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Supporting Information for

Rh(III)-catalyzed C-H bond activation/annulation reactions of arylacyl ammonium salts with 4-diazoisochroman-3-imines and 4-diazoisoquinolin-3-ones

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General Information:

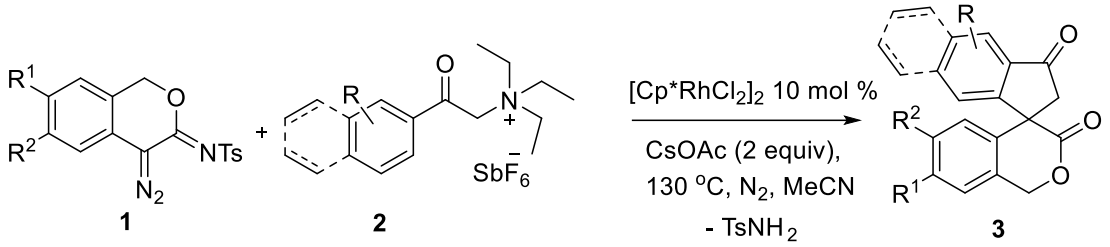
All chemicals, solvents and reagents were acquired from commercial sources and used as received. Melting points were recorded with a micro melting point apparatus. NMR spectral data were recorded at 400 MHz or 600 MHz for ^{1}H NMR, 100 MHz or 150 MHz for ^{13}C NMR. ^{1}H NMR chemical shift values were quoted in parts per million (ppm) referenced to 0.0 ppm for tetramethylsilane. ^{13}C{^{1}H} NMR spectral data were obtained by the same NMR spectrometers and chemical shifts were reported in ppm referenced to the center line of a triplet at 77.00 ppm of $CDCl_3$. The following abbreviations are used to describe peak patterns in NMR data as appropriate: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, td = triplet of doublets, ddd = doublet of doublet of doublets, dt = doublet of triplets. Coupling constants *J* are reported in hertz unit (Hz). All high-resolution mass spectra (HRMS) data were obtained by using ESI ionization on Quadrupole Time-of-Flight (Q-TOF) mass spectrometer. Flash column chromatography was performed using 300-400 mesh silica gel. Thin layer chromatography (TLC) was performed on silica gel HSGF254.

Diazo compounds **1** and **4** were synthesized according to our published procedures. [1] Arylacyl ammonium salts **2** were prepared according to the known procedure. [2]

References:

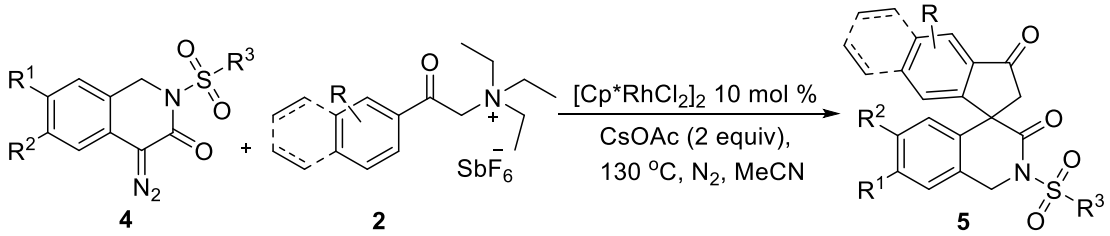
1) (a) A. Ren, P. Lu and Y. Wang, *Chem. Commun.*, 2017, ***53***, 3769-3772. (b) Z. Li, J. Chen, L. Wu, A. Ren, P. Lu and Y. Wang, *Org. Lett.*, 2020, ***22***, 26-30.
2) S. Yu, S. Liu, Y. Lan, B. Wan and X. Li, *J. Am. Chem. Soc.* 2015, ***137***, 1623−1631.

General Procedure for the synthesis of compounds 3



To an oven dried Schlenk flask equipped with a magnetic stirring bar were added 1.6 equiv of ammonium salts **2** (0.32 mmol), 10 mol% of $[Cp^*RhCl_2]_2$ (0.02 mmol) and 2 equiv of CsOAc (0.4 mmol). The air was removed by pump from Schlenk flask and nitrogen gas was introduced. The same procedure was repeated thrice and 2.5 mL of acetonitrile was added to the flask via syringe. The mixture was stirred for a few minutes at room temperature, followed by addition of diazo compound **1** (0.2 mmol) in small portions with continuous flow of nitrogen and stirring. After the complete addition of diazo compound, the schlenk flask was sealed and temperature was raised to 130 °C. After 24 hours, the solvent was removed under reduced pressure and the residue was purified by column chromatography over silica gel by using (PE/EA = 6:1 to 3:1, v/v) or (petroleum ether:ethyl acetate: dichloromethane 5:1:2) to get pure products **3** as off-white solids.

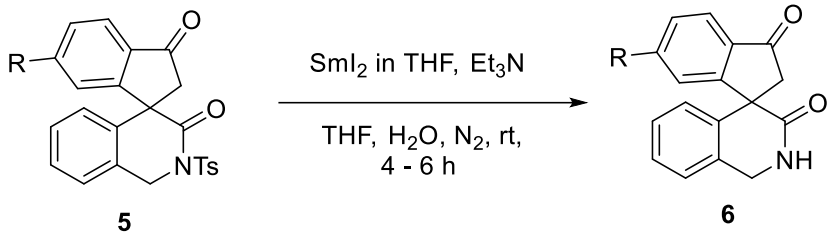
General procedure for preparation of compounds 5



To an oven dried Schlenk flask equipped with a magnetic stirring bar were added 1.6 equivalent of ammonium salts **2** (0.16 mmol), 10 mol% of $[Cp^*RhCl_2]_2$ (0.01 mmol) and 2 equivalents of CsOAc base (0.2 mmol). The air was removed by pump from Schlenk flask and nitrogen gas was introduced. The same procedure was repeated three times and 2.5 mL of acetonitrile solvent was added to the flask via syringe. The mixture was stirred for a few minutes at room temperature, followed by addition of measured amount of diazo compound **4** (0.1 mmol) in small portions with continuous flow of nitrogen and stirring. After the complete addition of diazo compound, the schlenk flask was sealed and temperature was raised to 130 ^oC. After 24 hours, the solvent was removed under reduced pressure and the residue was

purified by column chromatography over silica gel by using (PE/EA = 6:1 to 3:1, v/v) to get pure products **5** as off-white solids.

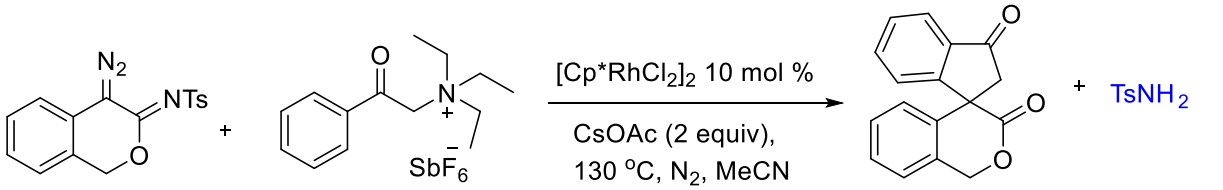
General procedure for preparation of compounds 6:



5 **6**

To an oven dried Schlenk flask equipped with a magnetic stirring bar, measured amount of compound **5** (0.1 mmol) was added. The air was removed by pump from the Schlenk flask and nitrogen gas was introduced (repeated thrice). Sequentially THF (1 mL), H_2O (36.0 equiv.), SmI_2 (6.0 equiv., 0.1 M in THF), and Et_3N (24.0 equiv.) were injected by syringe. The reaction mixture was stirred at room temperature for 4 to 6 hours. The reaction was quenched with saturated $NaHCO_3$ solution and organic layer was extracted with ethyl acetate, dried over $MgSO_4$, concentrated under reduced pressure and purified by flash column chromatography on silica gel (PE/EA, 1:1) to give products **6** as off white solids.

Preparation of Compound 3a at 4 mmole scale (Gram scale):



1a
1 equiv.
4 mmol

2a
1.6 equiv.
6.72 mmol

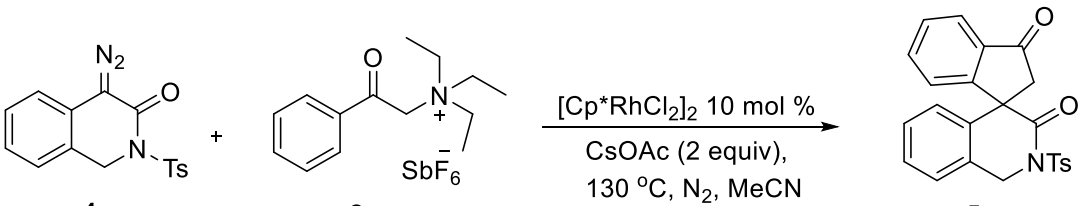
3a
0.637 g
60 % yield

0.386 g
56 % yield

To an oven dried Schlenk flask equipped with a magnetic stirring bar were added 1.6 equivalent of ammonium salts **2a** (6.72 mmol), 10 mol% of $[Cp^*RhCl_2]_2$ (0.4 mmol) and 2 equivalents of CsOAc base (8 mmol). The air was removed by pump from Schlenk flask and nitrogen gas was introduced. The same procedure was repeated thrice and 20 mL of acetonitrile solvent was added to the flask via syringe. The mixture was stirred for a few minutes at room temperature, followed by addition of measured amount of diazo compound **1a** (4 mmol) in small portions with continuous flow of nitrogen and stirring. After the complete addition of diazo compound, the schlenk flask was sealed

and temperature was raised to 130 °C. After 24 hours, the solvent was removed under reduced pressure and the residue was purified by column chromatography over silica gel by using (petroleum ether:ethyl acetate: dichloromethane 5:1:2) to get 0.637g of pure product **3a** as off-white solids along with tosyl-amine as side product.

Preparation of Compound 5a at 3.2 mmole scale (Gram scale):

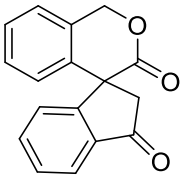


4a	**2a**		**5a**
1 equiv. 3.2 mmol	1.6 equiv. 5.12 mmol		1.035 g 77 % yield

To an oven dried Schlenk flask equipped with a magnetic stirring bar were added 1.6 equivalent of ammonium salt **2a** (5.12 mmol), 10 mol% of $[Cp^*RhCl_2]_2$ (0.32 mmol) and 2 equivalents of CsOAc base (6.4 mmol). The air was removed by pump from Schlenk flask and nitrogen gas was introduced. The same procedure was repeated three times and 15 mL of acetonitrile solvent was added to the flask via syringe. The mixture was stirred for a few minutes at room temperature, followed by addition of measured amount of diazo compound **4a** (3.2 mmol) in small portions with continuous flow of nitrogen and stirring. After the complete addition of diazo compound, the schlenk flask was sealed and temperature was raised to 130 °C. After 24 hours, the solvent was removed under reduced pressure and the residue was purified by column chromatography over silica gel by using (PE/EA = 6:1, v/v) to get 1.035 g of pure products **5a** as off-white solids.

Characterization data of products 3, 5 and 6

Spiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3a)



Off-White solid; R_f = 0.20 (PE/EA = 6:1); Yield: 34 mg, 64 %; M.p. 135 – 137 °C;

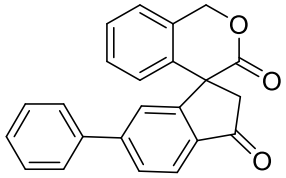
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.89 (m, 1H), 7.75 (td, *J* = 7.2 Hz, 1.6 Hz, 1H), 7.59 (td, *J* = 8.0 Hz, 0.8 Hz, 1H), 7.43 – 7.41 (m, 1H), 7.37 – 7.29 (m, 2H), 7.75 (td, *J* = 7.6 Hz, 2.0 Hz, 1H), 6.59 (d, *J* = 7.6 Hz, 1H), 5.62 (d, *J* = 14.8 Hz, 1H), 5.48 (d, *J* = 14.8 Hz, 1H), 3.42 (d, *J* = 18.0 Hz, 1H), 2.81 (d, *J* = 18.0 Hz, 1H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.7, 172.5, 153.7, 137.8, 136.9, 135.6, 130.8, 129.5, 129.1, 128.0, 127.7, 125.6, 124.9, 123.8, 69.6, 52.6, 48.8.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{12}NaO_3$ 287.0679; Found 287.0679.

IR (film): 2933, 1719, 1602, 1462, 1398, 1288, 1241, 1055, 983, 756 cm^{-1}.

6-Phenylspiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3b)



Off-White solid; R_f = 0.20 (PE/EA = 6:1); Yield: 41 mg, 60 %; M.p. 211 – 213 °C;

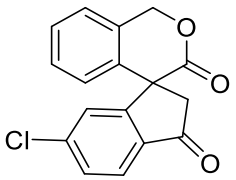
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.0 Hz, 1H), 7.82 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.63 – 7.59 (m, 3H), 7.48 – 7.39 (m, 3H), 7.37 – 7.29 (m, 2H), 7.24 – 7.22 (m, 1H), 6.68 (d, *J* = 8.0 Hz, 1H), 5.63 (d, *J* = 14.8 Hz, 1H), 5.49 (d, *J* = 14.8 Hz, 1H), 3.46 (d, *J* = 18.0 Hz, 1H), 2.84 (d, *J* = 18.0 Hz, 1H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.2, 172.5, 154.4, 148.7, 139.5, 137.8, 135.8, 130.8, 129.2, 128.9, 128.8, 128.6, 128.0, 127.6, 126.0, 125.6, 124.9, 124.2, 69.6, 52.7, 49.2.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{23}H_{16}NaO_3$ 363.0992; Found 363.0990.

IR (film): 3032, 1739, 1716, 1603, 1459, 1394, 1300, 1240, 1058, 980, 765 cm^{-1}.

6-Chlorospiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3c)



Off-White solid; R_f = 0.21 (PE/EA = 6:1); Yield: 30 mg, 50 %; M.p. 64 – 65 °C;

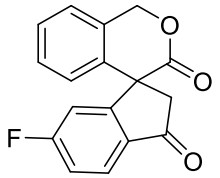
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.4 Hz, 1H), 7.57 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.39 (d, *J* = 1.6 Hz, 1H), 7.36 (dd, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.33 – 7.28 (m, 2H), 6.62 (d, *J* = 7.6 Hz, 1H), 5.61 (d, *J* = 14.4 Hz, 1H), 5.48 (d, *J* = 14.8 Hz, 1H), 3.42 (d, *J* = 18.4 Hz, 1H), 2.81 (d, *J* = 18.4 Hz, 1H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 200.1, 171.9, 155.1, 142.1, 137.1, 135.4, 130.6, 130.4, 129.3, 128.3, 127.9, 125.5, 125.0, 124.9, 69.6, 52.4, 49.0.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{11}ClNaO_3$ 321.094; Found 321.091.

IR (film): 2929, 1739, 1722, 1597, 1464, 1398, 1241, 1144, 1054, 983, 833, 754 cm^{-1}.

6-Fluorospiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3d)



Off-White solid; R_f = 0.24 (PE/EA = 6:1); Yield: 24 mg, 42 %; M.p. 170 – 172 °C;

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.89 (m, 1H), 7.37 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.33 – 7.29 (m, 2H), 7.28 – 7.25 (m, 1H), 7.07 (td, *J* = 7.6 Hz, 1.6 Hz, 1H), 6.62 (d, *J* = 7.6 Hz, 1H), 5.61 (d, *J* = 14.4 Hz, 1H), 5.47 (d, *J* = 14.8 Hz, 1H), 3.42 (d, *J* = 18.4 Hz, 1H), 2.81 (d, *J* = 18.4 Hz, 1H).

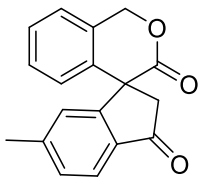
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 199.7, 171.9, 167.3 (d, C-F, $^1J_{C\text{-}F}$ = 256.7 Hz), 156.3 (d, C-F, $^3J_{C\text{-}F}$ = 10.1 Hz), 137.2, 133.5, 130.7, 129.3, 128.3, 126.1 (d, C-F, $^3J_{C\text{-}F}$ = 10.3 Hz), 125.4, 125.0, 118.1 (d, C-F, $^2J_{C\text{-}F}$ = 23.7 Hz), 114.6 (d, C-F, $^2J_{C\text{-}F}$ = 23.1 Hz), 69.6, 52.5, 49.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ - 99.91 ~ - 99.95 (m).

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{11}FNaO_3$ 305.0584; Found 305.0585.

IR (film): 2929, 1728, 1723, 1595, 1483, 1294, 1247, 1201, 1053, 856, 756 cm^{-1}.

6-Methylspiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3e)



Off-White solid; R_f = 0.24 (PE/EA = 6:1); Yield: 28 mg, 50 %; M.p. 71 – 73 °C;

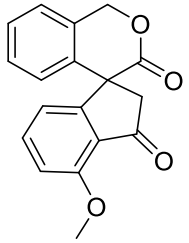
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.0 Hz, 1H), 7.40 (dd, *J* = 8.0 Hz, 2.0 Hz, 1H), 7.34 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.31 – 7.29 (m, 1H), 7.26 – 7.22 (m, 1H), 7.19 (bs, 1H), 6.61 (d, *J* = 7.6 Hz, 1H), 5.61 (d, *J* = 14.8 Hz, 1H), 5.47 (d, *J* = 14.4 Hz, 1H), 3.39 (d, *J* = 18.0 Hz, 1H), 2.78 (d, *J* = 18.0 Hz, 1H), 2.48 (s, 3H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.2, 172.6, 154.2, 147.1, 137.9, 134.8, 130.8, 130.7, 129.1, 127.9, 127.8, 125.7, 124.8, 123.6, 69.6, 52.4, 49.1, 22.3.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{14}NaO_3$ 301.0835; Found 301.0835.

IR (film): 2925, 1733, 1716, 1605, 1458, 1398, 1240, 1197, 1139, 1053, 984, 755 cm^{-1}.

4-Methoxyspiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3f)



Off-White solid; R_f = 0.11 (PE/EA = 3:1); Yield: 26 mg, 44 %; M.p. 182 – 184 °C;

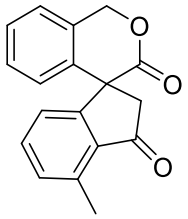
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (t, *J* = 8.0 Hz, 1H), 7.35 – 7.29 (m, 2H), 7.24 (td, *J* = 7.6 Hz, 1.6 Hz, 1H), 6.99 (d, *J* = 8.4 Hz, 1H), 6.92 (d, *J* = 7.6 Hz, 1H), 6.66 (d, *J* = 8.4 Hz, 1H), 5.58 (d, *J* = 14.8 Hz, 1H), 5.46 (d, *J* = 14.4 Hz, 1H), 4.02 (s, 3H), 3.39 (d, *J* = 18.0 Hz, 1H), 2.77 (d, *J* = 18.0 Hz, 1H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 199.2, 172.4, 157.8, 156.1, 137.8, 137.4, 130.6, 129.1, 127.9, 125.7, 125.2, 124.7, 119.3, 110.9, 69.6, 56.0, 52.0, 49.2.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{14}NaO_4$ 317.0784; Found 317.0784.

IR (film): 2937, 1737, 1713, 1594, 1480, 1399, 1285, 1198, 1032, 983, 757 cm^{-1}.

4-Methylspiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3g)



Off-White solid; R_f = 0.22 (PE/EA = 6:1); Yield: 22 mg, 39 %; M.p. 130 – 132 °C;

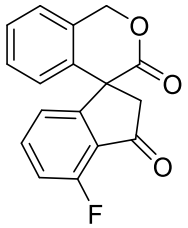
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (t, *J* = 7.6 Hz, 1H), 7.36 – 7.29 (m, 3H), 7.25 – 7.19 (m, 2H), 6.63 (d, *J* = 7.6 Hz, 1H), 5.60 (d, *J* = 14.4 Hz, 1H), 5.47 (d, *J* = 14.4 Hz, 1H), 3.39 (d, *J* = 18.0 Hz, 1H), 2.78 (d, *J* = 18.0 Hz, 1H), 2.72 (s, 3H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 202.6, 172.7, 154.5, 139.0, 138.0, 134.8, 134.3, 131.3, 130.7, 129.1, 127.9, 125.7, 125.0, 124.7, 69.6, 51.9, 49.2, 18.4.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{14}NaO_3$ 301.0835; Found 301.0835.

IR (film): 2920, 1738, 1713, 1593, 1475, 1398, 1241, 1196, 1145, 755 cm^{-1}.

4-Fluorospiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3h)



Off-White solid; R_f = 0.19 (PE/EA = 5:1); Yield: 26 mg, 46 %; M.p. 128 – 130 °C;

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (td, *J* = 8.0 Hz, 4.8 Hz, 1H), 7.37 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.32 – 7.26 (m, 2H), 7.25 – 7.17 (m, 2H), 6.64 (d, *J* = 7.6 Hz, 1H), 5.60 (d, *J* = 14.4 Hz, 1H), 5.48 (d, *J* = 14.8 Hz, 1H), 3.43 (d, *J* = 18.0 Hz, 1H), 2.82 (d, *J* = 18.4 Hz, 1H).

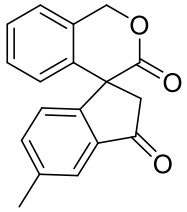
^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 197.8, 171.9, 158.5 (d, C-F, $^1J_{C\text{-}F}$ = 263.7 Hz), 155.6 (d, C-F, $^4J_{C\text{-}F}$ = 2.4 Hz), 137.5 (d, C-F, $^3J_{C\text{-}F}$ = 8.2 Hz), 137.2, 130.6, 129.3, 128.3, 126.5, 125.6, 124.9, 123.6 (d, C-F, $^4J_{C\text{-}F}$ = 4.3 Hz), 116.4 (d, C-F, $^2J_{C\text{-}F}$ = 18.8 Hz), 69.7, 52.5, 49.3.

^{19}F NMR (376 MHz, $CDCl_3$) δ - 114.01 ~ - 114.05 (m).

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{11}FNaO_3$ 305.0584; Found 305.0583.

IR (film): 2933, 1728, 1612, 1594, 1474, 1399, 1243, 1200, 1161, 1078, 978, 757 cm^{-1}.

5-Methylspiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3i)



Off-White solid; R_f = 0.19 (PE/EA = 5:1); Yield: 28 mg, 50 %; M.p. 192 – 193 °C;

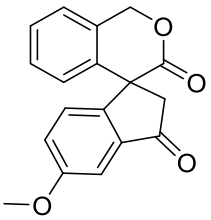
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (s, 1H), 7.56 (dd, *J* = 7.8 Hz, 1.8 Hz, 1H), 7.33 (td, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.30 – 7.28 (m, 2H), 7.22 (t, *J* = 7.8 Hz, 1H), 6.62 (d, *J* = 7.8 Hz, 1H), 5.59 (d, *J* = 14.4 Hz, 1H), 5.46 (d, *J* = 15.0 Hz, 1H), 3.40 (d, *J* = 18.0 Hz, 1H), 2.78 (d, *J* = 18.0 Hz, 1H), 2.49 (s, 3H).

^{13}C{^{1}H} **NMR (150 MHz, $CDCl_3$)** δ 201.7, 172.6, 151.2, 139.8, 137.9, 137.1, 136.9, 130.8, 129.0, 127.9, 127.3, 125.5, 124.8, 123.7, 69.6, 52.3, 49.2, 21.2.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{14}NaO_3$ 301.0835; Found 301.0836.

IR (film): 2942, 1739, 1719, 1489, 1399, 1286, 1241, 1181, 1057, 983, 756 cm^{-1}.

5-Methoxyspiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3j)



Off-White solid; R_f = 0.13 (PE/EA = 5:1); Yield: 30 mg, 51 %; M.p. 183 – 185 °C;

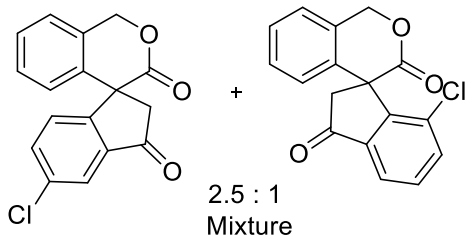
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.32 (m, 2H), 7.31 – 7.28 (m, 3H), 7.24 (td, *J* = 7.6 Hz, 1.6 Hz, 1H), 6.64 (d, *J* = 8.0 Hz, 1H), 5.60 (d, *J* = 14.4 Hz, 1H), 5.45 (d, *J* = 14.4 Hz, 1H), 3.91 (s, 3H), 3.42 (d, *J* = 18.0 Hz, 1H), 2.80 (d, *J* = 18.4 Hz, 1H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 201.6, 172.7, 160.8, 146.3, 138.4, 138.0, 130.9, 129.1, 128.4, 127.9, 125.5, 125.0, 124.9, 104.8, 69.5, 55.7, 52.1, 49.4.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{14}NaO_4$ 317.0784; Found 317.0785.

IR (film): 2937, 1738, 1716, 1614, 1490, 1333, 1284, 1242, 1054, 854, 779 cm^{-1}.

5-Chlorospiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione&7-chlorospiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3k & 3ka)



Off-White solid; R_f = 0.18 (PE/EA = 5:1); Yield: 36 mg, 60 %; M.p. 133 – 135 °C;

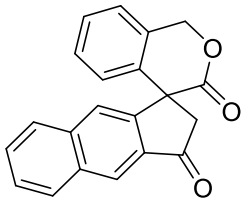
^{1}H NMR (600 MHz, $CDCl_3$) Major: δ 7.85 – 7.83 (m, 0.85H), 7.70 (dd, *J* = 8.4 Hz, 2.4 Hz, 0.86H), 7.37 – 7.34 (m, 2H), 7.33 – 7.30 (m, 1H), 7.25 – 7.22 (m, 1H), 6.60 (d, *J* = 7.8 Hz, 0.80H), 5.60 (d, *J* = 15.0 Hz, 1H), 5.46 (d, *J* = 14.4 Hz, 1H), 3.42 (d, *J* = 18.0 Hz, 1H), 2.82 (d, *J* = 18.0 Hz, 1H). **Minor:** δ 7.74 (dd, *J* = 7.8 Hz, 1.2 Hz, 0.42H), 7.57 (d, *J* = 7.8 Hz, 0.44H), 7.48 (t, *J* = 7.2 Hz, 0.45H), 7.28 (d, *J* = 7.8 Hz, 1H), 7.17 (t, *J* = 7.8 Hz, 0.53H), 6.40 (d, *J* = 7.8 Hz, 0.34H), 5.50 (d, *J* = 16.2 Hz, 0.51H), 5.02 (d, *J* = 16.2 Hz, 0.38H), 3.11 (d, *J* = 18.6 Hz, 0.40H), 2.71 (d, *J* = 18.6 Hz, 0.42H).

^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) Major: δ 200.2, 172.0, 151.7, 135.6, 131.0, 130.8, 129.4, 129.3, 128.9, 128.7, 128.2, 125.4, 125.0, 123.6, 69.6, 50.8, 49.2. **Minor:** δ 201.2, 151.2, 145.1, 139.4, 138.5, 137.3, 136.3, 136.2, 134.9, 133.0, 128.8, 129.6, 125.1, 124.8, 124.1, 52.3, 48.0.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{11}ClNaO_3$ 321.0289; Found 321.0289.

IR (film): 3075, 2929, 1724, 1596, 1460, 1400, 1241, 1199, 1053, 983, 755 cm^{-1}.

Spiro[cyclopenta[*b*]naphthalene-1,4'-isochromane]-3,3'(2*H*)-dione (3l)



Off-White solid; R_f = 0.20 (PE/EA = 6:1); Yield: 36 mg, 57 %; M.p. 160 – 162 °C;

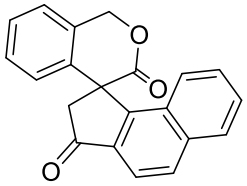
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (s, 1H), 8.08 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 8.4 Hz, 1H), 7.84 (s, 1H), 7.66 – 7.57 (m, 2H), 7.37 – 7. 31 (m, 2H), 7.22 (td, *J* = 7.6 Hz, 2.0 Hz, 1H), 6.69 (d, *J* = 7.6 Hz, 1H), 5.66 (d, *J* = 14.4 Hz, 1H), 5.57 (d, *J* = 14.8 Hz, 1H), 3.56 (d, *J* = 18.4 Hz, 1H), 2.96 (d, *J* = 18.4 Hz, 1H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.9, 172.7, 147.1, 138.1, 137.3, 133.8, 133.1, 130.6, 130.4, 129.14, 129.12, 128.5, 127.9, 126.6, 125.8, 124.9, 124.8, 69.7, 52.4, 49.9.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{21}H_{14}NaO_3$ 337.0835; Found 337.0836.

IR (film): 3059, 1721, 1627, 1451, 1393, 1339, 1239, 1175, 1051, 890, 753, 453 cm^{-1}.

Spiro[cyclopenta[*a*]naphthalene-1,4'-isochromane]-3,3'(2*H*)-dione (3la)



Off-White solid; R_f = 0.20 (PE/EA = 6:1); Yield: 16 mg, 25 %; M.p. 95 – 97 °C;

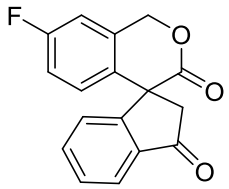
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.4 Hz, 1H), 8.01 (d, *J* = 8.0 Hz, 1H), 7.88 (d, *J* = 8.8 Hz, 1H), 7.62 (td, *J* = 8.0 Hz, 1.2 Hz, 1H), 7.40 – 7.27 (m, 4H), 7.09 (t, *J* = 7.6 Hz, 1H), 6.33 (d, *J* = 7.6 Hz, 1H), 5.72 (d, *J* = 14.4 Hz, 1H), 5.42 (d, *J* = 14.4 Hz, 1H), 3.50 (d, *J* = 18.4 Hz, 1H), 2.87 (d, *J* = 18.0 Hz, 1H).

^{13}C{^{1}H} **NMR (150 MHz, $CDCl_3$)** δ 201.4, 172.4, 153.4, 137.8, 137.7, 136.2, 131.4, 130.1, 129.4, 129.2, 129.1, 128.0, 127.4, 126.4, 125.8, 124.9, 128.96, 69.7, 52.1, 49.5.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{21}H_{14}NaO_3$ 337.0835; Found 337.0836.

IR (film): 2929, 1738, 1714, 1623, 1461, 1399, 1243, 1199, 1077, 981, 818, 751 cm^{-1}.

7'-Fluorospiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3m)



Off-White solid; R_f = 0.18 (PE/EA = 6:1); Yield: 18 mg, 32 %; M.p. 80 – 82 °C;

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.8 Hz, 1H), 7.75 (td, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.60 (t, *J* = 7.8 Hz, 1H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.03 (dd, *J* = 8.4 Hz, 2.4 Hz, 1H), 6.93 (td, *J* = 8.4 Hz, 2.4 Hz, 1H), 6.58 (dd, *J* = 9.0 Hz, 4.8 Hz, 1H), 5.58 (d, *J* = 14.4 Hz, 1H), 5.44 (d, *J* = 15.0 Hz, 1H), 3.41 (d, *J* = 18.0 Hz, 1H), 2.77 (d, *J* = 18.0 Hz, 1H).

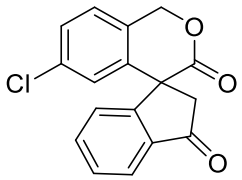
^{13}C{^{1}H} **NMR (150 MHz, $CDCl_3$)** δ 201.3, 171.9, 162.0, (d, C-F, $^{1}J_{C\text{-}F}$ = 247.9 Hz), 153.5, 136.9, 135.7, 133.6 (d, C-F, $^{4}J_{C\text{-}F}$ = 3.3 Hz), 132.9 (d, C-F, $^{3}J_{C\text{-}F}$ = 7.9 Hz), 129.6, 127.7 (d, C-F, $^{3}J_{C\text{-}F}$ = 8.5 Hz), 127.5, 123.9, 116.1 (d, C-F, $^{2}J_{C\text{-}F}$ = 21.7 Hz), 112.1 (d, C-F, $^{2}J_{C\text{-}F}$ = 22.9 Hz), 68.9 (d, *J* = 2.2 Hz), 52.2, 49.0.

^{19}F NMR (376 MHz, $CDCl_3$) δ - 112.88 – - 112.94.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{11}FNaO_3$ 305.0584; Found 305.0585.

IR (film): 3071, 2925, 1740, 1720, 1601, 1496, 1377, 1272, 1206, 1142, 1057, 961, 765 cm^{-1}.

6'-Chlorospiro[indene-1,4'-isochromane]-3,3'(2*H*)-dione (3n)



Off-White solid; R_f = 0.29 (PE/EA = 3:1); Yield: 24 mg, 40 %; M.p. 123 – 125 °C;

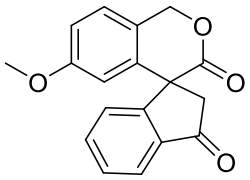
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 7.2 Hz, 1H), 7.79 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.63 (td, *J* = 7.2 Hz, 0.8 Hz, 1H), 7.41 (d, *J* = 7.6 Hz, 1H), 7.33 (dd, *J* = 8.0 Hz, 2.0 Hz, 1H), 7.25 - 7.24 (m, 1H), 6.57 (d, *J* = 2.0Hz, 1H), 5.57 (d, *J* = 14.4 Hz, 1H), 5.45 (d, *J* = 14.4 Hz, 1H), 3.41 (d, *J* = 18.0 Hz, 1H), 2.78 (d, *J* = 18.4 Hz, 1H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 200.9, 171.7, 152.8, 139.9, 136.9, 135.9, 135.2, 129.8, 129.3, 128.3, 127.6, 126.3, 125.8, 124.0, 69.0, 52.5, 48.6.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{11}ClNaO_3$ 321.0289; Found 321.0290.

IR (film): 2925, 1739, 1719, 1602, 1463, 1381, 1239, 100, 1146, 1056, 938, 763 cm^{-1}.

6'-Methoxyspiro[indene-1,4'-isochromane]-3,3'(2H)-dione (3o)



Off-White solid; R_f = 0.13 (PE/EA = 3:1); Yield: 28 mg, 48 %; M.p. 179 – 181 °C;

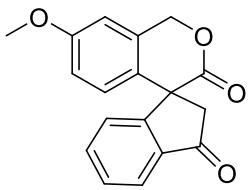
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.0 Hz, 1H), 7.75 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.68 (td, *J* = 7.6 Hz, 0.8 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 8.4 Hz, 1H), 6.85 (dd, *J* = 8.4 Hz, 2.8 Hz, 1H), 6.08 (d, *J* = 2.8 Hz, 1H), 5.56 (d, *J* = 14.4 Hz, 1H), 5.41 (d, *J* = 14.0 Hz, 1H), 3.66 (s, 3H), 3.40 (d, *J* = 18.0 Hz, 1H), 2.80 (d, *J* = 18.4 Hz, 1H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.6, 172.4, 160.1, 153.4, 139.5, 137.0, 135.6, 129.5, 127.8, 126.2, 123.8, 123.0, 112.7, 111.9, 69.3, 55.3, 52.9, 48.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{14}NaO_4$ 317.0784; Found 317.0786.

IR (film): 2941, 1739, 1716, 1615, 1491, 1336, 1283, 1241, 1054, 854, 779 cm^{-1}.

7'-Methoxyspiro[indene-1,4'-isochromane]-3,3'(2H)-dione (3p)



Off-White solid; R_f = 0.11 (PE/EA = 3:1); Yield: 22 mg, 37 %; M.p. 184– 186 °C;

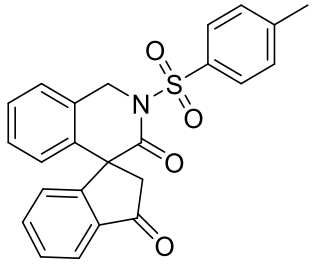
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.6 Hz, 1H), 7.73 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.58 (td, *J* = 7.6 Hz, 0.8 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 1H), 6.81 (d, *J* = 2.8 Hz, 1H), 6.74 (dd, *J* = 8.4 Hz, 2.4 Hz, 1H), 6.50 (d, *J* = 8.4 Hz, 1H), 5.58 (d, *J* = 14.4 Hz, 1H), 5.42 (d, *J* = 14.8 Hz, 1H), 3.81 (s, 3H), 3.39 (d, *J* = 18.0 Hz, 1H), 2.78 (d, *J* = 18.4 Hz, 1H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 202.1, 172.6, 159.3, 143.5, 139.0, 136.9, 135.6, 132.0, 129.4, 127.5, 126.9, 123.7, 114.5, 110.3, 69.6, 55.5, 52.1, 49.3.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{14}NaO_4$ 317.0784; Found 317.0785.

IR (film): 2941, 1741, 1718, 1603, 1463, 1358, 1288, 1175, 1063, 814, 779 cm^{-1}.

2'-Tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5a)



Off-White solid; R_f = 0.25 (PE/EA = 6:1); Yield: 33 mg, 79 %; M.p. 91 – 93 °C;

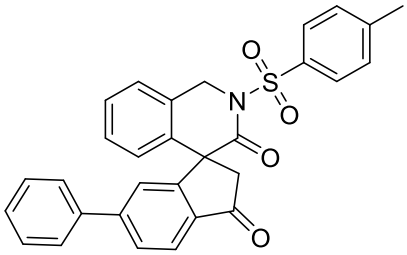
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.80 (dd, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.66 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.39 (d, *J* = 7.6 Hz, 1H), 7.35 – 7.28 (m, 4H), 7.19 (t, *J* = 7.2 Hz, 1H), 6.54 (d, *J* = 8.0 Hz, 1H), 5.52 (d, *J* = 16.0 Hz, 1H), 4.98 (d, *J* = 16.0 Hz, 1H), 3.04 (d, *J* = 18.4 Hz, 1H), 2.69 (d, *J* = 18.4 Hz, 1H), 2.41 (s, 3H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.9, 170.8, 153.7, 145.4, 138.1, 136.9, 135.5, 134.8, 130.3, 129.5, 129.3, 128.7, 128.5, 128.0, 127.5, 126.4, 125.8, 123.7, 55.7, 48.9, 48.2, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{19}NNaO_4S$ 440.0927; Found 440.0930.

IR (film): 2923, 1717, 1598, 1462, 1359, 1288, 1171, 1088, 1016, 778, 666, 551 cm^{-1}.

6-Phenyl-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5b)



Off-White solid; R_f = 0.17 (PE/EA = 6:1); Yield: 35 mg, 71 %; M.p. 114 – 116 °C;

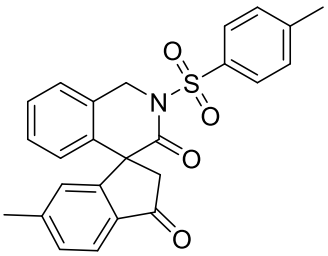
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.80 (dd, *J* = 8.0 Hz, 0.4 Hz, 1H), 7.73 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.54 – 7.51 (m, 2H), 7.46 – 7.38 (m, 5H), 7.33 (td, *J* = 7.2. Hz, 1.2 Hz, 1H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.20 (td, *J* = 7.6 Hz, 1.6 Hz, 1H), 6.63 (d, *J* = 8.0 Hz, 1H), 5.54 (d, *J* = 16.0 Hz, 1H), 5.01 (d, *J* = 16.0 Hz, 1H), 3.08 (d, *J* = 18.0 Hz, 1H), 2.73 (d, *J* = 18.0 Hz, 1H), 2.39 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 201.5, 170.9, 154.7, 148.6, 145.4, 139.4, 138.0, 135.7, 134.7, 130.2, 129.6, 128.9, 128.8, 128.6, 128.5, 128.0, 127.5, 126.4, 125.9, 125.7, 124.1, 55.7, 49.4, 48.2, 21.6.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{30}H_{23}NNaO_4S$ 516.1240; Found 516.1240.

IR (film): 3033, 2933, 2255, 1717, 1601, 1455, 1360, 1258, 1188, 1114, 1088, 1036, 910, 813, 765, 665, 552 cm^{-1}.

6-Methyl-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5c)



Off-White solid; R_f = 0.19 (PE/EA = 6:1); Yield: 30.2 mg, 70 %; M.p. 106 – 108 °C;

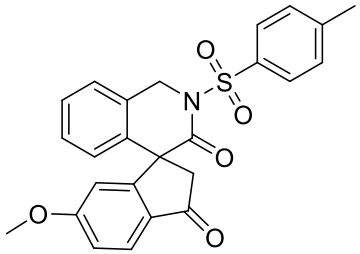
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.0 Hz, 1H), 7.38 (d, J = 7.6 Hz, 1H), 7.34 – 7.29 (m, 4H), 7.19 (d, J = 7.6 Hz, 1H), 7.07 (s, 1H), 6.55 (d, J = 8.0 Hz, 1H), 5.52 (d, J = 15.6 Hz, 1H), 4.96 (d, J = 16.0 Hz, 1H), 2.98 (d, J = 18.0 Hz, 1H), 2.65 (d, J = 18.0 Hz, 1H), 2.41 (s, 3H), 2.40 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 201.4, 171.0, 154.3, 146.9, 145.4, 138.3, 134.8, 134.7, 130.7, 130.3, 129.6, 128.7, 128.5, 127.9, 127.6, 126.3, 125.9, 123.5, 55.5, 49.2, 48.2, 22.2, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{21}NNaO_4S$ 454.1083; Found 454.1084.

IR (film): 2924, 2255, 1716, 1604, 1492, 1492, 1359, 1284, 1172, 1116, 1088, 1051, 817, 731, 666, 549 cm^{-1}.

6-Methoxy-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5d)



Off-White solid; R_f = 0.19 (PE/EA = 3:1); Yield: 35 mg, 71 %; M.p. 114 – 116 °C;

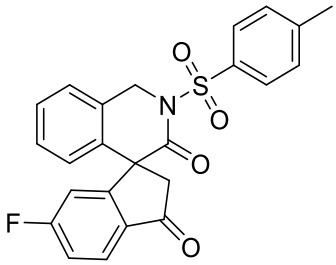
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 8.4 Hz, 2H), 7.73 (d, J = 8.4 Hz, 1H), 7.39 (d, J = 7.2 Hz, 1H), 7.34 (dd, J = 7.6 Hz, 1.6 Hz, 1H), 7.31 (d, J = 8.4 Hz, 2H), 7.20 (t, J = 7.6 Hz, 1H), (dd, J = 8.0 Hz, 2.0 Hz, 1H), 6.68 (d, J = 2.0 Hz, 1H), 6.57 (d, J = 7.6 Hz, 1H), 5.52 (d, J = 15.6 Hz, 1H), 4.93 (d, J = 15.6 Hz, 1H), 3.81 (s, 3H), 2.93 (d, J = 18.4 Hz, 1H), 2.62 (d, J = 18.0 Hz, 1H), 2.42 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 199.8, 170.8, 165.7, 156.5, 145.4, 138.3, 134.7, 130.5, 130.3, 129.6, 128.8, 128.5, 127.9, 126.3, 125.9, 125.3, 117.6, 110.6, 55.8, 55.6, 49.2, 48.1, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{21}NNaO_5S$ 470.1033; Found 470.1034.

IR (film): 2943, 1709, 1598, 1491, 1358, 1251, 1172, 1116, 1087, 1021, 911, 731, 669, 545 cm^{-1}.

6-Fluoro-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5e)



Off-White solid; R_f = 0.19 (PE/EA = 6:1); Yield: 28 mg, 64 %; M.p. 215 – 217 °C;

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.81 (dd, *J* = 8.8 Hz, 5.2 Hz, 1H), 7.41 (d, *J* = 7.2 Hz, 1H), 7.37 – 7.30 (m, 3H), 7.24 – 7.19 (m, 2H), 6.92 (dd, *J* = 8.4 Hz, 2.0 Hz, 1H), 6.56 (d, *J* = 8.0 Hz, 1H), 5.51 (d, *J* = 16.0 Hz, 1H), 4.97 (d, *J* = 16.0 Hz, 1H), 3.05 (d, *J* = 18.4 Hz, 1H), 2.71 (d, *J* = 18.4 Hz, 1H), 2.42 (s, 3H).

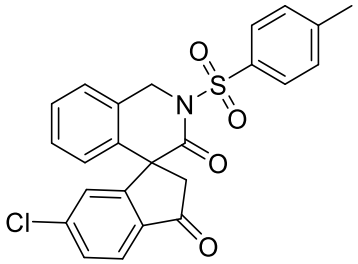
^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 199.9, 170.4, 167.1 (d, C-F, $^1J_{C\text{-}F}$ = 256.7 Hz), 156.4 (d, C-F, $^3J_{C\text{-}F}$ = 9.8 Hz), 145.5, 137.5, 134.7, 133.5 (d, C-F, $^4J_{C\text{-}F}$ = 1.8 Hz), 130.3, 129.6, 128.9, 128.6, 128.3, 126.5, 126.1 (d, C-F, $^3J_{C\text{-}F}$ = 10.3 Hz), 125.7, 117.9 (d, C-F, $^2J_{C\text{-}F}$ = 23.7 Hz), 114.4 (d, C-F, $^2J_{C\text{-}F}$ = 22.9 Hz), 55.5 (d, *J* = 1.8 Hz), 49.2, 48.1, 21.7.

^{19}F NMR (376 MHz, $CDCl_3$) δ - 100.13 ~ - 100.16 (m).

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{18}FNNaO_4S$ 458.0833; Found 458.0834.

IR (film): 2925, 1721, 1703, 1595, 1482, 1360, 1248, 1172, 1117, 1087, 1052, 911, 816, 732, 668, 552 cm^{-1}.

6-Chloro-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5f)



Off-White solid; R_f = 0.21 (PE/EA = 6:1); Yield: 35 mg, 71 %; M.p. 192 – 194 °C;

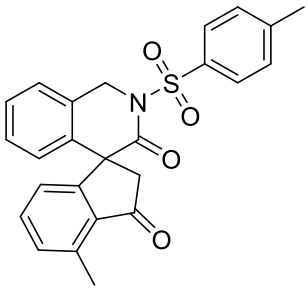
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.48 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.41 – 7.30 (m, 4H), 7.25 – 7.21 (m, 2H), 6.56 (d, *J* = 8.0 Hz, 1H), 5.51 (d, *J* = 15.6 Hz, 1H), 4.98 (d, *J* = 15.6 Hz, 1H), 3.05 (d, *J* = 18.4 Hz, 1H), 2.70 (d, *J* = 18.4 Hz, 1H), 2.42 (s, 3H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 200.3, 170.4, 155.1, 145.5, 141.9, 137.4, 135.4, 136.6, 130.3, 130.2, 129.6, 128.9, 128.5, 128.3, 127.6, 126.5, 125.8, 124.8, 55.5, 49.2, 48.1, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{18}ClNNaO_4S$ 474.0537; Found 474.0539.

IR (film): 2920, 2254, 1722, 1702, 1596, 1360, 1188, 1115, 1088, 911, 814, 732, 666, 550 cm^{-1}.

4-Methyl-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5g)



Off-White solid; R_f = 0.23 (PE/EA = 6:1); Yield: 21 mg, 49 %; M.p. 86 – 88 °C;

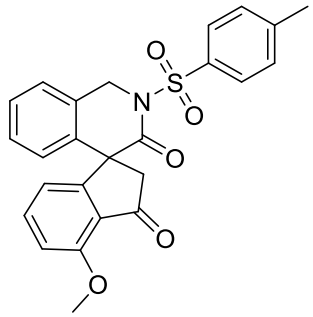
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.33 (dd, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 7.6 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 1H), 6.57 (d, *J* = 7.6 Hz, 1H), 5.51 (d, *J* = 15.6 Hz, 1H), 4.97 (d, *J* = 15.6 Hz, 1H), 3.01 (d, *J* = 18.0 Hz, 1H), 2.66 (d, *J* = 18.0 Hz, 1H), 2.64 (s, 3H), 2.42 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 202.8, 171.2, 154.6, 145.4, 139.0, 138.4, 134.7, 134.3, 131.2, 130.3, 129.6, 128.7, 128.6, 127.9, 126.3, 126.1, 124.9, 55.0, 49.3, 48.2, 21.7, 18.3.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{21}NNaO_4S$ 454.1083; Found 454.1084.

IR (film): 2916, 2262, 1710, 1594, 1474, 1358, 1257, 1171, 1115, 1088, 1046, 802, 667, 551 cm^{-1}.

4-Methoxy-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5h)



Off-White solid; R_f = 0.11 (PE/EA = 3:1); Yield: 28.3 mg, 63 %; M.p. 110 – 112 °C;

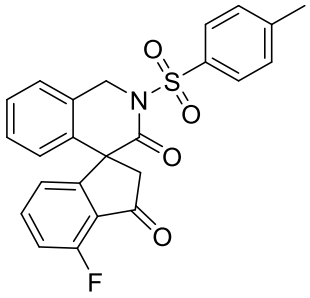
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.4 Hz, 2H), 7.58 (t, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 7.2 Hz, 1H), 7.34 – 7.29 (m, 3H), 7.19 (t, *J* = 7.6 Hz, 1H), 6.92 (d, *J* = 8.4 Hz, 1H), 6.81 (d, *J* = 7.6 Hz, 1H), 6.61 (d, *J* = 8.0 Hz, 1H), 5.49 (d, *J* = 15.6 Hz, 1H), 4.94 (d, *J* = 15.6 Hz, 1H), 3.97 (s, 3H), 2.97 (d, *J* = 18.0 Hz, 1H), 2.64 (d, *J* = 18.0 Hz, 1H), 2.41 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 199.5, 170.8, 157.8, 156.2, 145.4, 138.1, 137.2, 134.7, 130.1, 129.5, 128.7, 128.6, 127.9, 126.3, 125.9, 125.1, 119.1, 110.8, 55.9, 55.0, 49.2, 48.2, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{21}NNaO_5S$ 470.1033; Found 470.1032.

IR (film): 2938, 2252, 1712, 1594, 1480, 1358, 1285, 1188, 1116, 1031, 911, 732, 668, 546 cm^{-1}.

4-Fluoro-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5i)



Off-White solid; R_f = 0.16 (PE/EA = 6:1); Yield: 27 mg, 62 %; M.p. 246 – 248 °C;

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.4 Hz, 2H), 7.63 (td, J = 8.0 Hz, 4.8 Hz, 1H), 7.39 (d, J = 7.2 Hz, 1H), 7.36 (dd, J = 7.6 Hz, 1.2 Hz, 1H), 7.31 (d, J = 8.0 Hz, 2H), 7.22 (t, J = 7.6 Hz, 1H), 7.13 (t, J = 8.4 Hz, 1H), 7.05 (d, J = 8.0 Hz, 1H), 6.59 (d, J = 7.6 Hz, 1H), 5.51 (d, J = 16.0 Hz, 1H), 4.96 (d, J = 16.0 Hz, 1H), 3.04 (d, J = 18.4 Hz, 1H), 2.71 (d, J = 18.4 Hz, 1H), 2.42 (s, 3H).

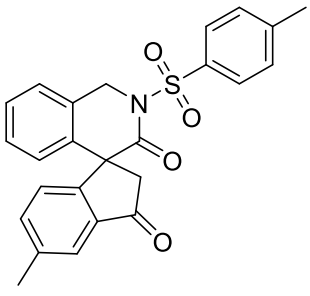
^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 198.0, 170.4, 158.4 (d, C-F, $^1J_{C\text{-}F}$ = 263.5 Hz), 155.7 (d, C-F, $^4J_{C\text{-}F}$ = 2.0 Hz), 145.5, 137.4, 137.3 (d, C-F, $^3J_{C\text{-}F}$ = 8.4 Hz), 134.6, 130.2, 129.6, 128.9, 128.6, 128.2, 126.5, 125.8, 124.9 (d, C-F, $^3J_{C\text{-}F}$ = 13.4 Hz), 123.3 (d, C-F, $^4J_{C\text{-}F}$ = 4.3 Hz), 116.3 (d, C-F, $^2J_{C\text{-}F}$ = 18.8 Hz), 55.5, 49.4, 48.2, 21.7.

^{19}F NMR (376 MHz, $CDCl_3$) δ - 114.07 ~ - 114.11 (m).

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{18}FNNaO_4S$ 458.0833; Found 458.0830.

IR (film): 2925, 2262, 1727, 1699, 1611, 1595, 1473, 1360, 1172, 1116, 1088, 902, 804, 667, 553 cm^{-1}.

5-Methyl-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5j)



Off-White solid; R_f = 0.25 (PE/EA = 5:1); Yield: 34 mg, 79 %; M.p. 129 – 131 °C;

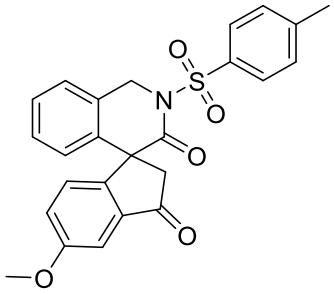
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 8.4 Hz, 2H), 7.59 (bs, 1H), 7.48 (dd, J = 8.0 Hz, 1.6 Hz, 1H), 7.38 (d, J = 7.6 Hz, 1H), 7.33 (dd, J = 7.6 Hz, 1.2 Hz, 1H), 7.29 (d, J = 8.4 Hz, 2H), 7.20 – 7.16 (m, 2H), 6.57 (d, J = 7.6 Hz, 1H), 5.51 (d, J = 15.6 Hz, 1H), 4.96 (d, J = 16.0 Hz, 1H), 3.00 (d, J = 18.4 Hz, 1H), 2.67 (d, J = 18.4 Hz, 1H), 2.43 (s, 3H), 2.41 (s, 3H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.9, 171.0, 151.1, 145.3, 139.7, 138.3, 137.2, 136.8, 134.8, 130.4, 129.5, 128.7, 128.6, 127.9, 127.2, 126.3, 125.8, 123.6, 55.4, 49.2, 48.2, 21.7, 21.2.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{21}NNaO_4S$ 454.1083; Found 454.1083.

IR (film): 2924, 2249, 1717, 1612, 1596, 1492, 1359, 1285, 1171, 1115, 1088, 911, 815, 732, 666, 552 cm^{-1}.

5-Methoxy-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione(5k)



Off-White solid; R_f = 0.12 (PE/EA = 5:1); Yield: 27 mg, 60 %; M.p. 95 – 97 °C;

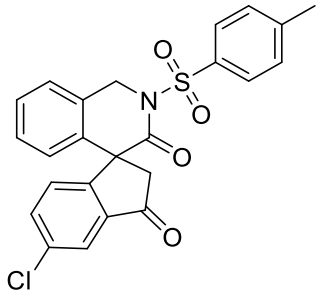
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, J = 8.4 Hz, 2H), 7.38 (d, J = 7.6 Hz, 1H), 7.33 (dd, J = 7.2 Hz, 1.2 Hz, 1H), 7.29 (d, J = 8.4 Hz, 2H), 7.24 (dd, J = 8.4 Hz, 2.4 Hz, 1H), 7.21 – 7.16 (m, 3H), 6.59 (d, J = 7.6 Hz, 1H), 5.50 (d, J = 15.6 Hz, 1H), 4.95 (d, J = 15.6 Hz, 1H), 3.86 (s, 3H), 3.02 (d, J = 18.0 Hz, 1H), 2.69 (d, J = 18.0 Hz, 1H), 2.42 (s, 3H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.8, 171.1, 160.7, 146.3, 145.3, 138.5, 138.4, 134.8, 130.5, 129.6, 128.7, 128.6, 128.3, 127.9, 126.4, 125.8, 124.8.104.8, 55.7, 55.2, 49.5, 48.1, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{21}NNaO_5S$ 470.1033; Found 470.1036.

IR (film): 2926, 2254, 1716, 1612, 1592, 1489, 1358, 1284, 1171, 1114, 1050, 780, 665, 544 cm^{-1}.

5-Chloro-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5l)



Off-White solid; R_f = 0.30 (PE/EA = 5:1); Yield: 20 mg, 44 %; M.p. 105 – 107 °C;

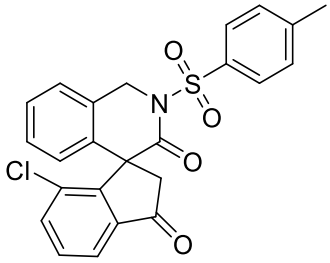
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.75 (d, *J* = 2.0 Hz, 1H), 7.61 (dd, *J* = 8.0 Hz, 2.0 Hz, 1H), 7.40 (d, *J* = 7.2 Hz, 1H), 7.34 (dd, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.24 – 7.19 (m, 2H), 6.55 (d, *J* = 7.6 Hz, 1H), 5.51 (d, *J* = 16.0 Hz, 1H), 4.95 (d, *J* = 16.0 Hz, 1H), 3.03 (d, *J* = 18.4 Hz, 1H), 2.71 (d, *J* = 18.4 Hz, 1H), 2.42 (s, 3H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 200.4, 170.5, 151.7, 145.5, 138.5, 137.6, 136.0, 135.5, 134.7, 130.4, 129.6, 128.9, 128.8, 128.6, 128.3, 126.5, 125.7, 123.6, 55.4, 49.3, 48.1, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{18}ClNNaO_4S$ 474.0537; Found 474.0540.

IR (film): 2924, 2285, 1723, 1698, 1596, 1465, 1359, 1257, 1171, 1115, 1088, 911, 732, 666, 552 cm^{-1}.

7-Chloro-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5la)



Off-White solid; R_f = 0.24 (PE/EA = 5:1); Yield: 22.2 mg, 49 %; M.p. 193 – 195 °C;

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.75 (dd, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.58 (dd, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.38 (d, *J* = 7.2 Hz, 1H), 7.33 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.17 (t, *J* = 7.6 Hz, 1H), 6.51 (d, *J* = 7.6

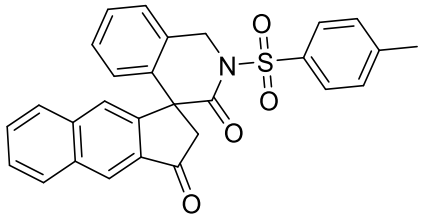
Hz, 1H), 5.51 (d, *J* = 16.0 Hz, 1H), 5.01 (d, *J* = 15.6 Hz, 1H), 3.10 (d, *J* = 18.4 Hz, 1H), 2.71 (d, *J* = 18.8 Hz, 1H), 2.41 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 201.2, 169.9, 151.2, 145.3, 139.3, 136.3, 136.3, 136.2, 134.9, 133.0, 131.0, 129.4, 129.3, 128.8, 128.6, 127.9, 126.2, 125.0, 122.1, 54.9, 50.7, 47.9, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{18}ClNNaO_4S$ 474.0537; Found 474.0540.

IR (film): 2924, 2245, 1724, 1699, 1595, 1460, 1359, 1260, 1172, 1121, 1073, 911, 732, 666, 552 cm^{-1}.

2'-Tosyl-1',2'-dihydro-3'*H*-spiro[cyclopenta[*b*]naphthalene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5m)



Off-White solid; R_f = 0.18 (PE/EA = 6:1); Yield: 25 mg, 53 %; M.p. 123 – 125 °C;

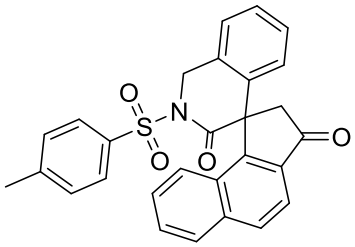
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.36 (s, 1H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.92 (d, *J* = 8.4 Hz, 2H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.68 (s, 1H), 7.62 – 7.53 (m, 2H), 7.41 (d, *J* = 7.6 Hz, 1H), 7.33 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.26 – 7.24 (m, 2H), 7.17 (t, *J* = 7.6 Hz,1H), 6.65 (d, *J* = 8.0 Hz, 1H), 5.56 (d, *J* = 15.6 Hz, 1H), 5.07 (d, *J* = 15.6 Hz, 1H), 3.21 (d, *J* = 18.4 Hz, 1H), 2.85 (d, *J* = 18.4 Hz, 1H), 2.37 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 202.1, 171.0, 147.3, 145.4, 138.3, 137.1, 134.7, 133.7, 133.1, 130.3, 130.0, 129.5, 129.0, 128.8, 128.6, 128.5, 128.4, 127.9, 127.2, 126.4, 126.3, 126.1, 124.9, 55.5, 49.8, 48.4, 21.6.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{28}H_{21}NNaO_4S$ 490.1083; Found 490.1085.

IR (film): 2924, 2258, 1720, 1696, 1627, 1599, 1452, 1359, 1173, 1116, 1088, 909, 753, 667, 546 cm^{-1}.

2'-Tosyl-1',2'-dihydro-3'*H*-spiro[cyclopenta[*a*]naphthalene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5ma)



Off-White solid; R_f = 0.15 (PE/EA = 6:1); Yield: 20.3 mg, 43 %; M.p. 119 – 121 °C;

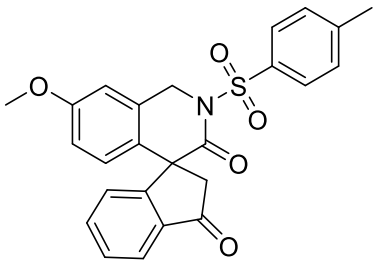
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.93 (m, 2H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 8.8 Hz, 1H), 7.57 – 7.53 (m, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.33 – 7.26 (m, 3H), 7.24 – 7.22 (m, 1H), 7.05 – 6.99 (m, 2H), 6.21 (d, *J* = 7.6 Hz, 1H), 5.69 (d, *J* = 15.6 Hz, 1H), 4.97 (d, *J* = 15.6 Hz, 1H), 3.04 (d, *J* = 18.0 Hz, 1H), 2.72 (d, *J* = 18.4 Hz, 1H), 2.41 (s, 3H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.6, 171.0, 153.6, 145.4, 138.1, 137.7, 136.4, 134.7, 131.3, 129.9, 129.5, 129.2, 129.1, 129.0, 128.9, 128.6, 128.0, 127.3, 126.3, 126.2, 125.9, 118.8, 55.3, 49.7, 47.9, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{28}H_{21}NNaO_4S$ 490.1083; Found 490.1084.

IR (film): 2924, 2254, 1713, 1595, 1460, 1360, 1188, 1172, 1114, 1076, 1015, 908, 815, 727, 665, 552 cm^{-1}.

7'-Methoxy-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5n)



Off-White solid; R_f = 0.19 (PE/EA = 3:1); Yield: 31 mg, 69 %; M.p. 118 – 120 °C;

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.79(d, *J* = 7.6 Hz, 1H), 7.64 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.50 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.30 – 7.26 (m, 3H), 6.89 (d, *J* = 2.4 Hz, 1H), 6.72 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 6.45 (d, *J* = 8.8 Hz, 1H), 5.46 (d, *J* = 16.0 Hz,

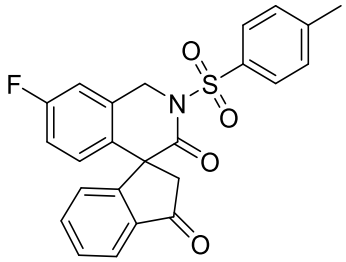
1H), 4.95 (d, *J* = 15.6 Hz, 1H), 3.81 (s, 3H), 3.02 (d, *J* = 18.0 Hz, 1H), 2.66 (d, *J* = 18.4 Hz, 1H), 2.41 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 202.2, 177.1, 159.1, 154.1, 145.3, 136.8, 135.4, 134.8, 131.6, 129.9, 129.6, 129.3, 128.5, 127.4, 127.2, 123.7, 114.4, 111.4, 55.5, 55.2, 49.3, 48.2, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{21}NNaO_5S$ 470.1033; Found 470.1033.

IR (film): 3071, 2935, 1718, 1604, 1597,1505, 1463, 1358, 1288, 1171, 1118, 1088, 813, 664, 553 cm^{-1}.

7'-Fluoro-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5o)



Off-White solid; R_f = 0.24 (PE/EA = 6:1); Yield: 30.2 mg, 69 %; M.p. 257 – 259 °C;

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.80 (d, *J* = 7.6 Hz, 1H), 7.67 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.53 (td, *J* = 7.6 Hz, 0.8 Hz, 1H), 7.31 – 7.26 (m, 3H), 7.11 (dd, *J* = 8.4 Hz, 2.8 Hz, 1H), 6.89 (td, *J* = 8.4 Hz, 2.8 Hz, 1H), 6.52 (dd, *J* = 7.2 Hz, 5.2 Hz, 1H), 5.48 (d, *J* = 15.6 Hz, 1H), 4.95 (d, *J* = 15.6 Hz, 1H), 3.02 (d, *J* = 18.4 Hz, 1H), 2.65 (d, *J* = 18.4 Hz, 1H), 2.42 (s, 3H).

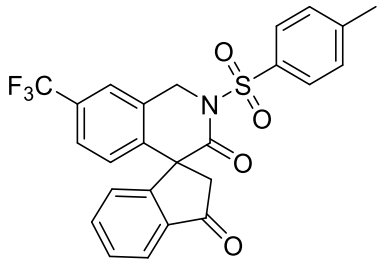
^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 201.6, 170.5, 161.9 (d, C-F, $^{1}J_{C\text{-}F}$ = 247.7 Hz), 153.6, 145.5, 136.9, 135.6, 134.6, 134.0 (d, C-F, $^{4}J_{C\text{-}F}$ = 3.2 Hz), 132.4 (d, C-F, $^{3}J_{C\text{-}F}$ = 8.1 Hz), 129.6, 129.5, 127.9 (d, C-F, $^{3}J_{C\text{-}F}$ = 8.3 Hz), 127.4, 123.8, 115.9 (d, C-F, $^{2}J_{C\text{-}F}$ = 21.4 Hz), 113.4 (d, C-F, $^{2}J_{C\text{-}F}$ = 22.7 Hz), 55.3, 49.1, 47.8 (d, *J* = 2.2 Hz), 21.7.

^{19}F NMR (376 MHz, CDCl$_3$) δ - 113.05 ~ - 113.06 (m).

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{18}FNNaO_4S$ 458.0833; Found 474. 458.0834.

IR (film): 2929, 2258, 1719, 1596, 1500, 1463, 1359, 1276, 1171, 1118, 1088, 734, 663, 551 cm^{-1}.

2'-Tosyl-7'-(trifluoromethyl)-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5p)



Off-White solid; R_f = 0.25 (PE/EA = 6:1); Yield: 34 mg, 70 %; M.p. 120 – 122 °C;

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.4 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 1H), 7.70 – 7.66 (m, 2H), 7.55 (td, *J* = 7.6 Hz, 0.8 Hz, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.28 – 7.25 (m, 1H), 6.69 (d, *J* = 8.4 Hz, 1H), 5.59 (d, *J* = 16.0 Hz, 1H), 5.01 (d, *J* = 16.0 Hz, 1H), 3.07 (d, *J* = 18.0 Hz, 1H), 2.67 (d, *J* = 18.0 Hz, 1H), 2.42 (s, 3H).

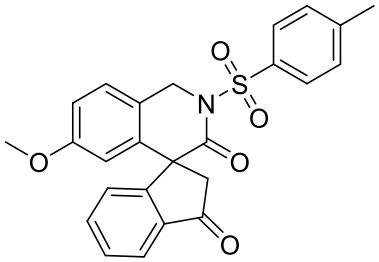
^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.1, 170.0, 153.0, 145.7, 142.2, 136.9, 135.9, 134.5, 131.3, 130.5 (q, CF_3, $^2J_{C-F}$ = 32.9 Hz), 129.7, 129.6, 128.6, 127.3, 126.7, 125.7 (q, CF_3, $^3J_{C-F}$ = 3.9 Hz), 124.8 (q, CF_3, $^1J_{C-F}$ = 270.9 Hz), 124.0, 123.5 (q, CF_3, $^3J_{C-F}$ = 3.8 Hz), 55.6, 48.8, 47.8, 21.7.

^{19}F NMR (376 MHz, $CDCl_3$) δ - 62.738 (s, 3F).

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{18}F_3NNaO_4S$ 508.0801; Found 508.0803.

IR (film): 3071, 2925, 2258, 1720, 1622, 1598, 1463, 1334, 1172, 1124, 1079, 910, 732, 671, 553 cm^{-1}.

6'-Methoxy-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5q)



Off-White solid; R_f = 0.21 (PE/EA = 6:1); Yield: 33.2 mg, 74 %; M.p. 107 – 109 °C;

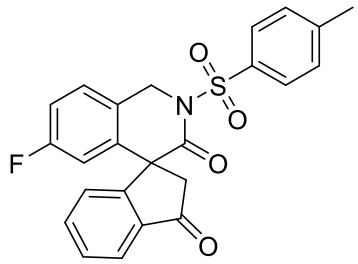
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.66 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.31 and 7.29 (2d, *J* = 7.2 Hz, 4H), 6.84 (dd, *J* = 8.8 Hz, 2.4 Hz,1H), 6.02 (d, *J* = 2.4 Hz, 1H), 5.46 (d, *J* = 15.6 Hz, 1H), 4.90 (d, *J* = 15.2 Hz, 1H), 3.63 (s, 3H), 3.00 (d, *J* = 18.4 Hz, 1H), 2.69 (d, *J* = 18.4 Hz, 1H), 2.41 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 201.8, 170.8, 159.7, 153.4, 145.3, 139.7, 137.0, 135.5, 134.9, 129.6, 129.4, 128.5, 127.7, 123.7, 122.6, 112.9, 112.1, 55.9, 55.3, 48.8, 47.7, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{21}NNaO_5S$ 470.1033; Found 470.1033.

IR (film): 3058, 2931, 1719, 1614, 1596, 1463, 1357, 1286, 1171, 1114, 1055, 814, 765, 664, 545 cm^{-1}.

6'-Fluoro-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5r)



Off-White solid; R_f = 0.23 (PE/EA = 6:1); Yield: 29.3 mg, 67 %; M.p. 113 – 115 °C;

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.81 (d, *J* = 7.6 Hz, 1H), 7.69 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.54 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.38 (dd, *J* = 8.8 Hz, 5.6 Hz, 1H), 7.31 – 7.28 (m, 3H), 7.03 (td, *J* = 8.4 Hz, 2.4 Hz, 1H), 6.24 (dd, *J* = 9.2 Hz, 2.4 Hz, 1H), 5.50 (d, *J* = 15.6 Hz, 1H), 4.91 (d, *J* = 15.6 Hz, 1H), 3.01 (d, *J* = 18.0 Hz, 1H), 2.67 (d, *J* = 18.4 Hz, 1H), 2.42 (s, 3H).

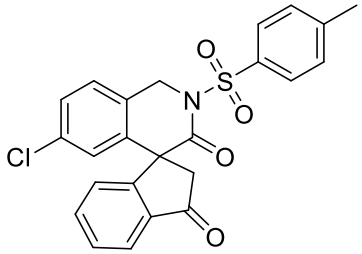
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 201.2, 170.3, 162.5 (d, C-F, $^{1}J_{C\text{-}F}$ = 247.2 Hz), 152.9, 145.5, 140.7 (d, C-F, $^{3}J_{C\text{-}F}$ = 7.3 Hz), 136.9, 135.7, 134.6, 129.7, 129.6, 128.6, 128.3 (d, C-F, $^{3}J_{C\text{-}F}$ = 8.5 Hz), 127.5, 126.4 (d, C-F, $^{4}J_{C\text{-}F}$ = 3.2 Hz), 123.9, 115.2 (d, C-F, $^{2}J_{C\text{-}F}$ = 22.1 Hz), 113.1 (d, C-F, $^{2}J_{C\text{-}F}$ = 23.7 Hz), 55.7 (d, *J* = 1.6 Hz), 48.6, 47.6, 21.7.

^{19}F NMR (376 MHz, $CDCl_3$) δ - 111.17 ~ - 111.23 (m).

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{18}FNNaO_4S$ 458.0833; Found 458.0834.

IR (film): 3067, 2929, 2262, 1719, 1622, 1598, 1463, 1359, 1171, 1117, 911, 767, 663, 573 cm^{-1}.

6'-Chloro-2'-tosyl-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5s)



Off-White solid; R_f = 0.23 (PE/EA = 6:1); Yield: 28 mg, 62 %; M.p. 117 – 119 °C;

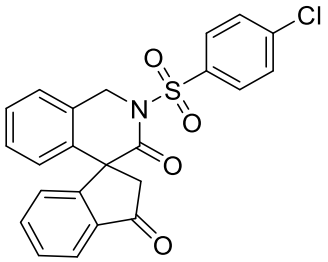
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.69 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.55 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.34 (d, *J* = 8.4 Hz, 1H), 7.32 – 7.27 (m, 4H), 6.51 (d, *J* = 2.0 Hz, 1H), 5.49 (d, *J* = 15.6 Hz, 1H), 4.92 (d, *J* = 15.6 Hz, 1H), 3.01 (d, *J* = 18.4 Hz, 1H), 2.66 (d, *J* = 18.8 Hz, 1H), 2.42 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 201.2, 170.2, 152.9, 145.6, 140.1, 136.9, 135.8, 134.8, 134.6, 129.7, 129.6, 128.9, 128.6, 128.3, 127.8, 127.4, 126.0, 123.9, 55.6, 48.7, 47.6, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{18}ClNNaO_4S$ 474.0537; Found 474.0537.

IR (film): 2925, 1719, 1597, 1463, 1360, 1288, 1171, 1118, 1088, 1019, 815, 681, 567, 544 cm^{-1}.

2'-((4-Chlorophenyl)sulfonyl)-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5t)



Off-White solid; R_f = 0.28 (PE/EA = 6:1); Yield: 33 mg, 75 %; M.p. 111 – 113 °C;

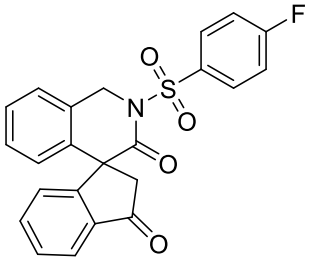
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.8 Hz, 2H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.66 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.53 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.47 (d, *J* = 8.8 Hz, 2H), 7.39 (d, *J* = 7.2 Hz, 1H), 7.33 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.28 – 7.25 (m, 1H), 7.20 (t, *J* = 7.6 Hz, 1H), 6.56 (d, *J* = 8.0 Hz, 1H), 5.49 (d, *J* = 15.6 Hz, 1H), 5.00 (d, *J* = 15.6 Hz, 1H), 3.10 (d, *J* = 18.0 Hz, 1H), 2.72 (d, *J* = 18.4 Hz, 1H).

^{13}C{^{1}H} **NMR (100 MHz, $CDCl_3$)** δ 201.6, 171.0, 153.5, 140.9, 137.9, 136.9, 136.2, 135.5, 130.1, 129.5, 129.3, 128.9, 128.1, 127.4, 126.4, 125.9, 123.9, 55.7, 49.0, 48.3.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{23}H_{16}ClNNaO_4S$ 460.0381; Found 460.0384.

IR (film): 3094, 2929, 2262, 1718, 1596, 1586, 1475, 1363, 1287, 1182, 1172, 1091, 1013, 757, 625, 549 cm^{-1}.

2'-((4-Fluorophenyl)sulfonyl)-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5u)



Off-White solid; R_f = 0.19 (PE/EA = 6:1); Yield: 30 mg, 71 %; M.p. 123 – 125 °C;

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 – 8.05 (m, 2H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.66 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.53 (td, *J* = 7.2 Hz, 0.8 Hz, 1H), 7.39 (d, *J* = 7.2 Hz, 1H), 7.33 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.27 – 7.25 (m, 1H), 7.22 – 7.15 (m, 3H), 6.57 (d, *J* = 7.6 Hz, 1H), 5.49

(d, *J* = 16.0 Hz, 1H), 4.99 (d, *J* = 16.0 Hz, 1H), 3.09 (d, *J* = 18.4 Hz, 1H), 2.72 (d, *J* = 18.0 Hz, 1H).

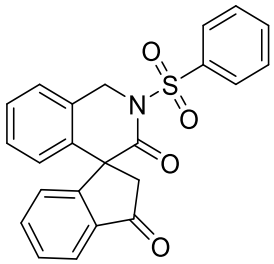
^{13}C{^{1}H} NMR (100 MHz, CDCl$_3$) δ 201.7, 171.0, 165.9 (d, C-F, $^{1}J_{C\text{-}F}$ = 255.9 Hz), 153.6, 137.9, 136.9, 135.5, 133.7 (d, C-F, $^{4}J_{C\text{-}F}$ = 3.1 Hz), 131.7 (d, C-F, $^{3}J_{C\text{-}F}$ = 9.8 Hz), 130.1, 129.5, 128.9, 128.1, 127.4, 126.4, 125.9, 123.9, 116.2 (d, C-F, $^{2}J_{C\text{-}F}$ = 22.6 Hz), 55.7, 49.1, 48.2.

^{19}F NMR (376 MHz, CDCl$_3$) δ - 101.95 ~ - 101.99 (m).

HRMS (ESI) m/z: [M + Na]$^+$ Calcd for $C_{23}H_{16}FNNaO_4S$ 444.0676; Found 444.0676.

IR (film): 3071, 2929, 2256, 1718, 1590, 1492, 1463, 1363, 1179, 1157, 1087, 838, 733, 671, 543 cm^{-1}.

2'-(Phenylsulfonyl)-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5v)



Off-White solid; R_f = 0.18 (PE/EA = 6:1); Yield: 36 mg, 89 %; M.p. 140 – 142 °C;

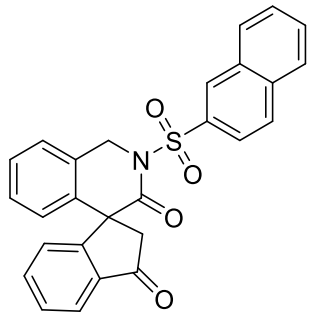
^{1}H NMR (600 MHz, CDCl$_3$) δ 8.03 (d, *J* = 7.2 Hz, 2H), 7.80 (d, *J* = 7.8 Hz, 1H), 7.66 – 7.61 (m, 2H), 7.53 – 7.49 (m, 3H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.33 (t, *J* = 7.8 Hz, 1H), 7.27 – 7.25 (m, 1H), 7.19 (t, *J* = 7.8 Hz, 1H), 6.55 (d, *J* = 7.8 Hz, 1H), 5.52 (d, *J* = 15.6 Hz, 1H), 5.01 (d, *J* = 15.6 Hz, 1H), 3.05 (d, *J* = 18.0 Hz, 1H), 2.70 (d, *J* = 18.6 Hz, 1H).

^{13}C{^{1}H} NMR (150 MHz, CDCl$_3$) δ 201.8, 170.9, 153.7, 138.0, 137.8, 136.9, 135.5, 134.2, 130.3, 129.4, 128.9, 128.8, 128.5, 128.1, 127.4, 126.4, 125.9, 123.8, 55.7, 49.0, 48.3.

HRMS (ESI) m/z: [M + Na]$^+$ Calcd for $C_{23}H_{17}NNaO_4S$ 426.0770; Found 426.0770.

IR (film): 3069, 2920, 2258, 1718, 1600, 1449, 1360, 1173, 1115, 1088, 911, 729, 609, 550 cm^{-1}.

2'-(Naphthalen-2-ylsulfonyl)-1',2'-dihydro-3'*H*-spiro[indene-1,4'-isoquinoline]-3,3'(2*H*)-dione (5w)



Off-White solid; R_f = 0.17 (PE/EA = 6:1); Yield: 42.2 mg, 93 %; M.p. 105 – 107 °C;

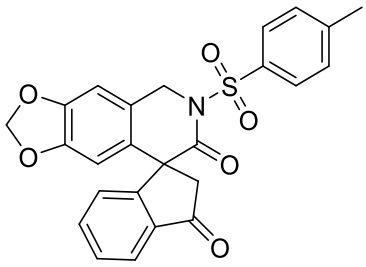
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.61 (bs, 1H), 7.96 (d, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 1.8 Hz, 2H), 7.88 (d, *J* = 8.4 Hz, 1H), 7.77 (d, *J* = 7.8 Hz, 1H), 7.65 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.62 – 7.58 (m, 2H), 7.47 (d, *J* = 7.2 Hz, 1H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.33 (td, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.26 (d, *J* = 1.8 Hz, 1H), 7.18 (td, *J* = 7.8 Hz, 1.2 Hz, 1H), 6.53 (d, *J* = 7.8 Hz, 1H), 5.59 (d, *J* = 16.2 Hz, 1H), 5.08 (d, *J* = 16.2 Hz, 1H), 3.07 (d, *J* = 18.0 Hz, 1H), 2.70 (d, *J* = 18.6 Hz, 1H).

^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 201.8, 170.9, 153.7, 138.1, 136.9, 135.4, 134.6, 131.7, 131.1, 130.3, 129.6, 129.5, 129.3, 129.2, 128.8, 128.0, 127.9, 127.7, 127.4, 126.4, 125.9, 123.7, 122.5, 55.7, 49.0, 48.4.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{27}H_{19}NNaO_4S$ 476.0927; Found 476.0927.

IR (film): 3060, 2925, 2254, 1718, 1600, 1462, 1352, 1287, 1172, 1115, 910, 733, 661, 547 cm^{-1}.

6'-Tosyl-5',6'-dihydro-7'*H*-spiro[indene-1,8'-[1,3]dioxolo[4,5-*g*]isoquinoline]-3,7'(2*H*)-dione (5x)



Off-White solid; R_f = 0.19 (PE/EA = 6:1); Yield: 31 mg, 71 %; M.p. 239 – 241 °C;

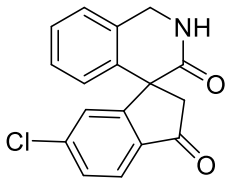
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 8.0 Hz, 1H), 7.66 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.51 (td, *J* = 7.6 Hz, 1.2 Hz, 1H), 7.30 – 7.27 (m, 3H), 6.81 (s, 1H), 5.94 (d, *J* = 1.2 Hz, 2H), 5.92 (d, *J* = 1.2 Hz, 1H), 5.38 (d, *J* = 15.6 Hz, 1H), 4.87 (d, *J* = 15.6 Hz, 1H), 2.99 (d, *J* = 18.4 Hz, 1H), 2.64 (d, *J* = 18.4 Hz, 1H), 2.42 (s, 3H).

^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ 201.9, 170.7, 153.9, 148.0, 147.5, 145.4, 136.9, 135.6, 134.7, 131.5, 129.6, 129.4, 128.5, 127.4, 123.9, 123.8, 106.5, 106.3, 101.7, 55.5, 49.1, 48.0, 21.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{25}H_{19}NNaO_6S$ 484.0825; Found 484.0830.

IR (film): 3075, 2923, 1717, 1597, 1487, 1359, 1270, 1231, 1188, 1120, 1037, 935, 766, 699, 553 cm^{-1}.

6-chloro-1',2'-dihydro-3'H-spiro[indene-1,4'-isoquinoline]-3,3'(2H)-dione (6a)



Off-White solid; R_f = 0.27 (PE/EA = 1:1); Yield: 8 mg, 25 %; M.p. 105 – 107 °C;

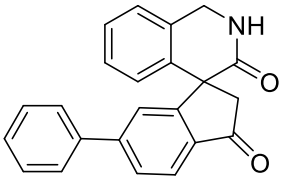
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.4 Hz, 1H), 7.45 (dd, *J* = 8.4 Hz, 1.8 Hz, 1H), 7.30 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.25 – 7.21 (m, 2H), 7.18 (d, *J* = 1.8 Hz, 1H), 6.73 (d, *J* = 7.2 Hz, 1H), 6.67 (bs, 1H), 4.77 (d, *J* = 2.4 Hz, 2H), 3.51 (d, *J* = 18.6 Hz, 1H), 2.88 (d, *J* = 18.6 Hz, 1H).

^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 172.3, 158.7, 141.8, 137.0, 135.0, 129.7, 129.5, 128.4, 127.6, 126.9, 126.3, 125.8, 124.8, 51.6, 45.1, 29.7.

HRMS (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{12}ClNNaO_2$ 320.0449; Found 320.0451.

IR (film): 3335, 2924, 2853, 1720, 1673, 1596, 1457, 1384, 1225, 1070, 958, 735 cm^{-1}.

6-phenyl-1',2'-dihydro-3'H-spiro[indene-1,4'-isoquinoline]-3,3'(2H)-dione (6b)



Off-White solid; R_f = 0.21 (PE/EA = 1:1); Yield: 10 mg, 28 %; M.p. 112 – 114 °C;

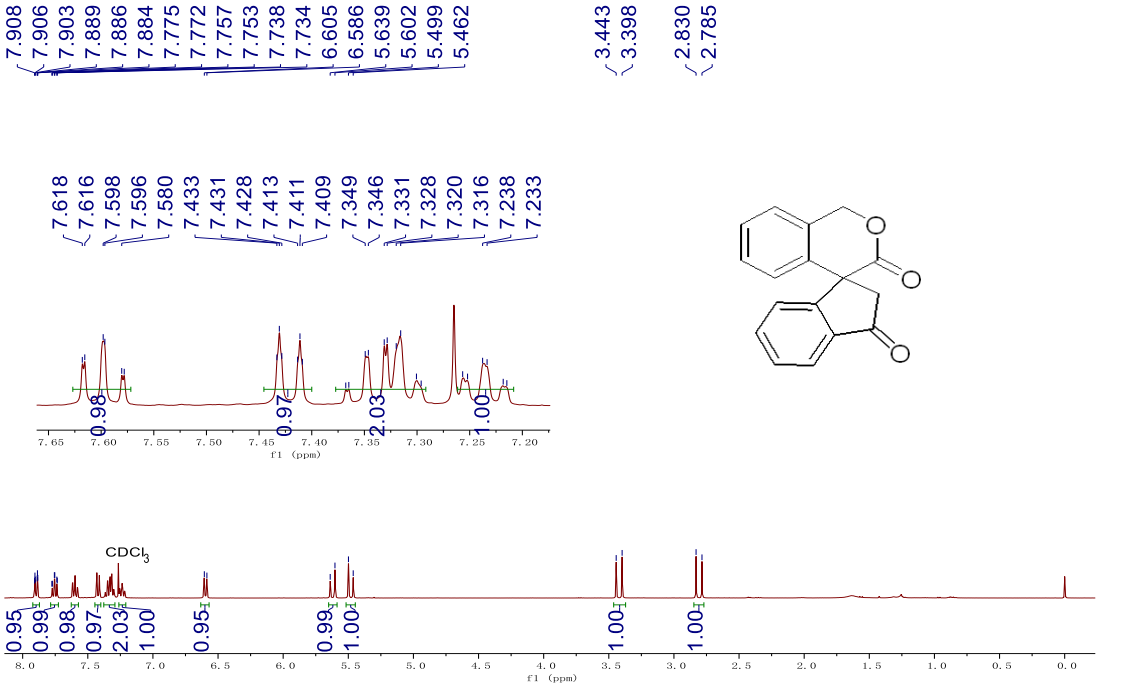
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.0 Hz, 1H), 7.70 (dd, J = 8.0 Hz, 1.6 Hz, 1H), 7.54 – 7.52 (m, 2H), 7.43 – 7.37 (m, 4H), 7.29 (d, J = 8.0 Hz, 1H), 7.24 – 7.18 (m, 2H), 6.79 (d, J = 7.6 Hz, 1H), 6.61 (bs, 1H), 4.79 (d, J = 2.0 Hz, 2H), 3.56 (d, J = 18.4 Hz, 1H), 2.92 (d, J = 18.4 Hz, 1H).

^{13}C{^{1}H} NMR (150 MHz, $CDCl_3$) δ 203.5, 172.9, 158.1, 148.6, 139.9, 137.8, 135.4, 129.6, 128.9, 128.4, 128.3, 128.2, 127.5, 127.3, 127.1, 125.7, 124.5, 124.0, 51.8, 45.2, 29.7.

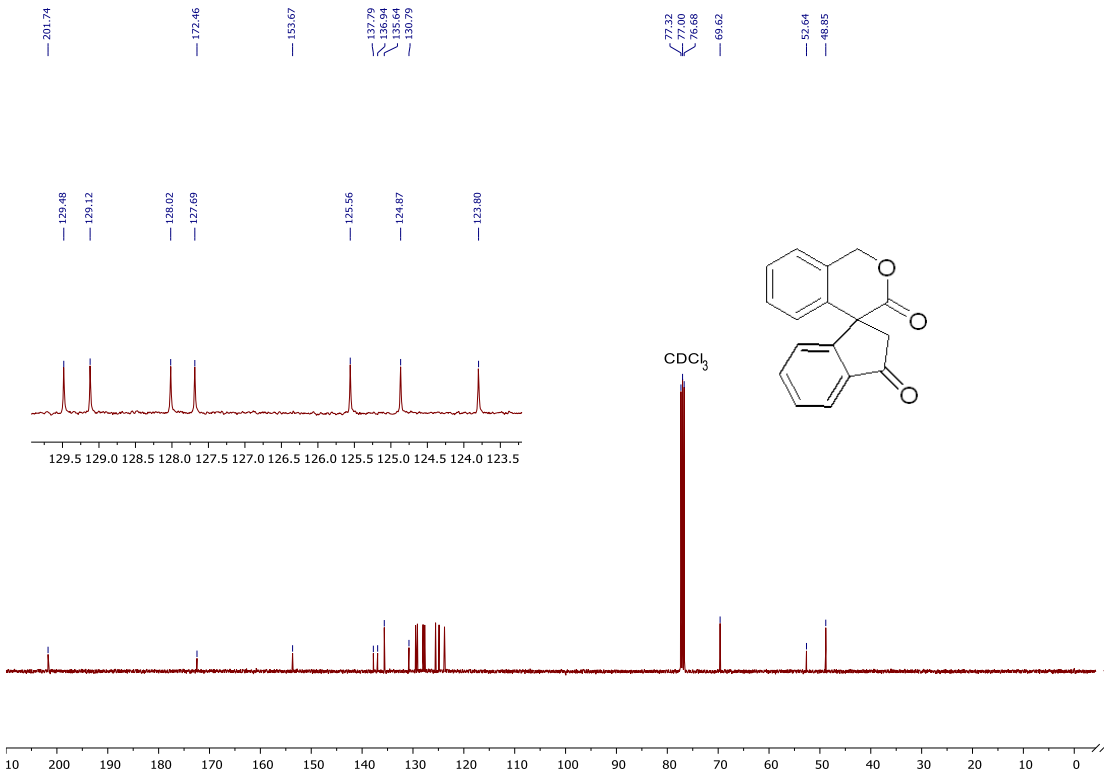
HRMS (ESI) m/z: $[M + Na]^+$ Calcd for; $C_{23}H_{17}NNaO_2$ 362.1151; Found. 362.1152.

IR (film): 3446, 2923, 2852, 1714, 1672, 1603, 1384, 1234, 1196, 1076, 763 cm^{-1}.

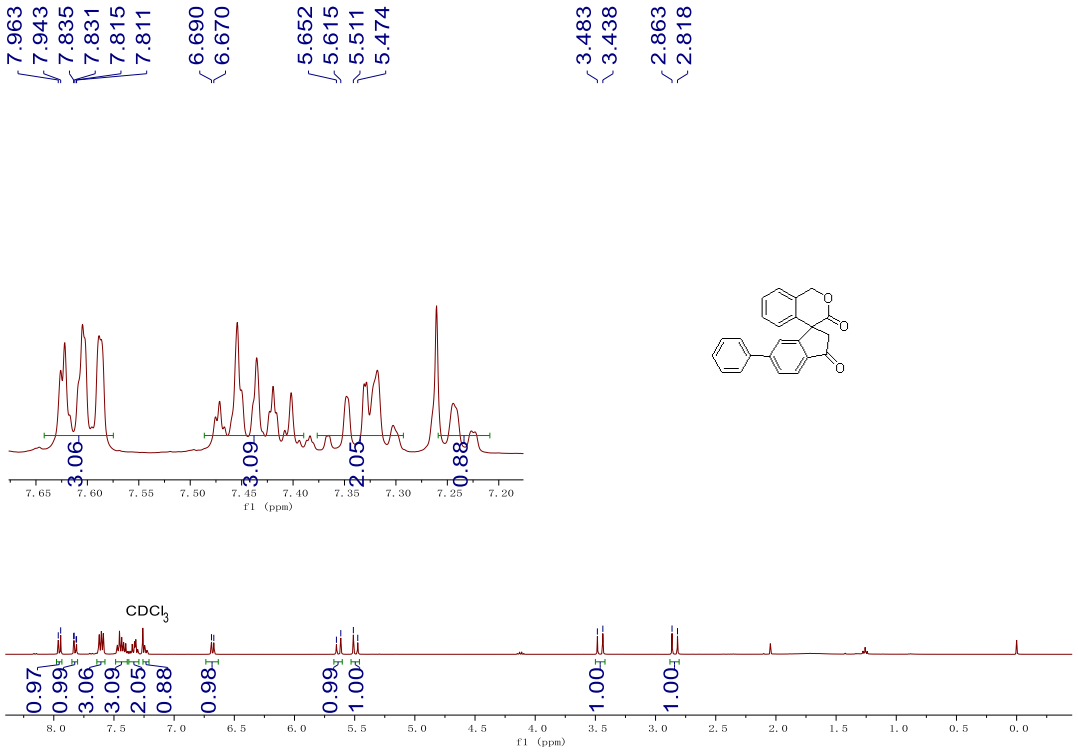
Copies of NMR Spectra



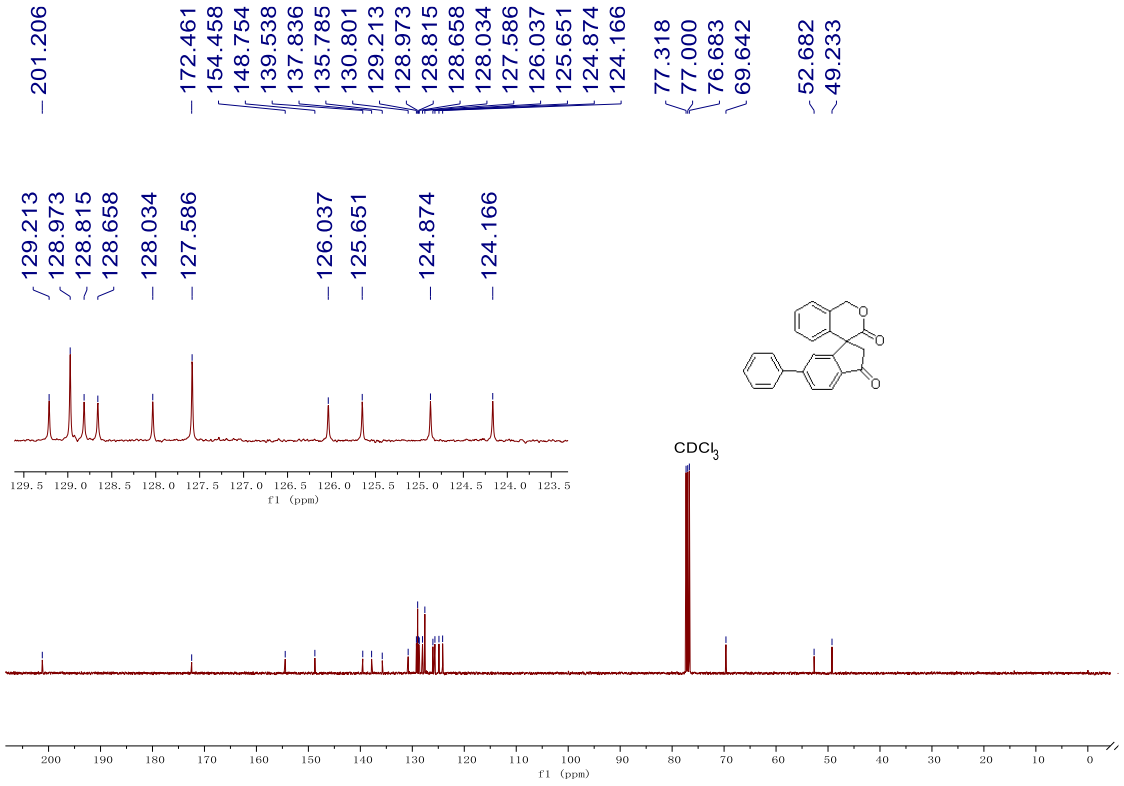
Compound **3a** ^{1}H NMR 400 MHz



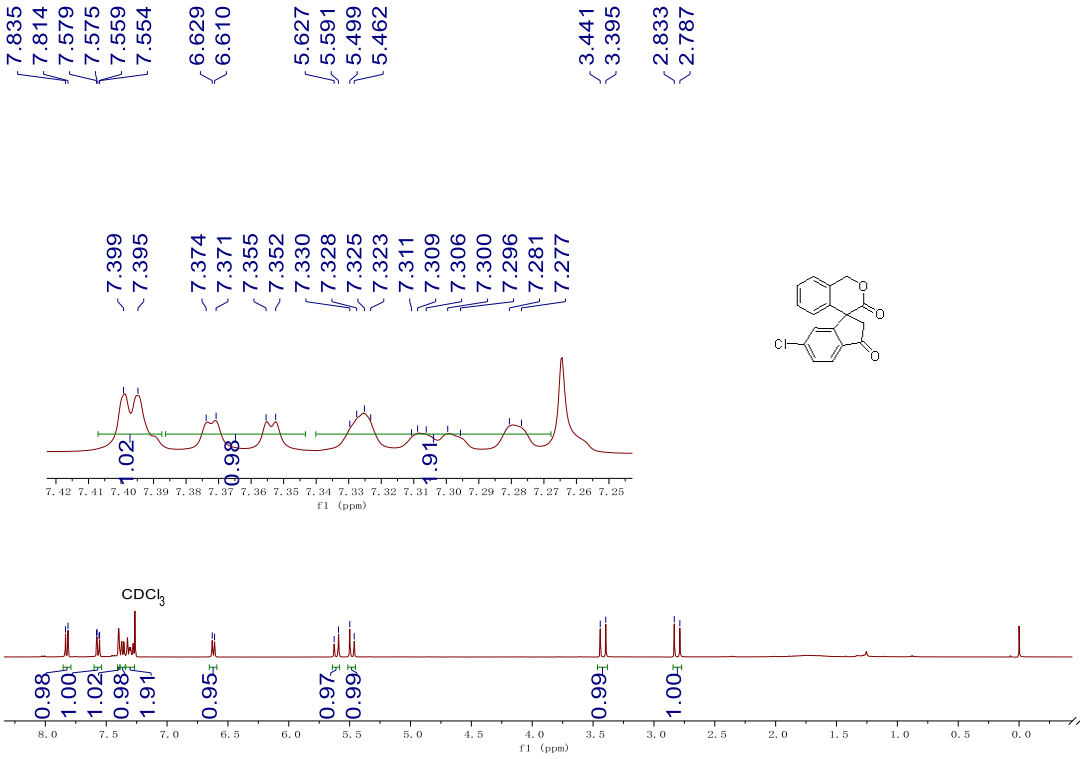
Compound **3a** ^{13}C NMR 100 MHz



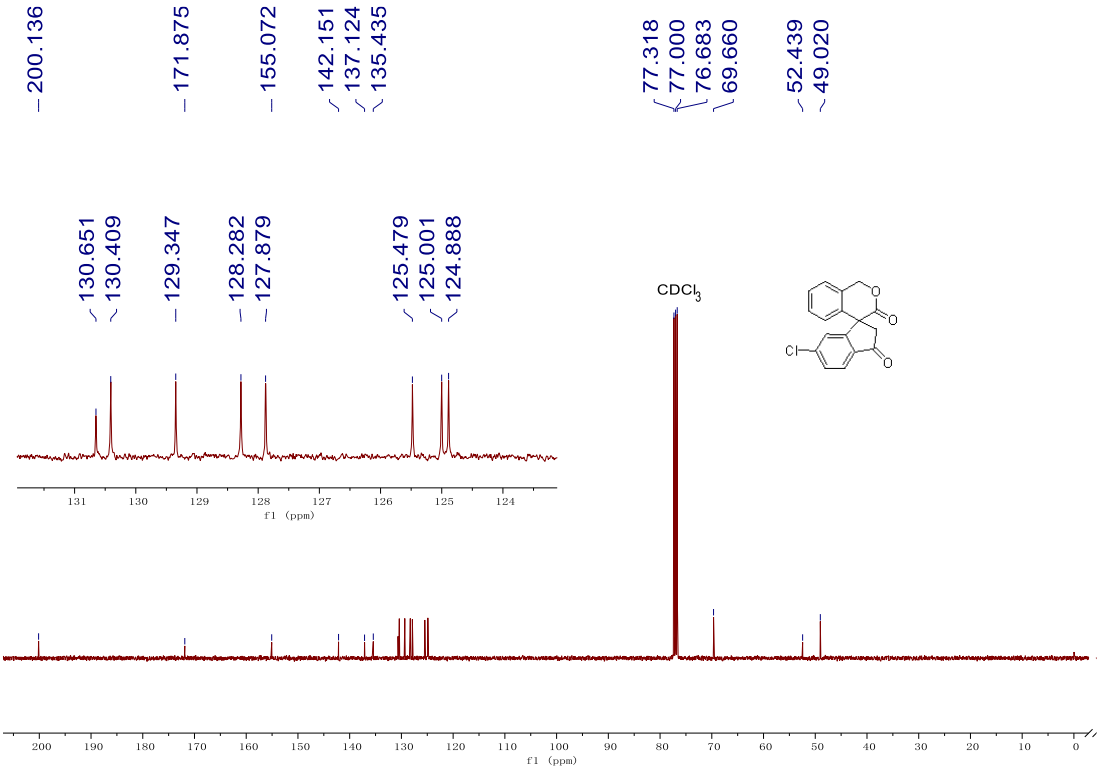
Compound **3b** ^{1}H NMR 400 MHz



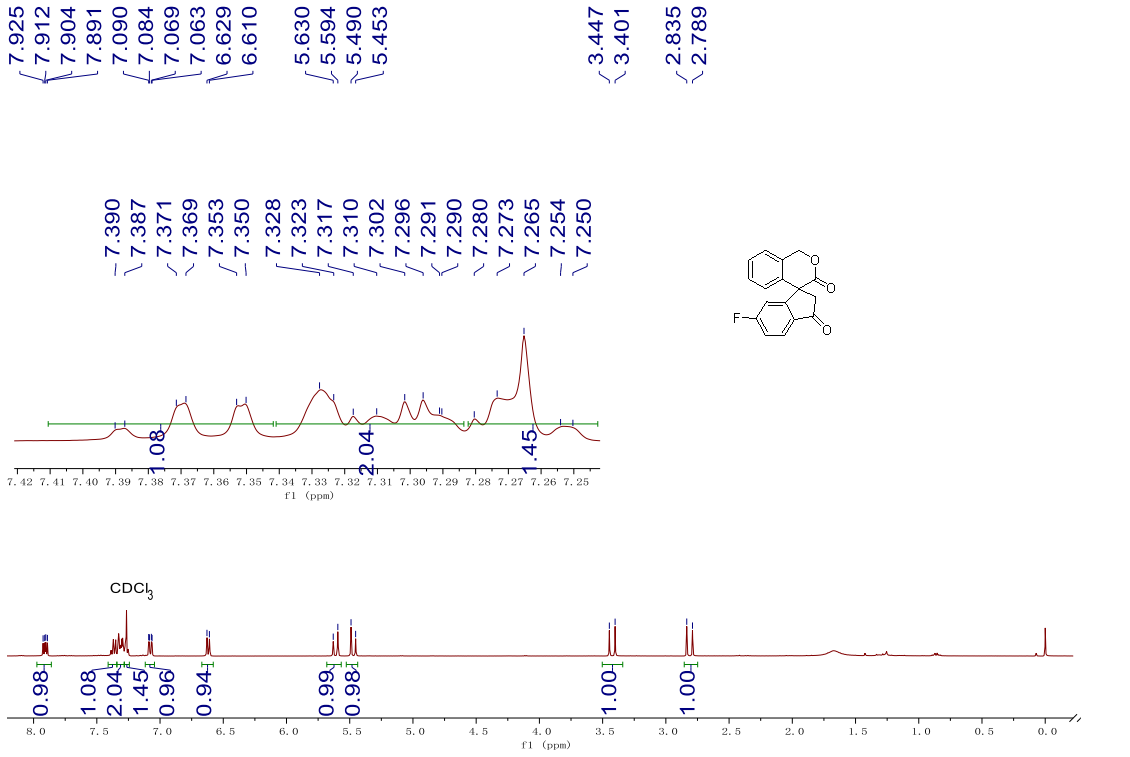
Compound **3b** ^{13}C NMR 100 MHz



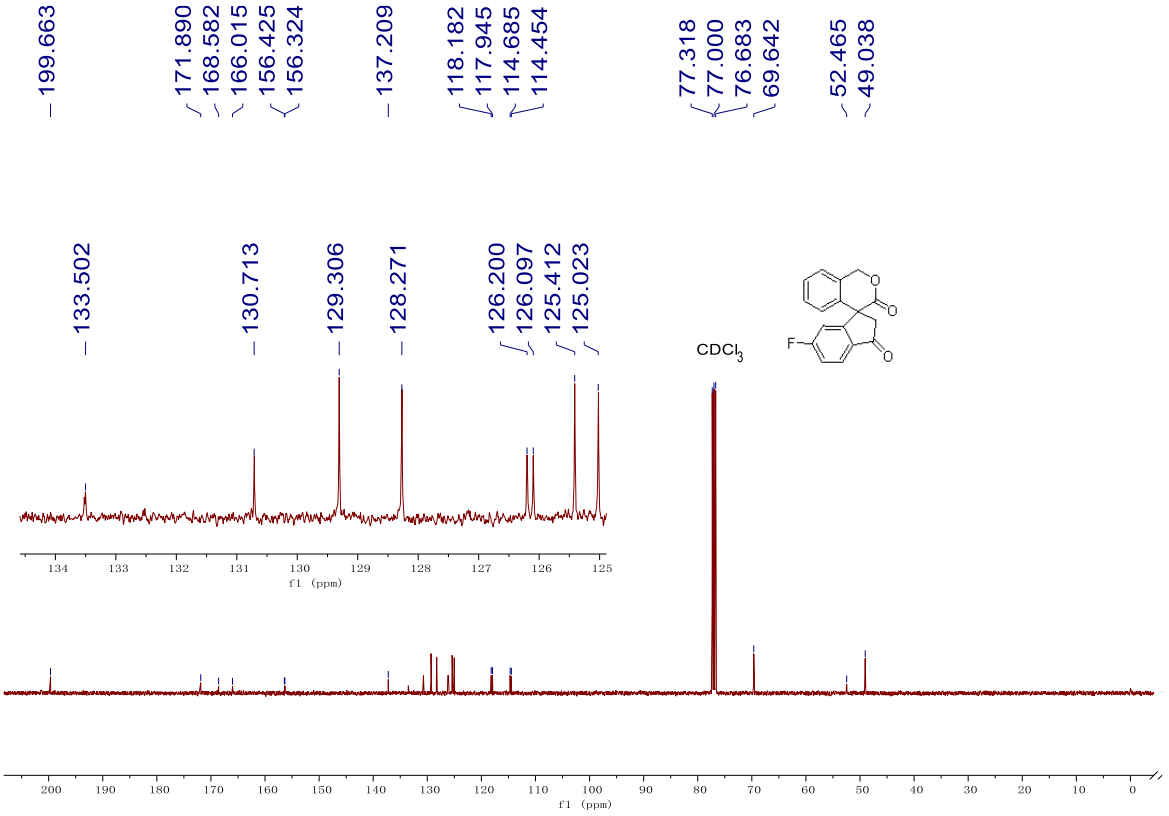
Compound **3c** ^{1}H NMR 400 MHz



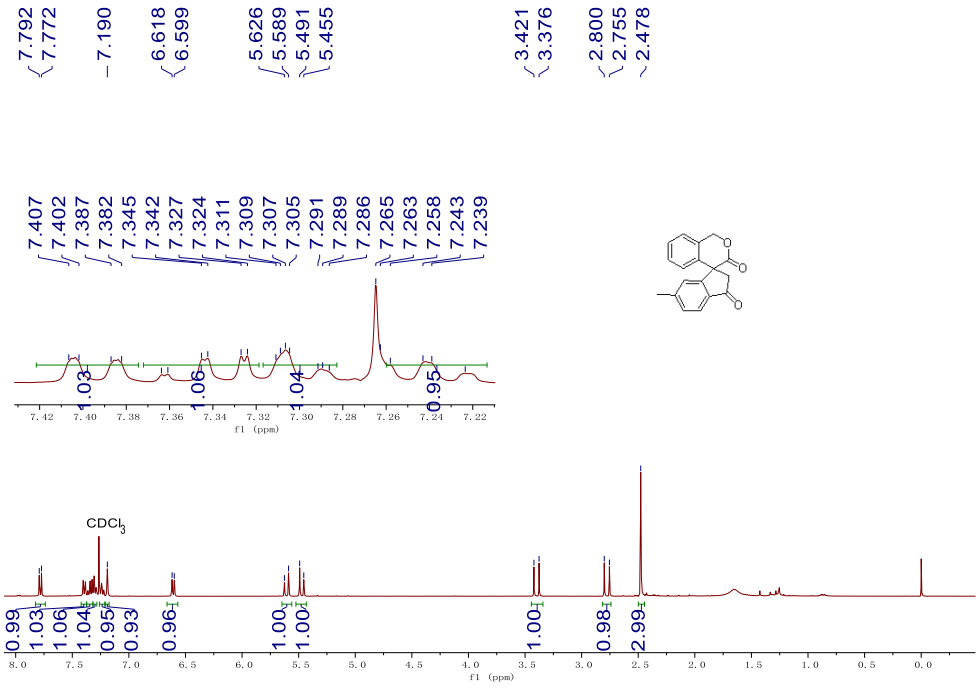
Compound **3c** ^{13}C NMR 100 MHz



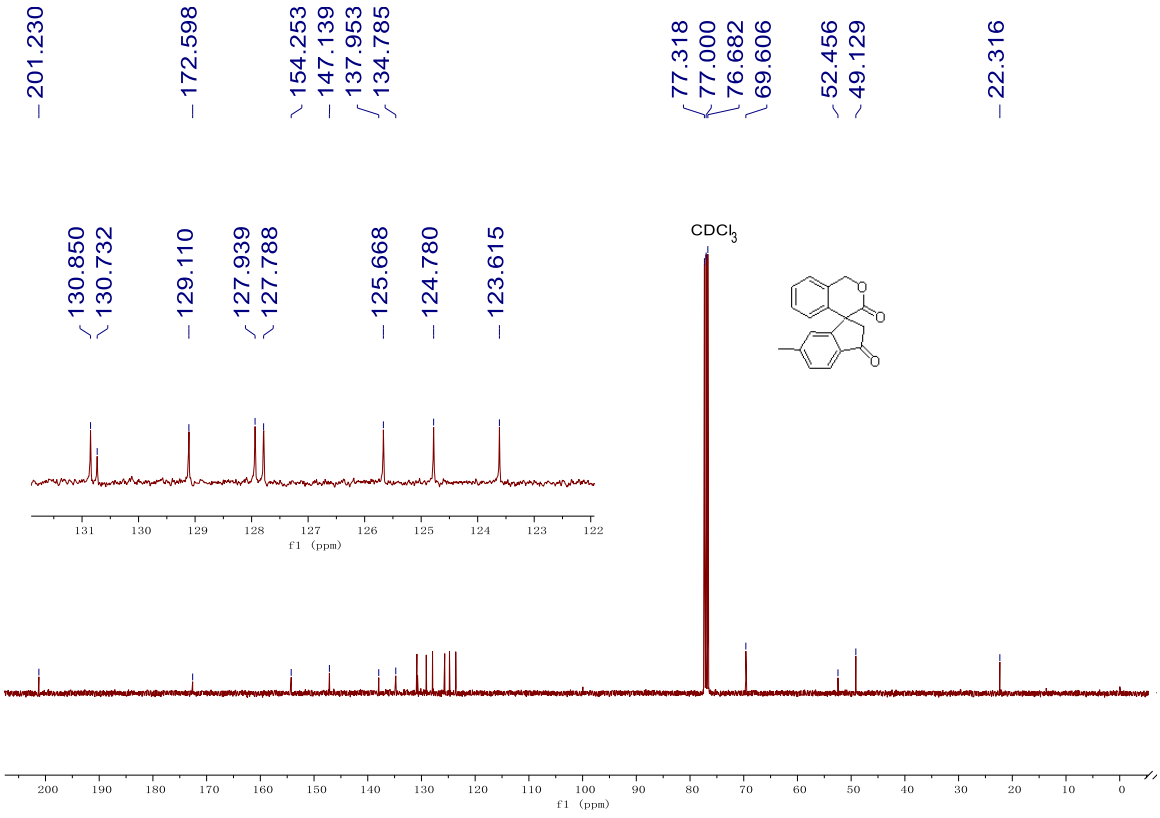
Compound **3d** ^{1}H NMR 400 MHz



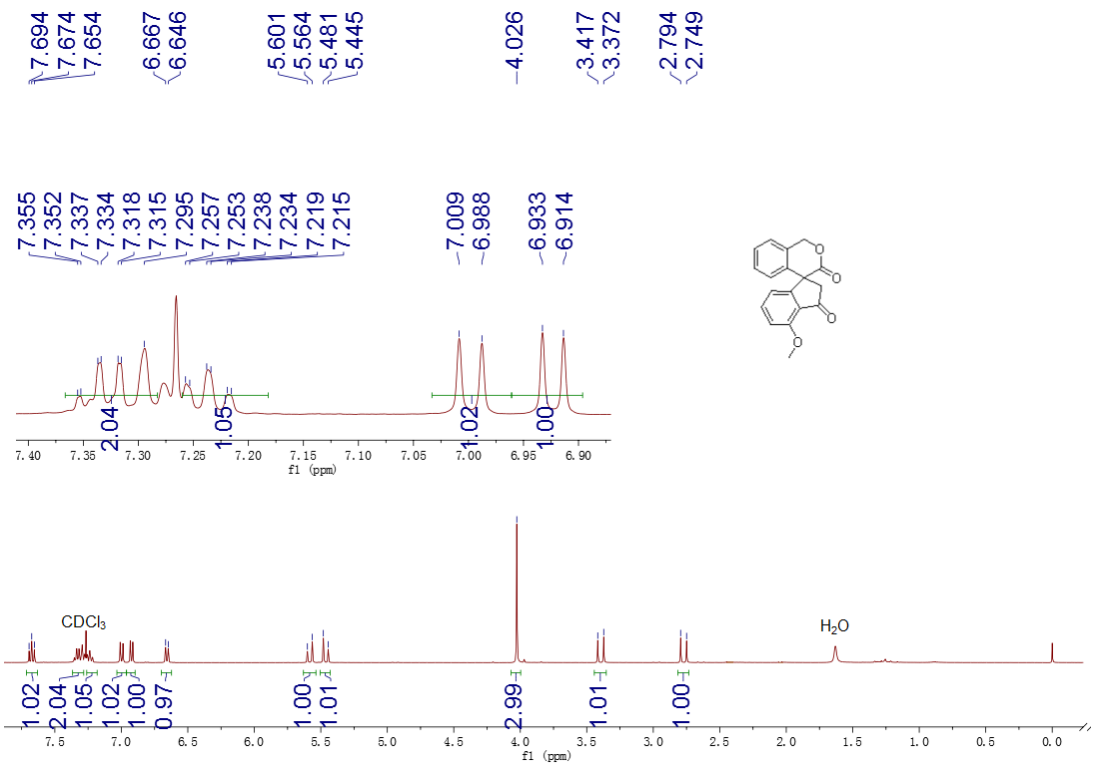
Compound **3d** ^{13}C NMR 100 MHz



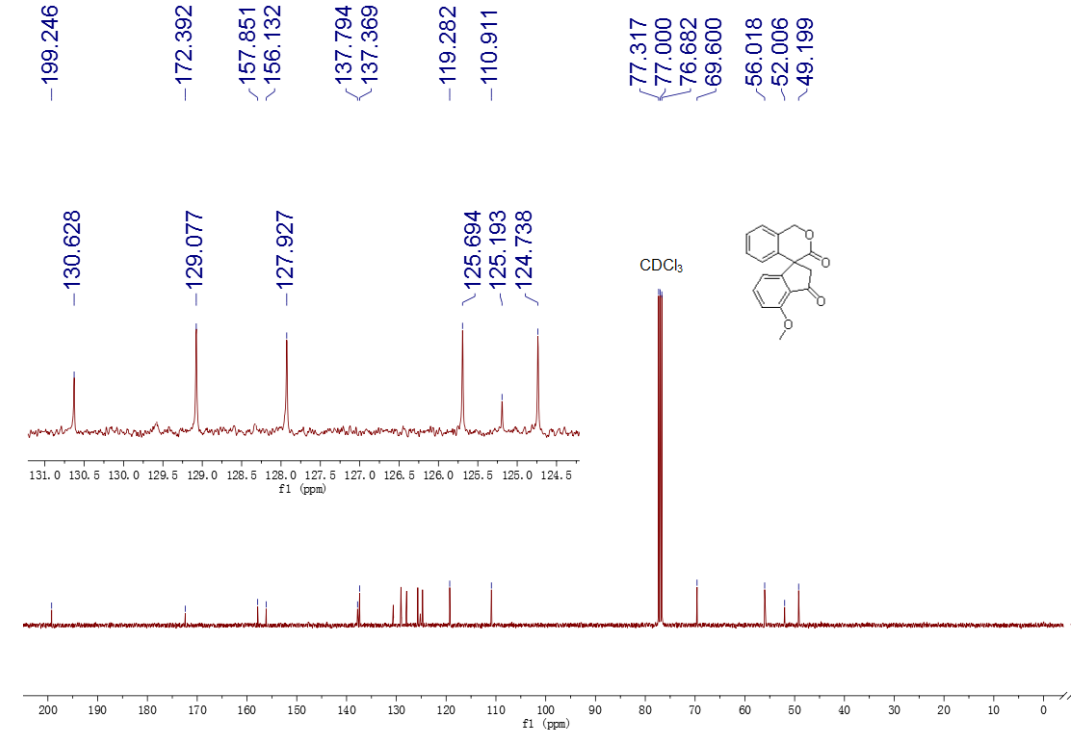
Compound **3e** ^{1}H NMR 400 MHz



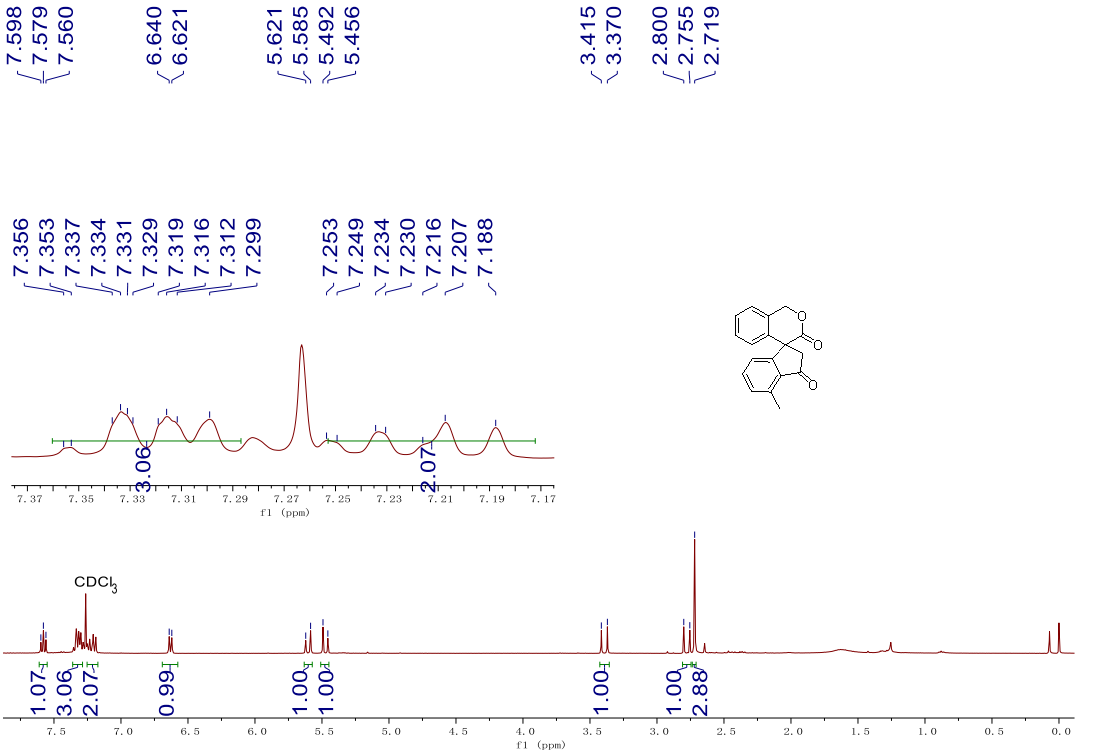
Compound **3e** ^{13}C NMR 100 MHz



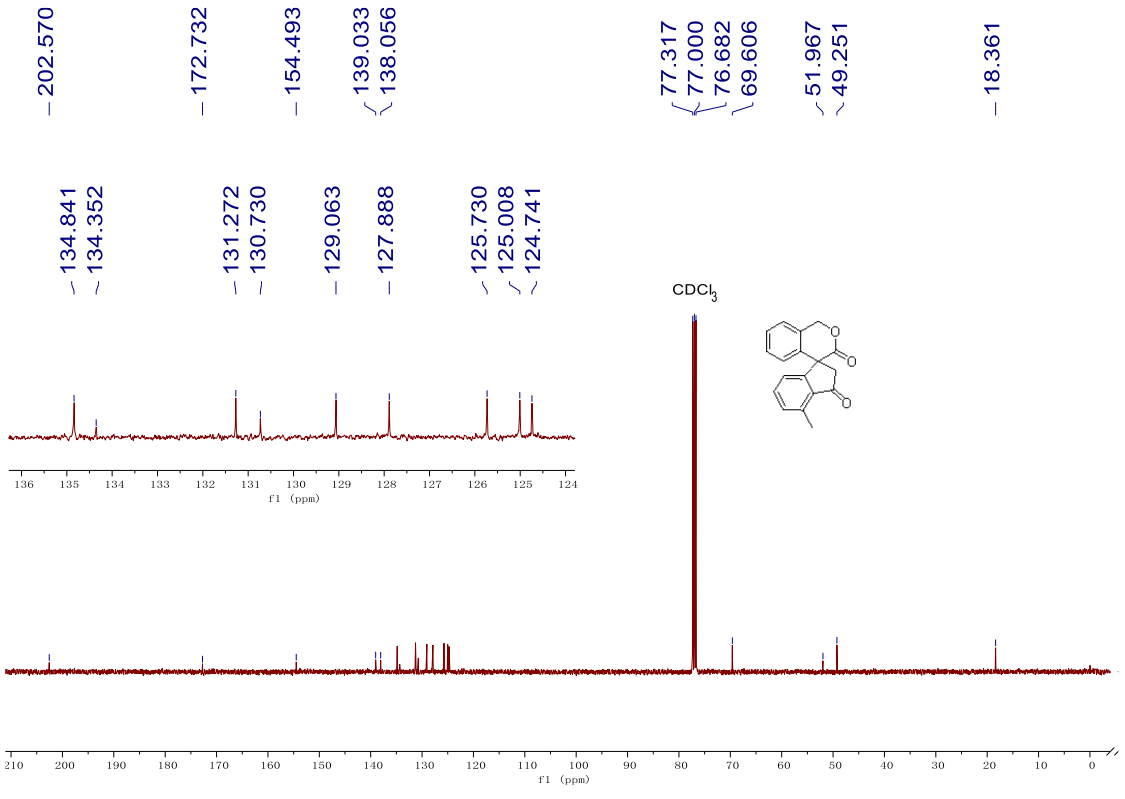
Compound **3f** ^{1}H NMR 400 MHz



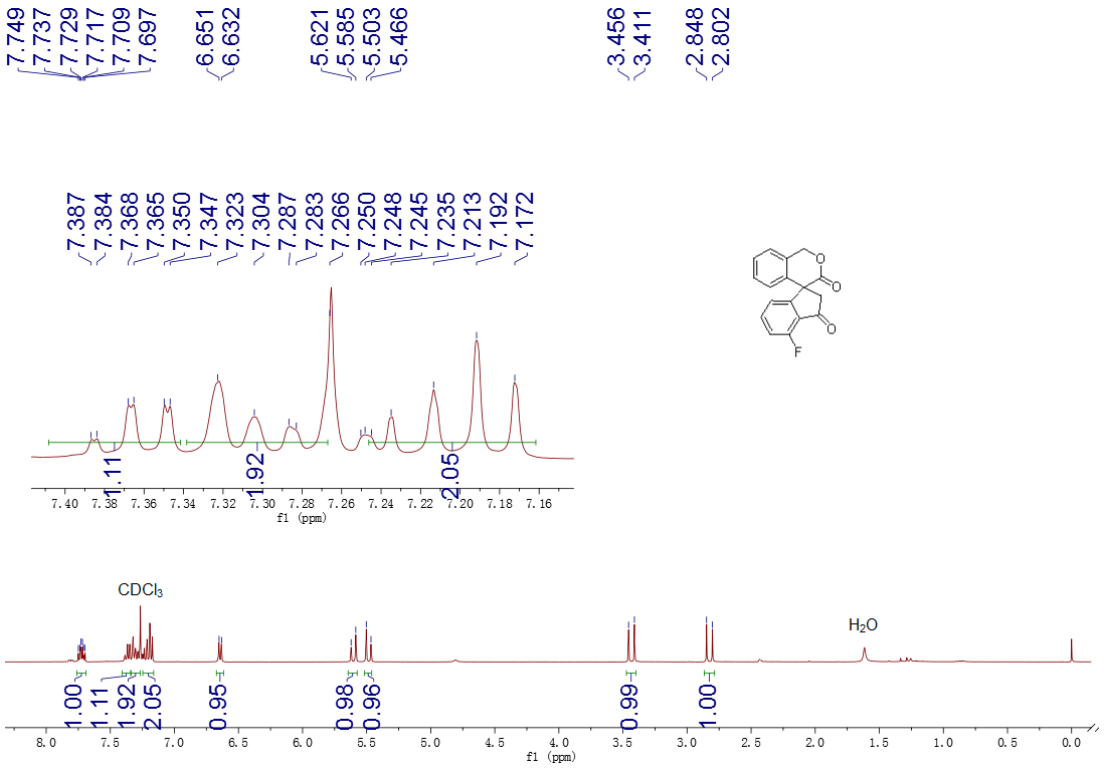
Compound **3f** ^{13}C NMR 100 MHz



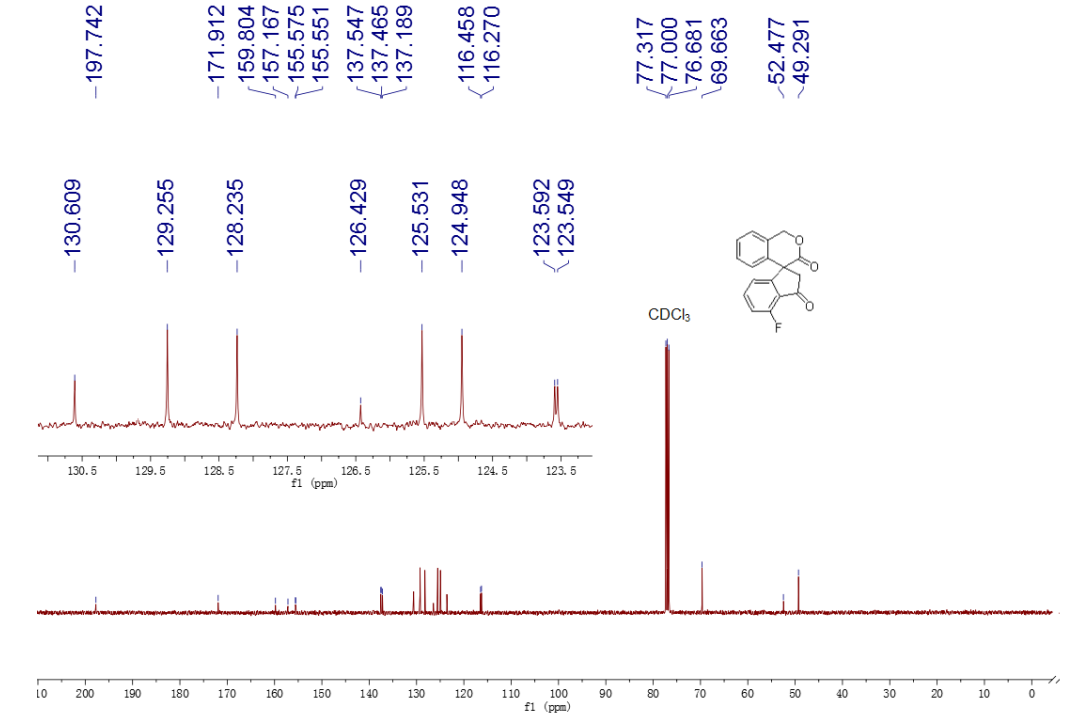
Compound **3g** ^{1}H NMR 400 MHz



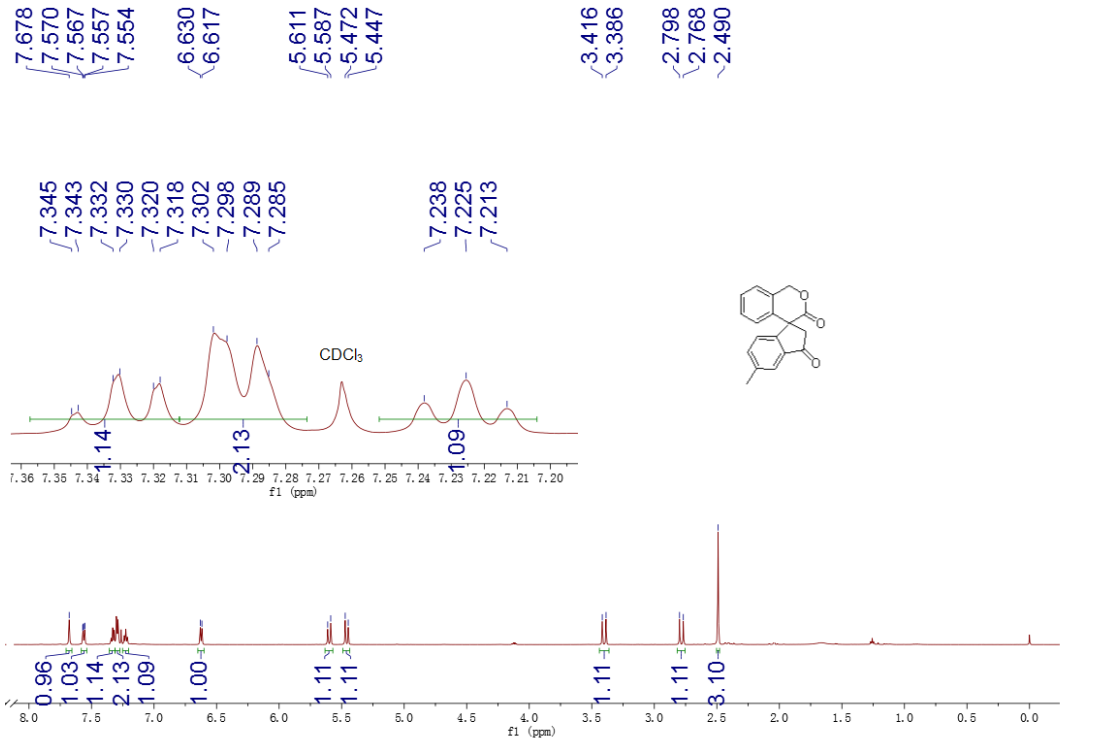
Compound **3g** ^{13}C NMR 100 MHz



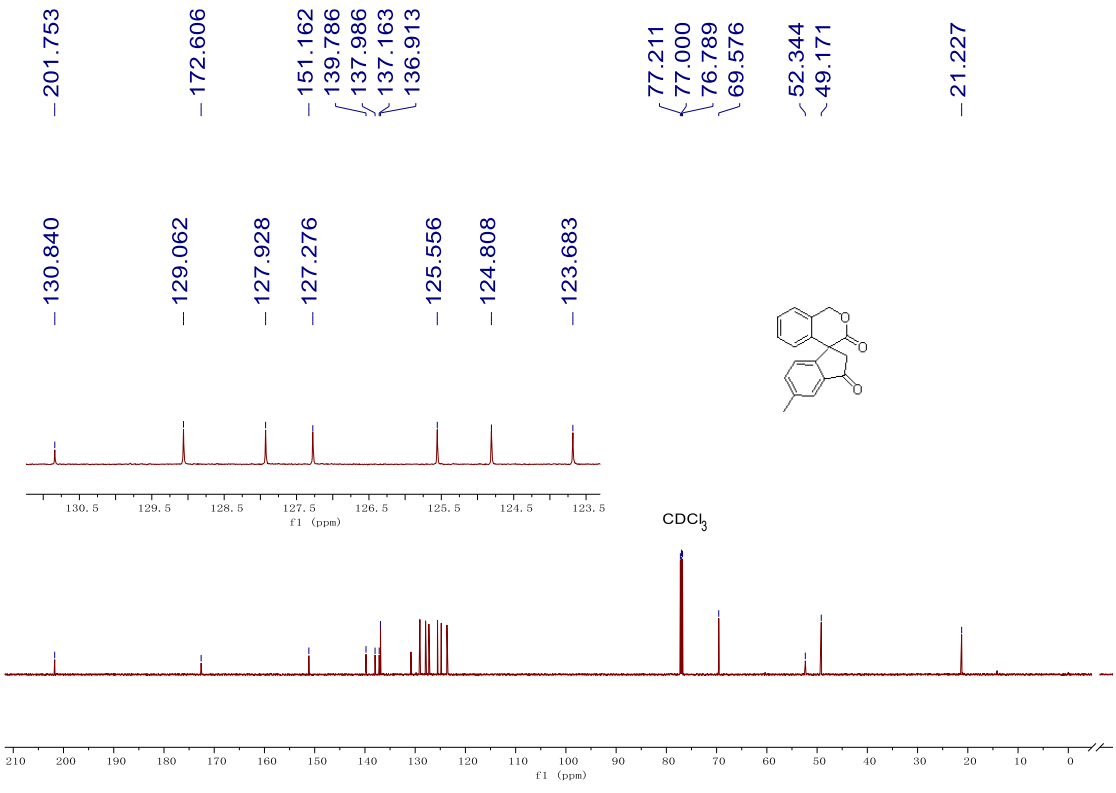
Compound **3h** ^{1}H NMR 400 MHz



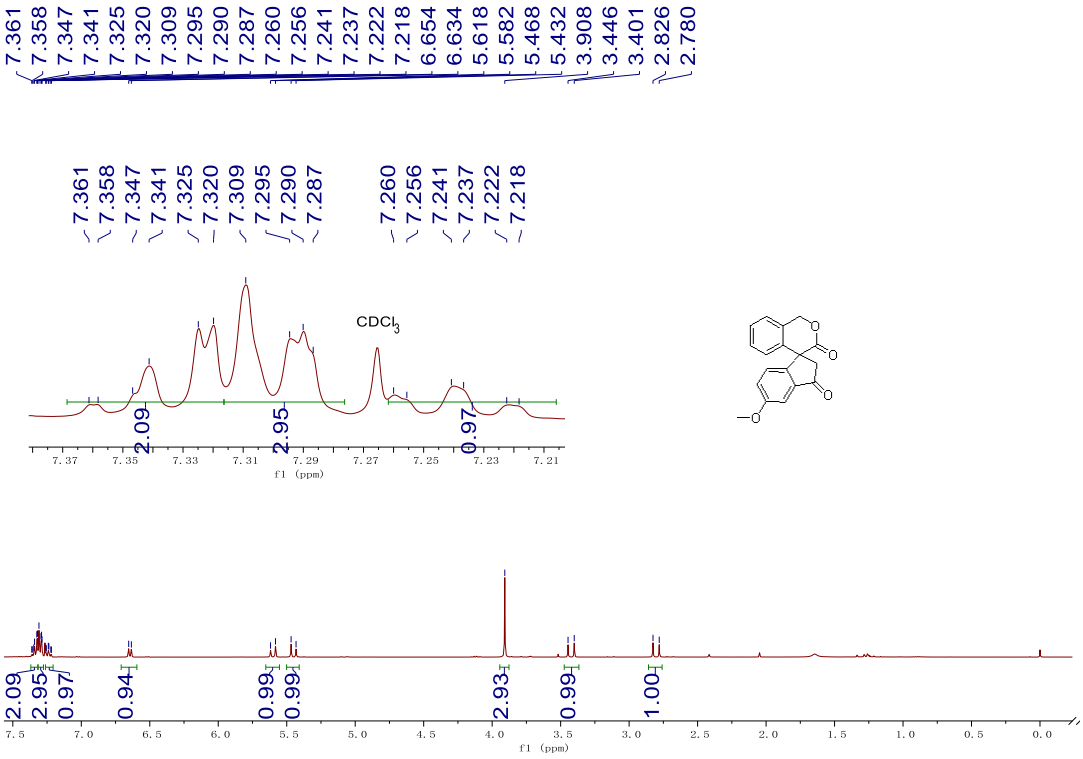
Compound **3h** ^{13}C NMR 100 MHz



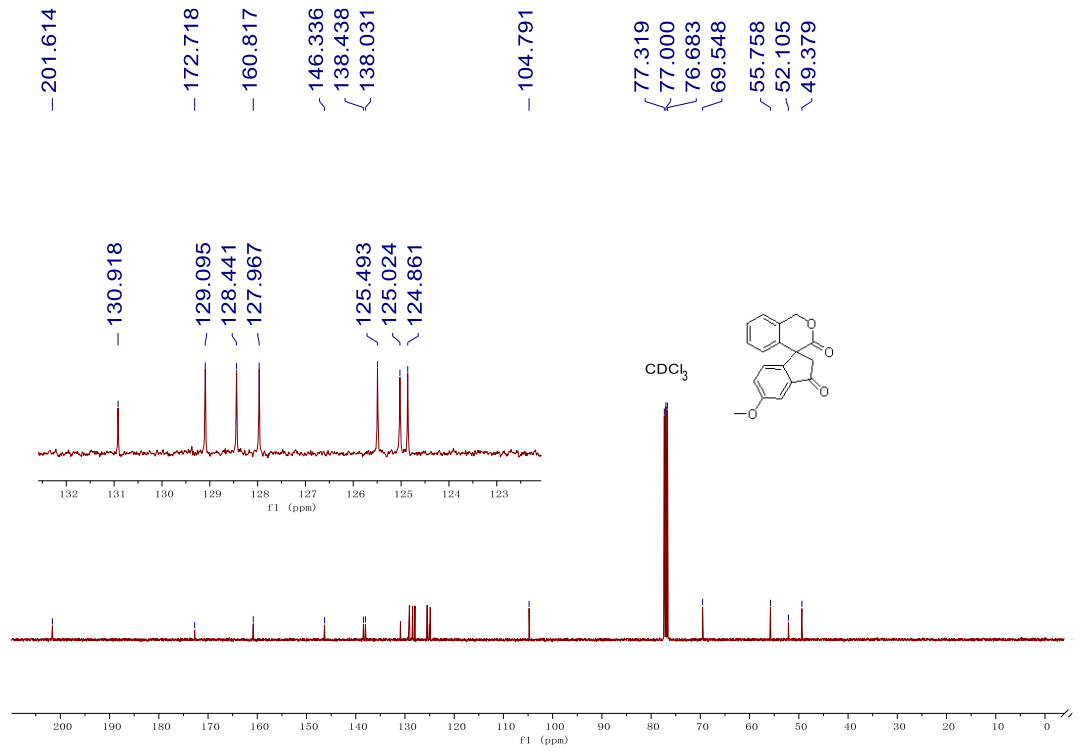
Compound **3i** ^{1}H NMR 600 MHz



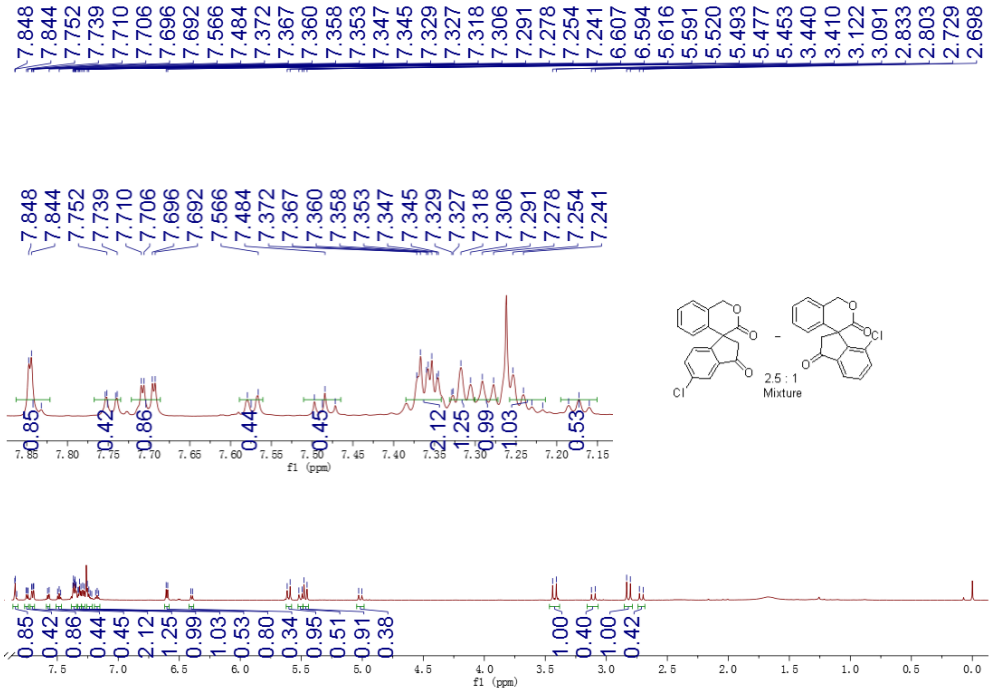
Compound **3i** ^{13}C NMR 150 MHz



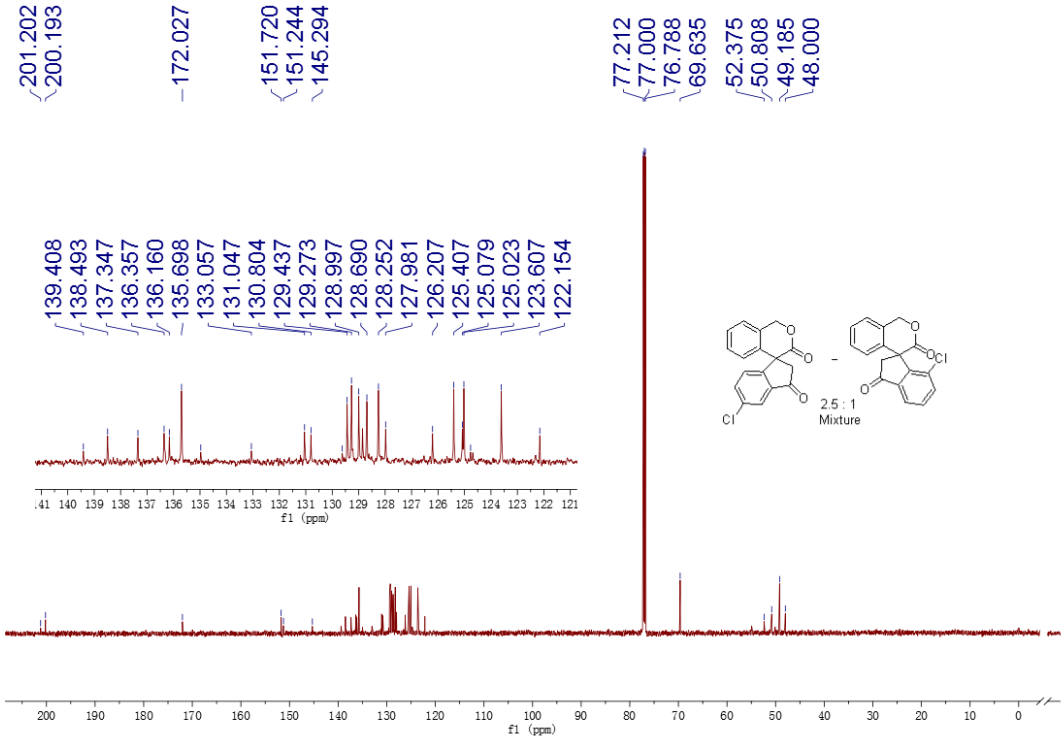
Compound **3j** ^{1}H NMR 400 MHz



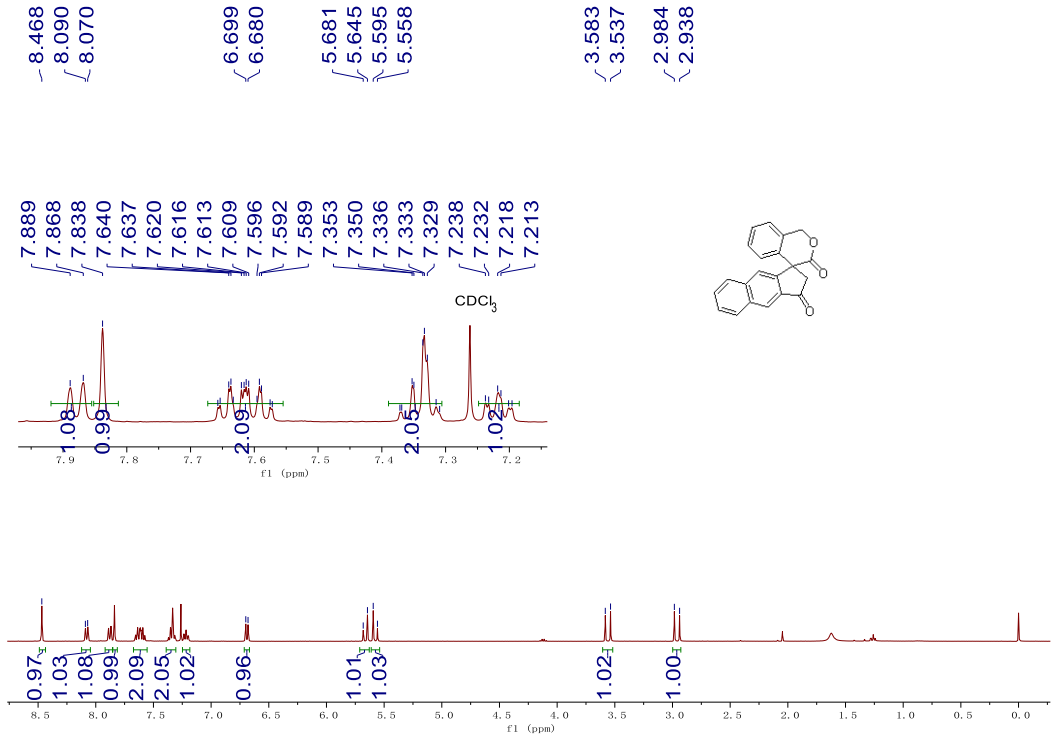
Compound **3j** ^{13}C NMR 100 MHz



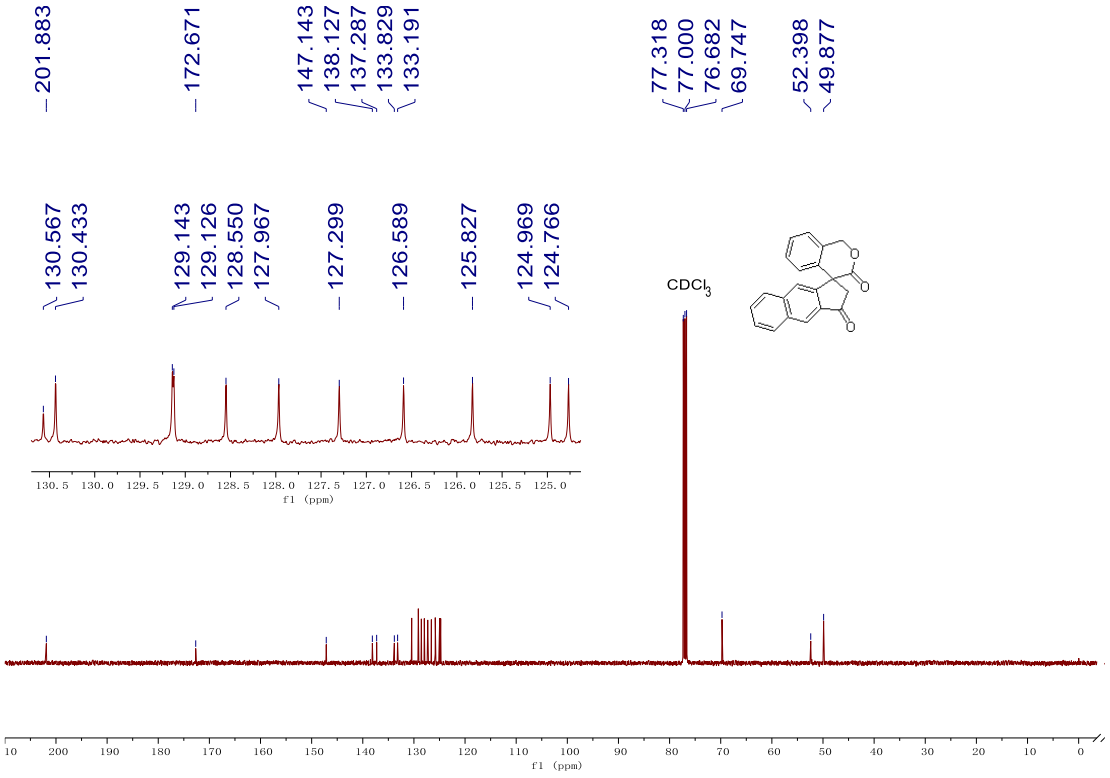
Compound **3k & 3ka** ^{1}H NMR 600 MHz



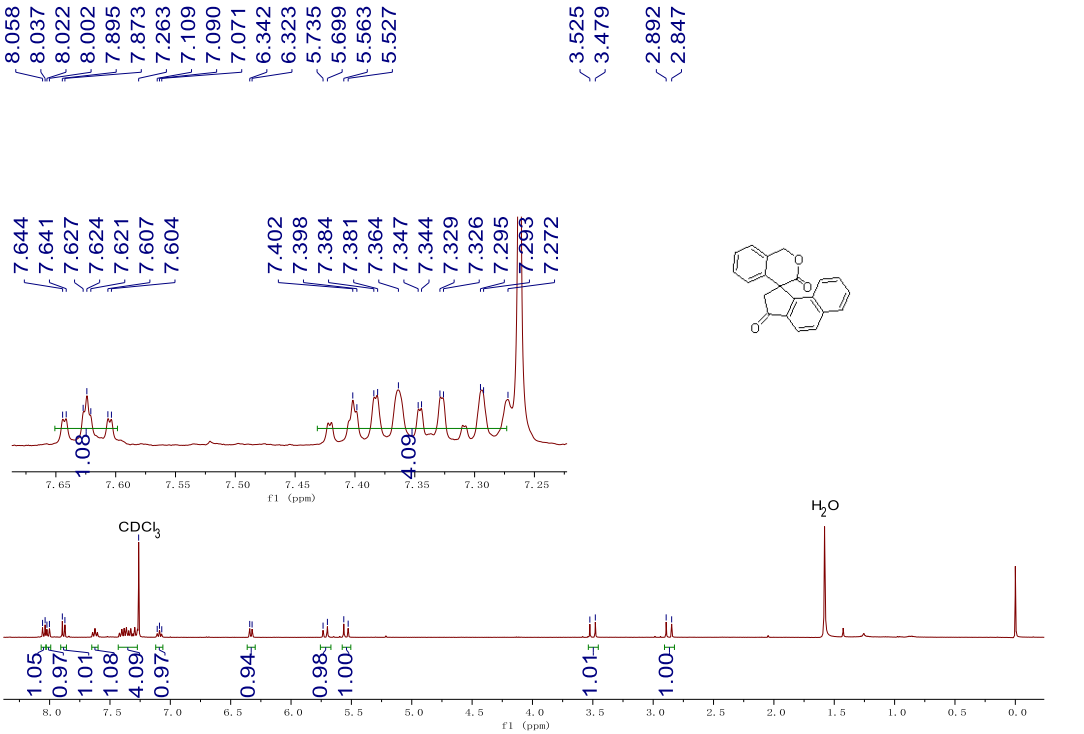
Compound **3k & 3ka** ^{13}C NMR 150 MHz



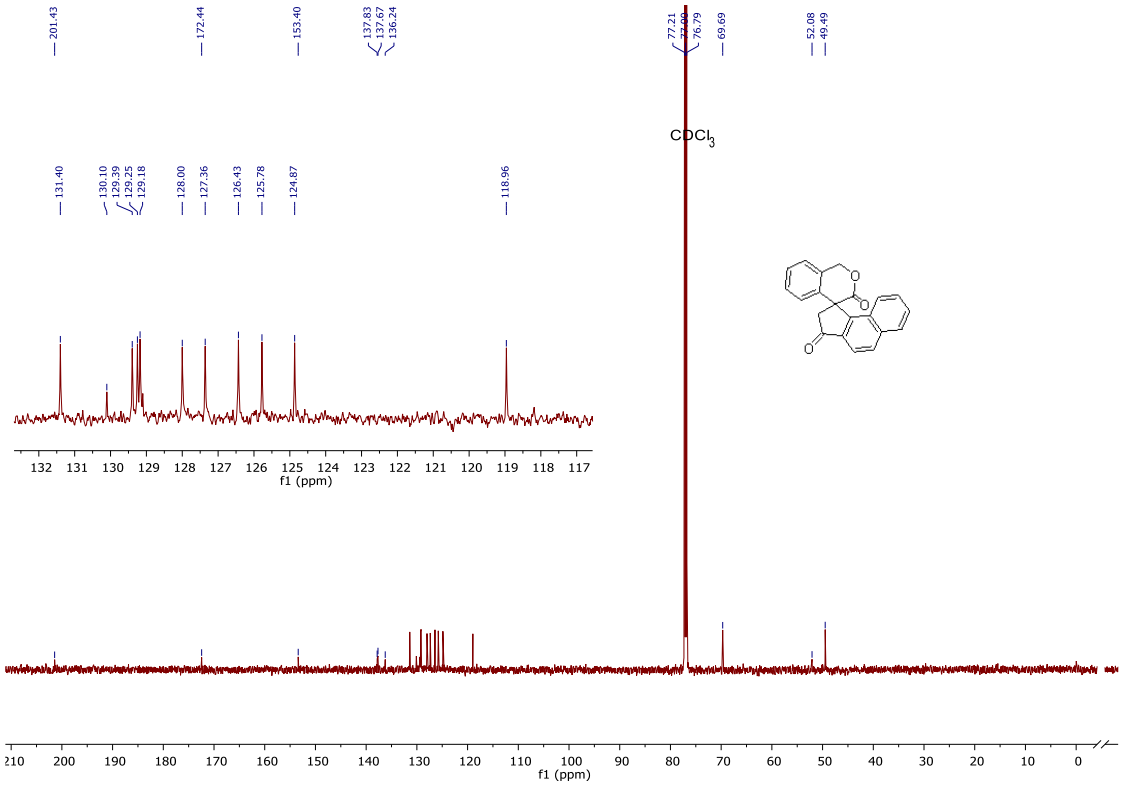
Compound **3l** ^{1}H NMR 400 MHz



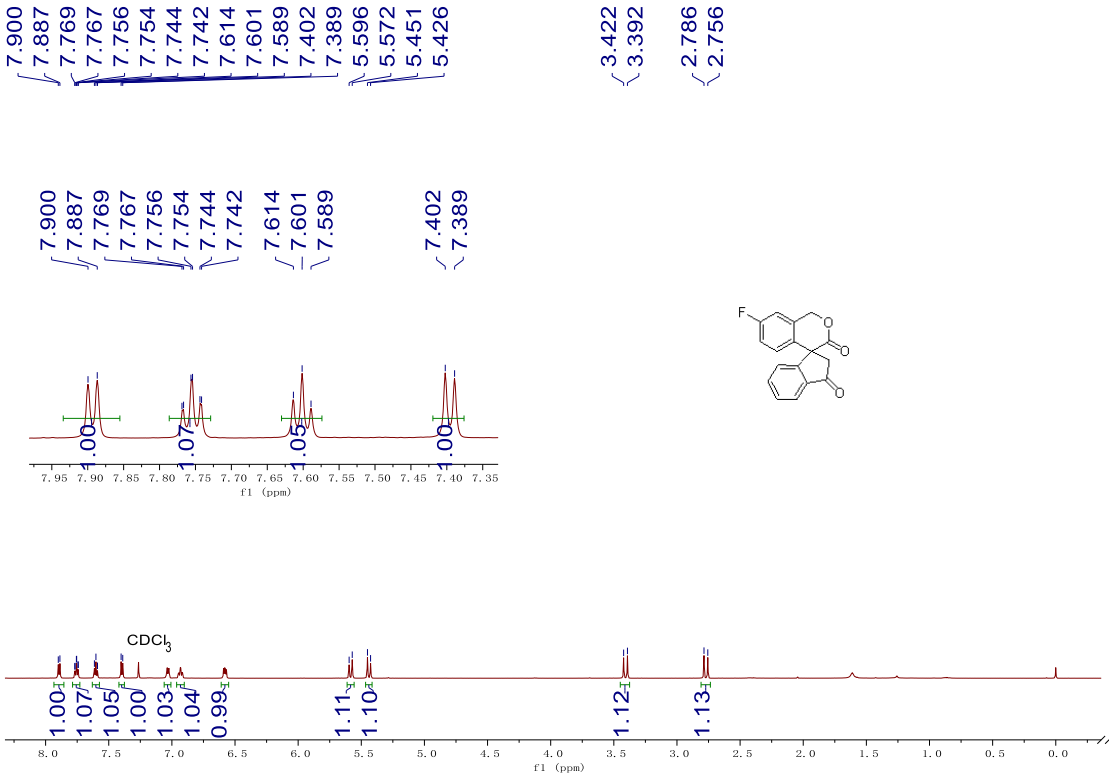
Compound **3l** ^{13}C NMR 100 MHz



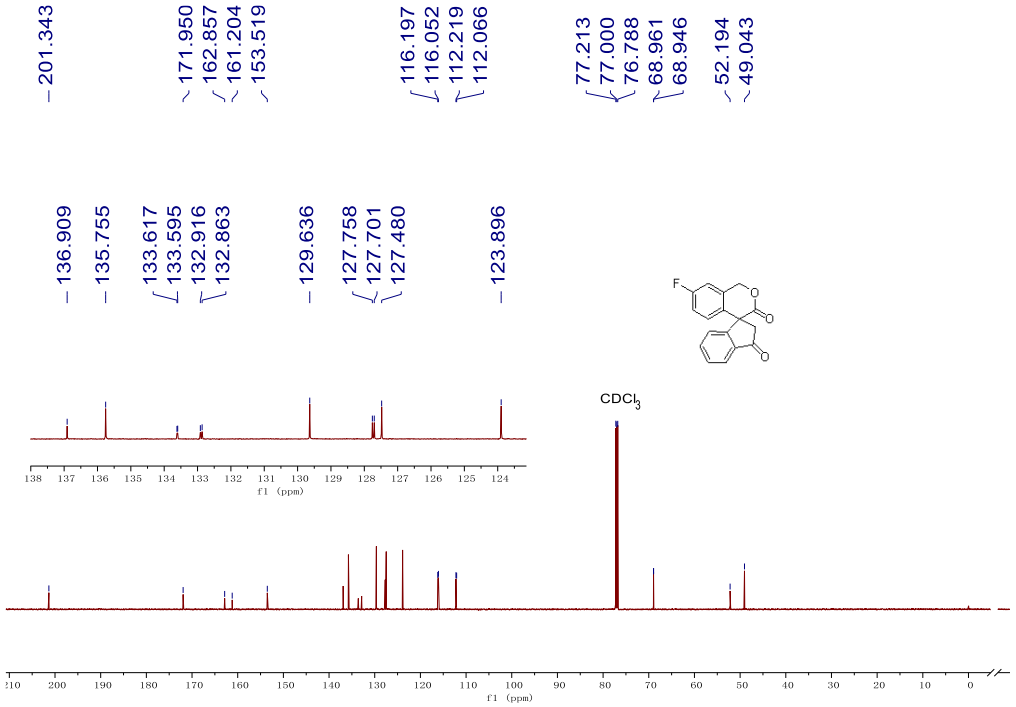
Compound **3la** ^{1}H NMR 600 MHz



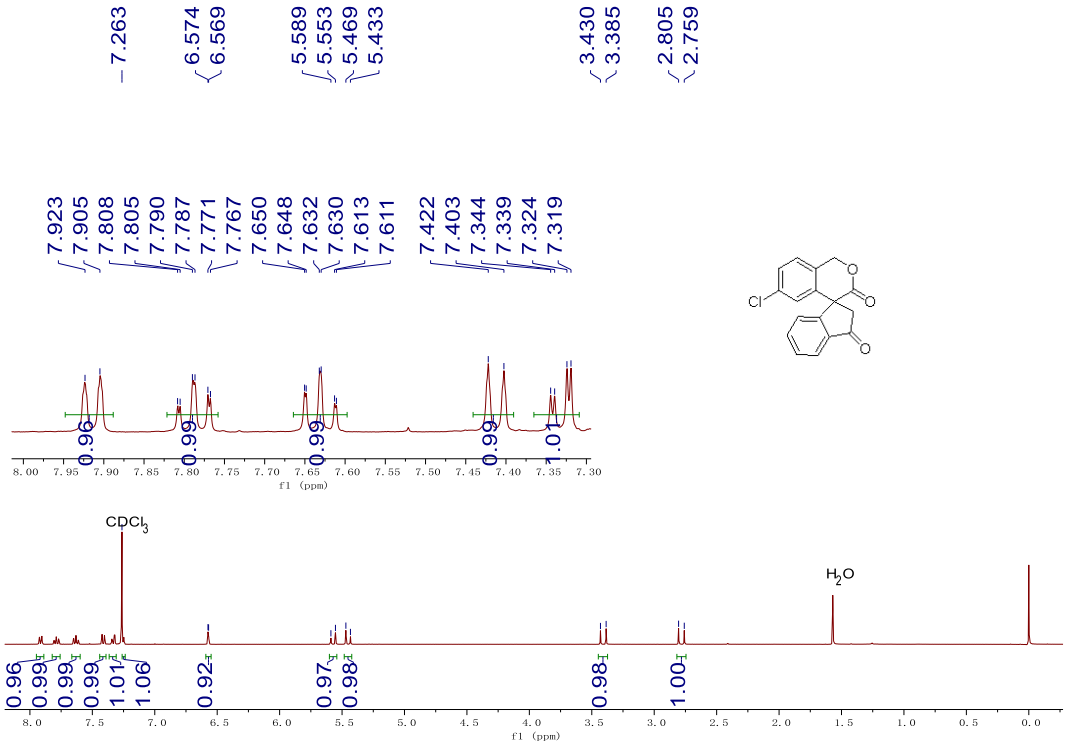
Compound **3la** ^{13}C NMR 150 MHz



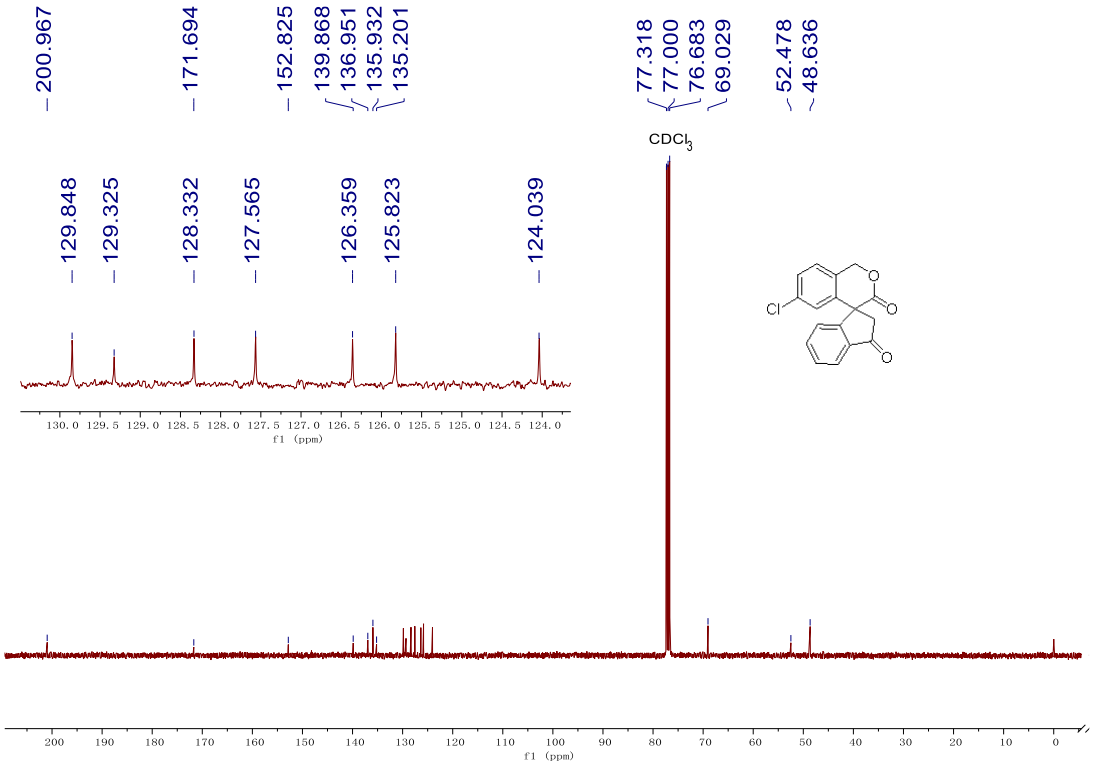
Compound **3m** ^{1}H NMR 600 MHz



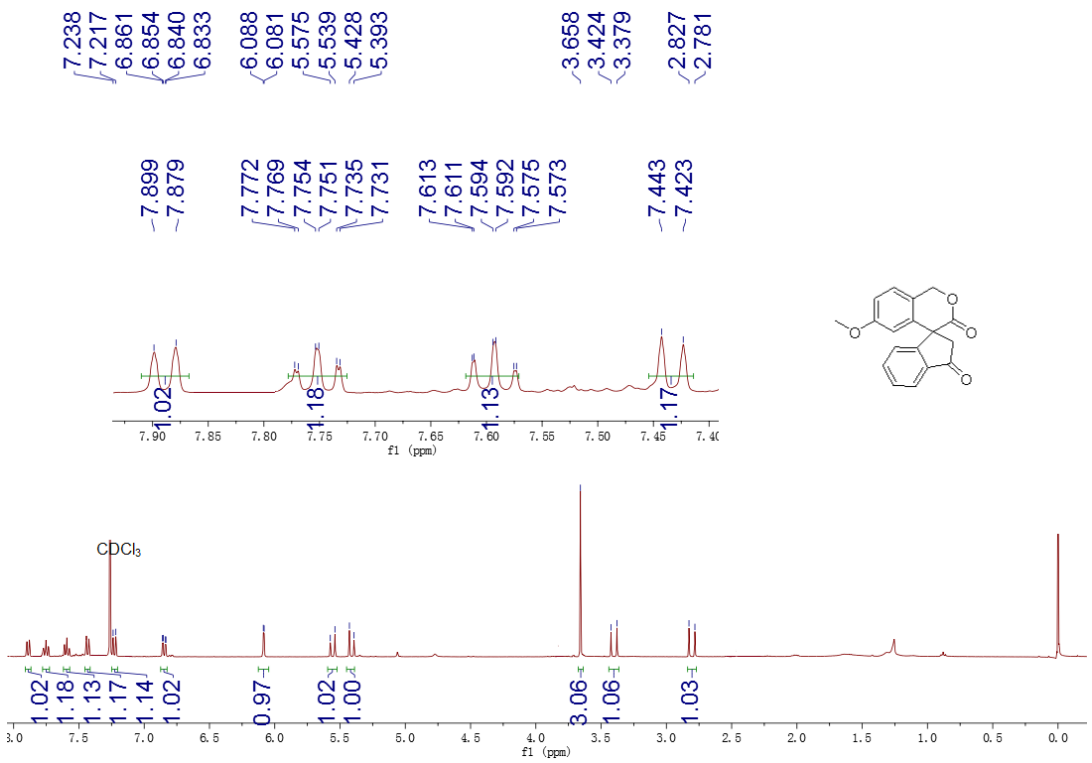
Compound **3m** ^{13}C NMR 150 MHz



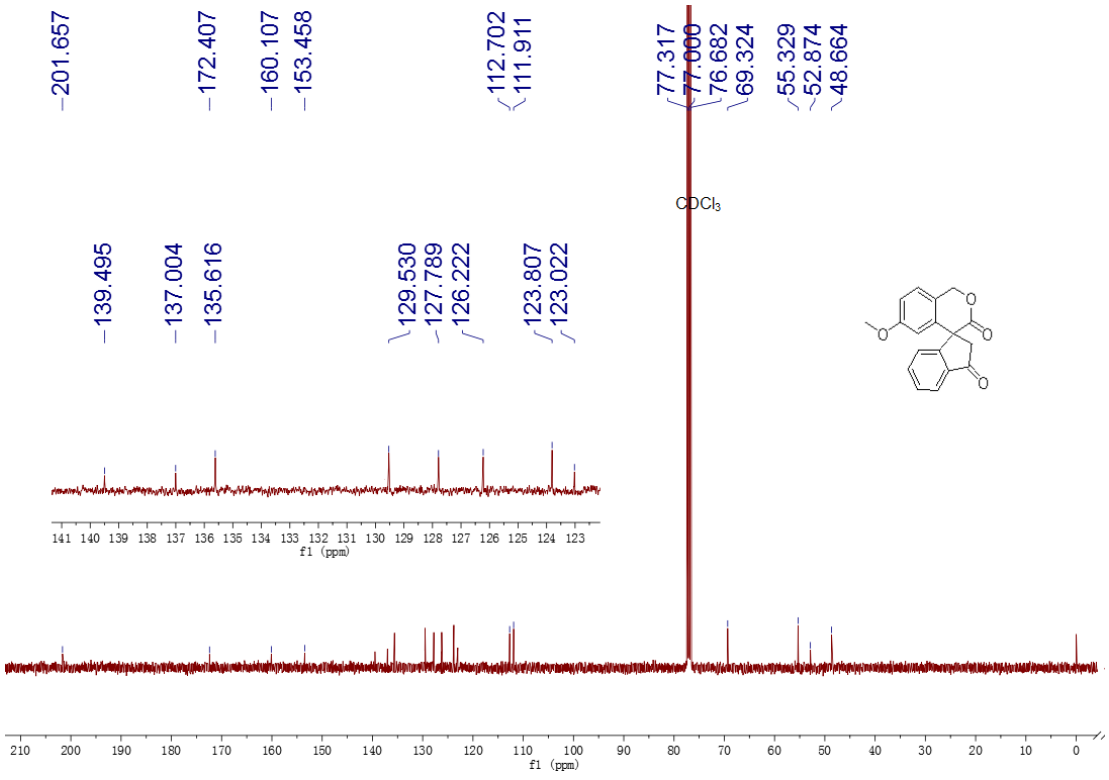
Compound **3n** ^{1}H NMR 400 MHz



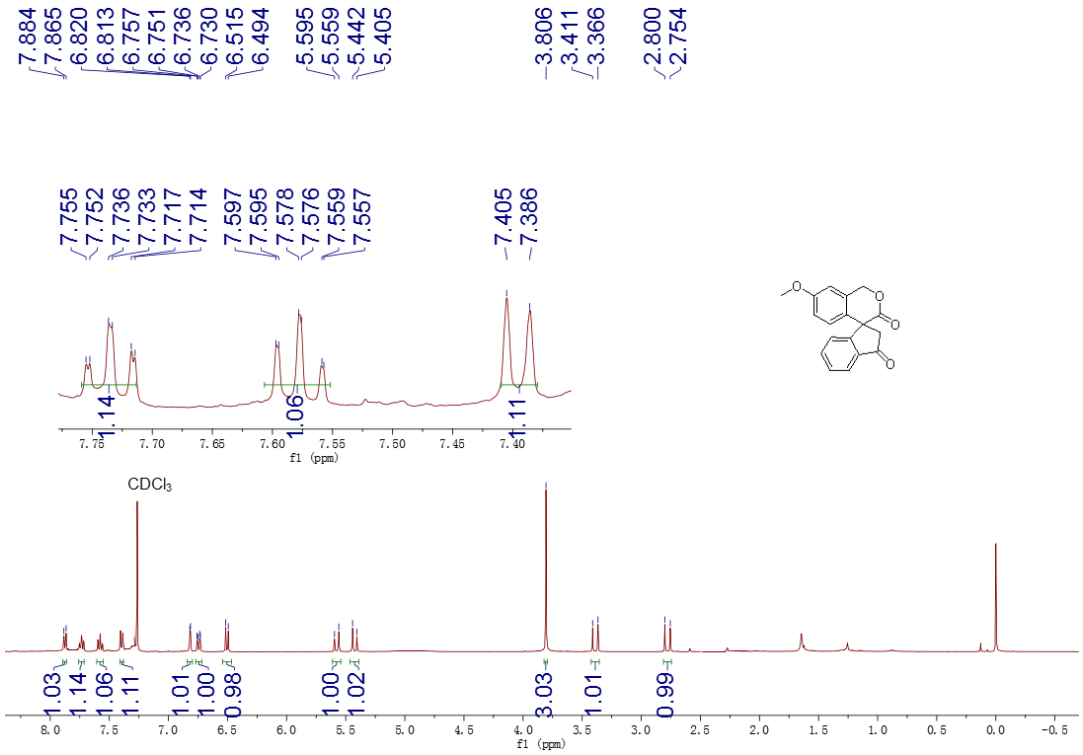
Compound **3n** ^{13}C NMR 100 MHz



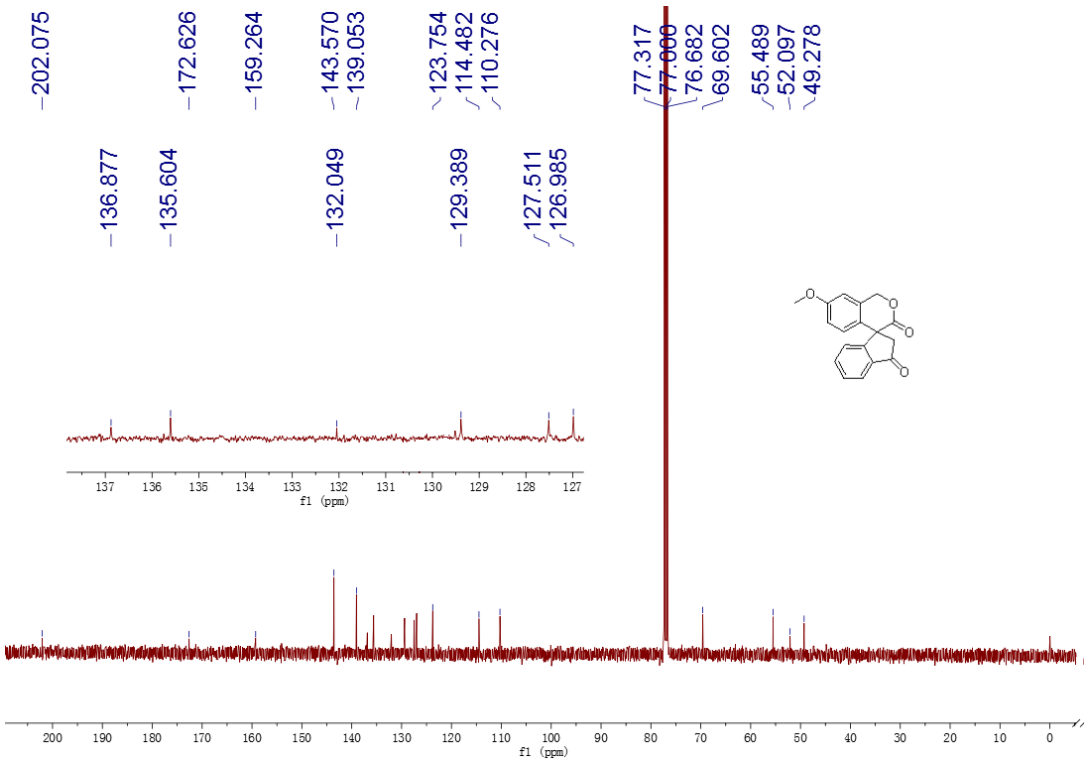
Compound **3o** ^{1}H NMR 400 MHz



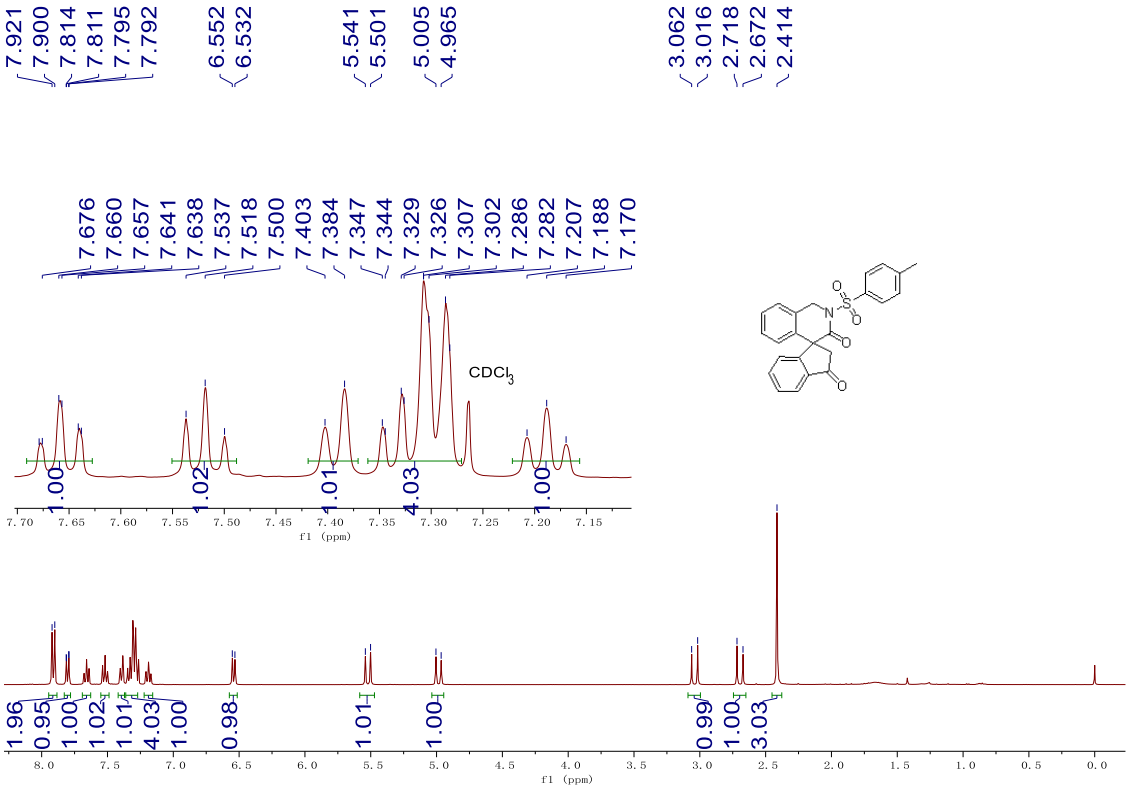
Compound **3o** ^{13}C NMR 100 MHz



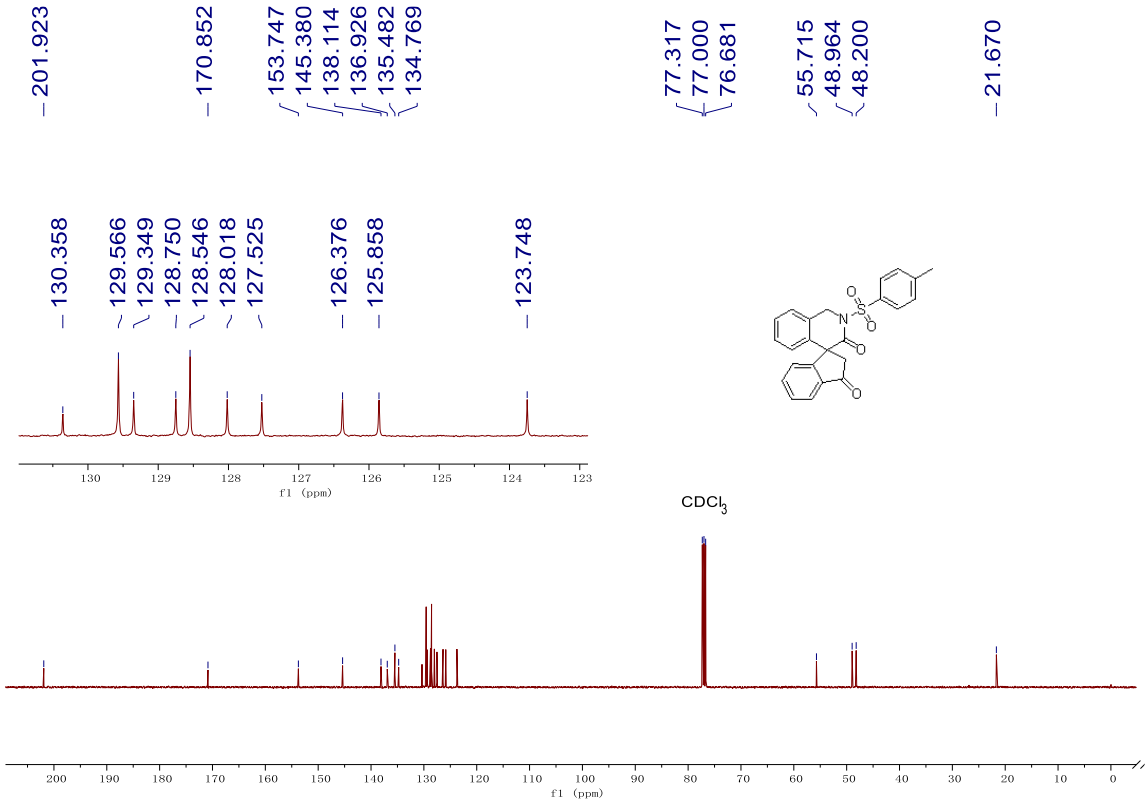
Compound **3p** ^{1}H NMR 400 MHz



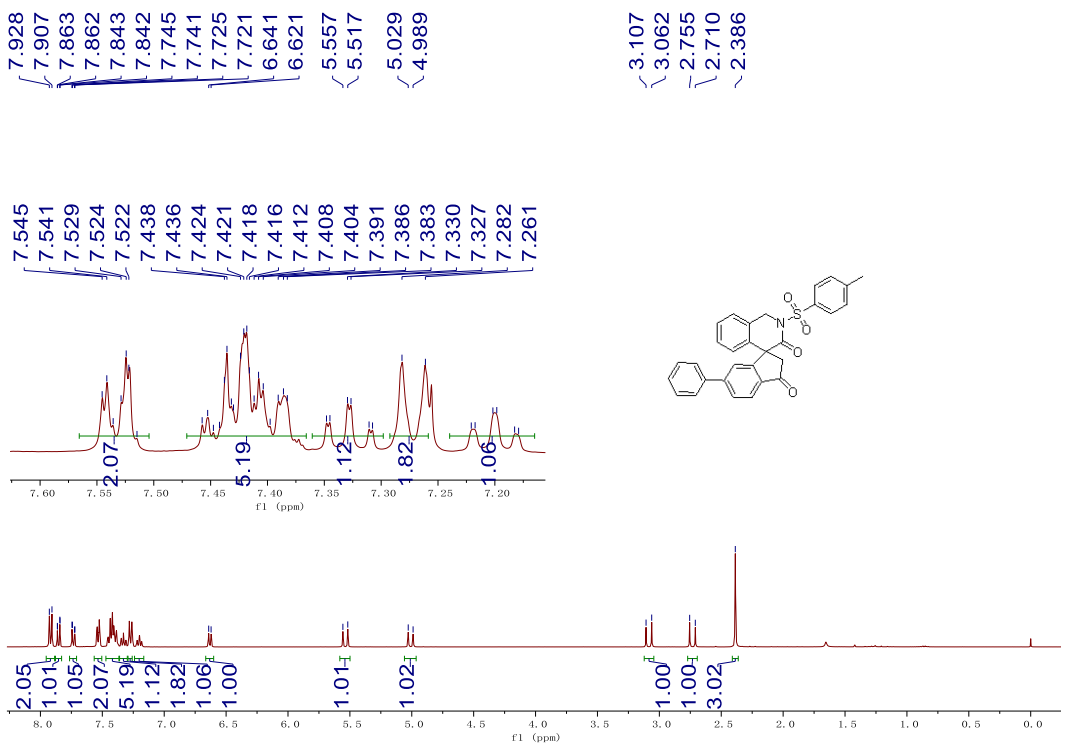
Compound **3p** ^{13}C NMR 100 MHz



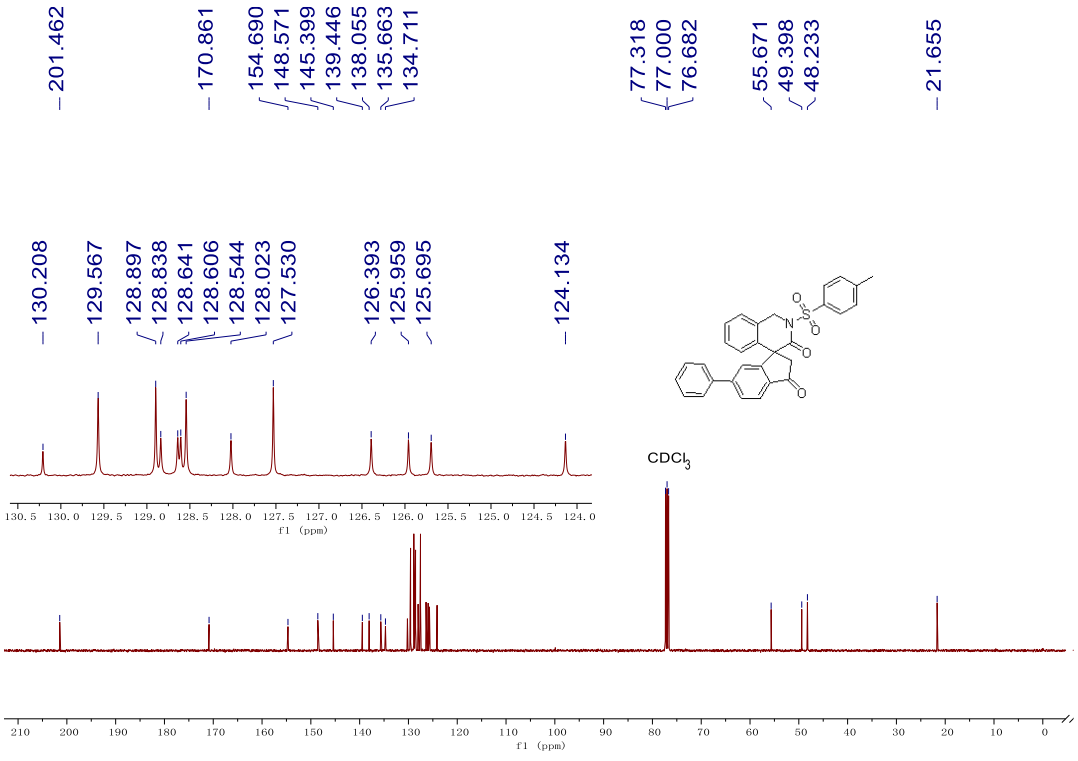
Compound **5a** ^{1}H NMR 400 MHz



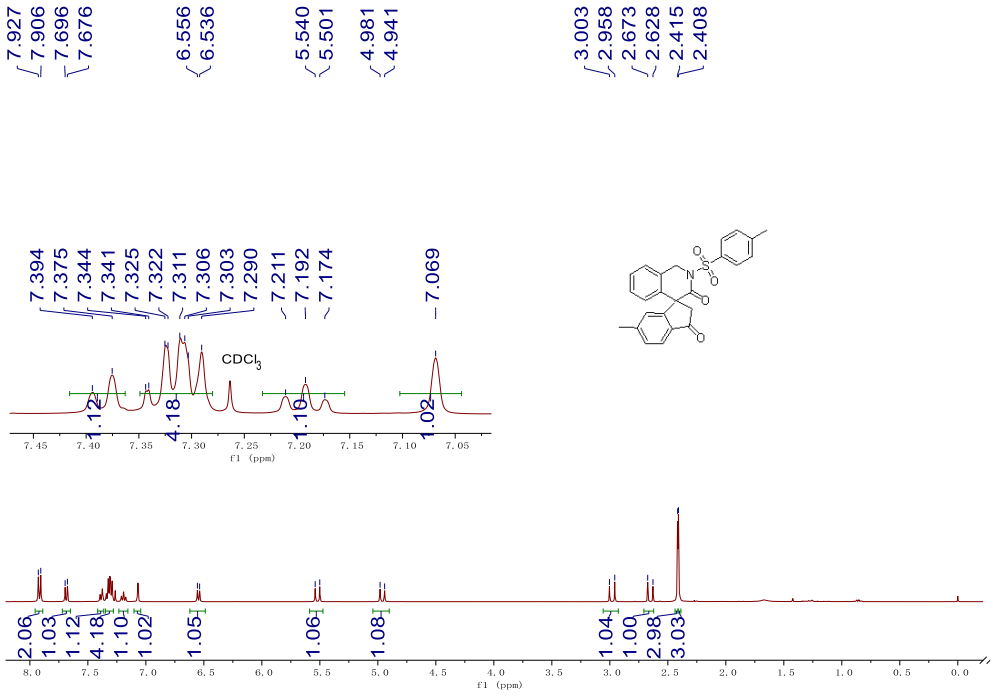
Compound **5a** ^{13}C NMR 100 MHz



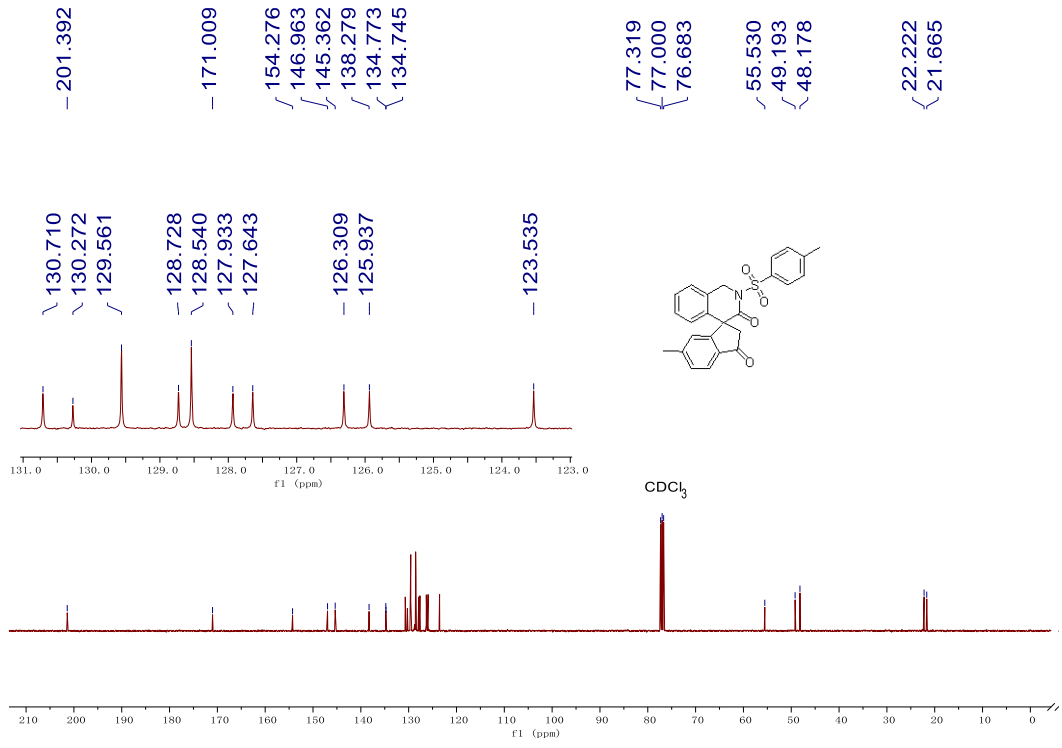
Compound **5b** ^{1}H NMR 400 MHz



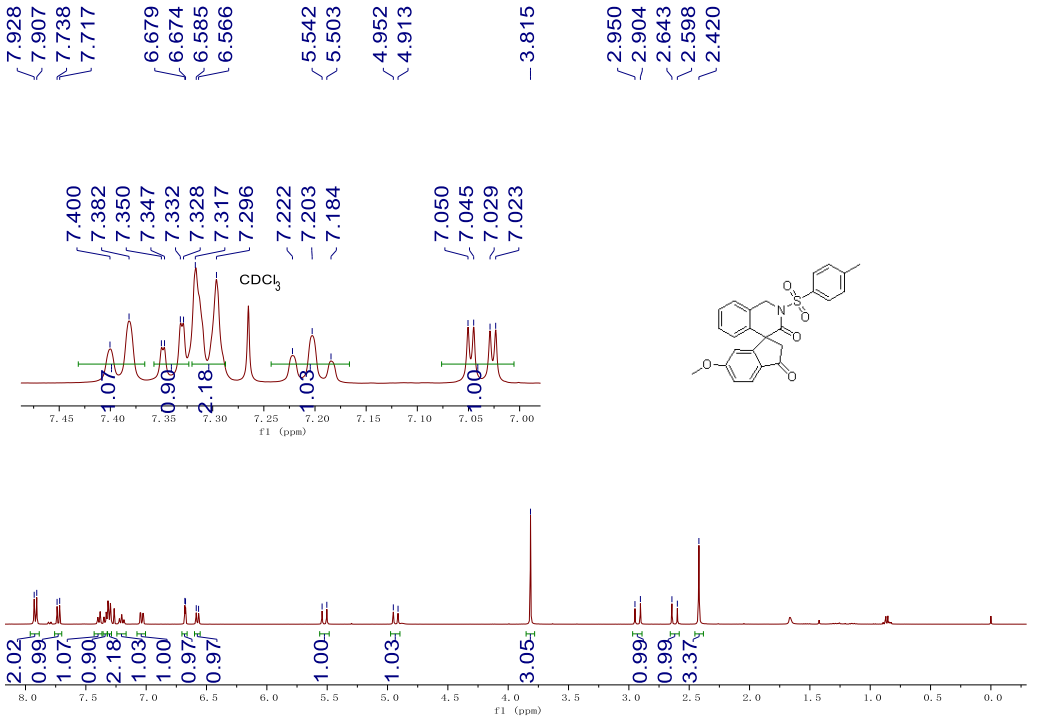
Compound **5b** ^{13}C NMR 100 MHz



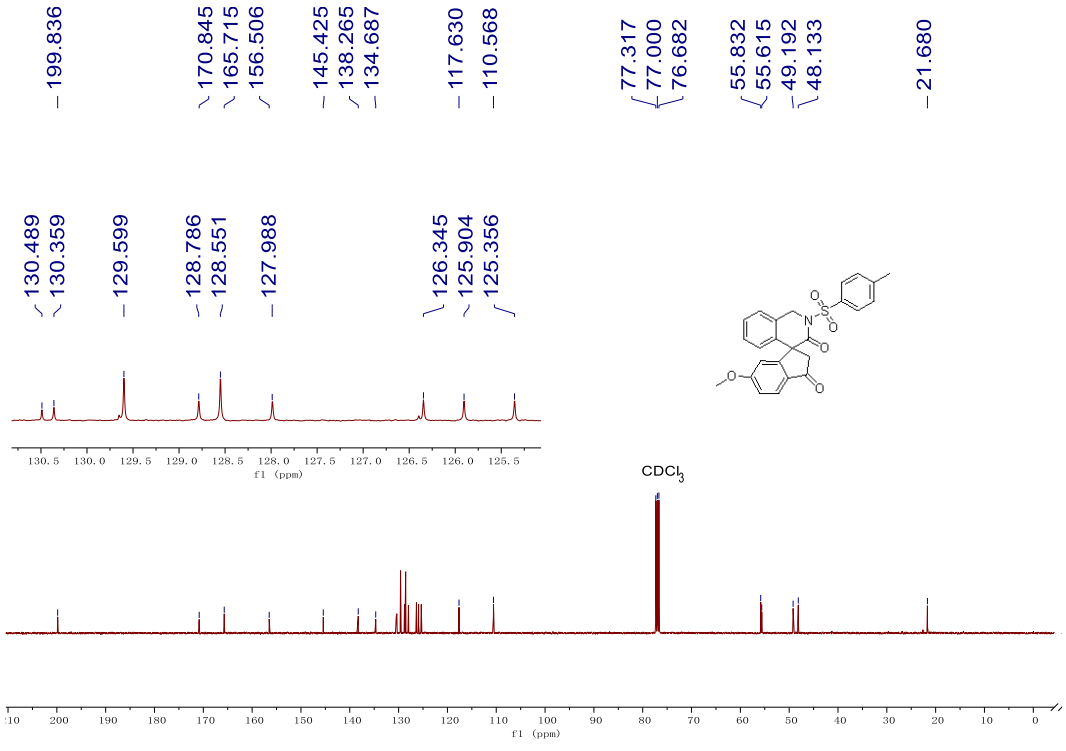
Compound **5c** ^{1}H NMR 400 MHz



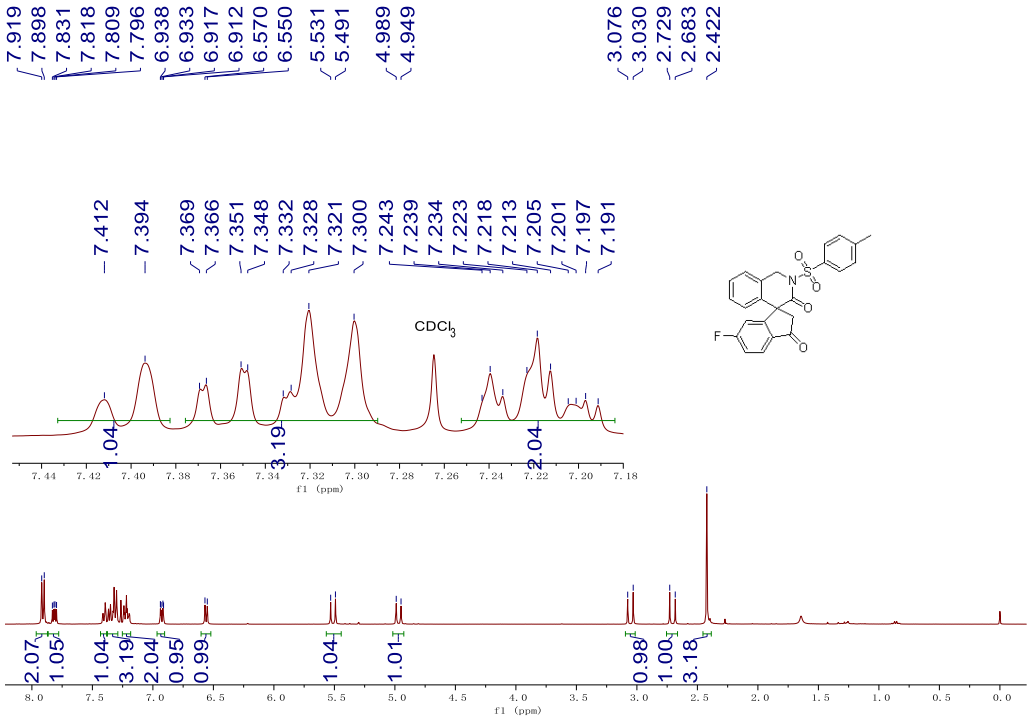
Compound **5c** ^{13}C NMR 100 MHz



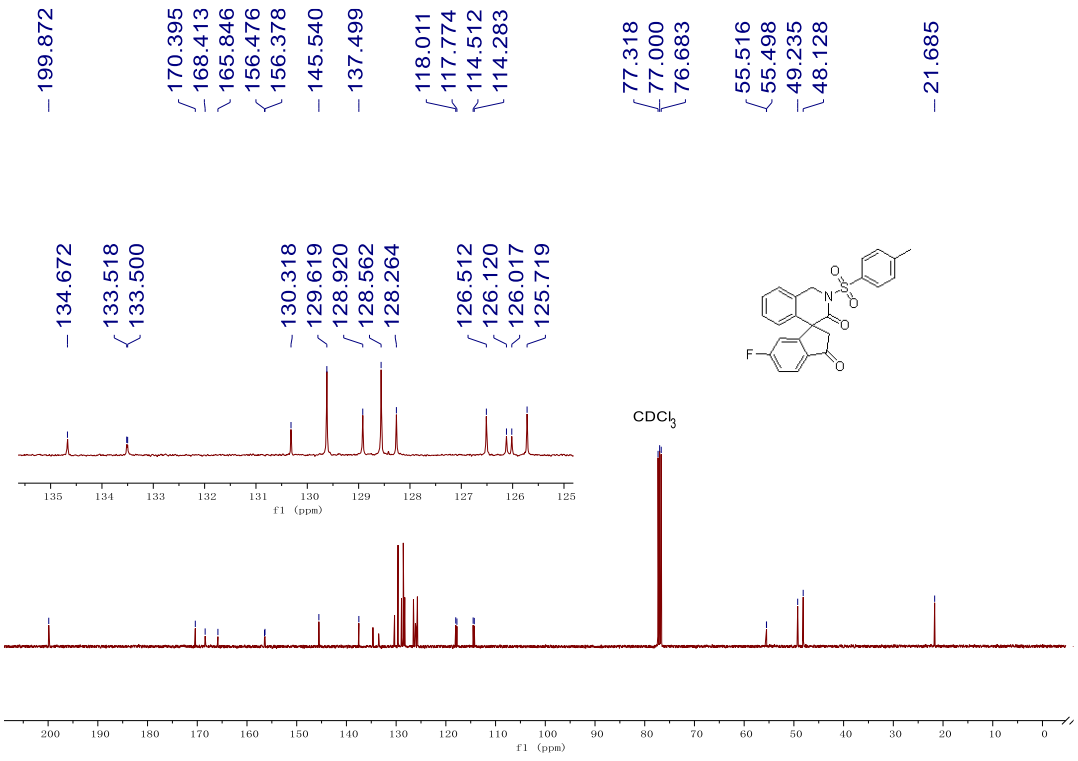
Compound **5d** ^{1}H NMR 400 MHz



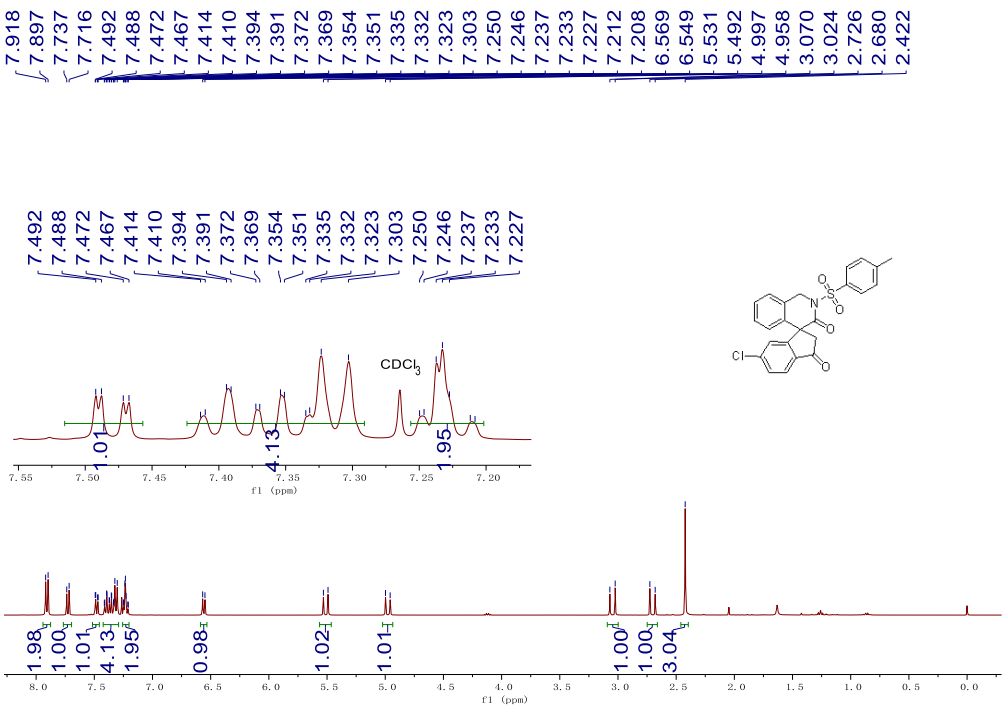
Compound **5d** ^{13}C NMR 100 MHz



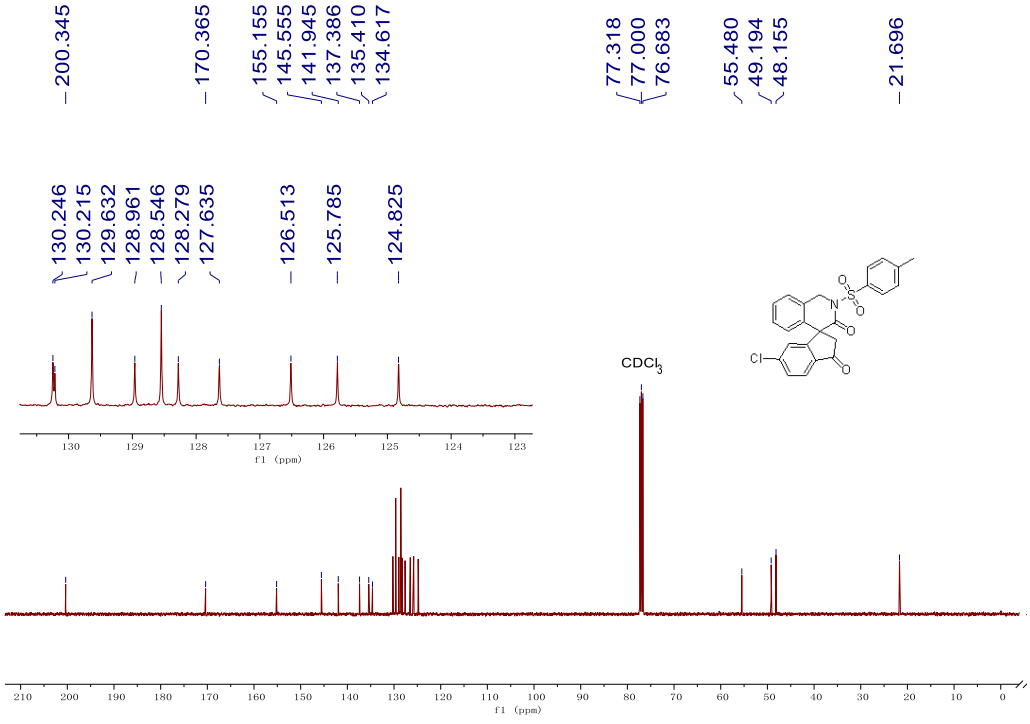
Compound **5e** ^{1}H NMR 400 MHz



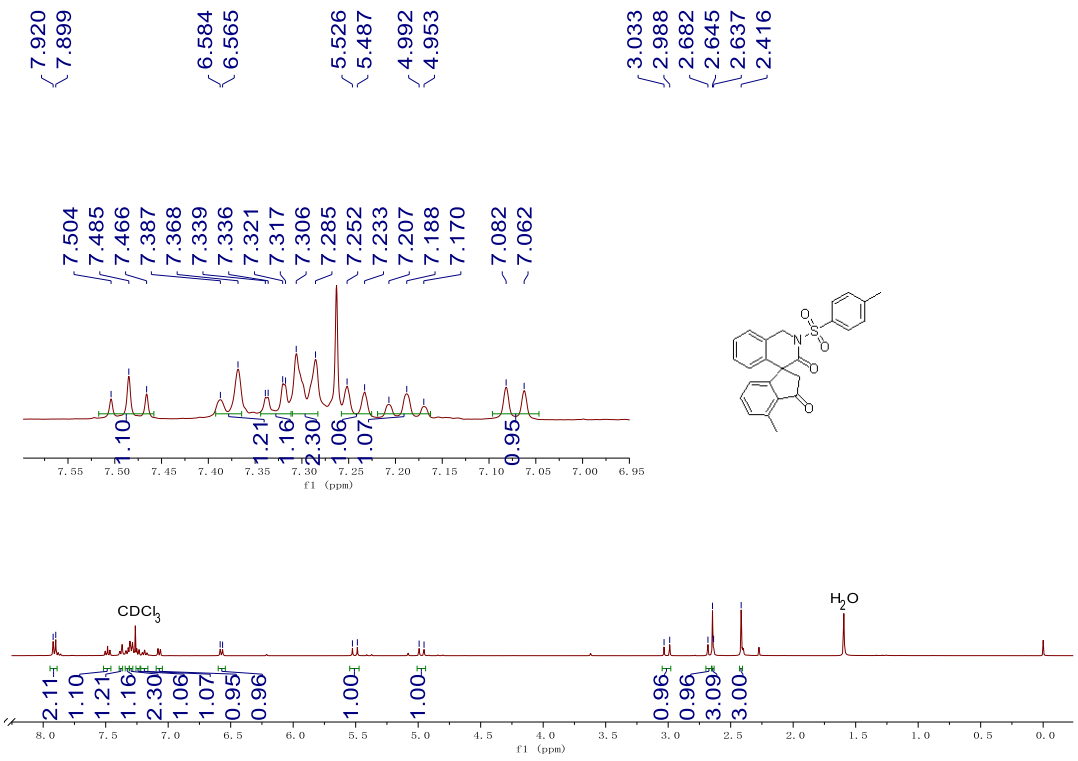
Compound **5e** ^{13}C NMR 100 MHz



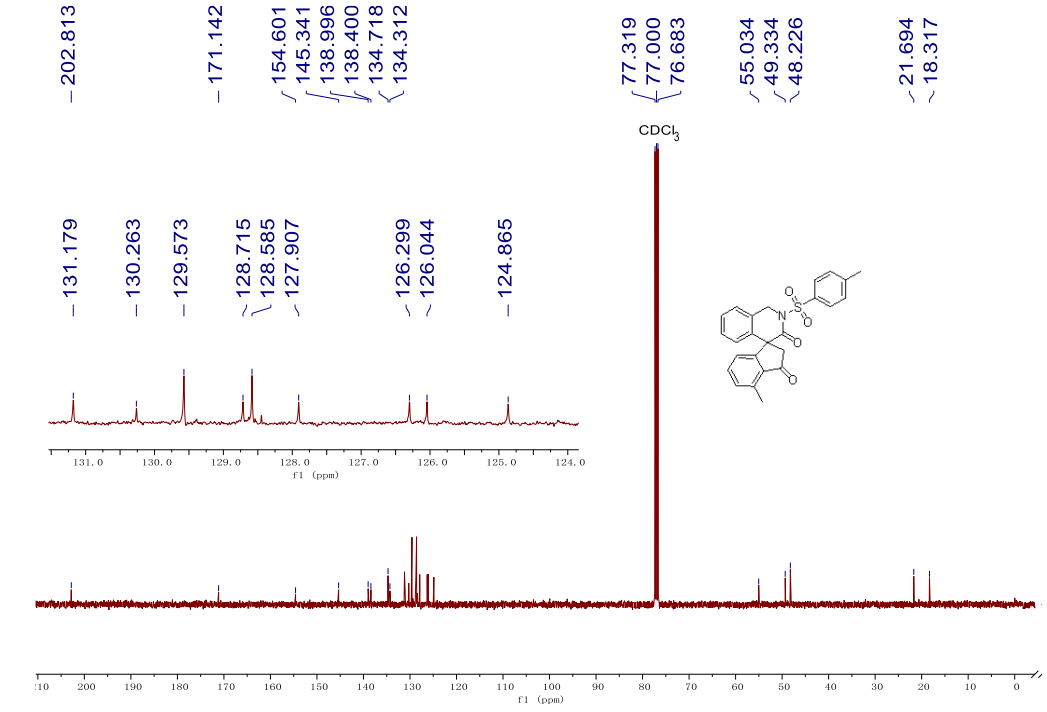
Compound **5f** ^{1}H NMR 400 MHz



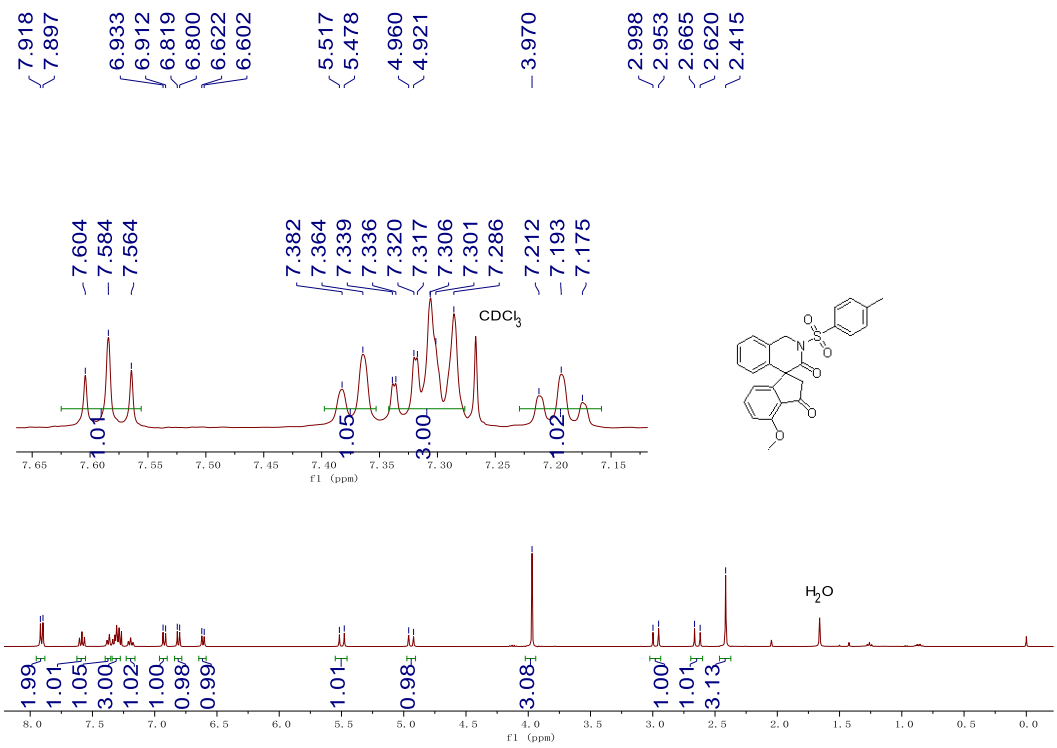
Compound **5f** ^{13}C NMR 100 MHz



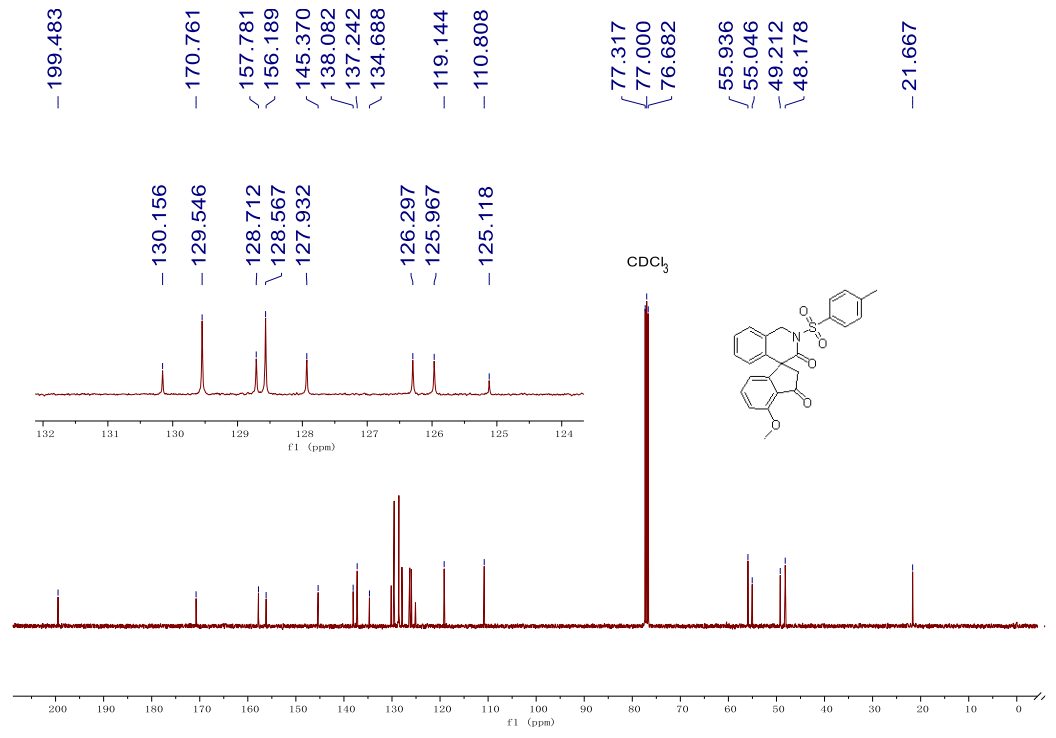
Compound **5g** ^{1}H NMR 400 MHz



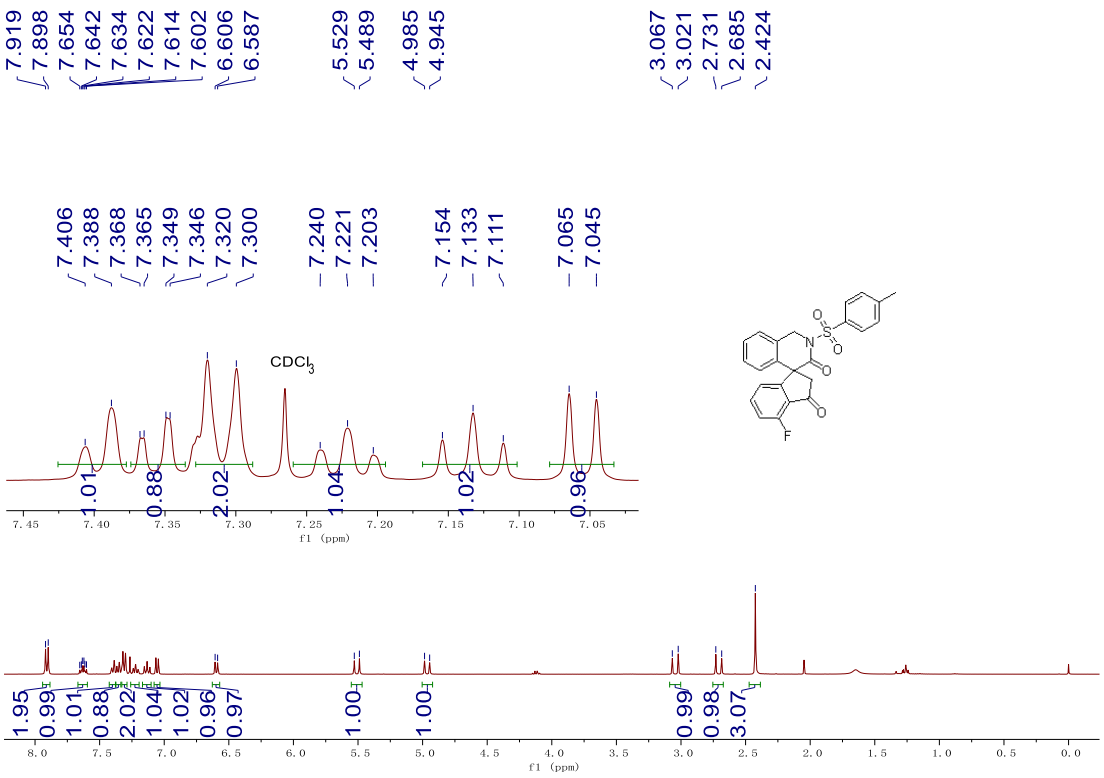
Compound **5g** ^{13}C NMR 100 MHz



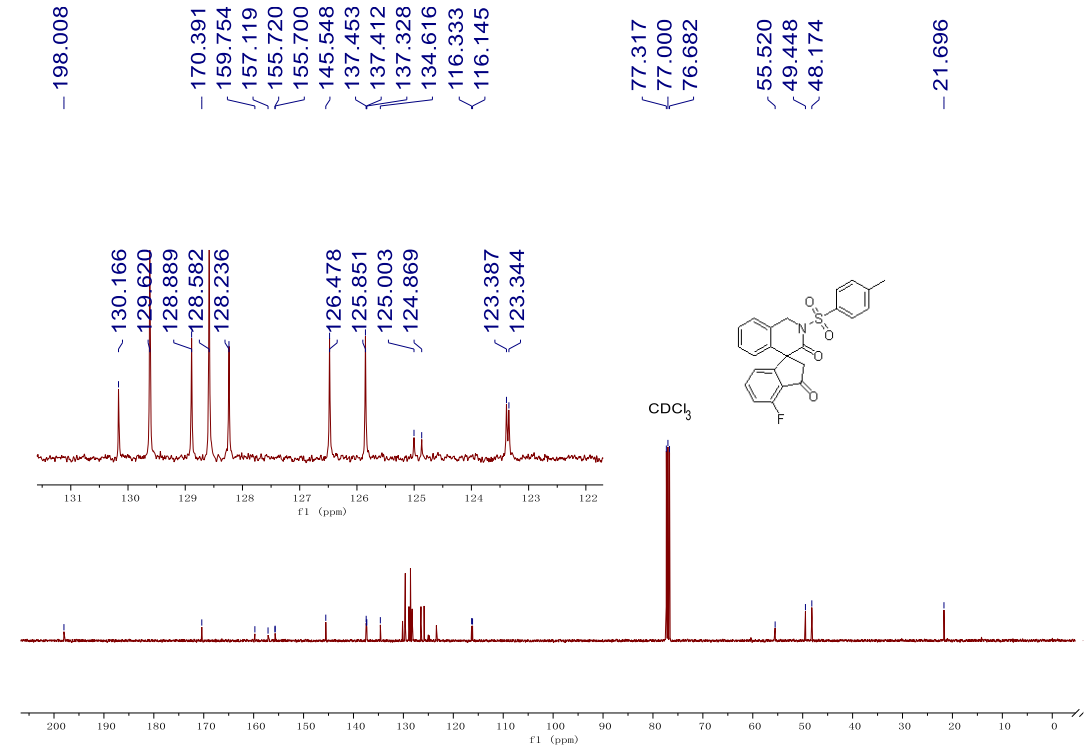
Compound **5h** ^{1}H NMR 400 MHz



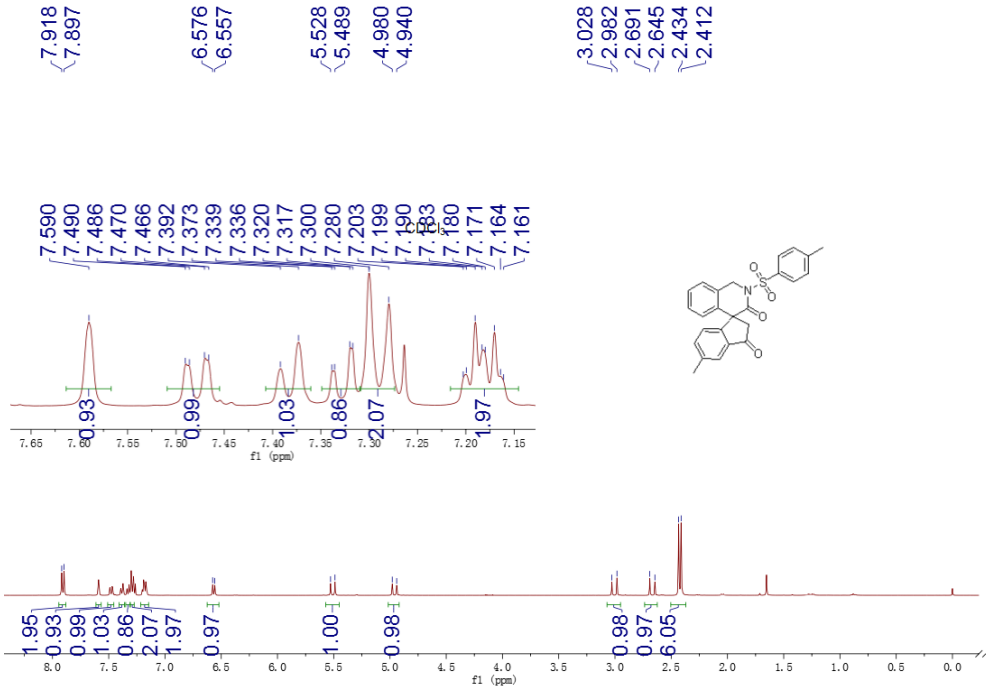
Compound **5h** ^{13}C NMR 100 MHz



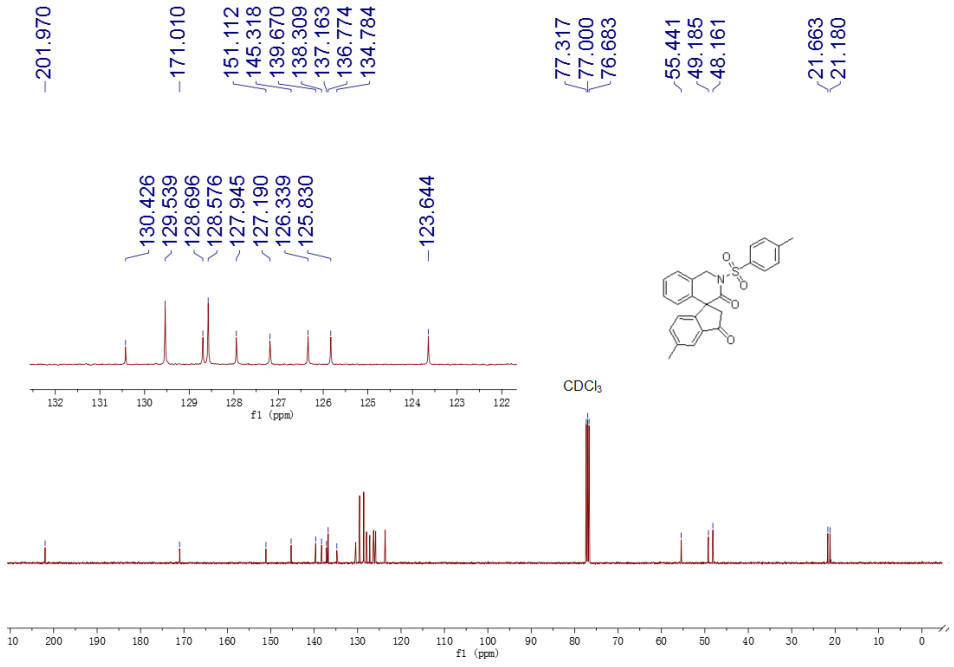
Compound **5i** ^{1}H NMR 400 MHz



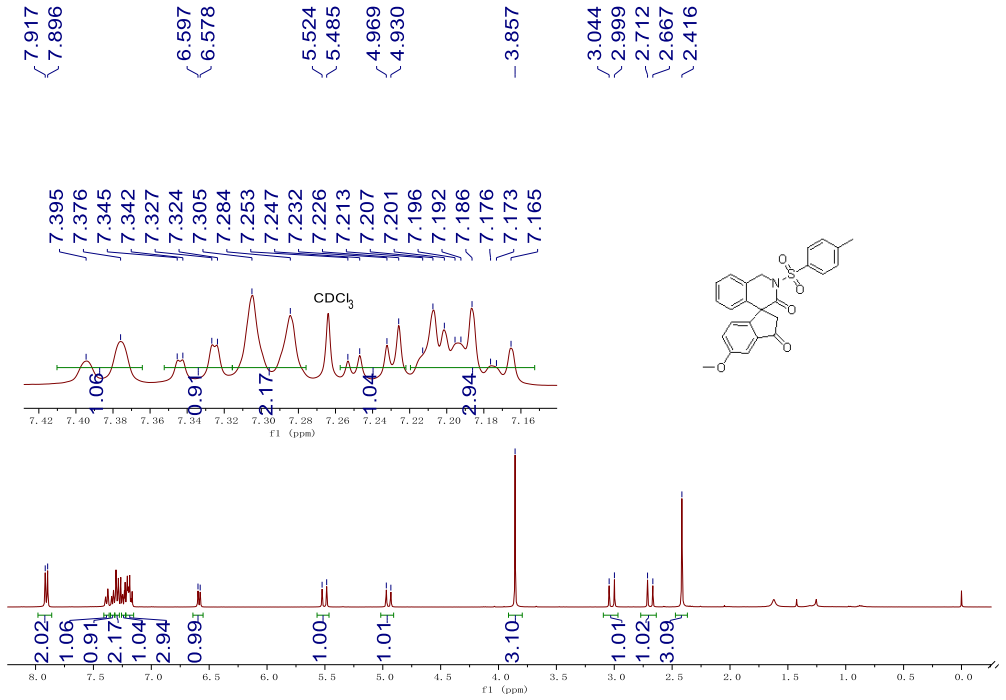
Compound **5i** ^{13}C NMR 100 MHz



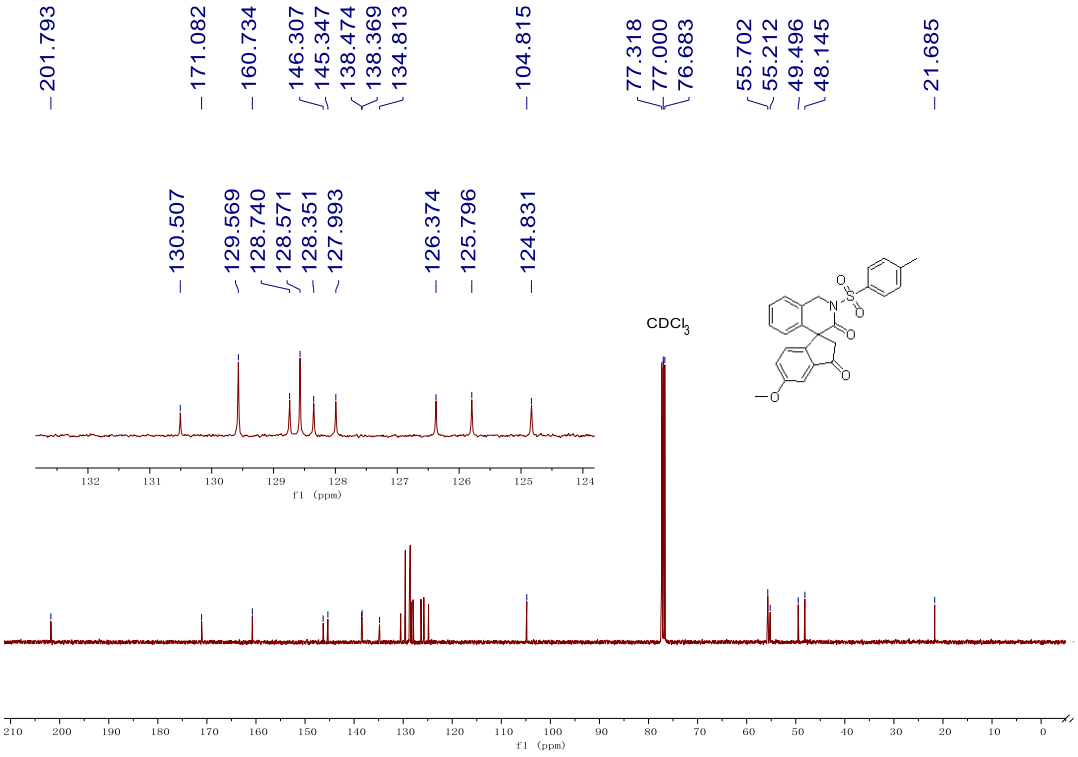
Compound **5j** ^{1}H NMR 400 MHz



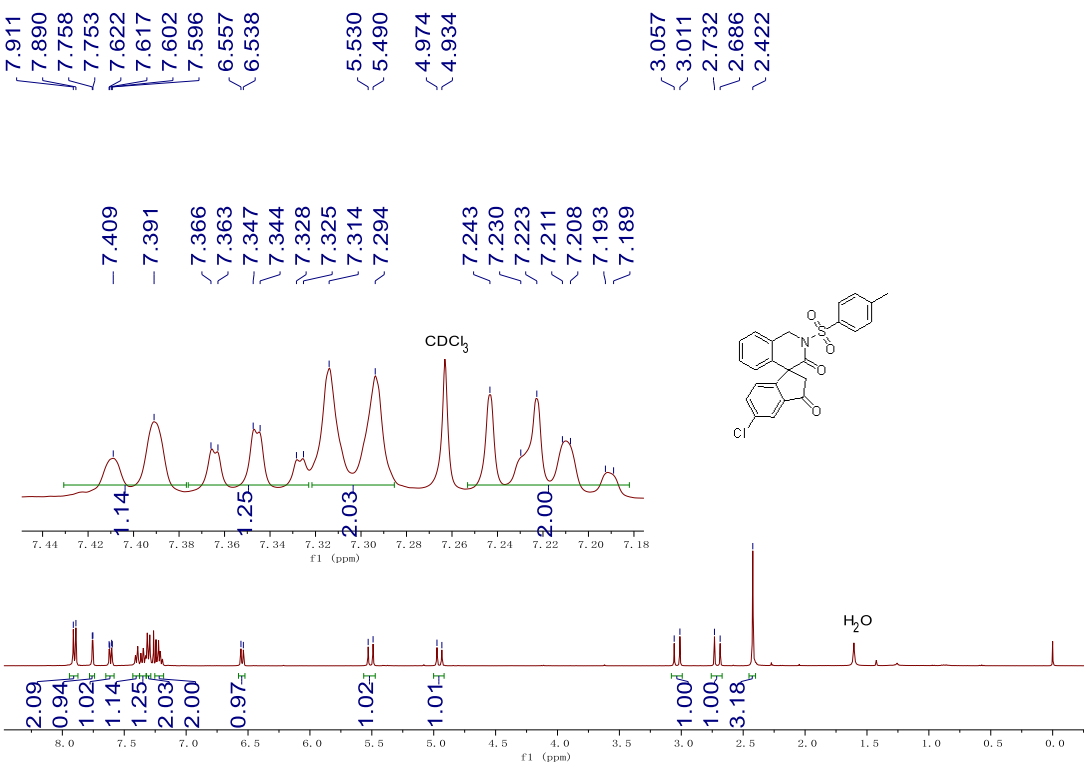
Compound **5j** ^{13}C NMR 100 MHz



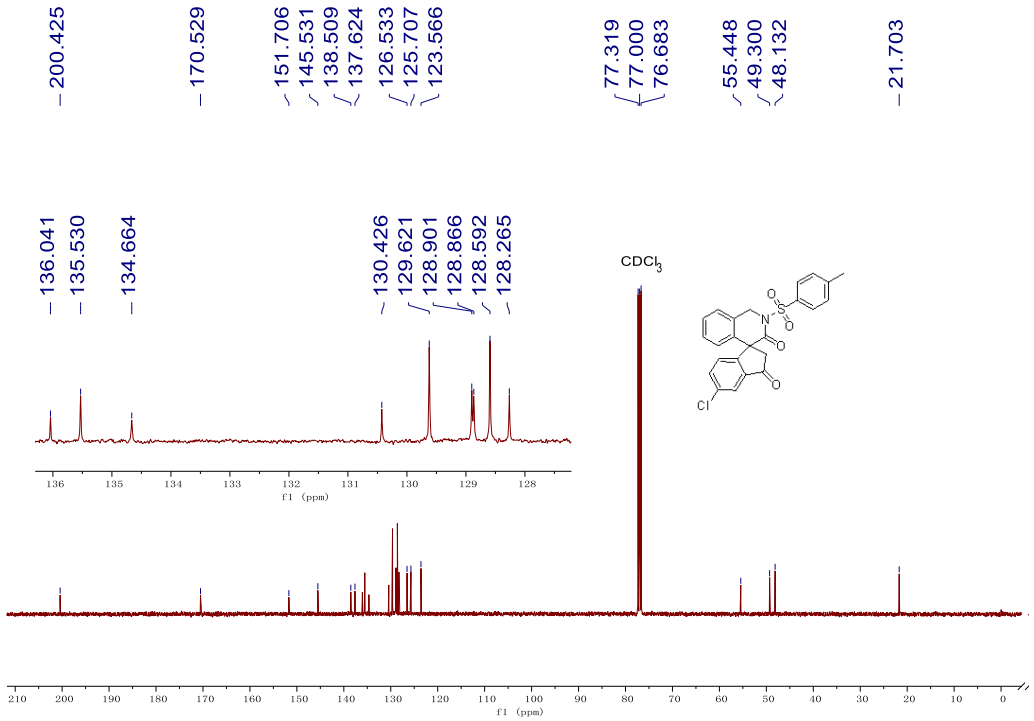
Compound **5k** ^{1}H NMR 400 MHz



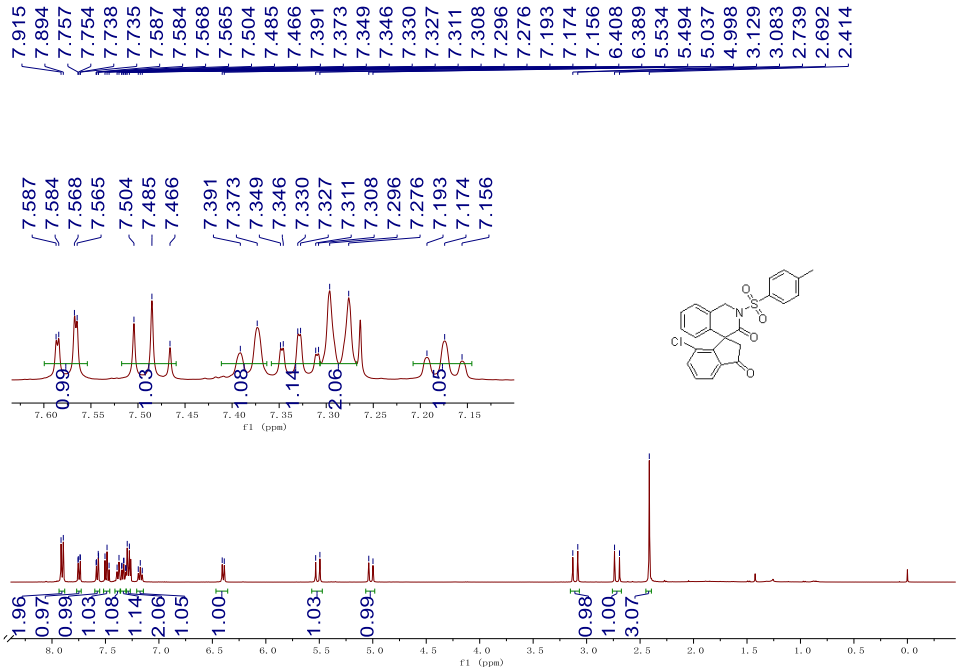
Compound **5k** ^{13}C NMR 100 MHz



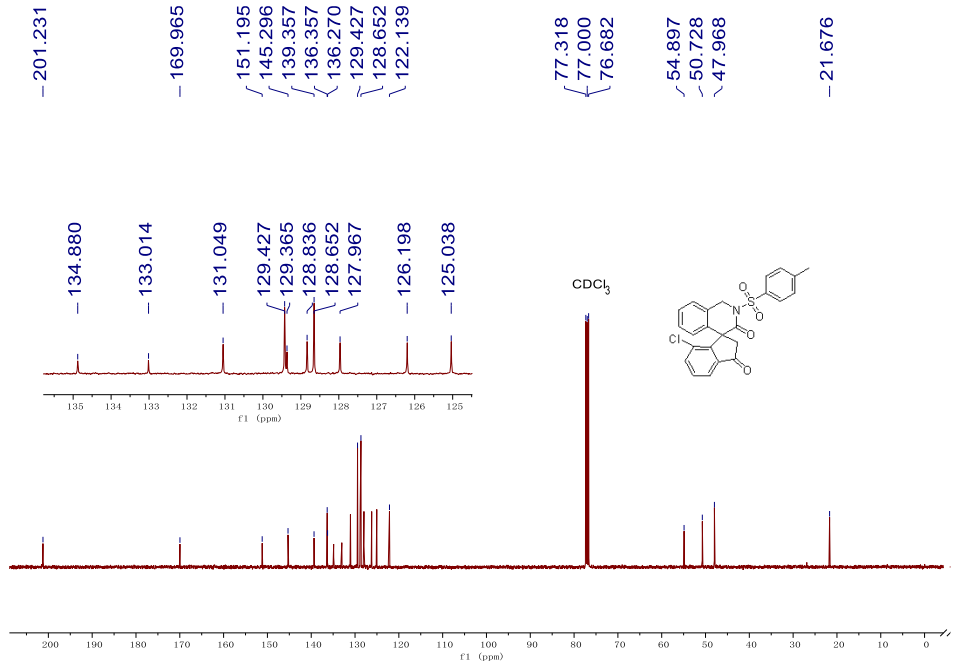
Compound **5l** ^{1}H NMR 400 MHz



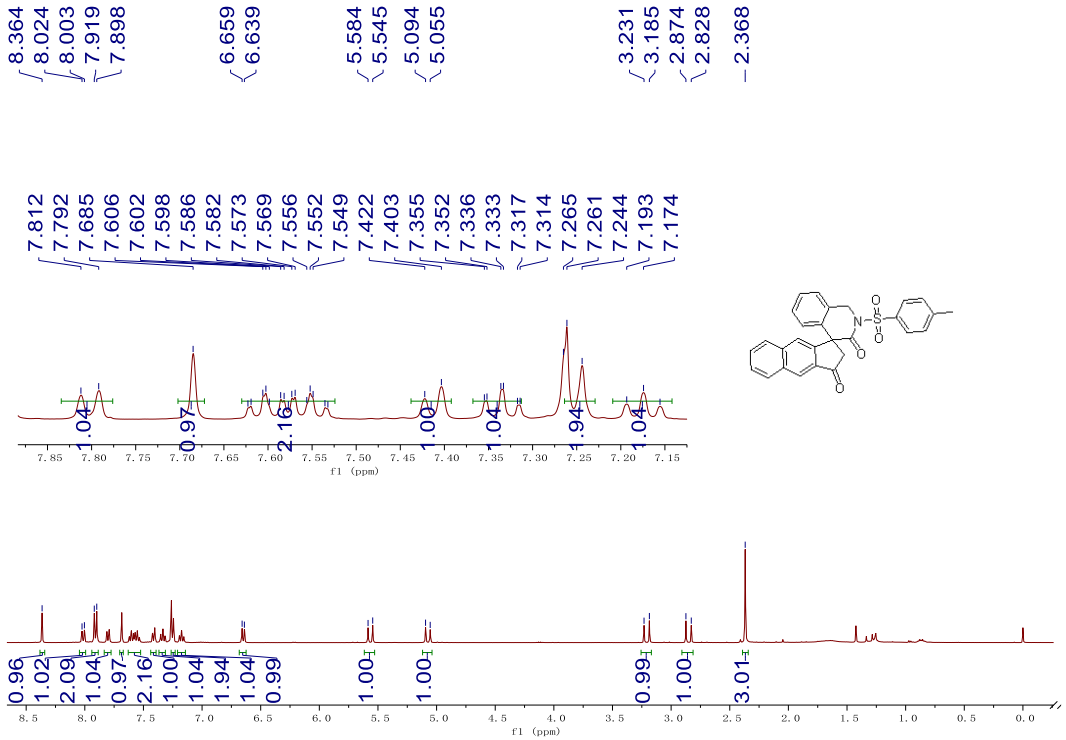
Compound **5l** ^{13}C NMR 100 MHz



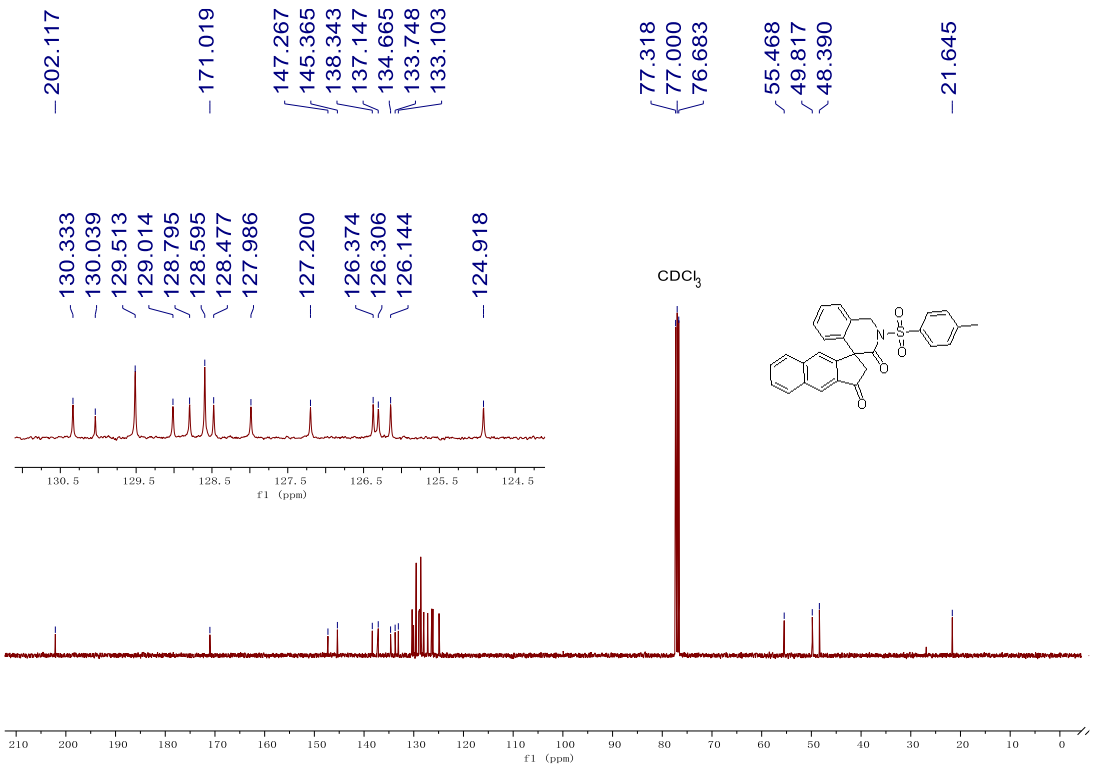
Compound **5la** ^{1}H NMR 400 MHz



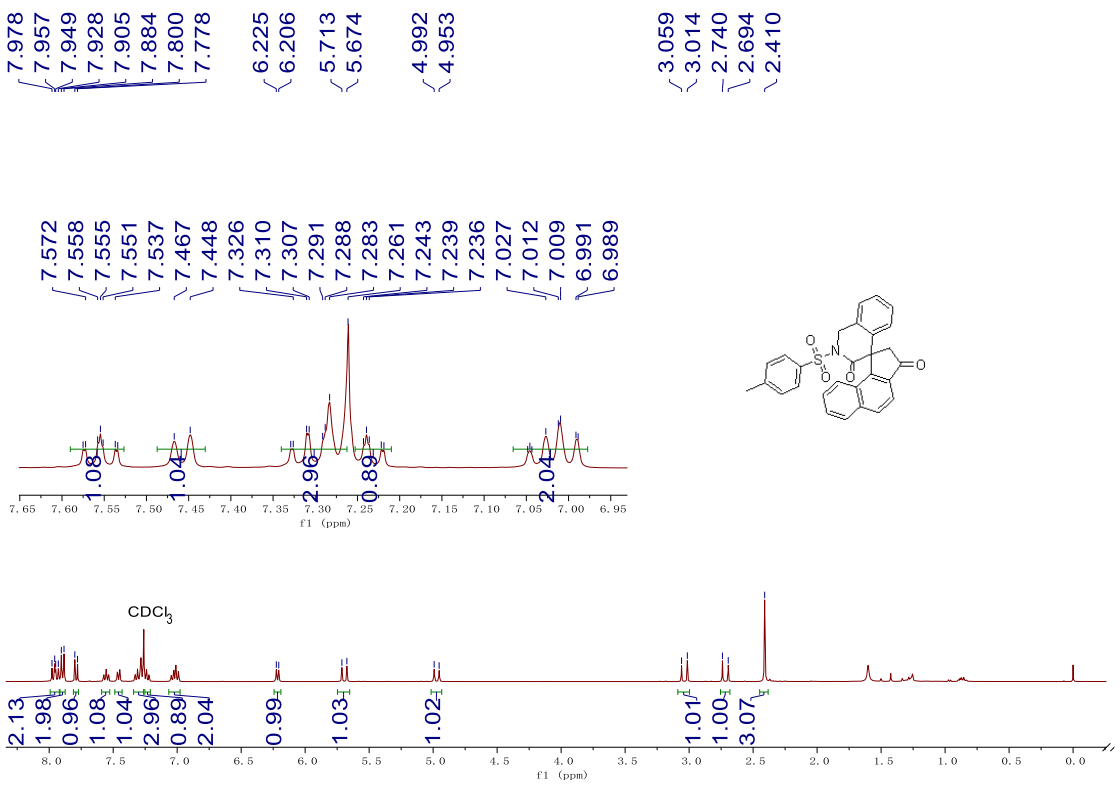
Compound **5la** ^{13}C NMR 100 MHz



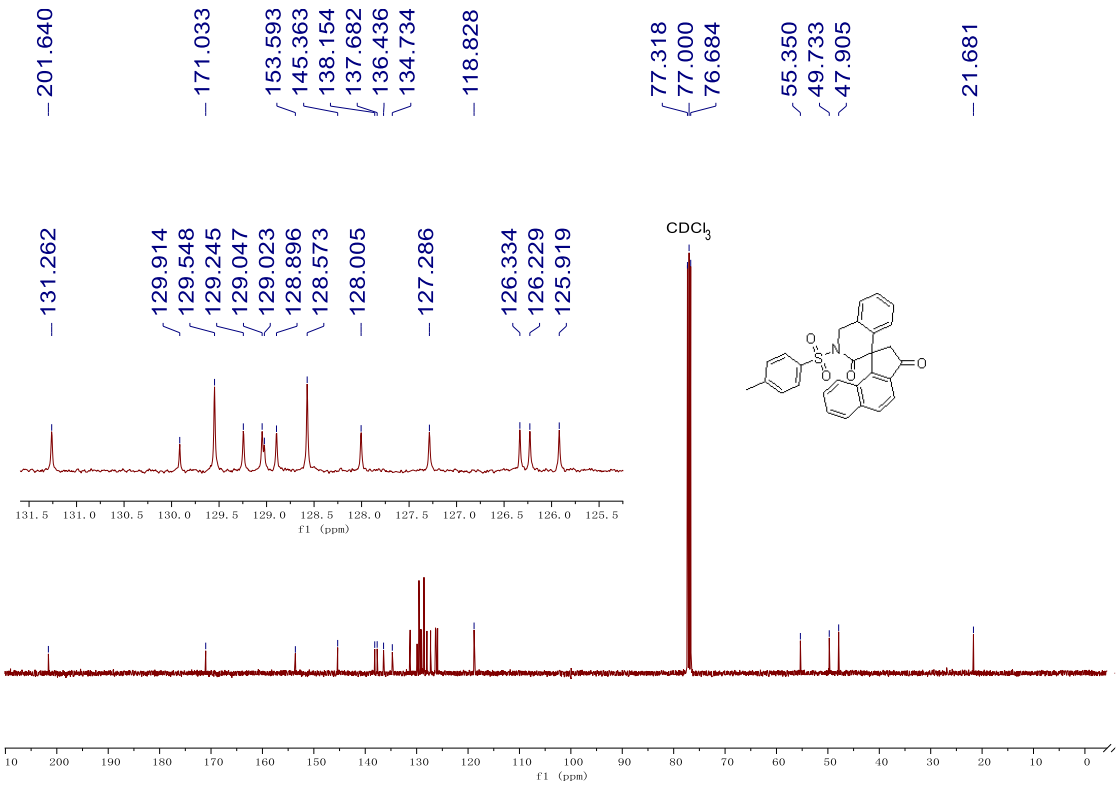
Compound **5m** ^{1}H NMR 400 MHz



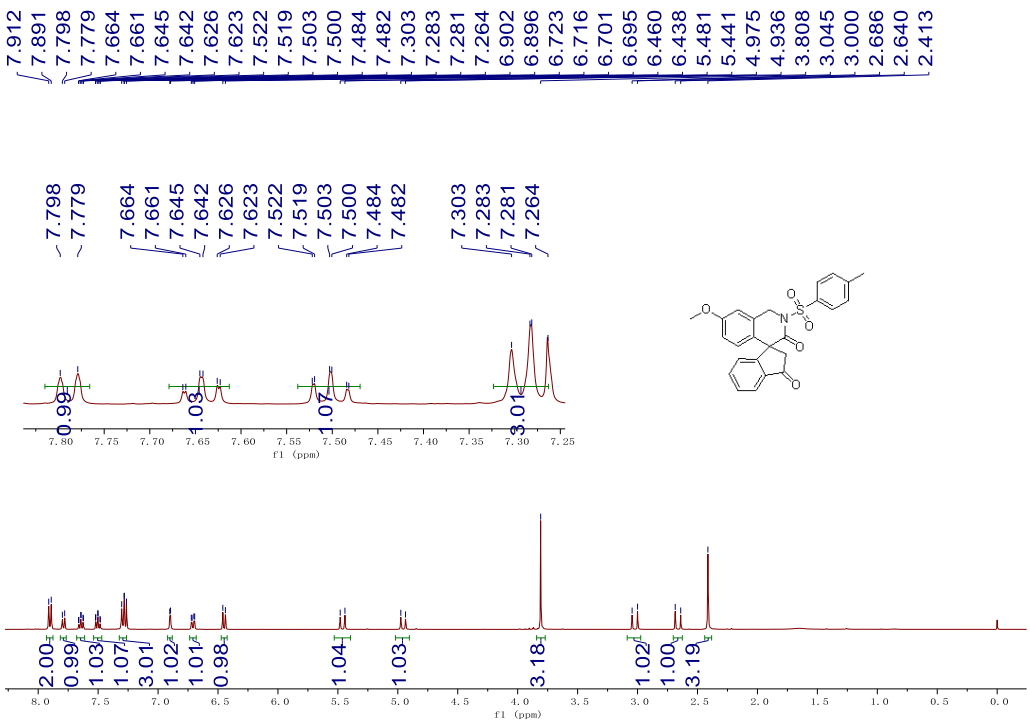
Compound **5m** ^{13}C NMR 100 MHz



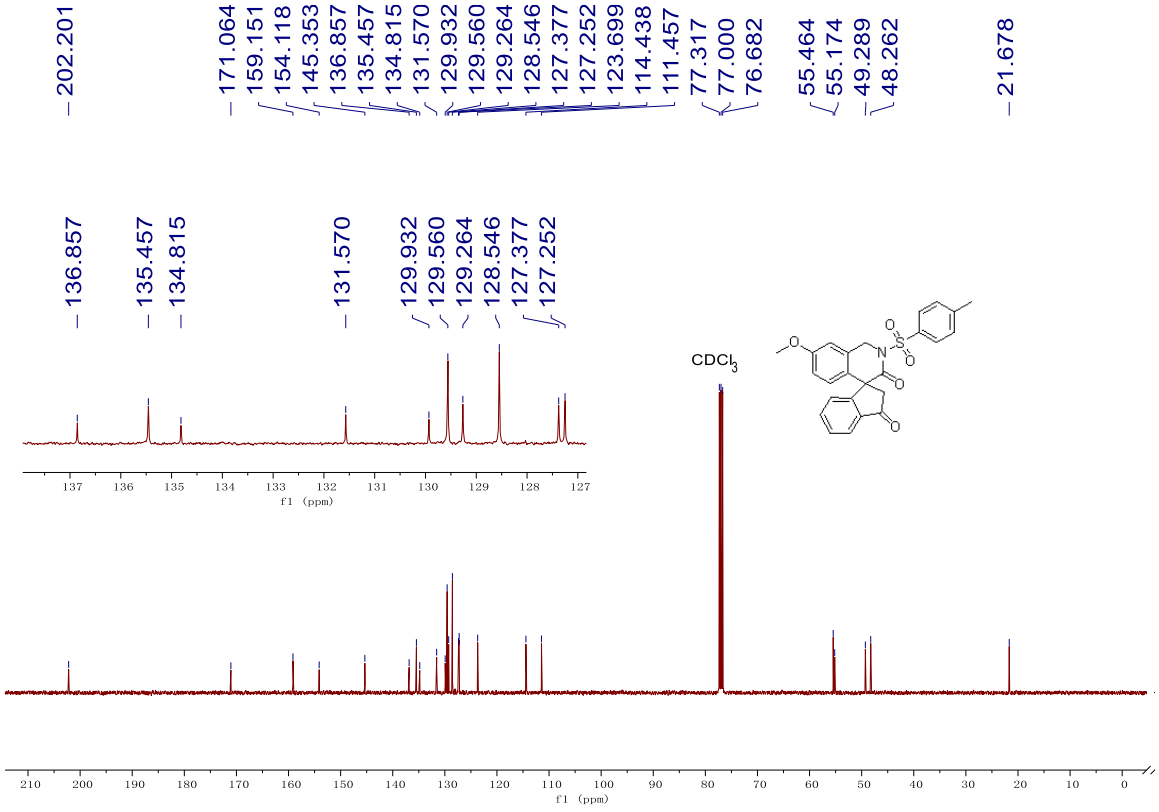
Compound **5ma** ^{1}H NMR 400 MHz



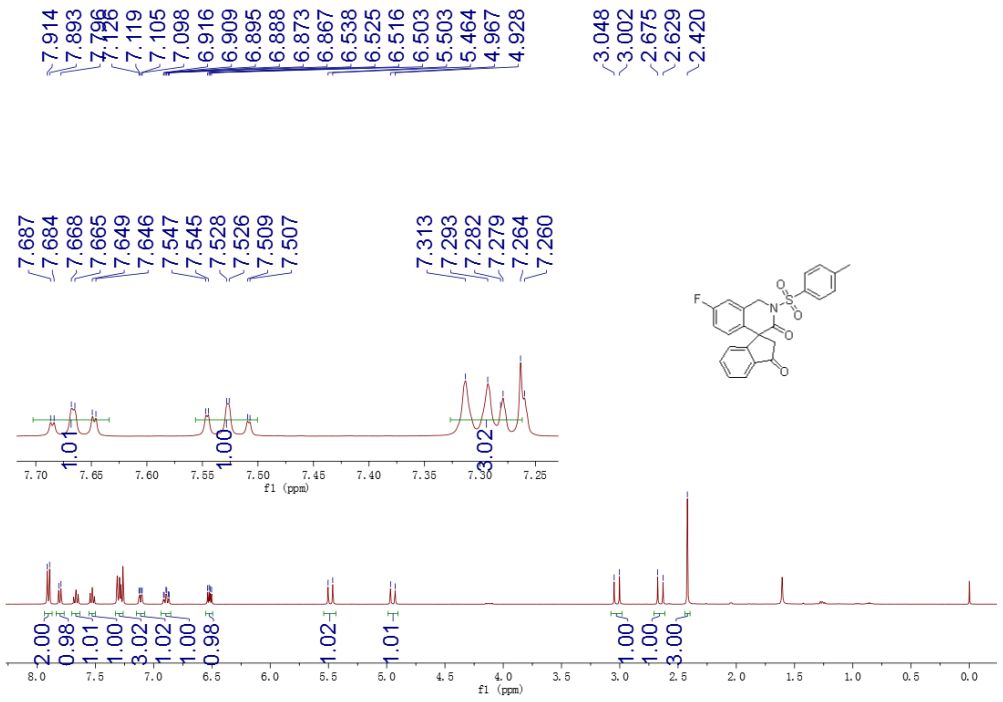
Compound **5ma** ^{13}C NMR 100 MHz



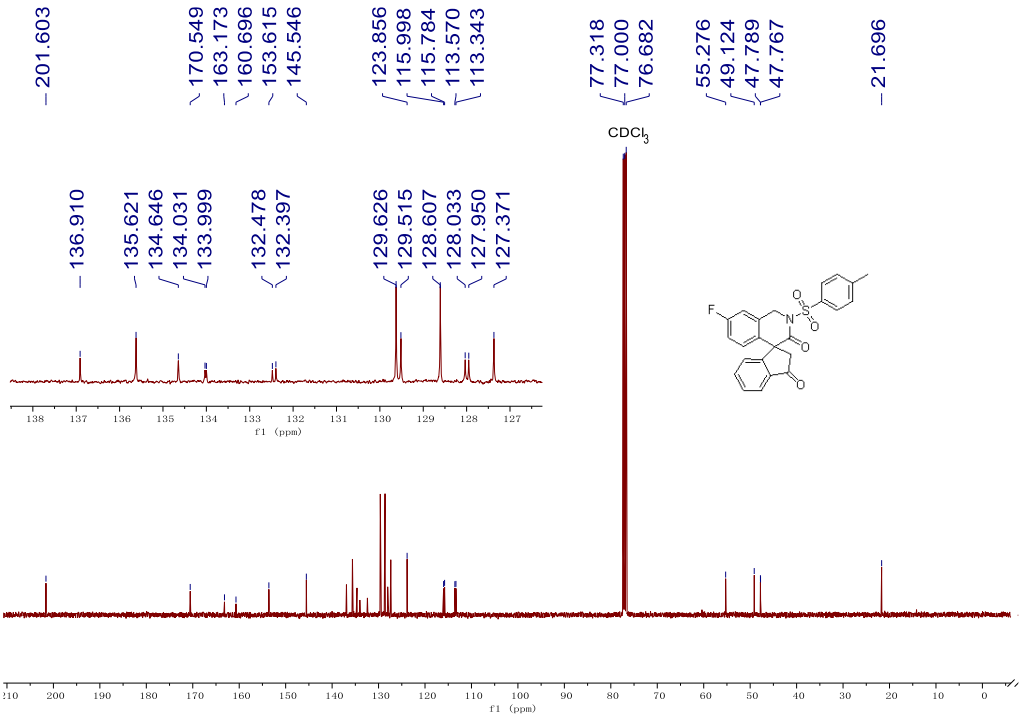
Compound **5n** ^{1}H NMR 400 MHz



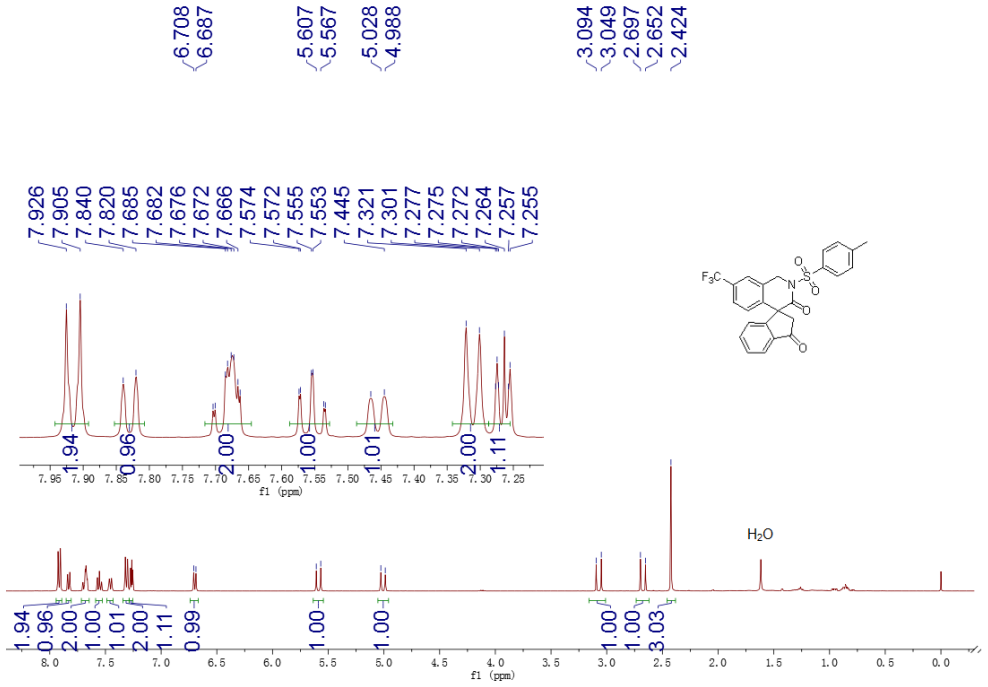
Compound **5n** ^{13}C NMR 100 MHz



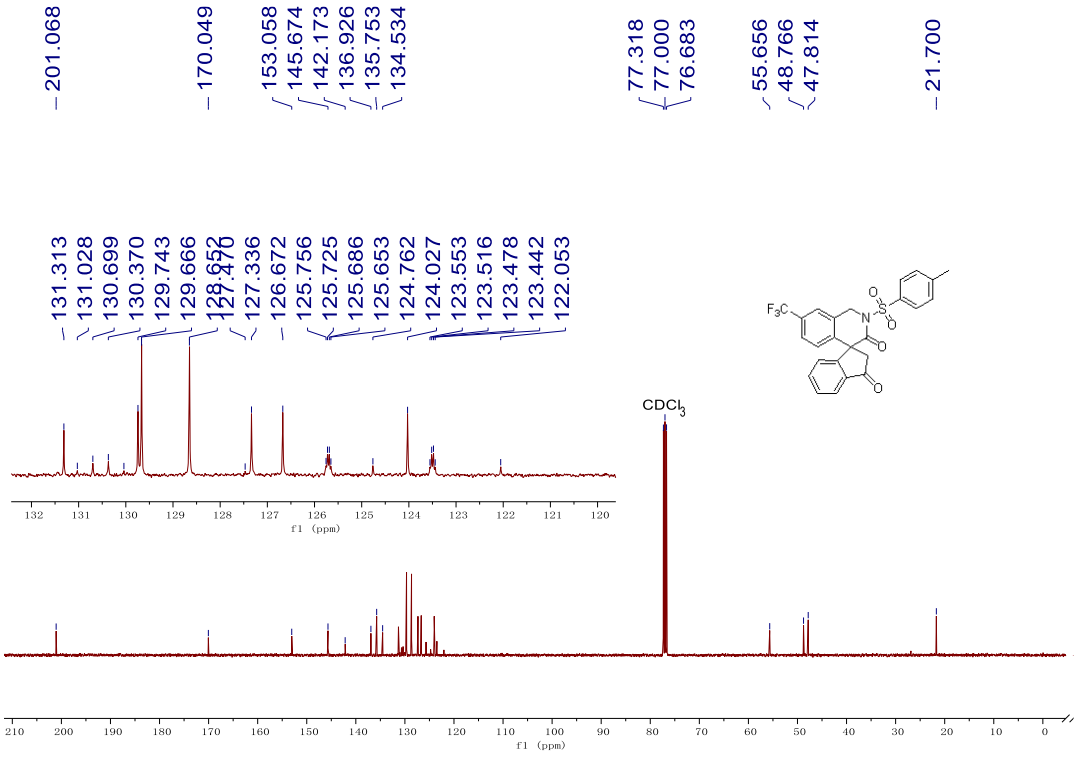
Compound **5o** ^{1}H NMR 400 MHz



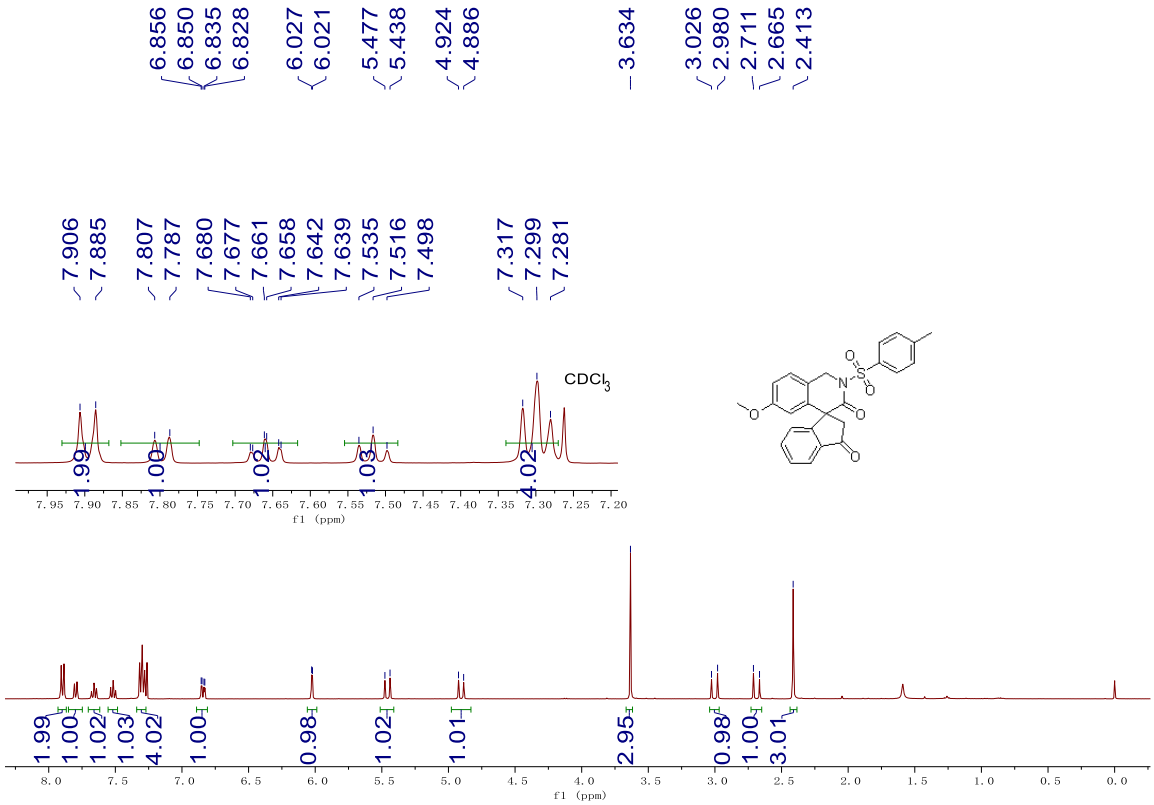
Compound **5o** ^{13}C NMR 100 MHz



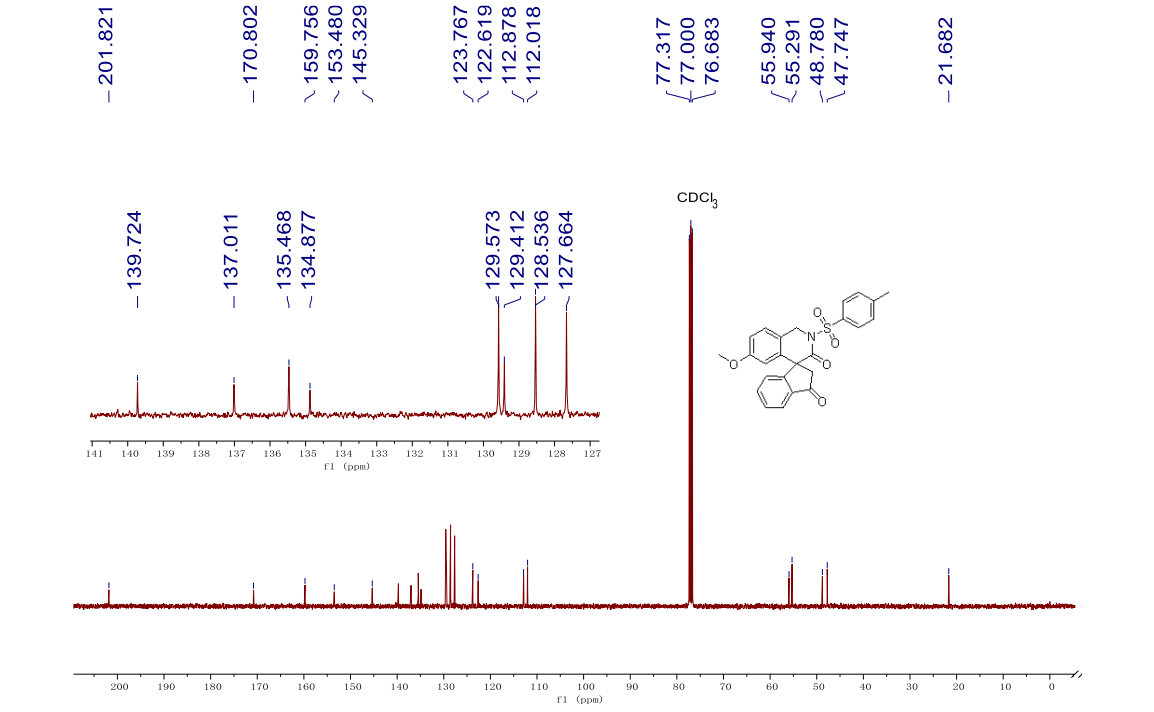
Compound **5p** ^{1}H NMR 400 MHz



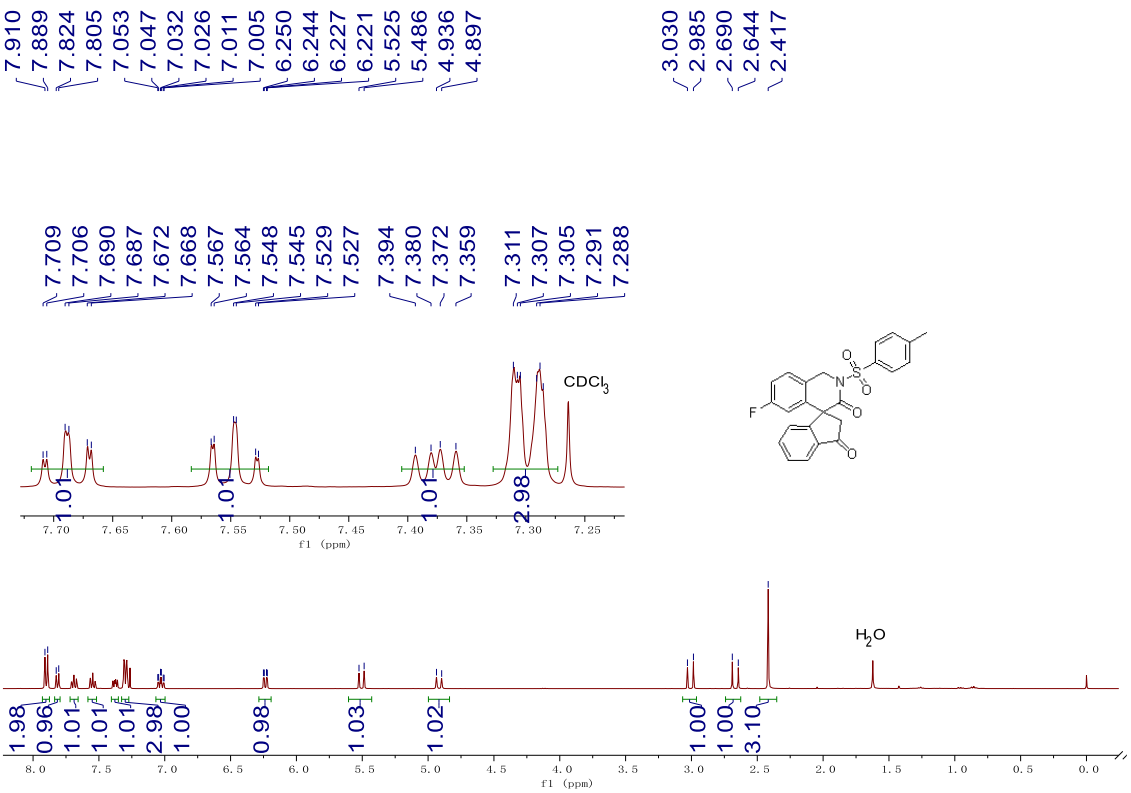
Compound **5p** ^{13}C NMR 100 MHz



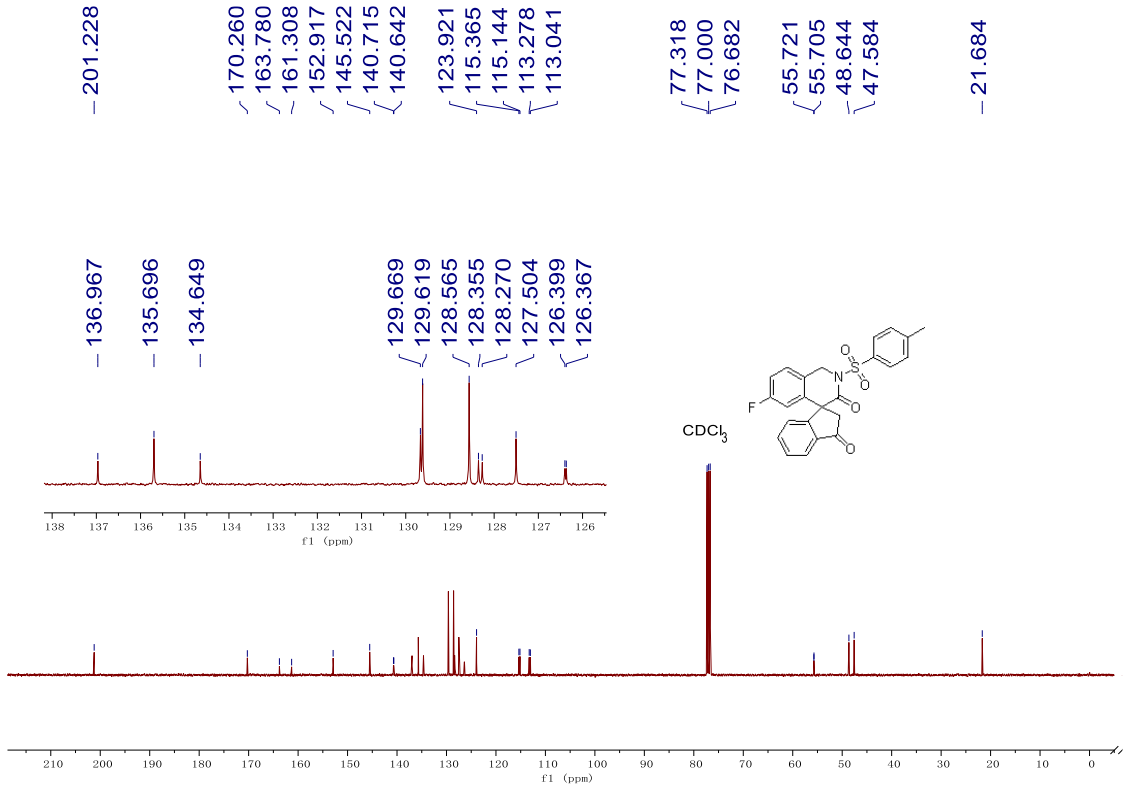
Compound **5q** ^{1}H NMR 400 MHz



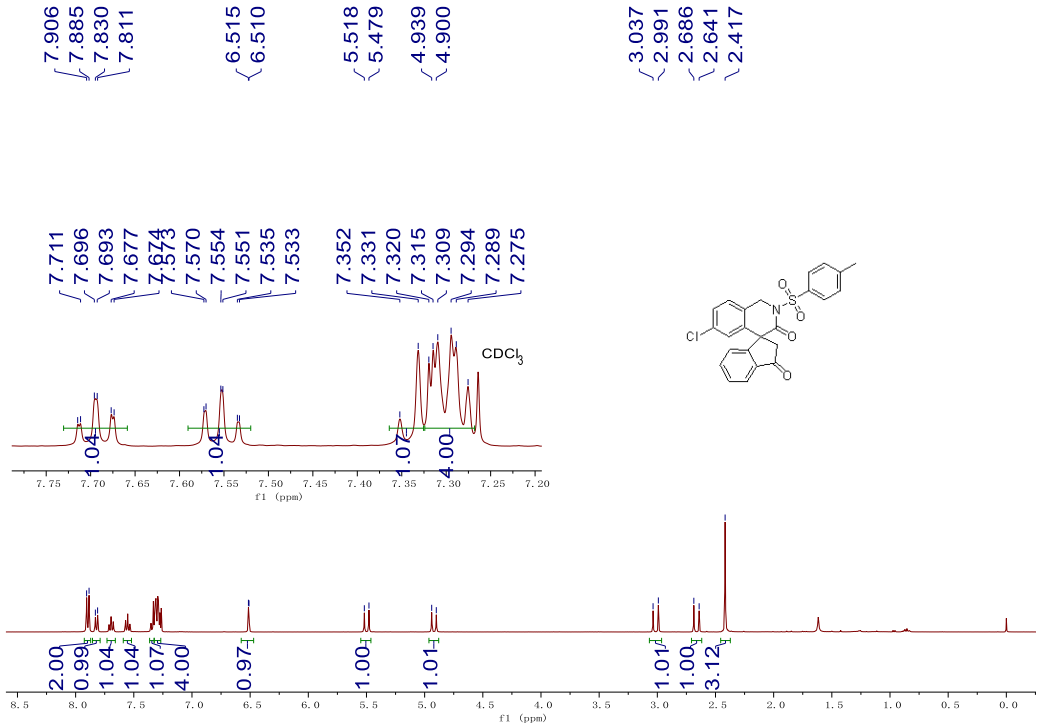
Compound **5q** ^{13}C NMR 100 MHz



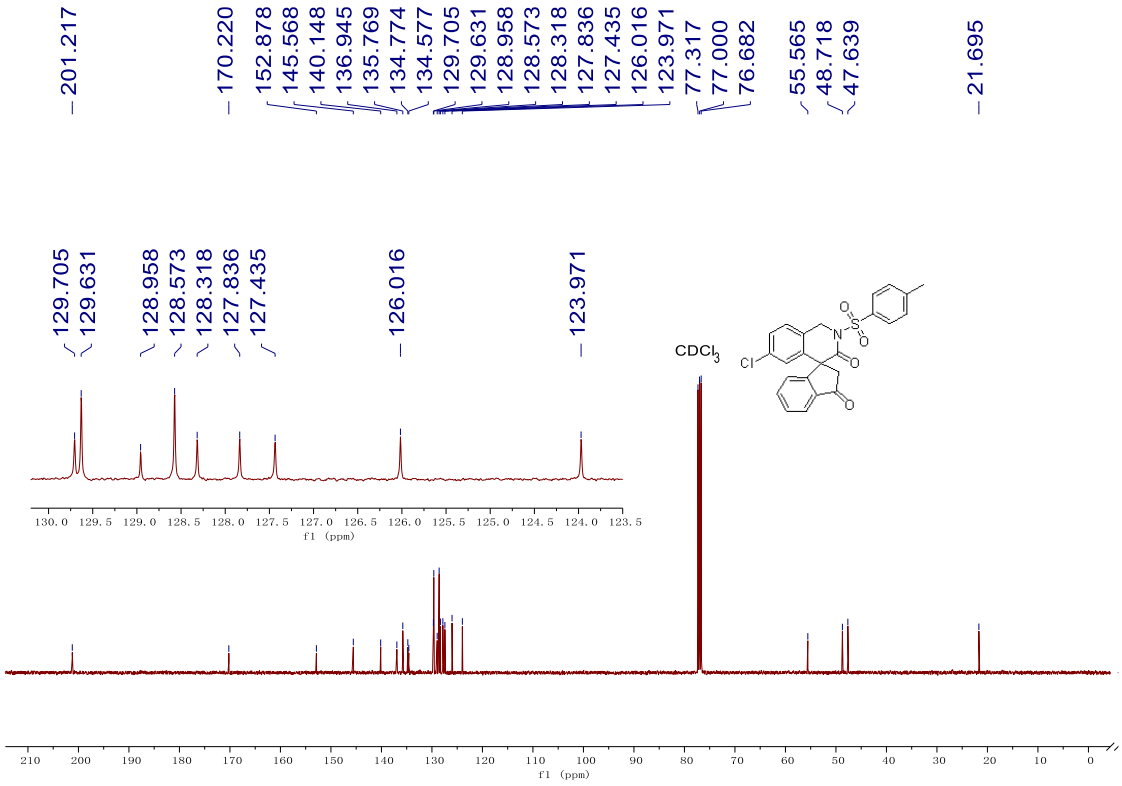
Compound **5r** ^{1}H NMR 400 MHz



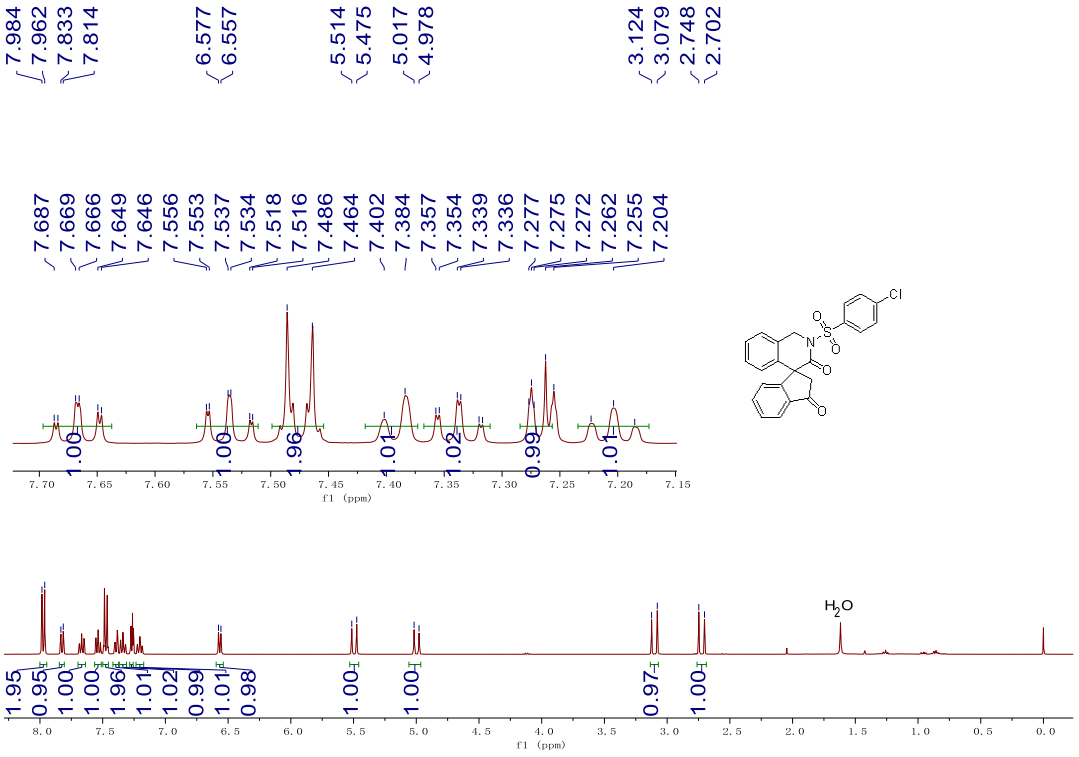
Compound **5r** ^{13}C NMR 100 MHz



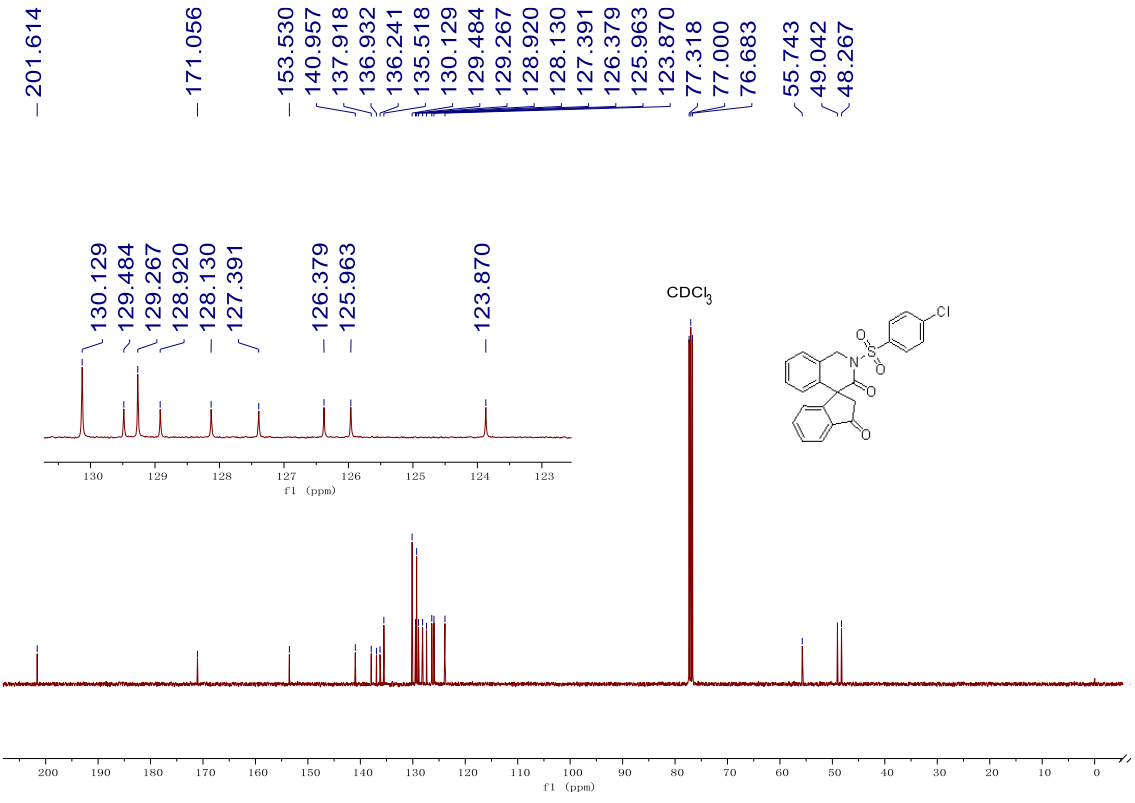
Compound **5s** ^{1}H NMR 400 MHz



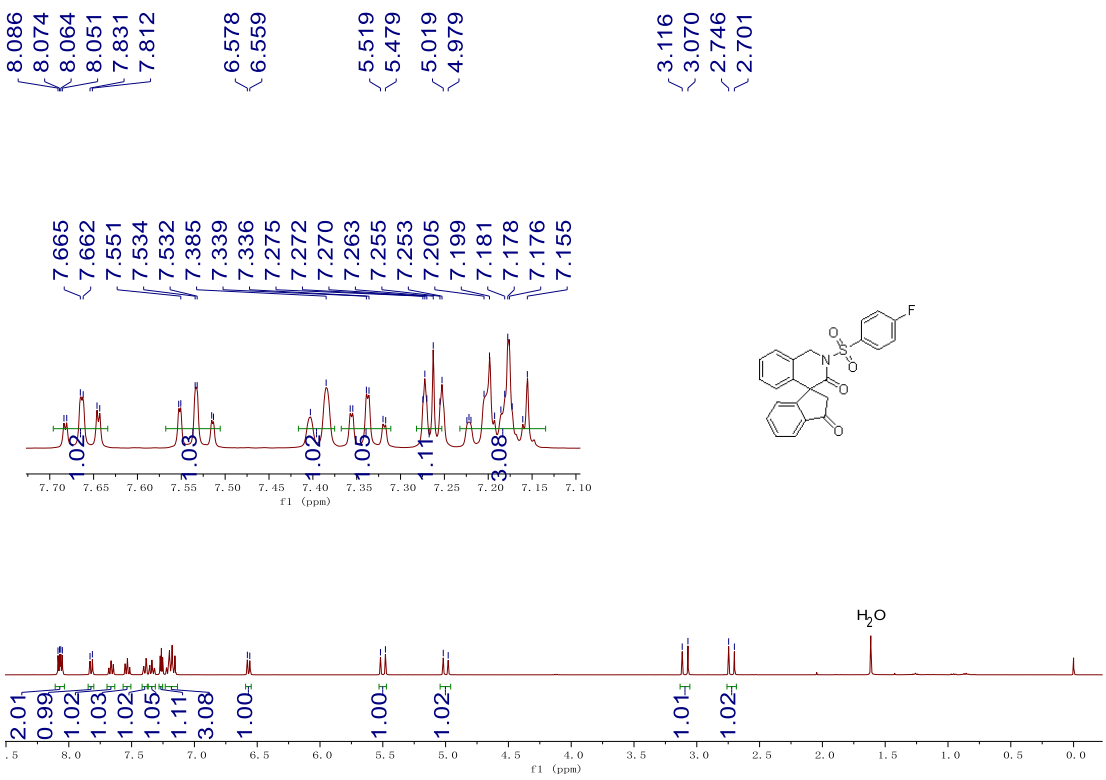
Compound **5s** ^{13}C NMR 100 MHz



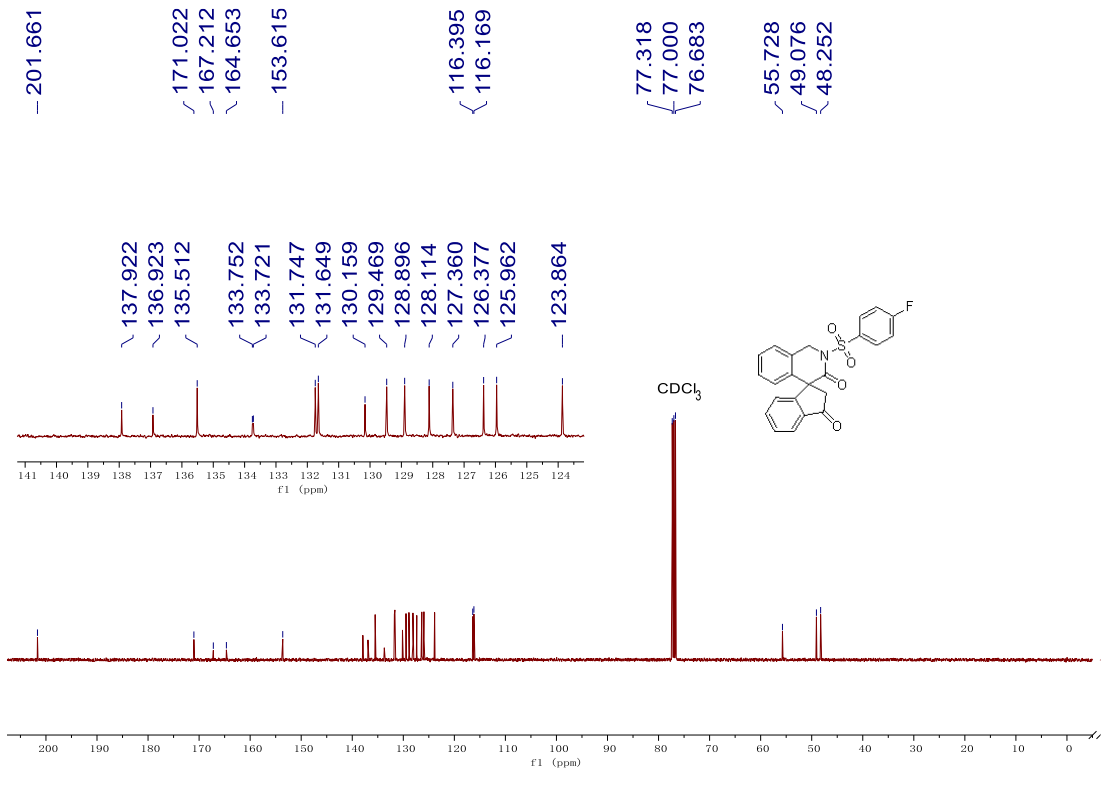
Compound **5t** ^{1}H NMR 400 MHz



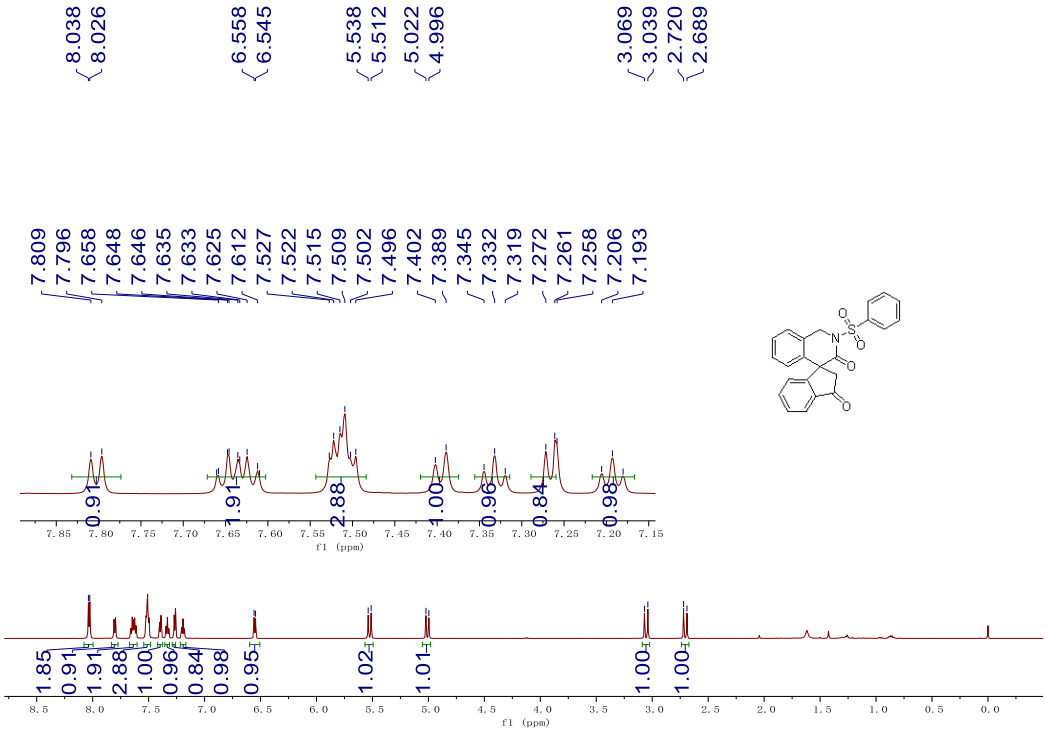
Compound **5t** ^{13}C NMR 100 MHz



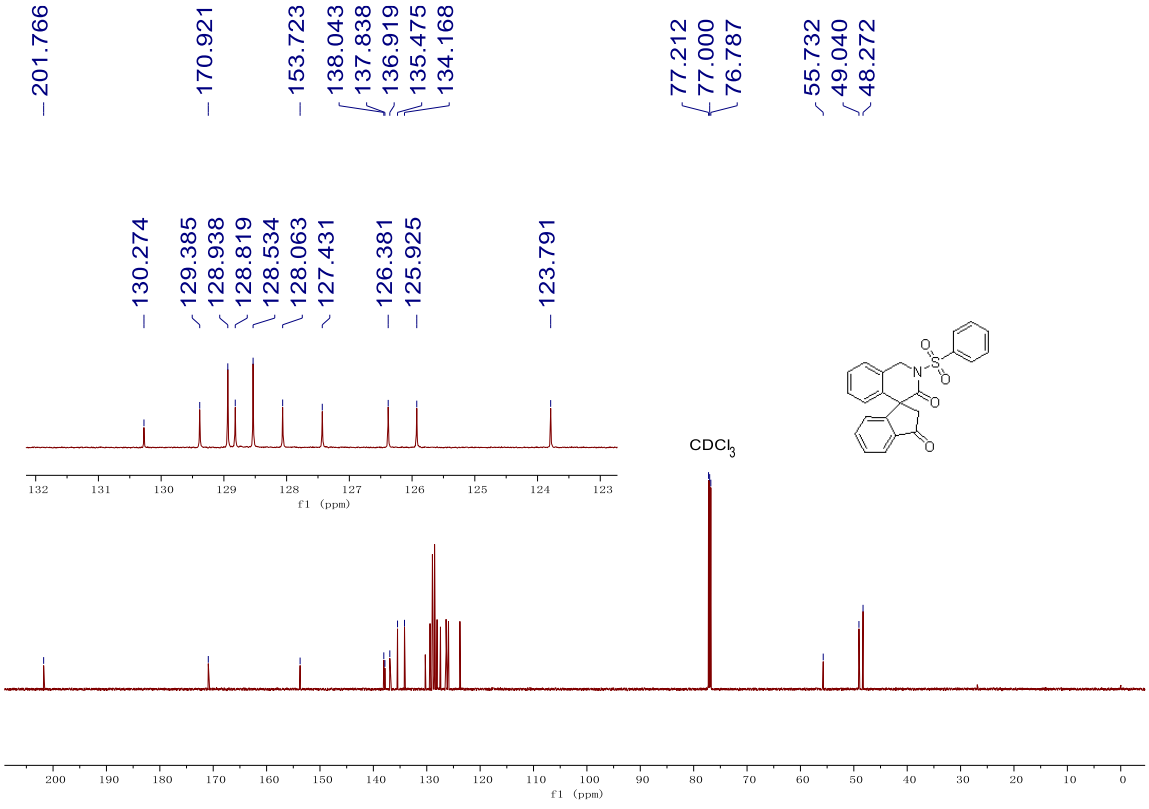
Compound **5u** ^{1}H NMR 400 MHz



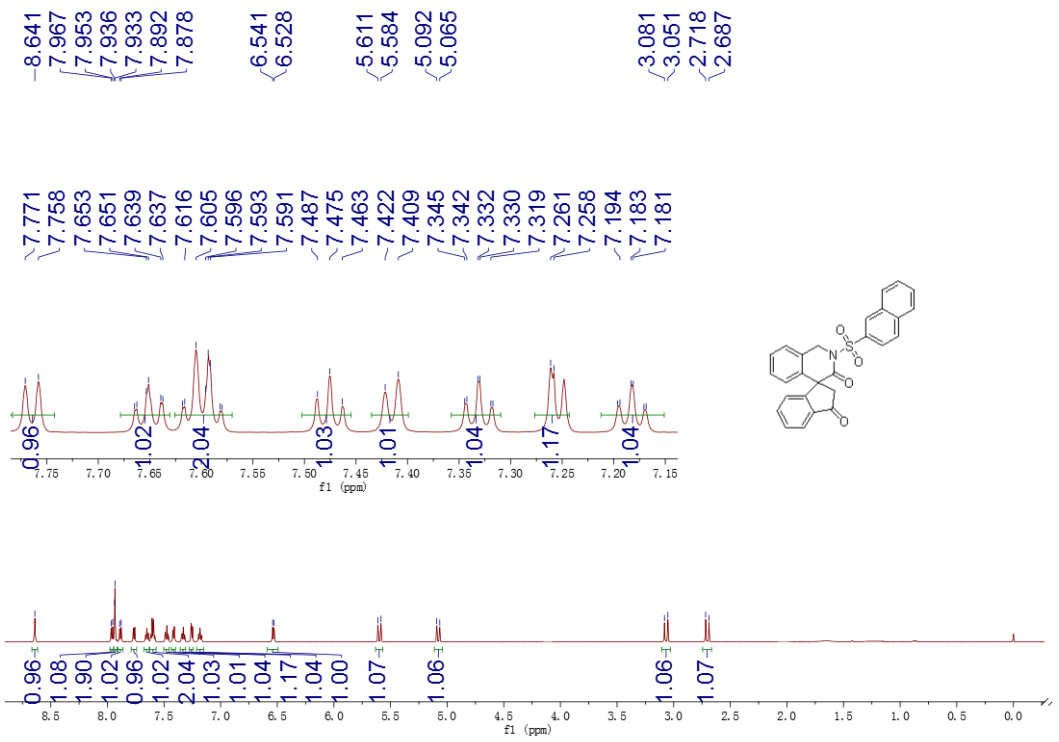
Compound **5u** ^{13}C NMR 100 MHz



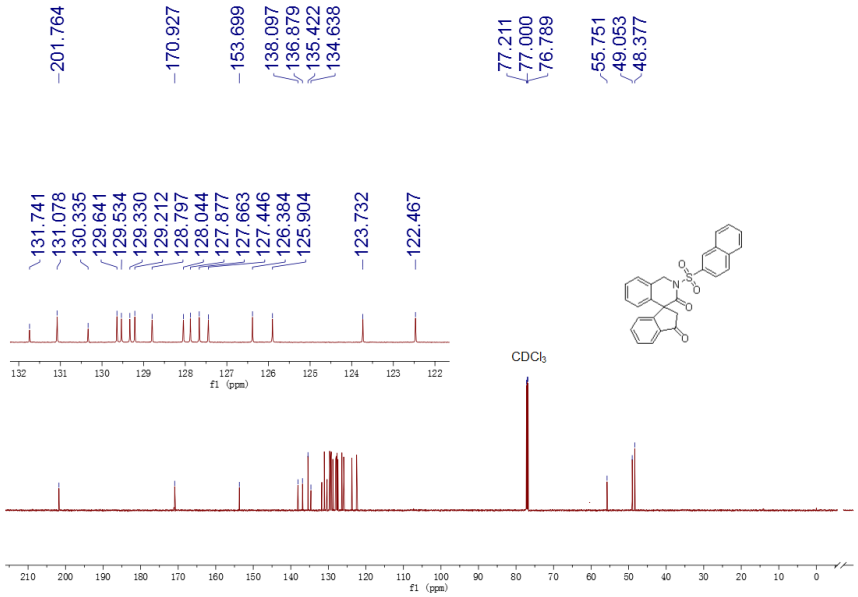
Compound **5v** ^{1}H NMR 600 MHz



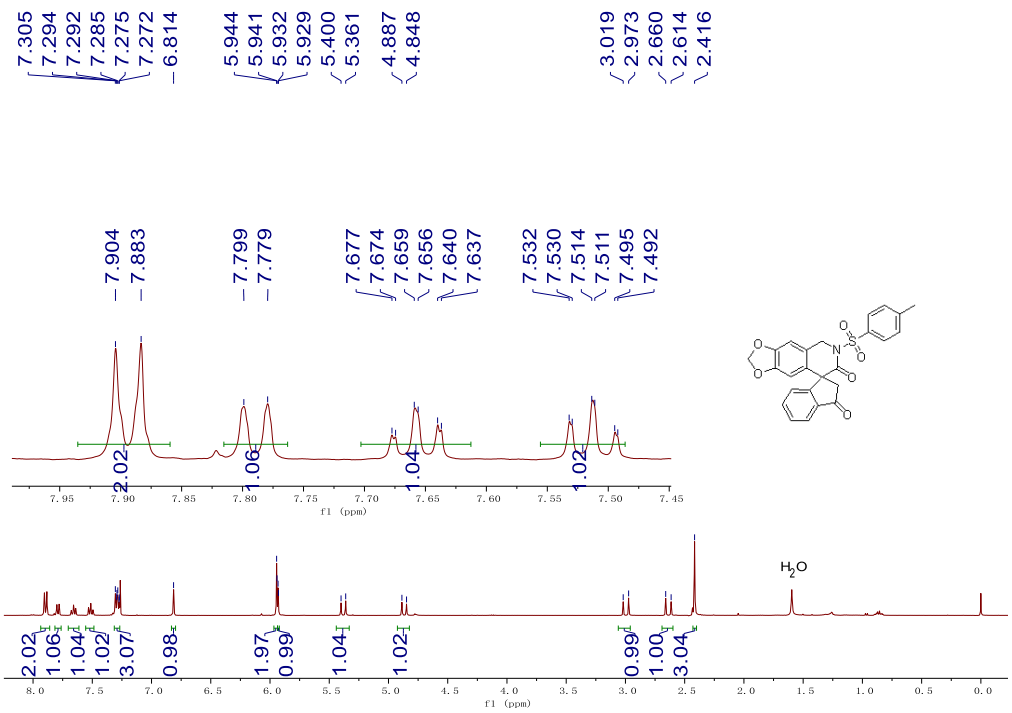
Compound **5v** ^{13}C NMR 150 MHz



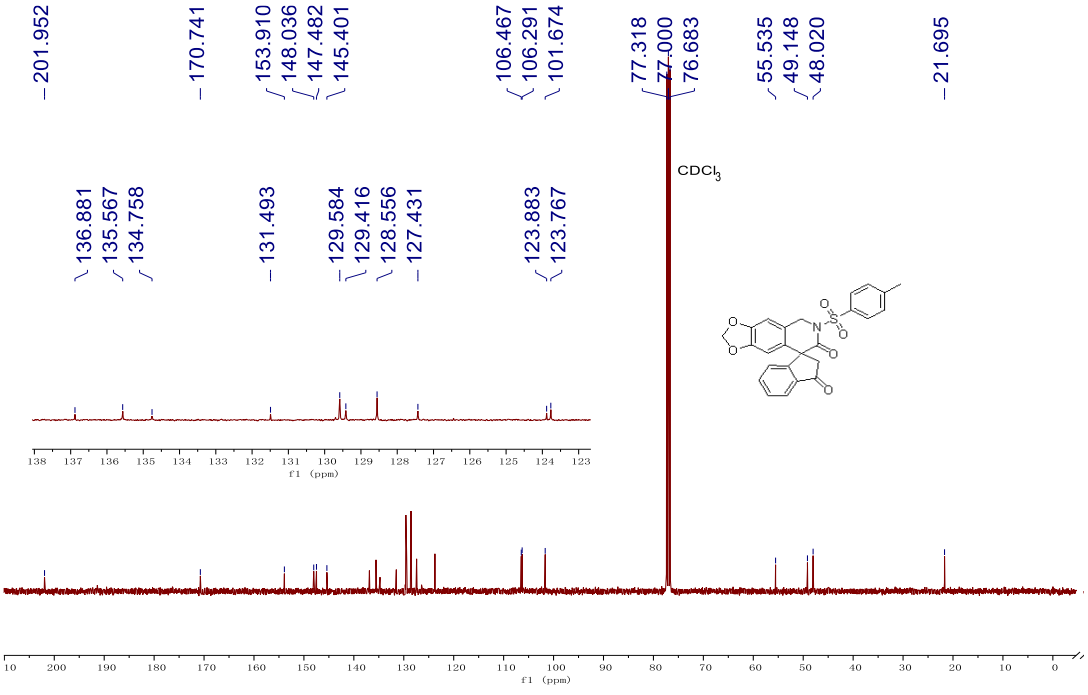
Compound **5w** ^{1}H NMR 600 MHz



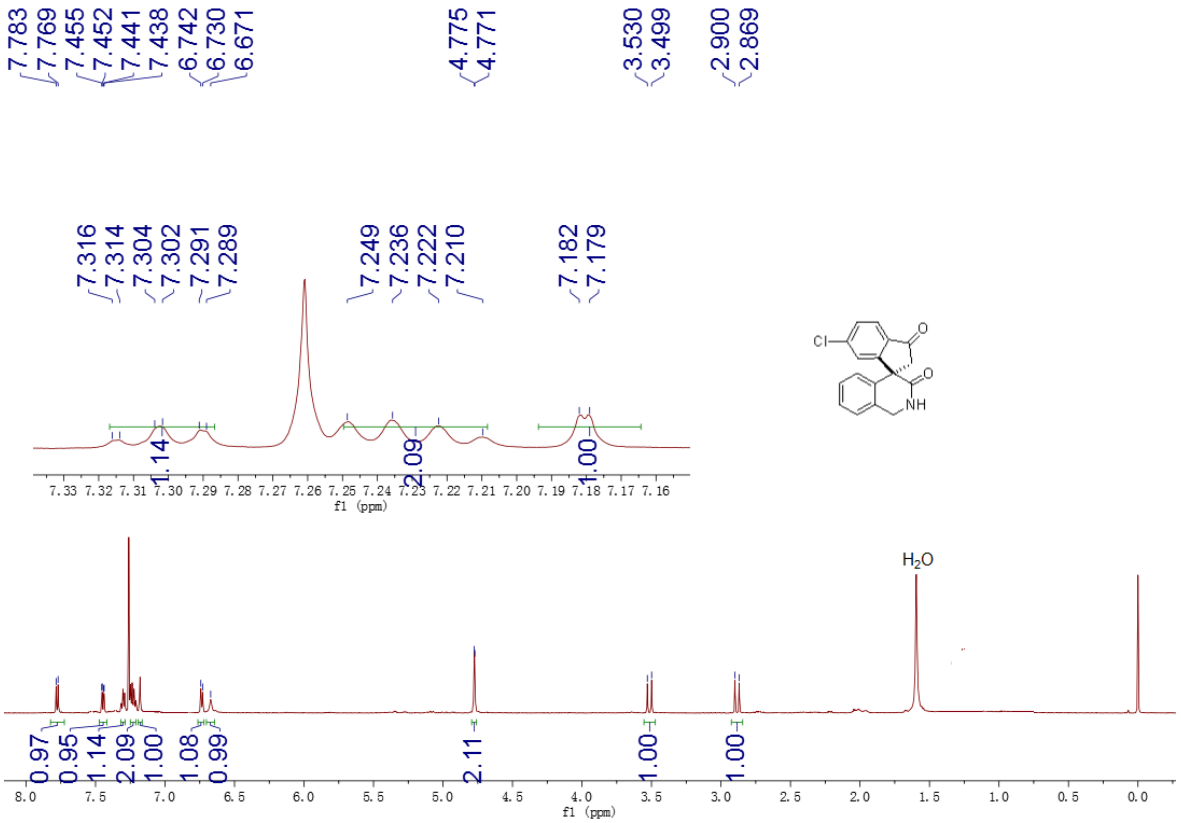
Compound **5w** ^{13}C NMR 150 MHz



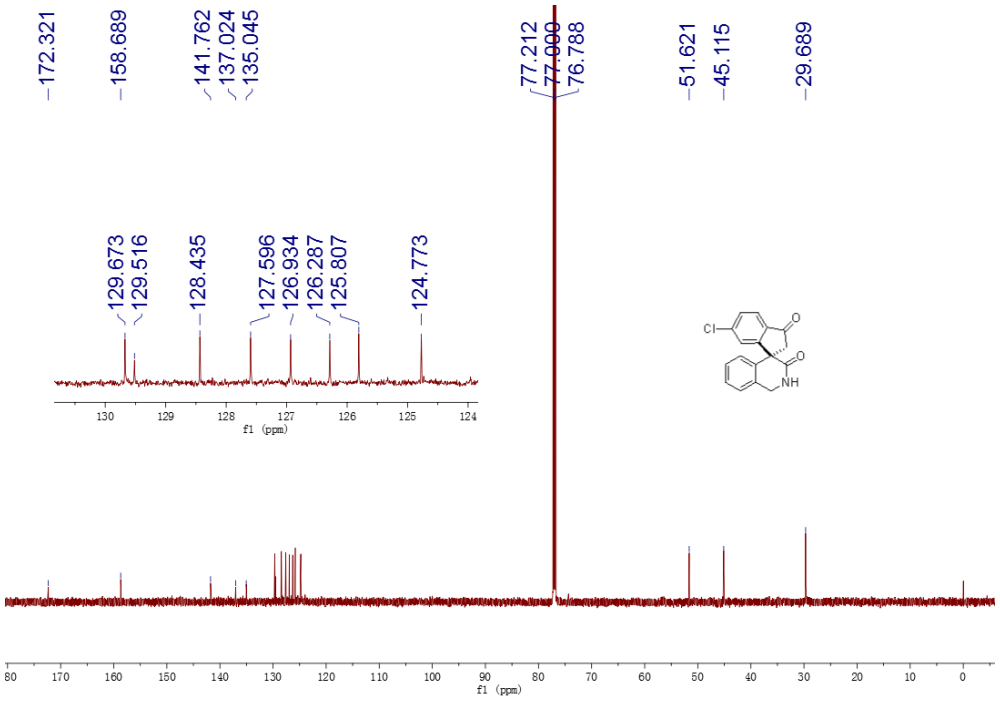
Compound **5x** ^{1}H NMR 400 MHz



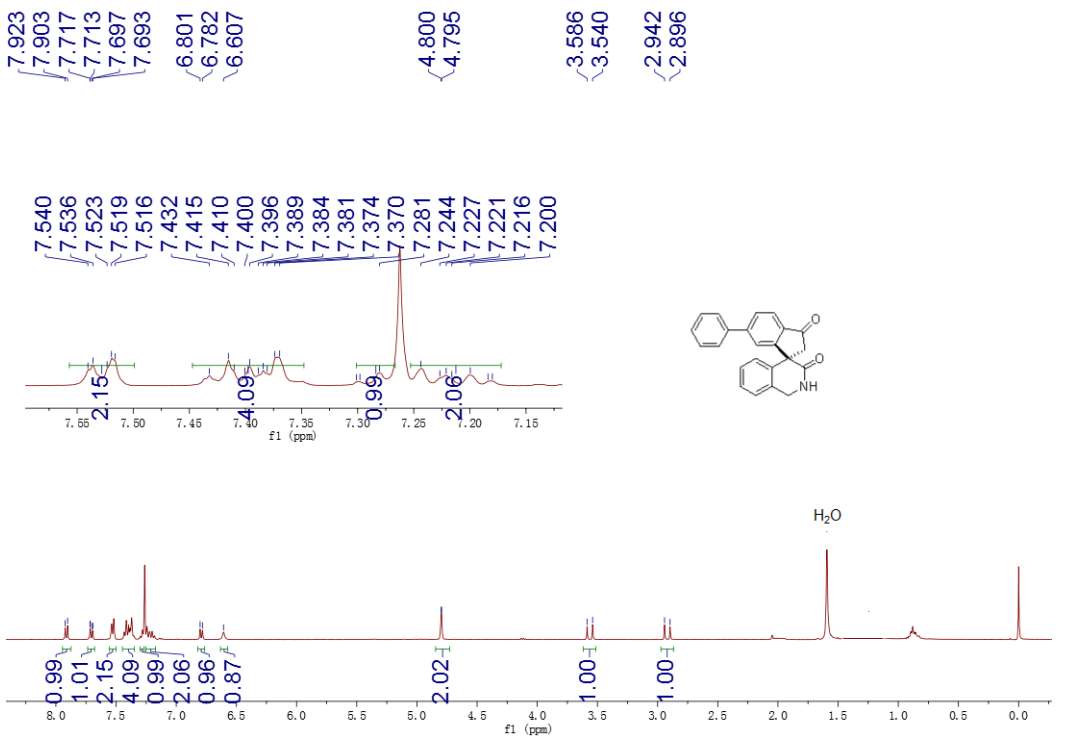
Compound **5x** ^{13}C NMR 100 MHz



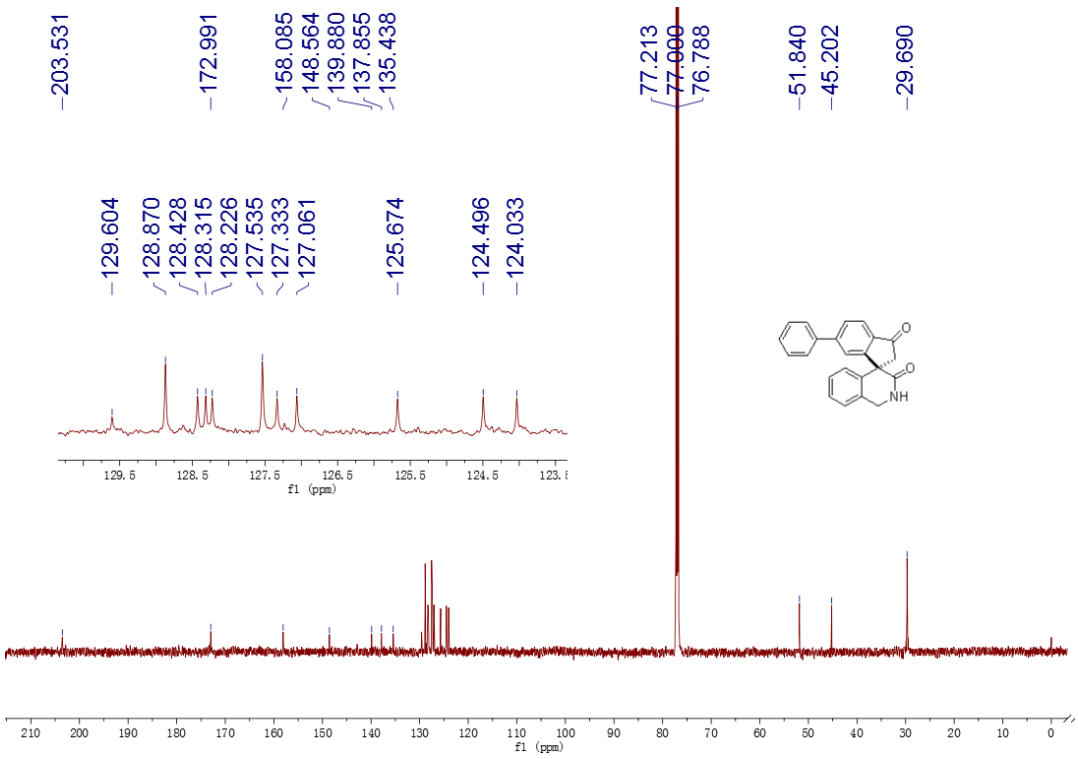
Compound **6a** ^{1}H NMR 600 MHz



Compound **6a** ^{13}C NMR 150 MHz



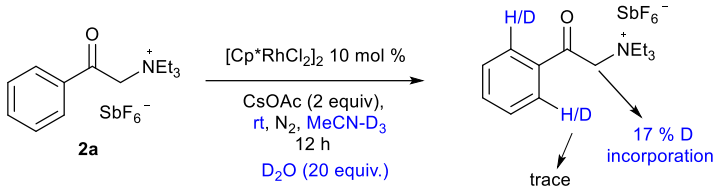
Compound **6b** ^{1}H NMR 400 MHz



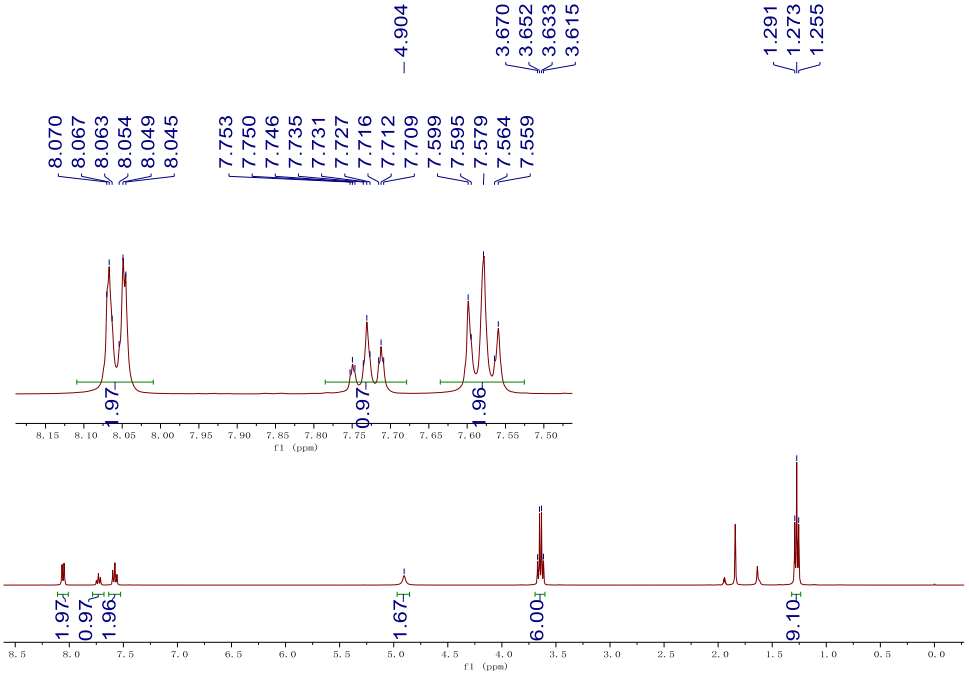
Compound **6b** ^{13}C NMR 150 MHz

Mechanistic studies

Deuterium labelling experiment:

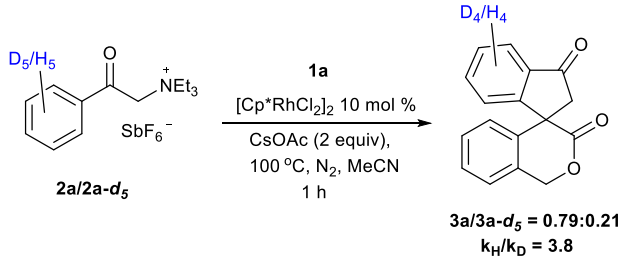


Quaternary-Ammonium compound **2a**-SbF_6^- (0.10 mmol), $[Cp^*RhCl_2]_2$ (0.01 mmol) and CsOAc (0.2 mmol) were added to a pressure tube, to which was added MeCN-*d*3 (1 mL) and D2O (40 μL) under N_2 atmosphere. The reaction mixture was stirred at rt for 12.0 h. After that, the mixture was characterized by 1H NMR spectroscopy.



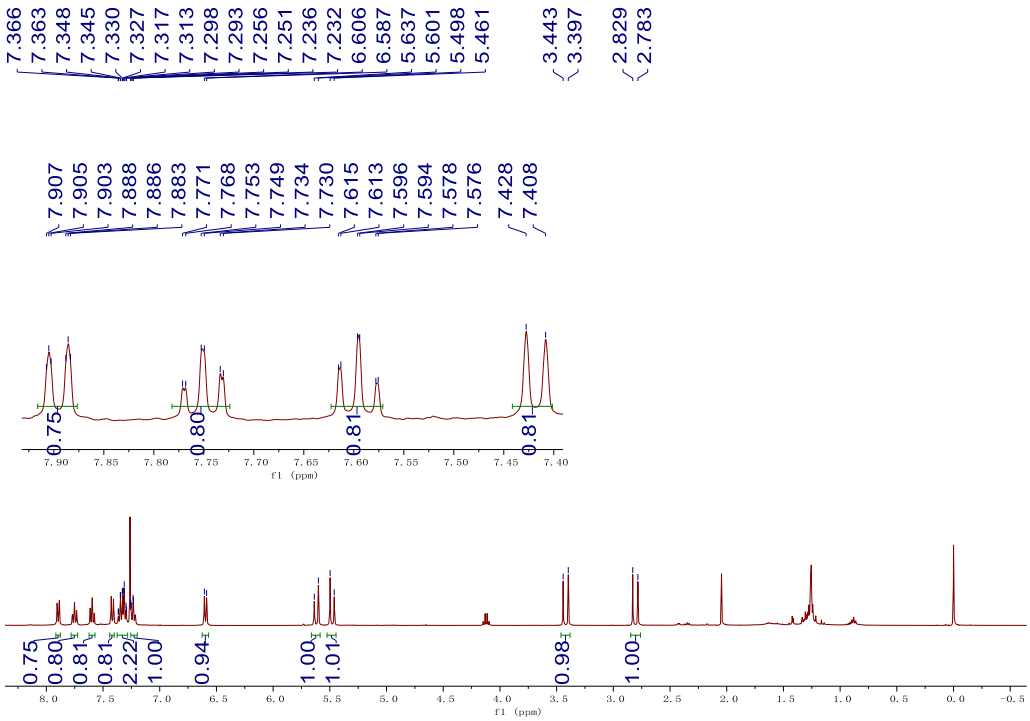
^{1}H NMR 400 MHz (CD_3CN)

Kinetic isotope effect experiment (3a)



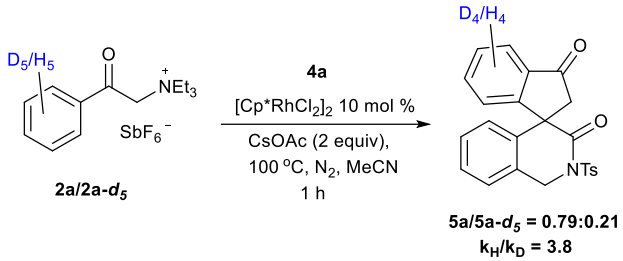
Two pressure tubes were separately charged with **2a**-SbF_6 and **2a**-SbF_6-*d*5 (0.32 mmol), and to each

tube were added CsOAc (0.4 mmol), $[Cp^*RhCl_2]_2$ (0.02 mmol), the diazo compound **1a** (0.2 mmol) and acetonitrile (4 mL) under nitrogen atmosphere. The two reaction mixtures were stirred side by side at 100 °C for one hour. The resulting mixtures in the two tubes were combined and the solvent was rapidly removed under reduced pressure. The residue was purified by silica gel chromatography using (PE/EA, 3:1) to afford the product. The KIE value was determined to be *k*H/*k*D = 3.8 on the basis of 1H NMR analysis.



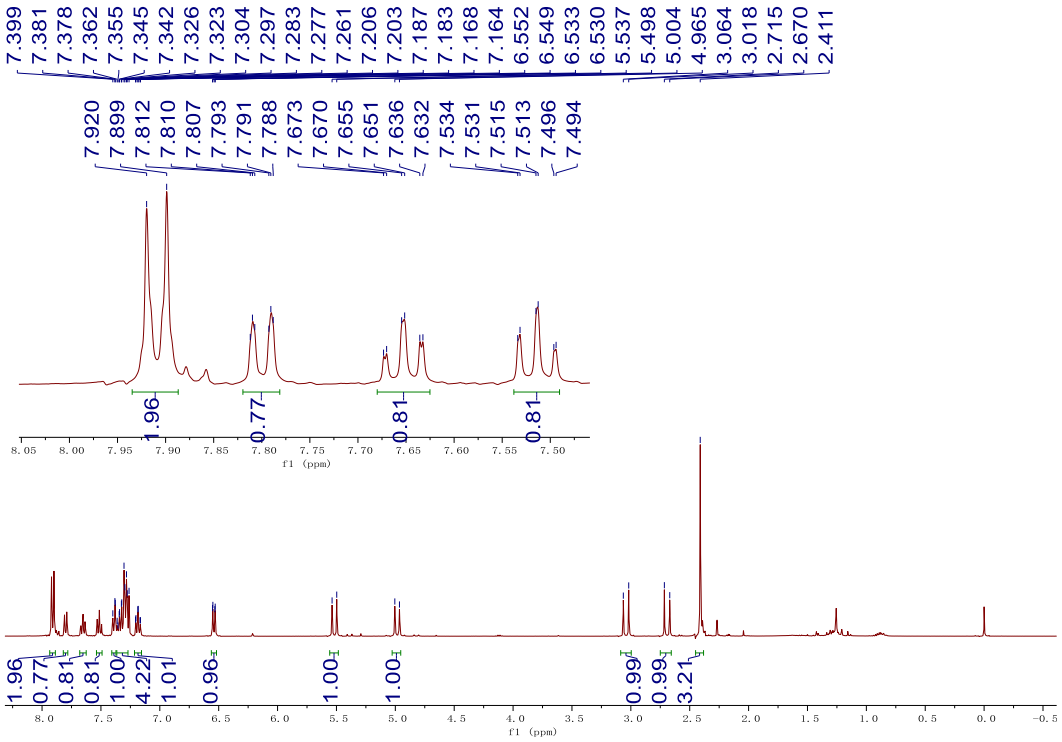
KIE (**3a/3a-d$_3$**) ^{1}H NMR 400 MHz ($CDCl_3$)

Kinetic isotope effect experiment (5a)



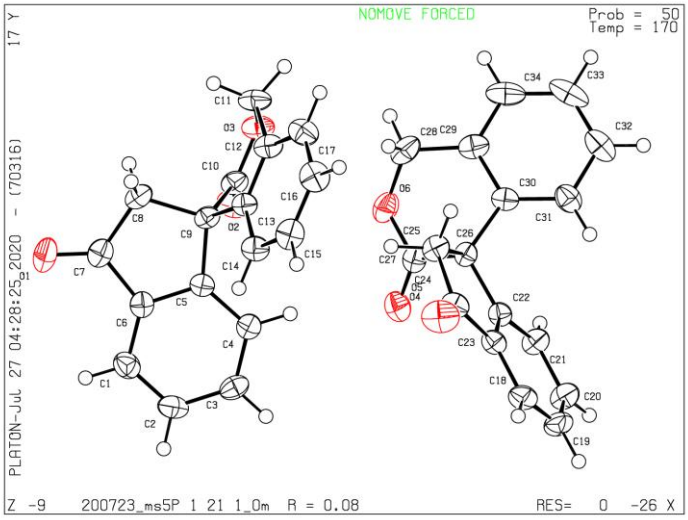
Two pressure tubes were separately charged with **2a**-SbF_6 and **2a**-SbF_6-*d*5 (0.32 mmol), and to each tube were added CsOAc (0.4 mmol), $[Cp^*RhCl_2]_2$ (0.02 mmol), the diazo compound **4a** (0.2 mmol) and acetonitrile (4 mL) under nitrogen atmosphere. The two reaction mixtures were stirred side by side at 100 °C for one hour. The resulting mixtures in the two tubes were

combined and the solvent was rapidly removed under reduced pressure. The residue was purified by silica gel chromatography using (PE/EA, 3:1) to afford the product. The KIE value was determined to be *k*H/*k*D = 3.8 on the basis of 1H NMR analysis.



KIE (**5a/5a-d$_3$**) ^{1}H NMR 400 MHz ($CDCl_3$)

Crystal Structure of 3a



Datablock: 200723_ms586_004_2_0m

Bond precision: C-C = 0.0049 A Wavelength=0.71073

Cell: a=11.849(4) b=8.586(3) c=12.730(5)
alpha=90 beta=94.872(14) gamma=90
Temperature: 170 K

	Calculated	Reported
Volume	1290.4(8)	1290.3(8)
Space group	P 21	P 1 21 1
Hall group	P 2yb	P 2yb
Moiety formula	C17 H12 O3	C17 H12 O3
Sum formula	C17 H12 O3	C17 H12 O3
Mr	264.27	264.27
Dx,g cm-3	1.360	1.360
Z	4	4
Mu (mm-1)	0.093	0.093
F000	552.0	552.0
F000'	552.29	
h,k,lmax	15,10,16	15,10,16
Nref	5644[3016]	5216
Tmin,Tmax	0.986,0.991	0.377,0.746
Tmin'	0.962	

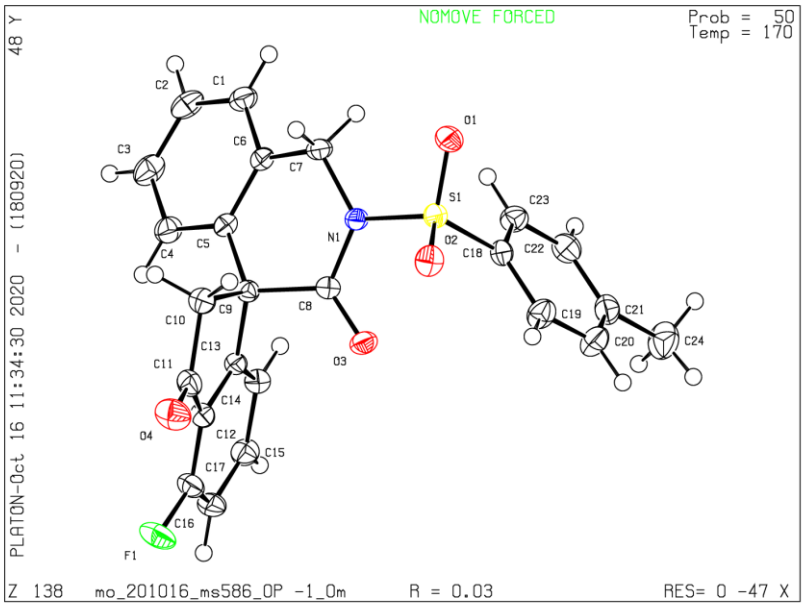
Correction method= # Reported T Limits: Tmin=0.377 Tmax=0.746
AbsCorr = NONE

Data completeness= 1.73/0.92 Theta(max)= 27.016

R(reflections)= 0.0791(4868) wR2(reflections)= 0.1783(5216)

S = 1.086 Npar= 361

Crystal Structure of 5i



Datablock: mo_201016_ms586_052_1_0m

Bond precision: C-C = 0.0019 A Wavelength=0.71073

Cell: a=7.1439(14) b=10.734(2) c=14.451(3)
alpha=72.259(9) beta=81.410(9) gamma=72.811(10)
Temperature: 170 K

	Calculated	Reported
Volume	1006.2(4)	1006.2(4)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C24 H18 F N O4 S	C24 H18 F N O4 S
Sum formula	C24 H18 F N O4 S	C24 H18 F N O4 S
Mr	435.45	435.45
Dx,g cm-3	1.437	1.437
Z	2	2
Mu (mm-1)	0.203	0.203
F000	452.0	452.0
F000'	452.48	
h,k,lmax	9,13,18	9,13,18
Nref	4425	4409
Tmin,Tmax	0.962,0.976	0.720,0.746
Tmin'	0.954	

Correction method= # Reported T Limits: Tmin=0.720 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.996 Theta(max)= 27.080

R(reflections)= 0.0322(4172) wR2(reflections)= 0.0871(4409)

S = 1.043 Npar= 281