## Superhydrophobic Nickel/Carbon Core-shell nanocomposites for the

### hydrogen transfer reactions of nitrobenzene and N-heterocycles

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# **Supporting Information**

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#### I. Materials and reagents

Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O was obtained from Xi long Chemical Co., Ltd.(Guangdong, China). PVP (K30) was purchased from Shanghai Maclean Biochemical Technology Co., Ltd., China. All other solvents and chemicals were obtained commercially and were used as received.

#### **II.** Characterization methods

The local (the Yu Zhong campus of Northwest Minzu University, China) atmospheric pressure was measured with the precision pressure gauge (FYP-2A), and the result was 0.0814 Mpa. The actual pressure in the model catalytic reaction at 145°C was measured by the autoclave with a large pressure range (0.01 MPa to 10 MPa) and the same volume as the pressure tube, the autogenic pressure was 0.35 Mpa. The structures of as-prepared samples were observed by FE-SEM. SEM was performed with a JEOL JSM-6701F equipped with a cold FEG (Field Emission Gun). Transmission electron microscopy (TEM) characterization was carried out by using a Tecnai G2 F30 S-Twin transmission electron microscope operating at 300 kV. Single-particle EDX analysis was performed by using a Tecnai G2 F30 S-Twin Field Emission TEM in STEM mode. For TEM investigations, the catalysts were dispersed in ethanol by ultrasonication and deposited on carbon-coated copper grids. XRD measurements were performed by using a STADIP automated transmission diffractometer (STOE) equipped with an incident beam curved germanium monochromator with CuKa1 radiation. The XRD patterns were scanned in the 20 range of 5-85°. For data interpretation, WinXpow software (STOE) and the database of powder diffraction files (PDF) of the International Centre of Diffraction Data (ICDD) were used. Raman spectra were measured with a 532 nm edge by using a LabRAM HR Evolution (HORIBA Jobin Yvon S.A.S.). FT-IR spectra were registered in the 400-4000 cm<sup>-1</sup> region with a resolution of 1 cm<sup>-1</sup> by a Nicolet 5700 spectrometer. For each FT-IR spectrum, the 0.5 mg of sample was uniformly mixed with 100 mg of potassium bromide, and then the mixture was laminated with a tablet press for further analysis. The BET surface areas and pore-size distribution was calculated from the desorption isotherm by using the Barrett, Joyner, and Halenda (BJH) method. WCA (water contact angle) was measured by a POWEREACH JC2000D goniometer (Made in China). The wettability of the samples were evaluated by WCA measurement of ~ 1  $\mu$ L water droplets on the surface using a micrometer syringe. At least five measurements were carried out to obtain each value. The error in contact angle measurements was  $\pm 2^{\circ}$ . The profiles of the contact angle were photographed using the digital camera of the goniometer. The Vario EL microanalyzer was used to analyze the element composition (N, C, O, H) of the samples. The X-ray photoelectron spectroscopy (XPS) measurements were performed by using a VG ES-CALAB 210 instrument equipped with a dual Mg/Al anode X-ray source, a hemispherical capacitor analyzer, and a 5 keV Ar<sup>+</sup> ion gun. All spectra were registered by using nonmonochromatic MgKa (1253.6 eV) radiation. The samples were attached to a stainless steel sample holder by using double-sided adhesive carbon tape. The electron binding energy was referenced to the C1s peak at 284.8 eV. The peaks were fitted by Gaussian-Lorentzian curves after Shirley background subtraction. For quantitative analysis, the peak area was shared by the element-specific Scofield factor and the transmission function of the analyzer. The background pressure in the chamber was less than 10<sup>-7</sup> Pa. The Ni content of the catalysts were measured by using inductively coupled plasma atomic emission spectrometry (ICP-AES), by using an Iris advantage Thermo Jarrel Ash device. NMR spectra were measured by using a Bruker ARX 400 or ARXS2100 spectrometer at 400 MHz (<sup>1</sup>H) and 100 MHz (<sup>13</sup>C). All spectra were recorded in CDCl<sub>3</sub> and chemical shifts (d) are reported in ppm relative to tetramethylsilane referenced to the residual solvent peaks. In addition. All the digital photos were shot with Canon EOS-60D camera.

Catalyst	Ni@NCF-600	Ni@NCF-700	Ni@NCF-800	Ni@NCF-900	Ni@1/2-NCF-600	Ni@2-NCF-600
S <sub>BET</sub> /m <sup>2</sup> •g <sup>-1</sup>	206	263	215	159	96	5

Table S1. The BET surface area of Ni@NCF

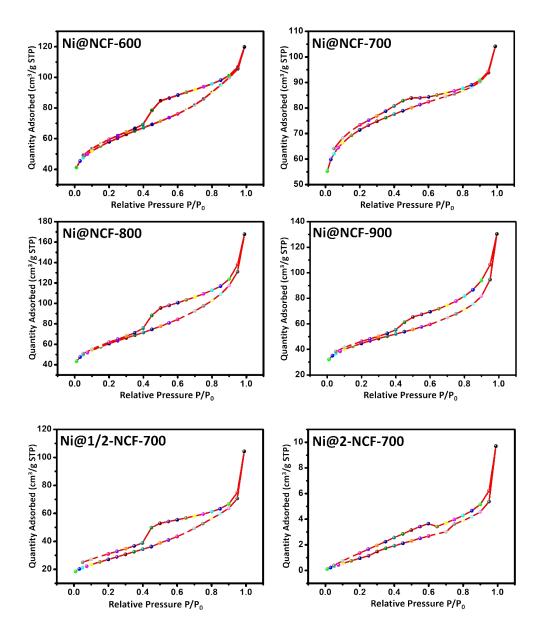


Figure S1. BJH Desorption patterns of prepared catalysts.

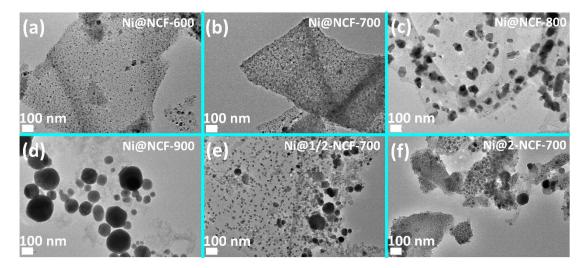


Figure S2. TEM images of Ni@NCFs.

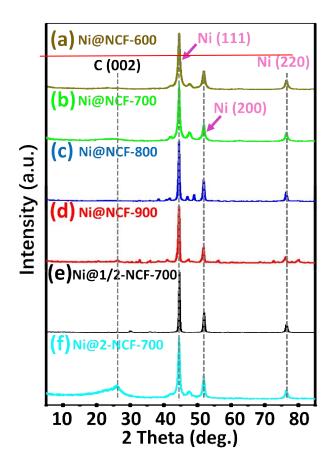


Figure S3. XRD diffraction patterns of Ni@NCFs.

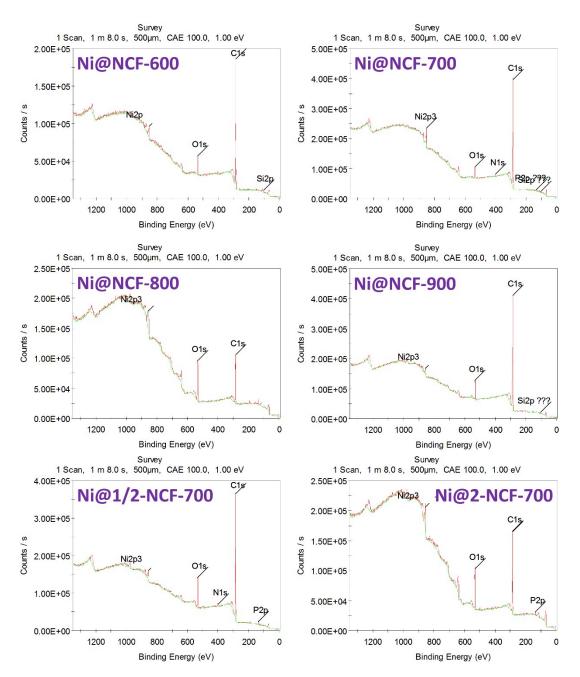


Figure S4. XPS spectra of prepared catalysts.

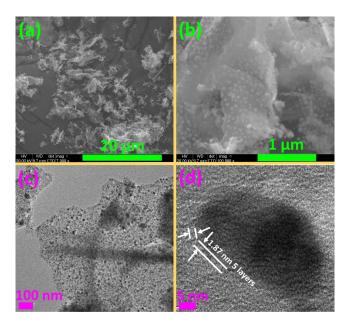


Figure S5. SEM images at low magnification (a), at high magnification (b), TEM (c)

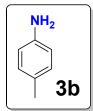
and HR-TEM (d) images after Ni@NCF-700 fresh catalyst used three times.

## IV. NMR peaks and MS-EI of all products

3b-3p and 4a-4m:

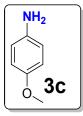
## NMR peaks and MS-EI of products

Acquired NMR peaks matched those of the literature.<sup>1-8</sup>



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 6.95 (d, *J* = 7.9 Hz, 2H), 6.74-6.31 (m, 2H), 3.50 (s, 2H), 2.22 (s, 3H).

 $^{13}C$  NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  143.74, 129.72, 127.78, 115.22, 20.45. MS-EI calculated for 107.0735, found 107.0762.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  6.73 (d, *J* = 8.9 Hz, 2H), 6.68-6.58 (m, 2H), 3.73 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  152.77, 139.81, 116.41, 114.74, 55.71. MS-EI calculated for 123.0684, found 123.0665.



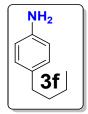
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.07 (t, *J* = 7.7 Hz, 1H), 6.76-6.21 (m, 3H), 3.58 (s, 2H), 2.56 (q, *J* = 7.6 Hz, 2H), 1.21 (t, *J* = 7.6 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 146.34, 145.55, 129.20, 118.26, 114.72, 112.50, 28.84, 15.51.

MS-EI calculated for 121.0891, found 121.0871.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.12-6.99 (m, 2H), 6.76 (td, J = 7.4, 1.0 Hz, 1H), 6.68 (dd, J = 7.8, 0.9 Hz, 1H), 3.62 (s, 2H), 2.52 (q, J = 7.5 Hz, 2H), 1.26 (t, J = 7.6 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.96, 128.32, 128.04, 126.80, 118.79, 115.34, 23.99, 12.99. MS-EI calculated for 121.0891, found 121.0864.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  6.96 (d, J = 8.4 Hz, 2H), 6.61 (d, J = 8.4 Hz, 2H), 3.37 (s, 2H), 2.55-2.38 (m, 2H), 1.59-1.45 (m, 2H), 1.32 (m, J = 14.5, 7.3 Hz, 2H), 0.90 (t, J = 7.3 Hz, 3H).

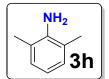
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.94, 133.07, 129.12, 115.19, 34.75, 33.99, 22.31, 14.00.

MS-EI calculated for 149.1204, found 149.1217.



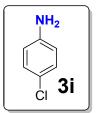
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  6.44-6.39 (m, 1H), 6.33 (d, J = 0.5 Hz, 2H), 3.53 (s, 2H), 2.23 (d, J = 0.5 Hz, 6H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 146.31, 139.00, 120.46, 113.05, 21.32. MS-EI calculated for 121.0891, found 121.0878.

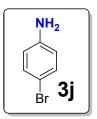


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  6.93 (d, *J* = 7.4 Hz, 2H), 6.62 (d, *J* = 7.5 Hz, 1H), 3.56 (s, 2H), 2.17 (s, 6H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 142.67, 128.20, 121.67, 117.95, 17.63. MS-EI calculated for 121.0891, found 121.0873.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.14-7.01 (m, 2H), 6.63-6.53 (m, 2H), 3.59 (s, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.87, 129.08, 123.13, 116.20. MS-EI calculated for 127.0189, found 127.0174.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.39-6.99 (m, 2H), 6.88-6.12 (m, 2H), 3.65 (s, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 145.36, 131.97, 116.67, 110.17. MS-EI calculated for 170.9684, found 170.9695.

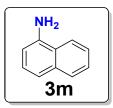


<sup>1</sup>H NMR (400 MHz, cdcl<sub>3</sub>)  $\delta$  8.04 (dd, *J* = 5.1, 1.0 Hz, 1H), 7.40 (m, *J* = 8.2, 7.3, 1.9 Hz, 1H), 7.03-6.05 (m, 2H), 4.71-3.88 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  158.31, 148.09, 137.73, 114.01, 108.58. MS-EI calculated for 94.0531, found 94.0543.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  6.81 (d, *J* = 7.8 Hz, 1H), 6.07 (dt, *J* = 5.7, 2.3 Hz, 2H), 3.47 (s, 4H), 2.05 (s, 3H).

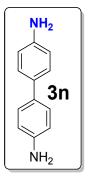
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 145.46, 145.23, 131.08, 112.89, 105.98, 102.11, 16.47. MS-EI calculated for 122.0844, found 122.0859.



<sup>1</sup>H NMR (400 MHz, cdcl<sub>3</sub>)  $\delta$  7.86-7.70 (m, 2H), 7.51-7.39 (m, 2H), 7.33-7.25 (m, 2H), 6.78 (dd, J = 6.8, 1.6 Hz, 1H), 4.14 (s, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 142.00, 134.33, 128.52, 126.30, 125.82, 124.85, 123.60, 120.75, 118.97, 109.66.

MS-EI calculated for 143.0735, found 143.0761.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 (d, J = 8.2 Hz, 4H), 6.71 (d, J = 8.2 Hz, 4H), 3.65 (s, 4H).

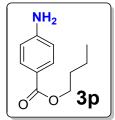
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.90, 131.77, 127.26, 115.41. MS-EI calculated for 184.1000, found 184.1017.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.69 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.29-7.17 (m, 1H), 6.74-6.53 (m, 2H), 6.26 (s, 2H), 2.56 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 200.77, 150.22, 134.37, 132.02, 118.18, 117.17, 115.72, 27.88.

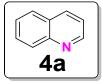
MS-EI calculated for 135.0684, found 135.0692.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.84 (d, J = 8.6 Hz, 2H), 6.62 (d, J = 8.6 Hz, 2H), 4.24 (t, J = 6.6 Hz, 2H), 4.02 (s, 2H), 1.70 (dt, J = 14.5, 6.6 Hz, 2H), 1.52-1.30 (m, 2H), 0.95 (t, J = 7.4 Hz, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 166.74, 150.63, 131.52, 120.11, 113.75, 64.21, 30.86, 19.29, 13.80.

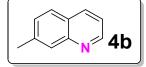
MS-EI calculated for 193.1103, found 193.1135.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.93 (dd, J = 4.2, 1.7 Hz, 1H), 8.24-8.01 (m, 2H), 7.82 (d, J = 8.2 Hz, 1H), 7.72 (m, J = 8.4, 6.9, 1.4 Hz, 1H), 7.61-7.48 (m, 1H), 7.40 (dd, J = 8.3, 4.2 Hz, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.43, 148.28, 136.08, 129.47, 128.30, 127.81, 126.56, 121.10.

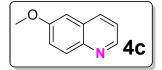
MS-EI calculated for 129.0578, found 129.0583.



<sup>1</sup>H NMR (400 MHz, cdcl<sub>3</sub>)  $\delta$  8.83 (dd, J = 4.2, 1.6 Hz, 1H), 8.02 (dd, J = 30.1, 8.4 Hz, 2H), 7.66-7.46 (m, 2H), 7.35 (dd, J = 8.3, 4.2 Hz, 1H), 2.53 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  149.52, 146.84, 136.38, 135.37, 131.74, 129.06, 128.28,

126.56, 121.06, 21.59.

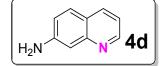
MS-EI calculated for 143.0735, found 143.0751.



<sup>1</sup>H NMR (400 MHz, cdcl<sub>3</sub>)  $\delta$  8.75 (dd, J = 4.2, 1.6 Hz, 1H), 8.14-7.81 (m, 2H), 7.48-7.26 (m, 2H), 7.05 (d, J = 2.8 Hz, 1H), 3.92 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 157.66, 147.93, 144.38, 134.77, 130.83, 129.25, 122.27, 121.35, 105.03, 55.52.

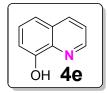
MS-EI calculated for 159.0684, found 159.0672.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.73 (dd, *J* = 4.3, 1.7 Hz, 1H), 7.95 (dd, *J* = 8.1, 1.3 Hz, 1H), 7.58 (d, *J* = 8.7 Hz, 1H), 7.18 (t, *J* = 5.0 Hz, 1H), 7.11 (dd, *J* = 8.1, 4.3 Hz, 1H), 7.00-6.86 (m, 1H), 4.16 (d, *J* = 94.4 Hz, 2H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 150.66, 149.92, 147.64, 135.70, 128.94, 122.25, 118.62, 117.76, 109.20.

MS-EI calculated for 144.0687, found 144.0673.



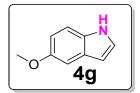
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.77 (dd, J = 4.2, 1.5 Hz, 1H), 8.14 (dd, J = 8.3, 1.5 Hz, 1H), 7.51-7.39 (m, 2H), 7.32 (dd, J = 8.3, 1.0 Hz, 1H), 7.17 (dd, J = 7.6, 1.1 Hz, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  152.15, 147.87, 138.26, 136.09, 128.46, 127.71, 121.82, 117.86, 109.93.

MS-EI calculated for 145.0528, found 145.0531.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.08 (dd, *J* = 4.2, 1.6 Hz, 1H), 9.00 (d, *J* = 2.2 Hz, 1H), 8.41-8.18 (m, 2H), 7.97 (d, *J* = 9.0 Hz, 1H), 7.59 (dd, *J* = 8.4, 4.2 Hz, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  152.67, 147.11, 135.91, 131.35, 129.46, 125.84, 123.92, 120.09.

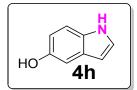
MS-EI calculated for 174.0429, found 174.0417.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.06 (s, 1H), 7.26 (d, J = 8.8 Hz, 1H), 7.14 (dd, J = 8.4, 2.7 Hz, 2H), 6.89 (dd, J = 8.8, 2.4 Hz, 1H), 6.54-6.42 (m, 1H), 3.87 (s, 3H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 154.12, 130.93, 128.24, 124.94, 112.32, 111.77, 102.31, 102.25, 55.85.

MS-EI calculated for 147.0684, found 147.0695.



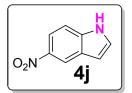
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.04 (s, 1H), 7.24 (t, *J* = 4.3 Hz, 2H), 7.17 (t, *J* = 2.8 Hz, 1H), 7.03 (d, *J* = 2.4 Hz, 1H), 6.76 (dd, *J* = 8.6, 2.5 Hz, 1H), 6.42 (s, 1H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  149.51, 125.21, 111.76, 111.58, 105.00, 102.00. MS-EI calculated for 133.0528, found 133.0516.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.25 (s, 1H), 7.40-7.25 (m, 2H), 7.26-7.19 (m, 1H), 7.05 (t, *J* = 7.9 Hz, 1H), 6.61 (dd, *J* = 3.8, 1.6 Hz, 1H).

<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 135.96, 128.63, 124.72, 122.91, 122.74, 114.72, 110.24, 103.00.

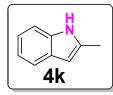
MS-EI calculated for 194.9684, found 194.9675.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.60 (s, 1H), 8.10 (dd, *J* = 9.0, 2.2 Hz, 1H), 7.43 (d, *J* = 9.0 Hz, 1H), 7.38-7.35 (m, 1H), 6.79-6.65 (m, 1H).

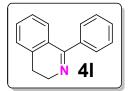
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 142.63, 138.80, 129.13, 127.19, 118.00, 117.70, 110.99, 105.08.

MS-EI calculated for 162.0429, found 162.0438.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.77 (s, 1H), 7.56-7.48 (m, 1H), 7.31-7.21 (m, 1H), 7.10 (m, J = 16.0, 7.1, 1.3 Hz, 2H), 6.23 (dt, J = 2.0, 0.9 Hz, 1H), 2.43 (d, J = 0.8 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  135.99, 135.07, 129.01, 120.89, 119.60, 110.21, 100.33, 13.71.

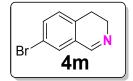
MS-EI calculated for 131.0735, found 131.0726.



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.63-7.53 (m, 2H), 7.45-7.33 (m, 4H), 7.24 (t, *J* = 3.9 Hz, 3H), 3.86-3.81 (m, 2H), 2.78 (d, *J* = 7.2 Hz, 2H).

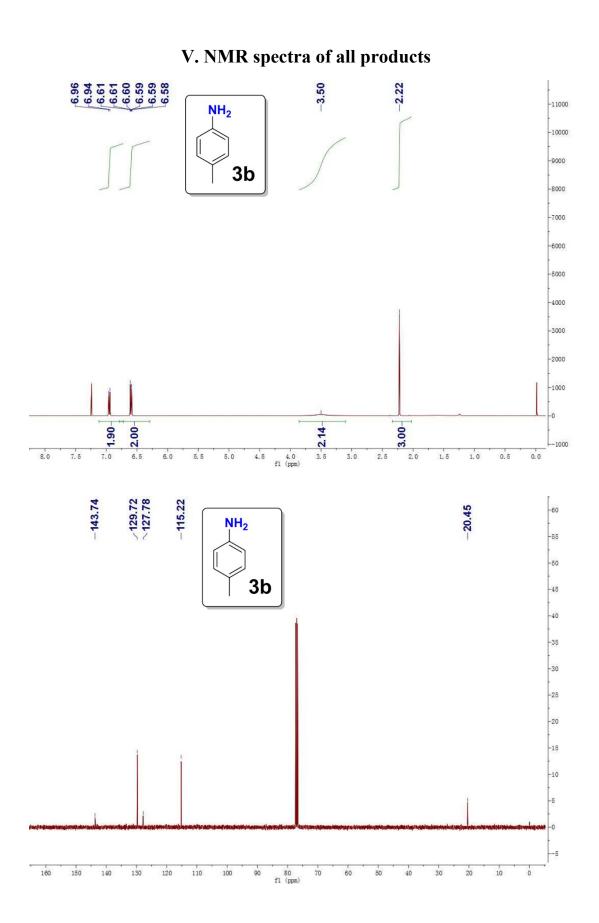
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 167.26, 138.81, 130.65, 129.29, 128.76, 128.12, 127.91, 127.37, 126.53, 47.60, 26.28.

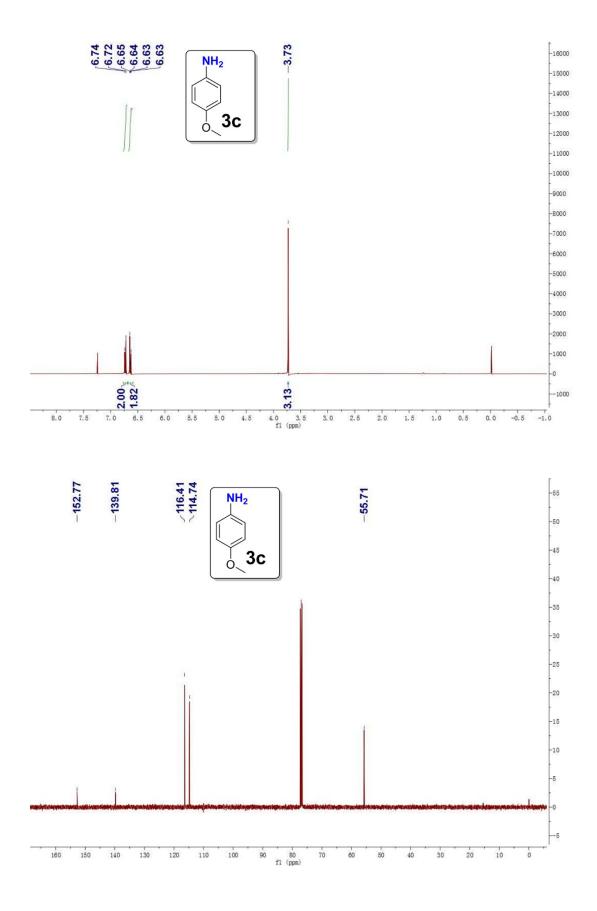
MS-EI calculated for 207.1048, found 207.1028.

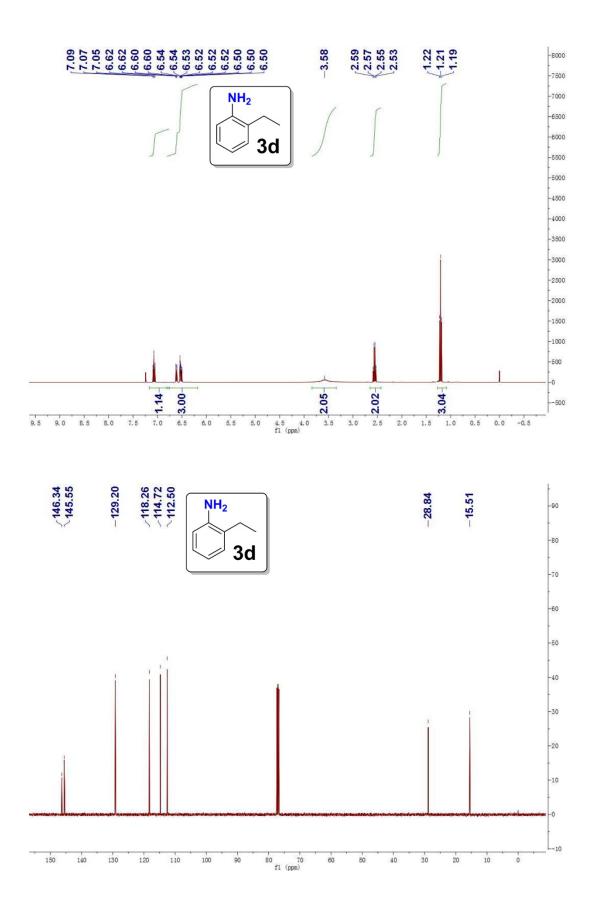


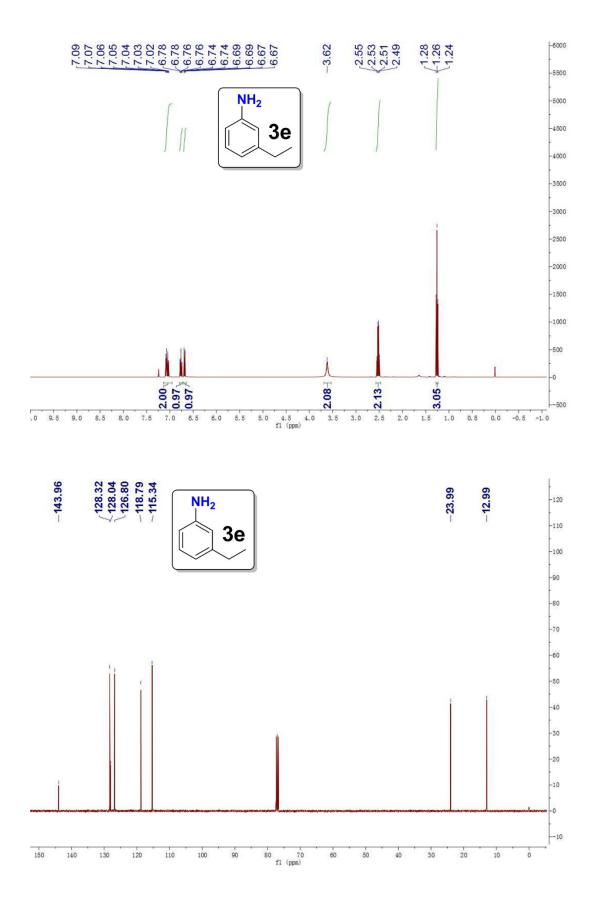
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.26 (s, 1H), 7.46 (dd, J = 8.0, 2.0 Hz, 1H), 7.39 (d, J = 1.9 Hz, 1H), 7.03 (d, J = 8.0 Hz, 1H), 3.82-3.63 (m, 2H), 2.79-2.53 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  158.90, 135.02, 133.81, 130.01, 129.11, 120.39, 47.21, 24.43.

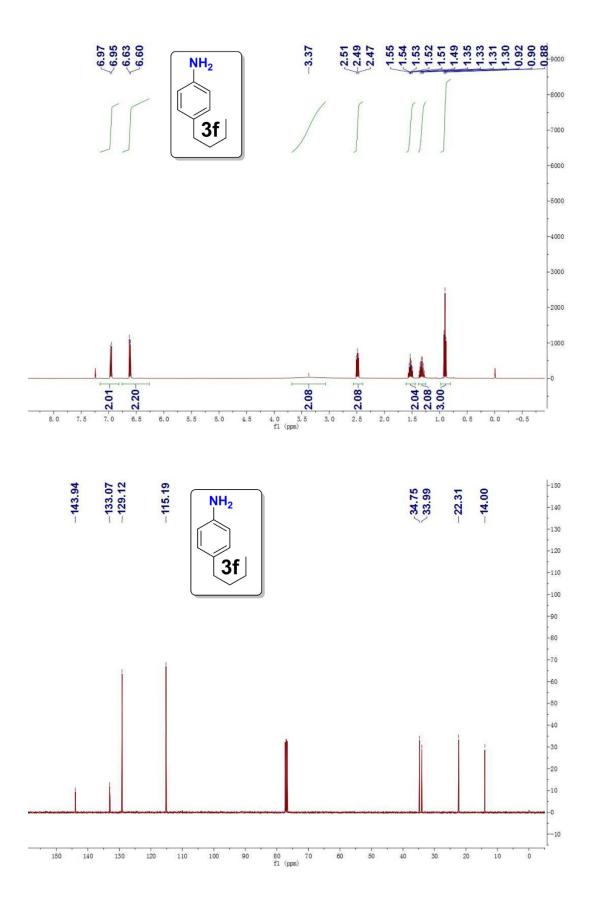
MS-EI calculated for 208.9840, found 208.9851.

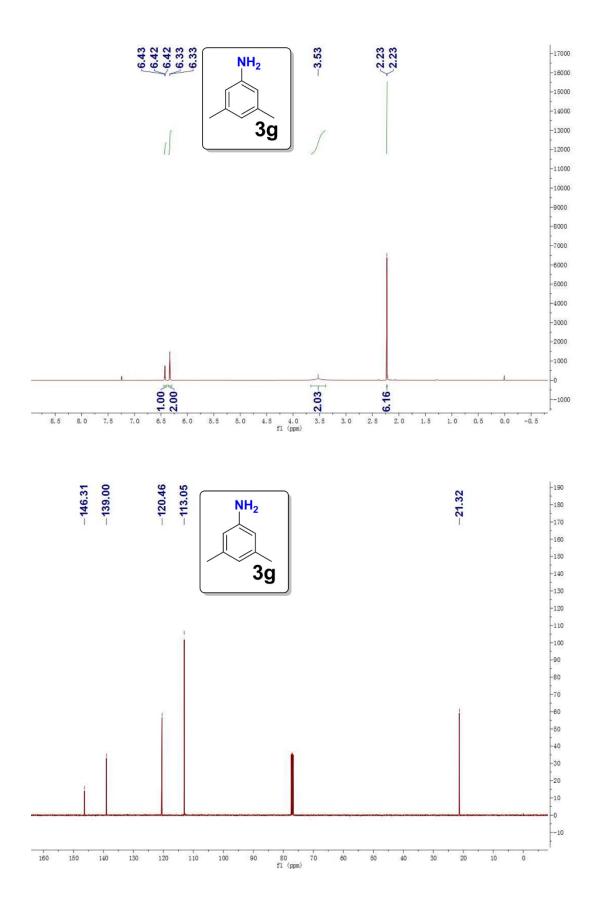




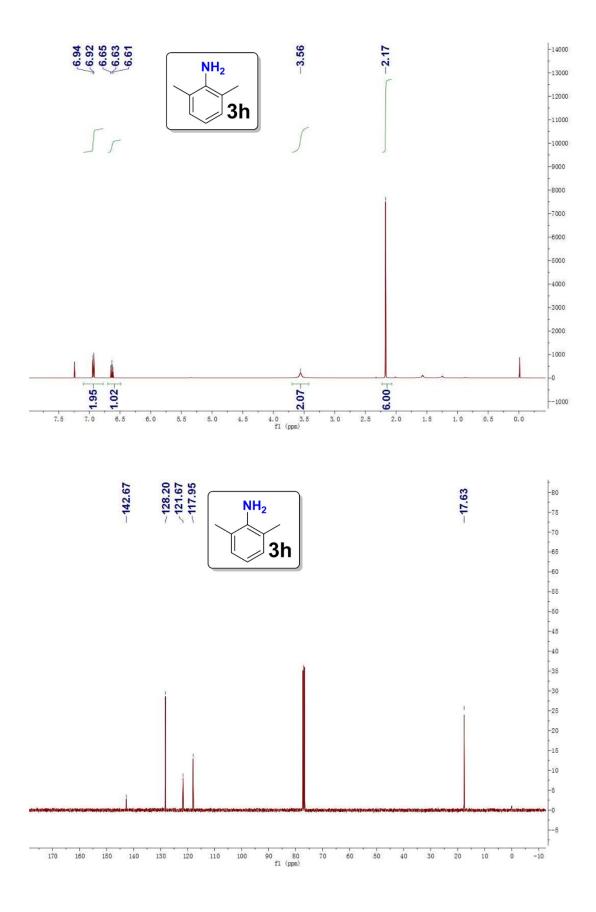


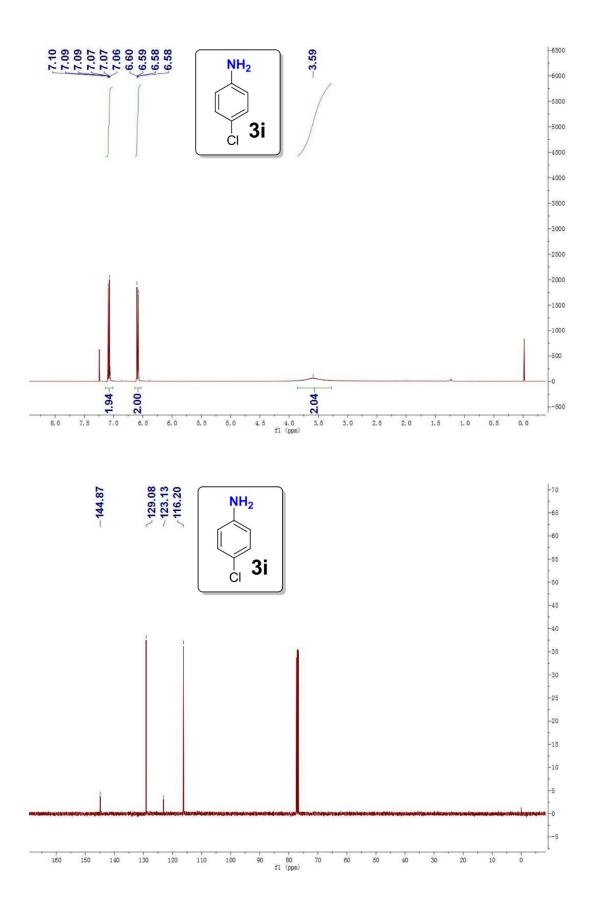


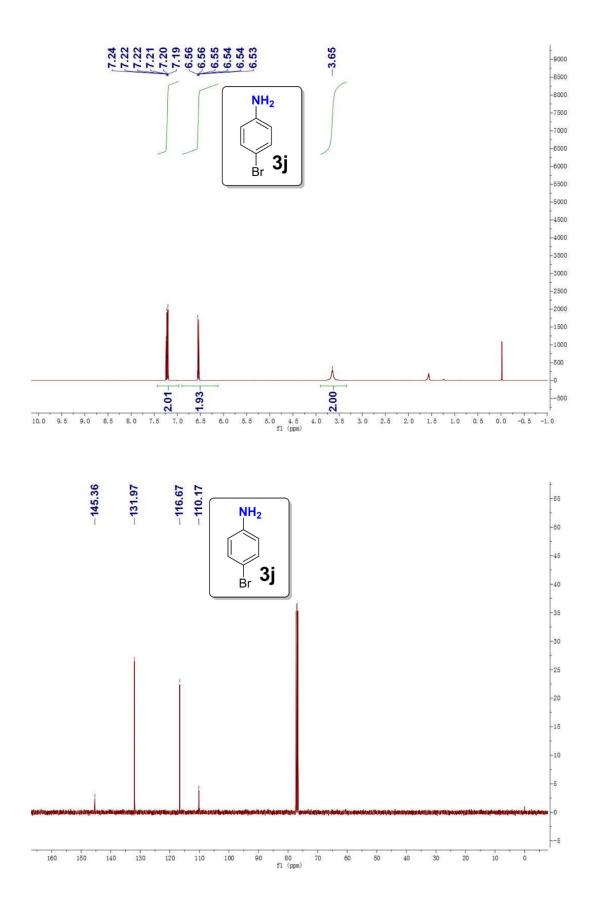


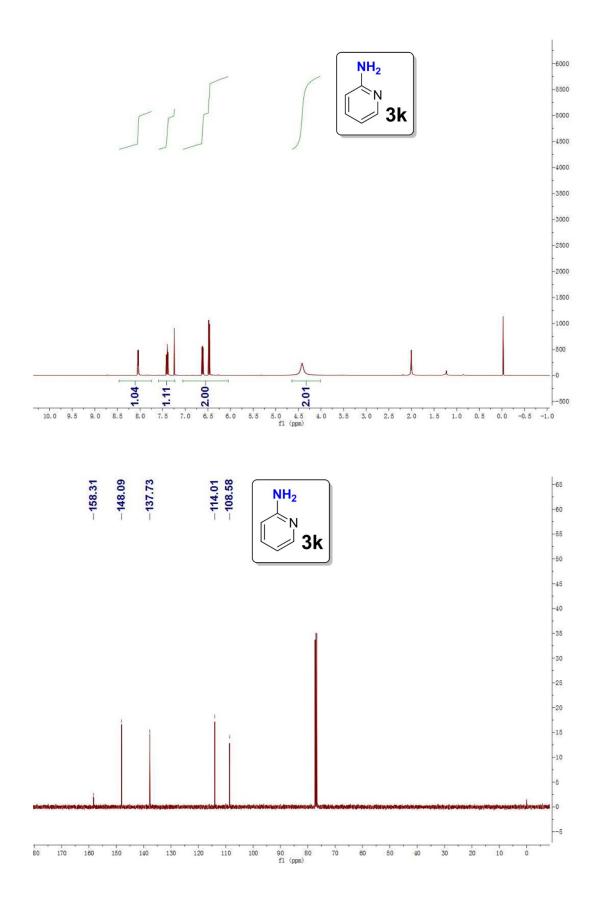


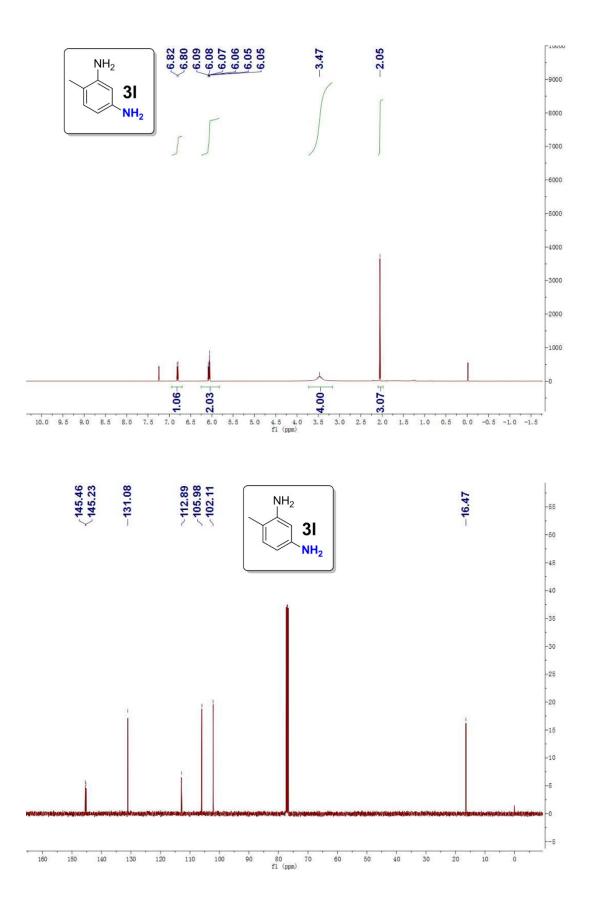
S19

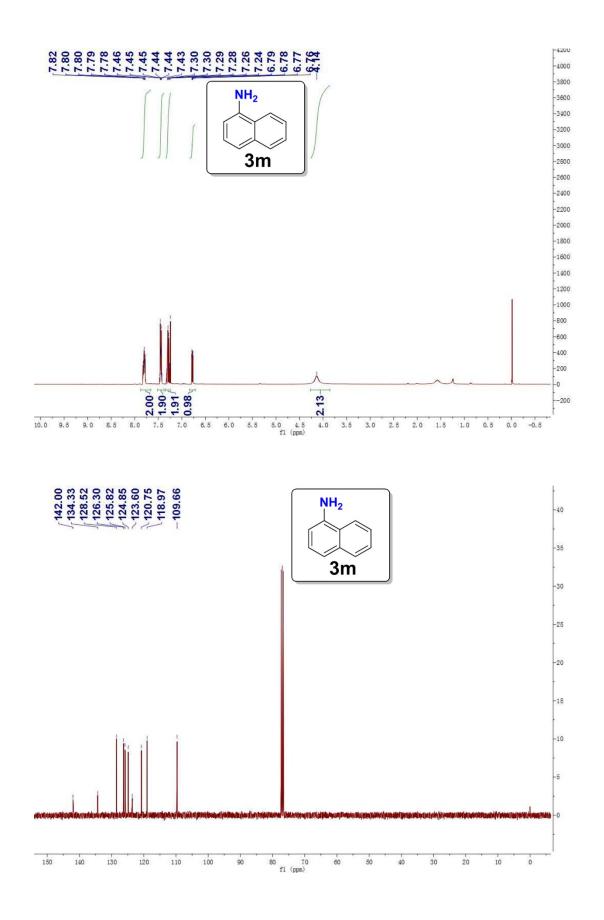


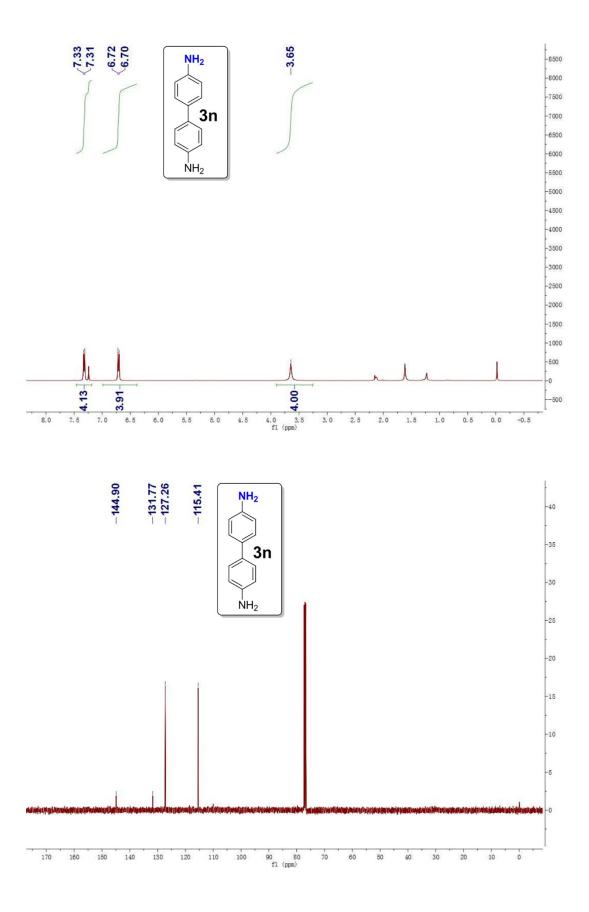


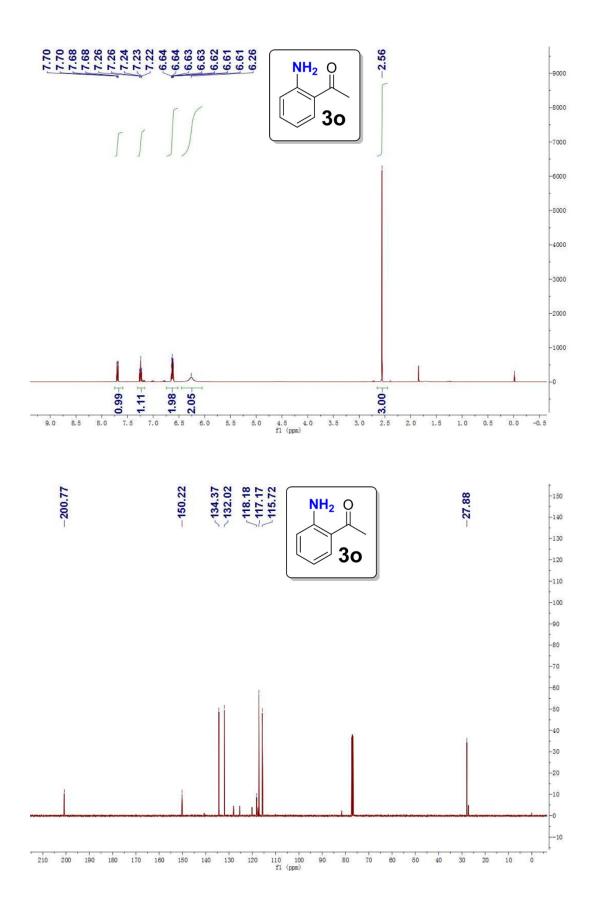


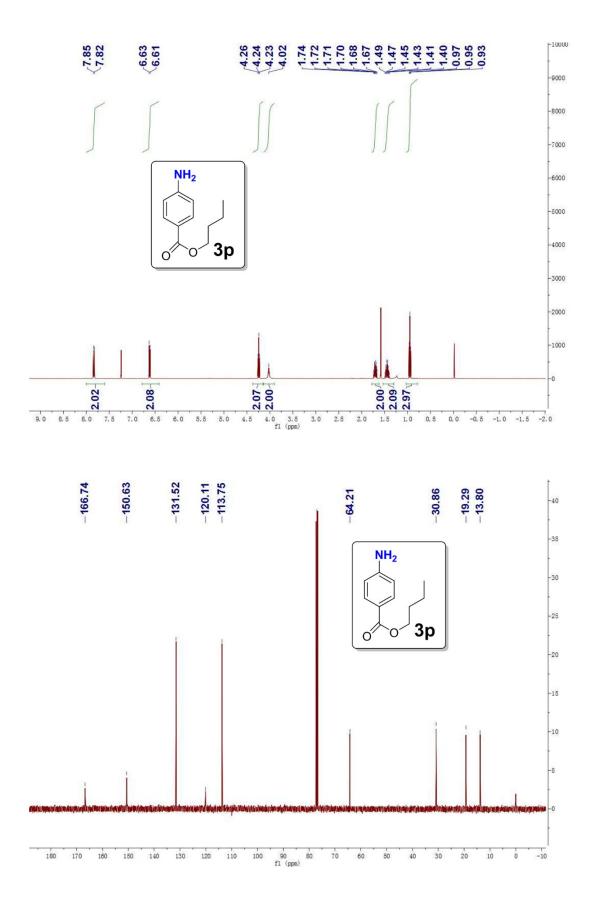


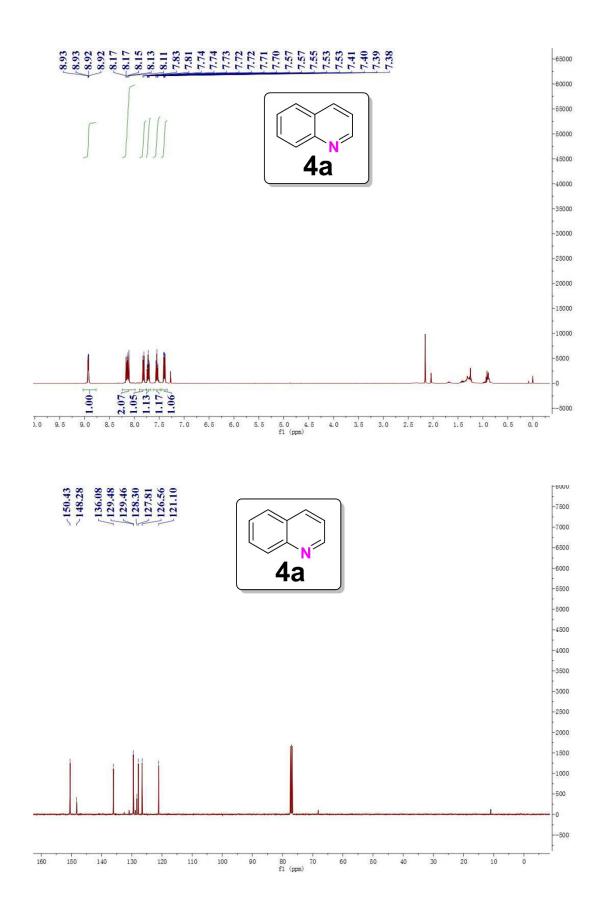


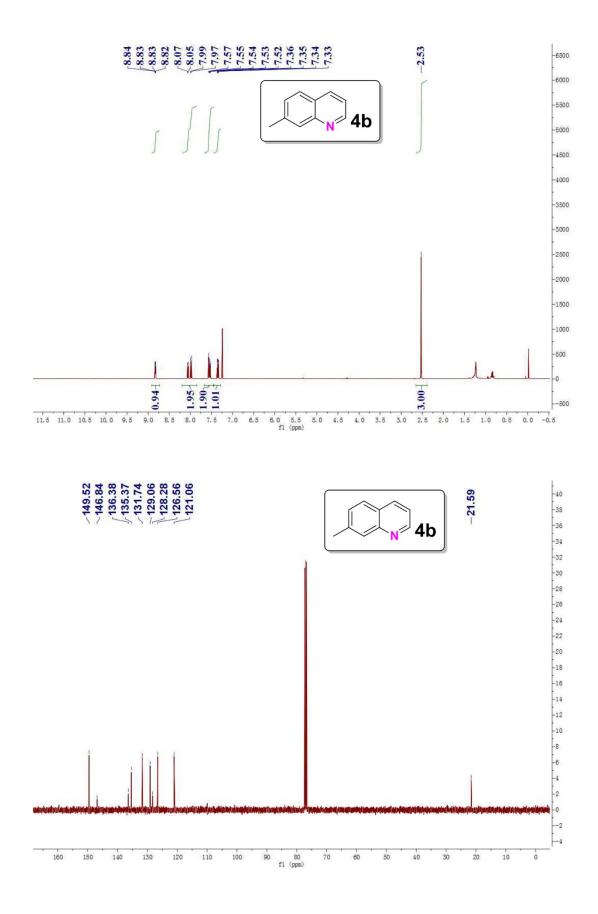


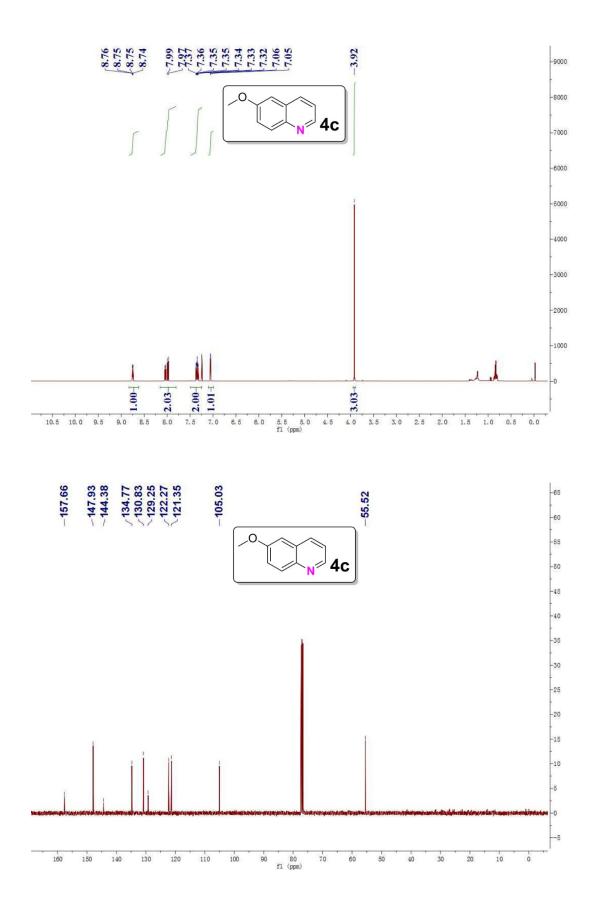




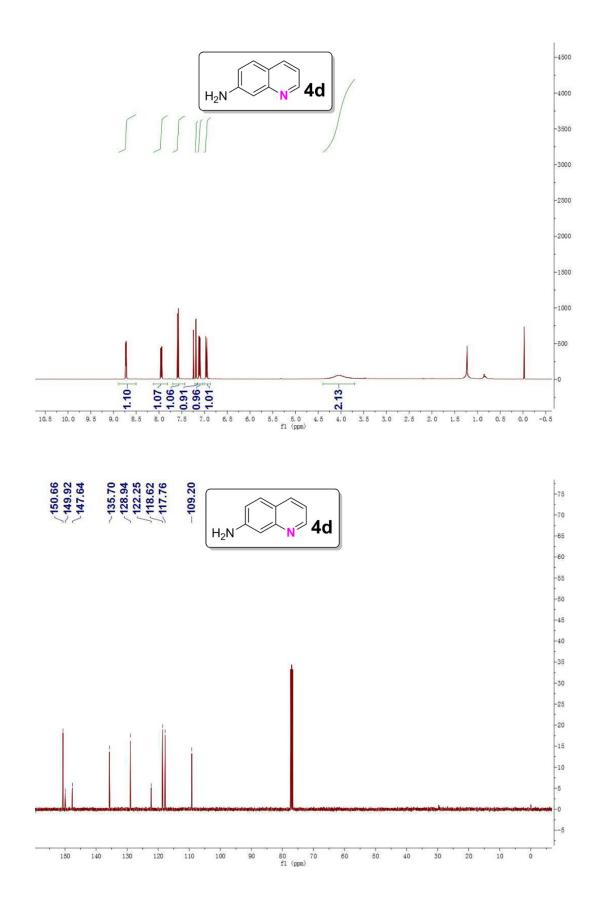


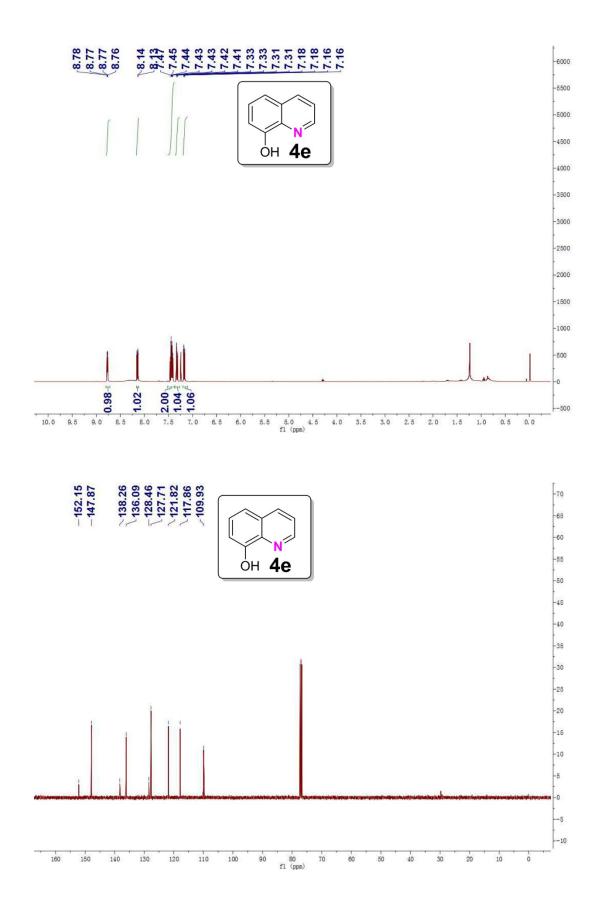


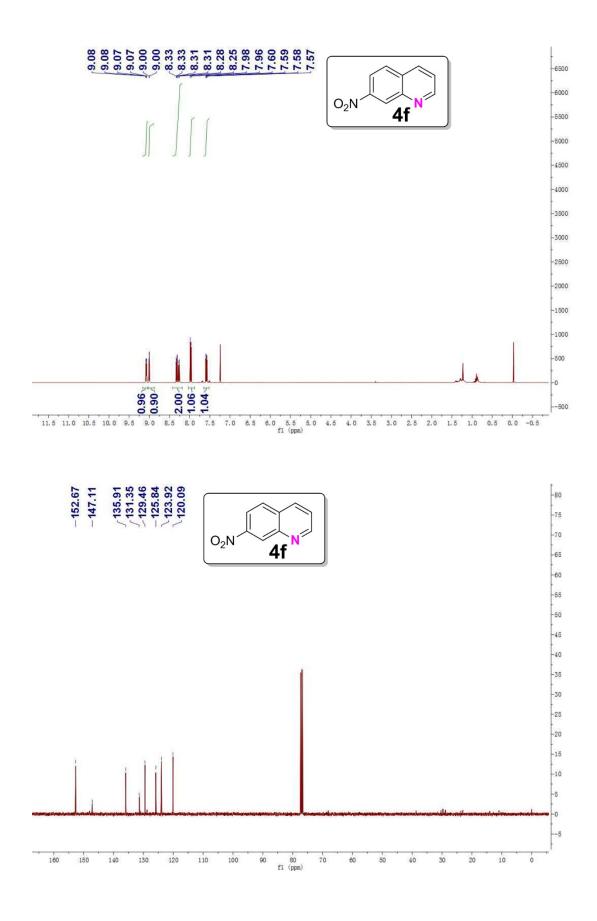


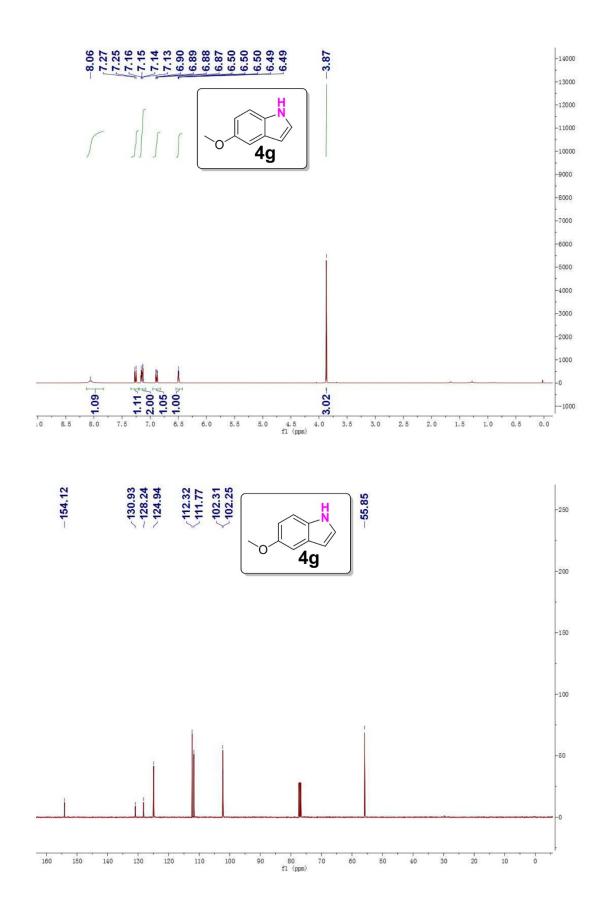


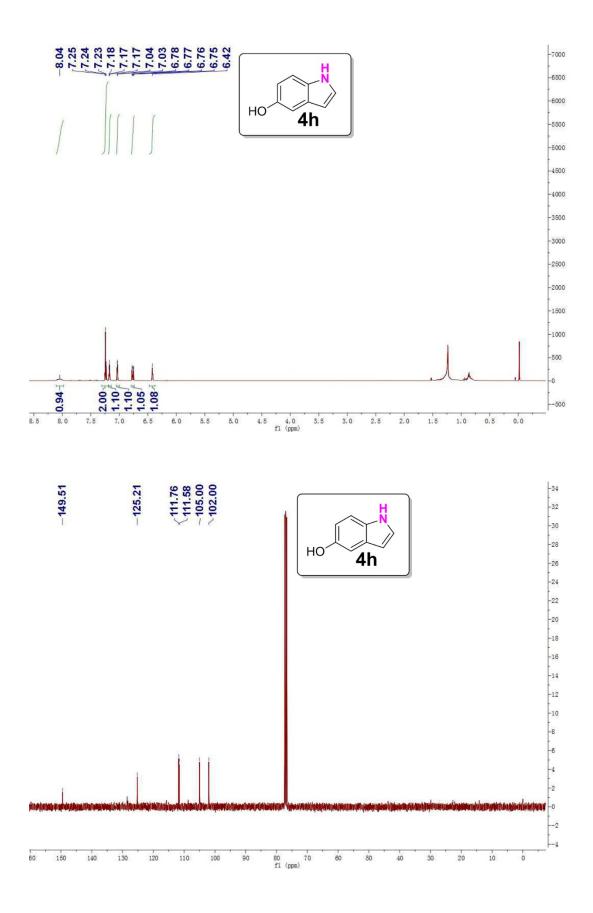
S31



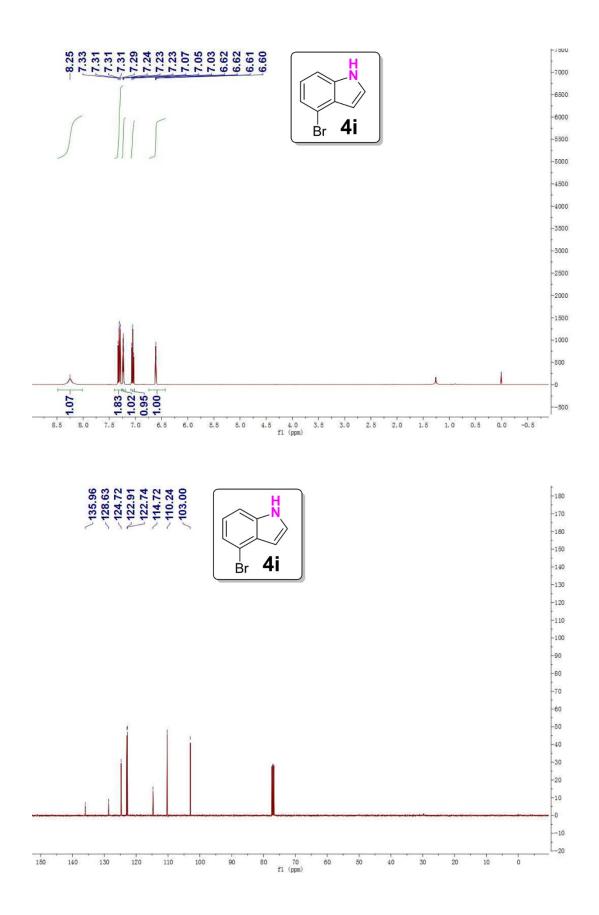


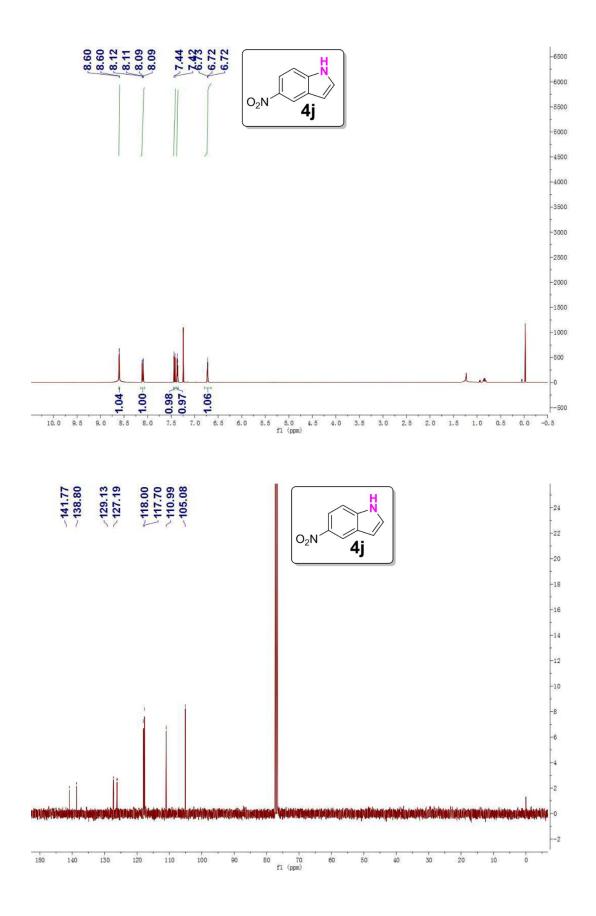




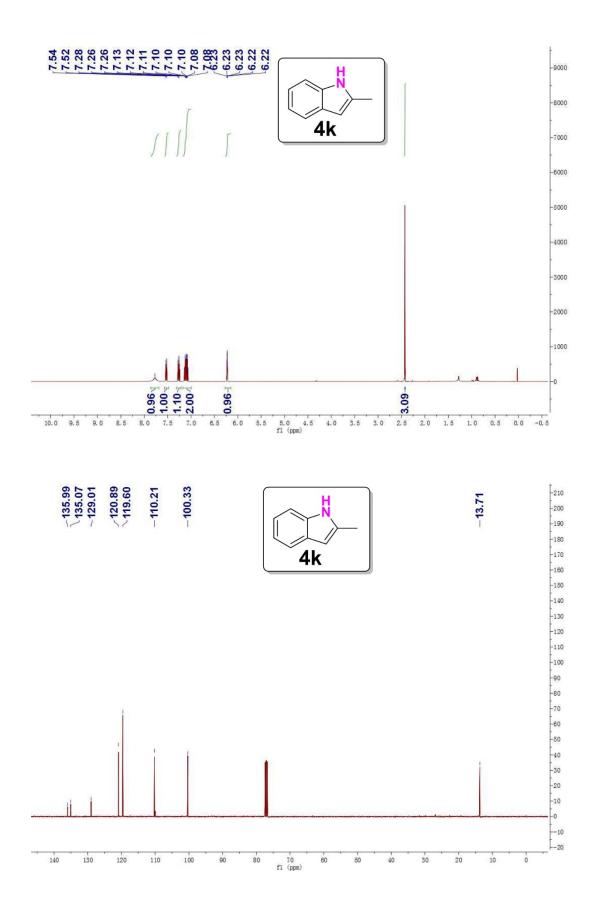


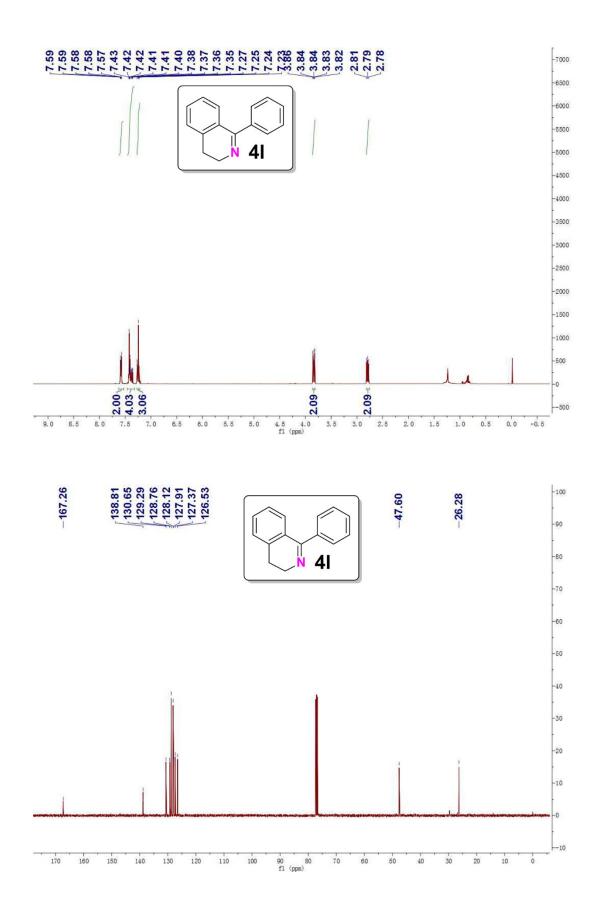
**S36** 

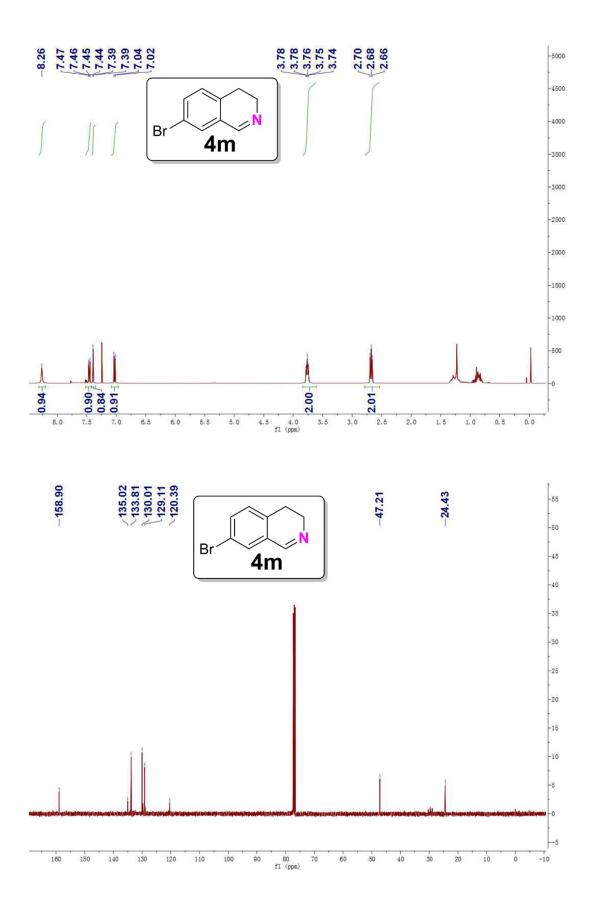




**S38** 







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