Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

A sustainable synthesis of 2-aryl-3-carboxylate indolines from *N*-aryl enamines under visible light irradiation

Cheng-Juan Wu,[a] Wen-Xiao Cao,[a] Tao Lei,[a] Zhi-Hua Li,[a] Qing-Yuan Meng,[a] Xiu-Long Yang,[a] Bin Chen,[a] Vaidhyanathan Ramamurthy,[b] Chen-Ho Tung[a] and Li-Zhu Wu*[a]

[a] Key Laboratory of Photochemical Conversion and Optoelectronic Materials, Technical Institute of Physics and Chemistry & University of Chinese Academy of Sciences, the Chinese Academy of Sciences, Beijing, 100190, P. R. China

[b] Department of Chemistry, University of Miami, 1301 Memorial Drive, Miami, FL 33124-0431, USA

Email: *lzwu@mail.ipc.ac.cn*

Table of contents

1. Experimental details

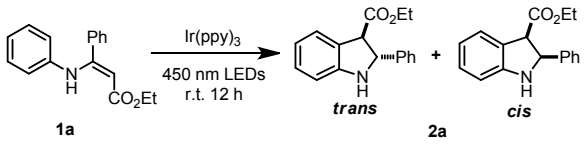
(a) General information:

^{1}H NMR spectra were recorded using a Bruker Avance DPX 400MHz instrument with tetramethylsilane (TMS) as an internal standard. ^{13}C NMR spectra were obtained at 100 MHz and referenced to the internal solvent signals. Mass spectra were recorded using a Trio-2000 GC-MS spectrometer. Blue LEDs (3 W, λ = 450 ± 10 nm, 145 lm @700mA) were used as the irradiation light. UV-Vis absorption spectra were recorded with a Shimadzu 1601PC spectrophotometer. Steady-state emission spectra were recorded using a Perkin–Elmer LS50B spectrofluorimeter. The values of lifetime were calculated by exponential function fitting with luminescence spectrometer software L900. Commercially available reagents and solvents were used without further purification.

(b) Preparation of *N*-aryl enamine substrates:

All the *N*-aryl enamine substrates needed for the indoline synthesis reactions were prepared using the reported procedure.[1-4] A mixture of ethyl benzoylacetate (1 mmol), aniline (4 mmol) and acetic acid (4 mmol) was stirred at 80 °C until TLC indicated the total consumption of the ethyl benzoylacetate. The reaction was cooled to room temperature, and the solvent was evaporated and the residue was purified by column chromatography on silica gel to get the *N*-aryl enamine substrates.

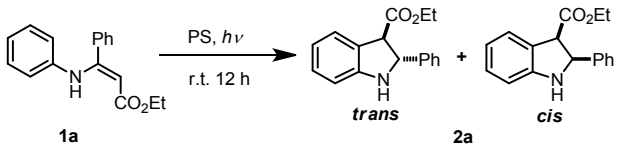
(c) General procedure for the indoline synthesis reaction:



A 10 mL Pyrex tube equipped with a magnetic stir bar was charged with *N*-aryl enamine **1a** (0.2 mmol), Ir(ppy)$_3$ (2 mol%) in 4 mL CH_3CN. The mixture was strictly deaerated and irradiated by blue LEDs (λ = 450 nm) for 12 hours at room temperature. After reaction, the solution was concentrated *in vacuo*. The diastereomer ratios were determined by ^{1}H NMR analysis of the unpurified reaction mixture, and the yield was determined using diphenylacetonitrile as an internal standard. The residue was purified by column chromatography on silica gel to get the products.

NOTE: *During the purification process by chromatography on silica gel, the indoline product was easily to be oxidized to indoles. Thus the isolated yield of the product was much less than the yield detected by ^{1}H NMR.*

Table S1. Optimization of the reaction conditions [a]

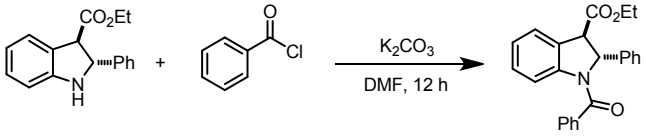


1a 2a

entry	PS	solvent	product
1	2 mol% $Ru(bpy)_3Cl_2$	4 mL CH_3CN	N.R.
2[b]	2 mol% Eosin Y	4 mL CH_3CN	N.R.
3	2 mol% Acr^+-Mes	4 mL CH_3CN	N.R.
4[c]	--	4 mL CH_3CN	N.R.

[a]Reaction conditions: 0.2 mmol **1a**, 2 mol% of photosensitizer (PS) were added in 4 mL CH_3CN, and the solution was strictly deaerated with N_2 and irradiated under blue LEDs (λ = 450 nm) for 12 hours at room temperature. [b]Green LEDs (λ = 525 nm) was used. [c]Mercury lamp (λ > 300 nm) was used.

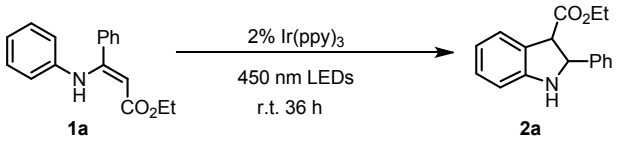
Scheme S1. Synthesis of 3a.



2a (major) 3a

X-ray crystallographic analysis was measured to determine the structure of two diastereomers. The major diastereomer was derived to **3a** firstly, which was then unambiguously established to be *trans*-indoline (Figure 1). **3a** was synthesized according to a reported literature method.[5] K_2CO_3 (0.88 mmol) was dissolved in dry DMF (1 mL) and the resulting mixture was cooled to 0 °C. Indoline **2a** (0.74 mmol) solution was added dropwise to the above mixture at 0 °C and stirred at room temperature for 30 min. Then benzoyl chloride (0.80 mmol) was added dropwise to the mixture at 0 °C. The resulting mixture was stirred at room temperature for 12 h, and TLC showed complete consumption of the starting indoline **2a**. After the reaction, the solution was extracted with ethyl acetate and water. The organic phase were combined together and washed with brine and dried over anhydrous sodium sulphate. Upon removal of solvent under vacuum, the residue was purified by chromatography on silica gel to get the product **3a**

Scheme S2. Gram-scale reaction of the synthesis of indolines[a]



1a 2a

4 mmol 1.068g isolated yield: 68%

[a]Reaction conditions: 4 mmol **1a**, 2% $Ir(ppy)_3$ were added in 50 mL CH_3CN, the solution was strictly deaerated with N_2, and irradiated under blue LEDs (λ = 450 nm) for 36 hours at room temperature.

2. Crystal structural data for 3a

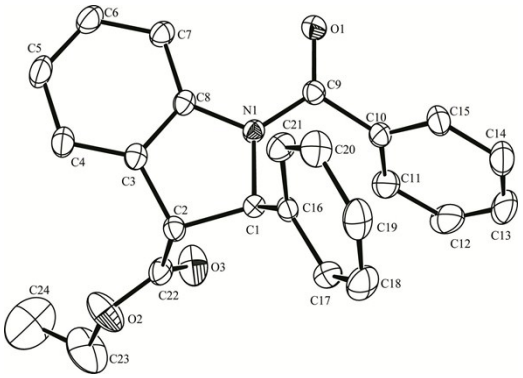


Figure S1. X-ray crystal structure of compound **3a** (CCDC No.1542178)

Empirical formula	$C_{24}H_{21}NO_3$
Formula weight	371.42
Temperature	179.99(10) K
Crystal system	monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 17.0796(14) Å α = 90°
	b = 13.6704(8) Å β = 116.272(9)°
	c = 18.9052(13) Å γ = 90°
Volume	3958.1(6) $Å^3$
Z	8
Density (calculated)	1.247 g/cm^3
Absorption coefficient	0.082 mm^{-1}
F(000)	1568.0
Crystal size	0.15 × 0.1 × 0.1 mm^3
2Θ range for data collection	7.1 to 52.038°
Index ranges	$-21 \le h \le 21$, $-16 \le k \le 15$, $-23 \le l \le 23$
Reflections collected	32710
Independent reflections	7752 [R_{int} = 0.0437, R_{sigma} = 0.0363]
Data/restraints/parameters	7752/0/507
Goodness-of-fit on F^2	1.029
Final R indexes [I>=2σ (I)]	R_1 = 0.0592, wR_2 = 0.1610
Final R indexes [all data]	R_1 = 0.0746, wR_2 = 0.1741
Largest diff. peak and hole	0.45 and -0.35 e.$Å^{-3}$

3. Mechanism study

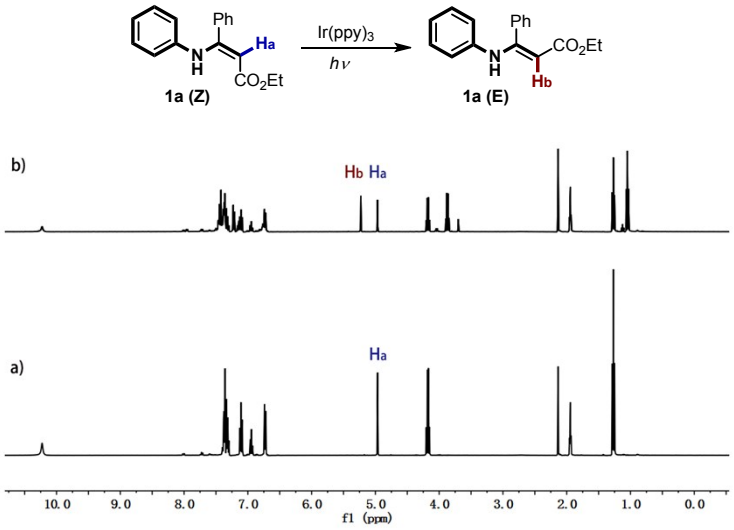
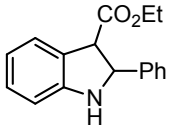


Figure S2. ^{1}H-NMR of system containing Ir(ppy)$_3$ (8 ×10^{-4} M) and **1a** (4 ×10^{-2} M) in degassed CD_3CN a) before irradiation, and b) after irradiated by blue LEDs for 1 minute.

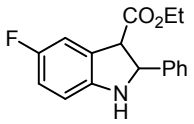
4. Characterization data for all compounds



2a

Ethyl 2-phenylindoline-3-carboxylate (2a)

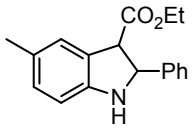
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 70%, 4.0:1 dr), R_f (pentane/EtOAc 4:1) = 0.6; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.38 (m, 2H), 7.33 – 7.19 (m, 3H), 7.15 – 7.01 (m, 2H), 6.78 – 6.62 (m, 2H), 5.38 (s, 1H), 5.26 (d, *J* = 10.0 Hz, 1H), 4.48 (d, *J* = 10.0 Hz, 1H), 3.79 – 3.49 (m, 2H), 0.80 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 169.99, 152.61, 140.71, 128.32, 127.77, 127.39, 127.35, 125.88, 125.62, 117.94, 108.88, 65.56, 59.63, 53.58, 13.18. HRMS (ESI) calculated for $C_{17}H_{17}NO_2$ [M + H]: 268.1338, Found: 268.1328. **(*cis*) Diastereomer** (minor): ^{1}H NMR (400 MHz, Acetone) δ 7.47 (m, 2H), 7.35 (t, *J* = 7.4 Hz, 2H), 7.28 (m, 1H), 7.18 (m, 1H), 7.08 (t, *J* = 7.6 Hz, 1H), 6.68 (m, 2H), 5.57 (s, 1H), 5.32 (d, *J* = 9.3 Hz, 1H), 4.43 – 4.09 (m, 2H), 3.96 (d, *J* = 9.3 Hz, 1H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.65, 150.25, 143.21, 128.92, 128.67, 127.79, 126.70, 125.09, 124.69, 118.98, 109.35, 65.19, 61.28, 56.66, 14.32. HRMS (ESI) calculated for $C_{17}H_{17}NO_2$ [M + H]: 268.1338, Found: 268.1324



2b

Ethyl 5-fluoro-2-phenylindoline-3-carboxylate (2b)

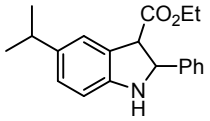
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 42%, 3.7:1 dr), R_f (pentane/EtOAc 4:1) = 0.5; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.36 (m, 2H), 7.31 – 7.18 (m, 3H), 6.93 (d, *J* = 8.5 Hz, 1H), 6.84 (m, 1H), 6.67 (m, 1H), 5.33 (s, 1H), 5.29 (d, *J* = 10.0 Hz, 1H), 4.51 (d, *J* = 10.0 Hz, 1H), 3.65 (m, 2H), 0.80 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 169.58, 156.44 (d, $J_{(C,F)}$ = 231 Hz), 148.99, 140.52, 127.84, 127.54, 127.46 (d, $J_{(C,F)}$ = 9 Hz), 127.30, 114.26 (d, $J_{(C,F)}$ = 23 Hz), 112.95 (d, $J_{(C,F)}$ = 25 Hz), 109.00 (d, *J* = 8 Hz), 66.20, 59.82, 53.57, 13.13. HRMS (ESI) calculated for $C_{17}H_{16}FNO_2$ [M + H]: 286.1243, Found: 286.1232.



2c

Ethyl 5-methyl-2-phenylindoline-3-carboxylate (2c)

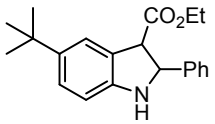
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 67%, 4.1:1 dr), R_f (pentane/EtOAc 4:1) = 0.6; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.46 – 7.33 (m, 2H), 7.32 – 7.15 (m, 3H), 6.94 (s, 1H), 6.89 (m, 1H), 6.61 (d, *J* = 7.9 Hz, 1H), 5.23 (d, *J* = 10.0 Hz, 1H), 5.18 (s, 1H), 4.43 (d, *J* = 9.9 Hz, 1H), 3.64 (m, 2H), 2.22 (s, 3H), 0.80 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.19, 150.53, 141.07, 128.86, 127.89, 127.52, 127.47, 127.08, 126.36, 109.00, 66.00, 59.72, 53.88, 20.15, 13.33. HRMS (ESI) calculated for $C_{18}H_{19}NO_2$ [M + H]: 282.1494, Found: 282.1482.



2d

Ethyl 5-isopropyl-2-phenylindoline-3-carboxylate (2d)

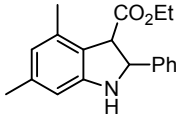
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 64%, 4.2:1 dr), R_f (pentane/EtOAc 4:1) = 0.5; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.37 (m, 2H), 7.32 – 7.10 (m, 3H), 7.01 (s, 1H), 6.95 (d, *J* = 8.0 Hz, 1H), 6.63 (d, *J* = 8.0 Hz, 1H), 5.24 (d, *J* = 10.0 Hz, 1H), 5.19 (s, 1H), 4.43 (d, *J* = 10.0 Hz, 1H), 3.62 (m, 2H), 2.89 – 2.72 (m, 1H), 1.19 (d, *J* = 6.9 Hz, 6H), 0.78 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.11, 150.63, 140.86, 138.66, 127.76, 127.39, 127.33, 126.16, 126.14, 123.55, 108.92, 65.84, 59.57, 53.72, 33.47, 24.01, 13.21. HRMS (ESI) calculated for $C_{20}H_{23}NO_2$ [M + H]: 310.1807, Found: 310.1794.



2e

Ethyl 5-(tert-butyl)-2-phenylindoline-3-carboxylate (2e)

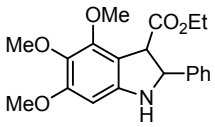
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 69%, 5.5:1 dr), R_f (pentane/EtOAc 4:1) = 0.4; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.37 (d, *J* = 7.3 Hz, 2H), 7.30 – 7.20 (m, 3H), 7.18 (s, 1H), 7.12 (d, *J* = 8.2 Hz, 1H), 6.64 (d, *J* = 8.2 Hz, 1H), 5.24 (d, *J* = 10.0 Hz, 1H), 5.19 (s, 1H), 4.43 (d, *J* = 10.0 Hz, 1H), 3.78 – 3.46 (m, 2H), 1.27 (s, 9H), 0.78 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.13, 150.20, 140.86, 127.74, 127.38, 127.31, 125.88, 125.03, 122.50, 108.58, 65.85, 59.55, 53.82, 33.81, 31.27, 13.21. HRMS (ESI) calculated for $C_{21}H_{25}NO_2$ [M + H]: 324.1964, Found: 324.1951.



2f

Ethyl 4,6-dimethyl-2-phenylindoline-3-carboxylate (2f)

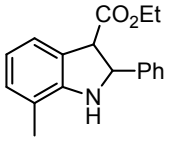
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 76%, 3.3:1 dr), R_f (pentane/EtOAc 4:1) = 0.5; **(*trans*) Diastereomer** (major) and **(*cis*) Diastereomer** as an inseparable mixture: ^{1}H NMR (400 MHz, Acetone) δ 7.48 (m, 6.6H$_{trans}$), 7.41 – 7.20 (m, 14.9H$_{overlap}$), 6.39 (s, 4.3H$_{overlap}$), 6.32 (s, 4.3H$_{overlap}$), 5.30 (s, 3.3H$_{trans}$), 5.28 (s, 3.3H$_{trans}$), 5.14 (s, 1H$_{cis}$), 5.12 (s, 1H$_{cis}$), 4.30 (d, J = 10.1 Hz, 3.3H$_{trans}$), 4.20 (q, J = 7.1 Hz, 2H$_{cis}$), 3.89 (d, J = 6.8 Hz, 1H$_{cis}$), 3.57 (m, 6.6H$_{trans}$), 2.21 (s, 12.9H$_{overlap}$), 2.09 (s, 3H$_{cis}$), 2.05 (s, 9.9H$_{trans}$), 1.25 (t, J = 7.1 Hz, 3H$_{cis}$), 0.76 (t, J = 7.1 Hz, 9.9H$_{trans}$). ^{13}C NMR (101 MHz, Acetone) δ 172.78, 170.17, 152.81, 152.03, 144.26, 140.34, 138.38, 138.10, 134.40, 134.10, 128.53, 127.70, 127.64, 127.47, 127.25, 126.11, 122.85, 121.29, 120.73, 120.37, 107.86, 107.43, 66.86, 65.93, 60.61, 59.57, 56.79, 52.88, 20.85, 20.78, 18.01, 17.75, 13.74, 13.28. HRMS (ESI) calculated for $C_{19}H_{21}NO_2$ [M + H]: 296.1651, Found: 296.1640.



2g

Ethyl 4,5,6-trimethoxy-2-phenylindoline-3-carboxylate (2g)

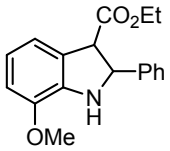
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 78%, >19:1 dr), R_f (pentane/EtOAc 2:1) = 0.3; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.44 (m, 2H), 7.34 – 7.20 (m, 3H), 6.19 (s, 1H), 5.30 (d, J = 10.3 Hz, 1H), 5.19 (s, 1H), 4.35 (d, J = 10.2 Hz, 1H), 3.77 (s, 6H), 3.68 (s, 3H), 3.63 – 3.40 (m, 2H), 0.74 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.99, 155.71, 151.09, 149.66, 140.84, 135.08, 128.28, 128.26, 127.91, 111.00, 91.19, 66.88, 60.69, 60.12, 60.04, 56.19, 52.61, 13.79. HRMS (ESI) calculated for $C_{20}H_{23}NO_5$ [M + H]: 358.1654, Found: 358.1642.



2h

Ethyl 7-methyl-2-phenylindoline-3-carboxylate (2h)

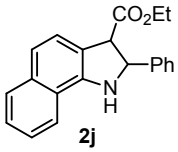
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 70%, 4.0:1 dr), R_f (pentane/EtOAc 4:1) = 0.6; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.08 (m, 5H), 6.96 (d, J = 7.4 Hz, 1H), 6.89 (d, J = 7.5 Hz, 1H), 6.65 (t, J = 7.4 Hz, 1H), 5.14 (d, J = 10.0 Hz, 1H), 4.43 (d, J = 10.0 Hz, 1H), 3.79 (s, 1H), 3.71 – 3.50 (m, 2H), 2.09 (s, 3H), 0.72 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.56, 149.98, 140.27, 129.66, 128.30, 128.00, 127.14, 124.89, 123.53, 119.56, 118.87, 65.64, 60.47, 54.07, 16.95, 13.73. HRMS (ESI) calculated for $C_{18}H_{19}NO_2$ [M + H]: 282.1494, Found: 282.1479.



2i

Ethyl 7-methoxy-2-phenylindoline-3-carboxylate (2i)

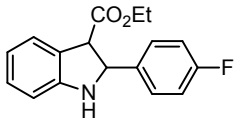
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 71%, 4.0:1 dr), R_f (pentane/EtOAc 4:1) = 0.6; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.48 – 7.36 (m, 2H), 7.34 – 7.18 (m, 3H), 6.78 (m, 2H), 6.69 (m, 1H), 5.30 (d, J = 10.0 Hz, 1H), 4.90 (s, 1H), 4.48 (d, J = 10.0 Hz, 1H), 3.83 (s, 3H), 3.74 – 3.48 (m, 2H), 0.78 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.59, 145.81, 142.22, 141.25, 128.39, 128.02, 127.96, 127.49, 119.50, 118.60, 111.29, 66.77, 60.25, 55.60, 54.89, 13.82. HRMS (ESI) calculated for $C_{18}H_{19}NO_3$ [M + H]: 298.1443, Found: 298.1433.



2j

Ethyl 2-phenyl-2,3-dihydro-1H-benzo[g]indole-3-carboxylate (2j)

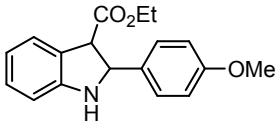
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 72%, 4.0:1 dr), R_f (pentane/EtOAc 4:1) = 0.7; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.99 (d, J = 7.2 Hz, 1H), 7.84 (d, J = 7.2 Hz, 1H), 7.48 – 7.37 (m, 4H), 7.37 – 7.19 (m, 5H), 6.02 (s, 1H), 5.50 (d, J = 10.5 Hz, 1H), 4.70 (d, J = 10.5 Hz, 1H), 3.64 (m, 2H), 0.79 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.29, 148.69, 140.62, 134.39, 128.21, 127.83, 127.51, 127.46, 125.55, 124.41, 124.03, 122.52, 120.47, 119.18, 117.88 , 66.07, 59.68, 54.49, 13.19. HRMS (ESI) calculated for $C_{21}H_{19}NO_2$ [M + H]: 318.1494, Found: 318.1476.



2k

Ethyl 2-(4-fluorophenyl)indoline-3-carboxylate (2k)

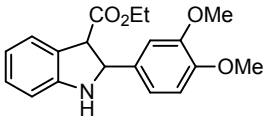
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 45%, 3.7:1 dr), R_f (pentane/EtOAc 4:1) = 0.5; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.42 (m, 2H), 7.21 – 6.92 (m, 4H), 6.68 (m, 2H), 5.44 (s, 1H), 5.29 (d, J = 10.0 Hz, 1H), 4.47 (d, J = 10.0 Hz, 1H), 3.67 (m, 2H), 0.84 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.88, 163.13 (d, $J_{(C,F)}$ = 242 Hz), 153.39, 137.69 (d, J = 3 Hz), 130.14 (d, J = 8 Hz), 129.30, 126.64, 126.55, 118.95, 115.30 (d, $J_{(C,F)}$ = 21 Hz), 109.81, 65.70, 60.63, 54.45, 14.13. HRMS (ESI) calculated for $C_{17}H_{16}FNO_2$ [M + H]: 286.1243, Found: 286.1234.



2l

Ethyl 2-(4-methoxyphenyl)indoline-3-carboxylate (2l)

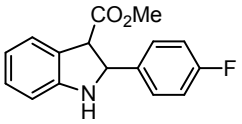
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 62%, 4.4:1 dr), R_f (pentane/EtOAc 4:1) = 0.6; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.28 (d, *J* = 8.2 Hz, 2H), 7.15 – 6.99 (m, 2H), 6.83 (d, *J* = 8.2 Hz, 2H), 6.74 – 6.57 (m, 2H), 5.30 (s, 1H), 5.20 (d, *J* = 10.0 Hz, 1H), 4.42 (d, *J* = 10.0 Hz, 1H), 3.75 (s, 3H), 3.65 (m, 2H), 0.85 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.06, 159.36, 152.63, 132.58, 128.44, 128.27, 125.85, 125.71, 117.86, 113.15, 108.76, 65.07, 59.63, 54.64, 53.59, 13.28. HRMS (ESI) calculated for $C_{18}H_{19}NO_3$ [M + H]: 298.1443, Found: 298.1431.



2m

Ethyl 2-(3,4-dimethoxyphenyl)indoline-3-carboxylate (2m)

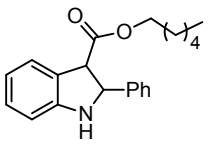
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 70%, 4.0:1 dr), R_f (pentane/EtOAc 4:1) = 0.6; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.16 – 6.96 (m, 3H), 6.95 – 6.79 (m, 2H), 6.75 – 6.60 (m, 2H), 5.32 (s, 1H), 5.20 (d, *J* = 10.0 Hz, 1H), 4.41 (d, *J* = 10.0 Hz, 1H), 3.75 (s, 3H), 3.71 (s, 3H), 3.71 – 3.60 (m, 2H), 0.84 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.19, 152.56, 149.08, 149.00, 133.11, 128.29, 126.10, 125.61, 119.66, 117.98, 111.61, 111.55, 108.97, 65.46, 59.66, 55.36, 55.24, 53.62, 13.32. HRMS (ESI) calculated for $C_{19}H_{21}NO_4$ [M + H]: 328.1549, Found: 328.1539.



2n

Eethyl 2-(4-fluorophenyl)indoline-3-carboxylate (2n)

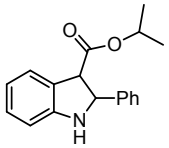
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 48%, 3.5:1 dr), R_f (pentane/EtOAc 4:1) = 0.5; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.41 (t, *J* = 6.4 Hz, 2H), 7.17 – 6.91 (m, 4H), 6.79 – 6.54 (m, 2H), 5.46 (s, 1H), 5.29 (d, *J* = 10.0 Hz, 1H), 4.48 (d, *J* = 10.0 Hz, 1H), 3.21 (s, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.40, 162.16 (d, $J_{(C,F)}$ = 242 Hz), 152.52, 136.66 (d, $J_{(C,F)}$ = 3 Hz), 129.11 (d, $J_{(C,F)}$ = 8 Hz), 128.46, 125.61, 125.58, 118.09, 114.42 (d, $J_{(C,F)}$ = 21 Hz), 108.95, 64.85, 53.73, 50.45. HRMS (ESI) calculated for $C_{16}H_{14}FNO_2$ [M + H]: 272.1087, Found: 272.1076.



2o

Propyl 2-phenylindoline-3-carboxylate (2o)

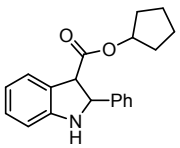
The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 30%, 3.6:1 dr), R_f (pentane/EtOAc 4:1) = 0.5; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.38 (m, 2H), 7.33 – 7.17 (m, 3H), 7.17 – 6.98 (m, 2H), 6.76 – 6.60 (m, 2H), 5.39 (s, 1H), 5.27 (d, *J* = 10.0 Hz, 1H), 4.48 (d, *J* = 10.0 Hz, 1H), 3.74 – 3.43 (m, 2H), 1.29 – 1.09 (m, 8H), 0.86 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.02, 152.56, 140.69, 128.30, 127.79, 127.38, 127.31, 126.02, 125.54, 117.95, 108.94, 65.63, 63.83, 53.67, 31.21, 28.08, 25.29, 22.25, 13.37. HRMS (ESI) calculated for $C_{21}H_{25}NO_2$ [M + H]: 324.1964, Found: 324.1957.



2p

Isopropyl 2-phenylindoline-3-carboxylate (2p)

The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 42%, 4.3:1 dr), R_f (pentane/EtOAc 4:1) = 0.5; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.37 (m, 2H), 7.30 – 7.17 (m, 3H), 7.15 – 7.00 (m, 2H), 6.67 (m, 2H), 5.35 (s, 1H), 5.23 (d, *J* = 10.0 Hz, 1H), 4.51 (m, 1H), 4.45 (d, *J* = 10.0 Hz, 1H), 0.94 (d, *J* = 6.3 Hz, 3H), 0.67 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 169.50, 152.57, 140.83, 128.26, 127.83, 127.46, 127.40, 126.07, 125.61, 117.88, 108.83, 67.11, 65.55, 53.50, 20.93, 20.48. HRMS (ESI) calculated for $C_{18}H_{19}NO_2$ [M + H]: 282.1494, Found: 282.1487.

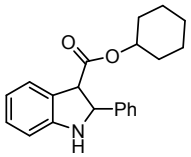


2q

Cyclopentyl 2-phenylindoline-3-carboxylate (2q)

The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 40%, 3.3:1 dr), R_f (pentane/EtOAc 4:1) = 0.5; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.40 (m, 2H), 7.34 – 7.23 (m, 3H), 7.21 – 7.03 (m, 2H), 6.71 (m, 2H), 5.35 (s, 1H), 5.25 (d, *J* = 10.0 Hz, 1H), 4.73 (s, 1H), 4.48 (d, *J* = 10.0 Hz, 1H), 1.79 – 1.08 (m, 8H). ^{13}C NMR (101 MHz, Acetone) δ 169.74, 152.55, 140.85, 128.29, 127.87, 127.44, 127.39, 126.10, 125.61, 117.92, 108.88, 76.57, 65.53, 53.48, 32.13, 31.87, 23.47, 23.41. HRMS (ESI) calculated for

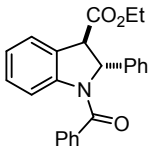
$C_{20}H_{21}NO_2$ [M + H]: 308.1651, Found: 308.1644.



2r

Cyclohexyl 2-phenylindoline-3-carboxylate (2r)

The product is isolated by column chromatography on silica gel as white solid (isolated total yield: 35%), R_f (pentane/EtOAc 4:1) = 0.5; **(*trans*) Diastereomer** (major): ^{1}H NMR (400 MHz, Acetone) δ 7.37 (m, 2H), 7.30 – 7.17 (m, 3H), 7.12 (d, *J* = 7.3 Hz, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 6.68 (m, 2H), 5.35 (s, 1H), 5.24 (d, *J* = 10.0 Hz, 1H), 4.46 (d, *J* = 10.0 Hz, 1H), 4.37 – 4.21 (m, 1H), 1.60 – 1.08 (m, 10H). ^{13}C NMR (101 MHz, Acetone) δ 169.34, 152.53, 140.78, 128.24, 127.81, 127.41, 127.36, 126.19, 125.53, 117.91, 108.90, 71.82, 65.58, 53.66, 31.11, 30.68, 25.13, 23.25, 23.17. HRMS (ESI) calculated for $C_{21}H_{23}NO_2$ [M + H]: 322.1807, Found: 322.1801.



3a

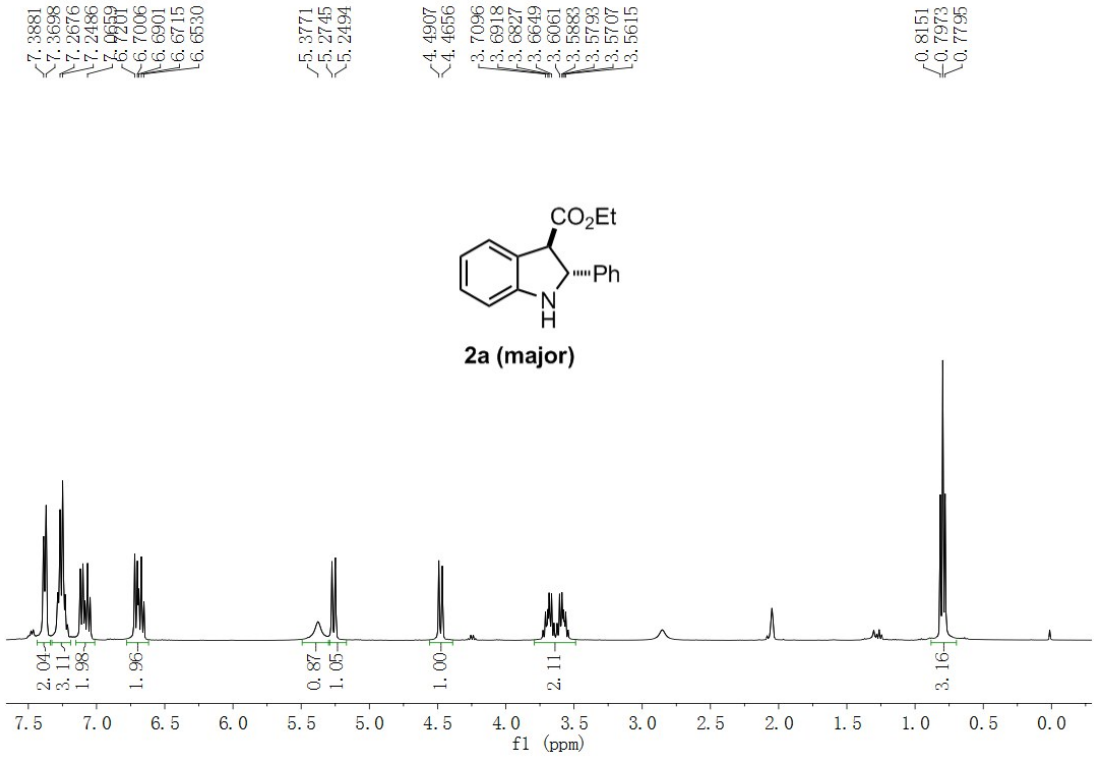
(*trans*)-Ethyl 1-benzoyl-2-phenylindoline-3-carboxylate (3a)

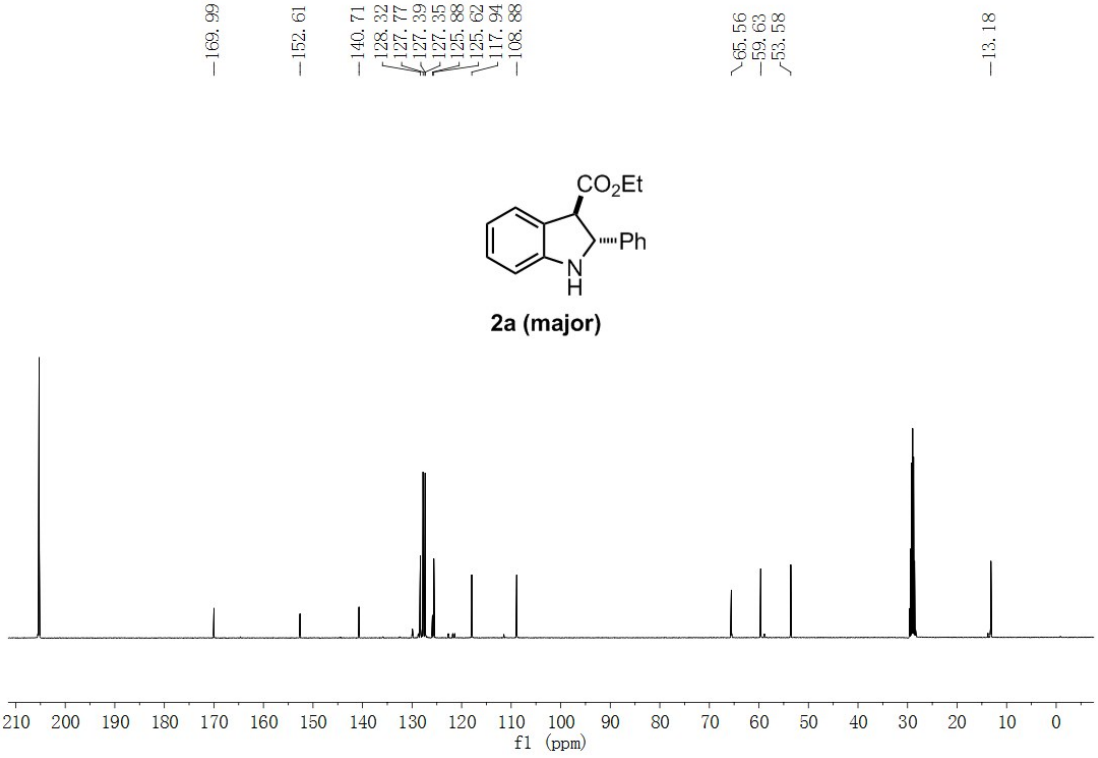
The product is isolated by column chromatography on silica gel as white solid, R_f (pentane/EtOAc 4:1) = 0.6; ^{1}H NMR (400 MHz, Acetone) δ 7.30 (m, 2H), 7.20 (m, 5H), 7.10 (m, 2H), 7.08 (m, 2H), 7.00 (t, *J* = 7.5 Hz, 1H), 6.89 (s, 2H), 5.82 (d, *J* = 0.9 Hz, 1H), 4.07 (q, *J* = 7.1 Hz, 2H), 3.87 (d, *J* = 1.4 Hz, 1H), 1.12 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Acetone) δ 170.67, 169.12, 143.49, 142.06, 137.31, 130.07, 128.97, 128.82, 128.34, 127.80, 127.02, 126.11, 125.52, 124.33, 116.84, 66.86, 61.41, 55.41, 13.65. HRMS (ESI) calculated for $C_{24}H_{21}NO_3$ [M + Na]: 394.1419, Found: 394.1406.

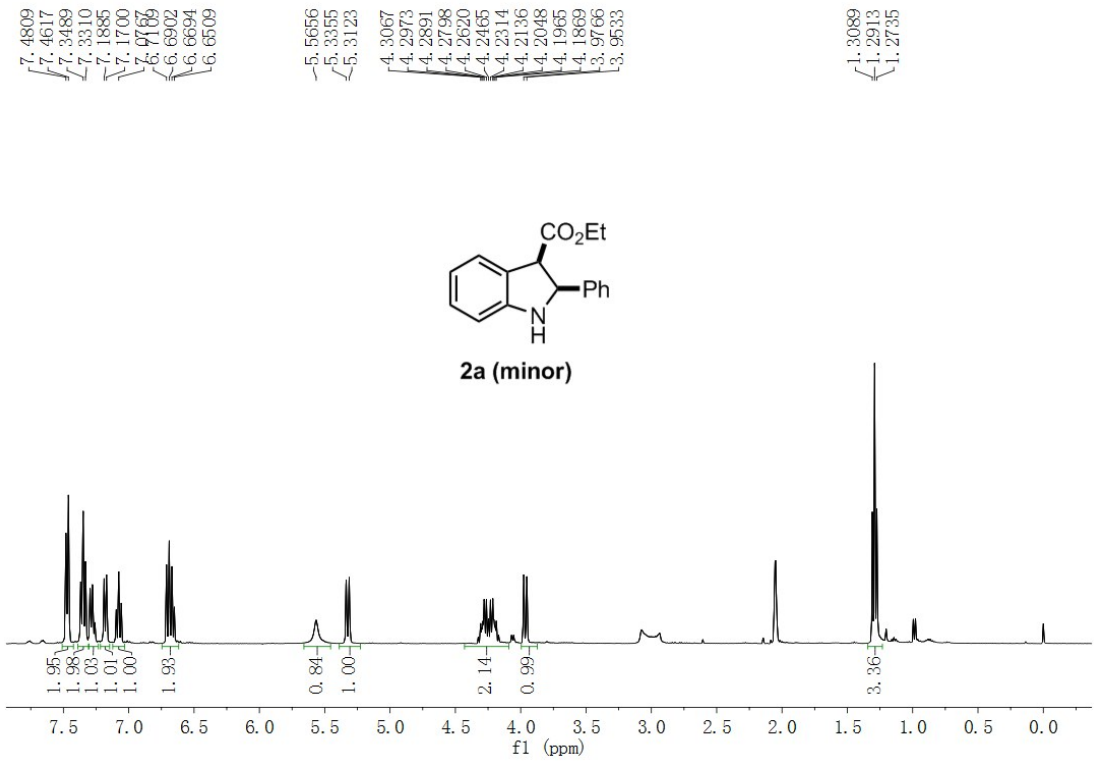
Reference

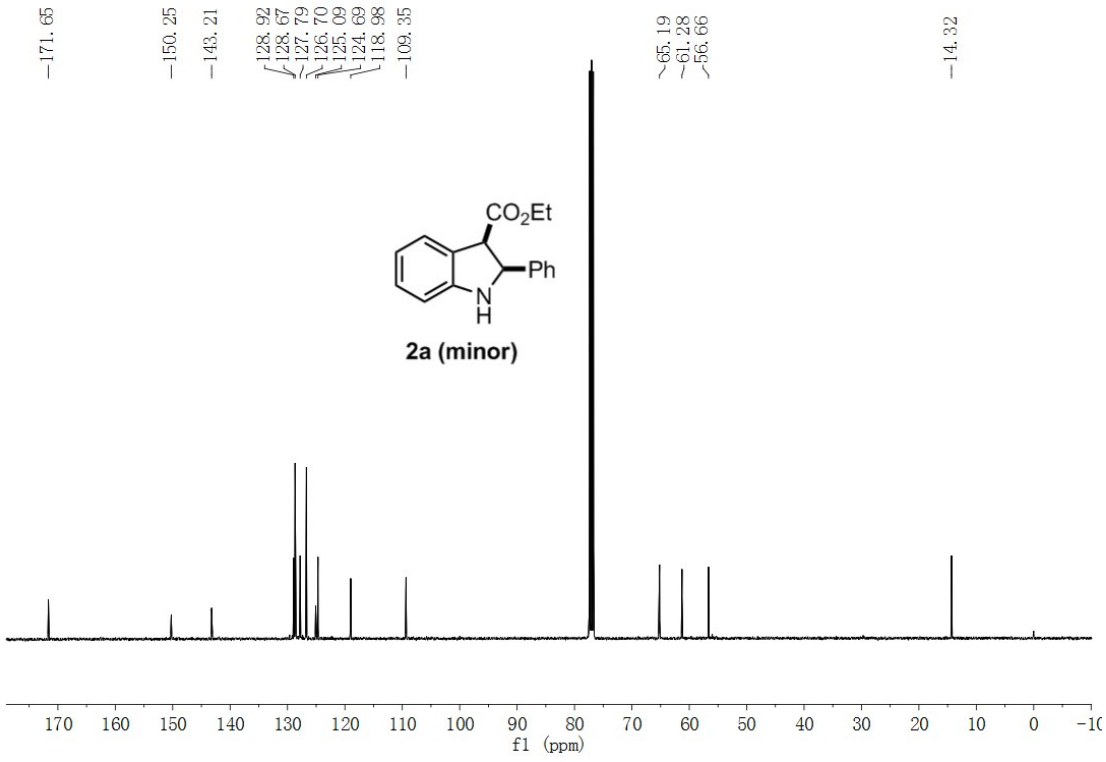
1. S. Würtz, S. Rakshit, J. J. Neumann, T. Dröge, F. Glorius, *Angew. Chem. Int. Ed.*, 2008, **47**, 7230.
2. G. Bartoli, M. Bosco, M. Locatelli, E. Marcantoni, P. Melchiorre, L. Sambri, *Synlett*, 2004, **2**, 239.
3. C. A. Brandt, A. C. M. P. Silva, C. G. Pancote, C. L. Brito, M. A. B. Silveira, *Synthesis*, 2004, **10**, 1557.
4. Z. He, W. Liu, Z. Li, *Chem. Asian J.*, 2011, **6**, 1340.
5. M. Chen, Z.-T. Huang, Q.-Y. Zheng, *Chem. Commun.*, 2012, **48**, 11686.

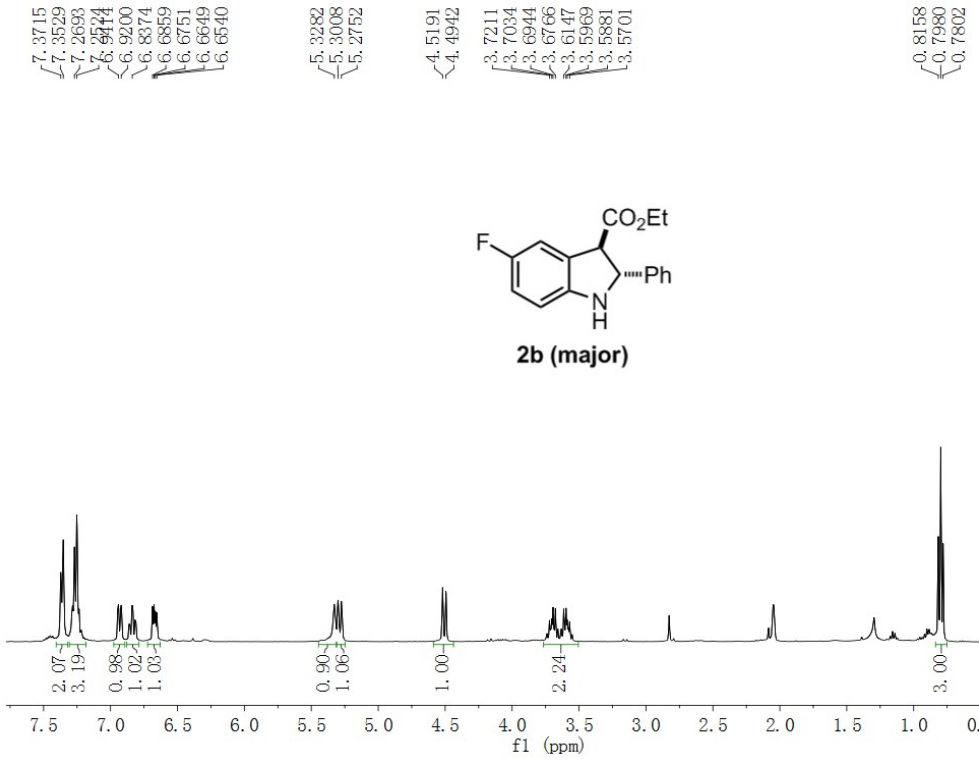
5. ^{1}H and ^{13}C NMR

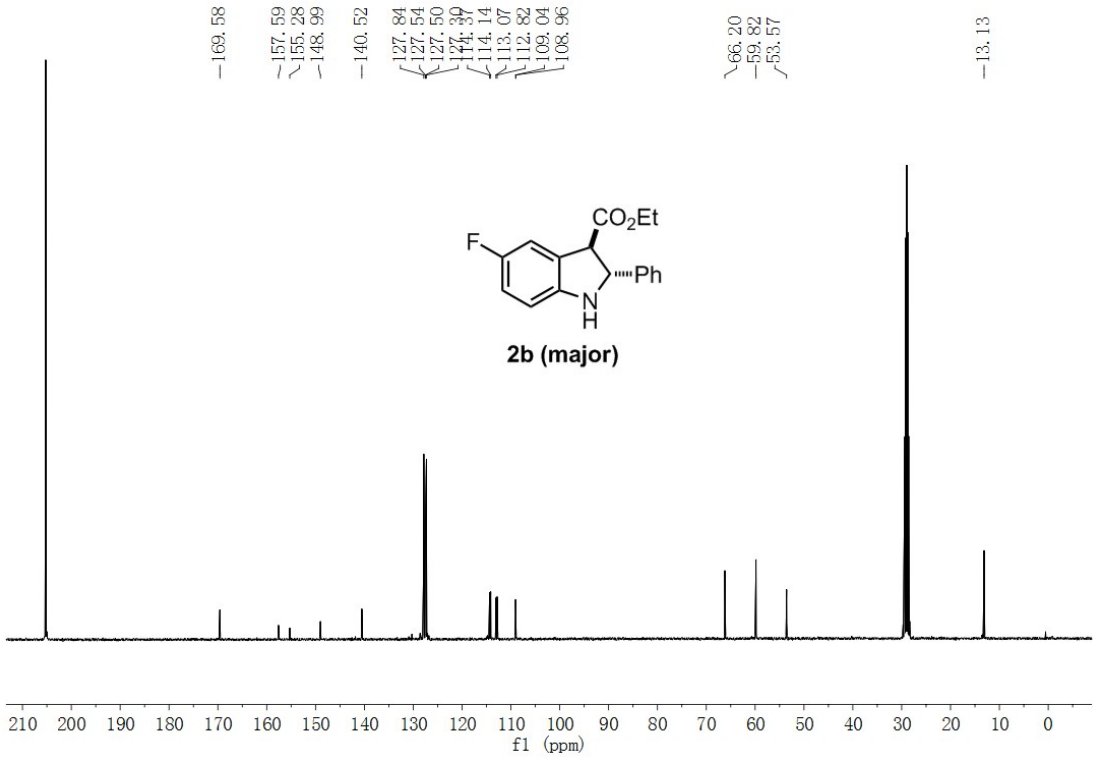


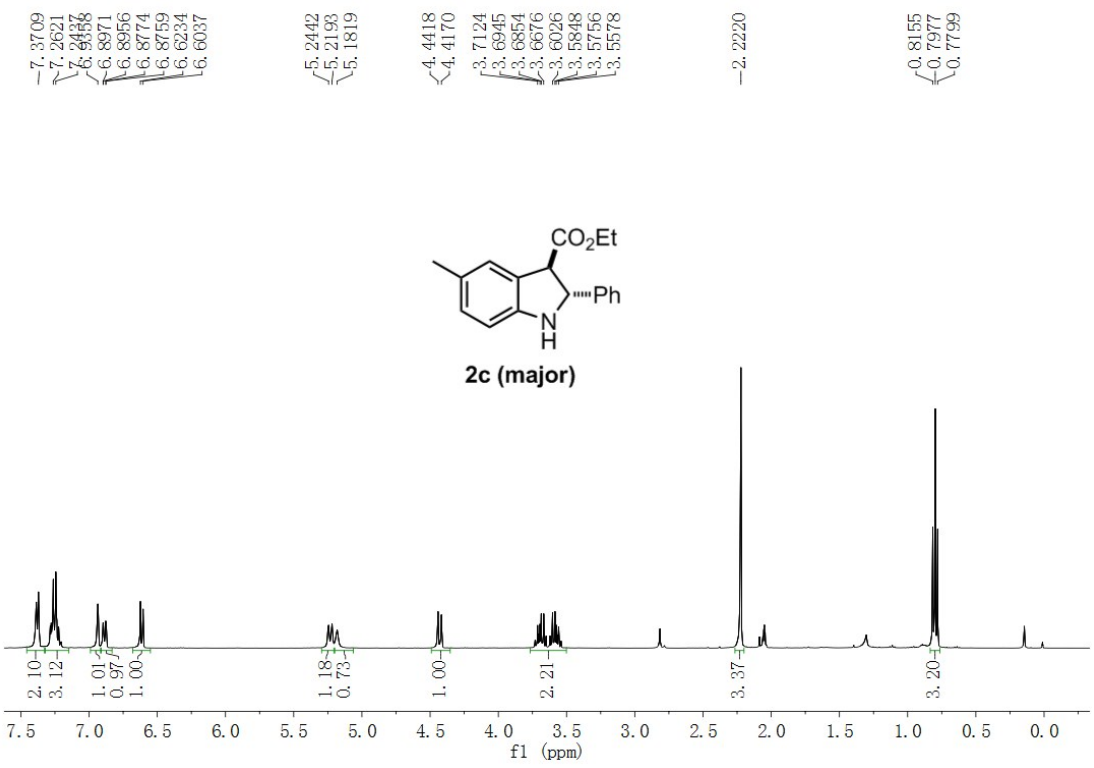


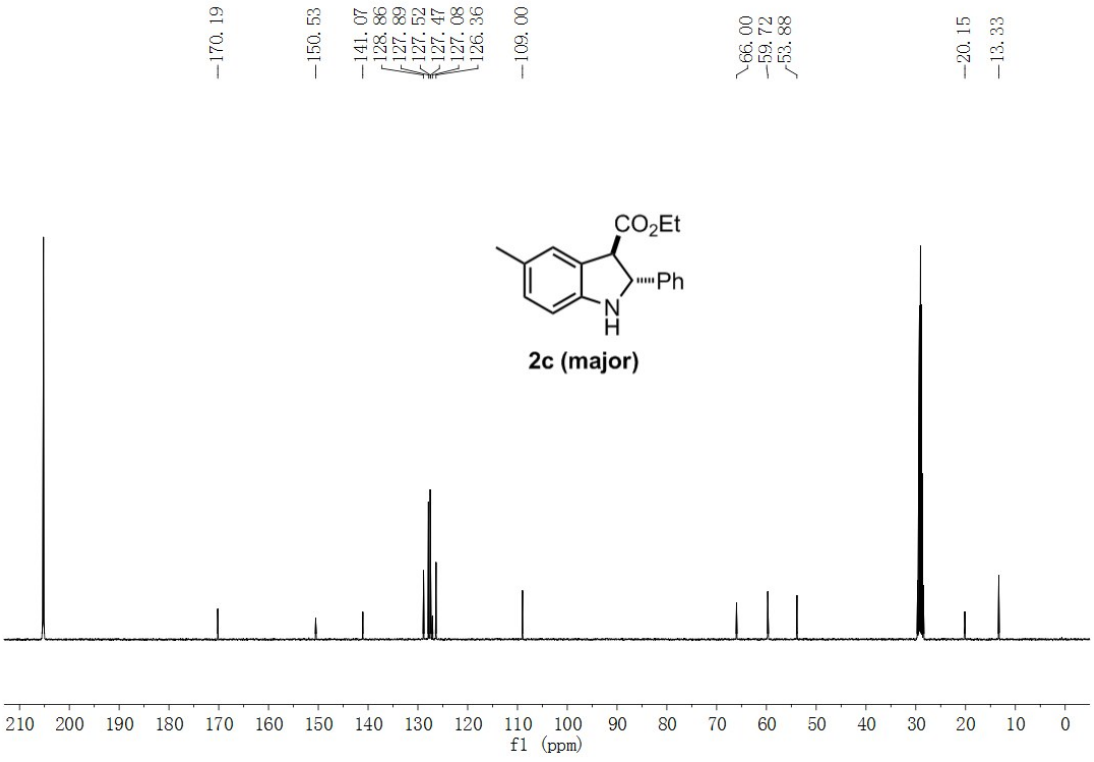

7.4809
7.4617
7.3489
7.3310
7.1885
7.1700
7.0767
7.0709
6.6902
6.6694
6.6509
5.5656
5.3355
5.3123
4.3067
4.2973
4.2891
4.2798
4.2620
4.2465
4.2314
4.2136
4.2048
4.1965
4.1869
3.9766
3.9533
1.3089
1.2913
1.2735
CO2Et
Ph
N
H
2a (minor)
1.95
1.98
1.03
1.01
1.00
1.93
0.84
1.00
2.14
0.99
3.36
7.5 7.0 6.5 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)

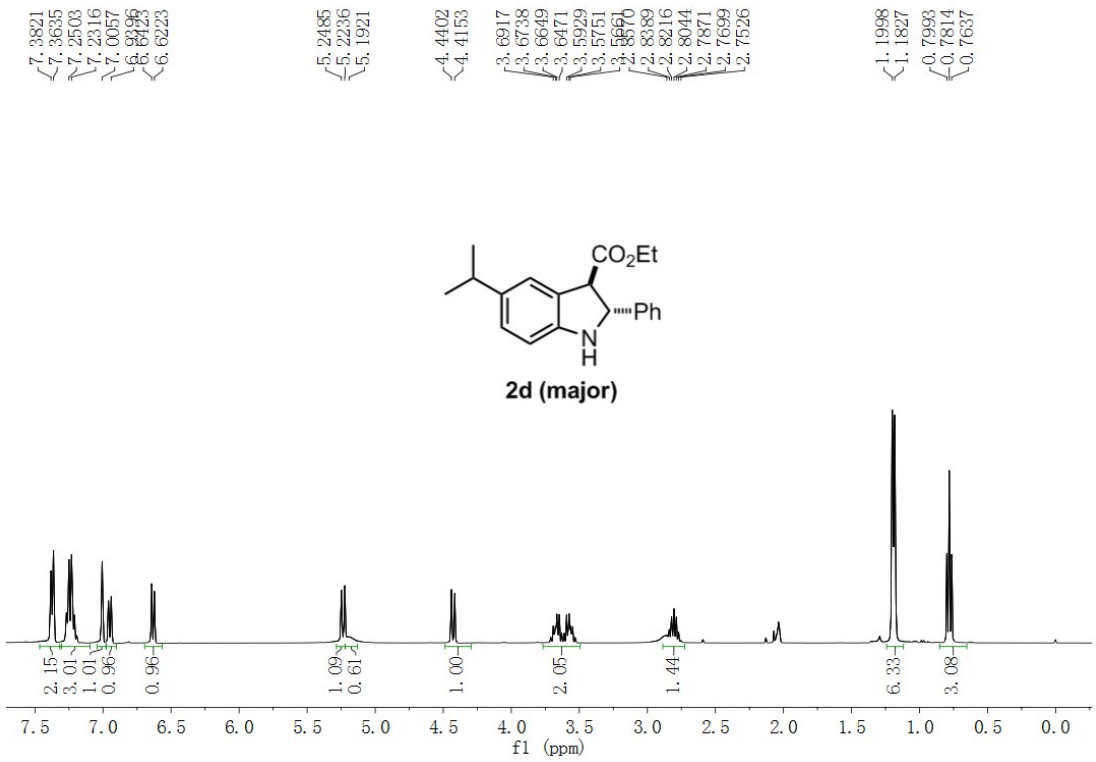

171.65
150.25
143.21
128.92
128.67
127.79
126.70
125.09
124.69
118.98
109.35
65.19
61.28
56.66
14.32
CO2Et
Ph
N
H
2a (minor)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

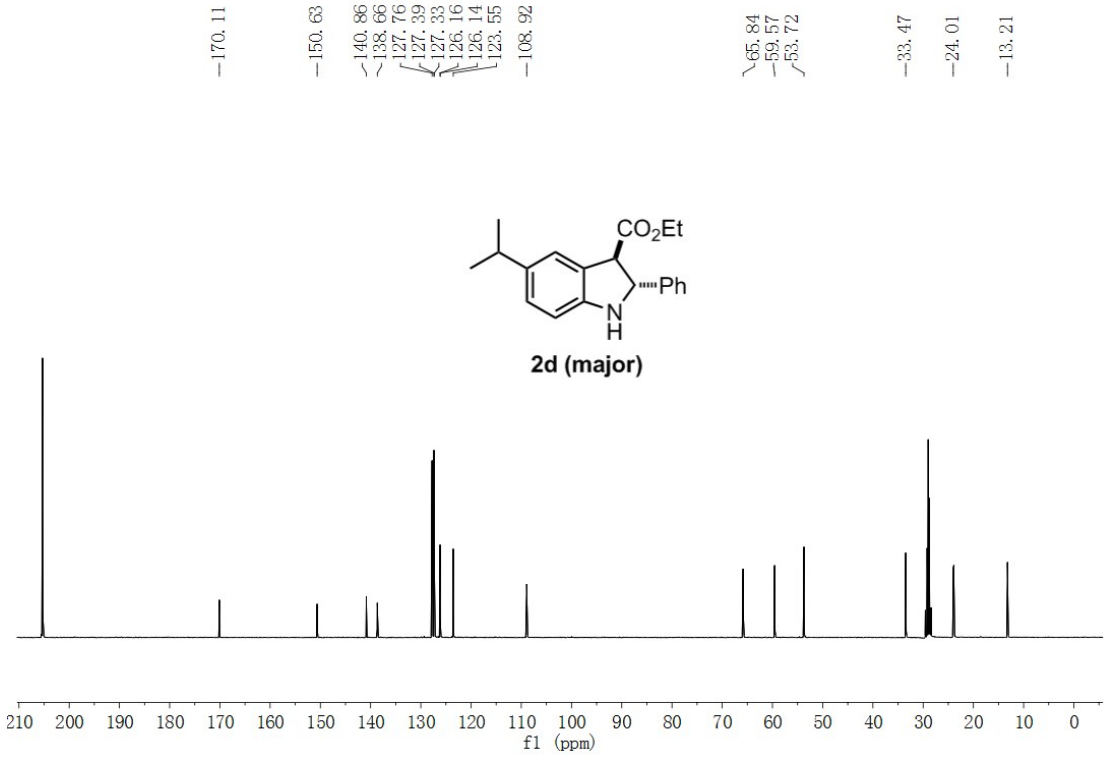

7.3715
7.3529
7.2693
6.9200
6.8374
6.6859
6.6751
6.6649
6.6540
5.3282
5.3008
5.2752
4.5191
4.4942
3.7211
3.7034
3.6944
3.6766
3.6147
3.5969
3.5881
3.5701
0.8158
0.7980
0.7802
CO2Et
F
Ph
N
H
2b (major)
2.07
3.19
0.98
1.02
1.03
0.90
1.06
1.00
2.24
3.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

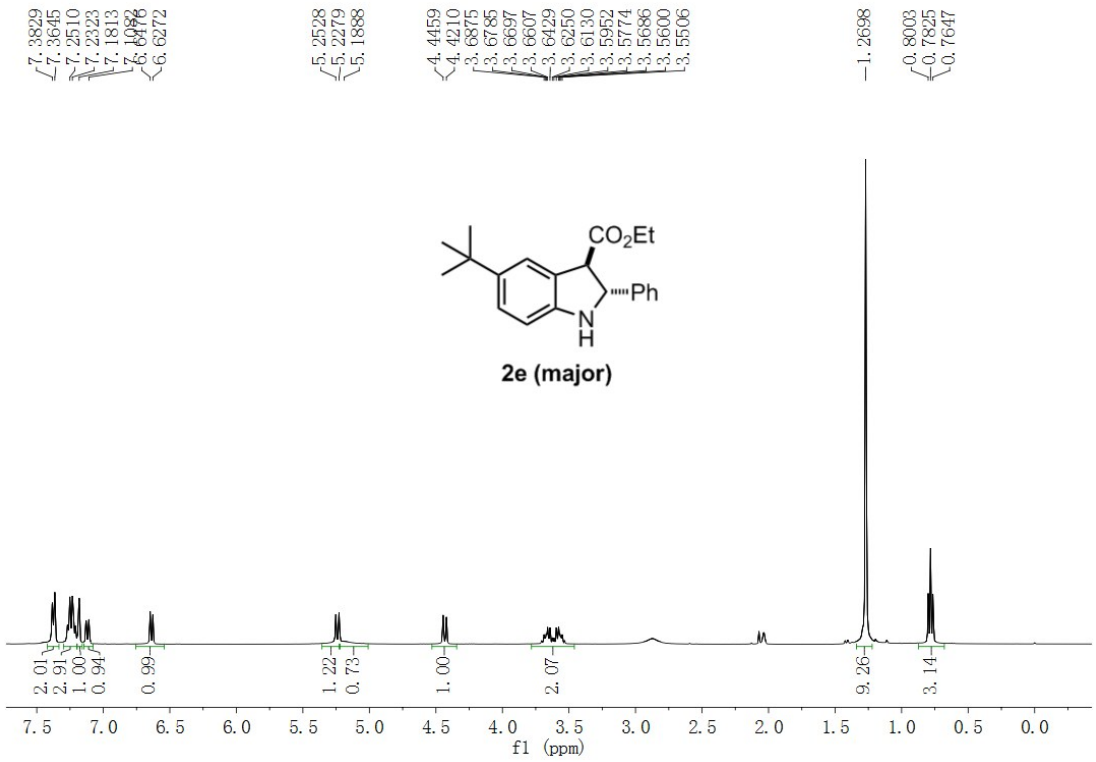

169.58
157.59
155.28
148.99
140.52
127.84
127.54
127.50
114.14
113.07
112.82
109.04
108.96
66.20
59.82
53.57
13.13
CO2Et
F
Ph
N
H
2b (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

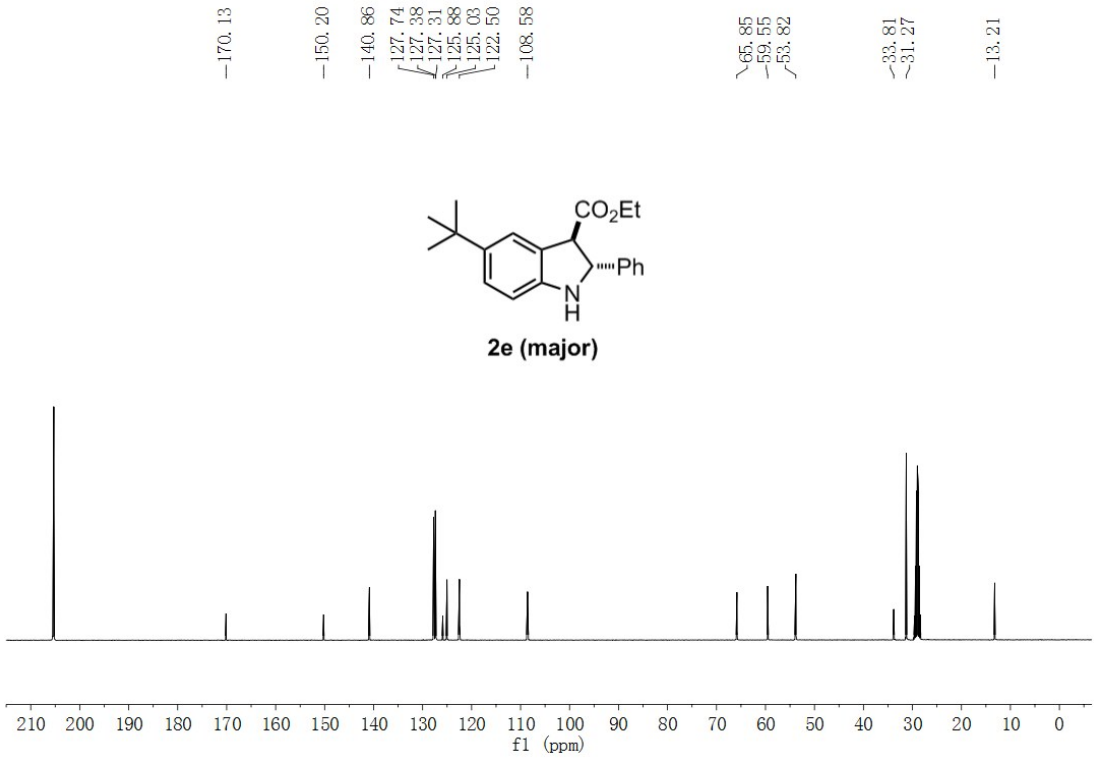

7.3709
7.2621
7.2437
6.8971
6.8956
6.8774
6.8759
6.6234
6.6037
5.2442
5.2193
5.1819
4.4418
4.4170
3.7124
3.6945
3.6854
3.6676
3.6026
3.5848
3.5756
3.5578
2.2220
0.8155
0.7977
0.7799
CO2Et
Ph
N
H
2c (major)
2.10
3.12
1.01
0.97
1.00
1.18
0.73
1.00
2.21
3.37
3.20
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

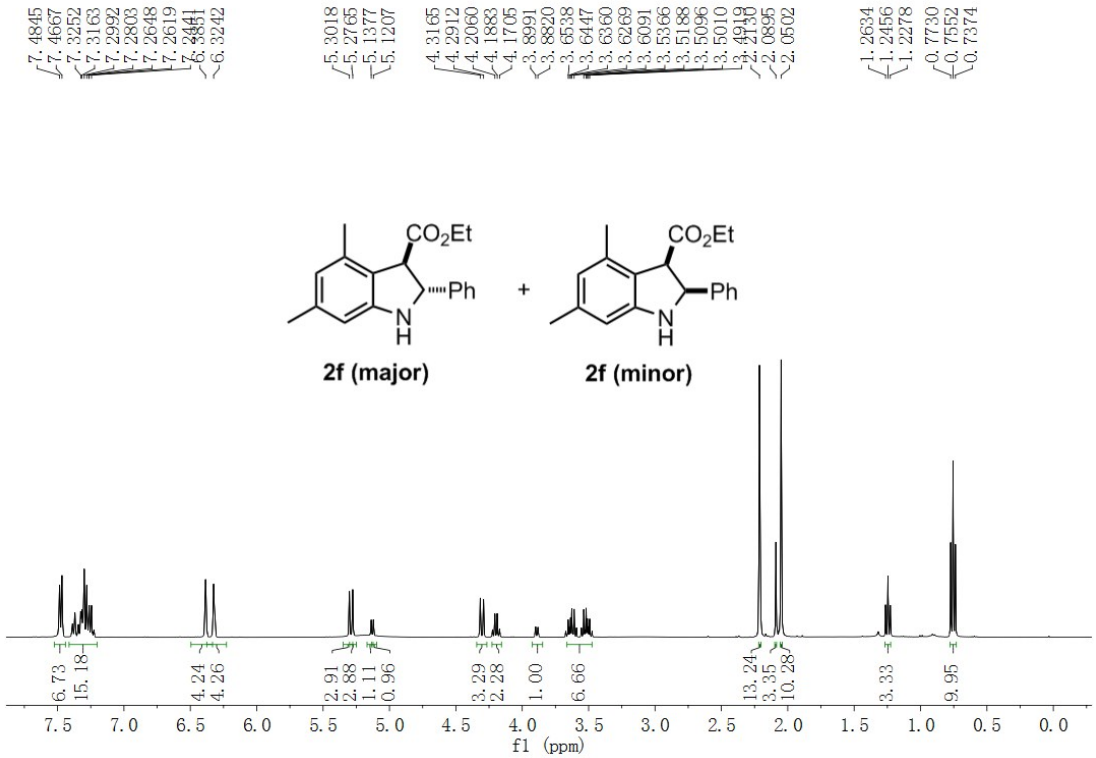

170.19
150.53
141.07
128.86
127.89
127.52
127.47
127.08
126.36
109.00
66.00
59.72
53.88
20.15
13.33
CO2Et
Ph
N
H
2c (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

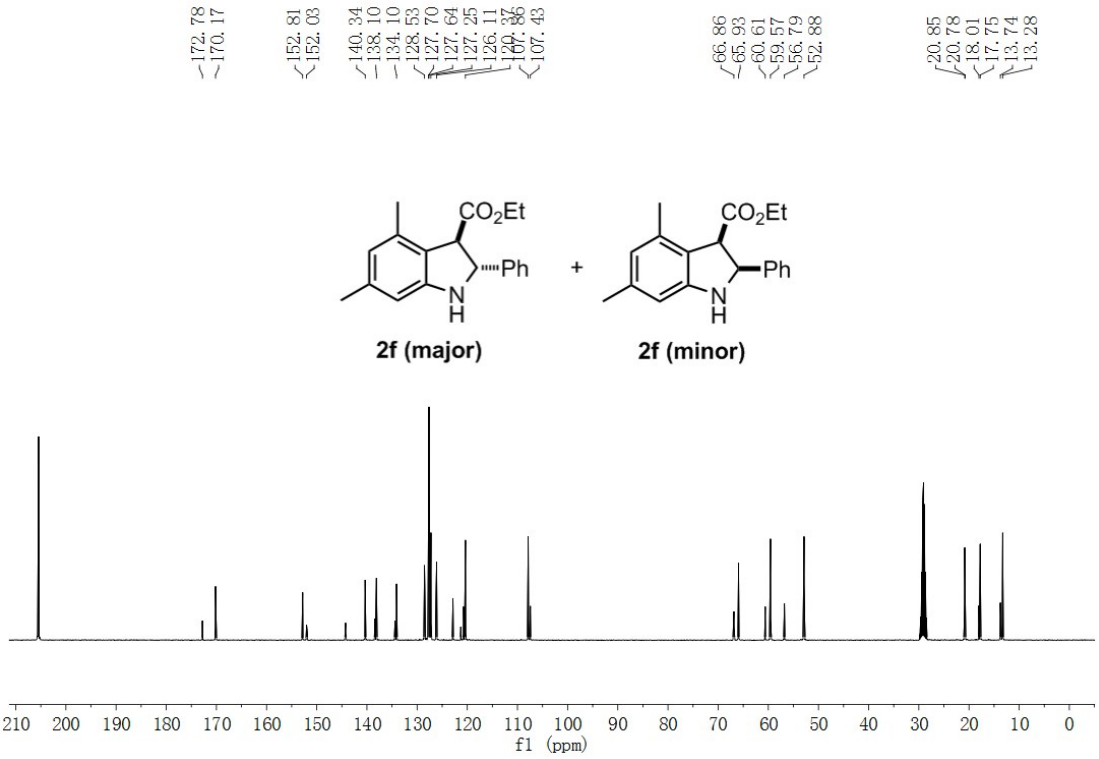

7.3821
7.3635
7.2503
7.2316
7.0057
6.9396
6.6423
6.6223
5.2485
5.2236
5.1921
4.4402
4.4153
3.6917
3.6738
3.6649
3.6471
3.5929
3.5751
3.5661
2.8570
2.8389
2.8216
2.8044
2.7871
2.7699
2.7526
1.1998
1.1827
0.7993
0.7814
0.7637
CO2Et
Ph
N
H
2d (major)
2.15
3.01
1.01
0.96
0.96
1.09
0.61
1.00
2.05
1.44
6.33
3.08
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)

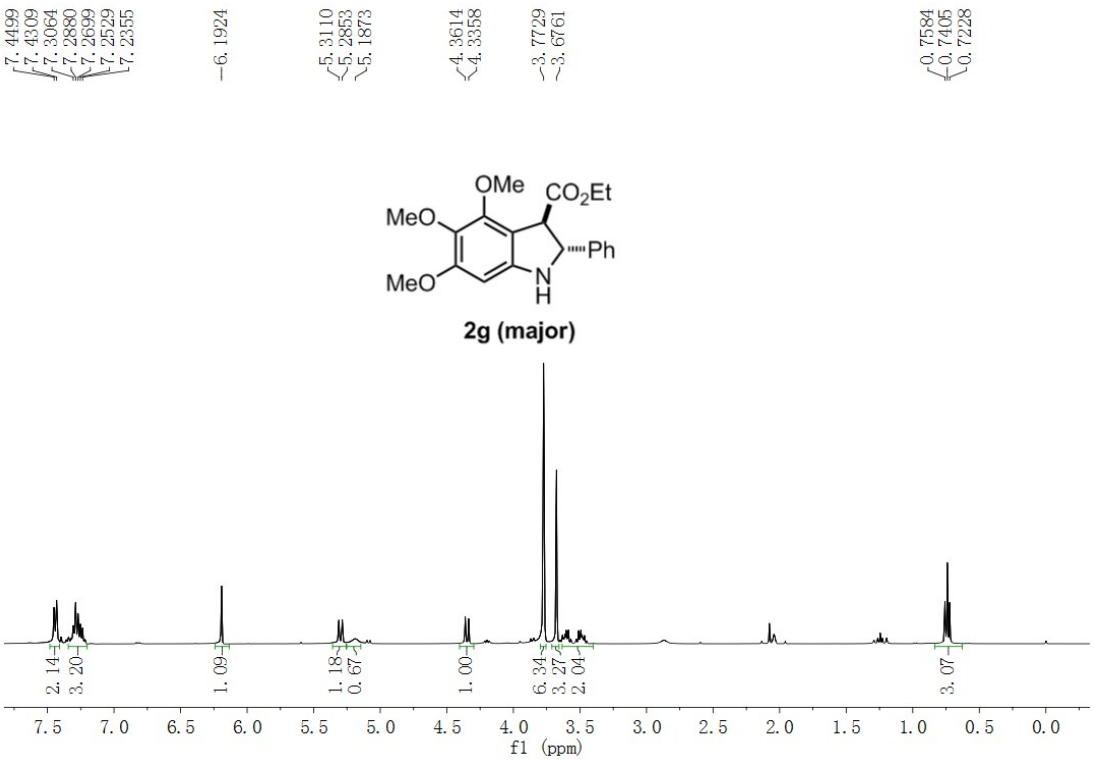

170.11
150.63
140.86
138.66
127.76
127.39
127.33
126.16
126.14
123.55
108.92
65.84
59.57
53.72
33.47
24.01
13.21
CO2Et
Ph
N
H
2d (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

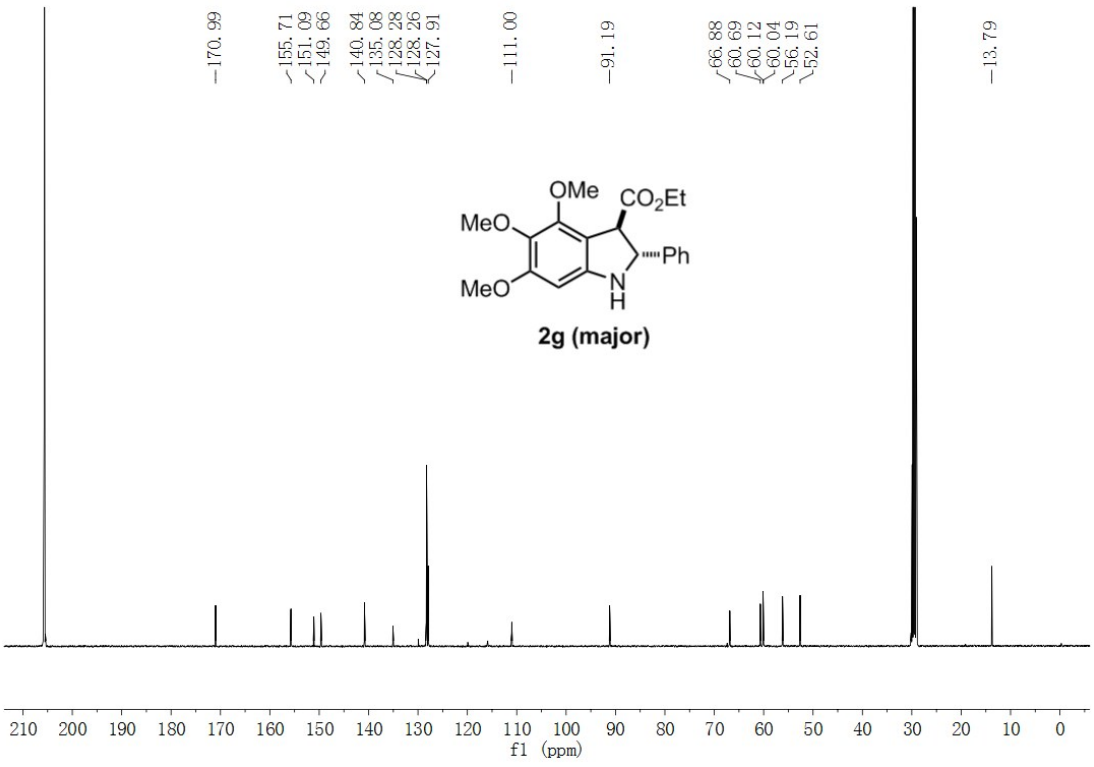

7.3829
7.3645
7.2510
7.2323
7.1813
6.6272
5.2528
5.2279
5.1888
4.4459
4.4210
3.6875
3.6785
3.6697
3.6607
3.6429
3.6250
3.6130
3.5952
3.5774
3.5686
3.5600
3.5506
1.2698
0.8003
0.7825
0.7647
CO2Et
Ph
N
H
2e (major)
2.01
2.91
1.00
0.94
0.99
1.22
0.73
1.00
2.07
9.26
3.14
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

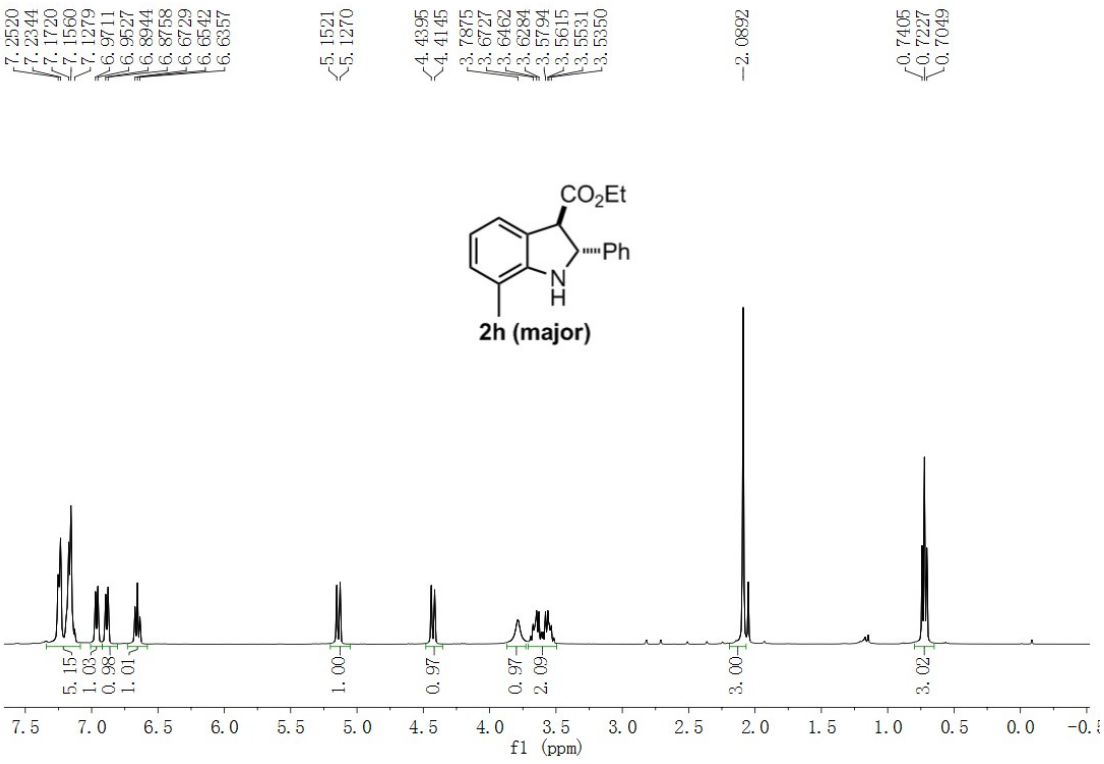

170.13
150.20
140.86
127.74
127.38
127.31
125.88
125.03
122.50
108.58
65.85
59.55
53.82
33.81
31.27
13.21
CO2Et
Ph
N
H
2e (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

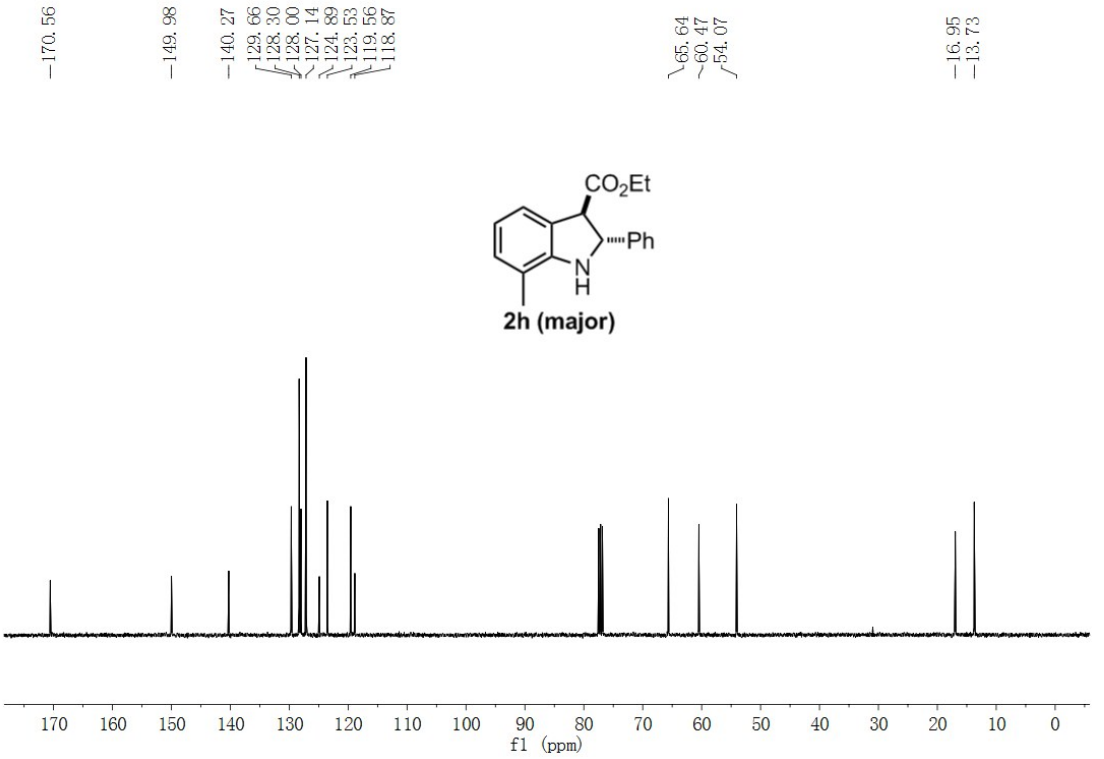

CO2Et
Ph
N
H
2f (major)
+
2f (minor)
f1 (ppm)

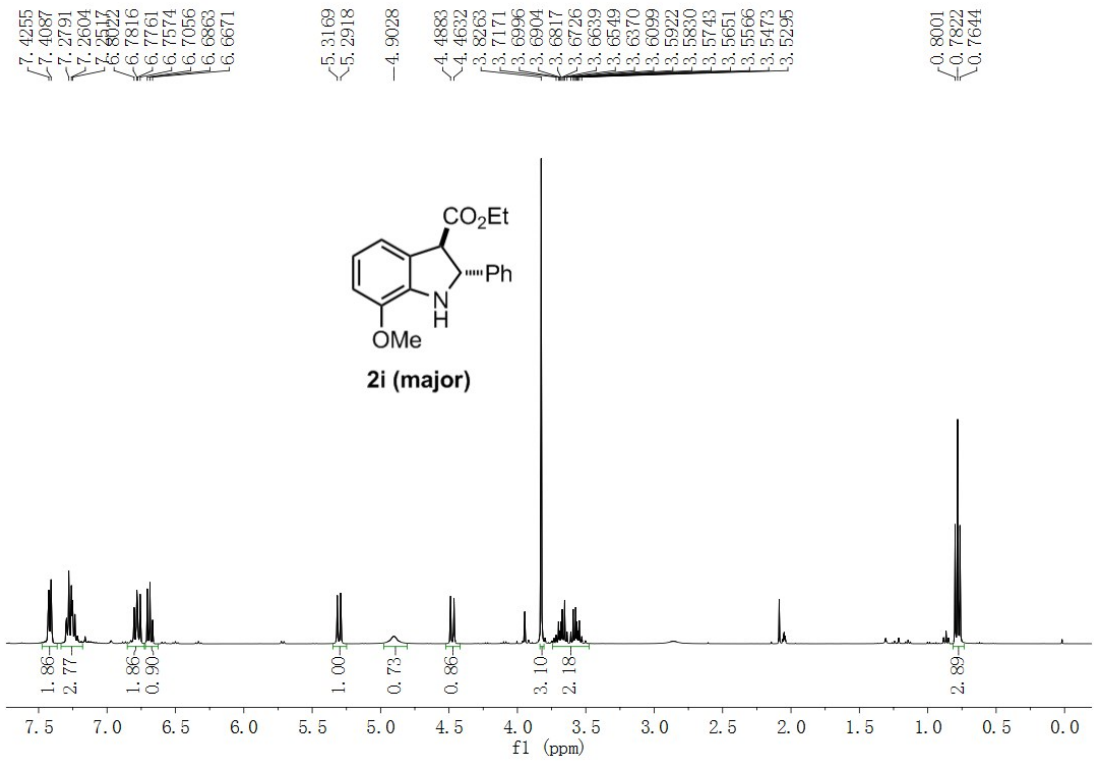

CO2Et
Ph
N
H
2f (major)
+
2f (minor)
f1 (ppm)

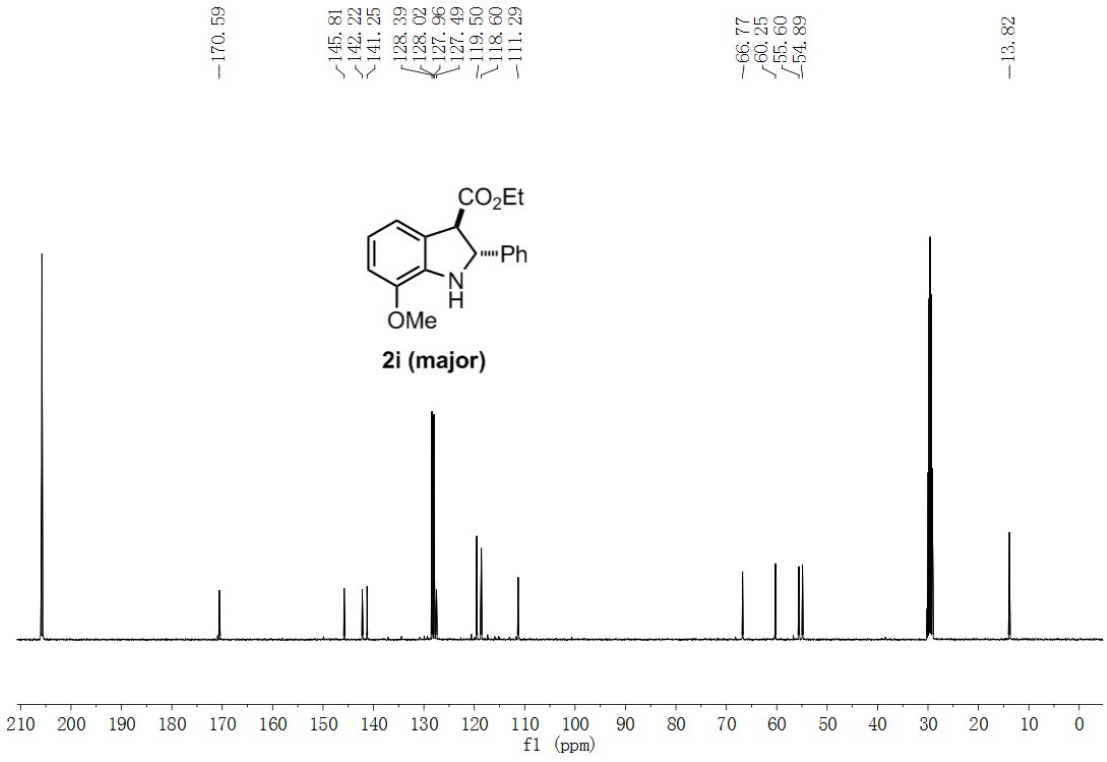

7.4499
7.4309
7.3064
7.2880
7.2699
7.2529
7.2355
6.1924
5.3110
5.2853
5.1873
4.3614
4.3358
3.7729
3.6761
0.7584
0.7405
0.7228
OMe
CO2Et
MeO
Ph
MeO
N
H
2g (major)
2.14
3.20
1.09
1.18
0.67
1.00
6.34
3.27
2.04
3.07
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

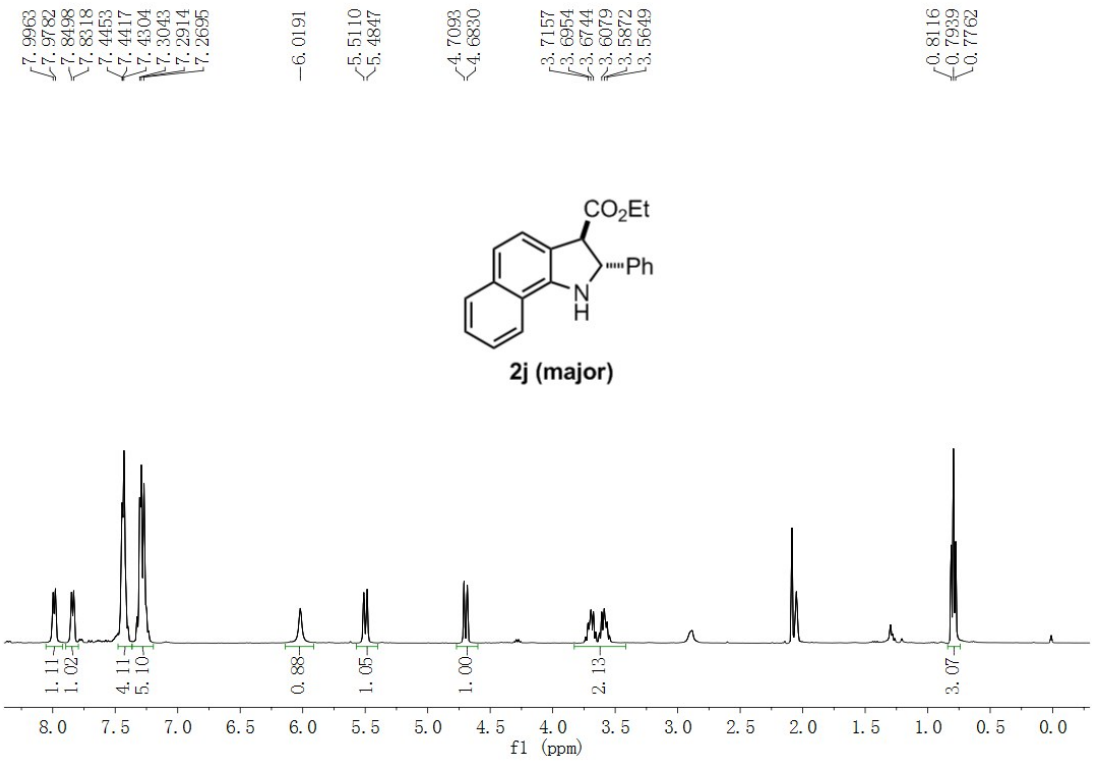

170.99
155.71
151.09
149.66
140.84
135.08
128.28
128.26
127.91
111.00
91.19
66.88
60.69
60.12
60.04
56.19
52.61
13.79
OMe
CO2Et
MeO
Ph
MeO
N
H
2g (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

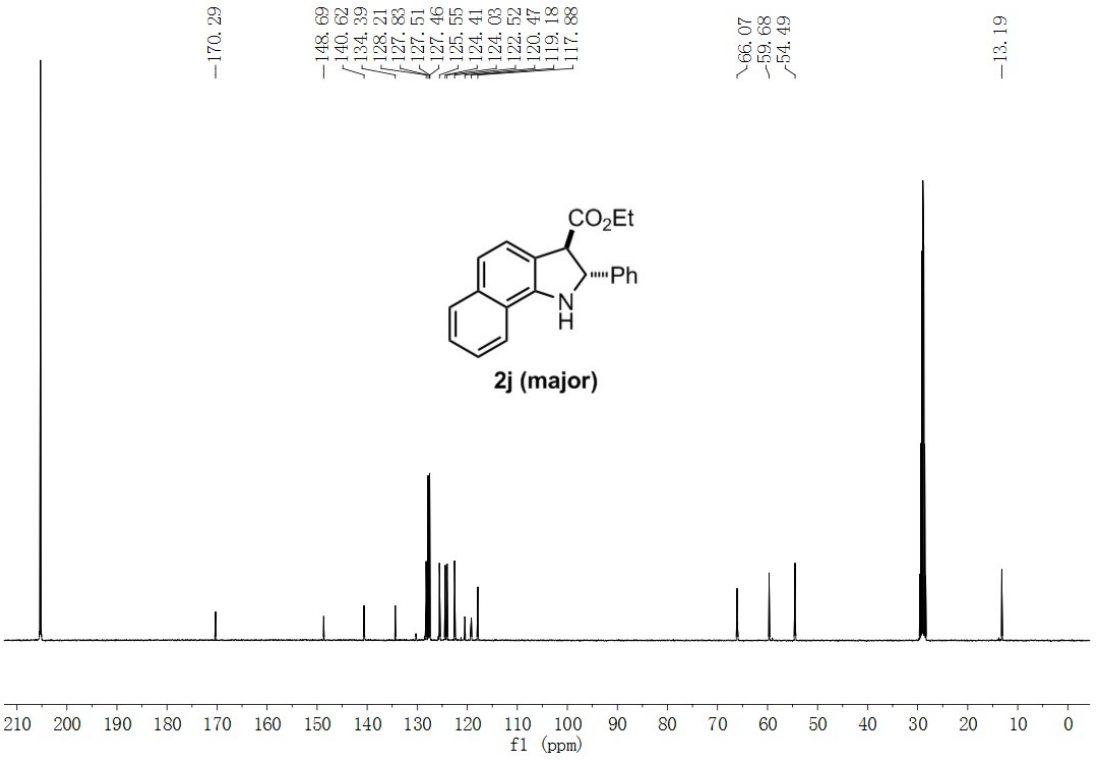

7.2520
7.2344
7.1720
7.1560
7.1279
6.9711
6.9527
6.8944
6.8758
6.6729
6.6542
6.6357
5.1521
5.1270
4.4395
4.4145
3.7875
3.6727
3.6462
3.6284
3.5794
3.5615
3.5531
3.5350
2.0892
0.7405
0.7227
0.7049
CO2Et
Ph
N
H
2h (major)
5.15
1.03
0.98
1.01
1.00
0.97
0.97
2.09
3.00
3.02
f1 (ppm)

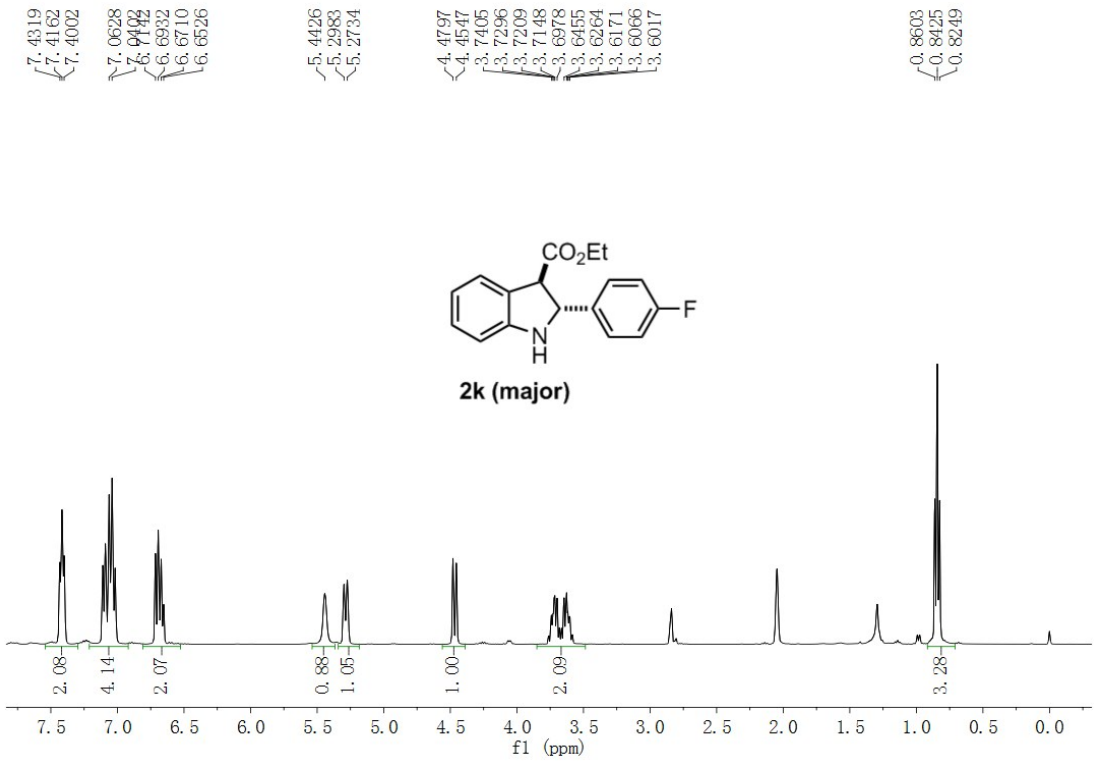

170.56
149.98
140.27
129.66
128.30
128.00
127.14
124.89
123.53
119.56
118.87
65.64
60.47
54.07
16.95
13.73
CO2Et
Ph
N
H
2h (major)
f1 (ppm)

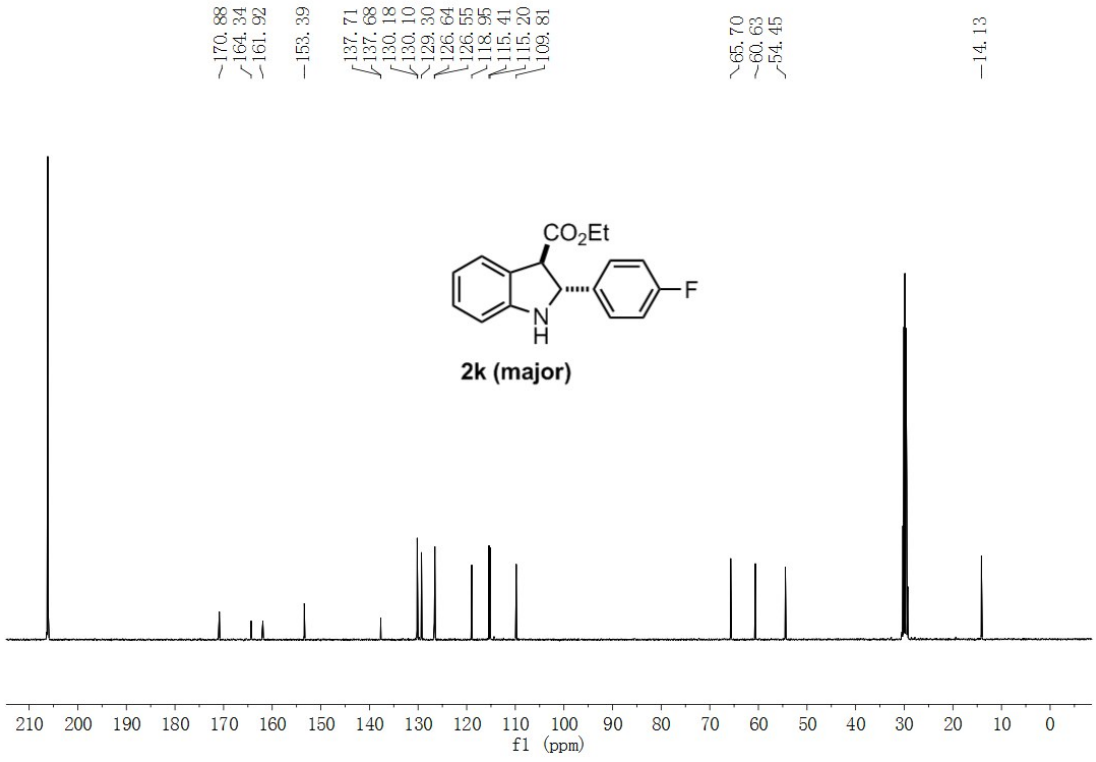

7.4255
7.4087
7.2791
7.2604
7.2517
7.2022
6.7816
6.7761
6.7574
6.7056
6.6863
6.6671
5.3169
5.2918
4.9028
4.4883
4.4632
3.8263
3.7171
3.6996
3.6904
3.6817
3.6726
3.6639
3.6549
3.6370
3.6099
3.5922
3.5830
3.5743
3.5651
3.5566
3.5473
3.5295
0.8001
0.7822
0.7644
CO2Et
Ph
N
H
OMe
2i (major)
1.86
2.77
1.86
0.90
1.00
0.73
0.86
3.10
2.18
2.89
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)

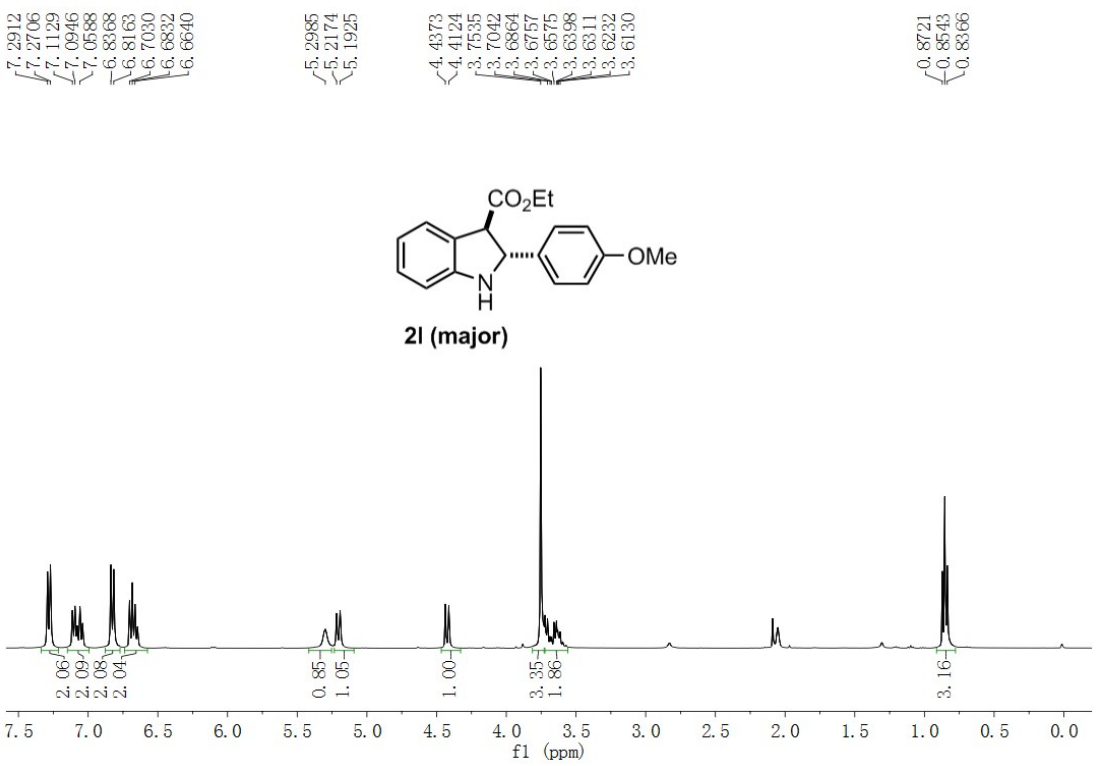

170.59
145.81
142.22
141.25
128.39
128.02
127.96
127.49
119.50
118.60
111.29
66.77
60.25
55.60
54.89
13.82
CO2Et
Ph
N
H
OMe
2i (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

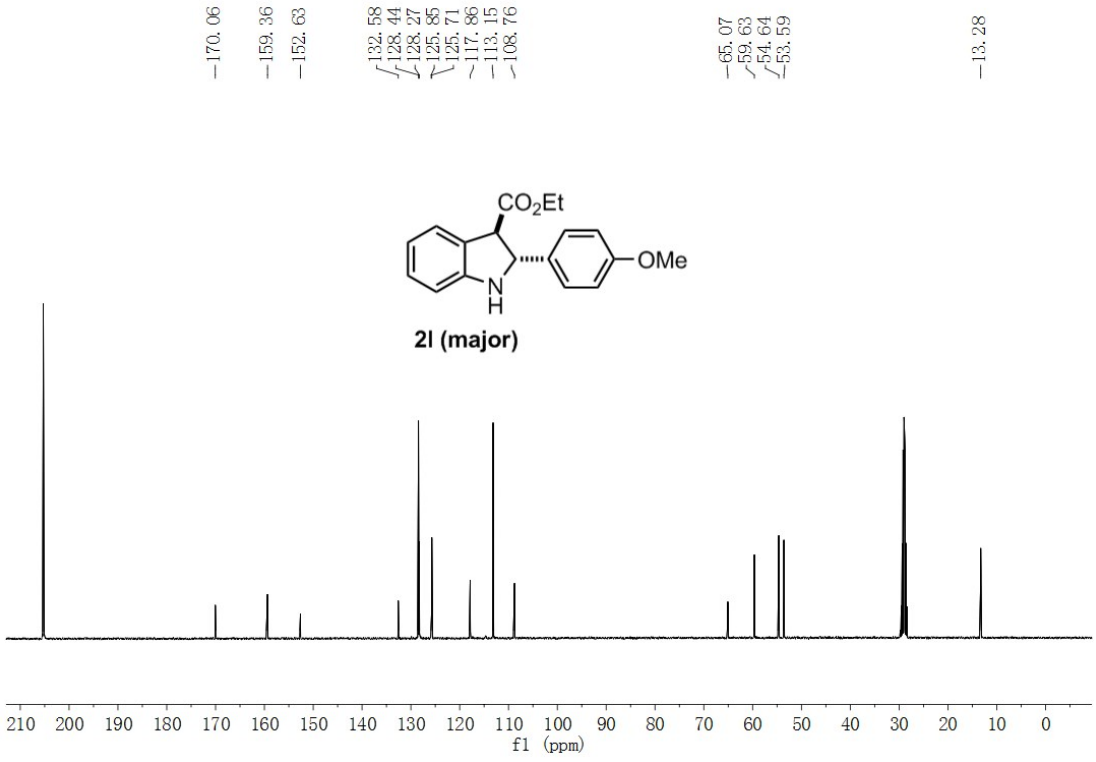

7.9963
7.9782
7.8498
7.8318
7.4453
7.4417
7.4304
7.3043
7.2914
7.2695
6.0191
5.5110
5.4847
4.7093
4.6830
3.7157
3.6954
3.6744
3.6079
3.5872
3.5649
0.8116
0.7939
0.7762
CO2Et
Ph
N
H
2j (major)
1.11
1.02
4.11
5.10
0.88
1.05
1.00
2.13
3.07
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

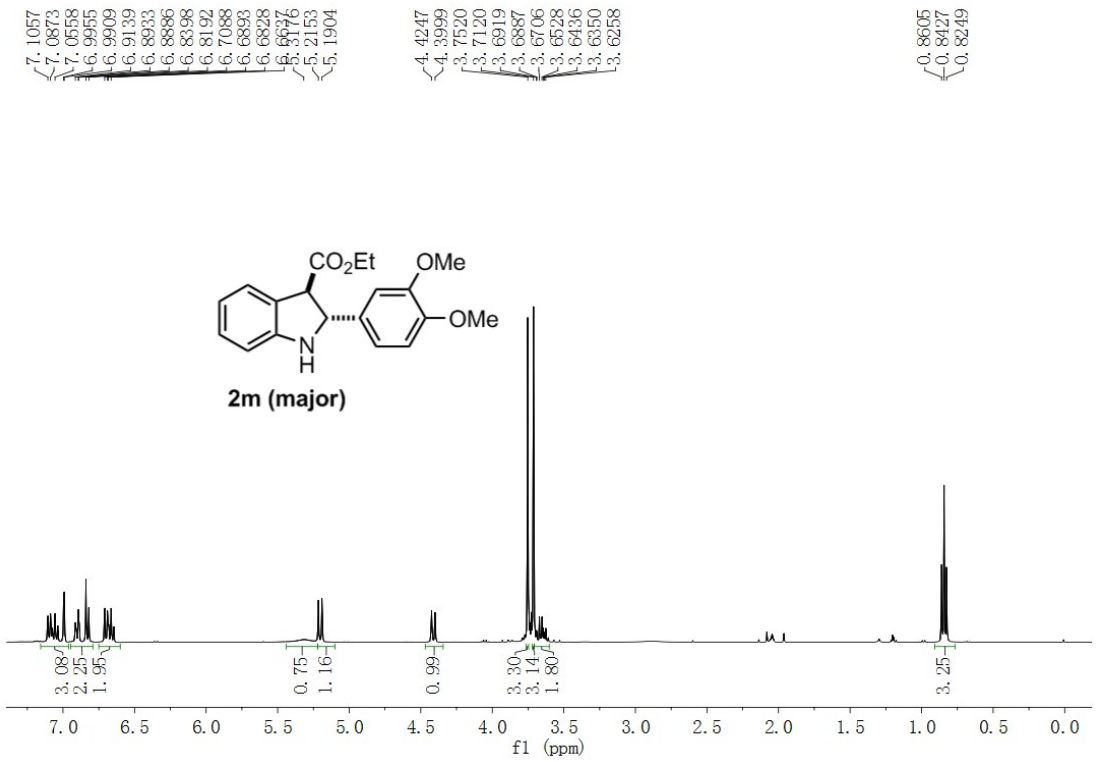

170.29
148.69
140.62
134.39
128.21
127.83
127.51
127.46
125.55
124.41
124.03
122.52
120.47
119.18
117.88
66.07
59.68
54.49
13.19
CO2Et
Ph
N
H
2j (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

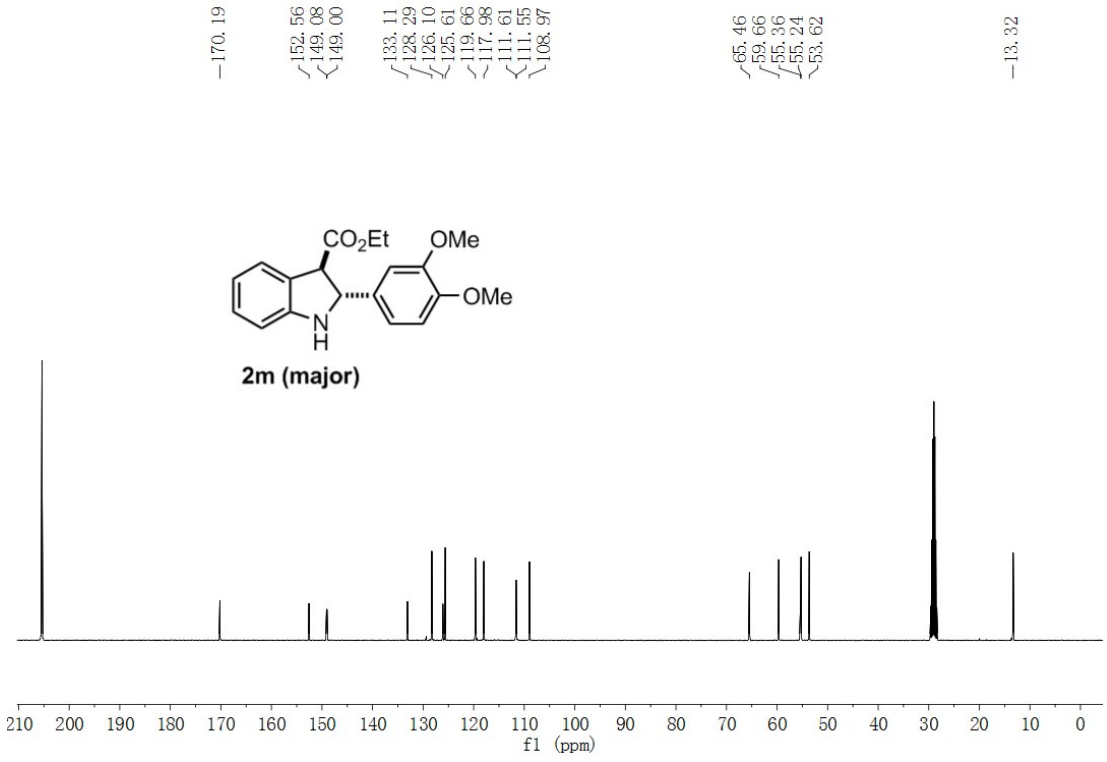

7.4319
7.4162
7.4002
7.0628
6.6932
6.6710
6.6526
5.4426
5.2983
5.2734
4.4797
4.4547
3.7405
3.7296
3.7209
3.7148
3.6978
3.6455
3.6264
3.6171
3.6066
3.6017
0.8603
0.8425
0.8249
CO2Et
F
N
H
2k (major)
2.08
4.14
2.07
0.88
1.05
1.00
2.09
3.28
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

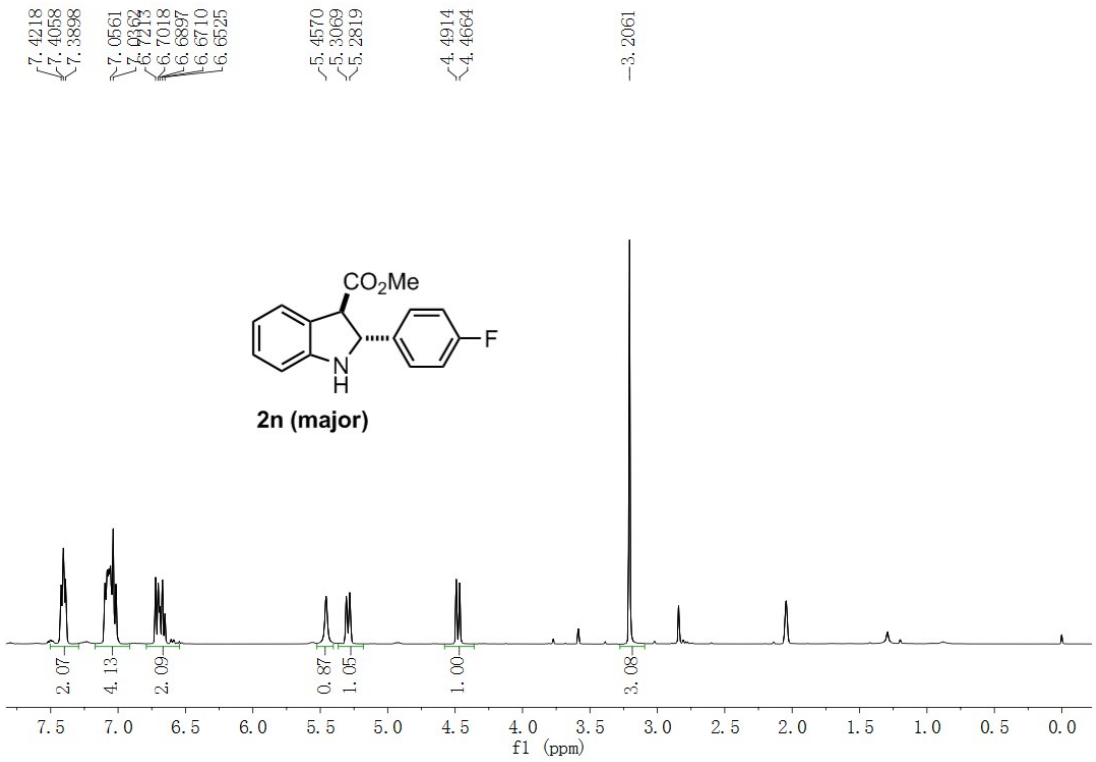

170.88
164.34
161.92
153.39
137.71
137.68
130.18
130.10
129.30
126.64
126.55
118.95
115.41
115.20
109.81
65.70
60.63
54.45
14.13
CO2Et
F
N
H
2k (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

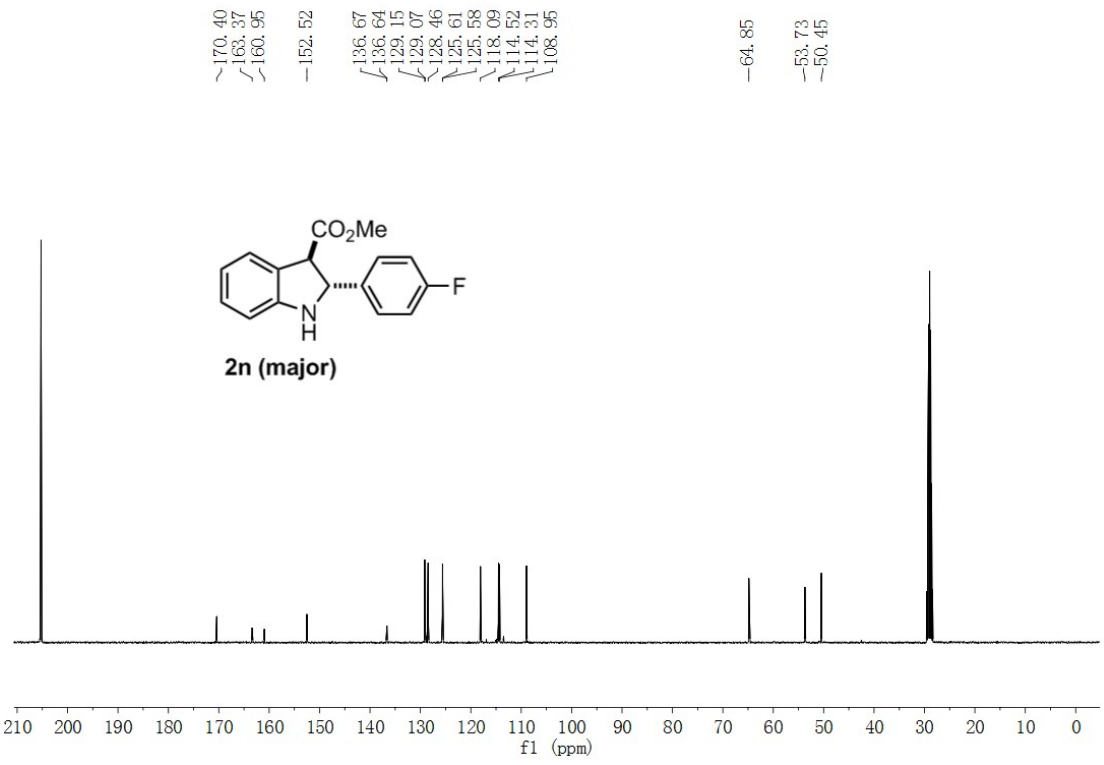

7.2912
7.2706
7.1129
7.0946
7.0588
6.8368
6.8163
6.7030
6.6832
6.6640
5.2985
5.2174
5.1925
4.4373
4.4124
3.7535
3.7042
3.6864
3.6757
3.6575
3.6398
3.6311
3.6232
3.6130
0.8721
0.8543
0.8366
CO2Et
OMe
N
H
2l (major)
2.06
2.09
2.08
2.04
0.85
1.05
1.00
3.35
1.86
3.16
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

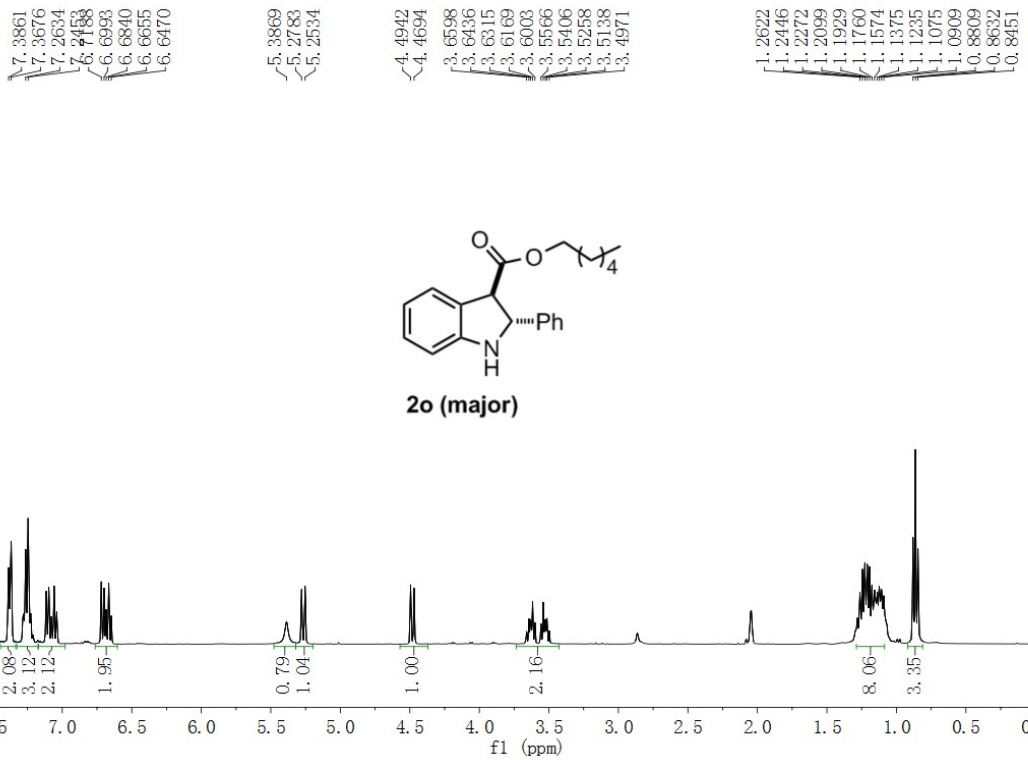

170.06
159.36
152.63
132.58
128.44
128.27
125.85
125.71
117.86
113.15
108.76
65.07
59.63
54.64
53.59
13.28
CO2Et
OMe
N
H
2l (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

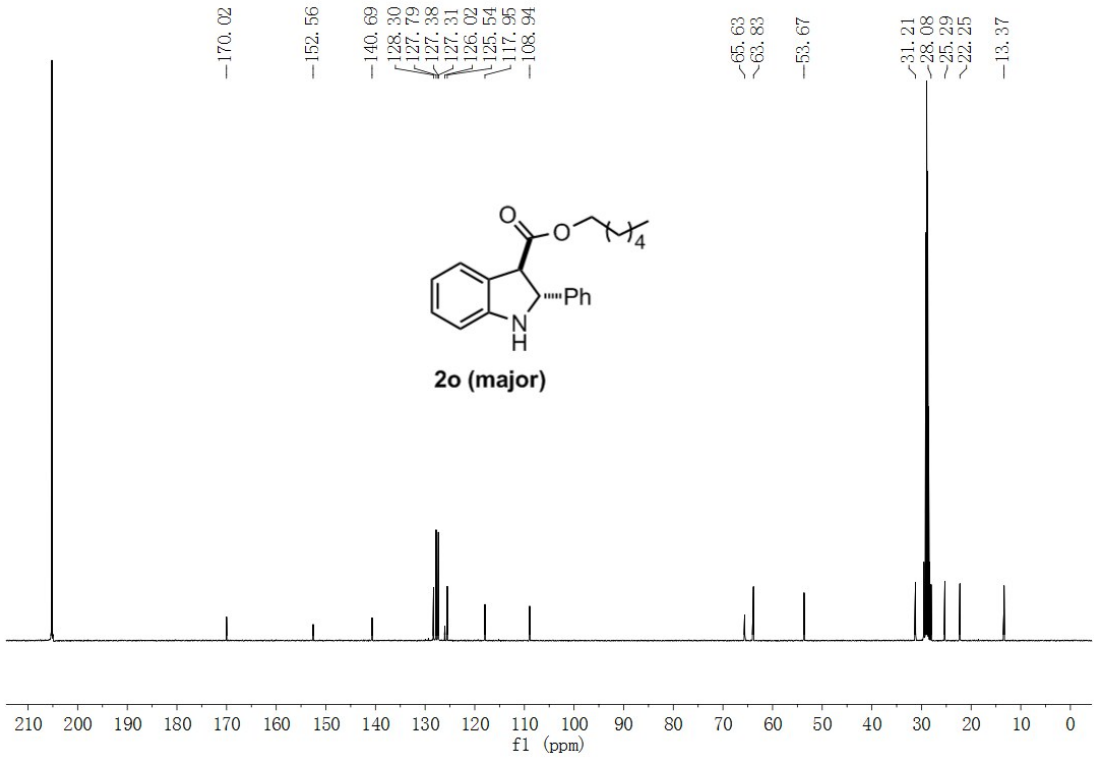

7.1057
7.0873
7.0558
6.9955
6.9909
6.9139
6.8933
6.8886
6.8398
6.8192
6.7088
6.6893
6.6828
6.6637
5.3176
5.2153
5.1904
4.4247
4.3999
3.7520
3.7120
3.6919
3.6887
3.6706
3.6528
3.6436
3.6350
3.6258
0.8605
0.8427
0.8249
CO2Et
OMe
OMe
N
H
2m (major)
3.08
2.25
1.95
0.75
1.16
0.99
3.30
3.14
1.80
3.25
7.0 6.5 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)

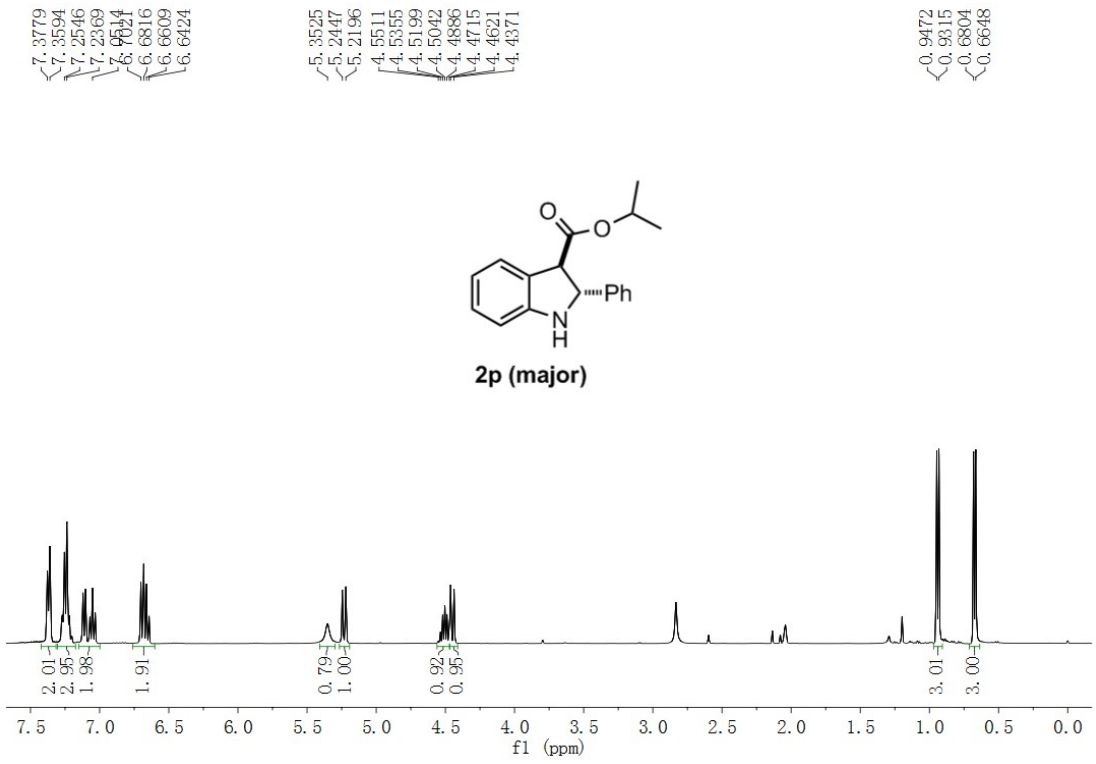

170.19
152.56
149.08
149.00
133.11
128.29
126.10
125.61
119.66
117.98
111.61
111.55
108.97
65.46
59.66
55.36
55.24
53.62
13.32
CO2Et
OMe
OMe
N
H
2m (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

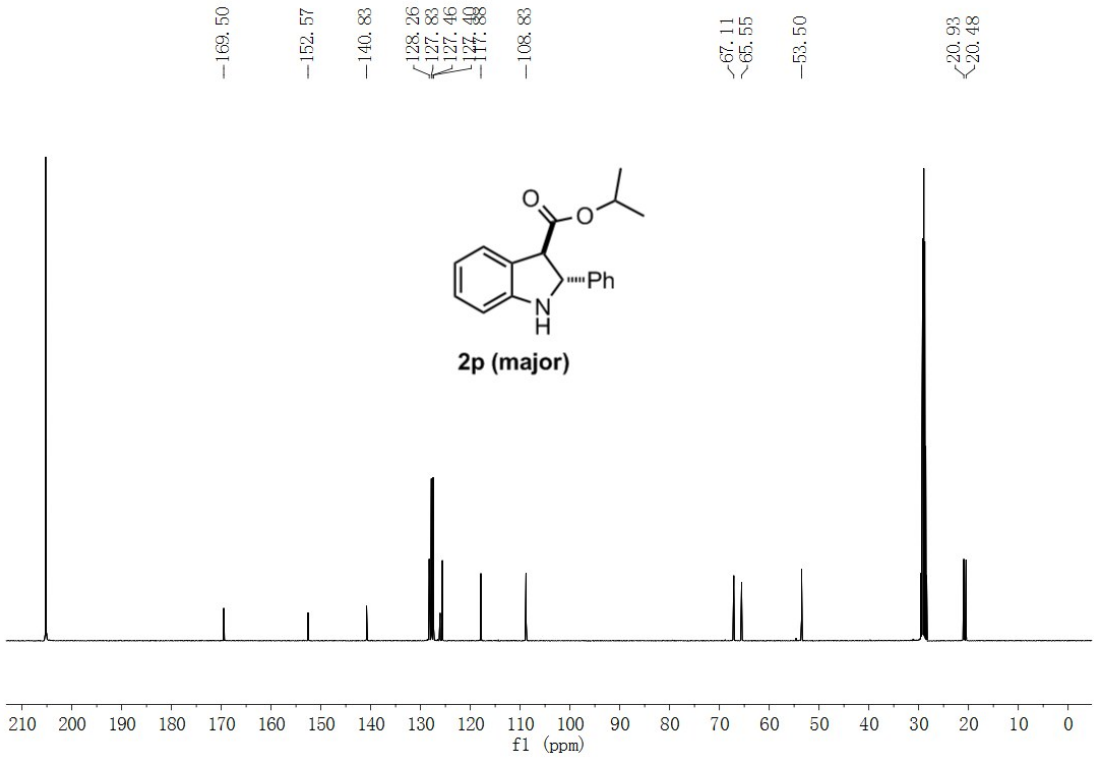

7.4218
7.4058
7.3898
7.0561
7.0362
7.0213
6.7018
6.6897
6.6710
6.6525
5.4570
5.3069
5.2819
4.4914
4.4664
3.2061
CO2Me
F
N
H
2n (major)
2.07
4.13
2.09
0.87
1.05
1.00
3.08
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

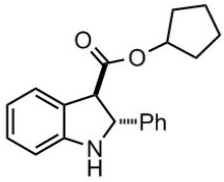

170.40
163.37
160.95
152.52
136.67
136.64
129.15
129.07
128.46
125.61
125.58
118.09
114.52
114.31
108.95
64.85
53.73
50.45
CO2Me
F
N
H
2n (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.3861
7.3676
7.2634
6.6993
6.6840
6.6655
6.6470
5.3869
5.2783
5.2534
4.4942
4.4694
3.6598
3.6436
3.6315
3.6169
3.6003
3.5566
3.5406
3.5258
3.5138
3.4971
1.2622
1.2446
1.2272
1.2099
1.1929
1.1760
1.1574
1.1375
1.1235
1.1075
1.0909
0.8809
0.8632
0.8451
O
O
4
Ph
N
H
2o (major)
2.08
3.12
2.12
1.95
0.79
1.04
1.00
2.16
8.06
3.35
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)

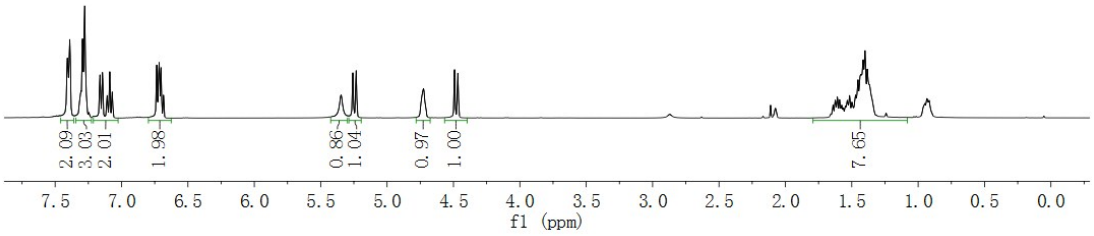

170.02
152.56
140.69
128.30
127.79
127.38
127.31
126.02
125.54
117.95
108.94
65.63
63.83
53.67
31.21
28.08
25.29
22.25
13.37
O
O
4
Ph
N
H
2o (major)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

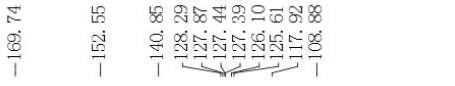

7.3779
7.3594
7.2546
7.2369
7.0514
7.0021
6.6816
6.6609
6.6424
5.3525
5.2447
5.2196
4.5511
4.5355
4.5199
4.5042
4.4886
4.4715
4.4621
4.4371
0.9472
0.9315
0.6804
0.6648
Ph
N
H
2p (major)
2.01
2.95
1.98
1.91
0.79
1.00
0.92
0.95
3.01
3.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

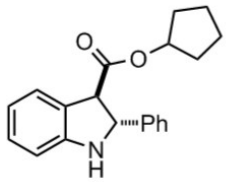

169.50
152.57
140.83
128.26
127.83
127.46
108.83
67.11
65.55
53.50
20.93
20.48
Ph
N
H
2p (major)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



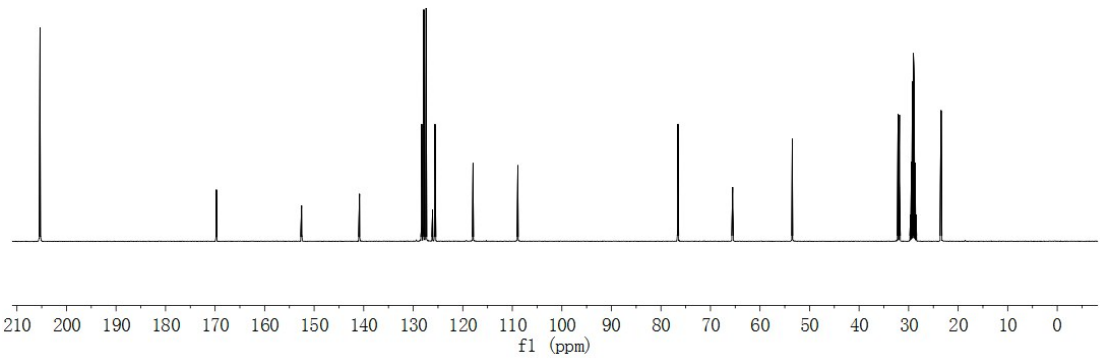
2q (major)

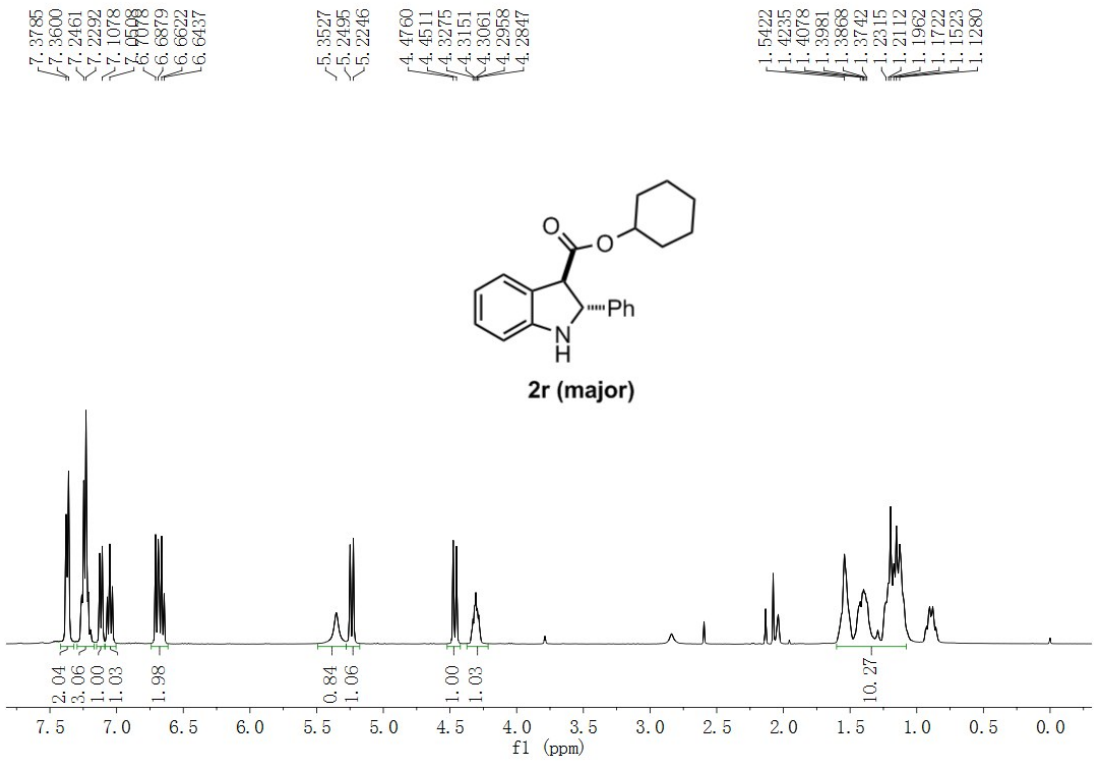


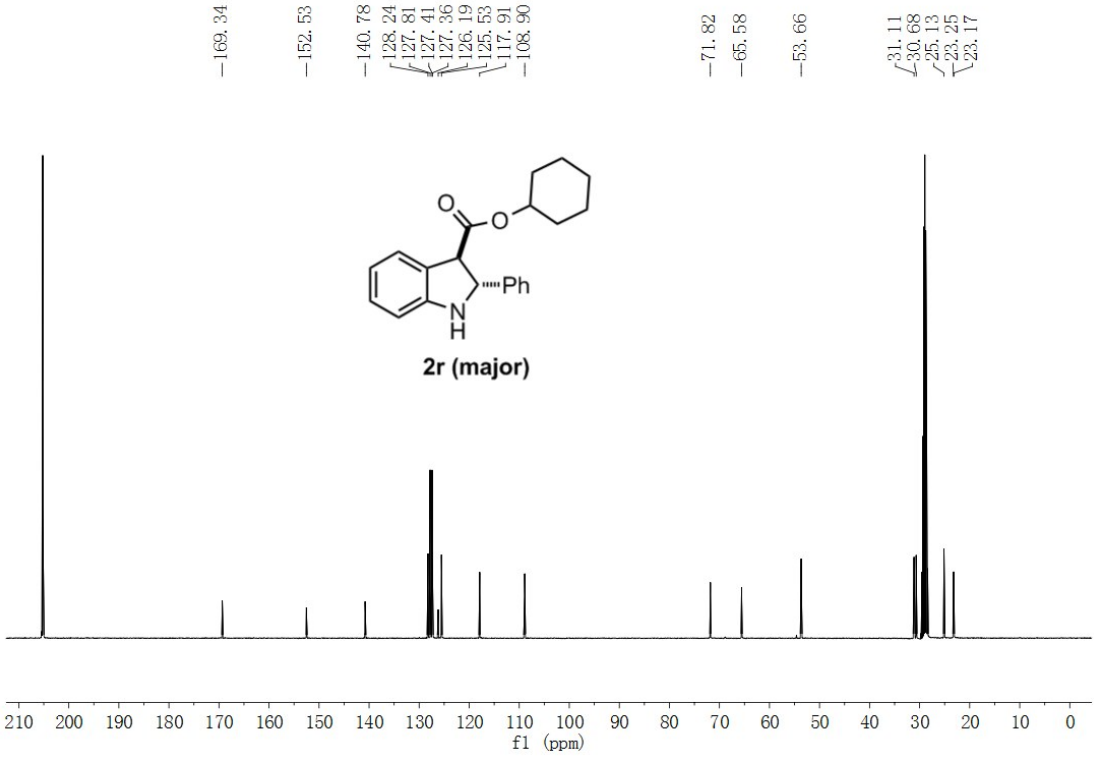


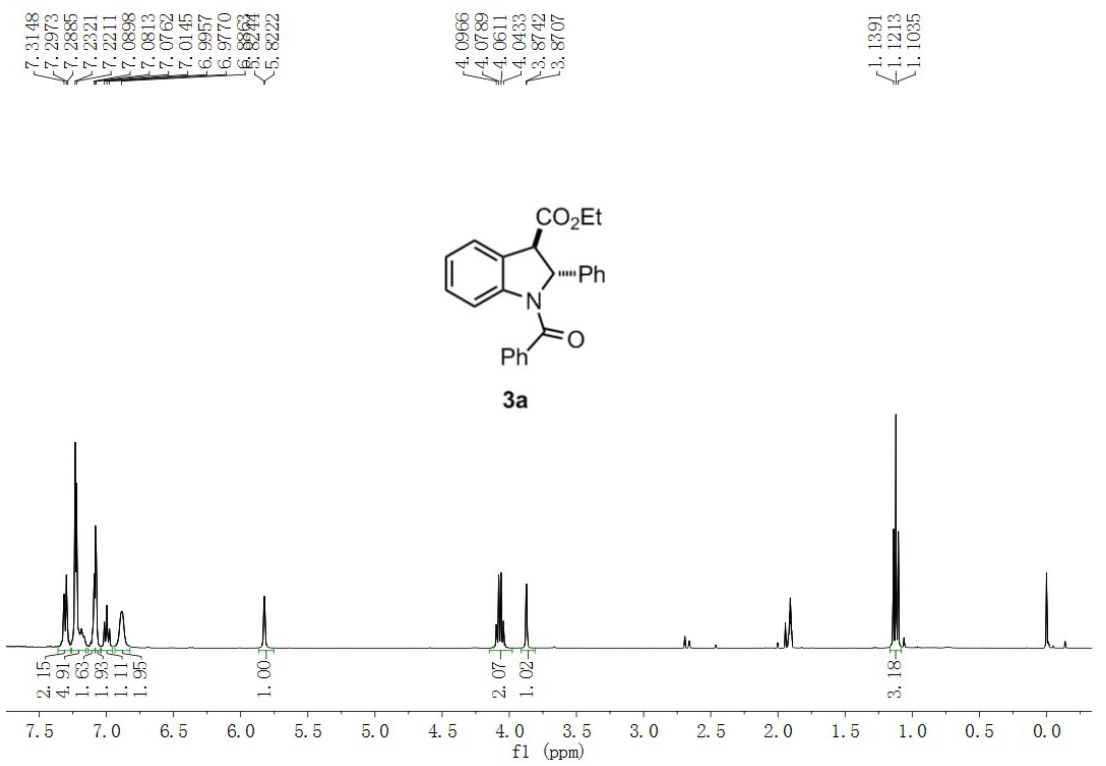


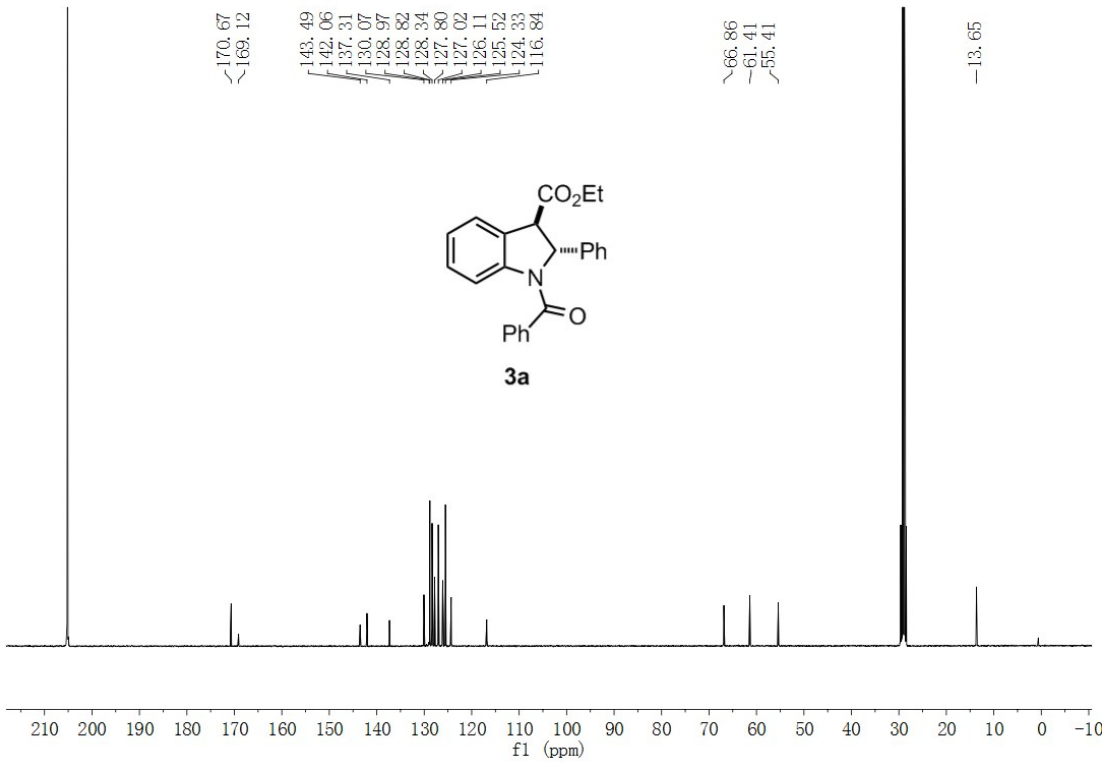
2q (major)




7.3785
7.3600
7.2461
7.2292
7.1078
7.0508
6.6879
6.6622
6.6437
5.3527
5.2495
5.2246
4.4760
4.4511
4.3275
4.3151
4.3061
4.2958
4.2847
1.5422
1.4235
1.4078
1.3981
1.3868
1.3742
1.2315
1.2112
1.1962
1.1722
1.1523
1.1280
Ph
2r (major)
2.04
3.06
1.00
1.03
1.98
0.84
1.06
1.00
1.03
10.27
f1 (ppm)


169.34
152.53
140.78
128.24
127.81
127.41
127.36
126.19
125.53
117.91
108.90
71.82
65.58
53.66
31.11
30.68
25.13
23.25
23.17
Ph
2r (major)
f1 (ppm)


7.3148
7.2973
7.2885
7.2321
7.2211
7.0898
7.0813
7.0762
7.0145
6.9957
6.9770
5.8222
4.0966
4.0789
4.0611
4.0433
3.8742
3.8707
1.1391
1.1213
1.1035
CO2Et
Ph
N
O
Ph
3a
2.15
4.91
1.63
1.93
1.11
1.95
1.00
2.07
1.02
3.18
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)


170.67
169.12
143.49
142.06
137.31
130.07
128.97
128.82
128.34
127.80
127.02
126.11
125.52
124.33
116.84
66.86
61.41
55.41
13.65
CO2Et
Ph
N
O
Ph
3a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)