Supporting Information

Palladium-Catalyzed 1,1-Aminoxylation of 3-Butenoic Acid with 2-Alkynylanilines

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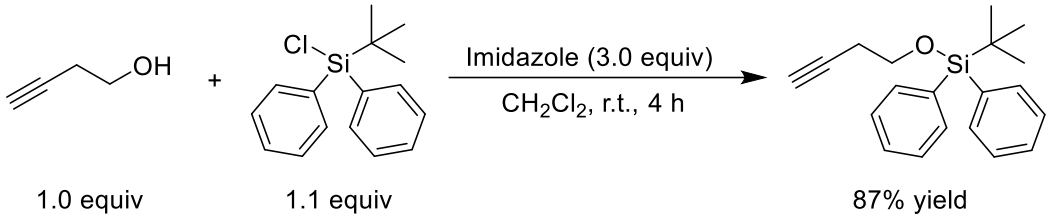
1. General information

NMR spectra were recorded on a Bruker Avance III 400 spectrometer or Bruker Ascend 400 spectrometer. The chemical shifts are referenced to TMS (δ 0.00 ppm) for ^{1}H NMR, and to $CDCl_3$ (δ 77.0 ppm) for ^{13}C NMR. Chemical shifts (δ) are reported in ppm and quoted to the nearest 0.01 ppm. Data are reported as follows: Chemical shift (multiplicity, coupling constants, number of protons). Coupling constants were quoted to the nearest 0.1 Hz and multiplicity reported according to the following convention: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, dt = doublet of triplet. br s = broad singlet. GC-MS analyses were performed on a Thermo Scientific ISQ gas chromatograph-mass spectrometer at an ionization voltage of 70 eV and equipped with a DB-WAX capillary column (internal diameter: 0.25 mm, length: 30 m). IR spectra were recorded on a Bruker Tensor 27 spectrometer and are reported in wavenumbers (cm^{-1}). High Resolution Mass Spectra (HRMS) were obtained from the high-resolution mass spectrometer (LCMS-ESI-TOF or LCMS-APCI-FTMS). Melting points were determined with a Büchi Melting Point B-545 instrument. All reagents were commercially purchased and used without further purification unless mentioned.

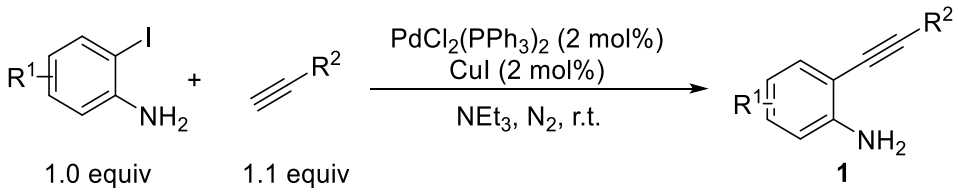
2. Experimental section

2.1 Synthesis of *N*-(2-((*tert*-butyldimethylsilyl)oxy)ethyl)aniline[1]



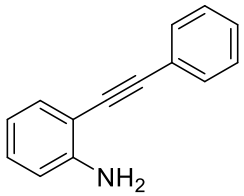
To a solution of but-3-yn-1-ol (376 μL, 5.0 mmol, 1.0 equiv) and imidazole (1.02 g, 15 mmol, 3.0 equiv) in CH_2Cl_2 (15 mL) was added TBDPSCl (1.43 mL, 11 mmol, 1.1 equiv) at room temperature. The reaction was stirred at room temperature for 4 h. After that, the reaction mixture was quenched with water and extracted with CH_2Cl_2. The combined organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated in vacuo to give a crude product, which was purified by silica gel column chromatography (100% hexane) to afford the product in 87% yield (1.34 g) as a colorless oil. NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[2] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.70-7.66 (m, 4H), 7.43-7.36 (m, 6H), 3.78 (t, J = 6.8 Hz, 2H), 2.45 (td, J = 7.2, 2.8 Hz, 2H), 1.94 (t, J = 2.8 Hz, 1H), 1.06 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 135.6, 133.5, 129.7, 127.7, 81.5, 69.3, 62.3, 26.8, 22.6, 19.2.

2.2 General procedure for synthesis of 2-alkynylanilines 1



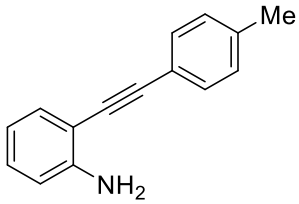
Following a reported procedure,[3] a solution of the corresponding 2-iodoaniline (3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and terminal alkyne (3.3 mmol, 1.1 equiv) in NEt_3 (12 mL) was stirred under nitrogen atmosphere at room temperature for 4 h. The mixture was filtered by short silica, then the solvent was evaporated under reduced pressure and the residue was purified by flash chromatography on silica gel with EtOAc and petroleum ether to give 2-alkynylanilines.

2-(phenylethynyl)aniline (1a)



2-(phenylethynyl)aniline (**1a**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (337.0 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (550.7 mg, 95% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[4] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.56-7.49 (m, 2H), 7.39-7.31 (m, 4H), 7.14 (td, J = 7.6, 1.6 Hz, 1H), 6.72 (t, J = 7.6 Hz, 2H), 4.13 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.7, 132.1, 131.5, 129.7, 128.4, 128.2, 123.3, 118.0, 114.4, 108.0, 94.7, 85.8.

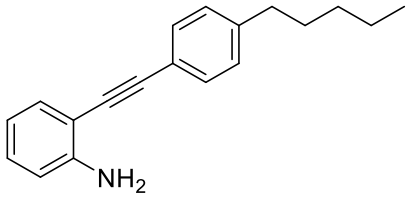
2-(*p*-tolylethynyl)aniline (1b)



2-(*p*-tolylethynyl)aniline (**1b**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-4-methylbenzene (383.3 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (590.7 mg, 95% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[4] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.42 (d, J

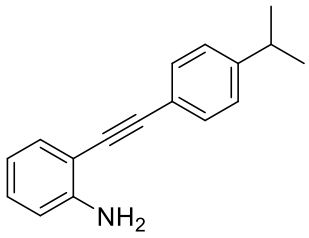
= 8.0 Hz, 2H), 7.35 (d, *J* = 7.6 Hz, 1H), 7.13 (dd, *J* = 13.6, 7.6 Hz, 3H), 6.71 (t, *J* = 7.6 Hz, 2H), 4.21 (s, 2H), 2.36 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.2, 138.3, 132.1, 131.3, 129.5, 129.1, 120.2, 118.0, 114.4, 108.3, 94.9, 85.1, 21.5.

2-((4-pentylphenyl)ethynyl)aniline (**1c**)



2-((4-pentylphenyl)ethynyl)aniline (**1c**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-4-pentylbenzene (568.5 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (734.8 mg, 93% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[5] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.43 (d, *J* = 8.0 Hz, 2H), 7.35 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.17-7.09 (m, 3H), 6.75-6.68 (m, 2H), 4.23 (s, 2H), 2.60 (t, *J* = 7.7 Hz, 2H), 1.61 (p, *J* = 7.4 Hz, 2H), 1.35-1.28 (m, 4H), 0.89 (t, *J* = 6.4 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.6, 143.4, 132.1, 131.34, 129.5, 128.5, 120.4, 118.0, 114.3, 108.2, 94.9, 85.1, 35.8, 31.4, 30.9, 22.5, 14.0.

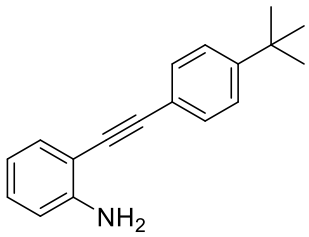
2-((4-isopropylphenyl)ethynyl)aniline (**1d**)



2-((4-isopropylphenyl)ethynyl)aniline (**1d**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-4-isopropylbenzene (475.9 mg, 3.3

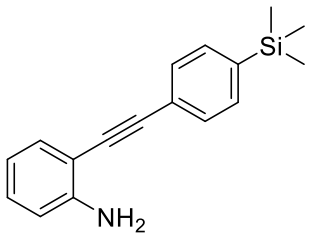
mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (642.5 mg, 91% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[6] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.48-7.42 (m, 2H), 7.35 (dd, J = 8.4, 2.0 Hz, 1H), 7.23-7.18 (m, 2H), 7.12 (td, J = 8.0, 1.6 Hz, 1H), 6.74-6.68 (m, 2H), 4.25 (s, 2H), 2.96-2.86 (m, 1H), 1.25 (d, J = 6.8 Hz, 6H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 149.3, 147.7, 132.1, 131.5, 129.5, 126.5, 120.6, 117.9, 114.3, 108.2, 94.9, 85.1, 34.1, 23.8.

2-((4-(*tert*-butyl)phenyl)ethynyl)aniline (1e)



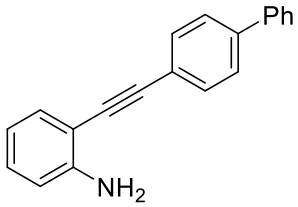
2-((4-(*tert*-butyl)phenyl)ethynyl)aniline (**1e**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-(*tert*-butyl)-4-ethynylbenzene (522.2 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (673.3 mg, 90% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[7] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.46 (d, J = 8.0 Hz, 2H), 7.35 (d, J = 8.4 Hz, 3H), 7.10 (td, J = 7.6, 1.6 Hz, 1H), 6.69 (t, J = 7.2 Hz, 2H), 4.16 (s, 2H), 1.31 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 151.6, 147.8, 132.2, 131.3, 129.6, 125.5, 120.4, 118.0, 114.4, 108.2, 94.9, 85.3, 34.9, 31.3.

2-((4-(trimethylsilyl)phenyl)ethynyl)aniline (1f)



2-((4-(trimethylsilyl)phenyl)ethynyl)aniline (**1f**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and (4-ethynylphenyl)trimethylsilane (575.2 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (700.7 mg, 88% yield). **m.p.** = 67.1-67.6 ℃. **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.50 (s, 4H), 7.37 (dd, J = 8.4, 1.6 Hz, 1H), 7.14 (td, J = 7.6, 1.6 Hz, 1H), 6.72 (t, J = 7.6 Hz, 2H), 4.27 (s, 2H), 0.28 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.8, 141.0, 133.2, 132.1, 130.5, 129.7, 123.5, 118.0, 114.3, 108.0, 94.9, 86.2, -1.3. **IR ν_{max}(KBr)/cm^{-1}** 2934, 2855, 1624, 1454, 1318, 1106, 749. **HRMS-APCI (m/z):** calcd for $C_{17}H_{20}NSi$, $[M+H]^+$: 266.1361, found 266.1360.

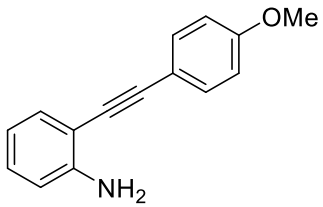
2-([1,1'-biphenyl]-4-ylethynyl)aniline (**1g**)



2-([1,1'-biphenyl]-4-ylethynyl)aniline (**1g**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 4-ethynyl-1,1'-biphenyl (588.2 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:40 v/v) as an eluent provided the titled compound as a yellow solid (686.8 mg, 85% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[8] **^{1}H NMR (400 MHz,**

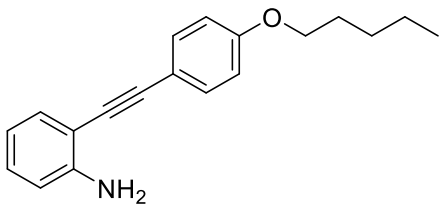
$CDCl_3$) δ 7.63-7.57 (m, 6H), 7.48-7.43 (m, 2H), 7.40-7.34 (m, 2H), 7.15 (ddd, J = 8.8, 7.2, 1.6 Hz, 1H), 6.78-6.72 (m, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.4, 140.9, 140.3, 132.2, 131.9, 129.7, 128.9, 127.6, 127.0, 126.9, 122.2, 118.2, 114.5, 108.2, 94.7, 86.5.

2-((4-methoxyphenyl)ethynyl)aniline (**1h**)



2-((4-methoxyphenyl)ethynyl)aniline (**1h**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-4-methoxybenzene (436.1 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:20 v/v) as an eluent provided the titled compound as a yellow solid (609.6 mg, 91% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[9] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.45 (d, J = 8.8 Hz, 2H), 7.34 (dd, J = 8.0, 1.6 Hz, 1H), 7.10 (td, J = 8.0, 1.6 Hz, 1H), 6.88-6.83 (m, 2H), 6.72-6.67 (m, 2H), 4.02 (s, 2H), 3.79 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 159.5, 147.6, 132.8, 131.9, 129.3, 117.9, 115.3, 114.2, 114.0, 108.2, 94.6, 84.4, 55.2.

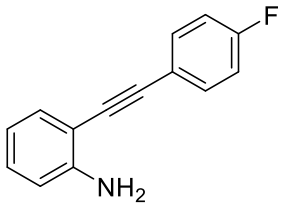
2-((4-(pentyloxy)phenyl)ethynyl)aniline (**1i**)



2-((4-(pentyloxy)phenyl)ethynyl)aniline (**1i**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg,

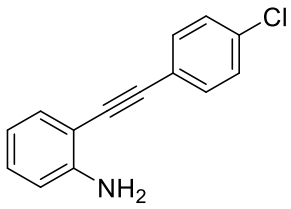
2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-4-(pentyloxy)benzene (621.3 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:20 v/v) as an eluent provided the titled compound as a yellow solid (771.1 mg, 92% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[10] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.44 (d, J = 8.4 Hz, 2H), 7.38-7.30 (m, 1H), 7.16-7.06 (m, 1H), 6.85 (d, J = 8.4 Hz, 2H), 6.72-6.68 (m, 2H), 4.25 (s, 2H), 3.95 (t, J = 6.4 Hz, 2H), 1.83-1.73 (m, 2H), 1.46-1.35 (m, 4H), 0.93 (t, J = 6.8 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 159.1, 147.6, 132.8, 131.9, 129.3, 117.9, 115.1, 114.5, 114.2, 108.3, 94.7, 84.3, 68.0, 28.8, 28.1, 22.4, 14.0.

2-((4-fluorophenyl)ethynyl)aniline (1j)



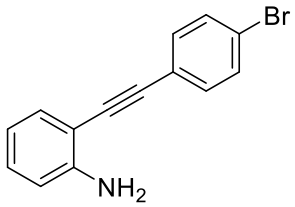
2-((4-fluorophenyl)ethynyl)aniline (**1j**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-4-fluorobenzene (396.4 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:40 v/v) as an eluent provided the titled compound as a yellow solid (608.3 mg, 96% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[7] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.53-7.47 (m, 2H), 7.37-7.33 (m, 1H), 7.14 (td, J = 7.6, 1.6 Hz, 1H), 7.07-7.00 (m, 2H), 6.75-6.70 (m, 2H), 3.95 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 162.5 (d, J = 249.0 Hz), 147.6, 133.3 (d, J = 8.0 Hz), 132.1, 129.8, 119.4 (d, J = 4.0 Hz), 118.1, 115.7 (d, J = 21.0 Hz), 114.4, 107.8, 93.6, 85.5.

2-((4-chlorophenyl)ethynyl)aniline (1k)



2-((4-chlorophenyl)ethynyl)aniline (**1k**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-chloro-4-ethynylbenzene (450.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:40 v/v) as an eluent provided the titled compound as a yellow solid (560.1 mg, 82% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[11] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.47-7.41 (m, 2H), 7.37-7.28 (m, 3H), 7.14 (td, J = 8.0, 1.6 Hz, 1H), 6.75-6.68 (m, 2H), 4.25 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.8, 134.1, 132.6, 132.1, 129.9, 128.7, 121.8, 118.0, 114.4, 107.5, 93.5, 86.9.

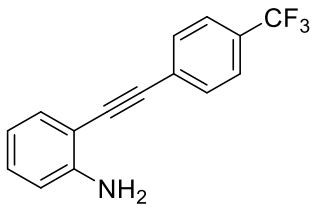
2-((4-bromophenyl)ethynyl)aniline (1l)



2-((4-bromophenyl)ethynyl)aniline (**1l**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-bromo-4-ethynylbenzene (597.4 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:30 v/v) as an eluent provided the titled compound as a yellow solid (694.0 mg, 85% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[7] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.50-7.45

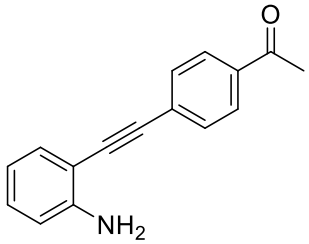
(m, 2H), 7.40-7.33 (m, 3H), 7.15 (td, J = 7.6, 1.6 Hz, 1H), 6.75-6.69 (m, 2H), 4.25 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.8, 132.8, 132.2, 131.6, 130.0, 122.4, 122.3, 118.1, 114.4, 107.5, 93.6, 87.1.

2-((4-(trifluoromethyl)phenyl)ethynyl)aniline (1m)



2-((4-(trifluoromethyl)phenyl)ethynyl)aniline (**1m**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-4-(trifluoromethyl)benzene (561.4 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:8 v/v) as an eluent provided the titled compound as a yellow solid (587.8 mg, 75% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[12] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.59 (s, 4H), 7.37 (dd, J = 8.0, 1.6 Hz, 1H), 7.16 (td, J = 8.0, 1.6 Hz, 1H), 6.72 (t, J = 7.6 Hz, 2H), 4.27 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 148.0, 132.3, 131.6, 130.3, 129.7 (q, J = 32.7 Hz), 127.1 (q, J = 1.6 Hz), 125.3 (q, J = 3.8 Hz), 118.0, 114.4, 107.1, 93.3, 88.4.

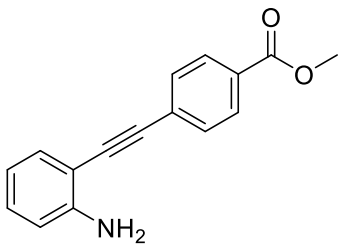
1-(4-((2-aminophenyl)ethynyl)phenyl)ethan-1-one (1n)



1-(4-((2-aminophenyl)ethynyl)phenyl)ethan-1-one (**1n**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$

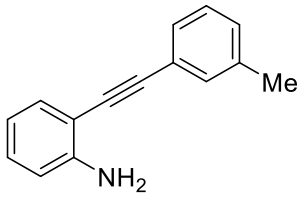
(42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-(4-ethynylphenyl)ethan-1-one (475.8 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:5 v/v) as an eluent provided the titled compound as a yellow solid (564.7 mg, 80% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[13] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.93 (d, J = 8.0 Hz, 2H), 7.59 (d, J = 8.0 Hz, 2H), 7.37 (d, J = 7.6 Hz, 1H), 7.17 (t, J = 7.6 Hz, 1H), 6.74 (t, J = 8.0 Hz, 2H), 4.02 (s, 2H), 2.61 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 197.2, 147.9, 136.1, 132.3, 131.4, 130.3, 128.3, 128.2, 118.1, 114.5, 107.2, 94.0, 89.4, 26.6.

methyl 4-((2-aminophenyl)ethynyl)benzoate (1o)



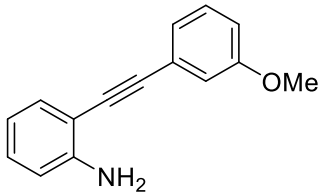
methyl 4-((2-aminophenyl)ethynyl)benzoate (**1o**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and methyl 4-ethynylbenzoate (528.6 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:5 v/v) as an eluent provided the titled compound as a yellow solid (655.7 mg, 87% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[11] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.05-7.99 (m, 2H), 7.57 (d, J = 8.4 Hz, 2H), 7.37 (dd, J = 8.1, 1.6 Hz, 1H), 7.17 (td, J = 7.7, 1.6 Hz, 1H), 6.76-6.70 (m, 2H), 4.40-4.23 (m, 2H), 3.93 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 166.5, 147.9, 132.3, 131.3, 130.3, 129.5, 129.3, 128.0, 118.0, 114.4, 107.2, 94.0, 89.0, 52.2.

2-(*m*-tolylethynyl)aniline (1p)



2-(*m*-tolylethynyl)aniline (**1p**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-3-methylbenzene (383.3 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (590.7 mg, 95% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[7] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.41-7.30 (m, 3H), 7.23 (t, J = 7.6 Hz, 1H), 7.17-7.10 (m, 2H), 6.72 (d, J = 7.6 Hz, 2H), 4.26 (s, 2H), 2.35 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.7, 138.0, 132.1, 132.0, 129.6, 129.1, 128.5, 128.3, 123.1, 117.9, 114.3, 108.0, 94.9, 85.5, 21.2.

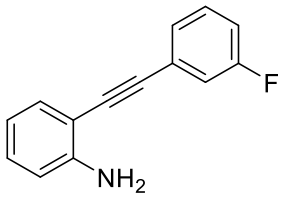
2-(*m*-tolylethynyl)aniline (1q)



2-(*m*-tolylethynyl)aniline (**1q**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-3-methoxybenzene (436.1 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:10 v/v) as an eluent provided the titled compound as a yellow solid (596.2 mg, 89% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[14] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.36 (dd, J = 8.0, 1.6 Hz, 1H), 7.27-7.23 (m, 1H), 7.19-7.09 (m, 2H), 7.05 (dd, J = 2.4, 1.2 Hz,

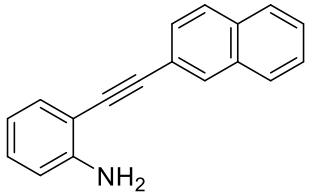
1H), 6.89 (ddd, J = 8.4, 2.6, 1.0 Hz, 1H), 6.79-6.66 (m, 2H), 3.82 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 159.3, 147.8, 132.2, 129.8, 129.4, 124.3, 124.0, 118.0, 116.2, 114.8, 114.3, 107.8, 94.6, 85.7, 55.3.

2-((3-fluorophenyl)ethynyl)aniline (1r)



2-((3-fluorophenyl)ethynyl)aniline (**1r**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 1-ethynyl-3-fluorobenzene (396.4 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:40 v/v) as an eluent provided the titled compound as a yellow solid (602.0 mg, 95% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[7] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.36 (dd, J = 7.6, 1.2 Hz, 1H), 7.32-7.29 (m, 2H), 7.25-7.19 (m, 1H), 7.16 (td, J = 7.6, 1.6 Hz, 1H), 7.08-7.00 (m, 1H), 6.73 (t, J = 7.6 Hz, 2H), 4.32 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 162.4 (d, J = 246.4 Hz), 147.9, 132.2, 130.1, 129.9 (d, J = 8.7 Hz), 127.3 (d, J = 3.0 Hz), 125.1 (d, J = 9.5 Hz), 118.3, 118.2 (d, J = 22.8 Hz), 115.5 (d, J = 21.2 Hz), 114.4, 107.3, 93.4 (d, J = 3.4 Hz), 86.9.

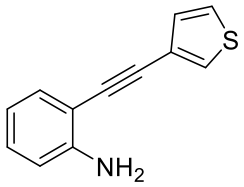
2-(naphthalen-2-ylethynyl)aniline (1s)



2-(naphthalen-2-ylethynyl)aniline (**1s**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 2-ethynylnaphthalene (502.2 mg, 3.3 mmol, 1.1 equiv) in

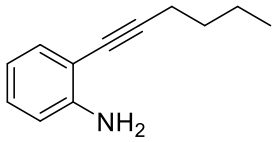
NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:40 v/v) as an eluent provided the titled compound as a yellow solid (605.8 mg, 83% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[15] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.04 (s, 1H), 7.80 (d, J = 8.0 Hz, 3H), 7.57 (d, J = 8.4 Hz, 1H), 7.52-7.46 (m, 2H), 7.41 (d, J = 7.6 Hz, 1H), 7.15 (t, J = 7.6 Hz, 1H), 6.76-6.72 (m, 2H), 4.32 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.8, 133.0, 132.7, 132.2, 131.1, 129.8, 128.3, 128.0, 127.8, 127.7, 126.6, 126.6, 120.6, 118.0, 114.4, 108.0, 95.1, 86.3.

2-(thiophen-3-ylethynyl)aniline (**1t**)



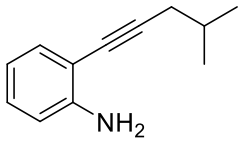
2-(thiophen-3-ylethynyl)aniline (**1t**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 3-ethynylthiophene (356.9 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:30 v/v) as an eluent provided the titled compound as a yellow solid (567.9 mg, 95% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[16] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.50 (d, J = 2.8 Hz, 1H), 7.34 (d, J = 7.6 Hz, 1H), 7.32-7.27 (m, 1H), 7.19 (d, J = 5.2 Hz, 1H), 7.13 (t, J = 8.0 Hz, 1H), 6.71 (d, J = 7.6 Hz, 2H), 4.25 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.7, 132.1, 129.8, 129.7, 128.3, 125.4, 122.3, 118.0, 114.3, 107.9, 89.6, 85.3.

2-(hex-1-yn-1-yl)aniline (**1u**)



2-(hex-1-yn-1-yl)aniline (**1u**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and hex-1-yne (271.1 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:60 v/v) as an eluent provided the titled compound as a yellow liquid (395.0 mg, 76% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[17] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.23 (dd, J = 7.6, 1.6 Hz, 1H), 7.06 (ddd, J = 8.0, 7.2, 1.6 Hz, 1H), 6.69-6.63 (m, 2H), 4.15 (s, 2H), 2.46 (t, J = 6.8 Hz, 2H), 1.64-1.55 (m, 2H), 1.53-1.43 (m, 2H), 0.95 (t, J = 7.6 Hz, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.6, 132.0, 128.7, 117.8, 114.1, 108.9, 95.7, 76.9, 31.0, 22.0, 19.3, 13.6.

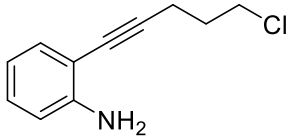
2-(4-methylpent-1-yn-1-yl)aniline (**1v**)



2-(4-methylpent-1-yn-1-yl)aniline (**1v**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 4-methylpent-1-yne (271.1 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:60 v/v) as an eluent provided the titled compound as a yellow liquid (337.9 mg, 65% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[18] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.24 (dd, J = 8.4, 1.6 Hz, 1H), 7.06 (td, J = 7.6, 1.6 Hz, 1H), 6.67-6.62 (m, 2H), 4.14 (s, 2H), 2.35 (d, J = 6.4 Hz, 2H), 1.96-1.86 (m, 1H), 1.04 (d, J = 6.8 Hz, 6H). **^{13}C NMR (100**

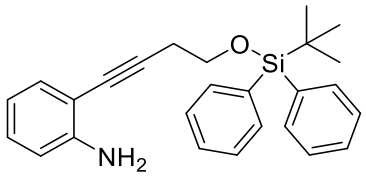
MHz, $CDCl_3$) δ 147.6, 132.0, 128.7, 117.7, 114.1, 108.9, 94.5, 77.8, 28.7, 28.2, 22.0.

2-(5-chloropent-1-yn-1-yl)aniline (**1w**)



2-(5-chloropent-1-yn-1-yl)aniline (**1w**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and 5-chloropent-1-yne (338.5 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:40 v/v) as an eluent provided the titled compound as a yellow liquid (435.8 mg, 75% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[19] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.24 (dd, J = 8.0, 1.6 Hz, 1H), 7.08 (ddd, J = 8.0, 7.2, 1.6 Hz, 1H), 6.69-6.64 (m, 2H), 4.13 (s, 2H), 3.72 (t, J = 6.4 Hz, 2H), 2.67 (t, J = 6.8 Hz, 2H), 2.06 (p, J = 6.4 Hz, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.7, 132.1, 129.1, 117.9, 114.2, 108.4, 93.3, 78.1, 43.7, 31.4, 17.0.

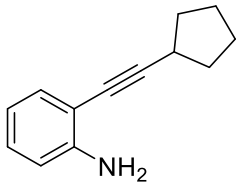
2-(4-((*tert*-butyldiphenylsilyl)oxy)but-1-yn-1-yl)aniline (**1x**)



2-(4-((*tert*-butyldiphenylsilyl)oxy)but-1-yn-1-yl)aniline (**1x**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and (but-3-yn-1-yloxy)(*tert*-butyl)diphenylsilane (1.02 g, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:20 v/v) as an eluent provided the titled compound as a yellow liquid (851.2 mg, 71% yield). **^{1}H NMR (400**

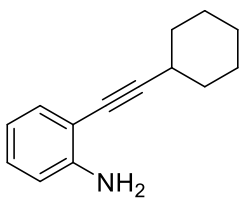
MHz, $CDCl_3$) δ 7.76-7.72 (m, 4H), 7.49-7.39 (m, 6H), 7.26 (dd, J = 7.6, 1.6 Hz, 1H), 7.11 (ddd, J = 8.0, 7.2, 1.6 Hz, 1H), 6.74-6.67 (m, 2H), 3.91 (t, J = 6.8 Hz, 2H), 2.77 (t, J = 6.4 Hz, 2H), 1.11 (s, 9H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.5, 135.6, 133.5, 132.0, 129.7, 129.0, 127.7, 117.9, 114.2, 108.7, 92.7, 78.0, 62.6, 26.8, 23.8, 19.2. **IR ν_{max}(KBr)/cm^{-1}** 2936, 2861, 1626, 1431, 1360, 1108, 745. **HRMS-APCI (m/z):** calcd for $C_{26}H_{30}NOSi$, $[M+H]^+$: 400.2091, found 400.2093.

2-(cyclopentylethynyl)aniline (**1y**)



2-(cyclopentylethynyl)aniline (**1y**) was prepared following the general procedure using 2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylcyclopentane (310.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow liquid (333.5 mg, 60% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[20] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.23 (dd, J = 7.6, 1.6 Hz, 1H), 7.07 (td, J = 7.6, 1.6 Hz, 1H), 6.71-6.63 (m, 2H), 3.42 (s, 2H), 2.92-2.85 (m, 1H), 2.06-1.97 (m, 2H), 1.81-1.67 (m, 4H), 1.66-1.57 (m, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.43 131.9, 128.7, 117.9, 114.1, 109.0, 100.1, 76.4, 34.2, 31.0, 25.0.

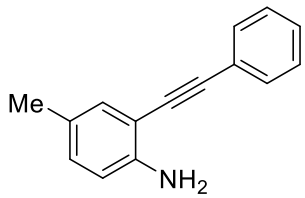
2-(cyclohexylethynyl)aniline (**1z**)



2-(cyclohexylethynyl)aniline (**1z**) was prepared following the general procedure using

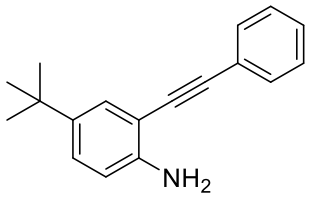
2-iodoaniline (657.1 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylcyclopentane (310.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow liquid (328.8 mg, 55% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[21] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.26-7.23 (m, 1H), 7.10-7.04 (m, 1H), 6.70-6.63 (m, 2H), 4.12 (s, 2H), 2.67-2.63 (m, 1H), 1.93-1.87 (m, 2H), 1.79-1.72 (m, 2H), 1.60-1.51 (m, 3H), 1.42-1.33 (m, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 147.5, 131.9, 128.7, 117.8, 114.1, 109.0, 99.9, 32.9, 29.9, 25.9, 24.9.

4-methyl-2-(phenylethynyl)aniline (1aa)



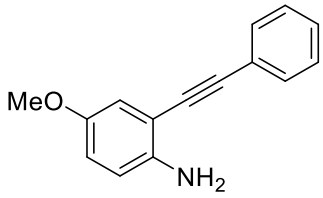
4-methyl-2-(phenylethynyl)aniline (**1aa**) was prepared following the general procedure using 2-iodo-4-methylaniline (699.2 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (597.0 mg, 97% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[7] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.55-7.48 (m, 2H), 7.38-7.30 (m, 3H), 7.19 (d, J = 2.0 Hz, 1H), 6.98-6.93 (m, 1H), 6.65 (d, J = 8.4 Hz, 1H), 4.04 (s, 2H), 2.23 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 145.4, 132.2, 131.4, 130.6, 128.3, 128.1, 127.3, 123.4, 114.6, 108.0, 94.4, 86.1, 20.3.

4-(*tert*-butyl)-2-(phenylethynyl)aniline (1ab)



4-(*tert*-butyl)-2-(phenylethynyl)aniline (**1ab**) was prepared following the general procedure using 4-(*tert*-butyl)-2-iodoaniline (825.4 mg, 3 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:40 v/v) as an eluent provided the titled compound as a yellow solid (710.7 mg, 95% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[22] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.64 (d, J = 7.2 Hz, 1H), 7.53-7.40 (m, 5H), 7.30 (d, J = 8.0 Hz, 1H), 7.26-7.18 (m, 2H), 6.69-6.47 (m, 2H), 2.92-2.82 (m, 1H), 2.80-2.63 (m, 2H), 2.58-2.46 (m, 1H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 145.3, 141.0, 131.5, 128.7, 128.3, 128.1, 127.1, 123.4, 114.4, 107.6, 94.2, 86.4, 33.9, 31.4.

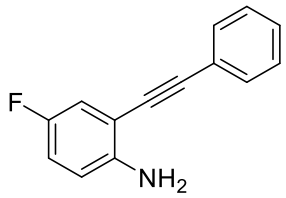
4-methoxy-2-(phenylethynyl)aniline (**1ac**)



4-methoxy-2-(phenylethynyl)aniline (**1ac**) was prepared following the general procedure using 2-iodo-4-methoxyaniline (747.2 mg, 3.0 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:10 v/v) as an eluent provided the titled compound as a yellow solid (602.9 mg, 90% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[23] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.54-7.49 (m, 2H), 7.36-7.30 (m, 3H), 6.92

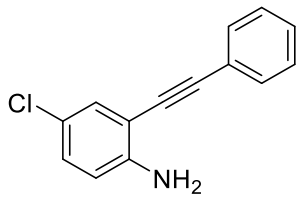
(d, *J* = 2.8 Hz, 1H), 6.77 (dd, *J* = 8.8, 3.2 Hz, 1H), 6.66 (d, *J* = 8.8 Hz, 1H), 4.01 (s, 2H), 3.73 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 151.8, 142.0, 131.4, 128.3, 128.2, 123.1, 117.4, 115.9, 115.7, 108.5, 94.6, 85.9, 55.7.

4-fluoro-2-(phenylethynyl)aniline (**1ad**)



4-fluoro-2-(phenylethynyl)aniline (**1ad**) was prepared following the general procedure using 4-fluoro-2-iodoaniline (711.0 mg, 3.0 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (614.7 mg, 97% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[24] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.57-7.46 (m, 2H), 7.40-7.32 (m, 1H), 7.14 (td, *J* = 7.6, 1.2 Hz, 1H), 7.04 (t, *J* = 8.4 Hz, 2H), 6.74-6.70 (m, 2H), 4.20 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 162.5 (d, *J* = 248.0 Hz), 147.7, 133.3 (d, *J* = 9.0 Hz), 132.1, 129.8, 119.4 (d, *J* = 3.0 Hz), 118.1, 115.7 (d, *J* = 22.0 Hz) 114.4, 107.8, 93.6, 85.5.

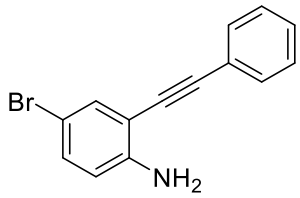
4-chloro-2-(phenylethynyl)aniline (**1ae**)



4-chloro-2-(phenylethynyl)aniline (**1ae**) was prepared following the general procedure using 4-chloro-2-iodoaniline (760.4 mg, 3.0 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room

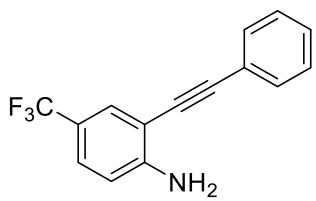
temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:30 v/v) as an eluent provided the titled compound as a yellow solid (580.6 mg, 85% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[25] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.52 (dd, J = 7.2, 3.6 Hz, 2H), 7.40-7.29 (m, 4H), 7.08 (dd, J = 8.8, 2.8 Hz, 1H), 6.67 (d, J = 8.8 Hz, 1H), 4.49 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 145.9, 131.5, 131.4, 129.6, 128.6, 128.4, 122.8, 122.6, 115.7, 109.6, 95.7, 84.5.

4-bromo-2-(phenylethynyl)aniline (**1af**)



4-bromo-2-(phenylethynyl)aniline (**1af**) was prepared following the general procedure using 4-bromo-2-iodoaniline (297.9 mg, 3.0 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:20 v/v) as an eluent provided the titled compound as a yellow solid (620.5 mg, 76% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[24] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.46-7.38 (m, 3H), 7.27 (dd, J = 4.0, 1.2 Hz, 3H), 7.13 (dd, J = 8.4, 2.0 Hz, 1H), 6.51 (d, J = 8.8 Hz, 1H), 4.20 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 146.7, 134.1, 132.4, 131.5, 128.5, 128.4, 122.7, 115.7, 109.8, 109.0, 95.7, 84.5.

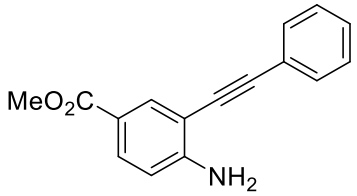
2-(phenylethynyl)-4-(trifluoromethyl)aniline (**1ag**)



2-(phenylethynyl)-4-(trifluoromethyl)aniline (**1ag**) was prepared following the general

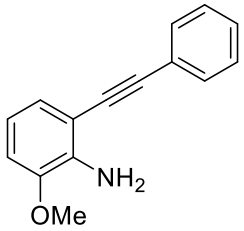
procedure using 2-iodo-4-(trifluoromethyl)aniline (861.1 mg, 3.0 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:20 v/v) as an eluent provided the titled compound as a yellow solid (666.2 mg, 85% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[26] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.61 (s, 1H), 7.51 (dd, J = 7.6, 3.6 Hz, 2H), 7.38-7.29 (m, 4H), 6.68 (d, J = 8.8 Hz, 1H), 4.54 (s, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 150.2, 131.5, 129.4 (q, J = 3.9 Hz), 128.6, 128.4, 126.5 (q, J = 3.7 Hz), 119.7 (q, J = 33.0 Hz), 119.2, 113.7, 107.4, 95.6, 84.3.

methyl 4-amino-3-(phenylethynyl)benzoate (**1ah**)



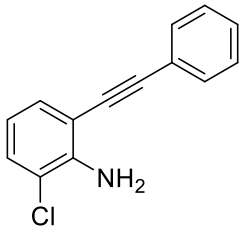
methyl 4-amino-3-(phenylethynyl)benzoate (**1ah**) was prepared following the general procedure using methyl 4-amino-3-iodobenzoate (681.2 mg, 3.0 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:5 v/v) as an eluent provided the titled compound as a yellow solid (640.8 mg, 85% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[24] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.08 (d, J = 2.0 Hz, 1H), 7.81 (dd, J = 8.4, 2.0 Hz, 1H), 7.52 (dd, J = 7.2, 3.6 Hz, 2H), 7.34 (dd, J = 4.8, 1.6 Hz, 3H), 6.69 (d, J = 8.4 Hz, 1H), 4.70 (s, 2H), 3.86 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 166.6, 151.4, 134.3, 131.4, 131.3, 128.4, 128.4, 122.8, 119.3, 113.2, 107.1, 95.0, 84.7, 51.7.

2-methoxy-6-(phenylethynyl)aniline (1ai)



2-methoxy-6-(phenylethynyl)aniline (**1ai**) was prepared following the general procedure using methyl 2-iodo-6-methoxyaniline (747.2 mg, 3.0 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:10 v/v) as an eluent provided the titled compound as a yellow solid (502.4 mg, 75% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[27] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.56-7.50 (m, 2H), 7.37-7.30 (m, 3H), 7.00 (dd, J = 8.0, 1.6 Hz, 1H), 6.76 (dd, J = 8.4, 1.6 Hz, 1H), 6.66 (t, J = 8.0 Hz, 1H), 4.42 (s, 2H), 3.85 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 146.6, 138.4, 131.4, 128.3, 128.1, 123.9, 123.3, 117.1, 110.4, 107.5, 94.6, 85.8, 55.6.

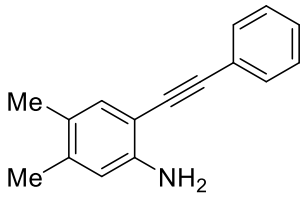
2-chloro-6-(phenylethynyl)aniline (1aj)



2-chloro-6-(phenylethynyl)aniline (**1aj**) was prepared following the general procedure using methyl 2-chloro-6-iodoaniline (760.4 mg, 3.0 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:20 v/v) as an eluent provided the titled compound as a yellow solid (505.5 mg, 74% yield). NMR spectra data of the isolated material matched

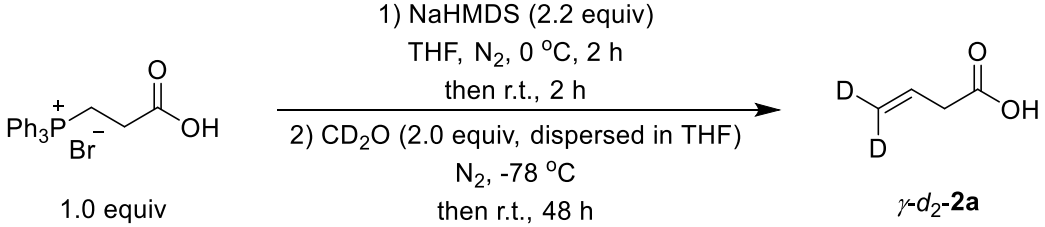
with the spectra data reported in the literature for this compound.[28] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.57-7.51 (m, 2H), 7.37 (dd, J = 4.8, 1.6 Hz, 2H), 7.28 (d, 2.4 Hz, 2H), 6.77-6.70 (m, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 148.6, 135.3, 133.1, 131.5, 128.4, 123.0, 118.2, 114.1, 106.5, 95.4, 84.9.

4,5-dimethyl-2-(phenylethynyl)aniline (1ak)



4,5-dimethyl-2-(phenylethynyl)aniline (**1ak**) was prepared following the general procedure using methyl 2-iodo-4,5-dimethylaniline (741.2 mg, 3.0 mmol, 1.0 equiv), $Pd(PPh_3)_2Cl_2$ (42.1 mg, 2 mol %), CuI (11.4 mg, 2 mol %) and ethynylbenzene (330.7 mg, 3.3 mmol, 1.1 equiv) in NEt_3 (12 mL). The resulting mixture was stirred under nitrogen atmosphere at room temperature for 4 h. Purification by column chromatography on silica gel using EtOAc: PE (1:50 v/v) as an eluent provided the titled compound as a yellow solid (524.5 mg, 97% yield). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[29] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.55-7.47 (m, 2H), 7.35-7.30 (m, 3H), 7.13 (s, 1H), 6.55 (s, 1H), 4.08 (s, 2H), 2.19 (s, 3H), 2.15 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 145.7, 138.8, 132.6, 131.4, 128.3, 127.9, 126.3, 123.6, 116.0, 105.4, 93.9, 86.2, 20.0, 18.6.

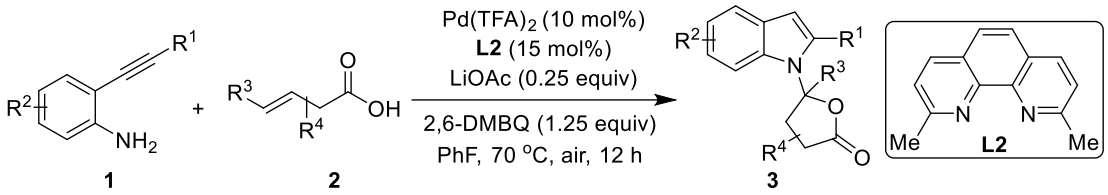
2.3 Synthesis of but-3-enoic-4,4-d_2 acid[2]



To a 100 mL dried two-neck flask equipped with a magnetic stir bar was added (2-carboxyethyl)triphenylphosphonium bromide (2.08 g, 5 mmol, 1.0 equiv), anhydrous

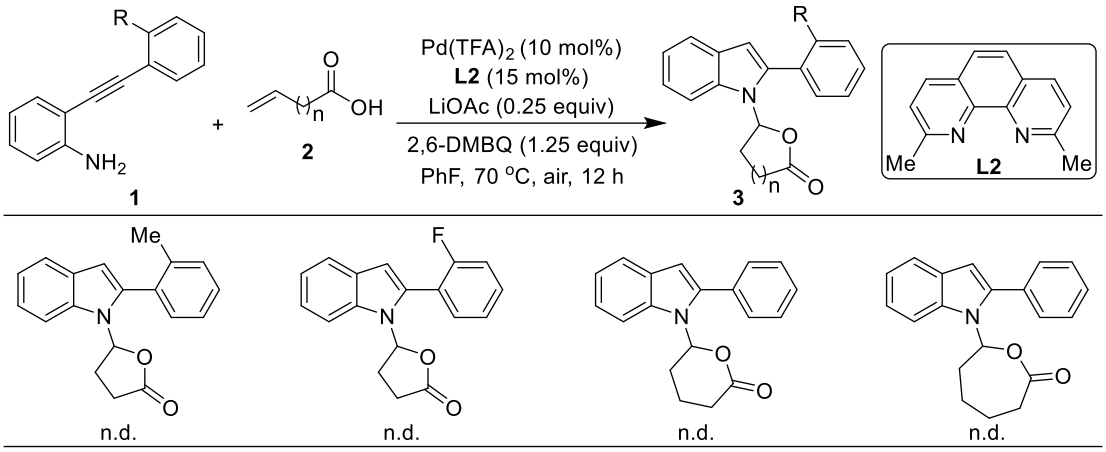
THF (30 mL) under nitrogen. Dropwise addition of NaHMDS solution (2 M in THF, 5.5 mL, 11 mmol, 2.0 equiv) at 0 ºC afforded a brilliant orange mixture. After being stirred for 1 h at this temperature and 2 h at room temperature, the resulting brown mixture was cooled to -78 ºC, and paraformaldehyde-d_2 (0.30 g, 10 mmol, 2.0 equiv) dispersed in anhydrous THF (4 mL) was added to the orange solution. The reaction was then stirred at room temperature for 48 h. After this time, the solvent was removed in vacuo. The residue was dissolved by water (40 mL) and washed with Et_2O (20 mL × 3). The aqueous phase was acidified to pH = 2 with aqueous HCl (6 N) and extracted with Et_2O (3 × 20 mL). The combined organic layer was dried over MgSO4, concentrated under reduced pressure. The residue was purified by flash chromatography (EtOAc: PE = 1:5 v/v) to afford γ-d_2-**2a** as an orange oil (96.9 mg, 22% yield, > 98% D). NMR spectra data of the isolated material matched with the spectra data reported in the literature for this compound.[2] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 10.93 (s, 1H), 5.87-5.82 (s, 1H), 3.08 (d, J = 7.0 Hz, 2H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 178.2, 129.3, 119.0-118.0 (m, the deuterium containing carbon, relatively low), 38.7.

3. General procedure for the synthesis of products



To a 25 mL dried round bottom flask equipped with a magnetic stir bar was added $Pd(TFA)_2$ (6.6 mg, 10 mol%), **L2** (6.2 mg, 15 mol%), LiOAc (3.3 mg, 0.25 equiv), substituted 2-alkynyl aniline **1** (0.2 mmol, 1.0 equiv), 2,6-DMBQ (34 mg, 1.25 equiv) and PhF (1.5 mL), followed by the addition of substituted 3-butenoic acid **2** (0.4 mmol, 2.0 equiv). The mixture was stirred at 70 °C for 12 h under air. After the reaction was completed, the mixture was cooled to room temperature, diluted with aqueous saturated $NaHCO_3$ (15 mL) and extracted with EtOAc (10 mL × 3). The organic layer was collected and dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The crude mixture was purified by flash chromatography on silica gel to afford the product **3**.

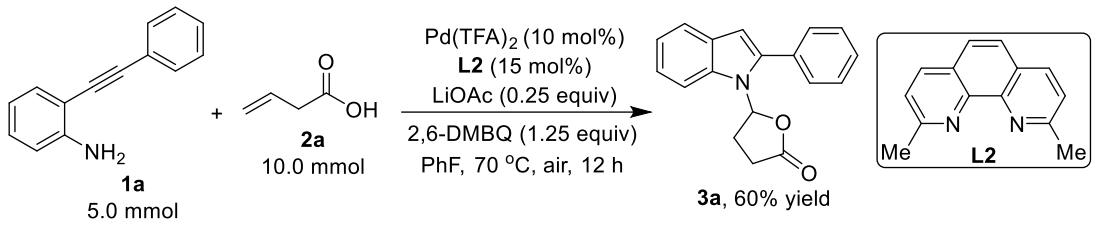
4. Unsuccessful substrates



Reaction conditions: **1** (0.2 mmol), **2** (2.0 equiv), $Pd(TFA)_2$ (10 mol%), **L2** (15 mol%), 2,6-DMBQ (1.25 equiv), LiOAc (0.25 equiv) in PhF (1.5 mL) at 70 °C under air for 12 h. n.d. = not detected.

When 2-(*o*-tolylethynyl)aniline or 2-((2-fluorophenyl)ethynyl)aniline was subjected, respectively, the corresponding products could not be detected by GC-MS, with a large amount of starting material remained. This may be attributed to the steric hindrance effects that prevent the coordination of alkyne with palladium. When 4-pentenoic acid or 5-hexenoic acid was subjected to the standard reaction conditions, respectively, the corresponding product could not be detected by GC-MS. We assume that increasing the carbon chain may weaken the coordination between carboxyl group and palladium, and thus the alkene could not be activated.

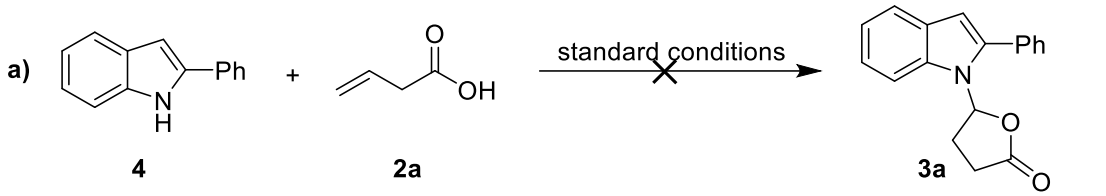
5. Larger-scale reaction



To a 100 mL dried round bottom flask equipped with a magnetic stir bar was added $Pd(TFA)_2$ (166.2 mg, 10 mol%), **L2** (156.2 mg, 15 mol%), LiOAc (82.5 mg, 0.25 equiv), 2-(phenylethynyl)aniline **1a** (996.3 mg, 5.0 mmol, 1.0 equiv), 2,6-DMBQ (8850.9 mg, 6.25 mmol, 1.25 equiv) and PhF (37.5 mL), followed by the addition of 3-butenoic acid **2a** (880.0 μL, 10.0 mmol, 2.0 equiv). The mixture was stirred at 70 °C for 12 h under air. After the reaction was completed, the mixture was cooled to room temperature, diluted with aqueous saturated $NaHCO_3$ (40 mL) and extracted with EtOAc (40 mL × 3). The combined organic layers were dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The crude mixture was purified by column chromatography on silica gel using Ether : Hexane (1:1 v/v) as an eluent to provide **3a** as a yellow solid (832.0 mg, 60% yield).

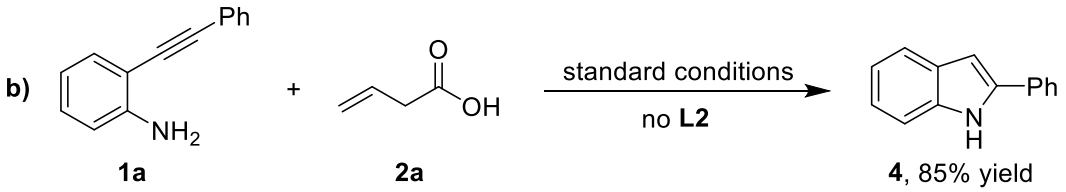
6. Mechanistic investigation

6.1 Controlling experiments



To a 25 mL dried round bottom flask equipped with a magnetic stir bar was added $Pd(TFA)_2$ (6.6 mg, 10 mol%), 2,9-dimethyl-1,10-phenanthroline (6.2 mg, 15 mol%), 2-phenyl-1*H*-indole **4** (38.6 mg, 0.2 mmol, 1.0 equiv), 3-butenoic acid **2a** (36 μL, 0.4 mmol, 2.0 equiv) and PhF (1.5 mL), followed by the addition of 2,6-DMBQ (34 mg, 1.25 equiv). The mixture was stirred at 70 °C for 12 h under air. After the reaction was completed, the mixture was detected by GC-MS, but the product **3a** was not detected.

The experimental result shows that the reaction is more likely to be initiated by the intermolecular aminopalladation of 2-phenylethynyl aniline with 3-butenoic acid than the intramolecular aminopalladation of 2-phenylethynyl aniline.

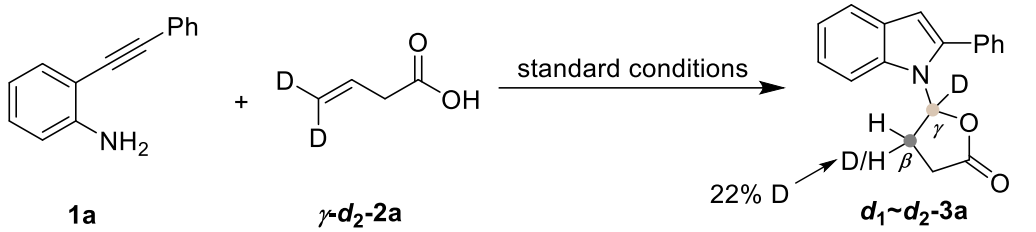


To a 25 mL dried round bottom flask equipped with a magnetic stir bar was added $Pd(TFA)_2$ (6.6 mg, 10 mol%), 2-phenylethynyl aniline **1a** (38.6 mg, 0.2 mmol, 1.0 equiv), 3-butenoic acid **2a** (36 μL, 0.4 mmol, 2.0 equiv) and PhF (1.5 mL), followed by the addition of 2,6-DMBQ (34 mg, 1.25 equiv). The mixture was stirred at 70 °C for 12 h under air. After the reaction was completed, the mixture was cooled to room temperature, diluted with EtOAc (10 mL) and washed with aqueous saturated $NaHCO_3$ (15 mL). The aqueous layer was further extracted with EtOAc (10 mL × 3) and dried over anhydrous $MgSO_4$. After removal of EtOAc in vacuum, the residue was purified by flash chromatography on silica gel using EtOAc: PE (1:10 v/v) as an eluent to afford the 2-phenyl-1*H*-indole **4** as a yellow solid (32.9 mg, 85%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 8.37 (s, 1H), 7.72-7.65 (m, 3H), 7.51-7.41 (m, 3H), 7.39-7.33 (m, 1H), 7.24 (ddd, J = 8.0, 6.8, 1.2 Hz, 1H), 7.17 (ddd, J = 8.0, 7.2, 1.2 Hz, 1H), 6.87 (dd, J = 2.0,

0.8 Hz, 1H).

The experimental result shows that ligand L2 may be play a critical role in effectively inhibiting the intramolecular aminopalladation of 2-phenylethynyl aniline 1a.

6.2 Deuterium-labeling experiments

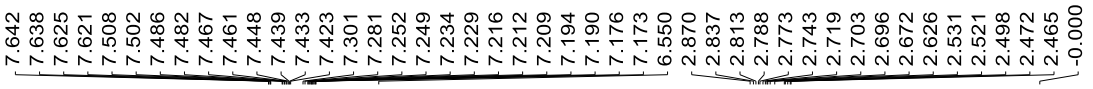


1a **γ-d_2-2a** **d_1~d_2-3a**

58 % yield (over all)

To a 25 mL dried round bottom flask equipped with a magnetic stir bar was added $Pd(TFA)_2$ (6.6 mg, 10 mol%), 2,9-dimethyl-1,10-phenanthroline (6.2 mg, 15 mol%), 2-phenylethynyl aniline **1a** (38.6 mg, 0.2 mmol, 1.0 equiv), but-3-enoic-4,4-d_2 acid *γ*-d_2-**2a** (36 μL, 0.4 mmol, 2.0 equiv) and PhF (1.5 mL), followed by the addition of 2,6-DMBQ (34 mg, 1.25 equiv). The mixture was stirred at 70 °C for 12 h under air. After the reaction was completed, the mixture was cooled to room temperature, diluted with EtOAc (10 mL) and washed with aqueous saturated $NaHCO_3$ (15 mL). The aqueous layer was further extracted with EtOAc (10 mL × 3) and dried over anhydrous $MgSO_4$. After removal of EtOAc in vacuum, the residue was purified by flash chromatography on silica gel using Ether : Hexane (1:1 v/v) as an eluent to afford the product d_1~d_2-**3a** as a yellow solid (15.6 mg, 58%). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.63 (dd, J = 6.8, 1.6 Hz, 1H), 7.50-7.40 (m, 5H), 7.29 (d, J = 8.0 Hz, 1H), 7.24-7.17 (m, 2H), 6.55 (s, 1H), 2.90-2.78 (m, 1H), 2.78-2.62 (m, 2H), 2.54-2.45 (m, **0.78H**).

The presence of 1.0 D at the γ position is in line with the proposed sequence of aminopalladation followed by β-H elimination, whereas the 0.22 D at the β position may be partially due to Pd-D participation in tautomerization from enamine to imine.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7.638
7.625
7.621
7.508
7.502
7.486
7.482
7.467
7.461
7.448
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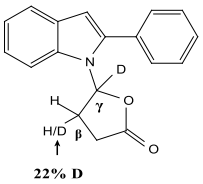
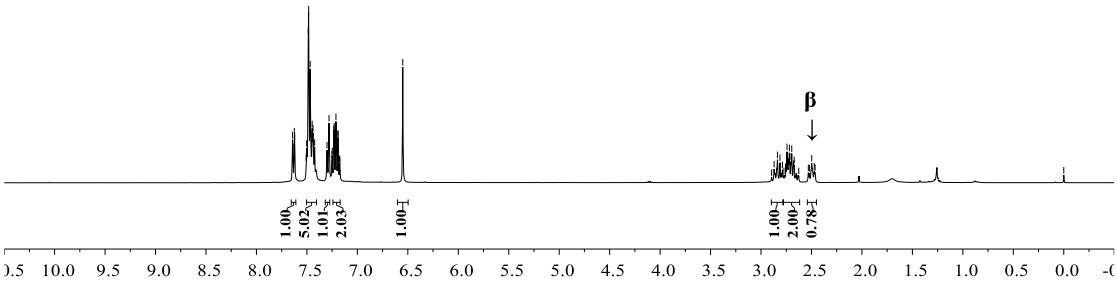
7.433
7.423
7.301
7.281
7.252
7.249
7.234
7.229
7.216
7.212
7.209
7.194
7.190
7.176
7.173
6.550
2.870
2.837
2.813
2.788
2.773
2.743
2.719
2.703
2.696
2.672
2.626
2.531
2.521
2.498
2.472
2.465
-0.000


N
D
H
γ
O
H/D
β
O

22% D


β
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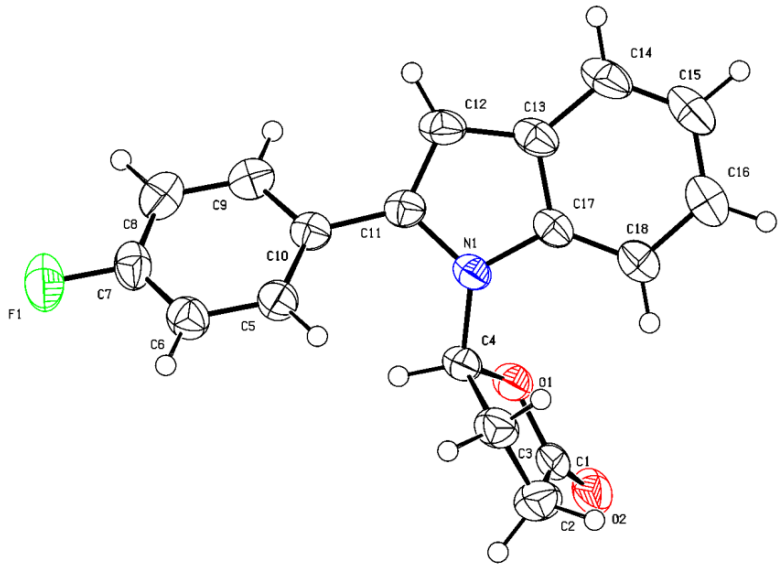
5.02
1.01
2.03
1.00
1.00
2.00
0.78
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

7. Crystal structure data of 3j

Compound **3j** was dissolved in DCM/Hexane (2 mL, 1:1) and the solvent was allowed to slowly evaporated at room temperature to give a crystal suitable for X-ray diffraction analysis. The intensity data was collected on a D8 VENTURE instrument. The data were outlined below.



(The thermal ellipsoids was drawn at the 50% probability level.)

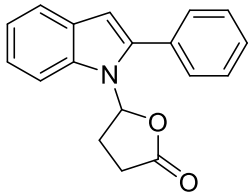
Table S1 Crysal data and structure refinements for **3j**

Compound	**3j**
Crystal Number	CCDC 2336722
Empirical formula	$C_{18}H_{14}FNO_2$
Formula weight	295.30
Temperature/K	170.0
Crystal system,	triclinic
Space group	P-1
a/Å	9.2770(6)
b/Å	9.5602(6)
c/Å	9.7665(6)
α/°	104.289(2)
β/°	110.312(2)
γ/°	107.388(2)

Volume/$Å^3$	713.26(8)
Z	2
$\rho_{calc}g/cm^3$	1.375
μ/ mm^{-1}	0.099
F (000)	308.0
Crystal size/mm^3	0.12 × 0.08 × 0.05
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/º	5.168 to 55.018
Index ranges	$-12 \leq h \leq 11$, $-12 \leq k \leq 11$, $-12 \leq l \leq 12$
Reflections collected	8815
Independent reflections	3229 [R_{int} = 0.0626, R_{sigma} = 0.0806]
Data/restraints/parameters	3229/0/199
Goodness-of-fit on F^2	1.050
Final R indices [I>=2σ (I)]	R_1 = 0.0529, wR_2 = 0.1009
Final R indexes [all data]	R_1 = 0.1178, wR_2 = 0.1309
Largest diff. peak/hole / e $Å^{-3}$	0.18/-0.24

8. Analytic data of products

5-(2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3a)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3a** as a yellow solid (42.2 mg, 76% yield).

m.p. = 101.7-102.2 ℃.

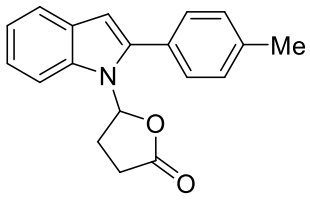
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 7.2 Hz, 1H), 7.53-7.40 (m, 5H), 7.30 (d, J = 8.0 Hz, 1H), 7.26-7.18 (m, 2H), 6.69-6.47 (m, 2H), 2.92-2.82 (m, 1H), 2.80-2.63 (m, 2H), 2.58-2.46 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 141.0, 135.3, 131.9, 129.6, 129.4, 128.8, 128.6, 122.7, 121.4, 111.4, 105.0, 85.9, 28.9, 26.2.

IR ν_{max}(KBr)/cm^{-1} 2923, 2817, 1771, 1626, 1516, 1352, 1162, 940, 689.

HRMS-APCI (m/z): calcd for $C_{18}H_{16}NO_2$, $[M+H]^+$: 278.1176, found 278.1171.

5-(2-(*p*-tolyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3b)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3b** as a yellow solid (38.5 mg, 66% yield).

m.p. = 139.8-140.2 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, J = 7.2 Hz, 1H), 7.39 (d, J = 8.0 Hz, 2H), 7.31-7.26 (m, 3H), 7.24-7.16 (m, 2H), 6.58 (t, J = 7.2 Hz, 1H), 6.53 (s, 1H), 2.91-2.81 (m, 1H), 2.79-2.64 (m, 2H), 2.55- 2.46 (m, 1H), 2.42 (s, 3H).

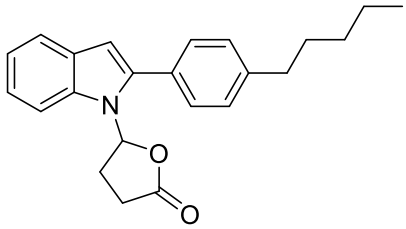
^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 141.2, 138.7, 135.2, 129.5, 129.5, 129.0, 122.5,

121.3, 121.3, 111.3, 104.6, 85.9, 29.0, 26.2, 21.3.

IR ν_{max}(KBr)/cm^{-1} 2937, 2877, 1770, 1660, 1514, 1294, 1163, 938, 690.

HRMS-APCI (m/z): calcd for $C_{19}H_{18}NO_2$, $[M+H]^+$: 292.1332, found 292.1327.

5-(2-(4-pentylphenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3c)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3c** as a yellow solid (42.0 mg, 63% yield).

m.p. = 64.5-64.9 ℃.

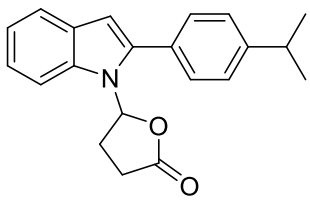
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (dd, J = 6.8, 1.6 Hz, 1H), 7.40 (d, J = 7.6 Hz, 2H), 7.32-7.26 (m, 3H), 7.26-7.17 (m, 2H), 6.60 (t, J = 7.6 Hz, 1H), 6.53 (s, 1H), 2.92-2.82 (m, 1H), 2.80-2.63 (m, 4H), 2.57-2.47 (m, 1H), 1.70-1.63 (m, 2H), 1.41-1.33 (m, 4H), 0.95-0.89 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 143.7, 141.2, 135.3, 129.5, 129.5, 129.2, 128.9, 122.5, 121.3, 121.3, 111.3, 104.6, 85.9, 35.7, 31.5, 31.1, 29.0, 26.1, 22.5, 14.0.

IR ν_{max}(KBr)/cm^{-1} 2947, 2816, 1770, 1622, 1516, 1326, 1153, 940, 620.

HRMS-APCI (m/z): calcd for $C_{23}H_{26}NO_2$, $[M+H]^+$: 348.1958, found 348.1953.

5-(2-(4-isopropylphenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3d)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3d** as a white solid (37.1 mg, 58% yield).

m.p. = 164.3-164.8 ℃.

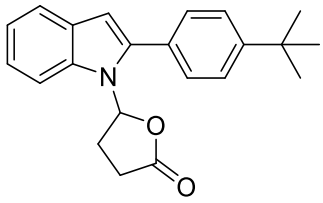
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (dd, J = 7.2, 2.0 Hz, 1H), 7.40 (d, J = 8.4 Hz, 2H), 7.33-7.26 (m, 3H), 7.22-7.15 (m, 2H), 6.57 (t, J = 7.6 Hz, 1H), 6.51 (s, 1H), 3.00-2.92 (m, 1H), 2.88-2.79 (m, 1H), 2.77-2.70 (m, 1H), 2.69-2.59 (m, 1H), 2.52-2.44 (m, 1H), 1.29 (d, J = 6.9 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 149.6, 141.2, 135.2, 129.6, 129.5, 129.3, 126.9, 122.5, 121.3, 121.3, 111.4, 104.6, 85.9, 33.9, 29.0, 26.1, 23.9, 23.9.

IR ν_{max}(KBr)/cm^{-1} 2930, 2816, 1772, 1628, 1441, 1353, 1159, 938, 754.

HRMS-APCI (m/z): calcd for $C_{21}H_{22}NO_2$, $[M+H]^+$: 320.1645, found 320.1639.

5-(2-(4-(*tert*-butyl)phenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3e)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3e** as a yellow solid (46.7 mg, 70% yield).

m.p. = 204.9-205.5 ℃.

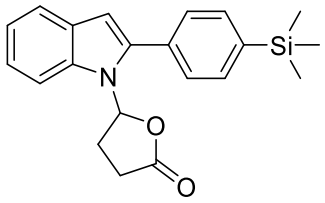
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, J = 6.8 Hz, 1H), 7.50 (d, J = 8.4 Hz, 2H), 7.43 (d, J = 8.4 Hz, 2H), 7.29 (d, J = 8.0 Hz, 1H), 7.24-7.15 (m, 2H), 6.60 (t, J = 7.6 Hz, 1H), 6.54 (s, 1H), 2.95-2.85 (m, 1H), 2.83-2.66 (m, 2H), 2.58-2.49 (m, 1H), 1.38 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 151.8, 141.2, 135.2, 129.6, 129.3, 129.0, 125.8, 122.5, 121.3, 121.3, 111.4, 104.6, 86.0, 34.7, 31.3, 29.0, 26.1.

IR ν_{max}(KBr)/cm^{-1} 2946, 2800, 1771, 1628, 1514, 1346, 1162, 931, 683.

HRMS-APCI (m/z): calcd for $C_{22}H_{22}NO_2$, $[M-H]^-$: 332.1656, found 332.1658.

5-(2-(4-(trimethylsilyl)phenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3f)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3f** as a yellow solid (44.7 mg, 64% yield).

m.p. = 198.7-199.4 ℃.

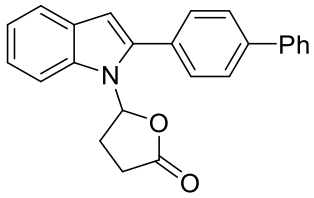
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65-7.63 (m, 3H), 7.49 (d, J = 8.0 Hz, 2H), 7.31-7.18 (m, 3H), 6.60 (t, J = 7.6 Hz, 1H), 6.56 (s, 1H), 2.97-2.87 (m, 1H), 2.86-2.66 (m, 2H), 2.58-2.49 (m, 1H), 0.32 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 141.3, 141.2, 135.3, 133.8, 132.2, 129.6, 128.7, 122.7, 121.4, 111.5, 104.9, 86.0, 29.0, 26.1, -1.2.

IR ν_{max}(KBr)/cm^{-1} 2930, 2855, 1784, 1629, 1456, 1342, 1252, 1111, 746.

HRMS-APCI (m/z): calcd for $C_{21}H_{24}NO_2Si$, $[M+H]^+$: 350.1571, found 350.1564.

5-(2-([1,1'-biphenyl]-4-yl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3g)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3g** as a white solid (44.5 mg, 63% yield).

m.p. = 190.4-191.2 ℃.

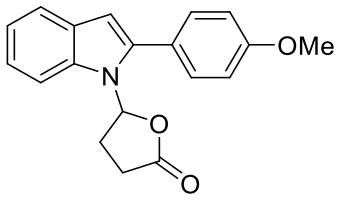
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, J = 8.0 Hz, 2H), 7.68-7.62 (m, 3H), 7.58 (d, J = 8.4 Hz, 2H), 7.48 (t, J = 7.6 Hz, 2H), 7.42-7.37 (m, 1H), 7.32 (d, J = 8.0 Hz, 1H), 7.28-7.19 (m, 2H), 6.65 (t, J = 7.6 Hz, 1H), 6.61 (s, 1H), 2.97-2.87 (m, 1H), 2.86-2.67 (m, 2H), 2.61-2.50 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 141.5, 140.8, 140.2, 135.4, 130.8, 129.9, 129.5, 128.9, 127.8, 127.5, 127.1, 122.7, 121.4, 121.4, 111.5, 105.0, 86.0, 29.0, 26.2.

IR ν_{max}(KBr)/cm^{-1} 2903, 2855, 1773, 1615, 1496, 1352, 1163, 938, 638.

HRMS-APCI (m/z): calcd for $C_{24}H_{20}NO_2$, $[M+H]^+$: 354.1489, found 354.1482.

5-(2-(4-methoxyphenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3h)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3h** as a yellow solid (30.7 mg, 50% yield).

m.p. = 201.7-202.2 ℃.

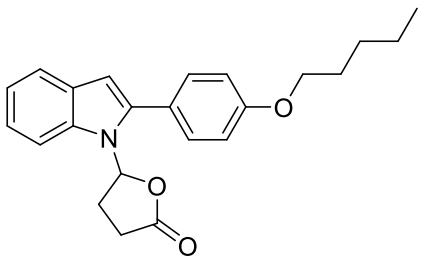
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, J = 7.6 Hz, 1H), 7.41 (d, J = 8.4 Hz, 2H), 7.29-7.16 (m, 3H), 6.99 (d, J = 8.4 Hz, 2H), 6.56 (t, J = 7.6 Hz, 1H), 6.49 (s, 1H), 3.85 (s, 3H), 2.90-2.81 (m, 1H), 2.79-2.61 (m, 2H), 2.54-2.45 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 159.9, 140.9, 135.1, 130.9, 129.4, 124.1, 122.4, 121.2, 121.1, 114.2, 111.2, 104.4, 85.9, 55.4, 28.9, 26.1.

IR ν_{max}(KBr)/cm^{-1} 2944, 2814, 1773, 1610, 1563, 1307, 1162, 939, 697.

HRMS-APCI (m/z): calcd for $C_{19}H_{18}NO_3$, $[M+H]^+$: 308.1281, found 308.1275.

5-(2-(4-(pentyloxy)phenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3i)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3i** as a yellow solid (30.1 mg, 43% yield).

m.p. = 105.8-106.3 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (dd, J = 6.8, 1.6 Hz, 1H), 7.40 (d, J = 8.4 Hz, 2H),

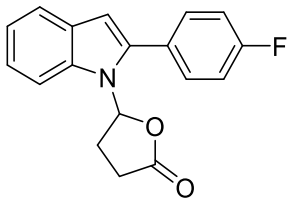
7.28 (d, *J* = 8.0 Hz, 1H), 7.24-7.17 (m, 2H), 6.99 (d, *J* = 8.4 Hz, 2H), 6.57 (t, *J* = 7.6 Hz, 1H), 6.49 (s, 1H), 4.00 (t, *J* = 6.4 Hz, 2H), 2.93-2.81 (m, 1H), 2.80-2.64 (m, 2H), 2.56-2.45 (m, 1H), 1.86-1.79 (m, 2H), 1.49-1.38 (m, 4H), 0.95 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 159.5, 141.0, 135.1, 130.9, 129.5, 123.9, 122.4, 121.2, 121.1, 114.8, 111.2, 104.4, 85.9, 68.1, 29.0, 28.9, 28.2, 26.1, 22.4, 14.0.

IR ν_{max}(KBr)/cm^{-1} 2957, 2816, 1774, 1617, 1476, 1353, 1162, 940, 681.

HRMS-APCI (m/z): calcd for $C_{23}H_{26}NO_3$, $[M+H]^+$: 364.1907, found 364.1900.

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



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3j** as a yellow solid (44.3 mg, 75% yield).

m.p. = 132.3-133.2 ℃.

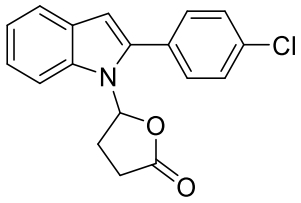
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67-7.62 (m, 1H), 7.48 (dd, *J* = 8.8, 5.6 Hz, 2H), 7.31-7.15 (m, 5H), 6.55-6.51 (m, 2H), 2.92-2.84 (m, 1H), 2.82-2.67 (m, 2H), 2.58-2.49 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 162.9 (d, *J* = 247.7 Hz), 139.9, 135.4, 131.4 (d, *J* = 8.2 Hz), 129.3, 128.0 (d, *J* = 3.3 Hz), 122.8, 121.5, 121.4, 116.0 (d, *J* = 21.6 Hz), 111.3, 105.2, 85.8, 28.9, 26.1.

IR ν_{max}(KBr)/cm^{-1} 2960, 2842, 1777, 1624, 1502, 1362, 1102, 936, 639.

HRMS-APCI (m/z): calcd for $C_{18}H_{15}FNO_2$, $[M+H]^+$: 296.1081, found 296.1078.

5-(2-(4-chlorophenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3k)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3k** as a yellow solid (41.2 mg, 66% yield).

m.p. = 136.8-137.2 ℃.

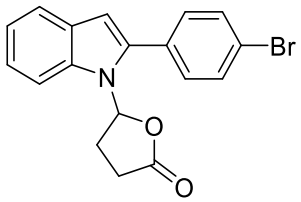
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (dd, J = 8.0, 1.2 Hz, 1H), 7.45-7.40 (m, 4H), 7.31-7.23 (m, 2H), 7.22-7.18 (m, 1H), 6.56-6.48 (m, 2H), 2.89-2.82 (m, 1H), 2.81-2.65 (m, 2H), 2.56-2.45 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3, 139.7, 135.4, 134.8, 130.7, 130.3, 129.3, 129.1, 122.9, 121.5, 121.4, 111.4, 105.3, 85.7, 28.9, 26.1.

IR ν_{max}(KBr)/cm^{-1} 2951, 2846, 1774, 1619, 1502, 1363, 1165, 942, 632.

HRMS-APCI (m/z): calcd for $C_{18}H_{15}ClNO_2$, $[M+H]^+$: 312.0786, found 312.0780.

5-(2-(4-bromophenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3l)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3l** as a yellow solid (43.5 mg, 61% yield).

m.p. = 156.8-157.2 ℃.

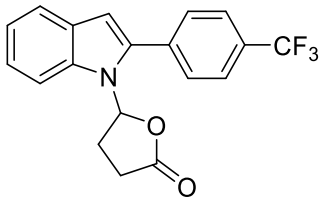
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 7.2 Hz, 1H), 7.60 (d, J = 8.4 Hz, 2H), 7.36 (d, J = 8.4 Hz, 2H), 7.32-7.18 (m, 3H), 6.56 (s, 1H), 6.52 (t, J = 7.6 Hz, 1H), 2.92-2.67 (m, 3H), 2.58-2.46 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3, 139.7, 135.4, 132.0, 131.0, 130.8, 129.3, 123.0, 122.9, 121.5, 121.5, 111.4, 105.3, 85.7, 28.9, 26.1.

IR ν_{max}(KBr)/cm^{-1} 2929, 2816, 1770, 1626, 1501, 1359, 1162, 940, 687.

HRMS-APCI (m/z): calcd for $C_{18}H_{15}BrNO_2$, $[M+H]^+$: 356.0115, found 356.0117.

5-(2-(4-(trifluoromethyl)phenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3m)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3m** as a yellow solid (41.4 mg, 60% yield).

m.p. = 158.7-159.2 ℃.

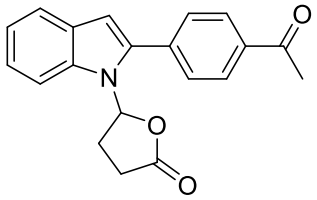
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, J = 8.0 Hz, 2H), 7.68-7.62 (m, 3H), 7.33-7.26 (m, 2H), 7.25-7.20 (m, 1H), 6.63 (s, 1H), 6.53 (t, J = 8.0 Hz, 1H), 2.93-2.87 (m, 1H), 2.87-2.70 (m, 2H), 2.59-2.50 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 139.4, 135.6, 135.5, 130.6 (q, J = 30.0 Hz), 129.8, 129.3, 125.9 (q, J = 3.7 Hz), 123.9 (q, J = 270.6 Hz), 123.3, 121.7, 111.6, 106.1, 85.8, 28.9, 26.2.

IR ν_{max}(KBr)/cm^{-1} 2936, 2829, 1772, 1605, 1495, 1354, 1165, 927, 669.

HRMS-APCI (m/z): calcd for $C_{19}H_{15}F_3NO_2$, $[M+H]^+$: 346.1049, found 346.1042.

5-(2-(4-acetylphenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3n)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3n** as a yellow solid (46.0mg, 72% yield).

m.p. = 154.2-154.7 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 8.4 Hz, 2H), 7.66 (d, J = 7.2 Hz, 1H), 7.61 (d, J = 8.4 Hz, 2H), 7.35-7.26 (m, 2H), 7.22 (td, J = 8.0, 6.8, 1.6 Hz, 1H), 6.64 (s, 1H), 6.58 (t, J = 7.6 Hz, 1H), 2.92-2.86 (m, 1H), 2.85-2.69 (m, 2H), 2.64 (s, 3H), 2.61-2.51 (m, 1H).

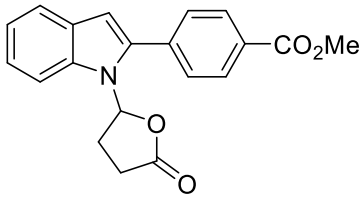
^{13}C NMR (100 MHz, $CDCl_3$) δ 197.3, 174.2, 139.8, 136.7, 136.5, 135.8, 129.5, 129.4,

128.8, 123.3, 121.7, 111.5, 106.2, 85.8, 28.9, 26.6, 26.2.

IR ν_{max}(KBr)/cm^{-1} 2935, 2825, 1771, 1607, 1494, 1362, 1162, 923, 666.

HRMS-APCI (m/z): calcd for $C_{20}H_{18}NO_3$, $[M+H]^+$: 320.1281, found 320.1275.

methyl 4-(1-(5-oxotetrahydrofuran-2-yl)-1*H*-indol-2-yl)benzoate (3o)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3o** as a yellow solid (40.2 mg, 60% yield).

m.p. = 171.2-172.1 ℃.

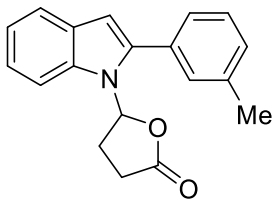
^{1}H NMR (400 MHz, CDCl$_3$) δ 8.14 (d, J = 8.4 Hz, 2H), 7.65 (d, J = 7.2 Hz, 1H), 7.58 (d, J = 8.4 Hz, 2H), 7.32-7.19 (m, 3H), 6.63 (s, 1H), 6.57 (t, J = 7.6 Hz, 1H), 3.95 (s, 3H), 2.91-2.70 (m, 3H), 2.60-2.49 (m, 1H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 174.3, 166.5, 139.9, 136.3, 135.7, 130.0, 130.0, 129.3, 129.3, 123.2, 121.6, 121.6, 111.5, 106.0, 85.8, 52.3, 28.9, 26.2.

IR ν_{max}(KBr)/cm^{-1} 2927, 2852, 1774, 1626, 1477, 1323, 1158, 915, 637.

HRMS-APCI (m/z): calcd for $C_{20}H_{18}NO_4$, $[M+H]^+$: 336.1230, found 336.1223.

5-(2-(*m*-tolyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3p)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3p** as a yellow liquid (36.1 mg, 62% yield).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.65-7.60 (m, 1H), 7.36 (t, J = 7.6 Hz, 1H), 7.32-7.26 (m, 3H), 7.26-7.17 (m, 3H), 6.59 (t, J = 7.6 Hz, 1H), 6.53 (s, 1H), 2.88-2.78 (m, 1H),

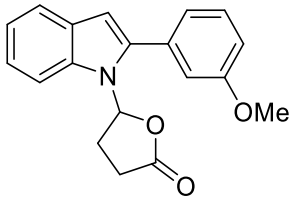
2.76-2.61 (m, 2H), 2.54-2.45 (m, 1H), 2.42 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 141.2, 138.6, 135.3, 131.9, 130.3, 129.4, 129.4, 128.7, 126.6, 122.5, 121.3, 111.3, 104.8, 85.9, 28.9, 26.2, 21.4.

IR ν_{max}(KBr)/cm^{-1} 2947, 2881, 1664, 1610, 1527, 1330, 1146, 947, 610.

HRMS-APCI (m/z): calcd for $C_{19}H_{18}NO_2$, $[M+H]^+$: 292.1332, found 292.1325.

5-(2-(3-methoxyphenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3q)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3q** as a yellow liquid (34.2 mg, 56% yield).

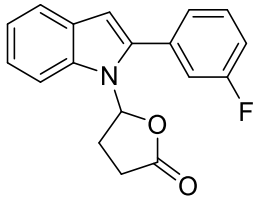
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 7.6 Hz, 1H), 7.38 (t, J = 7.6 Hz, 1H), 7.30 (d, J = 7.6 Hz, 1H), 7.26-7.18 (m, 2H), 7.10-7.02 (m, 2H), 6.98 (dd, J = 8.4, 2.8 Hz, 1H), 6.61 (t, J = 7.6 Hz, 1H), 6.56 (s, 1H), 3.85 (s, 3H), 2.92-2.81 (m, 1H), 2.81-2.64 (m, 2H), 2.58-2.48 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 159.8, 140.9, 135.4, 133.2, 129.9, 129.4, 122.7, 121.9, 121.4, 115.0, 114.3, 111.3, 105.0, 85.9, 55.3, 28.9, 26.2.

IR ν_{max}(KBr)/cm^{-1} 2947, 2890, 1750, 1640, 1525, 1330, 1114, 927, 675.

HRMS-APCI (m/z): calcd for $C_{19}H_{18}NO_3$, $[M+H]^+$: 308.1281, found 308.1274.

5-(2-(3-fluorophenyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3r)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3r** as a yellow liquid (38.4 mg, 65% yield).

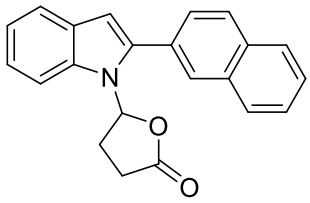
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65-7.63 (m, 1H), 7.47-7.41 (m, 1H), 7.32-7.24 (m, 3H), 7.24-7.19 (m, 2H), 7.13 (td, J = 8.4, 2.6, 1.0 Hz, 1H), 6.60-6.54 (m, 2H), 2.91-2.85 (m, 1H), 2.85-2.67 (m, 2H), 2.59-2.50 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3, 162.7 (d, J = 246.4 Hz), 139.6, 139.6, 135.4, 133.9 (d, J = 8.2 Hz), 130.4 (d, J = 8.4 Hz), 129.3, 125.2 (d, J = 3.0 Hz), 123.0, 121.5, 116.4 (d, J = 22.0 Hz), 115.6 (d, J = 20.9 Hz), 111.5, 105.5, 85.8, 28.9, 26.1.

IR ν_{max}(KBr)/cm^{-1} 2929, 2856, 1785, 1613, 1583, 1335, 1151, 939, 695.

HRMS-APCI (m/z): calcd for $C_{18}H_{15}FNO_2$, $[M+H]^+$: 296.1081, found 296.1076.

5-(2-(naphthalen-2-yl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3s)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3s** as a yellow solid (47.1 mg, 72% yield).

m.p. = 101.7-102.3 ℃

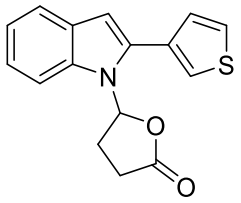
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98-7.95 (m, 1H), 7.94-7.87 (m, 3H), 7.66 (dd, J = 7.2, 1.6 Hz, 1H), 7.59 (dd, J = 8.4, 2.0 Hz, 1H), 7.56-7.52 (m, 2H), 7.32 (d, J = 8.0 Hz, 1H), 7.27-7.19 (m, 2H), 6.67-6.60 (m, 2H), 2.92-2.82 (m, 1H), 2.78-2.59 (m, 2H), 2.51-2.42 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 141.1, 135.5, 133.2, 133.0, 129.6, 129.3, 128.8, 128.6, 128.2, 127.8, 126.9, 126.9, 126.9, 122.8, 121.5, 121.4, 111.4, 105.4, 86.0, 29.0, 26.2.

IR ν_{max}(KBr)/cm^{-1} 2925, 2836, 1772, 1608, 1502, 1361, 1162, 942, 635.

HRMS-APCI (m/z): calcd for $C_{22}H_{18}NO_2$, $[M+H]^+$: 328.1332, found 328.1327.

5-(2-(thiophen-3-yl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3t)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3t** as a yellow liquid (36.3 mg, 64% yield).

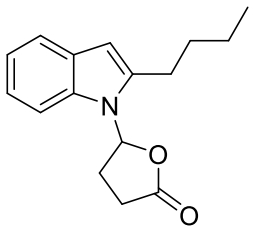
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63-7.61 (m, 1H), 7.46 (dd, J = 4.8, 3.2 Hz, 1H), 7.43 (dd, J = 3.2, 1.6 Hz, 1H), 7.30-7.27 (m, 1H), 7.26-7.17 (m, 3H), 6.65 (t, J = 7.6 Hz, 1H), 6.57 (s, 1H), 2.93-2.83 (m, 1H), 2.82-2.65 (m, 2H), 2.60-2.51 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 135.7, 135.2, 132.4, 129.3, 128.5, 126.7, 125.0, 122.7, 121.3, 121.3, 111.1, 105.1, 85.7, 8.9, 26.2.

IR ν_{max}(KBr)/cm^{-1} 2926, 2853, 1782, 1457, 1334, 1176, 1019, 939, 745.

HRMS-APCI (m/z): calcd for $C_{16}H_{14}NO_2S$, $[M+H]^+$: 284.0740, found 284.0734.

5-(2-butyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3u)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3u** as a yellow liquid (27.8 mg, 54% yield).

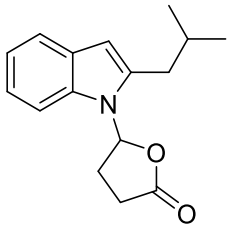
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55-7.51 (m, 1H), 7.19-7.09 (m, 3H), 6.58 (t, J = 7.6 Hz, 1H), 6.30 (s, 1H), 3.03-2.85 (m, 3H), 2.77-2.73 (m, 2H), 2.71-2.64 (m, 1H), 1.75-1.67 (m, 2H), 1.45 (q, J = 7.2 Hz, 2H), 0.97 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 140.5, 134.6, 129.5, 121.7, 120.7, 120.6, 110.7, 102.1, 85.1, 30.8, 29.1, 26.8, 26.6, 22.5, 13.8.

IR ν_{max}(KBr)/cm^{-1} 2929, 2861, 1782, 1682, 1455, 1349, 1115, 938, 746.

HRMS-APCI (m/z): calcd for $C_{16}H_{18}NO_2$, $[M-H]^-$: 256.1343, found 256.1342.

5-(2-isobutyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3v)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3v** as a black solid (25.2 mg, 49% yield).

m.p. = 103.1-103.8 ℃.

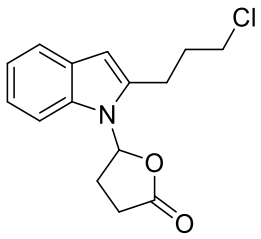
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57-7.51 (m, 1H), 7.18-7.10 (m, 3H), 6.53 (t, J = 7.2 Hz, 1H), 6.28 (s, 1H), 3.01-2.94 (m, 1H), 2.93-2.80 (m, 2H), 2.70-2.57 (m, 3H), 1.98-1.88 (m, 1H), 0.98 (t, J = 6.4 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 139.4, 134.4, 129.5, 121.6, 120.7, 120.6, 110.8, 103.3, 85.2, 36.3, 29.1, 28.4, 26.4, 22.6, 22.5.

IR ν_{max}(KBr)/cm^{-1} 2958, 2869, 1783, 1629, 1459, 1336, 1268, 1035, 784.

HRMS-APCI (m/z): calcd for $C_{16}H_{18}NO_2$, $[M-H]^-$: 256.1343, found 256.1343.

5-(2-(3-chloropropyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3w)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3w** as a black solid (30.0 mg, 54% yield).

m.p. = 121.8-122.2 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.51 (m, 1H), 7.19-7.11 (m, 3H), 6.57 (t, J = 7.6 Hz, 1H), 6.32 (s, 1H), 3.63-3.57 (m, 2H), 2.98-2.80 (m, 5H), 2.72-2.62 (m, 1H), 2.19-2.12 (m, 2H).

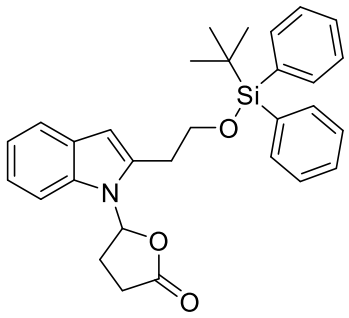
^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 138.4, 134.6, 129.2, 122.0, 120.9, 120.7, 110.7,

102.7, 85.0, 44.0, 31.3, 29.0, 26.5, 24.0.

IR ν_{max}(KBr)/cm^{-1} 2926, 2850, 1676, 1628, 1437, 1355, 1114, 866, 747.

HRMS-APCI (m/z): calcd for $C_{15}H_{17}ClNO_2$, $[M+H]^+$: 278.0942, found 278.0937.

5-(2-(2-((*tert*-butyldiphenylsilyl)oxy)ethyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3x)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3x** as a gray solid (49.3 mg, 51% yield).

m.p. = 157.7-158.2 ℃.

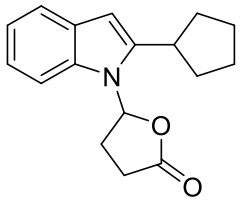
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67-7.63 (m, 2H), 7.62-7.58 (m, 2H), 7.52-7.48 (m, 1H), 7.44-7.40 (m, 2H), 7.37 (d, J = 7.6 Hz, 2H), 7.34-7.29 (m, 2H), 7.13-7.06 (m, 3H), 6.27 (s, 1H), 6.15 (t, J = 7.6 Hz, 1H), 4.00-3.86 (m, 2H), 3.01 (t, J = 7.2 Hz, 2H), 2.86-2.77 (m, 1H), 2.74-2.58 (m, 2H), 2.29-2.19 (m, 1H), 1.07 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 136.9, 135.5, 134.3, 133.5, 133.3, 129.8, 129.7, 129.4, 127.8, 127.7, 121.8, 120.8, 120.7, 110.8, 103.4, 85.0, 63.6, 30.6, 28.9, 26.9, 26.2, 19.2.

IR ν_{max}(KBr)/cm^{-1} 2934, 2858, 1784, 1628, 1431, 1355, 1110, 824, 700.

HRMS-APCI (m/z): calcd for $C_{30}H_{32}NO_3Si$, $[M-H]^-$: 482.2157, found 482.2159.

5-(2-cyclopentyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3y)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3y** as a gray solid (25.9 mg, 48% yield).

m.p. = 124.3-124.9 ℃.

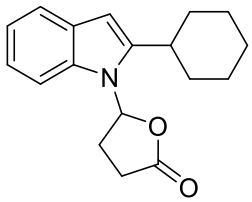
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.50 (m, 1H), 7.16-7.11 (m, 3H), 6.65 (t, J = 6.8 Hz, 1H), 6.31 (s, 1H), 3.19-3.12 (m, 1H), 3.03-2.85 (m, 3H), 2.72-2.62 (m, 1H), 2.17-2.05 (m, 2H), 1.85-1.68 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 144.9, 134.6, 129.5, 121.7, 120.7, 120.7, 110.9, 99.7, 85.3, 37.2, 32.9, 32.6, 29.2, 26.5, 25.1, 25.1.

IR ν_{max}(KBr)/cm^{-1} 2930, 2864, 1783, 1628, 1508, 1336, 1148, 991, 747.

HRMS-APCI (m/z): calcd for $C_{17}H_{18}NO_2$, $[M-H]^-$: 268.1343, found 268,1345.

5-(2-cyclohexyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3z)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3z** as a gray solid (26.1 mg, 46% yield).

m.p. = 128.6.7-129.2 ℃.

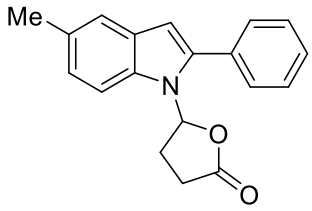
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56-7.51 (m, 1H), 7.16-7.08 (m, 3H), 6.56 (t, J = 7.6 Hz, 1H), 6.27 (s, 1H), 2.98-2.79 (m, 3H), 2.66-2.58 (m, 2H), 2.07 (d, J = 12.0 Hz, 1H), 1.97-1.75 (m, 4H), 1.51-1.26 (m, 5H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 145.9, 134.2, 129.6, 121.5, 120.7, 120.6, 110.9, 99.7, 84.9, 35.9, 33.5, 33.2, 29.1, 26.5, 26.4, 26.3, 26.0.

IR ν_{max}(KBr)/cm^{-1} 2928, 2854, 1782, 1627, 1456, 1336, 1038, 939, 741.

HRMS-APCI (m/z): calcd for $C_{18}H_{20}NO_2$, $[M-H]^-$: 282.1500, found 282.1501.

5-(5-methyl-2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3aa)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3aa** as a yellow solid (40.8 mg, 70% yield).

m.p. = 135.7-136.2 ℃.

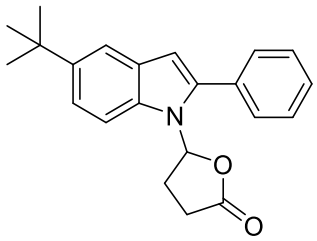
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52-7.38 (m, 6H), 7.18 (d, J = 8.4 Hz, 1H), 7.06 (d, J = 8.4 Hz, 1H), 6.56 (t, J = 7.6 Hz, 1H), 6.48 (s, 1H), 2.91-2.81 (m, 1H), 2.80-2.63 (m, 2H), 2.56-2.48 (m, 1H), 2.45 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 141.2, 133.6, 132.1, 130.8, 129.8, 129.5, 128.8, 128.5, 124.2, 121.1, 111.0, 104.6, 86.0, 29.0, 26.1, 21.3.

IR ν_{max}(KBr)/cm^{-1} 2950, 2859, 1781, 1465, 1331, 1178, 984, 875, 765.

HRMS-APCI (m/z): calcd for $C_{19}H_{18}NO_2$, $[M+H]^+$: 292.1332, found 292.1325.

5-(5-(*tert*-butyl)-2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3ab)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3ab** as a yellow solid (51.4 mg, 77% yield).

m.p. = 138.6-139.1 ℃

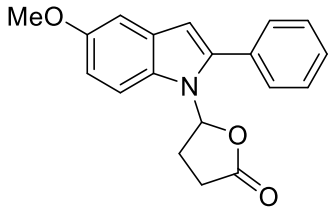
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 1.2 Hz, 1H), 7.49-7.42 (m, 5H), 7.33 (dd, J = 8.8, 2.0 Hz, 1H), 7.24 (d, J = 8.8 Hz, 1H), 6.59-6.51 (m, 2H), 2.91-2.81 (m, 1H), 2.79-2.63 (m, 2H), 2.56-2.47 (m, 1H), 1.39 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 144.4, 141.1, 133.4, 132.1, 129.6, 129.3, 128.8, 128.5, 120.9, 117.3, 110.9, 105.0, 85.9, 34.6, 31.8, 28.9, 26.2.

IR ν_{max}(KBr)/cm^{-1} 2917, 2865, 1785, 1605, 1470, 1332, 1179, 930, 759.

HRMS-APCI (m/z): calcd for $C_{22}H_{22}NO_2$, $[M-H]^-$: 332.1656, found 332.1660.

5-(5-methoxy-2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3ac)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3ac** as a yellow liquid (24.0 mg, 49% yield).

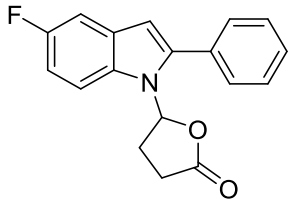
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51-7.41 (m, 5H), 7.19 (d, J = 8.8 Hz, 1H), 7.10 (d, J = 2.4 Hz, 1H), 6.89 (dd, J = 8.8, 2.4 Hz, 1H), 6.54 (t, J = 7.6 Hz, 1H), 6.49 (s, 1H), 3.86 (s, 3H), 2.88-2.82 (m, 1H), 2.81-2.65 (m, 2H), 2.57-2.47 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 155.2, 141.8, 132.0, 130.3, 130.2, 129.5, 128.8, 128.6, 112.4, 112.1, 104.8, 103.3, 86.0, 55.8, 29.0, 26.3.

IR ν_{max}(KBr)/cm^{-1} 2960, 2856, 1765, 1621, 1583, 1472, 1217, 1178, 936, 766.

HRMS-APCI (m/z): calcd for $C_{19}H_{16}NO_3$, $[M-H]^-$: 306.1136, found 306.1138.

5-(5-fluoro-2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3ad)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3ad** as a yellow solid (39.0 mg, 66% yield).

m.p. = 148.7-149.2 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51-7.43 (m, 5H), 7.28 (dd, J = 9.2, 2.4 Hz, 1H), 7.21 (dd, J = 8.8, 4.4 Hz, 1H), 6.98 (td, J = 9.2, 2.8 Hz, 1H), 6.55 (t, J = 8.0 Hz, 1H), 6.52

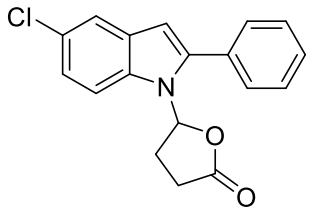
(s, 1H), 2.88-2.80 (m, 1H), 2.79-2.65 (m, 2H), 2.59-2.49 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3, 158.6 (d, J = 235.8 Hz), 142.7, 131.8, 131.6, 130.1 (d, J = 10.0 Hz), 129.5, 128.9, 128.9, 112.0 (d, J = 9.4 Hz), 110.8 (d, J = 25.8 Hz), 106.4 (d, J = 23.2 Hz), 104.8 (d, J = 4.3 Hz), 85.8, 28.9, 26.3.

IR ν_{max}(KBr)/cm^{-1} 2927, 2854, 1785, 1688, 1460, 1347, 1264, 1027, 769.

HRMS-APCI (m/z): calcd for $C_{18}H_{13}FNO_2$, $[M-H]^-$: 294.0936, found 294.0939.

5-(5-chloro-2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3ae)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3ae** as a yellow solid (38.0 mg, 61% yield).

m.p. = 146.7-147.3 ℃.

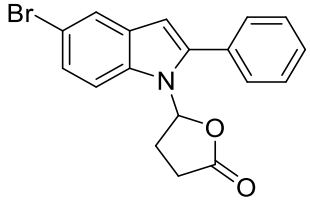
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (dd, J = 2.0, 0.8 Hz, 1H), 7.53-7.43 (m, 5H), 7.24-7.17 (m, 2H), 6.55 (t, J = 7.6 Hz, 1H), 6.50 (s, 1H), 2.85-2.79 (m, 1H), 2.79-2.66 (m, 2H), 2.60-2.50 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 142.4, 133.6, 131.4, 130.6, 129.6, 129.0, 128.9, 127.1, 122.8, 120.8, 112.3, 104.4, 85.7, 28.9, 26.4.

IR ν_{max}(KBr)/cm^{-1} 2924, 2856, 1784, 1629, 1438, 1355, 1113, 869, 757.

HRMS-APCI (m/z): calcd for $C_{18}H_{13}ClNO_2$, $[M-H]^-$: 310.0640, found 310.0642.

5-(5-bromo-2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3af)



The reaction was conducted on a 0.20 mmol scale with the general procedure.

Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3af** as a yellow solid (45.6 mg, 64% yield).

m.p. = 153.7-154.2 ℃.

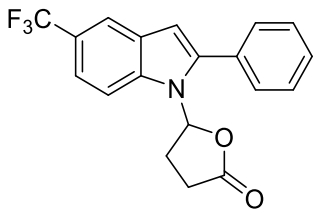
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, J = 1.2 Hz, 1H), 7.48-7.44 (m, 5H), 7.30 (dd, J = 8.8, 2.0 Hz, 1H), 7.15 (d, J = 8.8 Hz, 1H), 6.54 (t, J = 7.2 Hz, 1H), 6.48 (s, 1H), 2.82-2.76 (m, 1H), 2.75-2.63 (m, 2H), 2.57-2.48 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 142.2, 133.9, 131.3, 131.1, 129.5, 128.9, 128.9, 125.3, 123.8, 114.5, 112.7, 104.2, 85.6, 28.8, 26.3.

IR ν_{max}(KBr)/cm^{-1} 2927, 2853, 1785, 1687, 1452, 1355, 1266, 1166, 771.

HRMS-APCI (m/z): calcd for $C_{18}H_{13}BrNO_2$, $[M-H]^-$: 354.0135, found 354.0135.

5-(2-phenyl-5-(trifluoromethyl)-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3ag)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (3 : 1 v/v) as an eluent provided the titled compound **3ag** as a yellow solid (20.7 mg, 30% yield).

m.p. = 121.1-121.8 ℃.

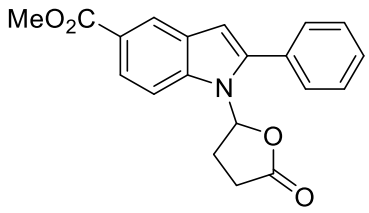
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97-7.90 (m, 1H), 7.51-7.49 (m, 5H), 7.47 (d, J = 1.6 Hz, 1H), 7.38 (d, J = 8.8 Hz, 1H), 6.64 (s, 1H), 6.60 (t, J = 7.6 Hz, 1H), 2.88-2.80 (m, 1H), 2.80-2.69 (m, 2H), 2.65-2.55 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 142.7, 136.6, 131.2, 129.6, 129.1, 129.0, 128.9, 124.9 (q, J = 270.1 Hz), 123.8 (q, J = 319.0 Hz), 119.3 (q, J = 3.5 Hz), 118.9 (q, J = 4.1 Hz), 111.5, 105.4, 85.5, 28.8, 26.5.

IR ν_{max}(KBr)/cm^{-1} 2926, 2859, 1789, 1628, 1445, 1337, 1270, 1116, 759.

HRMS-APCI (m/z): calcd for $C_{19}H_{15}F_3NO_2$, $[M+H]^+$: 346.1049, found 346.1044.

methyl 1-(5-oxotetrahydrofuran-2-yl)-2-phenyl-1*H*-indole-5-carboxylate (3ah)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (3 : 1 v/v) as an eluent provided the titled compound **3ah** as a yellow solid (16.8 mg, 25% yield).

m.p. = 164.3-165.1 °C.

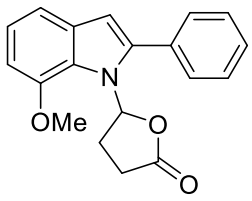
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, J = 1.6 Hz, 1H), 7.95 (dd, J = 8.8, 2.0 Hz, 1H), 7.53-7.43 (m, 5H), 7.32 (d, J = 8.8 Hz, 1H), 6.64 (s, 1H), 6.60 (t, J = 7.6 Hz, 1H), 3.94 (s, 3H), 2.90-2.82 (m, 1H), 2.80-2.67 (m, 2H), 2.65-2.55 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 167.6, 142.3, 137.9, 131.4, 129.6, 129.1, 129.0, 128.9, 123.9, 123.9, 123.4, 110.9, 105.8, 85.5, 52.0, 28.8, 26.5.

IR ν_{max}(KBr)/cm^{-1} 2926, 2851, 1786, 1628, 1437, 1350, 1111, 932, 763.

HRMS-APCI (m/z): calcd for $C_{20}H_{18}NO_4$, $[M+H]^+$: 336.1230, found 336.1223

5-(7-methoxy-2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3ai)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3ai** as a yellow liquid (19.7 mg, 32% yield).

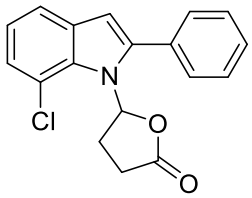
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53-7.42 (m, 5H), 7.26-7.23 (m, 1H), 7.14 (t, J = 7.6 Hz, 1H), 6.75 (d, J = 8.0 Hz, 1H), 6.58 (t, J = 7.6 Hz, 1H), 6.52 (s, 1H), 3.95 (s, 3H), 2.80-2.68 (m, 2H), 2.66-2.55 (m, 1H), 2.50-2.40 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 175.9, 146.6, 142.1, 132.1, 129.5, 128.8, 128.5, 125.5, 122.2, 113.9, 104.7, 104.6, 85.8, 55.8, 29.7, 28.7.

IR ν_{max}(KBr)/cm^{-1} 2929, 2845, 1782, 1581, 1420, 1326, 1151, 937, 733.

HRMS-APCI (m/z): calcd for $C_{19}H_{18}NO_3$, $[M+H]^+$: 308.1281, found 308.1278.

5-(7-chloro-2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3aj)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (2 : 1 v/v) as an eluent provided the titled compound **3aj** as a yellow solid (28.1 mg, 45% yield).

m.p. = 166.3-166.8 ℃.

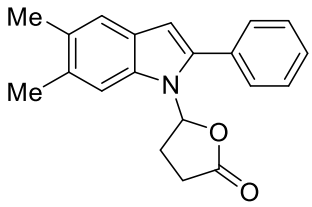
^{1}H NMR (400 MHz, CD_3Cl) δ 7.54 (d, J = 8.4 Hz, 1H), 7.50-7.43 (m, 5H), 7.29-7.28 (m, 1H), 7.17 (dd, J = 8.4, 1.6 Hz, 1H), 6.56-6.50 (m, 2H), 2.86-2.80 (m, 1H), 2.79-2.65 (m, 2H), 2.58-2.49 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.1, 141.7, 135.7, 131.5, 129.6, 128.9, 128.9, 128.4, 127.9, 122.1, 122.0, 111.4, 104.9, 85.6, 28.8, 26.3.

IR ν_{max}(KBr)/cm^{-1} 2926, 2859, 1785, 1629, 1453, 1339, 1269, 1143, 760.

HRMS-APCI (m/z): calcd for $C_{18}H_{15}ClNO_2$, $[M+H]^+$: 312.0786, found 312.0780.

5-(5,6-dimethyl-2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3ak)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3ak** as a yellow solid (42.8 mg, 70% yield).

Yield: 70% (42.8 mg) as a yellow solid;

m.p. = 144.7-145.2 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50-7.37 (m, 6H), 7.06 (s, 1H), 6.54 (t, J = 7.6 Hz, 1H),

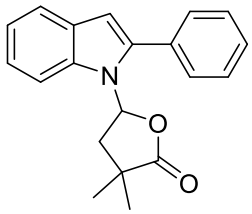
6.44 (s, 1H), 2.89-2.81 (m, 1H), 2.80-2.62 (m, 2H), 2.51-2.42 (m, 1H), 2.36 (d, *J* = 13.2 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 140.3, 134.4, 132.2, 131.7, 130.1, 129.5, 128.7, 128.3, 127.8, 121.4, 111.9, 104.5, 86.1, 29.0, 26.1, 20.8, 19.9.

IR ν_{max}(KBr)/cm^{-1} 2927, 2859, 1784, 1603, 1465, 1335, 1157, 929, 765.

HRMS-APCI (m/z): calcd for $C_{20}H_{18}NO_2$, $[M-H]^-$: 304.1343, found 304.1346.

3,3-dimethyl-5-(2-phenyl-1*H*-indol-1-yl)dihydrofuran-2(3*H*)-one (3an)



The reaction was conducted on a 0.20 mmol scale with the general procedure. Purification by column chromatography on silica gel using Ether : Hexane (1 : 1 v/v) as an eluent provided the titled compound **3ap** as a yellow solid (55.0 mg, 90% yield).

m.p. = 140.8.7-141.2 ℃.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.6 Hz, 1H), 7.54-7.42 (m, 5H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.25-7.18 (m, 2H), 6.62-6.51 (m, 2H), 2.83 (dd, *J* = 13.6, 9.6 Hz, 1H), 2.25 (dd, *J* = 13.6, 6.8 Hz, 1H), 1.32 (d, *J* = 6.0 Hz, 6H).

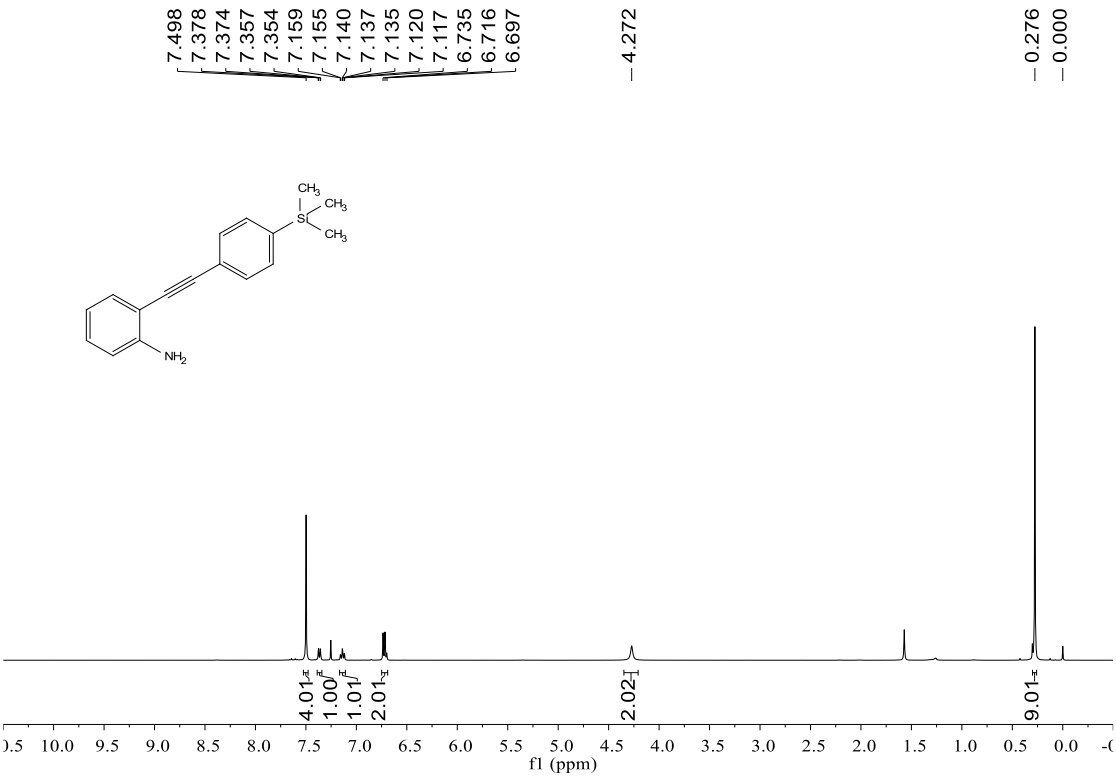
^{13}C NMR (100 MHz, $CDCl_3$) δ 179.6, 141.0, 135.7, 132.2, 129.5, 129.4, 128.9, 128.6, 122.6, 121.3, 121.3, 111.6, 105.2, 83.1, 41.3, 41.2, 25.5, 24.0.

IR ν_{max}(KBr)/cm^{-1} 2968, 2865, 1779, 1629, 1451, 1350, 1116, 1020, 749.

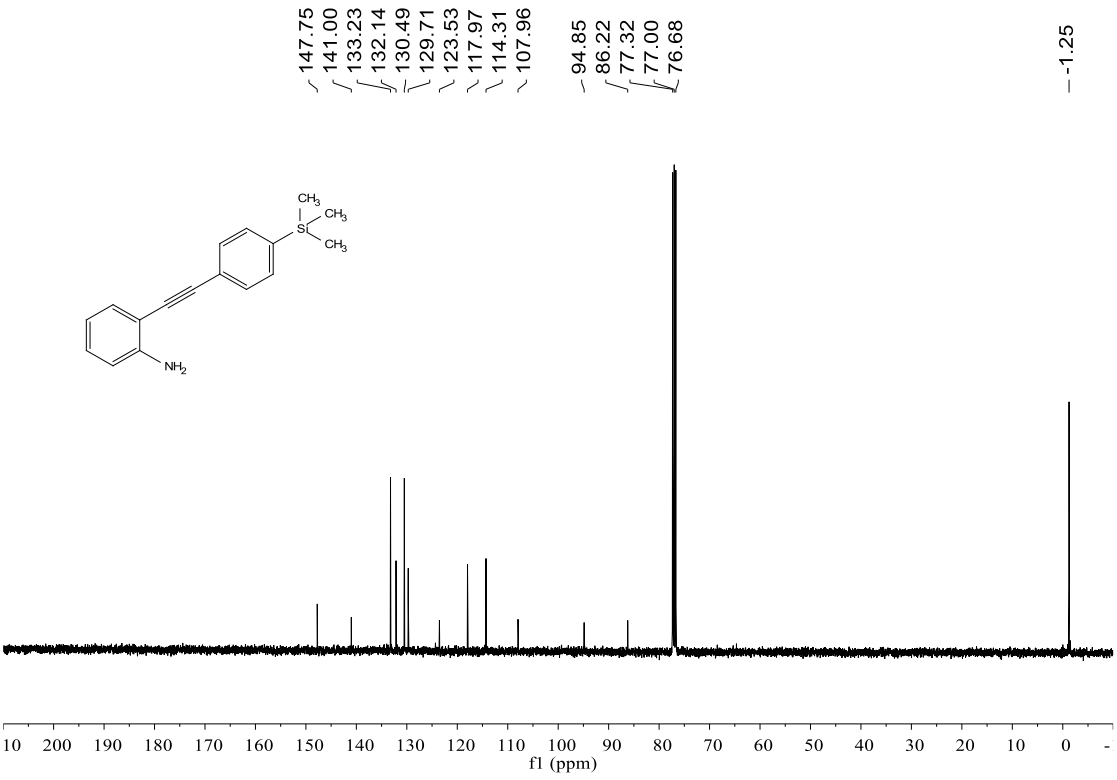
HRMS-APCI (m/z): calcd for $C_{20}H_{20}NO_2$, $[M+H]^+$: 306.1489, found 306.1482.

9. NMR spectra

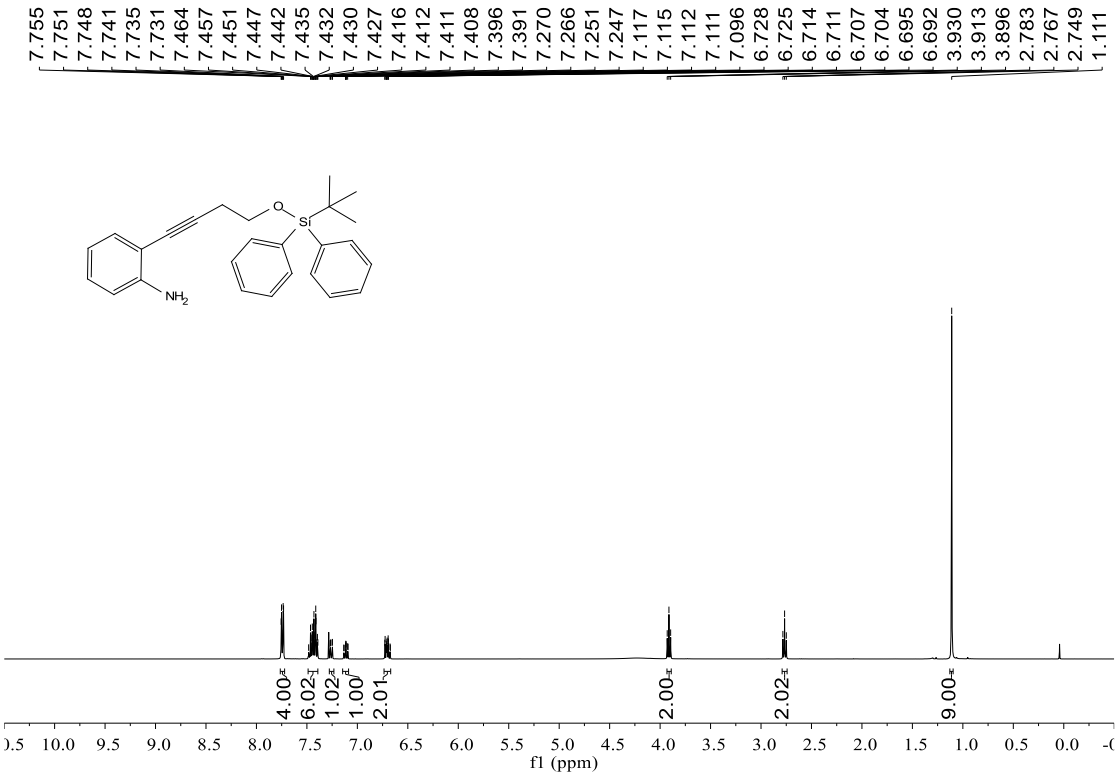
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1f



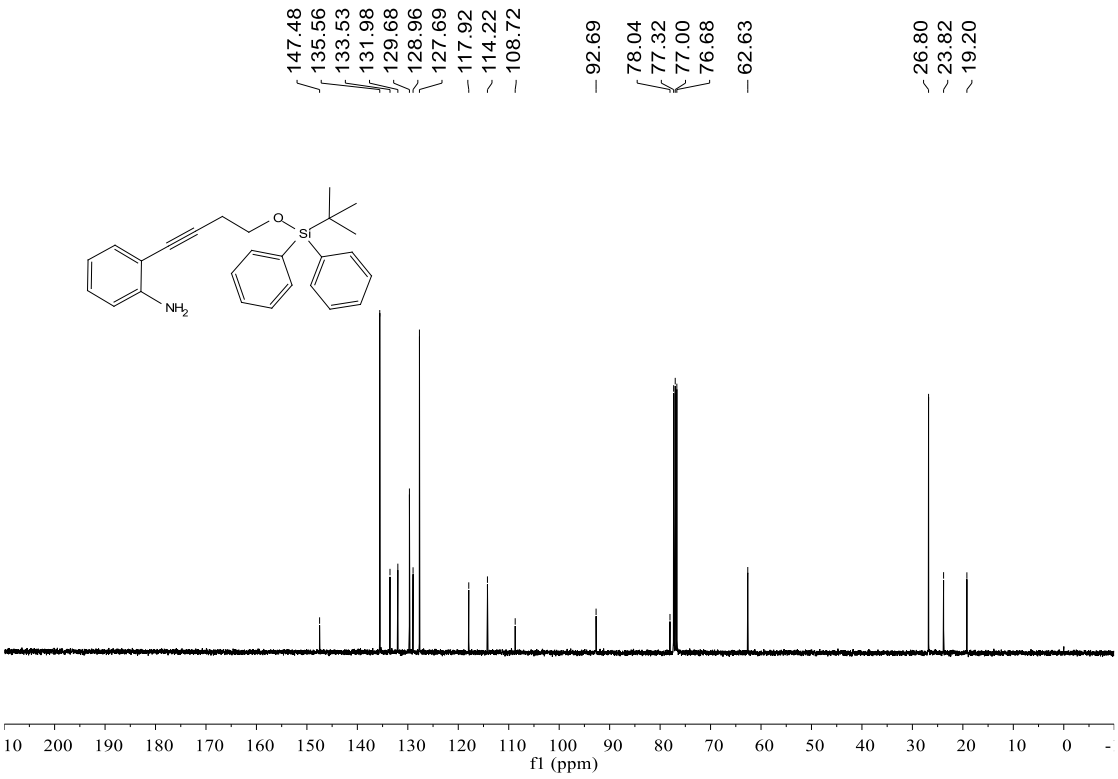
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 1f



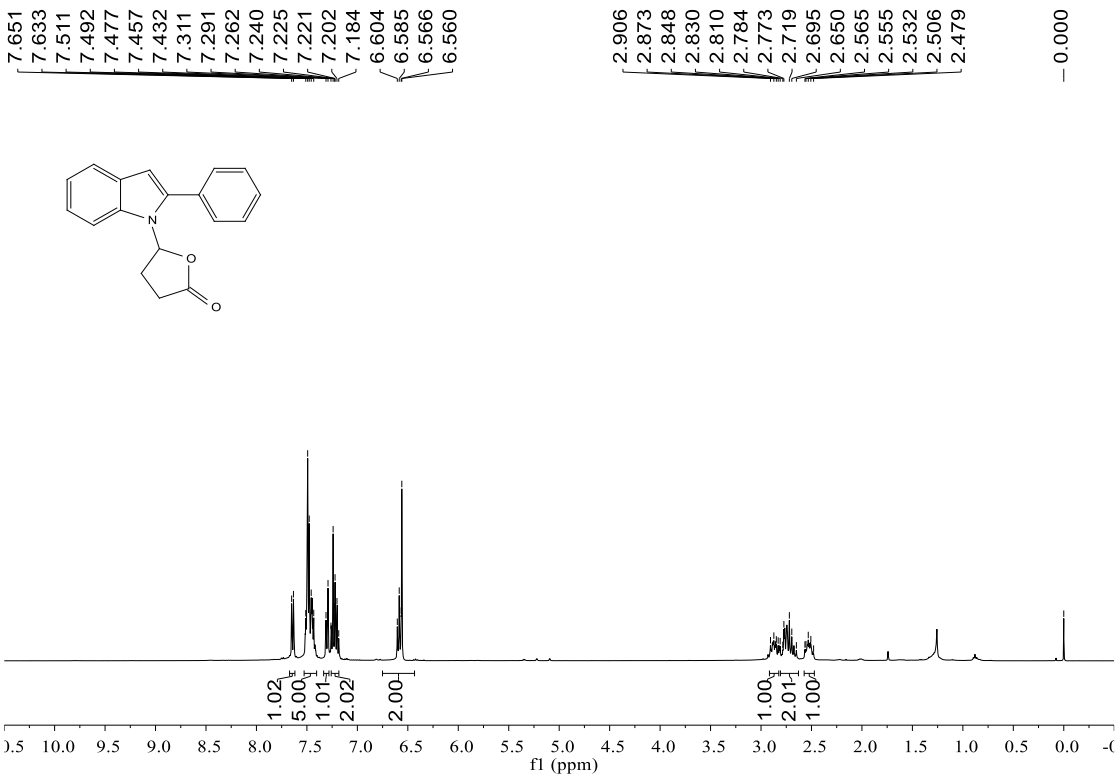
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1x



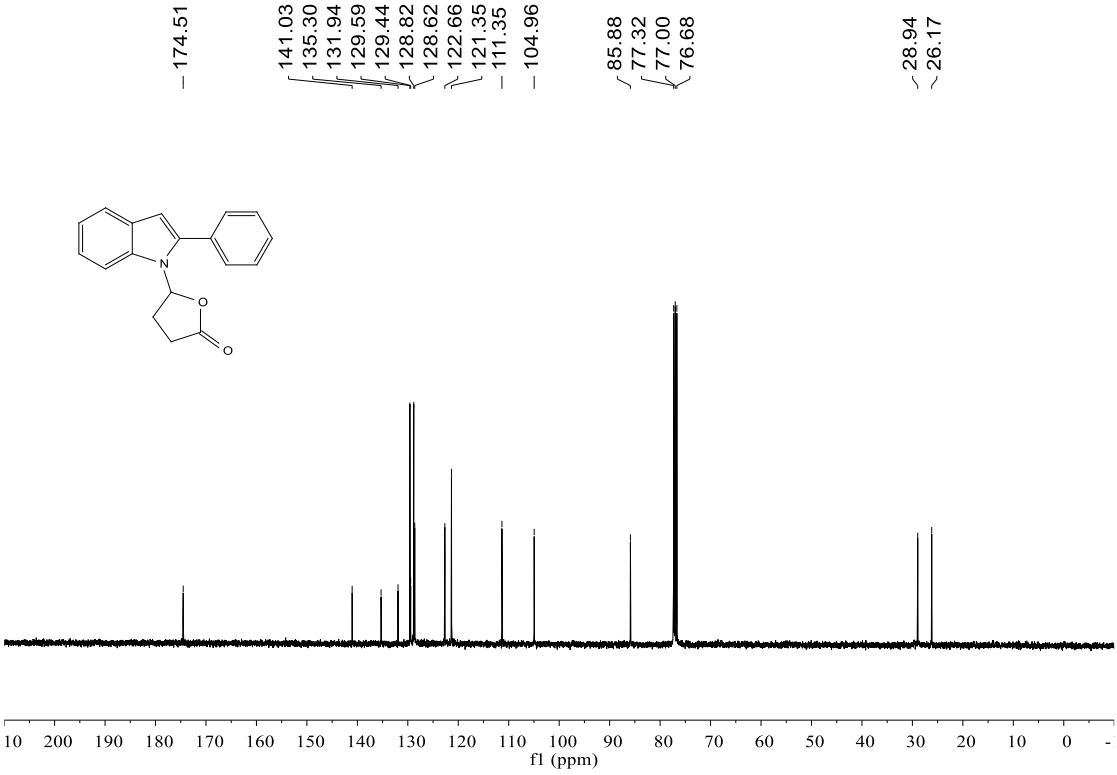
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 1x



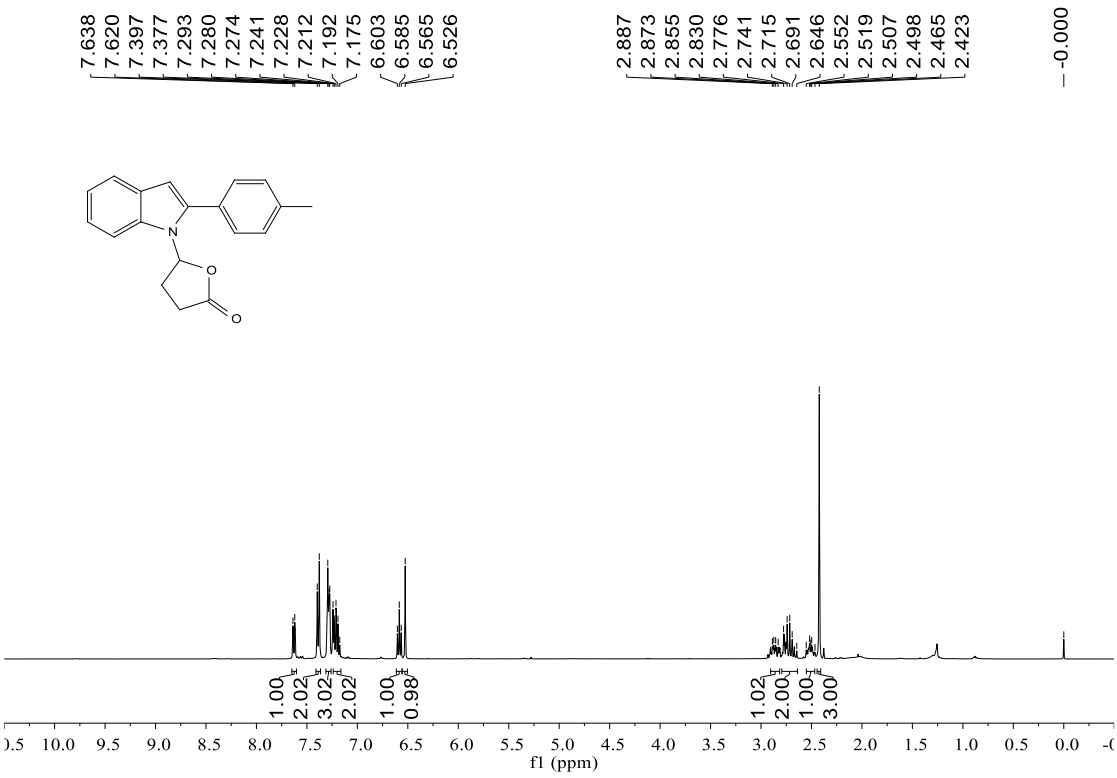
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3a



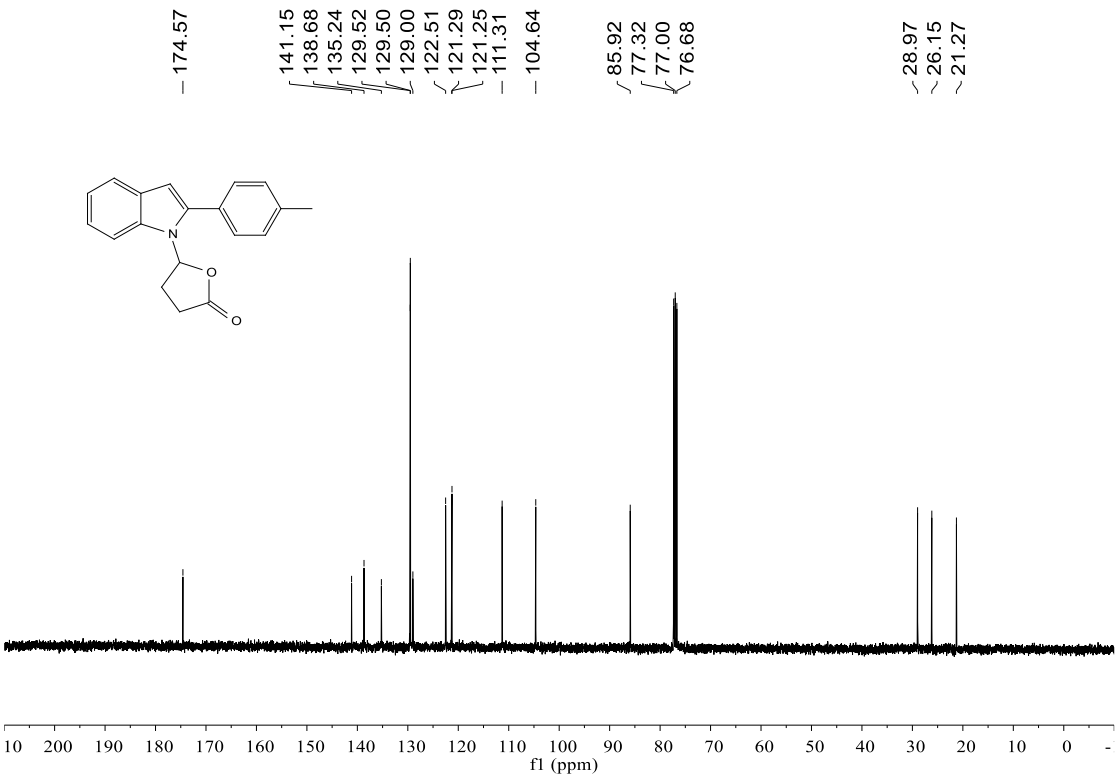
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3a



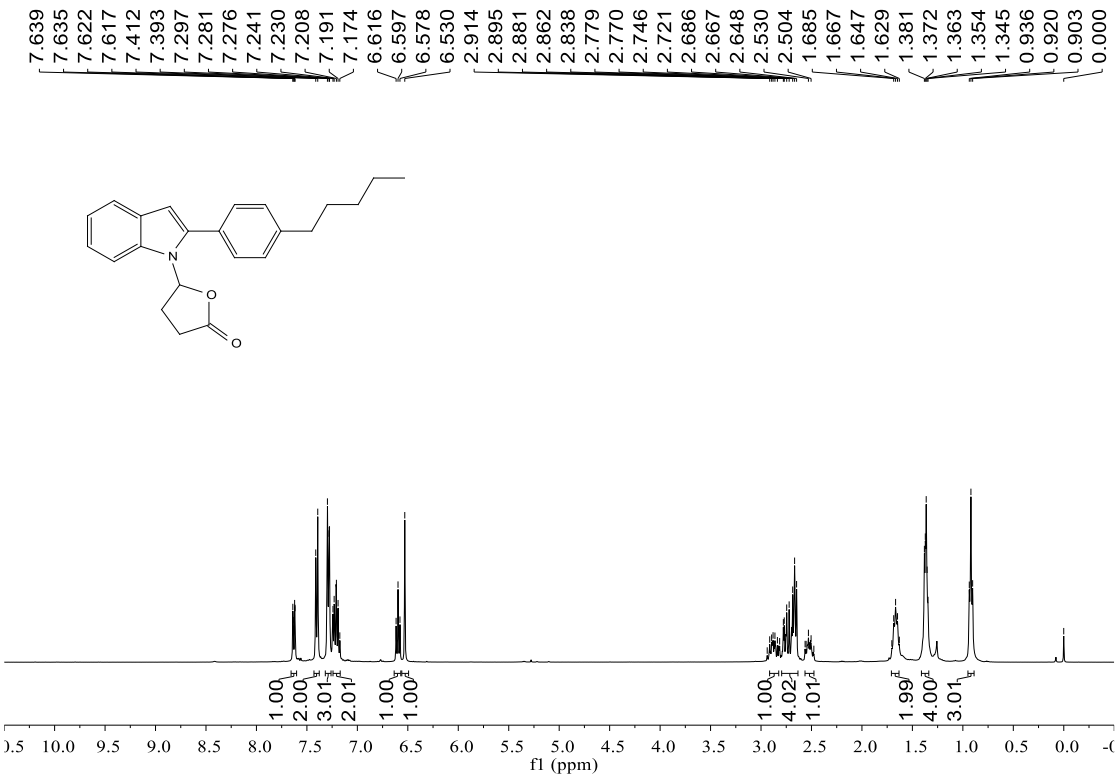
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3b



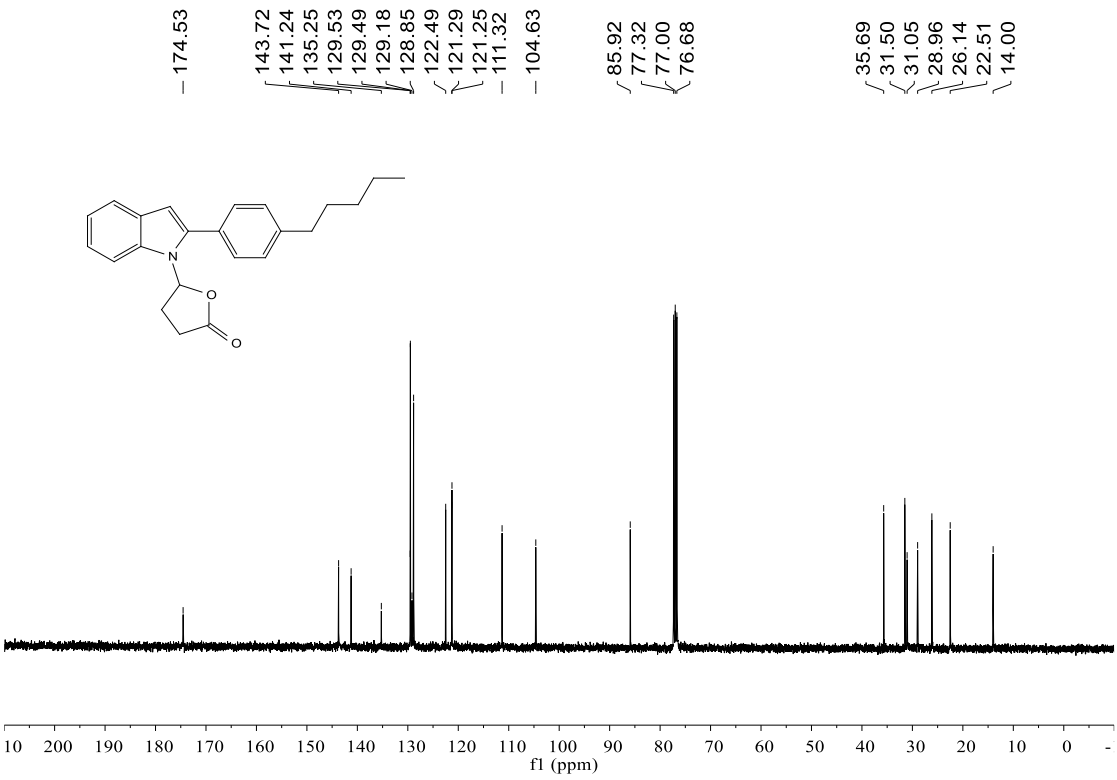
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3b



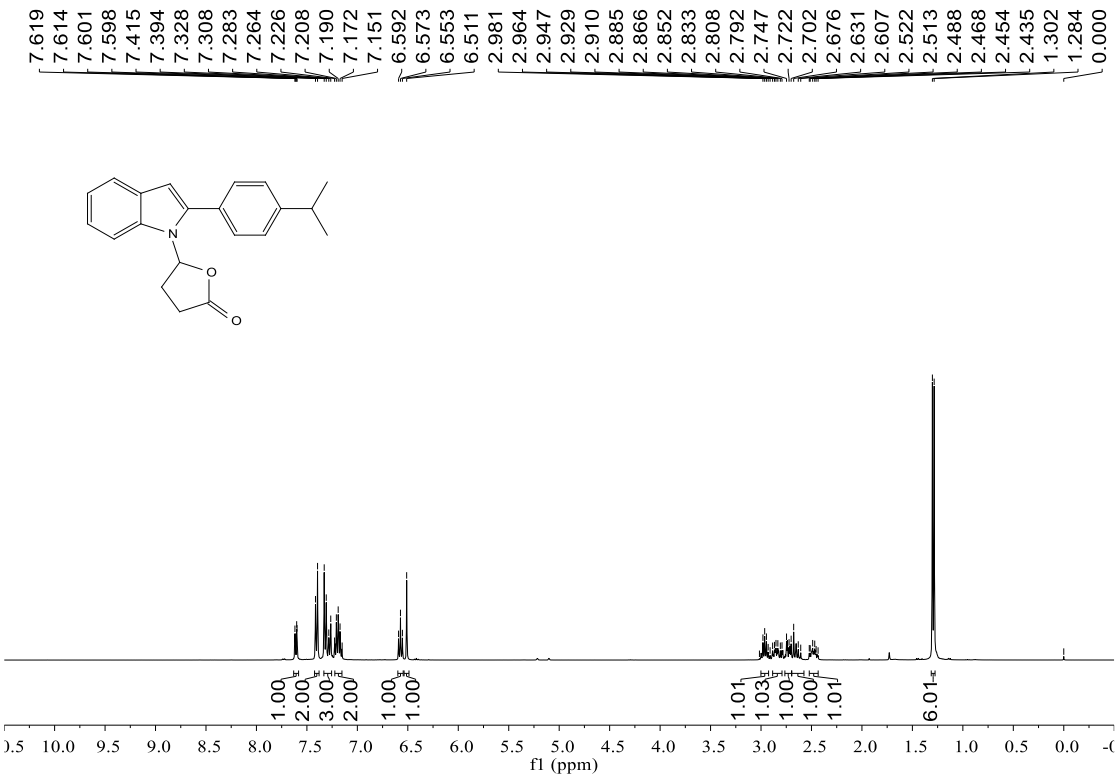
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3c



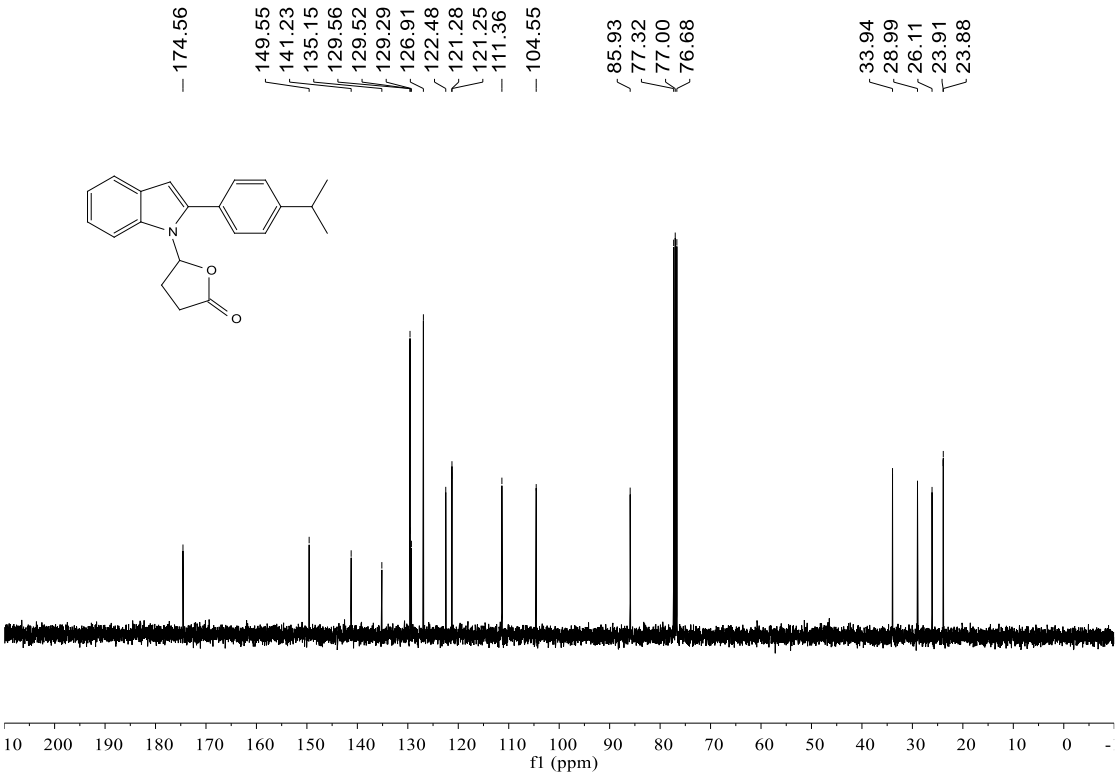
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3c



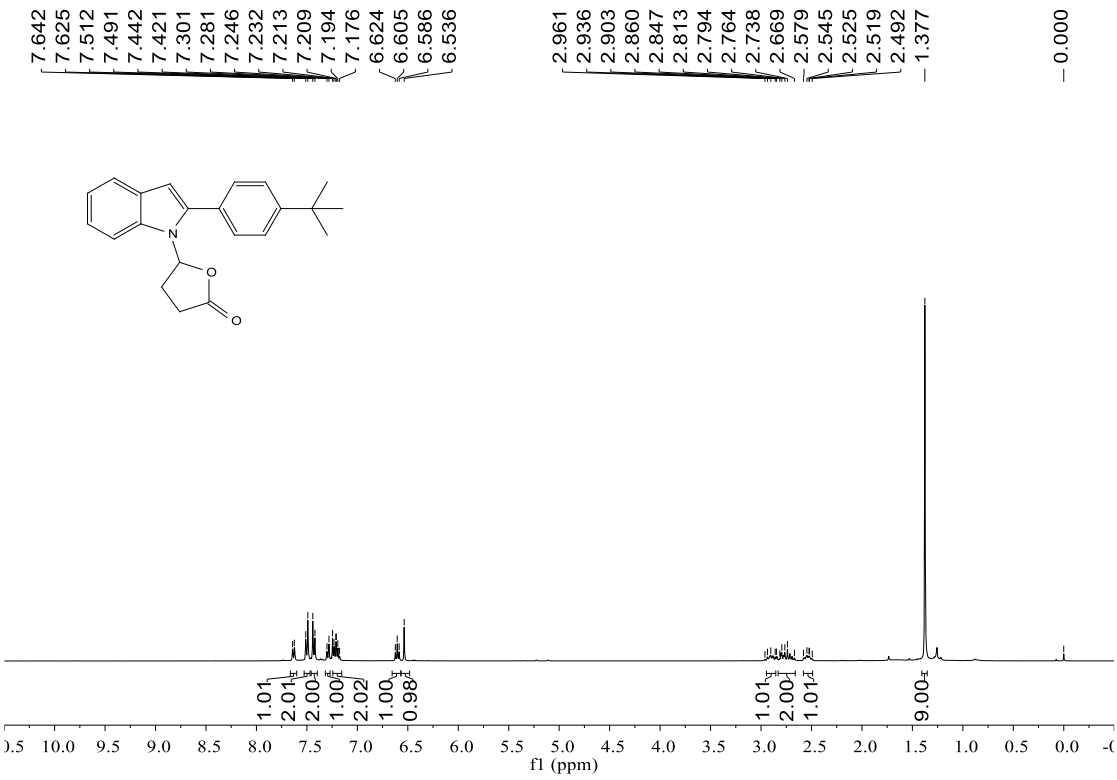
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3d



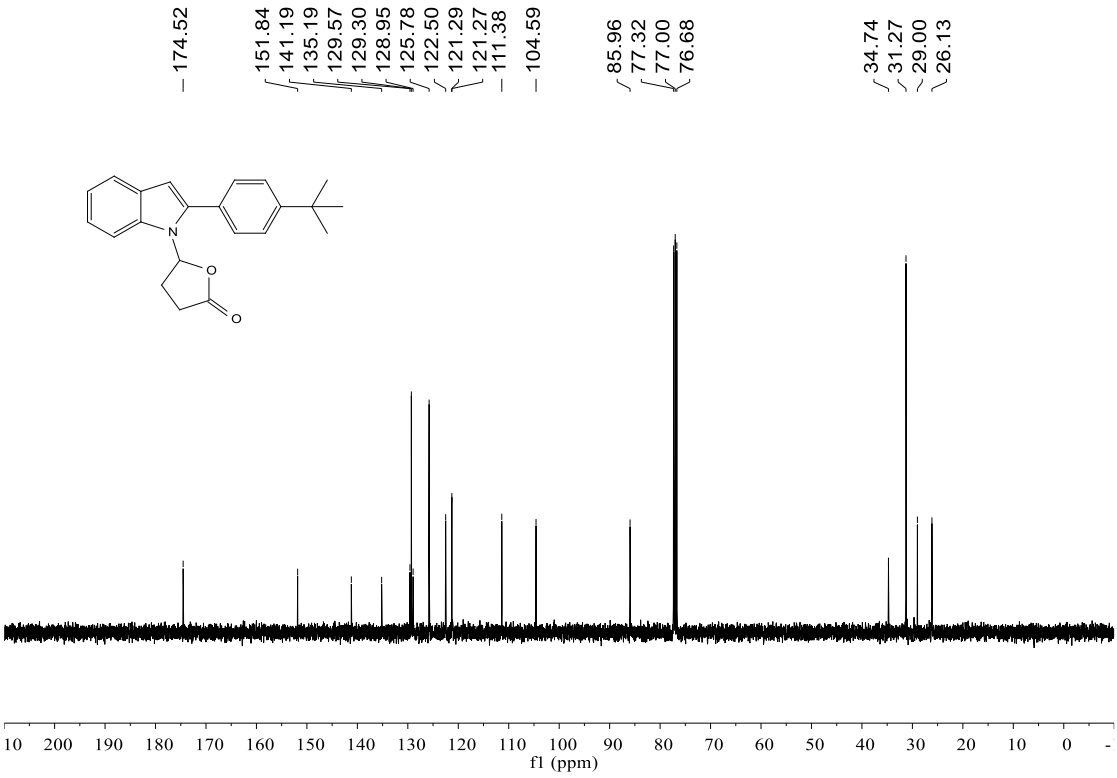
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3d



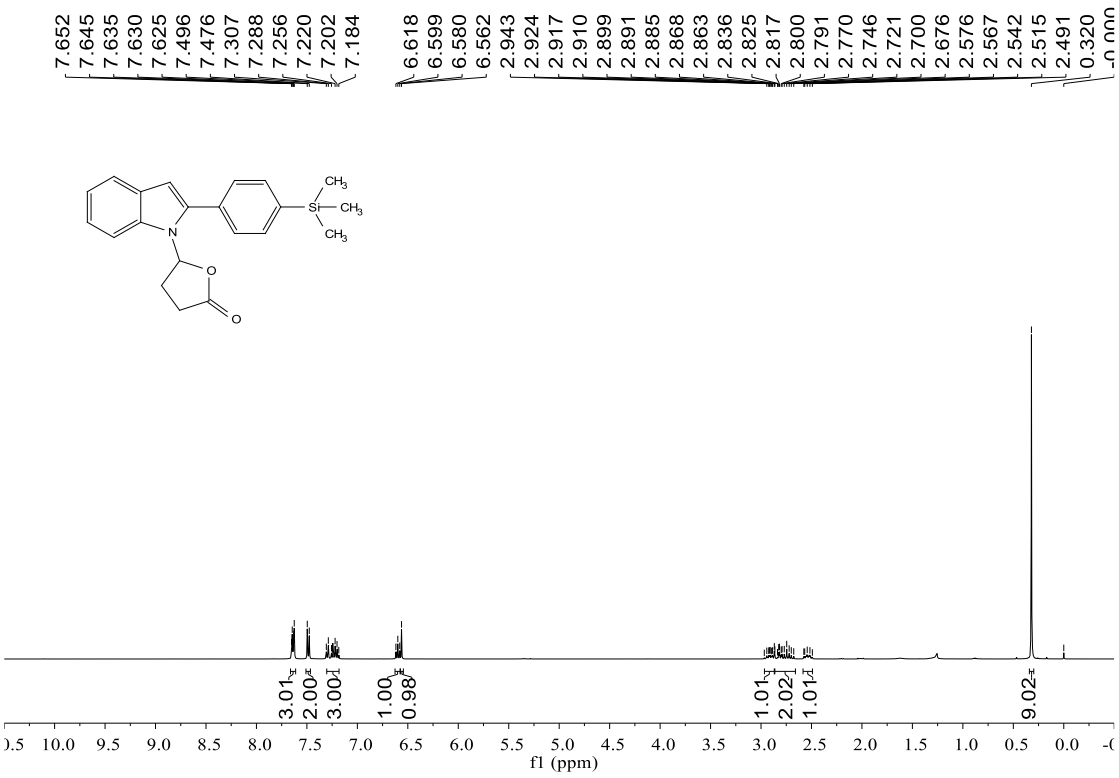
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3e



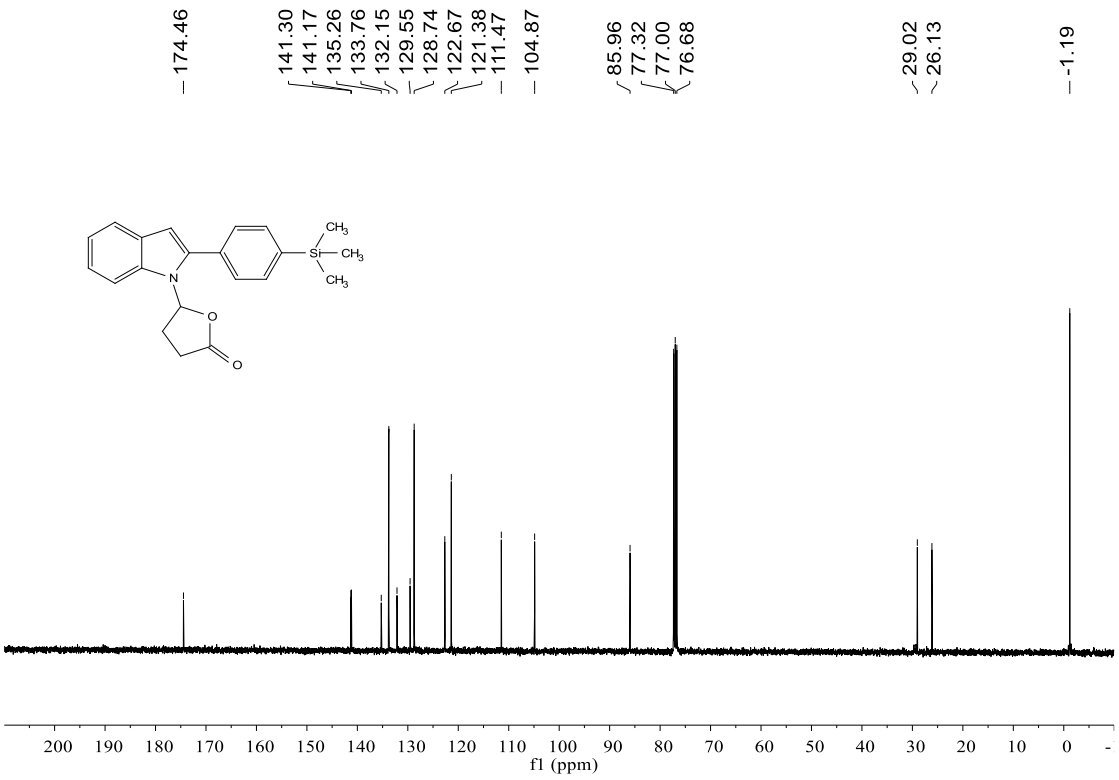
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3e



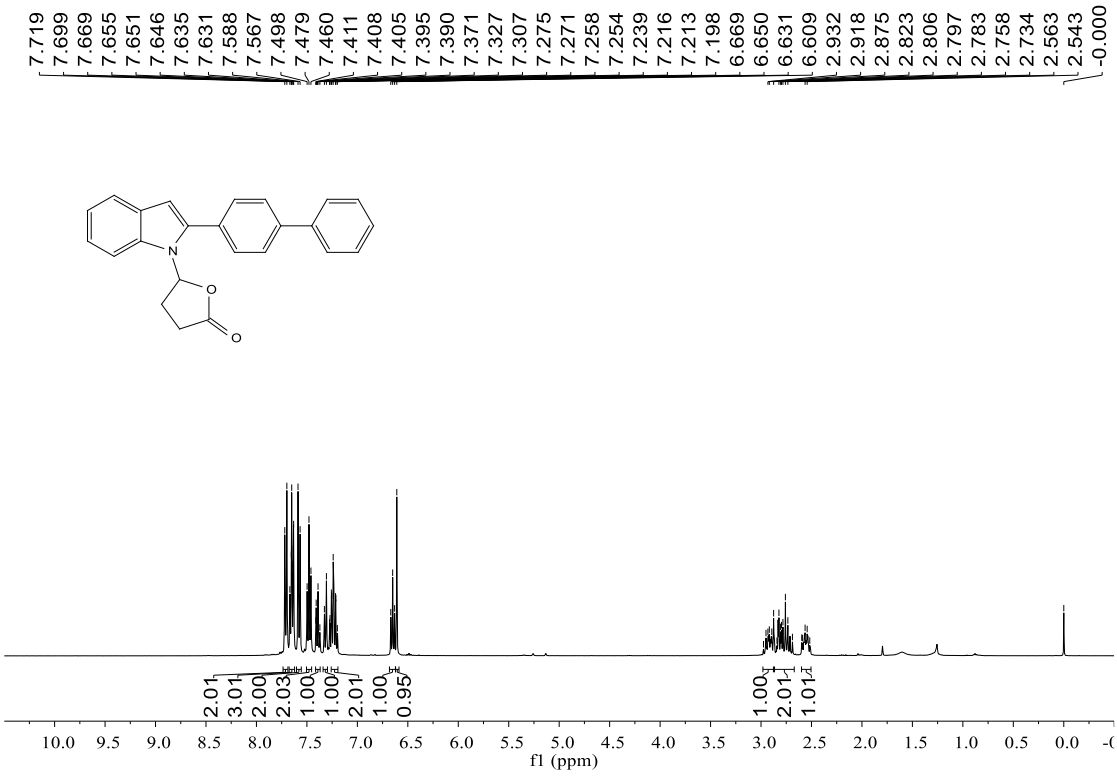
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3f



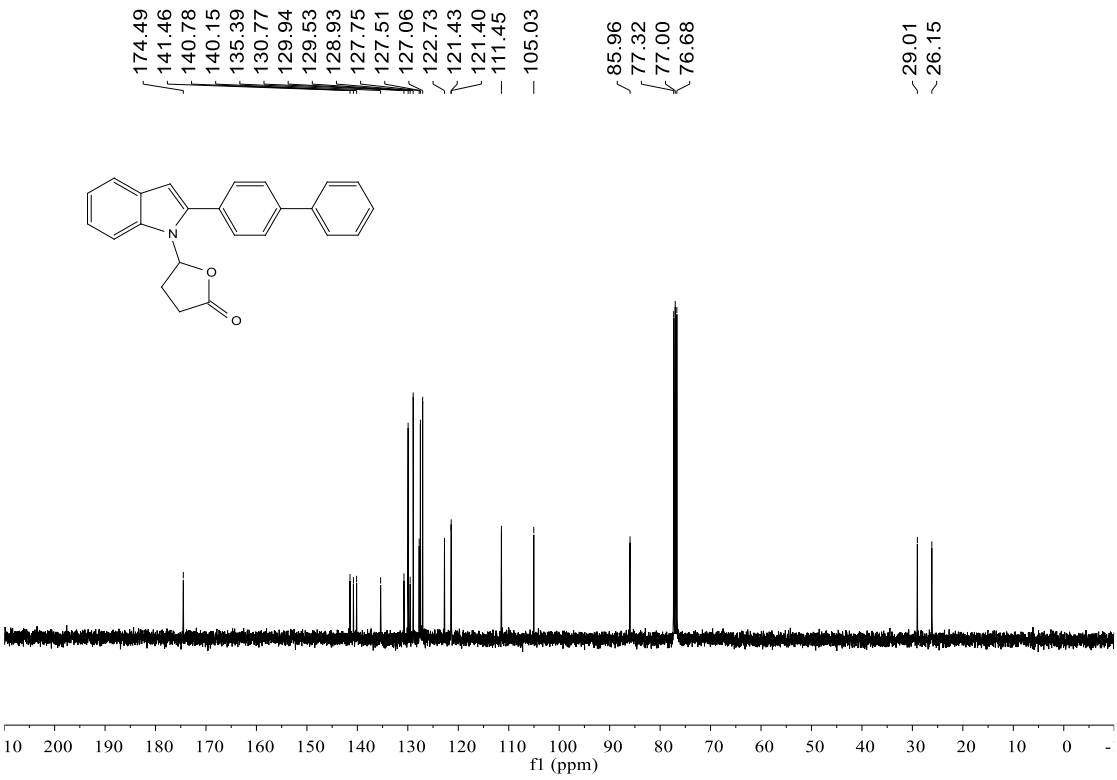
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3f



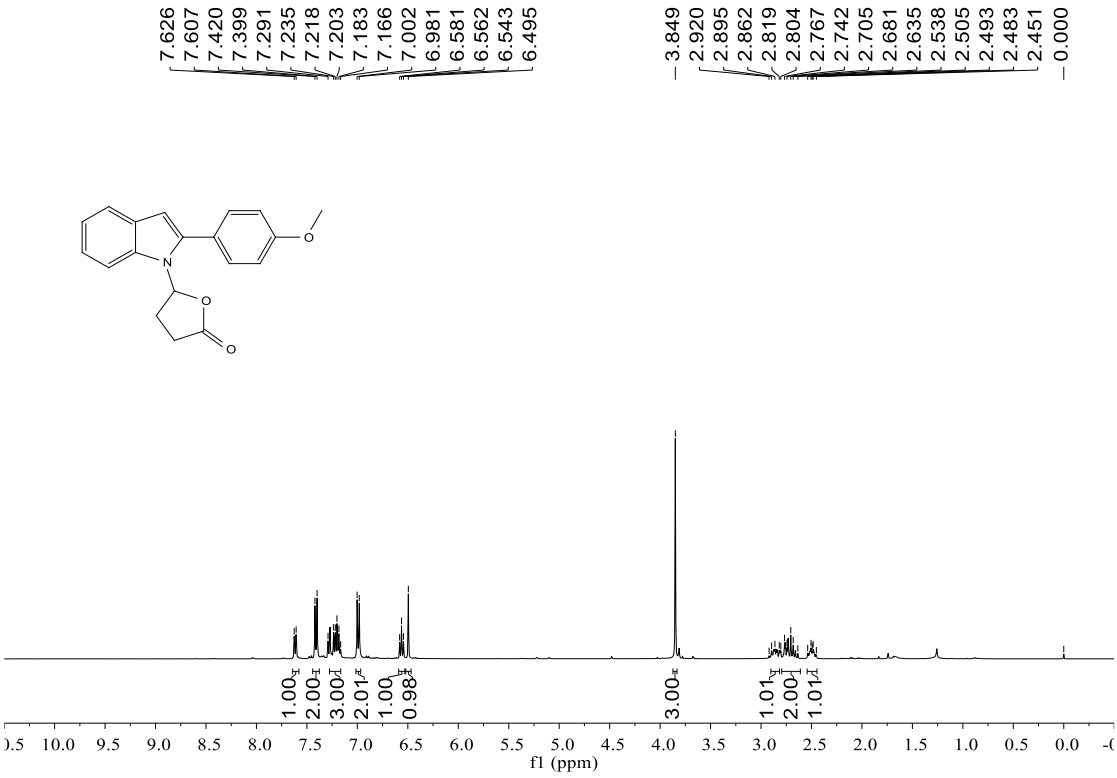
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3g



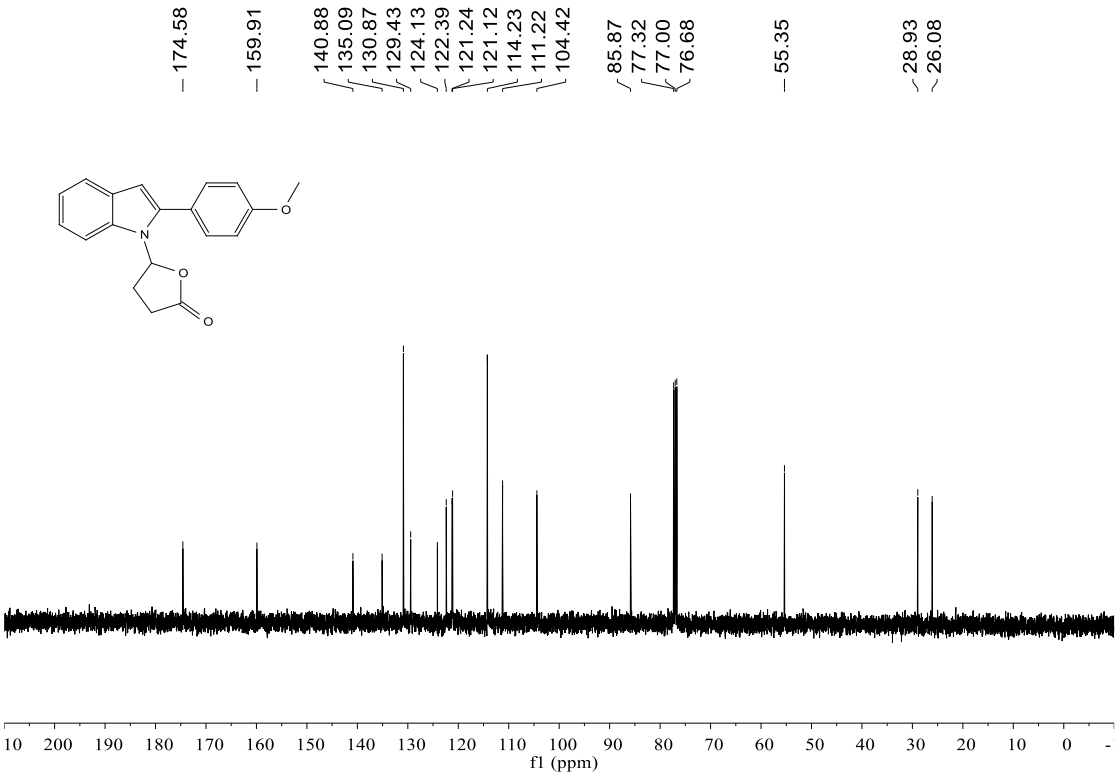
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3g



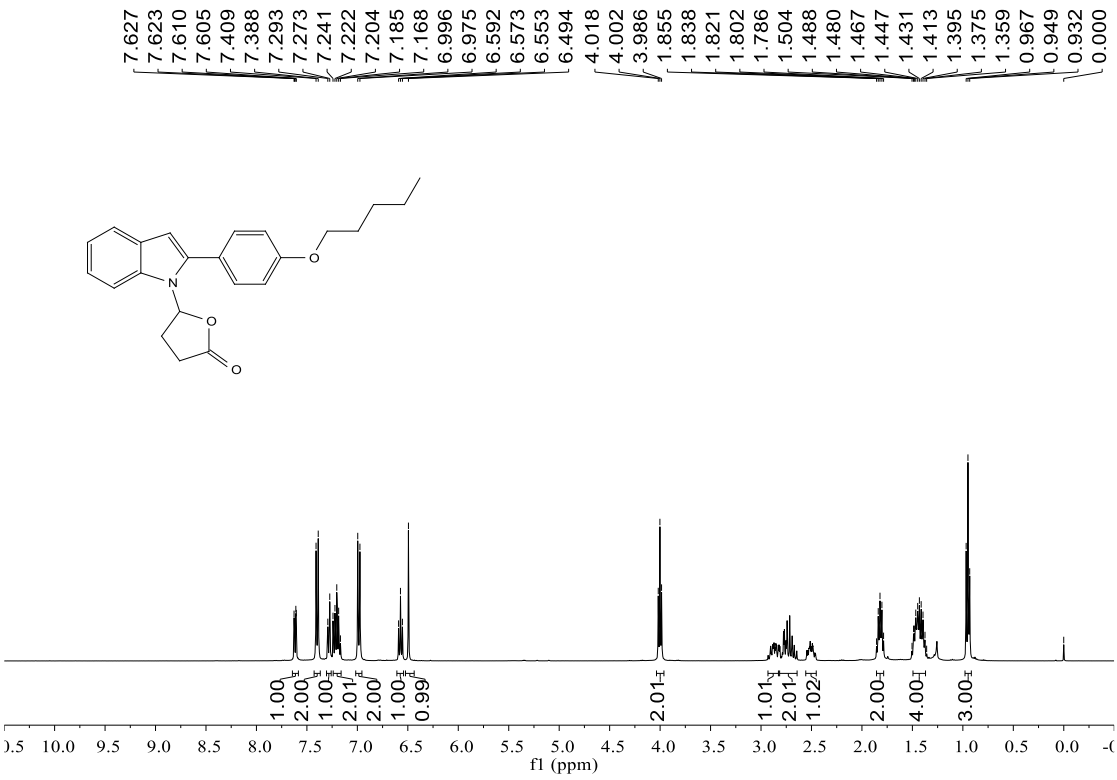
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3h



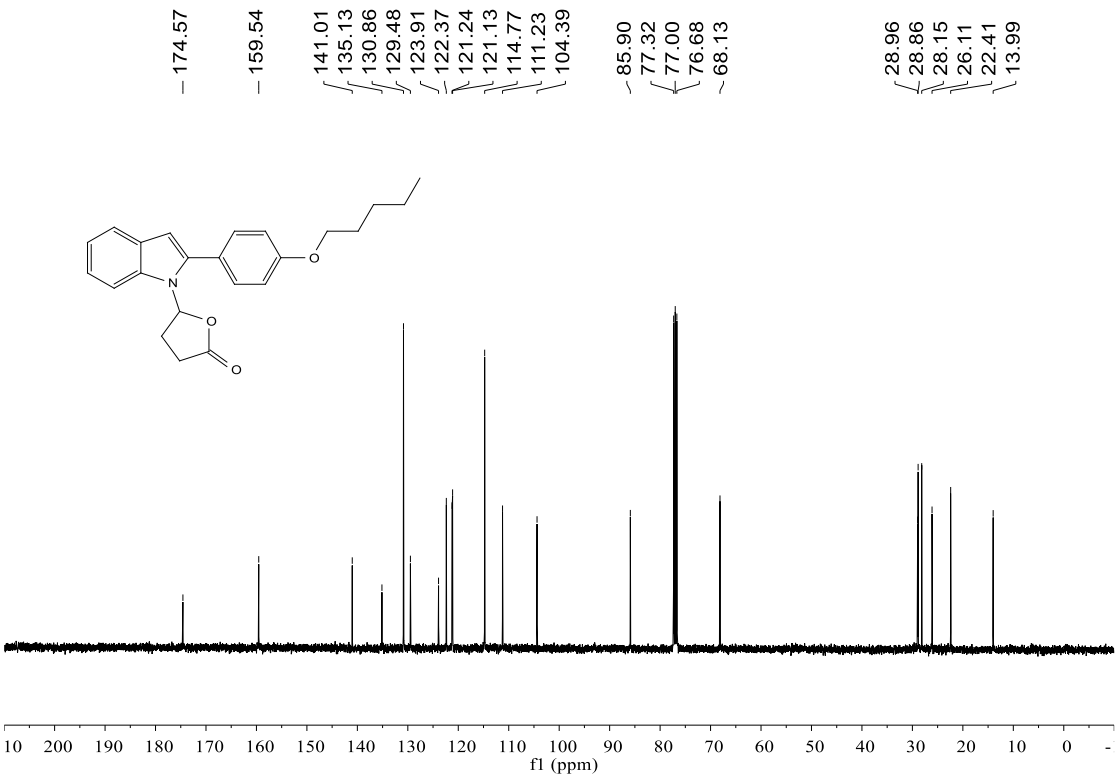
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3h



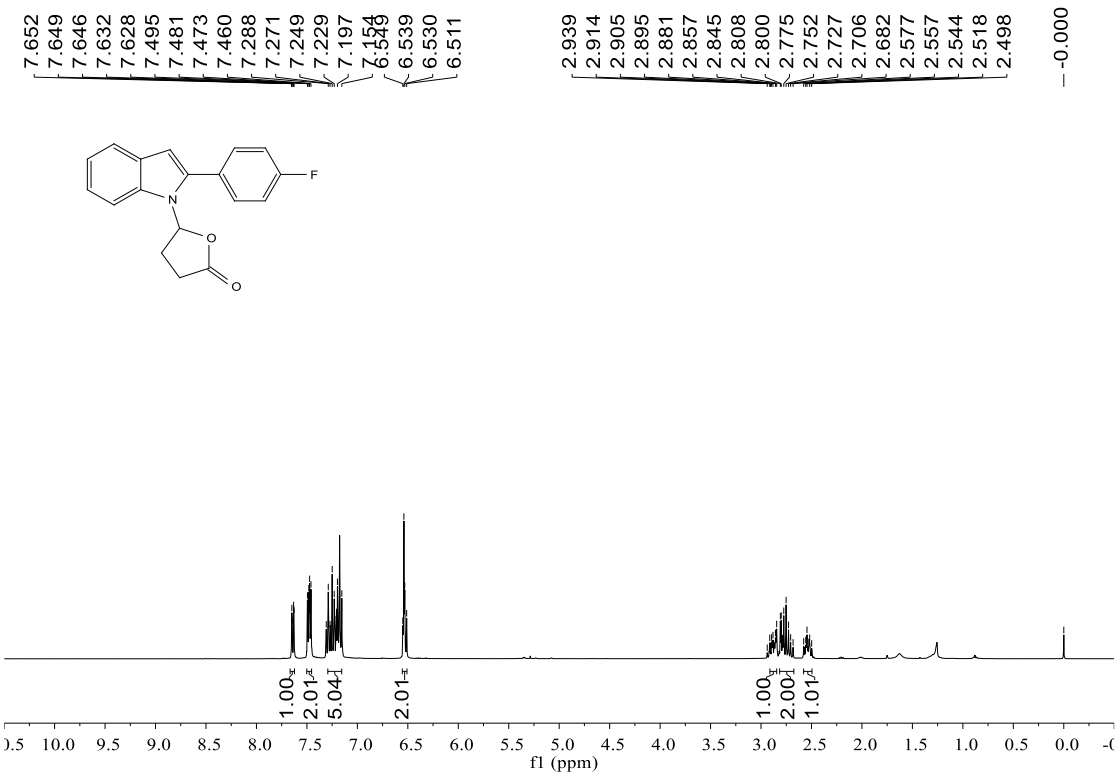
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3i



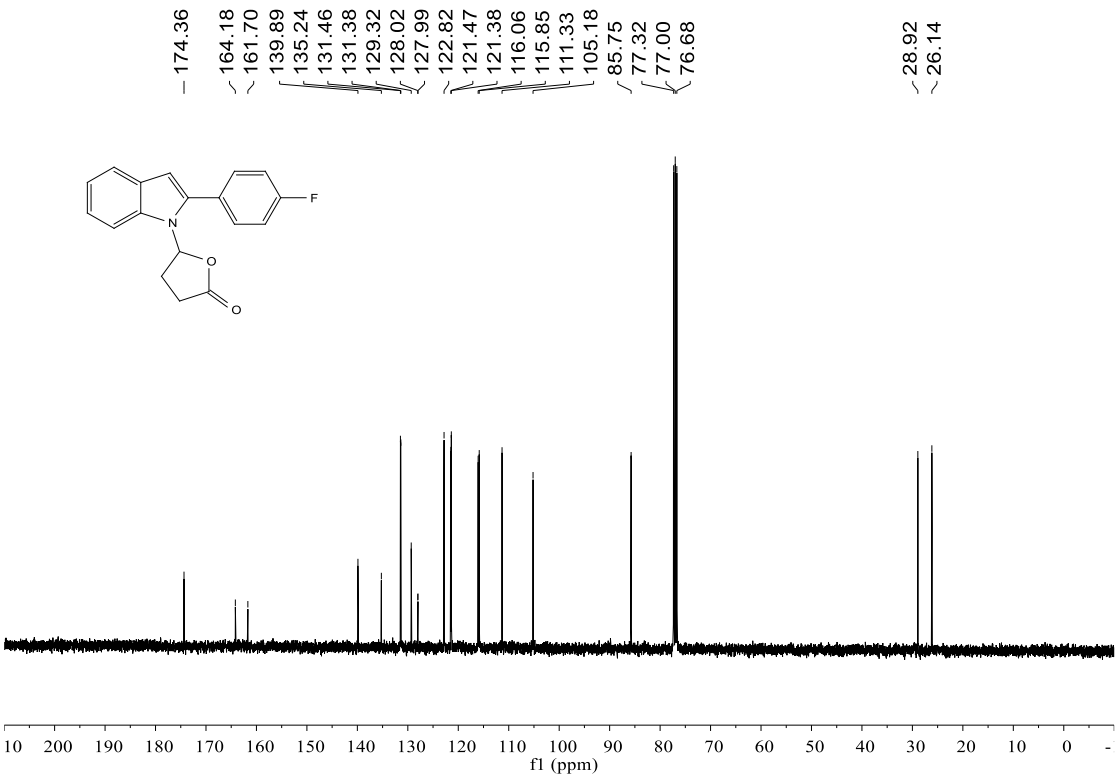
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3i



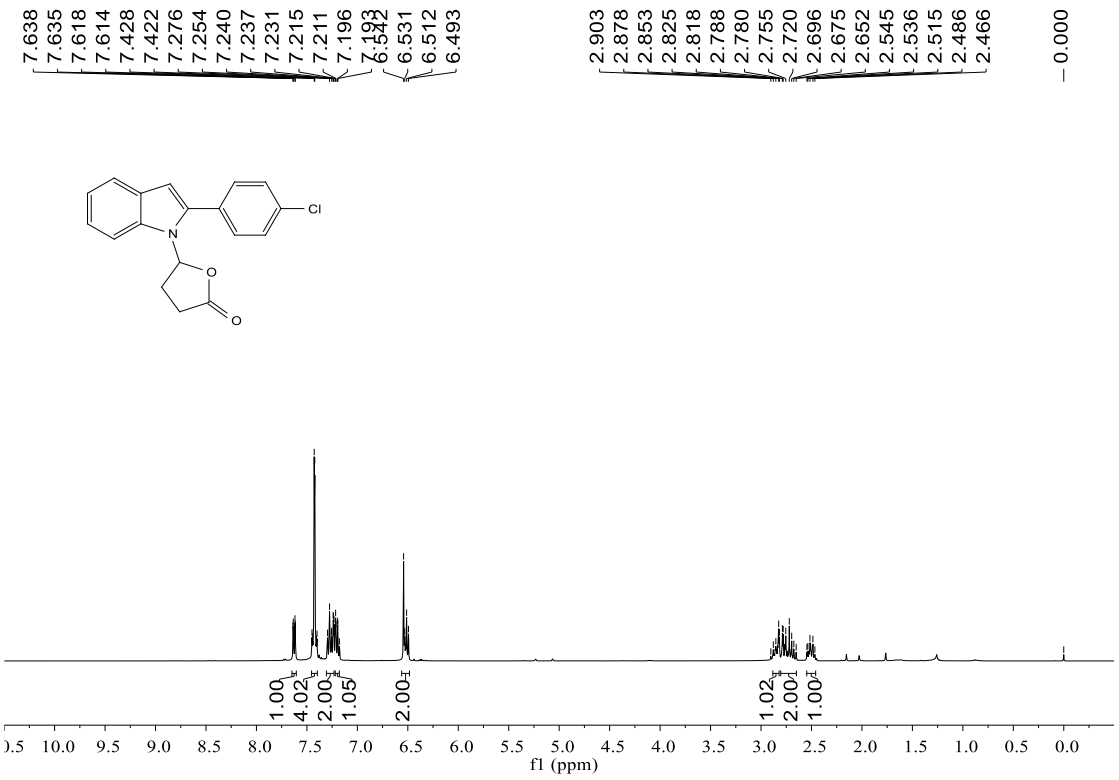
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3j



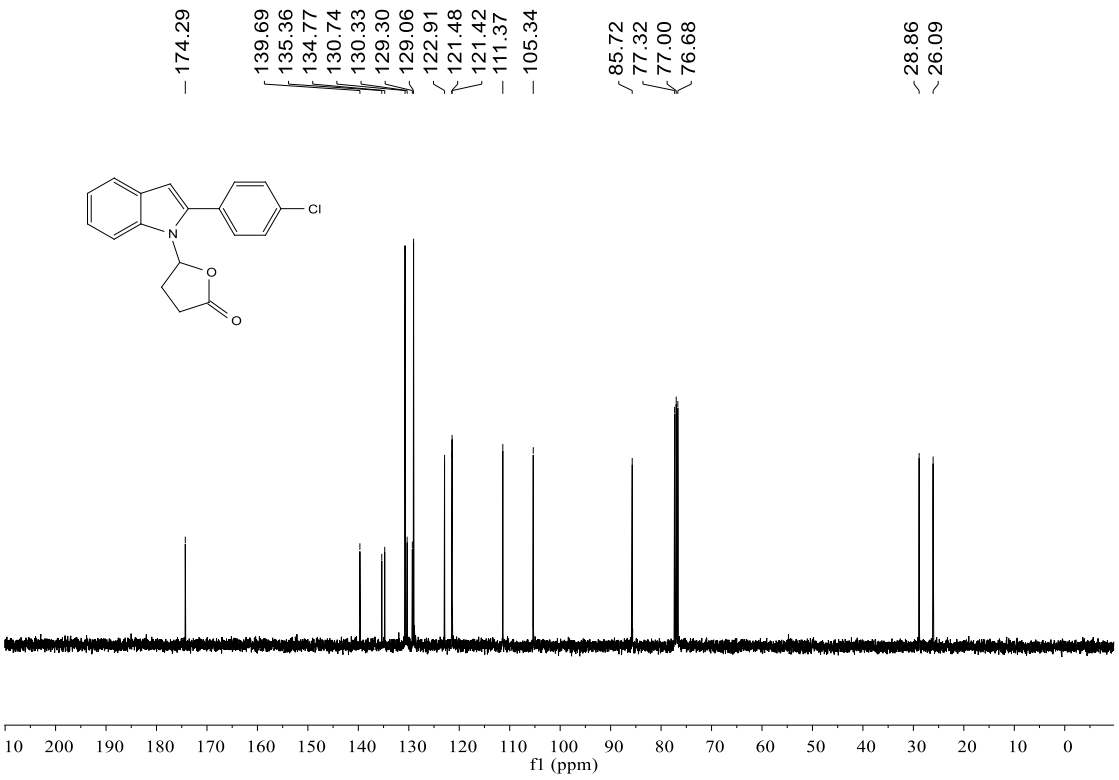
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3j



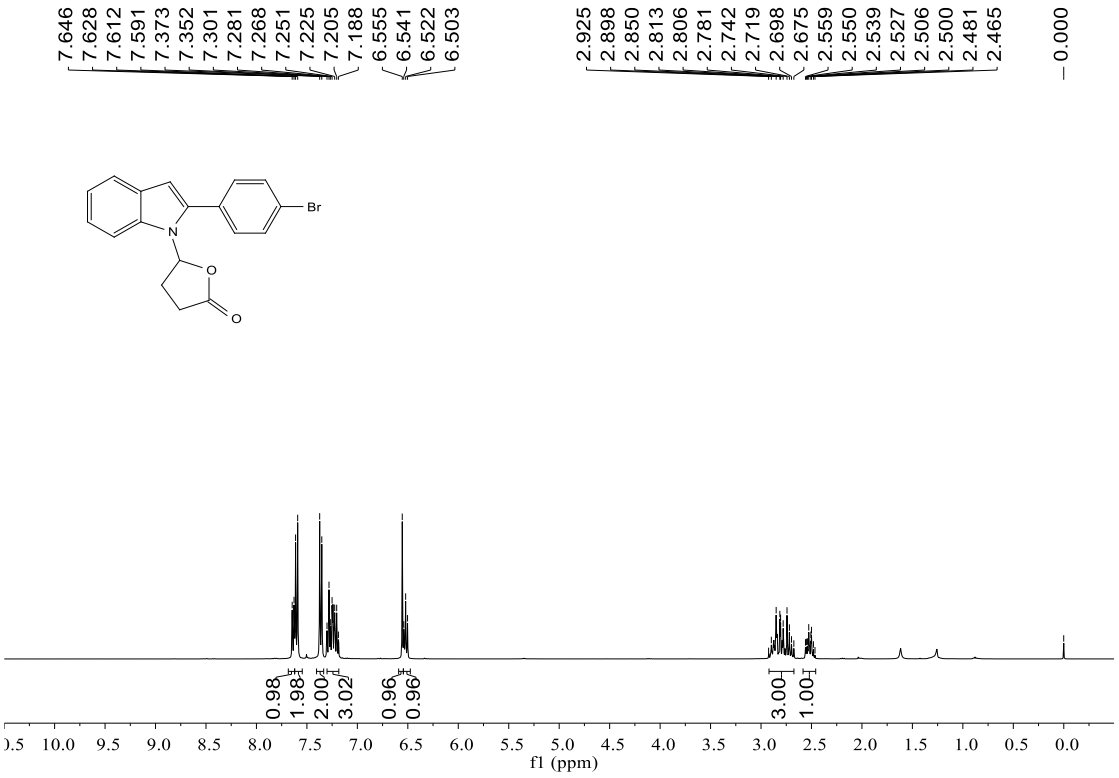
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3k



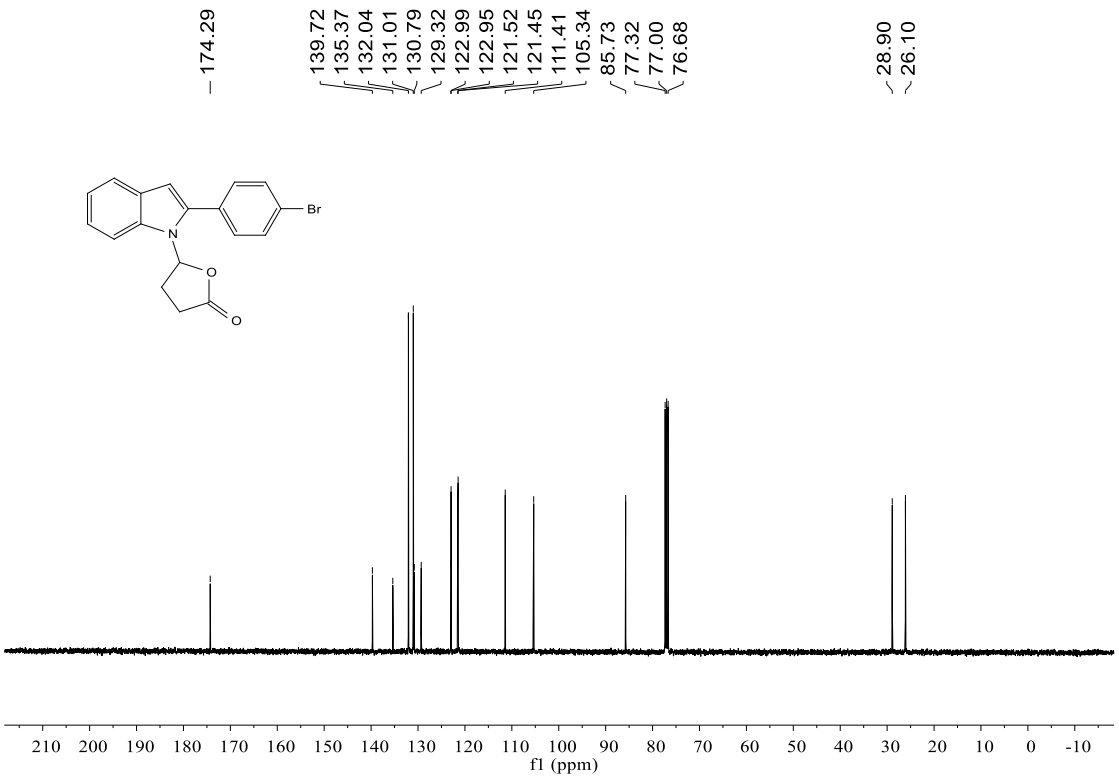
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3k



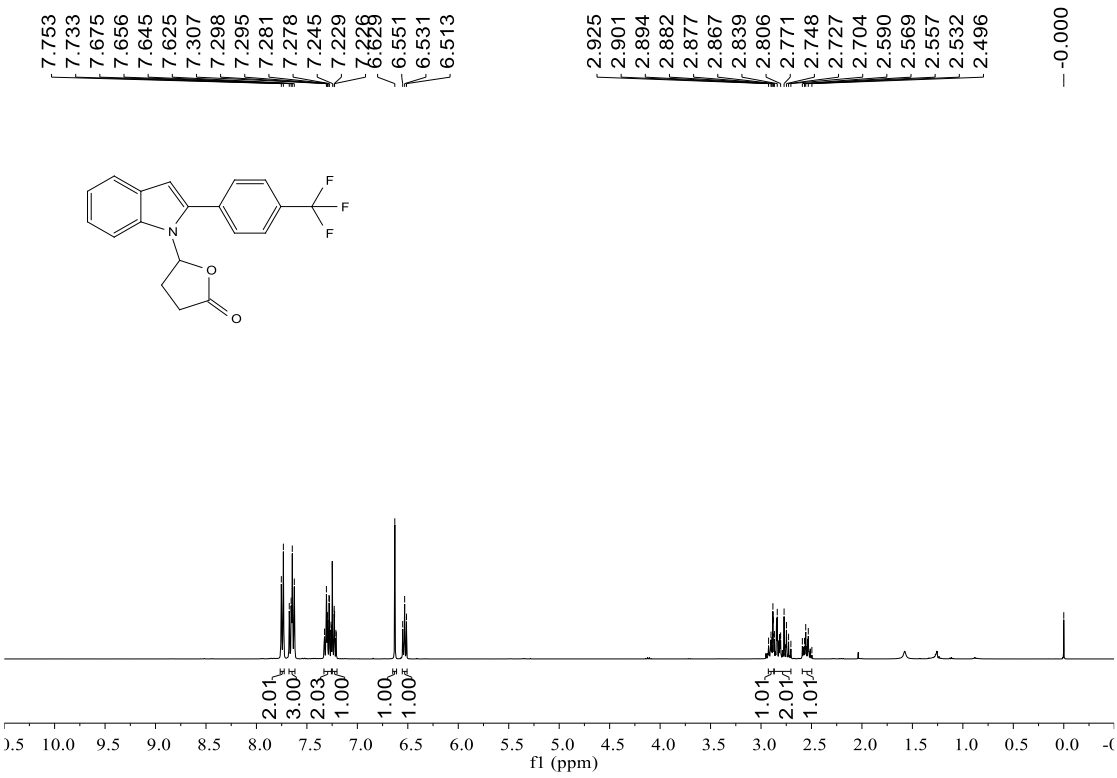
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3l



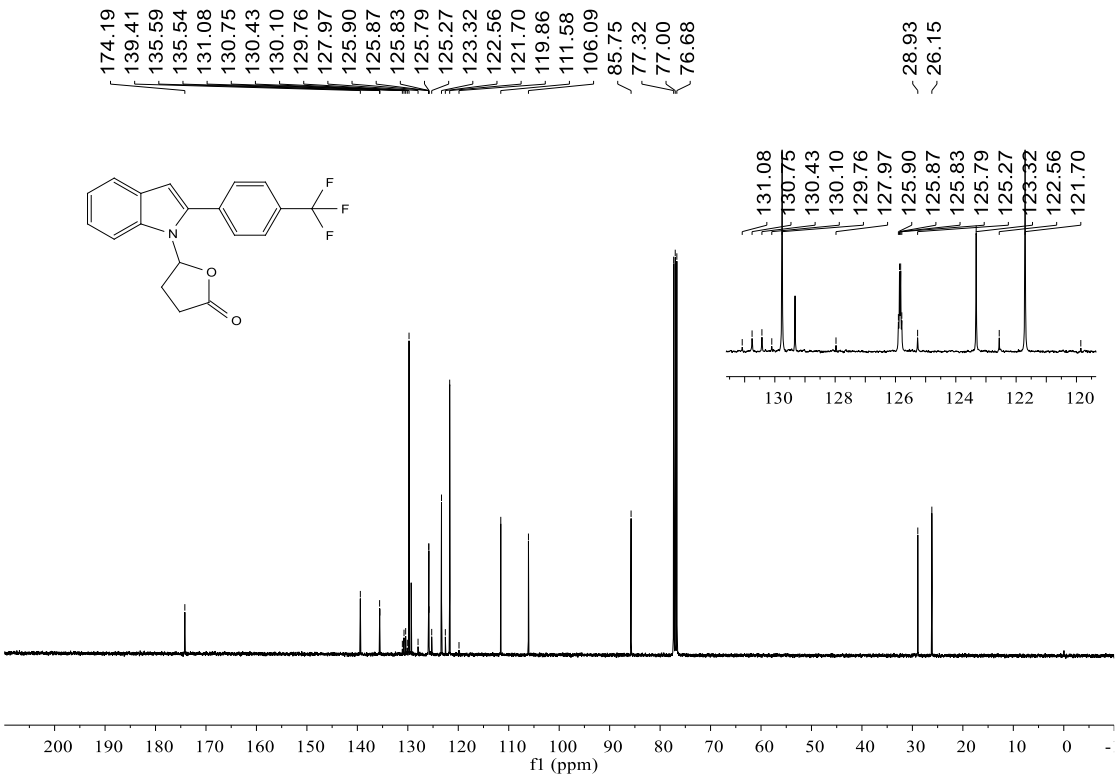
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3l



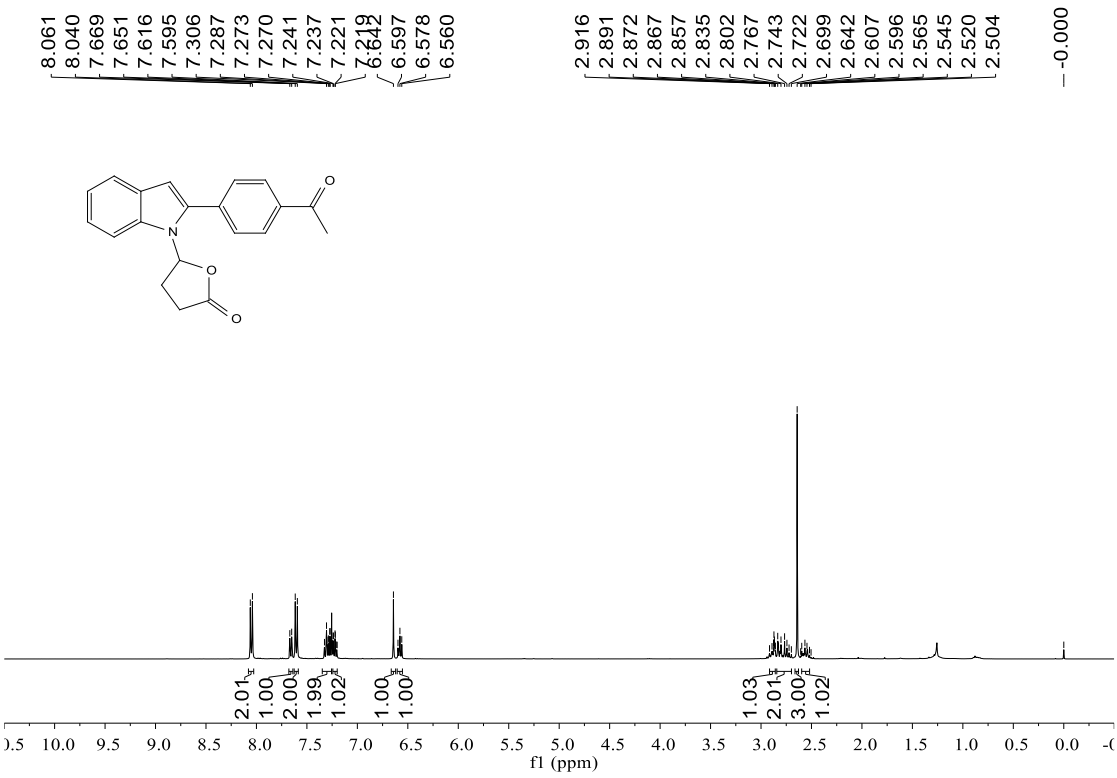
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3m



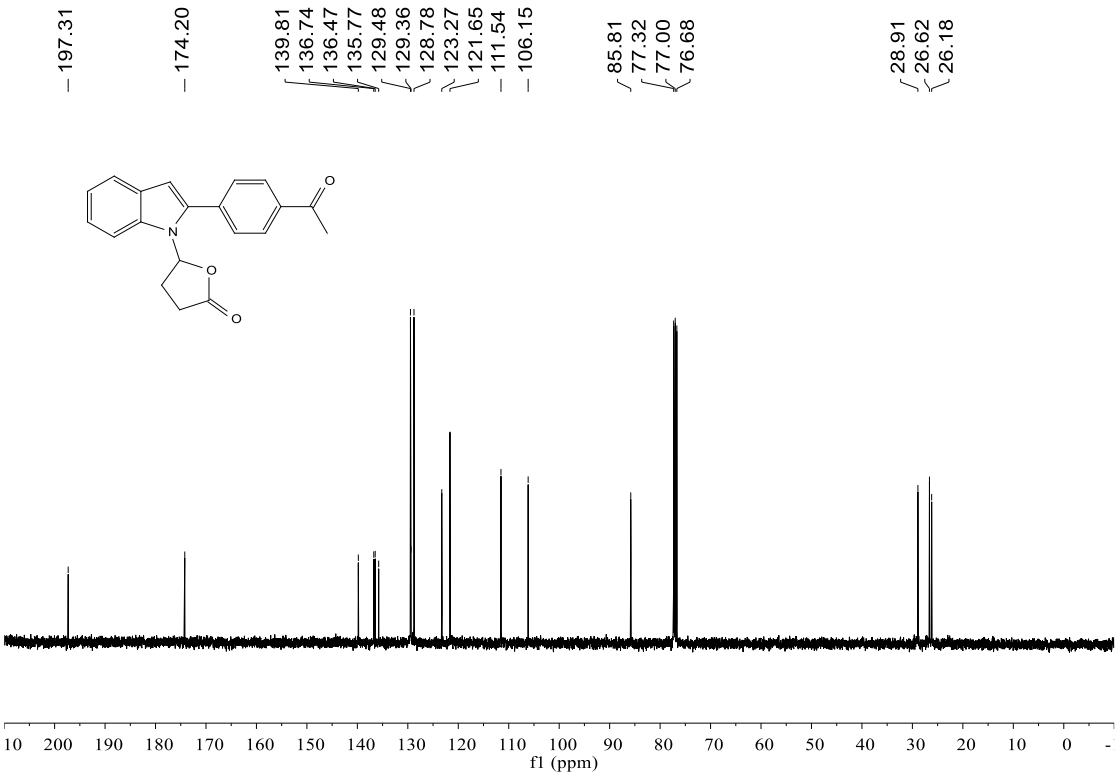
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3m



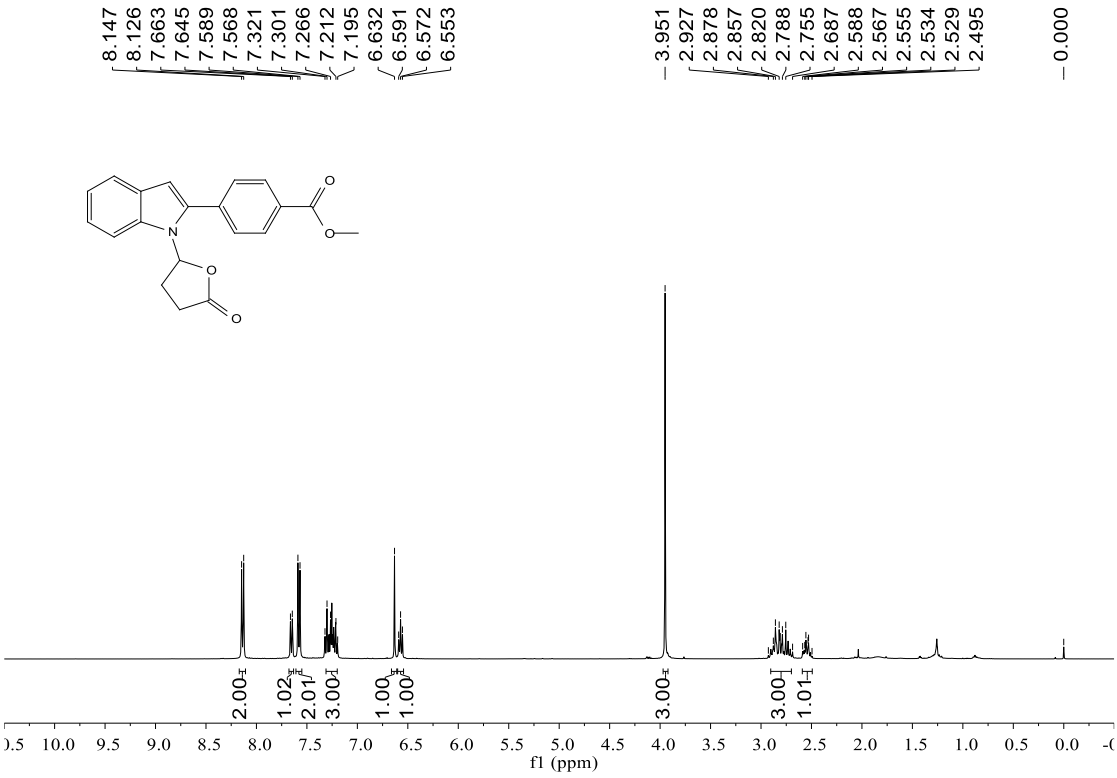
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3n



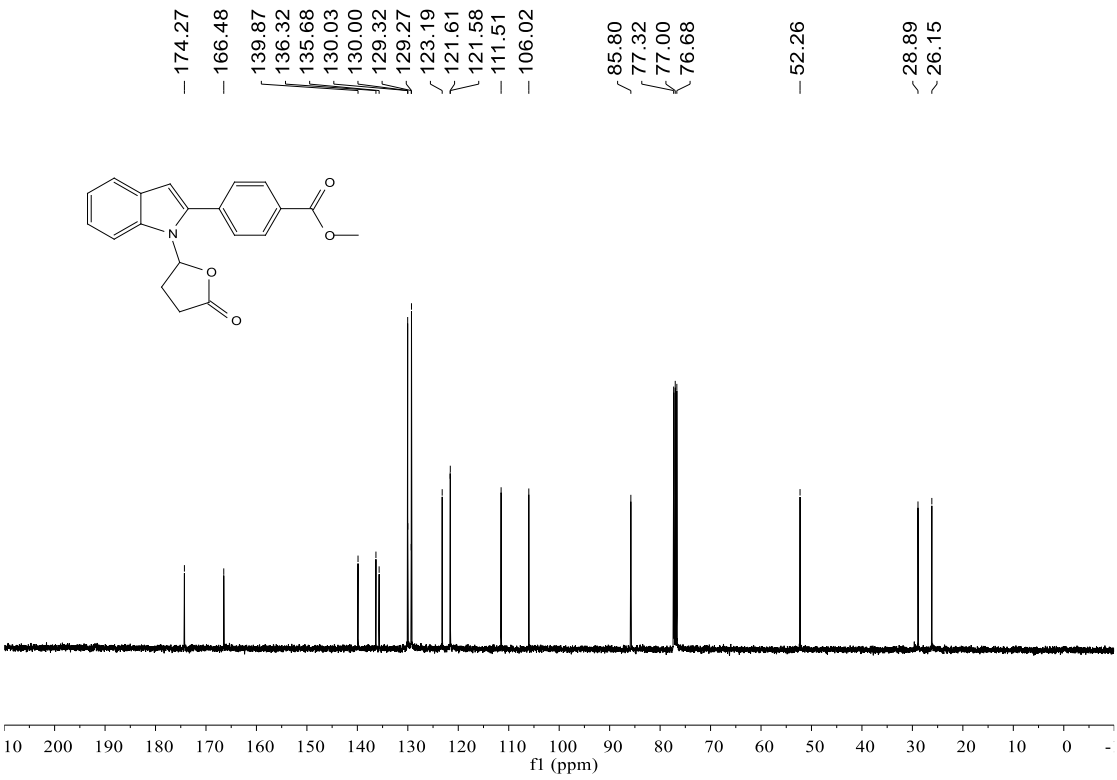
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3n



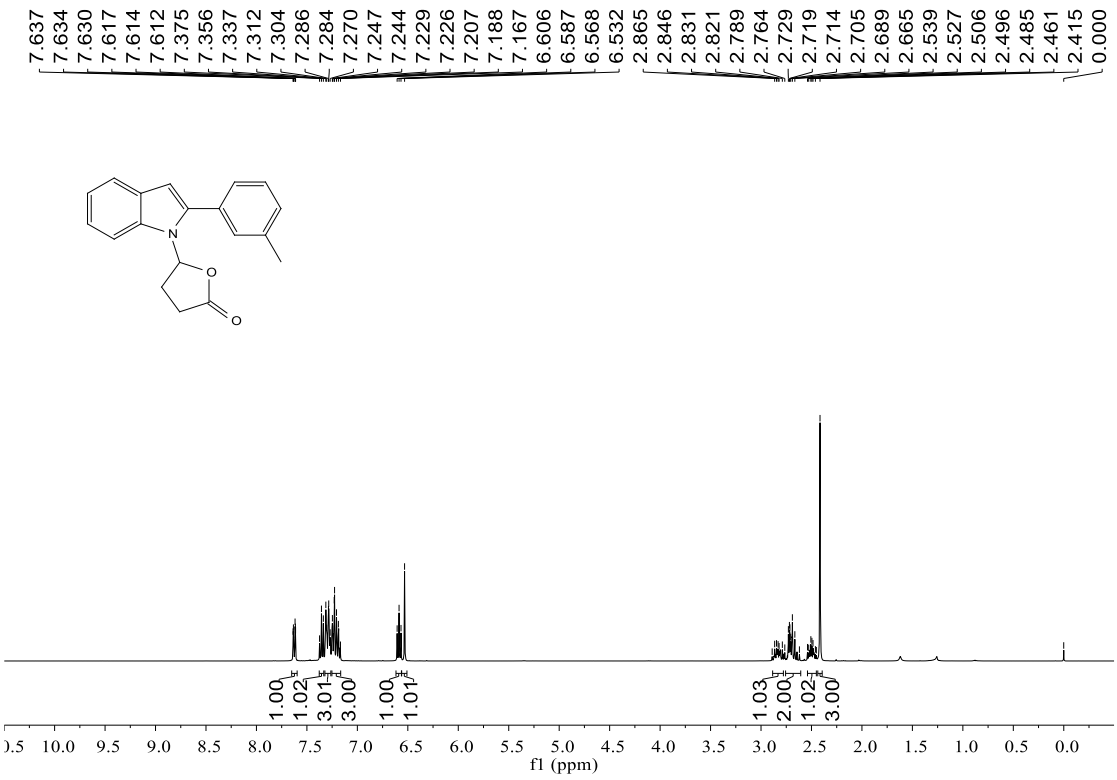
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3o



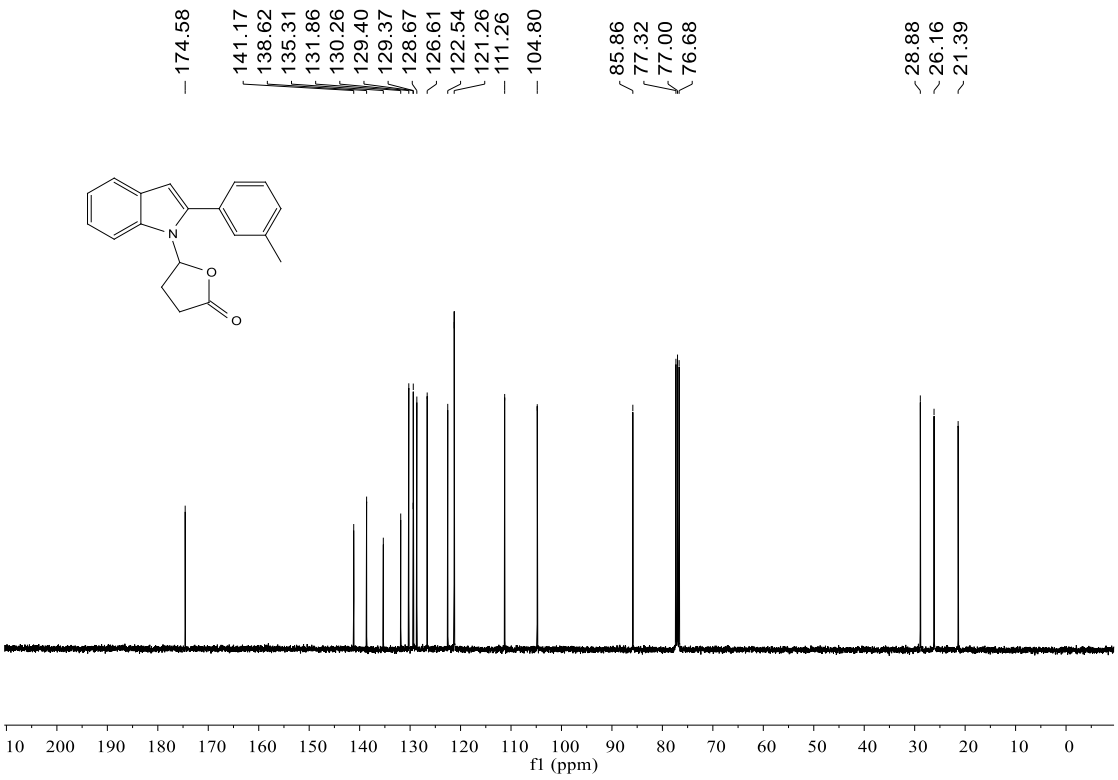
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3o



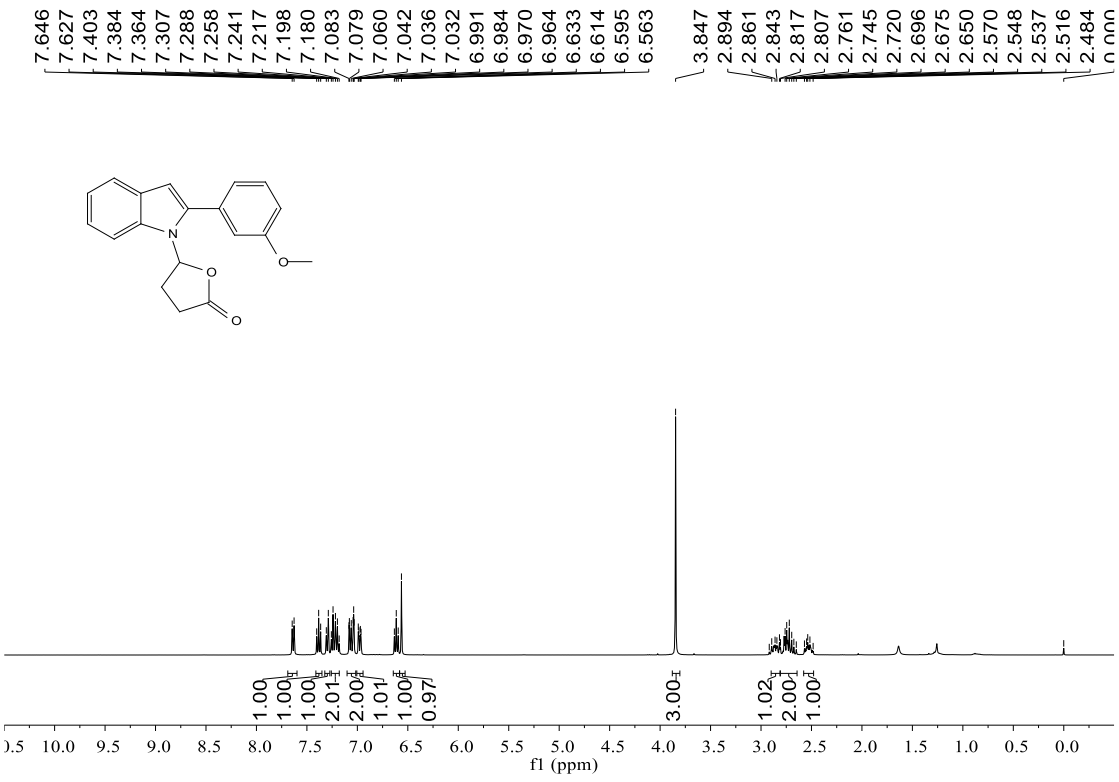
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3p



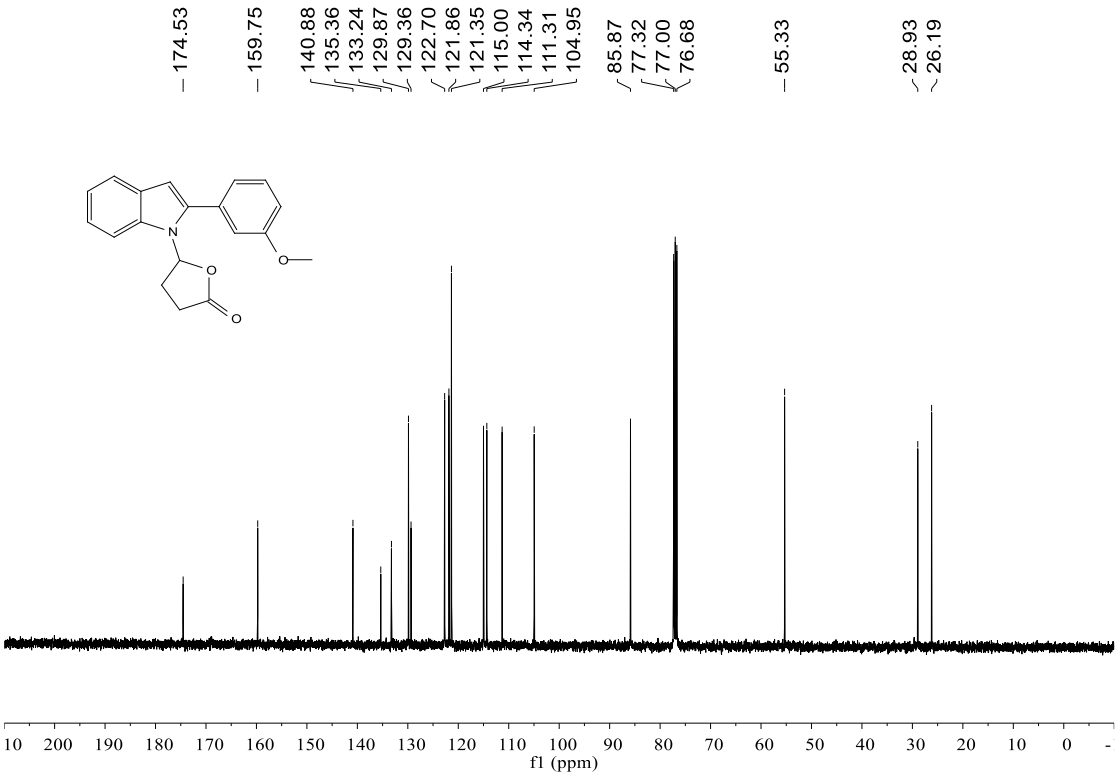
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3p



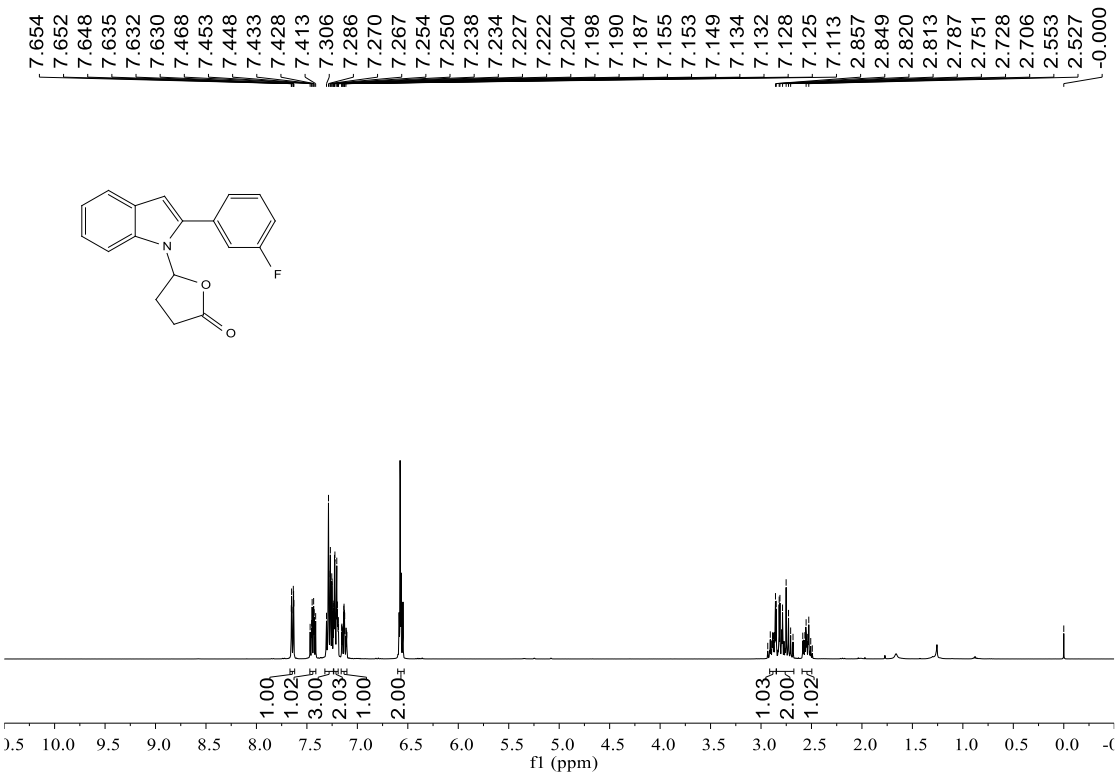
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3q



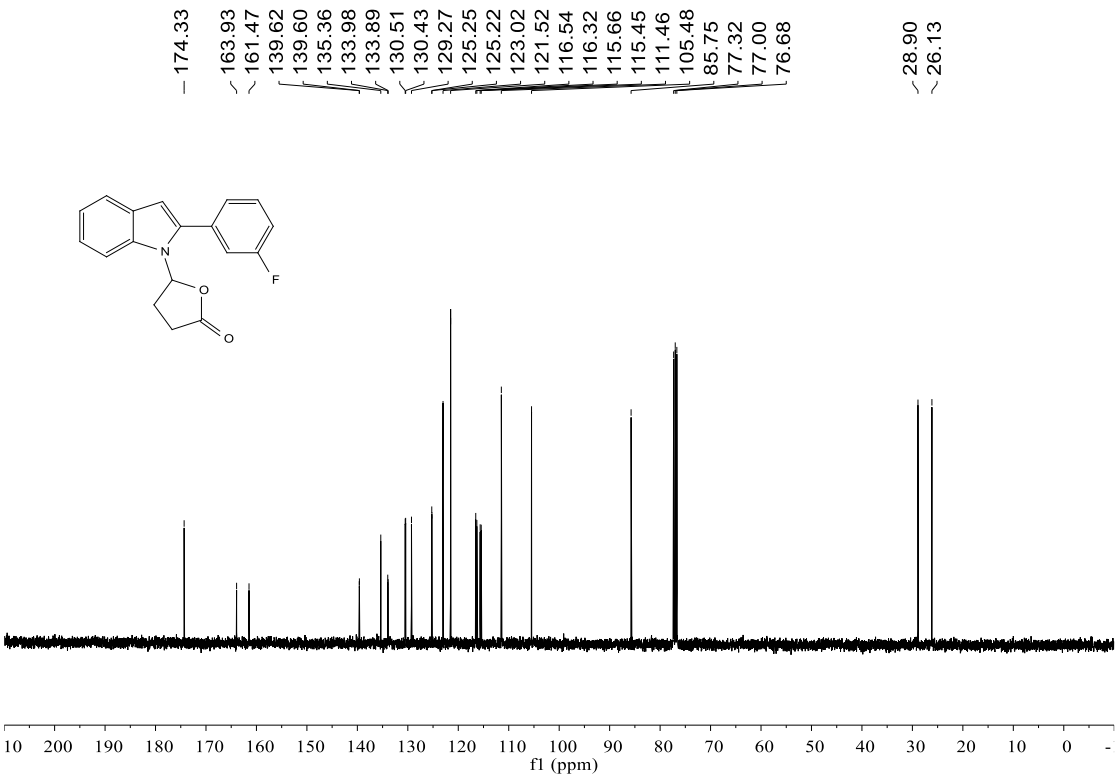
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3q



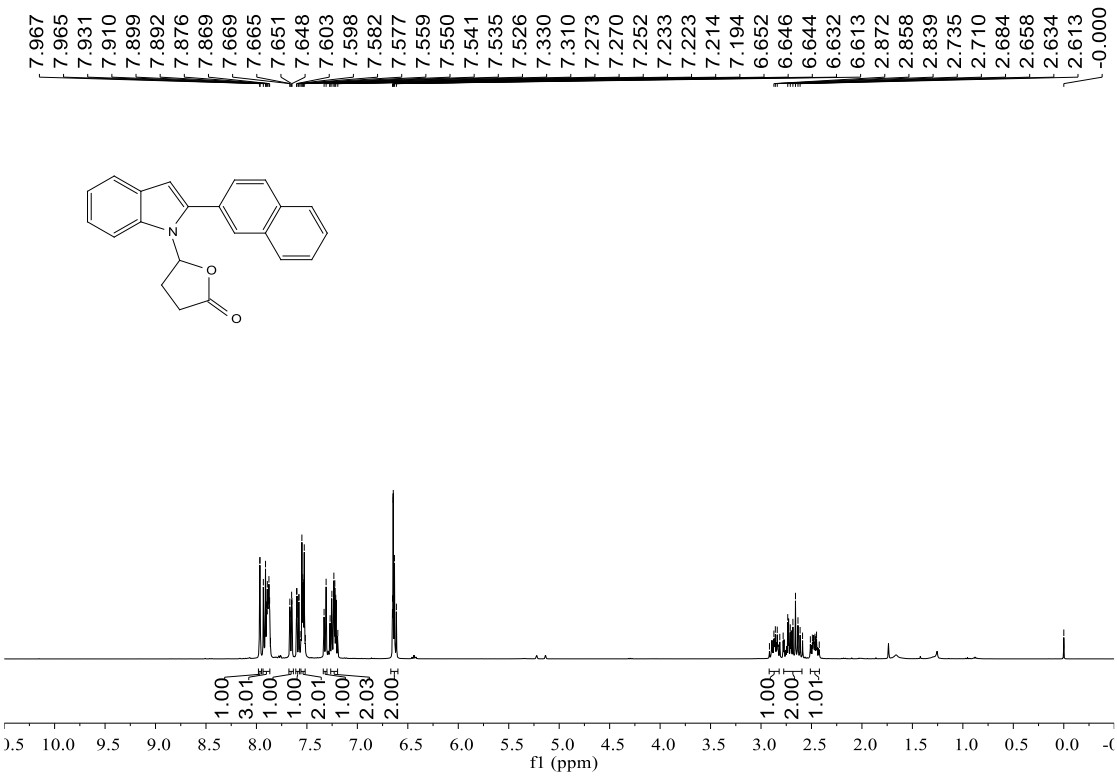
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3r



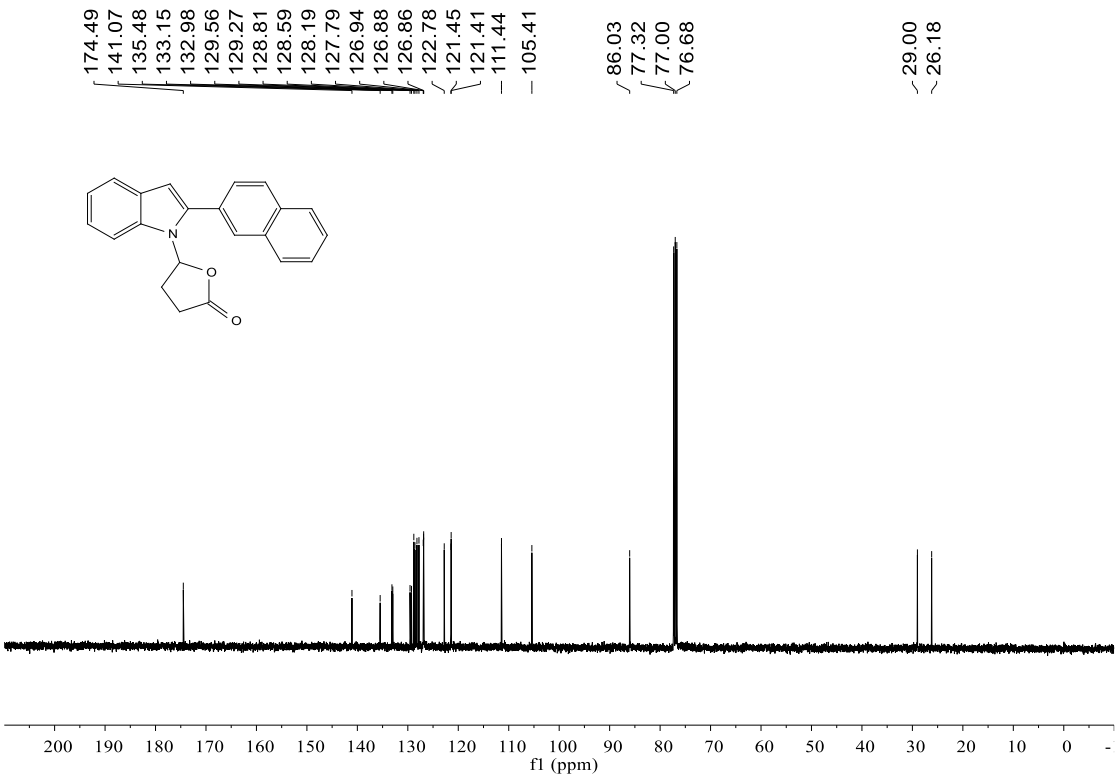
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3r



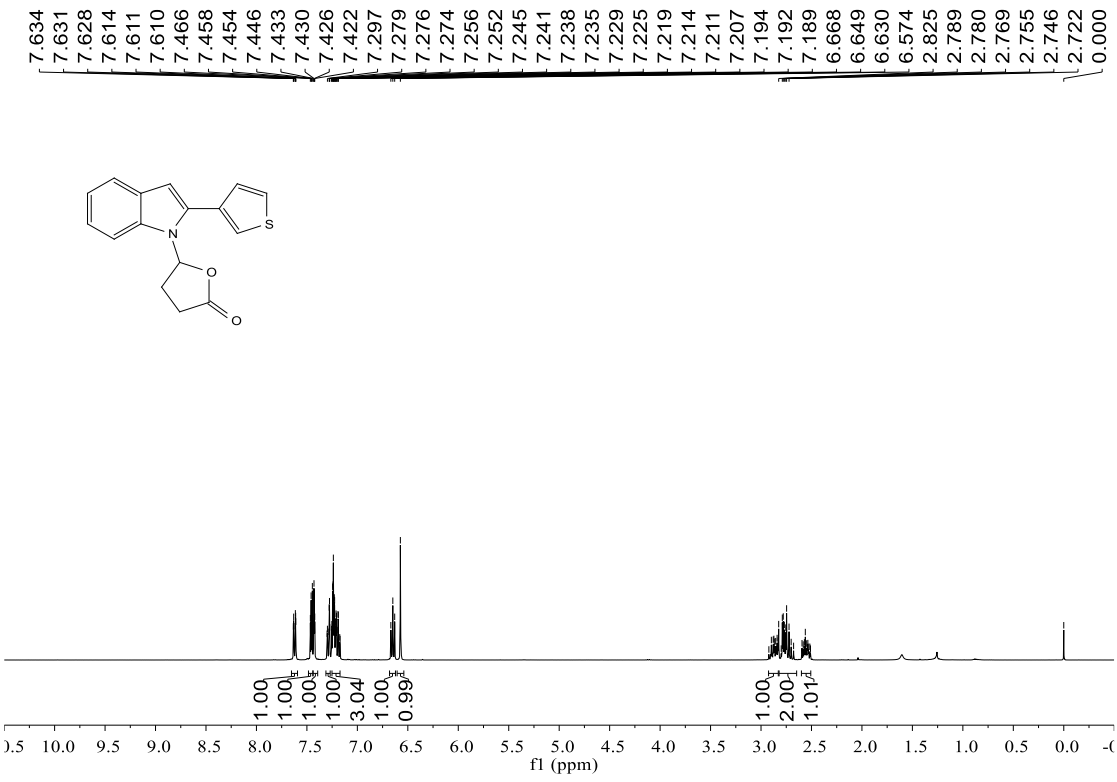
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3s



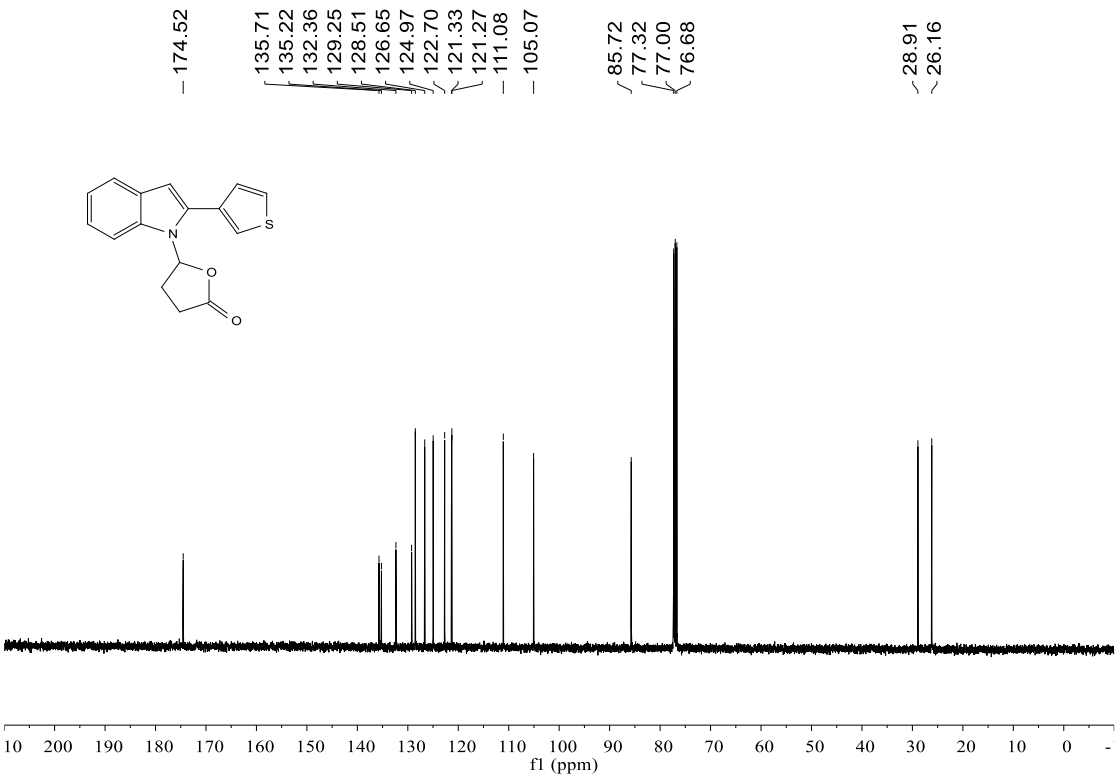
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3s



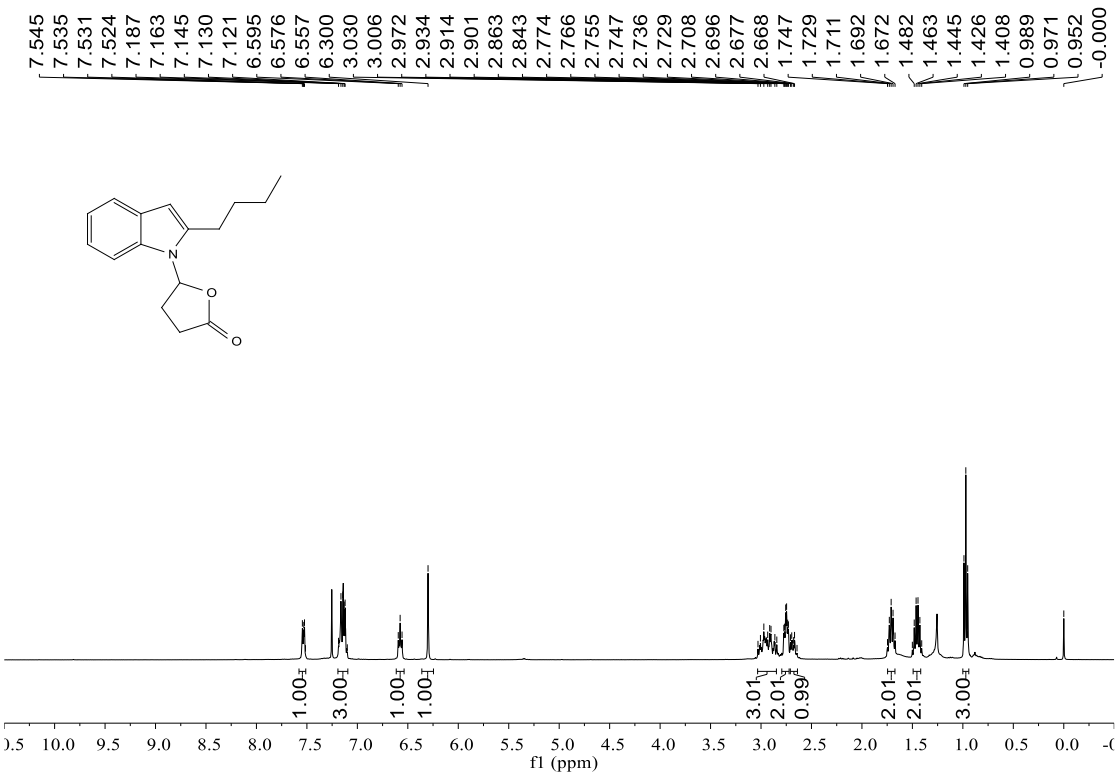
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3t



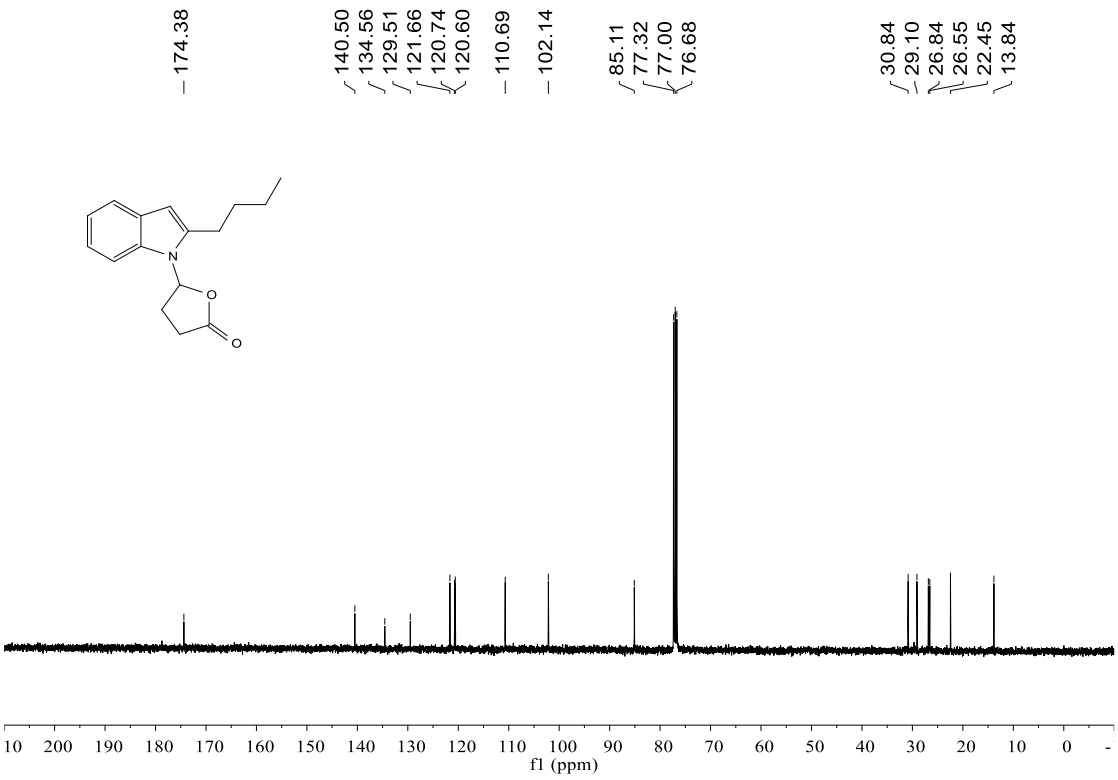
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3t



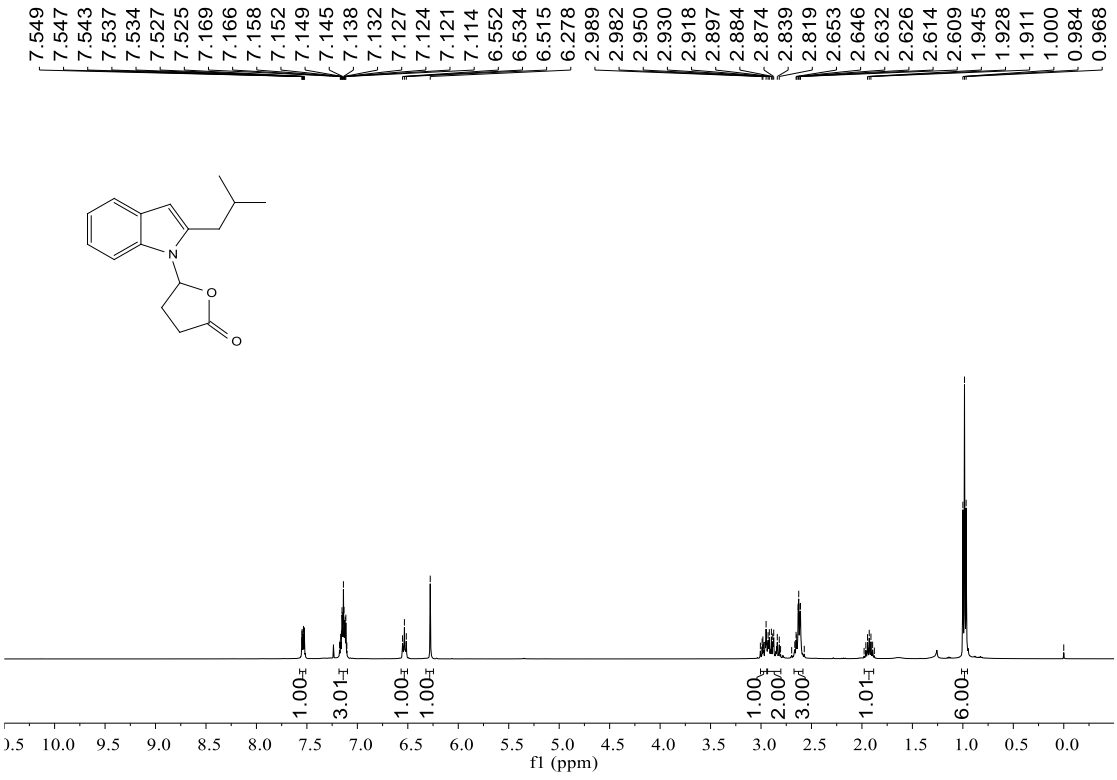
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3u



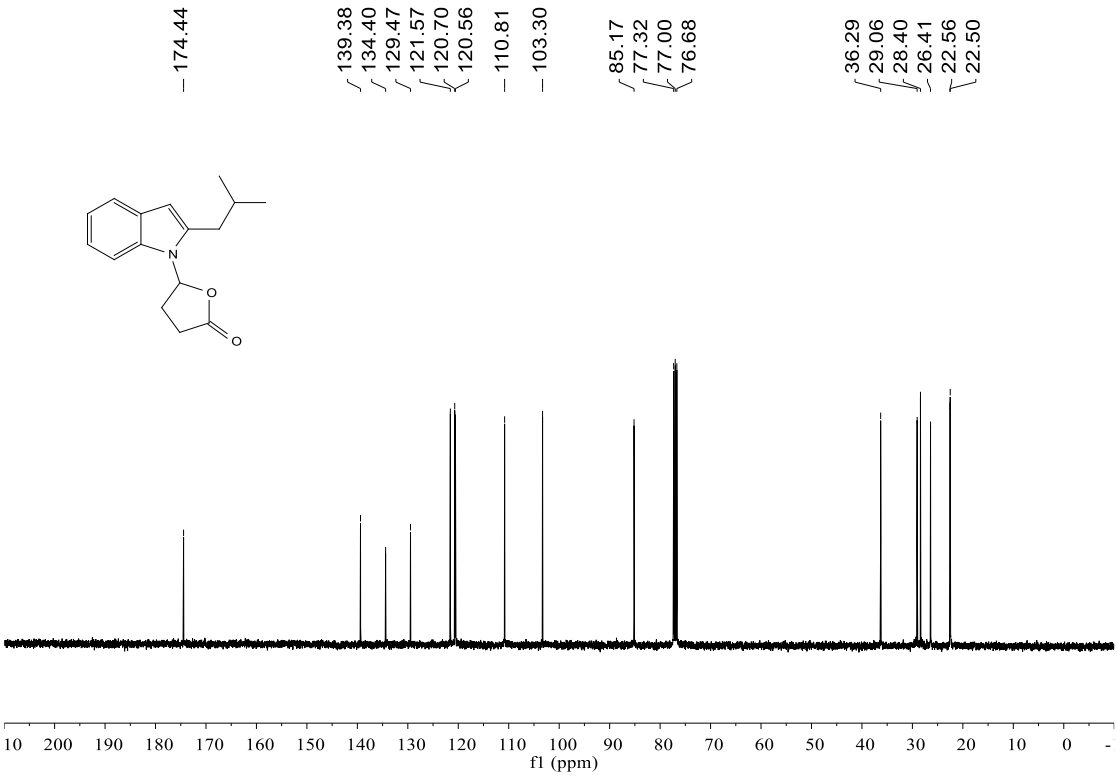
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3u



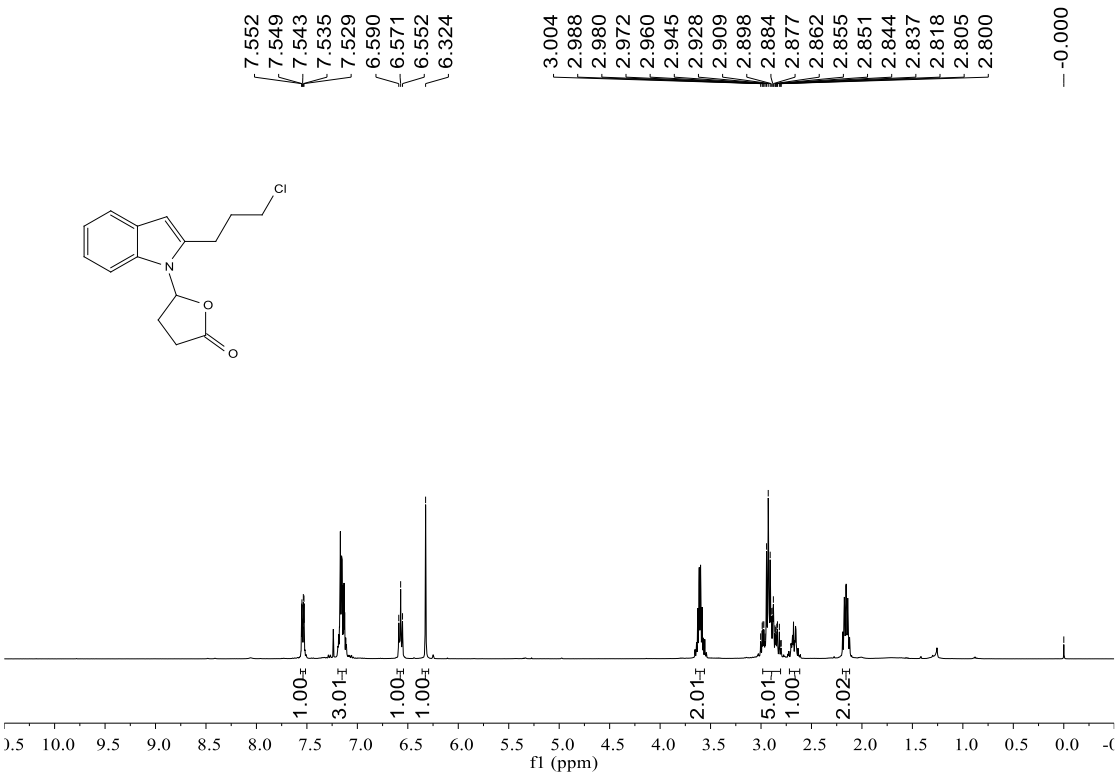
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3v



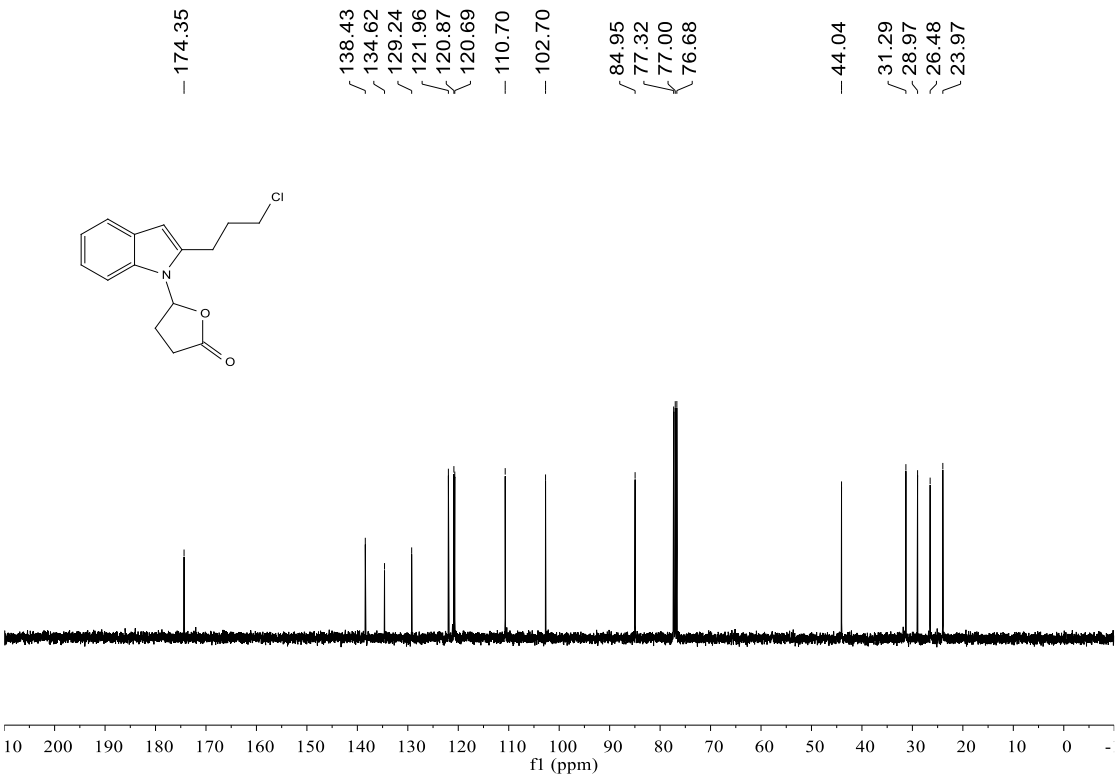
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3v



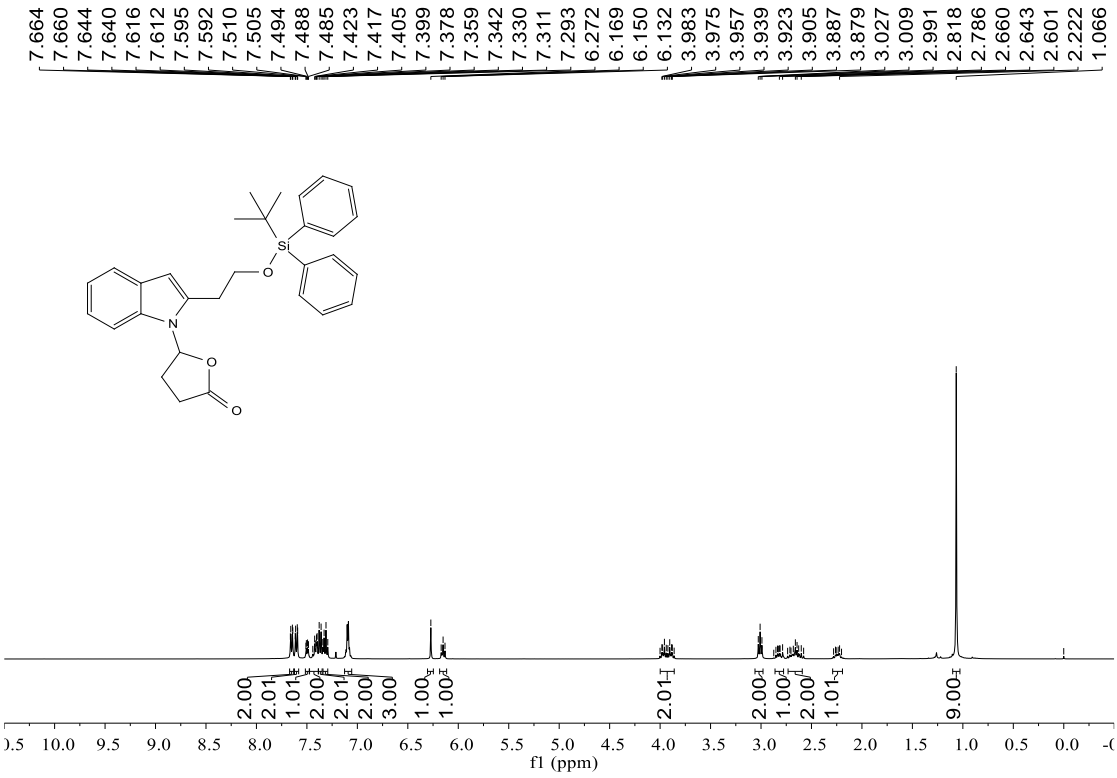
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3w



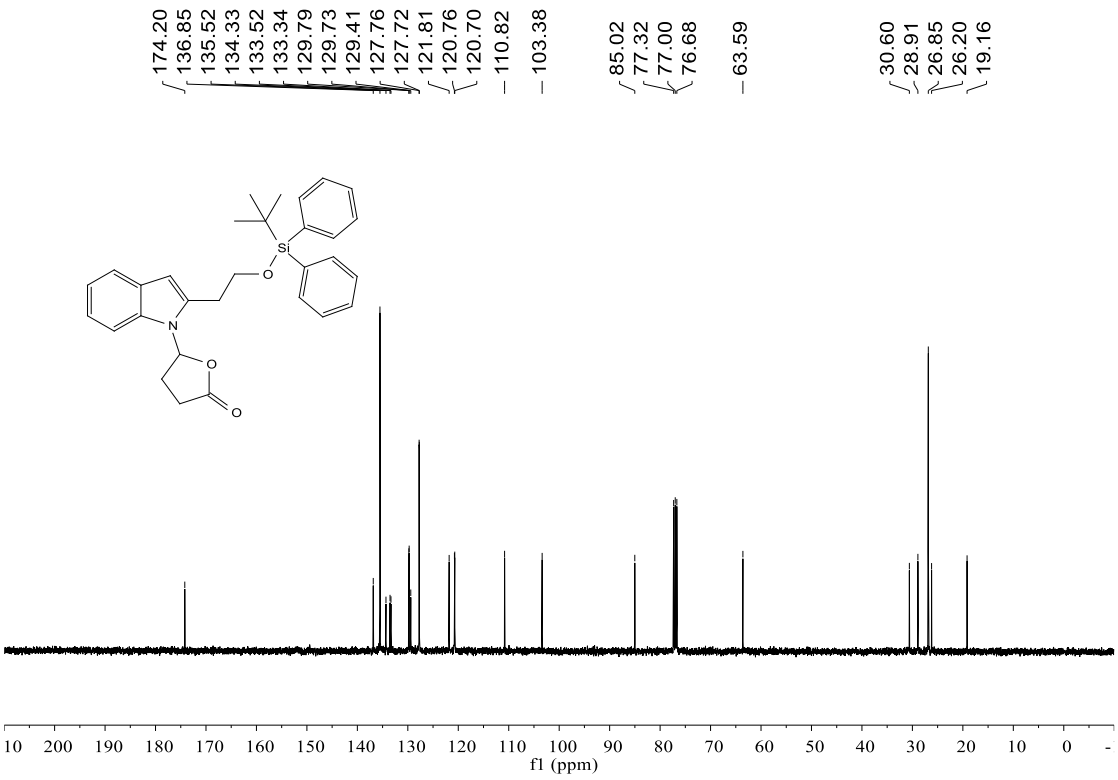
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3w



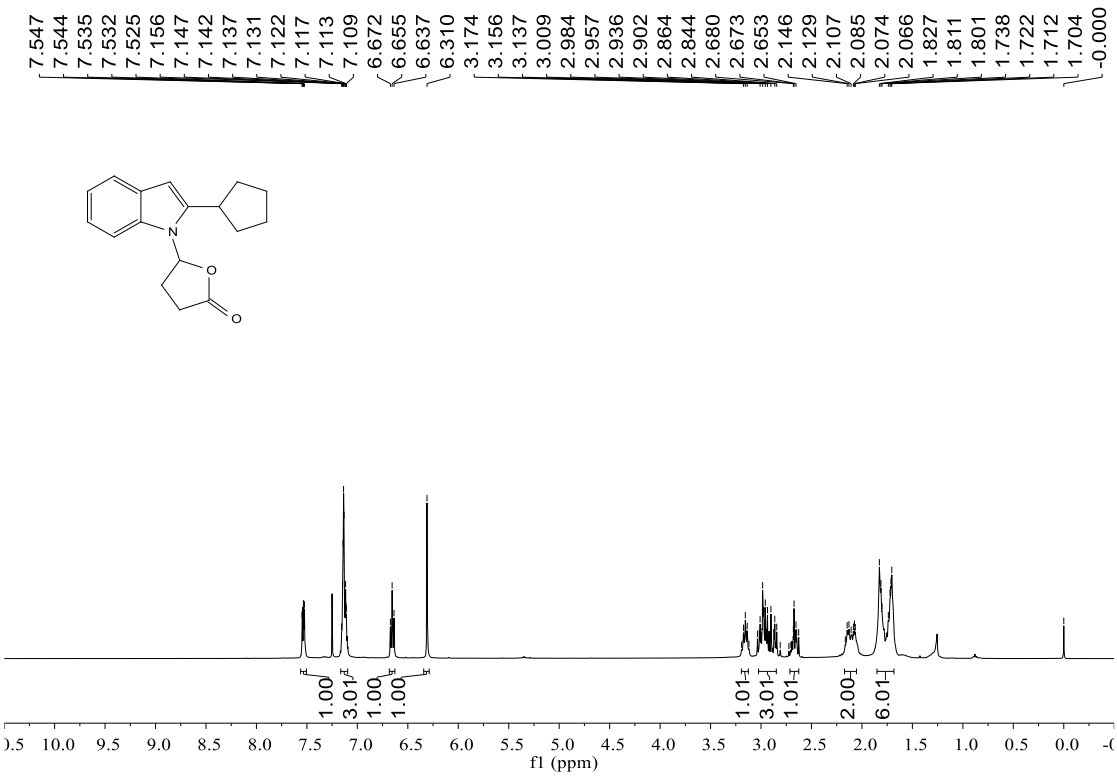
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3x



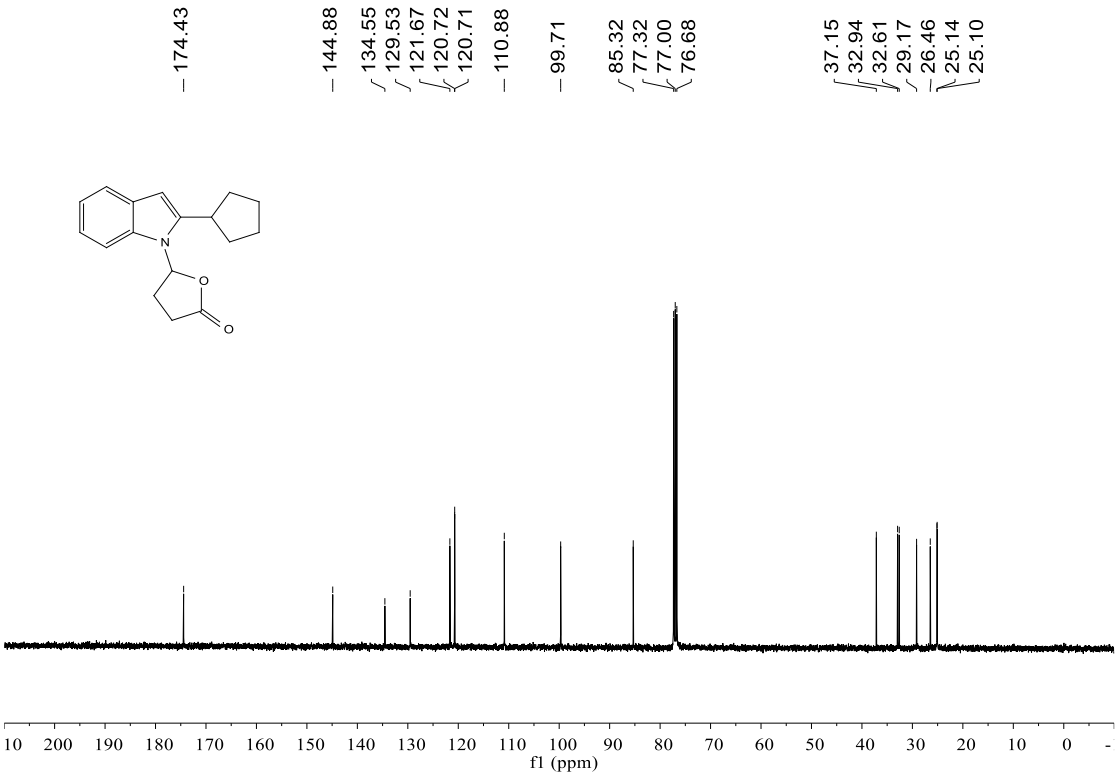
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3x



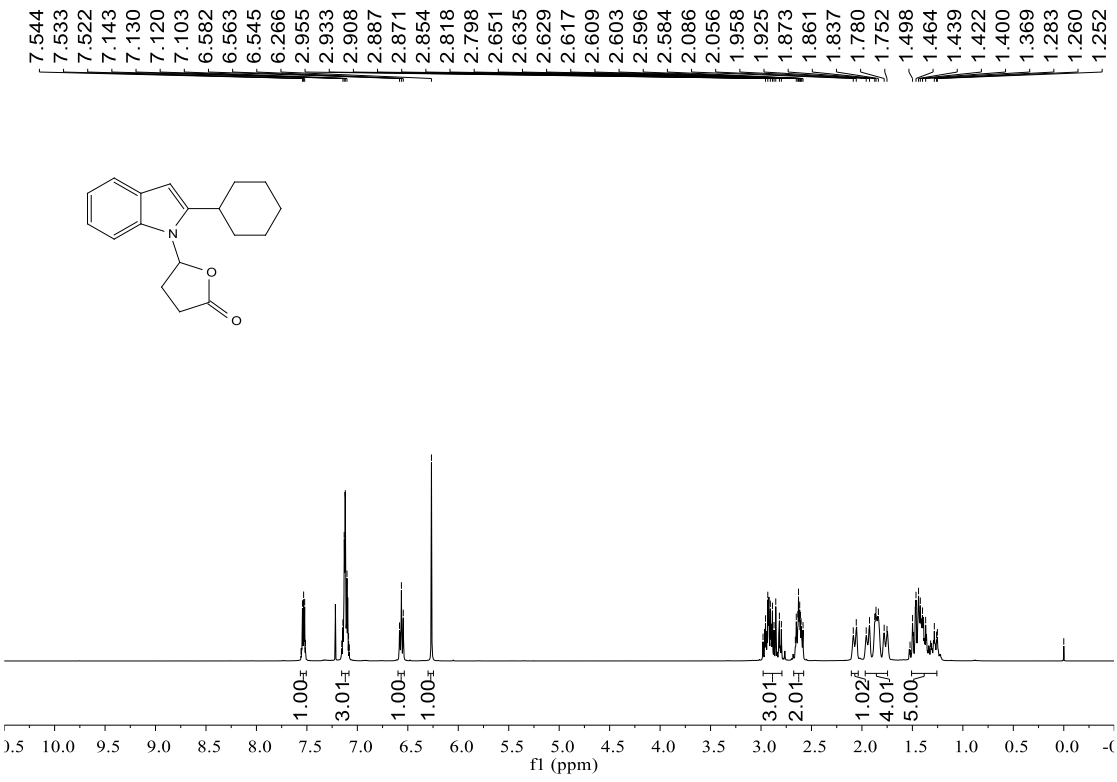
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3y



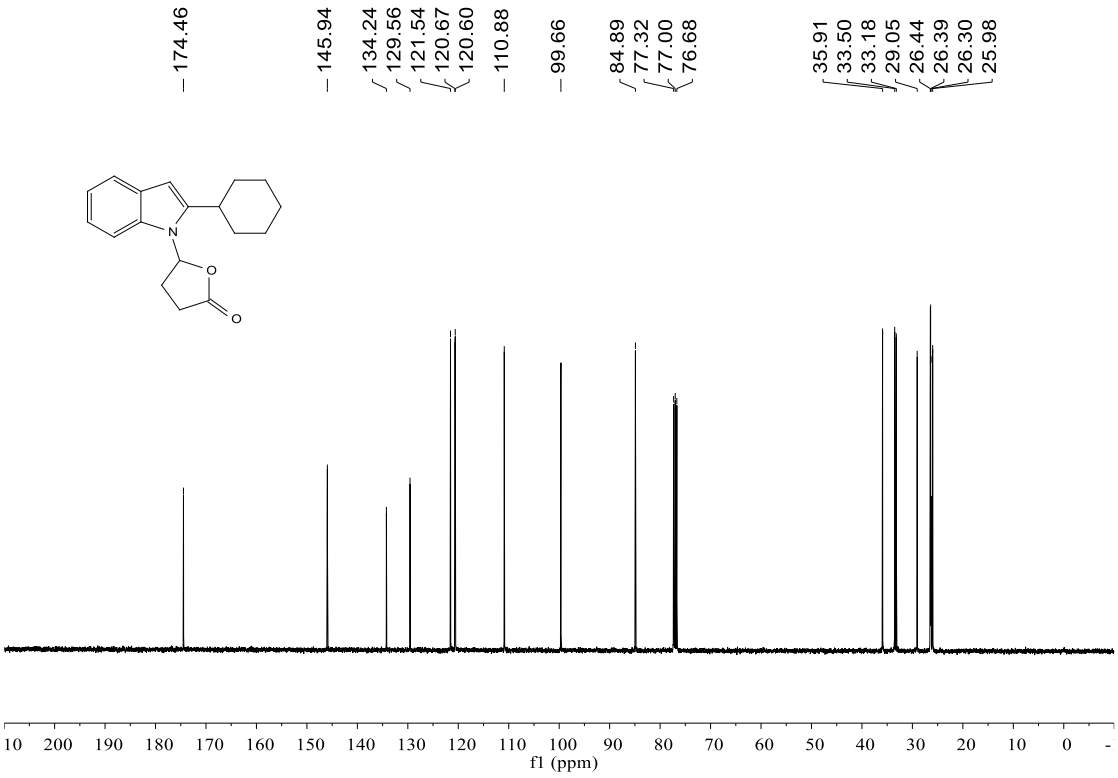
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3y



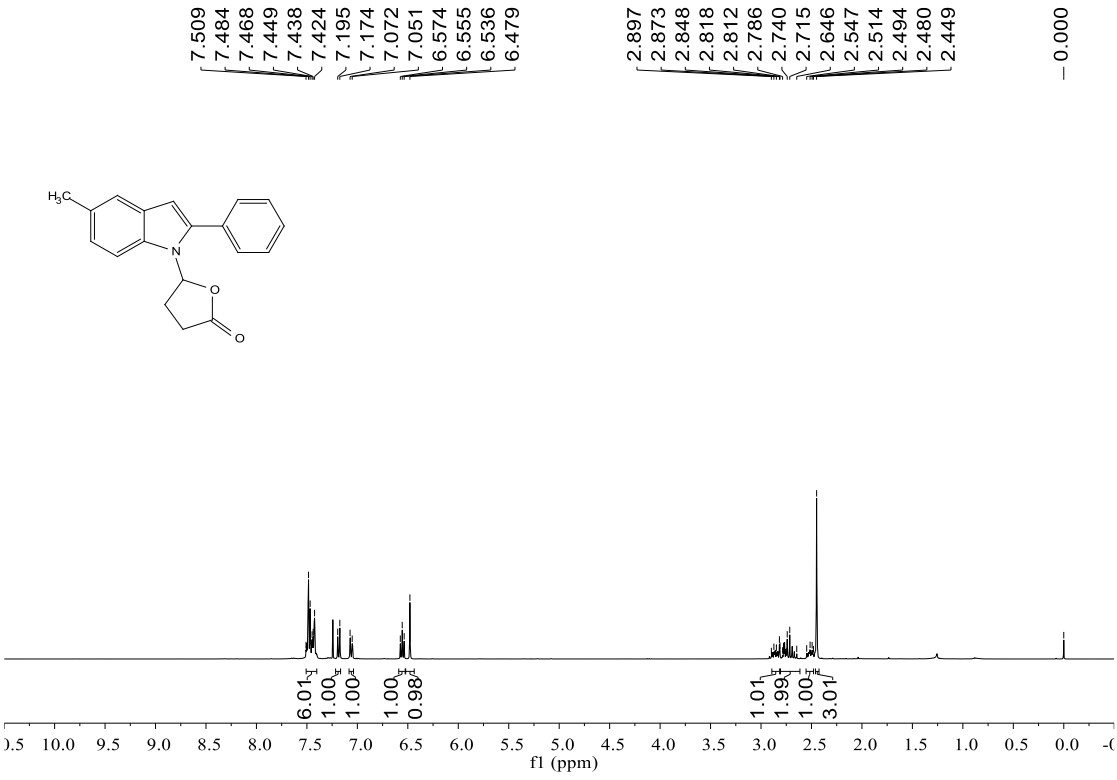
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3z



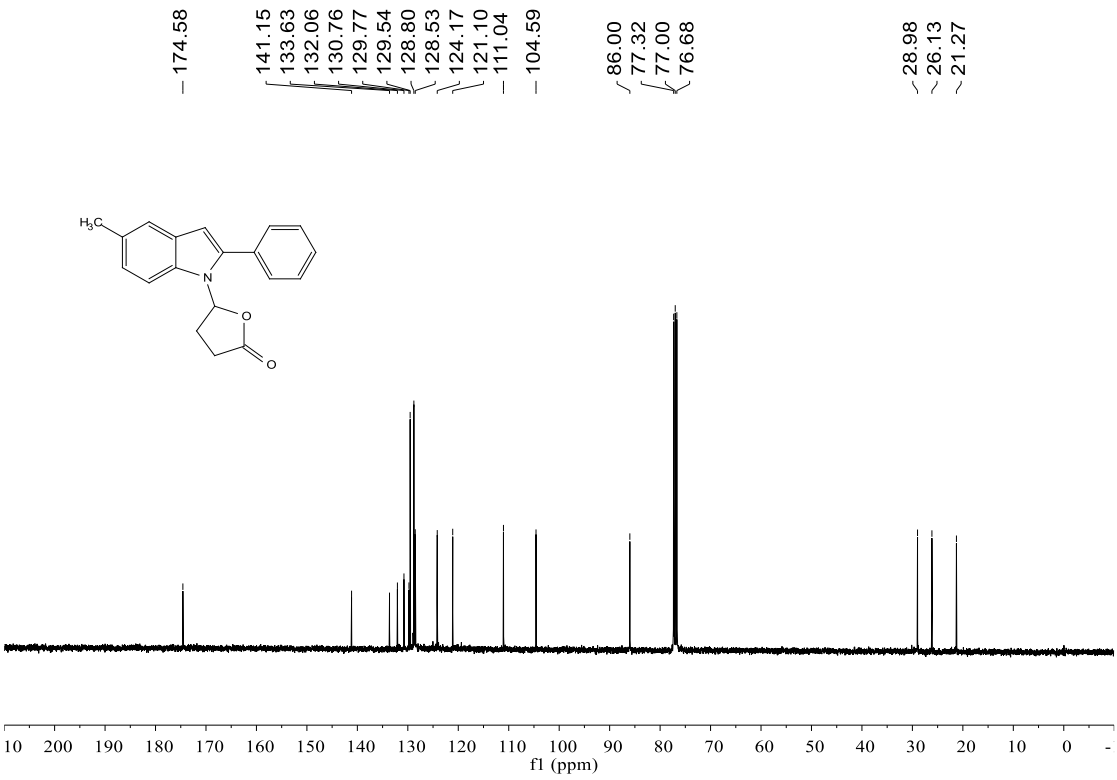
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3z



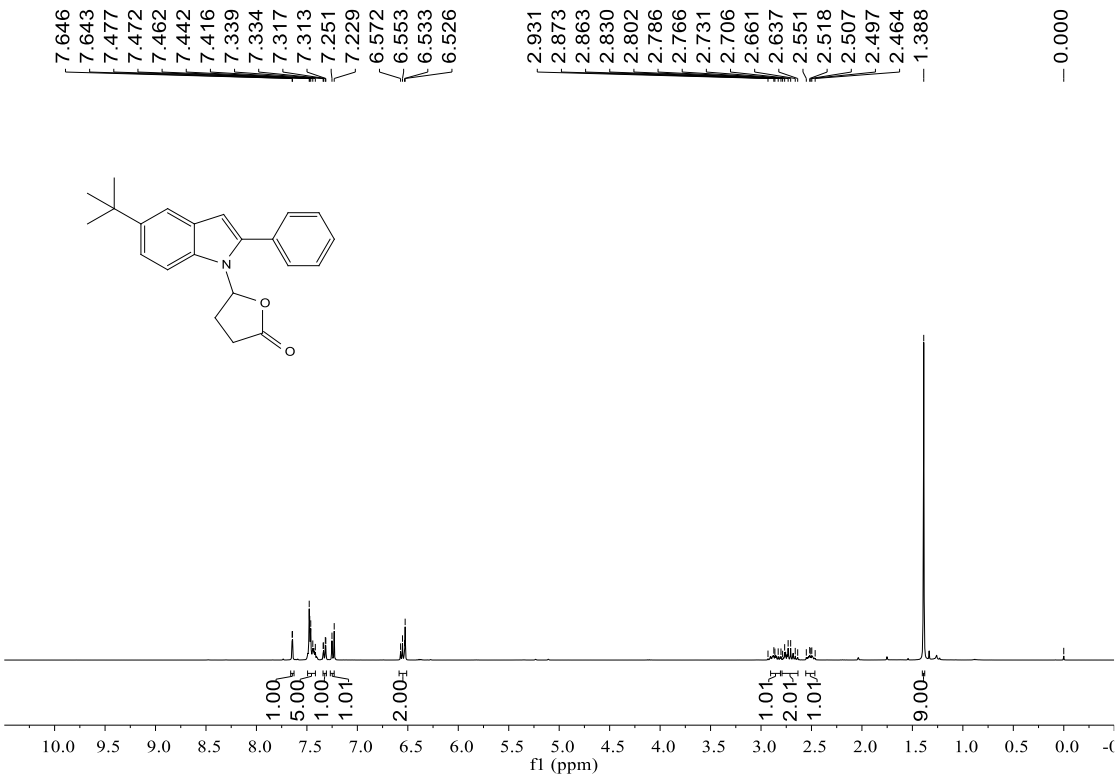
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3aa



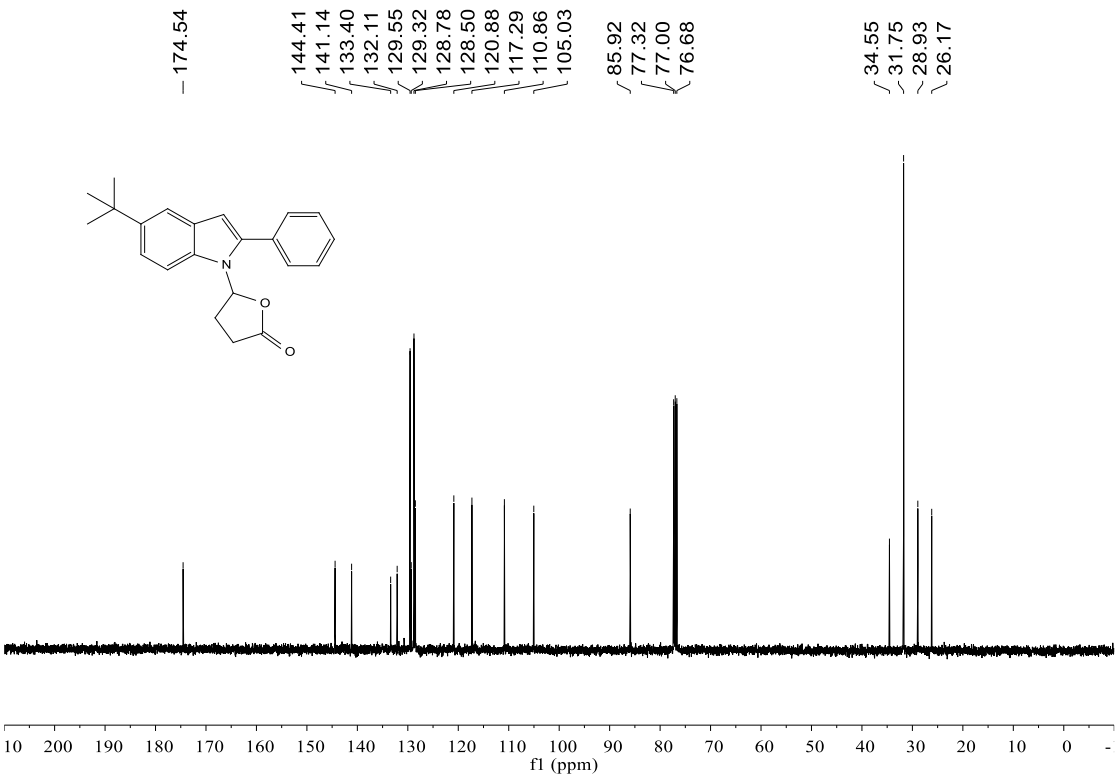
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3aa



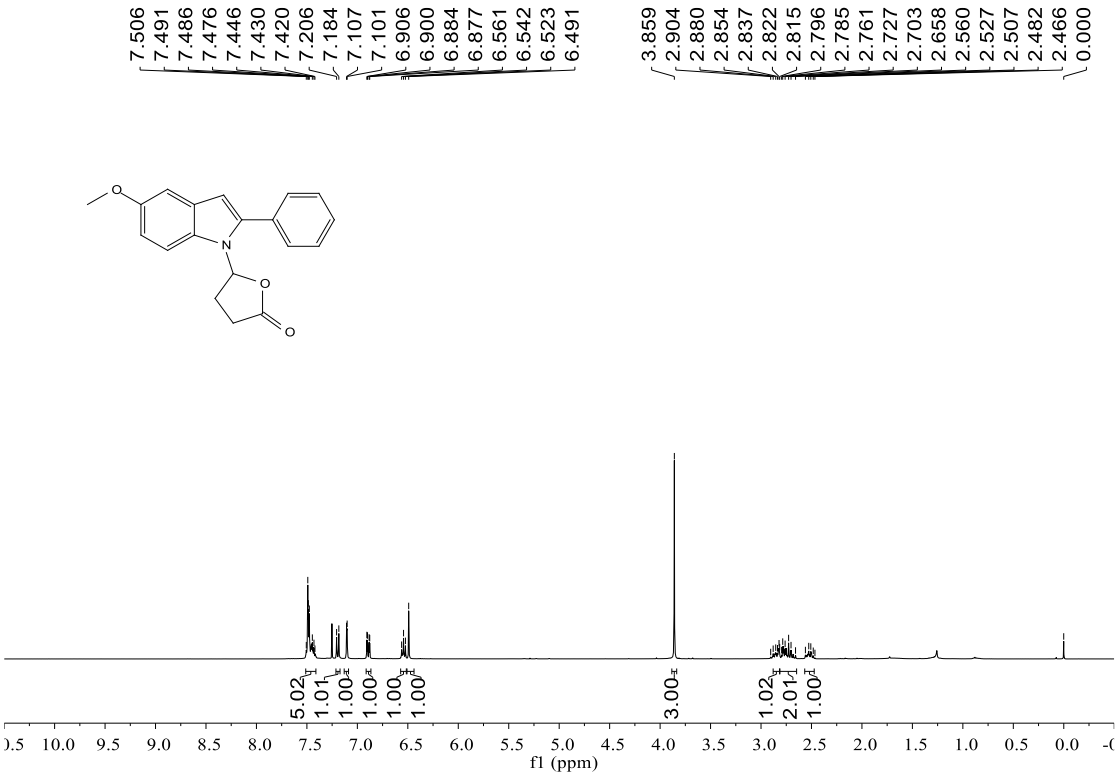
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ab



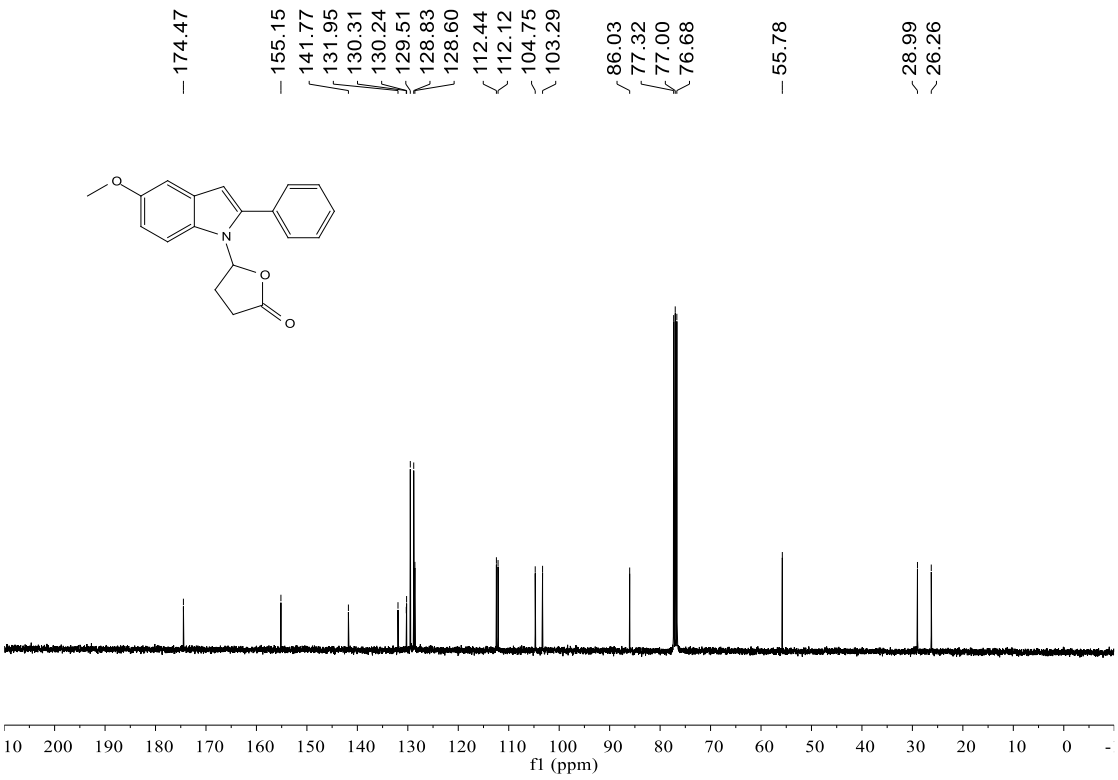
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3ab



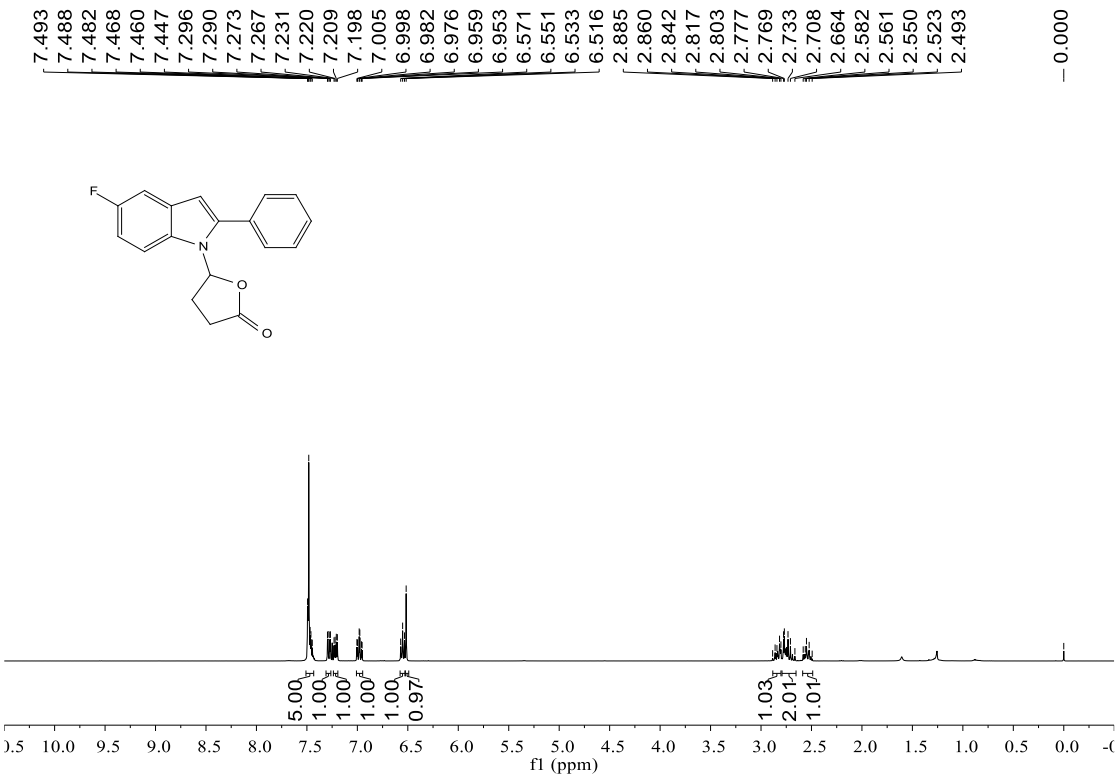
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ac



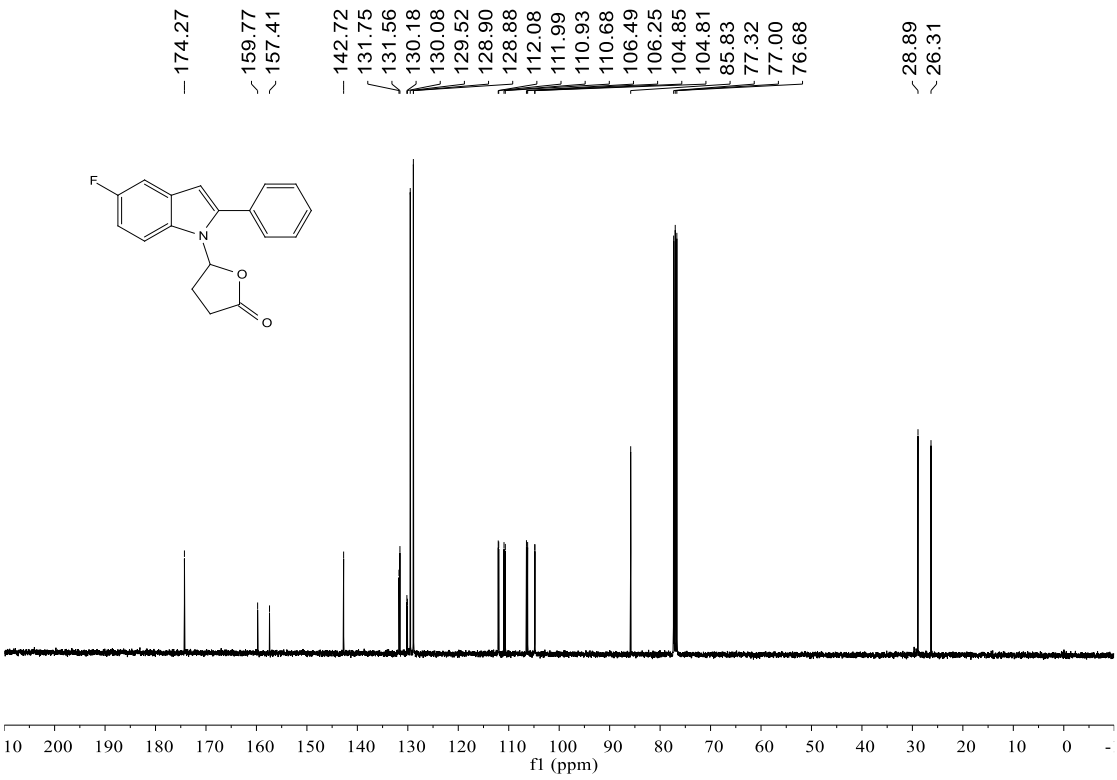
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3ac



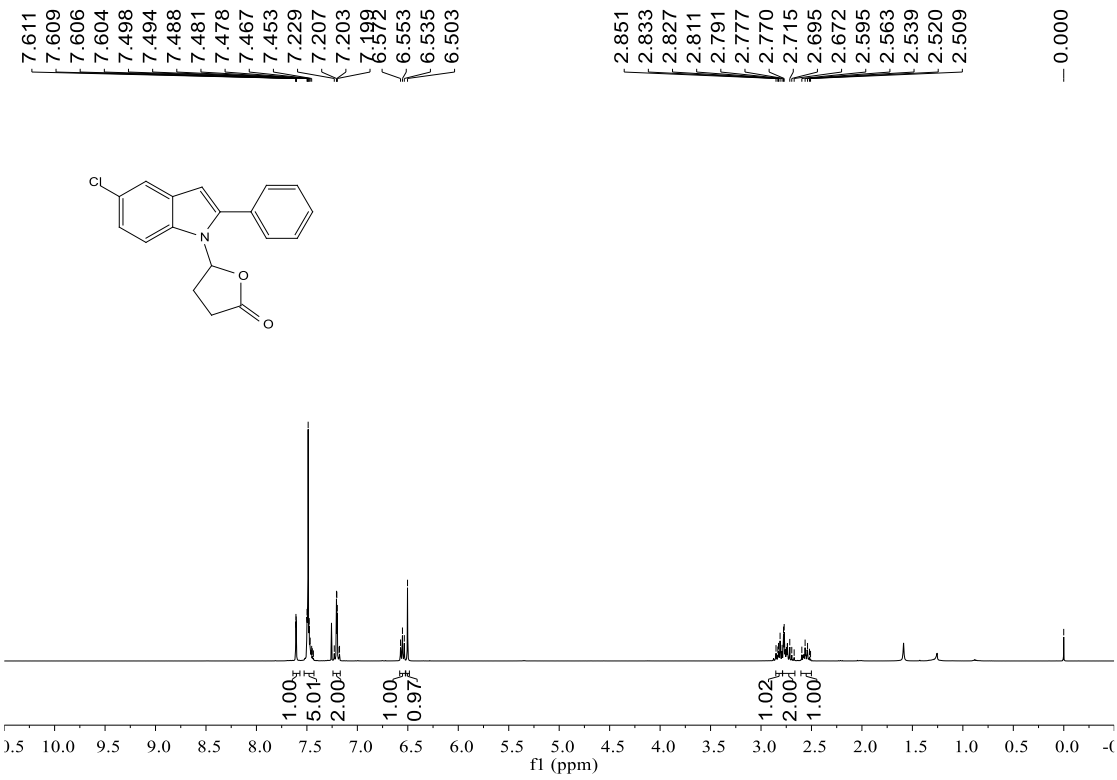
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ad



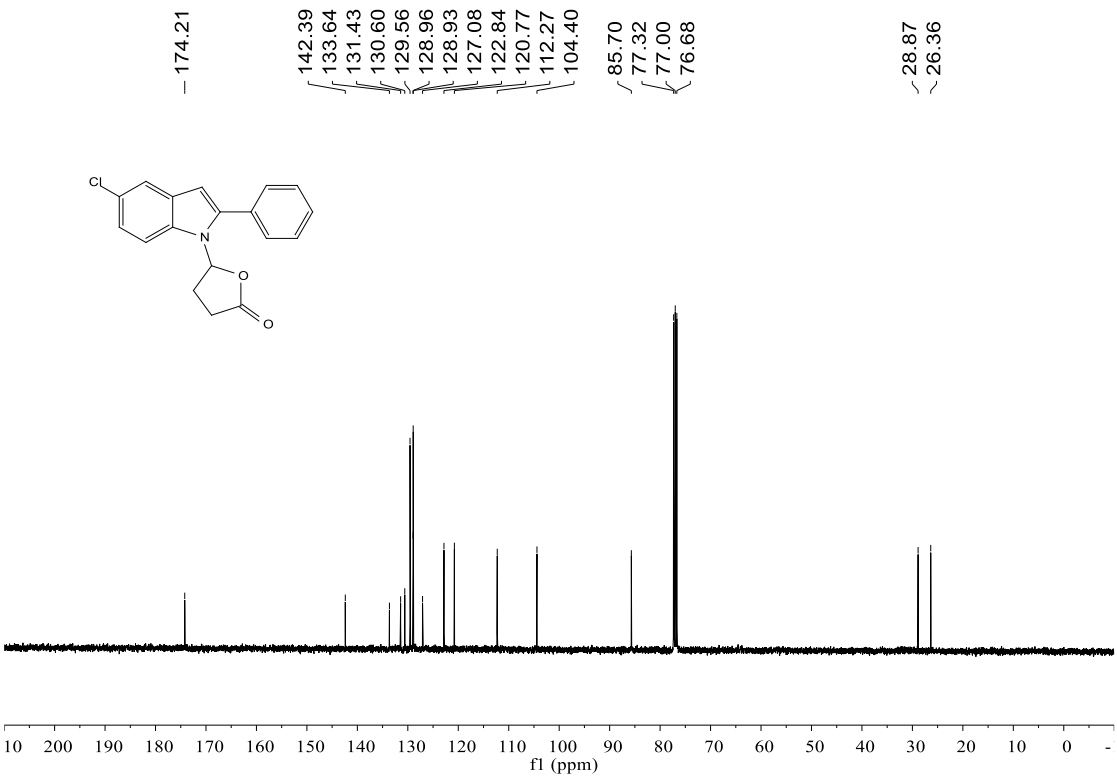
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3ad



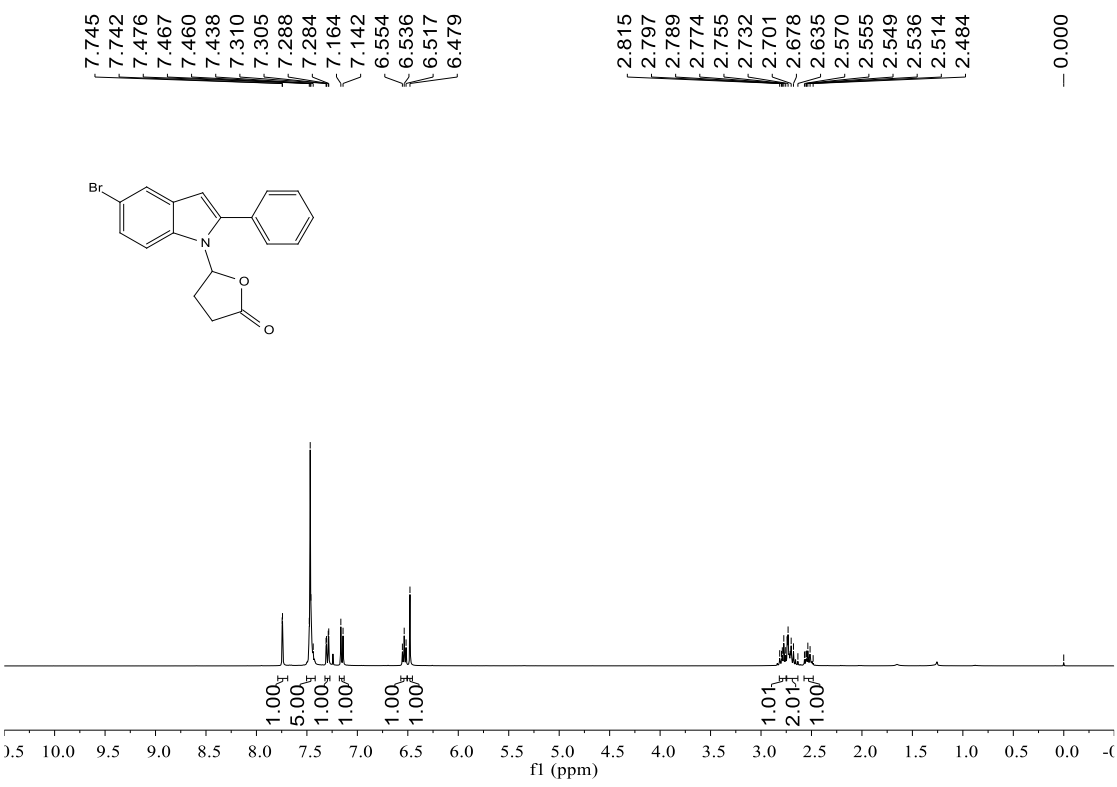
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ae



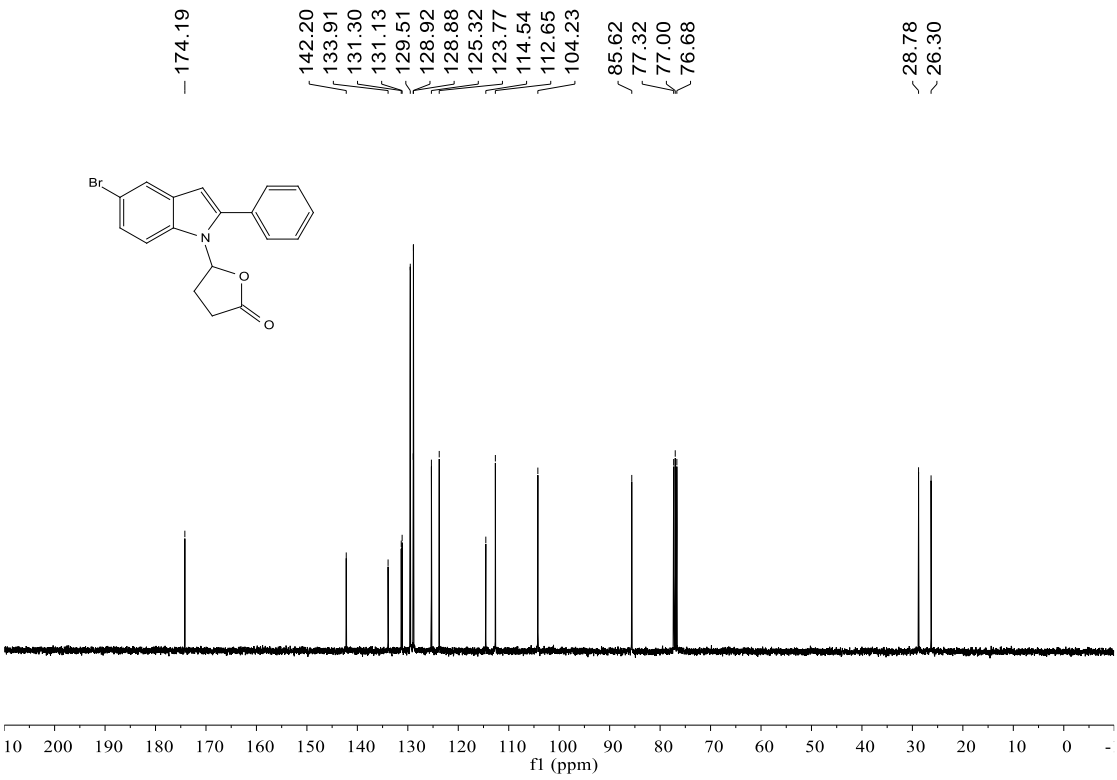
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3ae



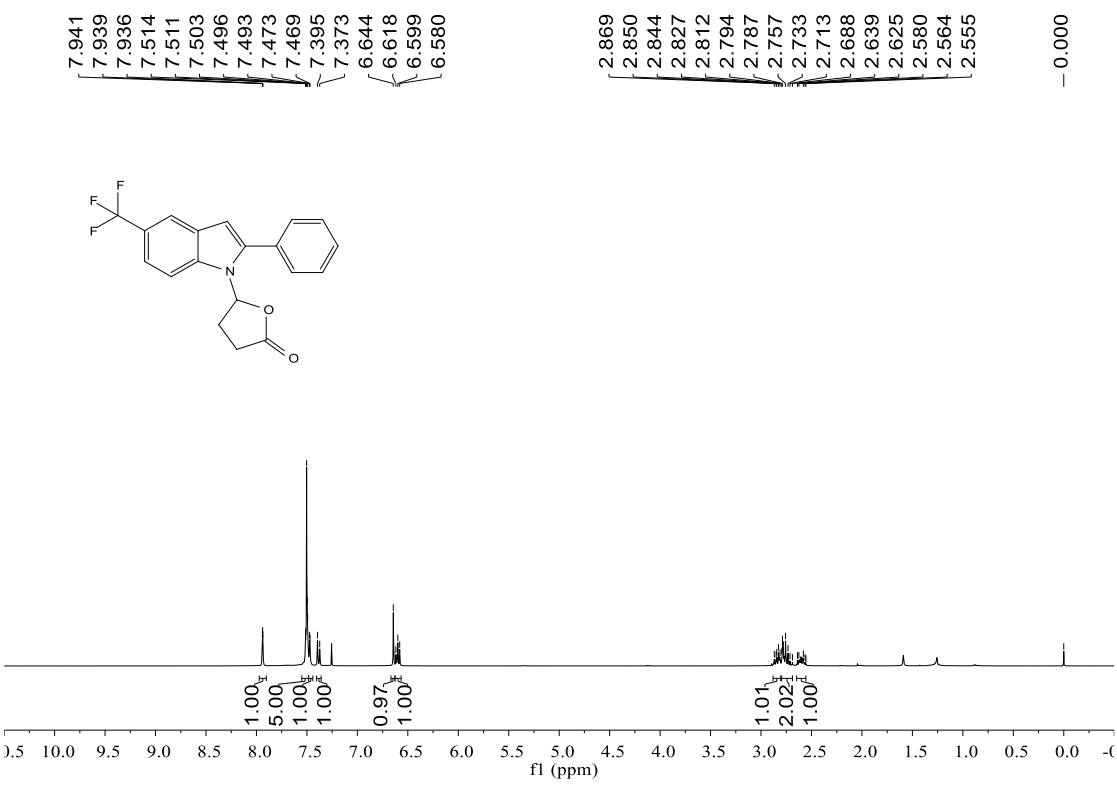
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3af



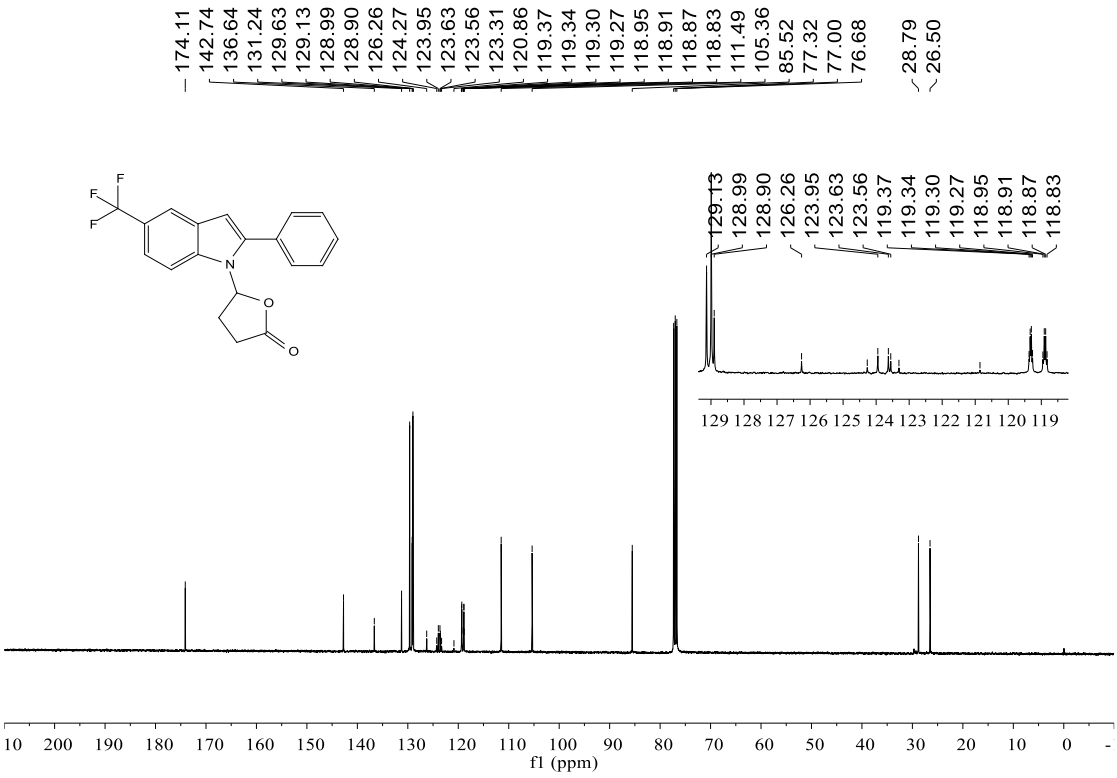
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3af



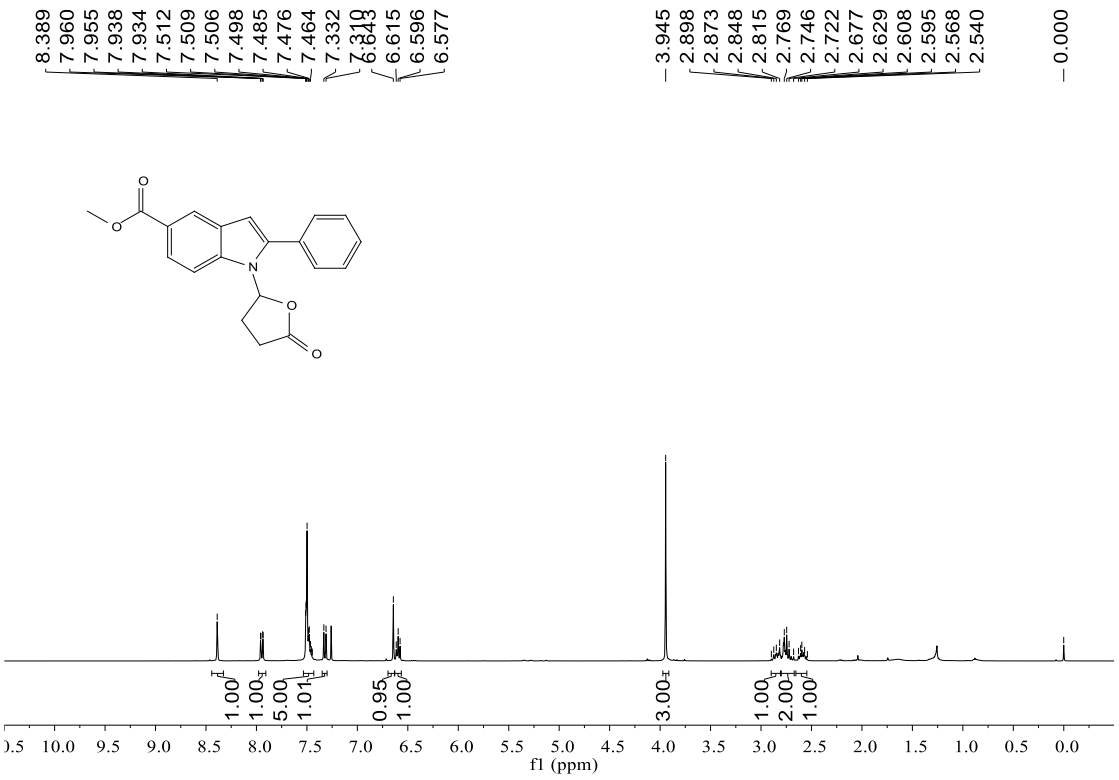
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ag



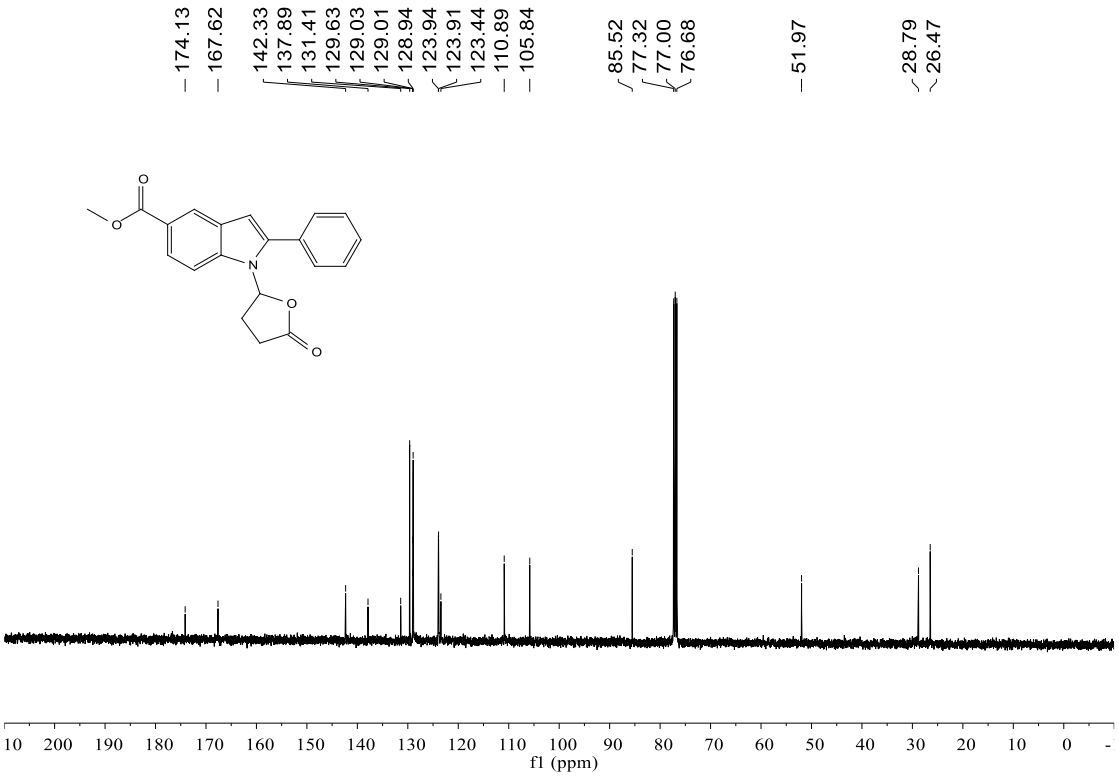
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3ag



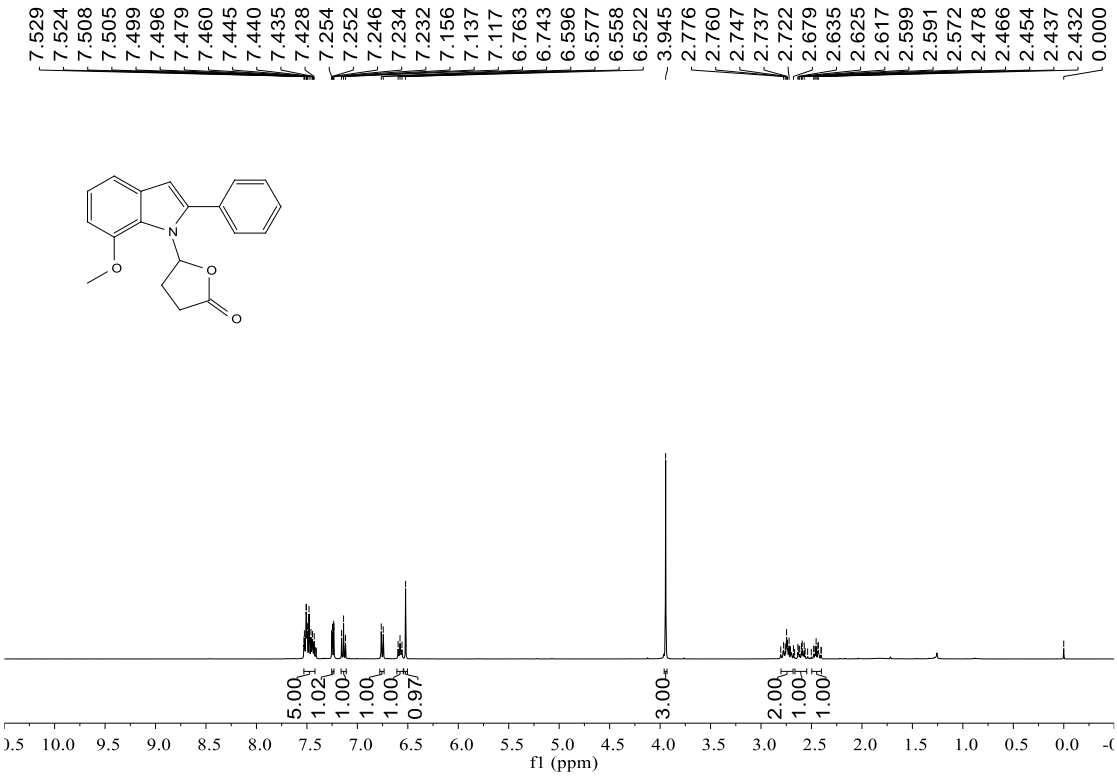
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ah



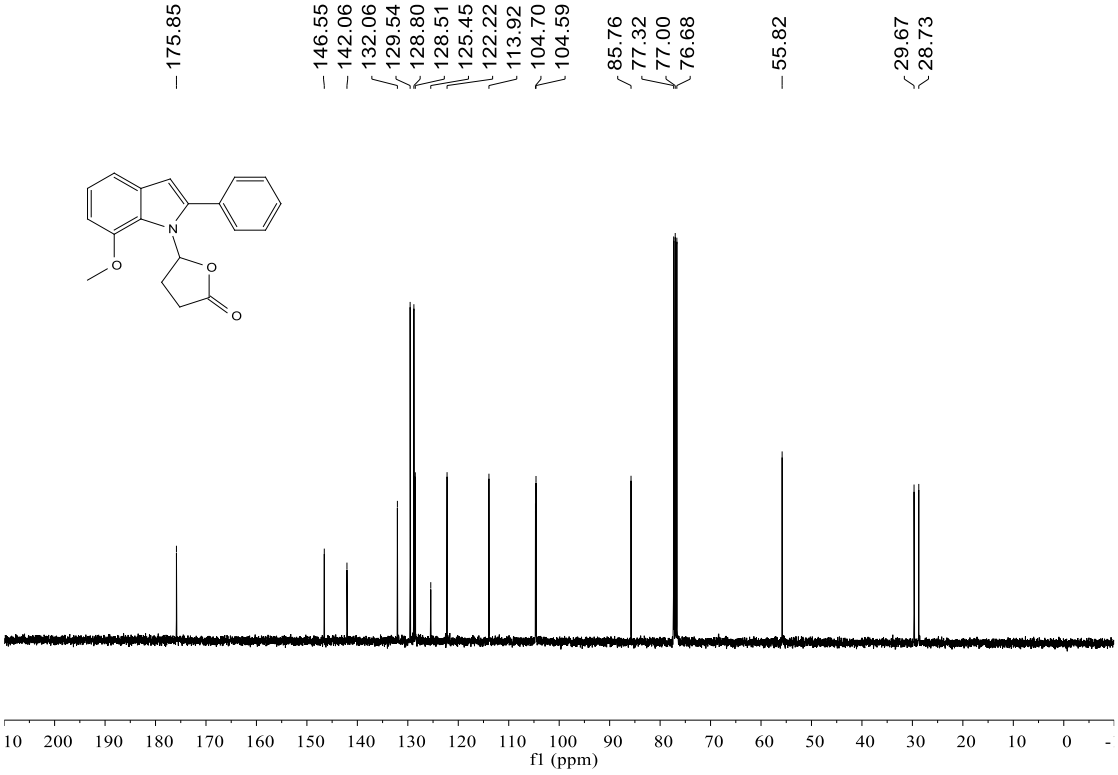
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3ah



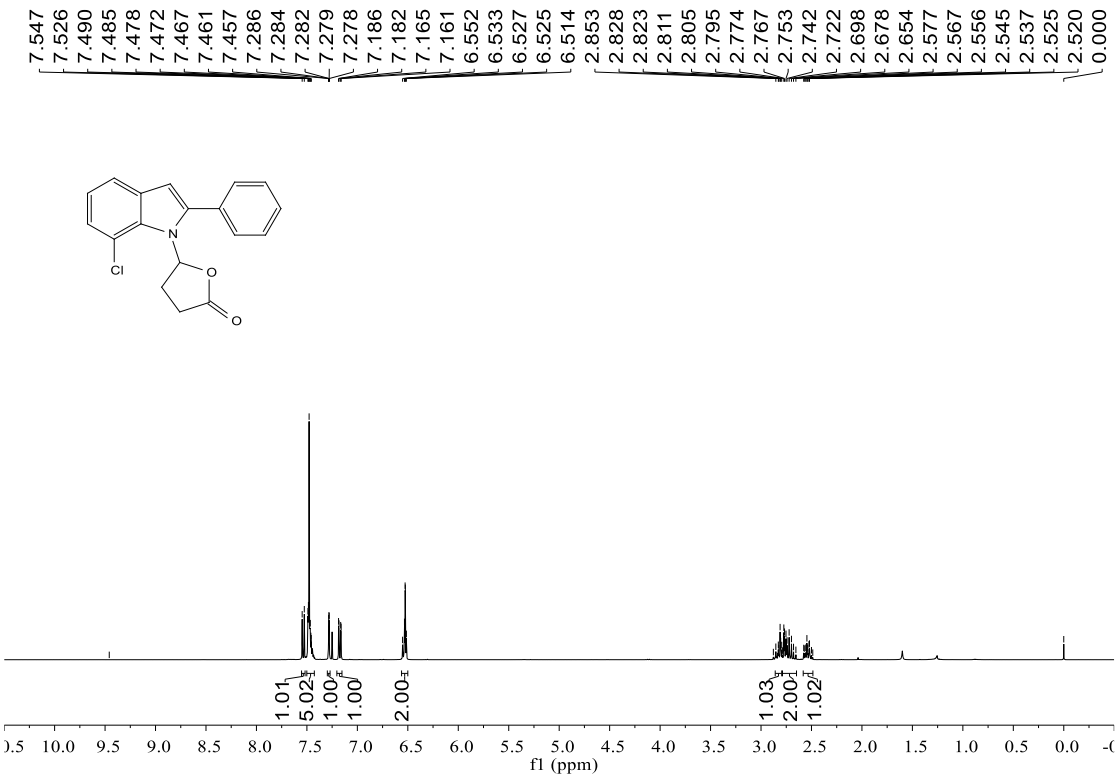
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ai



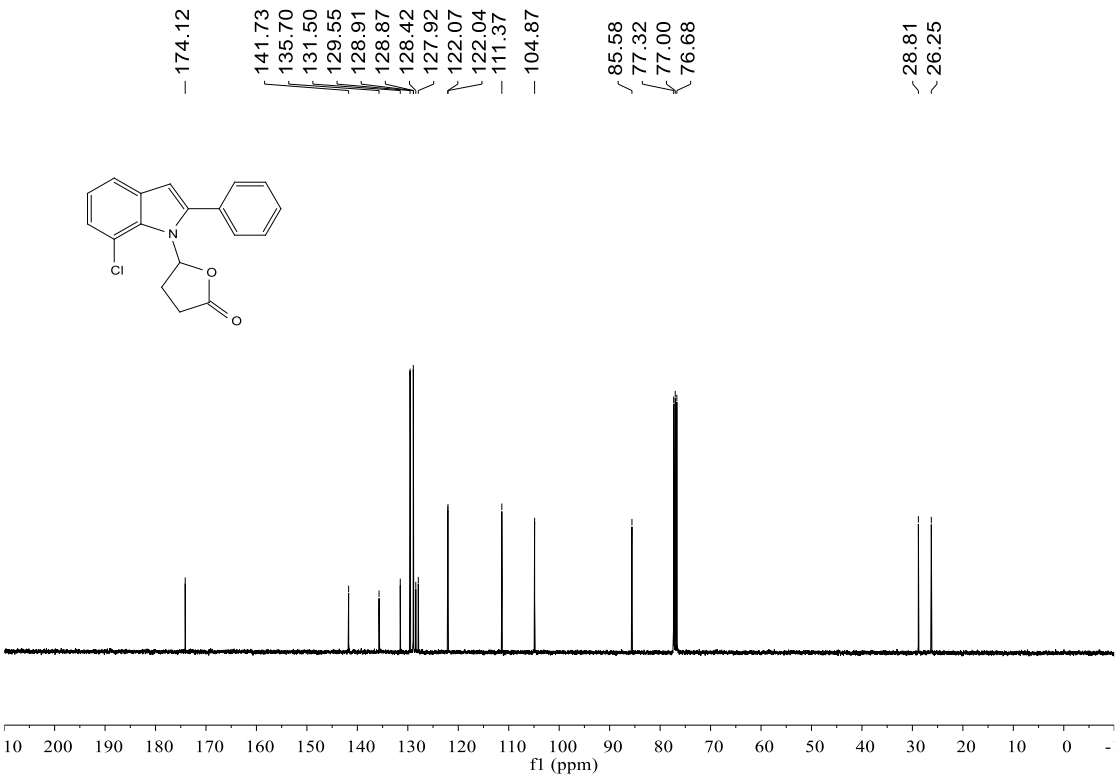
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3ai



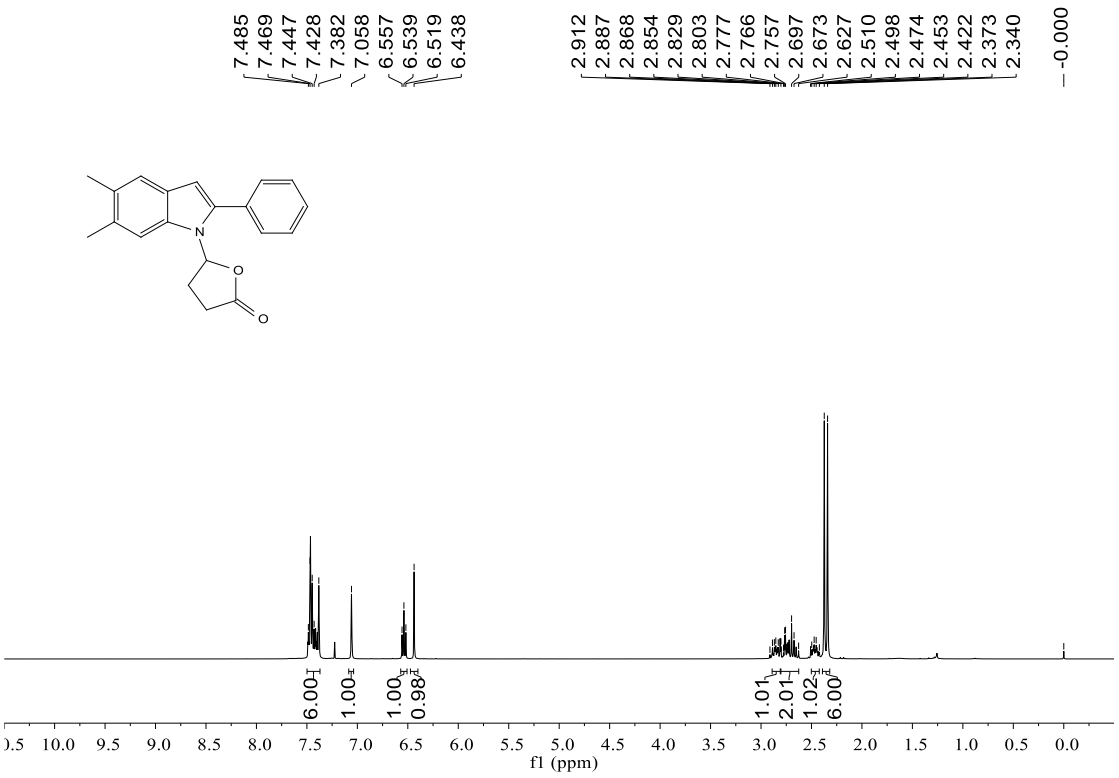
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3aj



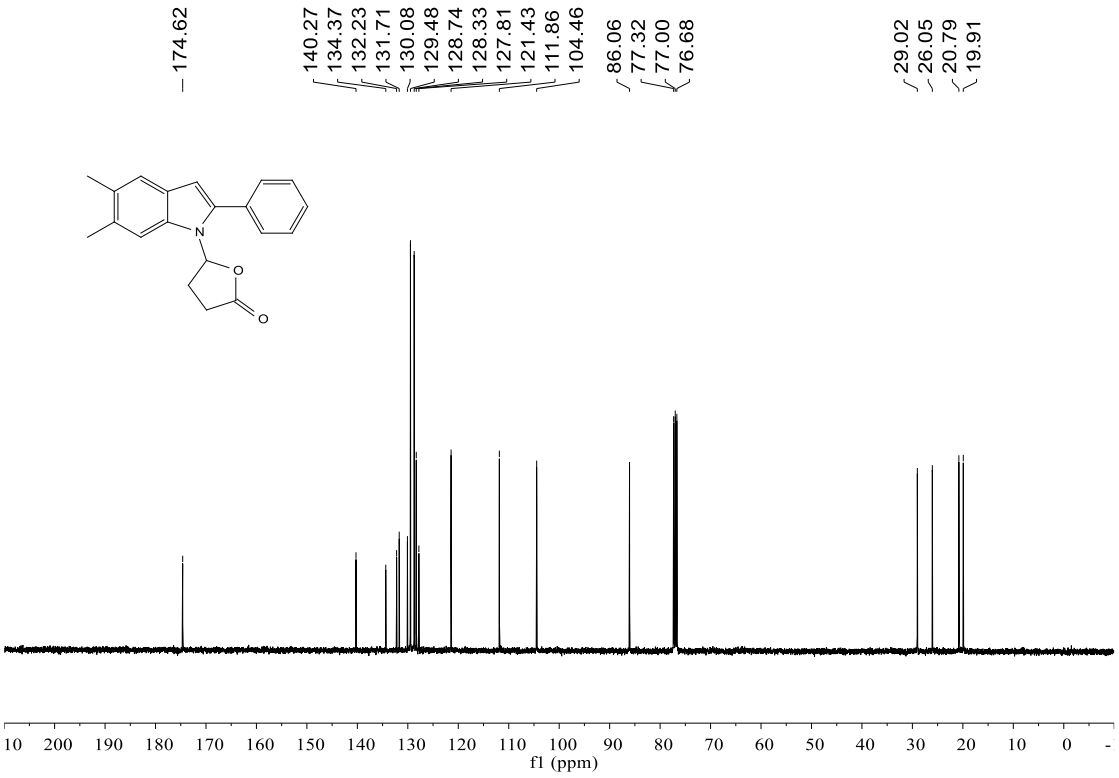
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3aj



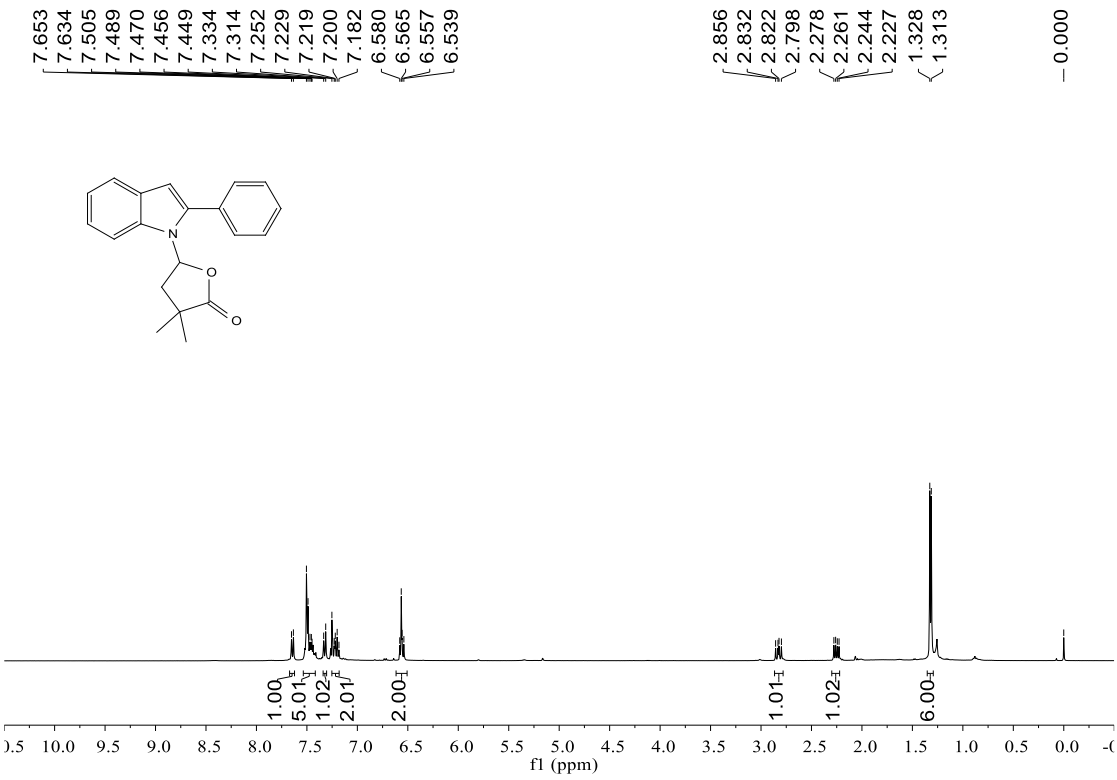
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3ak



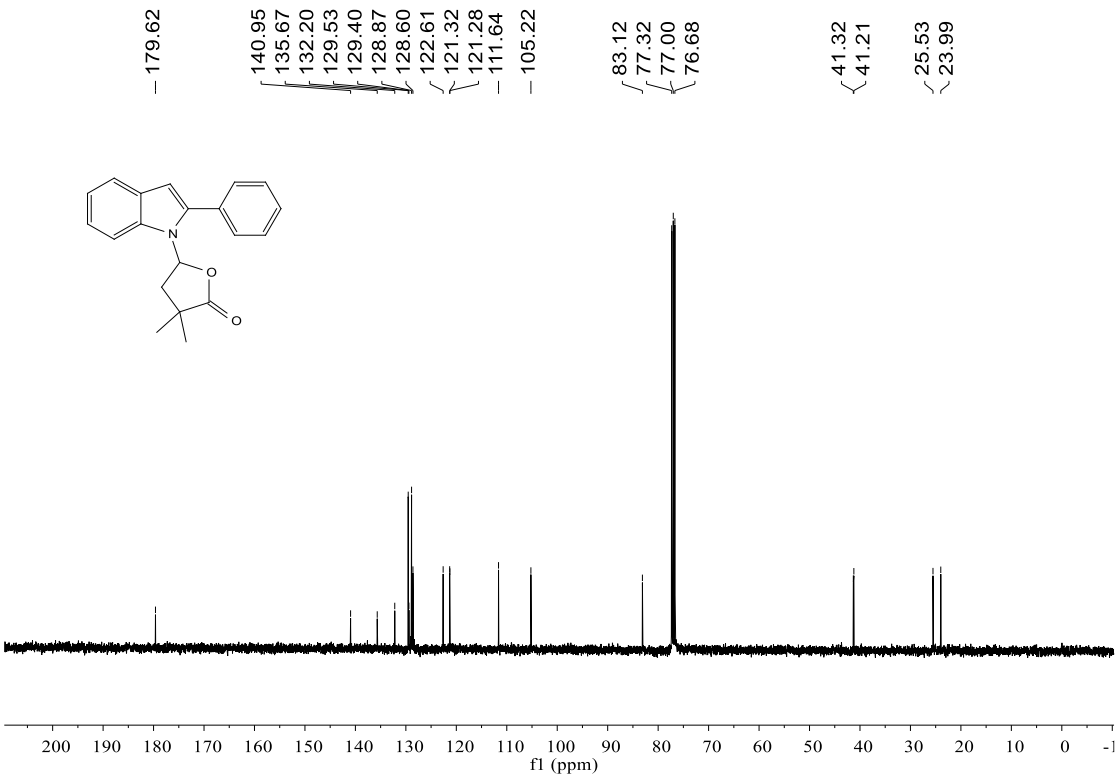
^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3ak



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3an



^{13}C NMR (100 MHz, $CDCl_3$) spectrum of 3an



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