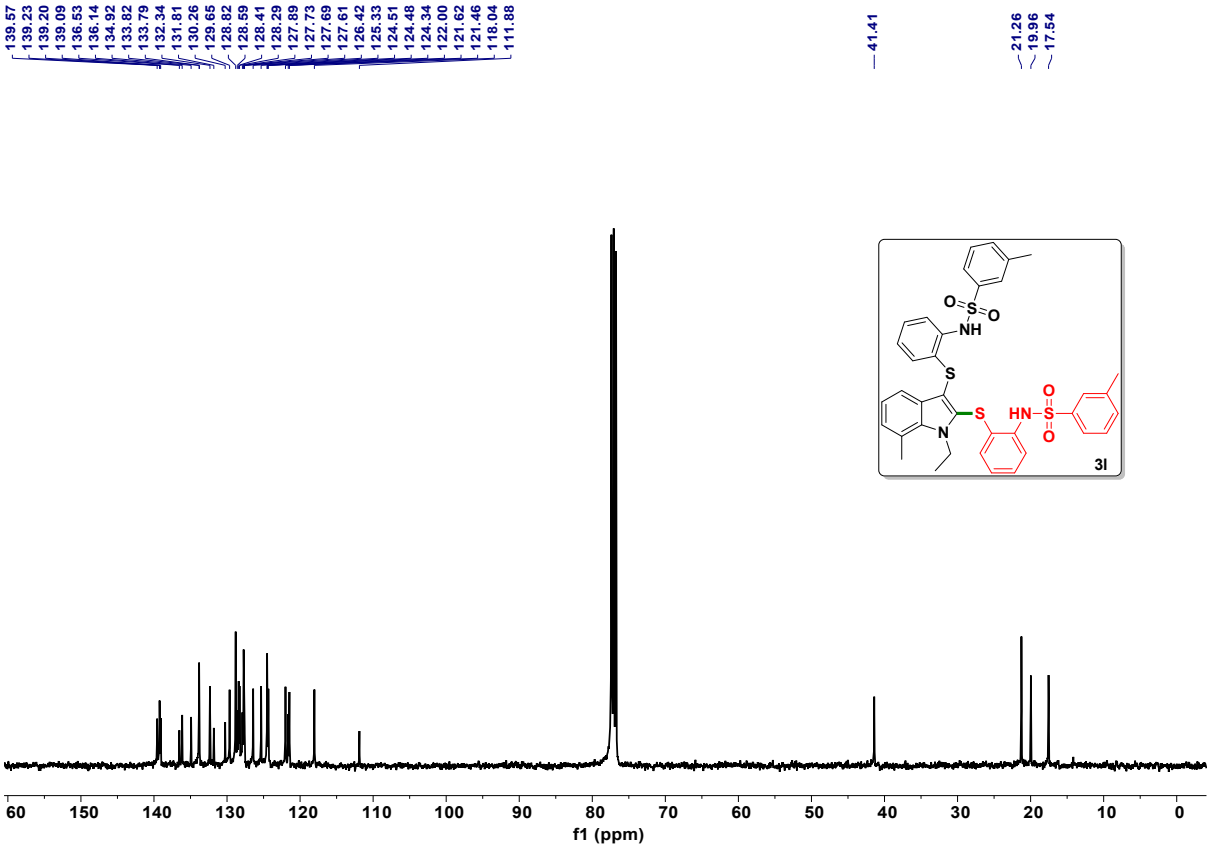


Figure S77. $^{13}C\{^{1}H\}$ NMR of compound **3l** (100 MHz, $CDCl_3$)

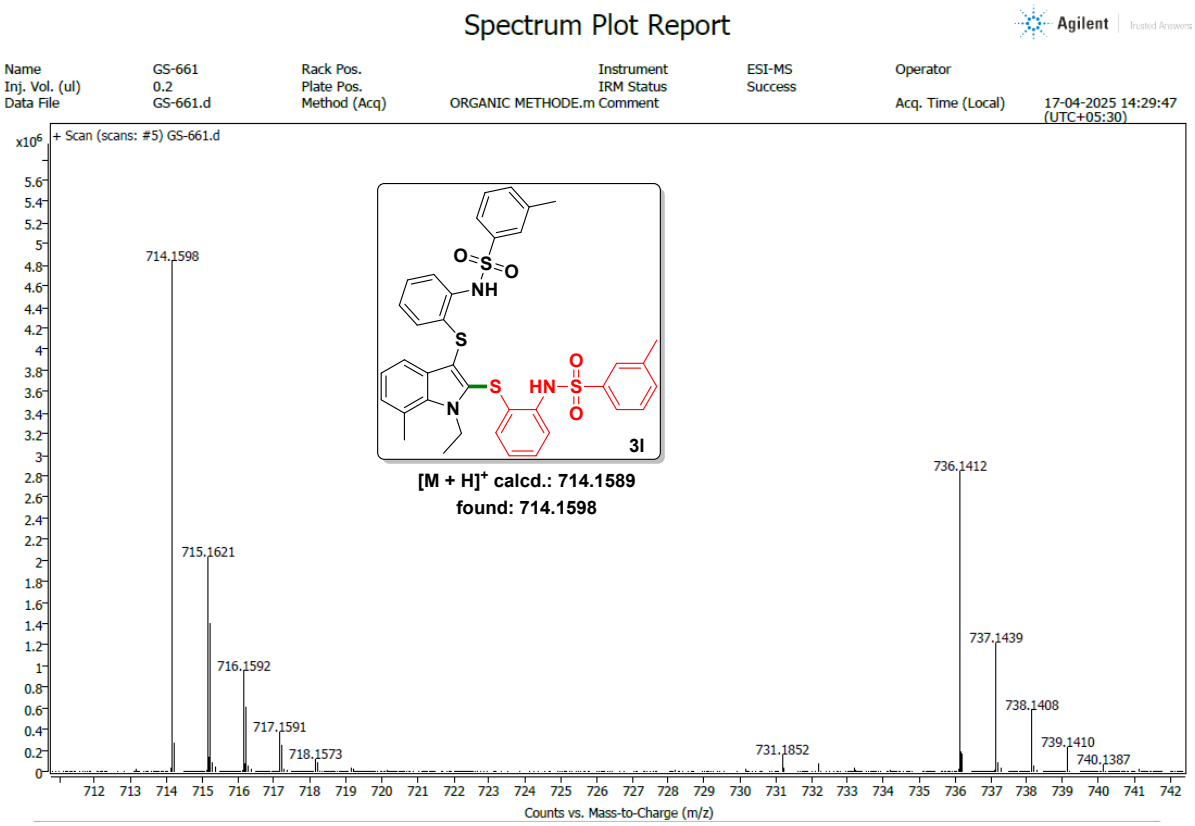


Figure S78. HRMS of compound **3l**

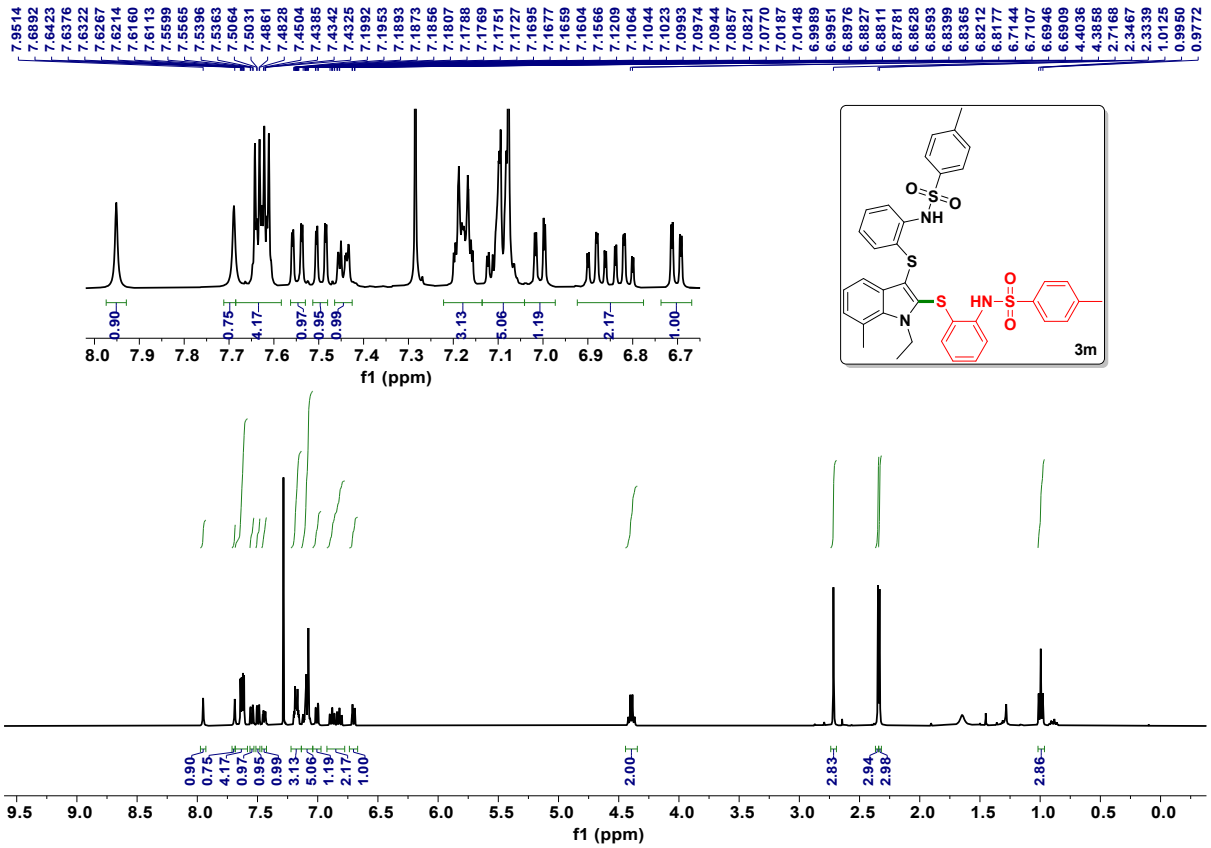


Figure S79. ^{1}H NMR of compound **3m** (400 MHz, $CDCl_3$)

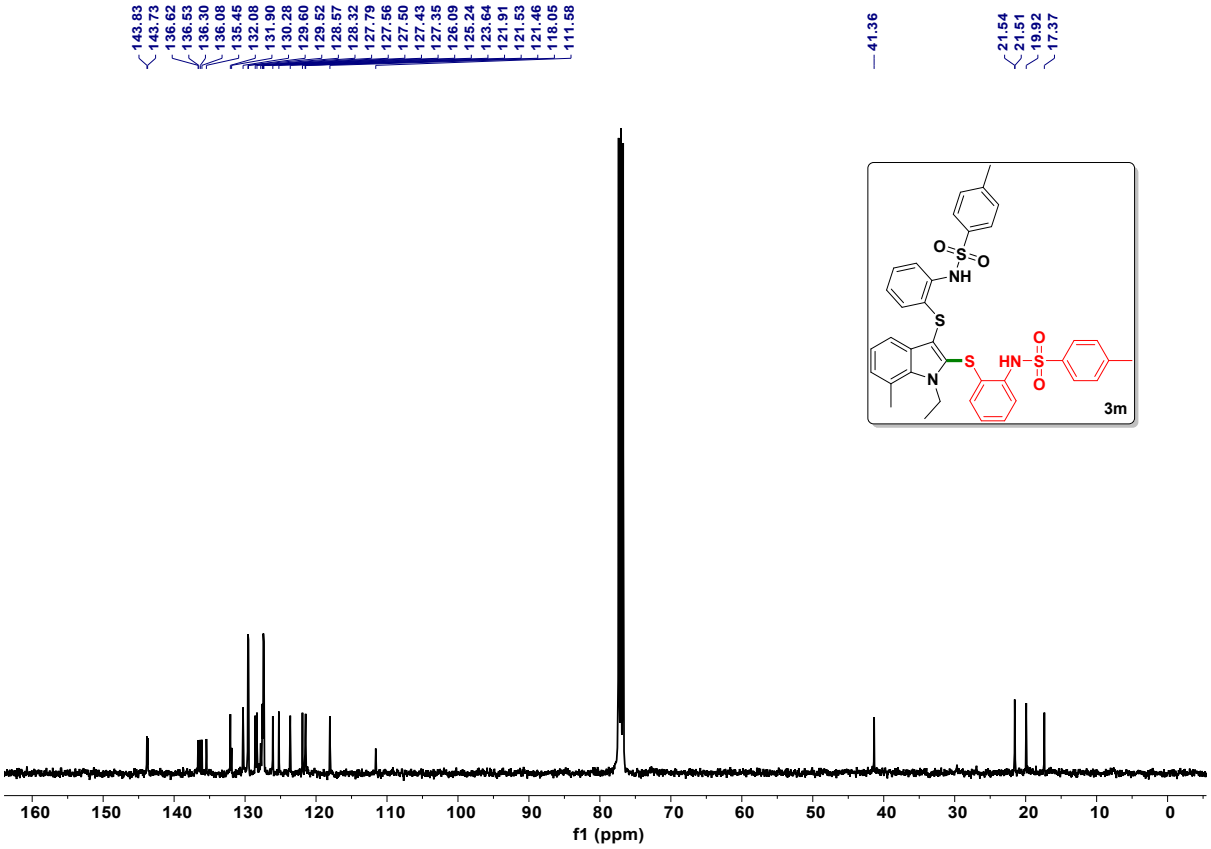


Figure S80. $^{13}C\{^{1}H\}$ NMR of compound **3m** (100 MHz, $CDCl_3$)

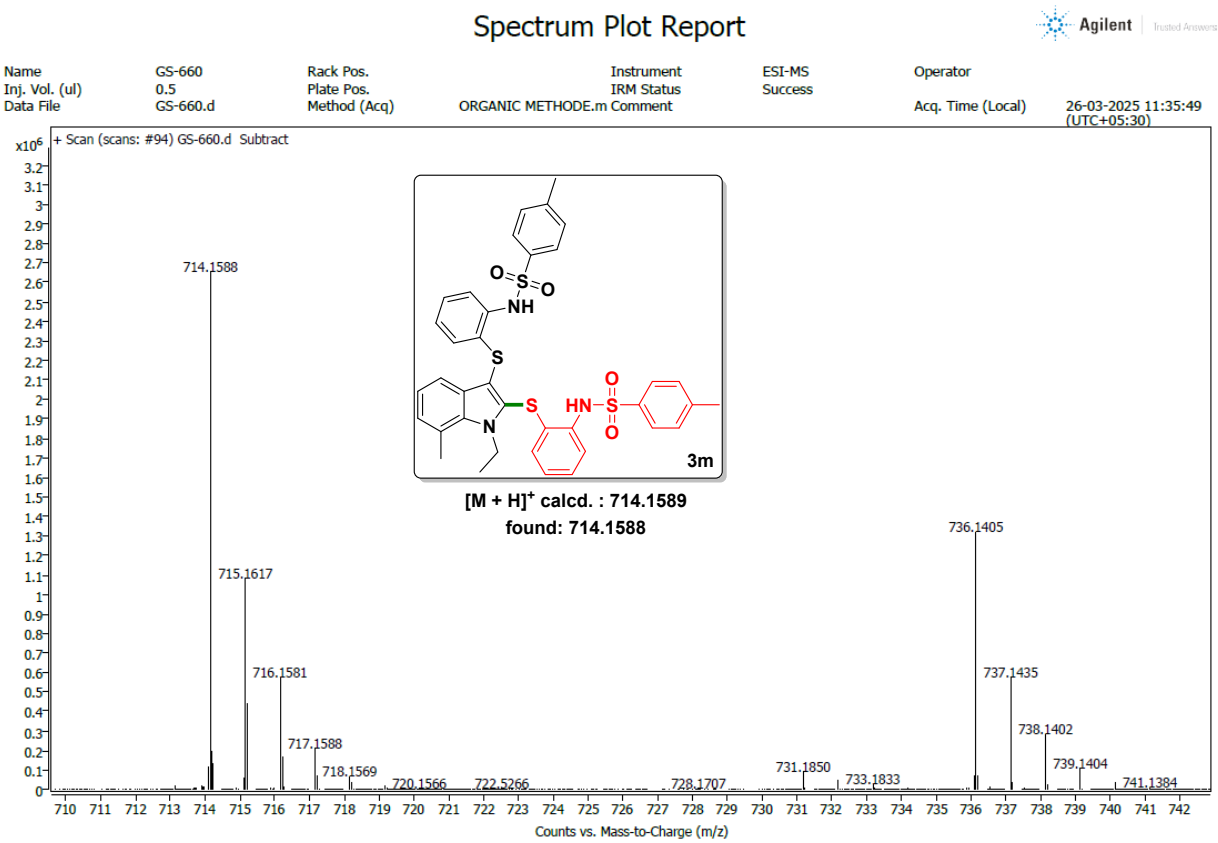


Figure S81. HRMS of compound **3m**

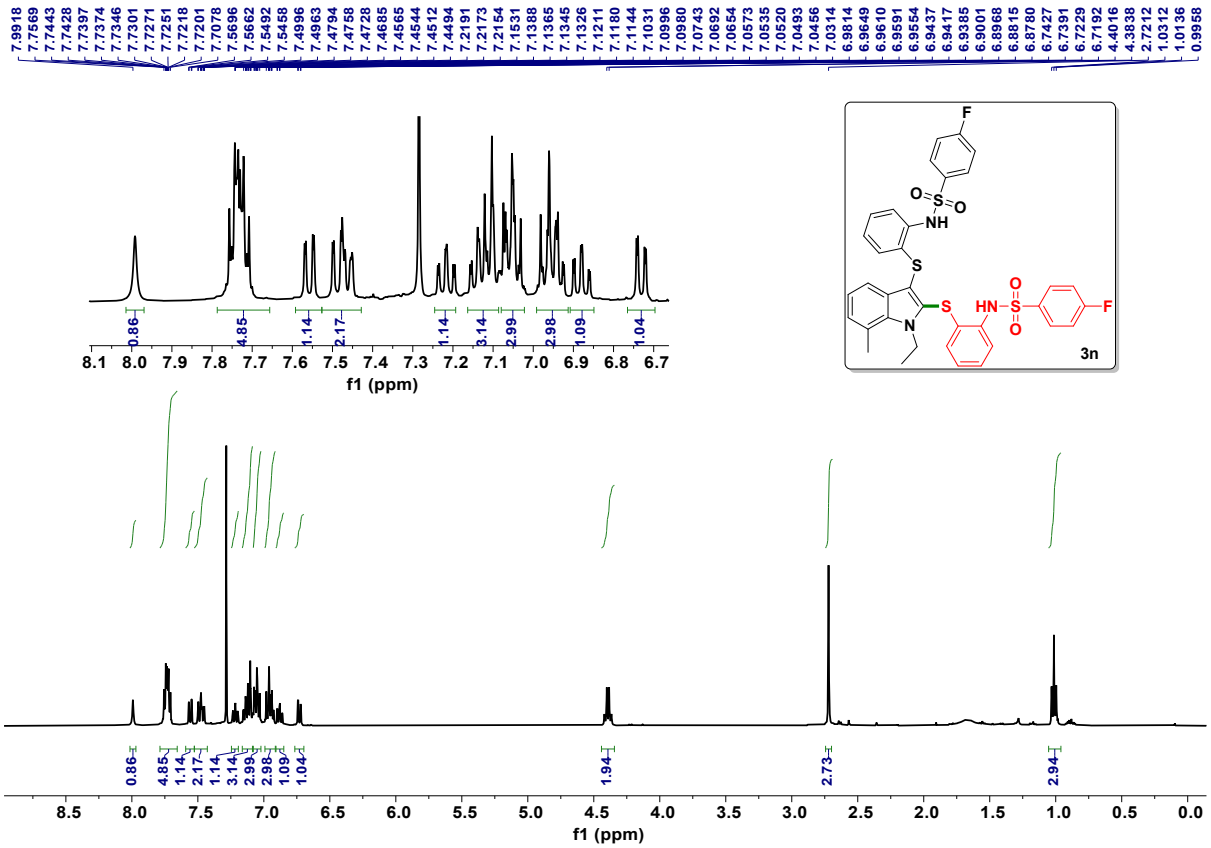


Figure S82. ^{1}H NMR of compound **3n** (400 MHz, $CDCl_3$)

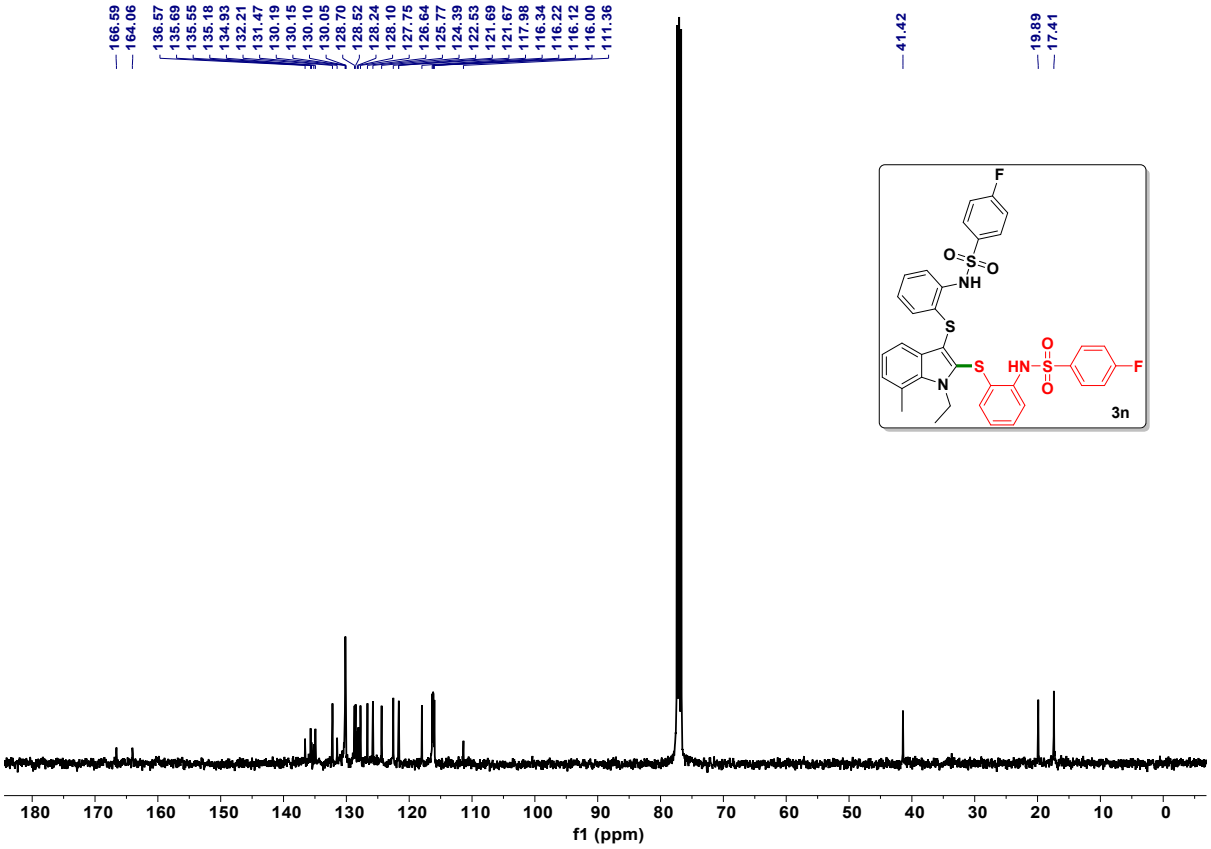


Figure S83. $^{13}C\{^{1}H\}$ NMR of compound **3n** (100 MHz, $CDCl_3$)

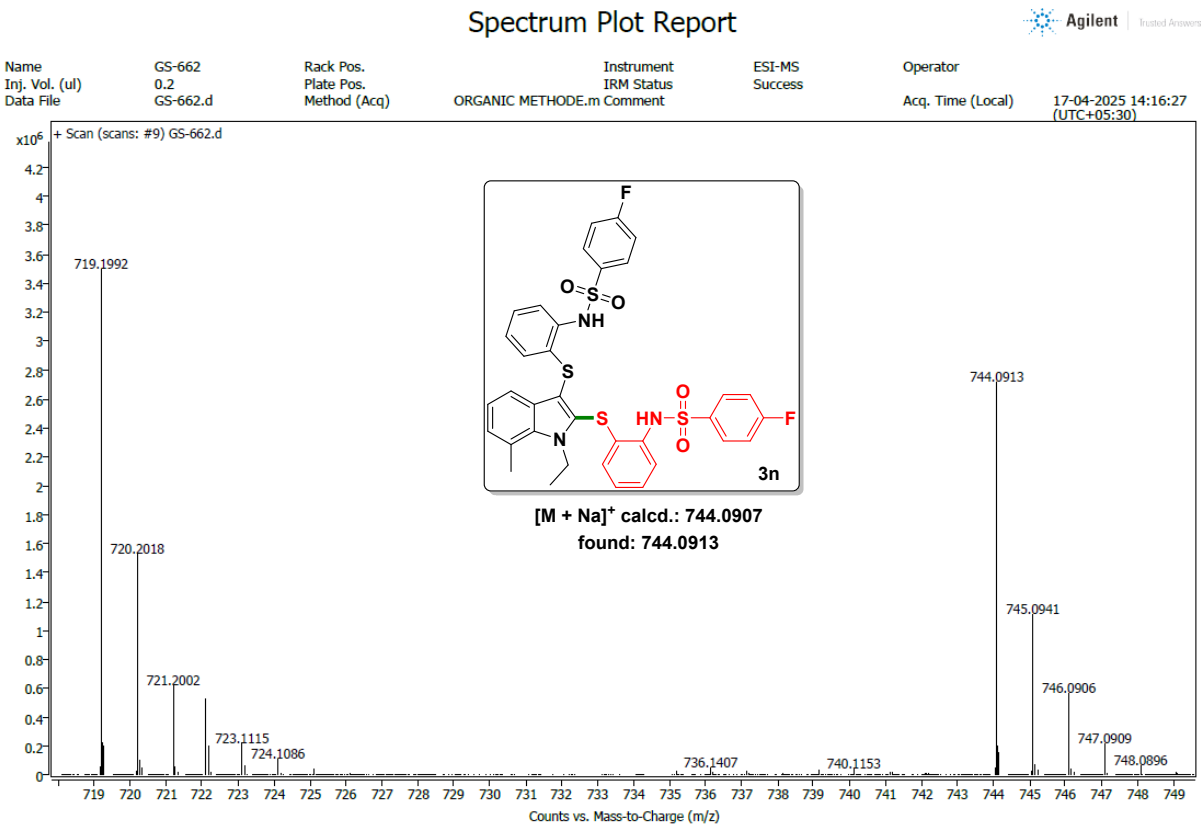


Figure S84. HRMS of compound **3n**

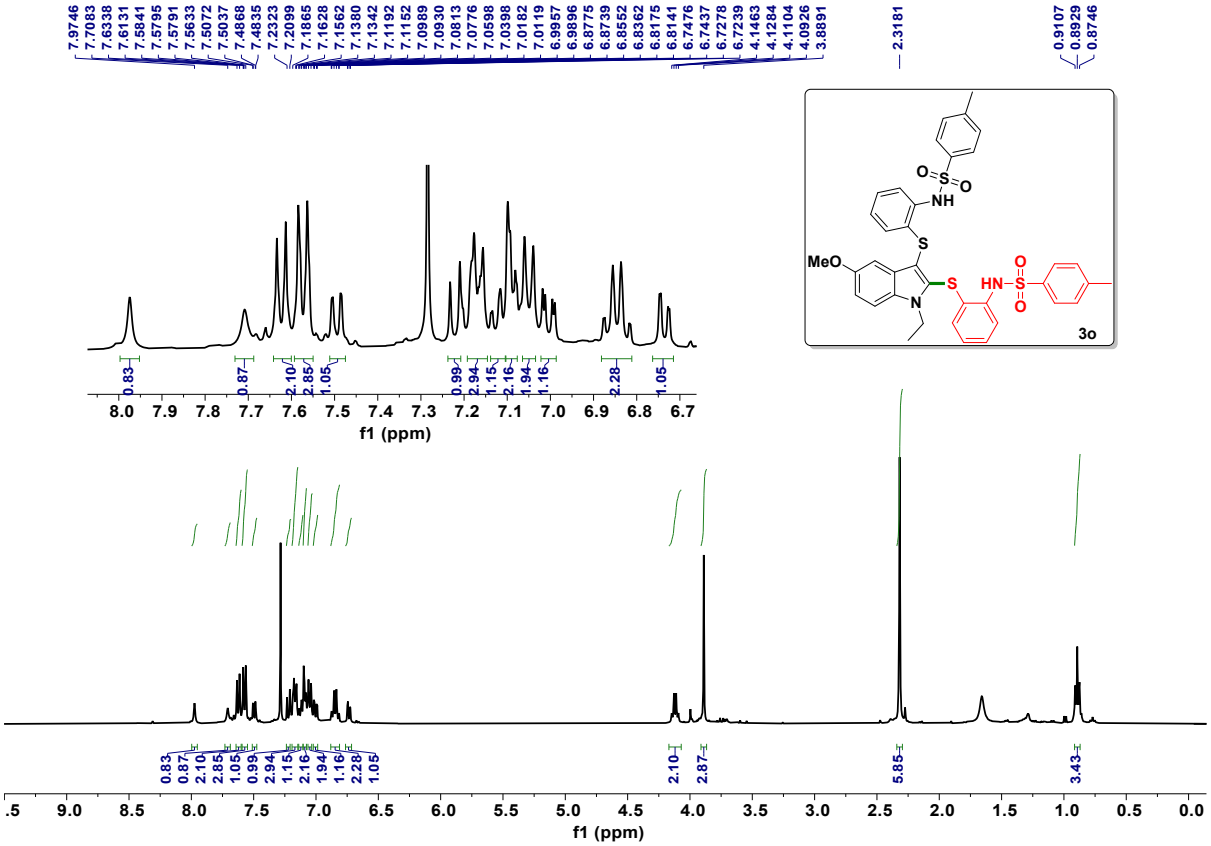


Figure S85. ^{1}H NMR of compound **3o** (400 MHz, $CDCl_3$)

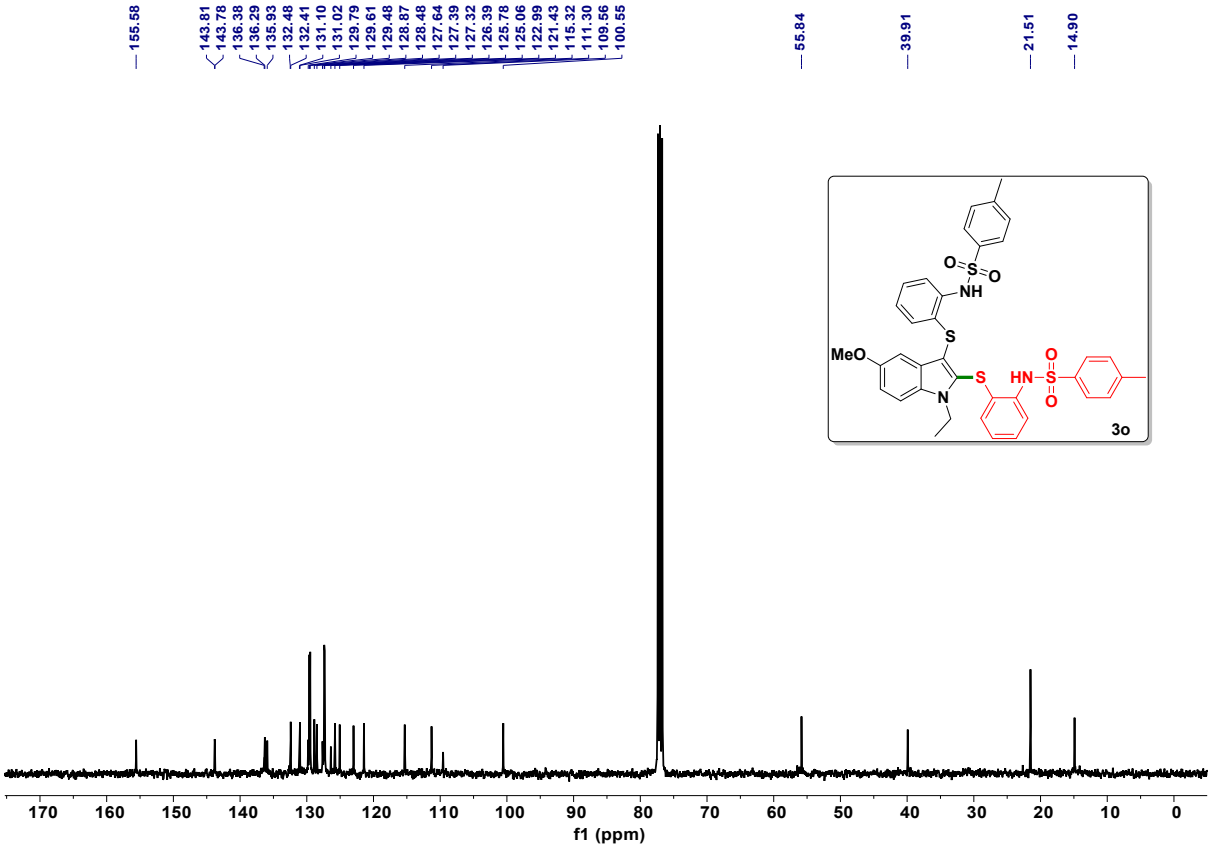


Figure S86. $^{13}C\{^{1}H\}$ NMR of compound **3o** (100 MHz, $CDCl_3$)

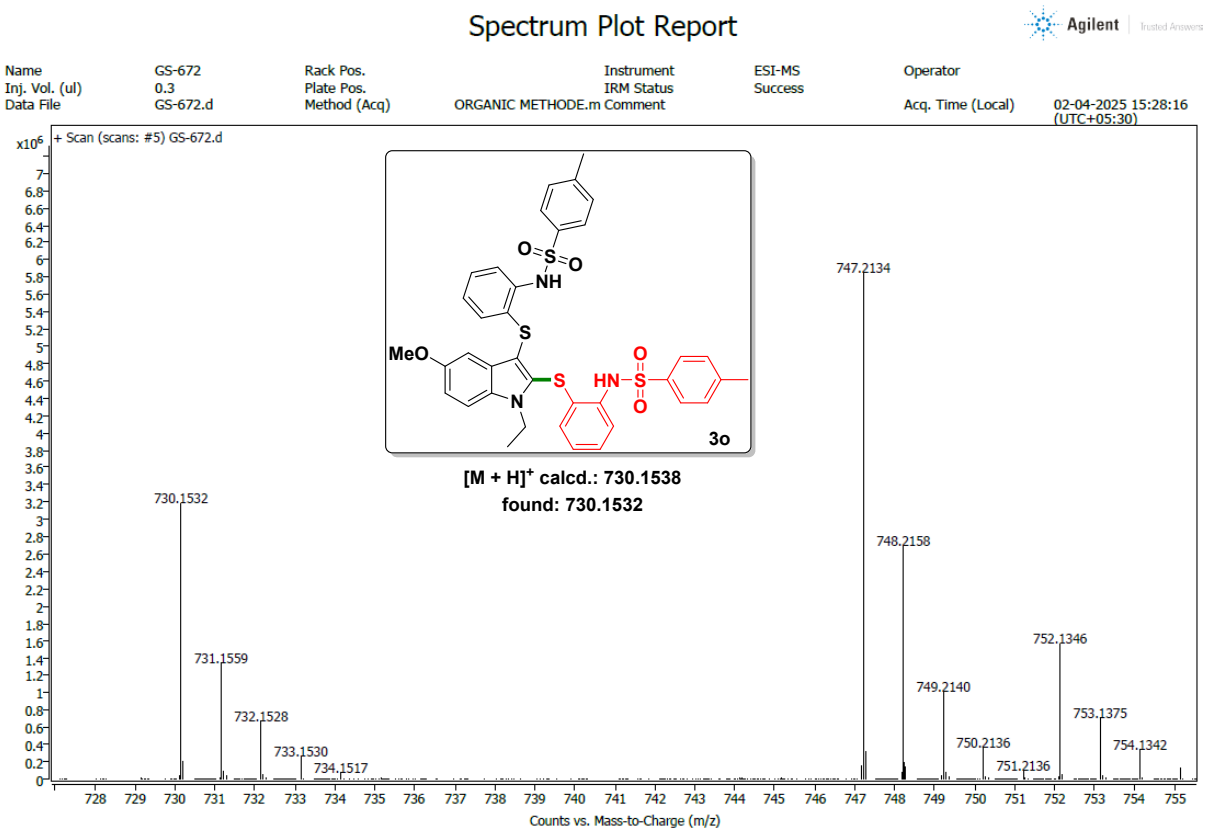


Figure S87. HRMS of compound **3o**

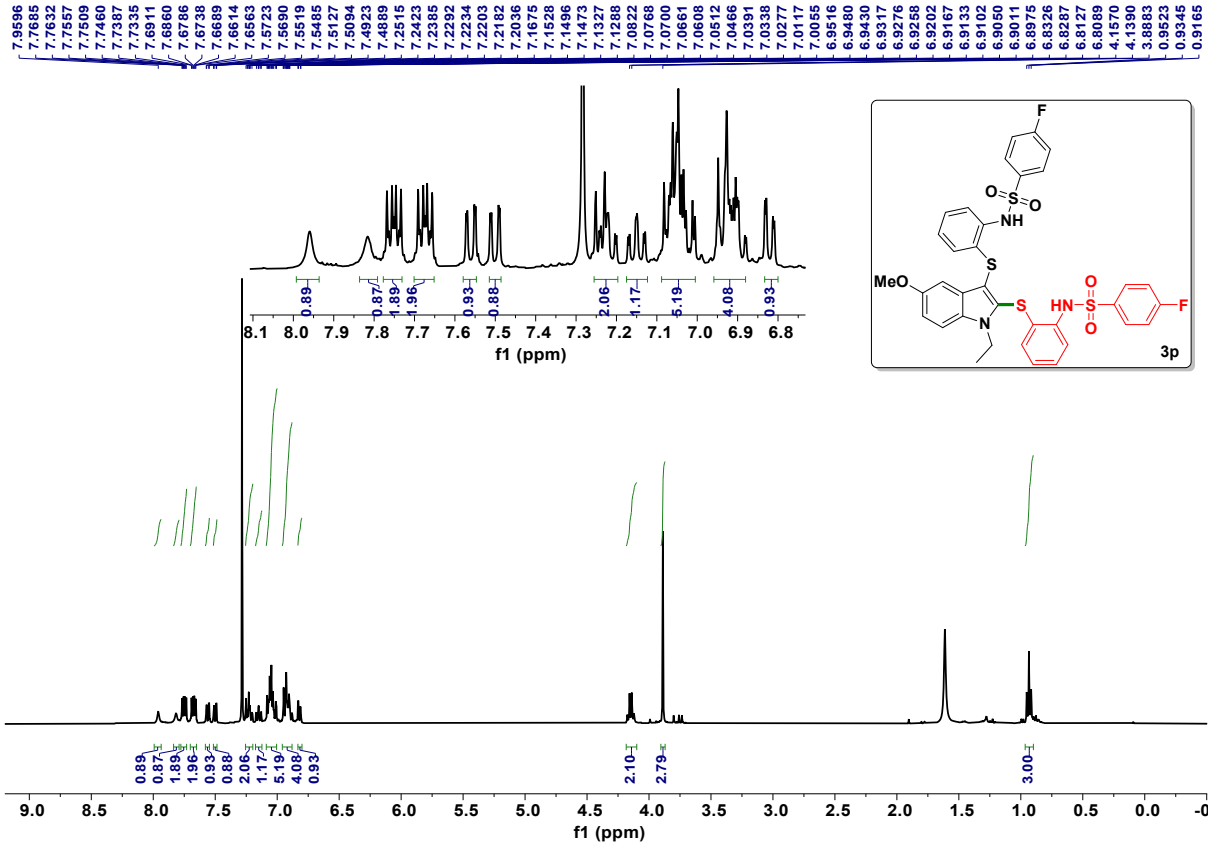


Figure S88. ^{1}H NMR of compound **3p** (400 MHz, $CDCl_3$)

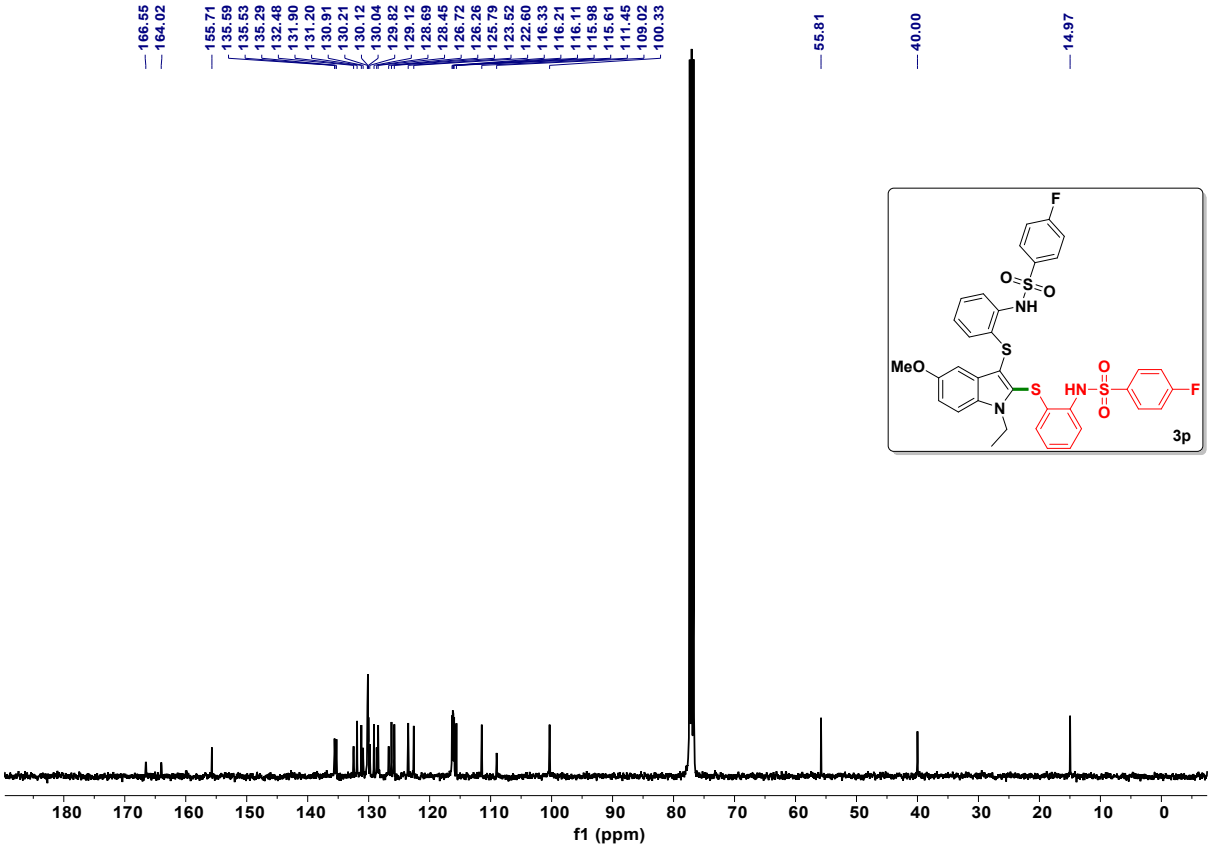


Figure S89. $^{13}C\{^{1}H\}$ NMR of compound **3p** (100 MHz, $CDCl_3$)

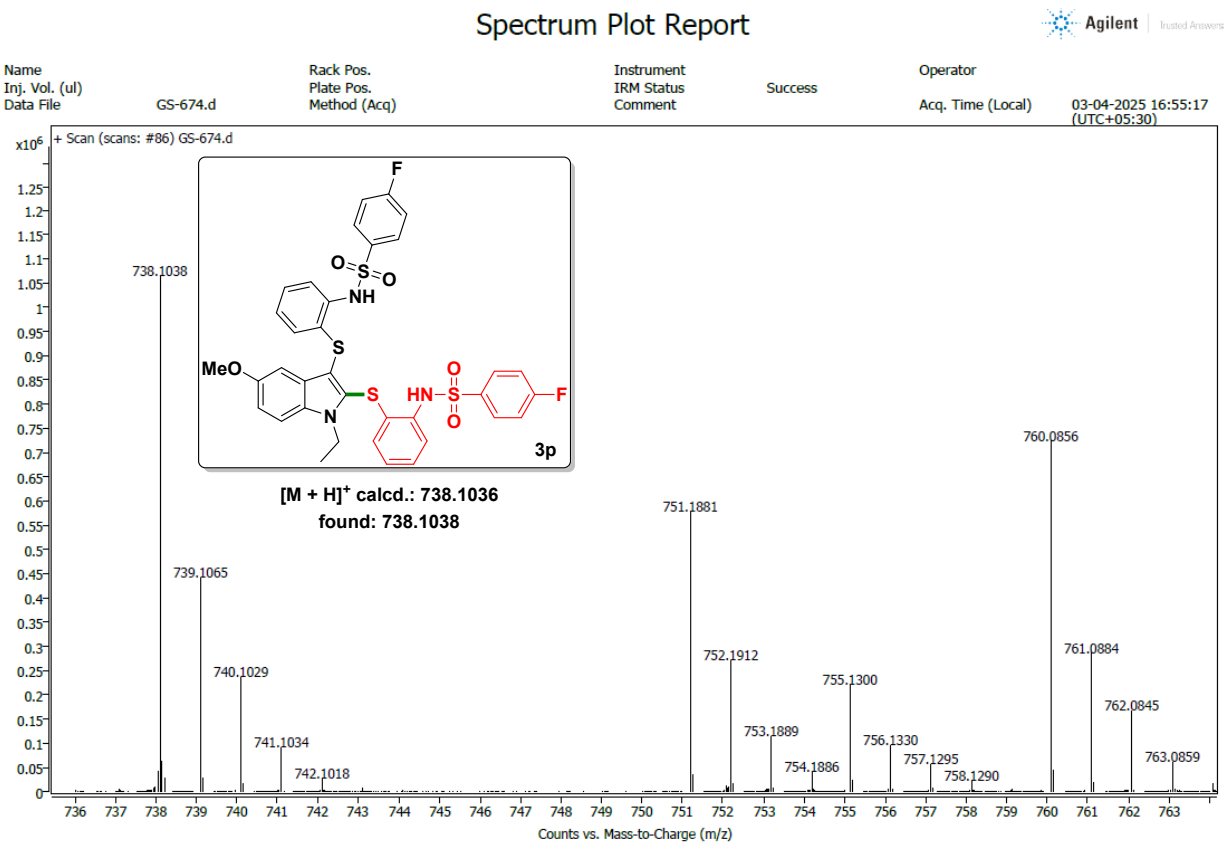


Figure S90. HRMS of compound **3p**

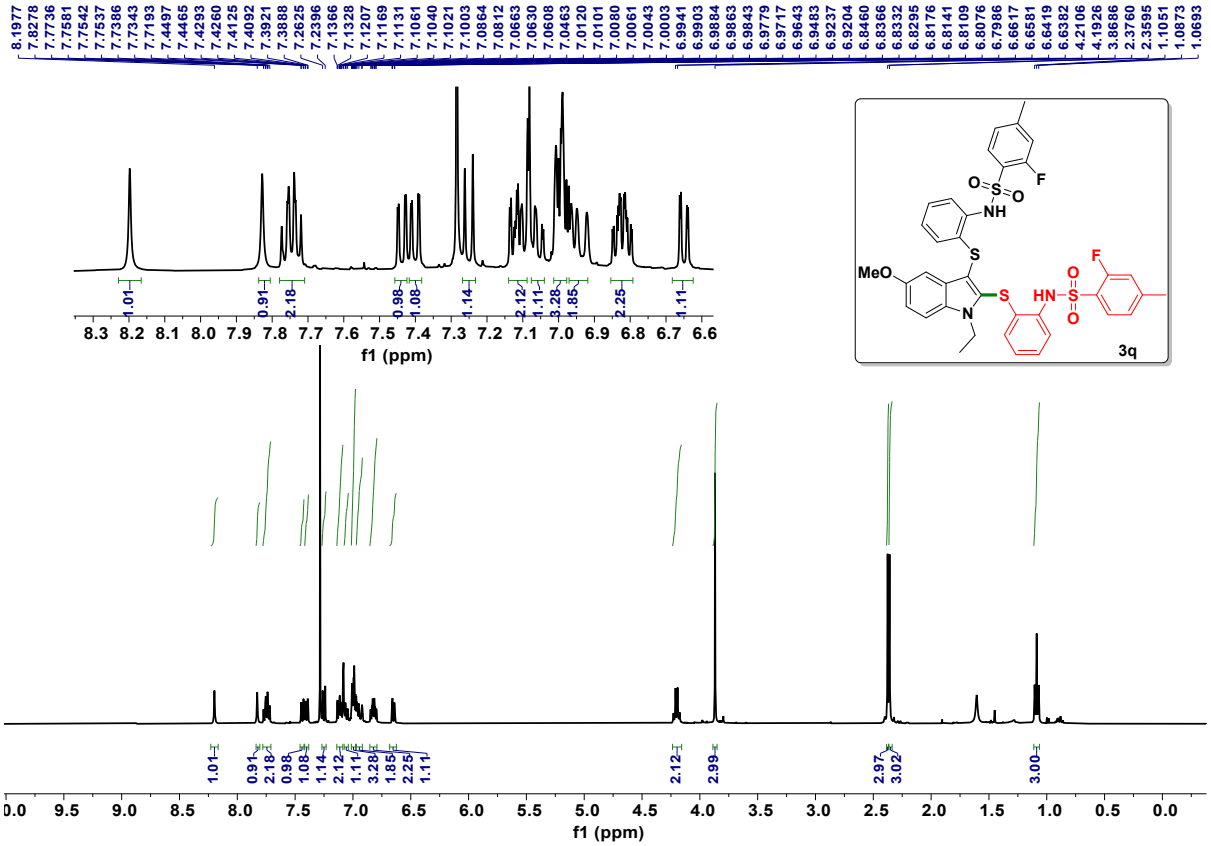


Figure S91. ^{1}H NMR of compound **3q** (400 MHz, $CDCl_3$)

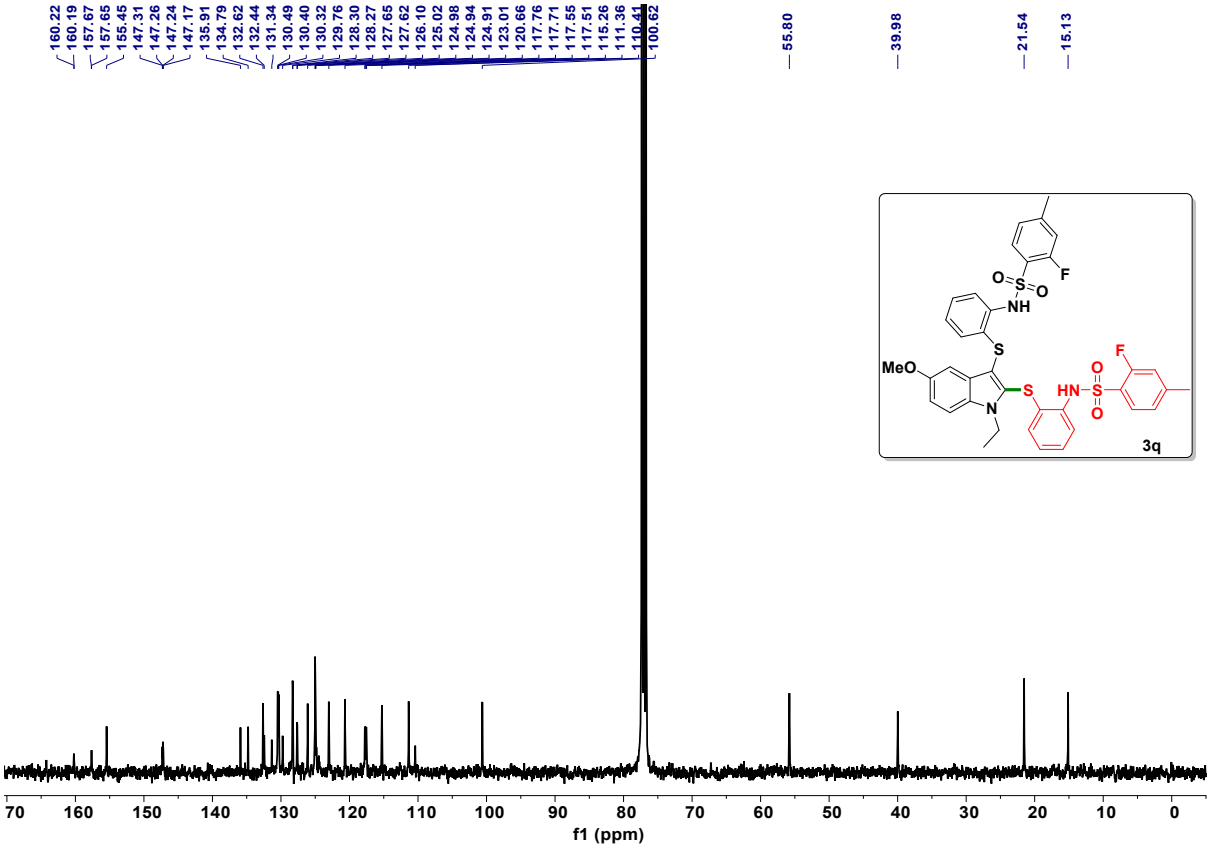


Figure S92. $^{13}C\{^{1}H\}$ NMR of compound **3q** (100 MHz, $CDCl_3$)

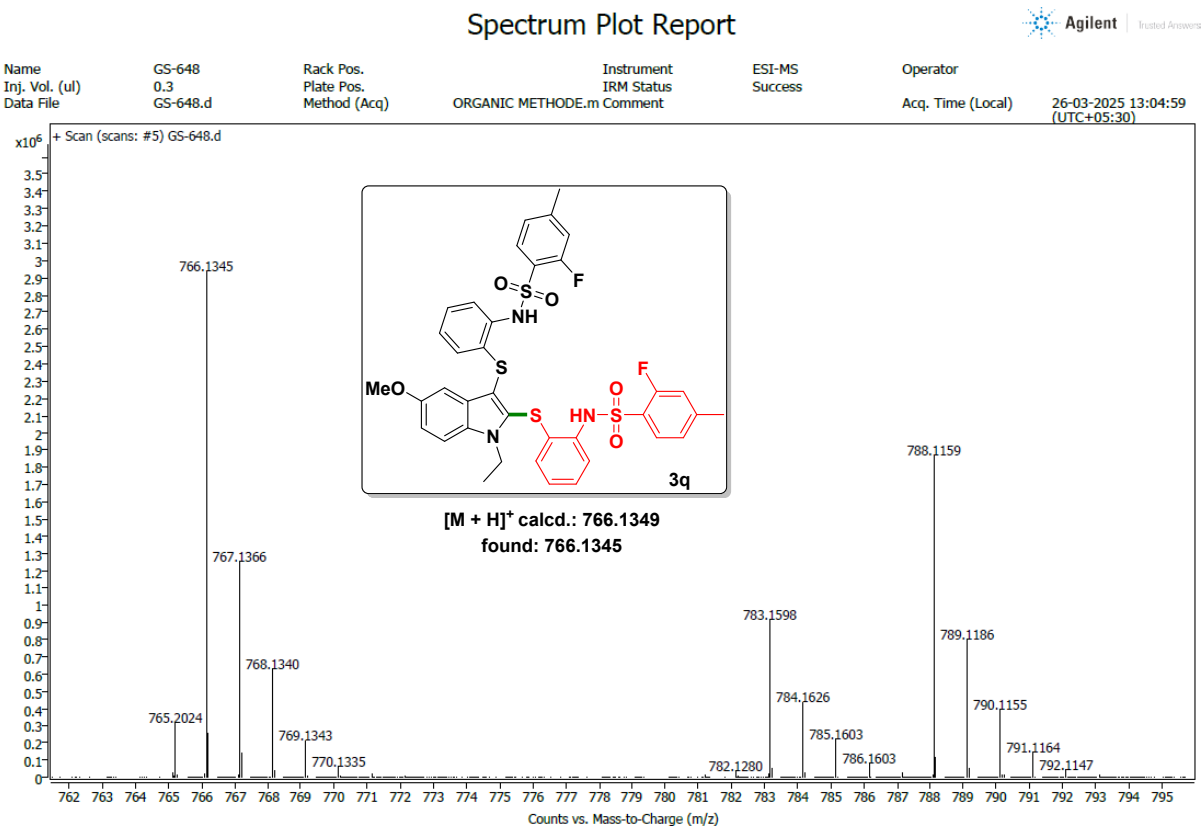


Figure S93. HRMS of compound **3q**

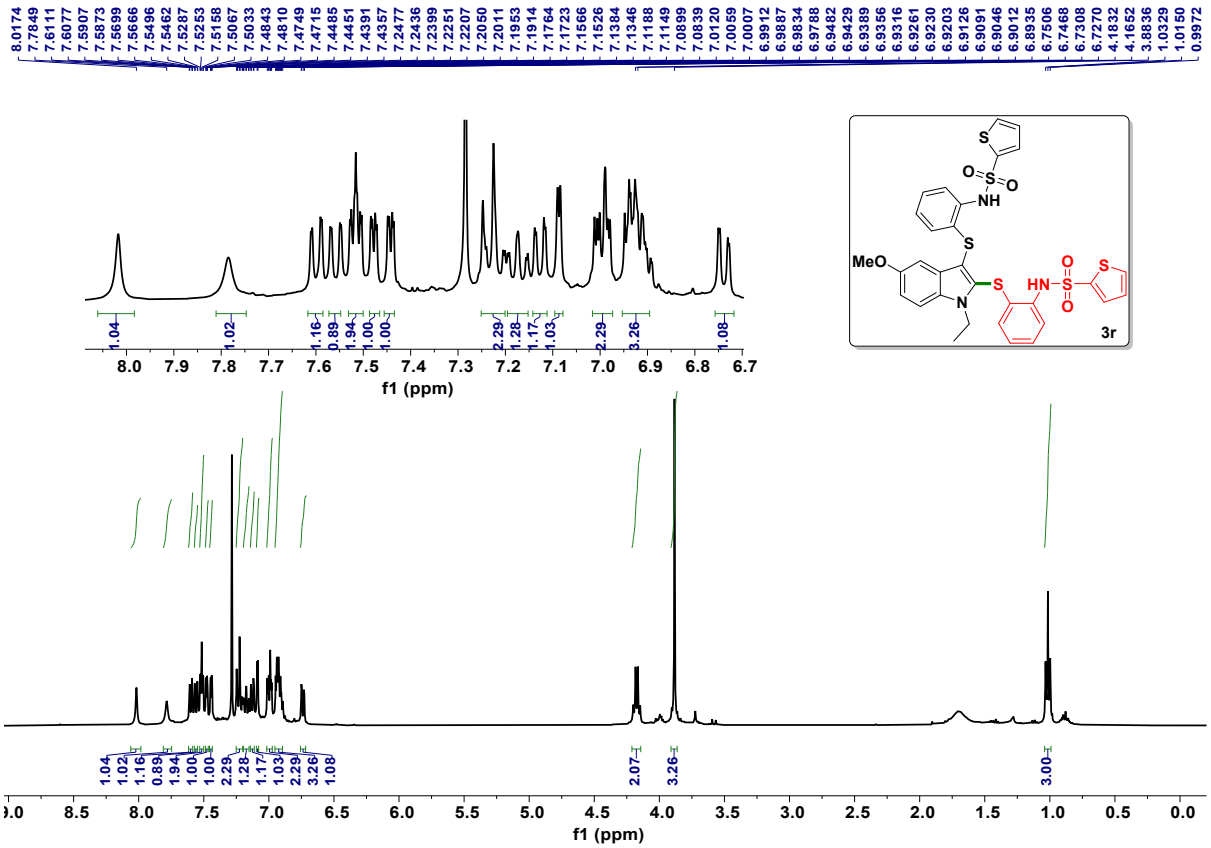


Figure S94. ^{1}H NMR of compound **3r** (400 MHz, $CDCl_3$)

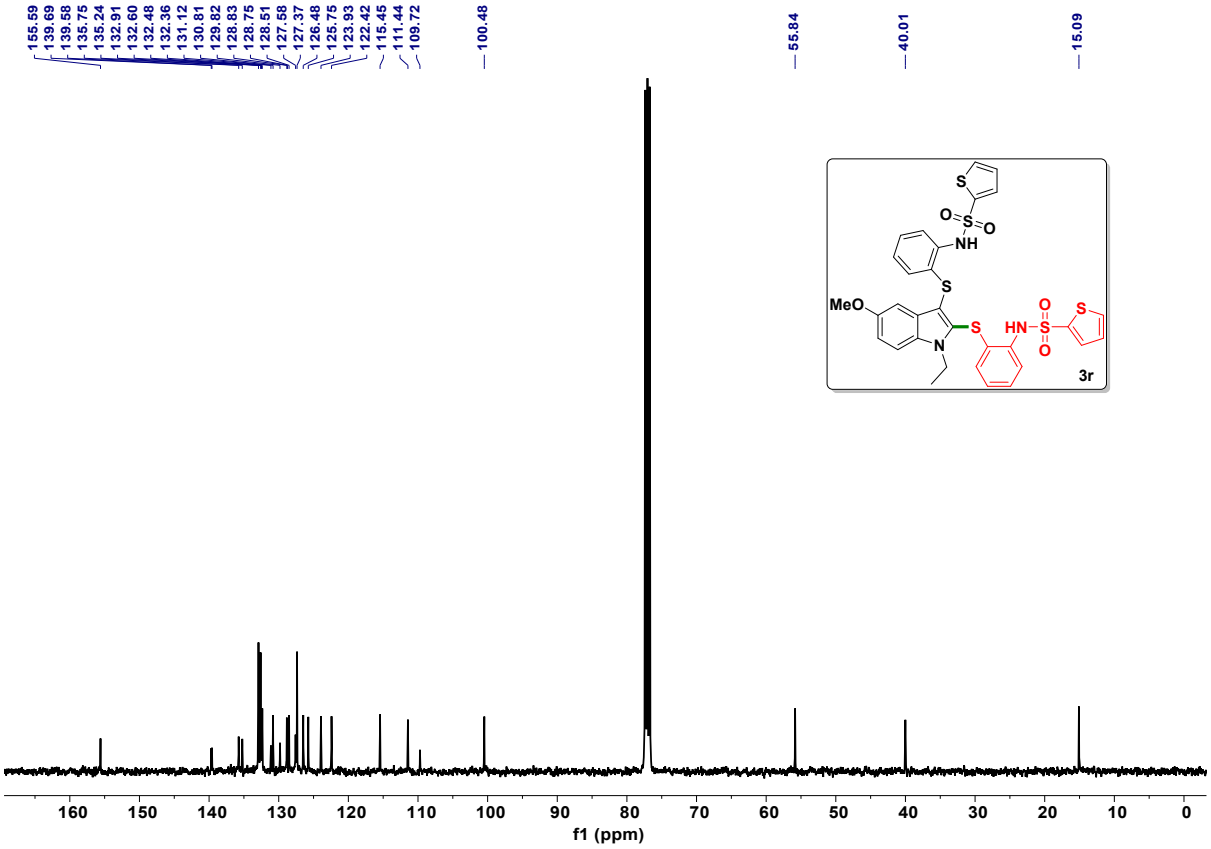


Figure S95. $^{13}C\{^{1}H\}$ NMR of compound **3r** (100 MHz, $CDCl_3$)

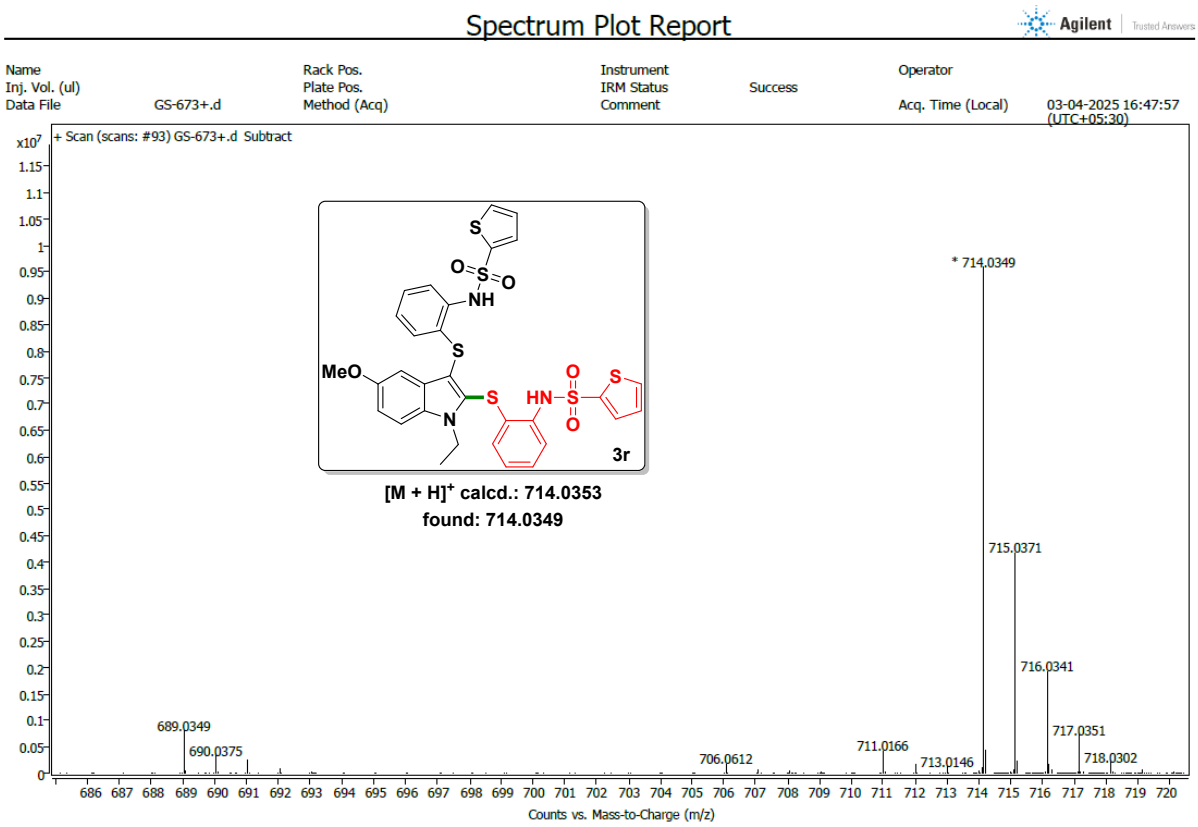


Figure S96. HRMS of compound **3r**

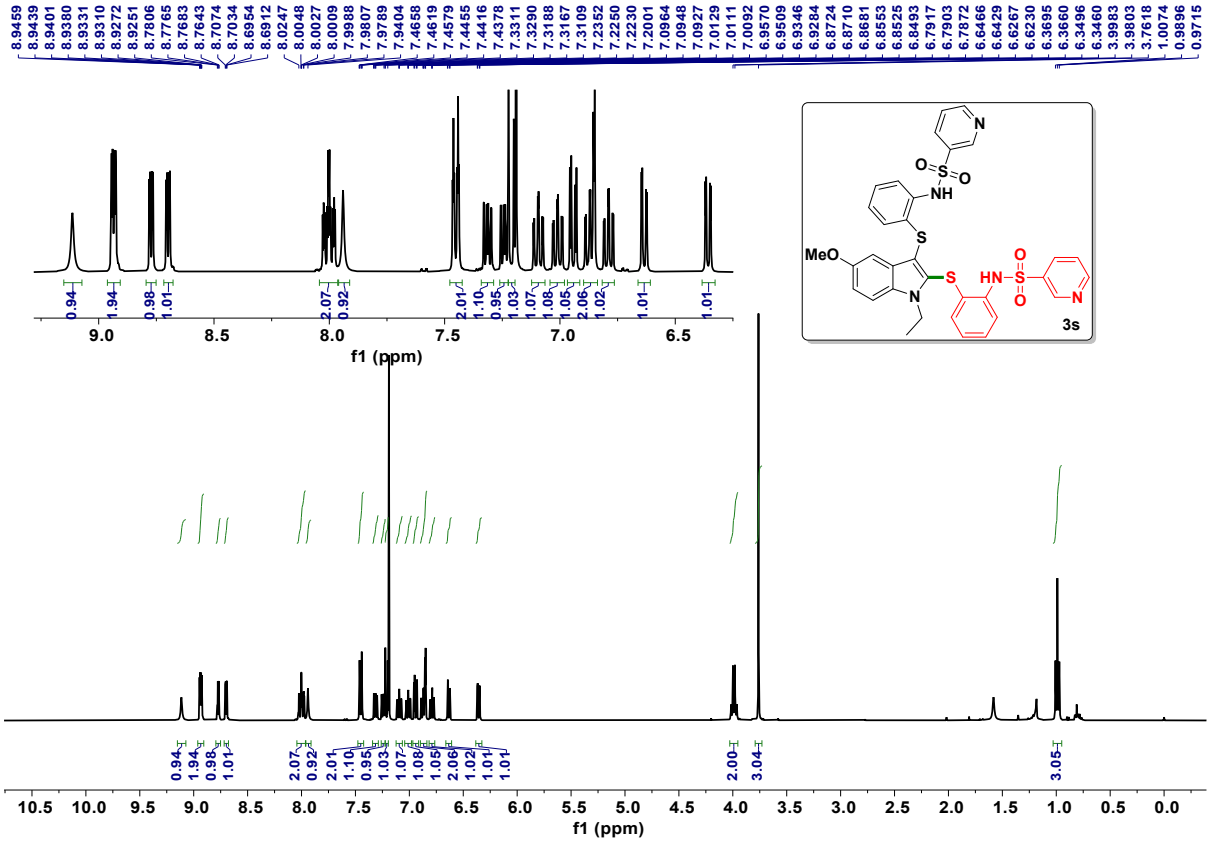


Figure S97. ^{1}H NMR of compound **3s** (400 MHz, $CDCl_3$)

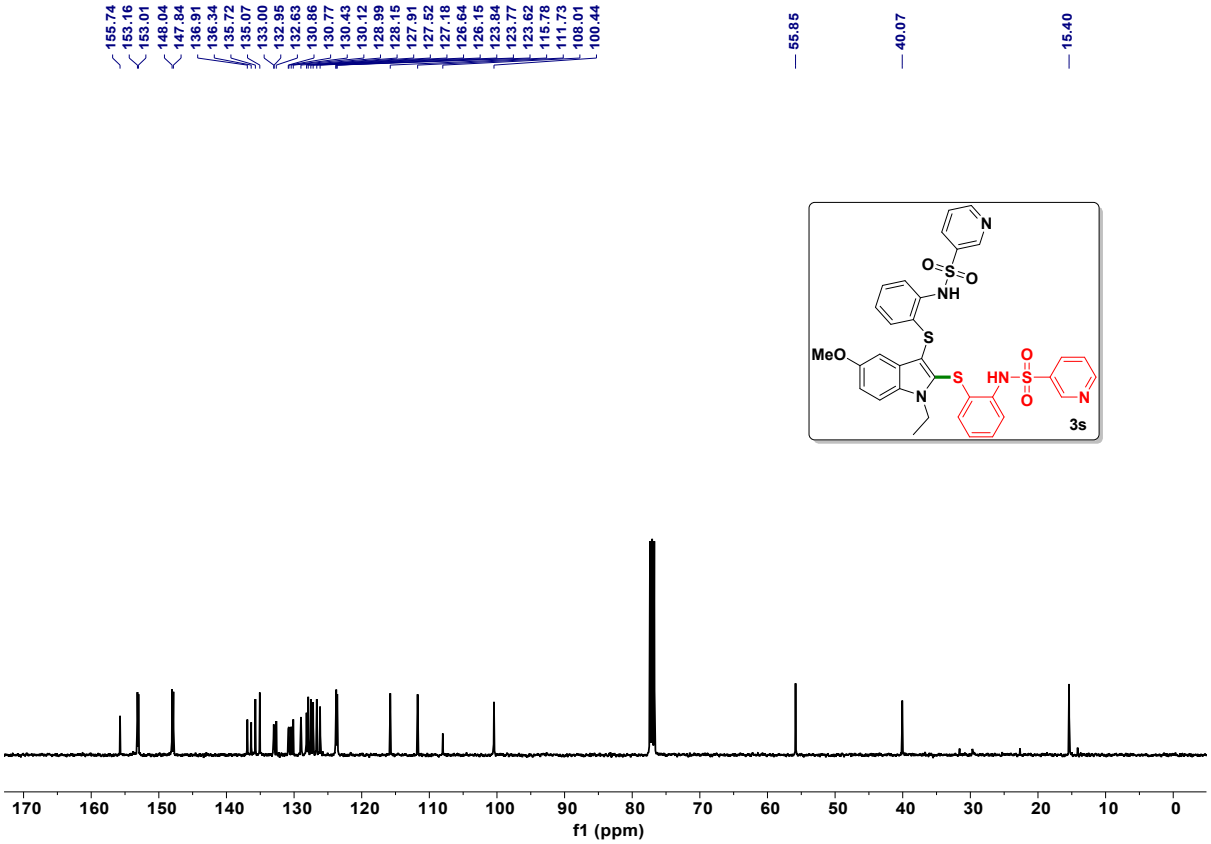


Figure S98. $^{13}C\{^{1}H\}$ NMR of compound **3s** (100 MHz, $CDCl_3$)

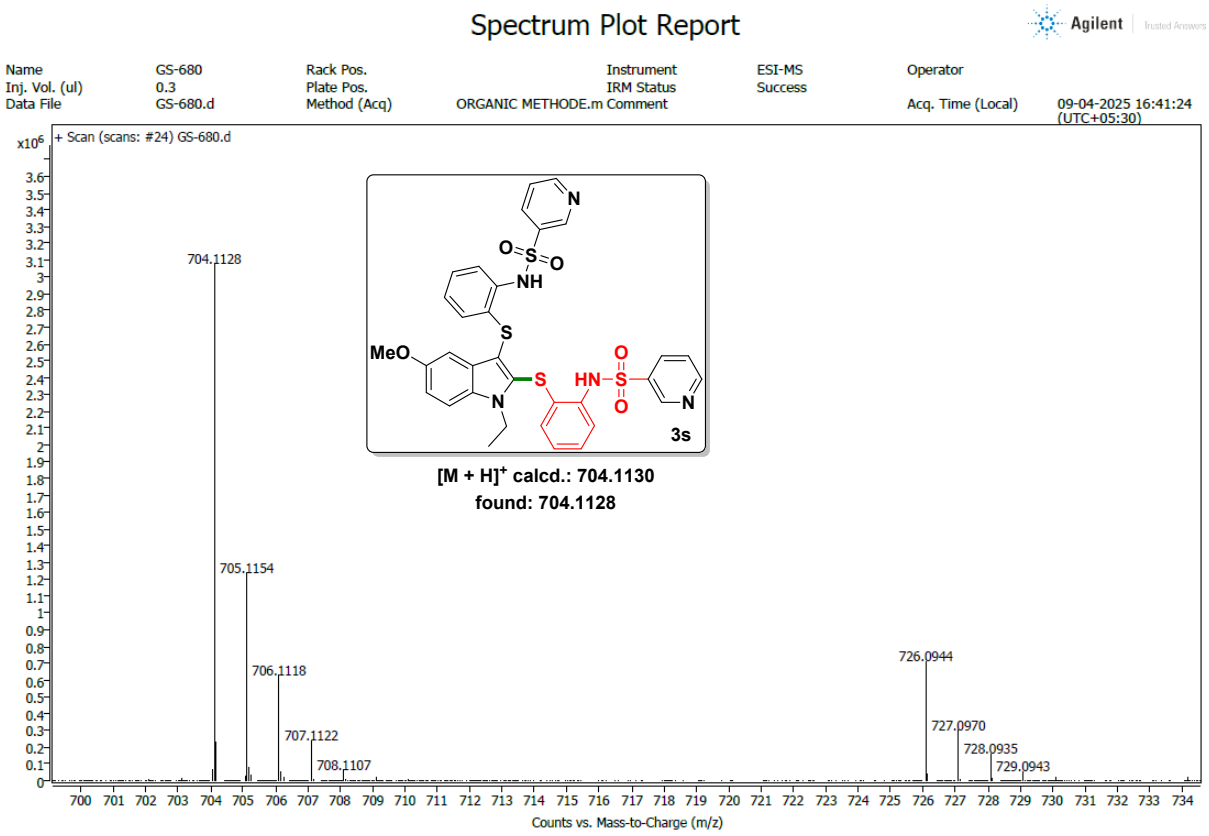


Figure S99. HRMS of compound **3s**

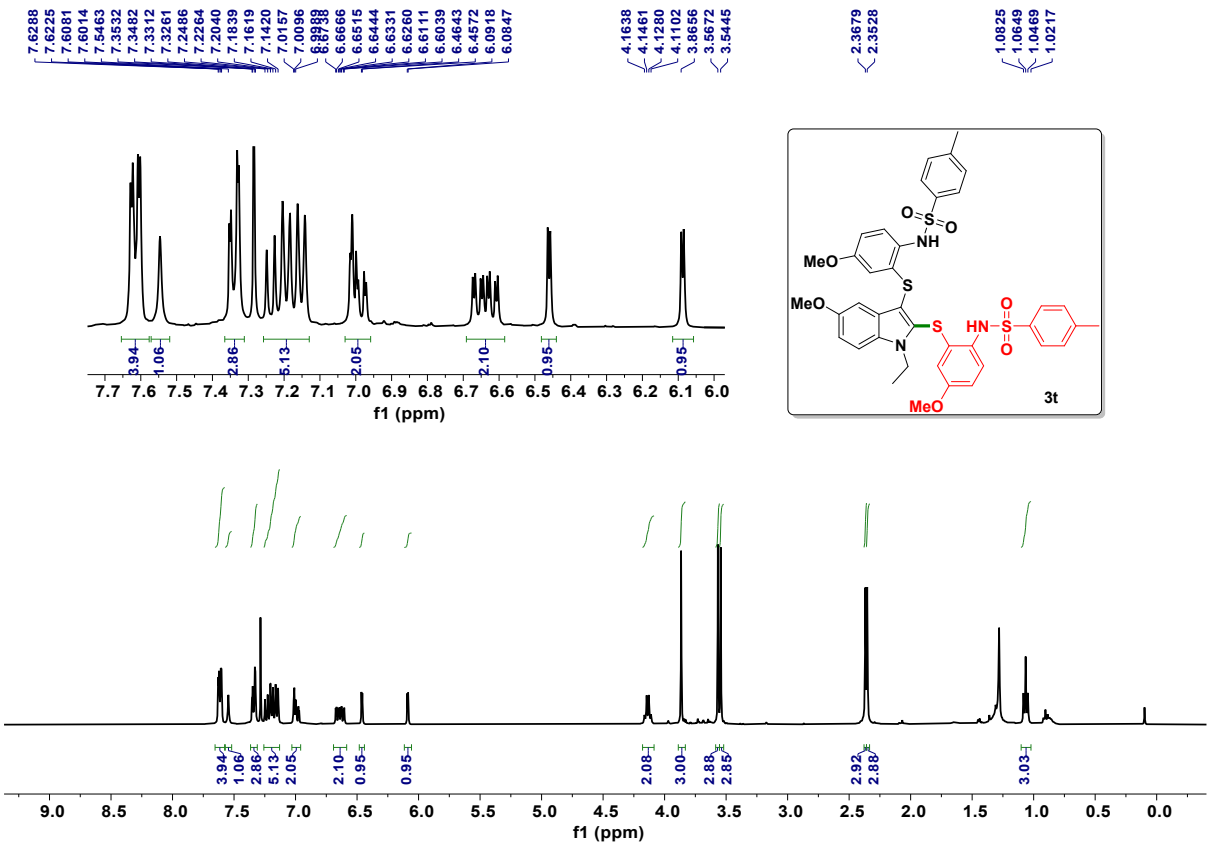


Figure S100. ^{1}H NMR of compound **3t** (400 MHz, $CDCl_3$)

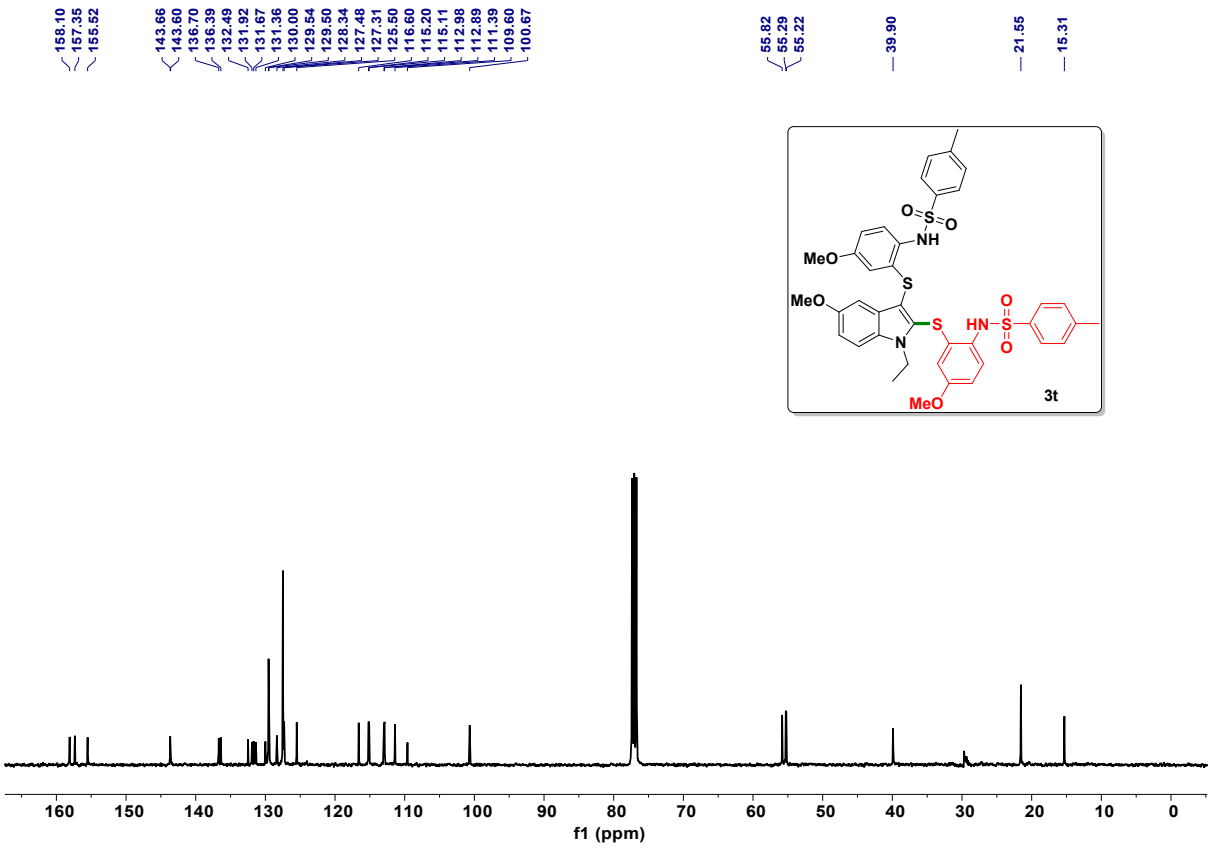


Figure S101. $^{13}C\{^{1}H\}$ NMR of compound **3t** (100 MHz, $CDCl_3$)

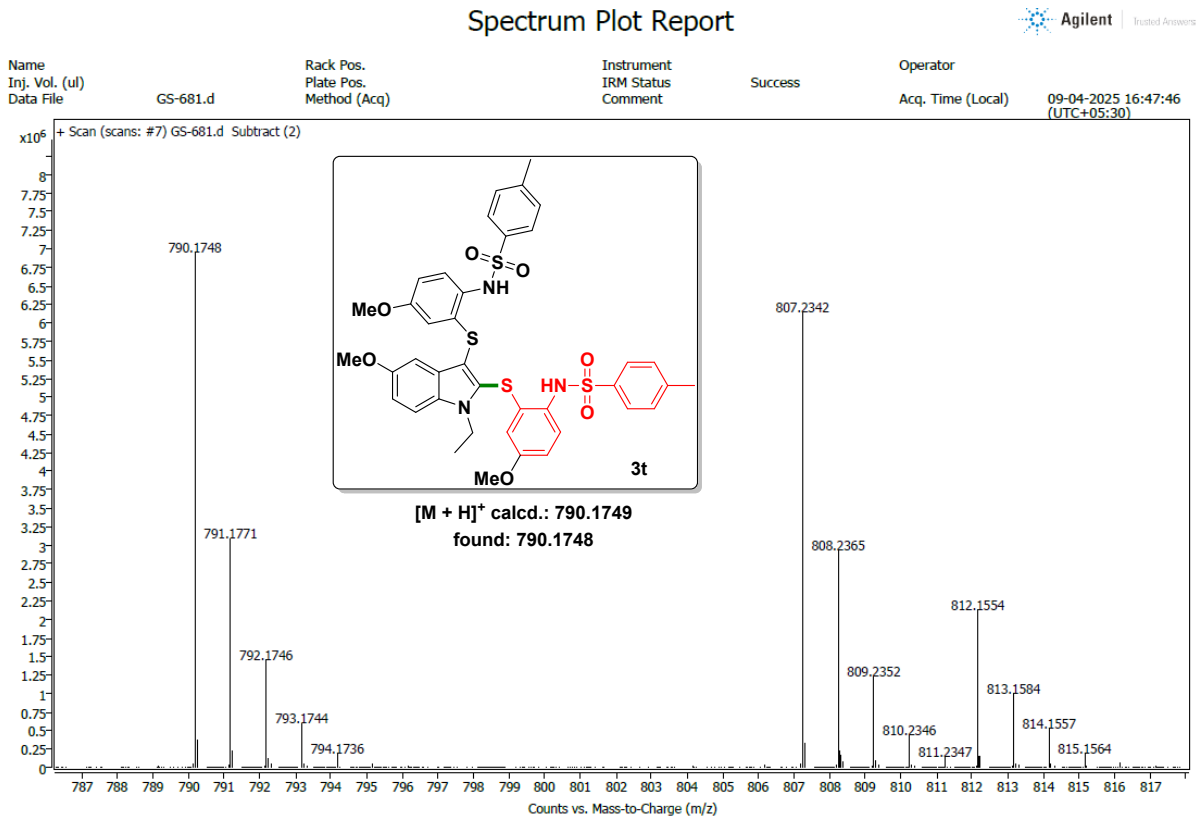


Figure S102. HRMS of compound **3t**

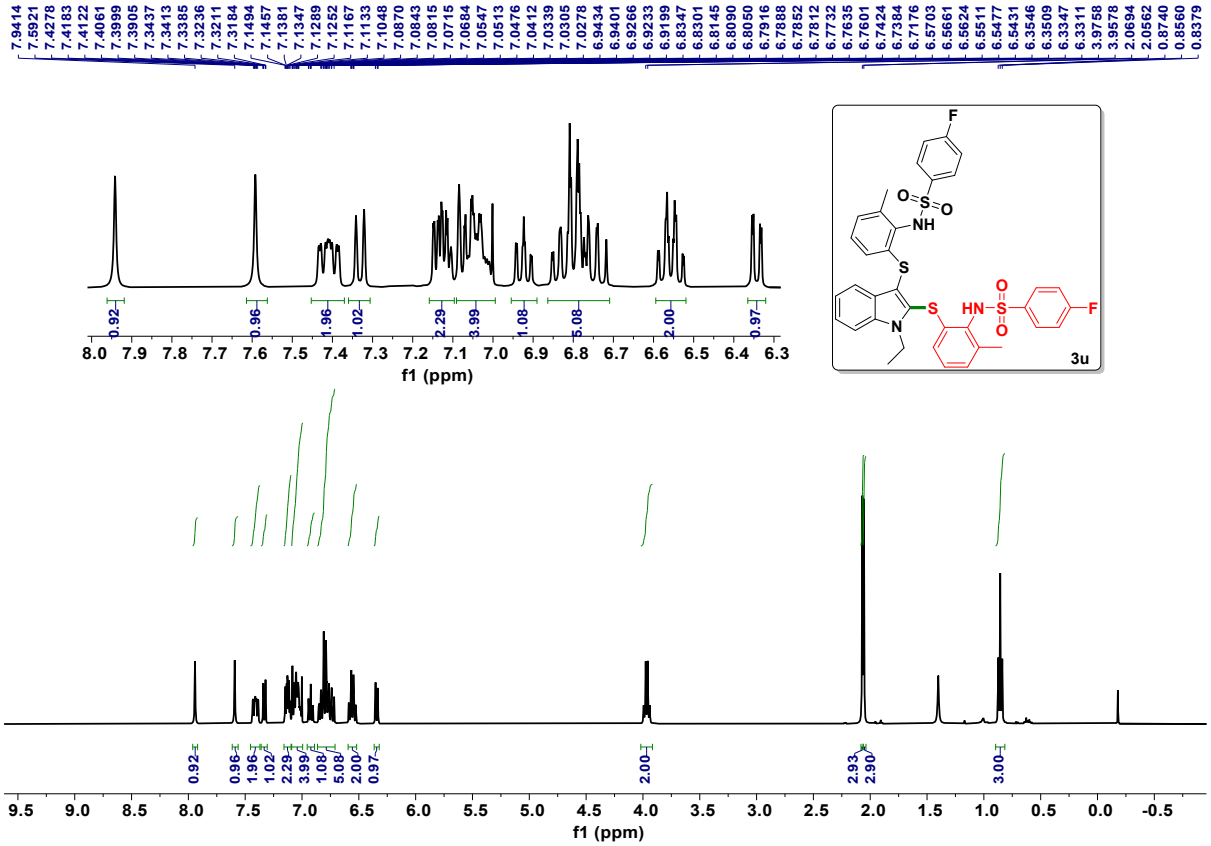


Figure S103. ^{1}H NMR of compound **3u** (400 MHz, $CDCl_3$)

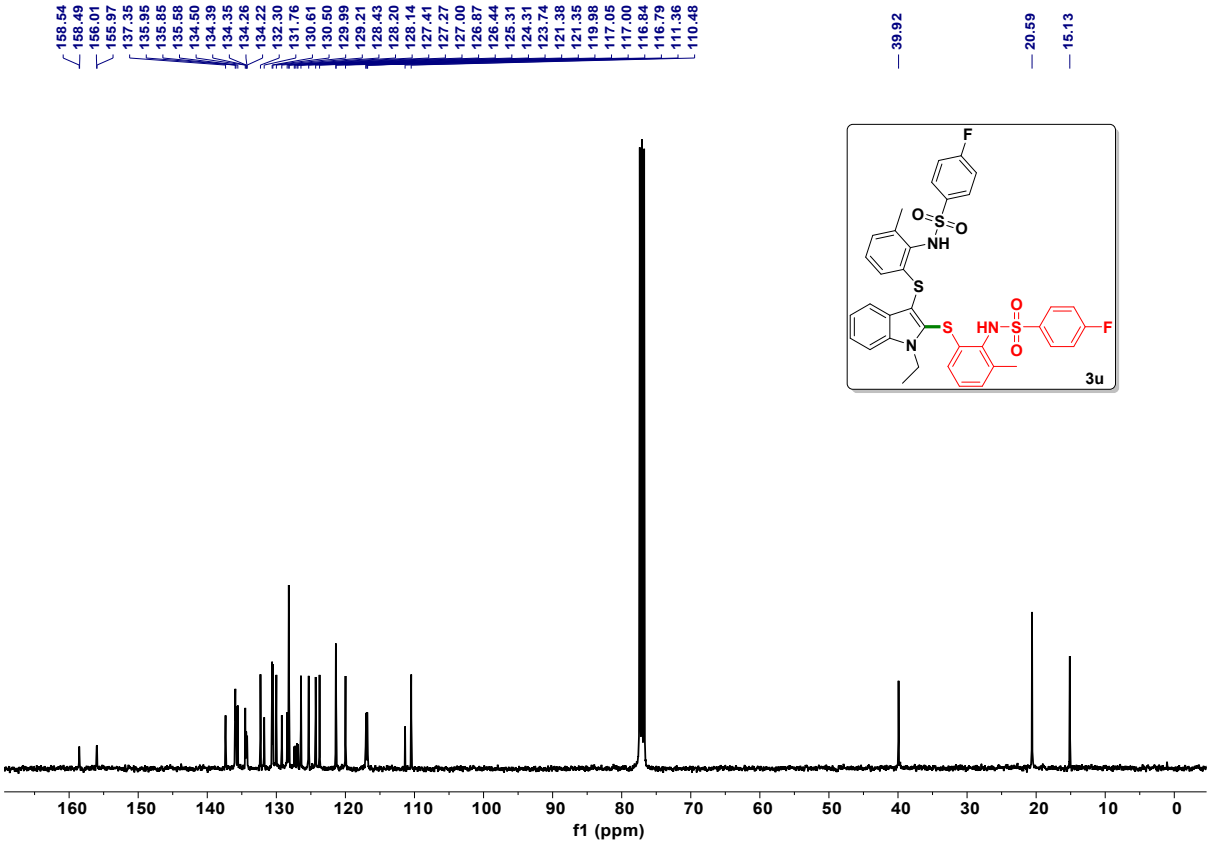


Figure S104. ^{13}C{^{1}H} NMR of compound **3u** (100 MHz, $CDCl_3$)

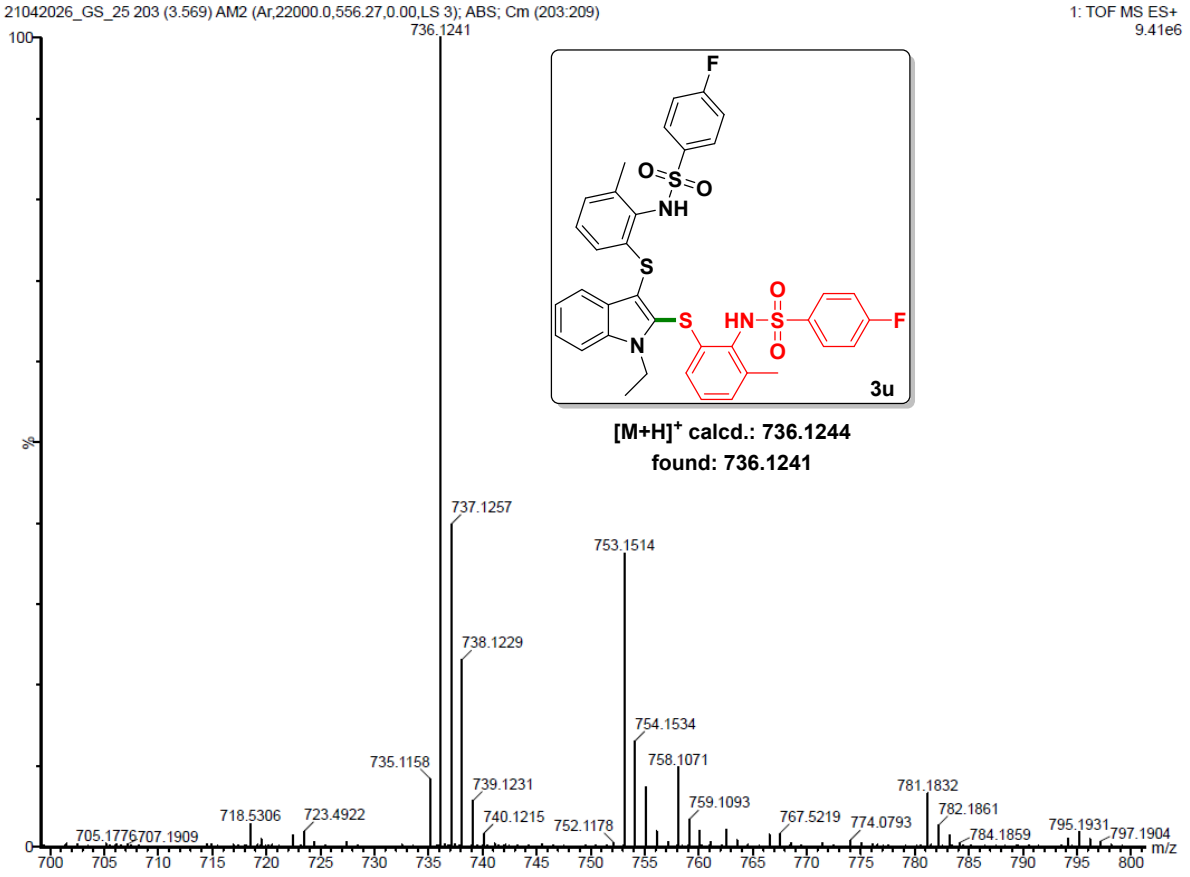


Figure S105. HRMS of compound **3u**

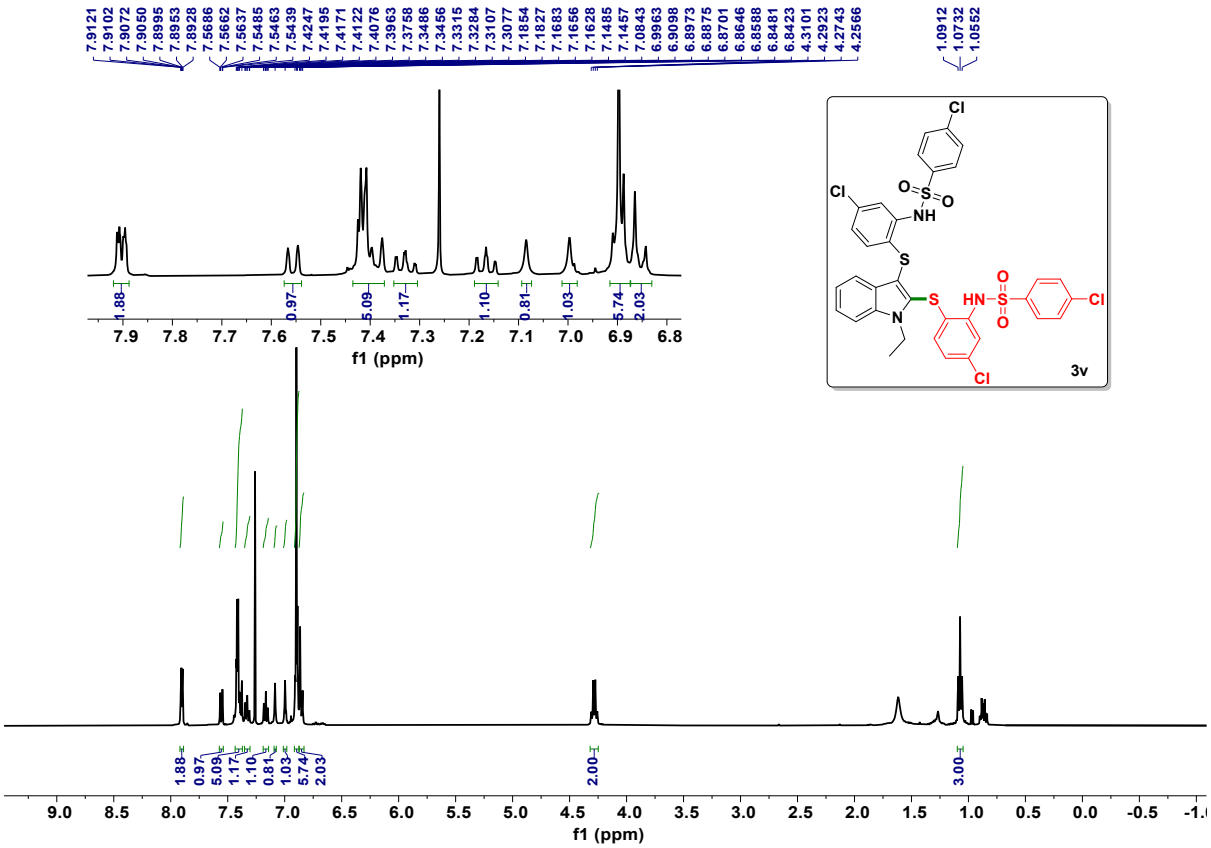


Figure S106. ^{1}H NMR of compound **3v** (400 MHz, $CDCl_3$)

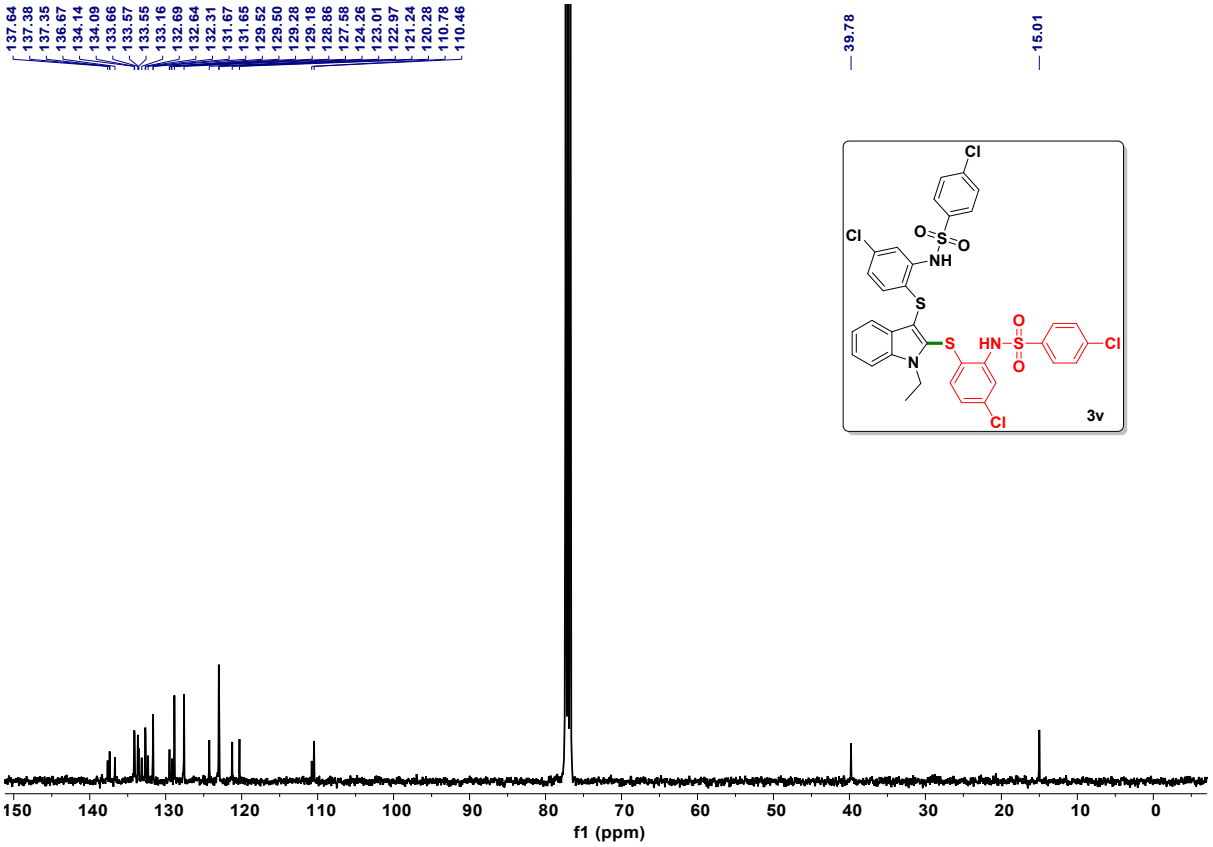


Figure S107. $^{13}C\{^{1}H\}$ NMR of compound **3v** (100 MHz, $CDCl_3$)

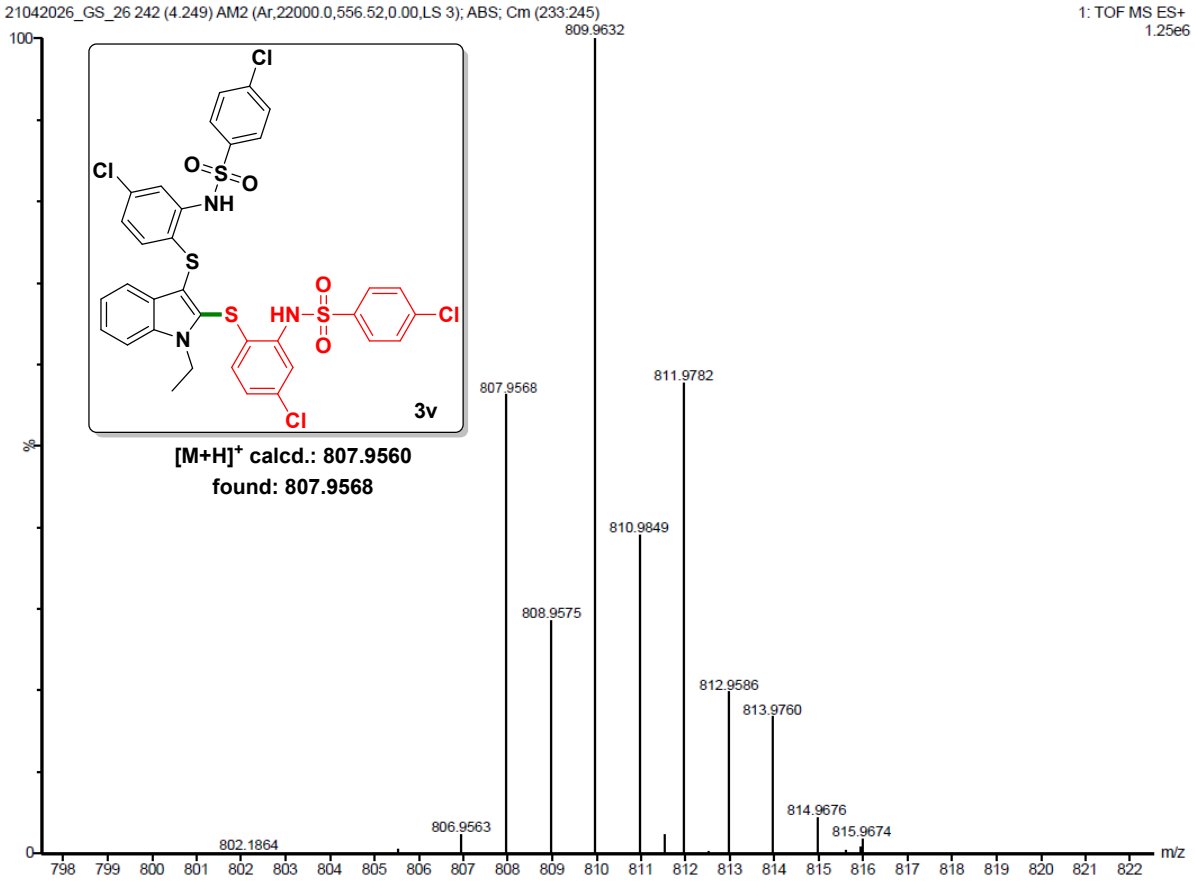


Figure S108. HRMS of compound **3v**

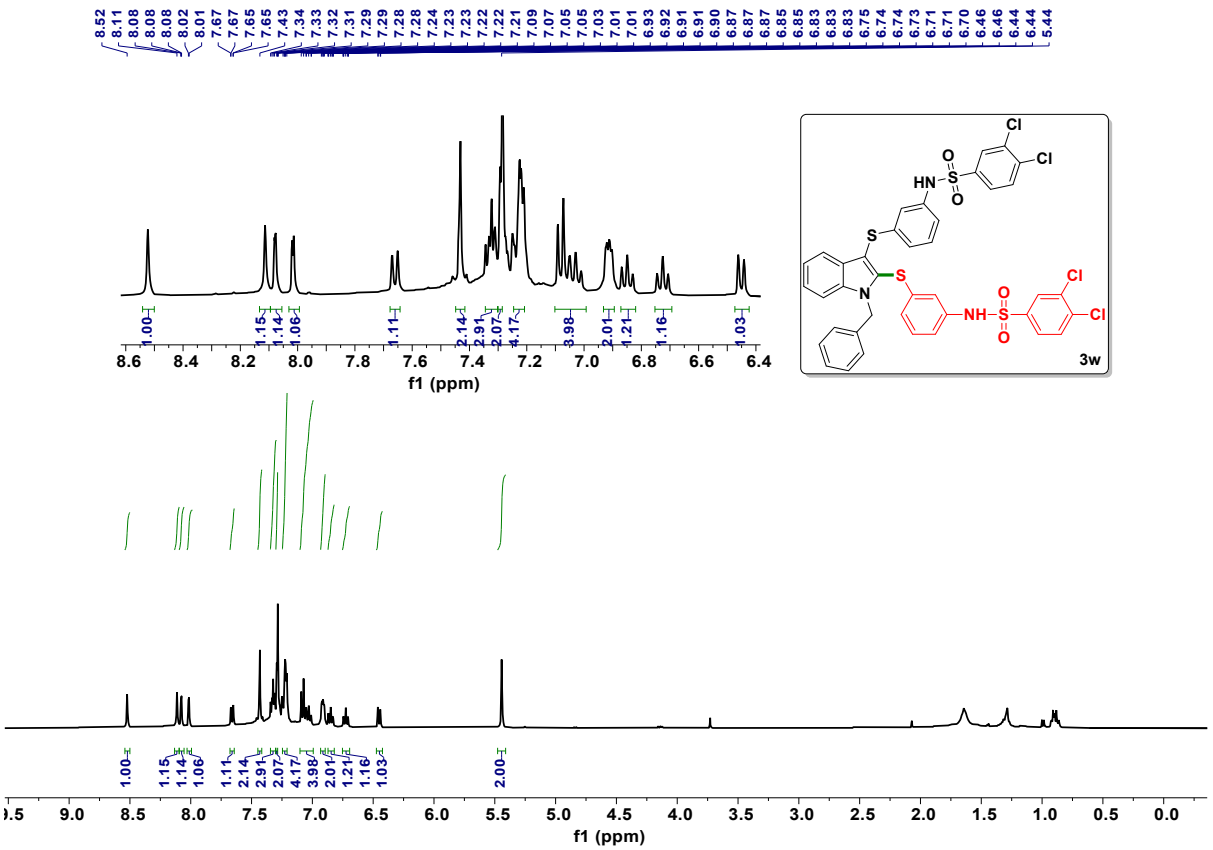


Figure S109. ^{1}H NMR of compound **3w** (400 MHz, $CDCl_3$)

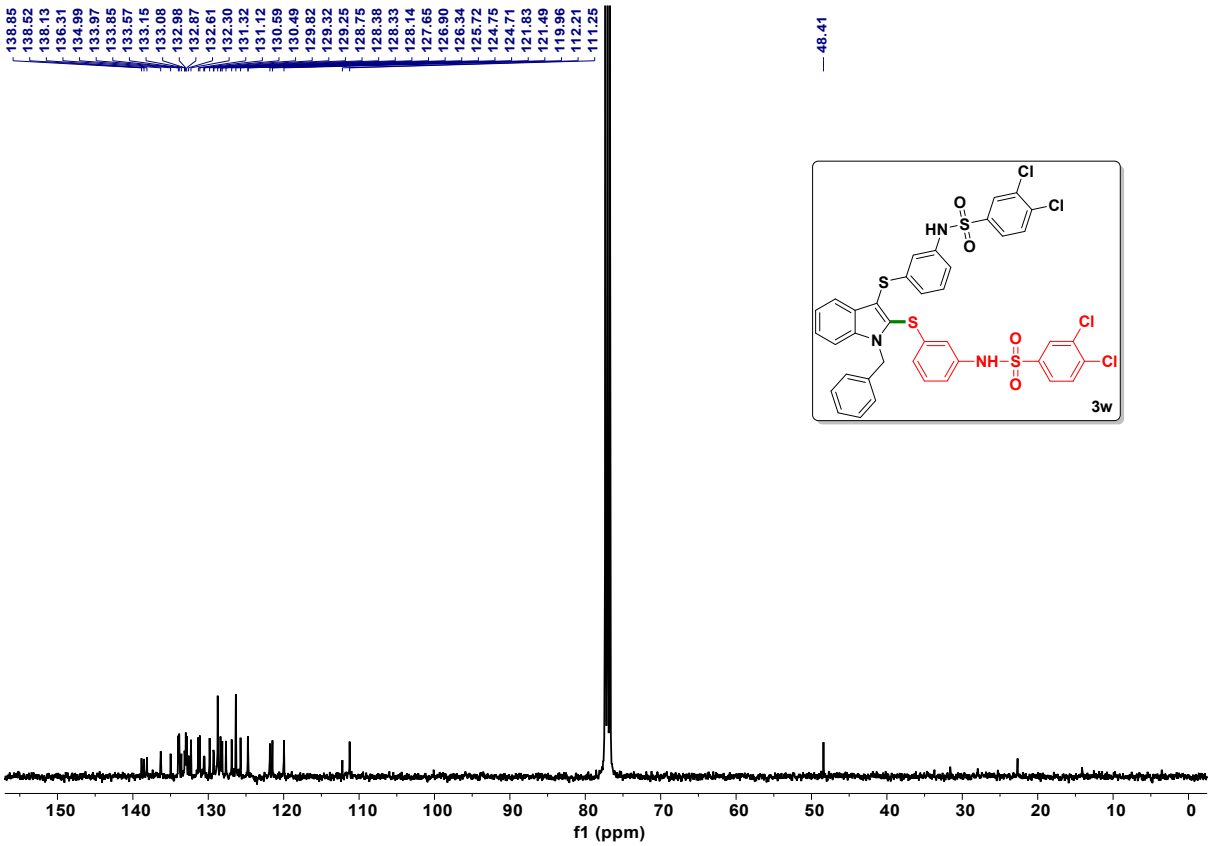


Figure S110. $^{13}C\{^{1}H\}$ NMR of compound **3w** (100 MHz, $CDCl_3$)

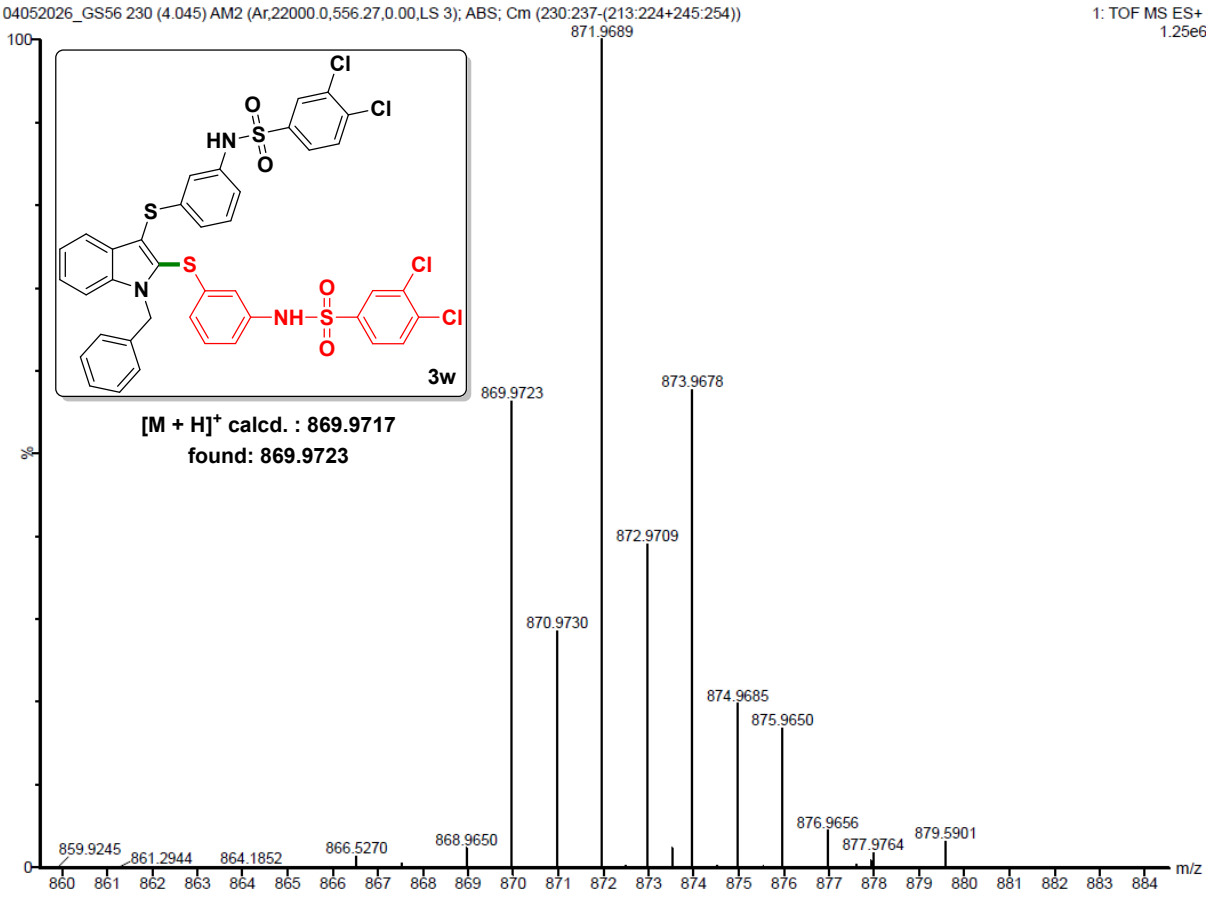


Figure S111. HRMS of compound **3w**

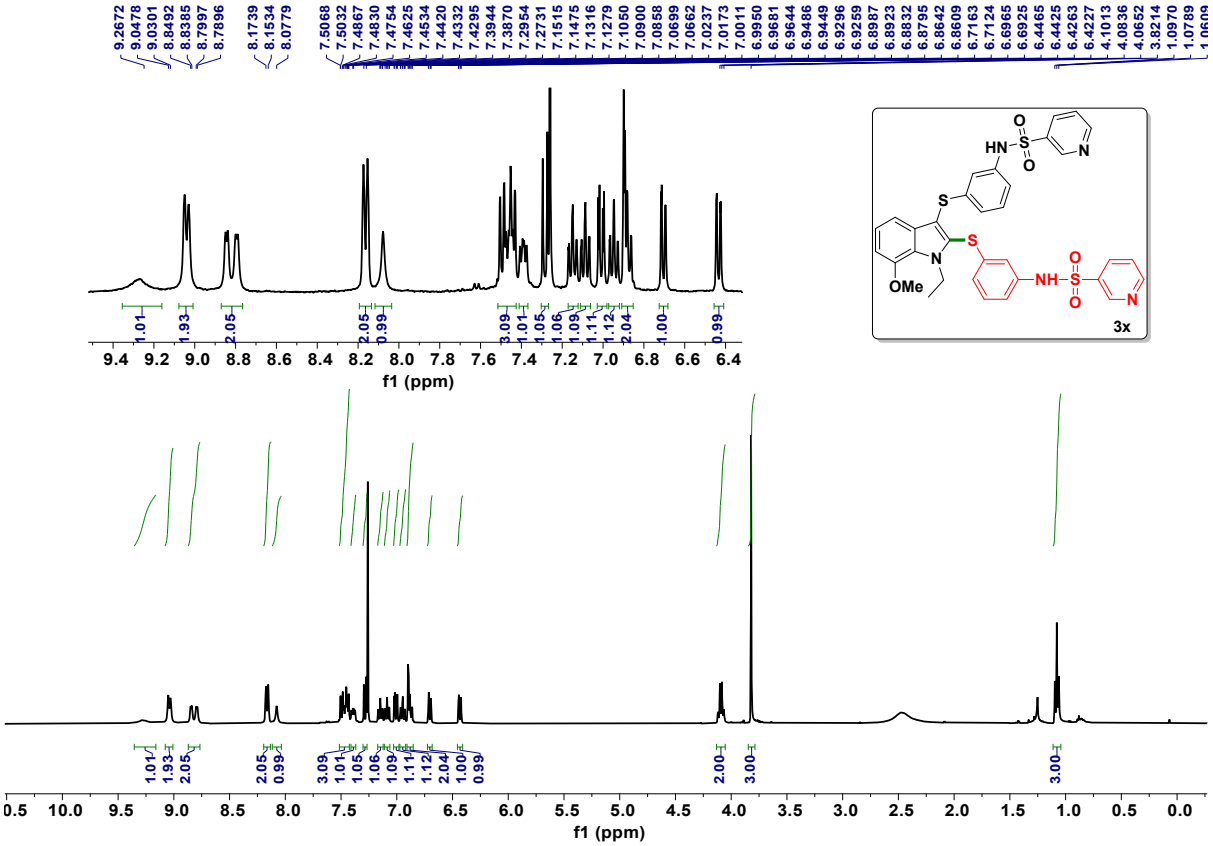


Figure S112. ^{1}H NMR of compound **3x** (400 MHz, $CDCl_3$)

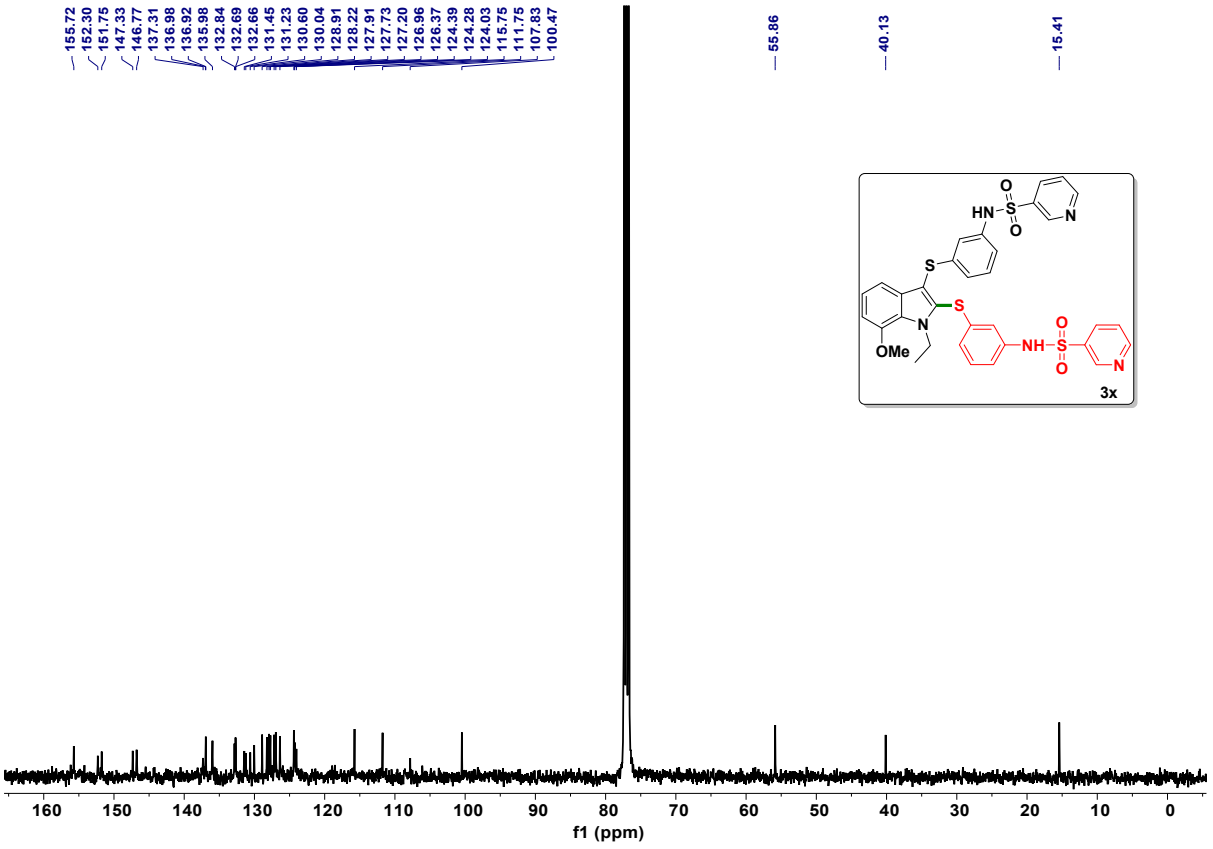


Figure S113. $^{13}C\{^{1}H\}$ NMR of compound **3x** (100 MHz, $CDCl_3$)

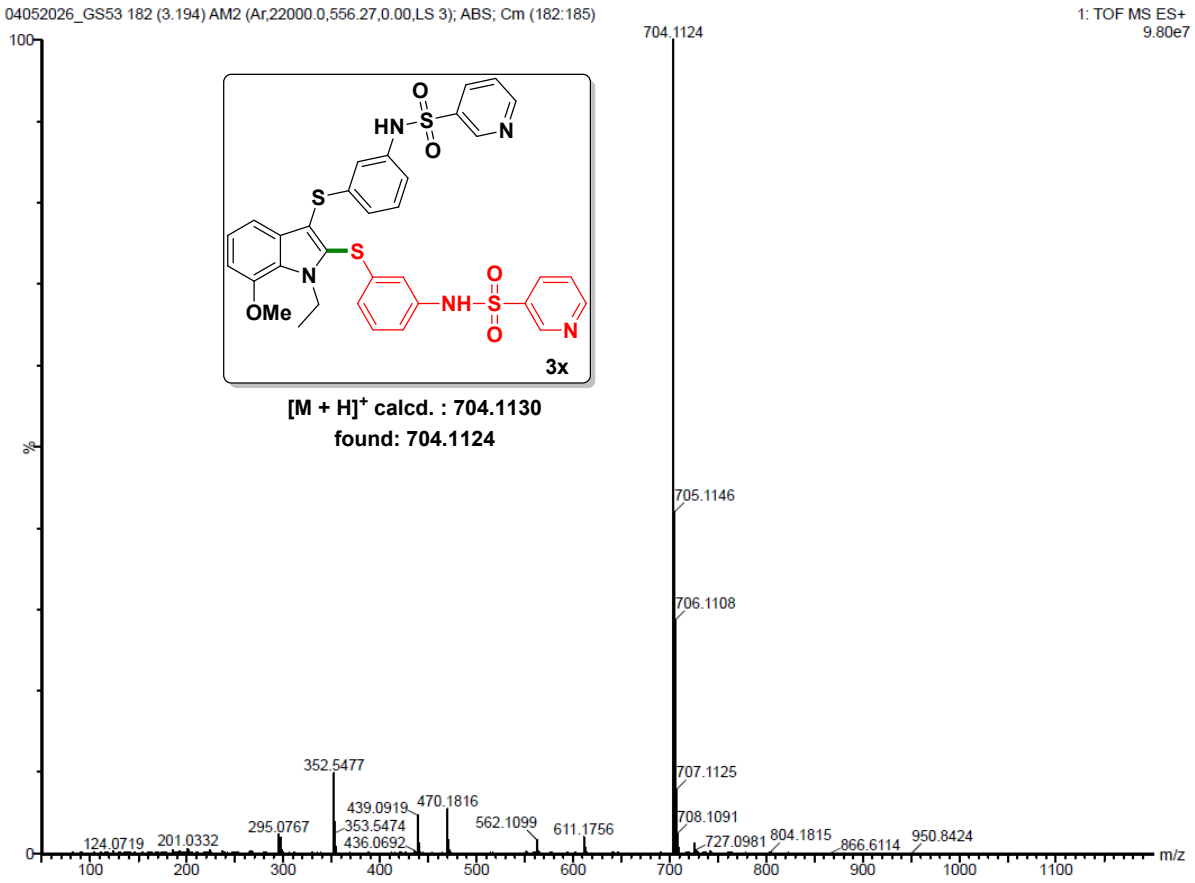


Figure S114. HRMS of compound **3x**

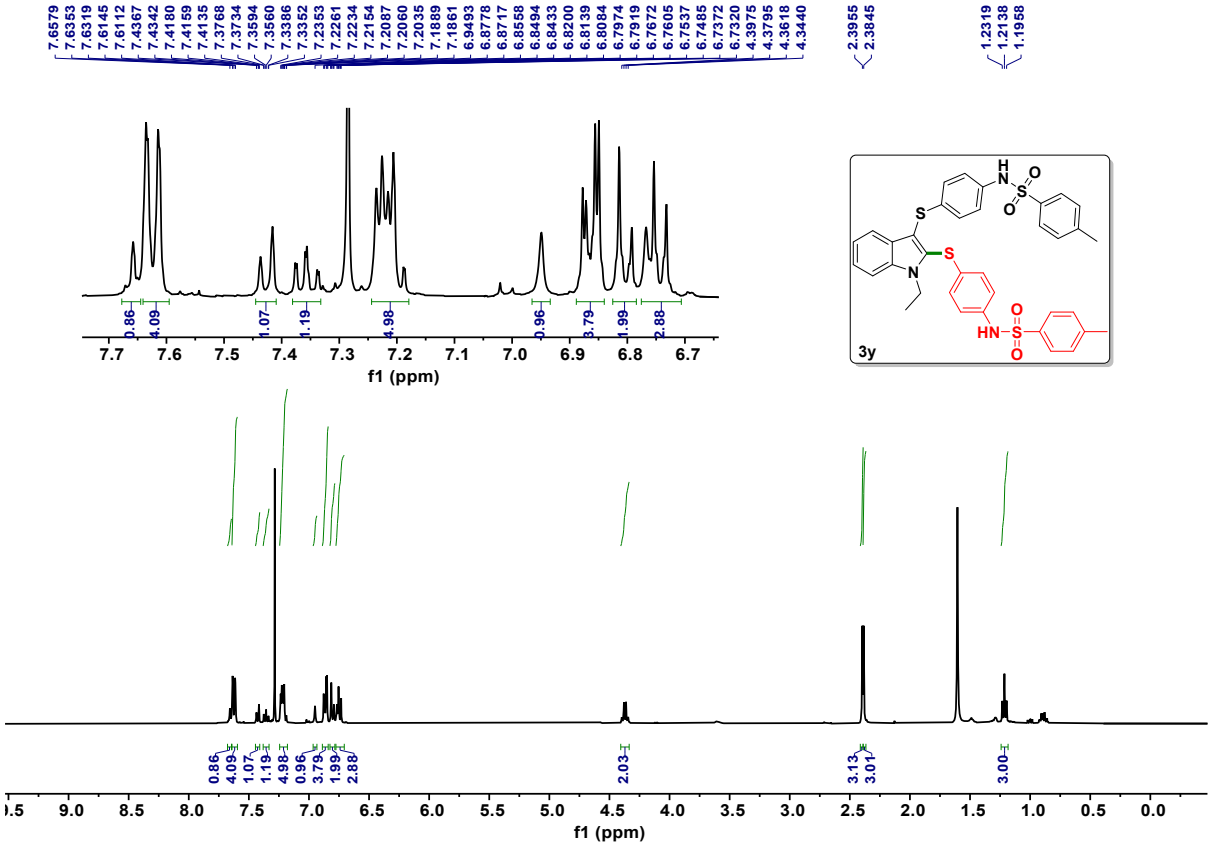


Figure S115. ^{1}H NMR of compound **3y** (400 MHz, $CDCl_3$)

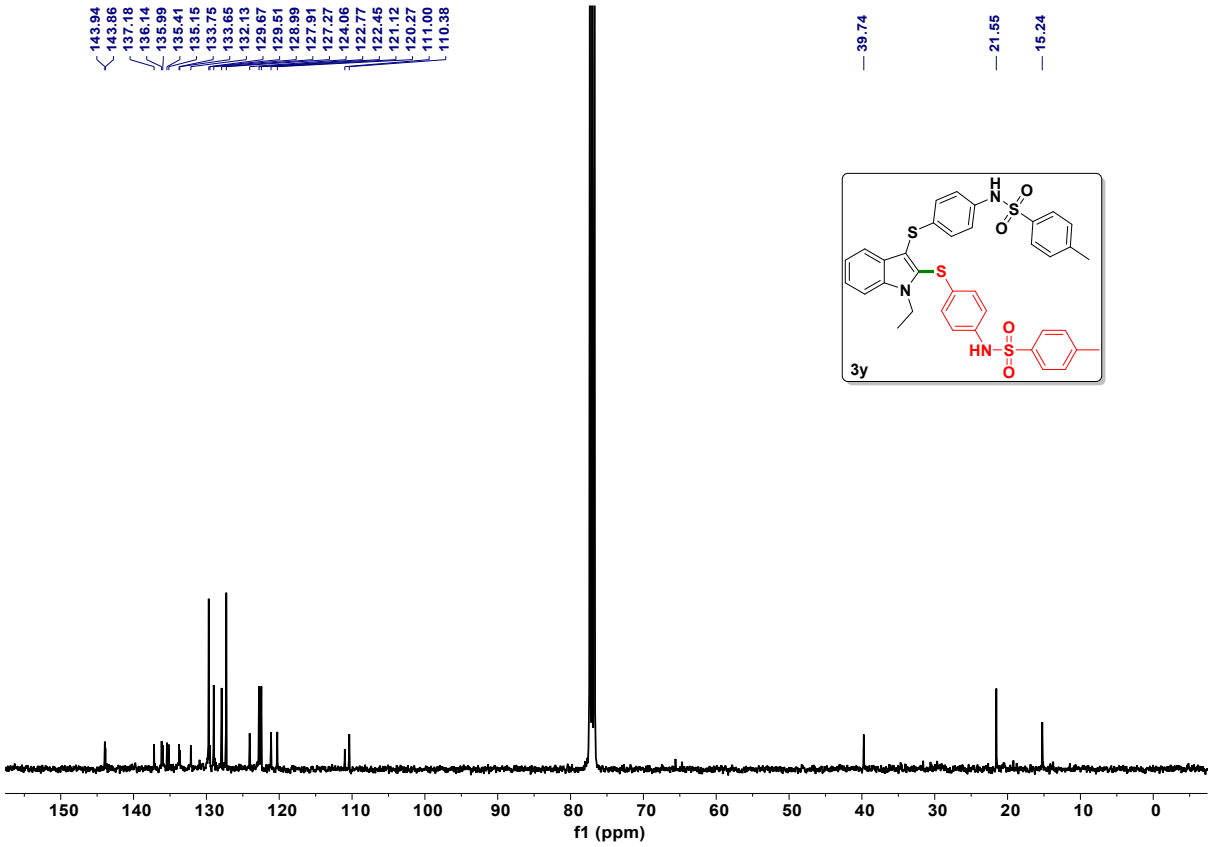


Figure S116. $^{13}C\{^{1}H\}$ NMR of compound **3y** (100 MHz, $CDCl_3$)

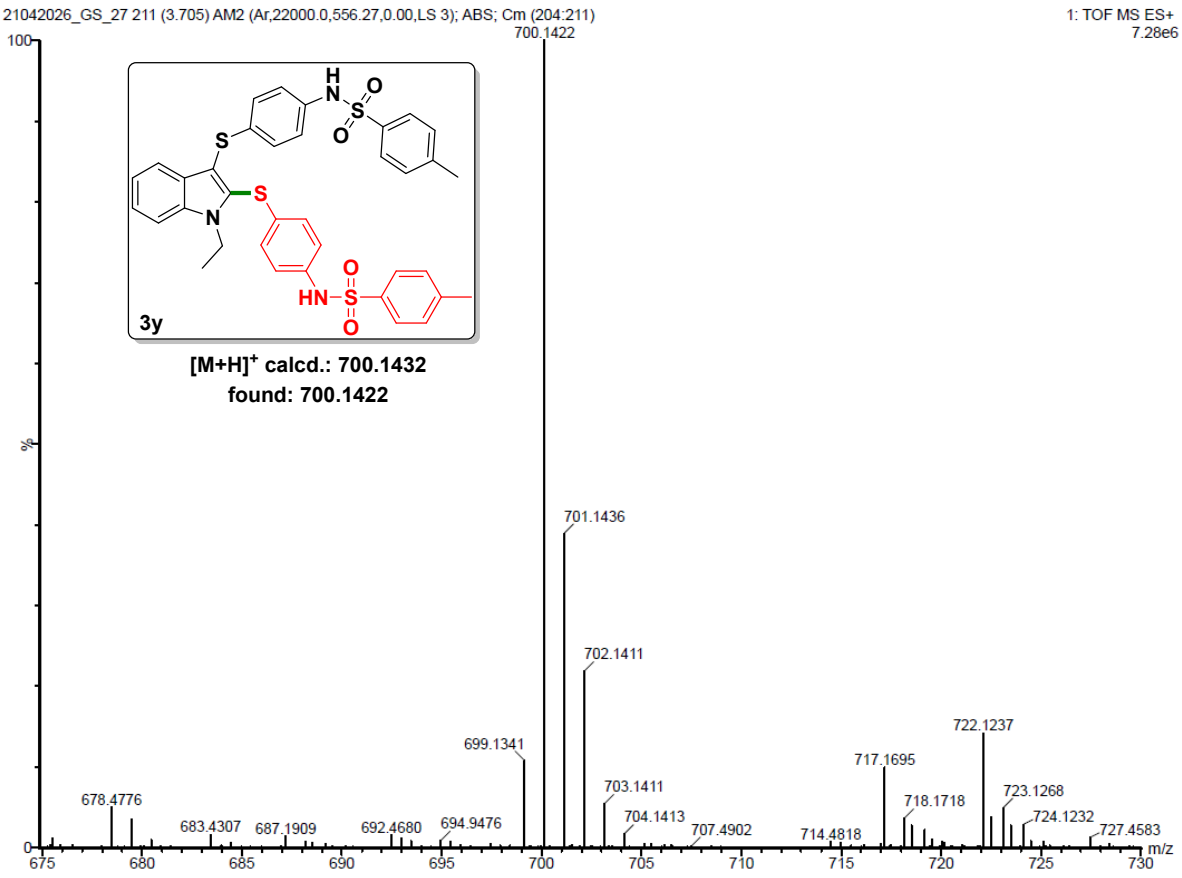


Figure S117. HRMS of compound **3y**

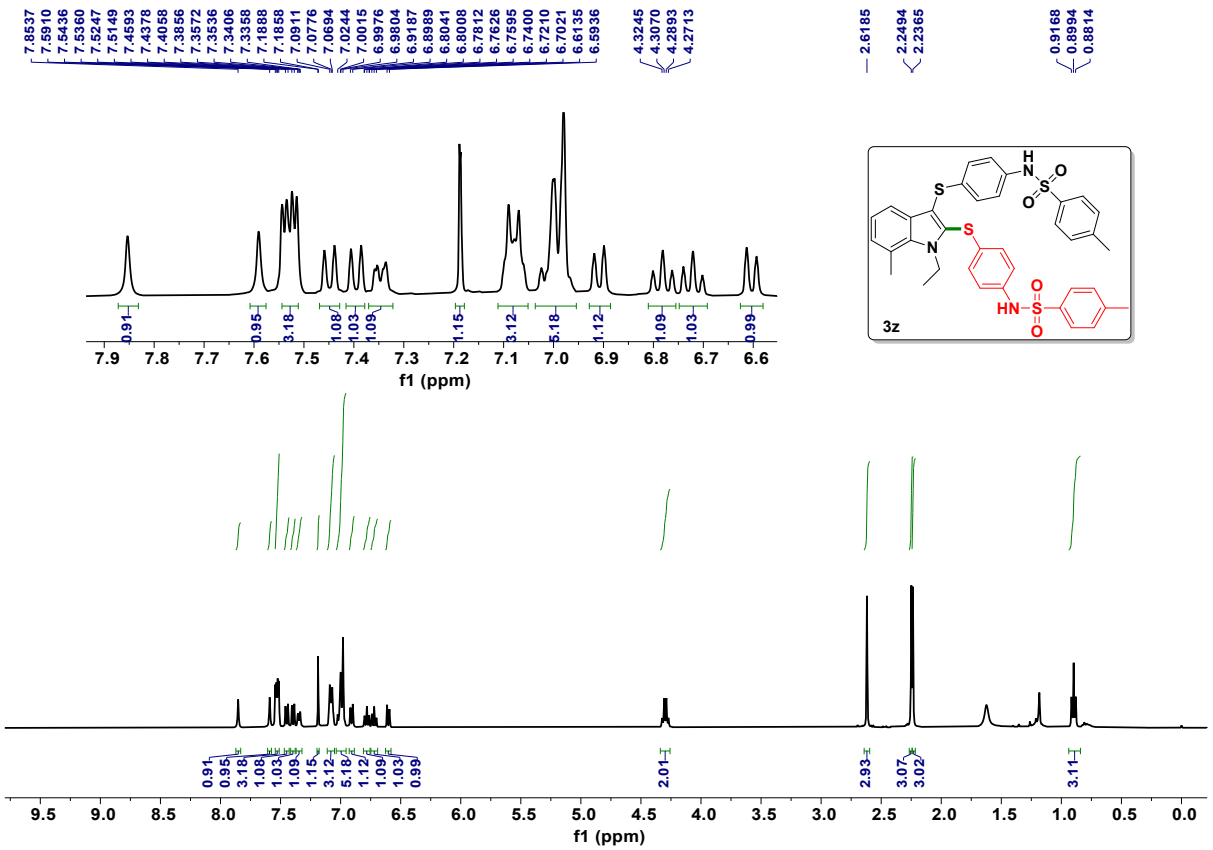


Figure S118. ^{1}H NMR of compound **3z** (400 MHz, $CDCl_3$)

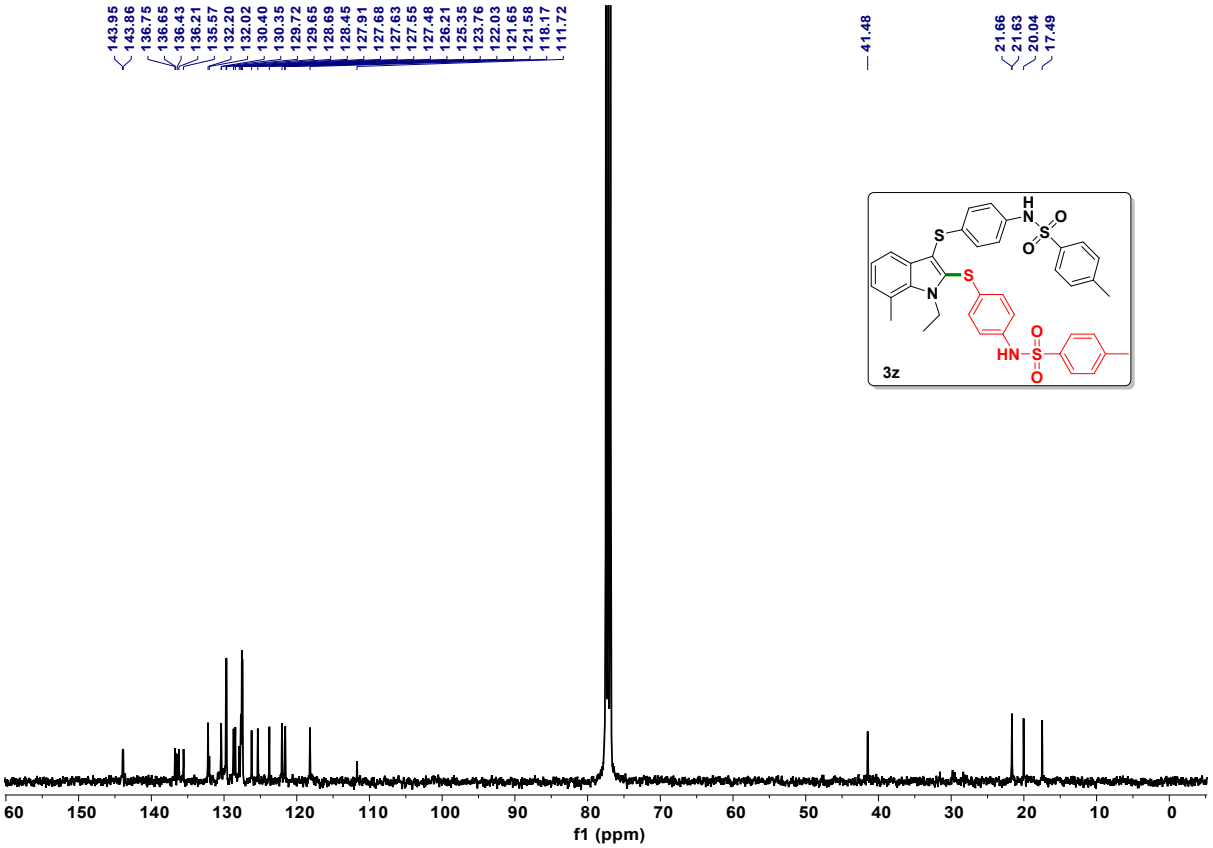


Figure S119. $^{13}C\{^{1}H\}$ NMR of compound **3z** (100 MHz, $CDCl_3$)

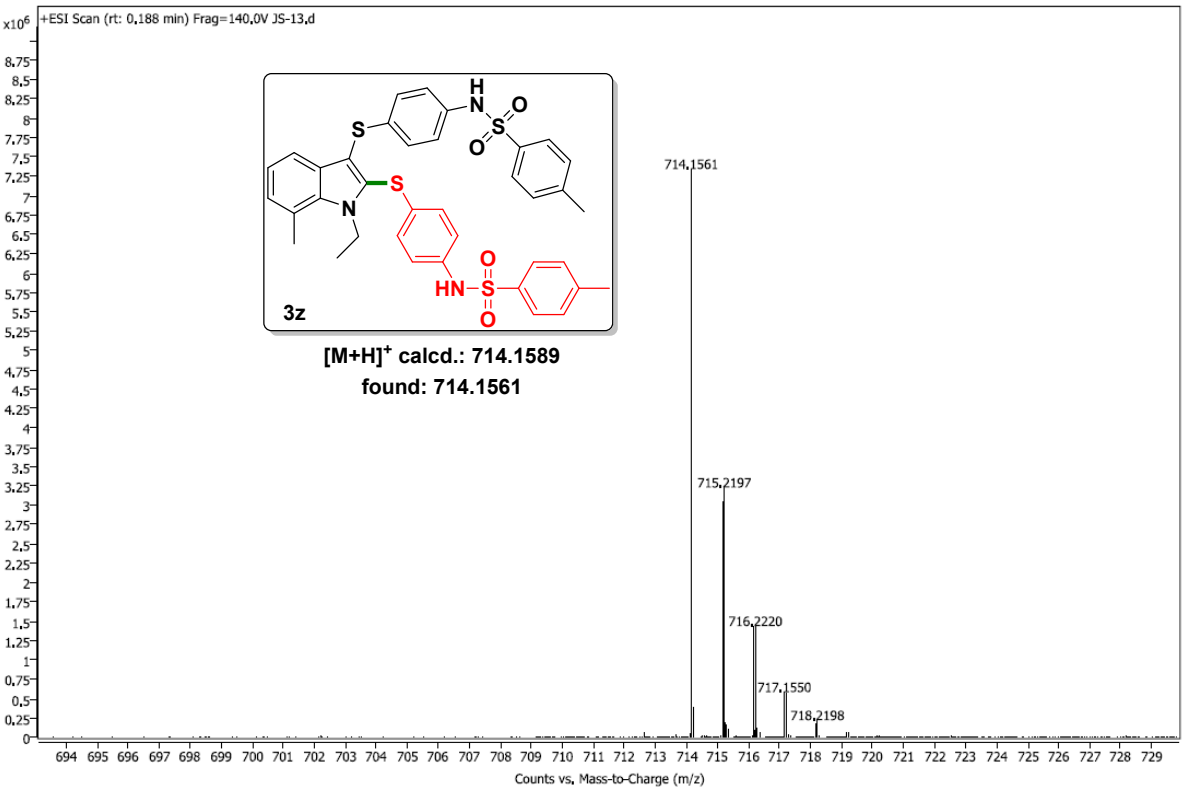


Figure S120. HRMS of compound **3z**

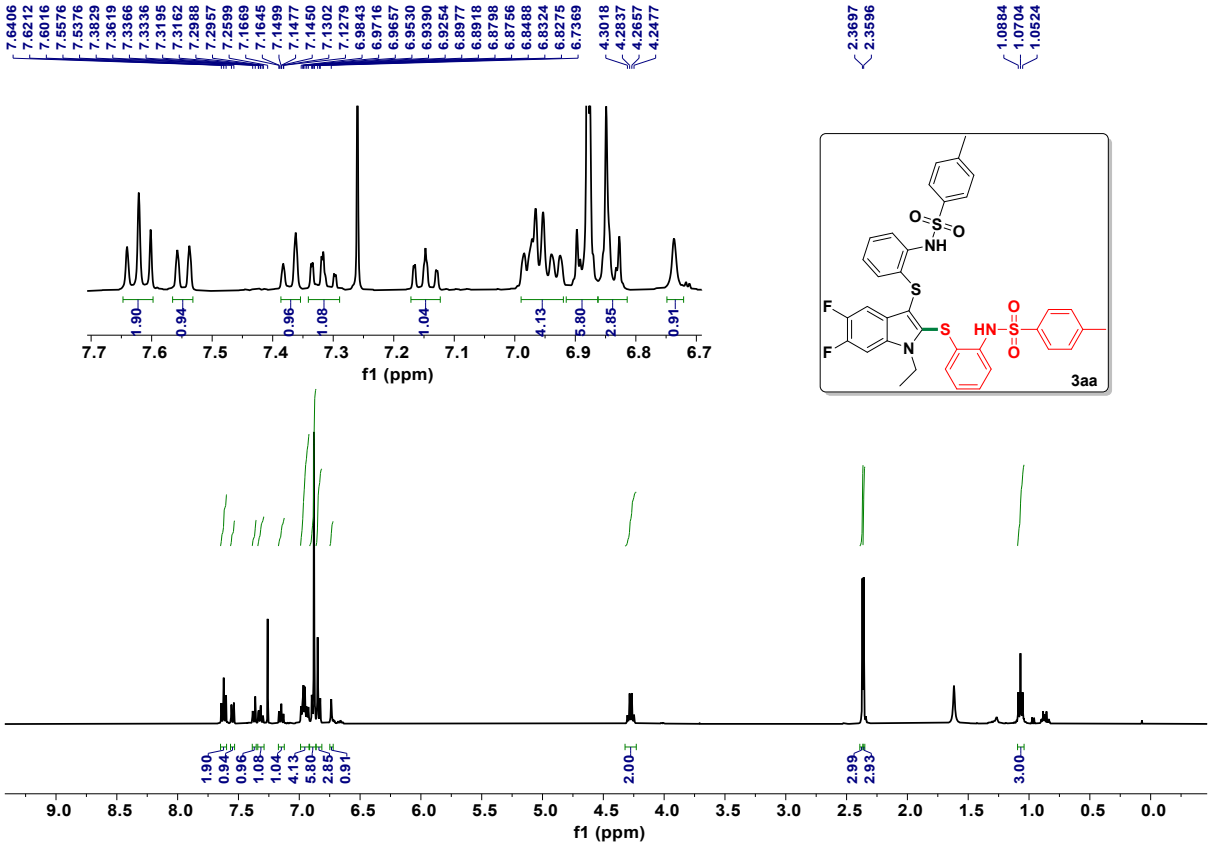


Figure S121. ^{1}H NMR of compound **3aa** (400 MHz, $CDCl_3$)

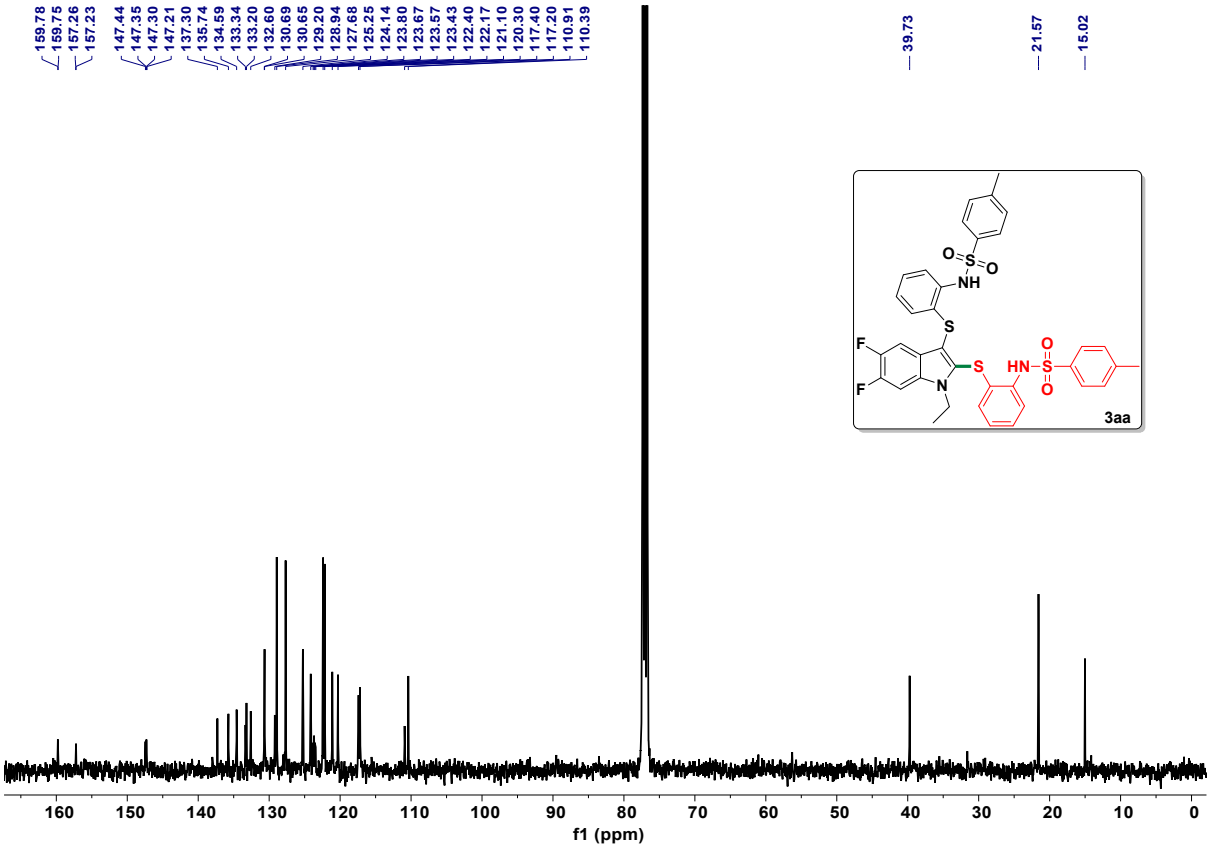


Figure S122. $^{13}C\{^{1}H\}$ NMR of compound **3aa** (100 MHz, $CDCl_3$)

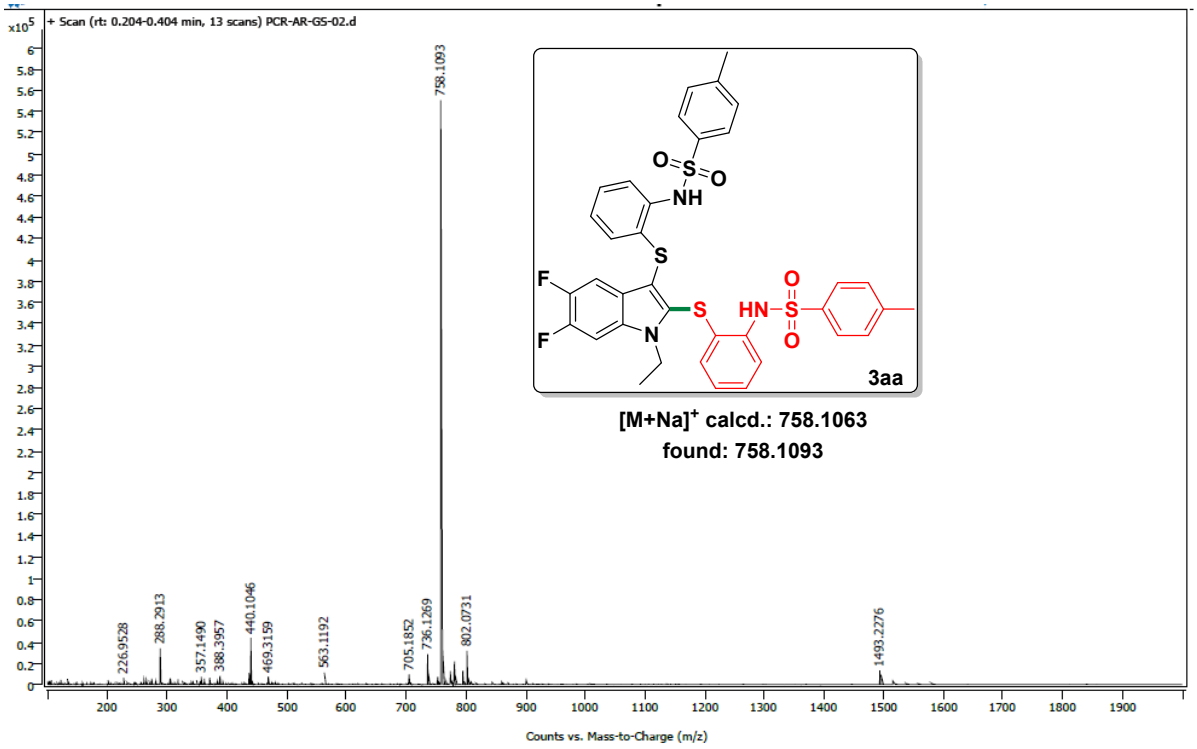


Figure S123. HRMS of compound **3aa**

Crystal growing procedure:

Compound **3a** was crystallized using the slow solvent evaporation method. A minimal amount of ethyl acetate was used to dissolve the compound, and the resulting solution was transferred to a glass vial. The vial was sealed and left undisturbed at room temperature, allowing the solvent to gradually evaporate and form crystals.

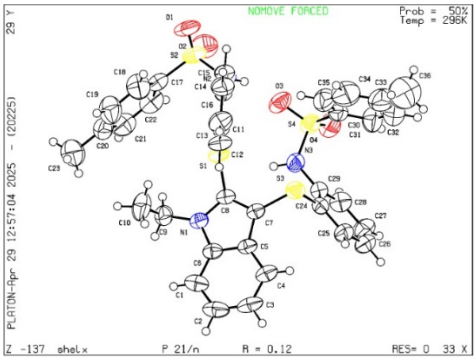


Figure S124. Single-crystal x-ray structure of **3a**. The thermal ellipsoid is scaled to the 50% probability level (CCDC 2531523)

Table S1. Crystal data and structure refinement for ex452gs.

Identification code	shelx
Empirical formula	$C_{36}H_{33}N_3O_4S_4$
Formula weight	699.89
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P 21/n
Unit cell dimensions	a = 13.1897(13) A alpha = 90 deg.
	b = 17.432(2) A beta = 107.838(4) deg.
	c = 17.3845(19) A gamma = 90 deg.
Volume	3805.0(7) A^3
Z, Calculated density	4, 1.222 Mg/m^3
Absorption coefficient	0.289 mm^-1
F(000)	1464
Crystal size	0.300 x 0.200 x 0.200 mm
Theta range for data collection	1.697 to 28.528 deg.
Limiting indices	-17<=h<=16, -23<=k<=23, -22<=l<=22
Reflections collected / unique	44463 / 8821 [R(int) = 0.0697]
Completeness to theta =	25.242 99.9 %
Absorption correction	Semi-empirical from equiv.alents
Max. and min. transmission	0.944 and 0.918
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8821 / 0 / 427
Goodness-of-fit on F^2	1.393
Final R indices [I>2sigma(I)]	R1 = 0.1176, wR2 = 0.3054
R indices (all data)	R1 = 0.2032, wR2 = 0.3596
Extinction coefficient	n/a
Largest diff. peak and hole	3.472 and -0.490 e.A^-3