

Metal-Free Borane-Catalyzed Highly Stereoselective Hydrogenation of Pyridines

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Supporting Information

General information: All air-sensitive compounds were handled under an atmosphere of argon or in a nitrogen-filled glovebox. ^1H NMR and ^{13}C NMR spectra were recorded on Bruker AV 400 at ambient temperature with CDCl_3 as solvent and TMS as internal standard. Chemical shifts (δ) were given in ppm, referenced to the residual proton resonance of TMS (0), to the carbon resonance of the CDCl_3 (77.23). Coupling constants (J) were given in Hertz (Hz). IR spectrums were recorded on Perkin-Elmer-983 spectrometer. Optical rotations were measured with PerkinElmer 341 polarimeter. Column chromatography was performed on silica gel (200-300 mesh). All solvents were purified by conventional methods, distilled before use. Commercially available reagents were used without further purification. Piers' borane $\text{H}(\text{C}_6\text{F}_5)_2$ and pyridines were prepared according to reported methods (Parks, D. J.; Piers, W. E.; Yap, G. P. A. *Organometallics* **1998**, *17*, 5492; Liu, C.; Han, N.; Song, X.; Qiu, J. *Eur. J. Org. Chem.* **2010**, 5548).

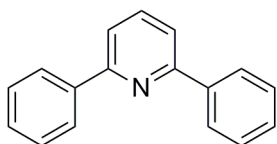
Representative procedure for hydrogenation of pyridines (Table 2, entry 1): To a glass test tube (10 mL) was added $\text{HB}(\text{C}_6\text{F}_5)_2$ (0.0086 g, 0.025 mmol), 2,3,4,5,6-pentafluorostyrene **4g** (0.0049 g, 0.025 mmol), and dry toluene (1.0 mL) in a nitrogen atmosphere glovebox. The resulting mixture was stirred at room temperature for 5 min followed by addition of 2,6-diphenylpyridine (**1a**) (0.0578 g, 0.25 mmol) and dry toluene (1.0 mL). The tube was then moved to a stainless-steel autoclave. After being sealed, the autoclave was purged three times with H_2 and the final pressure of hydrogen was adjusted to 50 bar. The reaction mixture was stirred at 100 °C for 20 h. After cooling to ambient temperature, the solvent was removed under reduced pressure. The crude residue was purified by column chromatography on silica gel using petroleum ether/ethyl acetate (10/1) to petroleum

ether/ethyl acetate/Et₃N (5/1/0.1) as the eluent to give 2,6-diphenylpiperidine (**3a**) as a white solid (0.0580 g, 98% yield).

Resolution of racemic piperidine 3C (Scheme 3): To a solution of L-(+)-tartaric acid (0.315 g, 2.10 mmol) in EtOH (4.0 mL) was added a solution of racemic piperidine **3C** in acetone (10 mL). After being stirred at room temperature for 10 min, the resulting mixture was kept still at the same temperature for 48 h, and clear crystals were formed. After filtration and washing with acetone, the crystal was treated with aq. NaOH (1N, 2 mL) and extracted with Et₂O (3 x 10 mL). The combined organic extracts were dried over anhydrous Na₂SO₄, filtered. Removal of solvents provided (*R,R*)-**3C** as a colorless liquid (0.147 g, 74% yield, >99% ee, $[\alpha]_{\text{D}}^{20} = +19.6$ (*c* 0.53, CH₂Cl₂)).

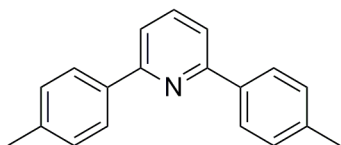
The mother liquid was treated with aq. NaOH to give (*S,S*)-**3C** as a pale yellow liquid (0.232 g, 67% ee). The resolution of (*S,S*)-**3C** (67% ee) (0.336 g, 1.77 mmol) with D-(-)-tartaric acid (0.266 g, 1.77 mmol) followed the aforementioned procedure afforded (*S,S*)-**3C** as a colorless liquid (0.203 g, 73% yield, >99% ee, $[\alpha]_{\text{D}}^{20} = -19.4$ (*c* 0.53, CH₂Cl₂)).

Characterization data of pyridines



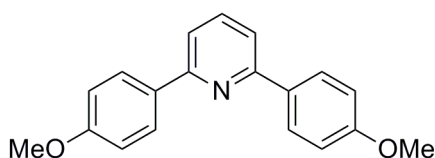
2,6-Diphenylpyridine (1a). ¹H NMR (400 MHz, CDCl₃, ppm): δ 8.15 (d, *J* = 8.0 Hz, 4H), 7.79 (t, *J* = 7.6 Hz, 1H), 6.68 (d, *J* = 7.6 Hz, 2H), 7.55-7.46 (m, 4H), 7.46-7.38 (m, 2H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 157.0, 139.7, 137.7, 129.2, 128.9, 127.2, 118.8.

Moreno-Mañas, M.; Pleixats, R.; Serra-Muns A. *Synlett* **2006**, 3001.



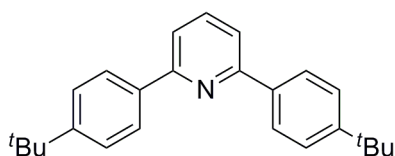
2,6-Di(*p*-tolyl)pyridine (1b). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.04 (d, $J = 8.0$ Hz, 4H), 7.73 (t, $J = 8.0$ Hz, 1H), 7.61 (d, $J = 8.0$ Hz, 2H), 7.28 (d, $J = 8.0$ Hz, 4H), 2.41 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 156.9, 139.0, 137.5, 137.0, 129.6, 127.0, 118.2, 21.5.

Spinella, S. M.; Guan, Z.-H.; Chen, J.; Zhang, X. *Synthesis* **2009**, 3094.



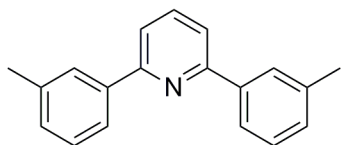
2,6-Bis(4-methoxyphenyl)pyridine (1c). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.14-8.06 (m, 4H), 7.73 (t, $J = 8.0$ Hz, 1H), 7.56 (d, $J = 8.0$ Hz, 2H), 7.05-6.97 (m, 4H), 3.87 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 160.6, 156.5, 137.5, 132.5, 128.4, 117.4, 114.2, 55.6.

Sindhuja, E.; Ramesh, R.; Liu, Y. *Dalton Trans.* **2012**, 41, 5351.



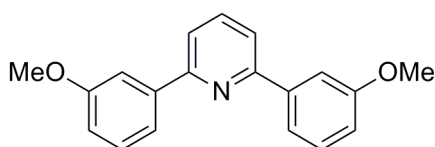
2,6-Bis(4-(*tert*-butyl)phenyl)pyridine (1d). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.08 (d, $J = 8.4$ Hz, 4H), 7.76 (t, $J = 7.6$ Hz, 1H), 7.63 (d, $J = 7.6$ Hz, 2H), 7.51 (d, $J = 8.4$ Hz, 4H), 1.37 (s, 18H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 157.0, 152.2, 137.5, 137.1, 126.9, 125.8, 118.3, 34.9, 31.6.

Wong, K. M.-C.; Hung, L.-L.; Lam, W. H.; Zhu, N.; Yam, V. W.-W. *J. Am. Chem. Soc.* **2007**, 129, 4350.



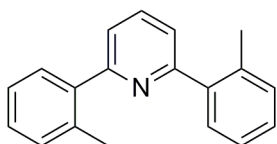
2,6-Di(*m*-tolyl)pyridine (1e). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.96 (s, 2H), 7.91 (d, J = 8.0 Hz, 2H), 7.78 (t, J = 8.0 Hz, 1H), 7.66 (d, J = 8.0 Hz, 2H), 7.42-7.34 (m, 2H), 7.24 (d, J = 7.6 Hz, 2H), 2.46 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 157.3, 139.8, 138.5, 137.5, 129.9, 128.8, 127.9, 124.4, 118.9, 21.8.

Spinella, S. M.; Guan, Z.-H.; Chen, J.; Zhang, X. *Synthesis* **2009**, 3094.



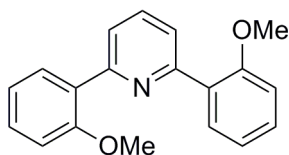
2,6-Bis(3-methoxyphenyl)pyridine (1f). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.83-7.73 (m, 3H), 7.72-7.64 (m, 4H), 7.44-7.36 (m, 2H), 6.98 (dd, J = 8.4, 2.4 Hz, 2H), 3.90 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 160.2, 156.7, 141.1, 137.7, 129.9, 119.6, 119.1, 114.9, 112.7, 55.6.

Han, P.; Zhang, H.; Qiu, X.; Ji, X.; Gao, L. *J. Mol. Catal. A: Chem.* **2008**, 295, 57.



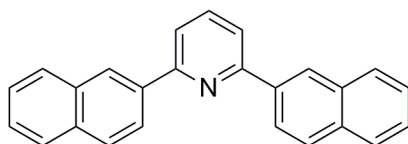
2,6-Di(*o*-tolyl)pyridine (1g). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.80 (t, J = 8.0 Hz, 1H), 7.48-7.41 (m, 2H), 7.36 (d, J = 8.0 Hz, 2H), 7.33-7.24 (m, 6H), 2.43 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 159.7, 140.9, 136.5, 136.1, 130.9, 130.0, 128.4, 126.0, 122.2, 20.8.

Fang, A. G.; Mello, J. V.; Finney, N. S. *Tetrahedron* **2004**, 60, 11075.



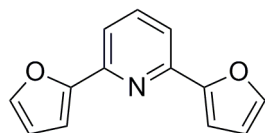
2,6-Bis(2-methoxyphenyl)pyridine (1h). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.93 (dd, $J = 7.6, 1.2$ Hz, 2H), 7.79-7.74 (m, 2H), 7.74-7.67 (m, 1H), 7.39-7.31 (m, 2H), 7.10-7.04 (m, 2H), 7.00 (d, $J = 8.4$ Hz, 2H), 3.87 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 157.3, 155.6, 135.4, 131.7, 129.9, 129.8, 123.3, 121.2, 111.6, 55.8.

Spinella, S. M.; Guan, Z.-H.; Chen, J.; Zhang, X. *Synthesis* **2009**, 3094.



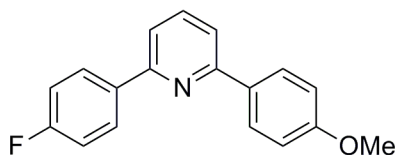
2,6-Di(naphthalen-2-yl)pyridine (1i). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.62 (s, 2H), 8.36 (d, $J = 8.8$ Hz, 2H), 8.02-7.95 (m, 4H), 7.91-7.79 (m, 5H), 7.55-7.48 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 157.0, 137.8, 137.1, 133.9, 133.7, 129.0, 128.6, 127.9, 126.7, 126.6, 126.5, 125.0, 119.2.

Kui, S. C. F.; Chui, S. S.-Y.; Che, C.-M.; Zhu, N. *J. Am. Chem. Soc.* **2006**, 128, 8297.

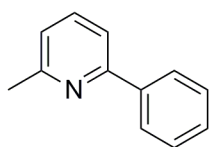


2,6-Di(furan-2-yl)pyridine (1j). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.73 (t, $J = 8.0$ Hz, 1H), 7.59-7.51 (m, 4H), 7.17-7.11 (m, 2H), 6.57-6.51 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 153.9, 149.4, 143.5, 137.4, 116.9, 112.2, 109.1.

Mousset, D.; Gillaizeau, I.; Sabatié, A.; Bouyssou, P.; Coudert, G. *J. Org. Chem.* **2006**, 71, 5993.

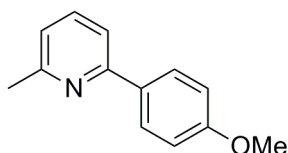


2-(4-Fluorophenyl)-6-(4-methoxyphenyl)pyridine (1k), white crystal, m.p. 157-159 °C. IR (film): 1601, 1510, 1253 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.16-8.06 (m, 4H), 7.79-7.72 (m, 1H), 7.61 (d, J = 7.6 Hz, 1H), 7.57 (d, J = 8.0 Hz, 1H), 7.20-7.13 (m, 2H), 7.02 (d, J = 8.8 Hz, 2H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 163.7 (d, J = 247.0 Hz), 160.8, 156.7, 155.8, 137.7, 135.9 (d, J = 3.0 Hz), 132.2, 128.9 (d, J = 8.0 Hz), 128.4, 118.0, 117.8, 115.7 (d, J = 22.0 Hz), 114.3, 55.6; HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{15}\text{NFO}$ ($\text{M}+\text{H}$): 280.1132, Found: 280.1131.



2-Methyl-6-phenylpyridine (1l). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.01-7.94 (m, 2H), 7.63 (t, J = 7.8 Hz, 1H), 7.51 (d, J = 7.8 Hz, 1H), 7.49-7.42 (m, 2H), 7.42-7.35 (m, 1H), 7.09 (d, J = 7.6 Hz, 1H), 2.63 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 158.6, 157.2, 140.0, 137.1, 128.9, 127.2, 121.8, 117.8, 25.0.

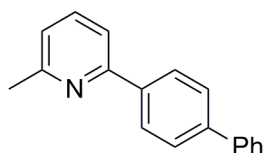
Liu, C.; Han, N.; Song, X.; Qiu, J. *Eur. J. Org. Chem.* **2010**, 5548.



2-(4-Methoxyphenyl)-6-methylpyridine (1m). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.94 (d, J = 8.8 Hz, 2H), 7.58 (dd, J = 8.0, 7.6 Hz, 1H), 7.45 (d, J = 8.0 Hz, 1H), 7.03 (d, J = 7.6 Hz, 1H), 6.98 (d, J

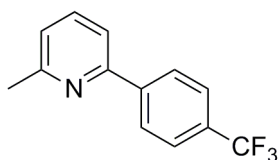
= 8.8 Hz, 2H), 3.85 (s, 3H), 2.60 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 160.5, 158.4, 156.8, 137.0, 132.6, 128.4, 121.1, 117.0, 114.2, 55.5, 25.0.

Gosmini, C.; Bassene-Ernst, C.; Durandetti, M. *Tetrahedron* **2009**, *65*, 6141.



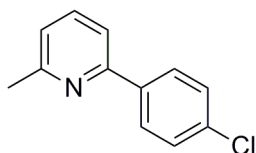
2-([1,1'-Biphenyl]-4-yl)-6-methylpyridine (1n). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.06 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.4 Hz, 2H), 7.66-7.60 (m, 3H), 7.55 (d, J = 8.0 Hz, 1H), 7.50-7.41 (m, 2H), 7.39-7.32 (m, 1H), 7.09 (d, J = 7.2 Hz, 1H), 2.64 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 158.6, 156.7, 141.7, 140.9, 138.9, 137.1, 129.0, 127.64, 127.61, 127.58, 127.3, 121.8, 117.7, 25.0.

Xia, C.; Alleyne, B.; Kwong, R. C.; Fiordeliso, J.; Brooks, J.; Adamovich, V.; Weaver, M. S. *U.S. Pat. Appl. Publ.* **2011**, US 20110227049 A1.



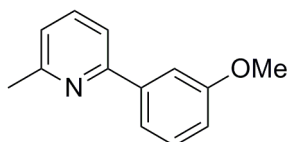
2-Methyl-6-(4-(trifluoromethyl)phenyl)pyridine (1o). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.09 (d, J = 8.0 Hz, 2H), 7.70 (d, J = 8.0 Hz, 2H), 7.69-7.61 (m, 1H), 7.53 (d, J = 7.6 Hz, 1H), 7.14 (d, J = 7.6 Hz, 1H), 2.63 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 159.0, 155.5, 143.3, 137.3, 130.7 (q, J = 32.0 Hz), 127.4, 125.8 (q, J = 4.0 Hz), 124.5 (q, J = 270.0 Hz), 122.7, 118.1, 24.9.

Yang, D. X.; Colletti, S. L.; Wu, K.; Song, M.; Li, G. Y.; Shen, H. C. *Org. Lett.* **2009**, *11*, 381.

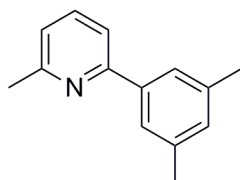


2-(4-Chlorophenyl)-6-methylpyridine (1p). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.92 (d, $J = 8.4$ Hz, 2H), 7.65-7.57 (m, 1H), 7.47 (d, $J = 8.0$ Hz, 1H), 7.41 (d, $J = 8.4$ Hz, 2H), 7.09 (d, $J = 7.6$ Hz, 1H), 2.61 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 158.7, 155.8, 138.3, 137.2, 135.0, 129.0, 128.4, 122.1, 117.5, 24.9.

Ackermann, L.; Potukuchi, H. K.; Kapdi, A. R.; Schulzke, C. *Chem. Eur. J.* **2010**, *16*, 3300.

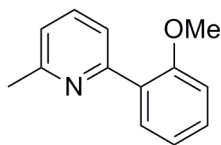


2-(3-Methoxyphenyl)-6-methylpyridine (1q), colorless oli. IR (film): 1576, 1491, 1465 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.64-7.55 (m, 2H), 7.53 (d, $J = 8.0$ Hz, 1H), 7.49 (d, $J = 7.6$ Hz, 1H), 7.39-7.31 (m, 1H), 7.08 (d, $J = 7.6$ Hz, 1H), 6.97-6.91 (m, 1H), 3.88 (s, 3H), 2.62 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 160.2, 158.5, 156.9, 141.5, 137.0, 129.8, 121.9, 119.6, 117.9, 114.8, 112.4, 55.5, 24.9; HRMS (ESI) calcd. for $\text{C}_{13}\text{H}_{14}\text{NO}$ ($\text{M}+\text{H}$): 200.1070, Found: 204.1069.



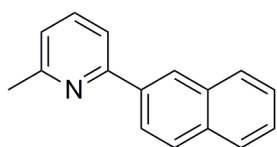
2-(3,5-Dimethylphenyl)-6-methylpyridine (1r). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.64-7.55 (m, 3H), 7.48 (d, $J = 7.6$ Hz, 1H), 7.06 (d, $J = 7.2$ Hz, 1H), 7.03 (s, 1H), 2.61 (s, 3H), 2.39 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 158.4, 157.6, 140.0, 138.3, 136.9, 130.6, 125.1, 121.6, 118.0, 25.0, 21.6.

Berman, A. M.; Lewis, J. C.; Bergman, R. G.; Ellman, J. A. *J. Am. Chem. Soc.* **2008**, *130*, 14926.



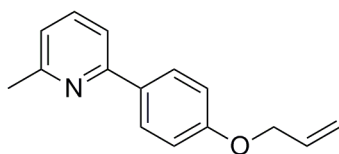
2-(2-Methoxyphenyl)-6-methylpyridine (1s). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.76-7.70 (m, 1H), 7.63-7.52 (m, 2H), 7.38-7.30 (m, 1H), 7.10-7.02 (m, 2H), 6.98 (d, $J = 8.4$ Hz, 1H), 3.83 (s, 3H), 2.61 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 158.2, 157.1, 155.7, 136.0, 131.4, 129.82, 129.77, 122.2, 121.4, 121.3, 111.5, 55.8, 24.9.

Lohse, O.; Thevenin, P.; Waldvogel, E. *Synlett* **1999**, 45.



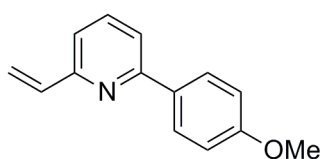
2-Methyl-6-(naphthalen-2-yl)pyridine (1t). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.47 (s, 1H), 8.14 (dd, $J = 8.4$ Hz, 1.2 Hz, 1H), 7.97-7.87 (m, 2H), 7.87-7.81 (m, 1H), 7.71-7.62 (m, 2H), 7.54-7.44 (m, 2H), 7.16-7.08 (m, 1H), 2.67 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 158.7, 157.0, 137.3, 137.1, 133.8, 133.7, 128.9, 128.6, 127.9, 126.53, 126.48, 126.4, 125.0, 121.9, 118.1, 25.0.

Milbank, J. B. J.; Knauer, C. S.; Augelli-Szafran, C. E. et al. *Bioorg. Med. Chem. Lett.* **2007**, *17*, 4415.

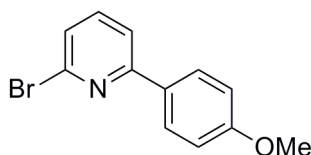


2-(4-(Allyloxy)phenyl)-6-methylpyridine (1u), white solid, m.p. 50-51°C. IR (film): 1649, 1608,

1513 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.96-7.89 (m, 2H), 7.56 (dd, $J = 7.6, 7.6$ Hz, 1H), 7.43 (d, $J = 7.6$ Hz, 1H), 7.01 (d, $J = 7.6$ Hz, 1H), 7.02-6.94 (m, 2H), 6.13-6.00 (m, 1H), 5.42 (dd, $J = 17.2, 1.6$ Hz, 1H), 5.29 (dd, $J = 10.8, 1.6$ Hz, 1H), 4.57 (d, 5.2 Hz, 2H), 2.59 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 159.4, 158.3, 156.7, 136.9, 133.3, 132.7, 128.4, 121.1, 117.9, 117.0, 115.0, 69.0, 24.9; HRMS (ESI) calcd. for $\text{C}_{15}\text{H}_{16}\text{NO}$ ($\text{M}+\text{H}$): 226.1227, Found: 226.1224.

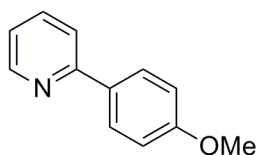


2-(4-Methoxyphenyl)-6-vinylpyridine (1v), white solid, m.p. 67-69 $^{\circ}\text{C}$. IR (film): 1608, 1452, 1260 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.05-7.97 (m, 2H), 7.68-7.61 (m, 1H), 7.53 (d, $J = 8.0$ Hz, 1H), 7.19 (d, $J = 7.6$ Hz, 1H), 7.02-6.94 (m, 2H), 6.87 (dd, $J = 17.6$ Hz, 10.8 Hz, 1H), 6.33 (dd, $J = 17.6$ Hz, 1.2 Hz, 1H), 5.49 (d, 10.8 Hz, 1H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 160.7, 156.7, 155.4, 137.4, 137.2, 132.2, 128.4, 119.2, 118.6, 118.2, 114.2, 55.3; HRMS (ESI) calcd. for $\text{C}_{14}\text{H}_{14}\text{NO}$ ($\text{M}+\text{H}$): 212.1070, Found: 212.1068.



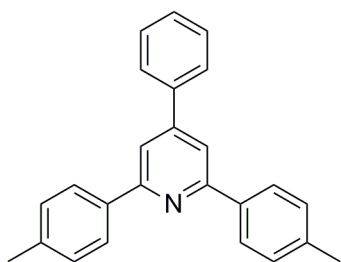
2-Bromo-6-(4-methoxyphenyl)pyridine (1w). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.65 (d, $J = 4.8$ Hz, 1H), 7.95 (d, $J = 8.8$ Hz, 2H), 7.75-7.62 (m, 2H), 7.20-7.13 (m, 1H), 7.00 (d, $J = 8.8$ Hz, 2H), 3.86 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 160.6, 157.3, 149.7, 136.8, 132.2, 128.4, 121.6, 120.0, 114.3, 55.5.

Smith, D. J.; Blake, A. J.; Wilson, C.; Champness N. R. *Dalton Trans.* **2011**, 40, 12257.



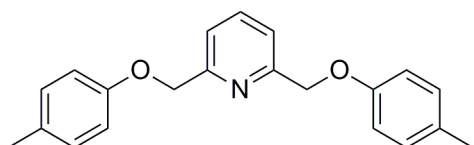
2-(4-Methoxyphenyl)pyridine (1x). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.65 (d, $J = 4.8$ Hz, 1H), 7.95 (d, $J = 8.8$ Hz, 2H), 7.75-7.62 (m, 2H), 7.20-7.13 (m, 1H), 7.00 (d, $J = 8.8$ Hz, 2H), 3.86 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 160.6, 157.3, 149.7, 136.8, 132.2, 128.4, 121.6, 120.0, 114.3, 55.5.

Ackermann, L.; Vicente, R.; Potukuchi, H. K.; Pirovano, V. *Org. Lett.* **2010**, 12, 5032.



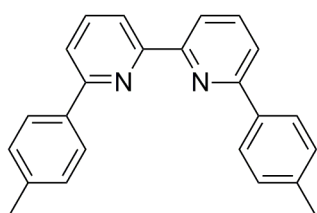
4-Phenyl-2,6-di(p-tolyl)pyridine (1y). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.09 (d, $J = 8.0$ Hz, 4H), 7.82 (s, 2H), 7.72 (d, $J = 7.2$ Hz, 2H), 7.54-7.47 (m, 2H), 7.47-7.41 (m, 1H), 7.30 (d, $J = 8.0$ Hz, 4H), 2.42 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 157.6, 150.2, 139.5, 139.1, 137.1, 129.6, 129.3, 129.1, 127.4, 127.2, 116.7, 21.5.

Huang, H.; Ji, X.; Wu, W.; Huang, L.; Jiang, H. *J. Org. Chem.* **2013**, 78, 3774.



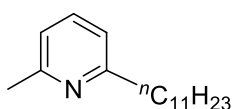
2,6-Bis((p-tolyloxy)methyl)pyridine (1z). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.70 (t, $J = 8.0$ Hz, 1H), 7.43 (d, $J = 8.0$ Hz, 2H), 7.08 (d, $J = 8.4$ Hz, 4H), 6.88 (d, $J = 8.4$ Hz, 4H), 5.18 (s, 4H), 2.28 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 157.3, 156.5, 137.8, 130.6, 130.2, 120.2, 114.8, 70.8, 20.7.

Carvalho, J. F. S.; Louvel, J.; Doornbos, M. L. J.; Klaasse, E.; Yu, Z.; Brussee, J.; Ijzerman, A. P. *J. Med. Chem.* **2013**, 56, 2828.



6,6'-Di(p-tolyl)-2,2'-bipyridine (1D). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 8.57 (d, $J = 7.6$ Hz, 2H), 8.07 (d, $J = 8.0$ Hz, 4H), 7.92-7.83 (m, 2H), 7.74 (d, $J = 7.6$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 4H), 2.43 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 156.5, 156.1, 139.1, 137.7, 136.9, 129.7, 127.0, 120.1, 119.4, 21.5.

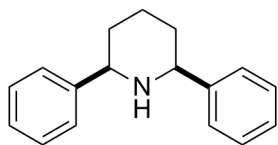
Kim, J.; Chang, S. *J. Am. Chem. Soc.* **2010**, 132, 10272-10274.



2-Methyl-6-undecylpyridine (1E). ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.46 (dd, $J = 7.6$, 7.6 Hz, 1H), 6.94 (d, $J = 7.6$ Hz, 1H), 6.93 (d, $J = 7.6$ Hz, 1H), 2.74 (t, $J = 8.0$ Hz, 2H), 2.53 (s, 3H), 1.76-1.61 (m, 2H), 1.43-1.16 (m, 16H), 0.88 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 162.2, 157.9, 136.6, 120.5, 119.6, 38.9, 32.1, 30.5, 29.85, 29.83, 29.78, 29.74, 29.71, 29.6, 24.8, 22.9, 14.3.

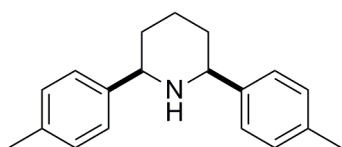
Pianaro, A.; Fox, E. G. P.; Bueno, O. C.; Marsaioli, A. J. *Tetrahedron: Asymmetry* **2012**, 23, 635.

Characterization data of piperidines



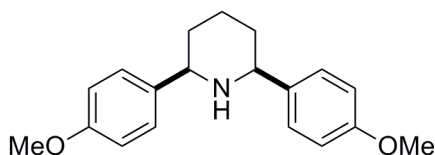
Cis-2,6-diphenylpiperidine (3a), white solid. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.45 (d, $J = 7.2$ Hz, 4H), 7.36-7.26 (m, 4H), 7.23-7.18 (m, 2H), 3.85-3.77 (m, 2H), 2.09-1.90 (m, 2H), 1.85-1.77 (m, 2H), 1.70-1.45 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 145.9, 128.5, 127.2, 127.0, 62.8, 34.9, 26.0.

Coia, N.; Mokhtari, N.; Vasse, J-L.; Szymoniak, J. *Org. Lett.* **2011**, *13*, 6292.



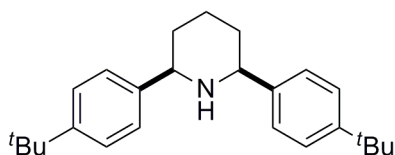
Cis-2,6-di(p-tolyl)piperidine (3b), white solid. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.33 (d, $J = 8.0$ Hz, 4H), 7.12 (d, $J = 8.0$ Hz, 4H), 3.81-3.74 (m, 2H), 2.32 (s, 6H), 2.01-1.90 (m, 1H), 1.88-1.65 (m, 3H), 1.65-1.47 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 143.2, 136.7, 129.2, 126.8, 62.6, 35.0, 26.1, 21.3.

Durant, G. J.; Maillard, M. ;Guo, J. Q. *PCT Int. Appl.* **1998**, WO 9806401 A1.

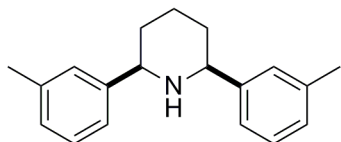


Cis-2,6-bis(4-methoxyphenyl)piperidine (3c), white solid, m.p. 115-118 °C. IR (film): 3309, 1611, 1583, 1514, 1252 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.35 (d, $J = 8.4$ Hz, 4H), 6.85 (d, $J =$

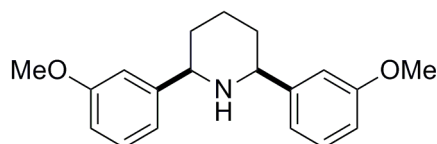
8.4 Hz, 4H), 3.79 (s, 6H), 3.82-3.70 (m, 2H), 2.00-1.90 (m, 1H), 1.81-1.66 (m, 3H), 1.65-1.40 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 158.8, 138.3, 128.0, 113.8, 62.3, 55.5, 34.9, 26.0; HRMS (EI) Calcd. for $\text{C}_{19}\text{H}_{23}\text{NO}_2$ (M): 297.1729, Found: 297.1732.



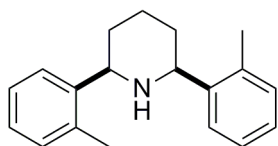
Cis-2,6-bis(4-(tert-butyl)phenyl)piperidine (3d), white solid, m.p. 137-139 °C. IR (film): 3357, 1659, 1632, 1361, 828 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.37 (d, J = 8.4 Hz, 4H), 7.33 (d, J = 8.4 Hz, 4H), 3.83-3.75 (m, 2H), 2.02-1.90 (m, 1H), 1.85-1.70 (m, 3H), 1.70-1.44 (m, 3H), 1.31 (s, 18H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 149.9, 143.0, 126.6, 125.4, 62.4, 34.7, 31.6, 26.1; HRMS (EI) Calcd. for $\text{C}_{25}\text{H}_{35}\text{N}$ (M): 349.2770, Found: 349.2773.



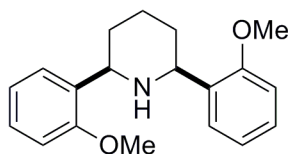
Cis-2,6-di(*m*-tolyl)piperidine (3e), colorless oil. IR (film): 3304, 1608, 783 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.30-7.15 (m, 6H), 7.09-7.00 (m, 2H), 3.82-3.72 (m, 2H), 2.35 (s, 6H), 2.02-1.92 (m, 1H), 1.85-1.73 (m, 3H), 1.72-1.44 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 145.9, 138.1, 128.4, 127.9, 127.6, 124.0, 62.9, 34.8, 26.1, 21.7; HRMS (EI) Calcd. for $\text{C}_{19}\text{H}_{23}\text{N}$ (M): 265.1830, Found: 265.1833.



Cis-2,6-bis(3-methoxyphenyl)piperidine (3f), colorless oil. IR (film): 3305, 1601, 1486 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.27-7.18 (m, 2H), 7.06-6.98 (m, 4H), 6.81-6.74 (m, 2H), 3.81 (s, 6H), 3.84-3.73 (m, 2H), 2.00-1.90 (m, 1H), 1.87-1.74 (m, 3H), 1.74-1.42 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 159.8, 147.8, 129.4, 119.3, 112.6, 112.4, 62.7, 55.4, 35.0, 26.0; HRMS (EI) Calcd. for $\text{C}_{19}\text{H}_{23}\text{NO}_2$ (M): 297.1729, Found: 297.1732.

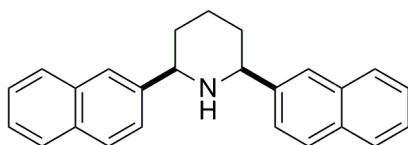


Cis-2,6-di(o-tolyl)piperidine (3g), white solid, m.p. 101-102 $^{\circ}\text{C}$. IR (film): 3302, 1602, 1460 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.73 (d, $J = 7.6$ Hz, 2H), 7.25-7.17 (m, 2H), 7.17-7.09 (m, 4H), 4.08-3.99 (m, 2H), 2.39 (s, 6H), 2.03-1.90 (m, 1H), 1.87-1.77 (m, 2H), 1.77-1.56 (m, 2H), 1.56-1.42 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 144.0, 134.8, 130.4, 126.7, 126.5, 126.4, 58.9, 33.5, 26.3, 19.4; HRMS (EI) Calcd. for $\text{C}_{19}\text{H}_{23}\text{N}$ (M): 265.1830, Found: 265.1834.

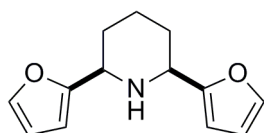


Cis-2,6-bis(2-methoxyphenyl)piperidine (3h), white solid, m.p. 117-119 $^{\circ}\text{C}$. IR (film): 3293, 1600, 1585, 1491, 1239 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.59 (d, $J = 7.2$ Hz, 2H), 7.24-7.15 (m, 2H), 6.99-6.91 (m, 2H), 6.85 (d, $J = 8.0$ Hz, 2H), 4.27-4.19 (m, 2H), 3.83 (s, 6H), 2.07-1.91 (m, 2H),

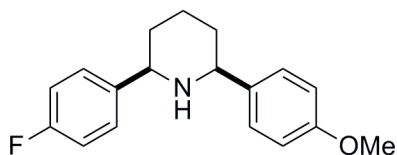
1.91-1.84 (m, 2H), 1.79-1.64 (m, 1H), 1.58-1.41 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 156.8, 134.0, 127.7, 127.2, 120.9, 110.3, 55.6, 55.5, 32.6, 26.1; HRMS (EI) Calcd. for $\text{C}_{19}\text{H}_{23}\text{NO}_2$ (M): 297.1729, Found: 297.1733.



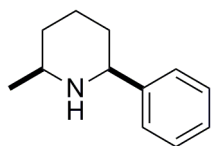
Cis-2,6-di(naphthalen-2-yl)piperidine (3i), white solid, m.p. 175-176 °C. IR (film): 3309, 3053, 1600 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.92 (s, 2H), 7.89-7.74 (m, 6H), 7.63 (d, J = 8.4 Hz, 2H), 7.50-7.37 (m, 4H), 4.08-4.00 (m, 2H), 2.18-1.85 (m, 4H), 1.84-1.57 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 143.4, 133.7, 133.0, 128.1, 128.0, 127.8, 126.1, 125.8, 125.6, 125.1, 62.9, 34.9, 26.1; HRMS (EI) Calcd. for $\text{C}_{25}\text{H}_{23}\text{N}$ (M): 337.1830, Found: 337.1834.



Cis-2,6-di(furan-2-yl)piperidine (3j), colorless oil. IR (film): 3323, 1597, 1505, 1297 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.37-7.28 (m, 2H), 6.35-6.25 (m, 2H), 6.20-6.13 (m, 2H), 3.94-3.85 (m, 2H), 2.22 (brs, 1H), 2.10-1.90 (m, 3H), 1.73-1.55 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 157.3, 141.5, 110.2, 104.7, 55.1, 30.4, 24.6; HRMS (EI) Calcd. for $\text{C}_{13}\text{H}_{15}\text{NO}_2$ (M): 217.1103, Found: 217.1105.

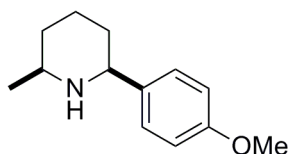


Cis-2-(4-fluorophenyl)-6-(4-methoxyphenyl)piperidine (3k), colorless oil. IR (film): 3309, 1609, 1510 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.43-7.36 (m, 2H), 7.35 (d, $J = 8.8$ Hz, 2H), 7.03-6.92 (m, 2H), 6.85 (d, $J = 8.8$ Hz, 2H), 3.78 (s, 3H), 3.81-3.70 (m, 2H), 2.03-1.89 (m, 1H), 1.81-1.70 (m, 3H), 1.67-1.40 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 162.0 (d, $J = 242.0$ Hz), 158.8, 141.8 (d, $J = 2.0$ Hz), 138.2, 128.4 (d, $J = 7.0$ Hz), 128.0, 115.2 (d, $J = 21.0$ Hz), 113.9, 62.17, 62.15, 55.4, 35.1, 34.8, 25.9; HRMS (EI) Calcd. for $\text{C}_{18}\text{H}_{20}\text{FNO}$ (M): 285.1529, Found: 285.1533.



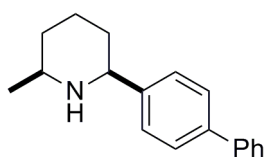
Cis-2-methyl-6-phenylpiperidine (3l), colorless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.37 (d, $J = 7.6$ Hz, 2H), 7.35-7.26 (m, 2H), 7.26-7.15 (m, 1H), 3.72-3.58 (m, 1H), 2.86-2.72 (m, 1H), 1.95-1.87 (m, 1H), 1.81-1.70 (m, 1H), 1.69-1.57 (m, 2H), 1.57-1.37 (m, 2H), 1.29-1.12 (m, 1H), 1.11 (d, $J = 6.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 145.8, 128.5, 127.2, 126.9, 62.7, 53.4, 34.5, 34.1, 26.0, 23.3.

Poerwono, H.; Higashiyama, K.; Yamauchi, T.; Kubo, H.; Ohmiya, S.; Takahashi, H. *Tetrahedron* **1998**, *54*, 13955.

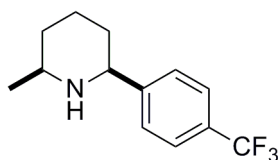


Cis-2-(4-methoxyphenyl)-6-methylpiperidine (3m), colorless oil. IR (film): 3312, 1611, 1513,

1246 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.29 (d, $J = 8.8$ Hz, 2H), 6.85 (d, $J = 8.8$ Hz, 2H), 3.78 (s, 3H), 3.65-3.53 (m, 1H), 2.86-2.72 (m, 1H), 1.92-1.80 (m, 1H), 1.76-1.68 (m, 1H), 1.68-1.61 (m, 1H), 1.61-1.37 (m, 3H), 1.21-1.10 (m, 1H), 1.10 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 158.7, 138.0, 127.9, 113.8, 62.0, 55.4, 53.4, 34.5, 34.0, 25.6, 23.3; HRMS (ESI) calcd. for $\text{C}_{13}\text{H}_{20}\text{NO}$ ($\text{M}+\text{H}$): 206.1539, Found: 206.1539.

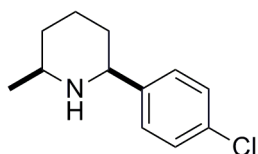


Cis-2-([1,1'-biphenyl]-4-yl)-6-methylpiperidine (3n), colorless oil. IR (film): 3307, 1599, 1486, 1302, 1114 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.57 (d, $J = 8.0$ Hz, 2H), 7.54 (d, $J = 8.0$ Hz, 2H), 7.47-7.37 (m, 4H), 7.35-7.28 (m, 1H), 3.75-3.66 (m, 1H), 2.88-2.75 (m, 1H), 1.94-1.82 (m, 1H), 1.82-1.73 (m, 1H), 1.70-1.55 (m, 2H), 1.55-1.40 (m, 2H), 1.26-1.14 (m, 1H), 1.13 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 144.9, 141.3, 140.1, 128.9, 127.36, 127.30, 127.26, 62.4, 53.4, 34.5, 34.1, 25.6, 23.3; HRMS (ESI) calcd. for $\text{C}_{18}\text{H}_{22}\text{N}$ ($\text{M}+\text{H}$): 252.1747, Found: 252.1746.

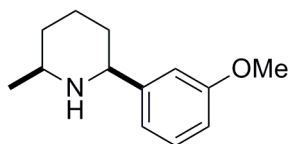


Cis-2-methyl-6-(4-(trifluoromethyl)phenyl)piperidine (3o), colorless oil. IR (film): 3268, 1621, 1326, 1125 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.56 (d, $J = 8.0$ Hz, 2H), 7.49 (d, $J = 8.0$ Hz, 2H), 4.15-3.98 (m, 1H), 2.88-2.73 (m, 1H), 1.93-1.83 (m, 1H), 1.80-1.66 (m, 1H), 1.66-1.54 (m, 1H), 1.54-1.33 (m, 3H), 1.31-1.12 (m, 1H), 1.12 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm):

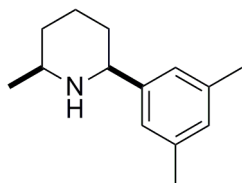
δ 149.8, 129.4 (q, $J = 32$ Hz), 127.2, 125.5 (q, $J = 4.0$ Hz), 124.5 (q, $J = 270$ Hz), 62.2, 53.2, 34.7, 33.9, 25.4, 23.2; HRMS (ESI) calcd. for $C_{13}H_{17}NF_3$ (M+H): 244.1308, Found: 204.1306.



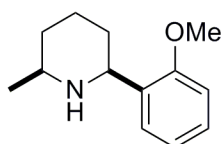
Cis-2-(4-chlorophenyl)-6-methylpiperidine (3p), colorless oil. IR (film): 3309, 1490, 1302, 1188 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$, ppm): δ 7.33-7.28 (m, 2H), 7.28-7.23 (m, 2H), 3.62 (dd, $J = 10.8$ Hz, 2.4 Hz, 1H), 2.86-2.73 (m, 1H), 1.91-1.81 (m, 1H), 1.76-1.68 (m, 1H), 1.68-1.55 (m, 1H), 1.55-1.26 (m, 3H), 1.23-1.12 (m, 1H), 1.12 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 144.3, 132.7, 128.6, 128.3, 62.0, 53.3, 34.6, 33.9, 25.5, 23.3; HRMS (ESI) calcd. for $C_{12}H_{17}NCl$ (M+H): 210.1044, Found: 210.1043.



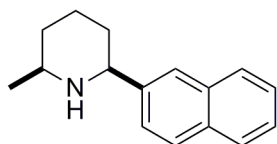
Cis-2-(3-methoxyphenyl)-6-methylpiperidine (3q), colorless oil. IR (film): 3311, 1601, 1465 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$, ppm): δ 7.26-7.18 (m, 1H), 6.99-6.92 (m, 2H), 6.77 (dd, $J = 8.0, 2.0$ Hz, 1H), 3.80 (s, 3H), 3.68-3.59 (m, 1H), 2.87-2.72 (m, 1H), 1.91-1.82 (m, 1H), 1.80-1.72 (m, 1H), 1.68-1.51 (m, 2H), 1.51-1.35 (m, 2H), 1.28-1.12 (m, 1H), 1.11 (d, $J = 6.0$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 159.9, 147.5, 129.5, 119.3, 112.6, 112.5, 62.7, 55.4, 53.3, 34.5, 34.0, 25.6, 23.3; HRMS (ESI) calcd. for $C_{13}H_{20}NO$ (M+H): 206.1539, Found: 206.1542.



Cis-2-(3,5-dimethylphenyl)-6-methylpiperidine (3r), colorless oil. IR (film): 3304, 1606, 1461 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 6.99 (s, 2H), 6.87 (s, 1H), 3.63-3.52 (m, 1H), 2.85-2.71 (m, 1H), 2.30 (s, 6H), 1.93-1.82 (m, 1H), 1.78-1.68 (m, 1H), 1.68-1.38 (m, 4H), 1.28-1.12 (m, 1H), 1.11 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 145.6, 138.0, 128.0, 124.7, 62.8, 53.5, 34.4, 34.1, 25.6, 23.3, 21.5; HRMS (ESI) calcd. for $\text{C}_{14}\text{H}_{22}\text{N}$ (M+H): 204.1747, Found: 204.1749.

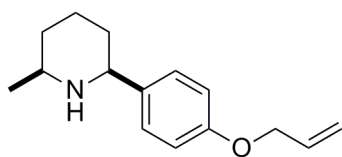


Cis-2-(2-methoxyphenyl)-6-methylpiperidine (3s), colorless oil. IR (film): 3305, 1601, 1492, 1241 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.45 (dd, $J = 7.6, 1.2$ Hz, 1H), 7.24-7.15 (m, 1H), 6.98-6.90 (m, 1H), 6.85 (d, $J = 8.4$ Hz, 1H), 4.07-3.97 (m, 1H), 3.83 (s, 3H), 2.90-2.76 (m, 1H), 1.92-1.83 (m, 1H), 1.83-1.61 (m, 3H), 1.61-1.32 (m, 2H), 1.28-1.12 (m, 1H), 1.11 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 156.7, 133.5, 127.8, 127.0, 120.9, 110.4, 55.5, 55.4, 53.4, 34.4, 32.3, 25.6, 23.4; HRMS (ESI) calcd. for $\text{C}_{13}\text{H}_{20}\text{NO}$ (M+H): 206.1539, Found: 206.1541.

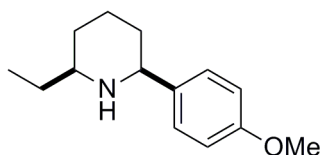


Cis-2-methyl-6-(naphthalen-2-yl)piperidine (3t), white solid, m.p. 61-62 $^{\circ}\text{C}$. IR (film): 3307, 1633, 1601, 1303, 1114 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.88-7.74 (m, 4H), 7.50 (d, $J = 8.4$ Hz,

1H), 7.48-7.38 (m, 2H), 3.89-3.72 (m, 1H), 2.95-2.76 (m, 1H), 1.97-1.88 (m, 1H), 1.88-1.77 (m, 1H), 1.74-1.63 (m, 2H), 1.62-1.45 (m, 2H), 1.29-1.17 (m, 1H), 1.15 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 143.3, 133.7, 133.0, 128.1, 128.0, 127.8, 126.1, 125.8, 125.6, 124.9, 62.7, 53.5, 34.6, 34.1, 25.7, 23.3; HRMS (ESI) calcd. for $\text{C}_{16}\text{H}_{20}\text{N}$ ($\text{M}+\text{H}$): 226.1590, Found: 226.1589.

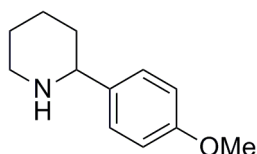


Cis-2-(4-(allyloxy)phenyl)-6-methylpiperidine (3u), colorless oil. IR (film): 3310, 1611, 1510, 1241 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.31-7.26 (m, 2H), 6.89-6.82 (m, 2H), 6.11-5.97 (m, 1H), 5.40 (dd, $J = 17.4, 1.6$ Hz, 1H), 5.26 (dd, $J = 10.4, 1.2$ Hz, 1H), 4.51 (d, $J = 5.2$ Hz, 2H), 3.63-3.55 (m, 1H), 2.85-2.72 (m, 1H), 1.91-1.80 (m, 1H), 1.79-1.68 (m, 1H), 1.68-1.34 (m, 4H), 1.27-1.12 (m, 1H), 1.10 (d, $J = 6.0$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 157.8, 138.2, 133.6, 127.9, 117.7, 114.7, 69.0, 62.0, 53.4, 34.4, 34.0, 25.6, 23.3; HRMS (ESI) calcd. for $\text{C}_{15}\text{H}_{22}\text{NO}$ ($\text{M}+\text{H}$): 232.1696, Found: 232.1694.



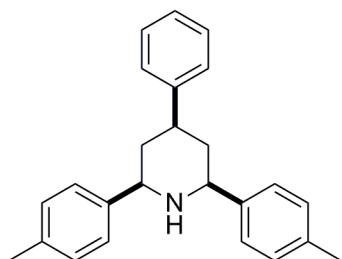
Cis-2-(4-methoxyphenyl)-6-ethylpiperidine (3v), colorless oil. IR (film): 3312, 1611, 1513, 1246 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.33-7.26 (m, 2H), 6.89-6.80 (m, 2H), 3.78 (s, 3H), 3.56 (dd, $J = 10.4, 2.0$ Hz, 1H), 2.60-2.49 (m, 1H), 1.91-1.80 (m, 1H), 1.80-1.60 (m, 3H), 1.58-1.33 (m, 4H), 1.18-1.07 (m, 1H), 0.91 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 158.8, 138.2,

128.0, 113.9, 62.1, 59.7, 55.4, 35.0, 31.9, 30.3, 25.5, 10.6; HRMS (ESI) calcd. for C₁₄H₂₂NO (M+H): 220.1696, Found: 220.1694.

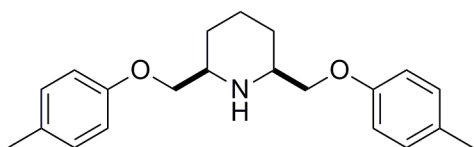


2-(4-Methoxyphenyl)piperidine (3w), colorless oil. ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.27 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 2H), 3.78 (s, 3H), 3.52 (dd, *J* = 10.4, 2.4 Hz, 1H), 3.20-3.14 (m, 1H), 2.83-2.73 (m, 1H), 1.95-1.69 (m, 3H), 1.69-1.59 (m, 1H), 1.59-1.40 (m, 3H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 158.7, 138.0, 127.8, 113.8, 61.9, 55.4, 48.1, 35.2, 26.1, 25.6.

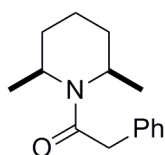
Kammler, R.; Polborn, K.; Wanner, K. Th. *Tetrahedron* **2003**, *59*, 3359.



Cis-4-phenyl-2,6-di-p-tolylpiperidine (3y), colorless oil. IR (film): 3307, 1601, 1513, 1450 cm⁻¹; ¹H NMR (400 MHz, CDCl₃, ppm): δ 7.36 (d, *J* = 7.6 Hz, 4H), 7.30-7.17 (m, 4H), 7.16-7.09 (m, 5H), 3.95 (d, *J* = 10.4 Hz, 2H), 3.00-2.89 (m, 1H), 2.31 (s, 6H), 2.03 (d, *J* = 11.6 Hz, 2H), 1.86 (brs, 1H), 1.80-1.65 (m, 2H); ¹³C NMR (100 MHz, CDCl₃, ppm): δ 146.2, 142.5, 136.8, 129.2, 128.6, 127.0, 126.8, 126.3, 62.0, 43.9, 42.4, 21.3; HRMS (ESI) calcd. for C₂₅H₂₈N (M+H): 342.2216, Found: 342.2214.

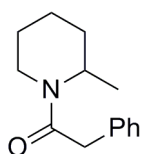


Cis-2,6-bis((p-tolyloxy)methyl)piperidine (3z), white solid, m.p. 115-117 °C. IR (film): 3332, 1614, 1513, 1241 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.06 (d, $J = 8.0$ Hz, 4H), 6.81 (d, $J = 8.0$ Hz, 4H), 3.94-3.85 (m, 2H), 3.84-3.75 (m, 2H), 3.11-3.01 (m, 2H), 2.70 (brs, 1H), 2.27 (s, 6H), 1.94-1.86 (m, 1H), 1.74-1.65 (m, 2H), 1.55-1.39 (m, 1H), 1.26-1.13 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 157.0, 130.2, 130.0, 114.7, 73.0, 55.6, 28.7, 24.0, 20.7; HRMS (ESI) calcd. for $\text{C}_{21}\text{H}_{28}\text{NO}_2$ (M+H): 326.2115, Found: 326.2111.



Cis-2,6-dimethylpiperidin-1-yl-2-phenylethanone (3A). Pale yellow solid. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.35-7.18 (m, 5H), 4.83 (br, s, 1H), 4.11 (br, s, 1H), 3.85-3.64 (m, 2H), 1.82-1.40 (m, 6H), 1.39-1.11 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 170.3, 136.0, 128.8, 127.7, 126.8, 48.2, 44.0, 41.5, 30.6, 30.1, 21.8, 20.7, 14.0.

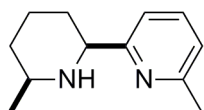
Chow, Y. L.; Colón, C. J.; Tam, J. N. S. *Can. J. Chem.* **1968**, *46*, 2821.



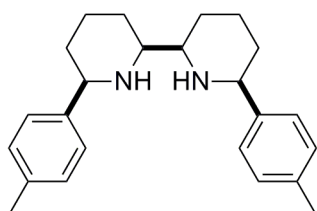
1-(2-Methylpiperidin-1-yl)-2-phenylethanone (3B) (The ratio of two rotational isomers = 1/1), pale yellow oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.35-7.19 (m, 5H), 4.96 (br, s, 0.5H), 4.65-4.40 (m, 0.5H), 4.15 (br, s, 0.5H), 3.72 (br, s, 2H), 3.69-3.49 (m, 0.5H), 3.11-2.85 (m, 0.5H), 2.76-2.50 (m,

0.5H), 1.55-1.03 (m, 9H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 169.5, 137.7, 128.8, 128.7, 126.8, 49.0, 44.0, 41.9, 41.6, 36.6, 30.7, 29.9, 26.2, 25.7, 18.8, 16.7, 15.6.

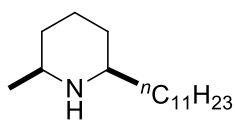
Chow, Y. L.; Colón, C. J.; Tam, J. N. S. *Can. J. Chem.* **1968**, *46*, 2821.



Cis-2-methyl-6-(6-methylpiperidin-2-yl)pyridine (3C), colorless oil. IR (film): 3263, 1591, 1455 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.57-7.48 (m, 1H), 7.16 (d, J = 7.6 Hz, 1H), 7.00 (d, J = 7.6 Hz, 1H), 3.82-3.71 (m, 1H), 2.90-2.79 (m, 1H), 2.53 (s, 3H), 2.02 (s, 1H), 1.99-1.85 (m, 2H), 1.71-1.62 (m, 1H), 1.62-1.36 (m, 2H), 1.28-1.07 (m, 1H), 1.14 (d, J = 6.0 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 162.9, 157.7, 137.0, 121.7, 117.4, 62.8, 52.9, 34.2, 32.3, 25.3, 24.7, 23.2; HRMS (ESI) calcd. for $\text{C}_{12}\text{H}_{19}\text{N}_2$ ($\text{M}+\text{H}$): 191.1543, Found: 191.1542.



Cis-6,6'-di(*p*-tolyl)-2,2'-bipiperidine (3D), white solid, m.p. 174-176 °C. IR (film): 3312, 1514, 1441 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3 , ppm): δ 7.27 (d, J = 8.0 Hz, 4H), 7.11 (d, J = 8.0 Hz, 4H), 3.63-3.54 (m, 2H), 2.69-2.60 (m, 2H), 2.32 (s, 6H), 1.97-1.85 (m, 2H), 1.77 (s, 2H), 1.73-1.65 (m, 2H), 1.65-1.53 (m, 2H), 1.52-1.30 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 143.5, 136.6, 129.1, 126.9, 62.6, 62.4, 35.5, 27.4, 25.5, 21.3; HRMS (ESI) calcd. for $\text{C}_{24}\text{H}_{33}\text{N}_2$ ($\text{M}+\text{H}$): 349.2638, Found: 349.2635.



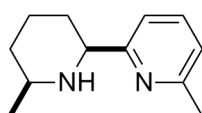
Isosolenopsin A, Colourless oil. ^1H NMR (400 MHz, CDCl_3 , ppm): δ 2.68-2.55 (m, 1H), 2.55-2.40 (m, 1H), 1.82-1.69 (m, 1H), 1.69-1.47 (m, 3H), 1.44-1.15 (m, 22H), 1.06 (d, $J = 6.4$ Hz, 3H), 0.95-1.02 (m, 1H), 0.88 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3 , ppm): δ 57.4, 52.7, 37.7, 34.7, 32.5, 32.1, 30.1, 29.9, 29.84, 29.82, 29.6, 26.2, 25.1, 23.3, 22.9, 14.3.

Pianaro, A.; Fox, E. G. P.; Bueno, O. C.; Marsaioli, A. J. *Tetrahedron: Asymmetry* **2012**, 23, 635.

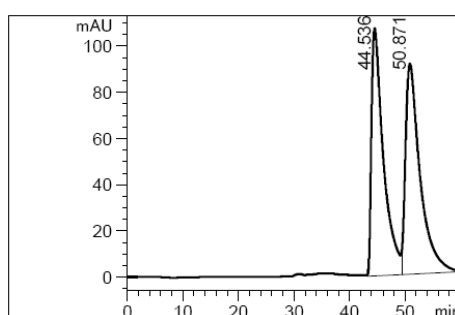
The chromatography for the determination of enantiomeric excess of optically active 3C

HPLC Conditions: Column: Chiralcel OB-H, Daicel Chemical Industries, Ltd., **Eluent:**

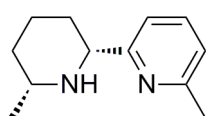
Hexanes/IPA (90/1); **Flow rate:** 0.1 mL/min; **Detection:** UV 254 nm



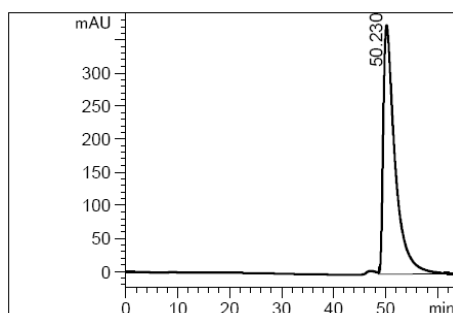
(±)-3C



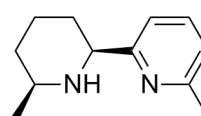
Peak #	RT [min]	Area %	Area
1	44.536	49.234	1.608e4
2	50.871	50.766	1.658e4



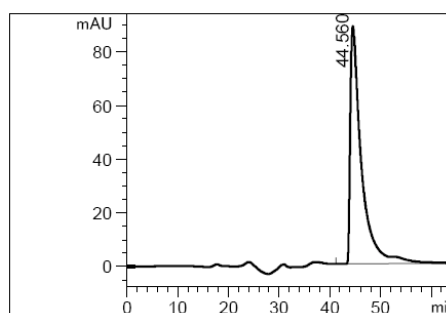
(R,R)-3C



Peak #	RT [min]	Area %	Area
1	50.230	100.000	5.955e4



(S,S)-3C



Peak #	RT [min]	Area %	Area
1	44.560	100.000	1.372e4

X-ray structure of piperidine 3c

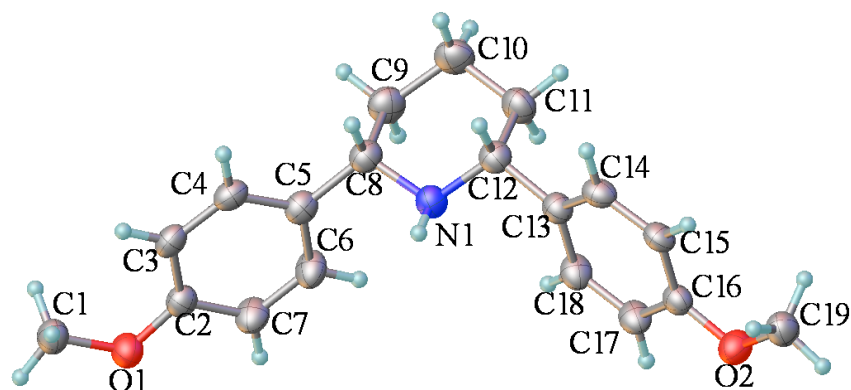


Table 1. Crystal data and structure refinement for piperidine 3c.

Identification code	sa2688
Empirical formula	C ₁₉ H ₂₃ N O ₂
Formula weight	297.38
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 7.5855(7) Å alpha = 90 deg. b = 8.6501(10) Å beta = 90 deg. c = 25.042(3) Å gamma = 90 deg.
Volume	1643.1(3) Å ³
Z, Calculated density	4, 1.202 Mg/m ³
Absorption coefficient	0.077 mm ⁻¹
F(000)	640
Crystal size	0.57 x 0.41 x 0.21 mm
Theta range for data collection	2.81 to 27.48 deg.
Limiting indices	-9<=h<=9, -11<=k<=11, -32<=l<=31
Reflections collected / unique	11391 / 2156 [R(int) = 0.0424]
Completeness to theta = 27.48	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.6357
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2156 / 0 / 202
Goodness-of-fit on F ²	1.195
Final R indices [I>2sigma(I)]	R1 = 0.0456, wR2 = 0.1242
R indices (all data)	R1 = 0.0475, wR2 = 0.1255
Absolute structure parameter	-10(10)
Largest diff. peak and hole	0.191 and -0.171 e.Å ⁻³

X-ray structure of compound 3D

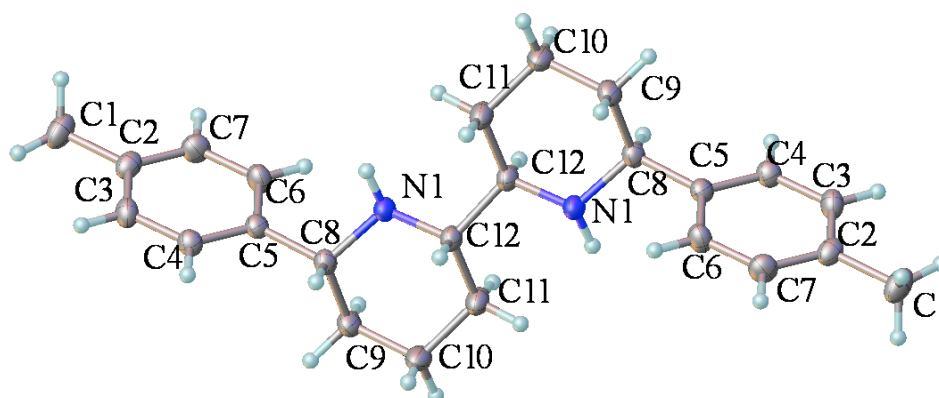


Table 2. Crystal data and structure refinement for compound **3D**

Identification code	sa2684
Empirical formula	C ₂₄ H ₃₂ N ₂
Formula weight	348.52
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 8.0673(13) Å alpha = 90 deg. b = 6.0026(7) Å beta = 95.849(9) deg. c = 20.713(3) Å gamma = 90 deg.
Volume	997.8(2) Å ³
Z, Calculated density	2, 1.160 Mg/m ³
Absorption coefficient	0.067 mm ⁻¹
F(000)	380
Crystal size	0.72 x 0.50 x 0.45 mm
Theta range for data collection	3.54 to 27.51 deg.
Limiting indices	-10 ≤ h ≤ 10, -7 ≤ k ≤ 7, -26 ≤ l ≤ 26
Reflections collected / unique	6635 / 2246 [R(int) = 0.0336]
Completeness to theta = 27.51	98.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.6110
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2246 / 0 / 119
Goodness-of-fit on F ²	1.133
Final R indices [I > 2sigma(I)]	R1 = 0.0530, wR2 = 0.1359
R indices (all data)	R1 = 0.0551, wR2 = 0.1379
Largest diff. peak and hole	0.334 and -0.185 e.Å ⁻³

X-ray structure of crystal 1

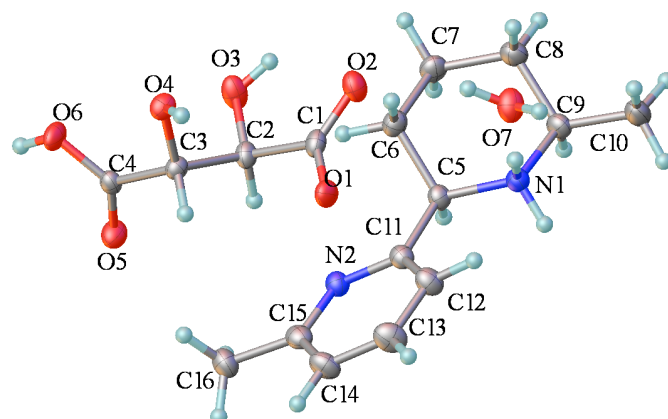
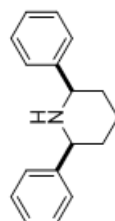
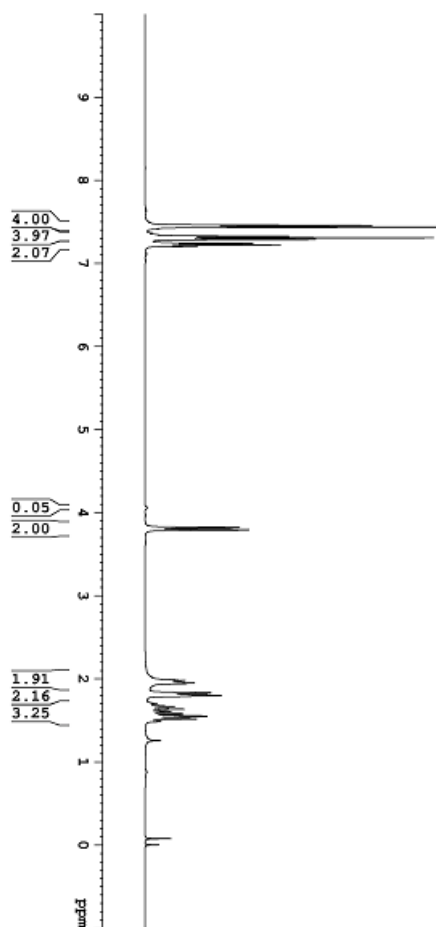


Table 3. Crystal data and structure refinement for crystal 1.

Identification code	sa2740a
Empirical formula	C ₁₆ H ₂₆ N ₂ O ₇
Formula weight	358.39
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P1
Unit cell dimensions	a = 7.451(3) Å alpha = 74.72(2) deg. b = 7.592(4) Å beta = 88.22(2) deg. c = 8.063(4) Å gamma = 83.52(2) deg.
Volume	437.2(4) Å ³
Z, Calculated density	1, 1.361 Mg/m ³
Absorption coefficient	0.107 mm ⁻¹
F(000)	192
Crystal size	0.50 x 0.28 x 0.27 mm
Theta range for data collection	2.80 to 27.49 deg.
Limiting indices	-9 ≤ h ≤ 9, -9 ≤ k ≤ 9, -10 ≤ l ≤ 10
Reflections collected / unique	7976 / 3911 [R(int) = 0.0429]
Completeness to theta = 27.49	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.0000 and 0.5407
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3911 / 3 / 229
Goodness-of-fit on F ²	1.062
Final R indices [I > 2sigma(I)]	R1 = 0.0370, wR2 = 0.0969
R indices (all data)	R1 = 0.0376, wR2 = 0.0976
Absolute structure parameter	0.4(7)
Largest diff. peak and hole	0.250 and -0.188 e.Å ⁻³

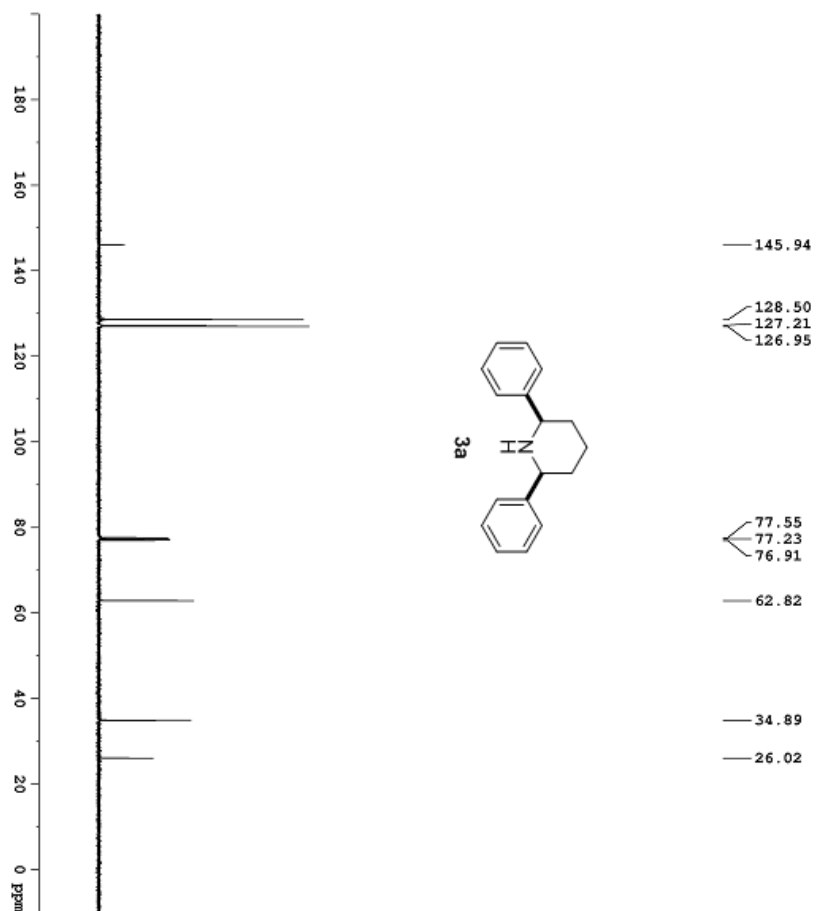


3a



```

NAME      1yb-130418-B
EXPNO     657
PROCNO    2
Date_     20130312
Time      6.26
INSTRUM    spect
PROBHD     5 mm PNUCL 13C
PULPROG    zg30
TD         32768
SOLVENT    CDCl3
NS         8
DS         0
SSMR       0
FIDRES     12019.230 Hz
AQ         0.366798 Hz
RG         1.363198 sec
RG         71.8
DE         41.600 usec
TE         295.4 K
D1         2.00000000 sec
TD0        1
===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300382 MHz
WDW        EM
SSB        0
ZS         0.50 Hz
GB         0
PC         1.00
  
```

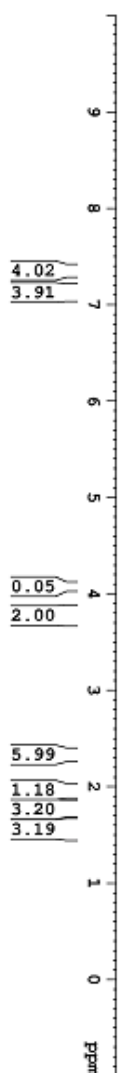
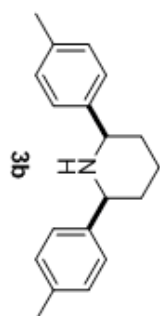


```

NAME      1p-130622-C
EXPNO     657
PROCNO    1
Date_     20130316
Time      6.28
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         43
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.648964 sec
RG         203
DPM        19.450 usec
DE         6.50 usec
TE         296.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127564 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

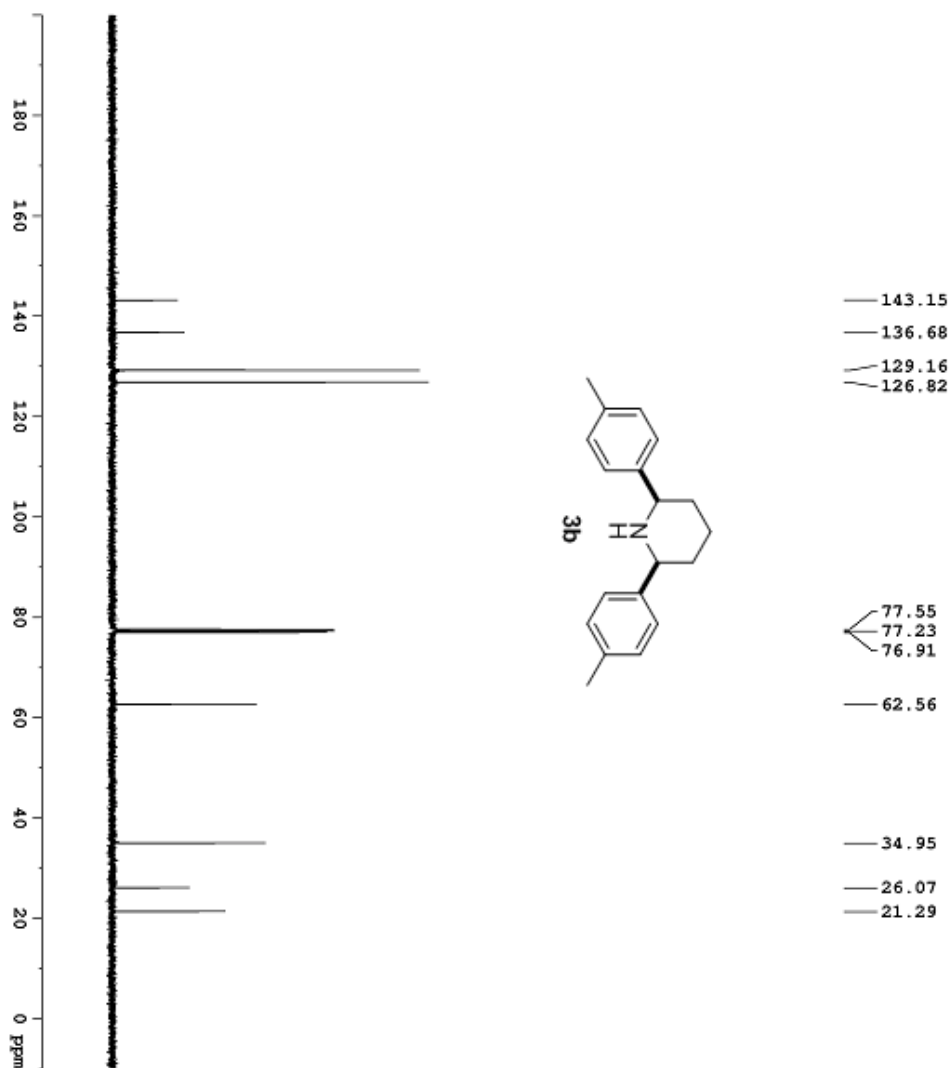
```



```

NAME      1yb-130418-H
EXPNO     665
PROCNO     3
Date_      20130328
Time       11.05
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD          32768
F2          32768
SOLVENT    CDCl3
NS          8
DS          0
SWH         12019.230 Hz
FIDRES     0.366798 Hz
AQ          1.3631988 sec
RG          203
RG          203
DW          41.600 usec
DE          6.50 usec
TE          295.4 K
D1          2.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300218 MHz
WCM        EX
SSB        0
GB          0
PC          1.00
  
```

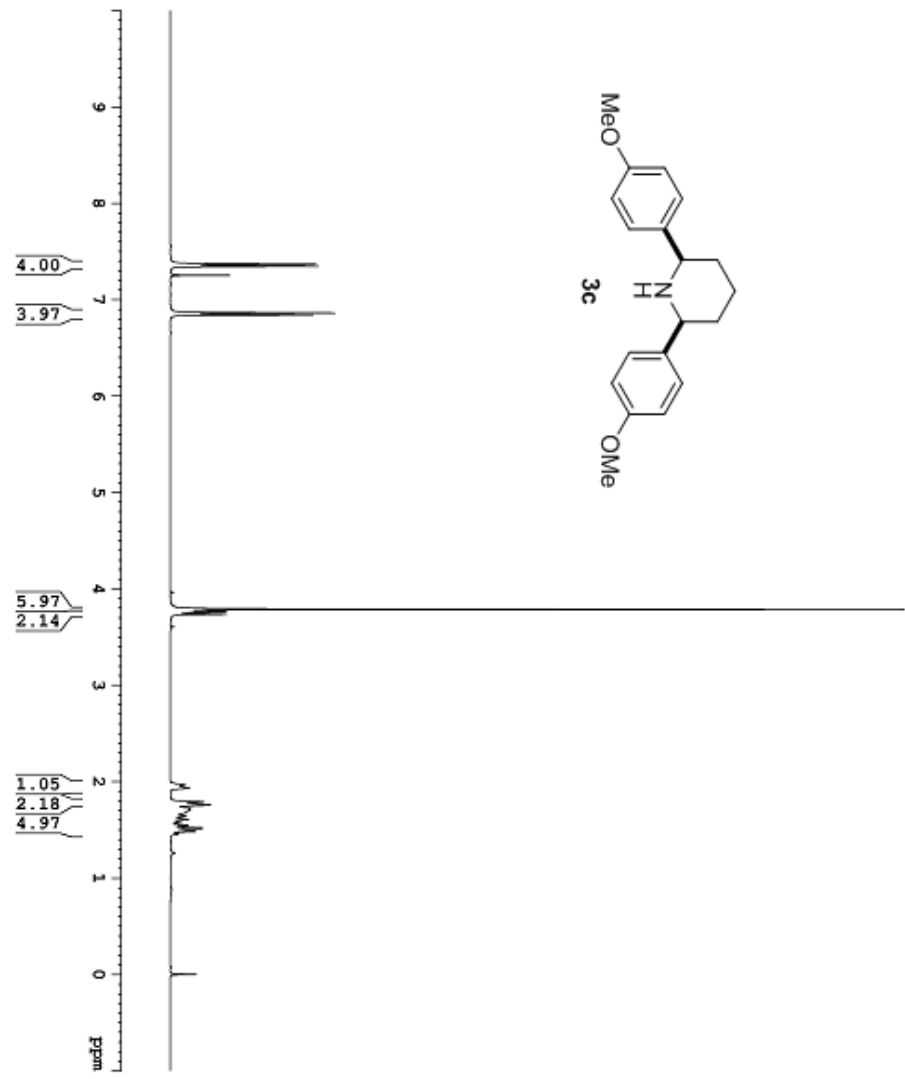
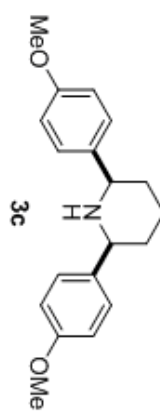


```

NAME      1yb-130622-C
EXPNO     665
PROCNO    1
Date_     20130317
Time      0.25
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         134
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.5 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127525 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         0
FC         1.40

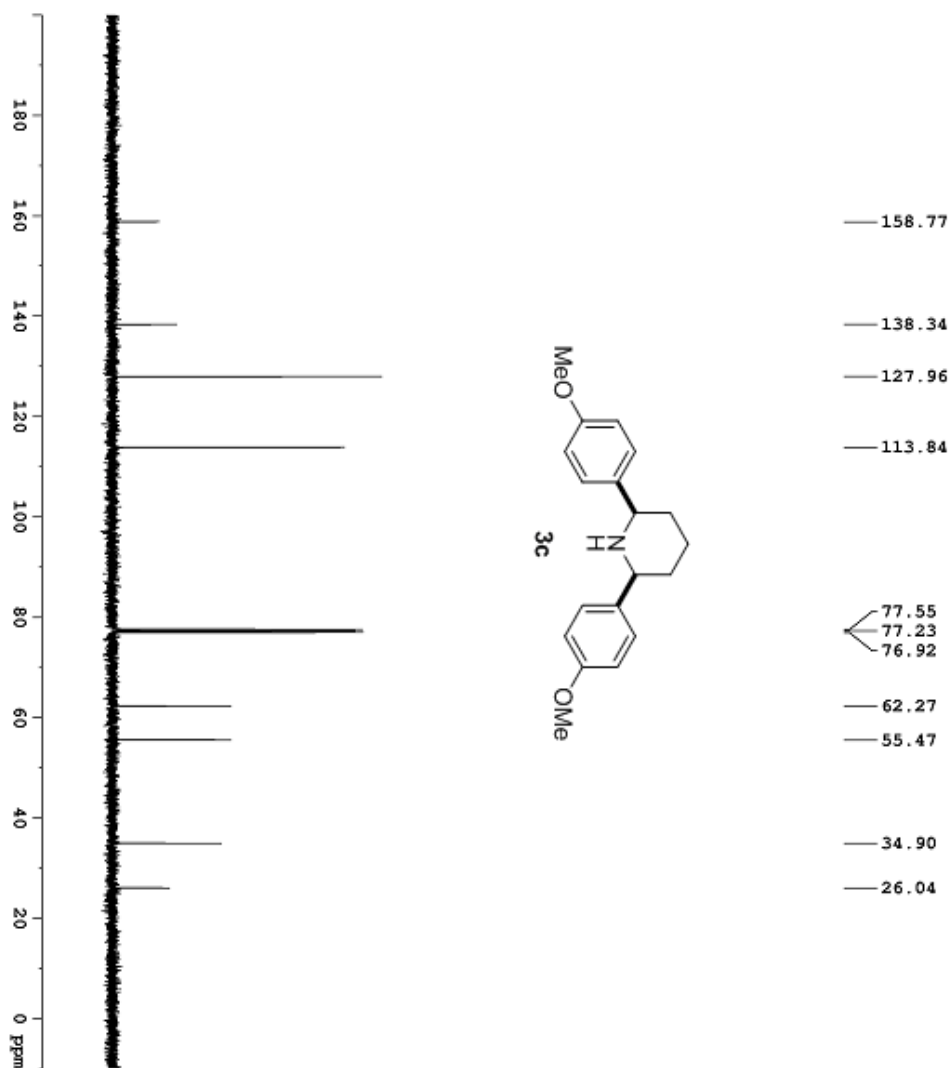
```



```

NAME      1yb-130622-B--2
EXPNO     3
PROCNO    3
Date_     20130607
Time      6.41
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         181
RG         181
DW         41.600 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300210 MHz
WCM        EX
SSB        0
GB         0
PC         1.00
  
```

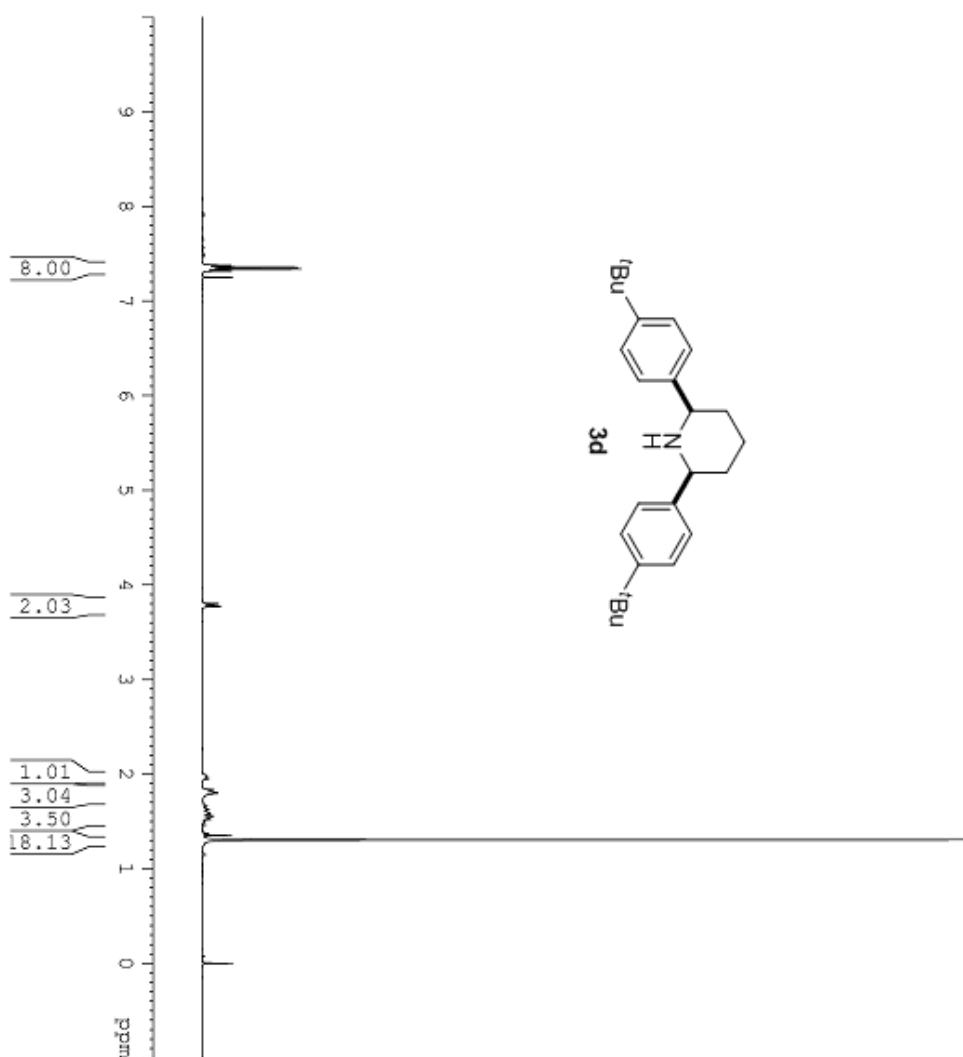
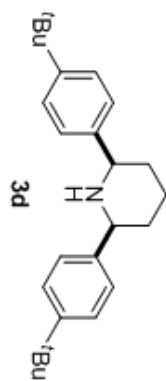


```

NAME      1yb-130622-C
EXPNO     681
PROCNO    2
Date_     20130607
Time      6.45
INSTRUM   spect
PROBHD    5 mm PABOL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         81
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127510 MHz
WDW       EM
SSB       0
GB        0
PC        1.00 Hz
EC        1.40

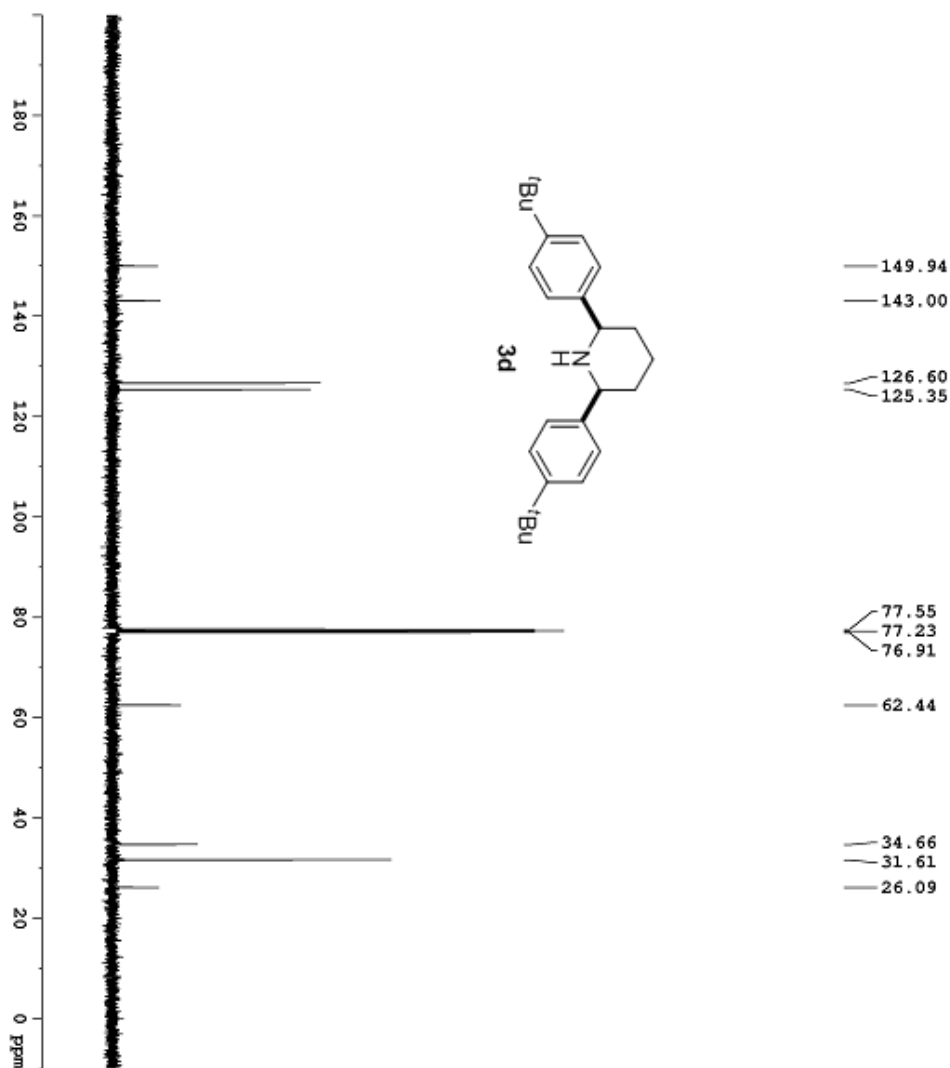
```



```

NAME      1y-b-130418-H
EXPNO     685
PROCNO    2
Date_     20130324
Time      12.35
INSTRUM    spect
PROBHD     5 mm PABUL 13C
PULPROG    zg30
TD          32768
SOLVENT    CDCl3
NS          8
DS          0
SMH         12019.230 Hz
FIDRES      0.366798 Hz
AQ          1.3631988 sec
RG          161
DE          41.600 usec
TE          294.8 K
D1          2.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1        1H
P1          12.60 usec
SI          65536
SF          400.1300211 MHz
WDW         EM
SSB         0
LB          0.50 Hz
GB          0
PC          1.00
  
```

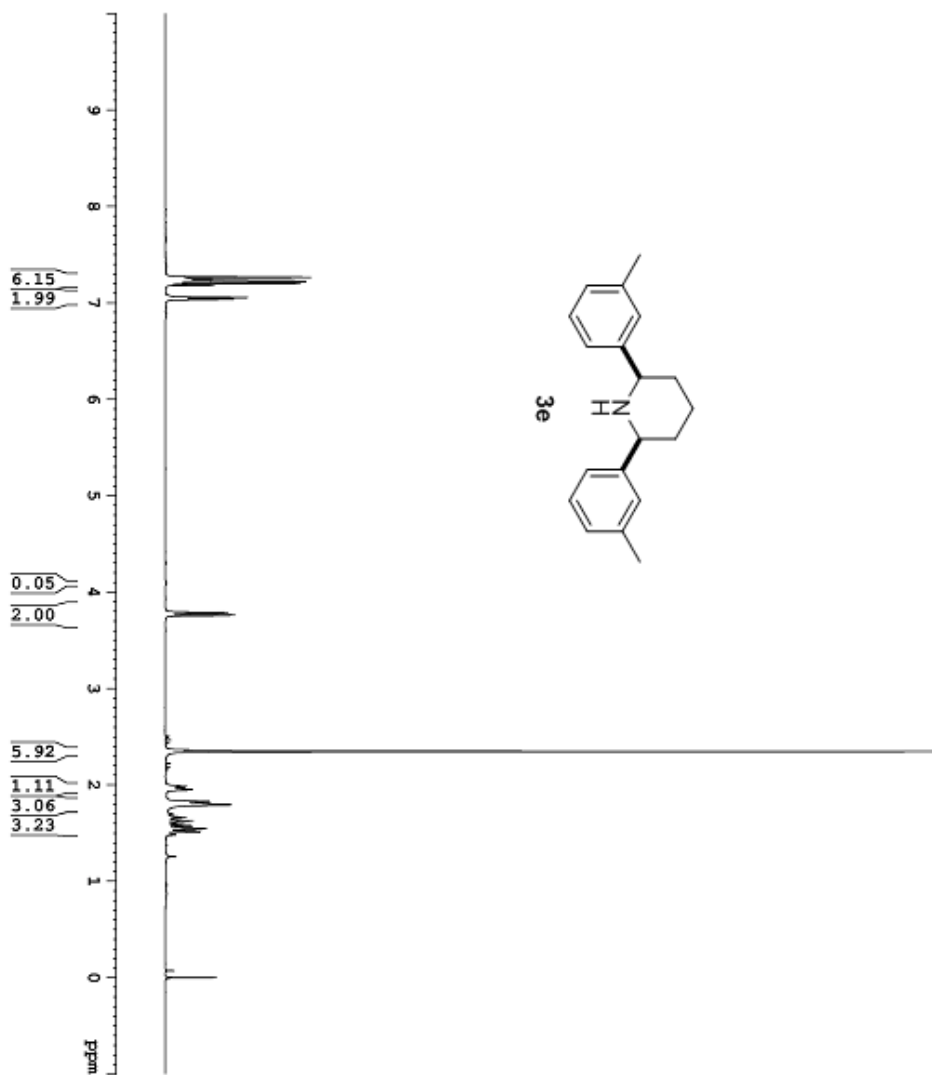
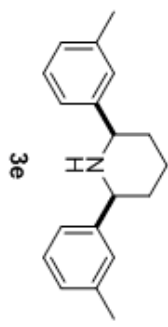


```

NAME      1yb-130622-C
EXPNO     685
PROCNO    1
Date_     20130324
Time      12.58
INSTRUM   spect
PROBHD    5 mm PABOL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         99
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         295.5 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127510 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

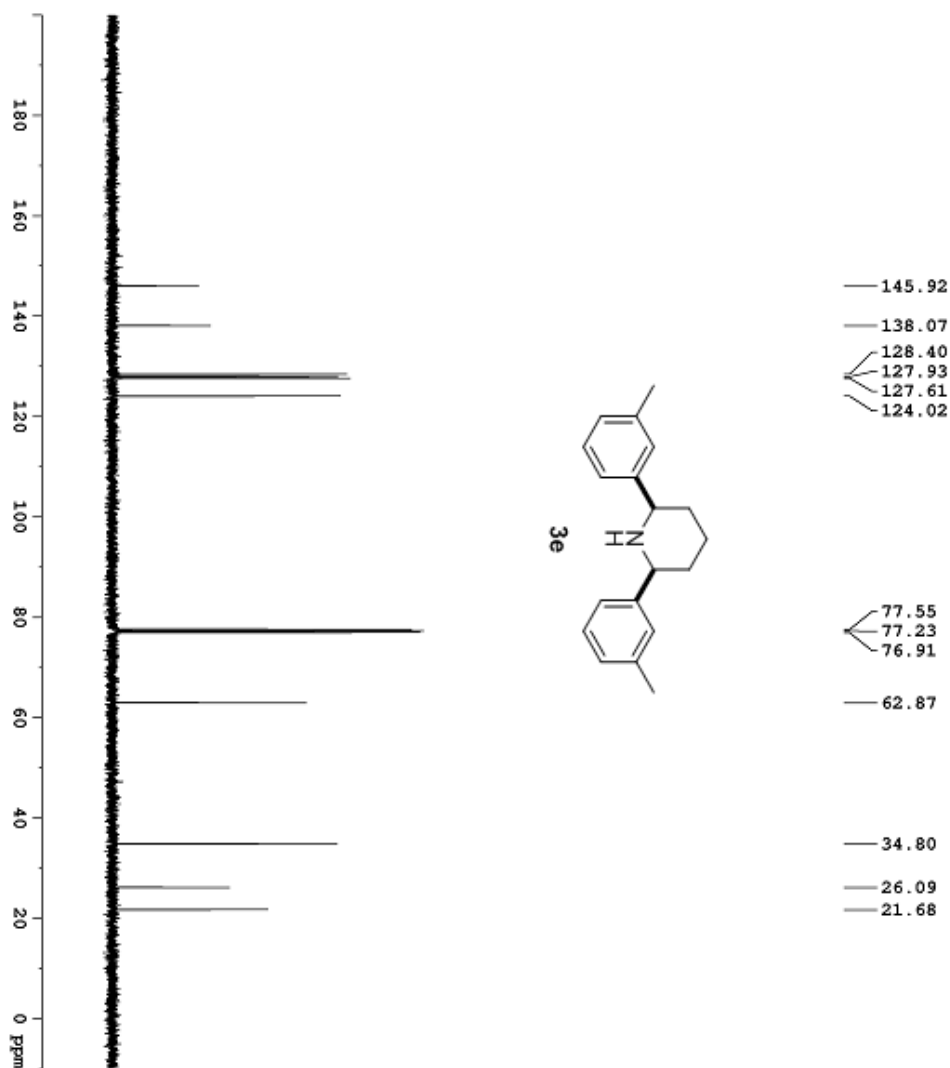
```



```

NAME      1yb-130418--H
EXPNO     2
PROCNO    2
Date_     20130323
Time      4.44
INSTRUM   spect
PROBHD    5 mm BBDPL 13C
PULPROG   zg30
TD         32768
F2         32768
SOLVENT   CDCl3
NS         9
DS         0
SWH        12019.230 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         114
RG         114
DW         41.600 usec
DE         6.50 usec
TE         295.5 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300272 MHz
WCMW       EM
SSB        0
GB         0
PC         1.00
  
```

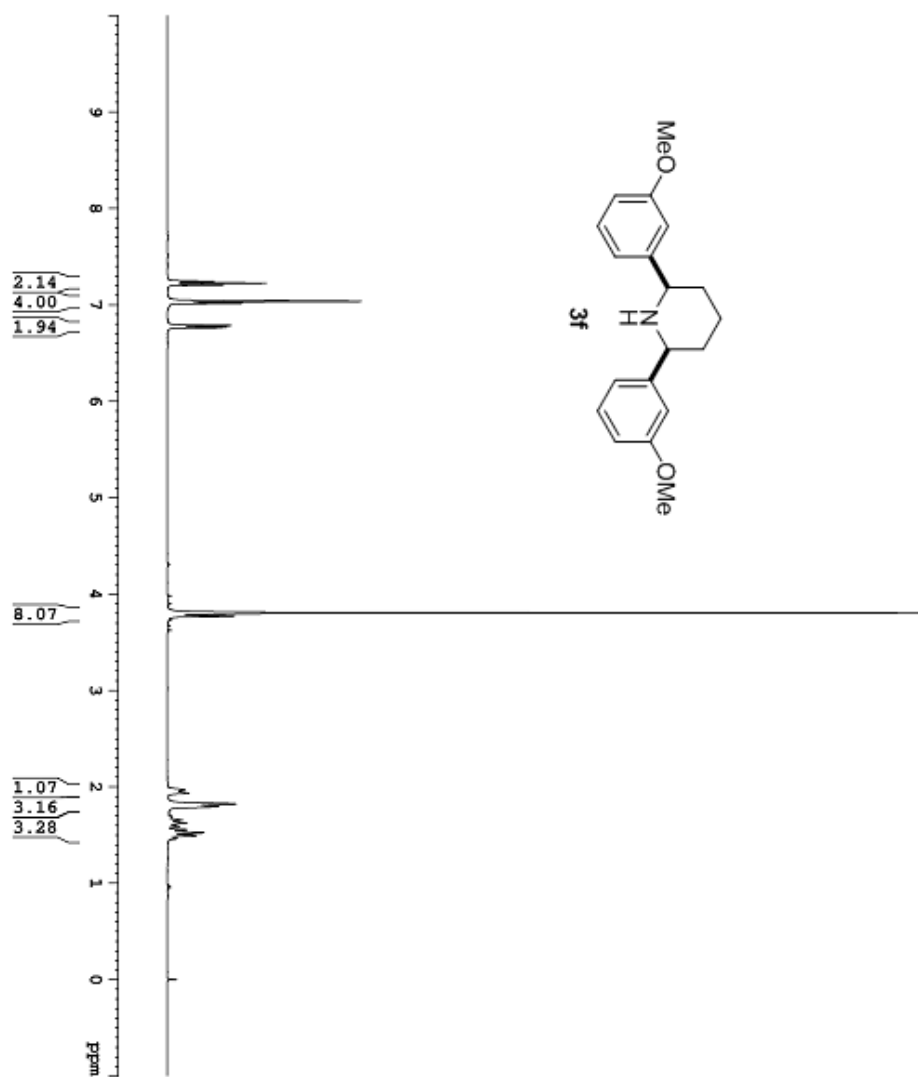
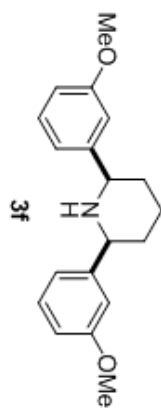


```

NAME      1yb-130622-C
EXPNO     679
PROCNO    1
Date_     20130323
Time      4.49
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         96
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127526 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

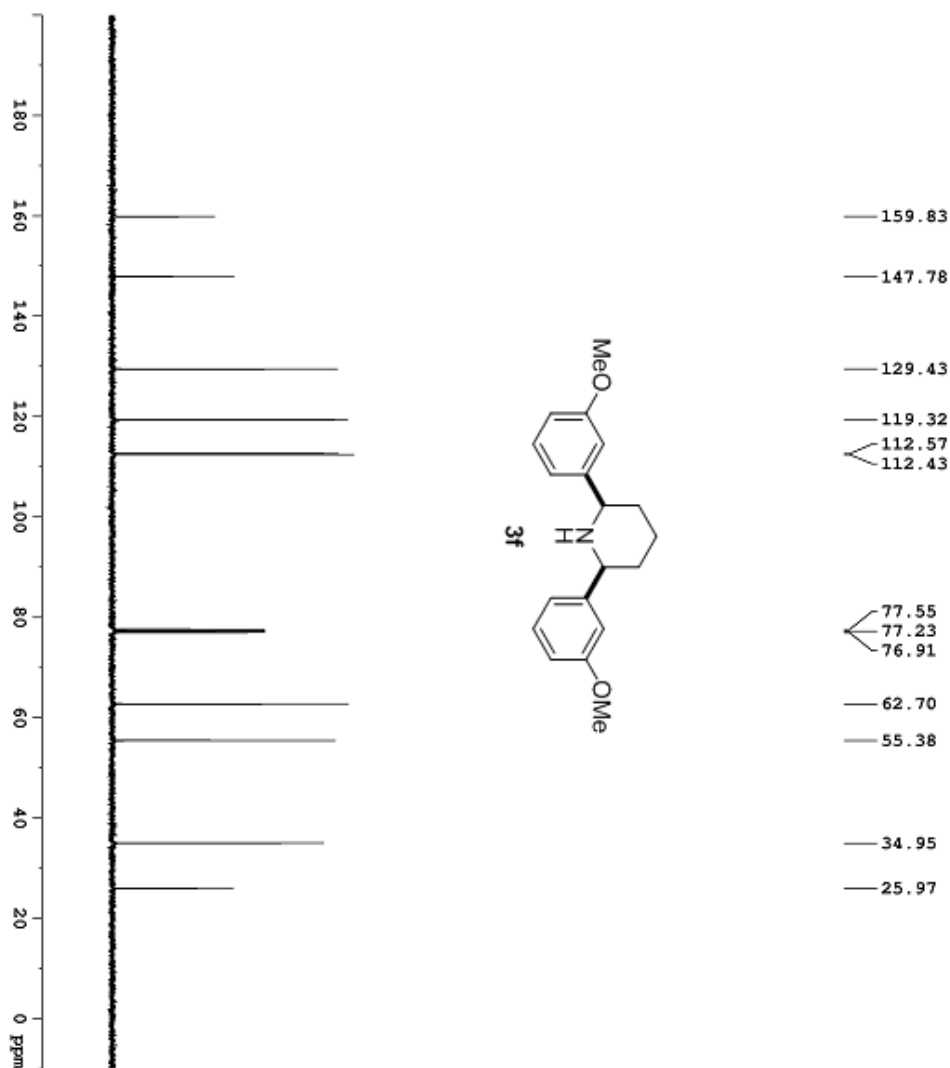
```



```

NAME      1yb-130418-H
EXPNO     2
PROCNO    2
Date_     20130331
Time      1.43
INSTRUM   spect
PROBHD    5 mm BBOUL-13C
PULPROG   zg30
TD         32768
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         71.8
DW         41.600 usec
DE         6.50 usec
TE         295.4 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300293 MHz
WCM        EX4
SSB        0
GB         0
PC         1.00
  
```

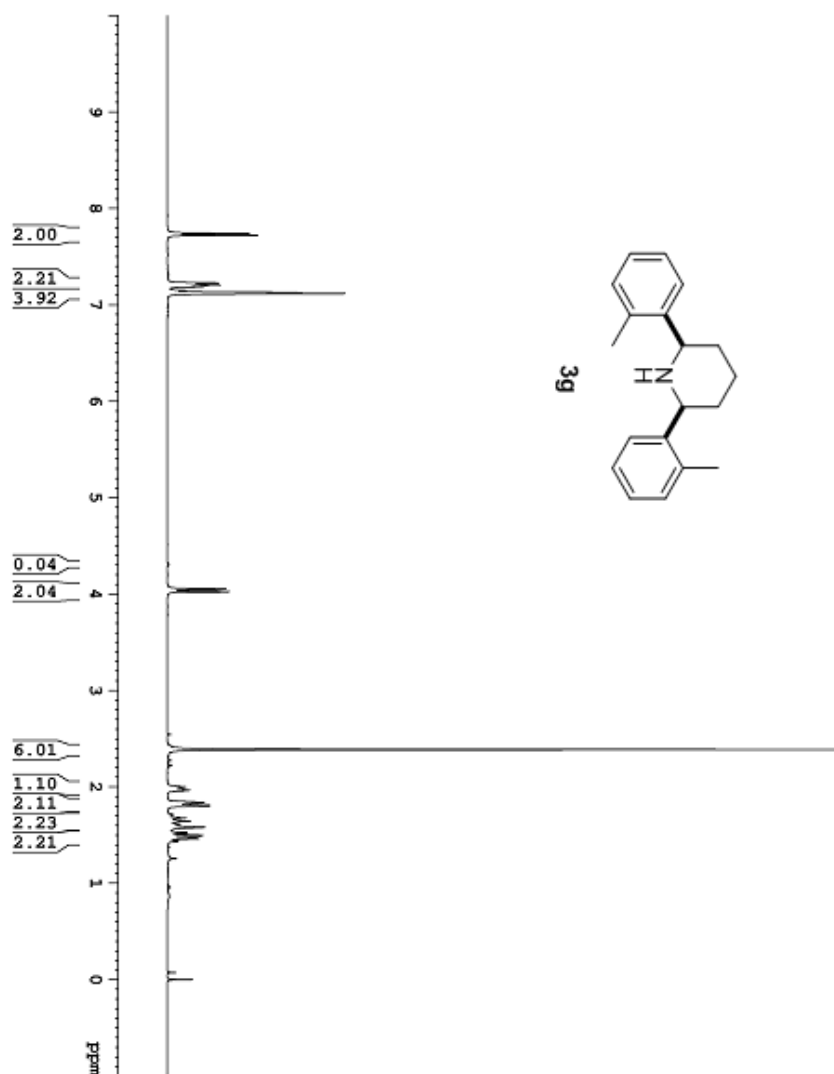
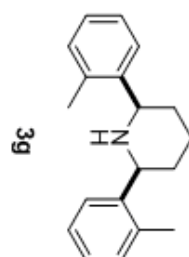


```

NAME      1yb-130622-C
EXPNO     694
PROCNO    1
Date_     20130331
Time      1.49
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         117
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127556 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

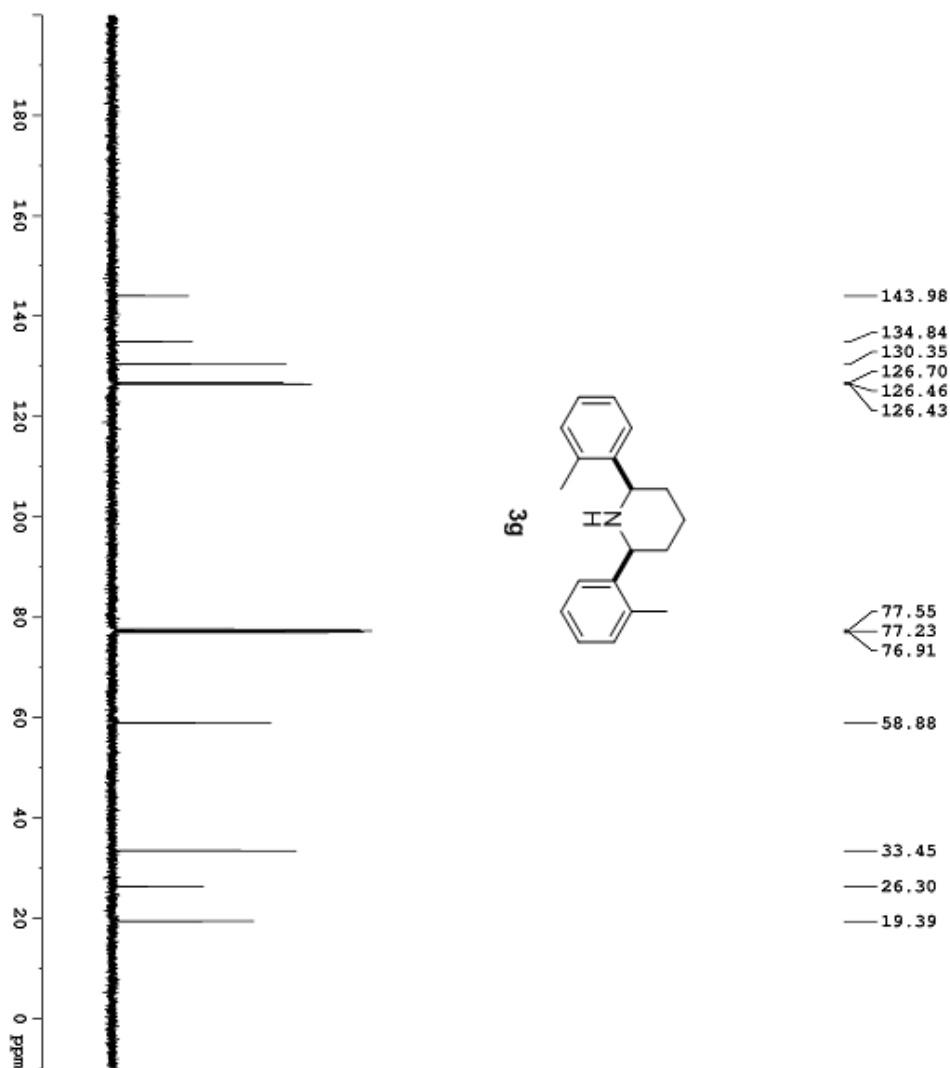
```



```

NAME      1yb-130418--H
EXPNO     684
PROCNO    2
Date_     20130324
Time      12.45
INSTRUM   spect
PROBHD    5 mm BBDPL 13C
PULPROG   zg30
TD         32768
FIDRES    0.36798 Hz
AQ         1.3631988 sec
RG         114
DW         41.600 usec
DE         6.50 usec
TE         294.7 K
D1         2.00000000 sec
TD0        1

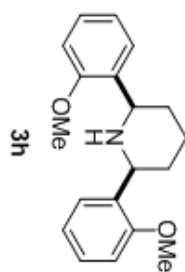
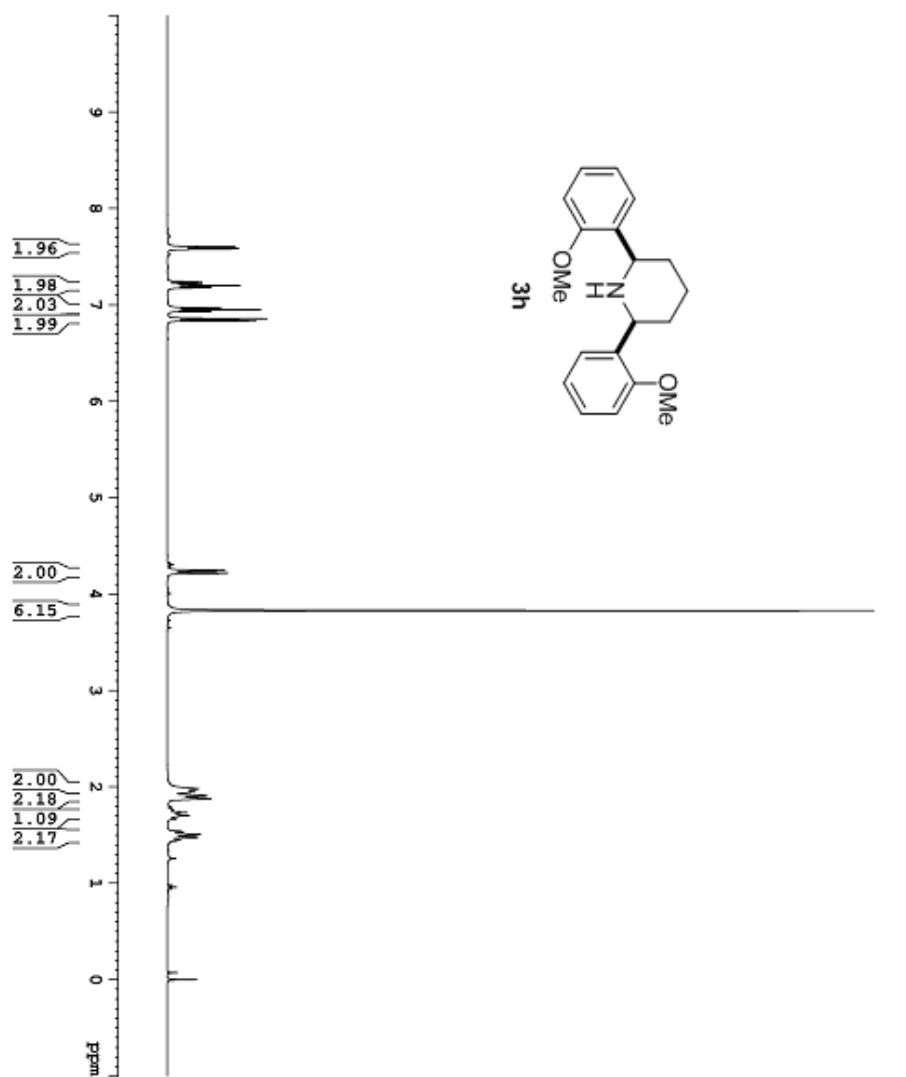
===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300305 MHz
WCM        EX4
SSB        0
GB         0
PC         1.00
  
```



```

NAME      lyb-130622-C
EXPNO     684
PROCNO    1
Date_     20130324
Time      12.48
INSTRUM    spect
PROBHD     5 mm PABD1 13C
PULPROG    zgpg30
TD          32768
SOLVENT    CDCl3
NS          52
DS          0
SWH         25252.525 Hz
FIDRES     0.770646 Hz
AQ          0.6488564 sec
RG          203
DM          19.800 usec
DE          6.50 usec
TE          295.4 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1

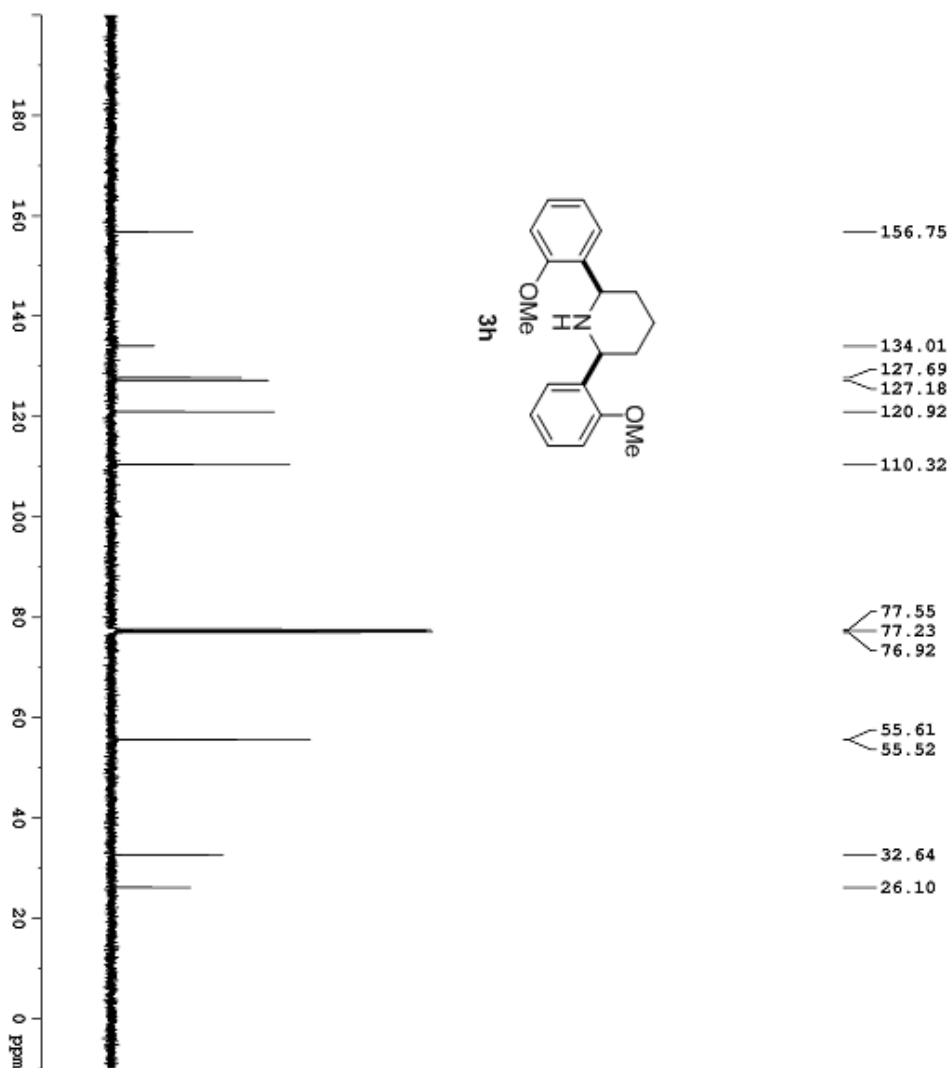
===== CHANNEL f1 =====
NUC1       13C
P1          9.40 usec
SI          32768
SF         100.6127541 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```



```

NAME      1yb-130418--H
EXPNO     2
PROCNO    2
Date_     20130327
Time      9.26
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
FIDRES     0.36798 Hz
AQ         1.3631988 sec
RG         114
DW         41.600 usec
DE         6.50 usec
TE         295.3 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300250 MHz
WCMW       EX
SSB        0
GB         0
PC         1.00
  
```

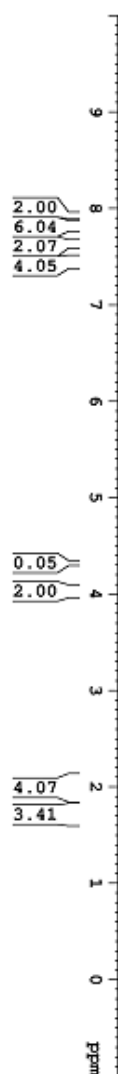
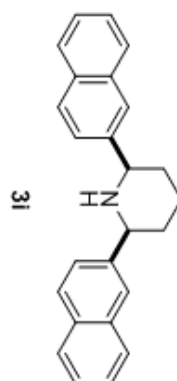


```

NAME      lyb-130622-C
EXPNO     608
PROCNO    1
Date_     20130327
Time      9.30
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         99
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1         9.40 usec
SI         32768
SF        100.6127526 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

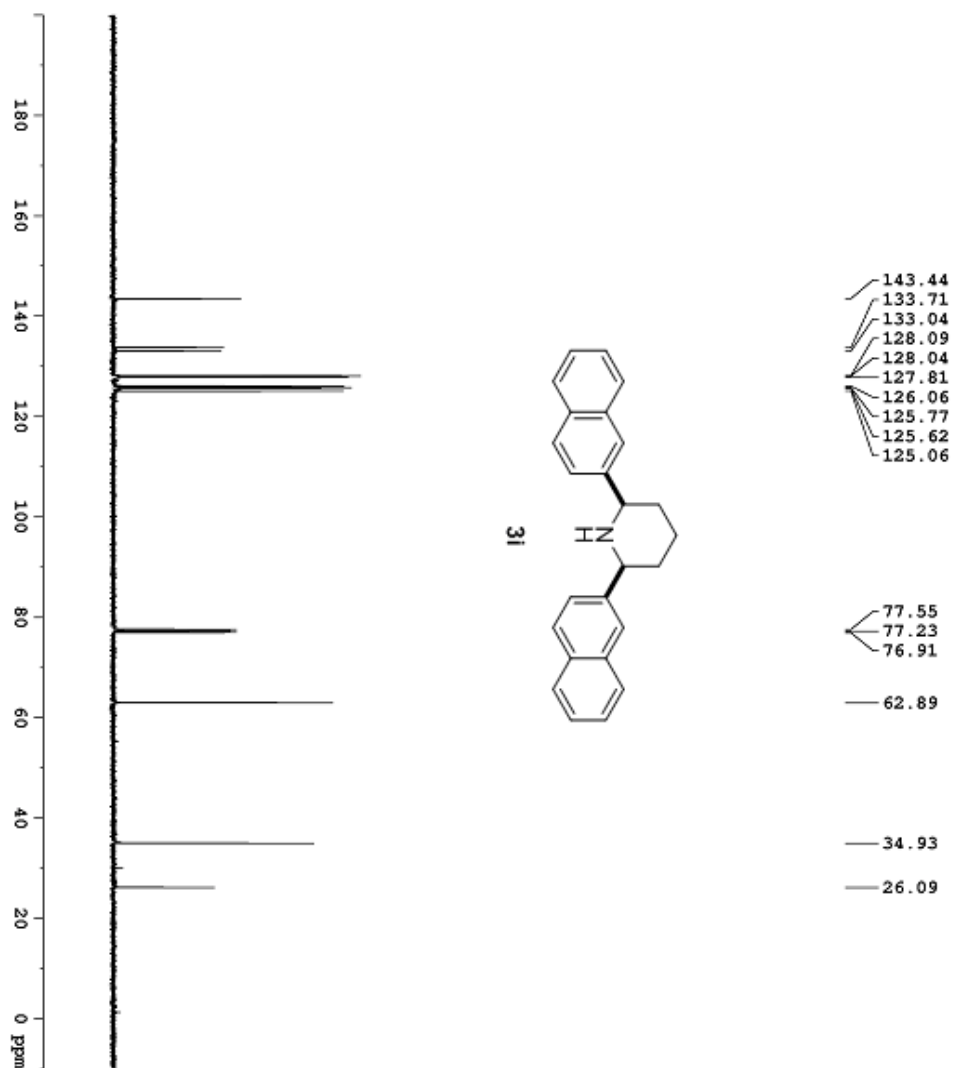
```



```

NAME      1yb-130418--H
EXPNO     3
PROCNO    3
Date_     20130328
Time      11.08
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
F2         32768
SOLVENT    CDCl3
NS         8
DS         0
SWH         12019.230 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         181
RG         181
DW         41.600 usec
DE         6.50 usec
TE         295.4 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300253 MHz
WCM        EX
SSB        0
GB         0
PC         1.00
  
```

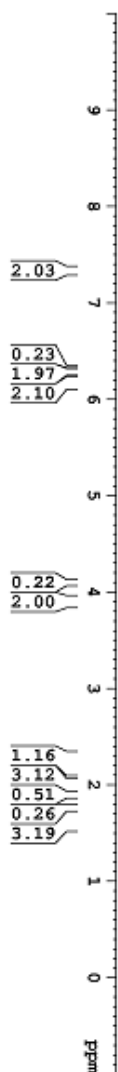
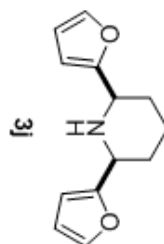


```

NAME      1yb-130622-C
EXPNO     689
PROCNO    1
Date_     20130327
Time      9.36
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         100
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ          0.6488564 sec
RG          203
DM         19.800 usec
DE          6.50 usec
TE         296.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1         9.40 usec
SI        32768
SF       100.6127621 MHz
WDW        EM
SSB         0
GB         0
PC         1.00 Hz
EC         1.40

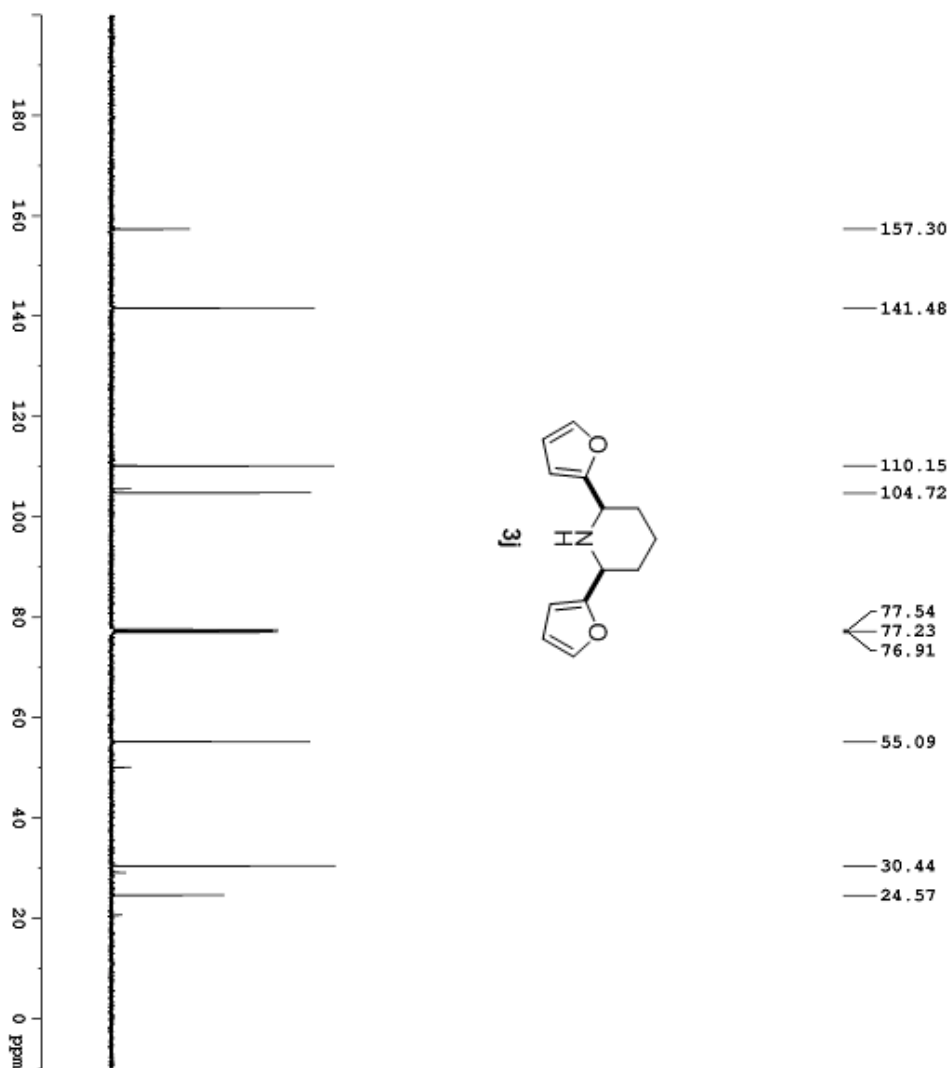
```



```

NAME      1yb-130418-H
EXPNO     2
PROCNO    2
Date_     20130402
Time      2.22
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD          32768
F2          32768
SOLVENT    CDCl3
NS          8
DS          0
SWH         12019.230 Hz
FIDRES     0.366798 Hz
AQ          1.3631988 sec
RG          203
RG          203
DW          41.600 usec
DE          6.50 usec
TE          295.6 K
D1          2.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300184 MHz
WCMW       EX
SSB        0
GB         0
PC         1.00
  
```

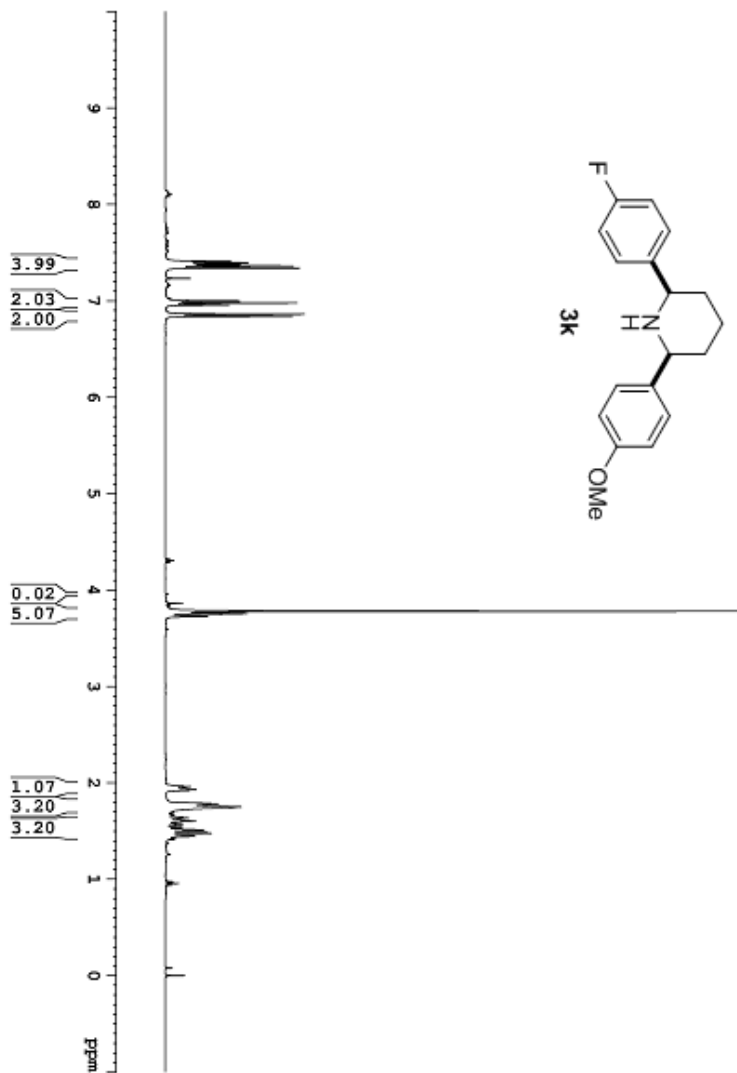
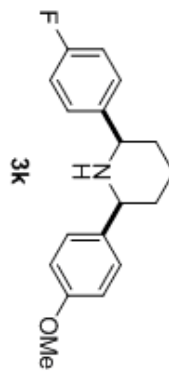


```

NAME      lyb-130622-C
EXPNO     698
PROCNO    1
Date_     20130331
Time      12.15
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         123
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.3 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127548 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

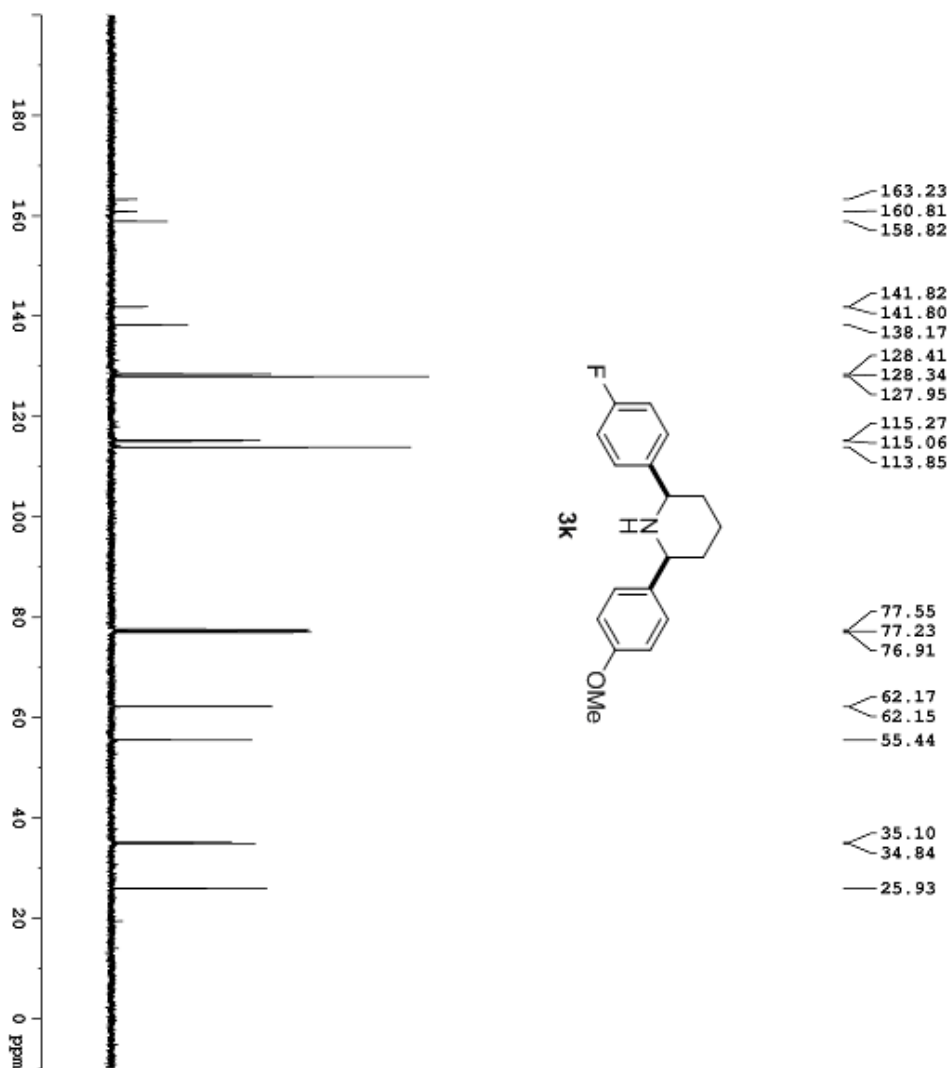
```



```

NAME      1yb-130418-H
EXPNO     2
PROCNO    2
Date_     20130330
Time      13.04
INSTRUM    spect
PROBHD     5 mm BBOUL-13C
PULPROG    zg30
TD         32768
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG          71.8
DW         41.600 usec
DE         6.50 usec
TE         295.3 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300285 MHz
WCM        EX4
SSB        0
GB         0
PC         1.00
  
```

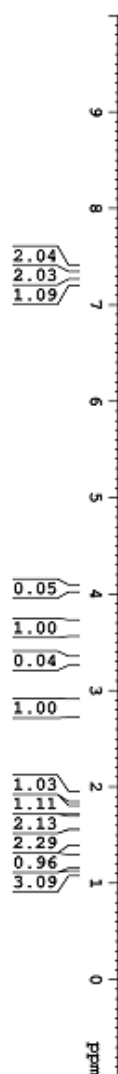
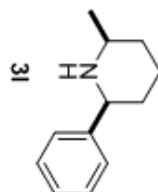


```

NAME      1yb-130622-C
EXPNO     695
PROCNO    1
Date_     20130330
Time      13.08
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         100
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127544 MHz
WDW       EM
SSB       0
GB        0
PC        1.00 Hz
EC        0
FC        1.40

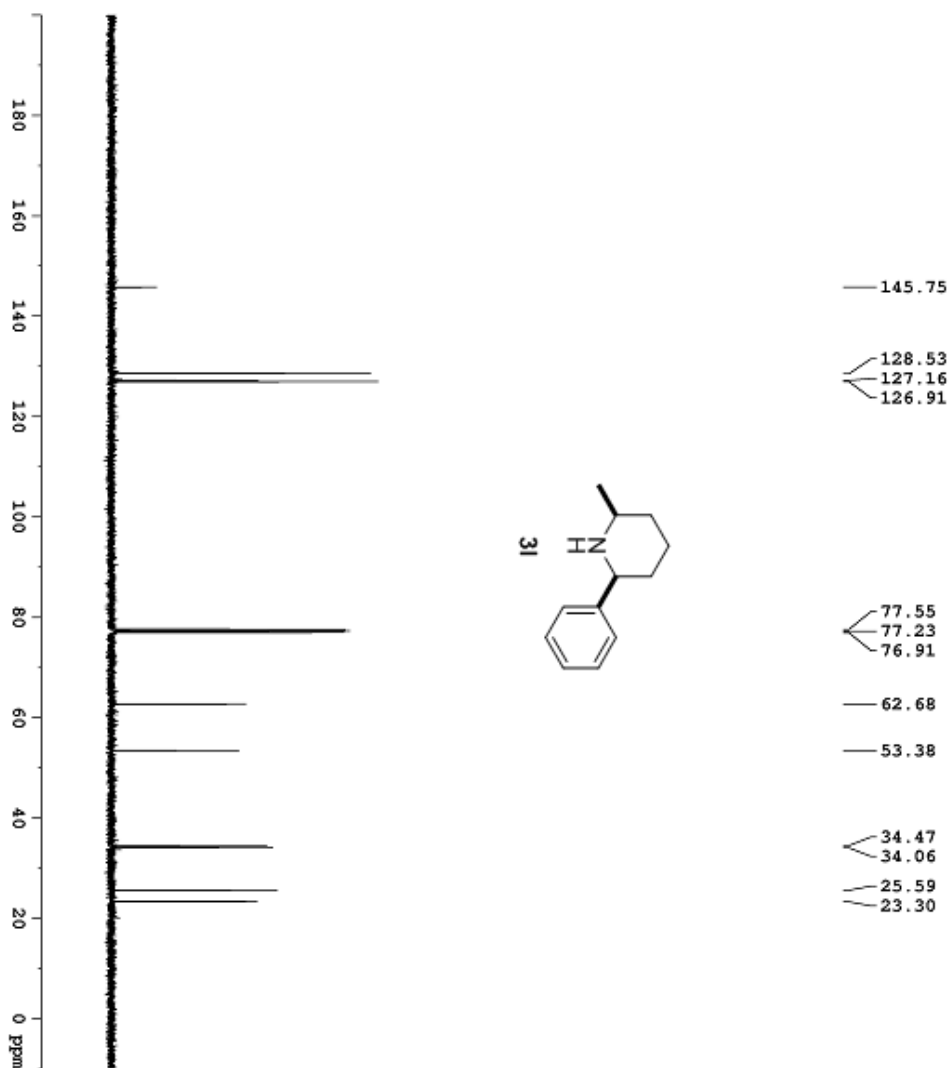
```



```

NAME      1y-130418-H
EXPNO     2
PROCNO    2
Date_     20130404
Time      11.07
INSTRUM    spect
PROBHD     5 mm BBOUL-13C
PULPROG    zg30
TD          32768
FIDRES     0.36798 Hz
AQ          1.363198 sec
RG          114
DW          41.600 usec
DE          6.50 usec
TE          295.7 K
D1          2.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.130023 MHz
WCM        EX4
SSB        0
GB          0
PC          1.00
  
```

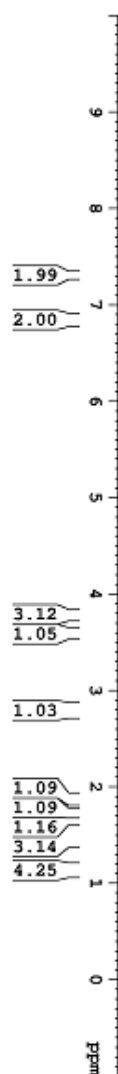
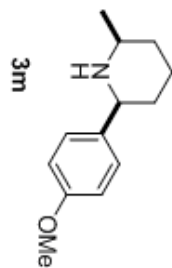


```

NAME      lyb-130622-C
EXPNO     1
PROCNO    1
Date_     20130404
Time      11.12
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         100
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.4 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127533 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

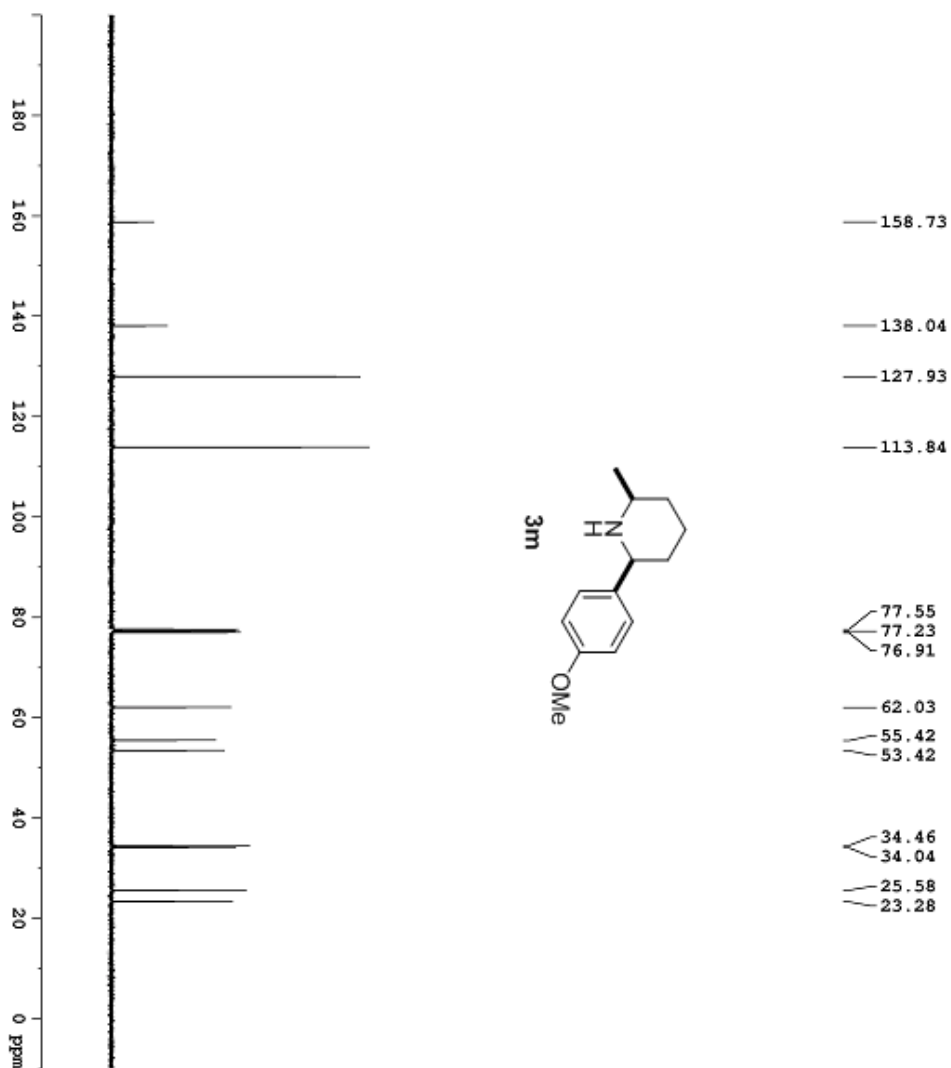
```



```

NAME      1yb-130418--H
EXPNO     703
PROCNO    2
Date_     20130403
Time      6.52
INSTRUM   spect
PROBHD    5 mm BBDPL 13C
PULPROG   zg30
TD         32768
F2         32768
SOLVENT   CDCl3
NS         8
DS         0
SWH        12019.230 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         71.8
RG         71.8
DW         41.600 usec
DE         6.50 usec
TE         295.5 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300204 MHz
WCM        EX4
SSB        0
GB         0
PC         1.00
  
```

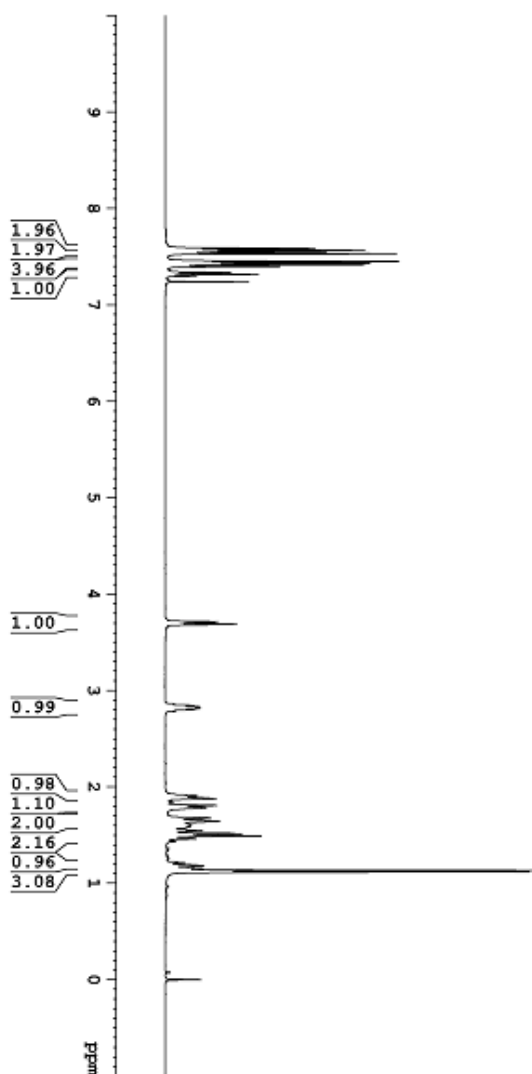
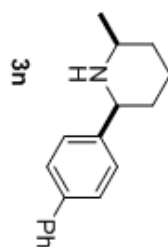


```

NAME      lyb-130622-C
EXPNO     703
PROCNO    1
Date_     20130403
Time      6.58
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         125
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ          0.6488564 sec
RG          203
DM         19.800 usec
DE         6.50 usec
TE         296.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127345 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

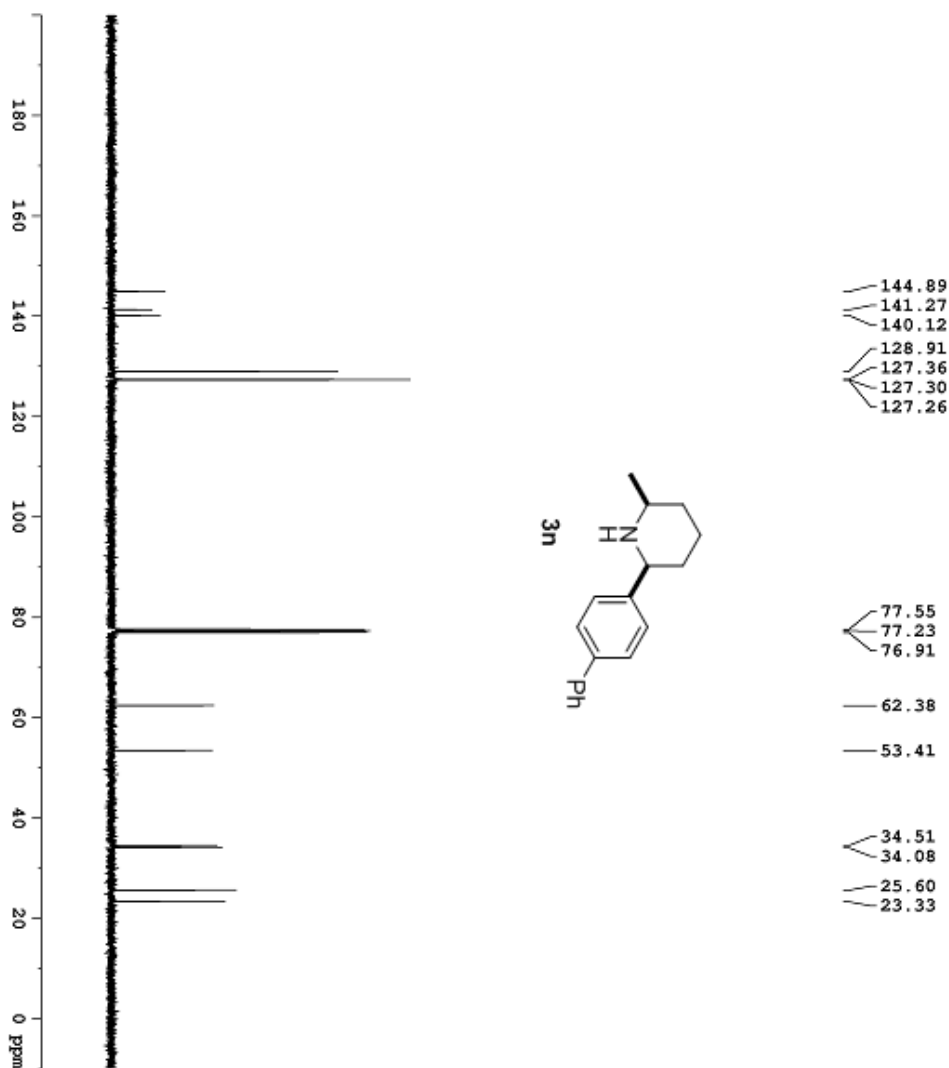
```



```

NAME      1yb-130622-B--2
EXPNO     12
PROCNO    12
Date_     20130524
Time      8.14
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD          32768
F2          32768
SOLVENT    CDCl3
NS          8
DS          0
SWH         12019.230 Hz
FIDRES      0.366798 Hz
AQ          1.3631988 sec
RG          203
RG          203
DW          41.600 usec
DE          6.50 usec
TE          300.0 K
D1          2.00000000 sec
TD0         1

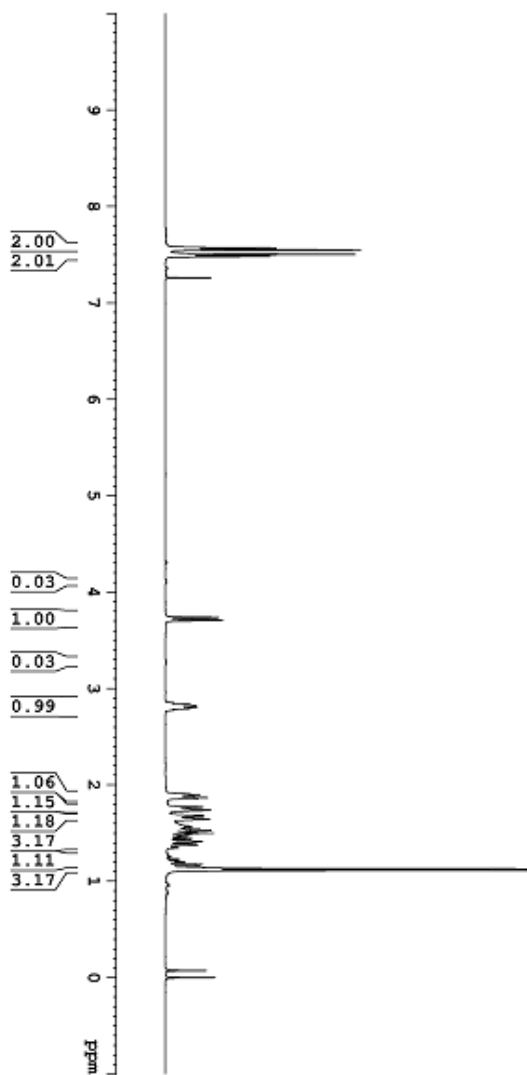
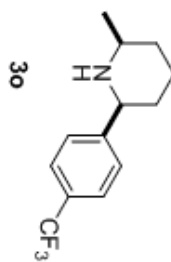
===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300250 MHz
WCM        EX4
SSB        0
GB          0
PC          1.00
  
```



```

NAME lyb-130622-C
EXPNO 777
PROCNO 1
Date_ 20130524
Time 8.22
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 413
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 0.6488564 sec
RG 203
DM 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

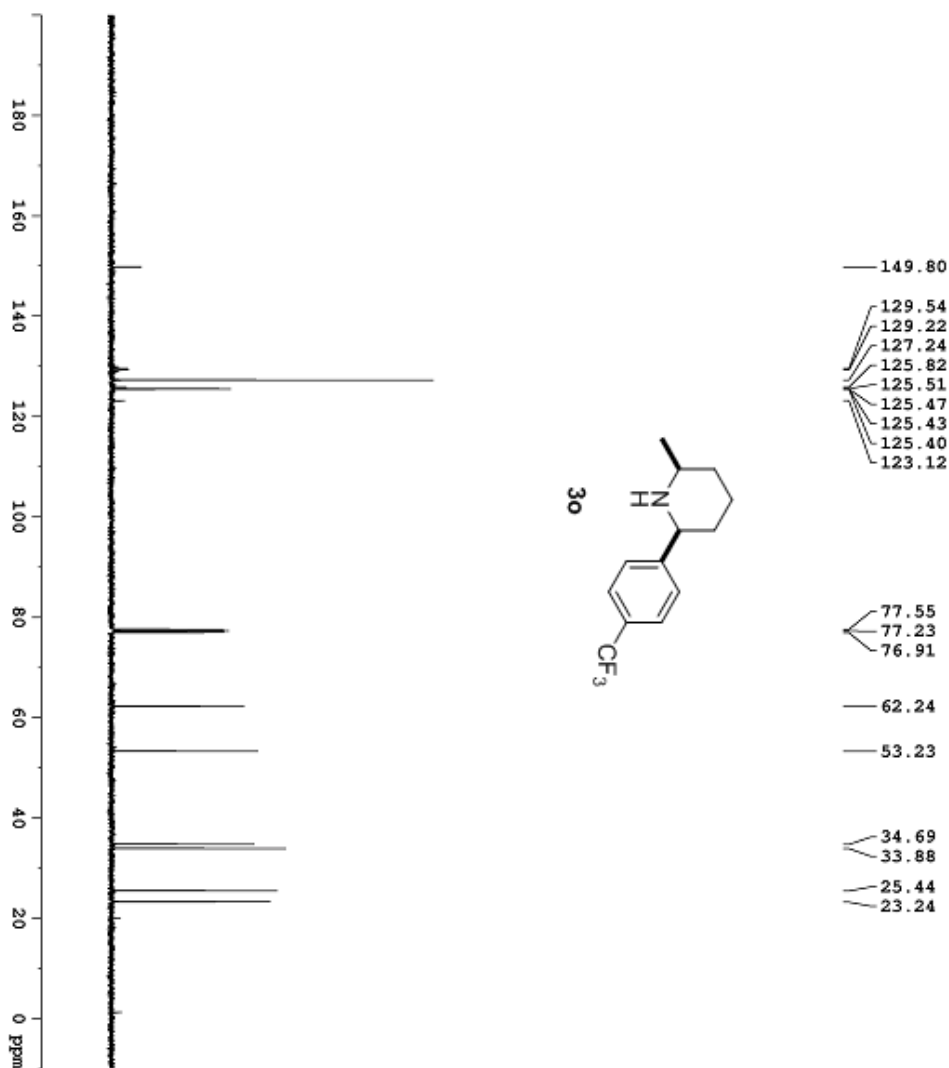
===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
SI 32768
SF 100.6127526 MHz
WDW EM
SSB 0
GB 0
PC 1.40
  
```



```

NAME      1yb-130418-H
EXPNO     725
PROCNO    2
Date_     20130416
Time      22.25
INSTRUM   spect
PROBHD    5 mm BBDPL 13C
PULPROG   zg30
TD         32768
FIDRES    0.36798 Hz
AQ         1.3631988 sec
RG         128
DW         41.600 usec
DE         6.50 usec
TE         295.5 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300178 MHz
WCM        EX4
SSB        0
GB         0
PC         1.00
  
```

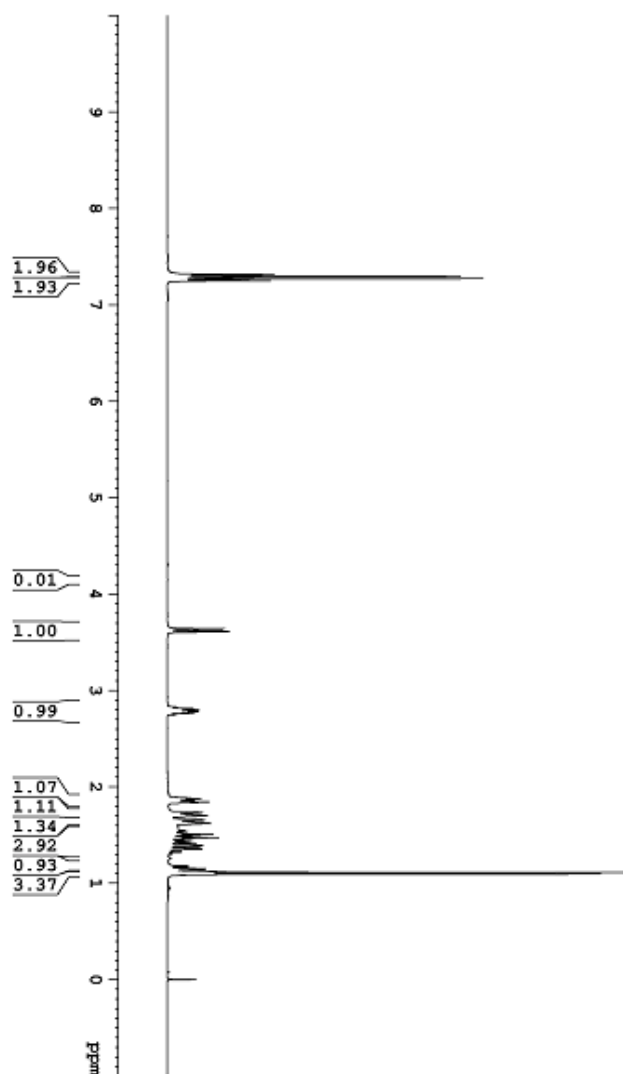
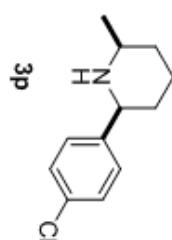


```

NAME      1yb-130622-C
EXPNO     725
PROCNO    2
Date_     20130417
Time      7.30
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         94
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127510 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         0
FC         1.40

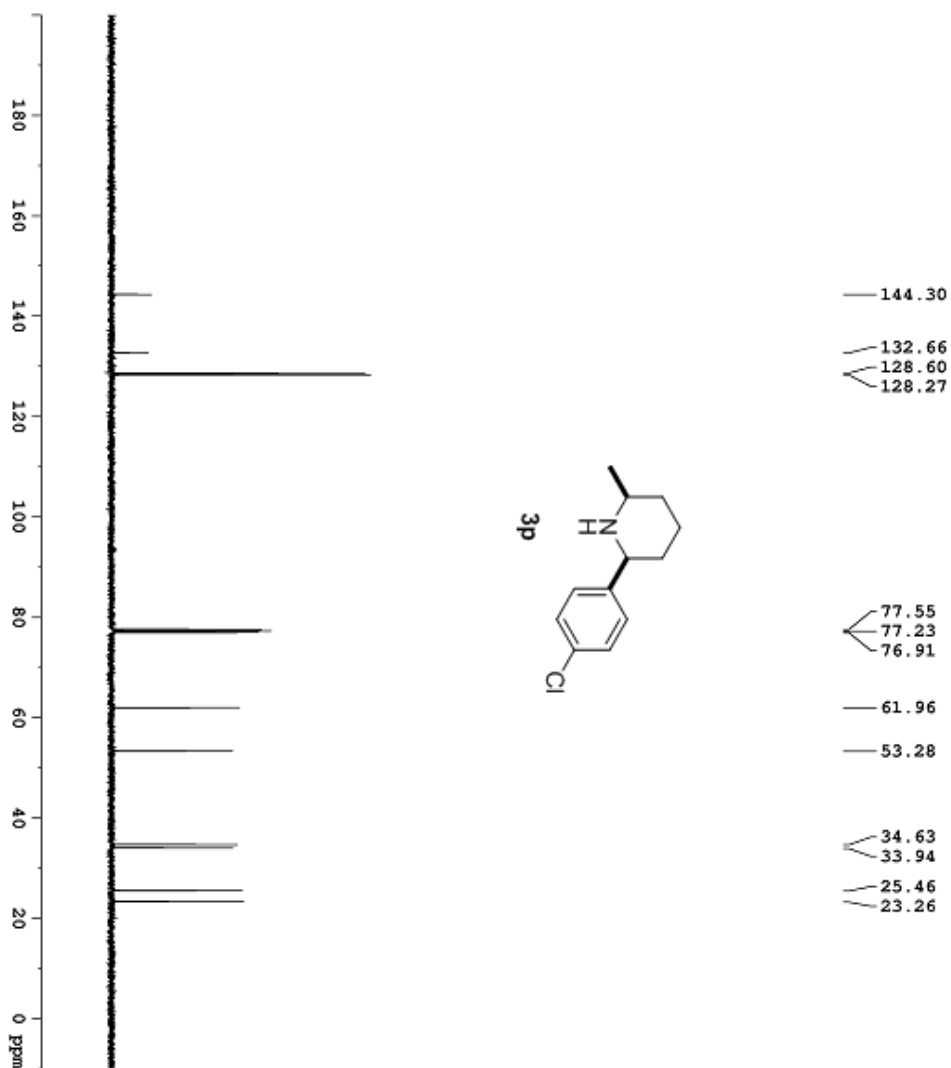
```



```

NAME      1yp-130622-B--2
EXPNO     1
PROCNO    1
Date_     20130507
Time      2.07
INSTRUM    spect
PROBHD     5 mm BBDU-13C
PULPROG    zg30
TD         32768
FIDRES     0.36798 Hz
AQ         1.3631988 sec
RG          80.6
DW         41.600 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300186 MHz
WCMW       EX
SSB        0
GB         0
PC         1.00
  
```

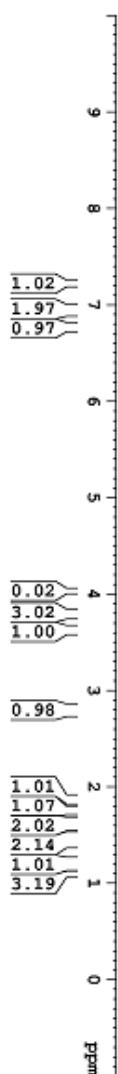
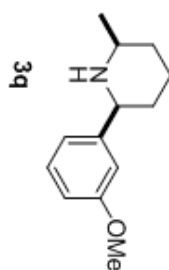


```

NAME      1yb-130622-C
EXPNO     735
PROCNO    1
Date_     20130507
Time      2.10
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         52
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127525 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

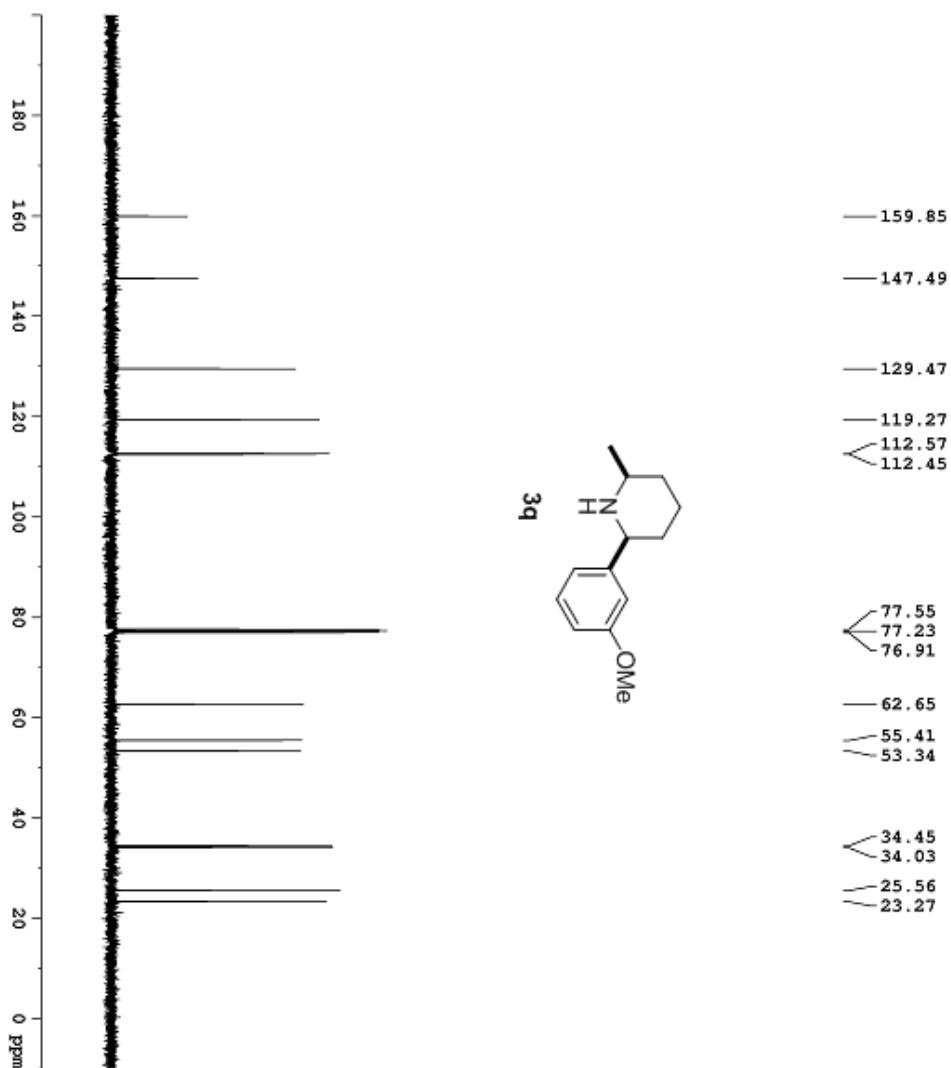
```



```

NAME      1yb-130418--H
EXPNO     712
PROCNO     2
Date_      20130408
Time       10.52
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD          32768
F2          32768
SOLVENT     CDCl3
NS          8
DS          0
SWH         12019.230 Hz
FIDRES     0.366798 Hz
AQ          1.3631988 sec
RG          71.8
RG          71.8
DW          41.600 usec
DE          6.50 usec
TE          295.5 K
D1          2.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300204 MHz
WCM        EX4
SSB        0
GB          0
PC          1.00
  
```

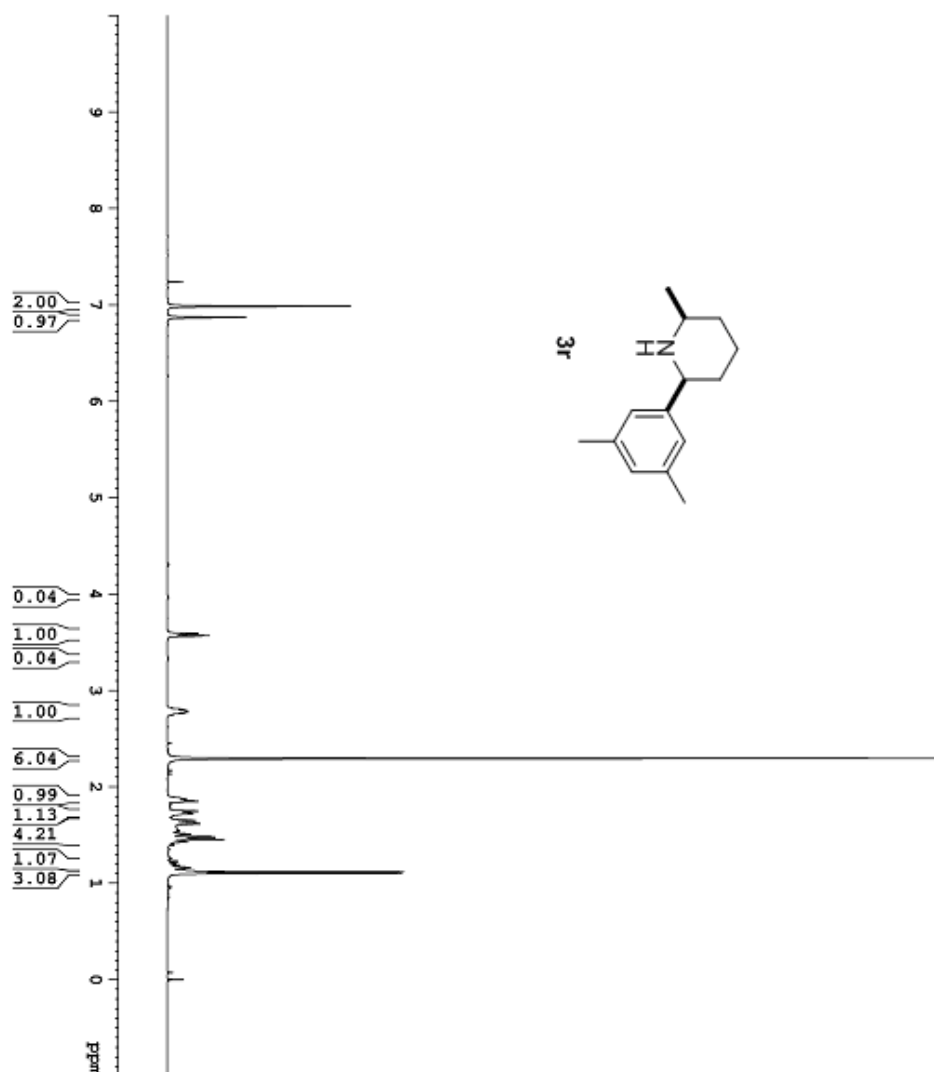
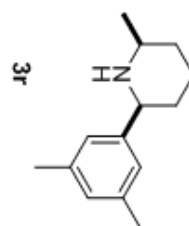


```

NAME      lyb-130622-C
EXPNO     712
PROCNO    1
Date_     20130408
Time      10.55
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         62
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

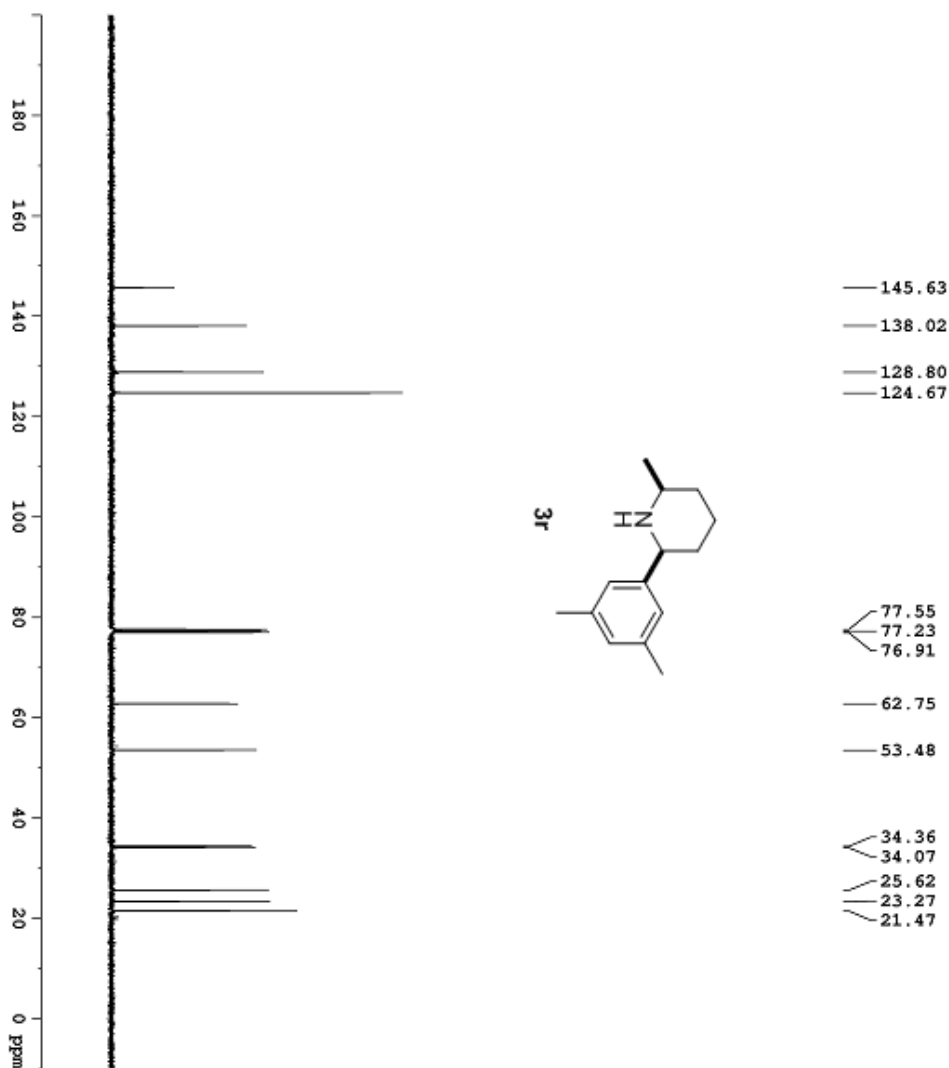
===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127541 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

```



NAME 1yb-130418--H
 EXPNO 707
 PROCNO 2
 Date_ 20130404
 Time 11.15
 INSTRUM spect
 PROBHD 5 mm BBOUL 13C
 PULPROG zg30
 TD 32768
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 64
 DW 41.600 usec
 DE 6.50 usec
 TE 295.7 K
 D1 2.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUCL 1H
 P1 12.60 usec
 SI 65536
 SF 400.1300262 MHz
 WCNW EX
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.00

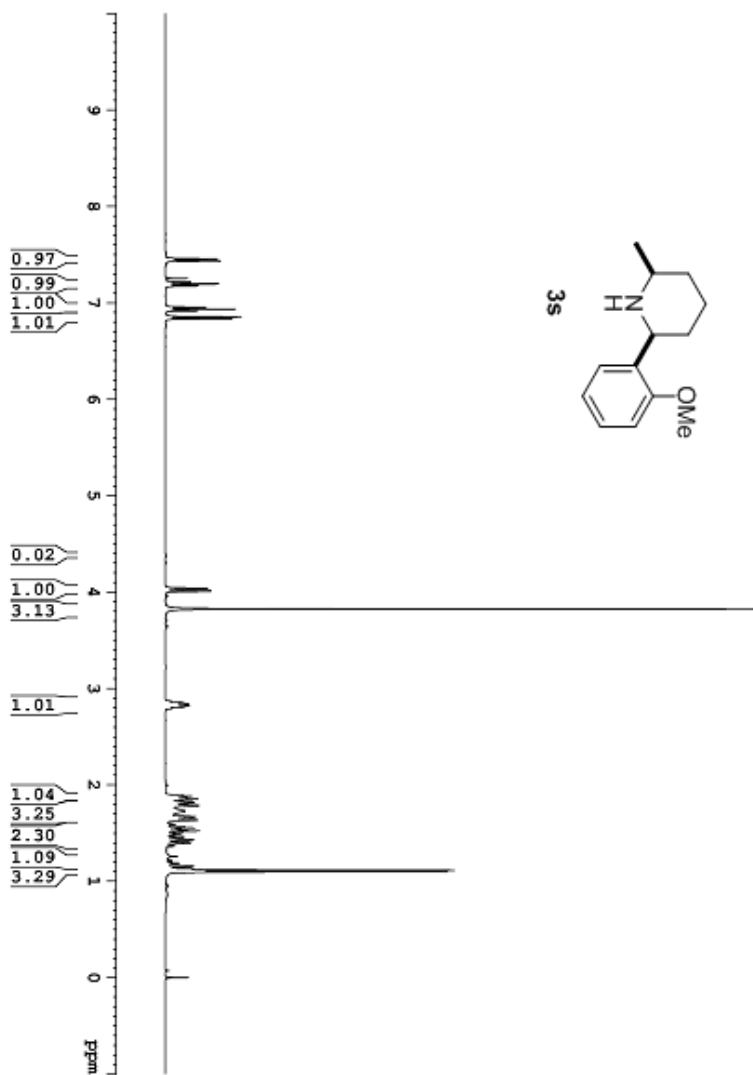
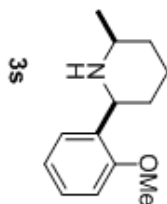


```

NAME      lyb-130622-C
EXPNO     1
PROCNO    1
Date_     20130404
Time      11.21
INSTRUM    spect
PROBHD     5 mm PABD1 13C
PULPROG    zgpg30
TD          32768
SOLVENT     CDCl3
NS          121
DS          0
SWH         25252.525 Hz
FIDRES     0.770646 Hz
AQ          0.6488564 sec
RG          203
DM          19.800 usec
DE          6.50 usec
TE          296.3 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127552 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

```

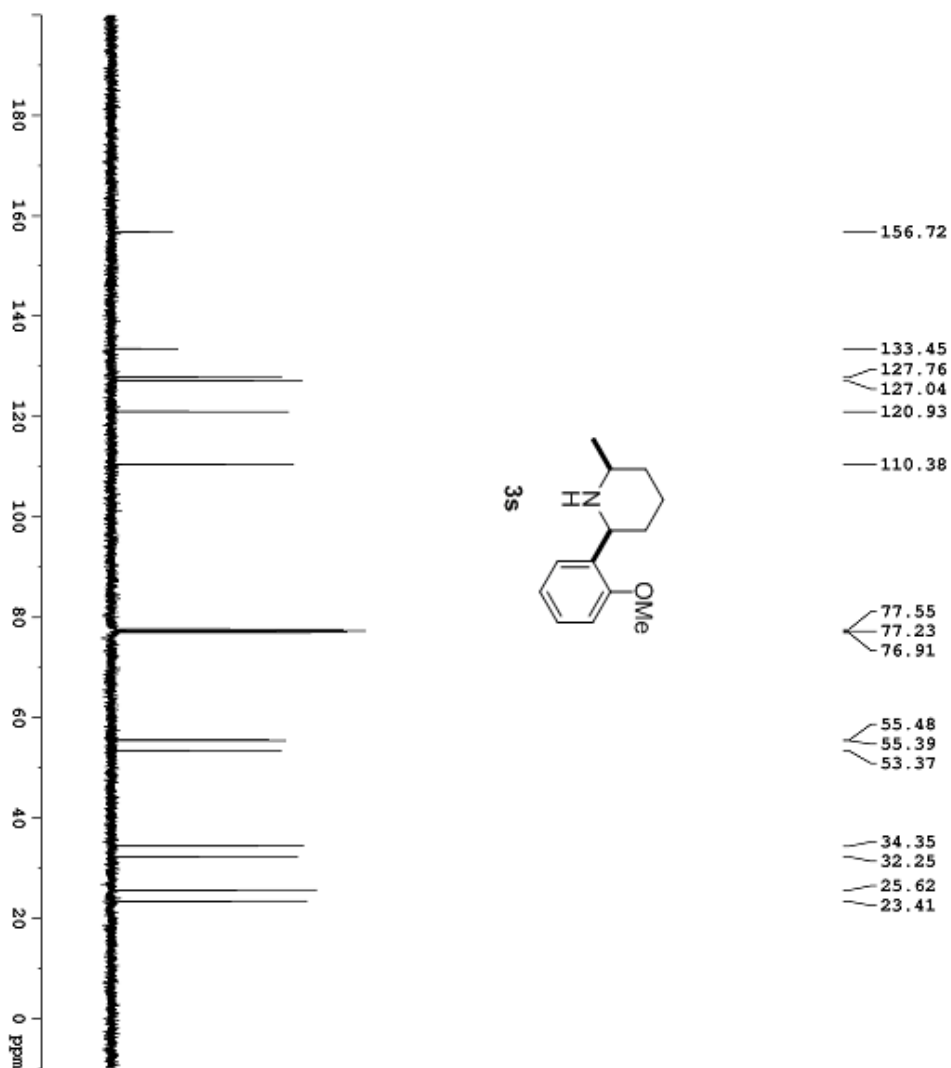


```

NAME      1yb-130418--H
EXPNO     713
PROCNO     2
Date_      20130408
Time       10.57
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD          32768
FIDRES     0.36798 Hz
AQ          1.3631988 sec
RG          41.600 usec
DE          6.50 usec
TE          295.7 K
D1          2.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300208 MHz
WCM        EX
SSB        0
GB         0
PC         1.00

```

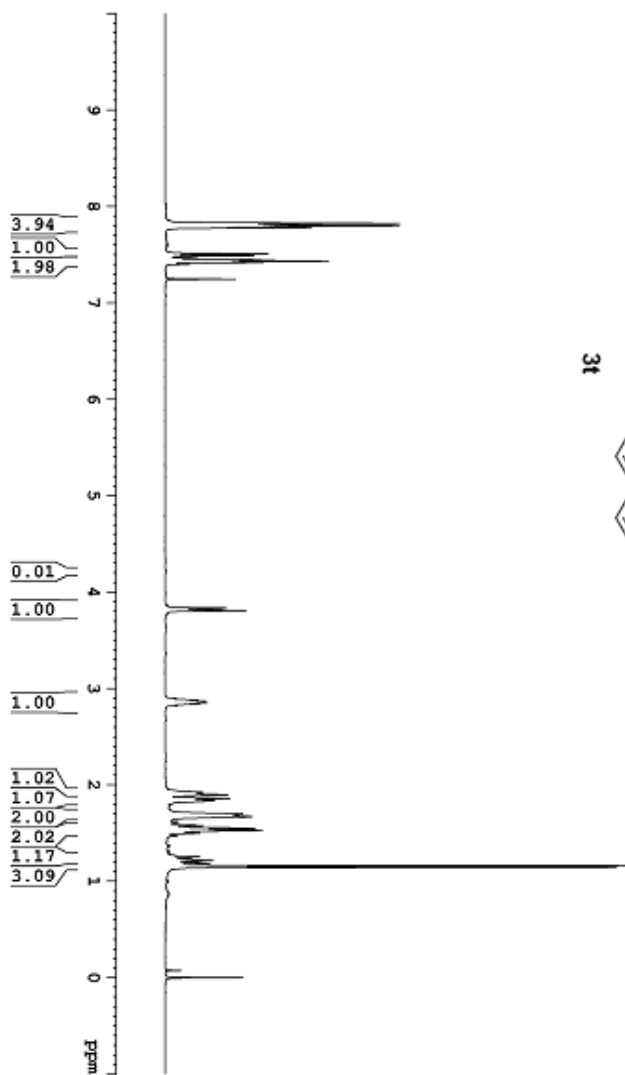
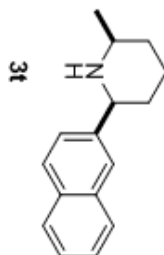


```

NAME      lyb-130622-C
EXPNO     713
PROCNO    1
Date_     20130408
Time      11.00
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         60
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.3 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127541 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

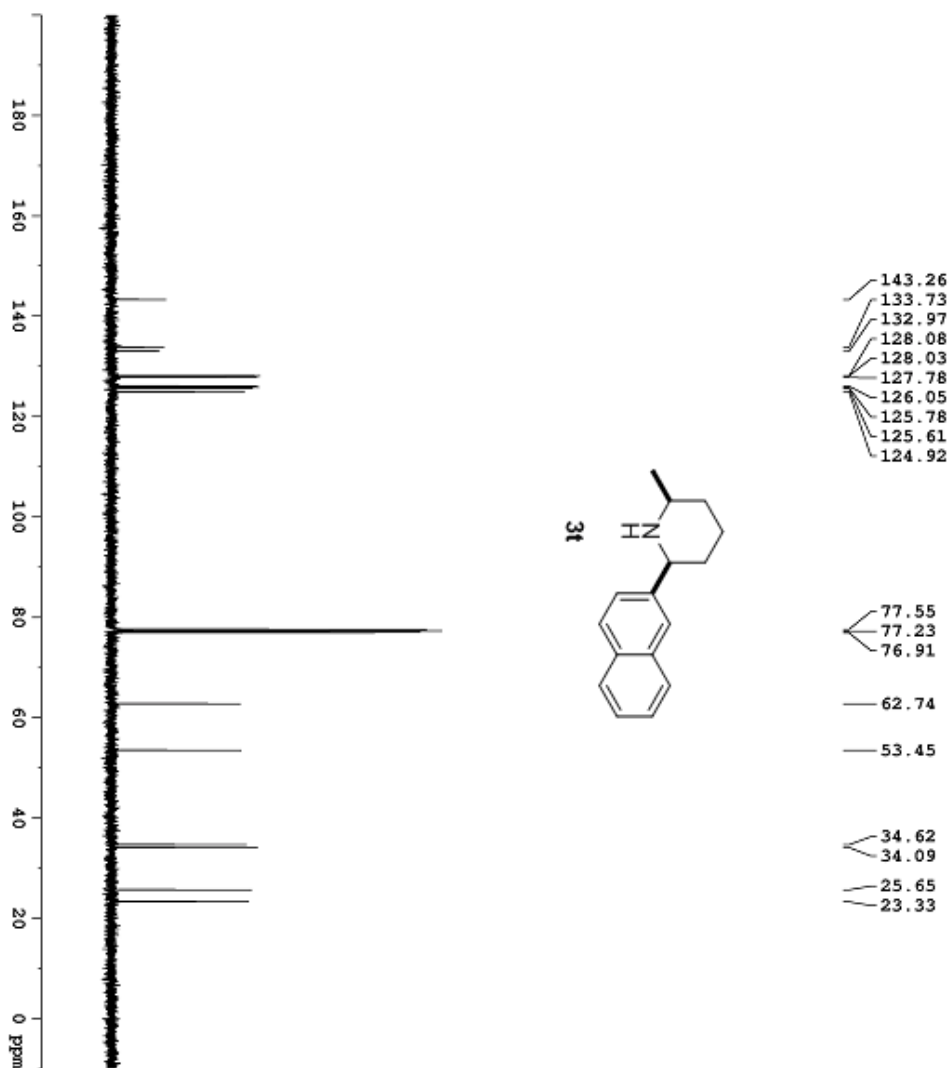
```



```

NAME      1yb-130418--H
EXPNO     702
PROCNO    2
Date_     20130403
Time      6.43
INSTRUM    spect
PROBHD     5 mm BBDP13C
PULPROG    zg30
TD         32768
FIDRES     0.36798 Hz
AQ         1.3631988 sec
RG         114
DW         41.600 usec
DE         6.50 usec
TE         295.5 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      1H
P1        12.60 usec
SI        65536
SF        400.1300231 MHz
WCMW      EX4
SSB       0
GB        0
PC        1.00
  
```

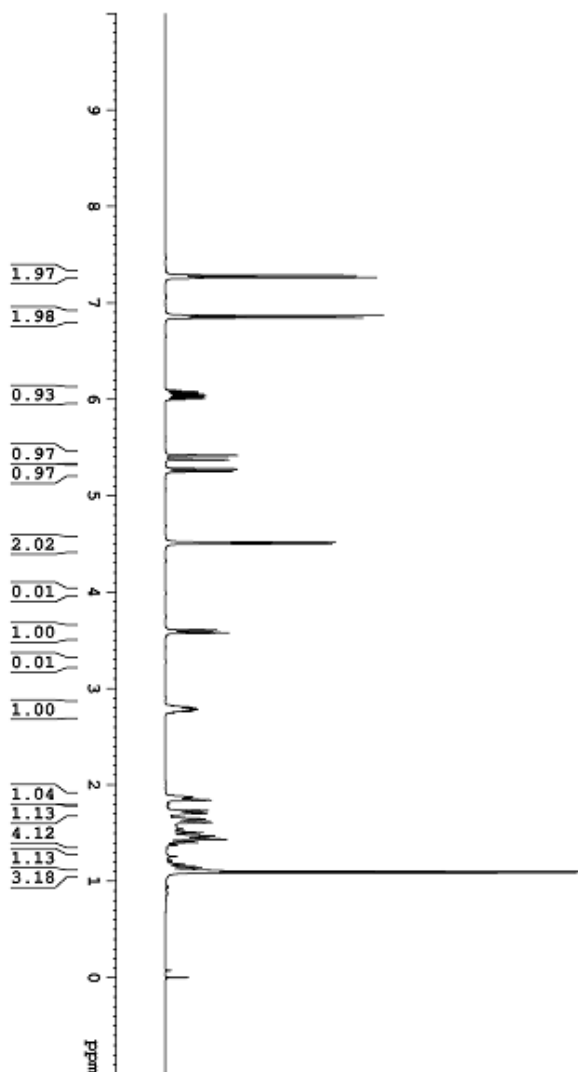
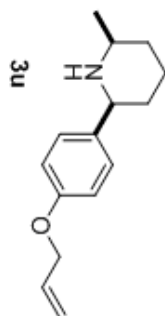


```

NAME      lyb-130622-C
EXPNO     702
PROCNO    1
Date_     20130403
Time      6.49
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         123
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.1 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127525 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40

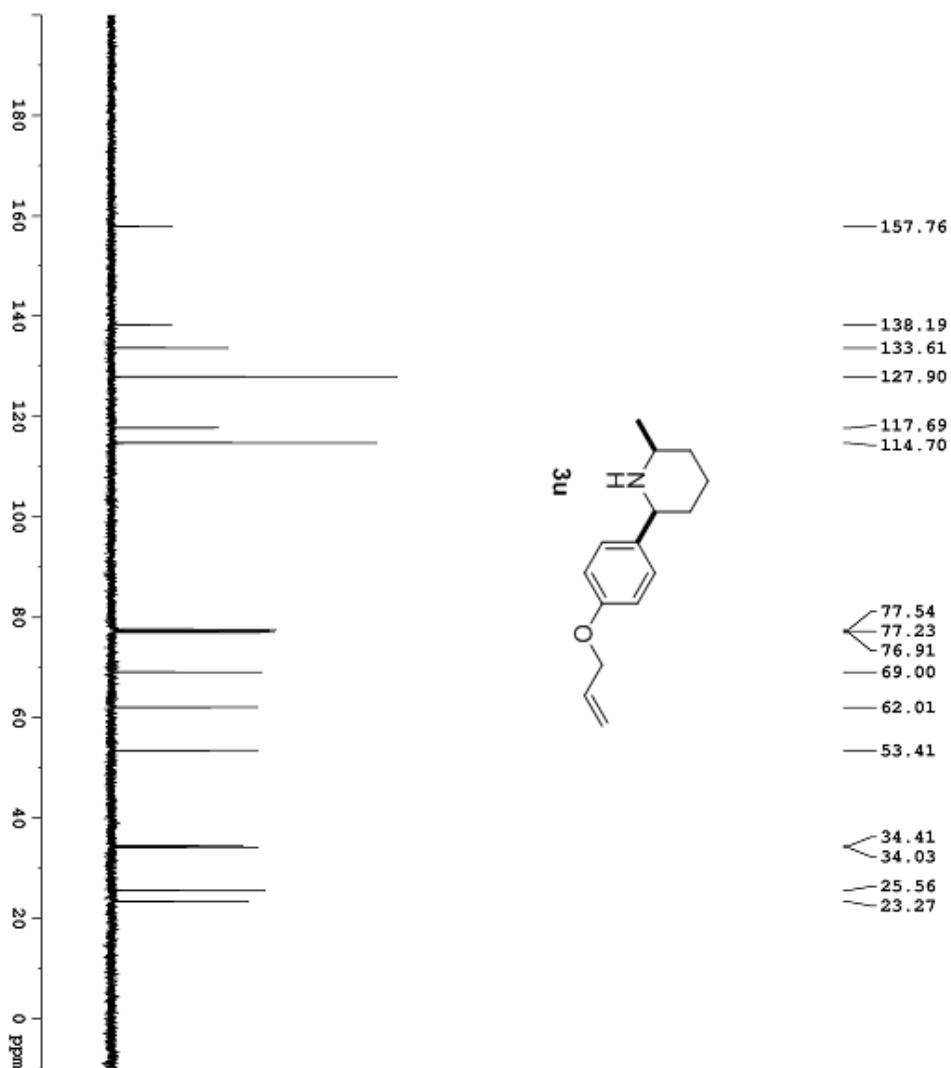
```



```

NAME      1yb-130622-B--2
EXPNO     2
PROCNO    2
Date_     20130514
Time      9.02
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
FIDRES     0.36798 Hz
AQ         1.3631988 sec
RG         71.8
DW         41.600 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
TD0        1

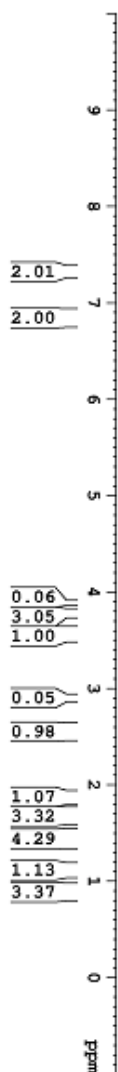
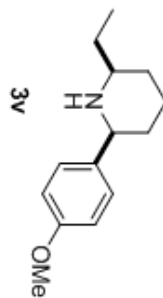
===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300206 MHz
WCMW       EX
SSB        0
GB         0
PC         1.00
  
```



```

NAME      lyb-130622-C
EXPNO     1
PROCNO    1
Date_     20130514
Time      9.08
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         110
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

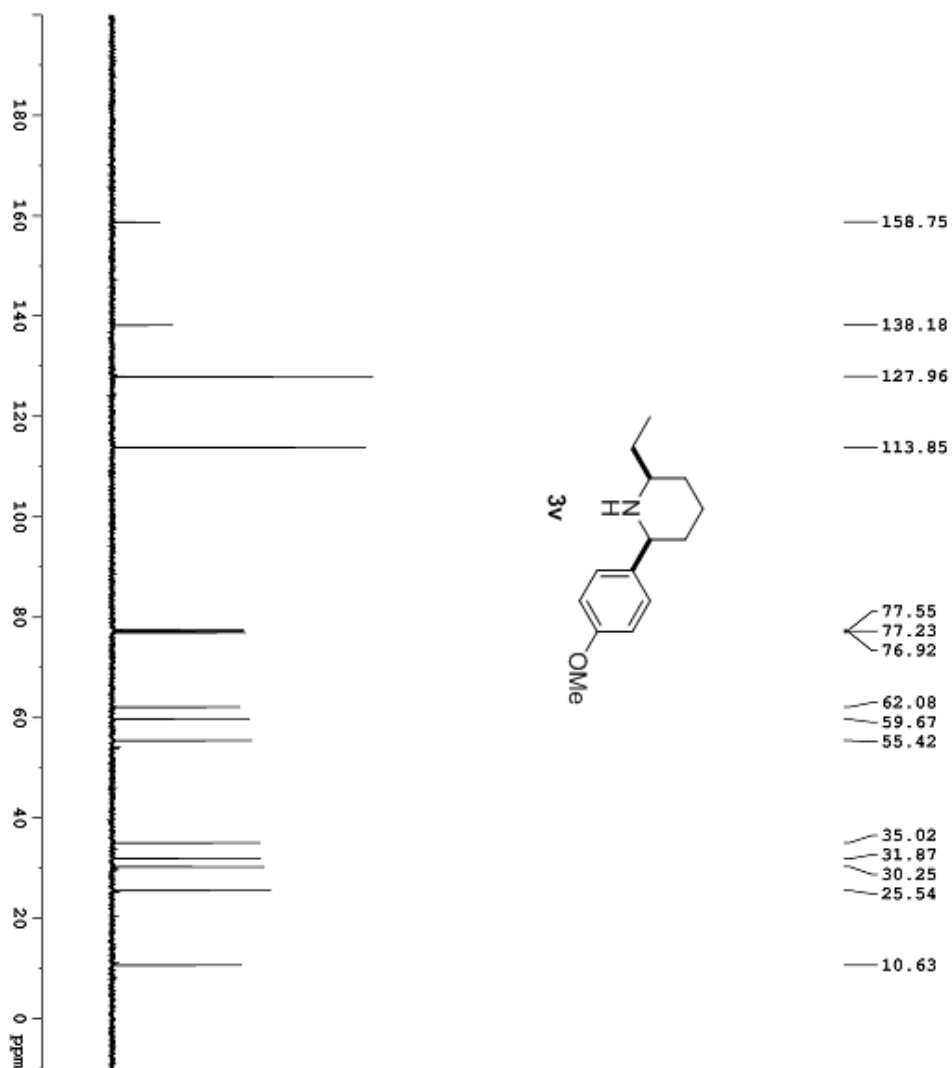
===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127549 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40
  
```



```

NAME      1yb-130622-B--2
EXPNO     748
PROCNO    2
Date_     20130508
Time      11.36
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD          32768
F2         32768
SOLVENT    CDCl3
NS          8
DS          0
SWH         12019.230 Hz
FIDRES     0.366798 Hz
AQ          1.3631988 sec
RG          57
RG          57
DW          41.600 usec
DE          6.50 usec
TE          300.0 K
D1          2.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300226 MHz
WCM        EX4
SSB        0
GB          0
PC          1.00
  
```

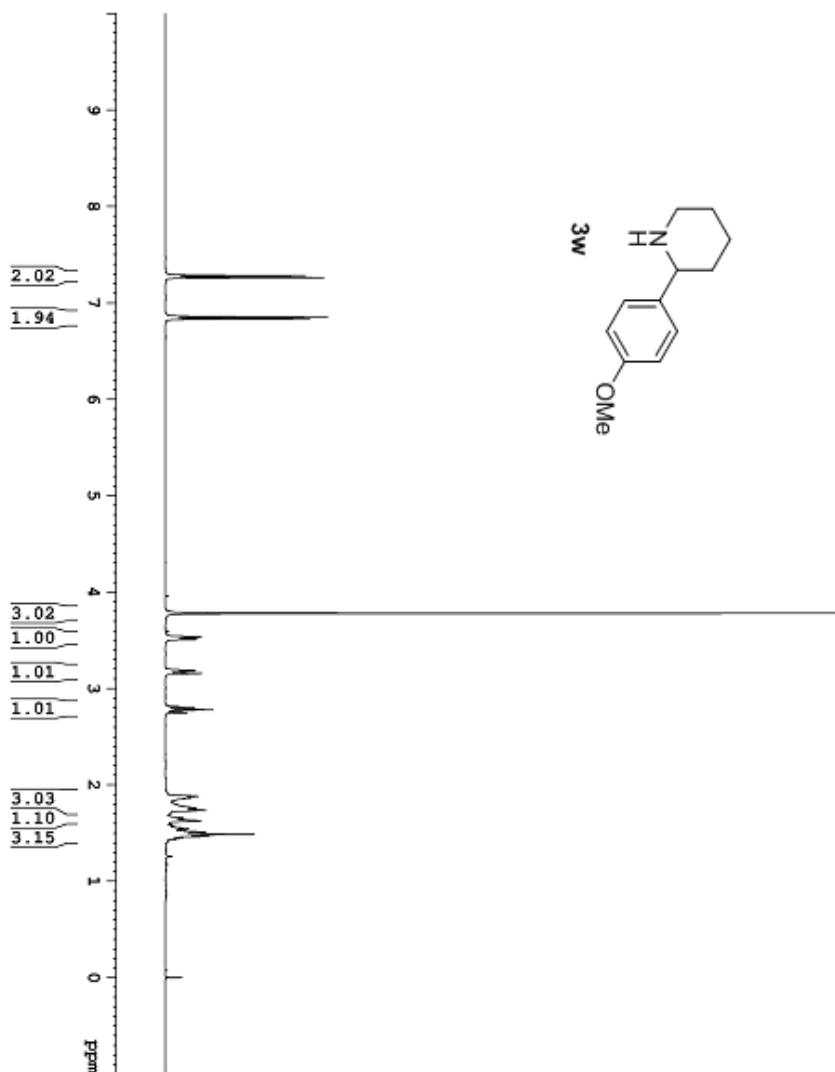
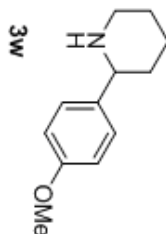


```

NAME      1yb-130622-C
EXPNO     748
PROCNO    1
Date_     20130508
Time      11.40
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         80
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127549 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

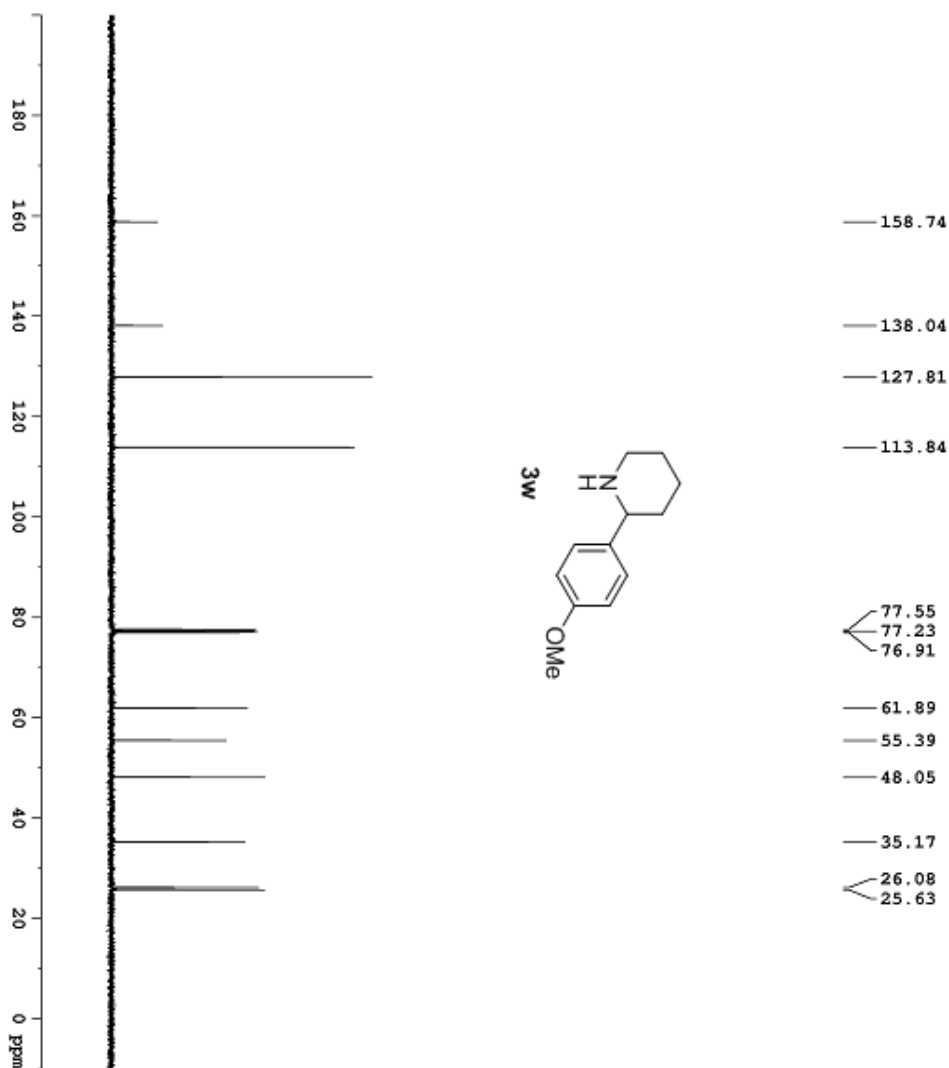
```



```

NAME      1yb-130622-B--2
EXPNO     2
PROCNO    2
Date_     20130522
Time      8.21
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
FIDRES     0.36798 Hz
AQ         1.3631988 sec
RG         71.8
DW         41.600 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300176 MHz
WCM        EX
SSB        0
GB         0
PC         1.00
  
```

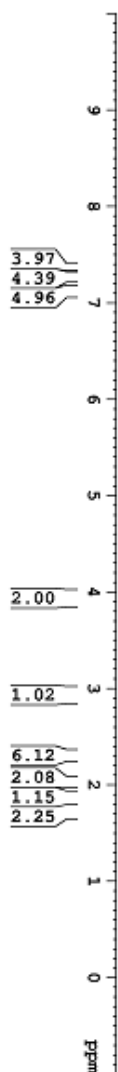
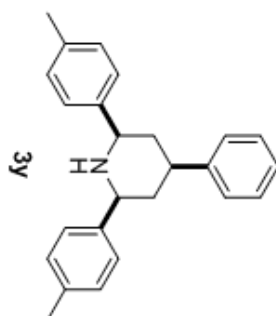


```

NAME      1yb-130622-C
EXPNO     1
PROCNO    1
Date_     20130522
Time      8.24
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         66
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127556 MHz
WDW       EM
SSB       0
GB        0
PC        1.00 Hz
EC        1.40

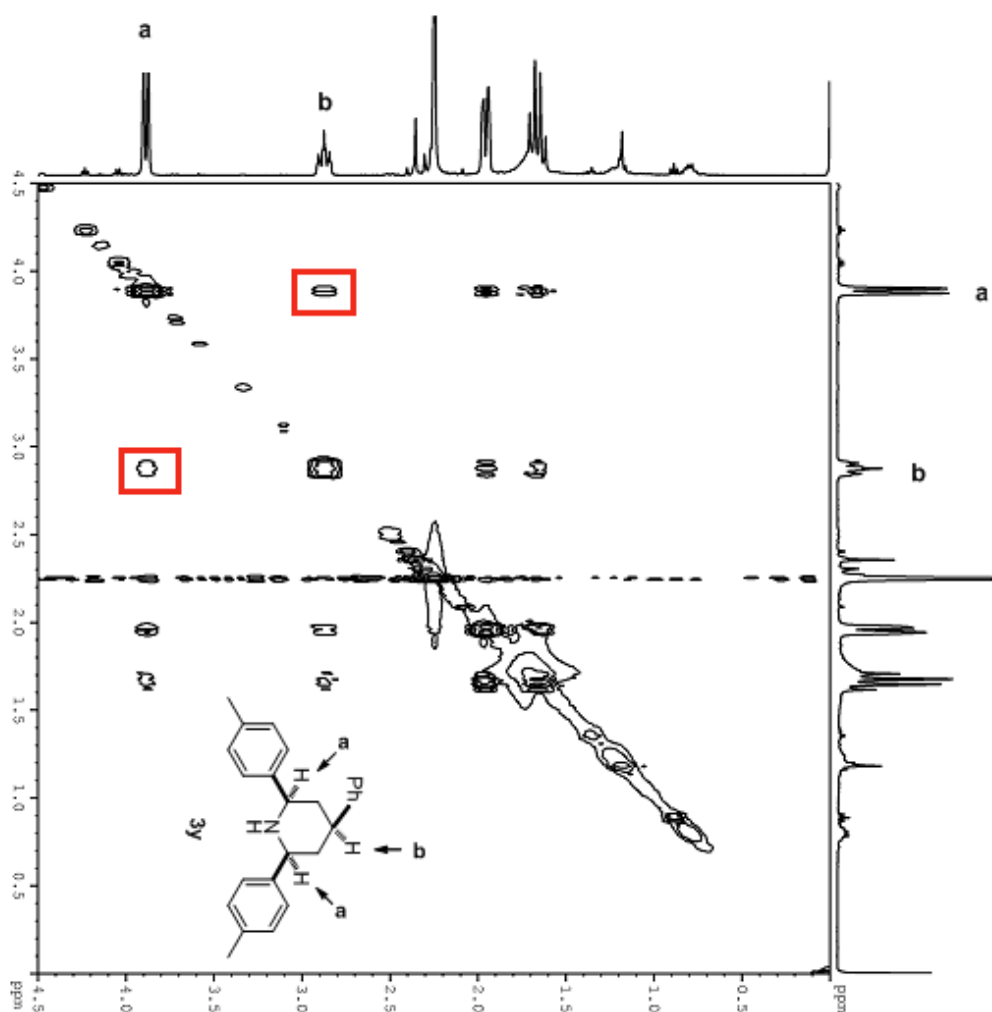
```



```

NAME      1yb-130622-B--2
EXPNO     3
PROCNO    3
Date_     20130607
Time      6.56
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
F2        32768
SOLVENT    CDCl3
NS          8
DS          0
SWH         12019.230 Hz
FIDRES      0.366798 Hz
AQ          1.3631988 sec
RG          71.8
RG          71.8
DW          41.600 usec
DE          6.50 usec
TE          300.0 K
D1          2.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300450 MHz
WCM        EX
SSB         0
GB          0
PC          1.00
  
```



Current Data Parameters
NAME H-H
EXPNO 7
PROCNO 1

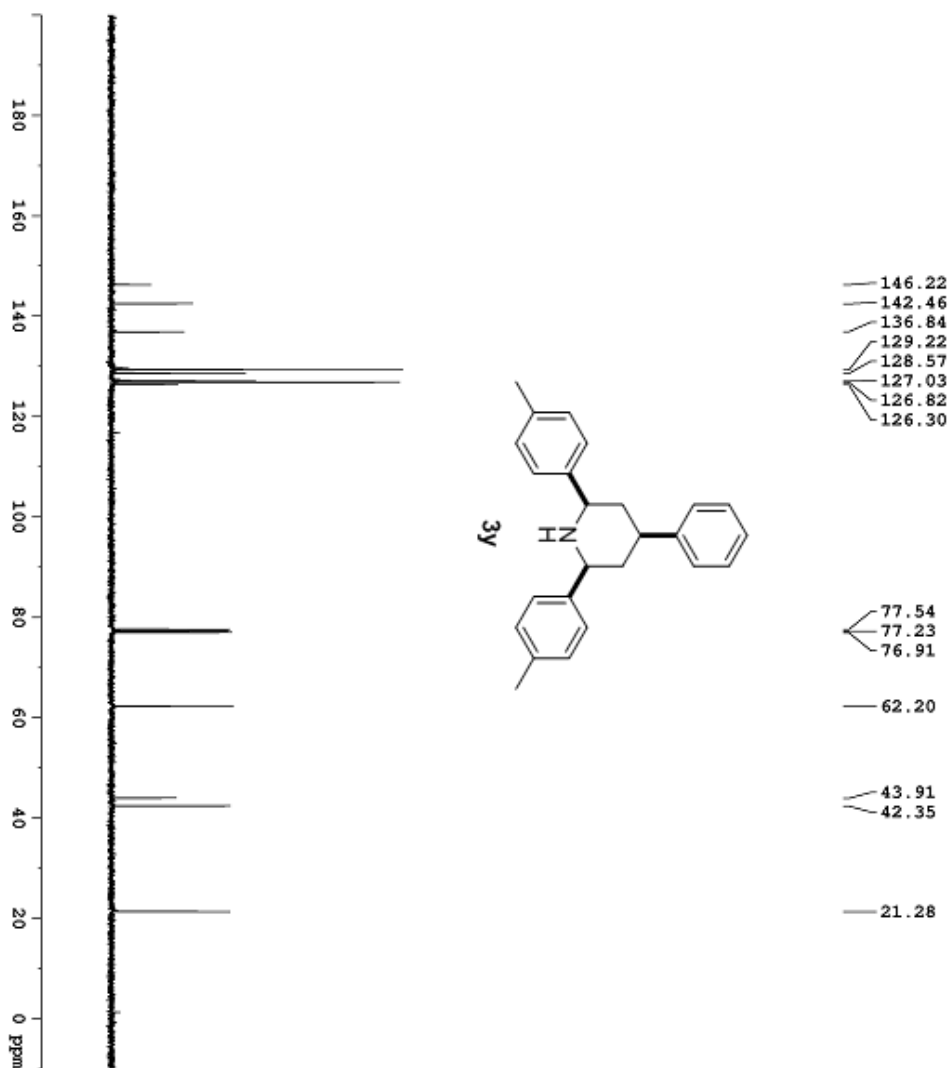
F2 - Acquisition Parameters
Date_ 20130620
Time 13.44
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG noesypppp
TD 2048
SOLVENT CDCl3
NS 4
DS 32
SWH 3759.398 Hz
FIDRES 1.835644 Hz
AQ 0.2724340 sec
RG 64
DE 133.000 usec
TE 300.0 K
DO 0.0001696 sec
D1 1.97829103 sec
D8 0.30000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
INO 0.00026600 sec

===== CHANNEL f1 =====
NUC1 1H
P1 12.60 usec
P2 25.20 usec
P17 2500.00 usec
PL1 13.0600042 W
PL12 3.06719995 W
SFO1 400.1316558 MHz

===== GRADIENT CHANNEL =====
GPM1 SSBQ10.100
GPZ1 48.00 V
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 400.1317 MHz
FIDRES 14.663150 Hz
SM 9.395 PPM
FWDOME States-TPE1

F2 - Processing parameters
SI 1024
SF 400.1300967 MHz
WDW OSINE
SSB 2
TB 0 Hz

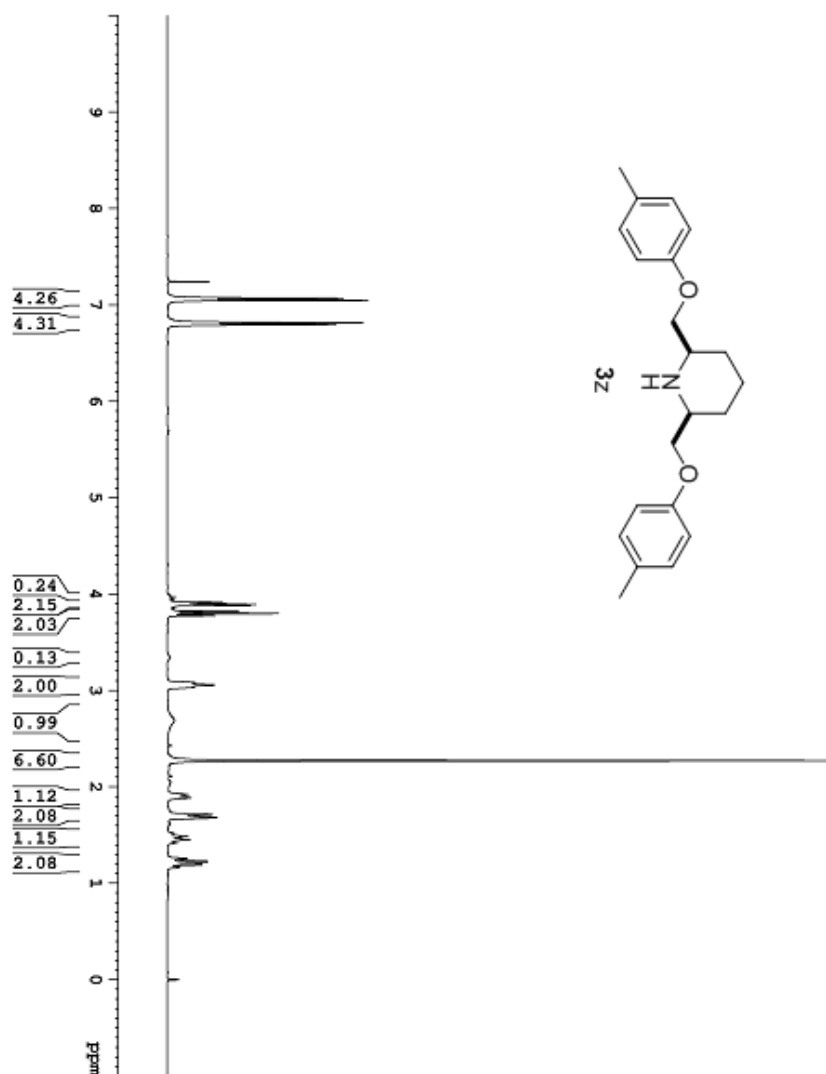
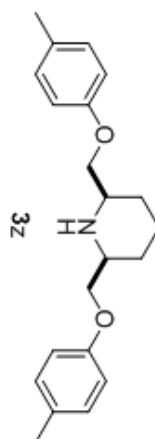


```

NAME      1y-130622-C
EXPNO     773
PROCNO    2
Date_     20130607
Time      6.59
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         52
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
SI        32768
SF        100.6127587 MHz
WDW       EM
SSB       0
GB        0
PC        1.00 Hz
EC        1.40

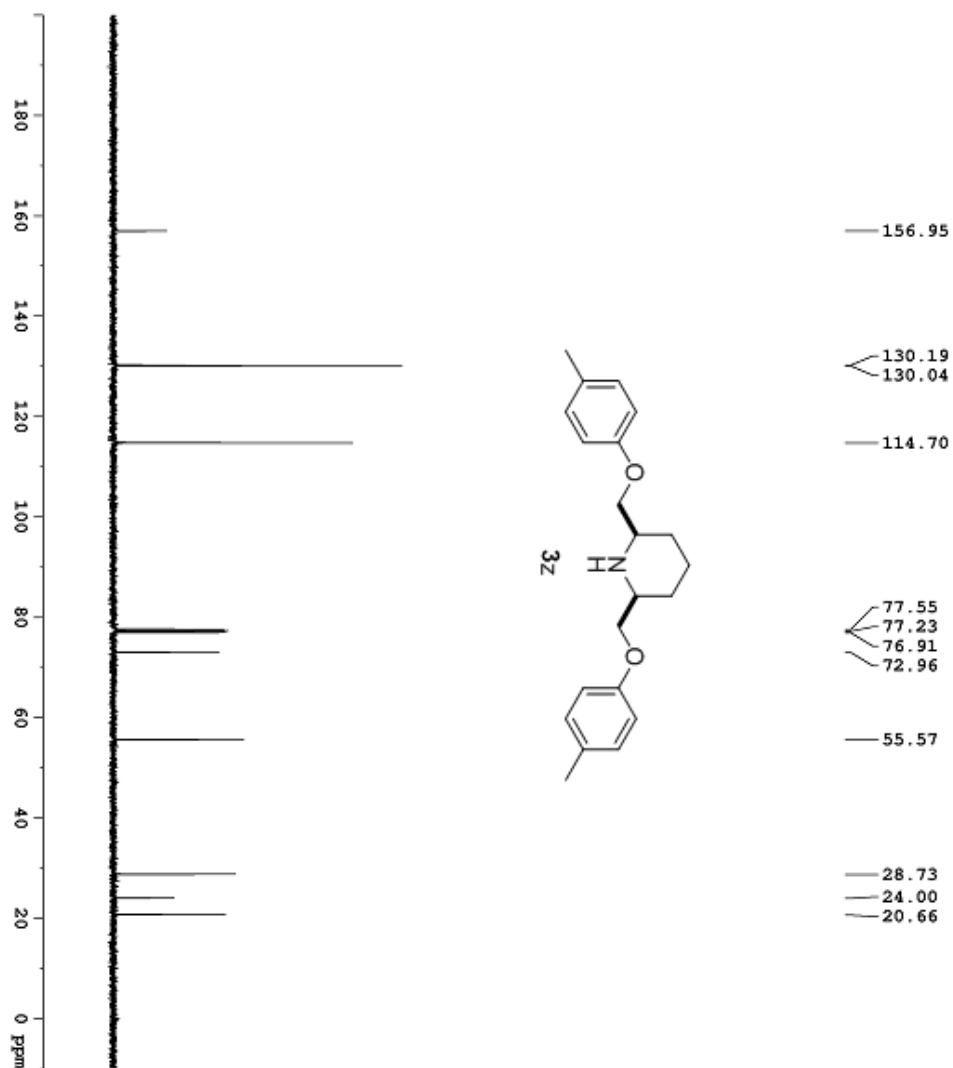
```



```

NAME      1yb-130622-B--2
EXPNO     2
PROCNO    2
Date_     20130606
Time      7.47
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD          32768
FIDRES     0.36798 Hz
AQ          1.3631988 sec
RG          90.5
DW          41.600 usec
DE          6.50 usec
TE          300.0 K
D1          2.00000000 sec
TD0         1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300255 MHz
WCMW       EX4
SSB        0
GB          0
PC          1.00
  
```

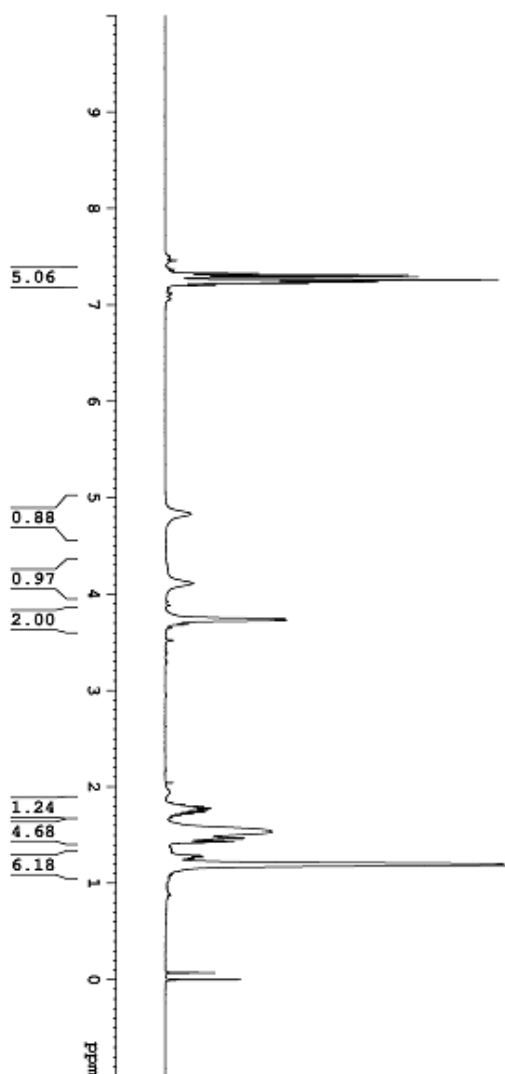
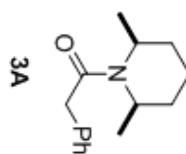


```

NAME      1yb-130622-C
EXPNO     799
PROCNO    1
Date_     20130606
Time      7.50
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         64
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ          0.6488564 sec
RG          203
DM         19.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1      13C
P1         9.40 usec
SI        32768
SF       100.6127549 MHz
WDW        EM
SSB         0
GB         0
PC         1.00 Hz
EC         1.40

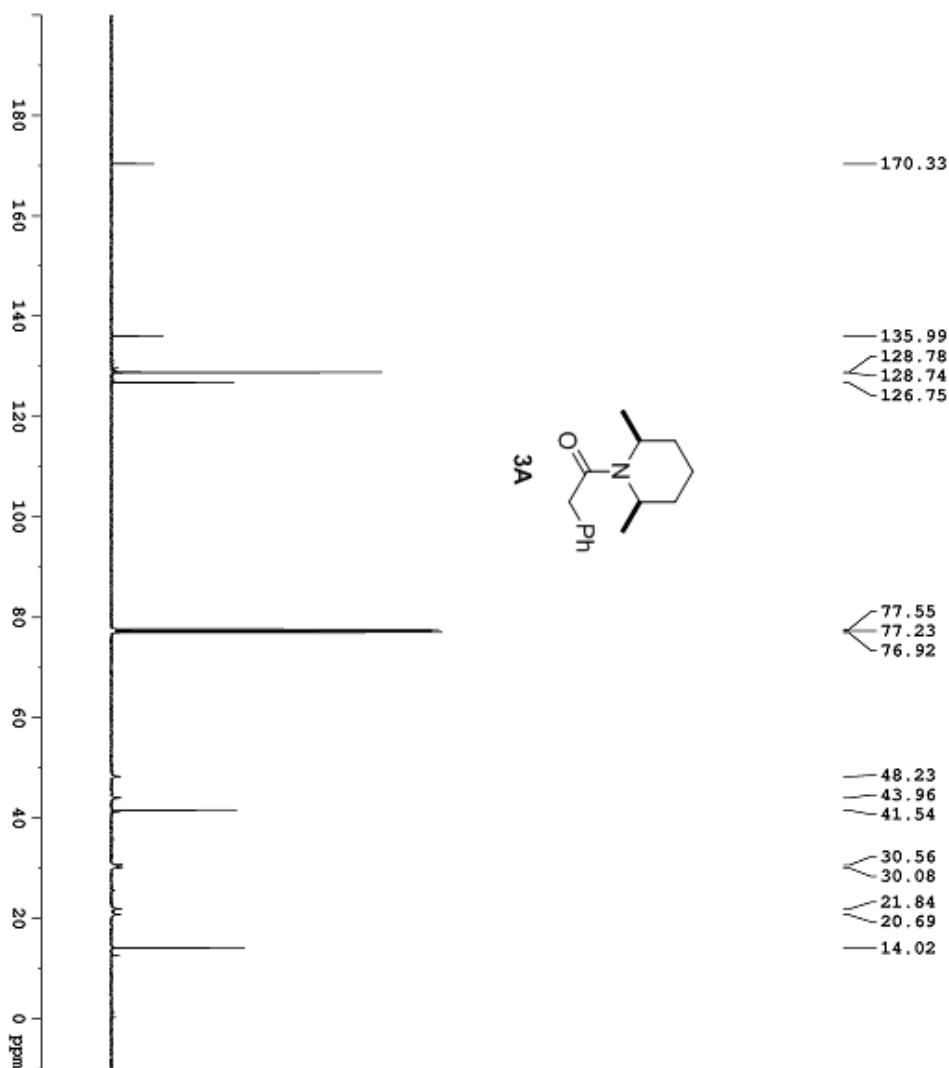
```



```

NAME      1yb-130622-B--2
EXPNO     4
PROCNO    778
Date_     20130524
Time      8.35
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
FIDRES     0.36798 Hz
AQ         1.3631988 sec
RG         128
RG         41.600 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300156 MHz
WCMW       EX
SSB        0
GB         0
PC         1.00
  
```

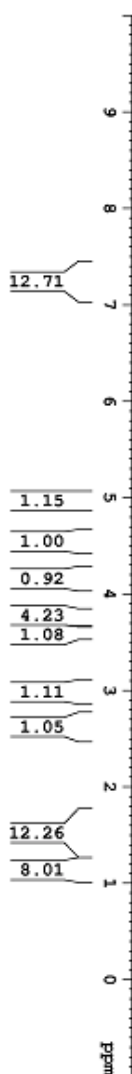
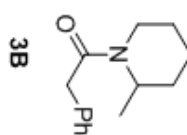


```

NAME      lyb-130622-C
EXPNO     1
PROCNO    1
Date_     20130524
Time      2.40
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         3108
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127518 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

```

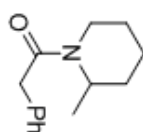
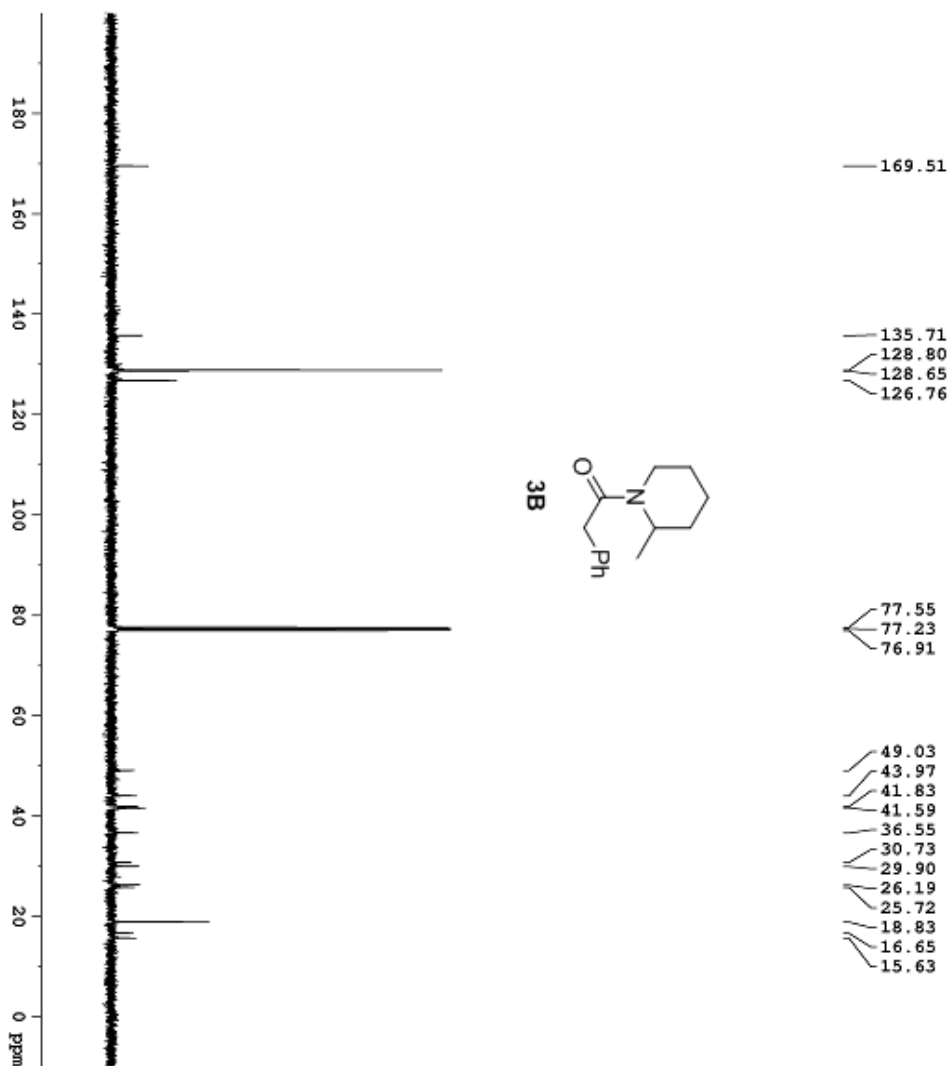


```

NAME      1yb-130622-B--2
EXPNO     1
PROCNO    1
Date_     20130528
Time      9.12
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
FIDRES     0.36798 Hz
AQ         1.3631988 sec
RG         203
DW         41.600 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300150 MHz
WCMW       EX
SSB        0
GB         0
PC         1.00

```

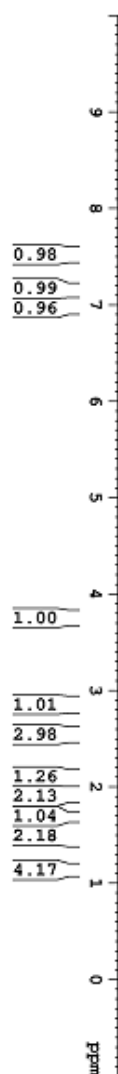
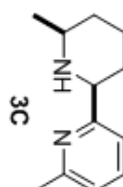


```

NAME      1yb-130622-C
EXPNO     783
PROCNO    1
Date_     20130528
Time      9.11
INSTRUM   spect
PROBHD    5 mm PABUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         1
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127533 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

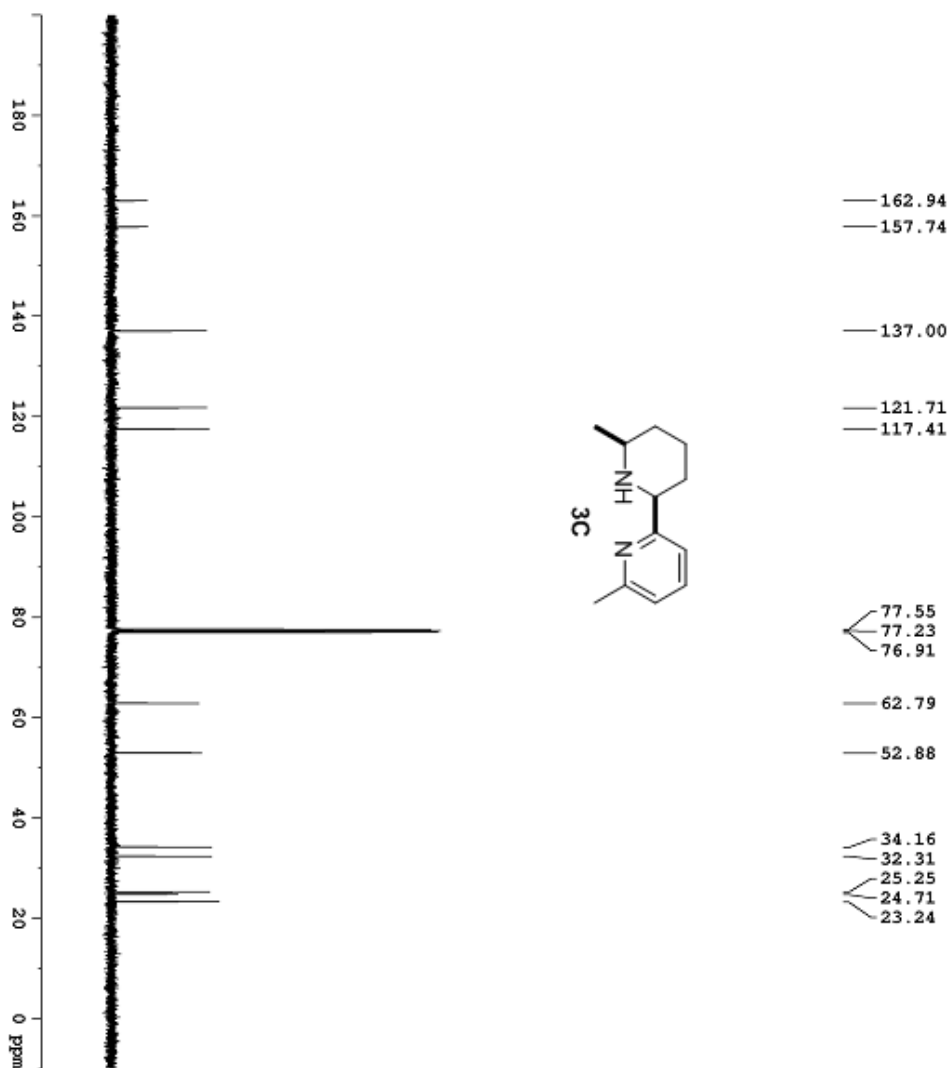
```



```

NAME      1yb-130622-B--2
EXPNO     1
PROCNO    1
Date_     20130530
Time      11.12
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         144
DW         41.600 usec
DE         6.50 usec
TE         300.0 K
D1         2.00000000 sec
TD0        1

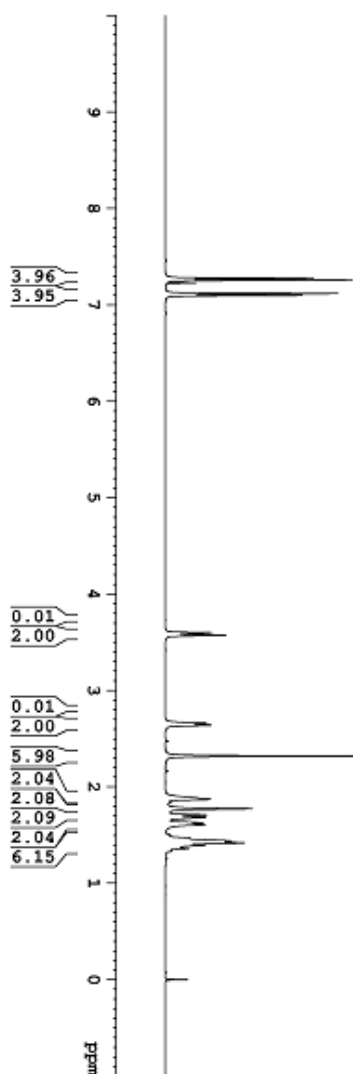
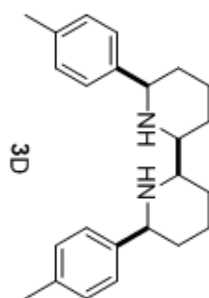
===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300134 MHz
WCMW       EX
SSB        0
GB         0
PC         1.00
  
```



```

NAME      lyb-130622-C
EXPNO     726
PROCNO    2
Date_     20130418
Time      9.20
INSTRUM   spect
PROBHD    5 mm PABDUL 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         112
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.3 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127510 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

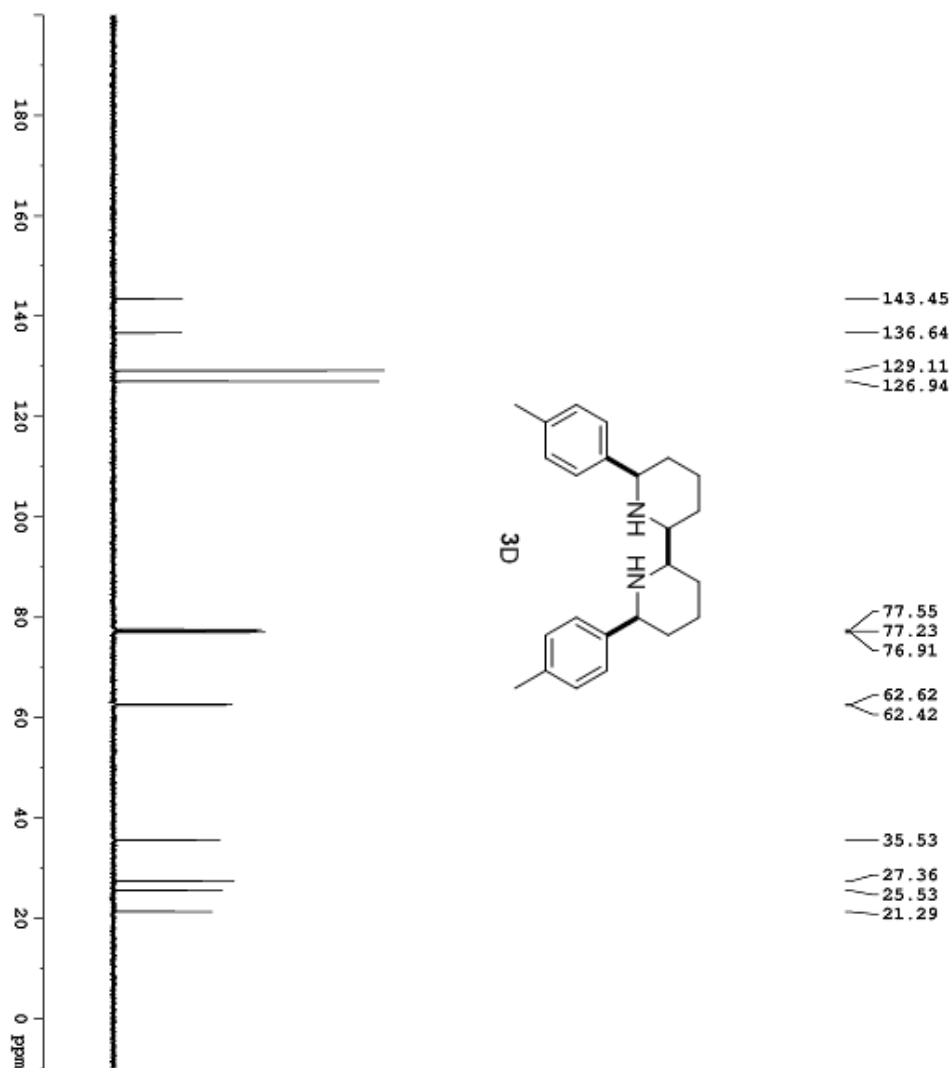


```

NAME      1yb-130418--H
EXPNO     2
PROCNO    2
Date_     20130418
Time      12.57
INSTRUM    spect
PROBHD     5 mm BBDPL 13C
PULPROG    zg30
TD         32768
FIDRES     0.36798 Hz
AQ         1.3631988 sec
RG         80.6
DW         41.600 usec
DE         6.50 usec
TE         295.7 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         12.60 usec
SI         65536
SF         400.1300307 MHz
WCM        EX4
SSB        0
GB         0
PC         1.00

```

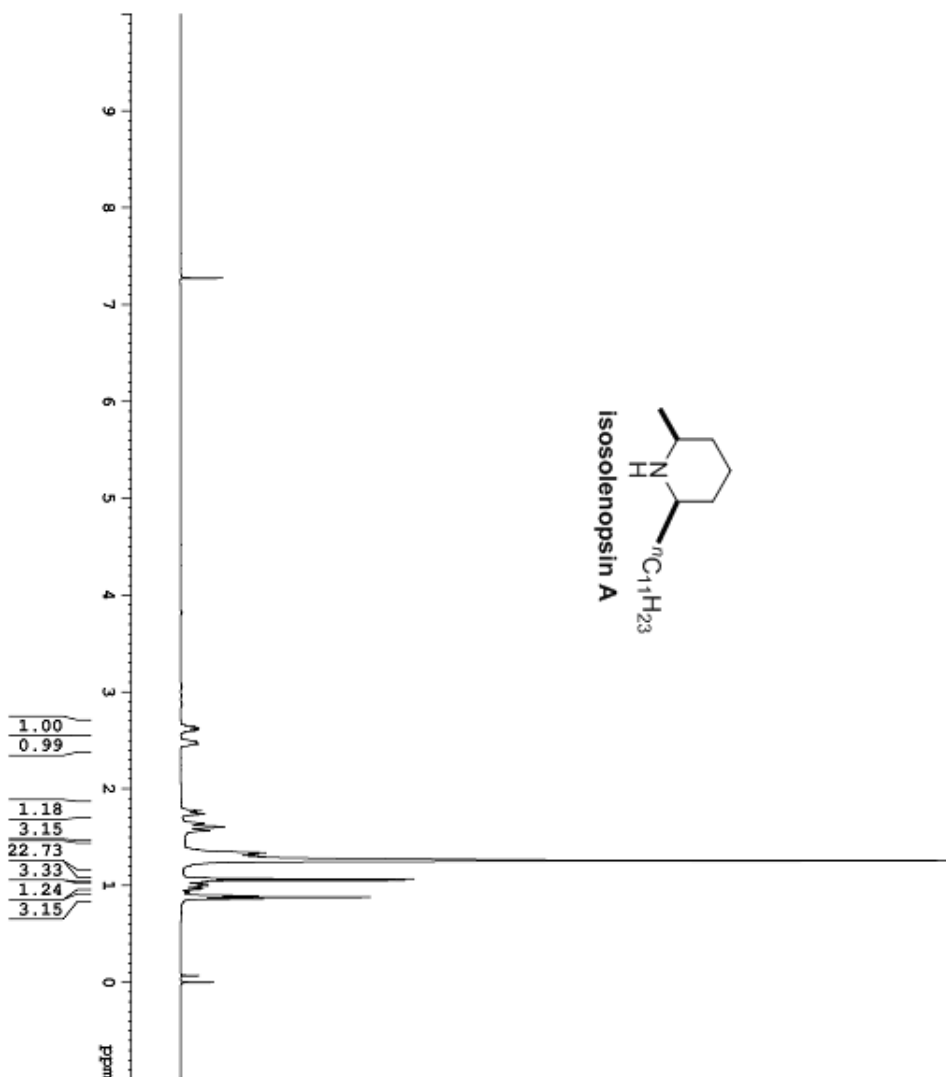
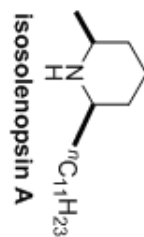


```

NAME      lyb-130622-C
EXPNO     727
PROCNO    1
Date_     20130418
Time      13.05
INSTRUM   spect
PROBHD    5 mm PABD1 13C
PULPROG   zgpg30
TD         32768
SOLVENT   CDCl3
NS         166
DS         0
SWH        25252.525 Hz
FIDRES     0.770646 Hz
AQ         0.6488564 sec
RG         203
DM         19.800 usec
DE         6.50 usec
TE         296.4 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.40 usec
SI         32768
SF         100.6127544 MHz
WDW        EM
SSB        0
GB         0
PC         1.00 Hz
EC         1.40

```



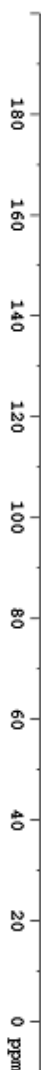
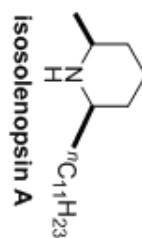
Current Data Parameters
NAME 1yb
EXPNO 814
PROCNO 23

F2 - Acquisition Parameters
Date_ 20130703
Time 9.26
INSTRUM spect
PROBHD 5 mm PABOL 13C
PULPROG zgpg30
TD 32768
FIDRES 0.36798 Hz
AQ 1.363198 sec
RG 90.5
DW 41.600 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL F1 =====
NUC1 1H
P1 12.60 usec
PL1 13.06000042 W
SF01 400.1320007 MHz

F2 - Processing parameters
SI 65536
SF 400.1300133 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

77.55
77.23
76.91
57.37
52.70
37.69
34.66
32.50
32.13
30.06
29.88
29.84
29.82
29.56
26.22
25.11
23.32
22.89
14.32



Current Data Parameters
NAME 1yb-C
EXPNO 815
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130703
Time 9.48
INSTRUM spect
PROBHD 5 mm PABUL 13C
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 180
DS 0
SWH 25252.525 Hz
FIDRES 0.770646 Hz
AQ 0.648564 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL #1 =====
NUC1 13C
P1 9.40 usec
PL1 43.6389994 W
SFO1 100.628298 MHz

===== CHANNEL #2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 13.06000042 W
PLW12 0.25598001 W
PLW13 0.20734000 W
SFO2 400.1316005 MHz

F2 - Processing Parameters
SI 32768
SF 100.6127495 MHz
WOW EN
SSB 0
IB 1.00 Hz
GB 0
PC 1.40