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Supporting Information

N-Heterocyclic nitrenium catalyzed photosynthesis of 3,3-disubstituted oxindoles from α-chloroanilides

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Contents

1. General Experimental Details.

Chemicals were purchased from Heowns, Innochem and Bidepharm. They were used without further purification unless otherwise noted. The α-chloroanilides were prepared according to the known literatures.[1-6] Solvents were purified using a solvent-purification system (VSPS-8, Vigor) that contained activated alumina and molecular sieves.

Chromatographic purification of the products was performed on Mietek 200-300 mesh silica gel.

High-resolution mass spectra (HRMS) were obtained with the mass analyzer of an orbitrap. The calculated values are based on the most abundant isotope.

IR spectra were taken on a Vertex 70 spectrophotometer and reported as wave numbers (cm^{-1}).

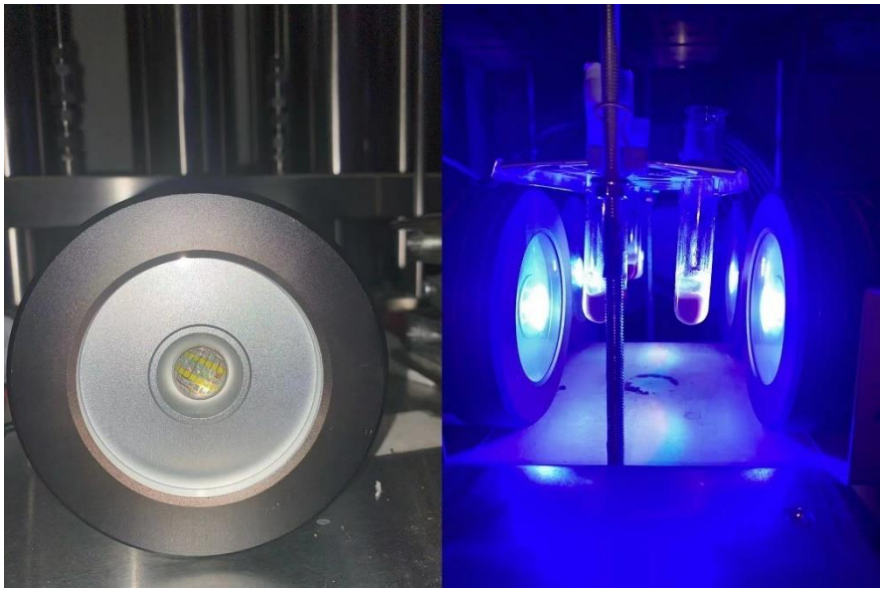
The GC-MS TQ8040 was used in the detection of the reaction mixture.

UV-vis absorption spectra were acquired on UV-2600 spectrophotometer (Shimadzu, Japan).

^{1}H, ^{19}F and ^{13}C- NMR spectra were recorded at ambient temperature on a JEOL JNM-LA400 Spectrometer and JEOL JNM-ECZ500R Spectrometer. The chemical shifts are reported in ppm downfield of tetramethylsilane (TMS) and referenced to residual solvent peaks resonance as the internal standard. The order of citation in parentheses is a) multiplicity (s = singlet, d = doublet, t = triplet, dd= doublet of doublet, td = triplet of doublet, m = multiplet), b) coupling constants, c) number of protons. Coupling constants (J) are reported in Hertz (Hz).

HRMS were obtained on an IonSpec FT-ICR mass spectrometer with ESI resources. The mass analysis mode of the HRMS was orbitrap.

Photochemical experiments were performed magnetically stirred in 10 mL glass tubes, sealed with a rubber septum. The tubes were irradiated with blue light (450 nm) using a LED lamp with a power output of 100 W. The distance from the light source to the irradiation vessel is 2 cm, keeping the reaction temperature at 75±5 °C. (The purchase link of LED lamp is:https://item.taobao.com/item.htm?spm=a1z10.5-c-s.w4002-21207510047.14.dbef5298YBVk03&id=522759747619).



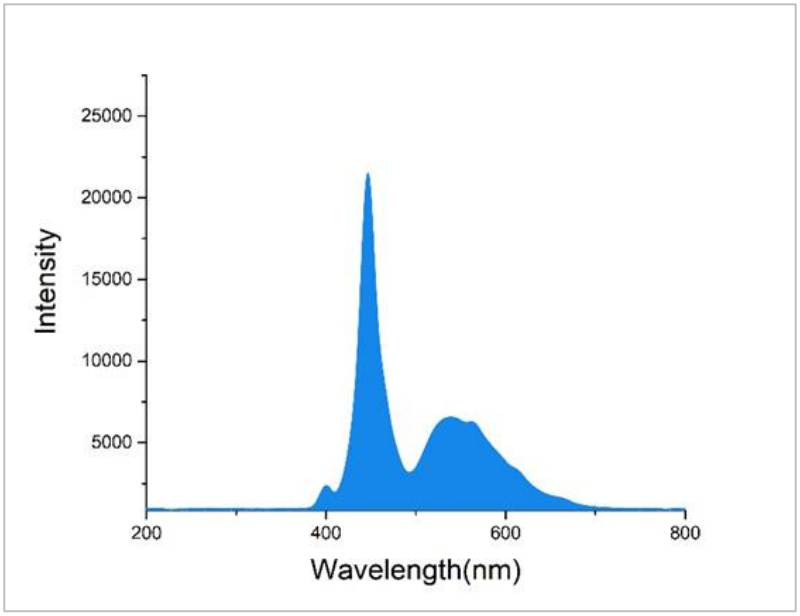
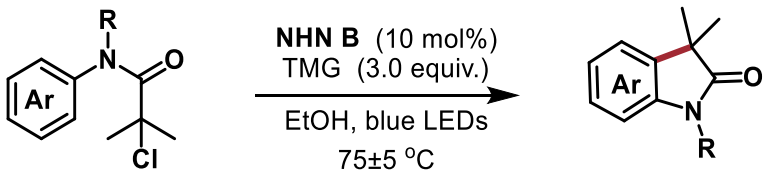


Figure S1. The spectrum of blue LEDs employed in the reaction.

2. Experimental procedures



In a nitrogen-filled glovebox, a dry tube equipped with a magnetic stirring bar was charged sequentially with substrate (0.20 mmol, 1.0 equiv.), TMG (0.60 mmol, 3.0 equiv.), NHN **B** (0.01 mmol, 10% mol) and EtOH (1.0 mL). The tube was closed and removed from the glovebox. The resulting mixture was stirred at 75±5 °C under blue LED (100 W) irradiation for 12 hours. Upon completion, the solvent was removed under vacuum and the residue was subjected to silica gel chromatography using petroleum ether and ethyl acetate as eluent to afford the desired products **2-23**.

3. Mechanism studies

3.1 UV-vis studies

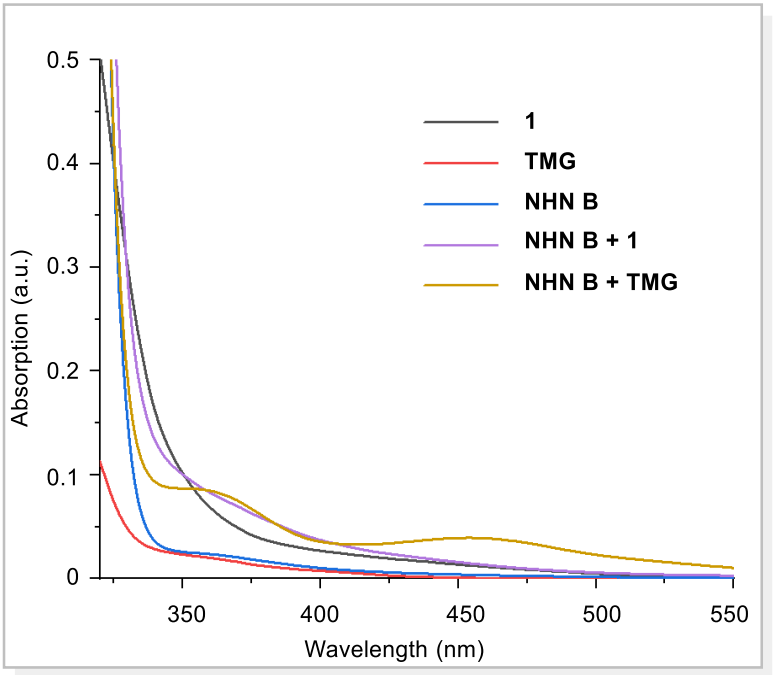
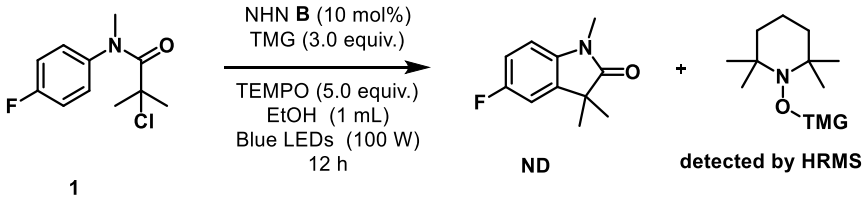
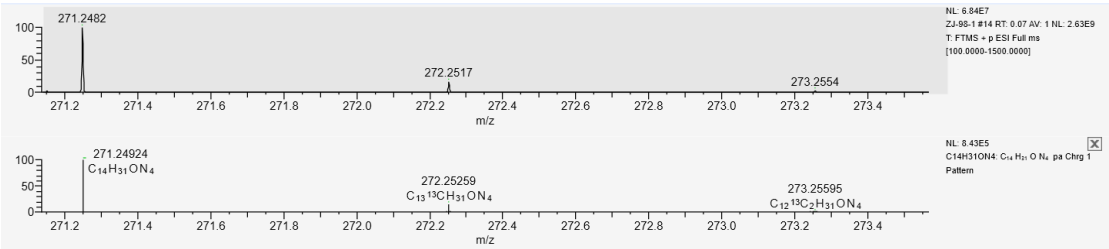


Figure S2. Absorption spectra of substrate **1**, TMG, NHN **B** and their mixture. The UV/vis spectra of **1** (0.2 M in EtOH), TMG (0.6 M in EtOH), NHN **B** (0.02 M in EtOH), and their mixtures.

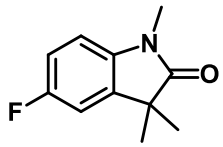
3.2 Radical trapping expriment



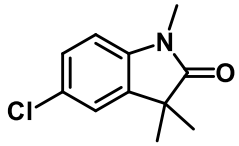
In a nitrogen-filled glovebox, a dry tube equipped with a magnetic stirring bar was charged sequentially with **1** (0.20 mmol, 1.0 equiv.), NHN **B** (0.01 mmol, 10%mol), TEMPO (1.0 mmol, 5.0 equiv.) and EtOH (1.0 mL). The tube was closed and removed from the glovebox. The resulting mixture was stirred at 75±5 °C under blue LED (100 W) irradiation for 12 hours. The adduct of TEMPO and Tetramethylguanidine radical was detected by HRMS. **HRMS** (ESI): m/z $[M+Na]^+$ calcd for $C_{14}H_{31}ON_4Na^+$: 271.2492; found: 271.2482.



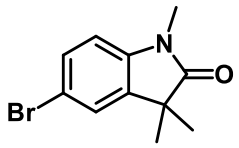
4. Compound Characterization Data.



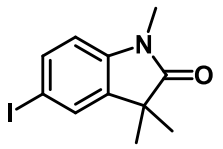
5-fluoro-1,3,3-trimethylindolin-2-one (2): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a light brown solid (35.9 mg, 0.186 mmol, 93%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.99 – 6.93 (m, 2H), 6.79 – 6.74 (m, 1H), 3.21 (s, 3H), 1.37 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 181.1, 159.5 (d, *J* = 240.3 Hz), 138.6, 137.6 (d, *J* = 7.9 Hz), 113.9 (d, *J* = 23.5 Hz), 110.6 (d, *J* = 24.5 Hz), 108.5 (d, *J* = 8.1 Hz), 44.8, 26.5, 24.4. These data are in agreement with those reported previously in the literature.[7]



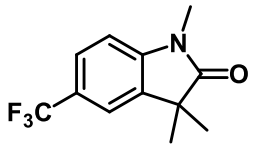
5-chloro-1,3,3-trimethylindolin-2-one (3): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 50:1) as a brownness solid (20.1 mg, 0.096 mmol, 48%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.24 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.18 (d, *J* = 2.1 Hz, 1H), 6.76 (d, *J* = 8.2 Hz, 1H), 3.20 (s, 3H), 1.37 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 181.0, 141.3, 137.6, 128.0, 127.7, 123.1, 109.1, 44.6, 26.5, 24.4.These data are in agreement with those reported previously in the literature.[8]



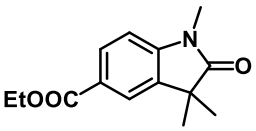
5-bromo-1,3,3-trimethylindolin-2-one (4)： Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a colorless solid (31.4 mg, 0.124 mmol, 64%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 (dd, *J* = 8.2, 2.0 Hz, 1H), 7.31 (d, *J* = 2.0 Hz, 1H), 6.72 (d, *J* = 8.2 Hz, 1H), 3.20 (s, 3H), 1.37 (s, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 180.8, 141.9, 138.1, 130.6, 125.8, 115.3, 109.6, 44.6, 26.4, 24.4. These data are in agreement with those reported previously in the literature.[7]



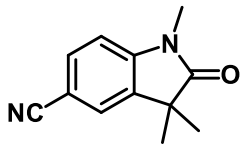
5-iodo-1,3,3-trimethylindolin-2-one （5）： Following the general procedure, general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a faint yellow solid (19.3 mg, 0.064 mmol, 32%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70 – 7.43 (m, 2H), 6.63 (d, *J* = 8.1 Hz, 1H), 3.19 (s, 3H), 1.36 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 180.9, 141.8, 138.0, 130.6, 125.8, 115.3, 109.6, 44.6, 26.4, 24.4. These data are in agreement with those reported previously in the literature.[9]



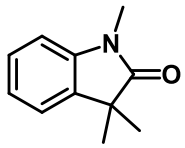
1,3,3-trimethyl-5-(trifluoromethyl)indolin-2-one (6): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a faint yellow oil (26.2 mg, 0.108 mmol, 54%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.57 – 7.53 (m, 1H), 7.44 – 7.42 (m, 1H), 6.91 (d, J = 8.2 Hz, 1H), 3.25 (s, 3H), 1.40 (s, 6H). **^{13}C NMR**(101 MHz, $CDCl_3$) δ 181.4, 145.8, 136.5, 125.7 (d, J = 4.0 Hz), 124.0 (d, J = 148.9 Hz), 119.5 (d, J = 3.8 Hz), 107.9, 44.3, 26.5, 24.4. **^{19}F NMR** (376 MHz, CDCl3) δ -61.26. These data are in agreement with those reported previously in the literature.[7]



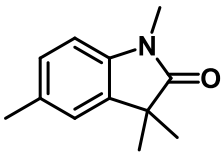
methyl 1,3,3-trimethyl-2-oxoindoline-5-carboxylatez (7): Following the general procedure, general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a faint yellow solid (34.58 mg, 0.140 mmol, 70%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.88 (d, *J* = 1.7 Hz, 1H), 6.88 (d, *J* = 8.2 Hz, 1H), 4.38 (q, *J* = 7.1 Hz, 2H), 3.25 (s, 3H), 1.44 – 1.38 (m, 9H). **^{13}C NMR** (101 MHz, CDCl3) δ 181.8, 166.7, 146.9, 135.8, 130.6, 125.0, 123.7, 107.6, 61.0, 44.2, 26.6, 24.4, 14.6.These data are in agreement with those reported previously in the literature.[10]



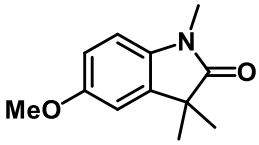
1,3,3-trimethyl-2-oxoindoline-5-carbonitrile (8): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a white solid (15.2 mg, 0.076 mmol, 38%) **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.45 (d, *J* = 1.6 Hz, 1H), 6.91 (d, *J* = 8.1 Hz, 1H), 3.25 (s, 3H), 1.39 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 181.1, 146.7, 136.9, 133.3, 125.9, 119.4, 108.6, 105.7, 44.2, 26.6, 24.3.These data are in agreement with those reported previously in the literature.[10]



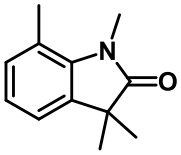
1,3,3-trimethylindolin-2-one (9): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow oil (26,3 mg, 0.15 mmol, 75%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 – 7.16 (m, 2H), 7.09 – 7.04 (m, 1H), 6.85 (d, *J* = 7.7, 0.9 Hz, 1H), 3.22 (s, 3H), 1.37 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 181.5, 142.7, 135.9, 127.8, 122.6, 122.3, 108.1, 44.3, 26.3, 24.5.These data are in agreement with those reported previously in the literature.[7]



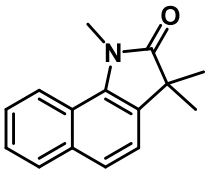
1,3,3,5-tetramethylindolin-2-one (10): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow solid (30.2 mg, 0.160mmol, 80%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.12 – 6.99 (m, 2H), 6.74 (d, *J* = 7.8 Hz, 1H), 3.20 (s, 3H), 2.35 (s, 3H), 1.36 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$)δ 181.5, 140.4, 136.0, 132.1, 128.0, 123.3, 107.9, 44.4, 26.4, 24.5, 21.3.These data are in agreement with those reported previously in the literature.[7]



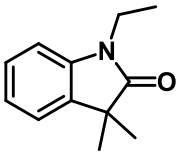
5-methoxy-1,3,3-trimethylindolin-2-one (11): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow oil (31.2 mg, 0.152 mmol, 76%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.83 (d, *J* = 2.4 Hz, 1H), 6.81 – 6.72 (m, 2H), 3.81 (s, 3H), 3.19 (s, 3H), 1.36 (s, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 181.2, 156.2, 137.4, 136.3, 111.7, 110.2, 108.4, 56.06, 44.8, 26.4, 24.5.These data are in agreement with those reported previously in the literature.[7]



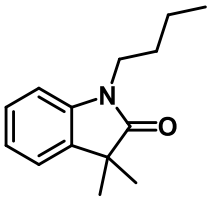
1,3,3,7-tetramethylindolin-2-one (12): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow solid (23.4 mg, 0.104 mmol, 52%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.07 – 7.02 (m, 1H), 7.01 – 6.91 (m, 2H), 3.50 (s, 3H), 2.59 (s, 3H), 1.35 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 182.2, 140.5, 136.6, 131.5, 122.5, 120.3, 119.8, 43.6, 29.6, 24.9, 19.2.These data are in agreement with those reported previously in the literature.[11]



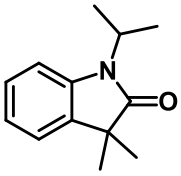
1,3,3-trimethyl-1,3-dihydro-2H-benzo[g]indol-2-one (13): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a faint yellow solid (38.3 mg, 0.170 mmol, 85%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.72 (d, J = 8.0 Hz, 1H), 7.52 (d, J = 7.8 Hz, 2H), 7.45 (d, *J* = 8.0 Hz, 2H), 6.96 (d, *J* = 7.6 Hz, 1H), 3.54 (s, 3H), 1.68 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 174.3, 140.1, 137.1, 133.5, 127.2, 126.5, 126.1, 122.5, 122.5, 119.0, 108.5, 43.5, 30.7, 29.9.These data are in agreement with those reported previously in the literature.[12]



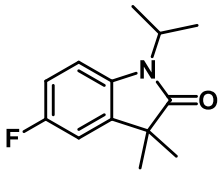
1-ethyl-3,3-dimethylindolin-2-one (14): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow oil (23.4 mg, 0.124 mmol, 62%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29 – 7.19 (m, 2H), 7.10 – 7.02 (m, 1H), 6.89 – 6.84 (m, 1H), 3.81 – 3.73 (m, 2H), 1.36 (s, 6H), 1.26 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 181.1, 141.8, 136.2, 127.7, 122.6, 122.4, 108.3, 44.2, 34.7, 24.5, 12.9. These data are in agreement with those reported previously in the literature.[11]



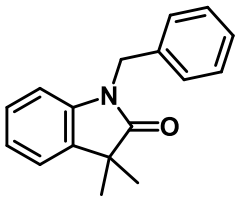
1-butyl-3,3-dimethylindolin-2-one (15): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a faint yellow oil (35.6 mg, 0.164 mmol, 82%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27 – 7.19 (m, 2H), 7.05 (d, 1H), 6.86 (d, *J* = 7.7, 0.8 Hz, 1H), 3.71 (t, *J* = 7.3 Hz, 2H), 1.71 – 1.62 (m, 2H), 1.36 (s, 8H), 0.95 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 181.4, 142.2, 136.2, 127.7, 122.5, 122.3, 108.5, 44.2, 39.7, 29.6, 24.6, 20.2, 13.9.These data are in agreement with those reported previously in the literature.[13]



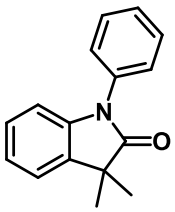
1- isopropyl-3,3-dimethylindolin-2-one (16): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow oil (34.9 mg, 0.172 mmol, 86%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.28 – 7.19 (m, 2H), 7.06 – 7.00 (m, 2H), 4.71 – 4.62 (m, 1H), 1.49 (s, 3H), 1.47 (s, 3H), 1.35 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 181.2, 141.3, 136.5, 127.4, 122.6, 122.0, 110.0, 44.0, 43.5, 24.6, 19.5. These data are in agreement with those reported previously in the literature.[11]



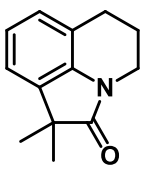
5-fluoro-1-isopropyl-3,3-dimethylindolin-2-one (17): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow solid (34.5 mg, 0.156 mmol, 78%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.96 – 6.90 (m, 3H), 4.72 – 4.59 (m, 1H), 1.47 (s, 3H), 1.45 (s, 3H), 1.34 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 180.8, 159.08 (d, *J* = 240.4 Hz), 138.4 (d, *J* = 7.6 Hz), 137.1, 113.5 (d, *J* = 23.1 Hz), 110.8 (d, *J* = 24.2 Hz), 110.4 (d, *J* = 7.8 Hz), 44.4, 43.6, 24.5, 19.5.**^{19}F NMR** (376 MHz, $CDCl_3$) δ -121.58. These data are in agreement with those reported previously in the literature.[7]



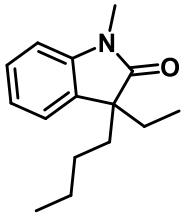
benzyl-3,3-dimethylindolin-2-one (18): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a faint yellow solid (42 mg, 0.166 mmol, 83%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 – 7.26 (m, 4H), 7.25 – 7.19 (m, 2H), 7.16 – 7.11 (m, 1H), 7.05 – 6.99 (m, 1H), 6.72 (d, *J* = 7.8, 0.7 Hz, 1H), 4.92 (s, 2H), 1.44 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 181.6, 141.8, 136.2, 135.9, 128.9, 127.7, 127.7, 127.3, 122.6, 122.5, 109.2, 44.3, 43.6, 24.7. These data are in agreement with those reported previously in the literature.[12]



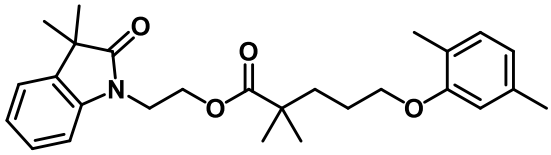
3,3-dimethyl-1-phenylindolin-2-one (19): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a faint yellow solid (19.0 mg, 0.08 mmol, 40%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 – 7.49 (m, 2H), 7.45 – 7.37 (m, 3H), 7.28 (d, J = 7.3, 1.2 Hz, 1H), 7.13 – 7.06 (m, 1H), 6.85 (d, *J* = 7.8 Hz, 1H), 1.49 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 180.9, 142.6, 135.8, 134.8, 129.7, 128.0, 127.7, 126.7, 123.1, 122.8, 109.5, 44.5, 24.9. These data are in agreement with those reported previously in the literature.[12]



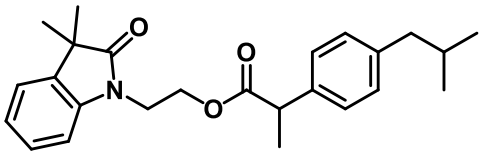
1,1-dimethyl-5,6-dihydro-4H-pyrrolo[3,2,1-ij]quinolin-2(1H)-one (20): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow oil (35.8 mg, 0.178 mmol, 89%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.06 – 7.00 (m, 2H), 6.98 – 6.90 (m, 1H), 3.74 – 3.70 (m, 2H), 2.82 – 2.76 (m, 2H), 2.06 – 1.98 (m, 2H), 1.38 (s, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 180.4, 138.6, 134.5, 126.5, 122.0, 120.2, 45.6, 38.9, 24.7, 24.3, 21.3. These data are in agreement with those reported previously in the literature.[12]



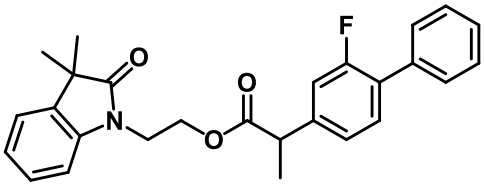
3-butyl-3-ethyl-1-methylindolin-2-one (21) : Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a yellow oil (21.3 mg, 0.092 mmol, 46%).**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 1H), 7.17 – 7.05 (m, 2H), 6.84 (d, *J* = 7.7 Hz, 1H), 3.22 (s, 3H), 1.98 – 1.69 (m, 4H), 1.33 – 0.87 (m, 4H), 0.76 (t, *J* = 7.3 Hz, 3H), 0.55 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 180.4, 144.3, 132.5, 127.6, 122.8, 122.5, 107.8, 53.9, 37.7, 31.1, 26.5, 26.1, 23.0, 14.0, 8.7. These data are in agreement with those reported previously in the literature.[14]



2-(3,3-dimethyl-2-oxoindolin-1-yl)ethyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (22): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 50:1) as a yellow oil (46.3 mg, 0.106 mmol, 53%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27 – 7.22 (m, 1H), 7.19 (d, *J* = 7.3, 1.3 Hz, 1H), 7.08 – 7.01 (m, 1H), 7.01 – 6.97 (m, 1H), 6.93 (d, *J* = 7.8 Hz, 1H), 6.65 (d, 1H), 6.56 (s, 1H), 4.36 – 4.27 (m, 2H), 4.00 – 3.93 (m, 2H), 3.81 – 3.75 (m, 2H), 2.31 (s, 3H), 2.14 (s, 3H), 1.63 – 1.59 (m, 4H), 1.35 (s, 6H), 1.12 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 181. 5, 177.8, 156.98, 141.9, 136.5, 135.9, 130.4, 127.7, 123.6, 122.7, 122.6, 120.8, 112.0, 108.5, 67.8, 61.4, 44.1, 42.1, 38.9, 37.0, 25.1, 24.5, 21.5, 15.9. **IR** (ART): 2968, 2924, 1713, 1612, 1458, 1352, 1263, 1189, 1129 cm^{-1}. **HRMS** (ESI): m/z $[M+H]^+$ calcd for $C_{27}H_{36}O_4N^+$: 438.2639; found: 438.2626.



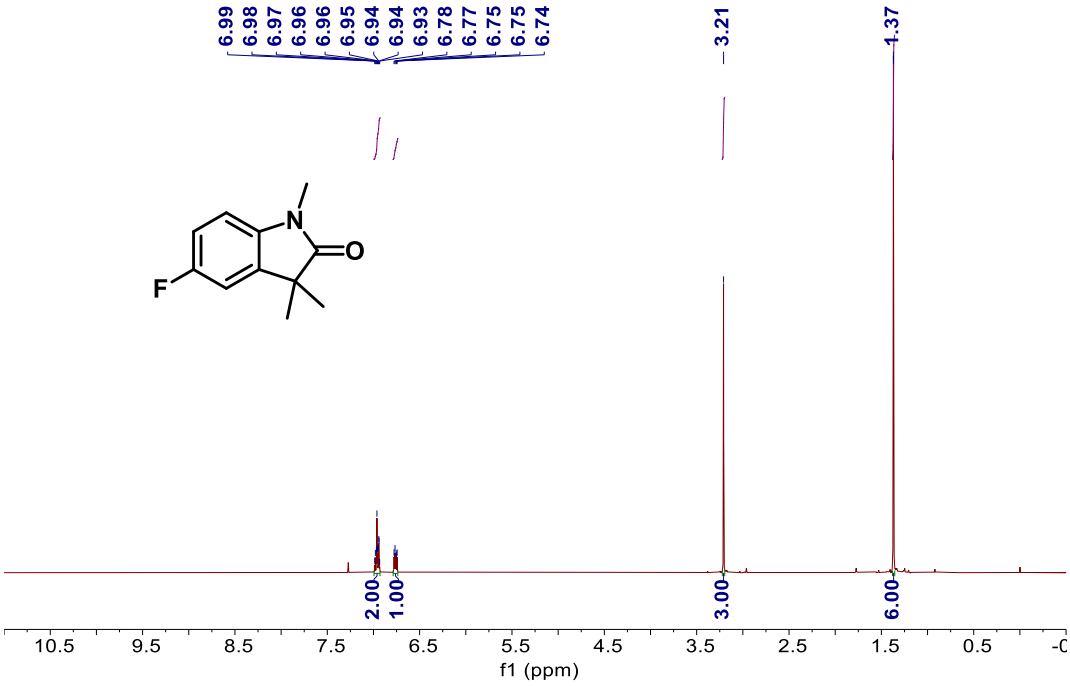
2-(3,3-dimethyl-2-oxoindolin-1-yl)ethyl 2-(4-isobutylphenyl)propanoate (23): Followingthe general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a faint yellow oil (42 mg, 0.068 mmol, 32%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.24 – 7.18 (m, 2H), 7.11 – 6.96 (m, 5H), 6.85 (d, J = 7.8 Hz, 1H), 4.45 – 4.18 (m, 2H), 4.04 – 3.83 (m, 2H), 3.63 – 3.55 (m, 1H), 2.41 (d, J = 7.2 Hz, 2H), 1.92 – 1.75 (m, 1H), 1.39 (d, J = 7.2 Hz, 3H), 1.33 (d, J = 6.3 Hz, 6H), 0.88 (d, J = 6.6 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 181.6, 174.7, 142.0, 140.7, 137.5, 135.9, 129.5, 127.7, 127.3, 122.6, 122.5, 108.5, 61.8, 45.2, 44.1, 38.9, 30.3, 24.5, 22.5, 18.4, 1.2. **IR** (ART): 2967, 2930, 1709, 1612, 1487, 1458, 1383, 1353, 1170, 1074, 926, 761, 697 cm^{-1}. **HRMS** (ESI): m/z $[M+H]^+$ calcd for $C_{27}H_{27}O_3NF^+$: 432.1970; found: 432.1959.



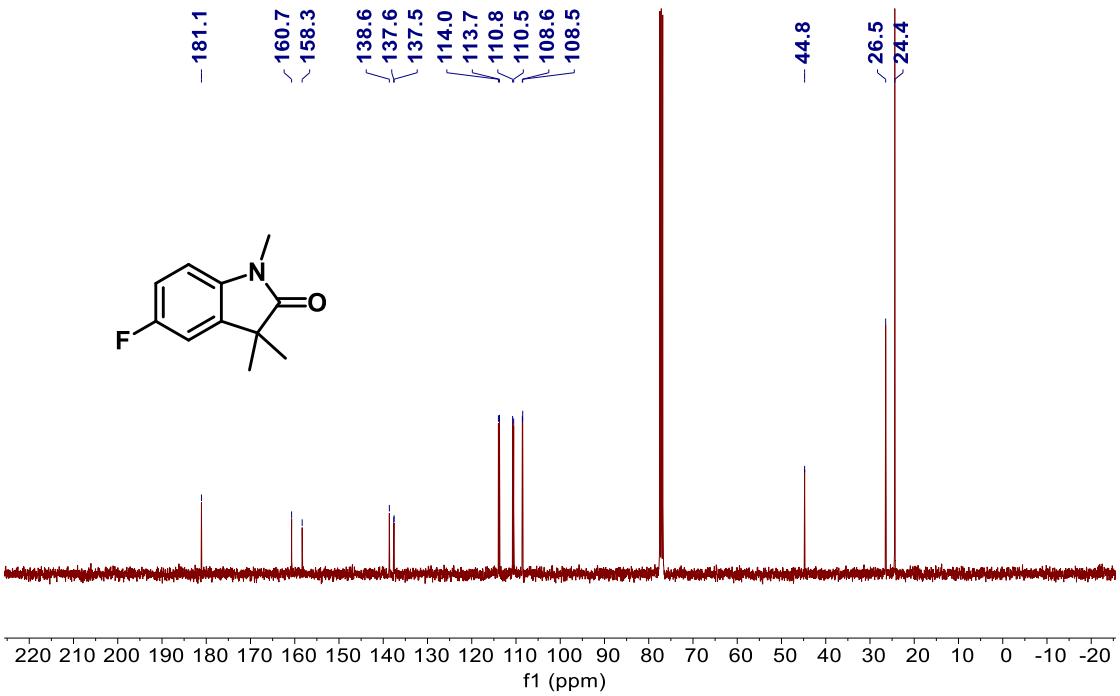
2-(3,3-dimethyl-2-oxoindolin-1-yl)ethyl 2-(2-fluoro-[1,1'-biphenyl]-4-yl)propanoate (24): Following the general procedure, the title product was obtained after purification by column chromatography (PE/EA = 20:1) as a faint yellow oil (29.3 mg, 0.166 mmol, 34%). **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.53 – 7.28 (m, 6H), 7.25 – 7.17 (m, 2H), 7.09 – 6.93 (m, 3H), 6.85 (d, *J* = 7.8 Hz, 1H), 4.47 – 4.26 (m, 2H), 4.07 – 3.88 (m, 2H), 3.70 – 3.60 (m, 1H), 1.44 (d, *J* = 7.2 Hz, 3H), 1.33 (d, *J* = 7.2 Hz, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 181.6, 173.9, 159.7 (d, *J* = 248.2 Hz), 141.9, 141.5 (d, *J* = 7.8 Hz), 135.9, 135.6, 129.1 (d, *J* = 2.9 Hz), 128.6, 127.8 (d, *J* = 15.3 Hz), 123.7 (d, *J* = 3.5 Hz), 122.7 (d, *J* = 12.2 Hz), 115.4 (d, *J* = 23.5 Hz), 108.4, 62.1, 45.1, 44.1, 38.9, 24.5, 18.3. **^{19}F NMR** (376 MHz, $CDCl_3$) δ -117.4. **IR** (ART): 2954, 2867, 1710, 1612, 1459, 1458, 1382, 1352, 1198, 1155, 1120, 1072, 1019, 944, 848, 755, 696 cm^{-1}. **HRMS** (ESI): m/z $[M+H]^+$ calcd for $C_{25}H_{32}O_3N^+$: 394.2377; found: 394.2365.

5. NMR Spectra.

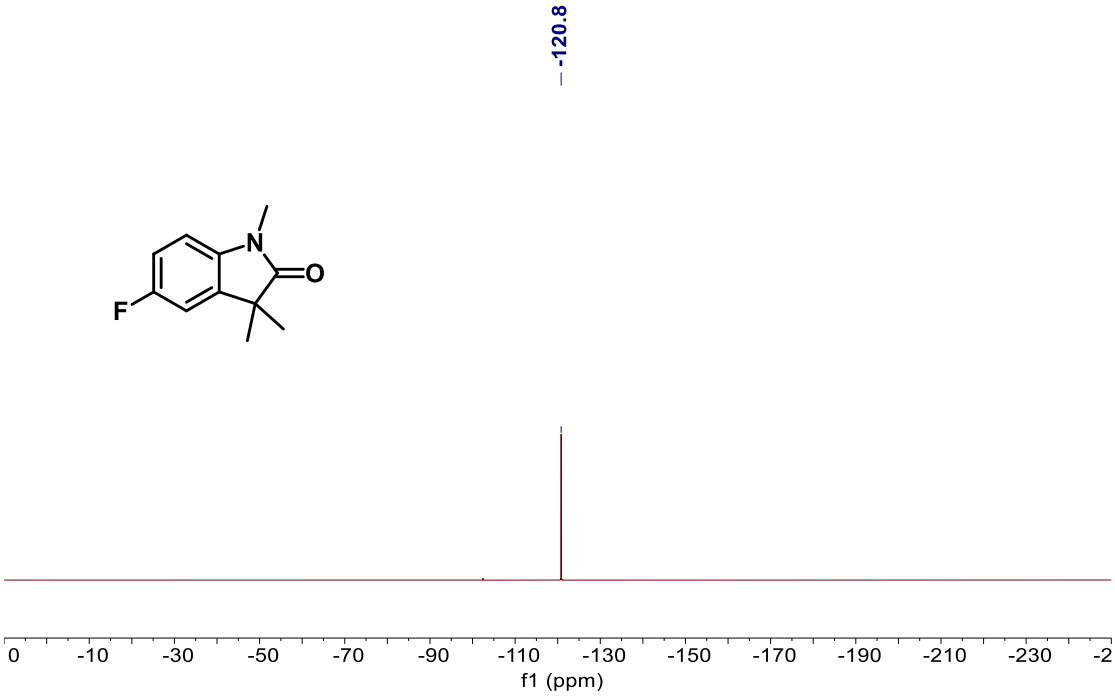
^{1}H NMR of compound **2** (400 MHz in $CDCl_3$)



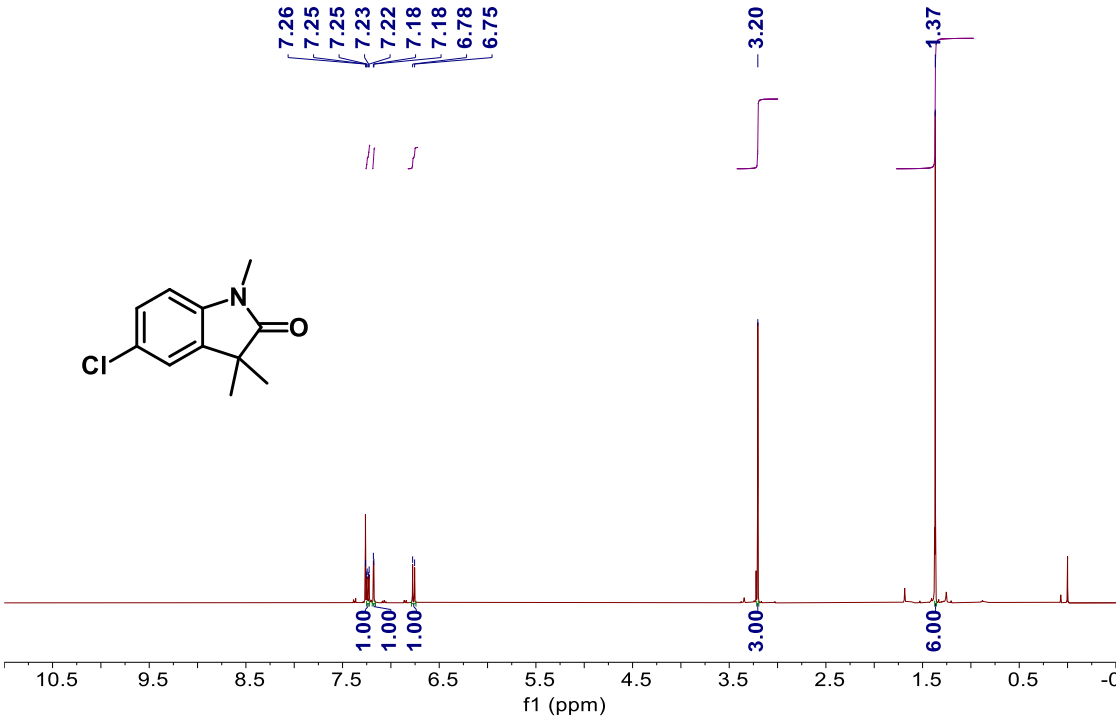
^{13}C NMR of compound **2** (101 MHz in $CDCl_3$)



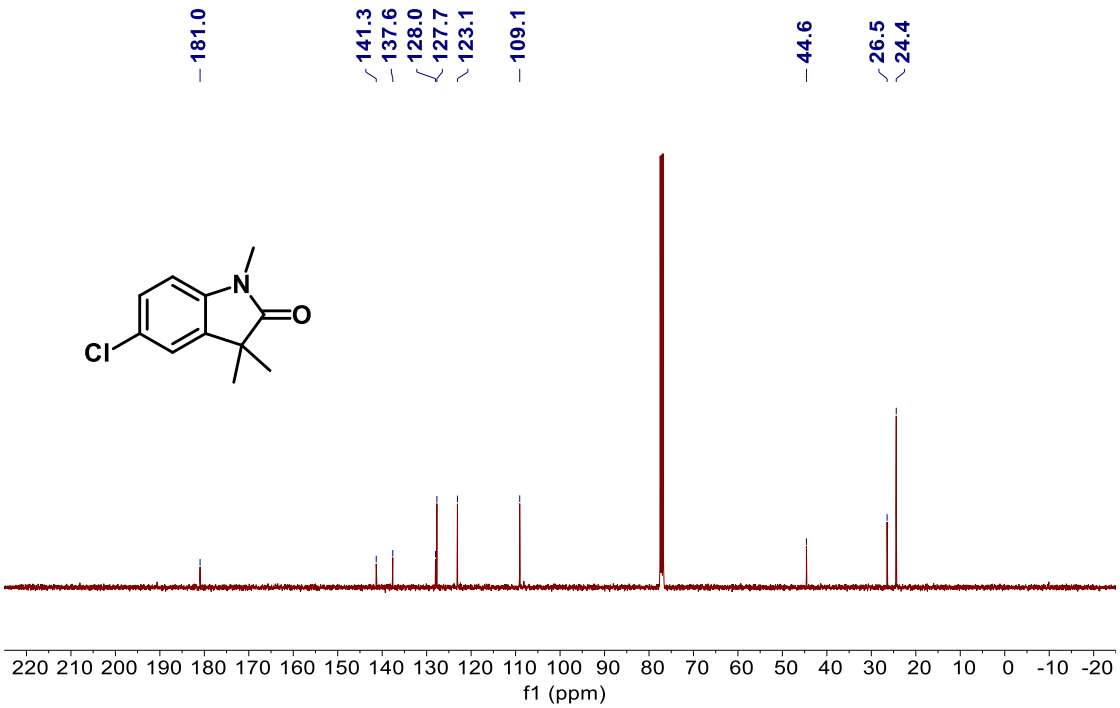
^{19}F NMR of compound **2** (565 MHz in $CDCl_3$)



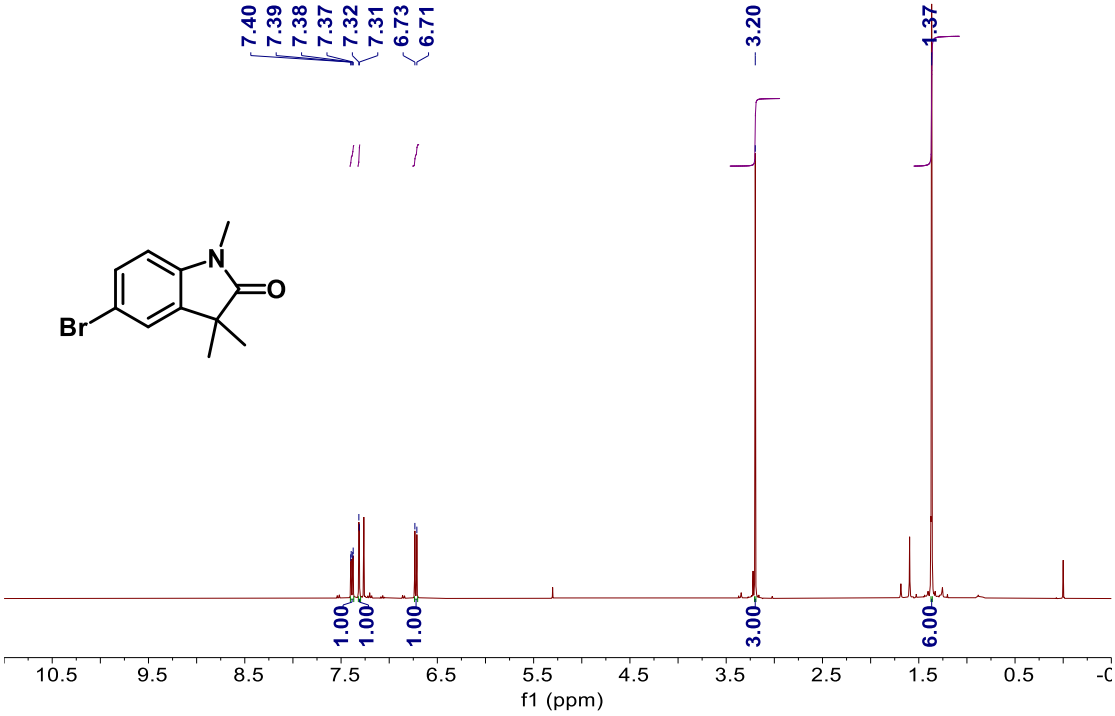
^{1}H NMR of compound **3** (400 MHz in $CDCl_3$)



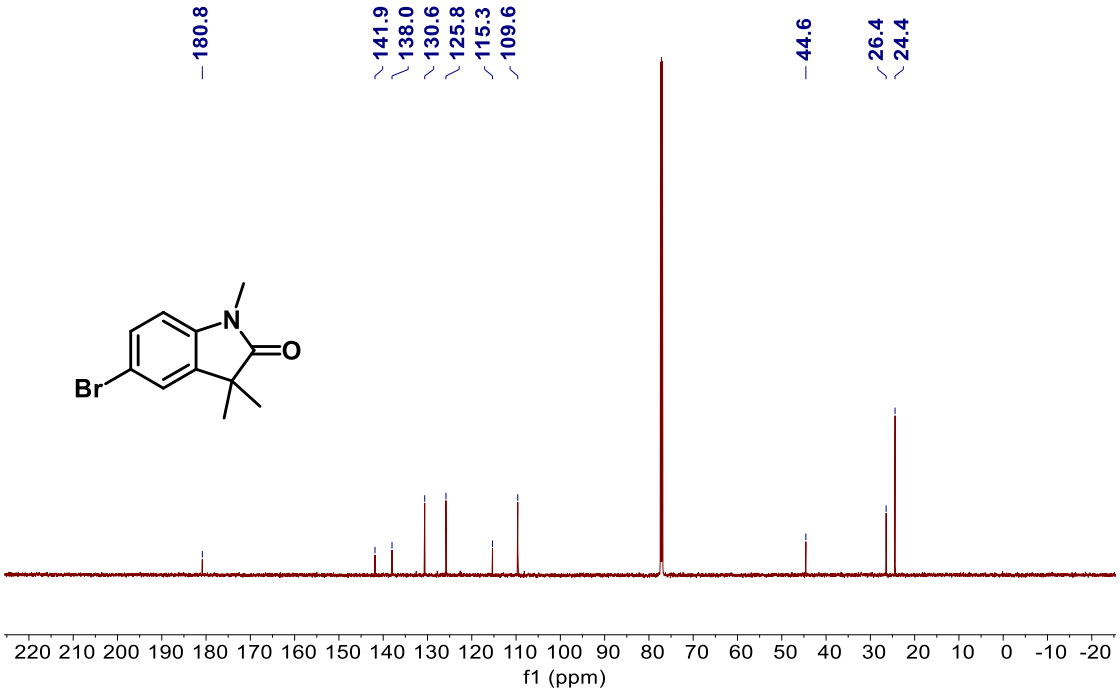
^{13}C NMR of compound **3** (101 MHz in $CDCl_3$)



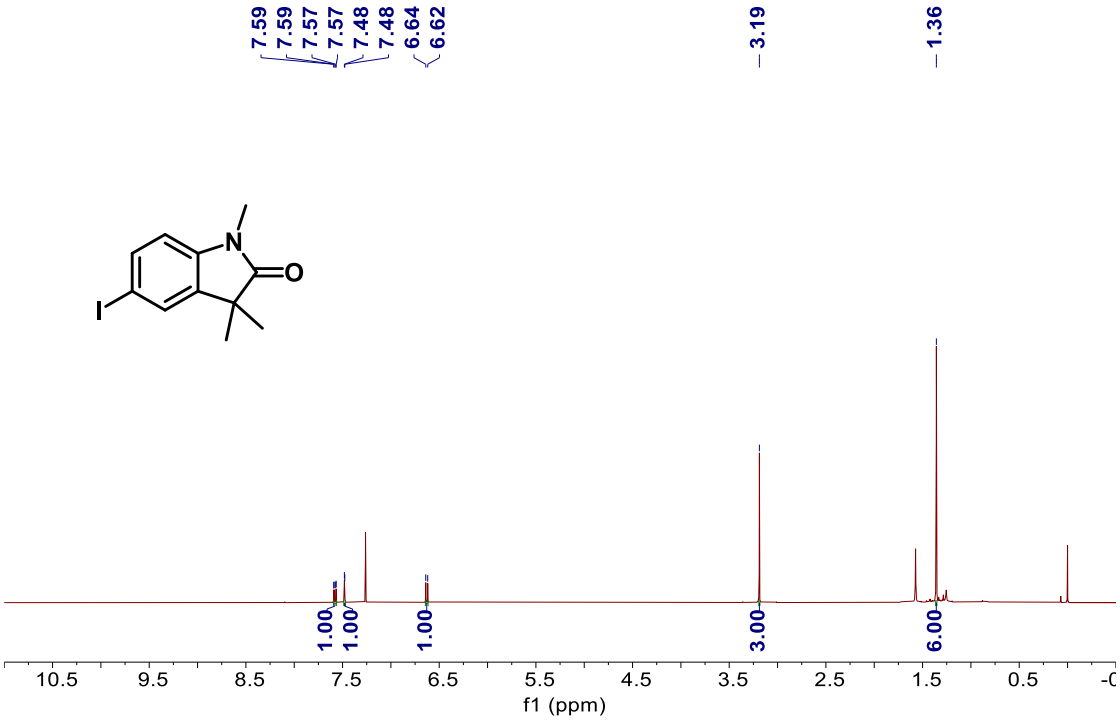
^{1}H NMR of compound **4** (400 MHz in $CDCl_3$)



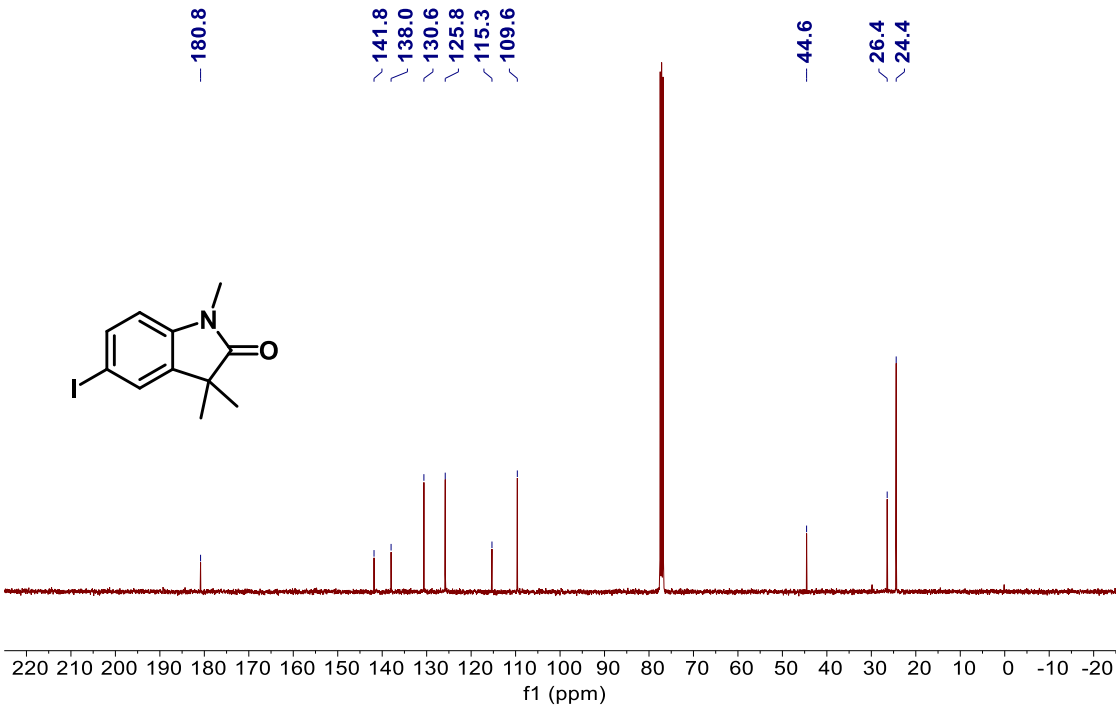
^{13}C NMR of compound **4** (151 MHz in $CDCl_3$)



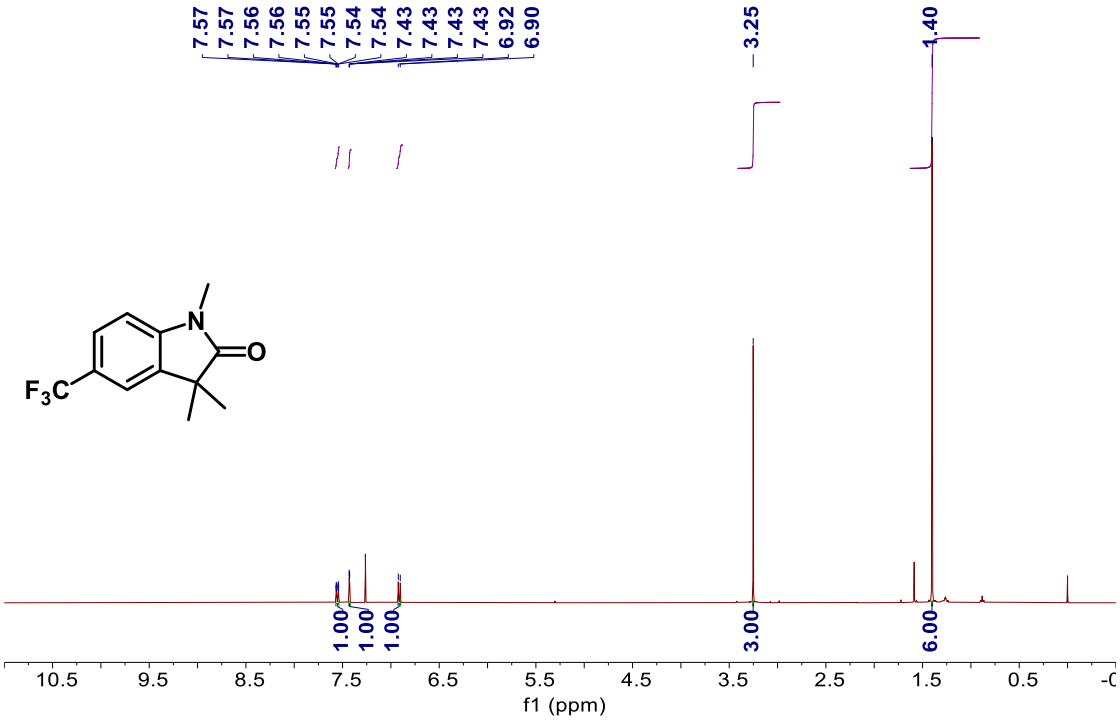
^{1}H NMR of compound **5** (400 MHz in $CDCl_3$)



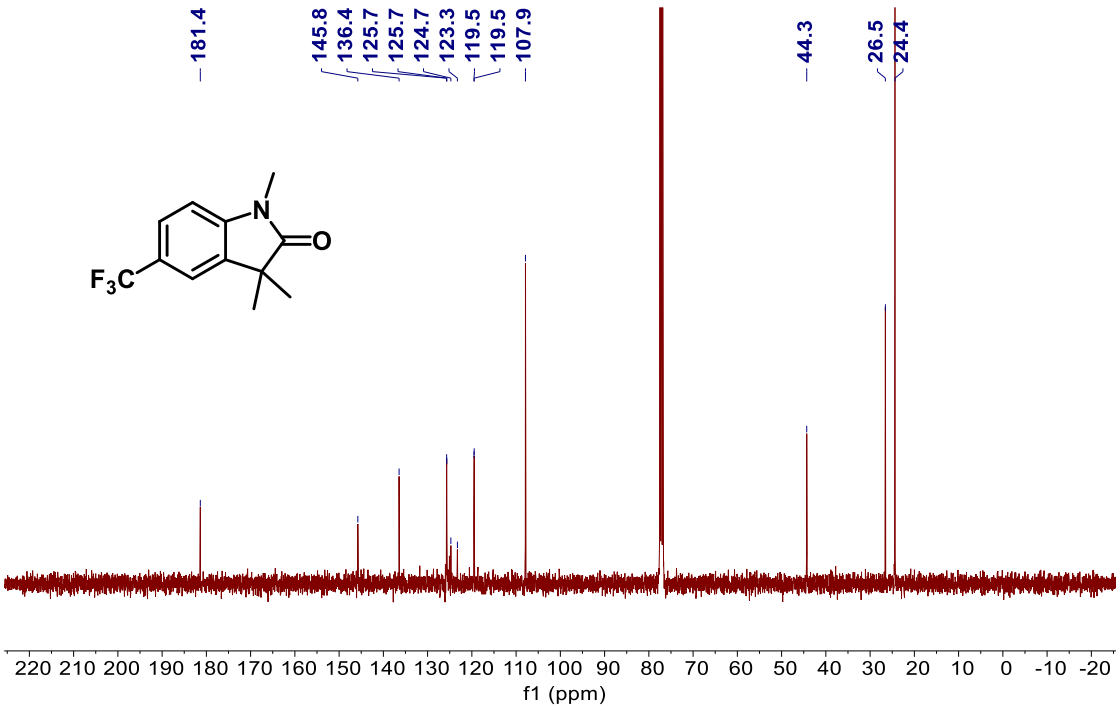
^{13}C NMR of compound **5** (151 MHz in $CDCl_3$)



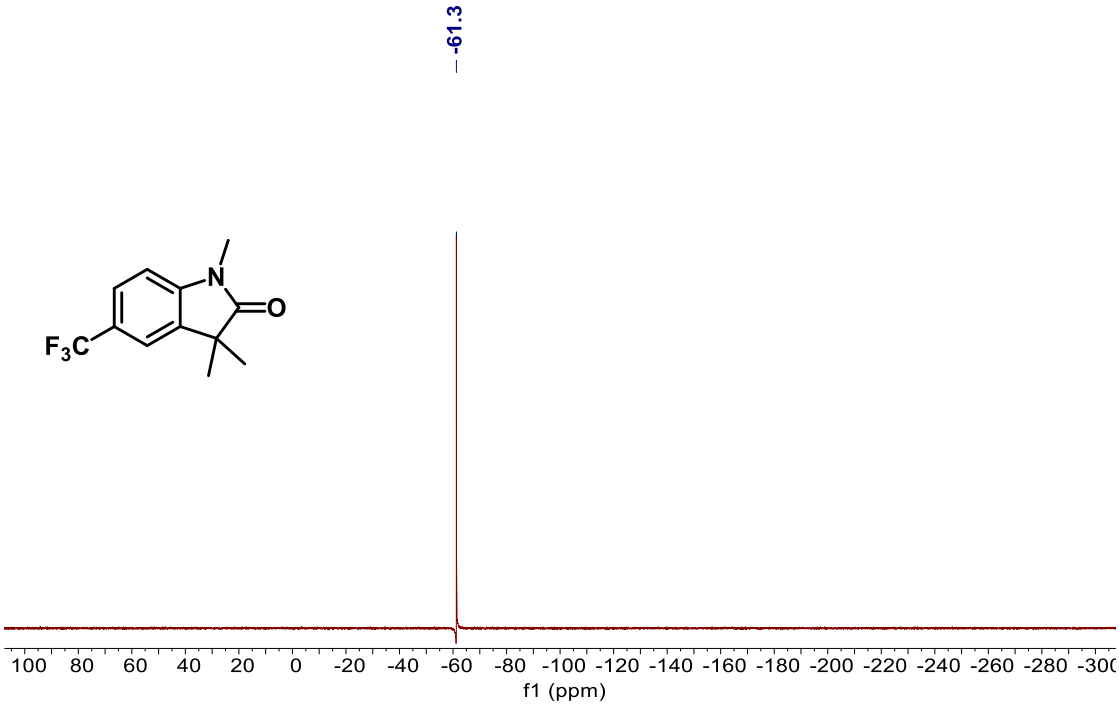
^{1}H NMR of compound **6** (400 MHz in $CDCl_3$)



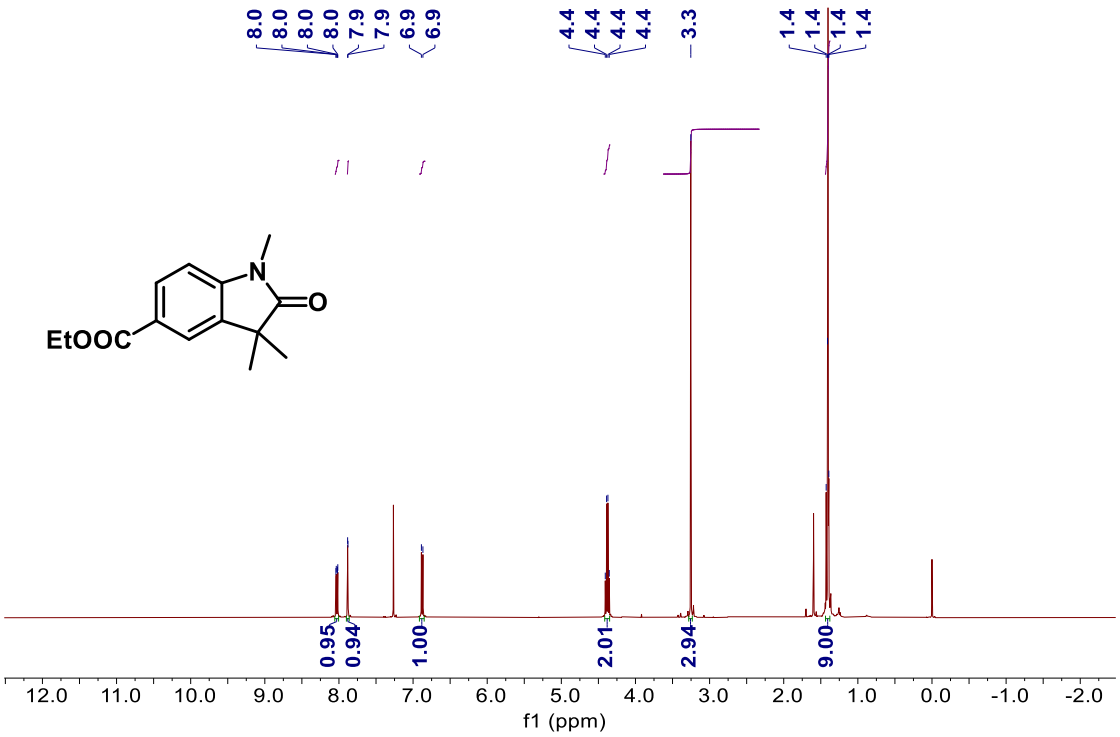
^{13}C NMR of compound **6** (101 MHz in $CDCl_3$)



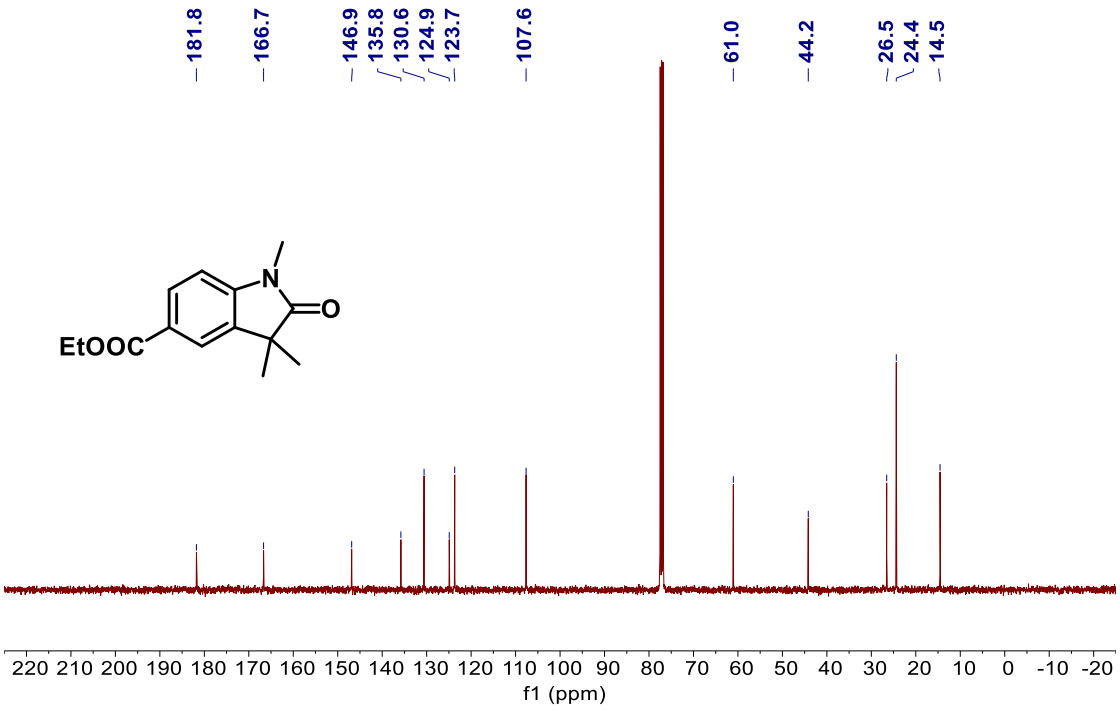
^{19}F NMR of compound **6** (101 MHz in $CDCl_3$)



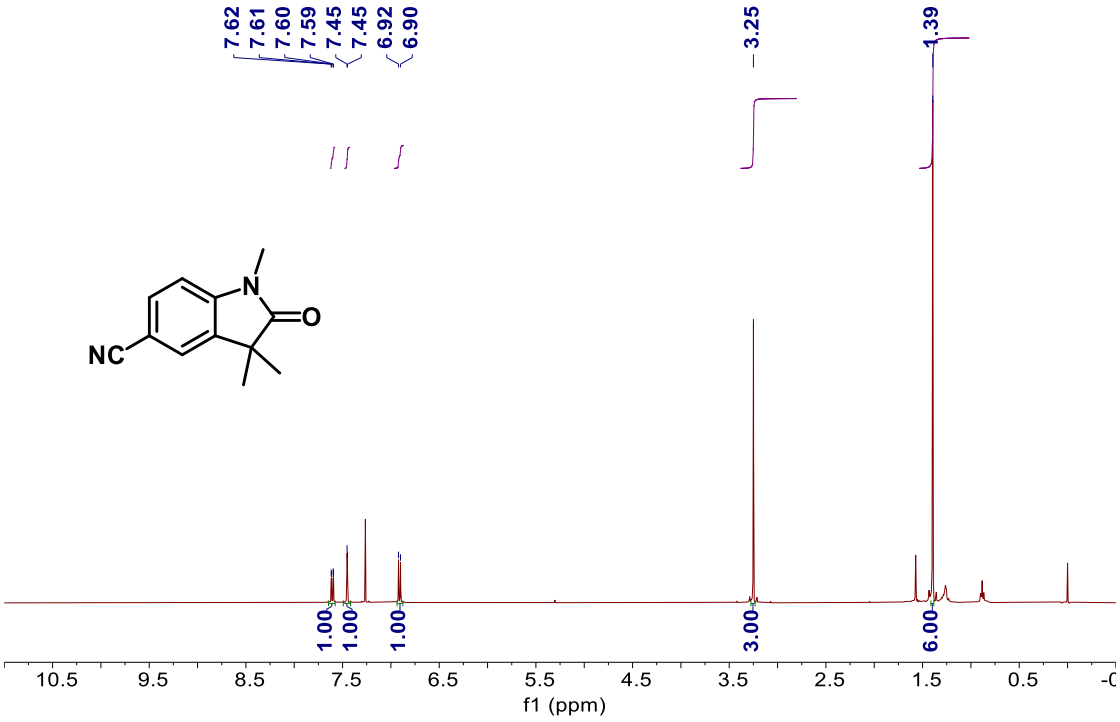
^{1}H NMR of compound **7** (400 MHz in $CDCl_3$)



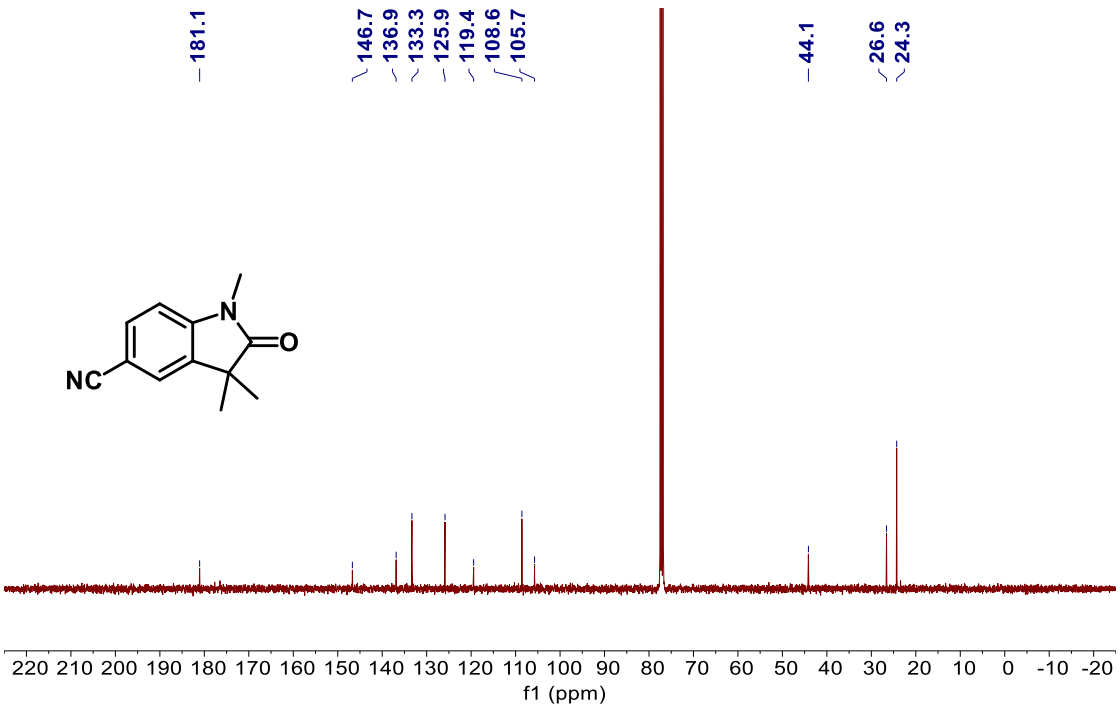
^{13}C NMR of compound **7** (101 MHz in $CDCl_3$)



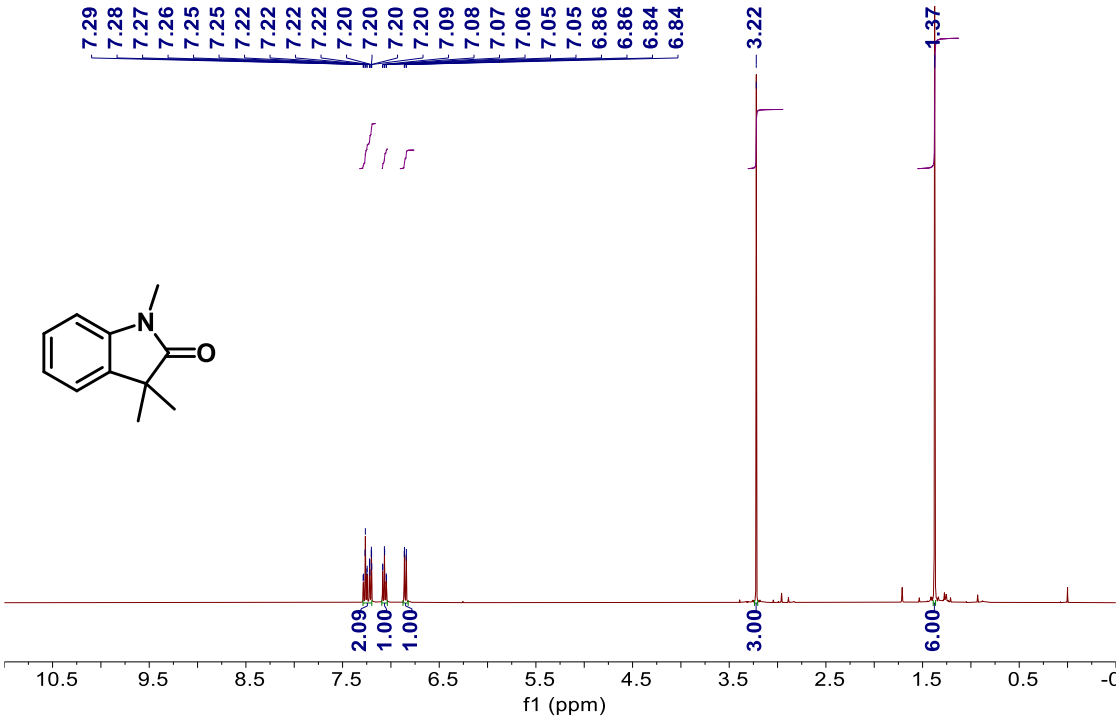
^{1}H NMR of compound **8** (400 MHz in $CDCl_3$)



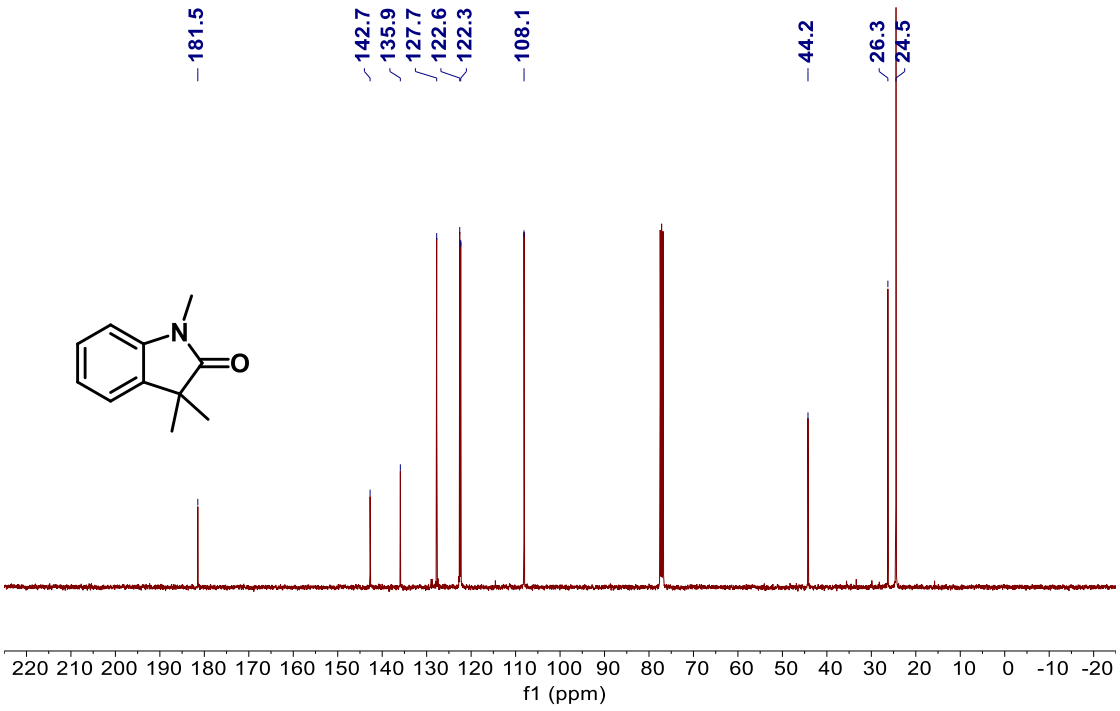
^{13}C NMR of compound **8** (101 MHz in $CDCl_3$)



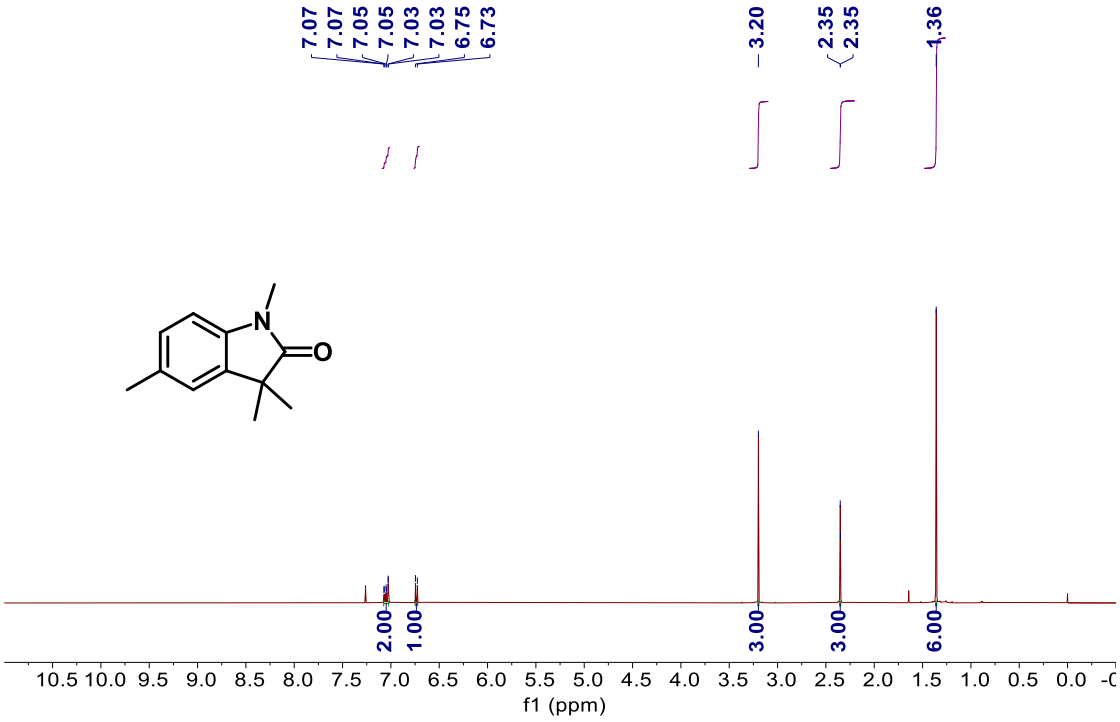
^{1}H NMR of compound **9** (400 MHz in $CDCl_3$)



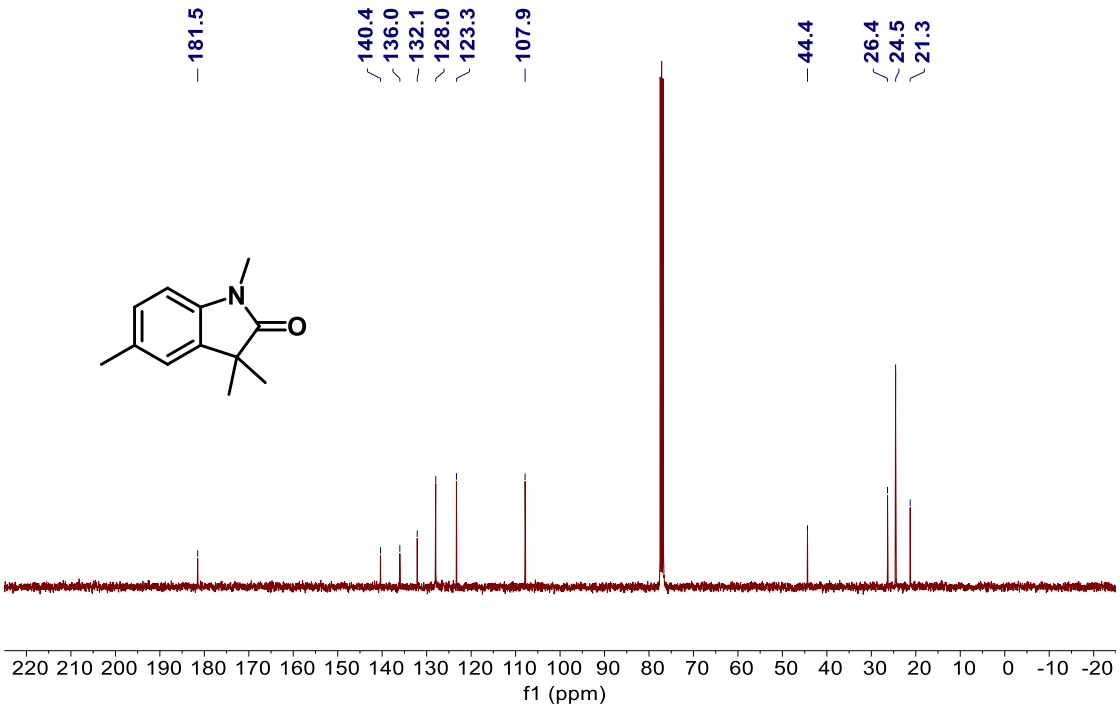
^{13}C NMR of compound **9** (101 MHz in $CDCl_3$)



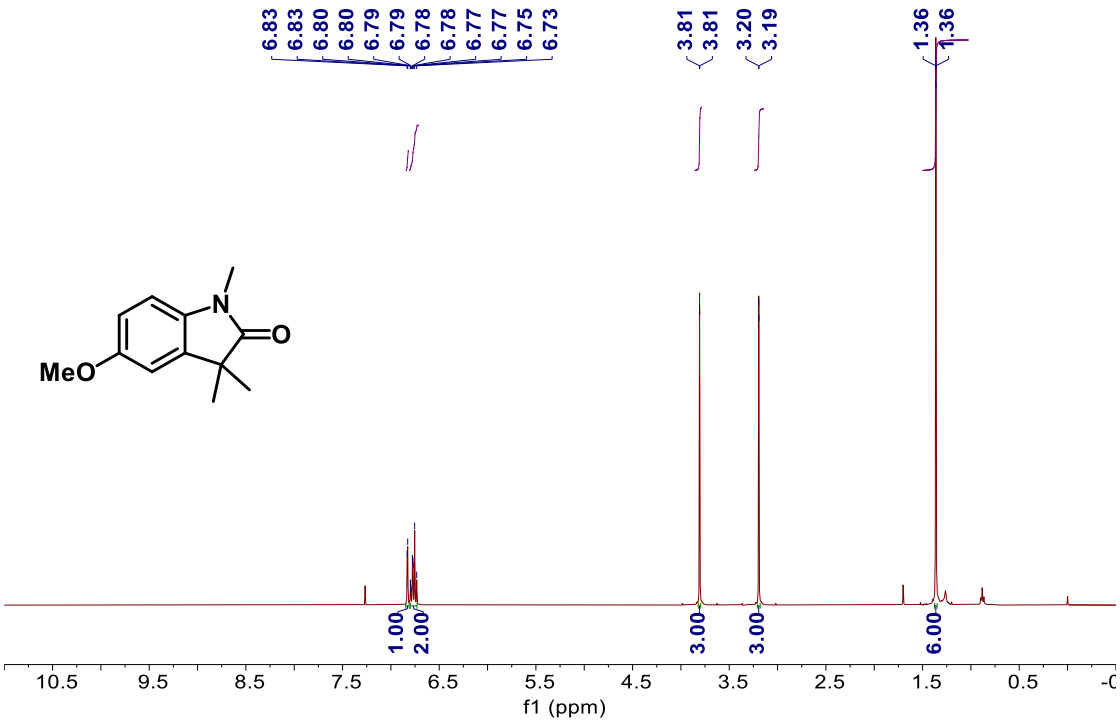
^{1}H NMR of compound **10** (400 MHz in $CDCl_3$)



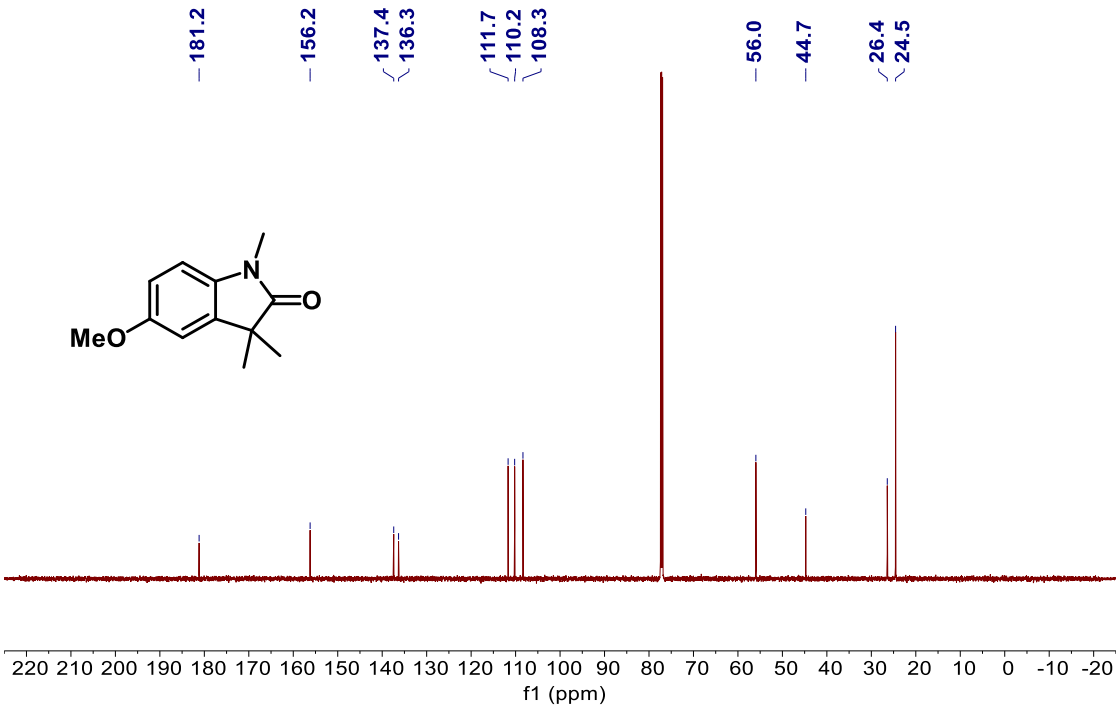
^{13}C NMR of compound **10** (101 MHz in $CDCl_3$)



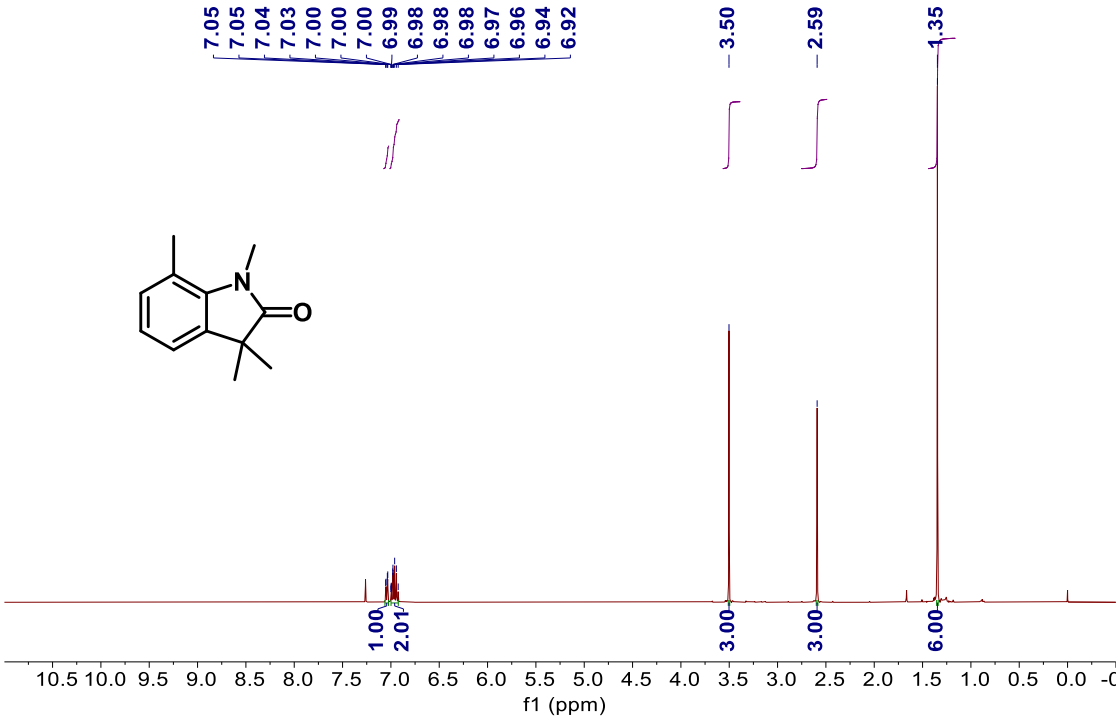
^{1}H NMR of compound **11** (500 MHz in $CDCl_3$)



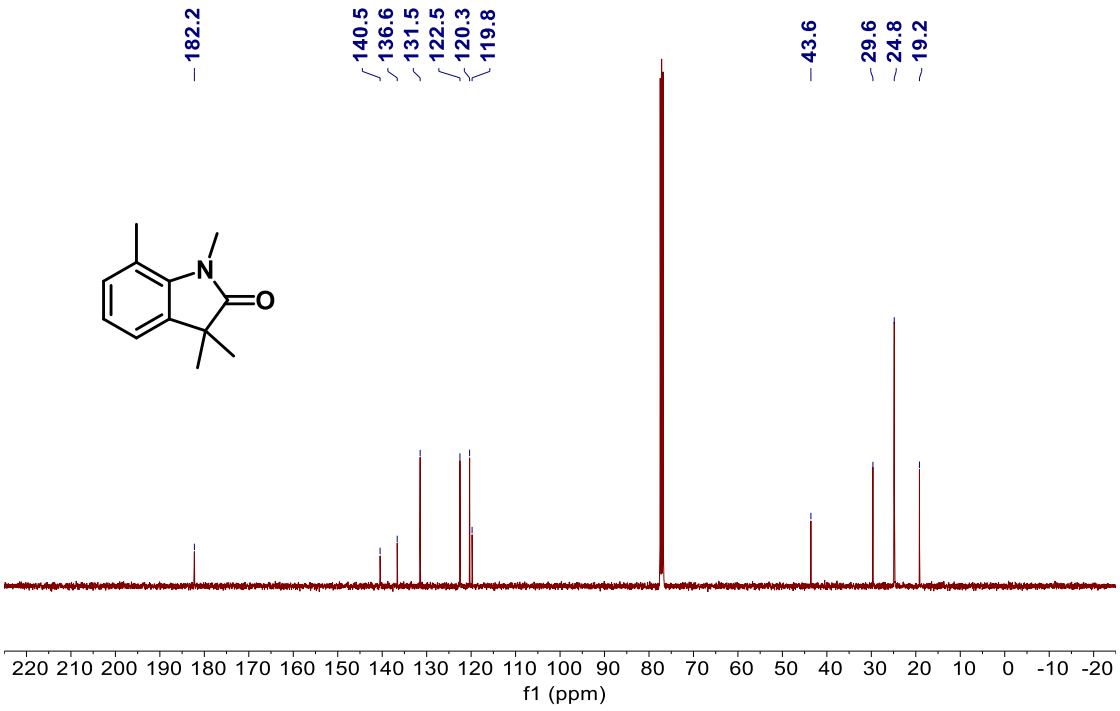
^{13}C NMR of compound **11** (151 MHz in $CDCl_3$)



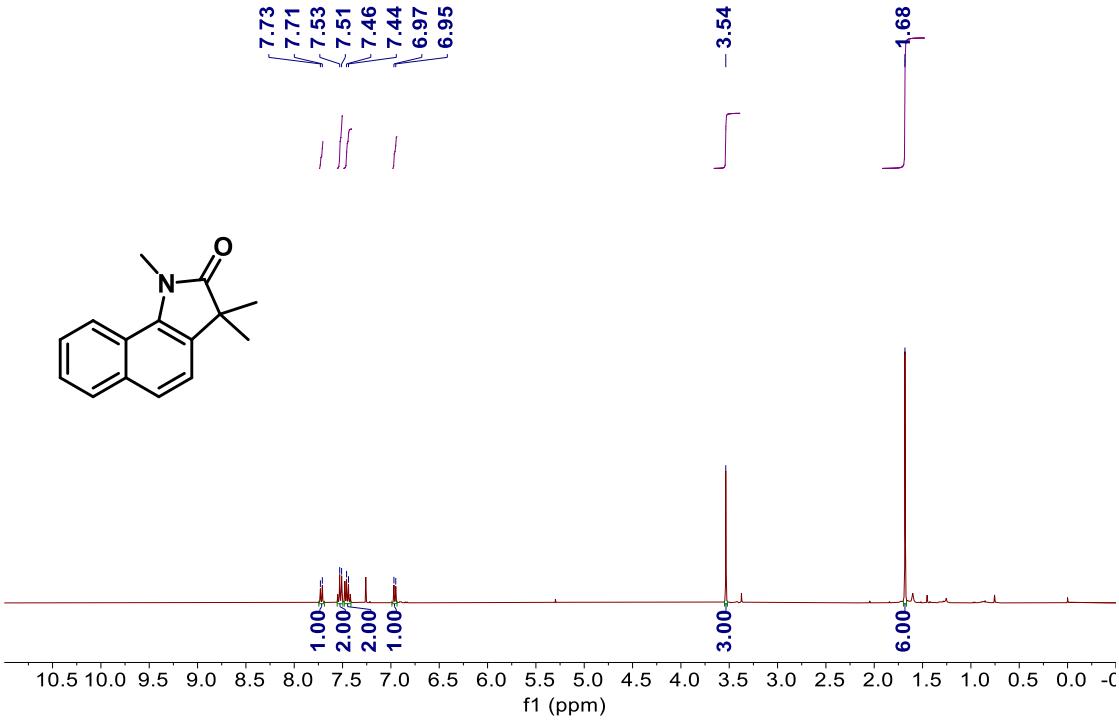
^{1}H NMR of compound **12** (400 MHz in $CDCl_3$)



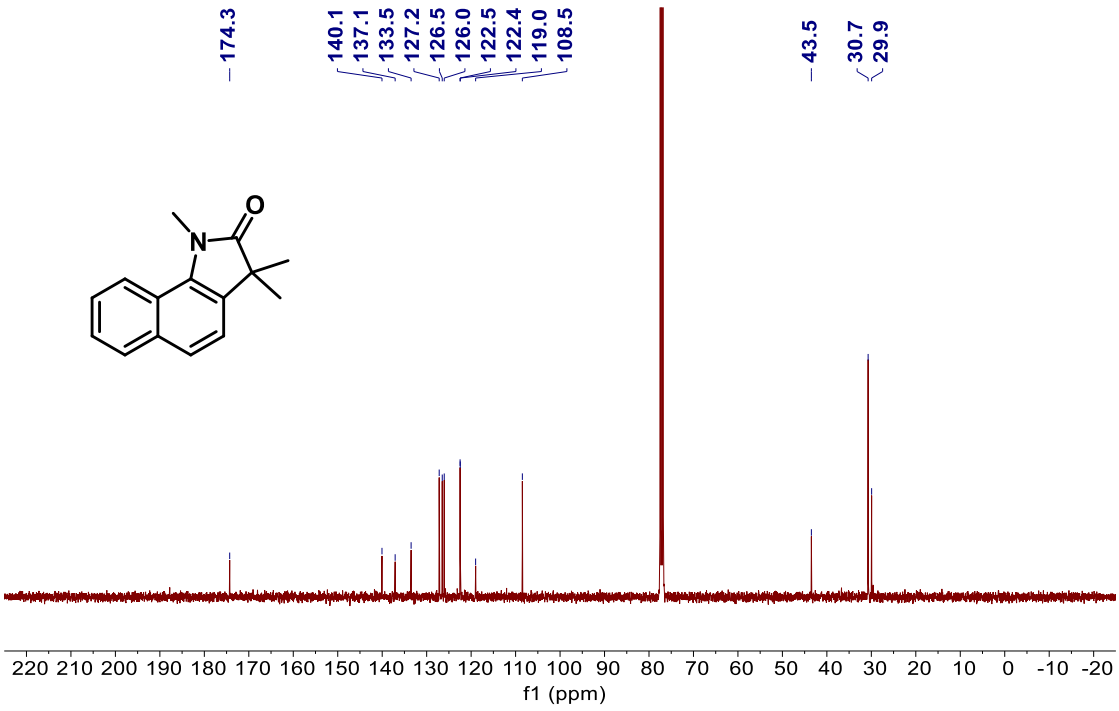
^{13}C NMR of compound **12** (101 MHz in $CDCl_3$)



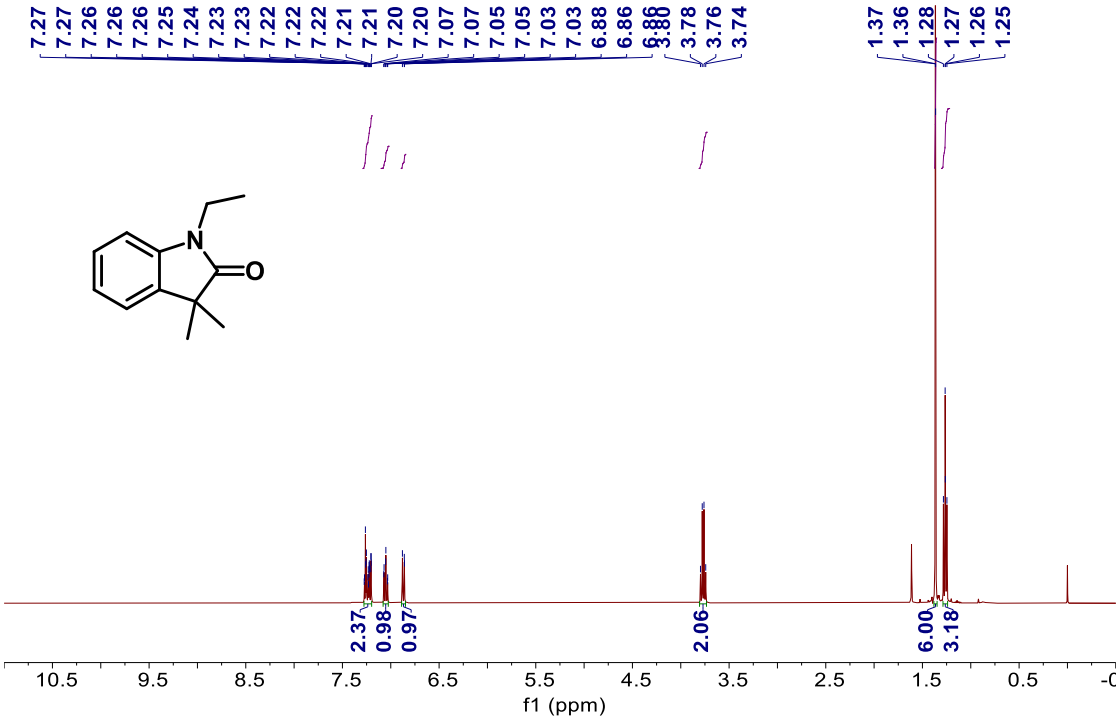
^{1}H NMR of compound **13** (400 MHz in $CDCl_3$)



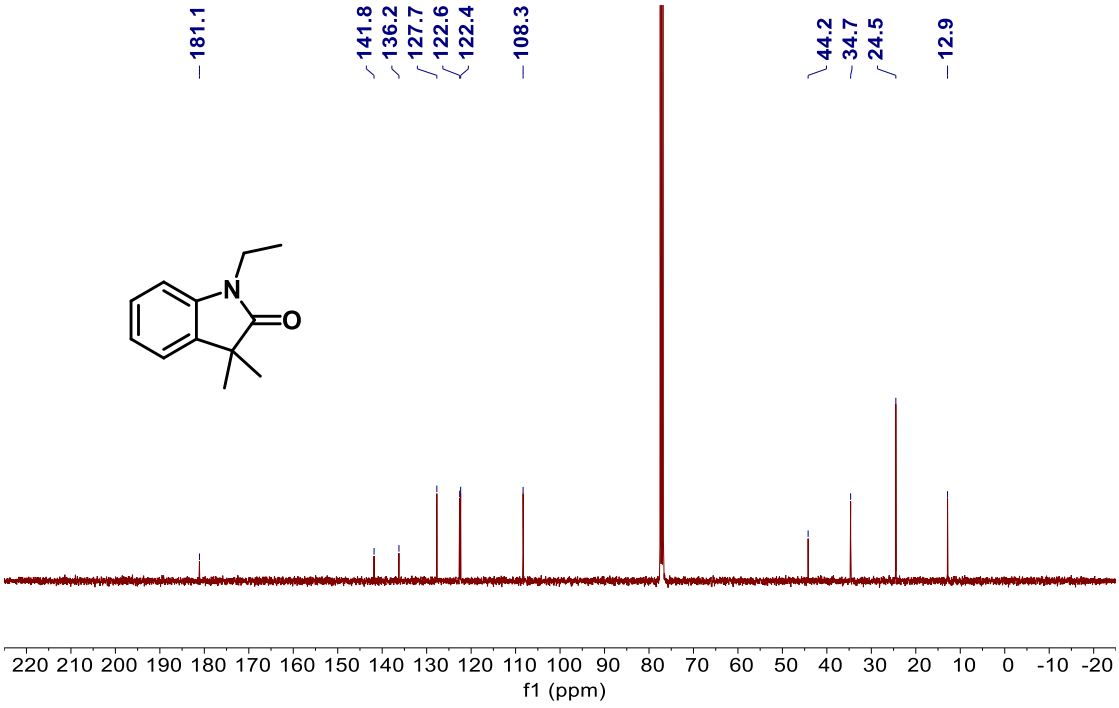
^{13}C NMR of compound **13** (101 MHz in $CDCl_3$)



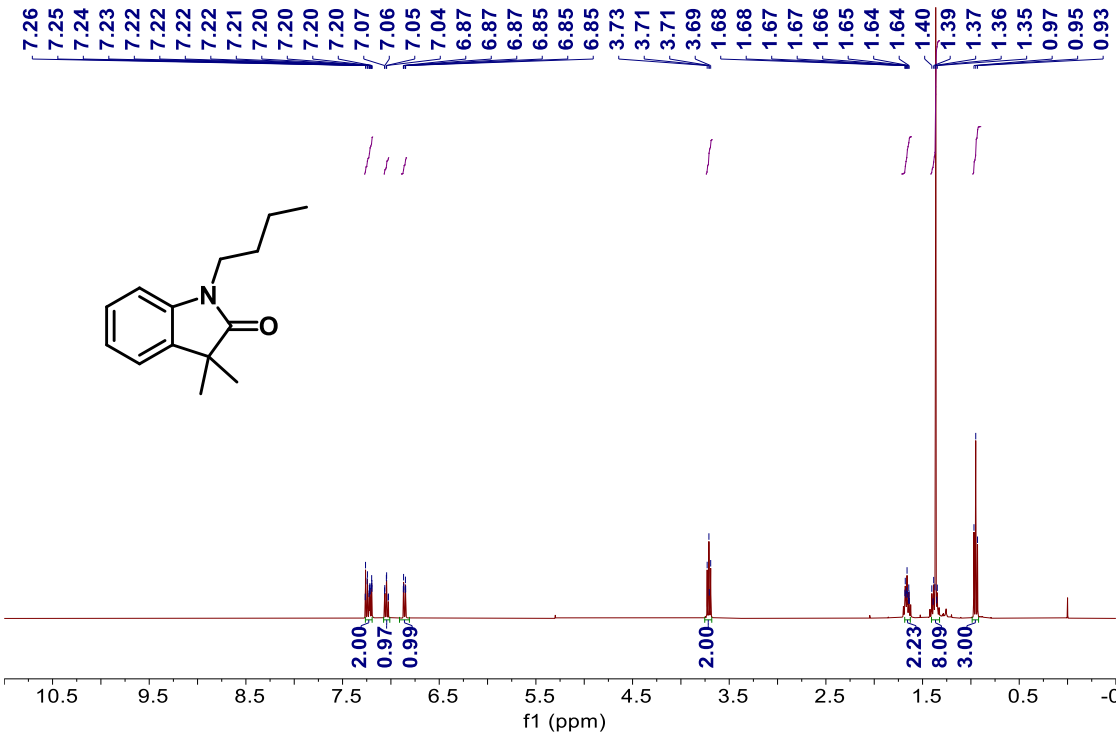
^{1}H NMR of compound **14** (400 MHz in $CDCl_3$)



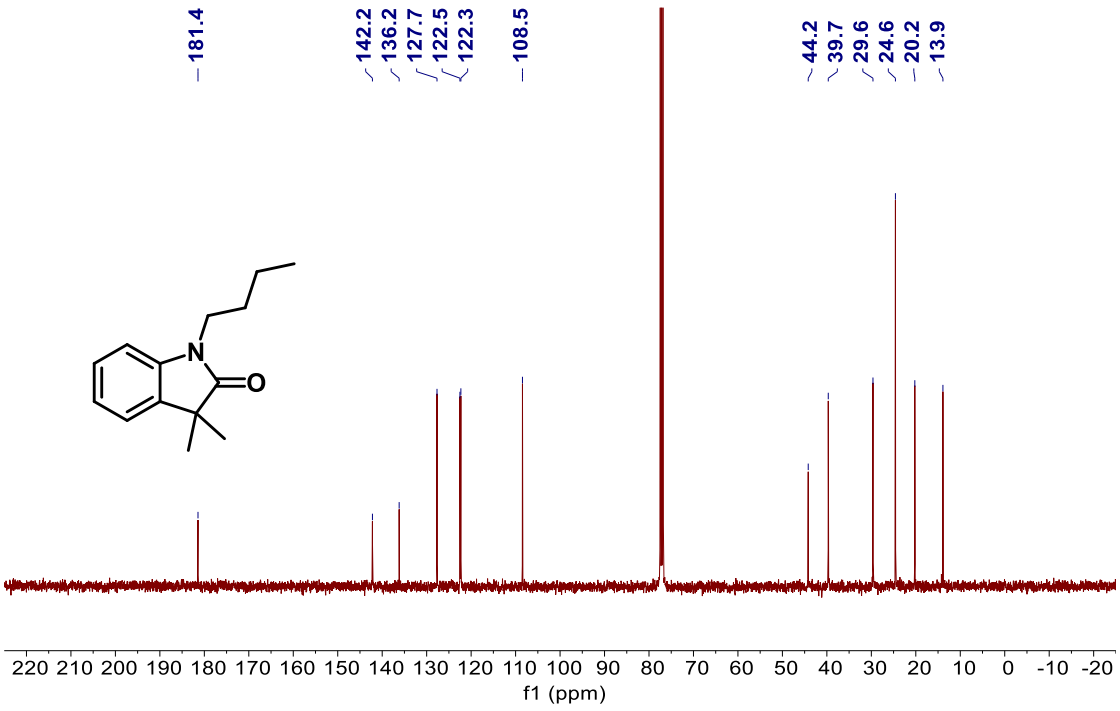
^{13}C NMR of compound **14** (101 MHz in $CDCl_3$)



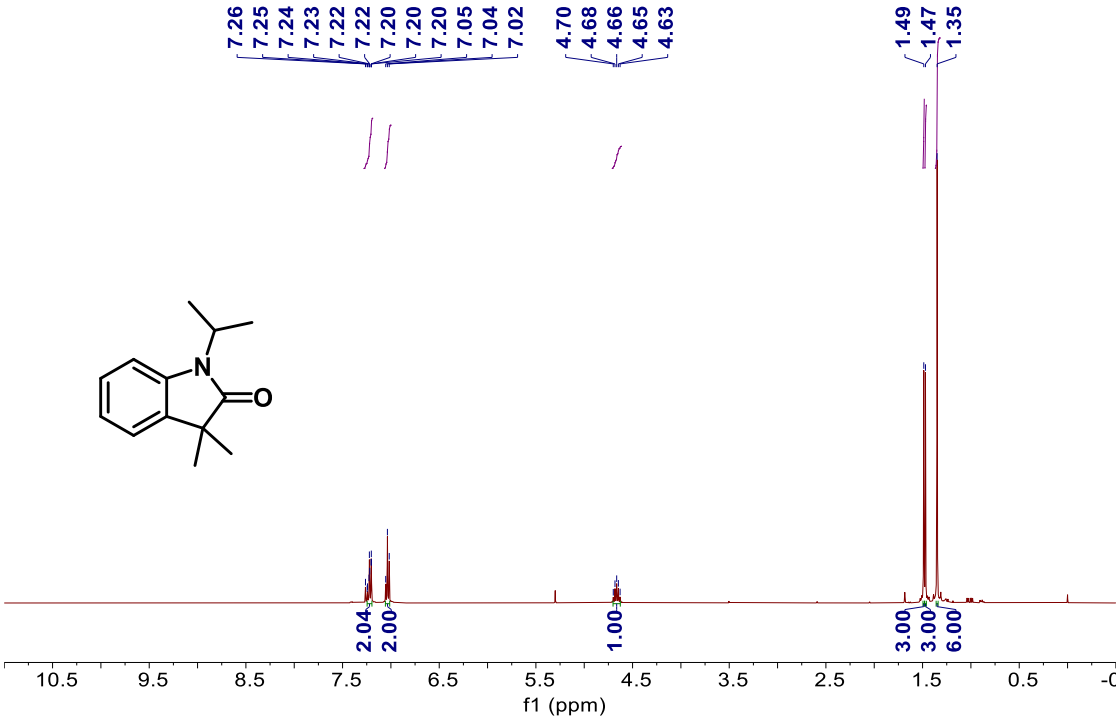
^{1}H NMR of compound **15** (400 MHz in $CDCl_3$)



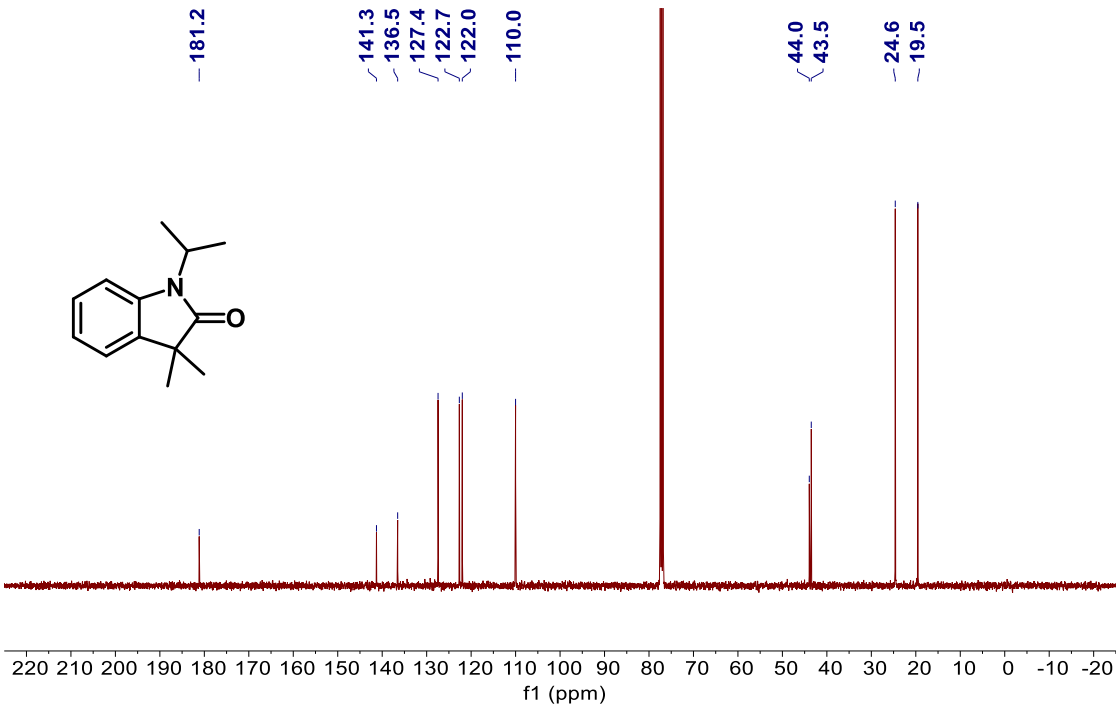
^{13}C NMR of compound **15** (101 MHz in $CDCl_3$)



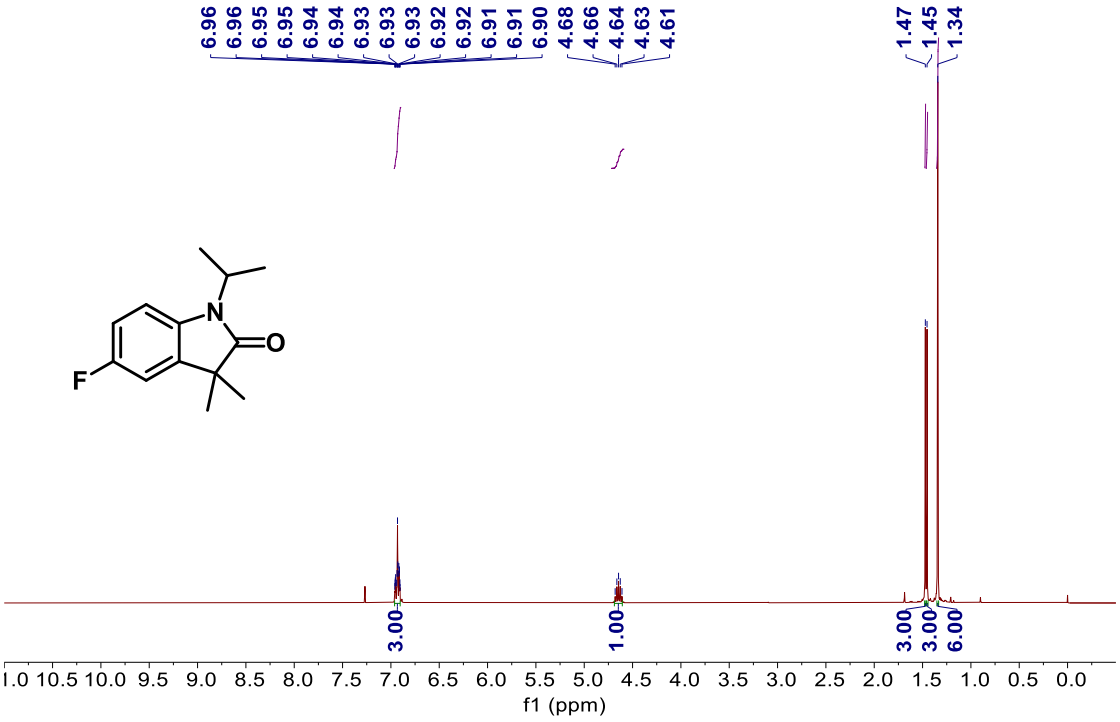
^{1}H NMR of compound **16** (400 MHz in $CDCl_3$)



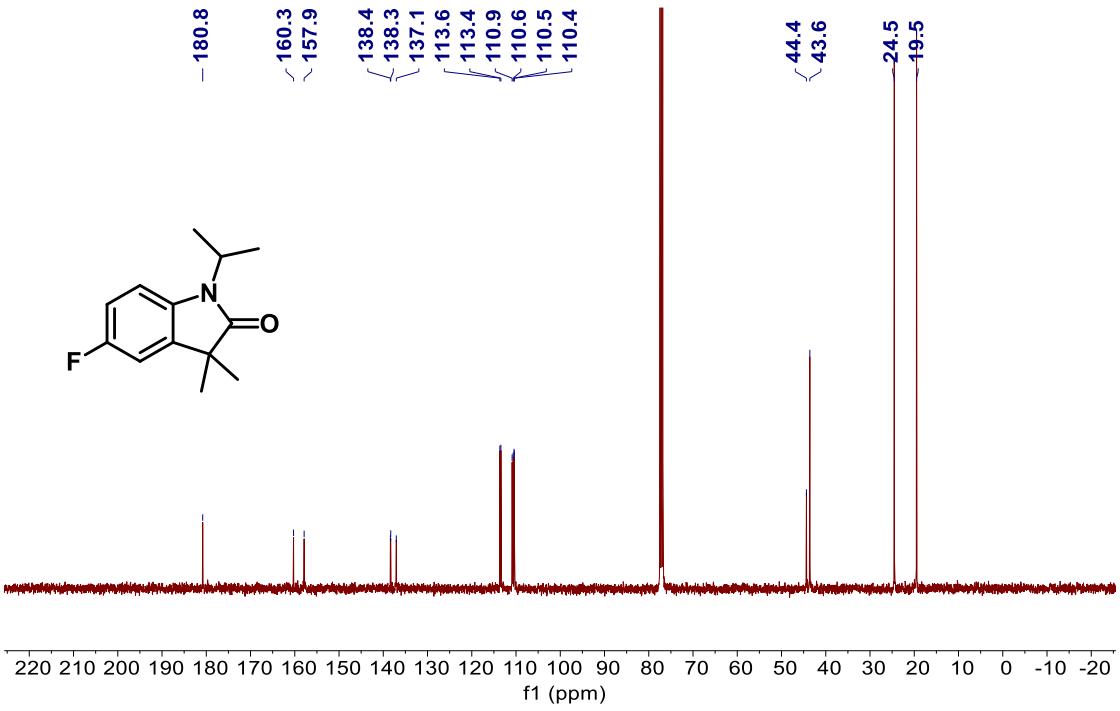
^{13}C NMR of compound **16** (101 MHz in $CDCl_3$)



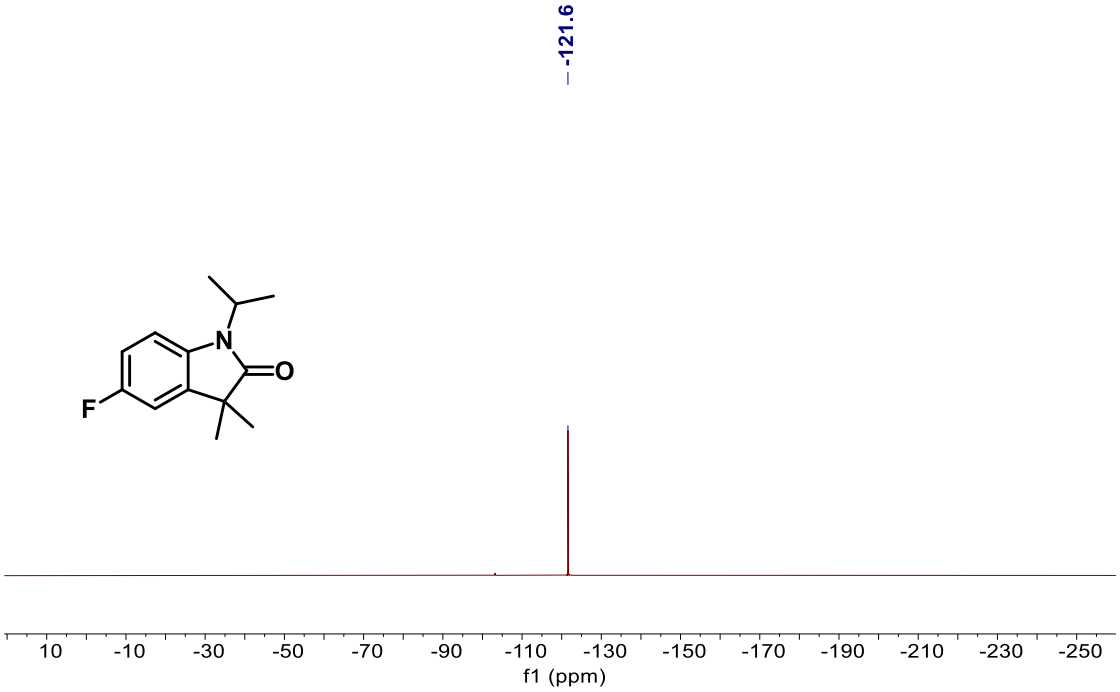
^{1}H NMR of compound **17** (400 MHz in $CDCl_3$)



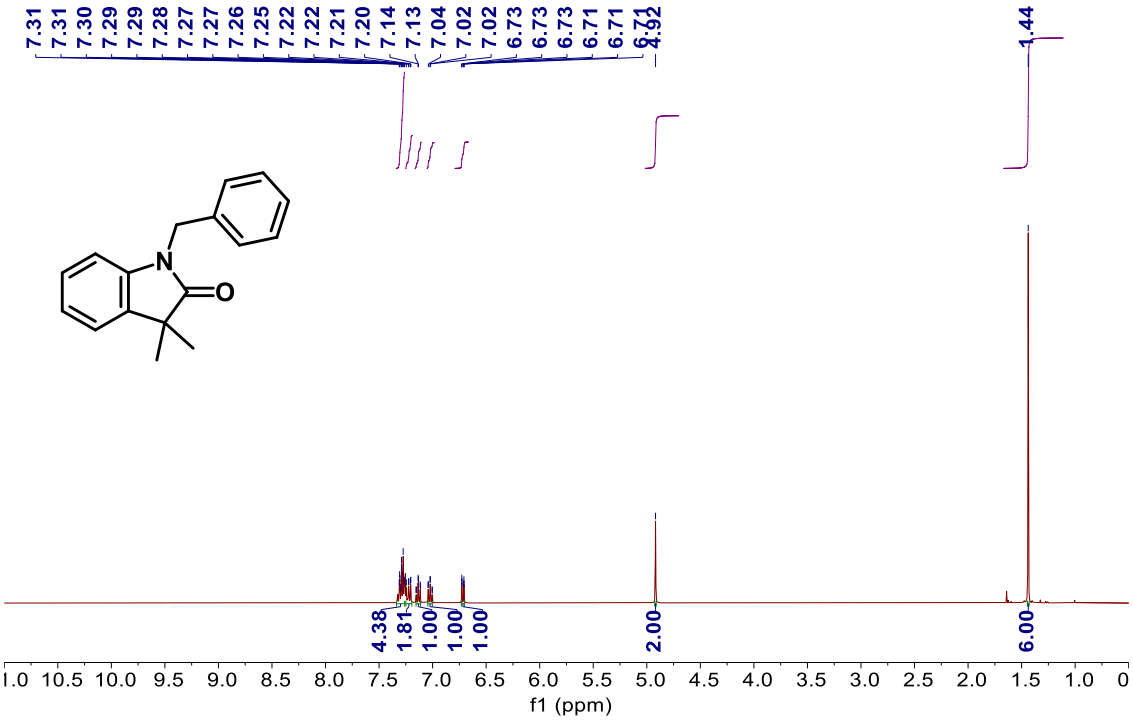
^{13}C NMR of compound **17** (101 MHz in $CDCl_3$)



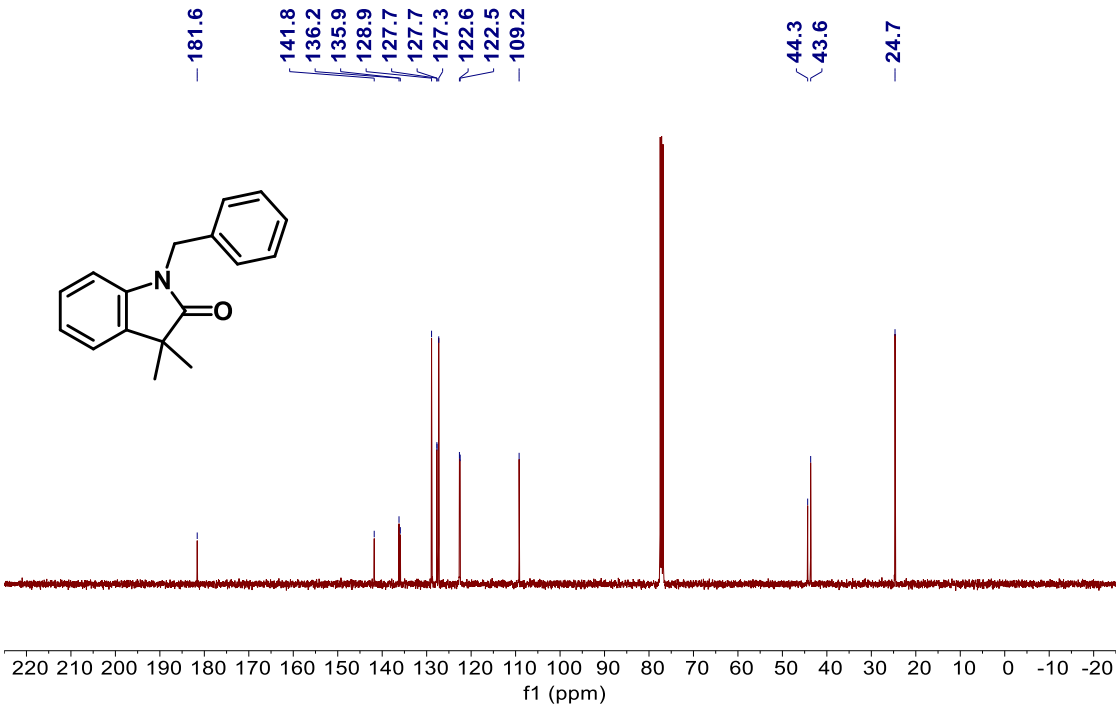
^{19}F NMR of compound **17** (101 MHz in $CDCl_3$)



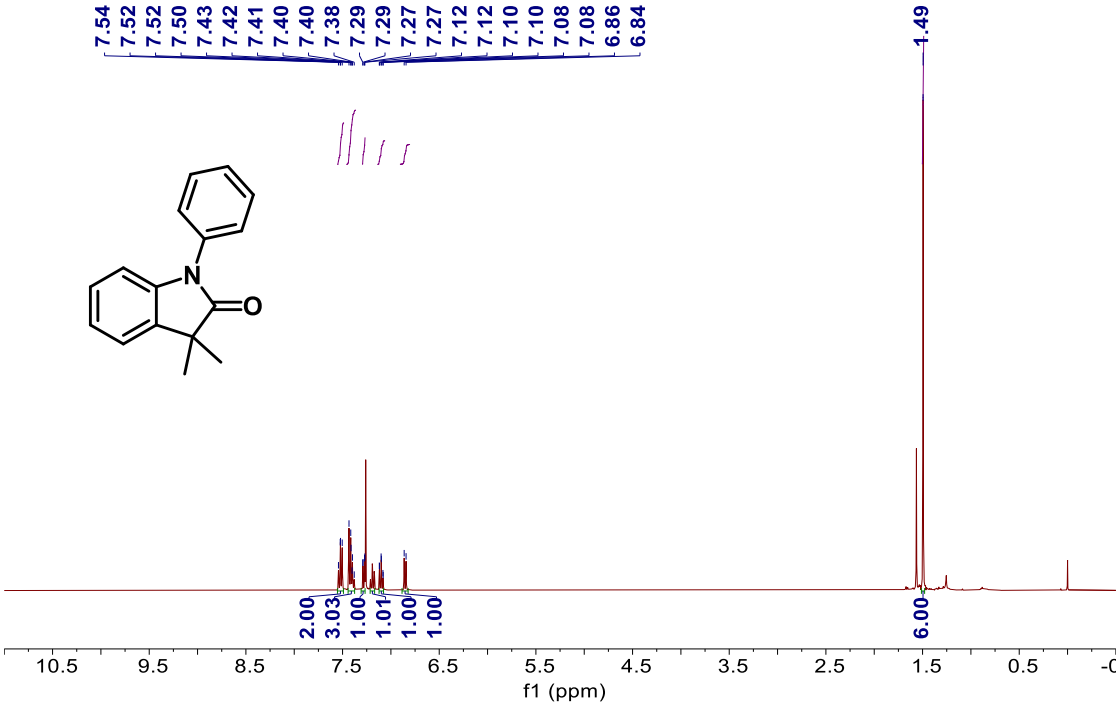
^{1}H NMR of compound **18** (400 MHz in $CDCl_3$)



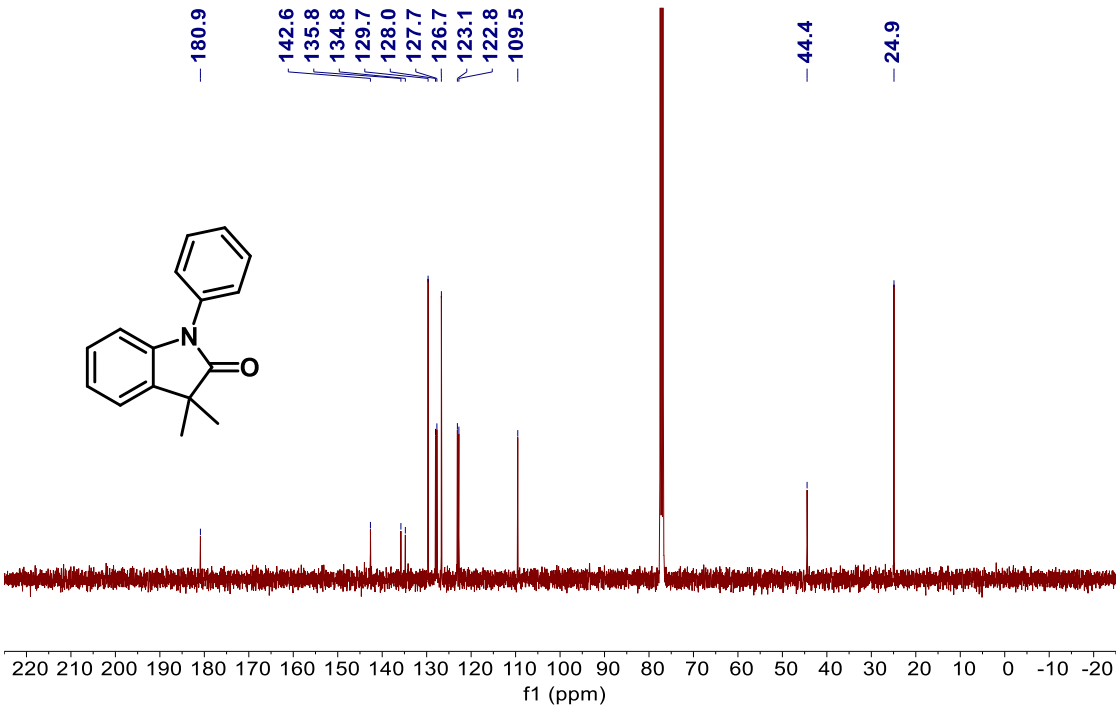
^{13}C NMR of compound **18** (101 MHz in $CDCl_3$)



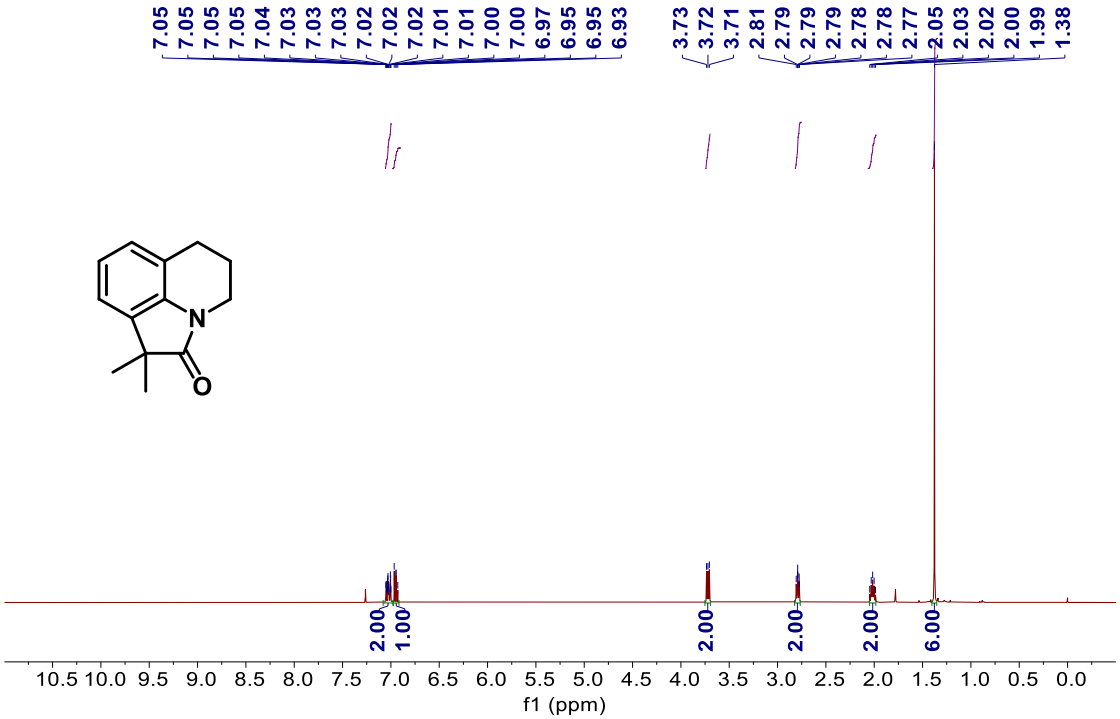
^{1}H NMR of compound **19** (400 MHz in $CDCl_3$)



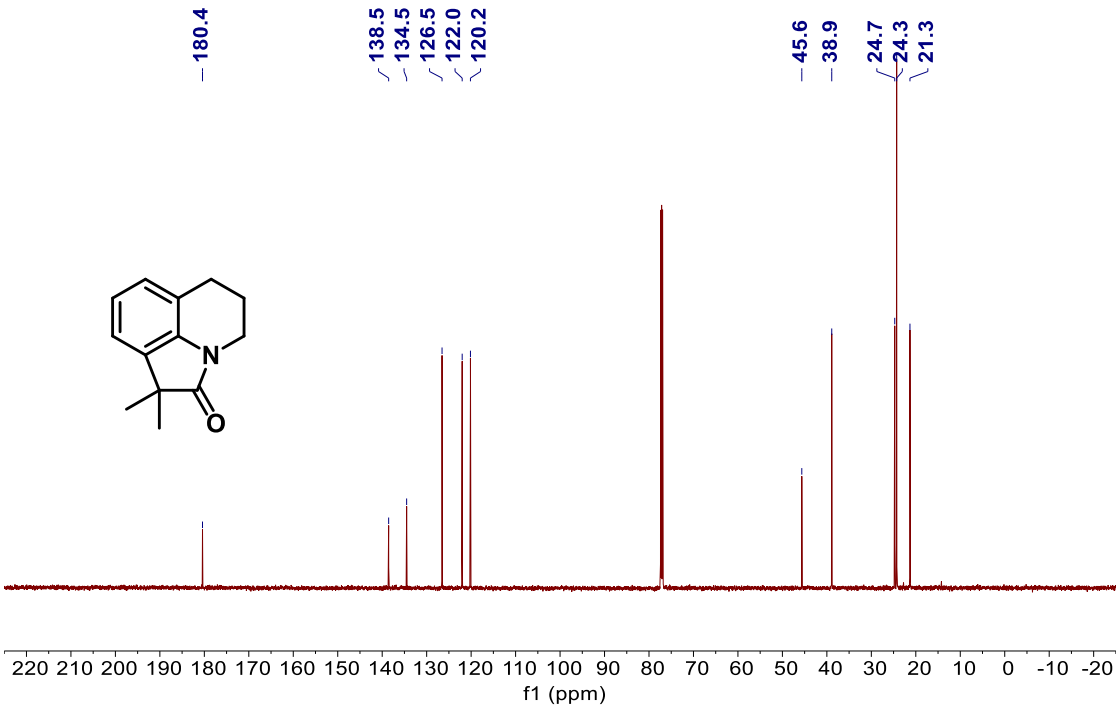
^{13}C NMR of compound **19** (101 MHz in $CDCl_3$)



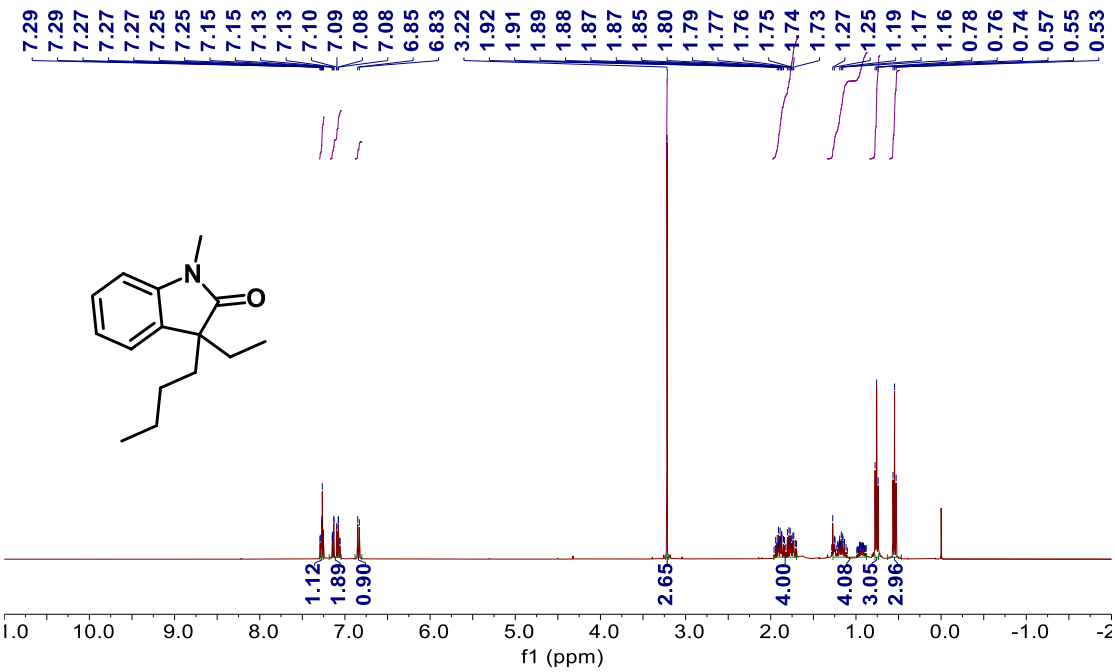
^{1}H NMR of compound **20** (400 MHz in $CDCl_3$)



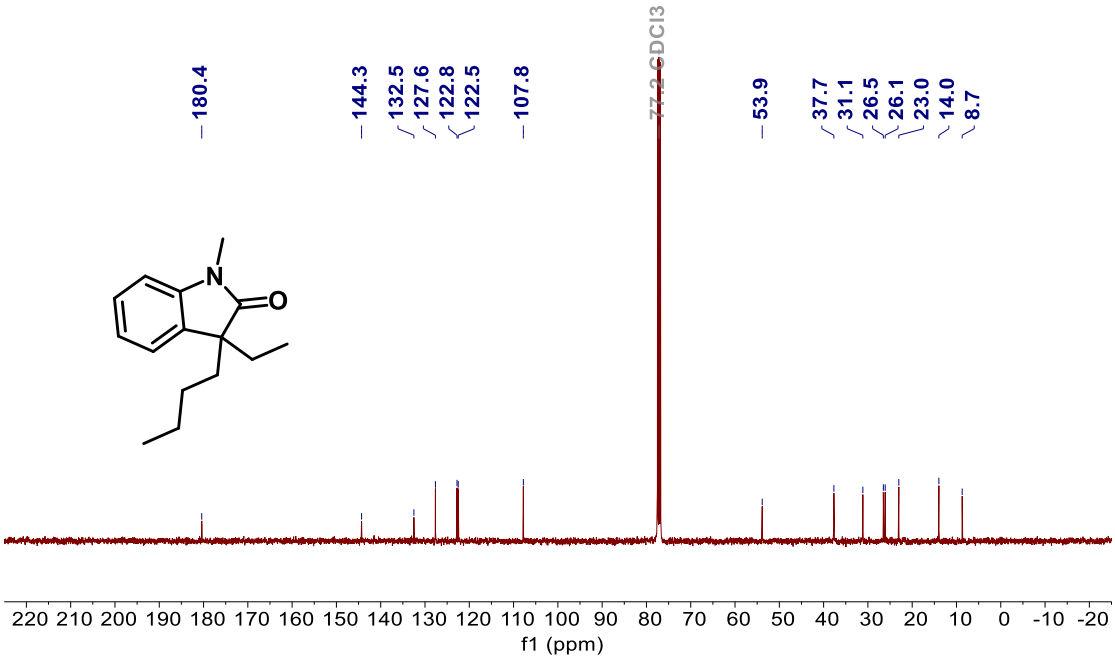
^{13}C NMR of compound **20** (151 MHz in $CDCl_3$)



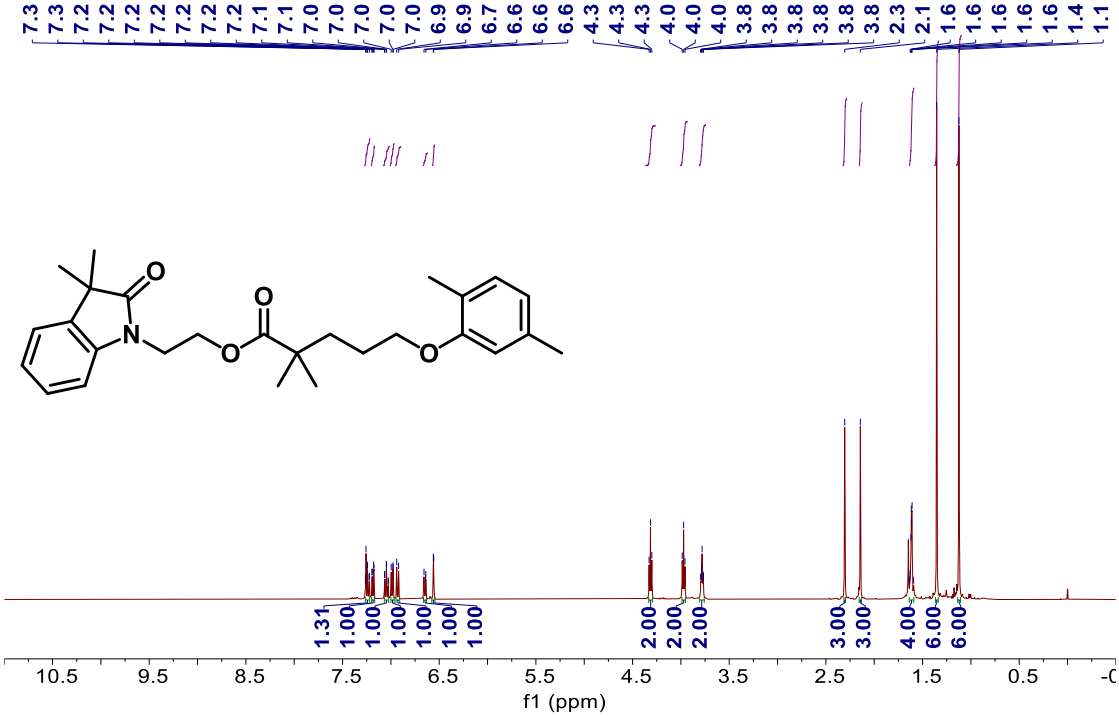
^{1}H NMR of compound **21** (400 MHz in $CDCl_3$)



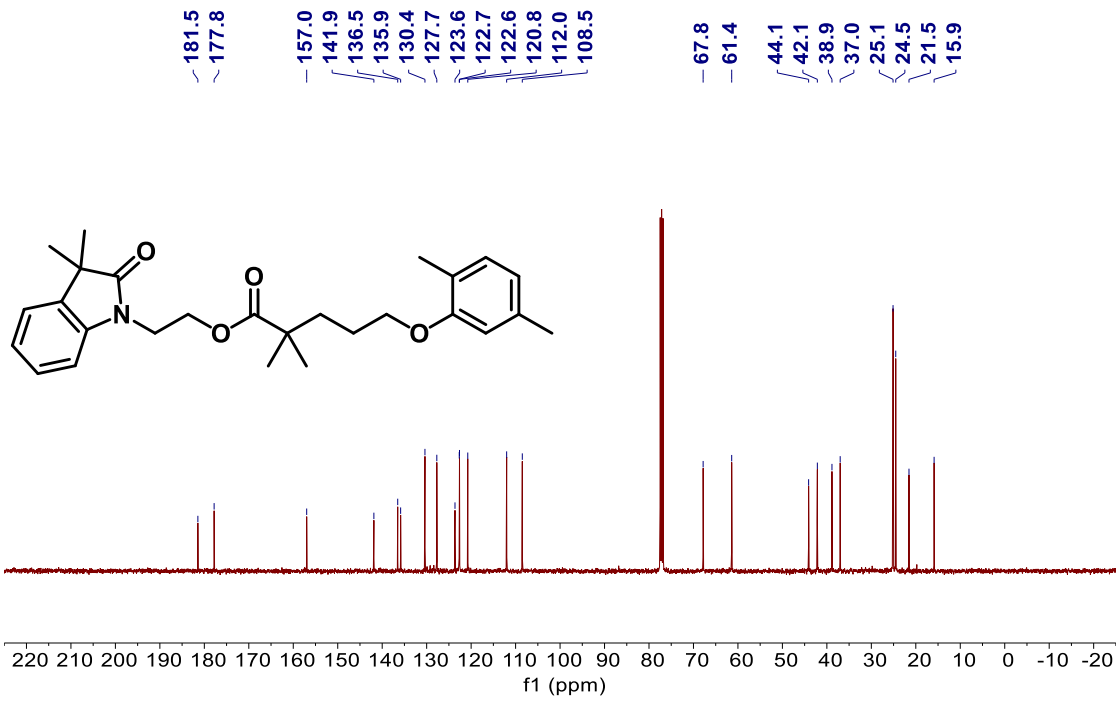
^{13}C NMR of compound **21** (101 MHz in $CDCl_3$)



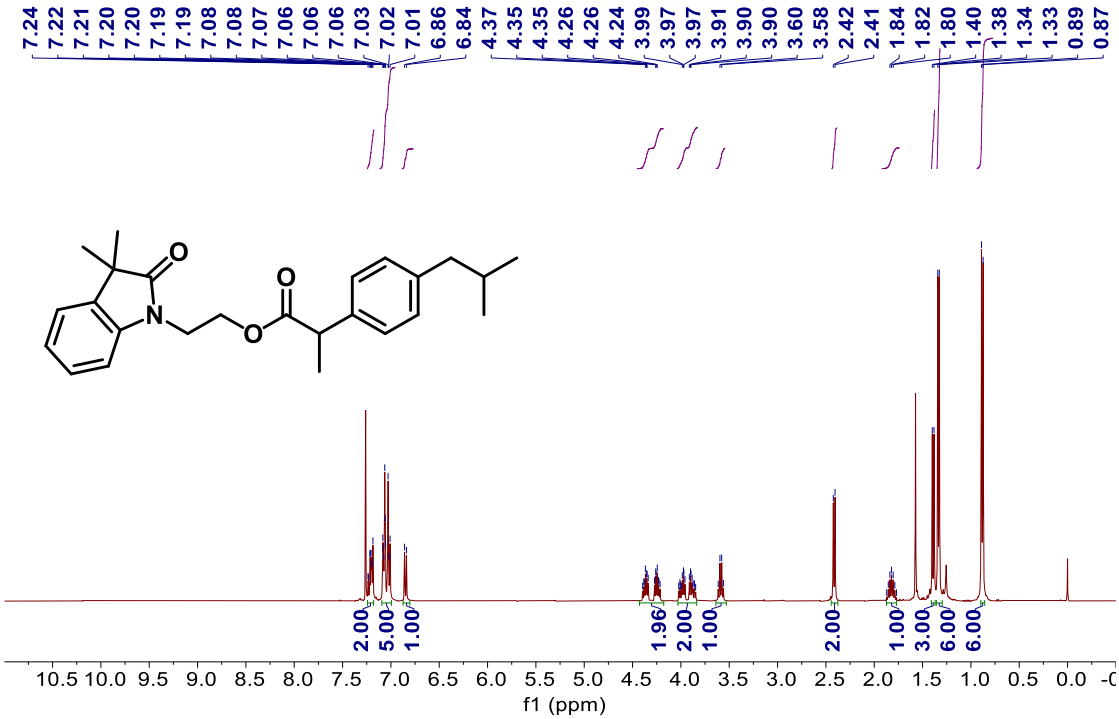
^{1}H NMR of compound **22** (400 MHz in $CDCl_3$)



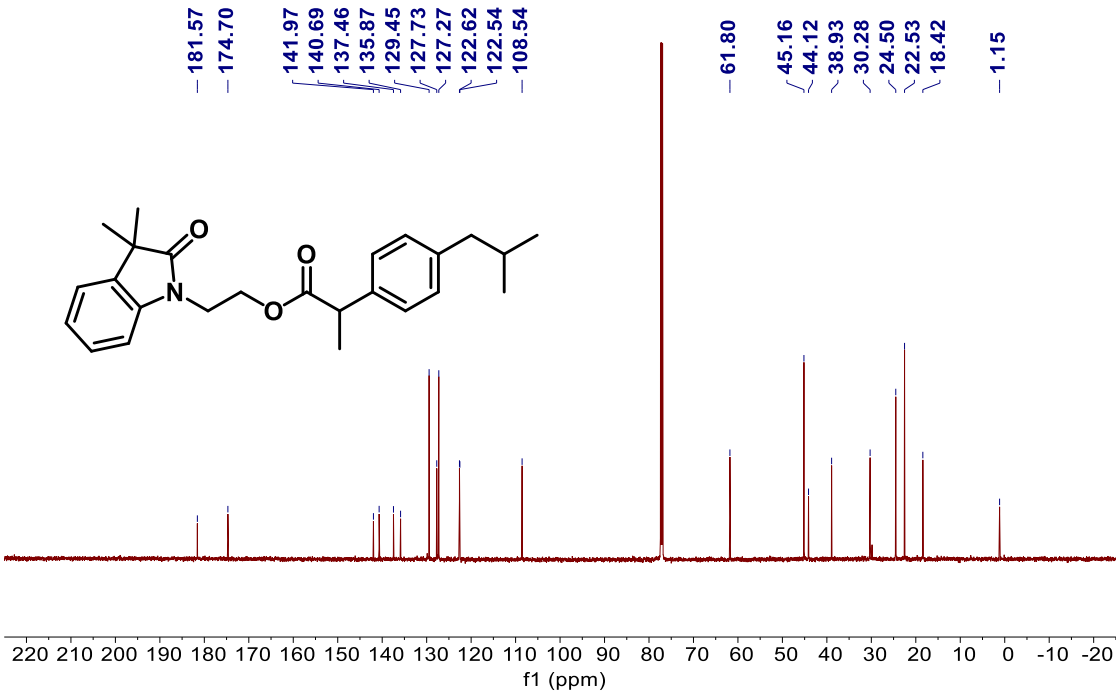
^{13}C NMR of compound **22** (101 MHz in $CDCl_3$)



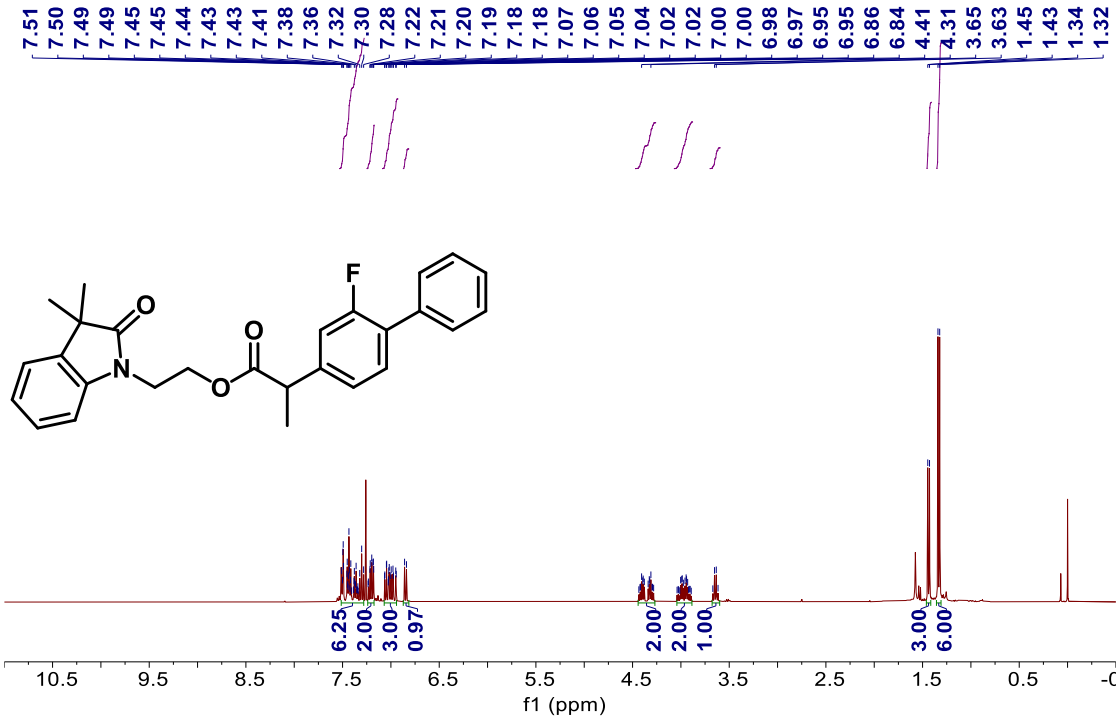
^{1}H NMR of compound **23** (400 MHz in $CDCl_3$)



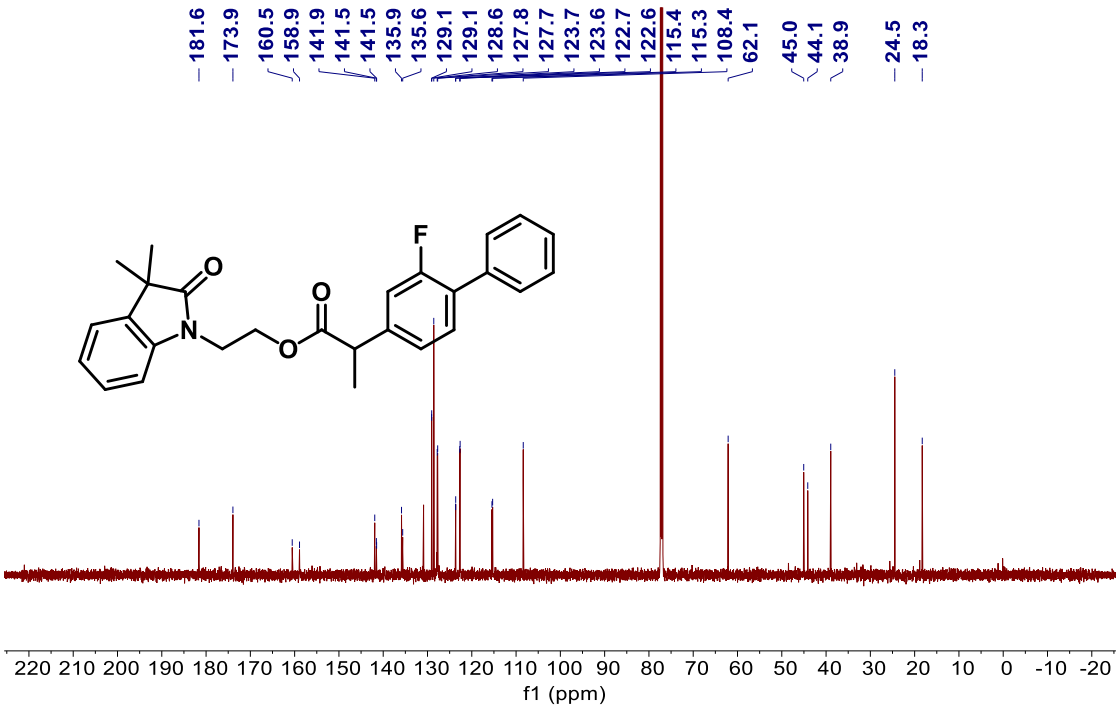
^{13}C NMR of compound **23** (151 MHz in $CDCl_3$)



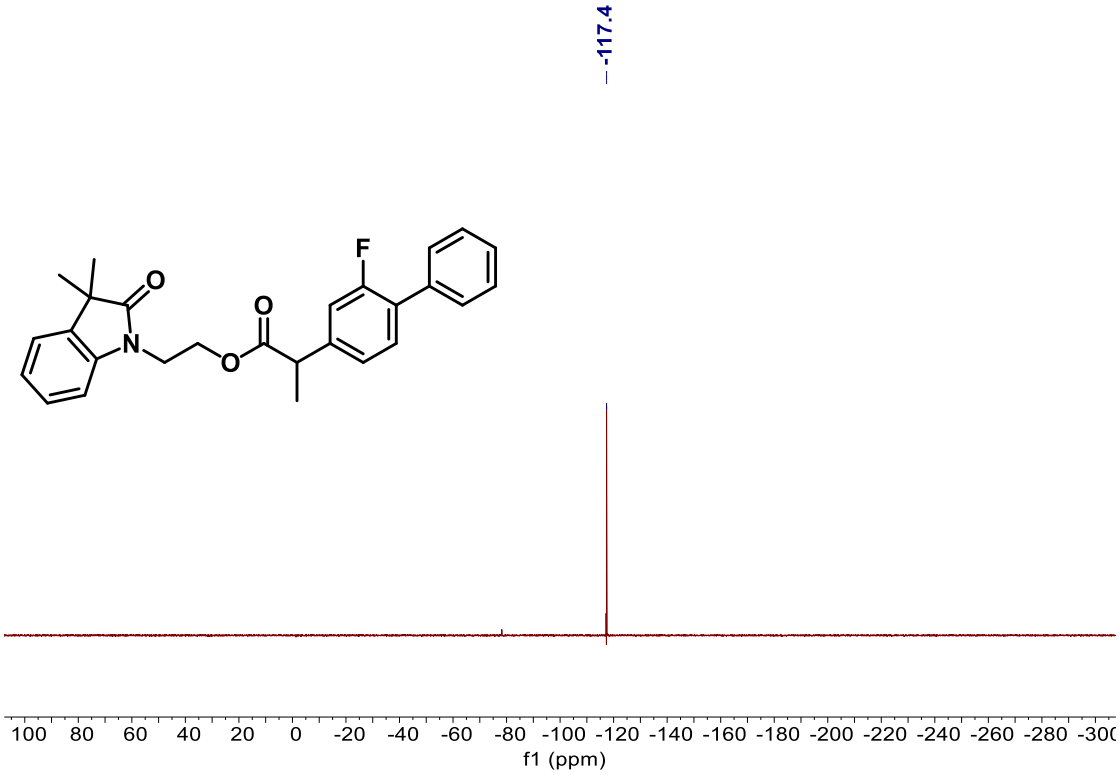
^{1}H NMR of compound **24** (400 MHz in $CDCl_3$)



^{13}C NMR of compound **24** (101 MHz in $CDCl_3$)



^{19}F NMR of compound **24** (376 MHz in $CDCl_3$)



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