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Scandium triflate-catalyzed selective ring opening and rearrangement reaction of spiro-epoxyoxindole and carbonyl compound

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1 General Information

The NMR spectra were recorded on Bruker AC – 500 spectrometer (500 MHz for ^{1}H NMR and 125 MHz for ^{13}C NMR) with $CDCl_3$ as the solvent and TMS as internal reference. ^{1}H NMR spectral data were reported as follows: chemical shift (δ, ppm), multiplicity, integration, and coupling constant (Hz). ^{13}C NMR spectral data were reported in terms of the chemical shift. The following abbreviations were used to indicate multiplicities: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet. Low-resolution mass spectra were obtained on a Shimadzu LCMS-2010EV spectrometer in ESI mode and reported as m/z. High-resolution mass spectra (HRMS) were recorded on a Bruker Daltonics, Inc. APEXIII 7.0 TESLA FTMS instrument. Melting points were obtained on a X-4 digital melting point apparatus without correction. Chemical yields referred to pure isolated product. Purification of products was accomplished by column chromatography packed with silica gel. Unless otherwise stated, all reagents were commercially purchased and used without further purification.

2 General Procedure

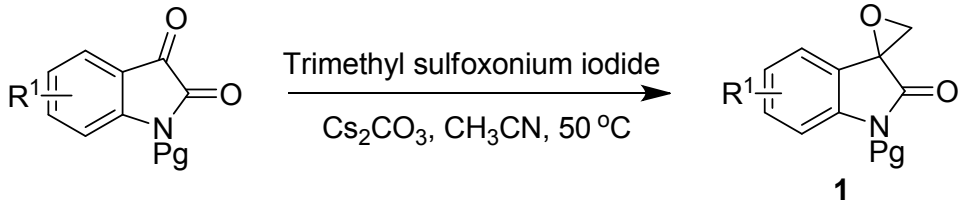
(a) General procedure for the formation of compound 3

To a solution of 1-methylspiro[indoline-3,2'-oxiran]-2-one **1a** (87.5 mg, 0.5 mmol) in acetone **2a** (5 mL) was slowly added $Sc(OTf)_3$ (24.6 mg，0.075 mmol) at room temperature. Then the mixture was warmed up to 60 °C and stirred for 10 min. After the reaction was complete, the solution was concentrated under reduced pressure. The residue was subjected to purification on silica gel chromatography (eluant: petroleum ether/ethyl acetate) to provide **3a** as product.

(b) General procedure for the formation of compound 5

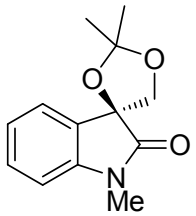
To a solution of 1-methylspiro[indoline-3,2'-oxiran]-2-one **1a** (87.5 mg, 0.5 mmol) and benzaldehyde **4a** (53.0 mg, 0.5 mmol) in 5 mL toluene was slowly added $Sc(OTf)_3$ (24.6 mg，0.075 mmol) at room temperature. Then the mixture was warmed up to 100 °C and stirred for several hours. After the reaction was complete, the solution was concentrated under reduced pressure. The residue was subjected to purification on silica gel chromatography (eluant: petroleum ether/ethyl acetate) to provide **5a** as product.

General procedure for the preparation of spiro-epoxyoxindole 1

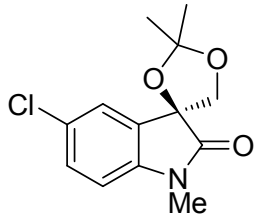


The solution of trimethyl sulfoxonium iodide (220 mg，1 mmol) and Cs_2CO_3 (652 mg，2 mmol) in CH_3CN (15 mL) was stirred for 1h at 50 °C under nitrogen. Then the mixture was added a solution of indoline-2,3-dione species. Stirring was continued monitored by TLC. The reaction mixture was filtered, and concentrated under reduced pressure. The residue was subjected to purification on silica gel chromatography (eluant: petroleum ether/ethyl acetate) to provide spiro[indoline-3,2'-oxiran]-2-one species 1 (50%~60% yield).

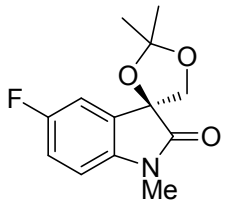
3 Characterization Data



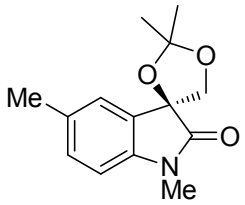
1',2,2-trimethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3a): White solid; melting point: 71-72 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.37 (dd, 1H, *J* = 7.5, 0.5 Hz), 7.34-7.30 (m, 1H), 7.09 (dt, 1H, *J* = 7.5, 0.5 Hz), 6.80 (d, 1H, *J* = 8.0 Hz), 4.34 (d, 1H, *J* = 9.0 Hz), 4.16 (d, 1H, *J* = 9.0 Hz), 3.17 (s, 3H), 1.67 (s, 3H), 1.63(s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 175.2, 143.8, 130.2, 129.0, 124.1, 123.5, 112.8, 108.5, 81.0, 73.0, 27.0, 26.5, 25.9; HRMS: Calcd for $C_{13}H_{15}NNaO_3$ $[M+Na]^+$ 256.0950, Found 256.0937.



5'-chloro-1',2,2-trimethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3b): White solid; m.p.: 130-131°C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.31 (d, 1H, *J* = 2.0 Hz), 7.28 (dd, 1H, *J* = 8.5, 2.0 Hz), 6.72 (d, 1H, *J* = 8.5 Hz), 4.32 (d, 1H, *J* = 9.0 Hz), 4.14 (d, 1H, *J* = 8.5 Hz), 3.15 (s, 3H), 1.65 (s, 3H), 1.62 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 174.8, 142.3, 130.8, 130.0, 128.8, 124.6, 113.1, 109.5, 80.8, 73.0, 27.0, 26.6, 25.7; HRMS: Calcd for $C_{13}H_{14}ClNNaO_3$ $[M+Na]^+$ 290.0560, Found 290.0558.

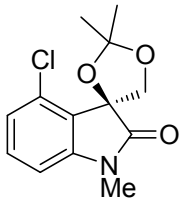


5'-fluoro-1',2,2-trimethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3c): Rufous solid; m.p.: 89-91 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.11 (dd, 1H, *J* = 7.5, 2.5 Hz), 7.02 (m, 1H), 6.73 (dd, 1H, *J* = 8.5, 4.0 Hz), 4.33 (d, 1H, *J* = 9.0 Hz), 4.15 (d, 1H, *J* = 8.5 Hz), 3.17 (s, 3H), 1.66 (s, 3H), 1.63 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 174.9, 160.7, 158.8, 139.63, 139.61, 130.9, 130.8, 116.4, 116.2, 113.1, 112.3, 112.1, 111.0, 109.2, 109.1, 81.0, 73.0, 27.0, 26.6, 25.7; HRMS: Calcd for $C_{13}H_{14}FNNaO_3$ $[M+Na]^+$ 274.0855, Found 274.0861.

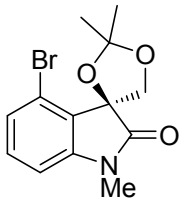


1',2,2,5'-tetramethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3d): Deepred solid; m.p.: 86-88 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.18 (t, 1H, *J* = 0.5 Hz), 7.12-7.10 (m, 1H), 6.69 (d, 1H, *J* = 8.0 Hz), 4.33 (d, 1H, *J* = 9.0 Hz), 4.15 (d, 1H, *J* = 8.5 Hz), 3.16 (s, 3H), 2.34 (s, 3H), 1.68 (s, 3H), 1.64(s, 3H); ^{13}C

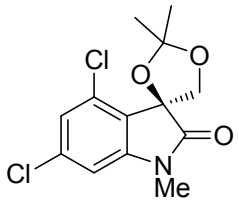
NMR (125 MHz, $CDCl_3$): δ (ppm) = 175.3, 141.4, 133.2, 130.4, 129.0, 124.9, 112.7, 108.3, 81.1, 73.0, 27.1, 26.5, 25.9, 21.2; HRMS: Calcd for $C_{14}H_{17}NNaO_3$ $[M+Na]^+$ 270.1106, Found 270.1106.



4'-chloro-1',2,2-trimethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3e): Yellowgreen oil; ^1H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.22 (t, 1H, *J* = 8.0 Hz), 6.99 (dd, 1H, *J* = 8.0, 0.5 Hz), 6.68 (dd, 1H, *J* = 8.0, 0.5 Hz), 4.47 (d, 1H, *J* = 9.0 Hz), 4.30 (d, 1H, *J* = 9.0 Hz), 3.13 (s, 3H), 1.69 (s, 3H), 1.66 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 175.1, 145.9, 131.6, 131.4, 124.5, 123.8, 113.9, 107.0, 81.5, 70.0, 26.6, 26.5, 26.2; HRMS: Calcd for $C_{13}H_{14}ClNNaO_3$ $[M+Na]^+$ 290.0560, Found 290.0558.

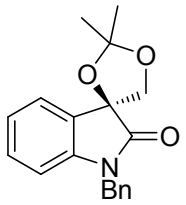


4'-bromo-1',2,2-trimethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3f): Yellowgreen oil; ^1H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.19-7.13 (m, 2H), 6.72 (d, 1H, *J* = 7.0 Hz), 4.50 (d, 1H, *J* = 8.5 Hz), 4.30 (d, 1H, *J* = 8.5 Hz), 3.13 (s, 3H), 1.72 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) =175.1, 146.1, 131.5, 127.8, 125.4, 119.7, 114.1, 107.5, 82.1, 69.8, 29.7, 26.6, 26.5, 26.4; HRMS: Calcd for $C_{13}H_{14}BrNNaO_3$ $[M+Na]^+$ 334.0055, Found 334.0051.

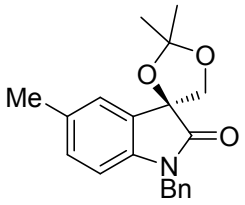


4',6'-dichloro-1',2,2-trimethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3g): White solid; m.p.: 98-99 °C; ^1H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.04 (dd, 1H, *J* = 5.0, 2.0 Hz), 6.71 (dd, 1H, *J* = 3.0, 2.0 Hz), 4.47-4.45 (m, 1H), 4.33-4.30 (m, 1H), 3.15 (s, 3H), 1.69 (d, 3H, *J* = 3.5 Hz), 1.67 (d, 3H, *J* = 3.5 Hz); ^{13}C

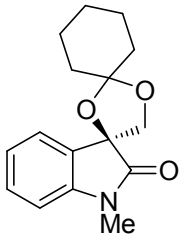
NMR (125 MHz, $CDCl_3$): δ (ppm) = 175.2, 146.7, 136.8, 132.3, 124.1, 122.5, 114.3, 108.0, 81.2, 70.1, 26.8, 26.5, 26.2; HRMS: Calcd for $C_{13}H_{13}Cl_2NNaO_3$ $[M+Na]^+$ 324.0170, Found 324.0173.



1'-benzyl-2,2-dimethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3h): White solid; m.p.: 86-88 °C; ^1H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.39-7.23 (m, 7H), 7.07 (t, 1H, *J* = 7.5 Hz), 6.79 (d, 1H, *J* = 8.0 Hz), 4.86 (d, 2H, *J* = 3.0 Hz), 4.32 (d, 1H, *J* = 9.5 Hz), 4.12 (d, 1H, *J* = 9.0 Hz), 1.61 (s, 3H), 1.60 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 176.3, 144.1, 137.3, 131.0, 129.9, 129.7, 128.7, 128.2, 125.1, 124.2, 113.5, 110.5, 82.0, 73.8, 44.3, 27.1, 26.4; HRMS: Calcd for $C_{19}H_{19}NNaO_3$ $[M+Na]^+$ 332.1263, Found 332.1262.

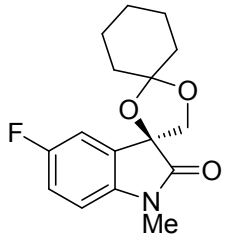


1'-benzyl-2,2,5'-trimethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3i): Rufous oil; ^1H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.32-7.20 (m, 6H), 7.00 (d, 1H, *J* = 8.0 Hz), 6.58 (d, 1H, *J* = 8.0 Hz), 4.85 (s, 2H), 4.41 (d, 1H, *J* = 8.5 Hz), 4.20 (d, 1H, *J* = 9.0 Hz), 2.31 (s, 3H), 1.73 (s, 3H), 1.68 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 175.3, 140.3, 135.5, 133.0, 130.2, 128.8, 127.7, 127.3, 124.7, 112.7, 109.3, 81.1, 73.1, 43.9, 27.0, 25.8, 21.0; HRMS: Calcd for $C_{20}H_{21}NNaO_3$ $[M+Na]^+$ 346.1419, Found 346.1427.

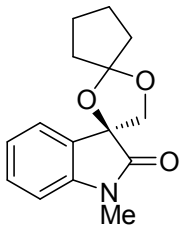


1'-methyl-2,2-cyclohexylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3j): Yellowgreen oil; ^1H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.35 (d, 1H, *J* = 7.5 Hz), 7.30 (dt, 1H, *J* = 7.5, 2.0 Hz), 7.07 (t, 1H, *J* = 7.5 Hz),

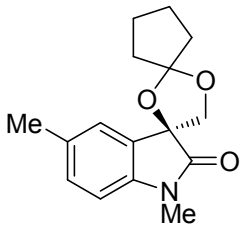
6.79 (d, 2H, J = 8.0 Hz), 4.31 (d, 1H, J = 8.5 Hz), 4.13 (d, 1H, J = 9.0 Hz), 3.16 (s, 3H), 1.96 (t, 2H, J = 6.0 Hz), 1.88-1.80 (m, 2H), 1.70-1.63 (m, 4H), 1.48-1.39 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 175.3, 143.8, 130.1, 129.1, 124.1, 123.4, 113.4, 108.5, 80.6, 72.6, 36.6, 35.2, 26.4, 25.1, 24.2, 23.9; HRMS: Calcd for $C_{16}H_{19}NNaO_3$ $[M+Na]^+$ 296.1263, Found 296.1272.



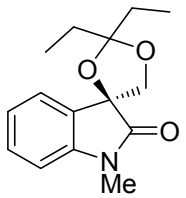
5'-fluoro-1'-methyl-2,2-cyclohexylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3k): White solid; m.p.: 92-94 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.10 (dd, 1H, J = 7.5, 2.5 Hz), 7.01 (dt, 1H, J = 9.0, 2.5 Hz), 6.72 (dd, 1H, J = 8.5, 4.0 Hz), 4.32 (d, 1H, J = 13.5 Hz), 4.12 (d, 1H, J = 14.0 Hz), 3.16 (s, 3H), 1.96-1.94 (m, 2H), 1.87-1.80 (m, 2H), 1.68-1.63 (m, 4H), 1.47-1.42 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 174.9, 160.6, 158.6, 139.5, 139.4, 130.9, 130.8, 116.2, 116.0, 113.6, 112.2, 111.9, 109.0, 108.9, 80.5, 80.4, 72.5, 36.5, 34.8, 26.5, 24.9, 24.1, 23.7; HRMS: Calcd for $C_{16}H_{18}FNNaO_3$ $[M+Na]^+$ 314.1168, Found 314.1170.



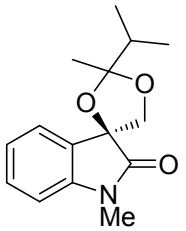
1'-methyl-2,2-cyclopentylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3l): Yellowgreen oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.35-7.33 (m, 1H), 7.29 (dt, 1H, J = 8.0, 2.0 Hz), 7.06 (t, 1H, J = 7.5 Hz), 6.78 (d, 1H, J = 8.0 Hz), 4.21(d, 1H, J = 9.0 Hz), 4.10 (d, 1H, J = 8.5 Hz), 3.15 (s, 3H), 2.21-2.16 (m, 1H), 2.07-1.95 (m, 3H), 1.77-1.69 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 175.0, 143.7, 130.1, 129.1, 124.0, 123.4, 122.5, 108.5, 80.2, 73.2, 36.9, 36.8, 26.4, 23.9, 23.5; HRMS: Calcd for $C_{15}H_{17}NNaO_3$ $[M+Na]^+$ 282.1106, Found 282.1113.



5'-methyl-1'-methyl-2,2-cyclopentylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3m): Yellowgreen oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.17 (s, 1H), 7.11 (dd, 1H, *J* = 8.0, 0.5 Hz), 6.89 (d, 1H, *J* = 8.0 Hz), 4.23(d, 1H, *J* = 8.5 Hz), 4.11 (d, 1H, *J* = 8.5 Hz), 3.16 (s, 3H), 2.33 (s, 3H), 2.24-2.21 (m, 2H), 2.19-1.98 (m, 4H), 1.77-1.75 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) =174.9, 141.2, 132.9, 130.2, 128.9, 124.6, 122.4, 108.1,1 80.2, 73.1, 36.8, 36.7, 26.4, 23.8, 23.4, 21.0; HRMS: Calcd for $C_{16}H_{19}NNaO_3$ $[M+Na]^+$ 296.1293, Found 296.1298.

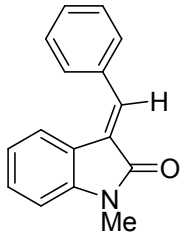


2,2-diethyl-1'-methylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3n): Yellowgreen oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.40 (dd, 1H, *J* = 7.5, 0.5 Hz), 7.32 (dt, 1H, *J* = 7.5, 1.5 Hz), 7.09 (dt, 1H, *J* = 7.5, 0.5 Hz), 6.80 (d, 1H, *J* = 8.0 Hz), 4.34 (d, 1H, *J* = 9.0 Hz), 3.18 (s, 3H), 2.05-1.99 (m, 2H), 1.91-1.87 (m, 2H), 1.04 (t, 3H, *J* = 7.5 Hz), 0.99 (t, 3H, *J* = 7.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 175.4, 143.9, 130.1, 129.3, 124.3, 123.4, 116.9, 108.5, 80.7, 72.9, 29.0, 28.6, 26.5, 8.8, 8.5; HRMS: Calcd for $C_{15}H_{19}NNaO_3$ $[M+Na]^+$ 284.1263, Found 284.1252.

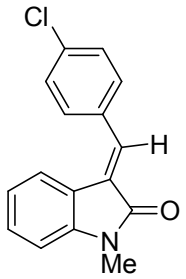


2-isopropyl-1',2-dimethylspiro[[1,3]dioxolane-4,3'-indolin]-2'-one (3o): Yellowgreen oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.38 (dd, 1H, *J* = 7.5, 1.0 Hz), 7.33-7.30 (m, 1H), 7.11-7.07 (m, 1H), 6.80 (d, 1H, *J* = 7.5 Hz), 4.36 (d, 0.6H, *J* = 9.0 Hz), 4.32 (d, 0.4H, *J* = 8.5 Hz), 4.16 (d, 0.6H, *J* = 2.0 Hz), 4.14

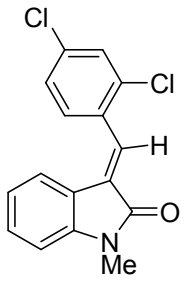
(d, 0.3H, J = 2.5 Hz), 3.18 (s, 3H), 2.48-2.43 (m, 0.3H), 2.11-2.06 (m, 0.6H), 1.58 (s, 1.8H), 1.48 (s, 1.2H), 1.07 (dd, 4H, J = 10.0, 2.0 Hz), 1.03 (dd, 2H, J = 7.0, 2.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 175.6, 175.2, 143.9, 143.8, 130.2, 130.1, 129.3, 129.2, 124.3, 124.0, 123.5, 123.4, 117.3, 116.8, 108.53, 108.51, 80.9, 80.8, 73.0, 72.9, 37.5, 35.5, 26.5, 19.6, 19.3, 18.2, 18.0, 17.9; HRMS: Calcd for $C_{15}H_{19}NNaO_3$ $[M+Na]^+$ 284.1263, Found 284.1256.



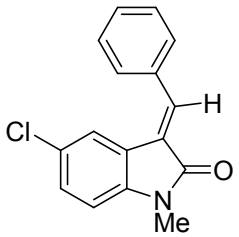
(*E*)-3-benzylidene-1-methylindolin-2-one (5a): [1],[2] Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.86 (s, 1H), 7.65-7.62 (m, 3H), 7.48-7.42 (m, 3H), 7.28 (d, 1H, J = 7.5 Hz), 6.88 (t, 1H, J = 7.5 Hz), 6.83 (d, 1H, J = 8.0 Hz), 3.29 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 168.5, 144.3, 137.2, 135.0, 129.8, 129.5, 129.3, 128.7, 127.3, 122.8, 121.8, 121.2, 108.2, 26.2.



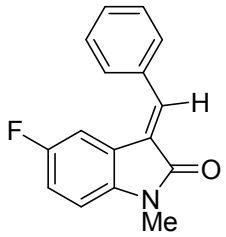
(*E*)-3-(4-chlorobenzylidene)-1-methylindolin-2-one (5b): Yellow solid; m.p.: 115-116 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.77 (s, 1H), 7.59-7.56 (m, 3H), 7.44 (d, 2H, J = 8.5 Hz), 7.28 (t, 1H, J = 7.5 Hz), 6.89 (t, 1H, J = 7.5 Hz), 6.84 (d, 1H, J = 7.5 Hz), 3.28 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 168.3, 144.4, 135.5, 135.4, 133.4, 130.6, 130.1, 128.9, 127.7, 122.7, 121.9, 120.9, 108.3, 26.2. HRMS: Calcd for $C_{16}H_{13}ClNO$ $[M+H]^+$ 270.0686, Found 270.0685.



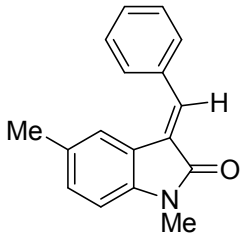
(*E*)-3-(2,4-dichlorobenzylidene)-1-methylindolin-2-one (5c): Yellow solid; m.p.: 155-157 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.78 (s, 1H), 7.62 (t, 1H, *J* = 7.5 Hz), 7.53 (s, 1H), 7.34-7.27 (m, 3H), 6.86 (t, 1H, *J* = 7.5 Hz), 6.83 (d, 1H, *J* = 8.0 Hz), 3.28 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 167.7, 144.6, 135.8, 135.3, 132.2, 132.1, 130.9, 130.5, 129.9, 129.2, 127.1, 122.9, 121.9, 120.6, 108.4, 26.2. HRMS: Calcd for $C_{16}H_{12}Cl_2NO$ $[M+H]^+$ 304.0296, Found 304.0290.



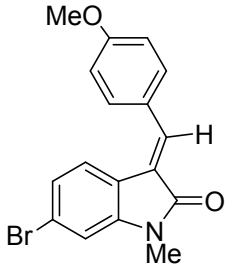
(*E*)-3-benzylidene-5-chloro-1-methylindolin-2-one (5d): [1] Yellow solid; m.p.: 137-139 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.91 (s, 1H), 7.63-7.59 (m, 3H), 7.51-7.46 (m, 3H), 7.24 (dd, 1H, *J* = 8.5, 2.0 Hz), 6.75 (d, 1H, *J* = 8.0 Hz), 3.27 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 168.1, 142.7, 138.9, 134.5, 130.0, 129.4, 129.3, 128.9, 127.2, 126.4, 122.8, 122.5, 109.0, 26.3.



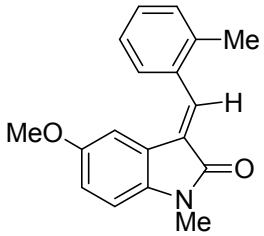
(*E*)-3-benzylidene-5-fluoro-1-methylindolin-2-one (5e): [1] Yellow solid; m.p.: 103-105 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.91 (s, 1H), 7.61 (d, 2H, *J* = 7.0 Hz), 7.51-7.45 (m, 3H), 7.34 (dd, 1H, *J* = 9.0, 2.5 Hz), 6.98 (dt, 1H, *J* = 9.0, 2.5 Hz), 6.74 (dd, 1H, *J* = 8.5, 4.5 Hz), 3.28 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 168.3, 159.4, 157.6, 138.7, 134.5, 129.9, 129.2, 128.8, 128.4, 127.0, 126.9, 126.7, 122.1, 122.0, 115.9, 115.8, 110.6, 110.4, 108.5, 108.4, 26.3.



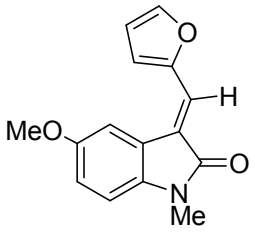
(*E*)-3-benzylidene-1,5-dimethylindolin-2-one (5f):[1] Yellow solid; m.p.: 78-80 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.83 (s, 1H), 7.65 (d, 1H, *J* = 7.0 Hz), 7.49-7.43 (m, 4H), 7.08 (d, 1H, *J* = 8.0 Hz), 6.72 (d, 1H, *J* = 8.0 Hz), 3.27 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 168.6, 142.1, 136.9, 135.1, 131.1, 130.2, 129.5, 129.3, 128.6, 127.4, 123.5, 121.2, 107.9, 26.2, 21.2.



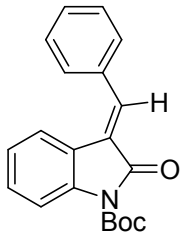
(*E*)-6-bromo-3-(4-methoxybenzylidene)-1-methylindolin-2-one (5g): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.80 (s, 1H), 7.61-7.56 (m, 3H), 7.02-6.95 (m, 4H), 3.87 (s, 3H), 3.24 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 168.5, 160.9, 145.0, 138.1, 131.4, 126.9, 124.4, 124.2, 123.3, 122.8, 120.3, 114.1, 111.5, 55.4, 26.2. HRMS: Calcd for $C_{17}H_{14}BrNNaO_2$ $[M+Na]^+$ 366.0106, Found 366.0103.



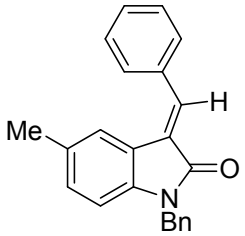
(*E*)-5-methoxy-1-methyl-3-(2-methylbenzylidene)indolin-2-one (5h): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.89 (s, 1H), 7.52 (d, 1H, *J* = 9.5 Hz), 7.32-7.21 (m, 3H), 6.84 (d, 1H, *J* = 3.5 Hz), 6.75 (dd, 1H, *J* = 11.5, 3.5 Hz), 6.66 (d, 1H, *J* = 11.5 Hz), 3.57 (s, 3H), 3.22 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 167.7, 154.9, 137.8, 137.0, 136.6, 133.8, 130.3, 129.3, 128.2, 128.0, 125.5, 121.8, 114.2, 109.5, 108.1, 55.3, 25.9, 19.7. HRMS: Calcd for $C_{18}H_{17}NNaO_2$ $[M+Na]^+$ 302.1157, Found 302.1165.



(*E*)-3-(furan-2-ylmethylene)-5-methoxy-1-methylindolin-2-one (5i): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 8.06 (d, 1H, *J* = 3.0 Hz), 7.71 (d, 1H, *J* = 2.0 Hz), 7.41 (s, 1H), 6.86 (d, 1H, *J* = 4.5 Hz), 6.80 (dd, 1H, *J* = 10.5, 3.5 Hz), 6.65 (d, 1H, *J* = 10.5 Hz), 6.58 (dd, 1H, *J* = 4.5, 2.0 Hz), 3.81 (s, 3H), 3.20 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 168.9, 155.3, 145.6, 137.8, 122.5, 122.2, 120.3, 119.8, 113.6, 113.0, 112.1, 107.7, 55.7, 26.1. HRMS: Calcd for $C_{15}H_{13}NNaO_3$ $[M+Na]^+$ 278.0793, Found 278.0790.



(*E*)-tert-butyl 3-benzylidene-2-oxoindoline-1-carboxylate (5j): Yellow solid; m.p.: 113-115 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.94 (d, 1H, *J* = 8.0 Hz), 7.90 (s, 1H), 7.70-7.64 (m, 3H), 7.51-7.46 (m, 3H), 7.33 (t, 1H, *J* = 8.0 Hz), 7.01 (t, 1H, *J* = 7.5 Hz), 1.69 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 166.7, 149.4, 140.1, 138.3, 134.6, 130.1, 129.8, 129.2, 128.7, 126.2, 123.7, 122.4, 121.5, 115.2, 84.2, 28.3. HRMS: Calcd for $C_{20}H_{19}NNaO_3$ $[M+Na]^+$ 344.1263, Found 344.1269.



(*E*)-1-benzyl-3-benzylidene-5-methylindolin-2-one (5k): Yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) = 7.94 (s, 1H), 7.70 (d, 1H, *J* = 7.0 Hz), 7.53-7.45 (m, 4H), 7.36-7.29 (m, 5H), 6.98 (d, 1H, *J* = 9.0 Hz), 6.64 (d, 1H, *J* = 8.0 Hz), 5.01 (s, 2H), 2.22 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) = 168.7,

141.2, 137.3, 136.1, 135.1, 131.2, 130.2, 129.6, 129.3, 128.8, 128.6, 127.5, 127.3, 127.2, 123.5, 121.3, 108.9, 43.8, 21.2. HRMS: Calcd for $C_{23}H_{19}NNaO$ $[M+Na]^+$ 348.1364, Found 348.1365.

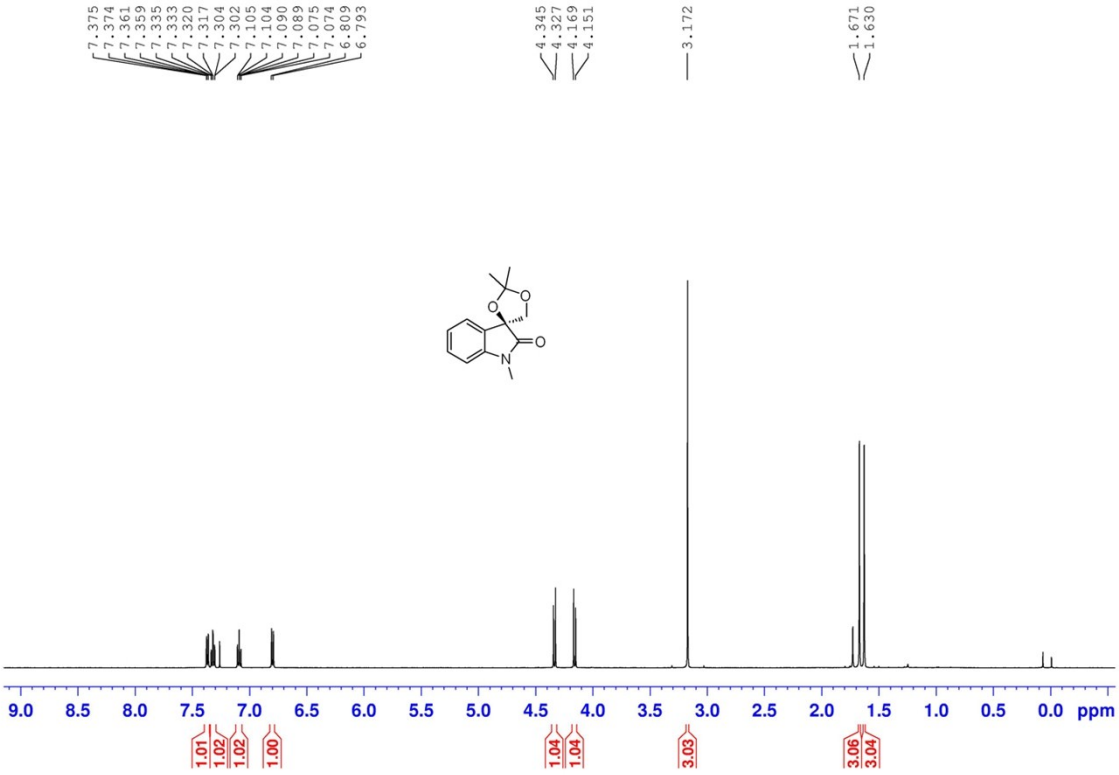
References:

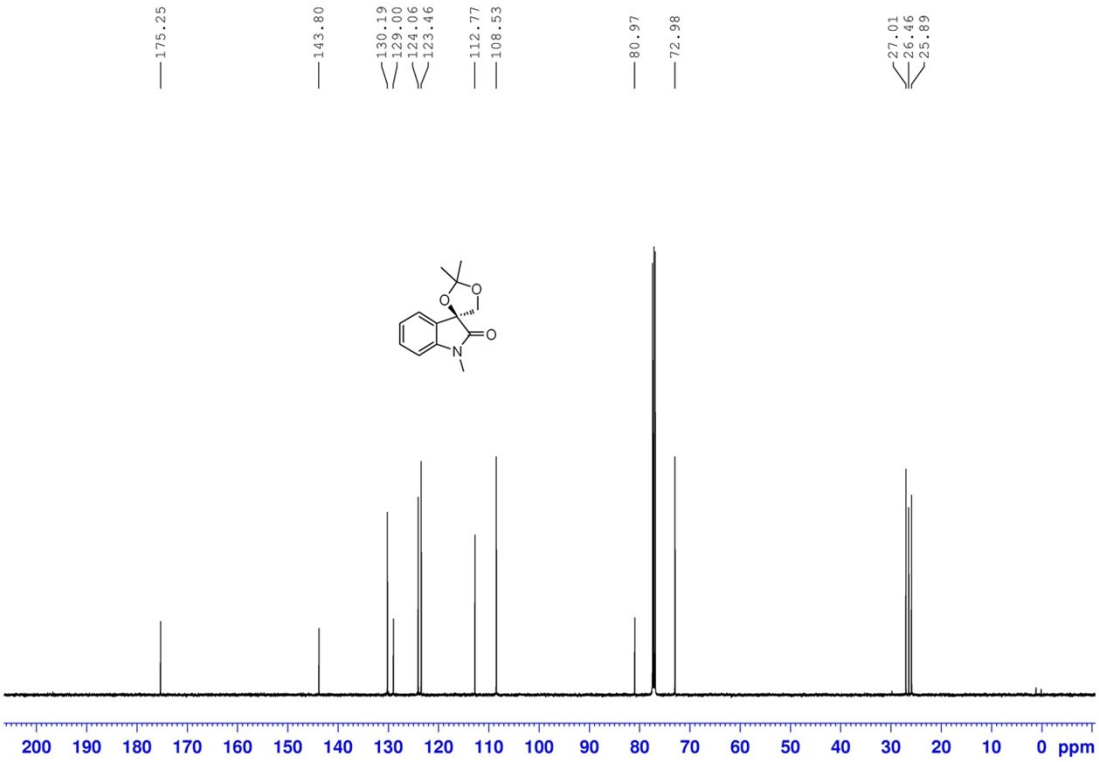
[1] H. Jin, J. Xie, C. Pan, Z. Zhu, Y. Cheng, C. Zhu, *ACS Catal.*, **2013**, *3*, 2195-2198.

[2] A. Teichert, K. Jantos, K. Harms, A. Studer, *Org. Lett.*, **2004**, *6*, 3477-3480.

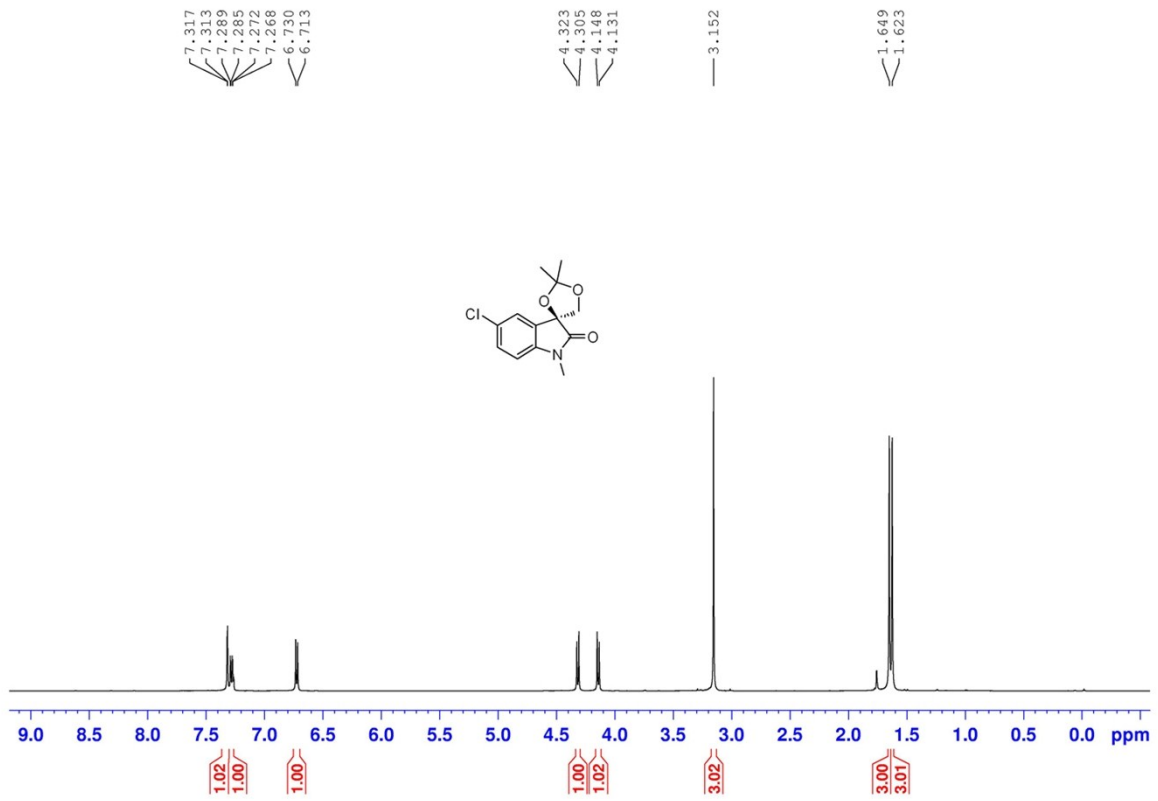
4 ^{1}H NMR and ^{13}C NMR Spectra of All Compounds

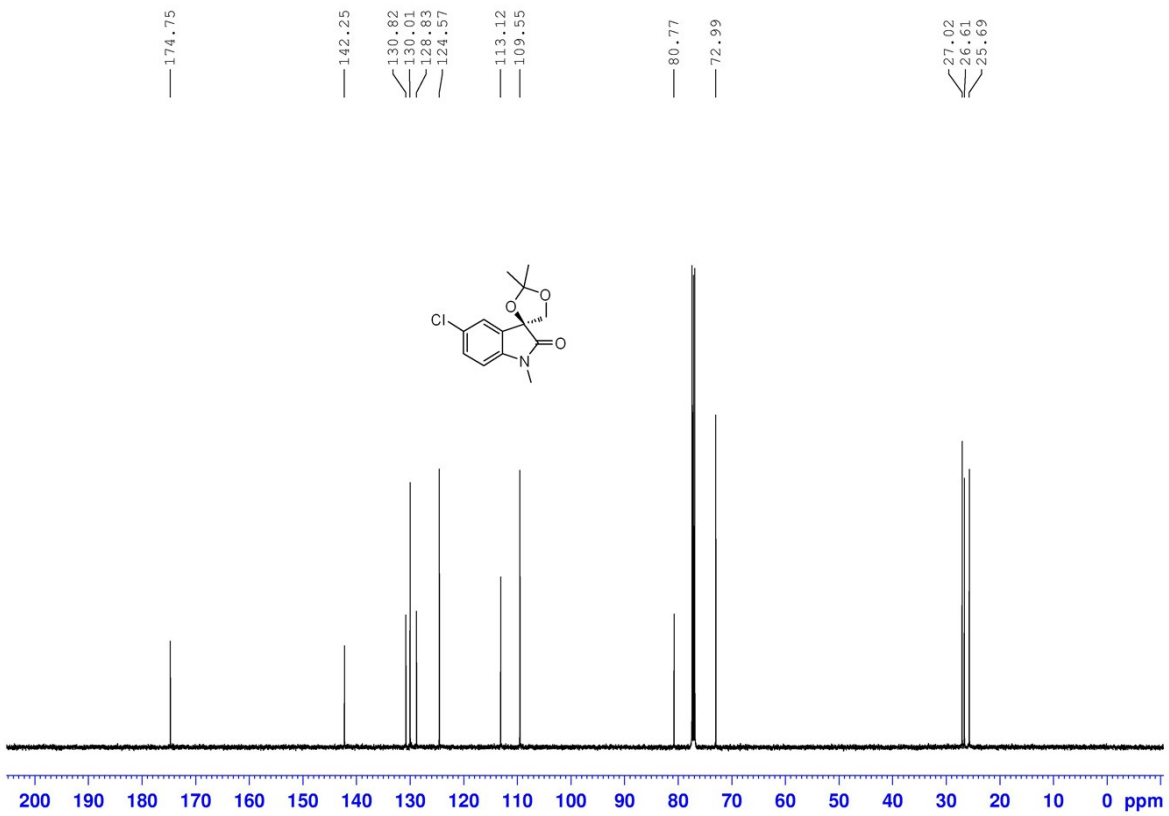
Compound **3a**



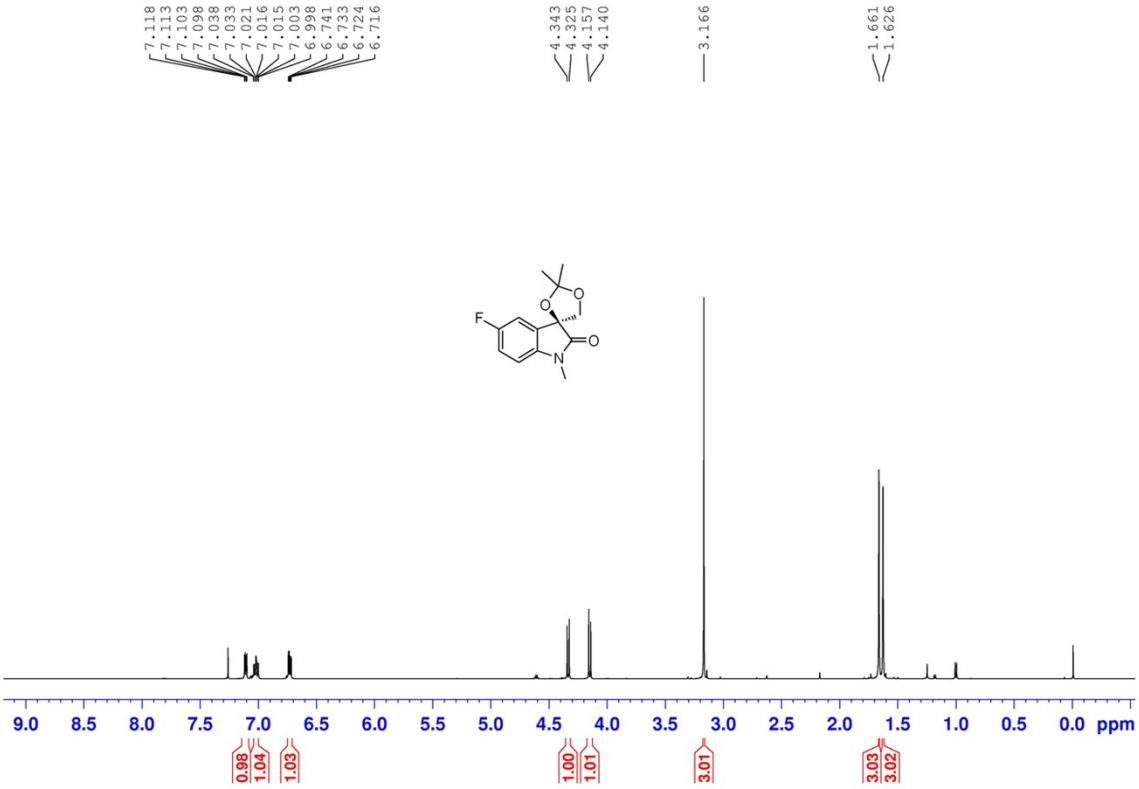


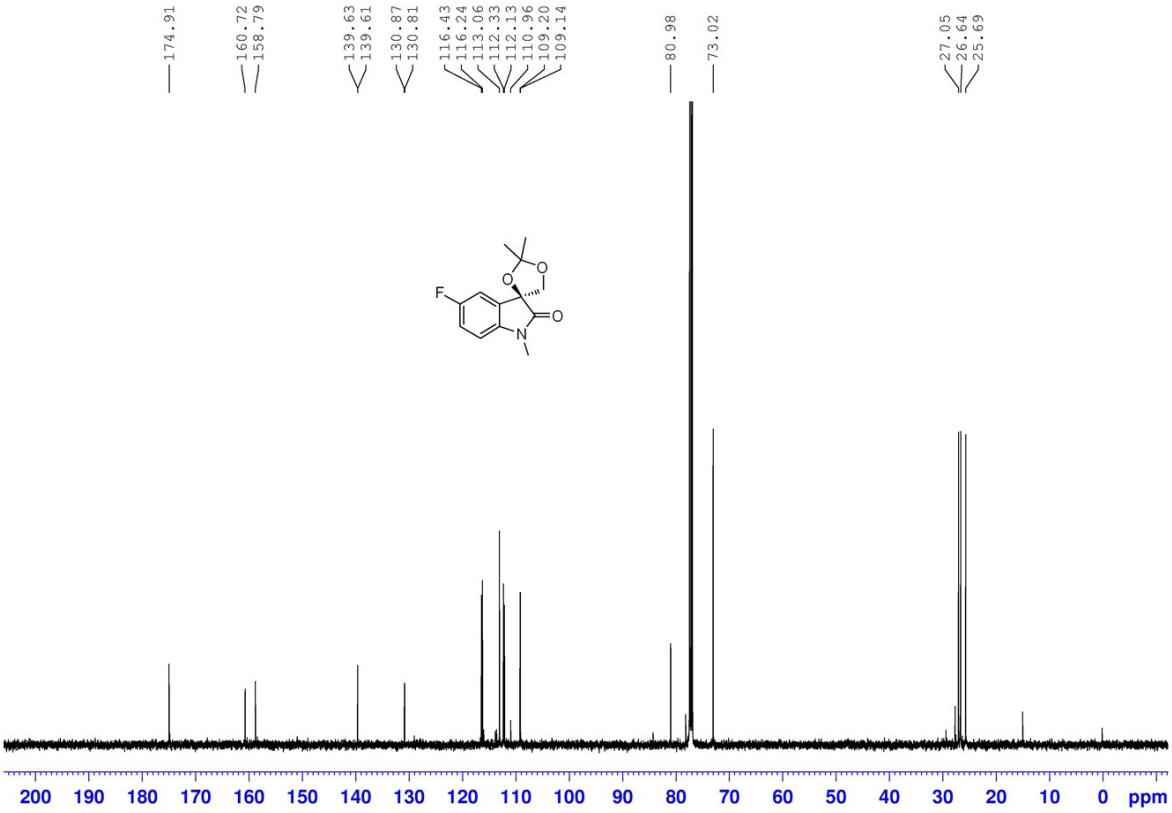
Compound **3b**


7.317
7.313
7.289
7.285
7.272
7.268
6.730
6.713
4.323
4.305
4.148
4.131
3.152
1.649
1.623
Cl
O
O
O
N
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.02
1.00
1.00
1.00
1.02
3.02
3.00
3.01

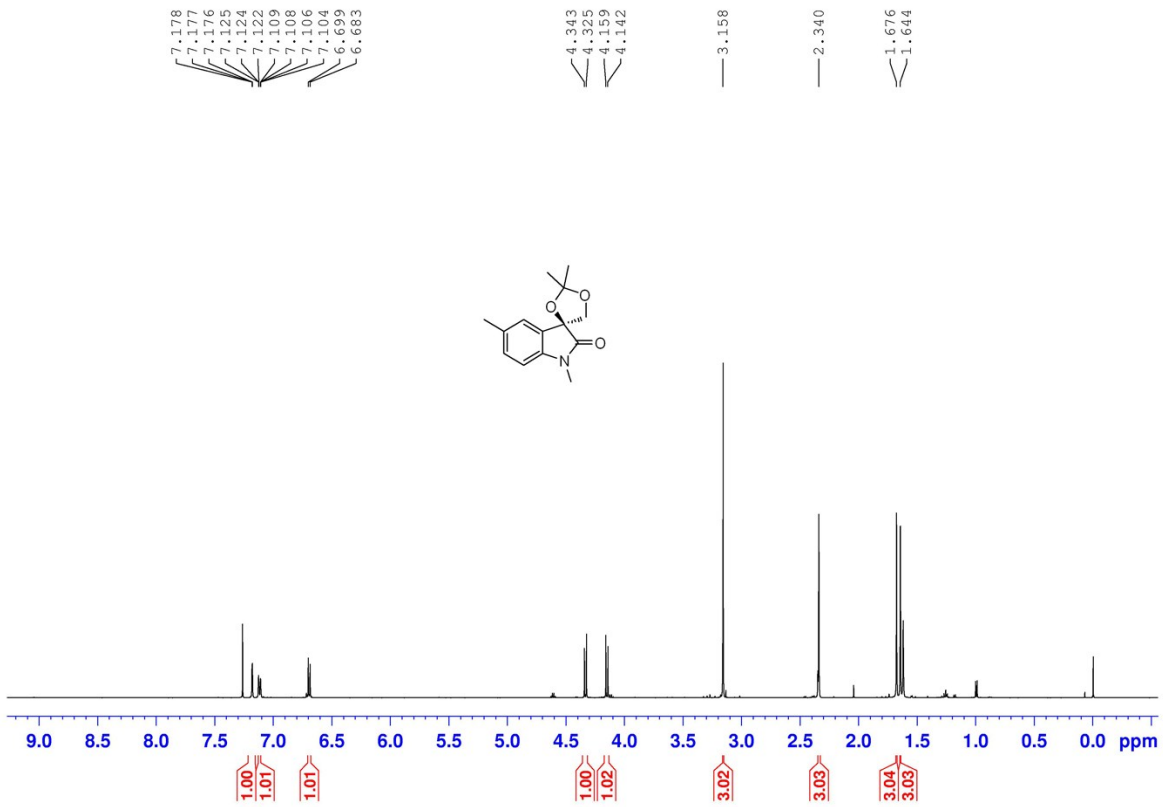

174.75
142.25
130.82
130.01
128.83
124.57
113.12
109.55
80.77
72.99
27.02
26.61
25.69
Cl
O
O
O
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

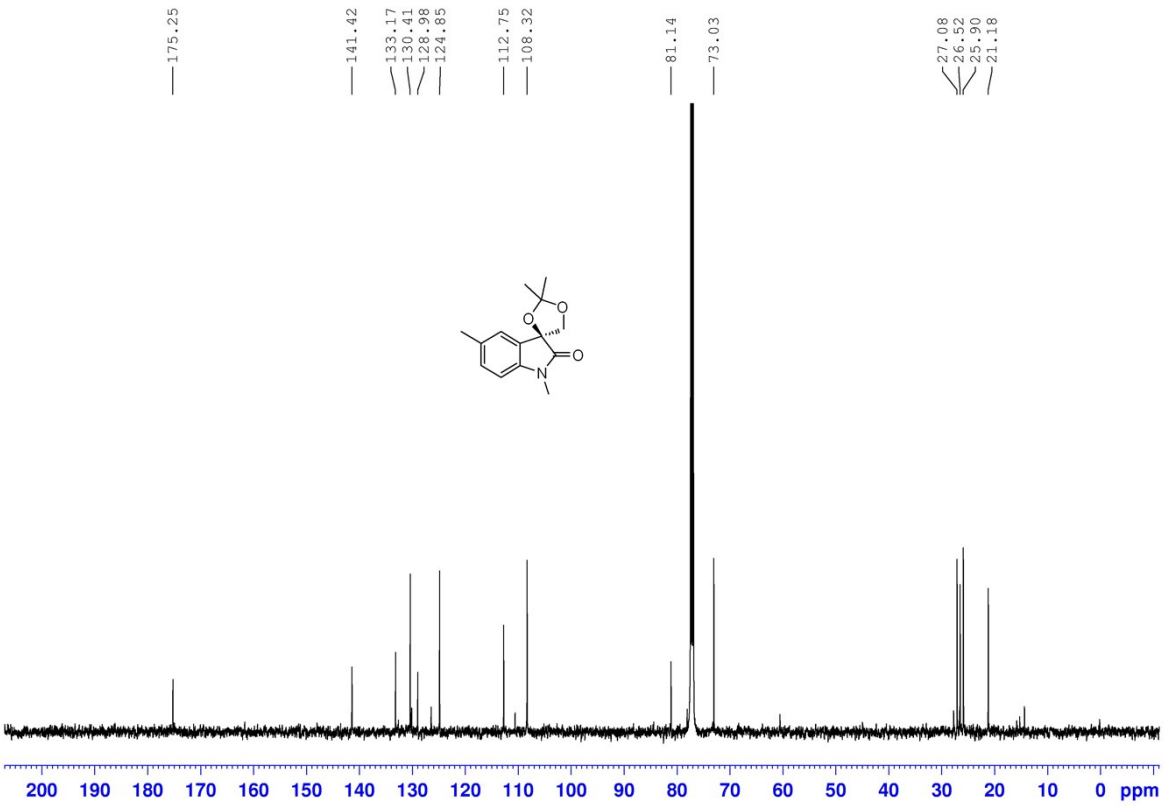
Compound **3c**


7.118
7.113
7.103
7.098
7.038
7.033
7.021
7.016
7.015
7.003
6.998
6.741
6.733
6.724
6.716
4.343
4.325
4.157
4.140
3.166
1.661
1.626
F
O
O
O
N
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
1.04
1.03
1.00
1.01
3.01
3.03
3.02

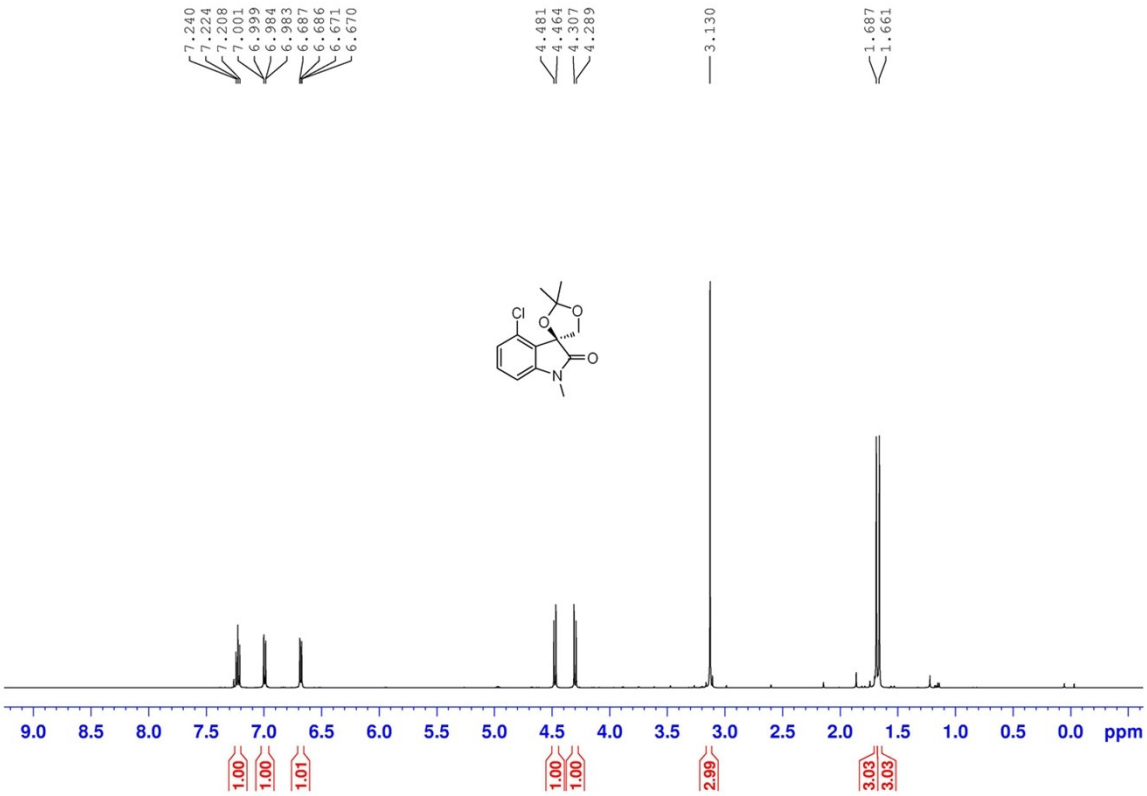

174.91
160.72
158.79
139.63
139.61
130.87
130.81
116.43
116.24
113.06
112.33
112.13
110.96
109.20
109.14
80.98
73.02
27.05
26.64
25.69
F
O
O
O
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

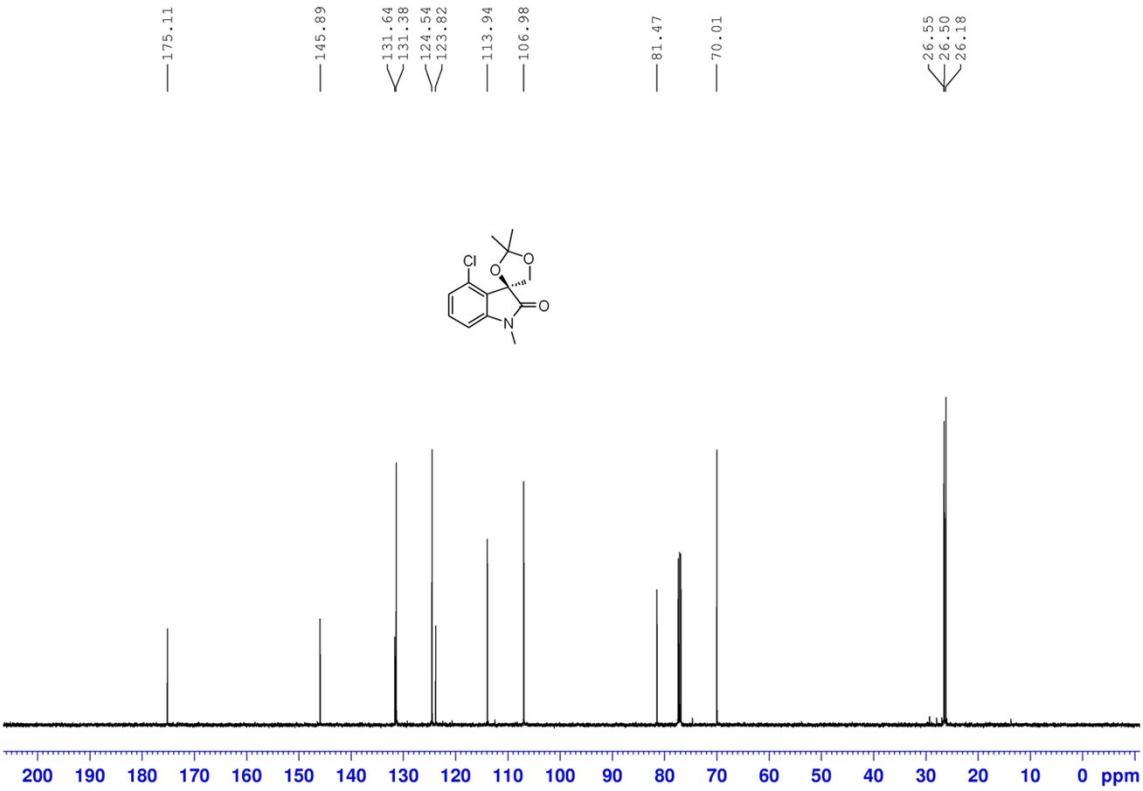
Compound **3d**


7.178
7.177
7.176
7.125
7.124
7.122
7.109
7.108
7.106
7.104
6.699
6.683
4.343
4.325
4.159
4.142
3.158
2.340
1.676
1.644
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.01
1.01
1.00
1.02
3.02
3.03
3.04
3.03

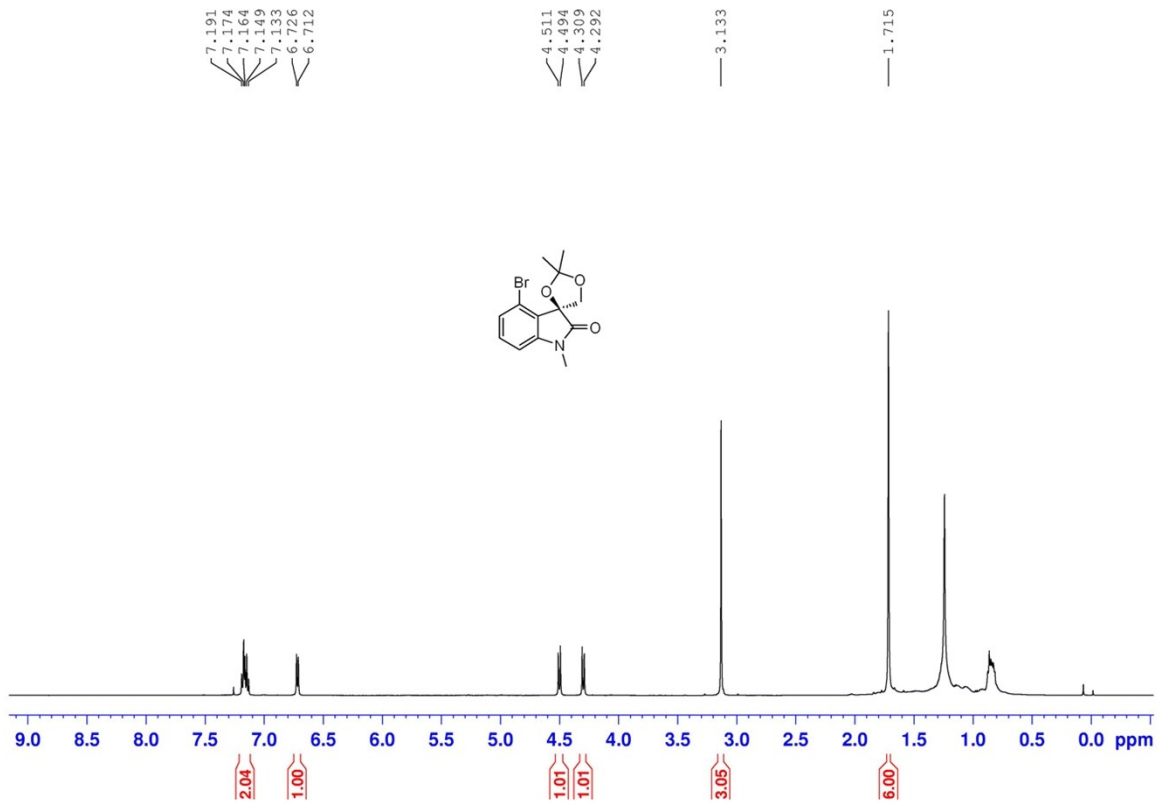

175.25
141.42
133.17
130.41
128.98
124.85
112.75
108.32
81.14
73.03
27.08
26.52
25.90
21.18
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

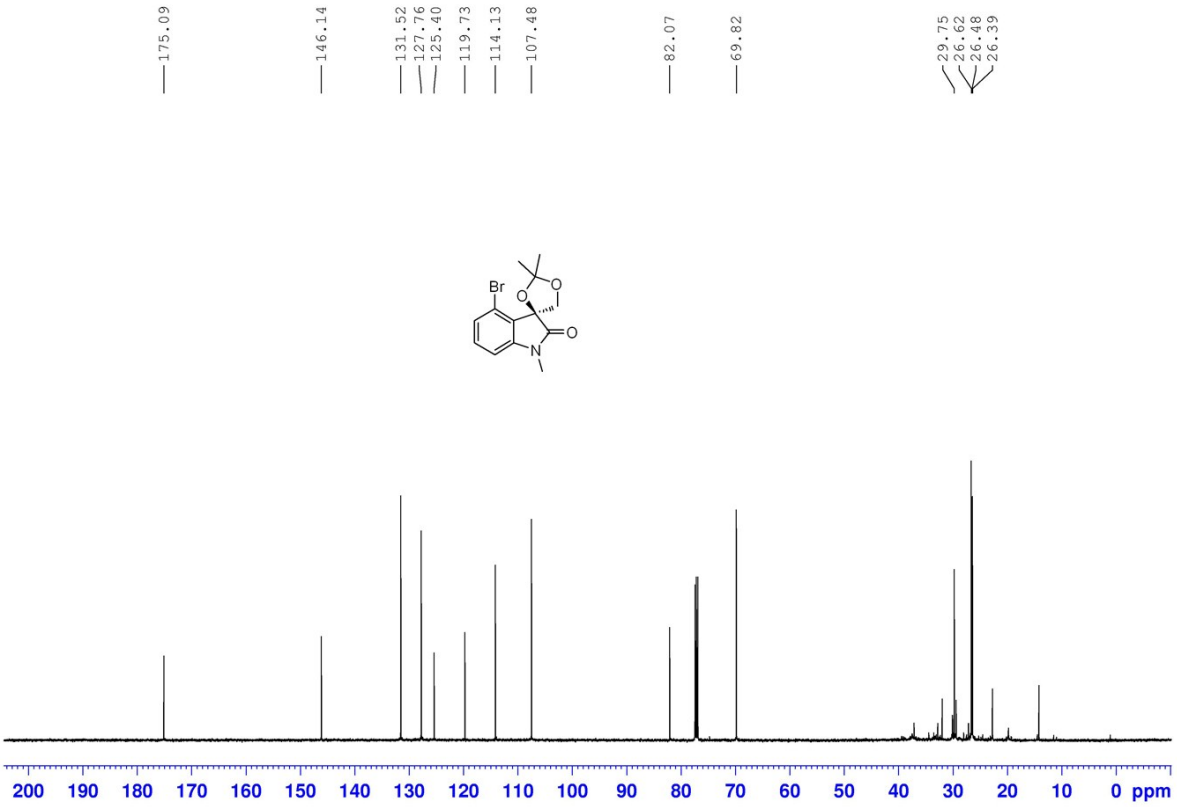
Compound **3e**


7.240
7.224
7.208
7.001
6.999
6.984
6.983
6.687
6.686
6.671
6.670
4.481
4.464
4.307
4.289
3.130
1.687
1.661
Cl
O
O
O
N
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.00
1.01
1.00
1.00
2.99
3.03
3.03

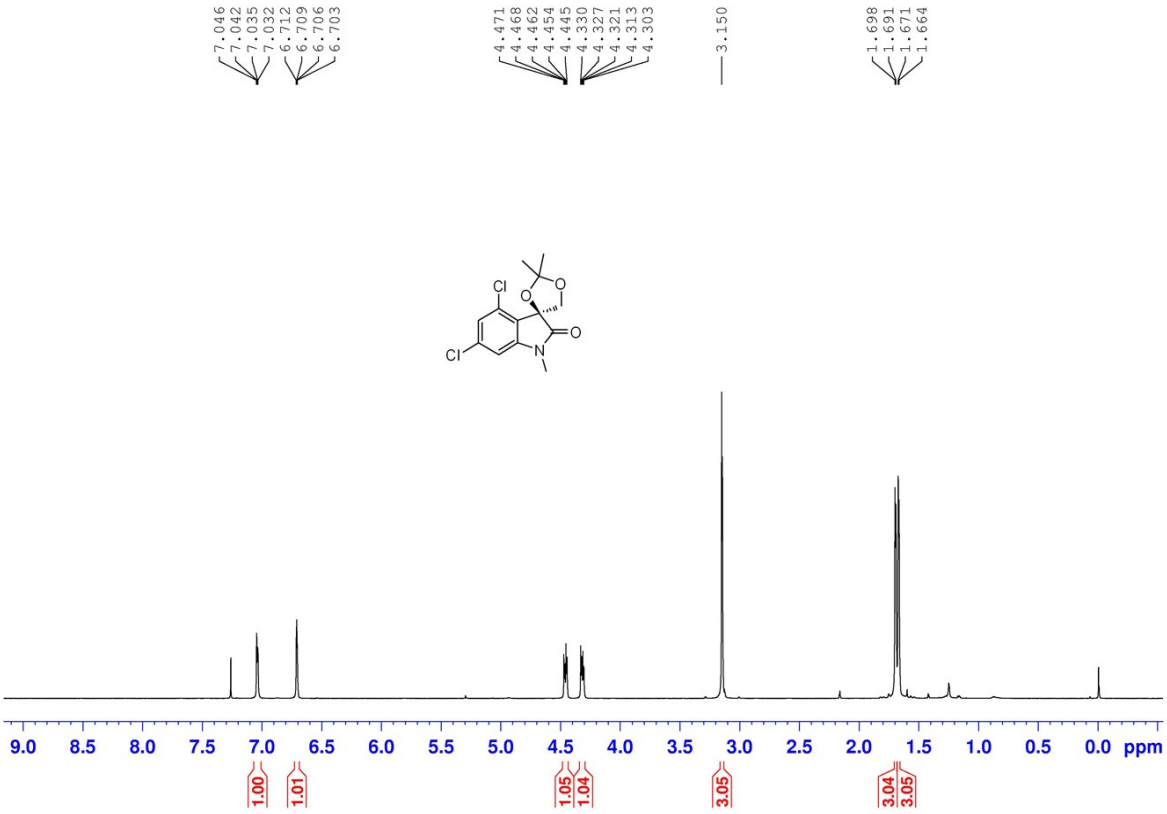

175.11
145.89
131.64
131.38
124.54
123.82
113.94
106.98
81.47
70.01
26.55
26.50
26.18
Cl
O
O
O
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

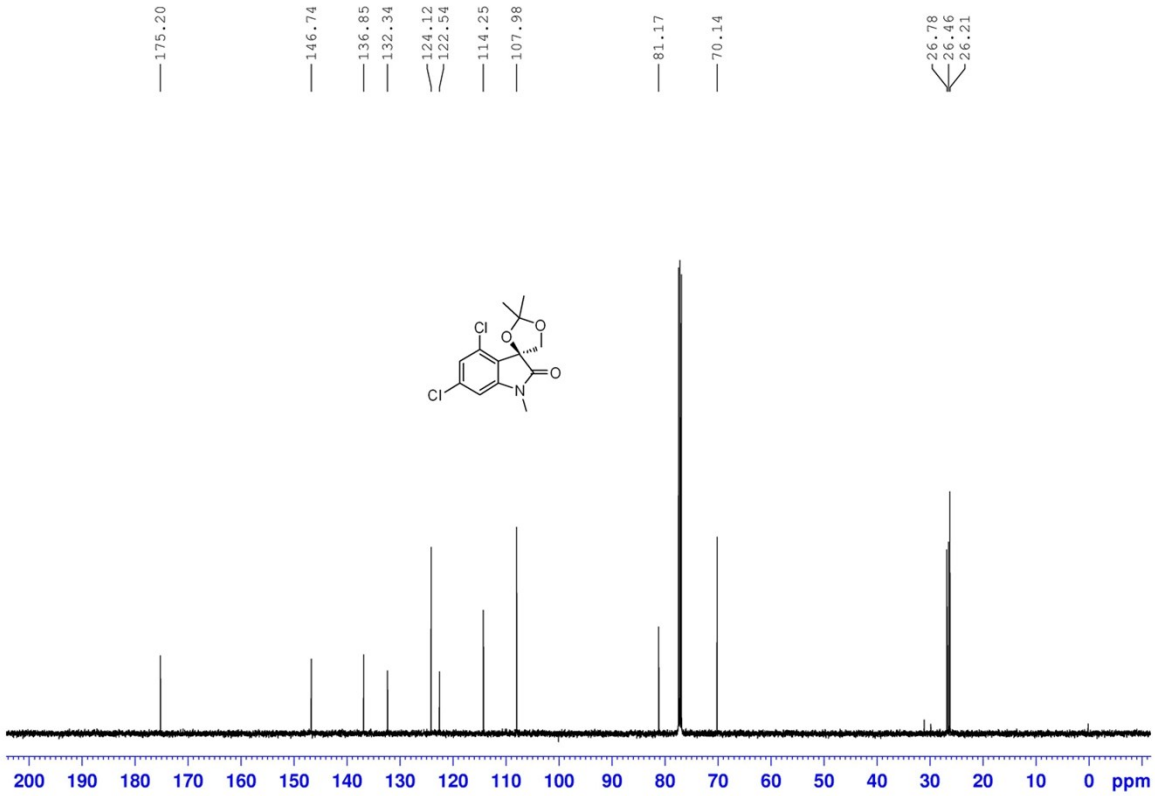
Compound **3f**


7.191
7.174
7.164
7.149
7.133
6.726
6.712
4.511
4.494
4.309
4.292
3.133
1.715
Br
O
O
O
N
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.04
1.00
1.01
1.01
3.05
6.00

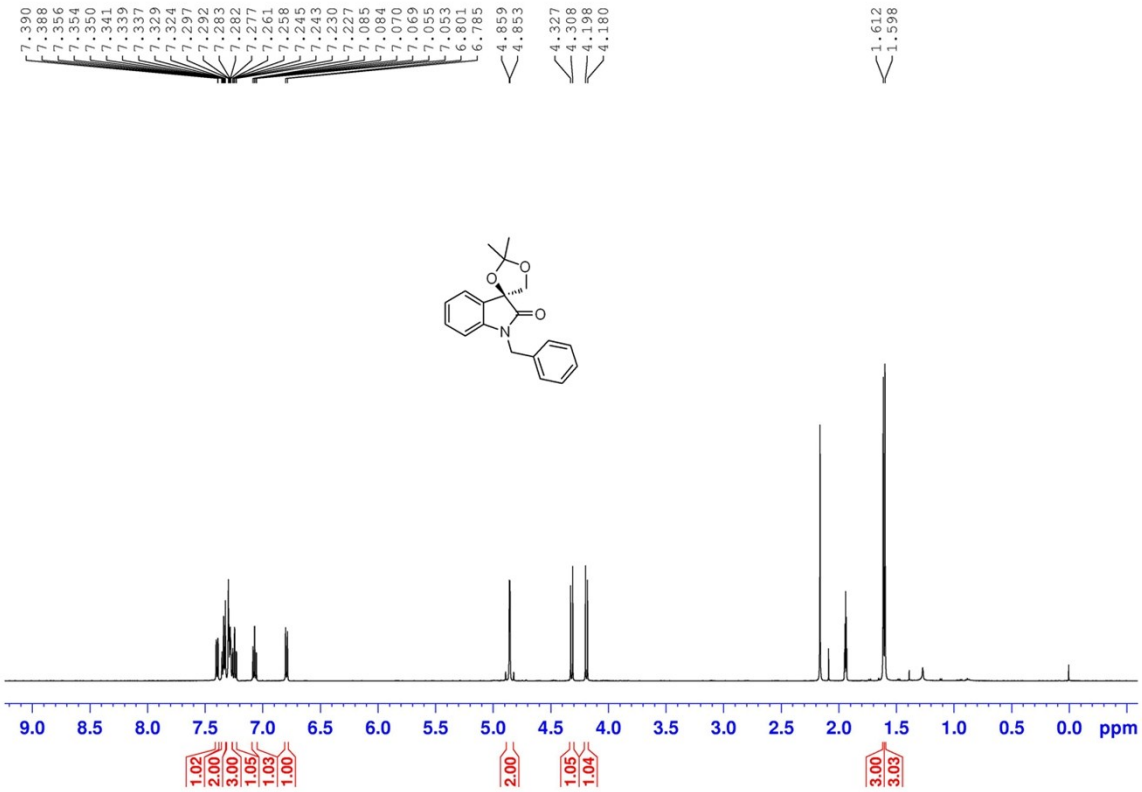

175.09
146.14
131.52
127.76
125.40
119.73
114.13
107.48
82.07
69.82
29.75
26.62
26.48
26.39
Br
O
O
O
N
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

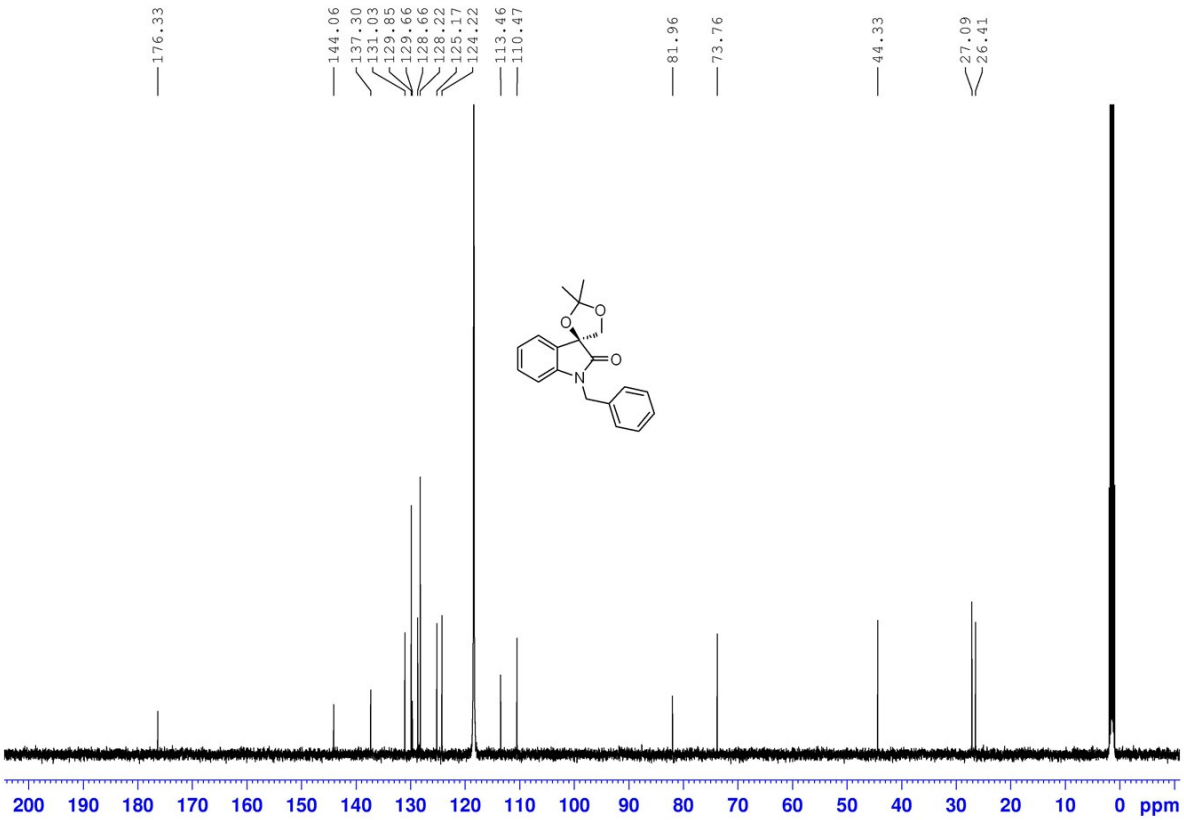
Compound **3g**


7.046
7.042
7.035
7.032
6.712
6.709
6.706
6.703
4.471
4.468
4.462
4.454
4.445
4.330
4.327
4.321
4.313
4.303
3.150
1.698
1.691
1.671
1.664
Cl
O
O
O
N
Cl
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.01
1.05
1.04
3.05
3.04
3.05

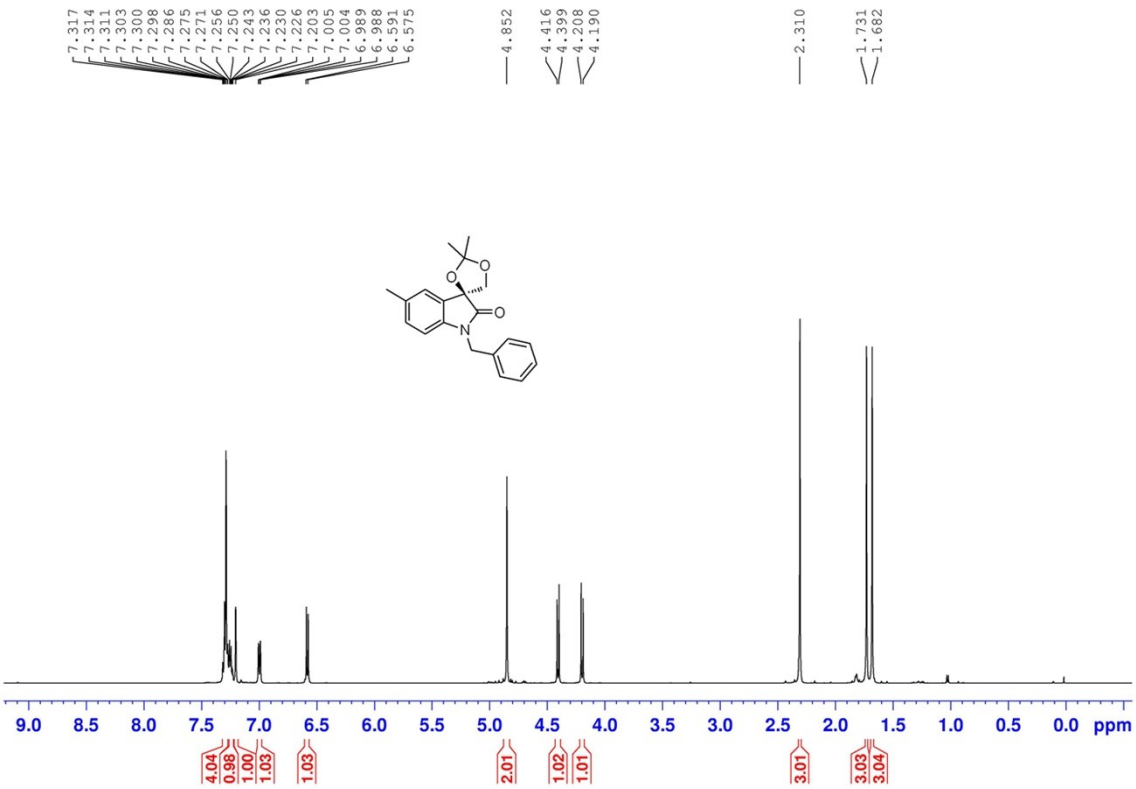

175.20
146.74
136.85
132.34
124.12
122.54
114.25
107.98
81.17
70.14
26.78
26.46
26.21
Cl
O
O
O
N
Cl
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

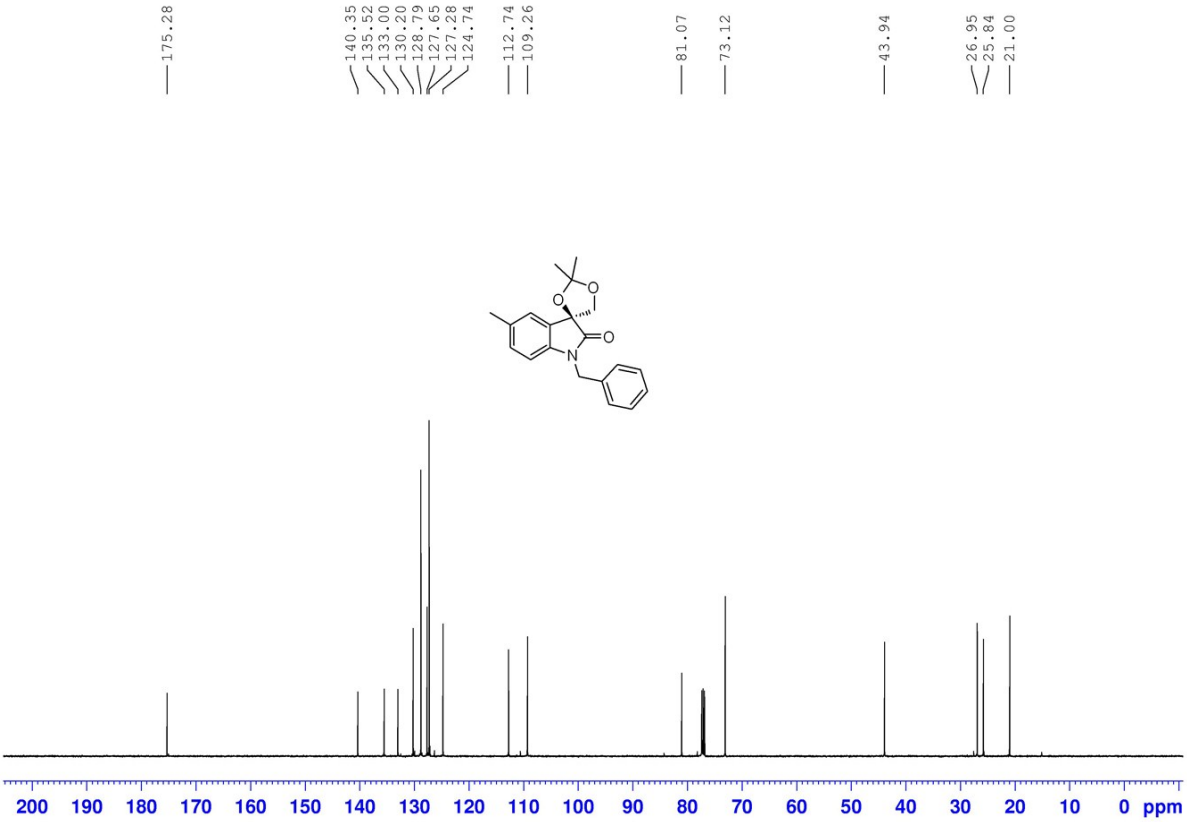
Compound **3h**


7.390
7.388
7.356
7.354
7.350
7.341
7.339
7.337
7.329
7.324
7.297
7.292
7.283
7.282
7.277
7.261
7.258
7.245
7.243
7.230
7.227
7.085
7.084
7.070
7.069
7.055
7.053
6.801
6.785
4.859
4.853
4.327
4.308
4.198
4.180
1.612
1.598
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.02
2.00
3.00
1.05
1.03
1.00
2.00
1.05
1.04
3.00
3.03

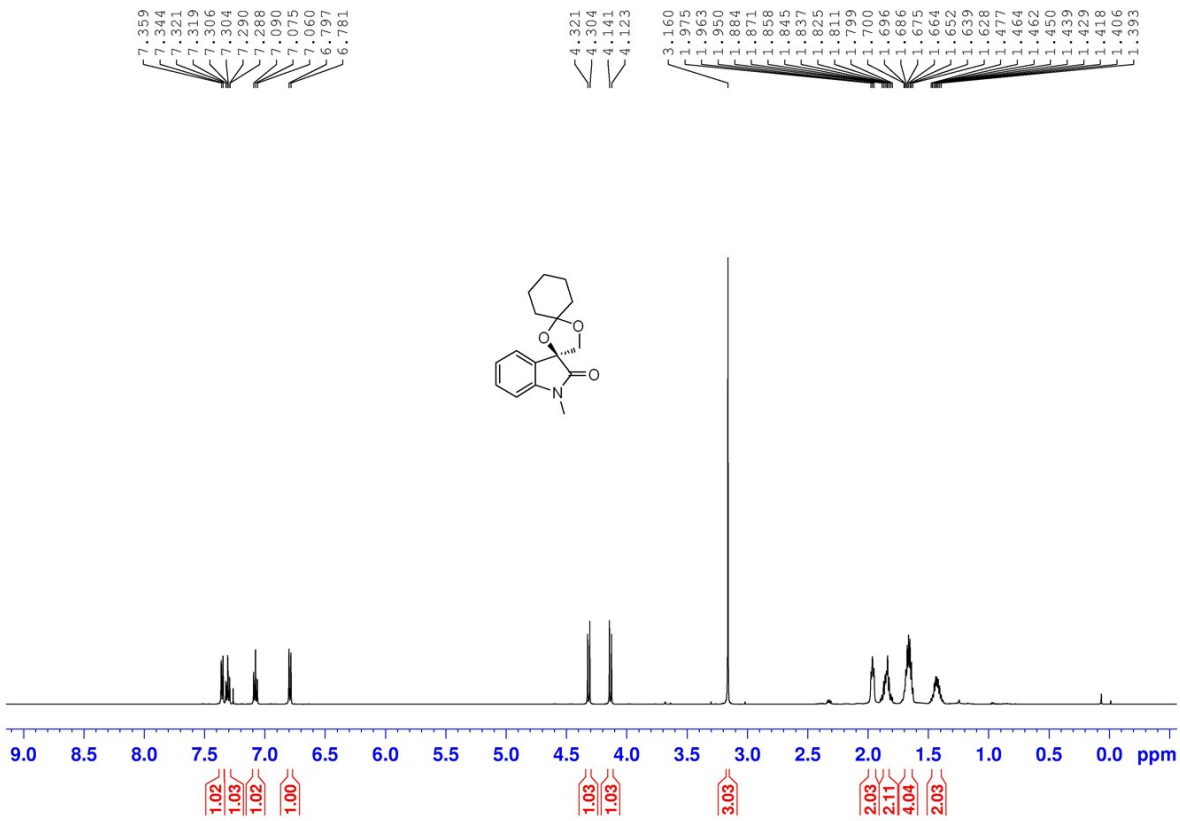

176.33
144.06
137.30
131.03
129.85
129.66
128.66
128.22
125.17
124.22
113.46
110.47
81.96
73.76
44.33
27.09
26.41
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

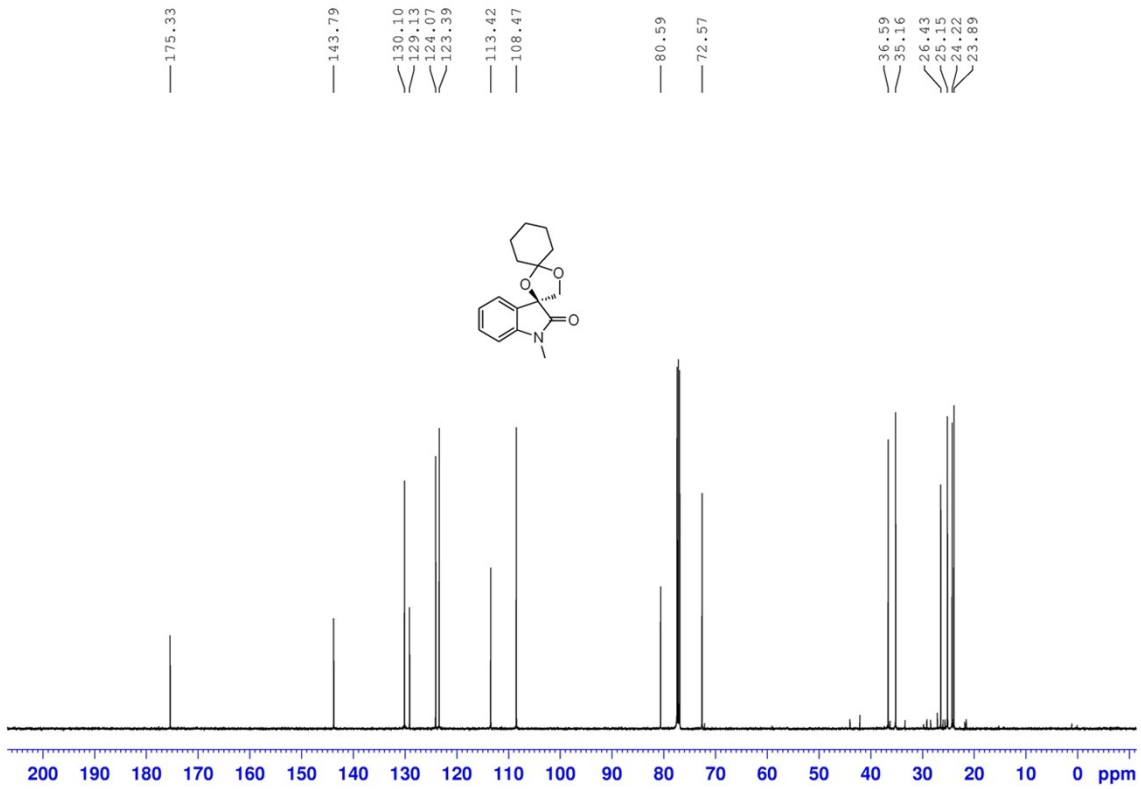
Compound **3i**


7.317
7.314
7.311
7.303
7.300
7.298
7.286
7.275
7.271
7.256
7.250
7.243
7.236
7.230
7.226
7.203
7.005
7.004
6.989
6.988
6.591
6.575
4.852
4.416
4.399
4.208
4.190
2.310
1.731
1.682
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
4.04
0.98
1.00
1.03
1.03
2.01
1.02
1.01
3.01
3.03
3.04

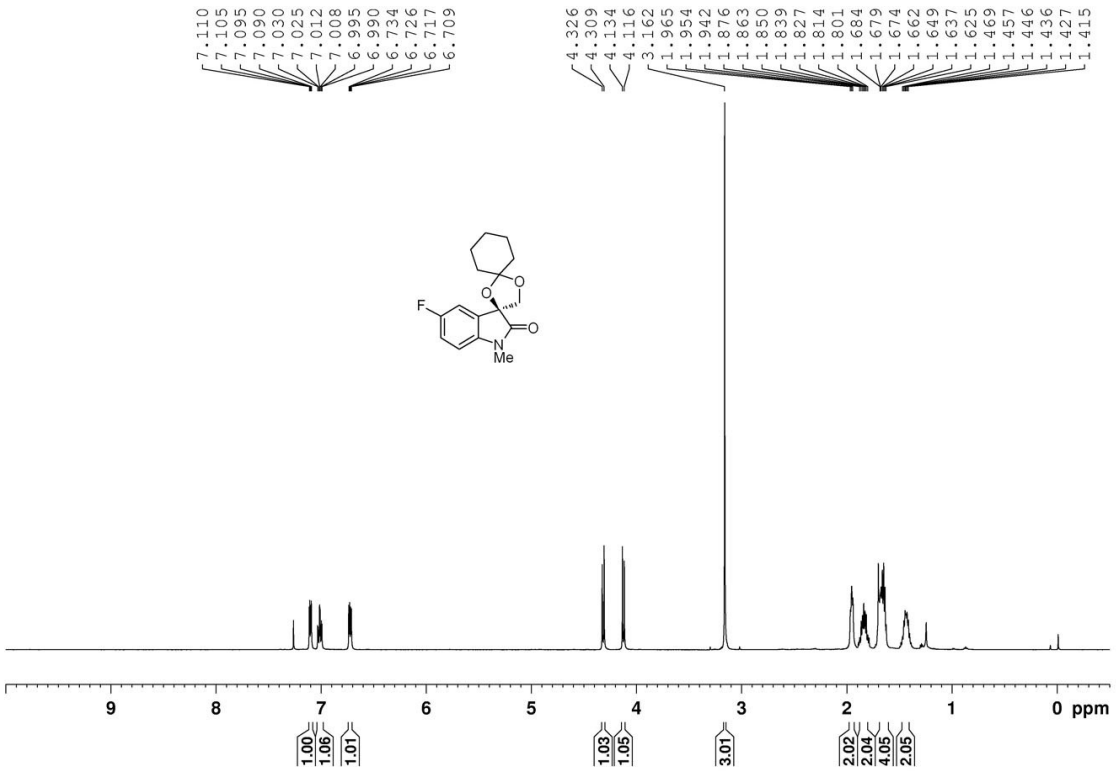

175.28
140.35
135.52
133.00
130.20
128.79
127.65
127.28
124.74
112.74
109.26
81.07
73.12
43.94
26.95
25.84
21.00
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

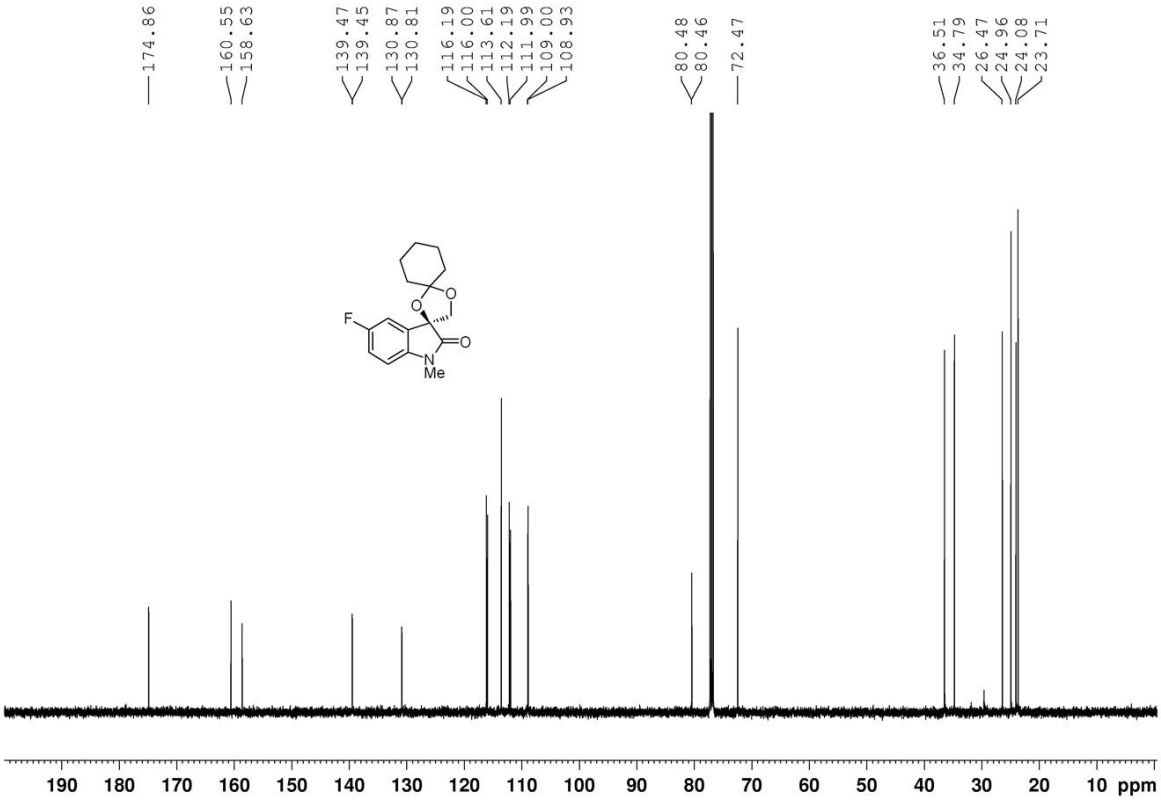
Compound **3j**


7.359
7.344
7.321
7.319
7.306
7.304
7.290
7.288
7.090
7.075
7.060
6.797
6.781
4.321
4.304
4.141
4.123
3.160
1.975
1.963
1.950
1.884
1.871
1.858
1.845
1.837
1.825
1.811
1.799
1.700
1.696
1.686
1.675
1.664
1.652
1.639
1.628
1.477
1.464
1.462
1.450
1.439
1.429
1.418
1.406
1.393
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.02
1.03
1.02
1.00
1.03
1.03
3.03
2.03
2.11
4.04
2.03

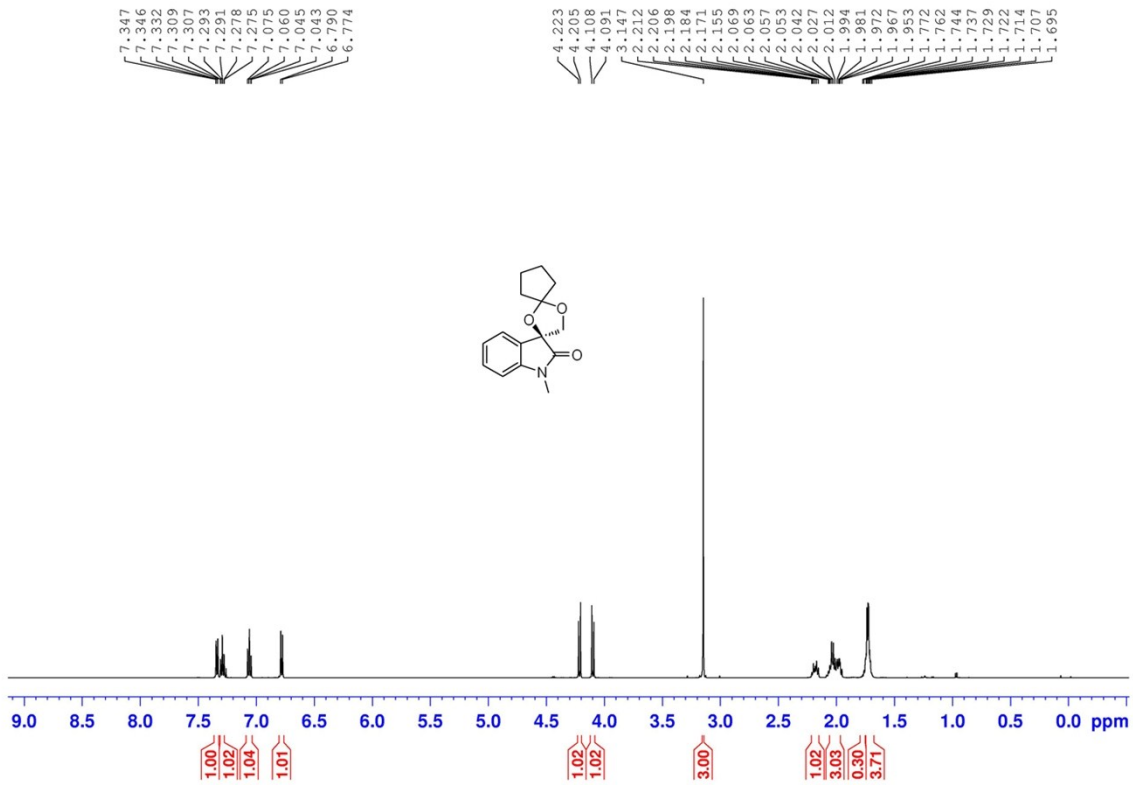

175.33
143.79
130.10
129.13
124.07
123.39
113.42
108.47
80.59
72.57
36.59
35.16
26.43
25.15
24.22
23.89
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

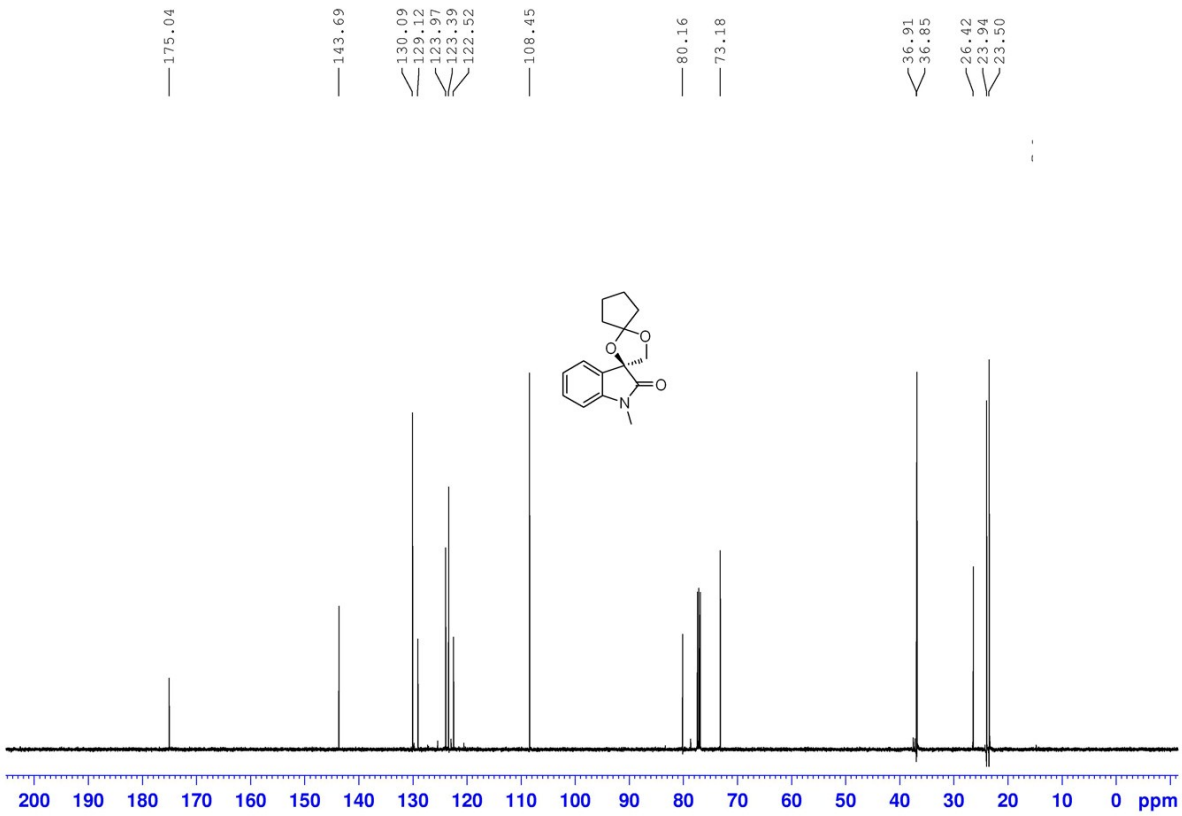
Compound **3k**


7.110
7.105
7.095
7.090
7.030
7.025
7.012
7.008
6.995
6.990
6.734
6.726
6.717
6.709
4.326
4.309
4.134
4.116
3.162
1.965
1.954
1.942
1.876
1.863
1.850
1.839
1.827
1.814
1.801
1.684
1.679
1.674
1.662
1.649
1.637
1.625
1.469
1.457
1.446
1.436
1.427
1.415
F
O
O
O
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.06
1.01
1.03
1.05
3.01
2.02
2.04
4.05
2.05

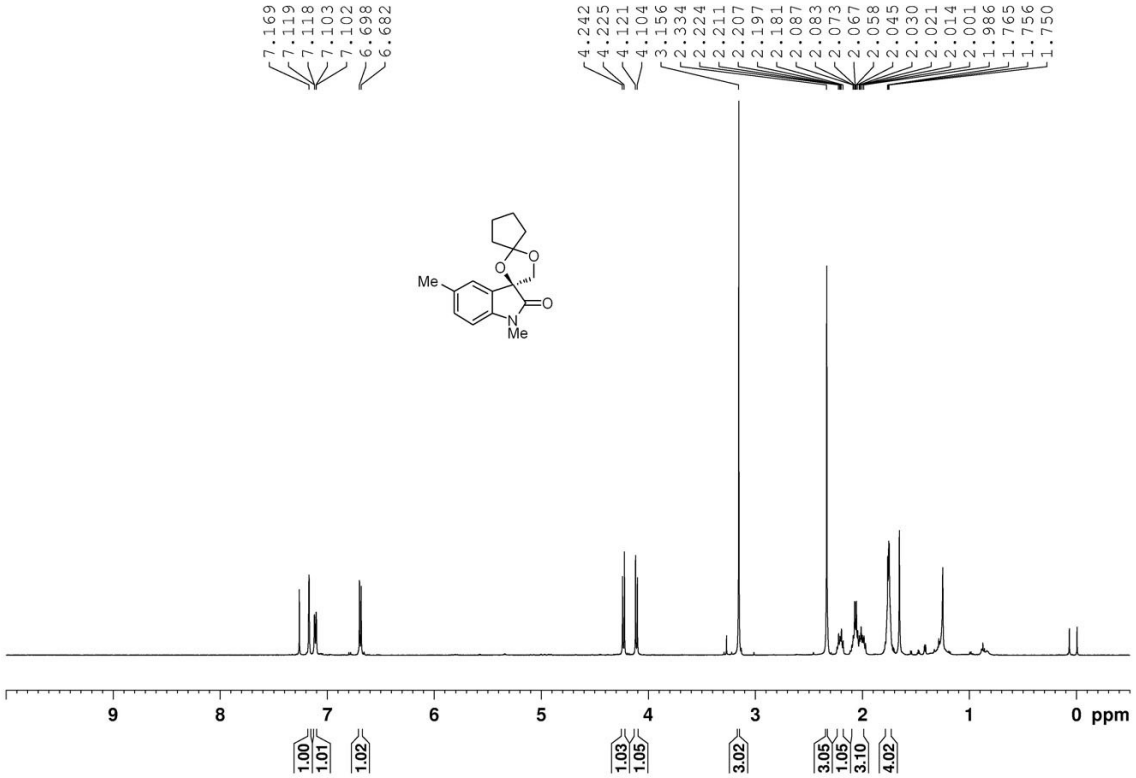

174.86
160.55
158.63
139.47
139.45
130.87
130.81
116.19
116.00
113.61
112.19
111.99
109.00
108.93
80.48
80.46
72.47
36.51
34.79
26.47
24.96
24.08
23.71
F
O
O
O
N
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm

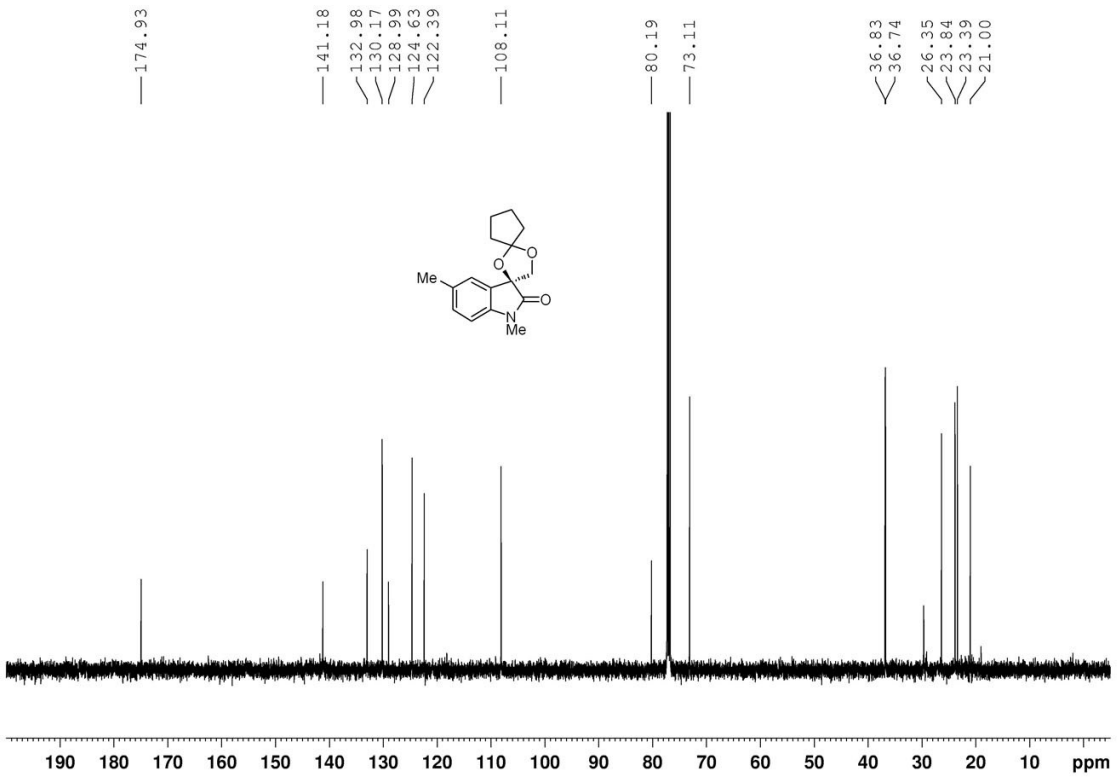
Compound **3l**


7.347
7.346
7.332
7.309
7.307
7.293
7.291
7.278
7.275
7.075
7.060
7.045
7.043
6.790
6.774
4.223
4.205
4.108
4.091
3.147
2.212
2.206
2.198
2.184
2.171
2.155
2.069
2.063
2.057
2.053
2.042
2.027
2.012
1.994
1.981
1.972
1.967
1.953
1.772
1.762
1.744
1.737
1.729
1.722
1.714
1.707
1.695
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.02
1.04
1.01
1.02
1.02
3.00
1.02
3.03
0.30
3.71

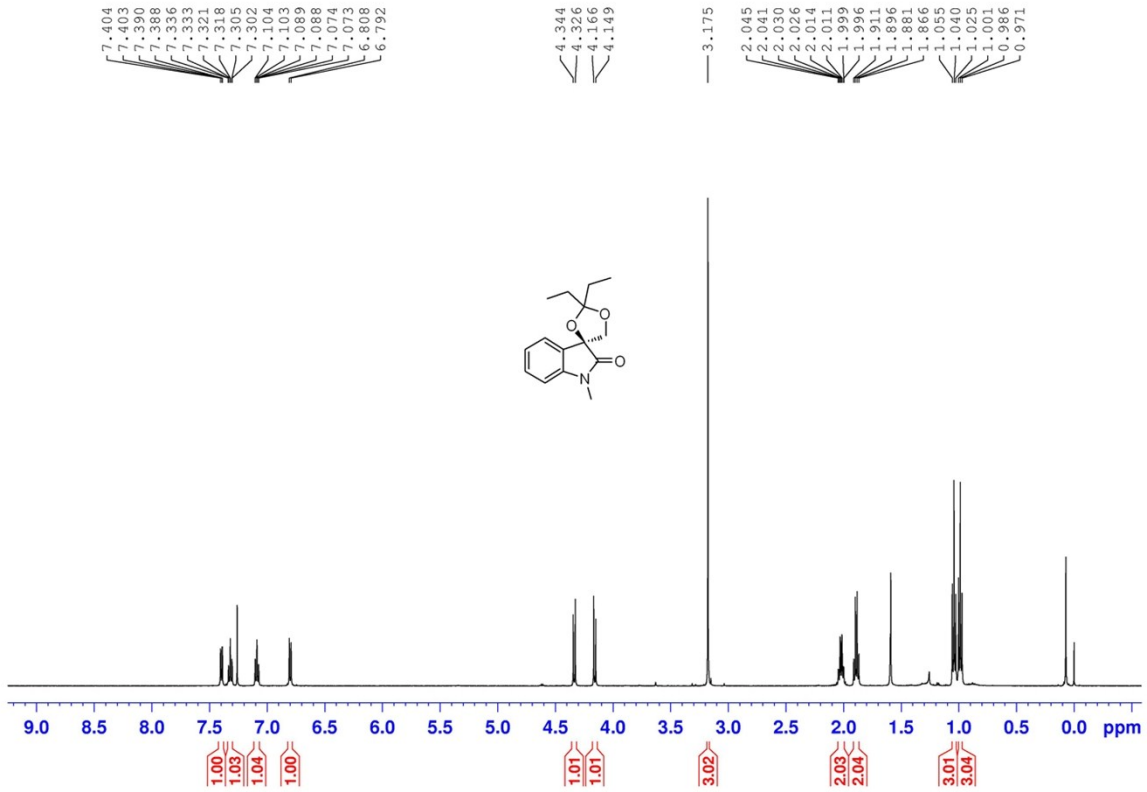

175.04
143.69
130.09
129.12
123.97
123.39
122.52
108.45
80.16
73.18
36.91
36.85
26.42
23.94
23.50
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

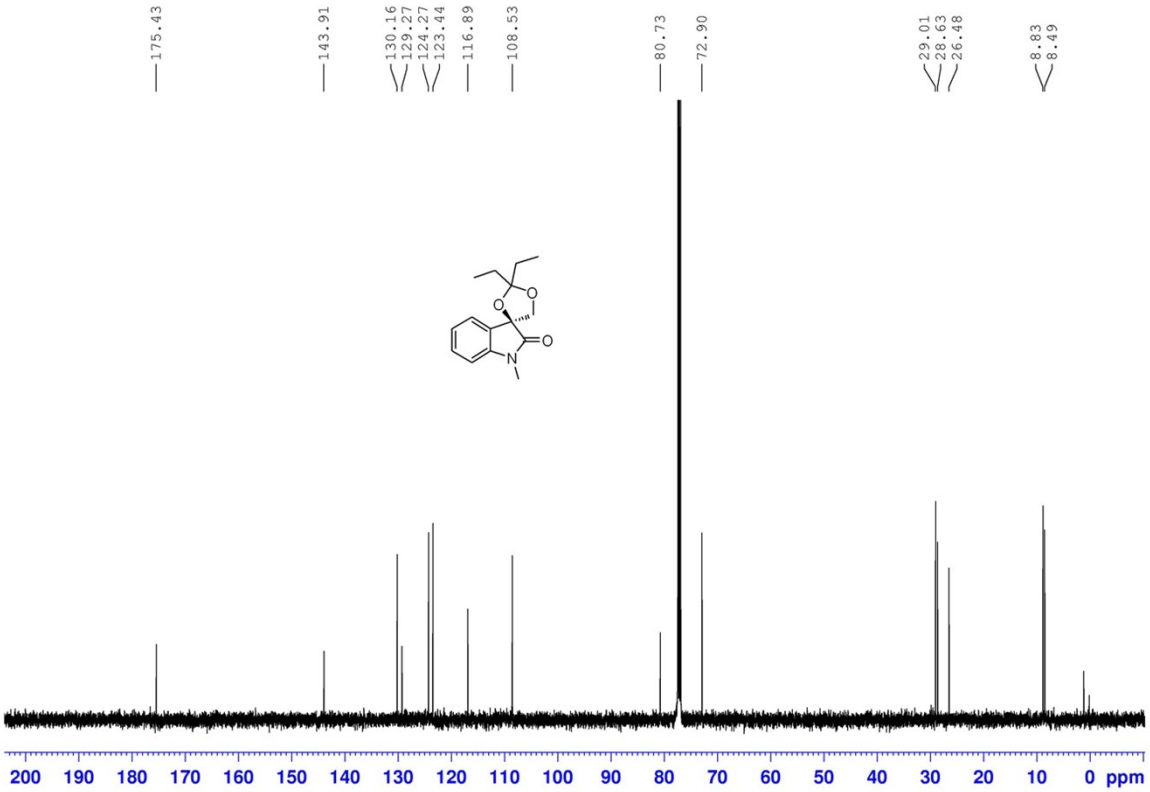
Compound **3m**


7.169
7.119
7.118
7.103
7.102
6.698
6.682
4.242
4.225
4.121
4.104
3.156
2.334
2.224
2.211
2.207
2.197
2.181
2.087
2.083
2.073
2.067
2.058
2.045
2.030
2.021
2.014
2.001
1.986
1.765
1.756
1.750
Me
O
O
O
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.01
1.02
1.03
1.05
3.02
3.05
1.05
3.10
4.02

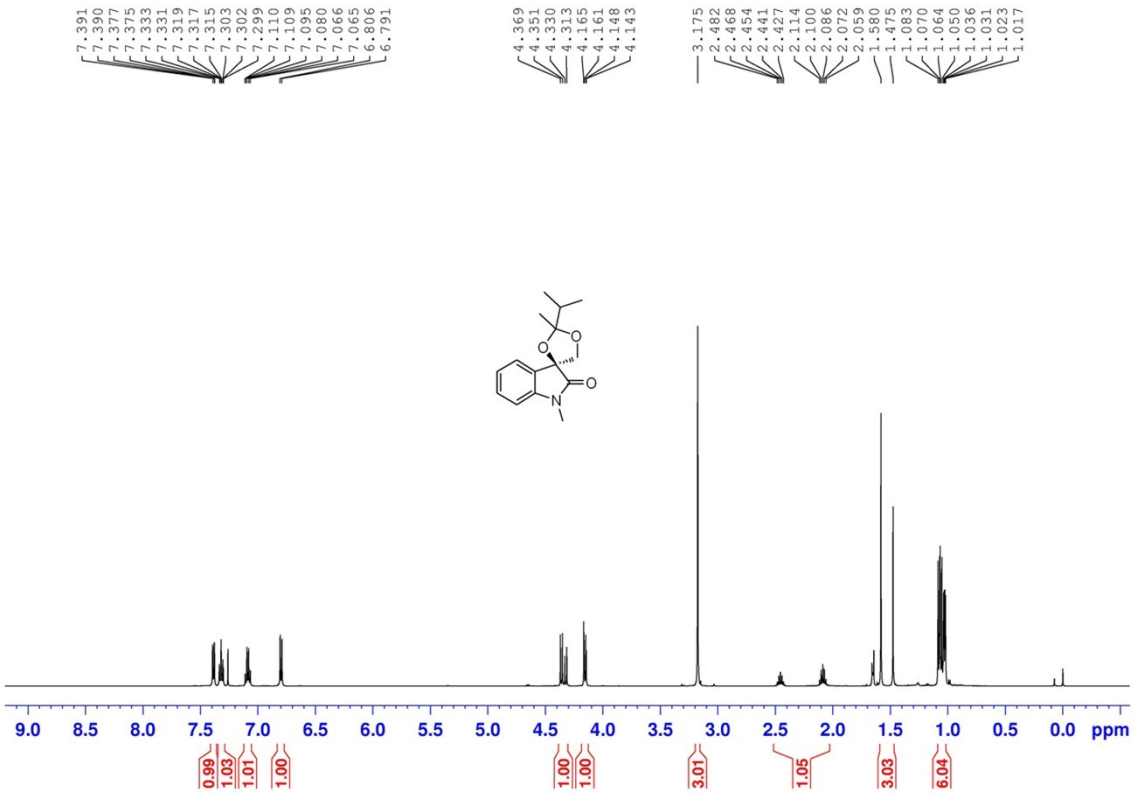

174.93
141.18
132.98
130.17
128.99
124.63
122.39
108.11
80.19
73.11
36.83
36.74
26.35
23.84
23.39
21.00
Me
O
O
O
N
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

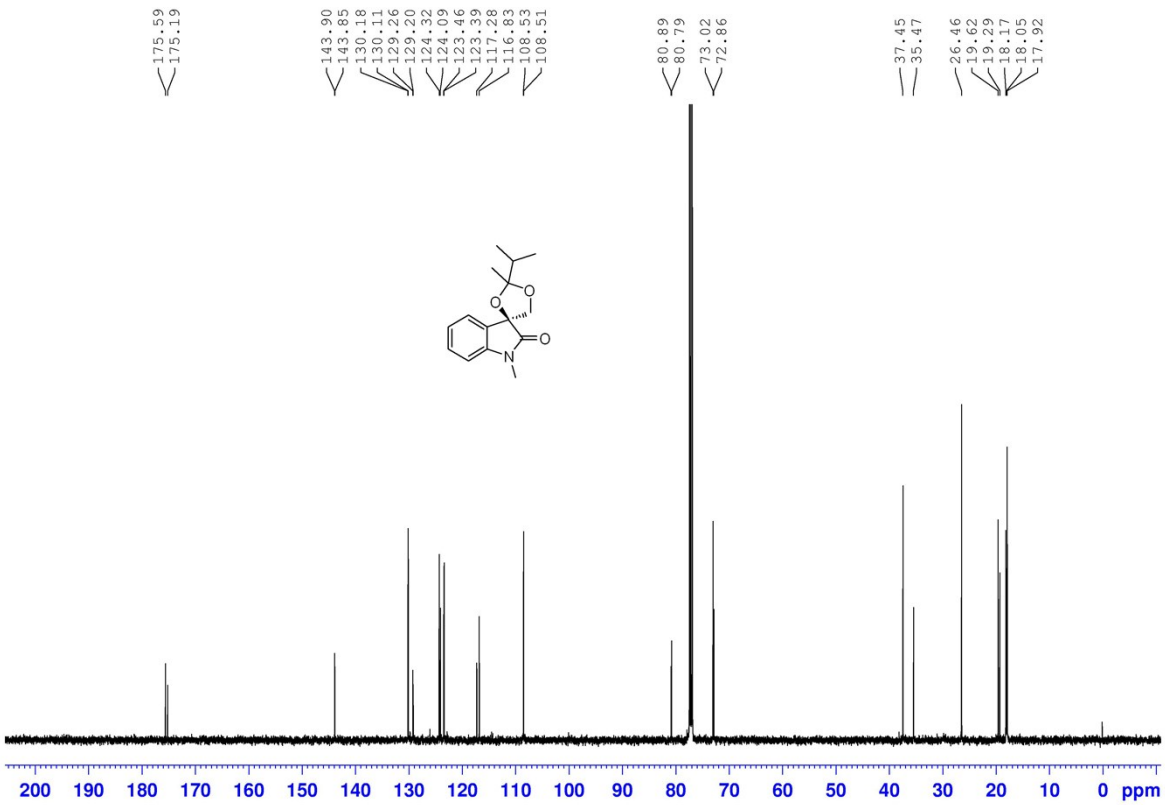
Compound **3n**


7.404
7.403
7.390
7.388
7.336
7.333
7.321
7.318
7.305
7.302
7.104
7.103
7.089
7.088
7.074
7.073
6.808
6.792
4.344
4.326
4.166
4.149
3.175
2.045
2.041
2.030
2.026
2.014
2.011
1.999
1.996
1.911
1.896
1.881
1.866
1.055
1.040
1.025
1.001
0.986
0.971
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.03
1.04
1.00
1.01
1.01
3.02
2.03
2.04
3.01
3.04

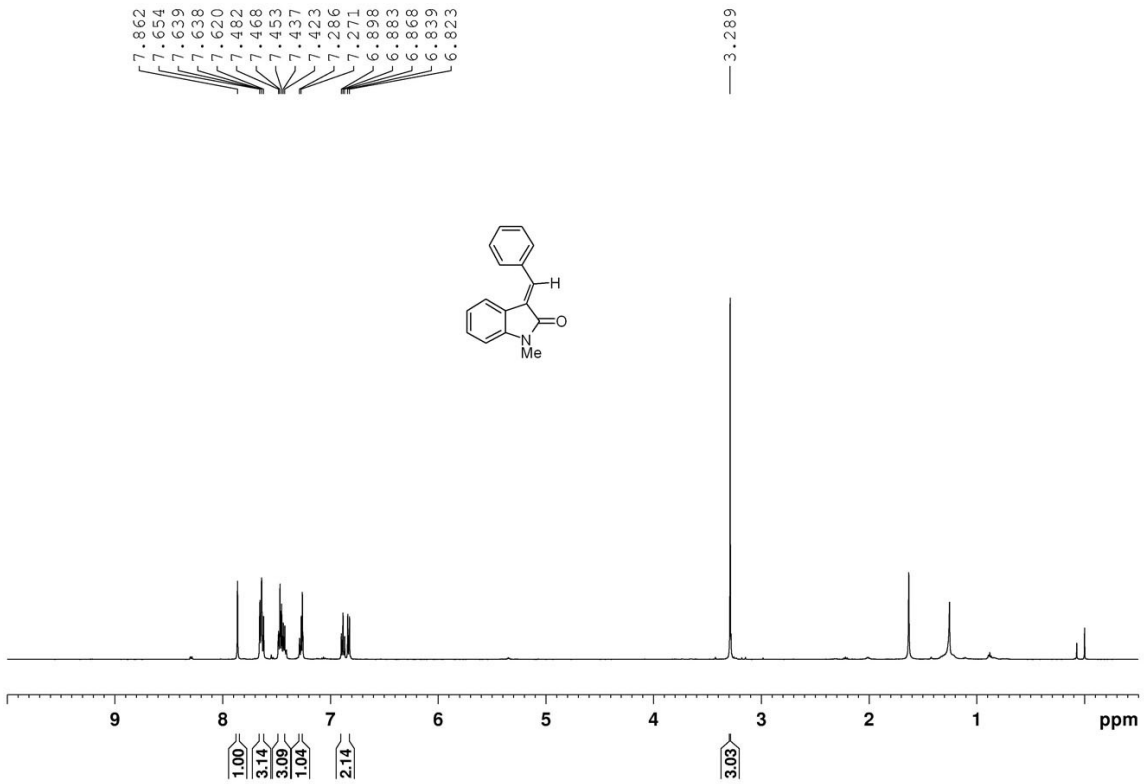

175.43
143.91
130.16
129.27
124.27
123.44
116.89
108.53
80.73
72.90
29.01
28.63
26.48
8.83
8.49
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

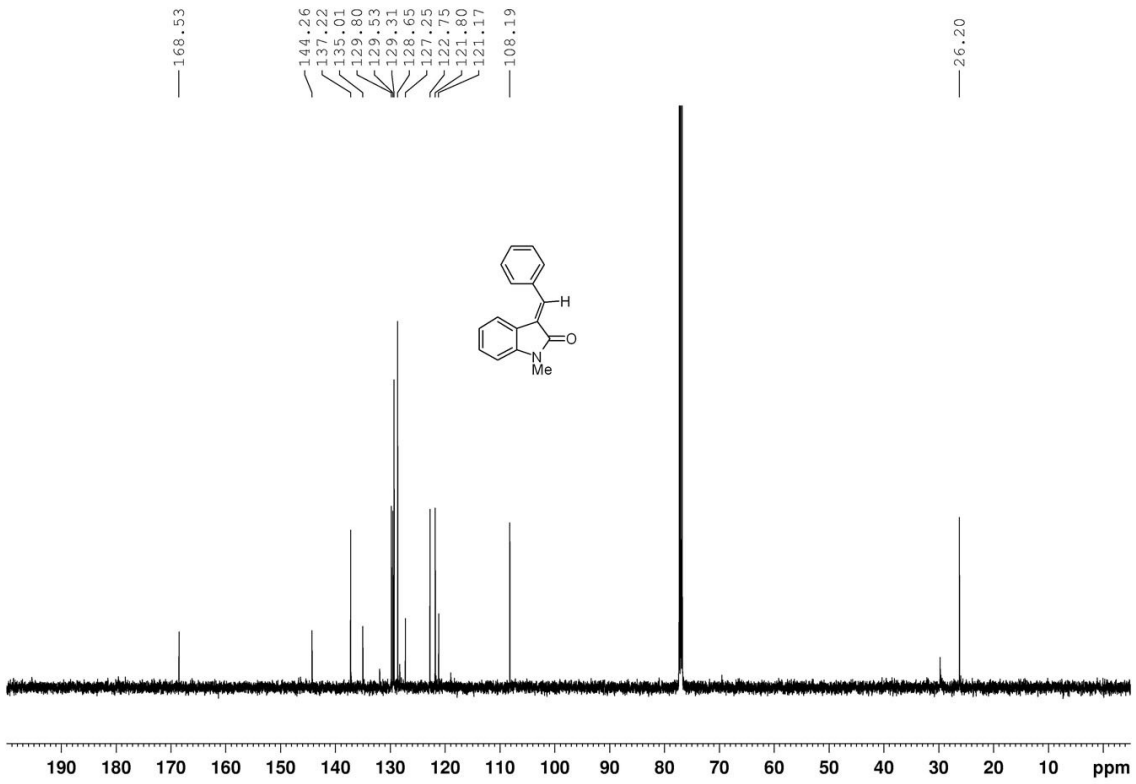
Compound **3o**


7.391 7.390 7.377 7.375 7.333 7.331 7.319 7.317 7.315 7.303 7.302 7.299 7.110 7.109 7.095 7.080 7.066 7.065 6.806 6.791
4.369 4.351 4.330 4.313 4.165 4.161 4.148 4.143
3.175
2.482 2.468 2.454 2.441 2.427 2.114 2.100 2.086 2.072 2.059
1.580 1.475
1.083 1.070 1.064 1.050 1.036 1.031 1.023 1.017
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.99 1.03 1.01 1.00 1.00 1.00 3.01 1.05 3.03 6.04

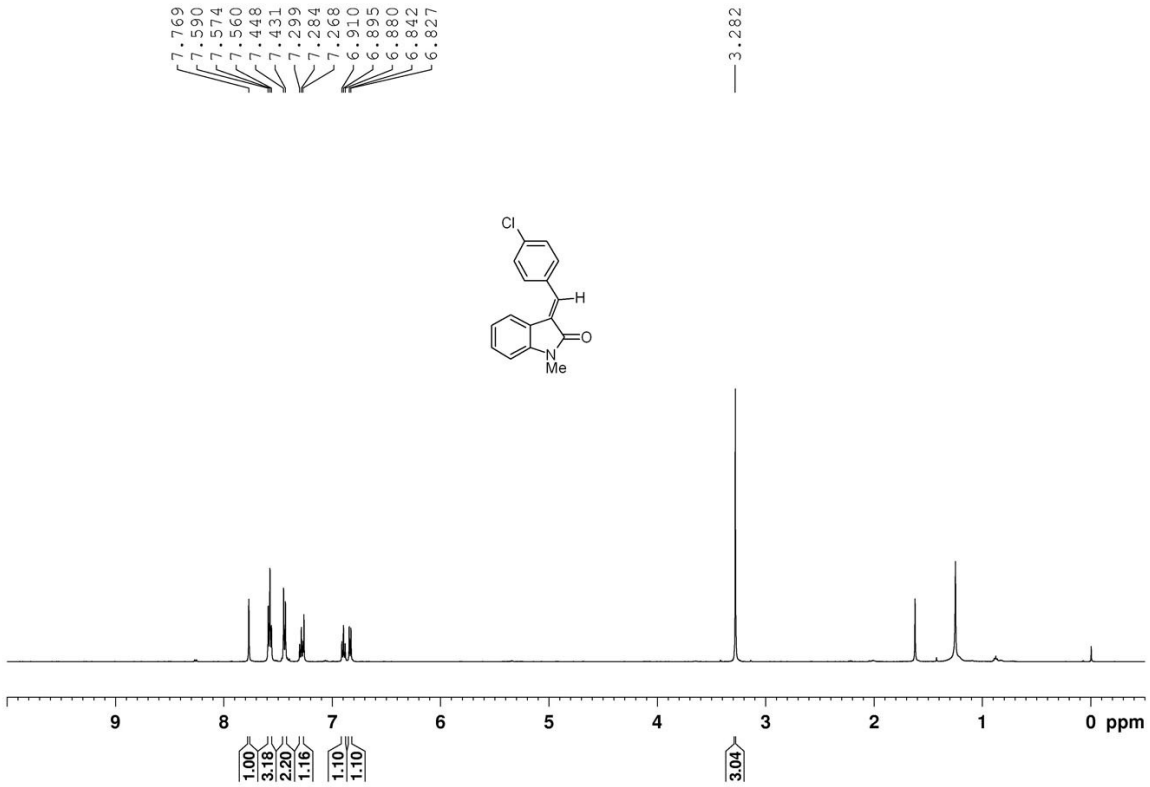

175.59 175.19
143.90 143.85 130.18 130.11 129.26 129.20 124.32 124.09 123.46 123.39 117.28 116.83 108.53 108.51
80.89 80.79 73.02 72.86
37.45 35.47
26.46 19.62 19.29 18.17 18.05 17.92
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

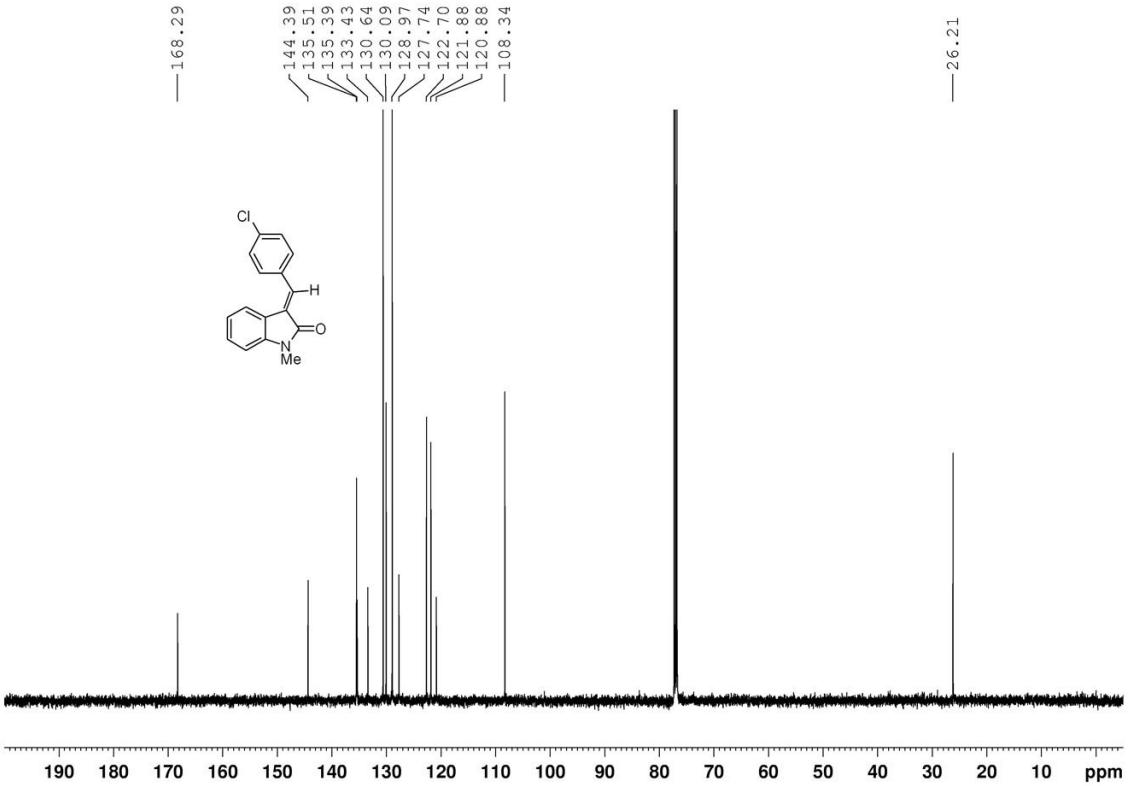
Compound **5a**


7.862
7.654
7.639
7.638
7.620
7.482
7.468
7.453
7.437
7.423
7.286
7.271
6.898
6.883
6.868
6.839
6.823
3.289
H
O
N
Me
9
8
7
6
5
4
3
2
1
ppm
1.00
3.14
3.09
1.04
2.14
3.03

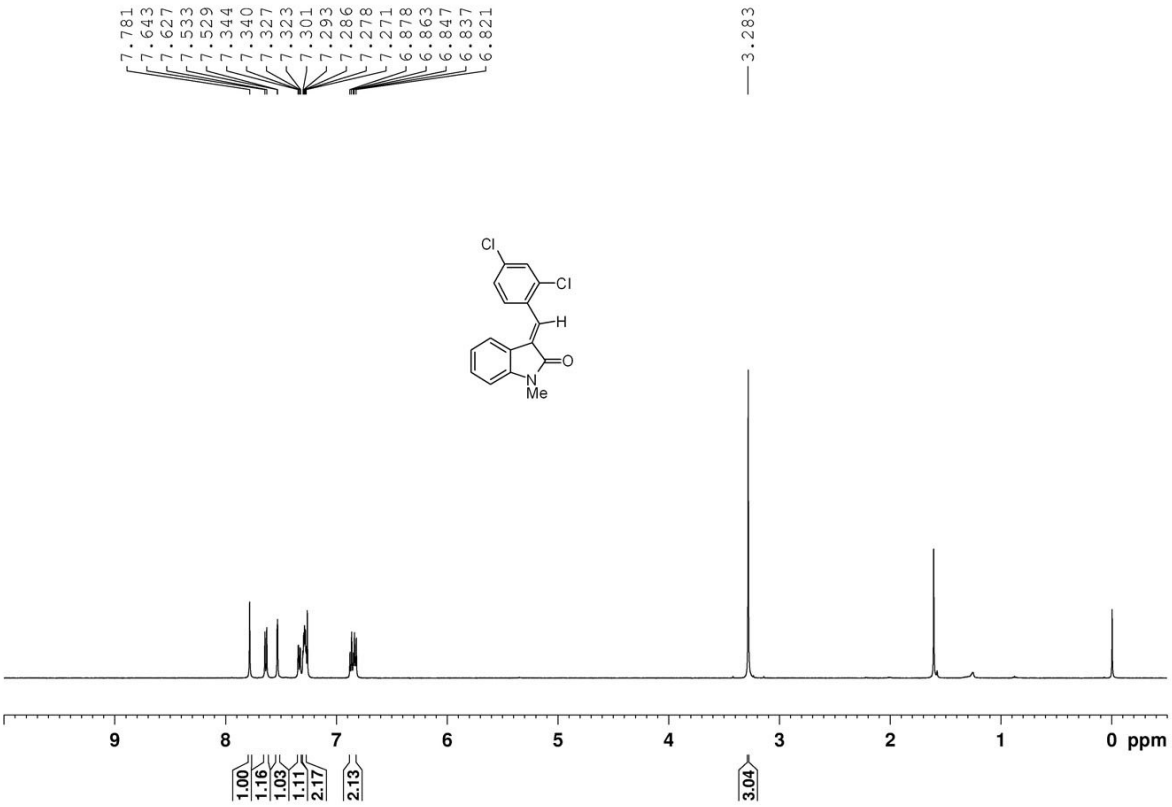

168.53
144.26
137.22
135.01
129.80
129.53
129.31
128.65
127.25
122.75
121.80
121.17
108.19
26.20
H
O
N
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

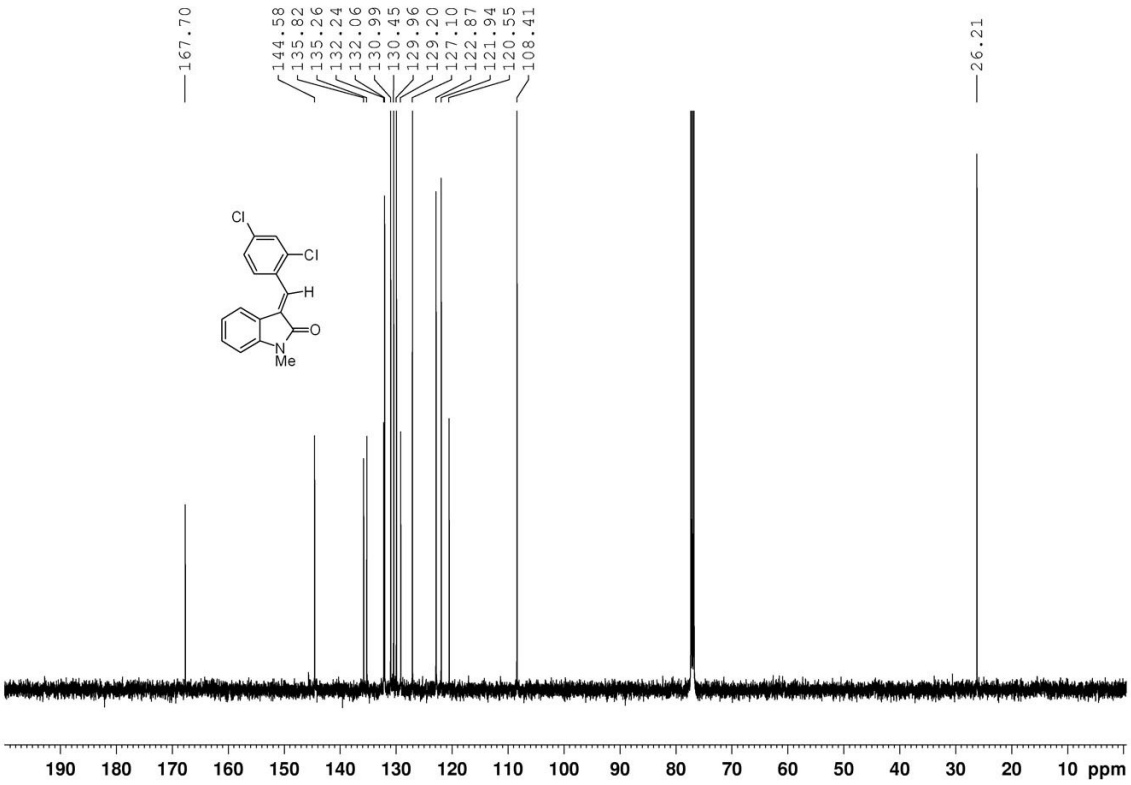
Compound **5b**


7.769
7.590
7.574
7.560
7.448
7.431
7.299
7.284
7.268
6.910
6.895
6.880
6.842
6.827
3.282
Cl
H
O
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
1.00
3.18
2.20
1.16
1.10
1.10
3.04

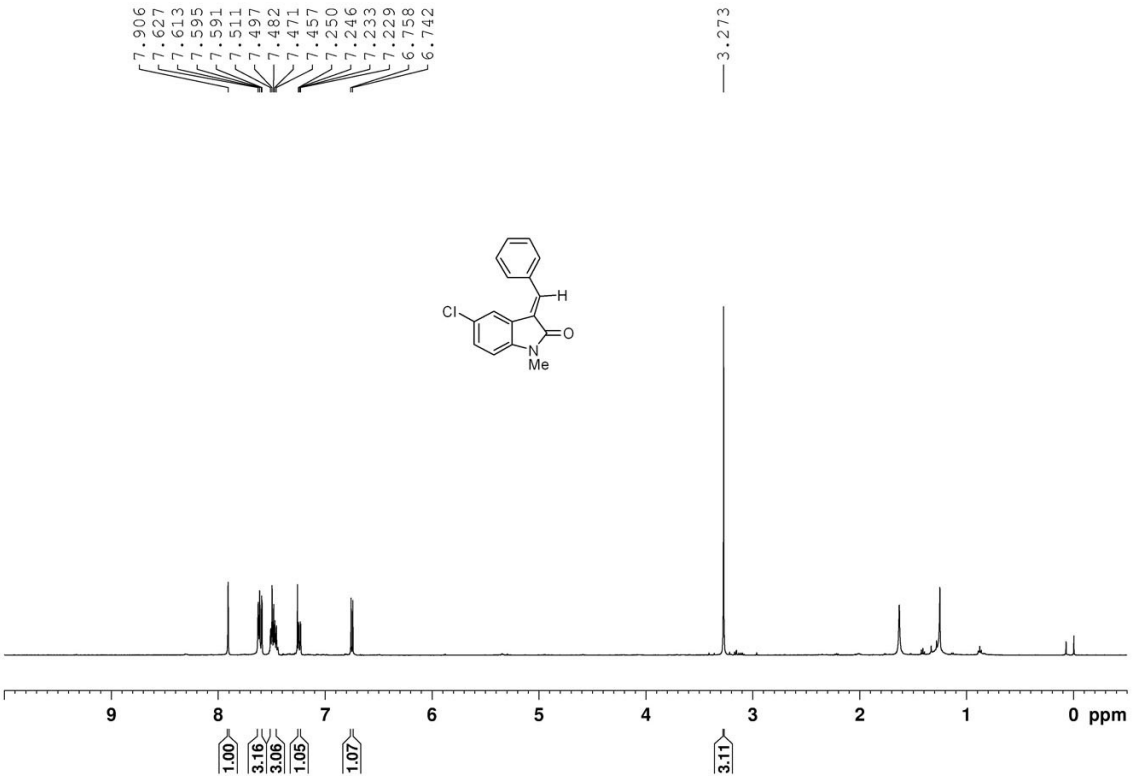

168.29
144.39
135.51
135.39
133.43
130.64
130.09
128.97
127.74
122.70
121.88
120.88
108.34
26.21
Cl
H
O
N
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

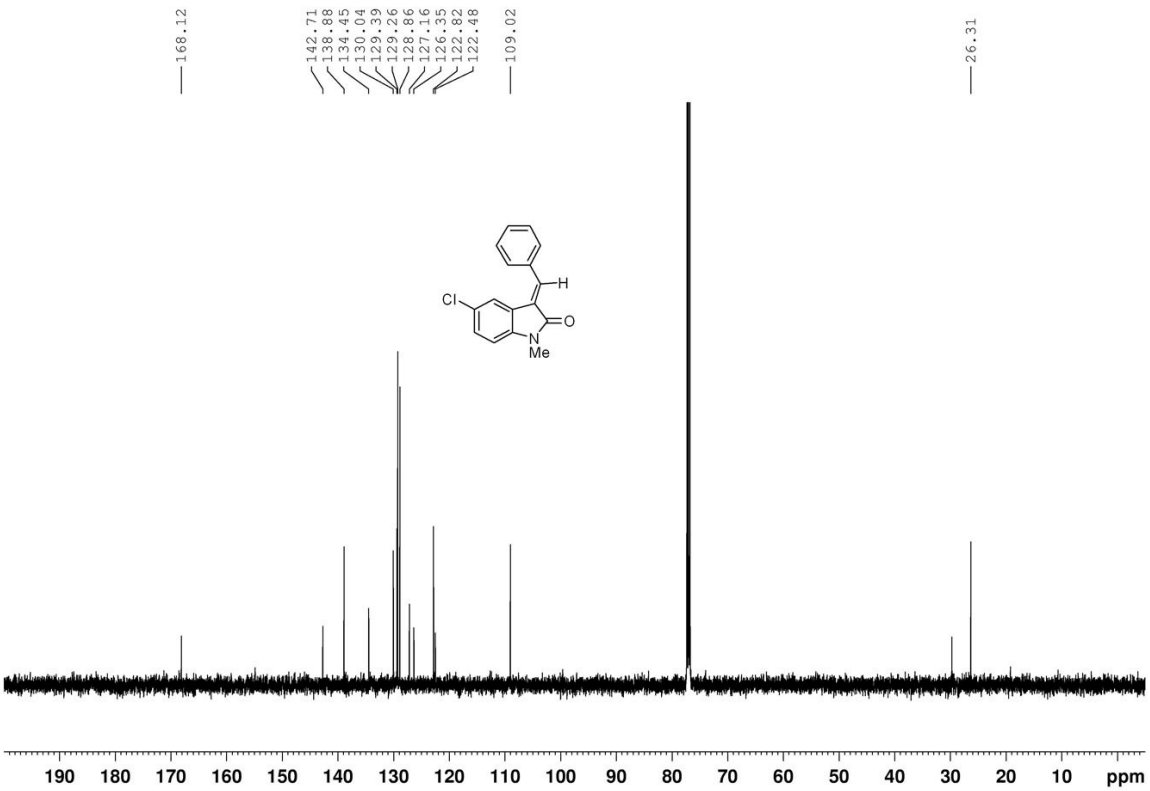
Compound **5c**


7.781
7.643
7.627
7.533
7.529
7.344
7.340
7.327
7.323
7.301
7.293
7.286
7.278
7.271
6.878
6.863
6.847
6.837
6.821
3.283
Cl
Cl
H
O
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
1.00
1.16
1.03
1.11
2.17
2.13
3.04

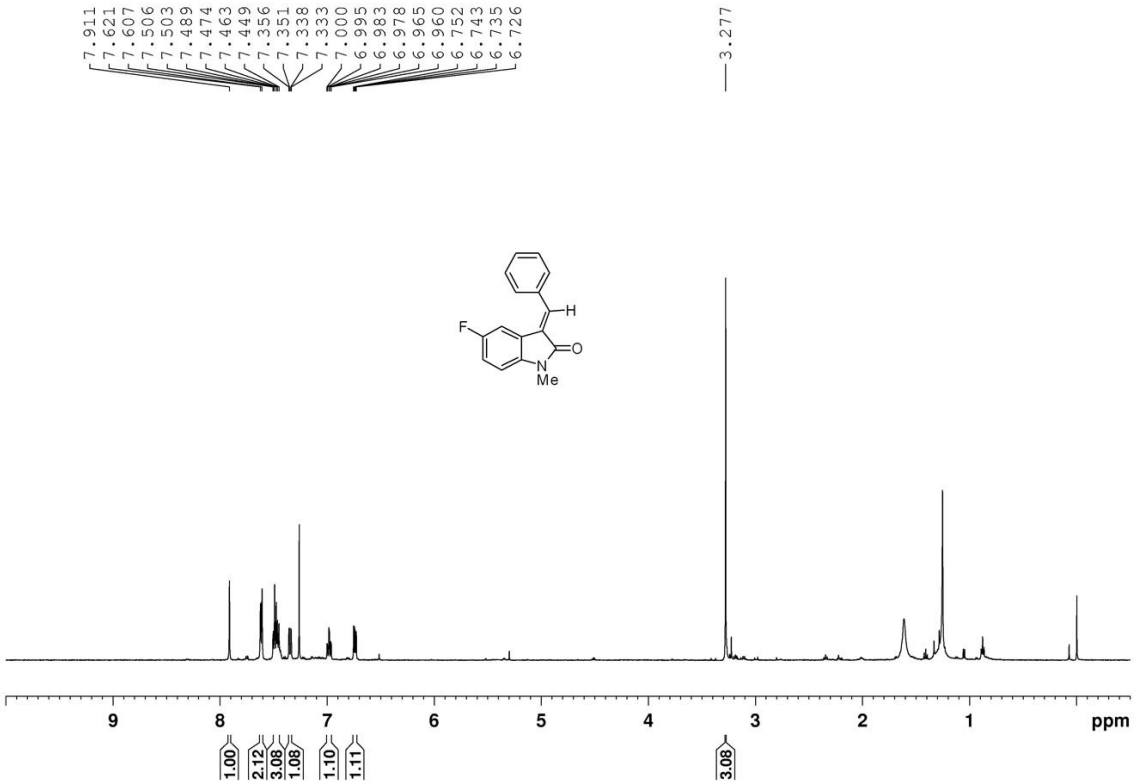

167.70
144.58
135.82
135.26
132.24
132.06
130.99
130.45
129.96
129.20
127.10
122.87
121.94
120.55
108.41
26.21
Cl
Cl
H
O
N
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10 ppm

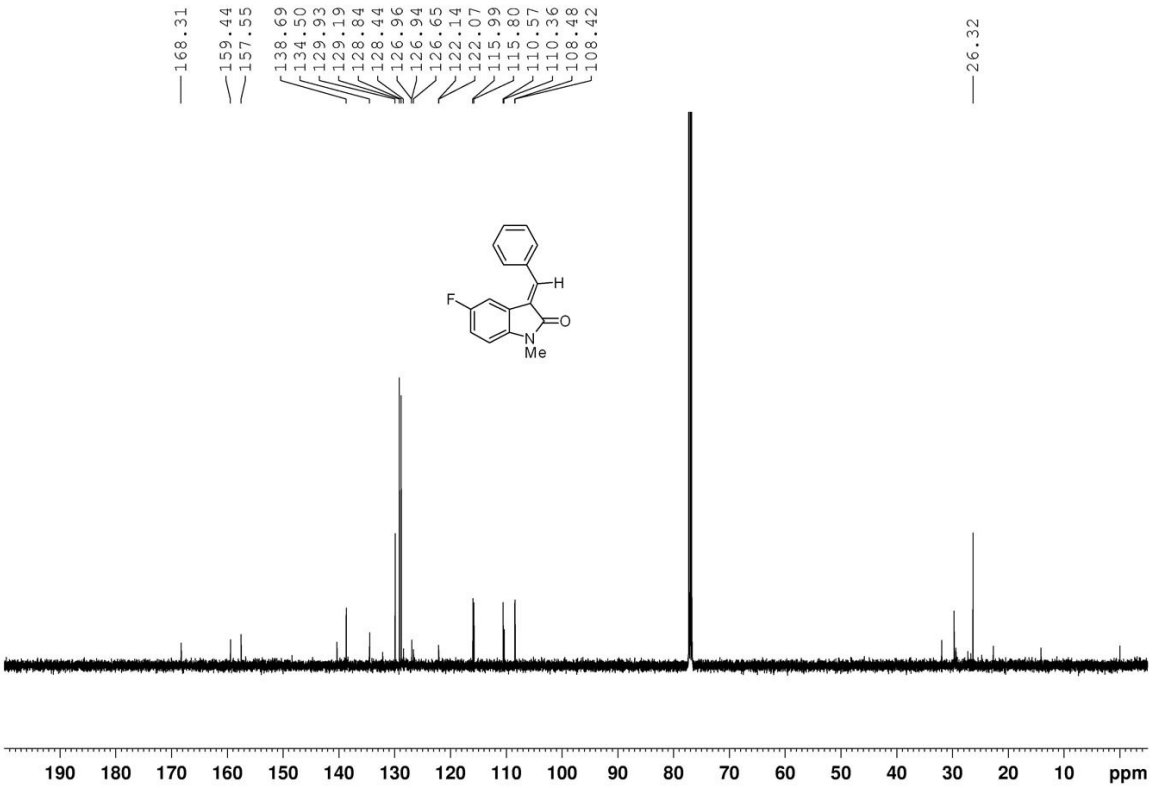
Compound **5d**


7.906
7.627
7.613
7.595
7.591
7.511
7.497
7.482
7.471
7.457
7.250
7.246
7.233
7.229
6.758
6.742
3.273
Cl
H
O
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
1.00
3.16
3.06
1.05
1.07
3.11

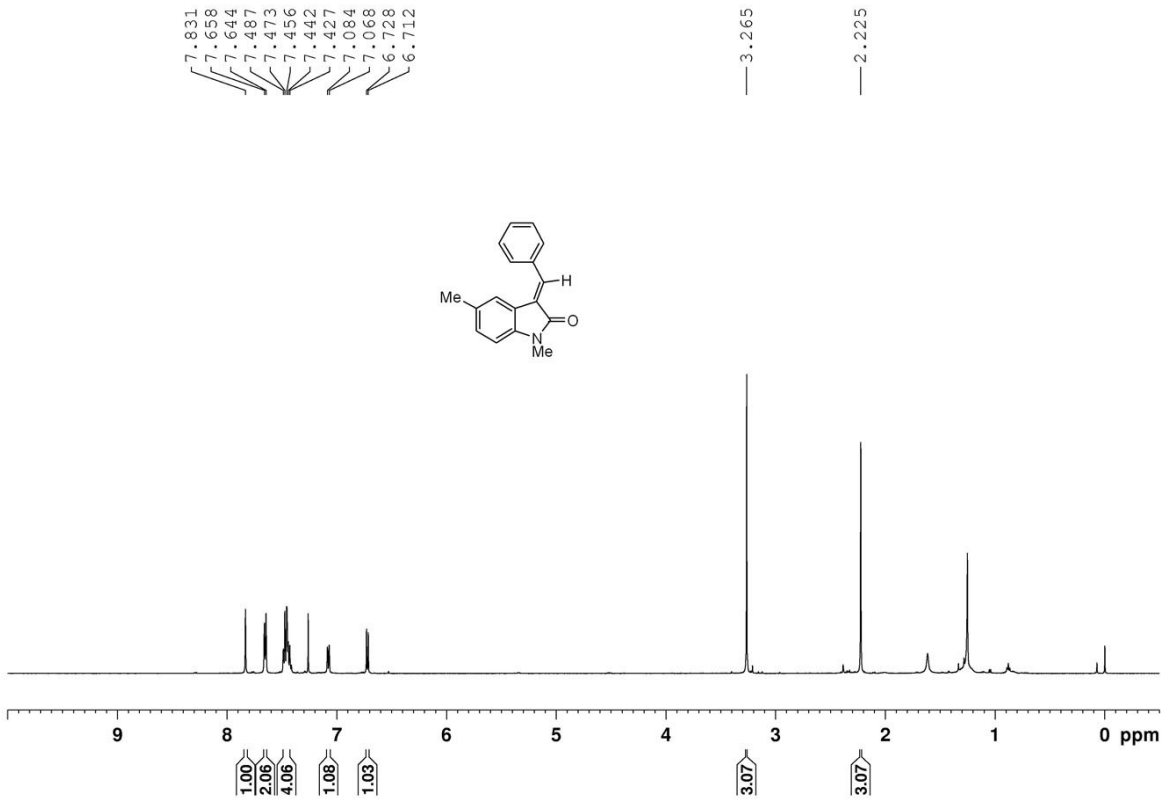

168.12
142.71
138.88
134.45
130.04
129.39
129.26
128.86
127.16
126.35
122.82
122.48
109.02
26.31
Cl
H
O
N
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

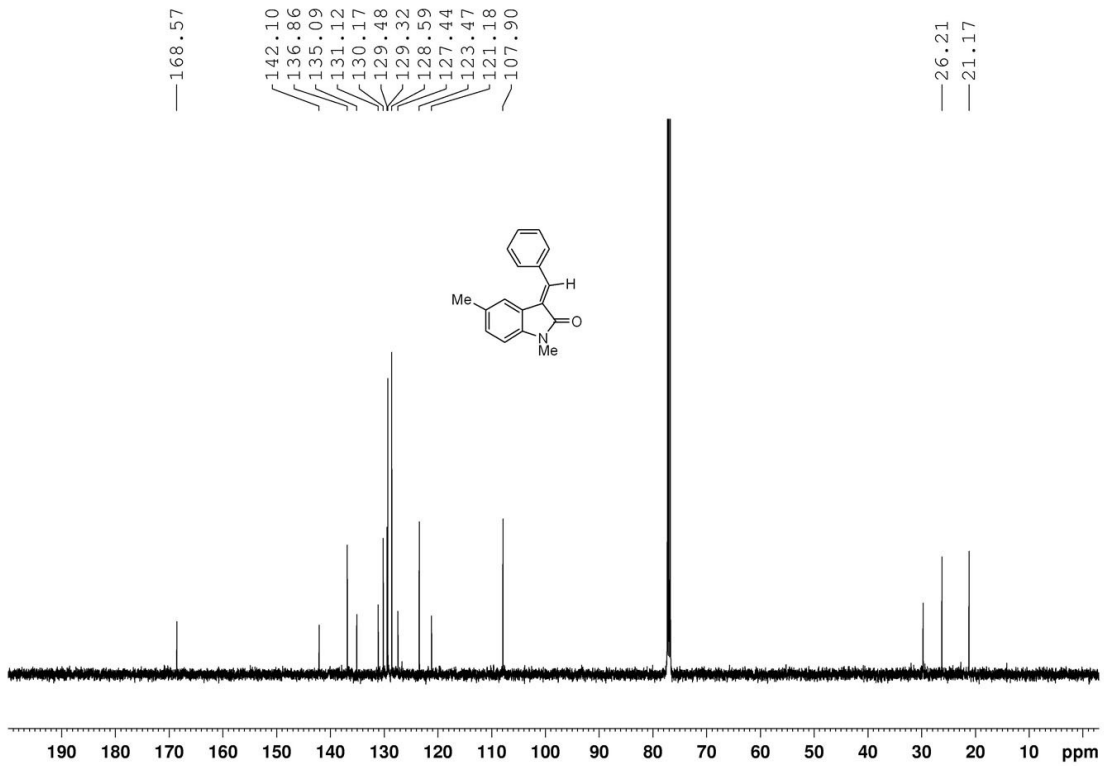
Compound **5e**


7.911
7.621
7.607
7.506
7.503
7.489
7.474
7.463
7.449
7.356
7.351
7.338
7.333
7.000
6.995
6.983
6.978
6.965
6.960
6.752
6.743
6.735
6.726
3.277
F
H
O
N
Me
9
8
7
6
5
4
3
2
1
ppm
1.00
2.12
3.08
1.08
1.10
1.11
3.08

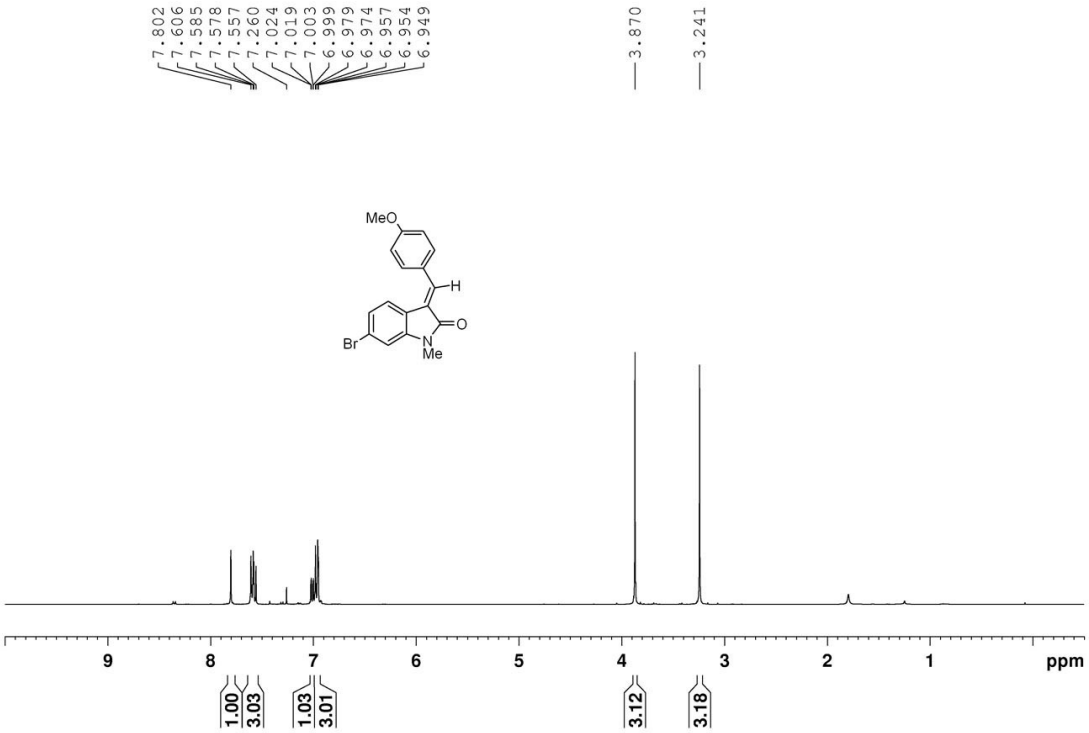

168.31
159.44
157.55
138.69
134.50
129.93
129.19
128.84
128.44
126.96
126.94
126.65
122.14
122.07
115.99
115.80
110.57
110.36
108.48
108.42
26.32
F
H
O
N
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

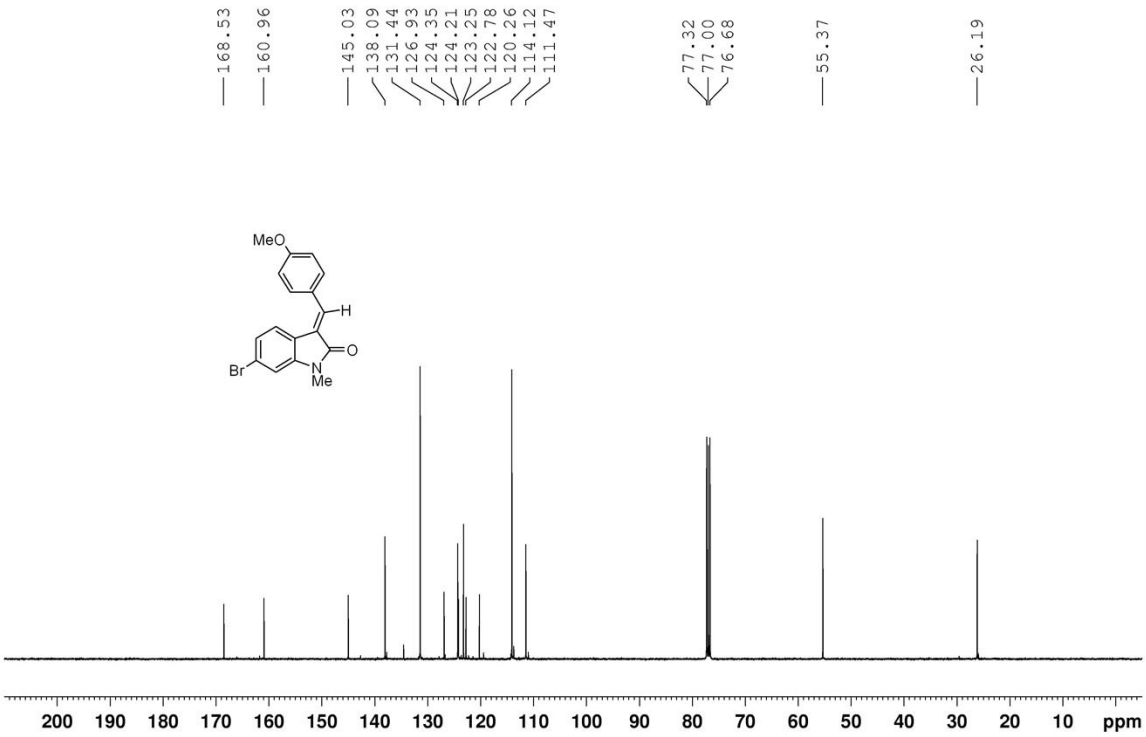
Compound **5f**


7.831
7.658
7.644
7.487
7.473
7.456
7.442
7.427
7.084
7.068
6.728
6.712
3.265
2.225
Me
H
O
N
Me
9
8
7
6
5
4
3
2
1
0 ppm
1.00
2.06
4.06
1.08
1.03
3.07
3.07

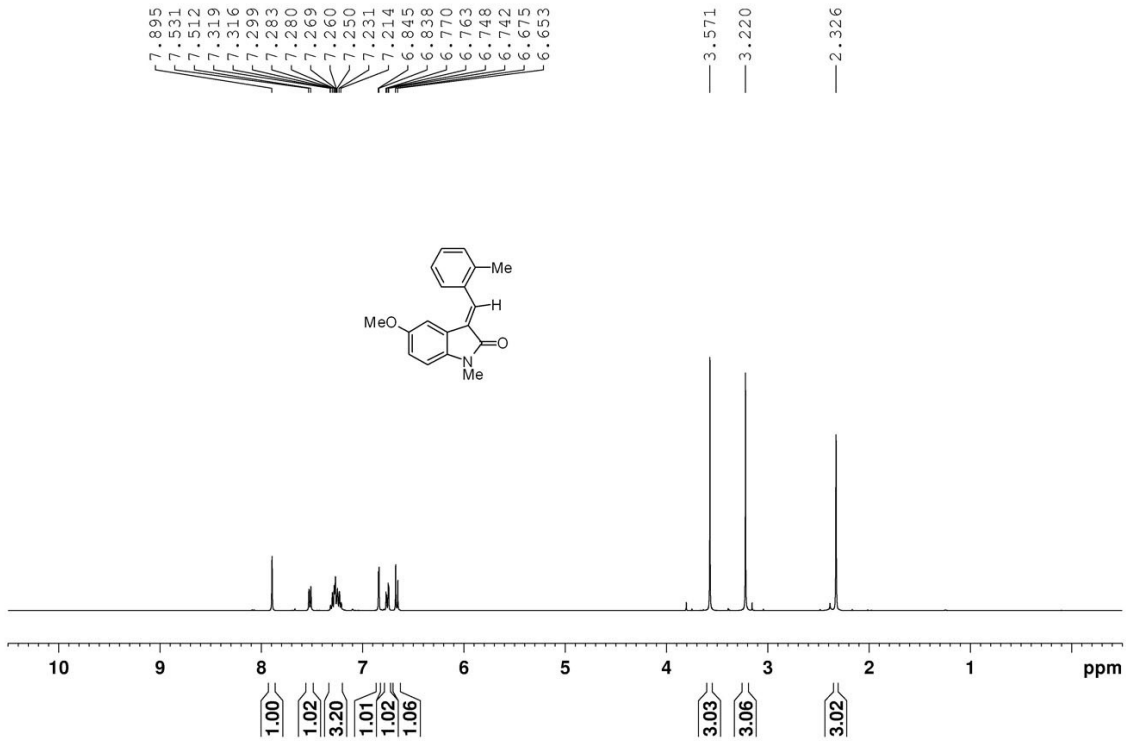

168.57
142.10
136.86
135.09
131.12
130.17
129.48
129.32
128.59
127.44
123.47
121.18
107.90
26.21
21.17
Me
H
O
N
Me
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

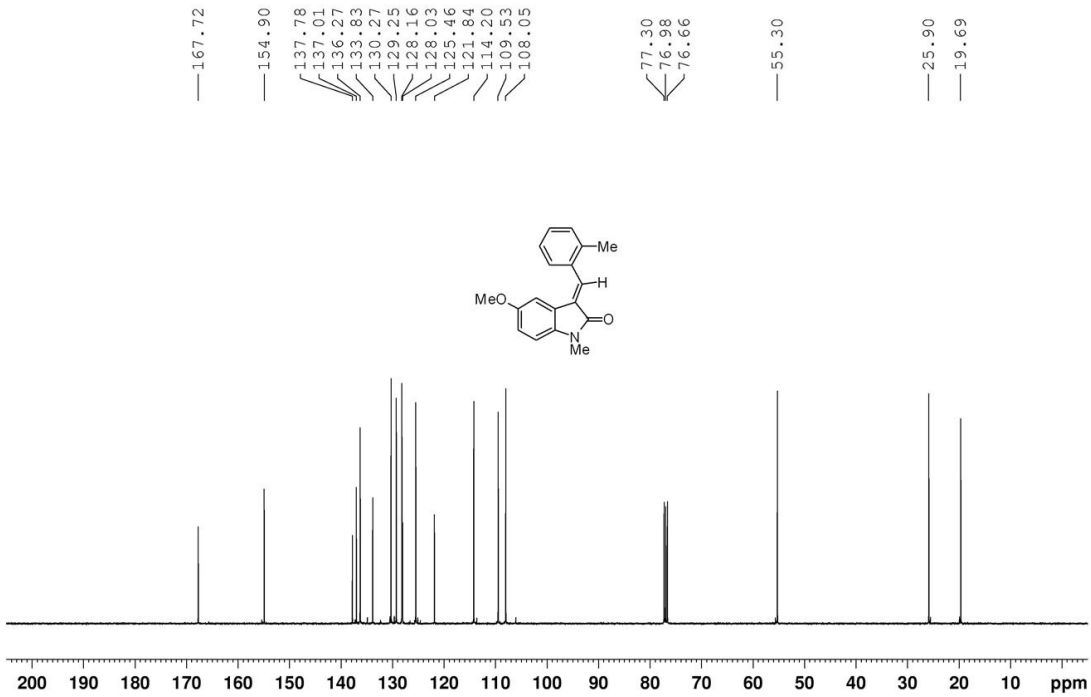
Compound **5g**


7.802
7.606
7.585
7.578
7.557
7.260
7.024
7.019
7.003
6.999
6.979
6.974
6.957
6.954
6.949
3.870
3.241
MeO
H
O
Br
N
Me
9
8
7
6
5
4
3
2
1
ppm
1.00
3.03
1.03
3.01
3.12
3.18

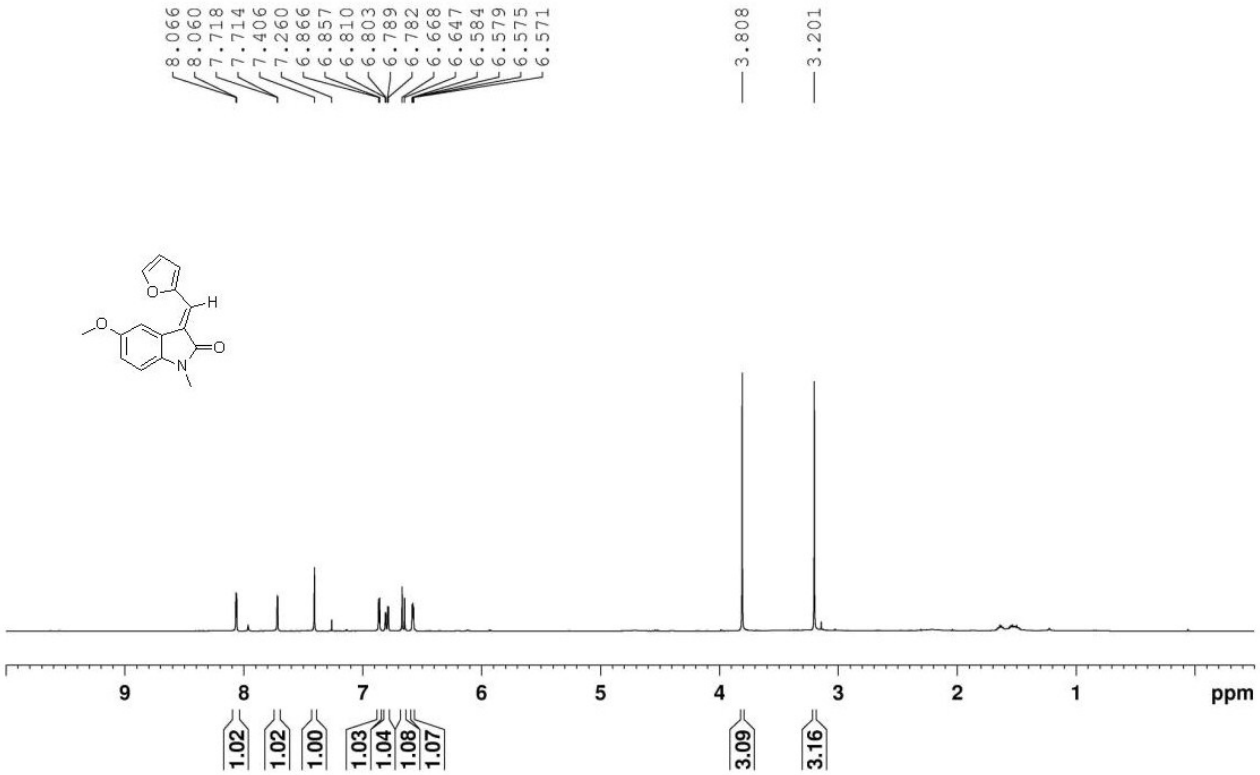

168.53
160.96
145.03
138.09
131.44
126.93
124.35
124.21
123.25
122.78
120.26
114.12
111.47
77.32
77.00
76.68
55.37
26.19
MeO
H
O
Br
N
Me
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

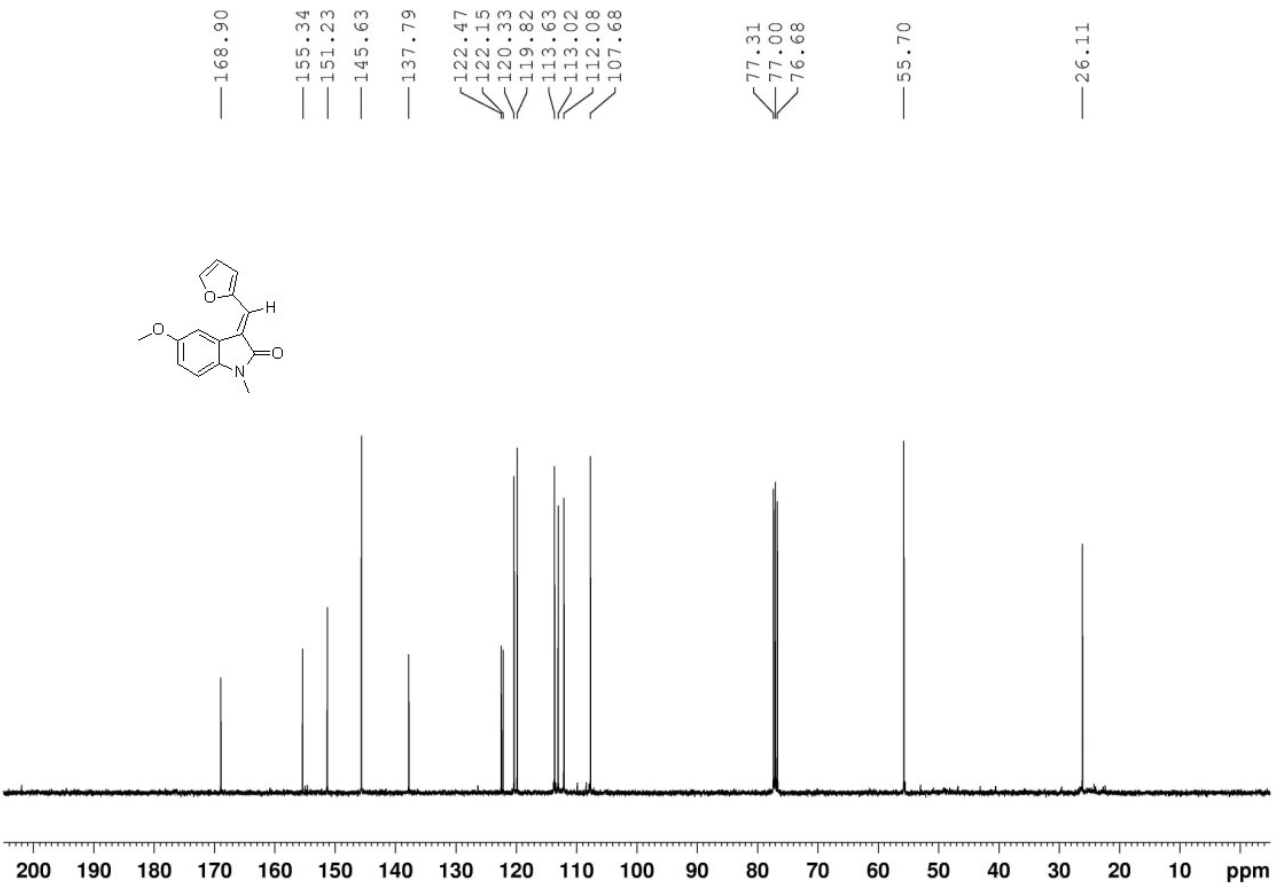
Compound **5h**


7.895
7.531
7.512
7.319
7.316
7.299
7.283
7.280
7.269
7.260
7.250
7.231
7.214
6.845
6.838
6.770
6.763
6.748
6.742
6.675
6.653
3.571
3.220
2.326
Me
H
MeO
O
N
Me
10 9 8 7 6 5 4 3 2 1 ppm
1.00
1.02
3.20
1.01
1.02
1.06
3.03
3.06
3.02

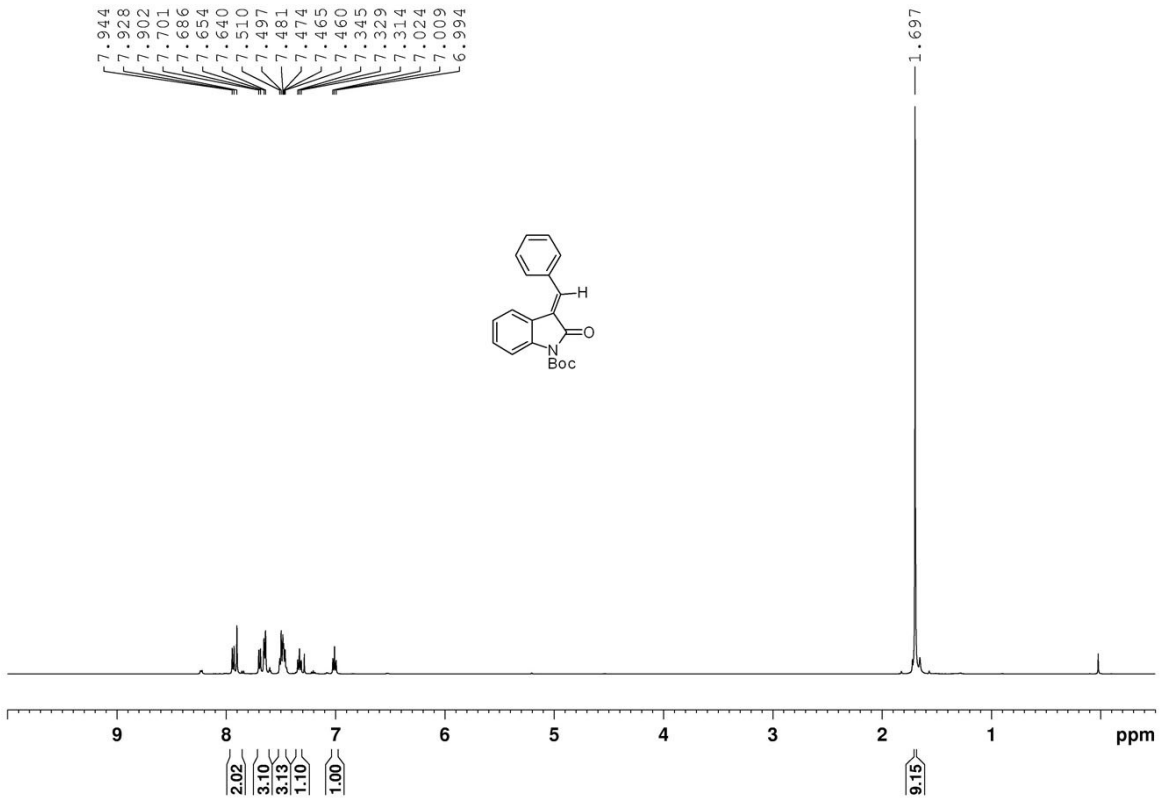

167.72
154.90
137.78
137.01
136.27
133.83
130.27
129.25
128.16
128.03
125.46
121.84
114.20
109.53
108.05
77.30
76.98
76.66
55.30
25.90
19.69
Me
H
MeO
O
N
Me
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

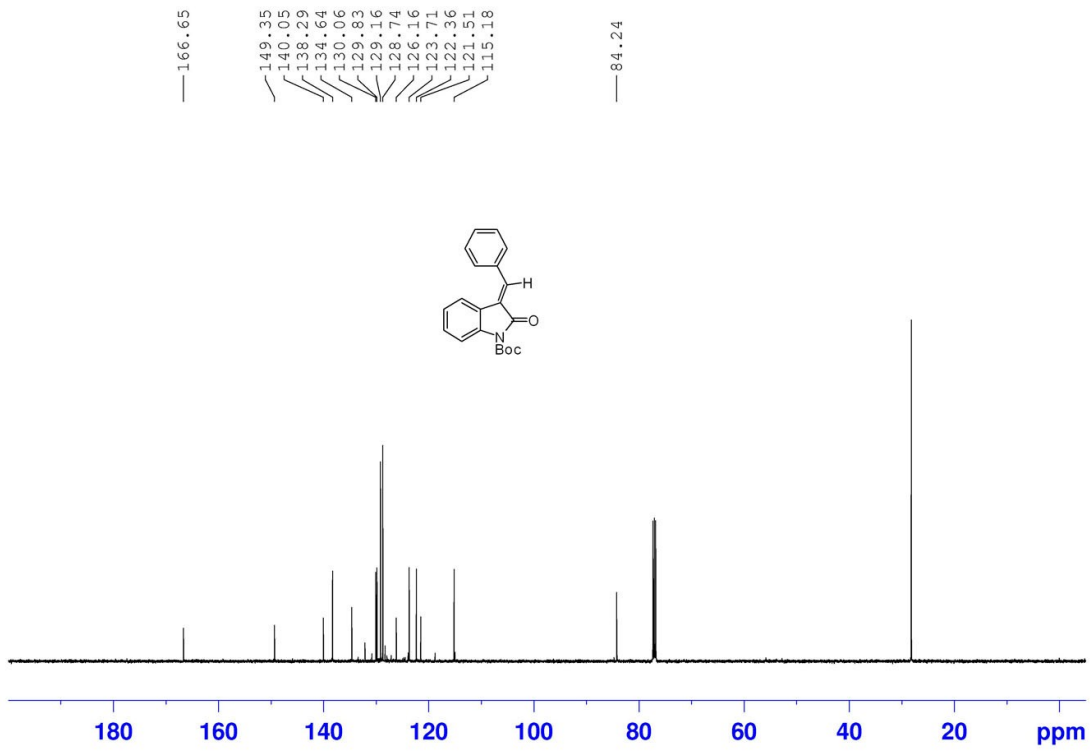
Compound **5i**


8.066
8.060
7.718
7.714
7.406
7.260
6.866
6.857
6.810
6.803
6.789
6.782
6.668
6.647
6.584
6.579
6.575
6.571
3.808
3.201
9
8
7
6
5
4
3
2
1
ppm
1.02
1.02
1.00
1.03
1.04
1.08
1.07
3.09
3.16

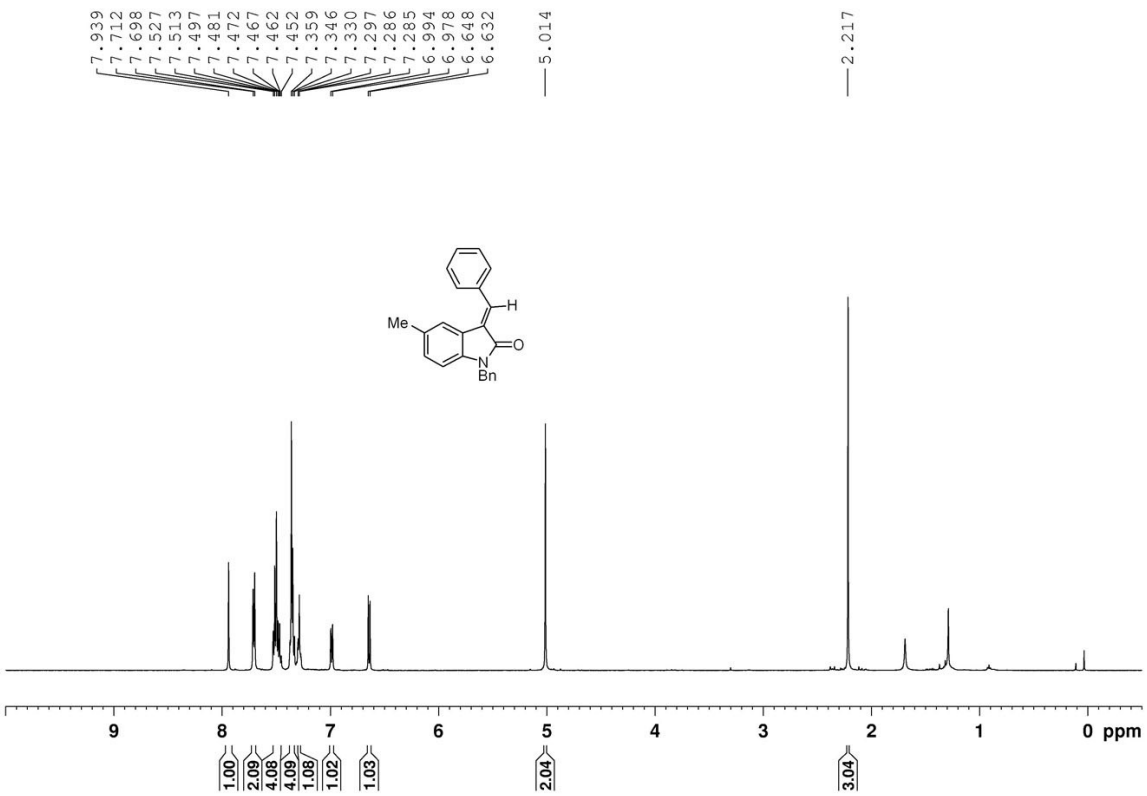

168.90
155.34
151.23
145.63
137.79
122.47
122.15
120.33
119.82
113.63
113.02
112.08
107.68
77.31
77.00
76.68
55.70
26.11
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

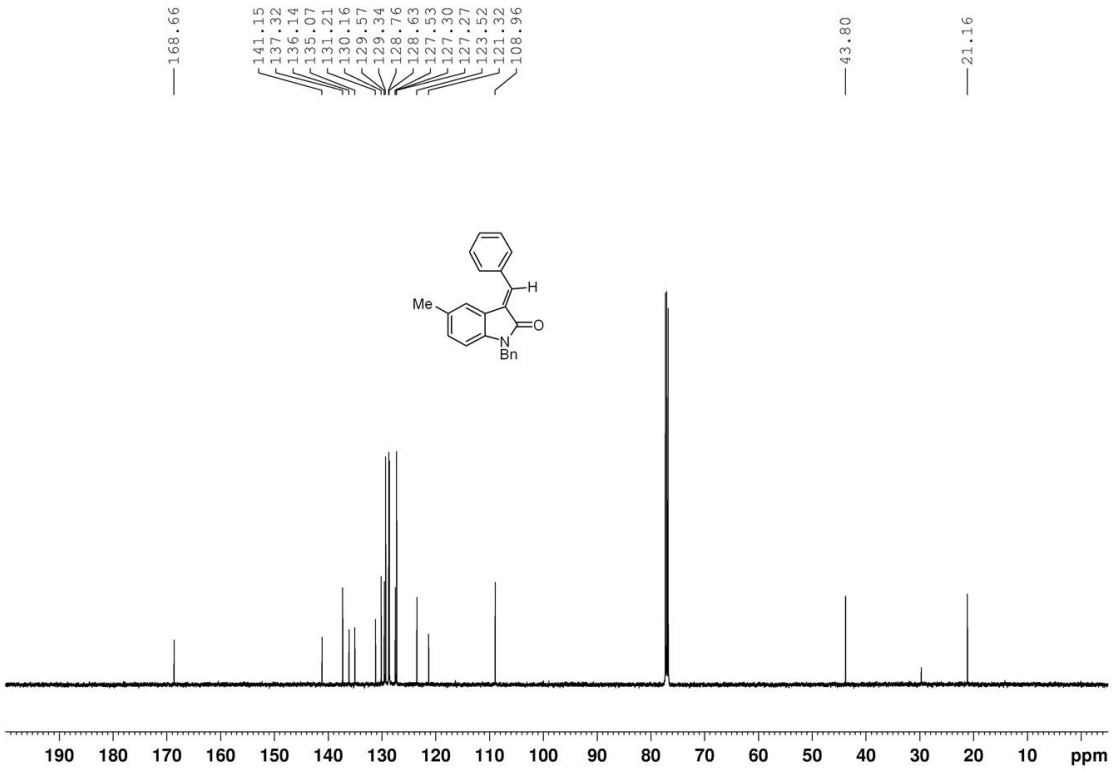
Compound **5j**


7.944
7.928
7.902
7.701
7.686
7.654
7.640
7.510
7.497
7.481
7.474
7.465
7.460
7.345
7.329
7.314
7.024
7.009
6.994
1.697
H
O
N
Boc
9
8
7
6
5
4
3
2
1
ppm
2.02
3.10
3.13
1.10
1.00
9.15


166.65
149.35
140.05
138.29
134.64
130.06
129.83
129.16
128.74
126.16
123.71
122.36
121.51
115.18
84.24
H
O
N
Boc
180
160
140
120
100
80
60
40
20
ppm

Compound **5k**


7.939
7.712
7.698
7.527
7.513
7.497
7.481
7.472
7.467
7.462
7.452
7.359
7.346
7.330
7.297
7.286
7.285
6.994
6.978
6.648
6.632
5.014
2.217
Me
H
O
N
Bn
9
8
7
6
5
4
3
2
1
0 ppm
1.00
2.09
4.08
4.09
1.08
1.02
1.03
2.04
3.04


168.66
141.15
137.32
136.14
135.07
131.21
130.16
129.57
129.34
128.76
128.63
127.53
127.30
127.27
123.52
121.32
108.96
43.80
21.16
Me
H
O
N
Bn
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

5 X-ray data for compound 3c

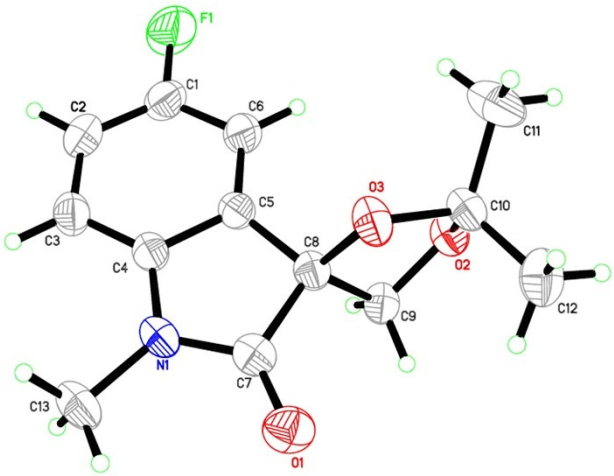


Figure 1 Single Crystal Structure of Compound **3c**

Table 1 Crystal data and structure refinement for **3c**

Identification code	121229b
Empirical formula	C13 H14 F N O3
Formula weight	251.25
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Pna2(1)
Unit cell dimensions	a = 9.209(7) A alpha = 90 deg.
	b = 8.898(7) A beta = 90 deg.
	c = 15.070(12) A gamma = 90 deg.
Volume	1234.8(17) A^3
Z, Calculated density	4, 1.352 Mg/m^3
Absorption coefficient	0.106 mm^-1
F(000)	528
Crystal size	0.20 x 0.20 x 0.09 mm
Theta range for data collection	2.66 to 25.01 deg.
Limiting indices	-10<=h<=10, -10<=k<=8, -17<=l<=16
Reflections collected / unique	5873 / 2065 [R(int) = 0.0221]
Completeness to theta = 25.01	100.0 %
Absorption correction	None
Max. and min. transmission	0.9905 and 0.9791
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2065 / 1 / 167
Goodness-of-fit on F^2	1.056
Final R indices [I>2sigma(I)]	R1 = 0.0285, wR2 = 0.0688
R indices (all data)	R1 = 0.0324, wR2 = 0.0715
Absolute structure parameter	0.1(9)
Extinction coefficient	0.0151(17)
Largest diff. peak and hole	0.080 and -0.090 e.A^-3