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A New Practical Approach Towards the Synthesis of Unsymmetric and Symmetric 1,10-Phenanthroline Derivatives at Room Temperature

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Supporting Information

Table of Contents **Page**

Materials and Methods

8-aminoquinoline was purchased from Aladdin and purified by column chromatography, other commercial materials (Alfa Aesar, Aladdin, J&K Chemical LTD., Beijing Ouhe Technology Co.Ltd.) were used without further purification. All solvents were analytical grade. CH_2Cl_2, MeCN, MeOH, PhMe, DMSO and $ClCH_2CH_2Cl$ were dried according to the procedure described in the fifth edition of <Purafication of Laboratory Chemicals > by W.L.F. Armarego & C.L.L. Chai. [1]The ^1H NMR and ^{13}C NMR spectra were recorded on a JOEL JNM-ECA 300 MHz or BRUKE AVANCE 400 MHz spectrometer in $CDCl_3$ using TMS or solvent peak as a standard. All ^{13}C NMR spectra were recorded with complete proton decoupling. Low-resolution mass spectral analyses were performed with a Waters AQUITY UPLC^{TM}/MS. Analytical TLC was performed on Yantai Chemical Industry Research Institute silica gel 60 F254 plates and flash column chromatography was performed on Qingdao Haiyang Chemical Co. Ltd silica gel 60 (200-300mesh).The rotavapor was BUCHI's Rotavapor R-3. Substituted 3-ethoxycyclobutanones[2],[3] and 2-azidoaniline[4] was prepared according to the literature reports.

General procedure

Ⅰ Synthesis of substituted 8-aminoquinolines

2-azidoaniline (1.0 equiv) and substituted 3-ethoxycyclobutanones (2.0 equiv) were dissolved in anhydrous dichloromethane in a flame-dried round bottom flask which was coated with aluminum foil. Following that, BF_3 -OEt_2 (1.0 equiv) was added into the reaction solution. Then the reaction mixture was stirred at room temperature under atmosphere of Argon for 6 - 12 h. The reaction was monitored by TLC and LC-MS. After completion of the reaction, Et_3N was added to quench the reaction. The reaction mixture was diluted with dichloromethane and washed once with saturated aqueous $NaHCO_3$. Then organic layer was dried over Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally the residue was purified by silical gel column chromatography to give substituted 8-azidoquinoline. then the substituted 8-azidoquinolines were added to a solution of triphenylphosphine in anhydrous methanol. The flask was also coated with aluminum foil. Then, the reaction was stirred at 50°C overnight. After all the starting material had disappeared, the reaction mixture was cooled to room temperature and the solvent was removed under reduced pressure. The residue was purified by silical gel column chromatography to give corresponding substituted 8-aminoquinolines.

Ⅱ Synthesis of 1,10-phenanthrolines

8-aminoquinoline or substituted 8-aminoquinolines (prepared by following the general procedure Ⅰ) (1.0 equiv) and substituted 3-ethoxycyclobutanones (2.0 equiv) were dissolved in anhydrous dichloromethane in a flame-dried round bottom flask. Following that, crushed 4 Å molecular sieves (150%wt) was added quickly, then BF_3 -OEt_2 (1.0 equiv) was dropwised into the reaction solution. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48 h. The reaction was monitored by TLC and LC-MS. After 48h, Et_3N was added to quench the reaction. The reaction mixture was diluted with dichloromethane and filtered through SiO_2 then washed once with saturated aqueous $NaHCO_3$. Then organic layer was dried over Na_2SO_4 and concentrated on rotavapor under reduced pressure. Finally the residue was purified by silical gel column chromatography (Petroleum ether : EtOAc = 2 : 1, then DCM : MeOH = 80 : 1) to give corresponding 1,10-phenanthroline.

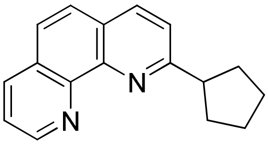
References:

[1] Wilfred L.F. Armarego .Christina L.L. Chai. <*Purification of Laboratory Chemicals*>, **fifth edition**.

[2] J. Matsuo, S. Sasaki, T. Hoshikawa, H. Ishibashi. *Org. Lett*. 2009. **11**. 3822.

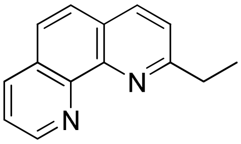
[3] J. Matsuo, R. Okuno, K. Takeuchi, M. Kawano, H. Ishibashi. *Tetrahedron Lett*. 2010. **51**. 3736.

[4] M. Shen, T. G. Driver. *Org. Lett.* 2008. **10** (15). 3367



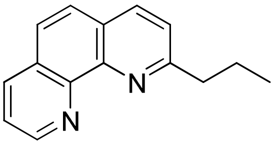
2-cyclopentyl-1,10-phenanthroline (3)

Following the general procedure Ⅱ, 3-ethoxy-spiro[3.4]octan-1-one (47 mg, 0.28 mmol), 8-aminoquinoline (20 mg, 0.14 mmol), BF_3 -OEt_2 (1.0 equiv, 0.14 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **3** (30 mg, colorless oil) was isolated in 87% yield.^{1}H-NMR (300 MHz, $CDCl_3$) δ (ppm) 9.21 (dd, *J* = 1.59 Hz, *J* = 4.32 Hz, 1H), 8.23 (dd, *J* = 1.62 Hz, *J* = 8.04 Hz, 1H), 8.17 (d, *J* = 8.37 Hz, 1H), 7.76 (d, *J* = 8.76 Hz, 1H), 7.72 (d, *J* = 8.76 Hz, 1H), 7.61 - 7.57 (m, 2H), 3.78 - 3.73 (m, 1H), 2.35 - 2.31 (m, 2H), 1.91 - 1.82 (m, 6H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) 167.4, 150.4, 146.2, 145.3, 136.5, 136.1, 128.9, 127.1, 126.5, 125.6, 122.7, 120.9, 49.5, 34.8, 26.3; MS (ESI) calcd for $C_{17}H_{16}N_2$ $[M+H]^+$: 249.13, found 249.07



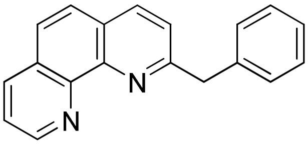
2-ethyl-1,10-phenanthroline (4)

Following the general procedure Ⅱ, 3-ethoxy-2-methylcyclobutanone (70 mg, 0.54 mmol), 8-aminoquinoline (39 mg, 0.27 mmol), BF_3 -OEt_2 (1.0 equiv, 0.27 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **4** (43 mg, colorless oil) was isolated in 76% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.30 (d, *J* = 4.36 Hz, 1H), 8.32 (d, *J* = 8.12 Hz, 1H), 8.22 (d, *J* = 8.24 Hz, 1H), 7.83 (d, *J* = 8.76 Hz, 1H), 7.78 (d, *J* = 8.76 Hz, 1H), 7.68 (dd, *J* = 4.48 Hz, *J* = 8.04 Hz, 1H), 6.62 (d, *J* = 8.24 Hz, 1H), 3.25 (q, *J* = 7.64 Hz, 2H), 1.46 (t, *J* = 7.64 Hz, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 164.7, 150.1, 145.8, 145.4, 136.4, 136.3, 128.8, 126.9, 126.5, 125.4, 122.7, 122.3, 32.5, 14.5; MS (ESI) calcd for $C_{14}H_{12}N_2$ $[M+H]^+$: 209.10, found 209.00



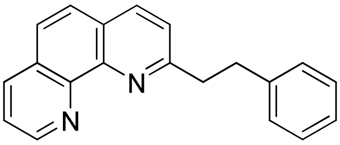
2-propyl-1,10-phenanthroline (5)

Following the general procedure Ⅱ, 3-ethoxy-2-ethylcyclobutanone (30 mg, 0.21 mmol), 8-aminoquinoline (15 mg, 0.10 mmol), BF_3 -OEt_2 (1.0 equiv, 0.10 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **5** (19 mg, colorless oil) was isolated in 82% yield.^{1}H-NMR (300 MHz, $CDCl_3$) δ (ppm) 9.22 (d, *J* = 4.38 Hz, 1H), 8.23 (d, *J* = 8.04 Hz, 1H), 8.16 (d, *J* = 8.16 Hz, 1H), 7.77 (d, *J* = 8.82 Hz, 1H), 7.72 (d, *J* = 8.73 Hz, 1H), 7.60 (dd, *J* = 4.29 Hz, *J* = 7.92 Hz, 1H), 7.54 (d, *J* = 8.22 Hz , 1H) 3.19 (t, *J* = 7.56 Hz, 2H), 1.93 - 1.89 (m, 2H), 1.08 (t, *J* = 6.54 Hz, 3H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) 163.6, 150.4, 146.2, 145.9, 136.3, 136.1, 128.9, 127.0, 126.6, 125.6. 122.9, 122.7, 41.6, 23.8, 14.2; MS (ESI) calcd for $C_{15}H_{14}N_2$ $[M+H]^+$: 223.12, found 223.00



2-benzyl-1,10-phenanthroline (6)

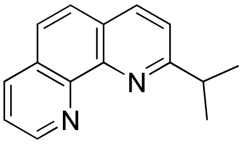
Following the general procedure Ⅱ, 3-ethoxy-2-phenylcyclobutanone (66 mg, 0.35 mmol), 8-aminoquinoline (25 mg, 0.17 mmol), BF_3 -OEt_2 (1.0 equiv, 0.17 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **6** (40 mg, yellow oil) was isolated in 85% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.27 (dd, *J* = 1.64 Hz, *J* = 4.36 Hz, 1H), 8.26 (dd, *J* = 1.04 Hz, *J* = 8.04 Hz, 1H), 8.10 (d, *J* = 8.28 Hz, 1H), 7.75 (s, 2H), 7.64 (dd, *J* = 4.36 Hz, *J* = 8.04 Hz, 1H), 7.38 - 7.32 (m, 5H), 7.25 (t, *J* = 7.00 Hz, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 162.2, 150.1, 145.7, 145.3, 139.5, 136.5, 129.6, 128.9, 128.7, 127.1, 126.6, 126.5, 125.8, 123.3, 122.9, 45.7; MS (ESI) calcd for $C_{19}H_{14}N_2$ $[M+H]^+$: 271.12, found 271.02



2-phenethyl-1,10-phenanthroline (7)

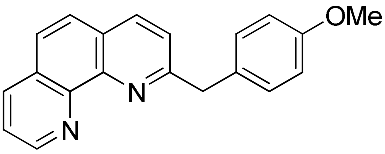
Following the general procedure Ⅱ, 3-ethoxy-2-benzylcyclobutanone (71 mg, 0.35 mmol), 8-aminoquinoline (25 mg, 0.17 mmol), BF_3 -OEt_2 (1.0 equiv, 0.17 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **7** (33 mg, yellow oil) was isolated in 67% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.24 (dd, *J* = 1.72 Hz, *J* =

4.36 Hz, 1H), 8.24 (dd, *J* = 1.56 Hz, *J* = 7.76 Hz, 1H), 8.13 (d, *J* = 8.20 Hz, 1H), 7.77 (d, *J* = 8.76 Hz, 1H), 7.74 (d, *J* = 8.76 Hz, 1H), 7.62 (dd, *J* = 4.36 Hz, *J* = 8.04 Hz, 1H), 7.46 (d, *J* = 8.20 Hz, 1H), 7.30 - 7.29 (m, 4H), 7.23 - 7.21 (m, 1H), 3.57 - 3.53 (m, 2H), 3.26 - 3.22 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 162.4, 150.4, 146.2, 145.9, 141.6, 136.3, 136.1, 128.9, 128.6, 128.4, 127.1, 126.5, 126.0, 125.7, 123.1, 122.8, 41.1, 36.5; MS (ESI) calcd for $C_{20}H_{16}N_2$ $[M+H]^+$: 285.13, found 285.02



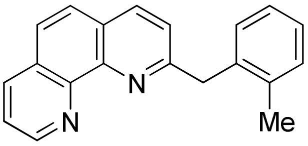
2-isopropyl-1,10-phenanthroline (8)

Following the general procedure Ⅱ, 3-ethoxy-2,2-dimethylcyclobutanone (49 mg, 0.35 mmol), 8-aminoquinoline (25 mg, 0.17 mmol), BF_3 -OEt_2 (1.0 equiv, 0.17 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **8** (29 mg, colorless oil) was isolated in 75% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.20 - 9.18 (m, *J* = 1H), 8.16 - 8.12 (m, 2H), 7.70 - 7.63 (m, 2H), 7.56 - 7.52 (m, 2H), 3.68 - 3.61 (m, 1H), 1.43 (d, *J* = 6.96 Hz, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 168.5, 150.2, 146.2, 145.3, 136.6, 135.9, 128.8, 127.1, 126.4, 125.5, 122.6, 120.0, 37.7, 23.2; MS (ESI) calcd for $C_{15}H_{14}N_2$ $[M+H]^+$: 223.12, found 223.03



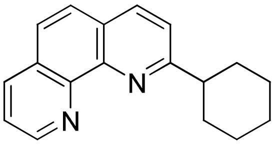
2-(4-methoxybenzyl)-1,10-phenanthroline (9)

Following the general procedure Ⅱ, 3-ethoxy-2-(4-methoxy-phenyl)-cyclobutanone (122 mg, 0.56 mmol), 8-aminoquinoline (40 mg, 0.28 mmol), BF_3-OEt_2 (1.0 equiv, 0.28 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **9** (62 mg, slight yellow oil) was isolated in 74% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.27 (dd, *J* = 1.76 Hz, *J* = 4.36 Hz, 1H), 8.27 (dd, *J*=1.76 Hz, *J* = 8.04 Hz, 1H), 8.12 (d, *J* = 8.28 Hz, 1H), 7.77 (s, 2H), 7.65 (dd, *J* = 4.36 Hz, *J* = 8.08 Hz, 1H), 7.38 (d, *J* = 8.32 Hz, 1H), 7.29 (d, *J* = 8.92 Hz, 2H), 6.89 (d, *J* = 8.68 Hz, 2H), 4.61 (s, 2H), 3.82 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 162.5, 158.3, 150.4, 146.2, 145.6, 136.4, 136.1, 131.7, 130.6, 128.9, 127.0, 126.5, 125.8, 123.1, 122.8, 114.1, 55.3, 44.8; MS (ESI) calcd for $C_{20}H_{16}N_2O$ $[M+H]^+$: 301.13, found 301.19



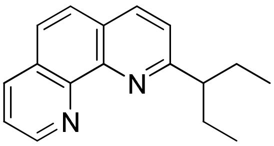
2-(2-methylbenzyl)-1,10-phenanthroline (10)

Following the general procedure Ⅱ, 3-ethoxy-2-o-tolyl-cyclobutanone (113 mg, 0.56 mmol), 8-aminoquinoline (40 mg, 0.28 mmol), BF$_3$ -OEt$_2$ (1.0 equiv, 0.28 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **10** (60 mg, slight yellow oil) was isolated in 76% yield.^{1}H-NMR (400 MHz, CDCl$_3$) δ (ppm) 9.17 (dd, *J* = 1.76 Hz, *J* = 4.36 Hz, 1H), 8.09 (dd, *J*=1.76 Hz, *J* = 8.08 Hz, 1H), 7.93 (d, *J* = 8.28 Hz, 1H), 7.59 (s, 2H), 7.50 (dd, *J* = 4.36 Hz, *J* = 8.04 Hz, 1H), 7.17 - 7.09 (m, 6H), 4.62 (s, 2H), 2.22 (s, 3H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ(ppm) 161.7, 150.3, 146.1, 145.7, 137.5, 136.5, 136.0, 130.5, 128.8, 126.9, 126.4, 126.2, 125.7, 122.7, 122.3, 43.5, 19.9; MS (ESI) calcd for $C_{20}H_{16}N_2$ $[M+H]^+$: 285.13, found 285.12



2-cyclohexyl-1,10-phenanthroline (11)

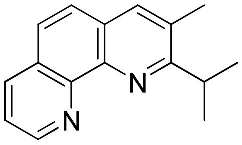
Following the general procedure Ⅱ, 3-ethoxy-spiro[3.5]nonan-1-one (38 mg, 0.21 mmol), 8-aminoquinoline (15 mg, 0.10 mmol), BF$_3$ -OEt$_2$ (1.0 equiv, 0.10 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **11** (18 mg, colorless oil) was isolated in 66% yield.^{1}H-NMR (400 MHz, CDCl$_3$) δ (ppm) 9.25 (dd, *J* = 1.76 Hz, *J* = 4.36 Hz, 1H), 8.25 (dd, *J* = 1.52 Hz, *J* = 8.04 Hz, 1H), 8.20 (d, *J* = 8.32 Hz, 1H), 7.78 (d, *J* = 8.76 Hz, 1H), 7.74 (d, *J* = 8.72 Hz, 1H), 7.62 (dd, *J* = 4.36 Hz, *J* = 8.00 Hz, 1H), 7.60 (d, *J* = 8.36 Hz, 1H), 3.40 - 3.34 (m, 1H), 2.17 - 2.13 (m, 2H), 1.93 - 1.89 (m, 2H), 1.86 - 1.82 (m, 1H), 1.72 - 1.59 (m, 2H), 1.58 - 1.48 (m, 2H), 1.42 - 1.36 (m, 1H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ (ppm) 167.6, 150.4, 146.3, 145.5, 136.5, 136.0, 128.8, 127.2, 126.5, 125.5, 122.6, 120.6, 47.9, 33.4, 26.5, 26.2; MS (ESI) calcd for $C_{18}H_{18}N_2$ $[M+H]^+$: 263.15, found 263.04



2-(pentan-3-yl)-1,10-phenanthroline (12)

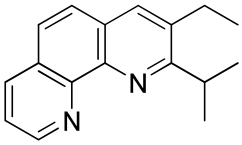
Following the general procedure Ⅱ, 3-ethoxy-2,2-diethylcyclobutanone (38 mg, 0.22 mmol), 8-aminoquinoline (16 mg, 0.11 mmol), BF$_3$ -OEt$_2$ (1.0 equiv, 0.11 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **12** (22 mg, yellow oil) was isolated in 79% yield.^{1}H-NMR (400 MHz, CDCl$_3$) δ (ppm) 9.27 (dd, *J* = 1.76 Hz, *J* = 4.36 Hz, 1H), 8.24 (dd, *J* = 1.64 Hz, *J* = 8.04 Hz, 1H), 8.19 (d, *J* = 8.32 Hz, 1H), 7.78 (d, *J* = 8.72 Hz, 1H), 7.74 (d, *J* = 8.76 Hz, 1H), 7.61 (dd, *J* = 4.36 Hz, *J* = 8.04 Hz, 1H), 7.53 (d, *J* = 8.32

Hz, 1H), 3.30 - 3.23 (m, 1H), 1.94 - 1.81 (m, 4H), 0.90 (t, *J* = 7.40 Hz, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 166.8, 150.4, 146.4, 145.7, 136.1, 136.0, 128.9, 127.2, 126.5, 125.5, 122.6, 121.0, 51.7, 28.2, 12.1; MS (ESI) calcd for $C_{17}H_{18}N_2$ [M+H]$^+$: 251.15, found 251.03



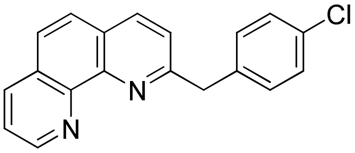
2-isopropyl-3-methyl-1,10-phenanthroline (13)

Following the general procedure Ⅱ, 3-ethoxy-2,2,4-trimethylcyclobutanone (44 mg, 0.28 mmol), 8-aminoquinoline (20 mg, 0.14 mmol), BF_3 -OEt_2 (1.0 equiv, 0.14 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **13** (16 mg, white solid) was isolated in 50% yield.^{1}H-NMR (300 MHz, $CDCl_3$) δ (ppm) 9.20 (d, *J* = 4.38 Hz,1H), 8.19 (d, *J* = 8.01 Hz, 1H), 7.91 (s, 1H), 7.69 (s, 2H), 7.56 (dd, *J* = 4.41 Hz, *J* = 8.04Hz, 1H), 3.59 - 3.54 (m, 1H), 2.59 (s, 3H), 1.55 (d, *J* = 6.84 Hz, 6H); ^{13}C-NMR (75 MHz, $CDCl_3$) δ (ppm) 166.5, 150.4, 146.6, 144.1, 136.7, 136.1, 131.0, 128.5, 127.4, 126.2, 125.6, 122.2, 33.3, 21.8, 19.3; MS (ESI) calcd for $C_{16}H_{16}N_2$ [M+H]$^+$: 237.13, found 237.07



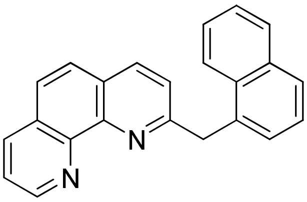
3-ethyl-2-isopropyl-1,10-phenanthroline (14)

Following the general procedure Ⅱ, 3-ethoxy-4-ethyl-2,2-dimethylcyclobutanone (71 mg, 0.42 mmol), 8-aminoquinoline (30 mg, 0.21 mmol), BF_3 -OEt_2 (1.0 equiv, 0.21 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **14** (27 mg, white solid) was isolated in 52% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 9.24 (dd, *J* = 1.76 Hz, *J* = 4.36 Hz, 1H), 8.22 (dd, *J* = 1.76 Hz, *J* = 8.04 Hz, 1H), 7.97 (s, 1H), 7.75 (d, *J* = 8.76 Hz, 1H), 7.70 (d, *J* = 8.72 Hz, 1H), 7.58 (dd, *J* = 4.36 Hz, *J* = 8.00 Hz, 1H), 3.65 - 3.58 (m, 1H), 2.97 (q, *J* = 7.44 Hz, 2H), 1.57 (d, *J* = 6.80 Hz, 6H), 1.40 (t, *J* = 7.48 Hz, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 166.2, 150.3, 146.5, 144.0, 136.6, 136.0, 134.7, 128.5, 127.5, 126.4, 125.5, 122.1, 32.3, 25.2, 22.5, 15.0; MS (ESI) calcd for $C_{17}H_{18}N_2$ [M+H]$^+$: 251.15, found 251.03



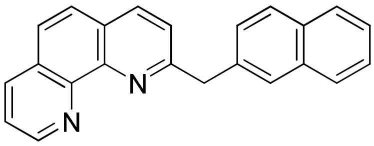
2-(4-chlorobenzyl)-1,10-phenanthroline (15)

Following the general procedure Ⅱ, 2-(4-chloro-phenyl)-3-ethoxy-cyclobutanone (125 mg, 0.56 mmol), 8-aminoquinoline (40 mg, 0.28 mmol), BF_3 -OEt_2 (1.0 equiv, 0.28 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **15** (54 mg, slight yellow oil) was isolated in 64% yield.^{1}H-NMR (400 MHz, CDCl$_3$) δ (ppm) 9.26 (dd, *J* = 1.76 Hz, *J* = 4.36 Hz, 1H), 8.27 (dd, *J* = 1.76 Hz, *J* = 8.08 Hz, 1H), 8.13 (d, *J* = 8.28 Hz, 1H), 7.77 (s, 2H), 7.65 (dd, *J* = 4.40 Hz, *J* = 8.08 Hz, 1H), 7.35 (d, *J* = 8.28 Hz, 1H), 7.29 (s, 4H), 4.61 (s, 2H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ (ppm) 161.4, 150.5, 146.1, 145.7, 138.0, 136.7, 136.1, 132.4, 130.9, 128.9, 128.8, 127.1, 126.4, 126.1, 123.0, 122.9, 45.0; MS (ESI) calcd for C$_{19}$H$_{13}$ClN$_2$ [M+H]$^+$: 305.08, found 305.04



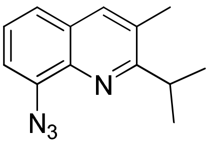
2-(naphthalen-1-ylmethyl)-1,10-phenanthroline (16)

Following the general procedure Ⅱ, 3-ethoxy-2-naphthalen-1-yl-cyclobutanone (133 mg, 0.56 mmol), 8-aminoquinoline (40 mg, 0.28 mmol), BF$_3$-OEt$_2$ (1.0 equiv, 0.28 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 40 : 1). Finally, compound **16** (42 mg, slight yellow oil) was isolated in 47% yield.^{1}H-NMR (400 MHz, CDCl$_3$) δ (ppm) 9.30 (dd, *J* = 1.68 Hz, *J* = 4.36 Hz, 1H), 8.27 (dd, *J* = 1.72 Hz, *J* = 8.08 Hz, 1H), 8.07 (d, *J* = 8.44 Hz, 1H), 7.99 (d, *J* = 8.32 Hz, 1H), 7.88 (d, *J* = 8.08 Hz, 1H), 7.83 (dd, *J* = 1.84 Hz, *J* = 7.20 Hz,1H), 7.76 (d, *J* = 8.76 Hz, 1H), 7.72 (d, *J* = 8.76 Hz, 1H), 7.66 (dd, *J* = 4.36 Hz, *J* = 8.04 Hz, 1H), 7.43-7.52 (m, 3H), 7.38-7.40 (m, 1H), 7,16 (d, *J* = 8.32 Hz, 1H), 5.12 (s, 2H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ (ppm) 162.0, 150.5, 146.2, 145.8, 136.5, 136.1, 135.3, 134.1, 132.4, 128.9, 128.6, 128.2, 127.6, 127.0, 126.5, 126.2, 125.8, 125.7, 125.7, 124.9, 122.9, 122.6, 43.4; MS (ESI) calcd for C$_{23}$H$_{16}$N$_2$ [M+H]$^+$: 321.13, found 321.11



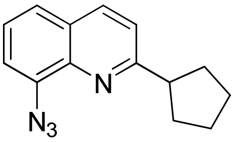
2-(naphthalen-2-ylmethyl)-1,10-phenanthroline (17)

Following the general procedure Ⅱ, 3-ethoxy-2-naphthalen-2-yl-cyclobutanone (133 mg, 0.56 mmol), 8-aminoquinoline (40 mg, 0.28 mmol), BF$_3$-OEt$_2$ (1.0 equiv, 0.28 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (DCM : MeOH = 80 : 1). Finally, compound **17** (39 mg, slight yellow oil) was isolated in 44% yield.^{1}H-NMR (400 MHz, CDCl$_3$) δ (ppm) 9.26 (dd, *J* = 1.68 Hz, *J* = 4.32 Hz, 1H), 8.20 (dd, *J* = 1.68 Hz, *J* = 8.08 Hz, 1H), 8.03 (d, *J* = 8.32 Hz, 1H), 7.76-7.80 (m, 4H), 7.70 (d, *J* = 1.44 Hz, 2H), 7.60 (dd, *J* = 4.36 Hz, *J* = 8.08 Hz,1H), 7.41-7.47 (m, 3H), 7.35 (d, *J* = 8.28 Hz ,1H), 4.80 (s, 2H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ (ppm) 161.9, 150.4, 146.2, 145.7, 137.1, 136.5, 136.1, 133.7, 132.3, 128.9, 128.3, 128.0, 127.7, 127.6, 127.1, 126.5, 126.1, 125.9, 125.6, 123.3, 122.8, 45.9; MS (ESI) calcd for $C_{23}H_{16}N_2$ $[M+H]^+$: 321.13, found 321.07



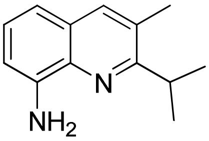
8-azido-2-isopropyl-3-methylquinoline (20)

Following the general procedure Ⅰ , 3-ethoxy-2,2,4-trimethylcyclobutanone (47 mg, 0.30 mmol), 2-azidoaniline(20 mg, 0.15 mmol) and BF_3 -OEt_2 (1.0 equiv, 0.15 mmol) were used. The flask was coated with aluminum foil. The reaction mixture was stirred at room temperature under atmosphere of Argon for 12h. After completion of the reaction, the residue was purified by silical gel column chromatography (Petroleum ether : EtOAc = 80 : 1). Finally, compound **20** (16 mg, colorless oil) was isolated in 48% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 7.83 (s, 1H), 7.44 (dd, *J* = 1.24 Hz, *J* = 8.12 Hz, 1H), 7.31 (t, *J* = 7.60 Hz, 1H), 7.13 (dd, *J* = 1.28 Hz, *J* = 7.48 Hz, 1H), 3.45 - 3.38 (m, 1H), 2.51 (s, 3H), 1.37 (d, *J* = 6.72Hz, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 165.8, 141.0, 136.4, 136.2, 129.7, 128.1, 125.6, 123.0, 118.0, 32.8, 21.8, 19.2; MS (ESI) calcd for $C_{13}H_{14}N_4$ $[M+H–N_2]^+$:199.12 , found 199.05



8-azido-2-cyclopentylquinoline (21)

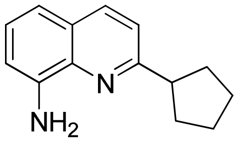
Following the general procedure Ⅰ , 3-ethoxyspiro[3.4]octan-1-one (75 mg, 0.45 mmol), 2-azidoaniline(30 mg, 0.22 mmol) and BF_3 -OEt_2 (1.0 equiv, 0.22 mmol) were used. The flask was coated with aluminum foil. The reaction mixture was stirred at room temperature under atmosphere of Argon for 6h. After completion of the reaction, the residue was purified by silical gel column chromatography (Petroleum ether : EtOAc = 80 : 1). Finally, compound **21** (38 mg, colorless oil) was isolated in 72% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 8.06 (d, *J* = 8.52 Hz, 1H), 7.53 (dd, *J* = 1.20 Hz, *J* = 8.12 Hz, 1H), 7.41 - 7.37 (m, 2H), 7.26 (dd, *J* = 1.28 Hz, *J* = 7.48 Hz, 1H), 3.48 - 3.39 (m, 1H), 2.20 - 2.15 (m, 2H), 1.99 - 1.88 (m, 4H), 1.77 - 1.74 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 165.5, 141.6, 136.4, 127.8, 125.5, 123.8, 121.5, 118.7, 48.6, 33.4, 26.0; MS (ESI) calcd for $C_{13}H_{16}N$ $[M+H-N_2]^+$: 211.12, found 211.06



2-isopropyl-3-methylquinolin-8-amine (23)

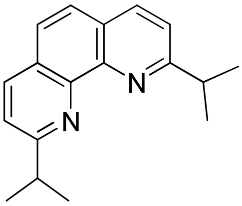
Following the general procedure Ⅰ , 8-azido-2-isopropyl-3-methylquinoline (18 mg, 0.08 mmol) were added to a solution of triphenylphosphine (25 mg, 0.10 mmol) in anhydrous methanol. The flask was coated with aluminum foil. Then, the reaction was stirred at 50°C overnight. After completion of the reaction, the residue was purified by silical gel column chromatography

(Petroleum ether : EtOAc = 40 : 1), Finally, compound **23** (14 mg, colorless oil) was isolated in 88% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 7.74 (s, 1H), 7.22 (t, *J* = 7.84 Hz, 1H), 7.04 (dd, *J* = 0.88 Hz, *J* = 8.12 Hz, 1H), 6.84 (dd, *J* = 0.96 Hz, *J* = 7.40 Hz, 1H), 4.97 (brs, 2H), 3.41 - 3.34 (m, 1H), 2.48 (s, 3H), 1.36 (d, *J* = 6.72 Hz, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 163.4, 143.5, 136.6, 135.8, 129.1, 127.5, 126.3, 115.1, 109.0, 32.0, 21.9, 19.0; MS (ESI) calcd for $C_{13}H_{16}N_2$ $[M+H]^+$: 201.13, found 201.05



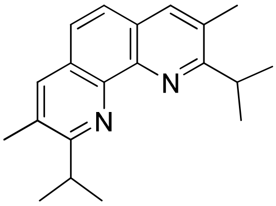
2-cyclopentylquinolin-8-amine (24)

Following the general procedure Ⅰ, 8-azido-2-cyclopentylquinoline (33 mg, 0.14 mmol) were added to a solution of triphenylphosphine (44 mg, 0.17 mmol) in anhydrous methanol. The flask was coated with aluminum foil. Then, the reaction was stirred at 50°C overnight. After completion of the reaction, the residue was purified by silical gel column chromatography (Petroleum ether : EtOAc = 40 : 1), Finally, compound **24** (28 mg, colorless oil) was isolated in 95% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 7.98 (d, *J* = 8.48 Hz, 1H), 7.32 - 7.26 (m, 2H), 7.13 (dd, *J* = 1.08 Hz, *J* = 8.08 Hz, 1H), 6.93 (dd, *J* = 1.16 Hz, *J* = 7.44 Hz, 1H), 5.00 (brs, 2H), 3.41 - 3.35 (m, 1H), 2.20 – 2.13 (m, 2H), 2.01 - 1.85 (m, 4H), 1.79 - 7.74 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 163.2, 143.6, 137.6, 136.0, 127.2, 126.2, 121.0, 115.9, 110.0, 48.3, 33.4, 26.0; MS (ESI) calcd for $C_{14}H_{16}N_2[M+H]^+$: 213.13, found 213.09



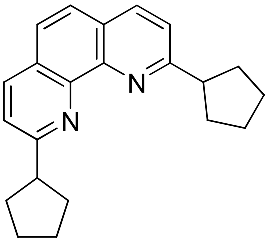
2,9-diisopropyl-1,10-phenanthroline (25)

Following the general procedure Ⅱ, 3-ethoxy-2,2-dimethylcyclobutanone (28 mg, 0.20 mmol), 2-isopropyl-8-aminoquinoline (18 mg, 0.10 mmol), BF_3-OEt_2 (1.0 equiv, 0.10 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (Petroleum ether : EtOAc = 2 : 1). Finally, compound **25** (16 mg, colorless oil) was isolated in 62% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 8.16 (d, *J* = 8.32 Hz, 2H), 7.70 (s, 2H), 7.56 (d, *J* = 8.32 Hz, 2H), 3.61 - 3.54 (m, 2H), 1.49 (d, *J* = 6.96 Hz, 12H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 167.9, 145.3, 136.5, 127.3, 125.5, 120.1, 37.3, 22.9; MS (ESI) calcd for $C_{18}H_{20}N_2$ $[M+H]^+$: 265.16, found 265.07



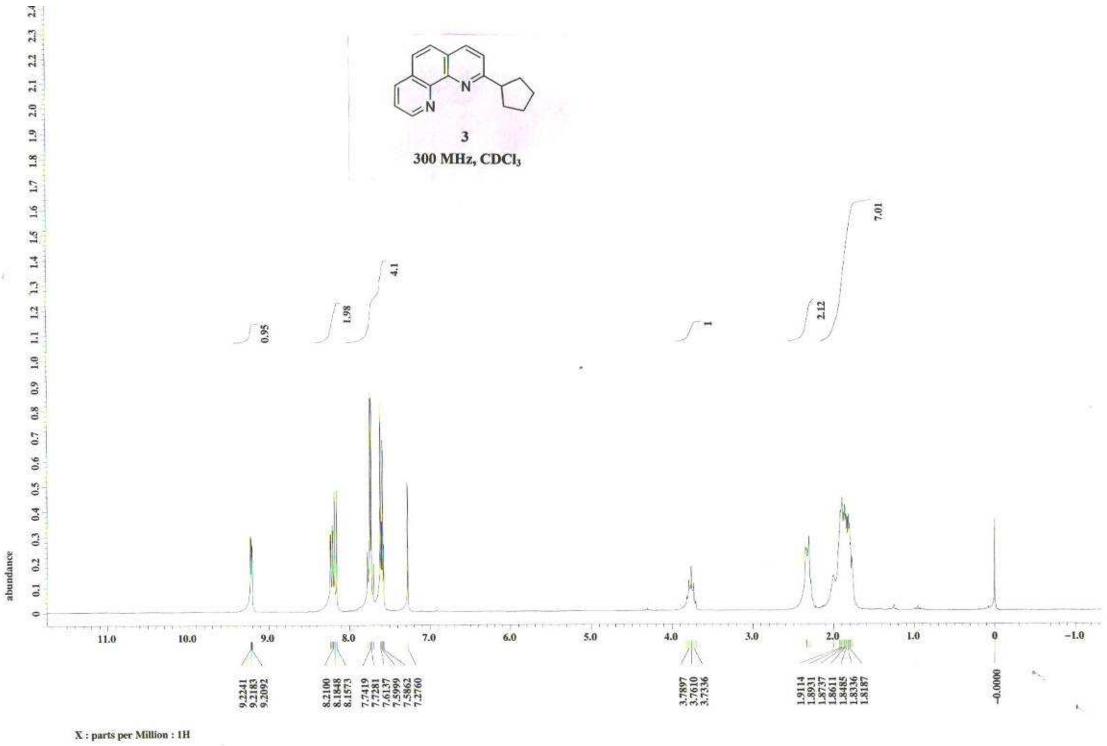
2,9-diisopropyl-3,8-dimethyl-1,10-phenanthroline (26)

Following the general procedure Ⅱ, 3-ethoxy-2,2,4-trimethylcyclobutanone (28 mg, 0.18 mmol), 2-isopropyl-3-methyl-8-aminoquinoline (18 mg, 0.09 mmol), BF_3 -OEt_2 (1.0 equiv, 0.09 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (Petroleum ether : EtOAc = 20 : 1). Finally, compound **26** (14 mg, white solid) was isolated in 53% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 7.85 (s, 2H), 7.59 (s, 2H), 3.53 – 3.46 (m, 2H), 2.55 (s, 6H), 1.53 (d, *J* = 6.72 Hz, 12H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 165.5, 144.0, 136.1, 129.8, 127.0, 124.9, 32.8, 21.7, 19.0; MS (ESI) calcd for $C_{18}H_{20}N_2$ $[M+H]^+$: 265.16, found 265.07



2,9-dicyclopentyl-1,10-phenanthroline (27)

Following the general procedure Ⅱ, 3-ethoxy-spiro[3.4]octan-1-one (40 mg, 0.24 mmol), 2-cyclopentyl-8-aminoquinoline (25 mg, 0.12 mmol), BF_3 -OEt_2 (1.0 equiv, 0.12 mmol) and crushed 4 Å molecular sieves (150%wt) were used. The reaction mixture was stirred at room temperature under atmosphere of Argon for 48h. After completion of the reaction, the residue was purified by silical gel column chromatography (Petroleum ether : EtOAc = 10 : 1). Finally, compound **27** (25 mg, white solid) was isolated in 68% yield.^{1}H-NMR (400 MHz, $CDCl_3$) δ (ppm) 8.21 (d, *J* = 8.36 Hz, 2H), 7.74 (s, 2H), 7.60 (d, *J* = 8.40 Hz, 2H), 3.80 - 3.76 (m, 2H), 2.40 - 2.33 (m, 4H), 2.01 - 1.92 (m, 8H), 1.91 - 1.81 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ (ppm) 167.0, 136.8, 127.3, 125.5, 121.4, 48.6, 34.2, 26.2; MS (ESI) calcd for $C_{22}H_{24}N_2$ $[M+H]^+$: 317.19, found 317.12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4.1
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2.12
7.01
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11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 −1.0
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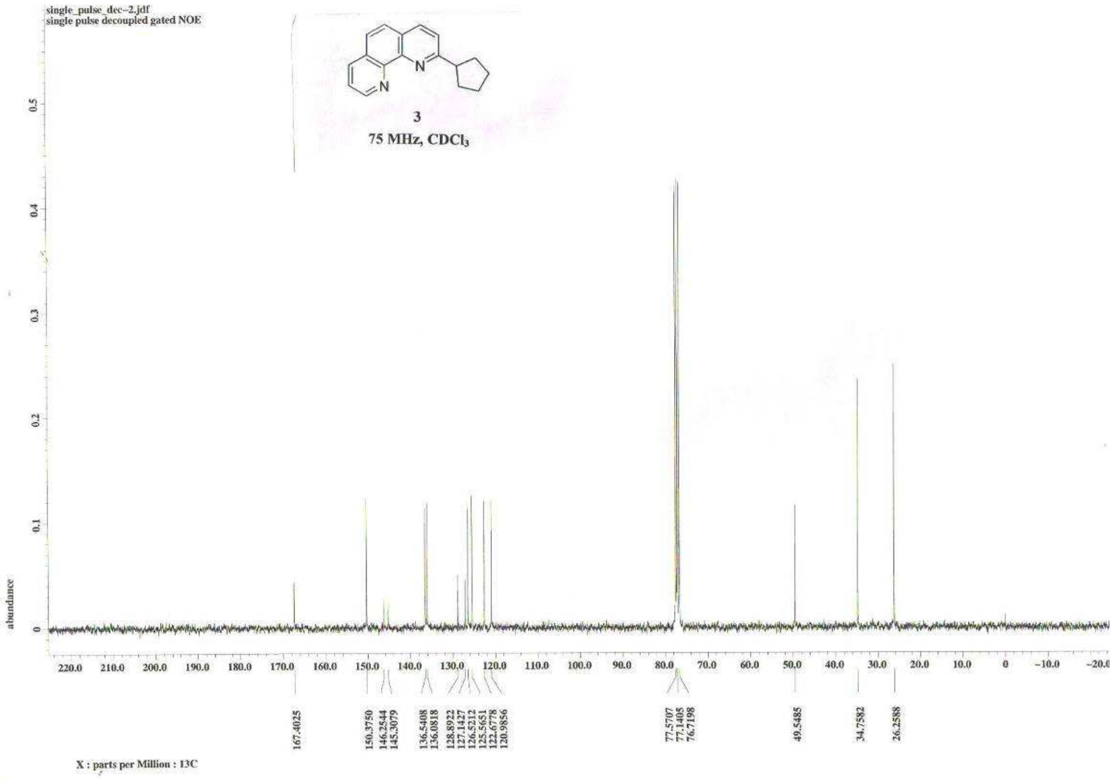
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N
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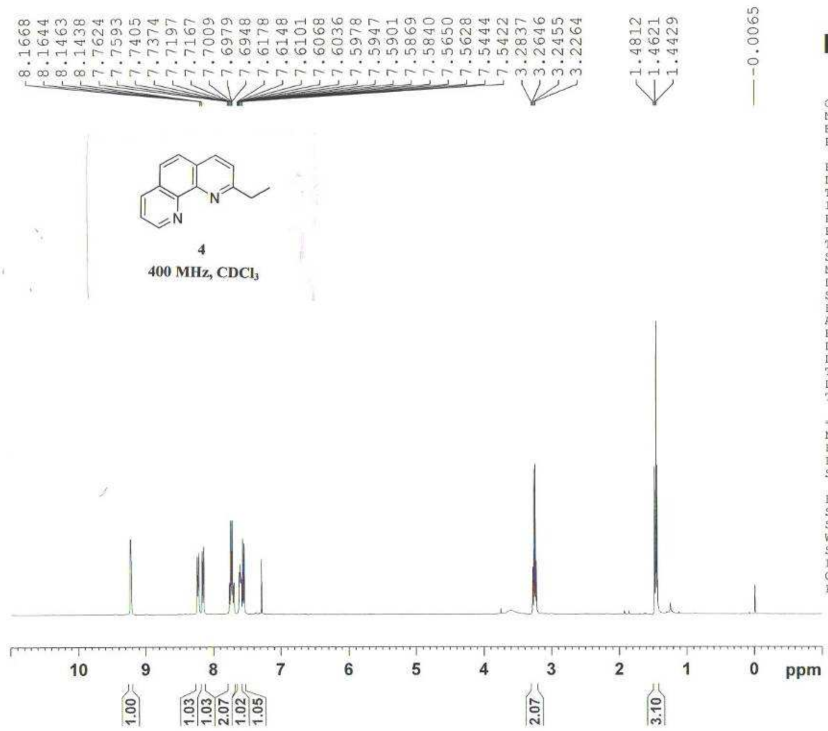

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DE                    6.50 usec
TE                   297.3 K
D1              1.00000000 sec
TD0                      1

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P1                   14.60 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

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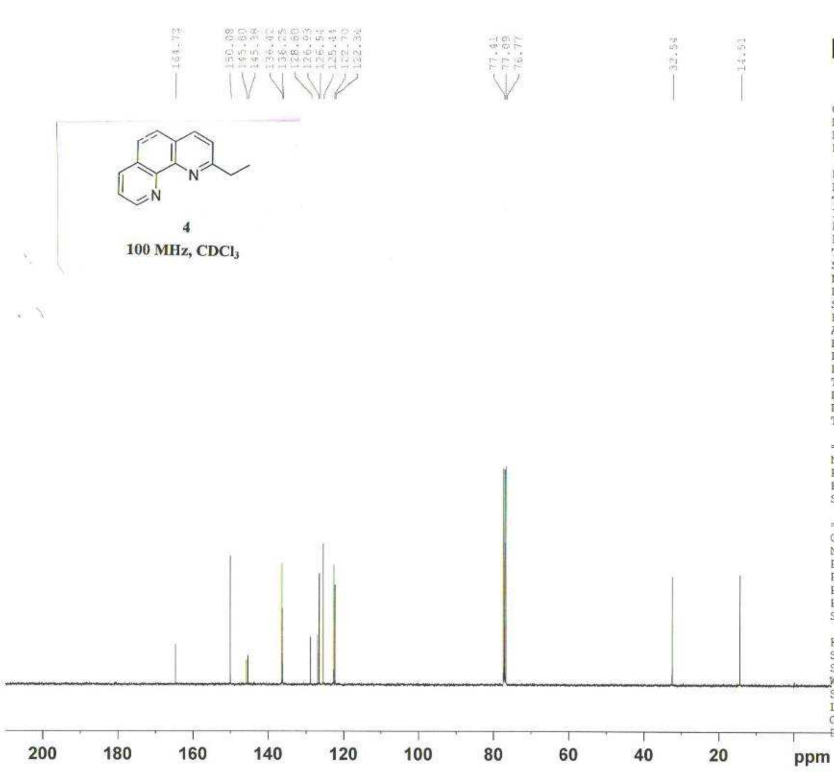

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D1              2.00000000 sec
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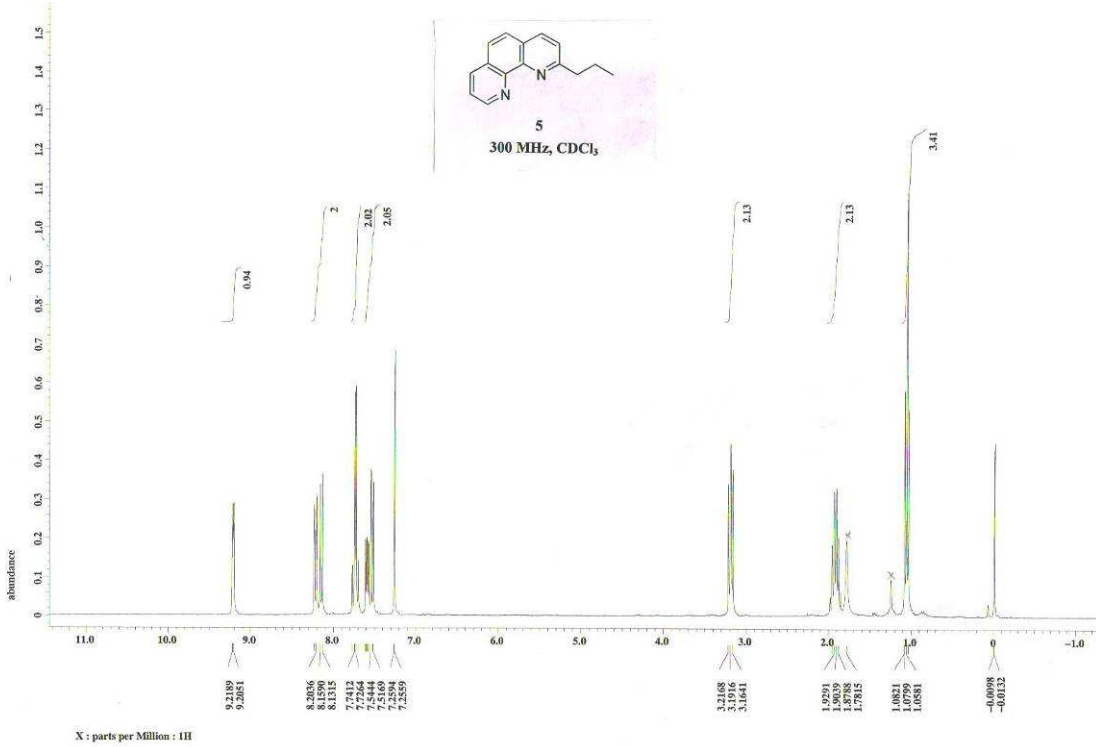
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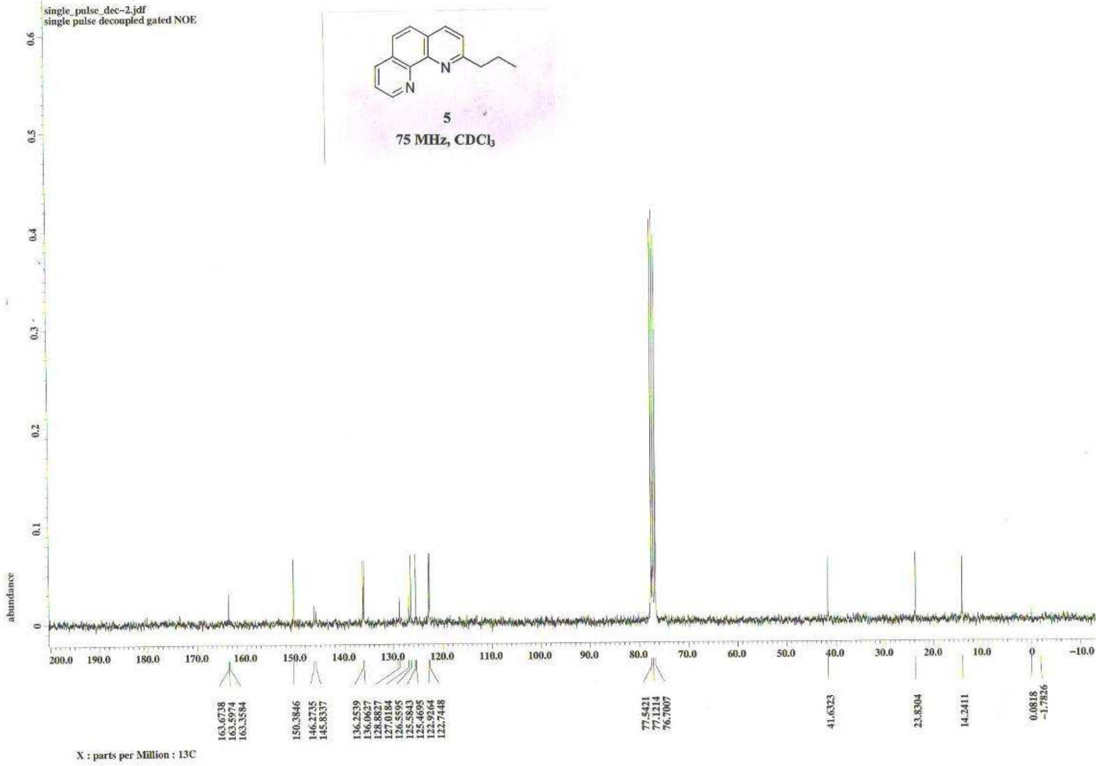
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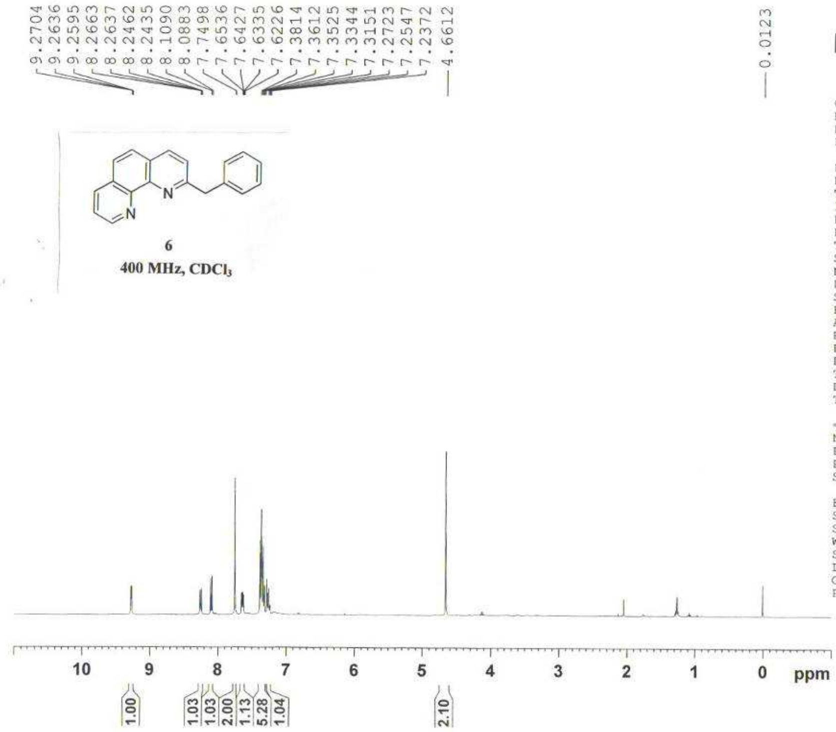
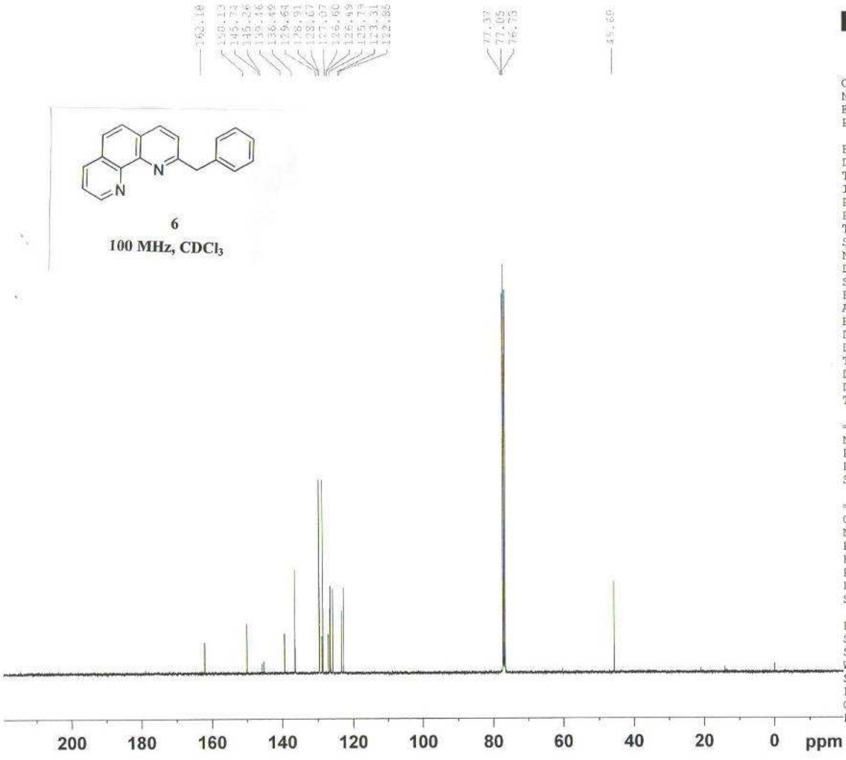
0.0818
-1.7826



6

400 MHz, CDCl$_3$

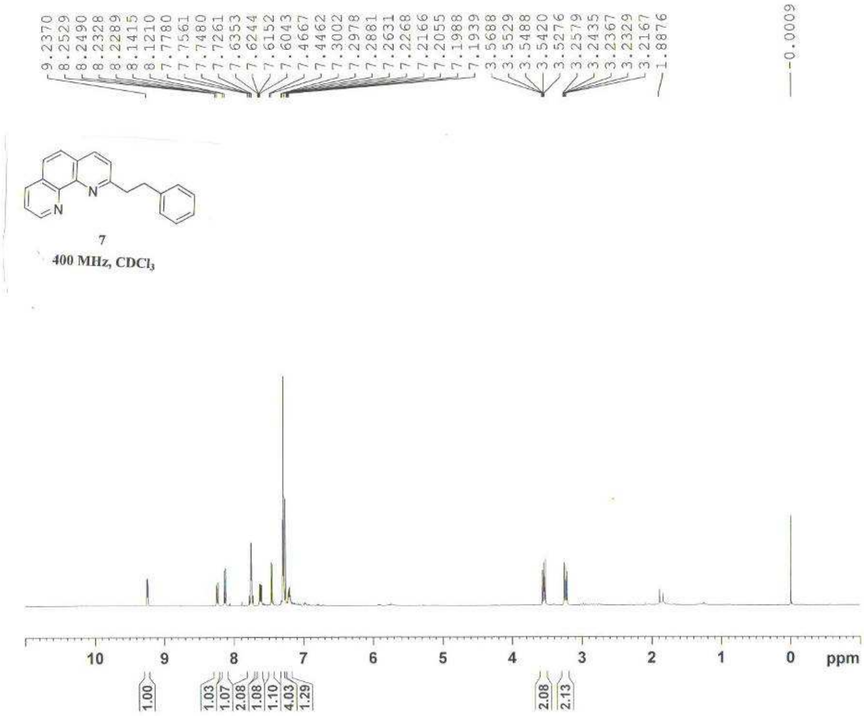
```
Current Data Parameters
NAME          CYF-3-87-1014
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111014
Time                  12.53
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                3.9846387 sec
RG                   102.67
DW                   60.800 usec
DE                     6.50 usec
TE                    296.9 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.60 usec
PLW1            13.00199986 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF              400.1300000 MHz
WDW                      EM
SSB      0
LB                     0.30 Hz
GB       0
PC                     1.00
```



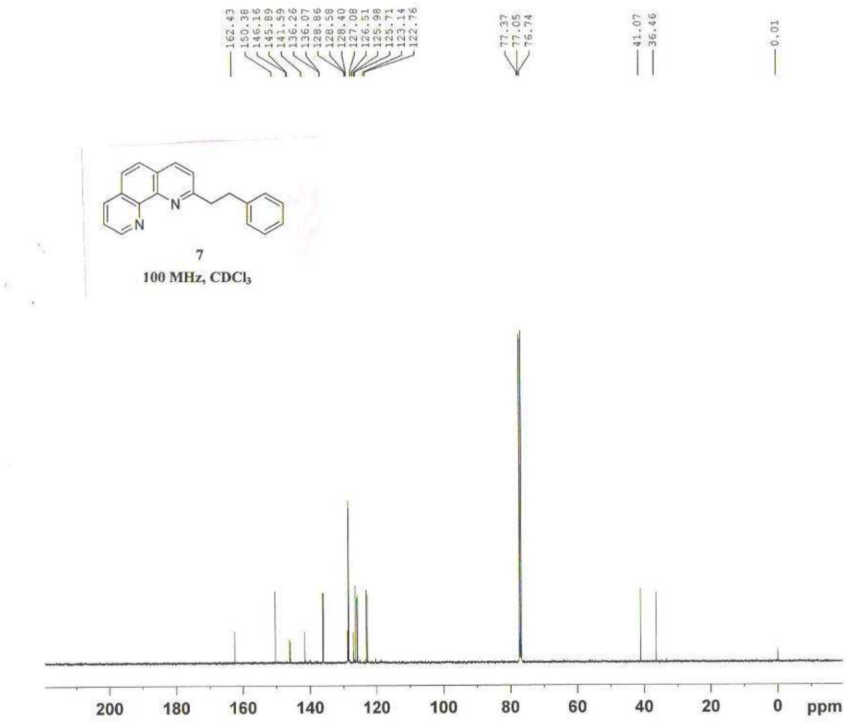
6

100 MHz, CDCl$_3$

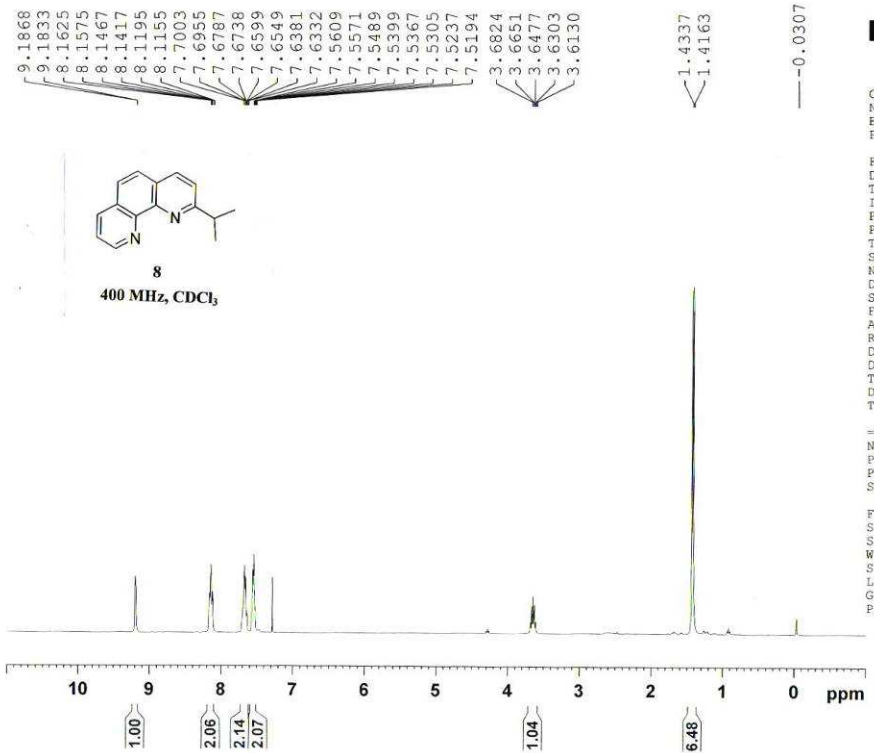
```
Current Data Parameters
NAME        CYF-3-87-C-1014
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111014
Time                  13.08
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631988 sec
RG                   184.16
DW                   20.800 usec
DE                     6.50 usec
TE                    297.6 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PLW1            50.00299835 W
SFO1            100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 90.00 usec
PLW2            13.00199986 W
PLW12            0.36116999 W
PLW13            0.29255000 W
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF              100.6127690 MHz
WDW                      EM
SSB      0
LB                     1.00 Hz
GB       0
PC                     1.40
```



```
Current Data Parameters
NAME     CYF-3-119-H-1021
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111021
Time                13.08
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ              3.9846387 sec
RG                 115.38
DW                 60.800 usec
DE                   6.50 usec
TE                  297.1 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.60 usec
PLW1          13.00199986 W
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300080 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



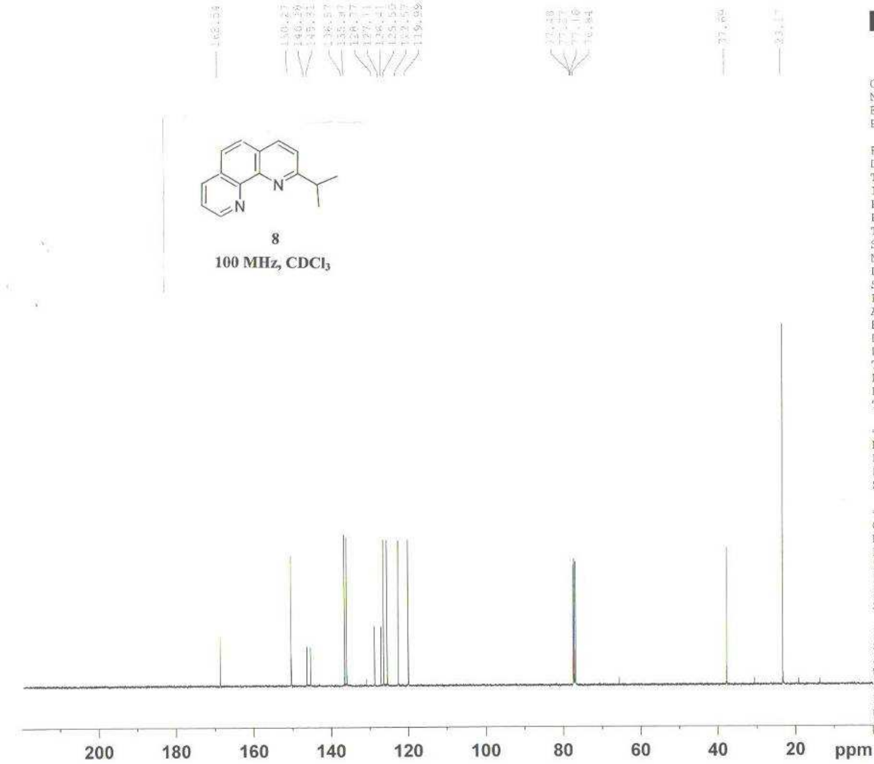
```
Current Data Parameters
NAME     CYF-3-119-C-1021
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111021
Time                15.19
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631988 sec
RG                     85
DW                 20.800 usec
DE                   6.50 usec
TE                  297.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PLW1          50.00299835 W
SFO1          100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PLW2          13.00199986 W
PLW12          0.36116999 W
PLW13          0.29255000 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127690 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```



8

400 MHz, CDCl$_3$

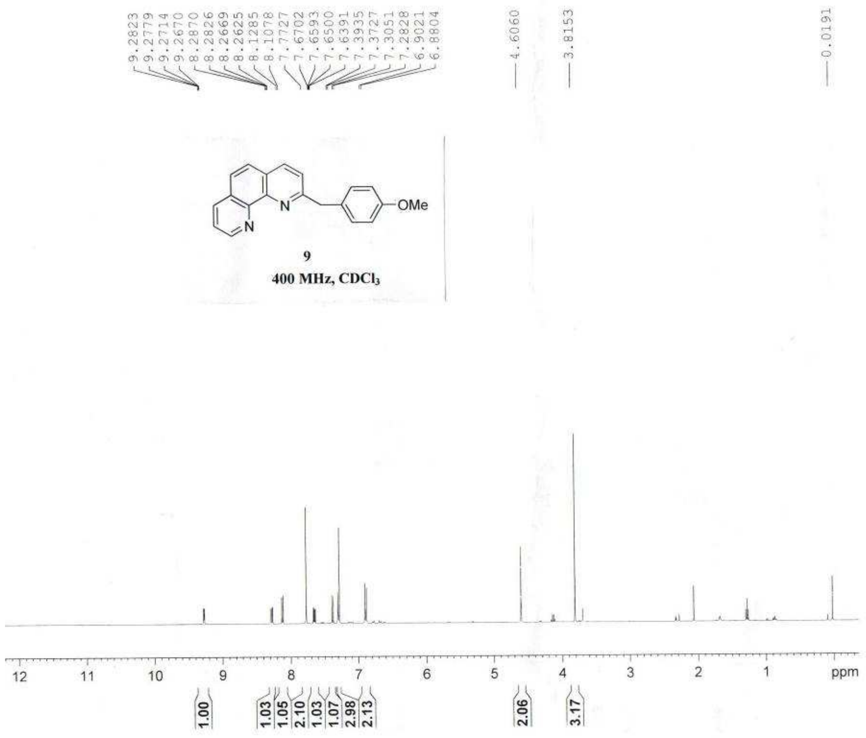
```
Current Data Parameters
NAME      CYF-3-47-H-1006
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111006
Time                20.59
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ              3.9846387 sec
RG                  33.21
DW                 60.800 usec
DE                   6.50 usec
TE                  296.7 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.60 usec
PLW1          13.00199986 W
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300000 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



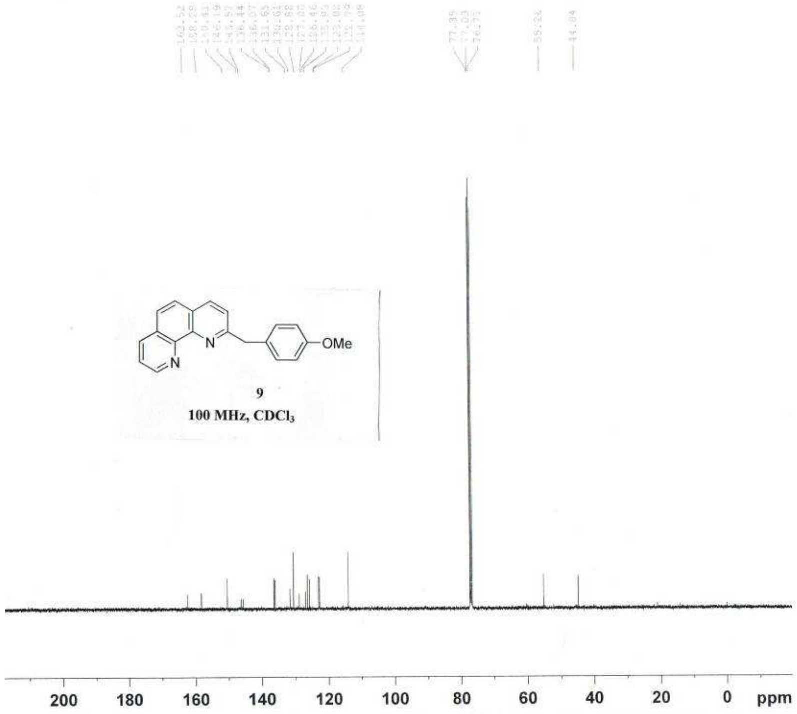
8

100 MHz, CDCl$_3$

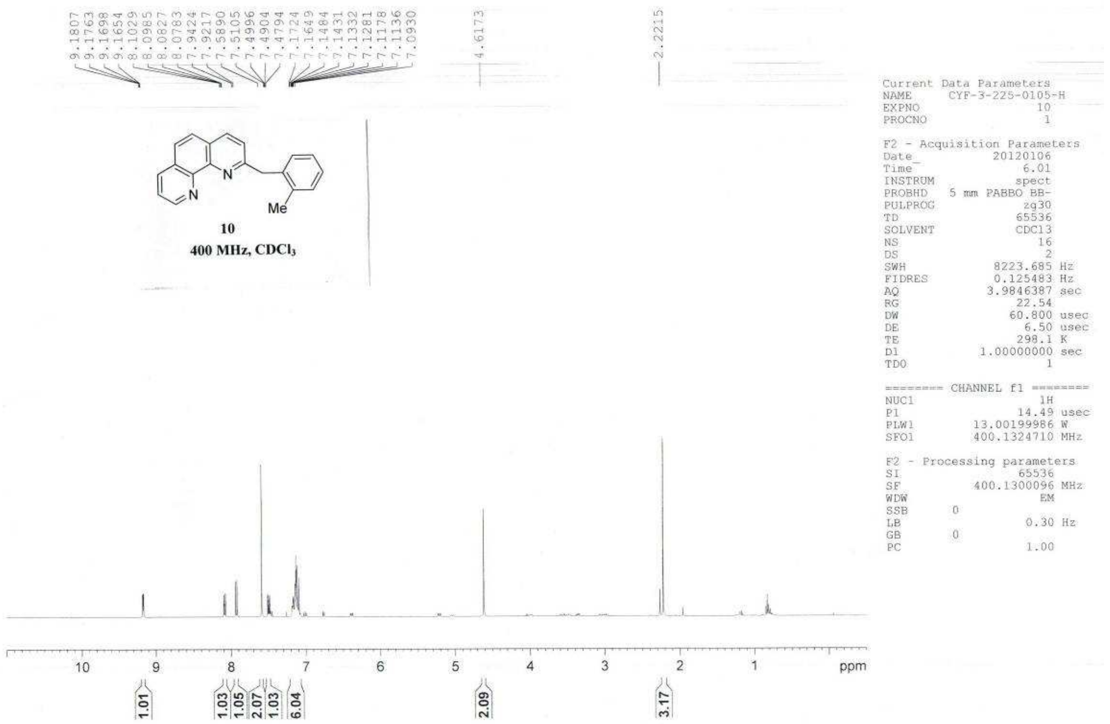
```
Current Data Parameters
NAME      CYF-3-47-C-1006
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111006
Time                21.21
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    256
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631988 sec
RG                 209.25
DW                 20.800 usec
DE                   6.50 usec
TE                  297.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PLW1          50.00299835 W
SFO1          100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PLW2          13.00199986 W
PLW12          0.36116999 W
PLW13          0.29255000 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127690 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```



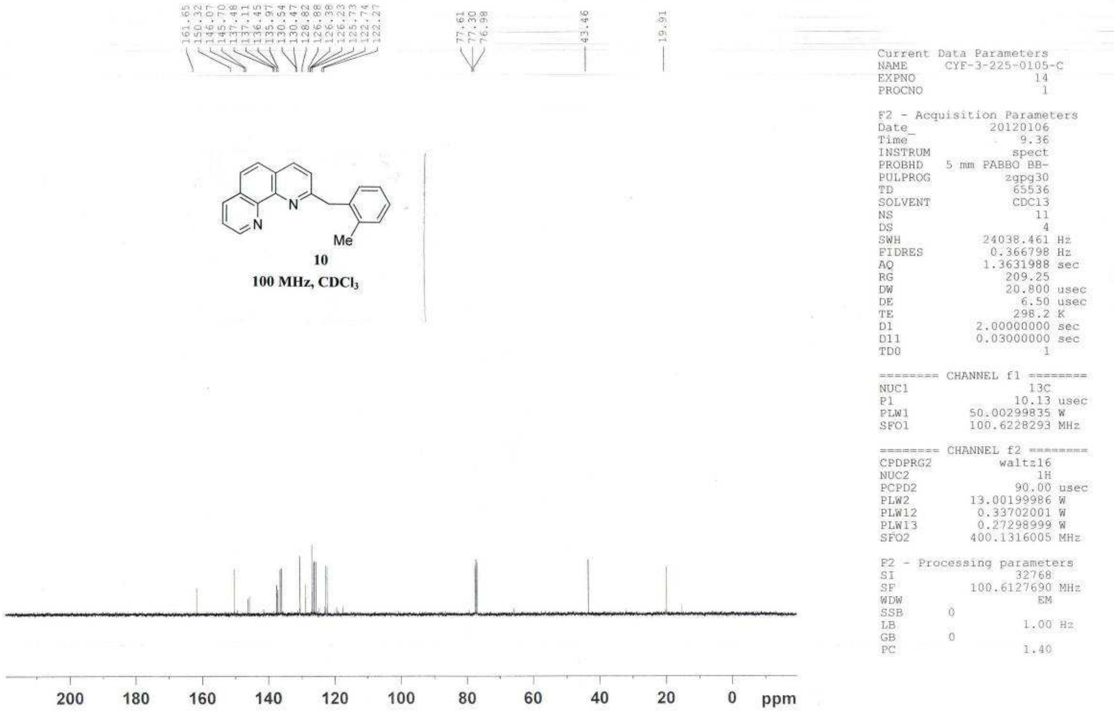
```
Current Data Parameters
NAME          CYF-3-197-H
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111223
Time                23.26
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ              3.9846387 sec
RG                 184.16
DW                 60.800 usec
DE                   6.50 usec
TE                  296.6 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.49 usec
PLW1          13.00199986 W
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300000 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



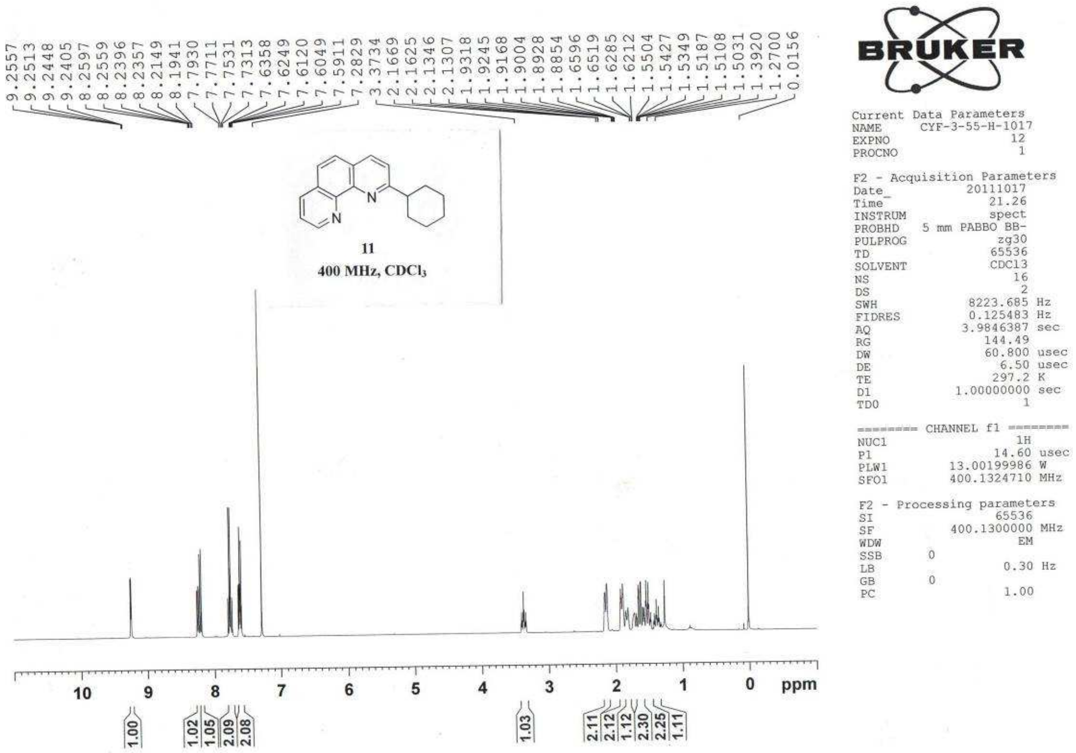
```
Current Data Parameters
NAME          CYF-3-197-H
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111224
Time                 1.55
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631988 sec
RG                 115.38
DW                 20.800 usec
DE                   6.50 usec
TE                  297.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.13 usec
PLW1          50.00299835 W
SFO1          100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PLW2          13.00199986 W
PLW12          0.33702001 W
PLW13          0.27298999 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127690 MHz
WDW                    EM
SSB      0
LB                   1.00 Hz
GB       0
PC                   1.40
```



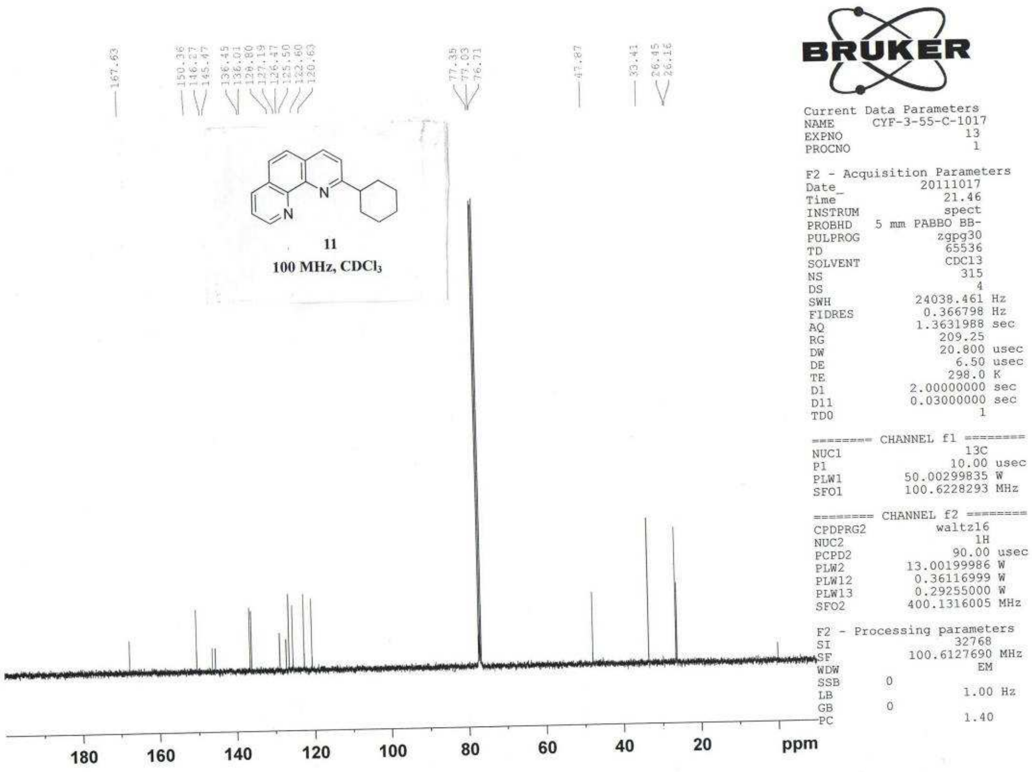
```
Current Data Parameters
NAME      CYF-3-225-0105-H
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20120106
Time                  6.01
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                   22.54
DW                  60.800 usec
DE                    6.50 usec
TE                   298.1 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.49 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300096 MHz
WDW                     EM
SSB      0
LB                    0.30 Hz
GB       0
PC                    1.00
```



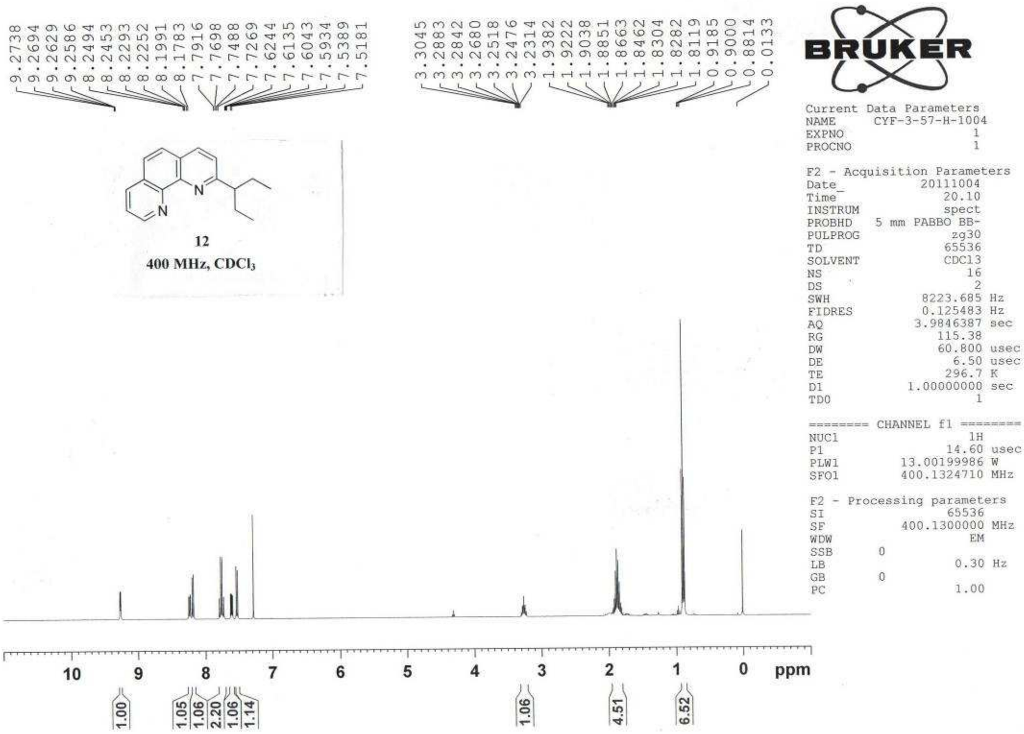
```
Current Data Parameters
NAME      CYF-3-225-0105-C
EXPNO                   14
PROCNO                   1

F2 - Acquisition Parameters
Date_             20120106
Time                  9.36
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      11
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  209.25
DW                  20.800 usec
DE                    6.50 usec
TE                   298.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.13 usec
PLW1           50.00299835 W
SFO1           100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.33702001 W
PLW13           0.27298999 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB      0
LB                    1.00 Hz
GB       0
PC                    1.40
```



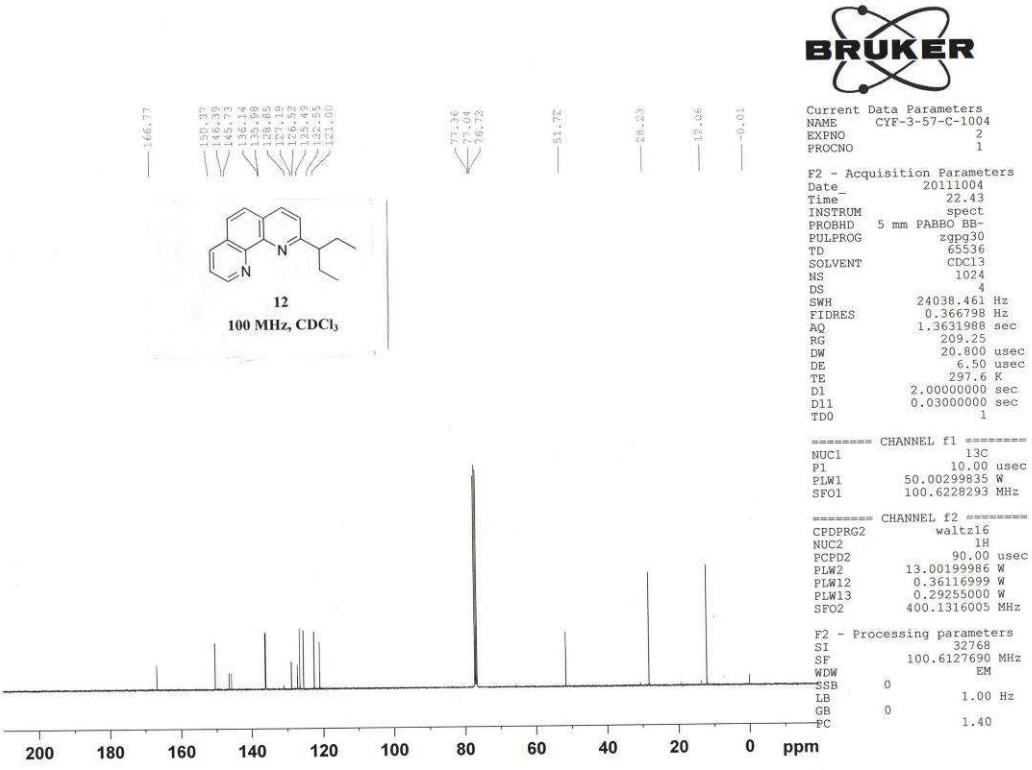
```
Current Data Parameters
NAME       CYF-3-55-H-1017
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111017
Time                 21.26
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                  144.49
DW                  60.800 usec
DE                    6.50 usec
TE                   297.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.60 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300000 MHz
WDW                     EM
SSB      0
LB                    0.30 Hz
GB       0
PC                    1.00
```



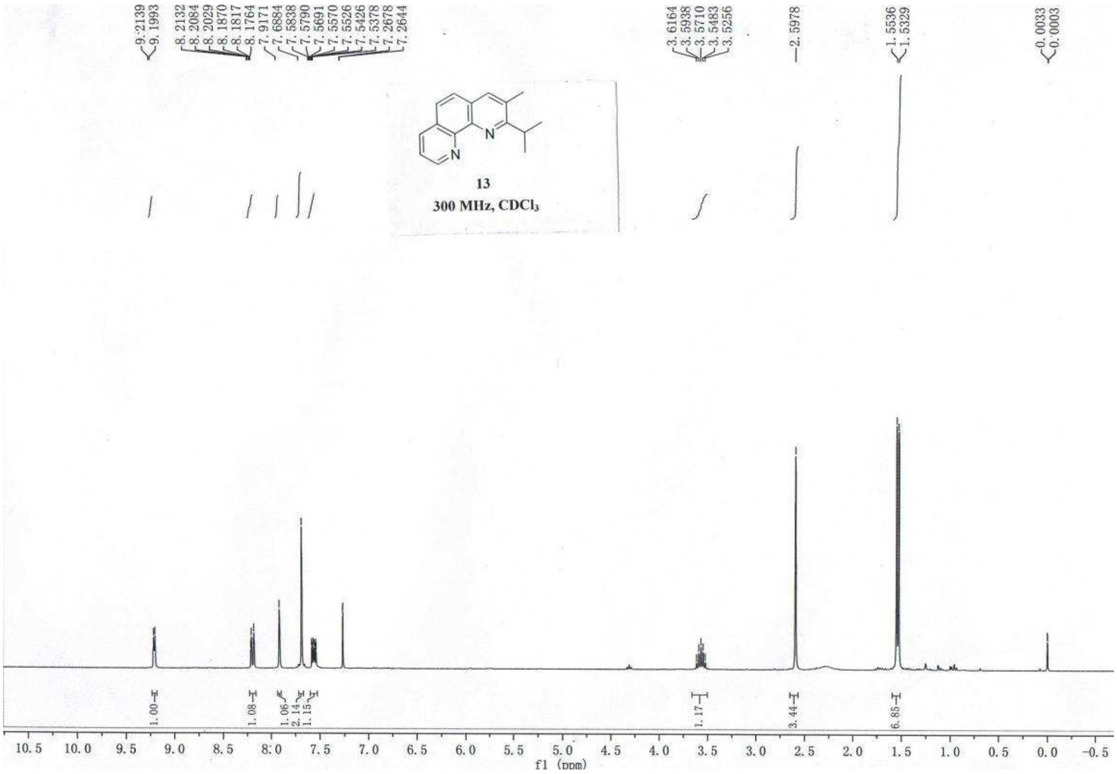
```
Current Data Parameters
NAME       CYF-3-55-C-1017
EXPNO                   13
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111017
Time                 21.46
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     315
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  209.25
DW                  20.800 usec
DE                    6.50 usec
TE                   298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.00 usec
PLW1           50.00299835 W
SFO1           100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.36116999 W
PLW13           0.29255000 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB      0
LB                    1.00 Hz
GB       0
PC                    1.40
```

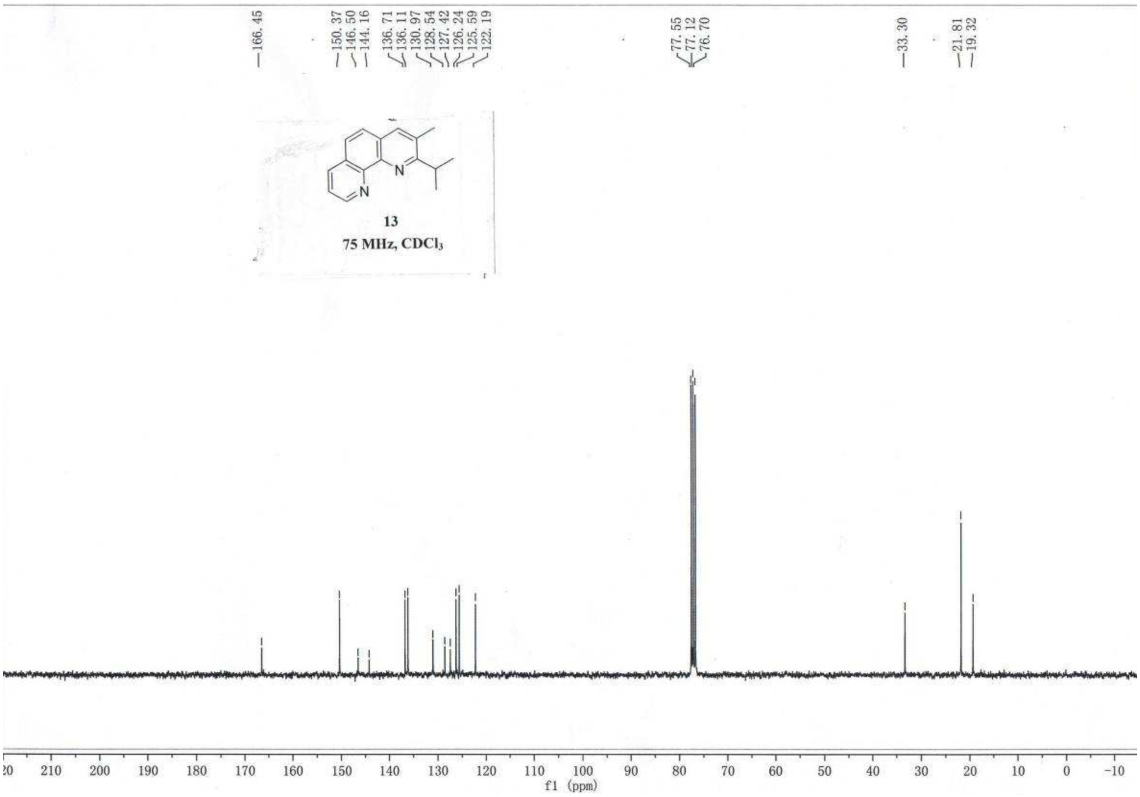


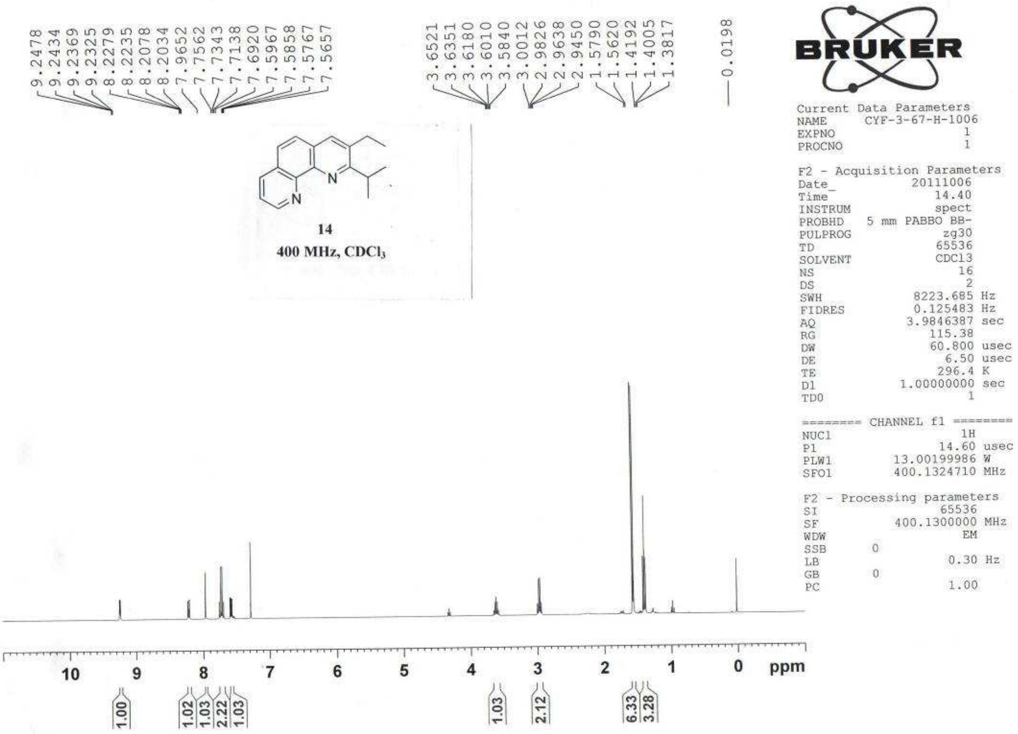
```
Current Data Parameters
NAME       CYF-3-57-H-1004
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111004
Time                 20.10
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                  115.38
DW                  60.800 usec
DE                    6.50 usec
TE                   296.7 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.60 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300000 MHz
WDW                     EM
SSB      0
LB                    0.30 Hz
GB       0
PC                    1.00
```



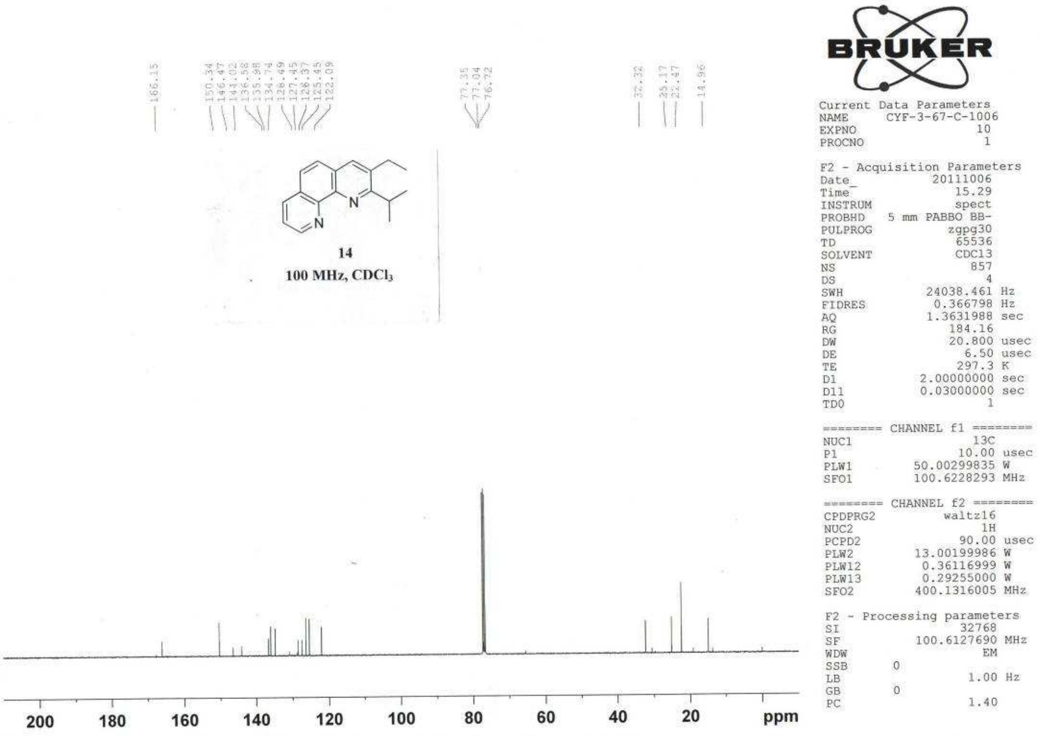
```
Current Data Parameters
NAME       CYF-3-57-C-1004
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111004
Time                 22.43
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  209.25
DW                  20.800 usec
DE                    6.50 usec
TE                   297.6 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.00 usec
PLW1           50.00299835 W
SFO1           100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.36116999 W
PLW13           0.29255000 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB      0
LB                    1.00 Hz
GB       0
PC                    1.40
```


9.2139
9.1993
8.2132
8.2084
8.2029
8.1870
8.1817
8.1764
7.9171
7.6884
7.5838
7.5790
7.5691
7.5570
7.5526
7.5426
7.5378
7.2678
7.2644
3.6164
3.5938
3.5710
3.5483
3.5256
2.5978
1.5536
1.5329
0.0033
0.0003
N
N
13
300 MHz, CDCl₃
1.00
1.08
1.06
2.14
1.15
1.17
3.44
6.85
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

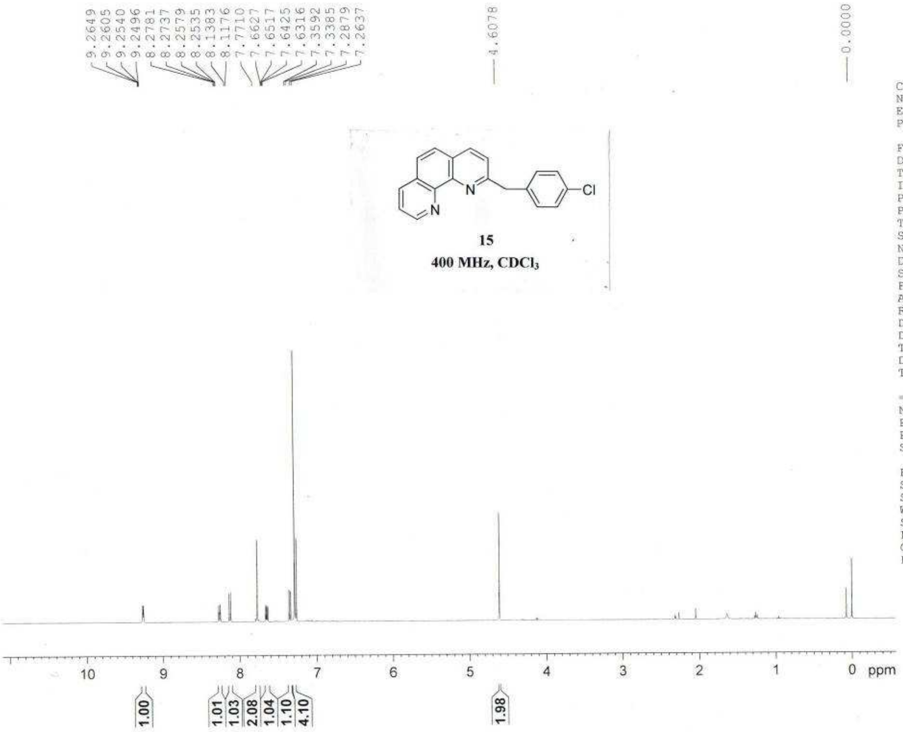

166.45
150.37
146.50
144.16
136.71
136.11
130.97
128.54
127.42
126.24
125.59
122.19
77.55
77.12
76.70
33.30
21.81
19.32
N
N
13
75 MHz, CDCl₃
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



```
Current Data Parameters
NAME       CYF-3-67-H-1006
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111006
Time                 14.40
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                  115.38
DW                  60.800 usec
DE                    6.50 usec
TE                   296.4 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.60 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300000 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

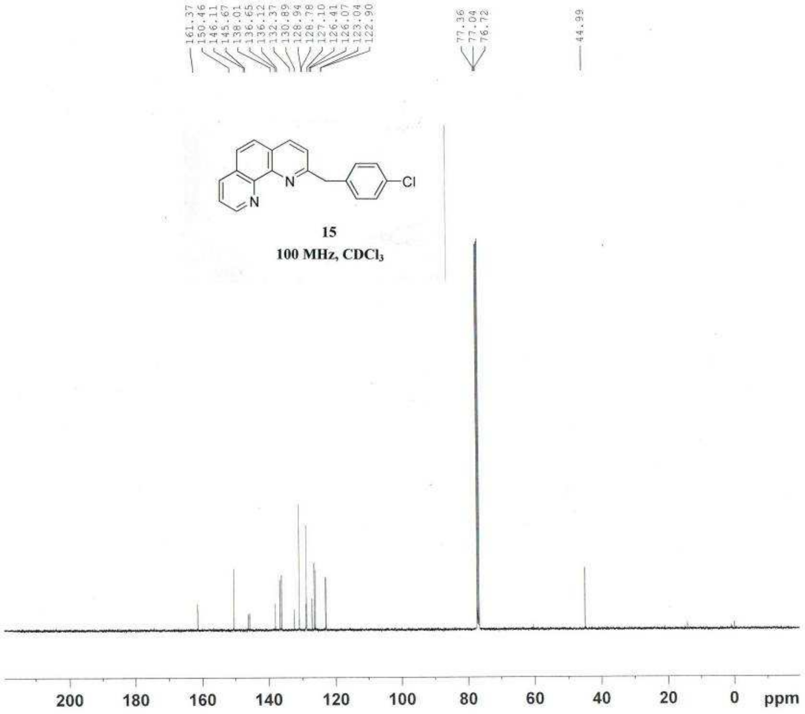


```
Current Data Parameters
NAME       CYF-3-67-C-1006
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111006
Time                 15.29
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     857
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  184.16
DW                  20.800 usec
DE                    6.50 usec
TE                   297.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.00 usec
PLW1           50.00299835 W
SFO1           100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.36116999 W
PLW13           0.29255000 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



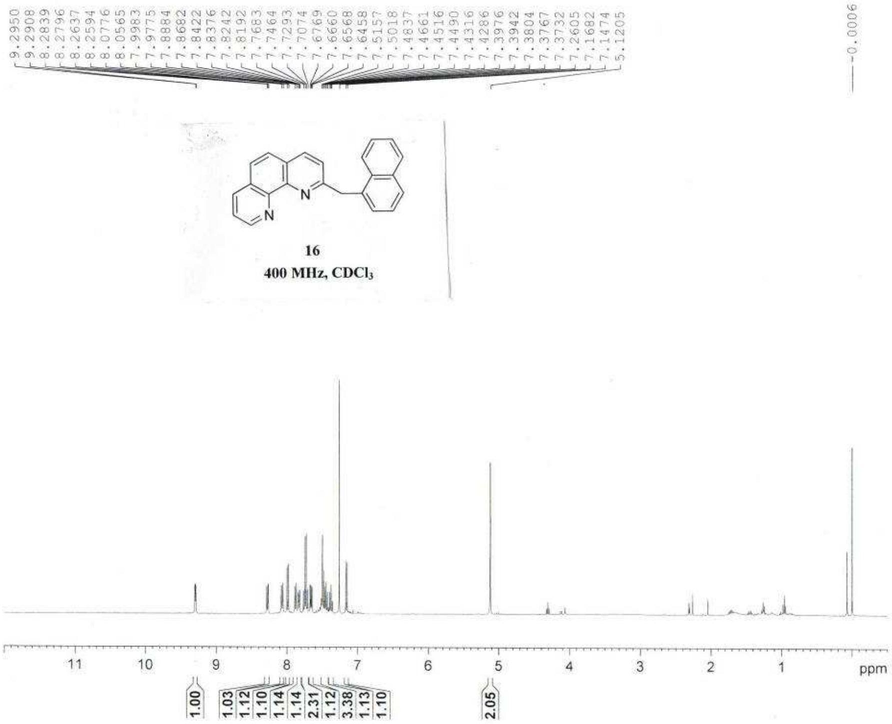
15
400 MHz, CDCl$_3$

```
Current Data Parameters
NAME      CYF-3-201-H-1224
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111224
Time                 21.50
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                  209.25
DW                  60.800 usec
DE                    6.50 usec
TE                   296.6 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.49 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300075 MHz
WDW                     EM
SSB      0
LB                    0.30 Hz
GB       0
PC                    1.00
```



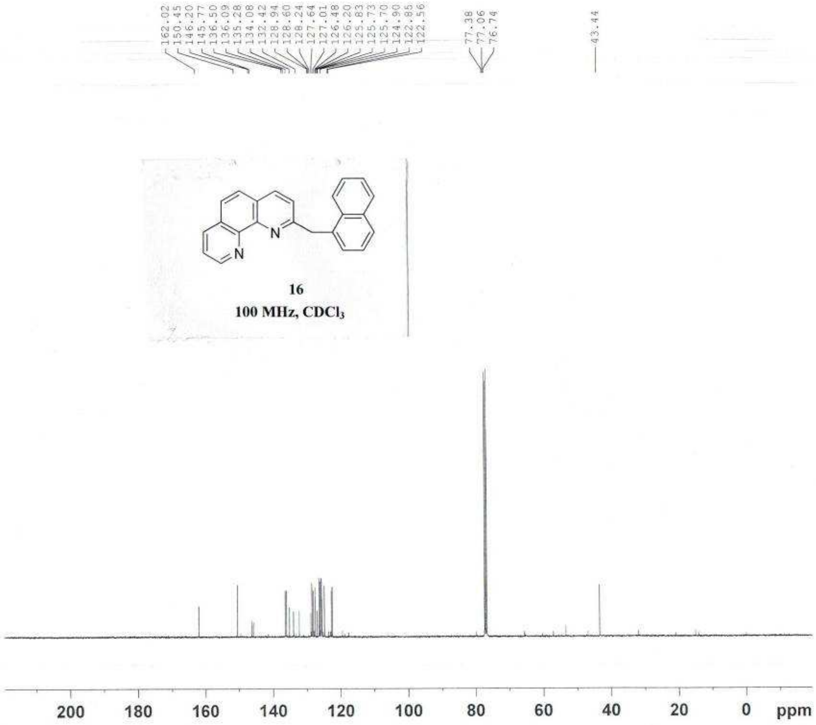
15
100 MHz, CDCl$_3$

```
Current Data Parameters
NAME      CYF-3-201-C-1226
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111227
Time                  5.41
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  184.16
DW                  20.800 usec
DE                    6.50 usec
TE                   297.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.13 usec
PLW1           50.00299835 W
SFO1           100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.33702001 W
PLW13           0.27298999 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB      0
LB                    1.00 Hz
GB       0
PC                    1.40
```



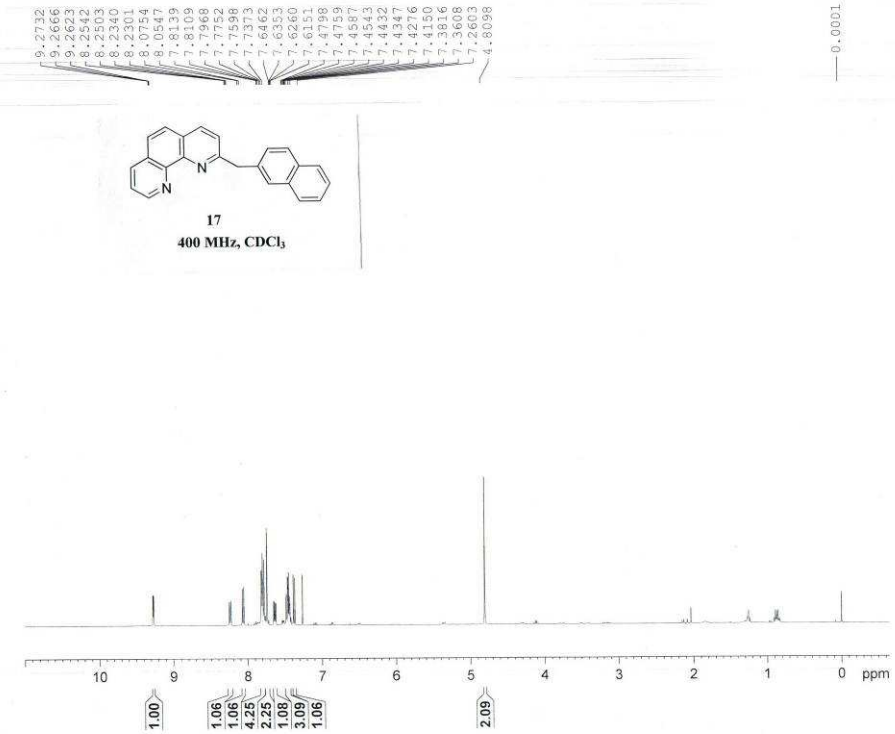
16
400 MHz, CDCl$_3$

```
Current Data Parameters
NAME       CYF-3-203-H-1226
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111227
Time                   7.16
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                3.9846387 sec
RG                   209.25
DW                   60.800 usec
DE                     6.50 usec
TE                    296.4 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.49 usec
PLW1            13.00199986 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF              400.1300088 MHz
WDW                      EM
SSB      0
LB                     0.30 Hz
GB       0
PC                     1.00
```



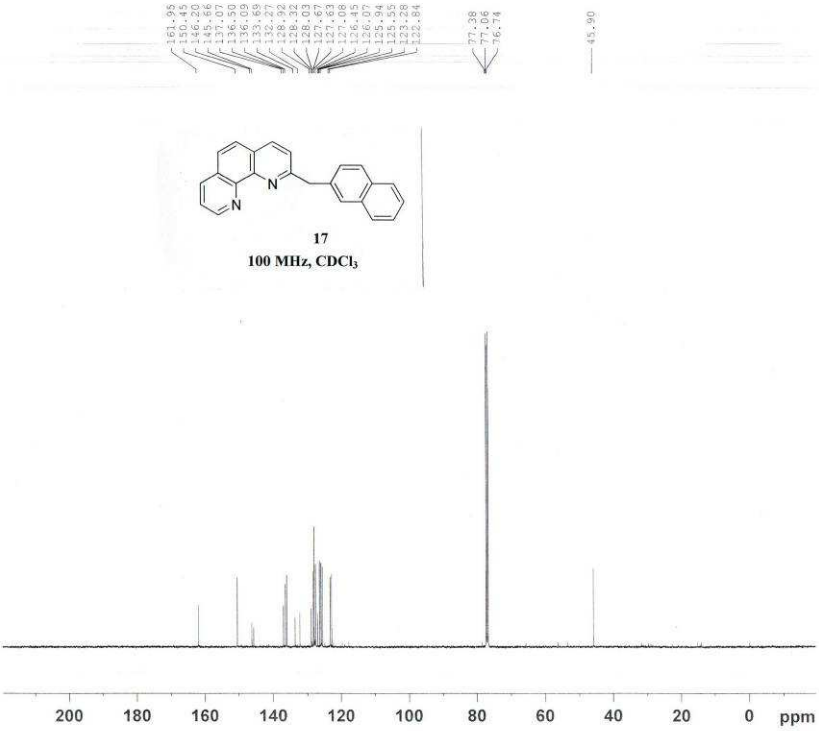
16
100 MHz, CDCl$_3$

```
Current Data Parameters
NAME       CYF-3-219-C-0105
EXPNO                    12
PROCNO                    1

F2 - Acquisition Parameters
Date_              20120105
Time                   2.34
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631988 sec
RG                   209.25
DW                   20.800 usec
DE                     6.50 usec
TE                    298.1 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.13 usec
PLW1            50.00299835 W
SFO1            100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 90.00 usec
PLW2            13.00199986 W
PLW12            0.33702001 W
PLW13            0.27298999 W
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF              100.6127690 MHz
WDW                      EM
SSB      0
LB                     1.00 Hz
GB       0
PC                     1.40
```



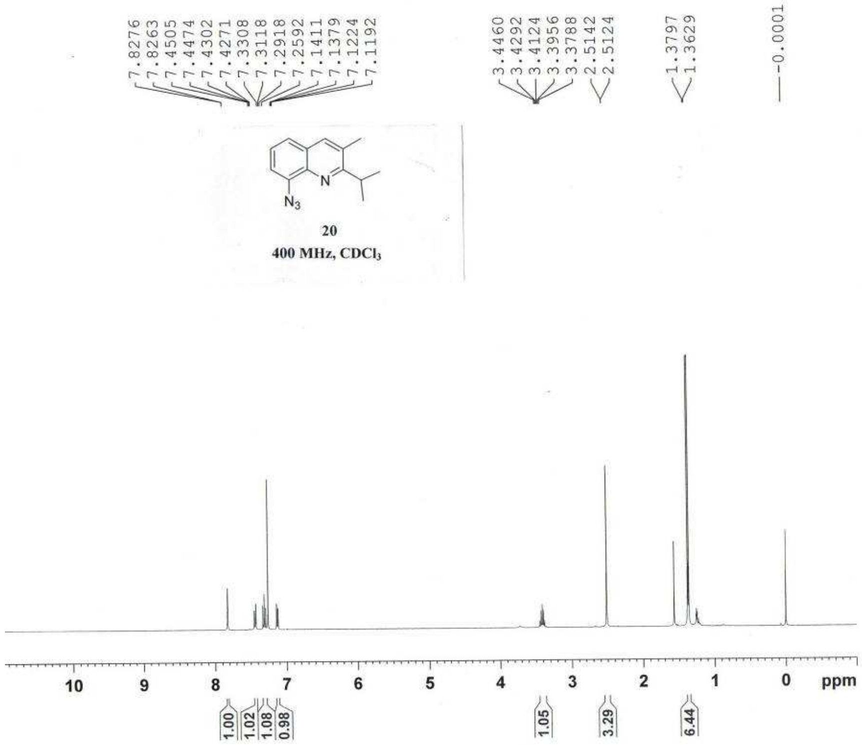
17
400 MHz, CDCl$_3$

```
Current Data Parameters
NAME      CYF-3-215-0105-H
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20120106
Time                  4.57
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                  115.38
DW                  60.800 usec
DE                    6.50 usec
TE                   298.1 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.49 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300090 MHz
WDW                     EM
SSB      0
LB                    0.30 Hz
GB       0
PC                    1.00
```

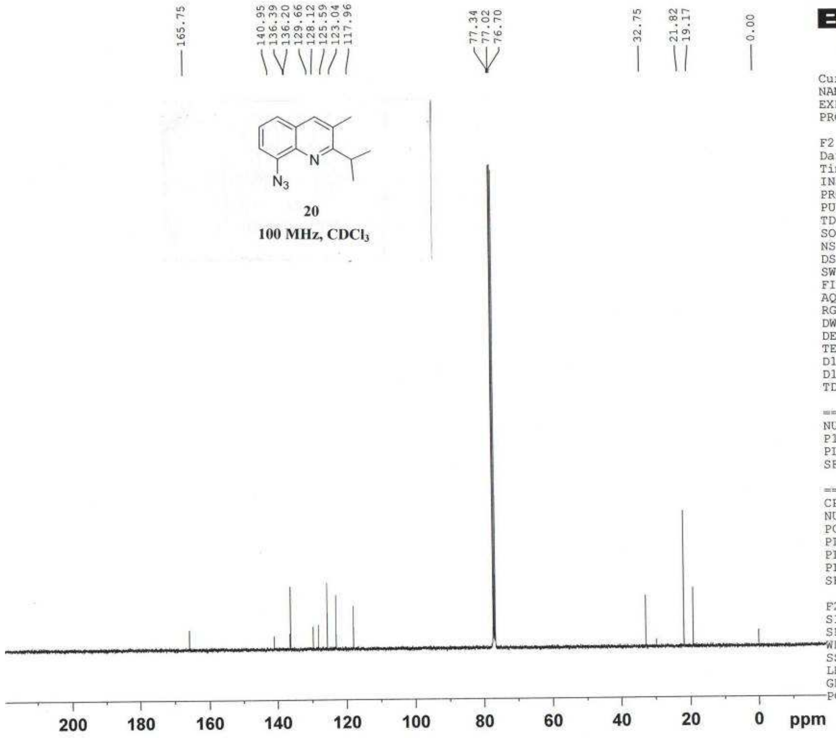


17
100 MHz, CDCl$_3$

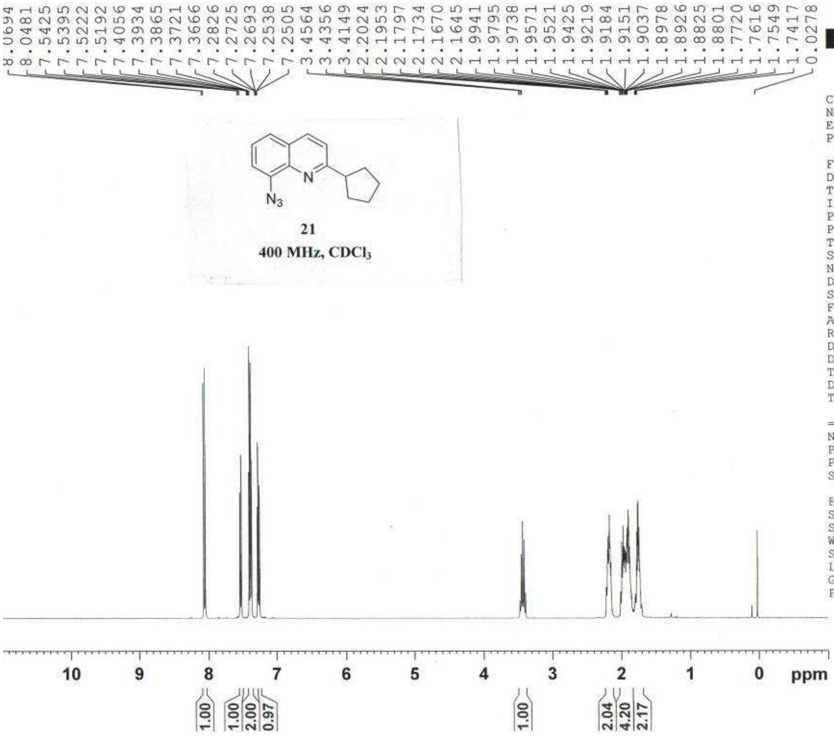
```
Current Data Parameters
NAME      CYF-3-215-0105-C
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_             20120106
Time                  5.57
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  209.25
DW                  20.800 usec
DE                    6.50 usec
TE                   298.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.13 usec
PLW1           50.00299835 W
SFO1           100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.33702001 W
PLW13           0.27298999 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB      0
LB                    1.00 Hz
GB       0
PC                    1.40
```



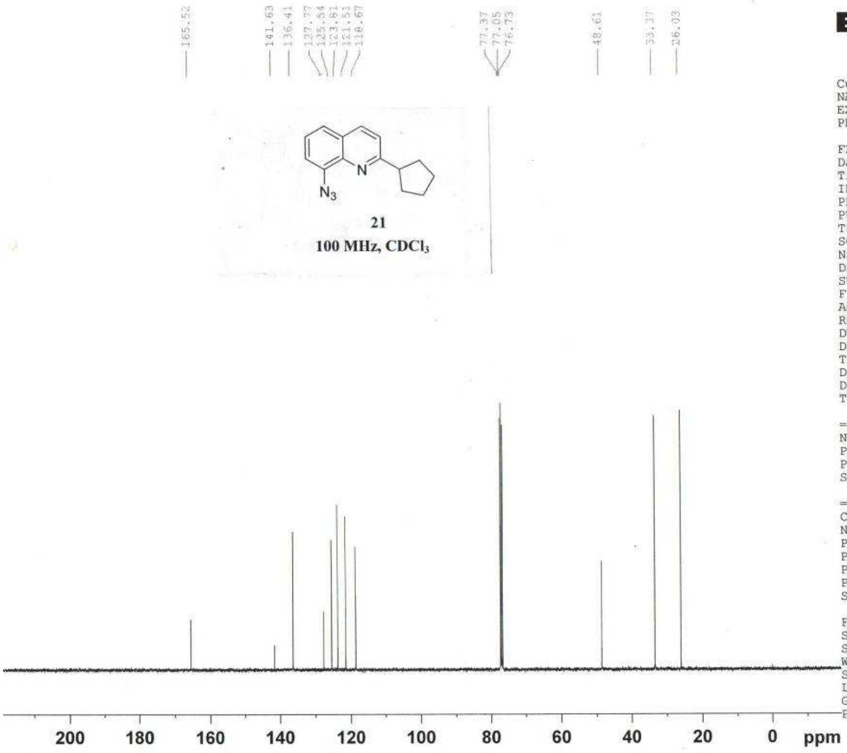
```
Current Data Parameters
NAME              CYF-3-127
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111024
Time                  11.31
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                3.9846387 sec
RG                   184.16
DW                   60.800 usec
DE                     6.50 usec
TE                    296.1 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.60 usec
PLW1            13.00199986 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF              400.1300093 MHz
WDW                      EM
SSB      0
LB                     0.30 Hz
GB       0
PC                     1.00
```



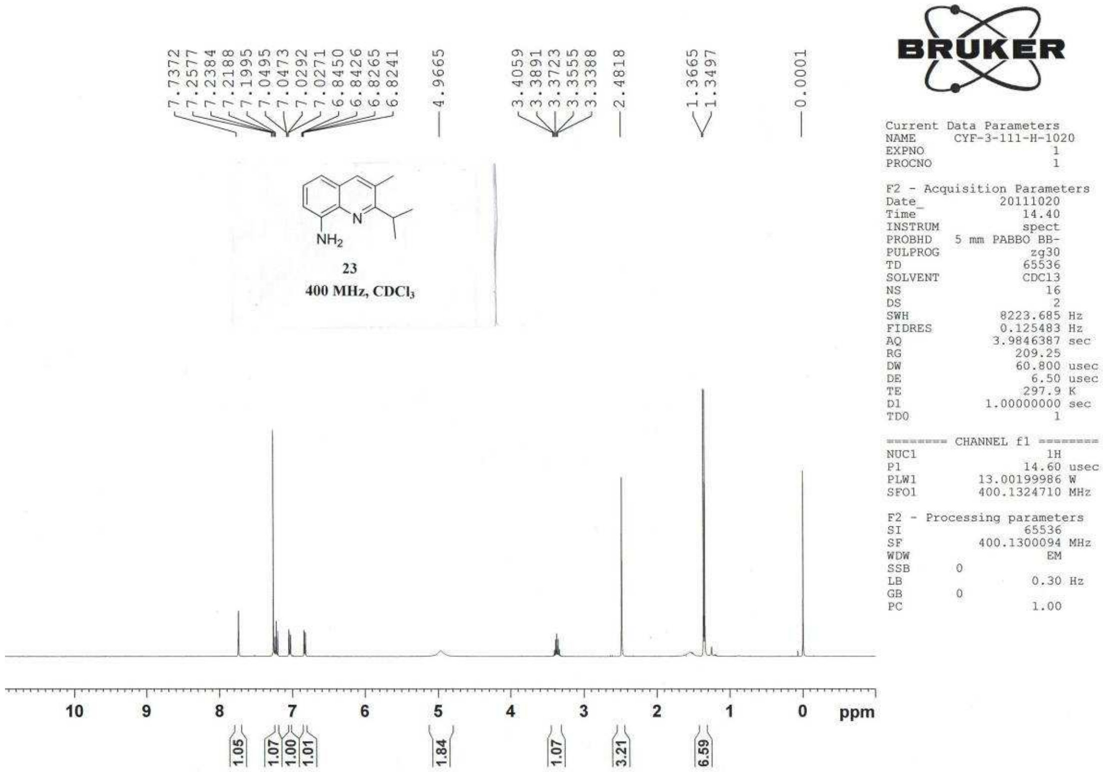
```
Current Data Parameters
NAME            CYF-3-127-C
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111024
Time                  11.41
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631988 sec
RG                   165.63
DW                   20.800 usec
DE                     6.50 usec
TE                    296.8 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PLW1            50.00299835 W
SFO1            100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 90.00 usec
PLW2            13.00199986 W
PLW12            0.36116999 W
PLW13            0.29255000 W
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF              100.6127690 MHz
WDW                      EM
SSB      0
LB                     1.00 Hz
GB       0
PC                     1.40
```



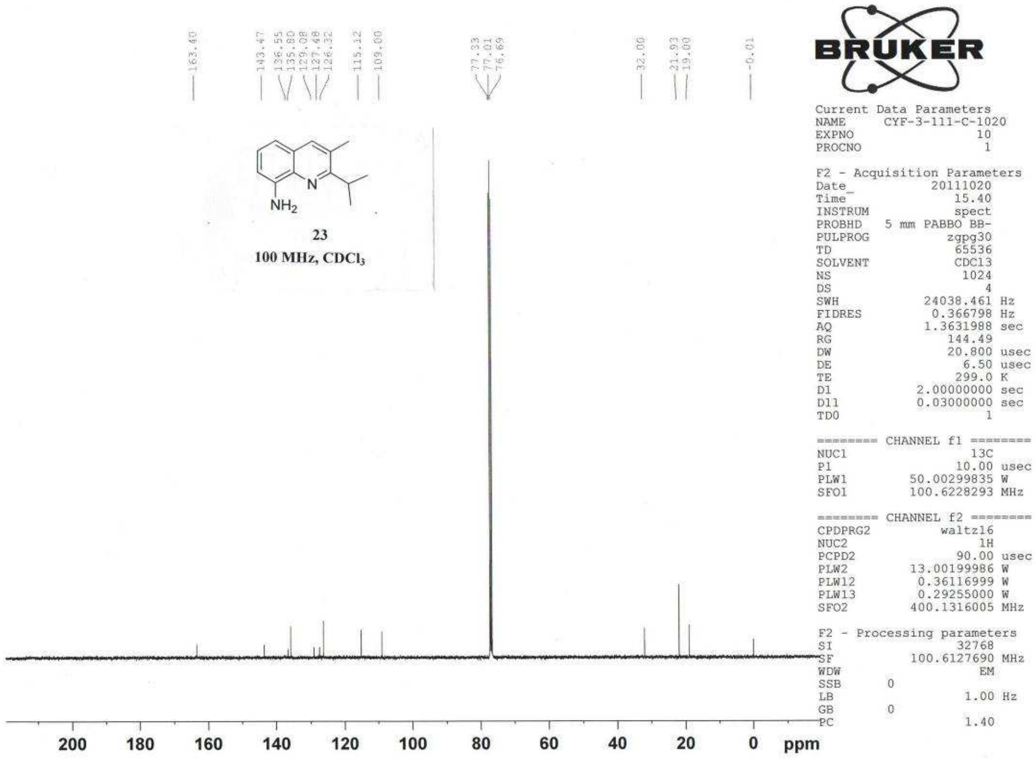
```
Current Data Parameters
NAME      CYF-3-89-H-1011
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111011
Time                22.31
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ              3.9846387 sec
RG                     85
DW                 60.800 usec
DE                   6.50 usec
TE                  297.4 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.60 usec
PLW1          13.00199986 W
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  65536
SF            400.1300000 MHz
WDW                    EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



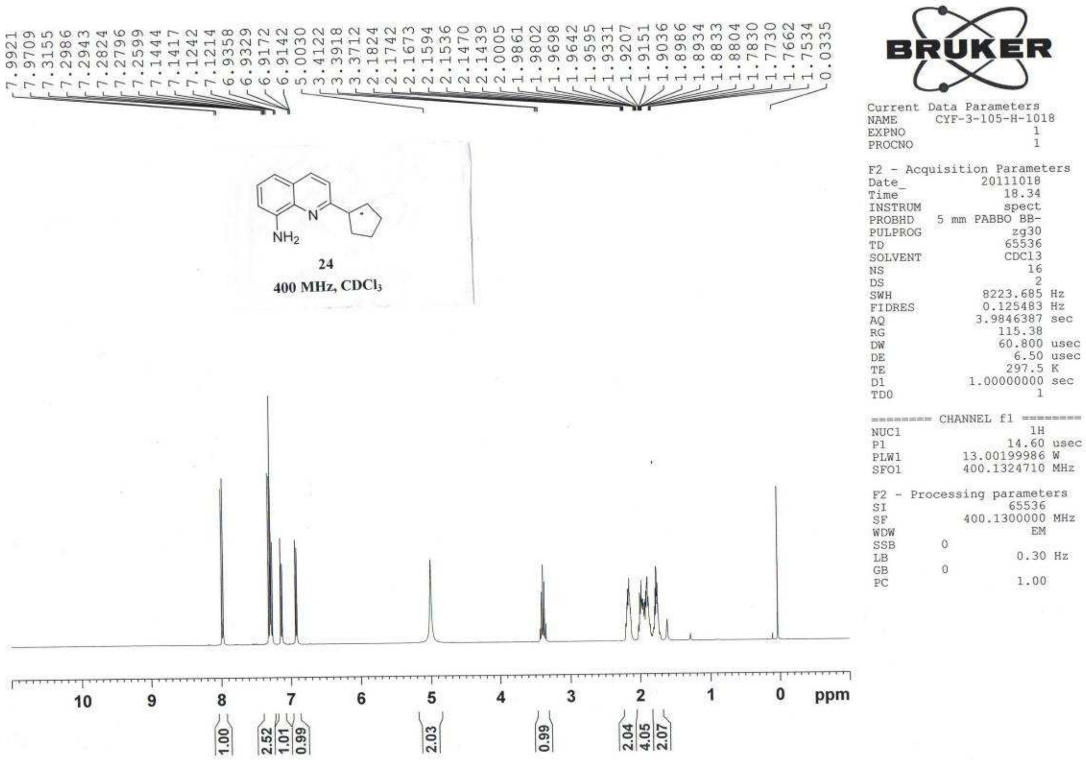
```
Current Data Parameters
NAME      CYF-3-89-C-1011
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111011
Time                22.48
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    287
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ              1.3631988 sec
RG                 209.25
DW                 20.800 usec
DE                   6.50 usec
TE                  298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PLW1          50.00299835 W
SFO1          100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PLW2          13.00199986 W
PLW12          0.36116999 W
PLW13          0.29255000 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127690 MHz
WDW                    EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```



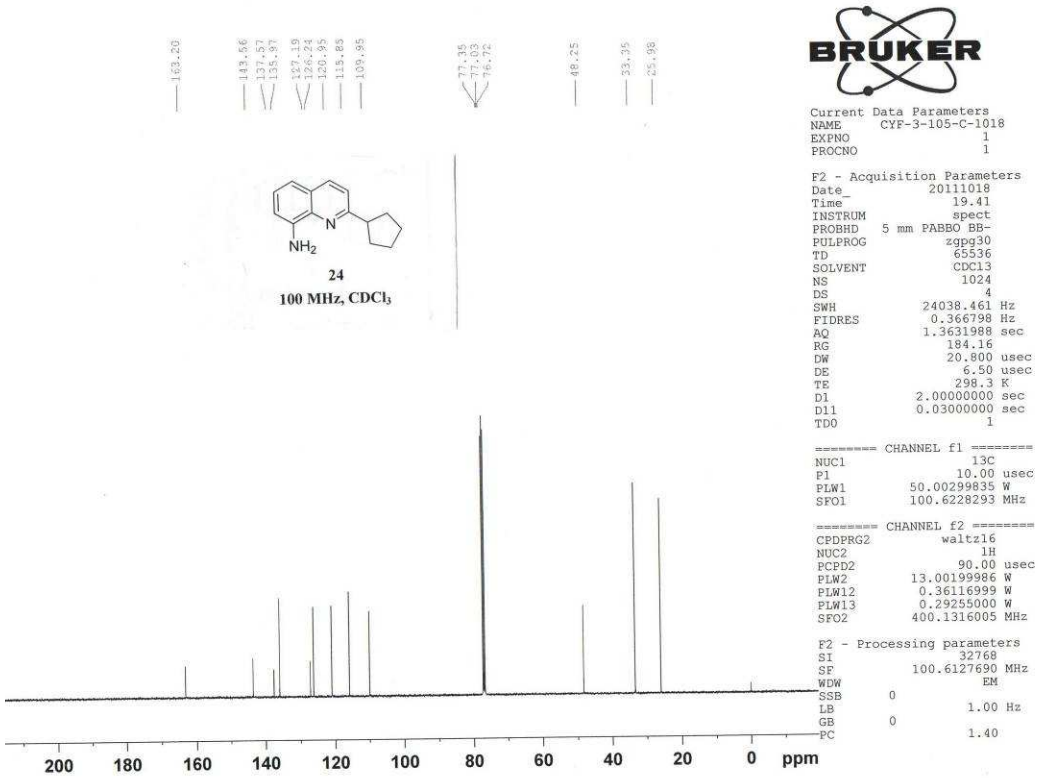
```
Current Data Parameters
NAME      CYF-3-111-H-1020
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111020
Time                 14.40
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                  209.25
DW                  60.800 usec
DE                    6.50 usec
TE                   297.9 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.60 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300094 MHz
WDW                     EM
SSB      0
LB                    0.30 Hz
GB       0
PC                    1.00
```



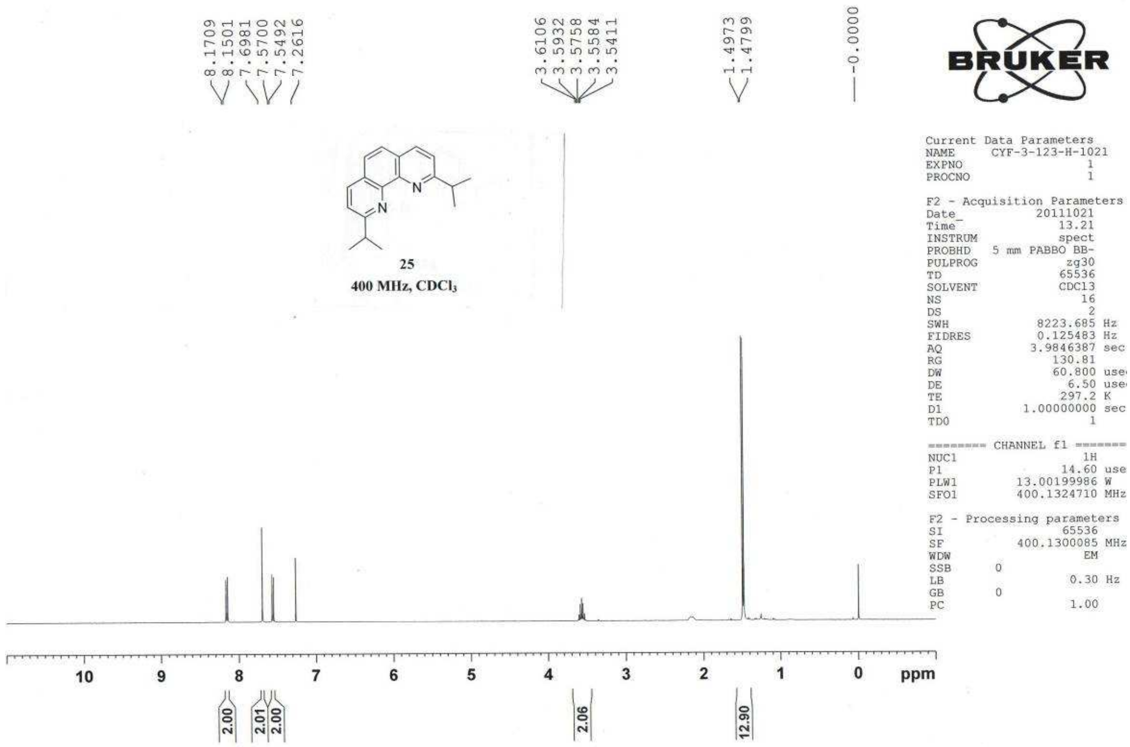
```
Current Data Parameters
NAME      CYF-3-111-C-1020
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111020
Time                 15.40
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  144.49
DW                  20.800 usec
DE                    6.50 usec
TE                   299.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.00 usec
PLW1           50.00299835 W
SFO1           100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.36116999 W
PLW13           0.29255000 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB      0
LB                    1.00 Hz
GB       0
PC                    1.40
```



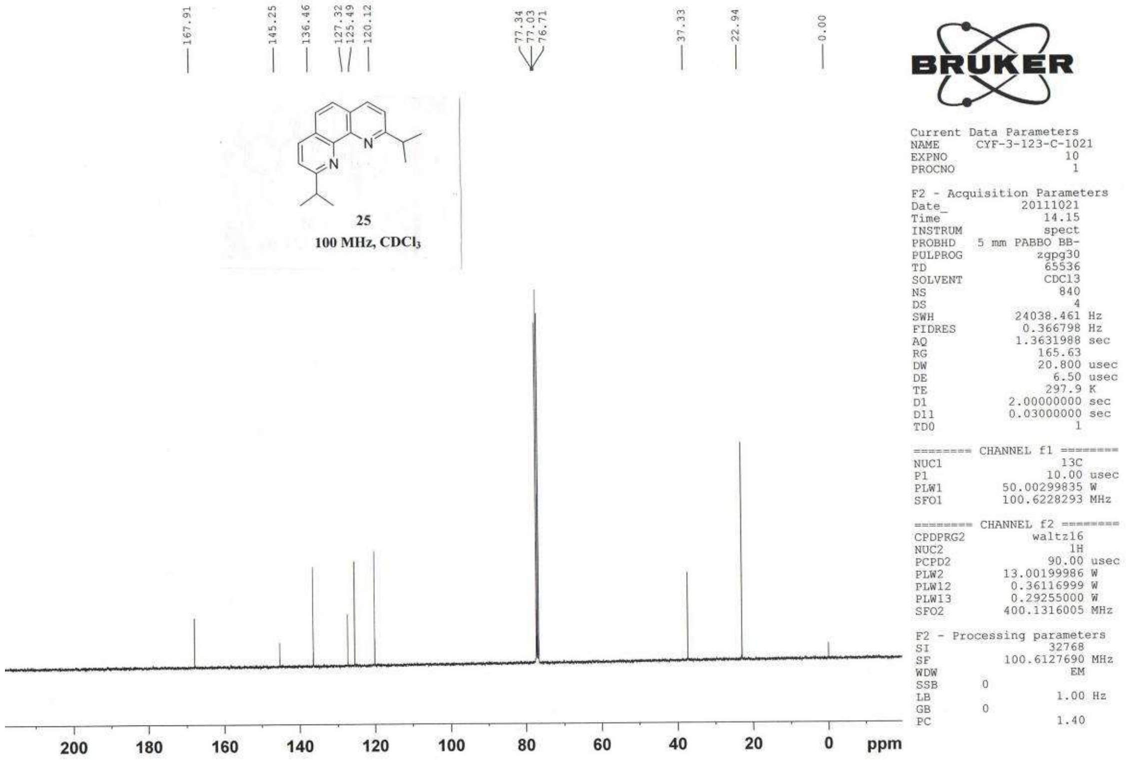
```
Current Data Parameters
NAME       CYF-3-105-H-1018
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111018
Time                  18.34
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                   115.38
DW                   60.800 usec
DE                     6.50 usec
TE                    297.5 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.60 usec
PLW1            13.00199986 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF              400.1300000 MHz
WDW                      EM
SSB       0
LB                     0.30 Hz
GB        0
PC                     1.00
```



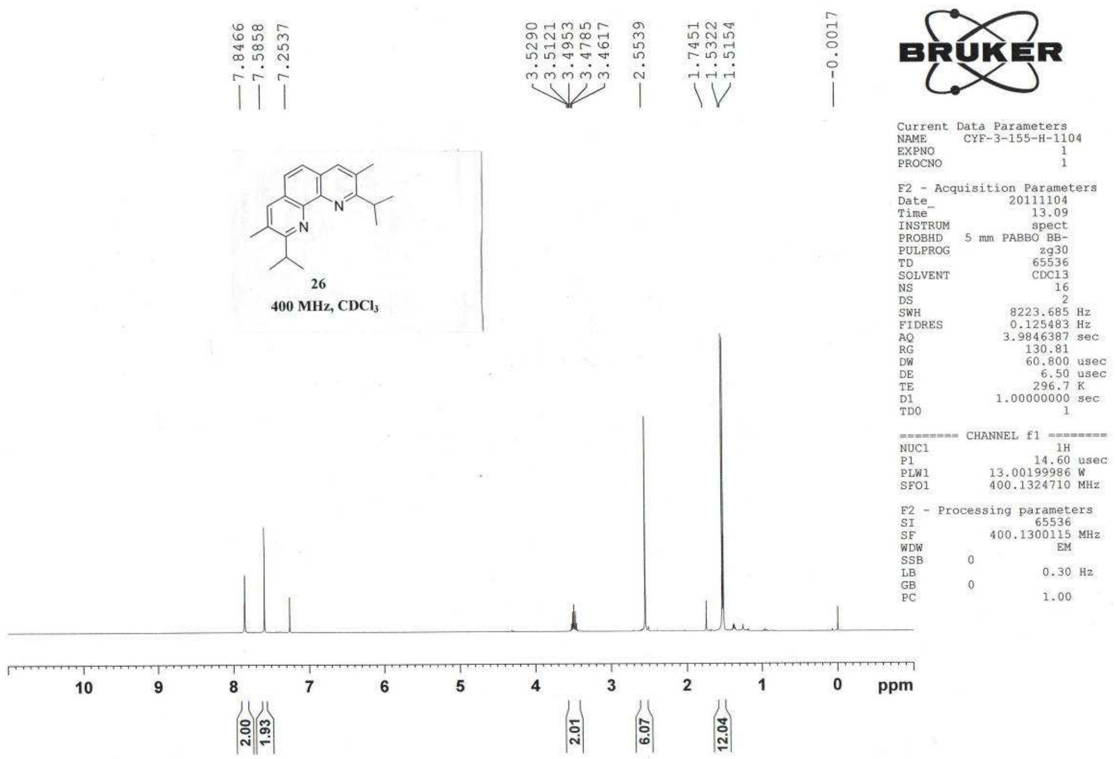
```
Current Data Parameters
NAME       CYF-3-105-C-1018
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111018
Time                  19.41
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                1.3631988 sec
RG                   184.16
DW                   20.800 usec
DE                     6.50 usec
TE                    298.3 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PLW1            50.00299835 W
SFO1            100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 90.00 usec
PLW2            13.00199986 W
PLW12            0.36116999 W
PLW13            0.29255000 W
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF              100.6127690 MHz
WDW                      EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

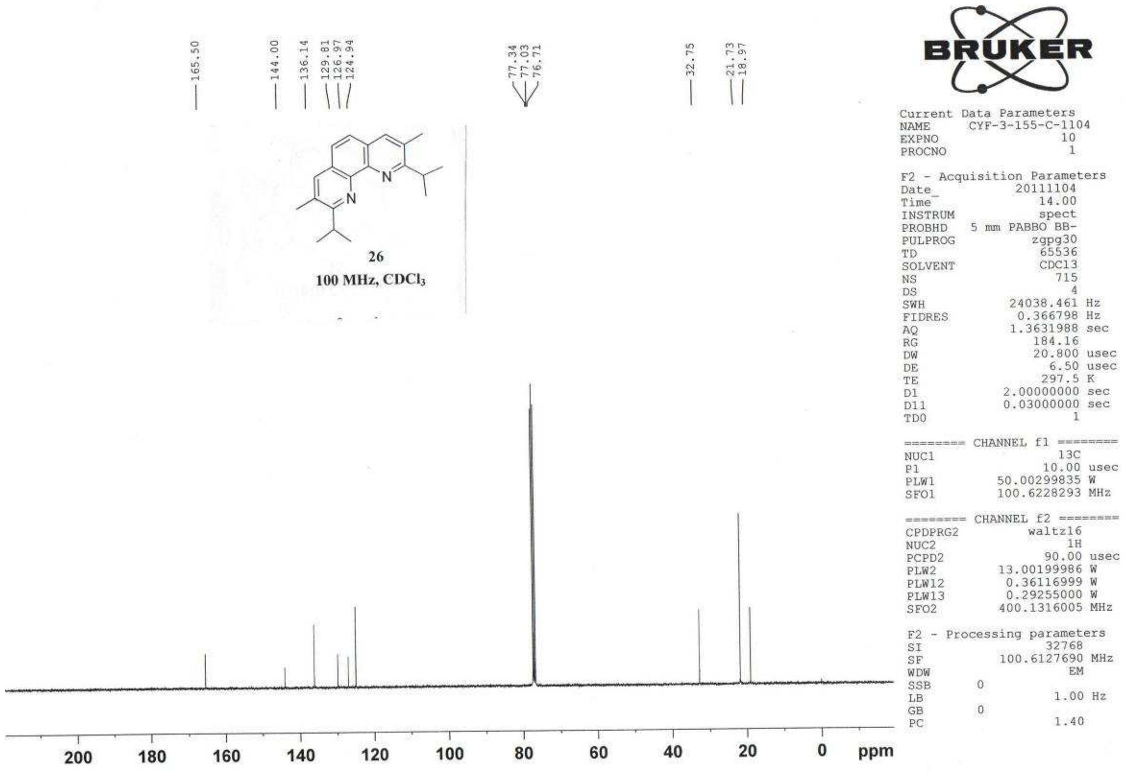


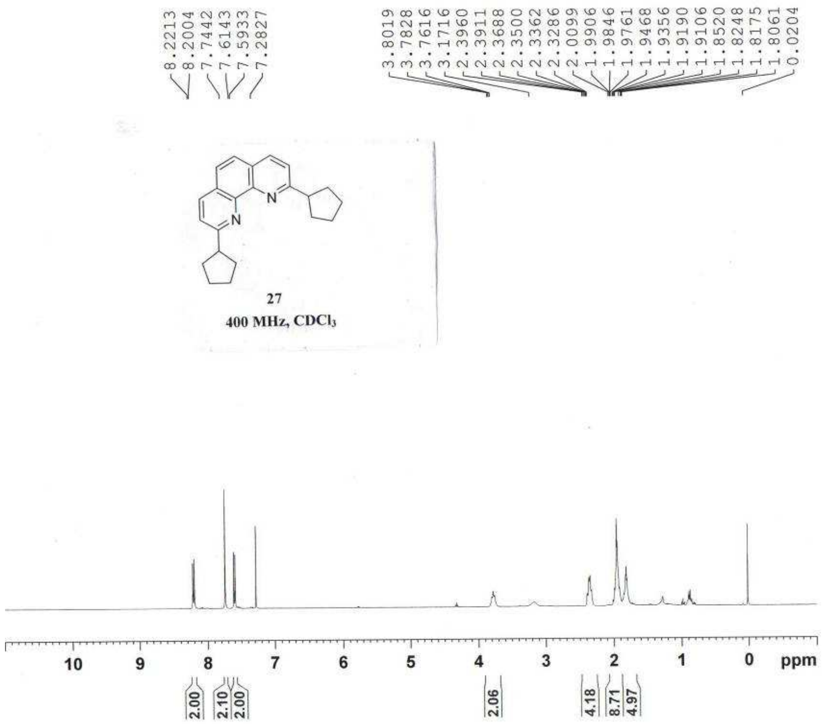
```
Current Data Parameters
NAME      CYF-3-123-H-1021
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111021
Time                 13.21
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                  130.81
DW                  60.800 use
DE                    6.50 use
TE                   297.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.60 use
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300085 MHz
WDW                     EM
SSB      0
LB                    0.30 Hz
GB       0
PC                    1.00
```



```
Current Data Parameters
NAME      CYF-3-123-C-1021
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111021
Time                 14.15
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     840
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  165.63
DW                  20.800 usec
DE                    6.50 usec
TE                   297.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.00 usec
PLW1           50.00299835 W
SFO1           100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.36116999 W
PLW13           0.29255000 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB      0
LB                    1.00 Hz
GB       0
PC                    1.40
```

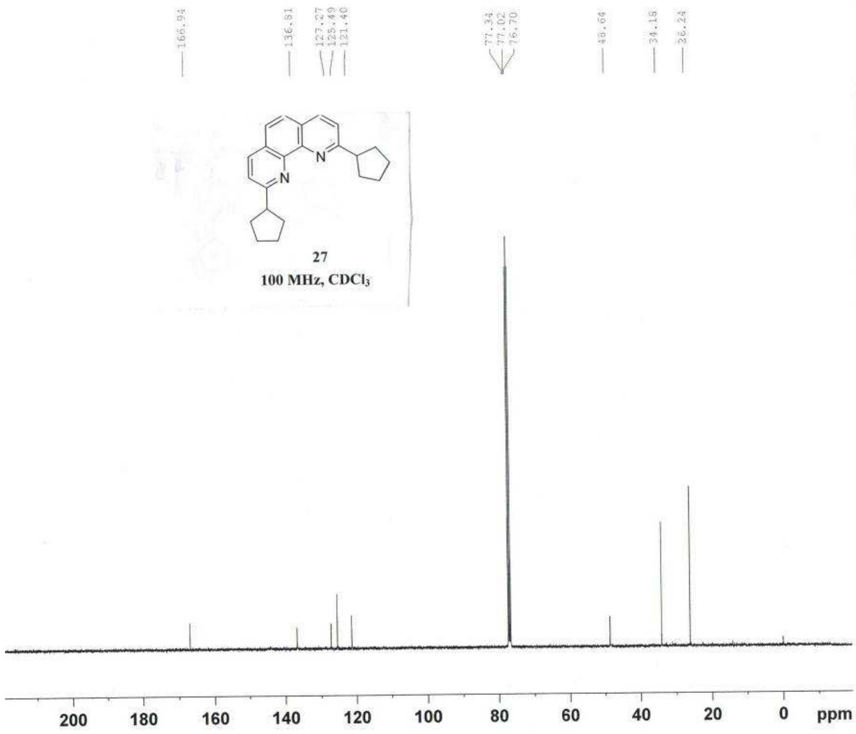

7.8466
7.5858
7.2537
3.5290
3.5121
3.4953
3.4785
3.4617
2.5539
1.7451
1.5322
1.5154
-0.0017
BRUKER
N
N
26
400 MHz, CDCl3
Current Data Parameters
NAME CYF-3-155-H-1104
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111104
Time 13.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 130.81
DW 60.800 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.60 usec
PLW1 13.00199986 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
1.93
2.01
6.07
12.04


165.50
144.00
136.14
129.81
126.97
124.94
77.34
77.03
76.71
32.75
21.73
18.97
BRUKER
N
N
26
100 MHz, CDCl3
Current Data Parameters
NAME CYF-3-155-C-1104
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111104
Time 14.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 715
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 184.16
DW 20.800 usec
DE 6.50 usec
TE 297.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 50.00299835 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 13.00199986 W
PLW12 0.36116999 W
PLW13 0.29255000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm



27
400 MHz, $CDCl_3$

```
Current Data Parameters
NAME       CYF-3-99-H-1015
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111015
Time                 22.22
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                  184.16
DW                  60.800 usec
DE                    6.50 usec
TE                   297.9 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   14.60 usec
PLW1           13.00199986 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                   65536
SF             400.1300000 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



27
100 MHz, $CDCl_3$

```
Current Data Parameters
NAME       CYF-3-99-C-1015
EXPNO                   10
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111015
Time                 22.32
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ               1.3631988 sec
RG                  209.25
DW                  20.800 usec
DE                    6.50 usec
TE                   298.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.00 usec
PLW1           50.00299835 W
SFO1           100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PLW2           13.00199986 W
PLW12           0.36116999 W
PLW13           0.29255000 W
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```