

Visible-Light-Promoted and One-Pot Synthesis of Phenanthridines and Quinolines from Aldehydes and O-Acyl Hydroxylamine

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Supporting Information

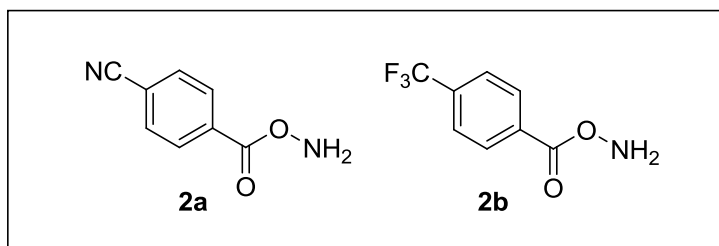
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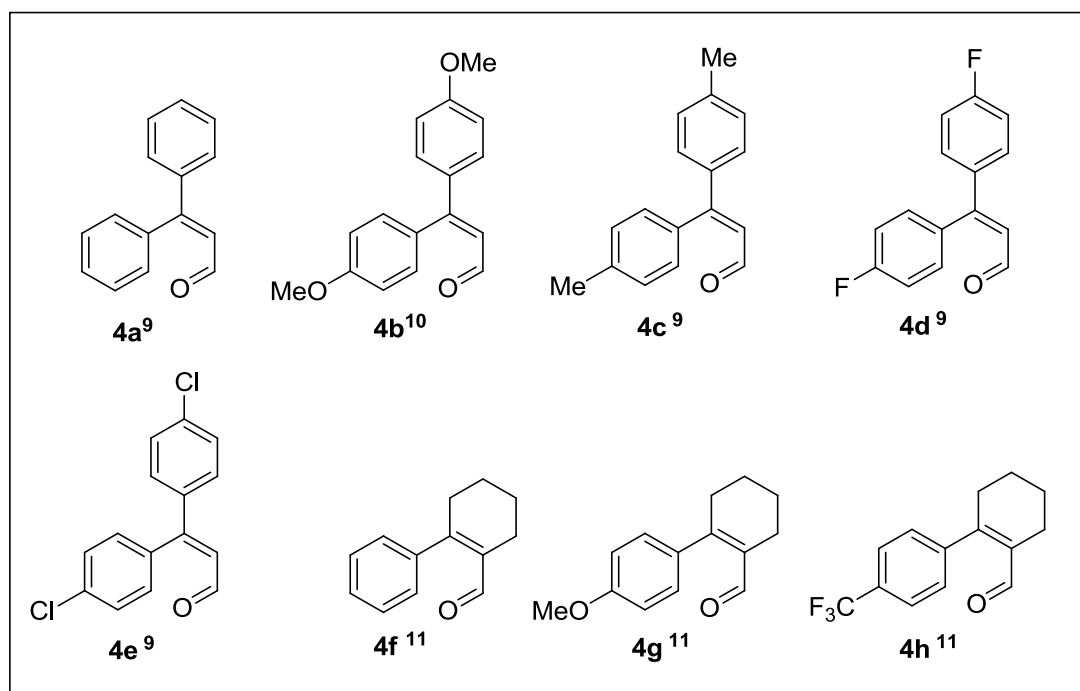
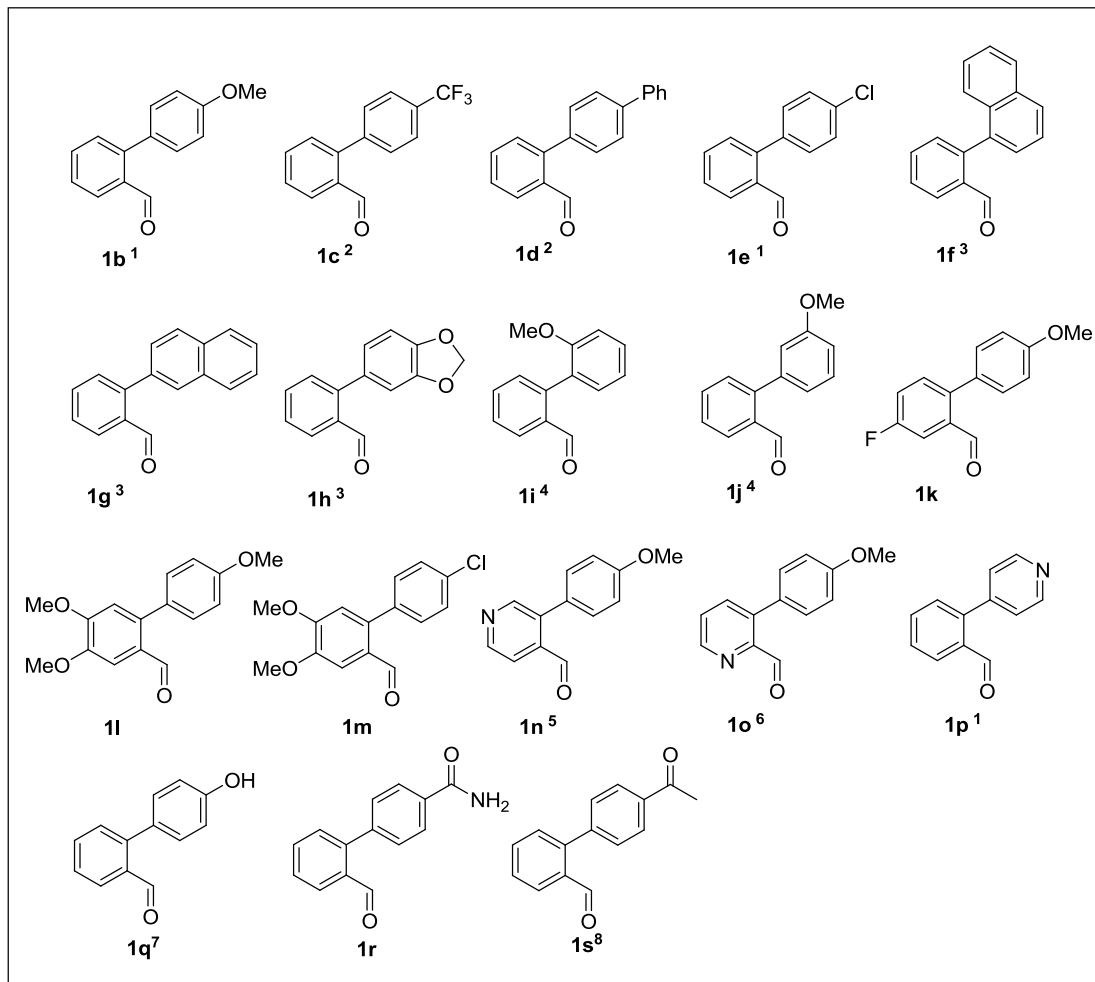
1. General methods.

The anhydrous DMF was purchased. Thin layer chromatography (TLC) was performed on EMD precoated plates (silica gel 60 F254, Art 5715) and visualized by fluorescence quenching under UV light and by staining with phosphomolybdic acid or potassium permanganate, respectively. Column chromatography was performed on EMD Silica Gel 60 (300 – 400 Mesh) using a forced flow of 0.5 – 1.0 bar. ^1H NMR (400 MHz), ^{13}C NMR (100MHz) and ^{19}F (376MHz) were measured on a Bruker AVANCE III-400 spectrometer. Chemical shifts are expressed in parts per million (ppm) with respect to the residual solvent peak. Coupling constants are reported as Hertz (Hz), signal shapes and splitting patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Infrared (IR) spectra were recorded on a Nicolet 6700 spectrophotometer and are reported as wavenumber (cm^{-1}).

2. List of the nitrogen sources

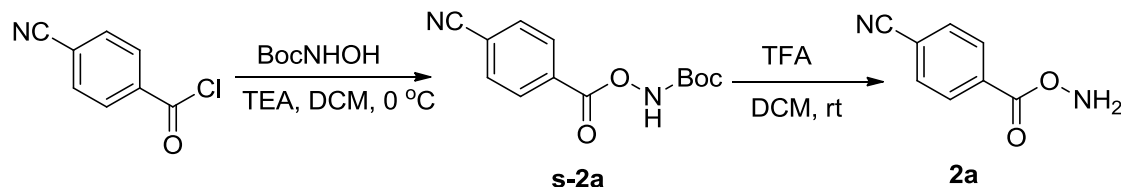


3. List of the substrates



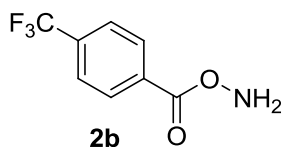
4. Synthesis of the nitrogen sources **2a** and **2b**

(1) Synthesis of the nitrogen sources **2a**



To a 100-mL round bottom flask equipped with a magnetic stir bar were charged with tert-Butyl N-hydroxycarbamate (804 mg, 6.04 mmol), Et₃N (840 μ L, 6.04 mmol) and CH₂Cl₂ (10mL). 4-Cyanobenzoyl chloride (500 mg, 3.02 mmol) was then added dropwise at 0 °C for 30 min and stirred at 0 °C for another 10 min. Then, the mixture was poured into saturated NaHCO₃ (25 mL) and extracted with 25 mL of CH₂Cl₂ twice. The combined organic phases were then dried over Na₂SO₄ and concentrated under vacuum. The residue was then dissolved in dry CH₂Cl₂ (2 mL). Trifluoroacetic acid (4 ml) was then added dropwise at room temperature and the reaction mixture was stirred at 0 °C for another 30 min. Then, the mixture was poured into saturated NaHCO₃ (25 mL) and extracted with 25 mL of CH₂Cl₂ twice. The combined organic phases were then dried over Na₂SO₄ and concentrated under vacuum. Further column chromatography on silica gel (EtOAc:hexane = 1:5) afforded 4-(aminooxycarbonyl)benzonitrile (**2a**, 450.3 mg, 92% for two steps) as a white solid. Mp 86-87 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.17 – 8.09 (m, 2H), 7.82 – 7.74 (m, 2H), 6.73 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 165.80, 132.43, 131.82, 129.92, 117.75, 116.96. IR (neat, cm⁻¹): 3309.93, 2231.91, 1727.11, 1591.43, 1269.54, 1075.20, 1017.75, 862.80, 760.71, 690.20.

(2) Synthesis of the nitrogen sources **2b**



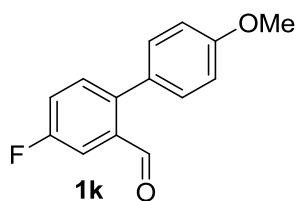
O-(4-(trifluoromethyl)benzoyl)hydroxylamine (2b): The synthesis of compound **2b** is similar to the method of **2a**. 88% for two steps as a white solid. Mp 49-50 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.10 (d, *J* = 7.8 Hz, 2H), 7.69 (d, *J* = 7.9 Hz, 2H), 6.99 (s, 2H). ¹⁹F NMR (376 MHz, CDCl₃) δ -63.27. ¹³C NMR (101 MHz, CDCl₃) δ 166.26, 134.85 (q, *J* = 33.3 Hz), 131.18, 129.74, 125.57 (q, *J* = 3.03 Hz), 123.43 (q, *J* = 273.7 Hz). IR (neat, cm⁻¹): 3290.00, 1729.49, 1558.37, 1411.56, 1324.87, 1270.99, 1064.19, 1015.02, 856.84, 766.89, 693.09.

5. Synthesis of substrates 1k-1m

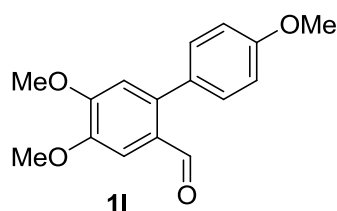
General Procedure:

A mixture of *p*-Br-substituted benzaldehyde (2 mmol), the corresponding substituted phenylboronic acid (2.2 mmol), Pd(PPh₃)₄ (0.02 mmol) and K₂CO₃ (4 mmol) was dissolved in a mixture of toluene/H₂O (5:1, 6 mL). The flask was evacuated and backfilled with N₂ for 3 times. The reaction was stirred at 90 °C for 3h. After the reaction was completed, the reaction mixture was diluted with ethyl acetate (20 mL). The EA layer was washed with saturated NH₄Cl solution (20 mL × 2) and saturated NaCl solution (20 mL × 1). The organic layer was dried over Na₂SO₄ and evaporated. The residue was purified by column chromatography on silica gel eluting to give the desired aldehyde.

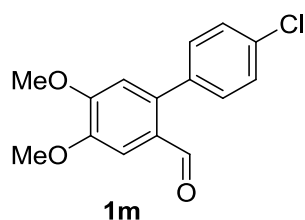
Data of compounds **1k-1m**:



4-fluoro-4'-methoxybiphenyl-2-carbaldehyde (1k); 90% yield, yellow solid; ^1H NMR (400 MHz, CDCl_3) δ 9.92 (d, $J = 3.3$ Hz, 1H), 7.66 (dd, $J = 8.9, 2.8$ Hz, 1H), 7.42 (dd, $J = 8.5, 5.2$ Hz, 1H), 7.32 (dt, $J = 7.8, 3.9$ Hz, 1H), 7.30 – 7.23 (m, 2H), 7.04 – 6.98 (m, 2H), 3.87 (s, 3H). ^{19}F NMR (376 MHz, CDCl_3) δ -113.84 (s, 1F). ^{13}C NMR (101 MHz, CDCl_3) δ 191.41 (d, $J = 1.0$ Hz), 163.21, 160.74, 159.81, 141.82 (d, $J = 3.0$ Hz), 135.19 (d, $J = 6.1$ Hz), 132.75 (d, $J = 7.1$ Hz), 131.32, 129.01, 127.72, 120.80 (d, $J = 23.2$ Hz), 114.06, 113.58 (d, $J = 22.2$ Hz), 55.41. IR (neat, cm^{-1}): 1677.41, 1601.79, 1482.74, 1247.23, 1183.11, 1037.27, 904.26, 829.09, 766.21. HRMS (ESI) ($[\text{M}+\text{H}]^+$) Calcd. for $\text{C}_{14}\text{H}_{12}\text{FO}_2$: 231.0821; found: 231.0815.

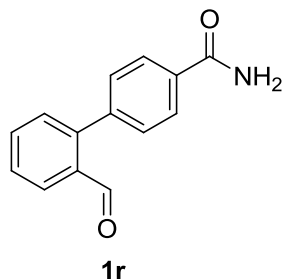


4,4',5-trimethoxybiphenyl-2-carbaldehyde (1l); 98% yield, yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 9.83 (s, 1H), 7.52 (s, 1H), 7.30 (d, $J = 8.5$ Hz, 2H), 6.99 (d, $J = 8.5$ Hz, 2H), 6.84 (s, 1H), 3.97 (s, 6H), 3.87 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 191.28, 159.60, 153.43, 148.56, 141.25, 131.33, 129.86, 126.96, 113.83, 112.63, 108.63, 56.19, 56.10, 55.40. IR (neat, cm^{-1}): 3002.99, 2978.40, 2863.32, 1667.10, 1598.72, 1502.26, 1349.75, 1187.85, 1135.91, 1031.63, 750.31, 604.11, 534.54. HRMS (ESI) ($[\text{M}+\text{Na}]^+$) Calcd. for $\text{C}_{16}\text{H}_{16}\text{O}_4\text{Na}$: 295.0946; found: 295.0941.



4'-chloro-4,5-dimethoxybiphenyl-2-carbaldehyde (1m); 93% yield, white solid. ^1H NMR (400 MHz, CDCl_3) δ 9.80 (s, 1H), 7.53 (s, 1H), 7.46 – 7.43 (m, 2H), 7.33 – 7.30 (m, 2H), 6.81 (s, 1H), 3.98 (d, $J = 1.9$ Hz, 7H). ^{13}C NMR (101 MHz, CDCl_3) δ 190.61, 153.51, 149.01, 140.01, 136.05, 134.35, 131.41, 128.58, 126.94, 112.47,

108.80, 56.27, 56.16. IR (neat, cm^{-1}): 1663.11, 1592.75, 1517.22, 1487.75, 1286.47, 1220.93, 1136.11, 1022.34, 830.57, 753.38, 717.68, 663.48. HRMS (ESI) ($[\text{M}+\text{Na}]^+$) Calcd. for $\text{C}_{15}\text{H}_{13}\text{ClNaO}_3$: 299.0451; found: 299.0444.

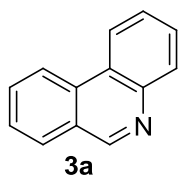


2'-formylbiphenyl-4-carboxamide (1r); 72% yield, white solid. ^1H NMR (400 MHz, CDCl_3) δ 9.96 (s, 1H), 8.05 (d, $J = 7.1$ Hz, 1H), 7.94 (d, $J = 8.1$ Hz, 2H), 7.67 (td, $J = 7.5, 1.1$ Hz, 1H), 7.55 (t, $J = 7.6$ Hz, 1H), 7.46 (dd, $J = 14.7, 7.9$ Hz, 3H), 6.21 (d, $J = 57.9$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 191.82, 168.87, 144.54, 141.62, 133.76, 133.66, 133.04, 130.67, 130.31, 128.44, 128.07, 127.51. IR (neat, cm^{-1}): 3168.46, 1701.30, 1640.67, 1610.08, 1408.82, 1190.52, 857.29, 754.05, 638.99, 615.54. HRMS (ESI) ($[\text{M}+\text{H}]^+$) Calcd. for $\text{C}_{16}\text{H}_{17}\text{O}_4$: 226.0868; found: 226.0865.

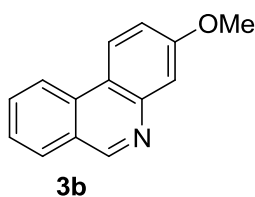
6. General Procedure: synthesis of phenanthridines and quinolines.

A 10-mL round bottom flask was equipped with a rubber septum and magnetic stir bar and was charged with **1** or **4** (0.2 mmol), **2a** (0.3mmol, 1.5 equiv.), *fac*-Ir(ppy)₃ (0.004 mmol, 0.02 equiv) and *p*-Cl-Benzenesulfonic acid (0.02mmol, 0.1 equiv). The flask was evacuated and backfilled with N_2 for 3 times. DMF (2.0 mL) was then added with syringe under N_2 . The mixture was then irradiated by white LED strips (for synthesis of phenanthridines) or blue LED strips (for synthesis of quinolines). After the reaction was complete (as judged by TLC analysis), the mixture was concentrated under vacuum to remove DMF. The residue was then purified by flash chromatography on silica gel to afford phenanthridines or quinolines.

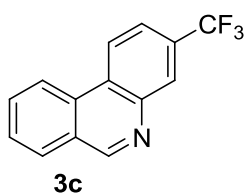
7. Data of compounds 3 and 5.



Phenanthridine (3a):¹² According to General Procedure (for 12h), **1a** (0.2 mmol, 36.4 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3a** (26.0 mg, 73%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1:10). ¹H NMR (400 MHz, CDCl₃) δ 9.28 (s, 1H), 8.58 (dd, *J* = 10.7, 8.5 Hz, 2H), 8.20 (d, *J* = 8.0 Hz, 1H), 8.03 (d, *J* = 7.9 Hz, 1H), 7.90 – 7.80 (m, 1H), 7.78 – 7.72 (m, 1H), 7.68 (td, *J* = 8.1, 0.9 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 153.57, 144.51, 132.57, 131.00, 130.18, 128.77, 128.69, 127.49, 127.09, 126.41, 124.12, 122.22, 121.88.

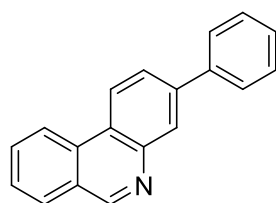


2-methoxyphenanthrene (3b):¹² According to General Procedure (for 18h), **1b** (0.2 mmol, 42.4 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3b** (36.7 mg, 88%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:6). ¹H NMR (400 MHz, CDCl₃) δ 9.23 (s, 1H), 8.45 (t, *J* = 6.3 Hz, 1H), 8.42 (d, *J* = 9.0 Hz, 1H), 7.98 (d, *J* = 7.9 Hz, 1H), 7.82 – 7.74 (m, 1H), 7.64 – 7.54 (m, 2H), 7.29 (dt, *J* = 8.4, 4.2 Hz, 1H), 3.97 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 160.07, 153.95, 146.06, 132.76, 131.08, 128.79, 126.39, 125.55, 123.39, 121.35, 118.14, 118.08, 109.93, 55.56.



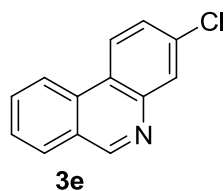
2-(trifluoromethyl)phenanthrene (3c):¹² According to General Procedure (for 12h), **1c** (0.2 mmol, 50.0 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid

(0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3c** (33.6 mg, 68%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:15). ¹H NMR (400 MHz, CDCl₃) δ 9.31 (s, 1H), 8.60 (dd, *J* = 15.3, 8.4 Hz, 2H), 8.45 (s, 1H), 8.06 (d, *J* = 7.9 Hz, 1H), 7.94 – 7.87 (m, 1H), 7.84 (dt, *J* = 10.9, 5.5 Hz, 1H), 7.77 (dt, *J* = 16.1, 4.5 Hz, 1H). ¹⁹F NMR (376 MHz, CDCl₃) δ -62.23 (s, 3F). ¹³C NMR (101 MHz, CDCl₃) δ 154.88, 143.73, 131.68, 131.58, 130.46 (q, *J* = 33.3 Hz), 128.95, 128.69, 127.63 (q, *J* = 4.0 Hz), 126.86, 126.40, 124.08 (q, *J* = 273.7 Hz), 123.26, 122.94 (q, *J* = 3.0 Hz), 122.20.

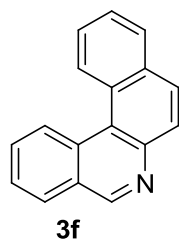


3d

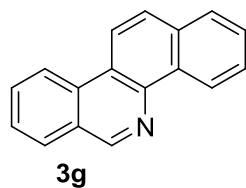
2-phenylphenanthrene (3d): According to General Procedure (for 12h), **1d** (0.2 mmol, 51.7 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3d** (41.8 mg, 82%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1:3). Mp 140-141 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.27 (s, 1H), 8.54 (dd, *J* = 8.4, 3.5 Hz, 2H), 8.42 (d, *J* = 1.8 Hz, 1H), 8.00 (d, *J* = 7.9 Hz, 1H), 7.89 (dd, *J* = 8.5, 1.9 Hz, 1H), 7.81 (dd, *J* = 11.8, 4.7 Hz, 1H), 7.80 – 7.74 (m, 2H), 7.65 (dd, *J* = 11.1, 3.9 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.4 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 154.02, 144.83, 141.39, 140.22, 132.39, 131.13, 129.04, 128.83, 127.97, 127.80, 127.48, 127.39, 126.37, 126.22, 123.14, 122.77, 121.90. IR (neat, cm⁻¹): 2908.13, 1613.10, 1568.18, 1478.08, 1276.76, 1053.57, 758.73, 680.38, 654.81, 580.81. HRMS (ESI) ([M+H]⁺) Calcd. for C₁₉H₁₅N: 256.1126; found: 256.1124.



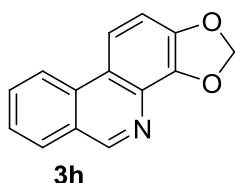
3-chlorophenanthridine (3e):¹² According to General Procedure (for 12h), **1e** (0.2 mmol, 43.3 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3e** (41.8 mg, 82%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:25). ¹H NMR (400 MHz, CDCl₃) δ 9.22 (s, 1H), 8.46 (d, *J* = 8.3 Hz, 1H), 8.39 (d, *J* = 8.8 Hz, 1H), 8.13 (t, *J* = 6.4 Hz, 1H), 7.99 (d, *J* = 7.9 Hz, 1H), 7.83 (ddd, *J* = 8.3, 7.2, 1.3 Hz, 1H), 7.73 – 7.64 (m, 1H), 7.57 (dt, *J* = 6.5, 3.3 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 154.65, 145.07, 134.26, 132.04, 131.40, 129.30, 128.87, 127.78, 127.58, 126.22, 123.53, 122.55, 121.73.



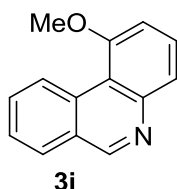
benzo[a]phenanthridine (3f):¹³ According to General Procedure (for 12h), **1f** (0.2 mmol, 46.5 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3f** (31.8 mg, 69%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:15). ¹H NMR (400 MHz, CDCl₃) δ 9.35 (s, 1H), 9.11 – 9.02 (m, 2H), 8.16 – 8.07 (m, 2H), 8.06 – 7.98 (m, 2H), 7.89 – 7.82 (m, 1H), 7.72 – 7.67 (m, 2H), 7.66 – 7.59 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 152.88, 144.33, 133.29, 132.65, 130.76, 129.80, 129.61, 128.81, 128.77, 128.41, 127.69, 127.56, 126.76, 126.64, 126.50, 126.43, 120.71.



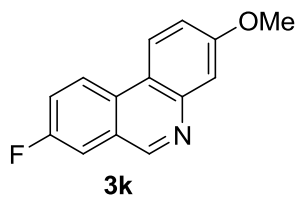
benzo[*c*]phenanthridine (3g):¹² According to General Procedure (for 12h), **1g** (0.2 mmol, 46.5 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3g** (37.6 mg, 82%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1:15). ¹H NMR (400 MHz, CDCl₃) δ 9.41 (d, *J* = 8.1 Hz, 1H), 9.38 (d, *J* = 8.3 Hz, 1H), 8.57 (d, *J* = 8.4 Hz, 1H), 8.45 (d, *J* = 9.0 Hz, 1H), 8.06 (d, *J* = 7.9 Hz, 1H), 7.94 (t, *J* = 7.7 Hz, 2H), 7.80 (ddd, *J* = 8.3, 7.1, 1.2 Hz, 1H), 7.78 – 7.72 (m, 1H), 7.69 – 7.62 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 151.97, 141.47, 133.28, 132.81, 132.07, 130.79, 128.68, 127.84, 127.71, 127.39, 127.13, 127.02, 126.88, 124.78, 122.19, 121.07, 119.92.



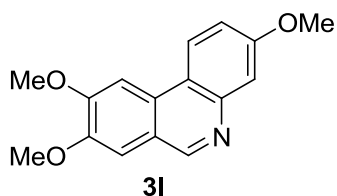
[1,3]dioxolo[4,5-*c*]phenanthridine (3h):¹² According to General Procedure, **1h** (0.2 mmol, 45.3 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3h** (32.8 mg, 71%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1:15). ¹H NMR (400 MHz, CDCl₃) δ 9.13 (s, 1H), 8.35 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 7.9 Hz, 1H), 7.82 (s, 1H), 7.77 (t, *J* = 7.6 Hz, 1H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.52 (s, 1H), 6.13 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 151.26, 149.24, 148.16, 141.76, 132.52, 130.58, 128.62, 126.56, 125.68, 121.58, 120.06, 107.73, 101.84, 99.41.



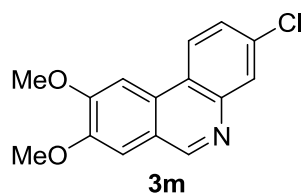
1-methoxyphenanthridine (3i): According to General Procedure (for 24h), **1i** (0.2 mmol, 42.4 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3i** (16.5 mg, 39%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:15). ¹H NMR (400 MHz, CDCl₃)



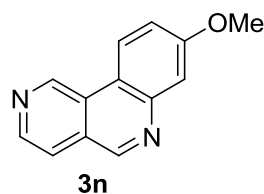
8-fluoro-3-methoxyphenanthridine (3k): According to General Procedure (for 12h), **1k** (0.2 mmol, 46.0 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3k** (35.9 mg, 79%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1: 8). Mp 155-156°C. ¹H NMR (400 MHz, CDCl₃) δ 9.07 (s, 1H), 8.34 (dd, *J* = 9.1, 5.0 Hz, 1H), 8.25 (d, *J* = 9.0 Hz, 1H), 7.54 – 7.40 (m, 3H), 7.21 (dd, *J* = 9.0, 2.7 Hz, 1H), 3.89 (s, 3H). ¹⁹F NMR (376 MHz, CDCl₃) δ -113.98 (s, 1F). ¹³C NMR (101 MHz, CDCl₃) δ 160.94, 158.89, 158.48, 151.78 (d, *J* = 3.0 Hz), 144.63, 128.39 (d, *J* = 1.0 Hz),, 122.89 (d, *J* = 8.1 Hz), 122.04, 119.33 (d, *J* = 24.2 Hz), 117.53, 111.61, 111.41, 108.91, 54.52. IR (neat, cm⁻¹): 2967.35, 1598.21, 1261.31, 1134.26, 1036.92, 847.16, 804.24, 652.69. HRMS (ESI) ([M+H]⁺) Calcd. for C₁₄H₁₁FNO: 228.0825; found: 228.0820.



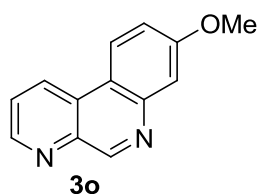
3,8,9-trimethoxyphenanthridine (3l):¹⁴ According to General Procedure (for 12h), **1l** (0.2 mmol, 54.5 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3l** (44.4 mg, 82%) as a white solid after purification on SiO₂ (DCM : MeOH = 40:1). ¹H NMR (400 MHz, CDCl₃) δ 8.97 (s, 1H), 8.18 (d, *J* = 9.1 Hz, 1H), 7.64 – 7.58 (m, 1H), 7.43 (dd, *J* = 7.4, 3.0 Hz, 1H), 7.16 (dd, *J* = 9.1, 2.5 Hz, 2H), 4.00 (s, 3H), 3.94 (s, 3H), 3.88 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 158.36, 151.99, 150.92, 148.14, 144.38, 127.49, 121.83, 119.76, 116.91, 116.84, 108.48, 106.58, 100.15, 55.07, 55.00, 54.46.



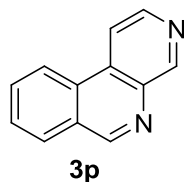
3-chloro-8,9-dimethoxyphenanthridine (3m): According to General Procedure (for 12h), **1m** (0.2 mmol, 43.3 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3m** (48.6 mg, 89%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1:25). ¹H NMR (400 MHz, CDCl₃) δ 9.12 (s, 1H), 8.31 (d, *J* = 8.8 Hz, 1H), 8.11 (d, *J* = 2.1 Hz, 1H), 7.76 (d, *J* = 5.3 Hz, 1H), 7.55 (dt, *J* = 14.0, 7.0 Hz, 1H), 7.32 (s, 1H), 4.13 (s, 3H), 4.07 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 153.21, 152.80, 150.16, 144.62, 133.30, 129.23, 127.80, 127.10, 123.09, 122.28, 121.73, 107.78, 101.62, 56.23, 56.16. IR (neat, cm⁻¹): 2916.24, 2848.44, 1498.33, 1262.74, 1156.24, 1016.57, 860.83, 803.79, 613.59, 582.26. HRMS (ESI) ([M+H]⁺) Calcd. for C₁₅H₁₃ClNO₂: 274.0635; found: 274.0632.



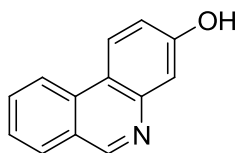
8-methoxybenzo[c][2,6]naphthyridine (3n): According to General Procedure (for 12h), **1n** (0.2 mmol, 42.6 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3n** (27.6 mg, 66%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:1.5). Mp 152-153°C. ¹H NMR (400 MHz, CDCl₃) δ 9.96 (s, 1H), 9.28 (d, *J* = 4.3 Hz, 1H), 8.81 (d, *J* = 5.2 Hz, 1H), 8.56 (t, *J* = 6.5 Hz, 1H), 7.81 (d, *J* = 5.3 Hz, 1H), 7.62 (t, *J* = 3.3 Hz, 1H), 7.39 (dt, *J* = 6.7, 3.3 Hz, 1H), 4.00 (d, *J* = 4.0 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 160.71, 152.51, 146.82, 146.36, 145.15, 127.97, 126.69, 122.75, 120.24, 119.47, 116.16, 110.46, 55.63. IR (neat, cm⁻¹): 3002.90, 2863.46, 1667.25, 1598.84, 1502.27, 1135.75, 834.45, 750.08, 669.20, 581.94. HRMS (ESI) ([M+H]⁺) Calcd. for C₁₃H₁₁N₂O: 211.0871; found: 211.0866.



8-methoxybenzo[f][1,7]naphthyridine (3o): According to General Procedure (for 12h), **1o** (0.2 mmol, 42.6 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3o** (20.4 mg, 51%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1:4). Mp 112-114°C. ¹H NMR (400 MHz, CDCl₃) δ 9.50 (s, 1H), 8.99 (dd, *J* = 4.3, 1.5 Hz, 1H), 8.79 (d, *J* = 8.4 Hz, 1H), 8.39 (d, *J* = 9.0 Hz, 1H), 7.72 (td, *J* = 8.8, 4.1 Hz, 1H), 7.64 (d, *J* = 2.6 Hz, 1H), 7.36 (dd, *J* = 9.0, 2.7 Hz, 1H), 4.00 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 160.71, 155.24, 149.96, 146.01, 141.22, 129.63, 128.21, 125.33, 123.33, 118.83, 117.05, 110.21, 55.64. IR (neat, cm⁻¹): 2863.52, 1667.42, 1599.00, 1502.42, 1135.89, 834.36, 750.14, 669.52, 635.64, 578.86. HRMS (ESI) ([M+H]⁺) Calcd. for C₁₃H₁₁N₂O: 211.0871; found: 211.0867.

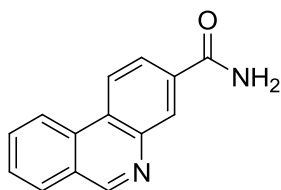


Benzo[c][1,7]naphthyridine (3p):¹⁵ According to General Procedure (for 44h), **1p** (0.2 mmol, 36.6 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3p** (19.9 mg, 55%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1:1.5). ¹H NMR (400 MHz, CDCl₃) δ 9.55 (d, *J* = 18.0 Hz, 1H), 9.35 (s, 1H), 8.80 (d, *J* = 5.5 Hz, 1H), 8.60 (d, *J* = 8.2 Hz, 1H), 8.32 (d, *J* = 5.6 Hz, 1H), 8.13 (t, *J* = 10.1 Hz, 1H), 7.98 – 7.90 (m, 1H), 7.86 (t, *J* = 7.2 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 155.10, 153.39, 145.63, 139.53, 131.69, 130.58, 129.80, 129.15, 128.99, 127.77, 122.52, 115.47.



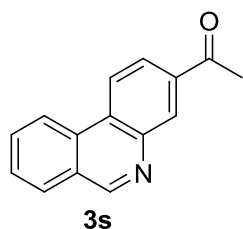
3q

Phenanthridin-3-ol (3q):¹⁶ According to General Procedure (for 12h), **1q** (0.2 mmol, 39.6 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3q** (26.1 mg, 67%) as a white solid after purification on SiO₂ (DCM : MeOH = 20:1). ¹H NMR (400 MHz, DMSO) δ 10.11 (s, 1H), 9.27 (s, 1H), 8.65 (dd, *J* = 20.5, 8.6 Hz, 2H), 8.15 (d, *J* = 7.9 Hz, 1H), 7.88 (t, *J* = 7.5 Hz, 1H), 7.68 (t, *J* = 7.4 Hz, 1H), 7.42 (d, *J* = 2.1 Hz, 1H), 7.25 (dd, *J* = 8.8, 2.2 Hz, 1H). ¹³C NMR (101 MHz, DMSO) δ 158.05, 153.69, 145.81, 132.18, 131.25, 128.76, 126.28, 124.86, 124.11, 121.42, 117.77, 116.38, 112.54.

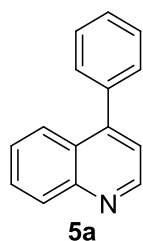


3r

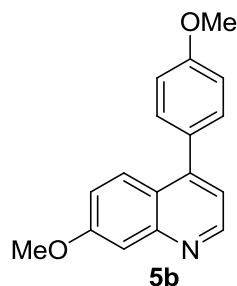
Phenanthridine-3-carboxamide (3r): According to General Procedure (for 12h), **1r** (0.2 mmol, 45.0 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3r** (25.5 mg, 57%) as a white solid after purification on SiO₂ (DCM : MeOH = 20:1). ¹H NMR (400 MHz, DMSO) δ 9.46 (s, 1H), 8.90 (dd, *J* = 8.4, 4.5 Hz, 2H), 8.68 (d, *J* = 1.7 Hz, 1H), 8.34 (s, 1H), 8.29 (d, *J* = 7.7 Hz, 1H), 8.23 (dd, *J* = 8.5, 1.8 Hz, 1H), 8.06 – 7.97 (m, 1H), 7.87 (dd, *J* = 14.3, 6.6 Hz, 1H), 7.62 (s, 1H). ¹³C NMR (101 MHz, DMSO) δ 167.38, 154.32, 143.50, 134.29, 131.67, 131.28, 128.93, 128.88, 128.63, 126.36, 125.84, 125.60, 123.08, 122.71. IR (neat, cm⁻¹): 2531.50, 2324.37, 1637.89, 1528.78, 1399.28, 1245.15, 1141.76, 960.39, 843.23, 743.45, 672.16, 629.84. HRMS (ESI) ([M+H]⁺) Calcd. for C₁₄H₁₁N₂O: 223.0871; found: 223.0869.



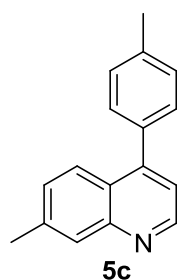
1-(phenanthridin-3-yl)ethanone (3s): According to General Procedure (for 12h), **1s** (0.2 mmol, 44.9 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **3r** (29.6 mg, 67%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1:5). ¹H NMR (400 MHz, CDCl₃) δ 9.32 (s, 1H), 8.72 (d, *J* = 1.6 Hz, 1H), 8.66 – 8.55 (m, 2H), 8.24 (dd, *J* = 8.6, 1.7 Hz, 1H), 8.08 (t, *J* = 8.8 Hz, 1H), 7.90 (td, *J* = 8.4, 4.3 Hz, 1H), 7.84 – 7.72 (m, 1H), 2.77 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 197.78, 154.48, 143.91, 136.82, 131.83, 131.51, 131.48, 128.91, 128.74, 127.54, 127.00, 125.36, 122.76, 122.46, 26.78. IR (neat, cm⁻¹): 1674.42, 1608.10, 1571.19, 1444.55, 1350.63, 1268.05, 1221.76, 1158.64, 927.59, 895.50, 825.79, 765.05, 654.50, 592.91. HRMS (ESI) ([M+H]⁺) Calcd. for C₁₅H₁₂NO: 222.0919; found: 222.0919.



4-phenylquinoline (5a):¹² According to General Procedure (for 12h), **4a** (0.2 mmol, 41.7 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **5a** (24.0 mg, 59%) as a white solid after purification on SiO₂ (EtOAc : hexane = 1:15). ¹H NMR (400 MHz, CDCl₃) δ 8.95 (d, *J* = 3.3 Hz, 1H), 8.19 (d, *J* = 8.4 Hz, 1H), 7.92 (dd, *J* = 8.5, 0.9 Hz, 1H), 7.73 (qd, *J* = 6.9, 3.4 Hz, 1H), 7.57 – 7.44 (m, 6H), 7.37 – 7.31 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 149.94, 148.64, 148.57, 137.99, 129.80, 129.56, 129.37, 128.60, 128.46, 126.79, 126.67, 125.90, 121.36.



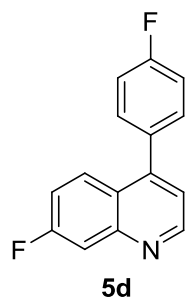
7-methoxy-4-(4-methoxyphenyl)quinoline (5b): According to General Procedure (for 16h), **4b** (0.2 mmol, 53.7 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **5b** (27.8 mg, 52%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:3). ¹H NMR (400 MHz, CDCl₃) δ 8.82 (d, *J* = 4.5 Hz, 1H), 7.85 (d, *J* = 9.3 Hz, 1H), 7.49 (d, *J* = 2.5 Hz, 1H), 7.43 (d, *J* = 8.7 Hz, 2H), 7.22 – 7.11 (m, 2H), 7.05 (d, *J* = 8.7 Hz, 2H), 3.97 (s, 3H), 3.89 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 160.44, 159.85, 150.49, 150.16, 148.15, 130.75, 130.45, 127.08, 122.02, 119.56, 119.42, 114.05, 107.58, 55.54, 55.41. IR (neat, cm⁻¹): 2998.61, 2834.93, 1609.86, 1506.15, 1431.74, 1287.57, 1224.56, 1026.72, 819.57, 812.04, 662.08.564.94. HRMS (ESI) ([M+H]⁺) Calcd. for C₁₇H₁₆NO₂: 266.1181; found: 266.1179.



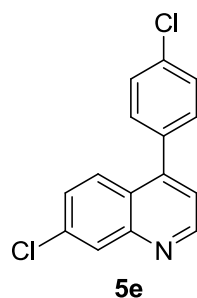
7-methyl-4-p-tolylquinoline (5c): According to General Procedure (for 16h), **4c** (0.2 mmol, 47.3 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **5c** (28.6 mg, 61%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:15). ¹H NMR (400 MHz, CDCl₃) δ 8.87 (d, *J* = 4.4 Hz, 1H), 7.95 (s, 1H), 7.83 (d, *J* = 8.6 Hz, 1H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 7.8 Hz, 3H), 7.24 (t, *J* = 9.3 Hz, 1H), 2.56 (s, 3H), 2.46 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 149.90, 148.86, 148.38, 139.58, 138.30, 135.23, 129.44, 129.27, 128.80, 128.71, 125.63, 124.88, 120.58, 21.72, 21.31. IR (neat, cm⁻¹):

2920.57, 1623.36, 1585.26, 1504.36, 1423.24, 885.24, 824.60, 782.80, 677.72, 659.00.

HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{17}H_{16}N$: 234.1283; found: 234.1283.

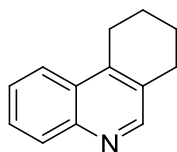


7-fluoro-4-(4-fluorophenyl)quinoline (5d): According to General Procedure (for 16h), **4d** (0.2 mmol, 48.8 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **5d** (26.8 mg, 56%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:15). Mp 101-102°C. ¹H NMR (400 MHz, CDCl₃) δ 8.93 (d, *J* = 4.4 Hz, 1H), 7.87 (dd, *J* = 9.3, 6.1 Hz, 1H), 7.80 (dd, *J* = 10.0, 2.6 Hz, 1H), 7.50 – 7.42 (m, 2H), 7.35 – 7.19 (m, 4H). ¹⁹F NMR (376 MHz, CDCl₃) δ -109.90 (s), -112.80 (s). ¹³C NMR (101 MHz, CDCl₃) δ 164.21 (d, *J* = 7.1Hz), 161.72 (d, *J* = 14.1Hz), 151.03, 149.79 (d, *J* = 12.1Hz), 147.63 (d, *J* = 1.0Hz), 133.67 (d, *J* = 3.0Hz), 131.19 (d, *J* = 8.1Hz, 2C), 127.95 (d, *J* = 10.1Hz), 123.82, 120.78 (d, *J* = 2.0Hz), 117.21 (d, *J* = 25.3Hz), 115.82 (d, *J* = 21.2Hz, 2C), 113.41 (d, *J* = 20.2Hz). IR (neat, cm⁻¹): 2924.84, 1619.49, 1501.75, 1425.86, 1215.05, 1159.96, 886.93, 830.61, 776.23, 645.50, 580.38, 550.71. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{15}H_{10}F_2N$: 242.0781; found: 242.0776.



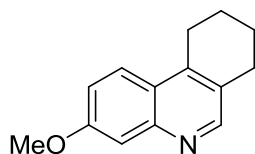
7-chloro-4-(4-chlorophenyl)quinoline (5e):¹² According to General Procedure (for 16h), **4e** (0.2 mmol, 55.4 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid

(0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **5e** (34.6 mg, 63%) as a yellow solid after purification on SiO₂ (EtOAc : hexane = 1:15). ¹H NMR (400 MHz, CDCl₃) δ 8.94 (d, *J* = 4.4 Hz, 1H), 8.17 (d, *J* = 2.1 Hz, 1H), 7.80 (d, *J* = 9.0 Hz, 1H), 7.55 – 7.50 (m, 2H), 7.46 (dt, *J* = 5.3, 2.7 Hz, 1H), 7.44 – 7.38 (m, 2H), 7.32 (dd, *J* = 10.7, 3.1 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 151.00, 149.11, 147.30, 135.87, 135.48, 135.01, 130.75, 129.03, 128.87, 127.87, 126.95, 124.97, 121.40.



5f

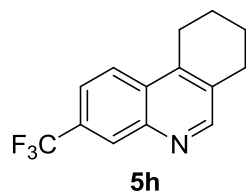
7,8,9,10-tetrahydrophenanthridine (5f):¹⁷ According to General Procedure (for 12h), **4f** (0.2 mmol, 37.2 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **5f** (20.5 mg, 56%) as a yellow oil after purification on SiO₂ (EtOAc : hexane = 1: 5). ¹H NMR (400 MHz, CDCl₃) δ 8.62 (s, 1H), 8.06 (d, *J* = 8.3 Hz, 1H), 7.92 (d, *J* = 8.3 Hz, 1H), 7.63 (t, *J* = 7.5 Hz, 1H), 7.53 (t, *J* = 7.5 Hz, 1H), 3.11 (t, *J* = 6.1 Hz, 2H), 2.90 (t, *J* = 5.9 Hz, 2H), 2.03 – 1.93 (m, 2H), 1.93 – 1.83 (m, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 152.38, 146.23, 141.43, 129.75, 129.70, 128.01, 127.62, 126.31, 122.59, 27.17, 24.93, 22.45, 22.34.



5g

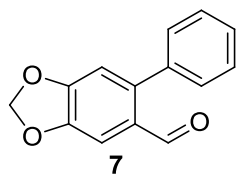
3-methoxy-7,8,9,10-tetrahydrophenanthridine (5g):¹⁸ According to General Procedure (for 12h), **4g** (0.2 mmol, 43.3 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **5g** (19.1 mg, 45%) as a yellow oil after purification on SiO₂ (EtOAc : hexane = 1: 5). ¹H NMR (400 MHz, CDCl₃) δ 8.54 (s, 1H), 7.80 (d, *J* = 9.2 Hz, 1H), 7.40 (d,

$J = 2.4$ Hz, 1H), 7.18 (dd, $J = 9.2, 2.5$ Hz, 1H), 3.94 (s, 3H), 3.07 (t, $J = 6.2$ Hz, 2H), 2.86 (t, $J = 6.0$ Hz, 2H), 1.94 (td, $J = 11.4, 5.8$ Hz, 2H), 1.91 – 1.81 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 159.51, 152.50, 147.86, 141.53, 127.69, 123.75, 122.60, 118.93, 107.88, 55.43, 26.91, 24.99, 22.51, 22.45.



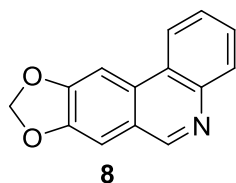
3-(trifluoromethyl)-7,8,9,10-tetrahydrophenanthridine (5h): According to General Procedure (for 12h), **4h** (0.2 mmol, 50.8 mg), **2a** (0.3 mmol, 48.6 mg), *p*-Cl-Benzenesulfonic acid (0.02mmol, 3.9 mg) and *fac*-Ir(ppy)₃ (0.004 mmol, 2.6 mg) afforded **5h** (27.3 mg, 54%) as a yellow oil after purification on SiO_2 (EtOAc : hexane = 1: 15). Mp 99-100°C. ^1H NMR (400 MHz, CDCl_3) δ 8.70 (s, 1H), 8.34 (s, 1H), 8.02 (d, $J = 8.8$ Hz, 1H), 7.69 (dd, $J = 8.8, 1.5$ Hz, 1H), 3.11 (t, $J = 6.3$ Hz, 2H), 2.92 (t, $J = 6.1$ Hz, 2H), 2.05 – 1.95 (m, 2H), 1.95 – 1.84 (m, 2H). ^{19}F NMR (376 MHz, CDCl_3) δ -62.46 (s). ^{13}C NMR (101 MHz, CDCl_3) δ 153.87, 145.42, 141.34, 131.78, 129.78 (d, $J = 33.3\text{Hz}$), 129.28, 127.66 (d, $J = 5.1\text{Hz}$), 124.13 (d, $J = 272.7\text{Hz}$), 123.87, 121.91 (d, $J = 3.0\text{Hz}$), 27.21, 24.91, 22.22, 22.08. IR (neat, cm^{-1}): 2941.82, 1739.05, 1513.17, 1461.39, 1322.14, 1267.38, 1157.08, 1111.41, 1064.84, 899.99, 808.45, 775.67, 732.66, 679.93. HRMS (ESI) ($[\text{M}+\text{H}]^+$) Calcd. for $\text{C}_{14}\text{H}_{11}\text{F}_3\text{N}$: 252.1000; found: 252.0995.

8. Synthesis of Alkaloid Trisphaeridine 8



6-phenylbenzo[d][1,3]dioxole-5-carbaldehyde:¹⁹ A mixture of 6-bromobenzo[d][1,3]dioxole-5-carbaldehyde (2.29 g, 10.0 mmol), phenylboronic acid (1.46 g, 12

mmol), Pd(PPh₃)₄ (116 mg, 0.1 mmol) and K₂CO₃ (2.1 g, 15 mmol) was dissolved in a mixture of toluene/H₂O (5:1, v/v). The flask was evacuated and backfilled with N₂ for 3 times. The reaction was stirred at 90 °C for 3 h. After the reaction was completed, the reaction mixture was diluted with ethyl acetate (20 mL). The organic layer was washed with saturated NH₄Cl solution (20 mL × 2) and saturated brine (20 mL × 1). The organic layer was dried over Na₂SO₄ and evaporated. The residue was purified by column chromatography on silica gel eluting (DCM:EA = 2:1) to give desired compound **7** as white solid (2.23g, 99%). ¹H NMR (300 MHz, CDCl₃) δ 9.77 (s, 1H), 7.52 – 7.40 (m, 4H), 7.36 (dd, J = 7.0, 2.3 Hz, 2H), 6.87 (s, 1H), 6.12 (s, 2H).

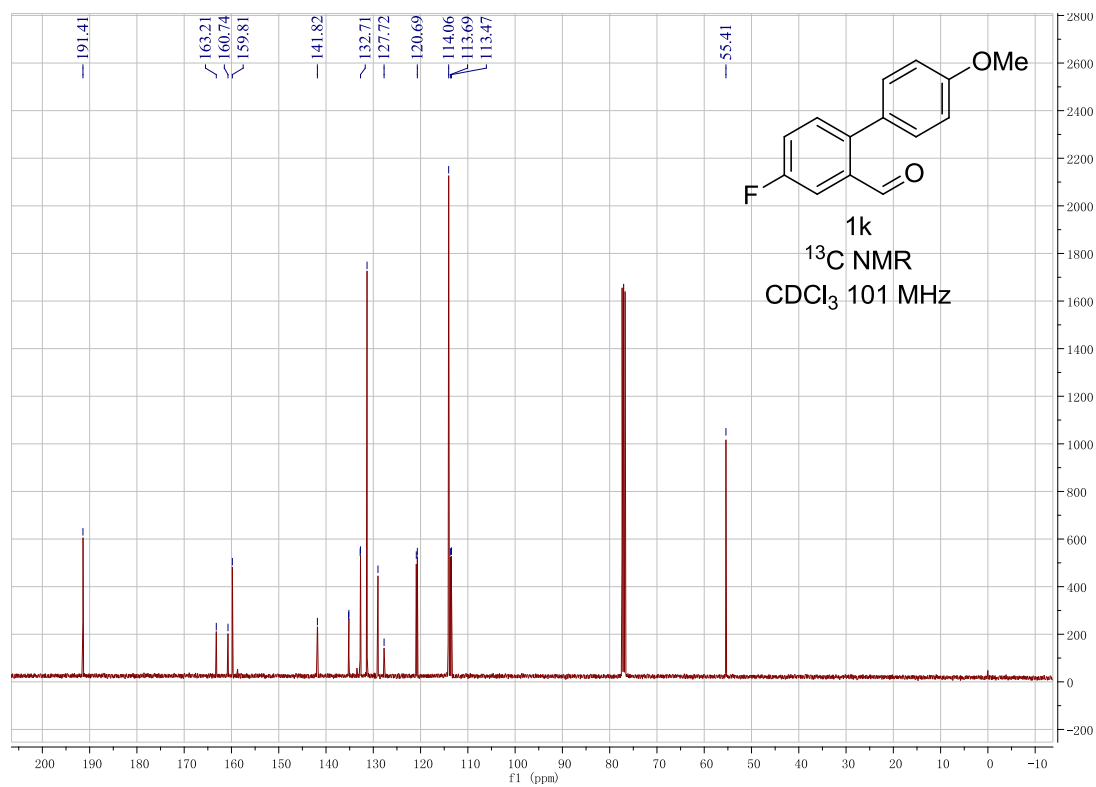
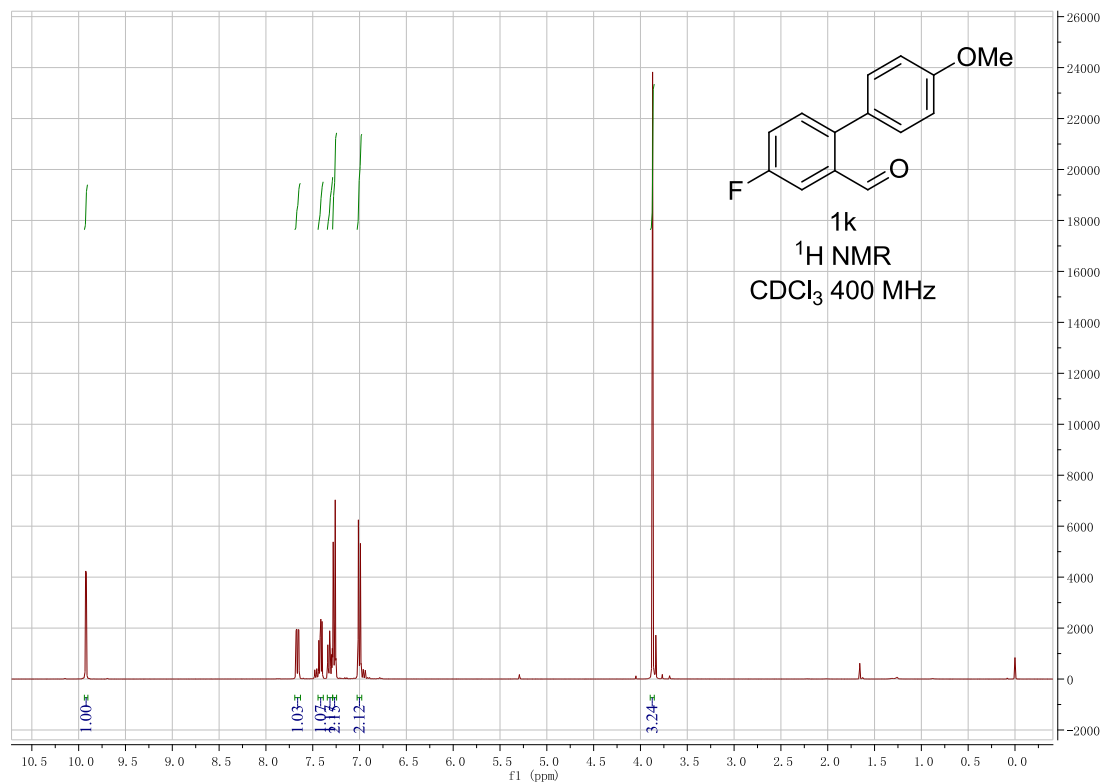


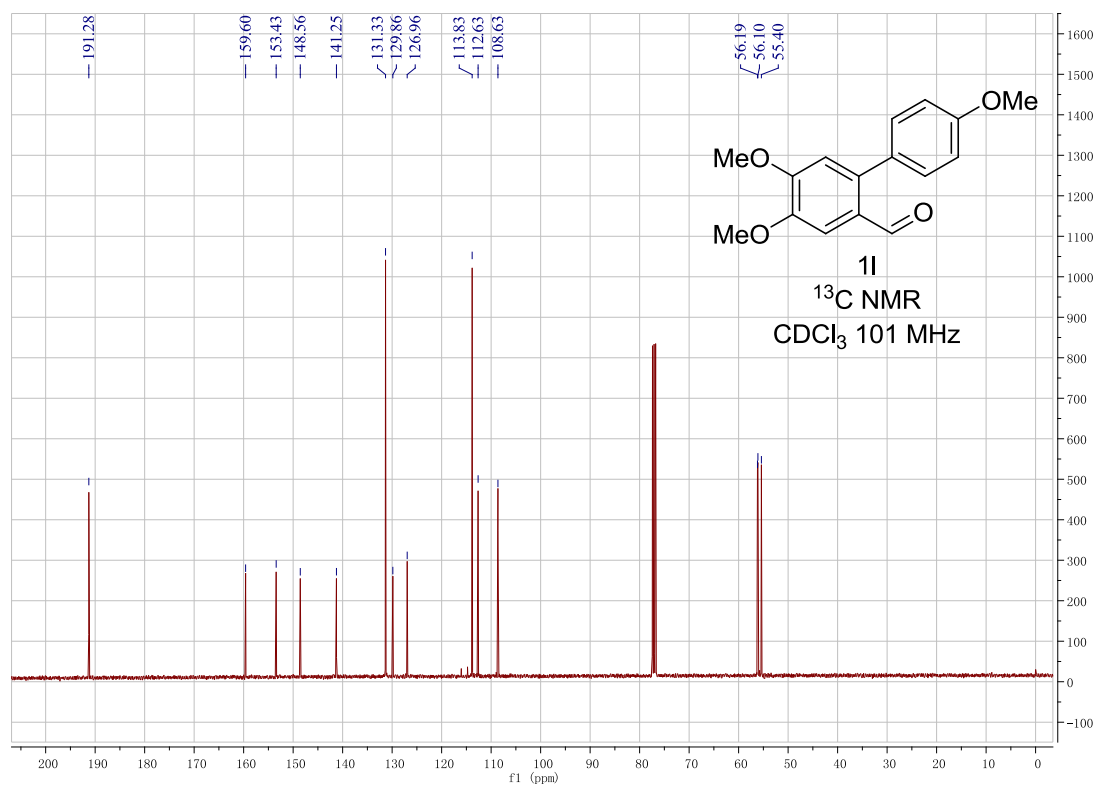
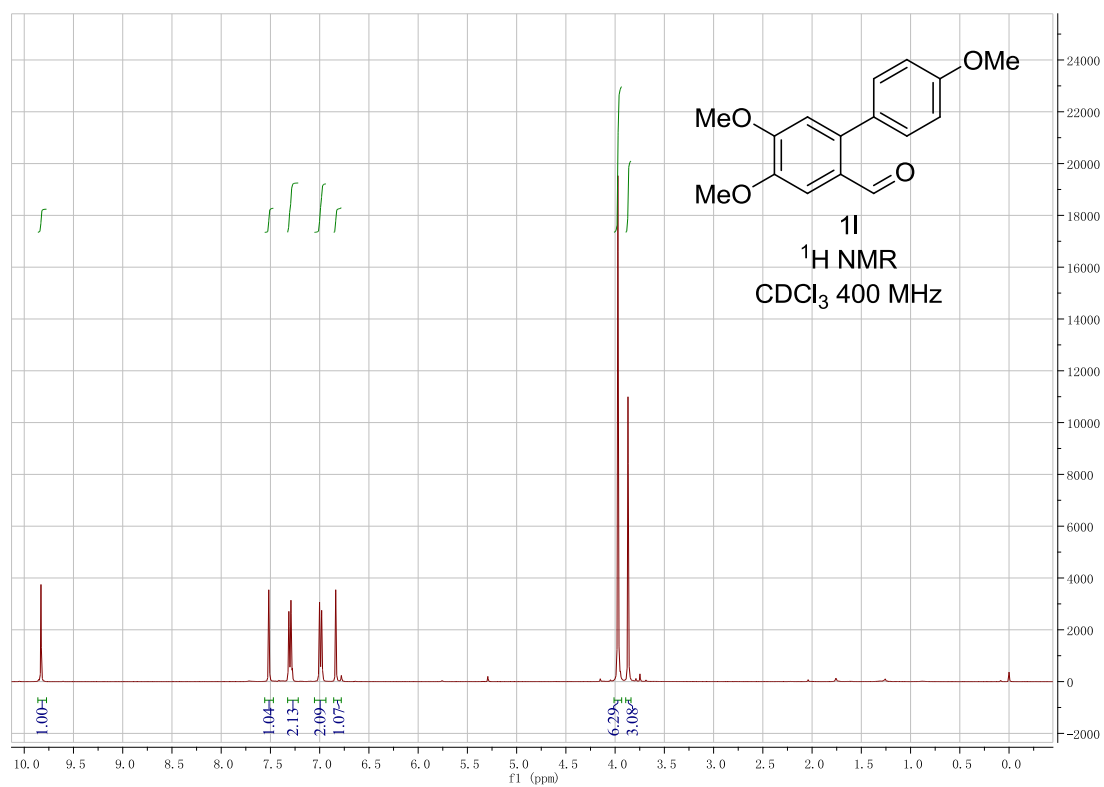
Trisphaeridine (8):¹⁷ A 100-mL round bottom flask was equipped with a rubber septum and magnetic stir bar and was charged with **7** (1.81 g, 8.0 mmol), **2a** (1.94g, 12.0 mmol), *fac*-Ir(ppy)₃ (1.5 mg, 1.16 mmol) and *p*-Cl-Benzenesulfonic acid (154mg, 0.8 mmol). The flask was evacuated and backfilled with N₂ for 3 times. DMF (80 mL) was then added with syringe under N₂. The mixture was then irradiated by white LED strips for 30 h. After the reaction was complete (as judged by TLC analysis), the mixture was concentrated under vacuum to remove DMF. The residue was then purified by flash chromatography on silica gel (EtOAc:hexane = 1:1) to afford **8** (1.43g, 80%) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 9.07 (s, 1H), 8.34 (d, *J* = 8.1 Hz, 1H), 8.15 (d, *J* = 8.1 Hz, 1H), 7.86 (s, 1H), 7.69 (t, *J* = 7.4 Hz, 1H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.29 (s, 1H), 6.14 (s, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 151.77, 151.38, 148.13, 144.12, 130.15, 130.05, 127.95, 126.63, 124.23, 123.03, 121.96, 105.41, 101.89, 99.86.

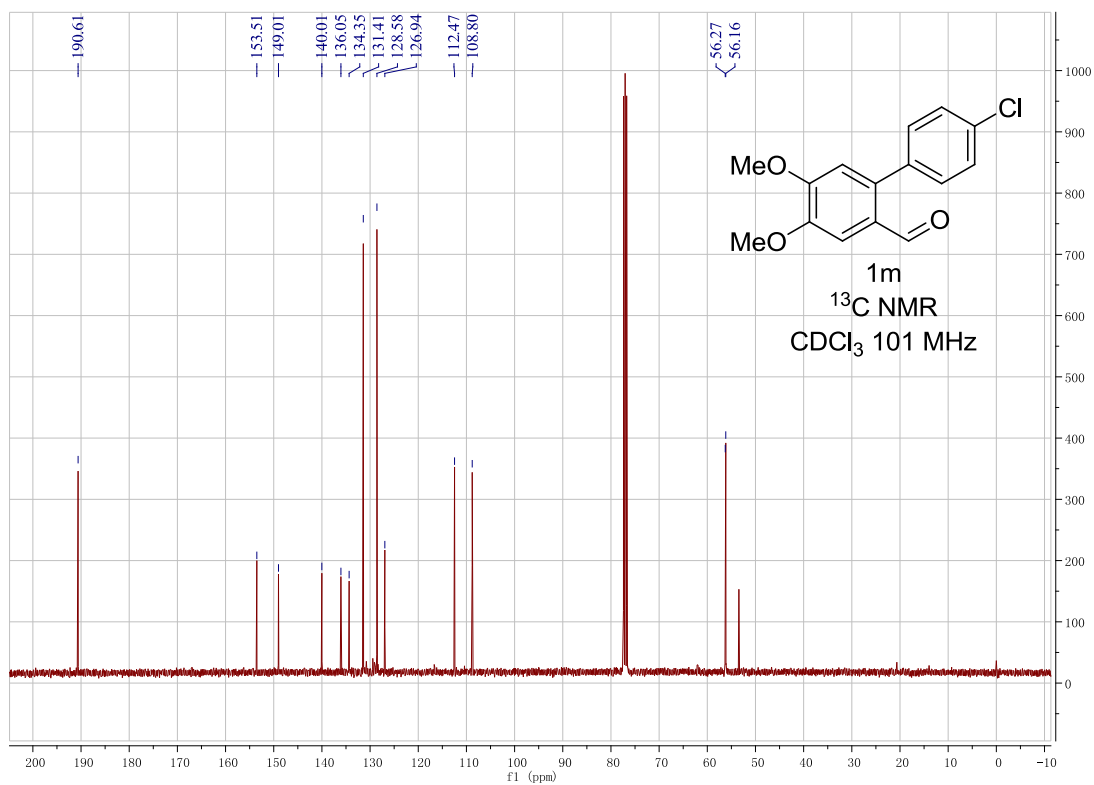
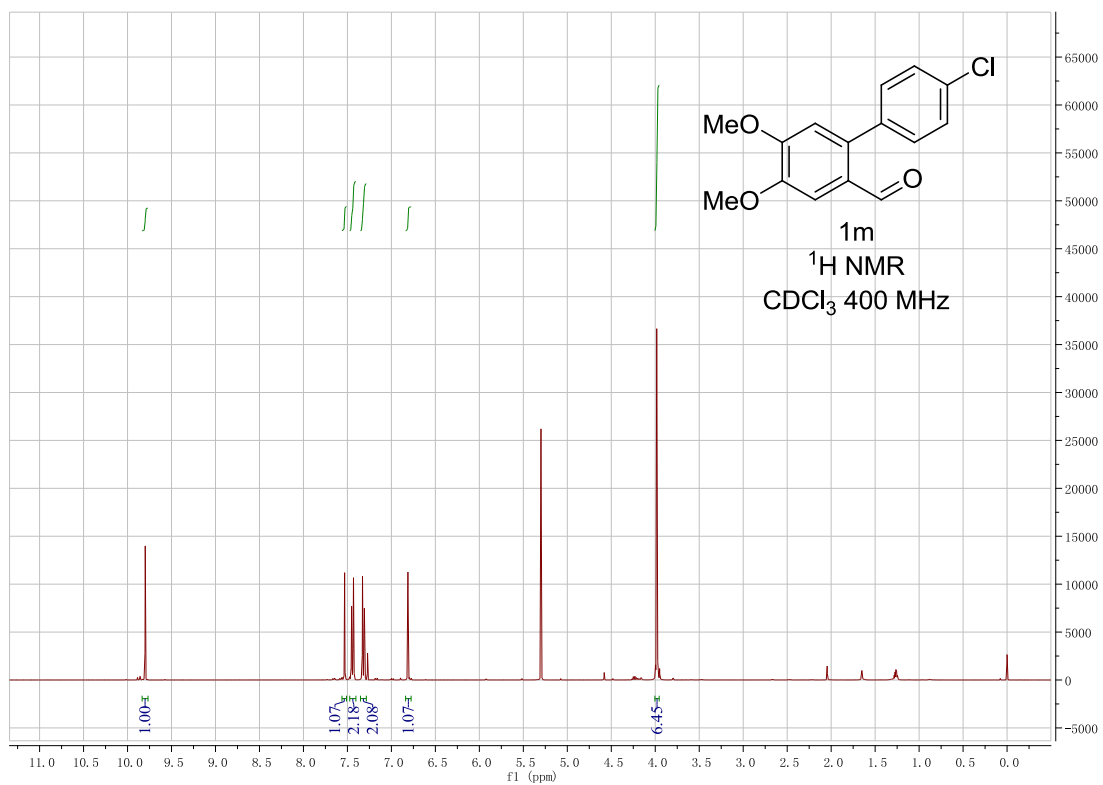
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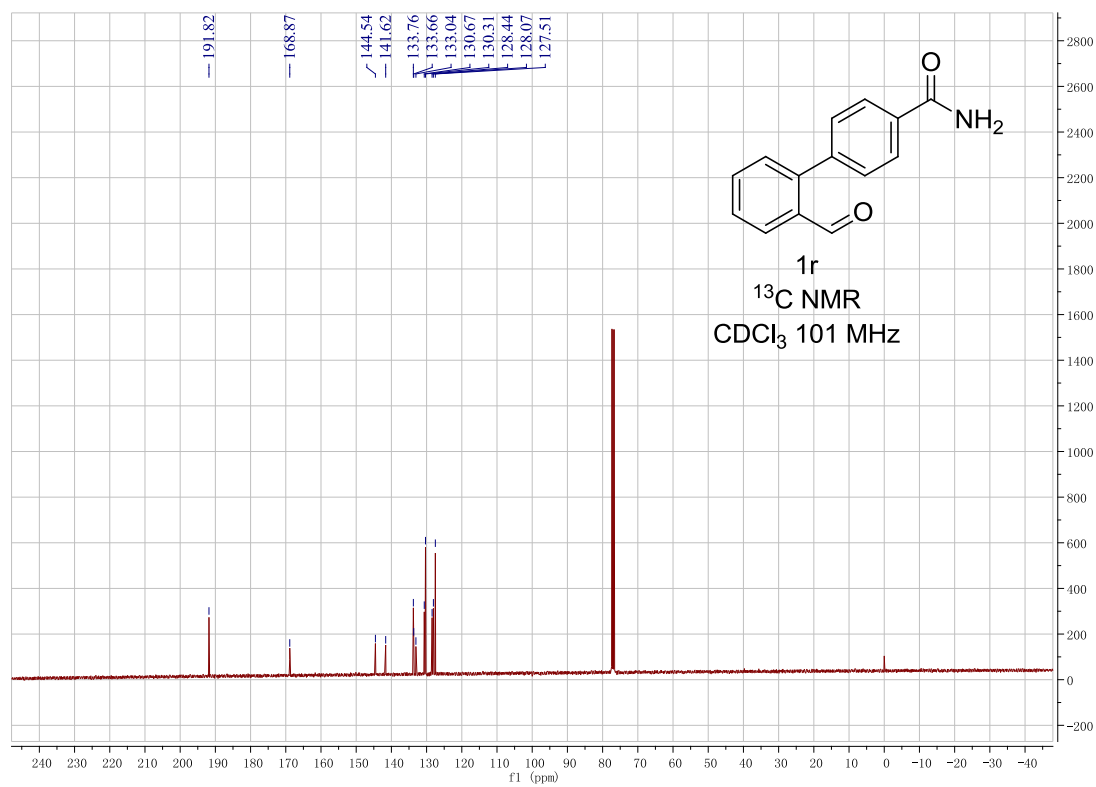
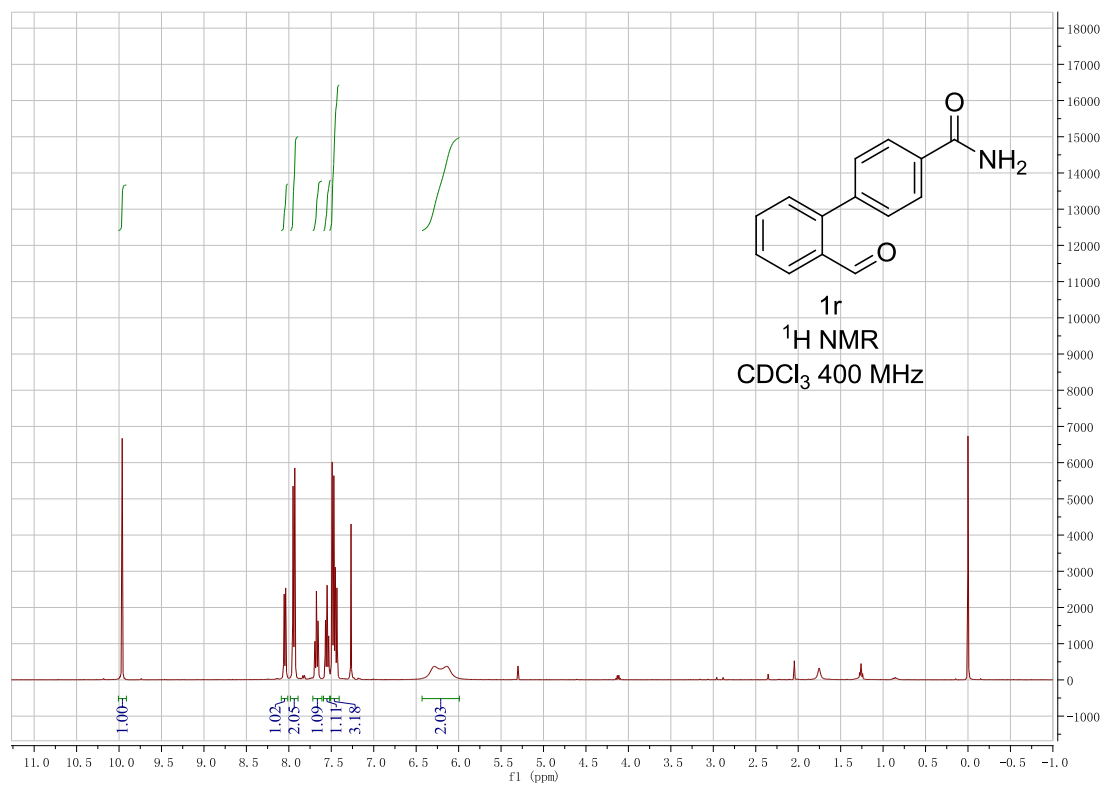
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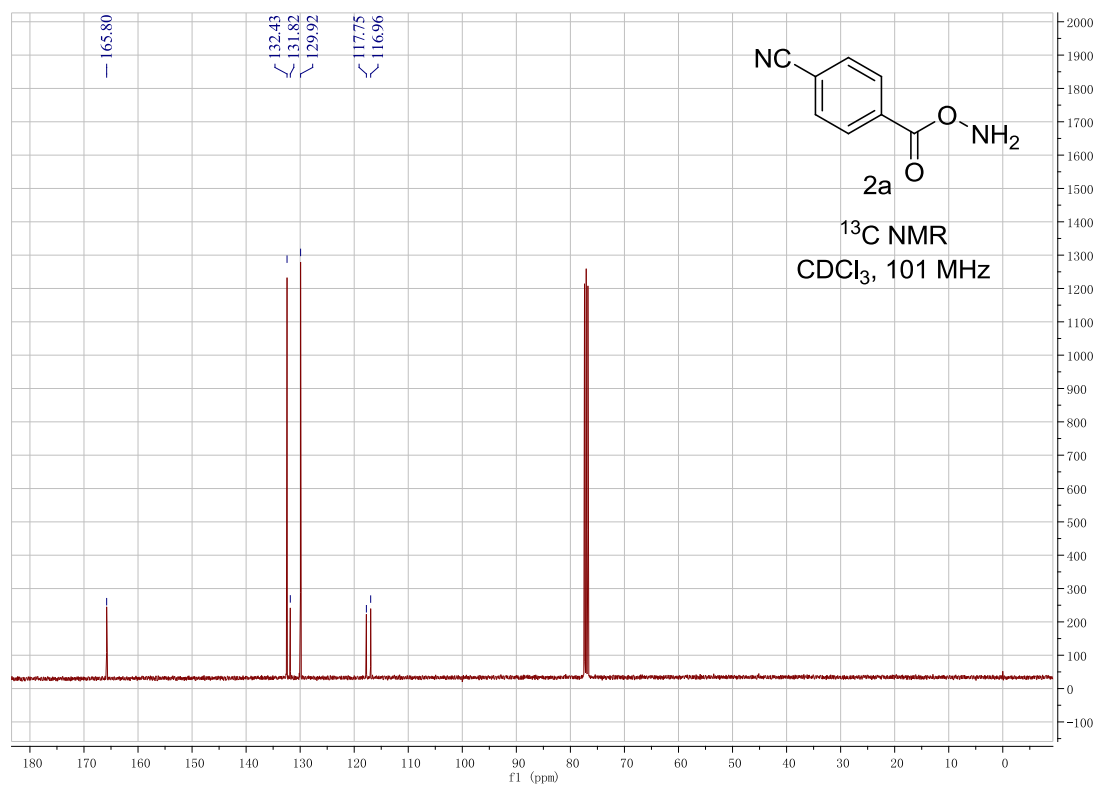
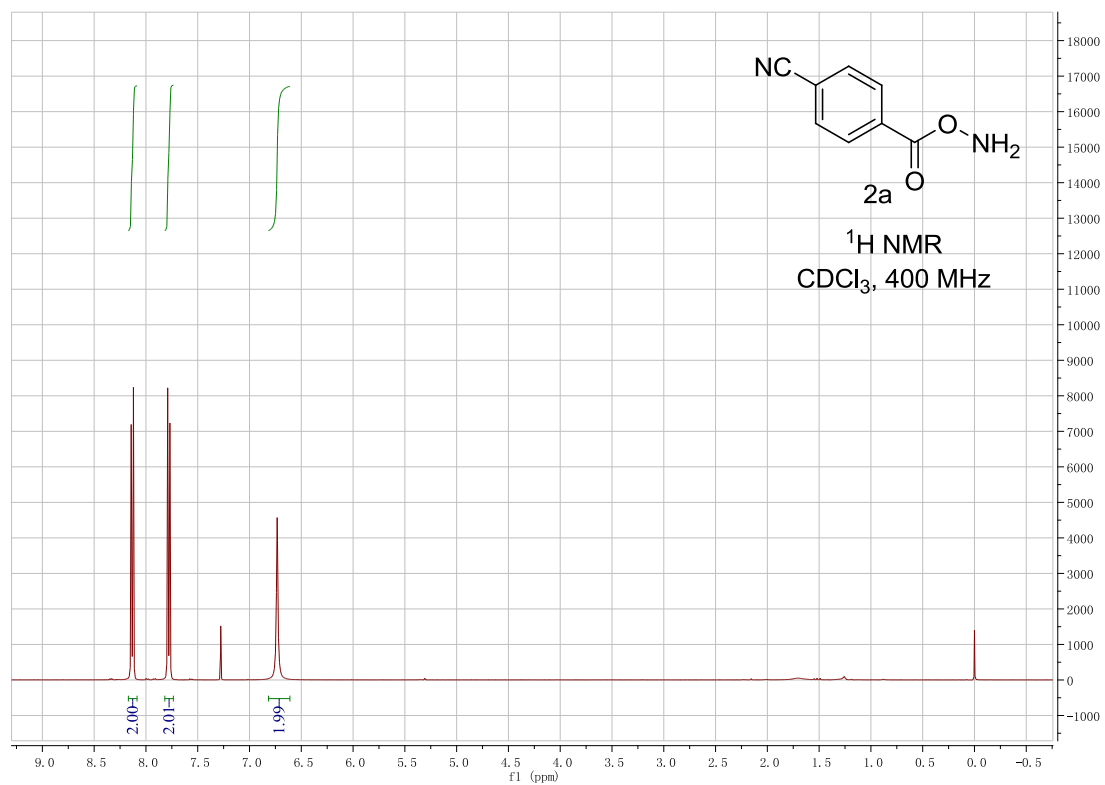
10. NMR spectra for all compounds

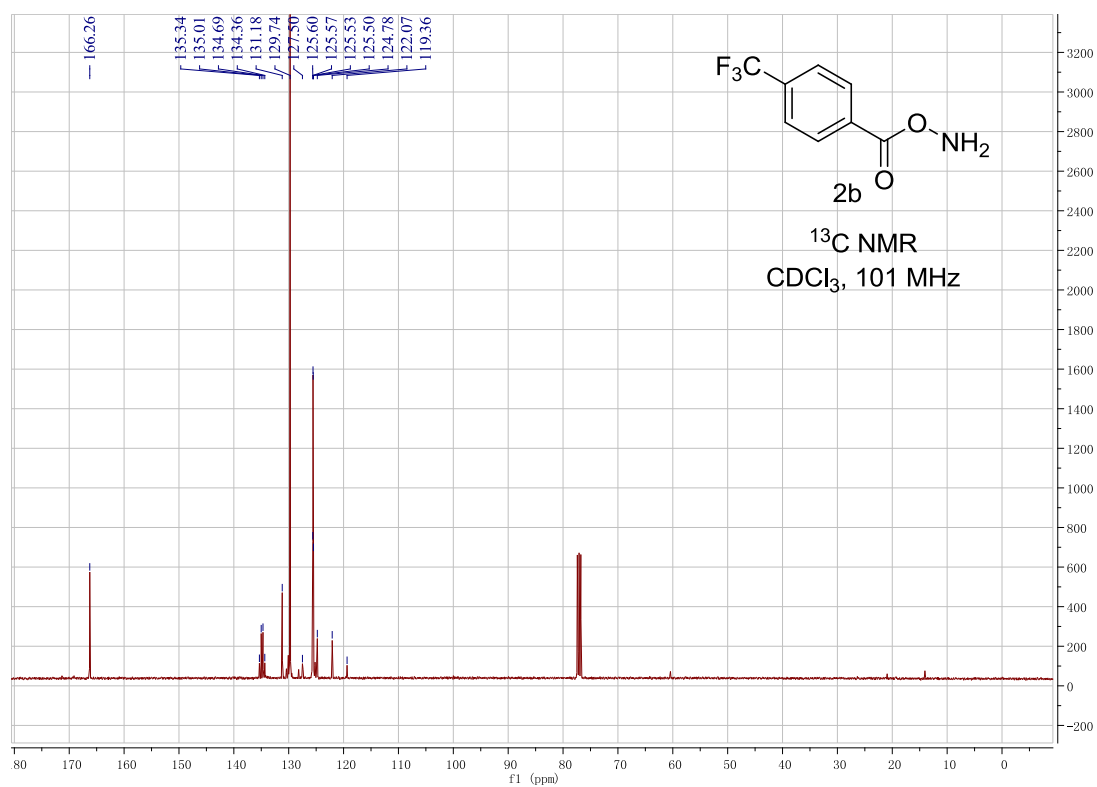
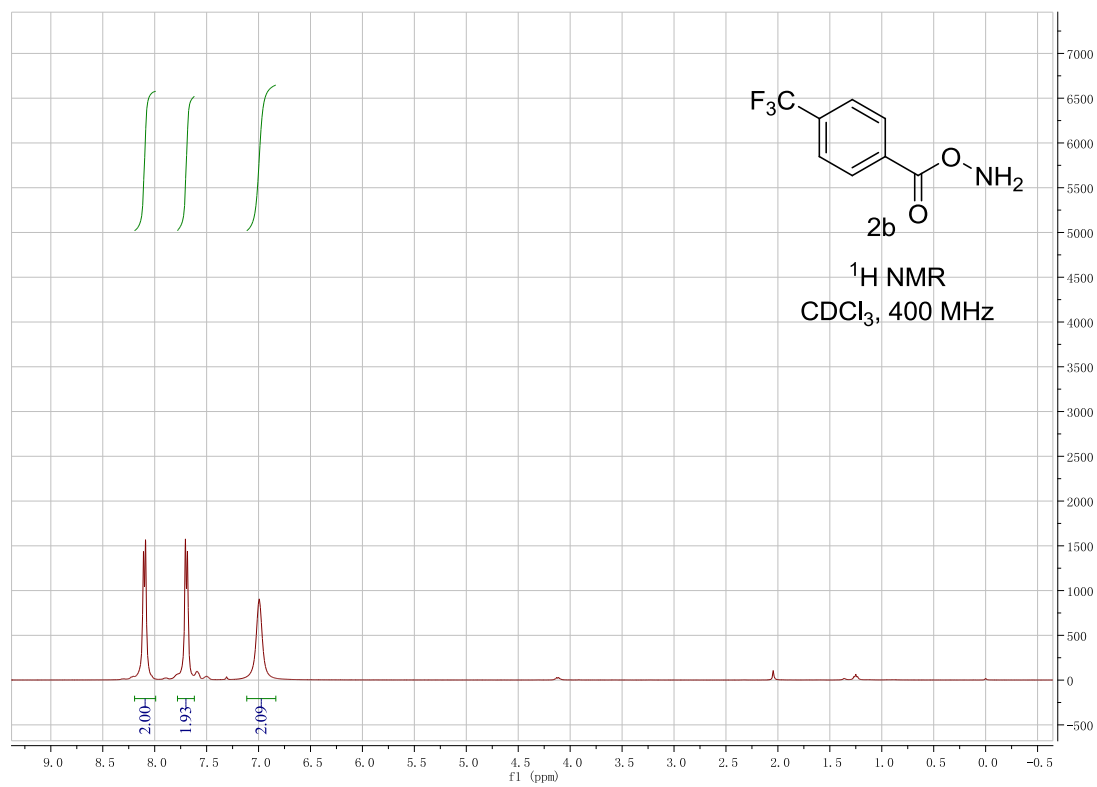


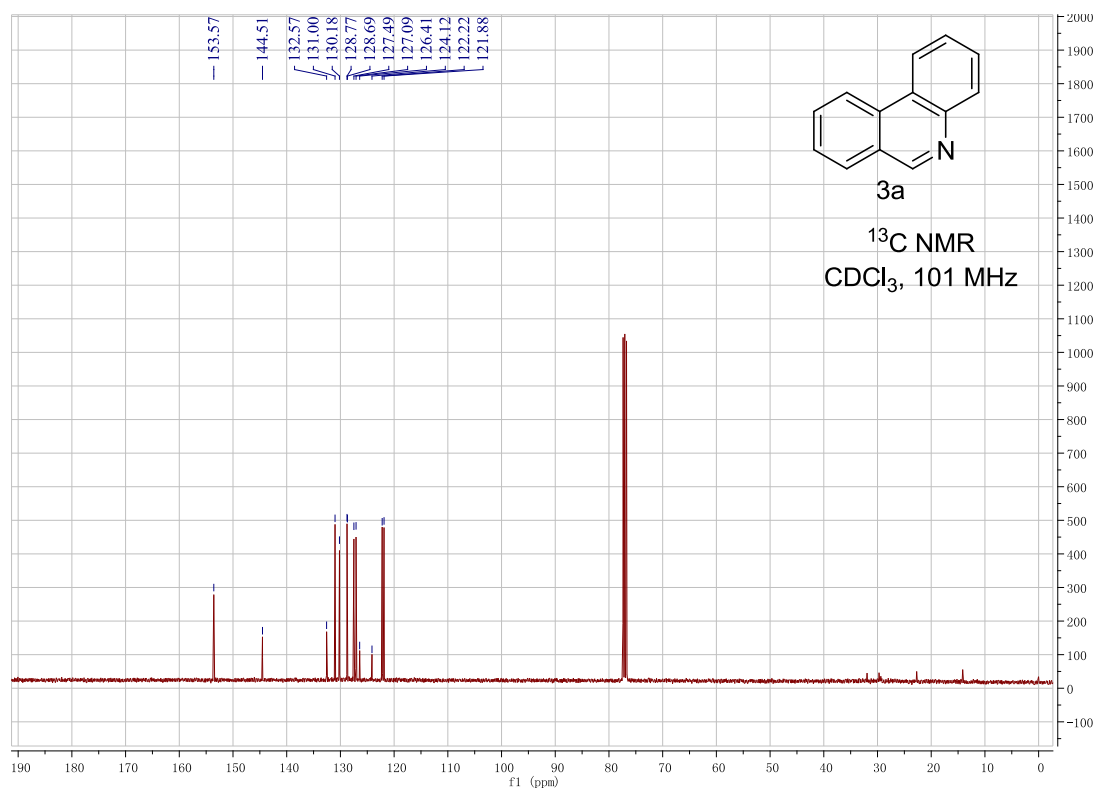
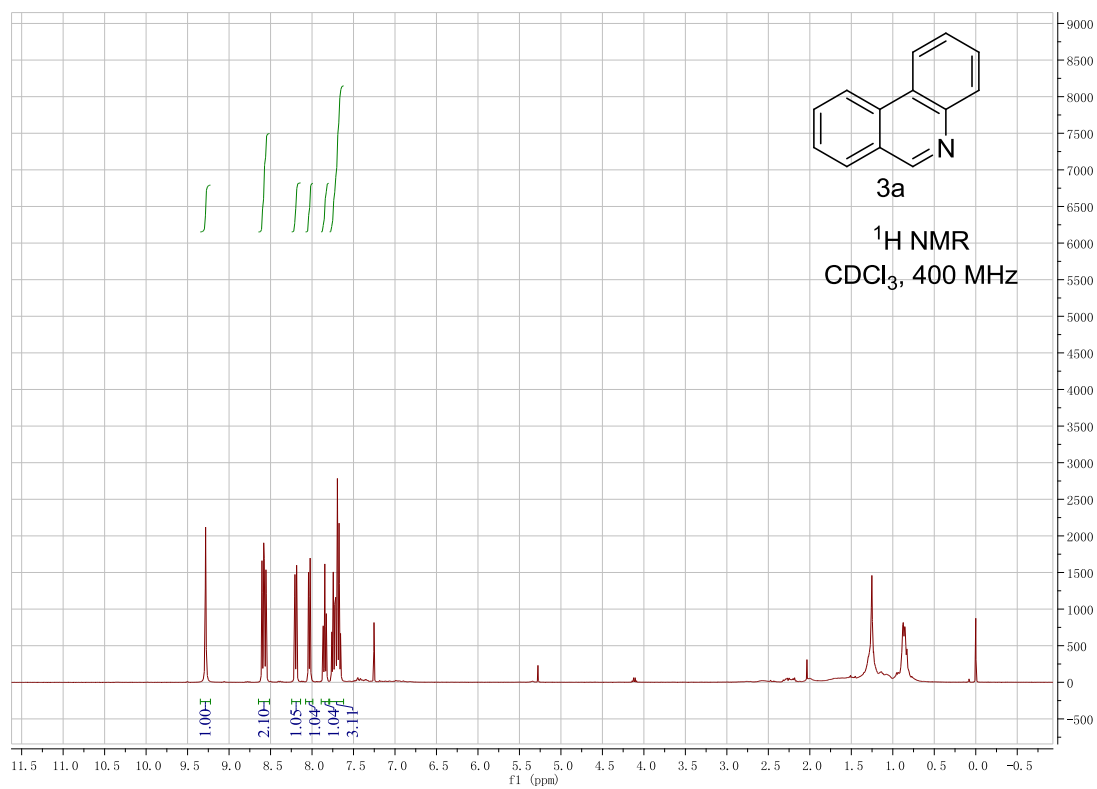


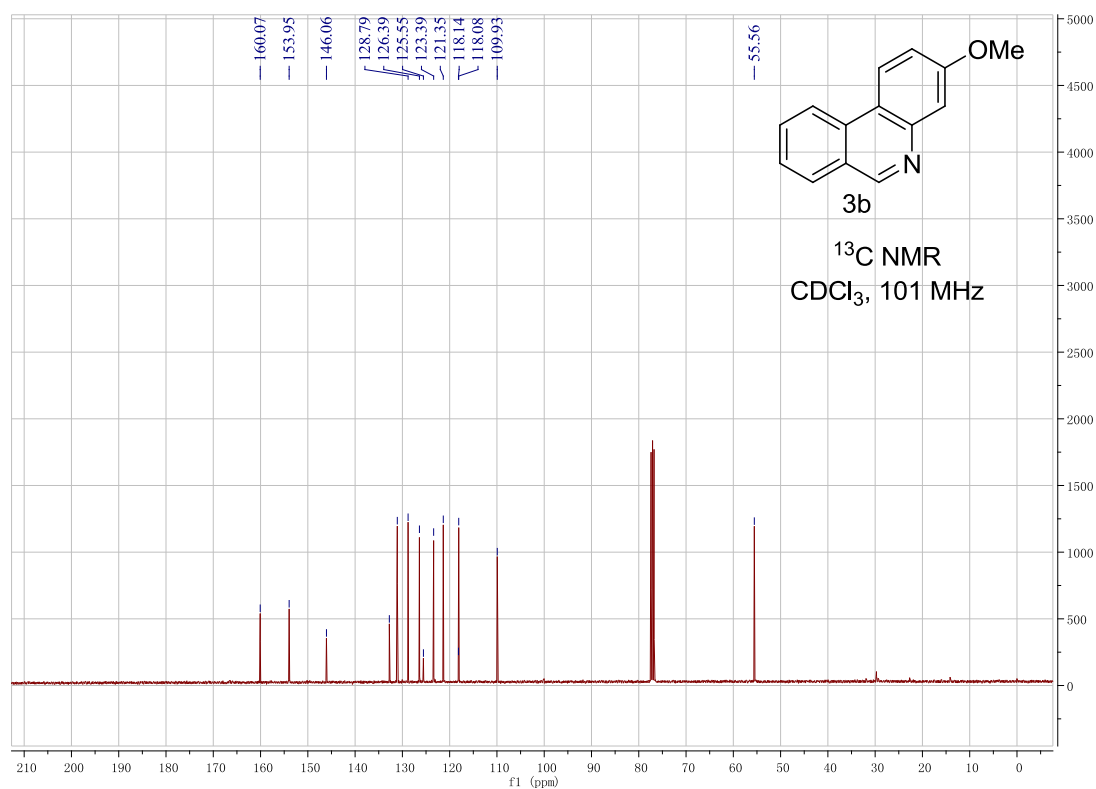
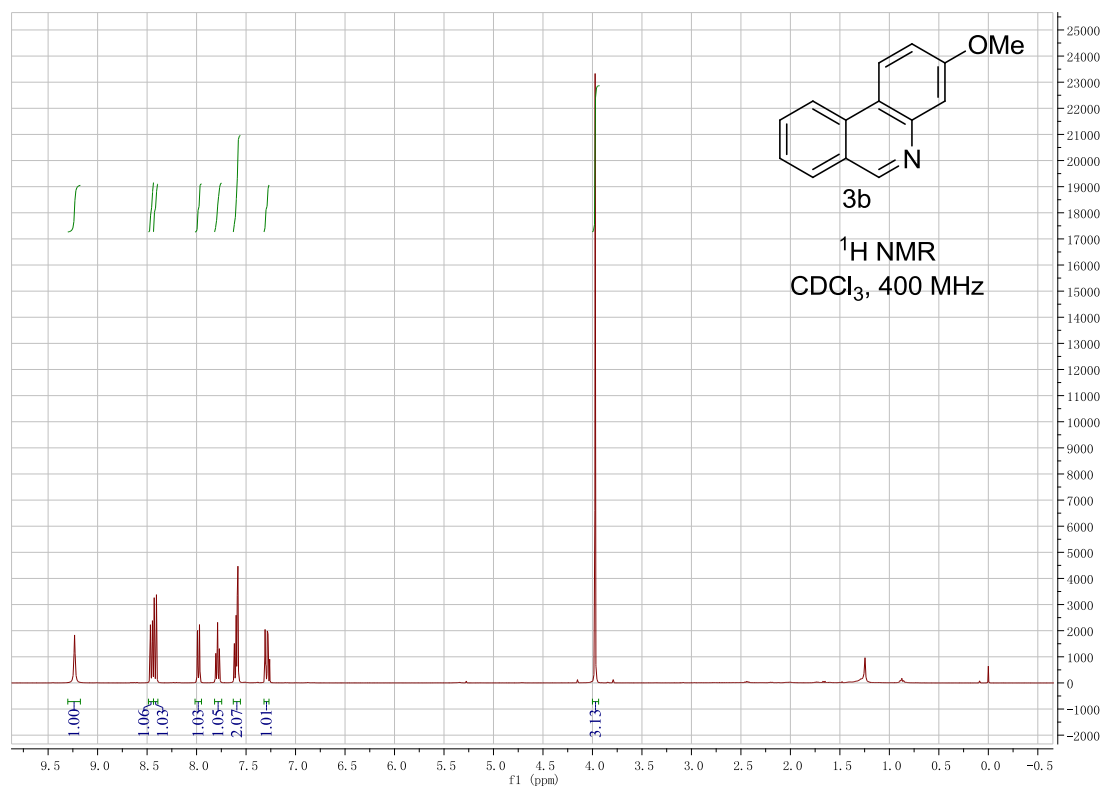


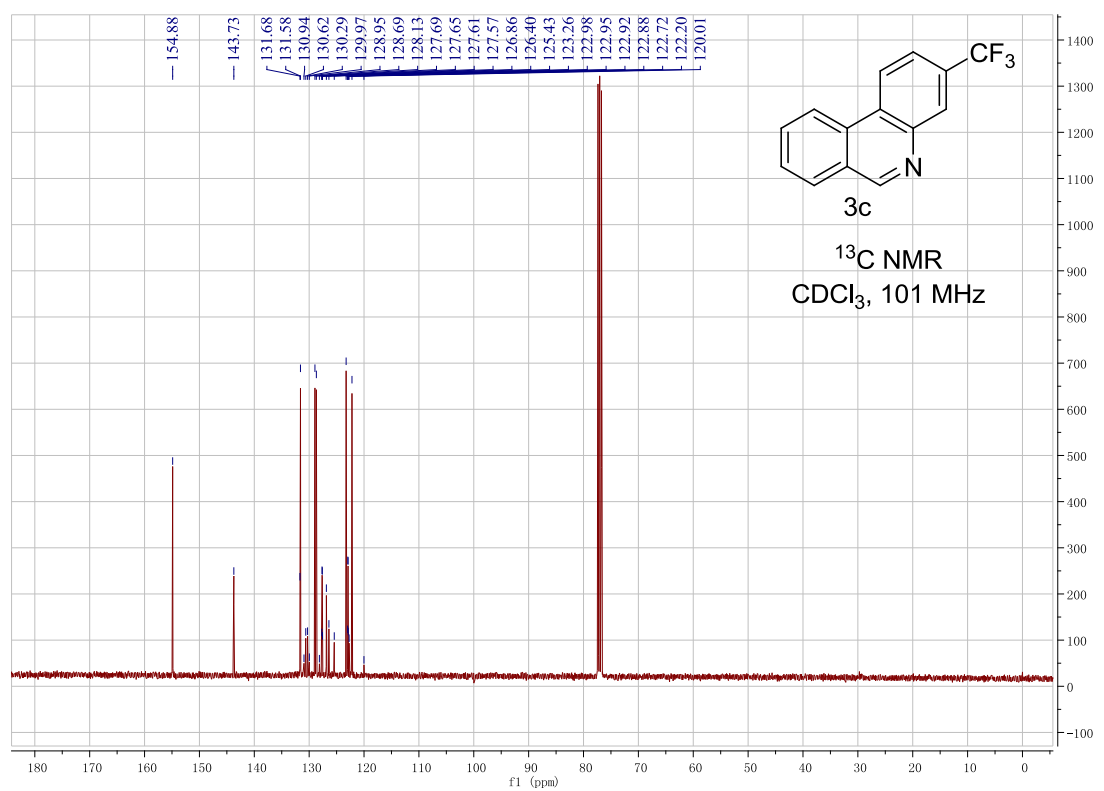
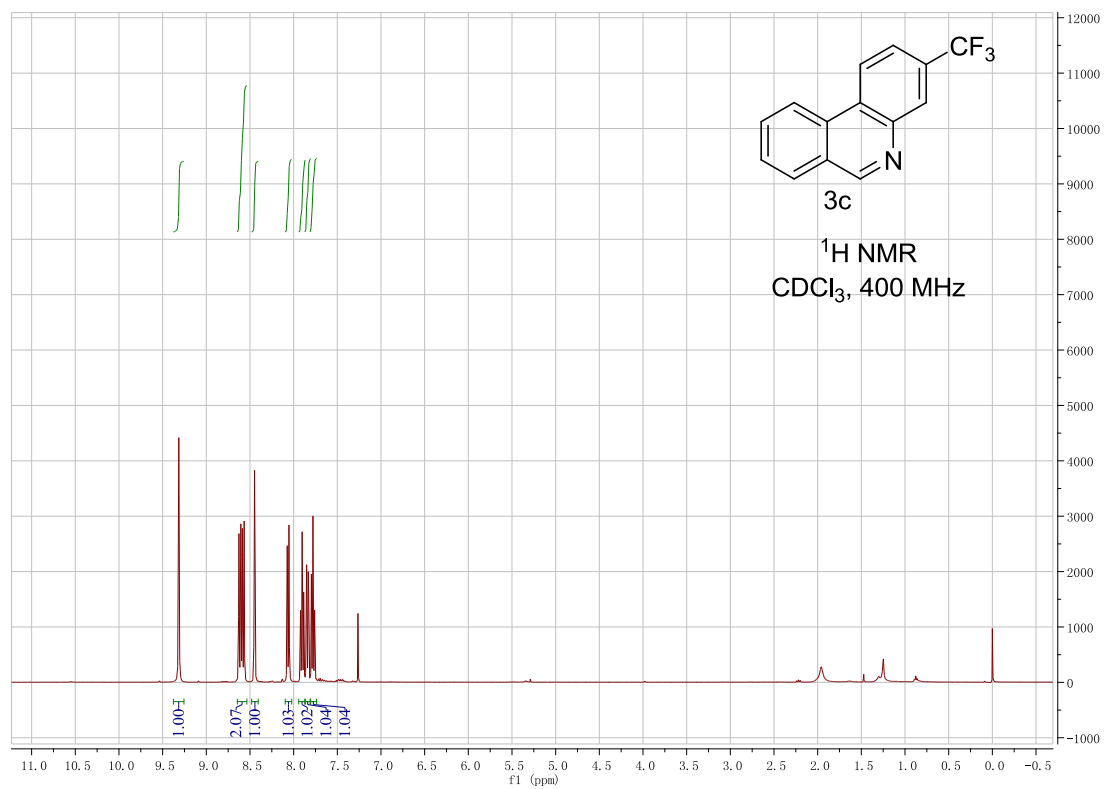


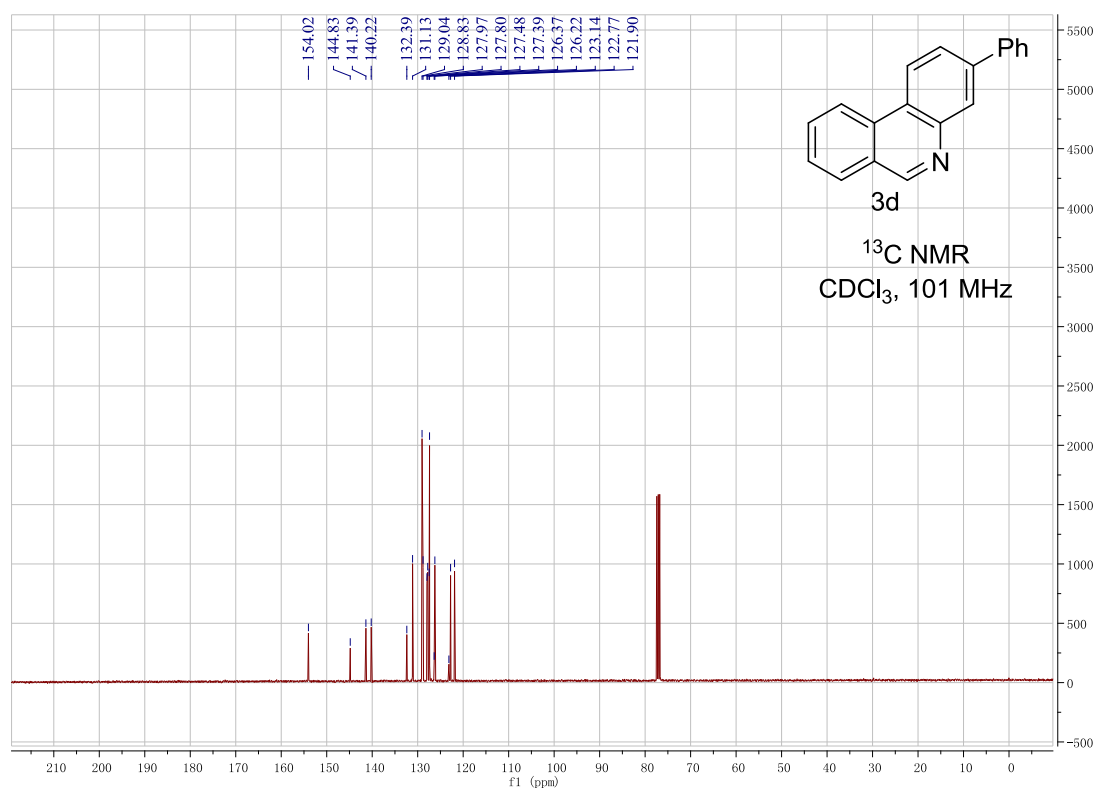
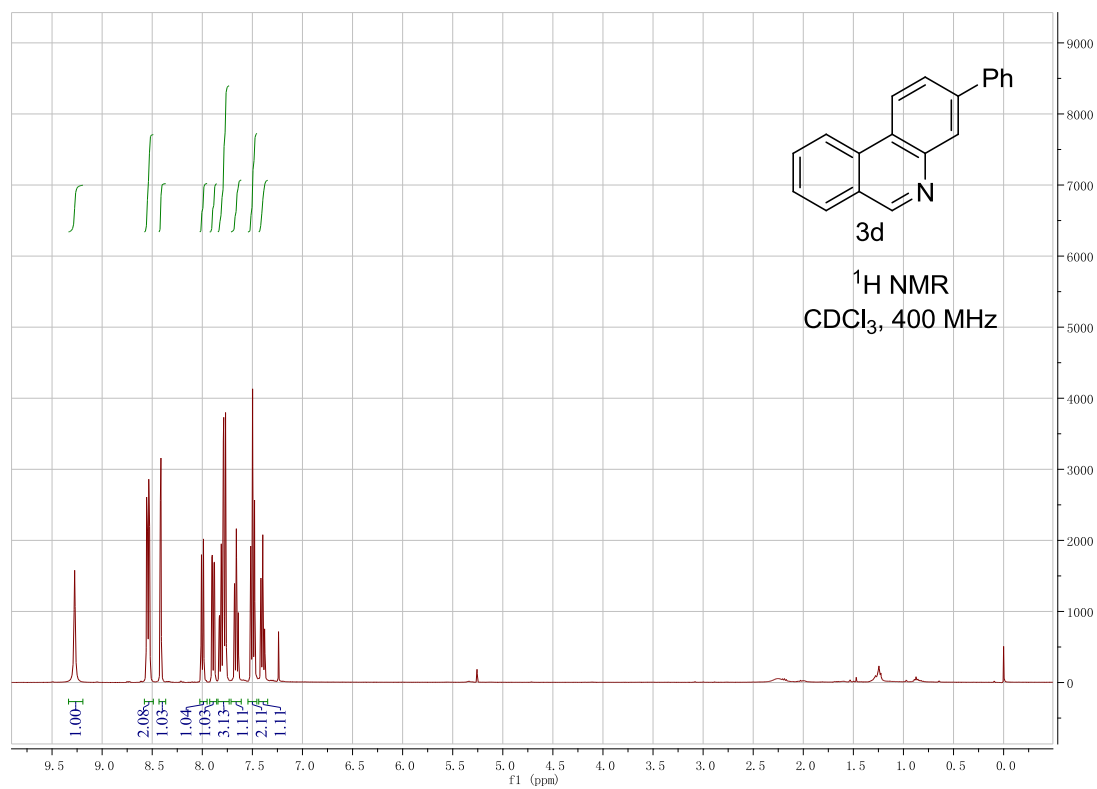


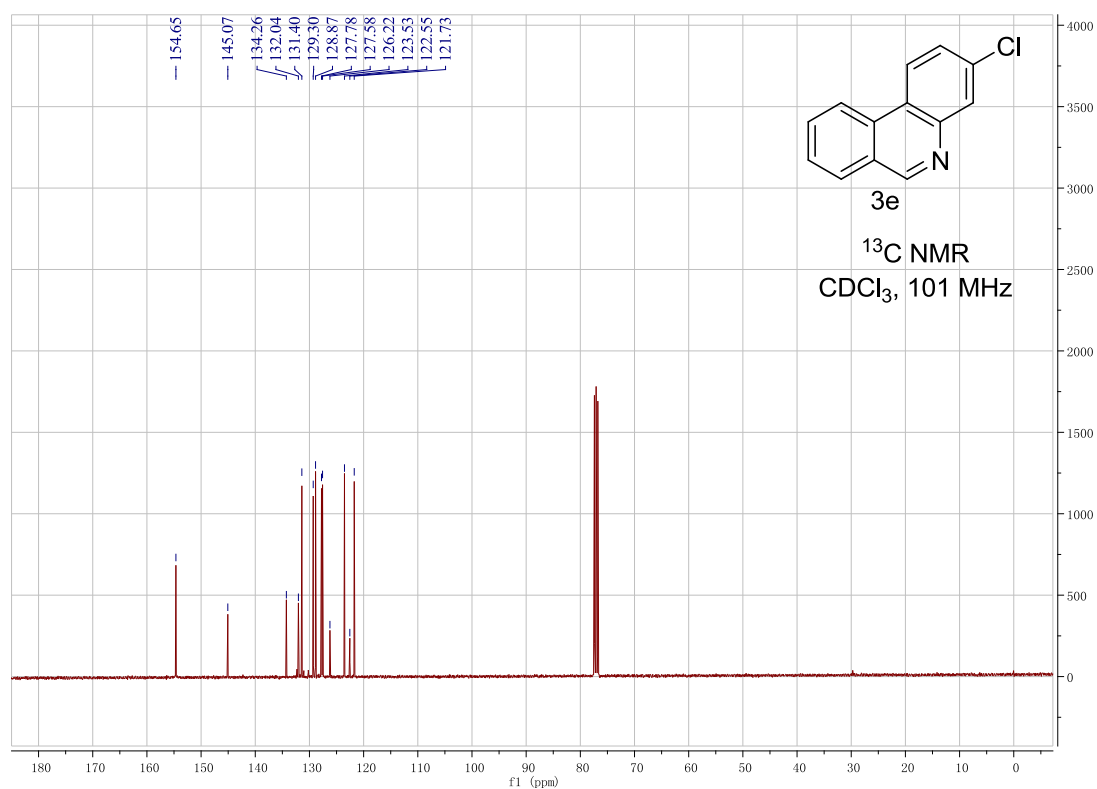
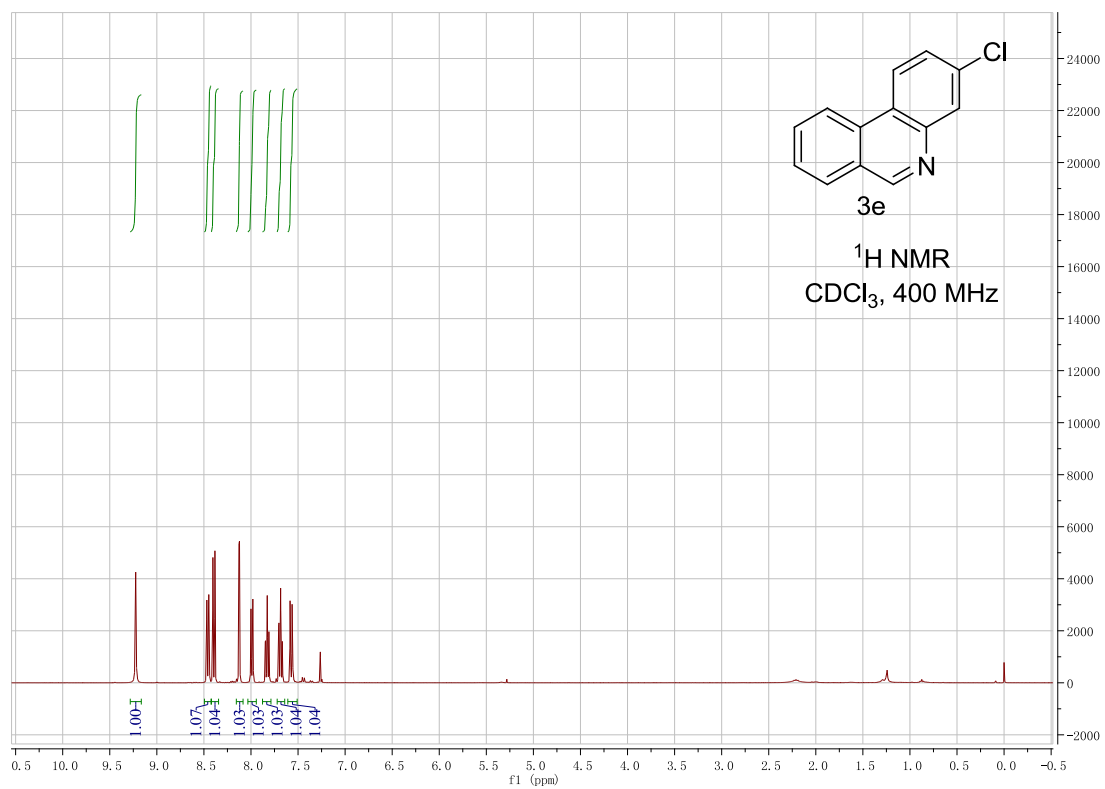


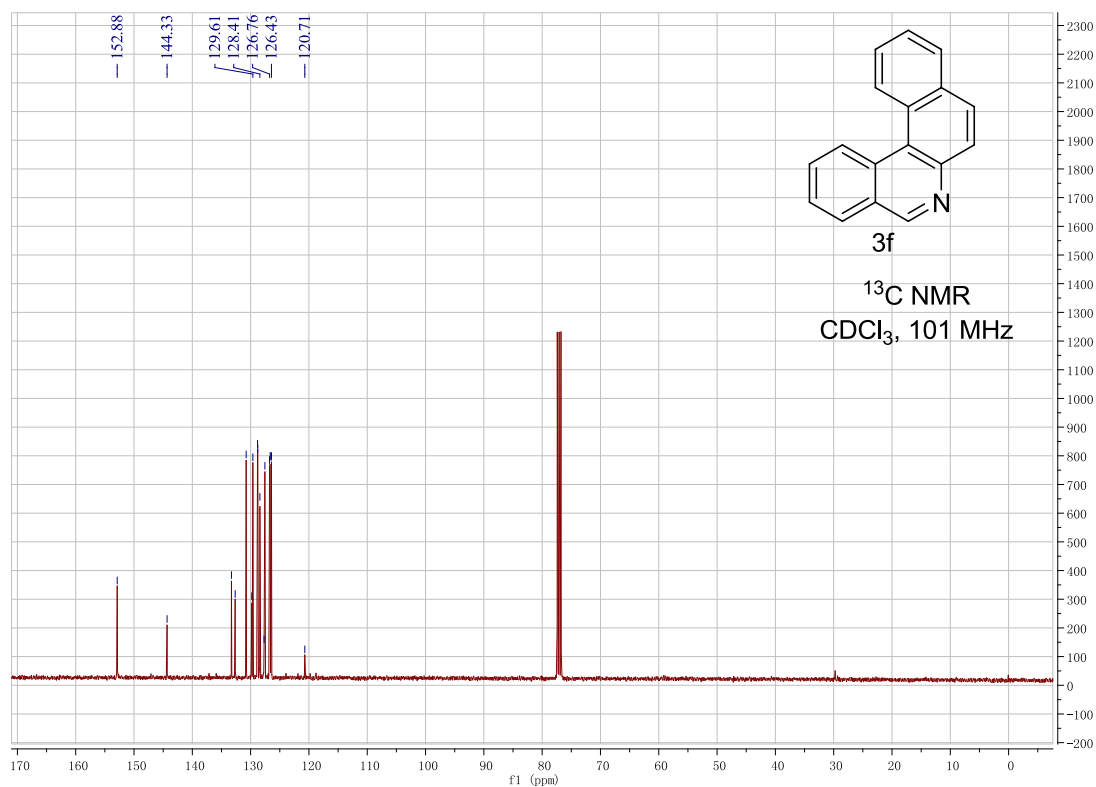
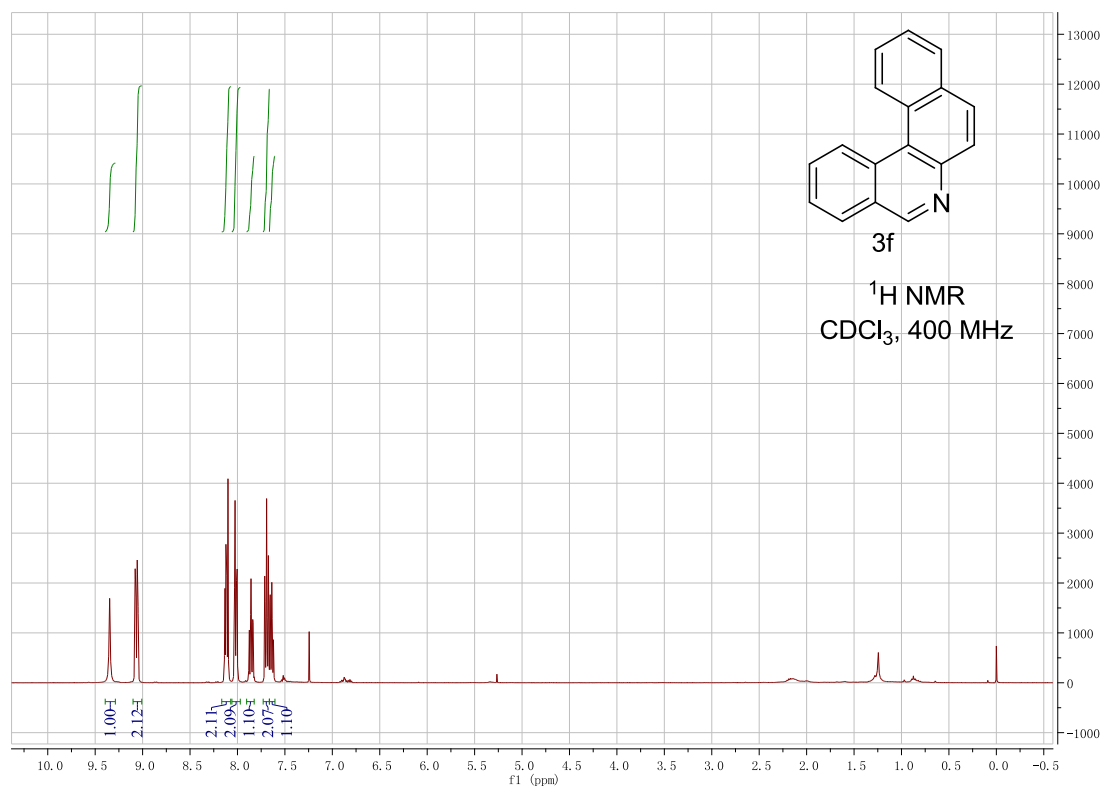


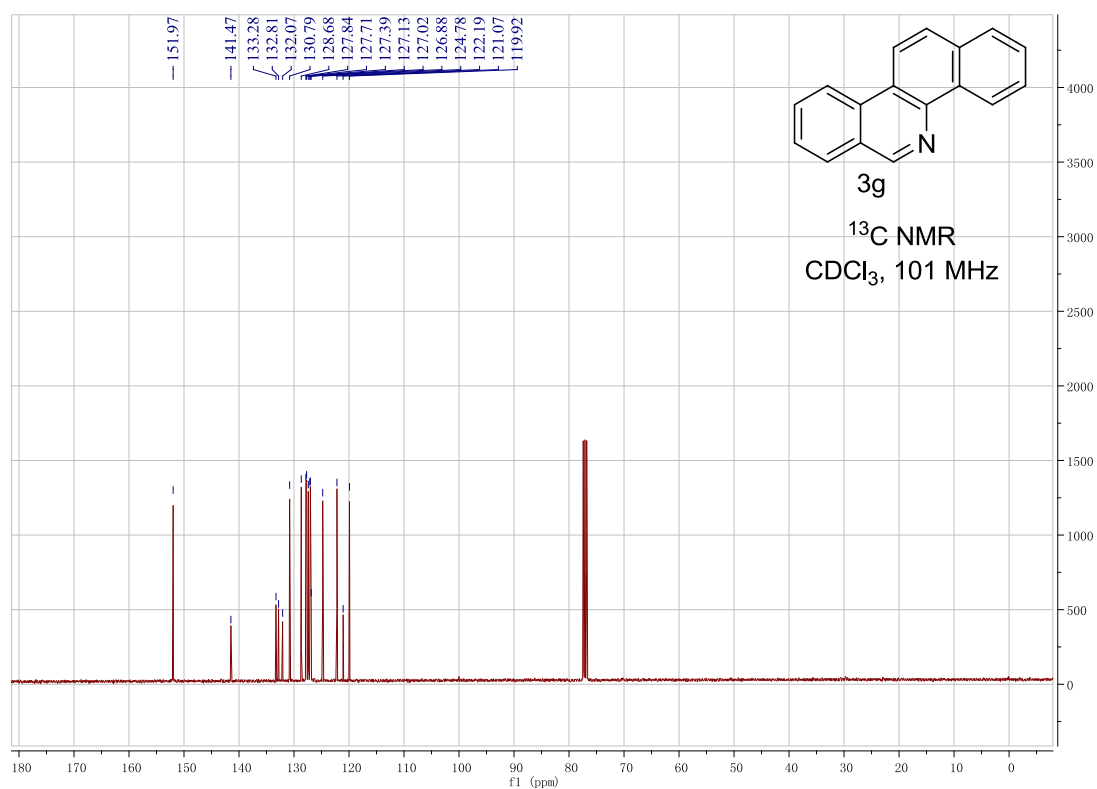
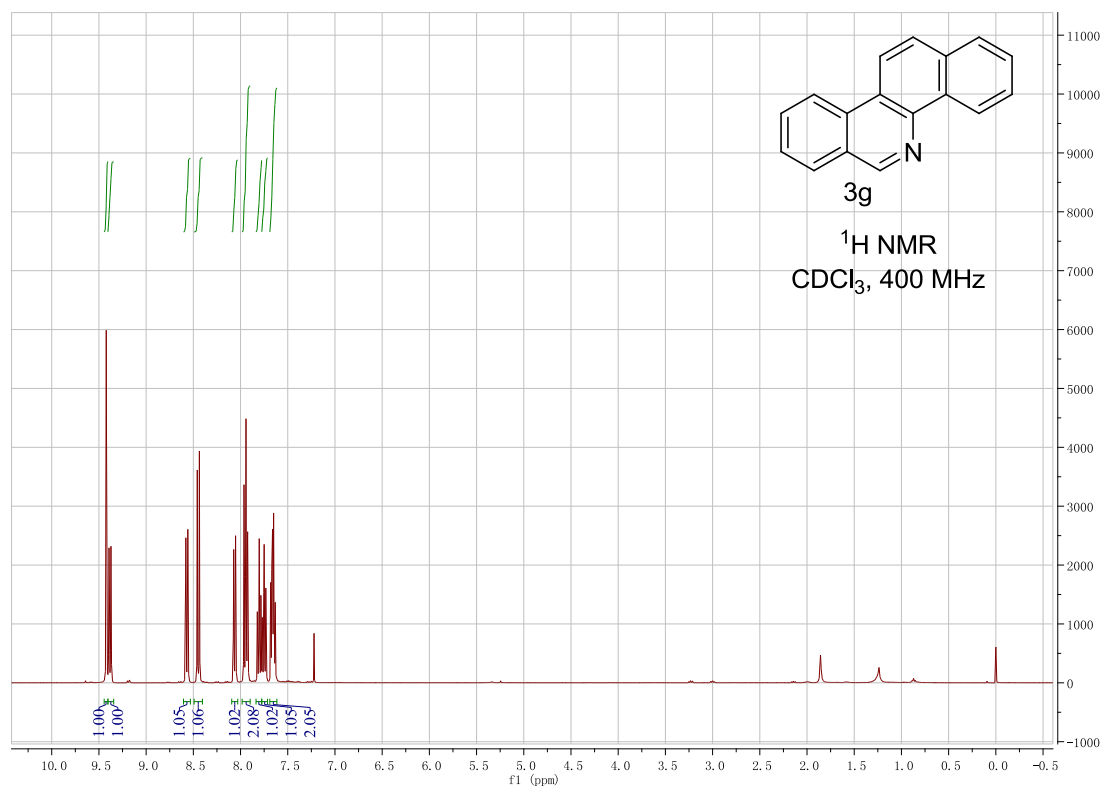


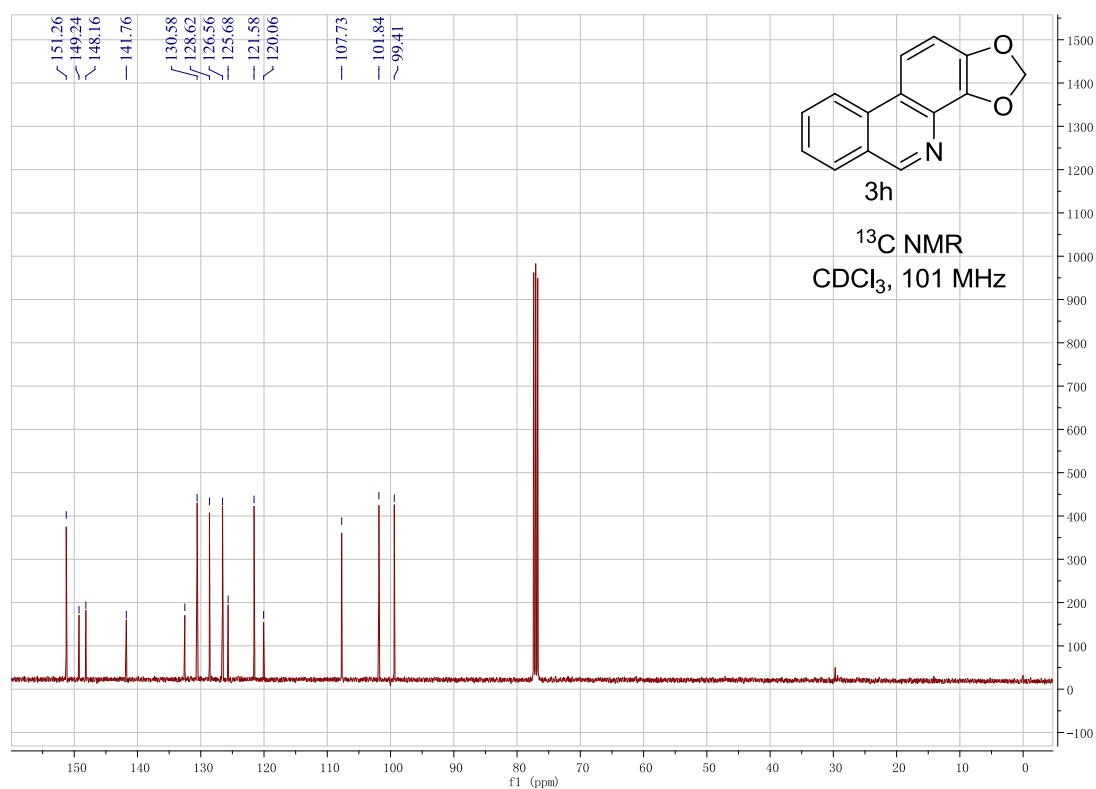
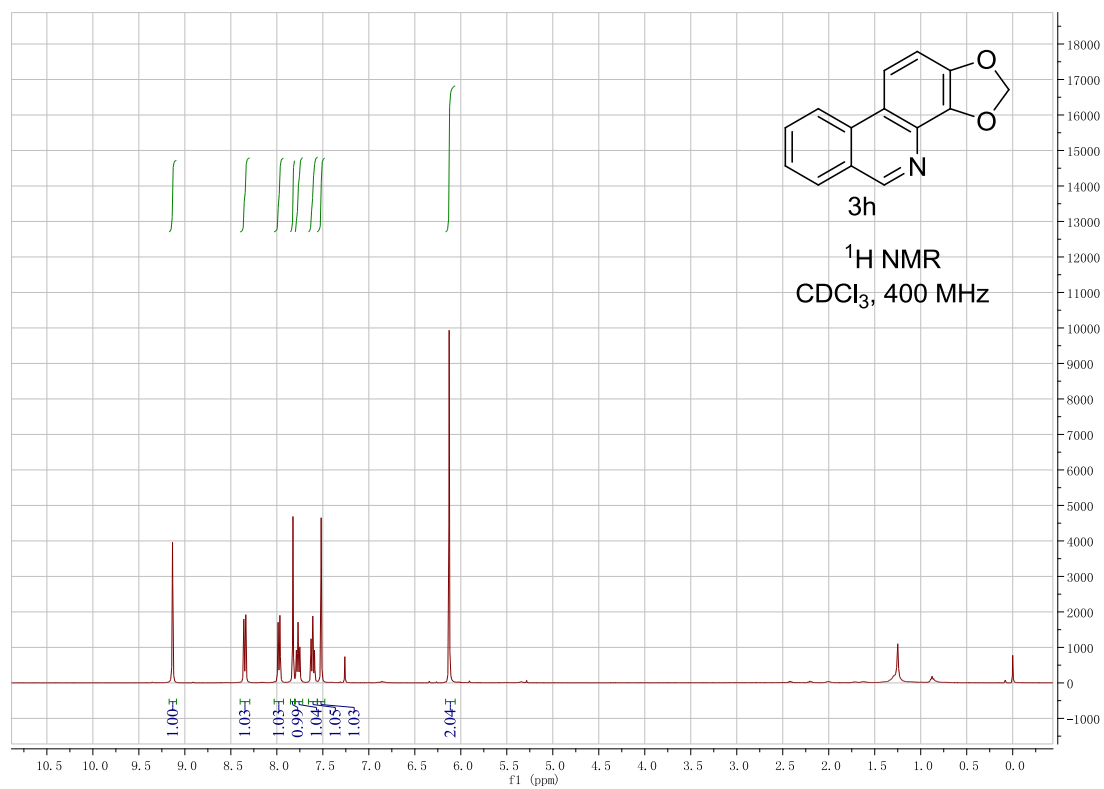


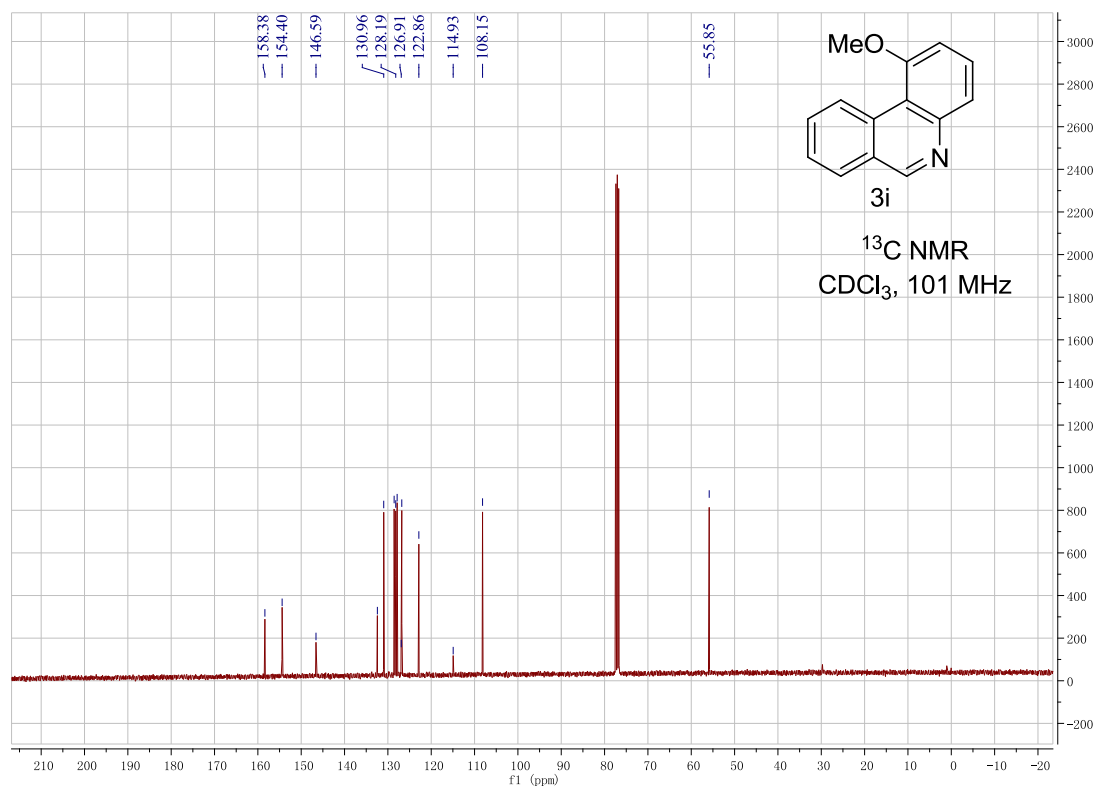
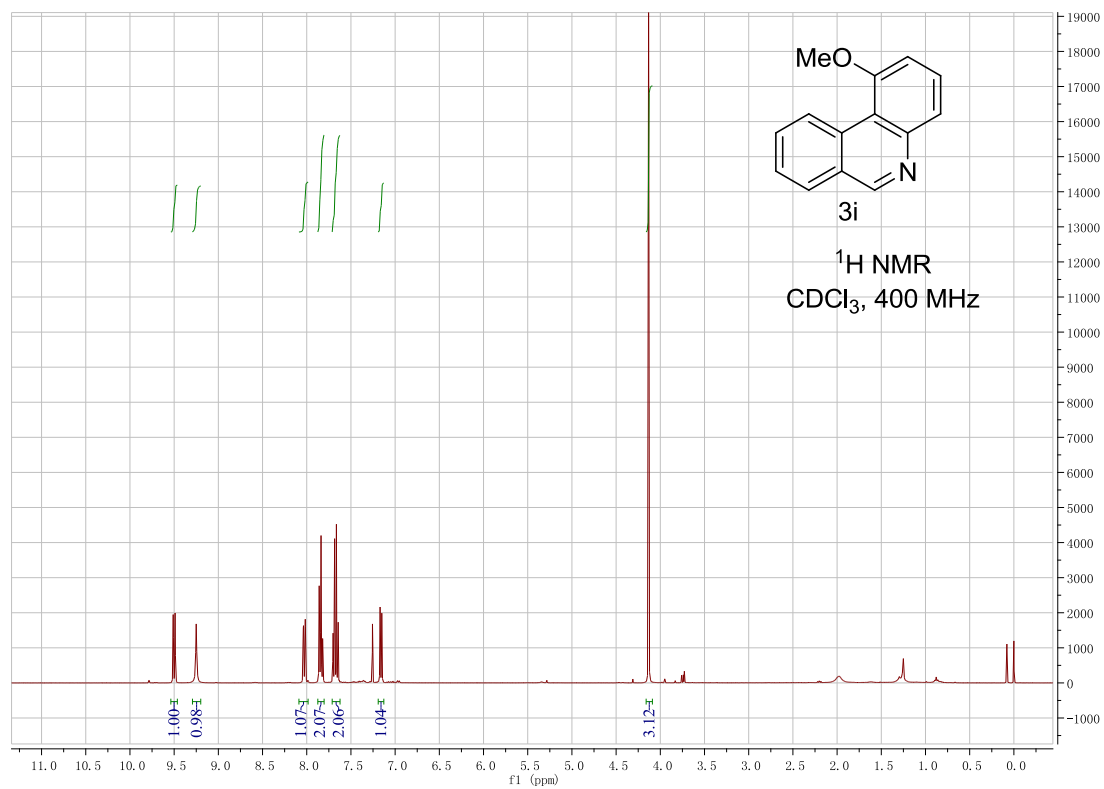


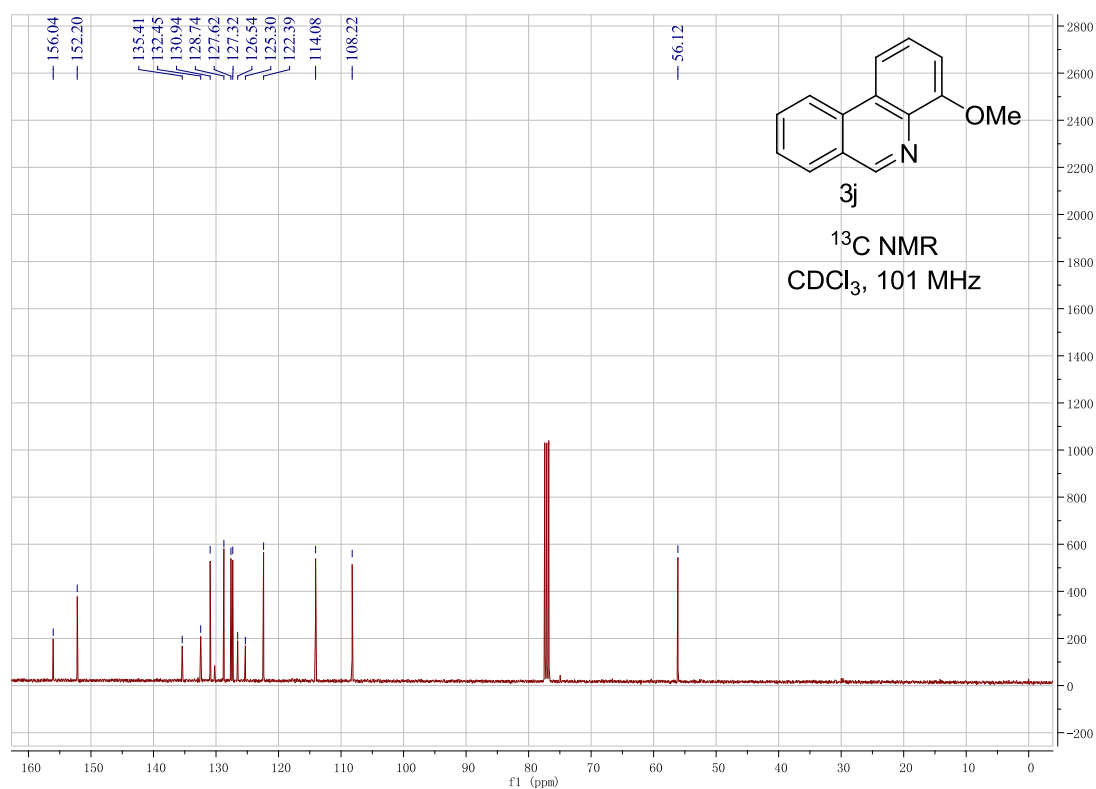
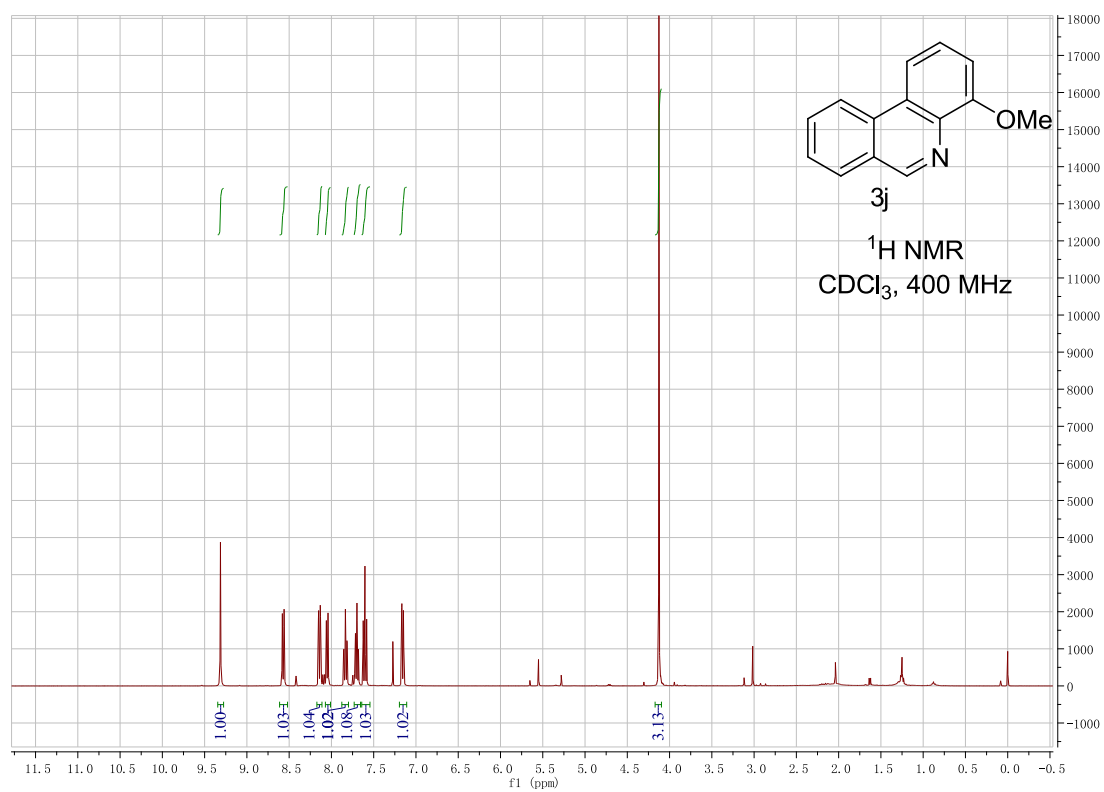


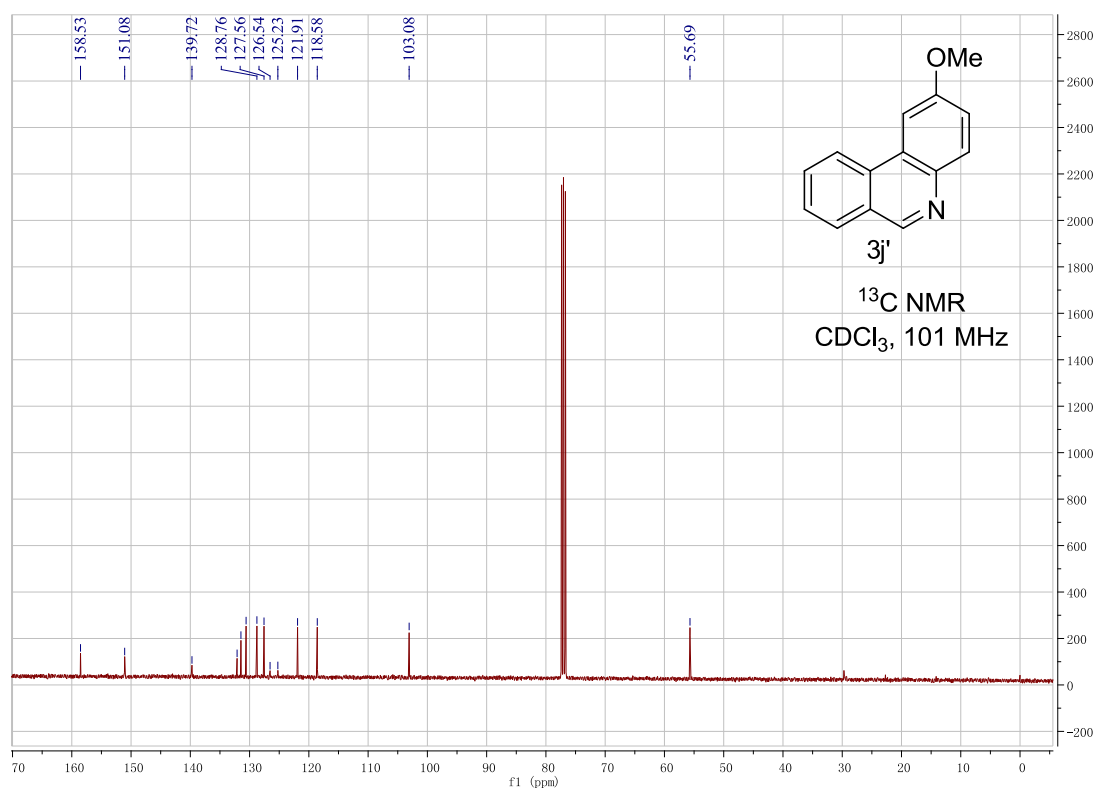
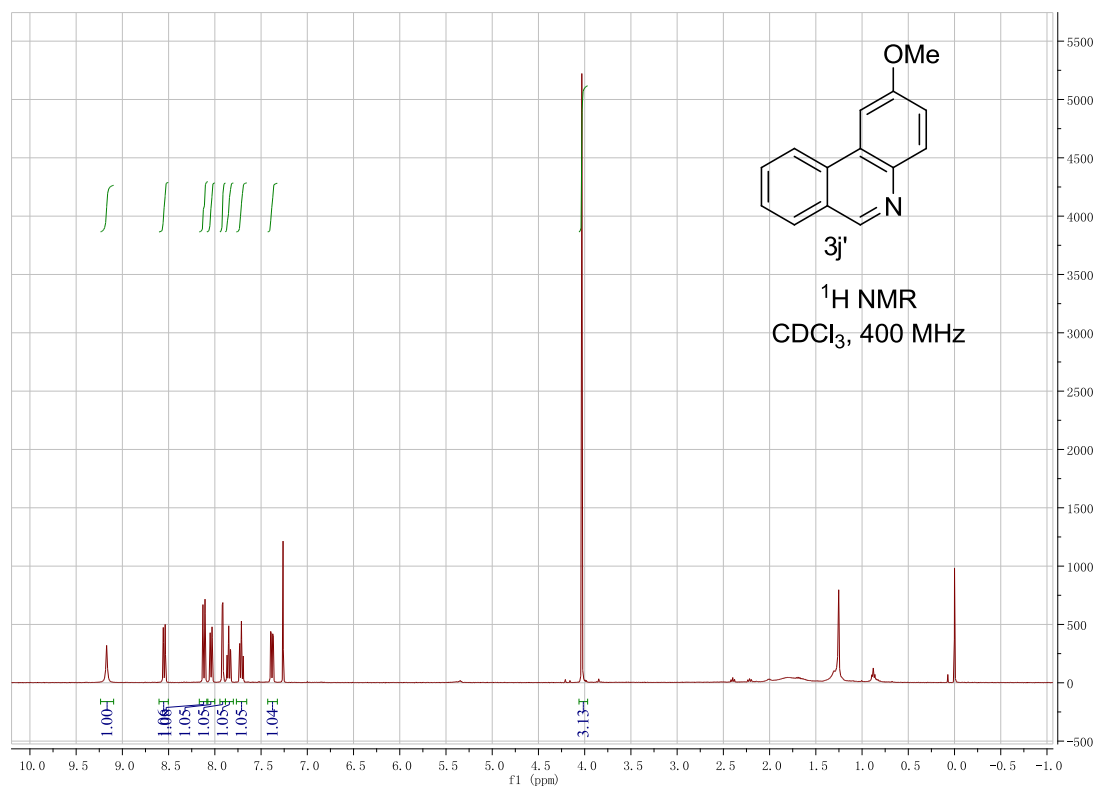


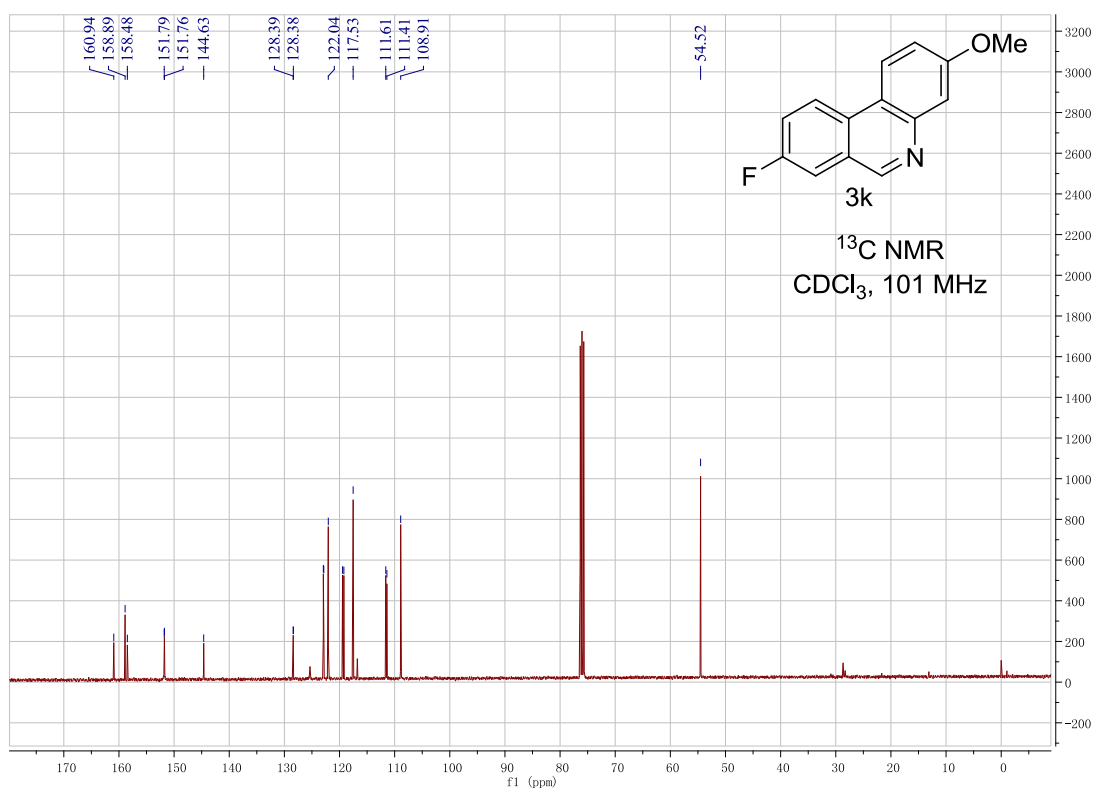
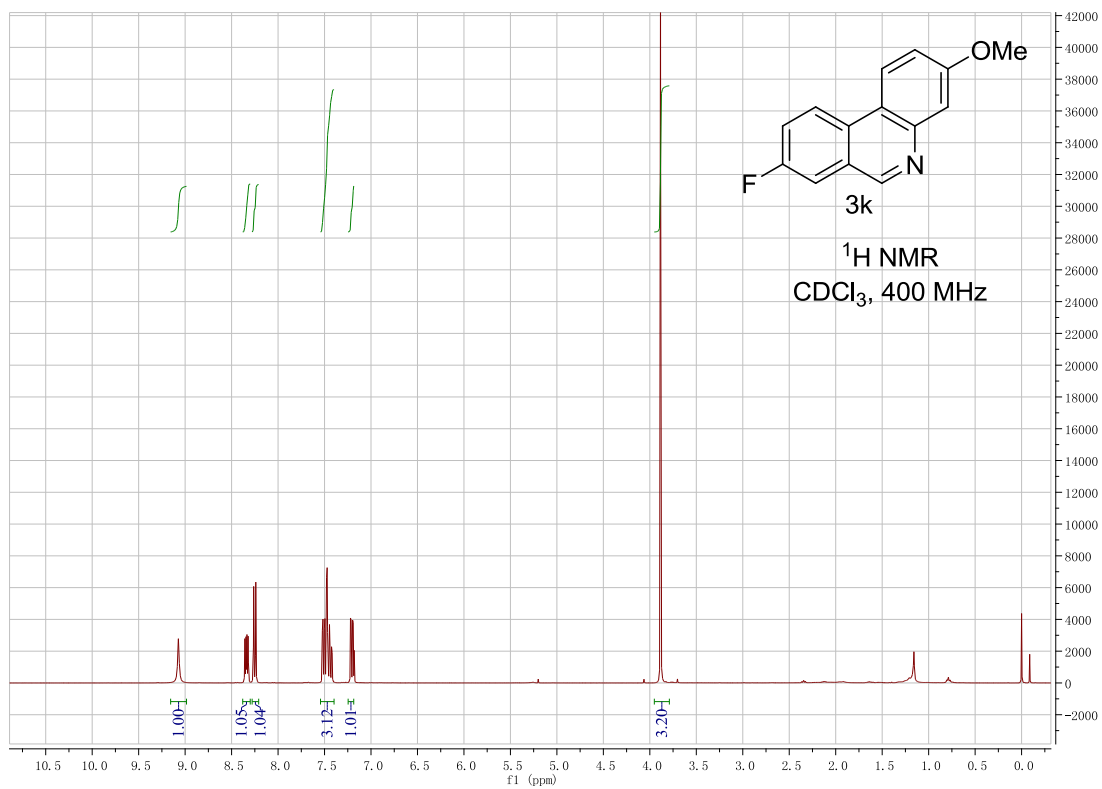


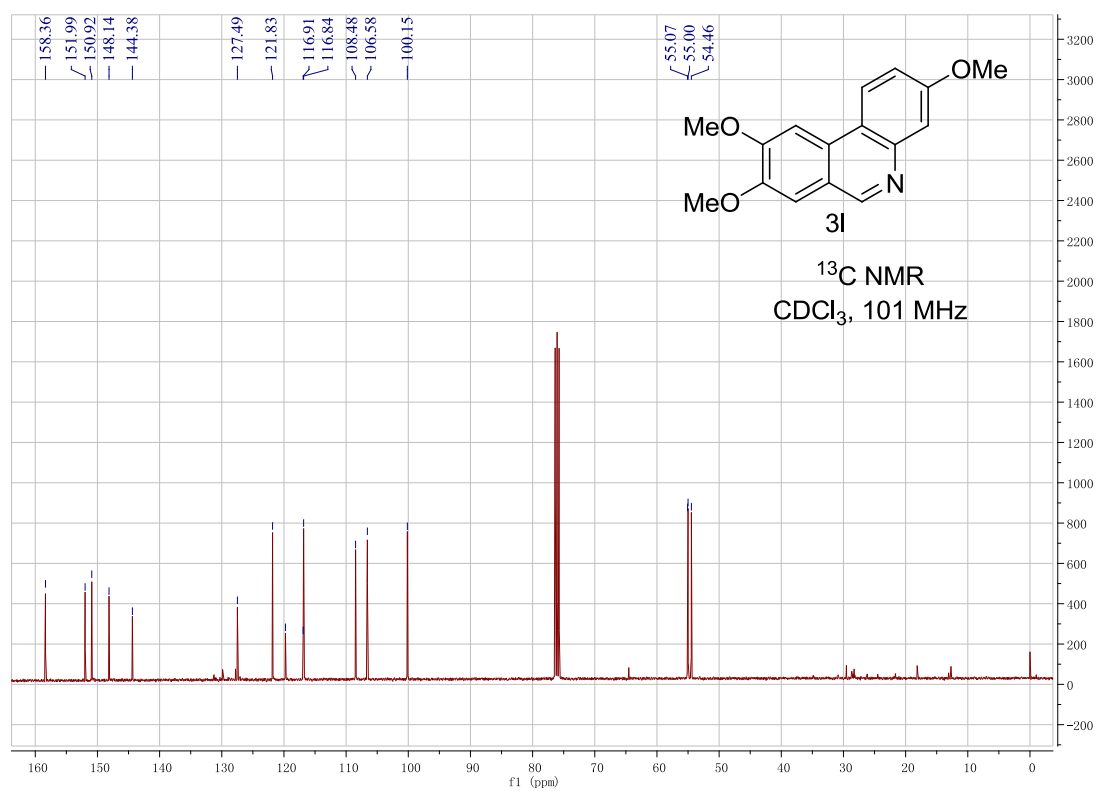
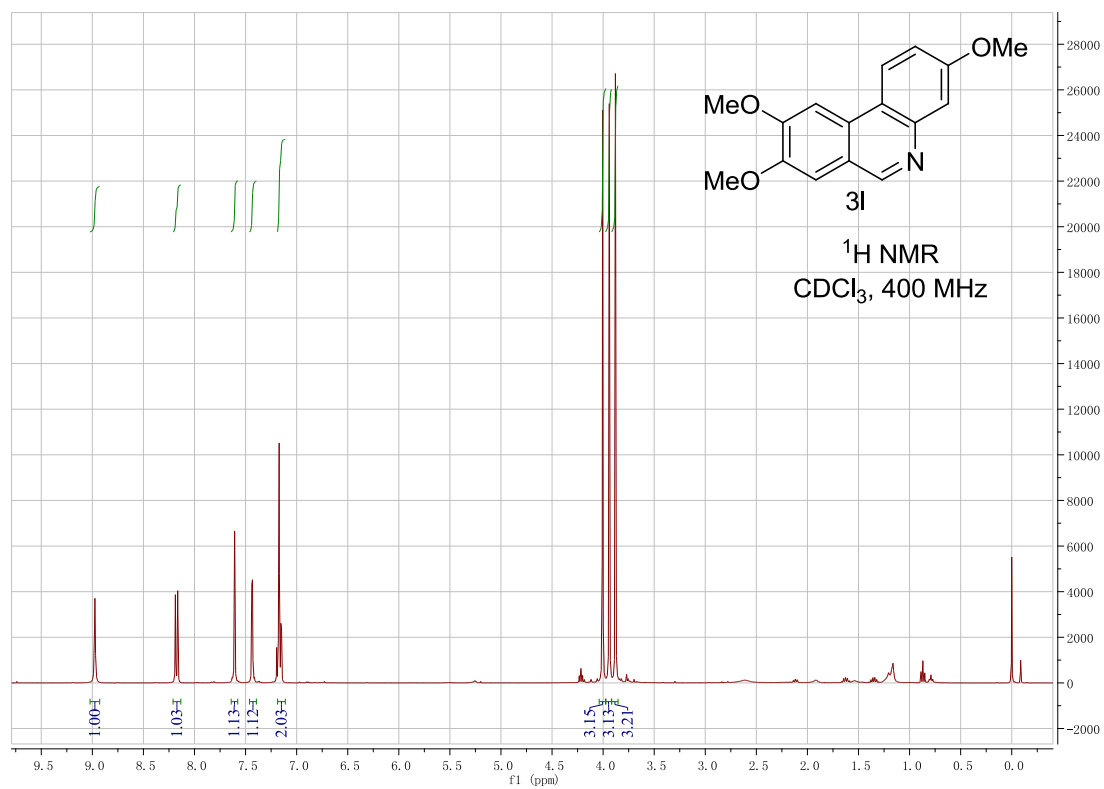


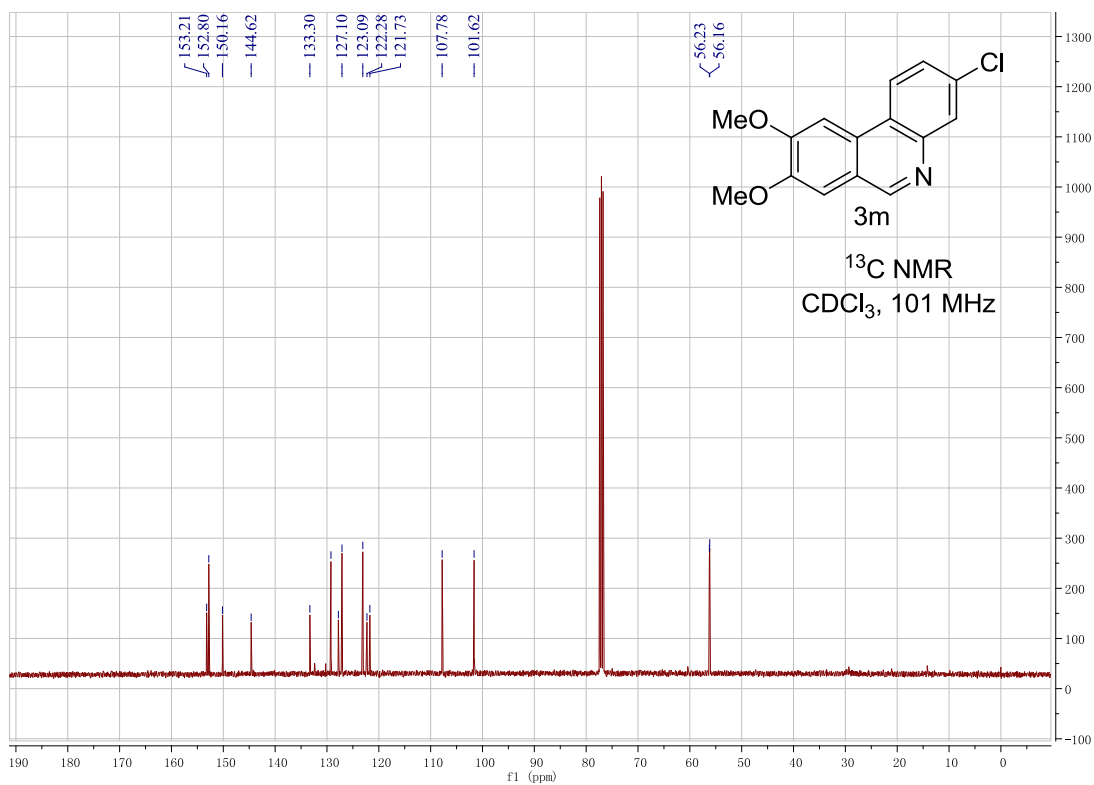
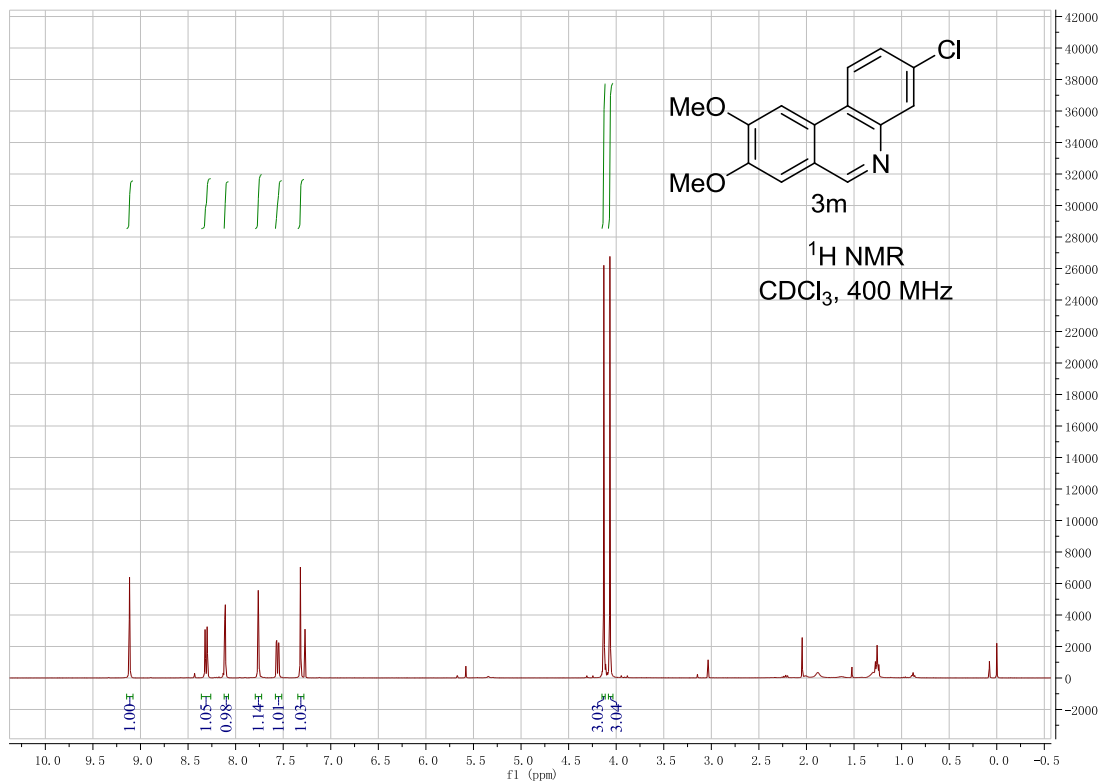


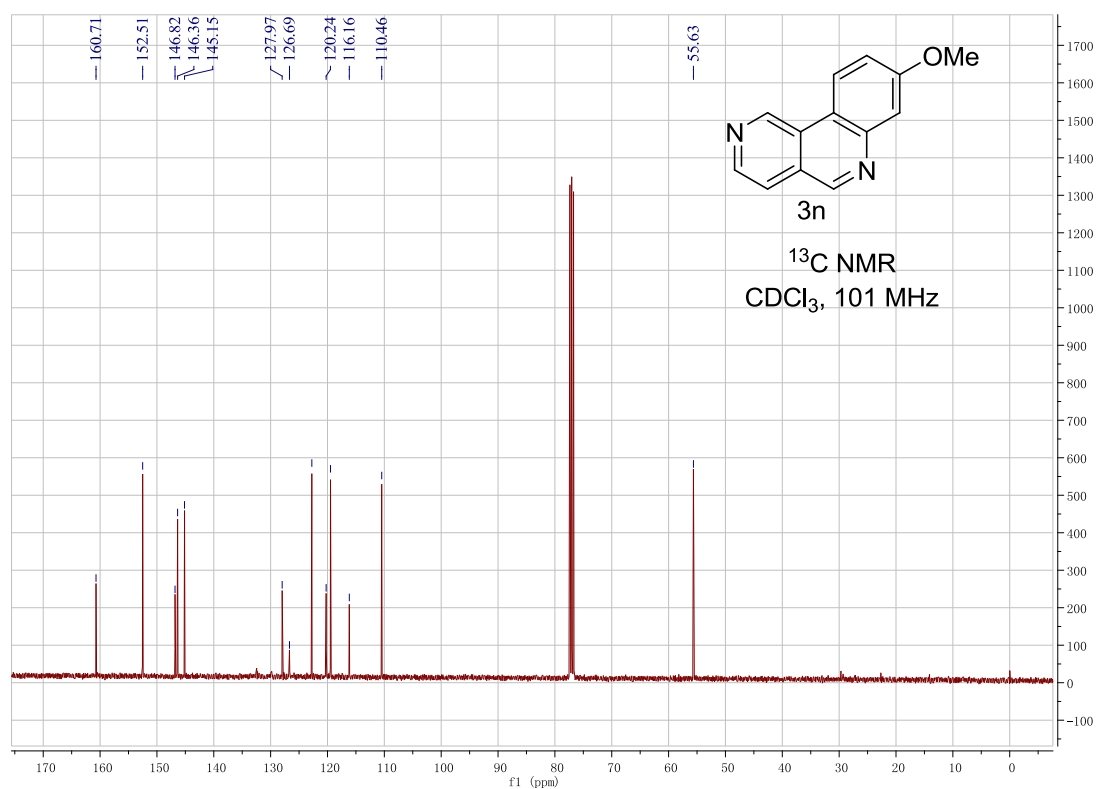
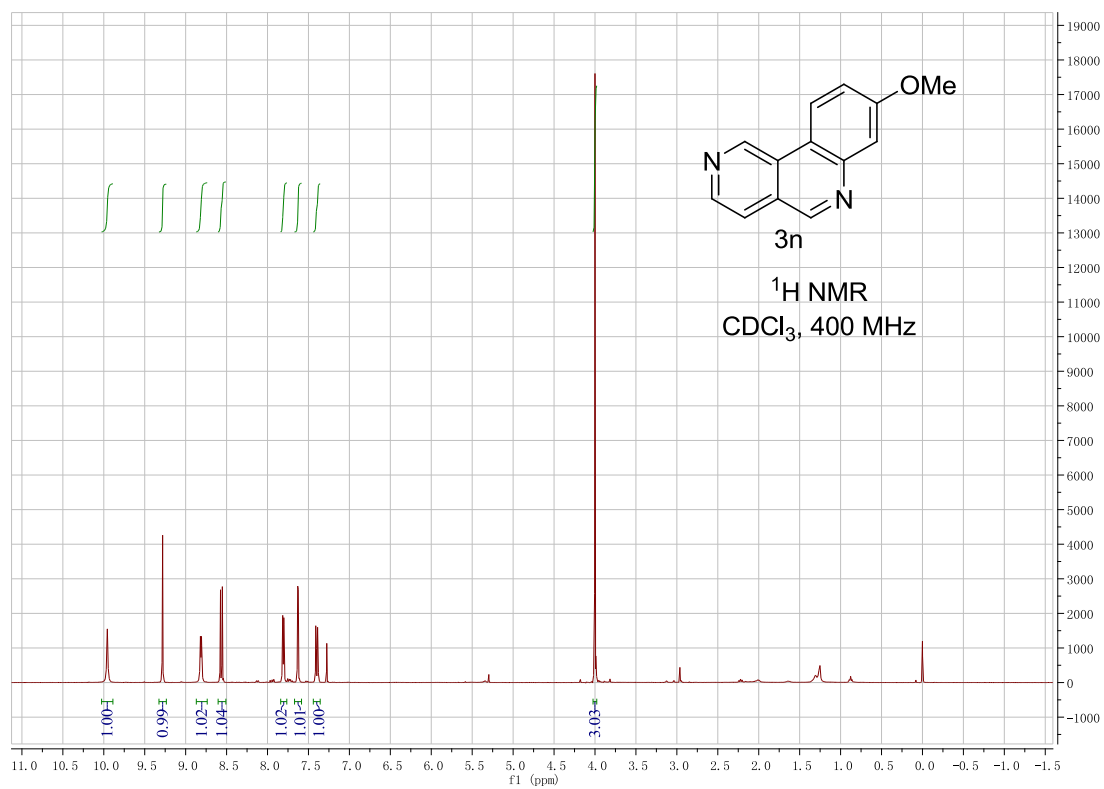


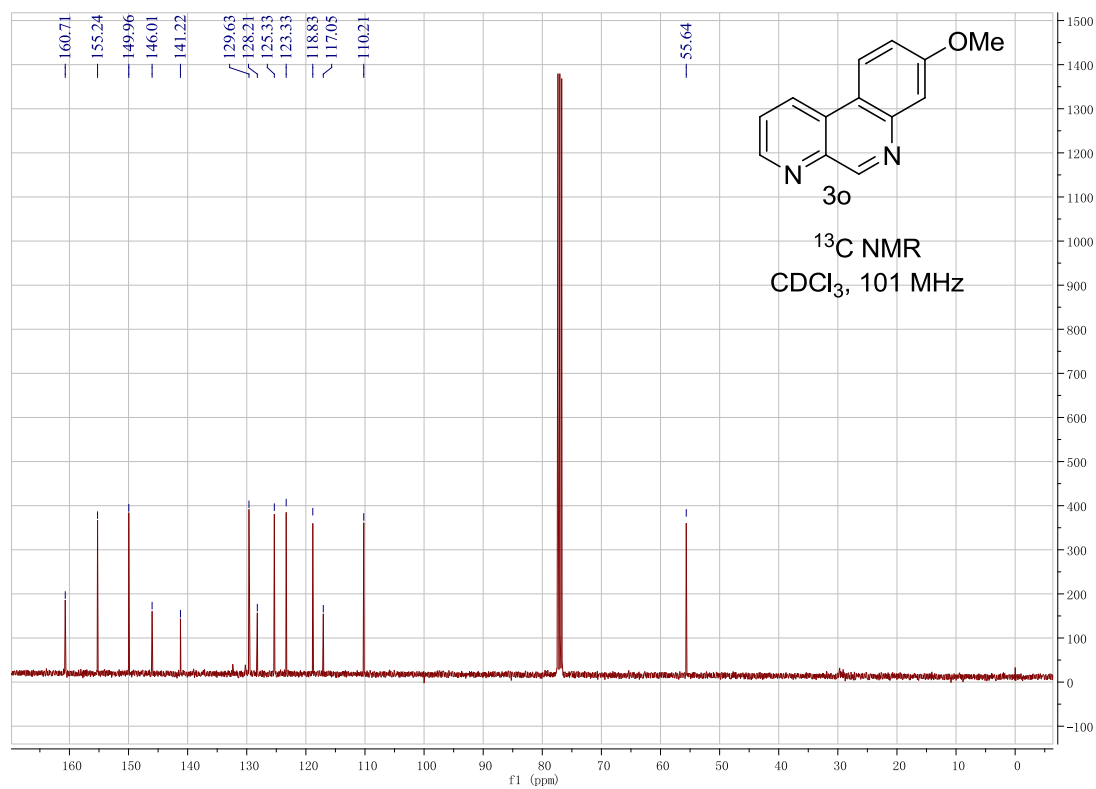
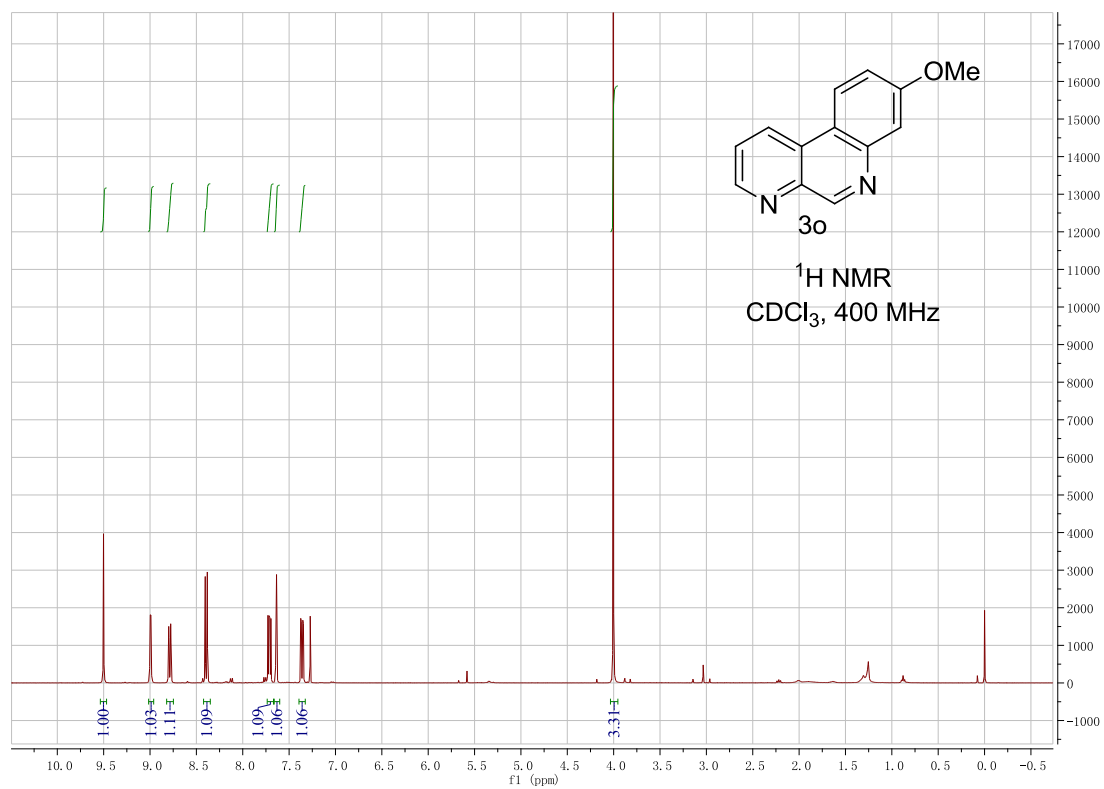


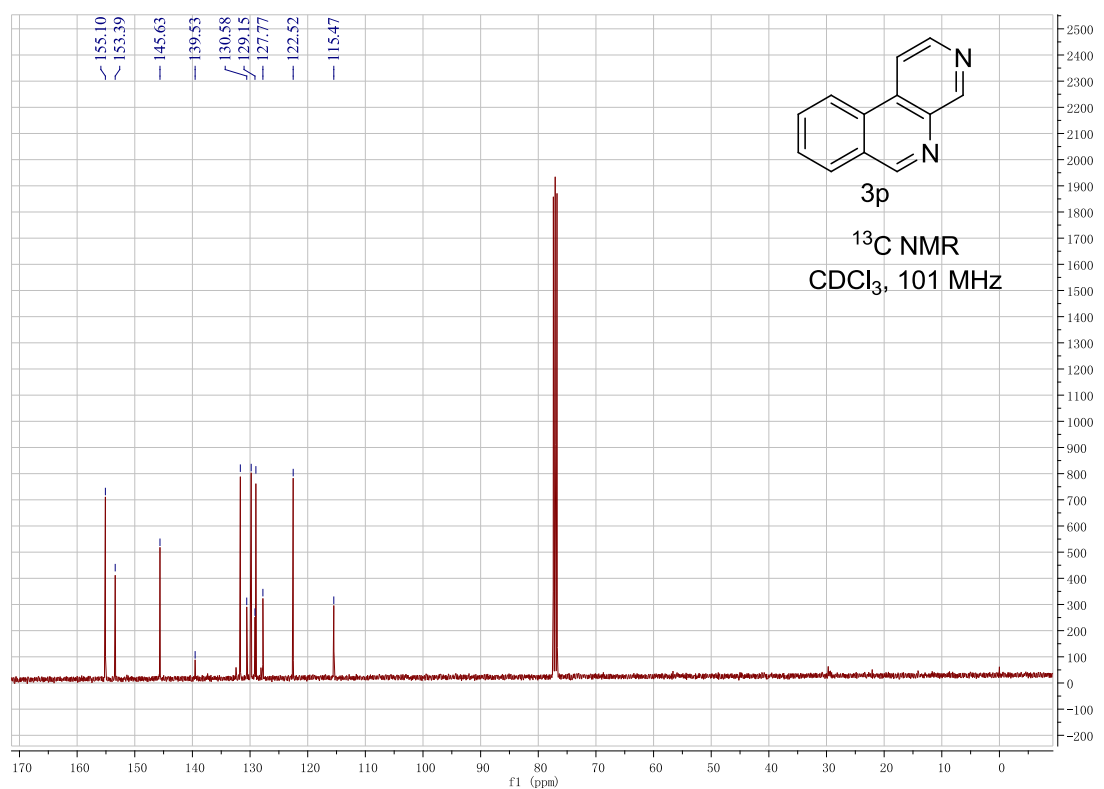
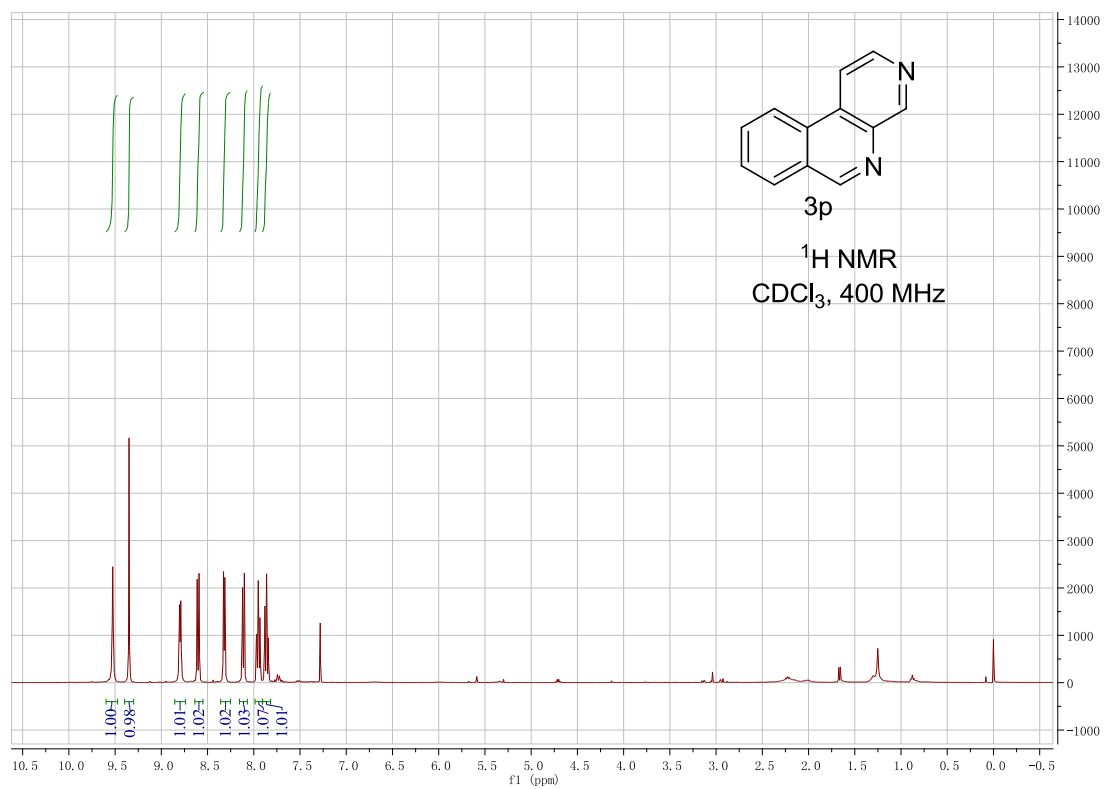


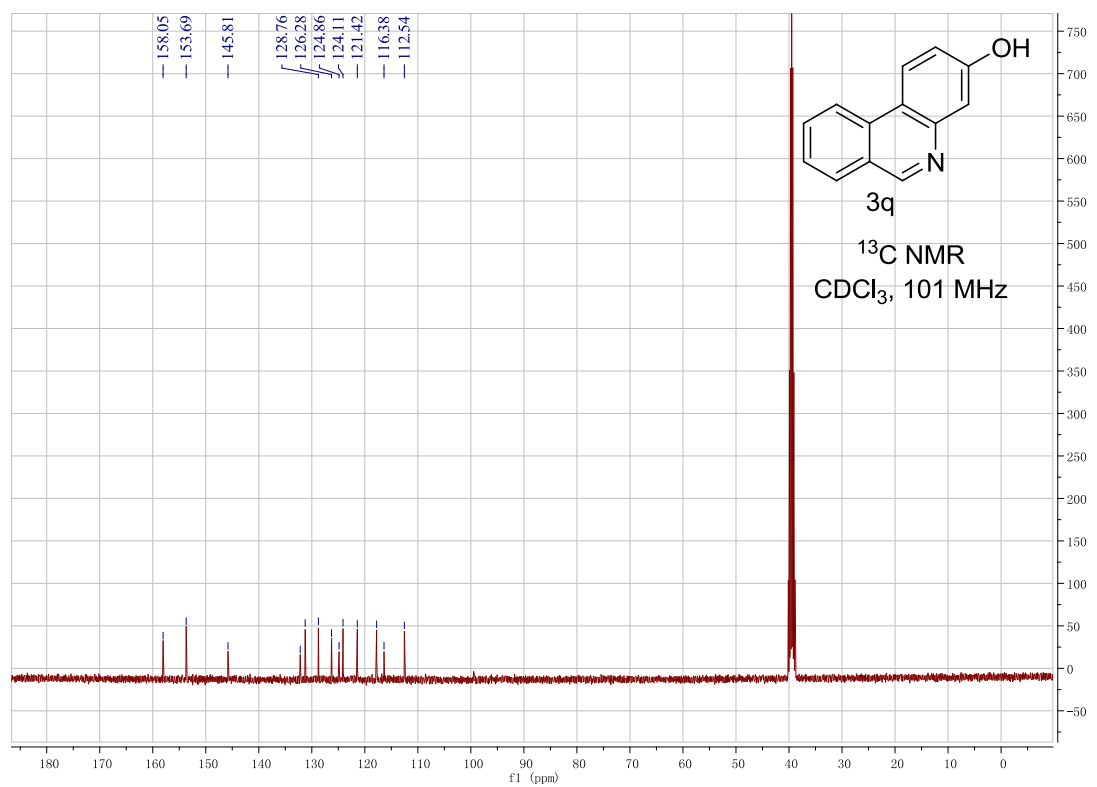
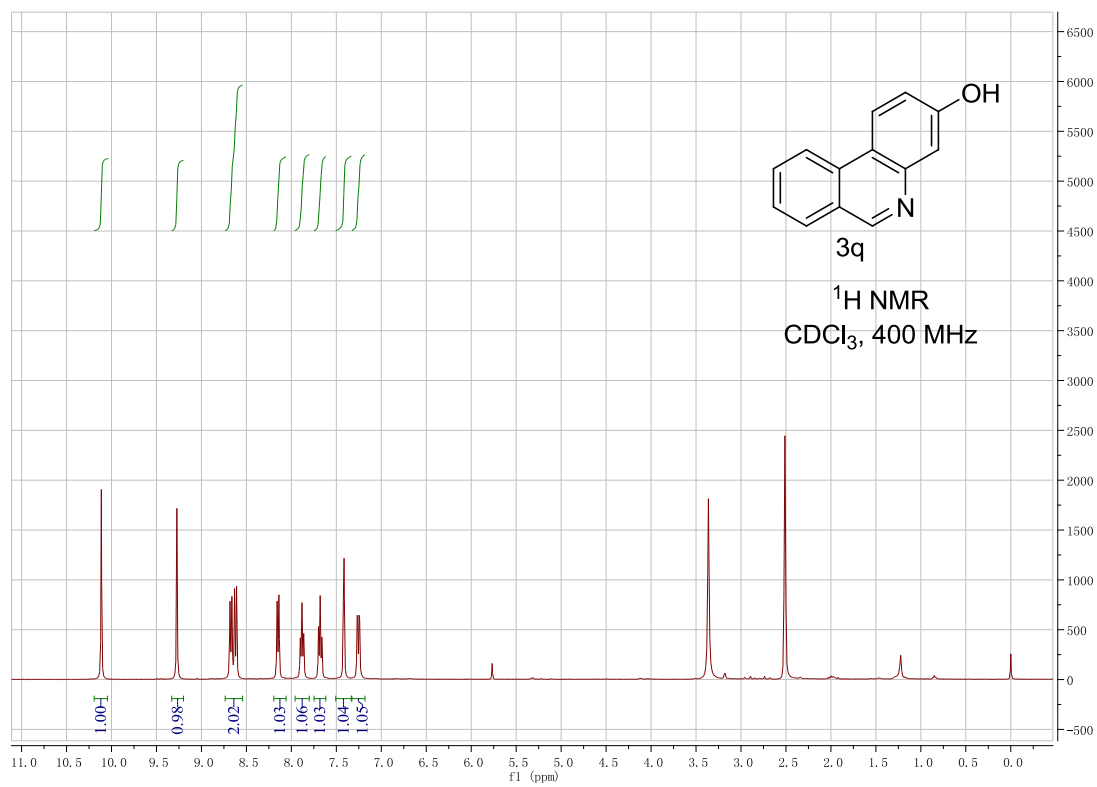


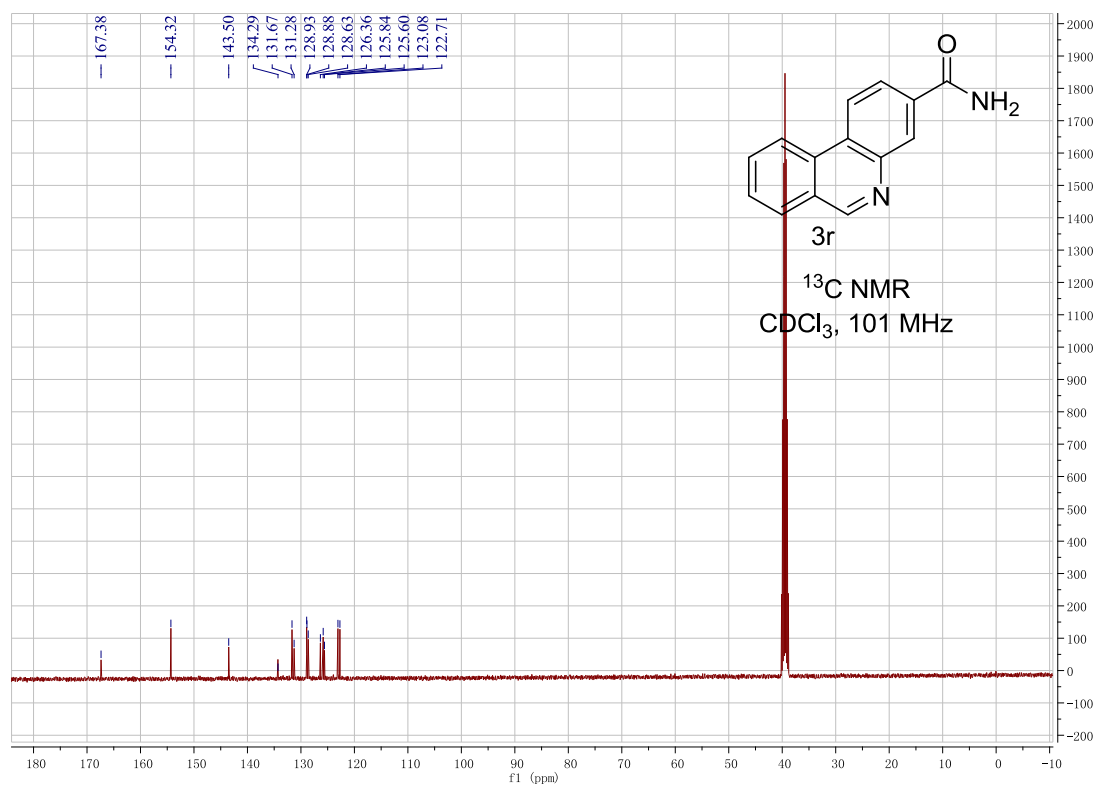
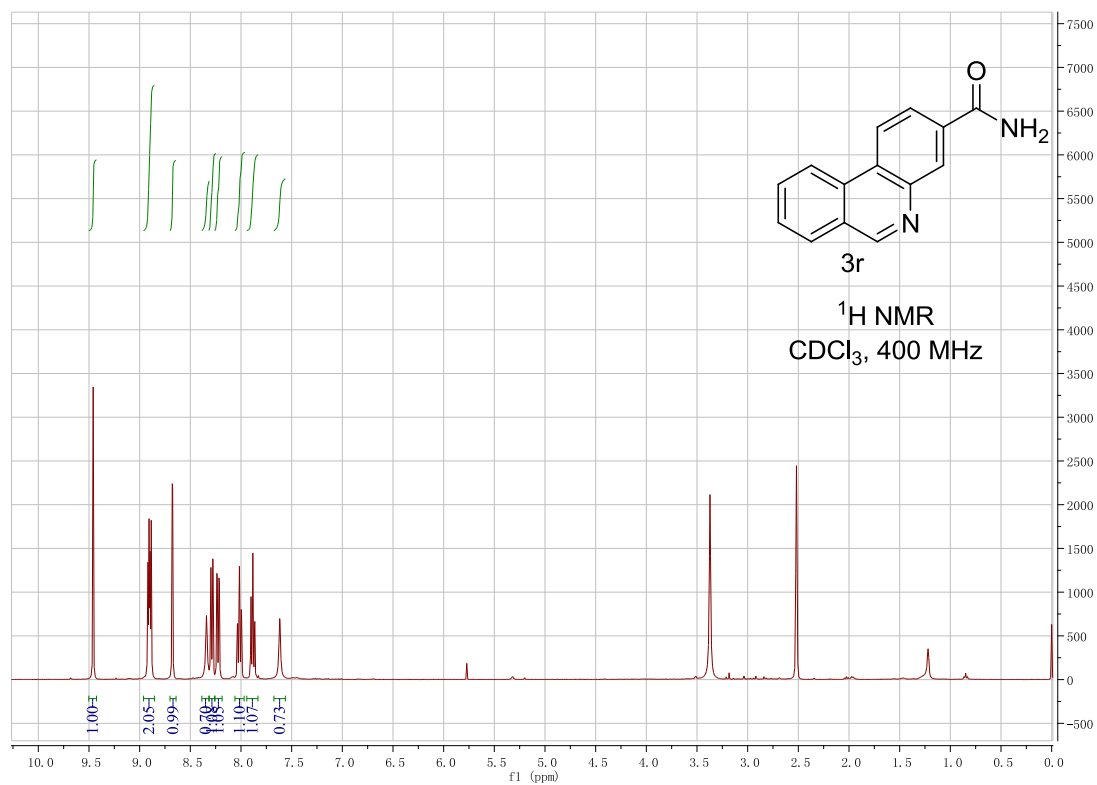


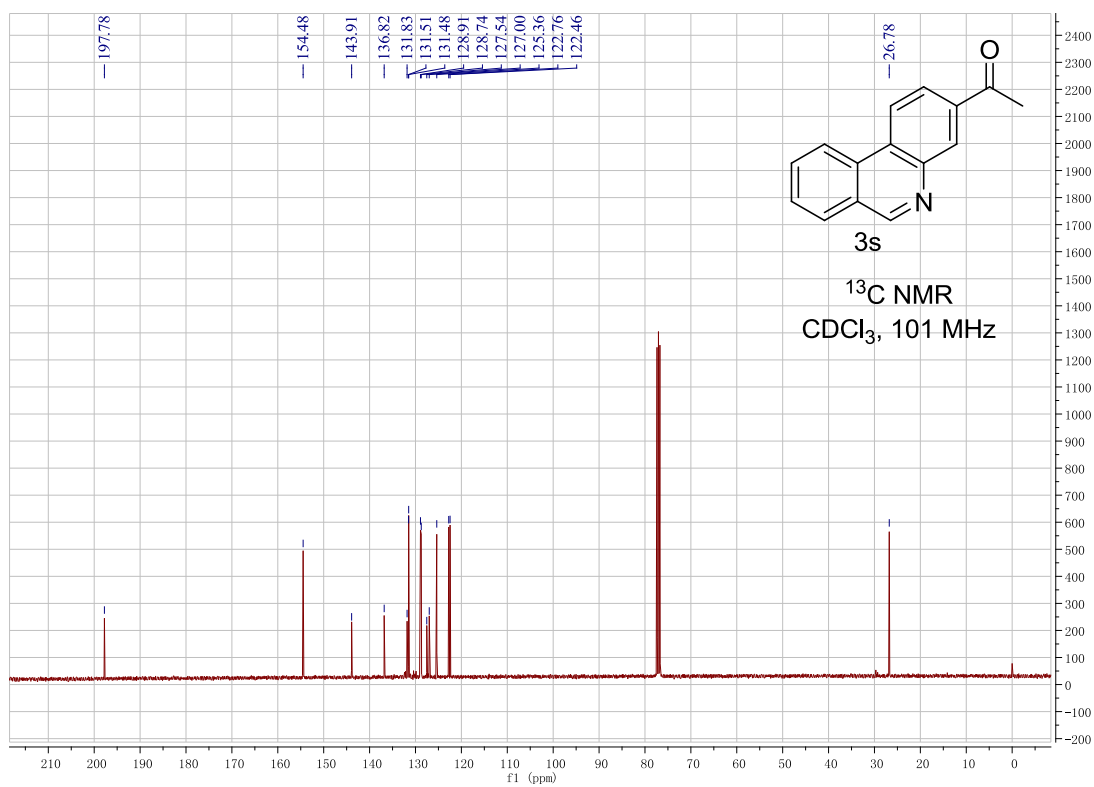
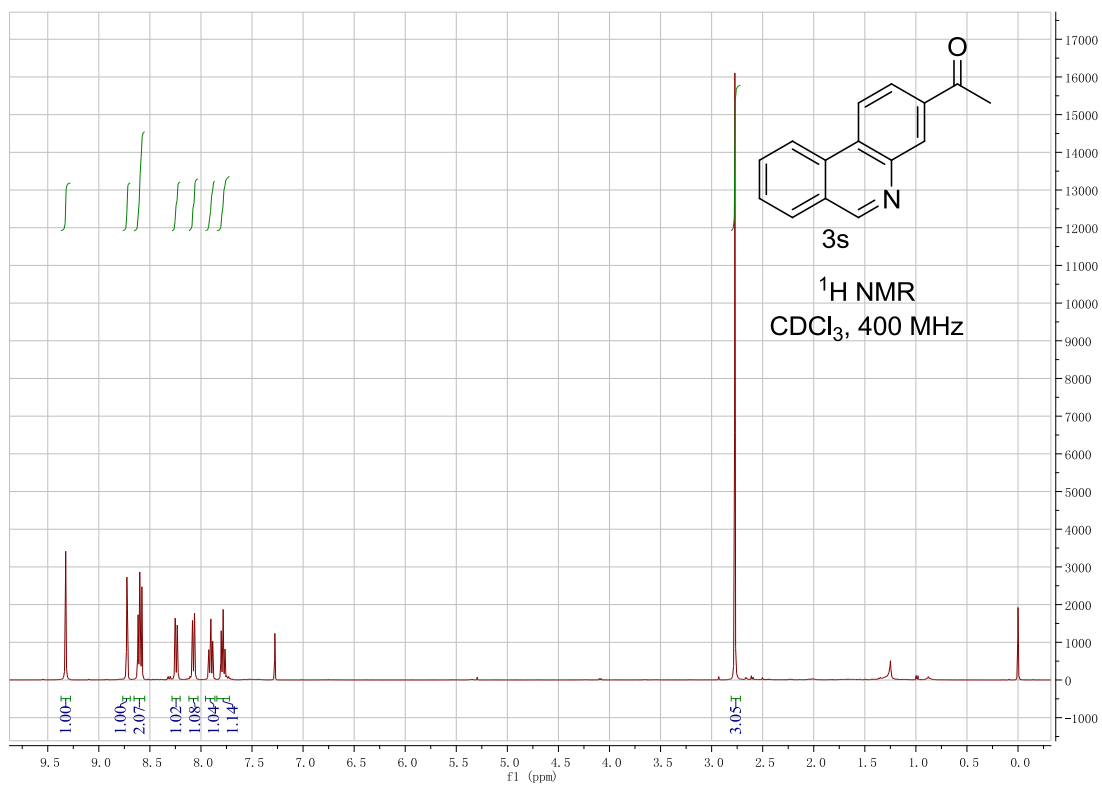


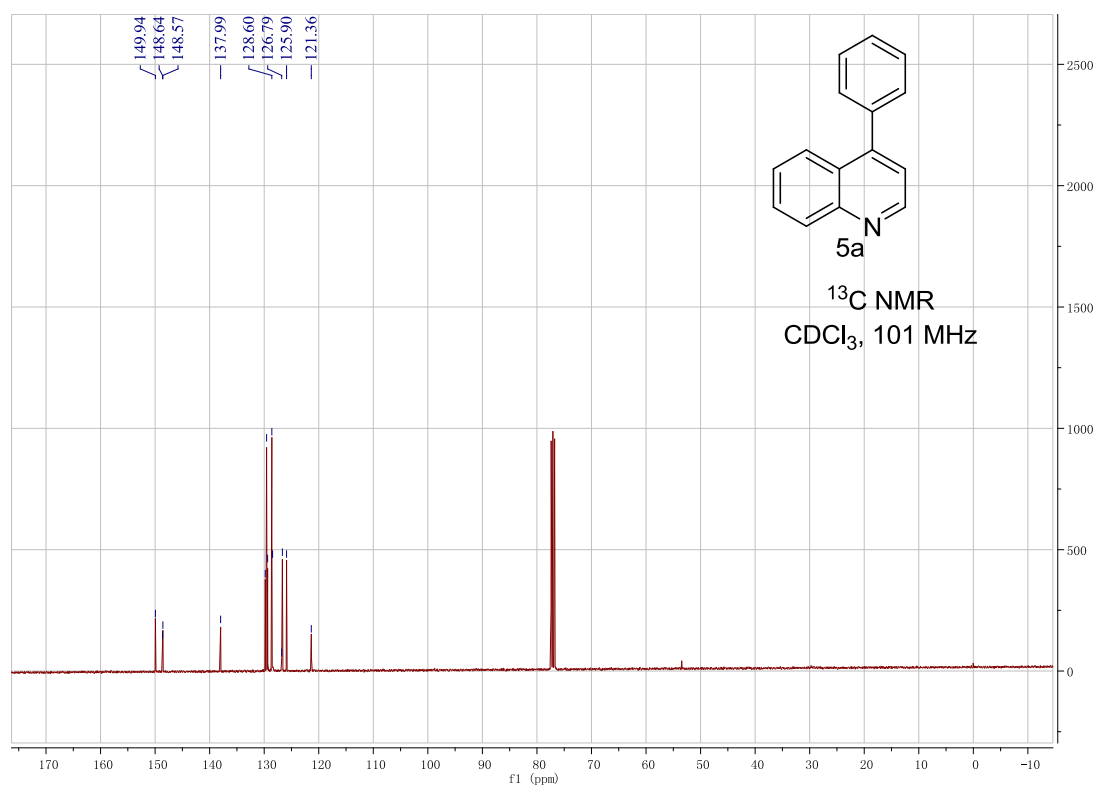
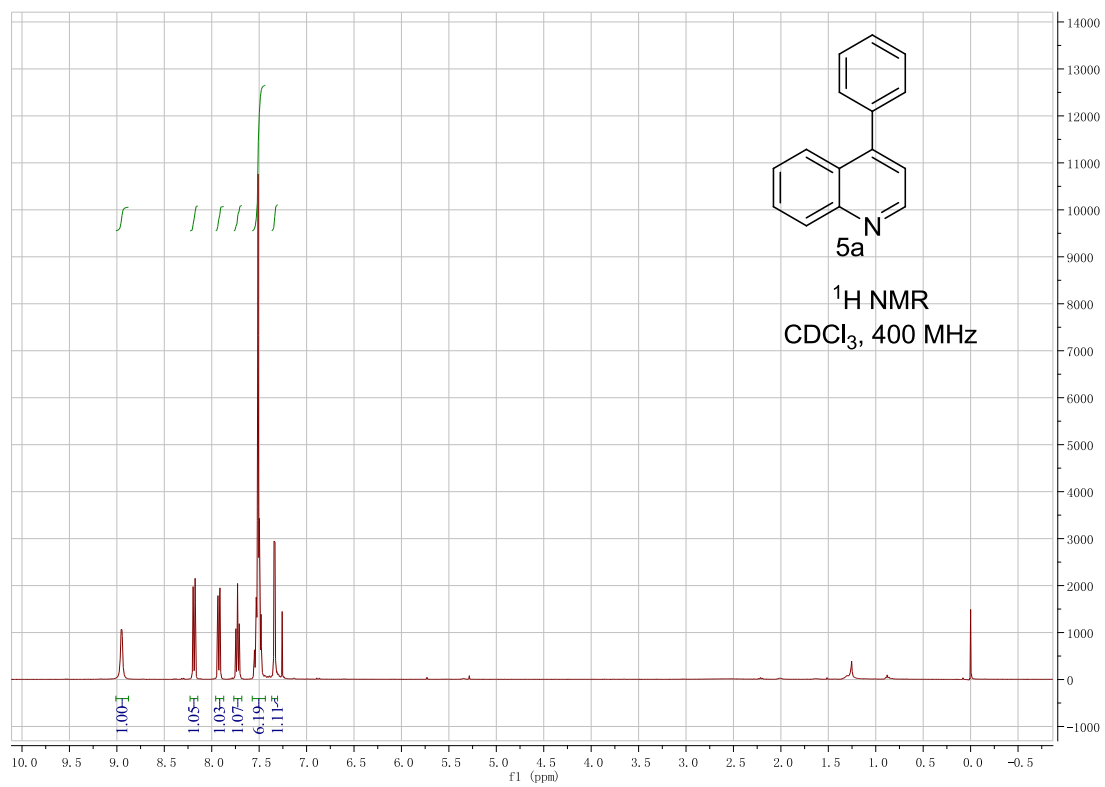


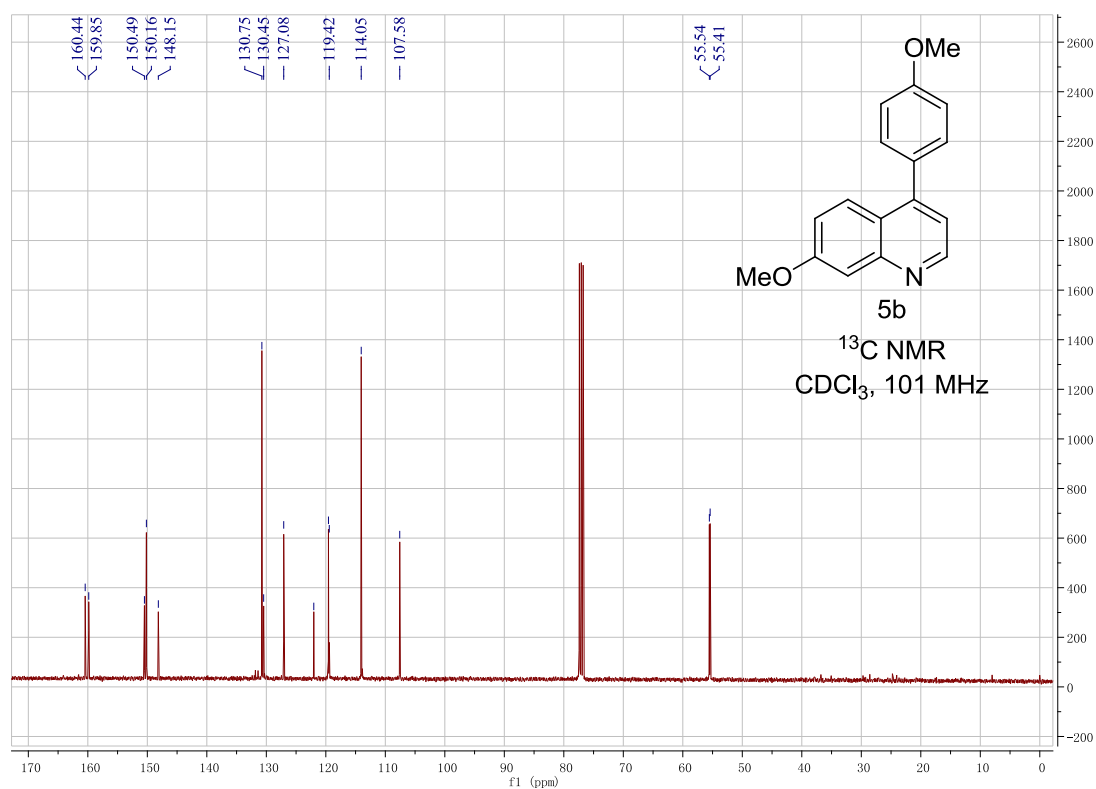
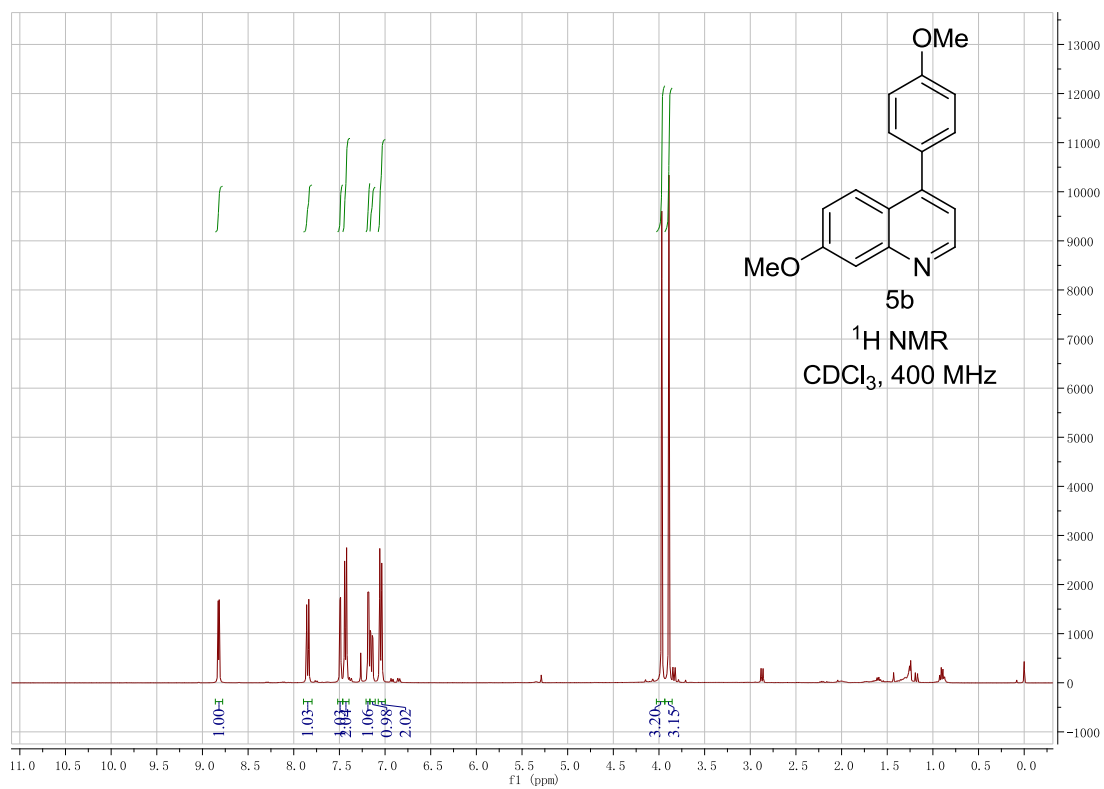


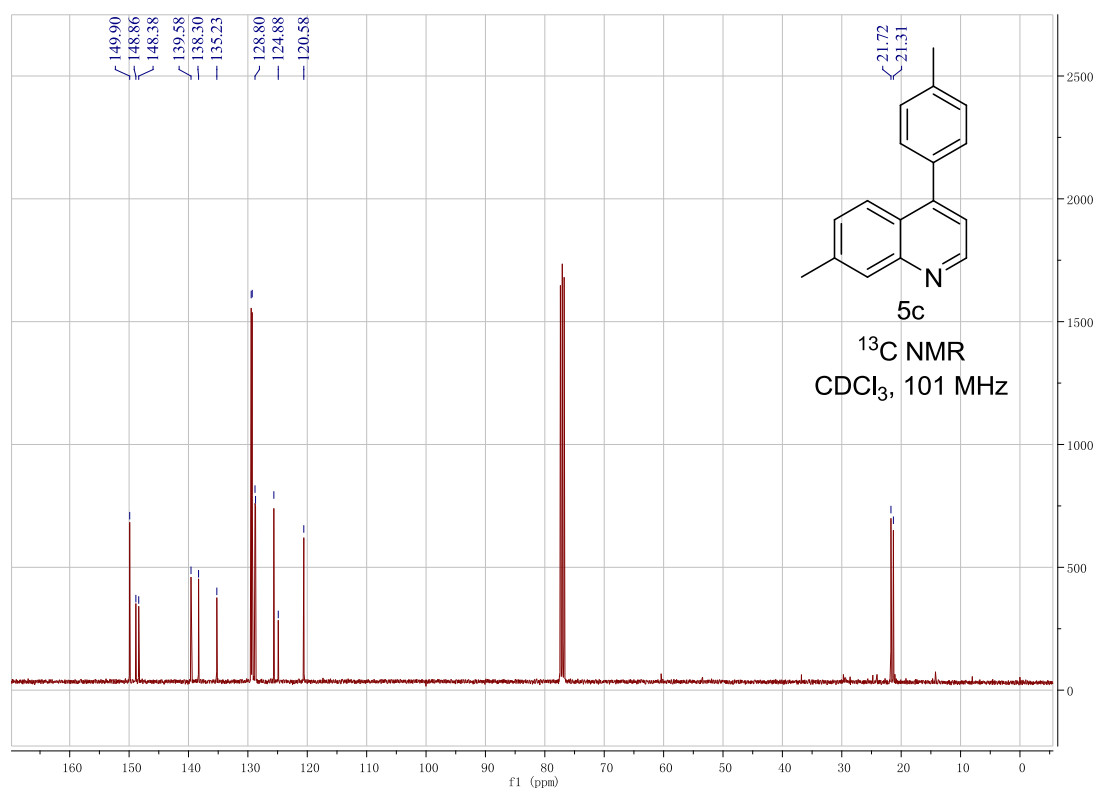
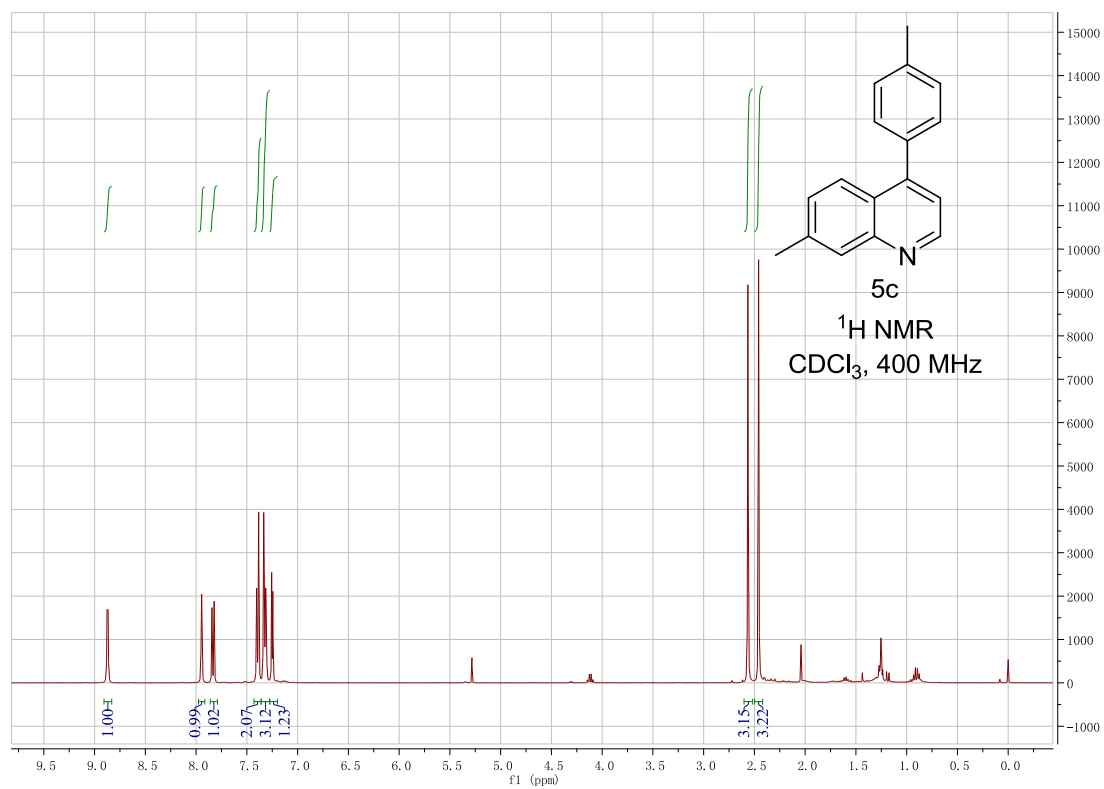


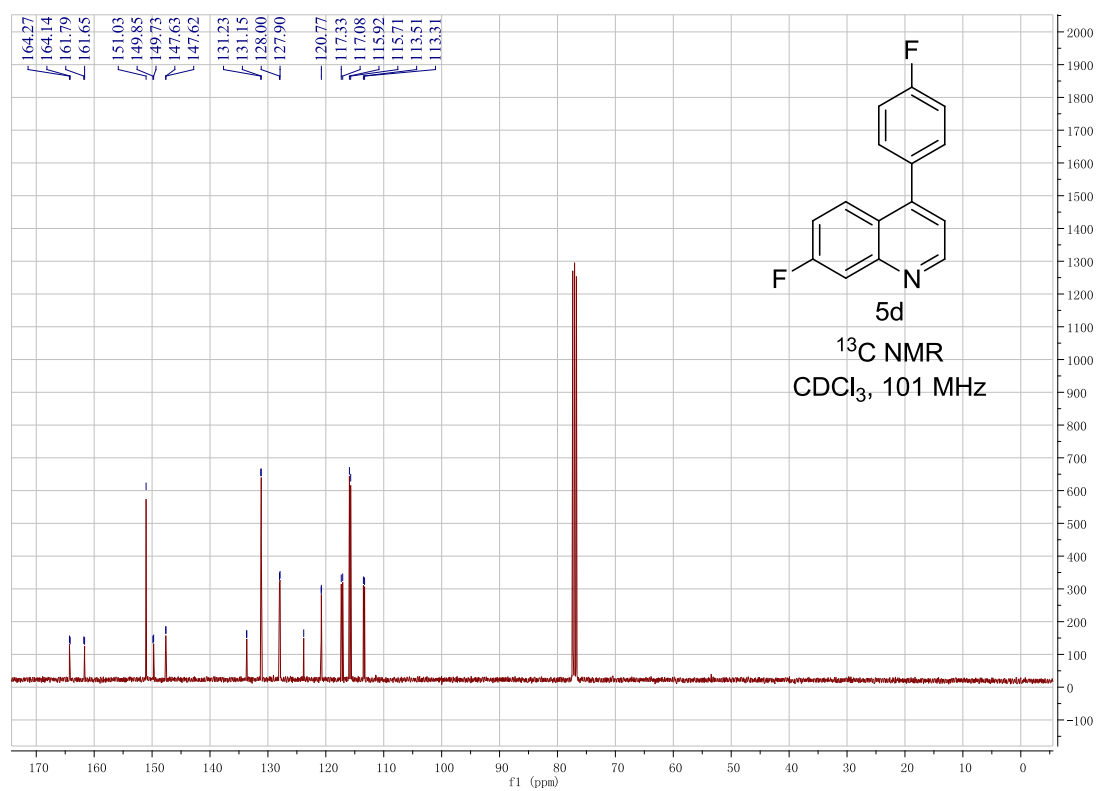
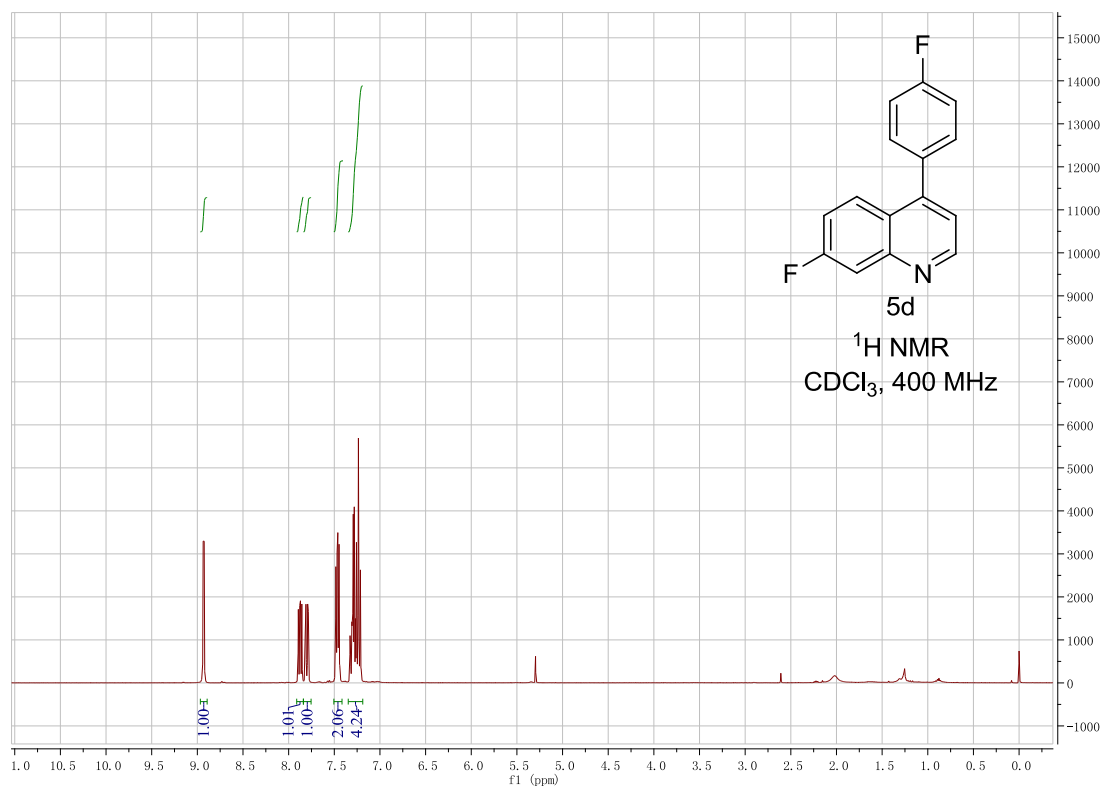


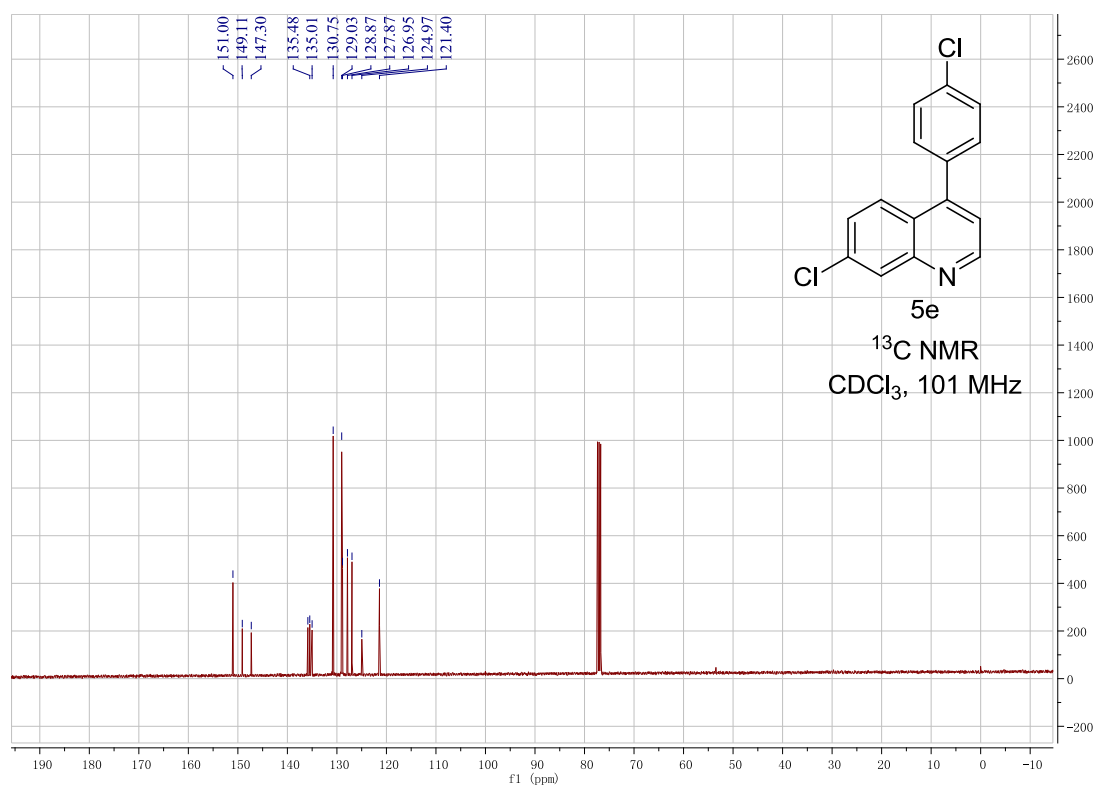
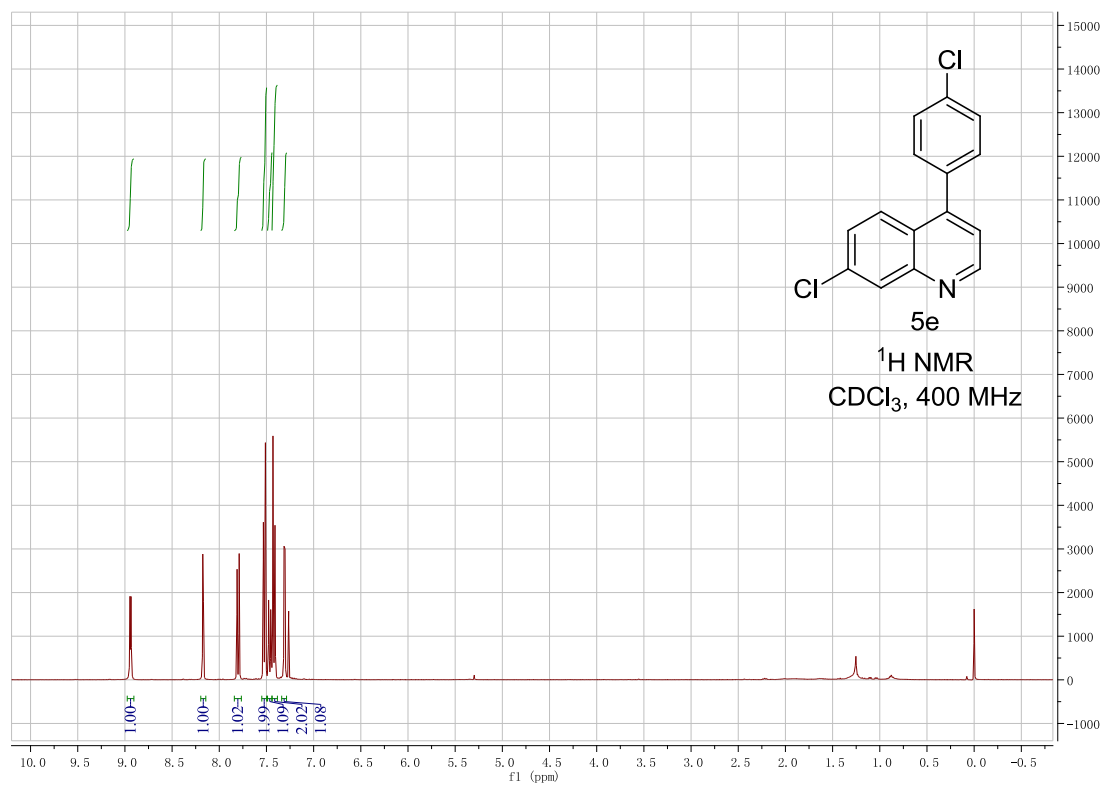


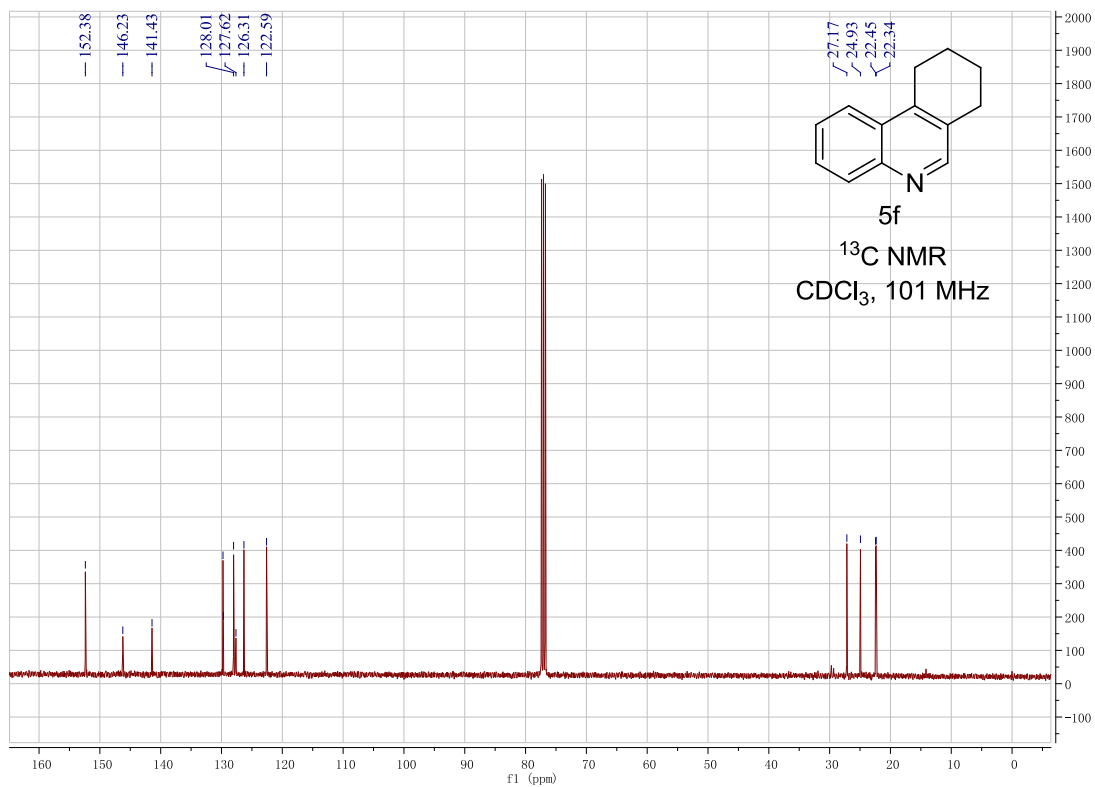
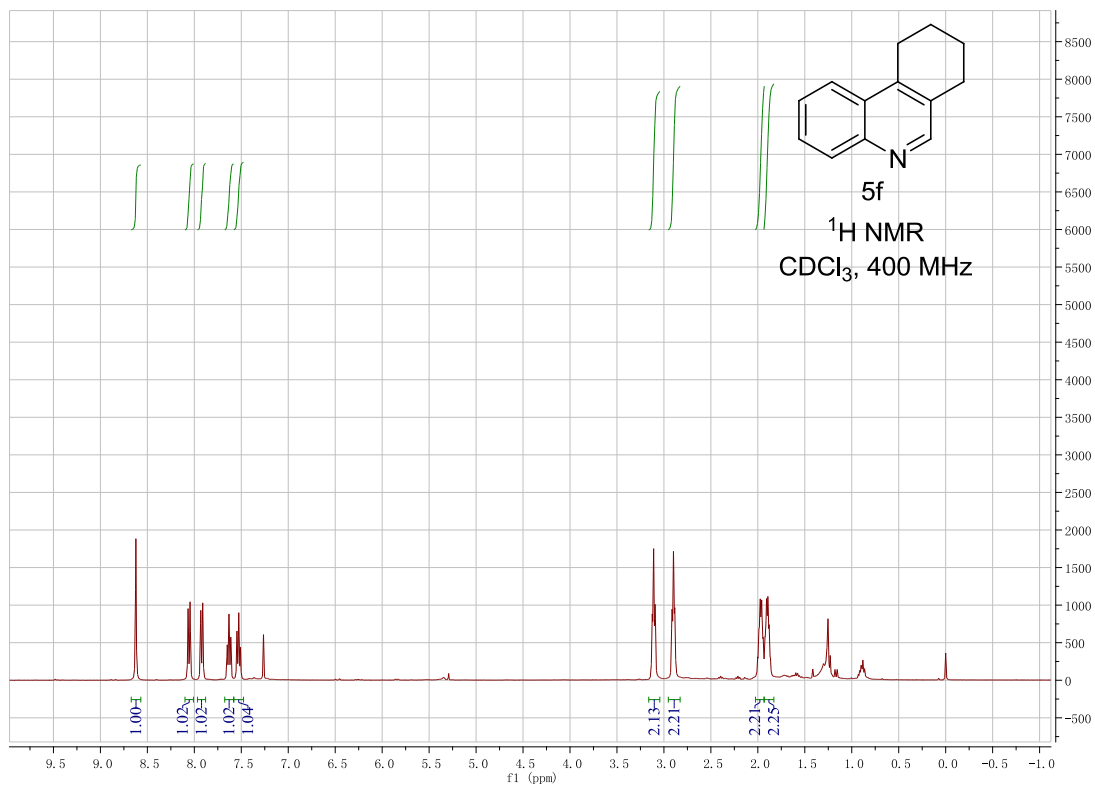


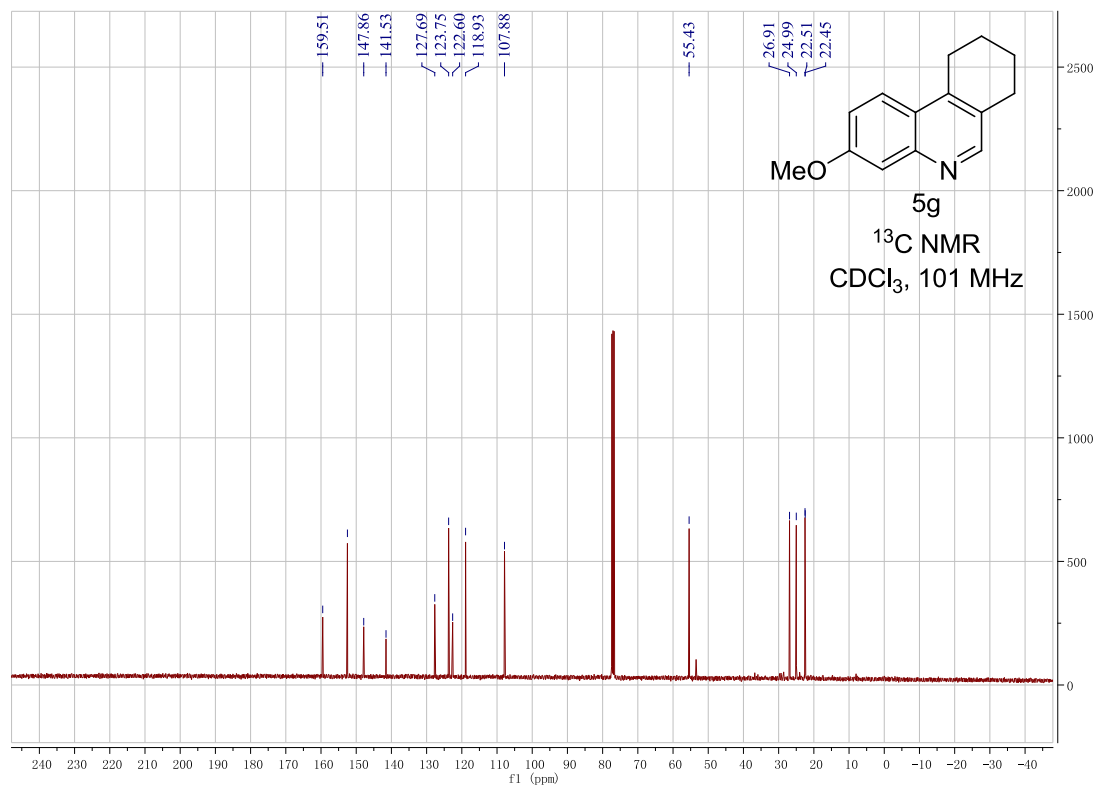
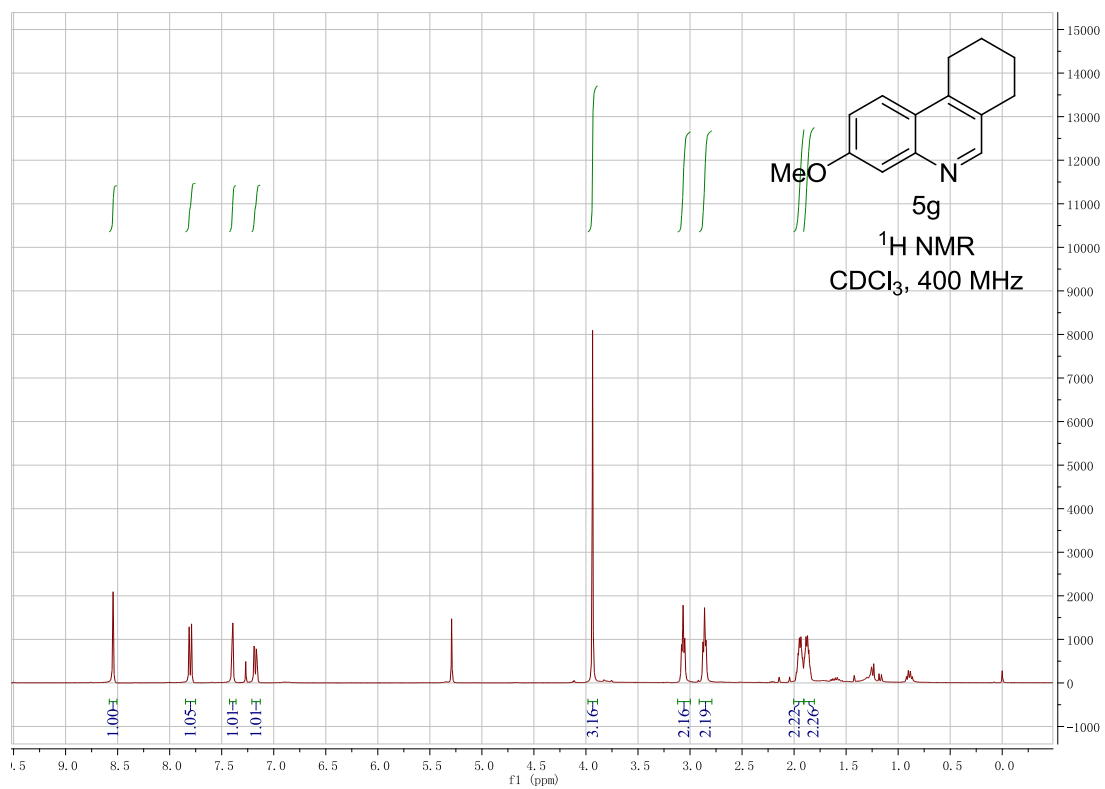


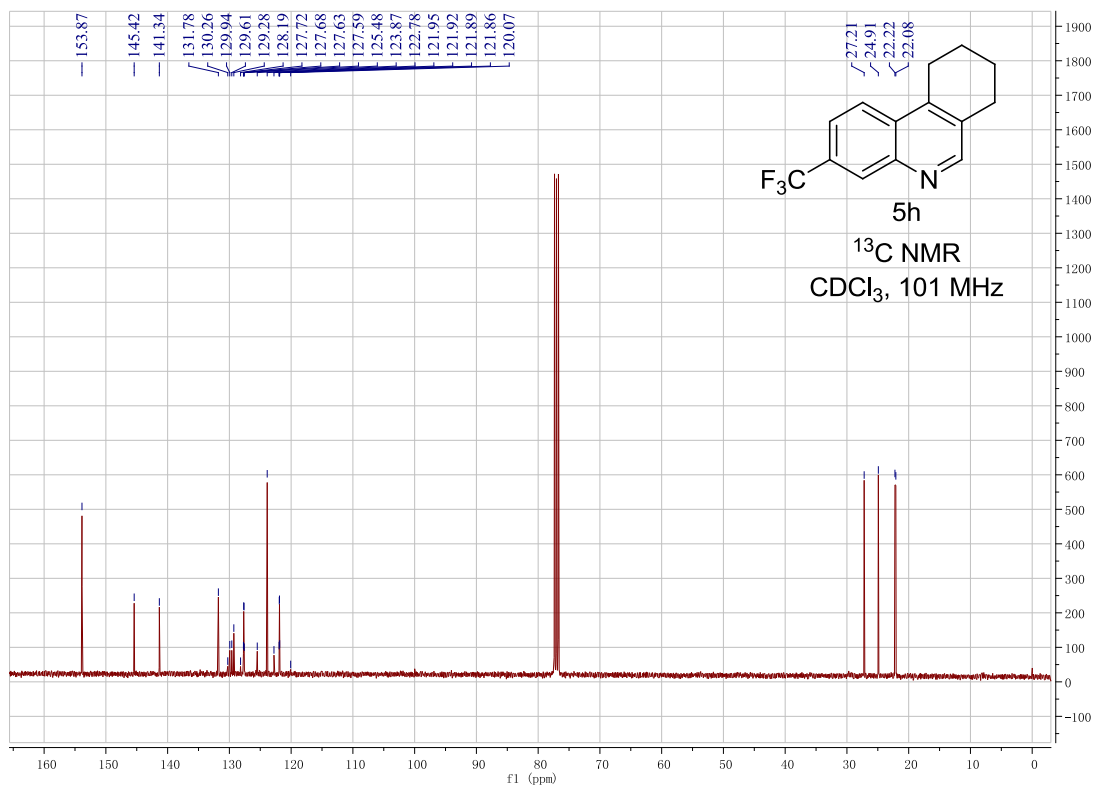
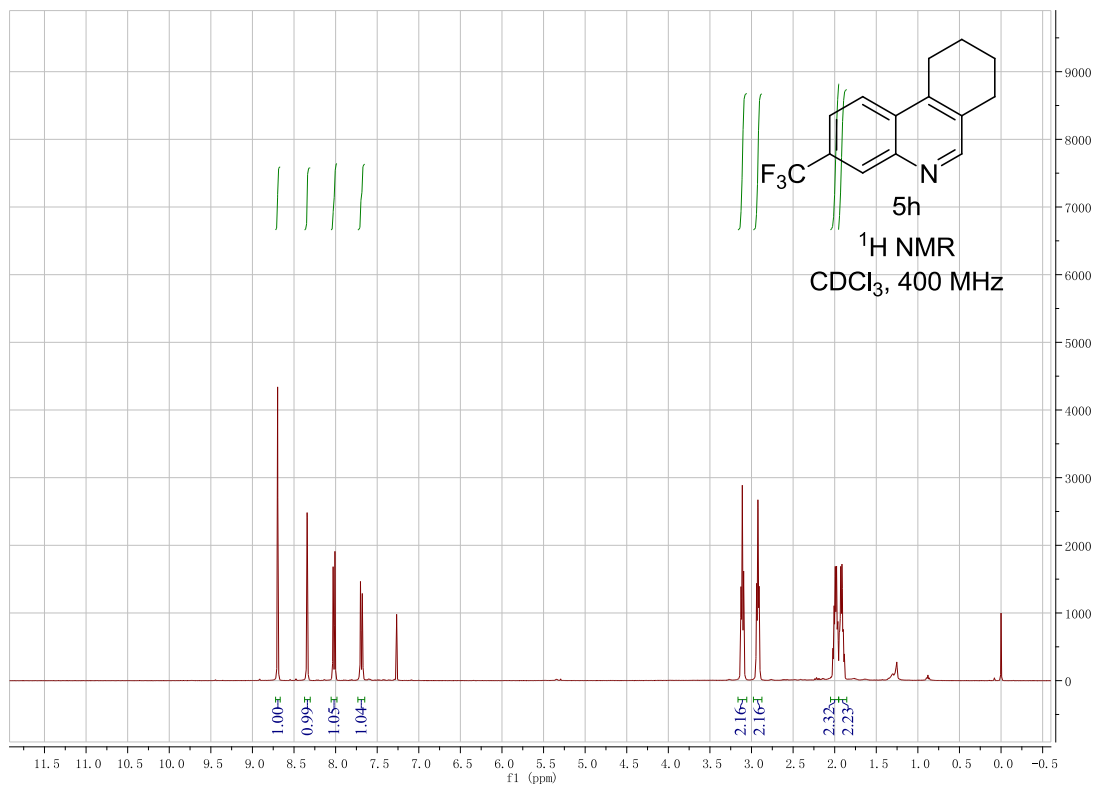


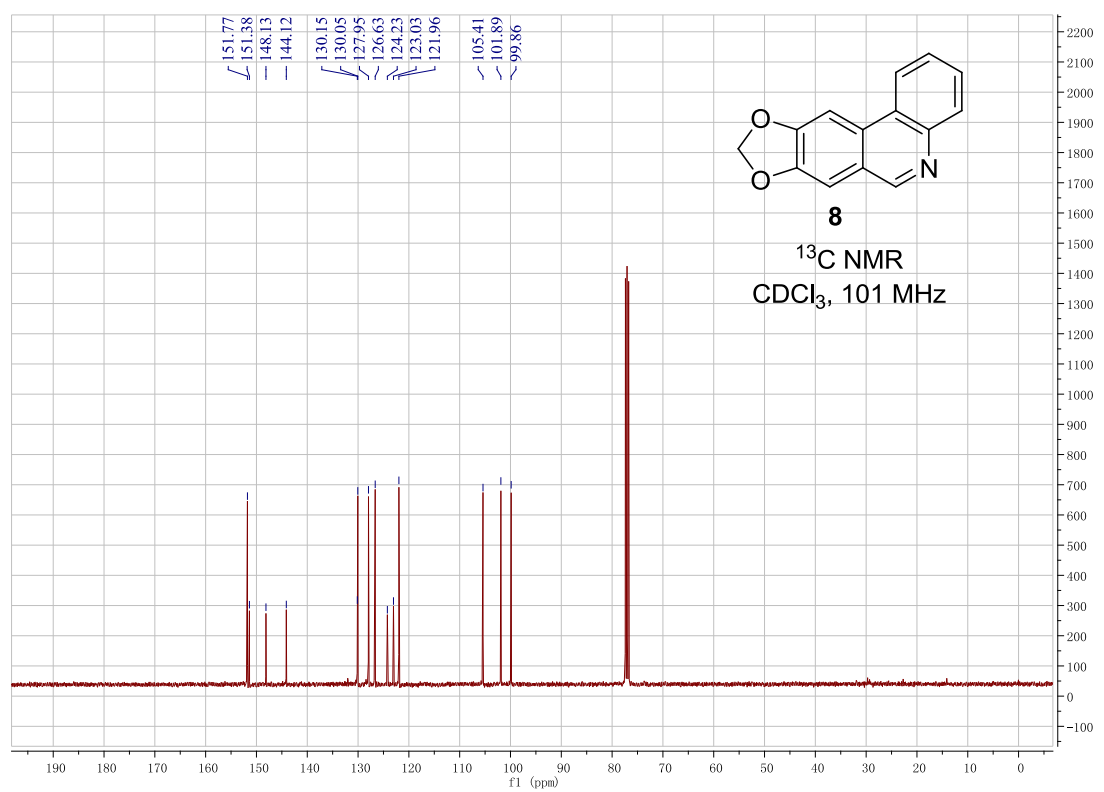
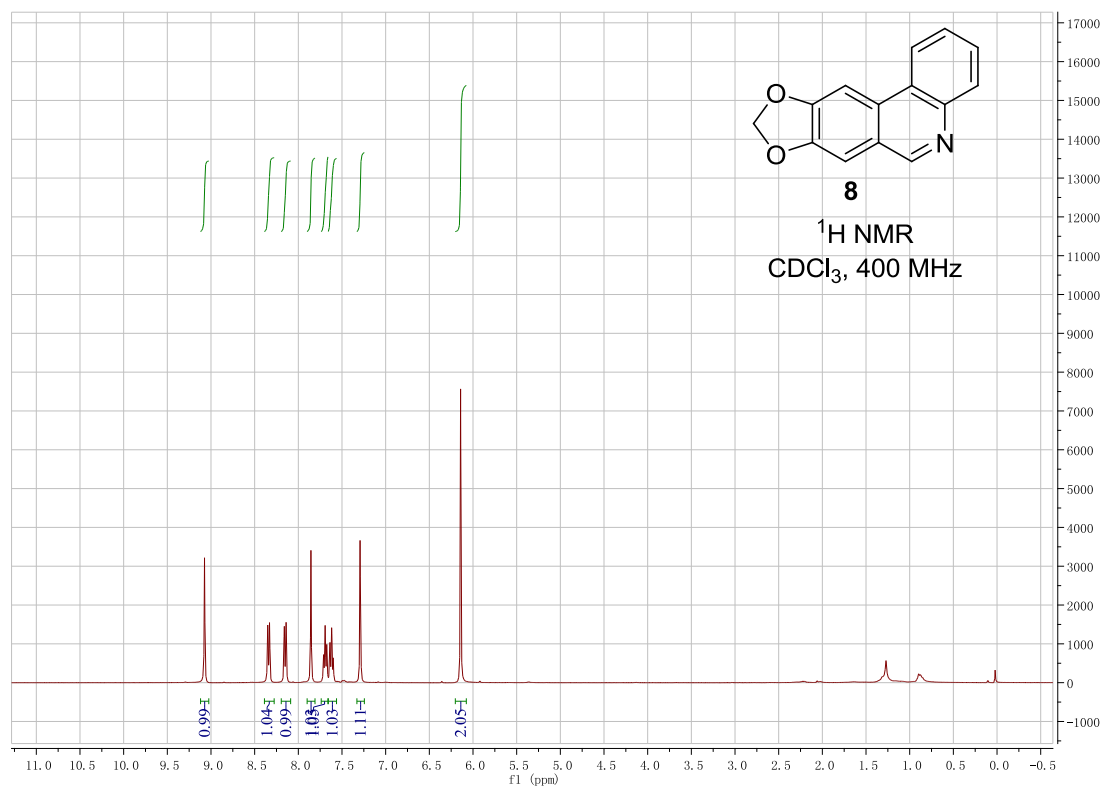




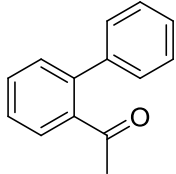
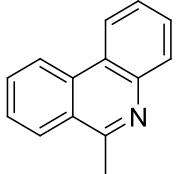
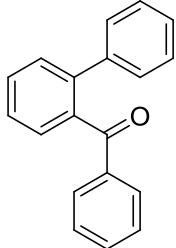
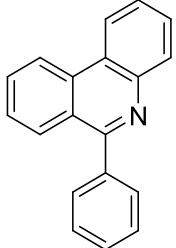
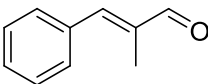
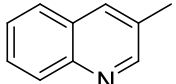
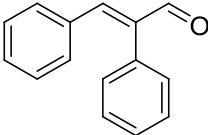
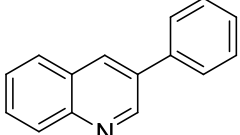
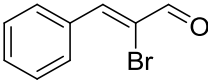
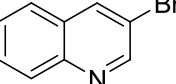
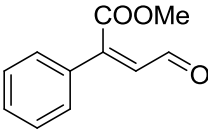
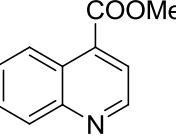
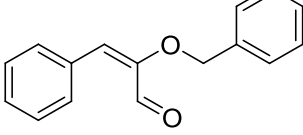
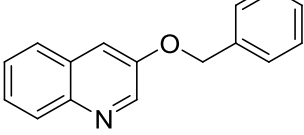
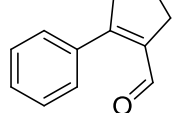
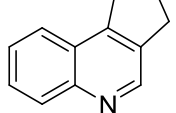
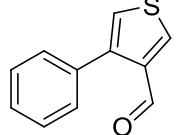
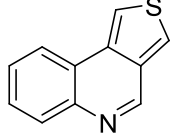


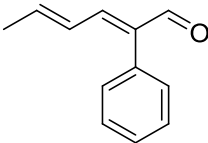
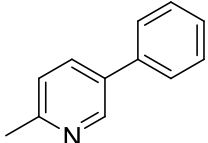
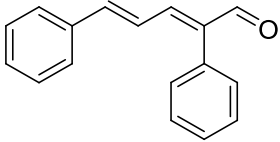
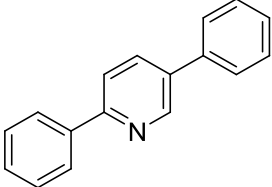
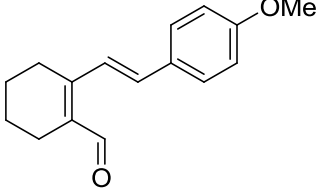
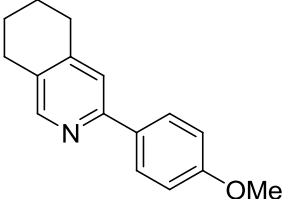






11. Unsuccessful substrates

SM	TM	Yield
		0 %
		0%
		0 %
		24%
		0%
		0 %
		0 %
		0 %
		0%

		18 %
		14%
		0%