

Surpporting Information

Copper-Catalyzed Arylsulfonylation of N-Arylsulfonylacrylamides with Arylsulfonylhydrazides: Synthesis of Sulfonated Oxindoles

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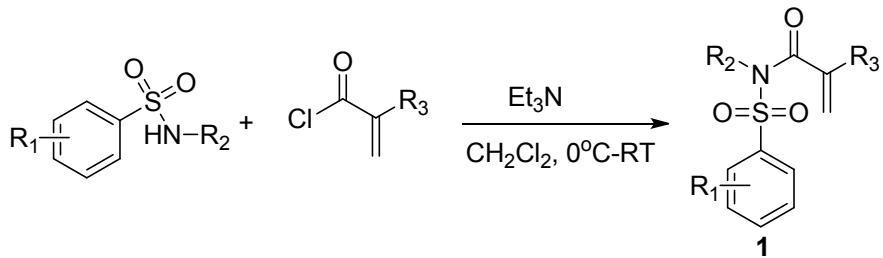
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General Experimental Details:

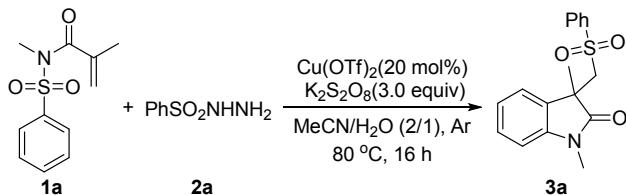
All manipulations were carried out under argon using standard Schlenk techniques. All glassware was oven or flame dried immediately prior to use. All solvents were purified and dried according to standard methods prior to use, unless stated otherwise. All reagents were obtained from commercial sources and used without further purification. Thin-layer chromatography (TLC) was performed using 60 mesh silica gel plates visualized with short-wavelength UV light (254 nm). Silica gel 60 (230 - 400 mesh) was used for column chromatography. ^1H NMR spectra were obtained at 400 MHz and recorded relative to the tetramethylsilane signal (0 ppm) or residual protio-solvent. ^{13}C NMR spectra were obtained at 100 MHz, and chemical shifts were recorded relative to the solvent resonance (CDCl_3 , 77.0 ppm). Data for ^1H NMR are recorded as follows: chemical shift (δ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, br = broad singlet, coupling constant(s) in Hz, integration). Data for ^{13}C NMR are reported in terms of chemical shift (δ , ppm). IR spectra were recorded on a Nicolet FT-IR spectrometer and only major peaks are reported in cm^{-1} . High resolution mass spectra were obtained using an Agilent 6210 Series TOF LC-MS equipped with electrospray ionization (ESI) probe operating in positive ion mode. Melting points were measured with SGW-4 micro melting point apparatus without further correction. Arylsulfonohydrazides **2** were prepared from arylsulfonyl chlorides.

General Procedure for preparation of substrats

All of compounds in Table 2 and Table 3 were synthesized according to the literature, and the NMR spectroscopy were consisted with those data.¹

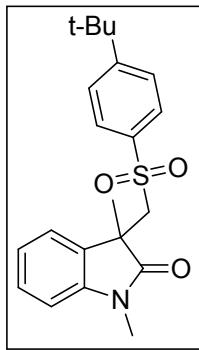


General Procedure of copper-catalyzed radical reaction of 1 and sulfonohydrazides.



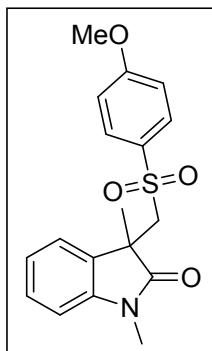
An oven-dried 15 mL screw-capped vial containing **1a** (0.2 mmol, 1.0 equiv), **2a** (0.4 mmol, 2.0 equiv), Cu(OTf)₂ (0.04 mmol, 0.2 equiv) and K₂S₂O₈ (0.6 mmol, 3.0 equiv) were evacuated and purged with Ar three times. Then, CH₃CN/H₂O(2/1, 1.00 mL) was added via syringe. The reaction mixture was stirred at 80 °C for 16 h. After cooling to room temperature, the solvent was evaporated and then the residue was purified on a silica gel column using petroleum ether /ethyl acetate (4/1) as eluent to give the desired product **3a**.

The data of characterization



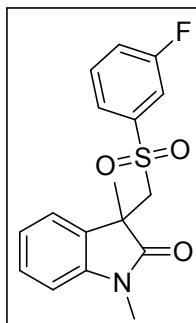
3-(((4-(tert-butyl)phenyl)sulfonyl)methyl)-1,3-dimethylindolin-2-one (3b)

White solid; mp 147°C; ¹H NMR (400 MHz, CDCl₃) δ 7.41 (q, J = 8.6 Hz, 4H), 7.28 (t, J = 3.3 Hz, 1H), 7.07 (d, J = 7.3 Hz, 1H), 6.89 (dd, J = 17.8, 7.7 Hz, 2H), 3.89 (d, J = 14.5 Hz, 1H), 3.69 (d, J = 14.5 Hz, 1H), 3.19 (s, 3H), 1.41 (s, 3H), 1.34 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 177.67, 157.21, 143.27, 136.88, 129.58, 128.58, 127.63, 125.94, 124.10, 122.50, 108.37, 61.87, 45.64, 35.19, 31.07, 26.58, 25.49; IR_{max}(thin film) (cm⁻¹)=3410, 2969, 1713, 1652, 1616, 1316, 1128, 753, 518, 620; HRMS (ESI-TOF) m/z: calcd for C₂₁H₂₅NNaO₃S⁺:394.1453 (M+Na)⁺, found: 394.1455.



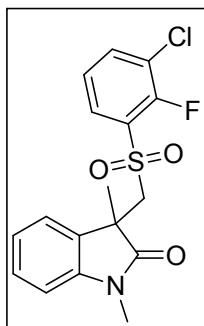
3-(((4-methoxyphenyl)sulfonyl)methyl)-1,3-dimethylindolin-2-one(3c)

White solid; mp 167°C; ¹H NMR (400 MHz, CDCl₃) δ 7.43 (d, J = 8.9 Hz, 2H), 7.32 (m, 1H), 7.13 (d, J = 7.4 Hz, 1H), 6.98 (t, J = 7.5 Hz, 1H), 6.86 (t, J = 8.2 Hz, 3H), 3.89 (d, J = 13.5 Hz, 4H), 3.67 (d, J = 14.5 Hz, 1H), 3.18 (s, 3H), 1.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 177.70, 163.50, 143.26, 131.54, 130.10, 129.67, 128.60, 124.21, 122.54, 114.06, 108.38, 62.07, 55.68, 45.70, 26.56, 25.58; IR: _{max}(thin film) (cm⁻¹)=2969, 2925, 1715, 1317, 1155, 752; HRMS (ESI-TOF) m/z: calcd for C₁₈H₁₄NNaO₄S⁺:368.0932 (M+Na)⁺, found: 368.0938.



3-(((3-fluorophenyl)sulfonyl)methyl)-1,3-dimethylindolin-2-one(3d)

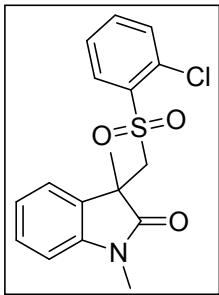
White solid; mp 135°C ; ¹H NMR (400 MHz, CDCl₃) δ 7.42-7.45 (m, 3H), 7.25 (s, 1H), 7.09 (d, J = 7.7 Hz, 1H), 6.99 (d, J = 7.2 Hz, 1H), 6.91 (d, J = 7.7 Hz, 1H), 3.93 (d, J = 14.6 Hz, 1H), 3.72 (d, J = 14.7 Hz, 1H), 3.24 (s, 3H), 1.42 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 177.46, 160.88 (d, J = 244.4Hz), 143.33, 141.62, 133.86, 131.48, 130.77 (d, J = 7.6 Hz), 129.09, 128.87, 128.41, 123.73, 122.76, 122.48, 120.74, 120.58, 116.27, 115.24 (d, J = 24.4 Hz), 108.51, 62.08, 45.56, 26.58, 25.36; ¹⁹F (376 MHz, CDCl₃) δ -109.43; IR: _{max}(thin film) (cm⁻¹)=2981, 2911, 1714, 1493, 1285, 805; HRMS (ESI-TOF) m/z: calcd for C₁₇H₁₆FNNaO₃S⁺:356.0733 (M+Na)⁺, found: 356.0731.



3-(((3-chloro-2-fluorophenyl)sulfonyl)methyl)-1,3-dimethylindolin-2-one (3e)

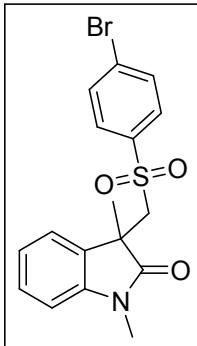
White solid; mp 144°C; ¹H NMR (400 MHz, CDCl₃) δ 7.56 (m, 1H), 7.18 (m, 1H), 7.08 (m, 1H),

6.97 (d, $J = 8.0$ Hz, 1H), 6.93 (m, 1H), 6.82 (d, $J = 7.8$ Hz, 1H), 6.74 (t, $J = 7.5$ Hz, 1H), 4.03 (d, $J = 14.9$ Hz, 1H), 3.97 (d, $J = 14.9$ Hz, 1H), 3.23 (s, 3H), 1.44 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 177.28, 155.79 (d, $J = 239.04$ Hz), 143.38, 135.93, 129.51 (d, $J = 14.7$ Hz), 128.95, 128.64, 128.22, 127.99, 127.05, 124.79 (d, $J = 4.8$ Hz), 123.24, 122.63 (d, $J = 18.0$ Hz), 122.33, 108.47, 61.30, 45.39, 26.62, 25.00; ^{19}F (376 MHz, CDCl_3) δ -110.87; IR: $\text{max}(\text{thin film}) (\text{cm}^{-1}) = 3414, 2969, 1715, 1611, 1652, 1616, 1128, 756, 516, 617$; HRMS (ESI-TOF) m/z: calcd for $\text{C}_{17}\text{H}_{15}\text{FClNNaO}_3\text{S}^+ : 390.0343$ ($\text{M}+\text{Na}^+$), found: 390.0344.



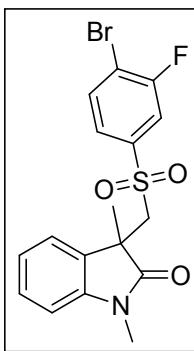
3-((2-chlorophenyl)sulfonyl)methyl-1,3-dimethylindolin-2-one (3f)

White solid; mp 152°C; ^1H NMR (400 MHz, CDCl_3) δ 7.49-7.41 (m, 2H), 7.31-7.29 (m, 1H), 7.20-7.16 (m, 1H), 7.13-7.09 (m, 1H), 6.84 (dd, $J = 16.4, 7.6$ Hz, 2H), 6.66 (t, $J = 7.5$ Hz, 1H), 4.23 (d, $J = 14.9$ Hz, 1H), 4.03 (d, $J = 14.9$ Hz, 1H), 3.25 (s, 3H), 1.43 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 177.55, 143.36, 137.49, 134.21, 132.11, 131.45, 130.87, 129.24, 128.67, 127.19, 123.22, 122.27, 108.29, 59.73, 45.45, 26.63, 25.11; IR: $\text{max}(\text{thin film}) (\text{cm}^{-1}) = 2960, 2925, 1711, 1614, 1530, 1281, 805$; HRMS (ESI-TOF) m/z: calcd for $\text{C}_{17}\text{H}_{16}\text{ClNNaO}_3\text{S}^+ : 372.0437$ ($\text{M}+\text{Na}^+$), found: 372.0439.



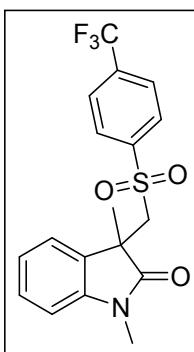
3-((4-bromophenyl)sulfonyl)methyl-1,3-dimethylindolin-2-one (3g)

White solid; mp 163°C; ^1H NMR (400 MHz, CDCl_3) δ 7.50 (d, $J = 8.6$ Hz, 2H), 7.30 (m, 3H), 6.99 (d, $J = 6.8$ Hz, 1H), 6.92 (dd, $J = 7.5, 0.6$ Hz, 1H), 6.86 (d, $J = 7.8$ Hz, 1H), 3.92 (d, $J = 14.7$ Hz, 1H), 3.70 (d, $J = 14.7$ Hz, 1H), 3.18 (s, 3H), 1.39 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 175.00, 163.27, 140.35, 133.03, 129.73, 128.76, 127.76, 126.85, 114.14, 64.41, 49.45, 26.94, 22.56; IR: $\text{max}(\text{thin film}) (\text{cm}^{-1}) = 3235, 2926, 1638, 1616, 622, 478$; IR: $\text{max}(\text{thin film}) (\text{cm}^{-1}) = 2959, 2924, 1713, 1615, 1531, 1280, 807$; HRMS (ESI-TOF) m/z: calcd for $\text{C}_{17}\text{H}_{16}\text{BrNNaO}_3\text{S}^+ : 415.9932$ ($\text{M}+\text{Na}^+$), found: 415.9935.



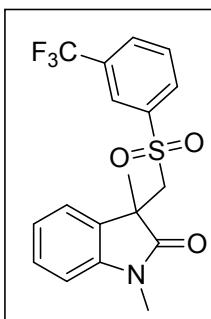
3-(((4-bromo-3-fluorophenyl)sulfonyl)methyl)-1,3-dimethylindolin-2-one(3h)

White solid; mp 139°C; ¹H NMR (400 MHz, CDCl₃) δ 7.62 (dd, J = 8.2, 6.5 Hz, 1H), 7.34 (t, J = 7.4 Hz, 1H), 7.21 (d, J = 7.2 Hz, 1H), 7.07 (m, 1H), 6.93 (dd, J = 16.1, 7.0 Hz, 3H), 3.95 (d, J = 14.7 Hz, 1H), 3.71 (d, J = 14.7 Hz, 1H), 3.23 (s, 4H), 1.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 177.31, 159.18(d, J = 254.52 Hz), 134.37 (d, J = 7.56 Hz), 143.43, 140.87, 134.31, 129.09, 127.84, 124.42, 123.75, 122.44, 116.26 (d, J = 25.20 Hz), 108.58, 62.19, 45.49, 26.62, 25.45; ¹⁹F (376 MHz, CDCl₃) δ -102.88; IR_{max}(thin film) (cm⁻¹)=3013, 2934, 1711, 1618, 1531, 1277, 799; HRMS (ESI-TOF) m/z: calcd for C₁₇H₁₅FBrNNaO₃S⁺:433.9838 (M+Na)⁺, found: 433.9840.



1,3-dimethyl-3-(((4-(trifluoromethyl)phenyl)sulfonyl)methyl)indolin-2-one(3i)

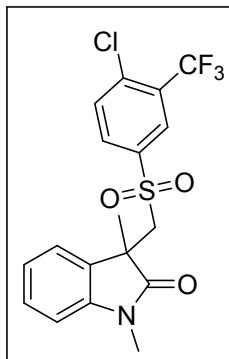
White solid; mp 155°C; ¹H NMR (400 MHz, CDCl₃) δ 7.62 (dd, J = 19.0, 8.4 Hz, 4H), 7.32 (d, J = 7.0 Hz, 1H), 6.94 (d, J = 7.2 Hz, 1H), 6.87 (m, 2H), 3.97 (q, J = 14.7 Hz, 1H), 3.74 (d, J = 14.7 Hz, 1H), 3.20 (s, 3H), 1.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 177.33, 143.37, 129.08, 128.89, 128.44, 125.97, 123.83(q, J = 269.8 Hz), 122.59, 108.54, 61.90, 45.52, 26.58, 25.55; ¹⁹F (376 MHz, CDCl₃) δ -62.63; IR_{max}(thin film) (cm⁻¹)=2990, 2932, 1713, 1619, 1521, 1270, 798; HRMS (ESI-TOF) m/z: calcd for C₁₈H₁₆F₃NNaO₃S⁺: 406.0701 (M+Na)⁺, found: 406.0705.



1,3-dimethyl-3-(((3-(trifluoromethyl)phenyl)sulfonyl)methyl)indolin-2-one (3j)

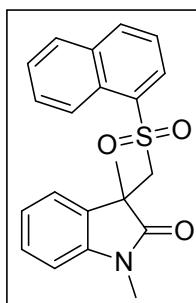
White solid; mp 148°C; ¹H NMR (400 MHz, CDCl₃) δ 7.81 (d, J = 7.7 Hz, 1H), 7.74 (d, J = 7.9 Hz, 1H), 7.65 (s, 1H), 7.58 (t, J = 7.8 Hz, 1H), 7.29 (m, 1H), 6.93 (d, J = 7.3 Hz, 1H), 6.86 (m,

2H), 3.99 (d, J = 14.7 Hz, 1H), 3.77 (d, J = 14.7 Hz, 1H), 3.21 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.98, 138.67, 138.25, 132.24, 132.06, 129.22, 128.66, 127.70, 123.69, 122.45, 108.65, 62.04, 26.57, 25.60; ^{19}F (376 MHz, CDCl_3) δ -62.61; IR: $\text{max}(\text{thin film}) (\text{cm}^{-1})$ = 3013, 2930, 1718, 1630, 1522, 1271, 801; HRMS (ESI-TOF) m/z: calcd for $\text{C}_{18}\text{H}_{16}\text{F}_3\text{NNaO}_3\text{S}^+$: 406.0701 ($\text{M}+\text{Na})^+$, found: 406.0702.



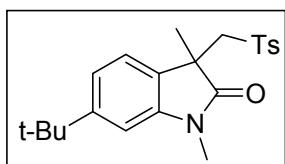
3-(((4-chloro-3-(trifluoromethyl)phenyl)sulfonyl)methyl)-1,3-dimethylindolin-2-one (3k)

White solid; mp 163 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.64-7.62 (m, 1H), 7.57-7.55 (m, 2H), 7.34-7.30 (m, 1H), 6.89-6.87 (dd, J = 6.7, 3.7 Hz, 3H), 4.02 (d, J = 14.8 Hz, 1H), 3.75 (d, J = 14.8 Hz, 1H), 3.21 (s, 3H), 1.41 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.98, 141.37, 138.38, 132.24, 132.06, 129.22, 128.49, 126.16(q, J = 4.50 Hz), 123.69 (q, J = 269.8 Hz), 122.45, 108.65, 61.95, 46.11, 26.34, 26.00; ^{19}F (376 MHz, CDCl_3) δ -62.90; IR: $\text{max}(\text{thin film}) (\text{cm}^{-1})$ = 3011, 2922, 1703, 1615, 1501, 1283, 813; HRMS (ESI-TOF) m/z: calcd for $\text{C}_{18}\text{H}_{15}\text{ClF}_3\text{NNaO}_3\text{S}^+$: 440.0311 ($\text{M}+\text{Na})^+$, found: 440.0311.



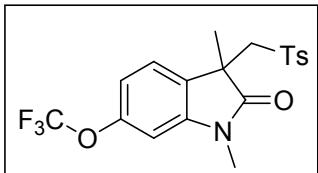
1,3-dimethyl-3-((naphthalen-1-ylsulfonyl)methyl)indolin-2-one (3l)

White solid; mp 151 °C ^1H NMR (400 MHz, CDCl_3) δ 7.89 (m, 3H), 7.79 (d, J = 8.1 Hz, 1H), 7.67 (m, 1H), 7.61 (m, 2H), 7.24 (m, 1H), 7.05 (d, J = 7.2 Hz, 1H), 6.79 (m, 2H), 4.01 (d, J = 14.6 Hz, 1H), 3.77 (d, J = 14.6 Hz, 1H), 2.99 (s, 3H), 1.40 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 177.55, 136.30, 135.15, 131.82, 130.00, 129.56, 129.25, 129.23, 129.18, 128.69, 127.84, 127.43, 124.18, 122.59, 122.39, 108.36, 61.61, 45.61, 26.37, 25.73; IR: $\text{max}(\text{thin film}) (\text{cm}^{-1})$ = 2991, 2932, 1712, 1620, 1525, 1269, 801; HRMS (ESI-TOF) m/z: calcd for $\text{C}_{21}\text{H}_{19}\text{NNaO}_3\text{S}^+$: 388.0983 ($\text{M}+\text{Na})^+$, found: 388.0985.



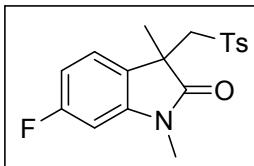
6-(tert-butyl)-1,3-dimethyl-3-(tosylmethyl)indolin-2-one (4b)

White solid; mp 131°C; ¹H NMR (400 MHz, CDCl₃) δ 7.34 (d, J = 8.2 Hz, 2H), 7.15 (d, J = 8.0 Hz, 2H), 6.91 (m, 2H), 6.85 (s, 1H), 3.87 (d, J = 14.6 Hz, 1H), 3.69 (d, J = 14.6 Hz, 1H), 3.20 (s, 3H), 2.41 (s, 3H), 1.38 (s, 9H); ¹³C NMR (100 MHz, CDCl₃) δ 152.23, 144.07, 129.39, 127.85, 123.61, 119.29, 105.61, 61.97, 38.45, 29.64, 25.95, 21.55; IR: max(thin film) (cm⁻¹)=3210, 2971, 1715, 1655, 1618, 1310, 1130, 780, 520; HRMS (ESI-TOF) m/z: calcd for C₂₂H₂₇NNaO₃S⁺: 408.1609 (M+Na)⁺, found: 408.1610.



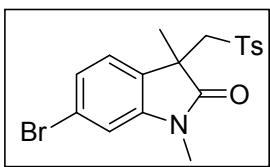
1,3-dimethyl-3-(tosylmethyl)-6-(trifluoromethoxy)indolin-2-one (4c)

Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.40 (d, J = 8.2 Hz, 2H), 7.21 (d, J = 8.1 Hz, 2H), 7.03 (d, J = 8.0 Hz, 1H), 6.74 (d, J = 11.8 Hz, 2H), 3.89 (d, J = 14.6 Hz, 1H), 3.69 (d, J = 14.6 Hz, 1H), 3.20 (s, 3H), 2.42 (s, 3H), 1.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 177.76, 149.65, 144.70, 136.99, 132.30, 130.66, 130.62, 130.00, 127.86, 125.03, 120.54, 114.28, 102.12, 61.83, 45.37, 26.71, 25.33, 21.53; ¹⁹F (376 MHz, CDCl₃) δ -57.72; IR: max(thin film) (cm⁻¹)=3013, 2969, 1715, 1642, 1617, 1300, 1130, 789, 622, 518; HRMS (ESI-TOF) m/z: calcd for C₁₉H₁₈F₃NNaO₄S⁺: 436.0306 (M+Na)⁺, found: 436.0310.



6-fluoro-1,3-dimethyl-3-(tosylmethyl)indolin-2-one (4d)

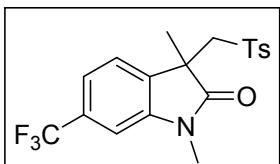
White solid; mp 149°C; ¹H NMR (400 MHz, CDCl₃) δ 7.44 (d, J = 8.2 Hz, 2H), 7.23 (d, J = 8.1 Hz, 2H), 7.04 (dd, J = 8.8, 5.3 Hz, 1H), 6.62 (m, 2H), 3.85 (d, J = 14.5 Hz, 1H), 3.66 (m, 1H), 3.17 (s, 3H), 2.43 (s, 3H), 1.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 178.05, 164.33 (d, J = 246.96 Hz), 144.84 (d, J = 11.7 Hz), 144.56, 137.11, 130.07, 129.75, 129.66 (d, J = 21.0 Hz), 128.23, 127.79, 127.31, 126.30 (d, J = 25.5 Hz), 125.23, 116.72, 116.54, 116.27, 116.14, 115.90 (d, J = 22.68 Hz), 108.54 (d, J = 22.5 Hz), 97.32, 97.10, 61.94, 45.30, 26.68, 25.43, 21.56; ¹⁹F (376 MHz, CDCl₃) δ -111.38; IR: max(thin film) (cm⁻¹)=2990, 2921, 1715, 1501, 11340, 807; HRMS (ESI-TOF) m/z: calcd for C₁₈H₁₈FNNaO₃S⁺: 370.0889 (M+Na)⁺, found: 370.0891.



6-bromo-1,3-dimethyl-3-(tosylmethyl)indolin-2-one(4e)

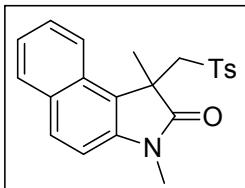
White solid; mp 169°C; ¹H NMR (400 MHz, CDCl₃) δ 7.44 (d, J = 8.2 Hz, 2H), 7.23 (d, J = 8.0 Hz, 2H), 7.06-7.02 (m, 2H), 6.95 (d, J = 7.9 Hz, 1H), 3.85 (d, J = 14.5 Hz, 1H), 3.65 (d, J = 14.5 Hz, 1H), 3.19 (s, 3H), 2.44 (s, 3H), 1.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 177.49, 144.64, 137.02, 129.61, 128.61, 127.76, 125.32, 122.32, 111.87, 61.85, 45.44, 26.66, 25.19, 21.59;

IR_{max}(thin film) (cm⁻¹)=3414, 2969, 1652, 1558, 1616, 617; HRMS (ESI-TOF) m/z: calcd for C₁₈H₁₈BrNNaO₃S⁺: 430.0088 (M+Na)⁺, found: 430.0091.



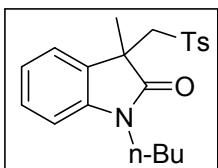
1,3-dimethyl-3-(tosylmethyl)-6-(trifluoromethyl)indolin-2-one (4f)

White solid; mp 126°C; ¹H NMR (400 MHz, CDCl₃) δ 7.41 (d, J = 8.2 Hz, 2H), 7.18 (dd, J = 15.0, 5.8 Hz, 4H), 7.07 (s, 1H), 3.90 (d, J = 14.6 Hz, 1H), 3.70 (d, J = 14.6 Hz, 1H), 3.26 (s, 3H), 2.42 (s, 3H), 1.43 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 177.34, 144.71, 143.92, 136.76, 129.66, 127.66 (q, JF-C=3.7Hz), 124.22 (q, JF-C=33.0Hz), 119.47 (q, JF-C=270.5Hz), 105.09, 61.70, 45.60, 26.76, 25.22, 21.55; ¹⁹F (376 MHz, CDCl₃) δ -62.46; IR_{max}(thin film) (cm⁻¹)=2989, 2930, 1710, 1623, 1532, 1269, 804; HRMS (ESI-TOF) m/z: calcd for C₁₉H₁₈F₃NNaO₃S⁺: 420.0857 (M+Na)⁺, found: 420.0855.



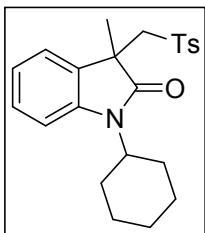
1,3-dimethyl-1-(tosylmethyl)-1H-benzo[e]indol-2(3H)-one (4g)

White solid; mp 189°C; ¹H NMR (400 MHz, CDCl₃) δ 8.43 (d, J = 9.2 Hz, 1H), 7.86 (dd, J = 6.4, 3.0 Hz, 1H), 7.51 (m, 2H), 7.44 (d, J = 8.2 Hz, 1H), 7.31 (d, J = 8.2 Hz, 2H), 7.20 (d, J = 8.2 Hz, 1H), 7.04 (d, J = 8.0 Hz, 2H), 3.97 (d, J = 14.5 Hz, 1H), 3.76 (d, J = 13.5 Hz, 4H), 2.38 (s, 3H), 1.45 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 179.22, 144.32, 138.38, 136.61, 134.77, 129.40, 129.36, 127.92, 125.92, 125.75, 125.42, 123.02, 121.59, 121.18, 120.86, 62.18, 45.45, 31.10, 25.67, 21.55; IR_{max}(thin film) (cm⁻¹)=2996, 2935, 1718, 1623, 1518, 1267, 798; HRMS (ESI-TOF) m/z: calcd for C₂₂H₂₁NNaO₃S⁺: 402.1140 (M+Na)⁺, found: 402.1144.



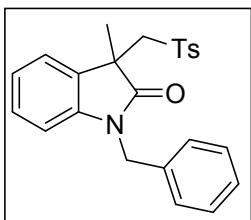
1-butyl-3-methyl-3-(tosylmethyl)indolin-2-one (4h)

White solid; mp 139°C; ¹H NMR (400 MHz, CDCl₃) δ 7.40 (d, J = 8.2 Hz, 2H), 7.25 (m, 1H), 7.17 (d, J = 8.1 Hz, 2H), 7.06 (d, J = 7.3 Hz, 1H), 6.88 (dd, J = 13.8, 7.5 Hz, 2H), 3.82 (d, J = 14.5 Hz, 1H), 3.64 (m, 3H), 2.39 (s, 3H), 1.67 (m, 2H), 1.40 (m, 5H), 0.96 (t, J = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 177.57, 144.28, 142.75, 137.27, 129.87, 129.53, 128.44, 127.80, 124.25, 122.18, 108.63, 61.79, 45.64, 40.09, 29.22, 25.69, 21.59, 20.22, 13.78; IR_{max}(thin film) (cm⁻¹)=2998, 2929, 1719, 1622, 1533, 1280, 805, 619; HRMS (ESI-TOF) m/z: calcd for C₂₁H₂₅NNaO₃S⁺: 394.1453 (M+Na)⁺, found: 394.1458.



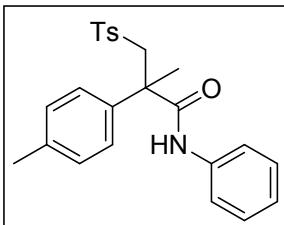
1-cyclohexyl-3-methyl-3-(tosylmethyl)indolin-2-one(4i)

Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.41 (d, *J* = 8.2 Hz, 2H), 7.23 (t, *J* = 7.3 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.10 (d, *J* = 7.9 Hz, 1H), 6.95 (d, *J* = 7.3 Hz, 1H), 6.81 (t, *J* = 7.4 Hz, 1H), 4.24 (m, 1H), 3.85 (d, *J* = 14.5 Hz, 1H), 3.65 (d, *J* = 14.5 Hz, 1H), 2.40 (s, 3H), 2.23-2.13 (m, 2H), 1.95-1.76 (m, 4H), 1.48-1.43 (m, 2H), 1.37 (s, 3H), 1.31-1.26 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 177.64, 144.13, 142.46, 137.55, 130.05, 129.57, 128.11, 127.62, 124.01, 121.70, 110.38, 62.05, 52.45, 45.35, 29.14, 28.57, 26.03, 26.00, 25.76, 25.51, 21.56; IR: _{max}(thin film) (cm⁻¹)=2997, 2928, 1715, 1623, 1519, 1274, 805; HRMS (ESI-TOF) m/z: calcd for C₂₃H₂₇NNaO₃S⁺: 420.1609 (M+Na)⁺; found: 420.1610.



1-benzyl-3-methyl-3-(tosylmethyl)indolin-2-one(4j)

Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.44 (d, *J* = 8.2 Hz, 2H), 7.36 (m, 4H), 7.29 (d, *J* = 6.0 Hz, 1H), 7.16 (m, 3H), 7.05 (d, *J* = 7.4 Hz, 1H), 6.86 (t, *J* = 7.5 Hz, 1H), 6.72 (d, *J* = 7.8 Hz, 1H), 5.02 (d, *J* = 15.7 Hz, 1H), 4.81 (d, *J* = 15.8 Hz, 1H), 3.93 (d, *J* = 14.5 Hz, 1H), 3.74 (d, *J* = 14.5 Hz, 1H), 2.41 (s, 3H), 1.47 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 177.88 (s), 144.36, 142.35, 137.17, 135.80, 129.62, 128.78, 128.40, 127.82, 127.61, 127.34, 124.00, 122.47, 109.55, 61.67, 45.78, 44.22, 26.02, 21.59; IR: _{max}(thin film) (cm⁻¹)=3412, 2973, 2926, 1711, 1613, 1450, 1383, 1309, 1150, 1126, 754, 523; HRMS (ESI-TOF) m/z: calcd for C₂₄H₂₃NNaO₃S⁺: 428.1296 (M+Na)⁺; found: 428.1294.



2-methyl-N,2-diphenyl-3-tosylpropanamide(6a)

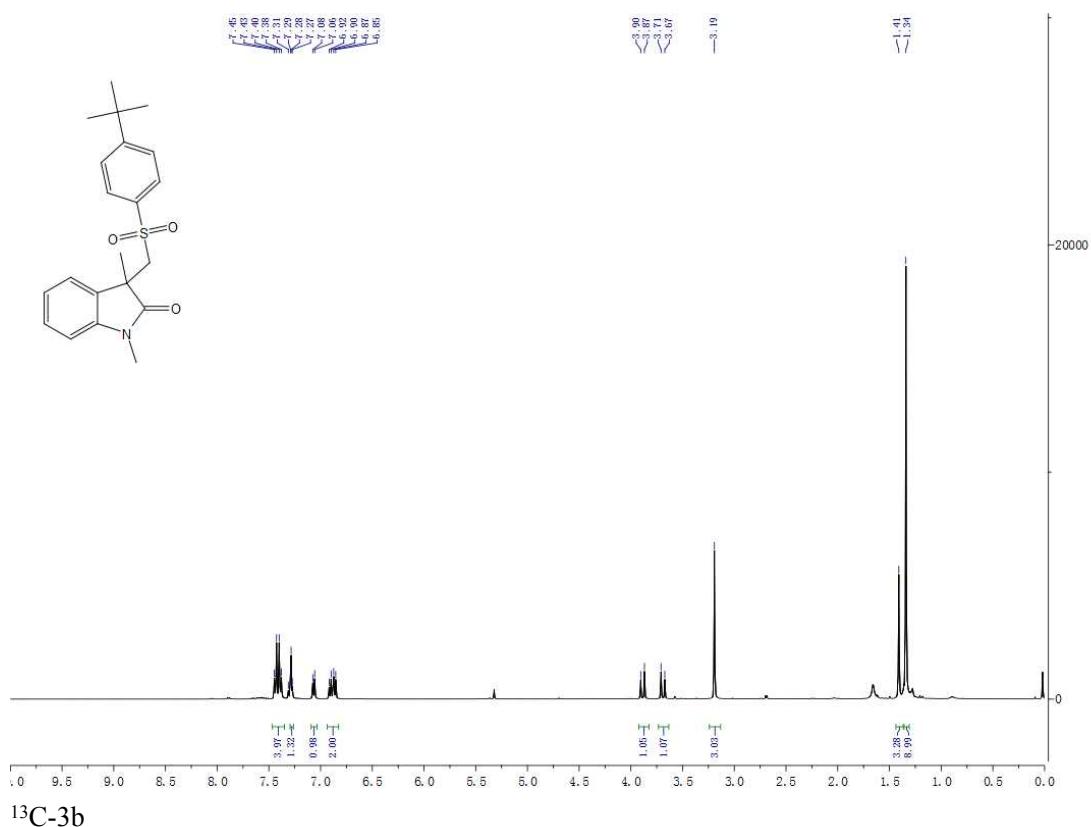
Colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 7.54 (d, *J* = 8.2 Hz, 2H), 7.35-7.27 (m, 5H), 7.22-7.18 (m, 3H), 7.12-7.07 (dd, *J* = 13.1, 7.5 Hz, 3H), 6.92 (s, 1H), 4.16 (d, *J* = 14.8 Hz, 1H), 3.85 (d, *J* = 14.9 Hz, 1H), 2.41 (s, 3H), 2.34 (s, 3H), 2.13 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 172.90, 143.97, 138.09, 137.38, 136.27, 129.57, 128.95, 127.60, 126.96, 124.66, 120.07, 64.05, 50.05, 22.73, 21.58, 21.03; HRMS (ESI-TOF) m/z: calcd for C₂₄H₂₅NNaO₃S⁺: 430.1453 (M+Na)⁺; found: 430.1455.

Reference

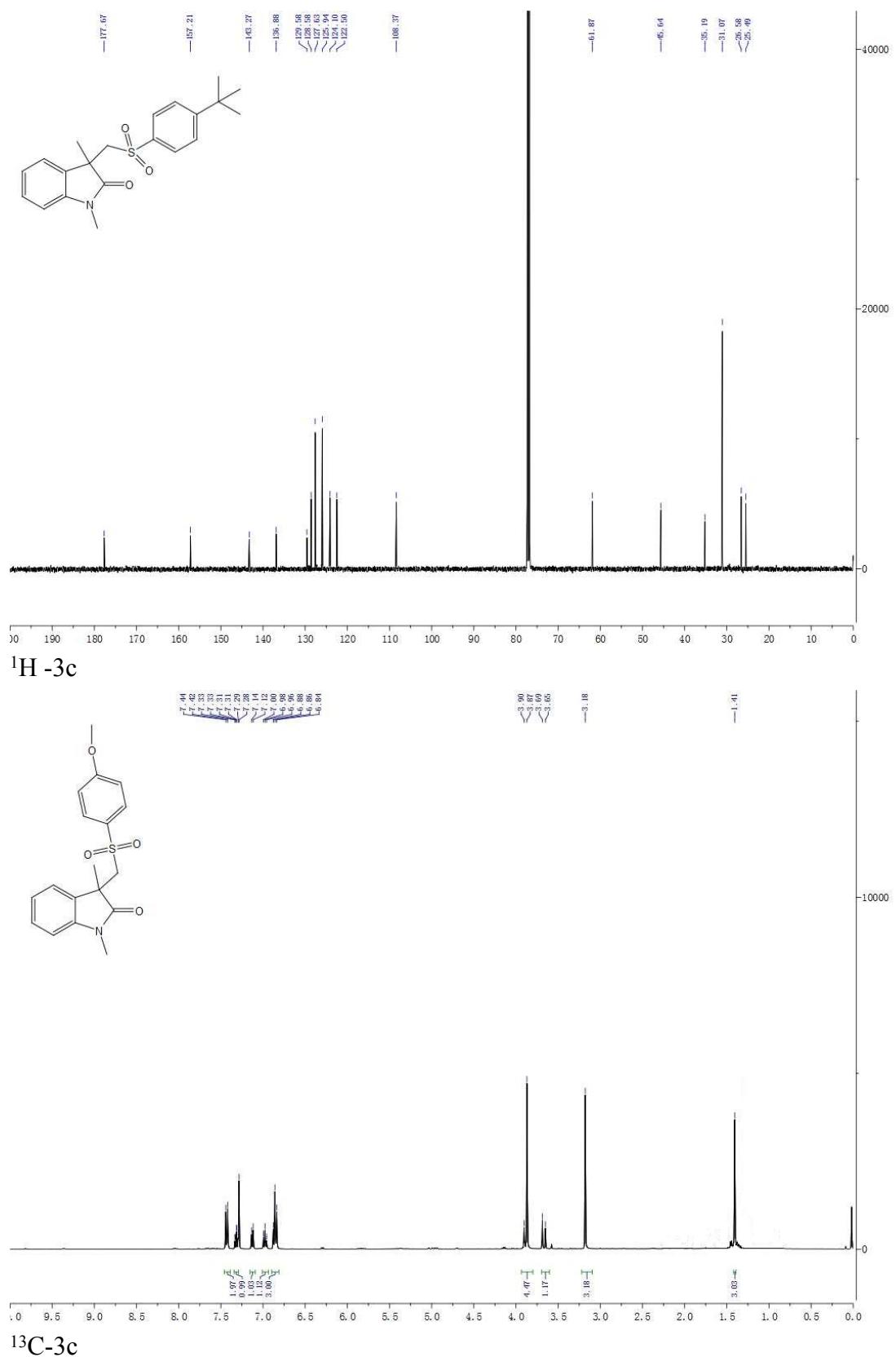
- W. Kong, M. Casimiro, E. Merino, C. Nevado, J. Am. Chem. Soc. 2013, 135, 14480.

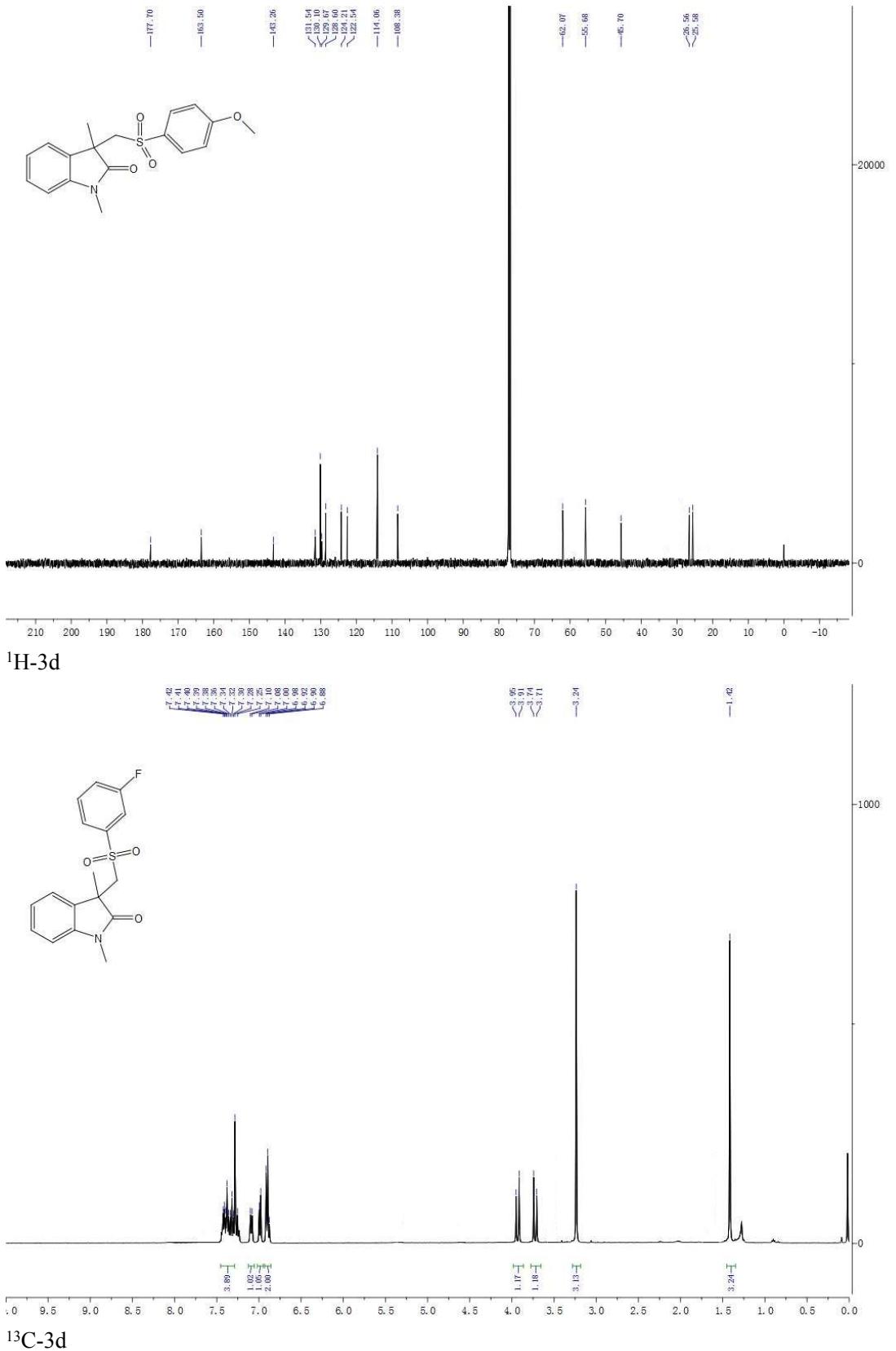
NMR Spectra of compounds

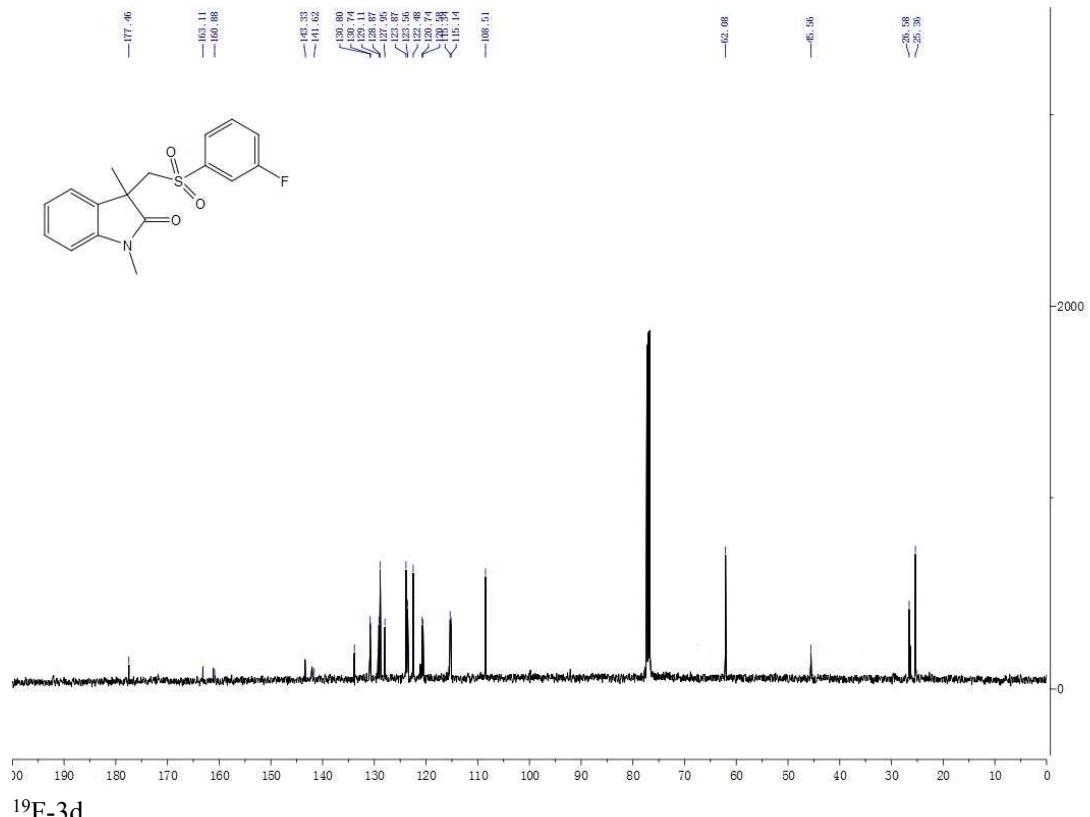
¹H -3b



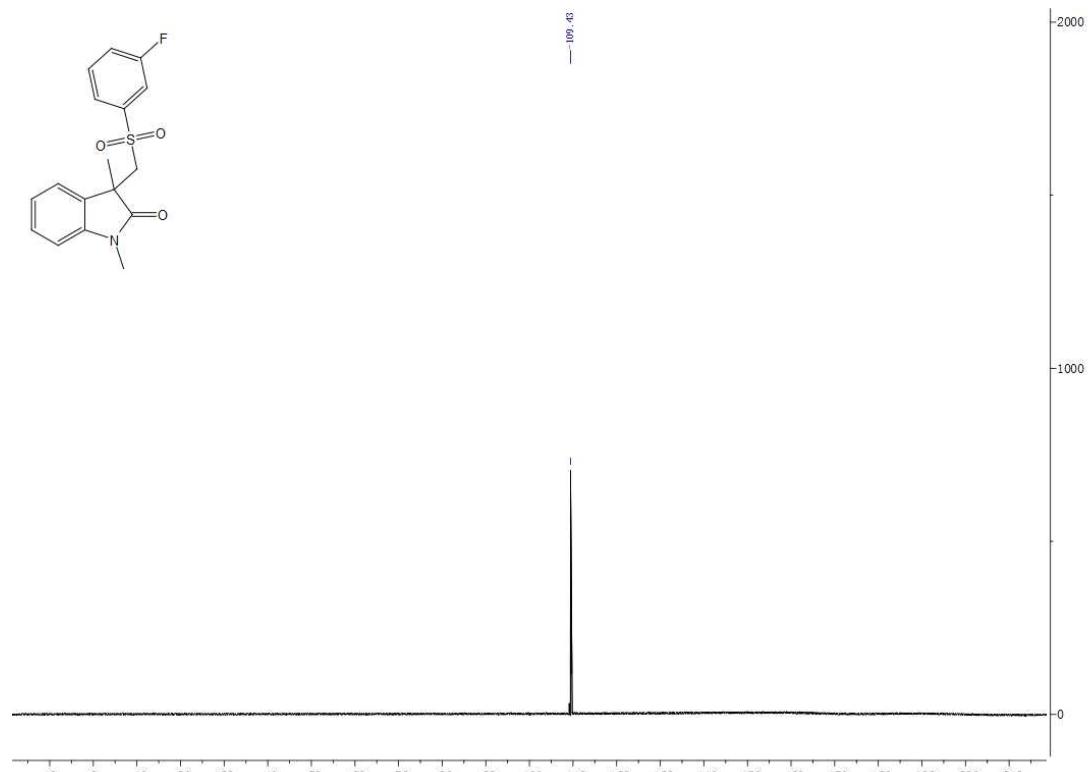
¹³C-3b



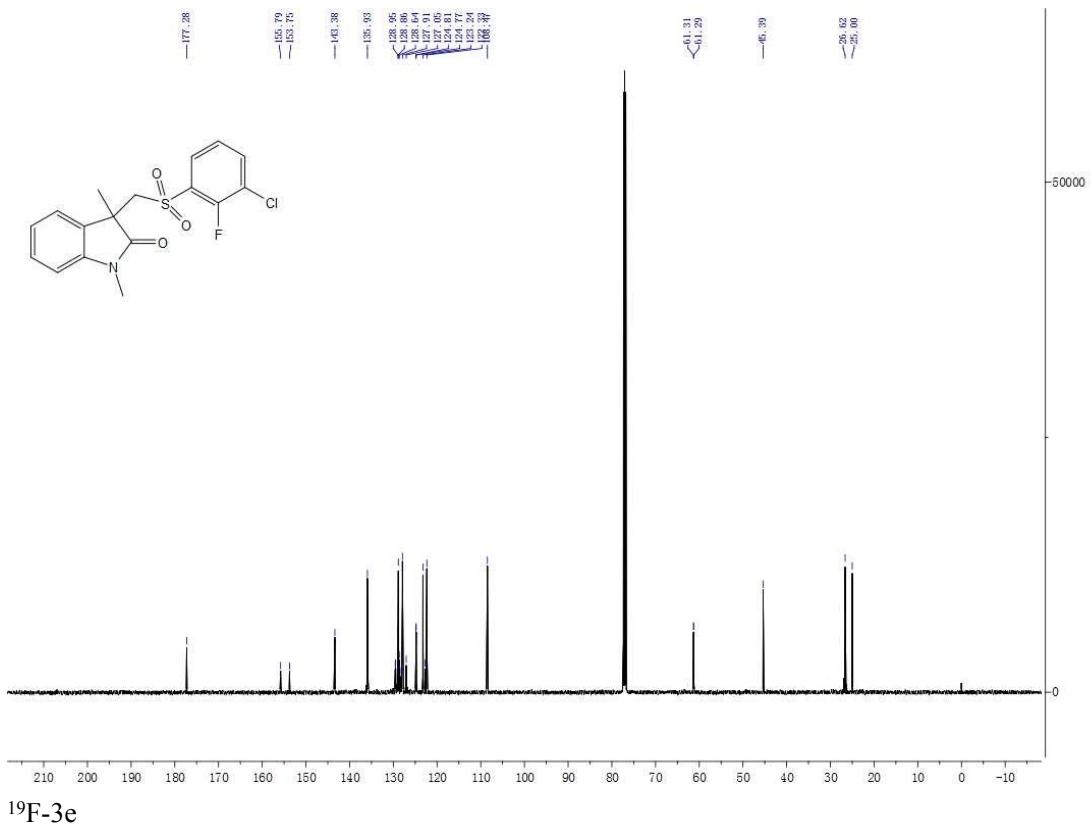
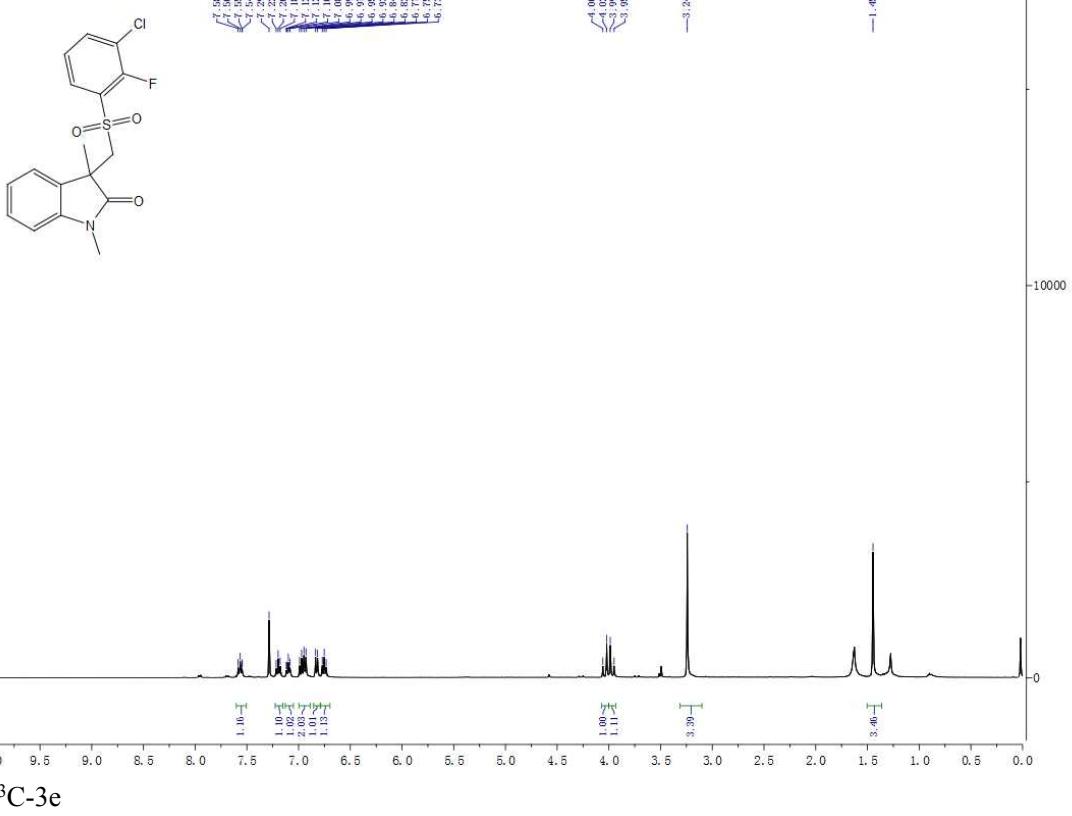


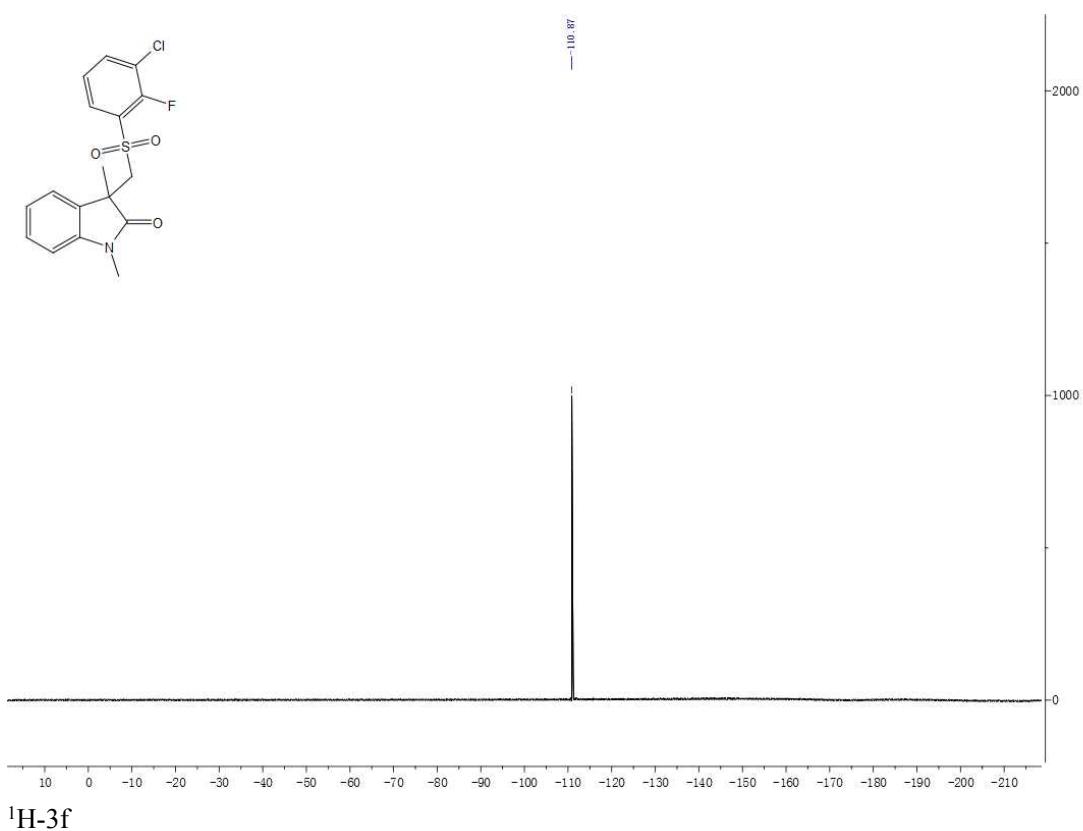
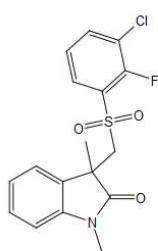


¹⁹F-3d

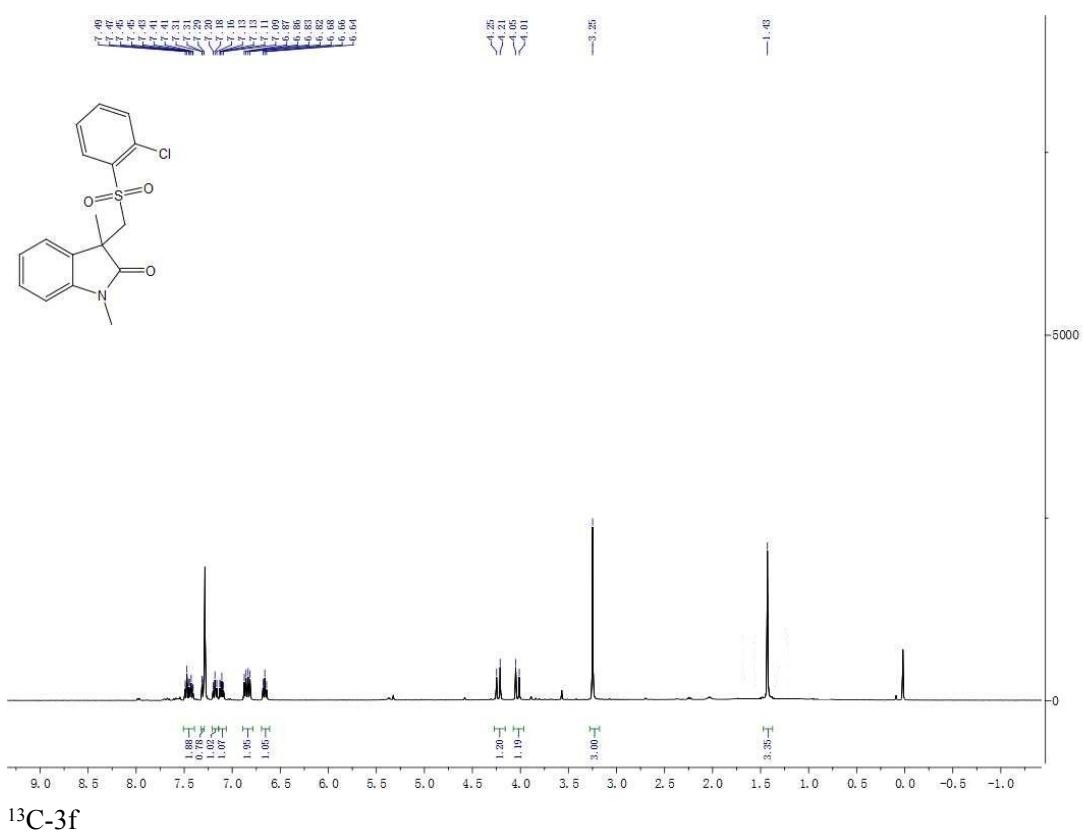
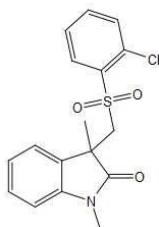


¹H-3e

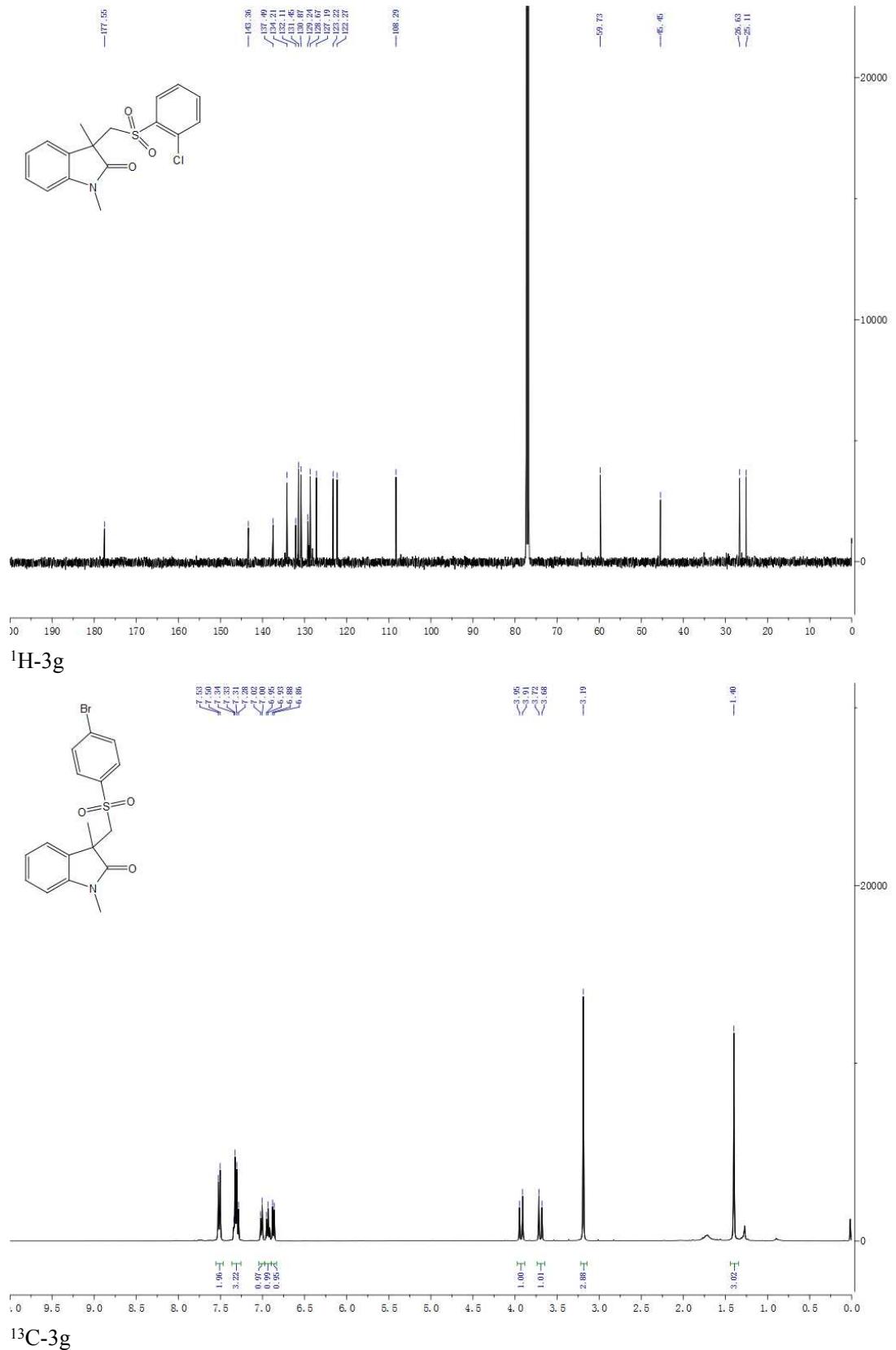


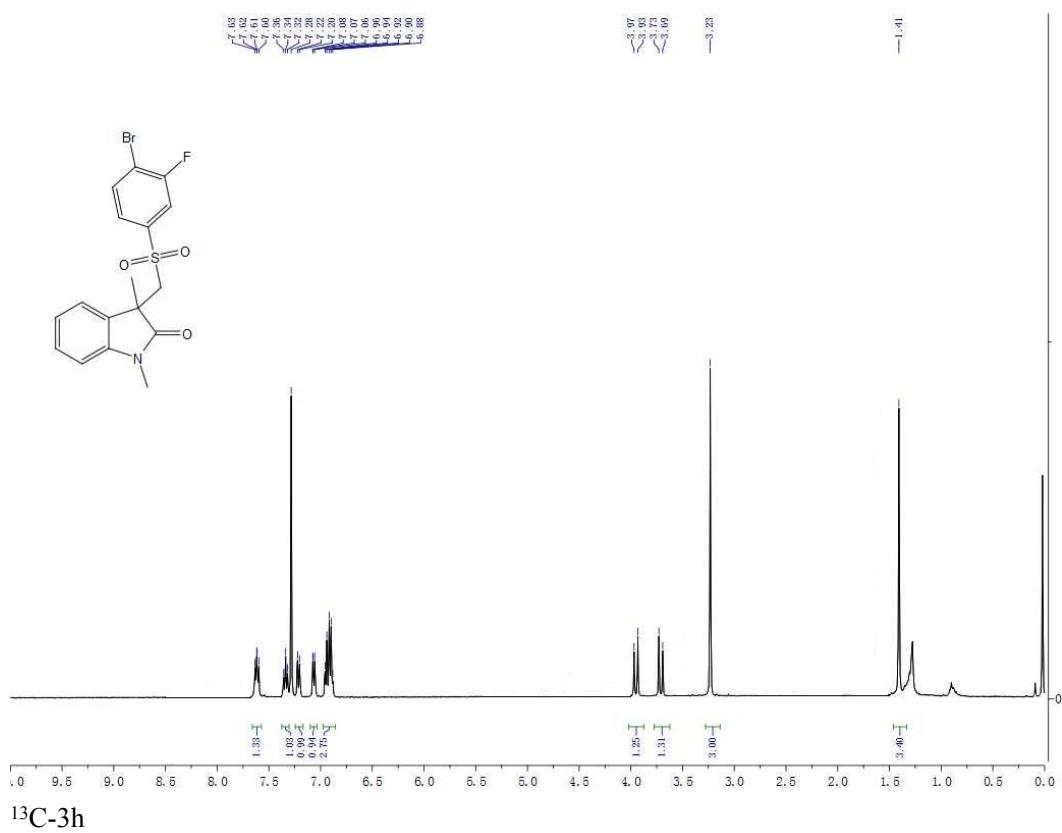
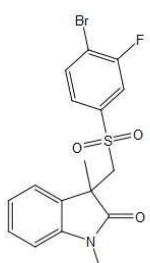
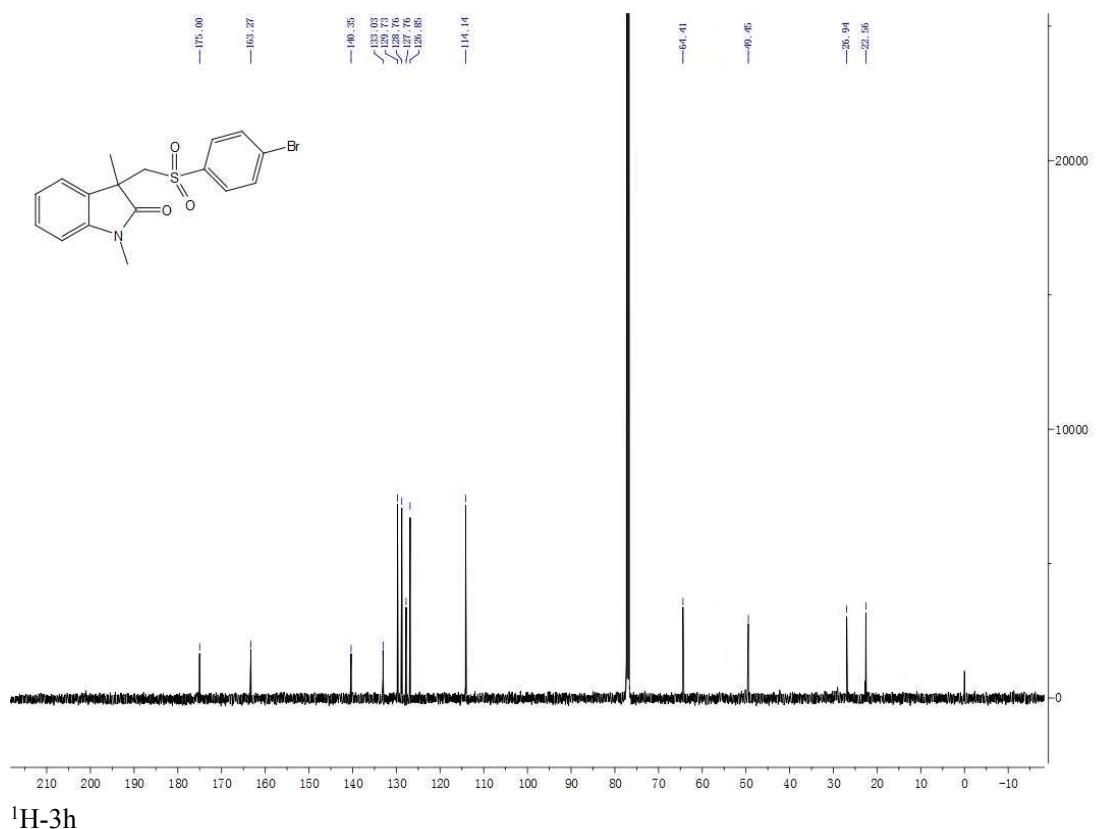


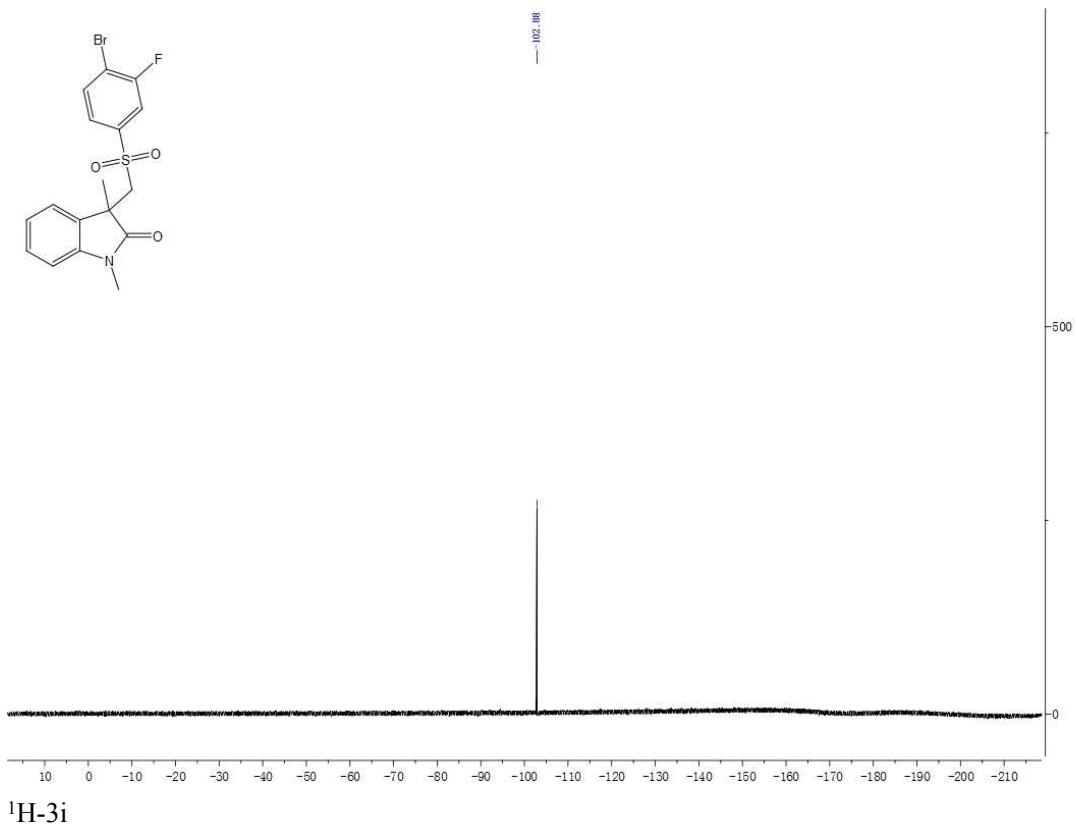
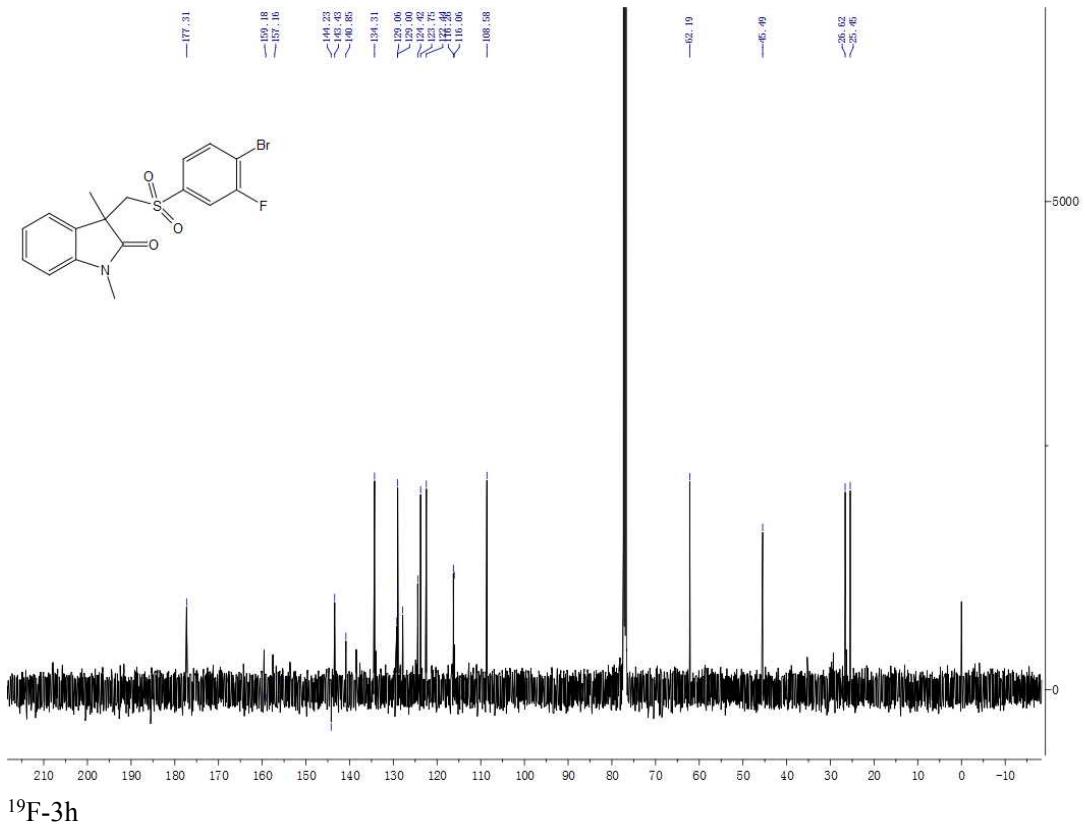
¹H-3f

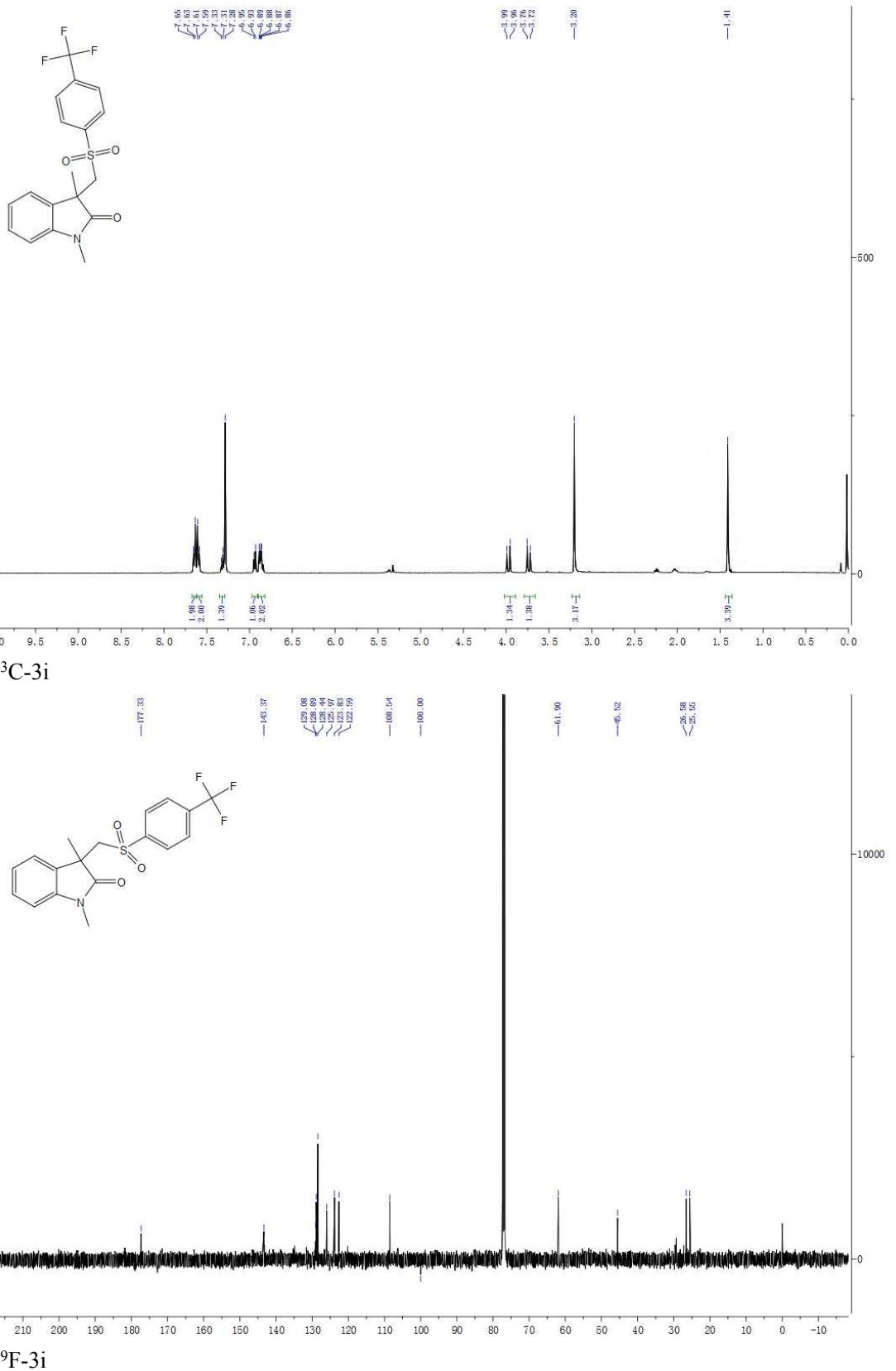


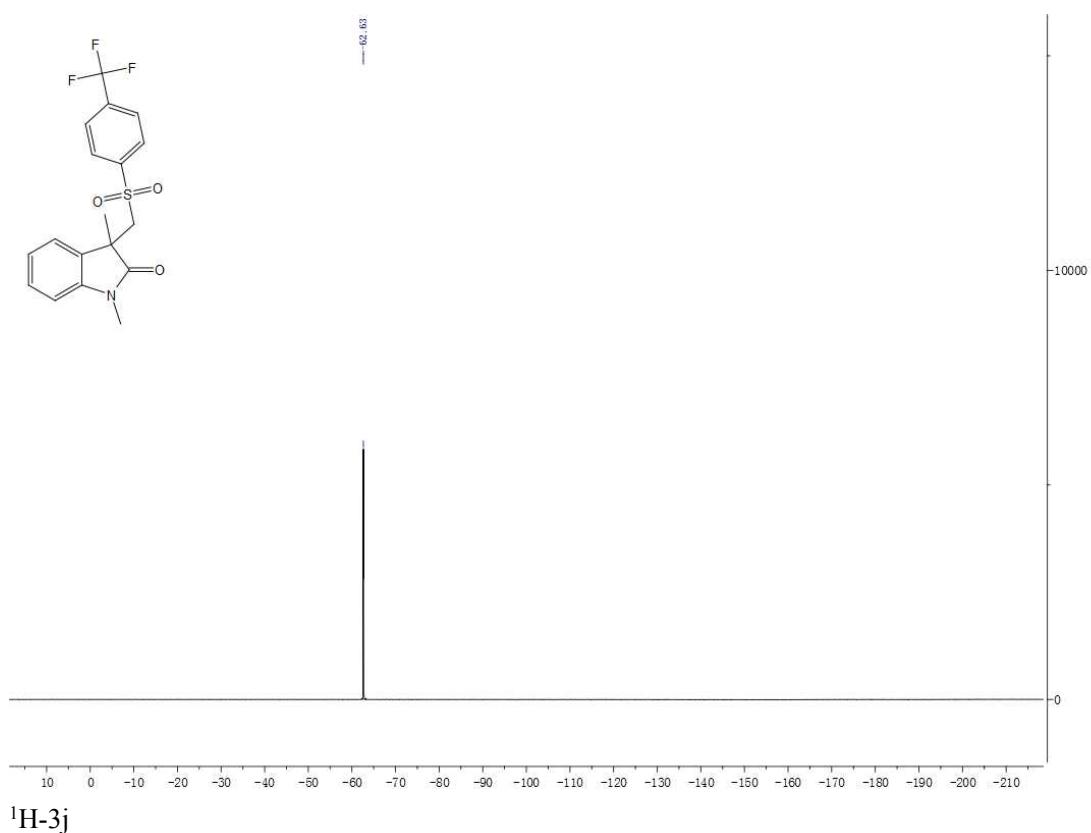
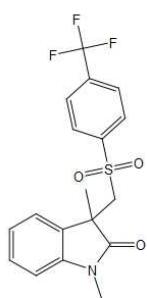
13C-3f



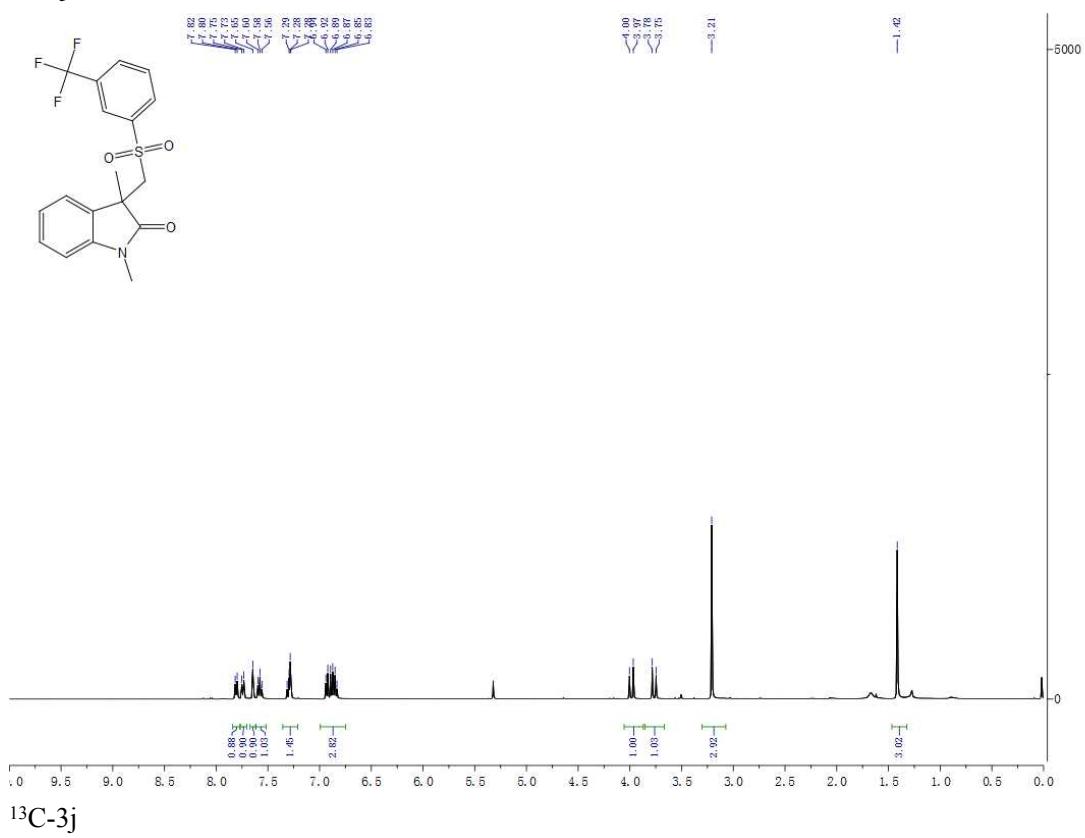
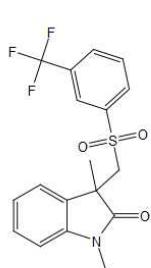




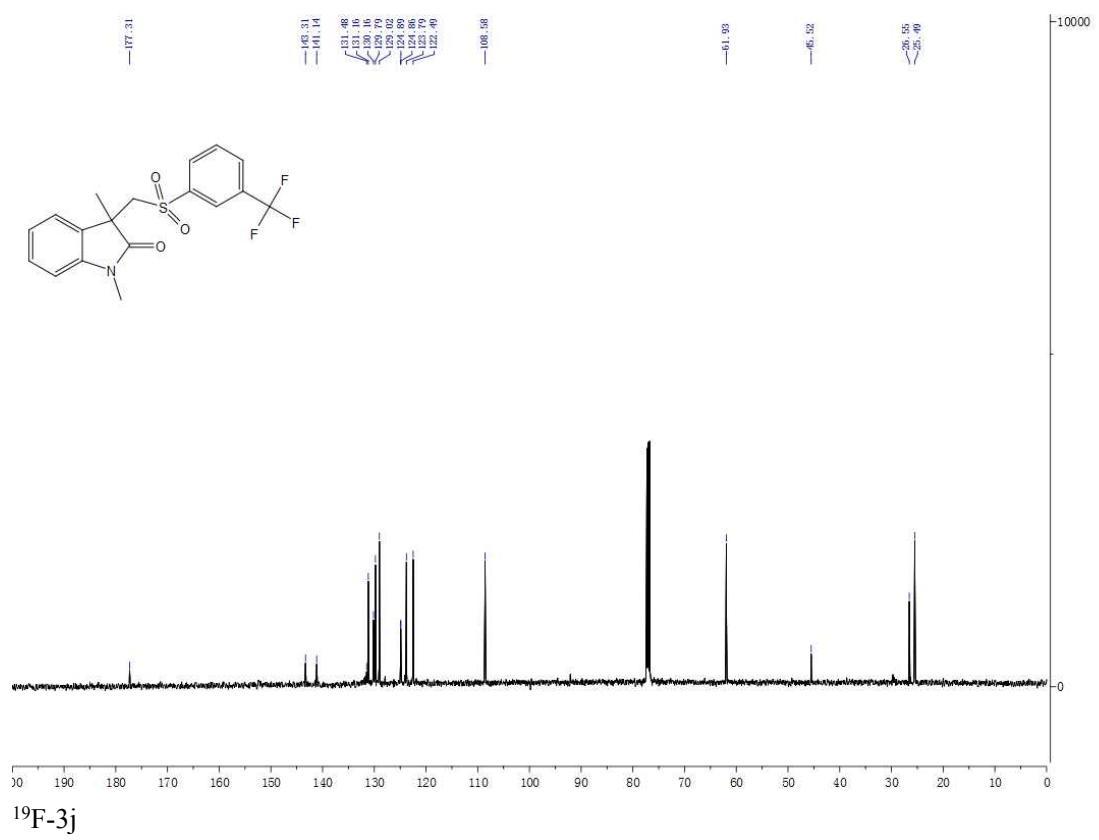




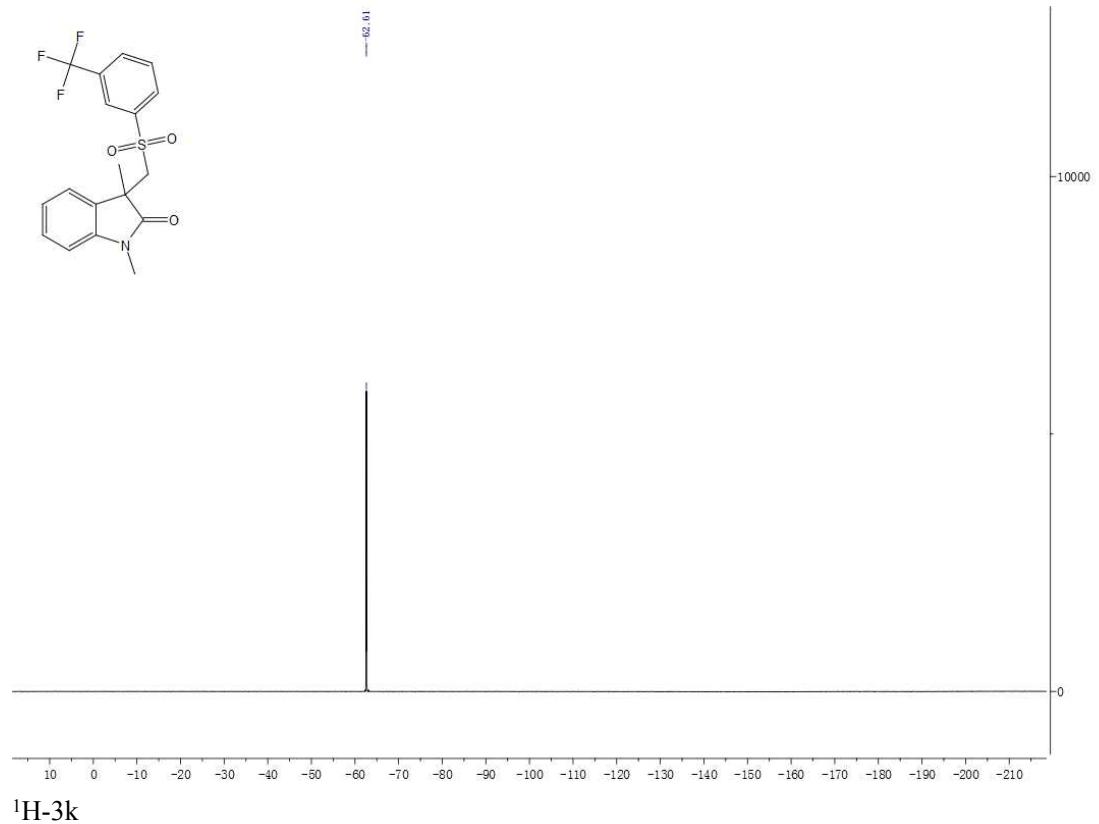
¹H-3j



13C-3j



¹⁹F-3j



¹H-3k

