

Supporting Information

Regioselective/Electro-oxidative Intermolecular [3+2] Annulation for the Preparation of Indolines

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1. General information

All glassware was oven dried at 110°C for hours and cooled down under vacuum. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. The instrument for electrolysis is dual display potentiostat (DJS-292B) (made in China). Cyclic voltammograms were obtained on a CHI 760E potentiostat. The anode electrode is graphite rod and cathode electrode is platinum electrode (1.5 cm × 1.5 cm × 0.3 mm). Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp. 60-90 °C). GC-MS spectra were recorded on Varian GC MS 3900-2100T or SHIMADZU GC MS-2010. ¹H and ¹³C NMR data were recorded with Bruker Advance III (400 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. All chemical shifts are reported relative to tetramethylsilane and d-solvent peaks (77.00 ppm, chloroform), respectively. EPR spectra were recorded on a Bruker X-band A200 spectrometer. High resolution mass spectra (HRMS) were measured with a Bruker UltiMate 3000 & Compact instrument and accurate masses were reported for the molecular ion + Hydrogen (M+H) or molecular ion + Sodium (M+Na).

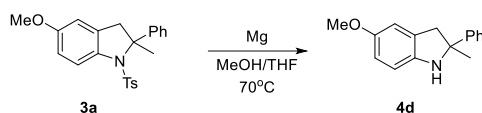
2. Experimental procedure

2.1 General procedure for Regioselective/Electro-oxidative Intermolecular [3+2] Annulation for the Preparation of Indolines

An undivided cell was equipped with a carbon anode and a platinum cathode and connected to a DC regulated power supply. *N*-(4-methoxyphenyl)-4-methylbenzenesulfonamide (0.20 mmol), prop-1-en-2-ylbenzene (0.4 mmol), ⁿBu₄NBF₄ (0.1 M), 2,3-Dichloro-5,6-dicyano-1,4-benzoquinone (0.02 mmol), AcOH (0.2 mmol) and CH₃CN/DCE (4/2 mL) were combined and added. The bottle was equipped with graphite electrode as the anode and platinum electrode (1.5×1.5×0.3 cm³) as the cathode and was then charged with nitrogen. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA under 15 °C for 2.0 h. When the reaction was finished, the solution was extracted with EtOAc (3×10 mL) and H₂O (3×10 mL). The combined organic layer was dried with Na₂SO₄, filtered. The solvent was removed with a rotary evaporator. The pure product was obtained by flash chromatography on silica gel using petroleum ether and ethyl acetate as the eluent (10:1).

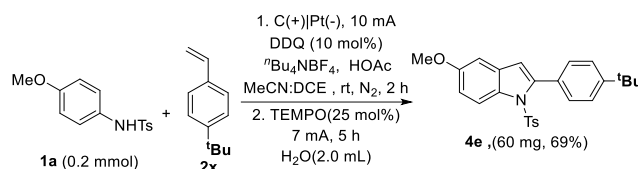
2.2 Procedure for gram scale synthesis: An undivided cell was equipped with a carbon anode and a platinum cathode and connected to a DC regulated power supply. *N*-(4-methoxyphenyl)-4-methylbenzenesulfonamide (5.0 mmol), prop-1-en-2-ylbenzene (10.0 mmol), 2,3-Dichloro-5,6-dicyano-1,4-benzoquinone (0.5 mmol), AcOH (5.0 mmol) and CH₃CN/DCE (40/20 mL) were combined and added. The bottle was equipped with graphite electrode as the anode and platinum electrodes (1.5×1.5×0.3 cm^{mm}) as the cathode and was then charged with nitrogen. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA under 2 °C for 30 h. When the reaction was finished, the solution was extracted with EtOAc (3×50 mL) and H₂O (3×50 mL). The combined organic layer was dried with Na₂SO₄, filtered. The solvent was removed with a rotary evaporator. The pure product was obtained by flash chromatography on silica gel using petroleum ether and ethyl acetate as the eluent (10:1).

2.3 General Procedure for the Deprotection of *N*-Ts Group¹



To a solution of the substrate **3a** (5 mmol, 1 equiv) in MeOH (20 mL) and THF (20 mL) was added Mg (150 mmol, 30 equiv). After being stirred at 70 °C for the reported time, the reaction mixture was cooled to rt. The resulting mixture was filtered through a pad of celite. The filtrate was diluted with water and washed with CH₂Cl₂ (three times). The combined organic layer was washed with brine, dried over Na₂SO₄. The residue was purified by column chromatography on silica gel to give the corresponding product **4d** in 99% yield.

2.4 General Procedure for the One pot two-step process for indole synthesis



N-(4-methoxyphenyl)-4-methylbenzenesulfonamide **1a** (0.20 mmol), 1-(tert-butyl)-4-vinylbenzene **2x** (0.4 mmol), ^tBu₄NBF₄ (0.1 M), 2,3-Dichloro-5,6-dicyano-1,4-benzoquinone (0.02 mmol), AcOH (0.2 mmol) and CH₃CN/DCE (4/2 mL) were combined and added. The bottle was equipped with graphite electrode as the anode and platinum electrode (1.5×1.5×0.3 cm³) as the cathode and was then charged with nitrogen. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA under 25 °C for 2.0 h. Then, 2,2,6,6-Tetramethylpiperidinoxy(TEMPO) (25 mol%, 7.8 mg) and H₂O (2.0 mL) were added to the mixture. The reaction mixture was stirred and electrolyzed at a constant current of 7 mA under 25 °C for 5.0 h. The pure product **4e** was obtained by flash column chromatography on silica gel.

2.5 EPR experiments: EPR spectra was recorded at 298 K on EPR spectrometer operated at 9.816 GHz. Typical spectrometer parameters are shown as follows, scan range: 100 G; center field set: 3503 G; time constant: 163.84 ms; S21 scan time: 30.72 s; modulation amplitude: 0.3 G; modulation frequency: 100 kHz; receiver gain: 1.00×10⁴; microwave power: 10 mW.

EPR studies of 1a in MeCN/DCE: Under constant current conditions, a dried three-necked flask equipped with a stir bar was loaded with **1a** (0.2 mmol), ${}^n\text{Bu}_4\text{NBF}_4$ (0.4 mmol) in 4.0 mL MeCN and 2.0 mL DCE was stirred under N_2 atmosphere at 25 °C. Then, the solution sample was taken out into a small tube and tested by EPR. An EPR signal (the parameters observed for the spin adduct are $\text{AN}=0.54$ G and $3 \text{ AH}=0.54$ G) which was mainly identified as a conjugated radical.

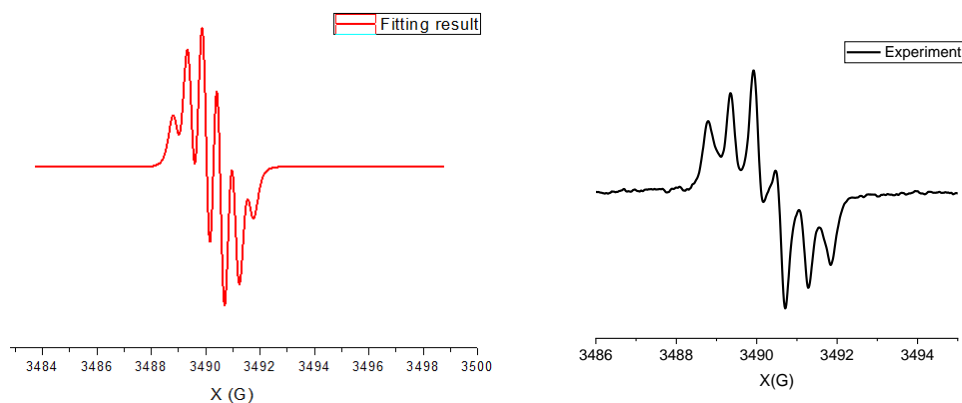


Figure S1. **1a** (0.2mmol), ${}^n\text{Bu}_4\text{NBF}_4$ (0.2 mmol), MeCN (4 mL), DCE (2 mL), undivided cell, constant current, 30 min.

2.6 General procedure for cyclic voltammetry (CV): Cyclic voltammetry was performed in a three-electrode cell connected to a schlenk line under nitrogen at room temperature. The working electrode was a platinum disk electrode, the counter electrode a platinum wire. The reference was an Ag/AgCl electrode submerged in saturated aqueous KCl solution, and separated from reaction by a salt bridge. 6 mL of CH_3CN : DCE containing 0.6 mmol ${}^n\text{Bu}_4\text{NBF}_4$ were poured into the electrochemical cell in all experiments. The scan rate is 0.1 V/s, ranging from 0 V to 2.0 V. The peak potentials vs. Ag/AgCl for used. The oxidation potentials of *N*-(4-methoxyphenyl)-4-methylbenzenesulfonamide (**1a**) and α -methylstyrenes (**2a**) are 1.49 V and higher than 2V, respectively.

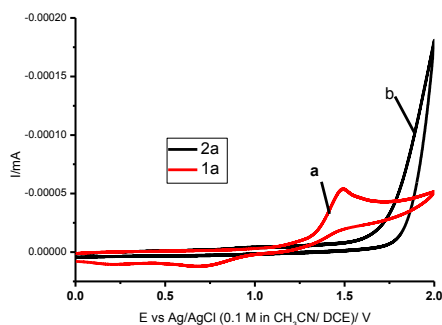


Figure S2. Cyclic voltammograms of **1a** and **2a** in 0.1 M ⁿBu₄NBF₄/CH₃CN: CH₂Cl₂ (2: 1 = v/ v)), using Pt working electrode, Pt wire and Ag/AgCl (0.1 M in CH₃CN) as counter and reference electrode at 100 mV/s scan rate. a: **1a** (1.0 mmol/L), b: **2a** (1.0 mmol/L)

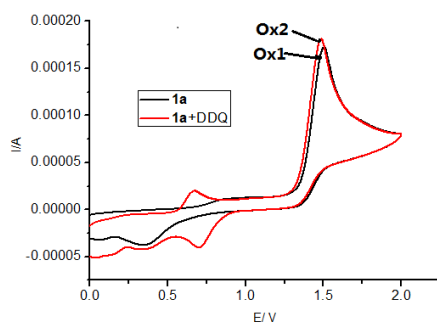


Figure S3. Cyclic voltammograms of **1a** and DDQ in 0.1 M ⁿBu₄NBF₄/CH₃CN: DCE, using glass carbon working electrode, Pt wire and Ag/AgCl (0.1 M in CH₃CN: DCE) as counter and reference electrode at 100 mV/s scan rate.

2.7 Ultraviolet(UV): In order to verify that DDQ did not work in the first step, we carried out the UV experiment (Figure S4.). As shown the Figure S4, when DDQ and **1a** were mixed for 1 minute, no red shift occurred.

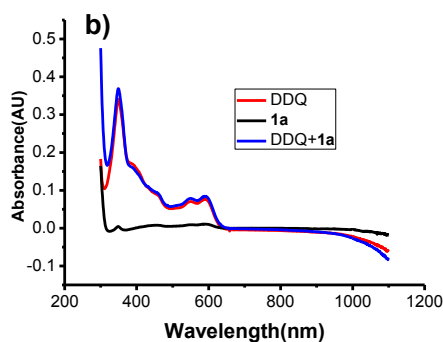


Figure S4. UV experiments of **1a** and DDQ. The red line: DDQ(10^{-5} mol/L); the black line: **1a** (10^{-5} mol/L); the blue line: **1a** (10^{-5} mol/L) + DDQ(10^{-5} mol/L).

3 Kinetic studies

3.1 Procedure for kinetic order in 1a and 2a substrate

The order in substrate *N*-(4-methoxyphenyl)-4-methylbenzenesulfonamide **1a** was determined by studying the initial rate of reaction with different concentrations of **1a**. Under N₂ atmosphere, **1a**, α -methylstyrenes (**2a**) (0.2 mmol, 0.3 mmol, 0.4 mmol, 0.5 mmol, 0.6 mmol), 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ, 10 mol%) and ⁿBu₄NBF₄ as the supporting electrolyte. We take six samples every three minutes for each group of experiments. The HPLC yields were determined using biphenyl as an internal standard. Finally, the initial rate for different concentration of **2a** vs relative concentrations could be obtained.

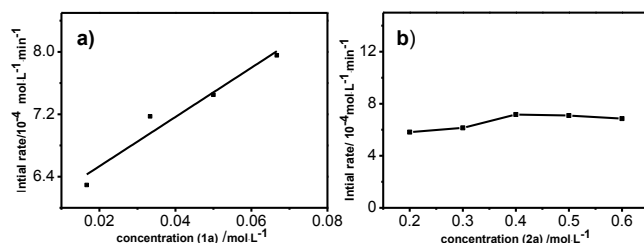
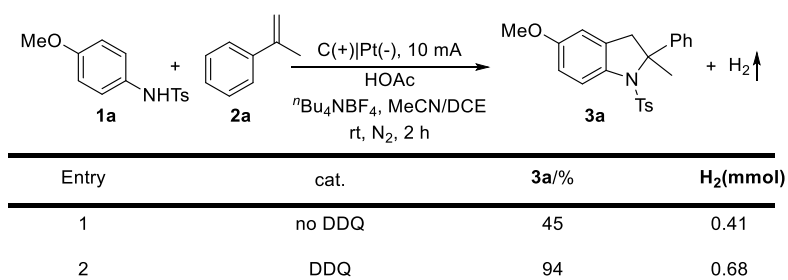


Figure S5. Kinetic studies

3.2 Procedure for hydrogen quantification

An undivided cell was equipped with a carbon anode and a platinum cathode and connected to a DC regulated power supply. *N*-(4-methoxyphenyl)-4-methylbenzenesulfonamide (0.50 mmol), prop-1-en-2-ylbenzene (1.0 mmol), ⁿBu₄NBF₄ (0.1 M), AcOH (0.2 mmol) and CH₃CN/DCE (4/2 mL) were combined and added. The bottle was equipped with graphite electrode as the anode and platinum electrode (1.5×1.5×0.3 cm³) as the cathode and was then charged with nitrogen. The reaction mixture was stirred and electrolyzed at a constant current of 10 mA under 15 °C for 2.0 h (Scheme S1).

Scheme S1. The quantitative experiment of hydrogen



At the same time, the reaction was finished, the reaction is quantified by hydrogen to investigate the cathodic reactions (as shown the **Figure S6**).

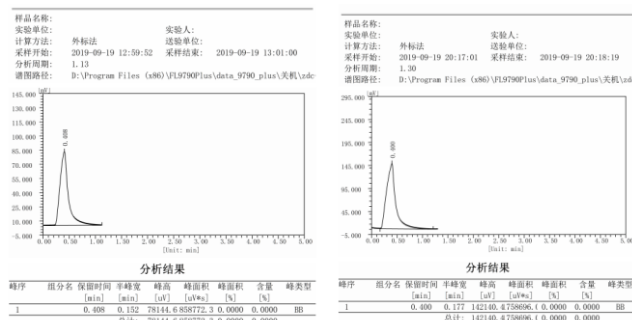


Figure S6: left picture: with DDQ; middle picture: without DDQ

3.3 Substrate Scope of other amines

At the same time, considering the effect of electronic effect, we also have made efforts to try other substituted amines in an undivided cell under constant current or constant voltage electrolysis. We found that a trace amount of products could be monitored in these reactions (Scheme S2, **1d-5**). To provide a justification for this outcome, the cyclic voltammetry (CV) experiments were conducted. **1a** gave oxidation wave at 1.47 V vs. Ag/AgCl. Whereas, higher oxidative potentials of **3-5** (>1.71 v) suggested that **3-5** were difficult to be oxidized under our conditions.

Scheme S2: the scop of other amines

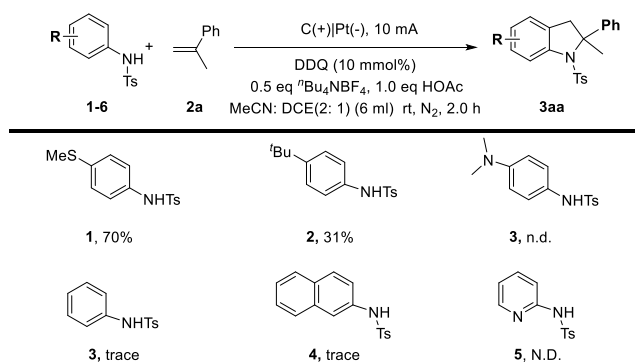


Table S1. the ratio of **3:2a**

Entry	3 : 2	Yield of 3 (%) ^[a]
1	1:1	N.D.
2	1:2	N.D.
3	1:3	N.D.
4	2:1	N.D.

^a undivided cell, nitrogen

Table S2. the effect of the Additive

Entry	Add.(0.2 mmol)	Yield of 3 (%) ^[a]
1	HOAc	N.D.
2	Na ₂ CO ₃	Trace, only detected by HRMS
3	TEA	N.D.
4	AlCl ₃	N.D.
5	HFIP	N.D.

^a undivided cell, nitrogen

Table S3. the effect of the Current

Entry	Current(mA)	t(h)	Yield of 3 (%) ^[a]
1	4	6	trace
2	6	4	trace
3	8	3	trace
4	10	2.4	trace
5	12	2	trace

^a undivided cell, nitrogen

After, the reaction was carried out in an undivided cell under constant potential electrolysis (CCE) with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ, 10 mol%). As the shown table S4. *N*-(4-(tert-butyl) phenyl) -4-methylbenzenesulfonamide (**1**) gave oxidation wave at 1.49 V vs. Ag/AgCl.

Table S4. The effect of the Voltage

Entry	T(°C)	Yield of 3 (%) ^[a]
1	RT	trace
2	60	< 3
3	80	< 5

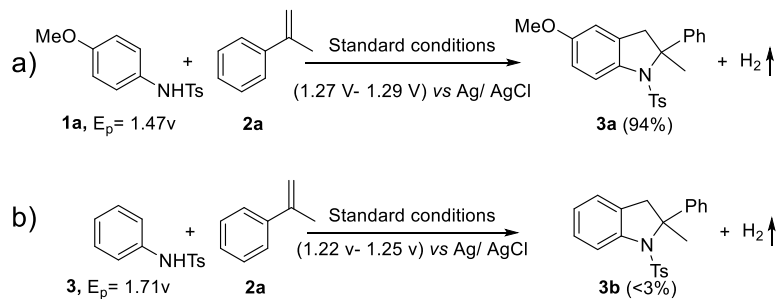
^a undivided cell, nitrogen

Table S5. The effect of the controlled oxidation potential

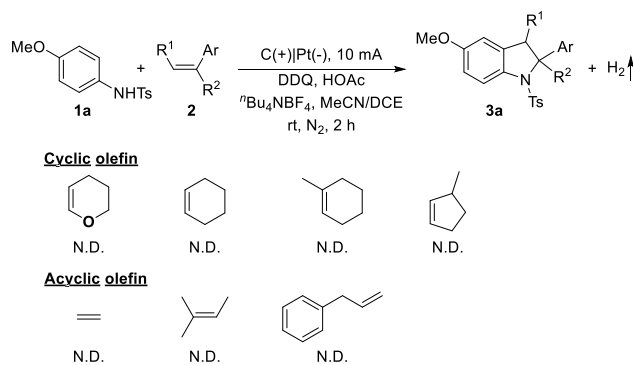
Entry	V(v)	Yield of 3 (%) ^[a]
1	1.4 V	only 1 and 3 was not detected
2	1.5 v	only 1 and 3 was not detected
3	1.58 v	only 1 and 3 was not detected
4	1.6 v	only 1 and 3 was not detected
5	1.7 v	No new point

^a undivided cell, nitrogen

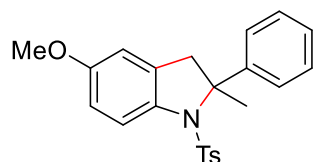
Scheme S3 Comparing the voltage of reaction a with reaction b



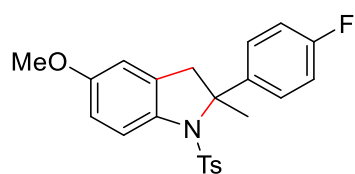
Scheme S4. Other Substrate Scope of alkenes



Detail descriptions for products

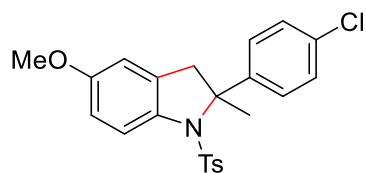


5-methoxy-2-methyl-2-phenyl-1-tosylindoline (3aa)^[2]: Yellow solid was obtained in 94% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, *J* = 8.9 Hz, 1H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.38 – 7.36 (m, 2H), 7.22 – 7.20 (m, 3H), 7.09 (d, *J* = 8.0 Hz, 2H), 6.75 (dd, *J* = 8.9, 2.7 Hz, 1H), 6.65 (dd, *J* = 2.6, 1.3 Hz, 1H), 3.75 (s, 3H), 3.39 – 3.22 (m, 2H), 2.34 (s, 3H), 2.10 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 155.98, 145.28, 143.08, 138.55, 136.07, 129.76, 129.25, 128.20, 127.28, 126.73, 125.81, 114.52, 112.67, 110.99, 72.87, 55.68, 49.14, 27.07, 21.51.

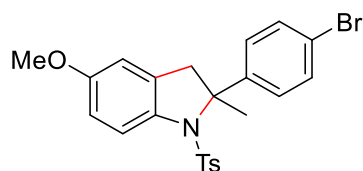


2-(4-fluorophenyl)-5-methoxy-2-methyl-1-tosylindoline (3ab)^[2]: A yellow oil was obtained in 87% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.60 (d, *J* = 8.9 Hz, 1H), 7.47 (d, *J* = 8.4 Hz, 2H), 7.37 – 7.34 (m, 2H), 7.15 (d, *J* = 8.1 Hz, 2H), 6.91 (t, *J* = 8.7 Hz, 2H), 6.80 (dd, *J* = 8.9, 2.8 Hz, 1H), 6.70 – 6.89 (m, 1H), 3.80 (s, 3H), 3.38 – 3.26 (m, 2H), 2.39 (s, 3H), 2.11 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 161.92 (d, *J*_{C-F} = 244.6), 156.06, 143.24, 141.13, 138.55, 135.95, 129.37 (d, *J*_{C-F} = 16.8), 127.60, 127.52, 126.60, 114.96, 114.67 (d, *J*_{C-F} = 16.0), 112.77, 111.01, 72.24, 55.68, 49.11, 27.21,

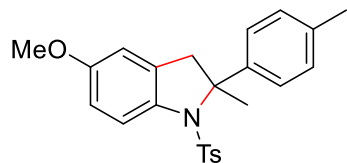
21.49. ^{19}F NMR (377 MHz, CDCl_3) δ -115.52.



2-(4-chlorophenyl)-5-methoxy-2-methyl-1-tosylindoline (3ac) ^[2]: A yellow oil was obtained in 81% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.55 (d, J = 8.9 Hz, 1H), 7.43 (d, J = 8.4 Hz, 2H), 7.38 – 7.36 (m, 2H), 7.22 – 7.21 (m, 3H), 7.10 – 7.08 (m, 2H), 6.75 (dd, J = 8.9, 2.8 Hz, 1H), 6.66 – 6.64 (m, 1H), 3.75 (s, 3H), 3.39 – 3.22 (m, 2H), 2.34 (s, 3H), 2.10 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.98, 145.29, 143.08, 138.55, 136.07, 129.76, 129.26, 128.20, 127.28, 126.73, 125.81, 114.52, 112.67, 110.99, 72.88, 55.68, 49.14, 27.07, 21.50.

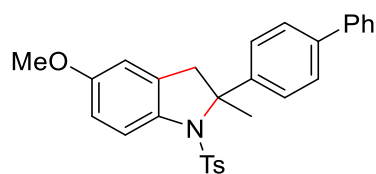


2-(4-bromophenyl)-5-methoxy-2-methyl-1-tosylindoline (3ad) ^[2]: A yellow oil was obtained in 83% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.58 (d, J = 8.9 Hz, 1H), 7.44 (d, J = 8.3 Hz, 2H), 7.30 – 7.28 (m, 2H), 7.23 – 7.20 (m, 2H), 7.12 (d, J = 8.1 Hz, 2H), 6.77 (dd, J = 8.9, 2.6 Hz, 1H), 6.65 (d, J = 2.6 Hz, 1H), 3.76 (s, 3H), 3.35 – 3.21 (m, 2H), 2.37 (s, 3H), 2.07 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.03, 144.22, 143.25, 138.40, 135.93, 131.11, 129.31, 129.27, 128.81, 127.50, 126.47, 121.32, 114.57, 112.77, 110.93, 72.11, 55.62, 48.87, 26.88, 21.47, 19.14.

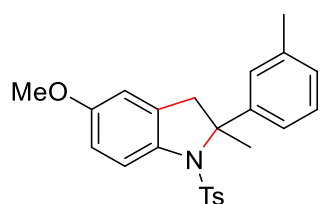


5-methoxy-2-methyl-2-(p-tolyl)-1-tosylindoline (3ae) ^[2]: A colorless oil was obtained in 95% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.60 (d, J = 8.9 Hz, 1H), 7.48 (d, J = 8.3 Hz, 2H), 7.31 (s, 1H), 7.29 (s, 1H), 7.13 (d, J = 8.1 Hz, 2H), 7.05 (d, J = 8.0 Hz, 2H), 6.79 (dd, J = 8.9, 2.7 Hz, 1H), 6.71 – 6.70 (m, 1H), 3.80 (s, 3H), 3.43 – 3.26 (m, 12H), 2.39 (s, 3H), 2.35 (s, 3H), 2.12 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ

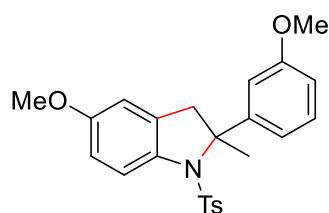
155.84, 142.89, 142.23, 138.53, 136.83, 136.01, 129.74, 129.07, 128.68, 126.61, 125.61, 114.42, 112.54, 110.86, 72.64, 55.56, 48.99, 26.99, 21.40, 20.93.



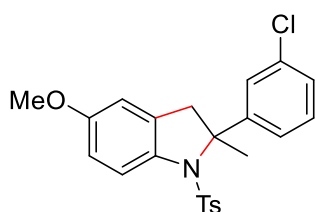
2-([1,1'-biphenyl]-4-yl)-5-methoxy-2-methyl-1-tosylindoline (3af): Colorless liquid was obtained in 83% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.68 (dd, $J = 8.9$, 1.9 Hz, 1H), 7.62 – 7.59 (m, 2H), 7.52 – 7.49 (m, 4H), 7.47 (d, $J = 1.3$ Hz, 4H), 7.42 – 7.40 (m, 1H), 7.11 (d, $J = 8.1$ Hz, 2H), 6.85 – 6.82 (m, 1H), 6.75 – 6.74 (m, 1H), 3.50 – 3.32 (m, 2H), 3.82 (s, 3H), 2.37 (s, 3H), 2.20 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.06, 144.14, 143.03, 140.63, 140.12, 138.63, 136.17, 129.76, 129.25, 128.83, 127.39, 127.02, 126.78, 126.67, 126.32, 114.60, 112.79, 111.03, 72.55, 55.70, 49.14, 27.23, 21.49. HRMS (EI) calculated for $\text{C}_{29}\text{H}_{27}\text{NO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 492.1604; found: 456.1609.



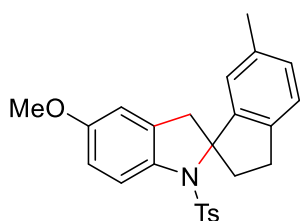
5-methoxy-2-methyl-2-(m-tolyl)-1-tosylindoline (3ag) ^[2]: A colorless oil was obtained in 92% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.59 (d, $J = 8.8$ Hz, 1H), 7.39 (d, $J = 7.8$ Hz, 2H), 7.22 (d, $J = 7.5$ Hz, 1H), 7.13 (t, $J = 7.6$ Hz, 1H), 7.09 – 7.01 (m, 4H), 6.77 (d, $J = 8.7$ Hz, 1H), 6.67 (s, 1H), 3.77 (s, 3H), 3.41 – 3.21 (m, 2H), 2.34 (s, 3H), 2.19 (s, 3H), 2.09 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.83, 144.81, 142.88, 138.46, 137.57, 135.99, 129.72, 129.06, 128.01, 127.93, 126.79, 126.61, 122.95, 114.41, 112.60, 110.90, 72.68, 55.61, 49.22, 27.14, 21.40.



5-methoxy-2-(3-methoxyphenyl)-2-methyl-1-tosylindoline (3ah) ^[2]: A colorless oil was obtained in 84% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.61 (d, *J* = 8.9 Hz, 1H), 7.49 (d, *J* = 8.4 Hz, 2H), 7.20 (t, *J* = 8.0 Hz, 1H), 7.14 (d, *J* = 8.1 Hz, 2H), 7.05 – 7.02 (m, 1H), 6.86 (t, *J* = 2.2 Hz, 1H), 6.80 – 6.77 (m, 2H), 6.69 – 6.68 (m, 1H), 3.79 (s, 3H), 3.68 (s, 3H), 3.42–3.24 (m, 2H), 2.38 (s, 3H), 2.12 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 159.68, 156.32, 147.17, 143.42, 138.89, 136.42, 130.08, 129.55, 129.49, 127.05, 118.54, 114.85, 113.05, 112.82, 112.36, 111.33, 73.18, 56.02, 55.36, 49.49, 27.46, 21.83.

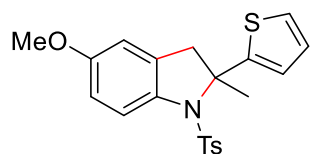


2-(3-chlorophenyl)-5-methoxy-2-methyl-1-tosylindoline (3ai) ^[2]: Colorless liquid was obtained in 61% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.59 (d, *J* = 8.9 Hz, 1H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.33-7.30 (m, 1H), 7.21 – 7.17 (m, 3H), 7.18 (d, *J* = 8.4 Hz, 2H), 6.78 (dd, *J* = 8.9, 2.7 Hz, 1H), 6.66 (d, *J* = 2.8 Hz, 1H), 3.77 (s, 3H), 3.35 – 3.21 (m, 2H), 2.36 (s, 3H), 2.07 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 156.03, 147.14, 143.34, 138.27, 135.80, 134.08, 129.36, 129.30, 129.21, 127.42, 126.43, 126.27, 124.02, 114.55, 112.83, 110.95, 72.16, 55.62, 49.00, 26.87, 21.44.

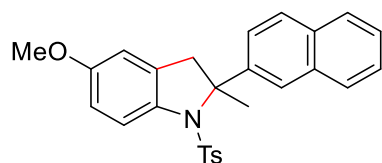


5'-methoxy-6-methyl-1'-tosyl-2,3-dihydrospiro[indene-1,2'-indoline] (3aj): Yellow oil was obtained in 62% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.67 (d, *J* = 8.9 Hz, 1H), 7.22 (d, *J* = 8.2 Hz, 2H), 7.16 (d, *J* = 7.7 Hz, 1H), 7.04 – 7.00 (m, 3H), 6.82 (dd, *J* = 8.8, 2.7 Hz, 1H), 6.75 (d, *J* = 2.6 Hz, 1H), 6.32 (s, 1H), 3.83 (s, 3H), 3.48 – 3.42 (m, 1H), 3.40 (s, 2H), 3.30 – 3.23 (m, 1H), 2.95 – 2.90 (m, 1H), 2.44 – 2.39 (m, 1H), 2.35 (s, 3H), 1.98 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 155.84, 143.76, 142.69,

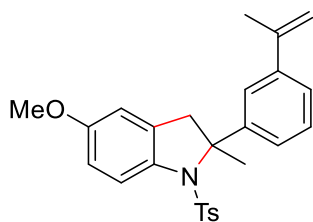
141.23, 137.86, 136.36, 135.92, 129.92, 129.61, 128.84, 126.72, 124.59, 123.84, 114.44, 112.53, 110.64, 80.76, 55.68, 47.67, 40.84, 30.10, 21.41, 20.90. HRMS (EI) calculated for $C_{25}H_{25}NO_3S$ $[M+Na]^+$: 442.1447; found: 442.1428.



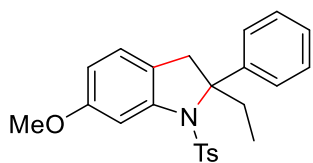
5-methoxy-2-methyl-2-(thiophen-2-yl)-1-tosylindoline (3ak): A colorless oil was obtained in 52% isolated yield. 1H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.48 (m, 3H), 7.14 – 7.11 (m, 3H), 7.06 (dd, $J = 3.6, 1.2$ Hz, 1H), 6.89 – 6.86 (m, 1H), 6.77 (dd, $J = 8.9, 2.7$ Hz, 1H), 6.72 (d, $J = 2.5$ Hz, 1H), 3.80 (s, 3H), 3.48 – 3.32 (m, 2H), 2.37 (s, 3H), 2.22 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.04, 149.24, 143.11, 138.49, 135.49, 129.23, 126.75, 126.17, 125.11, 124.49, 114.85, 112.72, 111.03, 70.86, 55.65, 49.50, 28.14. HRMS (EI) calculated for $C_{21}H_{21}NO_3S_2$ $[M+Na]^+$: 422.0855; found: 422.0840.



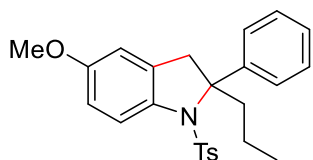
5-methoxy-2-methyl-2-(naphthalen-2-yl)-1-tosylindoline (3al): A colorless liquid was obtained in 67% isolated yield. 1H NMR (400 MHz, $DMSO-d_6$) δ 7.93 (d, $J = 2.0$ Hz, 1H), 7.90 – 7.86 (m, 2H), 7.81 (d, $J = 8.8$ Hz, 1H), 7.75 – 7.69 (m, 1H), 7.55 – 7.53 (m, 4H), 7.48 (dd, $J = 8.7, 2.0$ Hz, 1H), 7.43 – 7.41 (m, 1H), 7.24 – 7.22 (m, 2H), 6.82 (s, 1H), 4.15 (dd, $J = 5.6, 3.5$ Hz, 1H), 4.03 (d, $J = 6.6$ Hz, 1H), 3.70 (s, 3H), 4.16 – 4.14 (m, 1H), 4.03 (d, $J = 6.4, 0.52$ Hz, 1H), 3.66 – 3.61 (m, 0.56H), 2.33 (s, 3H), 2.14 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 155.91, 142.89, 141.93, 138.31, 136.12, 132.69, 132.46, 129.70, 128.97, 128.35, 127.84, 127.19, 126.47, 126.01, 125.97, 124.49, 124.12, 114.53, 112.71, 110.89, 72.64, 55.60, 48.95, 27.07, 21.31. HRMS (EI) calculated for $C_{27}H_{25}NO_3S$ $[M+Na]^+$: 466.1383; found: 466.1387.



5-methoxy-2-methyl-2-(3-(prop-1-en-2-yl) phenyl)-1-tosylindoline (3am): A colorless liquid was obtained in 53% isolated yield. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.61 (d, $J = 8.9$ Hz, 1H), 7.45 – 7.41 (m, 3H), 7.37 – 7.35 (m, 2H), 7.26 – 7.22 (m, 1H), 7.10 (d, $J = 8.1$ Hz, 2H), 6.87 – 6.78 (m, 1H), 6.70 (d, $J = 2.6$ Hz, 1H), 5.26 (s, 1H), 5.05 (s, 1H), 3.80 (s, 3H), 3.45 – 3.26 (m, 2H), 2.36 (s, 3H), 2.16 (s, 3H), 2.05 (s, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 155.94, 144.85, 143.06, 142.97, 140.92, 138.47, 136.07, 129.71, 129.23, 128.03, 126.62, 125.07, 124.45, 123.23, 114.49, 112.70, 112.57, 111.00, 72.94, 55.68, 49.20, 27.14, 21.72, 21.46. HRMS (ESI) calculated for $\text{C}_{26}\text{H}_{27}\text{NO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 456.1604; found: 456.1607.

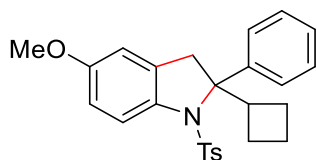


2-ethyl-6-methoxy-2-phenyl-1-tosylindoline (3an) ^[3]: A colorless liquid was obtained in 93% isolated yield. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.59 (d, $J = 8.8$ Hz, 1H), 7.31 – 7.28 (m, 2H), 7.24 – 7.21 (m, 1H), 7.17 – 7.13 (m, 4H), 7.00 – 6.98 (m, 2H), 6.80 (dd, $J = 8.8, 2.7$ Hz, 1H), 6.76 – 6.75 (m, 1H), 3.81 (s, 3H), 3.44 (s, 2H), 3.04 – 2.95 (m, 1H), 2.34 (s, 3H), 2.33 – 2.27 (m, 1H), 1.10 (t, $J = 7.3$ Hz, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 155.34, 143.60, 142.35, 137.54, 136.36, 129.80, 128.53, 127.63, 127.02, 126.40, 113.51, 112.23, 110.17, 74.91, 55.30, 45.38, 31.18, 21.06, 8.16.

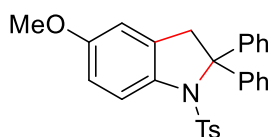


5-methoxy-2-phenyl-2-propyl-1-tosylindoline (3ao) ^[2]: Yellow oil was obtained in 89% isolated yield. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.57 (d, $J = 8.8$ Hz, 1H), 7.30 – 7.28 (m, 2H), 7.23 – 7.120 (m, 1H), 7.17 – 7.13 (m, 4H), 6.99 (d, $J = 8.2$ Hz, 2H), 6.79 (dd, $J = 8.9, 2.8$ Hz, 1H), 6.74 (d, $J = 2.6$ Hz, 1H), 3.81 (s, 3H), 3.45 (s, 2H), 2.96 – 2.89 (m,

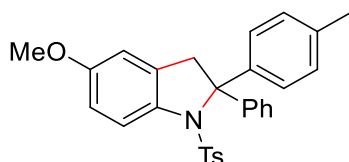
1H), 2.34 (s, 3H), 2.25 – 2.18 (m, 1H), 1.65 – 1.57 (m, 1H), 1.44 – 1.35 (m, 1H), 1.07 (t, $J = 7.3$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.61, 144.02, 142.64, 137.85, 136.54, 130.06, 128.81, 127.90, 127.29, 126.68, 126.64, 113.80, 112.50, 110.45, 74.72, 55.60, 46.32, 41.17, 21.35, 17.27, 14.40.



2-cyclobutyl-5-methoxy-2-phenyl-1-tosylindoline (3ap): Yellow oil was obtained in 81% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.55 (d, $J = 8.8$ Hz, 1H), 7.21 – 7.17 (m, 3H), 7.14 – 7.10 (m, 4H), 6.96 (d, $J = 8.1$ Hz, 2H), 6.79 (d, $J = 8.6$ Hz, 2H), 4.44 – 4.35 (m, 1H), 3.99 (d, $J = 19.6$, 1H), 3.83 (s, 3H), 3.37 (d, $J = 22.0$, 1H), 2.32 (s, 3H), 2.24 – 2.11 (m, 2H), 2.08 – 1.93 (m, 2H), 1.89 – 1.84 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.64, 143.19, 142.63, 137.82, 136.90, 130.09, 129.06, 128.85, 128.25, 128.05, 127.43, 126.96, 126.75, 125.32, 113.73, 112.56, 110.64, 55.69, 42.22, 40.17, 25.02, 22.55, 21.43, 17.18. HRMS (EI) calculated for $\text{C}_{26}\text{H}_{27}\text{NO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 456.1604; found: 456.1609.

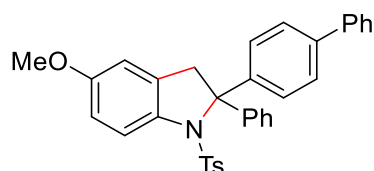


5-methoxy-2,2-diphenyl-1-tosylindoline (3aq) ^[2]: A colorless liquid was obtained in 83% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.81 (d, $J = 8.9$ Hz, 1H), 7.47 – 7.44 (m, 4H), 7.30 – 7.28 (m, 6H), 6.96 – 6.88 (m, 4H), 6.84 (dd, $J = 9.0$, 2.7 Hz, 1H), 6.66 (d, $J = 2.8$ Hz, 1H), 3.86 (s, 2H), 3.79 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.13, 143.50, 142.81, 137.76, 135.80, 129.33, 129.16, 128.79, 127.70, 127.27, 126.65, 114.90, 112.98, 110.47, 78.94, 55.66, 52.03, 21.46.

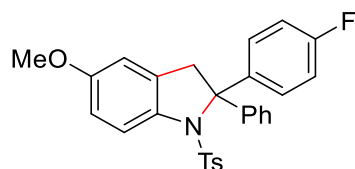


5-methoxy-2-phenyl-2-(p-tolyl)-1-tosylindoline (3ar) ^[2]: A white solid was obtained in 69% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.81 (d, $J = 8.9$ Hz, 1H), 7.47 (dd,

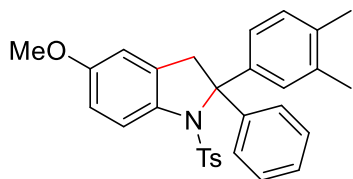
$J = 6.6, 2.9$ Hz, 2H), 7.32 – 7.29 (m, 5H), 7.06 (d, $J = 8.1$ Hz, 2H), 6.95 – 6.90 (m, 4H), 6.83 (dd, $J = 8.9, 2.6$ Hz, 1H), 6.66 (d, $J = 2.5$ Hz, 1H), 3.88 – 3.82 (m, 2H), 3.79 (s, 3H), 2.38 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.11, 143.81, 142.71, 140.35, 137.93, 137.04, 135.89, 129.40, 129.19, 129.01, 128.68, 128.27, 127.68, 127.17, 126.63, 114.94, 112.95, 110.46, 78.80, 55.65, 51.87, 21.46, 21.04.



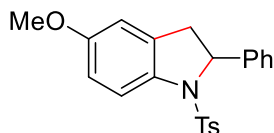
2-([1,1'-biphenyl]-4-yl)-5-methoxy-2-phenyl-1-tosylindoline (3as)^[2]: A white solid was obtained in 72% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.87 (d, $J = 8.9$ Hz, 1H), 7.64 – 7.60 (m, 3H), 7.52 – 7.49 (m, 8H), 7.34 – 7.32 (m, 3H), 6.97 – 6.92 (m, 4H), 6.87 (dd, $J = 9.0, 2.7$ Hz, 1H), 6.69 (d, $J = 2.5$ Hz, 1H), 3.96 – 3.83 (m, 2H), 3.81 (s, 3H), 2.32 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.18, 143.71, 142.75, 142.33, 140.56, 140.10, 137.94, 135.90, 129.72, 129.27, 128.97, 128.86, 128.79, 127.79, 127.45, 127.28, 127.05, 126.55, 126.23, 114.95, 113.07, 110.49, 78.60, 55.68, 52.06, 21.46. HRMS (ESI) calculated for $\text{C}_{34}\text{H}_{29}\text{NO}_3\text{S}$ [$\text{M}+\text{Na}$] $^+$: 554.1760; found: 554.1752.



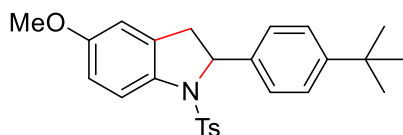
2-(4-fluorophenyl)-5-methoxy-2-phenyl-1-tosylindoline (3at): A yellow oil was obtained in 59% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.83 (d, $J = 9.0$ Hz, 1H), 7.45 – 7.38 (m, 4H), 7.30 – 7.29 (m, 2H), 6.99 – 6.91 (m, 7H), 6.85 (dd, $J = 8.9, 2.7$ Hz, 1H), 6.67 (d, $J = 2.7$ Hz, 1H), 3.82 (s, 2H), 3.79 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 161.93 (d, $J_{\text{C-F}} = 247.9$), 156.25, 143.45, 143.01, 139.14, 137.85, 135.74, 131.06 (d, $J_{\text{C-F}} = 8.0$), 129.07, 128.91, 128.84, 127.82, 127.39, 126.45, 115.05, 114.38 (d, $J_{\text{C-F}} = 21.5$), 113.10, 110.48, 78.34, 55.66, 51.95, 21.46. ^{19}F NMR (377 MHz, CDCl_3): δ -115.29. HRMS (ESI) calculated for $\text{C}_{28}\text{H}_{24}\text{FNO}_3\text{S}$ [$\text{M}+\text{Na}$] $^+$: 496.1353; found: 496.1351.



2-(3,4-dimethylphenyl)-5-methoxy-2-phenyl-1-tosylindoline(3au): A colorless liquid was obtained in 61% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.86 (d, $J = 8.9$ Hz, 1H), 7.53 – 7.47 (m, 2H), 7.32 – 7.29 (m, 3H), 7.16 – 7.14 (m, 1H), 7.07 – 7.03 (m, 2H), 6.93 (s, 4H), 6.85 (dd, $J = 8.9, 2.6$ Hz, 1H), 6.66 (d, $J = 2.7$ Hz, 1H), 3.92 (d, $J = 16.9$ Hz, 1H), 3.80 (s, 3H), 3.75 (d, $J = 16.9$ Hz, 1H), 2.34 (s, 3H), 2.28 (s, 3H), 2.12 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.01, 144.01, 142.52, 140.32, 138.00, 135.91, 135.66, 135.48, 130.26, 129.40, 128.77, 128.64, 128.50, 127.61, 127.30, 127.00, 126.43, 114.89, 112.87, 110.38, 78.65, 55.57, 51.92, 21.37, 19.80, 19.35. HRMS (ESI) calculated for $\text{C}_{30}\text{H}_{29}\text{NO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 506.1760; found: 506.1753.

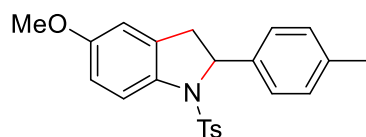


5-methoxy-2-phenyl-1-tosylindoline (3av) ^[3]: white solid was obtained in 59% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.67 (d, $J = 8.8$ Hz, 1H), 7.56 (d, $J = 8.3$ Hz, 2H), 7.37 – 7.32 (m, 5H), 7.21 (d, $J = 7.6$ Hz, 2H), 6.82 (dd, $J = 8.8, 2.6$ Hz, 1H), 6.62 (d, $J = 2.5$ Hz, 1H), 5.31 (dd, $J = 9.7, 2.6$ Hz, 1H), 3.79 (m, 3H), 3.12 (dd, $J = 16.2, 9.7$ Hz, 1H), 2.81 (dd, $J = 16.2, 2.5$ Hz, 1H), 2.41 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.52, 143.78, 142.52, 135.17, 135.01, 133.46, 129.57, 128.63, 127.60, 127.19, 125.99, 118.25, 112.94, 110.80, 65.00, 55.60, 37.84, 21.59.

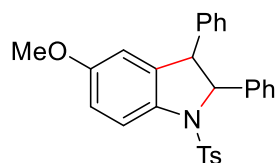


2-(4-(tert-butyl)phenyl)-5-methoxy-1-tosylindoline (3aw): A colorless oil was obtained in 78% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.67 (d, $J = 8.0$ Hz, 1H), 7.55 (d, $J = 8.3$ Hz, 2H), 7.34 – 7.27 (m, 4H), 7.19 (d, $J = 8.1$ Hz, 2H), 6.84 – 6.81 (m, 1H), 6.63 (d, $J = 2.6$ Hz, 1H), 5.31 (dd, $J = 9.6, 2.5$ Hz, 1H), 3.79 (s, 3H), 3.10 (dd, $J =$

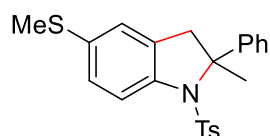
16.2, 9.7 Hz, 1H), 2.82 (dd, $J = 16.2, 2.1$ Hz, 1H), 2.40 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.47, 150.42, 143.69, 139.40, 135.18, 133.73, 129.54, 127.15, 125.69, 125.52, 118.26, 112.88, 110.80, 64.79, 55.60, 37.68, 34.49, 31.36, 21.60. HRMS (ESI) calculated for $\text{C}_{26}\text{H}_{29}\text{NO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 458.1760; found: 458.1767.



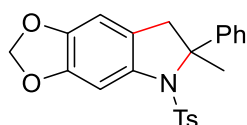
5-methoxy-2-(p-tolyl)-1-tosylindoline (3ax) ^[21]: A colorless oil was obtained in 73% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.63 (d, $J = 8.8$ Hz, 1H), 7.53 (d, $J = 8.2$ Hz, 2H), 7.19 (dd, $J = 17.0, 7.9$ Hz, 4H), 7.09 (d, $J = 7.9$ Hz, 2H), 6.79 (dd, $J = 8.8, 2.6$ Hz, 1H), 6.59 (d, $J = 2.6$ Hz, 1H), 5.25 (dd, $J = 9.7, 2.7$ Hz, 1H), 3.76 (s, 3H), 3.07 (dd, $J = 16.2, 9.7$ Hz, 1H), 2.77 (dd, $J = 16.2, 2.7$ Hz, 1H), 2.37 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.48, 143.75, 139.60, 137.28, 135.17, 135.03, 133.59, 129.56, 129.28, 127.19, 125.94, 118.26, 112.91, 110.77, 64.88, 55.60, 37.82, 21.60, 21.12.



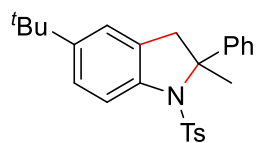
5-methoxy-2,3-diphenyl-1-tosylindoline (3ay): A colorless oil was obtained in 56% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.80 (d, $J = 8.9$ Hz, 1H), 7.46 – 7.44 (m, 4H), 7.30 – 7.28 (m, 5H), 6.95 – 6.88 (m, 5H), 6.84 (dd, $J = 9.0, 2.7$ Hz, 1H), 6.66 (d, $J = 2.8, 1\text{H}$) 3.85 (s, 2H), 3.79 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.12, 143.49, 142.79, 137.76, 135.80, 129.32, 129.15, 128.78, 127.69, 127.26, 126.64, 125.98, 125.98, 114.90, 112.96, 110.47, 78.93, 77.37, 77.05, 76.74, 55.66, 52.02, 21.45. HRMS (EI) calculated for $\text{C}_{28}\text{H}_{25}\text{NO}_3\text{S}_1$ $[\text{M}+\text{H}]^+$: 456.16299; found: 456.16279.



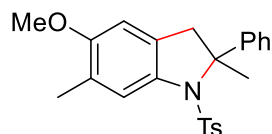
2-methyl-5-(methylthio)-2-phenyl-1-tosylindoline (3ba) Yellow solid was obtained in 70% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.55 (d, $J = 8.8$ Hz, 1H), 7.42-7.39 (m, 2H), 7.36 – 7.33(m, 2H), 7.22 – 7.20 (m, 3H), 7.16 – 7.14 (m, 1H), 7.08 (dd, $J = 8.9, 2.7$ Hz, 2H), 7.03-7.02 (m, 1H), 3.41 – 3.23 (m, 2H), 2.44 (s, 3H), 2.34 (s, 3H), 2.11 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 145.13, 143.40, 140.80, 138.46, 131.87, 129.39, 129.28, 128.34, 127.74, 127.49, 126.92, 125.96, 124.73, 114.25, 73.08, 49.14, 27.19, 21.58, 17.39. HRMS (EI) calculated for $\text{C}_{23}\text{H}_{23}\text{NO}_2\text{S}_2$ $[\text{M}+\text{H}]^+$: 410.12430; found: 410.12238.



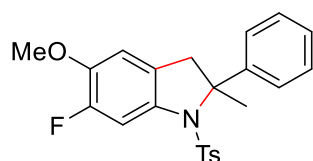
6-methyl-6-phenyl-5-tosyl-6,7-dihydro-5H- [1,3] dioxolo [4,5-f] indole (3ca): A yellow oil was obtained in 82% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.47 (d, $J = 8.3$ Hz, 2H), 7.42 (dd, $J = 6.7, 3.0$ Hz, 2H), 7.33 (s, 1H), 7.27 – 7.26 (m, 3H), 7.15 (d, $J = 8.1$ Hz, 2H), 6.58 (s, 1H), 5.97 (d, $J = 1.2$ Hz, 2H), 3.35 – 3.18 (m, 2H), 2.39 (s, 3H), 2.12 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 147.34, 145.30, 143.59, 143.19, 138.57, 136.40, 129.35, 128.20, 127.30, 126.72, 125.77, 120.21, 105.22, 101.37, 97.35, 73.49, 48.78, 27.10, 21.52. HRMS (ESI) calculated for $\text{C}_{23}\text{H}_{21}\text{NO}_4\text{S}$ $[\text{M}+\text{Na}]^+$: 430.1083; found: 430.1067.



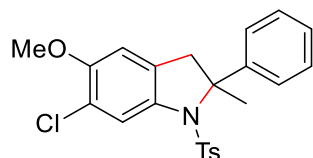
5-(tert-butyl)-2-methyl-2-phenyl-1-tosylindoline (3da) Yellow oil was obtained in 23% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.86 (d, $J = 8.3$ Hz, 1H), 7.68 (d, $J = 8.3$ Hz, 2H), 7.58 (d, $J = 9.2$ Hz, 2H), 7.51 (d, $J = 7.3$ Hz, 3H), 7.27 – 7.25 (m, 4H), 3.89 – 3.78 (m, 2H), 2.42 (s, 3H), 1.68 (s, 3H), 1.28 (s, 9H). HRMS (EI) calculated for $\text{C}_{26}\text{H}_{29}\text{NO}_2\text{S}$ $[\text{M}+\text{H}]^+$: 419.19190; found: 419.19200.



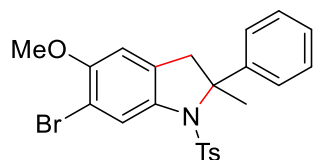
5-methoxy-2,6-dimethyl-2-phenyl-1-tosylindoline(3ea) ^[2]: white solid was obtained in 78% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.50 (s, 1H), 7.44 (d, *J* = 8.3 Hz, 2H), 7.40 – 7.37 (m, 2H), 7.24 – 7.20 (m, 3H), 7.10 (d, *J* = 8.1 Hz, 2H), 6.59 (s, 1H), 3.76 (s, 3H), 3.39 – 3.23 (m, 2H), 2.35 (s, 3H), 2.26 (s, 3H), 2.09 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 154.01, 145.36, 142.84, 138.70, 135.38, 129.14, 128.06, 127.11, 126.55, 126.15, 125.91, 125.71, 116.27, 107.02, 72.68, 55.64, 49.19, 26.99, 21.40, 16.81.



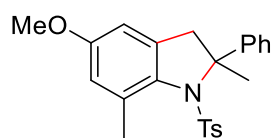
6-fluoro-5-methoxy-2-methyl-2-phenyl-1-tosylindoline(3fa) ^[2]: A white solid was obtained in 81% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.52 (d, *J* = 12.5 Hz, 1H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.38 (dd, *J* = 6.8, 2.9 Hz, 2H), 7.26 – 7.25 (m, 3H), 7.14 (d, *J* = 8.1 Hz, 2H), 6.75 (d, *J* = 8.4 Hz, 1H), 3.85 (s, 3H), 3.41 – 3.24 (m, 2H), 2.39 (s, 3H), 2.13 (s, 3H). ¹³C NMR (101 MHz, CDCl₃): δ (ppm) 152.4 (d, *J* = 243.7 Hz), 144.8, 143.6 (d, *J* = 11.9 Hz), 143.4, 138.3, 135.8 (d, *J* = 10.4 Hz), 129.4, 128.2, 127.4, 126.7, 125.8, 123.0, 110.5 (d, *J* = 8.5 Hz), 103.4 (d, *J* = 26.1 Hz), 73.4, 57.0, 48.9, 27.0, 21.5.



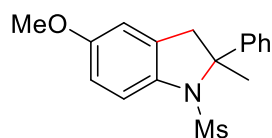
6-chloro-5-methoxy-2-methyl-2-phenyl-1-tosylindoline(3ga) ^[2]: A white solid was obtained in 69% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.77 (s, 1H), 7.44 (d, *J* = 8.3 Hz, 2H), 7.39 – 7.36 (m, 2H), 7.26 – 7.25 (m, 3H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.73 (s, 1H), 3.86 (s, 3H), 3.42 – 3.25 (m, 2H), 2.39 (s, 3H), 2.13 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 151.48, 144.78, 143.38, 138.25, 136.30, 129.37, 128.27, 127.74, 127.45, 126.66, 125.78, 121.54, 115.76, 109.23, 73.27, 56.62, 49.04, 27.05, 21.52.



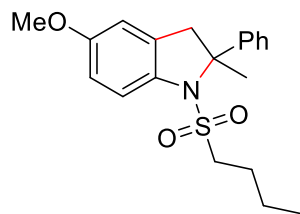
6-bromo-5-methoxy-2-methyl-2-phenyl-1-tosylindoline(3ha): A white solid was obtained in 75% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.93 (s, 1H), 7.43 (d, $J = 8.4$ Hz, 2H), 7.39 – 7.36 (m, 2H), 7.26 – 7.24 (m, 3H), 7.14 (d, $J = 8.2$ Hz, 2H), 6.71 (s, 1H), 3.85 (s, 3H), 3.40 – 3.23 (m, 2H), 2.39 (s, 3H), 2.13 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 152.14, 144.77, 143.38, 138.23, 136.72, 129.38, 128.69, 128.27, 127.45, 126.65, 125.78, 118.54, 110.45, 109.04, 73.27, 56.70, 49.06, 27.07, 21.53. HRMS (ESI) calculated for $\text{C}_{23}\text{H}_{22}\text{BrNO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 494.0396; found: 494.0450.



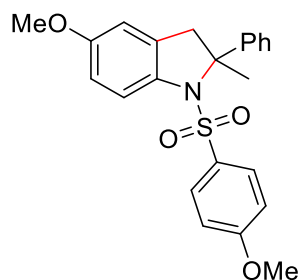
5-methoxy-2,7-dimethyl-2-phenyl-1-tosylindoline (3ia) ^[2]: white solid was obtained in 58% isolated yield. ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 7.51 – 7.49 (m, 2H), 7.36 (d, $J = 3.0$ Hz, 4H), 7.25 (t, $J = 7.7$ Hz, 2H), 7.15 (t, $J = 7.3$ Hz, 1H), 6.59 (d, $J = 2.3$ Hz, 2H), 4.03 (d, $J = 6.4$ Hz, 0.2H), 3.68 (s, 3H), 3.59 (s, 0.3H), 3.13 (d, $J = 15.8$ Hz, 1.2H), 2.95 (s, 0.31H), 2.46 (s, 3H), 2.39 (s, 3H), 1.72 (s, 3H). ^{13}C NMR (101 MHz, $\text{DMSO}-d_6$) δ 158.36, 146.79, 144.27, 139.52, 137.95, 136.30, 133.85, 130.12, 128.56, 127.52, 127.35, 125.39, 114.69, 108.46, 75.70, 55.61, 42.42, 27.73, 21.48, 20.37. HRMS (ESI) calculated for $\text{C}_{24}\text{H}_{25}\text{NO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 430.1447; found: 430.1434.



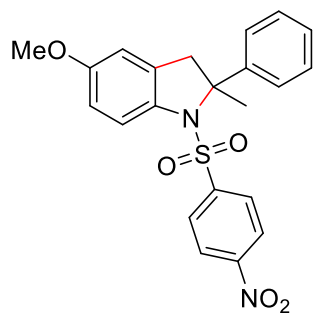
5-methoxy-2-methyl-1-(methylsulfonyl)-2-phenylindoline (3ja) ^[2]: white solid was obtained in 82% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.51 (dd, $J = 8.3, 1.4$ Hz, 2H), 7.38 – 7.31 (m, 4H), 6.83 – 6.80 (m, 2H), 3.83 (s, 3H), 3.60 – 3.31 (m, 2H), 2.60 (s, 3H), 2.15 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.06, 144.55, 135.72, 129.74, 128.43, 127.83, 125.73, 113.54, 112.85, 111.30, 72.30, 55.78, 49.03, 38.70, 26.63.



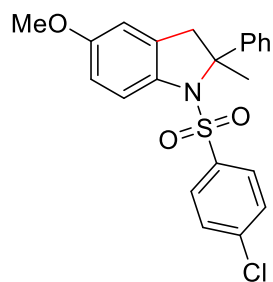
5-methoxy-2-methyl-2-phenyl-1-(propylsulfonyl) indoline (3ka) white solid was obtained in 80% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.54 – 7.52 (m, 2H), 7.38 – 7.29 (m, 4H), 6.81 – 6.79 (m, 2H), 3.82 (s, 3H), 3.55 – 3.37 (m, 2H), 2.89 – 2.82 (m, 1H), 2.61 – 2.51 (m, 1H), 2.15 (s, 3H), 1.79 – 1.71 (m, 2H), 1.37 – 1.30 (m, 2H), 0.87 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 155.74, 145.00, 135.96, 129.24, 128.23, 127.53, 125.59, 113.43, 112.62, 111.11, 72.21, 55.62, 52.18, 48.98, 26.72, 24.76, 21.45, 13.34. HRMS (ESI) calculated for $\text{C}_{20}\text{H}_{25}\text{NO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 382.1447; found: 382.1439.



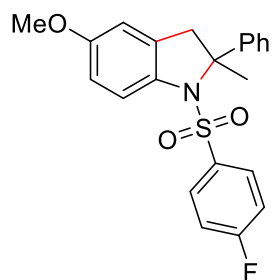
5-methoxy-1-((4-methoxyphenyl) sulfonyl)-2-methyl-2-phenylindoline (3la) A yellow solid was obtained in 84% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.58 (d, $J = 8.9$ Hz, 1H), 7.51 – 7.48 (m, 2H), 7.42 – 7.40 (m, 2H), 7.27 – 7.25 (m, 3H), 6.79 (dd, $J = 9.1, 2.4$ Hz, 3H), 6.70 (d, $J = 2.5$ Hz, 1H), 3.83 (s, 3H), 3.79 (s, 3H), 3.44 – 3.27 (m, 2H), 2.14 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 162.58, 155.93, 145.23, 136.09, 133.21, 129.77, 128.85, 128.20, 127.30, 125.85, 114.40, 113.76, 112.67, 111.00, 72.75, 55.68, 55.57, 49.20, 27.10. HRMS (ESI) calculated for $\text{C}_{23}\text{H}_{23}\text{NO}_4\text{S}$ $[\text{M}+\text{Na}]^+$: 432.1240; found: 432.1232.



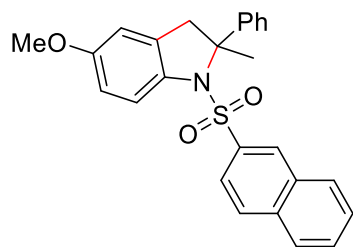
5-methoxy-2-methyl-1-((4-nitrophenyl) sulfonyl)-2-phenylindoline (3ma) ^[2] A yellow solid was obtained in 66% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, *J* = 8.9 Hz, 2H), 7.70 (d, *J* = 8.9 Hz, 1H), 7.57 – 7.54 (m, 2H), 7.29 – 7.27 (m, 2H), 7.25 – 7.22 (m, 1H), 7.18 – 7.14 (m, 2H), 6.85 (dd, *J* = 8.9, 2.7 Hz, 1H), 6.75 – 6.74 (m, 1H), 3.83 (s, 3H), 3.59 – 3.24 (m, 2H), 2.16 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 156.01, 148.92, 146.04, 143.19, 134.67, 129.35, 127.60, 127.27, 126.98, 125.62, 123.13, 113.91, 112.56, 110.60, 72.25, 55.15, 48.65, 26.96.



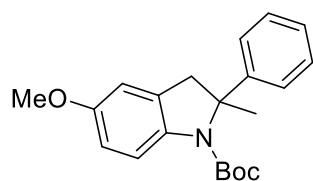
1-((4-chlorophenyl) sulfonyl)-5-methoxy-2-methyl-2-phenylindoline (3na): A yellow oil was obtained in 75% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.63 (d, *J* = 8.9 Hz, 1H), 7.41 (d, *J* = 8.7 Hz, 2H), 7.35 (dd, *J* = 8.1, 1.7 Hz, 2H), 7.27 – 7.22 (m, 5H), 6.82 (dd, *J* = 8.9, 2.7 Hz, 1H), 6.72 (d, *J* = 2.8 Hz, 1H), 3.81 (s, 3H), 3.48 – 3.29 (m, 2H), 2.15 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 156.21, 144.47, 139.75, 138.65, 135.68, 129.83, 128.82, 128.21, 127.99, 127.52, 125.99, 114.45, 112.86, 111.08, 72.78, 55.70, 49.20, 27.31. HRMS (ESI) calculated for C₂₂H₂₀ClNO₃S [M+Na]⁺: 436.0744; found: 436.0693.



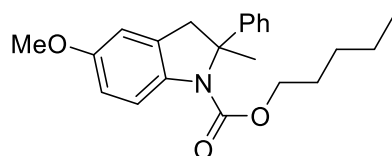
1-((4-fluorophenyl)sulfonyl)-5-methoxy-2-methyl-2-phenylindoline (3oa): A yellow oil was obtained in 76% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.62 (d, $J = 8.9$ Hz, 1H), 7.51 – 7.48 (m, 2H), 7.35 (dd, $J = 7.9, 1.9$ Hz, 2H), 7.26 – 7.12 (m, 3H), 6.96 (t, $J = 8.6$ Hz, 2H), 6.82 (dd, $J = 8.9, 2.7$ Hz, 1H), 6.73 – 6.69 (m, 1H), 3.81 (s, 3H), 3.48 – 3.29 (m, 2H), 2.15 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 164.8 (d, $J_{\text{C-F}} = 252.7$), 156.16, 144.51, 137.43 (d, $J_{\text{C-F}} = 3.2$), 135.78, 129.24 (d, $J_{\text{C-F}} = 9.2$), 129.19, 128.18, 127.48, 125.99, 115.71 (d, $J_{\text{C-F}} = 22.3$), 114.40, 112.83, 111.07, 72.72, 55.69, 49.23, 27.27.



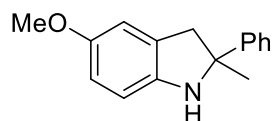
5-methoxy-2-methyl-1-(naphthalen-2-ylsulfonyl)-2-phenylindoline (3pa) ^[2]: A yellow solid was obtained in 83% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, $J = 1.9$ Hz, 1H), 7.85 (d, $J = 8.1$ Hz, 1H), 7.80 – 7.77 (m, 2H), 7.73 (d, $J = 8.9$ Hz, 1H), 7.63 – 7.55 (m, 3H), 7.40 – 7.37 (m, 2H), 7.19 – 7.14 (m, 3H), 6.83 (dd, $J = 8.9, 2.7$ Hz, 1H), 6.71 (d, $J = 2.6$ Hz, 1H), 3.80 (s, 3H), 3.46 – 3.25 (m, 2H), 2.19 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.05, 144.51, 138.27, 136.02, 134.63, 131.91, 129.79, 129.47, 128.79, 128.55, 128.10, 128.07, 127.69, 127.47, 127.16, 126.04, 122.02, 114.57, 112.80, 111.02, 72.86, 55.69, 49.22, 27.28.



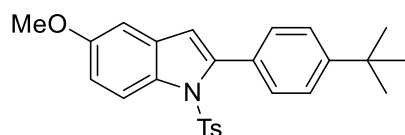
tert-butyl 5-methoxy-2-methyl-2-phenylindoline-1-carboxylate (3qa) A yellow solid was obtained in 56% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 8.01 (d, *J* = 12 Hz, 1H), 7.41 – 7.22 (m, 3H), 6.93 (d, *J* = 2.8 Hz, 1H), 6.84 (dd, *J* = 9.1, 2.9 Hz, 1H), 6.80 – 6.70 (m, 2H), 3.81 (s, 1H), 3.80 (s, 3H), 3.40 – 3.16 (m, 10H), 1.55 (s, 9H), 1.15 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 155.40, 152.77, 128.47, 128.21, 126.43, 124.59, 115.64, 114.43, 113.32, 112.30, 110.65, 80.80, 55.68, 29.73, 28.33, 27.94, 25.11. HRMS (ESI) calculated for C₂₁H₂₅NO₃ [M+H]⁺: 362.17267; found: 362.17266.



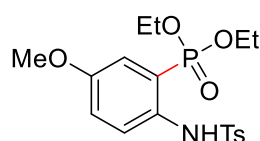
pentyl 5-methoxy-2-methyl-2-phenylindoline-1-carboxylate(3ra) A yellow solid was obtained in 53% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 8.01 (s, 1H), 7.37 – 7.32 (m, 4H), 7.26 – 7.24 (m, 1H), 6.83 (dd, *J* = 9.3, 2.8 Hz, 1H), 6.71 (d, *J* = 1.4 Hz, 1H), 4.00 – 3.89 (m, 2H), 3.82 (s, 3H), 3.36 (d, *J* = 16.6 Hz, 1H), 3.26 (d, *J* = 16.5 Hz, 1H), 1.98 (s, 3H), 1.74 – 1.73 (m, 2H), 1.42 – 1.39 (m, 2H), 0.97 – 0.94 (m, 2H), 0.83 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 155.82, 128.45, 126.63, 124.56, 115.75, 114.48, 113.40, 112.44, 110.67, 68.26, 64.98, 55.67, 48.79, 28.62, 28.01, 27.84, 22.26, 14.03. HRMS (ESI) calculated for C₂₂H₂₇NO₃ [M+Na]⁺: 376.1883; found: 376.1883.



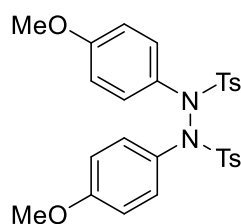
5-methoxy-2-methyl-2-phenylindoline (4a) A yellow liquid was obtained in 99% isolated yield. ¹H NMR (400 MHz, CDCl₃) δ 7.58 – 7.51 (m, 2H), 7.38 (d, *J* = 1.5 Hz, 2H), 7.32 – 7.25 (m, 1H), 6.76 – 6.75 (m, 1H), 6.68 – 6.67 (m, 2H), 3.79 (s, 3H), 3.28 – 3.19 (m, 2H), 1.68 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 153.50, 148.76, 143.93, 129.32, 128.37, 126.52, 125.24, 112.39, 111.80, 109.94, 66.67, 55.97, 46.20, 29.52. HRMS (ESI) calculated for C₁₆H₁₇NO [M+Na]⁺: 240.1383; found: 240.1377.



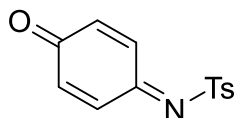
2-(4-(tert butyl) phenyl)-5-methoxy-1-tosylindoline (4b) A yellow liquid was obtained in 53% isolated yield. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.20 (d, $J = 9.1$ Hz, 1H), 7.47 (s, 4H), 7.27 (d, $J = 8.3$ Hz, 2H), 7.06 (d, $J = 8.1$ Hz, 2H), 6.97 (dd, $J = 9.1, 2.6$ Hz, 1H), 6.90 (d, $J = 2.5$ Hz, 1H), 6.48 (s, 1H), 3.85 (s, 3H), 2.32 (s, 3H), 1.42 (s, 9H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 157.01, 151.70, 144.34, 143.32, 134.29, 132.80, 131.83, 129.85, 129.48, 129.10, 126.86, 124.49, 117.76, 113.63, 113.21, 103.05, 55.58, 34.78, 31.39, 21.56. HRMS (ESI) calculated for $\text{C}_{26}\text{H}_{27}\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$: 434.1770; found: 434.1784.



diethyl (5-methoxy-2-((4-methylphenyl) sulfonamido) phenyl) phosphonate (4c): A yellow liquid was obtained in 50% isolated yield. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 9.86 (s, 1H), 7.80 (dd, $J = 9.1, 7.5$ Hz, 1H), 7.71 (d, $J = 8.3$ Hz, 2H), 7.21 (d, $J = 8.1$ Hz, 2H), 7.07 (dd, $J = 9.1, 3.0$ Hz, 1H), 6.93 (dd, $J = 15.5, 3.0$ Hz, 1H), 3.93 – 3.87 (m, 2H), 3.79 (s, 3H), 3.77 – 3.70 (m, 2H), 2.36 (s, 3H), 1.21 (t, $J = 7.1$ Hz, 6H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 155.64 (d, $J = 16.5$ Hz), 143.46, 136.52, 135.03 (d, $J = 7.1$ Hz), 129.44, 127.46, 122.87 (d, $J = 13.8$ Hz), 120.06 (d, $J = 2.7$ Hz), 117.32, 116.75 (d, $J = 6.9$ Hz), 115.55, 62.52 (d, $J = 4.9$ Hz), 55.61, 21.52, 16.13 (d, $J = 6.7$ Hz). HRMS (ESI) calculated for $\text{C}_{18}\text{H}_{24}\text{NO}_6\text{PS}$ $[\text{M}+\text{Na}]^+$: 436.0954; found: 436.0962.



***N, N'*-bis(4-methoxyphenyl)-4-methyl -*N'*-tosylbenzenesulfonohydrazide (4d):** A yellow liquid was obtained in 3% isolated yield. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.61 (d, $J = 8.3$ Hz, 4H), 7.28 – 7.20 (m, 4H), 7.01 – 6.99 (m, 4H), 6.80 – 6.77 (m, 4H), 3.78 (s, 6H), 2.41 (s, 6H). HRMS (ESI) calculated for $\text{C}_{28}\text{H}_{28}\text{N}_2\text{O}_6\text{S}_2$ $[\text{M}+\text{Na}]^+$: 575.1281; found: 575.1271.



4-methyl-N-(4-oxocyclohexa-2,5-dien-1-ylidene) benzenesulfonamide(4e), A yellow liquid was obtained in 53% isolated yield. ^1H NMR (400 MHz, CDCl_3) δ 8.24 (dd, $J = 10.5, 2.7$ Hz, 1H), 7.93 (d, $J = 8.3$ Hz, 2H), 7.41 (d, $J = 8.1$ Hz, 2H), 7.01 (dd, $J = 10.2, 2.7$ Hz, 1H), 6.74 – 6.70 (m, 2H), 2.49 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 185.91, 163.68, 144.99, 140.47, 136.68, 135.61, 135.21, 130.26, 129.85, 127.69, 77.07, 21.74. HRMS (ESI) calculated for $\text{C}_{13}\text{H}_{11}\text{NO}_3\text{S}$ $[\text{M}+\text{Na}]^+$: 284.0352; found: 284.0346.

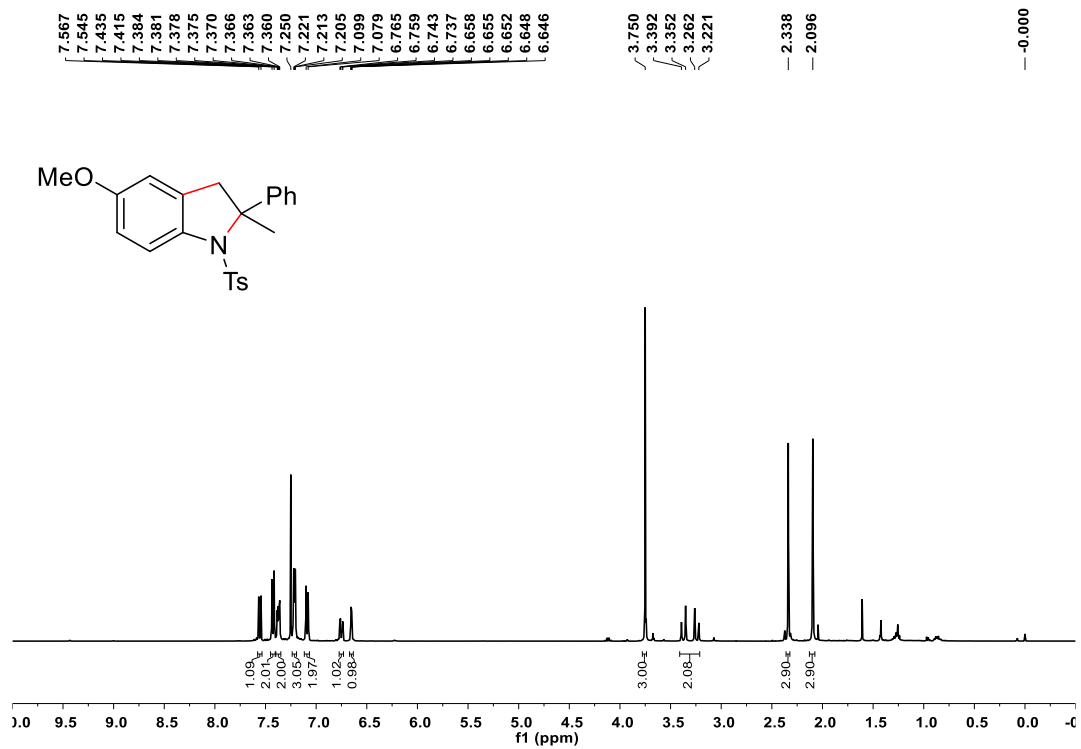
References

- [1] F. Zhou, D.-S. Wang, T. G. Driver, *Adv. Synth. Catal.* **2015**, 357, 3463.
- [2] N. Yang, Q. -L. Yu, Q.-H. Liu, H.-H. Zuo, H.-B. Yu, W.-J. Wei, R.-Z. Liao, F.-R. Zhang, *Org. Lett.* **2018**, 20, 1404.
- [3] Y.-J. Hu, T. Kamitanaka, Y. Mishima, T.Dohi, Y. Kita, *J. Org. Chem.* **2013**, 78, 5530.

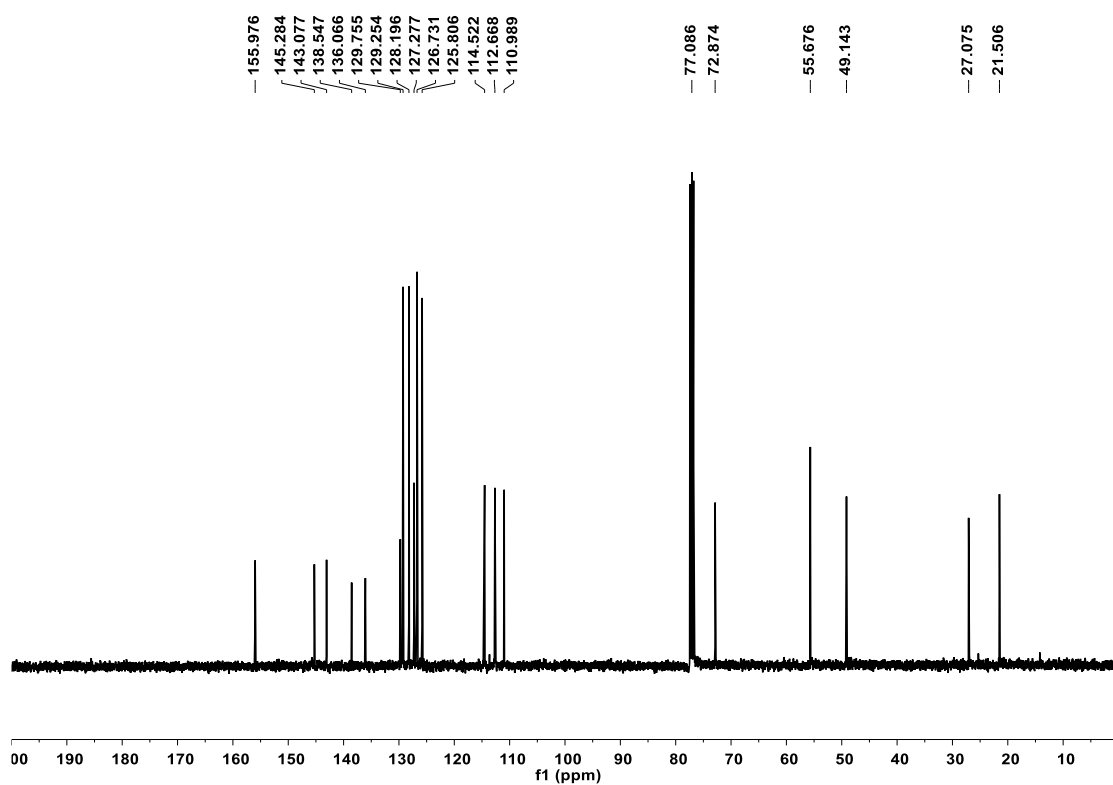
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3aa

¹H NMR

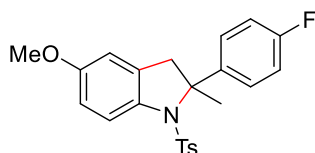
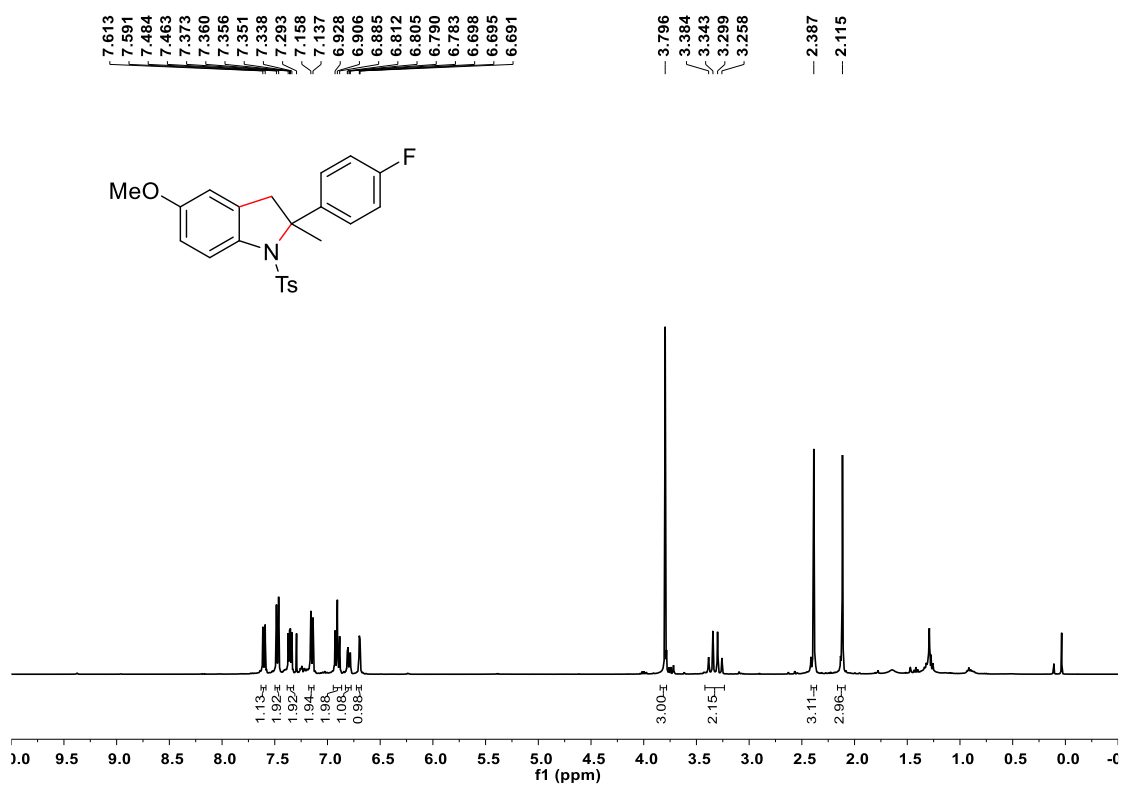


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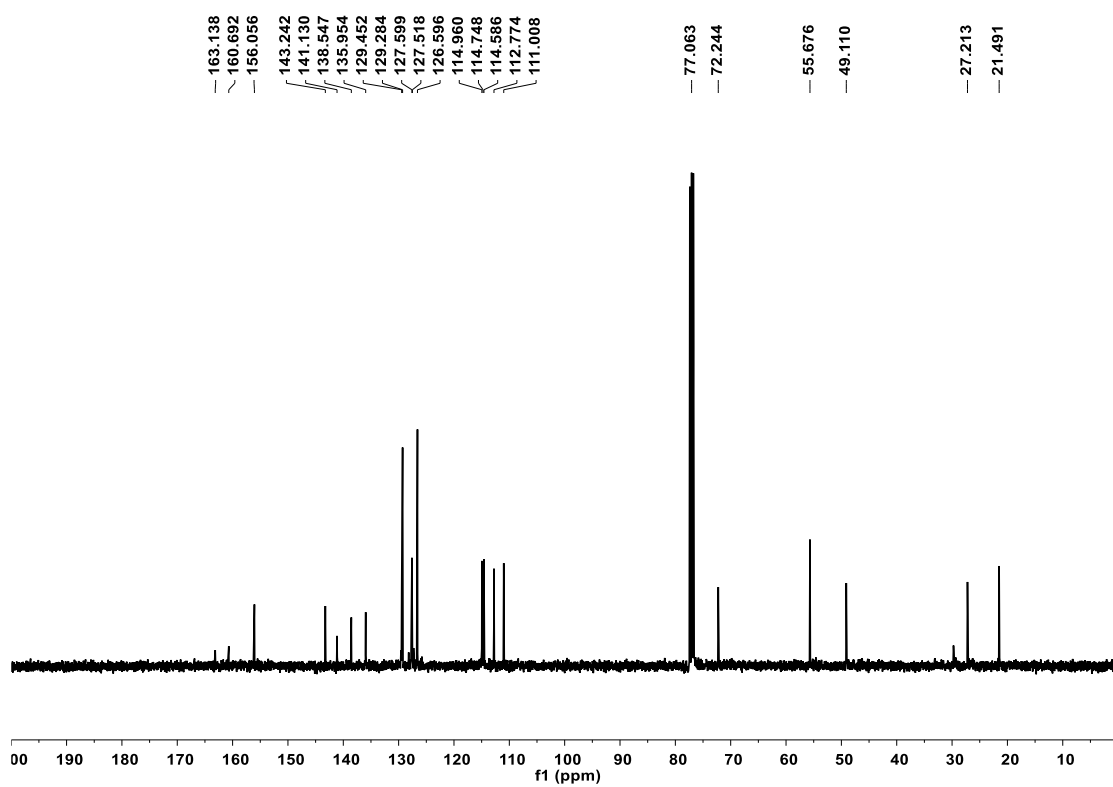


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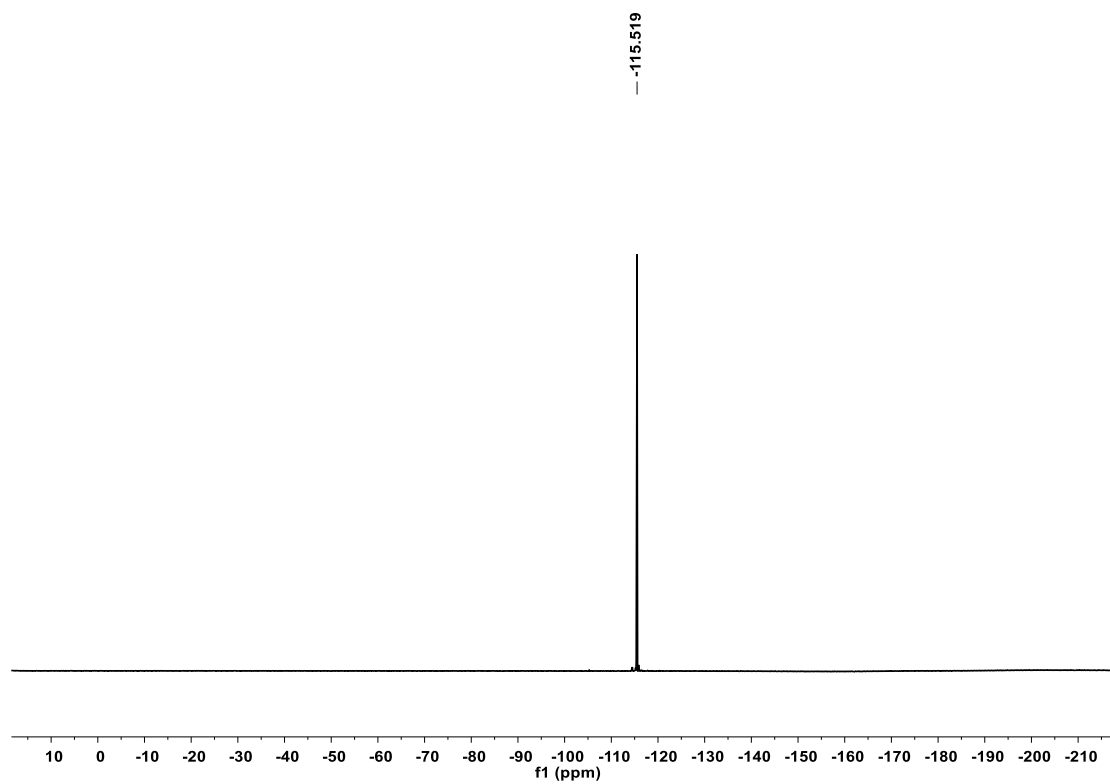
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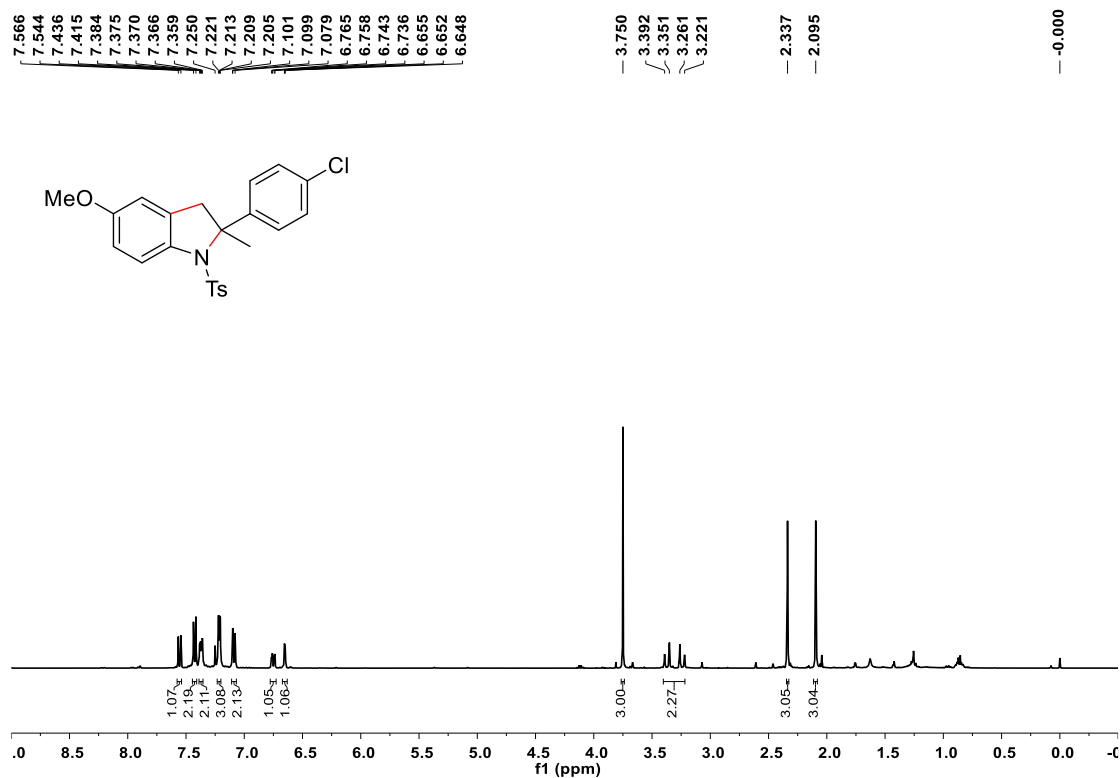


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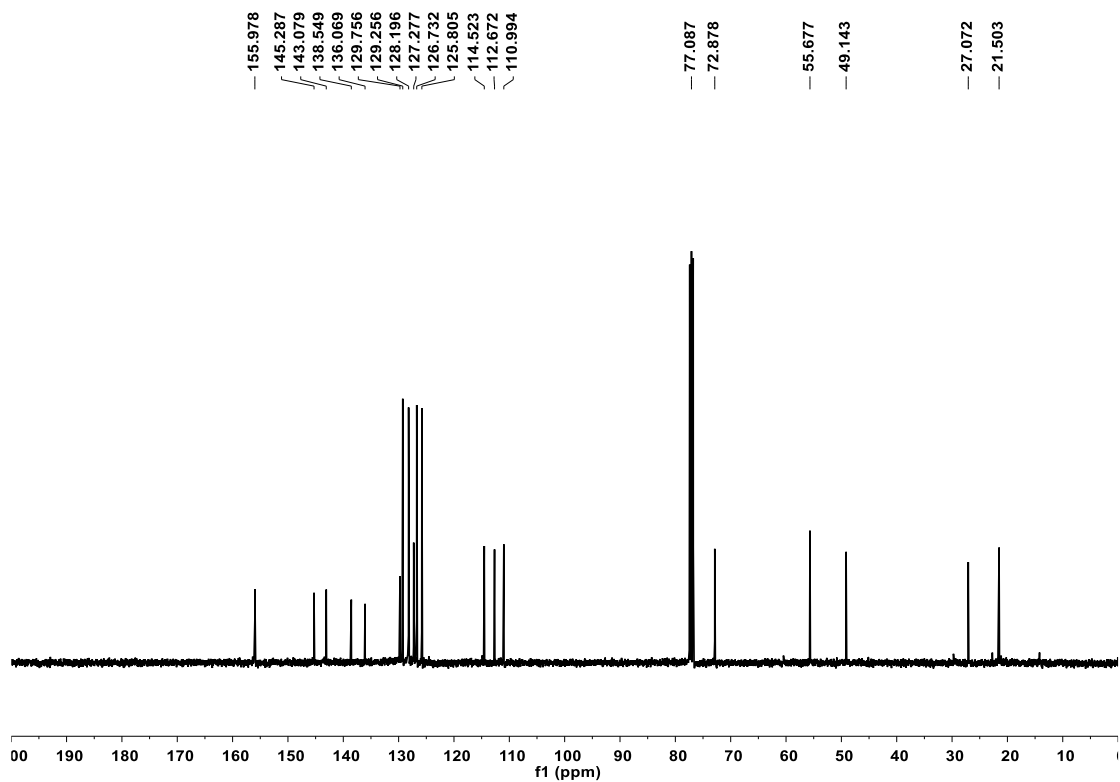


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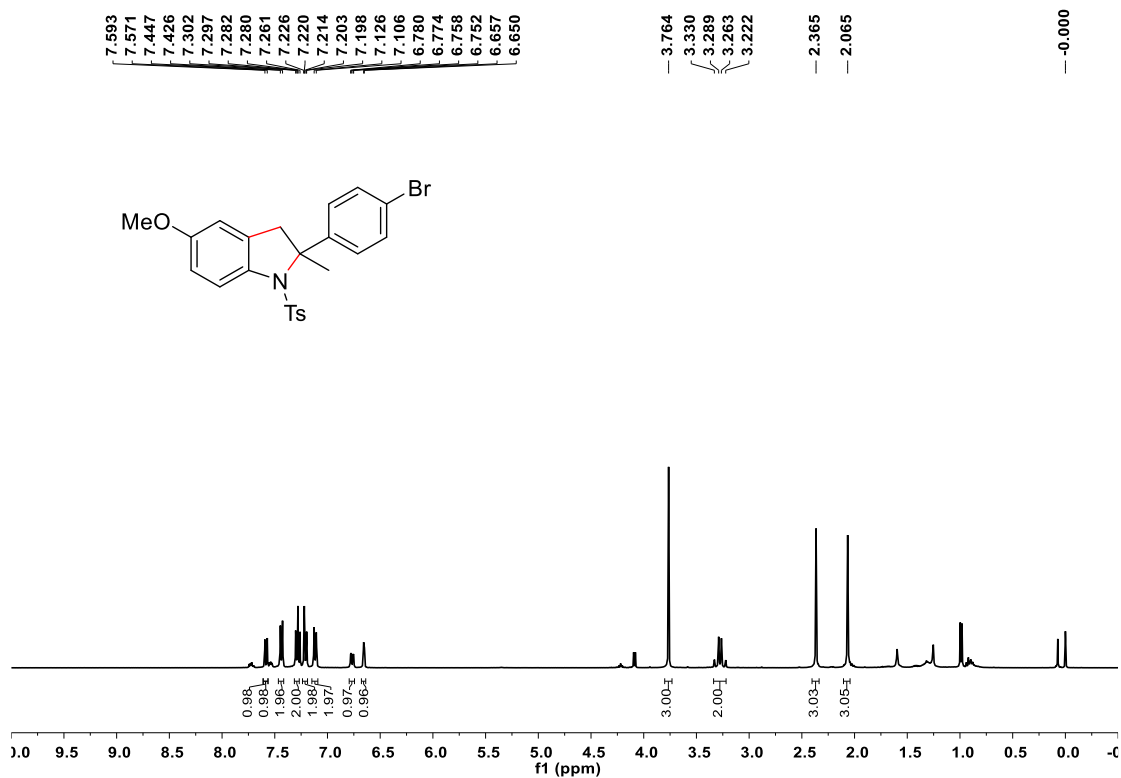


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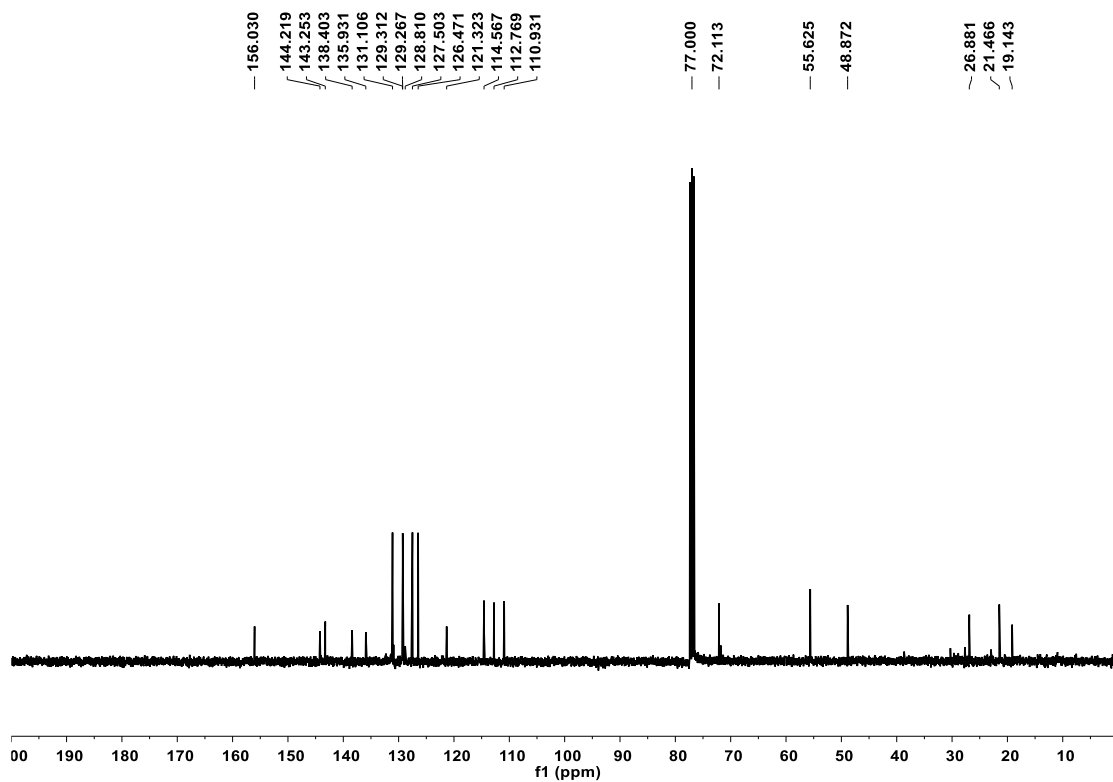


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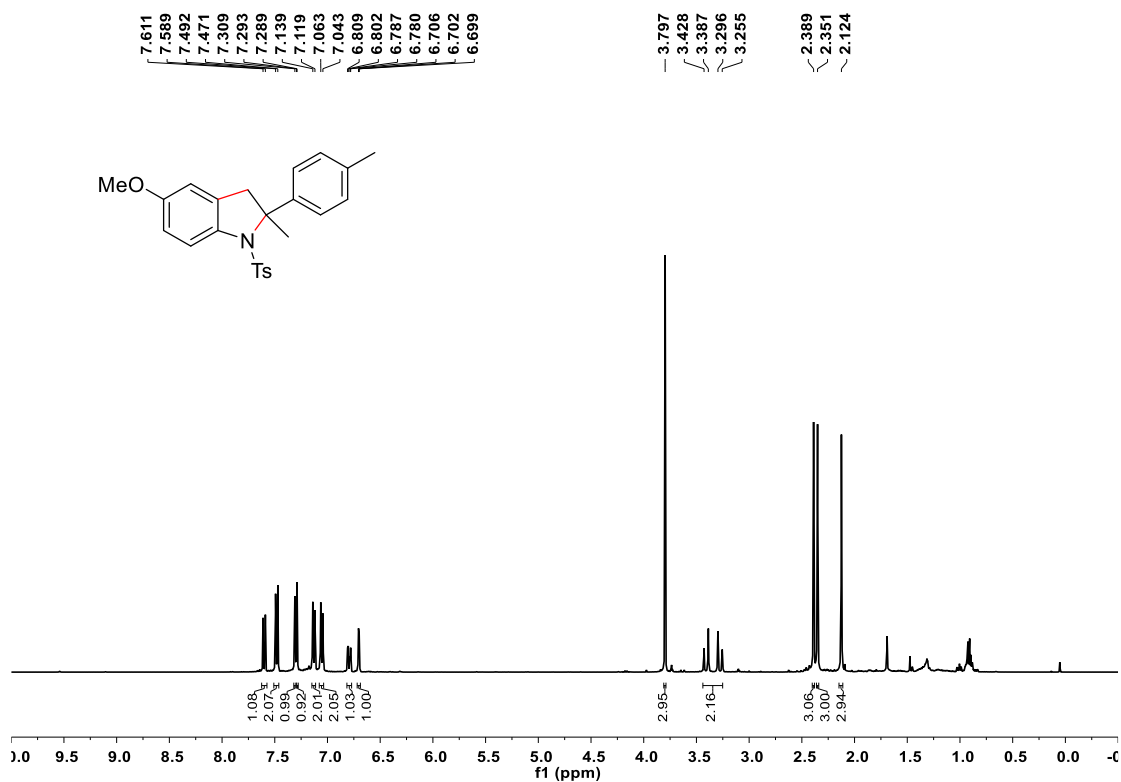


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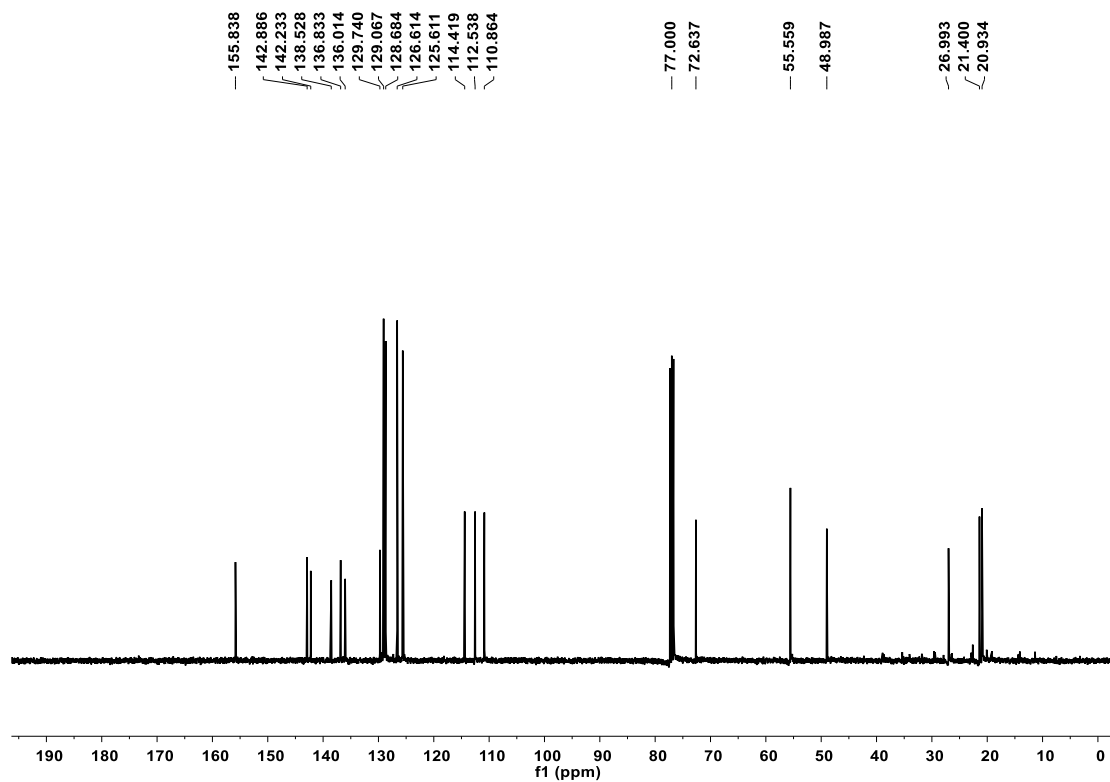


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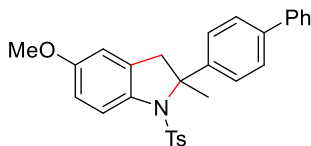
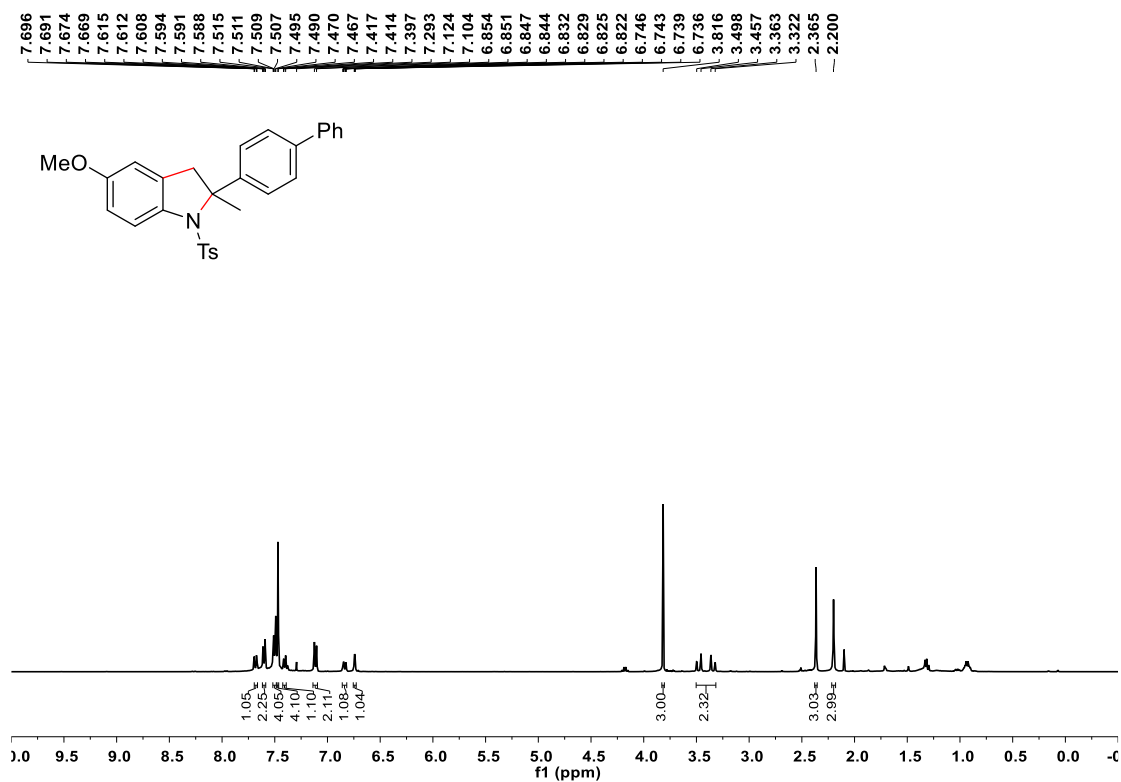


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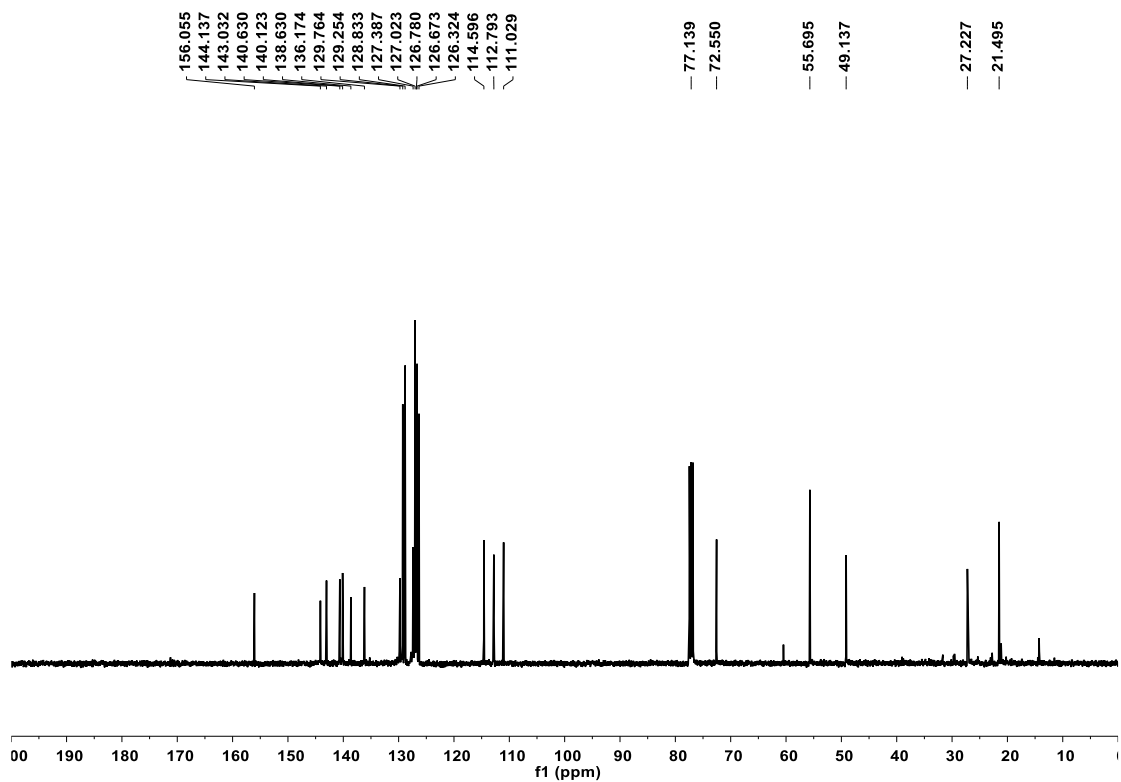


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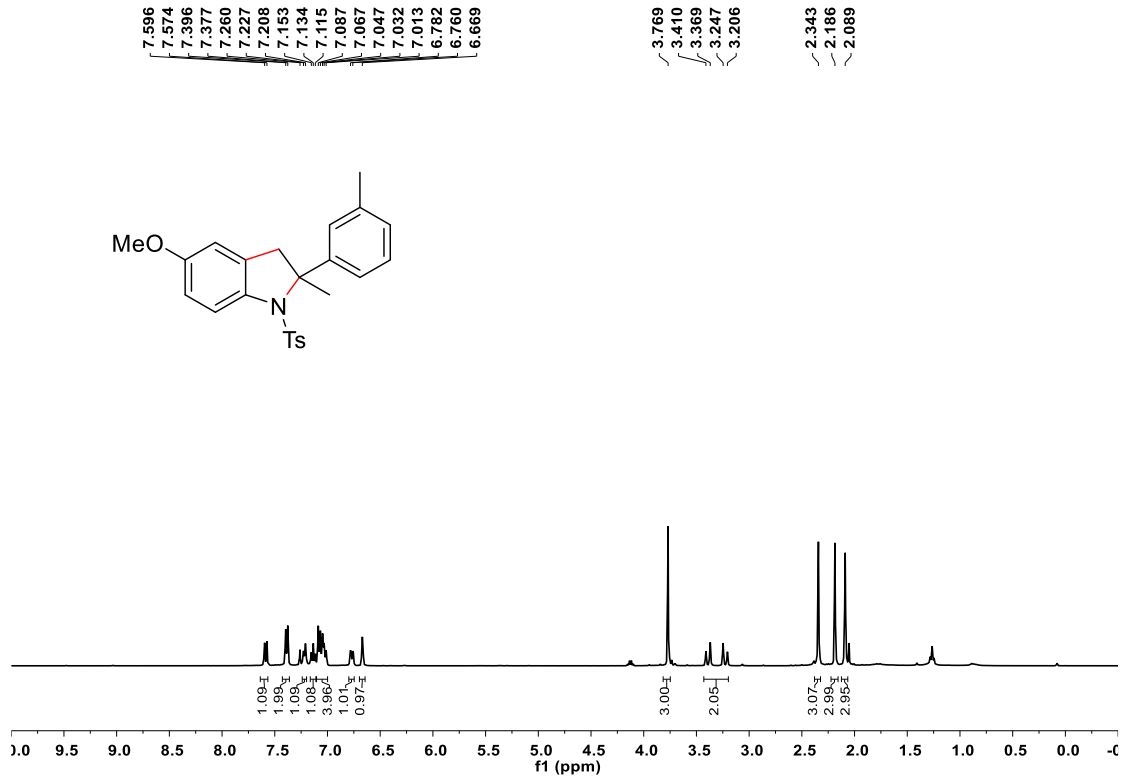


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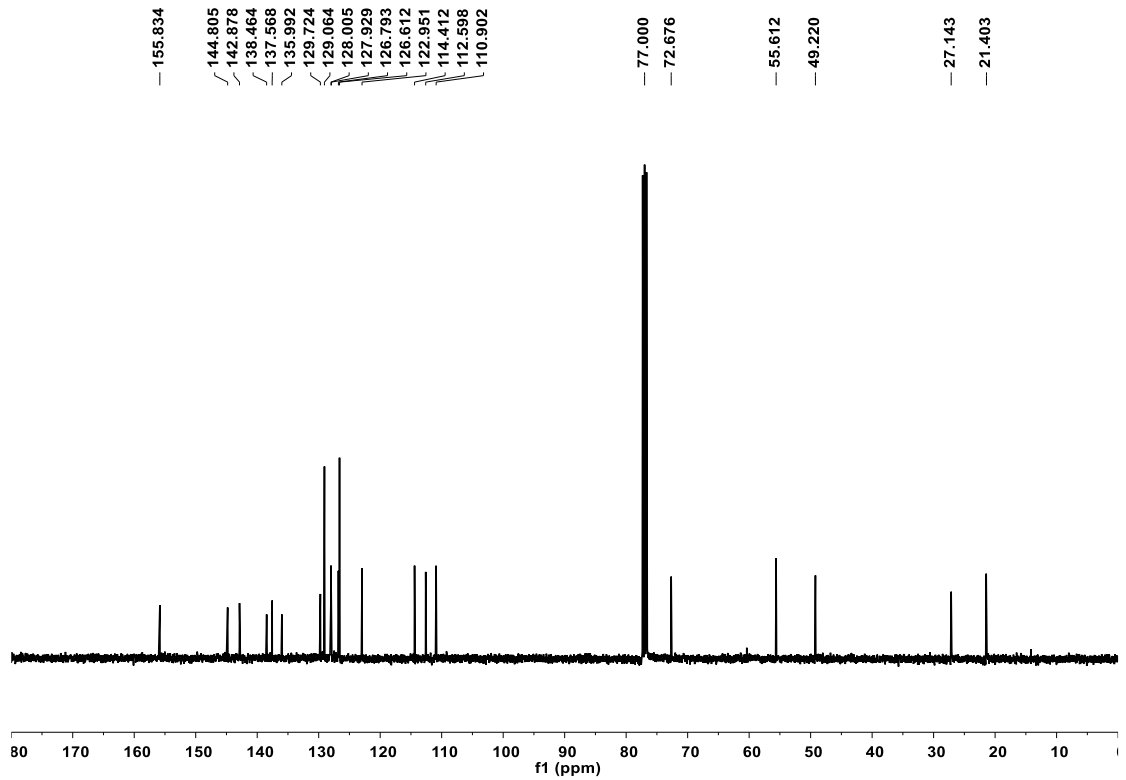


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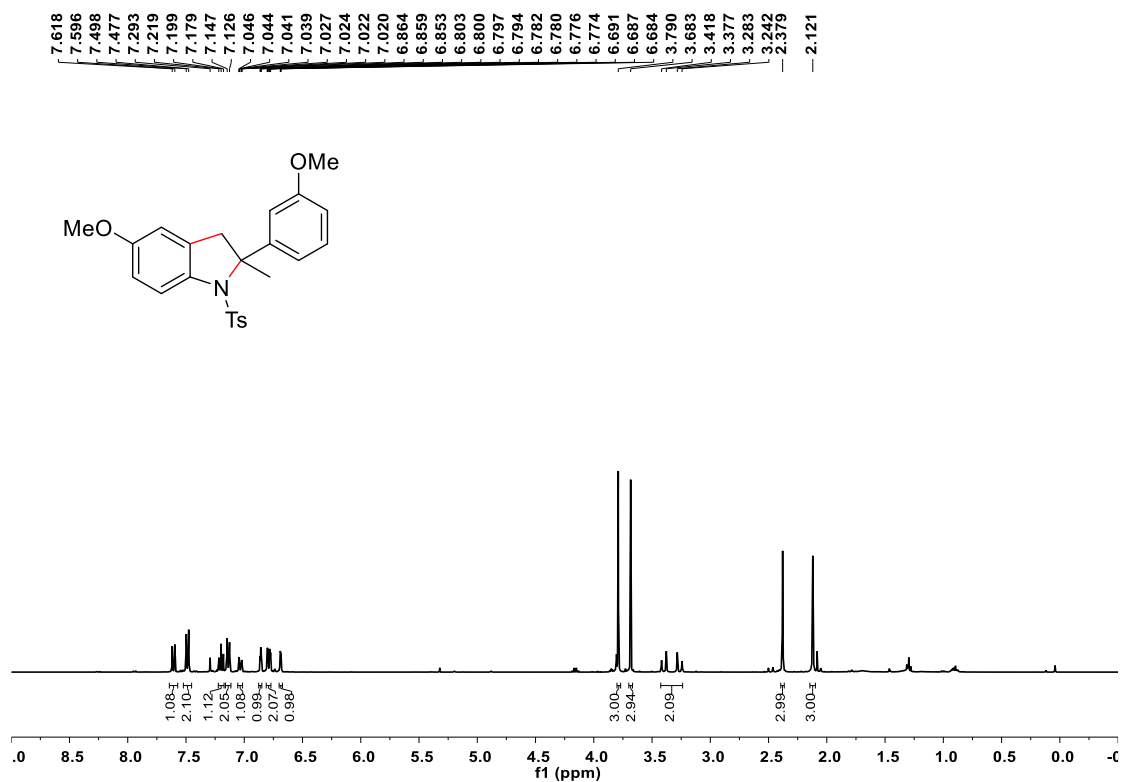


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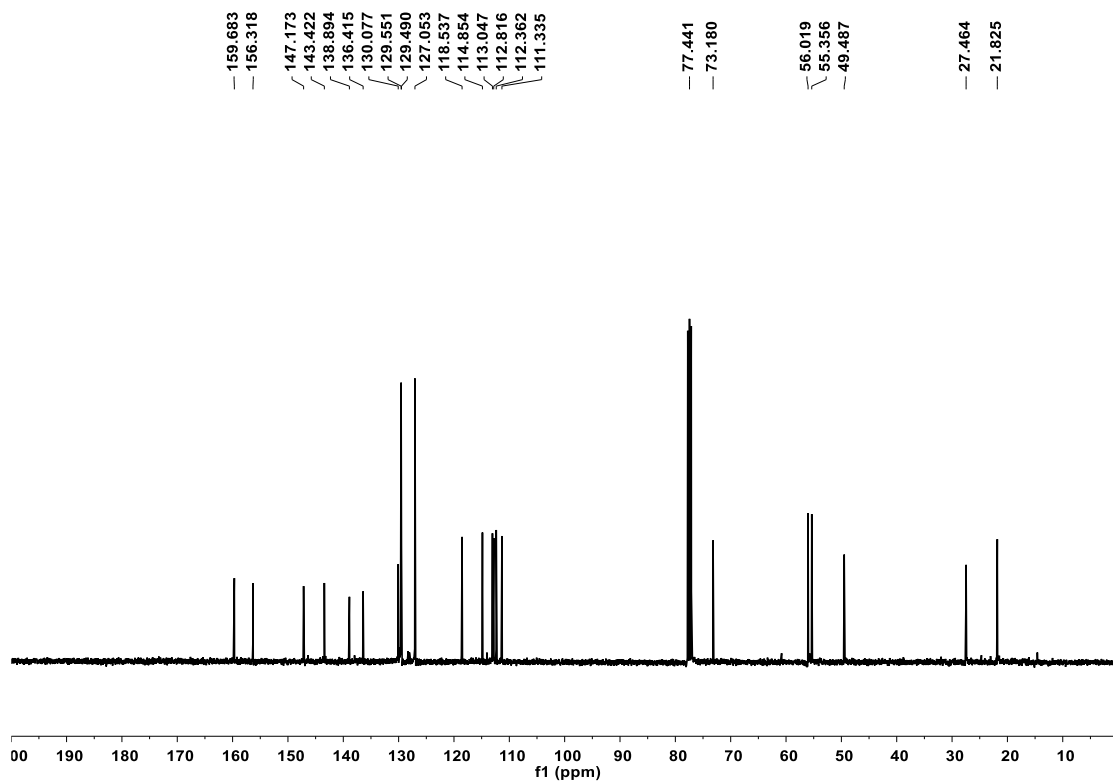


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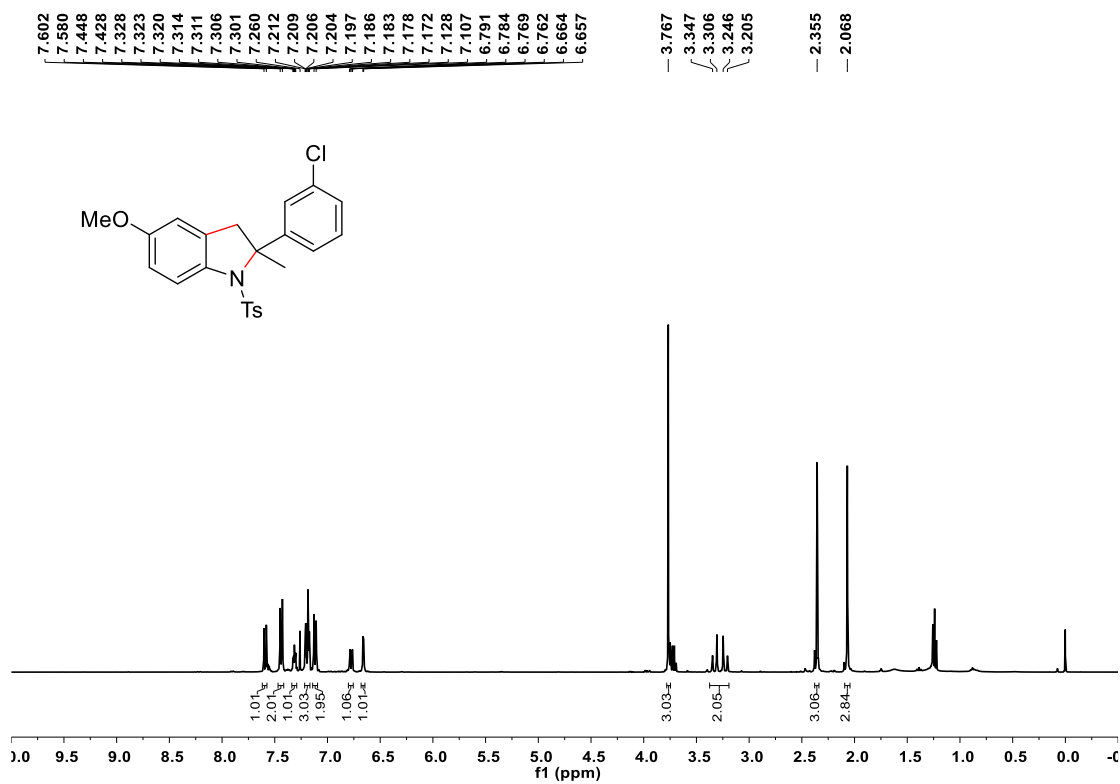


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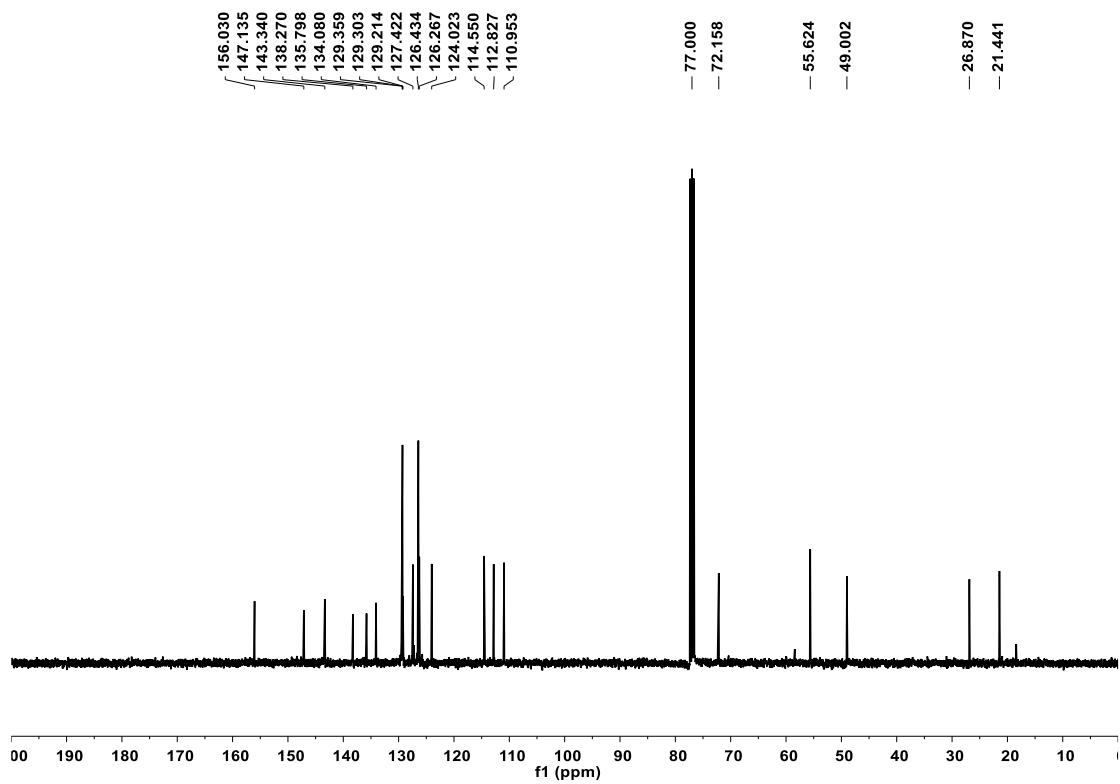


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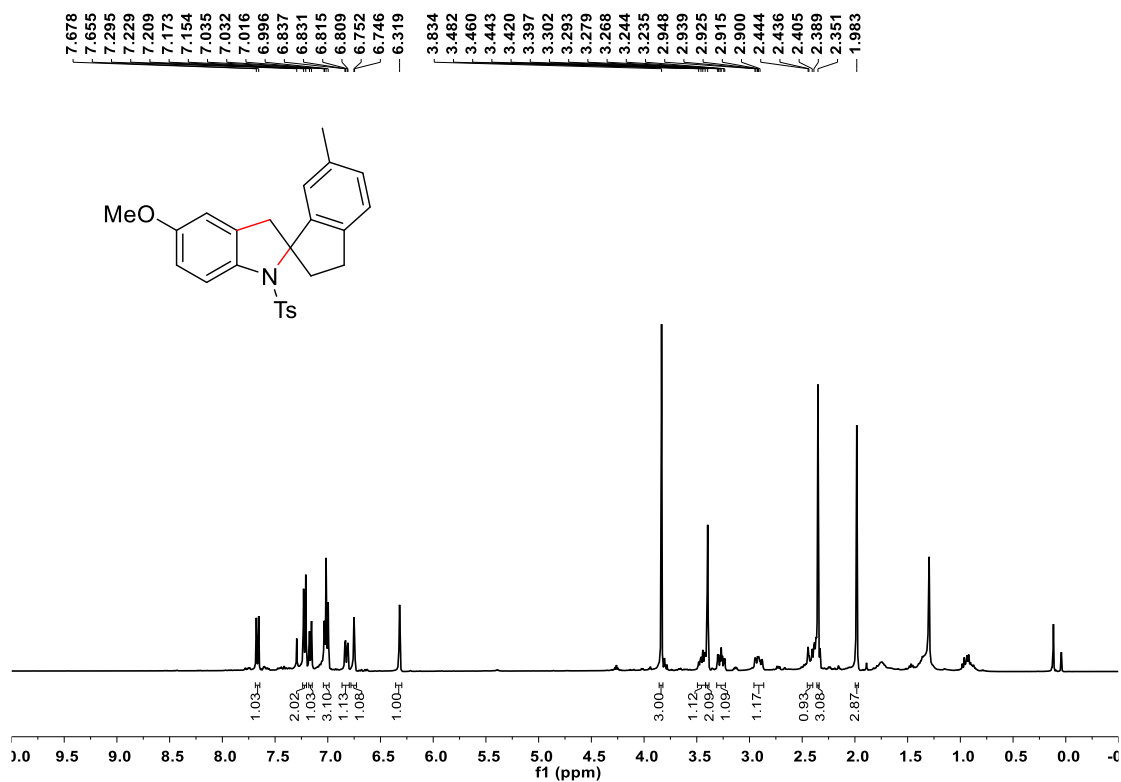


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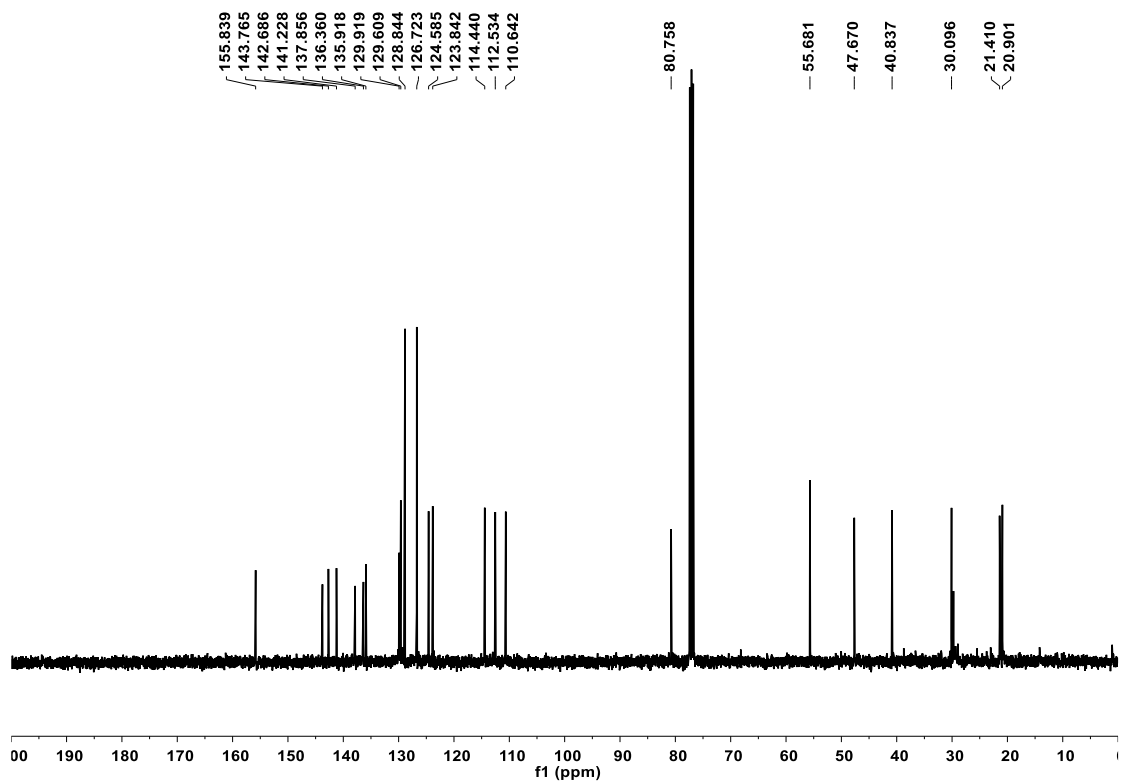


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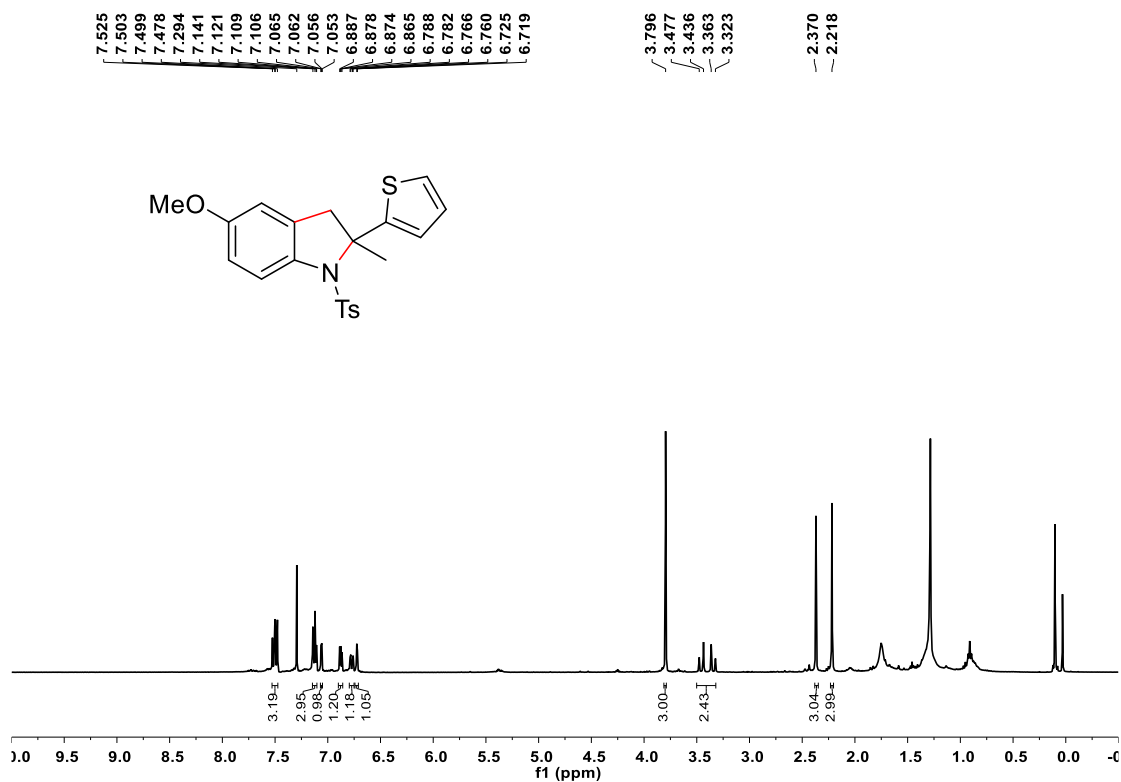


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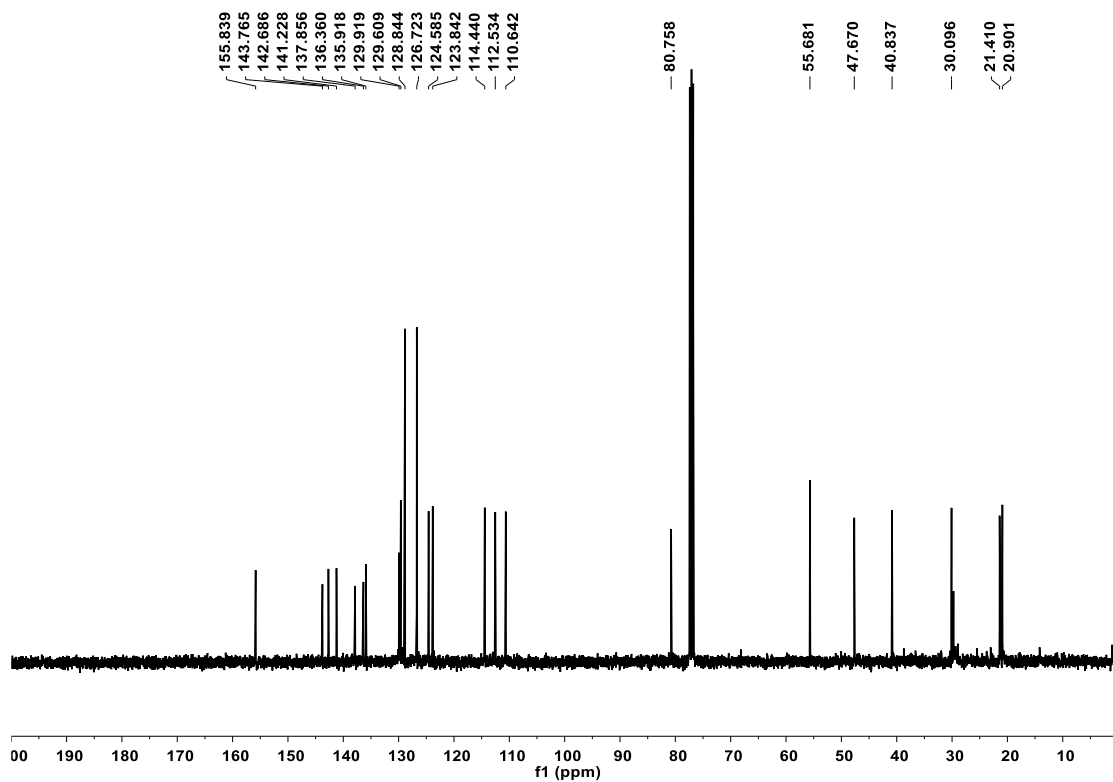


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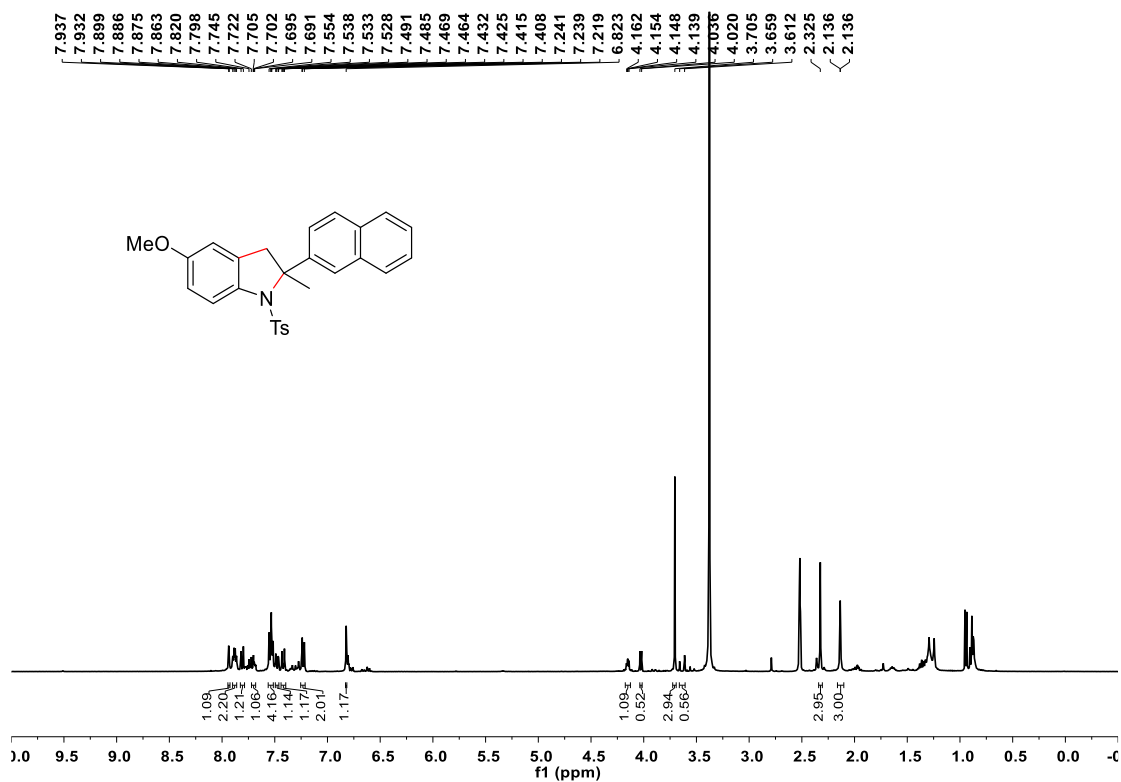


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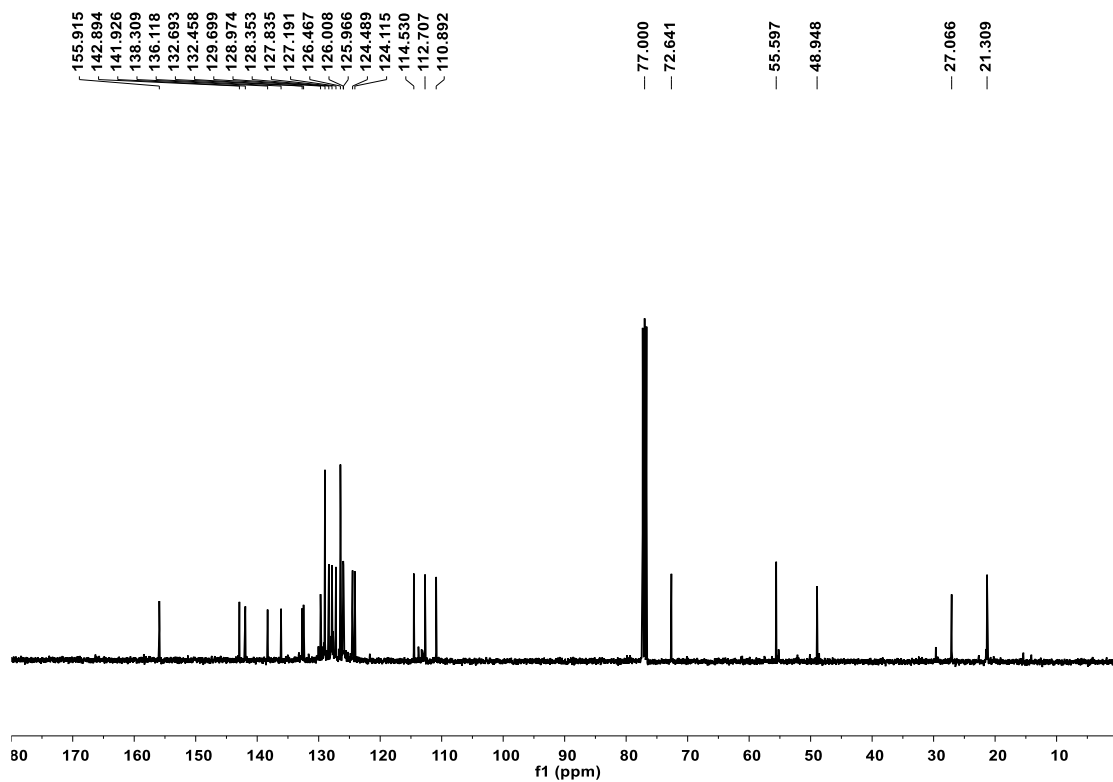


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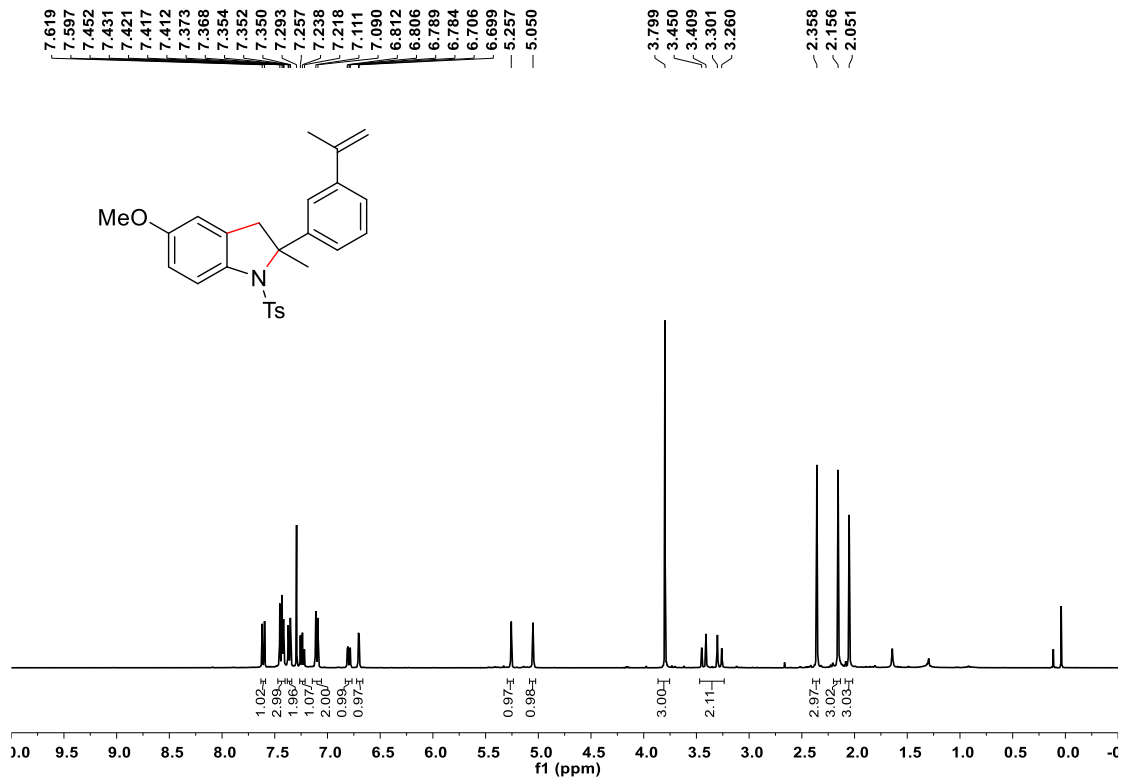


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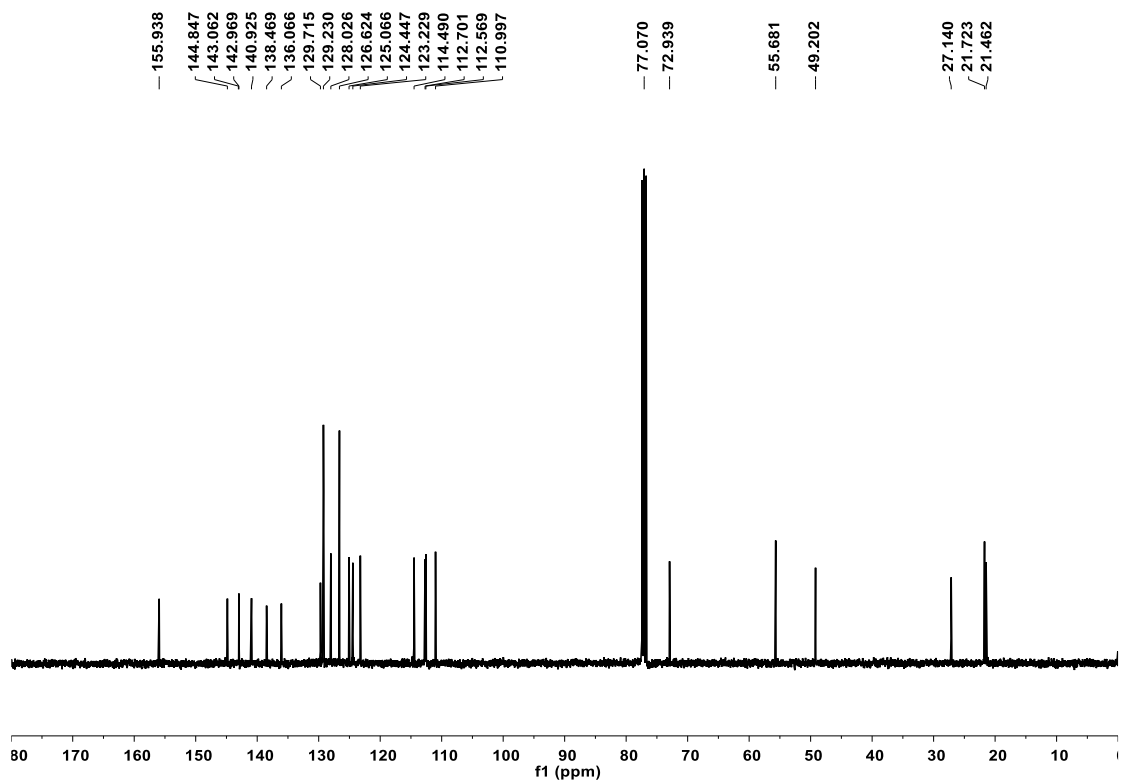


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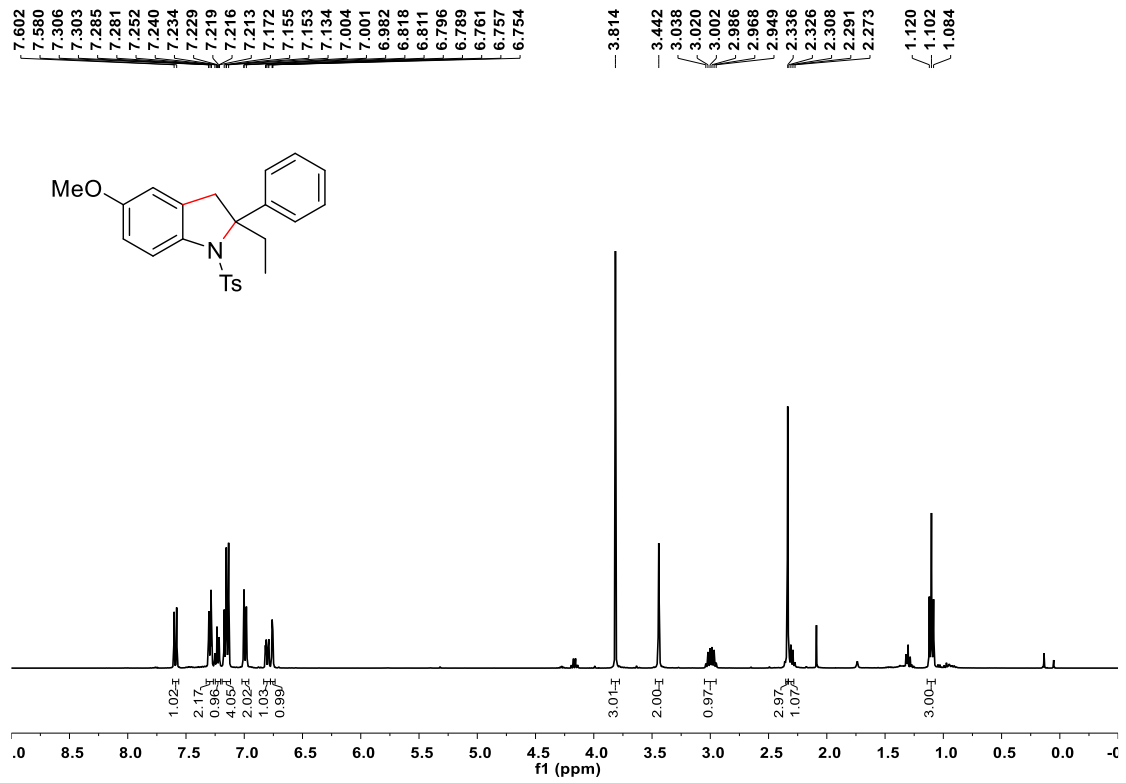


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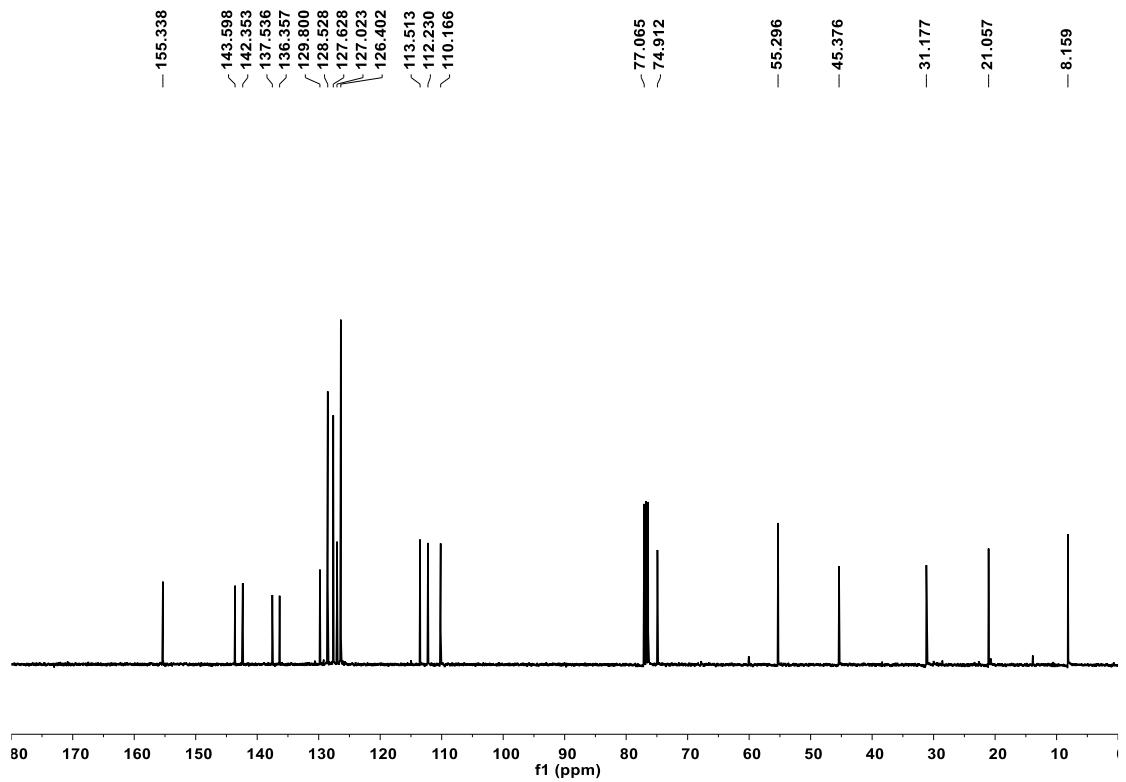


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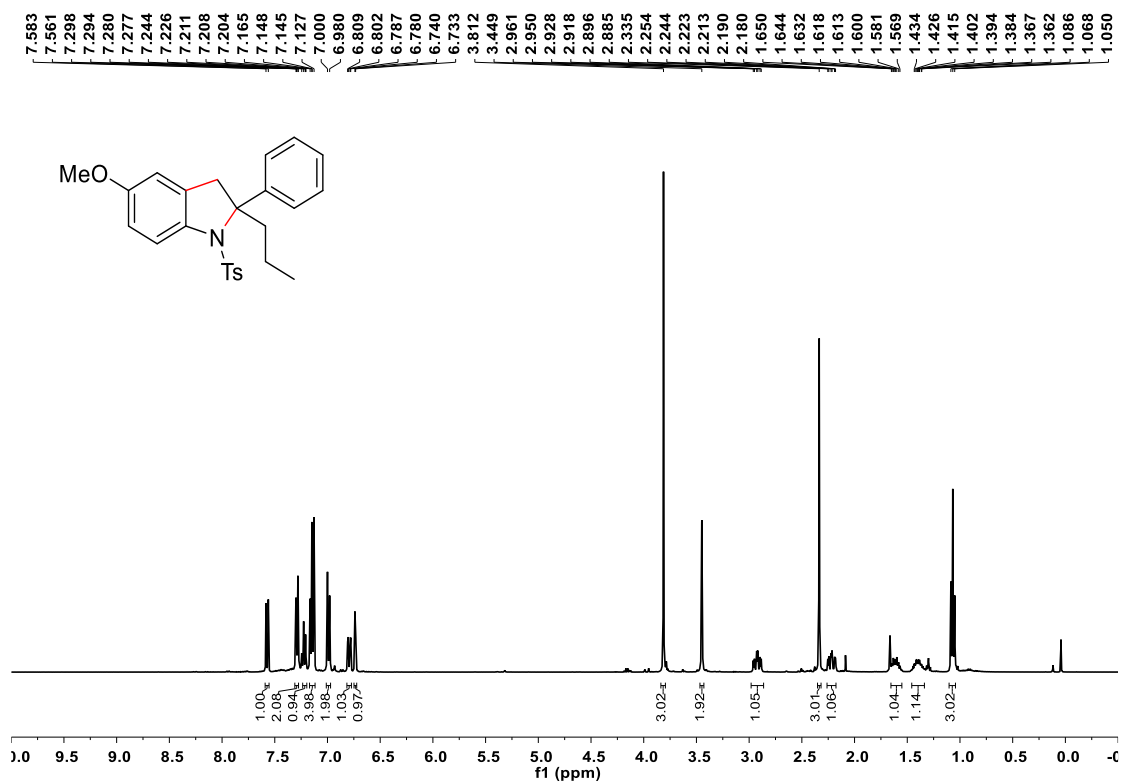


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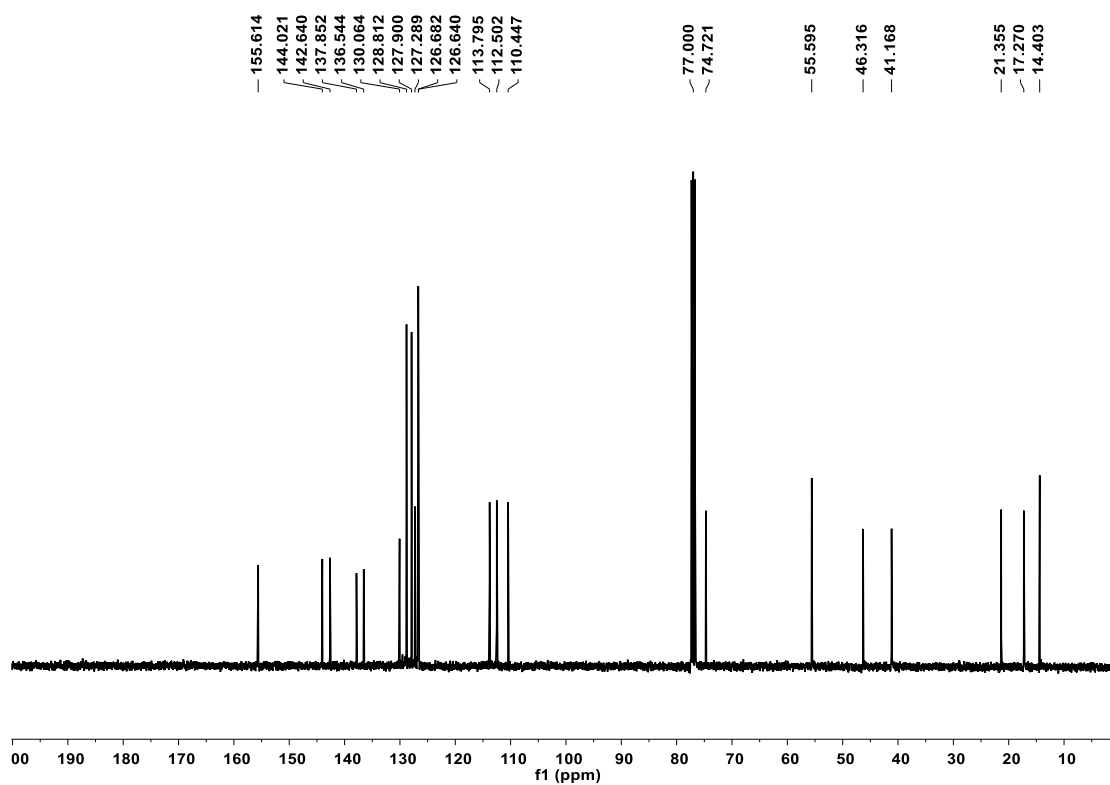


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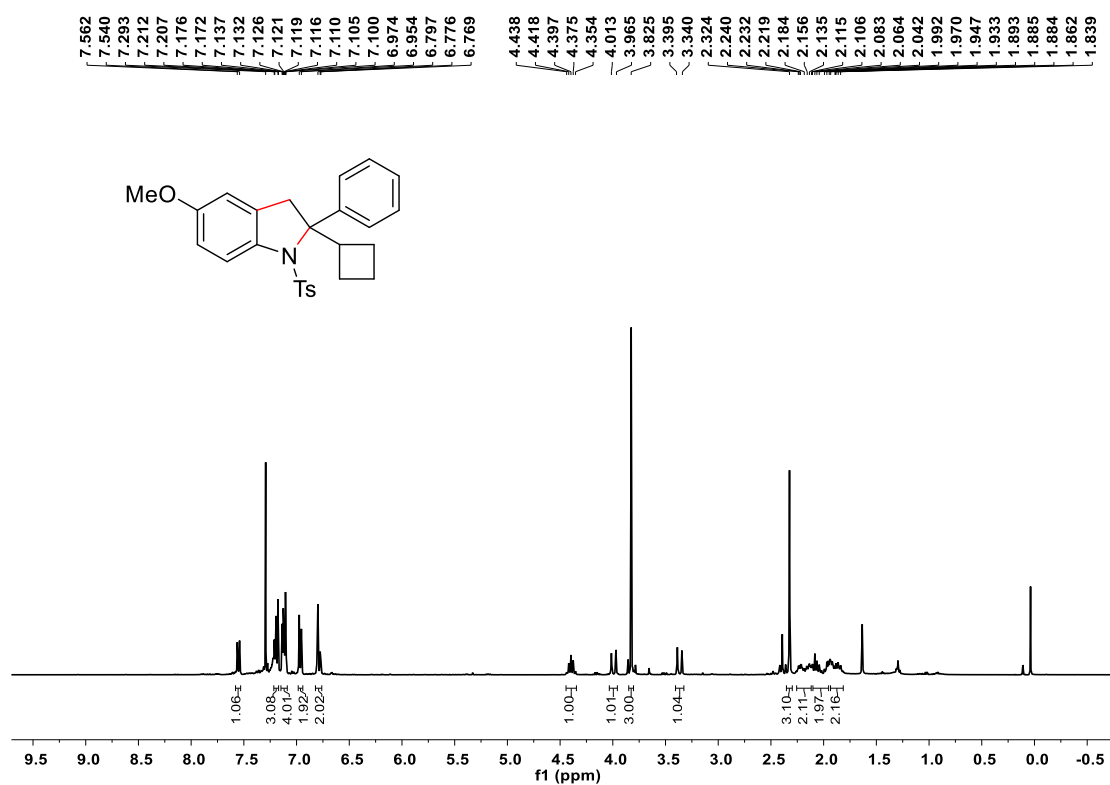


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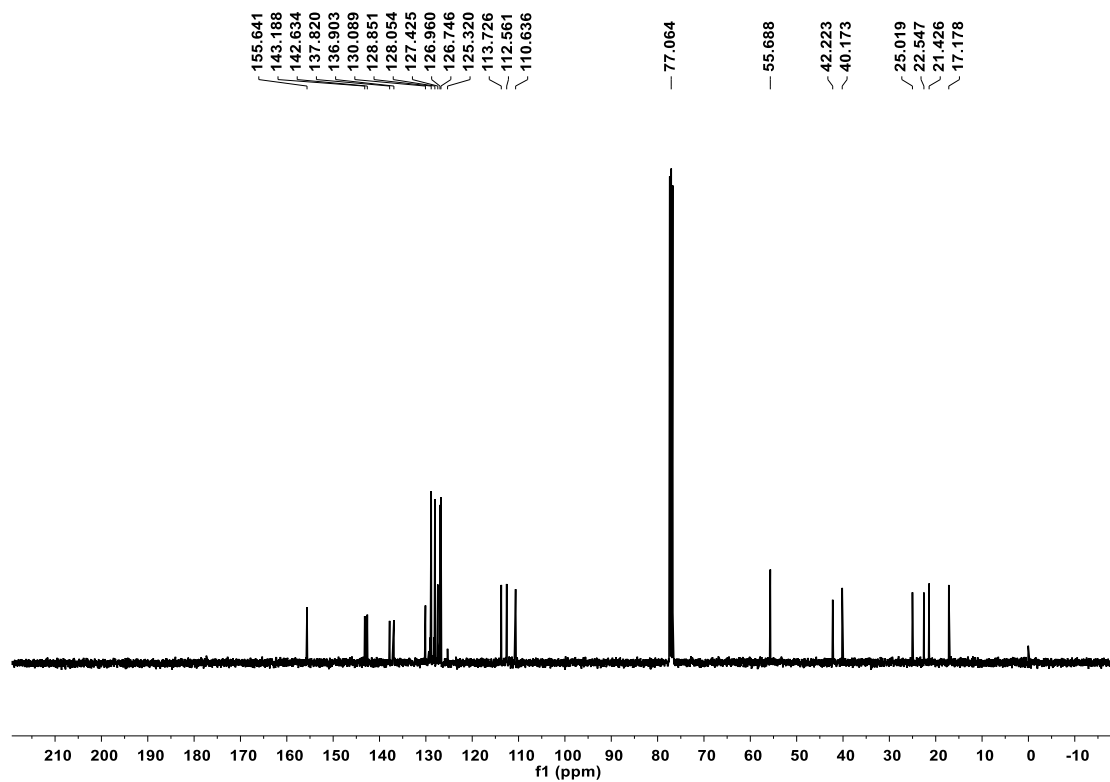


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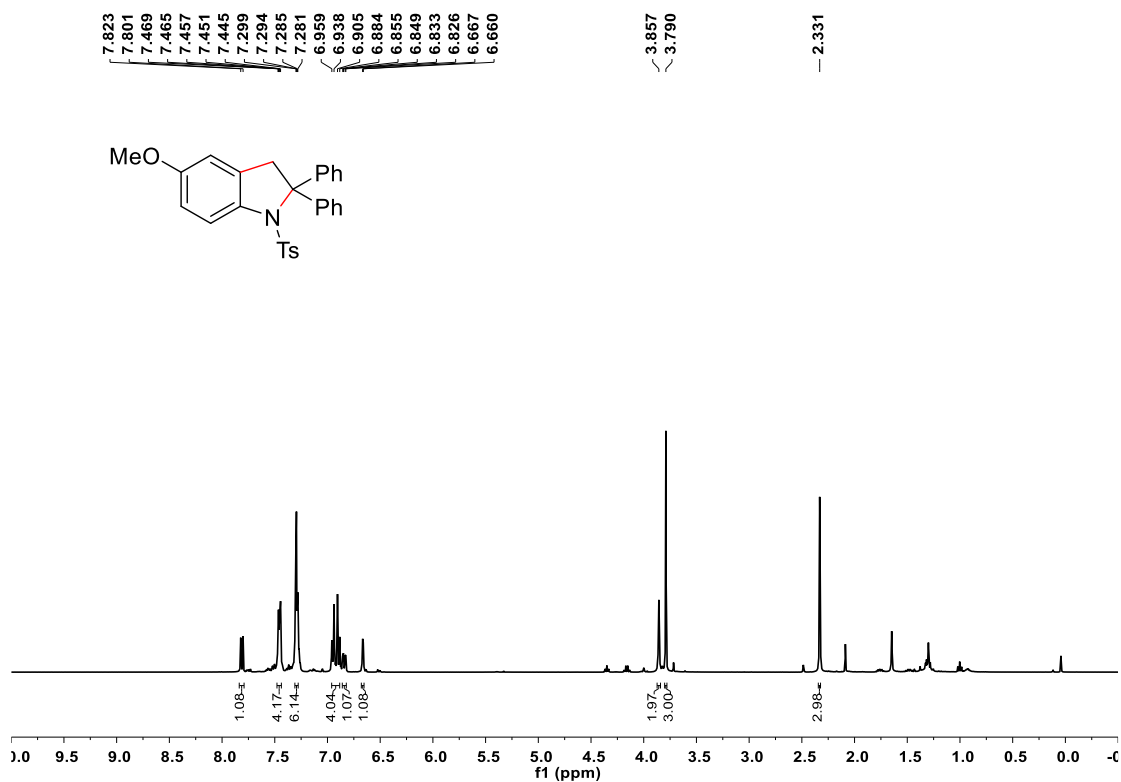


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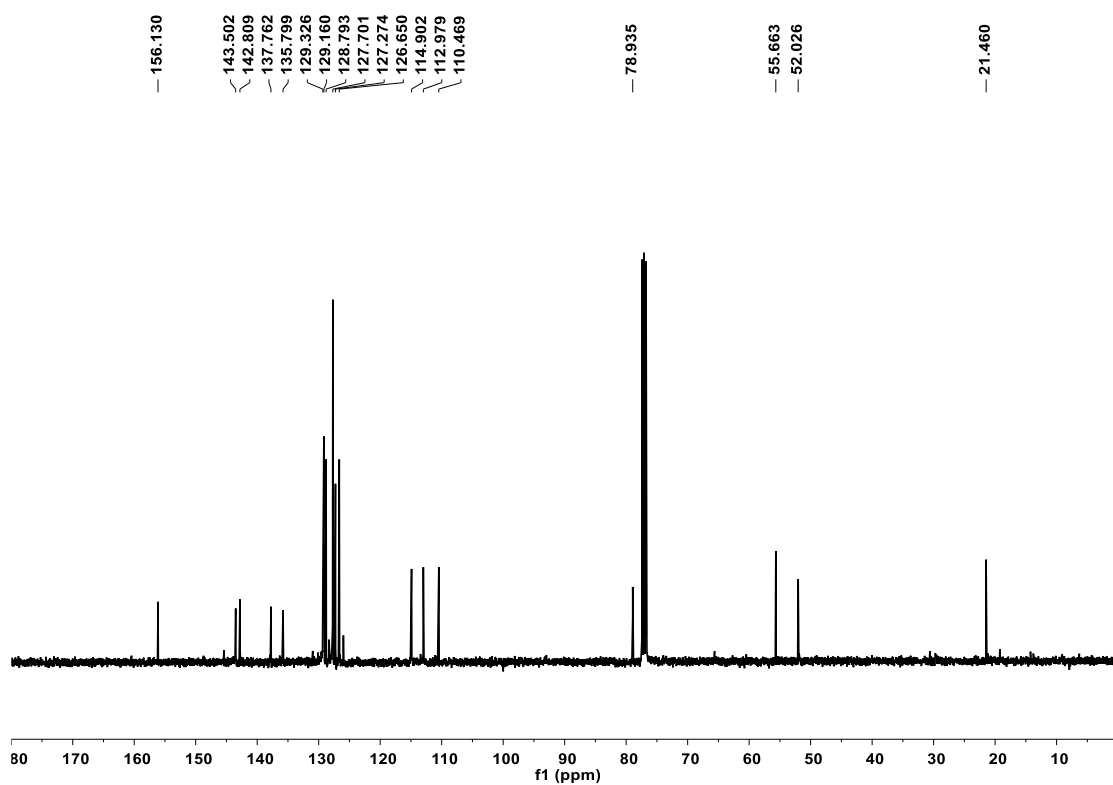


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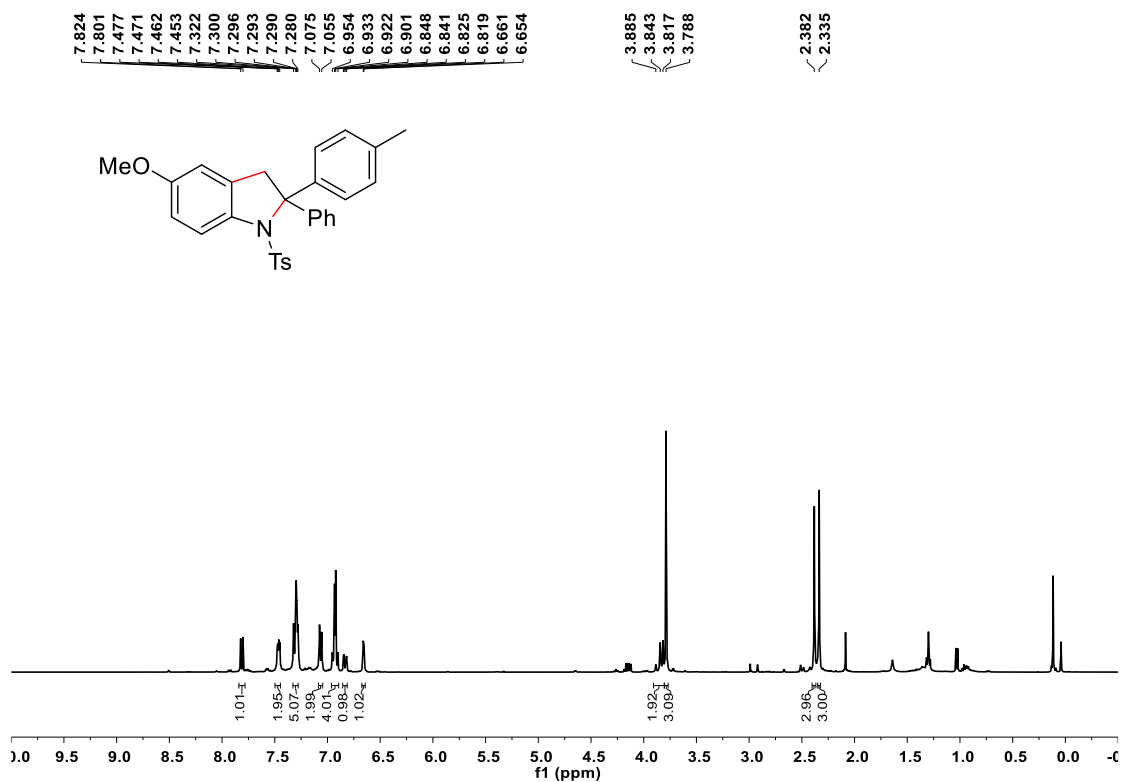


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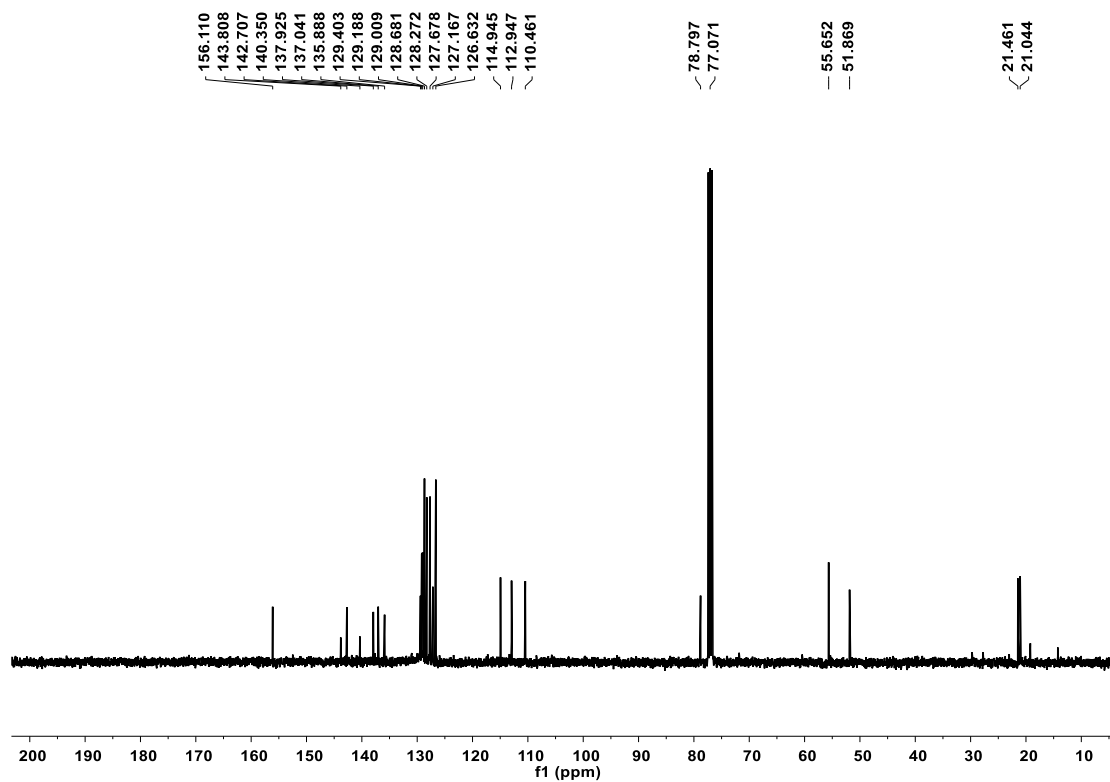


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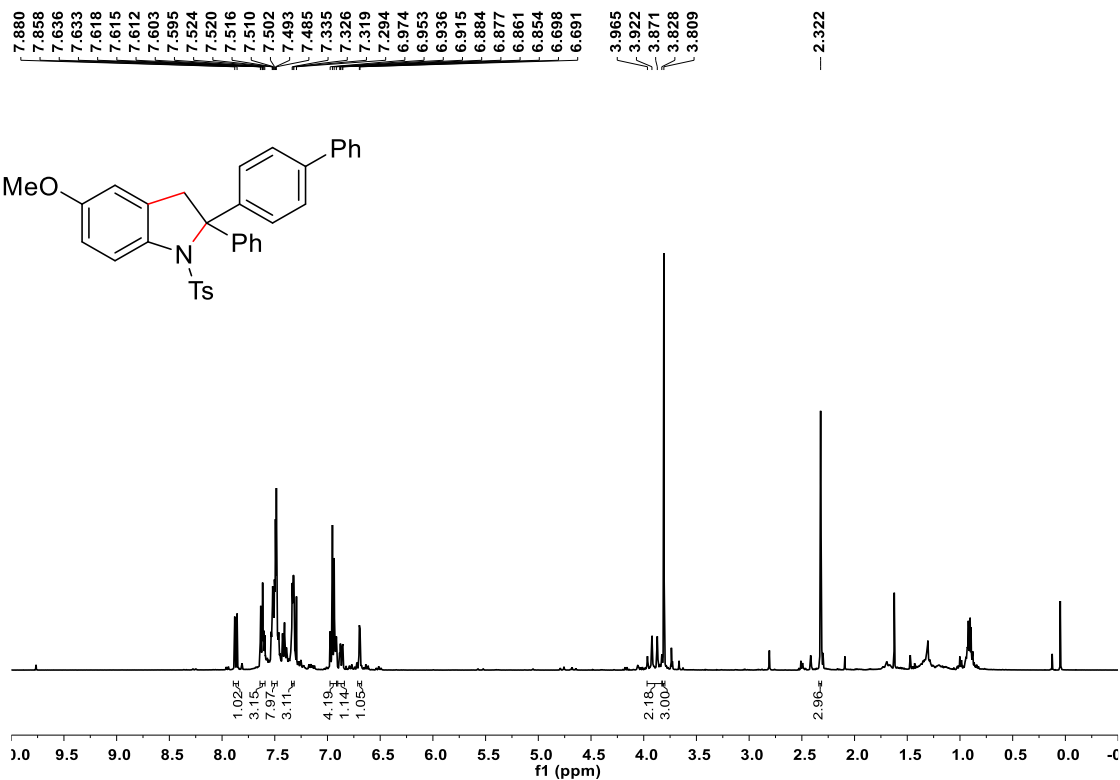


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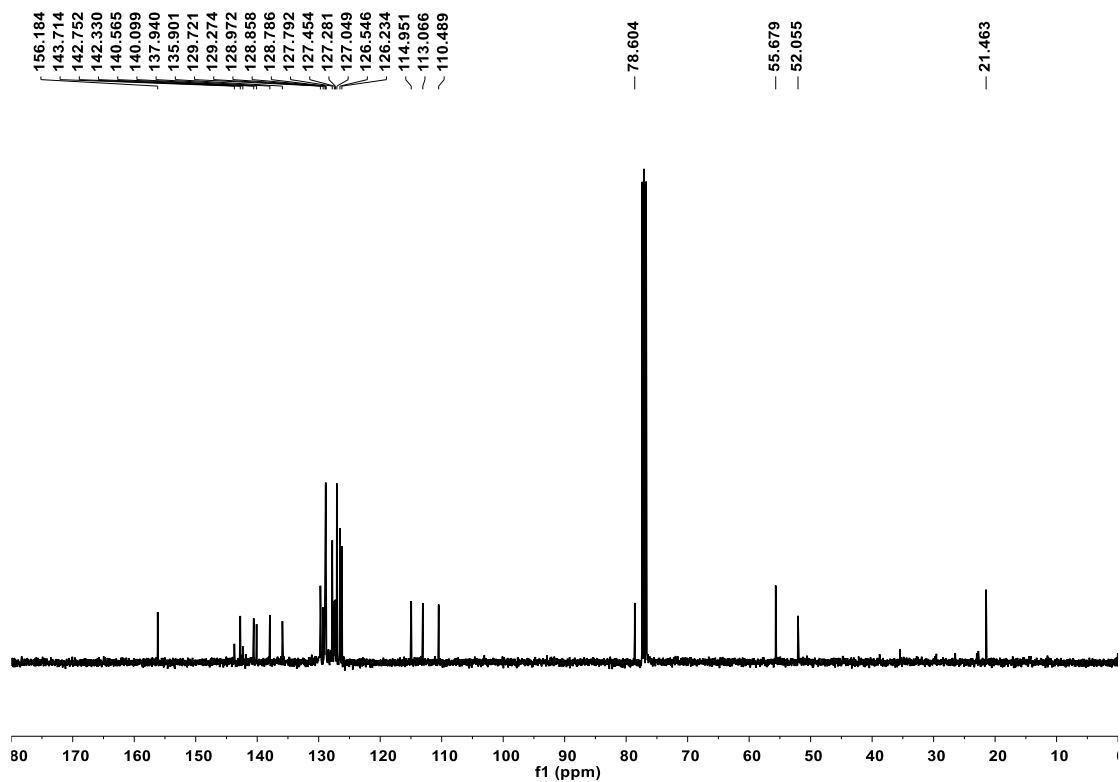


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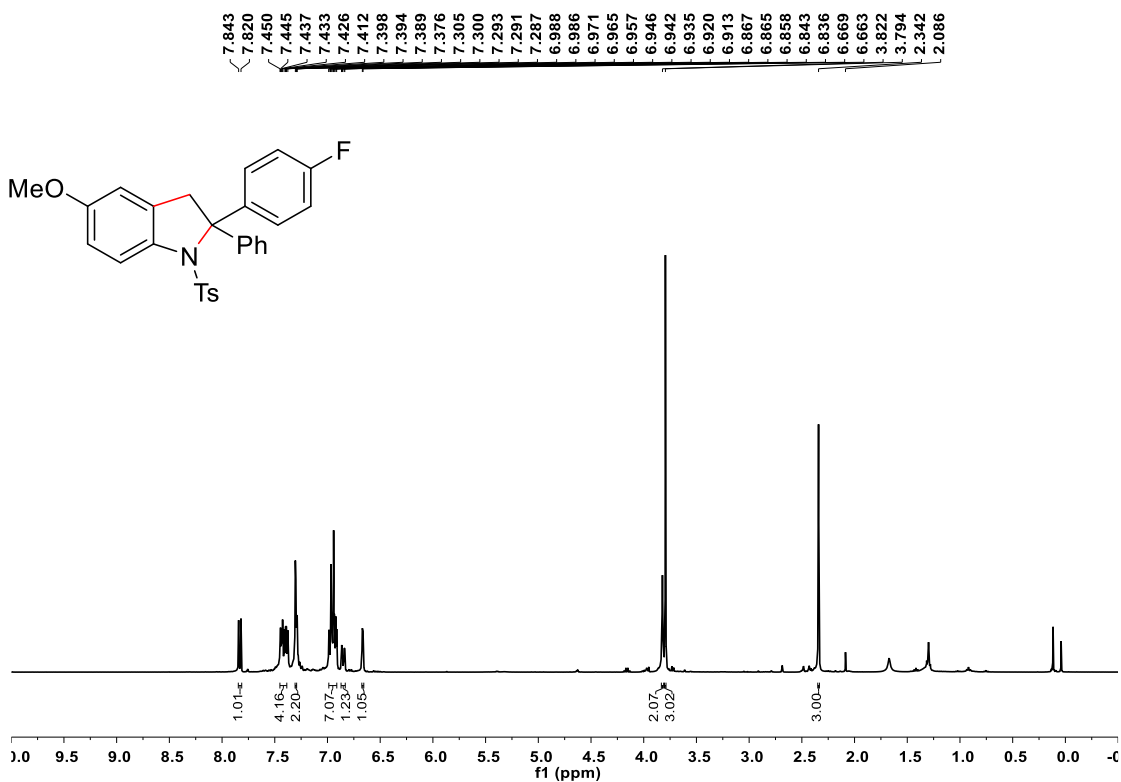


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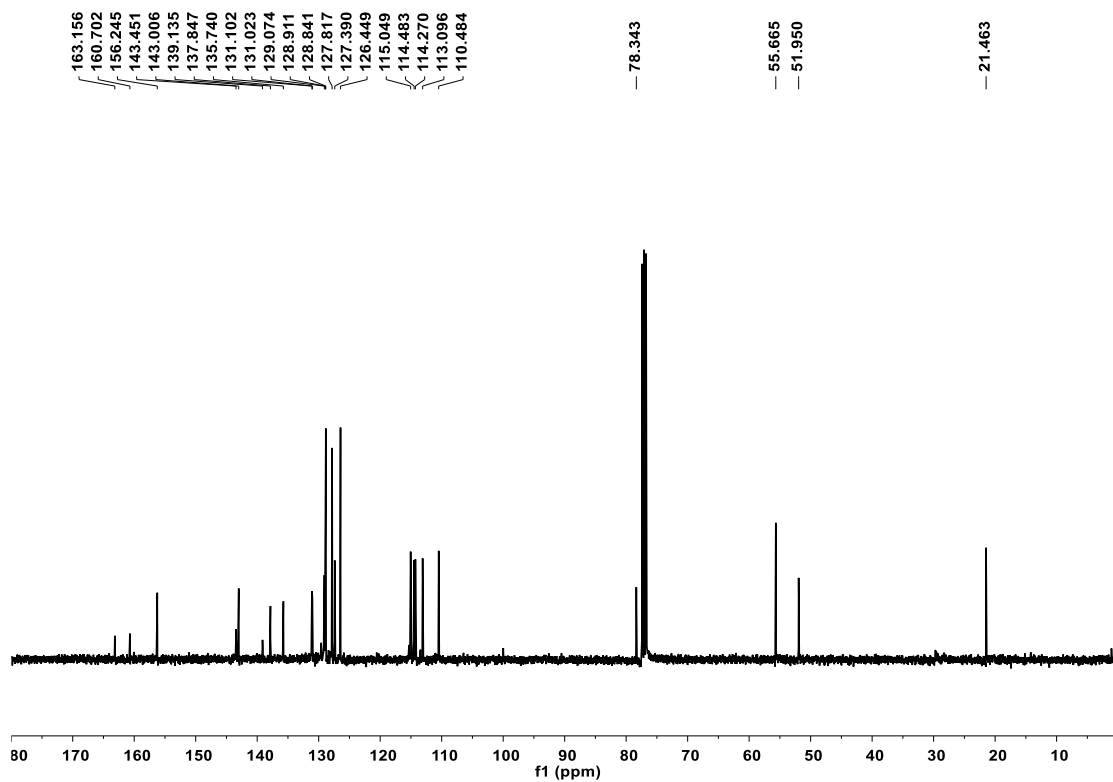


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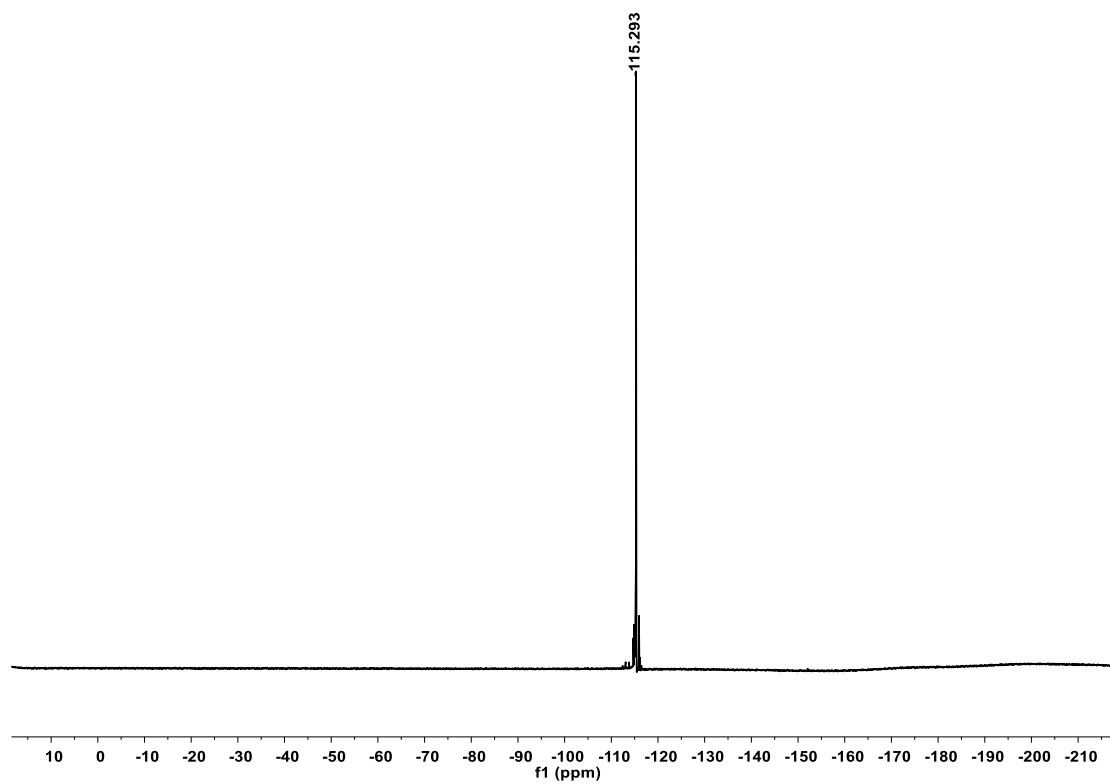
¹H NMR



¹³C NMR

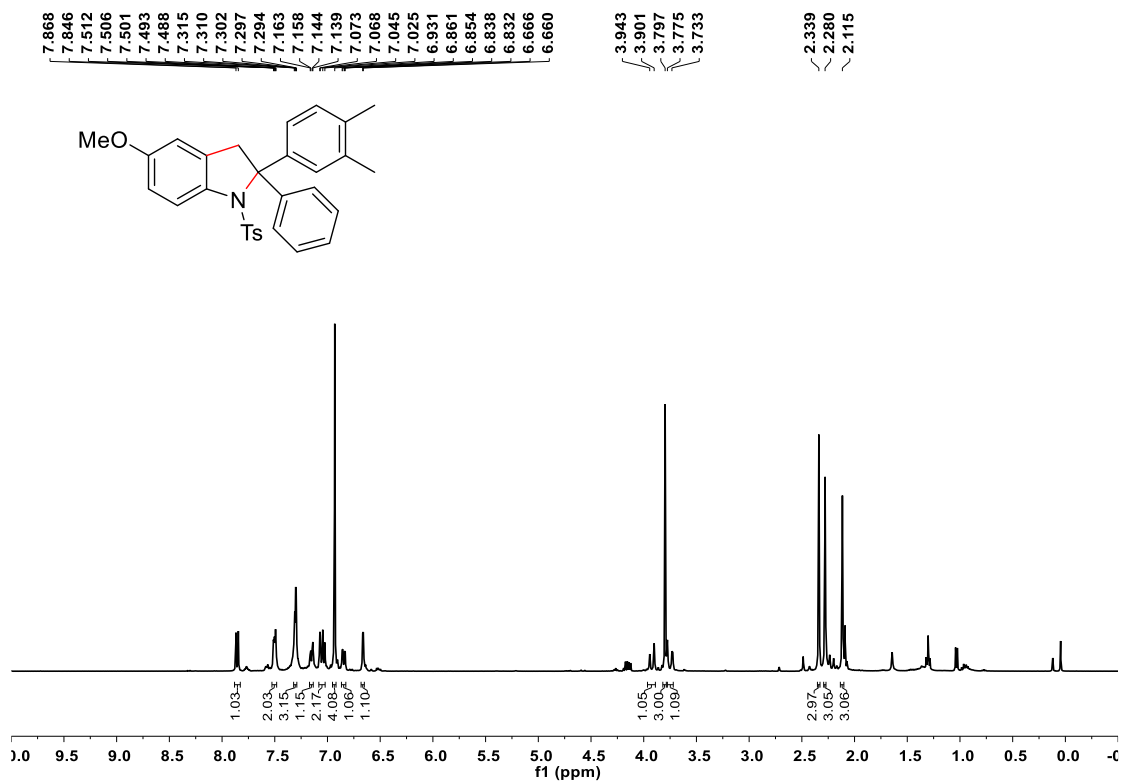


¹³C NMR



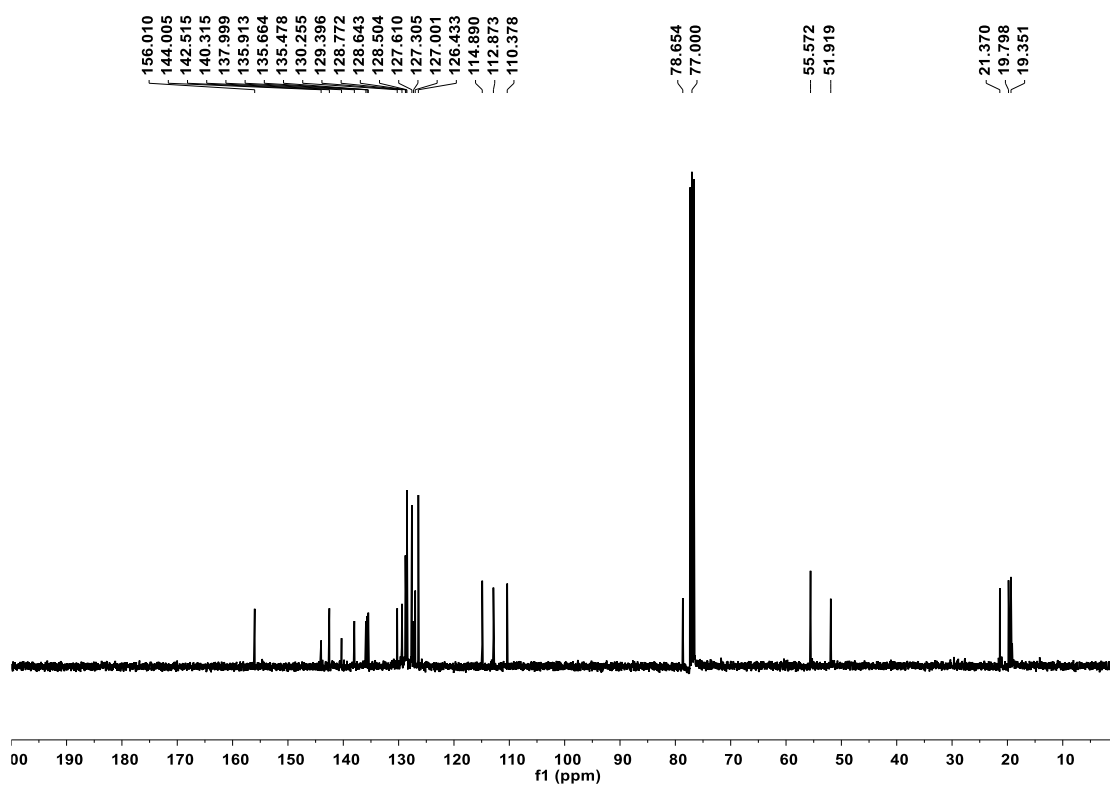
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¹H NMR



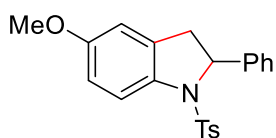
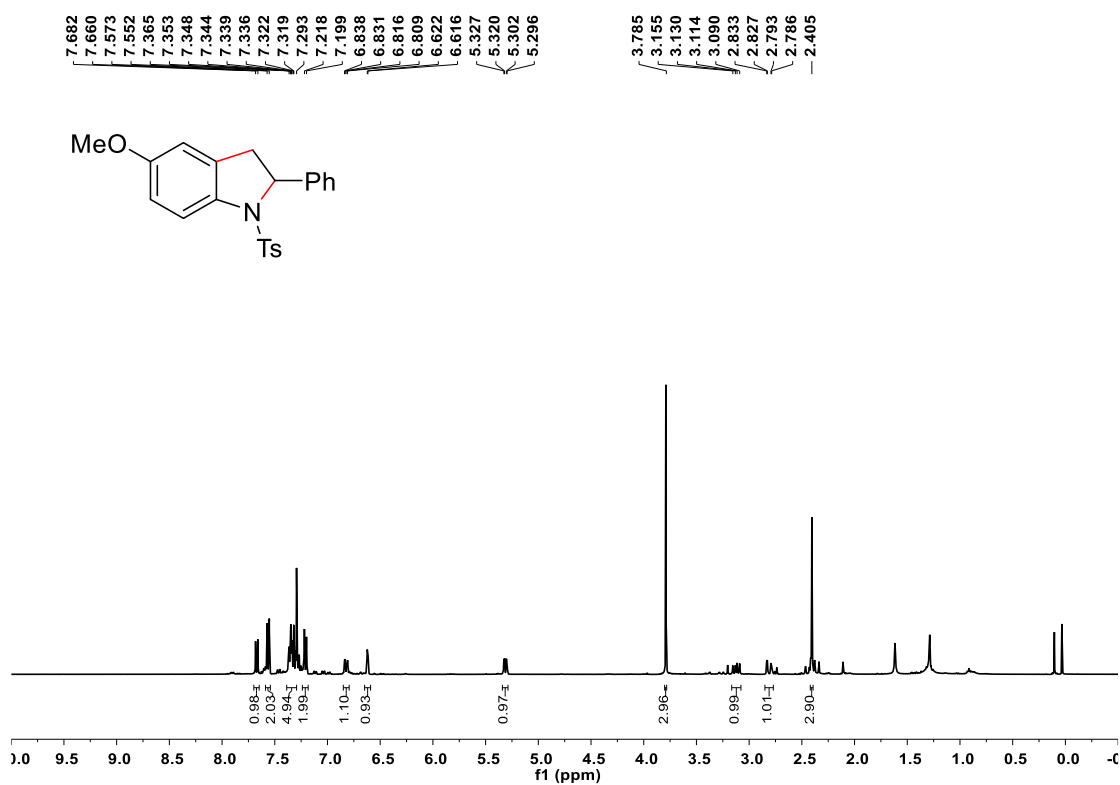
S53

¹³C NMR

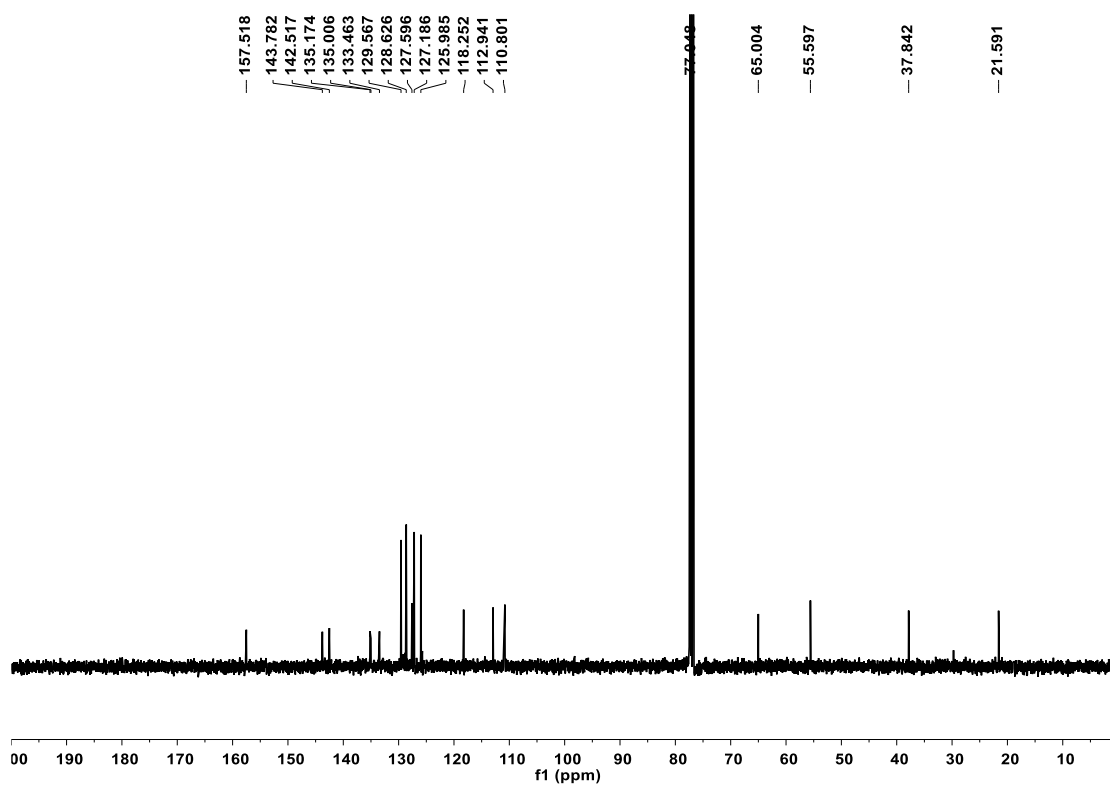


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¹H NMR

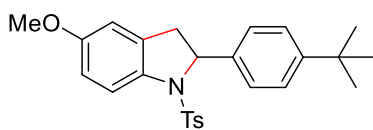
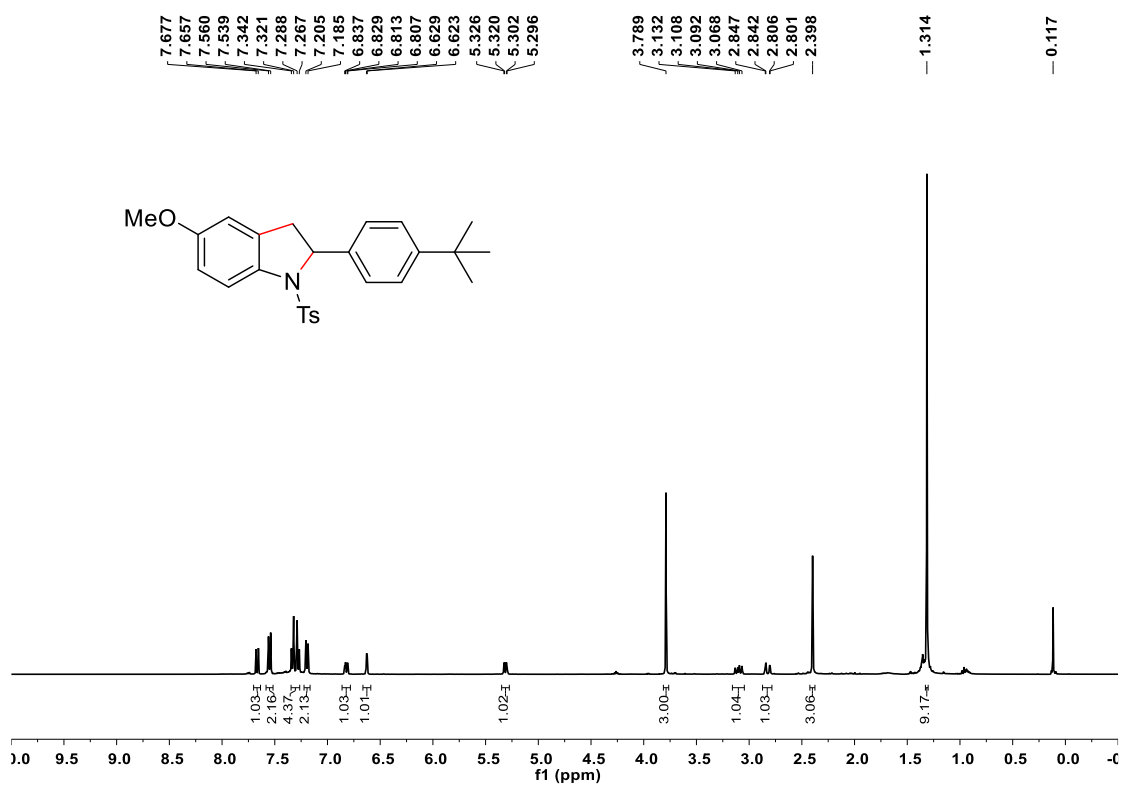


¹³C NMR



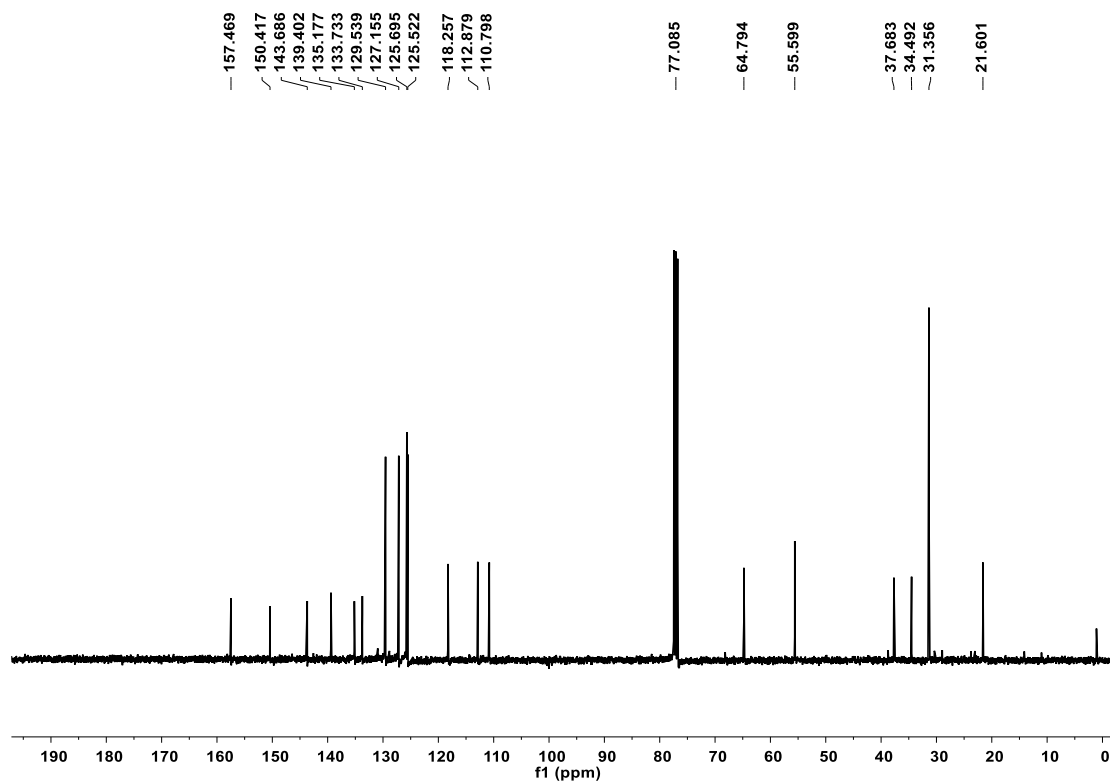
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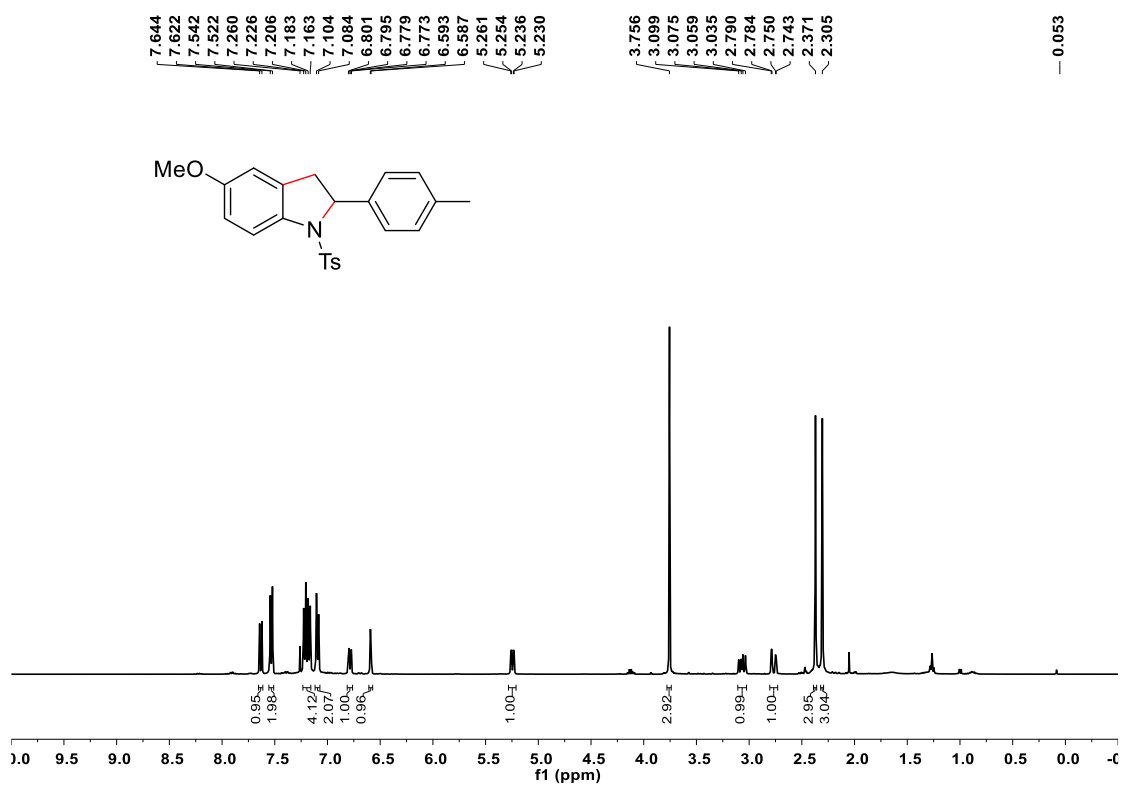
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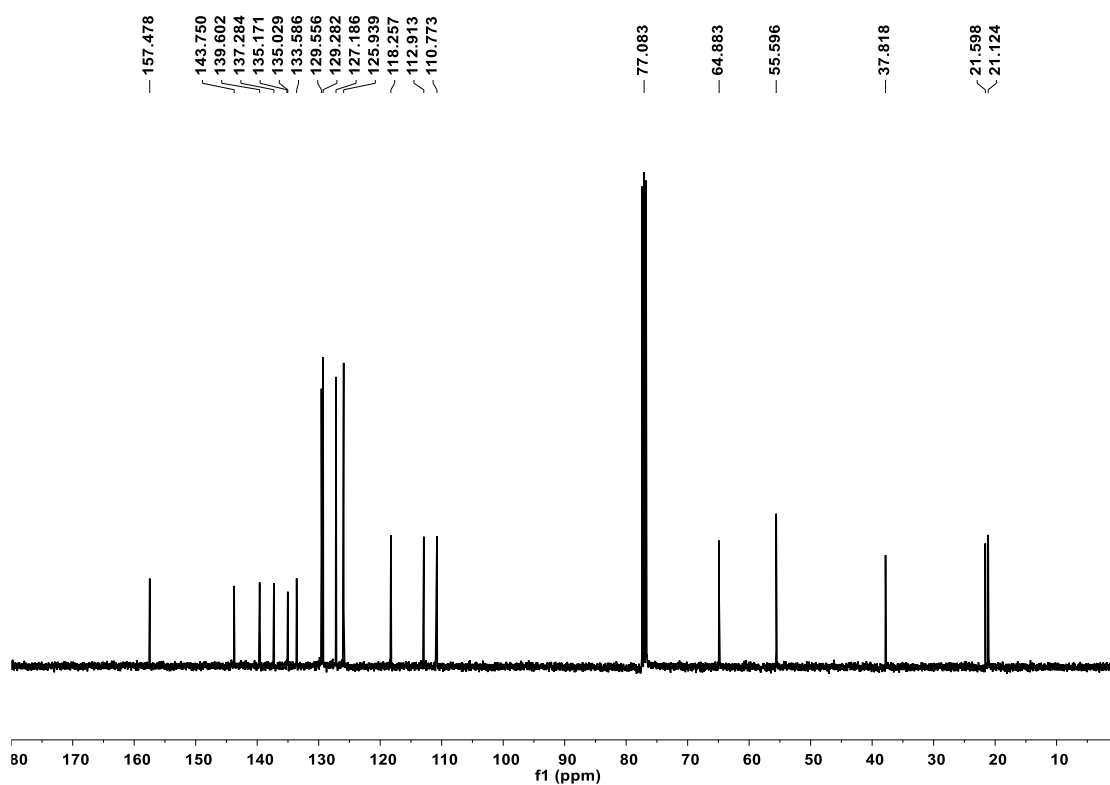


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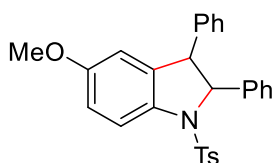
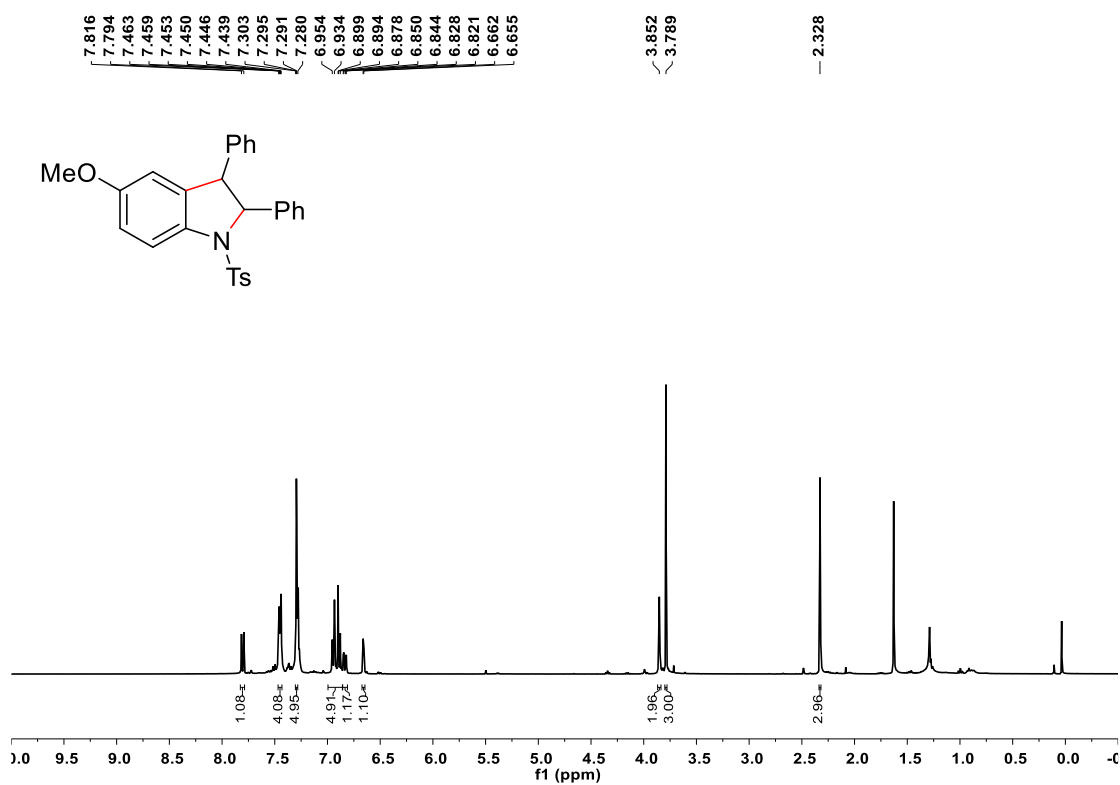


¹³C NMR



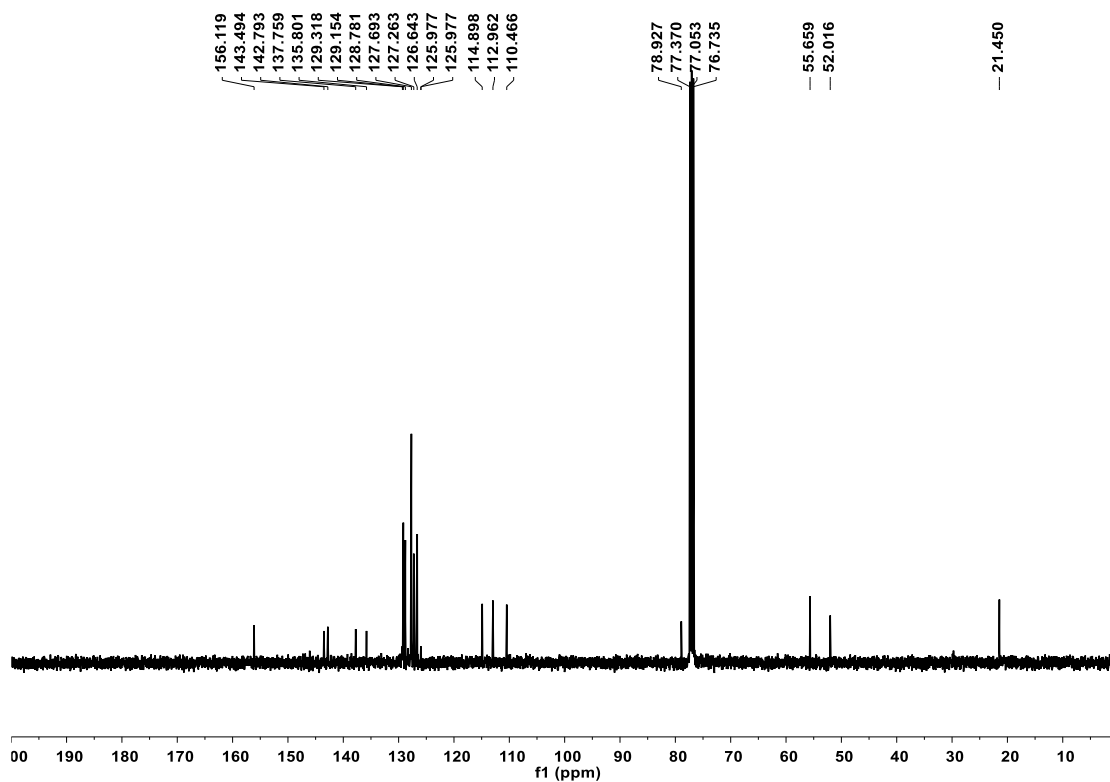
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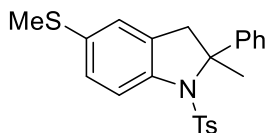
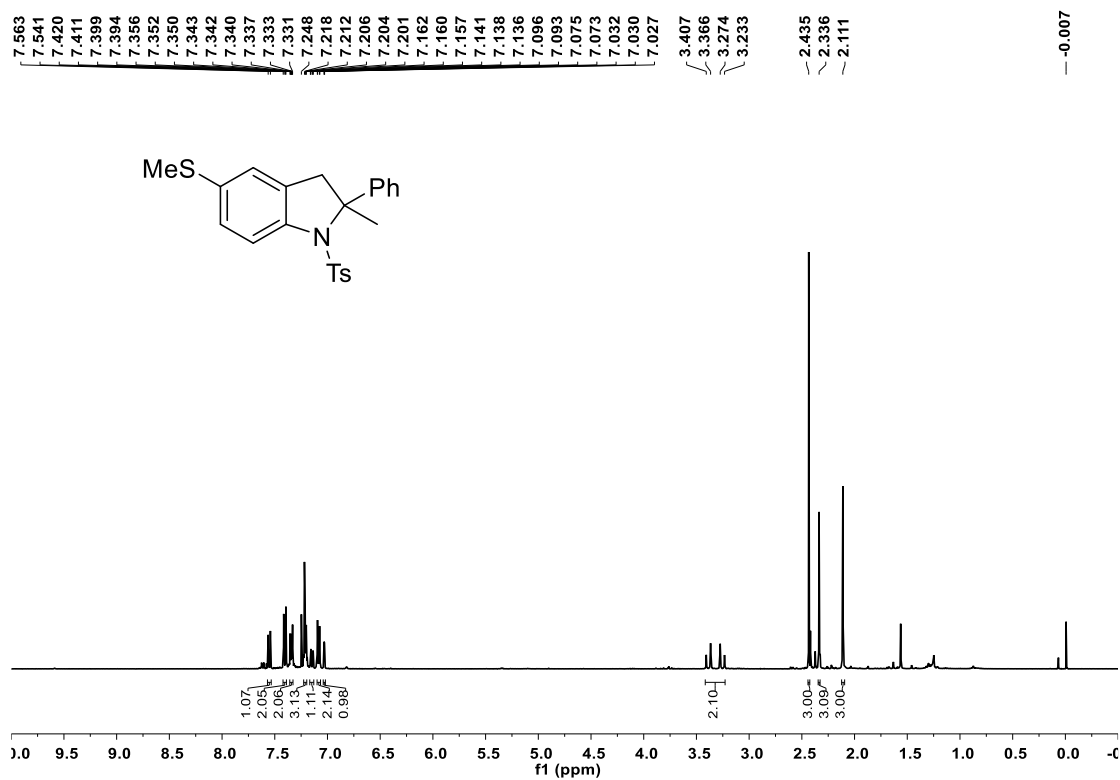
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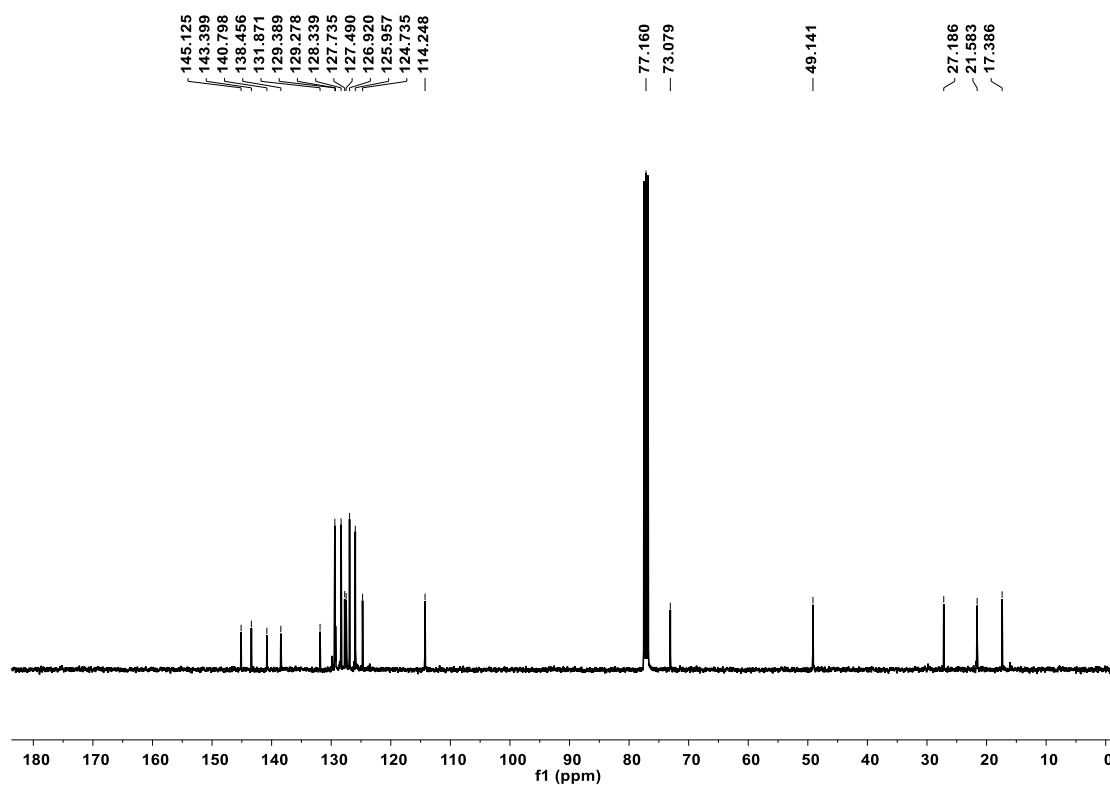


3ba

¹H NMR

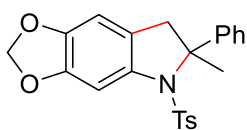
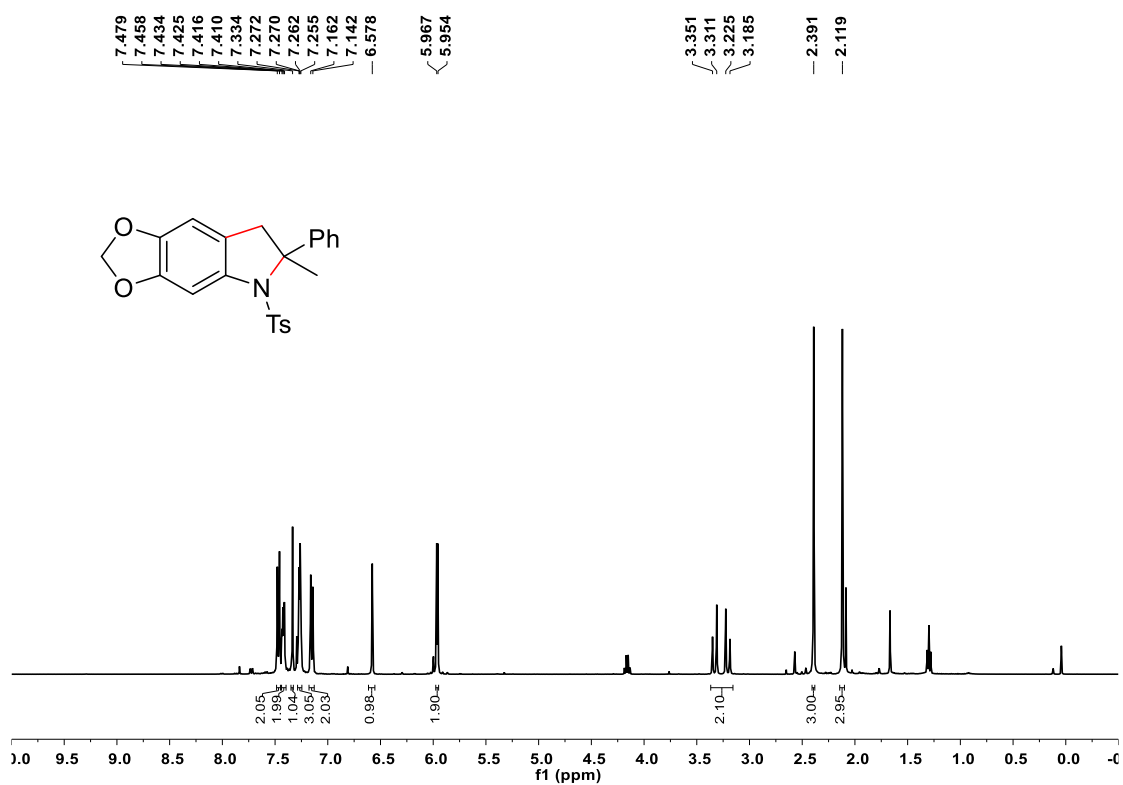


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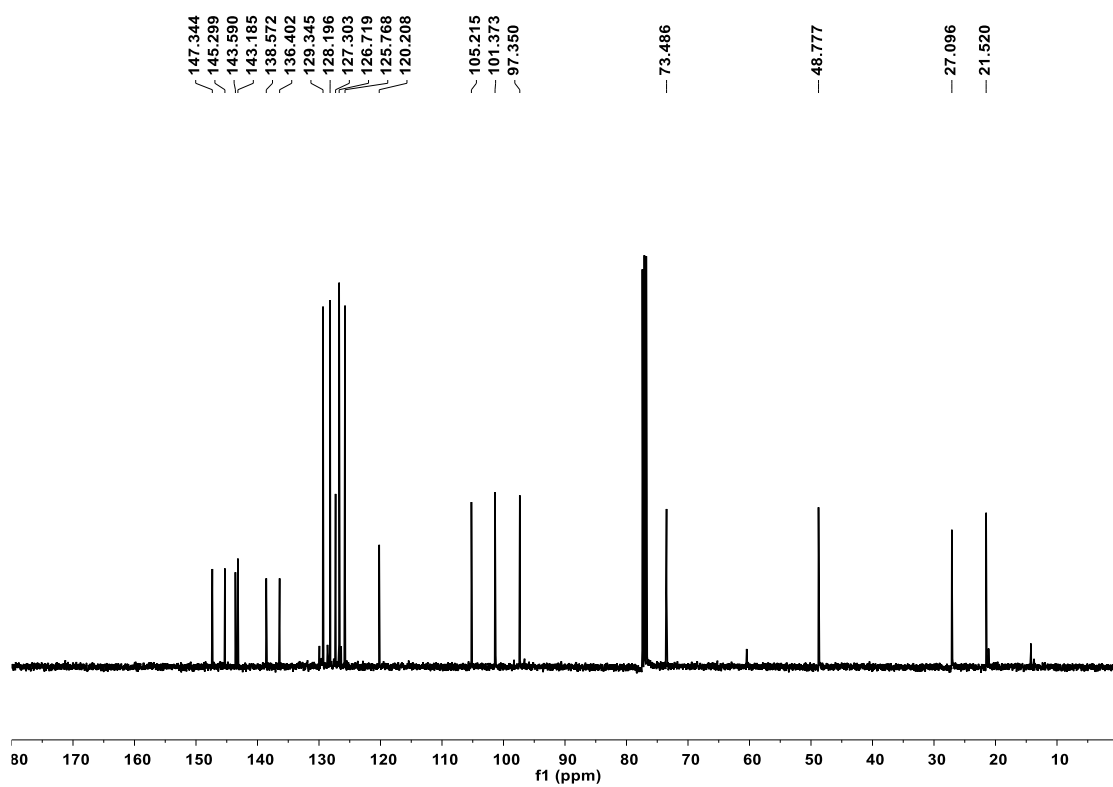


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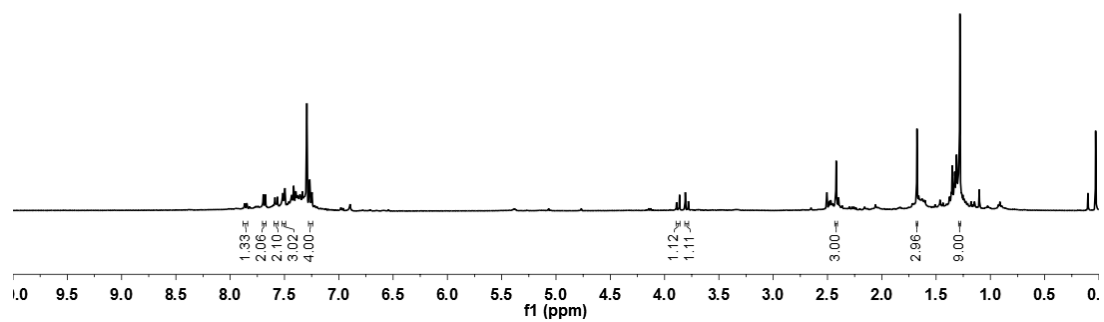
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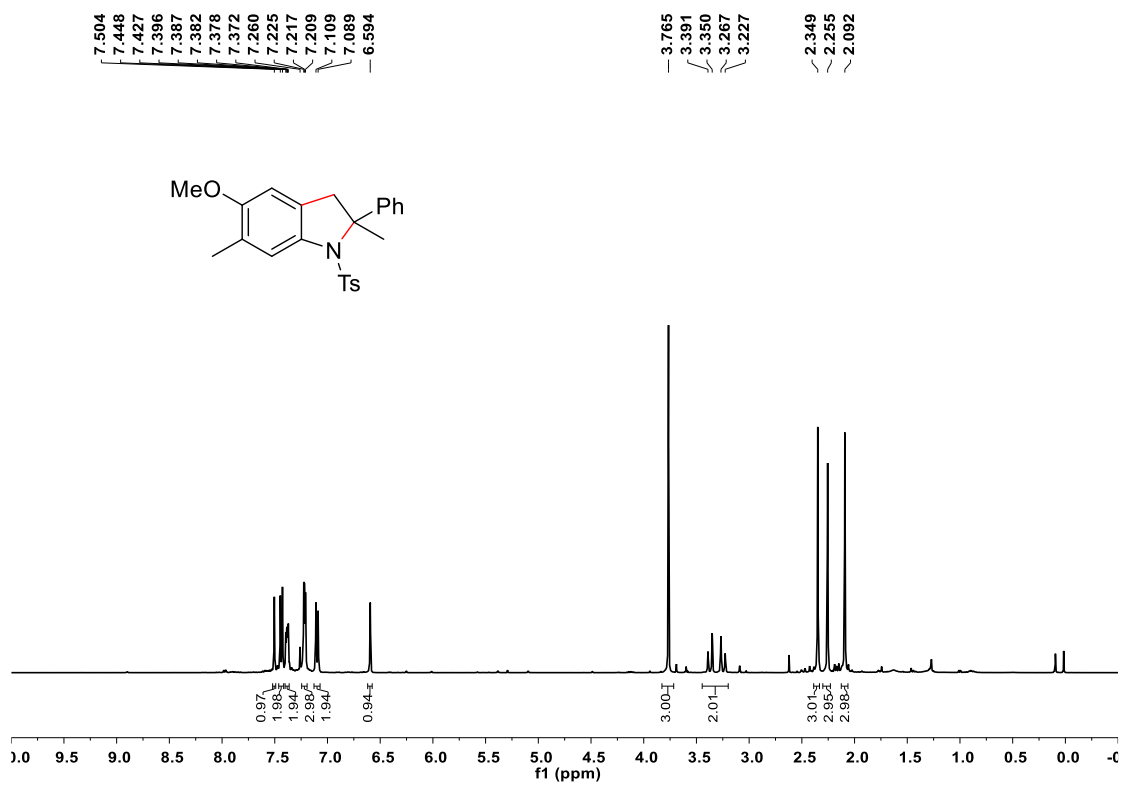


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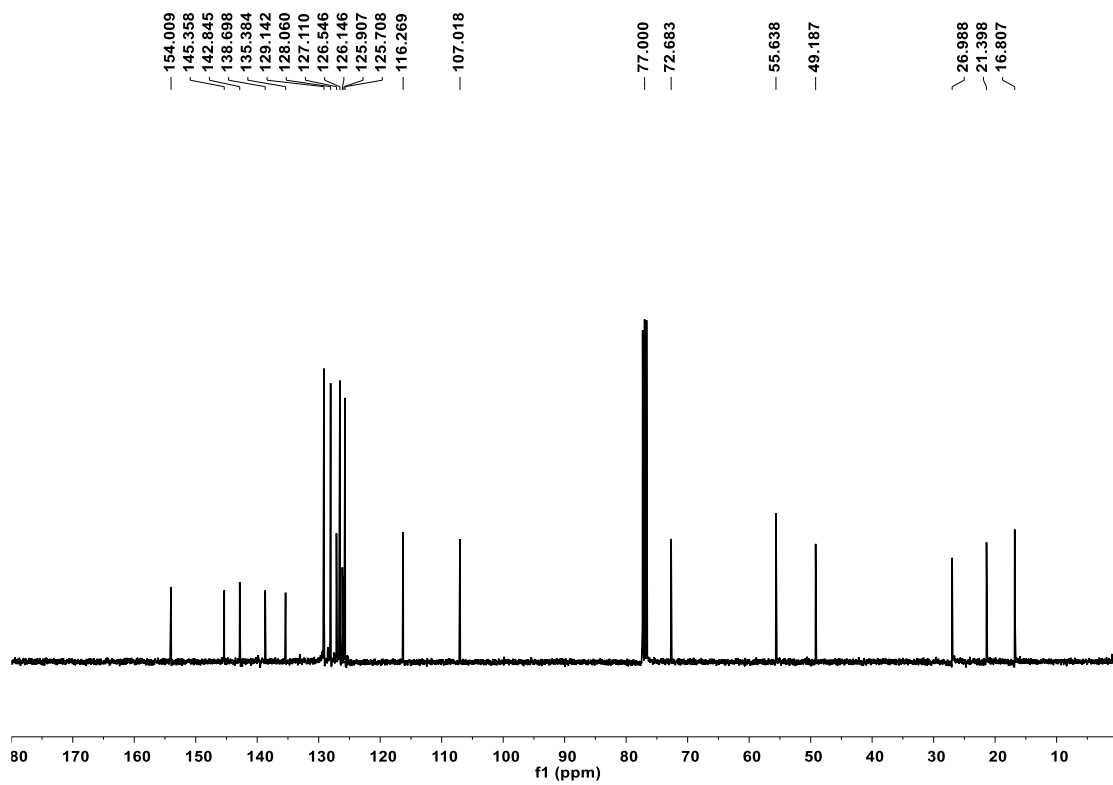


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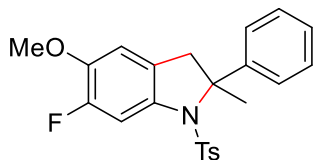
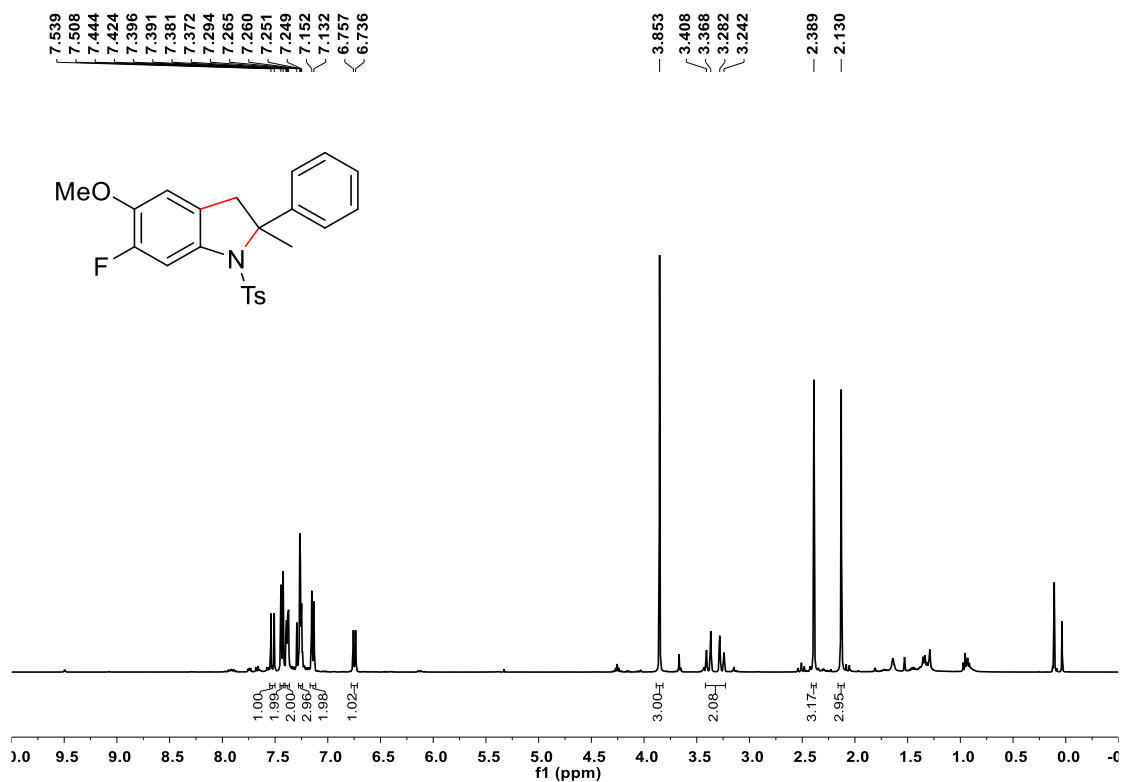


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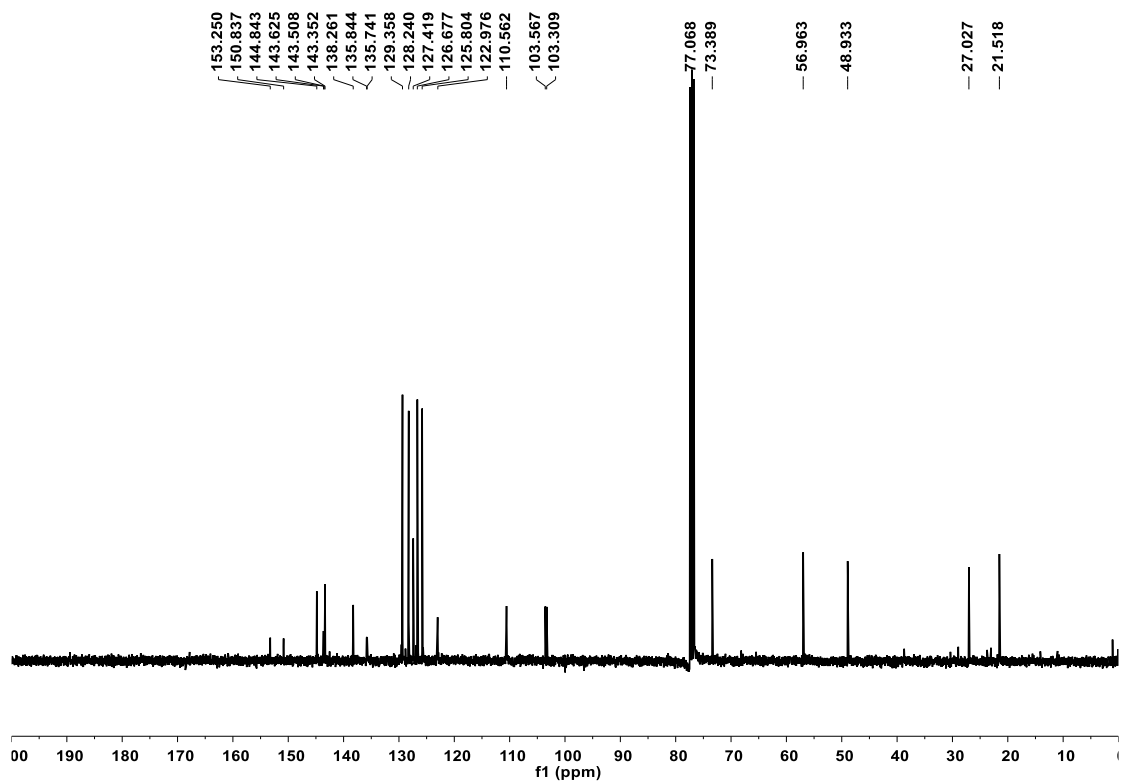


3fa

¹H NMR

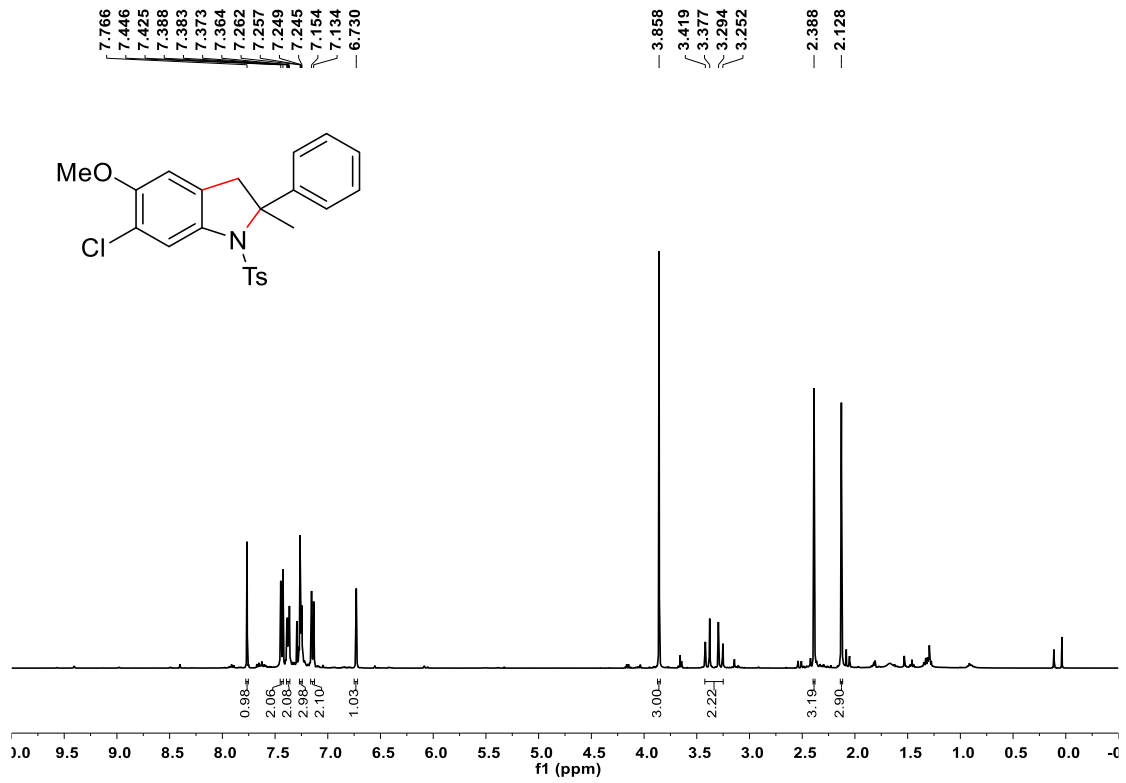


¹³C NMR

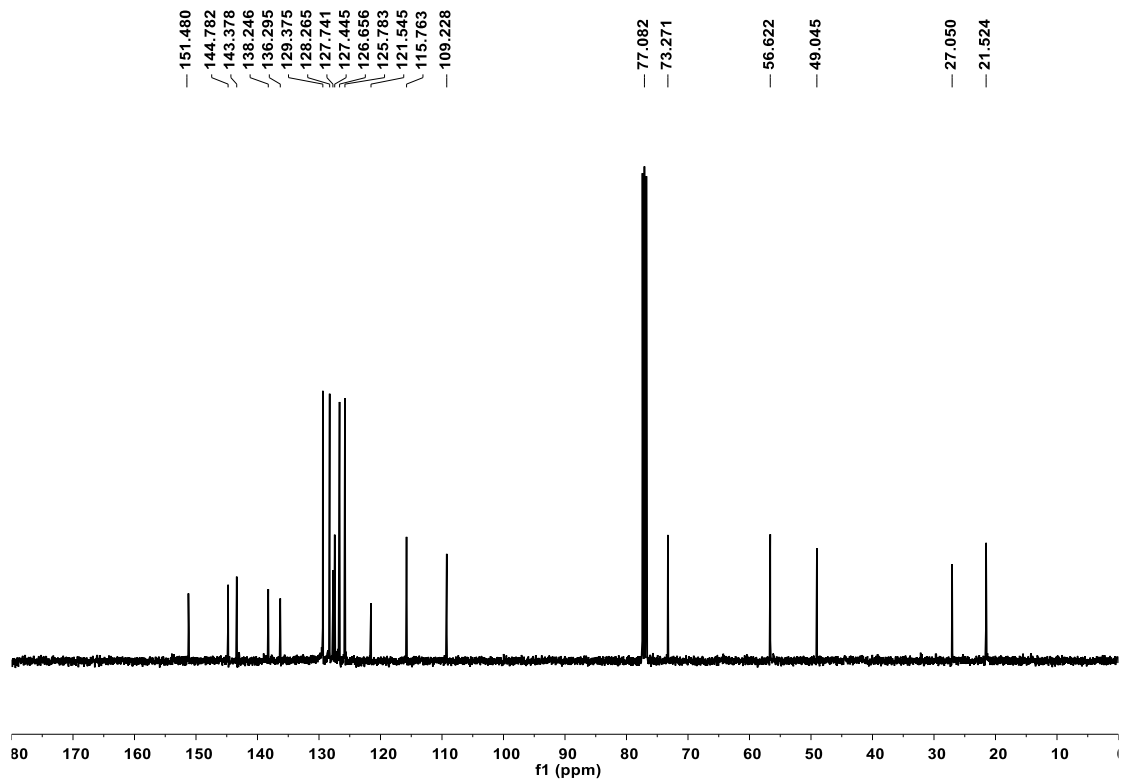


3ga

¹H NMR

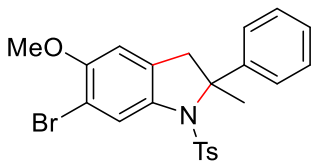
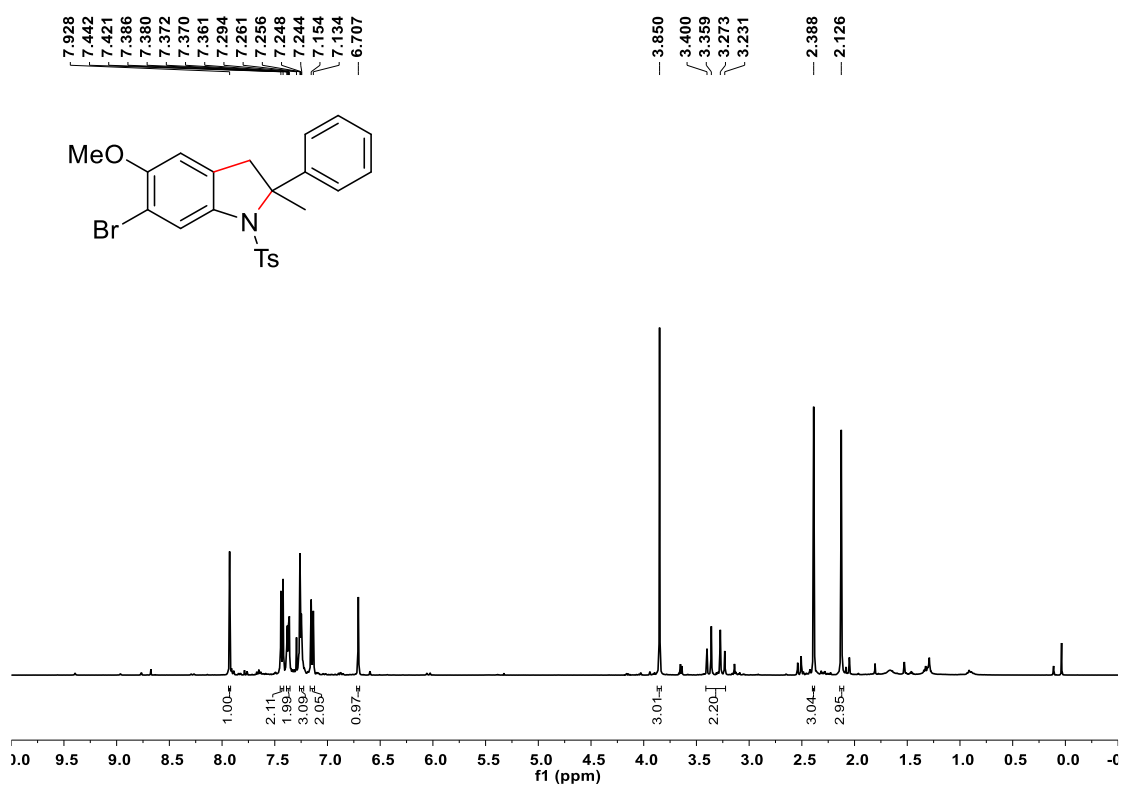


¹³C NMR

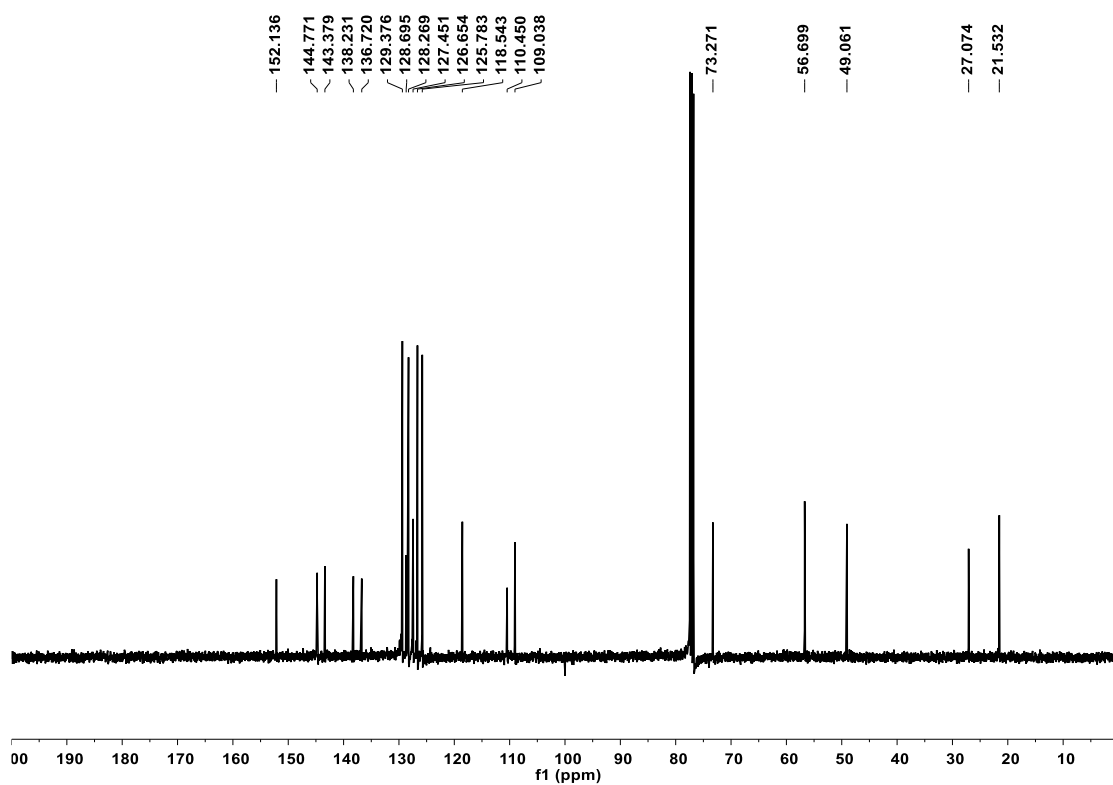


3ha

¹H NMR

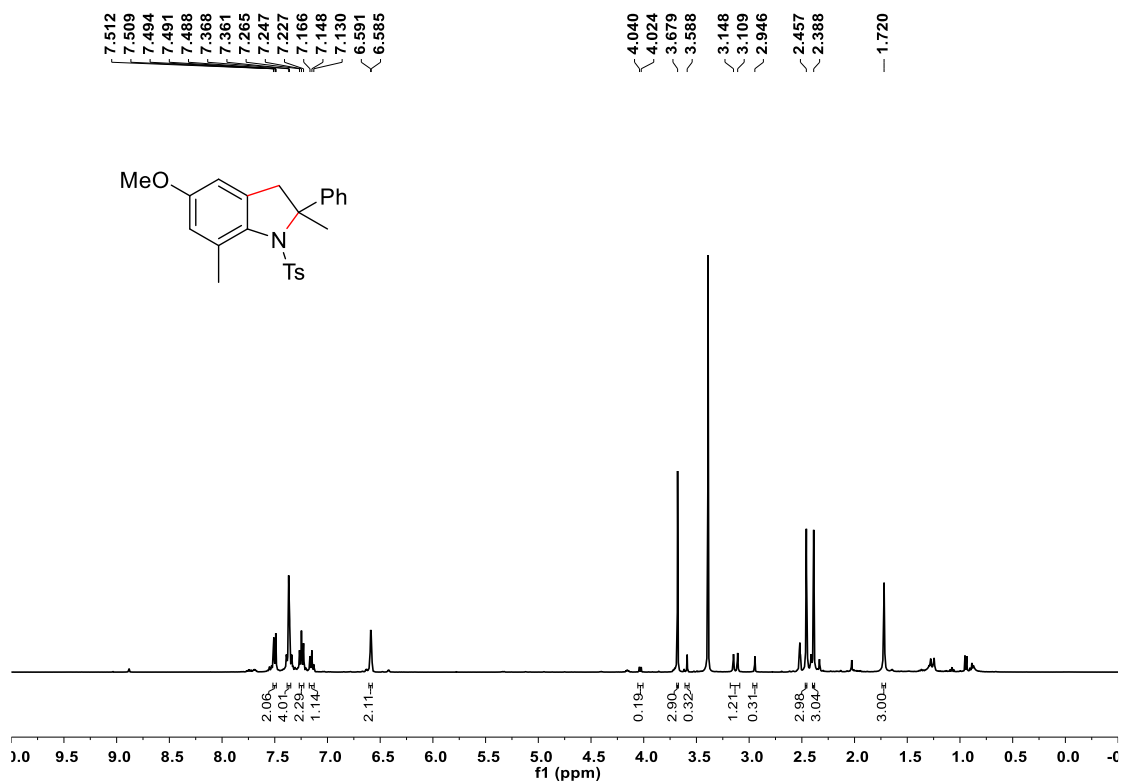


¹³C NMR

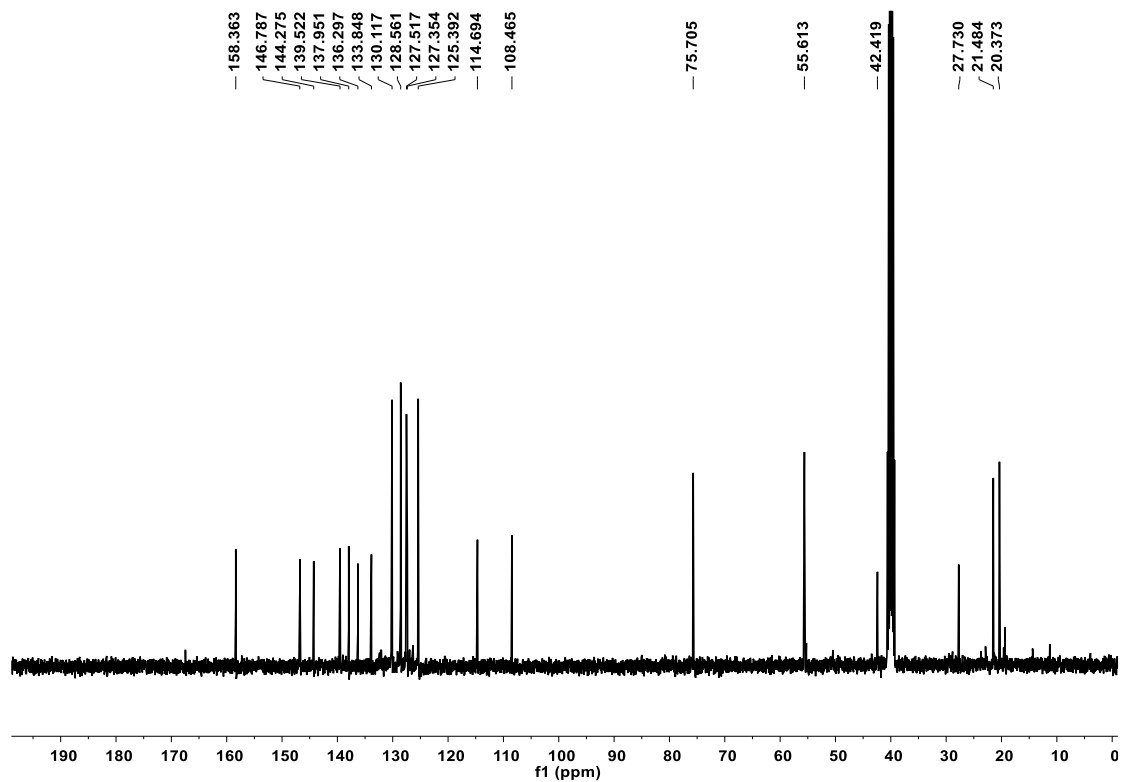


3ia

¹H NMR

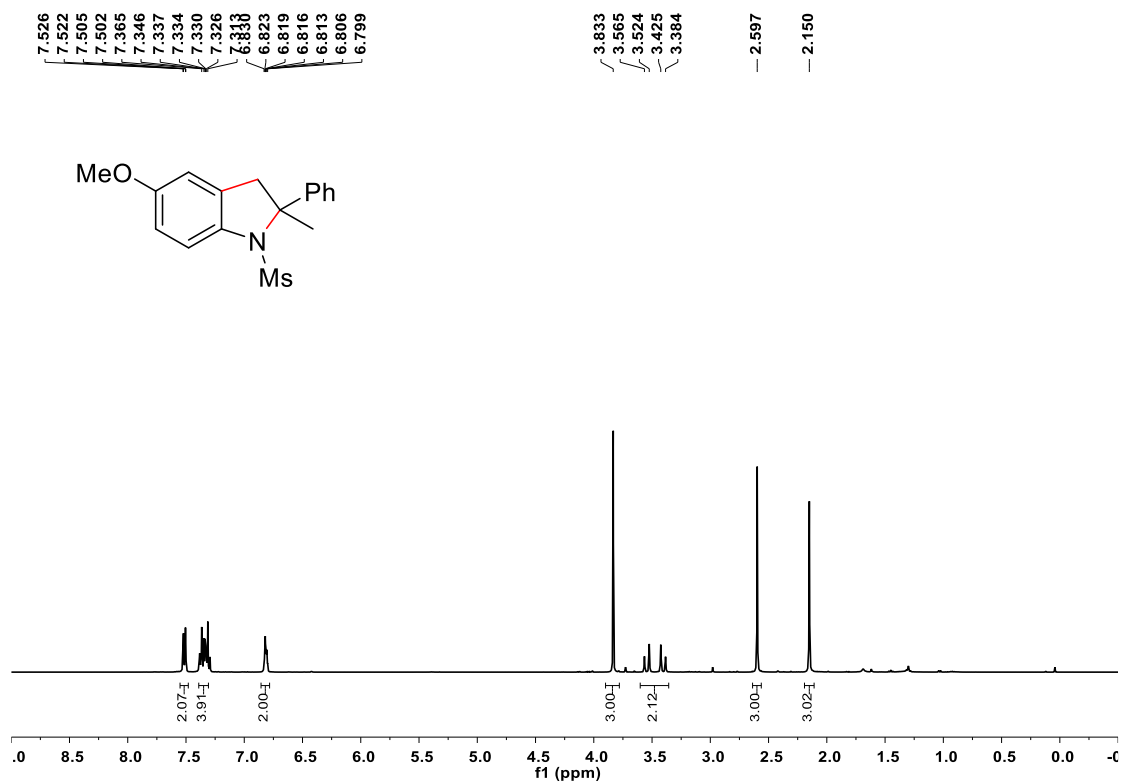


¹³C NMR

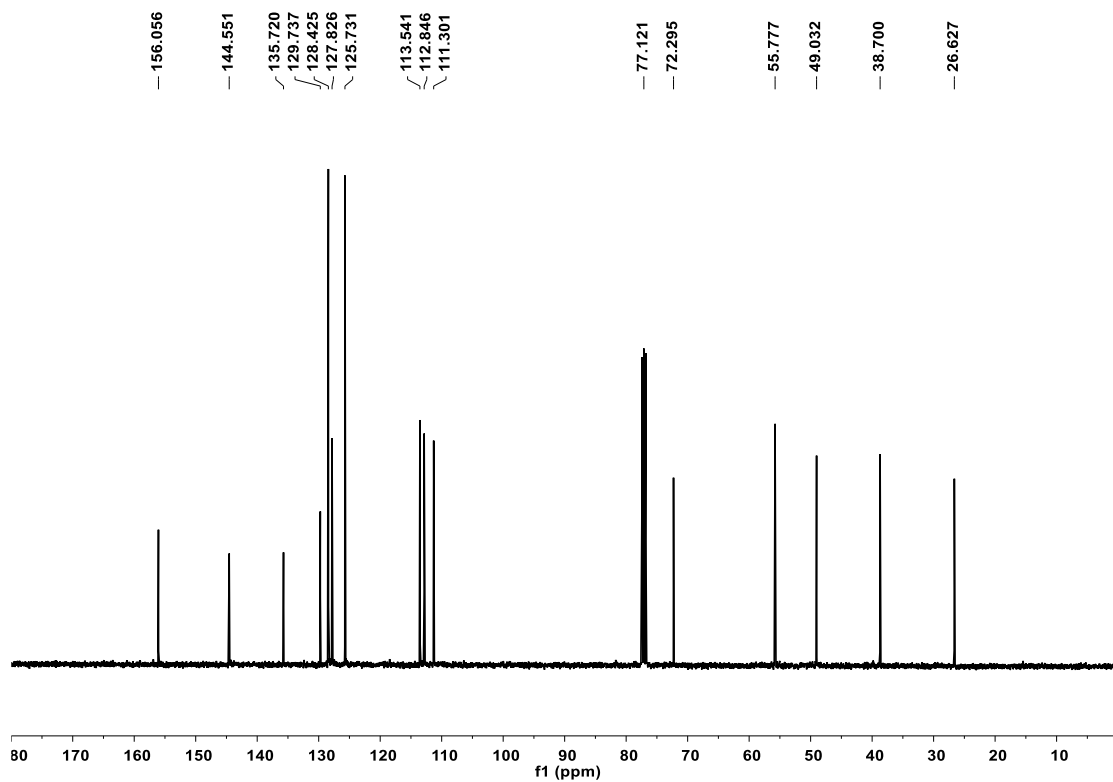


3ja

¹H NMR

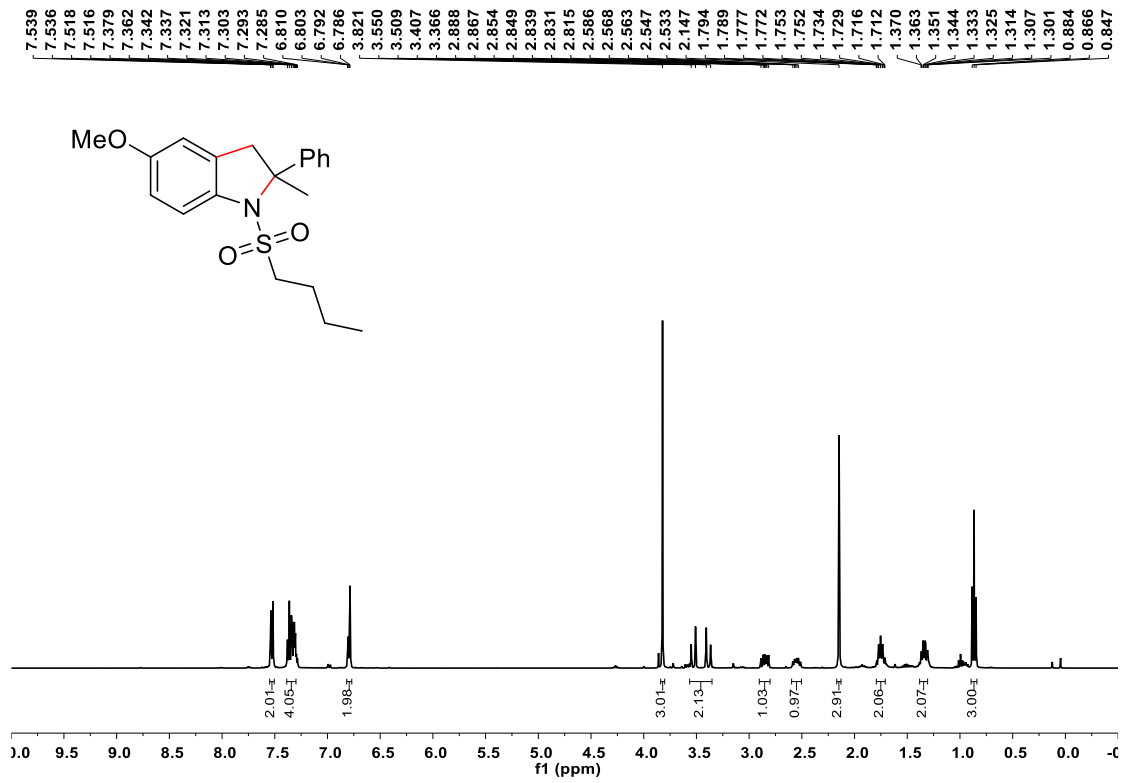


¹³C NMR

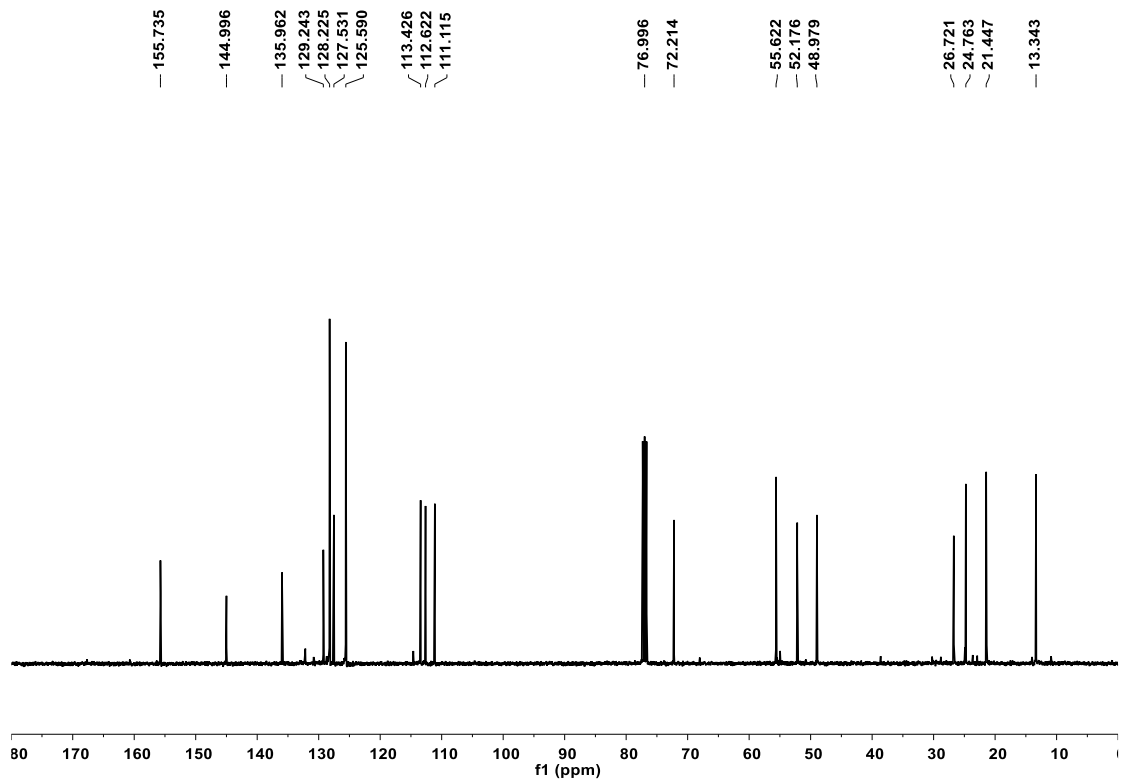


3ka

¹H NMR

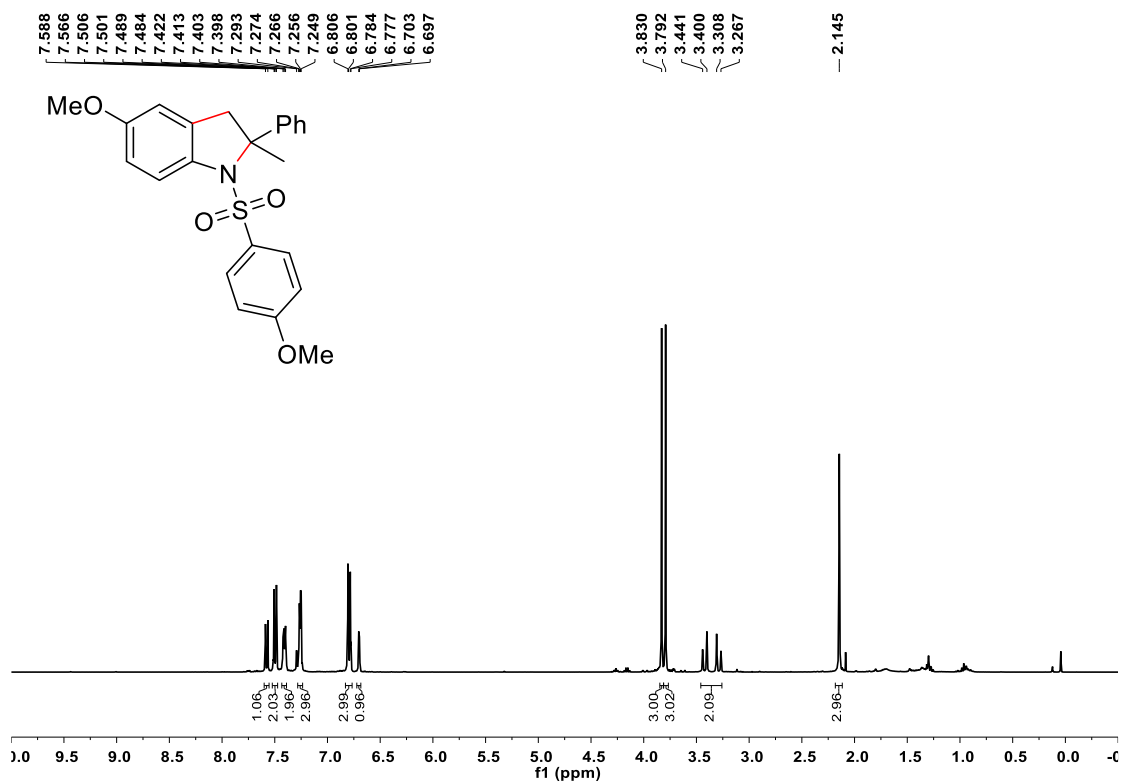


¹³C NMR

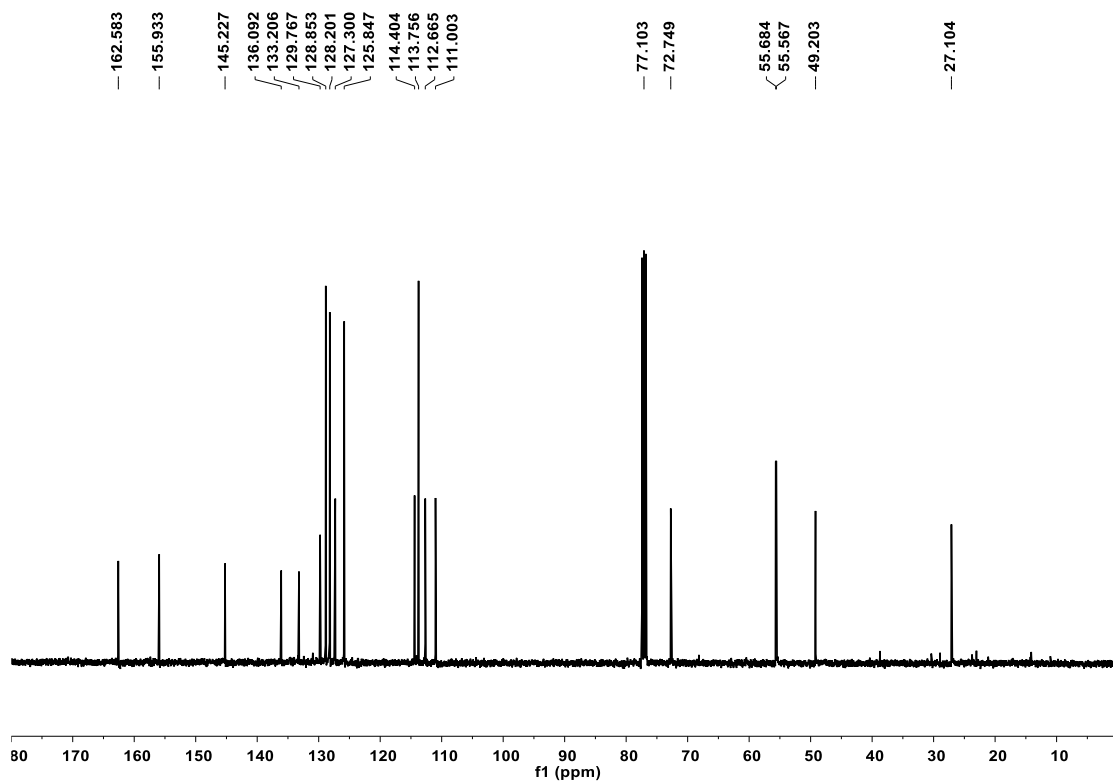


3la

¹H NMR

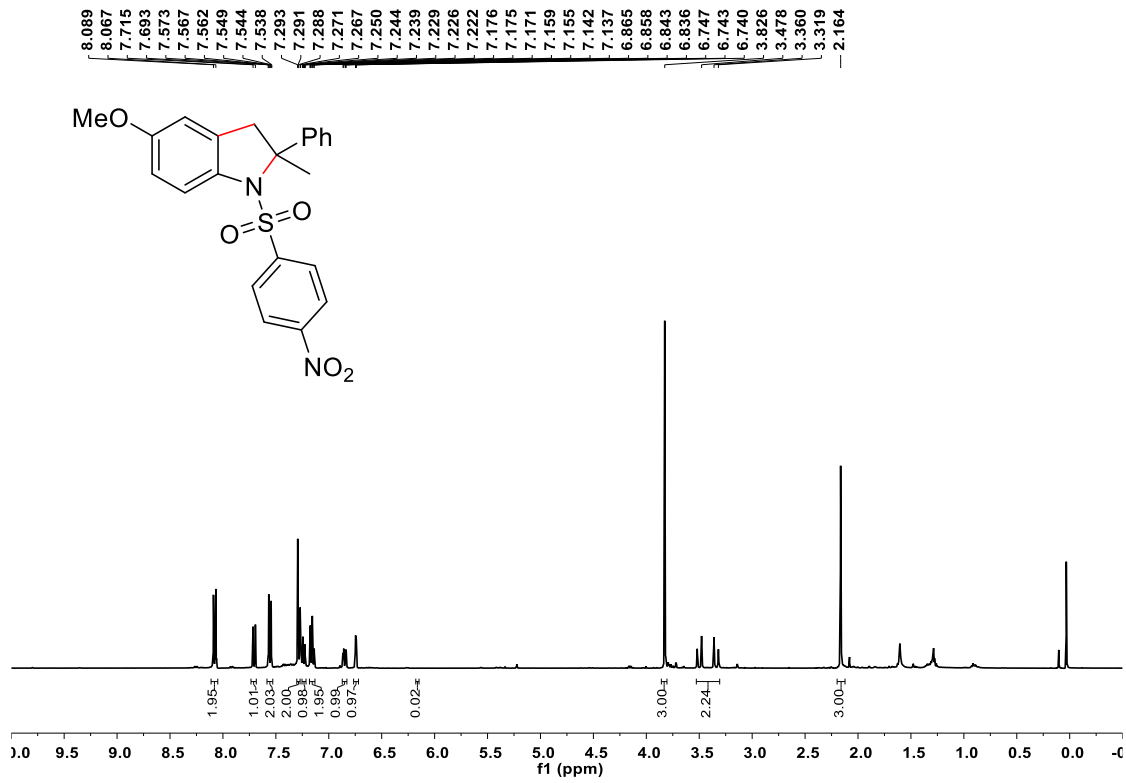


¹³C NMR

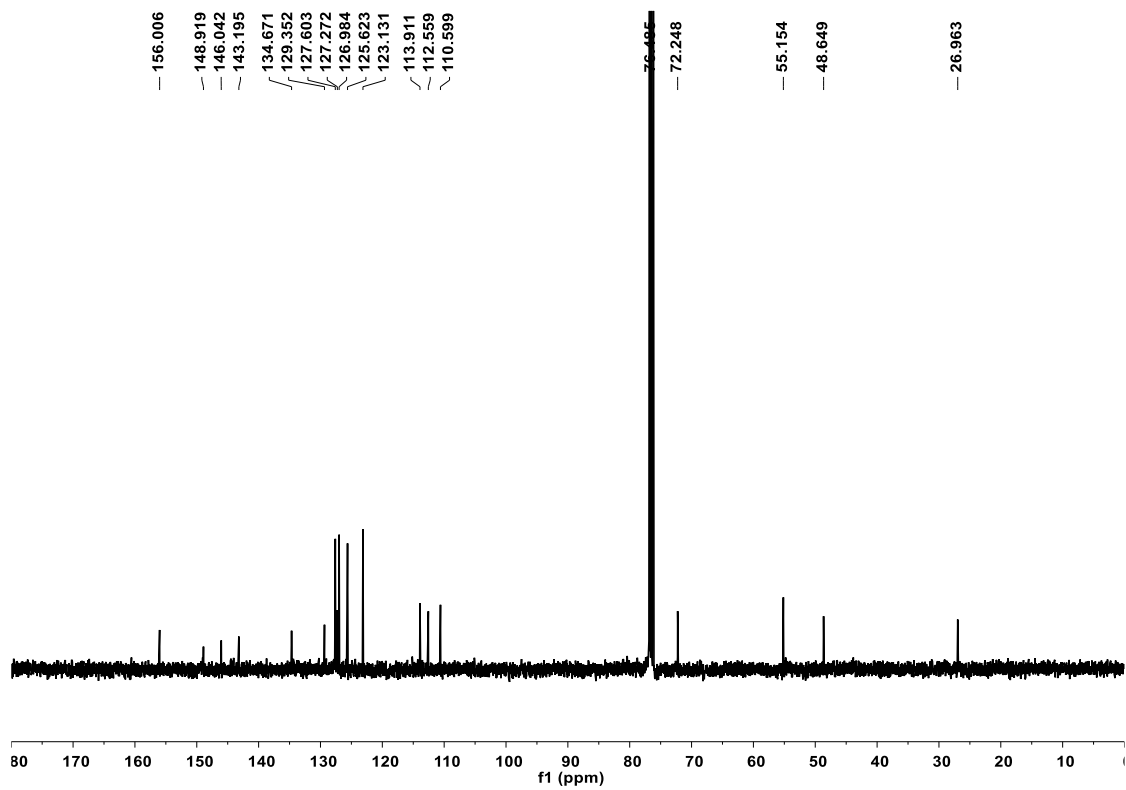


3ma

¹H NMR

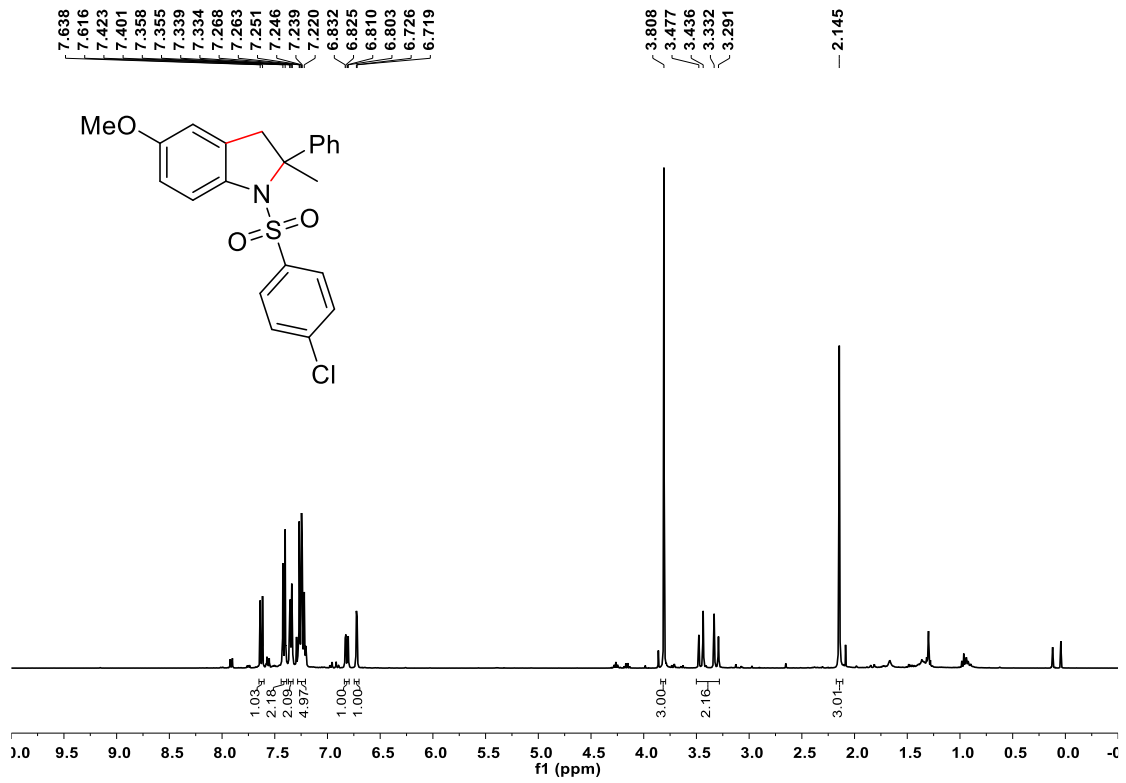


¹³C NMR

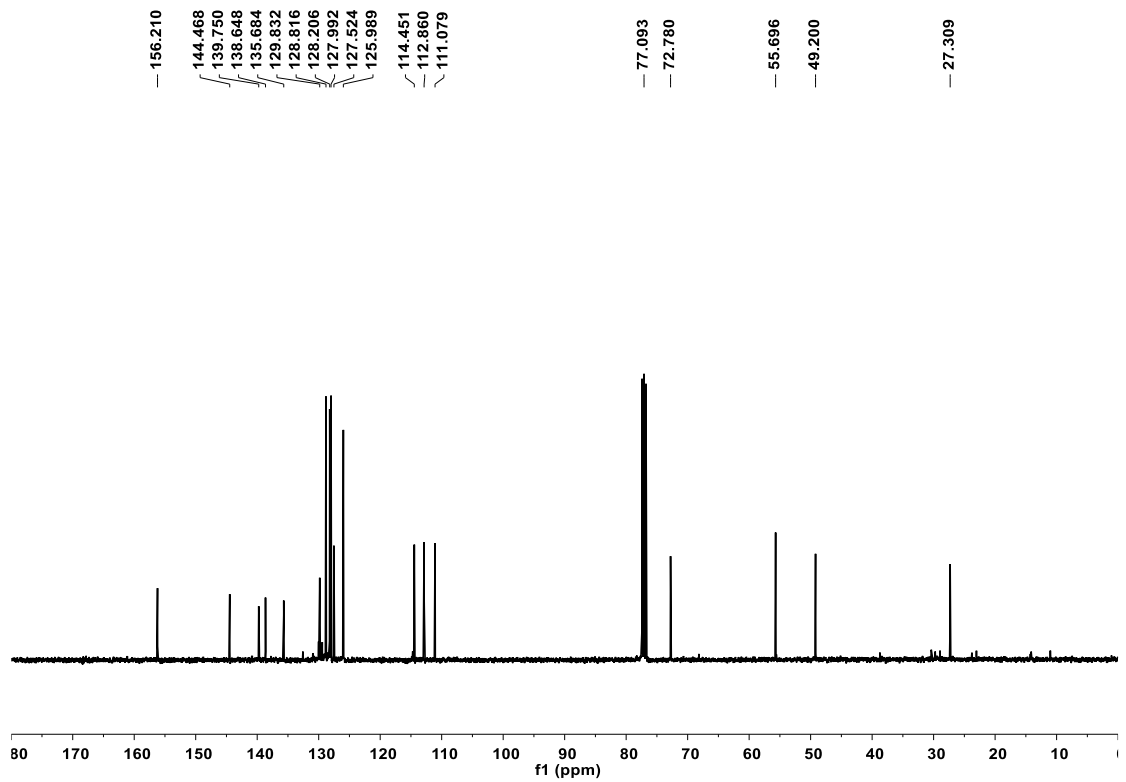


3na

¹H NMR

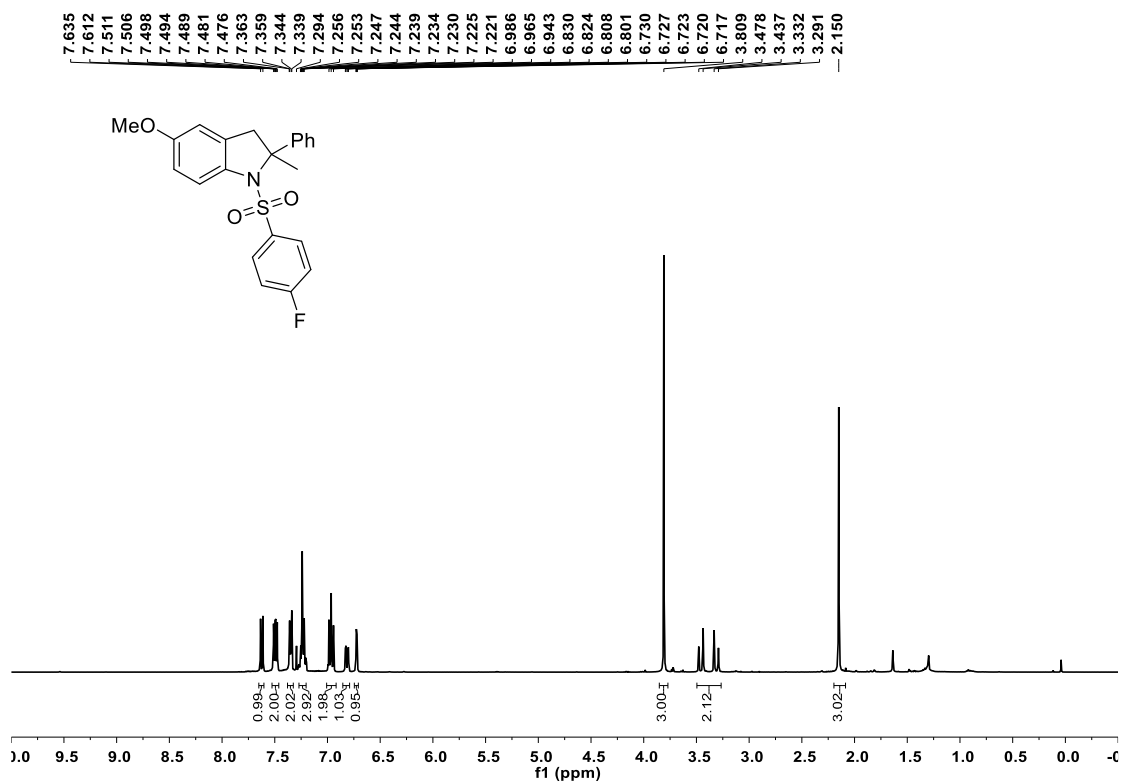


¹³C NMR

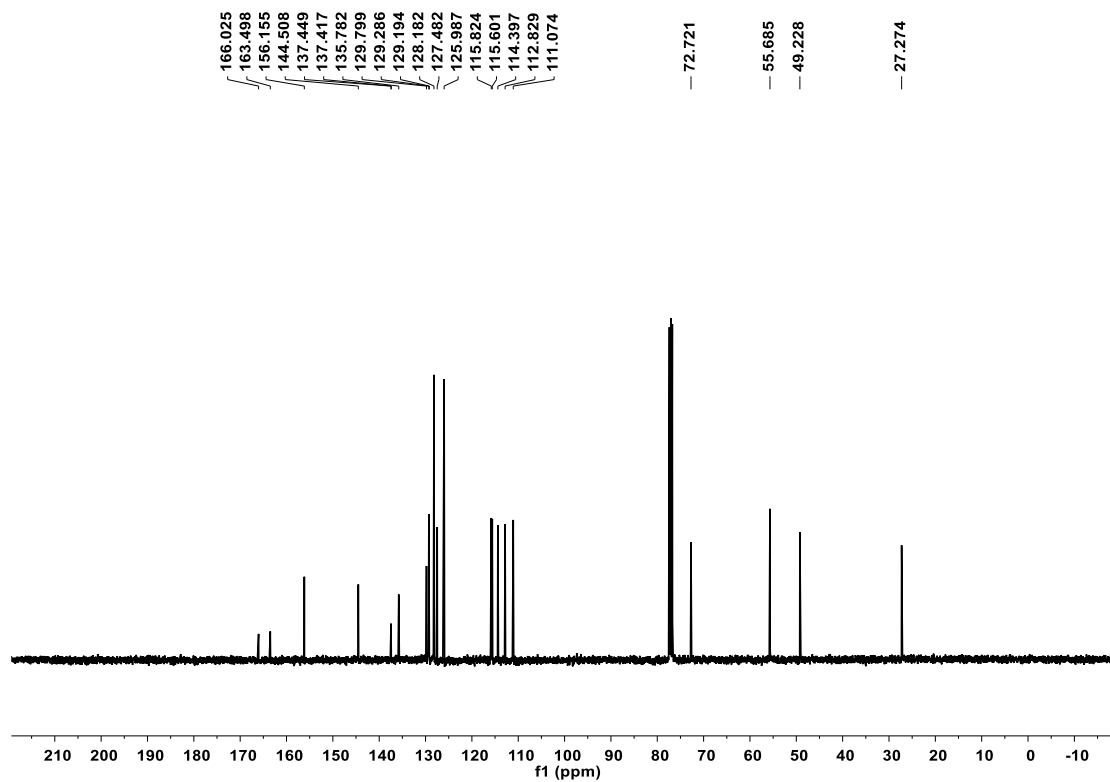


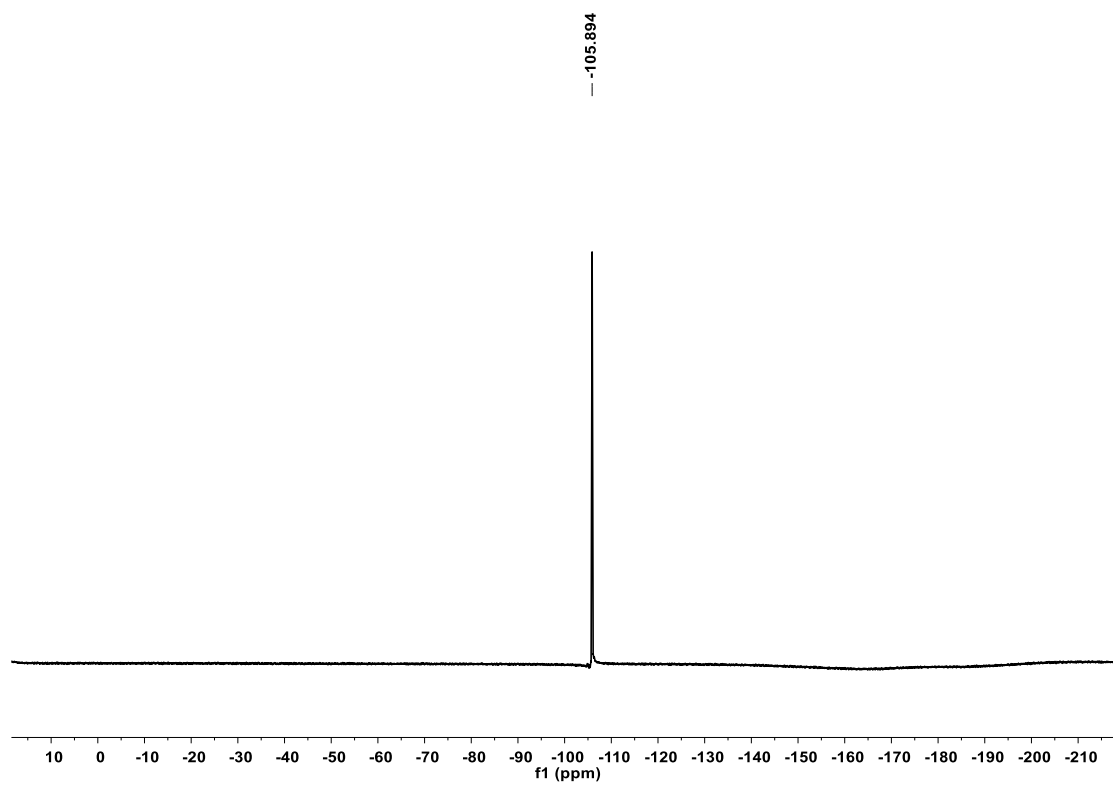
30a

¹H NMR



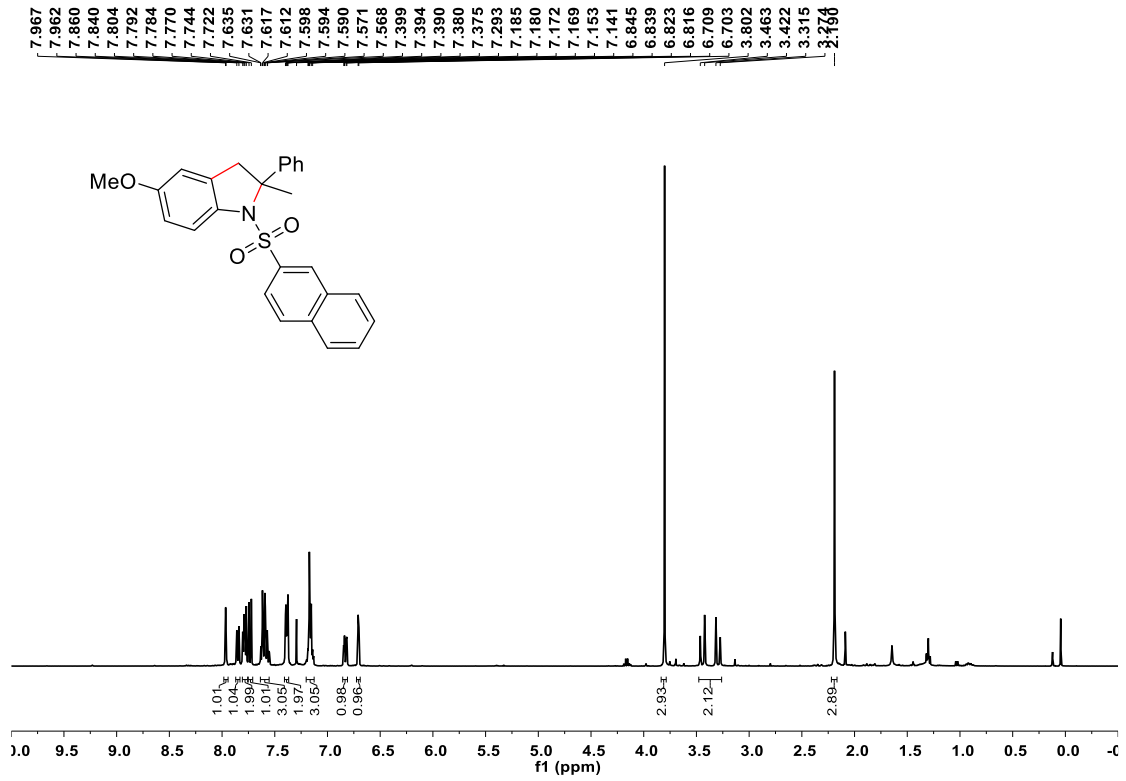
¹³C NMR



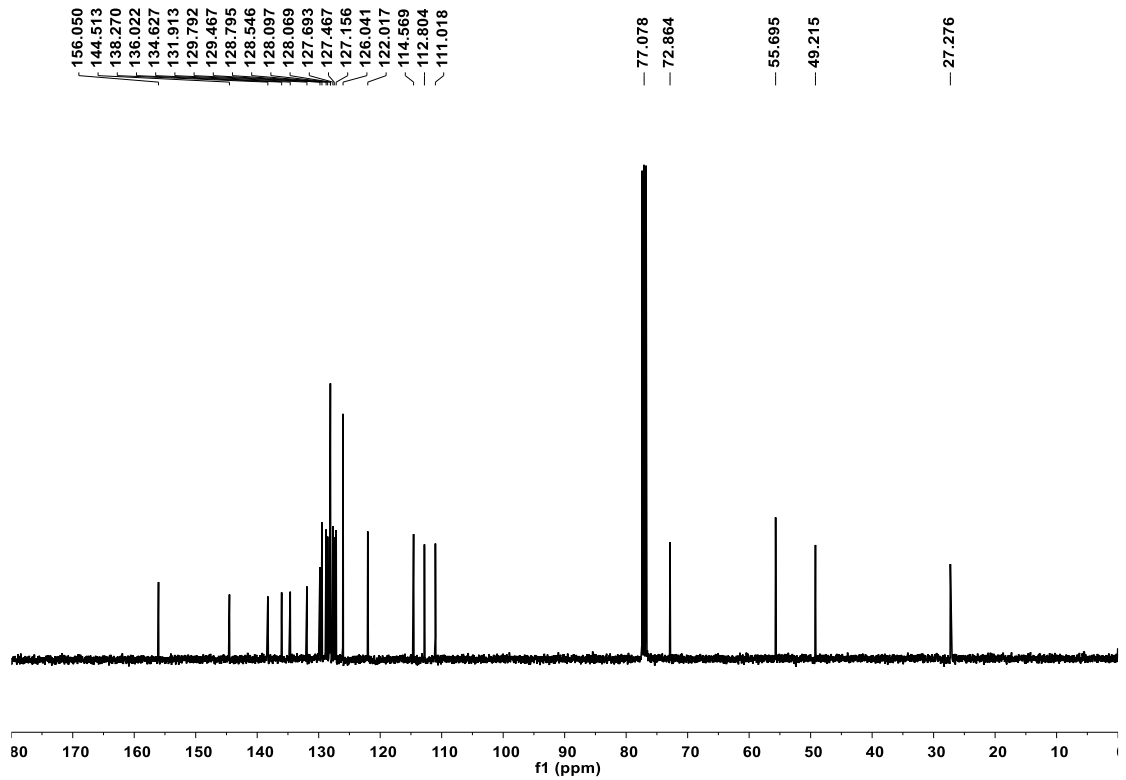


3pa

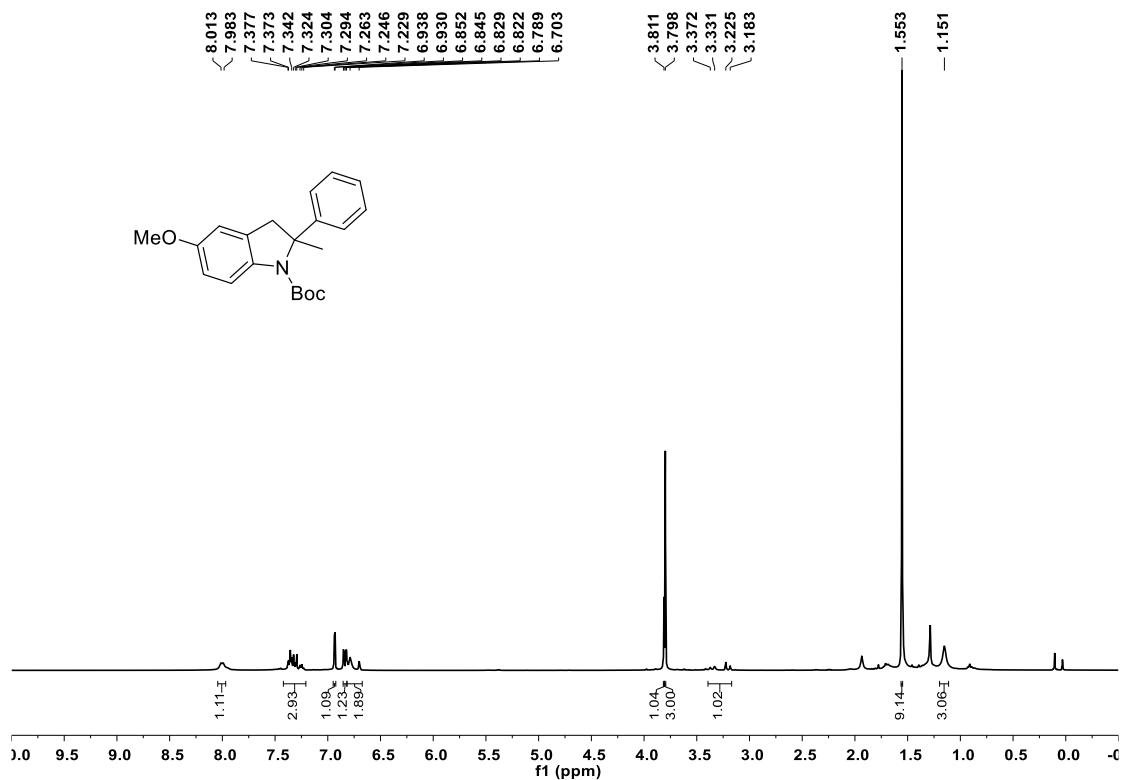
¹H NMR



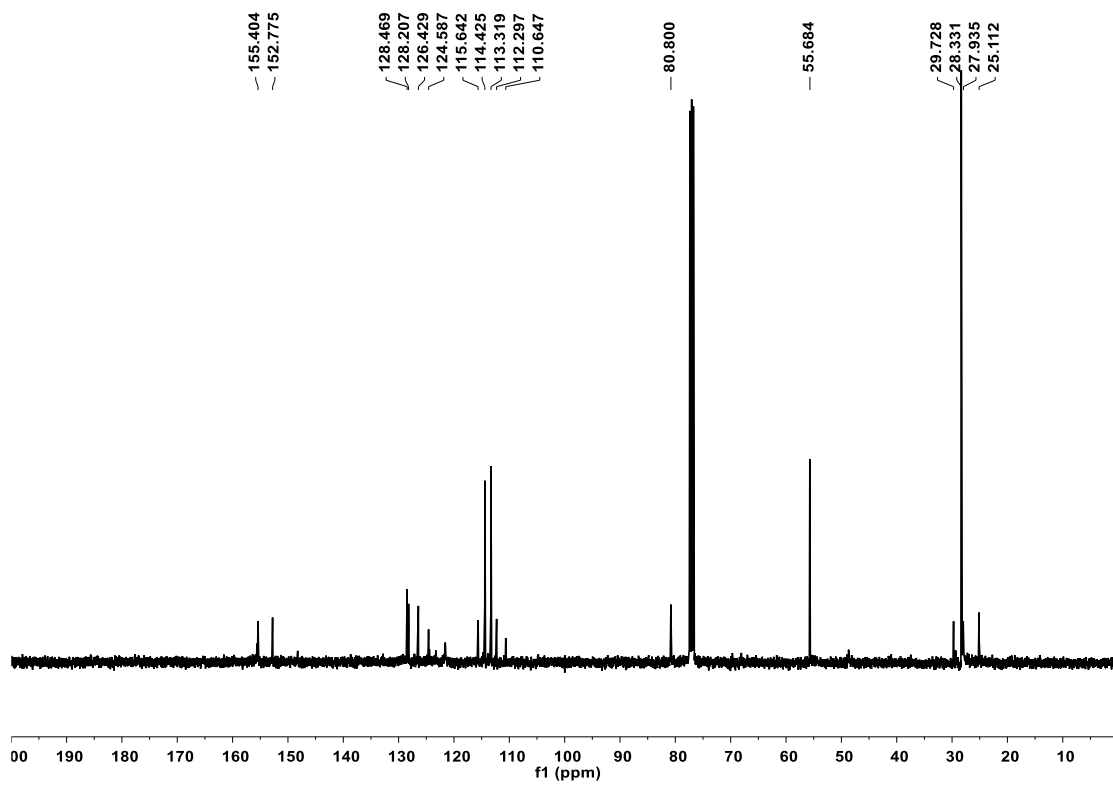
¹³C NMR



3qa

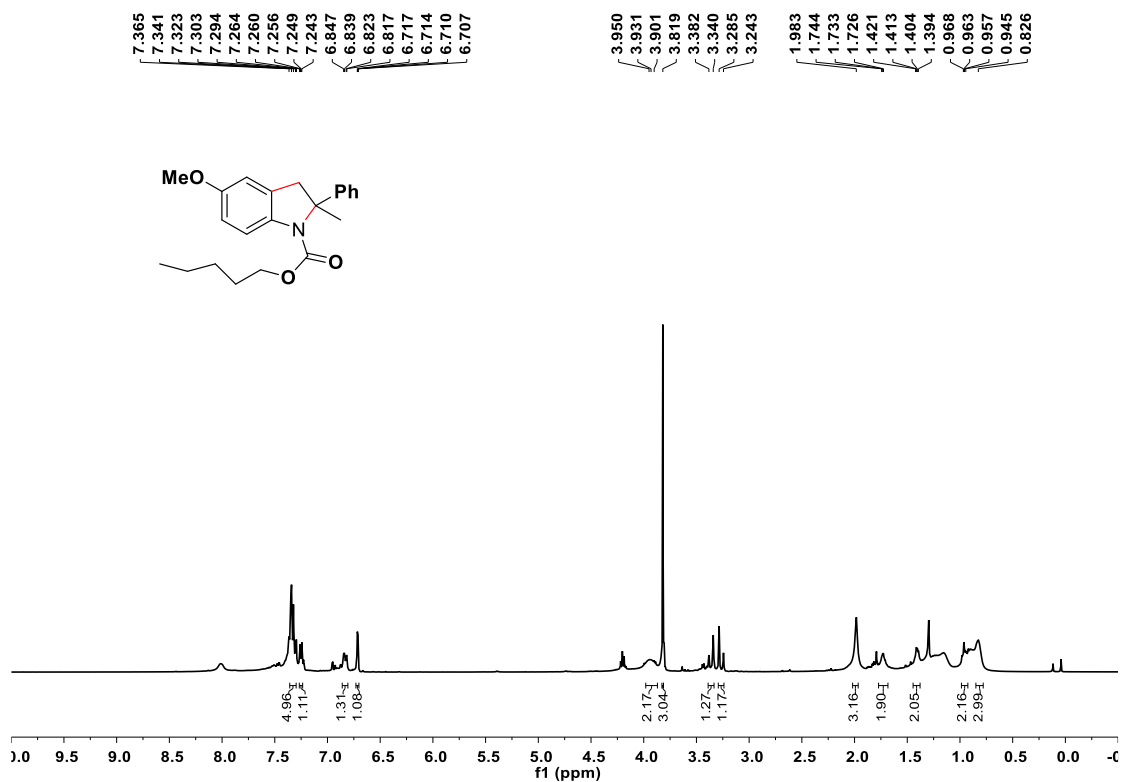


¹³CNMR

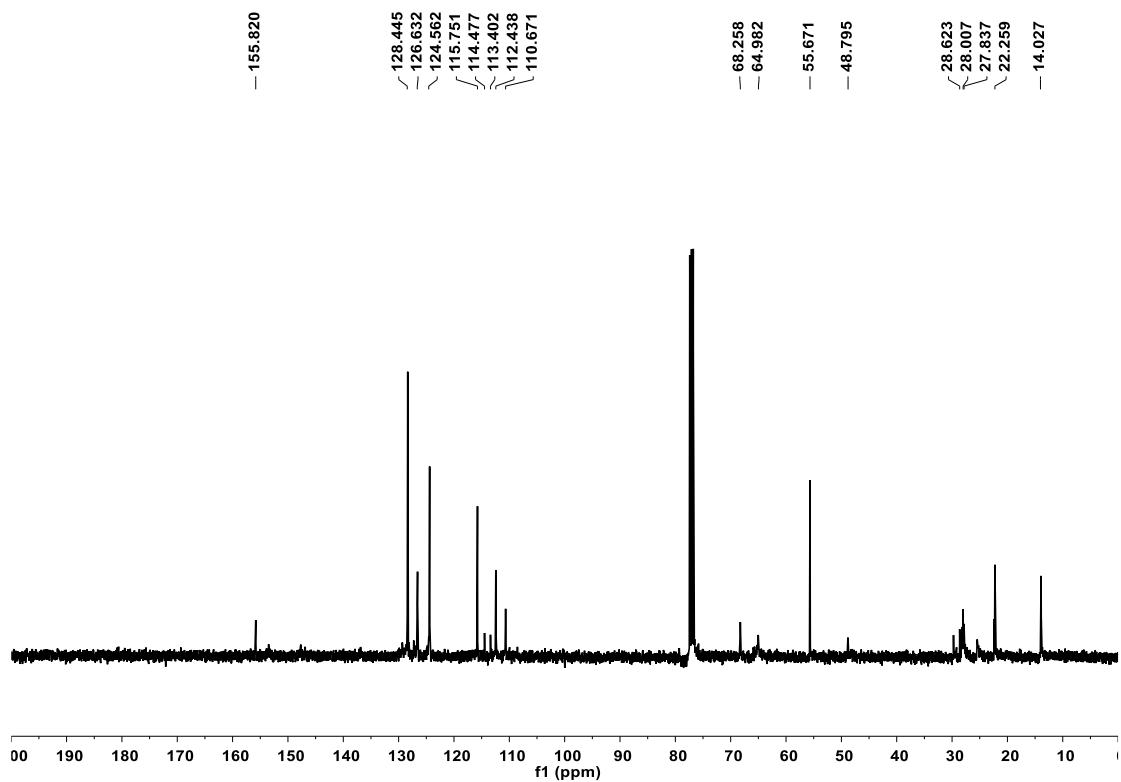


3ra

¹H NMR

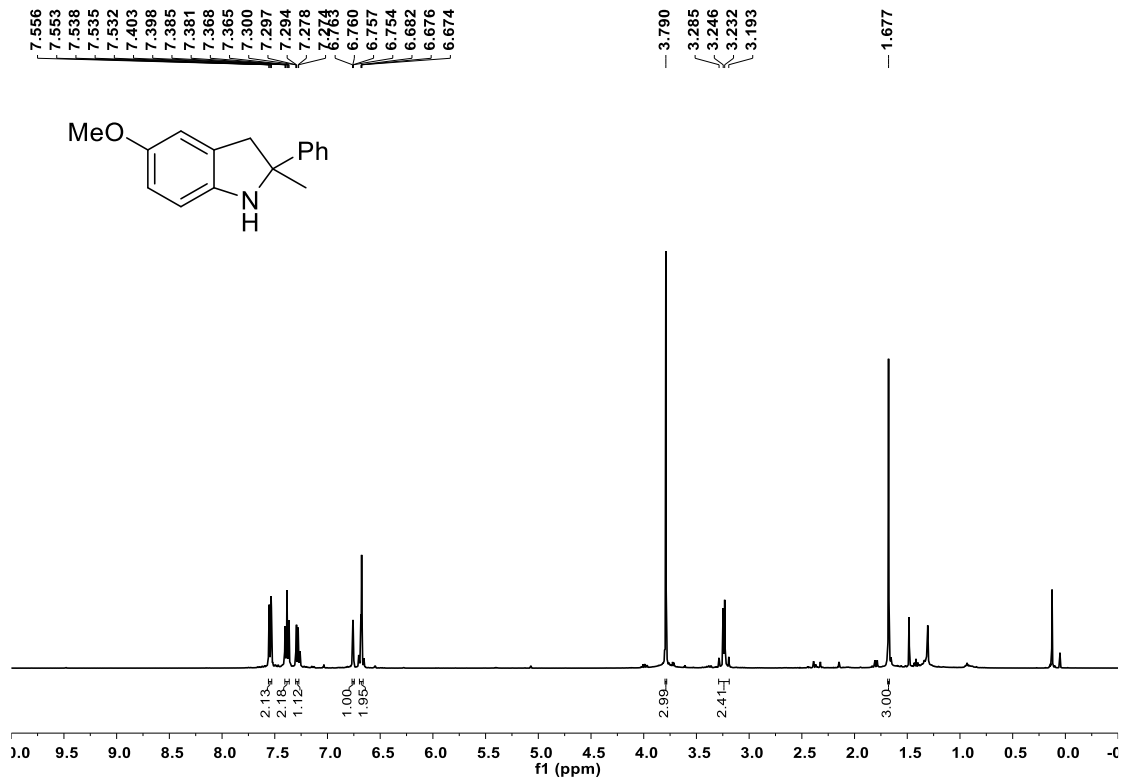


¹³C NMR

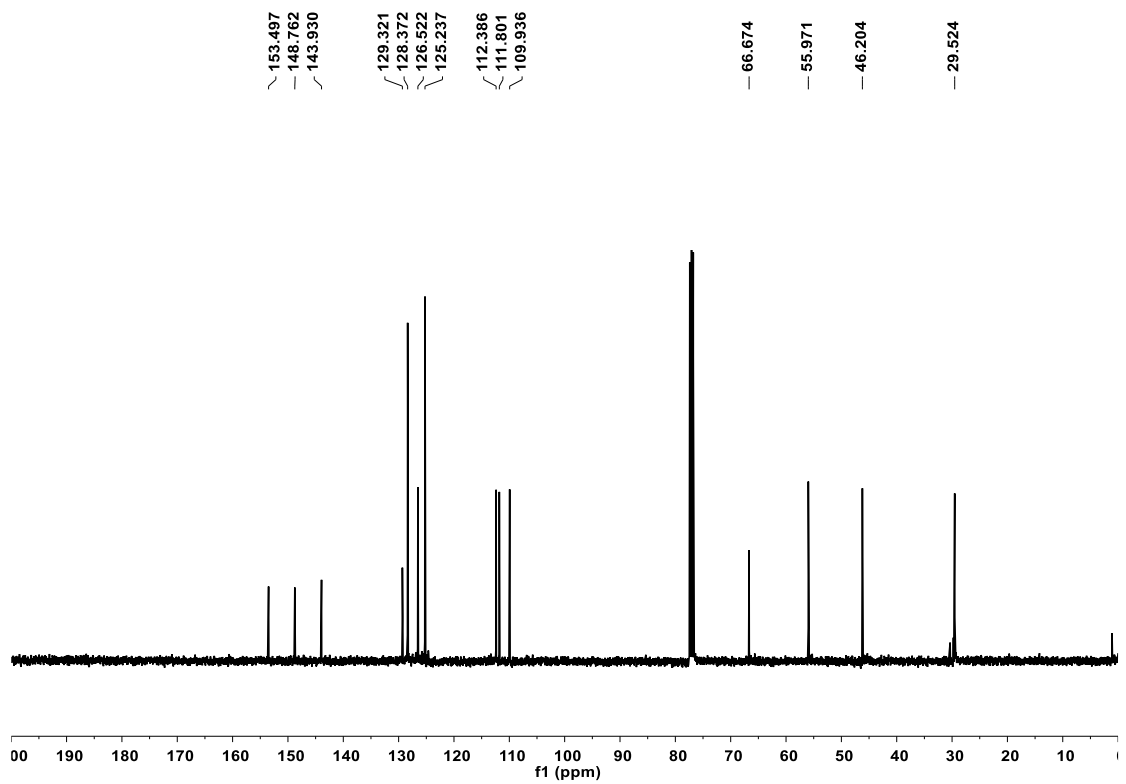


4a

¹H NMR

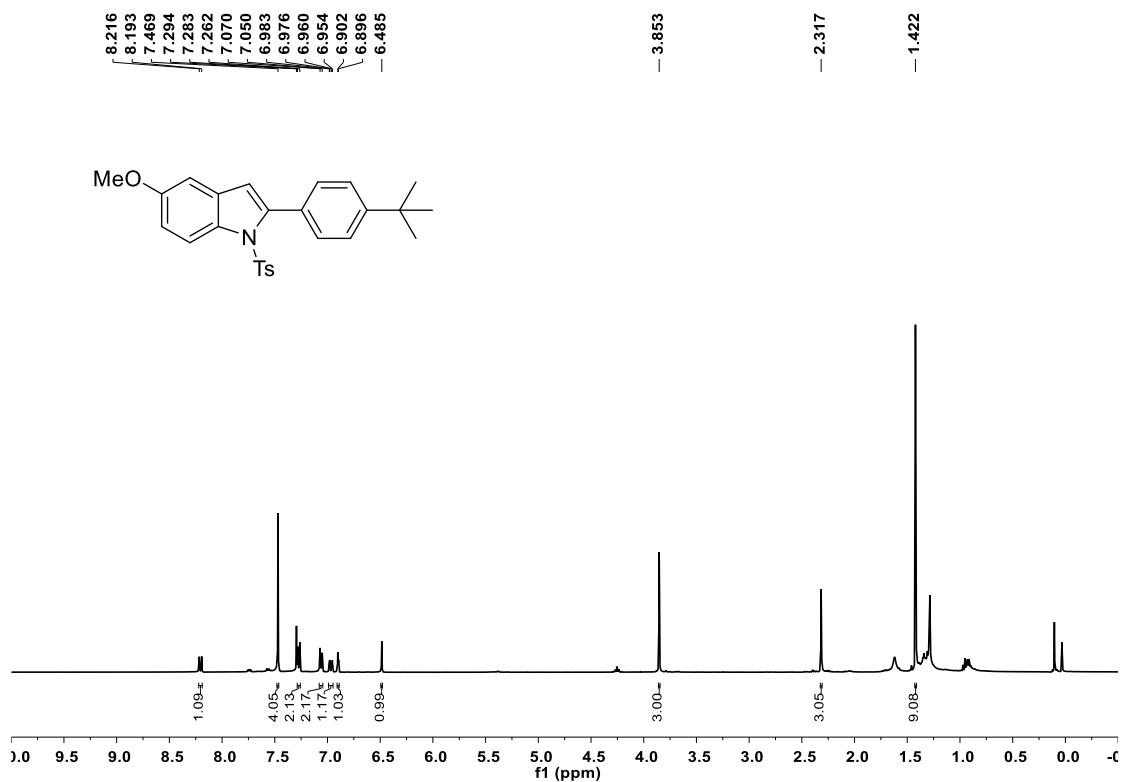


¹³C NMR

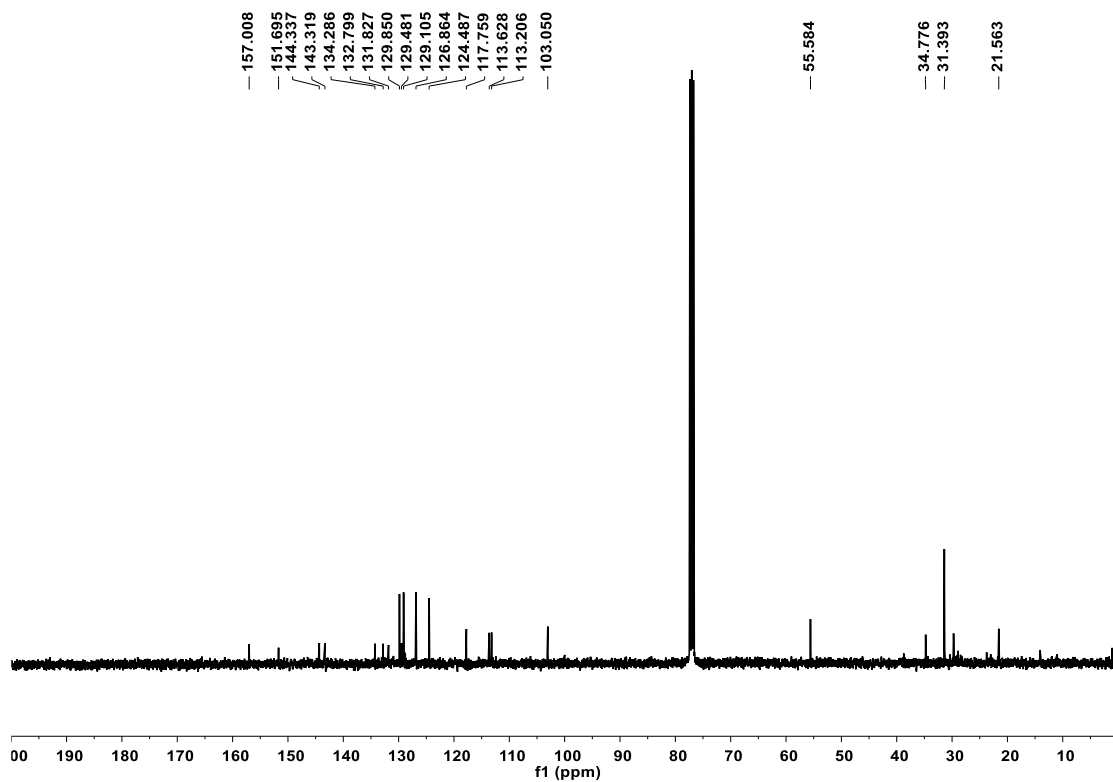


4b

¹H NMR

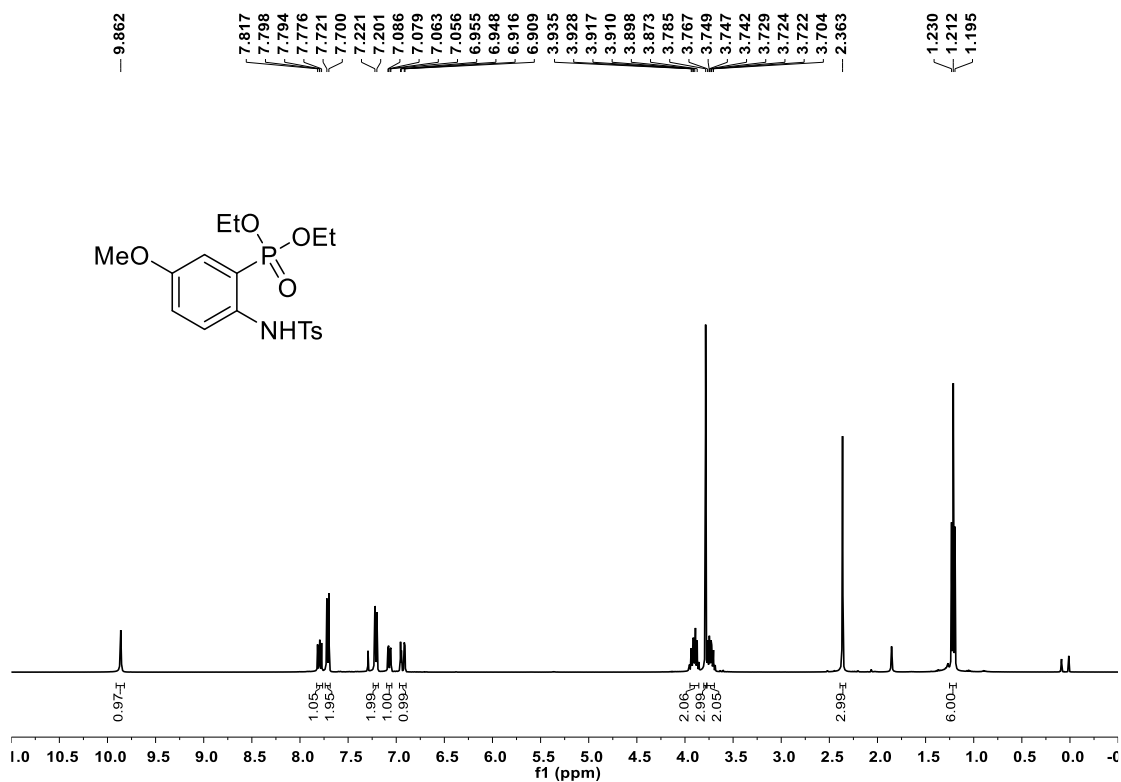


¹³C NMR

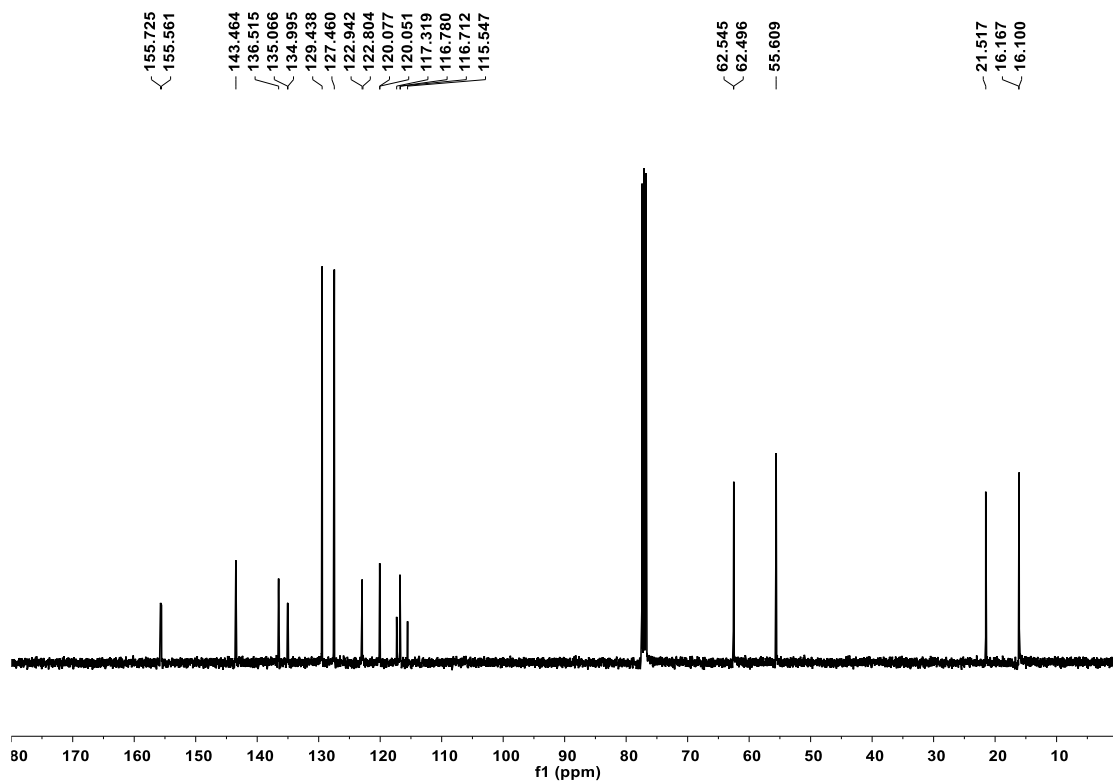


4c

¹H NMR

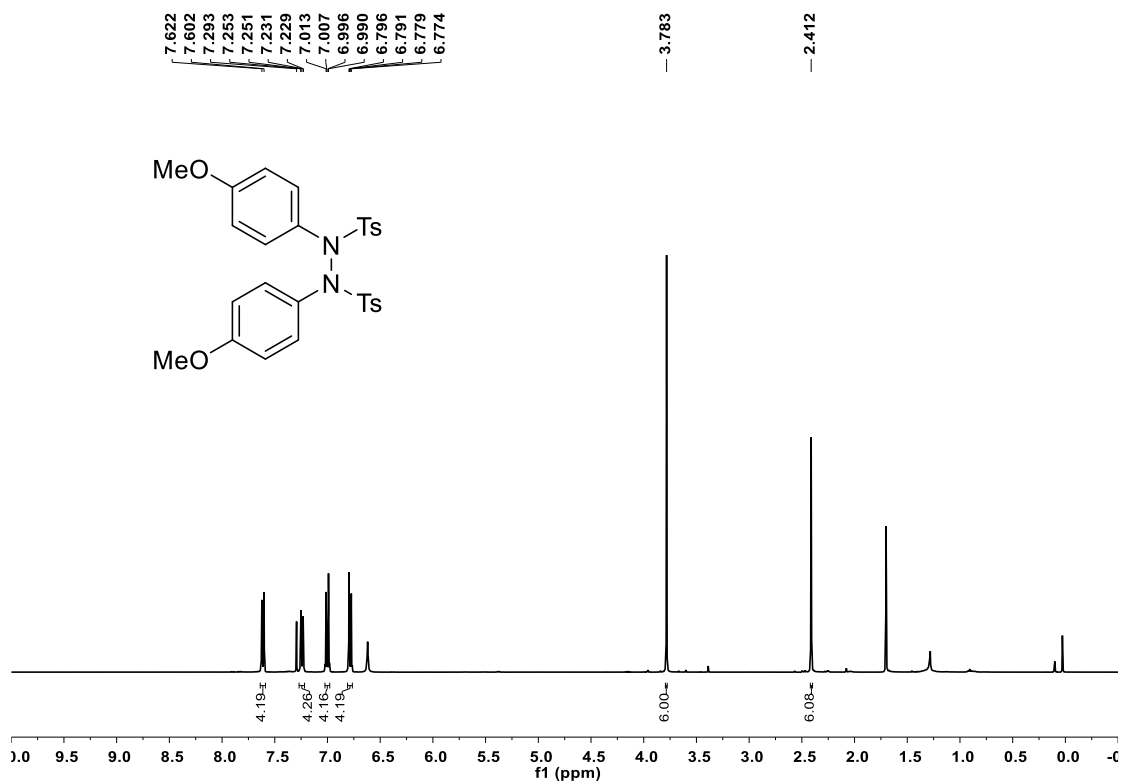


¹³C NMR

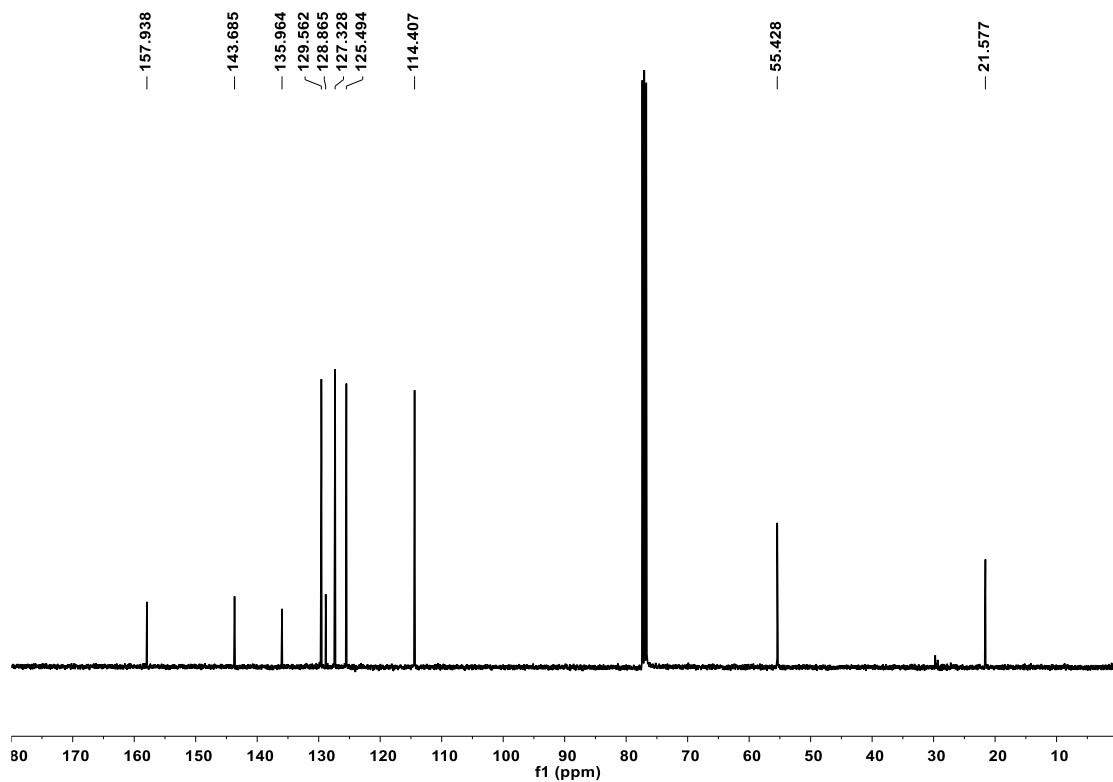


4d

¹H NMR

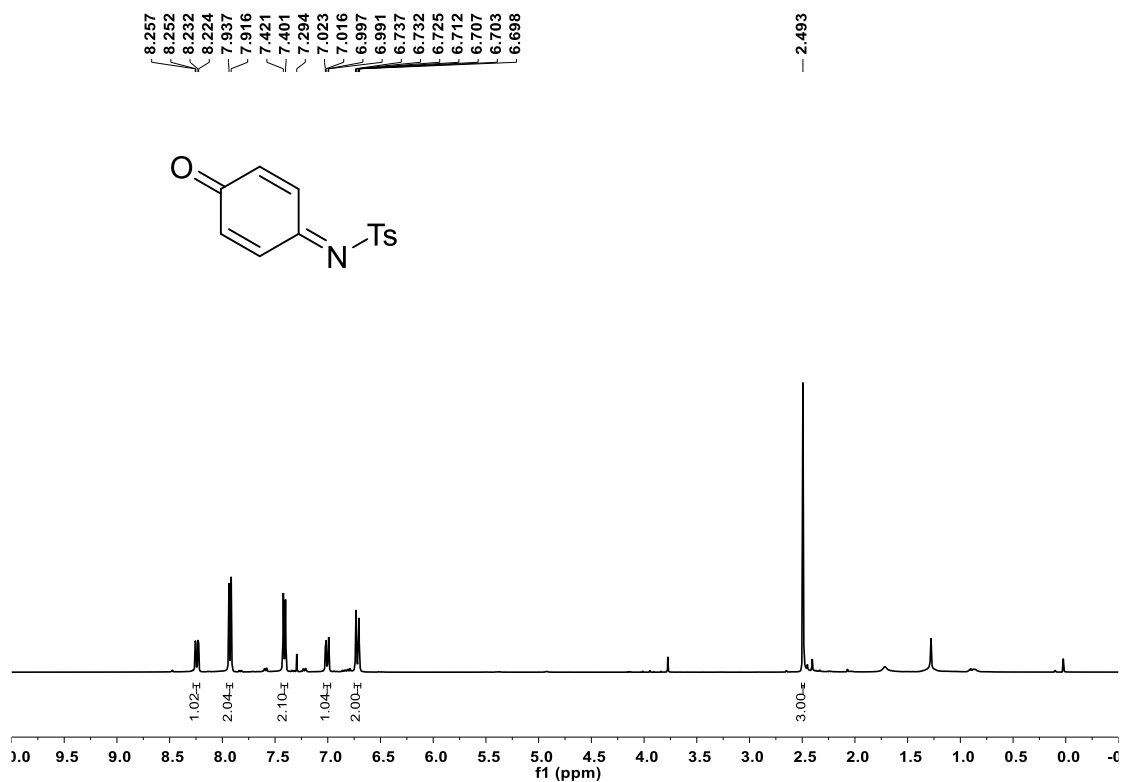


¹³C NMR



4e

¹H NMR



¹³C NMR

