

Palladium-Catalyzed Annulation of Benznes with *N*- Substituted-*N*-(2-halophenyl)formamides: Synthesis of Phenanthridinones

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Supporting Information

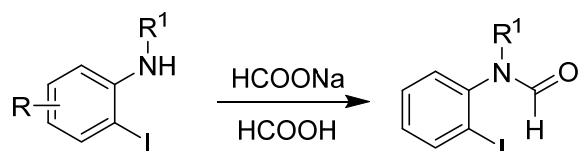
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1) General Information

^1H and ^{13}C NMR spectra were recorded on a Bruker ARX500 spectrometer (FT, 500 MHz for ^1H ; 125 MHz for ^{13}C) at room temperature, respectively. The ^1H NMR spectra were taken in CDCl_3 and the chemical shifts are given in *ppm* with respect to tetramethylsilane (TMS) used as an internal standard. The ^{13}C NMR spectra were taken in CDCl_3 and the central peak of the solvent was adjusted to 77.00 ppm and used as a reference. High-resolution mass spectra (HRMS) were recorded on a Bruker Apex IV FTMS mass spectrometer using ESI (electrospray ionization). Melting points were measured uncorrected. Reactions were monitored by thin-layer chromatography or GC-MS analysis. Column chromatography (petroleum ether/ethyl acetate) was performed on silica gel (200-300 mesh). Unless otherwise noted, all reactions were run under nitrogen atmosphere.

2) Synthesis of Starting Materials

Preparation of 1:¹

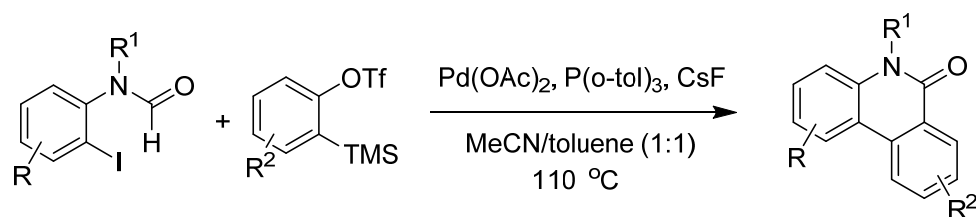


To a schlenk tube were added o-iodoaniline (1 mmol), HCOONa (0.5 mmol), and HCOOH (2 mL). The resulting mixture was stirred at 70°C for appropriate time. After completion of the reaction, water was added to the reaction mixture, The resulting solution was extracted with ethyl acetate. The organic layers were combined and dried over anhydrous Na_2SO_4 . The solvent was removed under vacuum and the residue was purified by flash column chromatography on silica gel using petroleum ether/ethyl acetate as the eluent.

Preparation of 2:

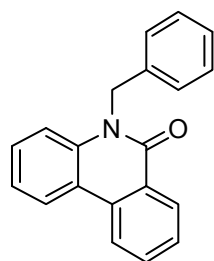
Compounds **2b-2e** were prepared according the literature method.²

3) Typical Procedures

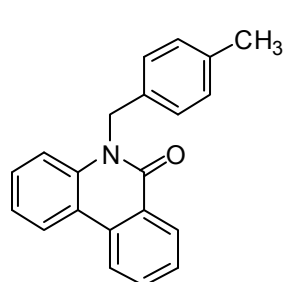


To a schlenk tube were added *N*-substituted-*N*-(2-iodophenyl)form-amide (0.3 mmol), aryne (0.36 mmol), Pd(OAc)₂ (5 mol%), P(*o*-tolyl)₃ (10 mol%), CsF (0.9 mmol), and MeCN/toluene (1:1, 2 mL). Then under the protection of nitrogen, the mixture was stirred at 110 °C (oil bath temperature) for the indicated time until complete consumption of starting material as monitored by TLC and GC-MS analysis. After the reaction was finished, the reaction mixture was filtered by a crude column with ethyl acetate as eluent, and evaporated under vacuum. The resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate = 6:1) to afford the desired product.

4) Characterization Data

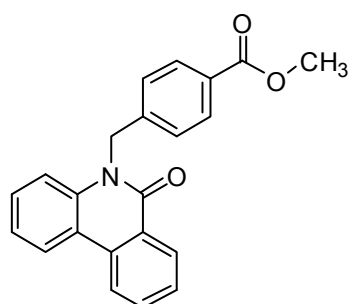


5-benzylphenanthridin-6(5H)-one (3a):³ Pale yellow solid, isolated yield 93% (79.5 mg); mp: 115.6-115.9 °C; ¹H NMR (500 MHz, CDCl₃) δ: 8.61 (d, *J* = 8.0 Hz, 1H), 8.24 (t, *J* = 8.8 Hz, 2H), 7.74 (t, *J* = 7.8 Hz, 1H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.35 (t, *J* = 7.8 Hz, 1H), 7.28-7.20 (m, 7H), 5.63 (s, 2H); ¹³C NMR (125 MHz, CDCl₃) δ: 161.81, 137.23, 136.51, 133.75, 132.60, 129.45, 129.07, 128.71, 127.94, 127.10, 126.45, 125.32, 123.19, 122.48, 121.61, 119.41, 115.92, 46.39; IR (KBr): 1635 (C=O) cm⁻¹.



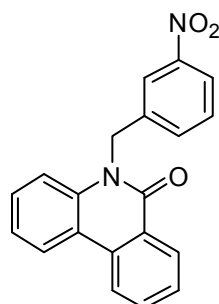
5-(4-methylbenzyl)phenanthridin-6(5H)-one (3b):³ Pale yellow solid, isolated yield 88% (79.1 mg); mp: 108.4-109.5 °C; ¹H NMR (500 MHz, CDCl₃) δ: 8.60 (dd, *J* = 7.5 Hz, 1.0 Hz, 1H), 8.22-8.18 (m, 2H), 7.71 (td, *J* = 7.8 Hz, 1.0 Hz,

1H), 7.56 (td, $J = 7.5$ Hz, 1.0 Hz, 1H), 7.33 (td, $J = 7.8$ Hz, 1.0 Hz, 1H), 7.28 (d, $J = 7.5$ Hz, 1H), 7.19 (td, $J = 7.5$ Hz, 1.0 Hz, 1H), 7.14 (d, $J = 8.5$ Hz, 2H), 7.06 (d, $J = 8.0$ Hz, 2H), 5.58 (s, 2H), 2.25 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.72, 137.23, 136.64, 133.69, 133.47, 132.48, 129.38, 129.33, 129.00, 127.84, 126.43, 125.33, 123.10, 122.35, 121.54, 119.33, 115.88, 46.11, 20.94; IR (KBr): 1652 (C=O) cm^{-1} .



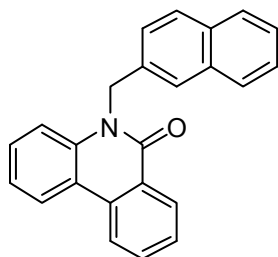
methyl 4-((6-oxophenanthridin-5(6H)-yl)methyl)benzoate (3c): Yellow solid, isolated yield 91% (93.6 mg); mp: 176.5-177.7 °C; ^1H NMR (500 MHz, CDCl_3) δ : 8.59 (d, $J = 8.0$ Hz, 1H), 8.23 (t, $J = 8.3$ Hz, 2H), 7.94 (d, $J = 8.0$ Hz, 2H), 7.74 (t, $J = 7.5$ Hz, 1H),

7.58 (t, $J = 7.5$ Hz, 1H), 7.34-7.29 (m, 3H), 7.21 (t, $J = 7.5$ Hz, 1H), 7.16 (d, $J = 8.5$ Hz, 1H), 5.66 (s, 2H), 3.84 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 166.52, 161.64, 141.83, 136.88, 133.63, 132.66, 129.96, 129.41, 129.03, 128.94, 127.94, 126.34, 125.07, 123.25, 122.57, 121.60, 119.34, 115.56, 51.88, 46.13; IR (KBr): 1708, 1649 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{22}\text{H}_{17}\text{NO}_3]\text{H}^+$: 344.1281; found 344.1284.



5-(3-nitrobenzyl)phenanthridin-6(5H)-one (3d): Pale yellow solid, isolated yield 85% (84.2 mg); mp: 155.2-156.1 °C; ^1H NMR (500 MHz, CDCl_3) δ : 8.58 (dd, $J = 8.0$ Hz, 1.0 Hz, 1H), 8.26 (d, $J = 8.0$ Hz, 2H), 8.14 (s, 1H), 8.05 (dd, $J = 8.0$ Hz, 1.0 Hz, 1H), 7.77 (td, $J = 7.5$ Hz, 1.0 Hz, 1H), 7.60 (t, $J = 7.5$ Hz, 0.5 Hz, 1H), 7.55 (d, $J = 8.0$ Hz, 1H), 7.42 (t, $J = 8.0$ Hz, 1H), 7.38 (td, $J = 8.0$ Hz, 1.0 Hz, 1H), 7.26 (t, $J = 7.3$ Hz, 1H), 7.17 (d, $J = 8.5$ Hz, 1H), 5.69 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.70, 148.44, 138.88, 136.68, 133.65, 132.87, 132.58, 129.73, 129.60, 128.96, 128.09, 124.95, 123.50, 122.84, 122.28, 121.69, 121.51, 119.47, 115.20, 45.71; IR (KBr): 1645 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{20}\text{H}_{14}\text{N}_2\text{O}_3]\text{H}^+$: 331.1077;

found 331.1079.



5-(naphthalen-2-ylmethyl)phenanthridin-6(5H)-one (3e):

Pale yellow solid, isolated yield 89% (89.7 mg); mp: 149.2-

150.5 °C; ^1H NMR (500 MHz, CDCl_3) δ : 8.64 (dd, J = 8.0 Hz,

1.0 Hz, 1H), 8.20 (d, J = 8.0 Hz, 1H), 8.17 (dd, J = 8.0 Hz, 1.0

Hz, 1H), 7.74-7.69 (m, 3H), 7.63 (t, J = 4.8 Hz, 1H), 7.61 (s, 1H), 7.57 (td, J = 7.5 Hz,

1.0 Hz, 1H), 7.39 (dd, J = 8.5 Hz, 1.5 Hz, 1H), 7.36-7.34 (m, 2H), 7.29-7.24 (m, 2H),

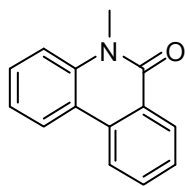
7.19-7.13 (m, 1H), 5.75 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.80, 137.16,

134.03, 133.74, 133.30, 132.57, 132.52, 129.40, 129.03, 128.56, 127.90, 127.55,

127.52, 126.03, 125.61, 125.28, 124.96, 124.61, 123.13, 122.43, 121.59, 119.34,

115.90, 46.55; IR (KBr): 1649 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for

$[\text{C}_{24}\text{H}_{17}\text{NO}]^+\text{H}^+$: 336.1383; found 336.1386.



5-methylphenanthridin-6(5H)-one (3f):⁴ Red brown solid, isolated

yield 93% (58.3 mg); mp: 113.4-114.5 °C; ^1H NMR (500 MHz,

CDCl_3) δ : 8.49 (dd, J = 8.0 Hz, 1.5 Hz, 1H), 8.13 (d, J = 8.5 Hz, 2H),

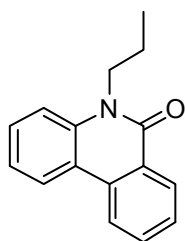
7.67 (td, J = 7.5 Hz, 1.0 Hz, 1H), 7.52 (td, J = 7.5 Hz, 1.0 Hz, 1H), 7.45 (td, J = 8.0

Hz, 1.0 Hz, 1H), 7.29 (d, J = 8.0 Hz, 1H), 7.22 (td, J = 7.5 Hz, 1.0 Hz, 1H), 3.72 (s,

2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.35, 137.74, 133.28, 132.14, 129.31, 128.63,

127.69, 125.33, 122.96, 122.22, 121.39, 118.98, 114.79, 29.74; IR (KBr): 1645 (C=O)

cm^{-1} .



5-propylphenanthridin-6(5H)-one (3g): Yellow solid, isolated

yield 85% (60.4 mg); mp: 53.5-54.5 °C; ^1H NMR (500 MHz, CDCl_3)

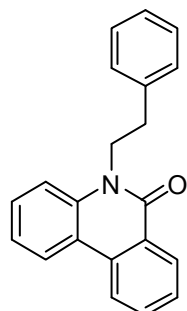
δ : 8.54 (dd, J = 8.0 Hz, 1.0 Hz, 1H), 8.23 (t, J = 9.5 Hz, 2H), 7.71 (td,

J = 7.8 Hz, 1.0 Hz, 1H), 7.55 (t, J = 7.5 Hz, 1H), 7.50 (td, J = 7.8 Hz,

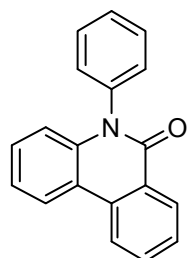
1.0 Hz, 1H), 7.36 (d, J = 8.5 Hz, 1H), 7.27 (t, J = 7.5 Hz, 1H), 4.33 (t, J = 7.8 Hz, 2H),

1.86-1.79 (m, 2H), 1.07 (t, J = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.22,

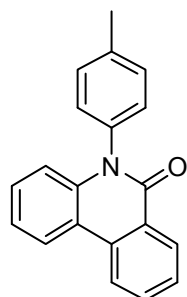
136.96, 133.40, 132.22, 129.37, 128.68, 127.76, 125.39, 123.30, 122.11, 121.42, 119.30, 114.98, 44.10, 20.63, 11.34; IR (KBr): 1645 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{16}\text{H}_{15}\text{NO}]\text{H}^+$: 238.1226; found 238.1230.



5-phenethylphenanthridin-6(5H)-one (3h): Pale yellow solid, isolated yield 88% (78.9 mg); mp: 81.2-82.2 $^{\circ}\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ : 8.54 (d, $J = 8.0$ Hz, 1H), 8.23 (d, $J = 8.0$ Hz, 1H), 8.20 (d, $J = 8.5$ Hz, 1H), 7.71 (td, $J = 7.5$ Hz, 1.0 Hz, 1H), 7.55 (t, $J = 7.8$ Hz, 1H), 7.52 (t, $J = 7.3$ Hz, 1H), 7.42 (d, $J = 8.5$ Hz, 1H), 7.39 (d, $J = 7.0$ Hz, 2H), 7.34 (t, $J = 7.5$ Hz, 2H), 7.29-7.24 (m, 2H), 4.54 (t, $J = 8.3$ Hz, 2H), 3.06 (t, $J = 8.5$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.09, 138.38, 136.77, 133.39, 132.33, 129.51, 128.71, 128.60, 128.56, 127.84, 126.56, 125.32, 123.43, 122.26, 121.48, 119.33, 114.64, 44.18, 33.48; IR (KBr): 1649 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{21}\text{H}_{17}\text{NO}]\text{H}^+$: 300.1383; found 300.1387.

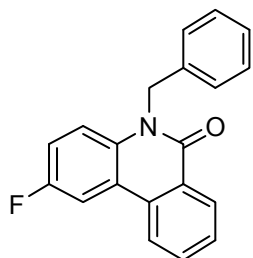


5-phenylphenanthridin-6(5H)-one (3i):⁴ Yellow solid, isolated yield 60% (48.8 mg); mp: 207.4-208.8 $^{\circ}\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ : 8.55 (dd, $J = 8.0$ Hz, 1.0 Hz, 1H), 8.29 (d, $J = 8.0$ Hz, 1H), 8.26 (dd, $J = 7.5$ Hz, 1.5 Hz, 1H), 7.77 (t, $J = 7.5$ Hz, 1.0 Hz, 1H), 7.61-7.56 (m, 3H), 7.51 (t, $J = 7.3$ Hz, 1H), 7.32-7.31 (m, 2H), 7.25 (td, $J = 7.5$ Hz, 1.5 Hz, 2H), 6.67 (dd, $J = 7.5$ Hz, 1.0 Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.51, 139.01, 138.15, 133.85, 132.68, 130.04, 128.96 (2C), 128.83, 128.62, 127.96, 125.70, 122.86, 122.51, 121.66, 118.85, 116.85; IR (KBr): 1655 (C=O) cm^{-1} .



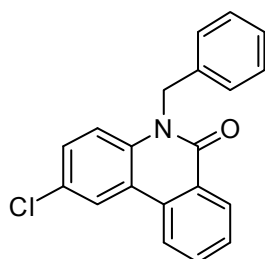
5-(p-tolyl)phenanthridin-6(5H)-one (3j):⁵ Pale yellow solid, isolated yield 54% (46.2 mg); mp: 173.5-174.7 $^{\circ}\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ : 8.56 (d, $J = 8.0$ Hz, 1H), 8.31 (d, $J = 8.0$ Hz, 1H), 8.28 (d, $J = 7.5$ Hz, 1H), 7.78 (td, $J = 7.5$ Hz, 1.0 Hz, 1H), 7.59 (t, $J =$

7.5 Hz, 1H), 7.40 (d, $J = 8.5$ Hz, 2H), 7.30-7.24 (m, 2H), 7.20 (d, $J = 8.0$ Hz, 2H), 6.73 (d, $J = 8.0$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.70, 139.19, 138.58, 135.46, 133.87, 132.67, 130.79, 128.96, 128.93, 128.62, 127.98, 125.78, 122.86, 122.47, 121.67, 118.89, 116.99, 21.25; IR (KBr): 1659 (C=O) cm^{-1} .



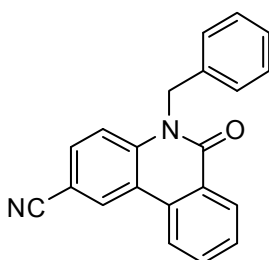
5-benzyl-2-fluorophenanthridin-6(5H)-one (3k): Red brown solid, isolated yield 93% (84.6 mg); mp: 163.6-164.9 °C; ^1H NMR (500 MHz, CDCl_3) δ : 8.59 (d, $J = 8.0$ Hz, 1H), 8.08 (d, $J = 8.0$ Hz, 1H), 7.82 (dd, $J = 9.5$ Hz, 3.0 Hz, 1H), 7.73 (t, $J = 7.8$

Hz, 1H), 7.60 (t, $J = 8.0$ Hz, 1H), 7.27 (d, $J = 7.5$ Hz, 2H), 7.22-7.18 (m, 4H), 7.03 (td, $J = 8.5$ Hz, 2.5 Hz, 1H), 5.59 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.31, 158.26 (d, $J = 240.63$ Hz), 136.22, 133.54, 132.74 (d, $J = 2.6$ Hz), 132.65, 129.14, 128.75, 128.56, 127.20, 126.37, 125.48, 121.74, 120.74 (d, $J = 7.8$ Hz), 117.38 (d, $J = 8.0$ Hz), 116.64 (d, $J = 23.0$ Hz), 109.09 (d, $J = 23.5$ Hz), 46.51; IR (KBr): 1635 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{20}\text{H}_{14}\text{FNO}]^+\text{H}^+$: 304.1132; found 304.1138.



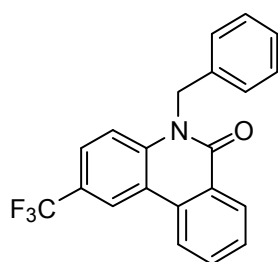
5-benzyl-2-chlorophenanthridin-6(5H)-one (3l): Yellow brown solid, isolated yield 88% (84.2 mg); mp: 167.1-168.3 °C; ^1H NMR (500 MHz, CDCl_3) δ : 8.57 (dd, $J = 8.0$ Hz, 1.5 Hz, 1H), 8.09-8.07 (m, 2H), 7.72 (td, $J = 7.5$ Hz, 1.0 Hz, 1H), 7.59

(td, $J = 7.5$ Hz, 0.5 Hz, 1H), 7.28-7.20 (m, 6H), 7.15 (d, $J = 9.0$ Hz, 1H), 5.57 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.33, 136.08, 135.64, 132.73, 132.44, 129.18, 129.10, 128.76, 128.57, 128.09, 127.24, 126.37, 125.40, 122.85, 121.61, 120.68, 117.22, 46.39; IR (KBr): 1635 (C=O) cm^{-1} .



5-benzyl-6-oxo-5,6-dihydrophenanthridine-2-carbonitrile (3m): Pale yellow solid, isolated yield 90% (83.7 mg); mp: 189.7-190.8 °C; ^1H NMR (500 MHz, CDCl_3) δ : 8.57 (d, $J =$

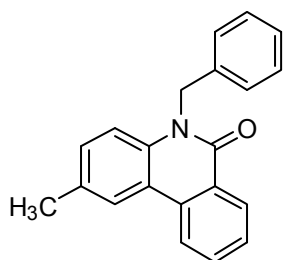
8.0 Hz, 1H), 8.47 (d, $J = 1.5$ Hz, 1H), 8.18 (d, $J = 8.0$ Hz, 1H), 7.81 (td, $J = 7.8$ Hz, 1.0 Hz, 1H), 7.66 (t, $J = 7.5$ Hz, 1H), 7.56 (dd, $J = 8.5$ Hz, 1.5 Hz, 1H), 7.33 (d, $J = 8.5$ Hz, 1H), 7.29 (t, $J = 7.3$ Hz, 2H), 7.25-7.21 (m, 3H), 5.62 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.43, 139.99, 135.46, 133.23, 132.04, 131.93, 129.17 (2C), 128.85, 127.69, 127.45, 126.29, 125.31, 121.63, 119.88, 118.50, 116.64, 105.87, 46.46; IR (KBr): 1655 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{21}\text{H}_{14}\text{N}_2\text{O}]\text{H}^+$: 311.1179; found 311.1183.



5-benzyl-2-(trifluoromethyl)phenanthridin-6(5H)-one

(3n): Pale yellow solid, isolated yield 84% (88.9 mg); mp: 142.8-143.8 $^{\circ}\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ : 8.58 (dd, $J = 8.0$ Hz, 1.0 Hz, 1H), 8.44 (d, $J = 1.0$ Hz, 1H), 8.23 (d, $J = 8.5$

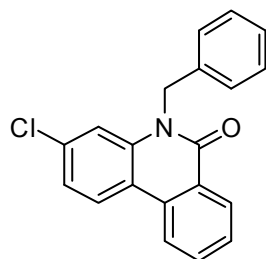
Hz, 1H), 7.78 (td, $J = 7.5$ Hz, 1.5 Hz, 1H), 7.63 (td, $J = 7.5$ Hz, 0.5 Hz, 1H), 7.56 (dd, $J = 9.0$ Hz, 1.5 Hz, 1H), 7.34 (d, $J = 9.0$ Hz, 1H), 7.30-7.27 (m, 2H), 7.22 (d, $J = 7.0$ Hz, 3H), 5.62 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.64, 139.43, 135.87, 133.03, 132.72, 129.20, 128.86 (2C), 127.39, 126.39, 125.85 (q, $J = 3.5$ Hz), 125.43, 124.51 (q, $J = 32.6$ Hz), 124.08 (q, $J = 270.0$ Hz), 121.69, 120.55 (q, $J = 3.6$ Hz), 119.40, 116.32, 46.52; IR (KBr): 1652 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{21}\text{H}_{14}\text{F}_3\text{NO}]\text{H}^+$: 354.1100; found 354.1108.



5-benzyl-2-methylphenanthridin-6(5H)-one (3o): Yellow

brown solid, isolated yield 86% (77.1 mg); mp: 162.5-163.7 $^{\circ}\text{C}$; ^1H NMR (500 MHz, CDCl_3) δ : 8.59 (dd, $J = 8.0$ Hz, 1.5 Hz, 1H), 8.21 (d, $J = 8.5$ Hz, 1H), 7.98 (s, 1H), 7.70 (td, $J = 7.8$ Hz, 1.5 Hz, 1H), 7.55 (td, $J = 7.5$ Hz, 1.0 Hz, 1H), 7.26-7.20 (m, 4H), 7.18 (t, $J = 7.0$ Hz, 1H), 7.13 (s, 2H), 5.59 (s, 2H), 2.37 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.57, 136.62, 135.03, 133.62, 132.38, 131.79, 130.41, 128.99, 128.62, 127.71, 127.00, 126.42, 125.34, 123.24, 121.50, 119.16, 115.74, 46.24, 20.78; IR (KBr): 1632

(C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{21}\text{H}_{17}\text{NO}]^+\text{H}^+$: 300.1383; found 300.1385.



5-benzyl-3-chlorophenanthridin-6(5H)-one (3p): Red

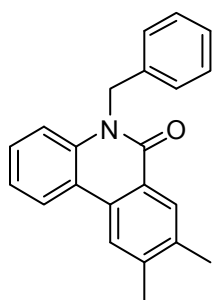
brown solid, isolated yield 95% (91.1 mg); mp: 191.6-192.8 °C;

^1H NMR (500 MHz, CDCl_3) δ : 8.56 (d, $J = 7.5$ Hz, 1H), 8.12 (d, $J = 8.0$ Hz, 1H), 8.07 (d, $J = 8.5$ Hz, 1H), 7.72 (t, $J = 7.5$ Hz,

1H), 7.58 (t, $J = 7.5$ Hz, 1H), 7.31-7.21 (m, 6H), 7.15 (d, $J = 8.5$ Hz, 1H), 5.56 (s, 2H);

^{13}C NMR (125 MHz, CDCl_3) δ : 161.63, 138.14, 135.91, 135.23, 132.98, 132.80, 129.13, 128.83, 128.20, 127.34, 126.50, 125.05, 124.36, 122.66, 121.53, 117.91,

115.78, 46.45; IR (KBr): 1645 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{20}\text{H}_{14}\text{ClNO}]^+\text{H}^+$: 320.0837; found 320.0844.



5-benzyl-8,9-dimethylphenanthridin-6(5H)-one (3q): Pale

yellow solid, isolated yield 83% (77.9 mg); mp: 186.8-187.9 °C; ^1H

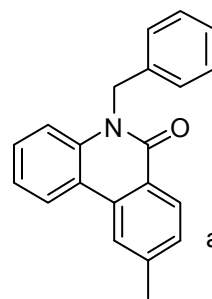
NMR (500 MHz, CDCl_3) δ : 8.33 (s, 1H), 8.17 (d, $J = 7.5$ Hz, 1H), 7.96 (s, 1H), 7.30-7.21 (m, 6H), 7.20-7.16 (m, 2H), 5.60 (s, 2H),

2.42 (s, 3H), 2.39 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.78,

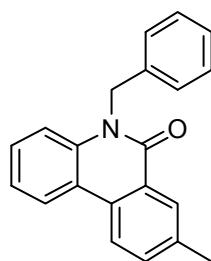
142.28, 137.30, 136.90, 136.66, 131.56, 129.16, 128.75, 128.61, 126.96, 126.40,

123.17, 122.80, 122.22 (2C), 119.41, 115.75, 46.15, 20.49, 19.65; IR (KBr): 1635

(C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{22}\text{H}_{19}\text{NO}]^+\text{H}^+$: 314.1539; found 314.1546.



and



5-benzyl-9-methylphenanthridin-6(5H)-

one (3r) and 5-benzyl-8-

methylphenanthridin-6(5H)-one (3r'):³

3r:3r' = 1.1:1; Pale yellow solid, isolated yield

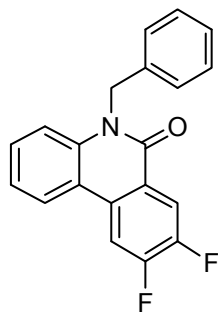
91% (81.6 mg); mp: 172.1-173.1 °C; ^1H NMR

(500 MHz, CDCl_3) δ : 8.48 (d, $J = 8.0$ Hz, 1H), 8.39 (s, 0.94H), 8.19 (d, $J = 7.5$ Hz,

1H), 8.15 (t, $J = 7.5$ Hz, 0.94H), 8.10 (d, $J = 8.5$ Hz, 0.94H), 8.01 (s, 1H), 7.53 (dd, J

= 8.0 Hz, 1.0 Hz, 0.94H), 7.39 (d, $J = 8.0$ Hz, 1H), 7.32-7.17 (m, 15.5H), 5.61 (s,

3.7H), 2.52 (s, 3H), 2.49 (s, 2.8H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.77, 163.73, 143.04, 138.00, 137.22, 136.70, 136.54, 136.51, 133.85, 133.61, 131.17, 129.29, 129.23, 128.94, 128.87, 128.67, 128.62, 126.99, 126.36, 125.01, 123.03, 122.91, 122.84, 122.33, 122.26, 121.58, 121.56, 119.42, 119.26, 115.80, 115.76, 46.25, 46.17, 22.05, 21.25; IR (KBr): 1642 ($\text{C}=\text{O}$) cm^{-1} .



5-benzyl-8,9-difluorophenanthridin-6(5H)-one (3s): Red brown

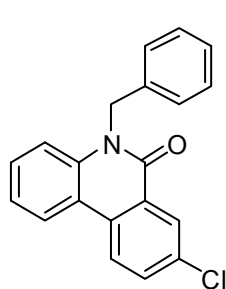
solid, isolated yield 24% (23.1 mg); mp: 170.2-171.2 $^{\circ}\text{C}$; ^1H NMR

(500 MHz, CDCl_3) δ : 8.41-8.37 (m, 1H), 8.09 (d, $J = 7.5$ Hz, 1H),

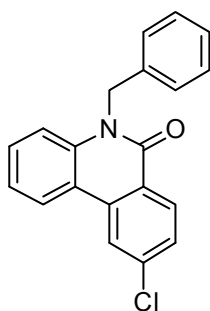
8.05-8.01 (m, 1H), 7.43 (t, $J = 7.5$ Hz, 1H), 7.33-7.29 (m, 4H),

7.26-7.24 (m, 3H), 5.64 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ :

160.34, 154.09 (dd, $J = 254.5$ Hz, 14.4 Hz), 150.49 (dd, $J = 250.6$ Hz, 13.1 Hz), 137.42, 136.15, 131.81 (dd, $J = 8.0$ Hz, 2.8 Hz), 130.12, 128.85, 127.33, 126.46, 123.41, 122.89, 122.75 (dd, $J = 5.4$ Hz, 1.9 Hz), 118.19, 117.53 (d, $J = 18.5$ Hz), 116.26, 110.29 (d, $J = 19.0$ Hz), 46.64; IR (KBr): 1648 ($\text{C}=\text{O}$) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{20}\text{H}_{13}\text{F}_2\text{NO}]^{\text{H}^+}$: 322.1038; found 322.1046.



and



5-benzyl-8-chlorophenanthridin-6(5H)-one (3t) and 5-benzyl-9-chlorophenanthridin-6(5H)-one (3t'):³

3t:3t' = 1.6:1; Red brown solid, isolated yield 32% (30.6 mg); mp: 153.2-154.7 $^{\circ}\text{C}$;

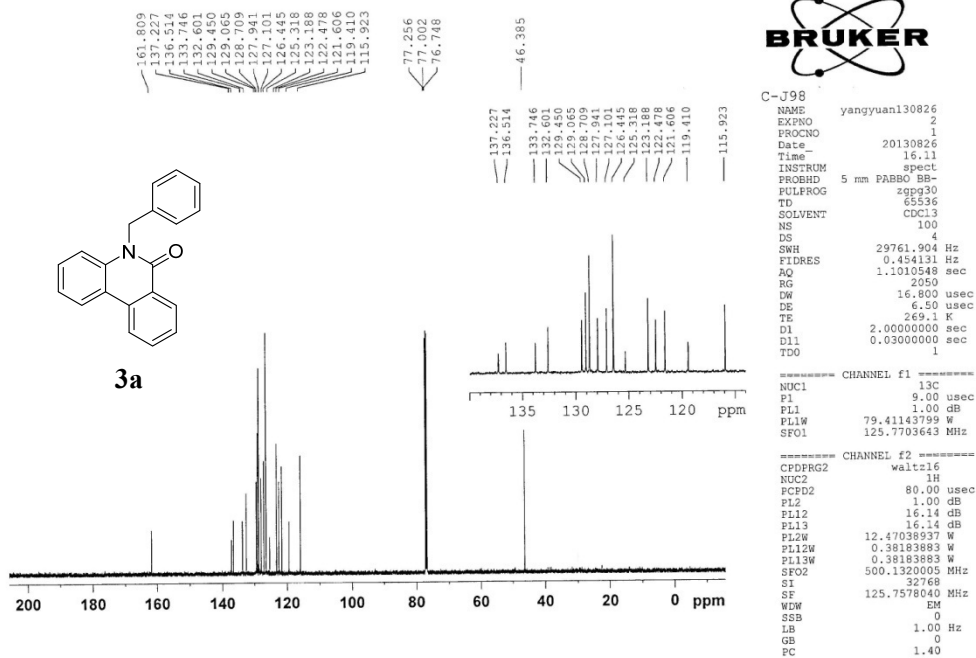
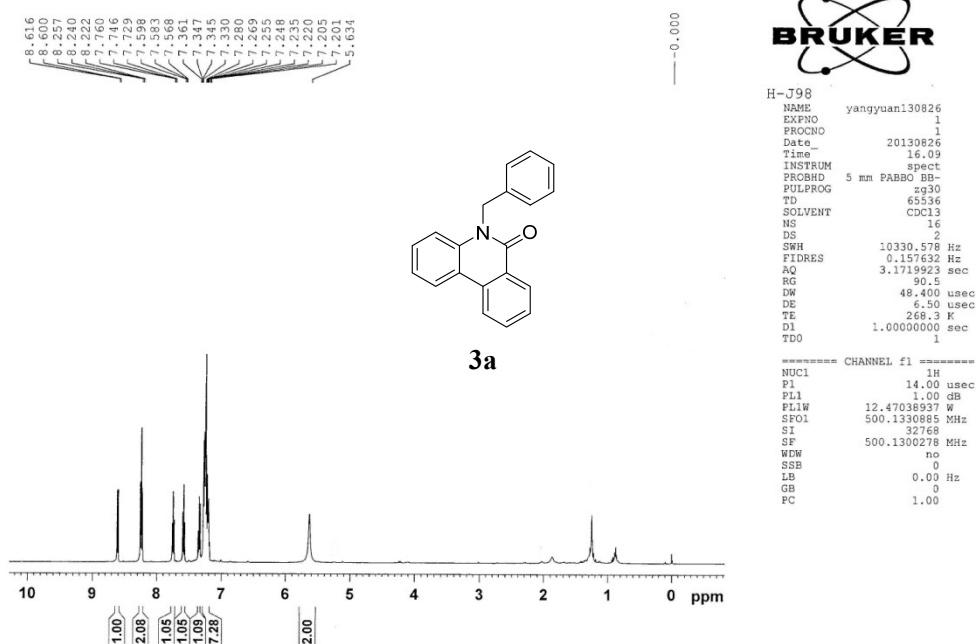
^1H NMR (500 MHz, CDCl_3) δ : 8.57 (d, $J = 2.0$ Hz, 1H), 8.52 (d, $J = 8.5$ Hz, 0.6H), 8.22-8.17 (m, 3.2H), 7.70 (dd, $J = 8.5$ Hz, 2.0 Hz, 1H), 7.53 (dd, $J = 9.0$ Hz, 2.0 Hz, 0.6H), 7.42-7.37 (m, 1.6H), 7.30-7.25 (m, 4.8H), 7.24-7.22 (m, 6.4H), 5.63 (s, 3.2H); ^{13}C NMR (125 MHz, CDCl_3) δ : 161.19, 160.77, 139.44, 137.72, 137.17, 136.30, 136.26, 135.26, 134.21, 132.95, 132.22, 130.93, 130.23, 129.82, 128.79, 128.64, 128.37, 127.26, 126.63, 126.48, 123.44, 123.38, 123.24, 122.77, 122.72, 121.61,

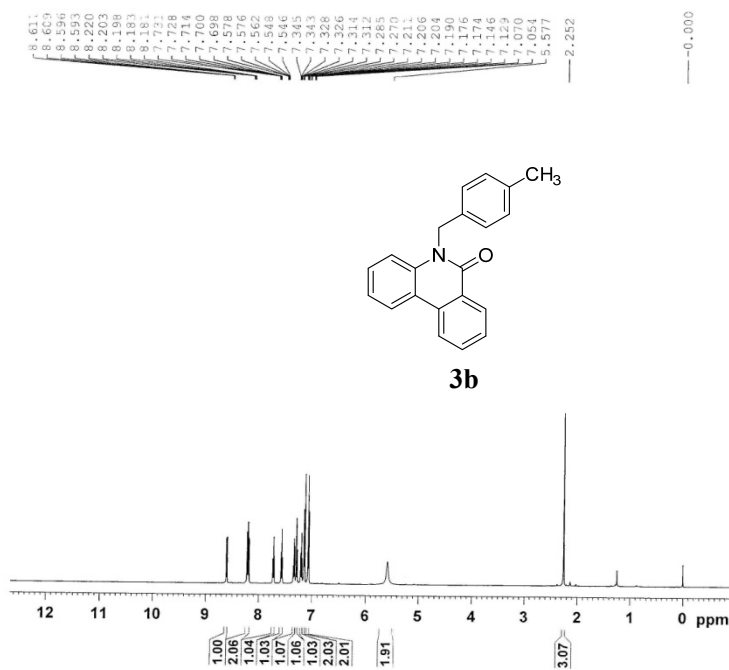
118.74, 118.35, 116.09, 46.57, 46.47; IR (KBr): 1638 (C=O) cm^{-1} ; HRMS (ESI, m/z) calcd for $[\text{C}_{20}\text{H}_{14}\text{ClNO}]\text{H}^+$: 320.0837; found 320.0837.

5) References

1. G. Brahmachari and S. Laskar, *Tetrahedron Lett.*, 2010, **51**, 2319.
2. D. Peña, A. Cobas, D. Pérez and E. Guitián, *Synthesis*, 2002, 1454.
3. C. Lu, A. V. Dubrovskiy and R. C. Larock, *J. Org. Chem.*, 2012, **77**, 8648.
4. T. Furuta, Y. Kitamura, A. Hashimoto, S. Fujii, K. Tanaka and T. Kan, *Org. Lett.*, 2007, **9**, 183.
5. D. H. Hey and T. M. Moynehan, *J. Chem. Soc.*, 1959, 1563.

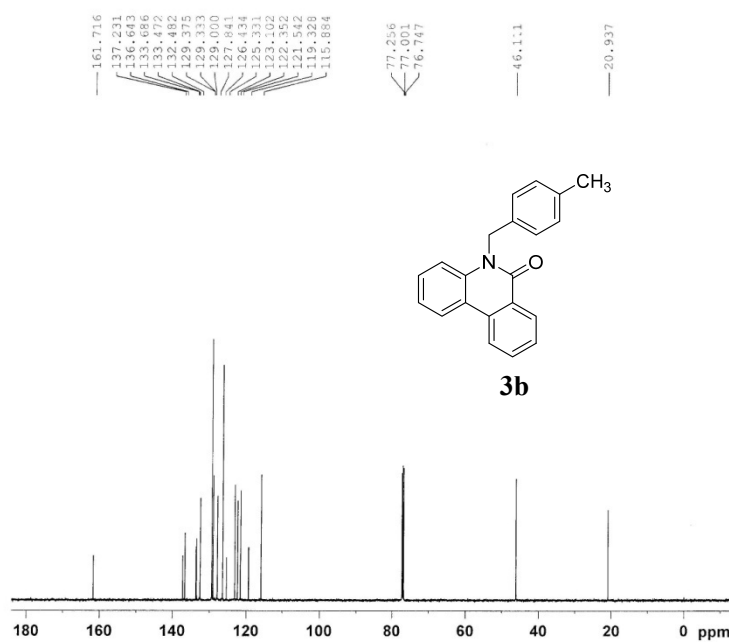
6) Scanned ^1H NMR and ^{13}C NMR Spectra of All New Compounds





J-H137
 NAME yangyuan130920
 EXPNO 1
 PROCNO 1
 Date 20130920
 Time 11.08
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 45.2
 DW 48.400 usec
 DE 6.50 usec
 TE 268.7 K
 D1 1.00000000 sec
 TD0 1

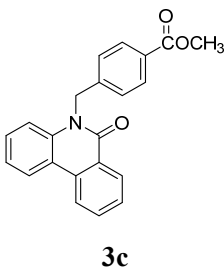
===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 1.00 dB
 PL1W 12.47038937 W
 SF01 500.1330885 MHz
 SI 32768
 SF 500.1300361 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 FC 1.00



J-C137
 NAME yangyuan130920
 EXPNO 2
 PROCNO 1
 Date 20130920
 Time 11.13
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 104
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 270.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 1.00 dB
 PL1W 79.41143799 W
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.00 dB
 PL12 16.14 dB
 PL13 16.14 dB
 PL1W 12.47038937 W
 PL12W 0.38183883 W
 PL13W 0.38183883 W
 SF02 500.1320005 MHz
 SI 32768
 SF 125.7578104 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 FC 1.40

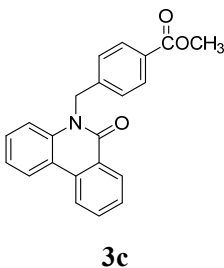


NAME	yangyuan130920	
EXPNO		3
PROCNO		1
Date_	20130920	
Time	11.20	
INSTRUM	spect	
PROBHD	5 mm	PABBO 5B
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS		
SWH	10330.578	Hz
FIDRES	0.157632	Hz
AQ	3.1719923	sec
RG	40.3	
DW	48.400	usec
DE	6.50	usec
TE	268.9	K
D1	1.00000000	sec
TD0	1	

```

NUC1                1H
P1                  14.00 usec
PL1                 1.00 dB
FLW                 12.47038937 W
SFO1               500.1330885 MHz
SI                  32768
SF                 500.1300136 MHz
WDW                 no
SSB                 0
LB                   0.00 Hz
GB                   0
PC                   1.00

```



```
NAME      yangyuan130920
EXPNO     1
PROCNO    1
Date_     20130920
Time      11.25
INSTRUM    spect
PROBHD     5 mm PABBO 1H
PULPROG    zgpg30
TD          65536
SOLVENT    CDCl3
NS          108
DS          4
SWH         29761.904 Hz
FIDRES      0.454131 Hz
AQ          1.1010548 sec
RG          2000
EQ          16.800 Hz
DE          6.50 sec
TE          270.2 K
D1          2.0000000 sec
D11         0.0300000 sec
TD0
```

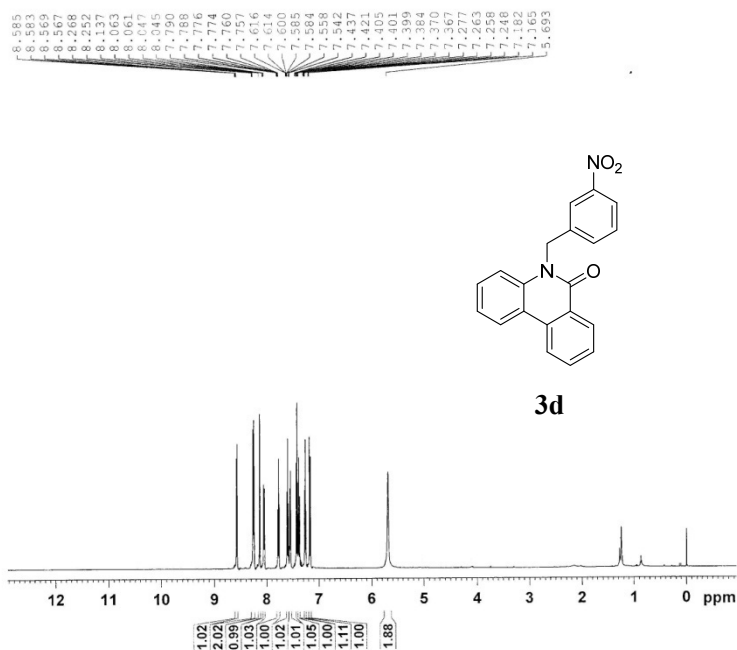
```

===== CHANNEL f1 15C =====
NUC1          1H
PL1           9.10 usec
PL1           1.00 dB
PL1W          79.4143799 W
SF01          125.7703643 MHz

===== CHANNEL f2          =====
CPDPRG2       waltz16
NUC2          1H
FCPR2         80.00 usec
PL1           1.00 dB
PL12          16.14 dB
PL13          16.14 dB
PL14          12.47036937 W
PL12W         0.38183883 W
PL13W         0.38183883 W
SF02          500.1320005 MHz
SI            32768
SF0F          125.7578120 MHz

WDW           EM
SSB           0
LC            1.00 Hz
GB            0
PB            1.40

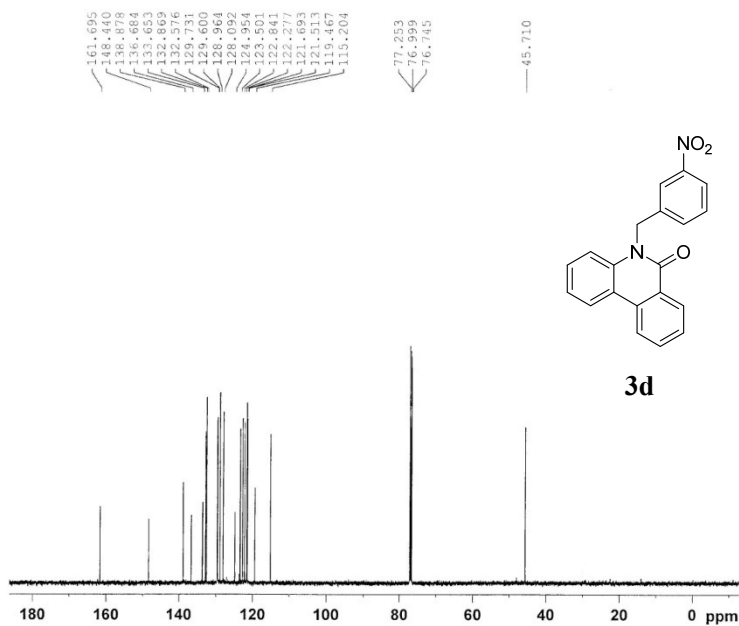
```



J-H169

NAME yangyuan131010
EXPNO 5
PROCNO 1
Date 20131010
Time 21.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.137632 Hz
AQ 3.1719923 sec
RG 161
DW 48.400 usec
DE 6.50 usec
TE 268.3 K
D1 1.00000000 sec
TDO 1

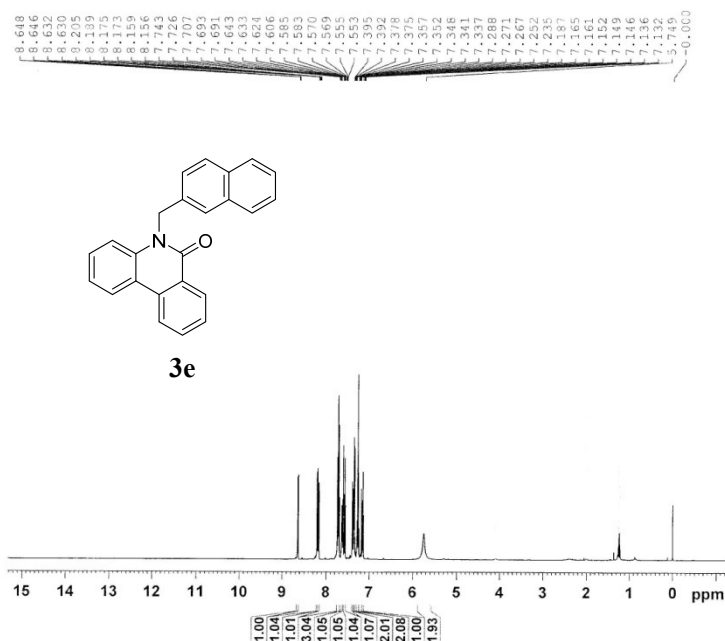
===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
PL1W 12.47038937 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300128 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



J-C169

NAME yangyuan131010
EXPNO 6
PROCNO 1
Date 20131010
Time 21.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 118
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 269.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

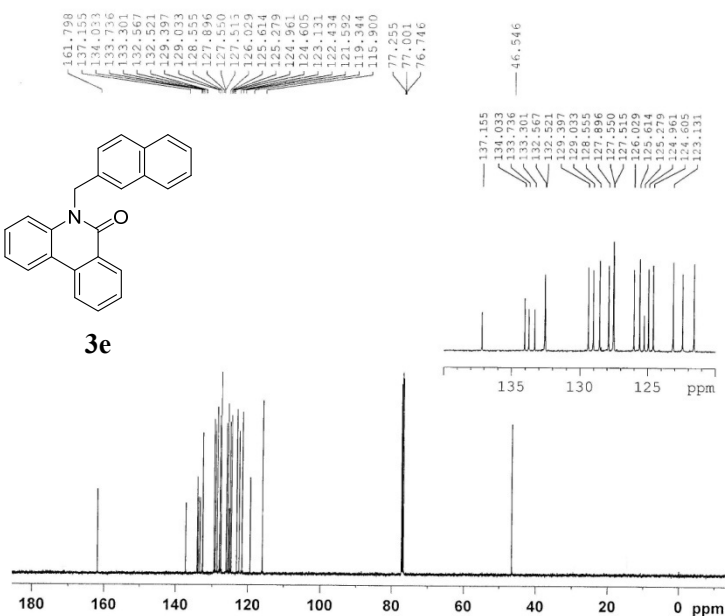
===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 1.00 dB
PL1W 79.41143799 W
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL2 16.14 dB
PL13 16.14 dB
PL2W 12.47038937 W
PL12W 0.38183883 W
PL13W 0.38183883 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578095 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



J-H155

NAME yangyuan130927
EXPNO 3
PROCNO 1
Date_ 20130927
Time 16.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 40.3
DW 48.400 usec
DE 6.50 usec
TE 268.9 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
PL1W 12.47038937 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300494 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

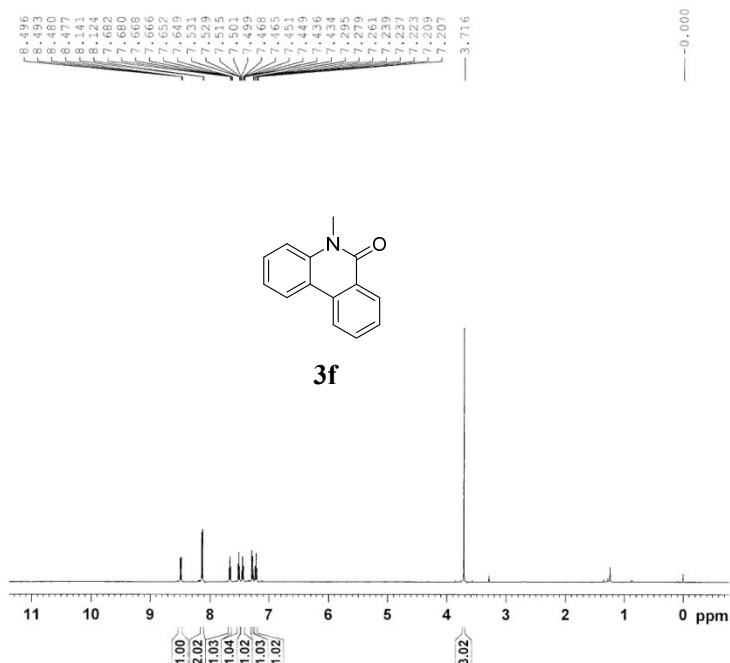


J-C155

NAME yangyuan130927
EXPNO 4
PROCNO 1
Date_ 20130927
Time 16.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 142
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 270.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 1.00 dB
PL1W 79.41143799 W
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.14 dB
PL13 16.14 dB
PL2W 12.47038937 W
PL12W 0.38183883 W
PL13W 0.38183883 W
SFO2 500.1330005 MHz
SI 32768
SF 125.7578158 MHz
WDW EM
SSB 0
TE 1.00 usec

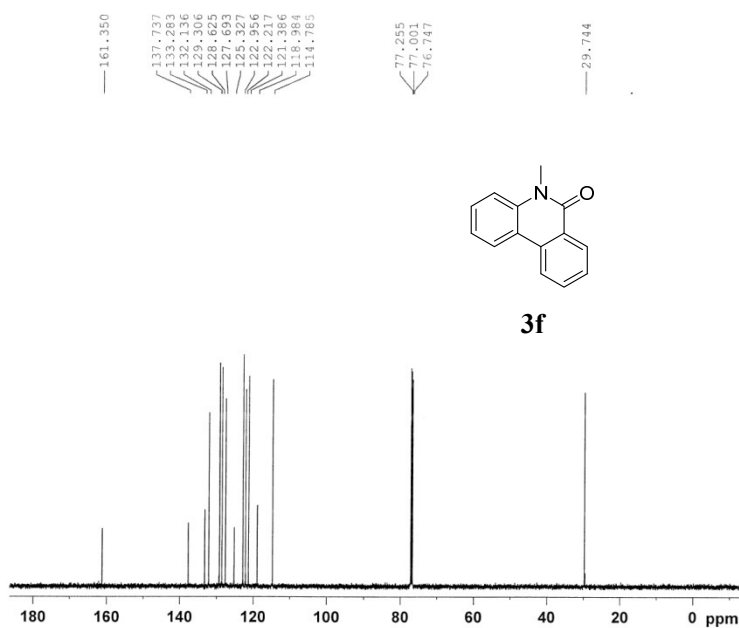


```

NAME      yangyuan131010
EXPNO     3
PROCNO    1
Date_     20131010
Time      20.58
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1719923 sec
RG          80.6
DW         48.400 usec
DE         6.50 usec
TE         268.0 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.00 usec
PL1       1.00 dB
PL1W      12.47038937 W
SFO1      500.1330885 MHz
SI        32768
SF        500.1300113 MHz
WDW       no
SSB       0
LB        0.00 Hz
GB        0
PC        1.00
  
```



```

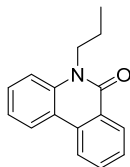
NAME      yangyuan131010
EXPNO     4
PROCNO    1
Date_     20131010
Time      21.01
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         80
DS         4
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         2050
CW         16.800 usec
DE         6.50 usec
TE         268.8 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        9.00 usec
PL1       1.00 dB
PL1W      79.41143799 W
SFO1      125.7703643 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       1.00 dB
PL12      16.14 dB
PL13      16.14 dB
PL2W      12.47038937 W
PL12W     0.38163883 W
PL13W     0.38163883 W
SFO2      500.1320005 MHz
SI        32768
SF        125.7578077 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```



3g



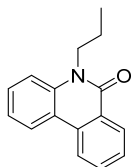
H-J186

```
H-J186
NAME yangyuan131212
EXPNO 3
PROCNO 1
Date_ 20131214
Time 16.13
INSTRUM spect
PROBHD 5 mm TXI 1H/1D
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
AQ 0.157632 Hz
FIDRES 3.1719923 sec
RG 32
DW 48.400 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TDO 1
```

```

===== CHANNEL f1 =====
NUC1              iH
P1                6.80 usec
PL1              0.80 dB
PL1W             13.05810070 W
SFO1             500.1330885 MHz
SI               32768
SF              500.1300138 MHz
WDW              no
SSB              0
LB              0.00 Hz
GB              0
PC              1.00

```



3g



C-J186

```

NAME          yangyuan131212
EXPNO         1
PROCNO        1
Date_         20131214
Time          16.17
INSTRUM       spect
PROBHD        5 mm TXI 1H/D-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            100
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            322
DW            16.800 usec
DE            6.50 usec
TE            673.2 K
D1            2.00000000 sec
D11           0.030000000 sec
TD0           1

```

```

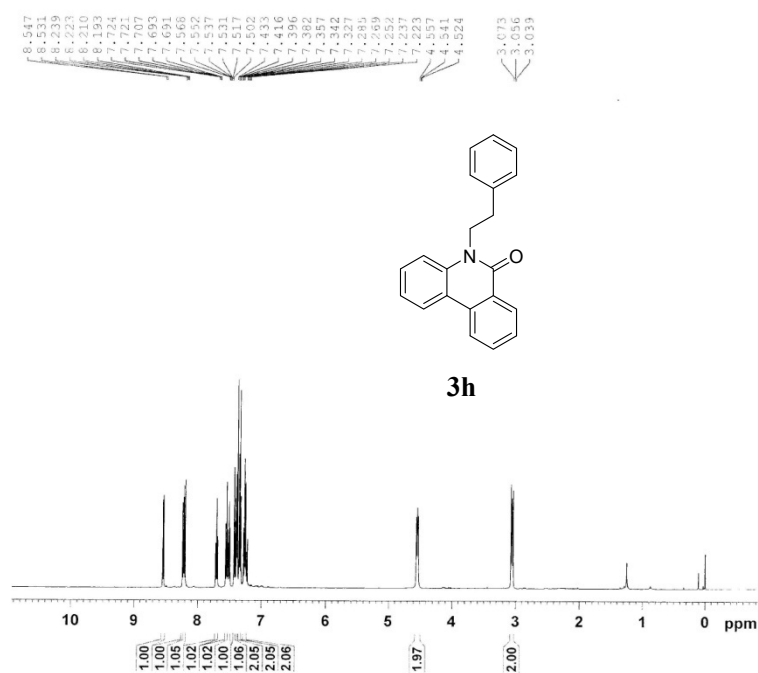
----- CHANNEL f1 -----
NUC1          13C
P1             11.90 usec
PL1            -3.50 dB
PL1W          223.81184387 W
SF01          125.7703643 MHz

```

```

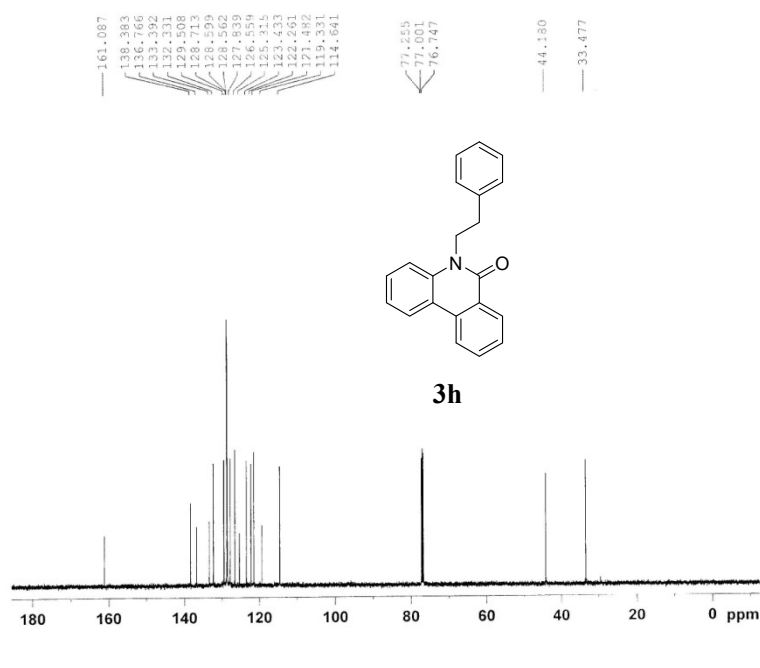
CPDRG2 = CHANNEL f2      waltz16
NUC2      1H
PCPD2      80.00 usec
PL2      4.15 dB
PL12      22.13 dB
PL13      22.13 dB
PL23      6.03781796 W
PL12W      0.09613468 W
PL13W      0.09613468 W
SFO2      570.1320005 MHz
SI      32768
SF      105.7578058 MHz
NDW      EM
SSB      0
LB      1.00 Hz
GB      0
PC      1.40

```



H-J187
 NAME yangyuan131212
 EXPNO 5
 PROCNO 1
 Date 20131214
 Time 16.24
 INSTRUM spect
 PROBHD 5 mm TXI 1H/0-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 25.4
 DW 48.400 usec
 DE 6.50 usec
 TE 673.2 K
 D1 1.0000000 sec
 TDO 1

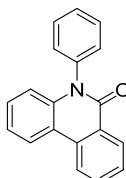
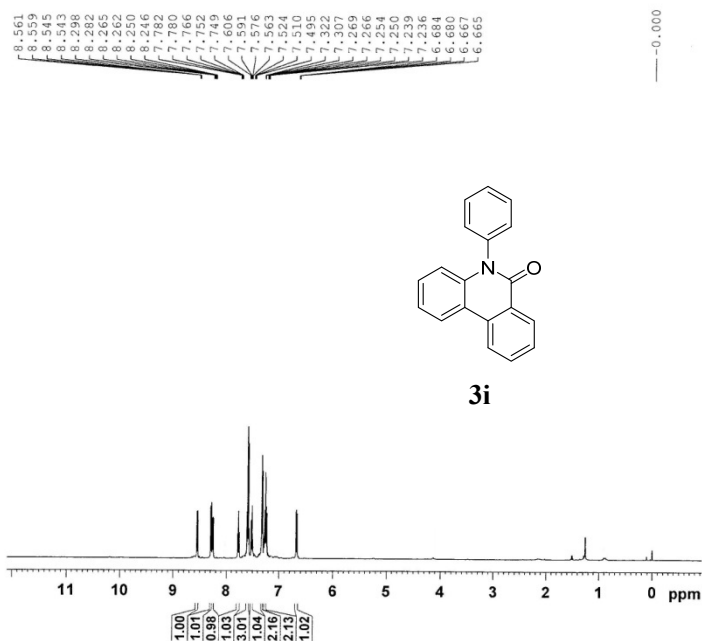
CHANNEL f1
 NUC1 1H
 P1 6.80 usec
 PL1 0.80 dB
 PL1W 13.05810070 W
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1300316 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00



C-J187
 NAME yangyuan131212
 EXPNO 6
 PROCNO 1
 Date 20131214
 Time 16.29
 INSTRUM spect
 PROBHD 5 mm TXI 1H/0-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 100
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 322
 DW 16.800 usec
 DE 6.50 usec
 TE 673.2 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

CHANNEL f1
 NUC1 13C
 P1 11.90 usec
 PL1 -3.50 dB
 PL1W 223.81184387 W
 SFO1 125.7703643 MHz

CHANNEL f2
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 4.15 dB
 PL12 22.13 dB
 PL13 22.13 dB
 PL1W 6.03781796 W
 PL12W 0.09613468 W
 PL13W 0.09613468 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7579122 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

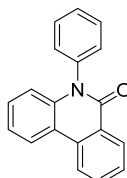
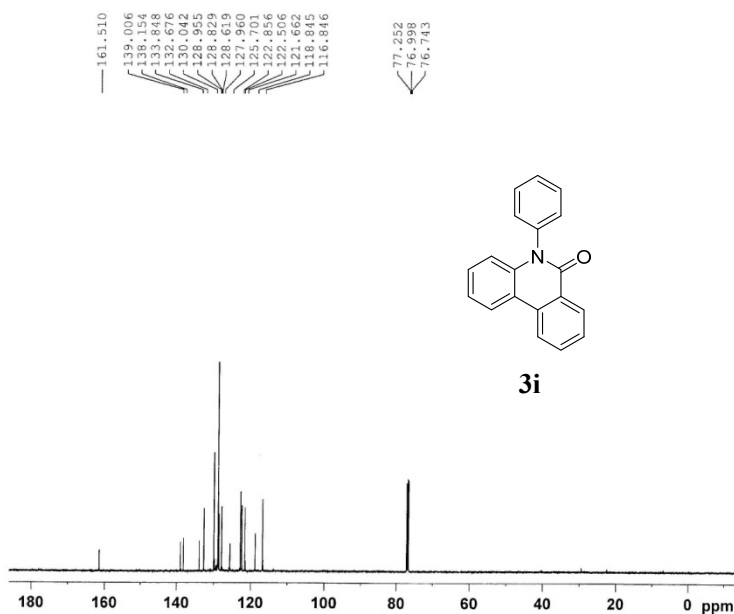


3i



J-H175
NAME yangyuan131015
EXPNO 3
PROCNO 1
Date_ 20131015
Time 10.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 45.2
DW 48.400 usec
DE 6.50 usec
TE 269.4 K
D1 1.00000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
PL1W 12.47038937 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300308 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



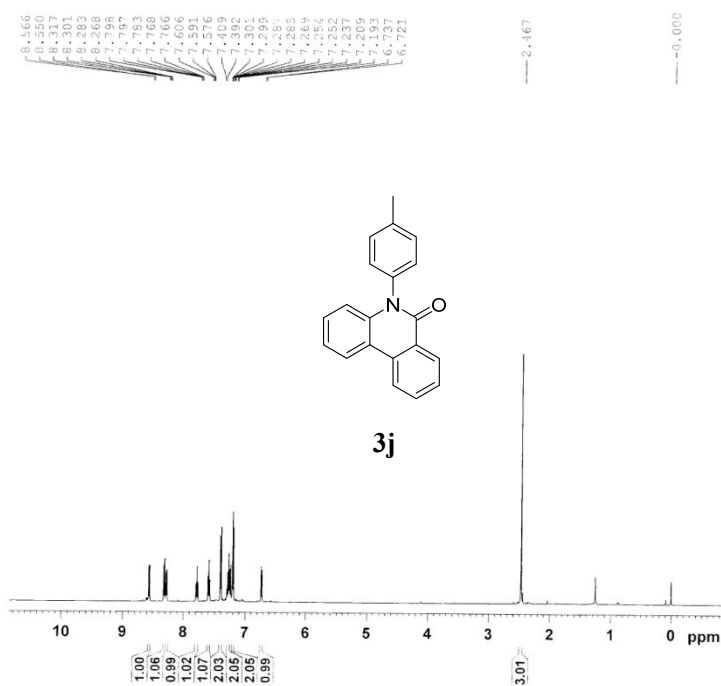
3i



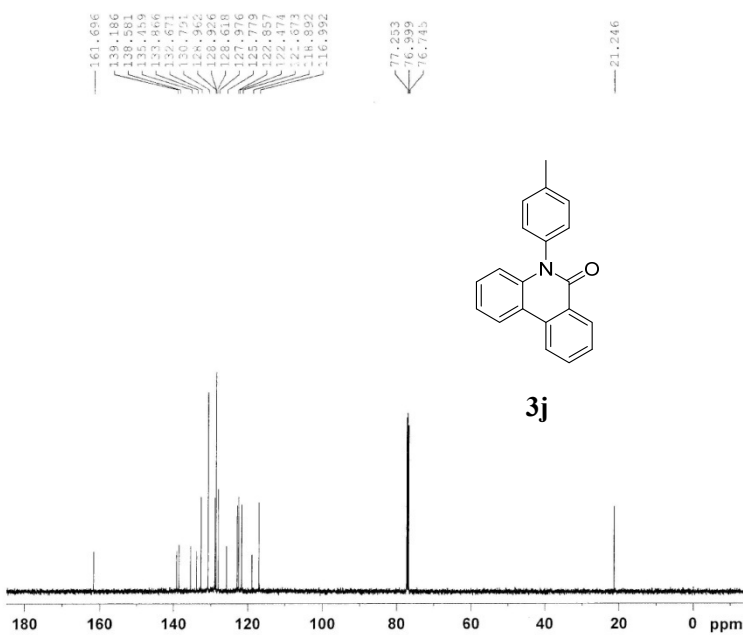
J-C175
NAME yangyuan131015
EXPNO 4
PROCNO 1
Date_ 20131015
Time 10.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 270.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 1.00 dB
PL1W 79.41143799 W
SFO1 125.7703643 MHz

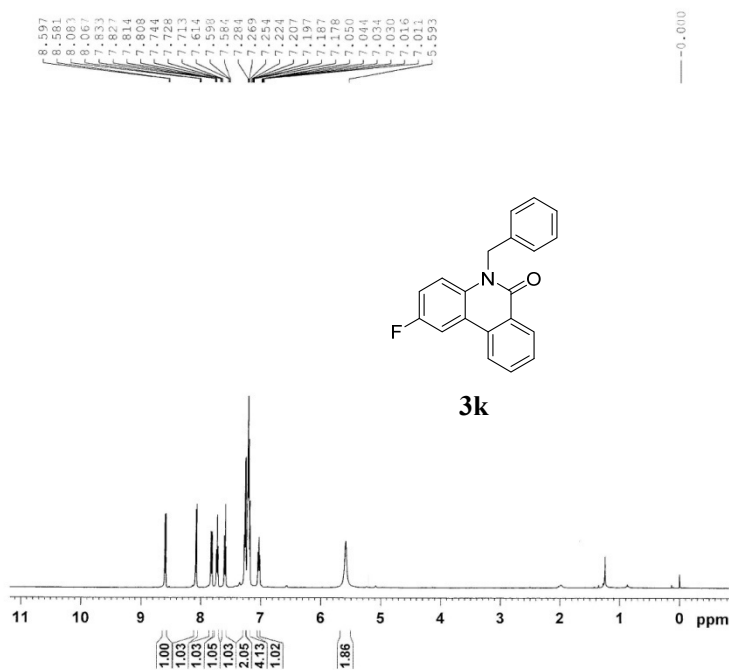
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.14 dB
PL13 16.14 dB
PL12W 12.47038937 W
PL12W 0.38183883 W
PL13W 0.38183883 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578149 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



H-J181
 NAME yangyuan131212
 EXPNO 1
 PROCNO 1
 Date 20131214
 Time 15.49
 INSTRUM spect
 PROBHD 5 mm TXI 1H/D-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 36
 DW 48.400 usec
 DE 6.50 usec
 TE 673.2 K
 D1 1.00000000 sec
 TDO 1
 CHANNEL f1
 NUC1 1H
 P1 6.80 usec
 PL1 0.80 dB
 PL1W 13.05810070 W
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1300253 MHz
 WDW no
 SSB 0
 LE 0.00 Hz
 GB 0
 PC 1.00

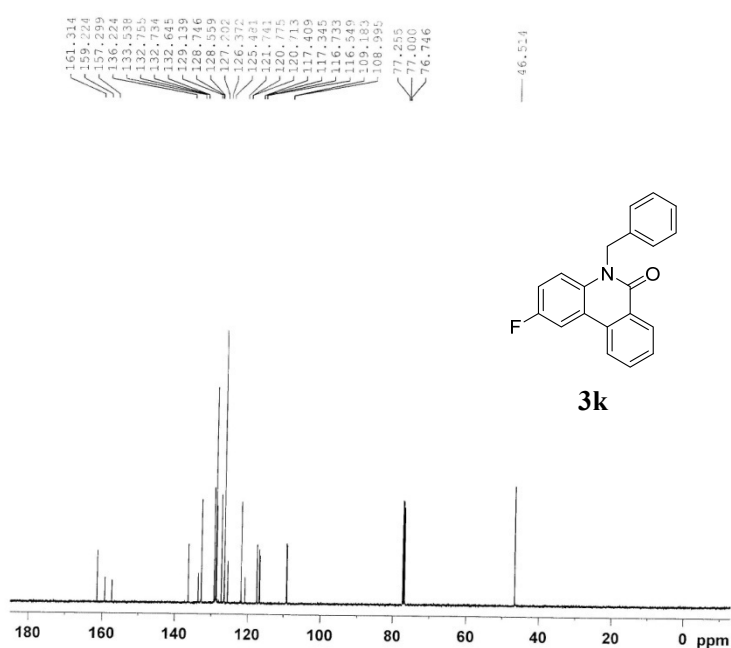


C-J181
 NAME yangyuan131212
 EXPNO 2
 PROCNO 1
 Date 20131214
 Time 15.54
 INSTRUM spect
 PROBHD 5 mm TXI 1H/D-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 116
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 322
 DW 16.800 usec
 DE 6.50 usec
 TE 673.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1
 CHANNEL f1
 NUC1 13C
 P1 11.90 usec
 PL1 -3.50 dB
 PL1W 223.81184387 W
 SFO1 125.7703643 MHz
 CHANNEL f2
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 4.15 dB
 PL12 22.13 dB
 PL12 22.13 dB
 PL12W 6.03761796 W
 PL12W 0.09613468 W
 PL13W 0.09613468 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7578077 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



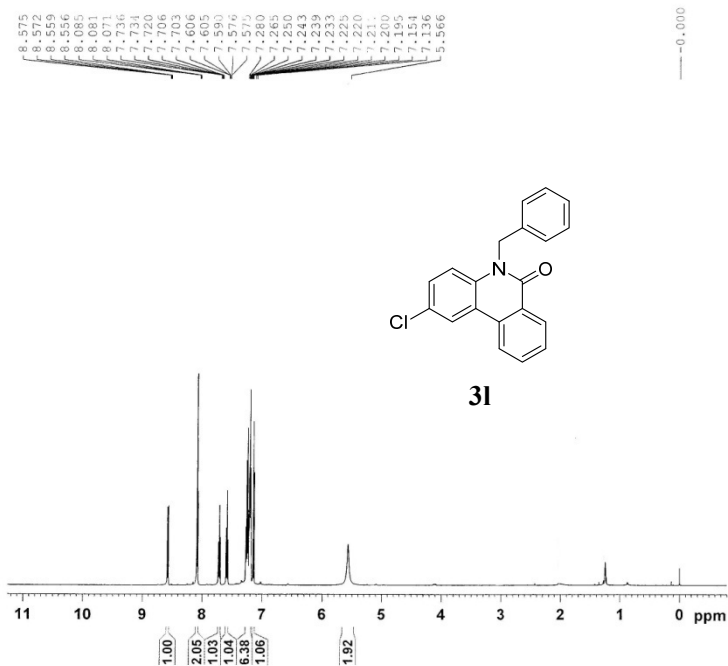
J-H144
NAME yangyuan130920
EXPNO 5
PROCNO 1
Date 20130920
Time 11.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 45.2
DW 48.400 usec
DE 6.50 usec
TE 268.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
PL1W 12.47038937 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300256 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
FC 1.00



J-C144
NAME yangyuan130920
EXPNO 6
PROCNO 1
Date 20130920
Time 11.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 270.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

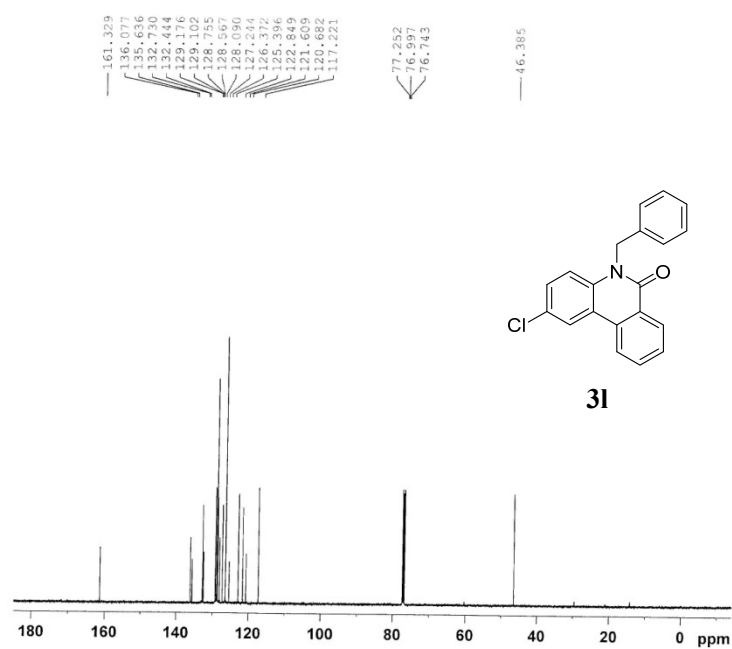
===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 1.00 dB
PL1W 79.41143799 W
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.14 dB
PL13 16.14 dB
PL1W 12.47038937 W
PL12W 0.38183883 W
PL13W 0.38183883 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578086 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
FC 1.40



J-H145

NAME yangyuan130920
EXPNO 7
PROCNO 1
Date 20130920
Time 11.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 50.8
DW 48.400 usec
DE 6.50 usec
TE 268.9 K
D1 1.00000000 sec
TDO 1

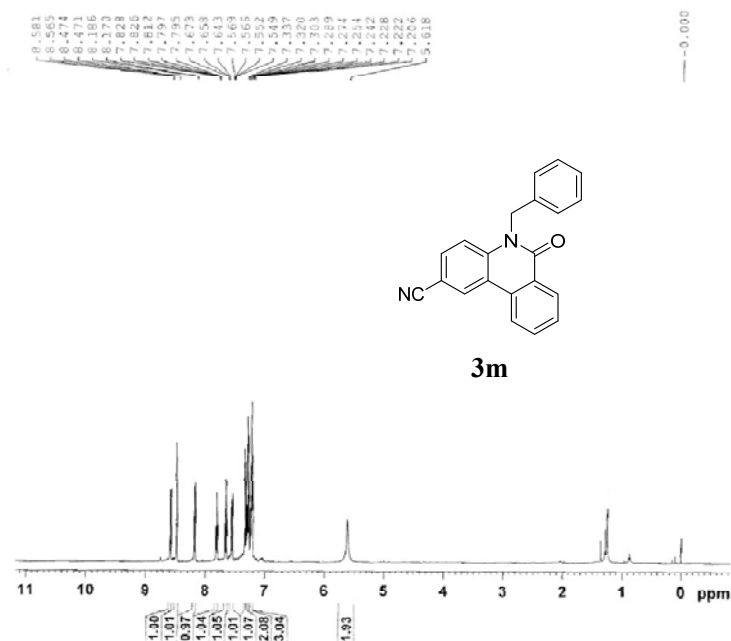
===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
PL1W 12.47038937 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300252 MHz
WDW RO
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



J-C145

NAME yangyuan130920
EXPNO 8
PROCNO 1
Date 20130920
Time 11.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 96
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 270.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

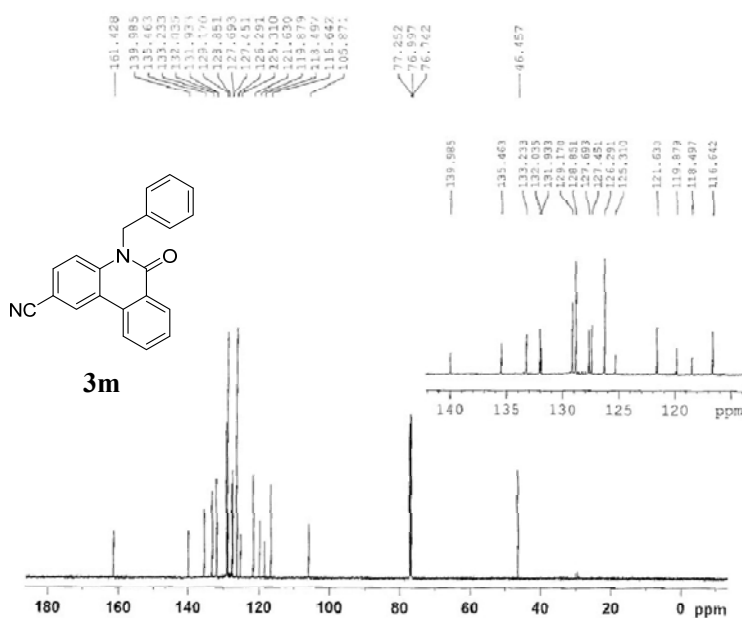
===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 1.00 dB
PL1W 79.41143799 W
SFO1 125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.14 dB
PL13 16.14 dB
PL1W 12.47038937 W
PL12W 0.38183883 W
PL13W 0.38183883 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7578086 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



J-H159

NAME yangyuan131006
EXPNO 1
PROCNO 1
Date_ 20131006
Time 19.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 50.8
DW 48.400 usec
DE 6.30 usec
TE 267.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
PL1W 12.47038937 W
SFO1 500.130885 MHz
SI 32768
SF 500.1308148 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

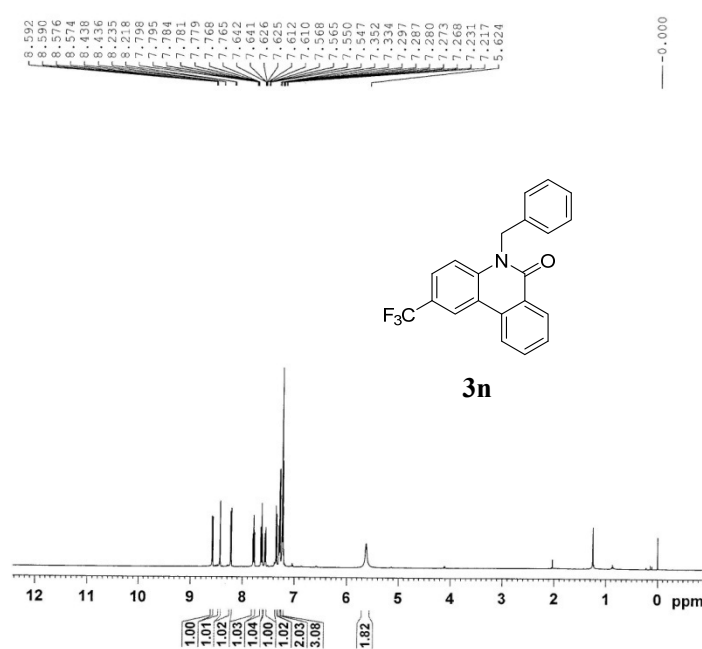


J-C159

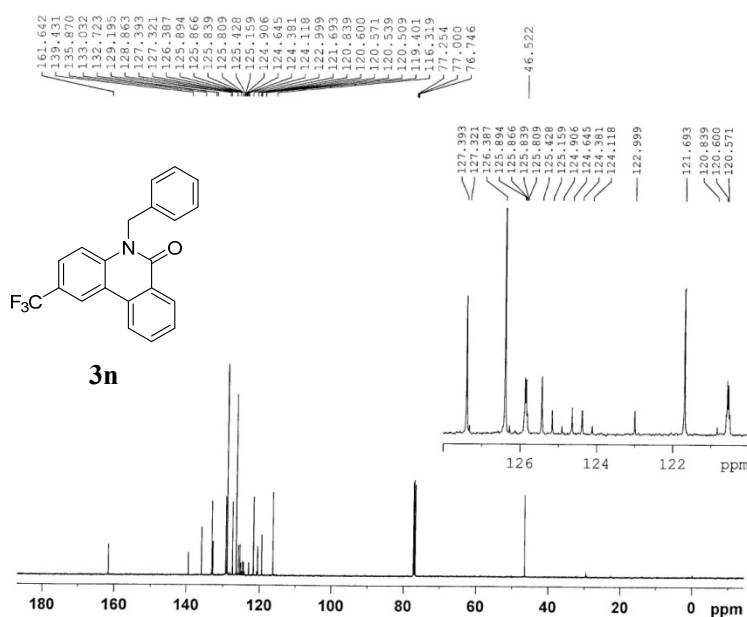
NAME yangyuan131006
EXPNO 2
PROCNO 1
Date_ 20131006
Time 19.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 151
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2030
DW 16.800 usec
DE 6.50 usec
TE 269.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 1.00 dB
PL1W 79.41145799 W
SFO1 125.7703643 MHz

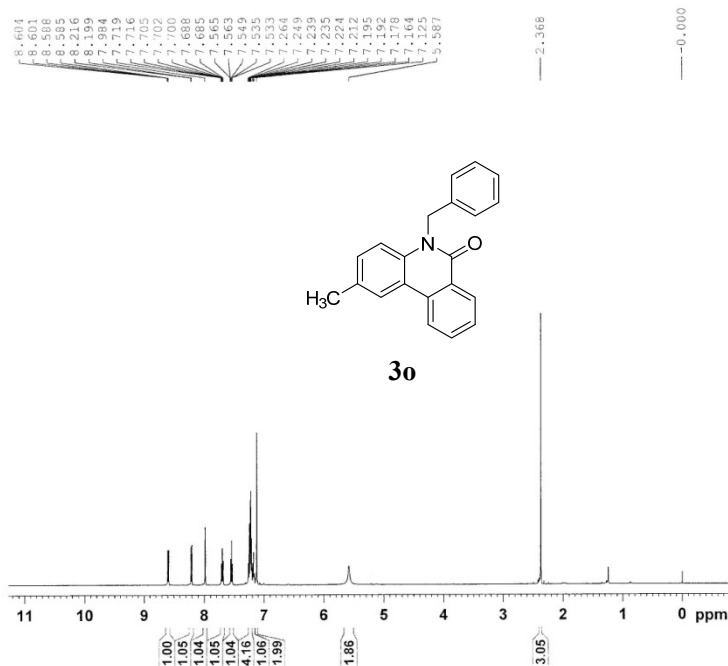
===== CHANNEL f2 =====
CPDPRG2 waltz16
WOC2 18
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.14 dB
PL13 16.14 dB
PL2W 12.47038937 W
PL12W 0.38183883 W
PL13W 0.38183883 W
SFO2 500.1320002 MHz
SI 32768
SF 125.7578122 MHz
WDW RM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



J-H151
 NAME yangyuan130927
 EXPNO 1
 PROCNO 1
 Date_ 20130927
 Time 16.06
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 50.8
 DW 48.400 usec
 DE 6.50 usec
 TE 268.5 K
 D1 1.0000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 1.00 dB
 PL1W 12.47038937 W
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1300273 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00



J-C151
 NAME yangyuan130927
 EXPNO 2
 PROCNO 1
 Date_ 20130927
 Time 16.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 170
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 270.2 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 1.00 dB
 PL1W 79.41143799 W
 SFO1 125.7703643 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.00 dB
 PL12 16.14 dB
 PL13 16.14 dB
 PL2W 12.47038937 W
 PL12W 0.38183883 W
 PL13W 0.38183883 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7578031 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

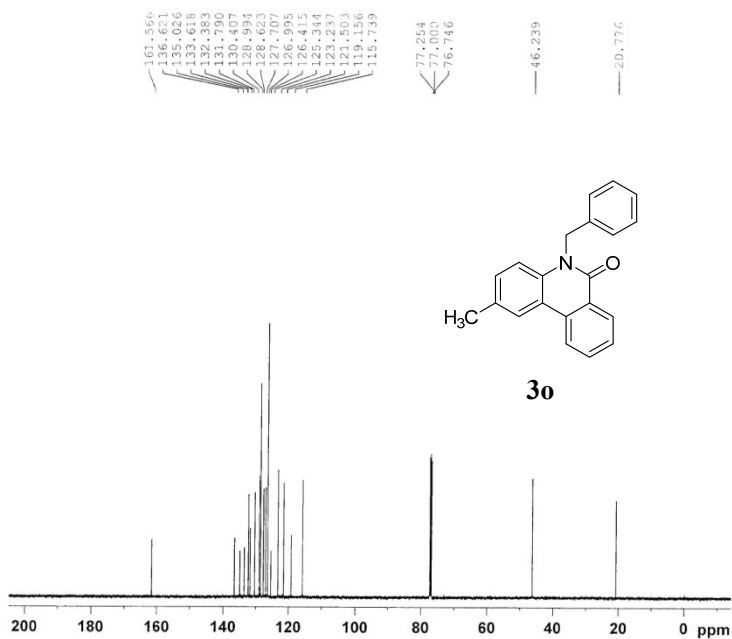


BRUKER
J-H163

```

NAME      yangyuan131010
EXPNO     1
PROCNO    1
Date_     20131010
Time      20.48
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1719923 sec
RG         45.2
DW         48.400 usec
DE         6.50 usec
TE         267.7 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.00 usec
PL1        1.00 dB
PL1W       12.47038937 W
SFO1       500.1330885 MHz
SI         32768
SF         500.1300358 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
  
```



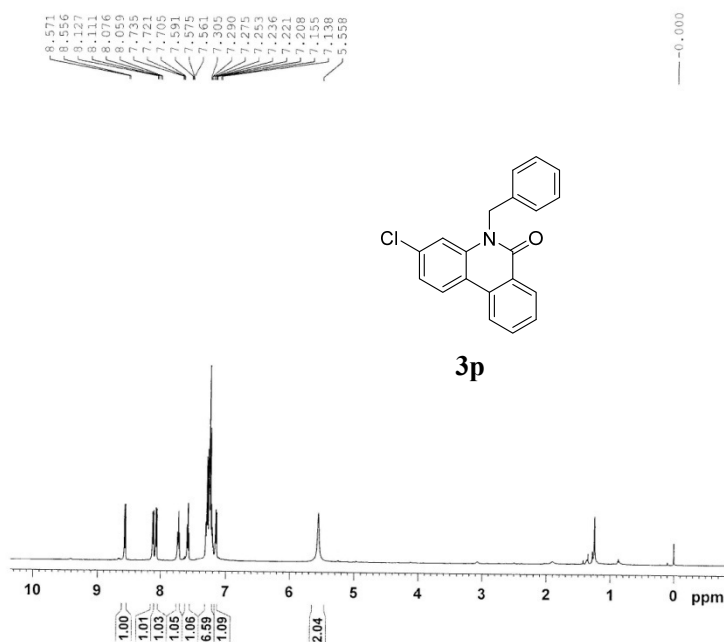
BRUKER
J-C163

```

NAME      yangyuan131010
EXPNO     2
PROCNO    1
Date_     20131010
Time      20.52
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         92
DS         4
SWH        29761.904 Hz
FIDRES     0.454131 Hz
AQ         1.1010548 sec
RG         2050
DW         16.800 usec
DE         6.50 usec
TE         269.3 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

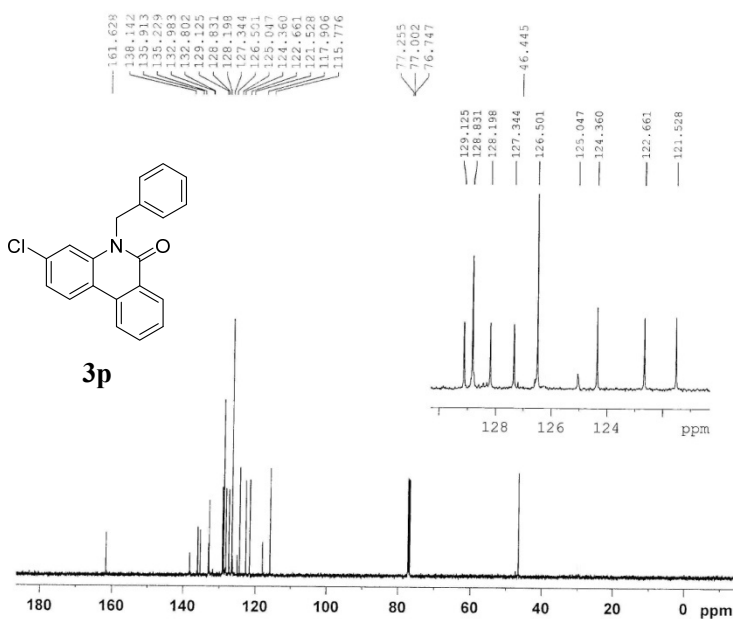
===== CHANNEL f1 =====
NUC1       13C
P1         9.00 usec
PL1        1.00 dB
PL1W       79.41143799 W
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        1.00 dB
PL12       16.14 dB
PL13       16.14 dB
PL2W       12.47038937 W
PL12W      0.38183883 W
PL13W      0.38183883 W
SFO2       500.1320005 MHz
SI         32768
SF         125.7578113 MHz
WDW        EM
SSB        0
TE         1.00 usec
  
```



NAME yangyuan130927
EXPNO 5
PROCNO 1
Date 20130927
Time 16.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 50.8
DW 48.400 usec
DE 6.50 usec
TE 269.2 K
D1 1.00000000 sec
TD0 1

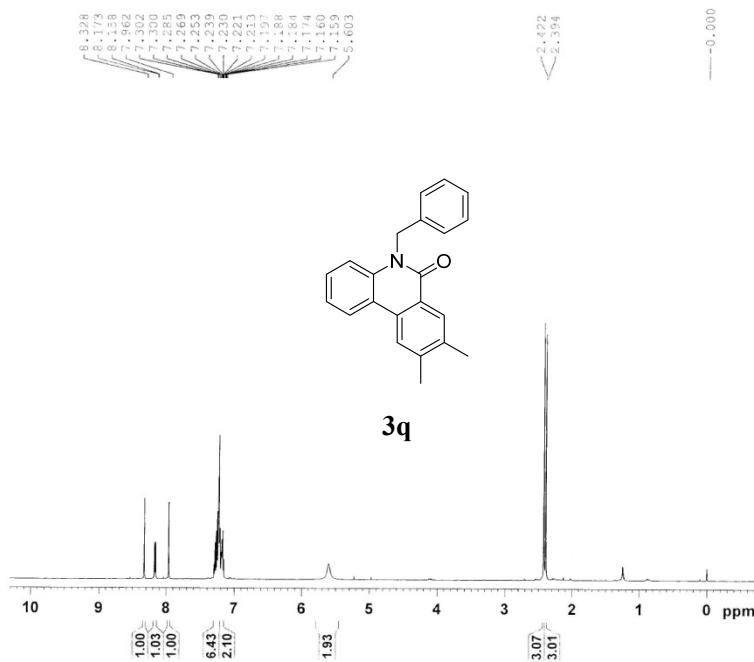
===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 1.00 dB
PL1W 12.47038937 W
SF01 500.1330885 MHz
SI 32768
SF 500.1330235 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



NAME yangyuan130927
EXPNO 6
PROCNO 1
Date 20130927
Time 16.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 112
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 271.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

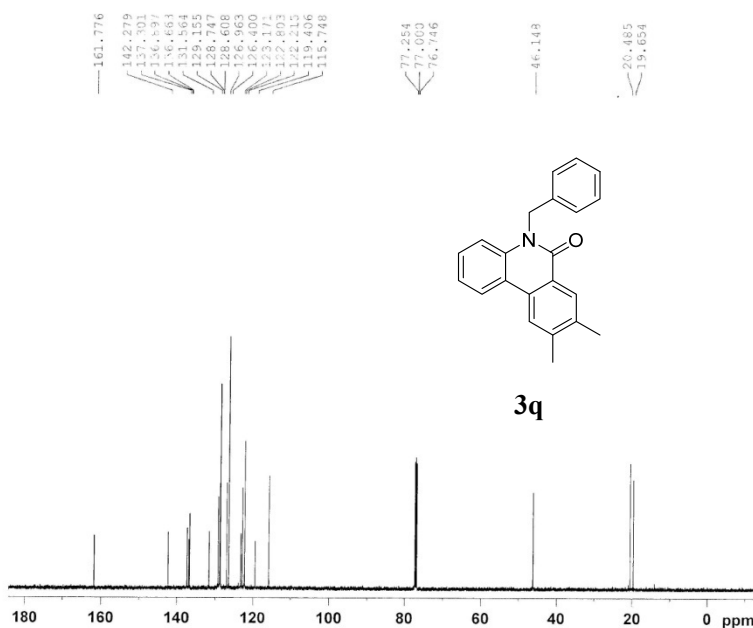
===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 1.00 dB
PL1W 79.41143799 W
SF01 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.14 dB
PL13 16.14 dB
PL2W 12.47038937 W
PL12W 0.38183883 W
PL13W 0.38183883 W
SF02 500.1320005 MHz
SI 32768
SF 125.7578049 MHz
WDW EM
SSB 0
TE 1.00 usec



H-J218
 NAME yangyuan131212
 EXPNO 7
 PROCNO 1
 Date 20131212
 Time 16.49
 INSTRUM spect
 PROBHD 5 mm TXI 1H/D-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 22.6
 DW 48.400 usec
 DE 6.50 usec
 TE 293.2 K
 D1 1.00000000 sec
 TDO 1

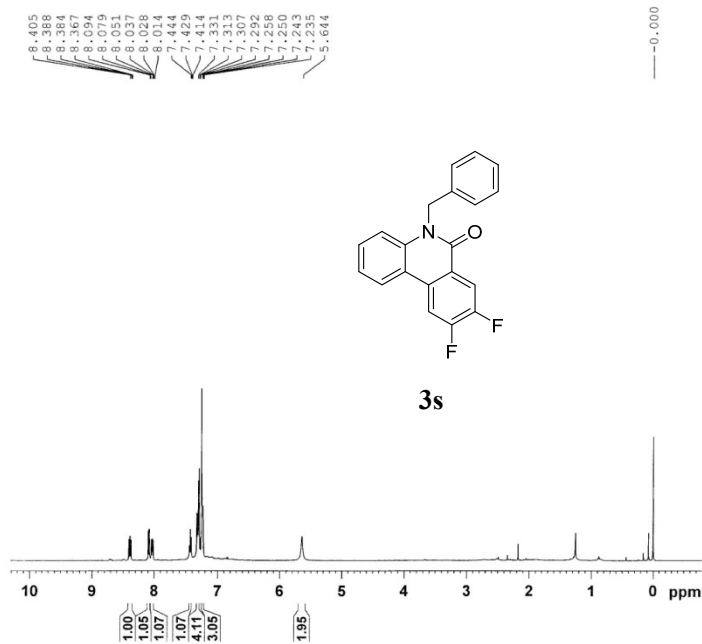
===== CHANNEL f1 =====
 NUC1 1H
 P1 6.80 usec
 PL1 0.80 dB
 PL1W 13.05810070 W
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1330326 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00



C-J218
 NAME yangyuan131212
 EXPNO 8
 PROCNO 1
 Date 20131212
 Time 16.52
 INSTRUM spect
 PROBHD 5 mm TXI 1H/D-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 100
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 322
 DW 16.800 usec
 DE 6.50 usec
 TE 293.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1

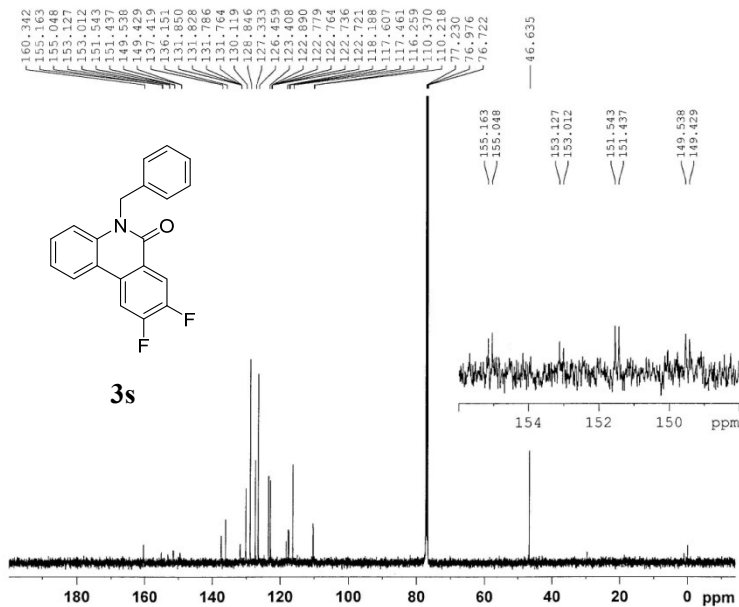
===== CHANNEL f1 =====
 NUC1 13C
 P1 11.90 usec
 PL1 -3.50 dB
 PL1W 223.81184387 W
 SFO1 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 4.15 dB
 PL12 22.13 dB
 PL13 22.13 dB
 PL2W 6.03781796 W
 PL12W 0.09613468 W
 PL13W 0.09613468 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7578131 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



H-J206
 NAME yangyuan131223
 EXPNO 1
 PROCNO 1
 Date_ 20131223
 Time_ 14.44
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 203
 DW 48.400 usec
 DE 6.50 usec
 TE 294.1 K
 D1 1.0000000 sec
 TDO 1

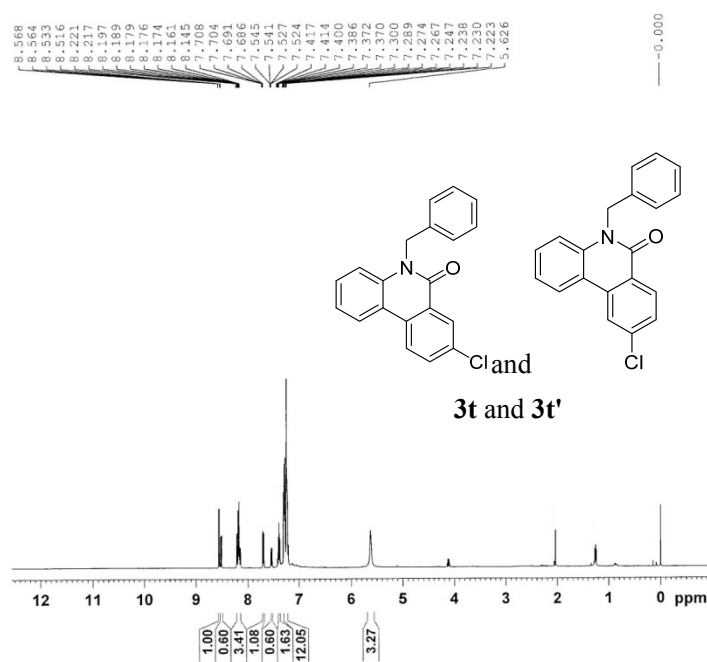
===== CHANNEL f1 =====
 NUC1 1H
 P1 16.00 usec
 PL1 1.00 dB
 PL1W 12.47038937 W
 SF01 500.130085 MHz
 SI 32768
 SF 500.1300141 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00



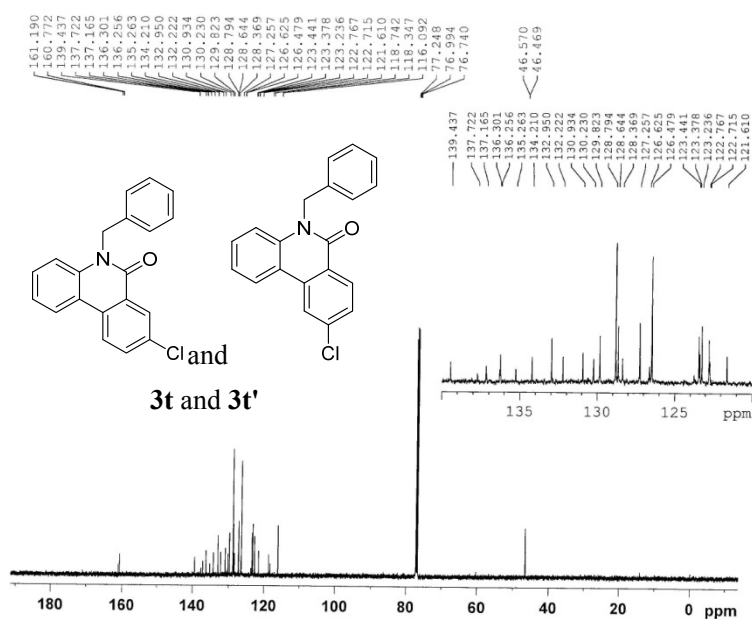
C-J206
 NAME yangyuan131223
 EXPNO 21
 PROCNO 1
 Date_ 20131223
 Time_ 14.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010348 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 296.6 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 1.00 dB
 PL1W 79.41143799 W
 SF01 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PD2 1.00 dB
 PL12 14.98 dB
 PL13 16.14 dB
 PL1W 12.47038937 W
 PL12W 0.49874672 W
 PL13W 0.38183883 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7577935 MHz
 WDW EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



H-J200
 NAME yangyuan131223
 EXPNO 3
 PROCNO 1
 Date_ 20131223
 Time 15.45
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 101
 DW 48.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.00000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 1H
 P1 16.00 usec
 PL1 1.00 dB
 PL1W 12.47038937 W
 SFO1 500.1330885 MHz
 SI 32768
 SF 500.1300197 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00



C-J200
 NAME yangyuan131223
 EXPNO 4
 PROCNO 1
 Date_ 20131223
 Time 15.49
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 164
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TDO 1
 ===== CHANNEL f1 =====
 NUC1 13C
 P1 9.00 usec
 PL1 1.00 dB
 PL1W 79.41143739 W
 SFO1 125.7703643 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.00 dB
 PL12 14.98 dB
 PL13 16.14 dB
 PL1W 12.47038937 W
 PL12W 0.49874472 W
 PL13W 0.38183883 W
 SFO2 500.1320005 MHz
 SI 32768
 SF 125.7577968 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40