Supporting Information

Montmorillonite K10 catalyzed facile synthesis of *N*-substituted indoles from primary amine and Morita–Baylis–Hillman acetate of cyclohexenone

Juqing Han,[a] Luxia Guo,[a] Fuwei Zhang,[a] Minghao Li,[a]* and Yanlong Gu [a,b,c]*

[a] Key Laboratory for Large-Format Battery Materials and System, Ministry of Education, School of Chemistry and Chemical Engineering, Huazhong University of Science and Technology, 430074, Wuhan, China.

[b] School of Chemistry and Chemical Engineering, The Key Laboratory for Green Processing of Chemical Engineering of Xinjiang Bingtuan, Shihezi University, Shihezi City 832004, China.

[c] State Key Laboratory for Oxo Synthesis and Selective Oxidation, Lanzhou Institute of Chemical Physics, 730000, Lanzhou, China.

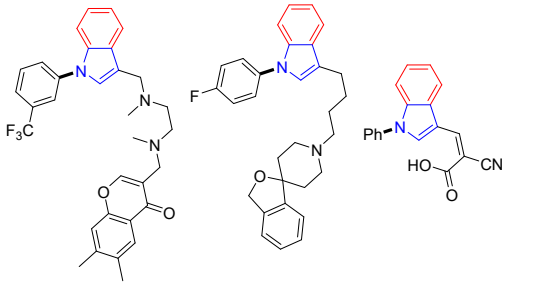
Corresponding authors: M. Li (liminghaochem@hust.edu.cn) and Y. Gu (klgyl@hust.edu.cn)

Table of contents

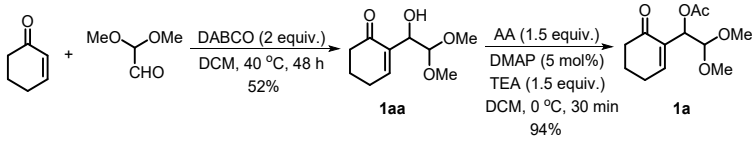
1. General information

Unless otherwise noted, all reagents were purchased from commercial suppliers and used without further purification. The reactions were monitored by TLC with Haiyang GF-254 silica gel plates (Qingdao Haiyang chemical industry Co. Ltd, Qingdao, China) using UV light or $KMnO_4$ as visualizing agents as needed. Flash column chromatography was performed using 200-300 mesh silica gel at increased pressure. ^1H NMR spectra and ^{13}C NMR spectra were respectively recorded on Brüker AV-400 spectrometers. Chemical shifts (δ) were expressed in ppm relative to Me_4Si in $CDCl_3$ or DMSO-d_6, and coupling constants (J) were reported in Hz. High-resolution mass spectra (HRMS) were obtained on Brüker Compass Data Analysis 4.0. Melting points were determined on a microscopic melting point apparatus and are uncorrected. IR spectra were recorded on a Bruker FT-IR (EQUINOX 55) using KBr pellets or neat liquid technology.

2. Some representative bioactive molecules synthesized from *N*-substituted indoles (Figure S1)



3. The procedure for synthesis of 1a.



Step I: Preparation of MBH alcohol 1aa: 2-Cyclohexen-1-one (1.44 g, 15 mmol) and DABCO (1.68 g, 15 mmol) was mixed with DCM (15 mL) in a 100 mL of round bottomed flask equipped with a magnetic stirring bar. The mixture was stirred at room temperature until dissolution completely. Then the 2,2-dimethoxyacetaldehyde (60 wt. % in H_2O, 1.73 g, 10 mmol) was added. The mixture was stirred at 40 °C for 48 h. After completion of reaction, the mixture washed with 0.5 M HCl solution (3 × 15 mL). The organic phase was washed with brine (30 mL) and dried over Na_2SO_4. After removing volatile components, the organic residue was subjected to silica column chromatography for isolation (eluting solution: petroleum ether / ethyl acetate = 5/1 (v/v)) to yield a colorless transparent liquid (MBH alcohol, 1.56 g, 52% yield).

Step II: Preparation of MBH acetates 1a: To a solution of MBH alcohol (2.00 g, 10 mmol) prepared by Step I, Ac_2O (1.53 g, 1.5 equiv.), 4-dimethylaminopyridine (DMAP, 0.06 g, 5 mol%), and Et_3N (1.52 g, 1.5 equiv.) in anhydrous dichloromethane (15 mL) were added. The mixture was stirred at 0 °C for 20 min. Upon completion of the reaction, the mixture was washed with a saturated solution of $NaHCO_3$ in 20 mL of water. The organic phase was collected and dried over

anhydrous sodium sulphate. After filtration and concentration, the organic residue was subjected to silica column chromatography (eluting solution: petroleum ether / ethyl acetate = 10/1 (v/v)) and the MBH acetate was obtained as a yellow liquid (MBH acetate, 2.28 g, 94% yield).

4. General procedure for the reaction of MBH acetates 1a and arylamine

The MBH acetate **1a** (0.3 mmol, 72.6 mg), *p*-toluidine (0.3 mmol, 32.1 mg), and Montmorillonite K10 (10 mg) were mixed in a 10 mL of V-type flask equipped with a triangle magnetic stirring bar. Then 1.0 mL diethyl carbonate was added. The mixture was stirred at 100 ℃ for 3 h. After the reaction was completed, the mixture was cooled to room temperature, and directly subjected to preparative TLC plate for isolation (eluting solution: petroleum ether / ethyl acetate = 20/1 (v/v)). The 1-(*p*-tolyl)-1*H*-indole **3a** was obtained as a pale-yellow liquid. Tests for substrate scope were all performed with an analogous procedure.

5. The procedure for the reaction of MBH acetates 1a and alkylamine

The MBH acetate **1a** (0.3 mmol, 72.6 mg), hexan-1-amine (0.3 mmol, 30.3 mg) and Montmorillonite K10 (10.0 mg) were mixed in a 10 mL of V-type flask equipped with a triangle magnetic stirring bar. Then 1.0 mL of diethyl carbonate (DEC) was added. The mixture was stirred at 120 ℃ for 3 h. After the reaction was completed, the mixture was cooled to room temperature, and directly subjected to preparative TLC plate for isolation (eluting solution: petroleum ether / ethyl acetate = 20/1 (v/v)). The 1-hexyl-1*H*-indole **3ac** was obtained as a pale-yellow liquid. Tests for substrate scope were all performed with an analogous procedure.

6. The procedure for the preparation of 1,1'-biindole

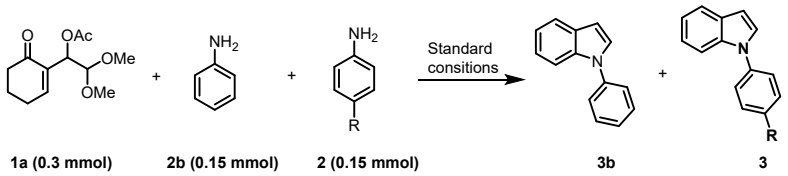
Step I: The MBH acetate **1a** (0.5 mmol, 121.0 mg) and benzoylhydrazine (0.5 mmol, 68.1 mg) and montmorillonite K10 (20.0 mg) were mixed in a 10 mL of V-type flask equipped with a triangle magnetic stirring bar. Then 3.3 mL of diethyl carbonate (DEC) was added. The mixture was stirred at 100 ℃ for 3 h. After the reaction was completed, the mixture was cooled to room temperature, and directly subjected to preparative TLC plate for isolation (eluting solution: petroleum ether / ethyl acetate = 16/1 (v/v)). The *N*-(1*H*-indol-1-yl)benzamide **5a** was obtained as a pale yellow solid (82.6 mg, yield =70%).

Step II: In a 10 mL of V-type flask equipped with a triangle magnetic stirring bar, *N*-(1*H*-indol-1-yl)benzamide **5a** (0.3 mmol, 70.8 mg) was dissolved in EtOH (1 mL), then $N_2H_4 \cdot H_2O$ (0.33 mmol, 10.6 mg) was added. The mixture was refluxed at 80 ℃ for 3 h. After the reaction was completed, the mixture was cooled to room temperature, and directly subjected to preparative TLC plate for isolation (eluting solution: petroleum ether / ethyl acetate = 15/1 (v/v)). The 1*H*-indol-1-amine (**5b**) was obtained as a pale-yellow solid (36.1 mg, yield = 91%).

Step III: The 1*H*-indol-1-amine (**5b**) (0.2 mmol, 26.1 mg) and MBH acetate **1a** (0.2 mmol, 48.4 mg) and montmorillonite K10 (6.6 mg) were mixed in a 10 mL of V-type flask equipped with a triangle magnetic stirring bar. Then 1.0 mL of diethyl carbonate (DEC) was added. The mixture was stirred at 100 ℃ for 3 h. After the reaction was completed, the mixture was cooled to room temperature, and directly subjected to preparative TLC plate for isolation (eluting solution:

petroleum ether / ethyl acetate = 20/1 (v/v)). 1,1'-biindole was obtained as a pale yellow solid (30.2 mg, yield = 65%).

7. The competitive experiments



The mixture of **1a** (0.3 mmol), **2b** (0.15 mmol), and para-substituted aniline (0.15 mmol), montmorillonite K10 (10 mg) were mixed in a 10 mL of V-type flask equipped with a triangle magnetic stirring bar. Then 1.0 mL diethyl carbonate was added. The mixture was stirred at 100 ^{o}C for 3 h. After the reaction was completed, the mixture was cooled to room temperature, and directly subjected to preparative TLC plate for isolation. The ratio of **3b** and **3** was analyzed by ^{1}H NMR in $CDCl_3$ and the result was summarized in **Table S1**. These data were plotted according to the Hammett equation to give the Hammett plot.

Table S1 The result of competitive experiments

Entry	R	Relative rate (3/3b) = k/k_0	log (k/k_0)	σ_{para}
1	OMe	3.94	0.595	-0.27
2	Me	1.74	0.241	-0.17
3	Cl	0.67	-0.174	0.227
4	CO_2Et	0.12	-0.921	0.45

8. Evaluation of green metrics

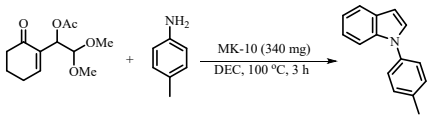
Table S2 The result of competitive experiments

Green metrics	Present work	Buchwald' work	Vaccaro's work	Bhalla's work	Karmakar's work
E-factor[a]	2.32	7.32	9.80	7.15	16.60
Eco-Scale	66.5	66.0	63.0	61.0	65.0

[a] For comparison, the drying agent and silica gel used for flash column chromatography were out of consideration because those were not reported in the otherwise available approaches.

The E-factor calculation is based on the equation: E-factor = m(waste)/m(product). The EcoScale calculation is based on the equation EcoScale = 100 – Sum of individual penalties (Score on EcoScale: > 75, Excellent; >50, Acceptable; <50, Inadequate). The calculation result was summarized in **Table S2**. The calculation details were displayed as following:

(a) Our current work

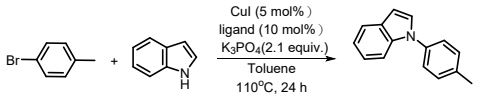


Reactant 1	MBH acetate	2.42 g	10 mmol	FW 242.12
Reactant 2	*p*-Toluidine	1.07 g	10 mmol	FW 107.07
Catalyst	MK-10	0.34 g	—	—
Solvent	DEC (18 ml)	17.55 g	—	—
Recycled	solvent 16 ml	15.60 g	—	—
	catalyst	0.34g	—	—
Product (79% yield)	1-(*p*-tolyl)-1*H*-indole	1.64 g	7.9 mmol	FW 207.10
E-factor	[(2.42+1.07+0.34+17.55)-(15.60+0.34+1.64)]/1.64 = 2.32			

Parameter	Penalty points
1. Yield (100-%yield)/2 = (100-79)/2	10.5
2. Price of reaction components	
(To obtain 10 mmol of end product)	
a. 1a = 2.42 g = $ 14.53 (estimated)	
b. *p*-Toluidine = 1.07 g = $0.12	
c. MK-10 = 0.34 g = $0.15	
d. DEC = 18ml = $1.62	
Total price (USD) = $16.42	
Thus, inexpensive (< $50)	0
3. Safety	
Solvent: DEC	
Toxic (T)	5
Highly flammable (F)	5
4. Technical Setup	
Common setup	0
5. Temperature and time	
Room temperature < 24 h	3
6. Workup and purification	
Removal of solvent with bp < 150°C	0
Classical Chromatography	10
Total Penalty Points	33.5

EcoScale = 100 – 33.5 = 66.5 (an acceptable synthesis)

(b) Buchwald' work (*J. Am. Chem. Soc.*, 2002, **124**, 11684.)

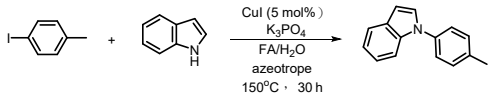


Reactant 1	indole	0.117 g	1.00 mmol	FW 117.06
Reactant 2	1-bromo-4-methylbenzene	0.203 g	1.20 mmol	FW 169.97
Catalyst	CuI	0.002 g	0.01 mmol	FW 191.46
Ligand	(1S,2S)-N,N'-Dimethyl-1,2-cyclohexanediamine	0.007 g	0.05 mmol	FW 142.24
Base	K_3PO_4	0.446 g	2.10 mmol	FW 212.27
Solvent	Toluene (1 ml)	0.872 g	—	—
Recycled solvent	—	—	—	—
Product (96% yield)	1-(*p*-tolyl)-1*H*-indole	0.198 g	0.96 mmol	FW 207.10
E-factor	(0.117+0.203+0.002+0.007+0.446+0.872-0.198) / 0.198 = 7.32			

Parameter	Penalty points
1. Yield (100-%yield)/2 = (100-96)/2	2
2. Price of reaction components	
(To obtain 10 mmol of end product)	
a. 1a = 1.17 g = $3.27	
b. 1-bromo-4-methylbenzene = 2.03 g = $24.89	
c. Ligand = 0.07 g = $20.58	
d. CuI = 0.02 g = $0.08	
e. K_3PO_4 = 4.46 g = $1.07	
f. Toluene = 8.72 g = $0.05	
Total price (USD) = $51.94	
Thus, expensive (> $50)	5
3. Safety	
Solvent: Toluene	3
Toxic (T)	5
Highly flammable (F)	5
4. Technical Setup	
Common setup	1
5. Temperature and time	
Heating, > 1 h	3
6. Workup and purification	
Removal of solvent with bp < 150°C	0
Classical Chromatography	10
Total Penalty Points	34.0

EcoScale = 100 – 33.0 = 66.0 (an acceptable synthesis)

(c) Vaccaro's work (*Green Chem.*, 2018, **20**, 1634)

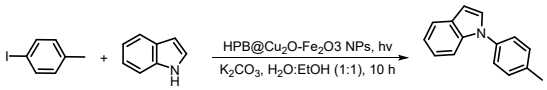


Reactant 1	indole	0.117 g	1.00 mmol	FW 117.06
Reactant 2	4-iodotoluene	0.218 g	1.00 mmol	FW 218.04
Base	K_3PO_4	0.424 g	2.10 mmol	FW 212.27
Catalyst	CuI	0.009 g	0.05 mmol	FW 191.46
Solvent	FA/H_2O azeotrope	2.050 g	2 mL	—
Extraction solvent	EtOAc	0.714 g	0.8 mL	
Recycled solvent	FA/H_2O azeotrope	1.74 g	—	—
Product (80% yield)	1-(*p*-tolyl)-1*H*-indole	0.166 g	0.80 mmol	FW 207.10
E-factor	(0.117+0.218+0.424+0.009+2.050+0.714-1.74-0.166)/0.166 = 9.80			

Parameter	Penalty points
1. Yield (100-%yield)/2 = (100-80)/2	10
2. Price of reaction components (To obtain 10 mmol of end product) a. indole = 1.17 g = \$3.27 b. 4-iodotoluene = 2.03 g = \$10.72 c. FA/H_2O azeotrope = 20.50 g = \$0.821 d. CuI = 0.09 g = \$0.36 e. K_3PO_4 = 4.24 g = \$1.02 f. EtOAc = 7.14 G = \$0.39 Total price (USD) = \$16.58 Thus, expensive (< \$50)	3
3. Safety	
Solvent: FA/H_2O azeotrope	0
Toxic (T)	5
Highly flammable (F)	5
4. Technical Setup	
Common setup	1
5. Temperature and time	
Heating, > 1 h	3
6. Workup and purification	
Removal of solvent with bp < 150°C	0
Classical Chromatography	10
Total Penalty Points	37.0

EcoScale = 100 – 37.0 = 63.0 (an acceptable synthesis)

(d) Bhalla's work (*Green Chem.,* 2018, **20,** 5346)

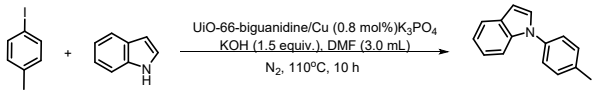


Reactant 1	indole	0.234 g	2.00 mmol	FW 117.06
Reactant 2	4-iodotoluene	0.087 g	0.40 mmol	FW 218.02
Catalyst	HPB@Cu_2O–Fe_2O_3 NPs	—	0.002 mmol	—
Base	K_2CO_3	0.138 g	1.00 mmol	FW 138.21
Solvent	H_2O (0.5 ml)+EtOH (0.5 ml)	0.499 g + 0.395 g		
Recycled Catalyst	HPB@Cu_2O–Fe_2O_3 NPs	—	0.002 mmol	—
Product (80% yield)	1-(*p*-tolyl)-1*H*-indole	0.166 g	0.80 mmol	FW 207.10
E-factor	(0.234+0.087+0.138+0.499+0.395-0.166)/0.166 = 7.15			

Parameter	Penalty points	
	1. Yield (100-%yield)/2 = (100-80)/2	10
	2. Price of reaction components	
	(To obtain 10 mmol of end product)	
	a. indole = 5.85 g = \$11.24	
	b. 4-iodotoluene = 2.18 g = \$10.72	
	c. Reactant 3 = \$34.55	
	d. K_2CO_3 = 3.45 g = \$1.77	
	f. H_2O = 4.99 g = \$0.22	
	g. EtOH = 3.95 g = \$0.66	
	Total price (USD) = \$59.16	
	Thus, expensive (>50)	5
	3. Safety	
	Solvent: H_2O:EtOH(1:1)	
	Toxic (T)	5
	Highly flammable (F)	5
	4. Technical Setup	
	Common setup	1
	5. Temperature and time	
	Room temperature < 24 h	3
	6. Workup and purification	
	Removal of solvent with bp < 150°C	0
	Classical Chromatography	10
	Total Penalty Points	39.0

EcoScale = 100 – 39.0 = 61.0 (an acceptable synthesis)

(e) Karmakar's work (*RSC Adv.*, 2021, **11**, 22278)



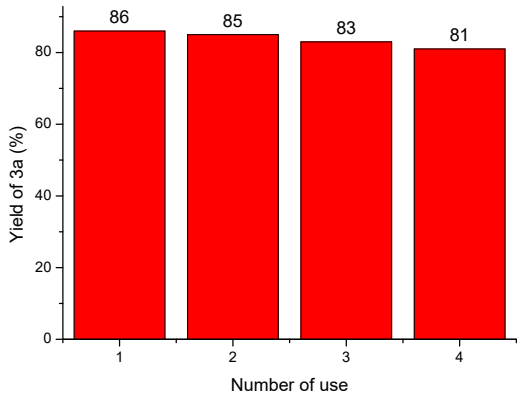
Reactant 1	indole	0.117 g	1.00 mmol	FW 117.06
Reactant 2	4-iodotoluene	0.240 g	1.10 mmol	FW 218.02
Catalyst	UiO-66-biguanidine/Cu	—	0.008 mmol	—
Base	KOH	0.084 g	1.5 mmol	FW 56.11
Solvent	DMF(3mL)	2.832 g		
Recovered Catalyst	UiO-66-biguanidine/Cu	—	0.008mmol	
Product (90% yield)	1-(*p*-tolyl)-1*H*-indole	0.186 g	0.90 mmol	FW 207.10
E-factor	(0.117+0.240+0.084+2.832-0.186)/0.186 = 16.6			

Parameter	Penalty points	
	1. Yield (100-%yield)/2 = (100-90)/2	5
	2. Price of reaction components	
	(To obtain 10 mmol of end product)	
	a. 1a = 1.17 g = $3.27	
	b. 4-iodotoluene = 2.18 g = $10.72	
	c. Reactant 3 = $2.11	
	d. KOH = 0.084 g = $0.10	
	f. DMF = 2.832 g = $0.53	
	Total price (USD) = $16.72	
	Thus, expensive (< 50)	3
	3. Safety	
	Solvent: DMF	3
	Toxic (T)	5
	Highly flammable (F)	5
	4. Technical Setup	
	Common setup	1
	5. Temperature and time	
	Heating, > 1 h	3
	6. Workup and purification	
	Removal of solvent with bp < 150°C	0
	Classical Chromatography	10
	Total Penalty Points	35.0

EcoScale = 100 – 35.0 = 65.0 (an acceptable synthesis)

9. The catalyst recycling procedure and reused results (Figure S2)

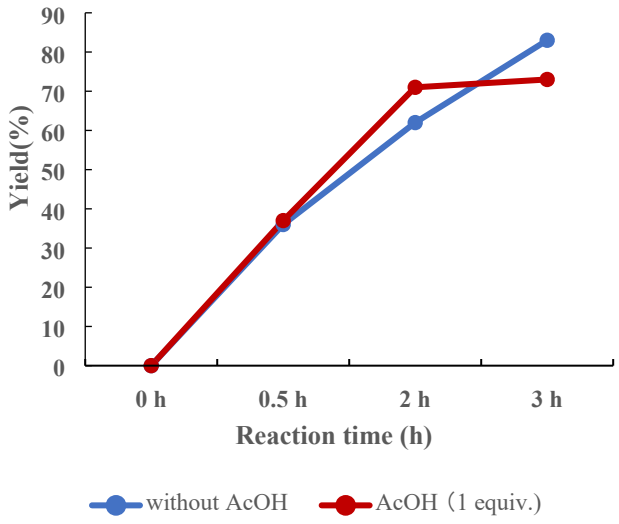
After the reaction, the Montmorillonite K10 was filtrated and washed with EtOAc for 3 times. Then it was calcined at 400 ℃ under air condition for 4 h. After the recovered catalyst was cooled down to room temperature, it was used for next run.



It is noted that the calcination is important to activate the recovered catalyst. Without calcination, the reaction yield decreased to 74% in the second run.

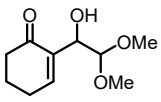
10. The effect of AcOH as a byproduct in the model reaction (Figure S3)

The byproduct AcOH could catalyze the model reaction to occur albeit in a poor yield (35% in table 1, entry 5). To evaluate the effect of AcOH as a cocatalyst, one equivalent of AcOH was added into the model reaction in the standard reaction conditions and the kinetics was studied. As shown in the following figure, the initial reaction rate didn't change with the addition of AcOH, but the final yield slightly decreased (73% *vs* 83%) indicative of the slightly negative effect of AcOH as a cocatalyst.



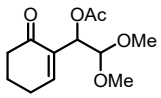
11. Characterization data of products

2-(1-hydroxy-2,2-dimethoxyethyl)cyclohex-2-en-1-one (1aa)



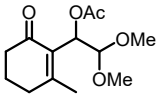
The crude mixture was purified by silica column chromatography (petroleum ether to petroleum ether/ethyl acetate = 5/1 v/v gradient elution) to yield the title compound as a colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 (t, *J* = 4.3 Hz, 1H), 4.39 (s, 2H), 3.35 (d, *J* = 11.1 Hz, 6H), 2.99 (s, 1H), 2.38 (dt, *J* = 8.5, 5.9 Hz, 4H), 1.94 (t, *J* = 6.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.75, 148.52, 136.82, 105.17, 70.74, 55.29, 55.25, 38.51, 25.87, 22.51. IR: v = 3660, 2937, 2826, 1740, 1661, 1432, 1365, 1231, 1140, 1057, 957, 969, 865, 771, 479 cm^{-1}. HRMS (ESI): m/z calcd for $C_{14}H_{18}O_5$, $[M + Na]^+$: 223.0941, found: 223.0941.

2,2-dimethoxy-1-(6-oxocyclohex-1-en-1-yl)ethyl acetate (1a)



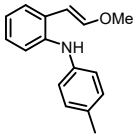
The crude mixture was purified by silica column chromatography using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 10/1) to yield the title compound as a colorless liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.96 (d, *J* = 4.3 Hz, 1H), 5.66 (d, *J* = 5.2 Hz, 1H), 4.56 (d, *J* = 5.2 Hz, 1H), 3.32 (s, 4H), 3.30 (s, 3H), 2.41 – 2.32 (m, 4H), 2.02 (s, 3H), 1.93 (p, *J* = 6.3 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.35, 169.66, 149.06, 135.01, 103.58, 69.84, 55.33, 54.98, 38.27, 25.82, 22.36, 21.01. IR: v = 3617, 2944, 2835, 1743, 1677, 1452, 1430, 1371, 1236, 1132, 1077, 1033, 974, 912, 865, 737, 462 cm^{-1}. HRMS (ESI): m/z calcd for $C_{12}H_{18}O_5$, $[M + Na]^+$: 265.1046, found: 265.1046.

2,2-dimethoxy-1-(2-methyl-6-oxocyclohex-1-en-1-yl)ethyl acetate (1b)



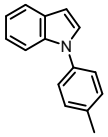
The 1b was prepared by the similar procedure as 1a from 1-methyl cyclohexenone. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 – 6.87 (m, 3H), 6.42 (d, *J* = 8.3 Hz, 2H), 4.58 (d, *J* = 4.0 Hz,1H), 4.43 (d, *J* = 3.9 Hz, 1H), 3.42 (s, 3H), 3.39 (s, 3H), 2.47 (td, *J* = 6.4, 2.4 Hz, 2H), 2.35 (p, *J* = 5.5 Hz, 2H), 2.21 (s, 3H), 1.97 (p, *J* = 6.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.55, 148.11, 144.52, 136.01, 129.63, 126.53, 113.35, 105.43, 55.58, 53.39, 38.54, 25.98, 22.72, 20.36. IR: v = 3400, 2923, 2866, 2833, 1732, 1668, 1618, 1521, 1455, 1383, 1218, 1249, 1171, 1123, 1077, 983, 809, 771, 723, 646, 516, 495, 443 cm^{-1}. HRMS (ESI): m/z calcd for $C_{13}H_{20}O_5$, $[M + Na]^+$: 279.1203, found: 279.1203.

2-(2-methoxyvinyl)-*N*-(*p*-tolyl)aniline (4a)



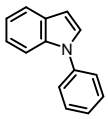
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 15/1) to yield the title compound as a brown liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 7.8 Hz, 2H), 7.18 (d, *J* = 7.1 Hz, 1H), 7.08 (dd, *J* = 11.2, 4.7 Hz, 3H), 6.90 (dd, *J* = 10.3, 7.4 Hz, 4H), 5.85 (d, *J* = 12.9 Hz, 1H), 3.67 (s, 3H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.43, 141.13, 140.77, 130.32, 129.83, 127.20, 127.01, 126.89, 121.51, 118.39, 117.94, 100.67, 56.64, 20.65. IR: v = 3372, 3030, 2922, 2854, 1737, 1719, 1638, 1611, 1515, 1465, 1372, 1300, 1217, 1154, 1088, 937, 810, 750, 503, 472 cm^{-1}. HRMS (ESI): m/z calcd for $C_{16}H_{17}NO$, $[M + Na]^+$: 262.1202, found: 262.1201.

1-(*p*-tolyl)-1*H*-indole[3] (3a)



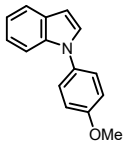
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.7 Hz, 1H), 7.44 (d, *J* = 8.1 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.25 – 7.18 (m, 3H), 7.10 (dt, *J* = 21.2, 7.2 Hz, 2H), 6.57 (d, *J* = 3.3 Hz, 1H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.33, 136.35, 136.02, 130.18, 129.21, 128.10, 124.36, 122.24, 121.09, 120.22, 110.55, 103.23, 21.10.

1-phenyl-1*H*-indole[4] (3b)



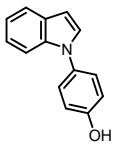
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a colorless oil;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.7 Hz, 1H), 7.53 – 7.40 (m, 5H), 7.33 – 7.25 (m, 2H), 7.18 – 7.08 (m, 2H), 6.61 (d, *J* = 3.2 Hz, 1H).^{13}C NMR (100 MHz, $CDCl_3$) δ 139.83, 135.85, 129.60, 129.30, 127.95, 126.44, 124.38, 122.33, 121.11, 120.34, 110.49, 103.55.

1-(4-methoxyphenyl)-1*H*-indole[5] (3c)



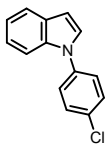
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a pale yellow oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.64 (d, *J* = 7.7 Hz, 1H), 7.55 (d, *J* = 3.2 Hz, 1H), 7.46 (dd, *J* = 18.0, 8.5 Hz, 3H), 7.21 – 7.02 (m, 4H), 6.66 (d, *J* = 3.2 Hz, 1H), 3.83 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 158.23, 135.95, 132.51, 129.21, 129.16, 125.94, 122.52, 121.27, 120.38, 115.36, 110.59, 103.24, 55.91.

4-(1*H*-indol-1-yl)phenol[6] (3d)



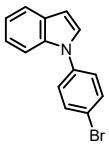
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 15/1) to yield the title compound as a orange oil; ^{1}H NMR (400 MHz, DMSO) δ 9.72 (s, 1H), 7.63 (d, *J* = 7.8 Hz, 1H), 7.52 (d, *J* = 3.2 Hz, 1H), 7.41 (d, *J* = 8.2 Hz, 1H), 7.35 (d, *J* = 8.4 Hz, 2H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.09 (t, *J* = 7.4 Hz, 1H), 6.95 (d, *J* = 8.3 Hz, 2H), 6.63 (d, *J* = 3.2 Hz, 1H).^{13}C NMR (100 MHz, $CDCl_3$) δ 154.22, 136.30, 133.01, 128.94, 128.27, 126.22, 122.17, 121.03, 120.10, 116.21, 110.35, 102.91.

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



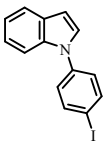
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a pale yellow oil;^{1}H NMR (400 MHz, DMSO) δ 7.65 (d, *J* = 13.0 Hz, 6H), 7.56 (d, *J* = 8.2 Hz, 1H), 7.21 (t, *J* = 7.3 Hz, 1H), 7.15 (t, *J* = 7.3 Hz, 1H), 6.73 (d, *J* = 3.1 Hz, 1H).^{13}C NMR (100 MHz, DMSO-d_6) δ 135.91 , 135.73 , 129.38 , 129.13 , 126.52 , 126.44 , 122.80 , 121.39 , 120.71 , 117.12 , 116.90 , 110.59 , 103.89.

1-(4-bromophenyl)-1*H*-indole[7] (3f)



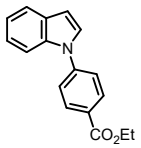
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a yellow oil; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.30 (d, J = 8.7 Hz, 2H), 7.24 – 7.18 (m, 2H), 7.12 (d, J = 8.8 Hz, 3H), 6.76 (t, J = 7.4 Hz, 1H), 6.69 (t, J = 7.3 Hz, 1H), 6.27 (d, J = 3.1 Hz, 1H), 2.92 (s, 3H).^{13}C NMR (100 MHz, DMSO-d_6) δ 158.23 , 135.95 , 132.51 , 129.21 , 129.16 , 125.95 , 122.52 , 121.27 , 120.38 , 115.36 , 110.59 , 103.24 , 55.91.

1-(4- iodophenyl) -1*H*-indole[8] (3g)



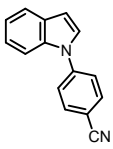
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, J = 8.0 Hz, 2H), 7.61 (d, J = 7.7 Hz, 1H), 7.45 (d, J = 8.2 Hz, 1H), 7.23 – 7.07 (m, 5H), 6.62 (d, J = 3.3 Hz, 1H).^{13}C NMR (100 MHz, $CDCl_3$) δ 139.56, 138.74, 135.58, 129.45, 127.56, 126.06, 122.67, 121.30, 120.67, 110.32, 104.24, 90.64.

ethyl 4-(1*H*-indol-1-yl)benzoate[9] (3h)



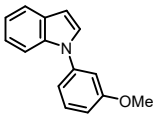
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 17/1) to yield the title compound as a colorless oil;^{1}H NMR (400 MHz, DMSO-d_6) δ 7.69 (d, J = 8.3 Hz, 2H), 7.36 – 7.28 (m, 3H), 7.23 (dd, J = 8.1, 3.4 Hz, 2H), 6.79 (t, J = 7.4 Hz, 1H), 6.72 (t, J = 7.5 Hz, 1H), 6.32 (d, J = 3.4 Hz, 1H), 3.90 (q, J = 7.1 Hz, 2H), 0.90 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 165.59, 143.46, 135.15, 131.36, 130.04, 128.65, 127.51, 123.56, 123.25, 121.63, 121.32, 111.10, 105.27, 61.30, 14.66.

4-(1*H*-indol-1-yl)benzonitrile[3] (3i)



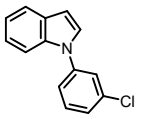
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 15/1) to yield the title compound as a yellow oil;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, J = 7.7 Hz, 1H), 7.55 (t, J = 8.7 Hz, 3H), 7.27 (d, J = 3.4 Hz, 1H), 7.24 – 7.12 (m, 2H), 6.68 (d, J = 3.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.59, 135.22, 133.81, 129.96, 127.08, 123.88, 123.24, 121.61, 121.39, 118.44, 110.36, 109.35, 105.76.

1-(3-methoxyphenyl)-1*H*-indole[3] (3j)



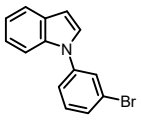
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a brown liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, J = 7.7 Hz, 1H), 7.53 (d, J = 8.2 Hz, 1H), 7.34 (t, J = 8.1 Hz, 1H), 7.27 (d, J = 3.3 Hz, 1H), 6.82 (dd, J = 8.4, 2.5 Hz, 1H), 6.60 (d, J = 3.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.56, 140.97, 135.80, 130.34, 129.36, 127.93, 122.38, 121.14, 120.39, 116.59, 112.00, 110.65, 110.23, 103.59, 55.50.

1-(3-chlorophenyl)-1*H*-indole[10] (3k)



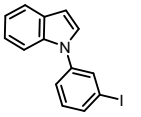
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a brown oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.7 Hz, 1H), 7.48 (d, *J* = 8.2 Hz, 1H), 7.43 (d, *J* = 2.1 Hz, 1H), 7.39 – 7.27 (m, 2H), 7.27 – 7.20 (m, 2H), 7.19 – 7.07 (m, 2H), 6.61 (d, *J* = 3.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.98, 135.65, 135.24, 130.64, 129.48, 127.61, 126.49, 124.39, 122.74, 122.30, 121.32, 120.76, 110.37, 104.36.

1-(3-bromophenyl)-1*H*-indole[11] (3l)



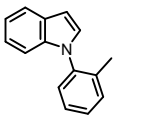
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.8 Hz, 2H), 7.48 (d, *J* = 8.2 Hz, 1H), 7.44 – 7.34 (m, 2H), 7.29 (t, *J* = 8.0 Hz, 1H), 7.22 (d, *J* = 3.3 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.61 (d, *J* = 3.3 Hz, 1H).^{13}C NMR (100 MHz, $CDCl_3$) δ 141.09, 135.64, 130.89, 129.46, 129.41, 127.60, 127.29, 123.10, 122.79, 122.74, 121.30, 120.76, 110.34, 104.37.

1-(3- iodophenyl) -1*H*-indole (3m)



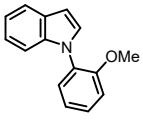
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (t, *J* = 1.9 Hz, 1H), 7.71 – 7.65 (m, 2H), 7.54 (d, *J* = 8.2 Hz, 1H), 7.48 (dd, *J* = 7.9, 2.1 Hz, 1H), 7.29 (d, *J* = 3.3 Hz, 1H), 7.27 – 7.11 (m, 3H), 6.68 (d, *J* = 3.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.96, 135.64, 135.41, 133.17, 131.04, 129.43, 127.62, 123.52, 122.72, 121.30, 120.74, 110.33, 104.31, 94.54. IR: v = 1588, 1516, 1491, 1455, 1334, 1310, 1233, 1211, 1133, 1101, 1072, 1009, 828, 762, 738, 722 cm^{-1}. HRMS (ESI): m/z calcd for $C_{14}H_{10}IN$, $[M + H]^+$: 319.9931, found: 319.9933.

1-(*o*-tolyl)-1*H*-indole[3] (3n)



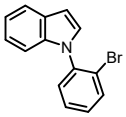
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.58 (m, 1H), 7.31 – 7.18 (m, 4H), 7.11 – 7.03 (m, 3H), 6.99 – 6.92 (m, 1H), 6.58 (d, *J* = 3.2 Hz, 1H), 1.98 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.31, 137.03, 135.89, 131.22, 128.70, 128.32, 128.25, 128.18, 126.81, 122.07, 120.89, 119.90, 110.57, 102.52, 17.70.

1-(2-methoxyphenyl)-1*H*-indole[5] (3o)



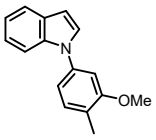
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.5 Hz, 1H), 7.36 (ddt, *J* = 9.7, 7.8, 3.2 Hz, 2H), 7.27 (d, *J* = 3.2 Hz, 1H), 7.16 (dqd, *J* = 20.8, 7.8, 7.0, 1.6 Hz, 3H), 7.05 (t, *J* = 7.8 Hz, 2H), 6.65 (d, *J* = 3.2 Hz, 1H), 3.73 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.54, 136.86, 129.39, 128.62, 128.56, 128.25, 128.15, 121.93, 120.91, 120.84, 119.96, 112.54, 110.91, 102.58, 55.79.

1-(2-bromophenyl)-1*H*-indole[11] (3p)



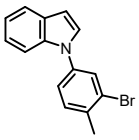
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.0 Hz, 1H), 7.73 – 7.65 (m, 1H), 7.44 (q, *J* = 5.2, 4.6 Hz, 2H), 7.33 (ddd, *J* = 8.6, 6.1, 3.0 Hz, 1H), 7.26 – 7.22 (m, 1H), 7.21 – 7.14 (m, 2H), 7.14 – 7.07 (m, 1H), 6.70 (d, *J* = 3.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.54, 136.78, 133.97, 129.77, 129.50, 128.73, 128.40, 128.32, 122.28, 122.02, 120.97, 120.30, 110.61, 103.14.

1-(3-methoxy-4-methylphenyl)-1*H*-indole (3q)



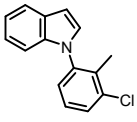
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a white oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.7 Hz, 1H), 7.48 (d, *J* = 8.1 Hz, 1H), 7.25 (d, *J* = 3.2 Hz, 1H), 7.20 – 7.05 (m, 3H), 6.94 – 6.85 (m, 2H), 6.59 (d, *J* = 3.2 Hz, 1H), 3.78 (s, 3H), 2.21 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.35, 138.60, 136.03, 131.09, 129.17, 128.16, 125.16, 122.24, 121.10, 120.21, 116.21, 110.58, 106.86, 103.14, 55.54, 15.96. IR: v = 3000, 2958, 2924, 2837, 1680, 1592, 1515, 1461, 1414, 1336, 1312, 1252, 1217, 1128, 1038, 855, 811, 742, 718, 644, 599, 427 cm^{-1}. HRMS (ESI): m/z calcd for $C_{16}H_{15}NO$, $[M + H]^+$: 238.1226, found: 284.1225.

1-(3-bromo-4-methylphenyl)-1*H*-indole (3r)



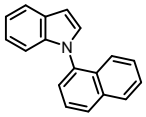
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a dark green oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 9.7 Hz, 2H), 7.44 (d, *J* = 8.2 Hz, 1H), 7.29 – 7.24 (m, 2H), 7.22 – 7.05 (m, 3H), 6.59 (d, *J* = 3.4 Hz, 1H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.61, 136.15, 135.80, 131.42, 129.31, 128.03, 127.75, 125.27, 123.13, 122.58, 121.23, 120.57, 110.35, 103.94, 22.53. IR: v = 3054, 3030, 2921, 2854, 1602, 1499, 1455, 1334, 1233, 1233, 1211, 1133, 1036, 882, 817, 763, 741, 693, 603, 586,427 cm^{-1}. HRMS (ESI): m/z calcd for C15H12BrN, $[M + H]^+$: 286.0226, found: 286.0225.

1-(3-chloro-2-methylphenyl)-1*H*-indole (3s)



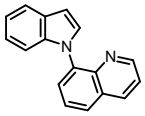
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 6.9 Hz, 1H), 7.40 (dd, *J* = 6.4, 2.9 Hz, 1H), 7.16 (q, *J* = 5.1, 4.6 Hz, 2H), 7.08 (ddd, *J* = 12.7, 7.9, 2.6 Hz, 3H), 6.94 (d, *J* = 8.9 Hz, 1H), 6.60 (d, *J* = 3.2 Hz, 1H), 1.99 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.61, 137.06, 135.84, 134.68, 129.15, 128.57, 128.32, 127.09, 126.76, 122.35, 120.99, 120.17, 110.46, 103.06, 15.39. IR: v = 3053, 2923, 2853, 1574, 1512, 1478, 1451, 1331, 1216, 1108, 1017, 960, 883, 791, 743, 711, 604, 427 cm^{-1}. HRMS (ESI): m/z calcd for $C_{15}H_{12}ClN$, $[M + H]^+$: 242.0731, found: 242.0730.

1-(naphthalen-1-yl)-1*H*-indole[12] (3t)



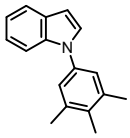
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a brown solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.2 Hz, 2H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.45 (dt, *J* = 12.5, 7.8 Hz, 3H), 7.37 (d, *J* = 8.4 Hz, 1H), 7.29 (dd, *J* = 19.7, 5.3 Hz, 2H), 7.13 – 7.02 (m, 2H), 6.94 (d, *J* = 8.1 Hz, 1H), 6.68 (d, *J* = 3.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.03, 136.09, 134.50, 130.60, 129.83, 128.50, 128.29, 126.98, 126.68, 126.68, 125.54, 125.18, 123.44, 122.17, 120.95, 120.15, 110.87, 102.94.

8-(1*H*-indol-1-yl)quinoline[13] (3u)



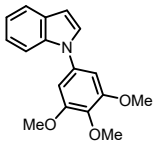
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a light brown solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.93 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.27 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.88 (td, *J* = 7.4, 1.5 Hz, 2H), 7.74 – 7.64 (m, 2H), 7.60 (d, *J* = 3.3 Hz, 1H), 7.47 (q, *J* = 8.3, 4.1 Hz, 1H), 7.30 – 7.24 (m, 1H), 7.19 – 7.13 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.83, 143.74, 137.44, 136.84, 136.27, 130.71, 129.61, 129.01, 127.27, 126.88, 126.23, 121.96, 121.84, 121.03, 120.20, 110.80, 103.04.

1-(3,4,5-trimethylphenyl)-1*H*-indole[12] (3v)



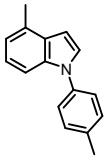
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.7 Hz, 1H), 7.46 (d, *J* = 8.1 Hz, 1H), 7.21 (d, *J* = 3.4 Hz, 1H), 7.15 – 7.03 (m, 4H), 6.55 (d, *J* = 3.4 Hz, 1H), 2.27 (s, 6H), 2.14 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.86, 136.90, 136.02, 133.59, 129.18, 128.18, 123.53, 122.09, 121.03, 120.08, 110.72, 102.94, 20.80, 15.21.

1-(3,4,5-trimethoxyphenyl)-1*H*-indole (3w)



The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.8 Hz, 1H), 7.55 (d, *J* = 8.2 Hz, 1H), 7.30 (d, *J* = 3.3 Hz, 1H), 7.26 – 7.20 (m, 1H), 7.16 (t, *J* = 7.6 Hz, 1H), 6.70 (s, 2H), 6.66 (d, *J* = 3.3 Hz, 1H), 3.91 (s, 3H), 3.88 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.85, 136.70, 136.07, 135.66, 129.17, 128.12, 122.40, 121.20, 120.36, 110.52, 103.34, 102.25, 61.07, 56.33. IR: v = 3100, 3055, 2964, 2939, 2830, 1596, 1510, 1461, 1421, 1281, 1232, 1128, 1010, 828, 763, 744, 646, 525, 428 cm^{-1}. HRMS (ESI): m/z calcd for $C_{17}H_{17}NO_3$, $[M + H]^+$: 284.1281, found: 284.1283.

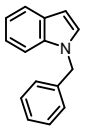
4-methyl-1-(*p*-tolyl)-1*H*-indole (3x)



The crude mixture was purified by silica column chromatography (petroleum ether to petroleum ether/ethyl acetate = 10/1 v/v gradient elution) to yield the title compound as a white oil with 78% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 3H), 7.23 (d, *J* = 7.5 Hz,3H), 7.05 (t, *J* = 7.7 Hz, 1H), 6.89 (d, *J* = 7.1 Hz, 1H), 6.61 (d, *J* =

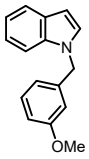
3.3 Hz, 1H), 2.52 (s, 3H), 2.36 (s, 3H). 13C NMR (100 MHz, CDCl3) δ 137.47, 136.28, 135.71, 130.50, 130.10, 128.96, 127.46, 124.35, 122.34, 120.35, 108.16, 101.62, 21.05, 18.76. IR: v = 3054, 2960, 2921, 1604, 1520, 1487, 1455, 1426, 1330, 1309, 1291, 1161, 1110, 1033, 925, 821, 751, 714, 550, 487, 446 cm-1. HRMS (ESI): m/z calcd for $C_{18}H_{15}NO_2$, $[M + H]^+$: 278.1176, found: 278.1174.

1-benzyl-1*H*-indole[14] (3y)



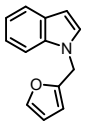
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.8 Hz, 1H), 7.19 (m, 5H), 7.13 – 6.97 (m, 5H), 6.48 (d, *J* = 3.2 Hz, 1H), 5.25 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.55, 136.31, 128.76, 128.72, 128.25, 127.59, 126.78, 121.68, 120.97, 119.52, 109.68, 101.69, 50.09.

1-(3-methoxybenzyl)-1*H*-indole[15] (3z)



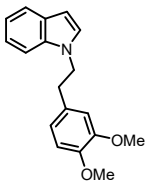
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.8 Hz, 1H), 7.28 (d, *J* = 8.2 Hz, 1H), 7.23 – 7.15 (m, 1H), 6.78 (d, *J* = 8.3, 4 Hz, 1H), 6.69 (d, *J* = 7.7 Hz, 1H), 6.65 (s, 1H), 6.55 (d, *J* = 3.2 Hz, 1H), 5.29 (s, 2H), 3.72 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.96, 139.18, 136.34, 129.81, 128.71, 128.24, 121.68, 120.96, 119.51, 119.08, 112.79, 112.64, 109.66, 101.71, 55.18, 50.03.

1-(furan-2-ylmethyl)-1*H*-indole[16] (3aa)



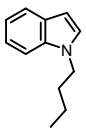
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a brown liquid;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.9 Hz, 1H), 7.34 (d, *J* = 8.2 Hz, 1H), 7.26 (s, 1H), 7.15 (m, 1H), 7.08 – 6.99 (m, 2H), 6.44 (d, *J* = 3.2 Hz, 1H), 6.22 (s, 1H), 6.14 (d, *J* = 3.2 Hz, 1H), 5.18 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.42, 142.56, 136.06, 128.70, 127.76, 121.72, 120.99, 119.60, 110.44, 109.42, 108.08, 101.81, 43.16

1-(3,4-dimethoxyphenethyl)-1*H*-indole[17] (3ab)



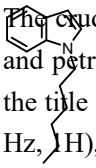
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.8 Hz, 1H), 7.32 (d, *J* = 8.2 Hz, 1H), 7.20 (t, *J* = 7.6 Hz, 1H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.89 (d, *J* = 3.2 Hz, 1H), 6.77 (d, *J* = 8.1 Hz, 1H), 6.65 (d, *J* = 8.1 Hz, 1H), 6.43 (d, *J* = 3.1 Hz, 1H), 6.35 (s, 1H), 4.33 (t, *J* = 7.1 Hz, 2H), 3.85 (s, 3H), 3.68 (s, 3H), 3.04 (t, *J* = 7.1 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.85, 147.77, 135.73, 131.17, 128.68, 128.05, 121.39, 120.97, 120.64, 119.29, 112.09, 111.28, 109.34, 100.91, 55.91, 55.71, 48.32, 36.21.

1-butyl-1*H*-indole[18] (3ac)



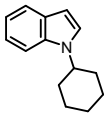
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a brown liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.9 Hz, 1H), 7.28 (d, *J* = 8.2 Hz, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.06 – 6.97 (m, 2H), 6.41 (d, *J* = 3.1 Hz, 1H), 4.05 (t, *J* = 7.1 Hz, 2H), 1.75 (p, *J* = 7.3 Hz, 2H), 1.27 (h, *J* = 7.4 Hz, 2H), 0.86 (t, *J* = 7.4 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 135.96, 128.56, 127.78, 121.26, 120.92, 119.13, 109.37, 100.81, 46.13, 32.34, 20.21, 13.71.

1-hexyl-1*H*-indole[19] (3ad)



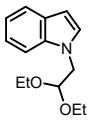
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a light-yellow viscous liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.9 Hz, 1H), 7.27 (d, *J* = 8.2 Hz, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 7.02 (q, *J* = 7.4, 5.9 Hz, 2H), 6.41 (d, *J* = 3.1 Hz, 1H), 4.04 (t, *J* = 7.2 Hz, 2H), 1.76 (t, *J* = 7.2 Hz, 2H), 1.23 (s, 8H), 0.81 (t, *J* = 6.6 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.91, 127.52, 126.73, 120.22, 119.88, 118.08, 108.33, 99.76, 45.39, 30.41, 29.19, 25.66, 21.51, 12.97.

1-cyclohexyl-1*H*-indole[20] (3ae)



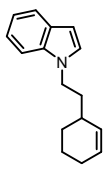
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a brown liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.8 Hz, 1H), 7.31 (d, *J* = 8.2 Hz, 1H), 7.19 – 7.10 (m, 2H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.43 (d, *J* = 3.2 Hz, 1H), 2.07 (d, *J* = 12.4 Hz, 2H), 1.86 (dd, *J* = 13.5, 4.5 Hz, 3H), 1.77 – 1.68 (m, 1H), 1.62 (m, 2H), 1.44 (m, 13.2, 3H), 1.22 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.47, 127.41, 122.98, 120.03, 119.89, 118.16, 108.38, 99.92, 54.04, 32.50, 24.95, 24.64.

1-(2,2-diethoxyethyl)-1*H*-indole (3af)



The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a white liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.9 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 1H), 7.21 (t, *J* = 7.7 Hz, 1H), 7.17 (d, *J* = 3.1 Hz, 1H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.49 (d, *J* = 3.2 Hz, 1H), 4.66 (t, *J* = 5.3 Hz, 1H), 4.22 (d, *J* = 5.3 Hz, 2H), 3.66 (q, *J* = 9.0, 7.1 Hz, 2H), 3.34 (q, *J* = 9.1, 7.1 Hz, 2H), 1.13 (t, *J* = 7.0 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.31, 128.79, 128.58, 121.43, 120.89, 119.38, 109.35, 101.67, 101.37, 63.72, 49.81, 15.25. IR: v = 3055, 2976, 2929, 1513, 1462, 1316, 1258, 1207, 1121, 1066, 884, 741, 675, 621, 473,427 cm^{-1}. HRMS (ESI): m/z calcd for $C_{14}H_{19}NO_2$, $[M + H]^+$: 234.1489, found: 234.1487.

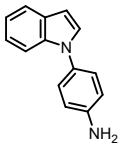
1-(2-(cyclohex-2-en-1-yl)ethyl)-1*H*-indole (3ag)



The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.9 Hz, 1H), 7.33 (d, *J* = 8.2 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.11 – 7.00 (m, 2H), 6.46 (d, *J* = 3.1 Hz, 1H), 5.37 (s, 1H), 4.15 (t, *J* = 7.5 Hz, 2H),

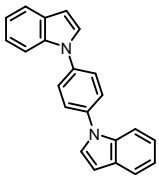
2.40 (t, *J* = 7.6 Hz, 2H), 1.94 – 1.89 (m, 4H), 1.60 (t, *J* = 2.7 Hz, 2H), 1.52 (m, 8.9, 4.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.92, 134.41, 128.63, 127.79, 123.83, 121.32, 120.98, 119.21, 109.42, 100.90, 45.46, 38.62, 28.52, 25.31, 22.97, 22.33. IR: v = 3363, 3043, 2926, 2858, 2663, 1624, 1510, 1405, 1361, 1172, 1172, 1043, 804 cm^{-1}. HRMS (ESI): m/z calcd for C16H19N, $[M + H]^+$: 226.1590, found: 226.1591.

4-(1*H*-indol-1-yl)aniline[21] (3ah)



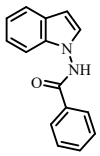
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 15/1) to yield the title compound as a brown solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.7 Hz, 1H), 7.36 (d, *J* = 8.1 Hz, 1H), 7.19 (dd, *J* = 6.1, 2.5 Hz, 3H), 7.14 – 7.04 (m, 2H), 6.72 (d, *J* = 8.6 Hz, 2H), 6.56 (d, *J* = 3.2 Hz, 1H), 3.70 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.28, 136.45, 130.94, 128.83, 128.44, 126.09, 121.96, 120.93, 119.90, 115.64, 110.49, 102.46.

1,4-di(1*H*-indol-1-yl)benzene[22] (3ai)



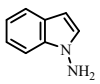
The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 18/1) to yield the title compound as a pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.8 Hz, 1H), 7.63 (d, *J* = 10.4 Hz, 3H), 7.39 (d, *J* = 3.3 Hz, 1H), 7.27 (t, *J* = 8.3, 7.0 Hz, 2H), 7.23 – 7.18 (m, 2H), 6.73 (d, *J* = 3.3 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.92, 134.83, 128.34, 126.79, 124.35, 121.57, 120.24, 119.56, 109.36, 102.98.

***N*-(1*H*-indol-1-yl)benzamide (5a)**



The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 16/1) to yield the title compound as a pale yellow solid; mp: 133 – 135 °C;^{1}H NMR (400 MHz, DMSO) δ 11.82 (s, 1H), 8.07 – 8.01 (d, *J* = 8.0 Hz, 2H), 7.68 (t, *J* = 7.4 Hz, 1H), 7.64 – 7.57 (m, 3H), 7.45 (d, *J* = 3.3 Hz, 1H), 7.28 (d, *J* = 8.1 Hz, 1H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.11 (t, *J* = 7.4 Hz, 1H), 6.59 – 6.53 (m, 1H). ^{13}C NMR (100 MHz, DMSO) δ 166.70, 136.17, 132.90, 132.30, 129.92, 129.19, 128.18, 126.49, 122.48, 121.13, 120.38, 109.44, 100.66. . HRMS (ESI): m/z calcd for $C_{15}H_{12}N_2O$, $[M + H]^+$: 237.1022, found: 237.1021. IR: v = 3434.77, 2256.10, 2129.18, 1659.79, 1050.38, 1027.36, 825.30, 763.87, 629.50 cm^{-1}. HRMS (ESI): m/z calcd for $C_{18}H_{15}NO_2$, $[M + H]^+$: 278.1176, found: 278.1174.

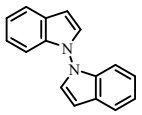
1*H*-indol-1-amine (5b)



The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 15/1) to yield the title compound as a pale yellow solid; mp: 40 – 42 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.9 Hz, 1H), 7.36 (d, *J* = 8.2 Hz, 1H), 7.23 (t, *J* = 7.6 Hz, 1H), 7.17 – 7.08 (m, 2H), 6.37 (d, *J* = 3.2 Hz, 1H), 4.68 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.70, 129.53, 126.56, 121.88, 120.99, 119.76, 108.34, 98.93. IR: v = 3331.44, 3053.37, 1459.43,

1325.15, 1325.15, 1325.15, 1231.63, 763.00, 743.97, 717.55 cm-1. HRMS (ESI): m/z calcd for $C_8H_8N_2$, $[M + H]^+$: 133.0760, found: 133.0763.

1,1'-biindole[23]



The crude mixture was purified by preparative TLC plate using a mixed solution of ethyl acetate and petroleum ether as eluting solvent (the ratio of petroleum ether/ethyl acetate is 20/1) to yield the title compound as a pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.65 (d, *J* = 8.0 Hz,2H), 7.32 (d, *J* = 3.4 Hz, 2H), 7.19 (pd, *J* = 7.1, 1.3 Hz, 4H), 6.89 (d, *J* = 7.8 Hz, 2H), 6.67 (d, *J* = 3.4 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.81, 127.97, 126.25, 123.27, 121.31, 121.22, 108.98, 102.26.

10. References

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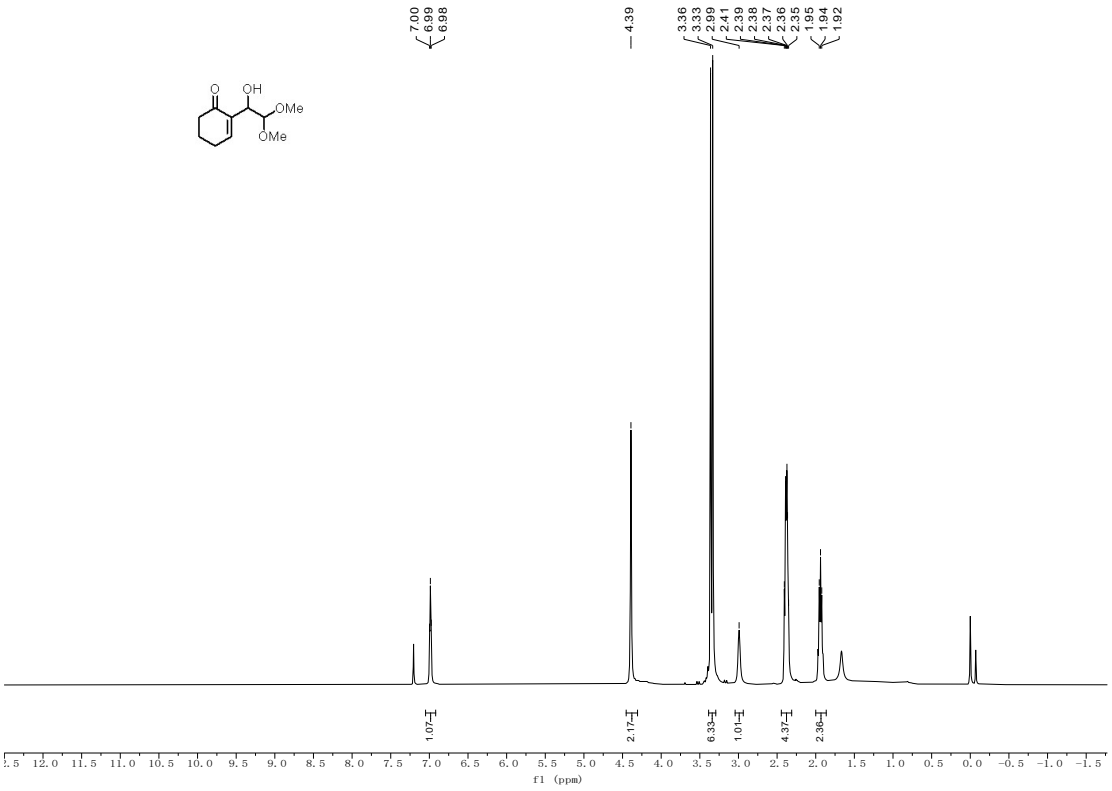
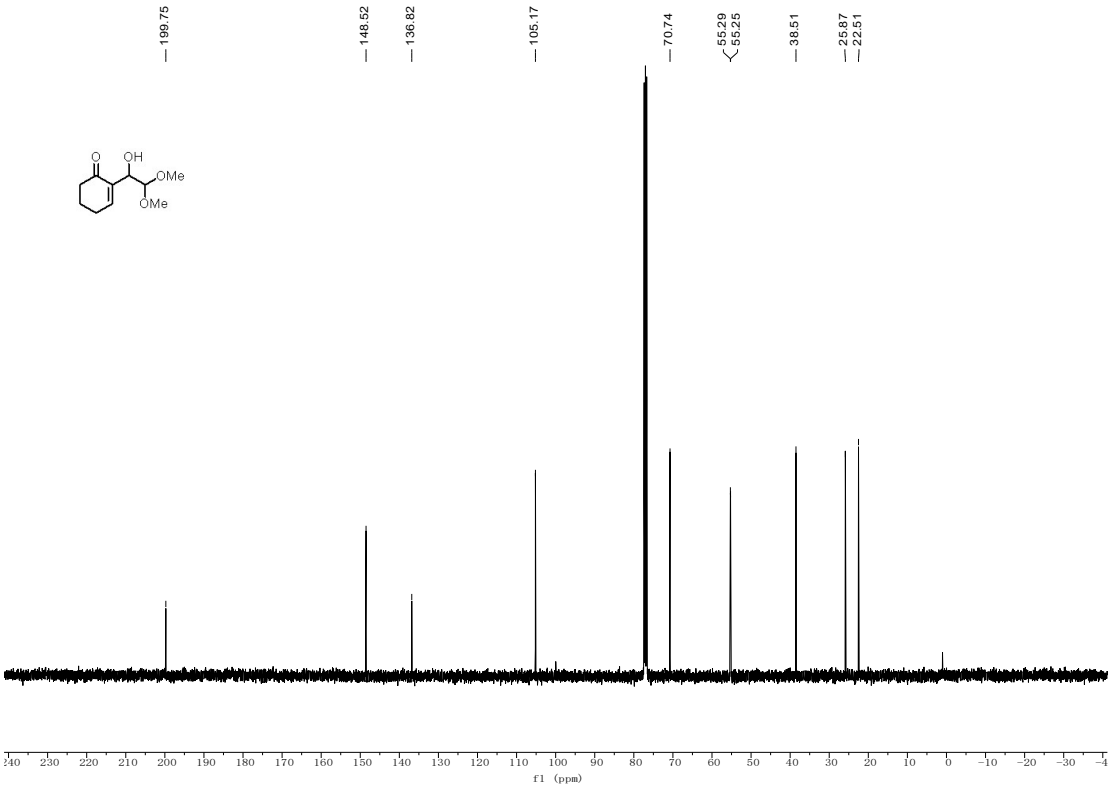
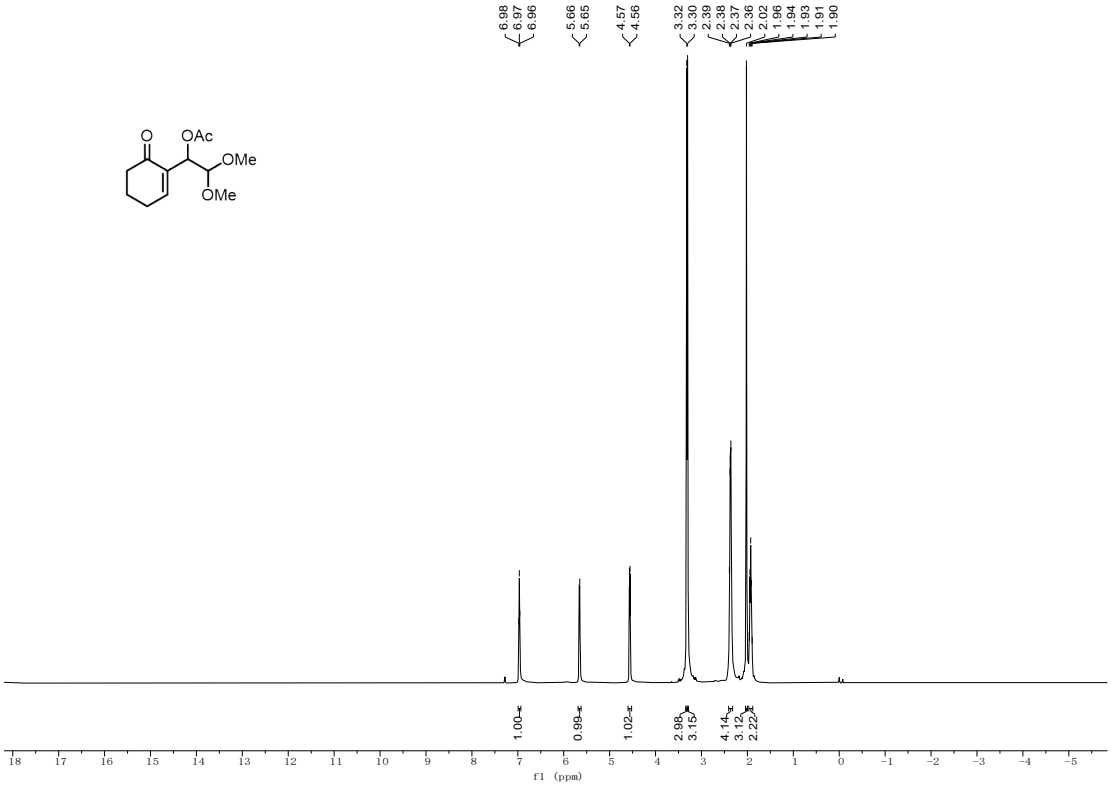
11. ^{1}H NMR and ^{13}C NMR spectra of products.

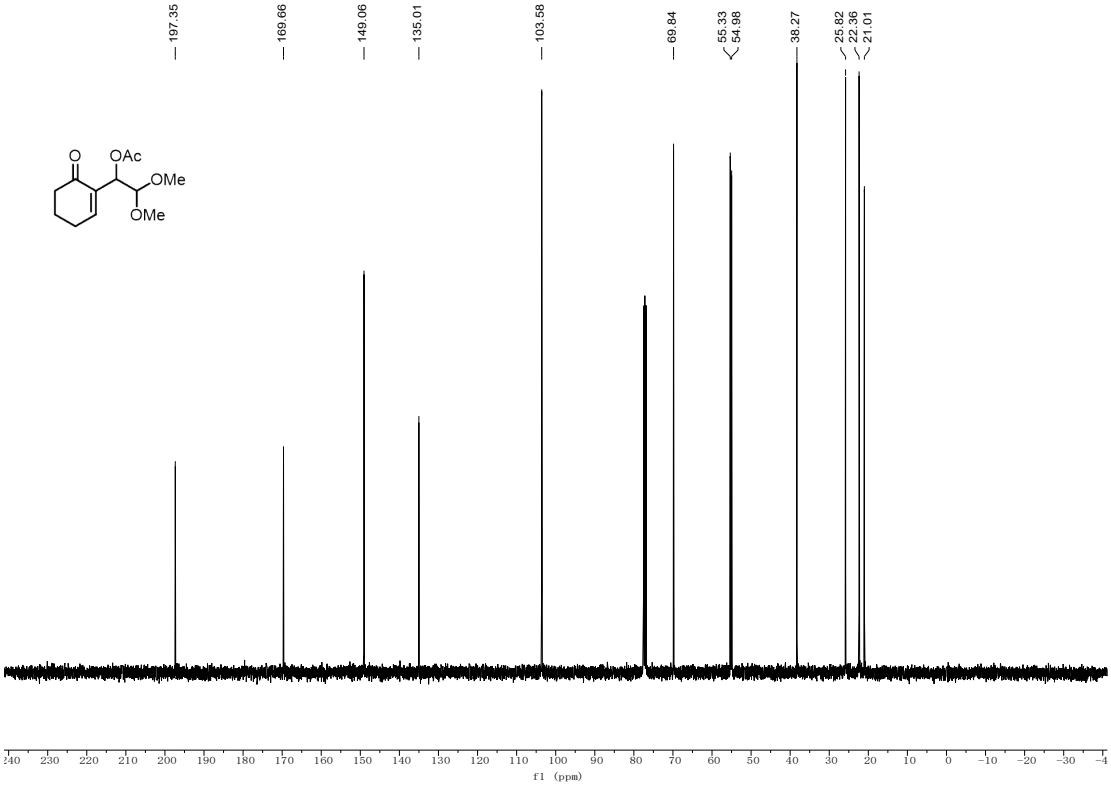
2-(1-hydroxy-2,2-dimethoxyethyl)cyclohex-2-en-1-one (1aa)



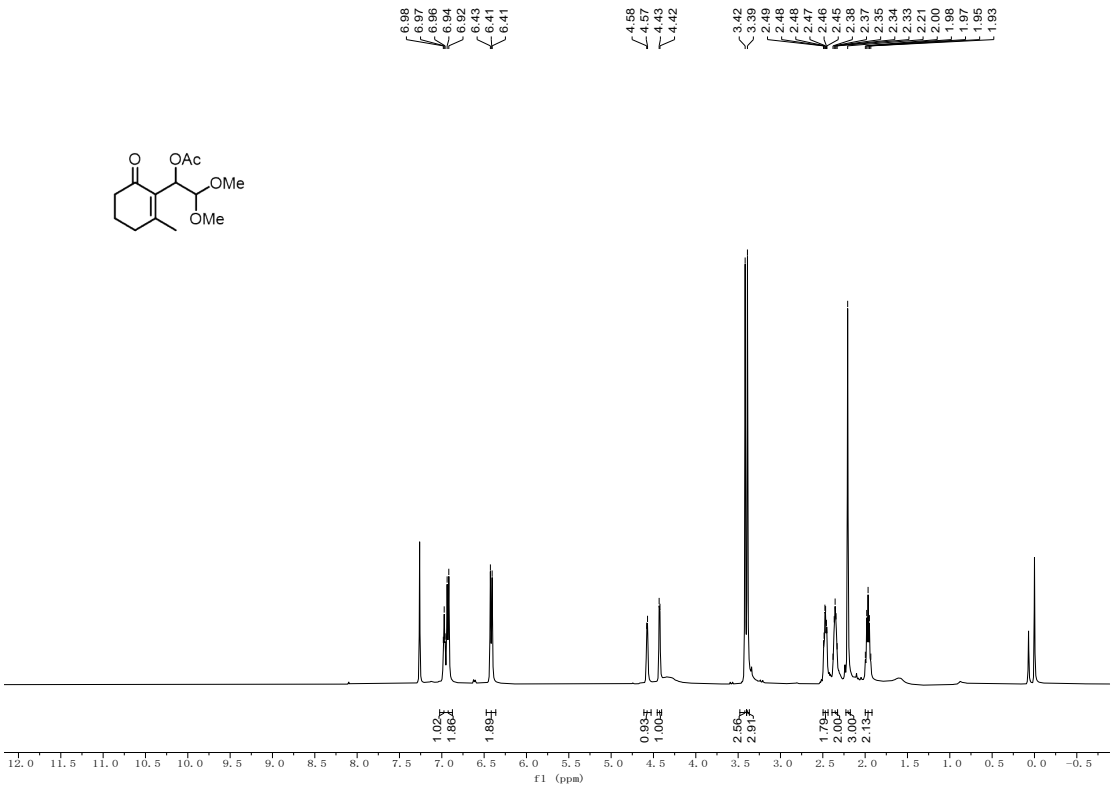


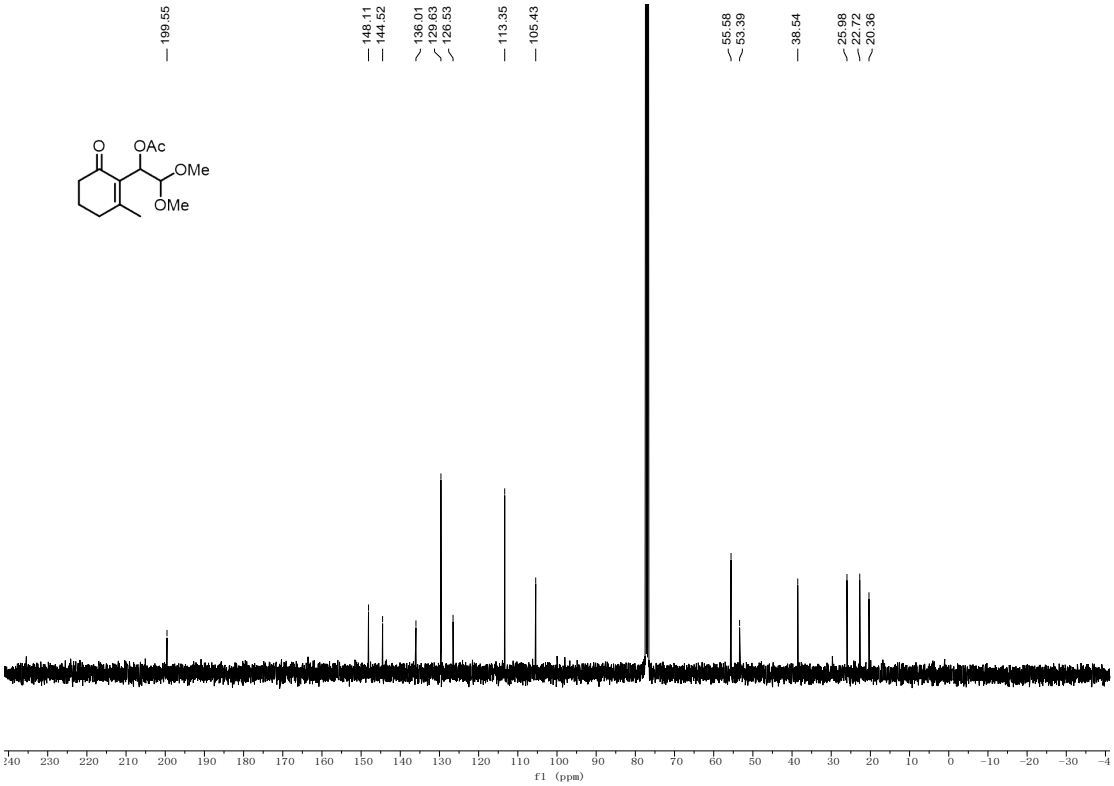
2,2-dimethoxy-1-(6-oxocyclohex-1-en-1-yl)ethyl acetate (1a)



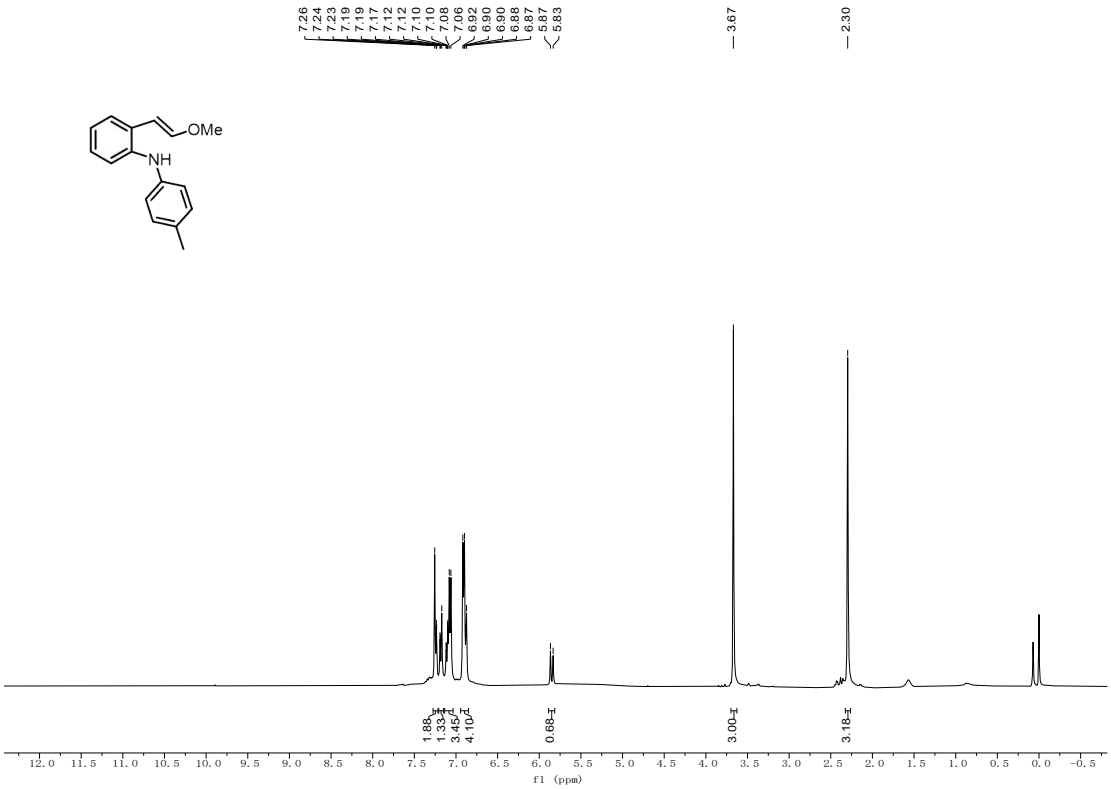


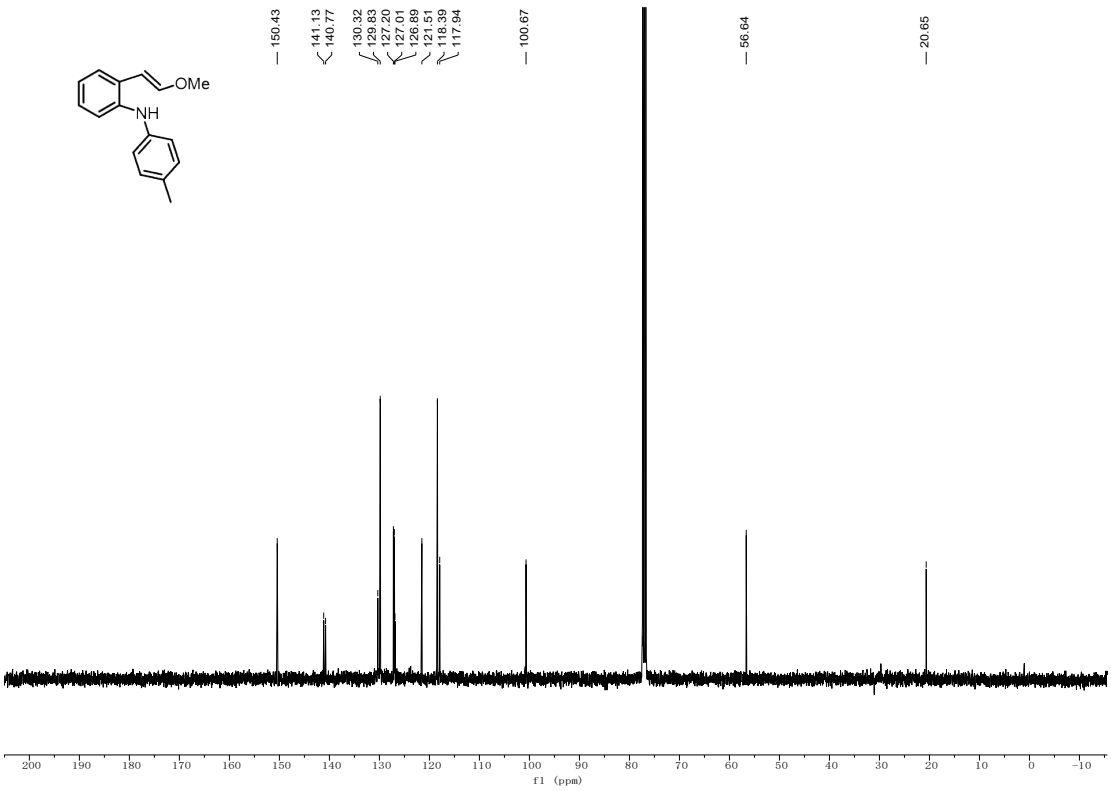
2,2-dimethoxy-1-(2-methyl-6-oxocyclohex-1-en-1-yl)ethyl acetate (1b)



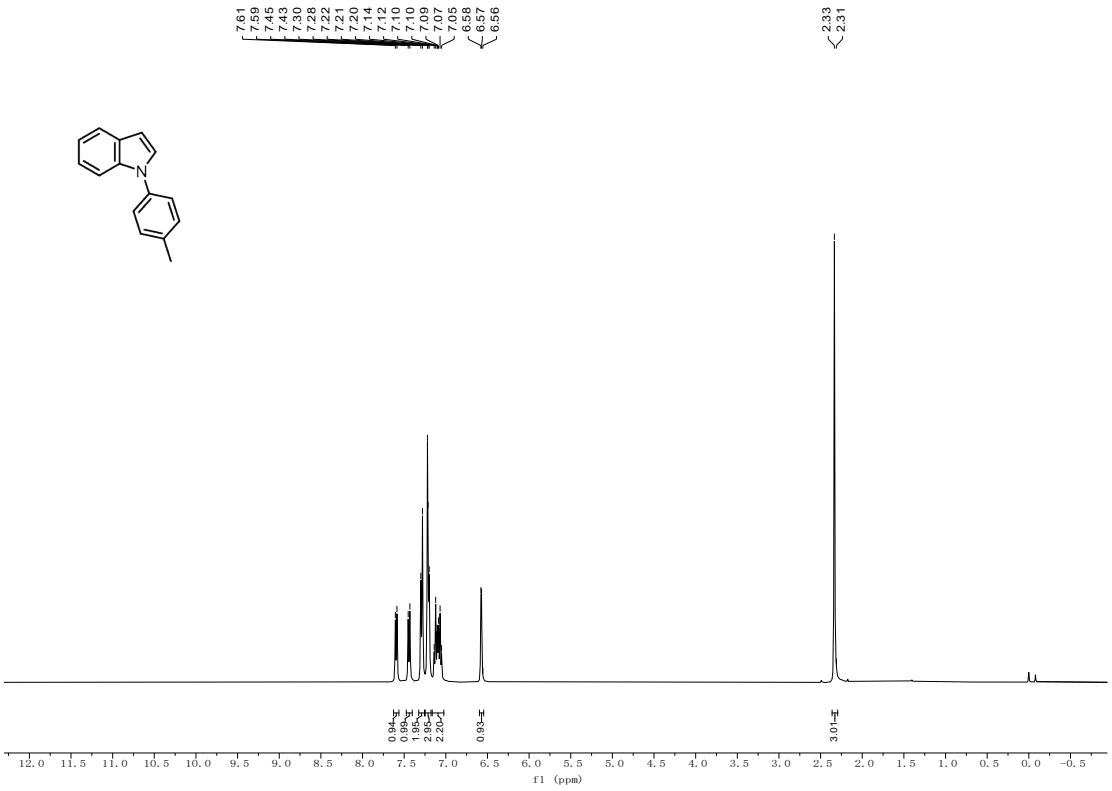


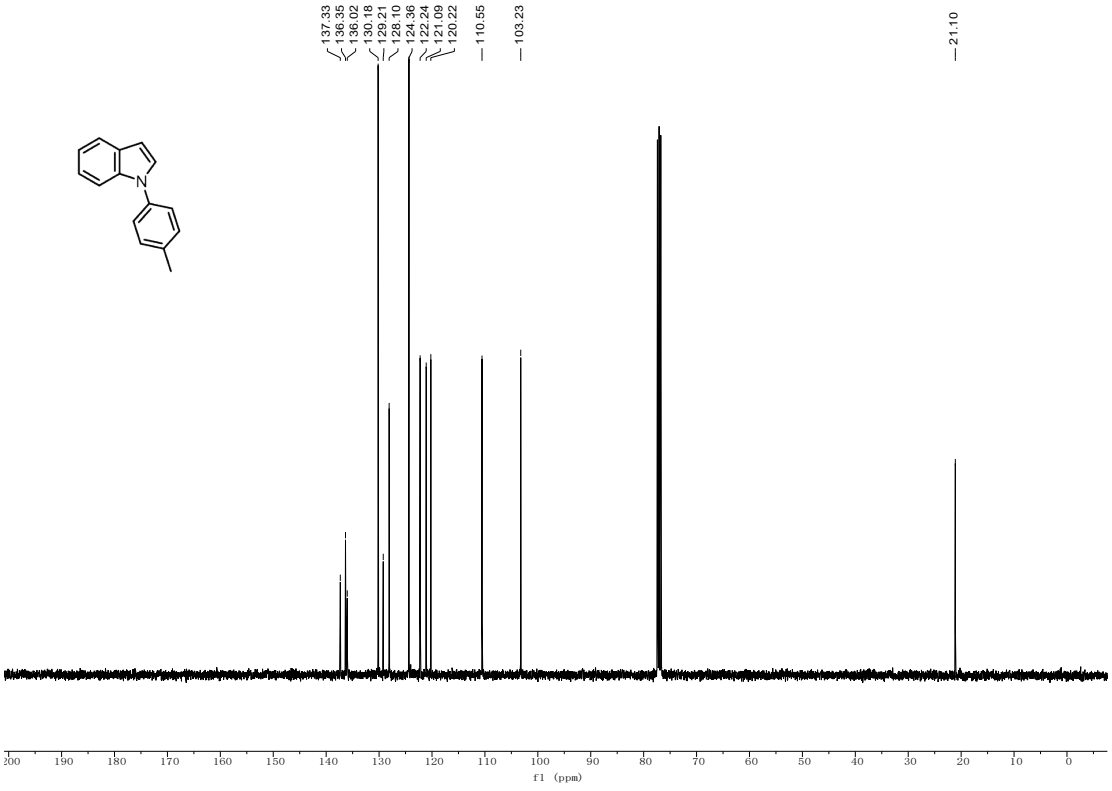
2-(2-methoxyvinyl)-*N*-(*p*-tolyl)aniline (4a)



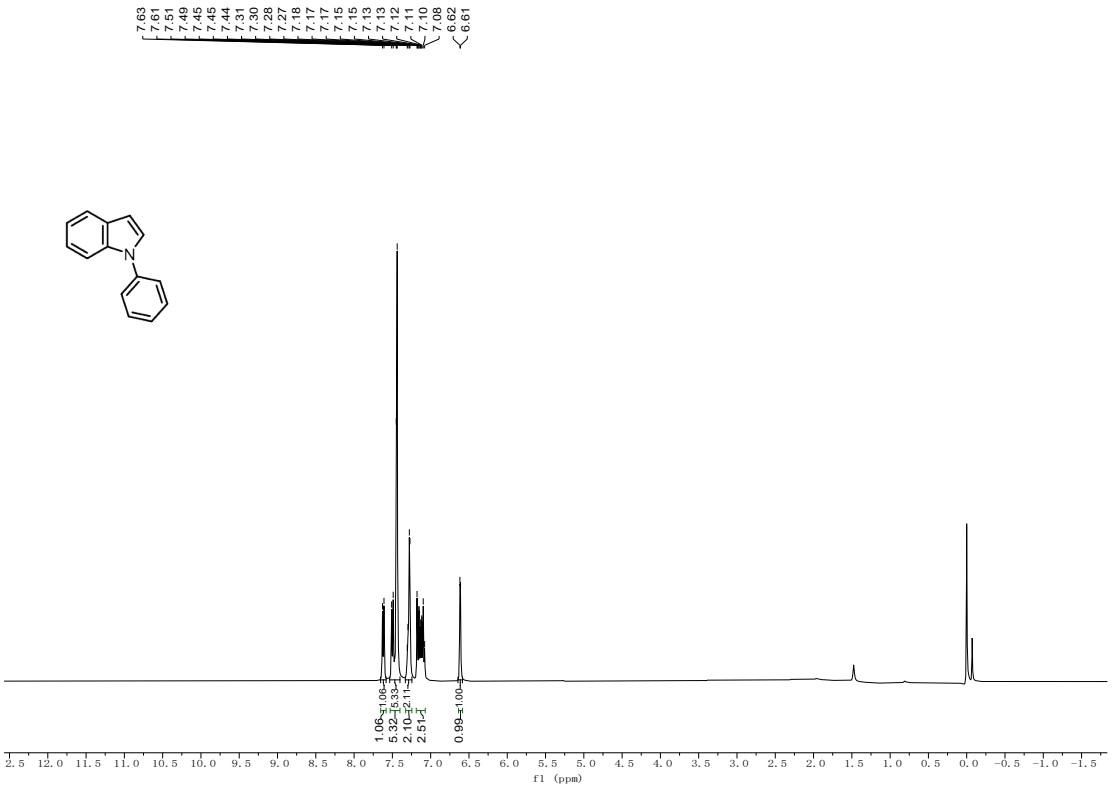


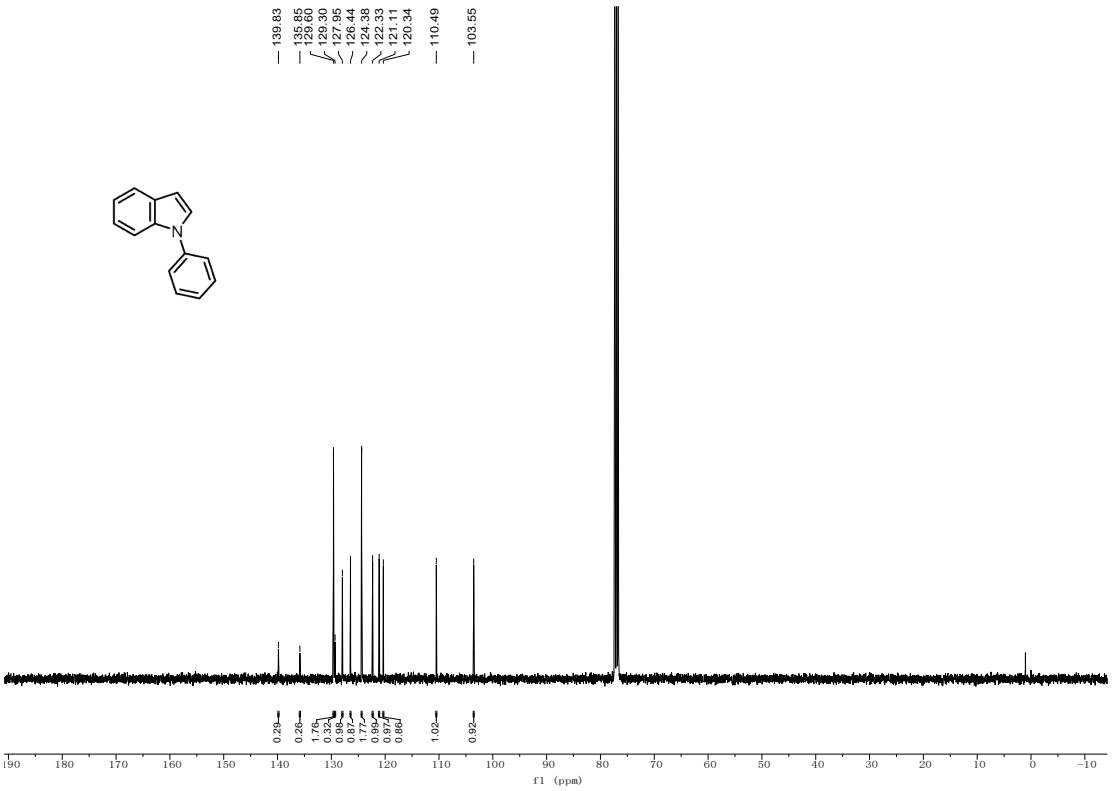
1-(*p*-tolyl)-1*H*-indole (3a)



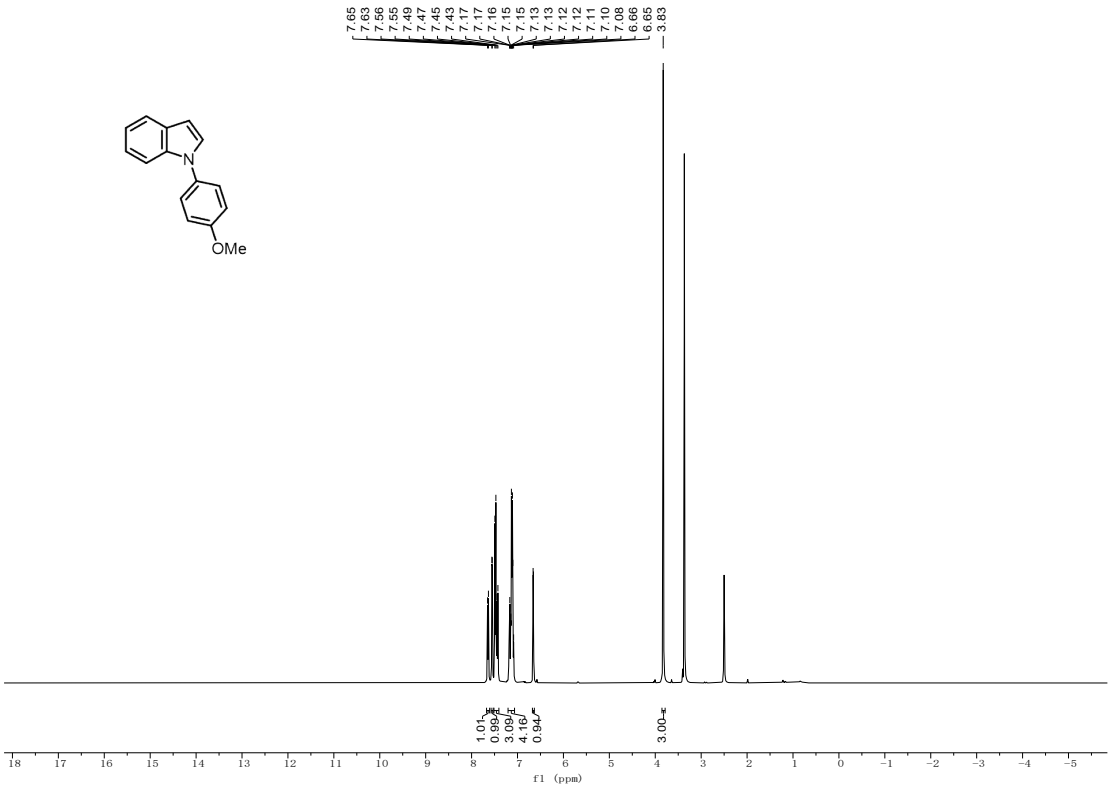


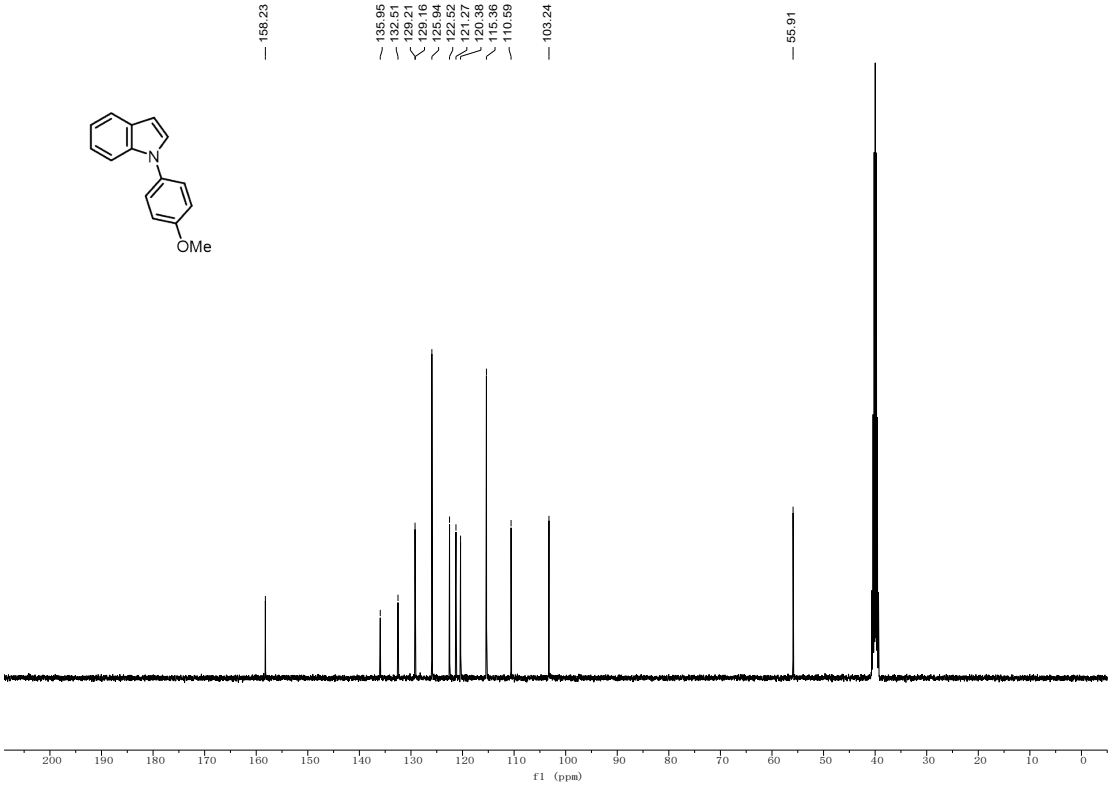
1-phenyl-1*H*-indole (3b)



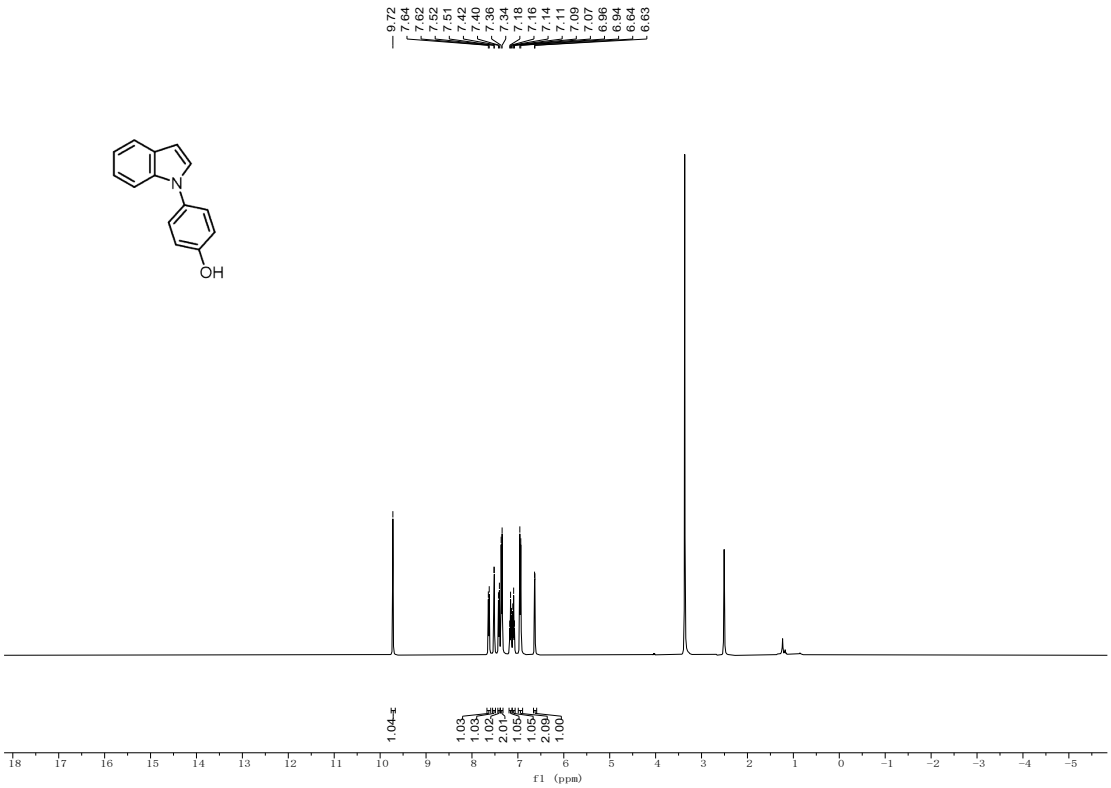


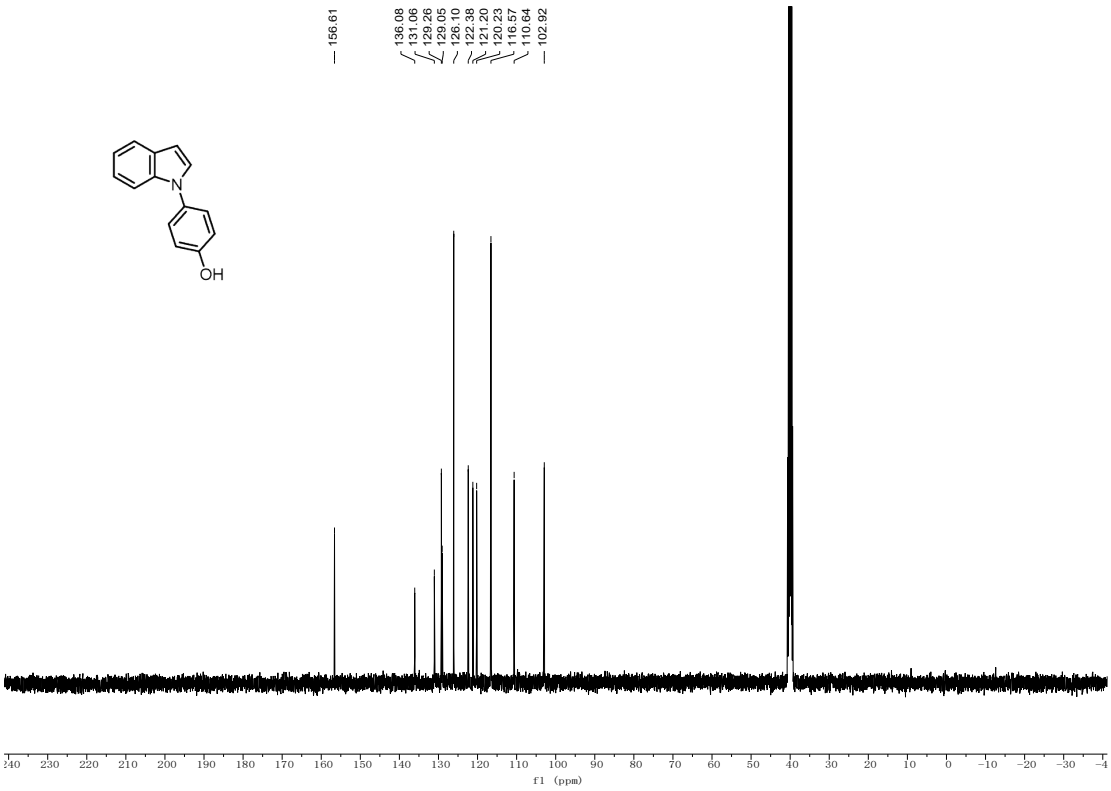
1-(4-methoxyphenyl)-1*H*-indole (3c)



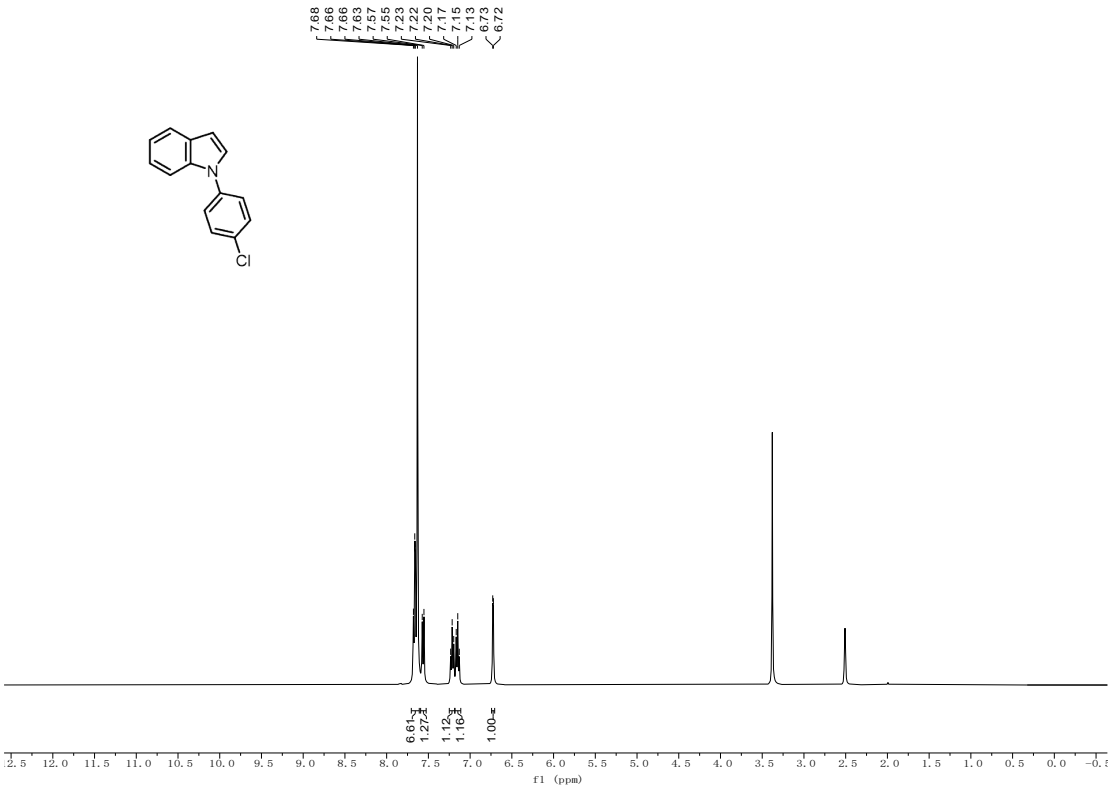


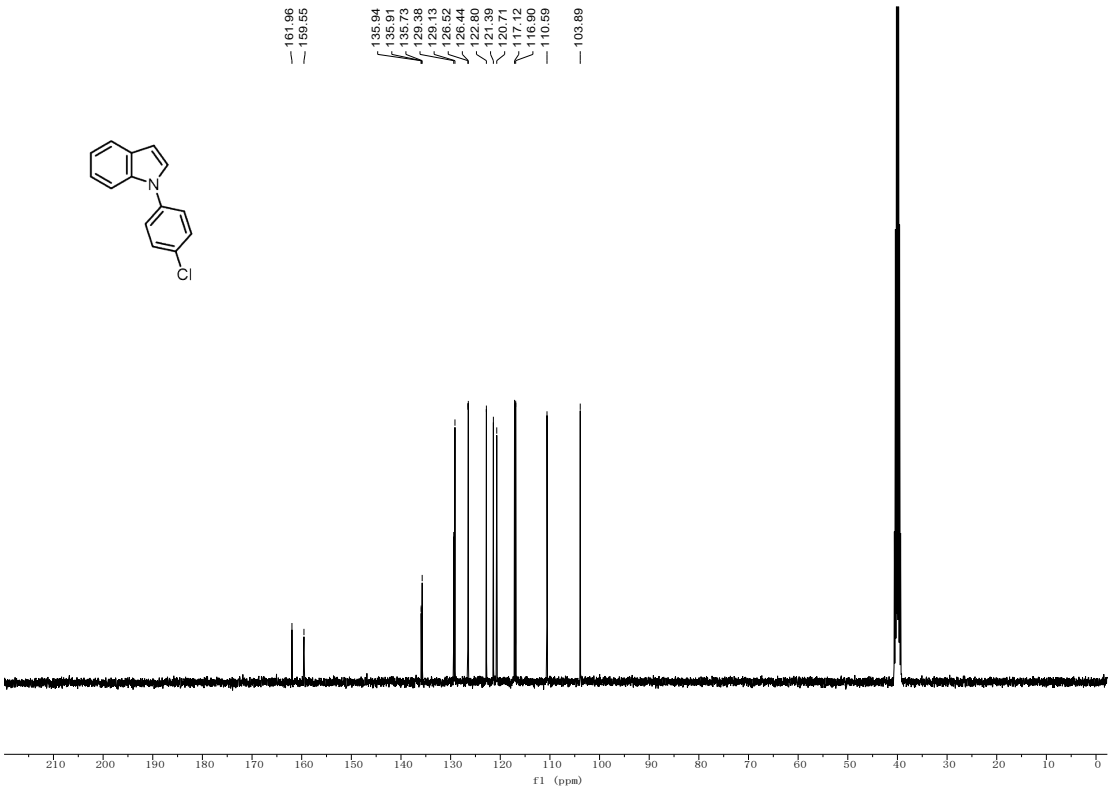
4-(1*H*-indol-1-yl)phenol (3d)



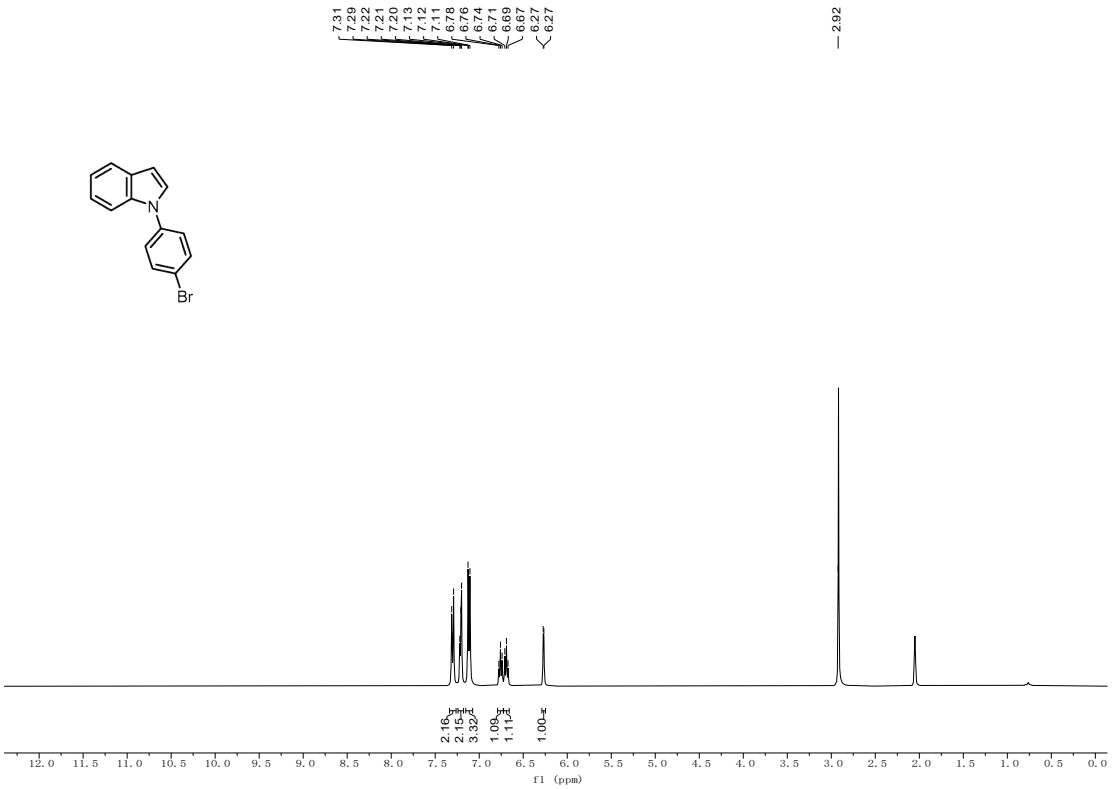


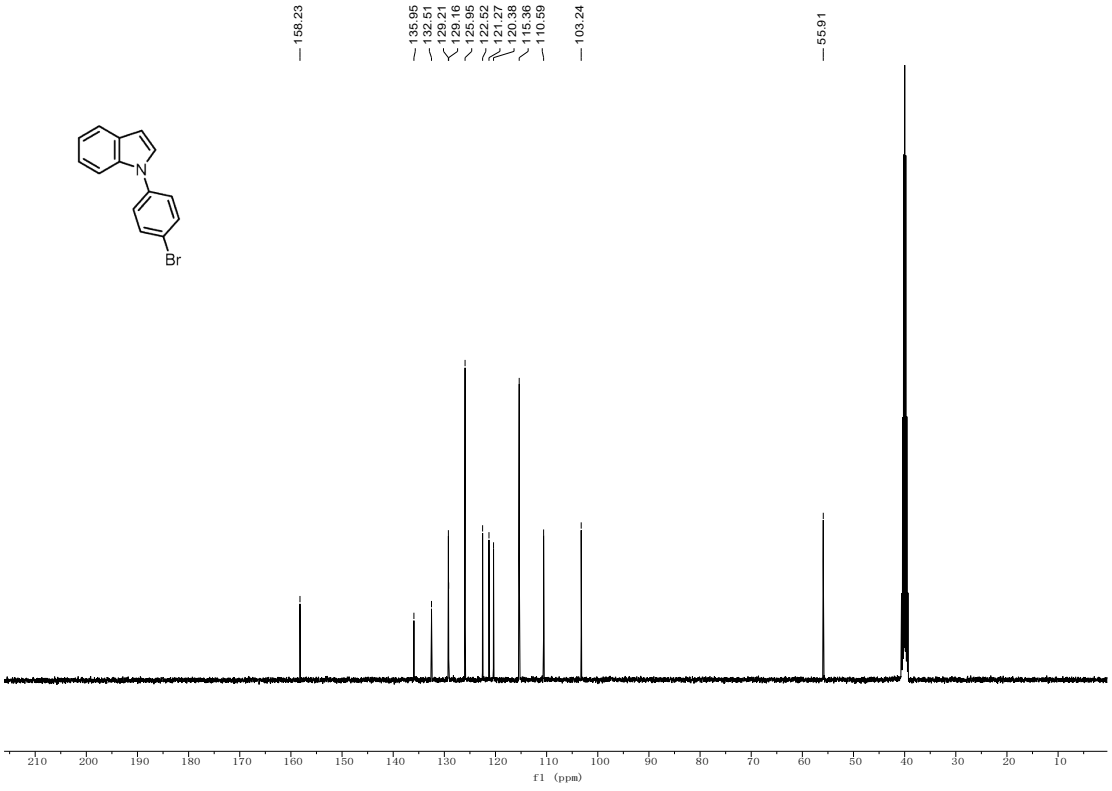
1-(4-chlorophenyl)-1*H*-indole (3e)



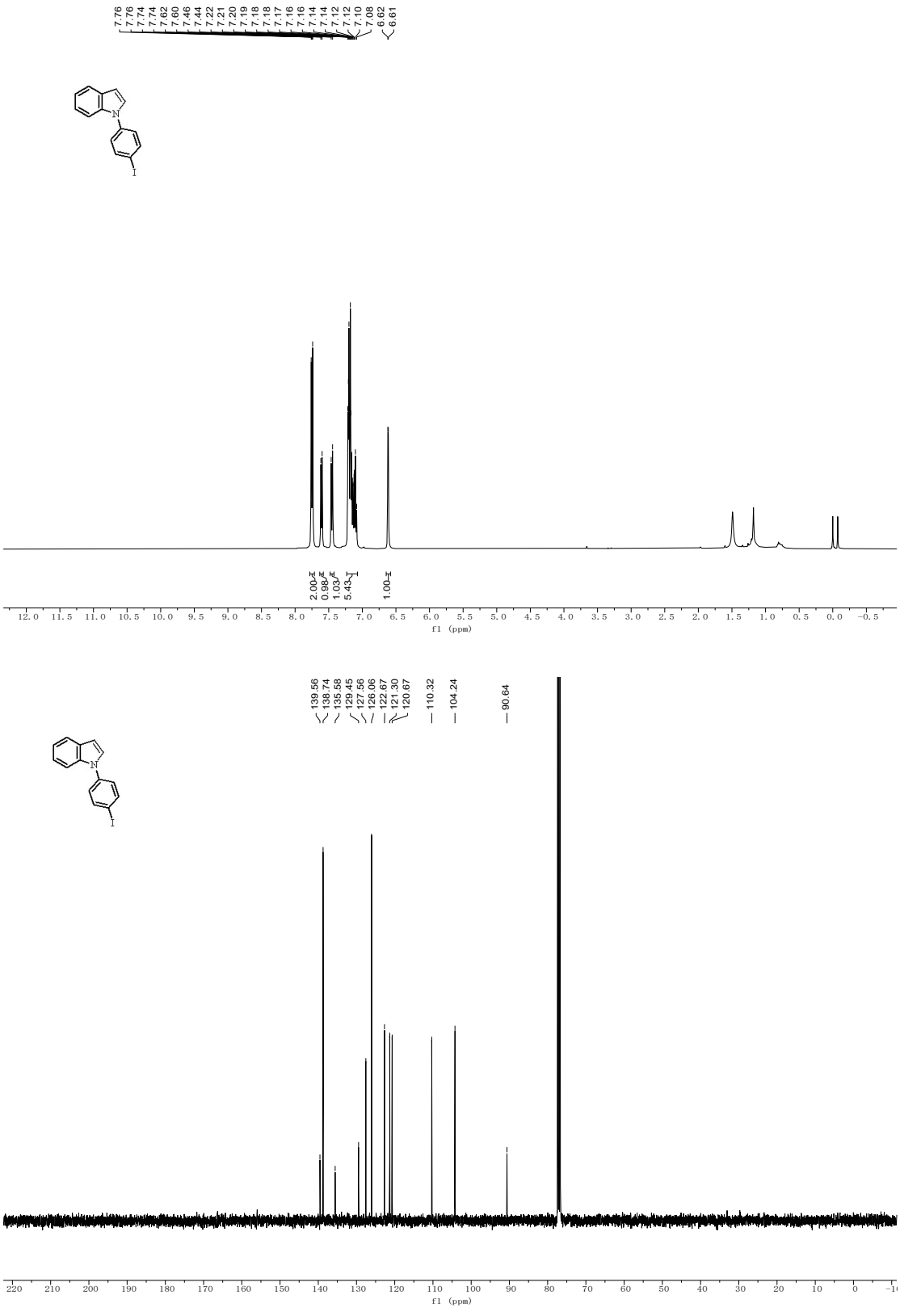


1-(4-bromophenyl)-1*H*-indole (3f)

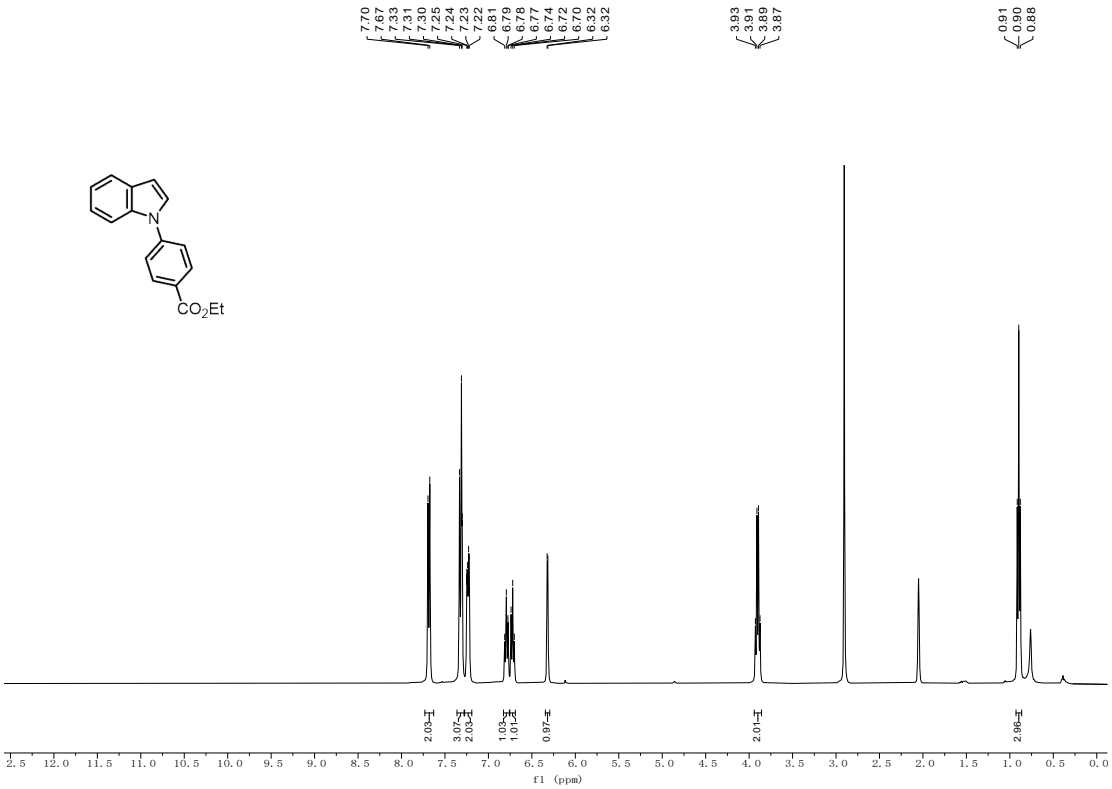


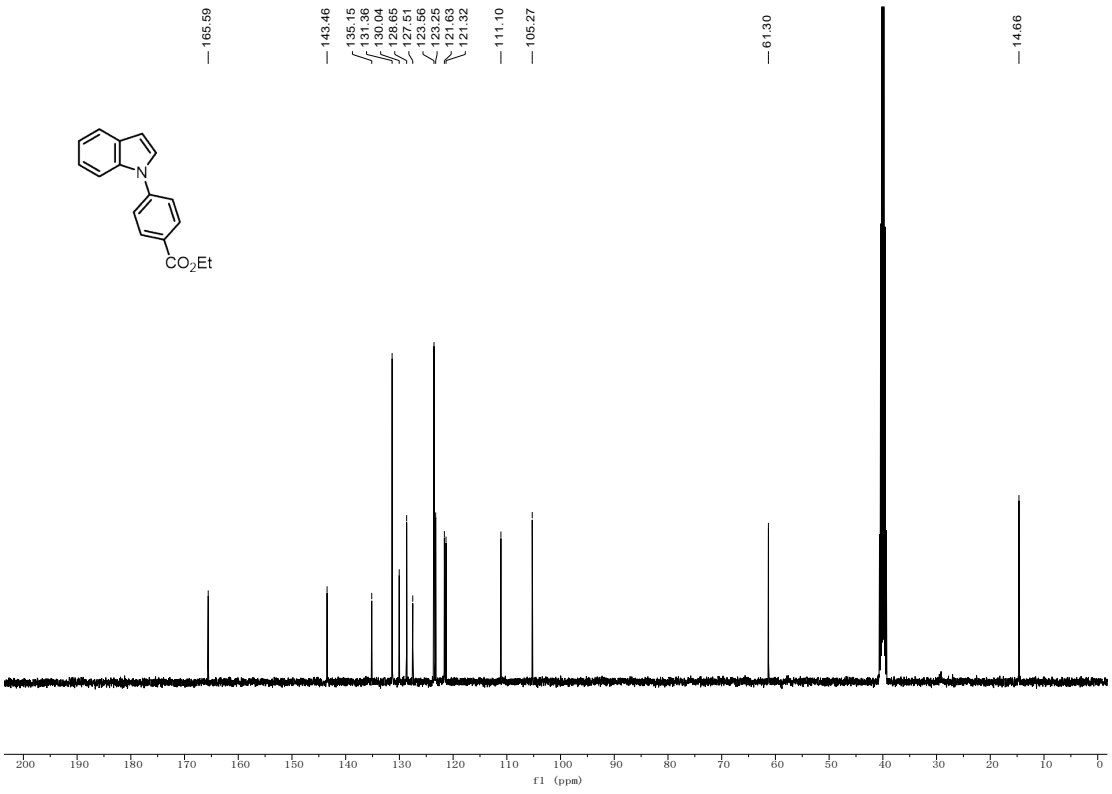


1-(4- iodophenyl)-1*H*-indole (3g)

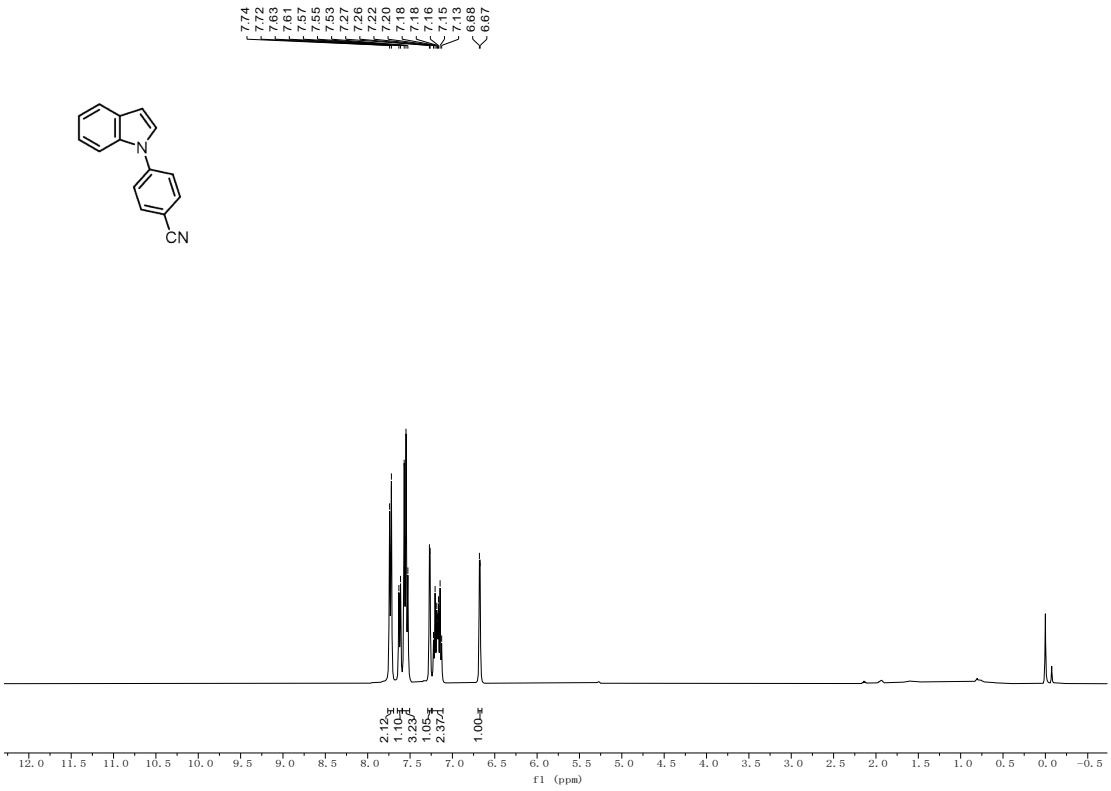


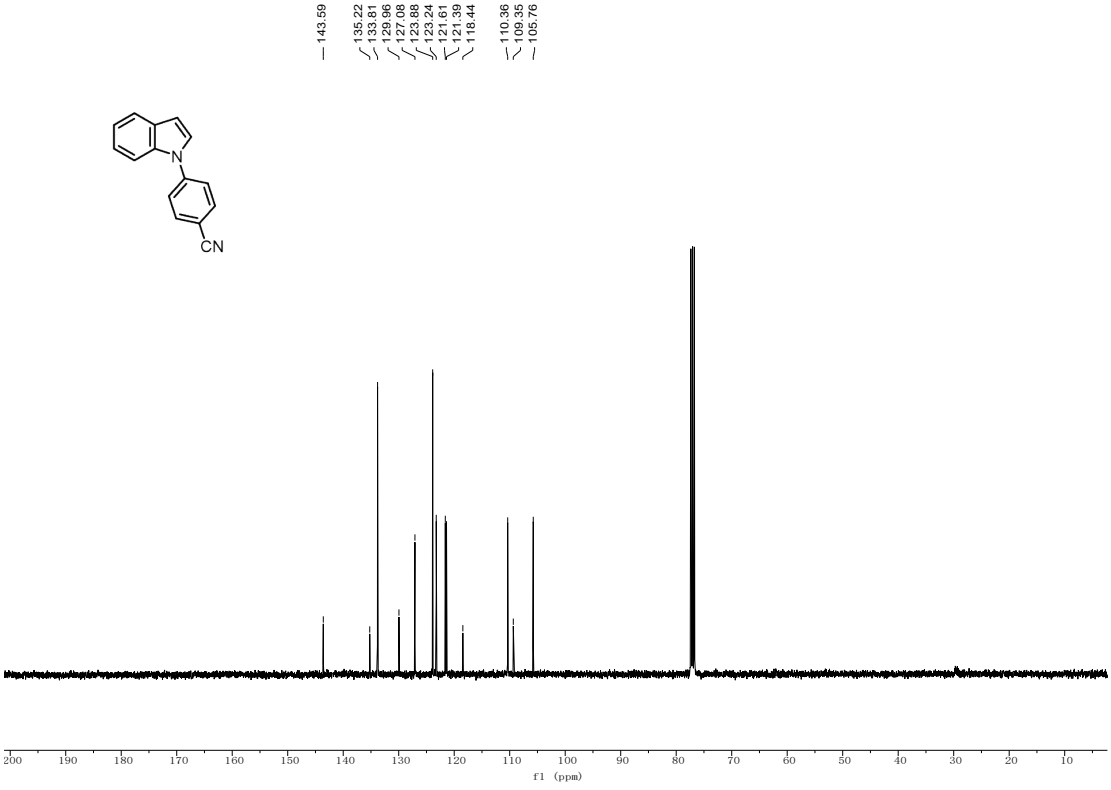
ethyl 4-(1*H*-indol-1-yl)benzoate (3h)



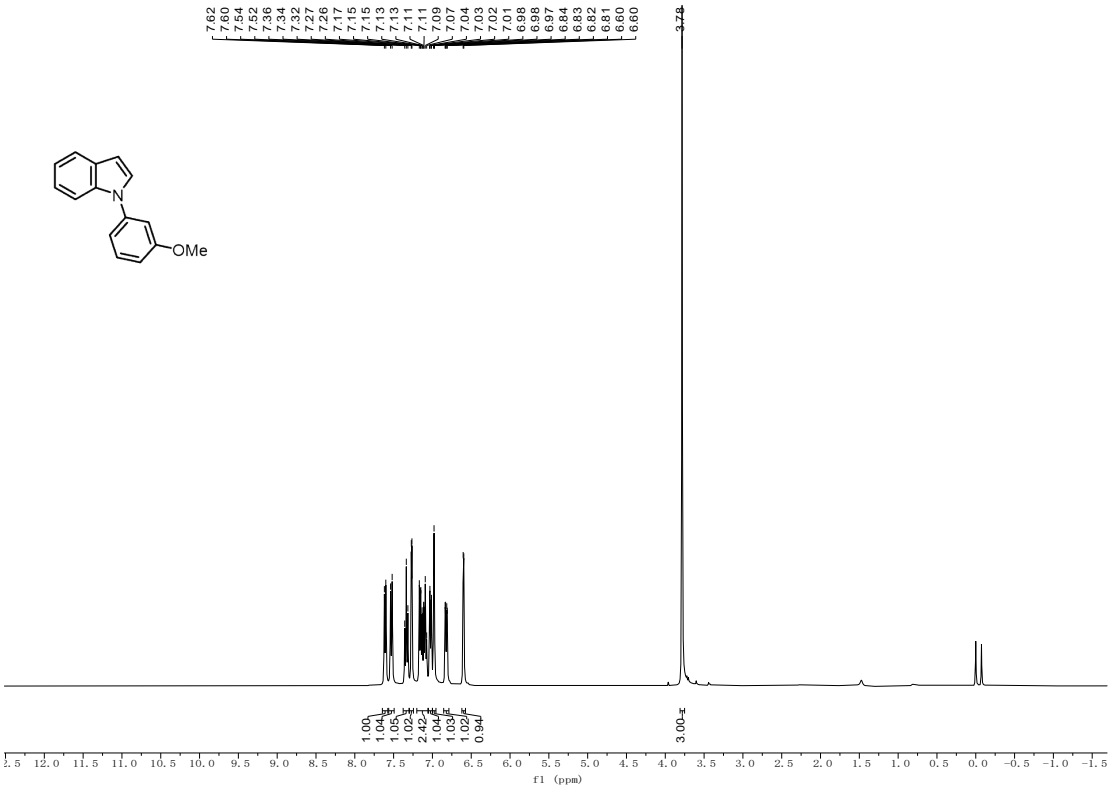


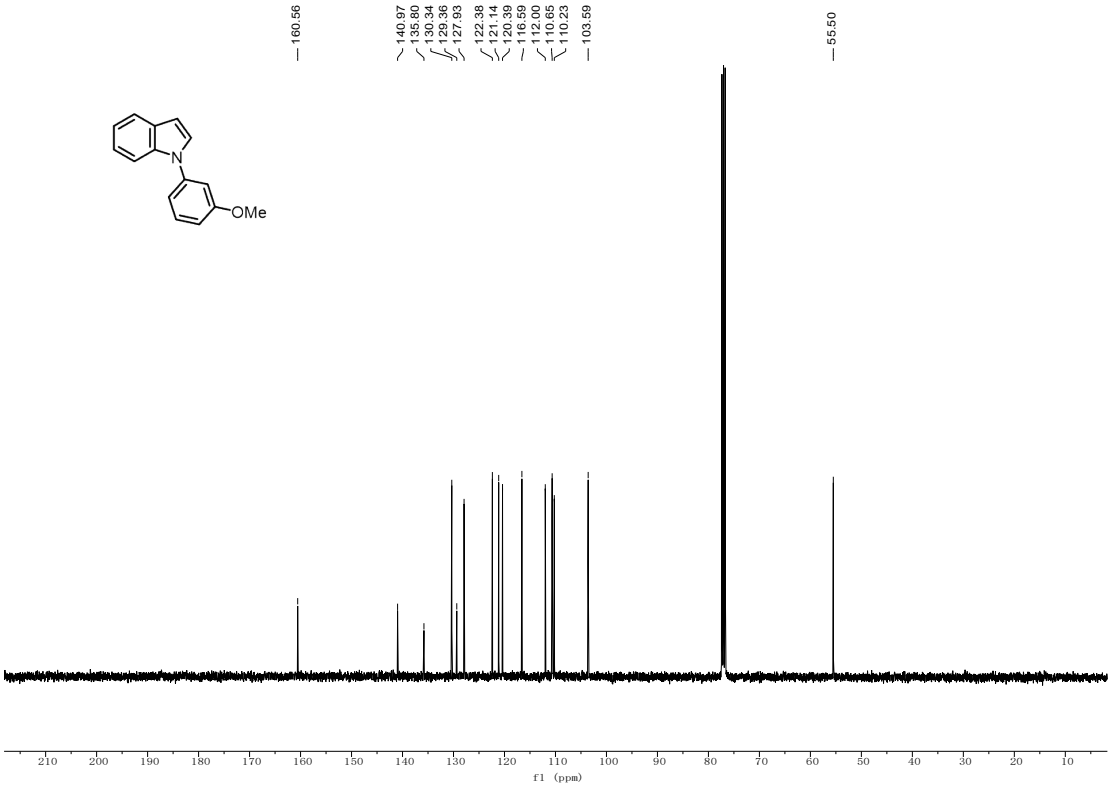
4-(1*H*-indol-1-yl)benzonitrile (3i)



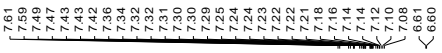


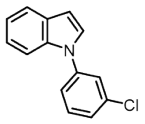
1-(3-methoxyphenyl)-1*H*-indole (3j)

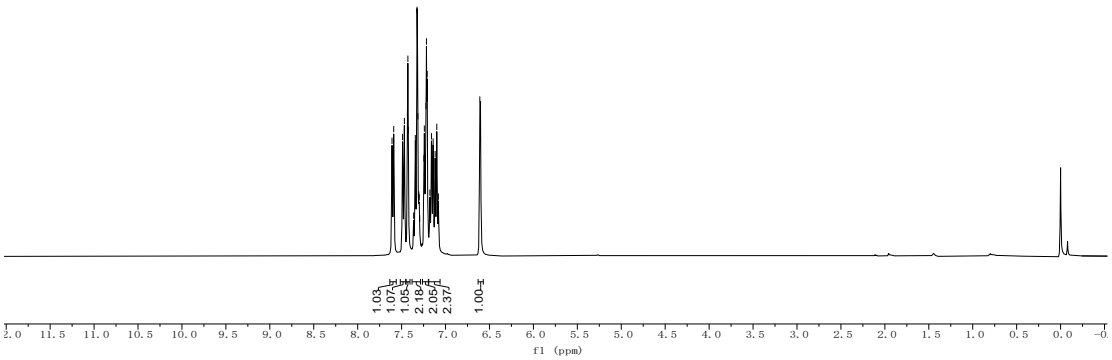


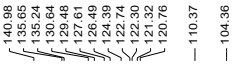


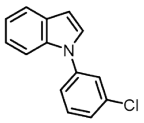
1-(3-chlorophenyl)-1*H*-indole (3k)

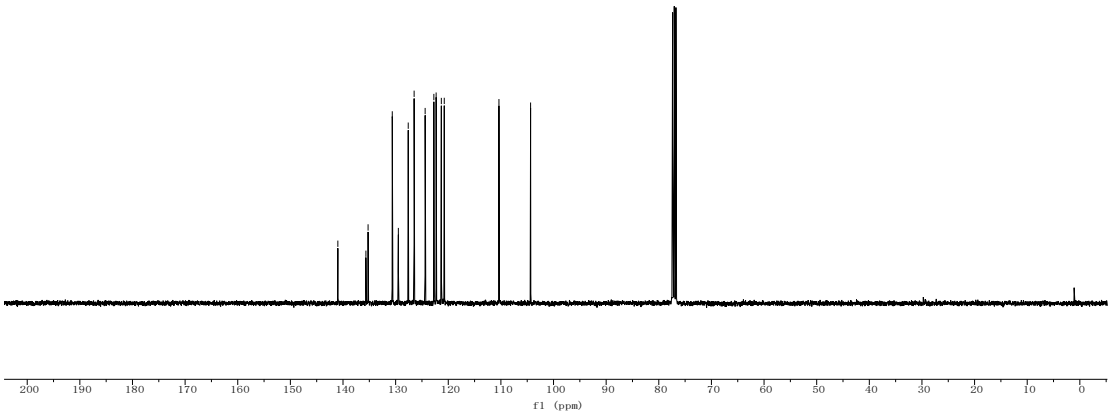




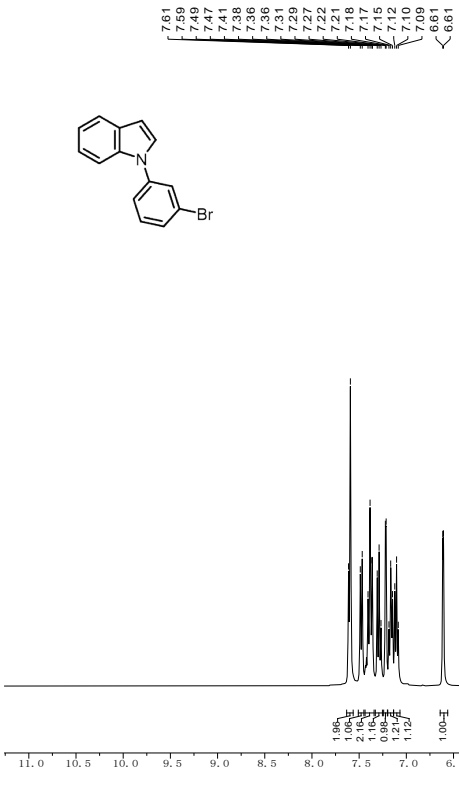


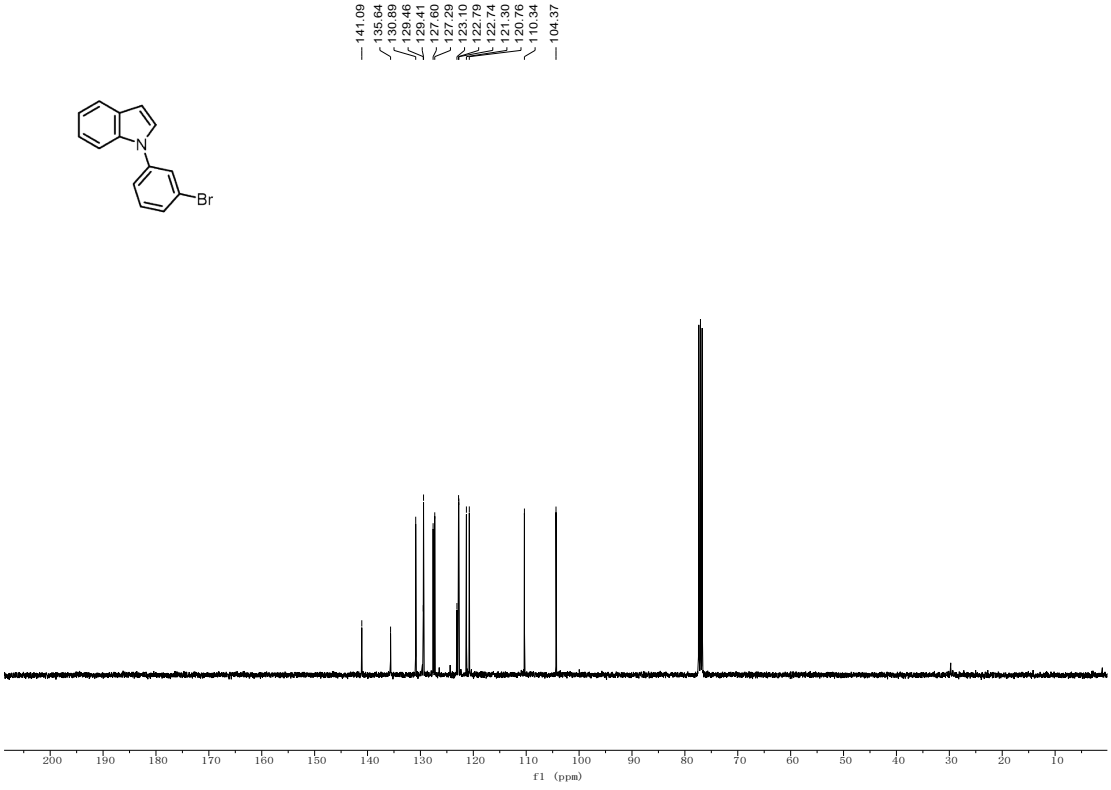




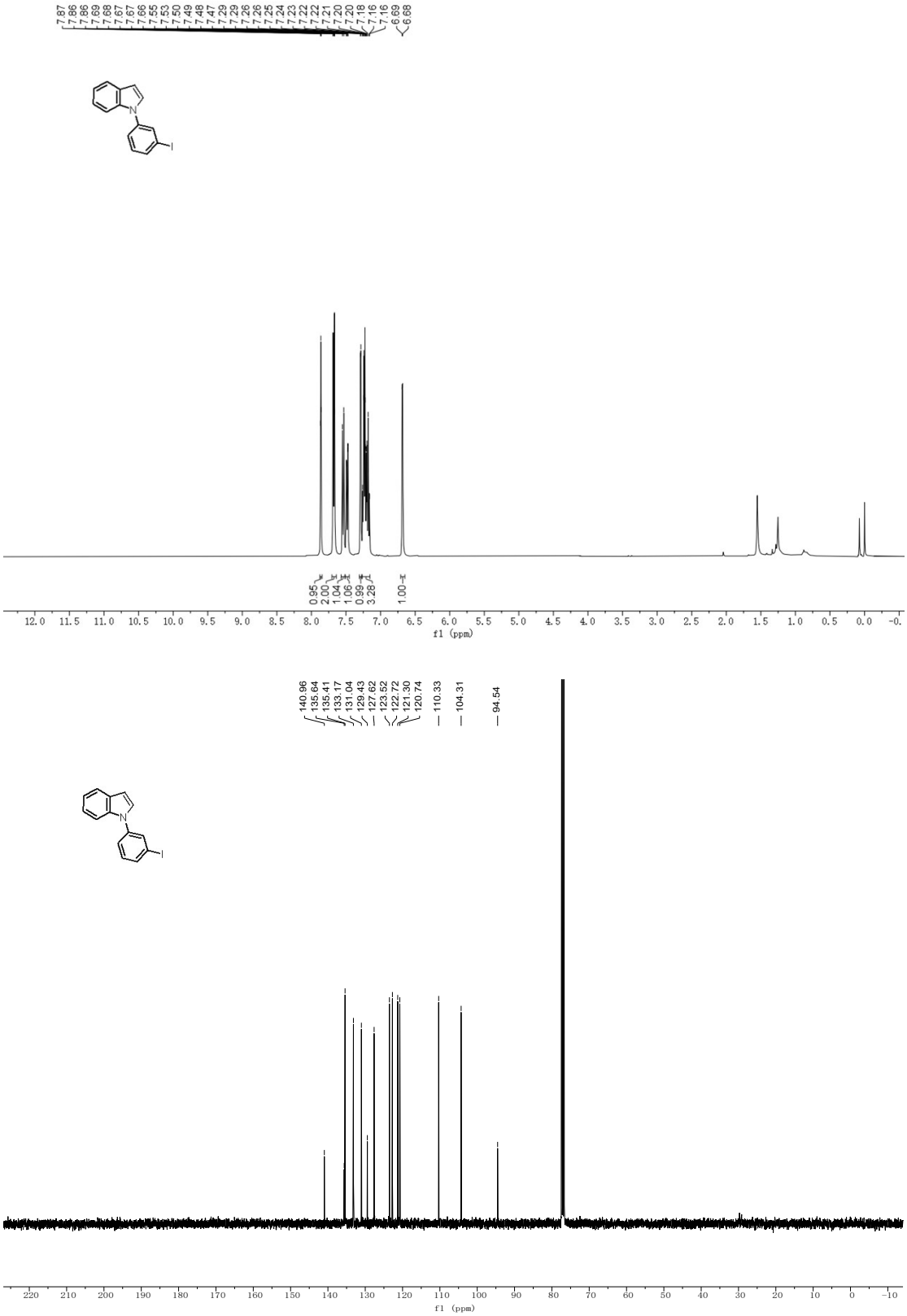


1-(3-bromophenyl)-1*H*-indole (3l)

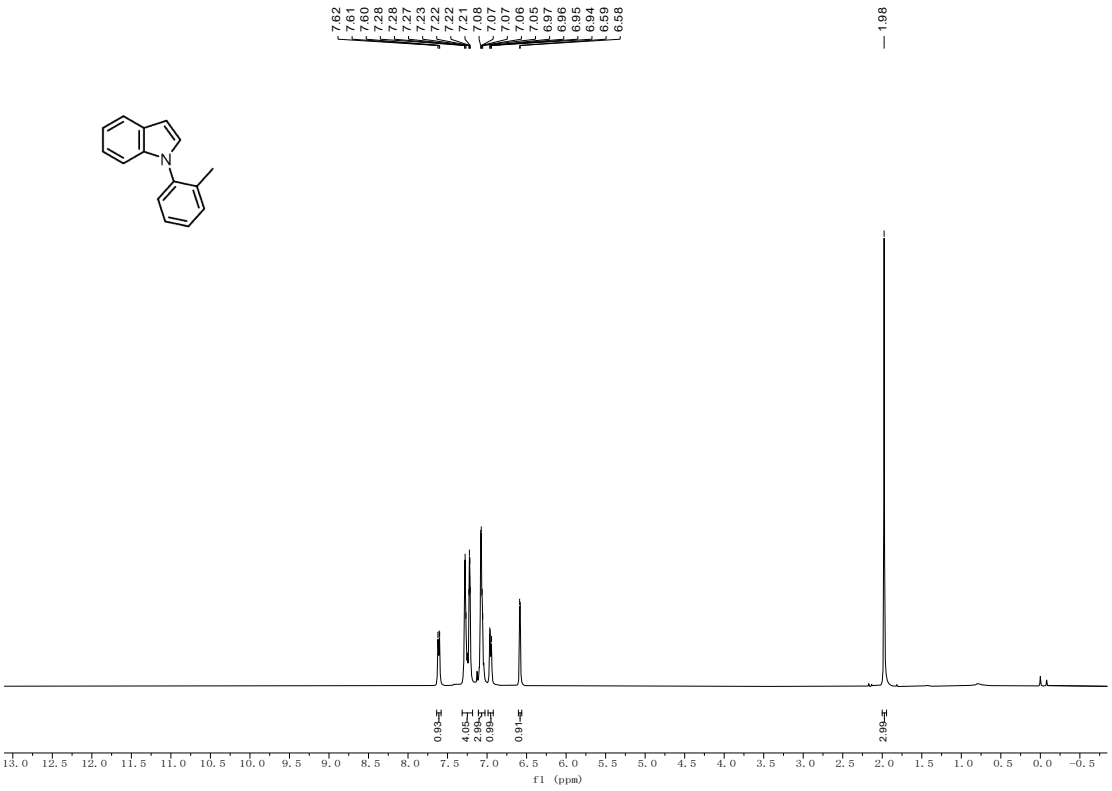


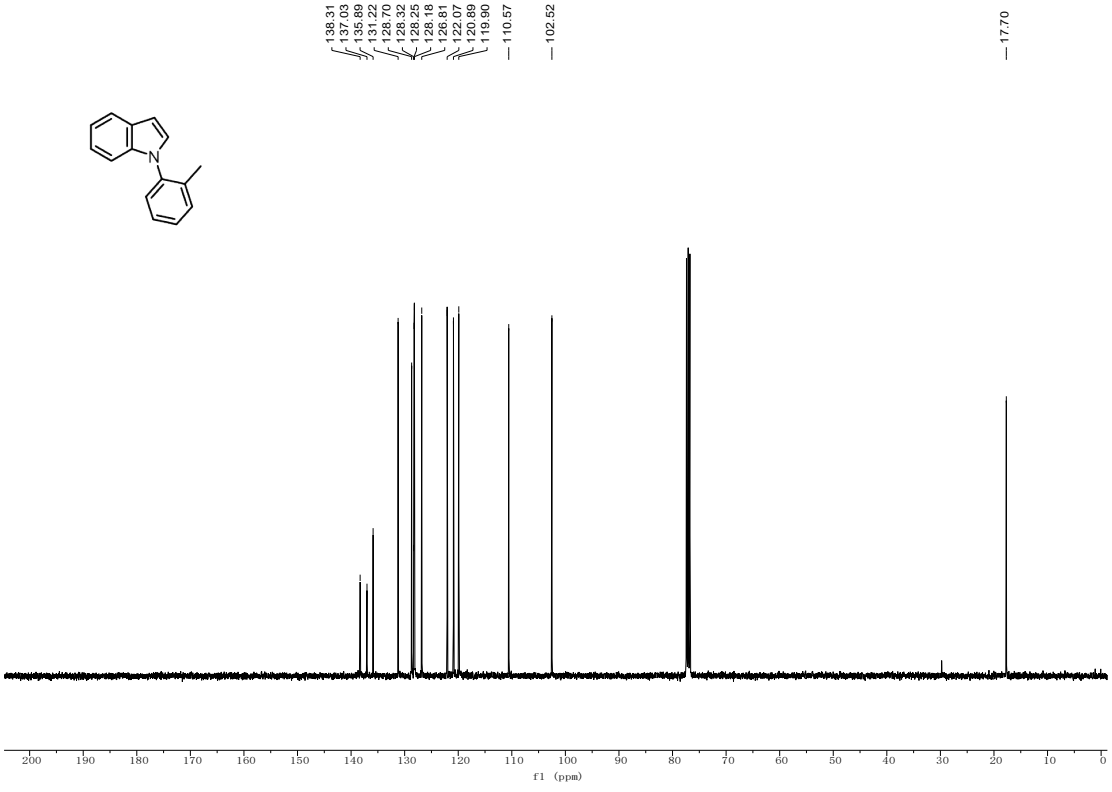


1-(3- iodophenyl) -1*H*-indole (3m)

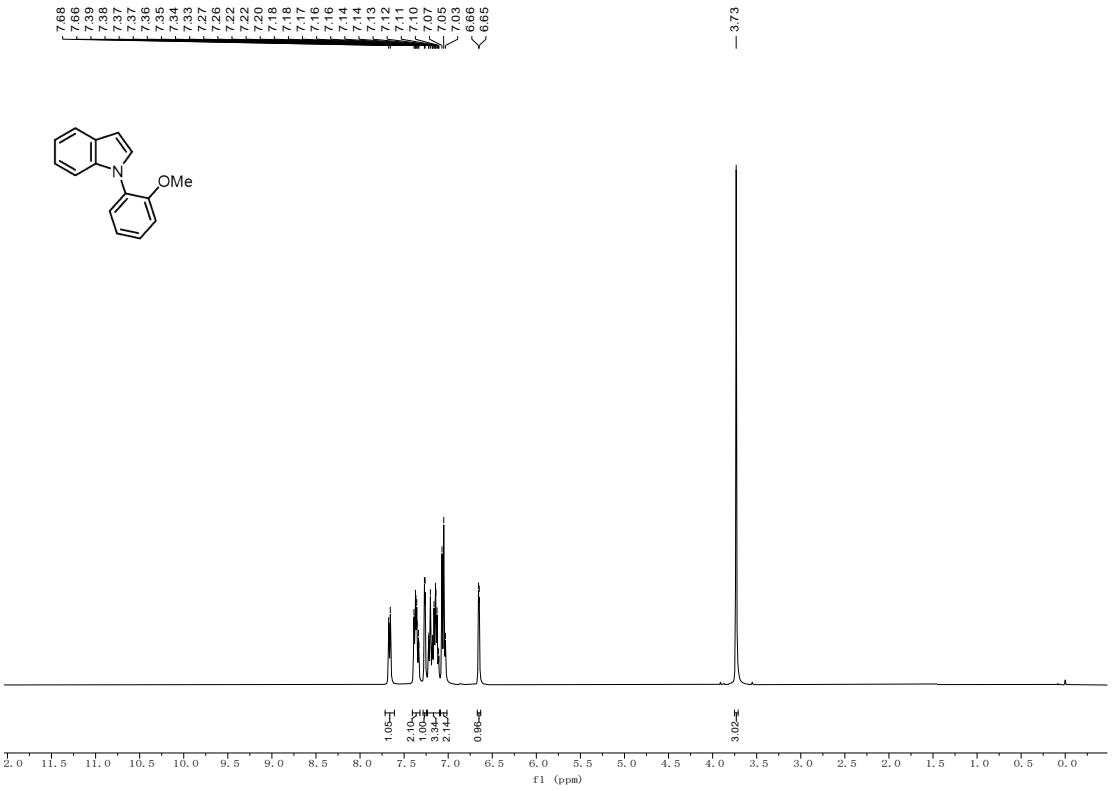


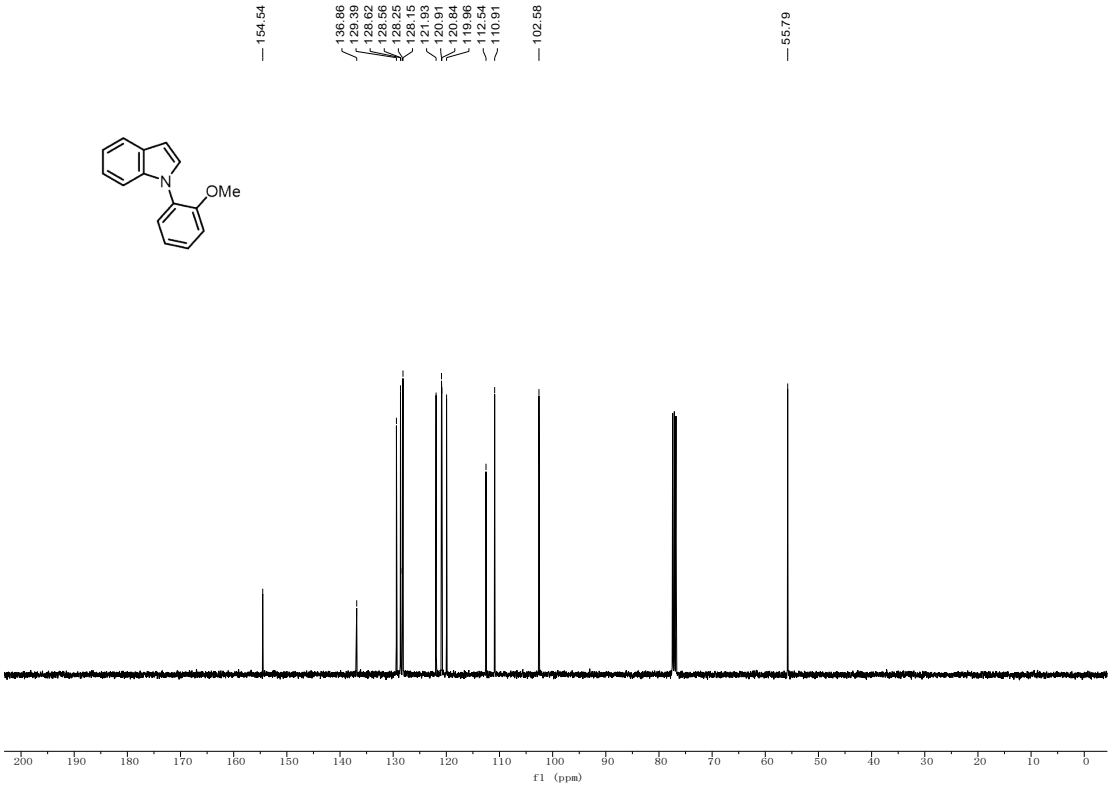
1-(*o*-tolyl)-1*H*-indole (3n)



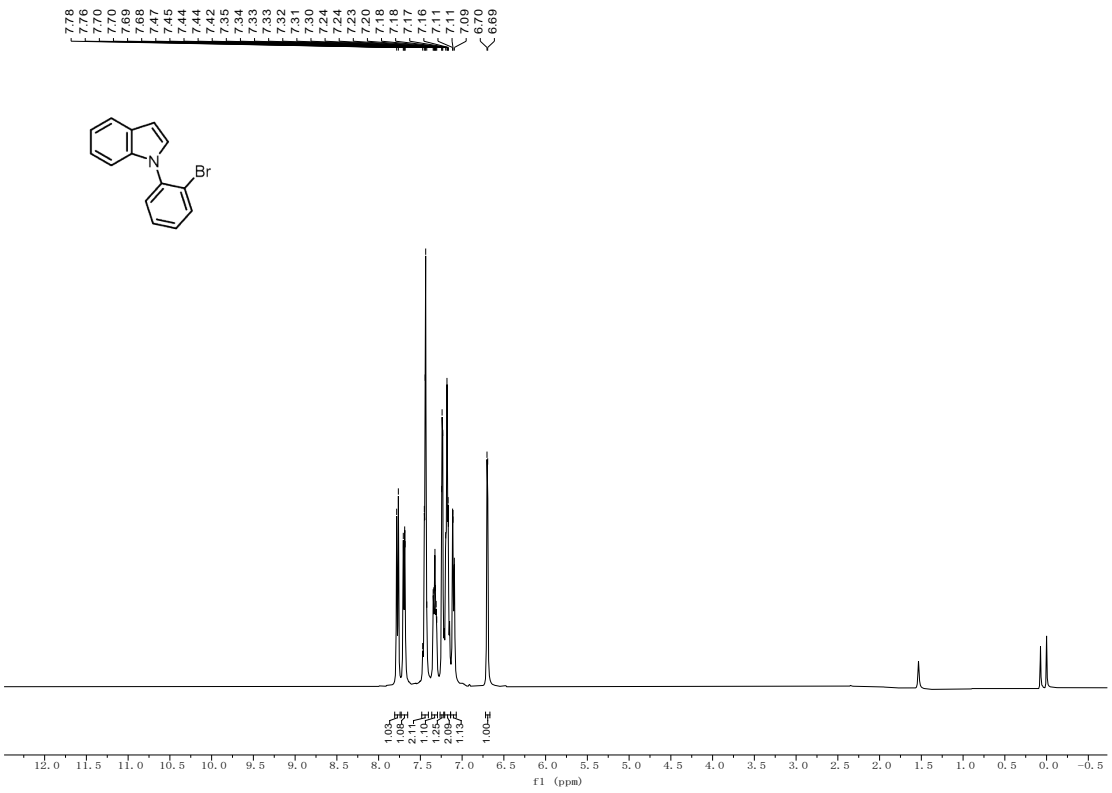


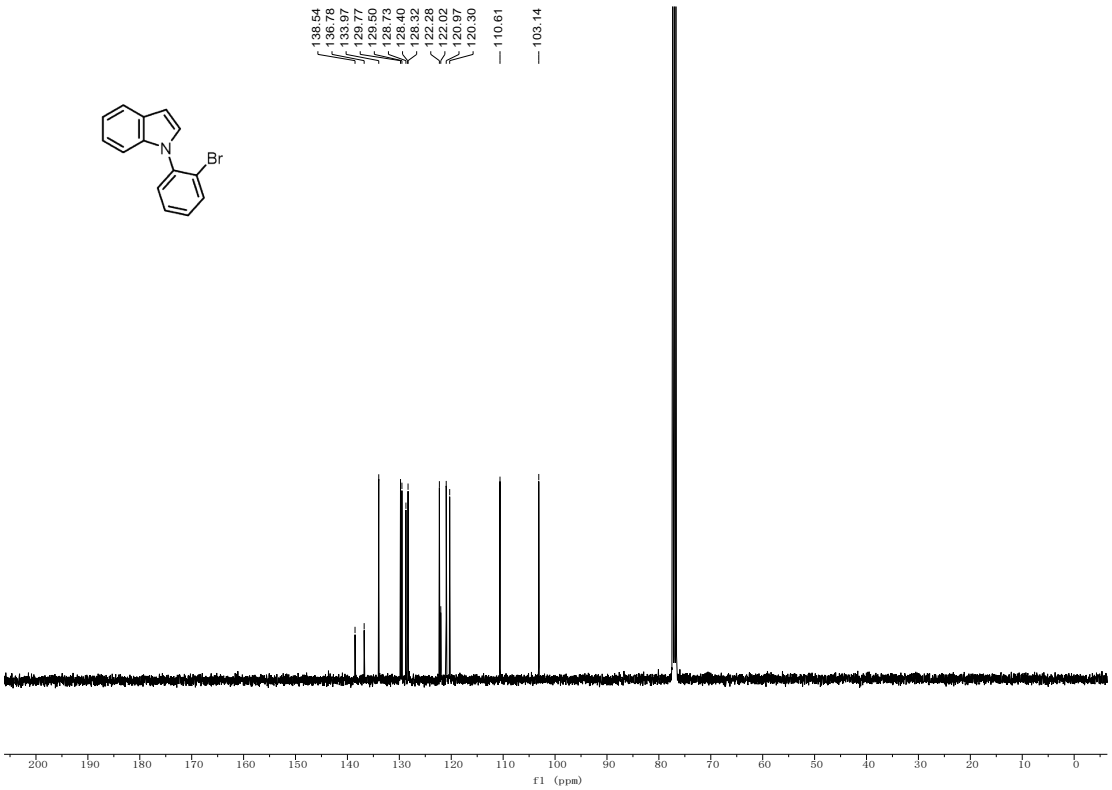
1-(2-methoxyphenyl)-1*H*-indole (3o)



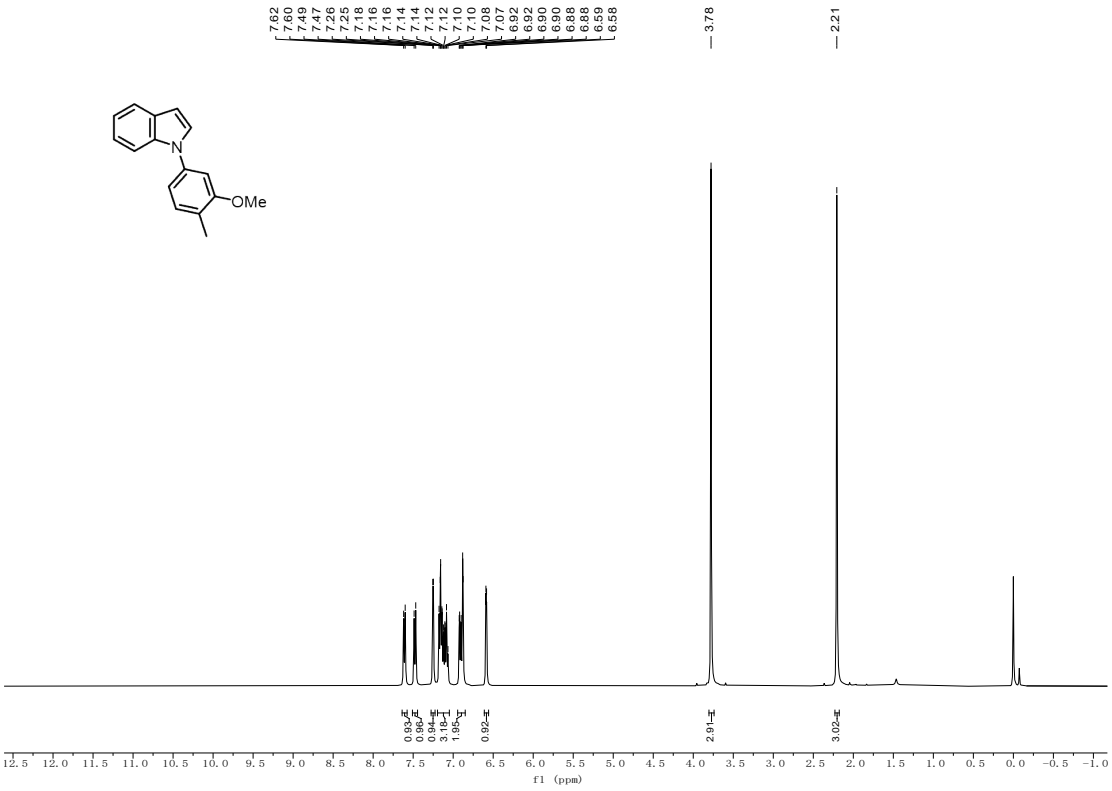


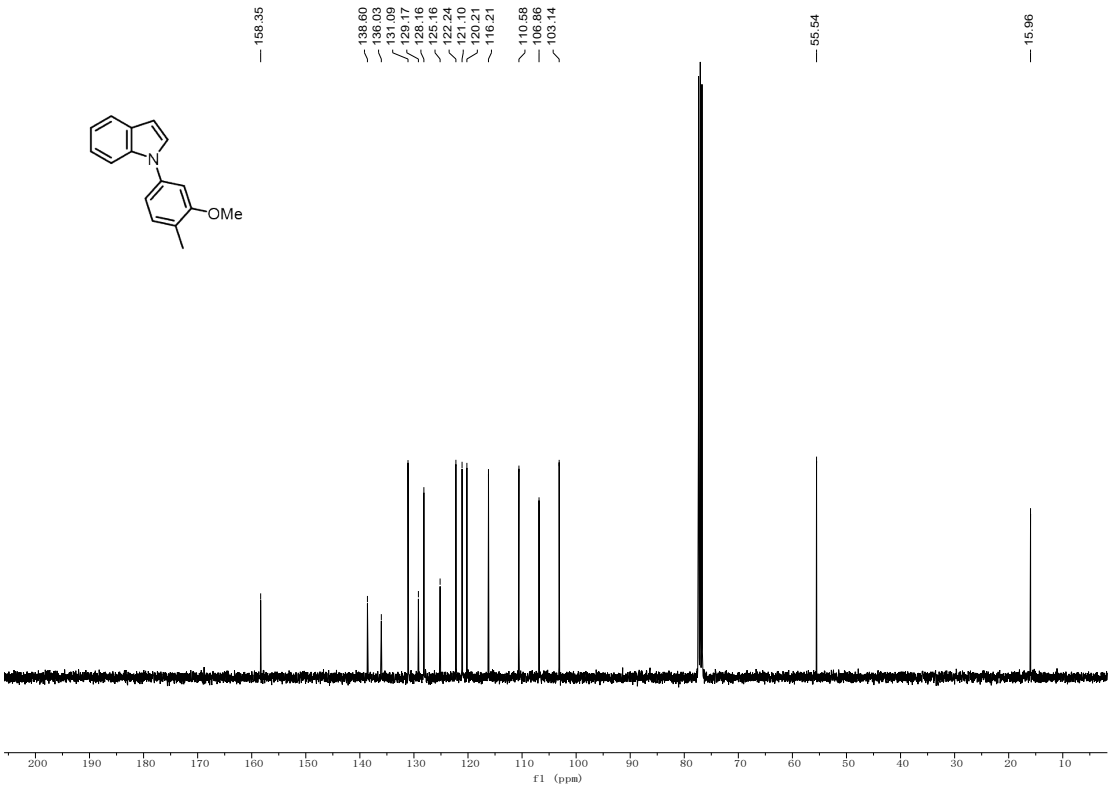
1-(2-bromophenyl)-1*H*-indole (3p)



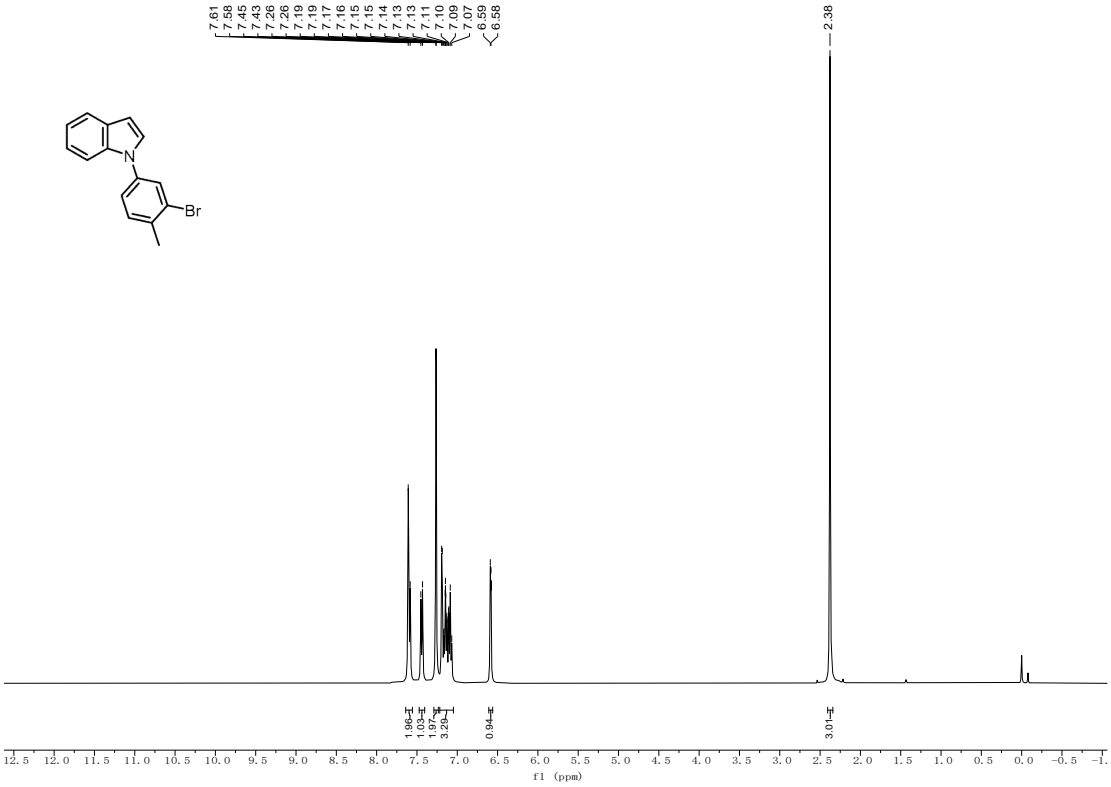


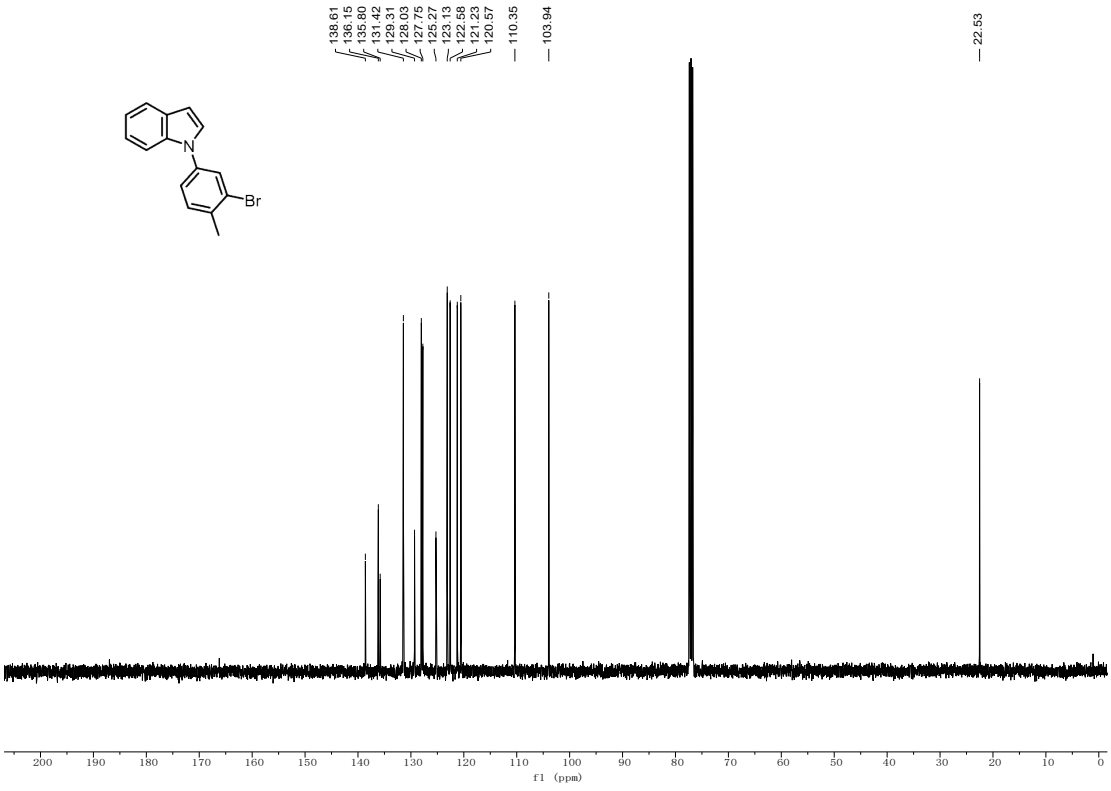
1-(3-methoxy-4-methylphenyl)-1*H*-indole (3q)



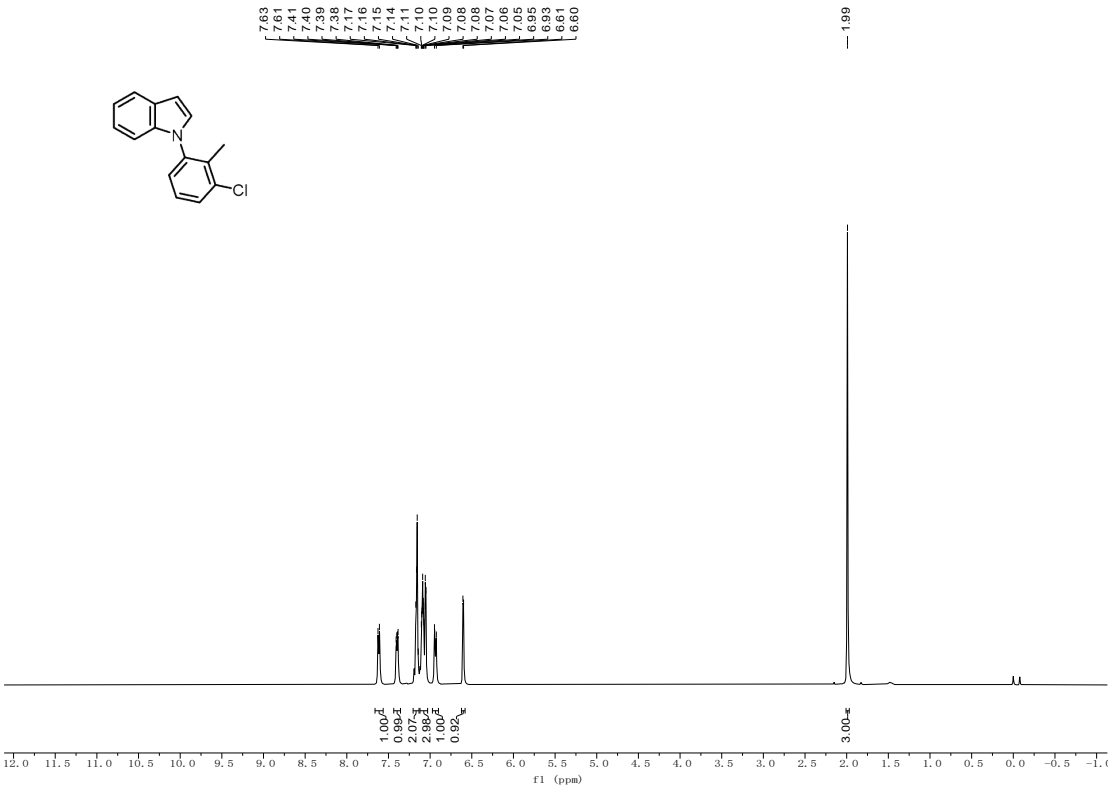


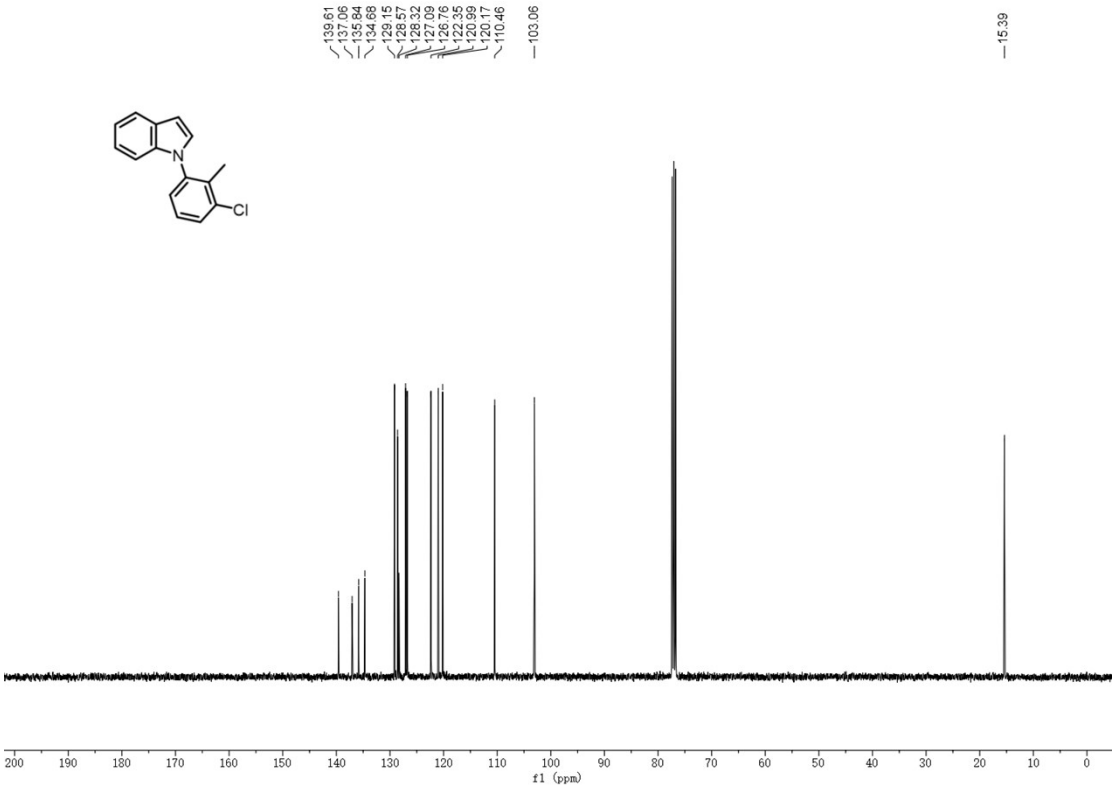
1-(3-bromo-4-methylphenyl)-1*H*-indole (3r)



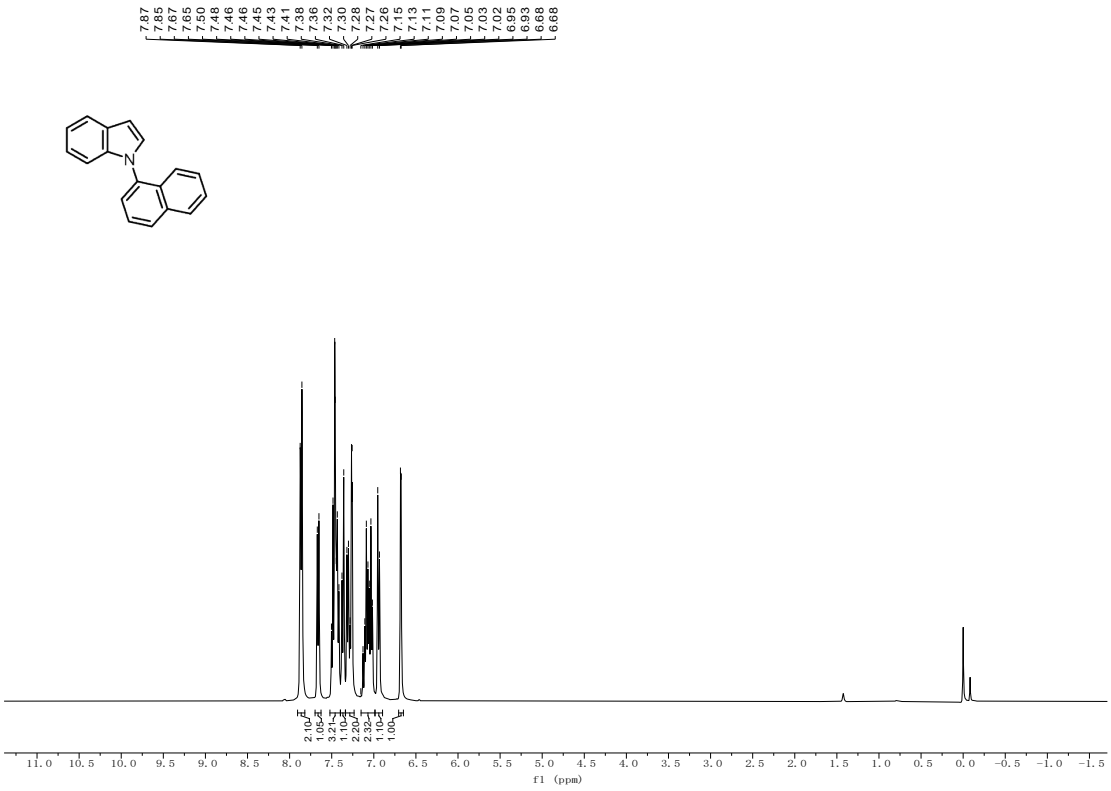


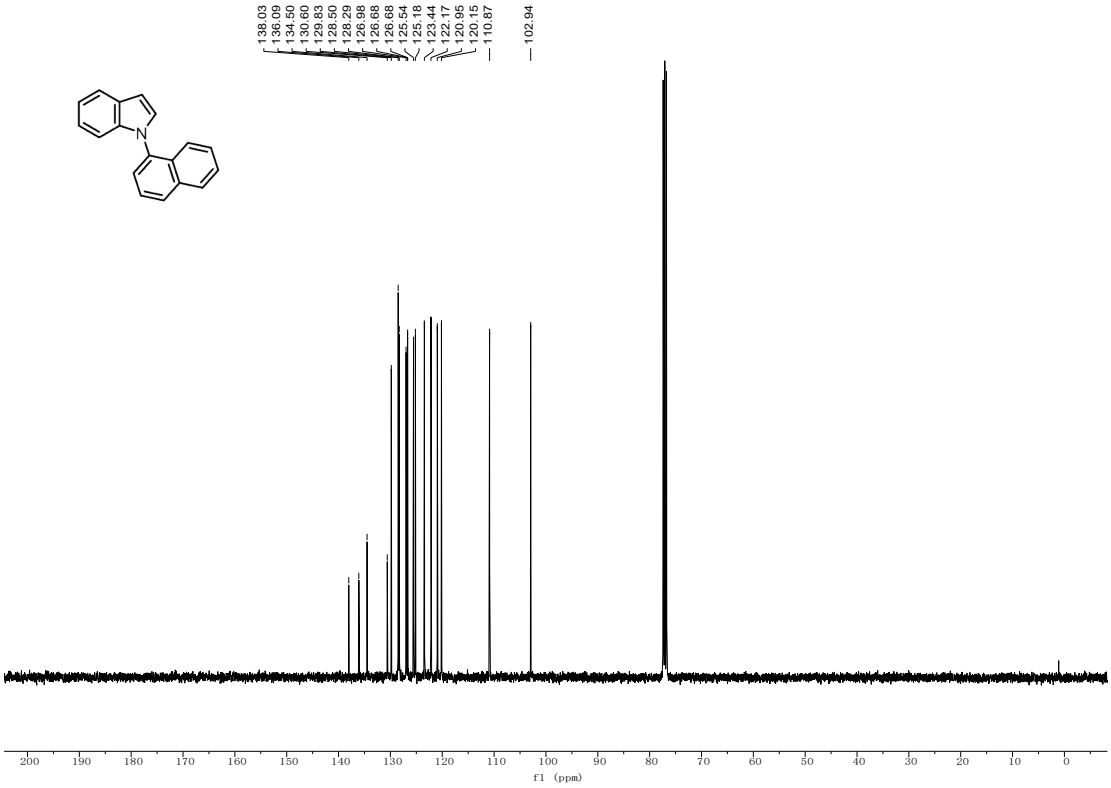
1-(3-chloro-2-methylphenyl)-1*H*-indole (3s)



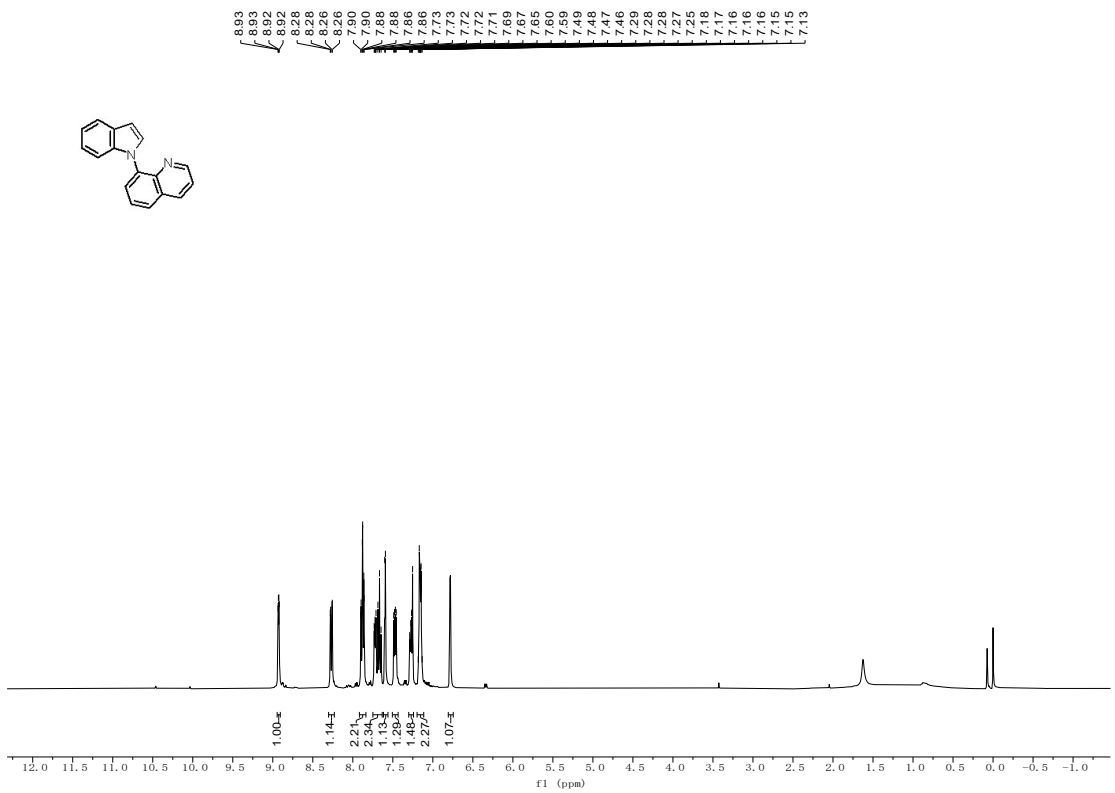


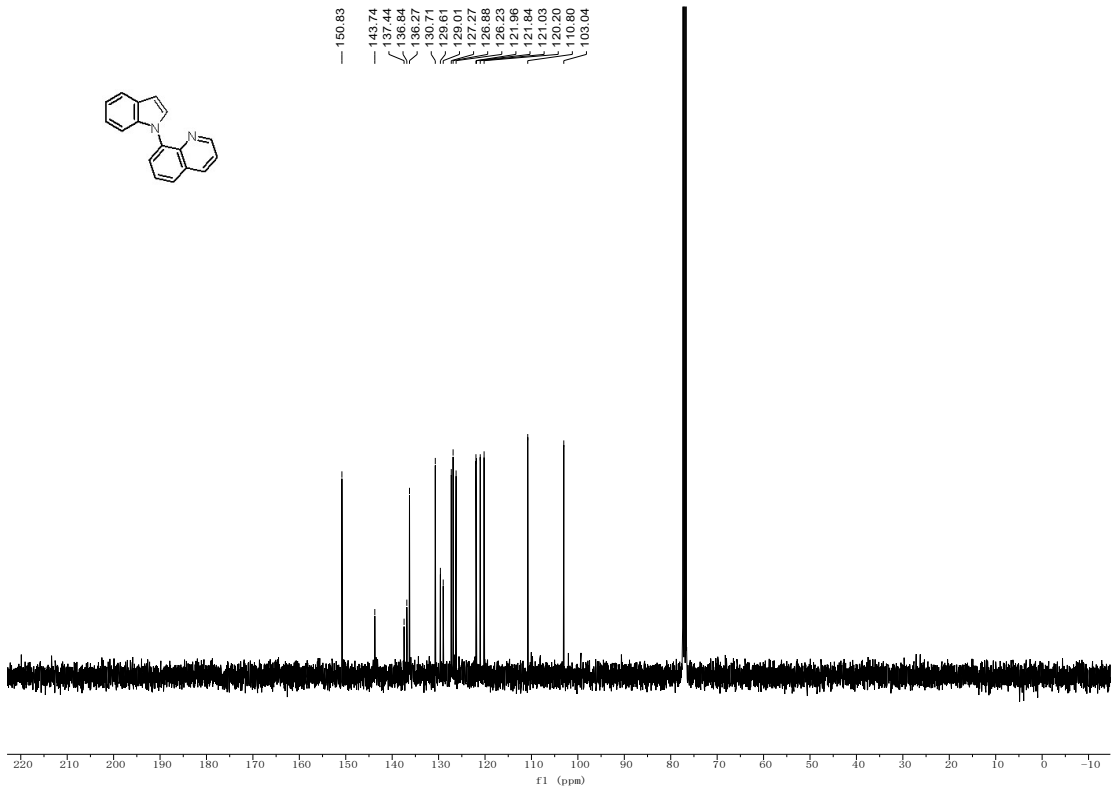
1-(naphthalen-1-yl)-1*H*-indole (3t)



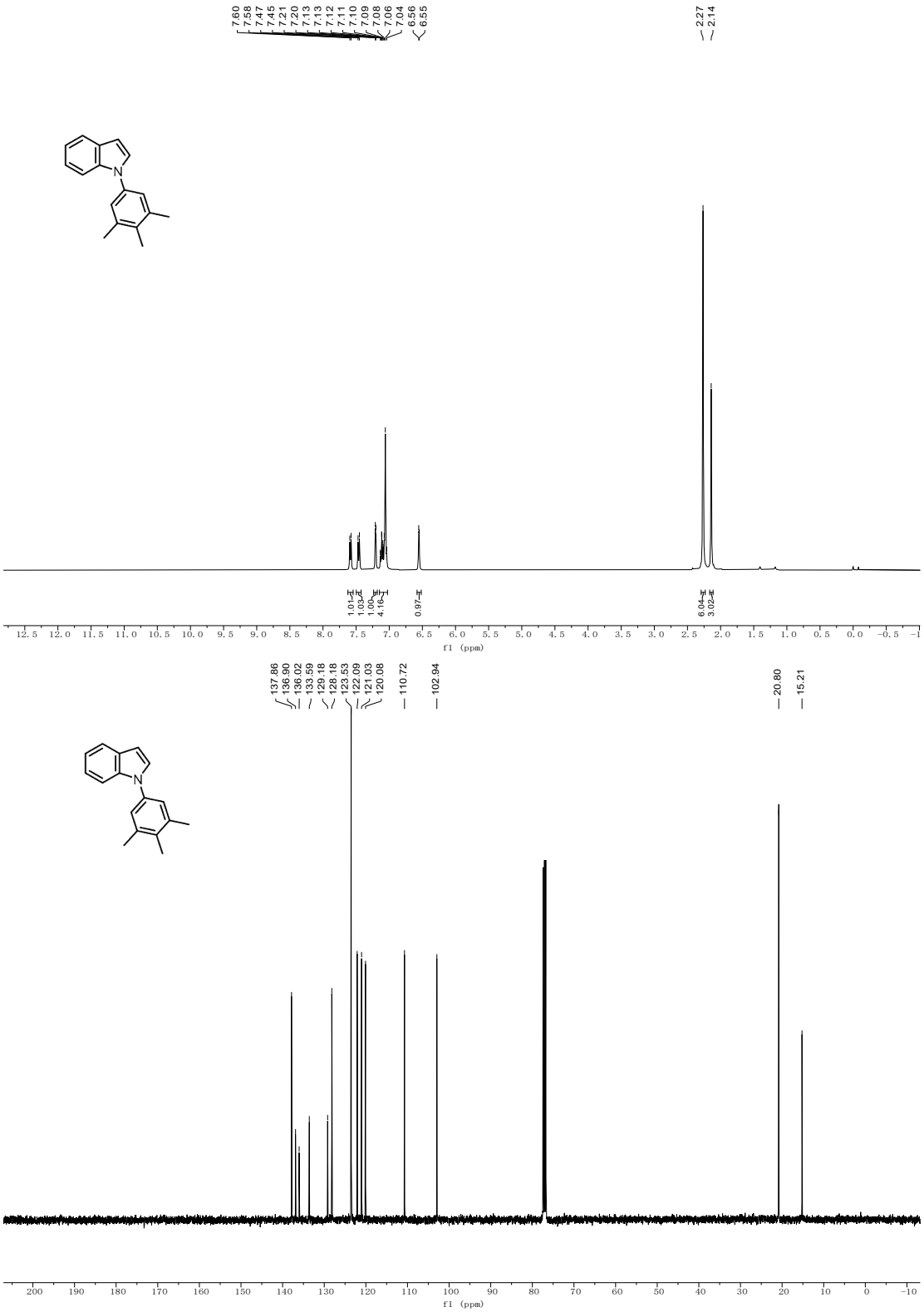


8-(1*H*-indol-1-yl)quinoline (3u)

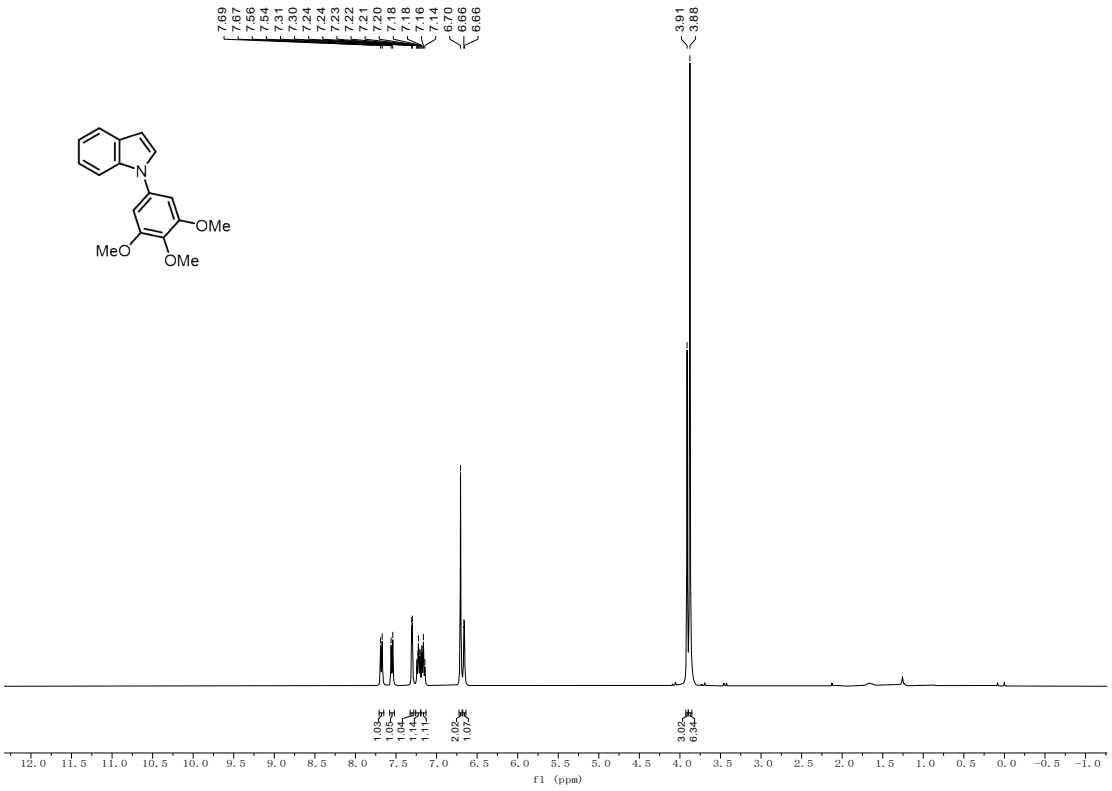


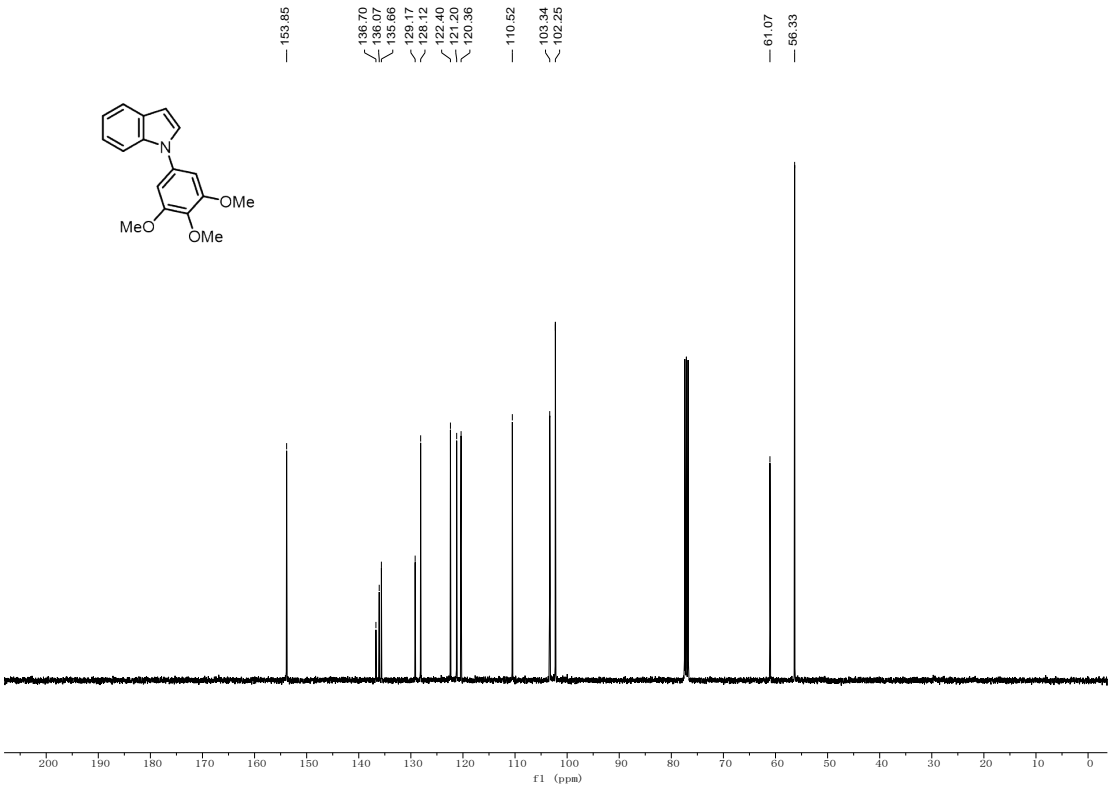


1-(3,4,5-trimethylphenyl)-1*H*-indole (3v)

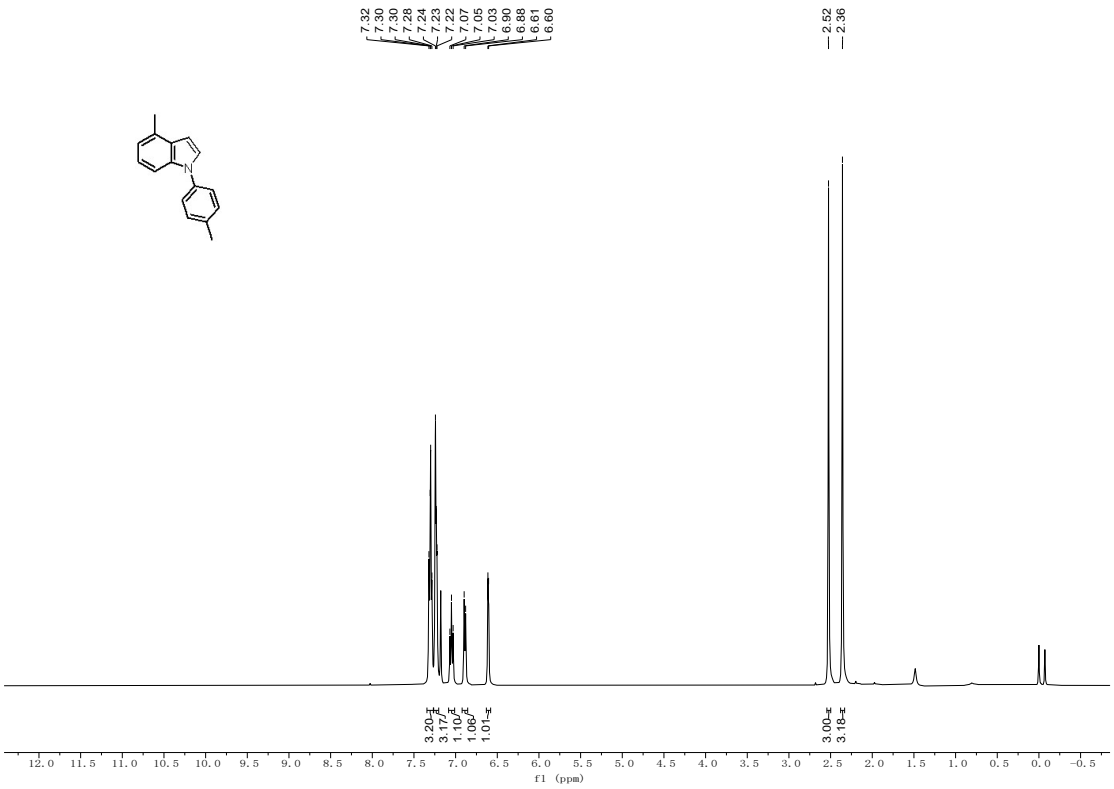


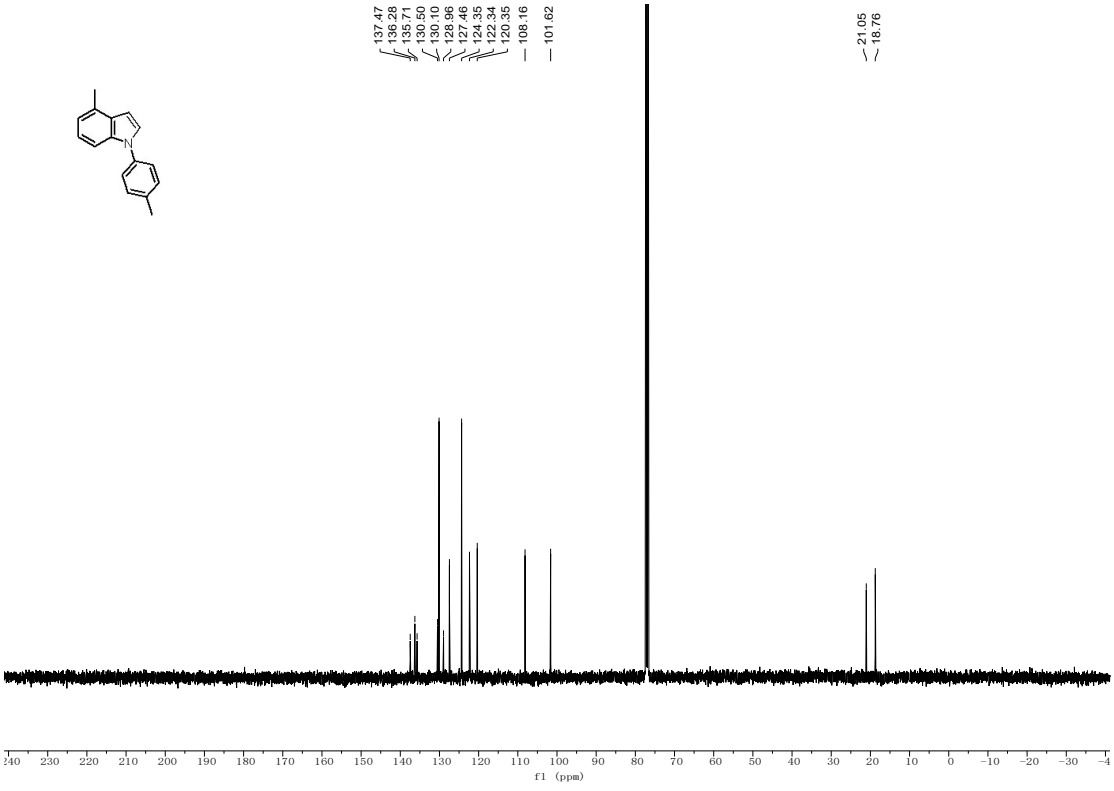
1-(3,4,5-trimethoxyphenyl)-1*H*-indole (3w)



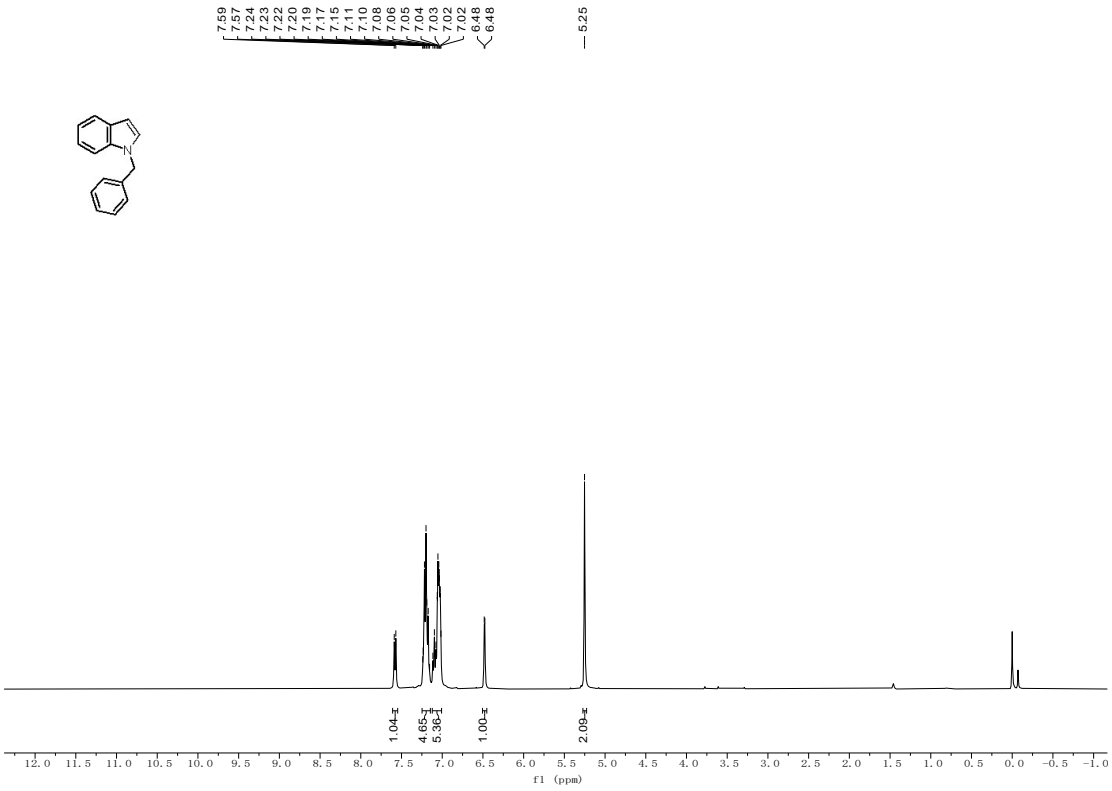


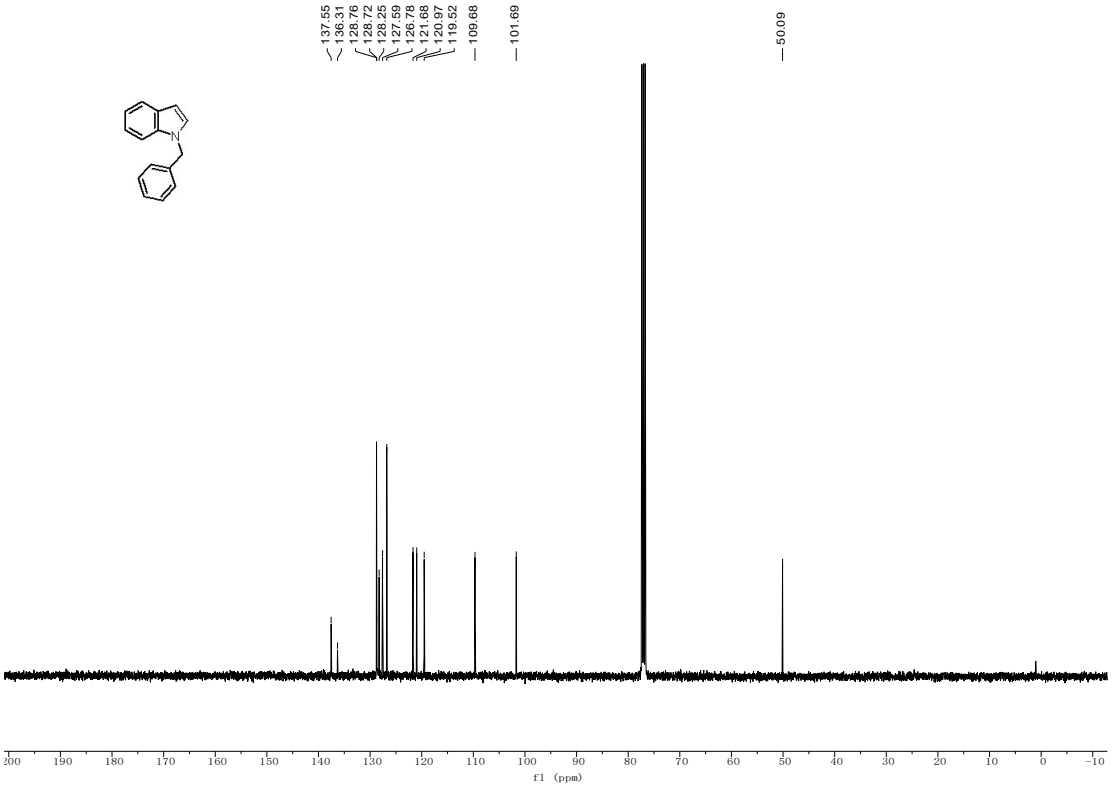
4-methyl-1-(*p*-tolyl)-1*H*-indole (3x)



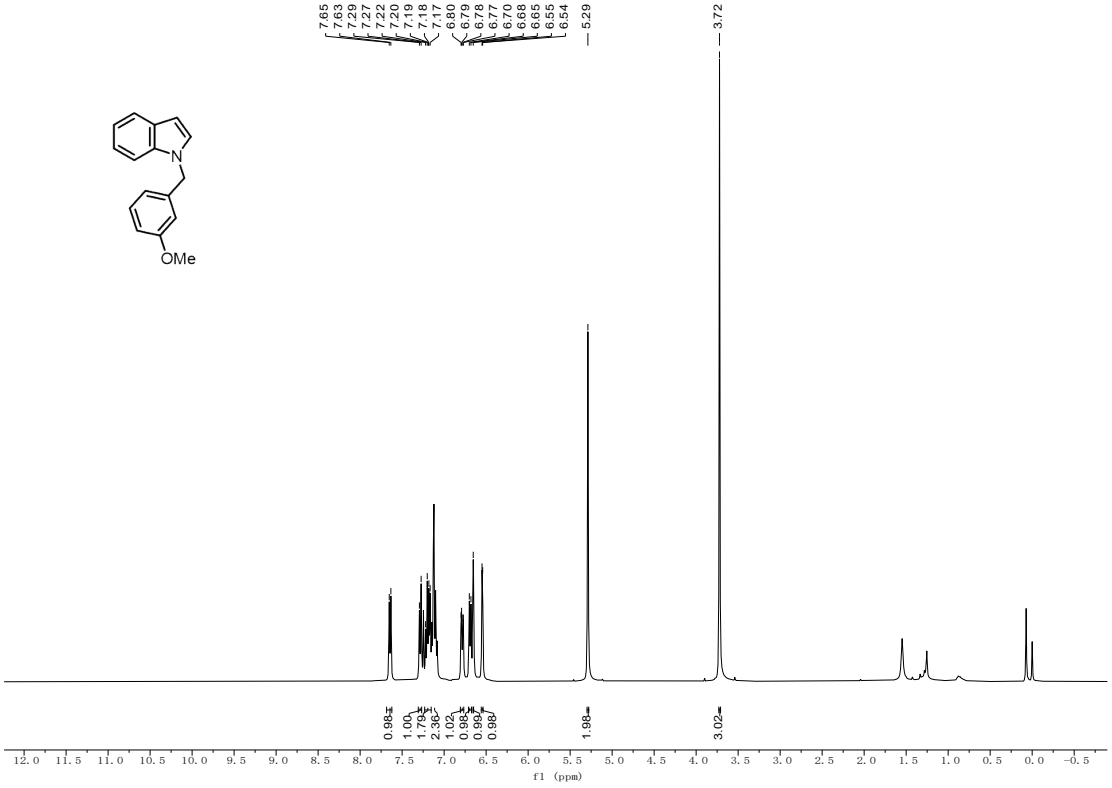


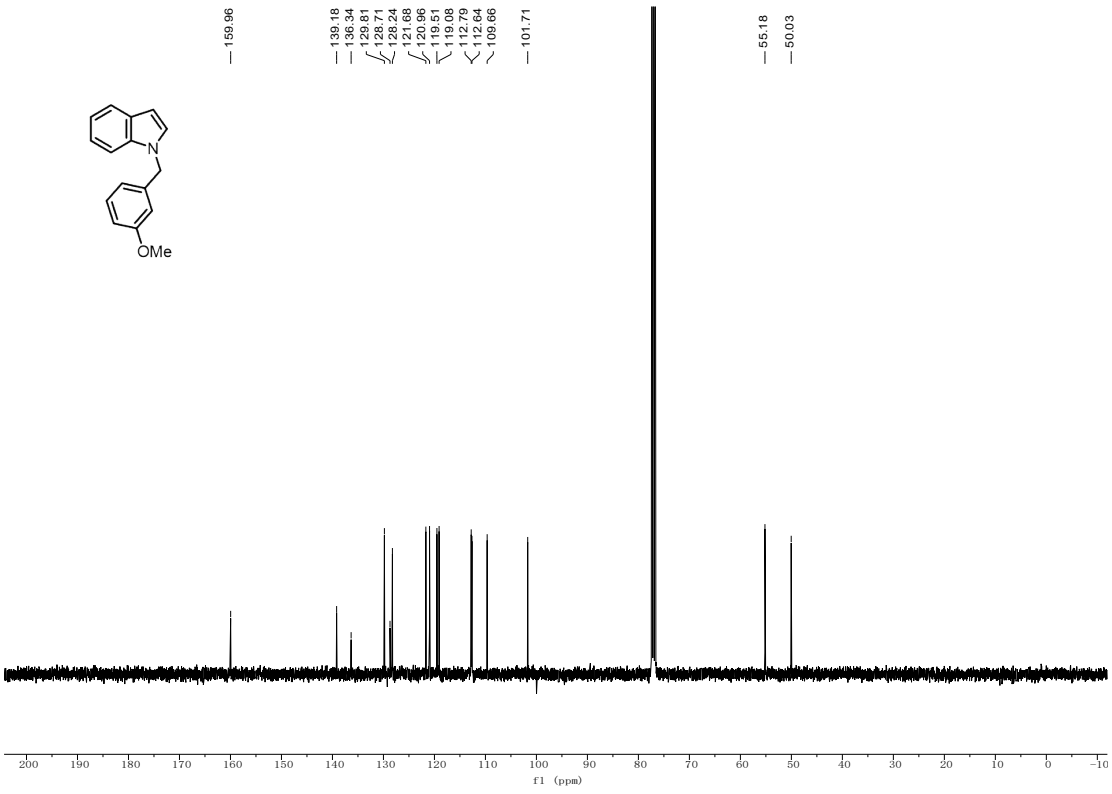
1-benzyl-1*H*-indole (3y)



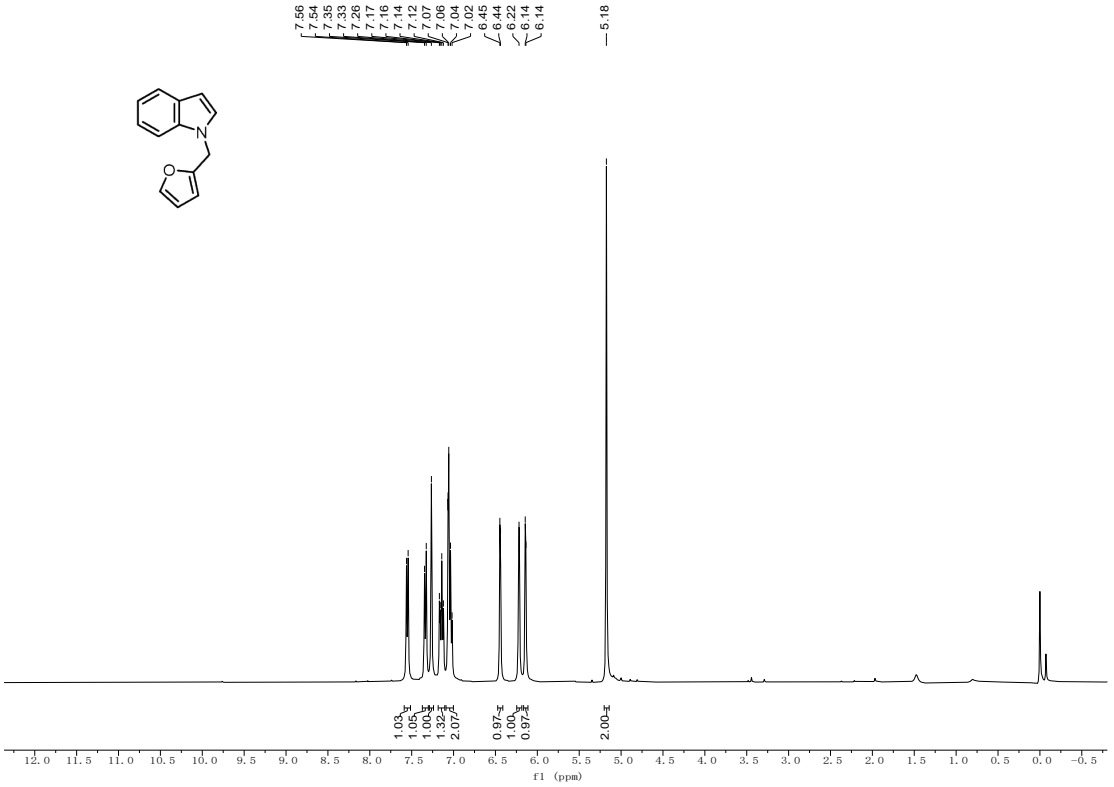


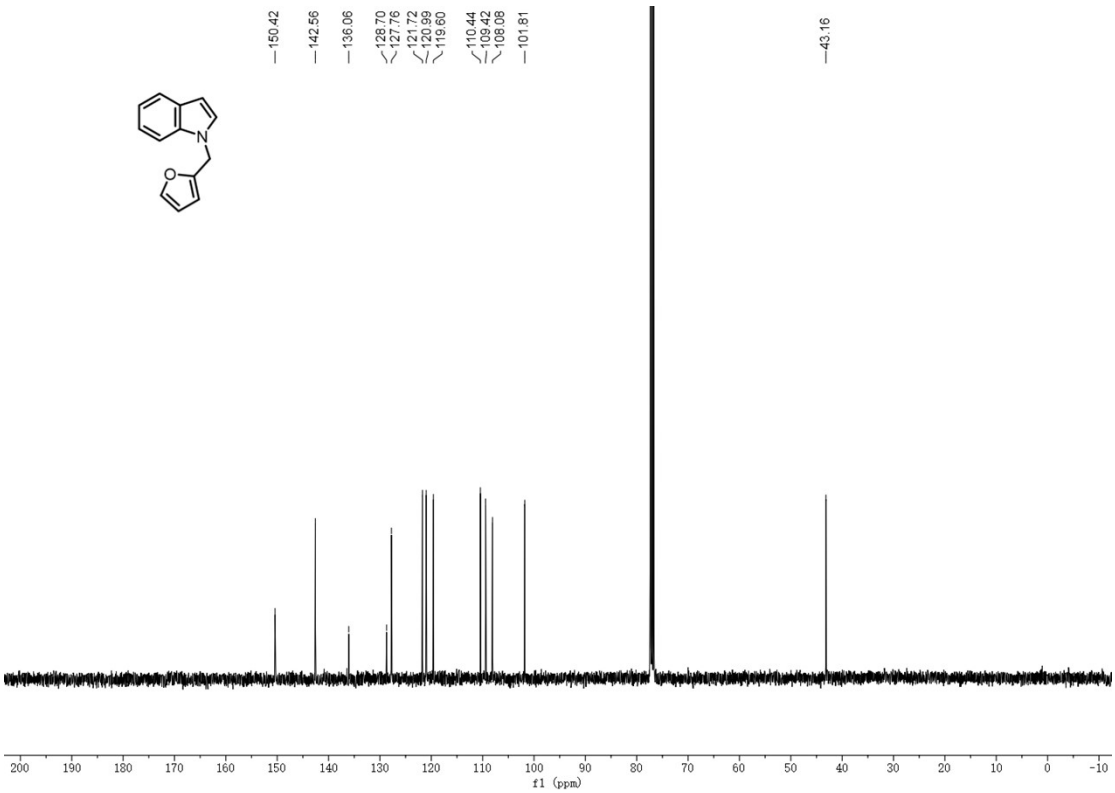
1-(3-methoxybenzyl)-1*H*-indole (3z)



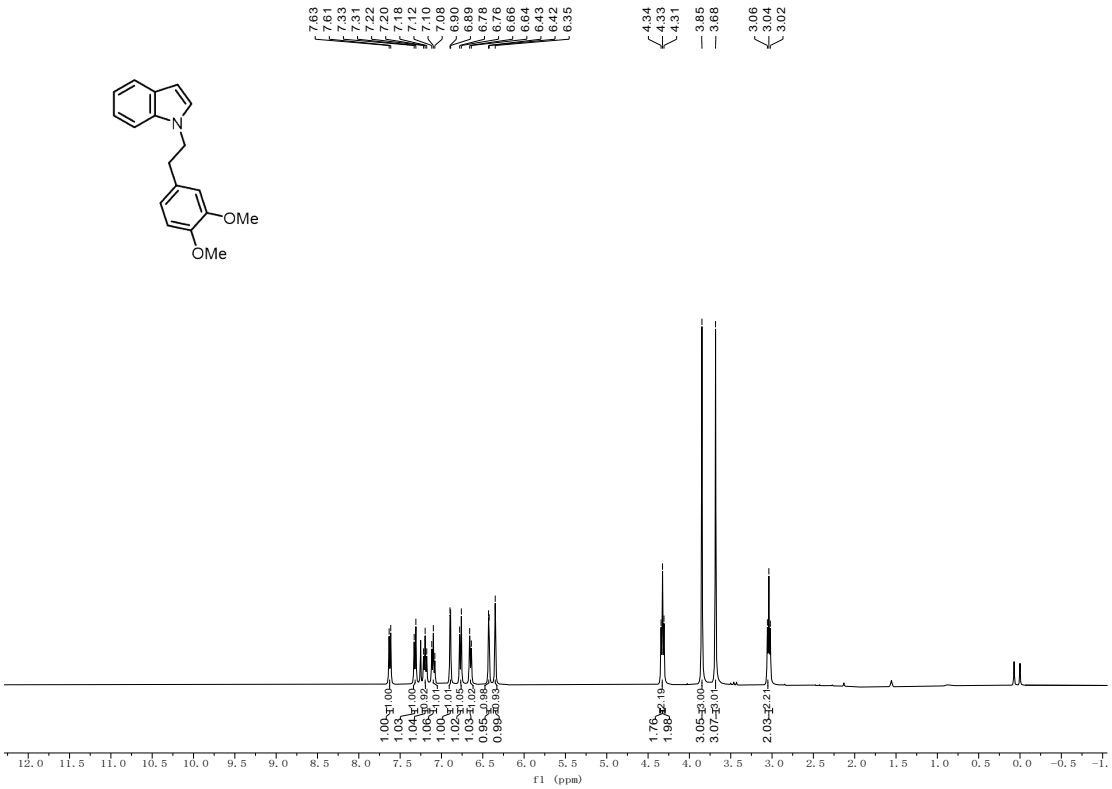


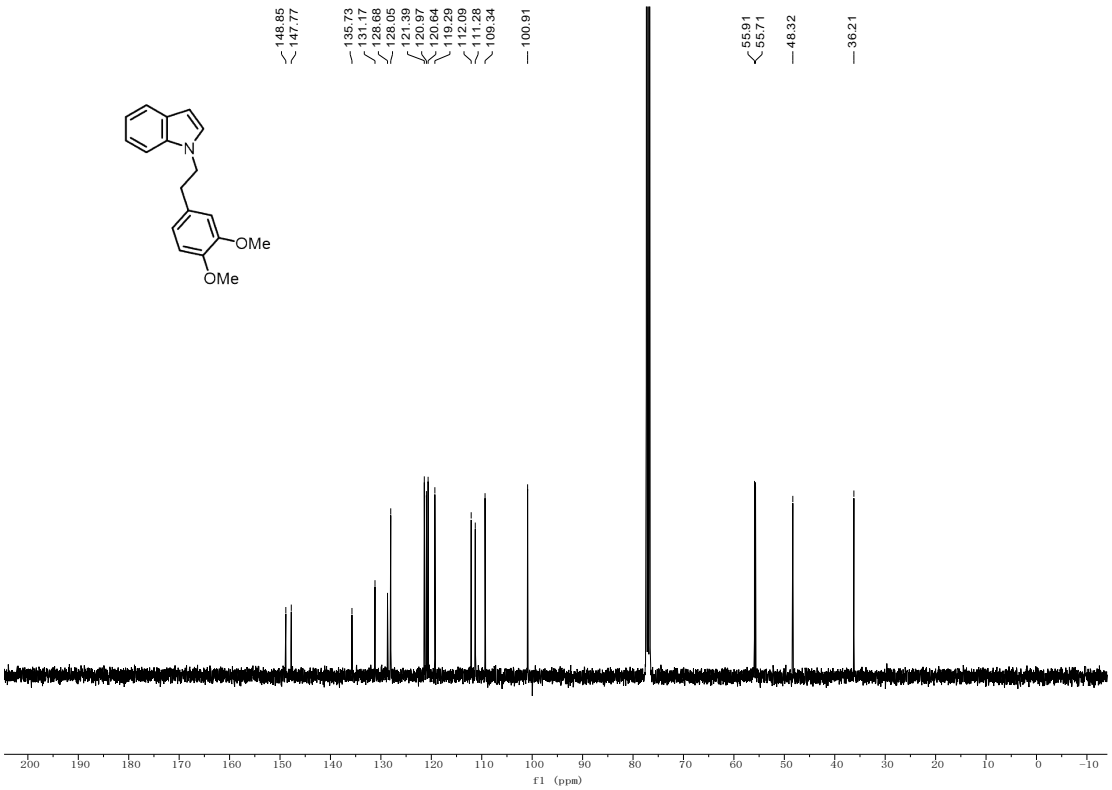
1-(furan-2-ylmethyl)-1*H*-indole (3aa)



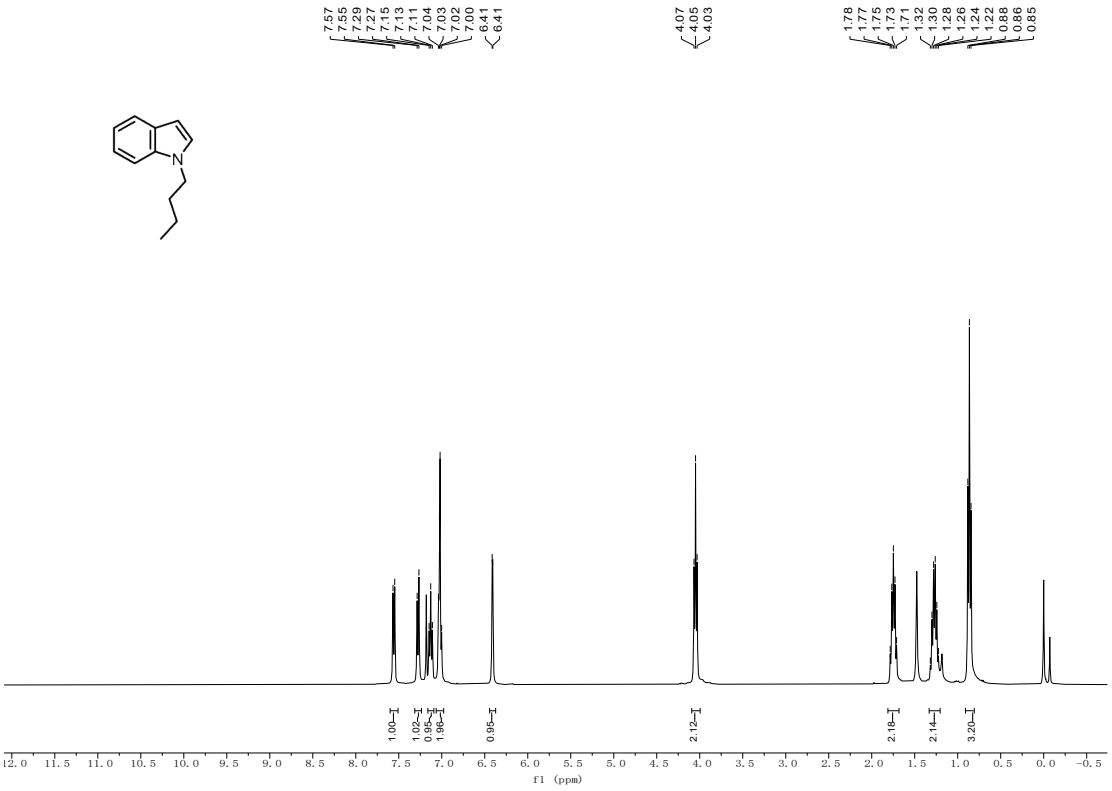


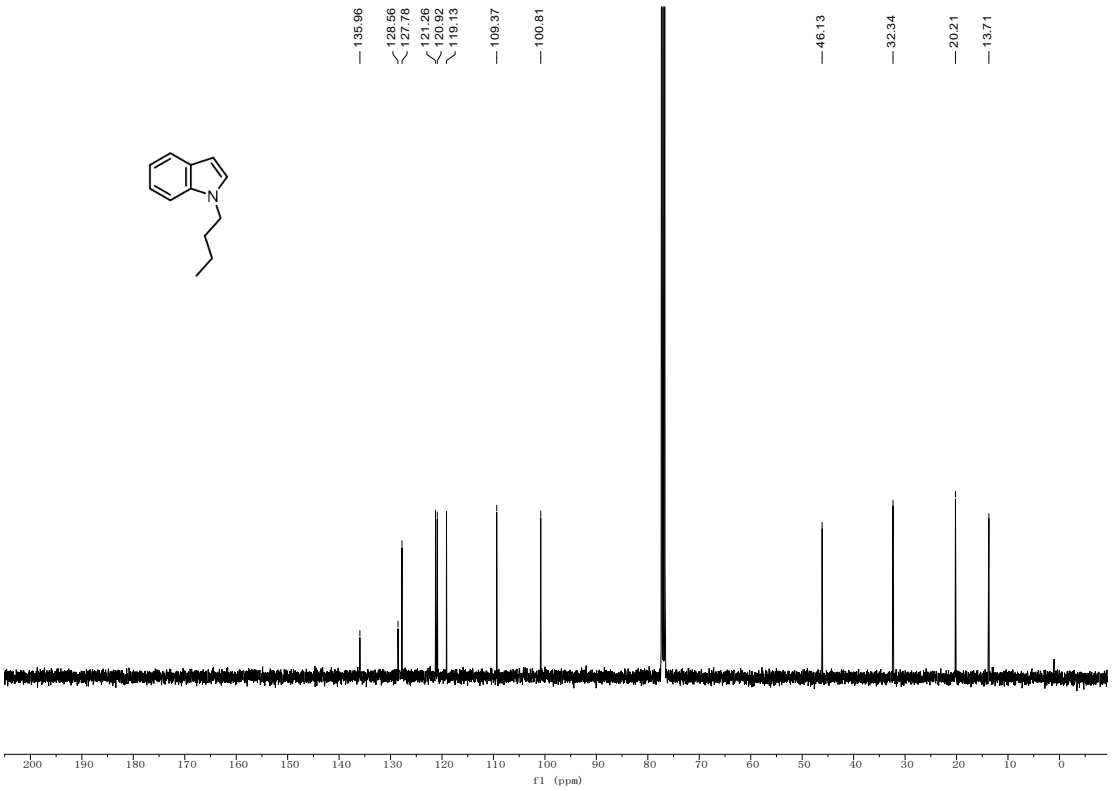
1-(3,4-dimethoxyphenethyl)-1*H*-indole (3ab)



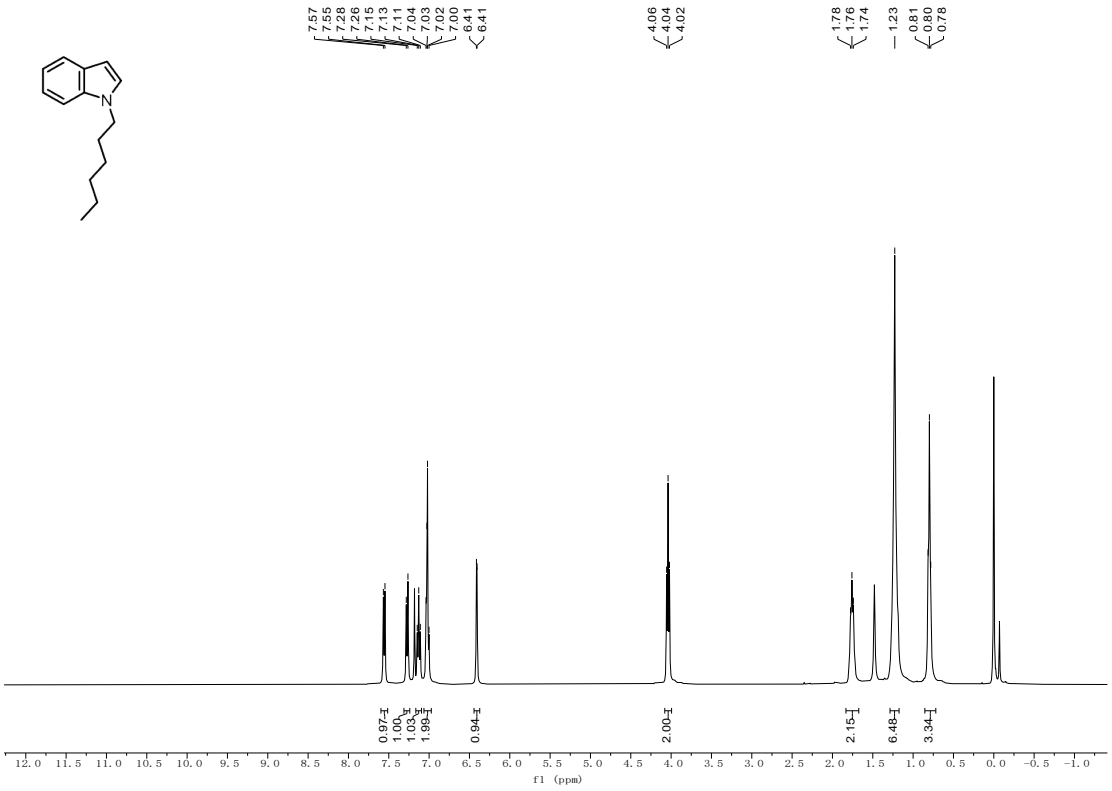


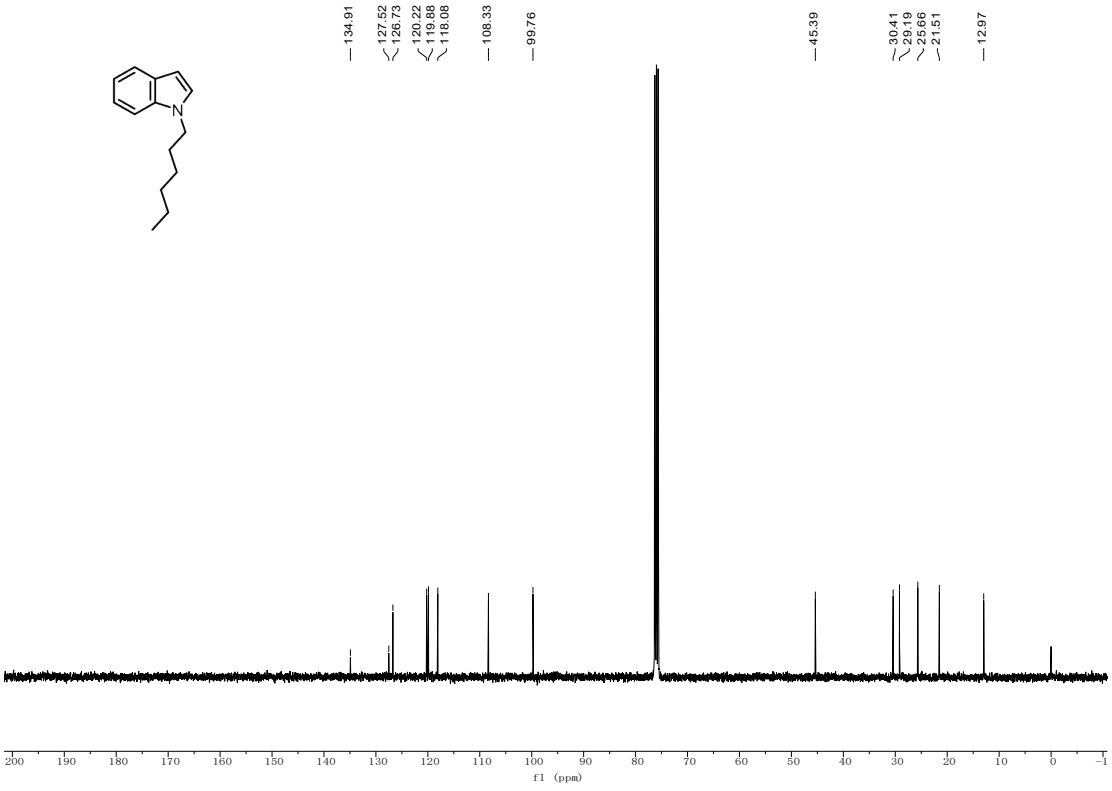
1-butyl-1*H*-indole (3ac)



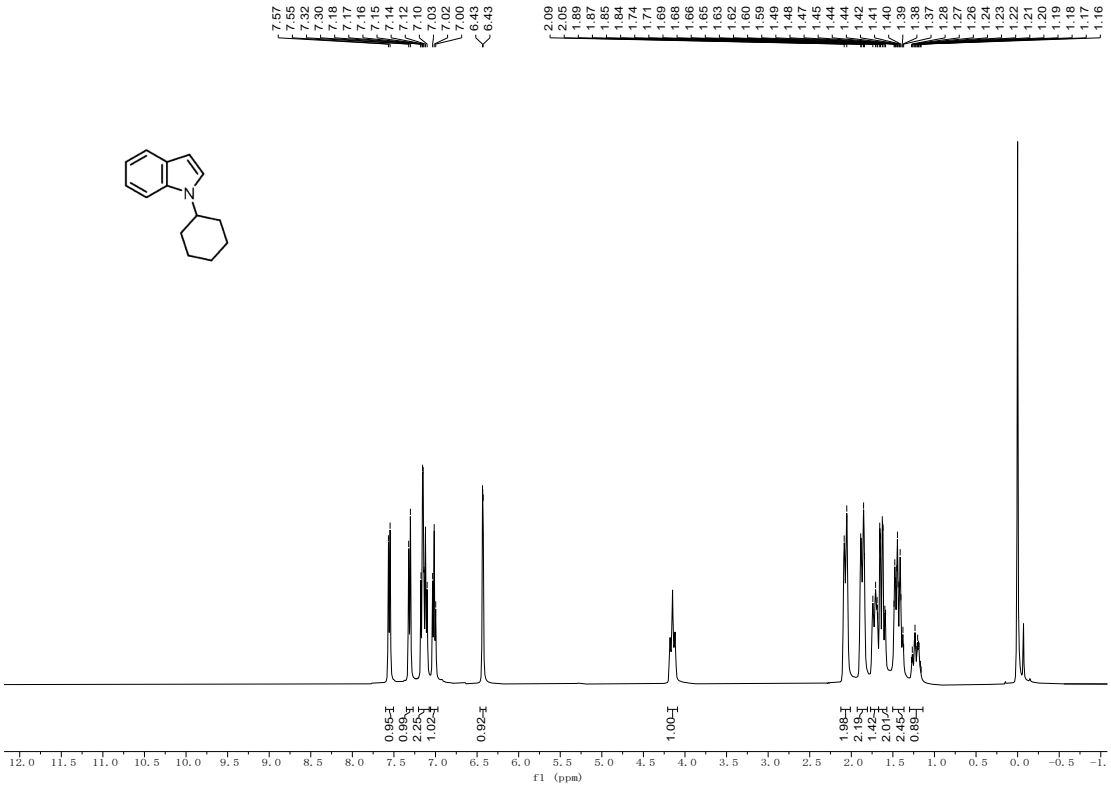


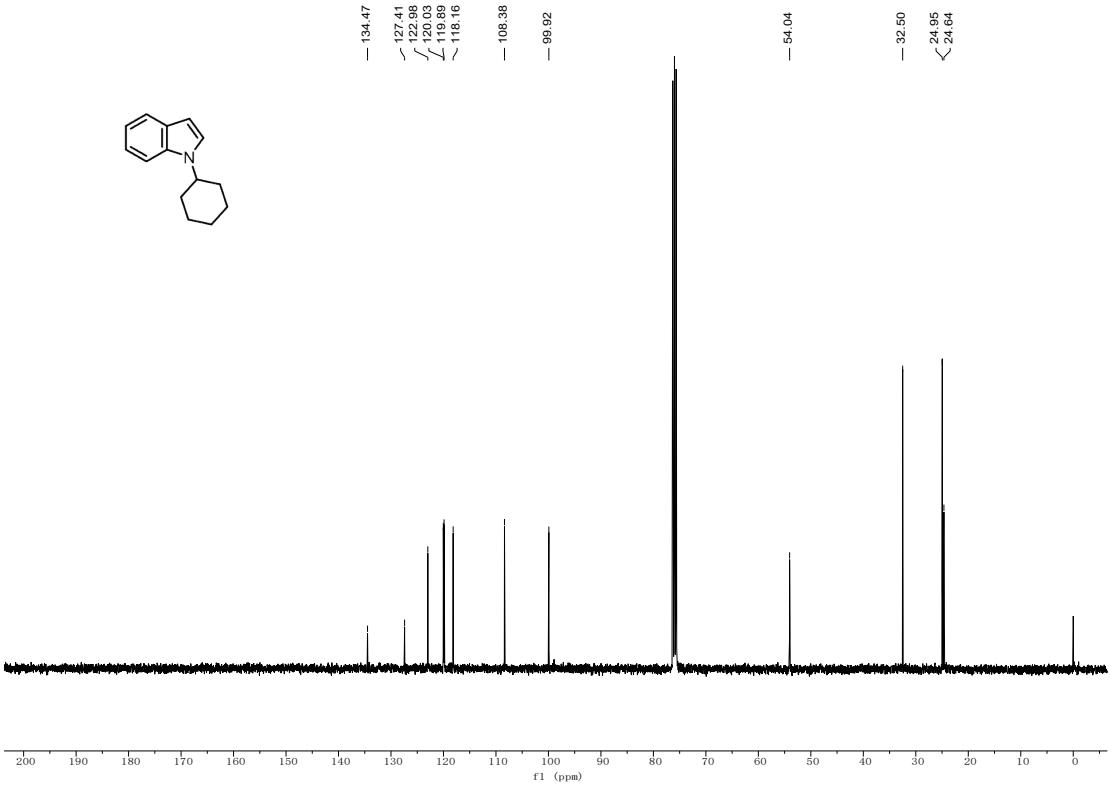
1-hexyl-1*H*-indole (3ad)



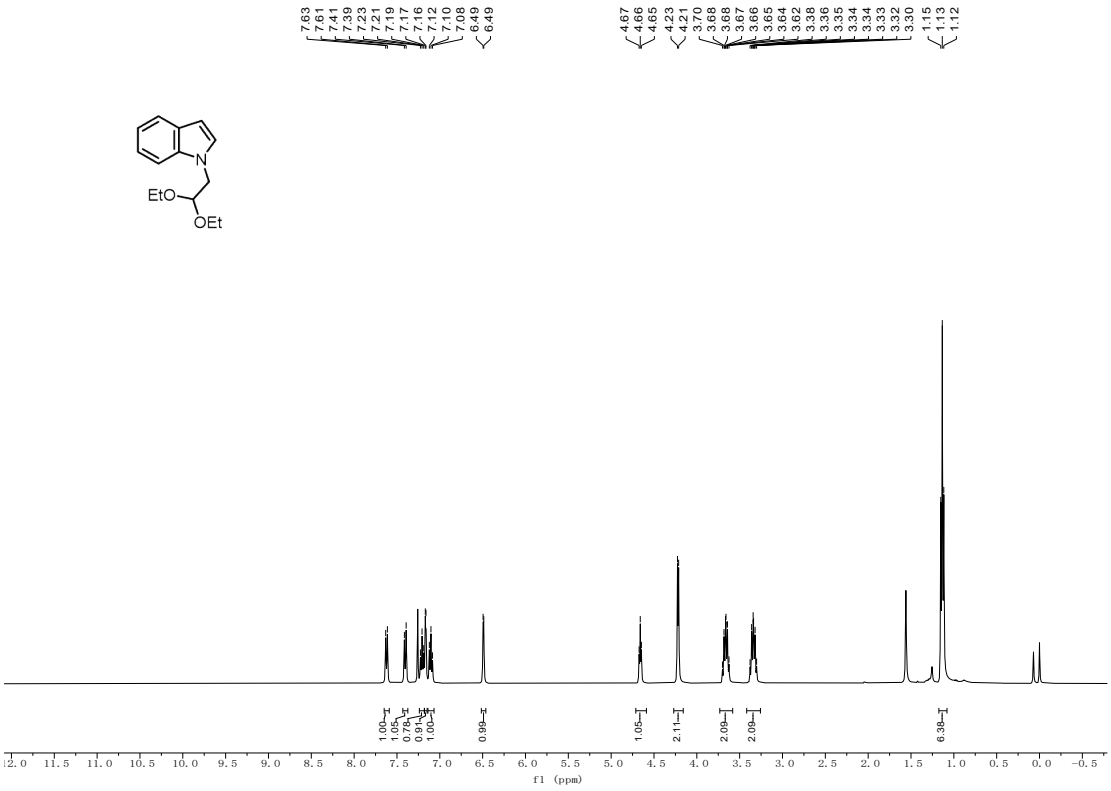


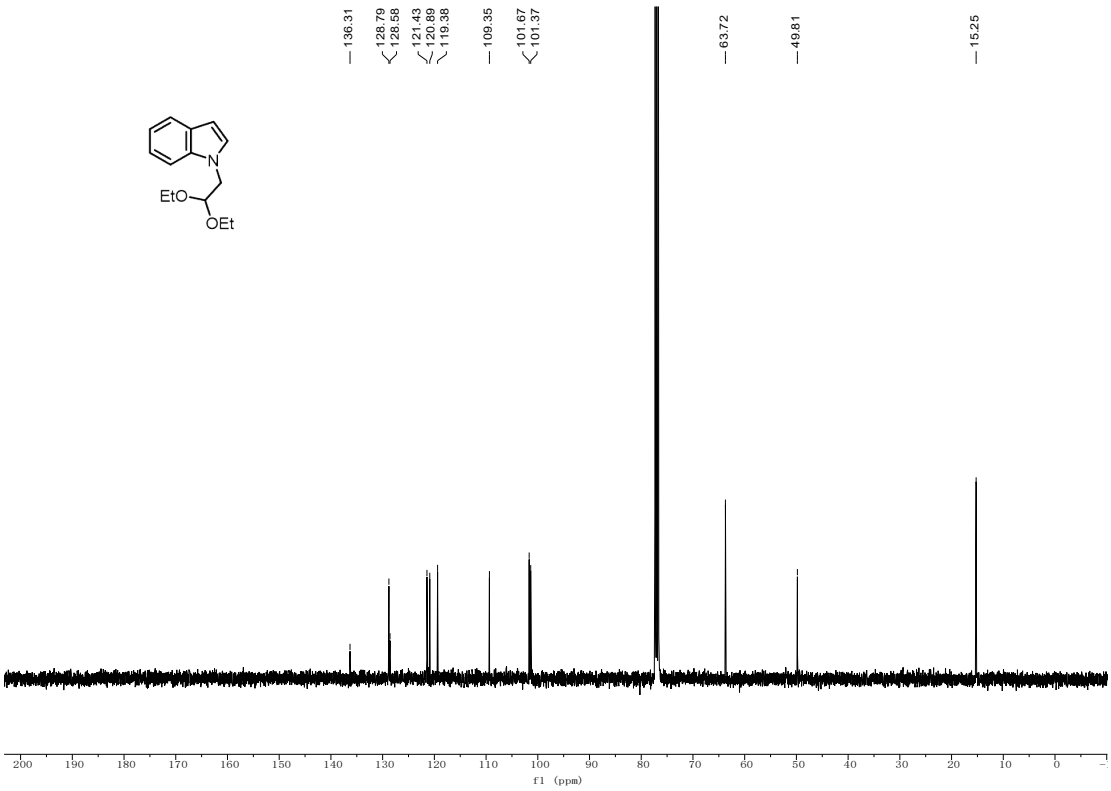
1-cyclohexyl-1*H*-indole (3ae)



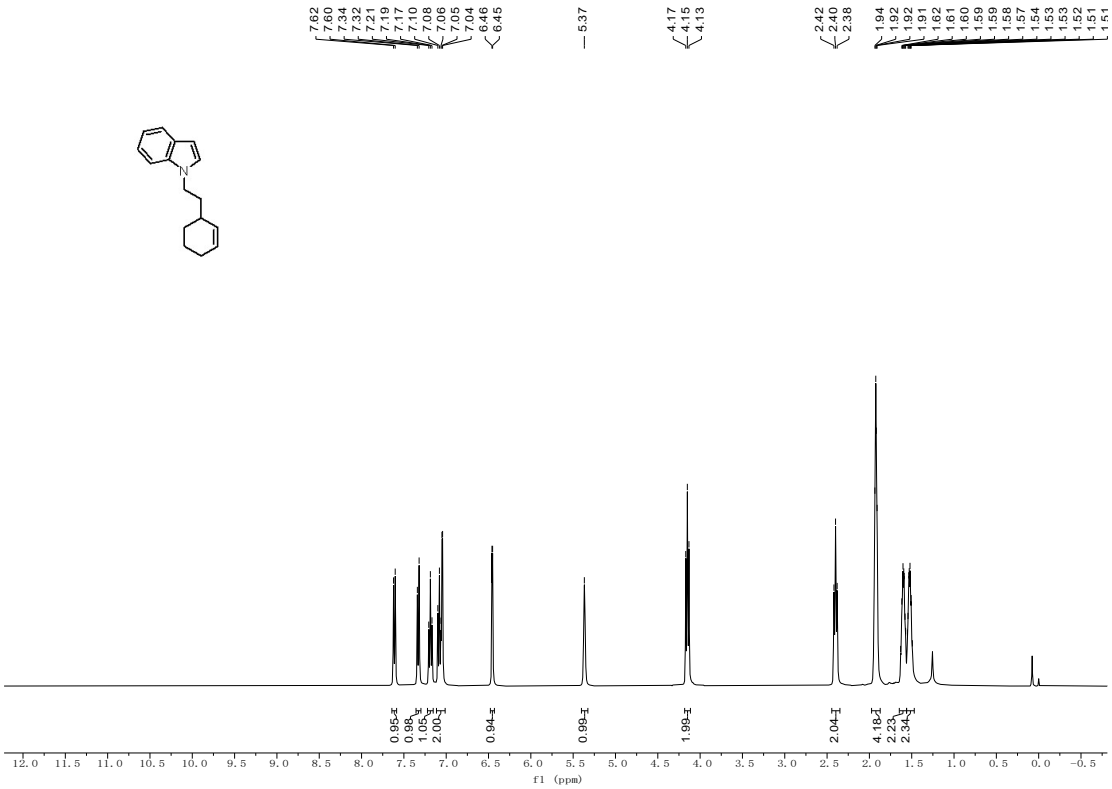


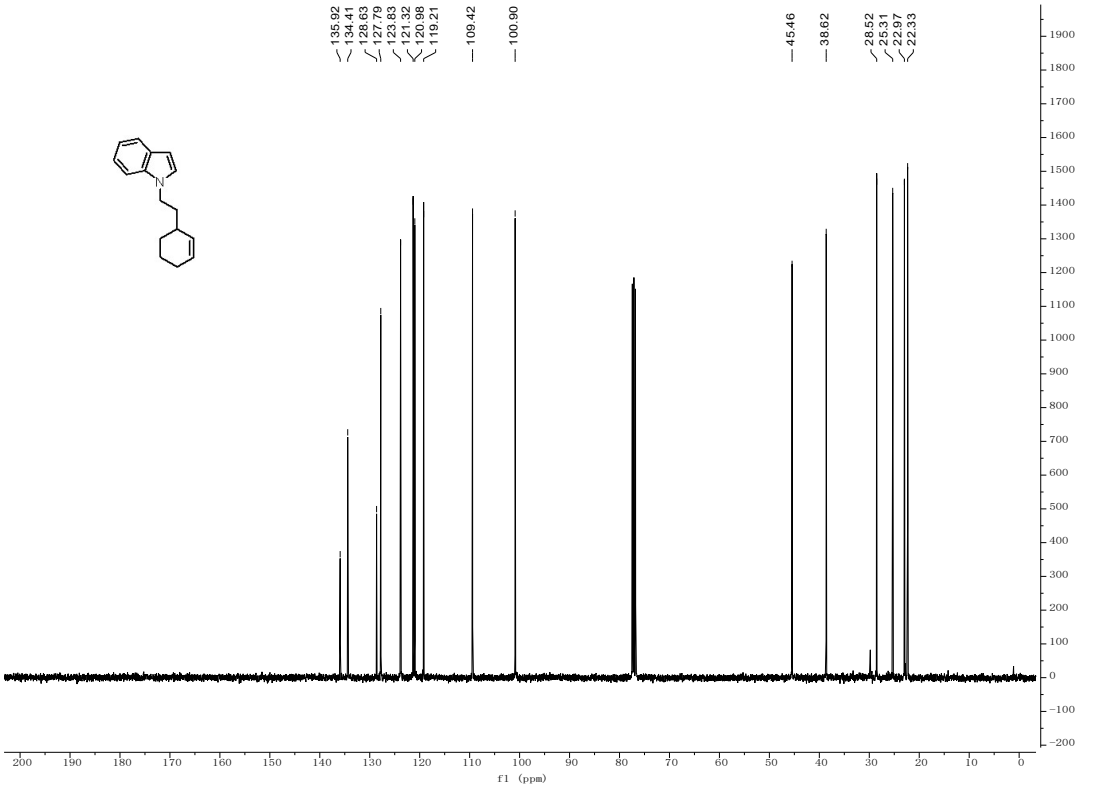
1-(2,2-diethoxyethyl)-1*H*-indole (3af)



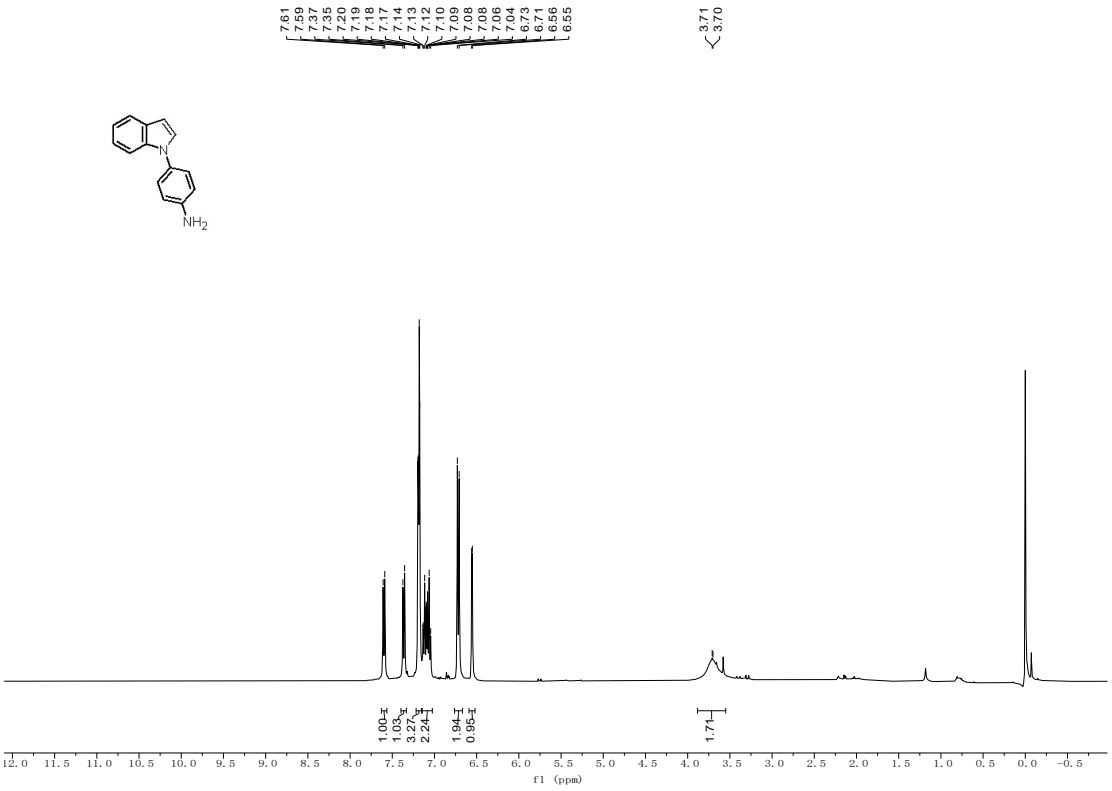


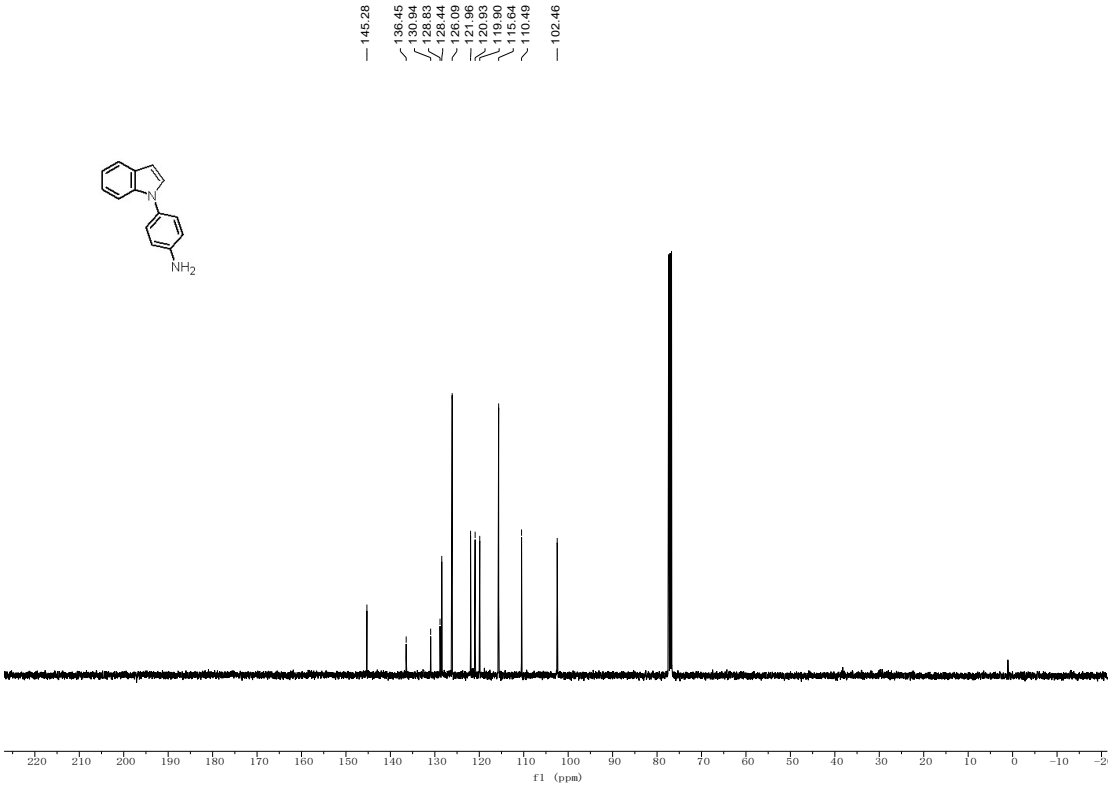
1-(2-(cyclohex-2-en-1-yl)ethyl)-1*H*-indole (3ag)



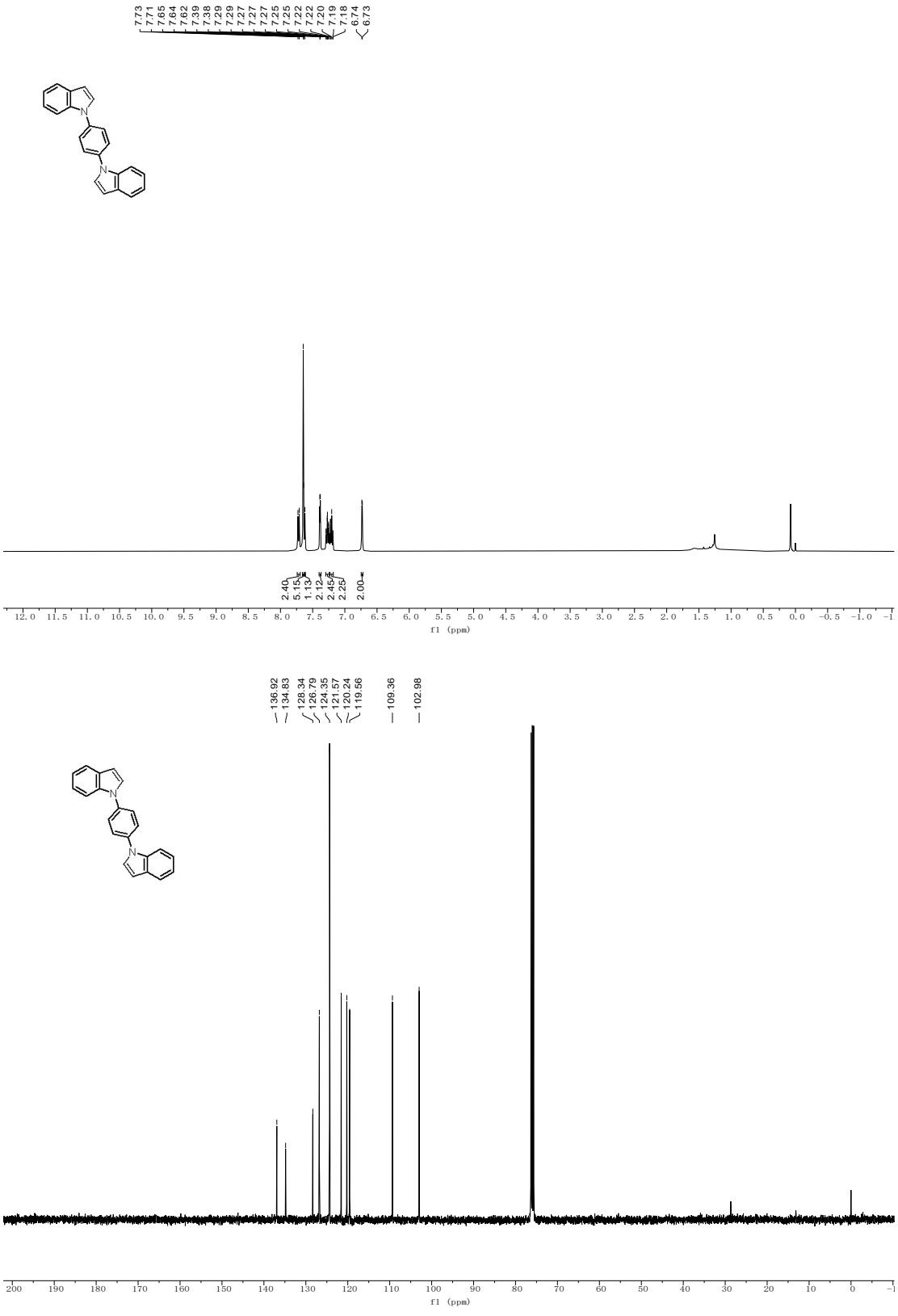


4-(1*H*-indol-1-yl)aniline (3ah)

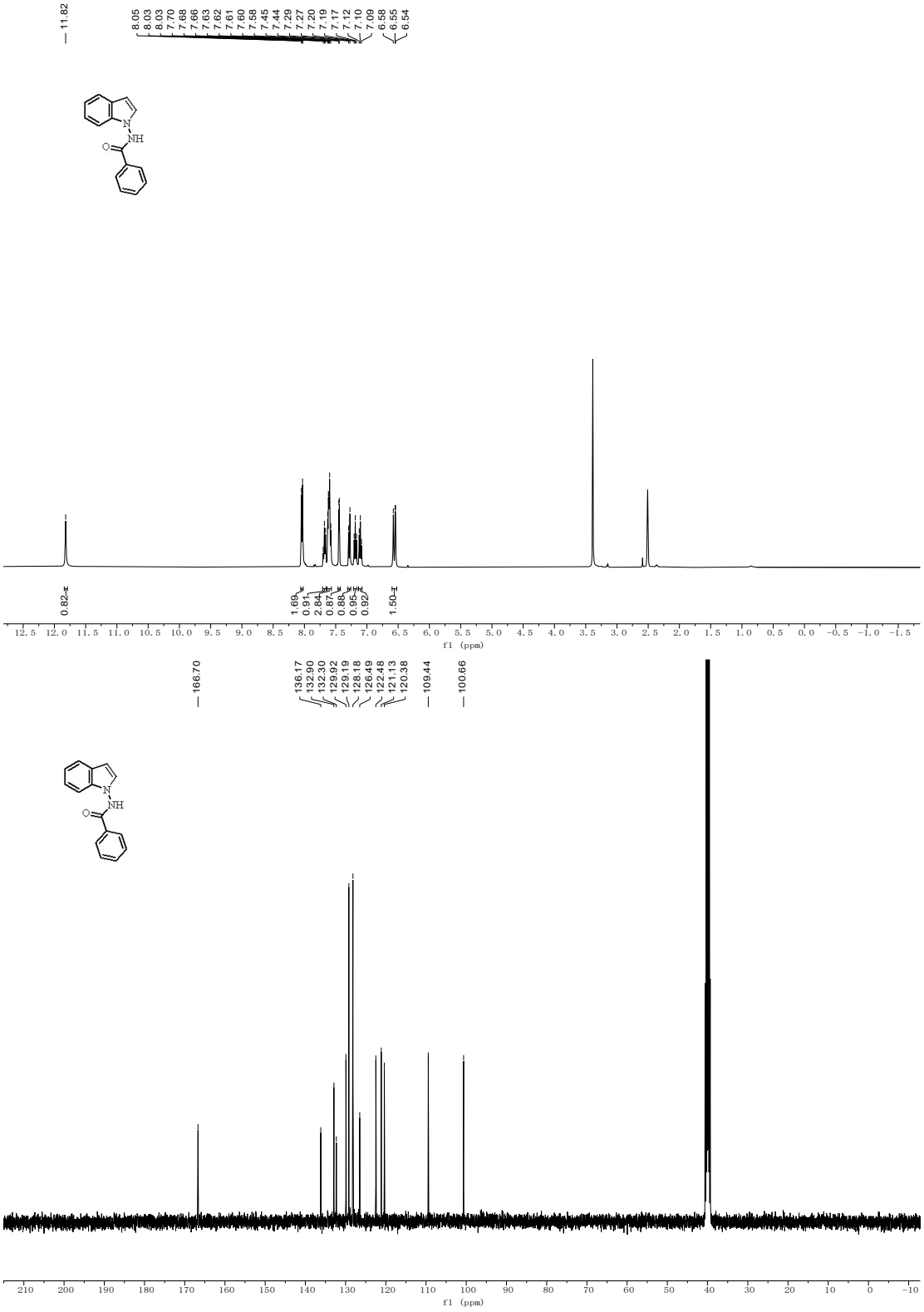




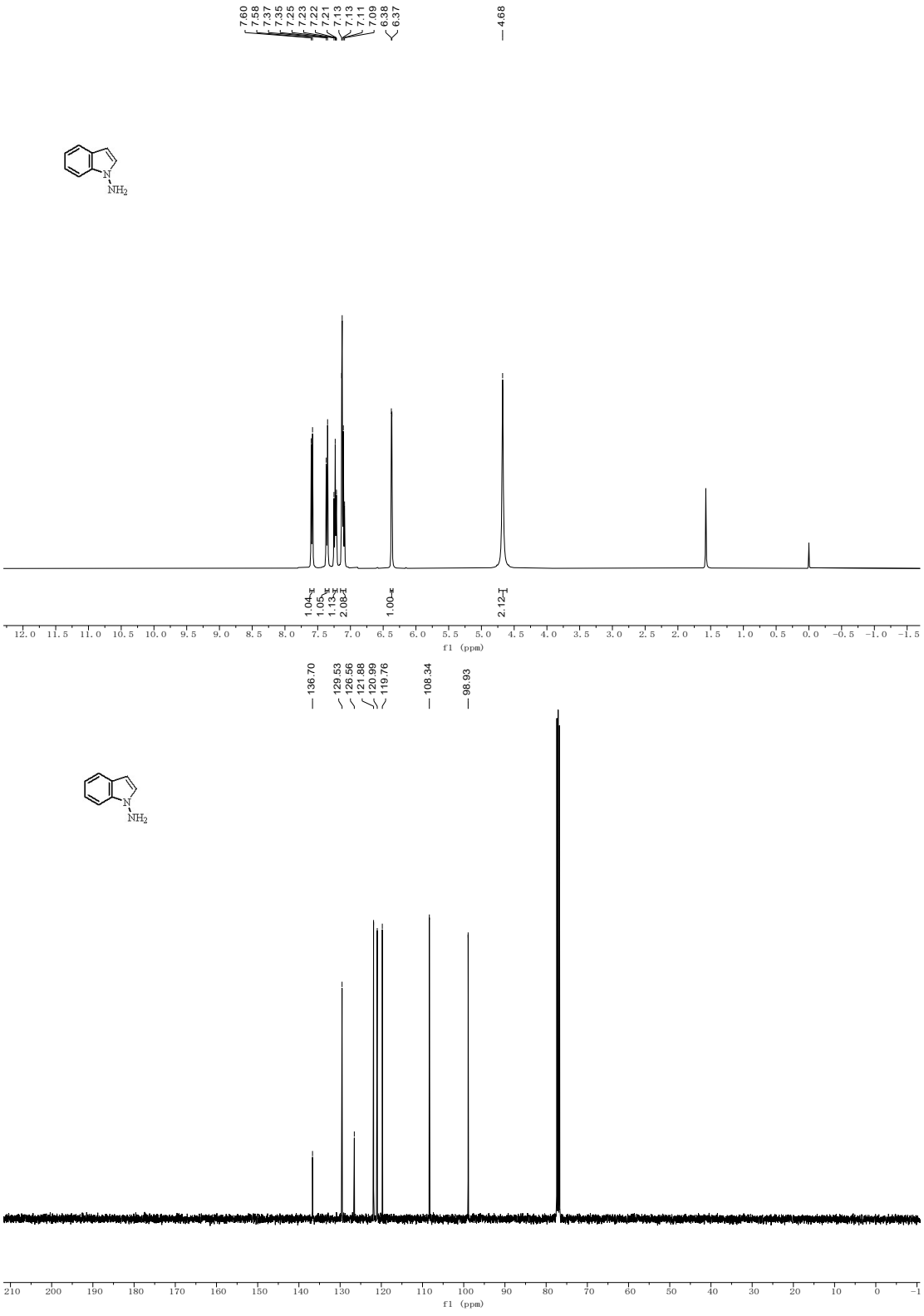
1,4-di(1*H*-indol-1-yl)benzene (3ai)



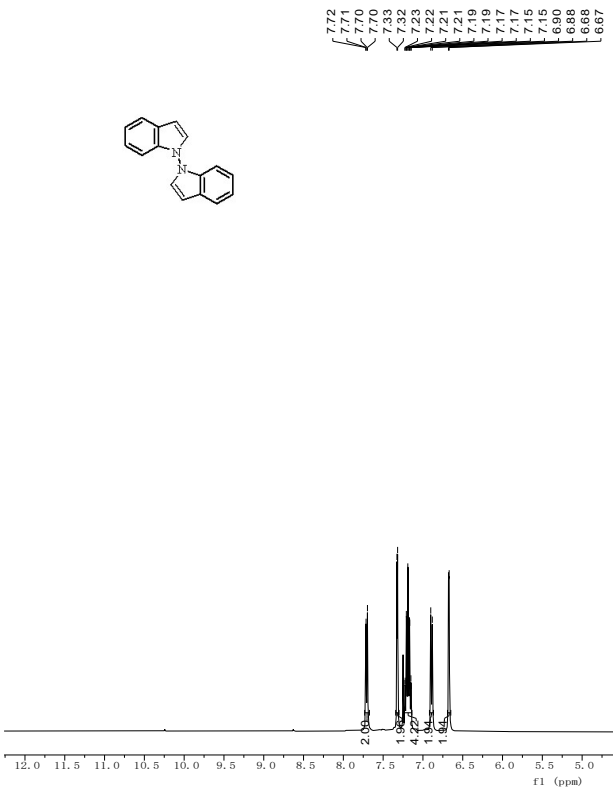
_N_-(1_H_-indol-1-yl)benzamide (5a)



1*H*-indol-1-amine (5b)



1,1'-biindole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7.70
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7.32
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7.22
7.21
7.21
7.19
7.19
7.17
7.17
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7.15
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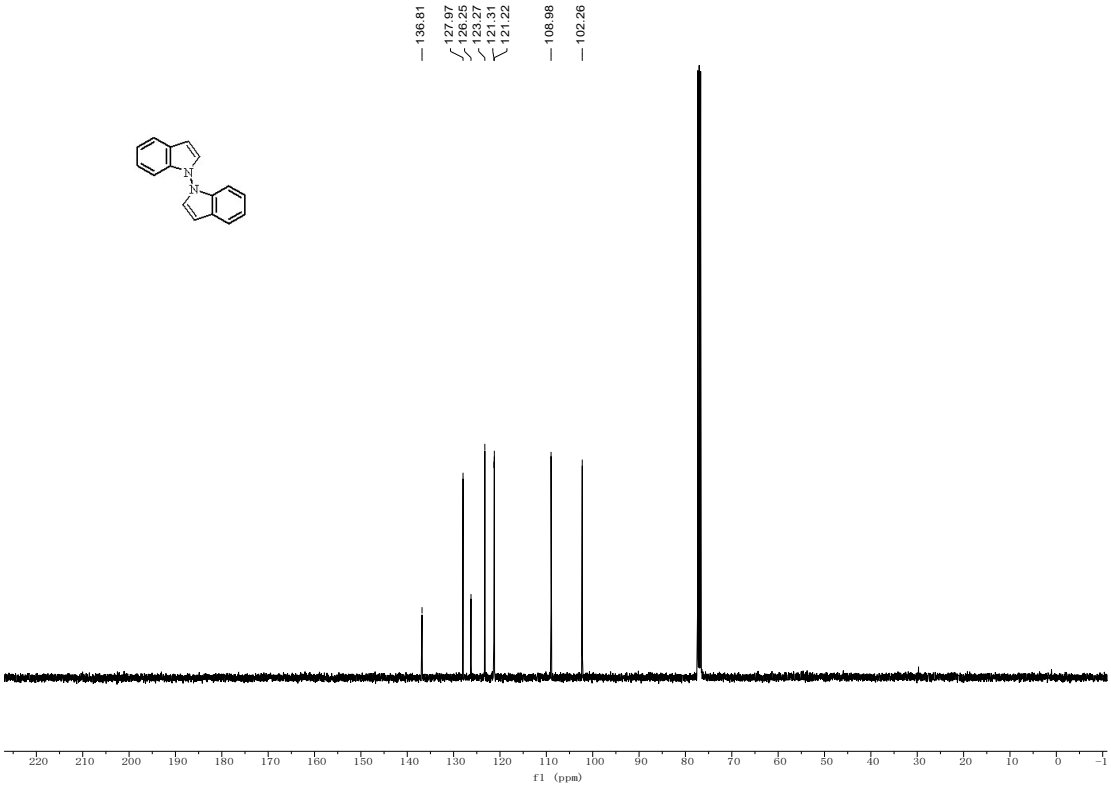
6.88
6.68
6.67
2.00
1.96
4.22
1.94
1.94
f1 (ppm)


136.81
127.97
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123.27
121.31
121.22
108.98
102.26
f1 (ppm)