Electronic Supplementary Material (ESI) for ChemComm.

Rhodium-catalyzed divergent dehydroxylation/alkenylation of hydroxyisoindolinones with vinylene carbonate

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Electronic Supplementary Information

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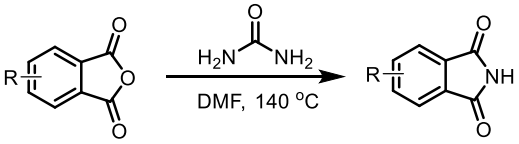
A. General information

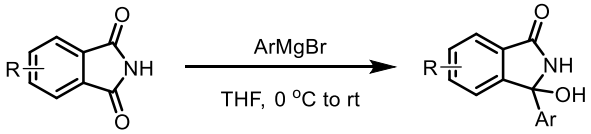
All reagents were used from commercial received unless otherwise noted. Analytical thin-layer chromatography was performed with 0.25 mm coated commercial silica gel plates (TLC Silica Gel 60 F_{254}); visualization of the developed chromatogram was performed by fluorescence. Flash Chromatography was performed with silica gel (300-400 mesh). Proton-1 nuclear magnetic resonance (^{1}H NMR) data were acquired at 400 MHz on a Bruker Ascend 400 (400 MHz) spectrometer, and chemical shifts are reported in delta (δ) units, in parts per million (ppm) downfield from tetramethylsilane. Splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet, coupling constants *J* are quoted in Hz. Carbon-13 nuclear magnetic resonance (^{13}C NMR) data were acquired at 100 MHz on a Bruker Ascend 400 spectrometer, chemical shifts are reported in ppm relative to the center line of a triplet at 77.0 ppm for $CDCl_3$, 39.5 ppm for DMSO. Fluorine-19 nuclear magnetic resonance (^{19}F NMR) data were acquired at 376 MHz on a Bruker Ascend 400 spectrometer. High resolution mass spectra were acquired on a Bruker Daltonics MicroTof-Q II mass spectrometer.

B. Preparation of substrates



1-I **1-II**

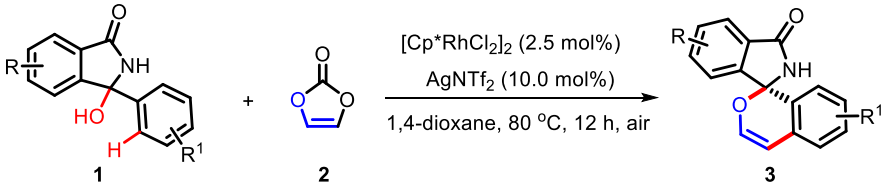
A round bottom flask equipped with a stir bar was charged with the mixture of substituted phthalic anhydride **1-I** (5.0 mmol), urea (3.0 mmol) and DMF (5.0 mL). After heating to reflux to a certain temperature, the solid portion was melted, stirring was started, and heating was continued. The solid was completely melted and heated to 140 ℃ for 3 h. Saturated NH_4Cl solution (3 × 20 mL) was added and the mixture was extracted with EtOAc. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated on a rotary evaporator. The residue was purified by column chromatography on silica gel to obtain the corresponding products **1-II**[1-3].



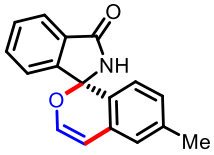
1-II **1**

A round bottom flask equipped with a stir bar was charged with the Magnesium turnings (3.3 mmol) were suspended in dry THF (5.0 mL) under argon, and further activated with a single crystall of iodine, then add the tetrahydrofuran solution of bromobenzenes (3.0 mmol) at 40 ℃ was stirred for 2 h. Phthalimide (1.0 mmol) was suspended in THF(5.0 mL) under argon at 0 °C, Grignard solution was cooled to room temperature and added dropwise to the phthalimide suspension. Resulting mixture was stirred at room temperature for 5 h. Saturated NH_4Cl solution (3 × 20 mL) was added and the mixture was extracted with EtOAc. The combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated on a rotary evaporator. The solid residue was recrystallized from CH_2Cl_2 and hexane to obtain corresponding products **1**[4-13].

C. Reaction results of cyclization reaction

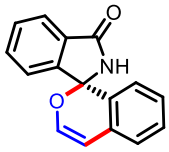


A pressure tube equipped with a stir bar was charged with $[Cp^*RhCl_2]_2$ (3.1 mg, 2.5 mol%), 3-hydroxy-3-(*p*-tolyl)isoindolin-1-one **1** (47.8 mg, 0.2 mmol), $AgNTf_2$ (7.8 mg, 10.0 mol%), vinylene carbonate **2** (51.6 mg, 0.6 mmol) in 1,4-dioxane (1.0 mL), The reaction mixture was stirred at 80 °C for 12 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford the products **3**.



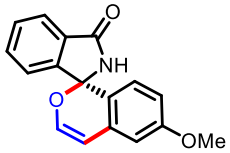
6-methylspiro[isochromene-1-1'-isoindolin]-3'-one (3a)

White solid (42.0 mg, 80% yield). PE/EA = 3:1, R_f = 0.40. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.2 Hz, 1H), 7.66 – 7.49 (m, 3H), 7.18 (s, 1H), 6.97 (s, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 6.71 (d, *J* = 5.7 Hz, 1H), 6.65 (d, *J* = 7.9 Hz, 1H), 6.00 (d, *J* = 5.7 Hz, 1H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 147.1, 143.8, 139.5, 133.1, 130.6, 130.2, 129.6, 128.3, 125.2, 124.7, 124.3, 123.7, 123.7, 104.8, 90.1, 21.1. HRMS (ESI) m/z Calcd for $C_{17}H_{14}NO_2$ $[M+H]^+$ 264.1019, found 264.1015.



spiro[isochromene-1,1'-isoindolin]-3'-one (3b)

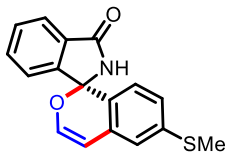
White solid (36.8 mg, 74% yield). PE/EA = 3:1, R_f = 0.39. ^{1}H NMR (400 MHz, DMSO) δ 9.91 (s, 1H), 7.77 – 7.69 (m, 1H), 7.67 – 7.58 (m, 2H), 7.50 (d, *J* = 6.9 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 7.6 Hz, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 5.7 Hz, 1H), 6.70 (d, *J* = 7.7 Hz, 1H), 6.13 (d, *J* = 5.7 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 168.0, 147.3, 144.0, 133.2, 130.5, 130.3, 129.8, 129.3, 128.1, 127.4, 124.1, 124.0, 123.4, 123.0, 104.4, 90.0. HRMS (ESI) m/z Calcd for $C_{16}H_{12}NO_2$ $[M+H]^+$ 250.0863, found 250.0860.



6-methoxyspiro[isochromene-1,1'-isoindolin]-3'-one (3c)

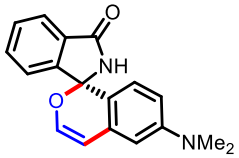
White solid (42.4 mg, 76% yield). PE/EA = 3:1, R_f = 0.37. ^{1}H NMR (400 MHz, DMSO) δ 9.79 (s, 1H), 7.73 (d, *J* = 5.9 Hz, 1H), 7.68 – 7.55 (m, 2H), 7.48 (d, *J* = 7.8 Hz, 1H), 6.85 (d, *J* =

5.8 Hz, 1H), 6.82 (d, *J* = 2.6 Hz, 1H), 6.71 (d, *J* = 8.6 Hz, 1H), 6.64 (d, *J* = 8.6 Hz, 1H), 6.09 (d, *J* = 5.8 Hz, 1H), 3.75 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 167.9, 159.8, 147.6, 144.3, 132.9, 131.2, 130.1, 125.4, 123.1, 122.8, 120.3, 113.0, 108.7, 104.1, 90.1, 55.1. HRMS (ESI) m/z Calcd for $C_{17}H_{14}NO_3$ $[M+H]^+$ 280.0968, found 280.0964.



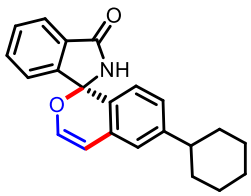
6-(methylthio)spiro[isochromene-1,1'-isoindolin]-3'-one (3d)

White solid (42.5 mg, 72% yield). PE/EA = 3:1, R_f = 0.38. ^{1}H NMR (400 MHz, DMSO) δ 9.96 (s, 1H), 7.77 (d, *J* = 6.8 Hz, 1H), 7.72 – 7.61 (m, 2H), 7.52 (d, *J* = 6.8 Hz, 1H), 7.18 (s, 1H), 7.05 (d, *J* = 8.2 Hz, 1H), 6.92 (d, *J* = 5.7 Hz, 1H), 6.67 (d, *J* = 8.1 Hz, 1H), 6.15 (d, *J* = 5.7 Hz, 1H), 3.49 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 168.1, 147.4, 144.7, 139.7, 133.2, 130.6, 130.5, 130.3, 124.7, 124.5, 124.5, 123.4, 123.1, 120.7, 104.0, 90.0, 14.5. HRMS (ESI) m/z Calcd for $C_{17}H_{14}NO_2S$ $[M+H]^+$ 296.0740, found 296.0735.



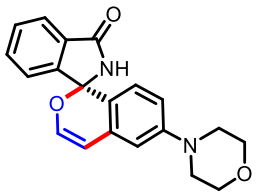
6-(dimethylamino)spiro[isochromene-1,1'-isoindolin]-3'-one (3e)

White solid (29.2 mg, 50% yield). PE/EA = 3:1, R_f = 0.38. ^{1}H NMR (400 MHz, DMSO) δ 9.66 (s, 1H), 7.72 – 7.67 (m, 1H), 7.65 – 7.52 (m, 2H), 7.44 (d, *J* = 7.1 Hz, 1H), 6.77 (d, *J* = 5.8 Hz, 1H), 6.57 – 6.43 (m, 3H), 6.01 (d, *J* = 5.8 Hz, 1H), 2.88 (s, 6H). ^{13}C NMR (101 MHz, DMSO) δ 150.9, 148.0, 143.8, 132.8, 130.4, 130.2, 130.0, 124.8, 123.1, 122.7, 115.7, 111.1, 107.2, 104.7, 90.3, 40.0, 39.9. HRMS (ESI) m/z Calcd for $C_{18}H_{17}N_2O_2$ $[M+H]^+$ 293.1285, found 293.1280.



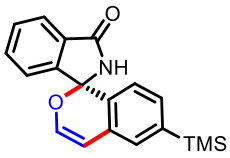
6-cyclohexylspiro[isochromene-1,1'-isoindolin]-3'-one (3f)

White solid (43.7 mg, 66% yield). PE/EA = 3:1, R_f = 0.41. ^{1}H NMR (400 MHz, DMSO) δ 9.92 (s, 1H), 7.79 (d, *J* = 6.9 Hz, 1H), 7.72 – 7.59 (m, 2H), 7.53 (d, *J* = 6.5 Hz, 1H), 7.13 (s, 1H), 7.02 (d, *J* = 8.0 Hz, 1H), 6.88 (d, *J* = 5.7 Hz, 1H), 6.66 (d, *J* = 7.9 Hz, 1H), 6.15 (d, *J* = 5.7 Hz, 1H), 2.61 – 2.38 (m, 1H), 1.89 – 1.65 (m, 5H), 1.50 – 1.18 (m, 5H). ^{13}C NMR (100 MHz, DMSO) δ 167.9, 148.7, 147.4, 143.8, 133.0, 130.4, 130.3, 129.7, 125.8, 125.7, 124.0, 123.4, 122.9, 122.2, 104.5, 90.0, 43.6, 33.8, 33.8, 26.3, 25.5. HRMS (ESI) m/z Calcd for $C_{22}H_{22}NO_2$ $[M+H]^+$ 332.1645, found 332.1639.



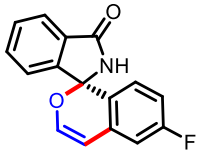
6-morpholinospiro[isochromene-1,1'-isoindolin]-3'-one (3g)

White solid (52.1 mg, 78% yield). PE/EA = 3:1, R_f = 0.35. ^{1}H NMR (400 MHz, DMSO) δ 9.80 (s, 1H), 7.71 (d, *J* = 6.5 Hz, 1H), 7.66 – 7.54 (m, 2H), 7.46 (d, *J* = 6.7 Hz, 1H), 6.82 (d, *J* = 5.7 Hz, 1H), 6.79 (d, *J* = 2.4 Hz, 1H), 6.71 (d, *J* = 8.6 Hz, 1H), 6.56 (d, *J* = 8.6 Hz, 1H), 6.04 (d, *J* = 5.8 Hz, 1H), 3.70 (t, *J* = 4.7 Hz, 4H), 3.08 (t, *J* = 4.7 Hz, 4H). ^{13}C NMR (100 MHz, DMSO) δ 168.0, 151.7, 147.8, 144.0, 133.0, 130.6, 130.3, 130.2, 124.9, 123.3, 122.9, 118.7, 113.9, 110.0, 104.7, 90.2, 66.0, 48.1. HRMS (ESI) m/z Calcd for $C_{20}H_{19}N_2O_3$ $[M+H]^+$ 335.1390, found 335.1385.



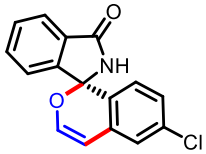
6-(trimethylsilyl)spiro[isochromene-1,1'-isoindolin]-3'-one (3h)

White solid (60.7 mg, 90% yield). PE/EA = 3:1, R_f = 0.39. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 6.3 Hz, 1H), 7.64 (t, *J* = 8.4 Hz, 1H), 7.58 (d, *J* = 8.1 Hz, 1H), 7.37 – 7.30 (m, 2H), 7.22 (s, 1H), 6.83 – 6.75 (m, 2H), 6.12 (d, *J* = 5.7 Hz, 1H), 0.30 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.6, 146.7, 143.8, 142.4, 133.1, 132.5, 130.5, 130.3, 129.1, 128.7, 128.2, 123.8, 123.7, 123.4, 105.0, 89.9, -1.3. HRMS (ESI) m/z Calcd for $C_{19}H_{20}NO_2Si$ $[M+H]^+$ 322.1258, found 322.1251.



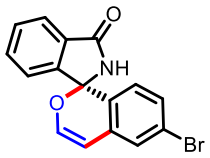
6-fluorospiro[isochromene-1,1'-isoindolin]-3'-one (3i)

White solid (36.3 mg, 68% yield). PE/EA = 3:1, R_f = 0.41. ^{1}H NMR (400 MHz, DMSO) δ 9.95 (s, 1H), 7.74 (d, *J* = 6.4 Hz, 1H), 7.69 – 7.58 (m, 2H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.13 (d, *J* = 9.6 Hz, 1H), 7.00 – 6.89 (m, 2H), 6.79 – 6.70 (m, 1H), 6.15 (d, *J* = 5.8 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 167.9 , 162.5 (d, *J* = 244.6 Hz), 147.1 , 145.3 , 133.3 , 132.4 (d, *J* = 9.5 Hz), 130.6 , 130.3 , 126.6 (d, *J* = 9.1 Hz), 124.1 (d, *J* = 2.9 Hz), 123.4 , 123.1, 114.0 (d, *J* = 22.4 Hz), 110.4 (d, *J* = 22.7 Hz), 103.7 , 89.9. ^{19}F NMR (376 MHz, DMSO) δ -112.8. HRMS (ESI) m/z Calcd for $C_{16}H_{11}FNO_2$ $[M+H]^+$ 268.0768, found 268.0764.



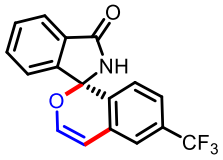
6-chlorospiro[isochromene-1,1'-isoindolin]-3'-one (3j)

White solid (39.6 mg, 70% yield). PE/EA = 3:1, R_f = 0.42. ^{1}H NMR (400 MHz, DMSO) δ 9.97 (s, 1H), 7.79 – 7.72 (m, 1H), 7.70 – 7.60 (m, 2H), 7.51 (d, *J* = 6.9 Hz, 1H), 7.37 (d, *J* = 2.2 Hz, 1H), 7.18 (d, *J* = 8.3 Hz, 1H), 6.94 (d, *J* = 5.7 Hz, 1H), 6.70 (d, *J* = 8.3 Hz, 1H), 6.15 (d, *J* = 5.7 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 167.9, 147.0, 145.4, 133.9, 133.3, 132.0, 130.7, 130.3, 127.0, 126.7, 126.2, 123.5, 123.4, 123.1, 103.4, 89.7. HRMS (ESI) m/z Calcd for $C_{16}H_{11}ClNO_2$ $[M+H]^+$ 284.0473, found 284.0467.



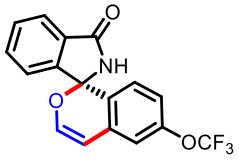
6-bromospiro[isochromene-1,1'-isoindolin]-3'-one (3k)

White solid (47.1 mg, 72% yield). PE/EA = 3:1, R_f = 0.43. ^{1}H NMR (400 MHz, DMSO) δ 9.97 (d, *J* = 2.9 Hz, 1H), 7.74 (d, *J* = 7.6 Hz, 1H), 7.68 – 7.58 (m, 2H), 7.55 – 7.46 (m, 2H), 7.31 (d, *J* = 8.3 Hz, 1H), 6.93 (d, *J* = 5.7 Hz, 1H), 6.65 (d, *J* = 8.3 Hz, 1H), 6.15 (d, *J* = 5.8 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 167.9, 146.9, 145.4, 133.3, 132.3, 130.6, 130.3, 129.9, 127.1, 126.4, 126.3, 123.4, 123.1, 122.5, 103.3, 89.8. HRMS (ESI) m/z Calcd for $C_{16}H_{11}BrNO_2$ $[M+H]^+$ 327.9968, found 327.9960.



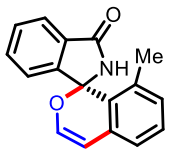
6-(trifluoromethyl)spiro[isochromene-1,1'-isoindolin]-3'-one (3l)

White solid (46.9 mg, 74% yield). PE/EA = 3:1, R_f = 0.40. ^{1}H NMR (400 MHz, DMSO) δ 10.05 (s, 1H), 7.77 (d, *J* = 5.8 Hz, 1H), 7.72 – 7.59 (m, 3H), 7.54 (d, *J* = 6.1 Hz, 1H), 7.48 (d, *J* = 8.1 Hz, 1H), 7.00 (d, *J* = 5.8 Hz, 1H), 6.91 (d, *J* = 8.1 Hz, 1H), 6.30 (d, *J* = 5.8 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 167.9, 146.8, 145.6, 133.4, 131.8, 131.0, 130.8, 130.3, 129.9 (q, *J* = 32.3 Hz), 125.3 , 124.0 (q, *J* = 273.7 Hz), 123.9 (d, *J* = 3.8 Hz), 123.5, 123.2, 120.7(d, *J* = 4.1 Hz), 103.5, 89.6. ^{19}F NMR (376 MHz, DMSO) δ -61.4. ^{19}F NMR (376 MHz, DMSO) δ -56.8. HRMS (ESI) m/z Calcd for $C_{17}H_{11}F_3NO_2$ $[M+H]^+$ 318.0736, found 318.0729.



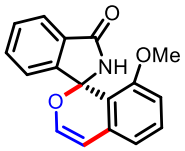
6-(trifluoromethoxy)spiro[isochromene-1,1'-isoindolin]-3'-one (3m)

White solid (54.6 mg, 82% yield). PE/EA = 3:1, R_f = 0.39.^{1}H NMR (400 MHz, DMSO) δ 10.02 (s, 1H), 7.85 – 7.71 (m, 1H), 7.69 – 7.59 (m, 2H), 7.53 (d, *J* = 6.8 Hz, 1H), 7.31 (s, 1H), 7.10 (d, *J* = 8.6 Hz, 1H), 6.97 (d, *J* = 5.7 Hz, 1H), 6.82 (d, *J* = 8.5 Hz, 1H), 6.23 (d, *J* = 5.7 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 168.4, 149.4, 147.4, 146.0, 133.8, 132.8, 131.1, 130.9, 127.4, 127.0, 123.9, 123.6, 120.5 (q, *J* = 256.7 HZ), 120.0, 116.5, 104.0, 90.2. ^{19}F NMR (376 MHz, DMSO) δ -56.8. HRMS (ESI) m/z Calcd for $C_{17}H_{11}F_3NO_3$ $[M+H]^+$ 334.0686, found 334.0683.



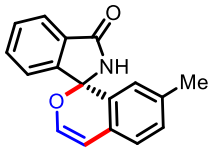
8-methylspiro[isochromene-1,1'-isoindolin]-3'-one (3n)

White solid (46.8 mg, 89% yield). PE/EA = 3:1, R_f = 0.40. ^{1}H NMR (400 MHz, DMSO) δ 9.90 (s, 1H), 7.76 (d, *J* = 5.1 Hz, 1H), 7.66 – 7.54 (m, 2H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.09 (d, *J* = 7.5 Hz, 1H), 6.95 (d, *J* = 7.5 Hz, 1H), 6.80 (d, *J* = 5.6 Hz, 1H), 6.03 (d, *J* = 5.6 Hz, 1H), 1.55 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 168.4, 148.3, 143.0, 134.4, 133.1, 131.1, 130.9, 130.4, 130.3, 129.1, 125.1, 123.2, 123.2, 123.1, 103.9, 89.9, 20.3. HRMS (ESI) m/z Calcd for $C_{17}H_{14}NO_2$ $[M+H]^+$ 264.1019, found 264.1014.



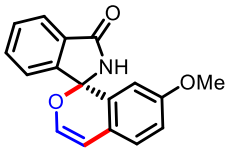
8-methoxyspiro[isochromene-1,1'-isoindolin]-3'-one (3o)

White solid (41.3 mg, 74% yield). PE/EA = 3:1, R_f = 0.38. ^{1}H NMR (400 MHz, DMSO) δ 9.59 (s, 1H), 7.67 (d, *J* = 7.1 Hz, 1H), 7.53 (t, *J* = 9.5 Hz, 2H), 7.38 – 7.25 (m, 2H), 6.87 – 6.74 (m, 3H), 5.99 (d, *J* = 2.9 Hz, 1H), 3.20 (d, *J* = 3.0 Hz, 3H). ^{13}C NMR (100 MHz, DMSO) δ 168.3, 155.4, 149.9, 143.5, 132.1, 131.4, 130.6, 130.0, 128.9, 122.2, 122.0, 116.9, 115.0, 111.1, 102.7, 87.9, 55.5. HRMS (ESI) m/z Calcd for $C_{17}H_{14}NO_3$ $[M+H]^+$ 280.0968, found 280.0962.



7-methylspiro[isochromene-1,1'-isoindolin]-3'-one (3p)

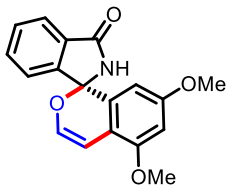
White solid (41.0 mg, 78% yield). PE/EA = 3:1, R_f = 0.39. ^{1}H NMR (400 MHz, DMSO) δ 9.78 (s, 1H), 7.72 (d, *J* = 6.0 Hz, 1H), 7.66 – 7.57 (m, 2H), 7.48 (d, *J* = 6.6 Hz, 1H), 7.13 (d, *J* = 2.6 Hz, 2H), 6.79 (d, *J* = 5.7 Hz, 1H), 6.54 (s, 1H), 6.08 (d, *J* = 5.7 Hz, 1H), 2.15 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 167.9, 147.4, 143.0, 136.6, 133.0, 130.2, 130.1, 129.8, 128.0, 127.1, 124.1, 124.0, 123.2, 122.9, 104.1, 90.0, 20.7. HRMS (ESI) m/z Calcd for $C_{17}H_{14}NO_2$ $[M+H]^+$ 264.1019, found 264.1015.



7-methoxyspiro[isochromene-1,1'-isoindolin]-3'-one (3q)

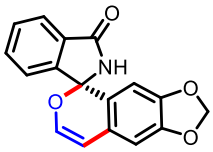
White solid (41.9 mg, 75% yield). PE/EA = 3:1, R_f = 0.37. ^{1}H NMR (400 MHz, DMSO) δ 9.89 (s, 1H), 7.73 (d, *J* = 6.1 Hz, 1H), 7.69 – 7.58 (m, 2H), 7.49 (d, *J* = 6.4 Hz, 1H), 7.10 (t, *J* = 8.0 Hz, 1H), 6.99 (d, *J* = 8.2 Hz, 1H), 6.85 (d, *J* = 5.8 Hz, 1H), 6.31 – 6.22 (m, 2H), 3.84 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 167.9, 152.6, 147.2, 143.4, 133.1, 130.4, 128.8, 128.1, 123.5,

123.0, 118.8, 116.0, 111.1, 98.7, 89.7, 55.7. HRMS (ESI) m/z Calcd for $C_{17}H_{14}NO_3$ $[M+H]^+$ 280.0968, found 280.0961.



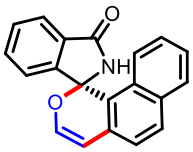
5,7-dimethoxyspiro[isochromene-1,1'-isoindolin]-3'-one (3r)

White solid (42.0 mg, 68% yield). PE/EA = 3:1, R_f = 0.36. ^{1}H NMR (400 MHz, DMSO) δ 9.84 (d, *J* = 6.3 Hz, 1H), 7.73 (d, *J* = 6.0 Hz, 1H), 7.67 – 7.56 (m, 2H), 7.50 (d, *J* = 5.7 Hz, 1H), 6.72 (d, *J* = 5.8 Hz, 1H), 6.60 (s, 1H), 6.20 (d, *J* = 5.5 Hz, 1H), 5.86 (d, *J* = 2.5 Hz, 1H), 3.84 (s, 3H), 3.60 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 168.2, 159.6, 154.3, 147.1, 141.2, 133.1, 130.4, 130.3, 129.9, 123.3, 123.0, 112.1, 100.6, 99.0, 98.6, 89.9, 55.8, 55.3. HRMS (ESI) m/z Calcd for $C_{18}H_{16}NO_4$ $[M+H]^+$ 310.1074, found 310.1070.



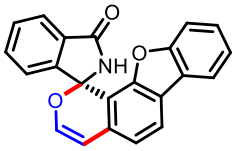
spiro[isoindoline-1,5'-[1,3]dioxolo[4,5-g]isochromen]-3-one (3s)

White solid (34.0 mg, 58% yield). PE/EA = 3:1, R_f = 0.36. ^{1}H NMR (400 MHz, DMSO) δ 9.84 (s, 1H), 7.71 (d, *J* = 5.9 Hz, 1H), 7.67 – 7.58 (m, 2H), 7.51 (d, *J* = 6.6 Hz, 1H), 6.91 (d, *J* = 5.8 Hz, 1H), 6.70 (d, *J* = 8.1 Hz, 1H), 6.27 (d, *J* = 8.1 Hz, 1H), 6.11 (s, 2H), 6.06 (d, *J* = 5.8 Hz, 1H).^{13}C NMR (100 MHz, DMSO) δ 168.0, 147.6, 147.2, 144.9, 140.8, 133.2, 130.5, 130.2, 123.3, 123.0, 122.2, 117.6, 112.8, 107.0, 101.8, 97.8, 90.0. HRMS (ESI) m/z Calcd for $C_{17}H_{12}NO_4^+$ [M+H] 294.0761, found 294.0759.



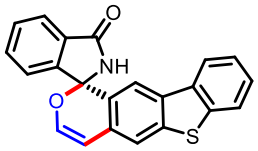
spiro[benzo[h]isochromene-1,1'-isoindolin]-3'-one (3t)

White solid (44.3 mg, 74% yield). PE/EA = 3:1, R_f = 0.37. ^{1}H NMR (400 MHz, DMSO) δ 10.16 (s, 1H), 8.00 – 7.80 (m, 3H), 7.73 – 7.57 (m, 2H), 7.49 – 7.34 (m, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.10 (t, *J* = 7.9 Hz, 1H), 6.98 (d, *J* = 5.5 Hz, 1H), 6.87 (d, *J* = 8.8 Hz, 1H), 6.22 (d, *J* = 5.5 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 168.2, 149.4, 144.0, 133.5, 133.3, 130.5, 130.5, 130.2, 130.1, 129.3, 128.6, 126.6, 124.7, 124.0, 123.6, 123.5, 122.9, 119.4, 104.2, 90.3. HRMS (ESI) m/z Calcd for $C_{20}H_{14}NO_2$ $[M+H]^+$ 300.1019, found 300.1014.



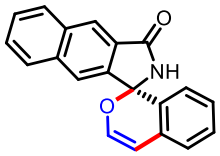
spiro[benzofuro[3,2-h]isochromene-1,1'-isoindolin]-3'-one (3u)

White solid (48.8 mg, 72% yield). PE/EA = 3:1, R_f = 0.37. ^{1}H NMR (400 MHz, DMSO) δ 10.04 (s, 1H), 8.11 (d, *J* = 7.9 Hz, 1H), 8.02 (d, *J* = 6.6 Hz, 1H), 7.84 (d, *J* = 7.3 Hz, 1H), 7.68 – 7.55 (m, 2H), 7.48 (d, *J* = 7.4 Hz, 1H), 7.40 – 7.25 (m, 3H), 7.20 – 7.13 (m, 1H), 6.99 (d, *J* = 5.7 Hz, 1H), 6.27 (d, *J* = 5.8 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 168.2, 155.1, 150.6, 147.9, 144.1, 132.9, 130.4, 130.3, 129.9, 127.4, 123.4, 123.4, 123.2, 123.1, 122.9, 122.1, 120.7, 119.9, 111.3, 111.3, 103.5, 87.6. HRMS (ESI) m/z Calcd for $C_{22}H_{14}NO_3$ $[M+H]^+$ 340.0968, found 340.0961.



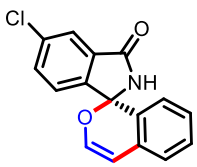
spiro[benzo[4,5]thieno[2,3-g]isochromene-1,1'-isoindolin]-3'-one (3v)

White solid (43.3 mg, 61% yield). PE/EA = 3:1, R_f = 0.35. ^{1}H NMR (400 MHz, DMSO) δ 9.98 (s, 1H), 8.14 (d, *J* = 7.8 Hz, 1H), 7.96 (t, *J* = 6.6 Hz, 1H), 7.90 (d, *J* = 4.6 Hz, 1H), 7.78 (d, *J* = 5.2 Hz, 2H), 7.67 – 7.58 (m, 2H), 7.58 – 7.51 (m, 1H), 7.47 – 7.39 (m, 1H), 7.39 – 7.31 (m, 1H), 6.94 (t, *J* = 5.7 Hz, 1H), 6.26 (t, *J* = 5.6 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 168.4, 147.6, 144.7, 139.7, 138.4, 134.7, 134.0, 133.2, 130.5, 129.9, 129.2, 127.0, 125.9, 124.8, 123.2, 123.1, 123.0, 122.0, 118.0, 117.6, 104.5, 90.7. HRMS (ESI) m/z Calcd for $C_{22}H_{14}NO_2S$ $[M+H]^+$ 356.0740, found 356.0736.



spiro[benzo[f]isoindole-1,1'-isochromen]-3(2H)-one (3a')

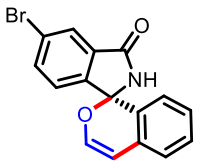
White solid (46.7 mg, 78% yield). PE/EA = 3:1, R_f = 0.38.^{1}H NMR (400 MHz, DMSO-d_6) δ 10.07 (s, 1H), 8.39 (s, 1H), 8.18 (dd, *J* = 6.2, 3.4 Hz, 1H), 8.03 (d, *J* = 9.3 Hz, 2H), 7.66 – 7.58 (m, 2H), 7.33 (t, *J* = 7.5, 1.2 Hz, 1H), 7.26 (dd, *J* = 7.7, 1.3 Hz, 1H), 7.12 (t, *J* = 7.6, 1.4 Hz, 1H), 6.89 (d, *J* = 5.7 Hz, 1H), 6.78 (d, *J* = 7.7 Hz, 1H), 6.18 (d, *J* = 5.7 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 167.5, 143.9, 142.8, 135.1, 133.5, 129.8, 129.5, 129.2, 128.7, 128.6, 128.3, 128.1, 127.3, 127.2, 124.4, 124.0, 123.6, 122.7, 104.3, 90.1. HRMS (ESI) m/z Calcd for $C_{20}H_{14}NO_2$ $[M+H]^+$ 356.0740, found 356.0736.



5'-chlorospiro[isochromene-1,1'-isoindolin]-3'-one (3b')

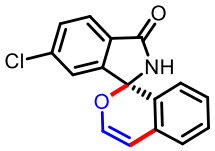
White solid (48.7 mg, 86% yield). PE/EA = 3:1, R_f = 0.38. ^{1}H NMR (400 MHz, DMSO) δ 10.11 (s, 1H), 7.76 (d, *J* = 1.9 Hz, 1H), 7.67 (d, *J* = 8.1 Hz, 1H), 7.51 (d, *J* = 8.1 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 8.2 Hz, 1H), 7.14 (t, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 5.7 Hz, 1H), 6.75 (d, *J* = 7.7 Hz, 1H), 6.15 (d, *J* = 5.7 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 166.5, 145.8, 143.9, 135.3, 133.1, 132.5, 129.7, 129.5, 127.5, 125.3, 124.2, 124.1, 123.0, 104.5, 89.7. HRMS (ESI) m/z

Calcd for $C_{16}H_{11}ClNO_2$ $[M+H]^+$ 284.0473, found 284.0470.



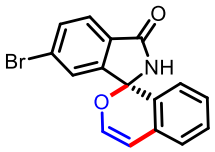
5'-bromospiro[isochromene-1,1'-isoindolin]-3'-one (3c')

White solid (53.6 mg, 82% yield). PE/EA = 3:1, R_f = 0.37.^{1}H NMR (400 MHz, DMSO) δ 10.09 (s, 1H), 7.88 (d, *J* = 1.9 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.46 (d, *J* = 8.1 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 7.14 (t, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 5.7 Hz, 1H), 6.74 (d, *J* = 7.8 Hz, 1H), 6.15 (d, *J* = 5.7 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 166.4, 146.2, 143.9, 135.9, 132.7, 129.7, 129.5, 127.5, 127.4, 125.9, 125.6, 124.2, 124.1, 123.7, 104.5, 89.8. HRMS (ESI) m/z Calcd for $C_{16}H_{11}BrNO_2$ $[M+H]^+$ 327.9968, found 327.9964.



6'-chlorospiro[isochromene-1,1'-isoindolin]-3'-one (3d')

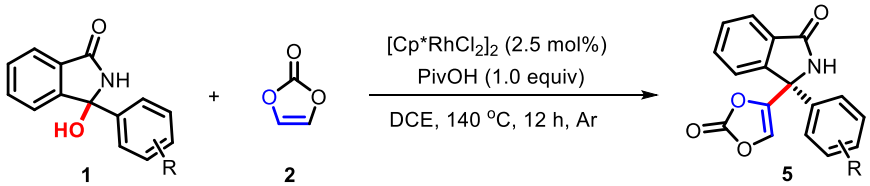
White solid (44.7 mg, 79% yield). PE/EA = 3:1, R_f = 0.38.^{1}H NMR (400 MHz, DMSO) δ 10.04 (s, 1H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 9.8 Hz, 1H), 7.55 (d, *J* = 1.7 Hz, 1H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 6.4 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 6.87 (d, *J* = 5.7 Hz, 1H), 6.76 (d, *J* = 7.7 Hz, 1H), 6.15 (d, *J* = 5.7 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 166.8, 149.0, 143.8, 137.8, 130.9, 129.7, 129.5, 129.2, 127.6, 127.4, 125.0, 124.2, 124.1, 123.4, 104.5, 89.5. HRMS (ESI) m/z Calcd for $C_{16}H_{11}ClNO_2$ $[M+H]^+$ 284.0473, found 284.0469.



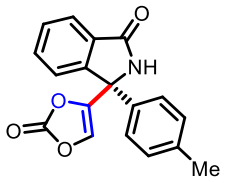
6'-bromospiro[isochromene-1,1'-isoindolin]-3'-one (3e')

White solid (51.0 mg, 78% yield). PE/EA = 3:1, R_f = 0.37.^{1}H NMR (400 MHz, DMSO) δ 10.04 (s, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 4.8 Hz, 2H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 7.6 Hz, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 6.87 (d, *J* = 5.7 Hz, 1H), 6.76 (d, *J* = 7.7 Hz, 1H), 6.15 (d, *J* = 5.7 Hz, 1H). ^{13}C NMR (100 MHz, DMSO) δ 167.0, 149.2, 143.9, 133.8, 129.7, 129.6, 129.5, 127.6, 127.4, 126.6, 126.3, 125.2, 124.3, 124.1, 104.5, 89.6. HRMS (ESI) m/z Calcd for $C_{16}H_{11}BrNO_2$ $[M+H]^+$ 327.9968, found 327.9962.

D. Reaction results of non-cyclization reaction

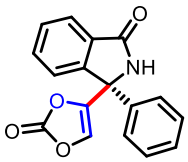


A pressure tube equipped with a stir bar was charged with $[Cp^*RhCl_2]_2$ (3.1 mg, 2.5 mol%), 3-hydroxy-3-(*p*-tolyl)isoindolin-1-one **1** (47.8 mg, 0.2 mmol), PivOH (20.4 mg, 1.0 equiv), vinylene carbonate **2** (51.6 mg, 0.6 mmol) in DCE (1.0 mL), The reaction mixture was stirred at 140 °C for 12 h under argon atmosphere in an oil bath. After cooling to room temperature, the reaction mixture was diluted with 15.0 mL aqueous saturated $NaHCO_3$ and extracted with DCM (3 × 5 mL). The combined organic phase was dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford the products **5**.



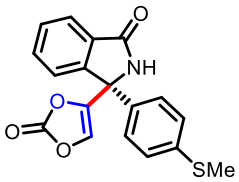
3-(2-oxo-1,3-dioxol-4-yl)-3-(p-tolyl)isoindolin-1-one (5a)

White solid (39.9 mg, 65% yield). PE/EA = 2:1, R_f = 0.31. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 7.5 Hz, 1H), 7.82 (s, 1H), 7.67 – 7.55 (m, 2H), 7.50 (d, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 8.2 Hz, 2H), 7.18 (d, *J* = 8.2 Hz, 2H), 7.00 (d, *J* = 1.5 Hz, 1H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 152.3, 145.5, 144.1, 139.4, 133.4, 133.1, 130.3, 129.9, 129.9, 127.8, 125.9, 124.8, 123.6, 63.6, 21.1. HRMS (ESI) m/z Calcd for $C_{18}H_{14}NO_4$ $[M+H]^+$ 308.0917, found 308.0911.



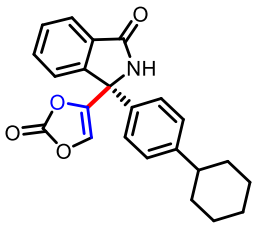
3-(2-oxo-1,3-dioxol-4-yl)-3-phenylisoindolin-1-one (5b)

White solid (41.0 mg, 70% yield). PE/EA = 2:1, R_f = 0.3. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 (s, 1H), 7.91 (d, *J* = 7.5 Hz, 1H), 7.67 – 7.56 (m, 2H), 7.52 (d, *J* = 7.3 Hz, 1H), 7.37 (s, 5H), 7.04 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 152.3, 145.3, 143.9, 136.5, 133.1, 130.4, 129.9, 129.3, 127.9, 126.0, 124.8, 123.7, 63.8. HRMS (ESI) m/z Calcd for $C_{17}H_{12}NO_4$ $[M+H]^+$ 294.0761, found 294.0757.



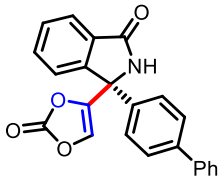
3-(4-(methylthio)phenyl)-3-(2-oxo-1,3-dioxol-4-yl)isoindolin-1-one (5c)

White solid (50.8 mg, 75% yield). PE/EA = 2:1, R_f = 0.29. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.90 (d, *J* = 7.5 Hz, 1H), 7.69 – 7.62 (m, 1H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.51 (d, *J* = 7.5 Hz, 1H), 7.30 – 7.26 (m, 2H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.04 (s, 1H), 2.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 152.3, 145.3, 143.8, 140.5, 133.1, 132.8, 130.4, 129.9, 127.9, 126.6, 126.5, 124.8, 123.6, 63.5, 15.3. HRMS (ESI) m/z Calcd for $C_{18}H_{14}NO_4S$ $[M+H]^+$ 340.0638, found 340.0633.



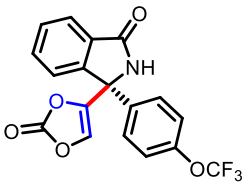
3-(4-cyclohexylphenyl)-3-(2-oxo-1,3-dioxol-4-yl)isoindolin-1-one (5d)

White solid (48.8 mg, 65% yield). PE/EA = 2:1, R_f = 0.31. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.5 Hz, 1H), 7.72 – 7.57 (m, 2H), 7.52 (d, *J* = 7.5 Hz, 1H), 7.29 (s, 2H), 7.23 (d, *J* = 8.7 Hz, 3H), 7.00 (s, 1H), 2.52 (s, 1H), 1.85 (d, *J* = 8.5 Hz, 5H), 1.50 – 1.33 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 152.3, 149.4, 145.5, 144.1, 133.6, 133.1, 130.3, 129.9, 127.7, 125.9, 124.8, 123.6, 63.6, 44.1, 34.2, 26.7, 26.0. HRMS (ESI) m/z Calcd for $C_{23}H_{22}NO_4$ $[M+H]^+$ 376.1543, found 376.1540.



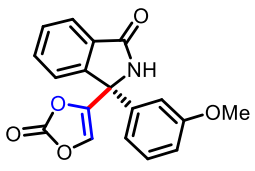
3-([1,1'-biphenyl]-4-yl)-3-(2-oxo-1,3-dioxol-4-yl)isoindolin-1-one (5e)

White solid (45.8 mg, 62% yield). PE/EA = 2:1, R_f = 0.32. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (s, 1H), 7.94 (d, *J* = 7.5 Hz, 1H), 7.66 (t, *J* = 7.4 Hz, 1H), 7.63 – 7.51 (m, 6H), 7.47 – 7.40 (m, 4H), 7.36 (t, *J* = 7.2 Hz, 1H), 7.07 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 152.3, 145.3, 143.9, 142.2, 139.8, 135.3, 133.2, 130.4, 130.0, 128.9, 127.9, 127.9, 127.8, 127.1, 126.5, 124.9, 123.7, 63.7. HRMS (ESI) m/z Calcd for $C_{23}H_{16}NO_4$ $[M+H]^+$ 370.1074, found 370.1069.



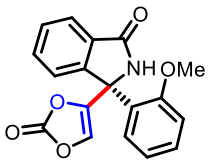
3-(2-oxo-1,3-dioxol-4-yl)-3-(4-(trifluoromethoxy)phenyl)isoindolin-1-one (5f)

Yellow solid (51.3 mg, 68% yield). PE/EA = 2:1, R_f = 0.31. ^{1}H NMR (400 MHz, DMSO) δ 9.90 (s, 1H), 7.78 (d, *J* = 8.2 Hz, 1H), 7.67 (d, *J* = 4.8 Hz, 2H), 7.65 – 7.58 (m, 4H), 7.40 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 168.5, 152.4, 148.4, 145.2, 142.8, 137.1, 133.1, 130.7, 129.9, 129.7, 128.4, 123.9, 123.8, 121.4, 120.0(q, *J* = 256.7 Hz), 62.7. HRMS (ESI) m/z Calcd for $C_{18}H_{11}F_3NO_5$ $[M+H]^+$ 378.0584, found 378.0577.



3-(3-methoxyphenyl)-3-(2-oxo-1,3-dioxol-4-yl)isoindolin-1-one (5g)

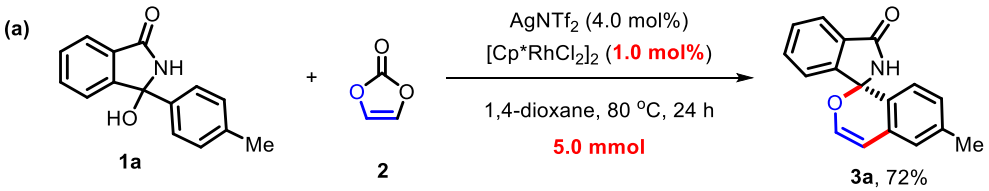
White solid (43.9 mg, 68% yield). PE/EA = 2:1, R_f = 0.28. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (s, 0H), 7.90 (d, *J* = 7.5 Hz, 1H), 7.63 (t, *J* = 7.5 Hz, 1H), 7.60 – 7.50 (m, 2H), 7.30 (t, *J* = 8.0 Hz, 1H), 7.02 (s, 1H), 6.96 (d, *J* = 7.9 Hz, 1H), 6.89 (d, *J* = 12.2 Hz, 2H), 3.76 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 160.1, 152.3, 145.1, 143.9, 137.9, 133.1, 130.4, 129.9, 127.9, 124.8, 123.6, 118.2, 113.9, 112.4, 63.7, 55.3. HRMS (ESI) m/z Calcd for $C_{18}H_{14}NO_5$ $[M+H]^+$ 324.0866, found 324.0860.



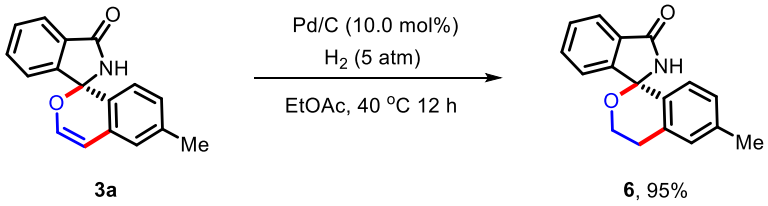
3-(2-methoxyphenyl)-3-(2-oxo-1,3-dioxol-4-yl)isoindolin-1-one (5h)

White solid (39.4 mg, 61% yield). PE/EA = 3:1, R_f = 0.28. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.6 Hz, 1H), 7.71 (s, 2H), 7.66 – 7.58 (m, 1H), 7.49 (s, 1H), 7.43 – 7.35 (m, 1H), 7.21 (d, *J* = 6.1 Hz, 1H), 7.00 (d, *J* = 8.3 Hz, 1H), 6.90 (t, *J* = 7.7 Hz, 1H), 6.85 (s, 1H), 3.90 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 169.1, 157.6, 152.6, 144.5, 144.2, 132.4, 131.1, 129.8, 128.0, 126.3, 125.0, 124.9, 123.0, 120.9, 111.8, 62.0, 55.6. HRMS (ESI) m/z Calcd for $C_{18}H_{14}NO_5$ $[M+H]^+$ 324.0866, found 324.0863.

E. Large-scale transformation and further derivatization

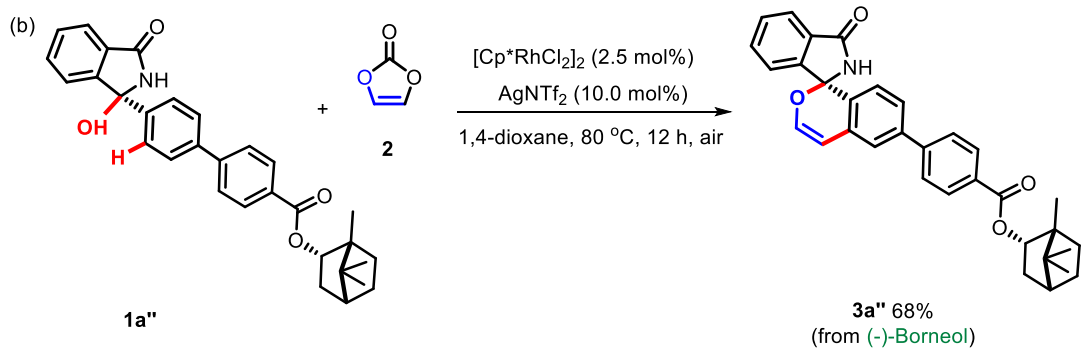


A pressure tube equipped with a stir bar was charged with $[Cp^*RhCl_2]_2$ (30.9 mg, 1.0 mol%), 3-hydroxy-3-(*p*-tolyl)isoindolin-1-one **1a** (1.2 g, 5.0 mmol), $AgNTf_2$ (194.0 mg, 4.0 mol%), vinylene carbonate **2** (1.3 g, 0.3 mmol) in 1,4-dioxane (10.0 mL), The reaction mixture was stirred at 80 °C for 24 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford the product **3a** (947.1 mg, 72% yield).

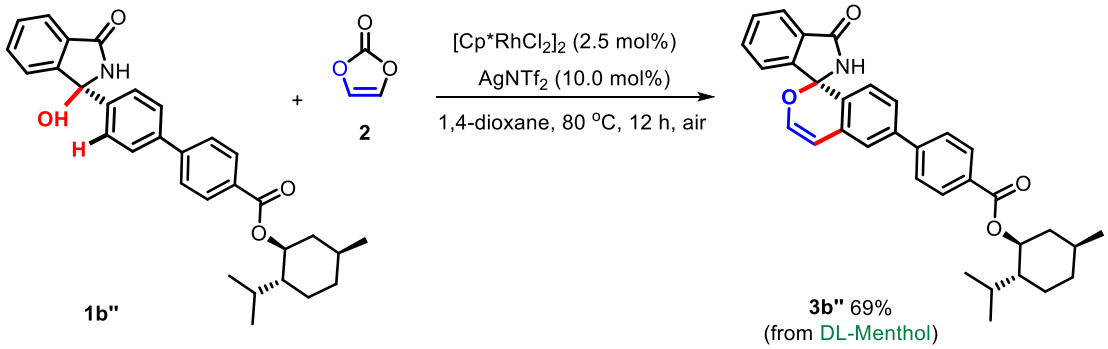


An autoclave equipped with a stir bar was charged with Pd/C (21.2 mg, 10.0 mol%), 6-methylspiro[isochromene-1,1'-isoindolin]-3'-one **3a** (52.6 mg, 0.2 mmol) in EtOAc (2.0 mL), The reaction mixture was stirred under H_2 atmosphere (5 atm) at rt for 12 h. After the reaction was complete (monitored by TLC), the crude reaction mixture was filtered with celite and washed with EtOAc. The solvent was removed under reduced pressure. Then the residue was purified by silica gel column chromatography (PE/EA = 5:1) to afford the desired product **6** (50.3 mg, 95% yield). White solid (20 mg, 75% yield). PE/EA = 5:1, R_f = 0.25.^{1}H NMR (400 MHz, DMSO-d_6) δ 9.59 (s, 1H), 7.78 – 7.63 (m, 1H), 7.52 (q, *J* = 5.0, 4.1 Hz, 2H), 7.16 (dt, *J* = 4.6, 3.1 Hz, 1H), 7.04 (s, 1H),

6.88 (d, *J* = 7.9 Hz, 1H), 6.44 (d, *J* = 8.0 Hz, 1H), 4.19 – 3.98 (m, 2H), 3.07 (ddd, *J* = 16.8, 11.0, 6.0 Hz, 1H), 2.71 (dt, *J* = 16.5, 2.6 Hz, 1H), 2.23 (s, 3H). ^{13}C NMR (100 MHz, DMSO) δ 168.4, 149.5, 137.0, 134.8, 132.6, 131.6, 131.2, 129.5, 129.0, 127.3, 126.4, 123.1, 122.5, 88.4, 61.4, 27.9, 20.5. HRMS (ESI) m/z Calcd for $C_{17}H_{16}NO_2$ $[M+H]^+$ 266.1102, found 266.1105.

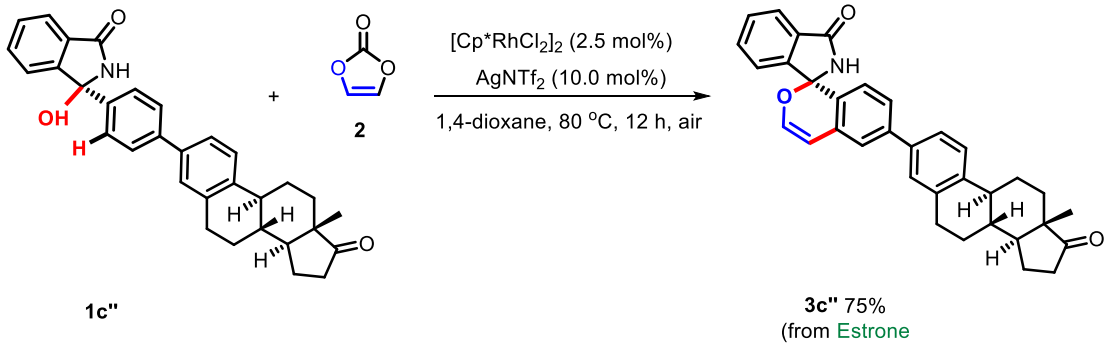


A pressure tube equipped with a stir bar was charged with $[Cp^*RhCl_2]_2$ (3.1 mg, 2.5 mol%),1,7,7-trimethylbicyclo[2.2.1]heptan-2-yl-4'-((S)-1-hydroxy-3-oxoisoindolin-1-yl)-[1,1'-biphenyl]-4-carboxylate **1a''** (96.2 mg, 0.2 mmol), $AgNTf_2$ (7.8 mg, 10.0 mol%), vinylene carbonate **2** (51.6 mg, 0.6 mmol) in 1,4-dioxane (1.0 mL), The reaction mixture was stirred at 80 °C for 12 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford the products **3a''**. White solid (67.7 mg, 68% yield). PE/EA = 2:1, R_f = 0.36. ^{1}H NMR (600 MHz, DMSO-*d*$_6$) δ 9.96 (s, 1H), 8.05 (d, *J* = 8.1 Hz, 2H), 7.82 (d, 2H), 7.75 (d, 1H), 7.68 – 7.60 (m, 3H), 7.55 (d, *J* = 7.2 Hz, 1H), 7.49 (d, *J* = 8.1, 2.0 Hz, 1H), 6.94 (d, *J* = 5.8 Hz, 1H), 6.81 (d, *J* = 7.6 Hz, 1H), 6.23 (d, *J* = 8.2, 5.7 Hz, 1H), 5.04 (d, *J* = 10.1, 2.7 Hz, 1H), 2.39 (t, *J* = 13.7, 4.0 Hz, 1H), 2.07 (t, *J* = 13.4, 9.5, 4.3 Hz, 1H), 1.81 – 1.67 (m, 2H), 1.44 – 1.34 (m, 1H), 1.33 – 1.25 (m, 1H), 1.20 (s, 1H), 0.92 (s, 3H), 0.87 (d, *J* = 12.2, 6.8 Hz, 6H). ^{13}C NMR (150 MHz, DMSO-*d*) δ 168.0, 164.9, 147.3, 144.6, 143.9, 139.8, 133.2, 130.6, 130.5, 130.3, 129.8, 129.1, 128.0, 127.0, 125.9, 124.9, 123.4, 123.0, 122.5, 104.1, 89.9, 74.2, 46.6, 40.5, 33.7, 30.9, 26.3, 23.3, 21.9, 20.4, 16.5. HRMS (ESI) m/z Calcd for $C_{33}H_{32}NO_4$ $[M+H]^+$ 506.2326, found 506.2329.



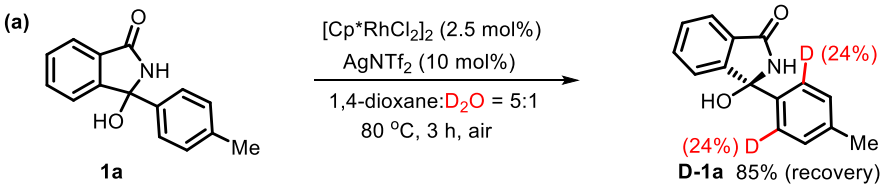
A pressure tube equipped with a stir bar was charged with $[Cp^*RhCl_2]_2$ (3.1 mg, 2.5 mol%), 2-isopropyl-5-methylcyclohexyl-4'-((S)-1-hydroxy-3-oxoisoindolin-1-yl)-[1,1'-biphenyl]-4-carboxylate **1b''** (96.6 mg, 0.2 mmol), $AgNTf_2$ (7.8 mg, 10.0 mol%), vinylene carbonate **2** (51.6 mg, 0.6 mmol) in 1,4-dioxane (1.0 mL), The reaction mixture was stirred at 80 °C for 12 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford the products **3b''**. White solid (69.9 mg, 69% yield). PE/EA = 2:1, R_f = 0.38. ^{1}H NMR (600 MHz, DMSO-*d*$_6$) δ

9.97 (s, 1H), 8.02 (d, *J* = 8.0 Hz, 2H), 7.80 (d, *J* = 8.0 Hz, 2H), 7.76 (d, *J* = 7.0 Hz, 1H), 7.66 – 7.62 (m, 3H), 7.53 (d, *J* = 7.3 Hz, 1H), 7.49 (d, *J* = 8.1 Hz, 1H), 6.93 (d, *J* = 5.7 Hz, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 6.23 (d, *J* = 5.8 Hz, 1H), 4.84 (t, *J* = 10.8, 4.2 Hz, 1H), 1.98 (d, *J* = 12.1 Hz, 1H), 1.92 – 1.82 (m, 1H), 1.65 (d, *J* = 12.5 Hz, 2H), 1.56 – 1.42 (m, 2H), 1.20 (s, 1H), 1.12 – 1.04 (m, 2H), 0.89 – 0.85 (m, 6H), 0.73 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (151 MHz, DMSO-*d*) δ 168.0, 165.6, 147.3, 144.6, 144.0, 139.9, 133.2, 130.6, 130.5, 130.3, 129.7, 129.6, 129.2, 128.0, 127.1, 126.0, 124.9, 123.4, 123.0, 122.5, 104.1, 89.9, 79.8, 48.7, 47.5, 44.3, 36.4, 27.6, 19.5, 18.7, 13.5. HRMS (ESI) m/z Calcd for $C_{33}H_{34}NO_4$ $[M+H]^+$ 508.2482, found 508.2477.

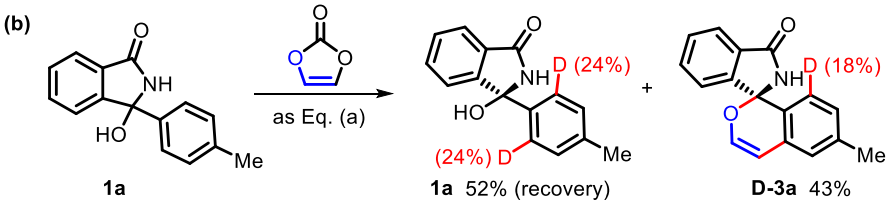


A pressure tube equipped with a stir bar was charged with $[Cp^*RhCl_2]_2$ (3.1 mg, 2.5 mol%), 3-hydroxy-3-(4-((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)phenyl)isoindolin-1-one **1c''** (95.4 mg, 0.2 mmol), $AgNTf_2$ (7.8 mg, 10.0 mol%), vinylene carbonate **2** (51.6 mg, 0.6 mmol) in 1,4-dioxane (1.0 mL), The reaction mixture was stirred at 80 °C for 12 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford the products **3c''**. White solid (75.1 mg, 75% yield). PE/EA = 2:1, R_f = 0.22. ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.91 (s, 1H), 7.74 (d, 1H), 7.66 – 7.59 (m, 2H), 7.53 (d, 2H), 7.45 – 7.32 (m, 4H), 6.90 (d, *J* = 5.7 Hz, 1H), 6.75 (d, *J* = 8.1 Hz, 1H), 6.21 (d, *J* = 5.8 Hz, 1H), 2.92 (d, 2H), 2.44 – 2.39 (m, 1H), 2.28 (s, 1H), 2.00 – 1.95 (m, 2H), 1.78 (d, *J* = 9.9 Hz, 1H), 1.55 (t, 3H), 1.40 (d, 3H), 1.33 – 1.26 (m, 2H), 0.83 (s, 3H).^{13}C NMR (101 MHz, DMSO) δ 219.6, 168.0, 147.4, 144.3, 141.0, 139.4, 136.8, 136.6, 133.2, 130.5, 130.3, 126.9, 126.8, 125.9, 125.4, 124.7, 123.9, 123.4, 123.0, 121.9, 104.3, 89.9, 49.6, 47.3, 43.8, 37.6, 35.4, 31.4, 29.0, 26.0, 25.3, 21.1, 13.5. HRMS (ESI) m/z Calcd for $C_{34}H_{32}NO_3$ $[M+H]^+$ 502.2377, found 506.2369.

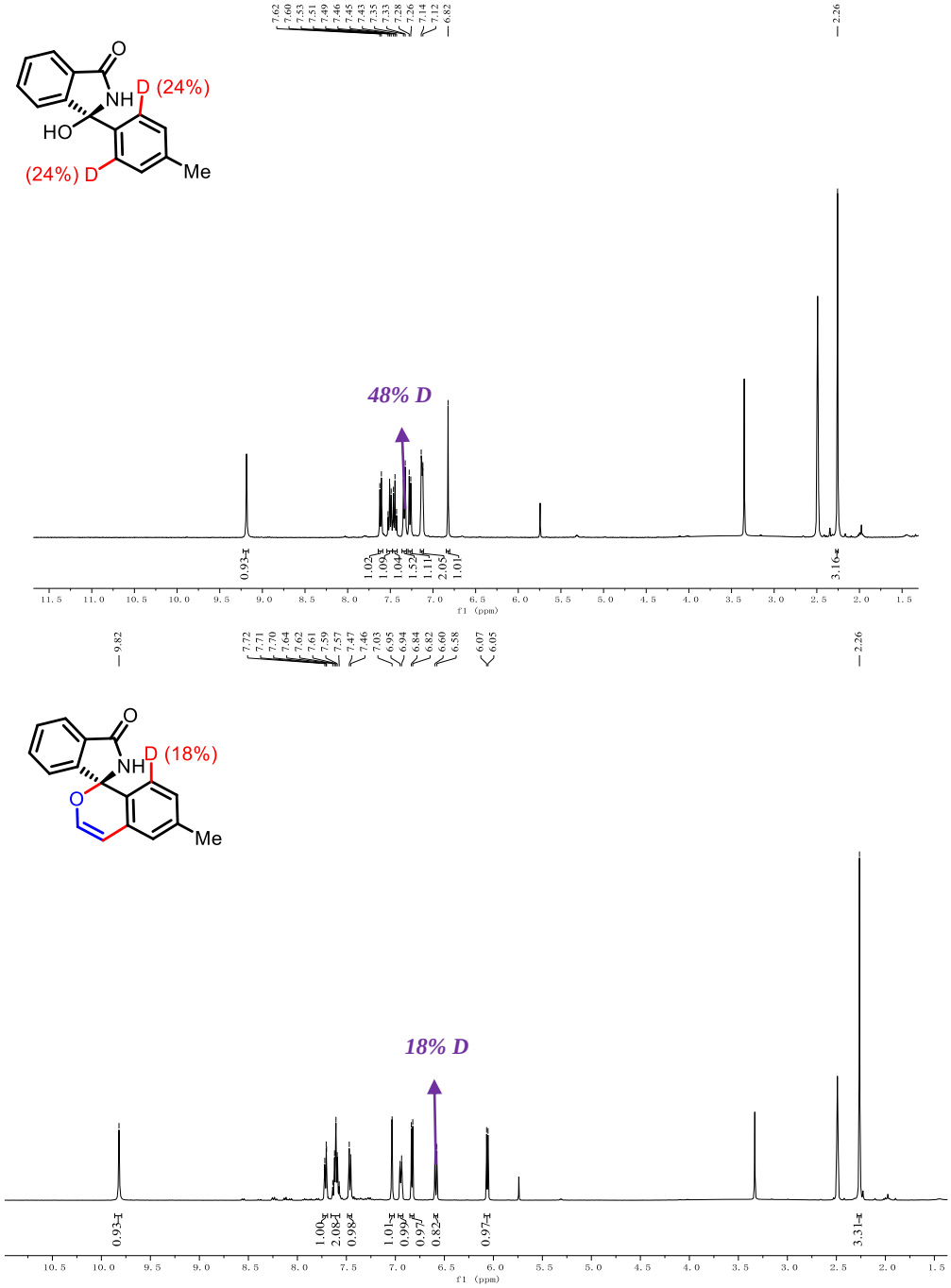
F. Mechanistic study

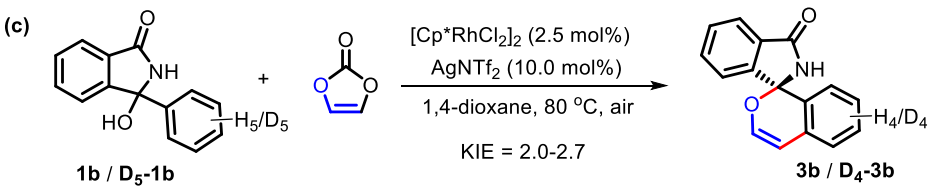


A pressure tube equipped with a stir bar was charged with **1a** (23.9 mg, 0.1 mmol), $[Cp^*RhCl_2]_2$ (1.6 mg, 2.5 mol%), $AgNTf_2$ (3.9 mg, 10.0 mol%) in 1,4-dioxane (0.5 mL) and D_2O (0.1 mL), The reaction mixture was stirred at 80 °C for 3 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford product **D-1a**.



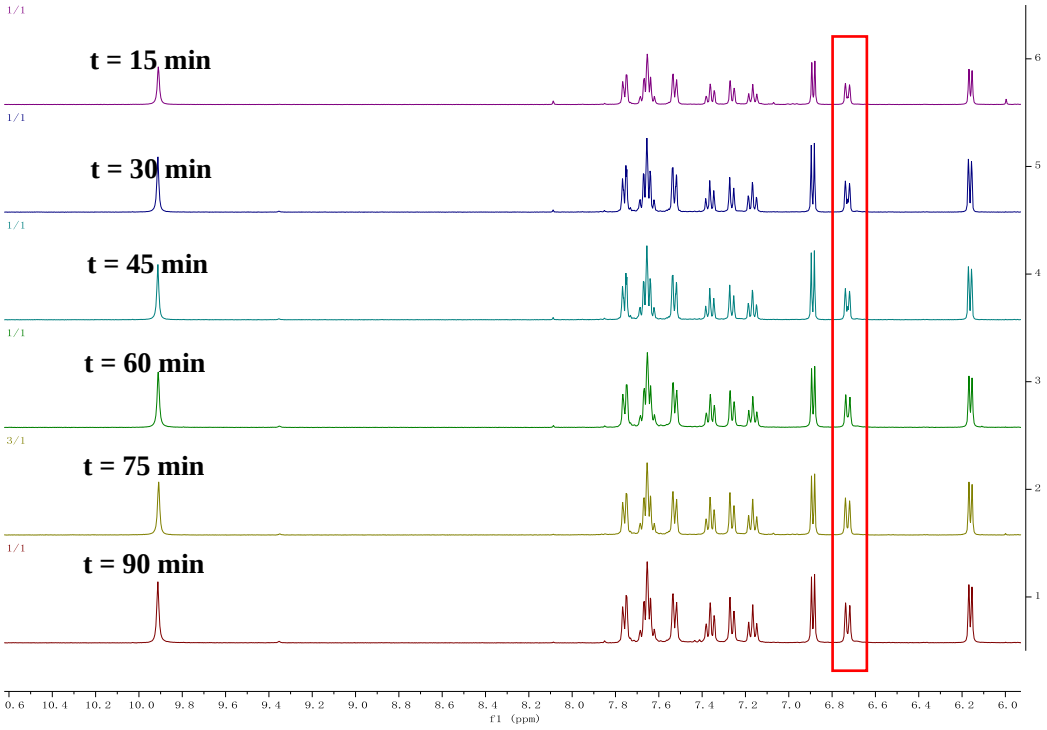
A pressure tube equipped with a stir bar was charged with **1a** (23.9 mg, 0.1 mmol), vinylene carbonate **2** (25.8 mg, 0.3 mmol), $[Cp^*RhCl_2]_2$ (1.6 mg, 2.5 mol%), $AgNTf_2$ (3.9 mg, 10.0 mol%) in 1,4-dioxane (0.5 mL) and D_2O (0.1 mL), The reaction mixture was stirred at 80 °C for 3 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford product **1a** and **D-3a.**



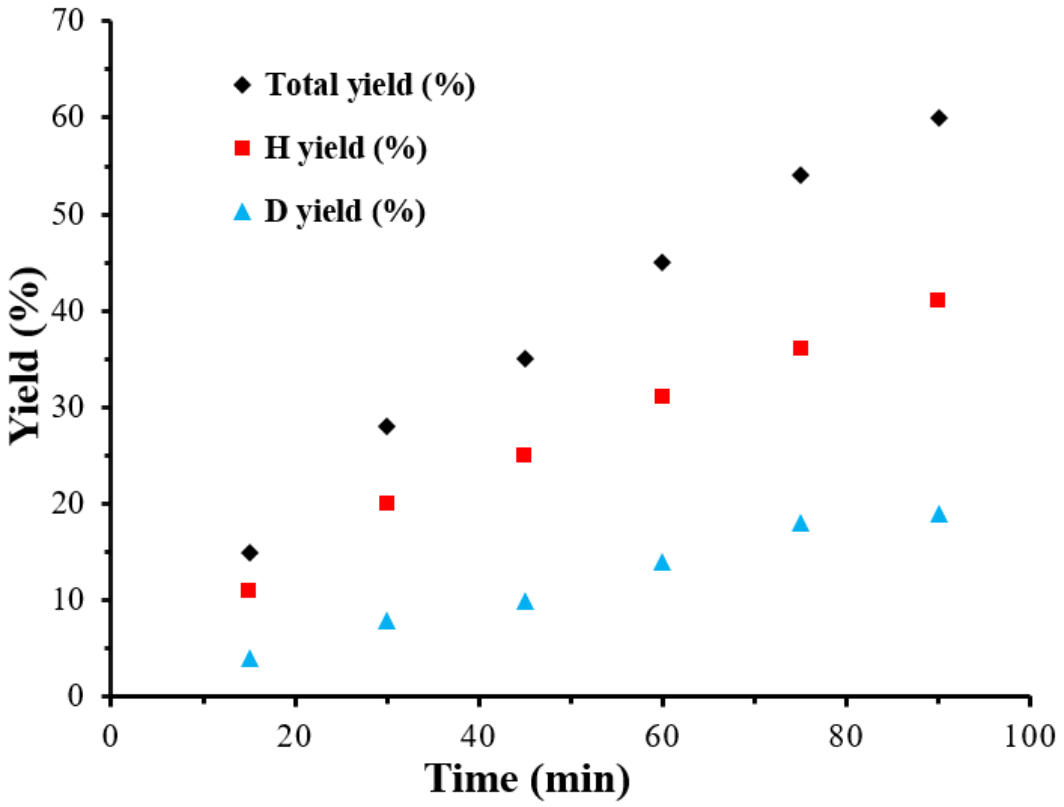


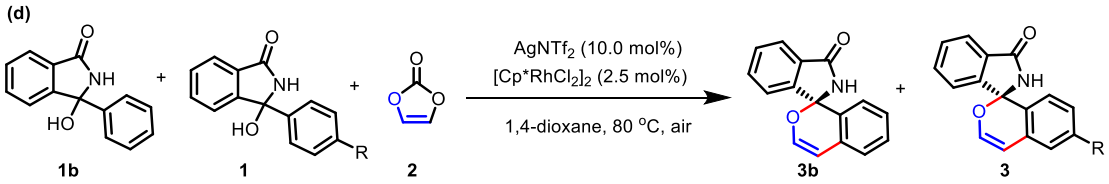
A pressure tube equipped with a stir bar was charged with **1b** (45.0 mg. 0.2 mmol), **D_5-1b** (46.0 mg, 0.2 mmol), vinylene carbonate **2** (17.2 mg, 0.2 mmol), $[Cp^*RhCl_2]_2$ (3.1 mg, 2.5 mol%), $AgNTf_2$ (7.8 mg, 10.0 mol%) in 1,4-dioxane (1.0 mL), The reaction mixture was stirred at 80 ℃ for 12 h under air in an oil bath. After cooling to room temperature, the reaction was monitored by ^{1}H NMR over time.

Table S1. The intermolecular KIE competition experiment over time.



t (min)	Total (%)	**3b** (%)	**D_4-1b** (%)	KIE
15	15	11	4	2.7
30	28	20	8	2.5
45	35	25	10	2.5
60	45	31	14	2.2
75	54	36	18	2
90	60	41	19	2.15

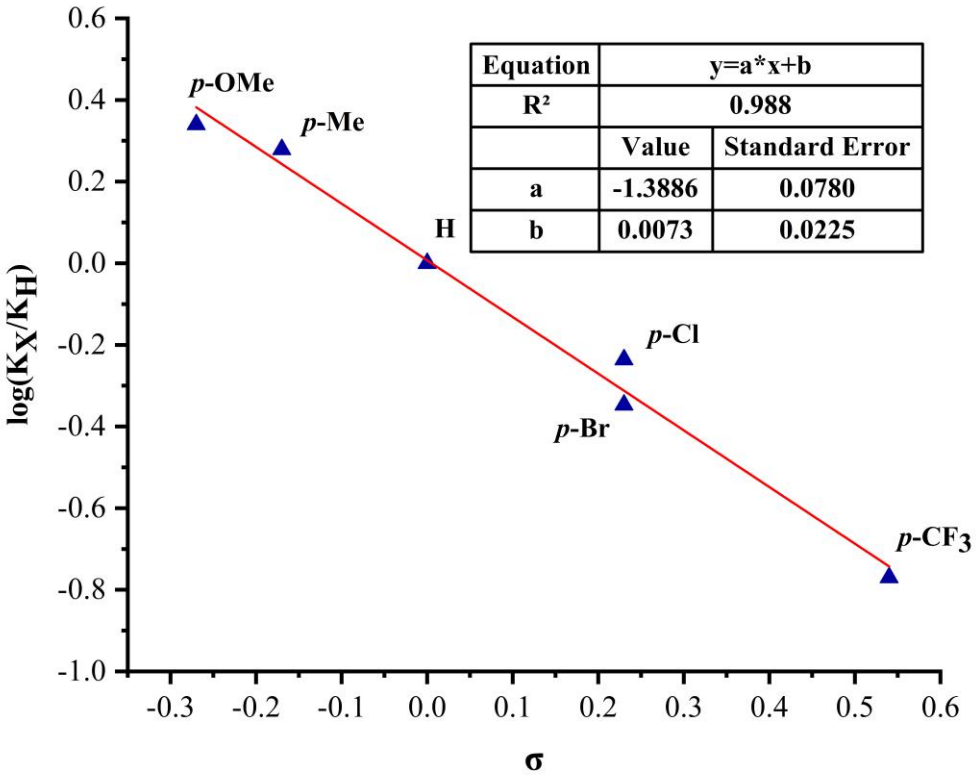




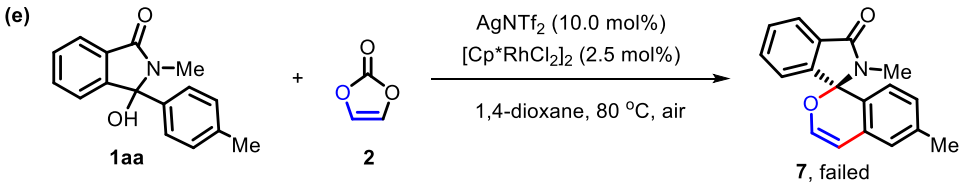
A pressure tube equipped with a stir bar was charged with **1b** (45.0 mg, 0.2 mmol), **1** (0.2 mmol), vinylene carbonate **2** (17.2 mg, 0.2 mmol), $[Cp^*RhCl_2]_2$ (3.1 mg, 2.5 mol%), $AgNTf_2$ (7.8 mg, 10.0 mol%) in 1,4-dioxane (1.0 mL), The reaction mixture was stirred at 80 °C for 12 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The ratio of different products was determined by the ^1H NMR analysis.

Table S2. Hammett plot.

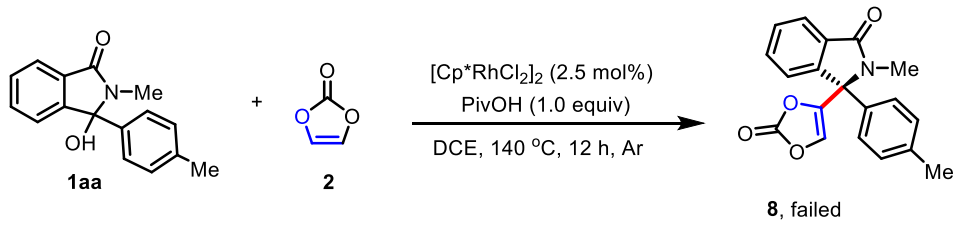
R	σ	Conv. of **2**	Ratio of(**3:3b**)	k_x/k_H	$\log(k_x/k_H)$
p-OMe	-0.27	61%	42%:19%	2.2	0.34
p-Me	-0.17	58%	38%:20%	1.9	0.279
p-Cl	0.23	57%	21%:36%	0.58	-0.236
p-Br	0.23	55%	17%:38%	0.45	-0.347
p-CF_3	0.54	55%	8%:55%	0.17	-0.770



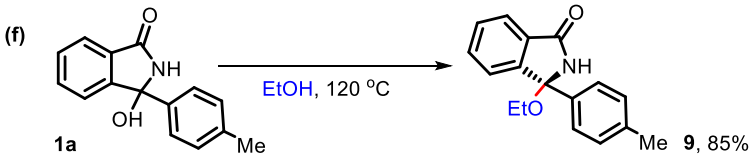
Equation	y=a*x+b	
R^2	0.988	
	Value	Standard Error
a	-1.3886	0.0780
b	0.0073	0.0225



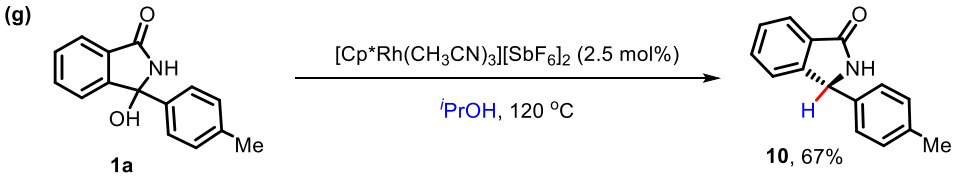
A pressure tube equipped with a stir bar was charged with **1aa** (25.3 mg, 0.1 mmol), vinylene carbonate **2** (25.8 mg, 0.3 mmol), $[Cp^*RhCl_2]_2$ (1.6 mg, 2.5 mol%), $AgNTf_2$ (7.8 mg, 10.0 mol%) in 1,4-dioxane (0.5 mL), The reaction mixture was stirred at 80 °C for 12 h under air in an oil bath. After cooling to room temperature, which was further detected by NMR spectroscopy and showed no desired product **7**.



A pressure tube equipped with a stir bar was charged with **1aa** (25.3 mg, 0.1 mmol), vinylene carbonate **2** (25.8 mg, 0.3 mmol), $[Cp^*RhCl_2]_2$ (1.6 mg, 2.5 mol%), PivOH (10.2 mg, 1.0 equiv) in DCE (0.5 mL), The reaction mixture was stirred at 140 °C for 12 h under argon atmosphere in an oil bath. After cooling to room temperature, which was further detected by NMR spectroscopy and showed no desired product **8**.

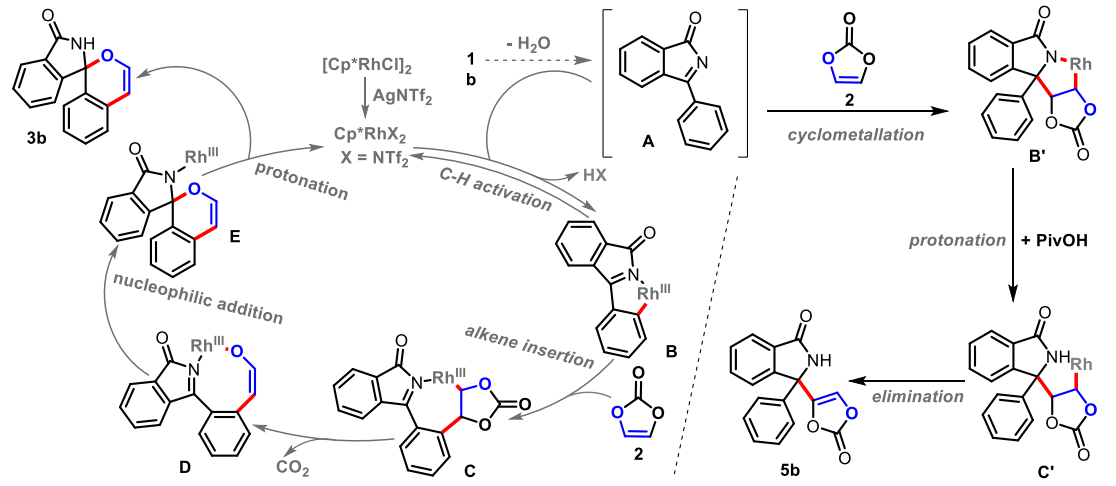


A pressure tube equipped with a stir bar was charged with **1a** (23.9 mg, 0.1 mmol) in EtOH (0.5 mL), The reaction mixture was stirred at 120 °C for 12 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford the product **9**. White solid (22.7 mg, 85% yield). PE/EA = 5:1, R_f = 0.40. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 7.3 Hz, 1H), 7.51 – 7.41 (m, 4H), 7.27 (d, *J* = 8.1 Hz, 1H), 7.13 (d, *J* = 7.8 Hz, 2H), 6.40 (s, 1H), 3.57 – 3.39 (m, 1H), 3.19 – 3.03 (m, 1H), 2.32 (s, 3H), 1.20 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.8, 147.4, 138.3, 137.2, 132.7, 130.9, 129.3, 129.2, 125.4, 123.6, 123.0, 91.8, 58.4, 21.0, 15.2. HRMS (ESI) m/z Calcd for $C_{17}H_{18}NO_2$ $[M+H]^+$ 268.1368, found 268.1361.



A pressure tube equipped with a stir bar was charged with **1a** (23.9 mg, 0.1 mmol), $[Cp^*Rh(CH_3CN)_3][SbF_6]_2$ (2.5 mol%) in ^{i}PrOH (0.5 mL), The reaction mixture was stirred at 120 °C for 12 h under air in an oil bath. After cooling to room temperature, all volatiles were removed under reduced pressure. The residue was purified by silica gel chromatography using PE/EA eluent to afford the product **10**. White solid (14.9 mg, 67% yield). PE/EA = 5:1, R_f = 0.35. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.86 (d, *J* = 7.4 Hz, 1H), 7.52 – 7.42 (m, 2H), 7.22 (d, *J* = 7.4 Hz, 1H), 7.14 (s, 5H), 5.58 (s, 1H), 2.32 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 148.2, 138.3, 135.4, 132.1, 130.9, 129.7, 128.2, 126.7, 123.7, 123.2, 60.6, 21.1. HRMS (ESI) m/z Calcd for $C_{15}H_{14}NO$ $[M+H]^+$ 224.1031, found 224.1035.

Possible reaction mechanism



Scheme S1. Proposed mechanism.

G. References

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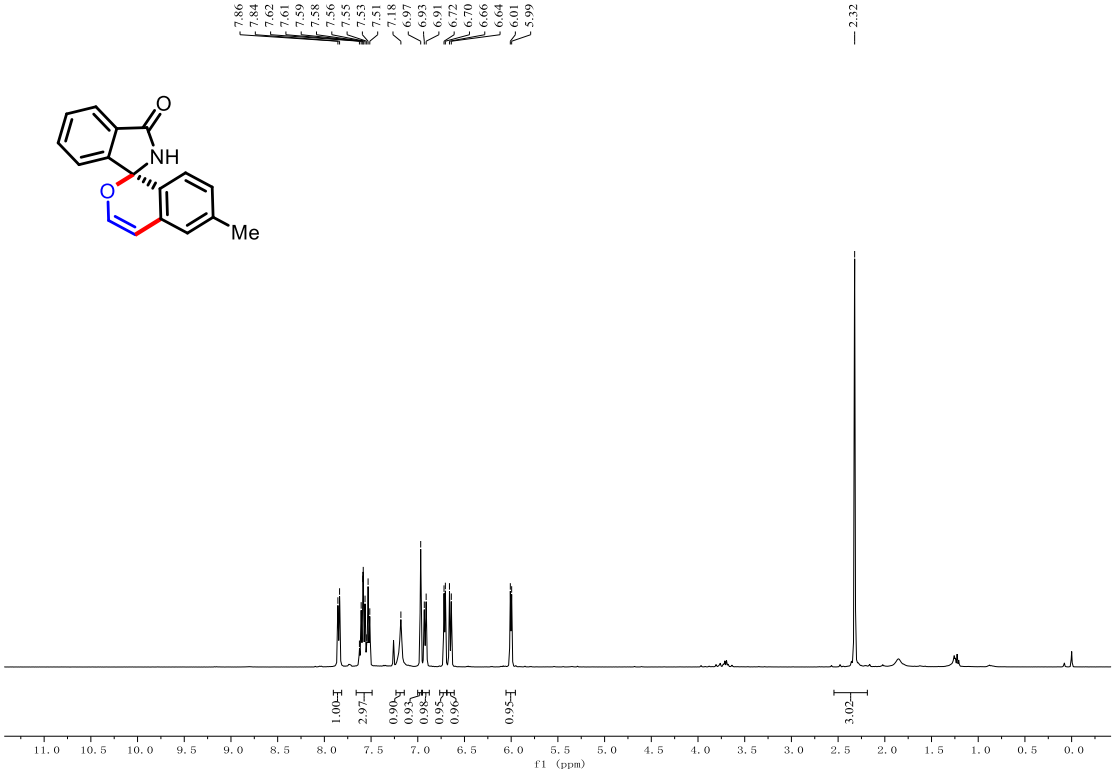
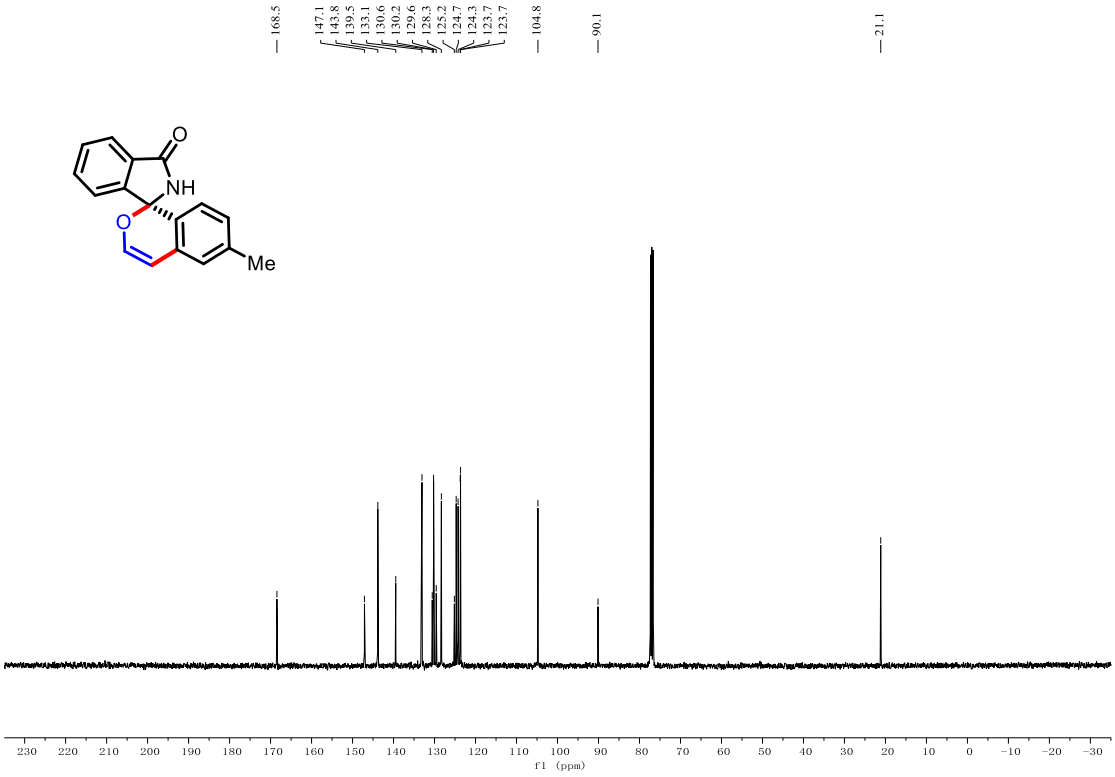
13) A. Suneja R. A. Unhale and V. K. Singh, *Org. Lett*., 2017, **19**, 476-479.

H. NMR spectra

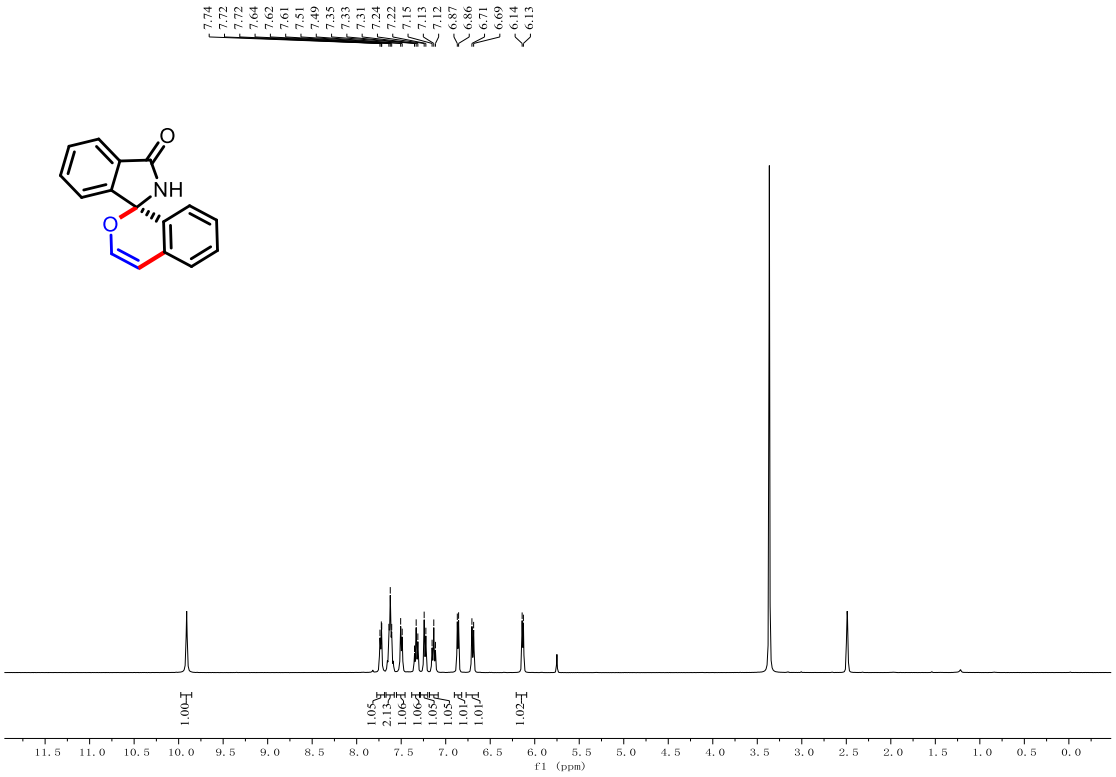
^{1}H NMR of **3a** (400 MHz, $CDCl_3$)



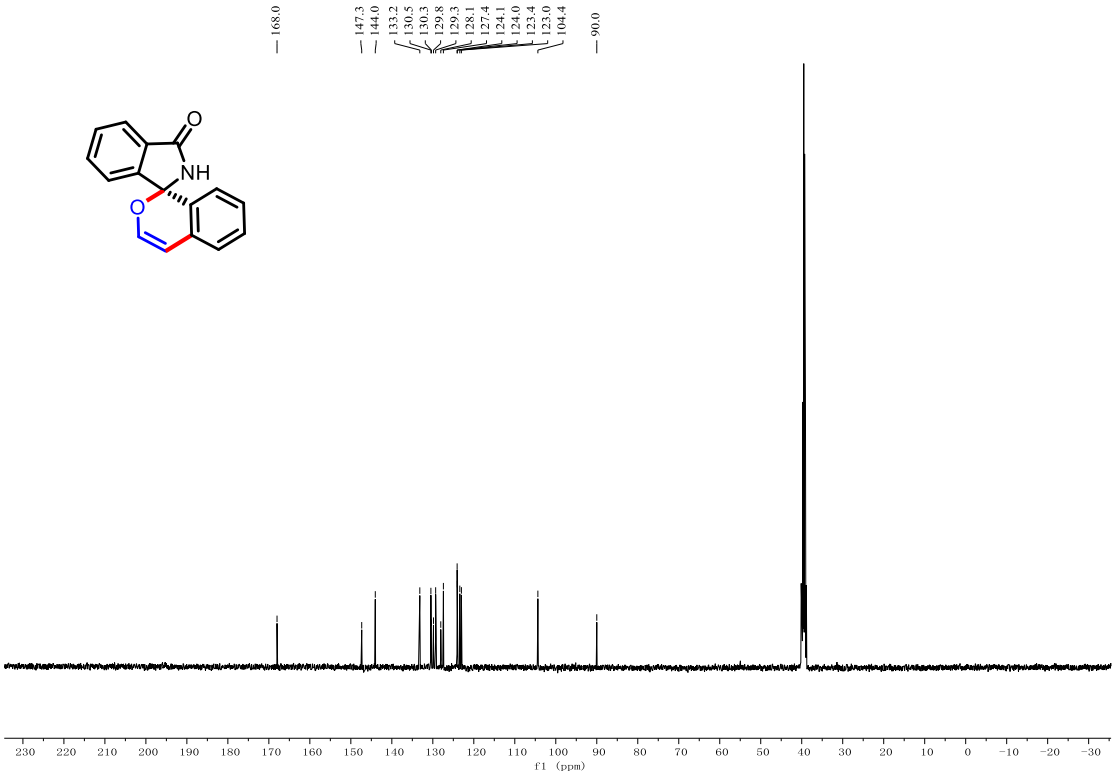
^{13}C NMR of **3a** (400 MHz, $CDCl_3$)



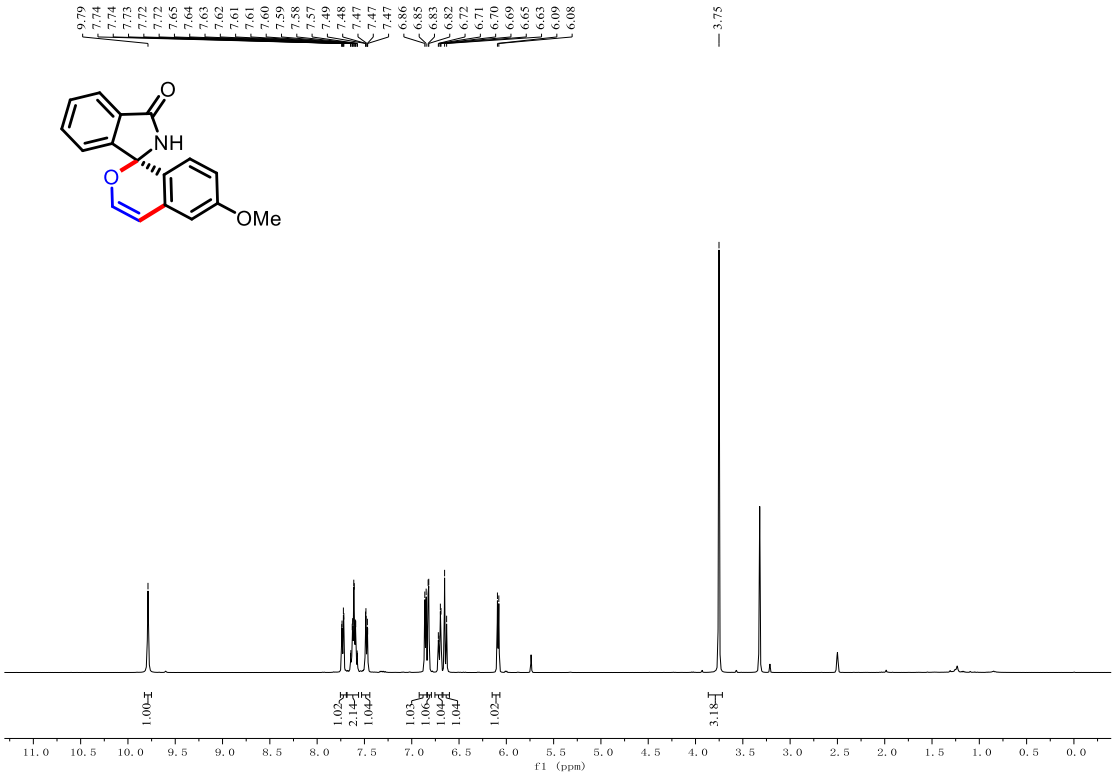
^{1}H NMR of **3b** (400 MHz, DMSO)



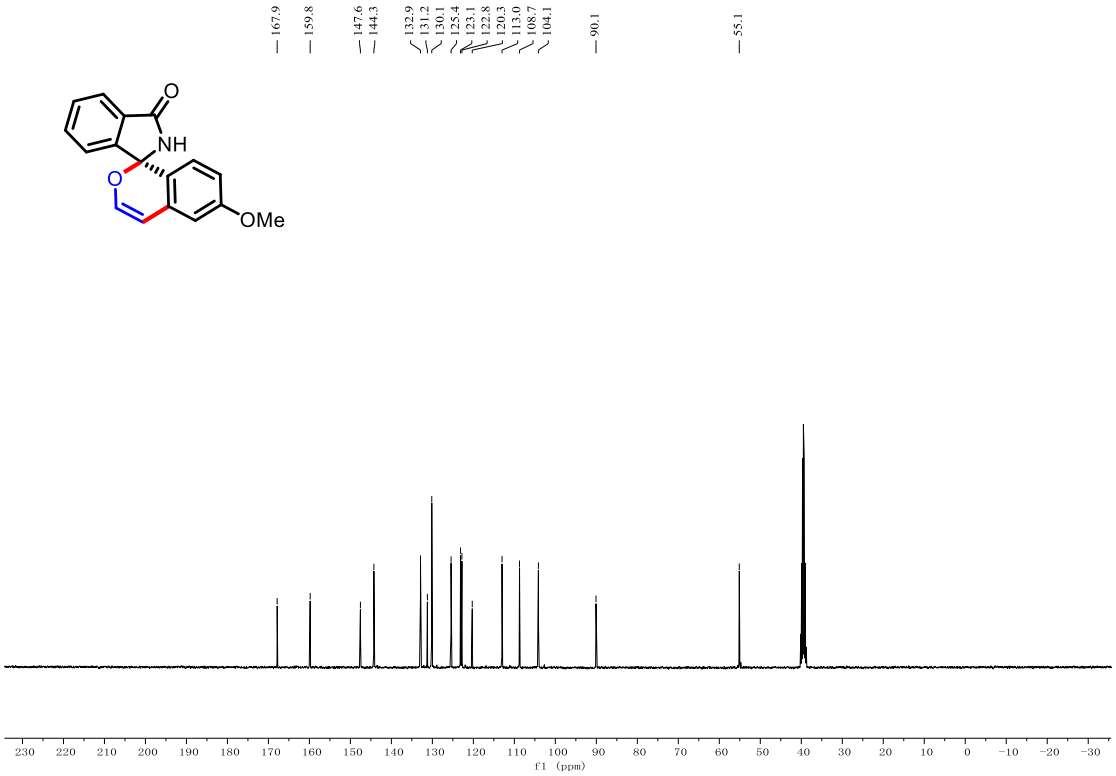
^{13}C NMR of **3b** (400 MHz, DMSO)



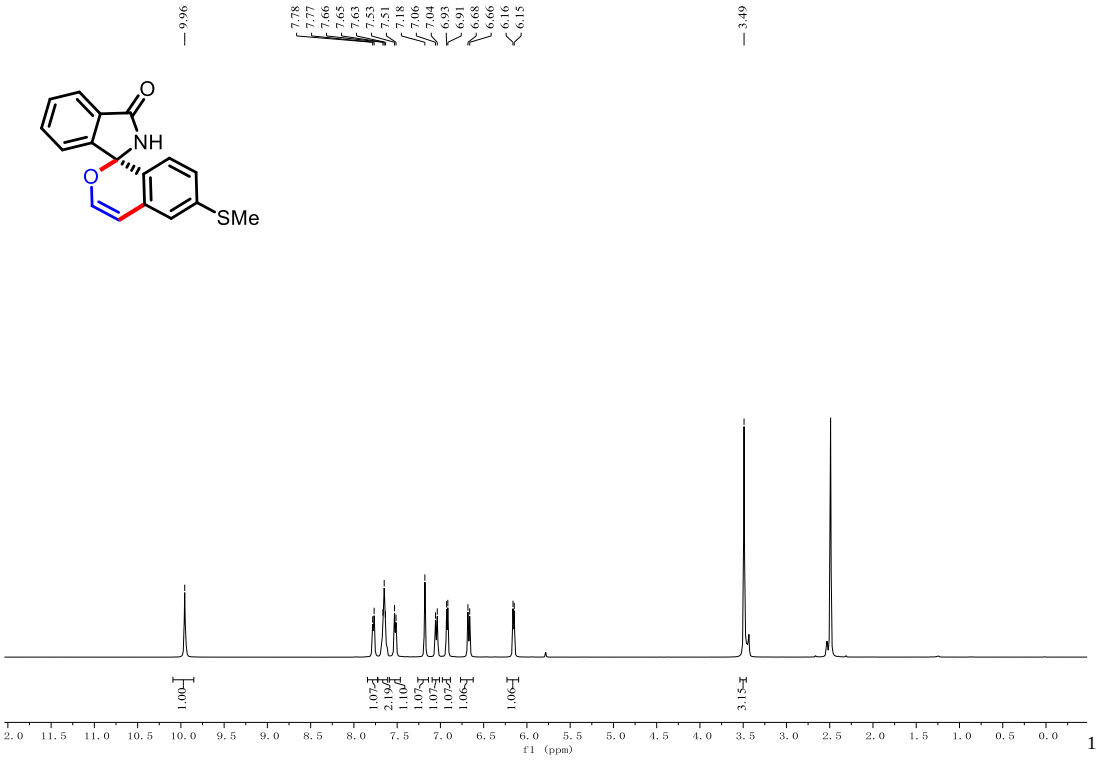
^{1}H NMR of **3c** (400 MHz, DMSO)



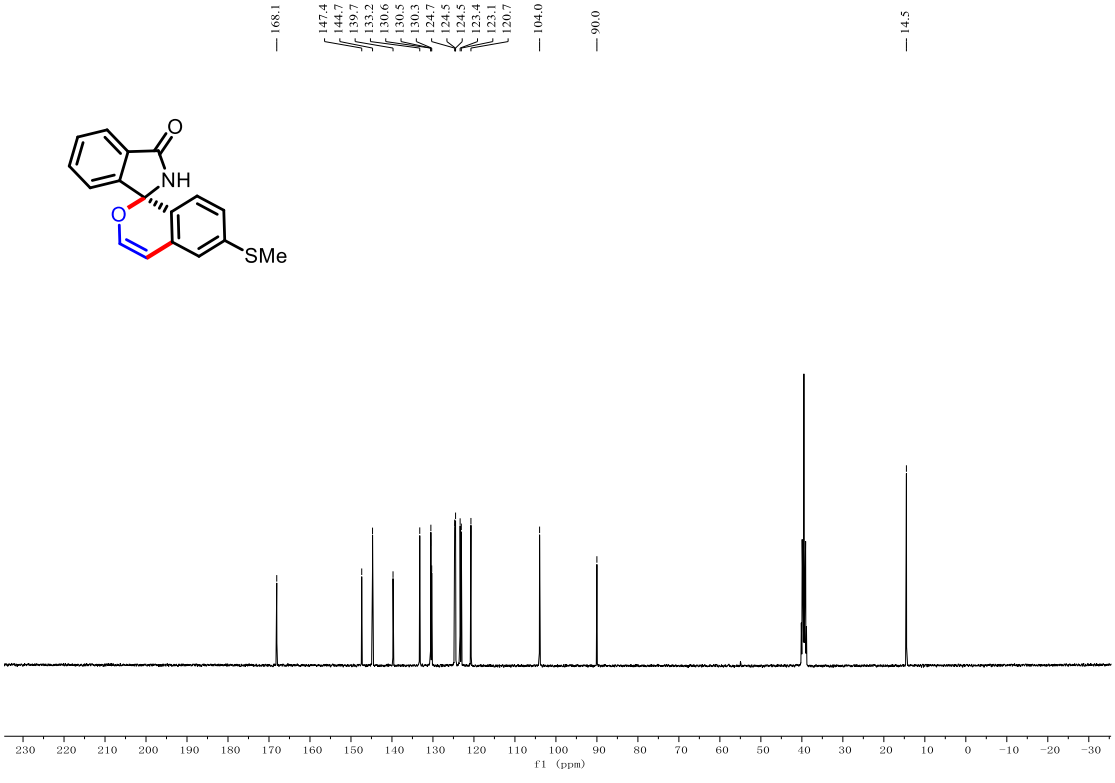
^{13}C NMR of **3c** (400 MHz, DMSO)



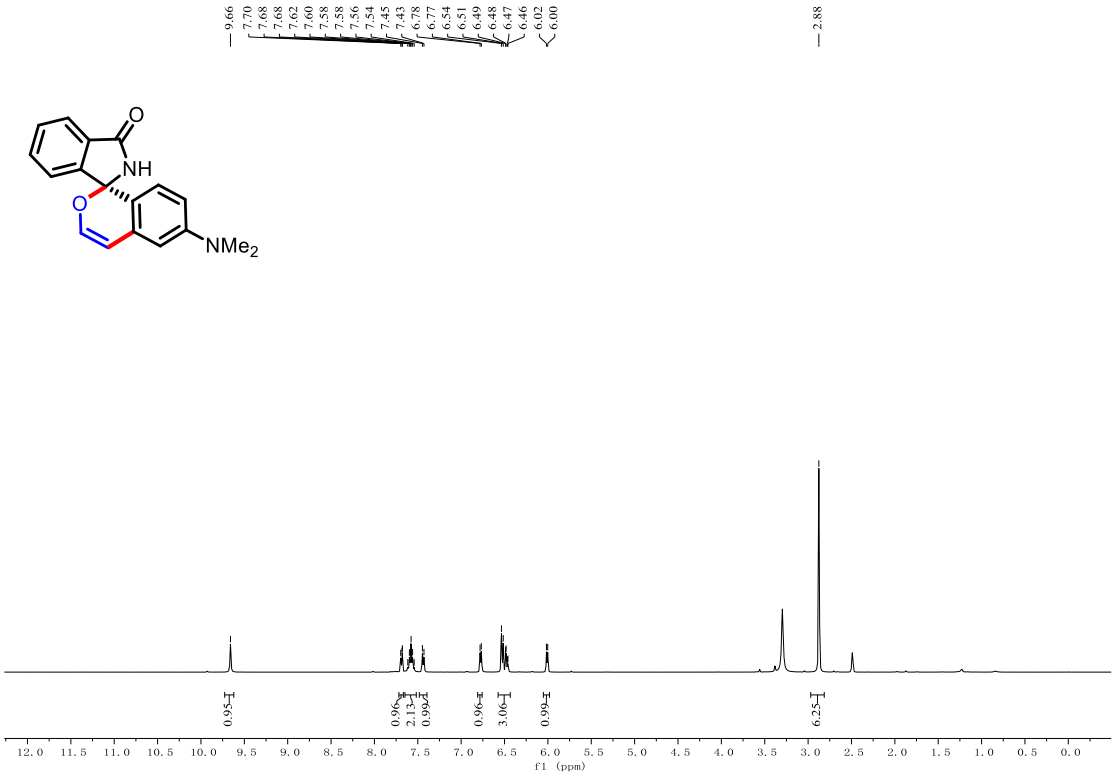
^{1}H NMR of **3d** (400 MHz, DMSO)



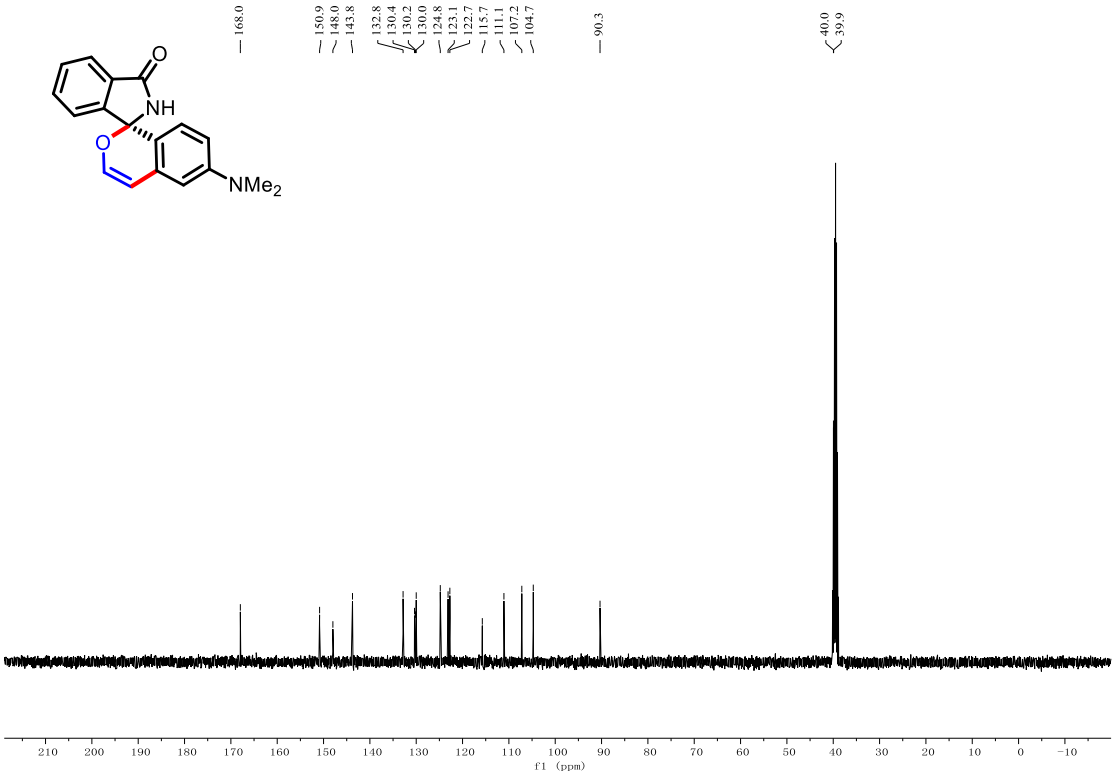
^{3}C NMR of **3d** (400 MHz, DMSO)



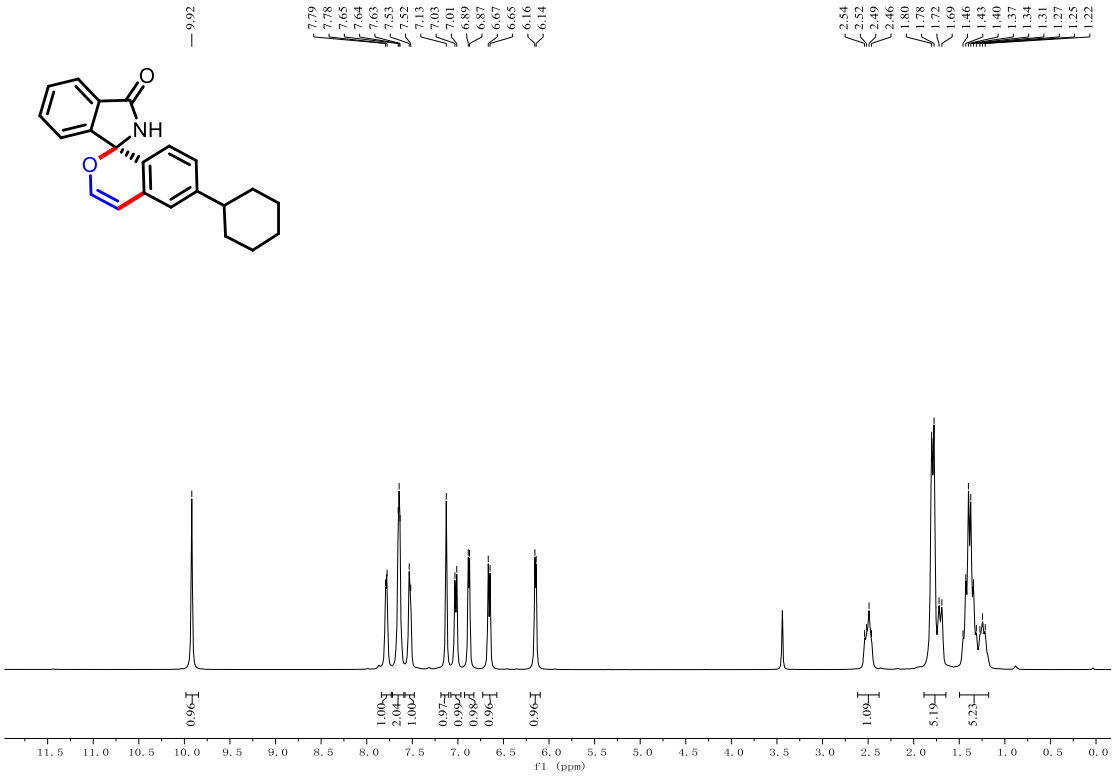
^{1}H NMR of **3e** (400 MHz, DMSO)



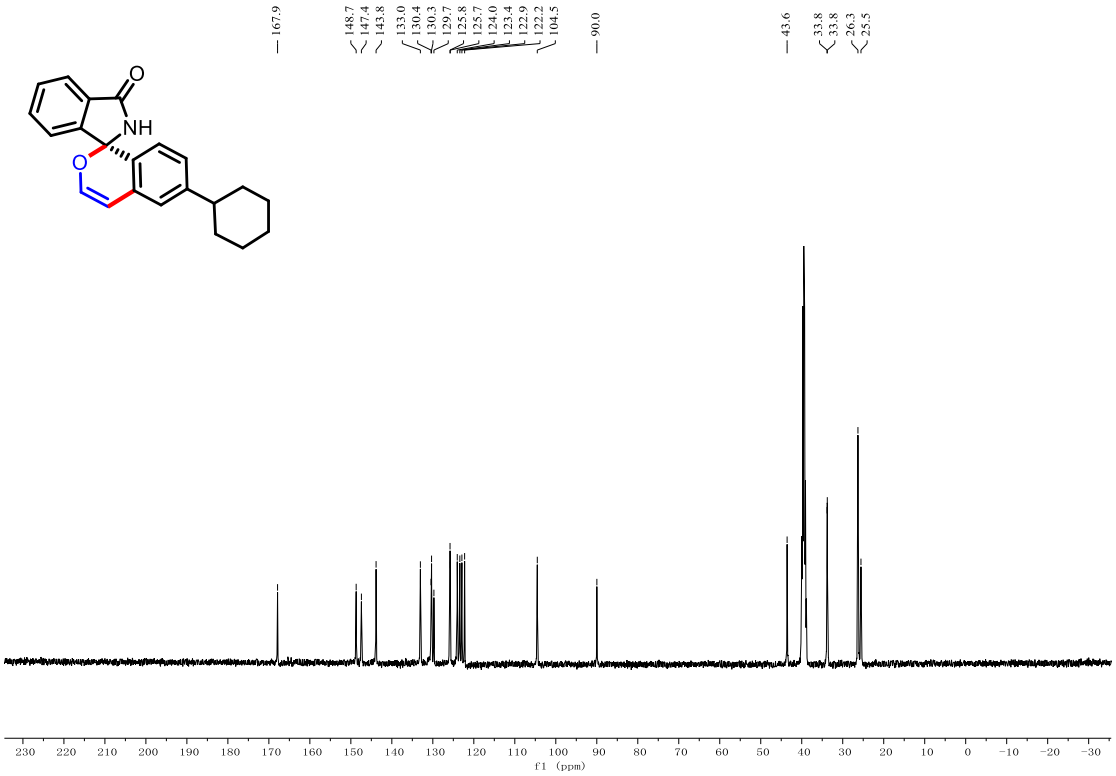
^{13}C NMR of **3e** (400 MHz, DMSO)



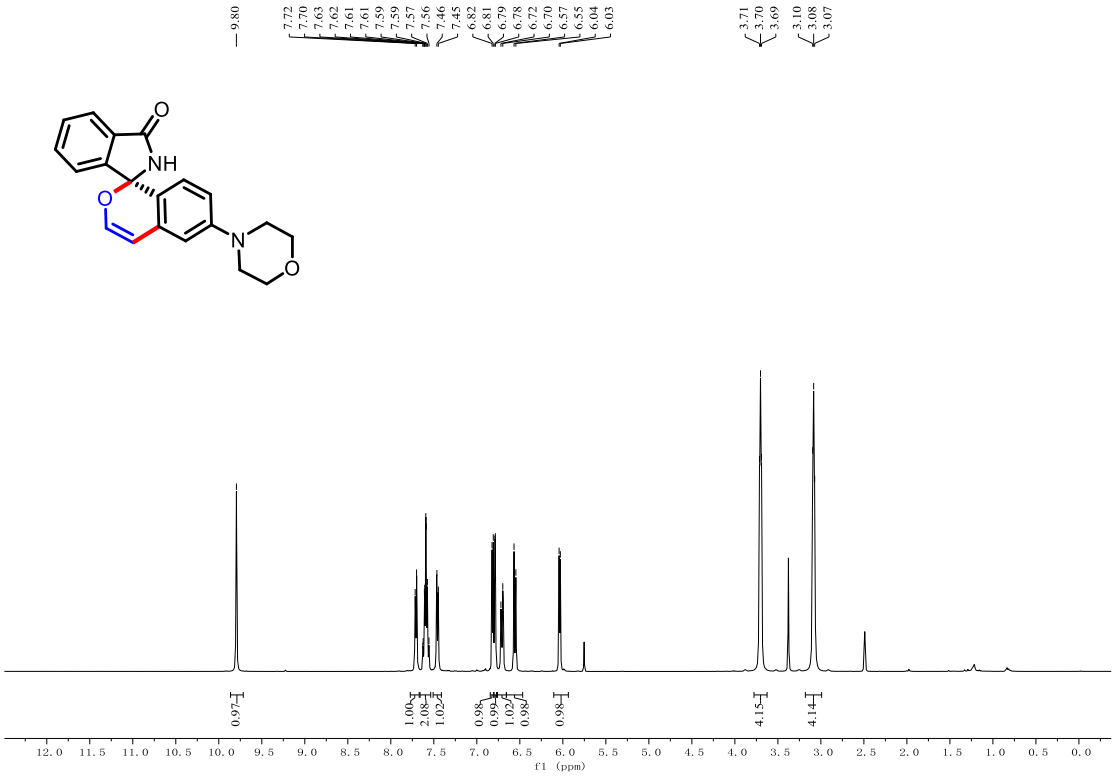
^{1}H NMR of **3f** (400 MHz, DMSO)



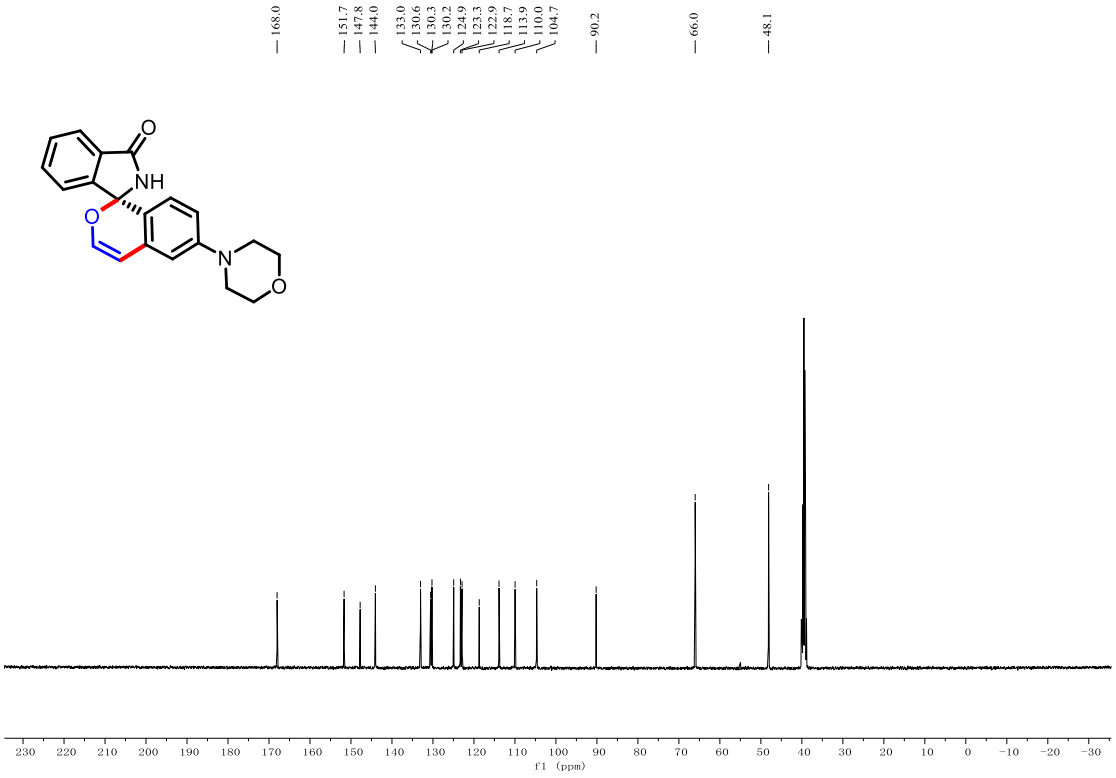
^{13}C NMR of **3f** (400 MHz, DMSO)



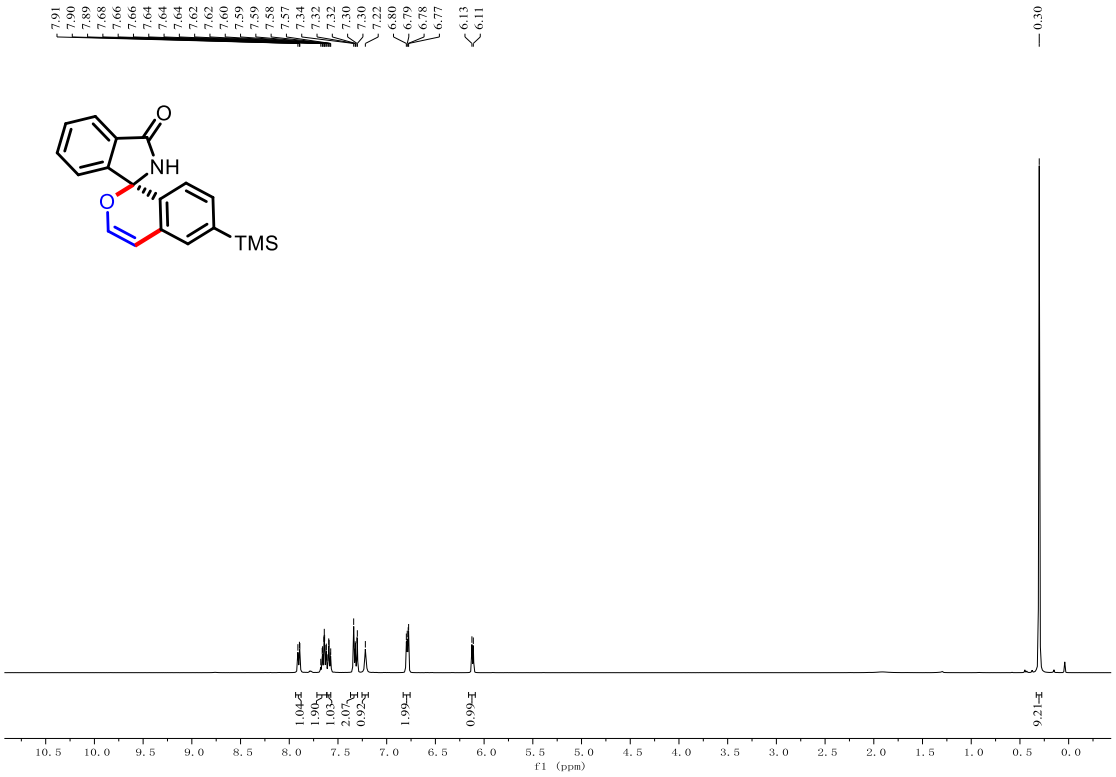
^{1}H NMR of **3g** (400 MHz, DMSO)



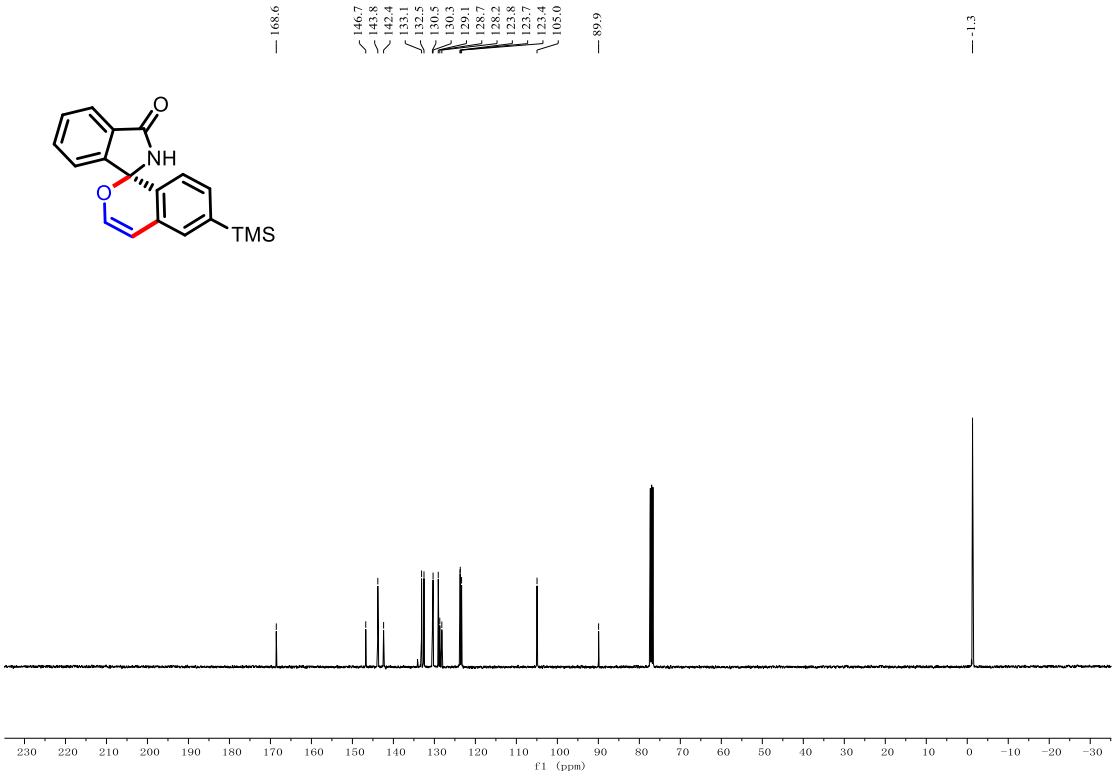
^{13}C NMR of **3g** (400 MHz, DMSO)



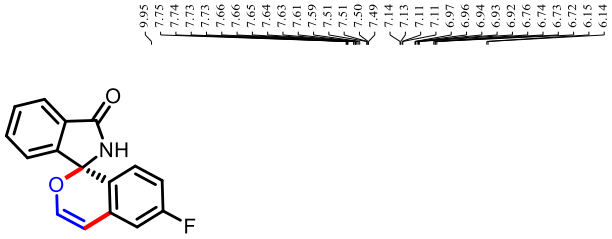
^{1}H NMR of **3h** (400 MHz, $CDCl_3$)

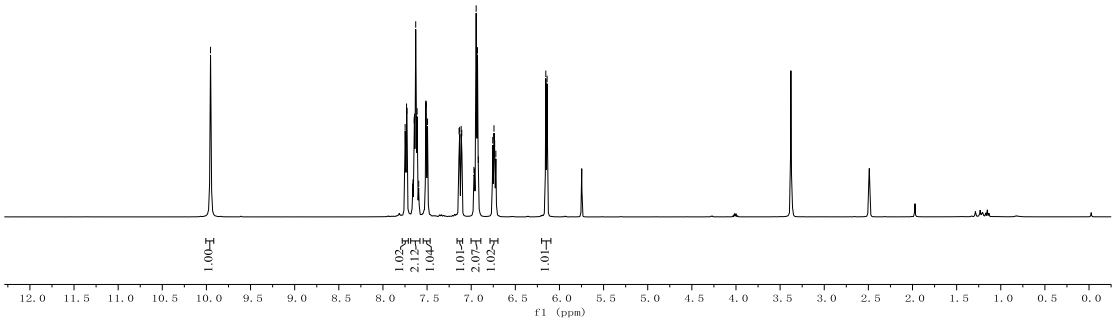


^{13}C NMR of **3h** (400 MHz, $CDCl_3$)

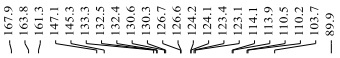


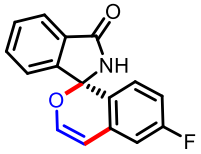
^{1}H NMR of **3i** (400 MHz, DMSO)

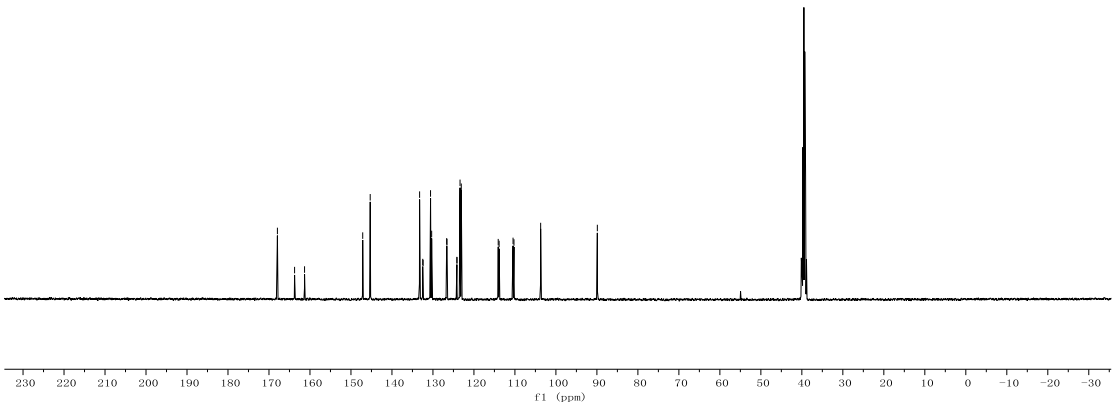




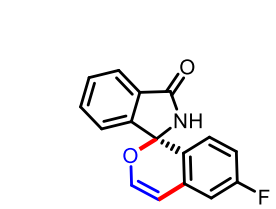
^{13}C NMR of **3i** (400 MHz, DMSO)



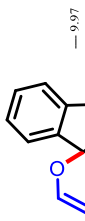




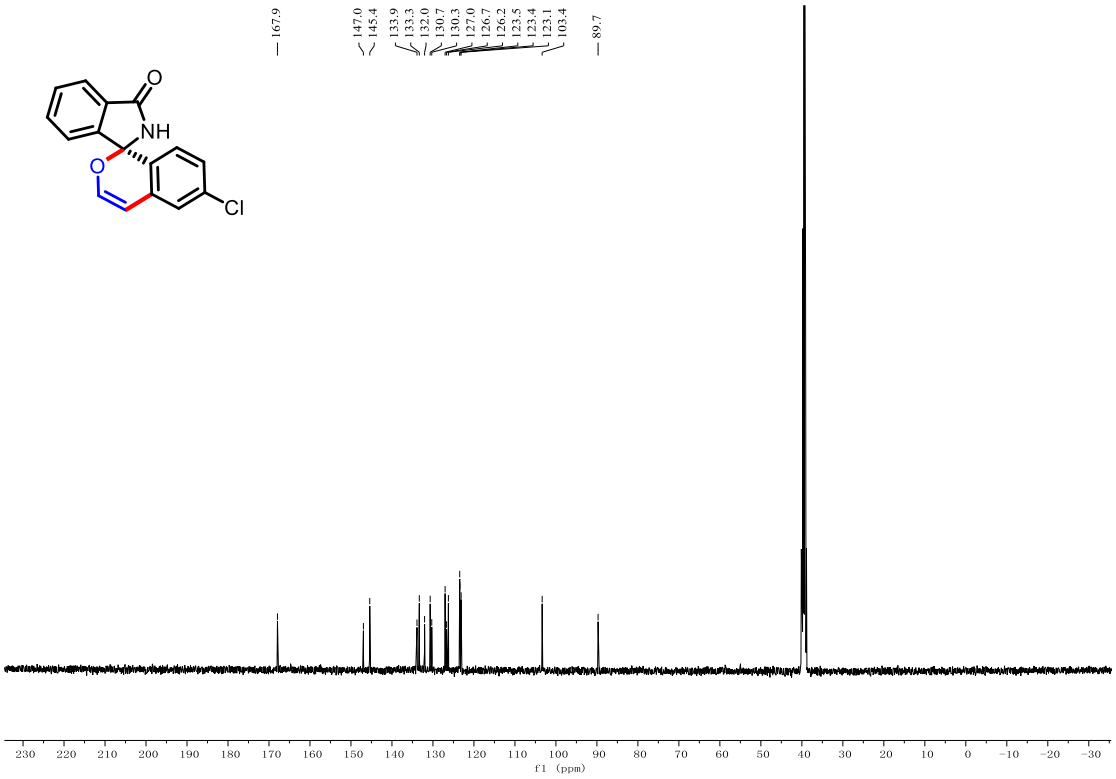
^{19}F NMR of **3i** (400 MHz, DMSO)



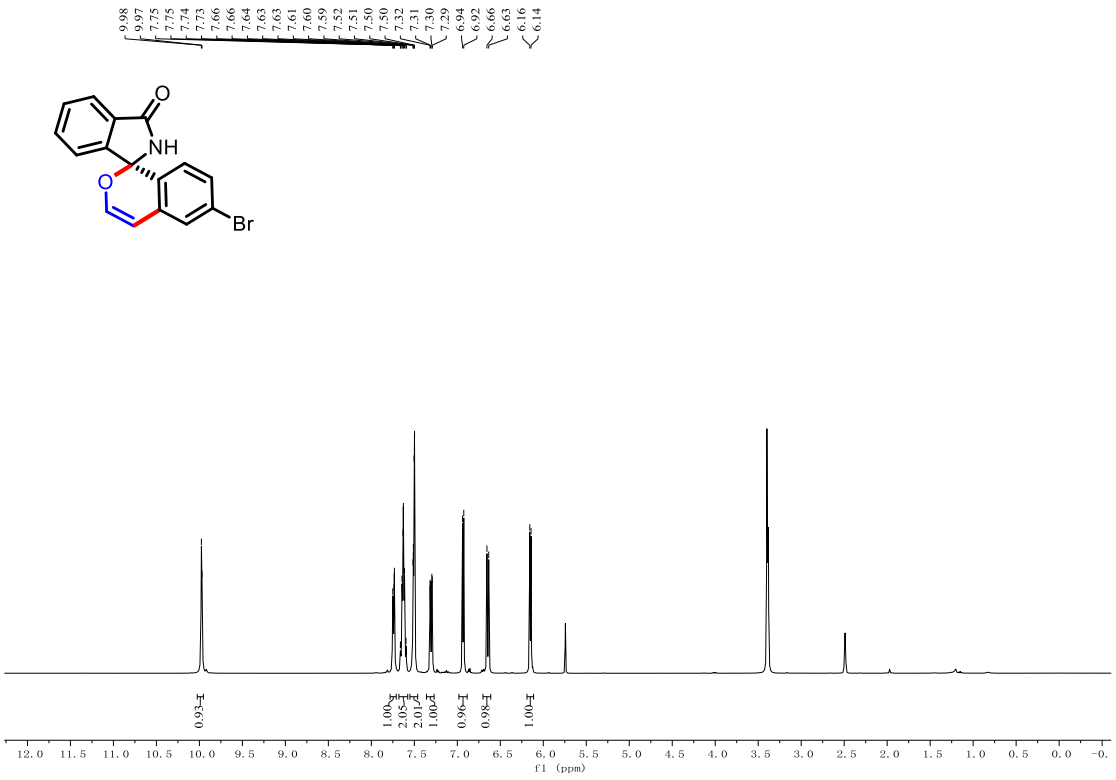
^{1}H NMR of **3j** (400 MHz, DMSO)



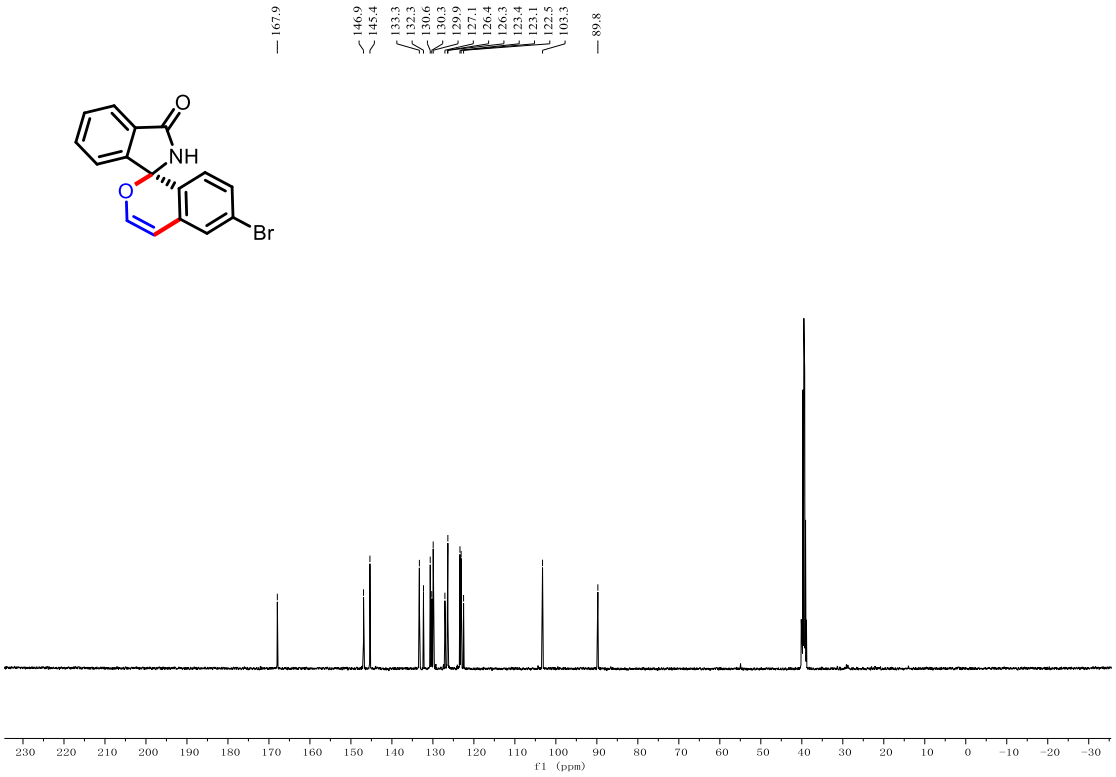
^{13}C NMR of **3j** (400 MHz, DMSO)



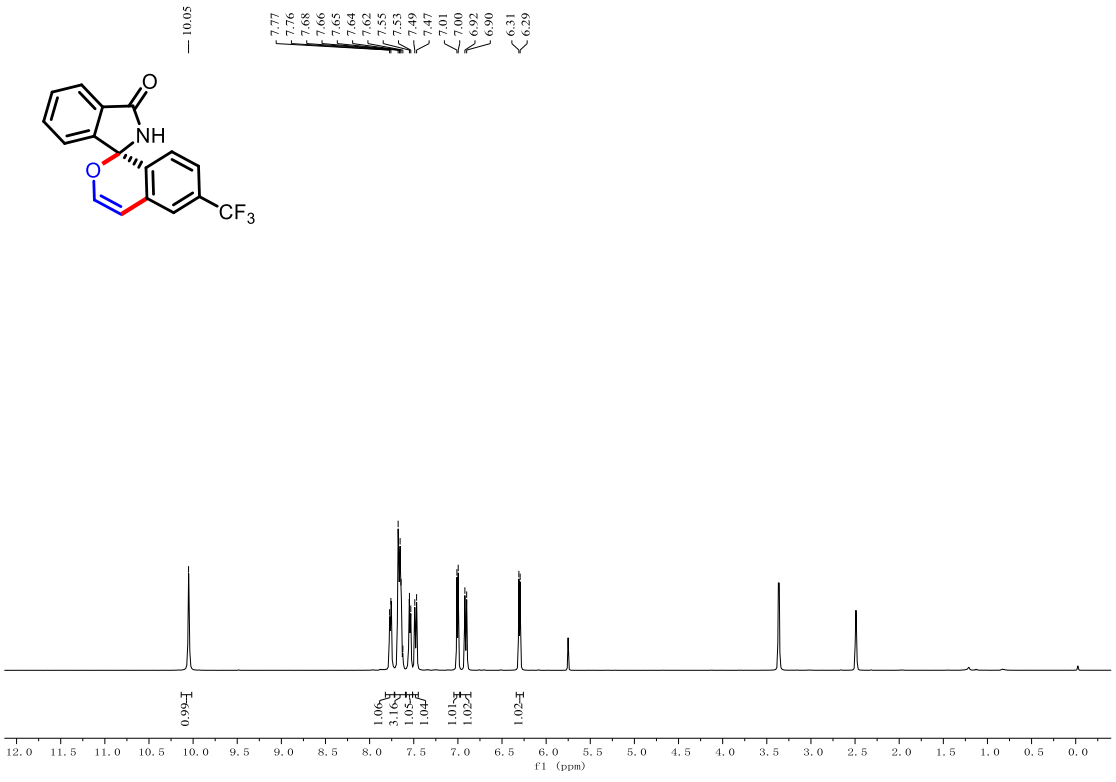
^{1}H NMR of **3k** (400 MHz, DMSO)



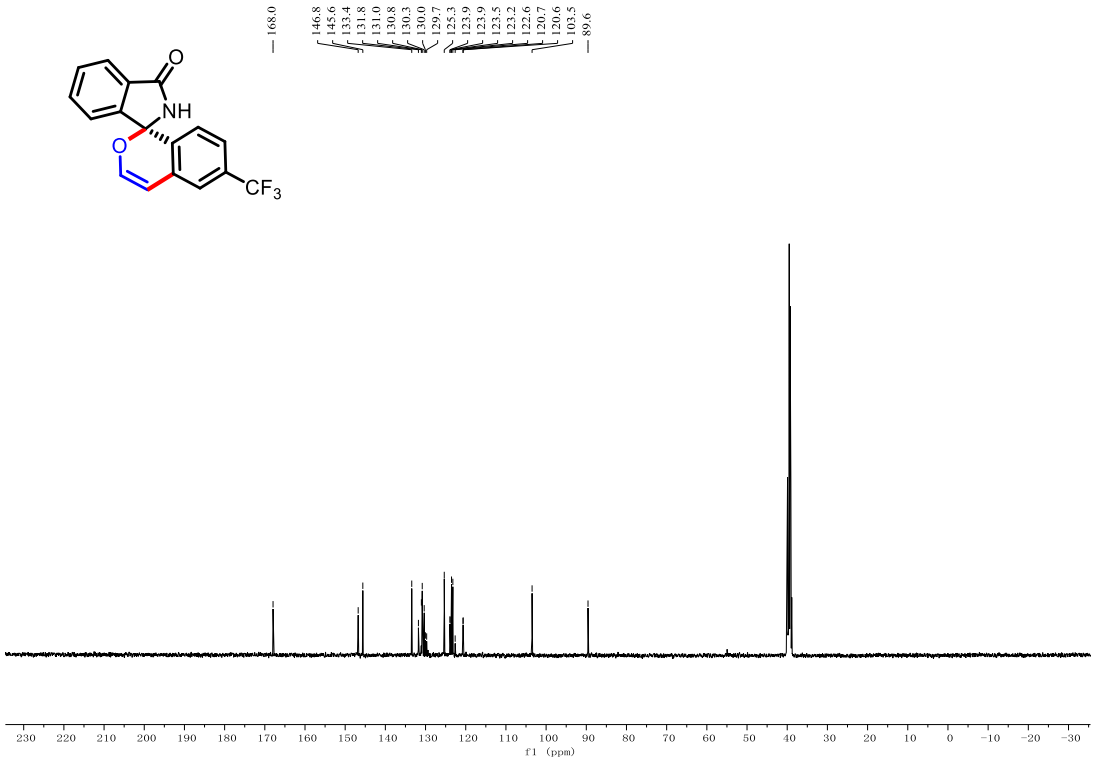
^{13}C NMR of **3k** (400 MHz, DMSO)



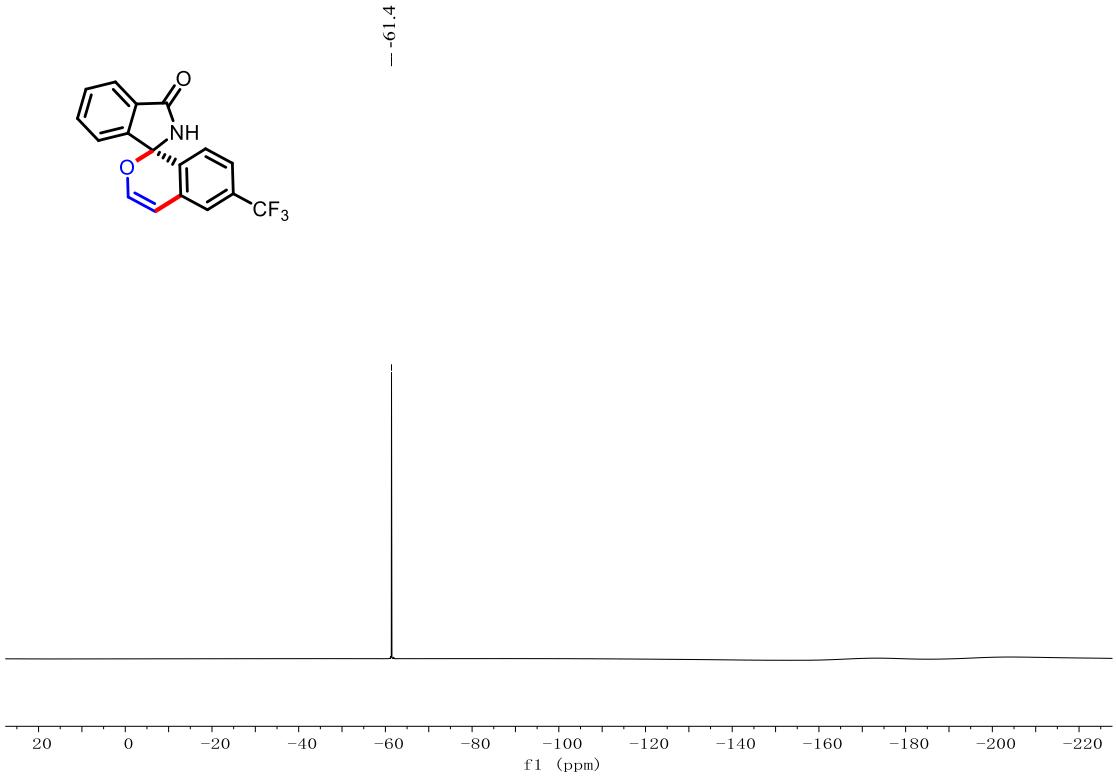
^{1}H NMR of **3l** (400 MHz, DMSO)



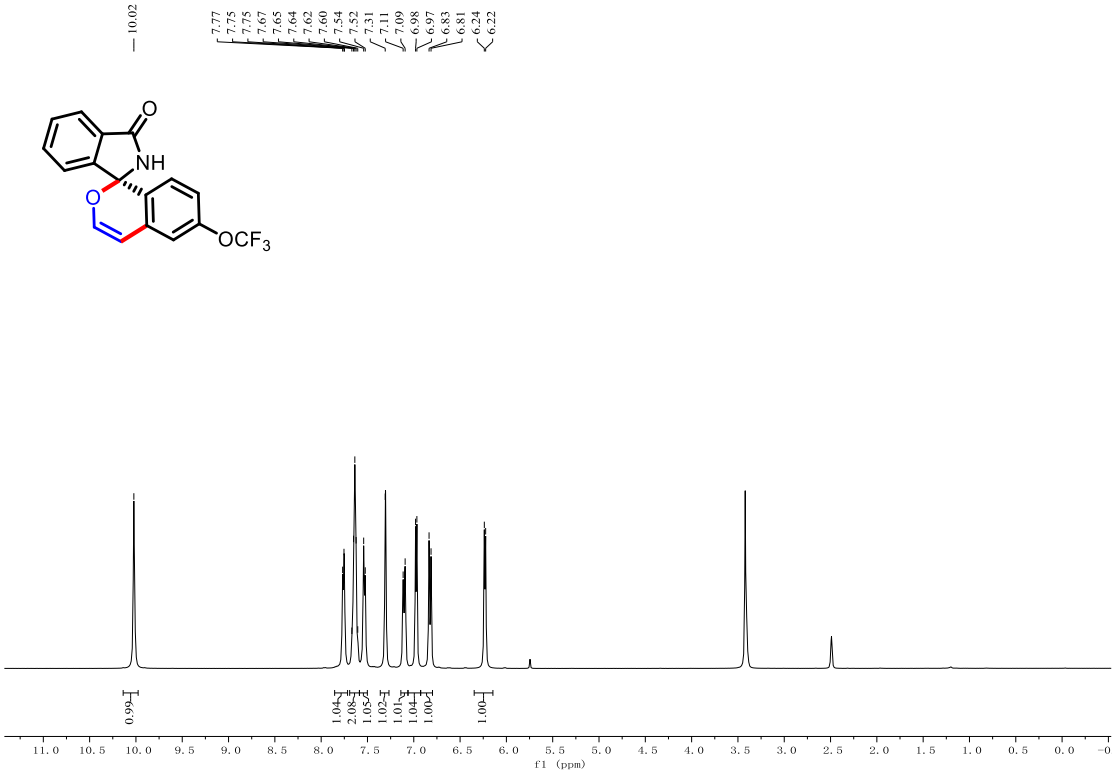
^{13}C NMR of **3l** (400 MHz, DMSO)



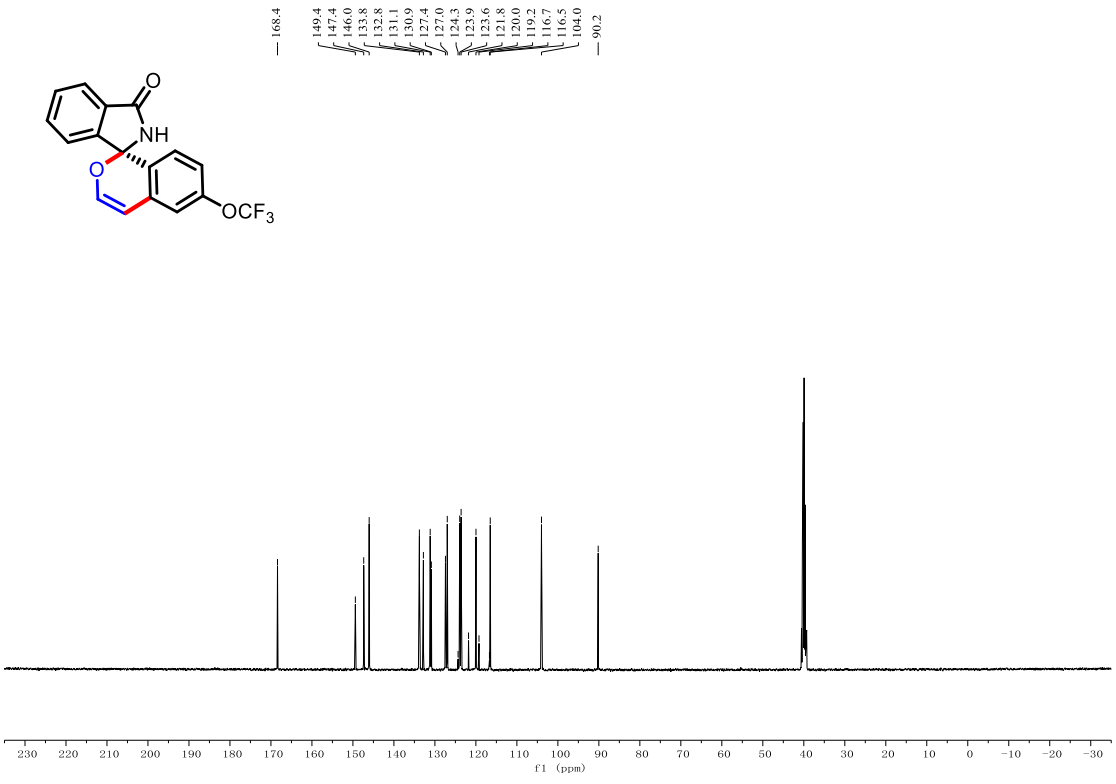
^{19}F NMR of **3l** (400 MHz, DMSO)



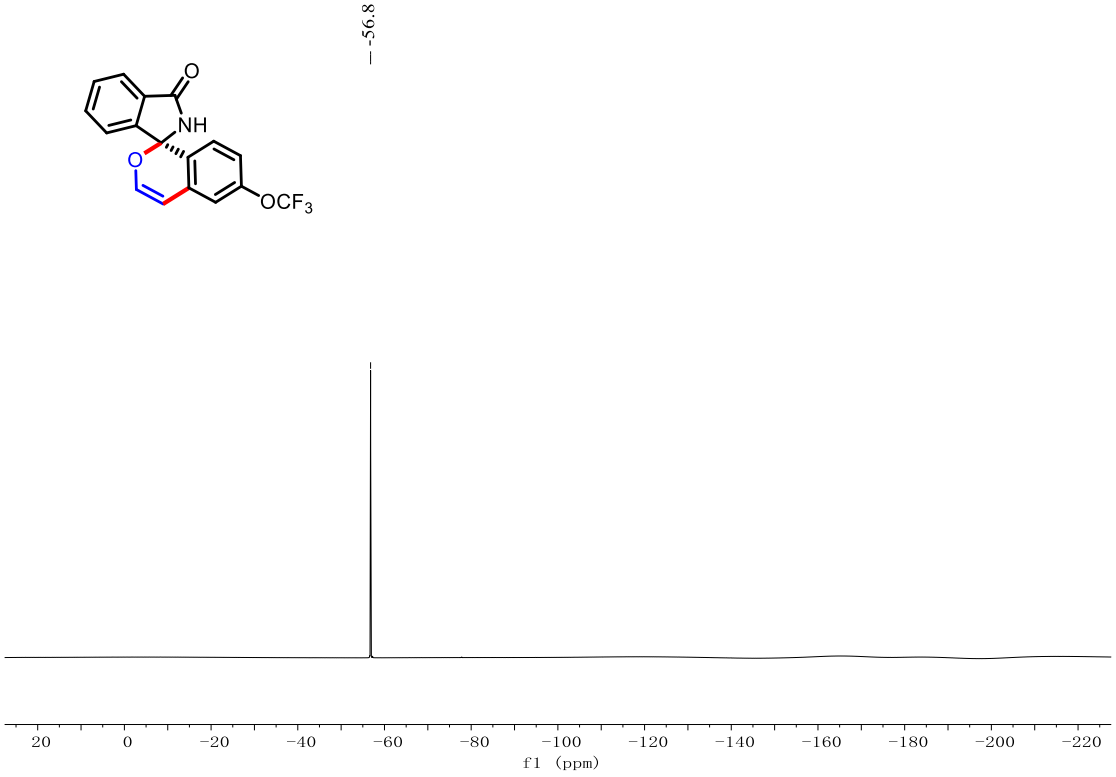
^{1}H NMR of **3m** (400 MHz, DMSO)



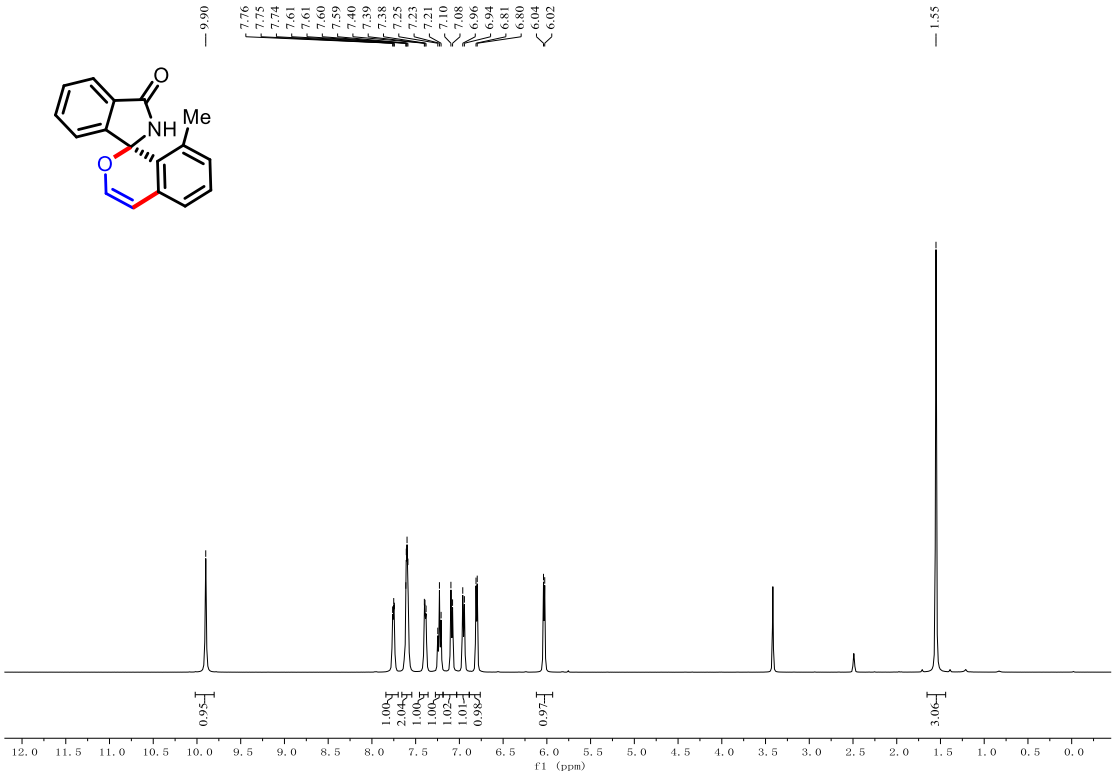
^{13}C NMR of **3m** (400 MHz, DMSO)



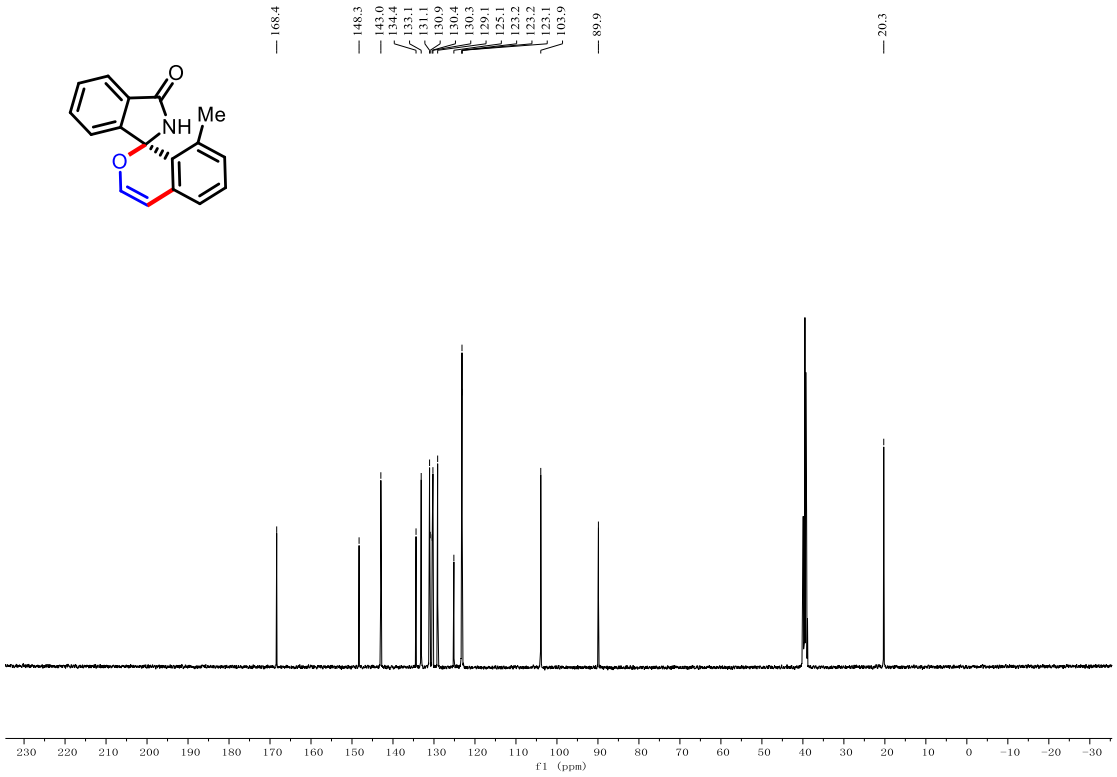
^{19}F NMR of **3m** (400 MHz, DMSO)



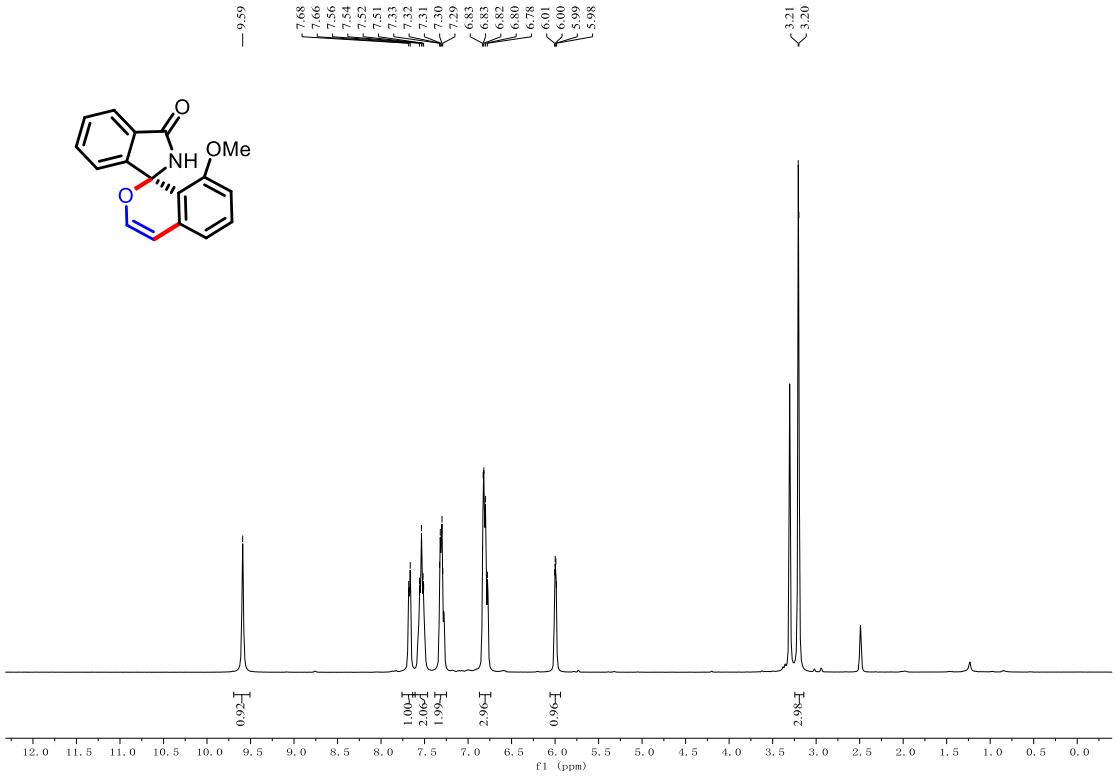
^{1}H NMR of **3n** (400 MHz, DMSO)



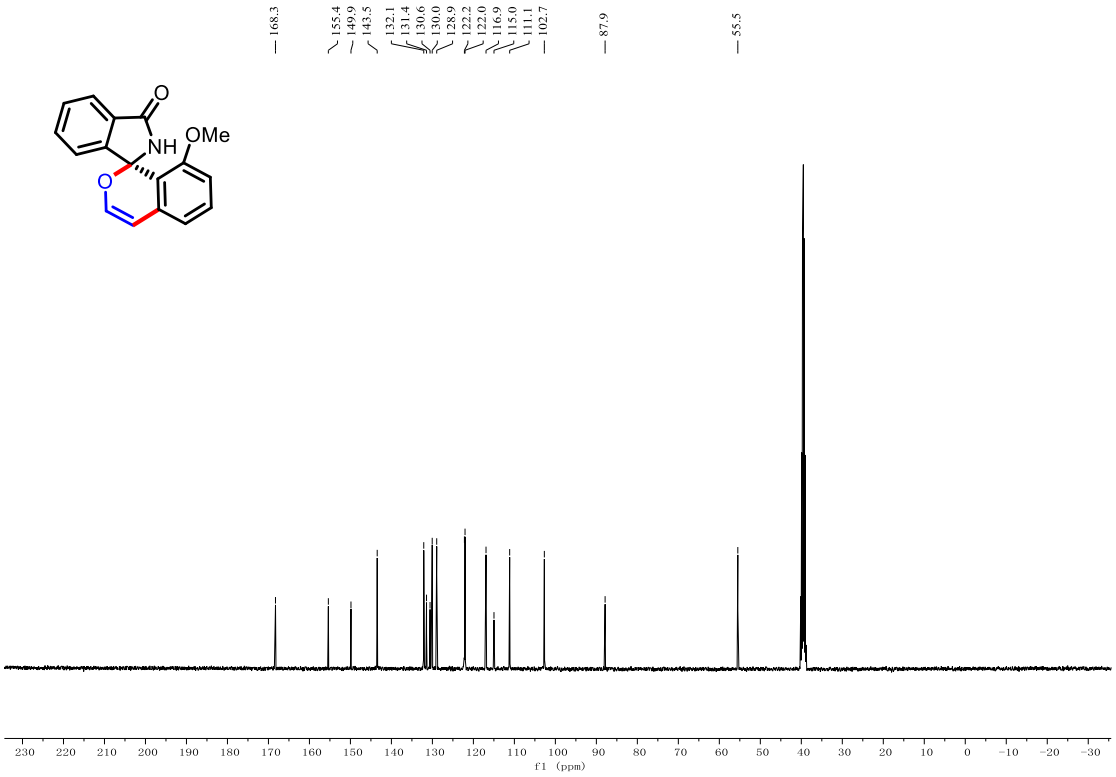
^{13}C NMR of **3n** (400 MHz, DMSO)



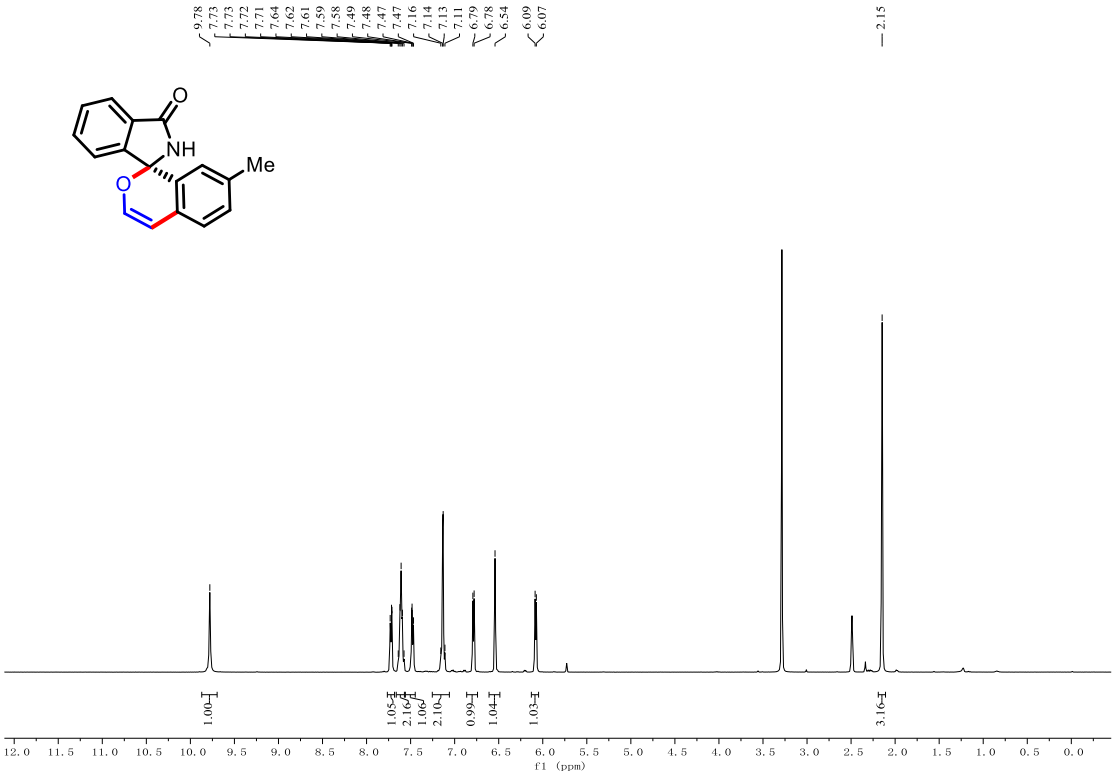
^{1}H NMR of **3o** (400 MHz, DMSO)



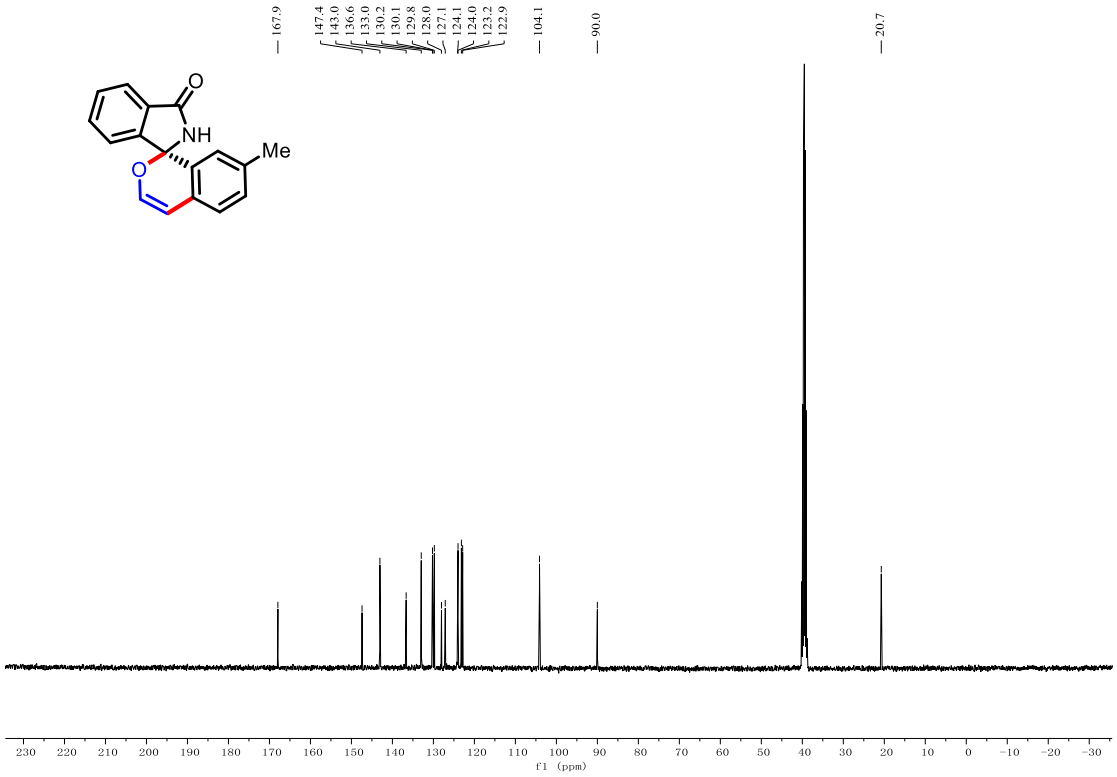
^{13}C NMR of **3o** (400 MHz, DMSO)



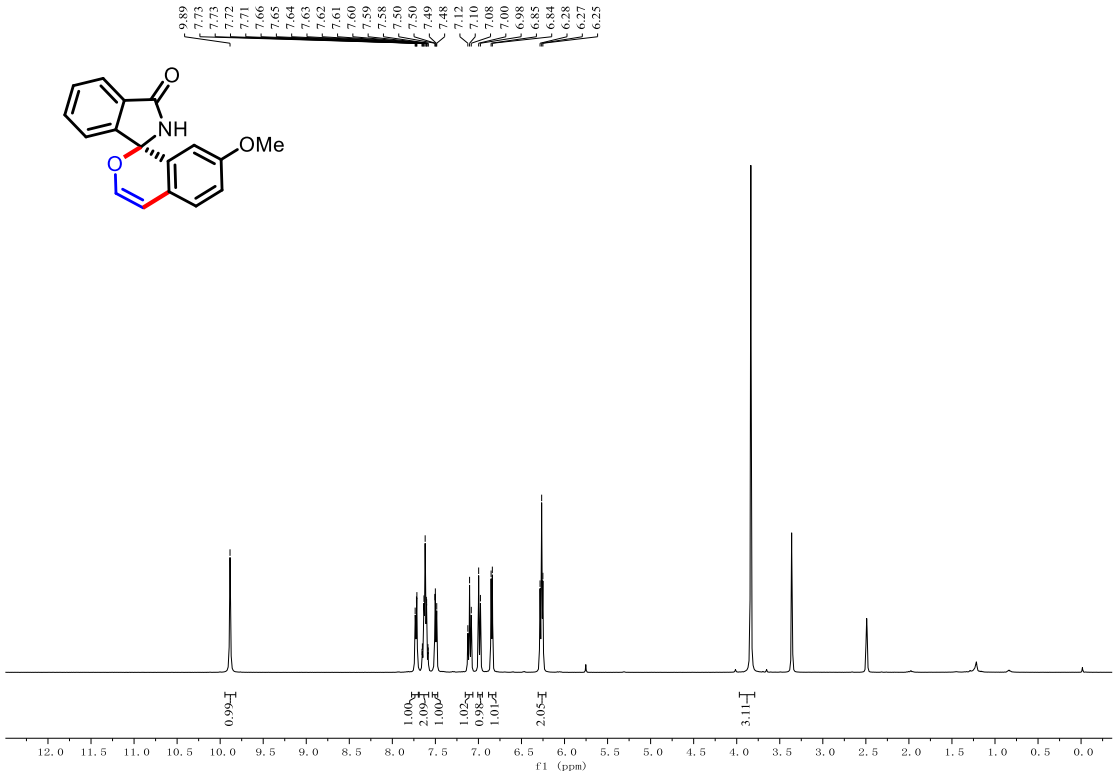
^{1}H NMR of **3p** (400 MHz, DMSO)



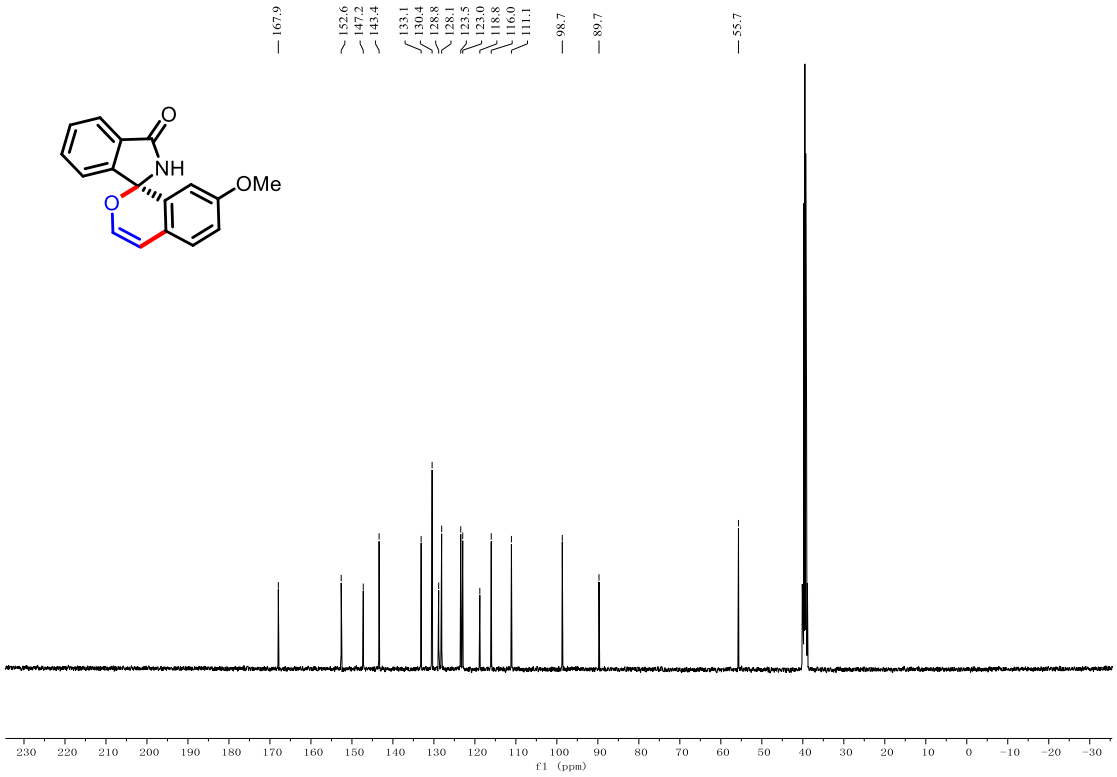
^{13}C NMR of **3p** (400 MHz, DMSO)



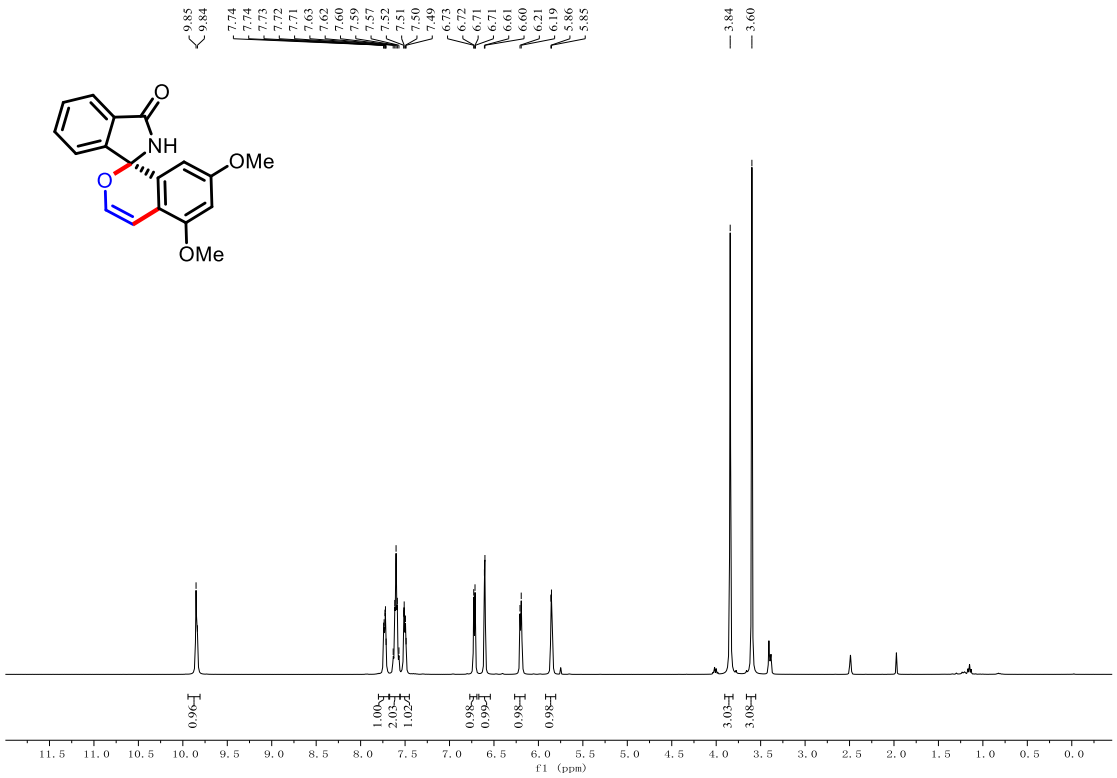
^{1}H NMR of **3q** (400 MHz, DMSO)



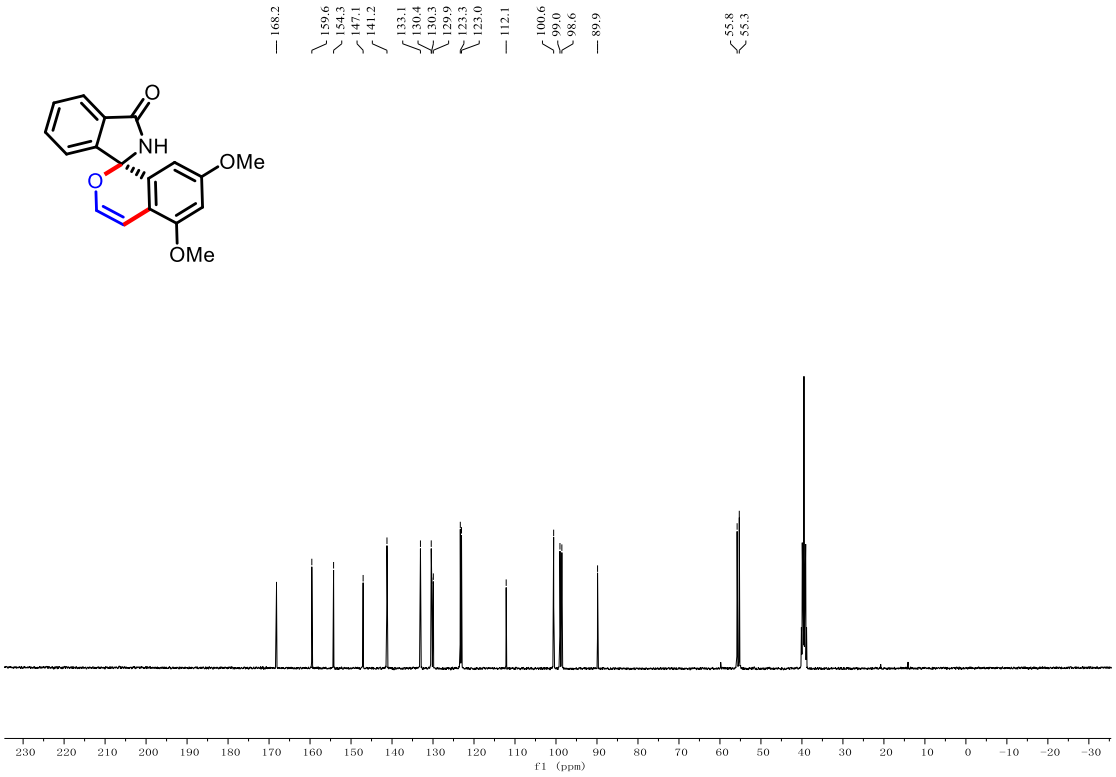
^{13}C NMR of **3q** (400 MHz, DMSO)



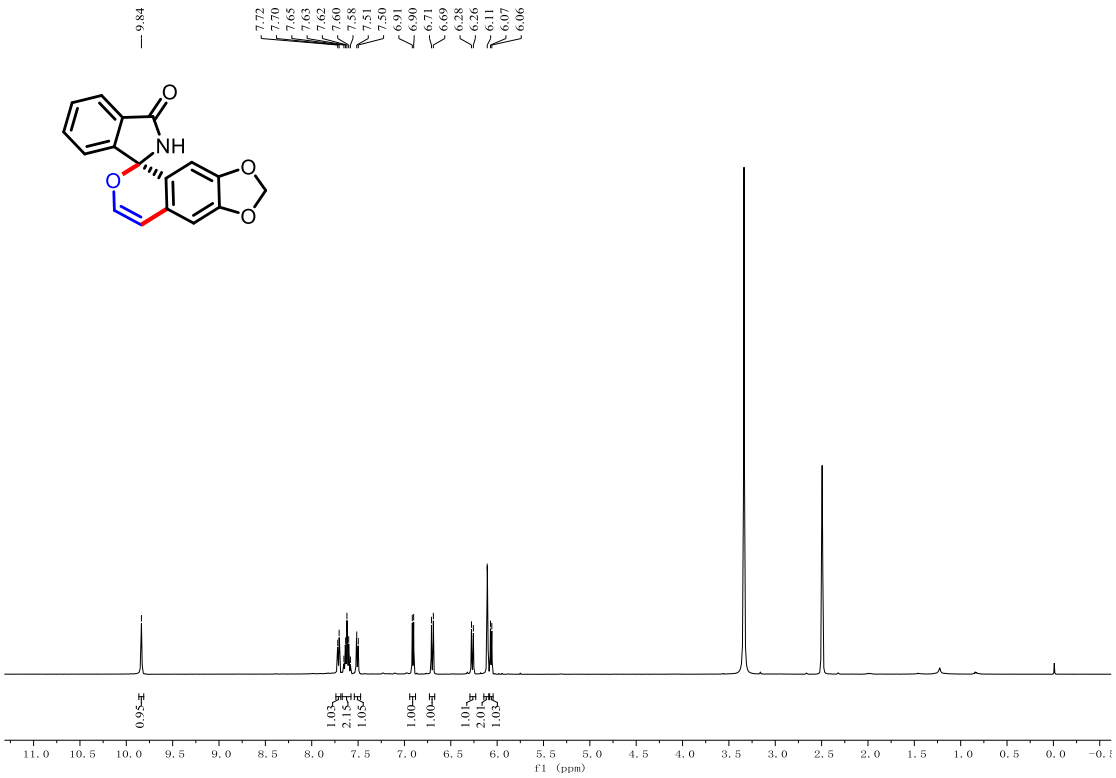
^{1}H NMR of **3r** (400 MHz, DMSO)



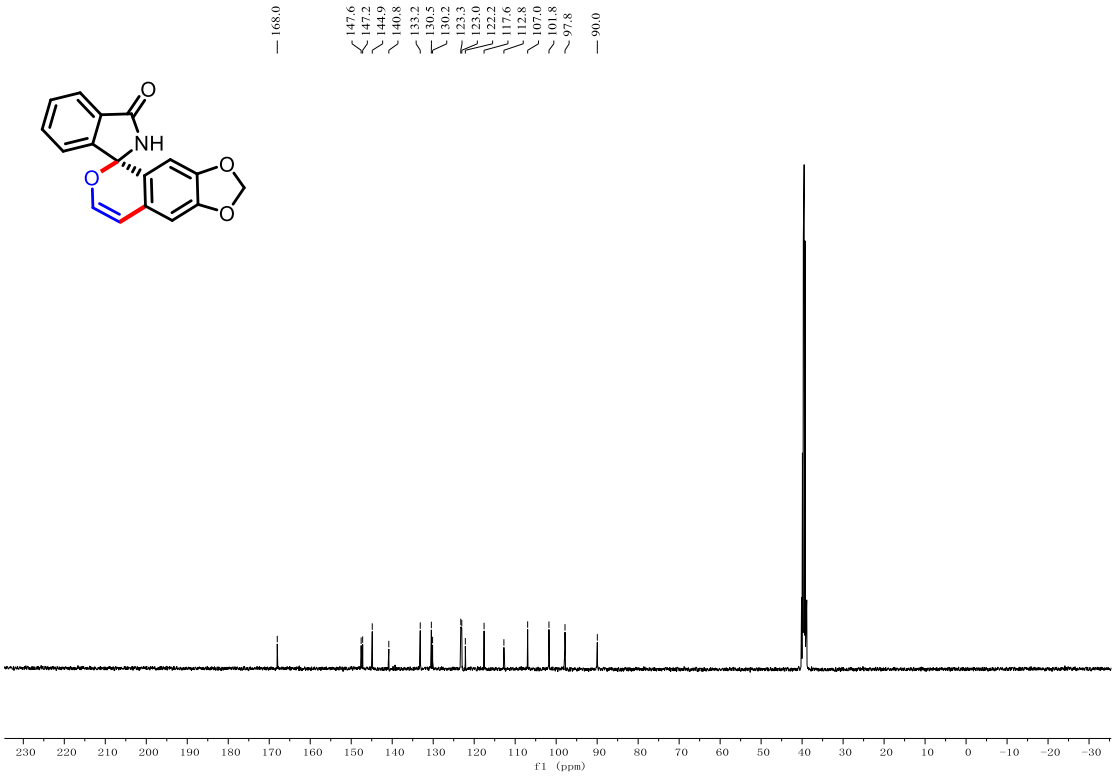
^{13}C NMR of **3r** (400 MHz, DMSO)



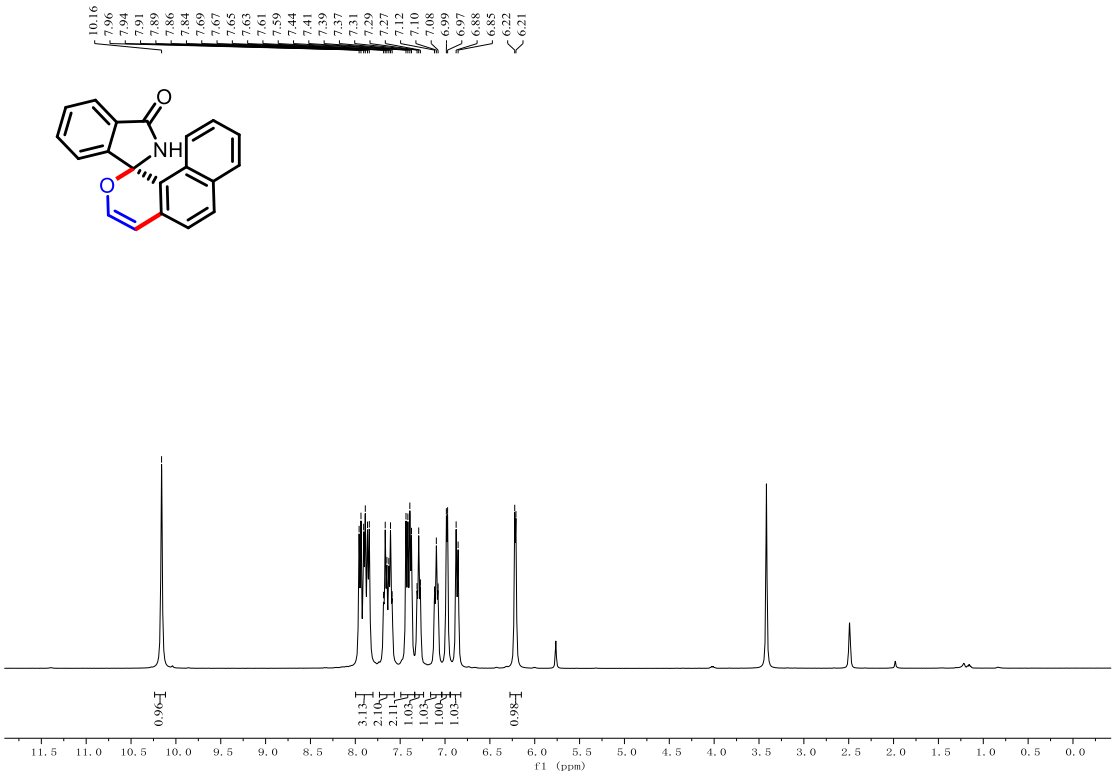
^{1}H NMR of **3s** (400 MHz, DMSO)



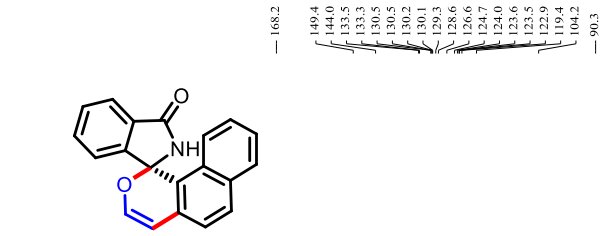
^{13}C NMR of **3s** (400 MHz, DMSO)

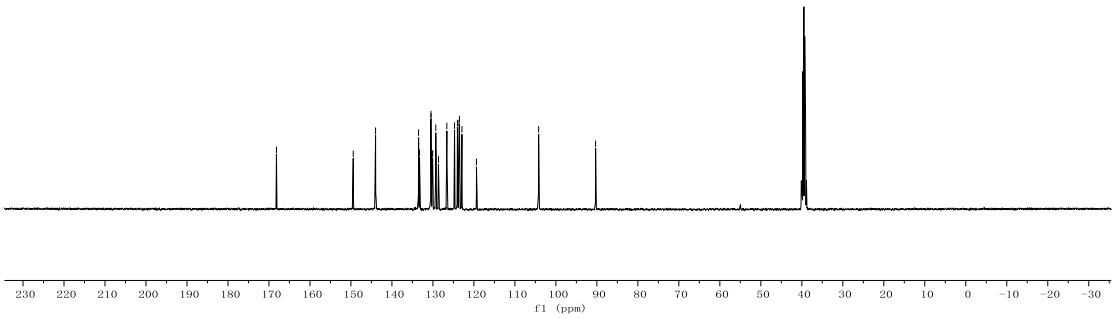


^{1}H NMR of **3t** (400 MHz, DMSO)

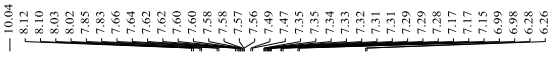


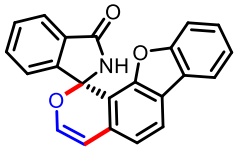
^{13}C NMR of **3t** (400 MHz, DMSO)

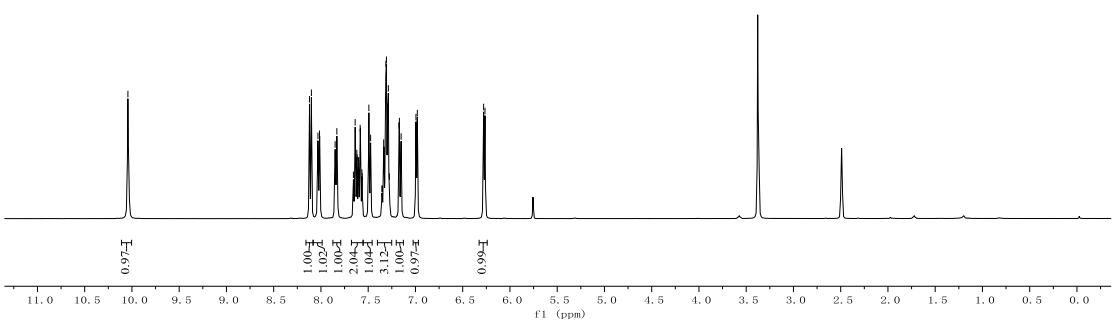




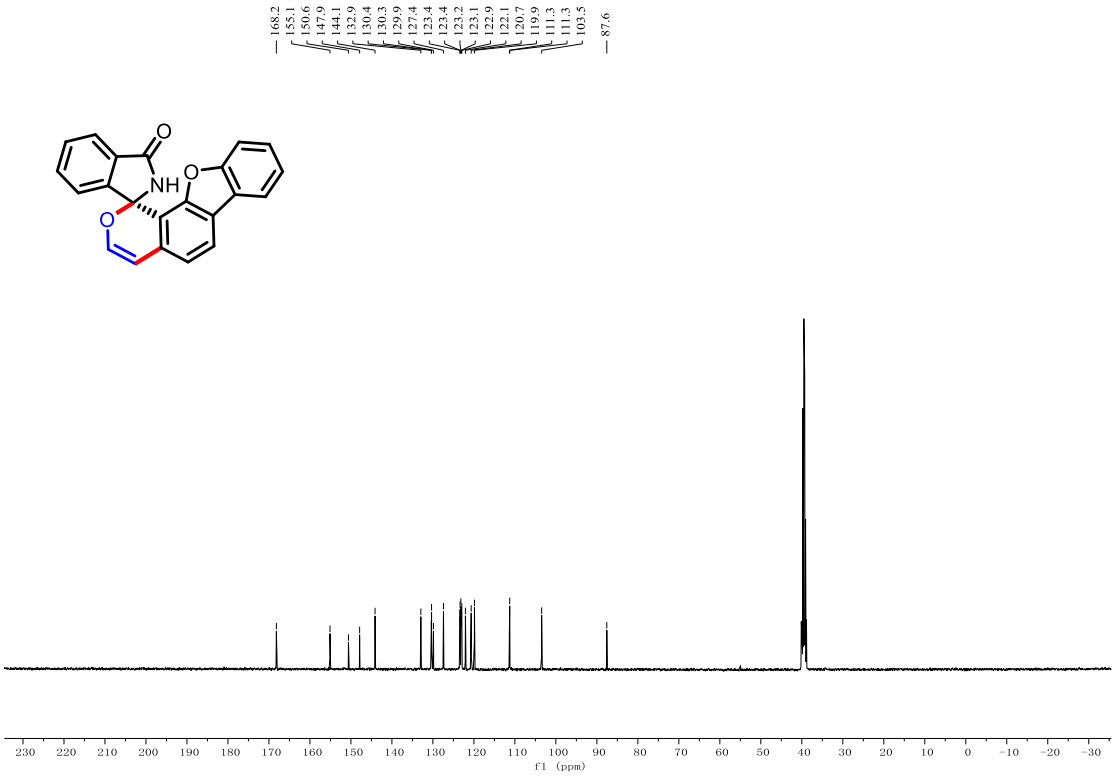
^{1}H NMR of **3u** (400 MHz, DMSO)



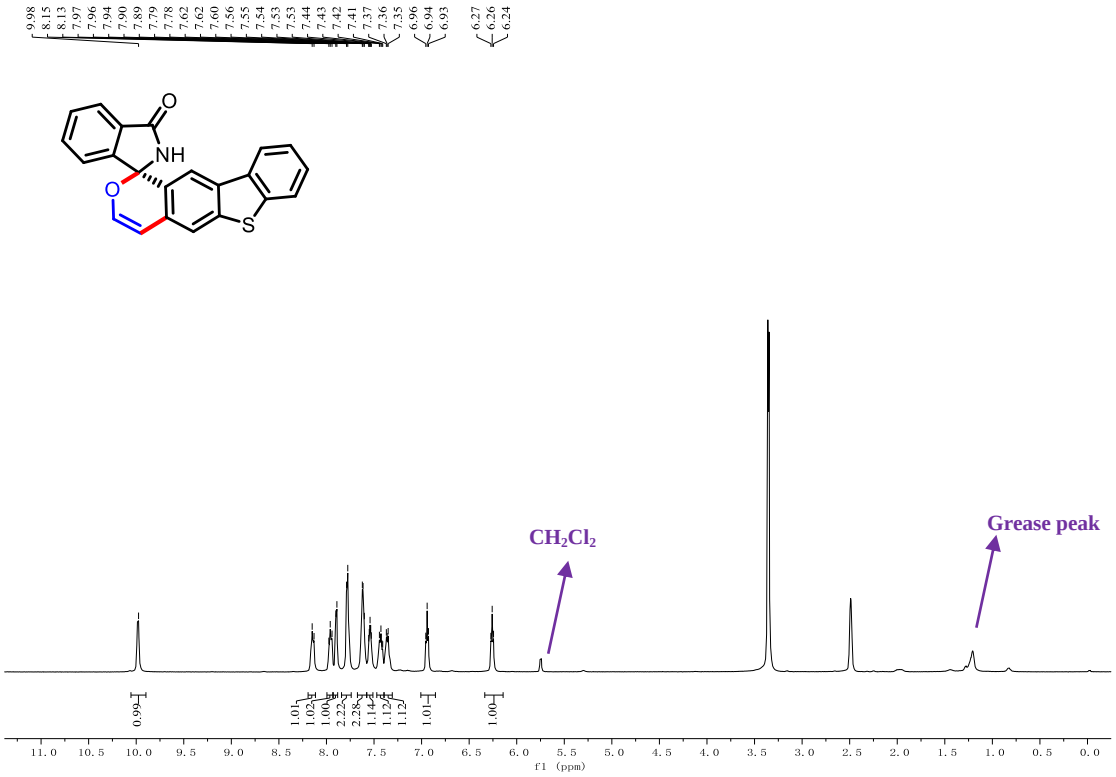




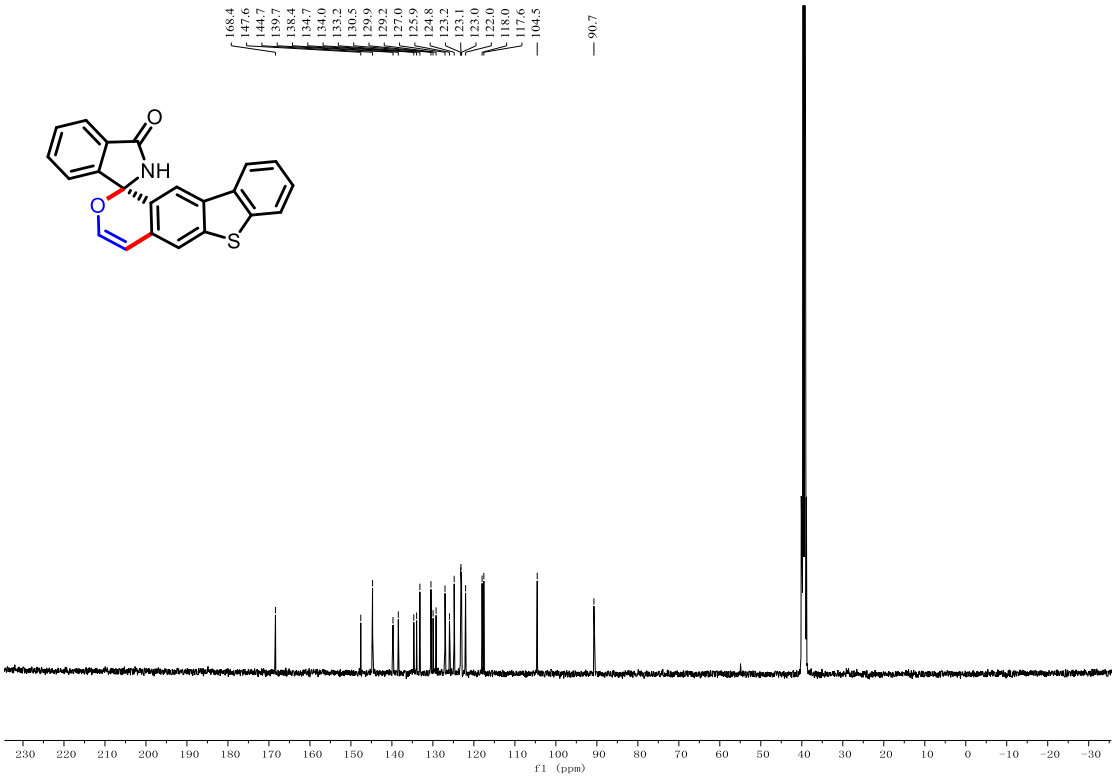
^{13}C NMR of **3u** (400 MHz, DMSO)



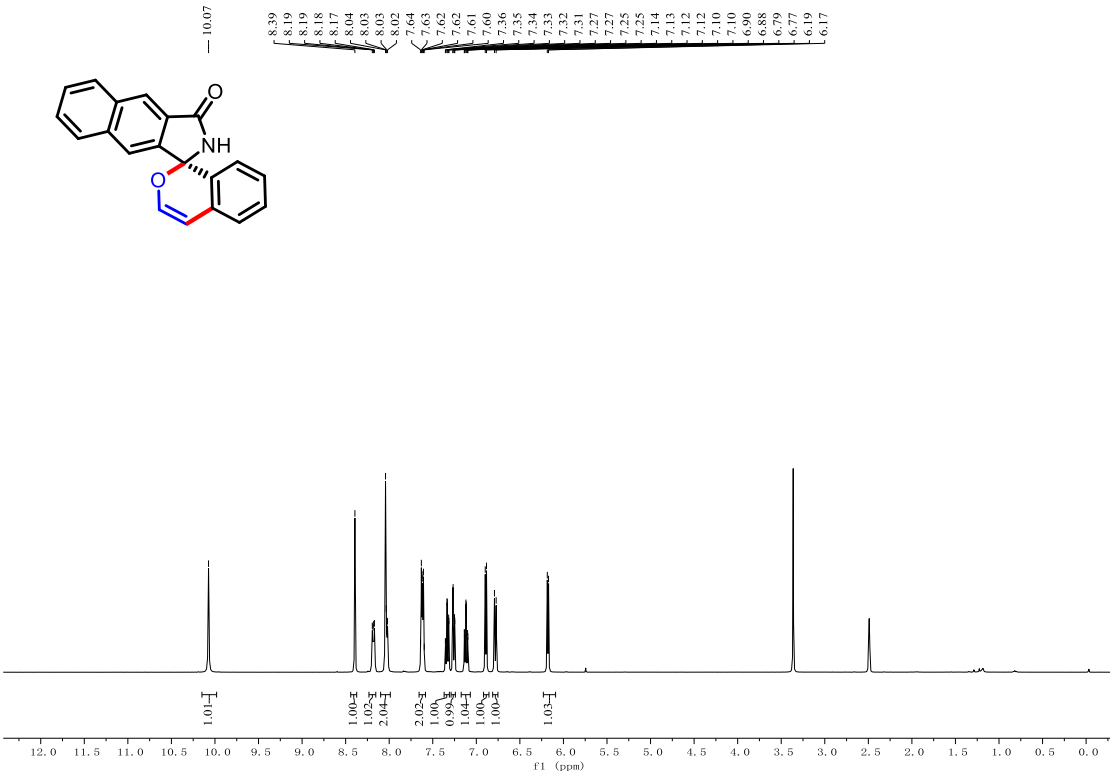
^{1}H NMR of **3v** (400 MHz, DMSO)



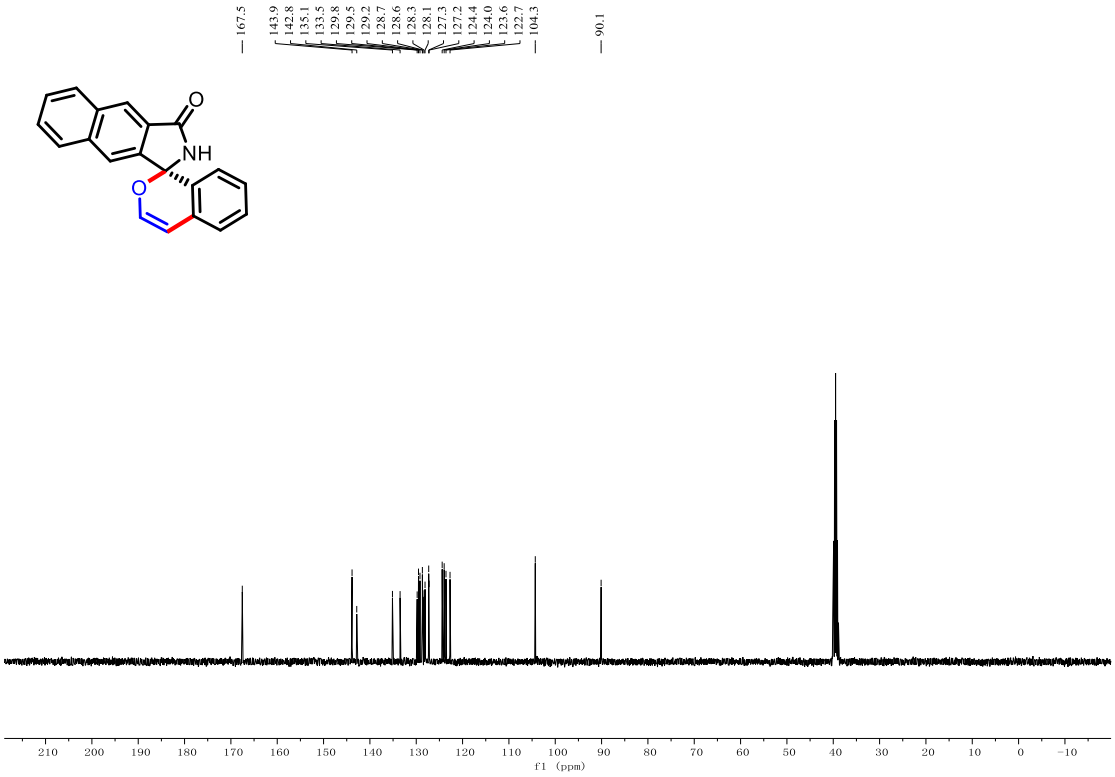
^{13}C NMR of **3v** (400 MHz, DMSO)



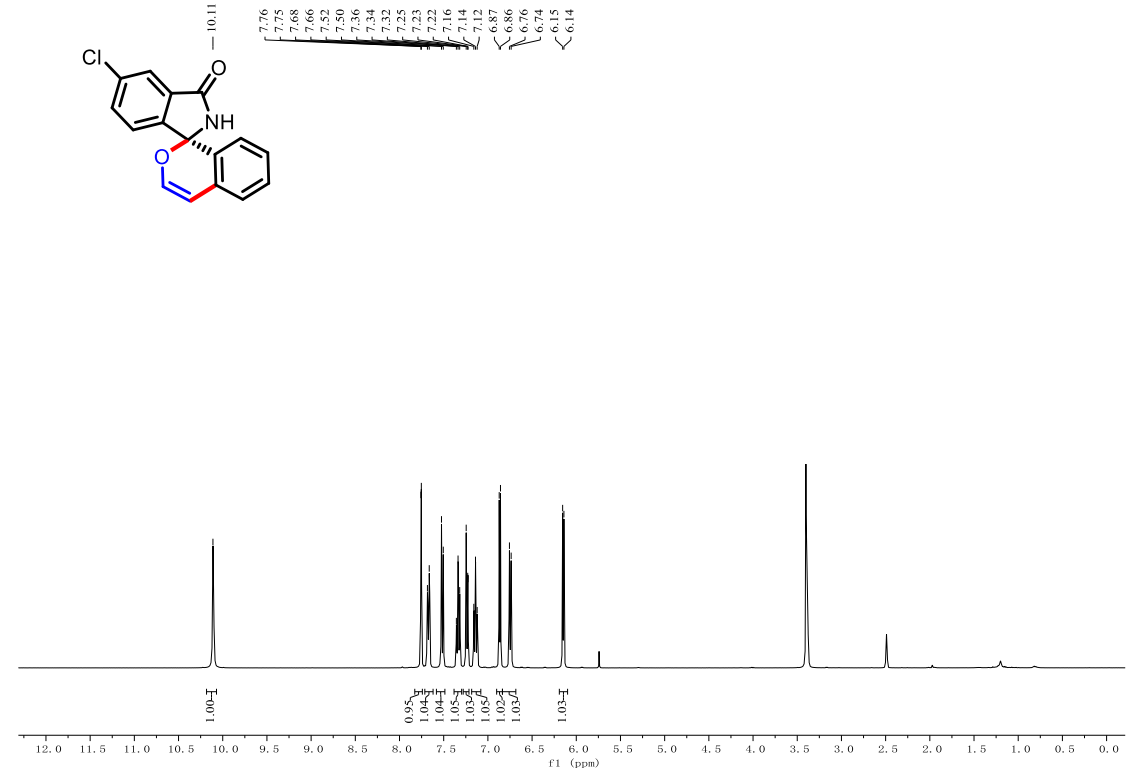
^{1}H NMR of **3a′** (400 MHz, DMSO)



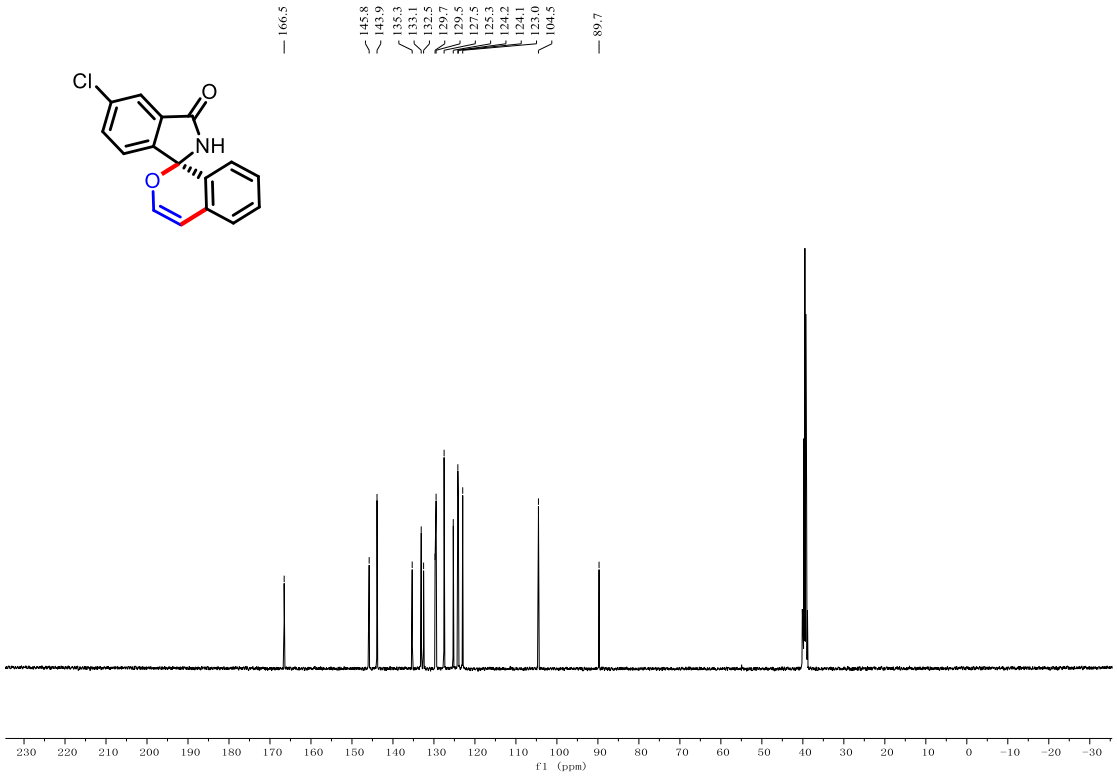
^{13}C NMR of **3a′** (400 MHz, DMSO)



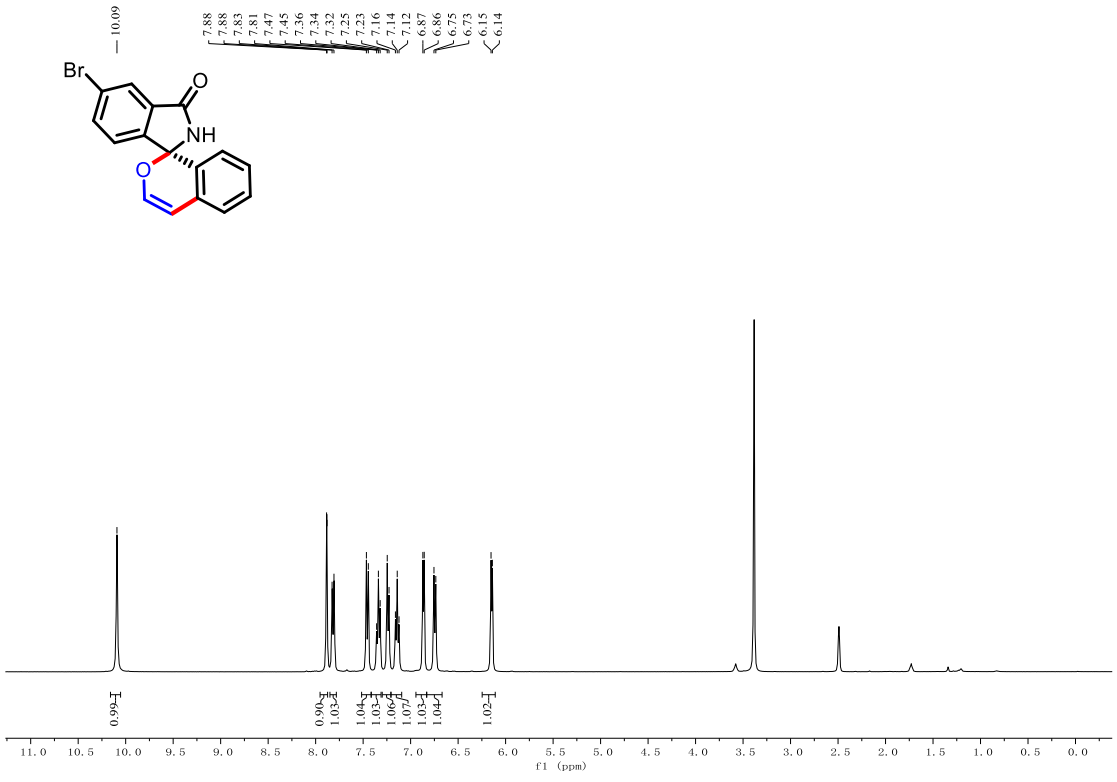
^{1}H NMR of **3b′** (400 MHz, DMSO)



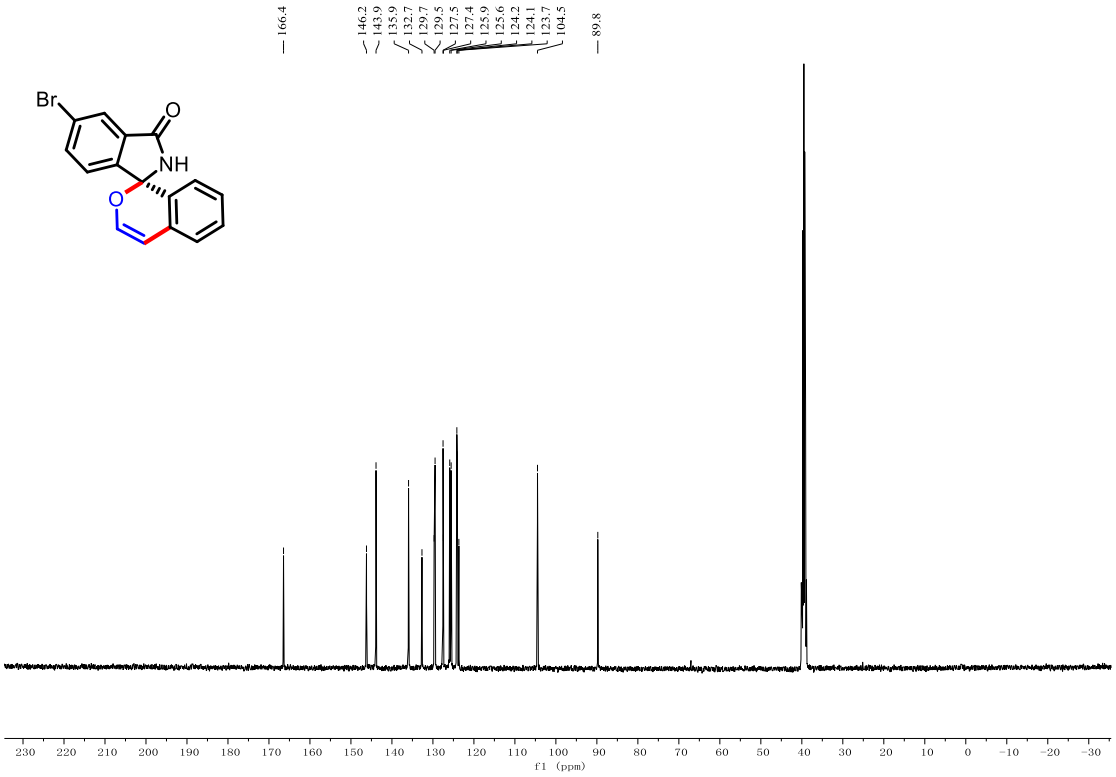
^{13}C NMR of **3b′** (400 MHz, DMSO)



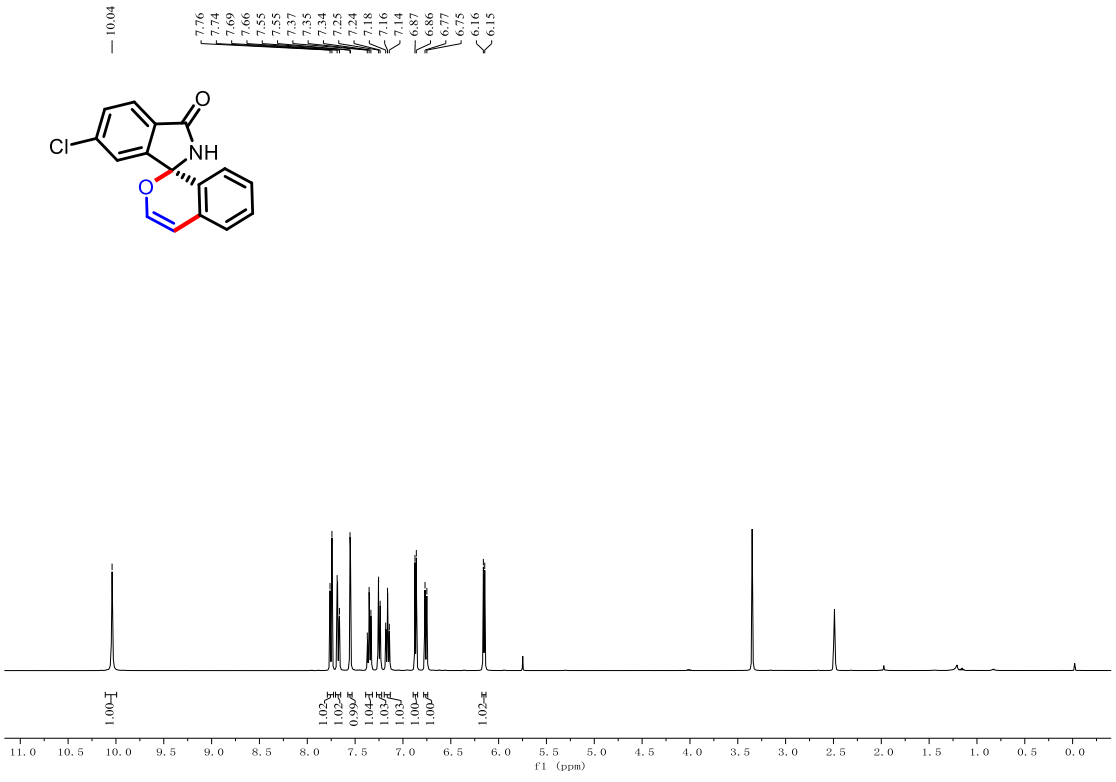
^{1}H NMR of **3c′** (400 MHz, DMSO)



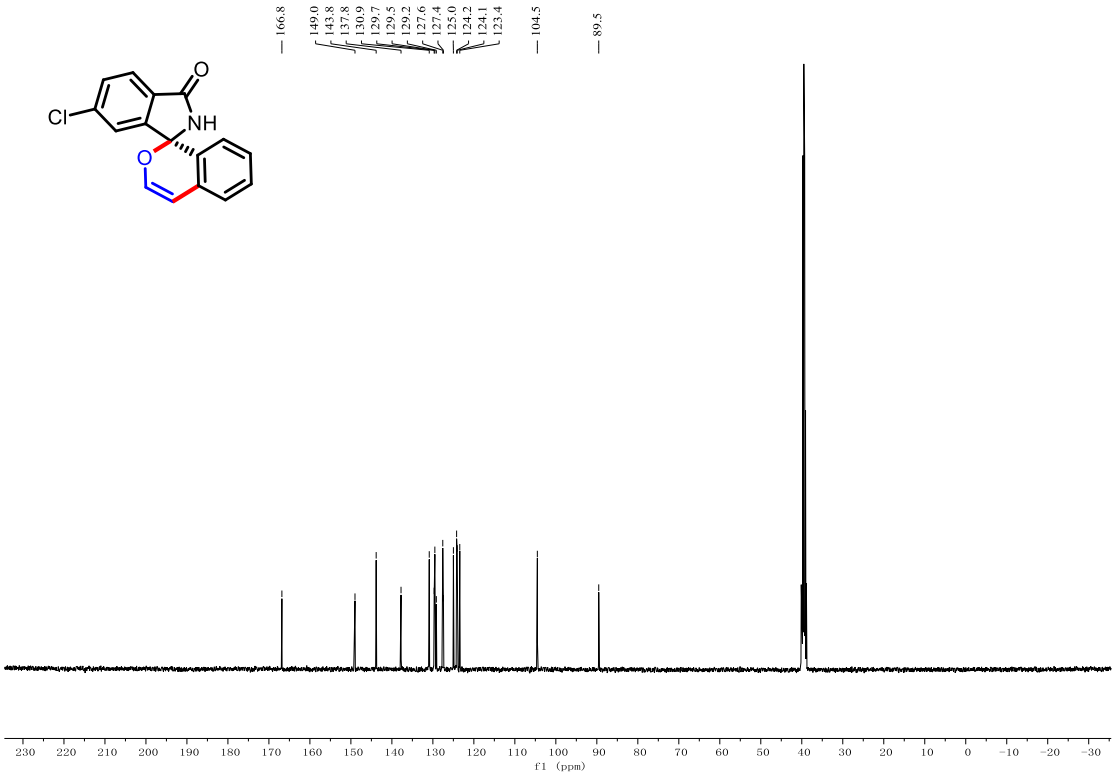
^{13}C NMR of **3c′** (400 MHz, DMSO)



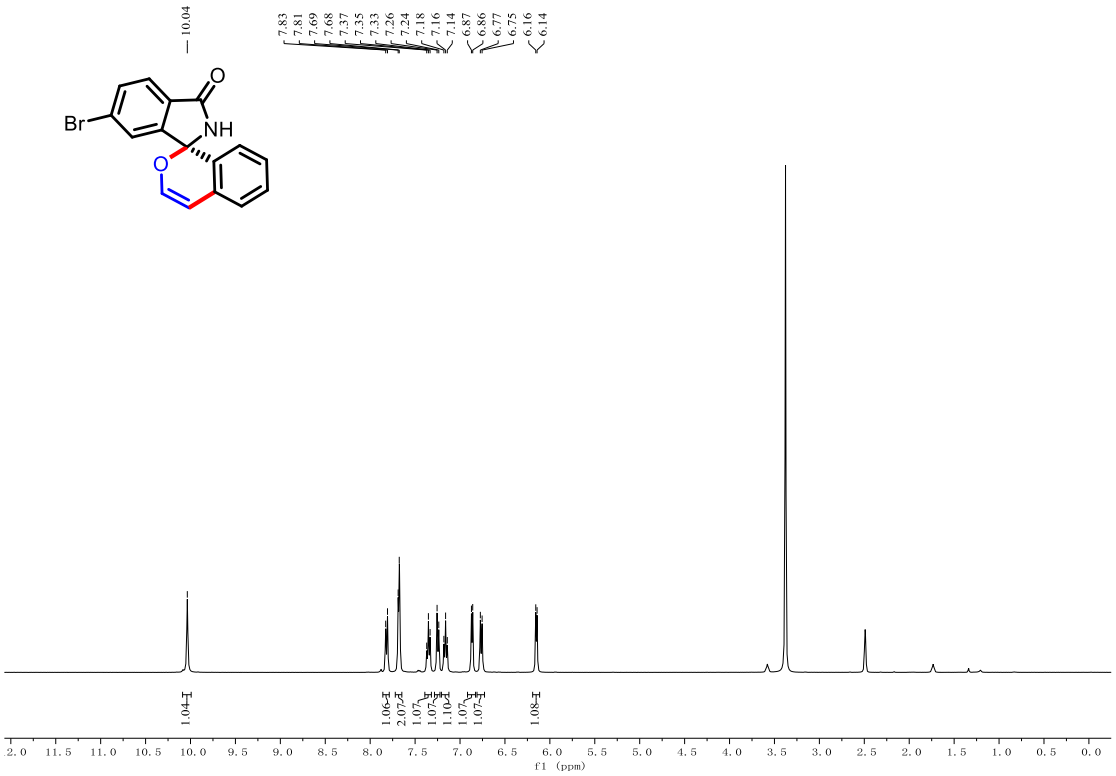
^{1}H NMR of **3d′** (400 MHz, DMSO)



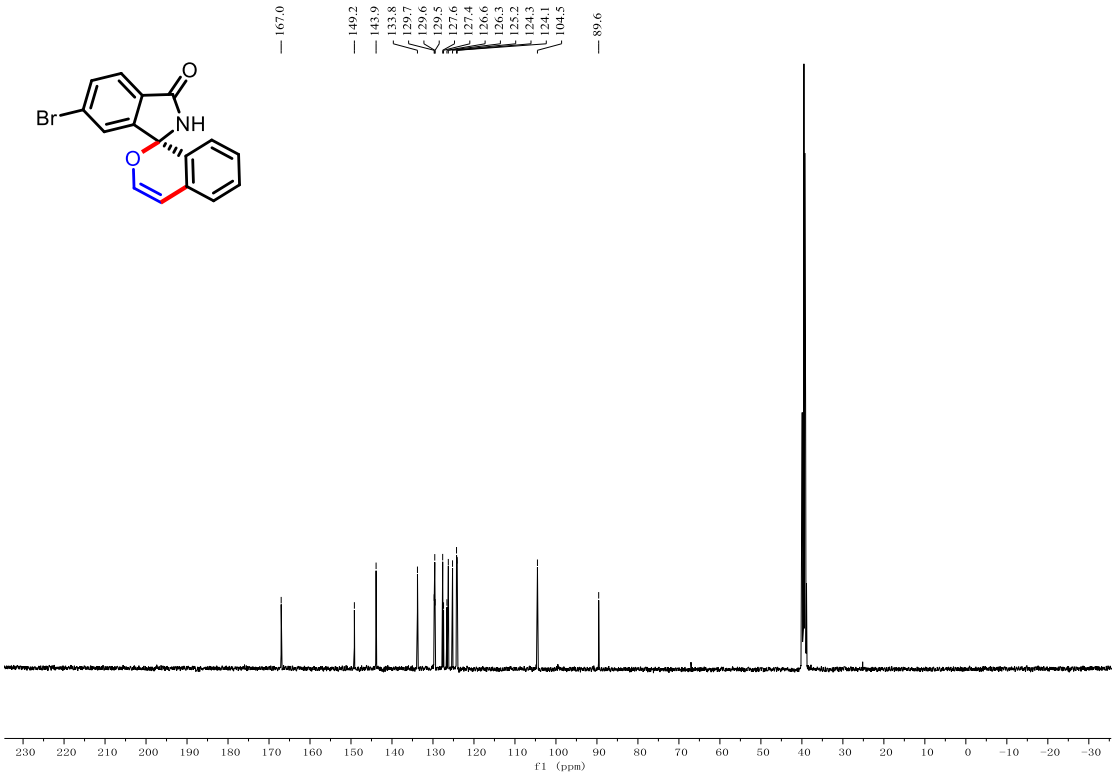
^{13}C NMR of **3d′** (400 MHz, DMSO)



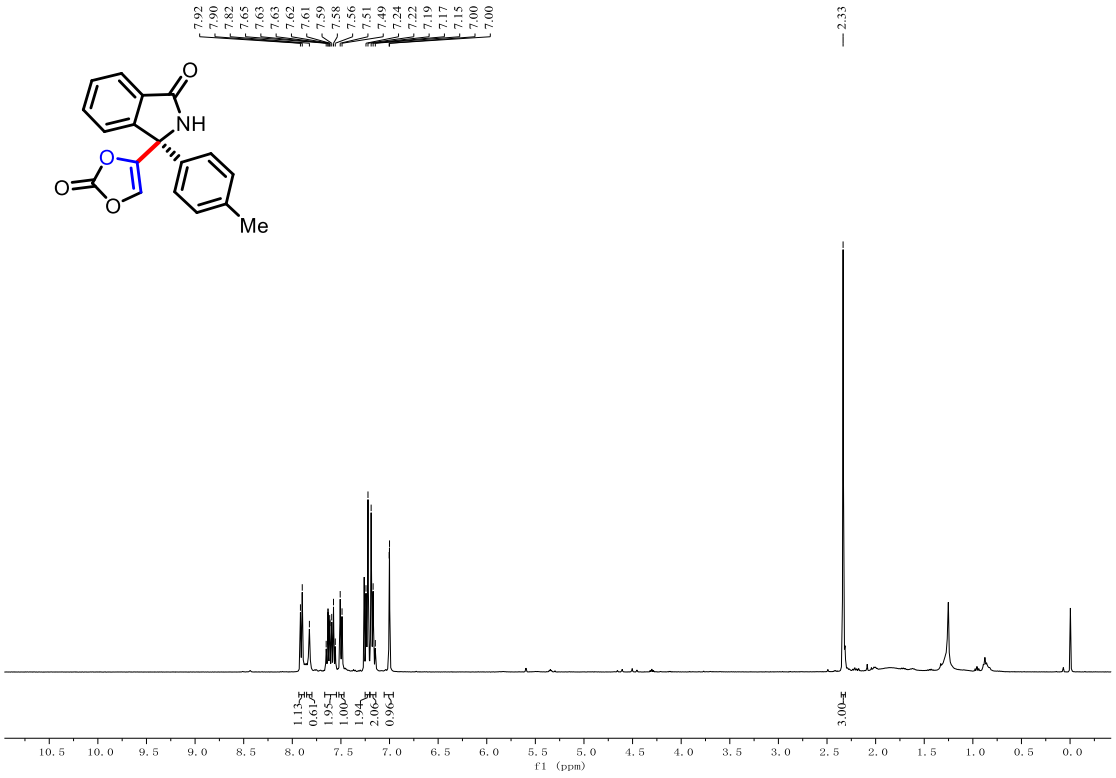
^{1}H NMR of **3e′** (400 MHz, DMSO)



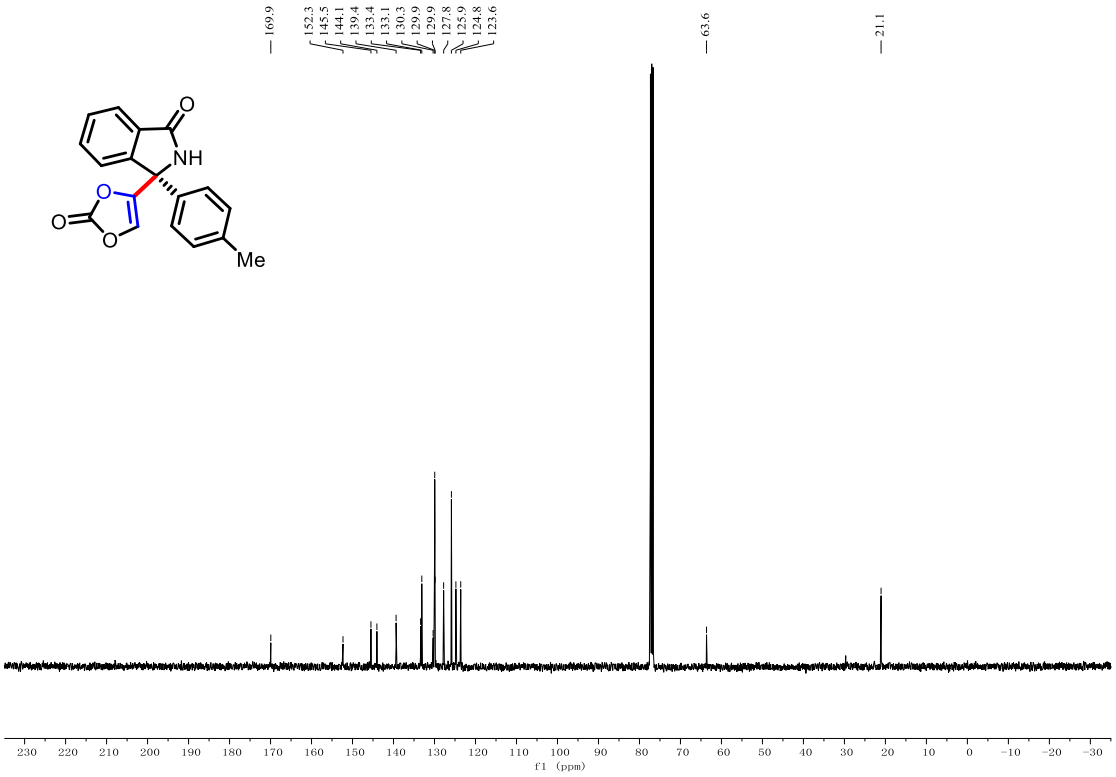
^{13}C NMR of **3e′** (400 MHz, DMSO)



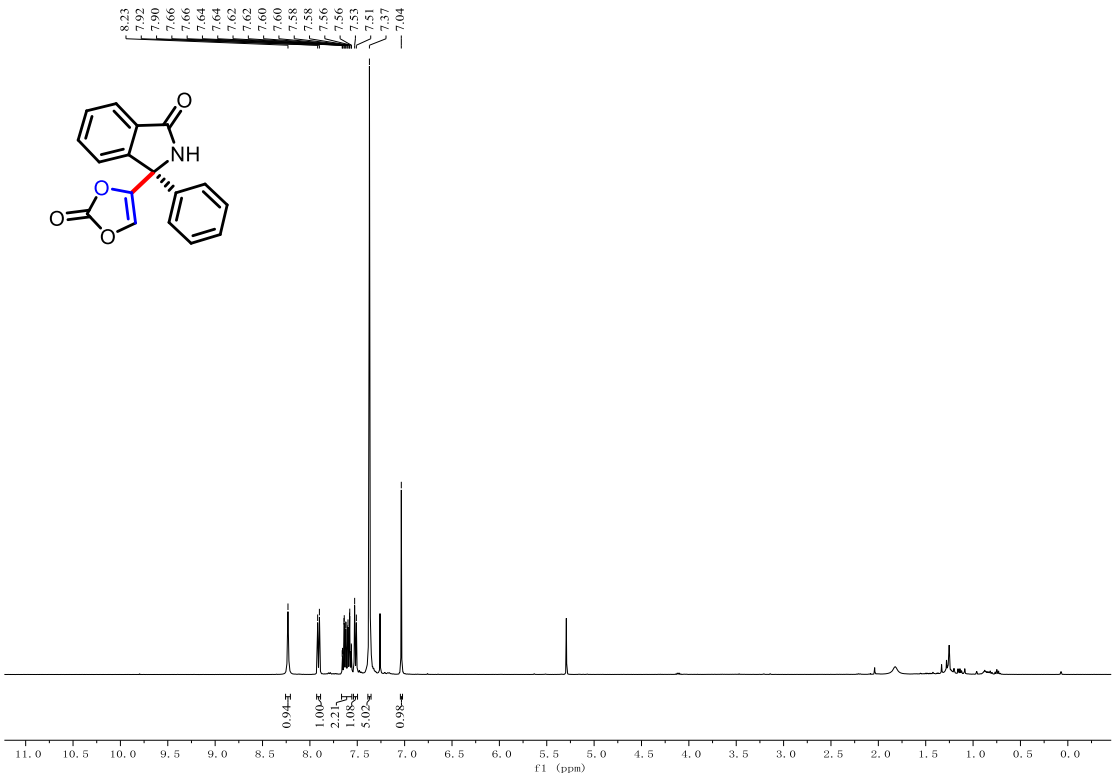
^{1}H NMR of **5a** (400 MHz, $CDCl_3$)



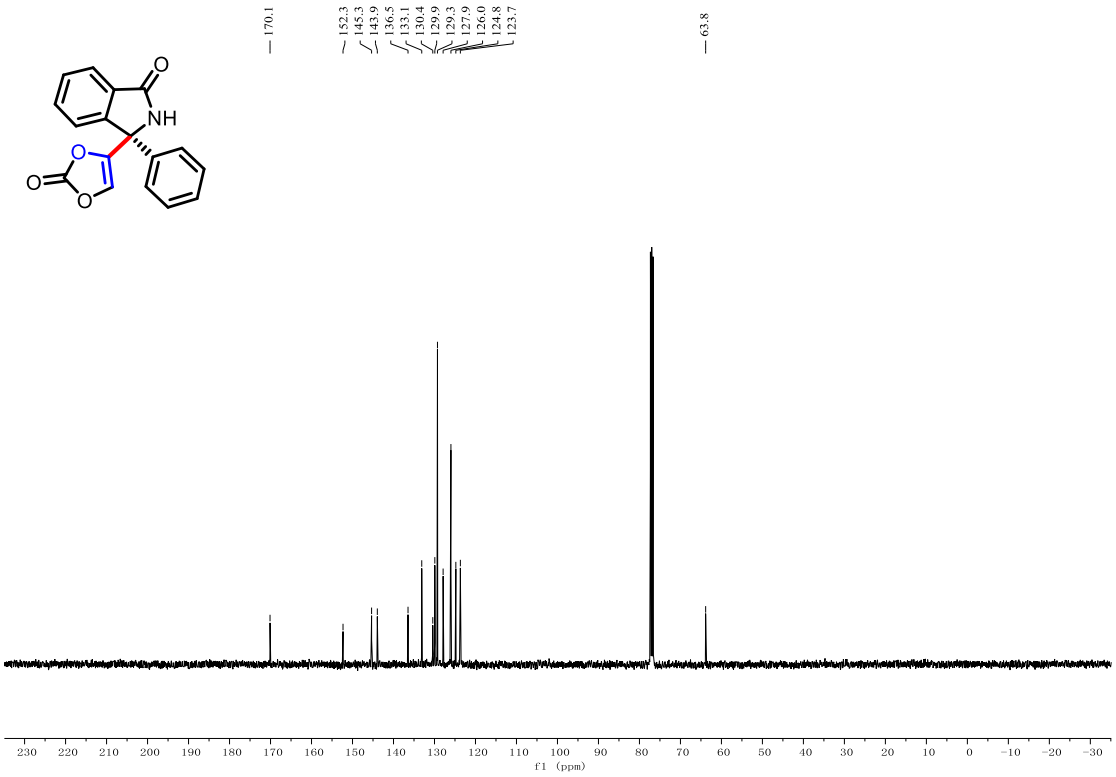
^{13}C NMR of **5a** (400 MHz, $CDCl_3$)



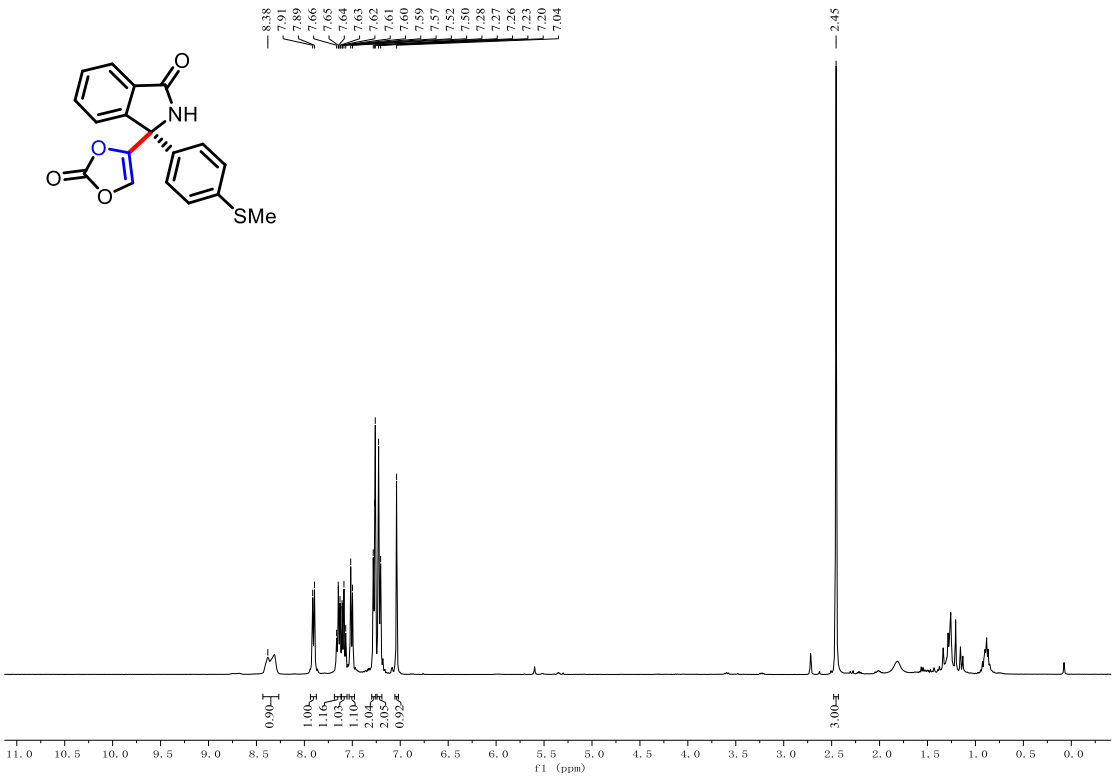
^{1}H NMR of **5b** (400 MHz, $CDCl_3$)



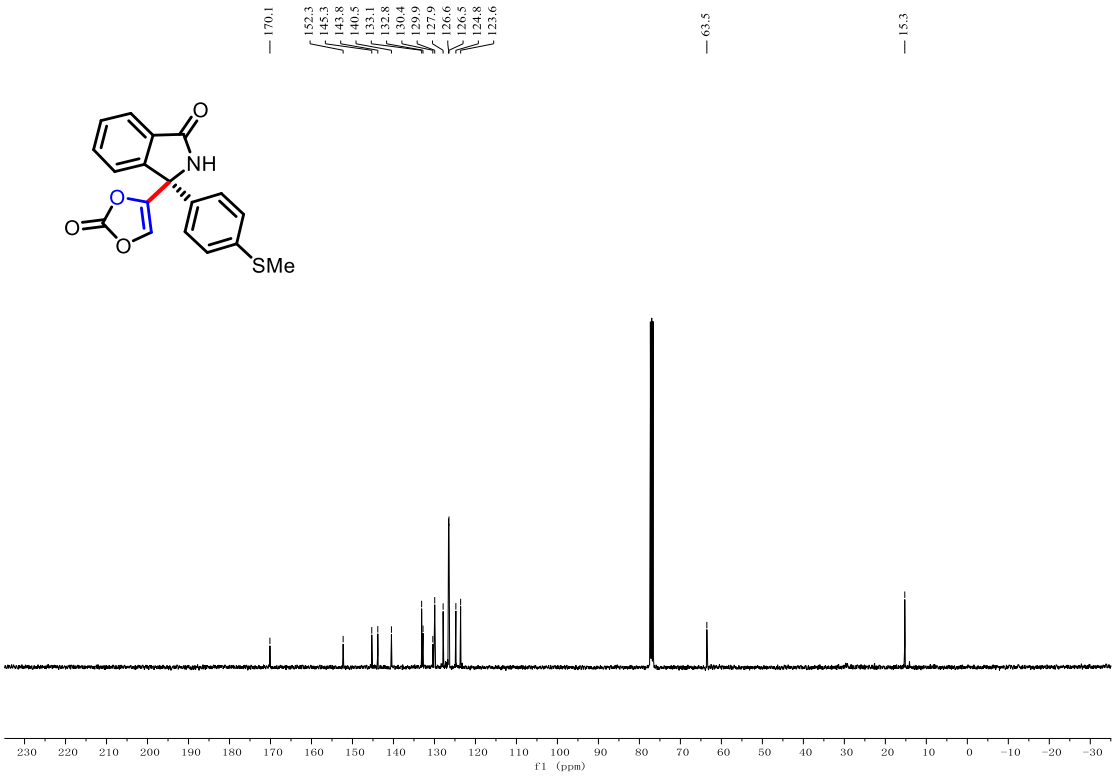
^{13}C NMR of **5b** (400 MHz, $CDCl_3$)



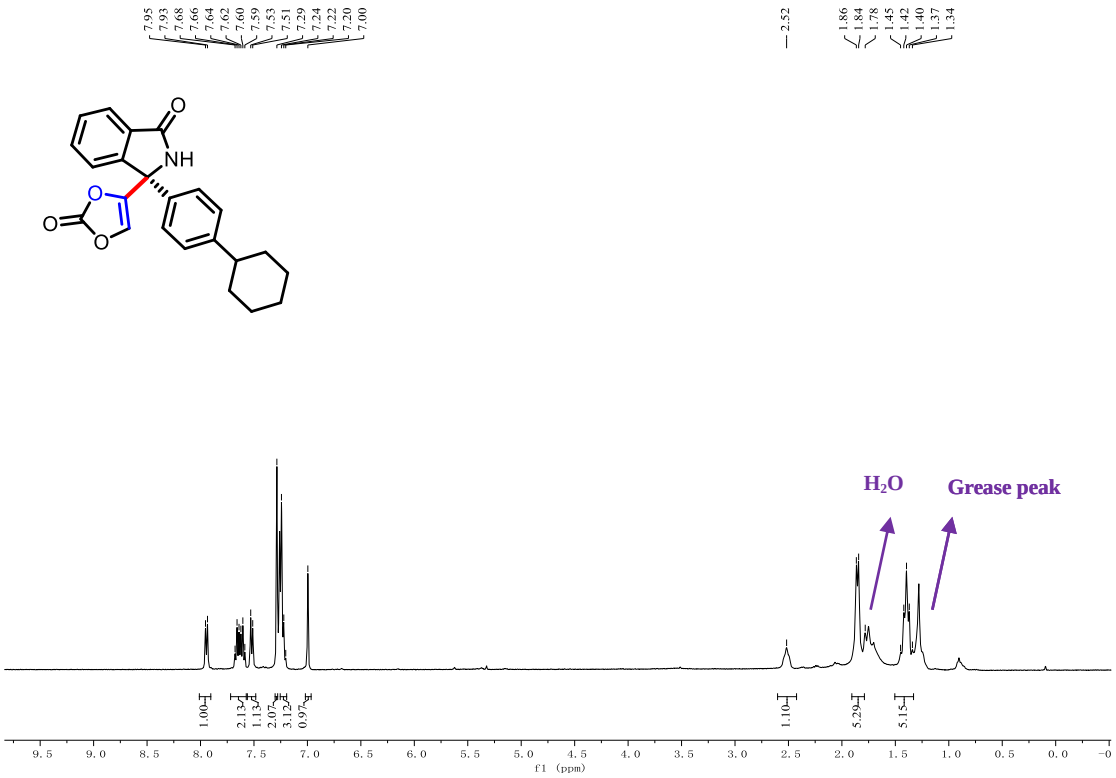
^{1}H NMR of **5c** (400 MHz, $CDCl_3$)



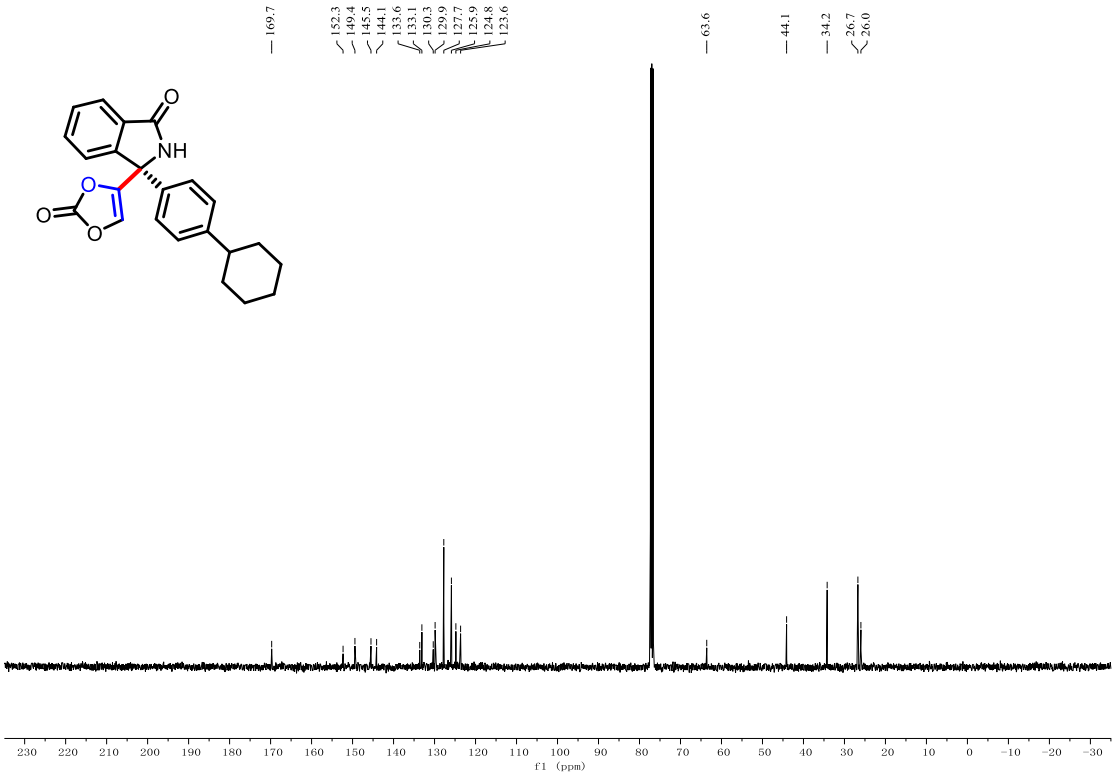
^{13}C NMR of **5c** (400 MHz, $CDCl_3$)



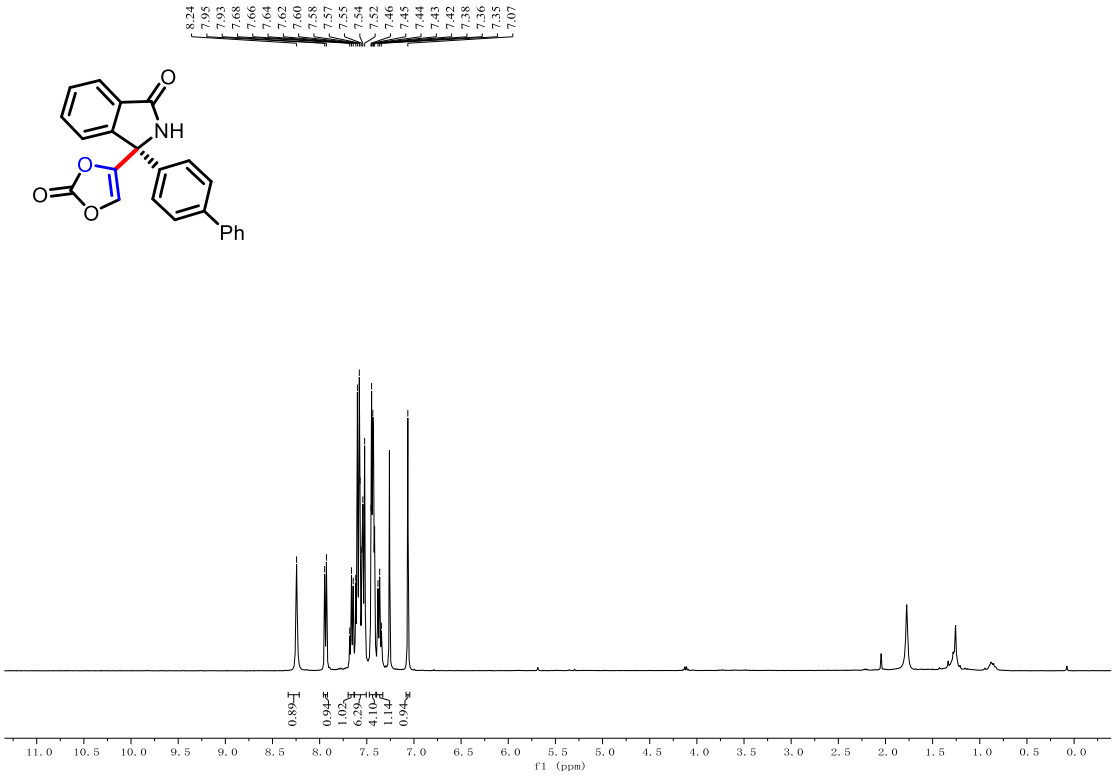
^{1}H NMR of **5d** (400 MHz, $CDCl_3$)



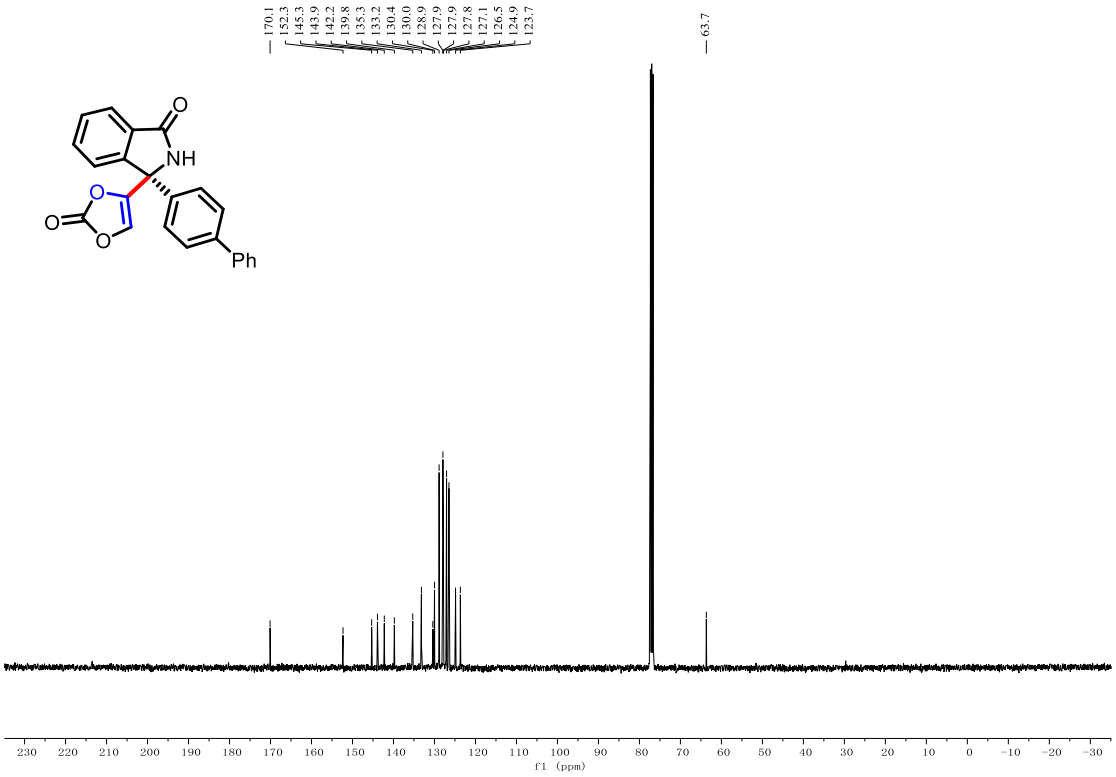
^{13}C NMR of **5d** (400 MHz, $CDCl_3$)



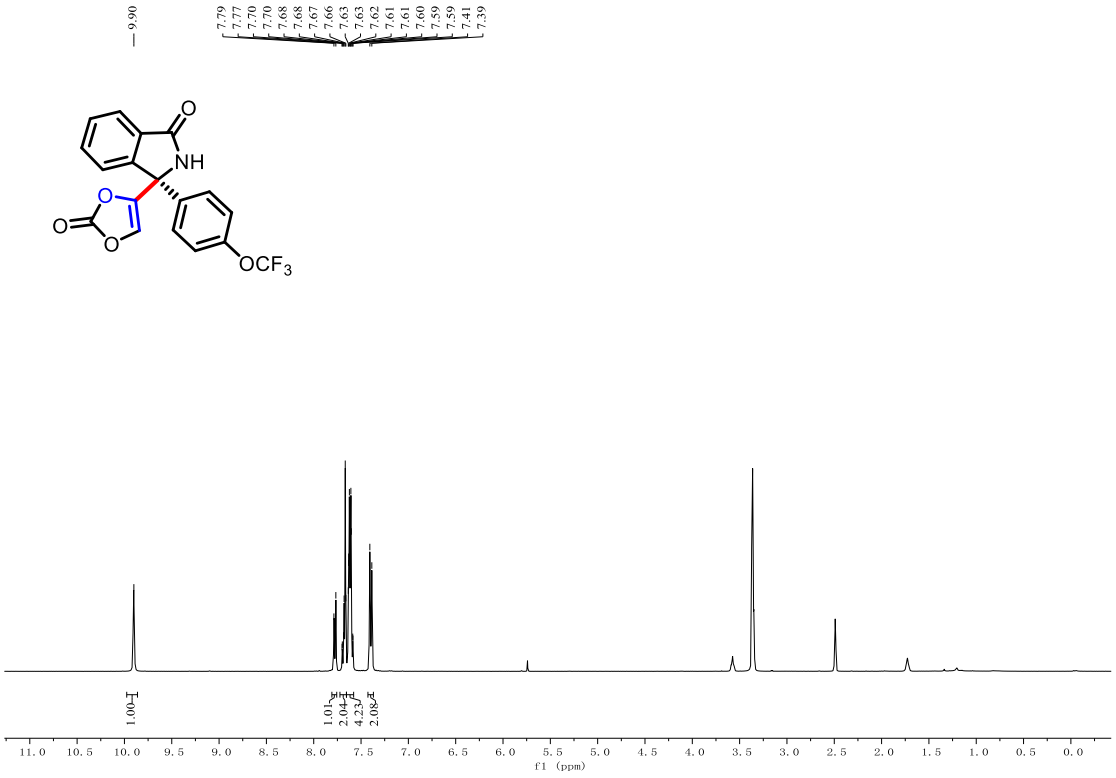
^{1}H NMR of **5e** (400 MHz, $CDCl_3$)



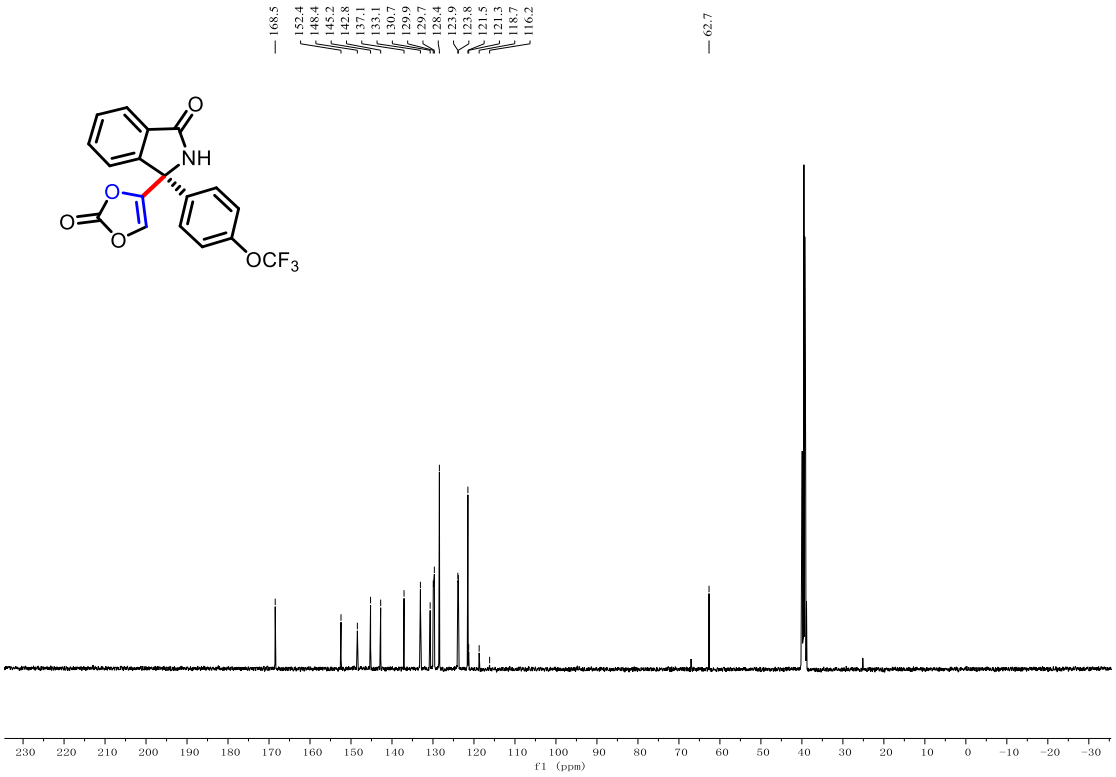
^{13}C NMR of **5e** (400 MHz, $CDCl_3$)



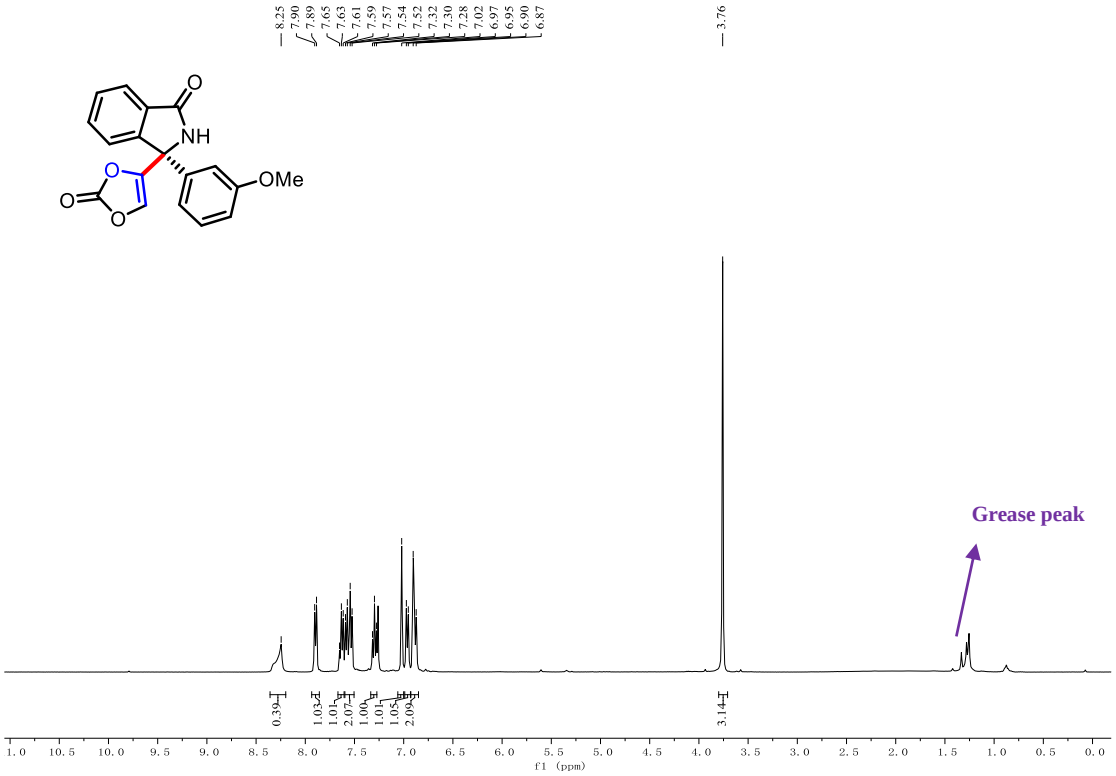
^{1}H NMR of **5f** (400 MHz, $CDCl_3$)



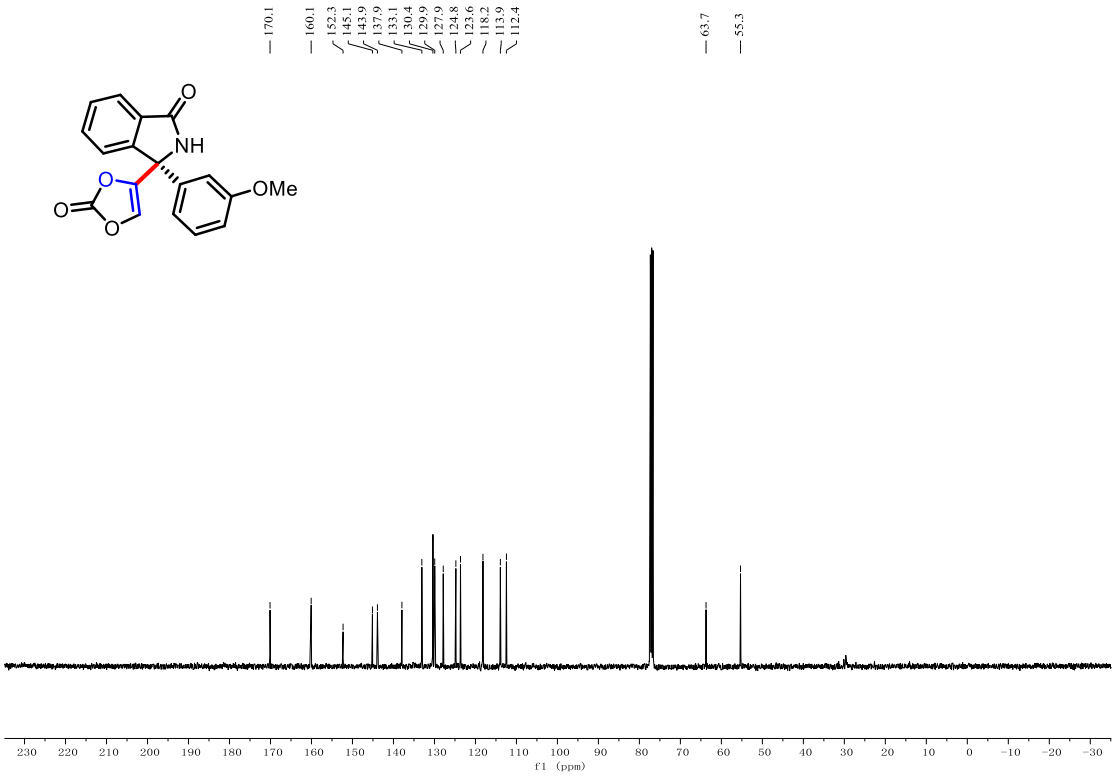
^{13}C NMR of **5f** (400 MHz, $CDCl_3$)



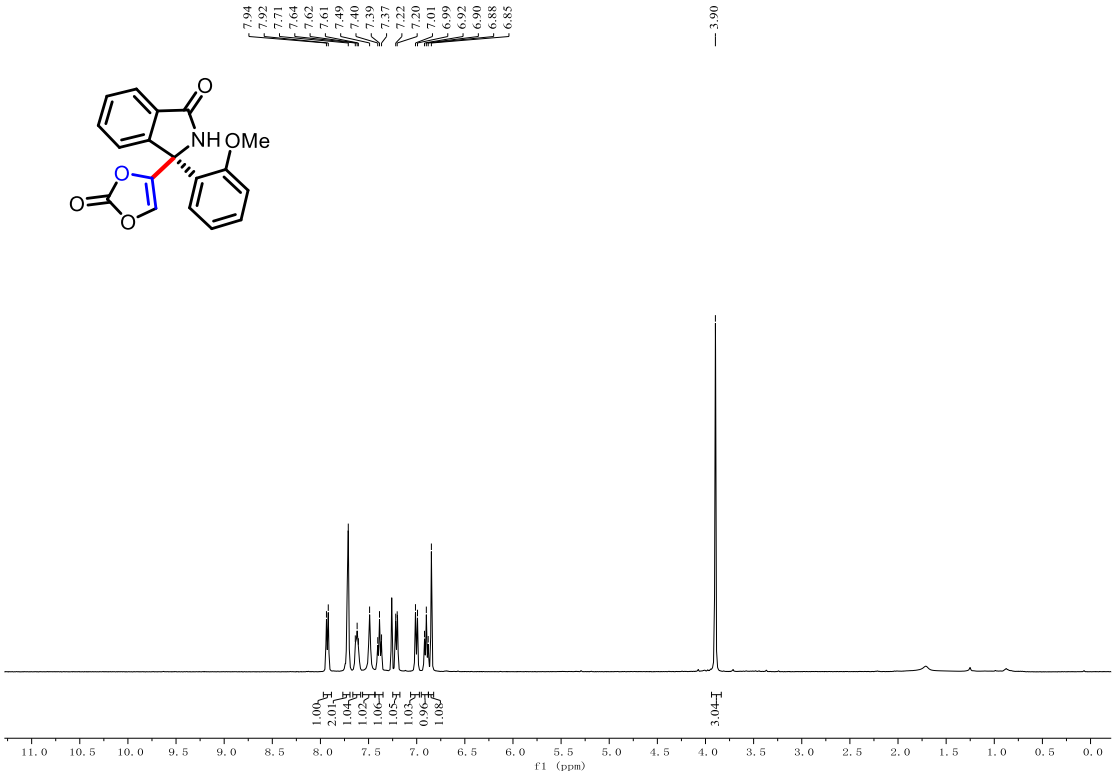
^{1}H NMR of **5g** (400 MHz, $CDCl_3$)



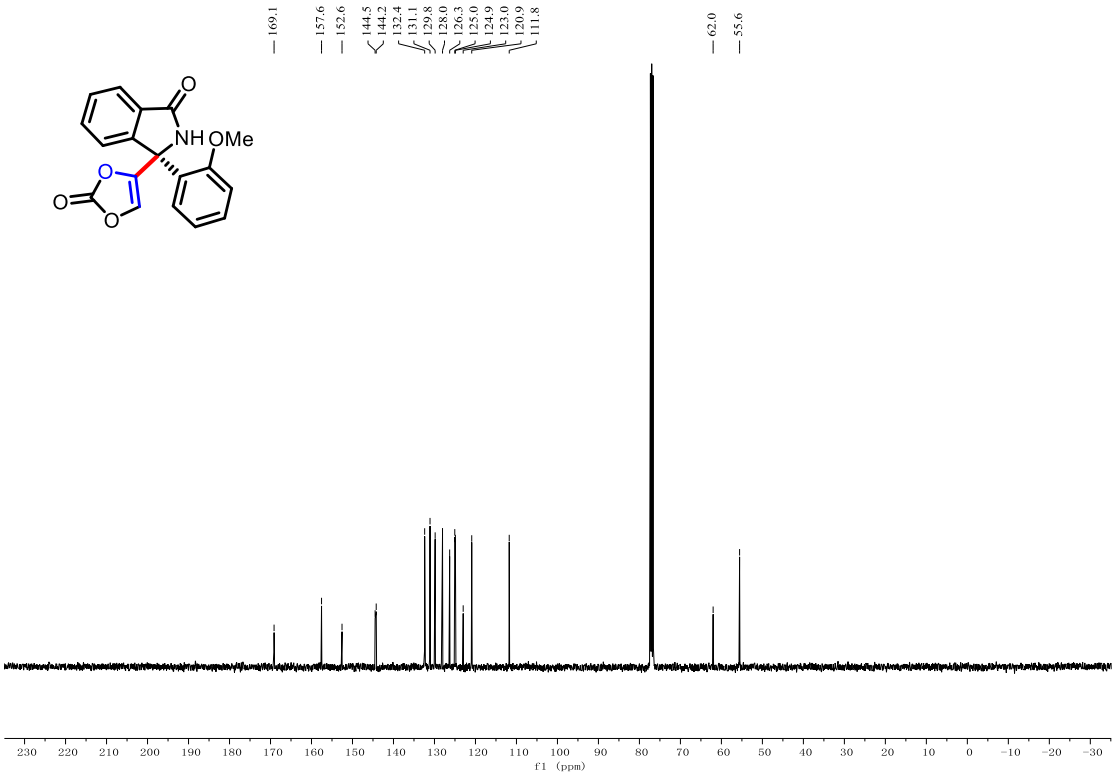
^{13}C NMR of **5g** (400 MHz, $CDCl_3$)



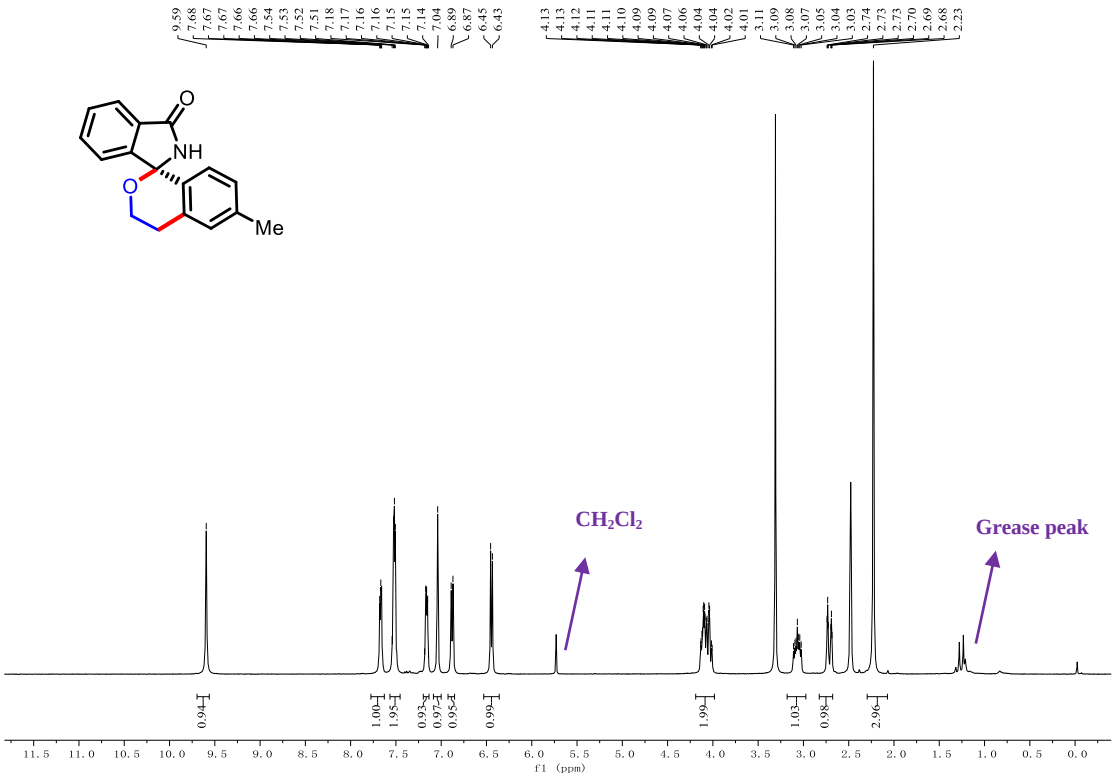
^{1}H NMR of **5h** (400 MHz, $CDCl_3$)



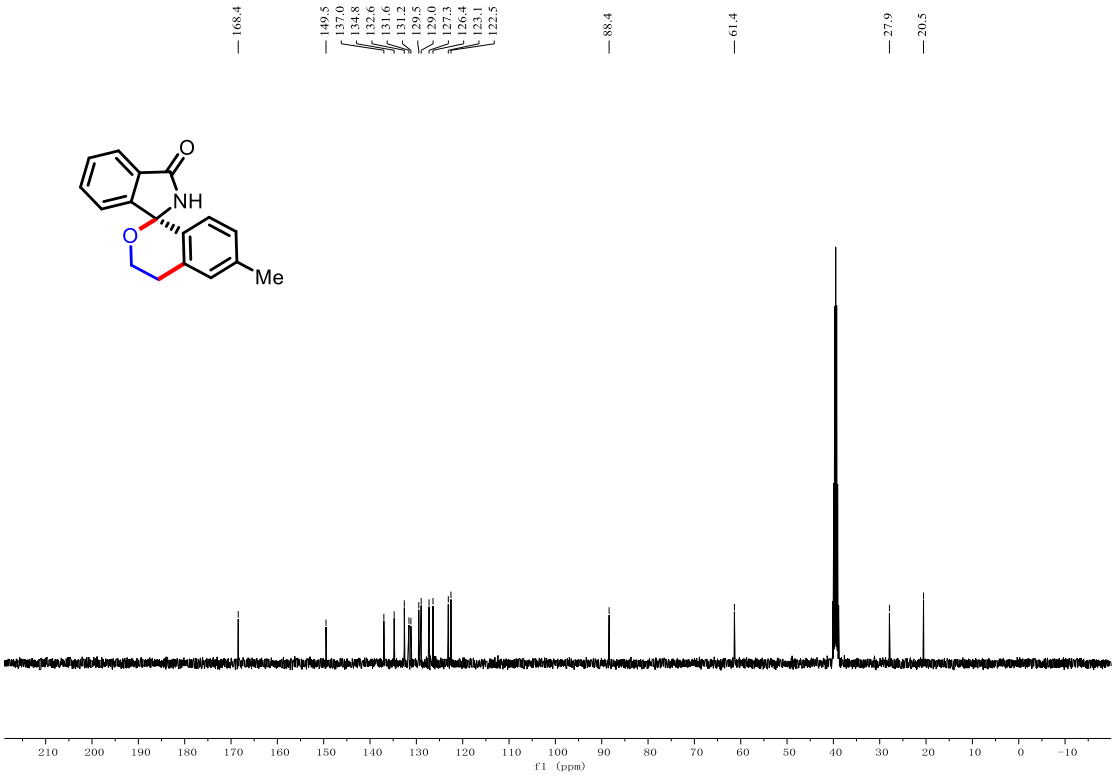
^{13}C NMR of **5h** (400 MHz, $CDCl_3$)



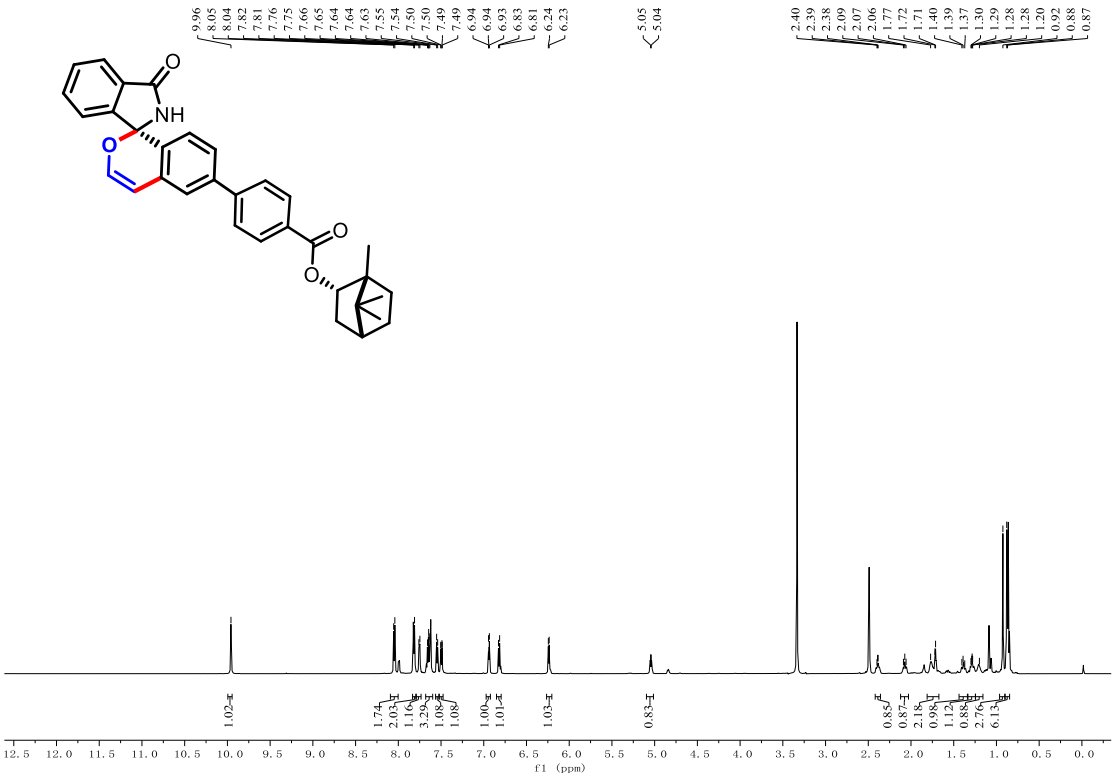
^{1}H NMR of **6** (400 MHz, DMSO)



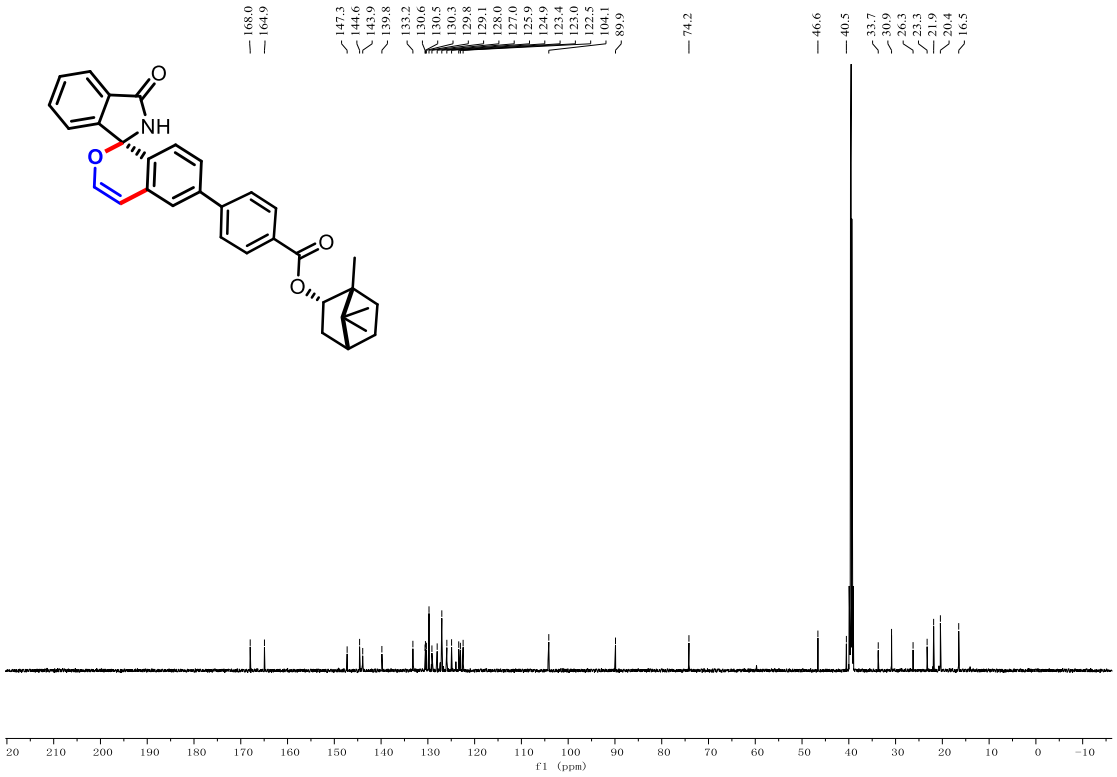
^{13}C NMR of **6** (400 MHz,DMSO)



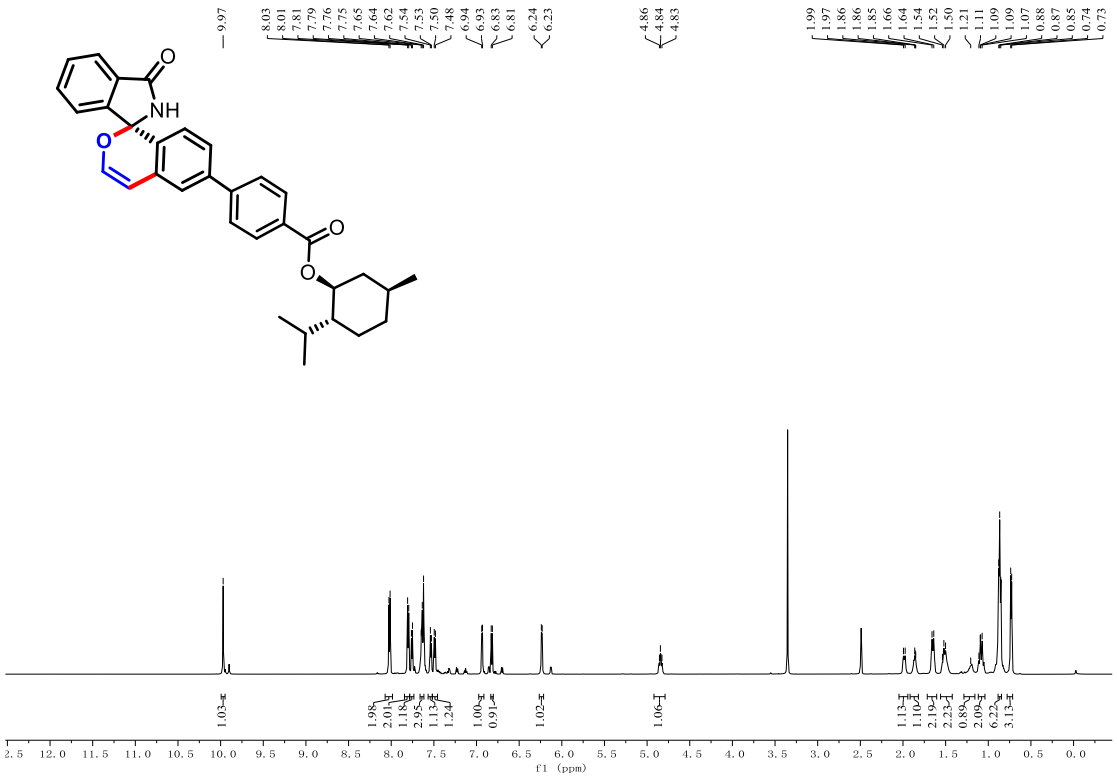
^{1}H NMR of **3a"** (600 MHz, DMSO)



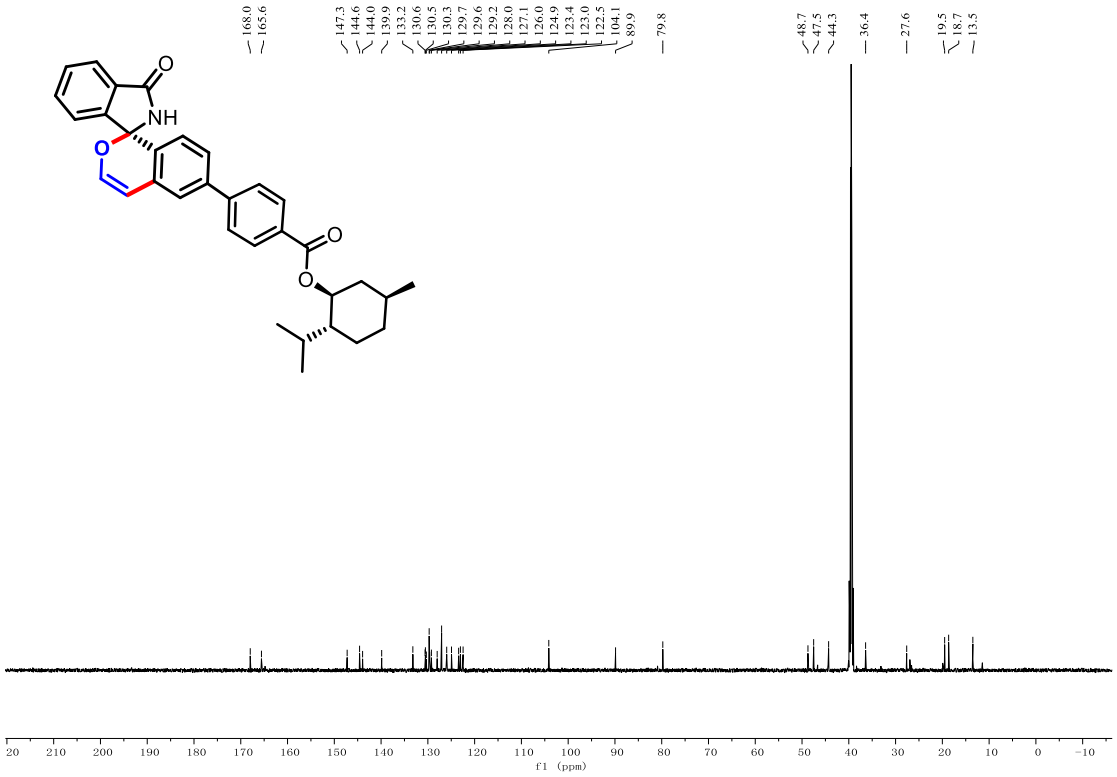
^{13}C NMR of **3a"** (600 MHz,DMSO)



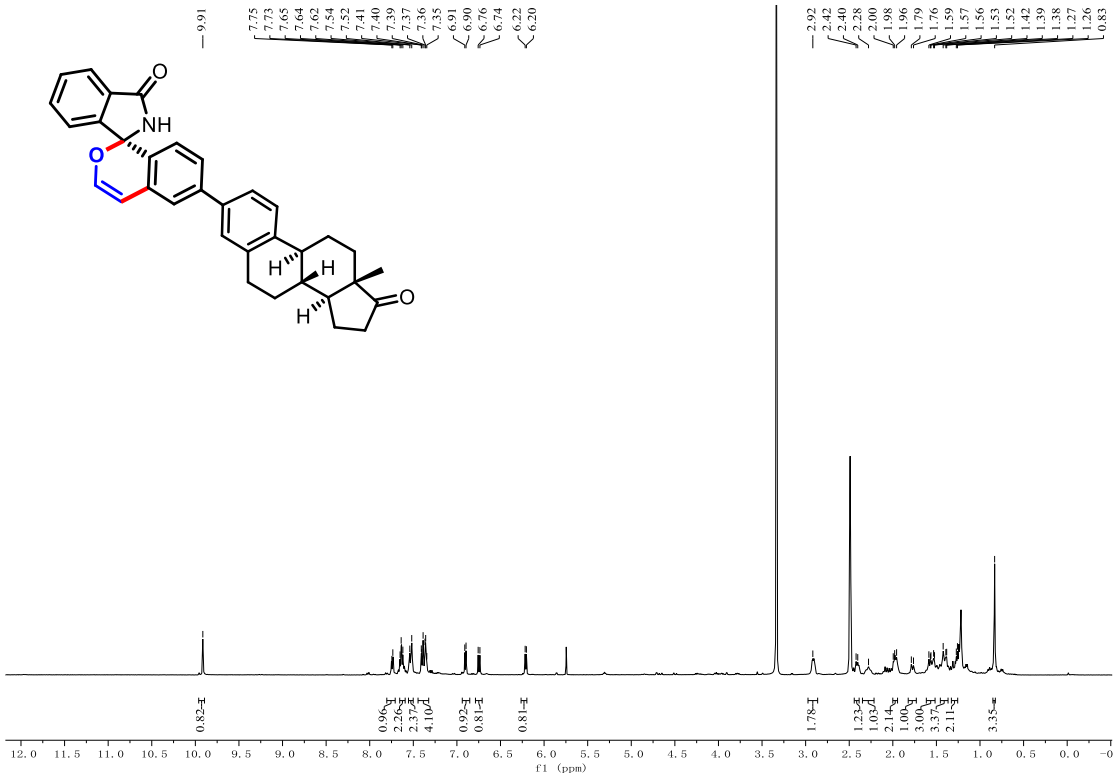
^{1}H NMR of **3b"** (600 MHz, DMSO)



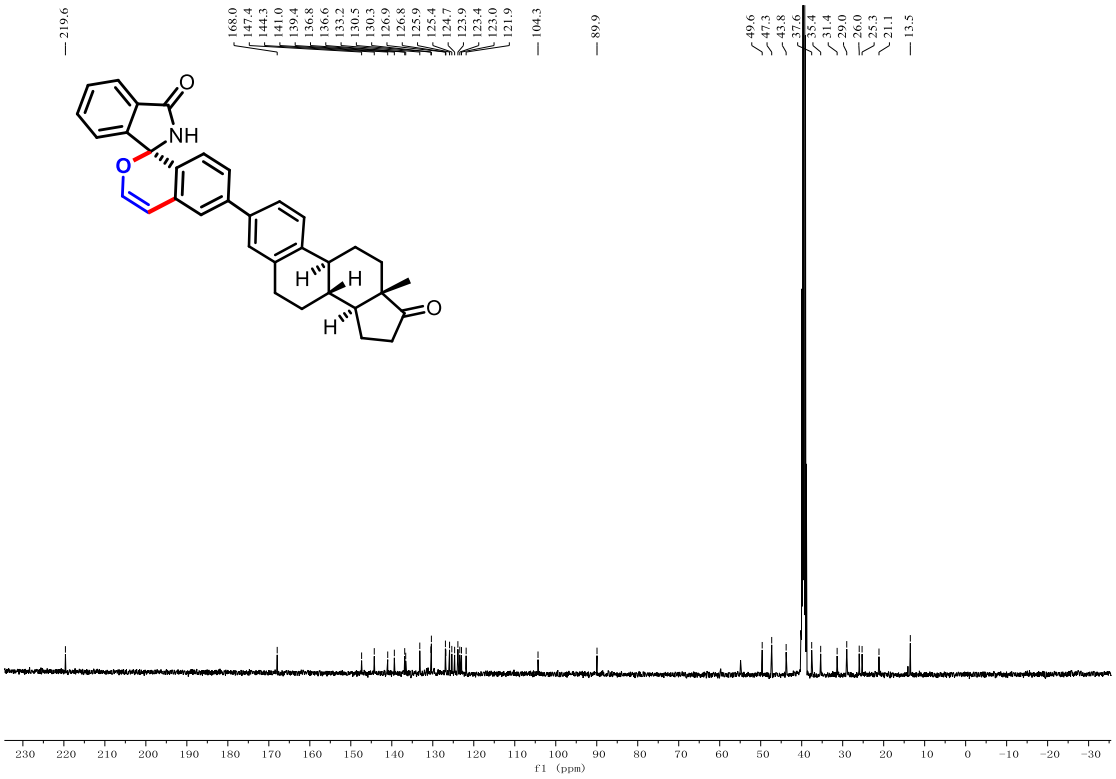
^{13}C NMR of **3b"** (600 MHz,DMSO)



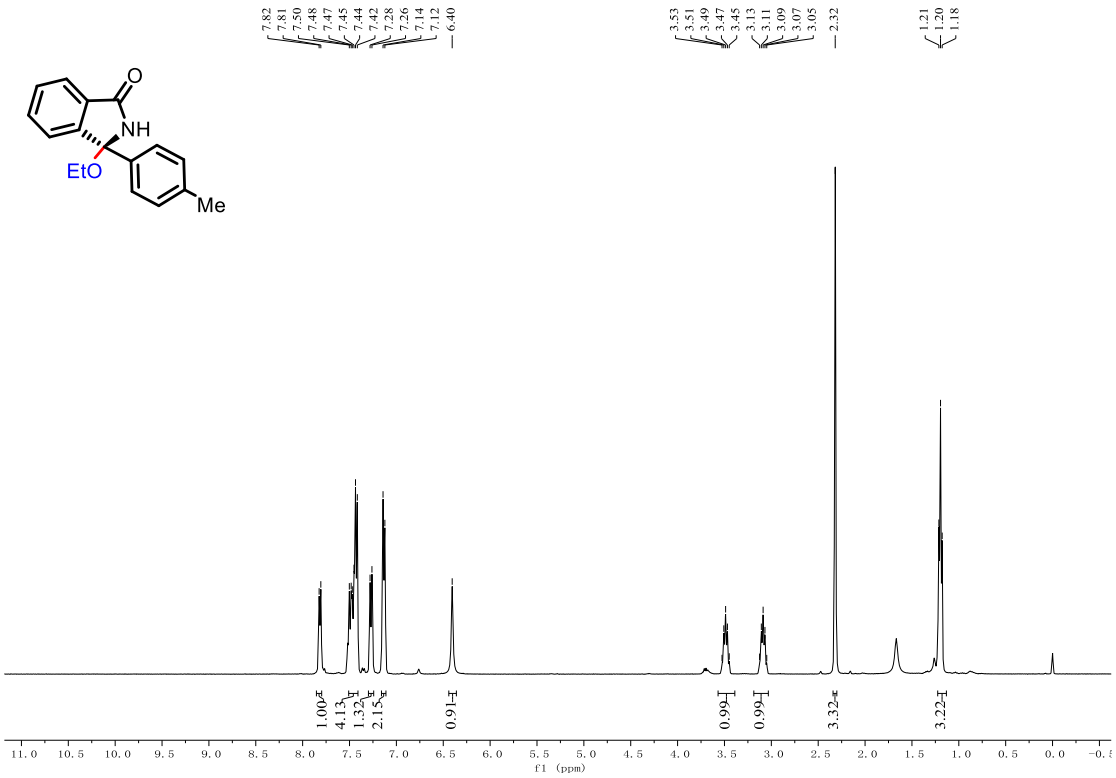
^{1}H NMR of **3c"** (400 MHz, DMSO)



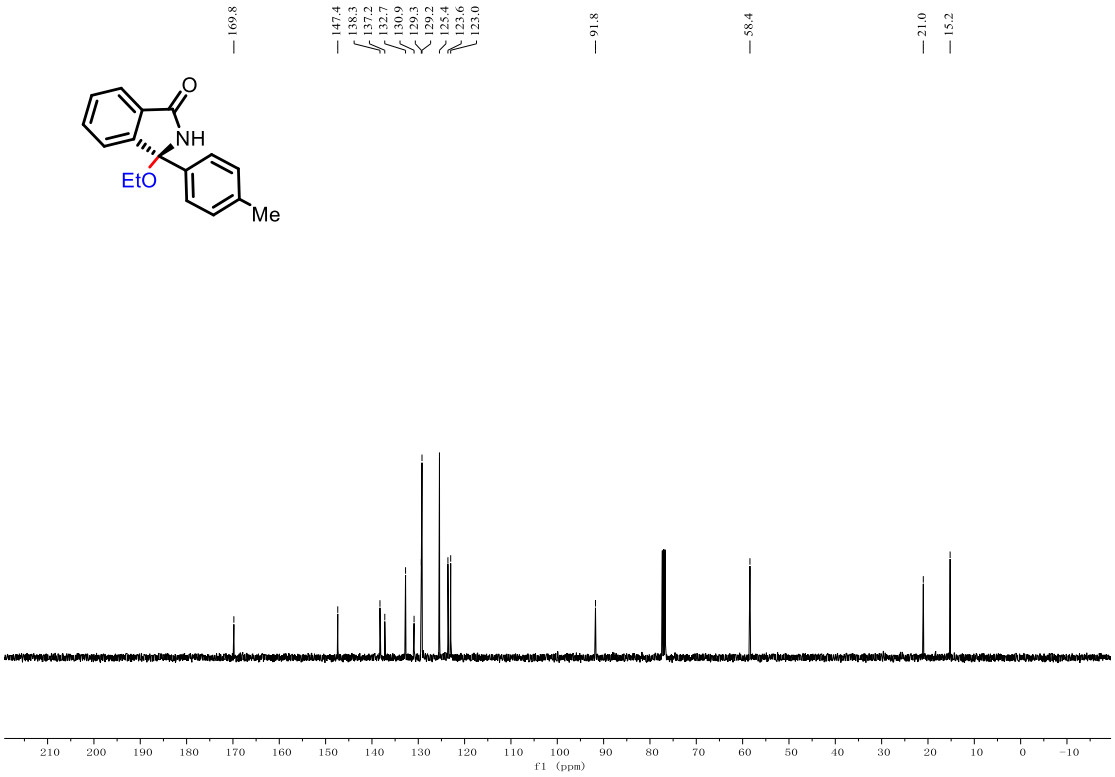
^{13}C NMR of **3c"** (600 MHz,DMSO)



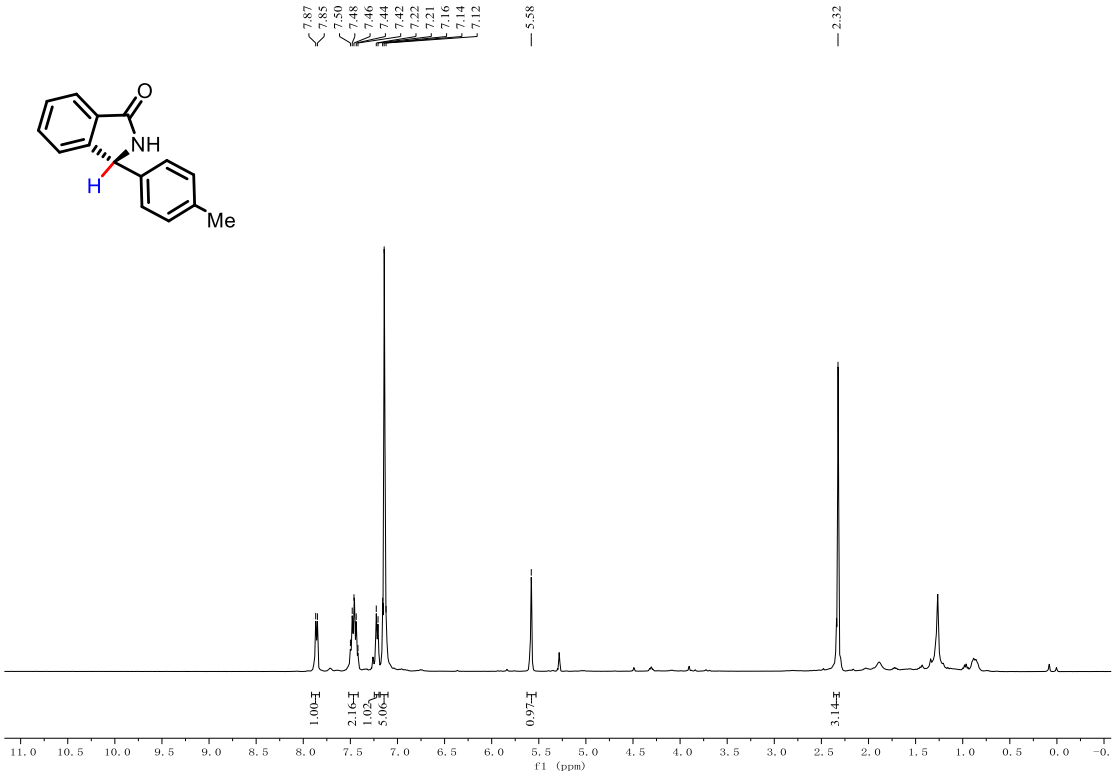
^{1}H NMR of **9** (400 MHz, $CDCl_3$)



^{13}C NMR of **9** (400 MHz, $CDCl_3$)



^{1}H NMR of **10** (400 MHz, $CDCl_3$)



^{13}C NMR of **10** (400 MHz, $CDCl_3$)