Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information for

Pd/β-cyclodextrin-catalyzed C-H functionalization in water: a greener approach to regioselective arylation of (NH)-indoles with aryl bromides

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1. Experiment Details

1) General Methods

All reagents were obtained from Sigma-Aldrich Chemical Co. or Alfa Aesar. ^{1}H-NMR spectra were recorded on a Bruker Avance DPX 400 (400 MHz) or 500 (500 MHz) spectrometer using $CDCl_3$ or DMSO-d6 as the solvent. The chemical shifts are reported in δ (ppm) values. Coupling constants are reported in hertz (Hz). The following abbreviations are used to designate the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet and m = multiplet. ^{13}C-NMR spectra were recorded on a Bruker Avance DPX 400 (400 MHz) or 500 (500 MHz) spectrometer at 100 or 125 MHz using $CDCl_3$ as the solvent. All compounds were characterized by ^{1}H-NMR, ^{13}C-NMR and melting point determination (for solids); melting points were uncorrected.

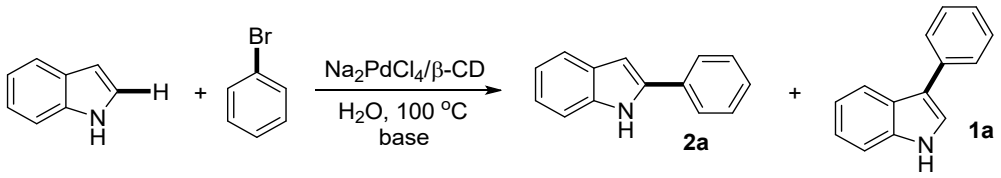
2) General procedures

(1) Procedure for 3-arylindoles (1a-u)

Na_2PdCl_4 (14.7 mg, 5.0 mol%), β-cyclodextrin (β-CD) (56.8 mg, 5.0 mol%), KOH (112 mg, 2.0 mmol) and 3.0 ml H_2O in 25 ml round-bottom flask equipped with condenser and magnetic stirring were preheated at 60 ^{o}C for 0.5h. Then, an indole (1.0 mmol) and an aryl bromide (1.0 mmol) were added. The resulting reaction mixture was stirred and heated at 80 ^{o}C until the coupling reaction reached completion (monitored by GC-MS). After extracted with EtOAc (3 x 15 mL), dried over Na_2SO_4, and concentrated in vacuo, the product was obtained by purification with silica gel flash chromatography. Spectral data matches that reported in the literature.

(2) Screening of the bases of C2-selective arylation

Table S1. Optimization of the bases.



Entry	Base	Time (h)	Yield (**2a**/**1a**, %)
1	Na_2CO_3	8	32/46
2	K_2CO_3	8	39/49
3	**KOAc**	**4**	**75/10**
4	NaOAc	4	52/14
5	LiOH	2	35/47
6	NaOH	2	26/52

(3) Procedure for 2-arylindoles (2a-i)

Na_2PdCl_4 (14.7 mg, 5.0 mol%), β-cyclodextrin (β-CD) (56.8 mg, 5.0 mol%), KOAc (196 mg, 2.0 mmol) and 3.0 ml H_2O in 25 ml round-bottom flask equipped with condenser and magnetic stirring were preheated at 60 ^{o}C for 0.5h. Then, an indole (1.0 mmol) and an aryl bromide (1.0 mmol) were added. The resulting reaction mixture was stirred and heated at 100 ^{o}C until the coupling reaction reached completion (monitored by GC-

MS). After extracted with EtOAc (3 x 15 mL), dried over Na_2SO_4, and concentrated in vacuo, the product was obtained by purification with silica gel flash chromatography. Spectral data matches that reported in the literature.

(4) Procedure for the coupling of 5-nitroindole and PhBr

Na_2PdCl_4 (14.7 mg, 5.0 mol%), β-cyclodextrin (β-CD) (56.8 mg, 5.0 mol%), KOH (112 mg, 2.0 mmol) and 3.0 ml H_2O in 25 ml round-bottom flask equipped with condenser and magnetic stirring were preheated at 60 °C for 0.5h. Then, 5-nitroindole (1.0 mmol) and PhBr (1.0 mmol) were added. The reaction mixture was stirred and heated at 80 °C for 24 hours and the reaction did not proceed under the conditions (monitored by GC-MS).

(5) Procedure for the coupling of *N*-methylindole and PhBr

Na_2PdCl_4 (14.7 mg, 5.0 mol%), β-cyclodextrin (β-CD) (56.8 mg, 5.0 mol%) or SDS (57.6 mg, 20.0 mol%), KOH (112 mg, 2.0 mmol) and 10 ml H_2O in 25 ml round-bottom flask equipped with condenser and magnetic stirring were preheated at 60 °C for 0.5h. Then, *N*-methylindole (1.0 mmol) and PhBr (1.0 mmol) were added. The resulting reaction mixture was stirred and heated at 100 °C for 24 hours. After extracted with EtOAc (3 x 15 mL), dried over Na_2SO_4, and concentrated in vacuo, the product was obtained by purification with silica gel flash chromatography.

(6) Procedure for the coupling of 2- or 3-phenylindole and PhBr

Na_2PdCl_4 (14.7 mg, 5.0 mol%), β-cyclodextrin (β-CD) (56.8 mg, 5.0 mol%), KOH (112 mg, 2.0 mmol) and 10 ml H_2O in 25 ml round-bottom flask equipped with condenser and magnetic stirring were preheated at 60 °C for 0.5h. Then, 2- or 3-phenylindole (1.0 mmol) and PhBr (1.0 mmol) were added. The resulting reaction mixture was stirred and heated at 100 °C for 24 hours. After extracted with EtOAc (3 x 15 mL), dried over Na_2SO_4, and concentrated in vacuo, the product was obtained by purification with silica gel flash chromatography.

(7) The proposed Na_2PdCl_4/β-CD-catalyzed mechanism of C2-arylation of indole with PhBr in water.

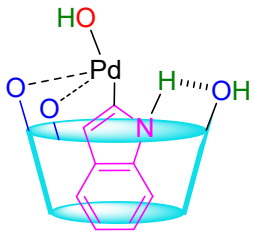
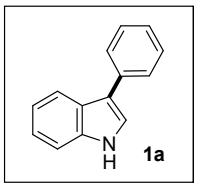


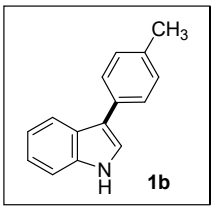
Fig. S1 the C2-palladation indole intermediate.

4) Characterization data



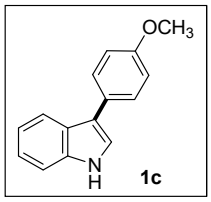
3-Pheny-1*H*-lindole[1] (**1a**)

White solid, m.p. 108-109 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.05 (br s, 1H), 7.94 (d, J = 7.7 Hz, 1H), 7.65 (d, J = 7.3 Hz, 2H), 7.45-7.41 (t, 2H), 7.35 (d, J = 7.5 Hz, 1H), 7.28 (d, J = 7.5 Hz, 1H), 7.26-7.16 (m, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 136.8, 135.7, 129.0, 127.6, 126.1, 125.8, 122.5, 122.1, 120.5, 119.9, 118.3, 111.6.



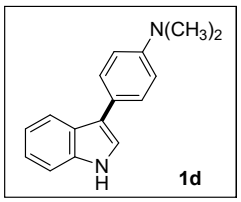
3-(*p*-Tolyl)-1*H*-indole[2] (**1b**)

Pale-yellow solid, m.p. 112-113 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.05 (br s, 1H), 7.93 (d, J = 7.8 Hz, 1H), 7.56 (d, J = 8.1 Hz, 2H), 7.36 (t, J = 8.9 Hz, 1H), 7.26-7.23 (m, 4H), 7.20-7.17 (m, 1H), 2.39 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 136.6, 135.6, 132.5, 129.5, 127.4, 125.7, 122.3, 122.2, 121.5, 120.2, 120.1, 119.8, 118.1, 111.4, 21.2.



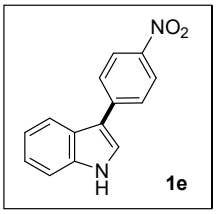
3-(4-Methoxyphenyl)-1*H*-indole[3] (**1c**)

Pale-yellow solid, m.p. 130-131 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.13 (br s, 1H), 7.95 (d, J = 5.0 Hz, 1H), 7.63 (dd, J = 1.4, 4.8 Hz, 2H), 7.40 (t, J = 6.6 Hz, 1H), 7.29-7.22 (m, 3H), 7.05 (dd, J = 1.4, 3.7 Hz, 2H), 3.89 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 158.2, 136.7, 128.7, 128.3, 126.0, 122.4, 121.4, 120.3, 119.9, 118.0, 114.4, 111.5, 55.5



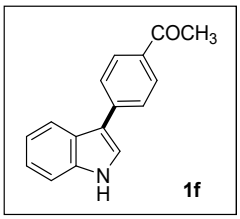
3-(4-N,N-dimethylaminophenyl)-1*H*-indole [4] (**1d**)

Pale-yellow solid, m.p. 88-90 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.15 (br s, 1H), 7.91 (dd, J = 1.2, 1.0 Hz, 1H,), 7.55 (d, J = 8.8 Hz, 2H), 7.39-7.32 (m, 2H), 7.24-7.14 (m, 2H), 6.86 (d, J = 8.8 Hz, 2H), 2.98 (s, 6H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 136.5, 130.8, 128.8, 128.3, 128.2, 126.0, 122.1, 120.7, 119.9, 119.8, 118.3, 113.3, 112.3, 111.3, 40.9.



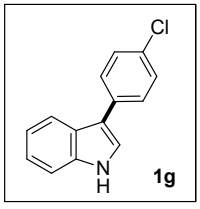
3-(4-Nitrophenyl)-1*H*-indole[2] (**1e**)

Light yellow solid, m.p. 149-151 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.48 (br s, 1H), 8.22 (d, J = 8.0Hz, 2H), 7.88 (d, J = 8.0Hz, 1H), 7.74 (d, J = 8.0Hz, 2H), 7.45-7.40 (m, 2H), 7.26-7.19 (m, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 145.6, 142.8, 136.9, 127.1, 125.1, 124.3, 123.7, 123.2, 121.3, 119.5, 116.3, 111.9.



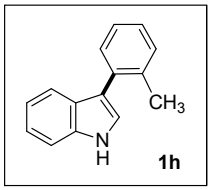
3-(4-Acetylphenyl)-1*H*-indole[4] (**1f**)

White solid, m.p. 140-141 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.61 (br s, 1H), 8.00 (d, J = 8.0 Hz, 2H), 7.94 (d, J = 7.3 Hz, 1H), 7.73 (d, J = 8.0 Hz, 2H), 7.40 (d, J = 7.6 Hz, 2H), 7.26-7.20 (m, 2H), 2.60 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 136.7, 134.1, 131.6, 128.9, 128.6, 125.5, 122.6, 121.9, 120.5, 119.6, 117.2, 111.5, 65.7.



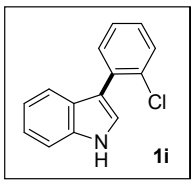
3-(4-Chlorophenyl)-1*H*-indole[5] (**1g**)

White solid, m.p. 138-140 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.19 (br s, 1H), 7.87 (d, J = 7.9 Hz, 1H), 7.57 (d, J = 7.9 Hz, 2H), 7.39-7.28 (t, 3H), 7.26 (s, 1H), 7.22-7.18 (m, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 136.7, 134.1, 131.6, 129.0, 128.6, 125.6, 122.6, 122.0, 120.6, 119.6, 117.1, 111.6



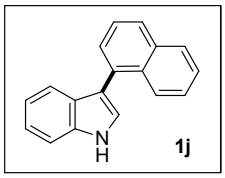
3-(*o*-Tolyl)-1*H*-indole[6] (**1h**)

Colorless oil. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.12 (br s, 1H), 7.58 (dd, J = 8.5, 1.3 Hz, 1H), 7.49-7.42 (m, 3H), 7.40-7.23 (m, 3H), 7.20 (t, J = 9.9 Hz, 1H), 7.17 (d, J = 3.0 Hz, 1H), 2.37 (s, 3 H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 137.0, 136.0, 134.6, 131.0, 130.5, 127.1, 126.9, 125.8, 123.0, 122.3, 120.3, 120.1, 117.6, 111.4, 21.0



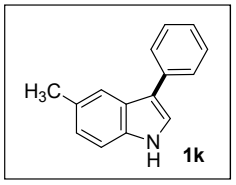
3-(2-Chlorophenyl)-1*H*-indole[7] (**1i**)

Light yellow solid, m.p 72-74 ^{o}C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 7.98 (br s, 1H), 7.81 (dt, J = 1.0, 3.4 Hz, 1H), 7.69-7.64 (m, 2H), 7.44-7.31 (m, 4H), 7.30-7.24 (m, 2H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 136.0, 134.0, 133.4, 132.2, 130.4, 128.0, 127.0, 126.7, 124.6, 122.6, 120.5, 120.3, 114.8, 111.4.



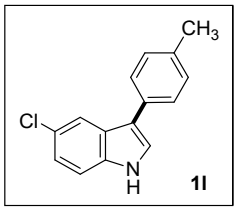
3-(Naphthalen-1-yl)-1*H*-indole[7] (**1j**)

Light brown solid, m.p 32-33 ^{o}C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.17 (d, J = 10.7 Hz, 1H), 8.12 (br s, 1H), 7.99 (dt, J = 6.6, 10.0 Hz, 2H), 7.70-7.57 (m, 4H), 7.50-7.46 (m, 2H), 7.37-7.30 (m, 2H), 7.23 (q, J = 9.8 Hz, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 136.2, 134.2, 133.2, 132.8, 128.5, 128.0, 127.9, 127.4, 126.8, 126.0, 125.9, 123.8, 122.6, 120.5, 120.3, 116.6, 111.5.



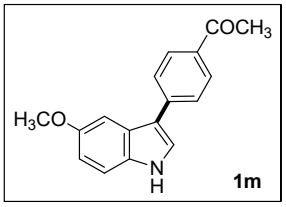
5-Methyl-3-phenyl-1*H*-indole[8] (**1k**)

White solid, m.p. 106-108 ^{o}C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 7.98 (br s, 1H), 7.72 (s, 1H), 7.68 (d, J = 7.4 Hz, 2H), 7.44-7.41 (t, 2H), 7.29-7.18 (m, 3H), 7.05 (d, J = 8.0 Hz, 1H), 2.46 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 135.8, 135.1, 129.6, 128.8, 127.5, 126.0, 125.9, 124.0, 122.0, 119.4, 117.9, 111.1, 21.6.



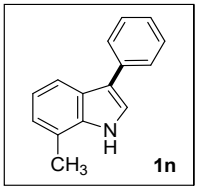
3-(4-Methylphenyl)-5-chloro-1*H*-indole (**1l**)

White solid, m.p. 99-100 ^{o}C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.12 (br s, 1H), 7.86 (d, J = 2.0 Hz, 1H), 7.48 (d, J = 8.1 Hz, 2H), 7.27-7.24 (m, 4H), 7.16 (dd, J = 2.0, 2.0 Hz, 1H), 2.39 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 136.0, 135.0, 132.0, 129.6, 127.3, 127.0, 126.8, 125.9, 122.7, 122.6, 119.3, 119.2, 118.0, 112.3, 21.2. HRMS (EI) [M^+]/z calcd. 241.72, found 241.00.



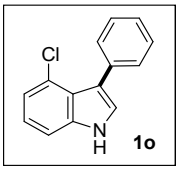
3-(4-Acetylphenyl)-5-methoxy-1*H*-indole (**1m**)

White solid, m.p. 118-119 ^{o}C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.45 (br s, 1H), 7.97 (d, J = 8.0 Hz, 2H), 7.65 (t, 2H), 7.35 (d, J = 10.5 Hz, 2H), 7.26 (d, J = 8.8 Hz, 1H), 6.87 (d, J = 8.7 Hz, 1H), 3.81 (s, 3H), 2.56 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 155.1, 141.1, 134.4, 132.0, 129.1, 129.0, 127.4, 126.8, 125.9, 123.8, 112.9, 112.4, 101.8, 56.0, 26.5. HRMS (EI) [M^+]/z calcd. 265.31, found 265.00.



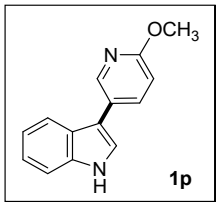
7-Methyl-3-phenyl-1*H*-indole[7] (**1n**)

Light yellow solid, m.p 109-111 ^{o}C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.99 (br s, 1H), 7.96 (d, J = 6.6 Hz, 1H), 7.79 (d, J = 6.1 Hz, 2H), 7.57 (t, J = 12.8 Hz, 2H), 7.42 (t, J = 12.3 Hz, 1H), 7.30 (d, J = 2.1 Hz, 1H), 7.26 (t, J = 12.6 Hz, 1H), 7.17 (d, J = 5.9 Hz, 1H), 2.54 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 136.4, 136.0, 129.0, 127.7, 126.2, 125.5, 123.1, 121.9, 120.8, 120.7, 118.8, 117.8, 16.7.



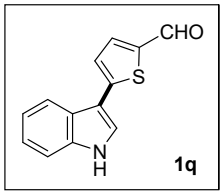
3-Phenyl-4-chloro-1*H*-indole (**1o**)

Colorless oil. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.14 (br s, 1H), 7.79 (dt, J = 2.0, 5.5 Hz, 2H), 7.66-7.56 (m, 3H), 7.40-7.20 (m, 3H), 7.16 (d, J = 2.9 Hz, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 137.8, 135.6, 131.4, 127.9, 127.0, 126.6, 125.1, 123.4, 123.1, 121.7, 119.0, 110.7.



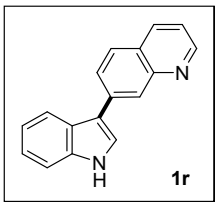
3-(6-Methoxylpyridin-3-yl)-1*H*-indole[10] (**1p**)

Yellow solid, m.p. 97-98 ºC. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.63 (br s, 1H), 8.48 (d, J = 2.1 Hz, 1H), 7.82 (dd, J = 3.0, 2.9 Hz, 1H), 7.64 (d, J = 9.7 Hz, 1H), 7.35 (d, J = 9.9 Hz, 1H), 7.26-7.13 (m, 2H), 6.81 (d, J = 10.8 Hz, 1H), 6.74 (s, 1H), 3.97 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ =163.7, 143.2, 137.0, 136.1, 135.1, 129.2, 122.5, 122.3, 120.6, 120.5, 111. 4, 111.0, 99.8, 55.9.



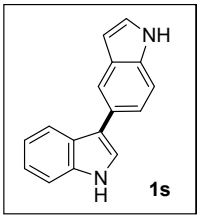
5-(1*H*-Indol-3-yl)thiophene-2-carbaldehyde (**1q**)

Yellow oil. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.99 (br s, 1H), 7.47 (d, J = 9.9 Hz, 1H), 7.35 (t, J = 11.1 Hz, 1H), 7.21-7.17 (t, 1H), 7.08-7.04 (t, 1H), 6.86 (t, J = 4.6 Hz, 1H), 6.81 (d, J = 2.8 Hz, 1H), 6.64 (t, J = 5.8 Hz, 1H), 6.06 (s, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 150.5, 136.7, 129.4, 126.7, 125.5, 123.4, 122.2, 119.7, 119.6, 118.9, 111.4, 110.0.



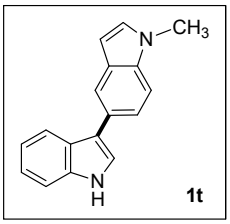
7-(1*H*-Indol-3-yl)quinoline (**1r**)

Brown solid, m.p. 238-240 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.33 (br s, 1H), 7.87 (s, 1H), 7.68-7.56 (m, 5H), 7.41 (t, J = 9.9 Hz, 1H), 7.21-7.15 (m, 2H), 7.52-7.50 (dt, J = 2.9, 1.2 Hz, 2H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 154.8, 146.0, 138.6, 136.9, 129.5, 128.2, 127.8, 122.4, 122.2, 120.6, 120.4, 117.9, 112.1, 110.9, 106.8, 99.7



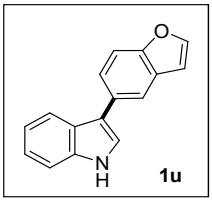
1*H*,1'*H*-3,5'-biindole (**1s**)

Pale-brown solid, m.p. 193-195 ºC. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.14 (br s, 2H), 7.99 (dd, J = 1.1, 1.1 Hz, 1H), 7.92 (d, J = 1.5 Hz, 1H), 7.51 (dd, J = 1.6, 1.9 Hz, 1H), 7.45-7.40 (m, 2H), 7.32 (d, J = 2.5 Hz, 1H), 7.22 (t, J = 3.2 Hz, 2H), 7.17 (t, J = 7.9 Hz, 1H), 6.59 (s, 1 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 136.6, 135.0, 128.4, 127.2, 126.2, 124.6, 122.7, 122.2, 121.3, 120.0, 119.4, 117.3, 111.3, 111.2, 102.7, 98.7.



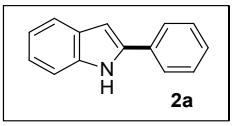
1'-Methyl-1*H*,1'*H*-3,5'-biindole[9] (**1t**)

Pale-brown solid, m.p. 163-164 ºC. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.64 (br s, 1H), 8.03 (d, J = 8.0 Hz, 3H), 7.74-7.72 (m, 3H), 7.41 (d, J = 9.9 Hz, 2H), 7.33 (d, J = 8.8 Hz, 1H), 6.93 (d, J = 8.6 Hz, 1H), 3.88 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 155.0, 141.1, 136.5, 134.2, 132.0, 129.1, 129.0, 127.4, 126.7, 125.7, 124.7, 123.9, 116.8, 112.8, 112.5, 101.6, 56.0.



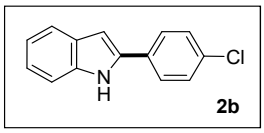
3-(Benzofuran-5-yl)-1*H*-indole[10] (**1u**)

Light yellow oil. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.81 (br s, 1H), 8.16 (d, J = 8.6 Hz, 2H), 8.06 (t, J = 11.0 Hz, 2H), 7.68 (d, J = 7.8 Hz, 1H), 7.44-7.41 (m, 2H), 7.22-7.21 (m, 1H), 7.15 (dd, J = 1.0, 7.0 Hz, 1H), 6.99 (d, J = 2.2 Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 150.4, 136.1, 130.7, 130.3, 129.3, 128.7, 127.6, 124.9, 123.0, 122.7, 121.9, 120.6, 119.8, 112.0, 111.1, 101.5.



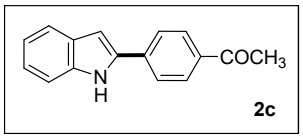
2-Pheny-1*H*-indole[11] (**2a**)

White solid, m.p. 188-190 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.32 (br s, 1H), 7.67 (d, J = 7.0 Hz, 3H), 7.48-7.40 (m, 3H), 7.37-7.33 (t, 1H), 7.26-7.21 (m, 1H), 7.17 (d, J = 8.0 Hz, 1H), 6.86 (s, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 136.9, 132.4, 129.3, 129.0, 127.7, 125.2, 122.4, 120.7, 120.3, 111.0, 100.4.



2-(4-Chlorophenyl)-1*H*-indole[12] (**2b**)

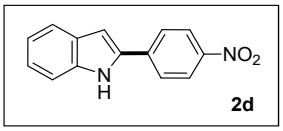
Off white solid, m.p. 201-203 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.30 (br s, 1H), 7.62 (dd, J = 1.0, 1.1 Hz, 1H), 7.55 (dd, J = 2.0, 2.0 Hz, 2H), 7.40-7.37 (m, 3H), 7.22-7.18 (m, 1H), 7.14-7.11 (m, 1H), 6.80 (dd, J = 0.9, 0.8 Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 137.0, 136.7, 133.4, 130.8, 129.2, 129.1, 128.8, 126.3, 122.6, 122.0, 120.7, 120.4, 111.0, 100.4.



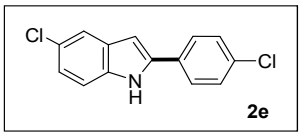
2-(4-Acetylphenyl)-1*H*-indole (**2c**)

White solid, m.p. 172-174 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.11 (br s, 1H), 7.97 (d, J = 6.6 Hz, 1H), 7.64 (dt, J = 5.8, 2.5 Hz, 2H), 7.39 (d, J = 6.7 Hz, 1H), 7.29 (t, J = 12.1 Hz, 1H), 7.25 (t, J = 10.0 Hz, 2H), 7.06 ((dd, J = 1.5, 2.4 Hz, 2H), 3.90 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 158.2, 136.8, 128.8, 128.3, 126.0, 122.4, 121.4, 120.3, 119.9, 118.0, 114.5, 111.6, 55.5.

2-(4-Nitrophenyl)-1*H*-indole[13] (**2d**)

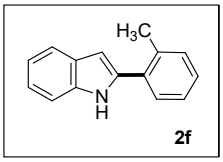


Yellow solid, m.p. > 230 °C. ^{1}H-NMR (400 MHz, DMSO-d6): δ = 11.85 (br s, 1H), 8.31 (d, J = 9.0 Hz, 2H), 8.11 (d, J = 8.9 Hz, 2H), 7.60 (d, J = 7.9 Hz, 1H), 7.45 (d, J = 8.1 Hz, 1H), 7.21-7.16 (m, 2H), 7.07-7.03 (t, 1H). ^{13}C-NMR (100 MHz, DMSO-d6): δ = 146.3, 139.0, 138.4, 135.7, 128.9, 125.9, 124.8, 123.6, 121.3, 120.4, 112.2, 102.9.



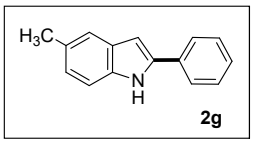
2-(4-Chlorophenyl)-5-chloro-1*H*-indole (**2e**)

Off white solid, m.p. 187-189 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.32 (br s, 1H), 7.61 (dd, J = 11.5, 5.6 Hz, 2H), 7.42 (d, J = 5.5, Hz, 2H), 7.31 (d, J = 2.1, Hz, 2H), 7.23 (d, J = 1.5, Hz, 1H), 7.15 (d, J = 5.7, Hz, 1H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 138.2, 135.3, 134.0, 130.5, 130.3, 129 .4, 126.5, 126.1, 123.0, 122.4, 120.2, 120.1, 112.0, 100.1.



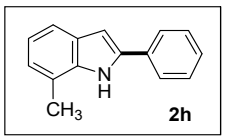
2-(2-Methylphenyl)-1*H*-indole[12] (**2f**)

White solid, m.p. 92-93 °C. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.24 (1H, br), 7.73 (t, J = 10.6 Hz, 2H), 7.54 (d, J = 9.7 Hz, 1H), 7.50-7.46 (t, 2H), 7.38-7.35 (t, 1H), 7.11 (t, J = 9.1 Hz, 1H), 7.09-7.04 (t, 1H), 6.88 (s, 1H), 2.58 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 136.6, 132.7, 130.3, 129.1, 129.0, 128.8, 128.6, 128.4, 127.8, 125.4, 123.1, 120.6, 118.6, 16.9.



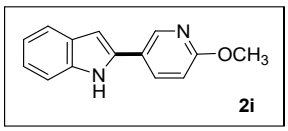
5-Methyl-2-phenyl-1*H*-indole[11] (**2g**)

Colorless solid, m.p. 214-215 °C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ = 8.25 (br s, 1H), 7.65 (dd, J = 1.4, 1.1 Hz, 2H), 7.46-7.41 (m, 3H), 7.33-7.28 (m, 2H), 7.02 (dd, J = 1.6, 1.6 Hz, 1H), 6.75 (dd, J = 2.1, 0.9 Hz, 1H), 2.45 (s, 3H). ^{13}C-NMR (100 MHz, $CDCl_3$): δ = 136.9, 136.7, 133.4, 130.8, 129.2, 129.1, 128.9, 126.3, 122.6, 122.0, 120.7, 120.4, 111.0, 100.4, 30.5.



7-Methyl-2-phenyl-1*H*-indole[11] (**2h**)

Colorless solid, m.p. 116-117 ºC. ^{1}H-NMR (500 MHz, $CDCl_3$): δ = 8.24 (br s, 1H), 7.75 (d, J = 6.6 Hz, 2H), 7.58 (d, J = 6.6 Hz, 1H), 7.51 (t, J = 12.8 Hz, 2H), 7.39 (t, J = 12.3 Hz, 1H), 7.15 (t, J = 12.4 Hz, 1H), 7.09 (d, J = 5.9 Hz, 1H), 6.9 (d, J = 1.7 Hz, 1H), 2.60 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): δ = 137.8, 136.6, 132.7, 129.2, 129.0, 127.8, 125.4, 123.1, 120.7, 120.3, 118.6, 100.8, 16.9.



2-(6-Methoxylpyridin-3-yl)-1*H*-indole (**2i**)

White solid, m.p. 122-124 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.69 (br s, 1H), 8.52 (s, 1H), 7.90-7.87 (m, 2H), 7.41 (d, J = 10.1 Hz, 1H), 7.29-7.20 (m, 3H), 6.88 (t, J = 13.2 Hz, 1H), 4.04 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 162.8, 145.0, 138.3, 136.8, 125.8, 125.0, 122.7, 121.7, 120.5, 119.4, 114.6, 111.7, 110.9, 53.7.

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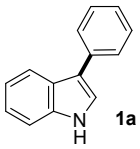
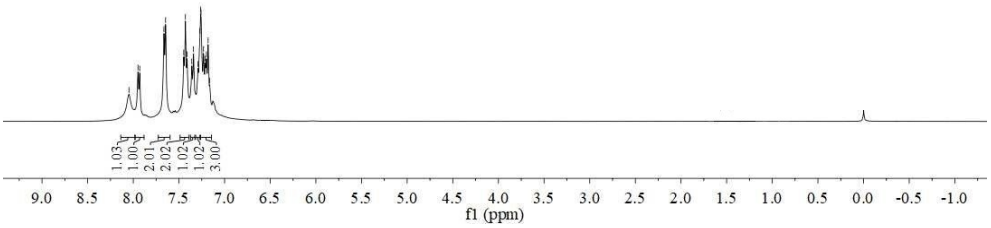
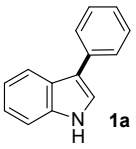
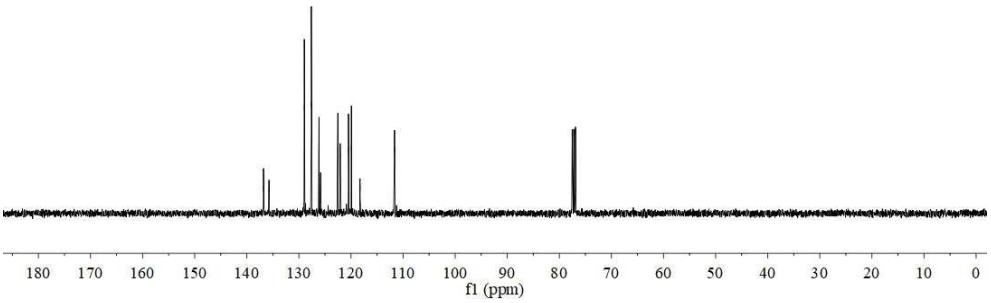
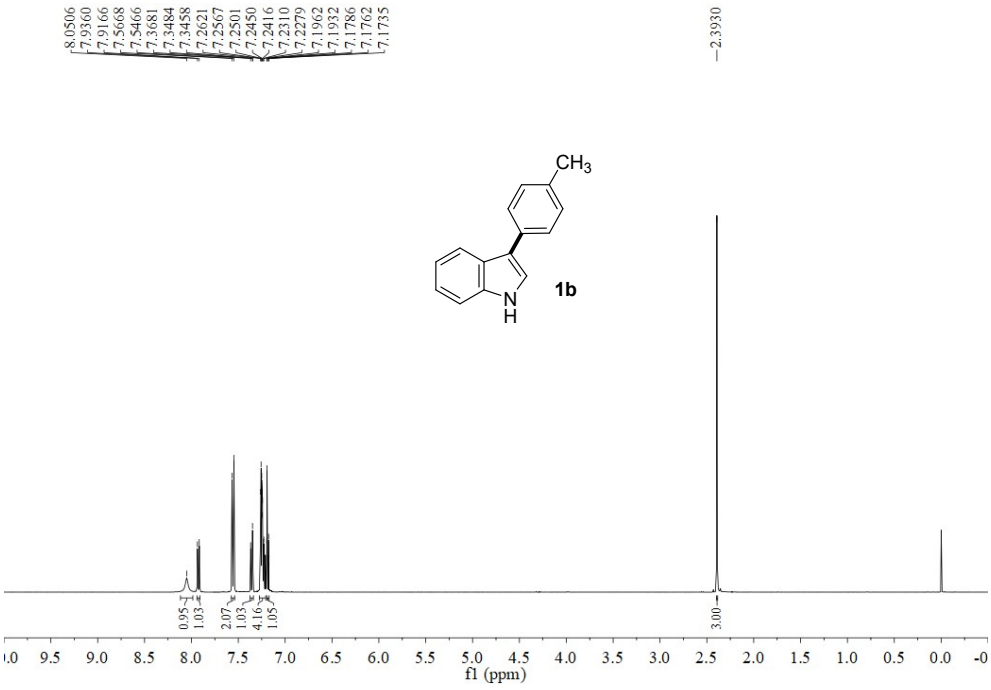
3 NMR Spectra







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7.1932
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7.1735
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H
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1.03
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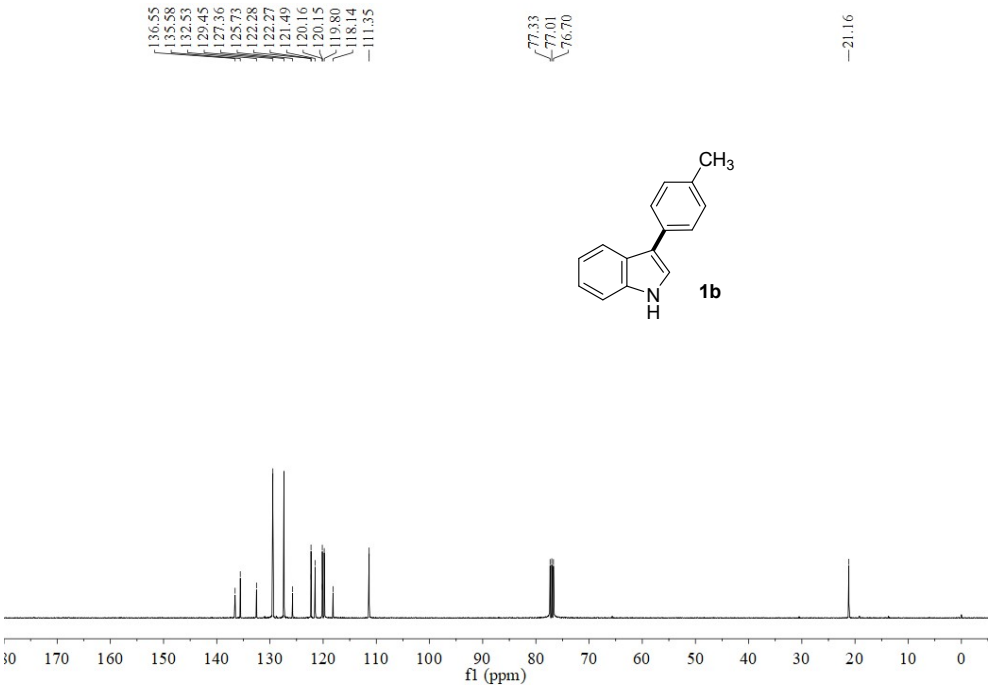
3.00
fl (ppm)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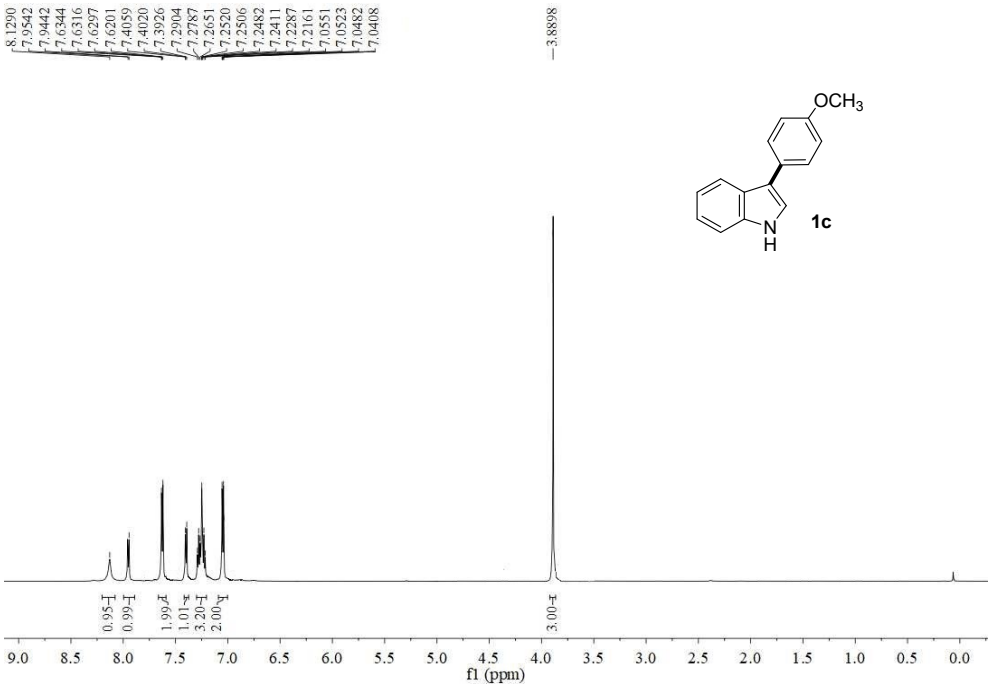
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7.0408
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N
H
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1.99
1.01
3.20
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3.00
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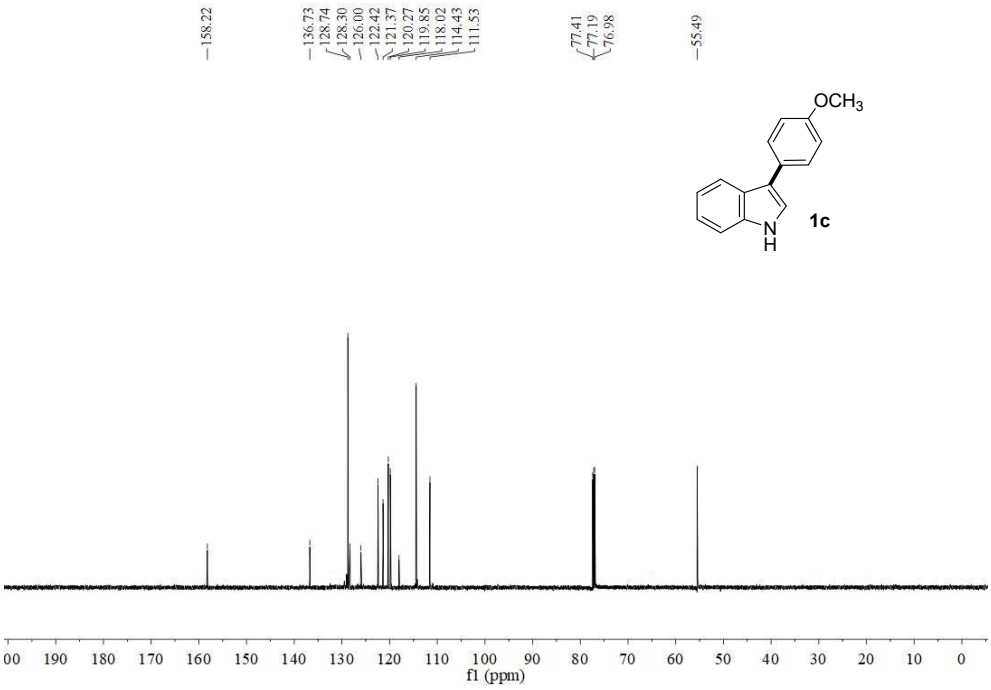
f1 (ppm)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H
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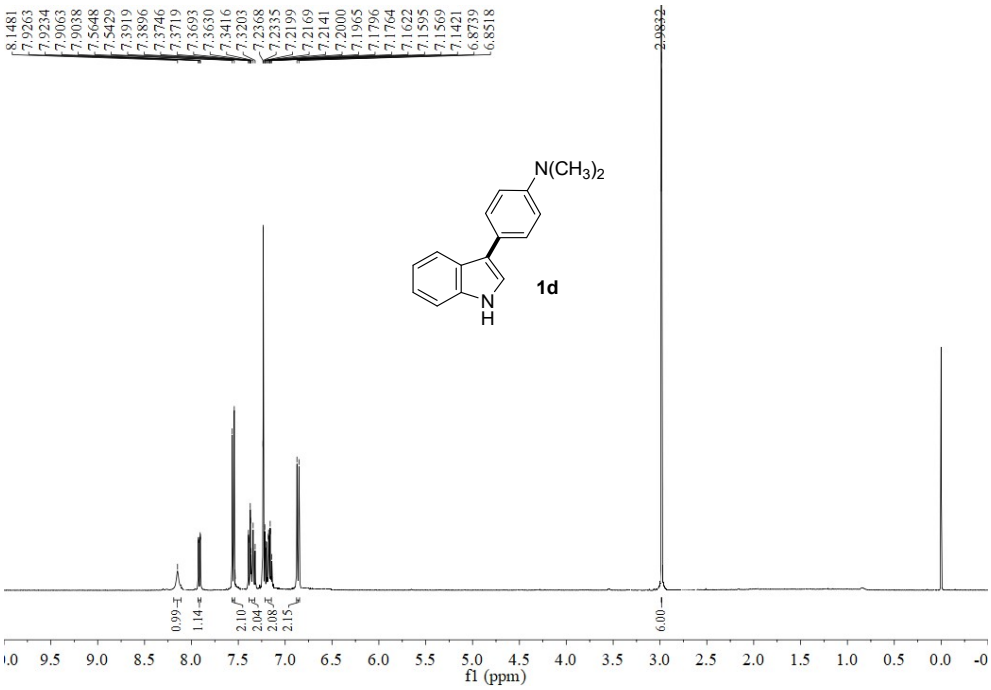
f1 (ppm)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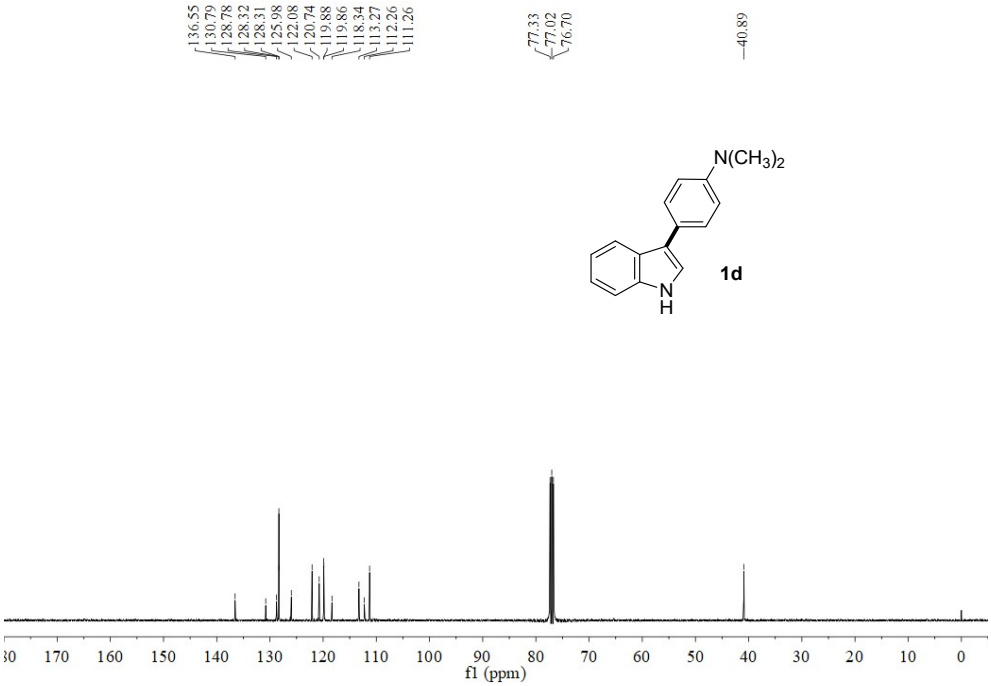
2.15
6.00
fl (ppm)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77.02
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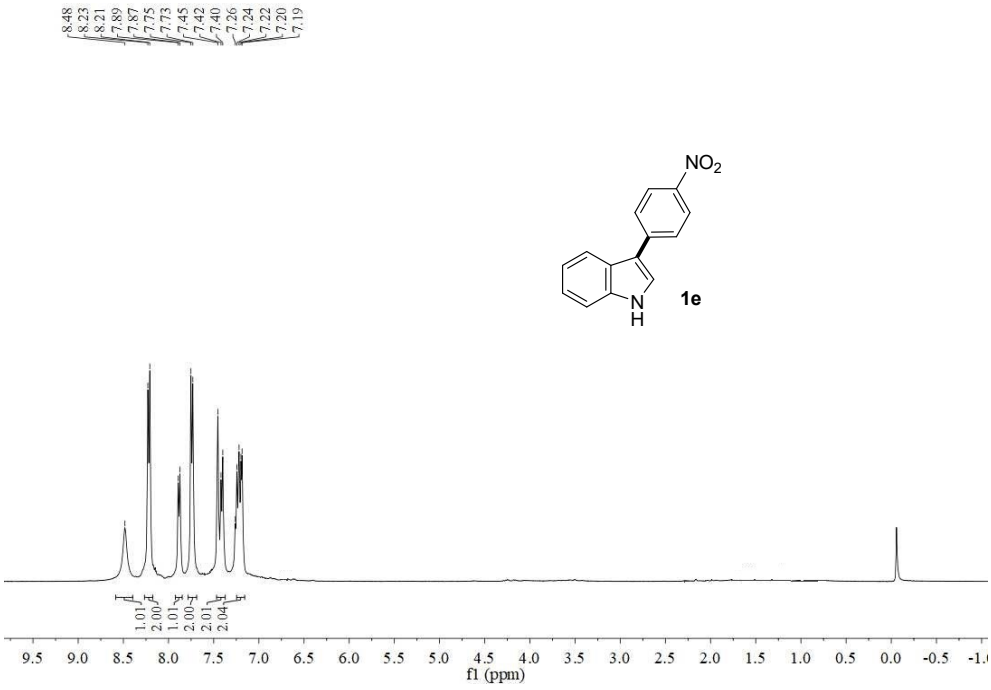
N(CH3)2
1d
N
H
fl (ppm)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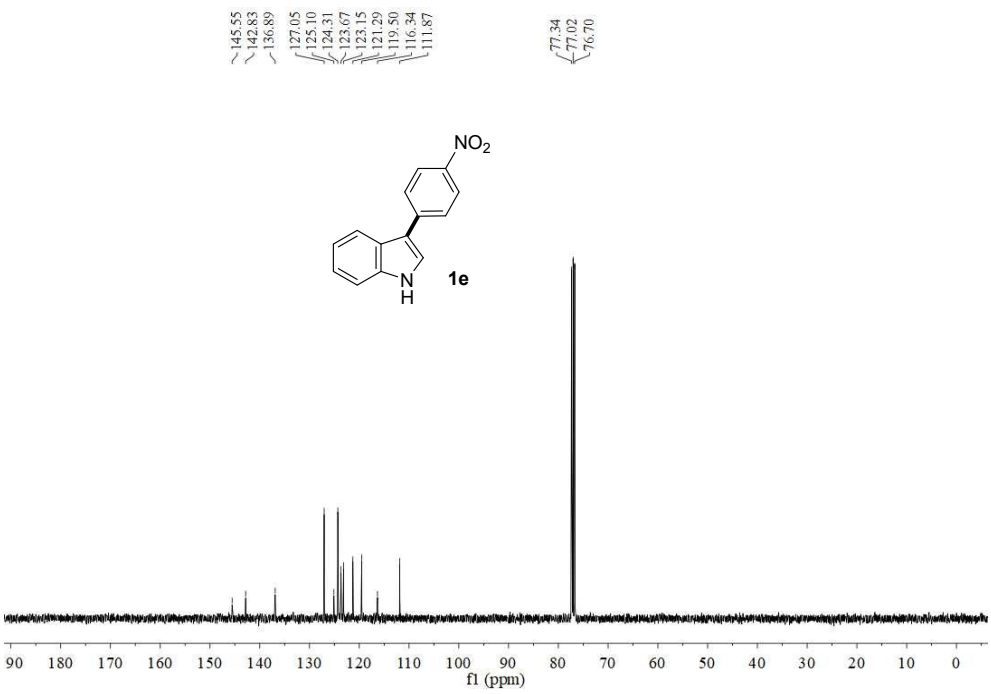
1.01
2.00
2.01
2.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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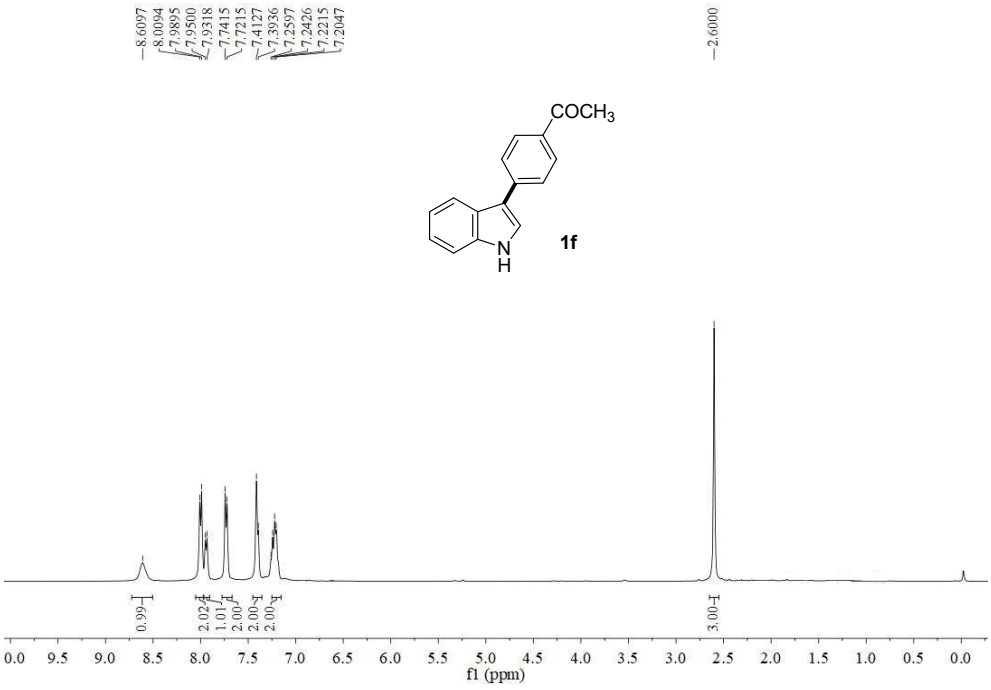
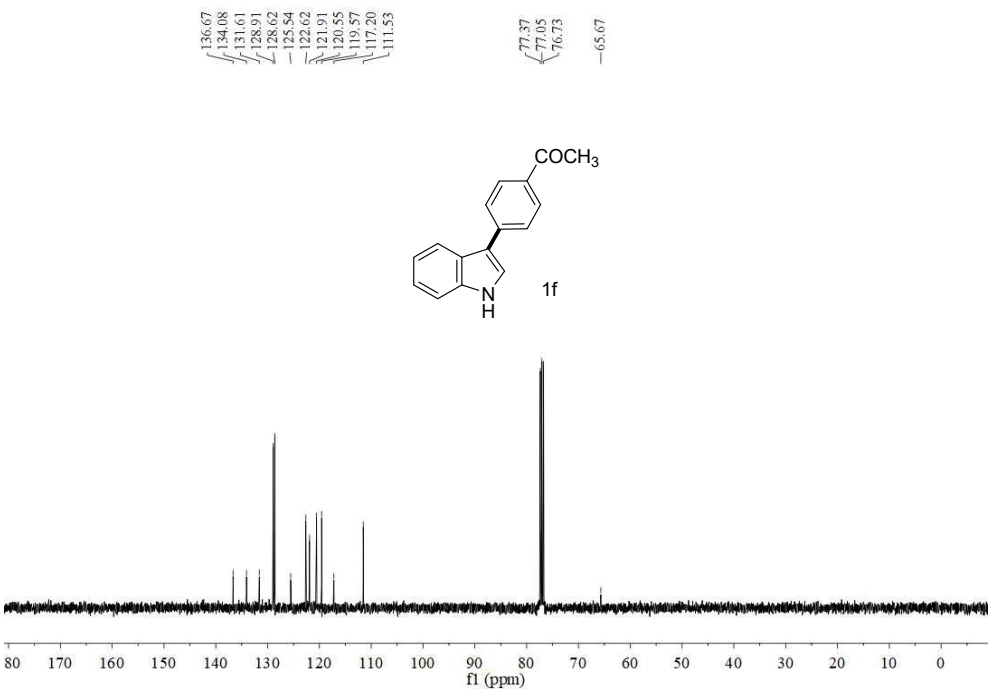
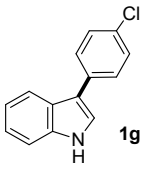
127.05
125.10
124.31
123.67
123.15
121.29
119.50
116.34
111.87
77.34
77.02
76.70
NO2
N
H
1e
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

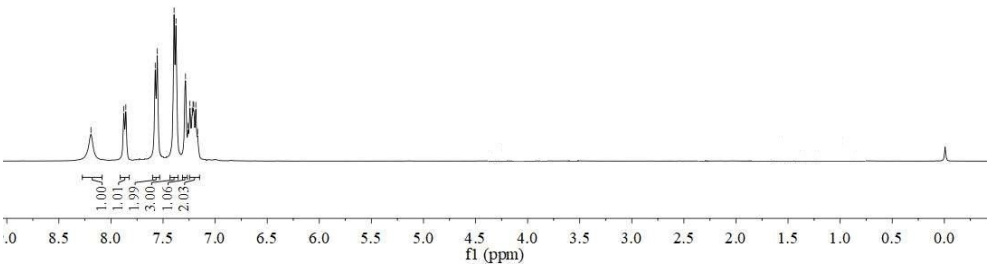

8.6097
8.0094
7.9895
7.9500
7.9318
7.7415
7.7215
7.4127
7.3936
7.2597
7.2426
7.2215
7.2047
2.6000
COCH3
N
H
1f
0.99
2.02
1.01
2.00
2.00
2.00
3.00
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


136.67
134.08
131.61
128.91
128.62
125.54
122.62
121.91
120.55
119.57
117.20
111.53
77.37
77.05
76.73
65.67
COCH3
N
H
1f
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.1939
7.8819
7.8621
7.5828
7.5631
7.3973
7.3821
7.2817
7.2619
7.2422
7.2231
7.2194
7.2156
7.1988
7.1786

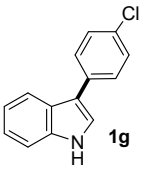

Cl
N
H

1g

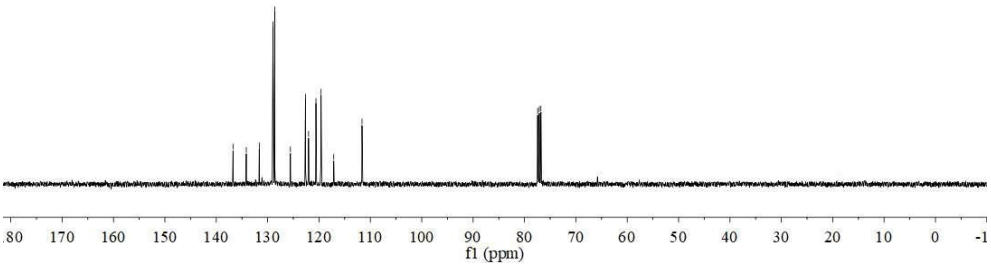

1.00
1.01
1.99
3.00
1.06
2.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

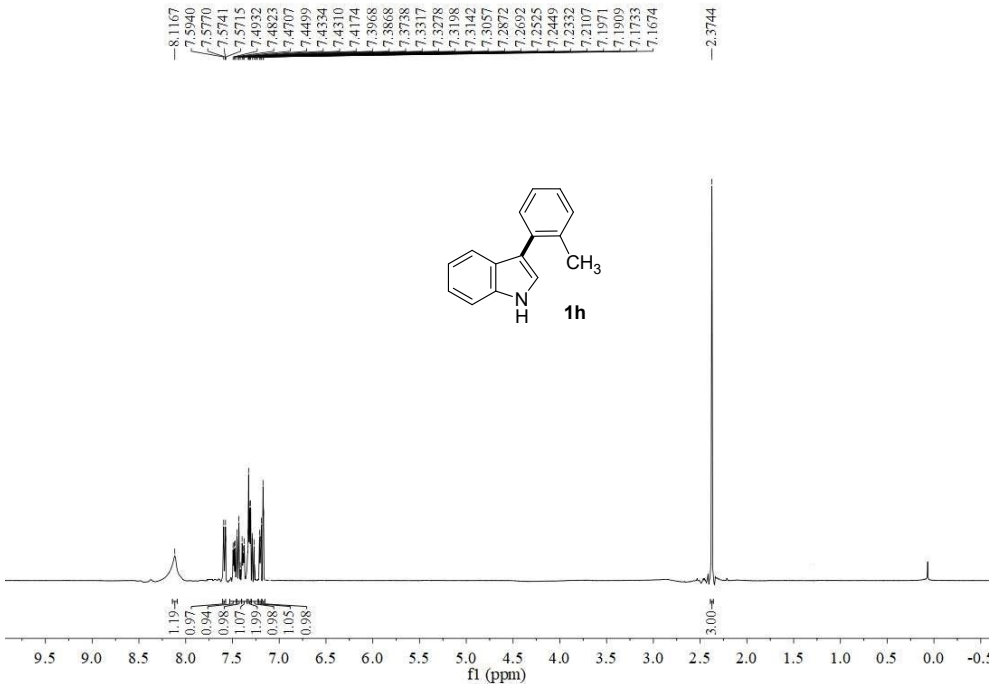

136.73
134.15
131.63
128.95
128.63
125.57
122.63
122.04
120.59
119.60
117.13
111.63

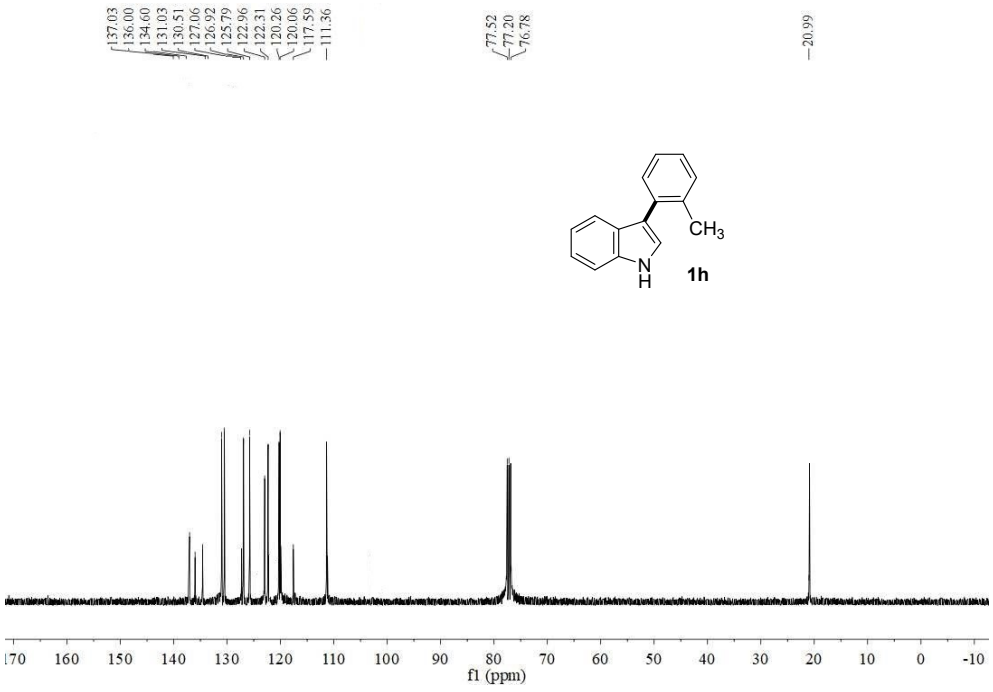
77.44
77.12
76.81


Cl
N
H

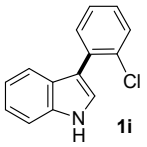
1g


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

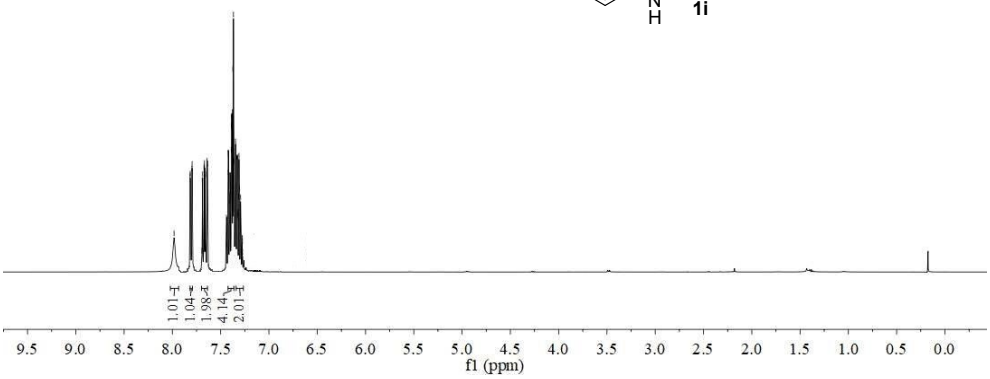

8.1167
7.5940
7.5770
7.5741
7.5715
7.4932
7.4823
7.4707
7.4499
7.4334
7.4310
7.4174
7.3968
7.3868
7.3738
7.3317
7.3278
7.3198
7.3142
7.3057
7.2872
7.2692
7.2525
7.2449
7.2332
7.2107
7.1971
7.1909
7.1733
7.1674
2.3744
CH3
N
H
1h
1.19
0.97
0.94
0.98
1.07
1.99
0.98
1.05
0.98
3.00
f1 (ppm)

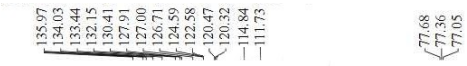

137.03
136.00
134.60
131.03
130.51
127.06
126.92
125.79
122.96
122.31
120.26
120.06
117.59
111.36
77.52
77.20
76.78
20.99
CH3
N
H
1h
f1 (ppm)

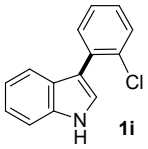

7.9825
7.8169
7.8150
7.8027
7.7973
7.7960
7.6936
7.6909
7.6865
7.6722
7.6718
7.6675
7.6603
7.6596
7.6570
7.6404
7.6372
7.6371
7.4405
7.4384
7.4210
7.4061
7.4043
7.3907
7.3883
7.3865
7.3850
7.3795
7.3740
7.3673
7.3602
7.3461
7.3416
7.3281
7.3258
7.3220
7.3136
7.3101
7.3061
7.2936
7.2797
7.2795
7.2776
7.2747
7.2604
7.2368


Cl
N
H

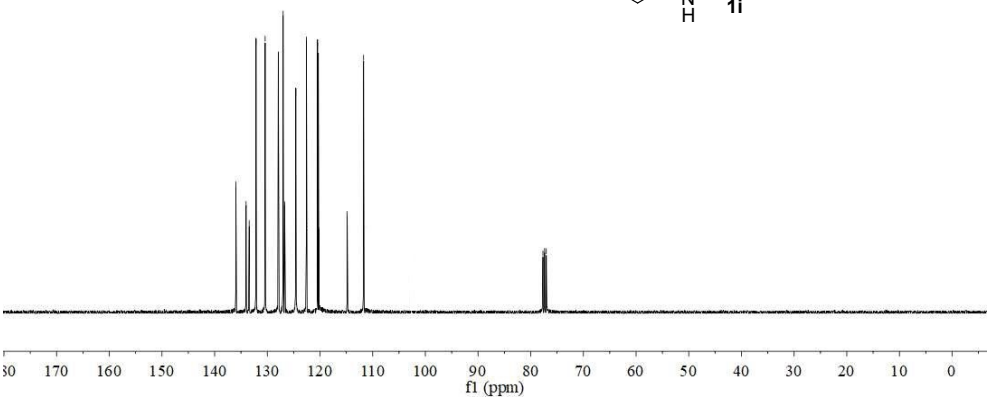
1i


1.01
1.04
1.98
4.14
2.01
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
f1 (ppm)

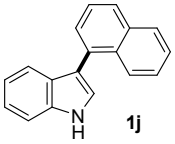

135.97
134.03
133.44
132.15
130.41
127.91
127.00
126.71
124.59
122.58
120.47
120.32
114.84
111.73
77.68
77.36
77.05


Cl
N
H

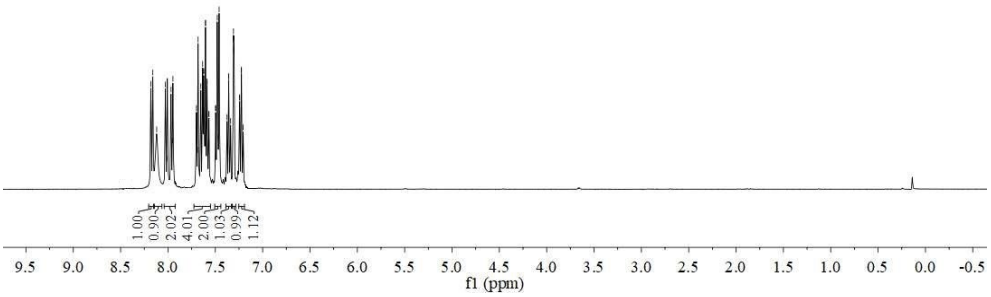
1i


170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.1813
8.1692
8.1600
8.1192
8.0269
8.0137
7.9676
7.9530
7.9476
7.6992
7.6886
7.6818
7.6527
7.6339
7.6229
7.6228
7.6035
7.6034
7.5871
7.5864
7.5672
7.5672
7.4967
7.4966
7.4796
7.4702
7.4598
7.3762
7.3583
7.3578
7.3383
7.3080
7.3059
7.3027
7.2432
7.2431
7.2242
7.2237

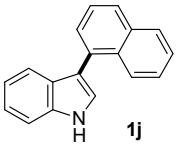

N
H

1j

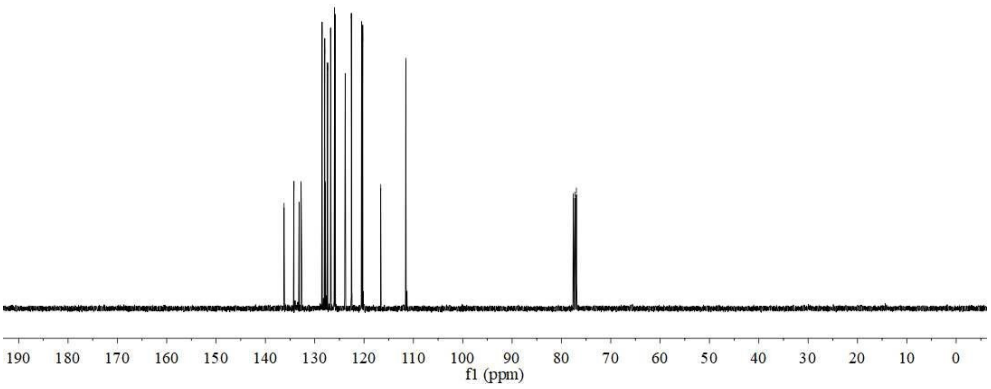

1.00
0.90
2.02
4.01
2.00
1.03
0.99
1.12
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

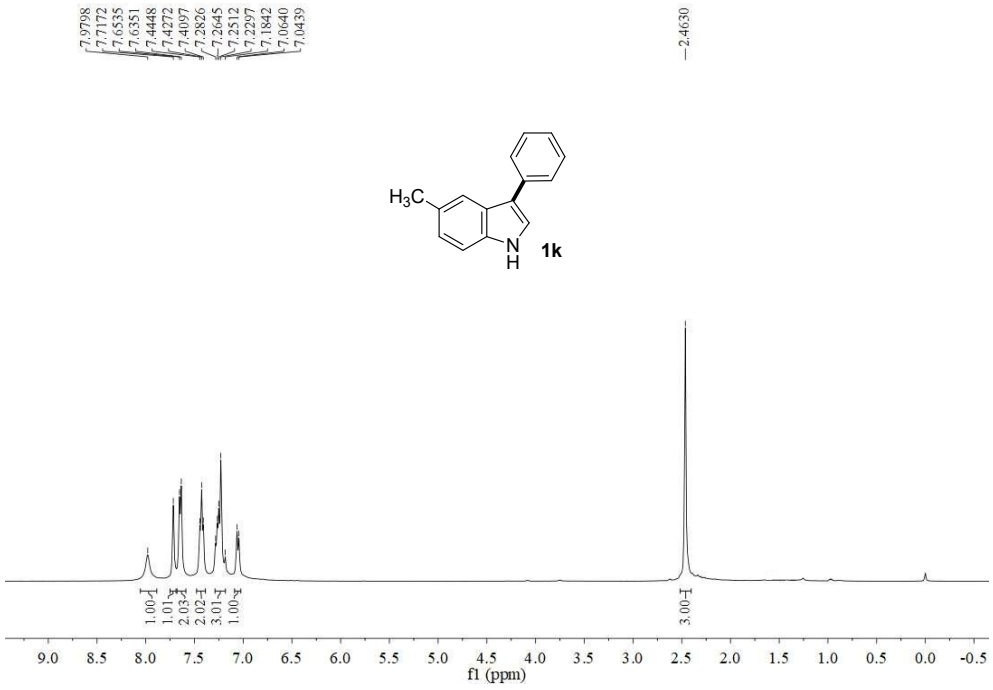

136.22
134.21
133.17
132.76
128.51
127.96
127.85
127.39
126.78
125.98
125.86
123.82
122.58
120.51
120.26
116.62
111.54

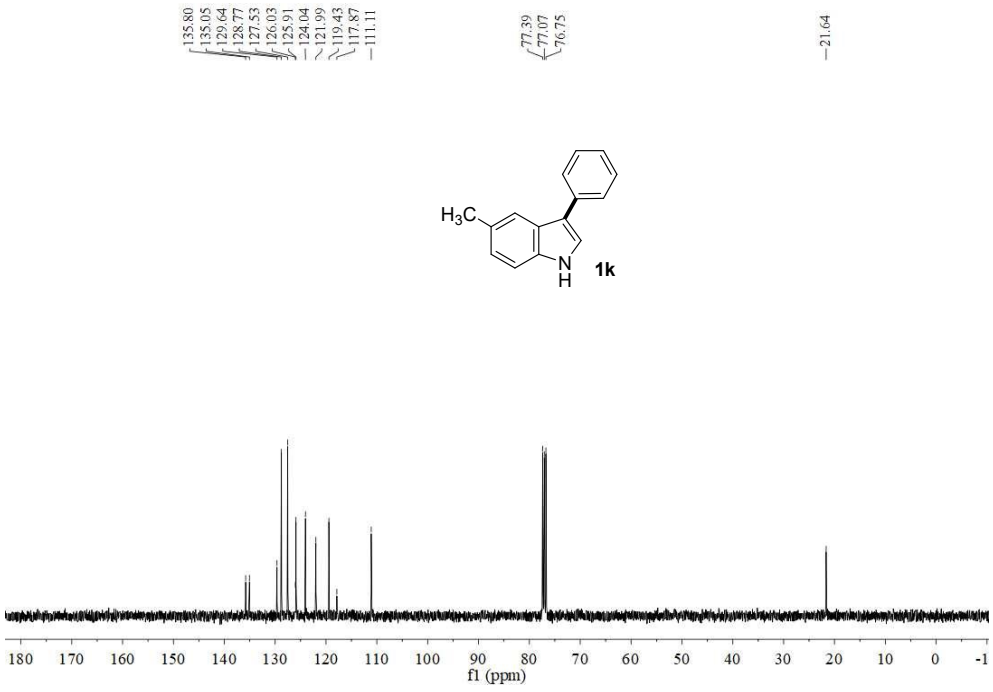
77.60
77.29
76.97

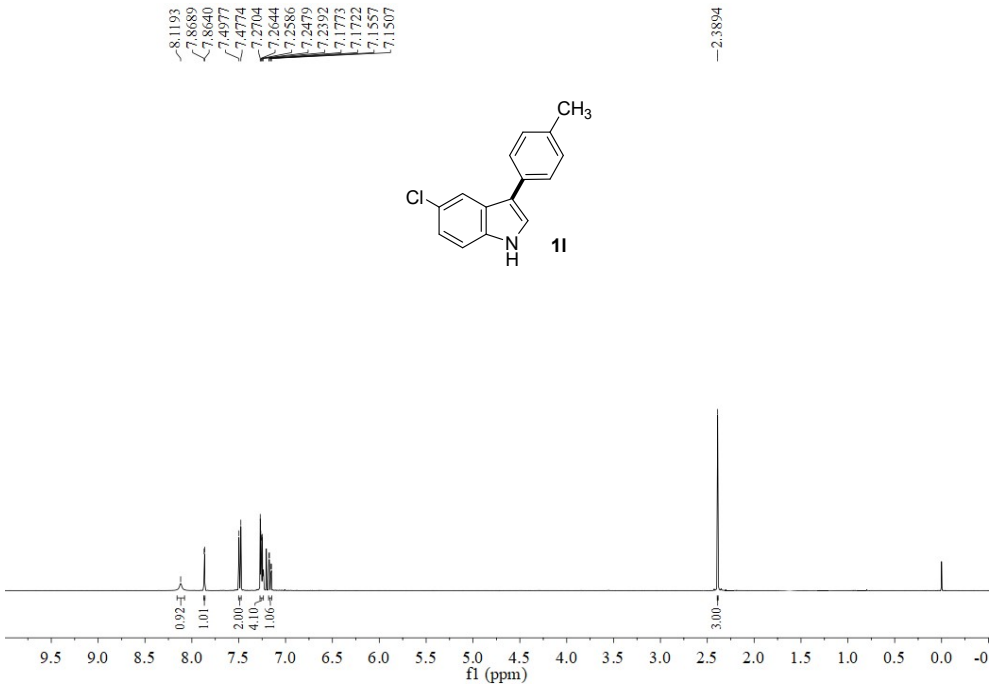

N
H

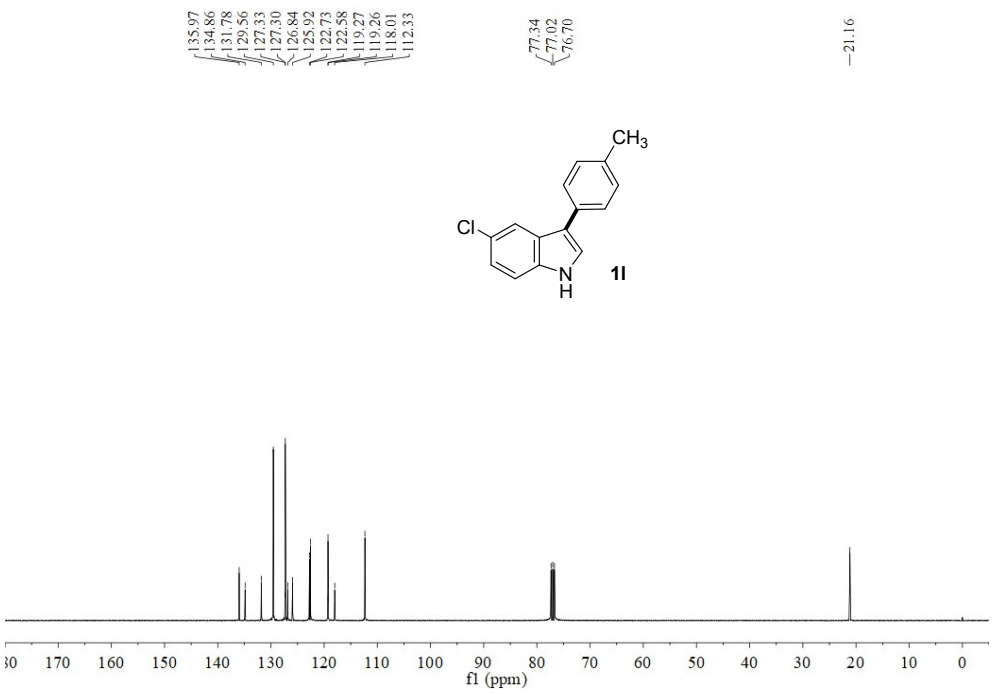
1j

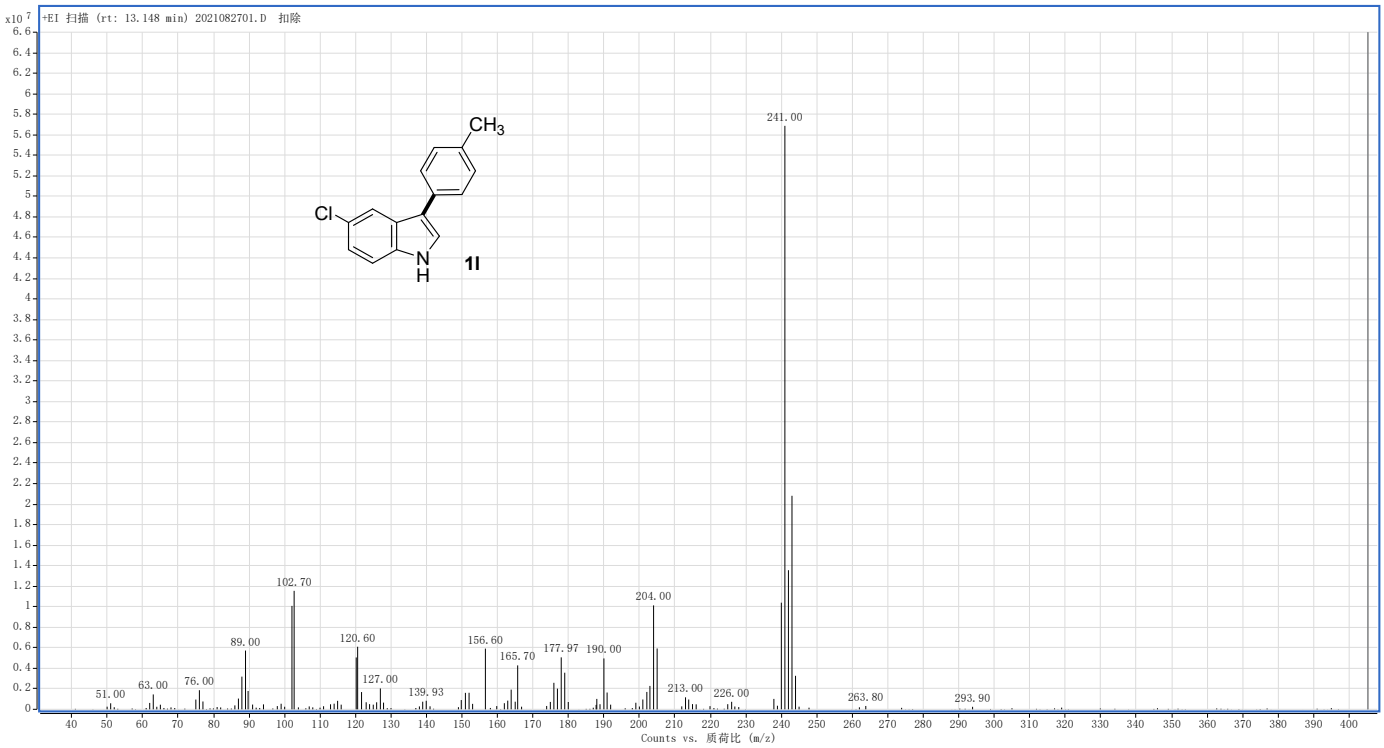

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

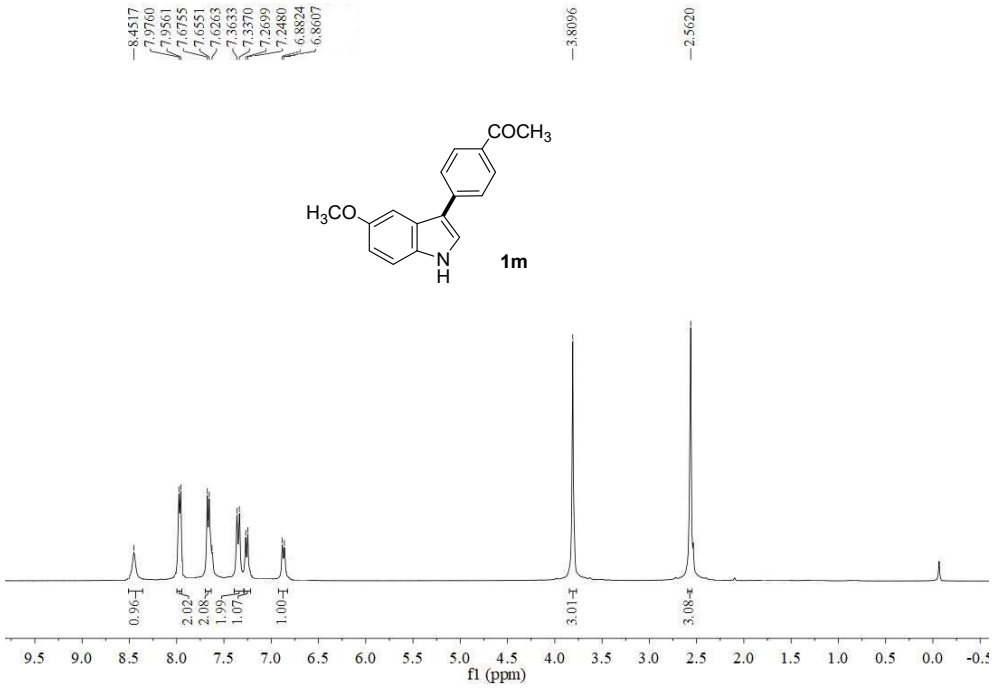

7.9798
7.7172
7.6535
7.6351
7.4448
7.4272
7.4097
7.2826
7.2645
7.2512
7.2297
7.1842
7.0640
7.0439
2.4630
H3C
N
H
1k
1.00
1.01
2.03
2.02
3.01
1.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

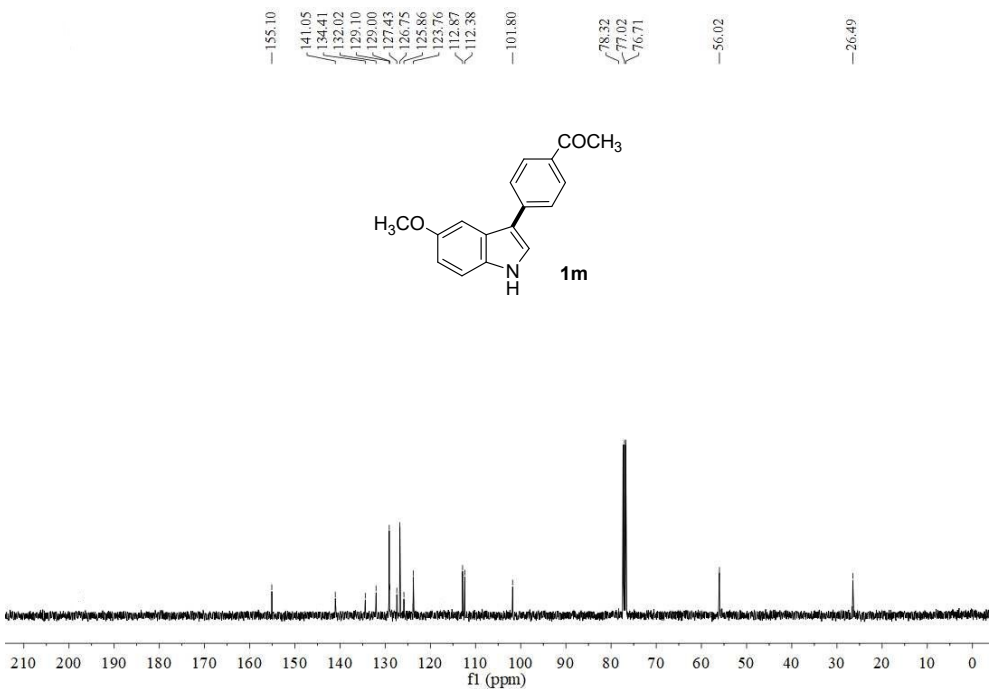

135.80
135.05
129.64
128.77
127.53
126.03
125.91
124.04
121.99
119.43
117.87
111.11
77.39
77.07
76.75
21.64
H3C
N
H
1k
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

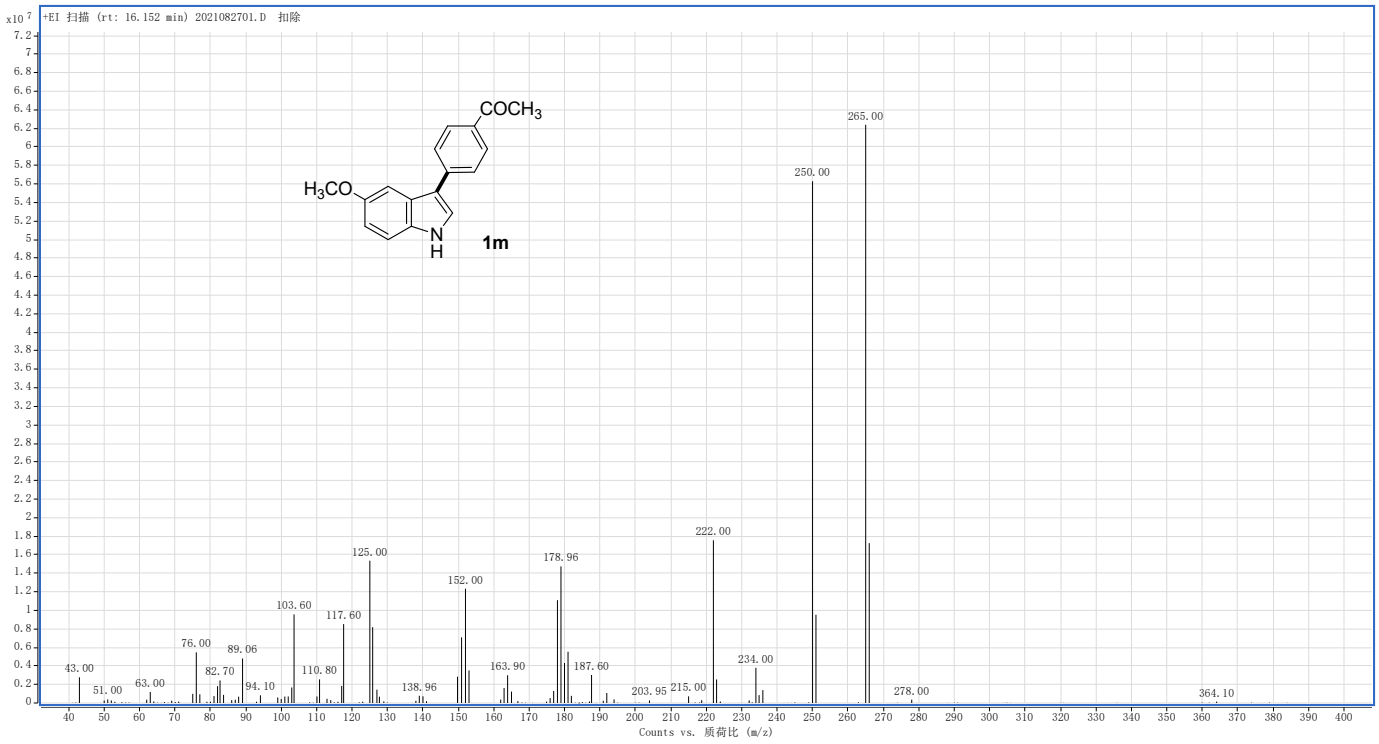

8.1193
7.8689
7.8640
7.4977
7.4774
7.2704
7.2644
7.2586
7.2479
7.2392
7.1773
7.1722
7.1557
7.1507
2.3894
CH3
Cl
N
H
1l
0.92
1.01
2.00
4.10
1.06
3.00
f1 (ppm)

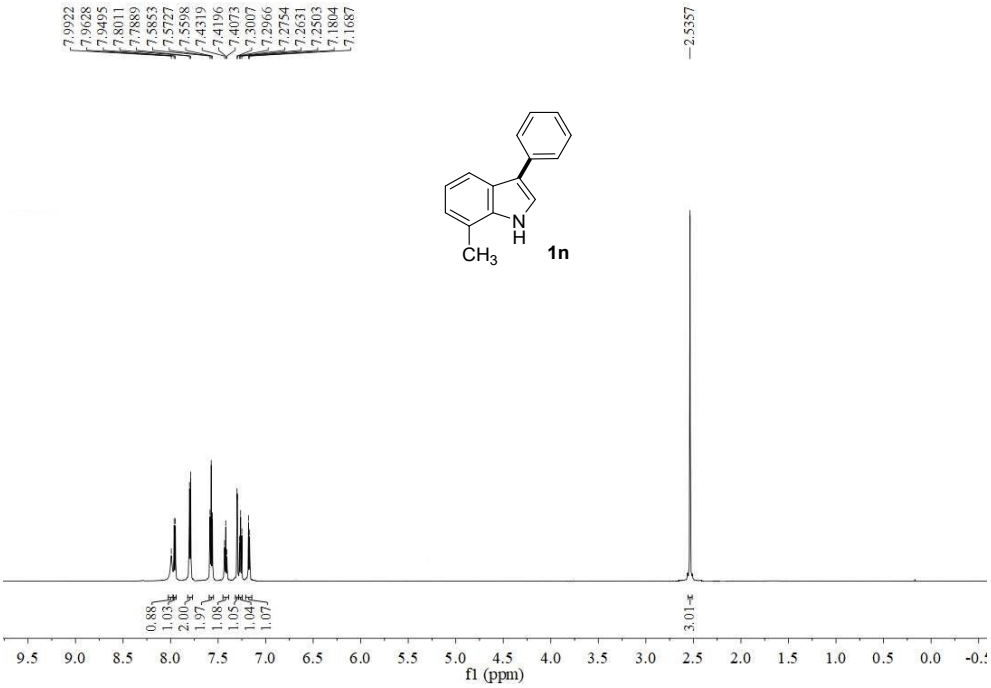

135.97
134.86
131.78
129.56
127.33
127.30
126.84
125.92
122.73
122.58
119.27
119.26
118.01
112.33
77.34
77.02
76.70
21.16
CH3
Cl
N
H
1l
f1 (ppm)

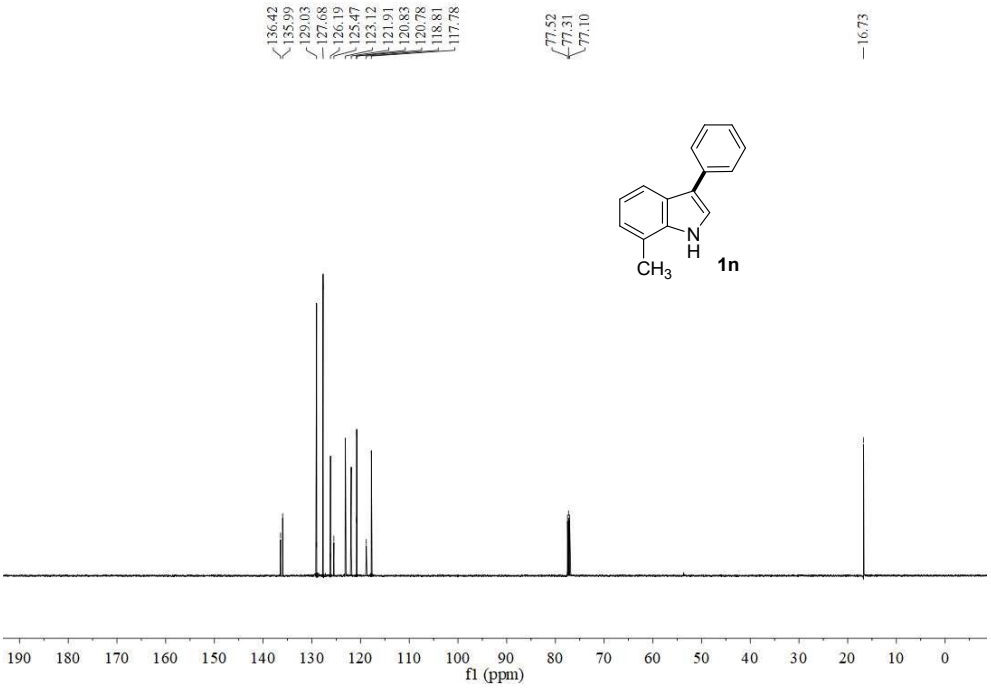

+EI 扫描 (rt: 13.148 min) 2021082701.D 扣除
x10 7
CH3
Cl
N
H
1l
241.00
102.70
204.00
89.00
120.60
156.60
165.70
177.97
190.00
63.00
76.00
127.00
139.93
213.00
226.00
263.80
293.90
51.00
Counts vs. 质荷比 (m/z)


8.4517
7.9760
7.9561
7.6755
7.6551
7.6263
7.3633
7.3370
7.2699
7.2480
6.8824
6.8607
3.8096
2.5620
COCH3
H3CO
N
H
1m
0.96
2.02
2.08
1.99
1.07
1.00
3.01
3.08
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
f1 (ppm)

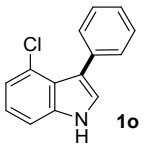

155.10
141.05
134.41
132.02
129.10
129.00
127.43
126.75
125.86
123.76
112.87
112.38
101.80
78.32
77.02
76.71
56.02
26.49
COCH3
H3CO
N
H
1m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


+EI 扫描 (rt: 16.152 min) 2021082701.D 扣除
x10 7
COCH3
H3CO
N
H
1m
265.00
250.00
222.00
125.00
178.96
152.00
103.60
117.60
76.00
89.06
82.70
110.80
43.00
51.00
63.00
94.10
138.96
163.90
187.60
203.95
215.00
234.00
278.00
364.10
Counts vs. 质荷比 (m/z)

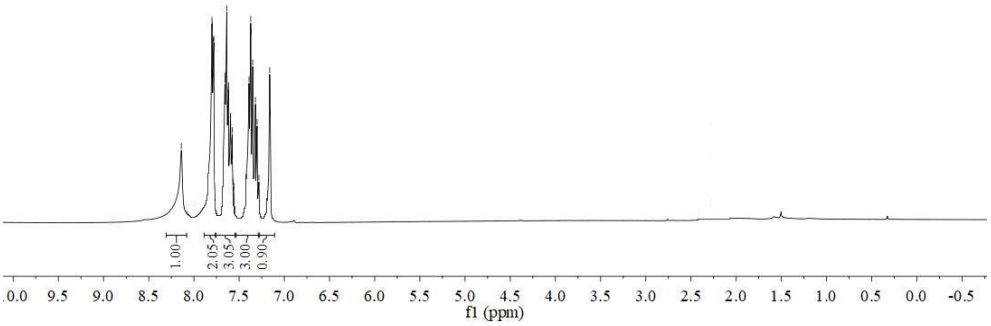

7.9922
7.9628
7.9495
7.8011
7.7889
7.5853
7.5727
7.5598
7.4319
7.4196
7.4073
7.3007
7.2966
7.2754
7.2631
7.2503
7.1804
7.1687
2.5357
1n
0.88
1.03
2.00
1.97
1.08
1.05
1.04
1.07
3.01
f1 (ppm)


136.42
135.99
129.03
127.68
126.19
125.47
123.12
121.91
120.83
120.78
118.81
117.78
77.52
77.31
77.10
16.73
1n
f1 (ppm)


8.1393
7.8004
7.7964
7.7904
7.7831
7.7795
7.6570
7.6536
7.6367
7.6207
7.6178
7.5973
7.5941
7.5823
7.5762
7.5688
7.5579
7.3903
7.3875
7.3717
7.3694
7.3500
7.3196
7.3010
7.2809
7.2012
7.1613
7.1555

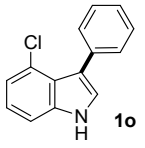

Cl
N
H

1o


1.00
2.05
3.05
3.00
0.90
f1 (ppm)

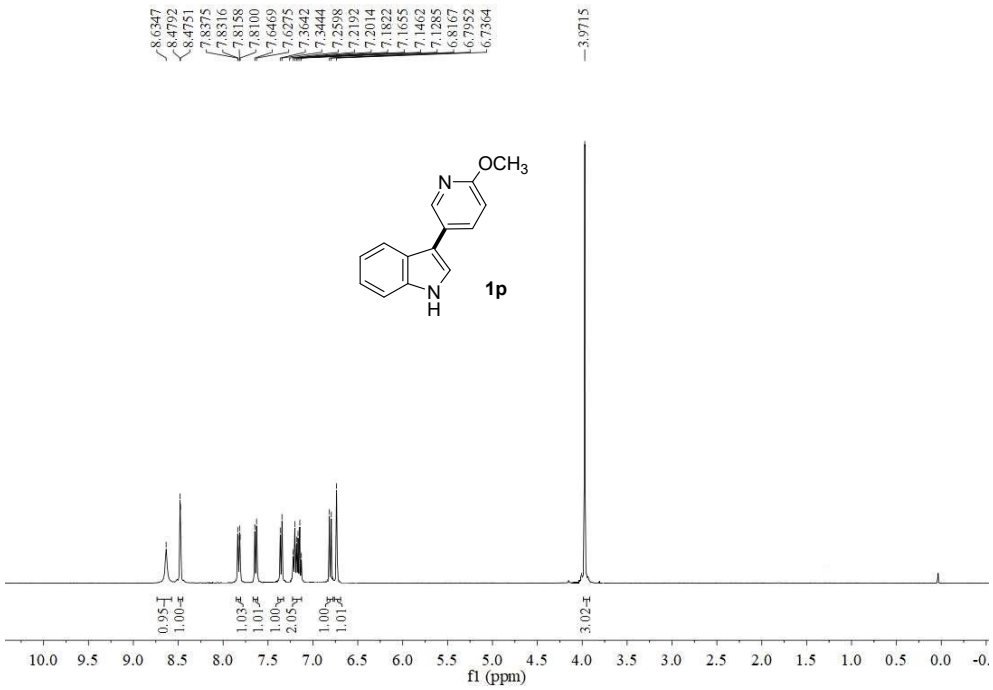

137.83
135.57
131.38
127.92
126.98
126.62
125.07
123.42
123.12
121.66
118.95
110.73

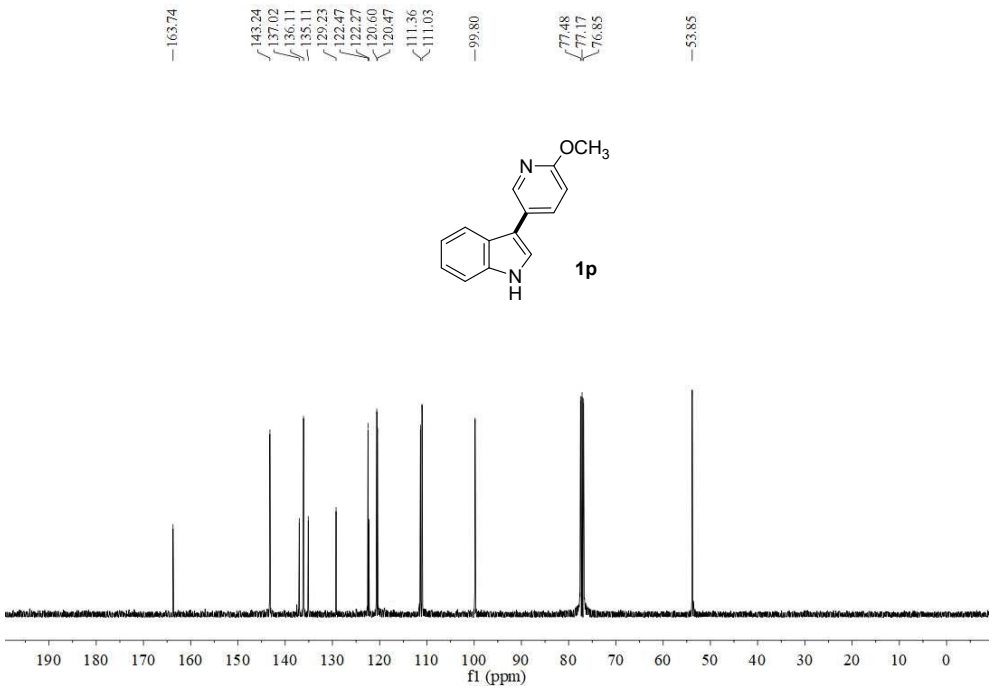

77.93
77.61
77.29


Cl
N
H

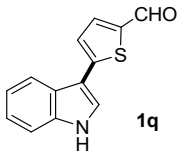
1o

f1 (ppm)

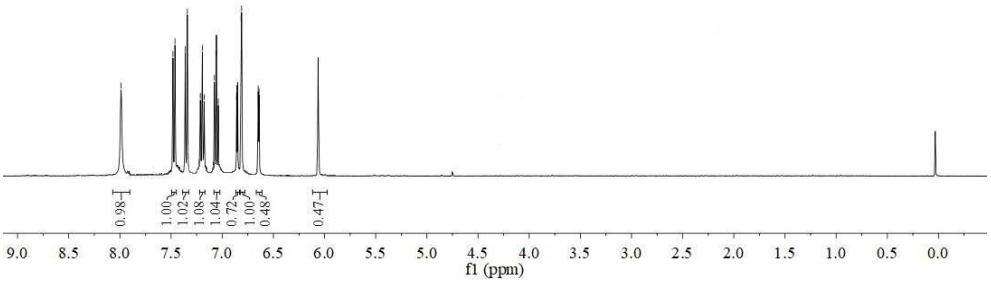

8.6347
8.4792
8.4751
7.8375
7.8316
7.8158
7.8100
7.6469
7.6275
7.3642
7.3444
7.2598
7.2192
7.2014
7.1822
7.1655
7.1462
7.1285
6.8167
6.7952
6.7364
3.9715
OCH3
N
N
H
1p
0.95
1.00
1.03
1.01
1.00
2.05
1.00
1.01
3.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


163.74
143.24
137.02
136.11
135.11
129.23
122.47
122.27
120.60
120.47
111.36
111.03
99.80
77.48
77.17
76.85
53.85
OCH3
N
N
H
1p
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.9885
7.4814
7.4616
7.3628
7.3611
7.3406
7.2600
7.2142
7.1945
7.1762
7.0763
7.0585
7.0387
6.8604
6.8586
6.8513
6.8160
6.8105
6.6491
6.6398
6.6376
6.0611

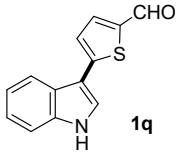

CHO
S
N
H

1q

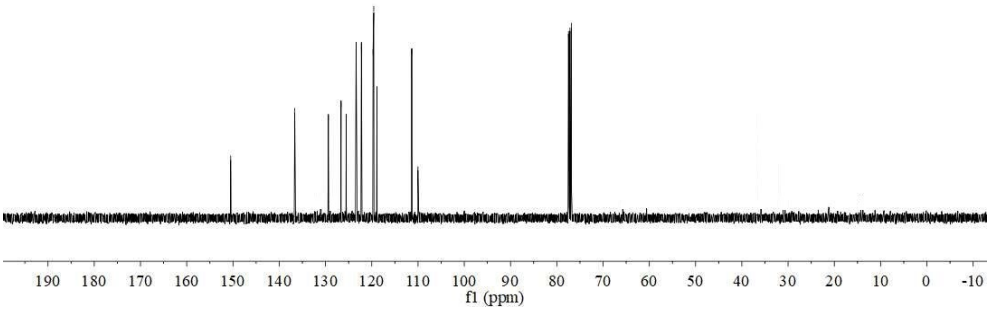

0.98
1.00
1.02
1.08
1.04
0.72
1.00
0.48
0.47
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


150.50
136.65
129.38
126.65
125.51
123.39
122.24
119.72
119.57
118.87
111.36
110.02

77.47
77.16
76.84


CHO
S
N
H

1q


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.3312
7.8706
7.6840
7.6798
7.6759
7.6679
7.6491
7.6379
7.6201
7.6162
7.6124
7.5883
7.5860
7.5667
7.5649
7.4261
7.4063
7.2147
7.2116
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7.1703
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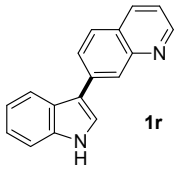
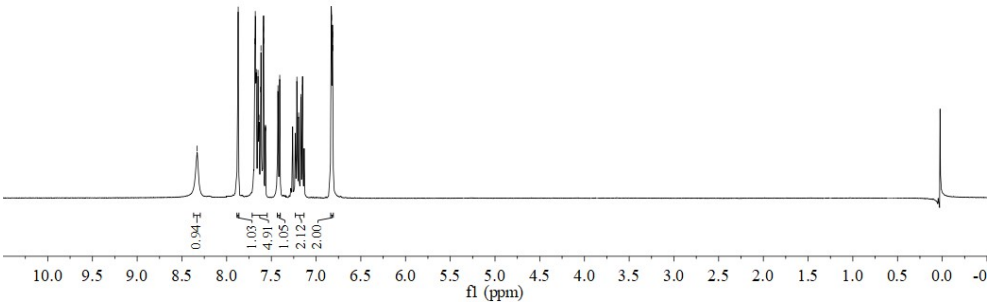
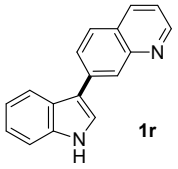
7.1490
6.8323
6.8266
6.8183
6.8155
6.8132


N
N
H

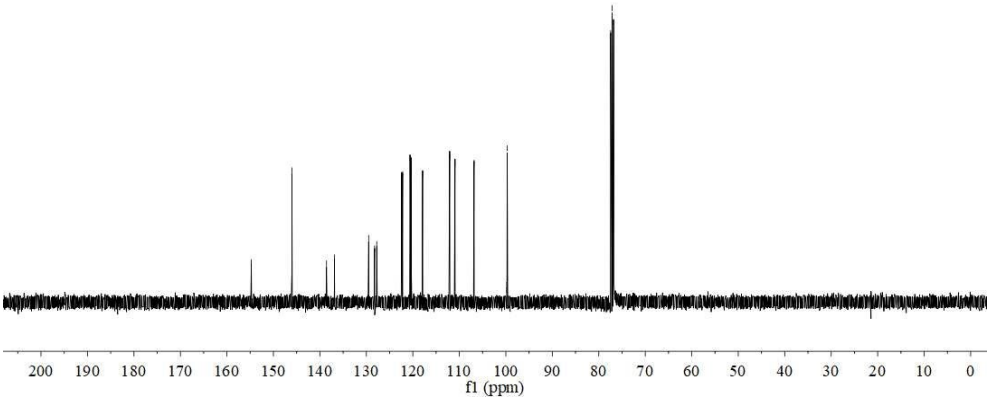
1r


0.94
1.03
4.91
1.05
2.12
2.00
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 -0
f1 (ppm)

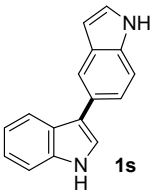

154.75
146.01
138.57
136.87
129.50
128.22
127.77
122.41
122.19
120.61
120.35
117.90
112.07
110.93
106.83
99.70
77.45
77.13
76.81


N
N
H

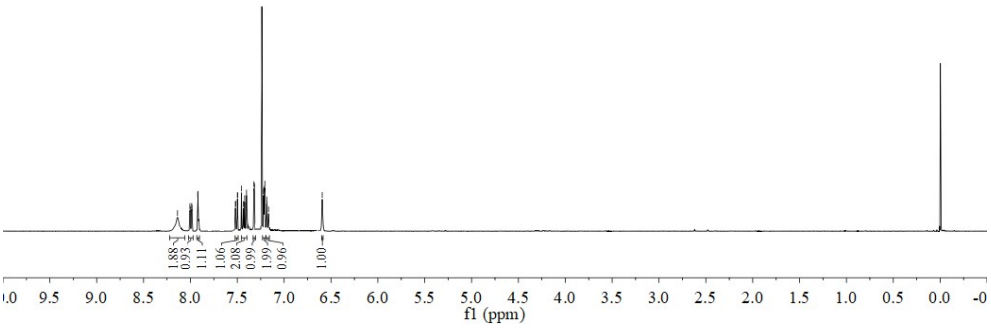
1r

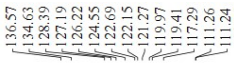

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.1379
8.0053
8.0025
7.9863
7.9835
7.9221
7.9184
7.5204
7.5163
7.4991
7.4950
7.4559
7.4349
7.4216
7.4016
7.3227
7.3164
7.2203
7.2133
7.2112
7.2054
7.1858
7.1832
7.1660
6.5938

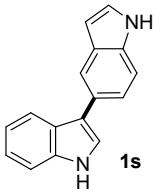

NH
N
H

1s

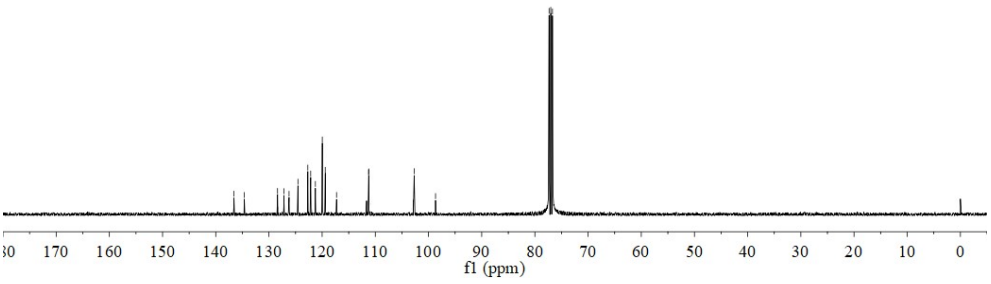

1.88
0.93
1.11
1.06
2.08
0.99
1.99
0.96
1.00
f1 (ppm)

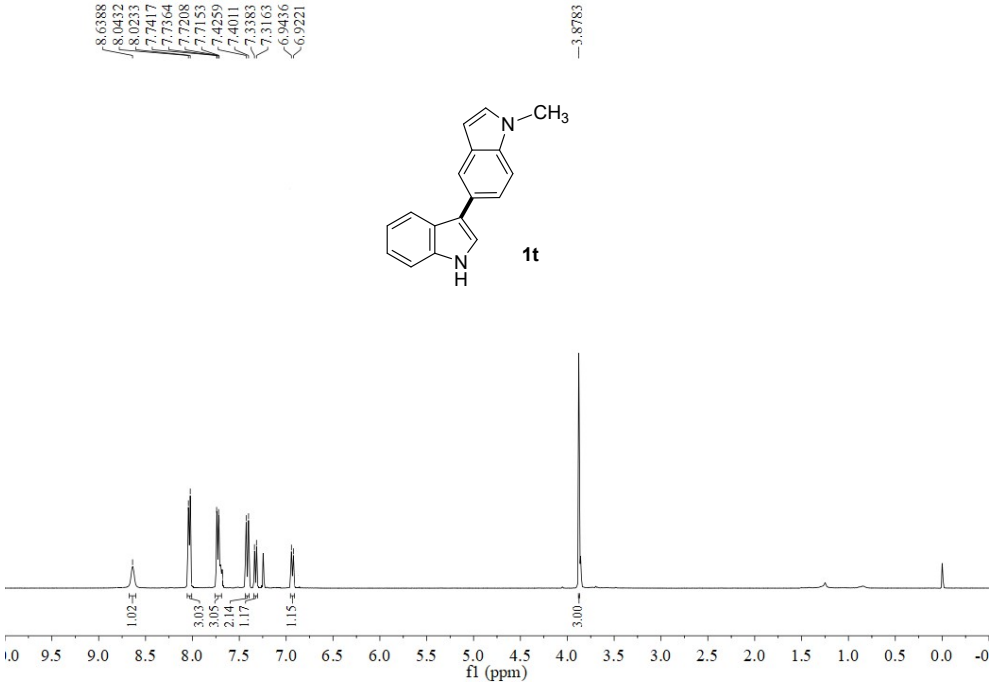

136.57
134.63
128.39
127.19
126.22
124.55
122.69
122.15
121.27
119.97
119.41
117.29
111.26
111.24
102.69
98.68

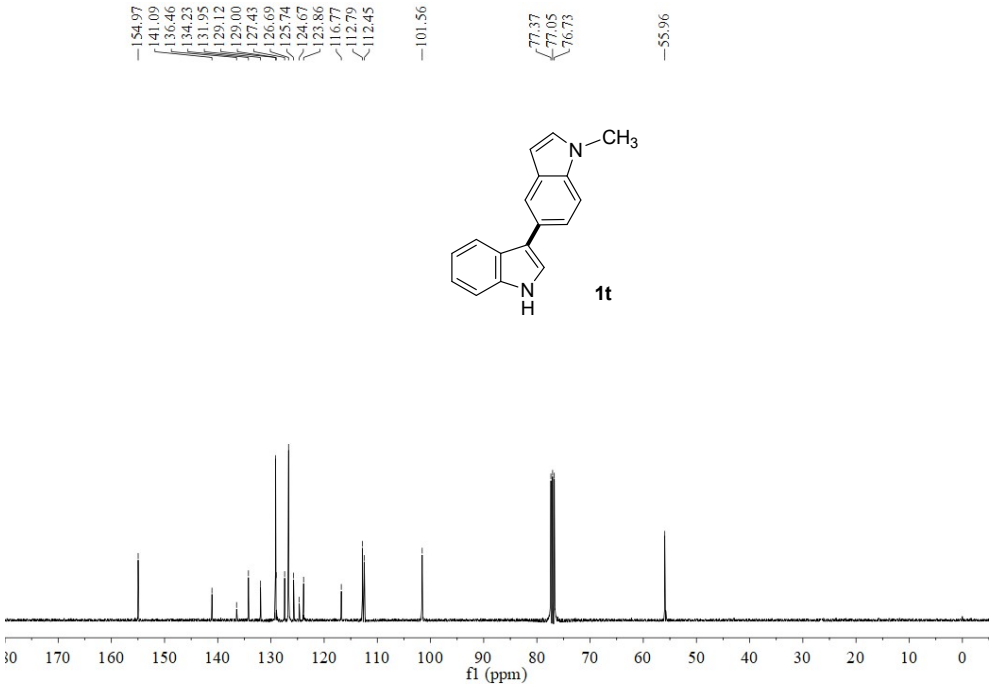

77.33
77.01
76.70


NH
N
H

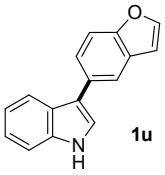
1s


f1 (ppm)

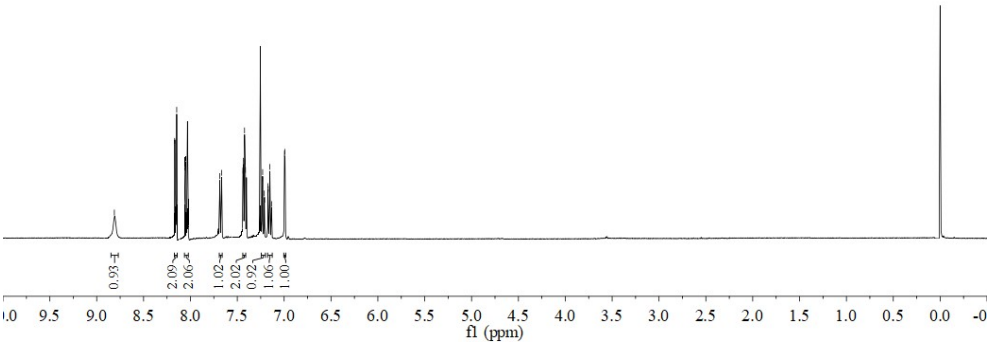

8.6388
8.0432
8.0233
7.7417
7.7364
7.7208
7.7153
7.4259
7.4011
7.3383
7.3163
6.9436
6.9221
3.8783
N
CH3
N
H
1t
1.02
3.03
3.05
2.14
1.17
1.15
3.00
f1 (ppm)


154.97
141.09
136.46
134.23
131.95
129.12
129.00
127.43
126.69
125.74
124.67
123.86
116.77
112.79
112.45
101.56
77.37
77.05
76.73
55.96
N
CH3
N
H
1t
f1 (ppm)

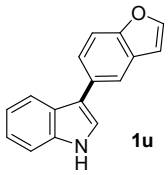

8.8118
8.1679
8.1465
8.0820
8.0768
8.0596
8.0546
7.6867
7.6671
7.4402
7.4338
7.4226
7.4187
7.4127
7.2323
7.2294
7.2121
7.2090
7.1719
7.1694
7.1522
7.1346
6.9963
6.9909


O
N
H

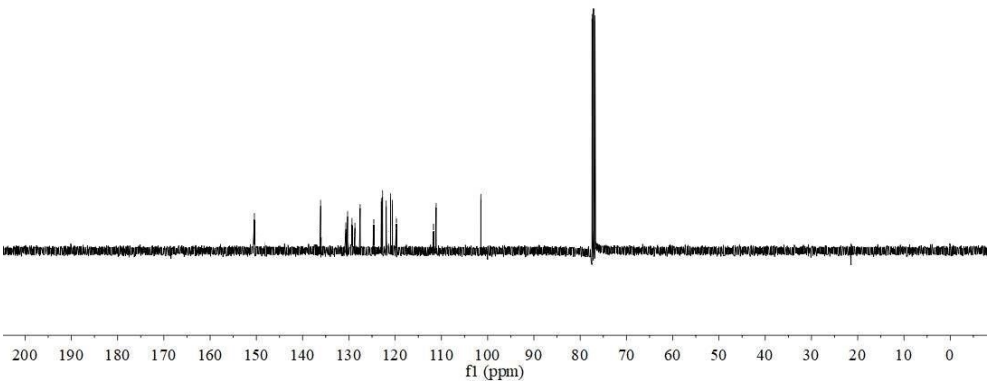
1u


0.93
2.09
2.06
1.02
2.02
0.92
1.06
1.00
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
f1 (ppm)

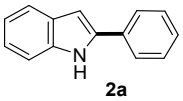

150.43
136.11
130.66
130.28
129.32
128.69
127.57
124.93
122.99
122.72
121.93
120.58
119.81
111.99
111.13
101.47
77.43
77.12
76.80


O
N
H

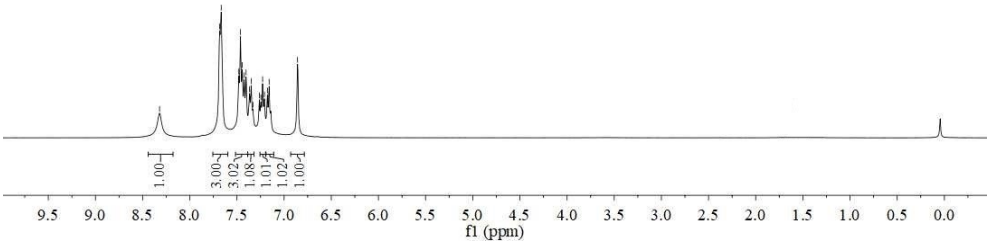
1u


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

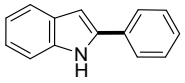

8.3204
7.6843
7.6668
7.4806
7.4624
7.4436
7.4229
7.4033
7.3670
7.3485
7.3309
7.2623
7.2457
7.2280
7.2086
7.1767
7.1586
6.8577



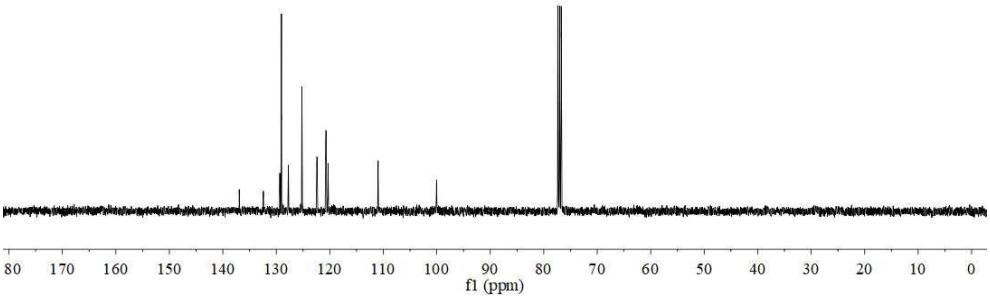
2a

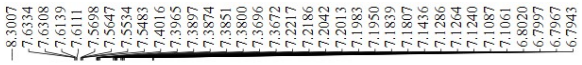

1.00
3.00
3.02
1.08
1.01
1.02
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

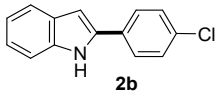

136.93
132.43
129.34
129.04
127.74
125.22
122.40
120.71
120.33
110.97
100.04
77.37
77.05
76.74



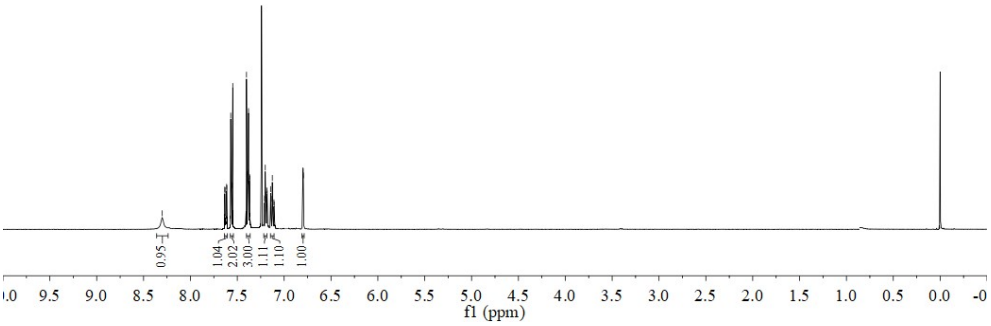
2a

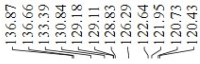

80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

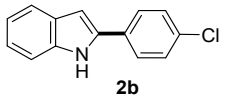

8.3007
7.6334
7.6308
7.6139
7.6111
7.5698
7.5647
7.5534
7.5483
7.4016
7.3965
7.3897
7.3874
7.3851
7.3800
7.3696
7.3672
7.2217
7.2186
7.2042
7.2013
7.1983
7.1950
7.1839
7.1807
7.1436
7.1286
7.1264
7.1240
7.1087
7.1061
6.8020
6.7997
6.7967
6.7943


Cl
N
H

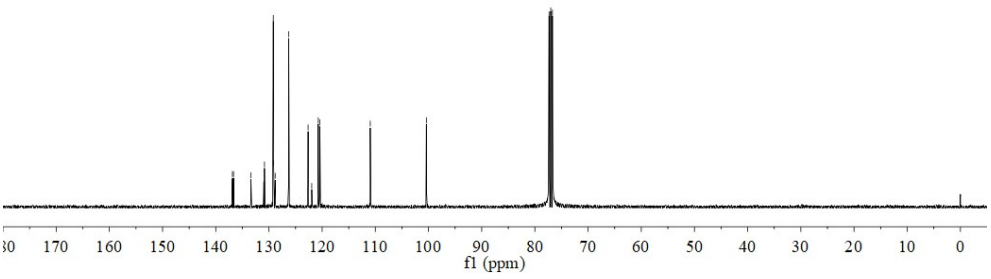
2b

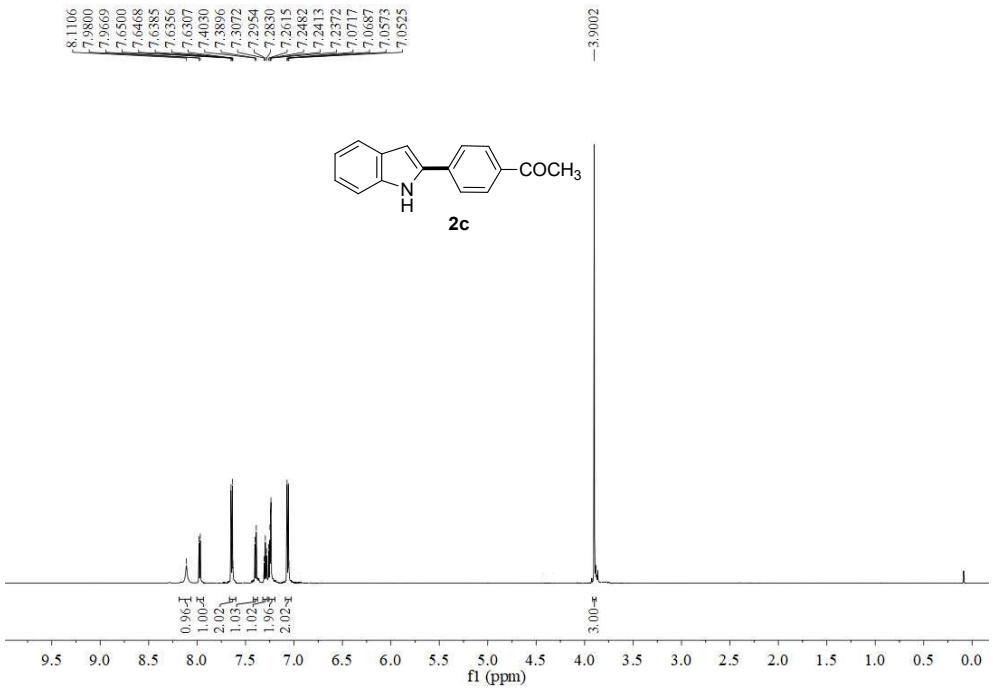

0.95
1.04
2.02
3.00
1.11
1.10
1.00
f1 (ppm)

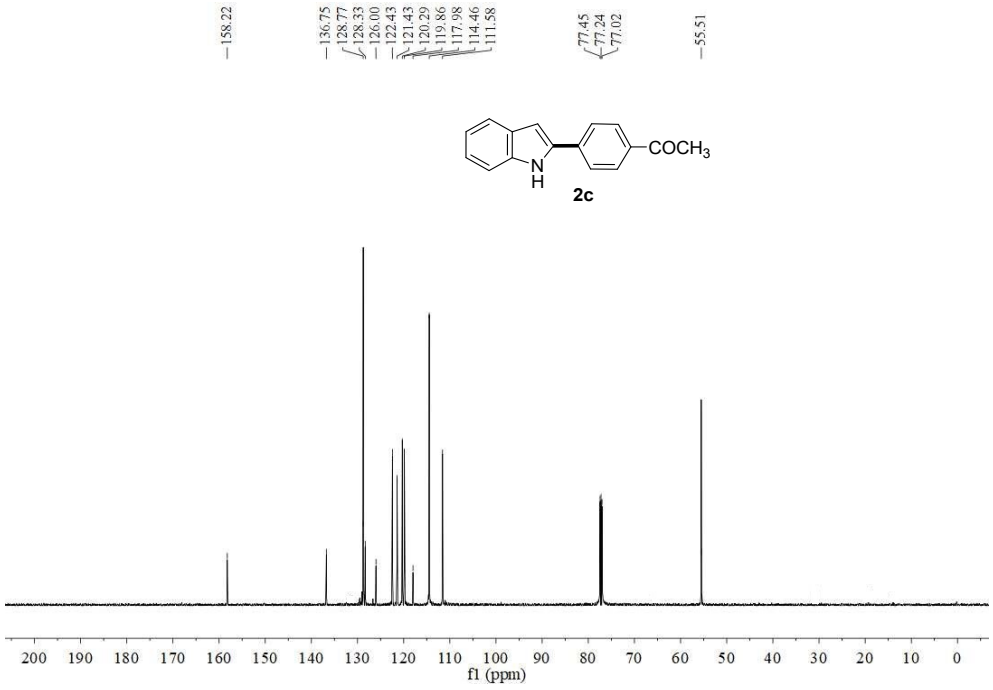

136.87
136.66
133.39
130.84
129.18
129.11
128.83
126.29
122.64
121.95
120.73
120.43
110.95
100.40
77.33
77.02
76.70

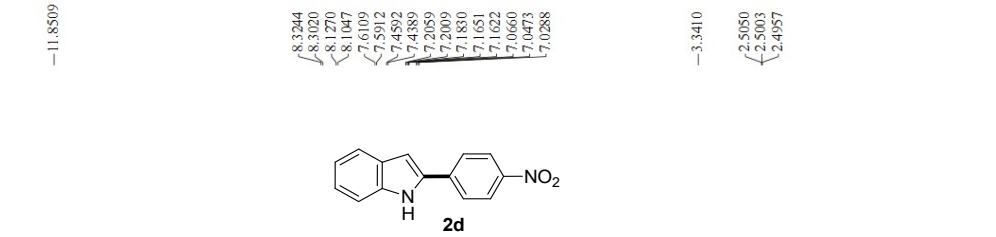

Cl
N
H

2b

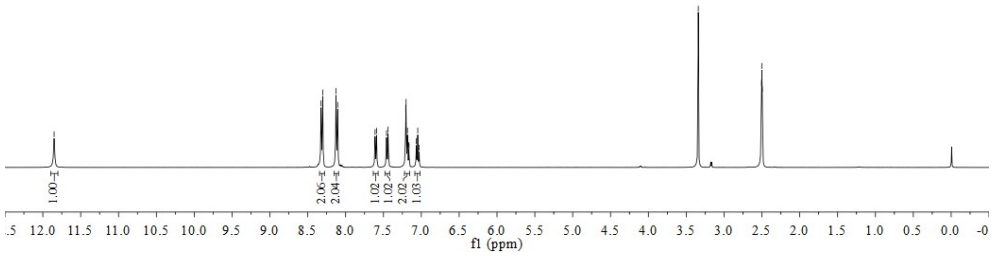

f1 (ppm)

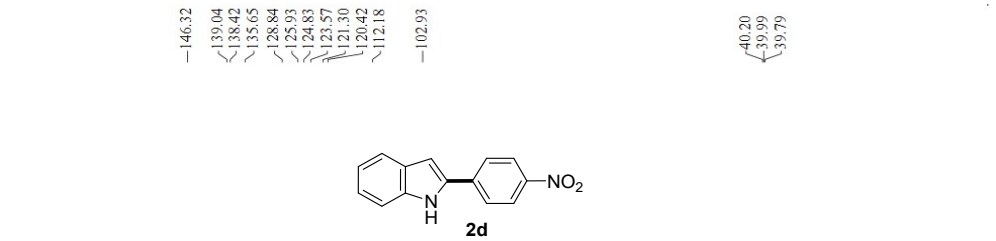

8.1106
7.9800
7.9669
7.6500
7.6468
7.6385
7.6356
7.6307
7.4030
7.3896
7.3072
7.2954
7.2830
7.2615
7.2482
7.2413
7.2372
7.0717
7.0687
7.0573
7.0525
3.9002
COCH3
N
H
2c
0.96
1.00
2.02
1.03
1.02
1.96
2.02
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


158.22
136.75
128.77
128.33
126.00
122.43
121.43
120.29
119.86
117.98
114.46
111.58
77.45
77.24
77.02
55.51
COCH3
N
H
2c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

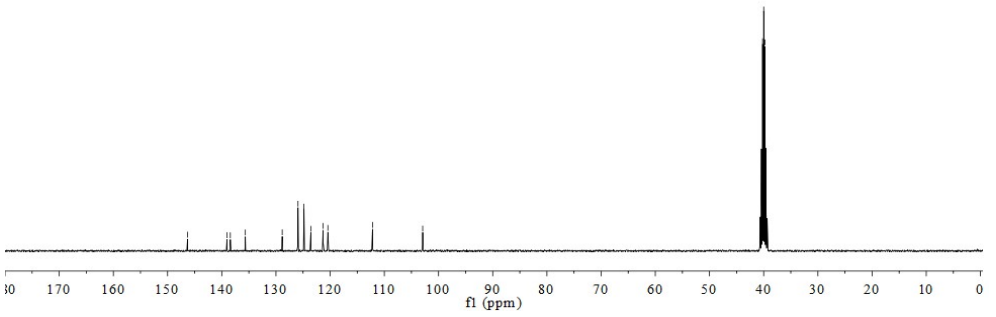

11.8509
8.3244
8.3020
8.1270
8.1047
7.6109
7.5912
7.4592
7.4389
7.2059
7.2009
7.1830
7.1651
7.1622
7.0660
7.0473
7.0288
3.3410
2.5050
2.5003
2.4957
NO2
N
H

2d

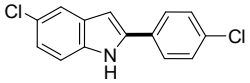

1.00
2.06
2.04
1.02
1.02
2.02
1.08
f1 (ppm)


146.32
139.04
138.42
135.65
128.84
125.93
124.83
123.57
121.30
120.42
112.18
102.93
40.20
39.99
39.79
NO2
N
H

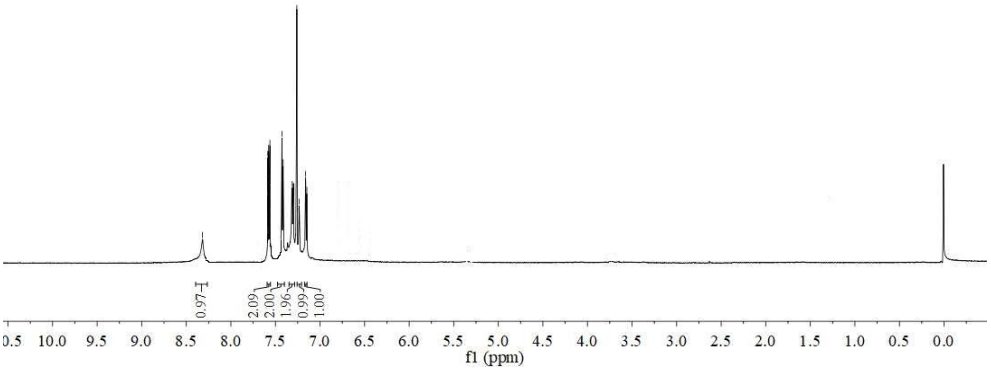
2d


f1 (ppm)

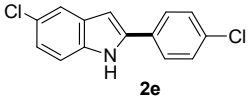

8.3182
7.6150
7.5862
7.5761
7.5622
7.4275
7.4138
7.3168
7.3115
7.2599
7.2370
7.2332
7.1614
7.1472


Cl
N
H
Cl

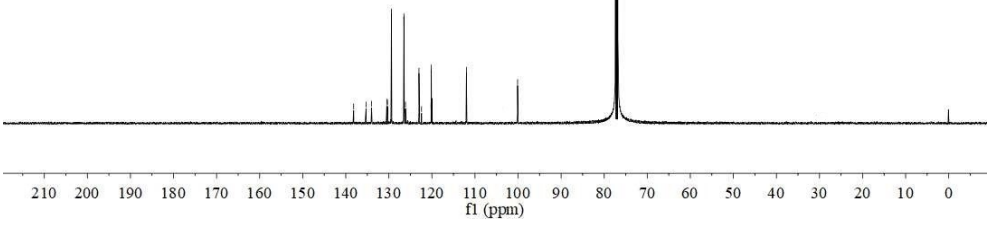
2e

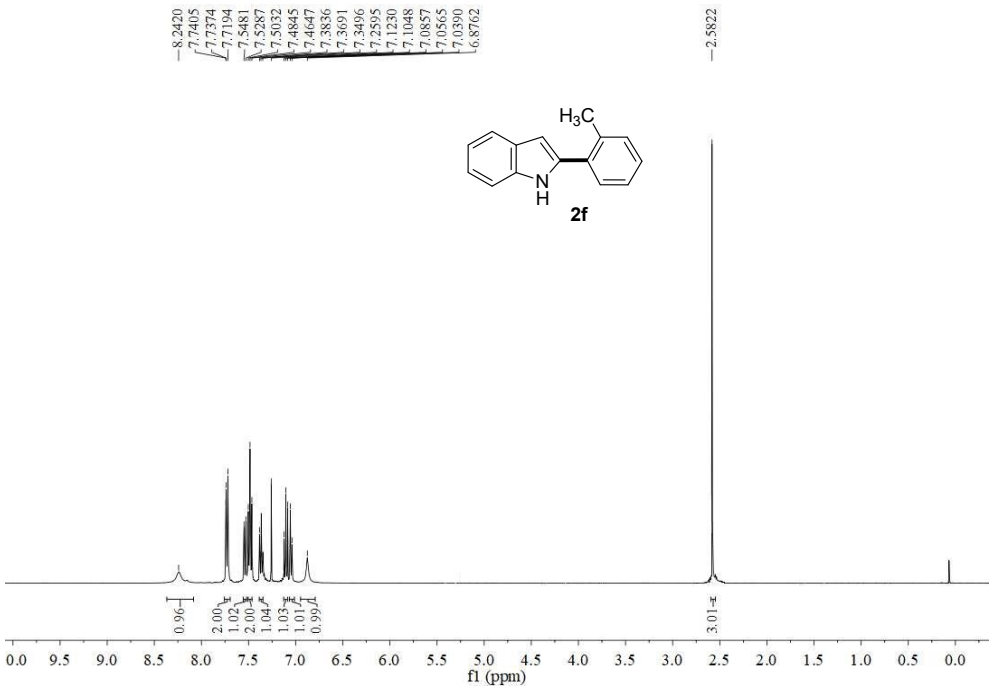

0.97
2.09
2.00
1.96
0.99
1.00
0.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

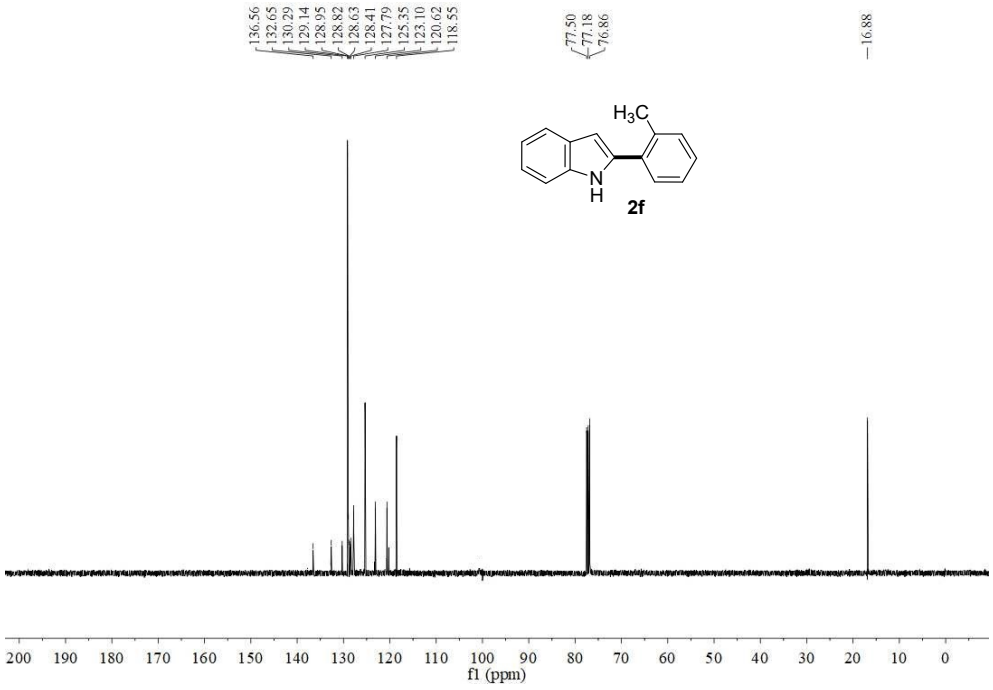

138.1707
135.3009
134.0073
130.4802
130.3112
129.3919
126.4961
126.1495
123.0012
122.4122
120.2070
120.1527
111.9856
100.1061
77.2999
77.0879
76.8758

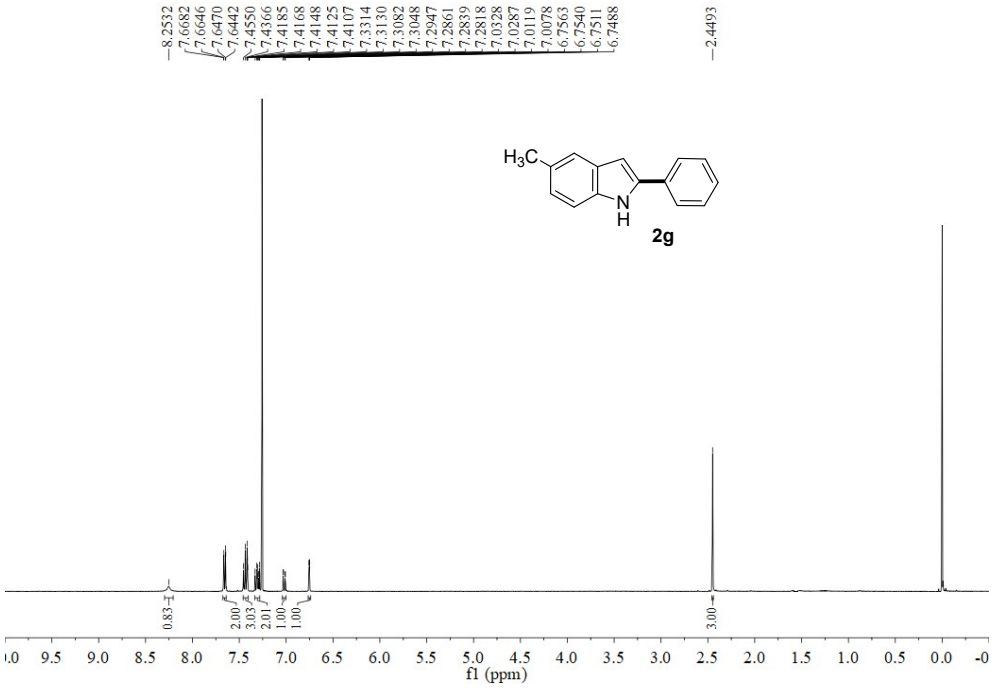

Cl
N
H
Cl

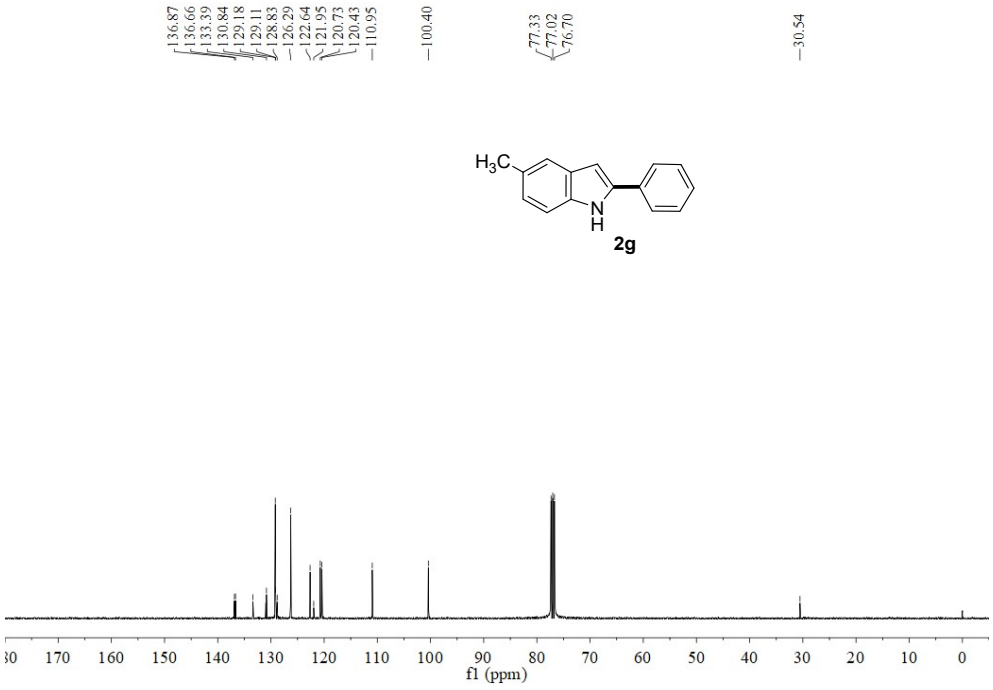
2e

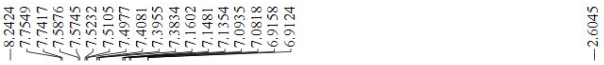

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

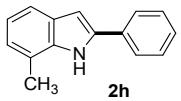

8.2420
7.7405
7.7374
7.7194
7.5481
7.5287
7.5032
7.4845
7.4647
7.3836
7.3691
7.3496
7.2595
7.1230
7.1048
7.0857
7.0565
7.0390
6.8762
2.5822
H3C
N
H
2f
0.96
2.00
1.02
2.00
1.04
1.03
1.01
0.99
3.01
0.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


136.56
132.65
130.29
129.14
128.95
128.82
128.63
128.41
127.79
125.35
123.10
120.62
118.55
77.50
77.18
76.86
16.88
H3C
N
H
2f
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

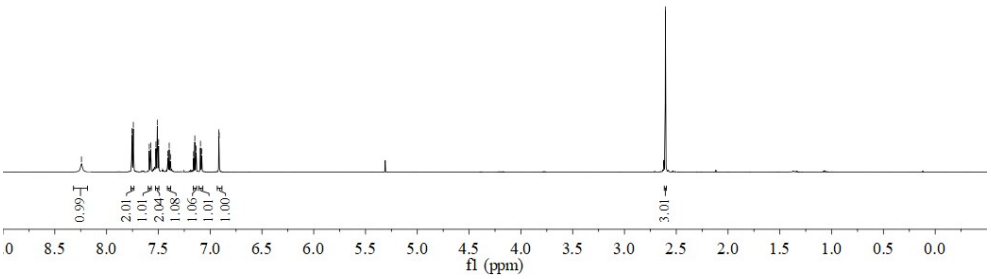

8.2532
7.6682
7.6646
7.6470
7.6442
7.4550
7.4366
7.4185
7.4168
7.4148
7.4125
7.4107
7.3314
7.3130
7.3082
7.3048
7.2947
7.2861
7.2839
7.2818
7.0328
7.0287
7.0119
7.0078
6.7563
6.7540
6.7511
6.7488
2.4493
H3C
N
H
2g
0.83
2.00
3.03
2.01
1.00
1.00
3.00
f1 (ppm)

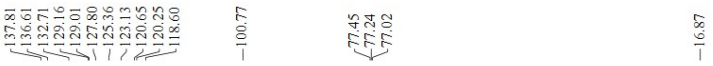

136.87
136.66
133.39
130.84
129.18
129.11
128.83
126.29
122.64
121.95
120.73
120.43
110.95
100.40
77.33
77.02
76.70
30.54
H3C
N
H
2g
f1 (ppm)

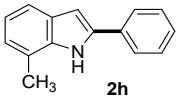

8.2424
7.7549
7.7417
7.5876
7.5745
7.5232
7.5105
7.4977
7.4081
7.3955
7.3834
7.1602
7.1481
7.1354
7.0935
7.0818
6.9158
6.9124
2.6045


N
H
CH3

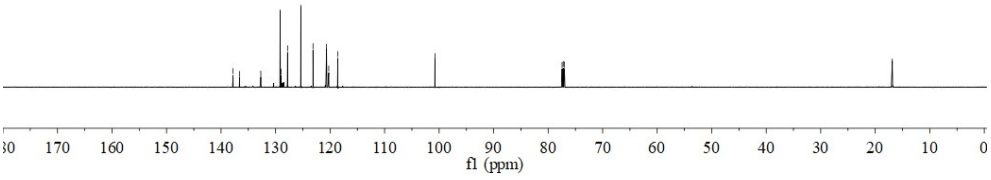
2h

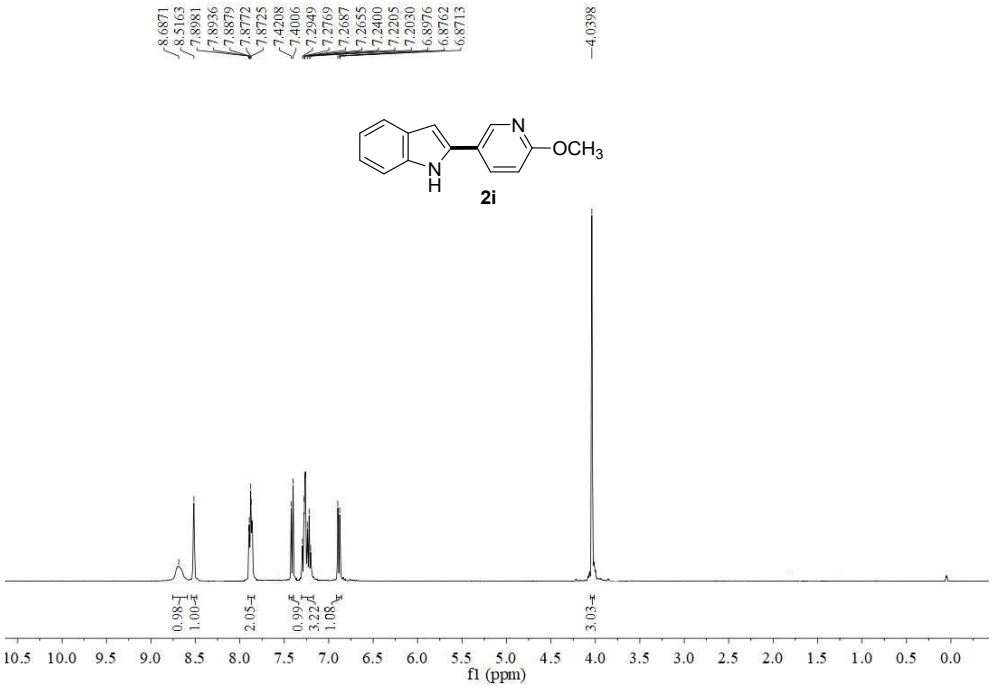

0.99
2.01
1.01
2.04
1.08
1.06
1.01
1.00
3.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

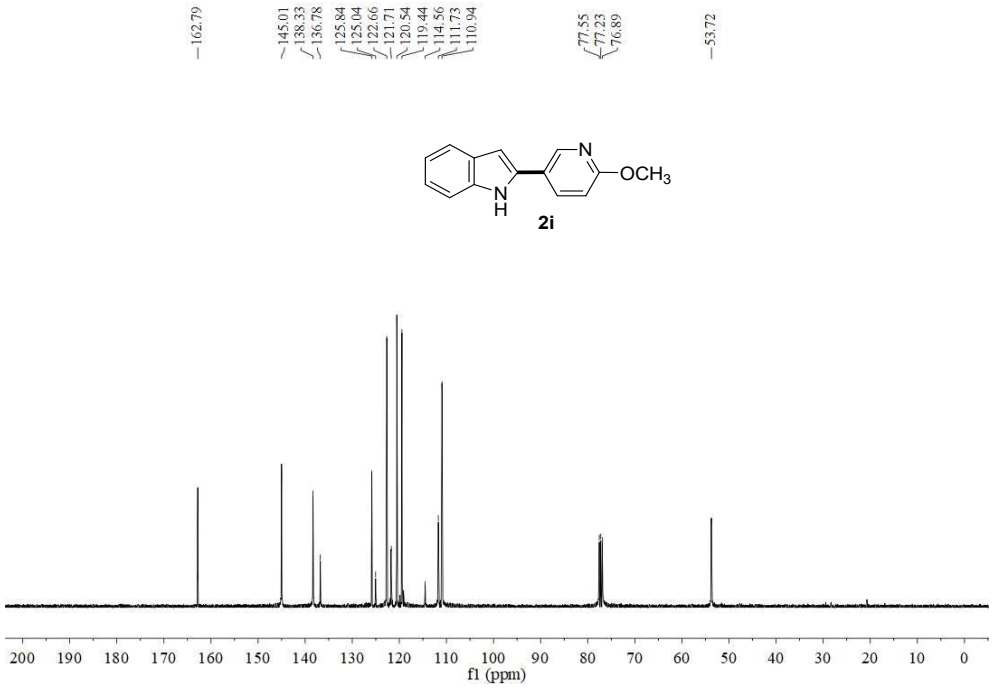

137.81
136.61
132.71
129.16
129.01
127.80
125.36
123.13
120.65
120.25
118.60
100.77
77.45
77.24
77.02
16.87


N
H
CH3

2h


170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.6871
8.5163
7.8981
7.8936
7.8879
7.8772
7.8725
7.4208
7.4006
7.2949
7.2769
7.2687
7.2655
7.2400
7.2205
7.2030
6.8976
6.8762
6.8713
4.0398
N
OCH3
N
H
2i
0.98
1.00
2.05
0.99
3.22
1.08
3.03
10.5 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
f1 (ppm)


162.79
145.01
138.33
136.78
125.84
125.04
122.66
121.71
120.54
119.44
114.56
111.73
110.94
77.55
77.23
76.89
53.72
N
OCH3
N
H
2i
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)