Supporting Information

Palladium-Catalyzed Ligand-Free Double Cyclization Reactions for the Synthesis of 3-(1'-Indolyl)-Phthalides

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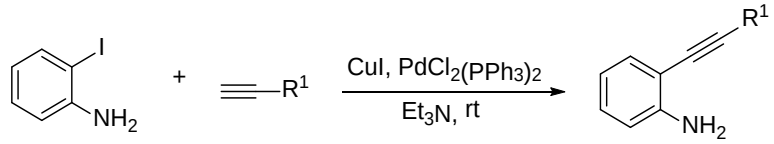
I. General Information

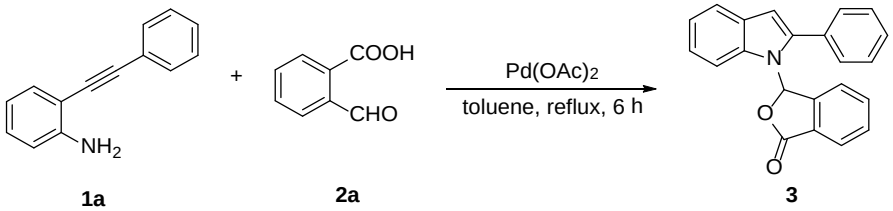
All the commercially available chemicals and solvents were used without further purification. All reagents used to prepare the substrates were purchased from Sigma-Aldrich, Innochem, Aladdin. The reactions for the synthesis of compounds **3-44** were performed in the sealed microwave reaction vials. TLC was performed using aluminum plates coated with SiO_2 (Merck 60, F-254) and visualized with UV light at 254 nm. Flash column chromatography was performed with silica gel (200-300 mesh). Melting points were determined on a Fargo MP-1D instrument. ^1H NMR and ^{13}C NMR spectra were recorded on Bruker-DRX (400 and 600 MHz for ^1H NMR, 100 and 150 MHz for ^{13}C NMR, respectively) instruments in $CDCl_3$, DMSO-*d6*, and the high temperature NMR spectrum was measured with a JOEL NMR spectrometer (JNM-ECZ400S, 400 MHz Japan). High-resolution mass spectrum (HRMS) were recorded on a Thermo Scientific Q Exactive (ESI).

II. Preparation of Substrates



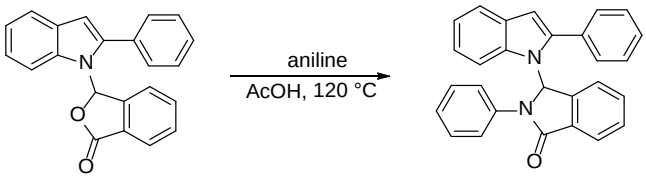
General Procedure A: To an oven dried round flask was added 2-iodoaniline (10 mmol) along with alkyne (10 mmol), CuI (5 mol%), $PdCl_2(PPh_3)_2$ (5 mol%) and Et_3N (20 mL). The mixture was stirred under argon at room temperature for 24 h. After evaporation of the solvent, the residue was purified by silica gel chromatography (PE/EA = 10/1) to afford the product 2-ethynylaniline. Other 2-ethynylaniline analogs were synthesized following this procedure.

III. General Procedure for the Synthesis of Compounds 3-44.



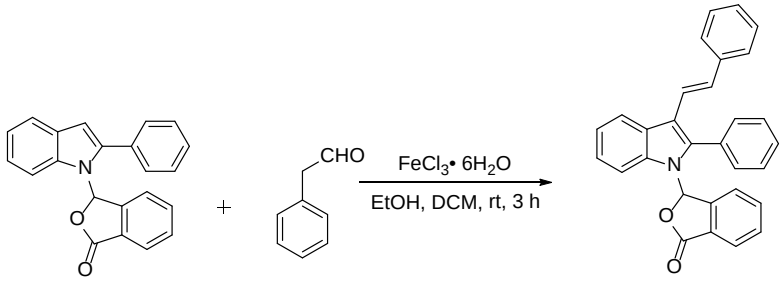
General Procedure B: To a microwave reaction tube were added 2-ethynylaniline (193 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), palladium acetate (2 mg, 1.0 mol%) and toluene (1 mL). After sealing the tube, the mixture was heated to 100 ℃ in an oil bath and stirred for 6 h. The sealed tube was cooled to room temperature, then the solvent was evaporated, and the residue was purified by silica gel chromatography (PE/EA = 10/1) to afford the product (292 mg, yield: 90%). Compounds **3-44** were prepared following the same procedure.

IV. Preparation of Compounds 45-50



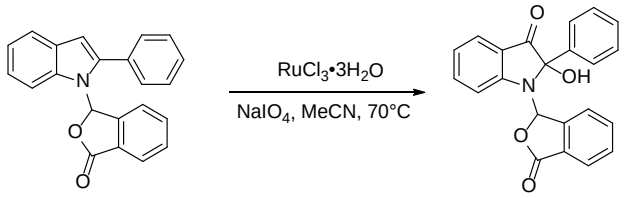
3 **45**

To a microwave reaction tube were added **3** (325 mg, 1 mmol), aniline (93 μL, 1 mmol) and glacial acetic acid (1 mL). After sealing the tube, the mixture was heated to 120 ℃ in an oil bath and stirred for 8 h. The sealed tube was cooled to room temperature, then the solvent was evaporated, and the residue was purified by silica gel chromatography (PE/EA = 5/1) to afford the product **45** (244 mg, yield: 61%).



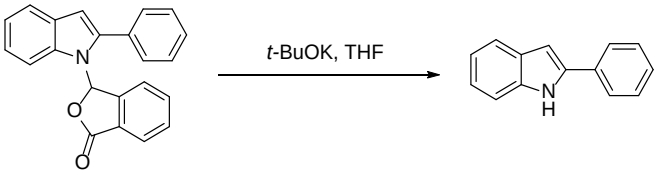
3 **46**

To a round bottom flask were added **3** (325 mg, 1 mmol), 2-phenylacetaldehyde (129 μL, 1.1 mmol), $FeCl_3{\cdot}6H_2O$ (7 mg, 2.5 mol%), EtOH (128 μL, 2.2 mmol) and DCM (3 mL). The mixture was stirred at room temperature for 3 h. Upon completion of the reaction (monitored by TLC), the solvent was evaporated, and the residue was purified by silica gel chromatography (PE/EA = 5/1) to afford the product **46** (183 mg, yield: 43%).



3 **47**

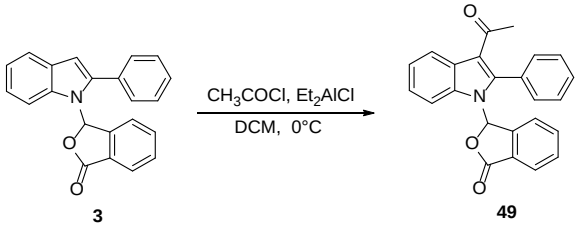
To a round bottom flask were added **3** (325 mg, 1 mmol), $NaIO_4$ (214 mg, 1 mmol), $RuCl_3{\cdot}3H_2O$ (13 mg, 5 mol%) and MeCN (5 mL). The mixture was heated to 70 ℃ in an oil bath and stirred for 9 h. Upon completion of the reaction (monitored by TLC), the solvent was evaporated, and the residue was purified by silica gel chromatography (PE/EA = 2/1) to afford the product **47** (250 mg, yield: 70%).



3 **48**

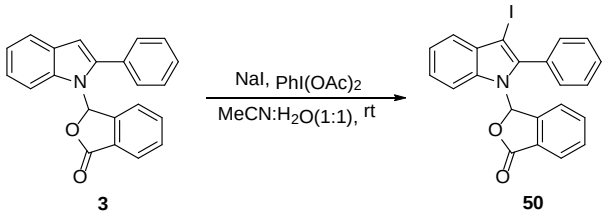
To a round bottom flask were added **3** (325 mg, 1 mmol), potassium-*t*-butoxide (112 mg, 1 mmol) and dry THF (10 mL). The mixture was under nitrogen atmosphere and

stirred at room temperature for 12 h. Upon completion of the reaction (monitored by TLC), the solvent was evaporated, and the residue was purified by silica gel chromatography (PE/EA = 20/1) to afford the product **48** (149 mg, yield: 77%).



3 **49**

To a solution of **3** (325 mg, 1 mmol) in DCM (5 mL) was added Et_2AlCl (1.53 mL, 1.5 mmol, 0.98 mol/L in hexane) at 0 ℃. The mixture was stirred at 0 ℃ for 30 min. To this solution was added acetyl chloride (85 μL, 1.2 mmol) in DCM (5 mL) dropwise at 0 ℃. The resulting solution was stirred at room temperature until completion of the reaction (monitored by TLC), and aq. NH_4Cl solution was added to quench the reaction. After the usual workup, the crude product was purified by silica gel chromatography (PE/EA = 10/1) to obtain the product **49** (260 mg, yield: 71%).

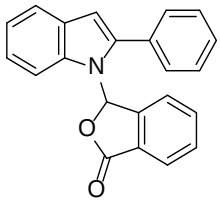


3 **50**

In a 25 mL round bottom flask, substrate **3** (325 mg, 1 mmol), NaI (150 mg, 1 mmol) and $PhI(OAc)_2$ (322 mg, 1 mmol) were dissolved in 4 mL of $MeCN:H_2O$ (v/v = 1:1). The reaction mixture was stirred at room temperature for 1-2 h. Upon completion of the reaction (monitored by TLC), the reaction mixture was diluted with 20 mL of ethyl acetate and then treated with 10 mL of saturated Na_2S aqueous solution. The organic layer was dried over Na_2SO_4 and concentrated under vacuum. The residue was purified by silica gel chromatography (PE/EA = 10/1) to give the iodinated product **50** (427 mg, yield: 95%).

V. Characterization Data

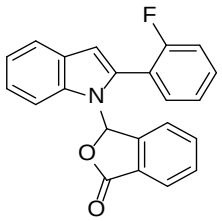
3-(2-phenyl-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (3)



The reaction was performed following the General Procedure B with 2-(phenylethynyl)aniline (193 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (292 mg, 90% yield) as a white solid; **mp** 150 - 151 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.62 - 7.72 (m, 4H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.56 - 7.43 (m, 3H), 7.42 (s, 1H), 7.33 (d, *J* = 4.5 Hz, 1H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.88 (s, 1H), 6.68 (s, 1H), 6.09 (s, 1H). **^{13}C NMR** (100

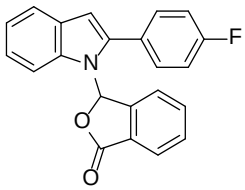
MHz, $CDCl_3$) δ 168.2, 144.9, 142.1, 134.8, 131.6, 130.9, 129.9, 129.5, 128.9, 128.8, 127.5, 126.0, 123.5, 122.7, 121.5, 121.1, 111.7, 105.6, 85.0. **HRMS (ESI) m/z**: calculated for $C_{22}H_{15}NO_2$ $[M+H]^+$: 326.1181, found: 326.1170.

3-(2-(2-fluorophenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (4)



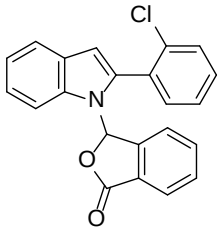
The reaction was performed following the General Procedure B with 2-((2-fluorophenyl)ethynyl)aniline (211 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (294 mg, 86% yield) as a white solid; **mp** 169 - 170 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.02 (s, 1H), 7.73 - 7.65 (m, 2H), 7.62 (d, *J* = 7.9 Hz, 2H), 7.54 (d, *J* = 6.7 Hz, 1H), 7.43 (s, 1H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.23 (s, 1H), 7.17 (s, 1H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.93 (s, 1H), 6.70 (s, 1H), 6.17 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 159.9 (d, *J* = 246 Hz), 145.1, 134.9, 132.9 (d, *J* = 2 Hz), 131.2 (d, *J* = 8 Hz), 131.0, 129.6, 127.5, 125.8, 124.9, 123.9 (d, *J* = 2 Hz), 123.1, 121.3 (d, *J* = 19 Hz), 119.5 (d, *J* = 15 Hz), 116.1, 115.8, 111.3, 107.2, 85.1. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}FNO_2$ $[M+H]^+$: 344.1087, found: 344.1071.

3-(2-(4-fluorophenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (5)



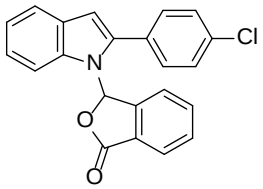
The reaction was performed following the General Procedure B with 2-((4-fluorophenyl)ethynyl)aniline (211 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (315 mg, 92% yield) as a white solid; **mp** 120 - 121 ℃; **^{1}H NMR** (600 MHz, DMSO-*d6*) δ 8.00 (s, 1H), 7.90-7.67 (m, , 3H), 7.65 (d, *J* = 8.0 Hz, 2H), 7.58 (s, 2H), 7.38 (s, 2H), 7.12 (s, 1H), 6.99 (s, 1H), 6.78 (s, 1H), 6.01 (s, 1H). **^{13}C NMR** (150 MHz, DMSO-*d6*) δ 168.3, 159.8 (d, *J* = 242 Hz), 145.4, 135.6, 133.0, 131.8 (d, *J* = 81), 127.2, 125.8, 124.1, 123.4, 121.8, 121.6, 119.4 (d, *J* = 9 Hz), 116.5 (d, *J* = 14 Hz), 111.2, 107.3, 85.2. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}FNO_2$ $[M+H]^+$: 344.1087, found: 344.1067.

3-(2-(2-chlorophenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (6)



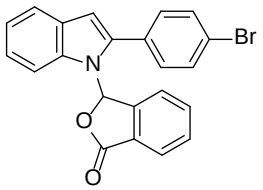
The reaction was performed following the General Procedure B with 2-((2-chlorophenyl)ethynyl)aniline (227 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (283 mg, 79% yield) as a white solid; **mp** 205 - 206 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 6.6 Hz, 1H), 7.76 - 7.68 (m, 2H), 7.68 - 7.58 (m, 3H), 7.54 (d, *J* = 6.9 Hz, 1H), 7.48 - 7.38 (m, 2H), 7.09 (t, *J* = 7.5 Hz, 1H), 7.03 (s, 1H), 6.87 (t, *J* = 7.6 Hz, 1H), 6.66 (s, 1H), 6.00 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 144.8, 138.5, 135.3, 134.9, 134.6, 133.9, 131.1, 130.9, 130.7, 129.7, 127.6, 127.4, 126.0, 124.3, 122.9, 121.4, 121.3, 111.5, 106.6, 85.1. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}ClNO_2$ $[M+H]^+$: 360.0791, found: 360.0776.

3-(2-(4-chlorophenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (7)



The reaction was performed following the General Procedure B with 2-((4-chlorophenyl)ethynyl)aniline (227 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (326 mg, 91% yield) as a white solid; **mp** 173 - 174 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.71 - 7.62 (m, 3H), 7.59 (d, *J* = 7.8 Hz, 2H), 7.46 (s, 2H), 7.35 (s, 1H), 7.30 (d, *J* = 5.3 Hz, 1H), 7.10 (t, *J* = 7.3 Hz, 1H), 6.89 (s, 1H), 6.66 (s, 1H), 6.04 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 144.6, 140.8, 135.0, 134.8, 131.1, 131.0, 130.1, 129.4, 129.1, 127.4, 126.0, 123.4, 122.9, 121.6, 121.1, 111.7, 106.0, 84.8. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}ClNO_2$ $[M+H]^+$: 360.0791, found: 360.0767.

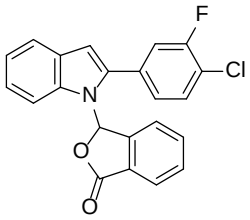
3-(2-(4-bromophenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (8)



The reaction was performed following the General Procedure B with 2-((4-bromophenyl)ethynyl)aniline (272 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (347 mg, 86% yield) as a white solid; **mp** 158 - 159 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.80 - 7.63 (m, 3H), 7.62 - 7.45 (m, 4H), 7.36 (s, 1H), 7.30 (d, *J* = 5.6 Hz, 1H), 7.10 (t, *J* = 7.3 Hz,

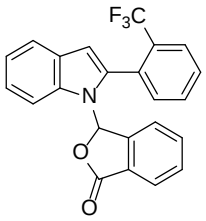
1H), 6.89 (s, 1H), 6.67 (s, 1H), 6.04 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 144.6, 140.7, 134.8, 132.1, 131.3, 131.0, 130.5, 129.4, 127.5, 126.0, 123.4, 123.2, 122.9, 121.6, 121.2, 111.7, 105.9, 84.8. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}BrNO_2$ $[M+H]^+$: 404.0286, found: 404.0266.

3-(2-(4-chloro-3-fluorophenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (9)



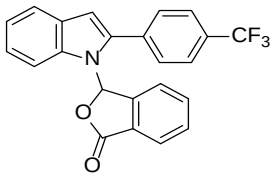
The reaction was performed following the General Procedure B with 2-((4-chloro-3-fluorophenyl)ethynyl)aniline (245 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (339 mg, 90% yield) as a white solid; **mp** 176 - 177 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.67 (s, 3H), 7.60 (d, *J* = 7.8 Hz, 2H), 7.31 (s, 3H), 7.12 (s, 1H), 6.91 (s, 1H), 6.66 (s, 1H), 6.02 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.9, 158.4 (d, *J* = 251 Hz), 144.5, 139.4, 134.8, 132.0, 131.1, 129.8 (d, *J* = 8 Hz), 129.2, 128.9, 127.5, 126.0, 123.3 (d, *J* = 29 Hz), 121.5 (d, *J* = 46 Hz), 117.0 (d, *J* = 18 Hz), 111.6, 106.5, 84.7. **HRMS (ESI) m/z**: calculated for $C_{22}H_{13}ClFNO_2$ $[M+H]^+$: 378.0697, found: 378.0671.

3-(2-(2-(trifluoromethyl)phenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (10)



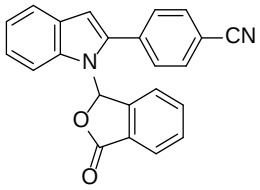
The reaction was performed following the General Procedure B with 2-((2-(trifluoromethyl)phenyl)ethynyl)aniline (261 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (326 mg, 83% yield) as a white solid; **mp** 169 - 170 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.10 - 8.01 (m, 1H), 7.86 (d, *J* = 7.8 Hz, 1H), 7.76 - 7.71 (m, 2H), 7.70 - 7.66 (m, 2H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.39 - 7.32 (m, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.89 (s, 1H), 6.87 (t, *J* = 7.4 Hz, 1H), 6.68 (s, 1H), 5.96 (d, *J* = 8.4 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 144.5, 136.9, 135.0, 134.9, 134.4, 133.1, 131.9, 131.1, 130.7 (q, *J* = 30 Hz), 130.0 (d, *J* = 2 Hz), 129.7, 128.4 (q, *J* = 190 Hz), 126.2 (q, *J* = 5 Hz), 125.9, 123.9 (d, *J* = 1 Hz), 123.2, 122.9, 122.6, 121.6, 121.4, 121.3, 111.4, 107.3, 85.0. **HRMS (ESI) m/z**: calculated for $C_{23}H_{14}F_3NO_2$ $[M+H]^+$: 394.1055, found: 394.1026.

3-(2-(4-(trifluoromethyl)phenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (11)



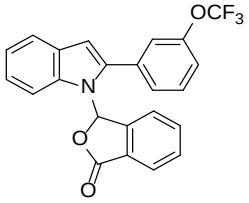
The reaction was performed following the General Procedure B with 2-((4-(trifluoromethyl)phenyl)ethynyl)aniline (261 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (377 mg, 96% yield) as a white solid; **mp** 203 - 204 ℃; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.75 (s, 4H), 7.66 (s, 2H), 7.62 (d, *J* = 7.8 Hz, 1H), 7.36 (s, 1H), 7.30 (s, 1H), 7.12 (s, 1H), 6.91 (s, 1H), 6.73 (s, 1H), 6.03 (s, 1H). **^{13}C NMR** (150 MHz, $CDCl_3$) δ 168.0, 144.5, 135.3, 134.9, 131.1, 130.1, 129.3, 127.5, 126.0 (d, *J* = 32 Hz), 123.9 (q, *J* = 270 Hz), 123.4 (d, *J* = 27 Hz), 121.8, 121.4, 111.9, 106.8, 84.8. **HRMS (ESI) m/z**: calculated for $C_{23}H_{14}F_3NO_2$ $[M+H]^+$: 394.1055, found: 394.1029.

4-(1-(3-oxo-1,3-dihydroisobenzofuran-1-yl)-*1H*-indol-2-yl)benzonitrile (12)



The reaction was performed following the General Procedure B with 4-((2-aminophenyl)ethynyl)benzonitrile (218 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (297 mg, 85% yield) as a pink solid; **mp** 194 - 195 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.77 (s, 3H), 7.67 (s, 2H), 7.63 (d, *J* = 8.0 Hz, 2H), 7.40 - 7.26 (m, 2H), 7.14 (t, *J* = 7.3 Hz, 1H), 6.94 (s, 1H), 6.76 (s, 1H), 6.05 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.8, 144.3, 134.9, 132.7, 131.2, 130.2, 127.4, 126.2, 125.0, 123.6, 123.5, 121.9, 121.5, 118.4, 84.7. **HRMS (ESI) m/z**: calculated for $C_{23}H_{14}N_2O_2$ $[M+H]^+$: 351.1134, found: 351.1110.

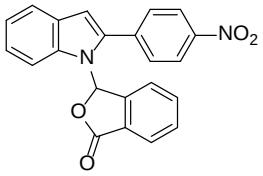
3-(2-(3-(trifluoromethoxy)phenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (13)



The reaction was performed following the General Procedure B with 2-((3-(trifluoromethoxy)phenyl)ethynyl)aniline (277 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (270 mg, 66% yield) as a yellow solid; **mp** 161 - 162 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.67 (s, 2H), 7.61 (d, *J* = 7.9 Hz, 2H), 7.52 (s, 2H), 7.38 (s, 1H), 7.32 (s, 2H), 7.12 (t, *J* = 7.2 Hz, 1H), 6.92 (s, 1H), 6.71 (s, 1H), 6.05 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 149.5, 144.6, 140.2, 134.9, 133.7, 131.1, 130.4, 129.3, 128.2, 127.5, 126.0,

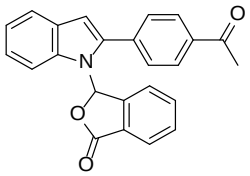
123.5, 123.2, 121.6 (q, *J* = 40 Hz), 121.8, 119.2, 111.7, 106.6, 84.8. **HRMS (ESI) m/z**: calculated for $C_{23}H_{14}F_3NO_3$ $[M+H]^+$: 410.1004, found: 410.0974.

3-(2-(4-nitrophenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (14)



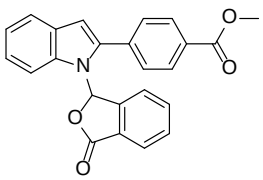
The reaction was performed following the General Procedure B with 2-((4-nitrophenyl)ethynyl)aniline (238 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (303 mg, 82% yield) as a yellow solid; **mp** 189 - 190 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.34 (s, 2H), 8.06 (s, 1H), 7.86 (s, 2H), 7.68 (s, 2H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.37 (s, 1H), 7.30 (d, *J* = 4.8 Hz, 1H), 7.14 (t, *J* = 6.9 Hz, 1H), 6.95 (s, 1H), 6.81 (s, 1H), 6.05 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.8, 147.6, 144.3, 134.9, 131.3, 130.3, 127.4, 126.2, 124.1, 123.8, 123.5, 122.0, 121.6, 107.8, 84.7. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}N_2O_4$ $[M+H]^+$: 371.1032, found: 371.1001.

3-(2-(4-acetylphenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (15)



The reaction was performed following the General Procedure B with 1-(4-((2-aminophenyl)ethynyl)phenyl)ethan-1-one (235 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (260 mg, 71% yield) as a white solid; **mp** 188 - 189 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.08 (s, 3H), 7.89 - 7.74 (m, 2H), 7.73 - 7.64 (m, 2H), 7.62 (d, *J* = 7.9 Hz, 1H), 7.40 (s, 1H), 7.32 (d, *J* = 4.5 Hz, 1H), 7.12 (t, *J* = 7.3 Hz, 1H), 6.92 (s, 1H), 6.76 (s, 1H), 6.05 (s, 1H), 2.65 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 197.5, 168.1, 144.6, 136.8, 136.2, 134.9, 131.1, 129.8, 128.9, 127.5, 126.1, 123.5, 123.3, 121.7, 121.4, 111.9, 106.9, 84.9, 26.7. **HRMS (ESI) m/z**: calculated for $C_{24}H_{17}NO_3$ $[M+H]^+$: 368.1287, found: 368.1259.

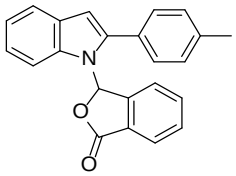
methyl 4-(1-(3-oxo-1,3-dihydroisobenzofuran-1-yl)-*1H*-indol-2-yl)benzoate (16)



The reaction was performed following the General Procedure B with methyl 4-((2-aminophenyl)ethynyl)benzoate (251 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (356 mg, 93% yield) as a

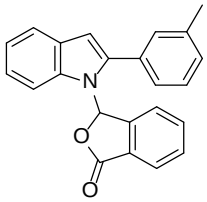
white solid; **mp** 135 - 136 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.15 (s, 2H), 8.06 (s, 1H), 7.75 (s, 2H), 7.67 (s, 2H), 7.62 (d, *J* = 7.9 Hz, 1H), 7.40 (s, 1H), 7.32 (d, *J* = 4.3 Hz, 1H), 7.12 (t, *J* = 7.3 Hz, 1H), 6.92 (s, 1H), 6.75 (s, 1H), 6.07 (s, 1H), 3.96 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 166.6, 144.6, 136.1, 134.9, 131.1, 130.1, 129.6, 127.5, 126.1, 123.5, 123.2, 121.7, 121.3, 106.7, 84.9, 52.3. **HRMS (ESI) m/z**: calculated for $C_{24}H_{17}NO_4$ $[M+H]^+$: 384.1236, found: 384.1211.

3-(2-(p-tolyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (17)



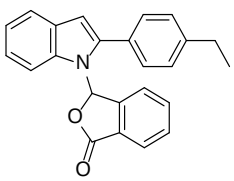
The reaction was performed following the General Procedure B with 2-(p-tolylethynyl)aniline (207 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (247 mg, 73% yield) as a white solid; **mp** 179 - 180 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.77 - 7.61 (m, 2H), 7.61 - 7.51 (m, 3H), 7.41 (s, 1H), 7.38 - 7.26 (m, 3H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.86 (s, 1H), 6.64 (s, 1H), 6.06 (s, 1H), 2.42 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 145.0, 142.2, 138.9, 134.8, 130.9, 129.8, 129.6, 128.7, 127.5, 126.0, 123.5, 122.5, 121.4, 121.0, 111.7, 105.2, 85.1, 21.3. **HRMS (ESI) m/z**: calculated for $C_{23}H_{17}NO_2$ $[M+H]^+$: 340.1338, found: 340.1319.

3-(2-(m-tolyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (18)



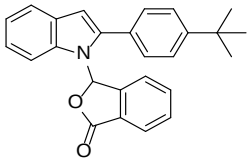
The reaction was performed following the General Procedure B with 2-(m-tolylethynyl)aniline (207 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (274 mg, 81% yield) as a yellow solid; **mp** 108 - 109 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.65 (s, 2H), 7.59 (d, *J* = 7.9 Hz, 1H), 7.55 - 7.35 (m, 4H), 7.32 (d, *J* = 4.2 Hz, 1H), 7.25 (s, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.88 (s, 1H), 6.65 (s, 1H), 6.09 (s, 1H), 2.43 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 145.0, 142.3, 138.7, 134.7, 131.6, 130.9, 130.6, 129.6, 128.7, 127.5, 127.0, 125.9, 123.4, 122.6, 121.4, 121.0, 111.6, 105.4, 85.0, 21.5. **HRMS (ESI) m/z**: calculated for $C_{23}H_{17}NO_2$ $[M+H]^+$: 340.1338, found: 340.1314.

3-(2-(4-ethylphenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (19)



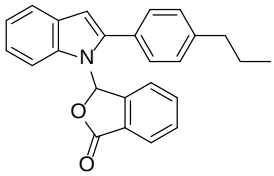
The reaction was performed following the General Procedure B with 2-((4-ethylphenyl)ethynyl)aniline (221 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (271 mg, 77% yield) as a white solid; **mp** 175 - 176 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.72 - 7.61 (m, 2H), 7.58 (d, *J* = 7.8 Hz, 3H), 7.42 (s, 1H), 7.38 - 7.26 (m, 3H), 7.08 (t, *J* = 7.4 Hz, 1H), 6.86 (s, 1H), 6.64 (s, 1H), 6.07 (s, 1H), 2.71 (q, *J* = 7.6 Hz, 2H), 1.28 (t, *J* = 7.6 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 145.2, 145.0, 142.3, 134.8, 130.9, 129.9, 129.6, 128.9, 128.4, 127.5, 125.9, 123.5, 122.5, 121.4, 121.0, 111.7, 105.2, 85.1, 28.7, 15.5. **HRMS (ESI) m/z**: calculated for $C_{24}H_{19}NO_2$ $[M+H]^+$: 354.1494, found: 354.1472.

3-(2-(4-(tert-butyl)phenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (20)



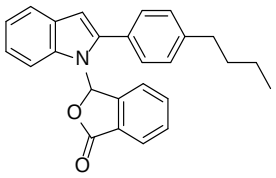
The reaction was performed following the General Procedure B with 2-((4-(tert-butyl)phenyl)ethynyl)aniline (249 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (289 mg, 76% yield) as a white solid; **mp** 225 - 226 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.63 (s, 3H), 7.58 (d, *J* = 7.8 Hz, 2H), 7.52 (s, 2H), 7.43 (s, 1H), 7.31 (d, *J* = 4.5 Hz, 1H), 7.08 (t, *J* = 7.4 Hz, 1H), 6.86 (s, 1H), 6.64 (s, 1H), 6.08 (s, 1H), 1.36 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 152.0, 145.0, 142.2, 134.7, 130.9, 129.7, 128.7, 127.6, 125.9, 125.9, 123.5, 122.5, 121.4, 121.0, 111.6, 105.2, 85.1, 34.8, 31.3. **HRMS (ESI) m/z**: calculated for $C_{26}H_{23}NO_2$ $[M+H]^+$: 382.1807, found: 382.1781.

3-(2-(4-propylphenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (21)



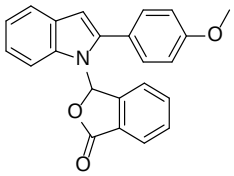
The reaction was performed following the General Procedure B with 2-((4-propylphenyl)ethynyl)aniline (235 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (267 mg, 73% yield) as a yellow solid; **mp** 165 - 166 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.70 - 7.61 (m, 2H), 7.58 (d, *J* = 7.8 Hz, 3H), 7.42 (s, 1H), 7.36 - 7.25 (m, 3H), 7.08 (t, *J* = 7.4 Hz, 1H), 6.86 (s, 1H), 6.64 (s, 1H), 6.07 (s, 1H), 2.64 (t, *J* = 7.5 Hz, 2H), 1.73 - 1.64 (m, 2H), 0.98 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 145.0, 143.6, 142.2, 134.7, 130.9, 129.8, 129.6, 129.0, 128.9, 127.5, 125.9, 123.5, 122.4, 121.4, 120.9, 111.6, 105.2, 85.1, 37.8, 24.5, 13.9. **HRMS (ESI) m/z**: calculated for $C_{25}H_{21}NO_2$ $[M+H]^+$: 368.1651, found: 368.1626.

3-(2-(4-butylphenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (22)



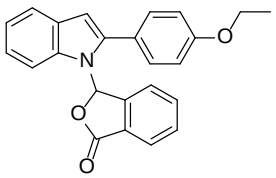
The reaction was performed following the General Procedure B with 2-((4-butylphenyl)ethynyl)aniline (249 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (297 mg, 78% yield) as a white solid; **mp** 130 - 131 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.71 - 7.60 (m, 2H), 7.58 (d, *J* = 7.8 Hz, 3H), 7.42 (s, 1H), 7.36 - 7.24 (m, 3H), 7.08 (t, *J* = 7.4 Hz, 1H), 6.86 (s, 1H), 6.64 (s, 1H), 6.07 (s, 1H), 2.67 (t, *J* = 7.5 Hz, 2H), 1.68 - 1.60 (m, 2H), 1.44 - 1.34 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 145.0, 143.8, 142.3, 134.7, 130.9, 129.8, 129.6, 128.9, 128.8, 127.5, 125.9, 123.5, 122.4, 121.4, 120.9, 111.6, 105.2, 85.1, 35.4, 33.5, 22.4, 13.9. **HRMS (ESI) m/z**: calculated for $C_{26}H_{23}NO_2$ $[M+H]^+$: 382.1807, found: 382.1780.

3-(2-(4-methoxyphenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (23)



The reaction was performed following the General Procedure B with 2-((4-methoxyphenyl)ethynyl)aniline (223 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (291 mg, 82% yield) as a white solid; **mp** 139 - 140 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.71 - 7.63 (m, 3H), 7.58 (d, *J* = 7.8 Hz, 2H), 7.40 (s, 1H), 7.32 (d, *J* = 4.9 Hz, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 7.03 (s, 2H), 6.86 (s, 1H), 6.62 (s, 1H), 6.04 (s, 1H), 3.86 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 160.1, 144.9, 142.0, 134.7, 131.3, 130.9, 129.6, 127.5, 126.0, 123.9, 123.5, 122.4, 121.4, 120.9, 114.4, 111.6, 105.0, 85.0, 55.4. **HRMS (ESI) m/z**: calculated for $C_{23}H_{17}NO_3$ $[M+H]^+$: 356.1287, found: 356.1268.

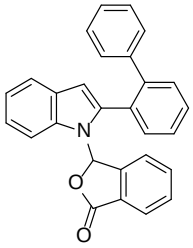
3-(2-(4-ethoxyphenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (24)



The reaction was performed following the General Procedure B with 2-((4-ethoxyphenyl)ethynyl)aniline (237 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (291 mg, 79% yield) as a white solid; **mp** 158 - 159 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.70 - 7.62 (m, 2H), 7.57 (d, *J* = 7.8 Hz, 3H), 7.40 (s, 1H), 7.31 (d, *J* = 5.1 Hz, 1H), 7.08 (t, *J* = 7.5

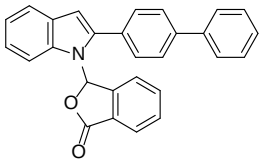
Hz, 1H), 7.01 (s, 2H), 6.85 (s, 1H), 6.61 (s, 1H), 6.04 (s, 1H), 4.09 (q, *J* = 6.9 Hz, 2H), 1.45 (t, *J* = 7.0 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 159.5, 145.0, 142.0, 134.7, 131.2, 130.9, 129.6, 127.5, 126.0, 123.7, 123.5, 122.3, 121.4, 120.9, 114.9, 111.6, 105.0, 85.1, 63.7, 14.8. **HRMS (ESI) m/z**: calculated for $C_{24}H_{19}NO_3$ $[M+H]^+$: 370.1443, found: 370.1419.

3-(2-([1,1'-biphenyl]-2-yl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (25)



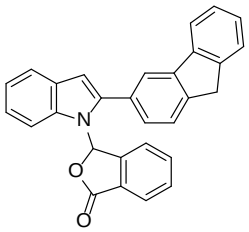
The reaction was performed following the General Procedure B with 2-([1,1'-biphenyl]-2-ylethynyl)aniline (269 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (280 mg, 70% yield) as a white solid; **mp** 207 - 208 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.6 Hz, 1H), 7.69 (d, *J* = 7.3 Hz, 1H), 7.63 - 7.55 (m, 3H), 7.54 - 7.48 (m, 2H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.38 (d, *J* = 7.0 Hz, 2H), 7.34 (d, *J* = 6.9 Hz, 1H), 7.32 - 7.25 (m, 2H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.87 (s, 1H), 6.81 - 6.67 (m, 2H), 5.83 (d, *J* = 7.6 Hz, 1H), 5.63 (d, *J* = 8.3 Hz, 1H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 144.6, 141.6, 141.1, 140.8, 135.2, 134.3, 133.6, 130.7, 130.5, 130.0, 129.9, 129.6, 129.3, 128.9, 128.1, 127.3, 125.6, 123.7, 122.4, 121.2, 120.9, 111.4, 107.1, 85.0. **HRMS (ESI) m/z**: calculated for $C_{28}H_{19}NO_2$ $[M+H]^+$: 402.1494, found: 402.1468.

3-(2-([1,1'-biphenyl]-4-yl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (26)



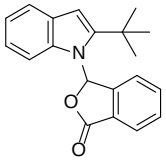
The reaction was performed following the General Procedure B with 2-([1,1'-biphenyl]-4-ylethynyl)aniline (269 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (352 mg, 88% yield) as a yellow solid; **mp** 151 - 152 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.82 - 7.69 (m, 4H), 7.68 - 7.58 (m, 5H), 7.53 - 7.43 (m, 3H), 7.41 - 7.35 (m, 1H), 7.33 (d, *J* = 4.4 Hz, 1H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.89 (s, 1H), 6.72 (s, 1H), 6.08 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 144.9, 141.8, 140.3, 134.8, 131.0, 130.5, 130.3, 129.0, 127.8, 127.6, 127.1, 126.0, 123.5, 122.7, 121.5, 121.1, 111.8, 105.7, 85.1. **HRMS (ESI) m/z**: calculated for $C_{28}H_{19}NO_2$ $[M+H]^+$: 402.1494, found: 402.1470.

3-(2-(*9H*-fluoren-3-yl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (27)



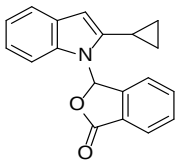
The reaction was performed following the General Procedure B with 2-((9H-fluoren-3-yl)ethynyl)aniline (281 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (276 mg, 67% yield) as a white solid; **mp** 191 - 192 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.94 - 7.76 (m, 3H), 7.64 (s, 2H), 7.63 - 7.54 (m, 3H), 7.50 (s, 1H), 7.41 (t, *J* = 7.3 Hz, 1H), 7.36 (dd, *J* = 7.4, 0.9 Hz, 1H), 7.34 - 7.29 (m, 1H), 7.11 (t, *J* = 7.4 Hz, 1H), 6.90 (s, 1H), 6.71 (s, 1H), 6.09 (s, 1H), 3.98 (s, 2H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 144.9, 143.6, 140.9, 134.7, 130.9, 129.8, 128.7, 127.5, 127.3, 127.0, 126.5, 125.9, 125.2, 123.5, 122.6, 121.4, 121.0, 120.2, 111.7, 105.6, 85.1, 37.0. **HRMS (ESI) m/z**: calculated for $C_{29}H_{19}NO_2$ $[M+H]^+$: 414.1494, found: 414.1465.

3-(2-(tert-butyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (28)



The reaction was performed following the General Procedure B with 2-(3,3-dimethylbut-1-yn-1-yl)aniline (173 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (213 mg, 73% yield) as a white solid; **mp** 158 - 159 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.16 - 8.07 (m, 1H), 7.84 (s, 1H), 7.76 - 7.61 (m, 2H), 7.47 (d, *J* = 7.7 Hz, 1H), 7.44 - 7.37 (m, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.74 (t, *J* = 7.8 Hz, 1H), 6.42 (s, 1H), 5.92 (d, *J* = 8.4 Hz, 1H), 1.62 (s, 9H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 149.4, 145.3, 136.3, 135.0, 131.0, 129.1, 127.6, 126.1, 123.2, 122.0, 120.9, 120.6, 111.5, 102.1, 85.6, 32.4, 31.1. **HRMS (ESI) m/z**: calculated for $C_{20}H_{19}NO_2$ $[M+H]^+$: 306.1494, found: 306.1477.

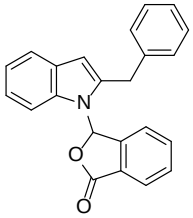
3-(2-cyclopropyl-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (29)



The reaction was performed following the General Procedure B with 2-(cyclopropylethynyl)aniline (157 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (216 mg, 75% yield) as a white solid; **mp** 95 - 96 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.80 (s, 1H), 7.76 - 7.64 (m, 2H), 7.55 - 7.38 (m, 2H), 7.02 (s, 1H), 6.78 (s, 1H), 6.29 (s, 1H), 5.88 (s, 1H), 2.03 (s, 1H), 1.18 - 0.70 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.5, 145.2,

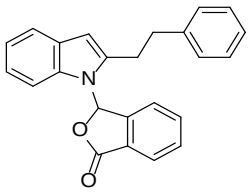
143.3, 134.8, 131.0, 127.7, 126.0, 123.6, 122.1, 121.0, 120.6, 111.0, 102.9, 84.4, 29.7, 6.8. **HRMS (ESI) m/z**: calculated for $C_{19}H_{15}NO_2$ $[M+H]^+$: 290.1181, found: 290.1160.

3-(2-benzyl-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (30)



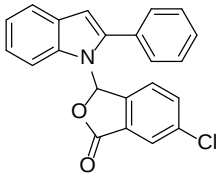
The reaction was performed following the General Procedure B with 2-(3-phenylprop-1-yn-1-yl)aniline (207 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (267 mg, 79% yield) as a yellow solid; **mp** 133 - 134 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 7.4 Hz, 1H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.50 (d, *J* = 7.9 Hz, 1H), 7.43 - 7.23 (m, 6H), 7.03 (s, 1H), 6.94 (s, 1H), 6.78 (s, 1H), 6.47 (s, 1H), 5.90 (s, 1H), 4.41 (s, 1H), 4.32 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 144.7, 139.2, 137.9, 134.8, 131.0, 129.5, 128.9, 128.5, 127.4, 127.1, 125.9, 123.5, 122.2, 121.1, 120.7, 111.2, 105.7, 84.4, 33.6. **HRMS (ESI) m/z**: calculated for $C_{23}H_{17}NO_2$ $[M+H]^+$: 340.1338, found: 340.1317.

3-(2-phenethyl-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (31)



The reaction was performed following the General Procedure B with 2-(4-phenylbut-1-yn-1-yl)aniline (221 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (250 mg, 71% yield) as a white solid; **mp** 162 - 163 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.76 - 7.65 (m, 2H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.41 - 7.15 (m, 7H), 7.02 (s, 1H), 6.75 (s, 1H), 6.43 (s, 1H), 5.83 (s, 1H), 3.44 - 3.02 (m, 4H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 144.7, 140.6, 134.9, 131.1, 128.6, 128.4, 127.6, 126.4, 126.1, 123.6, 121.9, 121.0, 120.5, 111.1, 103.6, 84.1, 35.1, 28.9. **HRMS (ESI) m/z**: calculated for $C_{24}H_{19}NO_2$ $[M+H]^+$: 354.1494, found: 354.1472.

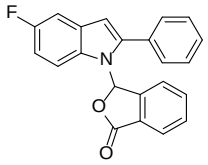
6-chloro-3-(2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (32)



The reaction was performed following the General Procedure B with 2-(phenylethynyl)aniline (193 mg, 1 mmol), 5-chloro-2-formylbenzoic acid (277 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel

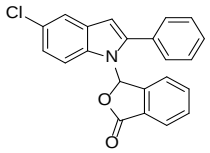
chromatography (PE/EA = 10/1) to give the desired product (298 mg, 83% yield) as a white solid; **mp** 154 - 155 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.70 - 7.57 (m, 4H), 7.55 - 7.42 (m, 3H), 7.40 (s, 1H), 7.25 (d, *J* = 6.0 Hz, 1H), 7.13 (t, *J* = 7.4 Hz, 1H), 6.95 (s, 1H), 6.68 (s, 1H), 6.12 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 166.7, 143.0, 142.0, 137.4, 135.0, 131.5, 129.9, 129.4, 128.9, 126.0, 124.7, 122.9, 121.7, 121.2, 111.5, 105.8, 84.9. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}ClNO_2$ $[M+H]^+$: 360.0791, found: 360.0792.

3-(5-fluoro-2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (33)



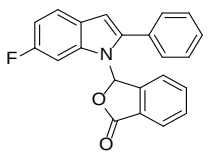
The reaction was performed following the General Procedure B with 4-fluoro-2-(phenylethynyl)aniline (211 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (278 mg, 81% yield) as a white solid; **mp** 184 - 185 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.73 - 7.61 (m, 4H), 7.56 - 7.42 (m, 3H), 7.39 (s, 1H), 7.36 - 7.30 (m, 1H), 7.27 - 7.21 (m, 1H), 6.68 - 6.58 (m, 2H), 6.00 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 158.6 (d, *J* = 236 Hz), 144.6, 143.8, 134.9, 131.3, 131.1, 129.9, 129.0 (d, *J* = 6 Hz), 127.5, 126.1, 123.4, 112.3 (d, *J* = 9 Hz), 110.8 (d, *J* = 26 Hz), 106.1 (d, *J* = 23 Hz), 105.4, 84.9. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}FNO_2$ $[M+H]^+$: 344.1087, found: 344.1088.

3-(5-chloro-2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (34)



The reaction was performed following the General Procedure B with 4-chloro-2-(phenylethynyl)aniline (227 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (276 mg, 77% yield) as a white solid; **mp** 167 - 168 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.76 - 7.60 (m, 4H), 7.59 - 7.54 (m, 1H), 7.54 - 7.40 (m, 3H), 7.38 (s, 1H), 7.31 (d, *J* = 4.6 Hz, 1H), 6.82 (s, 1H), 6.62 (s, 1H), 5.98 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 144.5, 143.5, 134.9, 131.1, 129.9, 129.1, 129.0, 127.4, 127.2, 126.1, 125.5, 123.4, 122.8, 120.5, 112.5, 104.9, 102.3, 84.8. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}ClNO_2$ $[M+Na]^+$: 382.0611, found: 382.0610.

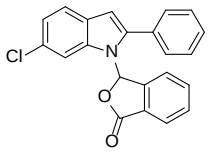
3-(6-fluoro-2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (35)



The reaction was performed following the General Procedure B with 5-fluoro-2-(phenylethynyl)aniline (211 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol),

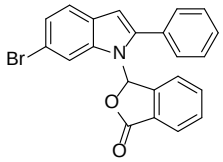
and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (260 mg, 76% yield) as a white solid; **mp** 151 - 152 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.76 - 7.60 (m, 4H), 7.57 - 7.41 (m, 4H), 7.38 (s, 1H), 7.37 - 7.32 (m, 1H), 6.86 (td, *J* = 9.2, 1.9 Hz, 1H), 6.64 (s, 1H), 5.74 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 159.5 (d, *J* = 238 Hz), 144.3, 142.6, 134.9, 131.3, 131.2, 129.9, 128.9 (d, *J* = 6 Hz), 127.4, 126.2, 125.9, 123.4, 121.7 (d, *J* = 10 Hz), 110.0 (d, *J* = 25 Hz), 105.3, 98.6 (d, *J* = 30 Hz), 84.8. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}FNO_2$ $[M+H]^+$: 344.1087, found: 344.1089.

3-(6-chloro-2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (36)



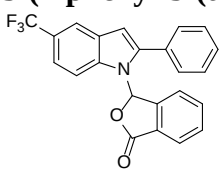
The reaction was performed following the General Procedure B with 5-chloro-2-(phenylethynyl)aniline (227 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (293 mg, 82% yield) as a white solid; **mp** 138 - 139 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.76 - 7.58 (m, 4H), 7.56 - 7.41 (m, 4H), 7.37 (s, 1H), 7.36 - 7.29 (m, 1H), 7.07 (d, *J* = 8.2 Hz, 1H), 6.64 (s, 1H), 6.01 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 144.3, 142.8, 135.0, 131.2, 131.2, 129.9, 129.0, 129.0, 128.4, 128.0, 127.4, 126.2, 123.4, 122.2, 121.8, 111.7, 105.4, 84.7. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}ClNO_2$ $[M+H]^+$: 360.0791, found: 360.0791.

3-(6-bromo-2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (37)



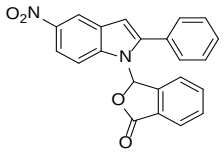
The reaction was performed following the General Procedure B with 5-bromo-2-(phenylethynyl)aniline (272 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (319 mg, 79% yield) as a white solid; **mp** 159 - 160 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.77 - 7.59 (m, 4H), 7.56 - 7.42 (m, 4H), 7.37 (s, 1H), 7.35 - 7.29 (m, 1H), 7.21 (d, *J* = 7.8 Hz, 1H), 6.64 (s, 1H), 6.15 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.9, 144.3, 142.7, 134.9, 131.2, 129.9, 128.9, 128.3, 127.4, 126.2, 124.8, 123.4, 122.1, 116.0, 114.7, 105.5, 100.0, 84.6. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}BrNO_2$ $[M+H]^+$: 404.0286, found: 404.0285.

3-(2-phenyl-5-(trifluoromethyl)-1H-indol-1-yl)isobenzofuran-1(3H)-one (38)



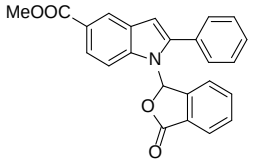
The reaction was performed following the General Procedure B with 2-(phenylethynyl)-4-(trifluoromethyl)aniline (261 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (358 mg, 91% yield) as a white solid; **mp** 163 - 164 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.08 (s, 1H), 7.89 (s, 1H), 7.75 - 7.63 (m, 4H), 7.58 - 7.46 (m, 3H), 7.42 (s, 1H), 7.35 - 7.29 (m, 1H), 7.13 (d, *J* = 7.7 Hz, 1H), 6.75 (s, 1H), 6.17 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.9, 144.4, 143.9, 137.3, 135.0, 131.2, 130.9, 129.9, 129.3, 129.0, 127.4, 126.2, 123.9 (q, *J* = 32 Hz), 123.4, 120.8, 119.3 (q, *J* = 3 Hz), 118.6 (q, *J* = 4 Hz), 111.8, 105.8, 84.6.**HRMS (ESI) m/z**: calculated for $C_{23}H_{14}F_3NO_2$ $[M+H]^+$: 394.1055, found: 394.1056.

3-(5-nitro-2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (39)



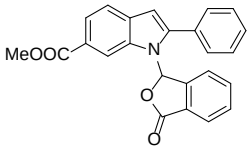
The reaction was performed following the General Procedure B with 4-nitro-2-(phenylethynyl)aniline (238 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (247 mg, 67% yield) as a yellow solid; **mp** 209 - 210 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.54 (d, *J* = 2.1 Hz, 1H), 8.09 (s, 1H), 7.81 (d, *J* = 8.6 Hz, 1H), 7.77 - 7.60 (m, 4H), 7.58 - 7.49 (m, 3H), 7.42 (s, 1H), 7.32 (d, *J* = 6.0 Hz, 1H), 6.83 (s, 1H), 6.19 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 144.1, 142.9, 135.2, 131.5, 130.4, 129.9, 129.6, 129.2, 127.2, 126.4, 123.3, 118.0, 117.6, 111.5, 106.6, 84.3. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}N_2O_4$ $[M+Na]^+$: 393.0852, found: 393.0854.

methyl 1-(3-oxo-1,3-dihydroisobenzofuran-1-yl)-2-phenyl-1H-indole-5-carboxylate (40)



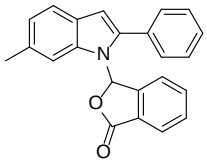
The reaction was performed following the General Procedure B with methyl 4-amino-3-(phenylethynyl)benzoate (251 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (344 mg, 90% yield) as a white solid; **mp** 172 - 173 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.34 (d, *J* = 0.9 Hz, 1H), 8.07 (s, 1H), 7.76 - 7.63 (m, 4H), 7.59 (d, *J* = 6.5 Hz, 1H), 7.56 - 7.44 (m, 3H), 7.41 (s, 1H), 7.31 (d, *J* = 6.1 Hz, 1H), 6.75 (s, 1H), 6.11 (s, 1H), 3.89 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 167.5, 144.5, 134.9, 131.2, 131.0, 129.9, 129.1, 129.0, 127.4, 126.2, 123.9, 123.6, 123.6, 123.4, 111.2, 106.2, 84.6, 51.9. **HRMS (ESI) m/z**: calculated for $C_{24}H_{17}NO_4$ $[M+Na]^+$: 406.1056, found: 406.1056.

methyl 1-(3-oxo-1,3-dihydroisobenzofuran-1-yl)-2-phenyl-1H-indole-6-carboxylate (41)



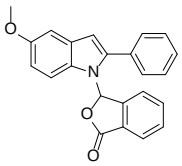
The reaction was performed following the General Procedure B with methyl 3-amino-4-(phenylethynyl)benzoate (251 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (333 mg, 87% yield) as a white solid; **mp** 154 - 155 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.76 - 7.65 (m, 4H), 7.62 (d, *J* = 8.3 Hz, 1H), 7.56 - 7.41 (m, 5H), 7.33 (d, *J* = 6.0 Hz, 1H), 6.71 (s, 1H), 3.78 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.0, 167.3, 144.4, 134.9, 131.1, 129.9, 129.2, 128.9, 127.5, 126.2, 124.2, 123.4, 122.6, 120.6, 84.6, 51.9. **HRMS (ESI) m/z**: calculated for $C_{24}H_{17}NO_4$ $[M+Na]^+$: 406.1056, found: 406.1056.

3-(6-methyl-2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (42)



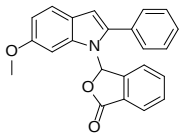
The reaction was performed following the General Procedure B with 5-methyl-2-(phenylethynyl)aniline (207 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (254 mg, 75% yield) as a white solid; **mp** 119 - 120 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.66 (s, 4H), 7.56 - 7.36 (m, 5H), 7.32 (d, *J* = 3.1 Hz, 1H), 6.93 (d, *J* = 7.7 Hz, 1H), 6.62 (s, 1H), 5.80 (s, 1H), 2.15 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.3, 145.0, 141.4, 134.7, 132.5, 131.8, 130.8, 129.9, 128.8, 128.6, 127.6, 127.3, 125.9, 123.5, 123.1, 120.6, 111.8, 105.5, 85.1, 21.9. **HRMS (ESI) m/z**: calculated for $C_{23}H_{17}NO_2$ $[M+H]^+$: 340.1338, found: 340.1337.

3-(5-methoxy-2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (43)



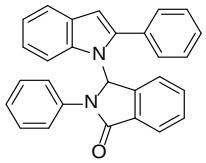
The reaction was performed following the General Procedure B with 4-methoxy-2-(phenylethynyl)aniline (223 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (234 mg, 66% yield) as a yellow solid; **mp** 212 - 213 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05 (s, 1H), 7.75 - 7.61 (m, 4H), 7.58 - 7.42 (m, 3H), 7.38 (s, 1H), 7.36 - 7.30 (m, 1H), 7.05 (d, *J* = 2.4 Hz, 1H), 6.61 (s, 1H), 6.52 (d, *J* = 6.7 Hz, 1H), 5.94 (s, 1H), 3.79 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 155.1, 144.9, 142.9, 134.8, 131.7, 131.0, 130.3, 129.8, 128.9, 128.8, 127.6, 126.0, 123.5, 112.3, 105.4, 103.1, 85.1, 55.7. **HRMS (ESI) m/z**: calculated for $C_{23}H_{17}NO_3$ $[M+H]^+$: 356.1287, found: 356.1287.

3-(6-methoxy-2-phenyl-1H-indol-1-yl)isobenzofuran-1(3H)-one (44)



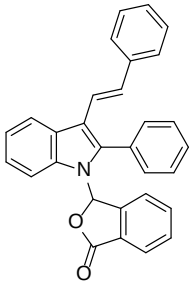
The reaction was performed following the General Procedure B with 5-methoxy-2-(phenylethynyl)aniline (223 mg, 1 mmol), 2-formylbenzoic acid (225 mg, 1.5 mmol), and $Pd(OAc)_2$ (2 mg, 1 mol%). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (245 mg, 69% yield) as a white solid; **mp** 161 - 162 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.68 (s, 4H), 7.57 - 7.39 (m, 5H), 7.39 - 7.32 (m, 1H), 6.74 (d, *J* = 7.7 Hz, 1H), 6.60 (s, 1H), 5.51 (s, 1H), 3.43 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 156.3, 144.9, 141.0, 134.8, 131.8, 130.9, 129.8, 128.9, 128.5, 127.6, 125.9, 123.7, 121.5, 110.9, 105.4, 95.9, 85.1, 55.2. **HRMS (ESI) m/z**: calculated for $C_{23}H_{17}NO_3$ $[M+H]^+$: 356.1287, found: 356.1286.

2-phenyl-3-(2-phenyl-*1H*-indol-1-yl)isoindolin-1-one (45)



The reaction was performed following the General Procedure C with 3-(2-phenyl-1*H*-indol-1-*yl*)isobenzofuran-1(3H)-one (325 mg, 1 mmol), and aniline (93 μL, 1 mmol). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (244 mg, 61% yield) as a white solid; **mp** 248 - 249 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.14 (s, 1H), 8.10 - 8.03 (m, 1H), 7.59 - 7.48 (m, 7H), 7.46 - 7.41 (m, 1H), 7.19 - 6.98 (m, 7H), 6.88 - 6.77 (m, 2H), 6.46 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 139.5, 134.8, 130.6, 130.5, 130.2, 129.3, 129.2, 129.1, 128.9, 127.9, 125.8, 125.0, 124.5, 123.4, 122.8, 120.3, 119.4, 118.1, 115.8, 110.6, 29.7. **HRMS (ESI) m/z**: calculated for $C_{28}H_{20}N_2O$ $[M+H]^+$: 401.1654, found: 401.1628.

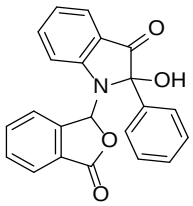
(*E*)-3-(2-(2-styrylphenyl)-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (46)



The reaction was performed following the General Procedure D with 3-(2-phenyl-1*H*-indol-1-*yl*)isobenzofuran-1(3H)-one (325 mg, 1 mmol), 2-phenylacetaldehyde (129 μL, 1 mmol), $FeCl_3·6H_2O$ (7 mg, 2.5 mol%), and EtOH (128 μL, 2.2 mmol). The crude product was purified by silica gel chromatography (PE/EA = 5/1) to give the desired product (183 mg, 43% yield) as a white solid; **mp** 197 - 198 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.0 Hz, 1H), 8.02 (s, 1H), 7.67 (s, 3H), 7.52 (s, 4H), 7.43 - 7.34 (m, 3H), 7.33 - 7.27 (m, 3H), 7.21 (t, *J* = 6.4 Hz, 2H), 7.17 (s, 1H), 7.04 (d, *J* = 17.2 Hz, 2H), 6.15 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.1, 144.8, 138.4, 134.8, 131.5,

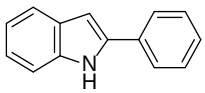
130.9, 130.3, 128.9, 128.6, 128.4, 127.5, 127.0, 126.0, 123.4, 122.0, 121.4, 121.0, 84.9. **HRMS (ESI) m/z**: calculated for $C_{30}H_{21}NO_2$ $[M+H]^+$: 428.1651, found: 428.1624.

2-hydroxy-1-(3-oxo-1,3-dihydroisobenzofuran-1-yl)-2-phenylindolin-3-one (47)



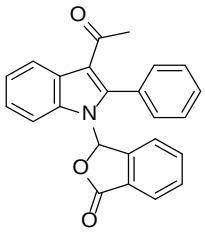
The reaction was performed following the General Procedure E with 3-(2-phenyl-1*H*-indol-1-*yl*)isobenzofuran-1(3H)-one (325 mg, 1 mmol), $NaIO_4$ (214 mg, 1 mmol), and $RuCl_3 \cdot 3H_2O$ (13 mg, 5 mol%). The crude product was purified by silica gel chromatography (PE/EA = 2/1) to give the desired product (250 mg, 70% yield) as a yellow solid; **mp** 233- 234 ℃; **^{1}H NMR** (400 MHz, DMSO-*d6*) δ 7.99 - 7.89 (m, 1H), 7.86 - 7.65 (m, 3H), 7.64 - 7.52 (m, 2H), 7.48 (s, 2H), 7.45 - 7.33 (m, 4H), 7.03 - 6.74 (m, 2H), 6.05 - 5.84 (m, 1H). **^{13}C NMR** (100 MHz, DMSO-*d6*) δ 198.2, 198.1, 168.4, 168.3, 155.8, 155.3, 144.8, 144.7, 138.1, 137.9, 137.5, 136.9, 134.7, 134.5, 130.8, 130.6, 128.7, 128.5, 128.4, 127.5, 127.2, 126.4, 126.1, 125.4, 125.3, 125.1, 123.8, 123.7, 120.6, 120.3, 119.2, 118.6, 110.6, 110.3, 90.4, 90.1, 85.4, 85.2. **HRMS (ESI) m/z**: calculated for $C_{22}H_{15}NO_4$ $[M+H]^+$: 358.1079, found: 358.1053.

2-phenyl-*1H*-indole (48)



The reaction was performed following the General Procedure F with 3-(2-phenyl-1*H*-indol-1-*yl*)isobenzofuran-1(3H)-one (325 mg, 1 mmol), and *t*-BuOK (112 mg, 1 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20/1) to give the desired product (149 mg, 77% yield) as a white solid; **mp** 192 - 193 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.32 (s, 1H), 7.68 - 7.61 (m, 3H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.39 (d, *J* = 8.1 Hz, 1H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.19 (t, *J* = 7.4 Hz, 1H), 7.12 (t, *J* = 7.5 Hz, 1H), 6.83 (d, *J* = 1.5 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 137.9, 136.8, 132.4, 129.3, 129.0, 127.7, 125.2, 122.3, 120.7, 120.3, 110.9, 100.0. **HRMS (ESI) m/z**: calculated for $C_{14}H_{11}N$ $[M+H]^+$: 194.0970, found: 194.0944.

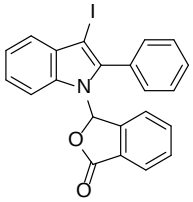
3-(3-acetyl-2-phenyl-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (49)



The reaction was performed following the General Procedure G with 3-(2-phenyl-1*H*-indol-1-*yl*)isobenzofuran-1(3H)-one (325 mg, 1 mmol), Et_2AlCl (1.53 mL, 1.5 mmol, 0.98 mol/L in hexane), and acetyl chloride (85 μL, 1.2 mmol). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (260 mg, 71% yield) as a white solid; **mp** 214 - 215 ℃; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.45

(d, *J* = 8.0 Hz, 1H), 8.02 (s, 1H), 7.77 - 7.46 (m, 7H), 7.35 (d, *J* = 6.8 Hz, 1H), 7.25 (s, 1H), 6.99 (s, 2H), 6.05 (s, 1H), 1.97 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 195.2, 167.7, 144.3, 135.0, 131.1, 130.7, 129.1, 127.2, 126.1, 124.2, 123.5, 123.2, 84.2, 30.4. **HRMS (ESI) m/z**: calculated for $C_{24}H_{17}NO_3$ $[M+H]^+$: 368.1287, found: 368.1259.

3-(3-iodo-2-phenyl-*1H*-indol-1-yl)isobenzofuran-1(*3H*)-one (50)

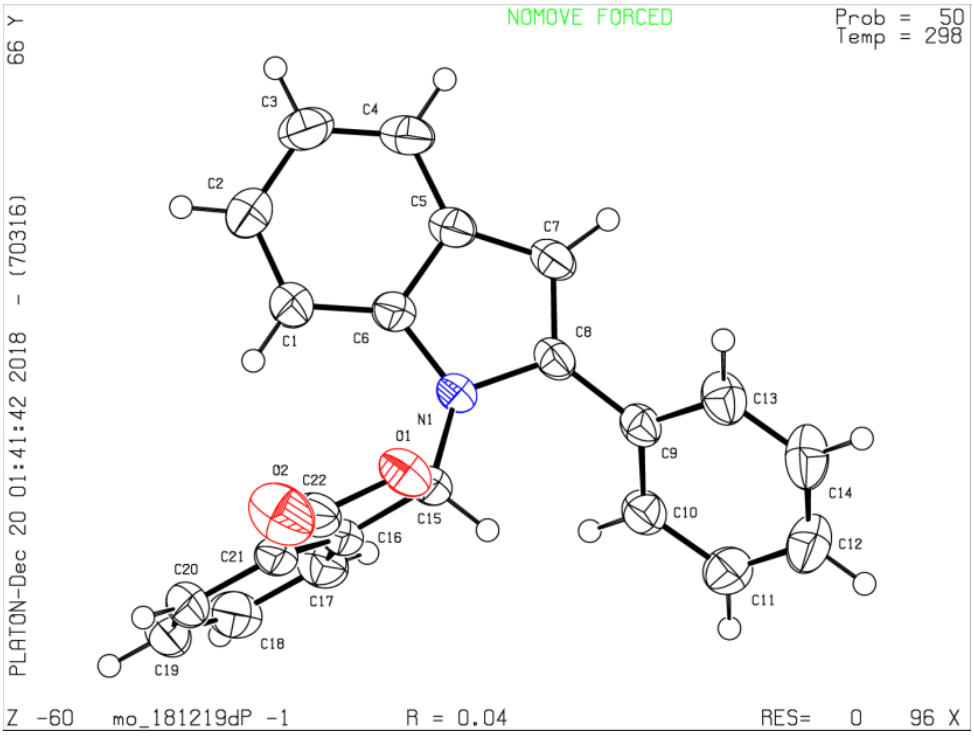


The reaction was performed following the General Procedure H with 3-(2-phenyl-1*H*-indol-1-*yl*)isobenzofuran-1(3H)-one (325 mg, 1 mmol), NaI (150 mg, 1 mmol), and $PhI(OAc)_2$ (322 mg, 1 mmol). The crude product was purified by silica gel chromatography (PE/EA = 10/1) to give the desired product (427 mg, 95% yield) as a yellow solid; **mp** 208 - 209 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (s, 1H), 7.72 - 7.63 (m, 2H), 7.57 (s, 2H), 7.50 (d, *J* = 7.9 Hz, 4H), 7.35 (d, *J* = 5.7 Hz, 1H), 7.27 (s, 1H), 7.19 (t, *J* = 7.5 Hz, 1H), 6.99 (s, 1H), 6.17 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.9, 144.6, 142.3, 134.9, 131.7, 131.1, 130.7, 129.5, 128.8, 127.3, 126.0, 124.0, 123.4, 122.2, 111.4, 85.3. **HRMS (ESI) m/z**: calculated for $C_{22}H_{14}INO_2$ $[M+H]^+$: 452.0147, found: 452.0124.

VI. X-ray Crystallographic Data

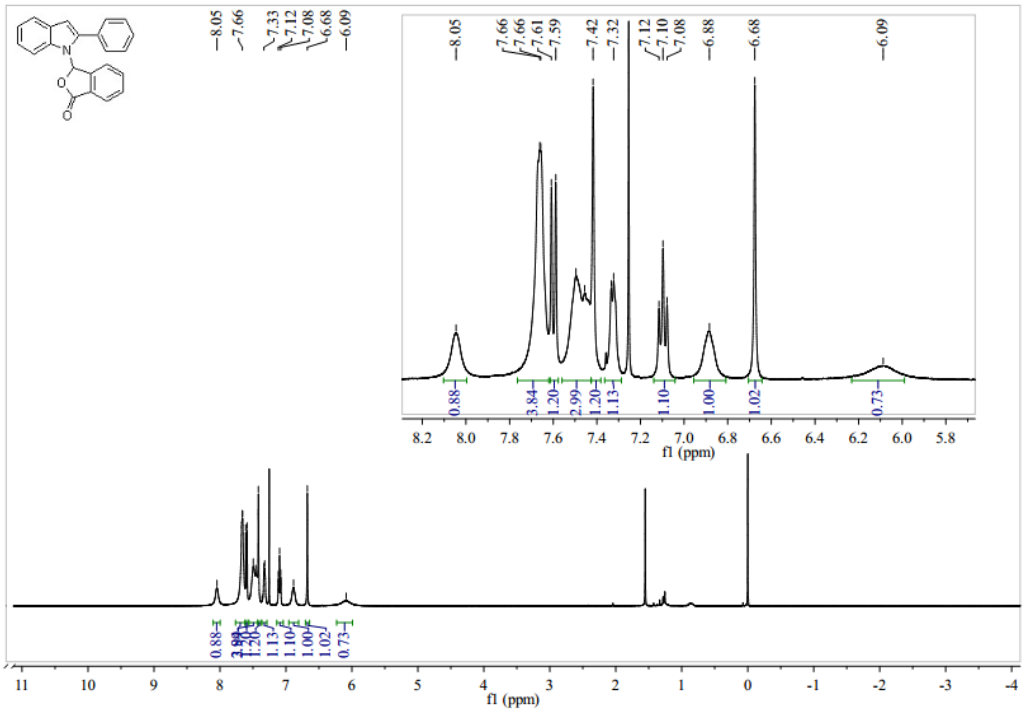
X-ray Single Crystal Structure Analysis of **3**

X-ray crystallographic data of **3**: CCDC (1886364), T = 298K, $C_{22}H_{15}NO_2$, Mr = 325.35, monoclinic, space group: P-1, *a* = 8.8838(15), *b* = 9.5186(15), *c* = 11.063(2), *α* = 114.342(7), *β* = 93.693(7), *γ* = 95.255(5), V = 843.4(3), Z = 2.

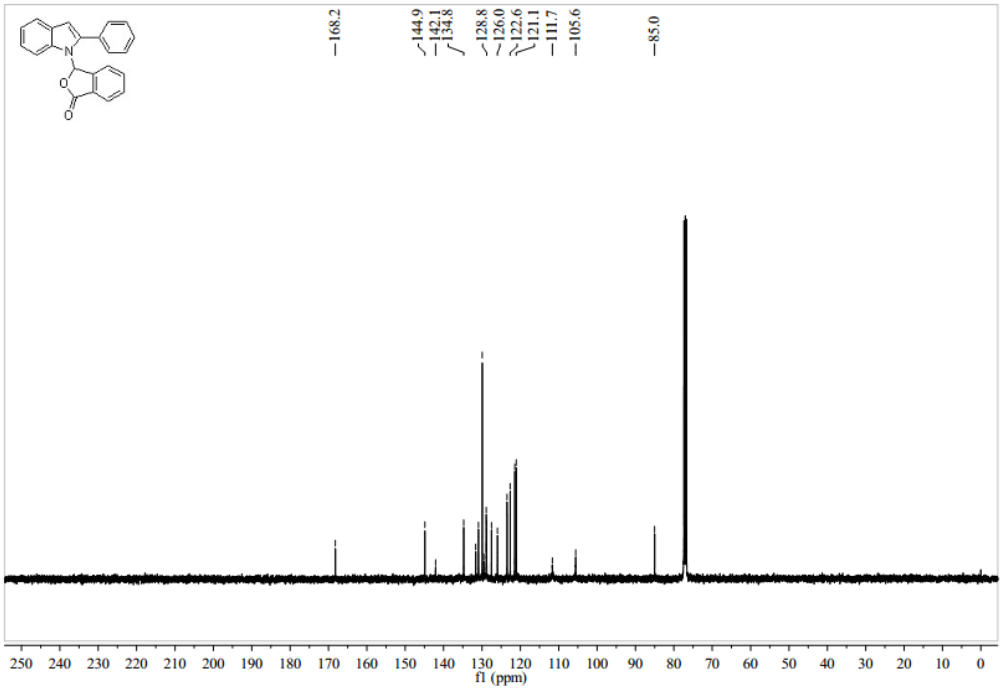


VII. Representative NMR Spectra

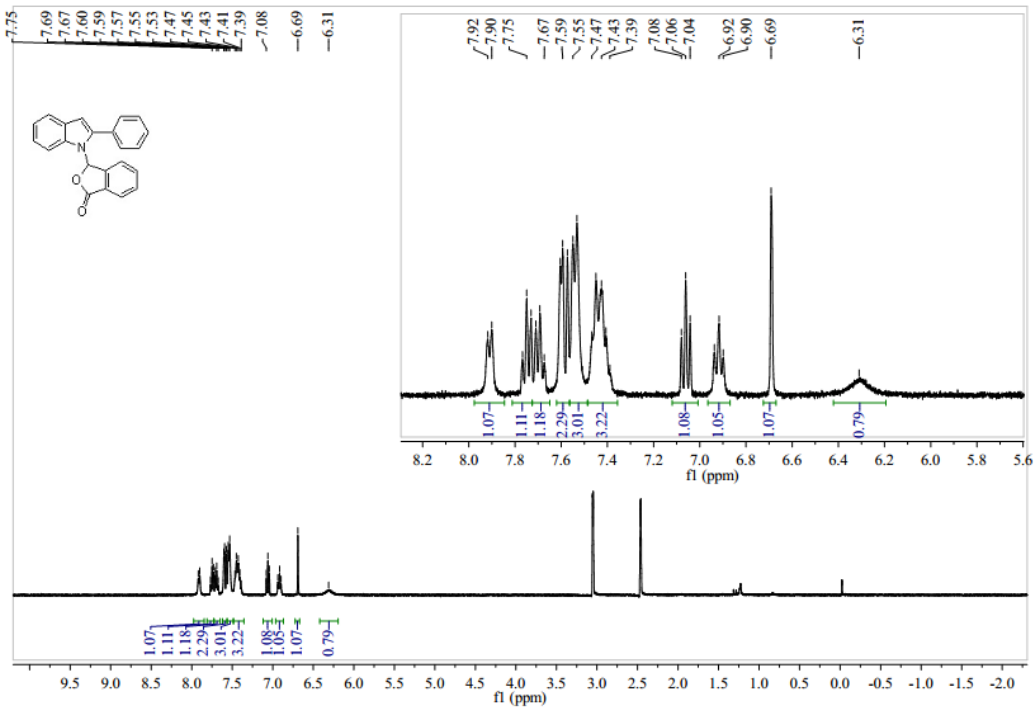
^{1}H NMR spectrum of compound 3



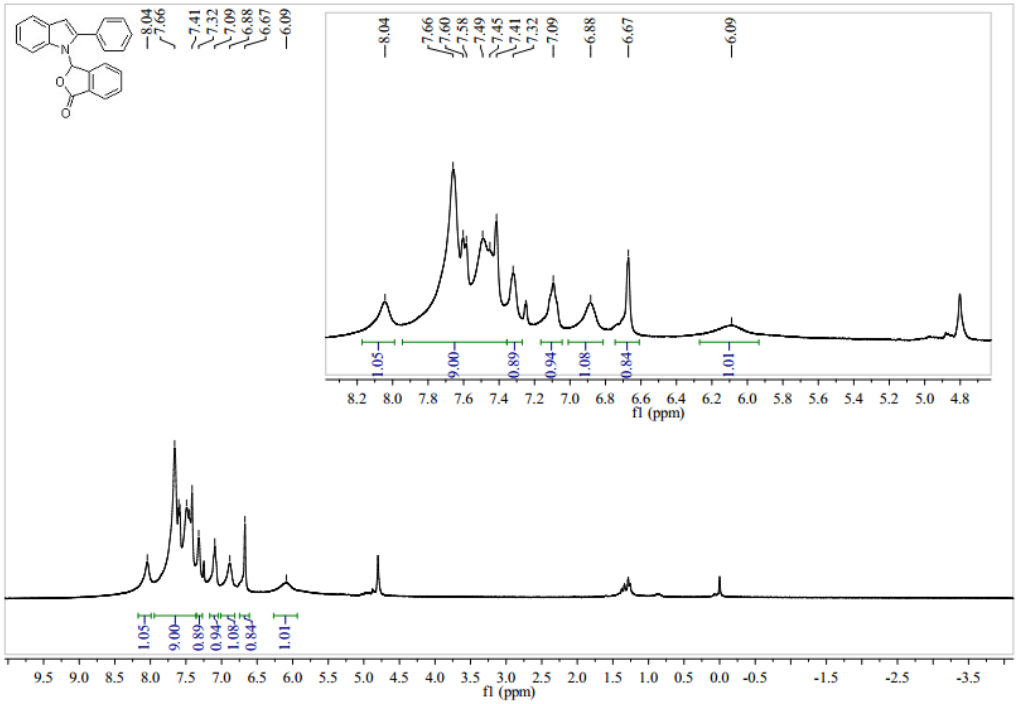
^{13}C NMR spectrum of compound 3



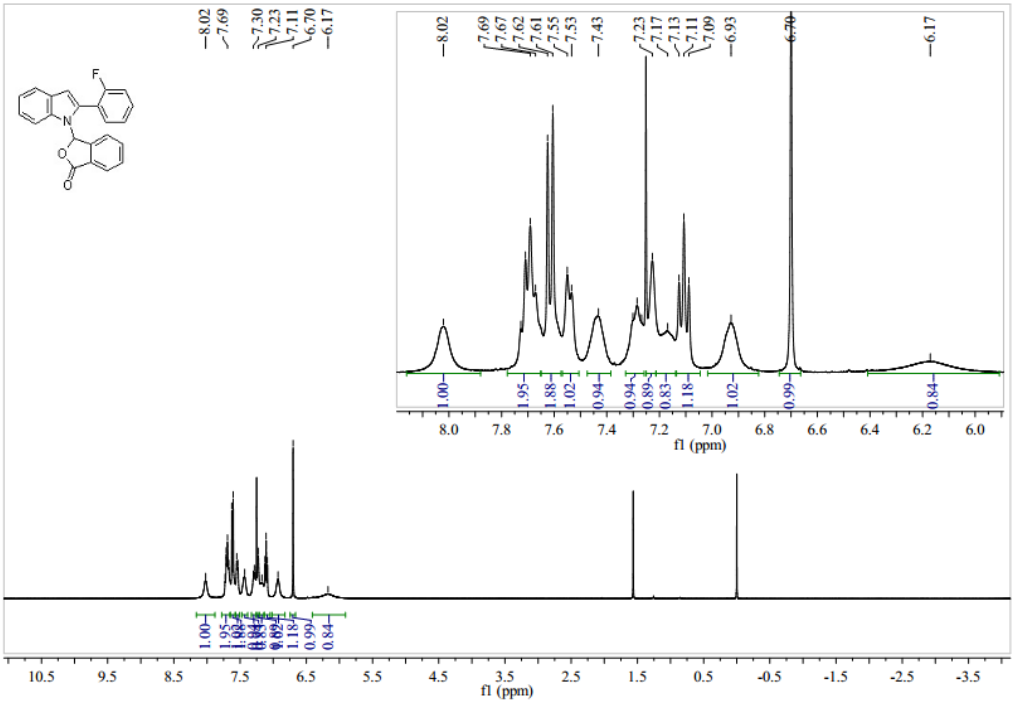
^{1}H NMR spectrum of compound 3 (80 °C in DMSO-*d*6)



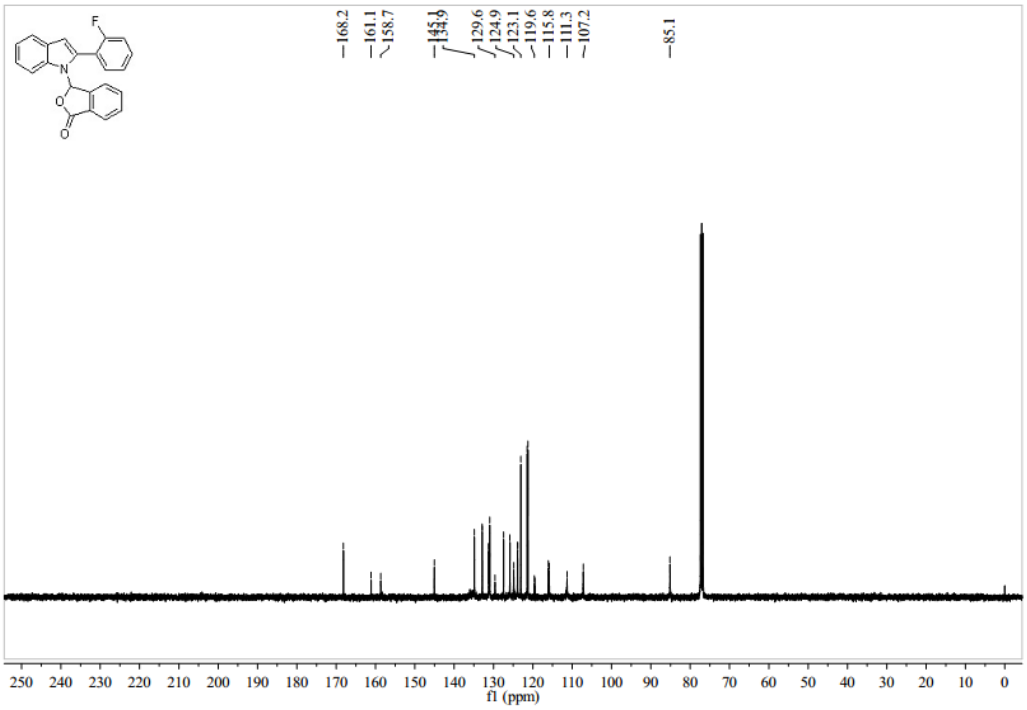
^{1}H NMR spectrum of compound 3 (additional treatment with D_2O in $CDCl_3$)



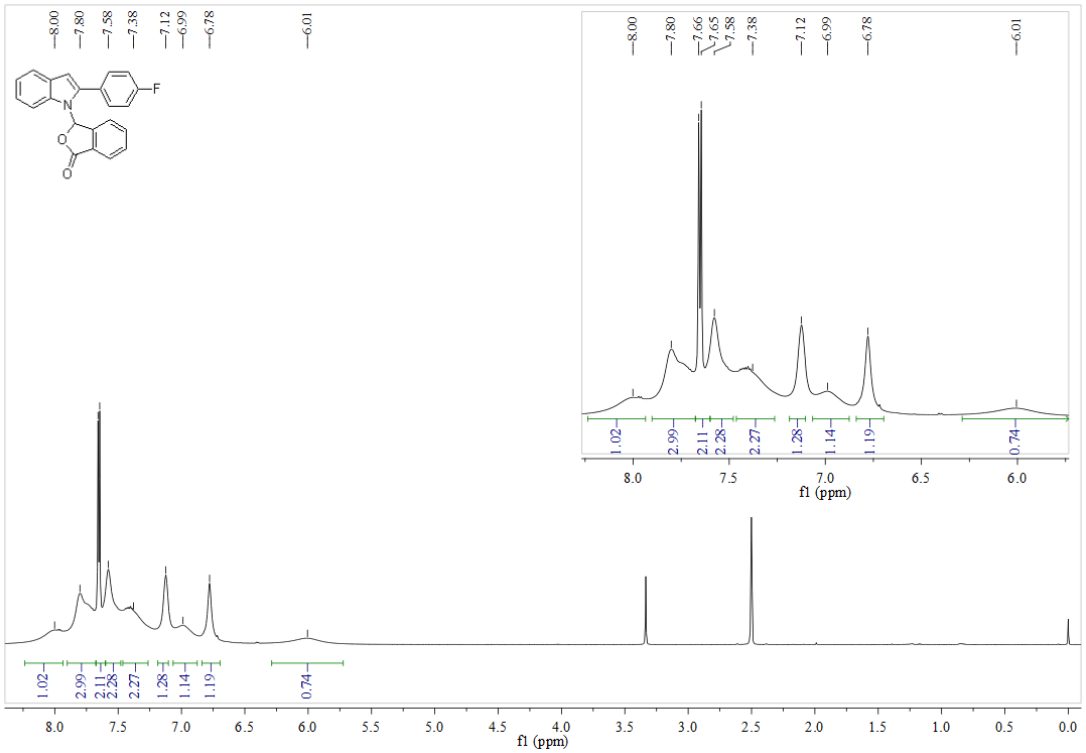
^{1}H NMR spectrum of compound 4



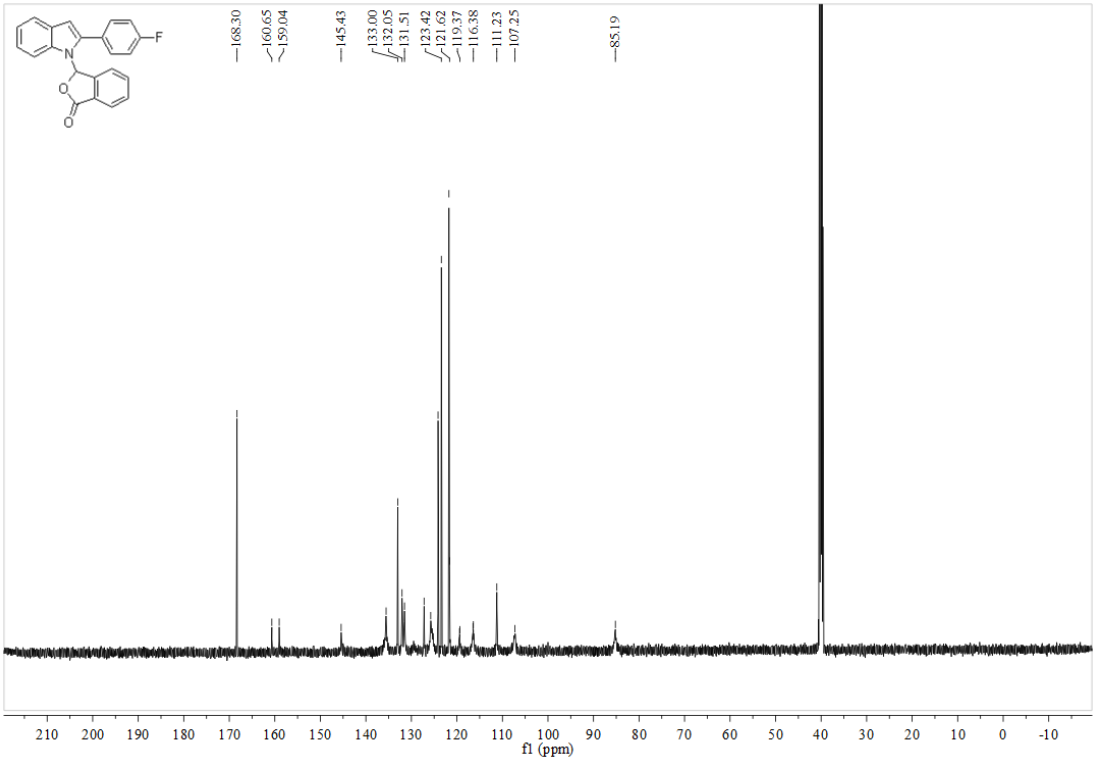
^{13}C NMR spectrum of compound 4



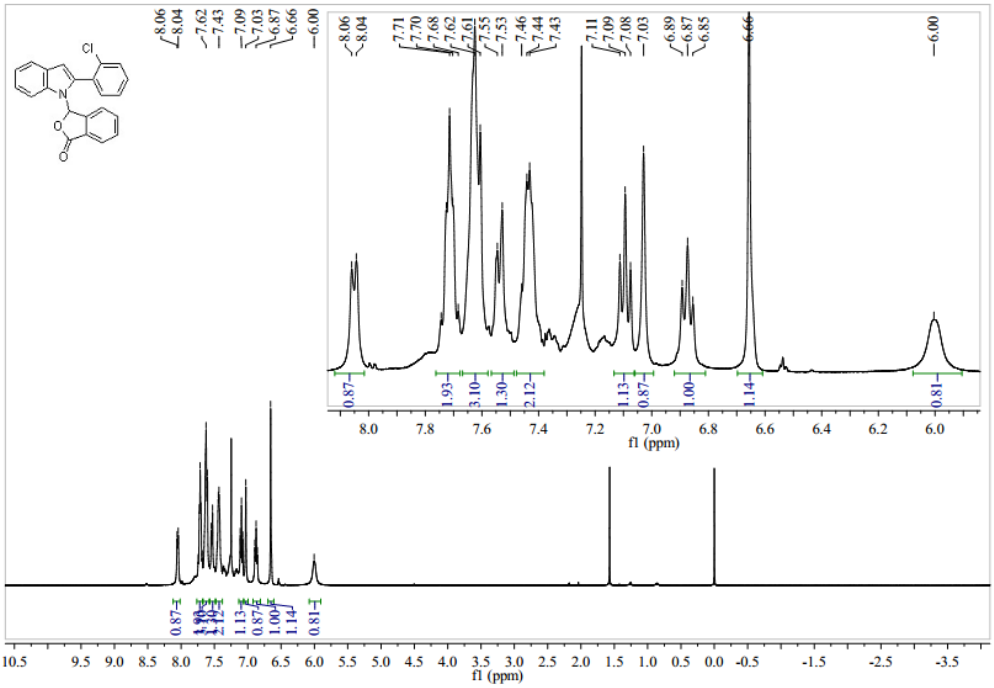
^{1}H NMR spectrum of compound 5



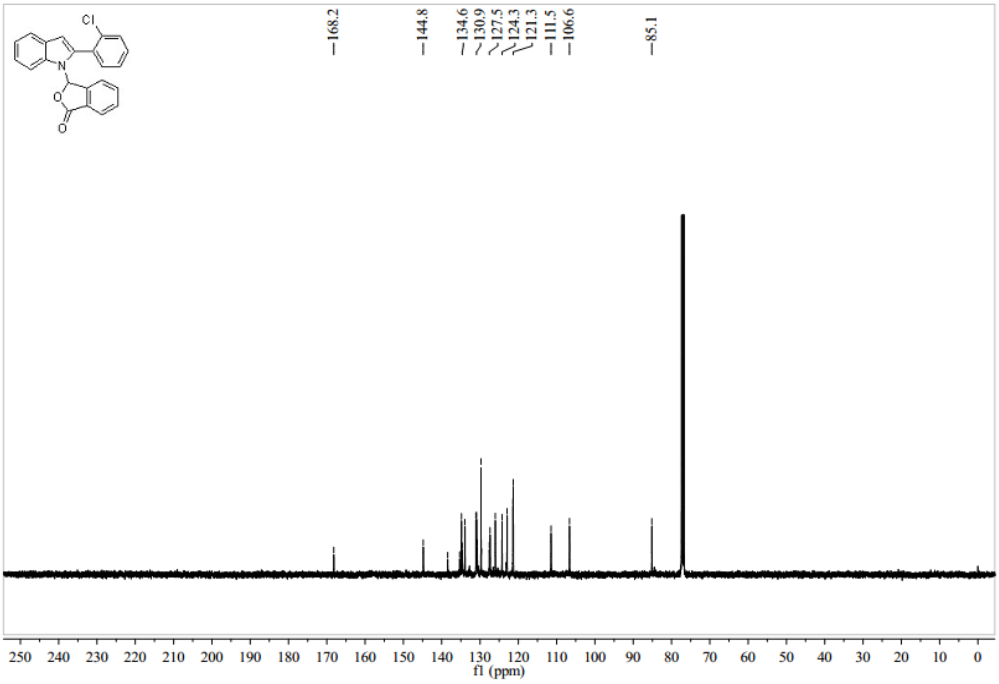
^{13}C NMR spectrum of compound 5



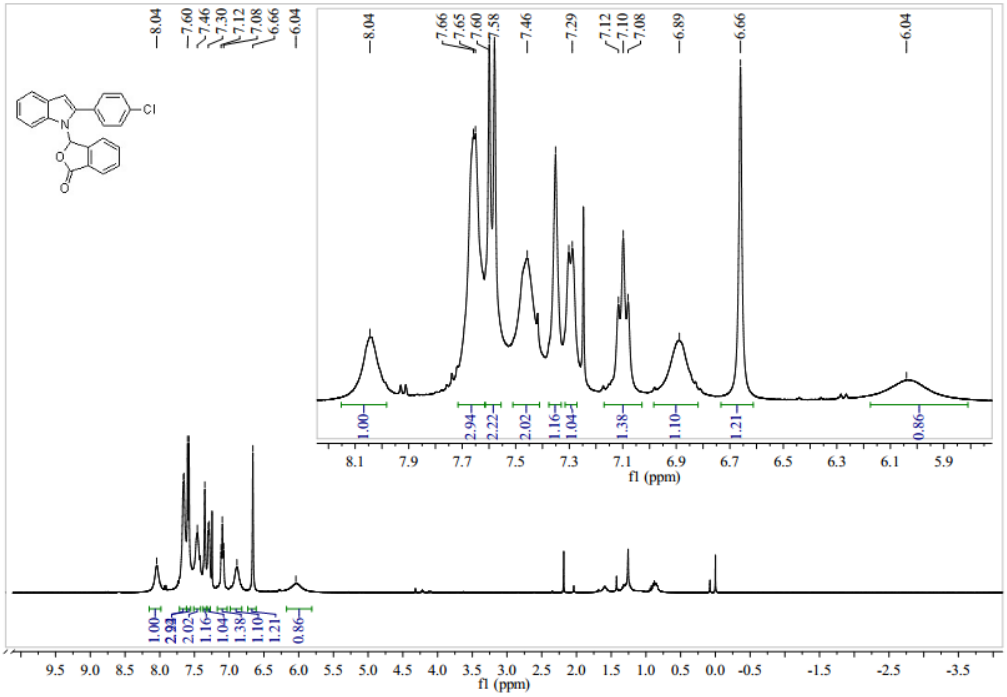
^{1}H NMR spectrum of compound 6



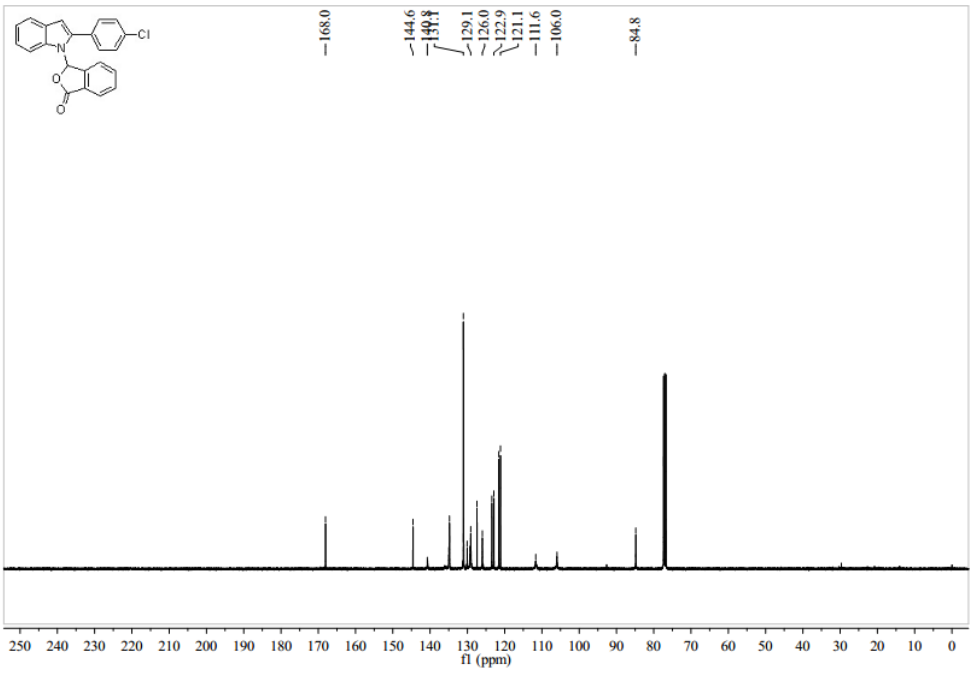
^{13}C NMR spectrum of compound 6



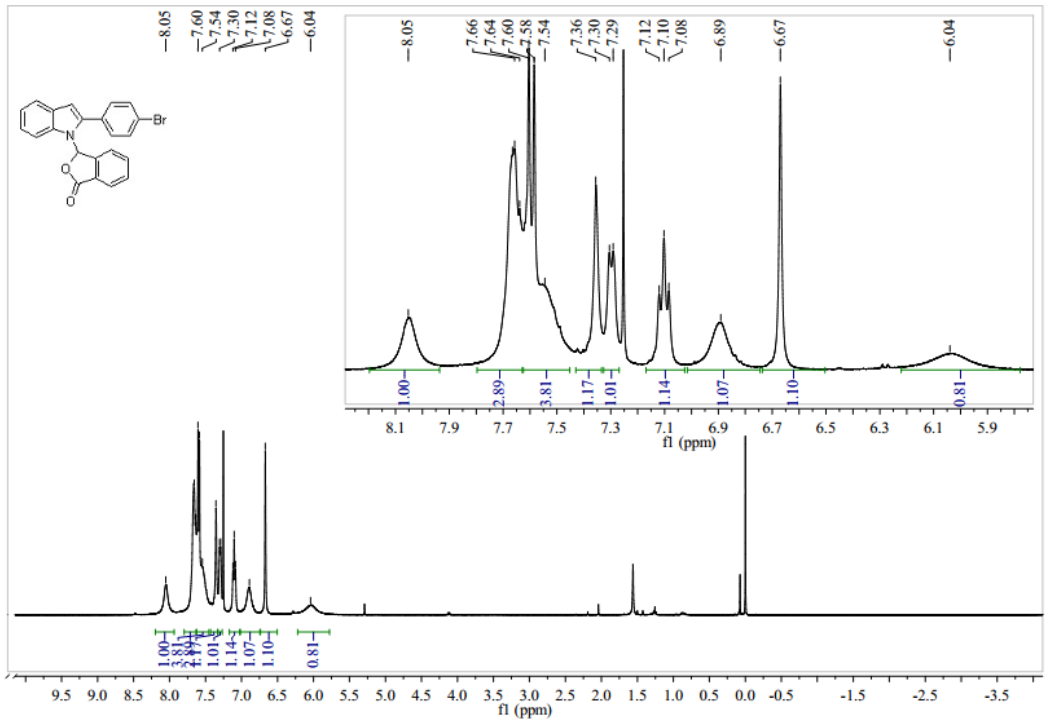
^{1}H NMR spectrum of compound 7



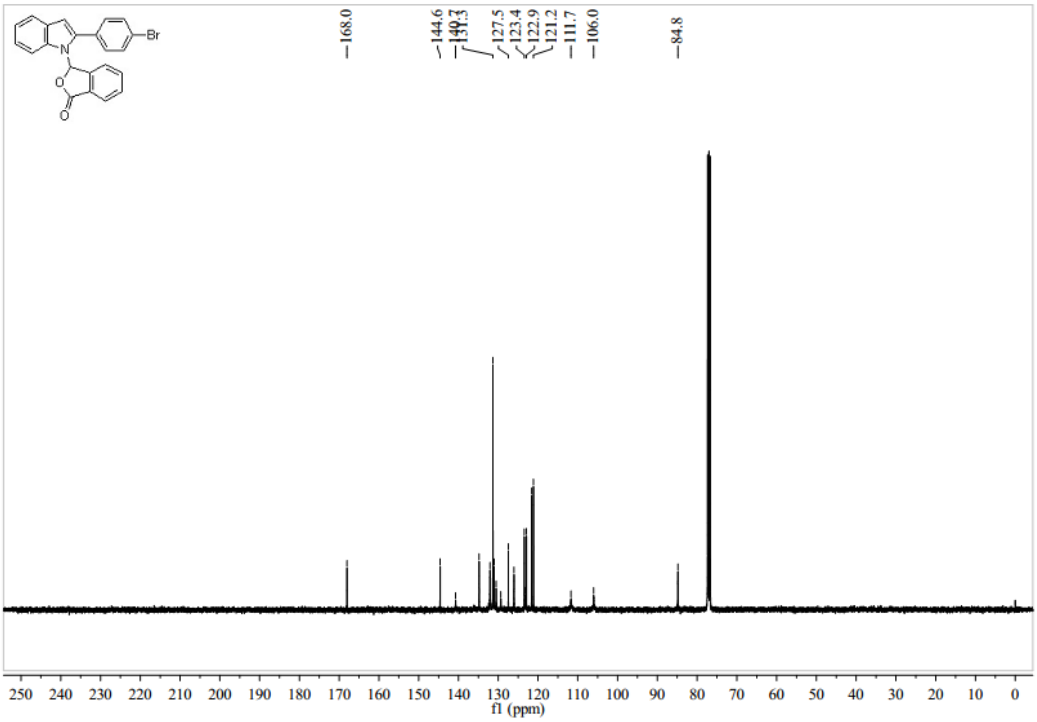
^{13}C NMR spectrum of compound 7



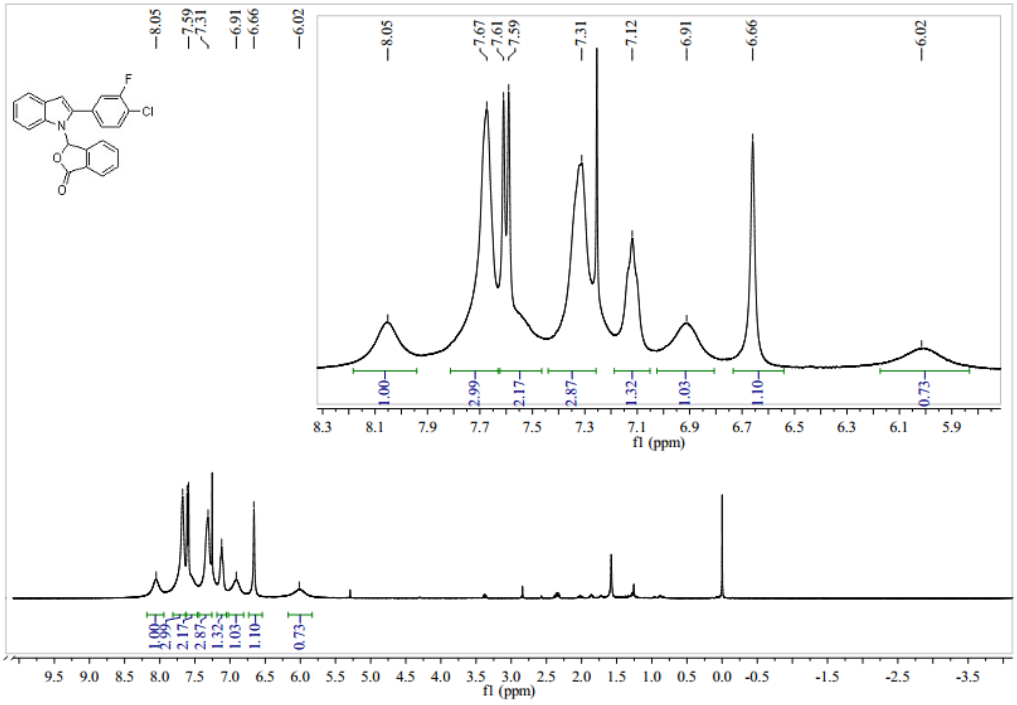
^{1}H NMR spectrum of compound 8



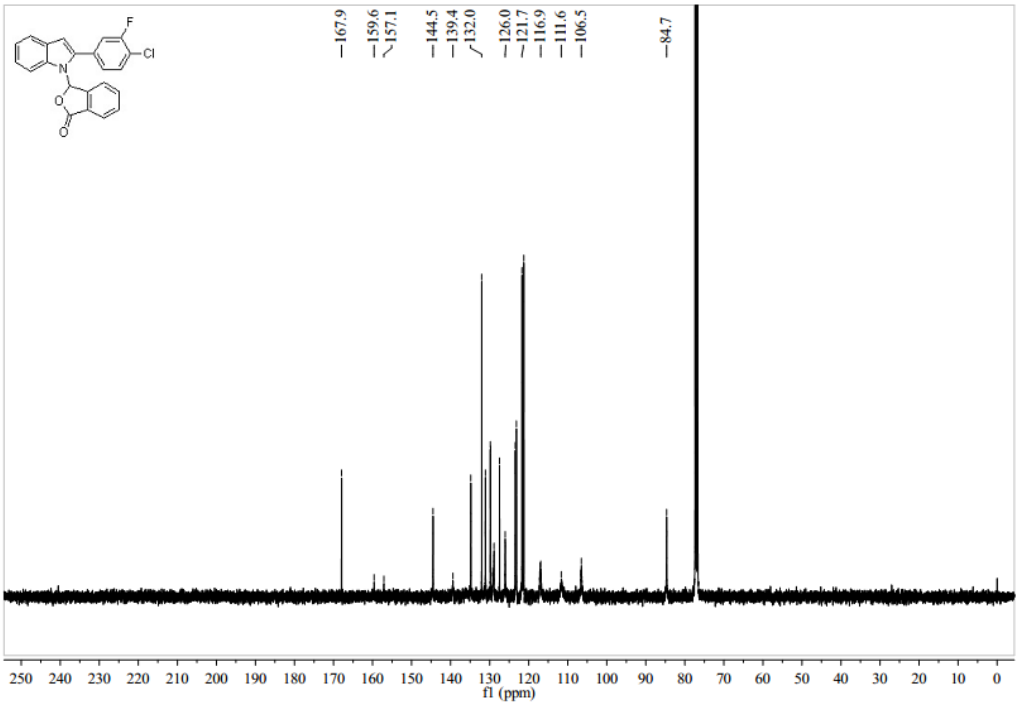
^{13}C NMR spectrum of compound 8



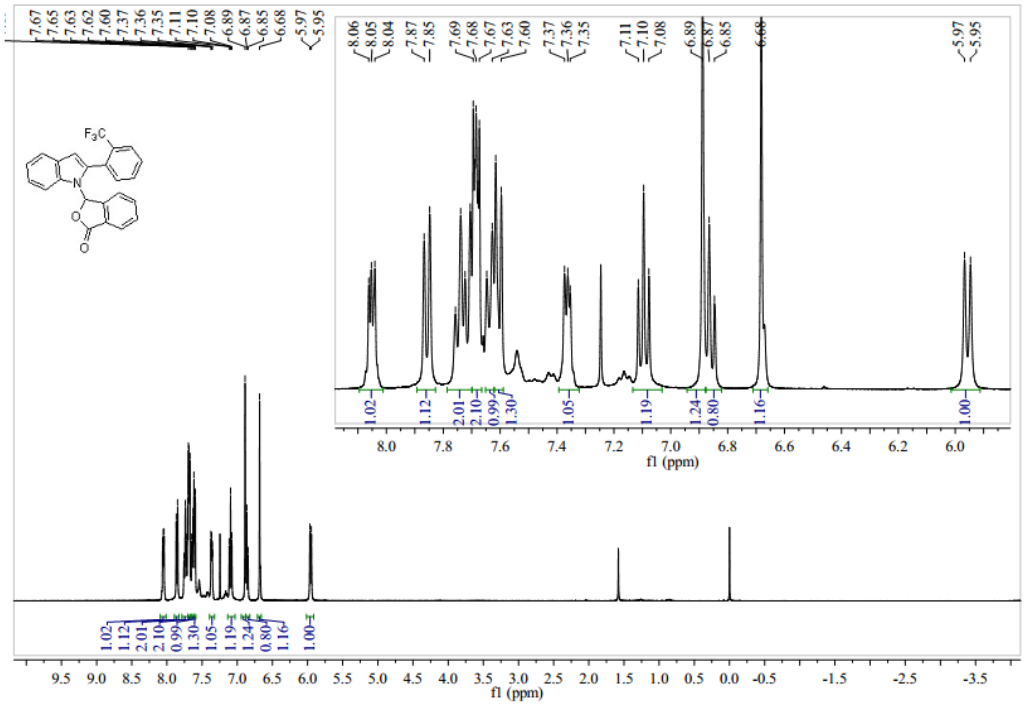
^{1}H NMR spectrum of compound 9



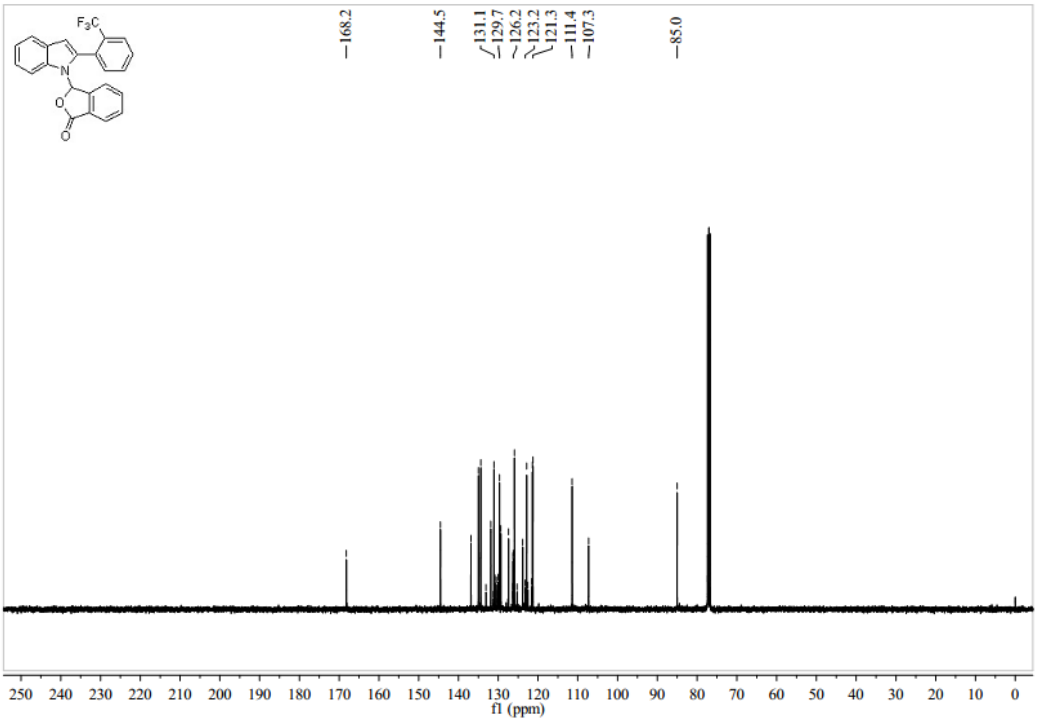
^{13}C NMR spectrum of compound 9



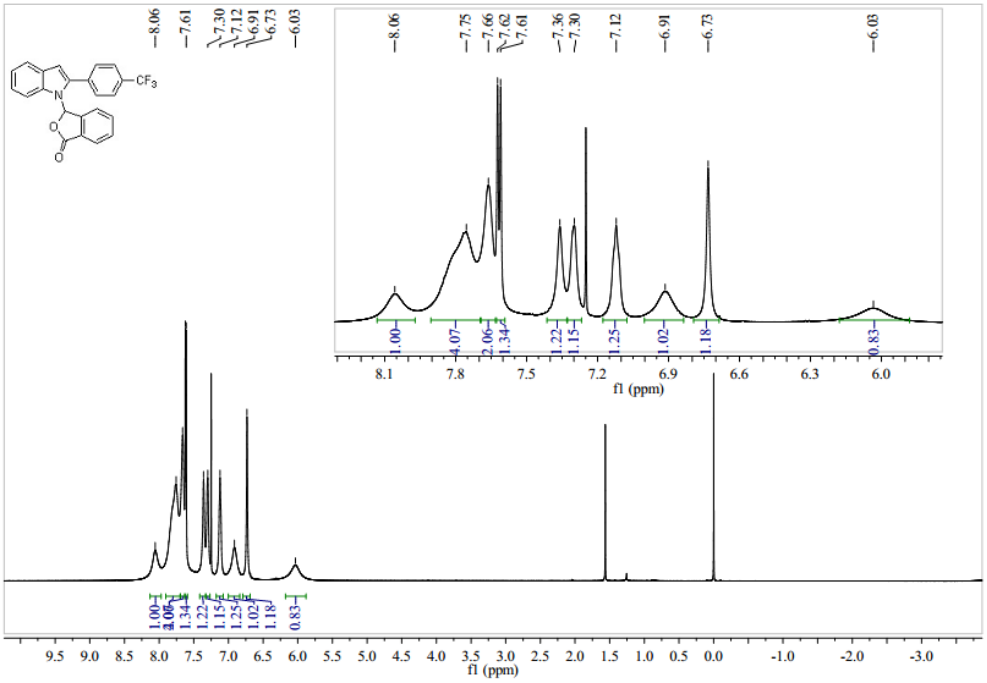
^{1}H NMR spectrum of compound 10



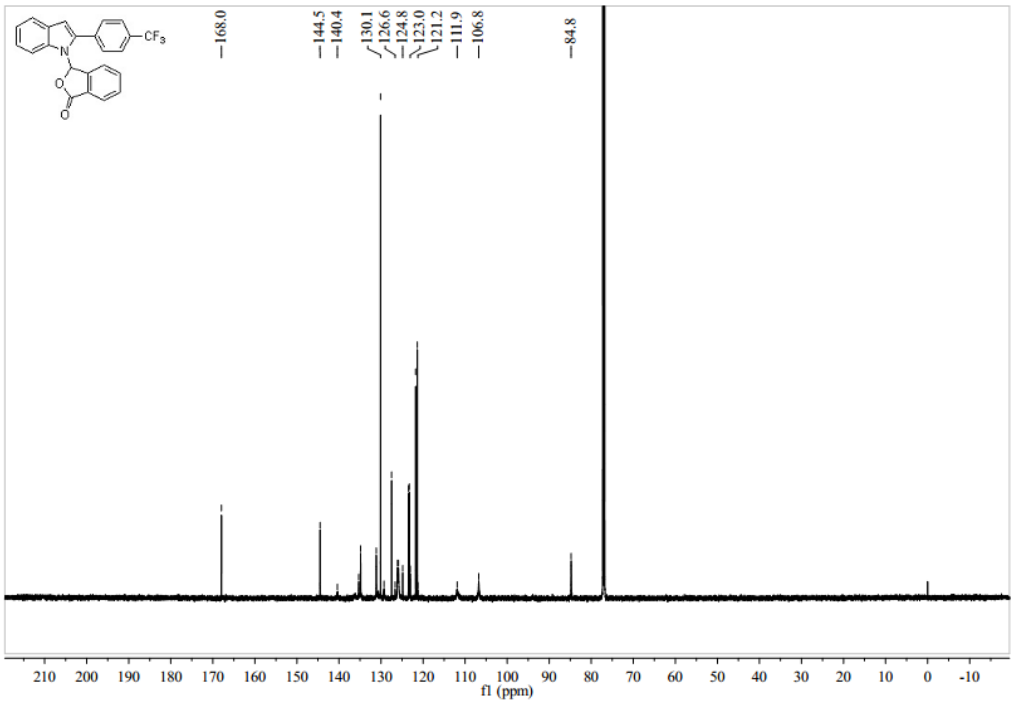
^{13}C NMR spectrum of compound 10



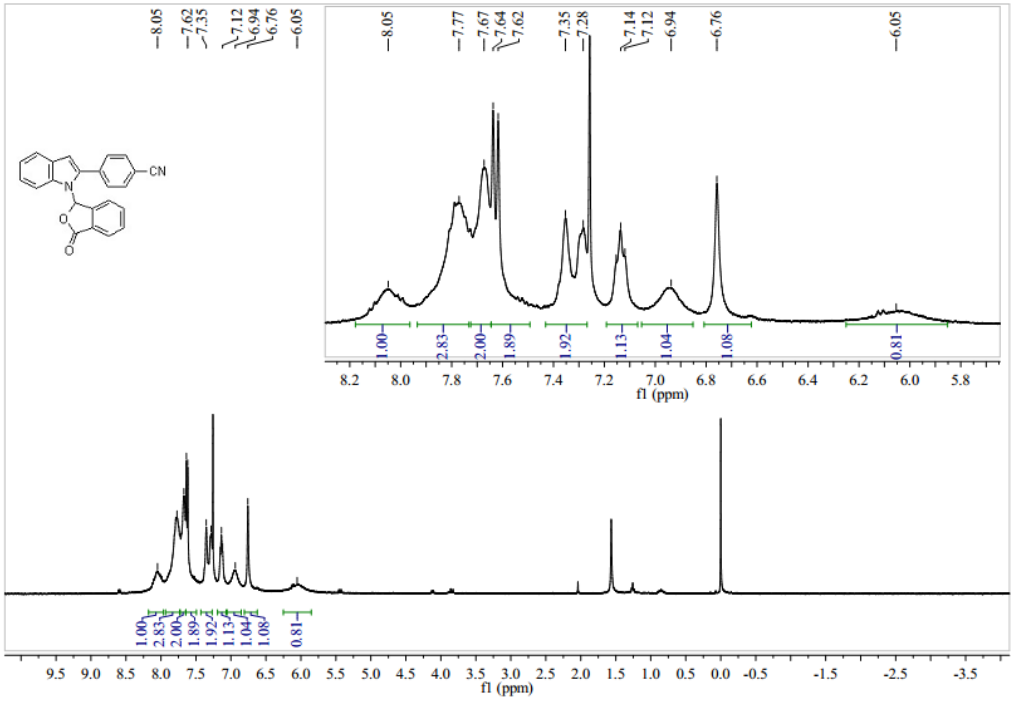
^{1}H NMR spectrum of compound 11



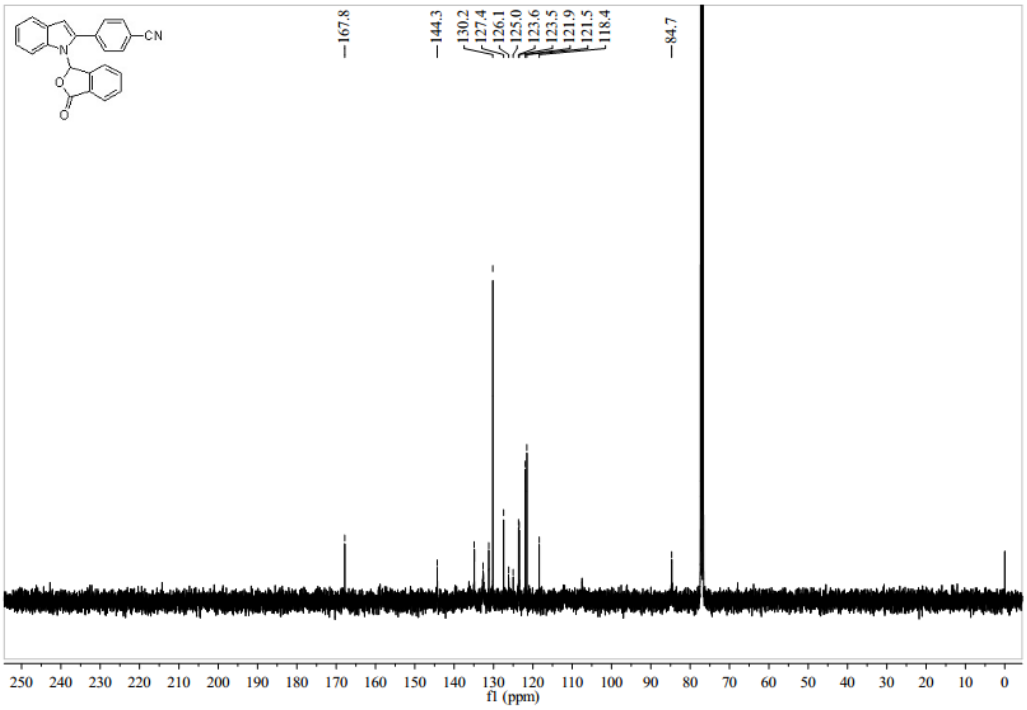
^{13}C NMR spectrum of compound 11



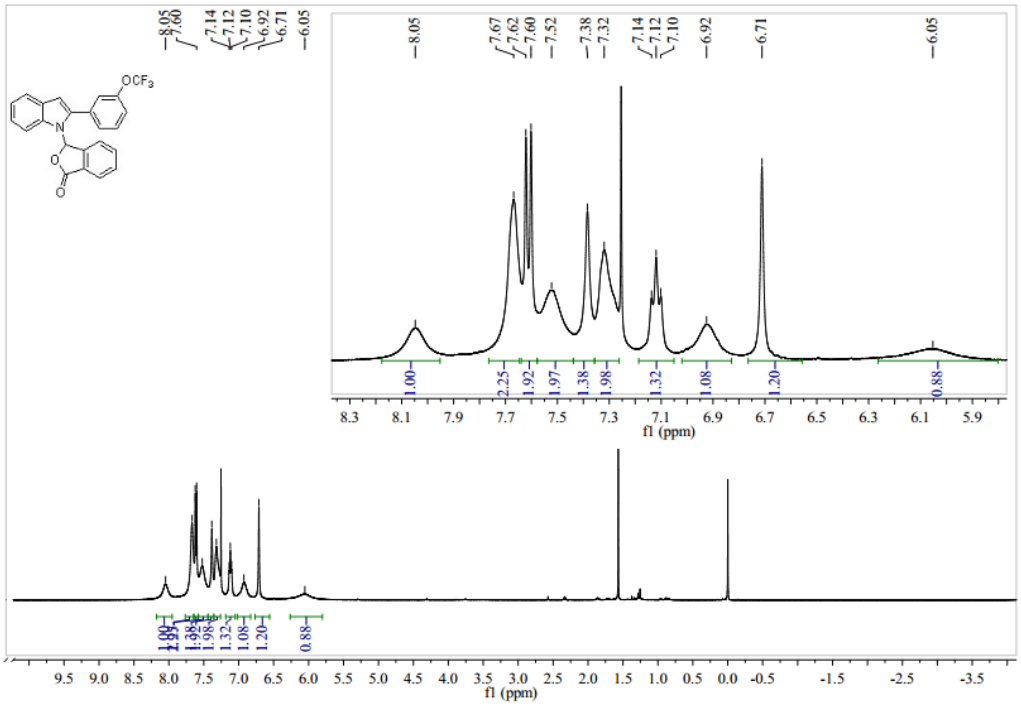
^{1}H NMR spectrum of compound 12



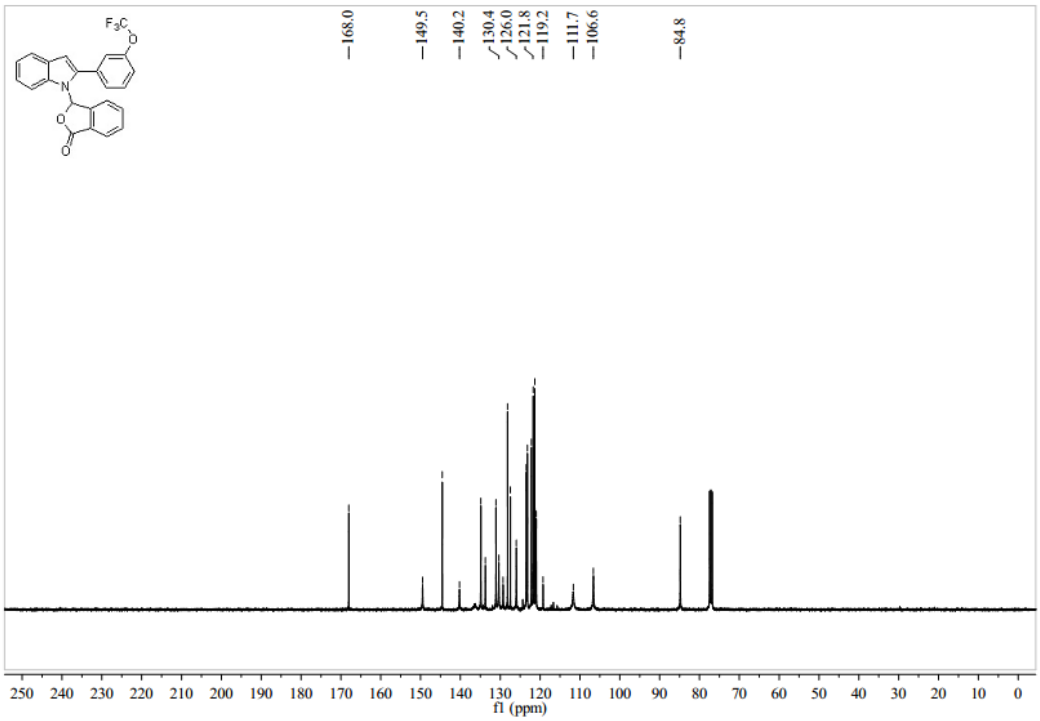
^{13}C NMR spectrum of compound 12



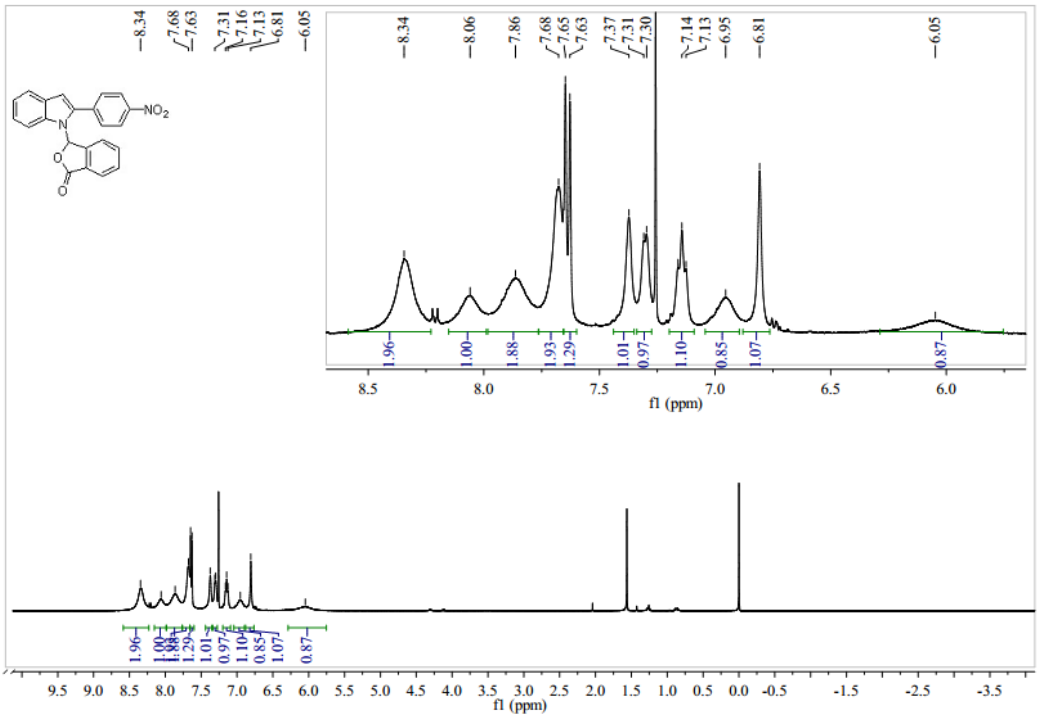
^{1}H NMR spectrum of compound 13



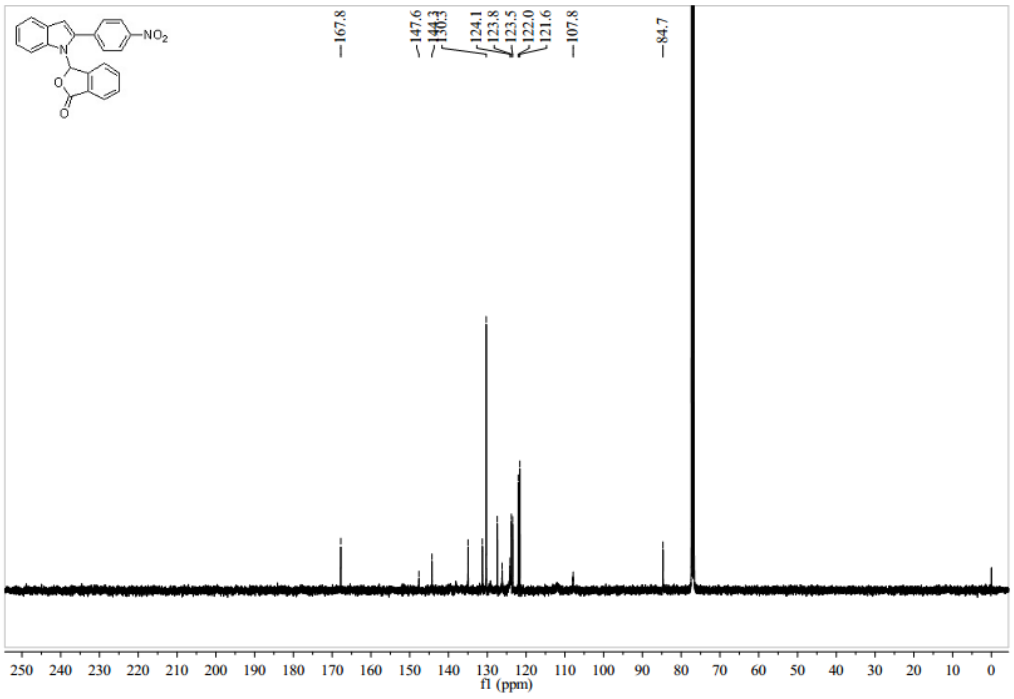
^{13}C NMR spectrum of compound 13



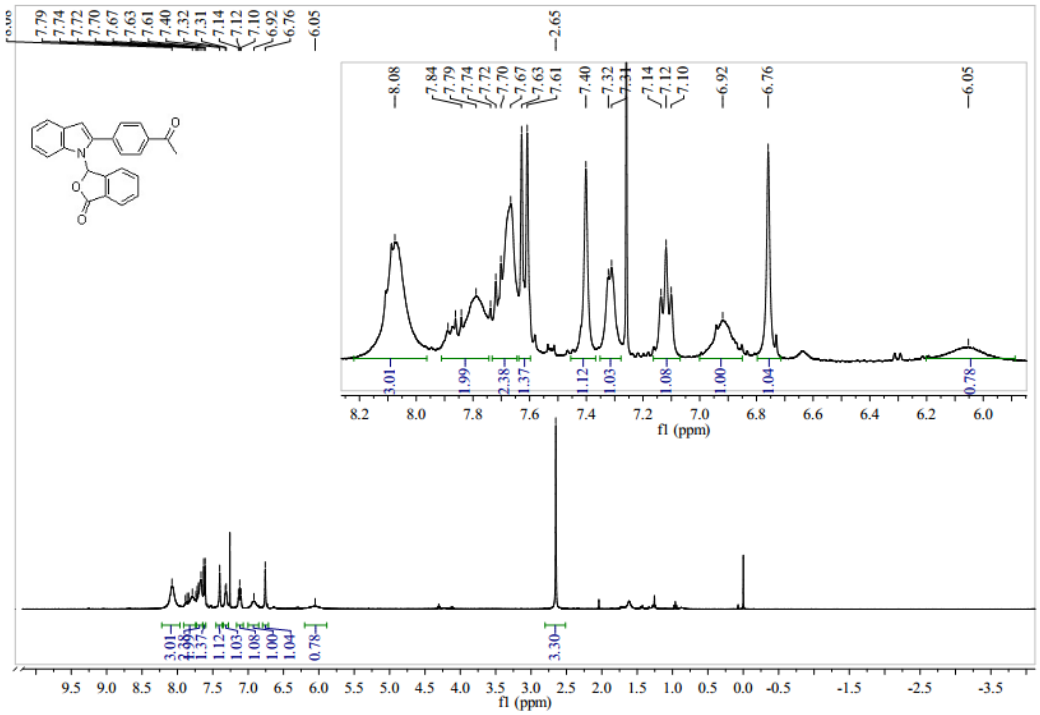
^{1}H NMR spectrum of compound 14



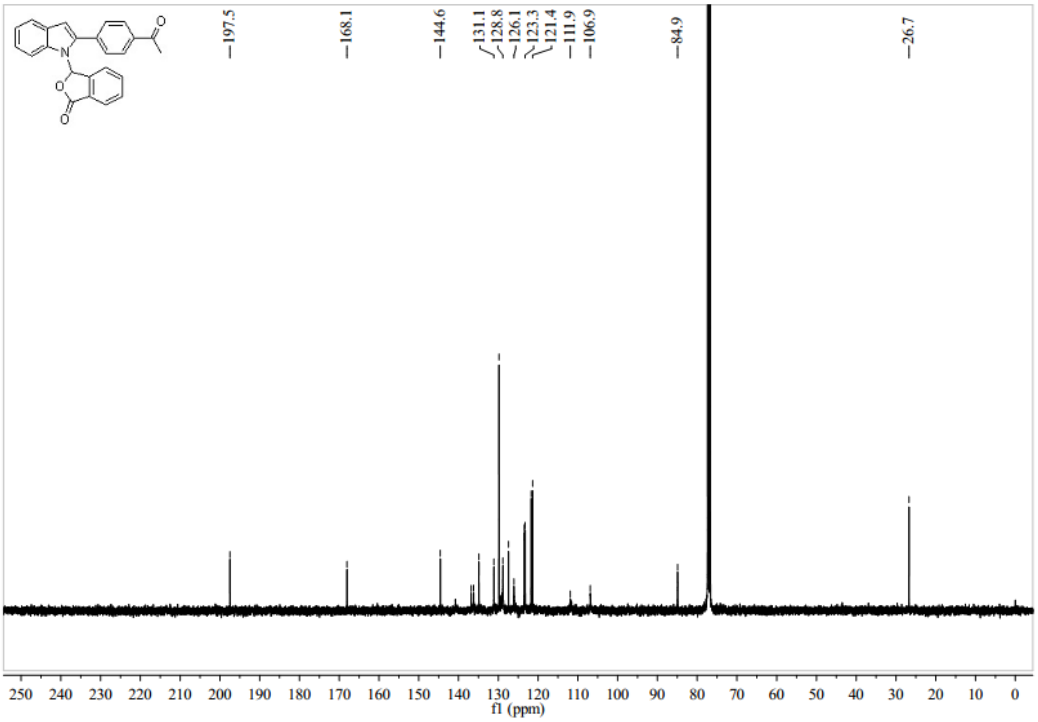
^{13}C NMR spectrum of compound 14



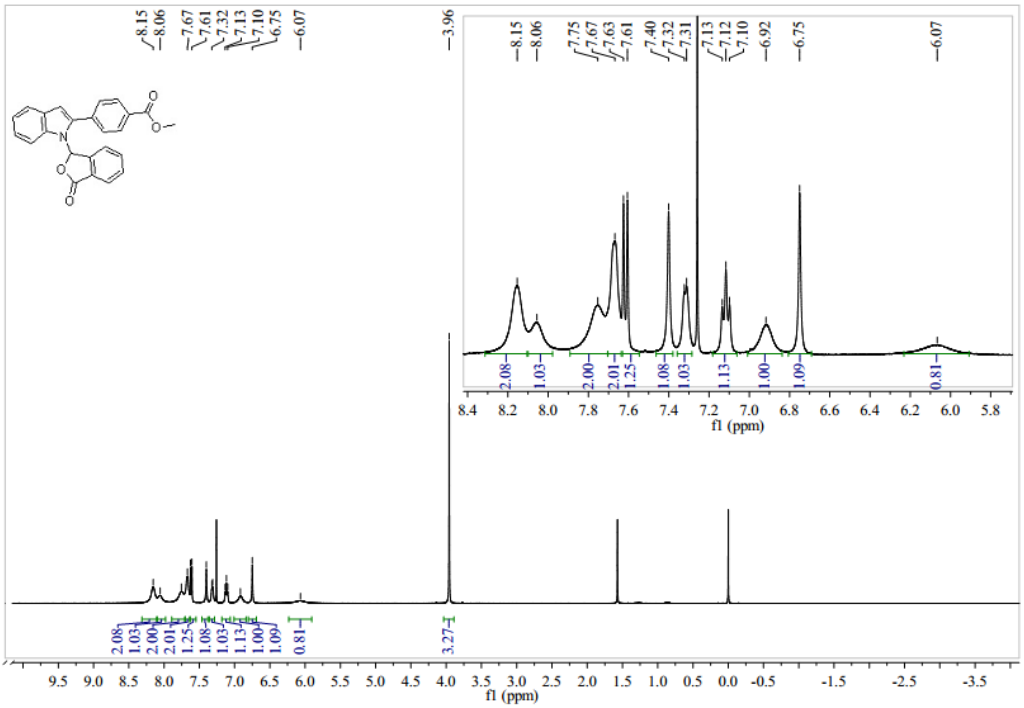
^{1}H NMR spectrum of compound 15



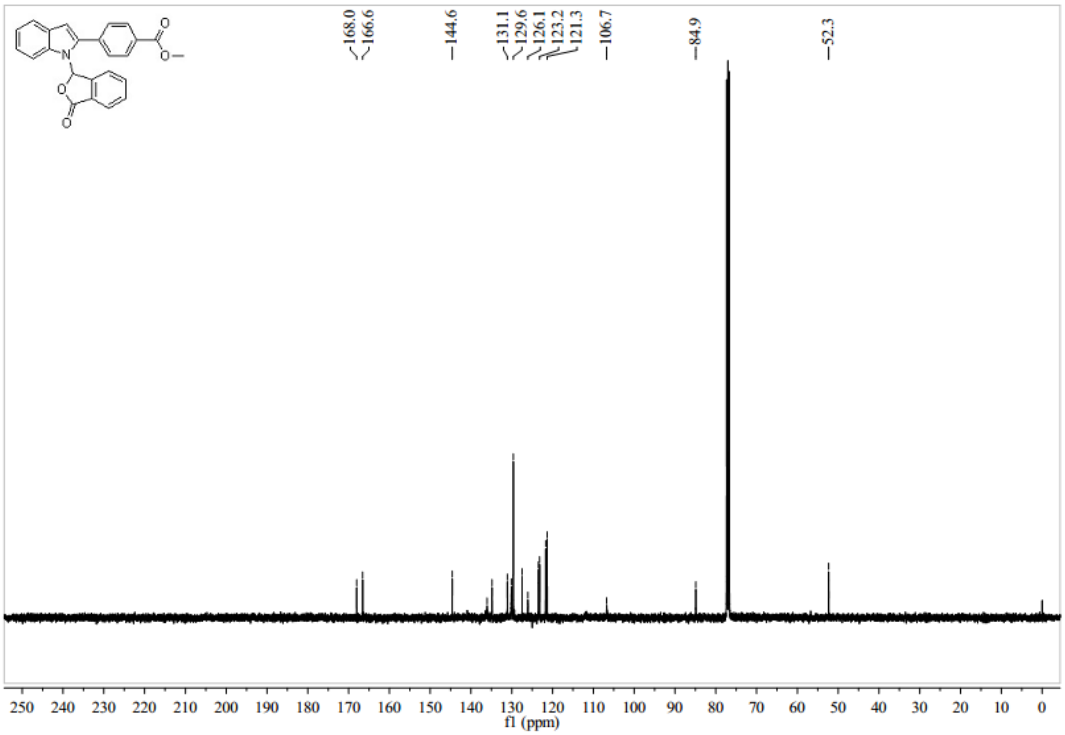
^{13}C NMR spectrum of compound 15



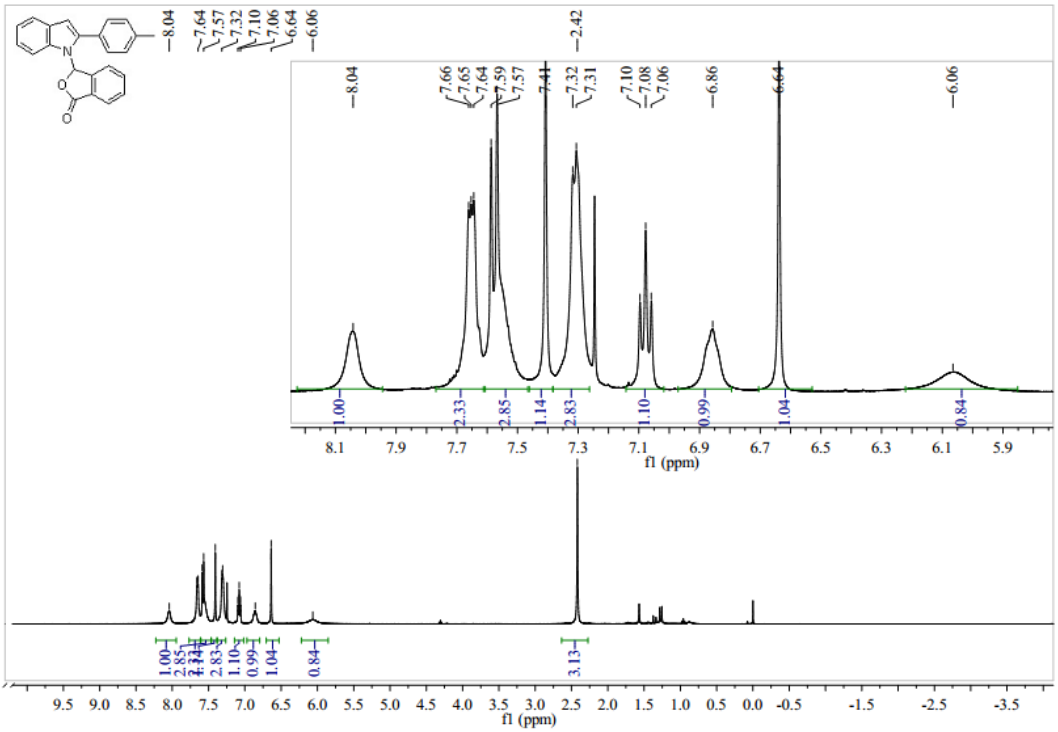
^{1}H NMR spectrum of compound 16



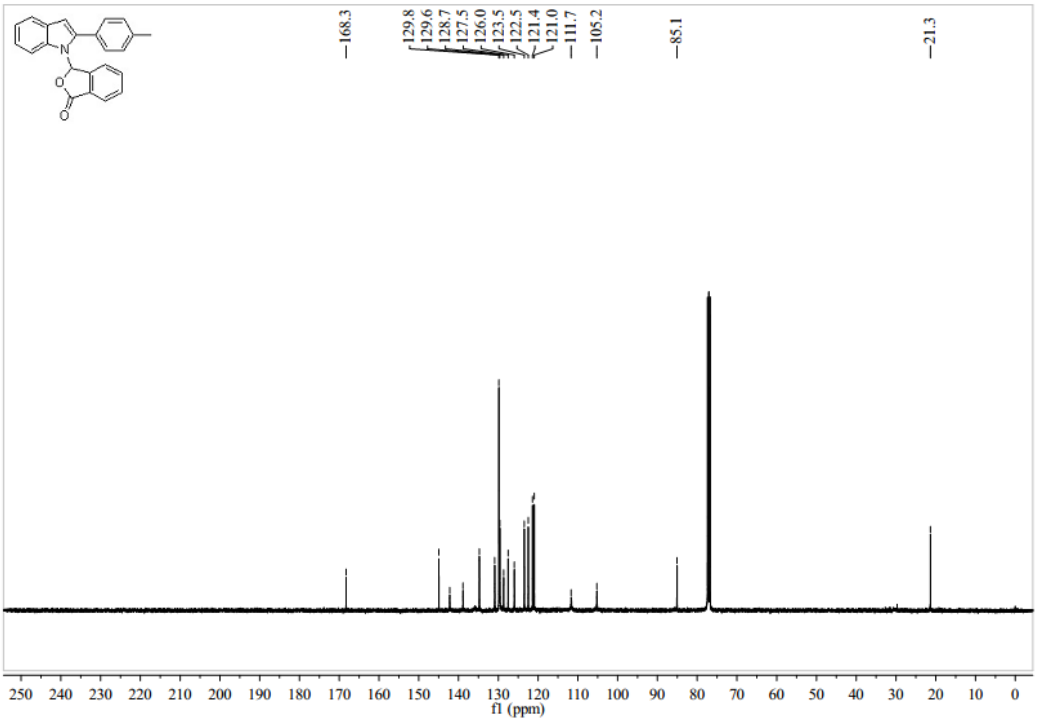
^{13}C NMR spectrum of compound 16



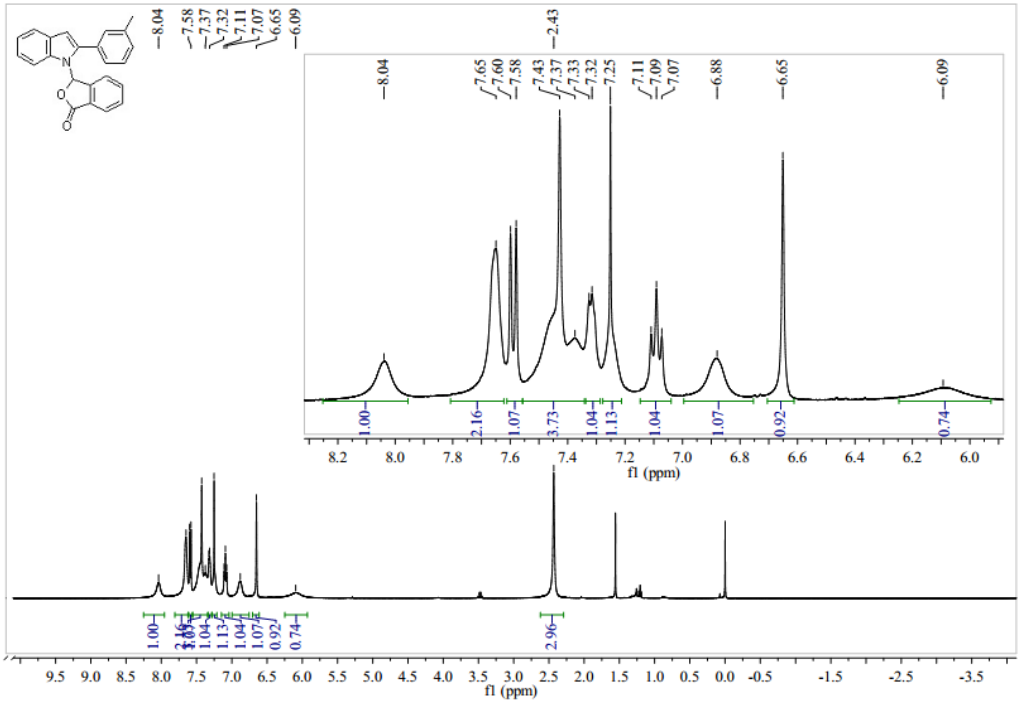
^{1}H NMR spectrum of compound 17



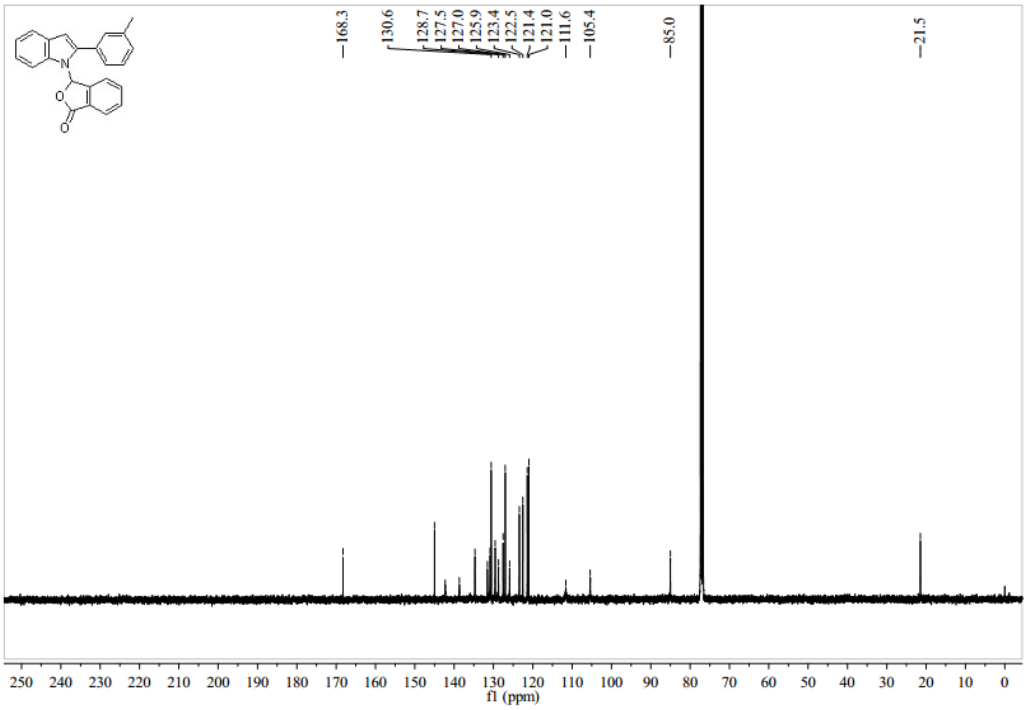
^{13}C NMR spectrum of compound 17



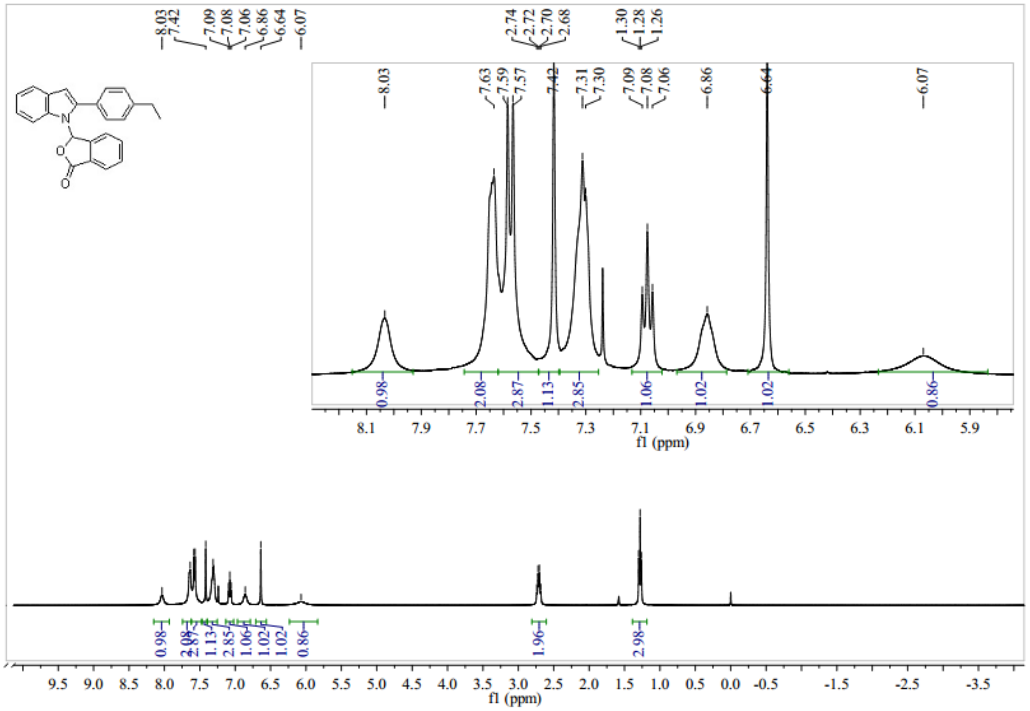
^{1}H NMR spectrum of compound 18



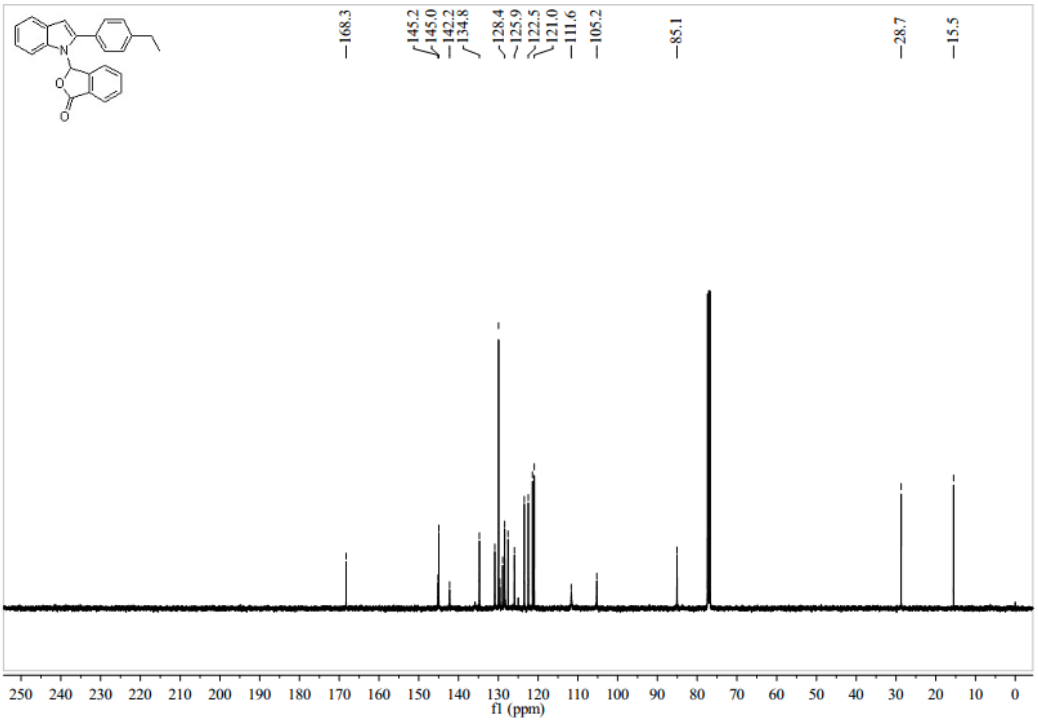
^{13}C NMR spectrum of compound 18



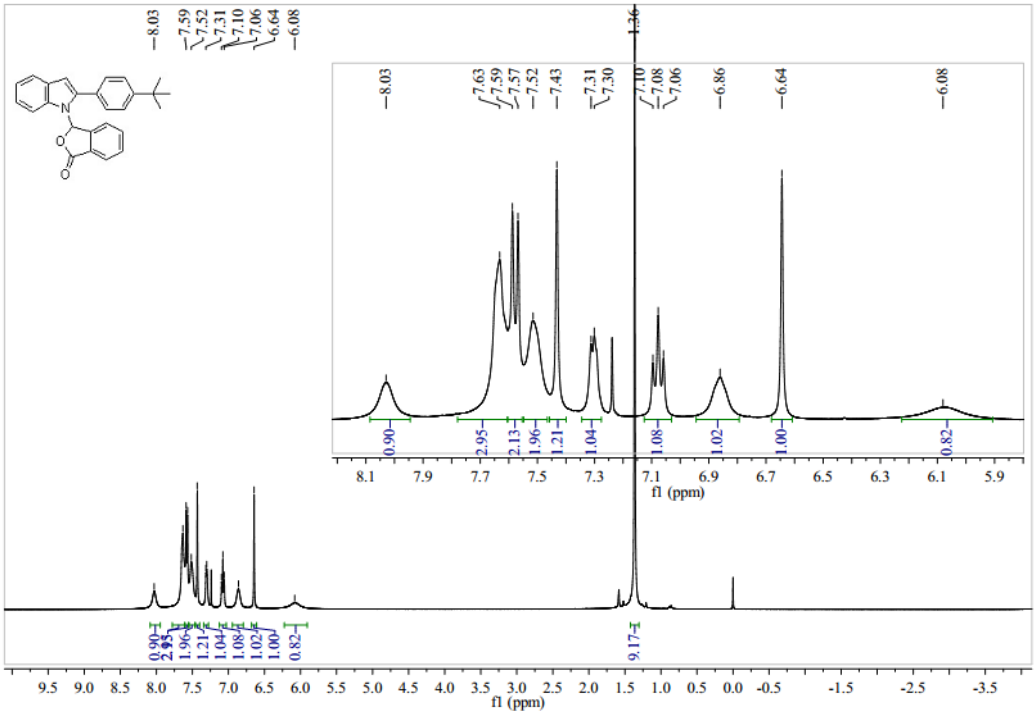
^{1}H NMR spectrum of compound 19



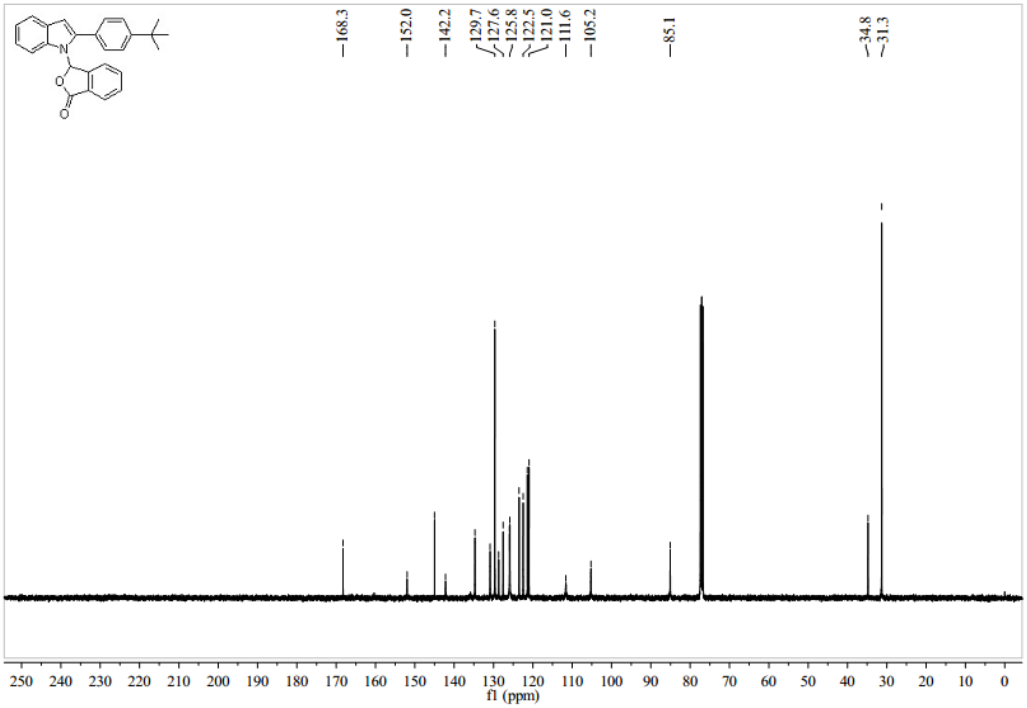
^{13}C NMR spectrum of compound 19



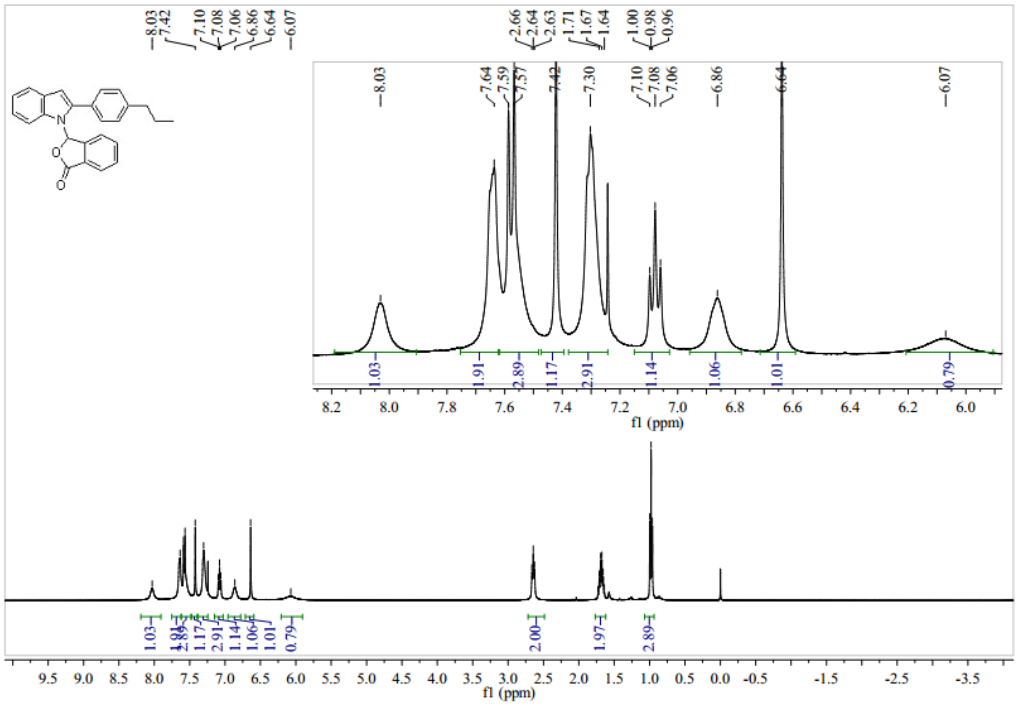
^{1}H NMR spectrum of compound 20



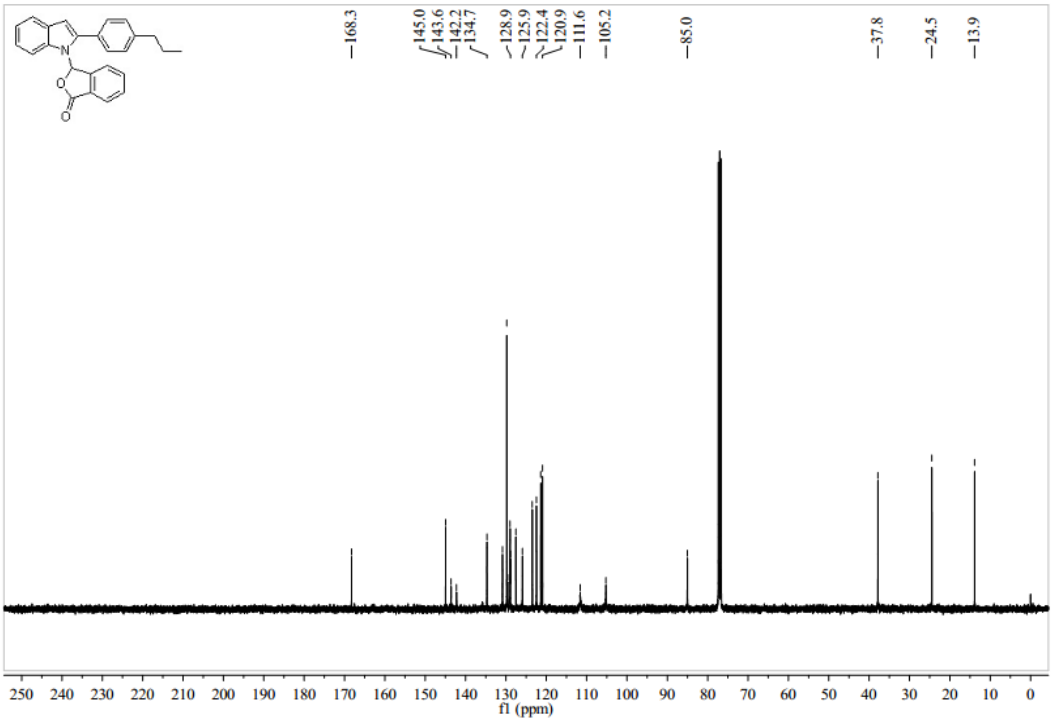
^{13}C NMR spectrum of compound 20



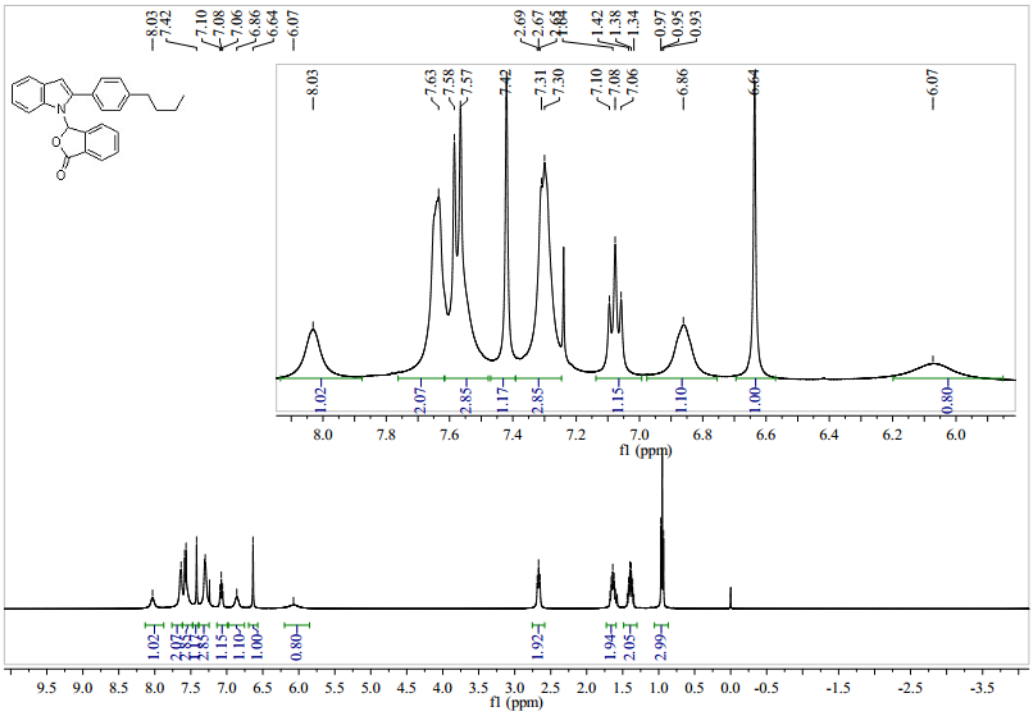
^{1}H NMR spectrum of compound 21



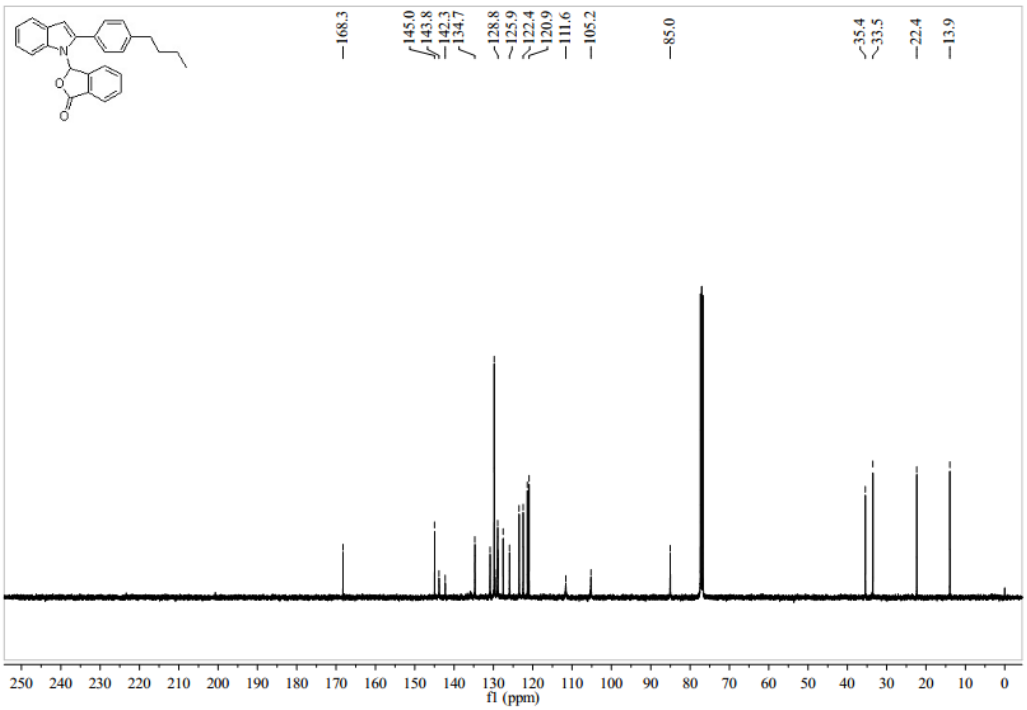
^{13}C NMR spectrum of compound 21



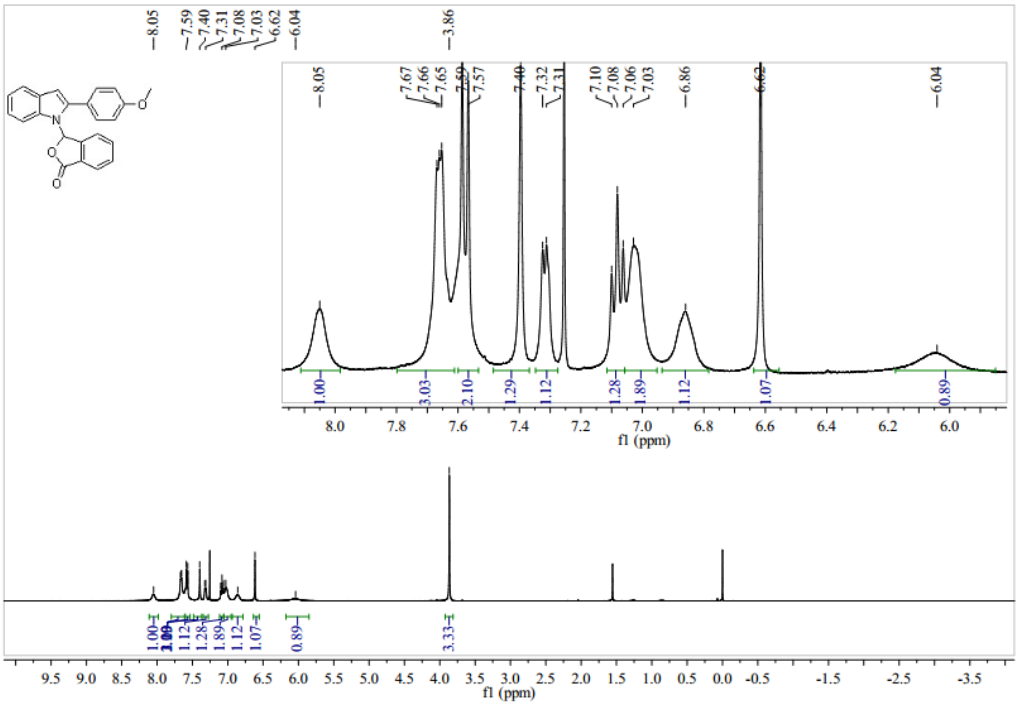
^{1}H NMR spectrum of compound 22



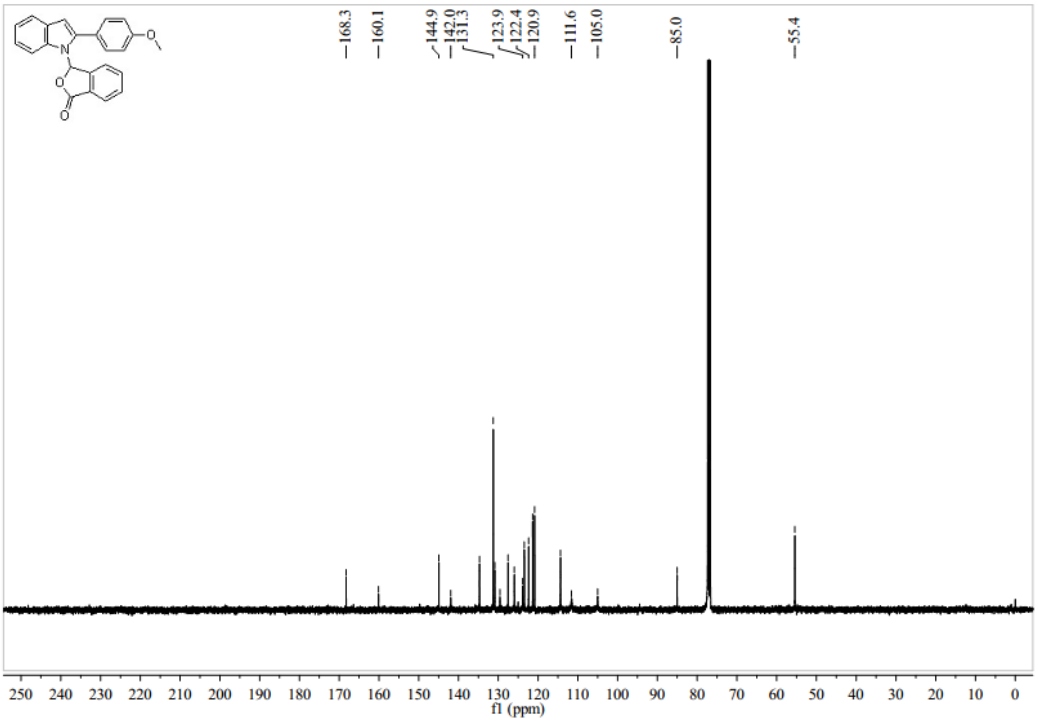
^{13}C NMR spectrum of compound 22



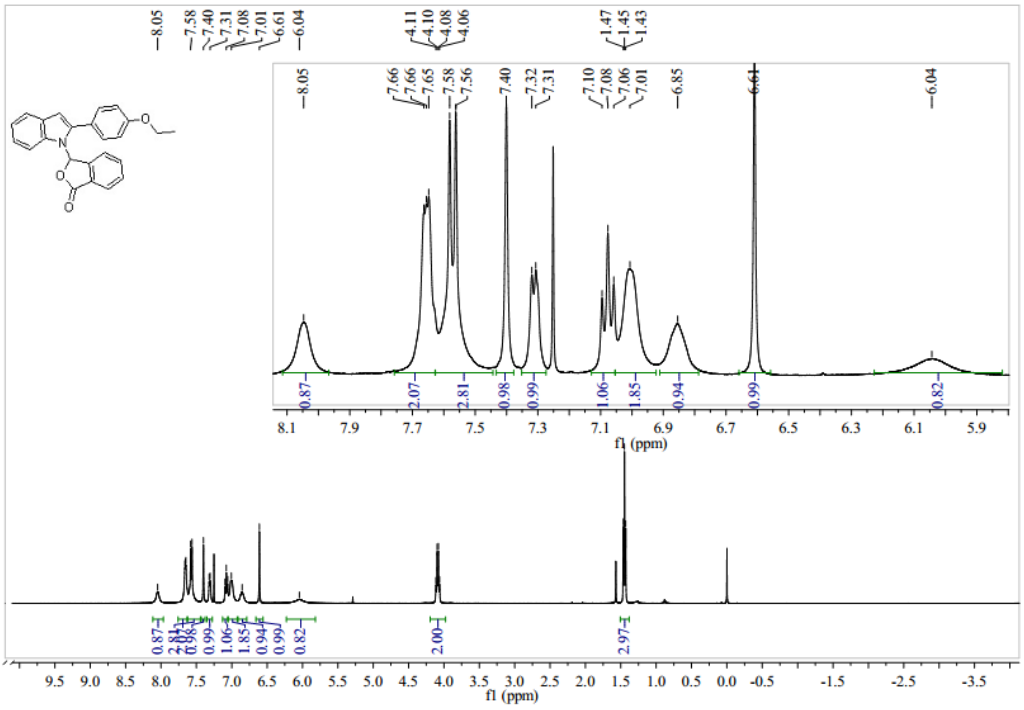
^{1}H NMR spectrum of compound 23



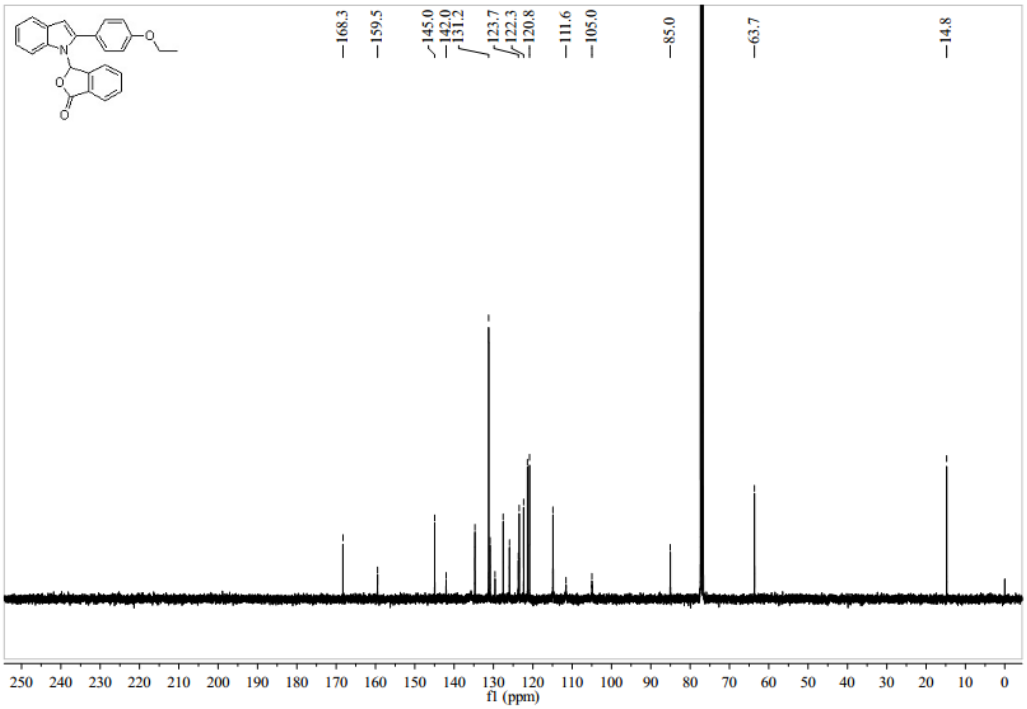
^{13}C NMR spectrum of compound 23



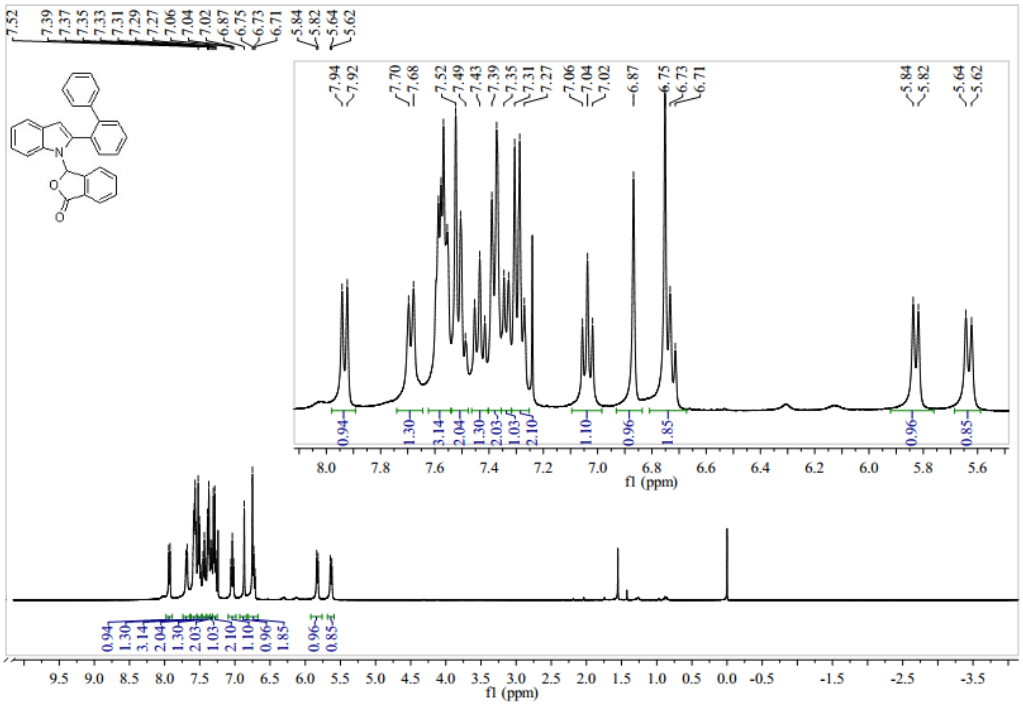
^{1}H NMR spectrum of compound 24



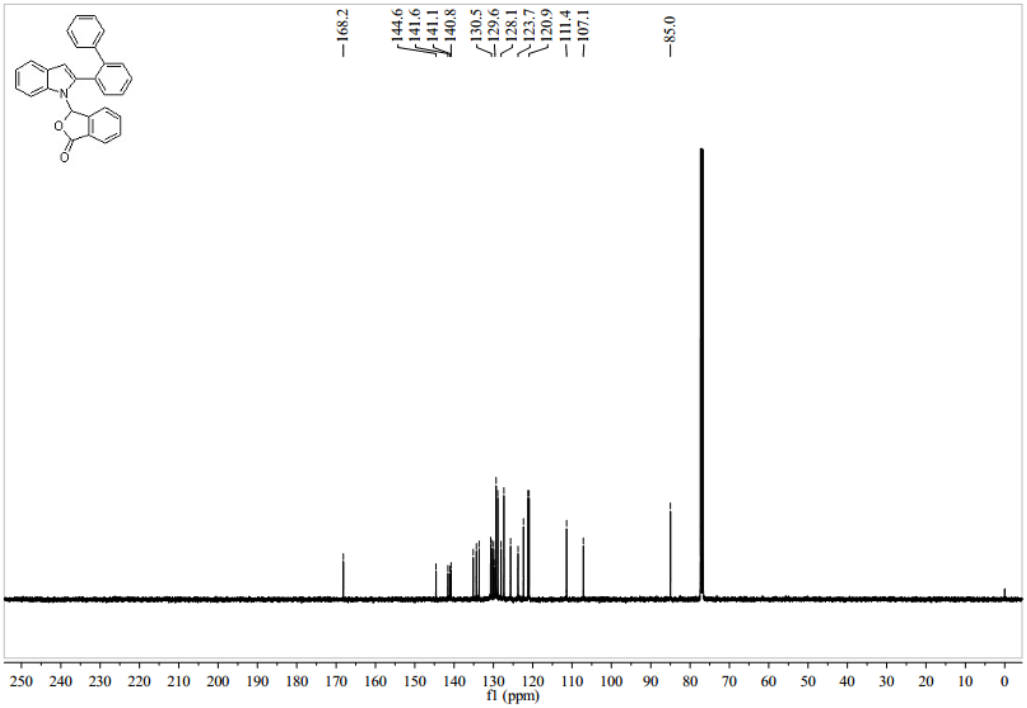
^{13}C NMR spectrum of compound 24



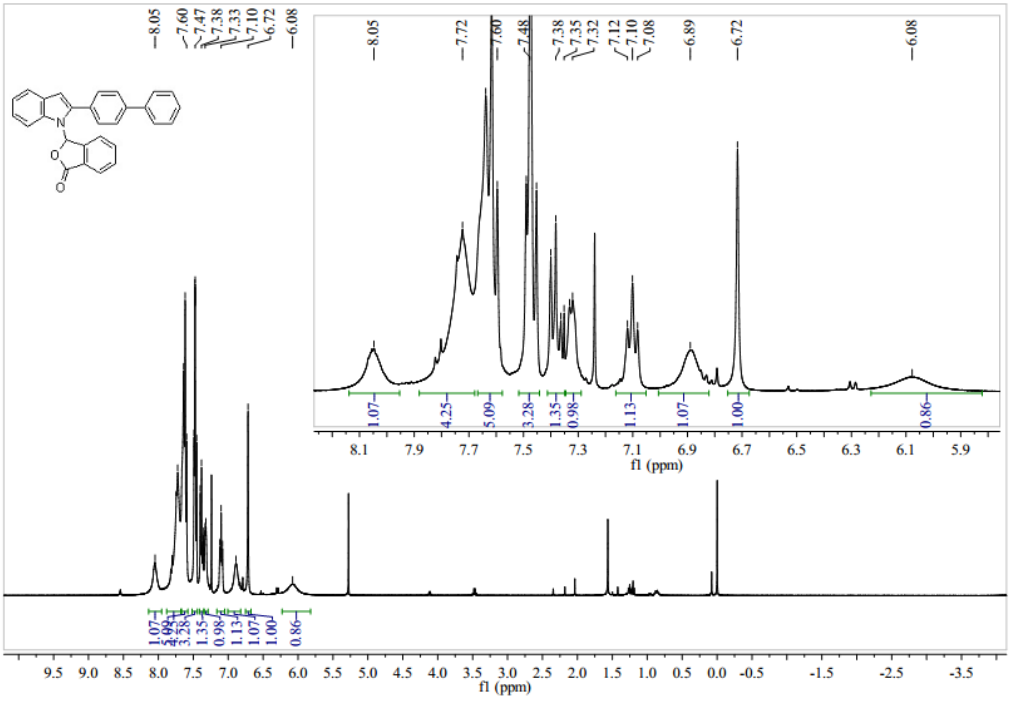
^{1}H NMR spectrum of compound 25



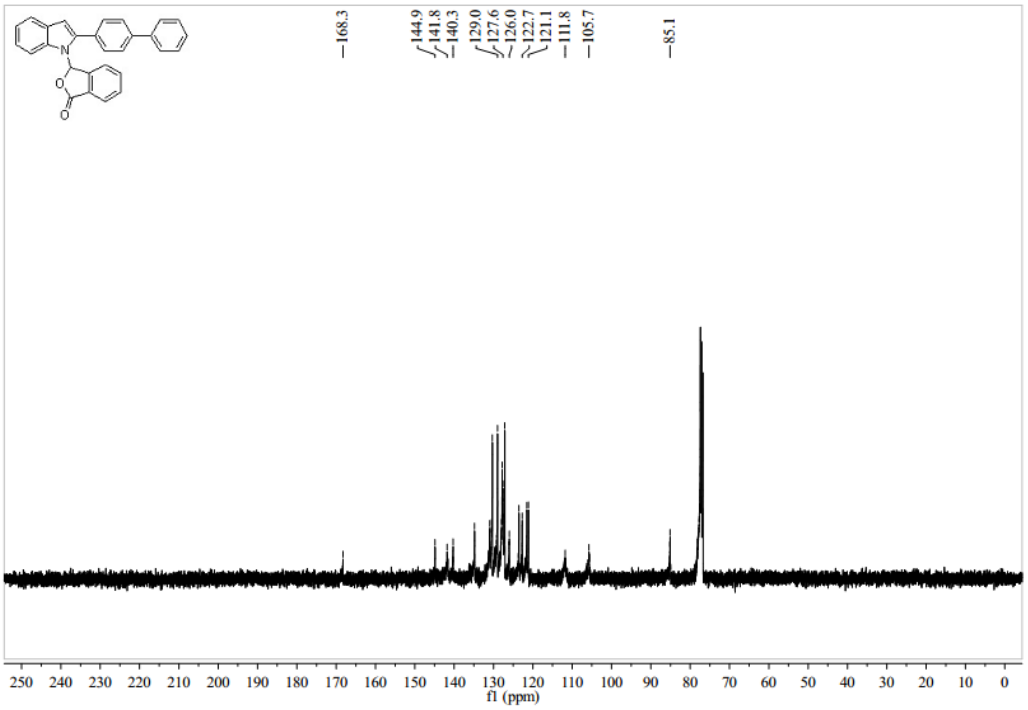
^{13}C NMR spectrum of compound 25



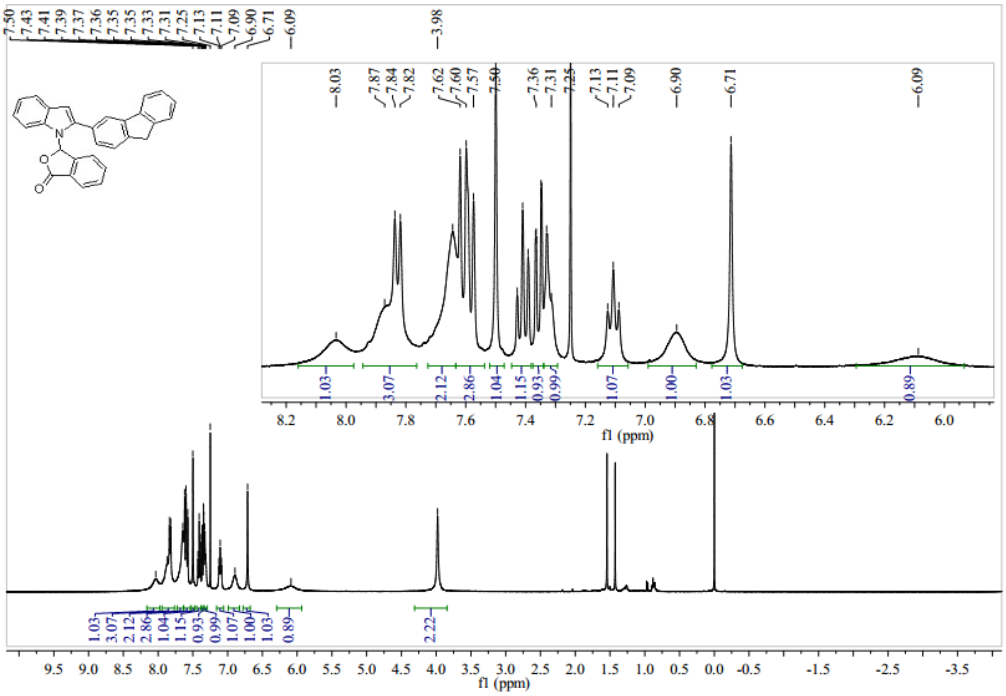
^{1}H NMR spectrum of compound 26



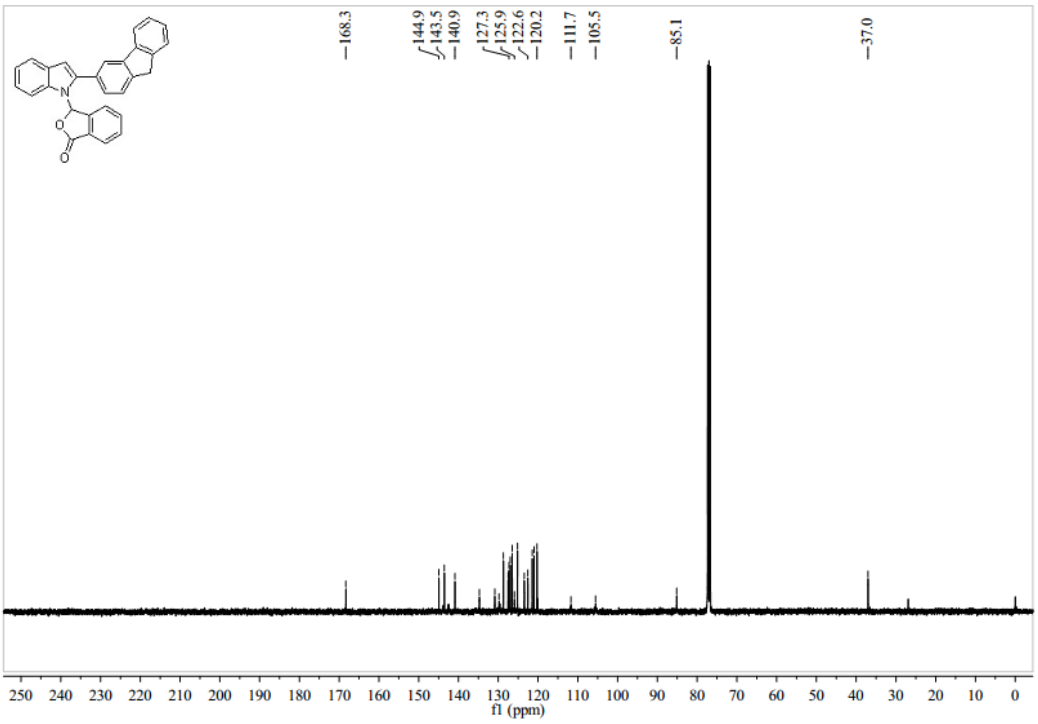
^{13}C NMR spectrum of compound 26



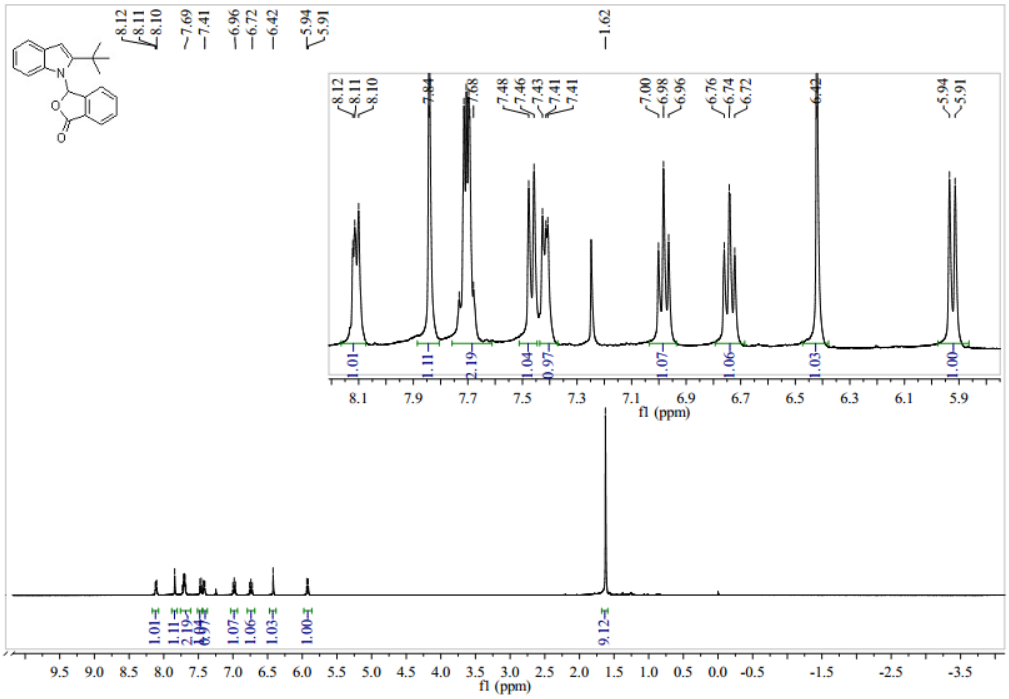
^{1}H NMR spectrum of compound 27



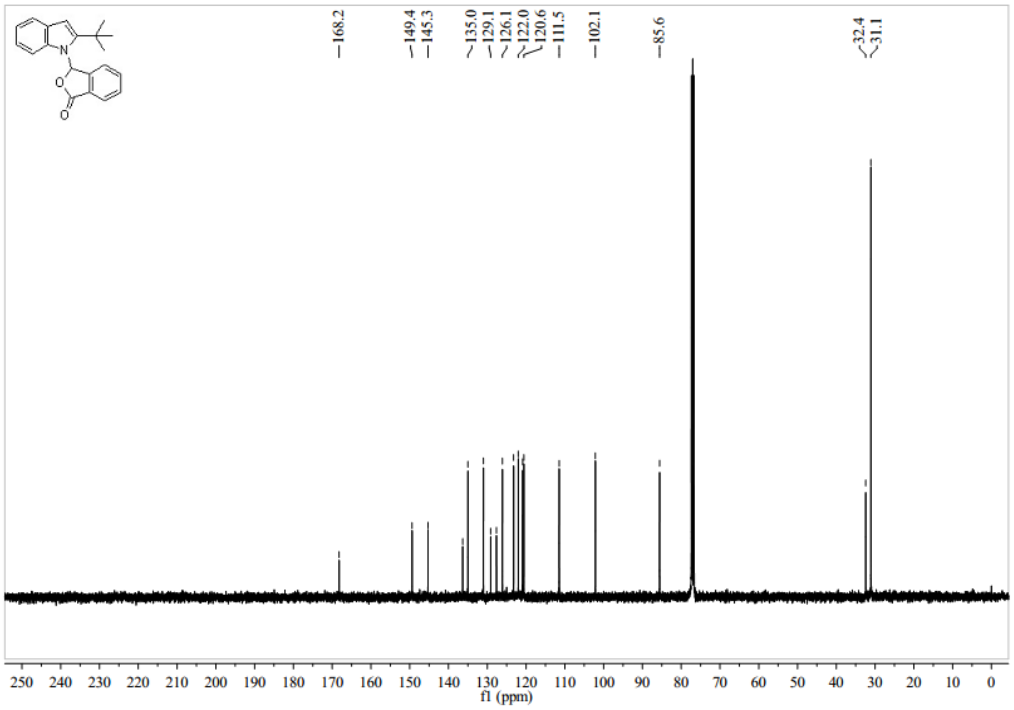
^{13}C NMR spectrum of compound 27



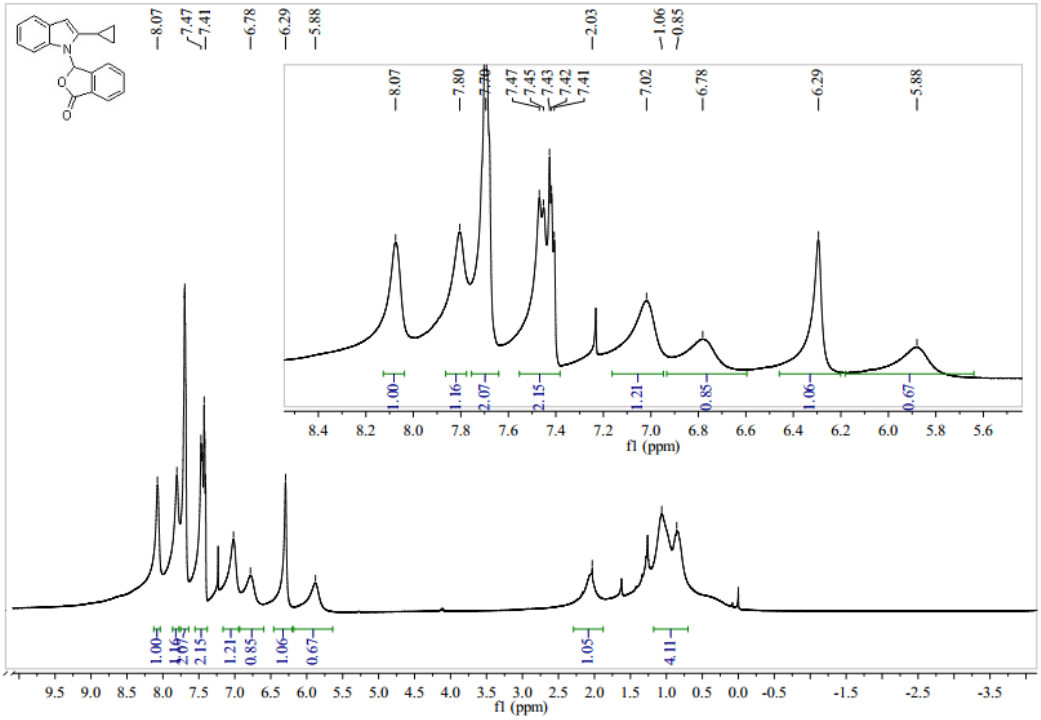
^{1}H NMR spectrum of compound 28



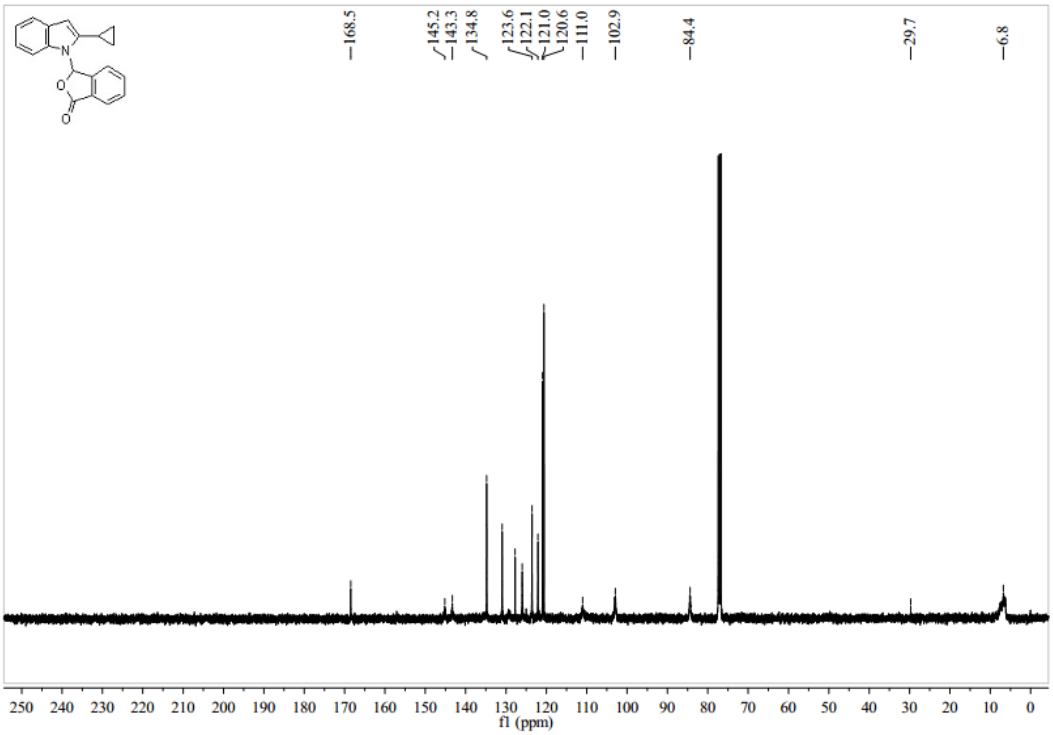
^{13}C NMR spectrum of compound 28



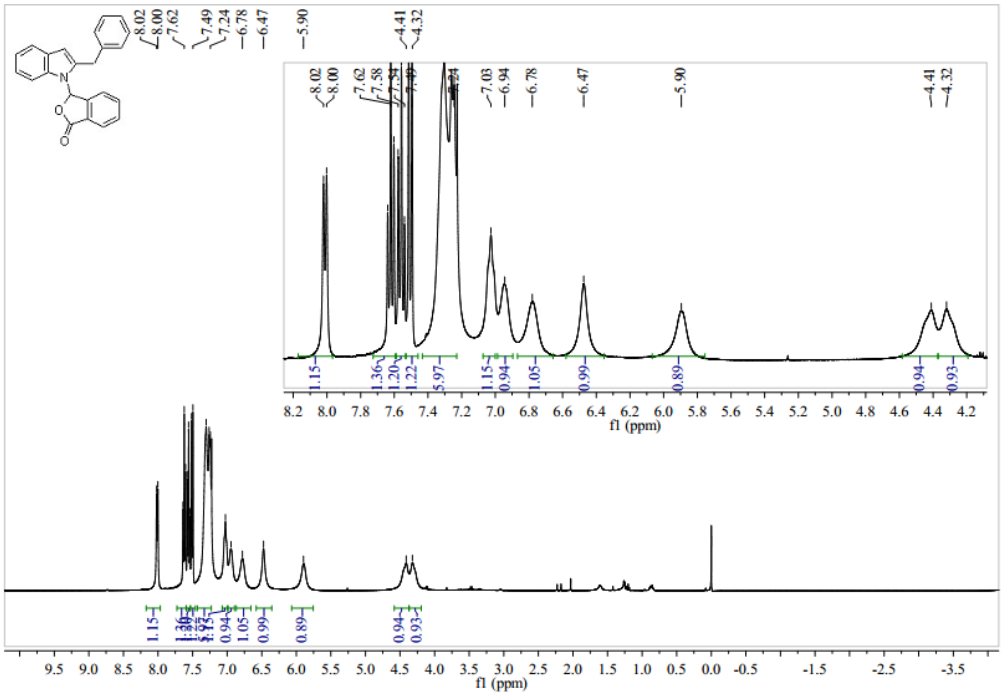
^{1}H NMR spectrum of compound 29



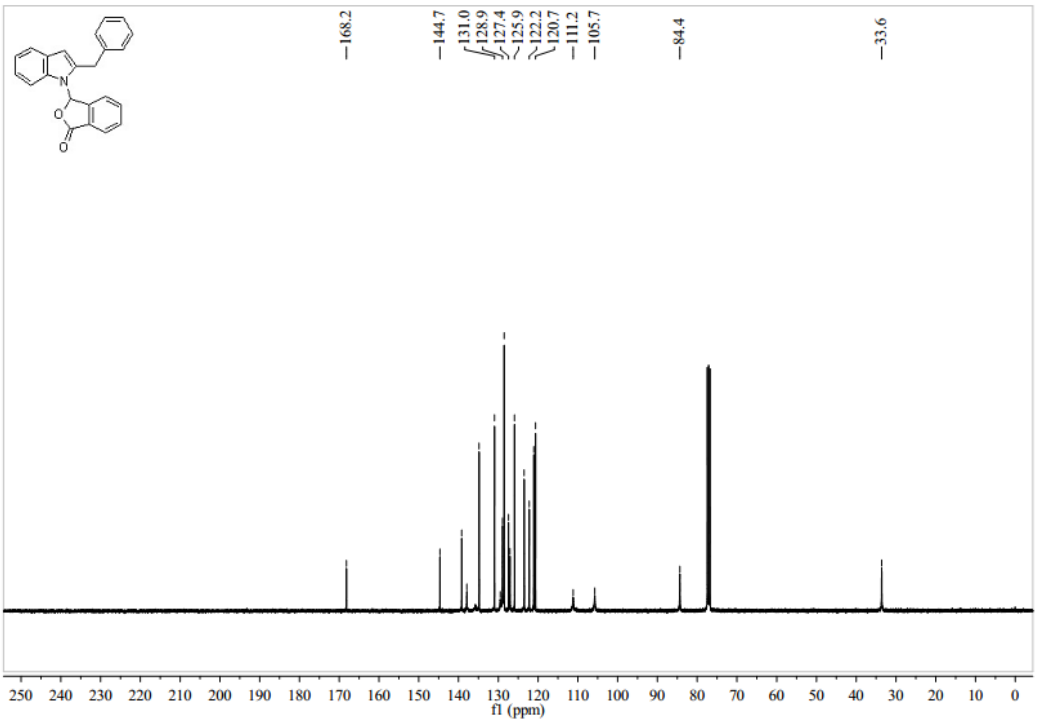
^{13}C NMR spectrum of compound 29



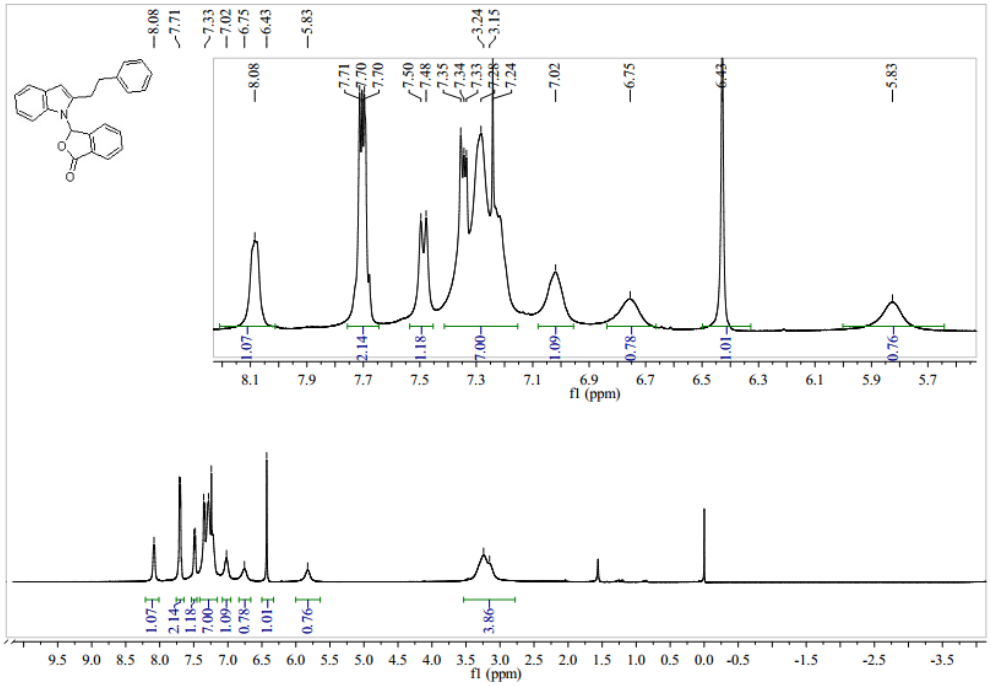
^{1}H NMR spectrum of compound 30



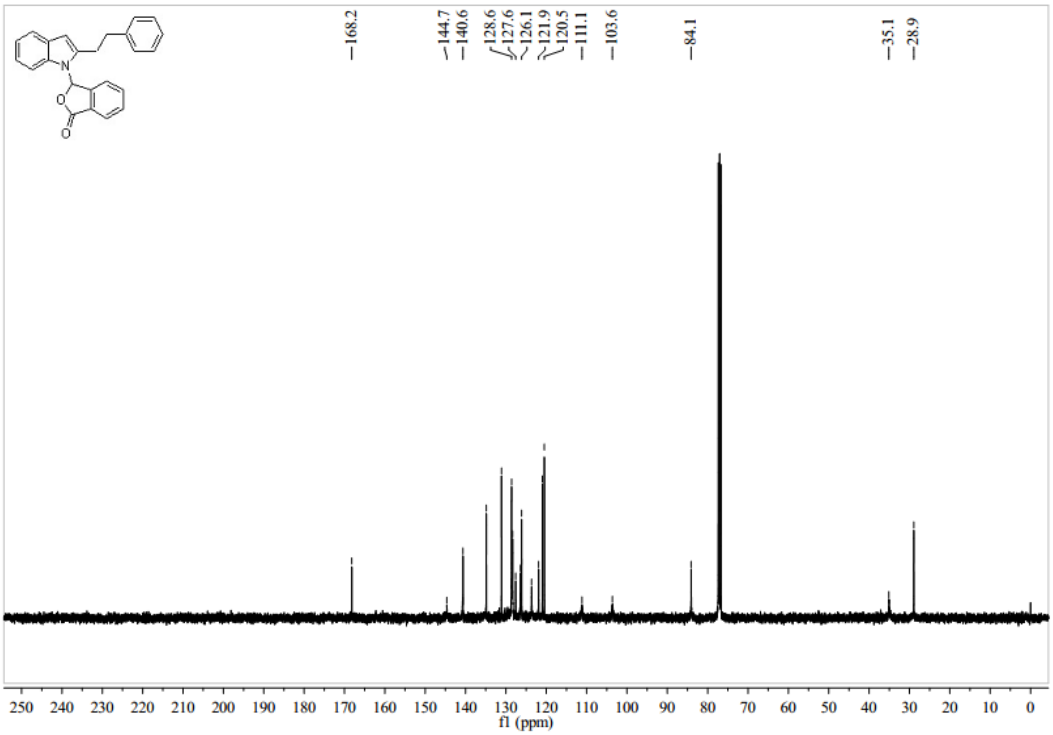
^{13}C NMR spectrum of compound 30



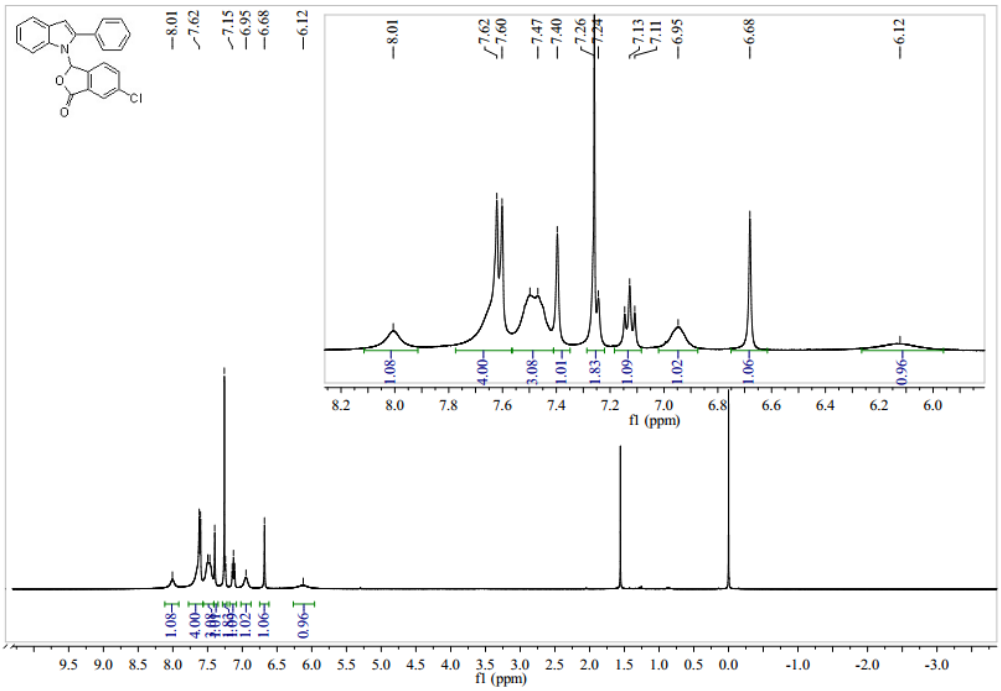
^{1}H NMR spectrum of compound 31



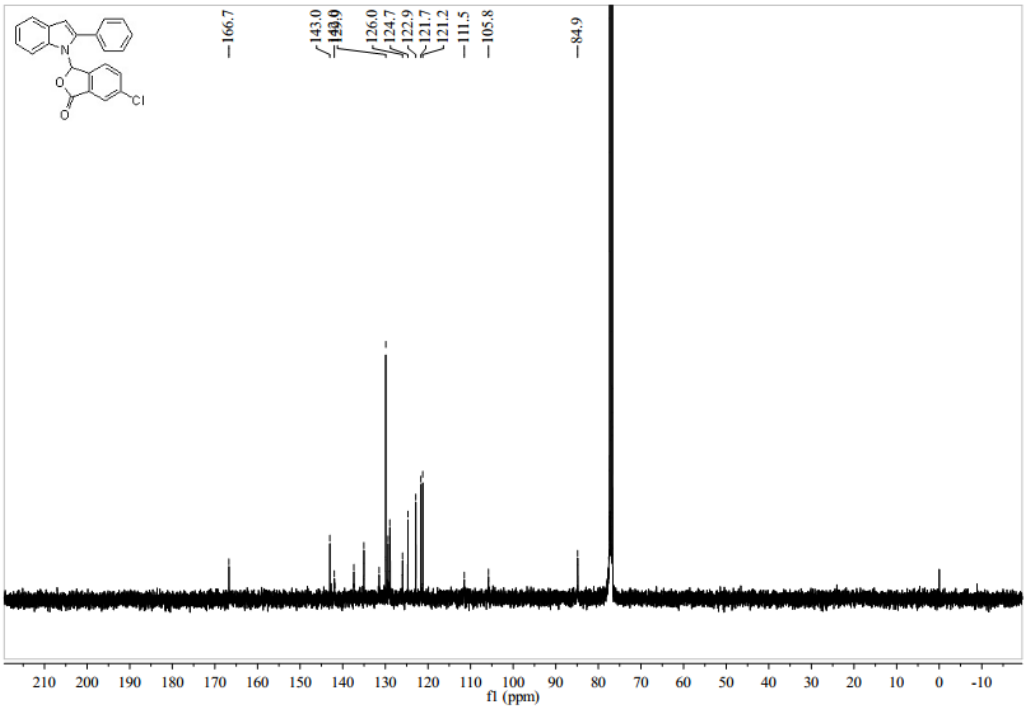
^{13}C NMR spectrum of compound 31



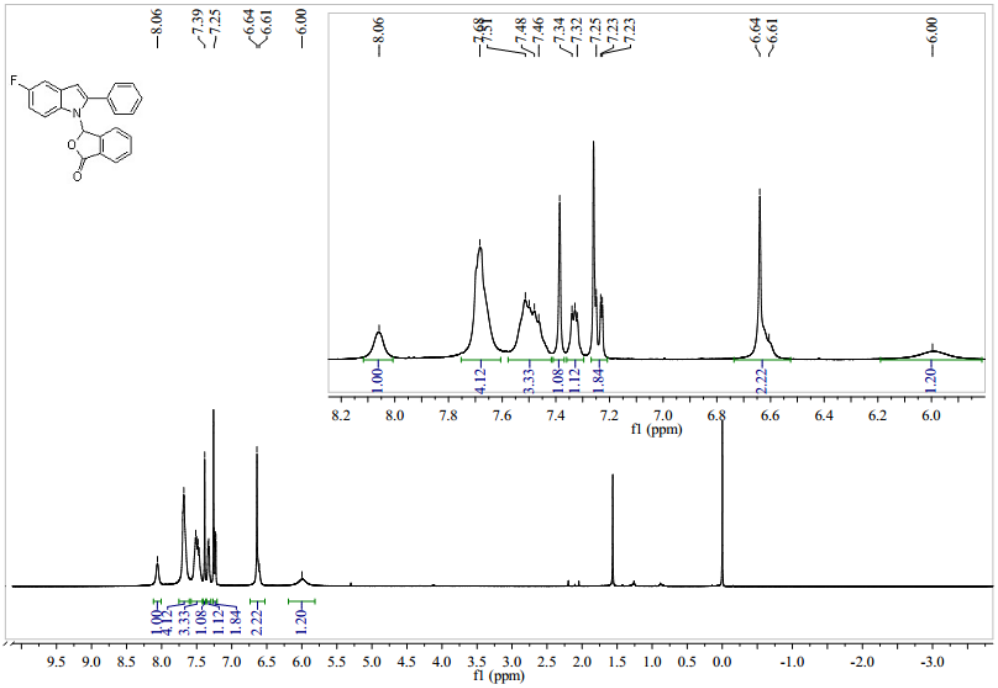
^{1}H NMR spectrum of compound 32



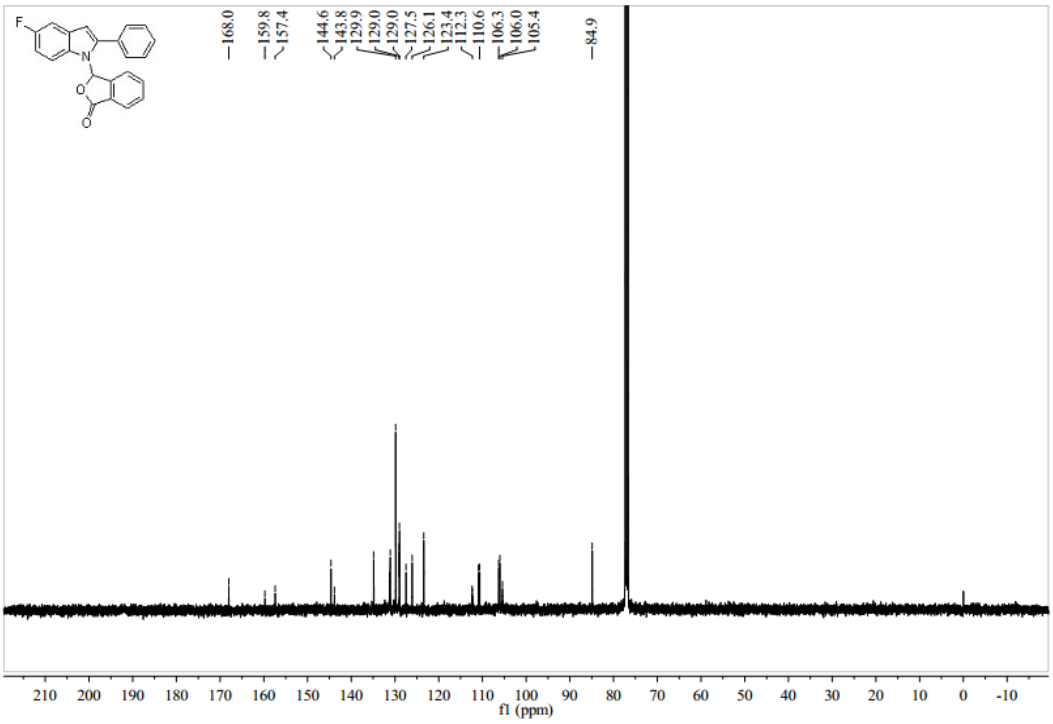
^{13}C NMR spectrum of compound 32



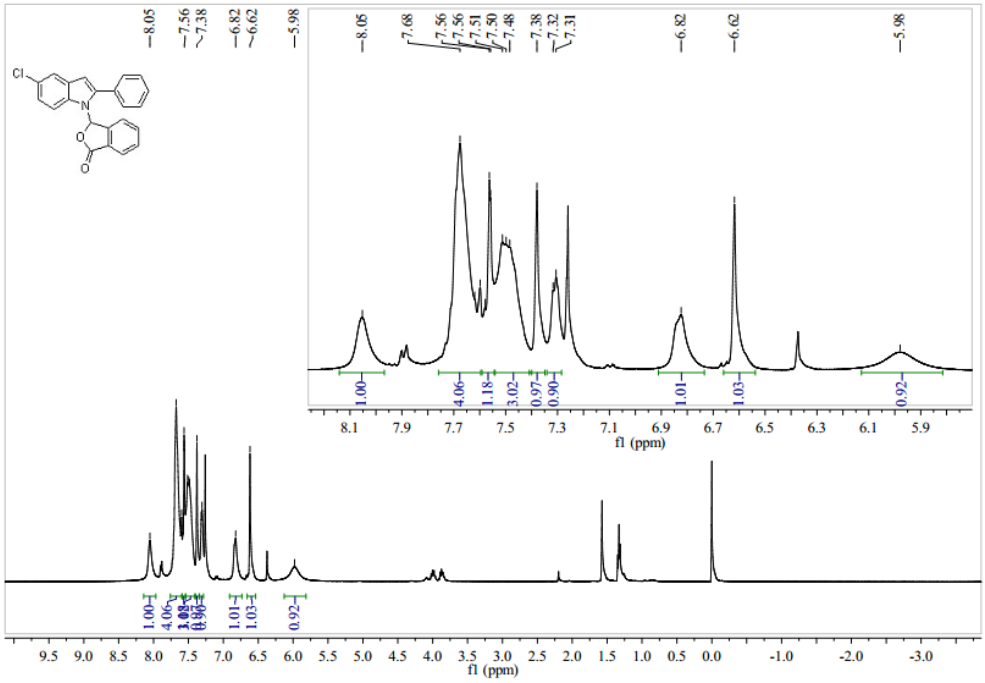
^{1}H NMR spectrum of compound 33



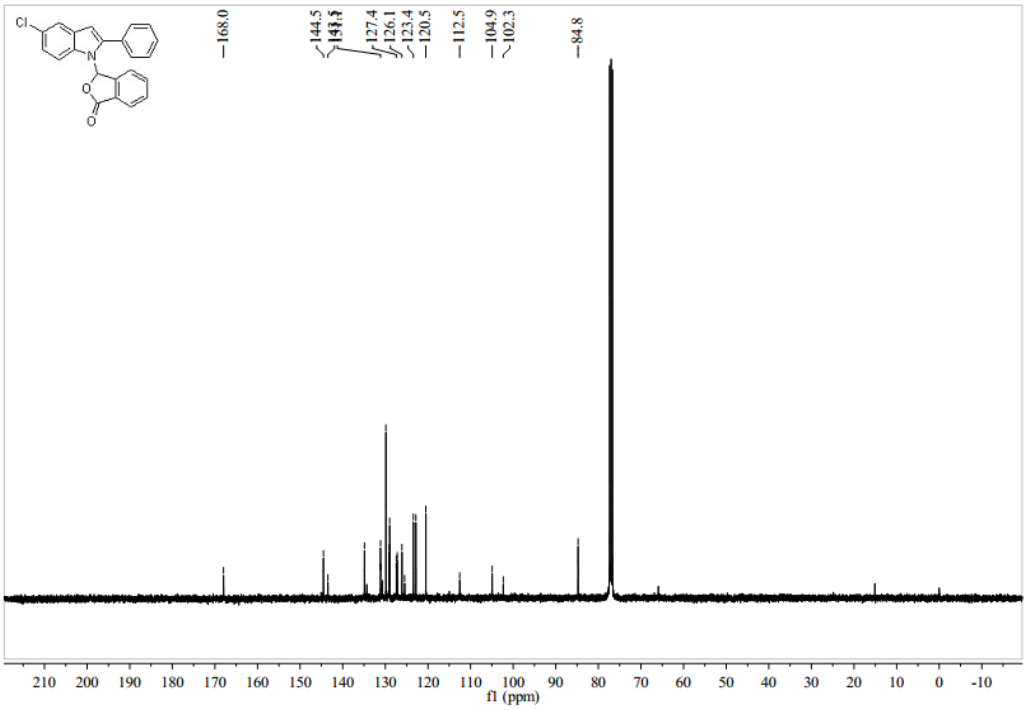
^{13}C NMR spectrum of compound 33



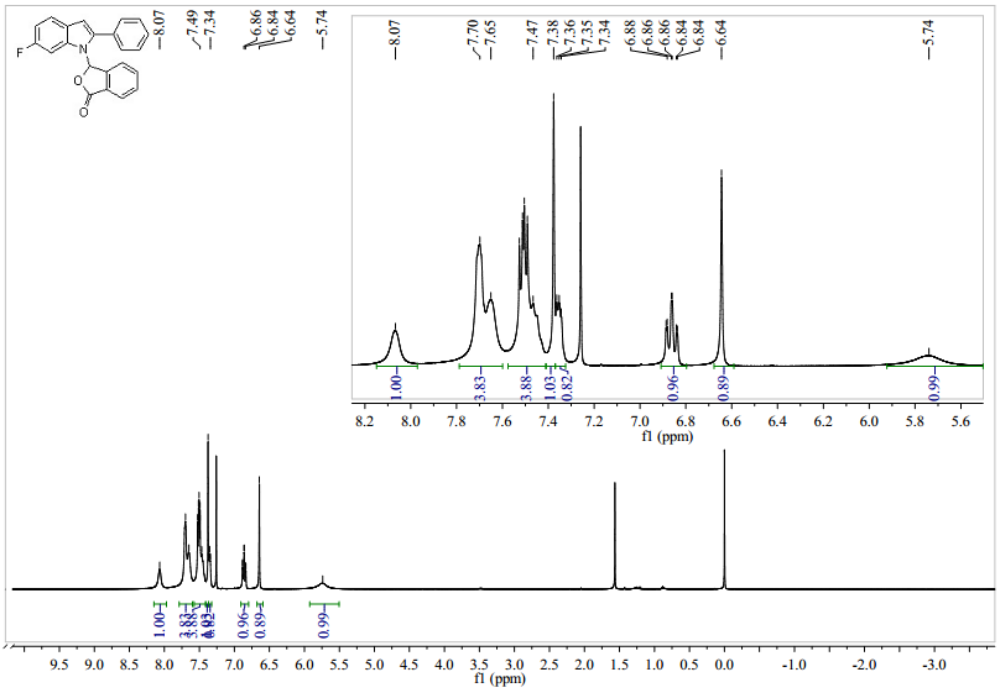
^{1}H NMR spectrum of compound 34



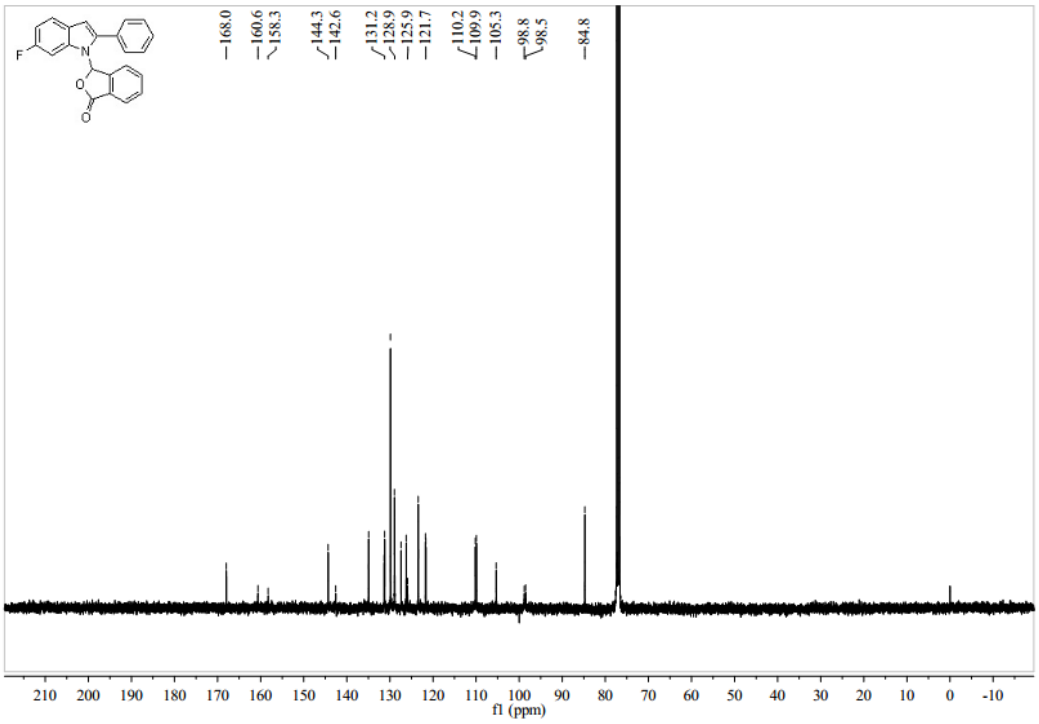
^{13}C NMR spectrum of compound 34



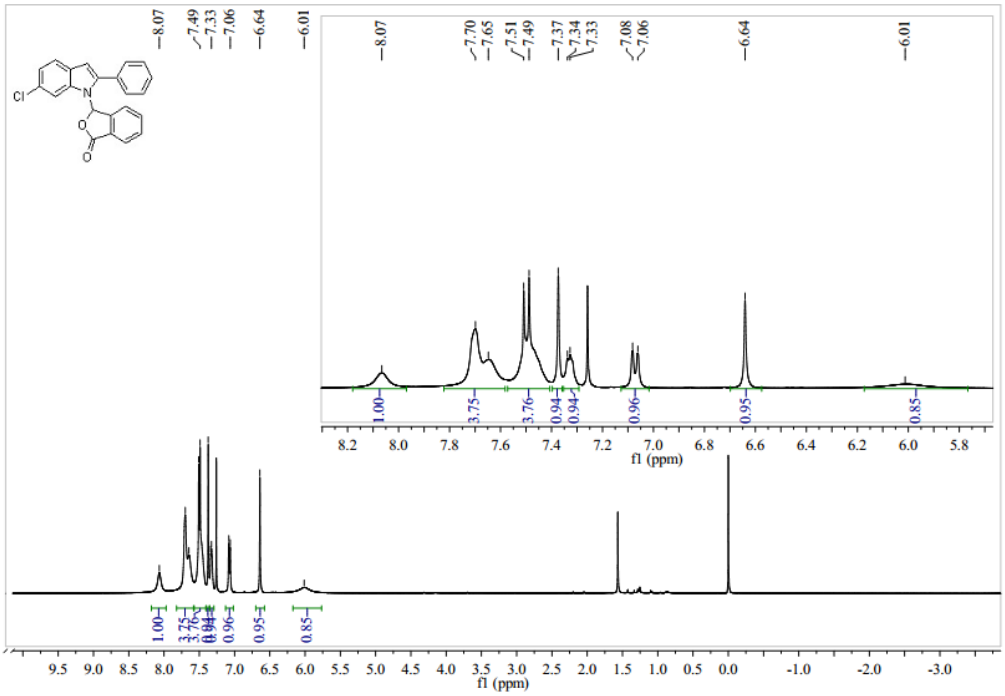
^{1}H NMR spectrum of compound 35



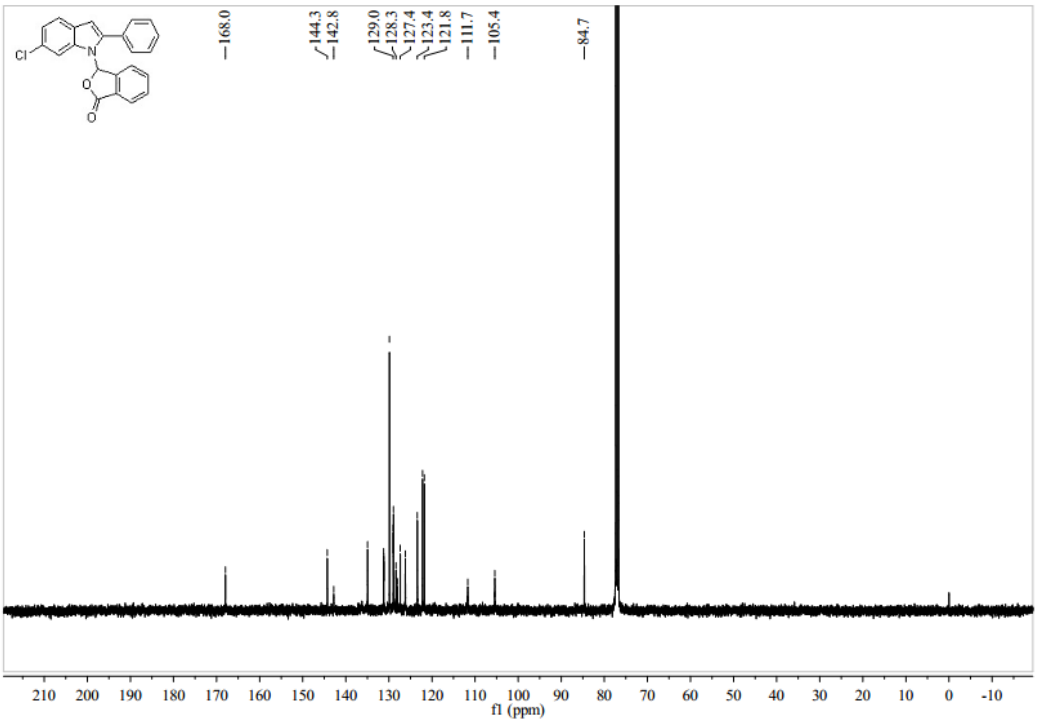
^{13}C NMR spectrum of compound 35



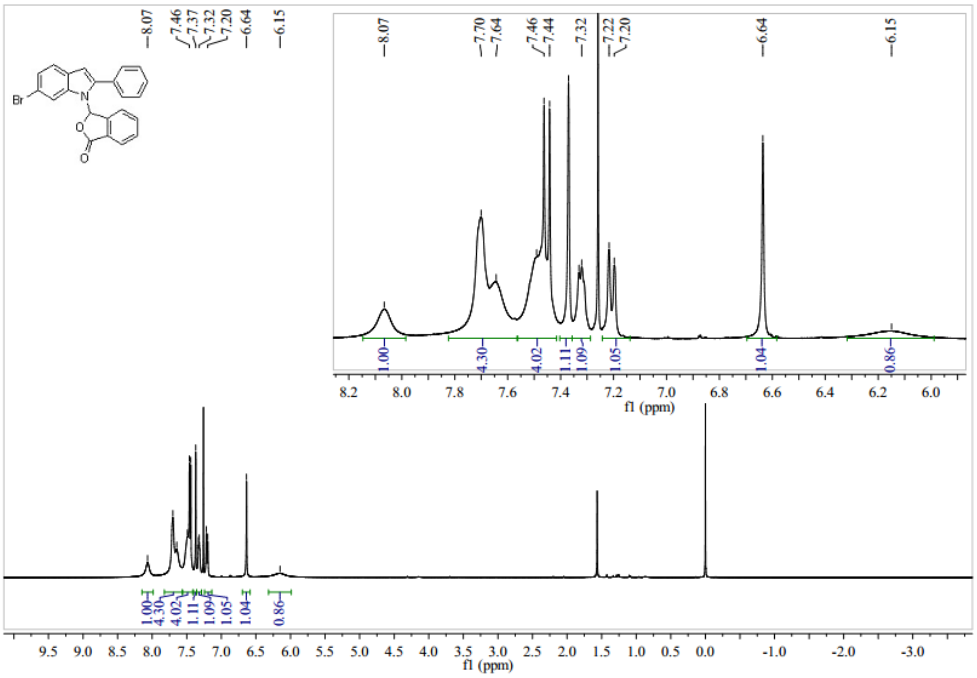
^{1}H NMR spectrum of compound 36



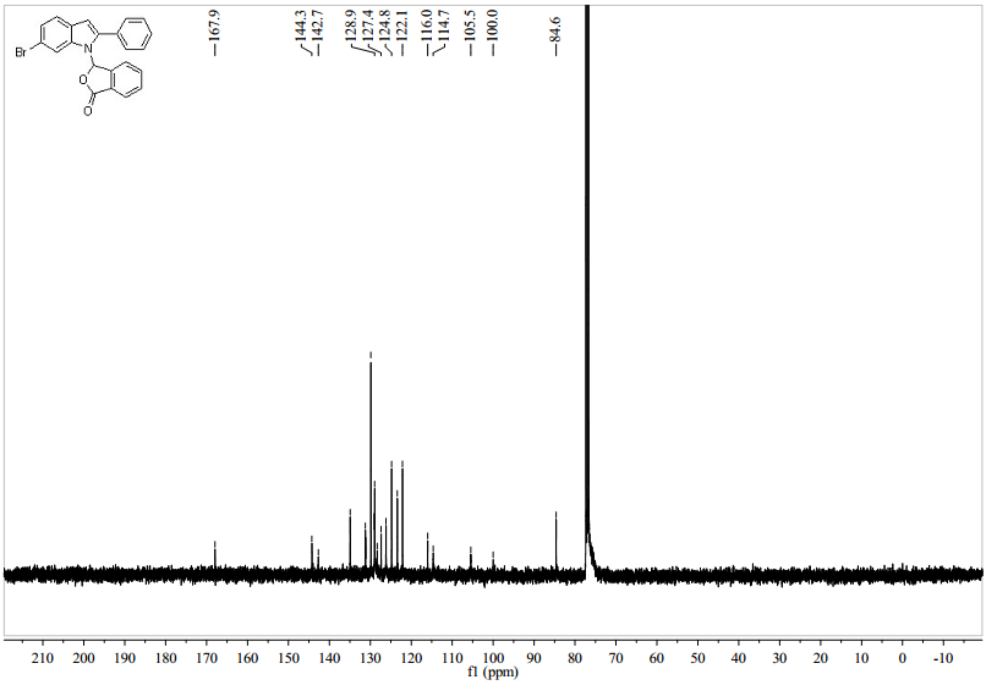
^{13}C NMR spectrum of compound 36



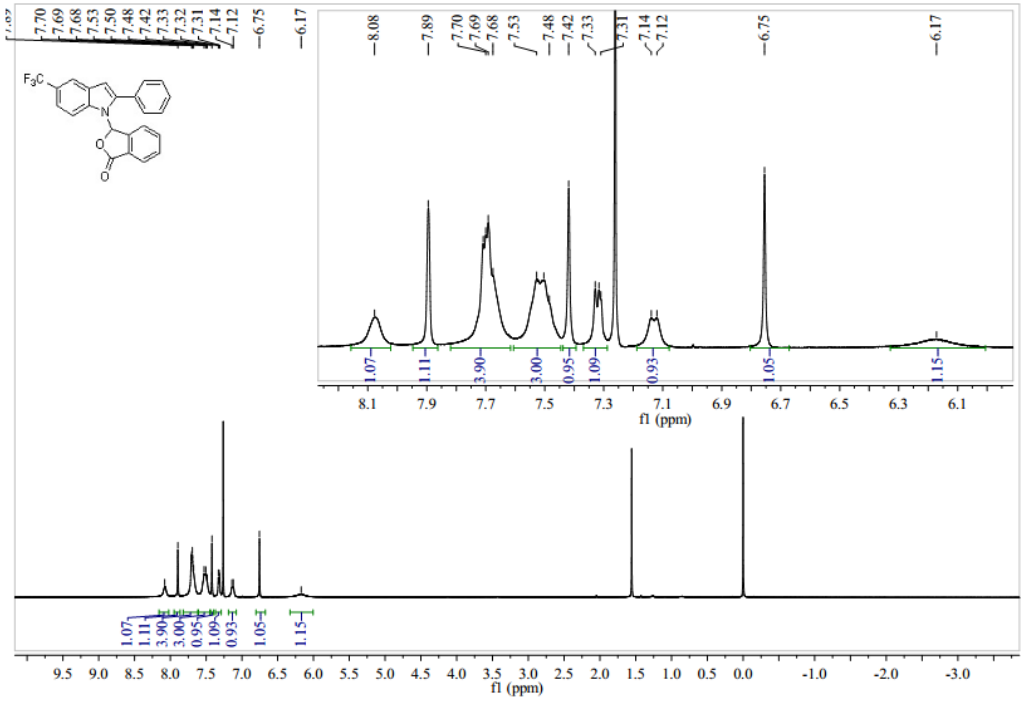
^{1}H NMR spectrum of compound 37



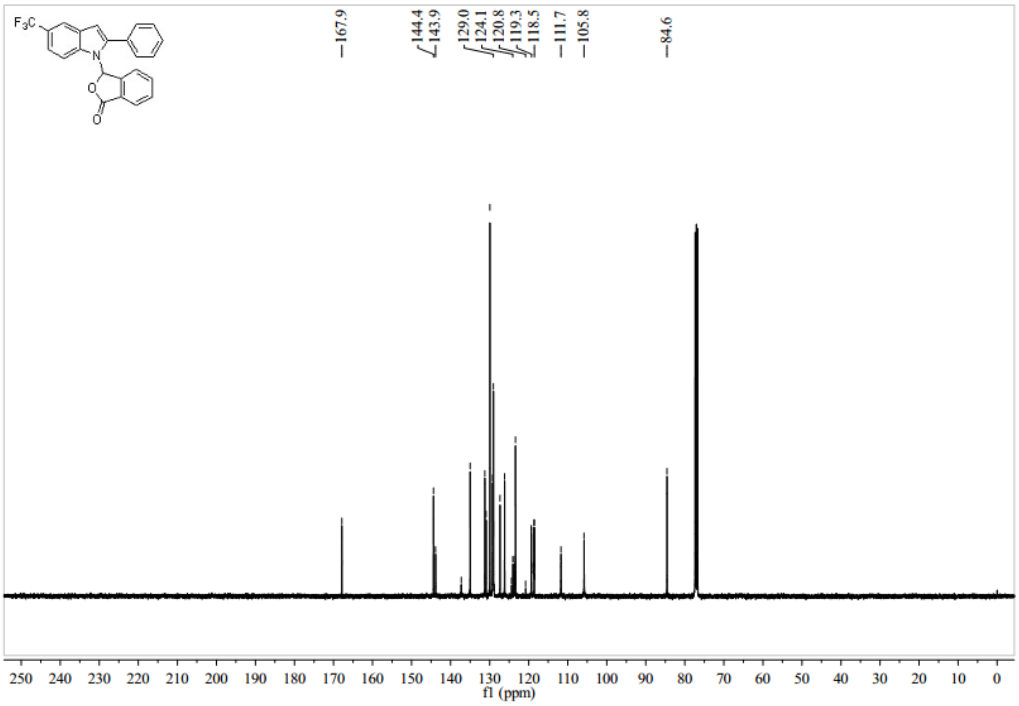
^{13}C NMR spectrum of compound 37



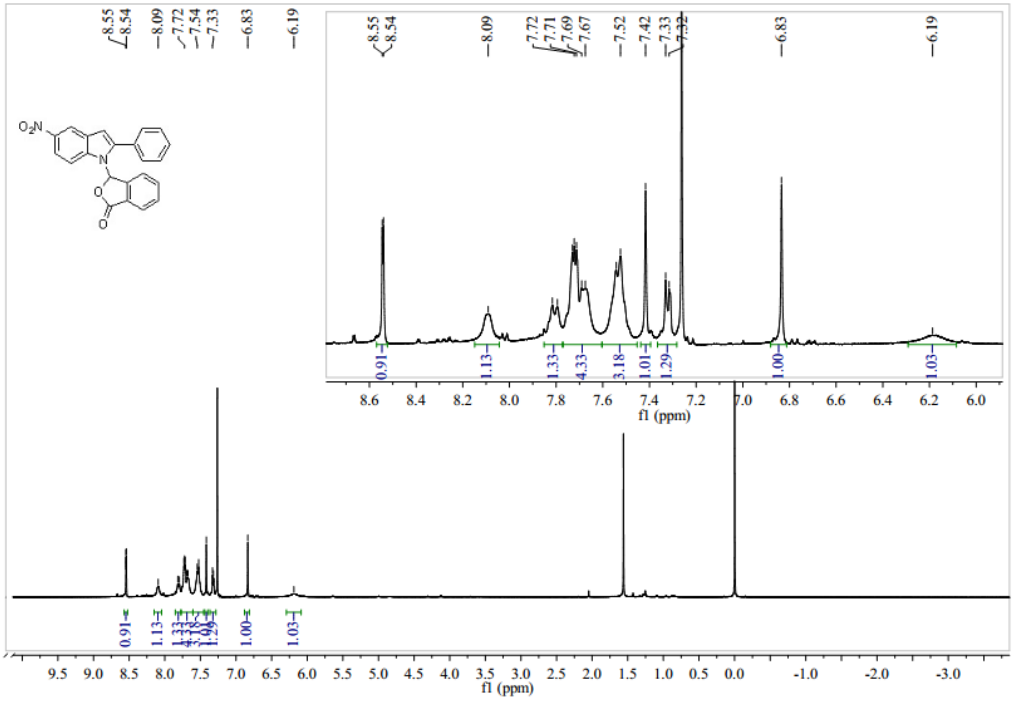
^{1}H NMR spectrum of compound 38



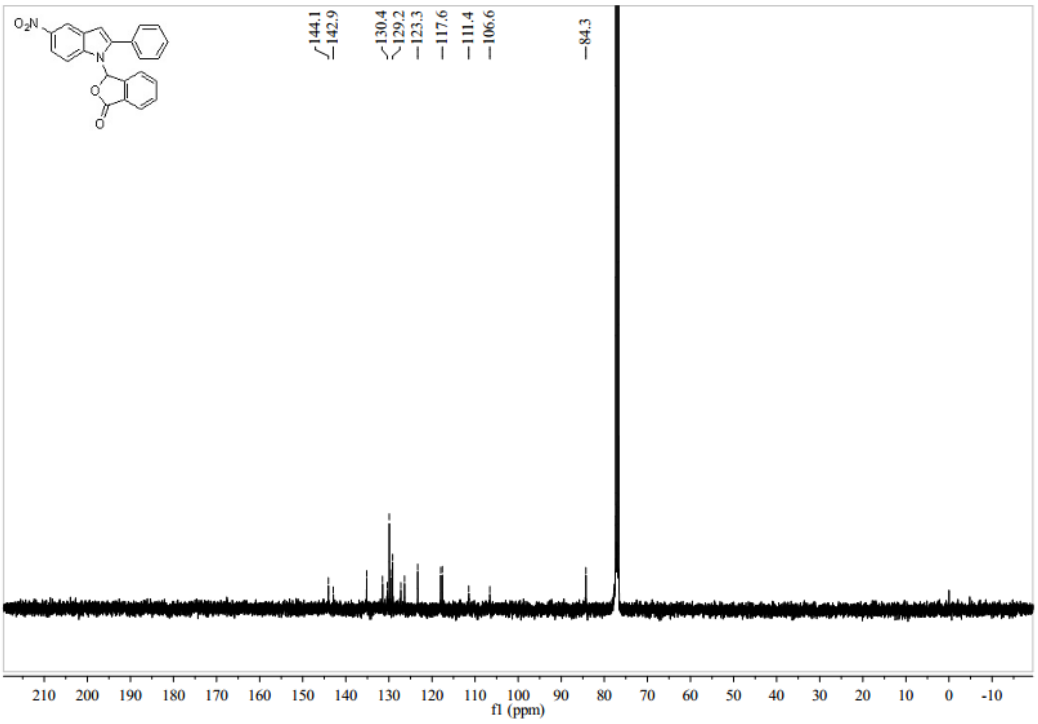
^{13}C NMR spectrum of compound 38



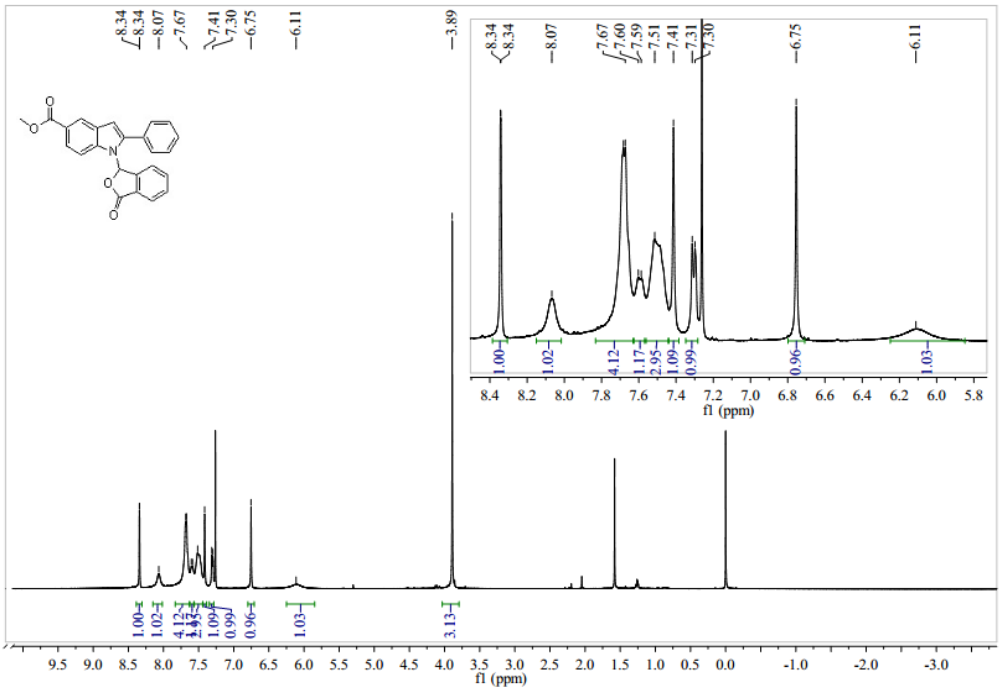
^{1}H NMR spectrum of compound 39



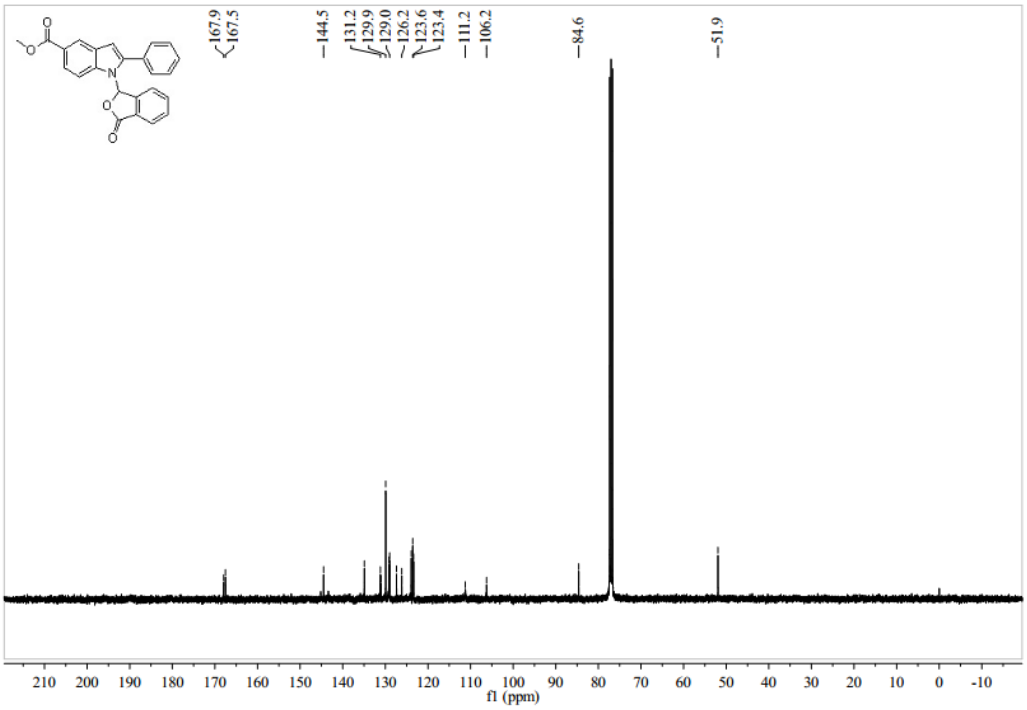
^{13}C NMR spectrum of compound 39



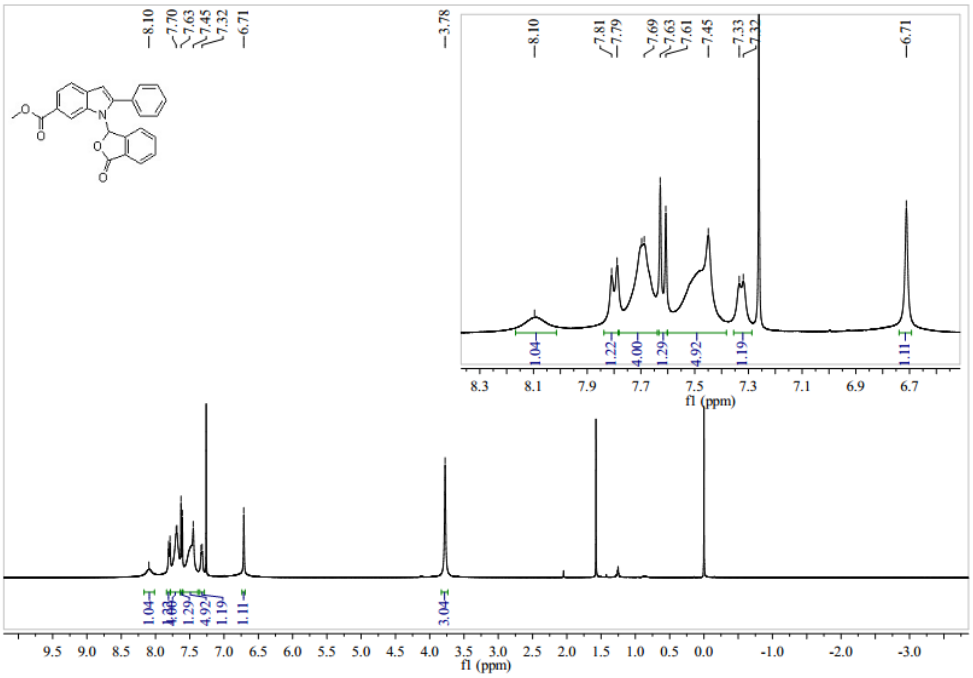
^{1}H NMR spectrum of compound 40



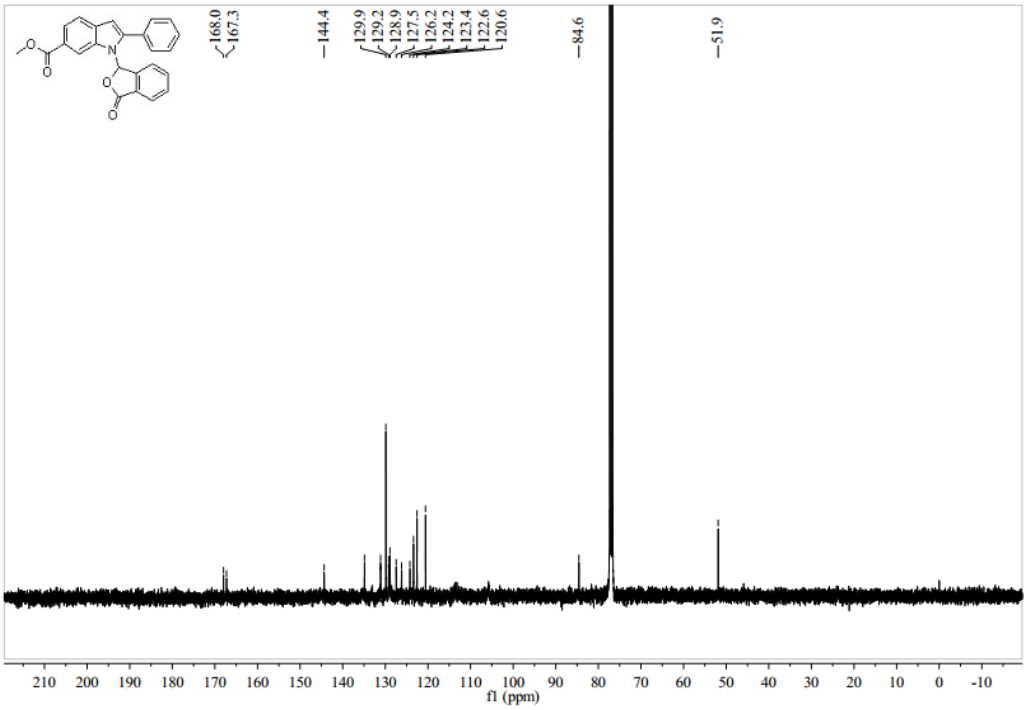
^{13}C NMR spectrum of compound 40



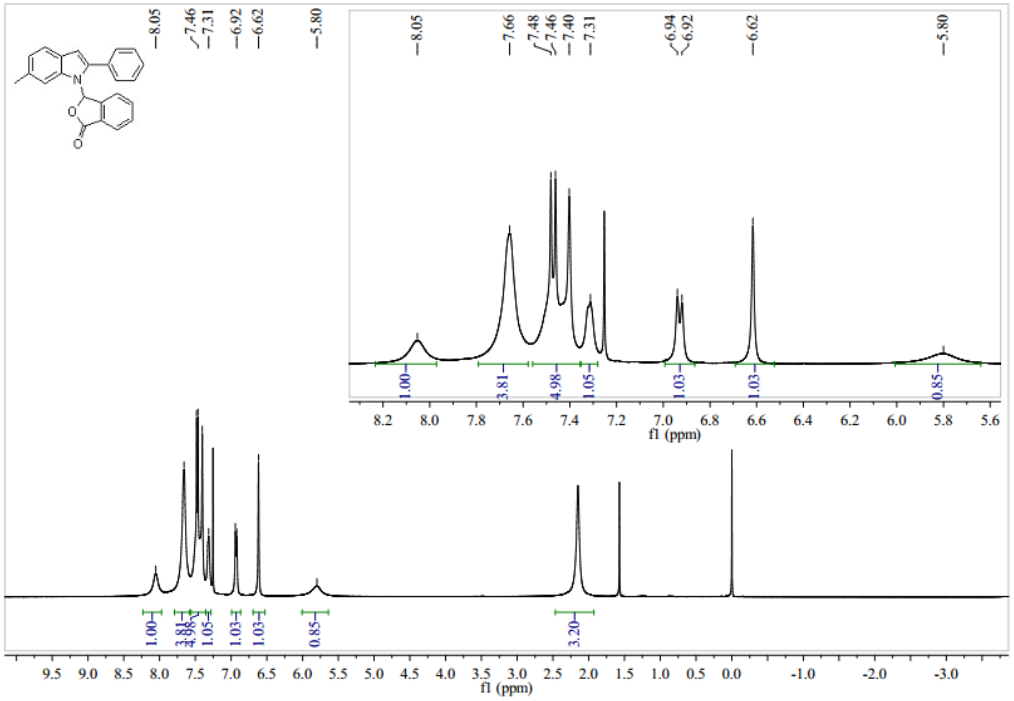
^{1}H NMR spectrum of compound 41



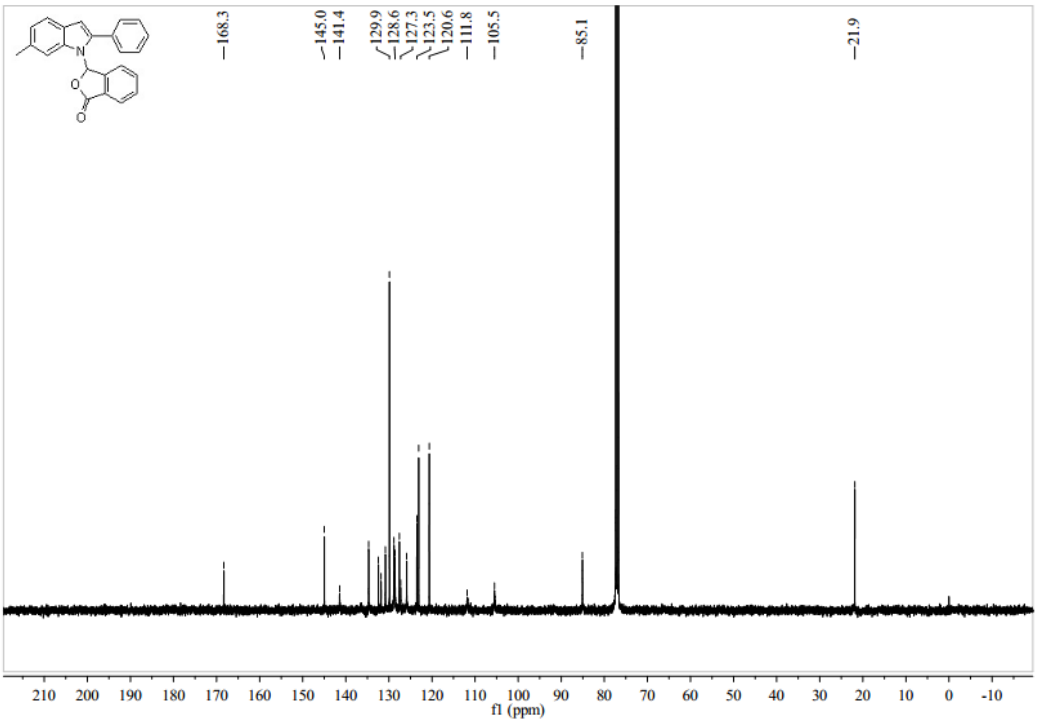
^{13}C NMR spectrum of compound 41



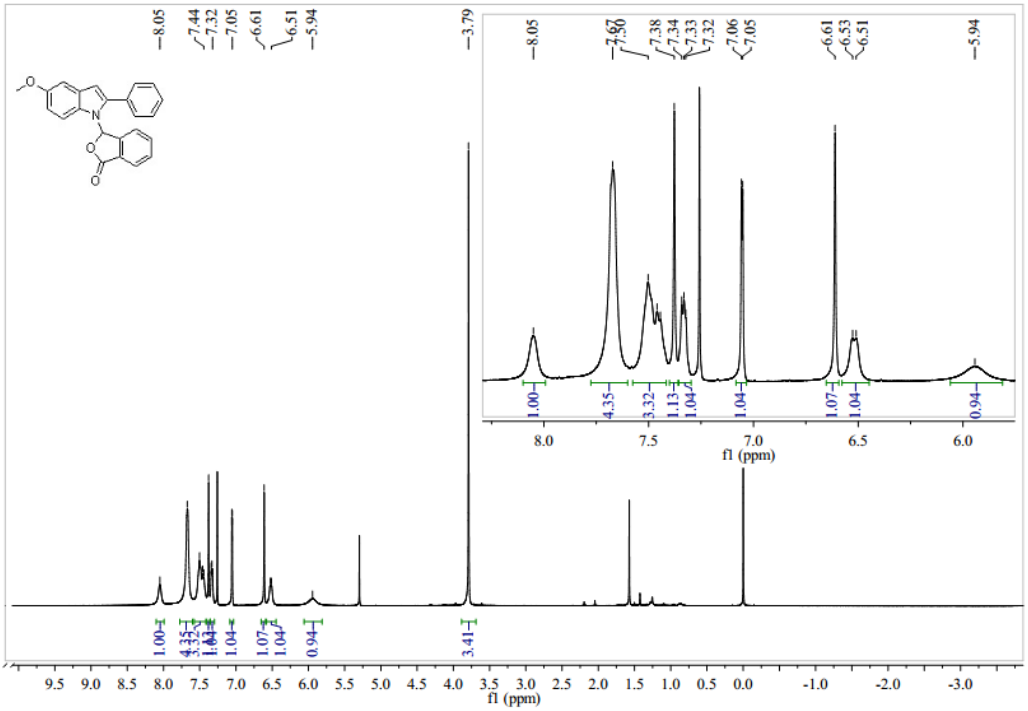
^{1}H NMR spectrum of compound 42



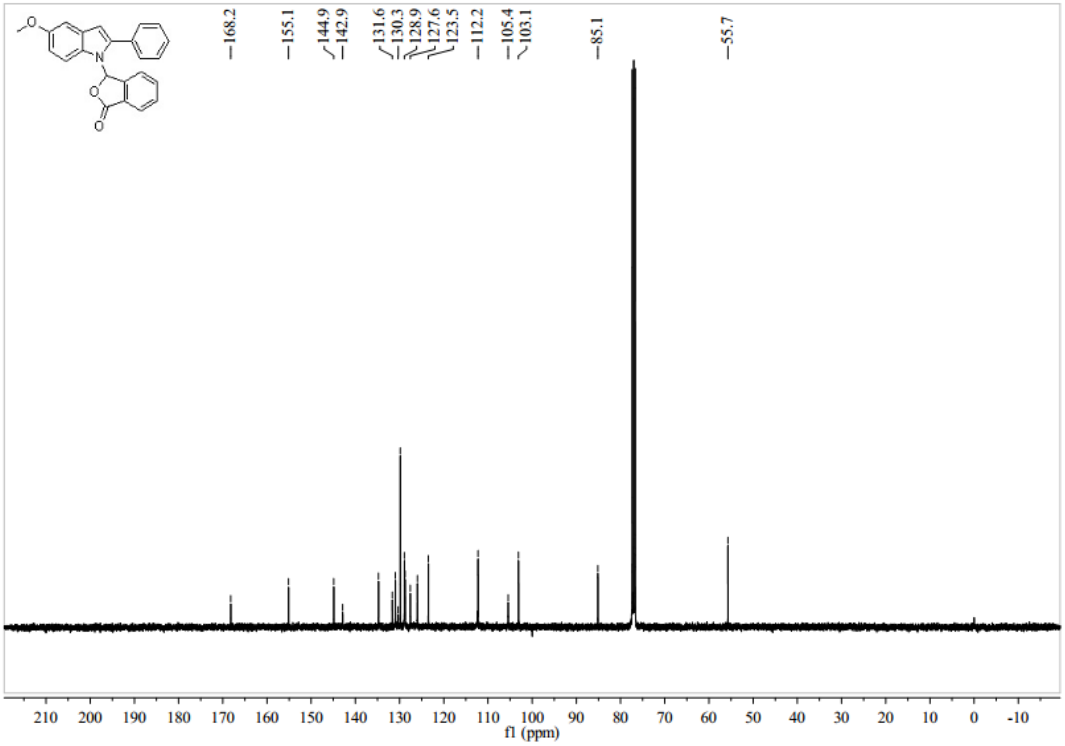
^{13}C NMR spectrum of compound 42



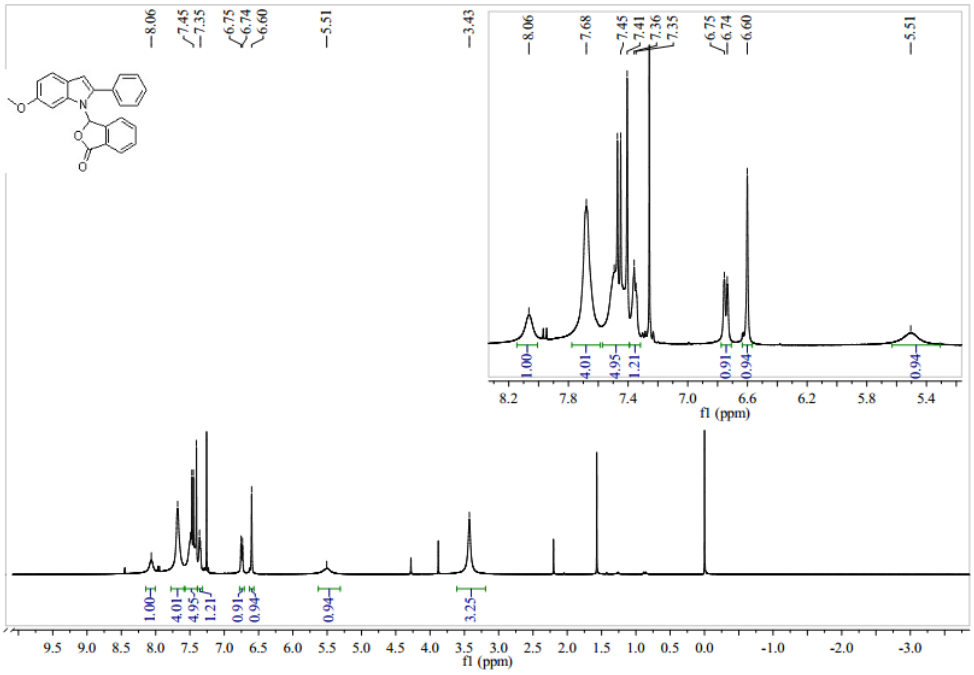
^{1}H NMR spectrum of compound 43



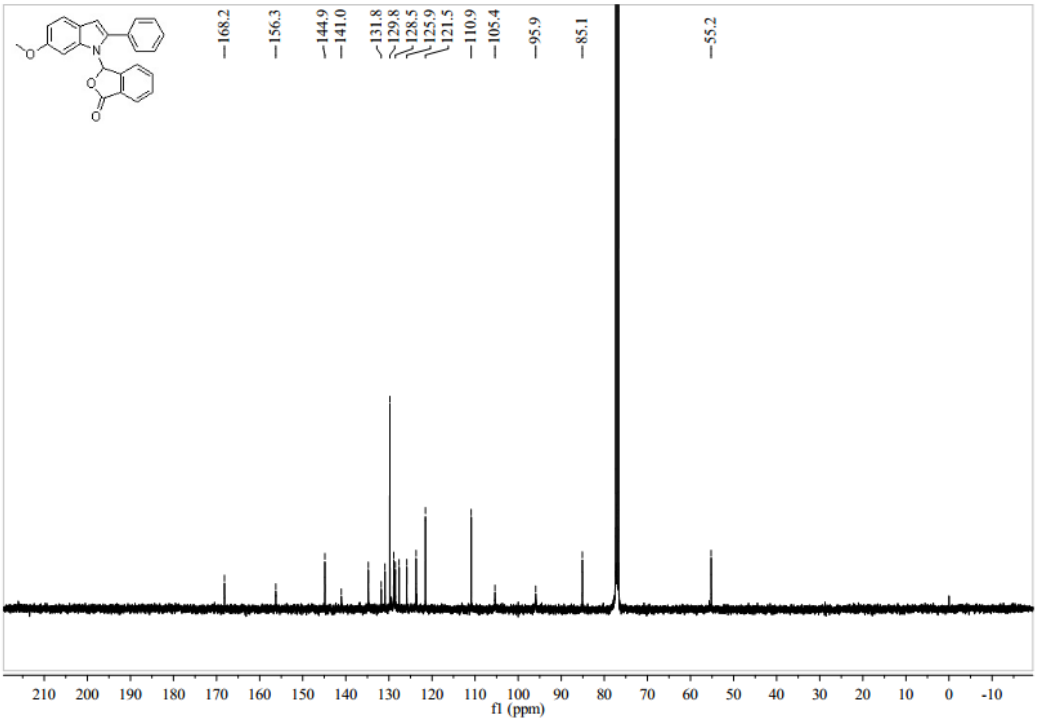
^{13}C NMR spectrum of compound 43



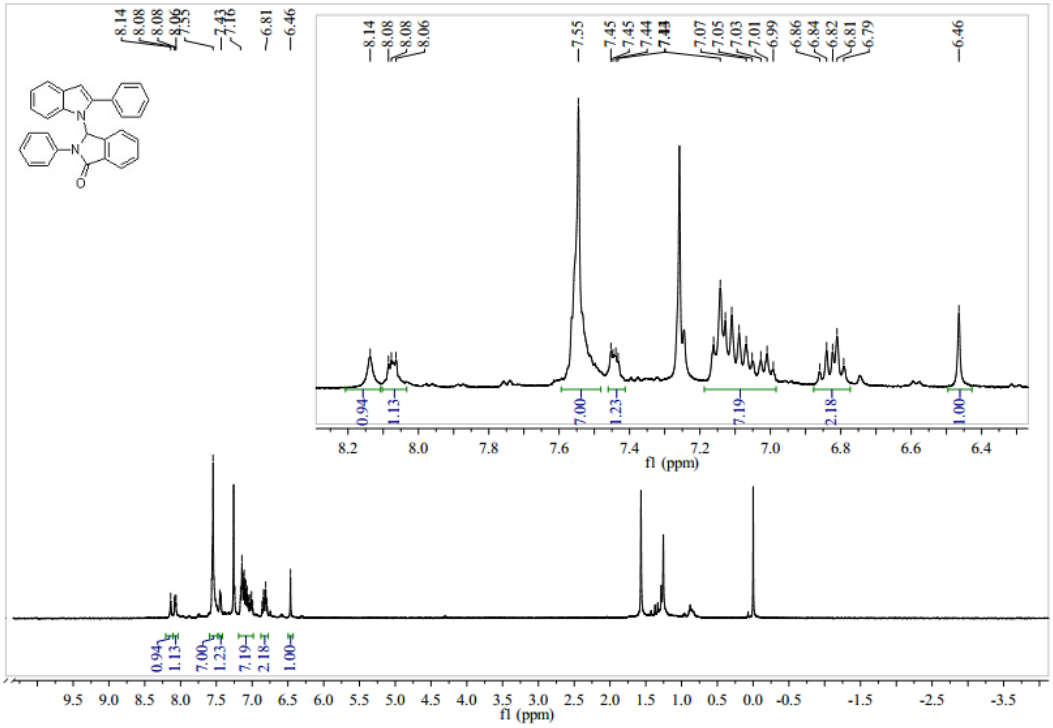
^{1}H NMR spectrum of compound 44



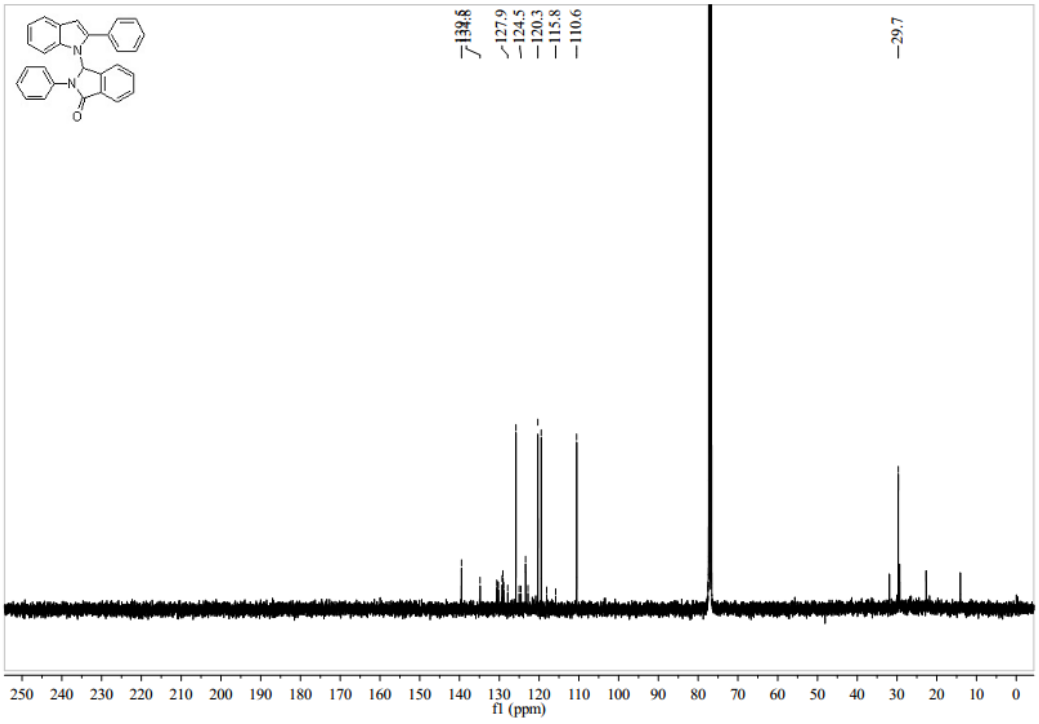
^{13}C NMR spectrum of compound 44



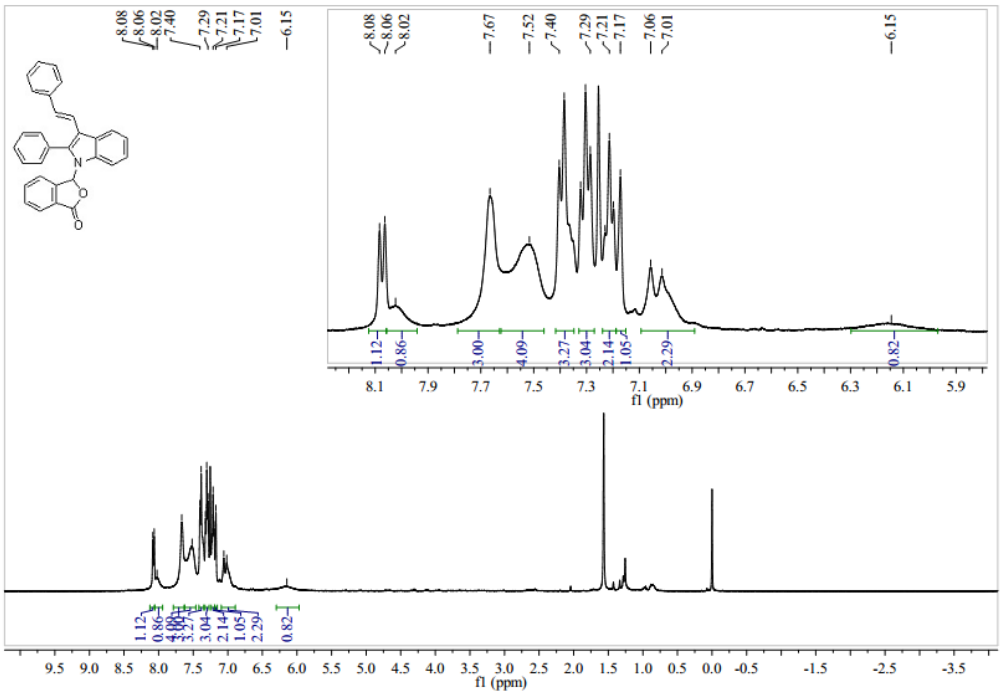
^{1}H NMR spectrum of compound 45



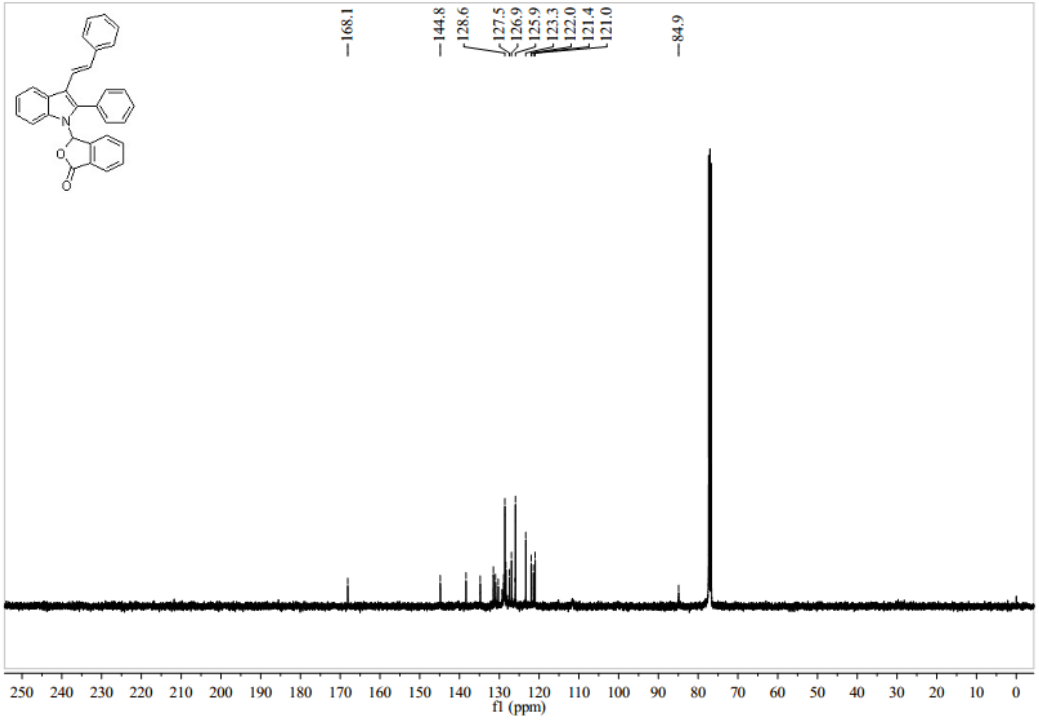
^{13}C NMR spectrum of compound 45



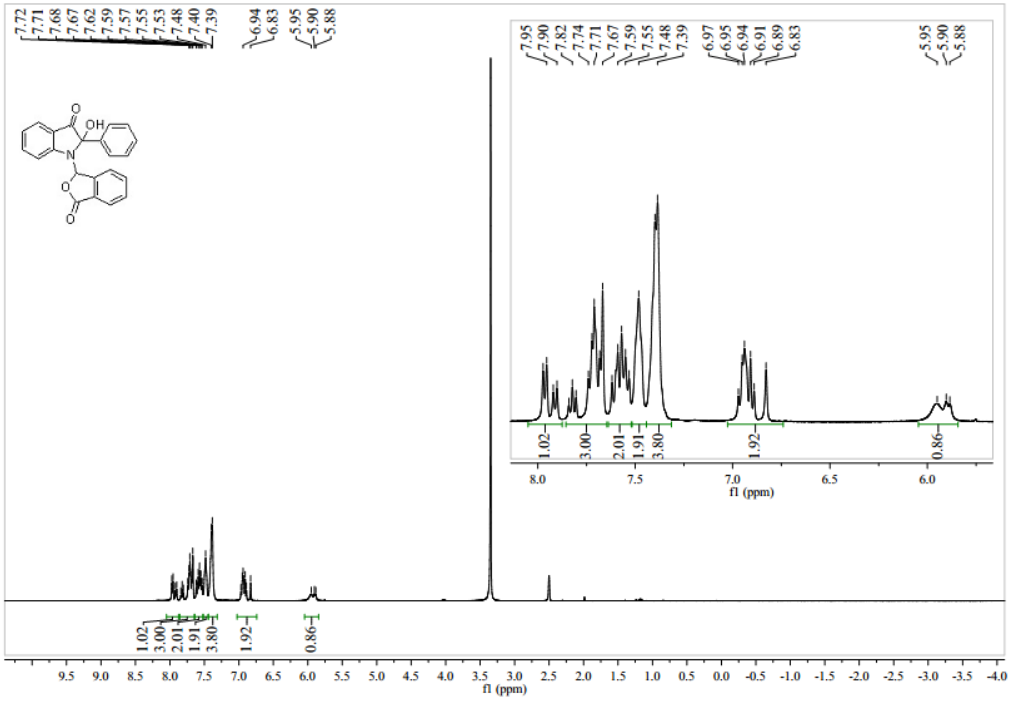
^{1}H NMR spectrum of compound 46



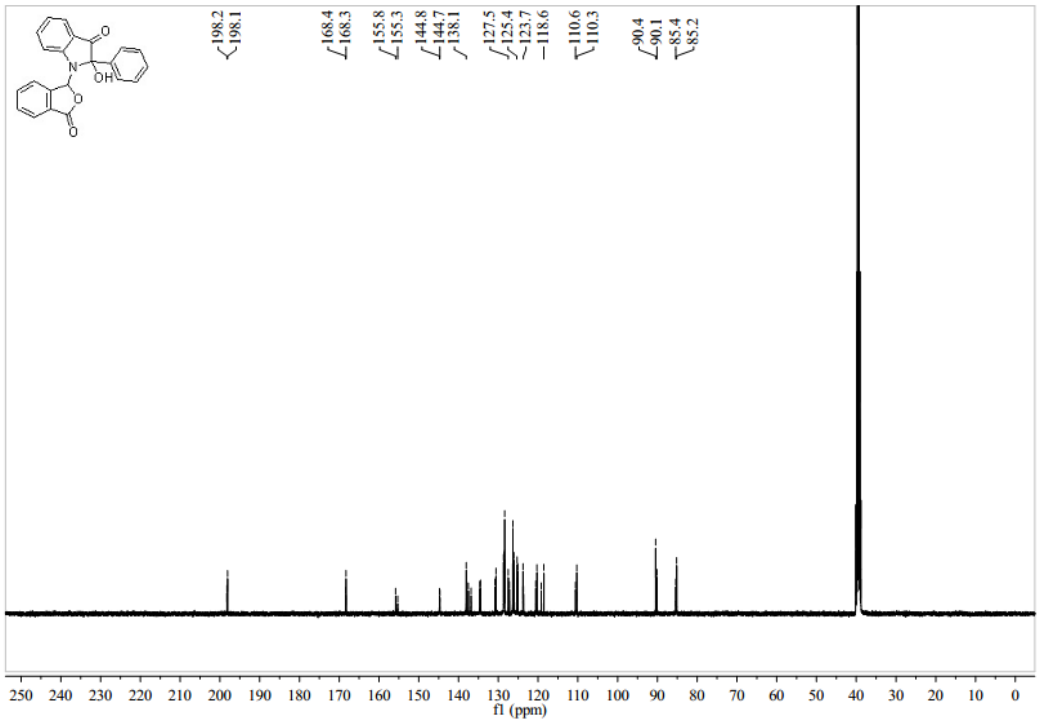
^{13}C NMR spectrum of compound 46



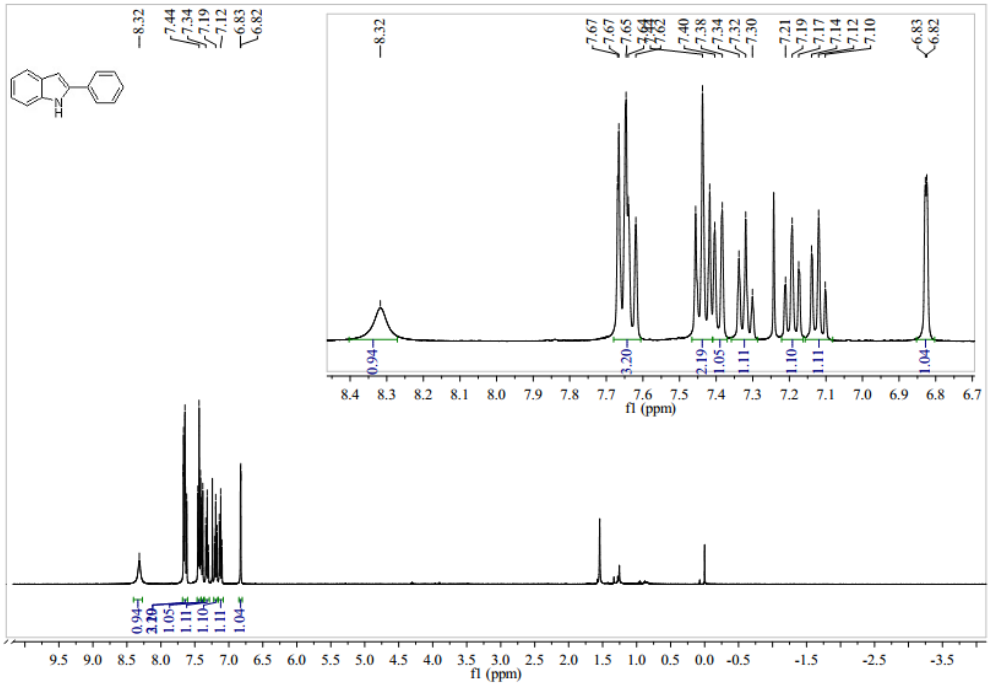
^{1}H NMR spectrum of compound 47



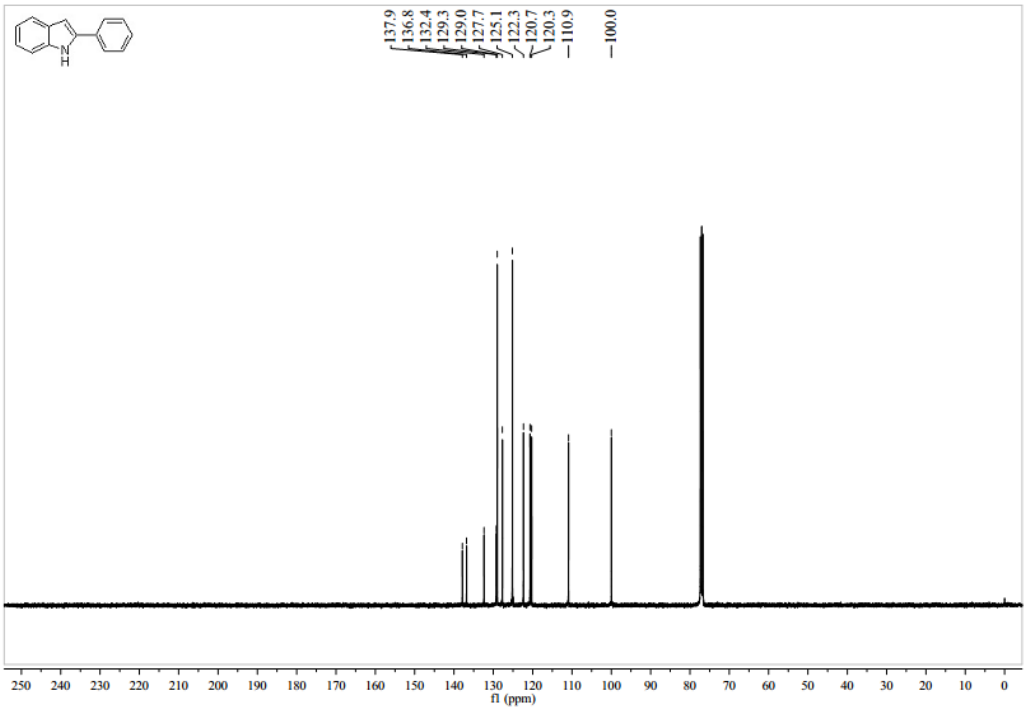
^{13}C NMR spectrum of compound 47



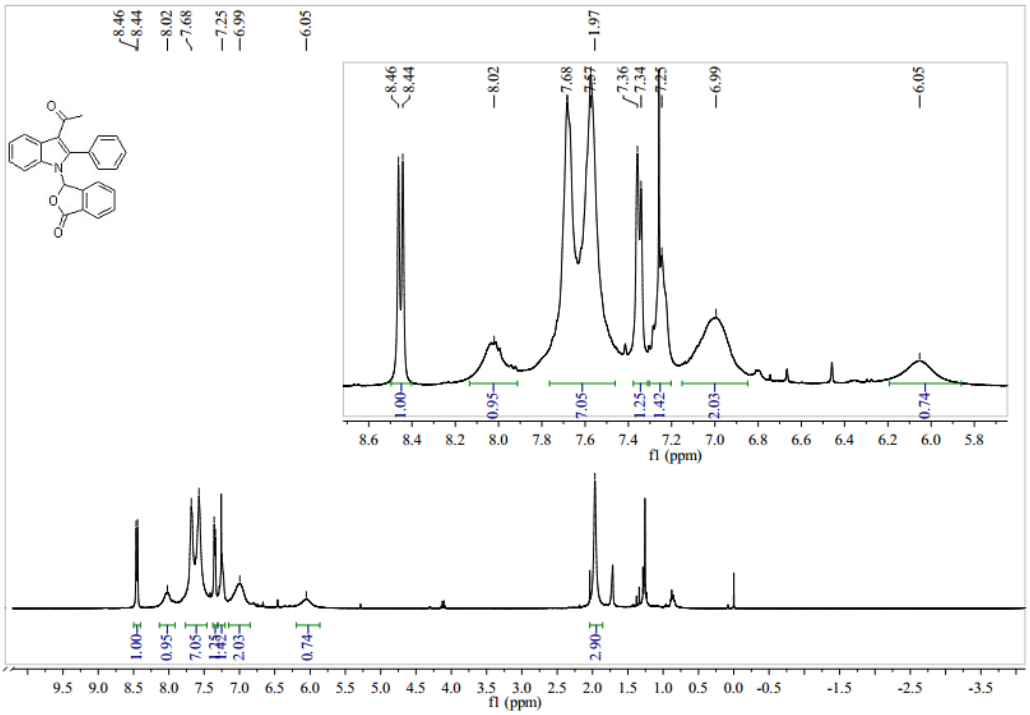
^{1}H NMR spectrum of compound 48



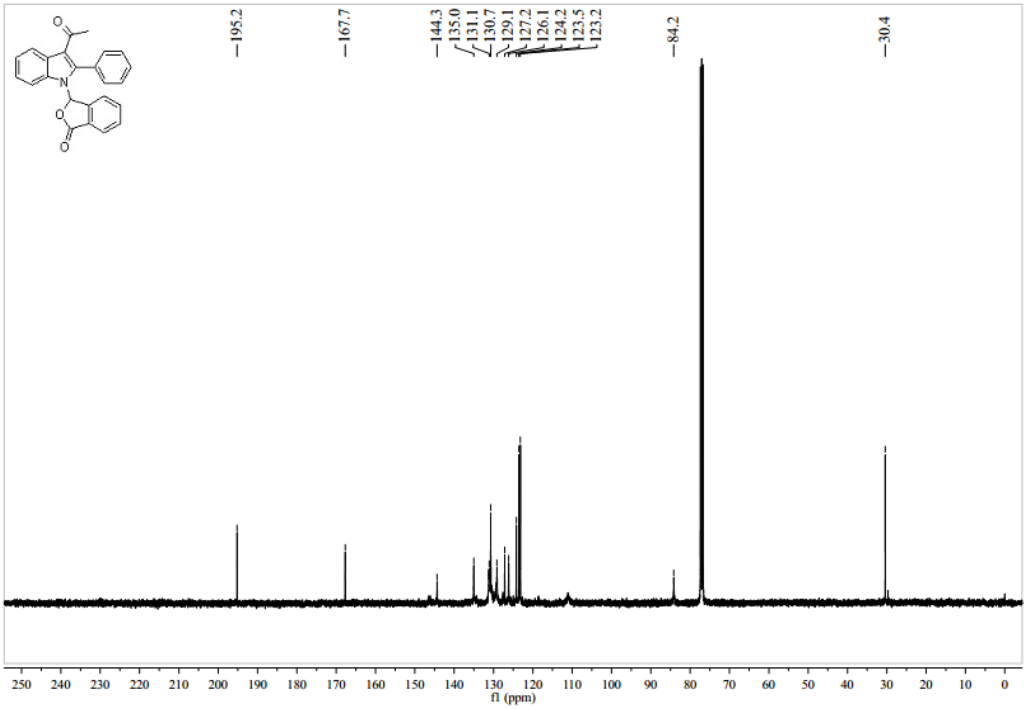
^{13}C NMR spectrum of compound 48



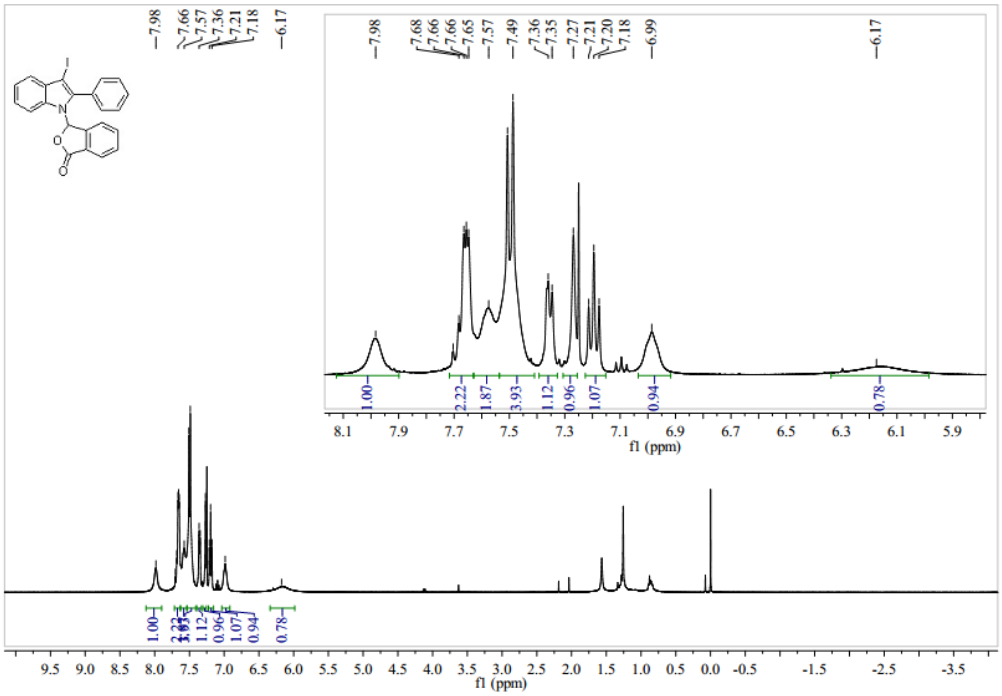
^{1}H NMR spectrum of compound 49



^{13}C NMR spectrum of compound 49



^{1}H NMR spectrum of compound 50



^{13}C NMR spectrum of compound 50