

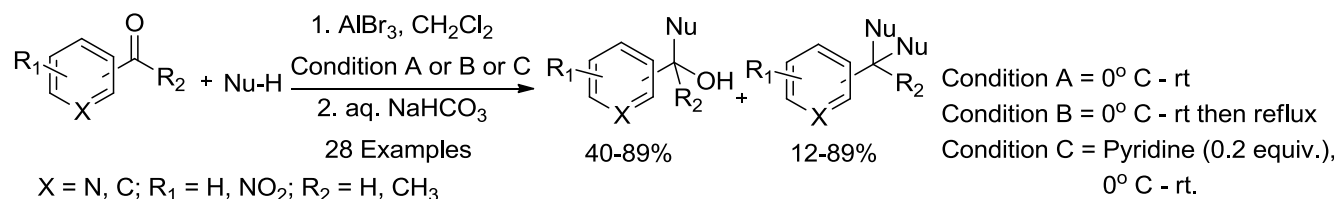
## Electronic Supplementary Information

### **Friedel-Crafts hydroxyalkylation through activation of carbonyl group using AlBr<sub>3</sub>: An easy access to pyridyl aryl / heteroaryl carbinols†**

**Adhikesavan Hari Krishnan, Jayaraman Selvakumar, Elumalai Gnanamani, Suman Bhattacharya and Chinnasamy Ramraj Ramanathan\***

Department of Chemistry, Pondicherry University, Puducherry – 605 014, India.

*E-mail: crrnath.che@pondiuni.edu.in*



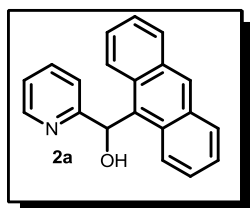
**General information:** Melting points reported in this paper are uncorrected and were determined using EZ Melt, Stanford Research Systems, USA. Infrared spectra were recorded on Thermo Nicolet 6700 FT-IR Spectrophotometer and are reported in frequency of absorption ( $\text{cm}^{-1}$ ). Mass spectra were measured with micro mass Q-TOF (ESI-HRMS),  $^1\text{H}$  and  $^{13}\text{C}$  NMR were recorded on Bruker AVANCE 400 spectrometer. NMR spectra for all the samples were measured in  $\text{CDCl}_3$  using TMS as an internal standard. The chemical shifts are expressed in  $\delta$  ppm down field from the signal of internal TMS.

Aluminum bromide (solid), pyridine was purchased from Aldrich and used without further purification. Carboxaldehydes were purchased from Aldrich and purified by distillation under reduced pressure. Nucleophiles **1d**, **1e**, **1f**, **1g**, **1h**, **1i** and **1j** were prepared from corresponding phenols using reported procedure.<sup>1</sup> Solvents used for the reactions were dried using standard procedures.<sup>2</sup> Analytical thin layer chromatographic tests were carried out on glass plates (3 x 10 cm) coated with Himedia's silica gel GF<sub>254</sub> containing calcium sulphate as binder for TLC. The spots were visualized by short exposure to iodine vapor or UV light. Column chromatography was carried out using Merck silica gel (100-200 mesh). All the glassware were pre-dried at 120 °C for at least 6 h and assembled while hot and cooled under stream of dry nitrogen gas. In all experiments, round bottom flasks of appropriate size were used.

**General procedure for the addition of  $\pi$ -nucleophiles to pyridine-2-carboxaldehyde condition A:**

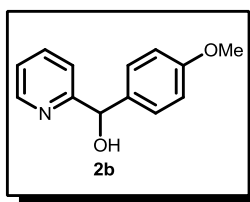
An oven dried two neck round bottom flask bearing septum in side arm and fitted with condenser was cooled to room temperature under a steady stream of nitrogen gas flow. The flask was charged with stirring bar,  $\text{AlBr}_3$  (266 mg, 1.0 mmol) and dry dichloromethane (3 mL) and cooled down to 0 °C (using ice). Then pyridine-2-carboxaldehyde (1 mmol) was added. The mixture was stirred for 30 minutes at 0 °C under nitrogen atmosphere. To this mixture was added dichloromethane (5 mL) solution of nucleophile (1.2 mmol) in drops. The resulting suspension was stirred at room temperature for 24 h. The reaction mixture was poured into aq.  $\text{NaHCO}_3$  and stirred for 5 min., organic layer was separated and the aqueous layer was extracted with dichloromethane (2 x 15 mL). The combined organic layer was washed with brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , filtered and concentrated on rotary evaporator under reduced pressure. The residue was purified through silica gel column chromatography using hexane/ethyl acetate as an eluent to afford the pure products.

### Reaction of anthracene (1a) with pyridine-2-carboxaldehyde<sup>3</sup> (Scheme 1)



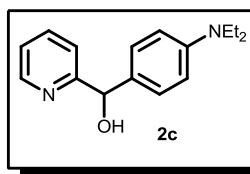
Reaction of anthracene (1a) with pyridine-2-carboxaldehyde gave 142 mg (49%) of anthracen-9-yl(pyridin-2-yl)methanol (2a) as yellow color solid; m.p. 89 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3350, 3097, 2978, 2838, 1584, 1441, 1148, 1350, 1097, 842;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.71 (dd,  $J = 3.6, 1.2$  Hz, 1H), 8.49 (s, 1H), 8.33-8.30 (m, 2H), 8.04-8.01 (m, 2H), 7.45-7.37 (m, 5H), 7.25 (s, 1H), 7.20-7.17 (m, 1H), 6.71 (dd,  $J = 8.0, 0.8$  Hz, 1H), 5.88 (br. s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.9, 147.7, 137.2, 132.3, 131.9, 130.8, 129.4, 129.1, 126.2, 124.9, 124.9, 122.3, 121.0, 69.8.

### Reaction of anisole (1b) with pyridine-2-carboxaldehyde<sup>4</sup> (Table 2)



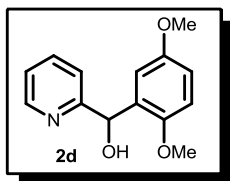
Reaction of anisole (1b) with pyridine-2-carboxaldehyde gave 175 mg, (81%) of (4-methoxyphenyl)(pyridine-2-yl)methanol (2b) as colorless solid; m.p. 134-136 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3265, 2976, 1603, 1517, 1427, 1328, 1256, 1183, 1043, 815, 763;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.54-8.53 (m, 1H), 7.60 (td,  $J = 8.0, 1.2$  Hz, 1H), 7.29-7.25 (m, 2H), 7.19-7.16 (m, 1H), 7.14 (d,  $J = 8.0$  Hz, 1H), 6.87-6.84 (m, 2H), 5.70 (s, 1H), 5.24 (br. s, 1H), 3.77 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.2, 159.2, 147.7, 136.8, 135.4, 128.3, 122.3, 121.2, 113.9, 74.5, 55.2.

### Reaction of *N,N*-diethylaniline (1c) with pyridine-2-carboxaldehyde (Table 2)



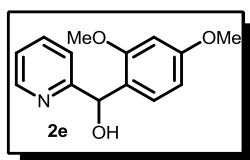
Reaction of *N,N*-diethylaniline (1c) with pyridine-2-carboxaldehyde gave 228 mg, (89%) of (4-(diethylamino)phenyl)(pyridine-2-yl)methanol (2c) as colorless solid; m.p. 91-92 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3211, 2971, 2880, 2675, 1609, 1518, 1470, 1264, 1144, 799;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.54 (d,  $J = 4.0$  Hz, 1H), 7.59 (t,  $J = 7.6$  Hz, 1H), 7.20-7.14 (m, 4H), 6.64 (d,  $J = 8.4$  Hz, 2H), 5.67 (s, 1H), 5.01 (br. s, 1H), 3.32 (q,  $J = 6.8$  Hz, 4H), 1.13 (t,  $J = 6.8$  Hz, 6H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.9, 147.7, 147.5, 136.7, 130.0, 128.4, 122.1, 121.4, 111.8, 74.9, 44.4, 12.6; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{16}\text{H}_{21}\text{N}_2\text{O}$  ( $\text{M}+\text{H}$ ): 257.1654, Found ( $\text{M}+\text{H}$ ): 257.1658.

### Reaction of 1,4-dimethoxybenzene (**1d**) with pyridine-2-carboxaldehyde (Table 2)



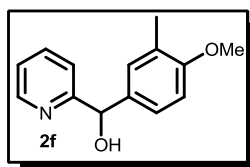
Reaction of 1,4-dimethoxybenzene (**1d**) with pyridine-2-carboxaldehyde gave 189 mg, (77%) of (2,5-dimethoxyphenyl)(pyridin-2-yl)methanol (**2d**) as pale yellow solid; m.p. 118 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3294, 1601, 1436, 1236, 998, 741, 543;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.52 (dt,  $J = 4.8, 1.2$  Hz, 1H), 7.58 (td,  $J = 8.0, 2.0$  Hz, 1H), 7.29-7.27 (m, 1H), 7.17-7.13 (m, 1H), 6.90 (d,  $J = 3.2$  Hz, 1H), 6.83 (d,  $J = 8.8$  Hz, 1H), 6.76 (dd,  $J = 8.8, 3.2$  Hz, 1H), 6.17 (s, 1H), 5.30 (br. s, 1H), 3.82 (s, 3H), 3.71 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.0, 154.0, 150.9, 147.7, 136.8, 132.9, 122.3, 121.3, 113.6, 113.4, 112.1, 69.0, 56.2, 55.7; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{14}\text{H}_{16}\text{NO}_3$  (M+H): 246.1130, Found (M+H): 246.1123.

### Reaction of 1,3-dimethoxybenzene (**1e**) with pyridine-2-carboxaldehyde<sup>5</sup>(Table 2)



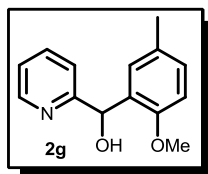
Reaction of 1,3-dimethoxybenzene (**1e**) with pyridine-2-carboxaldehyde gave 205 mg, (84%) of (2,4-Dimethoxyphenyl)(pyridine-2-yl)methanol (**2e**) as yellow oil; IR (KBr,  $\text{cm}^{-1}$ ): 3398, 3003, 2938, 2836, 1611, 1590, 1504, 1464, 1293, 1207, 1156, 1035, 796;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.60-8.58 (m, 1H), 7.66 (td,  $J = 8.0, 2.0$  Hz, 1H), 7.33 (dd,  $J = 8.0, 0.4$  Hz, 1H), 7.23-7.21 (m, 2H), 6.55-6.51 (m, 2H), 6.20 (s, 1H), 5.26 (br. s, 1H), 3.88 (s, 3H), 3.84 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.6, 160.3, 157.8, 147.7, 136.6, 128.6, 124.2, 122.0, 121.1, 104.6, 98.5, 69.1, 55.4, 55.3.

### Reaction of 2-methoxytoluene (**1f**) with pyridine-2-carboxaldehyde (Table 2)



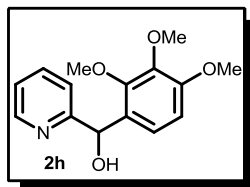
Reaction of 2-methoxytoluene (**1f**) with pyridine-2-carboxaldehyde gave 184 mg (80%) of (4-methoxy-3-methylphenyl)(pyridin-2-yl)methanol (**2f**) as colorless solid; m.p. 92-93 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3377, 3006, 2950, 1595, 1503, 1251, 1130, 1035;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.55 (d,  $J = 4.8$  Hz, 1H), 7.61 (td,  $J = 7.6, 2.0$  Hz, 1H), 7.19-7.14 (m, 3H), 7.11 (d,  $J = 2.0$  Hz, 1H), 6.77 (d,  $J = 8.4$  Hz, 1H), 5.68 (s, 1H), 5.21 (br. s, 1H), 3.80 (s, 3H), 2.18 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.3, 157.4, 147.7, 136.7, 134.9, 129.4, 126.8, 125.6, 122.2, 121.3, 109.7, 74.6, 55.3, 16.3; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{14}\text{H}_{15}\text{NO}_2$  (M+Na): 252.1000, Found (M+Na): 252.0999.

### Reaction of 4-methoxytoluene (**1g**) with pyridine-2-carboxaldehyde (Table 2)



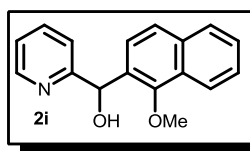
Reaction of 4-methoxytoluene (**1g**) with pyridine-2-carboxaldehyde gave 200 mg (87%) (2-methoxy-5-methylphenyl)(pyridine-2-yl)methanol (**2g**) as colorless solid; m.p. 116 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3401, 3005, 2941, 2836, 1592, 1500, 1247, 1037, 808;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.53 (ddd,  $J = 4.8, 1.6, 0.8$  Hz, 1H), 7.59 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.30-7.28 (m, 1H), 7.17-7.14 (m, 1H), 7.11 (d,  $J = 2.0$  Hz, 1H), 7.04-7.01 (m, 1H), 6.80 (d,  $J = 8.4$  Hz, 1H), 6.17 (d,  $J = 2.8$  Hz, 1H), 5.23 (d,  $J = 4.4$  Hz, 1H), 3.83 (s, 3H), 2.23 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.4, 154.6, 147.7, 136.7, 131.4, 130.2, 129.1, 128.3, 122.2, 121.3, 110.9, 69.2, 55.7, 20.6; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{14}\text{H}_{16}\text{NO}_2$  (M+H): 230.1181, Found (M+H): 230.1191

### Reaction of 1,2,3-trimethoxybenzene (**1h**) with pyridine-2-carboxaldehyde (Table 2)



Reaction of 1,2,3-trimethoxybenzene (**1h**) with pyridine-2-carboxaldehyde gave 220 mg (79%) of pyridin-2-yl-(2,3,4-trimethoxyphenyl)methanol (**2h**) as yellow oil; IR (KBr,  $\text{cm}^{-1}$ ): 3397, 3047, 3003, 2939, 2834, 1595, 1494, 1465, 1282, 1096, 1021, 755;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.54 (d,  $J = 4.8$  Hz, 1H), 7.61 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.28-7.25 (m, 1H), 7.18-7.15 (m, 1H), 6.95 (d,  $J = 8.8$  Hz, 1H), 6.63 (d,  $J = 8.8$  Hz, 1H), 6.01 (s, 1H), 5.22 (br. s, 1H), 3.86 (s, 3H), 3.82 (s, 3H), 3.81 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.4, 153.3, 151.5, 147.7, 142.0, 136.7, 129.3, 122.6, 122.1, 121.2, 107.4, 70.1, 61.0, 60.7, 55.9; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{15}\text{H}_{17}\text{NO}_4$  (M+H): 298.1055, Found (M+H): 298.1061.

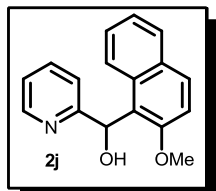
### Reaction of 1-methoxynaphthalene (**1i**) with pyridine-2-carboxaldehyde (Table 2)



Reaction of 1-methoxy naphthalene (**1i**) with pyridine-2-carboxaldehyde gave 195 mg (73%), of (1-methoxynaphthalen-2-yl)(pyridine-2-yl)methanol (**2i**) as colorless solid; m.p. 130 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3444, 3040, 2922, 2850, 1585, 1468, 1428, 1245, 1182, 1090, 841, 759;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.64 (ddd,  $J = 4.9, 1.5, 1.0$  Hz, 1H), 8.31-8.28 (m, 1H), 8.03-8.01 (m, 1H), 7.54 (td,  $J = 8.0, 2.0$  Hz, 1H), 7.46-7.42 (m, 2H), 7.36 (d,  $J = 8.0$  Hz, 1H), 7.21-7.18 (m, 1H), 7.05 (dd,  $J = 8.0, 0.8$  Hz, 1H), 6.77 (d,  $J = 8.0$  Hz, 1H), 6.30 (s, 1H), 5.30 (br. s, 1H),

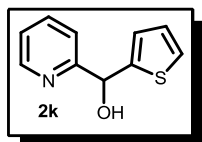
4.00 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.5, 155.8, 147.9, 136.9, 132.2, 130.2, 126.8, 126.7, 126.3, 125.0, 124.3, 122.6, 122.3, 121.4, 103.0, 73.8, 55.6; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{17}\text{H}_{15}\text{NO}_2$  ( $\text{M}+\text{Na}$ ): 288.1000, Found ( $\text{M}+\text{Na}$ ): 288.0990.

### Reaction of 2-methoxy naphthalene (1j) with pyridine-2-carboxaldehyde (Table 2)



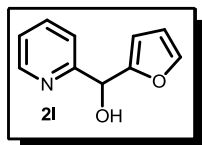
Reaction of 2-methoxy naphthalene (**1j**) with pyridine-2-carboxaldehyde gave 207 mg, (78%) of (2-methoxynaphthalen-1-yl)(pyridine-2-yl)methanol (**2j**) as colorless solid; m.p. 133 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3418, 3054, 2926, 2850, 1624, 1593, 1511, 1467, 1434, 1249, 1082, 1057, 1026, 810, 749;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.62 (dt,  $J = 4.8, 1.2$  Hz, 1H), 7.94-7.91 (m, 1H), 7.83 (d,  $J = 8.8$  Hz, 1H), 7.76-7.73 (m, 1H), 7.50 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.33-7.24 (m, 3H), 7.16-7.13 (m, 1H), 7.00 (dd,  $J = 8.0, 0.8$  Hz, 1H), 6.86 (s, 1H), 5.51 (br. s, 1H), 3.93 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  162.0, 155.7, 147.7, 136.8, 132.6, 130.6, 130.0, 128.6, 126.5, 124.8, 123.6, 122.9, 121.9, 120.8, 113.7, 67.9, 57.1; LRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{17}\text{H}_{16}\text{NO}_2$  ( $\text{M}+\text{H}$ ): 266.11 Found ( $\text{M}+\text{H}$ ): 265.93

### Reaction of thiophene (1k) with pyridine-2-carboxaldehyde (Table 2)



Reaction of thiophene (**1k**) with pyridine-2-carboxaldehyde gave 160 mg (84%) of pyridine-2-yl(thiophen-2-yl)methanol (**2k**) as yellow color solid; m.p. 69 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3145, 2864, 2702, 2360, 1593, 1480, 1433, 1253, 1154, 1102, 1049, 1002, 826, 707;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.58-8.56 (m, 1H), 7.66 (td,  $J = 7.6, 2.0$  Hz, 1H), 7.28 (d,  $J = 7.6$  Hz, 1H), 7.27-7.22 (m, 2H), 7.03-7.02 (m, 1H), 6.96 (dd  $J = 5.2, 3.6$  Hz, 1H), 6.02 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  160.1, 148.0, 147.4, 137.1, 126.7, 125.7, 125.2, 122.9, 121.3, 71.0. HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{10}\text{H}_9\text{NOS}$  ( $\text{M}+\text{H}$ ): 192.0483, Found ( $\text{M}+\text{H}$ ): 192.0477.

### Reaction of furan (1l) with pyridine-2-carboxaldehyde (Table 2)



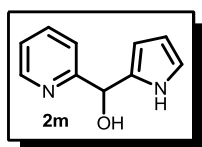
Reaction of furan (**1l**) with pyridine-2-carboxaldehyde gave 158 mg (88%) of furan-2-yl(pyridin-2-yl)methanol (**2l**) as brown color solid; m.p. 61 °C IR (KBr,  $\text{cm}^{-1}$ ): 3414, 2875, 1647, 1589, 1469, 1392, 1316, 1146, 1012, 748;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.58-8.56 (m, 1H), 7.68 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.36 (dd,  $J = 1.6,$

0.8 Hz, 1H), 7.29-7.23 (m, 2H), 6.32 (m, 1H), 6.26 (d,  $J = 3.2$  Hz, 1H), 5.80 (s, 1H), 5.05 (br. s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  158.1, 155.2, 148.1, 142.8, 137.0, 123.0, 121.4, 110.3, 107.7, 68.8. HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{10}\text{H}_9\text{NO}_2$  (M+H):175.0633, Found (M+H): 175.0621

### Reaction of pyrrole (1m) with pyridine-2-carboxaldehyde (Table 2)

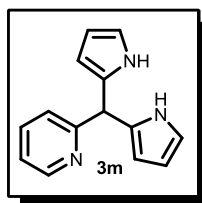
Reaction of pyrrole (1m) with pyridine-2-carboxaldehyde as mentioned in the general procedure gave 180 mg mixture of pyridin-2-yl(1H-pyrrol-2-yl)methanol (2m) and 2-(di(1H-pyrrol-2-yl)methyl)pyridine (3m) as black color solid. The reaction was performed at 0 °C. These two compounds were separated through silica gel column chromatography using hexane / ethyl acetate (80:20) mixture as eluent.

#### Pyridin-2-yl(1H-pyrrol-2-yl)methanol (2m)



118 mg (67% Yield) of as black color solid; m.p. 104 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3435, 3165, 3107, 2983, 1592, 1568, 1437, 1260, 1136, 1089, 841, 759, 724;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.71 (br. s, 1H), 8.55 (d,  $J = 4.8$  Hz, 1H), 7.68 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.30 (d,  $J = 7.6$  Hz, 1H), 7.24-7.21 (m, 1H), 6.75-6.73 (m, 1H), 6.13 (dd,  $J = 6.0, 2.8$  Hz, 1H), 6.10-6.09 (m, 1H), 5.83 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  159.9, 148.2, 137.2, 132.7, 122.9, 121.5, 118.4, 108.3, 106.8, 69.2.

#### 2-(Di(1H-pyrrol-2-yl)methyl)pyridine (3m)<sup>6</sup>



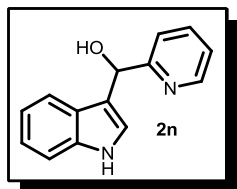
34 mg (24% Yield), colorless solid; m.p. 116 °C, (*Lit.*<sup>3</sup> m.p.: 115 °C); IR (KBr,  $\text{cm}^{-1}$ ): 3259, 3093, 2999, 1627, 1584, 1464, 1431, 1248, 1004, 755;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.89 (br. s, 2H), 8.60 (d,  $J = 4.0$  Hz, 1H), 7.66 (td,  $J = 7.6, 2.0$  Hz, 1H), 7.30 (dt,  $J = 7.6, 0.8$ , Hz, 1H), 7.18 (ddd,  $J = 6.4, 4.8, 0.8$  Hz, 1H), 6.71-6.69 (m, 2H), 6.12 (q,  $J = 2.8$  Hz, 2H), 5.93-5.92 (m, 2H), 5.51 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.3, 149.4, 137.3, 131.7, 123.4, 122.1, 117.5, 108.3, 106.5, 45.4.

### Reaction of indole (1n) with pyridine-2-carboxaldehyde (Table 2)

Reaction of indole (1n) with pyridine-2-carboxaldehyde as mentioned in the general procedure gave 225 mg mixture of pyridin-2-yl(1H-pyrrol-2-yl)methanol (2n) and 2-(di(1H-pyrrol-2-yl)methyl)pyridine (3n) as colorless solid. The reaction was performed at 0 °C. These two

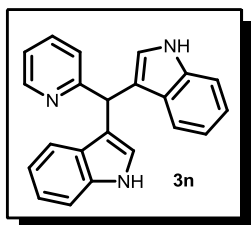
compounds were separated through silica gel column chromatography using hexane / ethyl acetate (55:45) mixture as eluent.

### (1*H*-Indol-3-yl)(pyridin-2-yl)methanol (**2n**)



Yield; 160 mg (71%), colorless solid; m.p. 160 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3290, 1596, 1436, 1350, 1236, 1104, 1000, 836, 745, 596;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.62 (dt,  $J = 4.8, 1.2$  Hz, 1H), 8.17 (br. s, 1H), 7.60 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.44 (dd,  $J = 8.0, 0.8$  Hz, 1H), 7.34-7.32 (m, 1H), 7.27-7.25 (m, 1H), 7.22-7.19 (m, 1H), 7.18-7.13 (m, 2H), 7.04-7.00 (m, 1H), 6.08 (s, 1H), 5.05 (br. s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.1, 147.7, 136.7, 125.7, 123.2, 122.3, 121.3, 119.8, 119.7, 118.3, 111.2, 69.1; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{14}\text{H}_{13}\text{N}_2\text{O}$  (M+H): 225.1028, Found (M+H): 225.1021.

### 2,2'-(Pyridin-2-ylmethylene)bis(1*H*-indole) (**3n**)



Yield; 55 mg (17%), colorless solid; m.p. 218 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3143, 2916, 2863, 1583, 1451, 1343, 1218, 1098, 1002, 744;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.61-8.59 (m, 1H), 8.02 (br. s, 2H), 7.60 (td,  $J = 7.6, 1.0$  Hz, 1H), 7.41 (d,  $J = 4.0$  Hz, 1H), 7.35-7.31 (m, 3H), 7.18-7.11 (m, 3H), 7.02 (td,  $J = 8.0, 1.2$  Hz, 2H), 6.78 (dd,  $J = 2.0, 0.8$  Hz, 2H), 6.06 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100MHz):  $\delta$  163.7, 149.6, 136.8, 127.1, 123.6, 122.1, 121.5, 119.9, 119.4, 118.4, 111.2, 43.4; LRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{22}\text{H}_{18}\text{N}_3$  (M+H): 324.15, Found (M+H): 324.07.

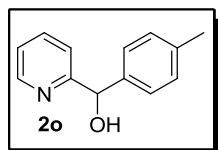
### General procedure for the addition of $\pi$ -nucleophiles to pyridine-2-carboxaldehyde condition B:

An oven dried two neck round bottom flask bearing septum in side arm and fitted with condenser was cooled to room temperature under a steady stream of nitrogen gas flow. The flask was charged with stirring bar,  $\text{AlBr}_3$  (266 mg, 1.0 mmol) and dry dichloromethane (3 mL) and cooled down to 0 °C (using ice). Then pyridine-2-carboxaldehyde (1 mmol) was added. The mixture was stirred for 30 minutes at 0 °C under nitrogen atmosphere. To this mixture was added the dichloromethane (5 mL) solution of nucleophiles (1.2 or 2.0 mmol) in drops. The resulting suspension was stirred at room temperature for 30 min followed by reflux for 24 h. After cooling to room temperature, the reaction mixture was poured into aq.  $\text{NaHCO}_3$  and stirred for 5



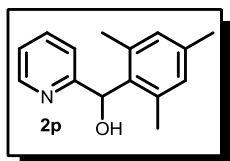
min. The organic layer was separated and the aqueous layer was extracted with dichloromethane (2 x 15 mL). The combined organic layer was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and concentrated on rotary evaporator under reduced pressure. The residue was purified through silica gel column chromatography using hexane/ethyl acetate as an eluent to afford the pure products.

### Reaction of toluene (**1o**) with pyridine-2-carboxaldehyde <sup>7</sup>(Table 3)



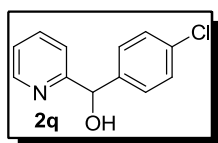
Reaction of toluene (**1o**) with pyridine-2-carboxaldehyde gave 105 mg (52%) of pyridin-2-yl(p-tolyl)methanol (**2o**) as oil; IR (KBr cm<sup>-1</sup>): 3473, 2888, 2844, 1591, 1570, 1491, 1470, 1438, 1113, 1055, 999, 770; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz): δ 8.61 (d, *J* = 4.8 Hz, 1H), 7.66 (td, *J* = 7.6, 1.6 Hz, 1H), 7.33-7.31 (m, 1H), 7.24-7.20 (m, 4H), 5.78 (s, 1H), 5.31(br, s, 1H), 2.39 (s, 3H); <sup>13</sup>C (CDCl<sub>3</sub>, 100 MHz): δ 161.2, 147.9, 140.4, 137.6, 136.9, 129.3, 127.1, 122.4, 121.4, 74.9, 21.2.

### Reaction of mesitylene (**1p**) with pyridine-2-carboxaldehyde (Table 3)



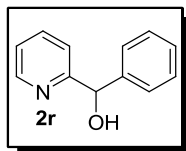
Reaction of mesitylene (**1p**) with pyridine-2-carboxaldehyde gave 140 mg (62%) of mesityl(pyridine-2-yl)methanol (**2p**) as yellow color solid; m.p. 58 °C; IR (KBr, cm<sup>-1</sup>): 3399, 3007, 2918, 2864, 1591, 1467, 1434, 1202, 1145, 1044, 851, 753; <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz): δ 8.58 (dt, *J* = 4.8, 1.2 Hz, 1H), 7.56 (td, *J* = 7.6, 1.6 Hz, 1H), 7.20-7.17 (m, 1H), 6.91-6.88 (m, 1H), 6.83 (s, 2H), 6.19 (s, 1H), 5.40 (br. s, 1H), 2.26 (s, 3H), 2.18 (s, 6H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz): δ 161.3, 147.5, 137.6, 137.5, 137.0, 135.1, 130.1, 122.0, 120.5, 70.5, 21.0, 20.5; HRMS-ESI (*m/z*): Calculated for C<sub>15</sub>H<sub>17</sub>NO (M+Na): 250.1208, Found (M+Na): 250.1197.

### Reaction of chlorobenzene (**1q**) with pyridine-2-carboxaldehyde <sup>7</sup>(Table 3)



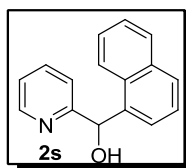
Reaction of chlorobenzene (**1q**) with pyridine-2-carboxaldehyde gave 95 mg (43%) of (4-chlorophenyl)(pyridin-2-yl)methanol (**2q**) as white solid; m.p. 80 °C; IR (KBr cm<sup>-1</sup>): 3469, 2884, 1591, 1570, 1491, 1090, 1055, 812, 770; <sup>1</sup>H (CDCl<sub>3</sub>, 400 MHz): δ 8.55 (d, *J* = 4.4 Hz, 1H), 7.62 (td, *J* = 8.0, 1.6 Hz, 1H), 7.32-7.27 (m, 4H), 7.21-7.19 (m, 1H), 7.12 (d, *J* = 8.0 Hz, 1H), 5.71 (s, 1H), 5.37 (br, s, 1H); <sup>13</sup>C (CDCl<sub>3</sub>, 100 MHz): δ 160.5, 148.0, 141.8, 137.1, 133.7, 128.8, 128.5, 122.7, 121.3, 74.4.

### Reaction of benzene (**1r**) with pyridine-2-carboxaldehyde <sup>7</sup>(Table 3)



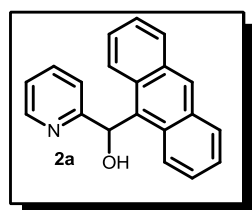
Reaction of benzene (**1r**) with pyridine-2-carboxaldehyde gave 70 mg (37%) of (phenyl(pyridin-2-yl)methanol (**2r**) as oil; IR (KBr  $\text{cm}^{-1}$ ): 3178, 3079, 2875, 1592, 1451, 1432, 1148, 1052, 1001, 761, 749, 698;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz) :  $\delta$  8.56 (dt,  $J = 4.8, 1.6$  Hz, 1H ), 7.63 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.39-7.31 (m, 4H), 7.29-7.25 (m, 1H), 7.21-7.19 (m, 1H), 7.17-7.14 (m, 1H), 5.75 (s, 1H), 5.28 (br, s, 1H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.1, 147.9, 143.3, 136.9, 128.6, 127.9, 127.1, 122.5, 121.4, 75.1.

### Reaction of naphthalene (**1s**) with pyridine-2-carboxaldehyde (Table 3)<sup>8</sup>



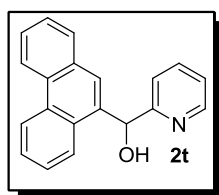
Reaction of naphthalene (**1s**) with pyridine-2-carboxaldehyde gave 120 mg (51%) of naphthalen-2-yl(pyridine-2-yl)methanol **2s** as colorless solid; m.p. 87 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3379, 3052, 2920, 2851, 1592, 1435, 1396, 1051, 798, 778, 751;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz) :  $\delta$  8.63 (d,  $J = 4.8$  Hz, 1H), 8.13-8.10 (m, 1H), 7.87-7.81 (m, 2H), 7.55-7.50 (m, 2H), 7.47-7.41 (m, 3H), 7.20-7.18 (m, 1H), 7.05 (d,  $J = 8.0$  Hz, 1H), 6.43 (s, 1H), 5.47 (br, s, 1H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.0, 147.9, 138.2, 136.9, 134.3, 131.9, 128.9, 128.8, 126.3, 126.2, 125.7, 125.4, 124.5, 122.5, 121.4, 73.6.

### Reaction of anthracene (**1a**) with pyridine-2-carboxaldehyde<sup>3</sup> (Table 3)



Reaction of anthracene (**1a**) with pyridine-2-carboxaldehyde gave 179 mg (61%) of anthracen-9-yl(pyridin-2-yl)methanol (**2a**) as yellow color solid; m.p. 89 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3350, 3097, 2978, 2838, 1584, 1441, 1148, 1350, 1097, 842;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.71 (dd,  $J = 3.6, 1.2$  Hz, 1H), 8.49 (s, 1H), 8.33-8.30 (m, 2H), 8.04-8.01 (m, 2H), 7.45-7.37 (m, 5H), 7.25 (s, 1H), 7.20-7.17 (m, 1H), 6.71 (dd,  $J = 8.0, 0.8$  Hz, 1H), 5.88 (br. s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.9, 147.7, 137.2, 132.3, 131.9, 130.8, 129.4, 129.1, 126.2, 124.9, 124.9, 122.3, 121.0, 69.8.

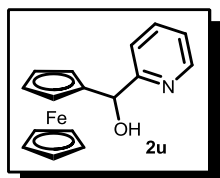
### Reaction of phenanthrene (**1t**) with pyridine-2-carboxaldehyde<sup>3</sup> (Table 3)



Reaction of phenanthrene (**1t**) with pyridine-2-carboxaldehyde gave 118 mg (41%) of phenanthren-9-yl(pyridin-2-yl)methanol (**2t**) as yellow color solid; m.p. 78 °C; IR (KBr  $\text{cm}^{-1}$ ): 3458, 3076, 2844, 2695, 1597, 1440, 1073, 1002,

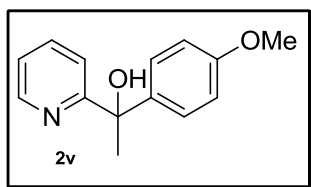
788, 743, 721;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.72 (d,  $J = 8.4$  Hz, 1H), 8.68-8.66 (m, 2H), 8.12 (d,  $J = 8.0$  Hz, 1H), 7.87 (d,  $J = 7.6$  Hz, 1H), 7.81 (s, 1H), 7.67-7.57 (m, 3H), 7.55-7.49 (m, 2H), 7.23-7.20 (m, 1H), 7.11 (d,  $J = 8.0$  Hz, 1H), 6.43 (s, 1H), 5.43 (br, s, 1H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  160.9, 148.1, 137.0, 136.2, 131.4, 131.3, 130.7, 130.2, 129.0, 127.6, 127.1, 126.8, 126.7, 126.4, 125.5, 123.2, 122.68, 122.63, 121.5, 74.5.

### Reaction of ferrocene (**1u**) with pyridine-2-carboxaldehyde (Table 3)



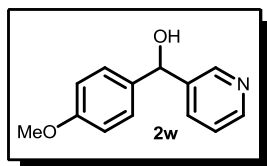
Reaction of ferrocene (**1u**) with pyridine-2-carboxaldehyde gave 120 mg (40%) of (1-ferrocenyl)(2-pyridyl)methanol (**2u**) as yellow color solid; m.p. 98 °C; IR (KBr  $\text{cm}^{-1}$ ): 3434, 3083, 2890, 2835, 1592, 1475, 1432, 1102, 1074, 999;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.55 (d,  $J = 4.8$  Hz, 1H), 7.66 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.34 (d,  $J = 7.6$  Hz, 1H), 7.20-7.17 (m, 1H), 5.49 (s, 1H), 4.27-4.26 (m, 1H), 4.19 (s, 5H), 4.16-4.13 (m, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.1, 148.1, 136.5, 122.4, 120.7, 92.4, 71.4, 68.6, 68.0, 67.9, 67.2, 65.8; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{16}\text{H}_{16}\text{NOFe}$  ( $\text{M}+\text{H}$ ): 294.0581, Found ( $\text{M}+\text{H}$ ): 294.0588.

### Reaction of anisole (**1b**) with 2-acetyl pyridine (Scheme 2)<sup>9</sup>



Reaction of anisole (**1b**) with 2-acetyl pyridine as mentioned in the general procedure (condition **B**) gave 108 mg (47%) of 1-(4-methoxyphenyl)-1-(pyridin-2-yl)methanol (**2v**) as colorless solid. This compound was separated though silica gel column chromatography using hexane / ethyl acetate (80:20) mixture as eluent; m.p. 67-68 °C; IR (KBr  $\text{cm}^{-1}$ ): 3431, 2973, 2932, 1609, 1589, 1510, 1367, 1249, 1178, 1031;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.51 (dq,  $J = 4.8, 1.6, 0.8$  Hz, 1H), 7.63 (td,  $J = 8.0, 2.0$  Hz, 1H), 7.39-7.36 (m, 2H), 7.26-7.24 (m, 1H), 7.18-7.15 (m, 1H), 6.85-6.81 (m, 2H), 3.76 (s, 3H), 1.90 (s, 3H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  165.1, 158.6, 147.4, 139.4, 137.0, 127.2, 122.0, 120.3, 113.6, 74.8, 55.3, 29.4.

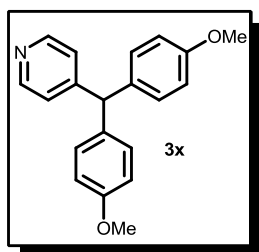
### Reaction of anisole (**1b**) with pyridine-3-carboxaldehyde (Figure 2)



Reaction of anisole (**1b**) with pyridine-3-carboxaldehyde as mentioned in the general procedure (condition **A**) gave 70 mg (31%) of (4-methoxyphenyl)(pyridine-3-yl)methanol (**2w**) as colorless solid. This

compound was separated through silica gel column chromatography using hexane / ethyl acetate (80:20) mixture as eluent; m.p. 101 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3201, 2994, 2832, 1586, 1511, 1251, 1173, 1029, 808, 715;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.50 (d,  $J = 2.4$  Hz, 1H), 8.38 (dd,  $J = 4.8$ , 1.6 Hz, 1H), 7.70-7.67 (m, 1H), 7.27-7.23 (m, 2H), 7.22-7.20 (m, 1H), 6.87-6.85 (m, 2H), 5.79 (s, 1H), 3.78 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  159.3, 148.4, 148.1, 139.9, 135.6, 134.3, 128.0, 123.5, 114.1, 73.6, 55.4; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{13}\text{H}_{14}\text{NO}_2$  (M+H): 216.1025, Found (M+H): 216.1016.

### Reaction of anisole (1b) with pyridine-4-carboxaldehyde (Figure 2)



Reaction of anisole (**1b**) with pyridine-4-carboxaldehyde as mentioned in the general procedure (condition A) gave 272 mg (89%) of 4-(bis(4-methoxyphenyl)methyl)pyridine (**3x**) as yellow solid; m.p. 117 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3069, 2953, 2834, 1610, 1510, 1412, 1302, 1249, 1181, 1032, 817;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.50-8.48 (m, 2H) 7.03-7.02 (m, 2H), 7.00-6.98 (m, 4H), 6.85-6.82 (m, 4H), 5.39 (s, 1H), 3.78 (s, 6H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  158.4, 153.7, 149.8, 134.7, 130.3, 124.6, 114.0, 55.4, 54.7; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{20}\text{H}_{20}\text{NO}_2$  (M+H): 306.1494, Found (M+H): 306.1483.

### General procedure for the addition of anisole to electron deficient aldehydes condition C:

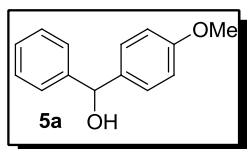
An oven dried two neck round bottom flask bearing septum in side arm and fitted with condenser was cooled to room temperature under a steady stream of nitrogen gas flow. The flask was charged with stirring bar,  $\text{AlBr}_3$  (266 mg, 1.0 mmol) and dry dichloromethane (3 mL) and cooled down to 0 °C (using ice bath). Then electron deficient aldehydes (1 mmol) in dry dichloromethane (5 mL) at 0 °C with stirring was added followed by the addition of dichloromethane solution of pyridine (0.016 mL, 2mmol). Stirring was continued for 30 minutes. To this mixture was added the dichloromethane (5 mL) solution of anisole (1.2 mmol) in drops. The resulting suspension was stirred at room temperature for 30 min followed by reflux for 24 h. After cooling to room temperature, the reaction mixture was poured into aq.  $\text{NaHCO}_3$  and stirred for 5 min. The organic layer was separated and the aqueous layer was extracted with dichloromethane (2 x 15 mL). The combined organic layer was dried over anhydrous  $\text{Na}_2\text{SO}_4$ , filtered and concentrated on rotary evaporator under reduced pressure. The

residue was purified through silica gel column chromatography using hexane/ethyl acetate as an eluent to afford the pure products.

### Reaction of anisole (**1b**) with benzaldehyde (Table 4)

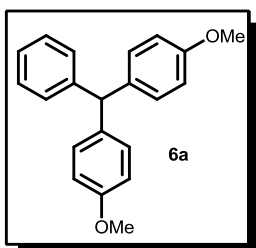
Reaction of anisole (**1b**) with benzaldehyde as mentioned in the general procedure gave 180 mg mixture of (4-methoxyphenyl)(phenyl)methanol (**5a**) and 4,4'-(phenylmethylene)-bis(methoxybenzene) (**6a**) as yellow color solid. The reaction was performed at 0 °C-room temperature. These two compounds were separated through silica gel column chromatography using hexane / ethyl acetate (90:10) mixture as eluent.

#### (4-methoxyphenyl)(phenyl)methanol (**5a**)<sup>10</sup>



125 mg (58% Yield) of as colorless solid; m.p. 104 °C; IR (KBr  $\text{cm}^{-1}$ ): 3409, 3006, 2950, 2834, 1610, 1586, 1515, 1255, 1175, 1032, 809, 726;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.35-7.22 (m, 7H), 6.83 (d,  $J = 8.8$  Hz, 1H), 5.76 (s, 1H), 3.75 (s, 3H), 2.30 (br, s, 1H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  159.1, 144.1, 136.3, 128.5, 128.0, 127.5, 126.5, 114.0, 75.9, 55.4.

#### 4,4'-(phenylmethylene)bis-(methoxybenzene) (**6a**)



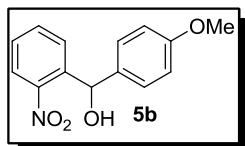
43 mg (14% yield) as colorless solid, m.p. 79 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3015, 2945, 2835, 1601, 1503, 1446, 1241, 1184, 1025, 816;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.30-7.25 (m, 2H), 7.22-7.17 (m, 1H), 7.12-7.09 (m, 2H), 7.03-7.01 (m, 4H), 6.84-6.80 (m, 4H), 5.45 (s, 1H), 3.78 (s, 6H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  157.9, 144.5, 136.4, 130.2, 129.2, 128.2, 126.1, 113.6, 55.2, 55.1; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{21}\text{H}_{20}\text{O}_2$  (M+K): 343.1100, Found (M+K): 343.1103.

### Reaction of anisole (**1b**) with *o*-nitrobenzaldehyde (Table 4)<sup>11</sup>

Reaction of anisole (**1b**) with *o*-nitrobenzaldehyde as mentioned in the general procedure gave 164 mg mixture of (4-methoxyphenyl)(2-nitrophenyl)methanol (**5b**) and 4,4'-((2-nitrophenyl)methylene)bis(methoxybenzene) (**6b**) as yellow color solid. The reaction was

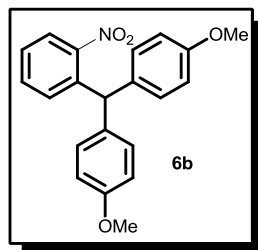
performed at 0 °C-room temperature. These two compounds were separated though silica gel column chromatography using hexane / ethyl acetate (90:10) mixture as eluent.

#### (4-methoxyphenyl)(2-nitrophenyl)methanol (**5b**)



110 mg, (42% yield) (**5b**) as yellow oil; IR (KBr,  $\text{cm}^{-1}$ ): 3432, 2935, 2837, 1609, 1529, 1349, 1249, 1175, 1029, 733;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.91 (dd,  $J = 8.0, 1.2$  Hz, 1H), 7.79 (dd,  $J = 8.0, 1.2$  Hz, 1H), 7.64 (td,  $J = 8.0, 1.2$  Hz, 1H), 7.46-7.42 (m, 1H), 7.24-7.21 (m, 2H), 6.86-6.84 (m, 2H), 6.40 (s, 1H), 3.78 (s, 3H), 2.69 (br, s, 1H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  159.4, 148.3, 138.8, 133.9, 133.4, 129.1, 128.4, 124.7, 114.0, 71.2, 55.3.

#### 4,4'-((2-nitrophenyl)methylene)bis(methoxybenzene) (**6b**)

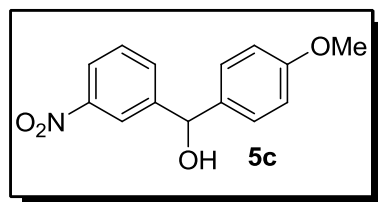


46 mg, (13% yield) (**6b**) as yellow solid; m.p. 114°C; IR (KBr,  $\text{cm}^{-1}$ ): 3005, 2952, 2837, 1602, 1520, 1462, 1360, 1245, 1032, 751;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.83 (dd,  $J = 8.0, 1.2$  Hz, 1H), 7.47 (td,  $J = 8.0, 1.2$  Hz, 1H), 7.36 (td,  $J = 8.0, 1.2$  Hz, 1H), 7.03 (dd,  $J = 8.0, 1.2$  Hz, 1H), 6.97-6.95 (m, 4H), 6.83-6.81 (m, 4H), 6.16 (s, 1H), 3.78 (s, 6H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  158.4, 139.0, 134.5, 132.4, 131.9, 130.4, 127.4, 124.7, 114.0, 55.3, 49.8; LRMS- ESI ( $m/z$ ): Calculated for  $\text{C}_{21}\text{H}_{19}\text{NO}_4$  (M+Na): 372.1212, Found (M+Na): 372.1217.

#### Reaction of anisole (**1b**) with *m*-nitrobenzaldehyde (Table 4)<sup>12</sup>

Reaction of anisole (**1b**) with *m*-nitrobenzaldehyde as mentioned in the general procedure gave 176 mg mixture of (4-methoxyphenyl)(3-nitrophenyl)methanol (**5c**) and 4,4'-((3-nitrophenyl)methylene)bis(methoxybenzene) (**6c**) as yellow color solid. The reaction was performed at 0 °C-room temperature. These two compounds were separated though silica gel column chromatography using hexane / ethyl acetate (90:10) mixture as eluent.

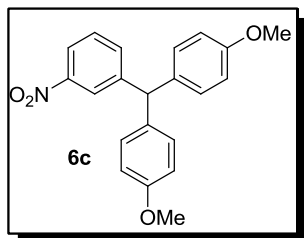
#### (4-methoxyphenyl)(3-nitrophenyl)methanol (**5c**)



115 mg, (42% yield) (**5c**) as yellow solid; m.p. 59 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3344, 3109, 3020, 2965, 2894, 1606, 1513, 1348, 1249, 1175, 1037, 798;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.27 (t,  $J = 2.0$

Hz, 1H), 8.10 (ddd,  $J = 8.0, 2.0, 0.8$  Hz, 1H), 7.71-7.68 (m, 1H), 7.48 (t,  $J = 8.0$  Hz, 1H), 7.27-7.25 (m, 2H), 6.90-6.86 (m, 2H), 5.87 (d,  $J = 2.8$  Hz, 1H), 3.79 (s, 3H), 2.42 (d,  $J = 3.2$  Hz, 1H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  159.5, 148.3, 146.0, 135.0, 132.3, 129.2, 128.0, 122.2, 121.2, 114.2, 74.9, 55.3.

#### 4,4'-((3-nitrophenyl)methylene)bis(methoxybenzene) (**6c**)

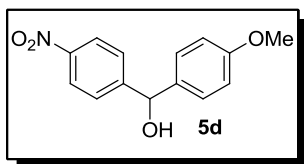


60 mg, (17% yield) (**6c**) as yellow solid ; m.p. 110 °C; IR (KBr  $\text{cm}^{-1}$ ): 3002, 2956, 2835, 1609, 1529, 1509, 1461, 1350, 1248, 1178, 1035, 842;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.06-8.05 (m, 1H), 7.98-7.97 (m, 1H), 7.46-7.41 (m, 2H), 7.02-6.98 (m, 4H), 6.86-6.83 (m, 4H), 5.54 (s, 1H), 3.79(s, 6H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  158.3, 148.3, 146.9, 135.4, 134.8, 130.1, 129.1, 124.0, 121.4, 114.0, 55.2, 54.8; HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{21}\text{H}_{19}\text{NO}_4$  ( $\text{M}+\text{Na}$ ): 372.1212, Found ( $\text{M}+\text{Na}$ ): 372.1219.

#### Reaction of anisole (**1b**) with *p*-nitrobenzaldehyde (Table 4)<sup>13</sup>

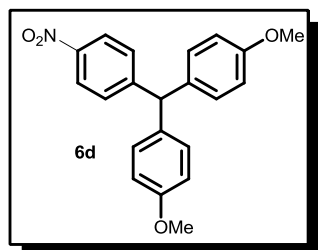
Reaction of anisole (**1b**) with *p*-nitrobenzaldehyde as mentioned in the general procedure gave 208 mg mixture of (4-methoxyphenyl)(4-nitrophenyl)methanol (**5d**) and 4,4'-((4-nitrophenyl)methylene)bis(methoxybenzene) (**6d**) as yellow color solid. The reaction was performed at 0 °C-room temperature. These two compounds were separated though silica gel column chromatography using hexane / ethyl acetate (90:10) mixture as eluent.

#### (4-methoxyphenyl)(4-nitrophenyl)methanol (**5d**)



125 mg, (48% yield) as yellow solid m.p. 55 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3504, 3093, 2933, 2841, 1609, 1526, 1348, 1237, 1040, 1020, 732;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz) :  $\delta$  8.16-8.13 (m, 2H), 7.55-7.51 (m, 2H), 7.25-7.20 (m, 2H), 6.87-6.84 (m, 2H), 5.83 (s, 1H), 3.77 (s, 3H), 2.66 (br, s, 1H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  159.6, 151.2, 147.1, 135.1, 128.2, 127.0, 123.6, 114.3, 75.1, 55.4.

#### 4,4'-((4-nitrophenyl)methylene)bis(methoxybenzene) (**6d**)



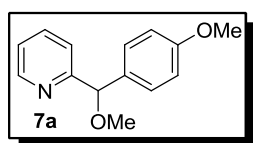
75 mg (21 %) (**6d**) as yellow solid; m.p.118 °C; IR (KBr,  $\text{cm}^{-1}$ ): 3001, 2938, 2837, 1603, 1514, 1345, 1298, 1249, 1179, 1030, 816;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.14-8.12 (m, 2H), 7.28-7.25 (m,

2H), 7.00-6.97 (m, 4H), 6.86-6.83 (m, 4H), 5.53 (s, 1H), 3.79 (s, 6H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  158.5, 152.5, 146.5, 134.9, 130.3, 130.2, 123.6, 114.1, 55.4, 55.1. HRMS-ESI ( $m/z$ ): Calculated for  $\text{C}_{21}\text{H}_{19}\text{NO}_4$  ( $\text{M}+\text{Na}$ ): 372.1212, Found ( $\text{M}+\text{Na}$ ): 372.1211.

### General procedure for the formation of ethers by quenching with nucleophiles: (Scheme 3)

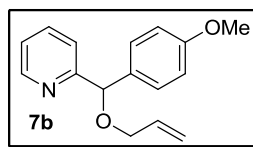
An oven dried two neck round bottom flask bearing septum in side arm and fitted with condenser was cooled to room temperature under a steady stream of nitrogen gas flow. The flask was charged with stirring bar,  $\text{AlBr}_3$  (266 mg, 1.0 mmol) and dry dichloromethane (3 mL) and cooled down to 0 °C (using ice bath). Dichloromethane solution of pyridine-2-carboxaldehyde (1 mmol) was added and stirred for 30 minutes at 0 °C under nitrogen atmosphere. To this mixture was added dichloromethane (5 mL) solution of anisole (1.2 mmol) in drops. The resulting suspension was stirred at room temperature for 24 h. The reaction mixture was quenched with nucleophiles (methanol, allyl alcohol or thiophenol) and stirred for 12 h – 24 h. The organic layer was separated and the aqueous layer was extracted with dichloromethane (2 x 15 mL). The combined organic layer was washed with brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , filtered and concentrated on rotary evaporator under reduced pressure. The residue was purified through silica gel column chromatography using hexane/ethyl acetate as an eluent to afford the pure products.

### 2-(methoxy(4-methoxyphenyl)methyl)pyridine (7a) (Scheme 3)<sup>14</sup>



185 mg, (80% yield) (**7a**) as colorless oil; IR (KBr,  $\text{cm}^{-1}$ ): 3059, 3003, 2933, 2903, 2824, 1610, 1588, 1511, 1466, 1436, 1247, 1174, 1095, 1033, 974, 820, 754;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz) :  $\delta$  8.53-8.52 (m, 1H), 7.66 (td,  $J = 8.0, 1.6$  Hz, 1H), 7.48 (d,  $J = 8.0$  Hz, 1H), 7.35-7.31 (m, 2H), 7.15-7.11 (m, 1H), 6.87-6.84 (m, 2H), 5.32 (s, 1H), 3.76 (s, 3H), 3.40 (s, 3H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  161.8, 159.2, 149.1, 136.8, 133.0, 128.3, 122.3, 120.5, 113.9, 86.1, 57.1, 55.3.

### 2-((allyloxy)(4-methoxyphenyl)methyl)pyridine (7b) (Scheme 3)

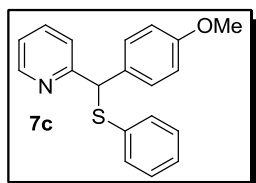


150 mg, (59% yield) (**7b**) as colorless oil; IR (KBr,  $\text{cm}^{-1}$ ): 3070, 3007, 2931, 2836, 1609, 1589, 1511, 1466, 1434, 1247, 1175, 753;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz) :  $\delta$  8.52 (ddd,  $J = 4.8, 1.6, 0.8$  Hz, 1H), 7.67 (td,  $J = 7.6, 2.0$



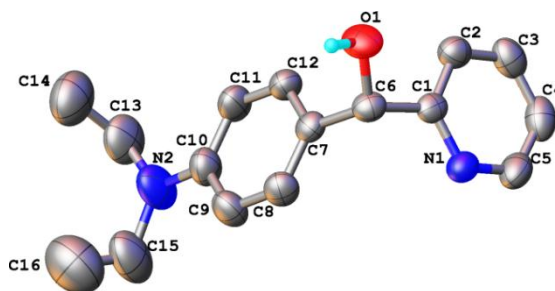
Hz, 1H), 7.55 (d,  $J = 7.6$  Hz, 1H), 7.36-7.31 (m, 2H), 7.12 (ddd,  $J = 7.2, 4.8, 1.2$  Hz, 1H), 6.86-6.84 (m, 2H), 6.01-5.92 (m, 1H), 5.50 (s, 1H), 5.31 (dq,  $J = 17.2, 1.6$  Hz, 1H), 5.19 (dq,  $J = 10.4, 1.6$  Hz, 1H), 4.09-3.99 (m, 2H), 3.76 (s, 3H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  162.0, 159.2, 149.0, 136.8, 134.6, 133.2, 128.4, 122.2, 120.5, 117.0, 113.9, 83.5, 69.8, 55.2.

### 2-((4-methoxyphenyl)(phenylthio)methyl)pyridine (**7c**) (Scheme 3)<sup>15</sup>

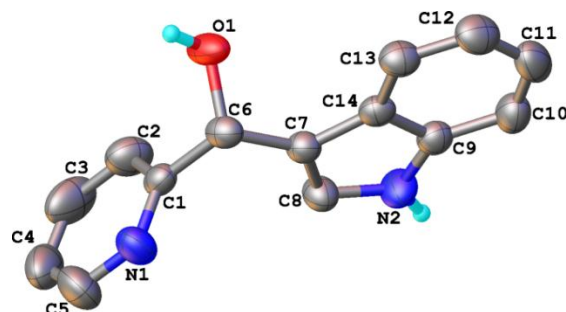


132 mg, (43% yield) (**7c**) as yellow oil; IR (KBr,  $\text{cm}^{-1}$ ): 3057, 3002, 2927, 2835, 1607, 1585, 1509, 1465, 1434, 1251, 1176, 1031, 744;  $^1\text{H}$  ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  8.55 (d,  $J = 4.4$  Hz, 1H), 7.59 (td,  $J = 7.6, 1.6$  Hz, 1H), 7.46 (d,  $J = 8.0$  Hz, 1H), 7.36 (d,  $J = 8.8$  Hz, 2H), 7.28-7.24 (m, 2H), 7.18-7.06 (m, 4H), 6.84-6.80 (m, 2H), 5.62 (s, 1H), 3.75 (s, 3H);  $^{13}\text{C}$  ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  160.6, 159.0, 149.4, 136.9, 135.7, 132.1, 131.0, 129.6, 128.8, 126.8, 122.6, 122.1, 114.1, 58.5, 55.3.

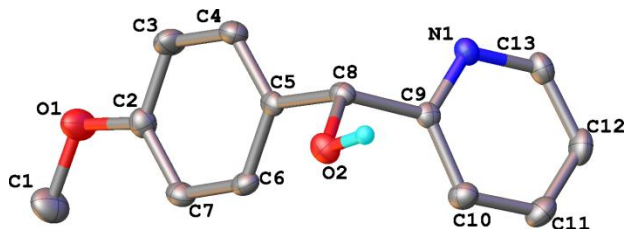
### Crystal Structure of the compounds **2c**, **2b**, **2n** and **6a**<sup>16</sup>



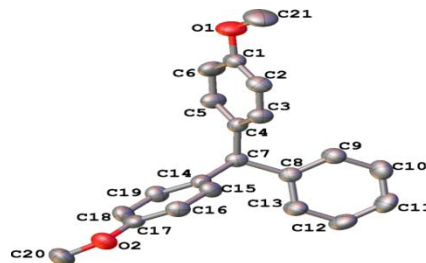
**2c**, CCDC 847587



**2n**, CCDC 847590



**2b**, CCDC 847589



**6a**, CCDC 847592

ORTEPs have been drawn at 50% probability level of the ellipsoids. Caption “Thermal ellipsoid plots at 50 % probability level only hydrogen bonded hydrogen atoms have been shown for clarity.”

## References:

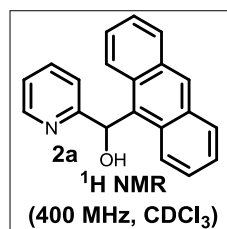
1. A. Barbara, N. Stoochnoff and L. Benoiton, *Tetrahedron Lett.*, 1973, **1**, 21.
2. W. L. F. Armarego and C. L. L. Chai, *Purification of Laboratory Chemicals*, 6<sup>th</sup> ed.; Elsevier, UK, 2009.
3. A. Solladie-Cavallo, C. Marsol, K. Azyat, A. Klein, M. Roje, C. Welch, J. Chilenski, P. Taillasson and H. D'Orchymont, *Eur. J. Org. Chem.*, 2007, **53**, 826.
4. (a) B. Agai, A. Proszenyak, G. Tarkanyi, L. Vida and F. Faig, *Eur. J. Org. Chem.*, 2004, 3623; (b) F. Shibahara, R. Sugiura, E. Yamaguchi, A. Kitagawa and T. Murai, *J. Org. Chem.*, 2009, **74**, 3566.
5. A. S. Gothelf, T. Hansen and K. A. Jorgensen, *J. Chem. Soc., Perkin. Trans. 1*, 2001, 854.
6. L. Zoli and P. Cozzi, *ChemSusChem*, 2009, **2**, 218.
7. (a) M. Froimowitz, Y. Gu, L. A. Dakin, P. M. Nagafuji, C. J. Kelley, D. Parrish, J. R. Deschamps and A. Janowsky, *J. Med. Chem.*, 2006, **50**, 219; (b) A. Doudouh, C. Woltermann and P. C. Gos, *J. Org. Chem.*, 2007, **72**, 4978.
8. A. Solladie-Cavallo, C. Marsol, K. Azyat, M. Yaakoup, K. Azyat, A. Klein, M. Roje, C. Suteu, T. B. Freedman, X. Cao and L. A. Naite, *J. Org. Chem.*, 2003, **68**, 7308.
9. X. Wang, M. Zak, M. Maddess, P. O'Shea, R. Tillyer, E. J. J. Grabowski and P. J. Reider, *Tetrahedron Lett.*, 2000, **41**, 4865.
10. (a) S.K. Khim, M. Dai, X. Zhang, L. Chen, L. Pettus, K. Thakkar and A. G. Schultz, *J. Org. Chem.*, 2004, **69**, 7728; (b) A. Fürstner and H. Krause, *Adv. Synth. Catal.*, 2001, **343**, 343.
11. P. J. Serafinowski and P. B. Garland, *J. Am. Chem. Soc.*, 2003, **125**, 962.
12. J. Xuefeng, F. Ling, L. Aijun, P. Yi and Z. Chengjian, *Synlett*, 2009, **3**, 495.
13. (a) G. E. Job, A. Shvets, W. H. Pirkle, S. Kuwahara, M. Kosaka, Y. Kasai, H. Taji, K. Fujita, M. Watanabe and N. Harada, *J. Chrom. A* 2004, **1055**, 41; (b) C. M. Qin, H. Y. Wu, J. Cheng, X. A. Chen, M. C. Liu, W. W. Zhang, W. K. Su and J. C. Ding, *J. Org. Chem.*, 2007, **72**, 4102.
14. T. Kuroda, M. Takahashi, T. Ogiku, H. Ohmizu, T. Nishitani, K. Kondo and T. Iwasaki, *J. Org. Chem.*, 1994, **59**, 7353.

15. S. Kumar, S. K. Das, S. Dey, P. Maity, M. Guha, V. Choubey, G. Panda and U. Bandyopadhyay, *Antimicrob. Agents Chemother.*, 2008, **52**, 705.
16. (a) CrysAlisPro, Version 1.171.33.66; Oxford Diffraction Ltd.:Abingdon, U.K., 2010. (b) G. M. Sheldrick SHELXS-97, *Programs for the Solution of Crystal Structures*; University of Gottingen: Germany, 1997. (c) G. M. Sheldrick XL, *Acta Crystallogr.* **2008**, *A64*, 112–122. (d) O. V. Dolomanov, L. J. Bourhis, R. J Gildea, J. A. K. Howard and H. Puschmann, OLEX2: A complete structure solution, refinement and analysis program, *J. Appl. Cryst.*, **2009**, *42*, 339–341.
17. CCDC & X-ray crystallography details for compounds 2b, 2c, 2n, and 6a, CCDC reference numbers 847587, 847589, 847590, 847592 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

<b>Crystal data:</b>	<b>2b</b>	<b>2c</b>	<b>2n</b>	<b>6a</b>
Empirical formula	C <sub>12</sub> H <sub>12</sub> NO <sub>2</sub>	C <sub>16</sub> H <sub>20</sub> N <sub>2</sub> O	C <sub>14</sub> H <sub>12</sub> N <sub>2</sub> O	C <sub>21</sub> H <sub>20</sub> O <sub>2</sub>
Formula weight	215.24	256.34	224.26	304.37
Temperature/K	150.0	298	298	298
Crystal system	monoclinic	monoclinic	orthorhombic	orthorhombic
Space group	P2 <sub>1</sub> /c	P2 <sub>1</sub> /c	Pca2 <sub>1</sub>	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
a/Å	8.4890(3)	11.7951(6)	10.6147(5)	5.9087(2)
b/Å	17.2434(6)	9.4577(5)	11.5927(5)	12.7700(5)
c/Å	8.1339(3)	12.8525(7)	9.2553(4)	21.8373(7)
α/°	90.00	90.00	90.00	90.00
β/°	110.610(4)	91.093(5)	90.00	90.00
γ/°	90.00	90.00	90.00	90.00
Volume/Å <sup>3</sup>	1114.43(7)	1433.49(13)	1138.90(9)	1647.71(10)
Z	4	4	4	4
ρ <sub>calc</sub> /mg/mm <sup>3</sup>	1.205	1.188	1.308	1.227
m/mm <sup>-1</sup>	0.083	0.075	0.084	0.077
F(000)	456.0	552.0	472.0	648.0

Crystal size/mm <sup>3</sup>	0.4 × 0.35 × 0.2	0.45 × 0.4 × 0.25	0.45 × 0.4 × 0.08	0.45 × 0.4 × 0.35
2 $\Theta$ range for data collection	5.64 to 50°	5.52 to 50°	6.82 to 50°	6.38 to 58.54°
Index ranges	-10 ≤ h ≤ 9, -13 ≤ k ≤ 20, -9 ≤ l ≤ 9	-14 ≤ h ≤ 9, -10 ≤ k ≤ 10, -7 ≤ l ≤ 15	-12 ≤ h ≤ 12, -13 ≤ k ≤ 13, -11 ≤ l ≤ 11	-7 ≤ h ≤ 7, -15 ≤ k ≤ 15, -30 ≤ l ≤ 14
Reflections collected	4704	4143	4040	5893
Independent reflections	1965[R(int) = 0.0209]	2523[R(int) = 0.0203]	1944[R(int) = 0.0248]	3621[R(int) = 0.0138]
Data/restraints/parameters	1965/0/150	2523/0/178	1944/1/155	3621/0/210
Goodness-of-fit on F <sup>2</sup>	1.043	1.025	1.057	1.046
Final R indexes [I ≥ 2σ (I)]	R <sub>1</sub> = 0.0355, wR <sub>2</sub> = 0.0818	R <sub>1</sub> = 0.0567, wR <sub>2</sub> = 0.1473	R <sub>1</sub> = 0.0389, wR <sub>2</sub> = 0.0949	R <sub>1</sub> = 0.0400, wR <sub>2</sub> = 0.0857
Final R indexes [all data]	R <sub>1</sub> = 0.0433, wR <sub>2</sub> = 0.0864	R <sub>1</sub> = 0.0803, wR <sub>2</sub> = 0.1650	R <sub>1</sub> = 0.0456, wR <sub>2</sub> = 0.1028	R <sub>1</sub> = 0.0509, wR <sub>2</sub> = 0.0932
Largest diff. peak/hole / e Å <sup>-3</sup>	0.18/-0.18	0.23/-0.25	0.11/-0.13	0.14/-0.15

PROTON CDC13 {D:\CRR} crr 1

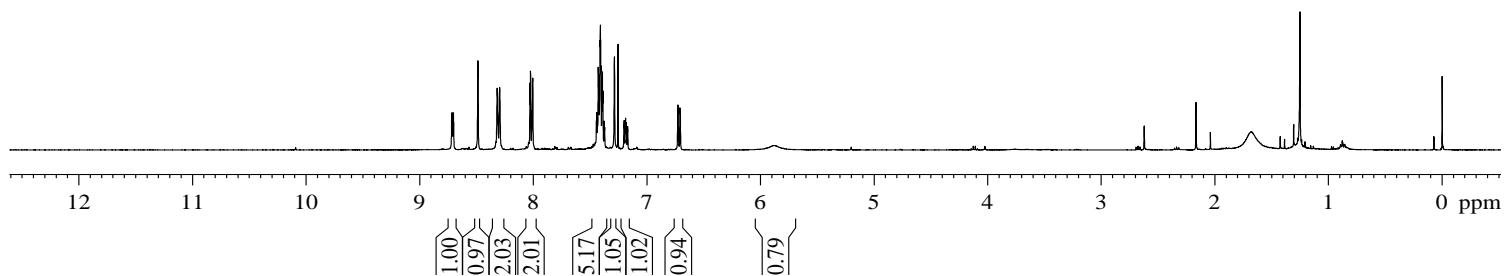


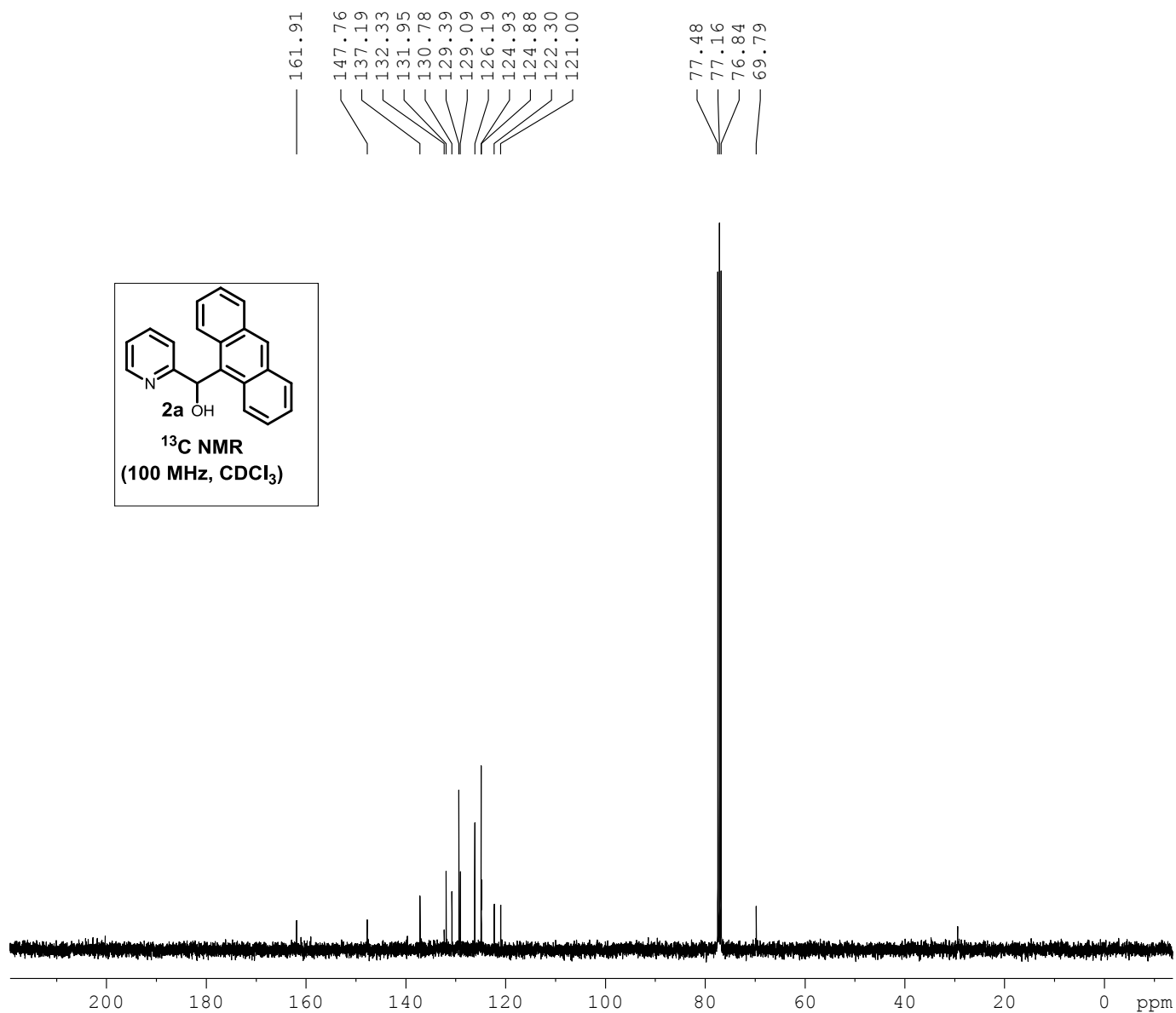
Current Data Paramete  
NAME AHK-I  
EXPNO  
PROCNO

F2 - Acquisition Para  
Date\_ 201001  
Time\_ 22.  
INSTRUM spe  
PROBHD 5 mm BBO BB-  
PULPROG zg  
TD 327  
SOLVENT CDC  
NS  
DS  
SWH 8223.6  
FIDRES 0.2509  
AQ 1.99234  
RG 3  
DW 60.8  
DE 6.  
TE 297  
D1 2.000000  
TD0

==== CHANNEL f1 =  
NUC1  
P1 14.  
PL1 -0.  
SFO1 400.13247

F2 - Processing param  
SI 327  
SF 400.13000  
WDW  
SSB  
LB 0.  
GB  
PC 1.





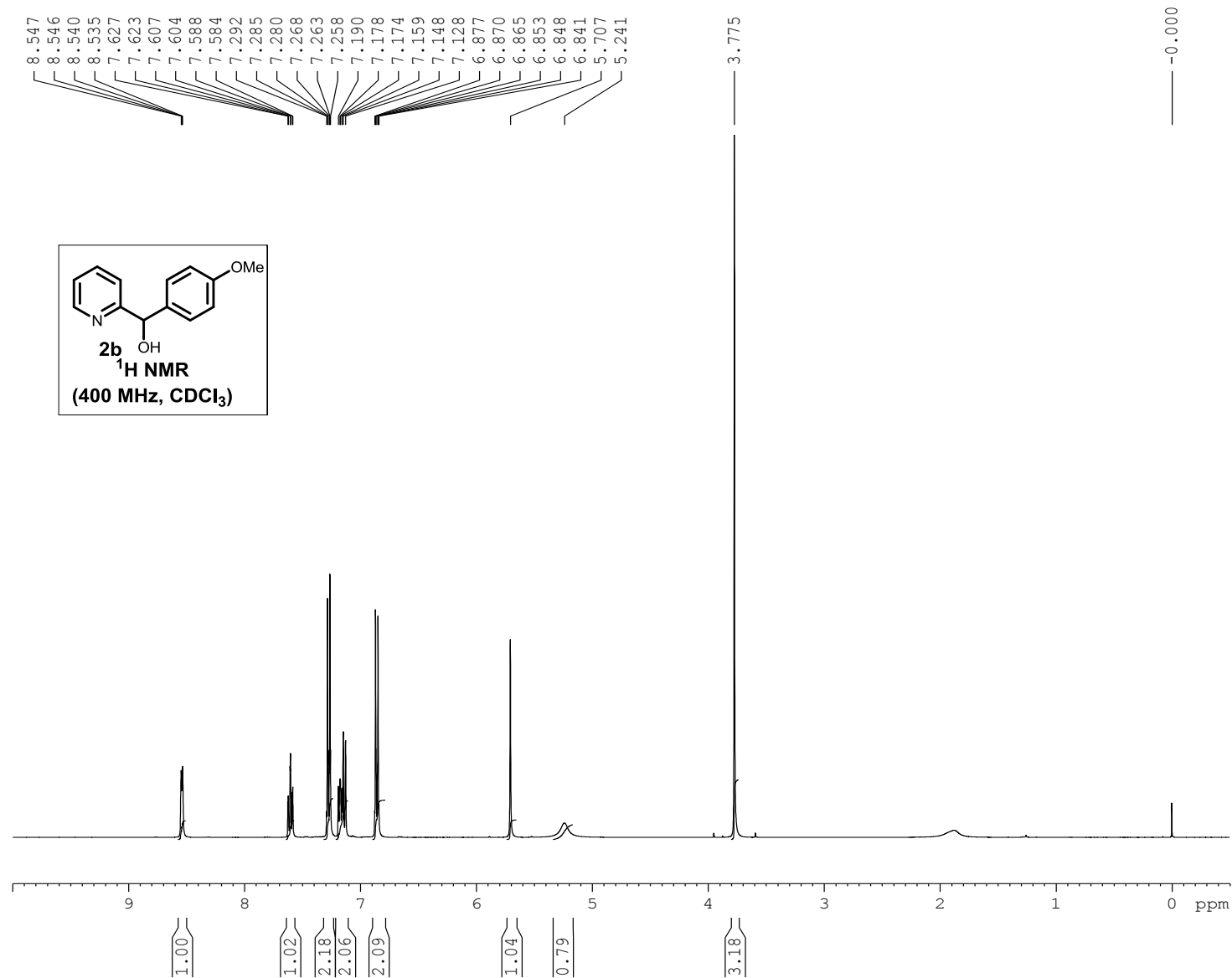
```
Current Data Parameters
NAME          EG-AHK-I-5B
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20100127
Time          12.39
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            812
DW            20.800 usec
DE            6.00 usec
TE            297.1 K
D1            2.0000000 sec
d11           0.0300000 sec
DELTA         1.89999998 sec
TD0           1

===== CHANNEL f1 =====
NUC1           13C
P1             9.50 usec
PL1            -0.60 dB
SFO1           100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2          90.00 usec
PL12           15.60 dB
PL13           15.60 dB
PL2            -0.90 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI             32768
SF            100.6127543 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```



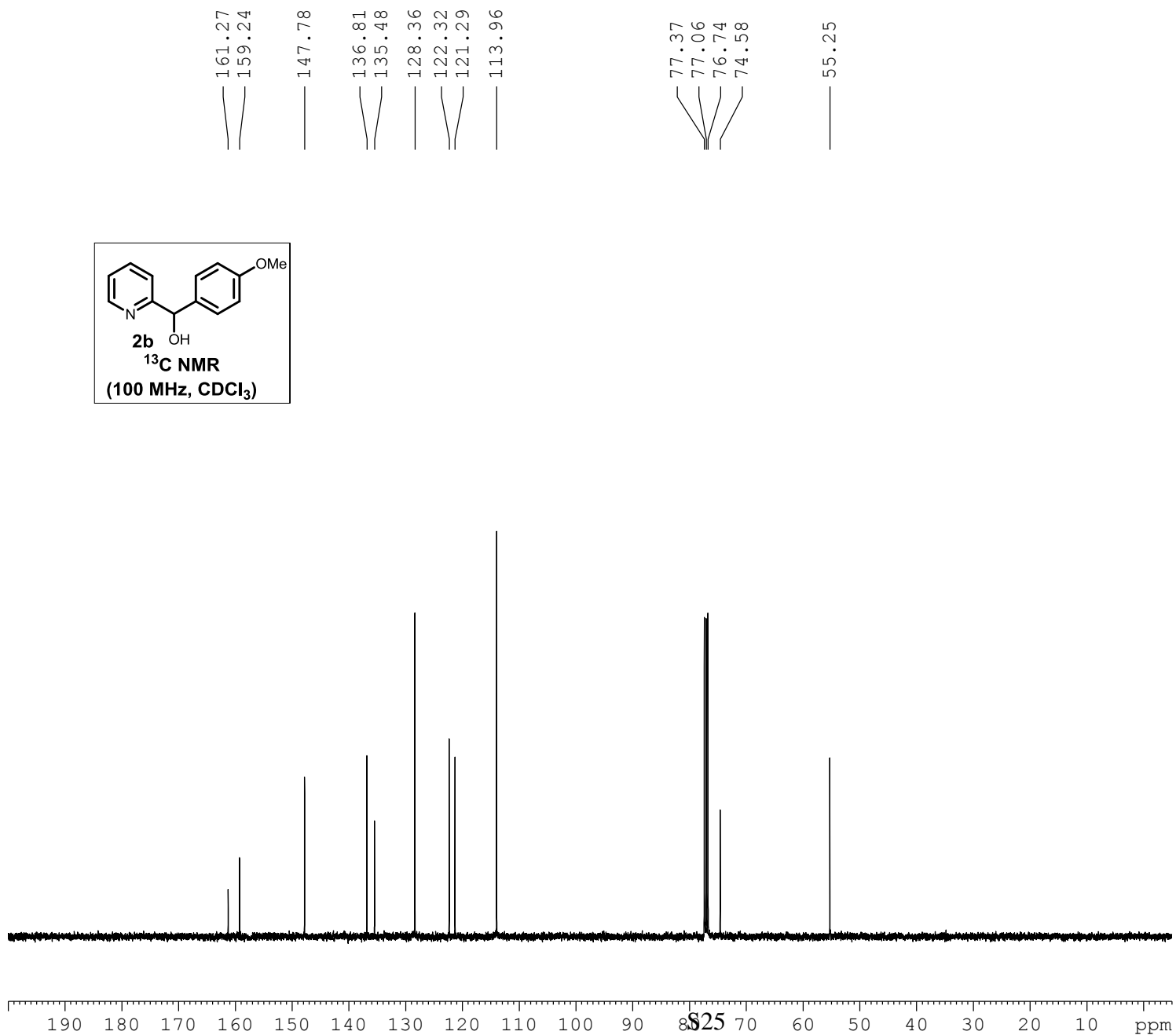
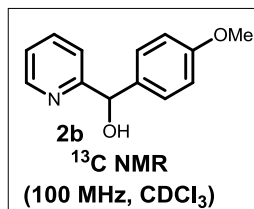
Current Data Parameters  
NAME AHK-71B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100914  
Time\_ 11.20  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 128  
DW 60.800 usec  
DE 6.00 usec  
TE 296.0 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300050 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





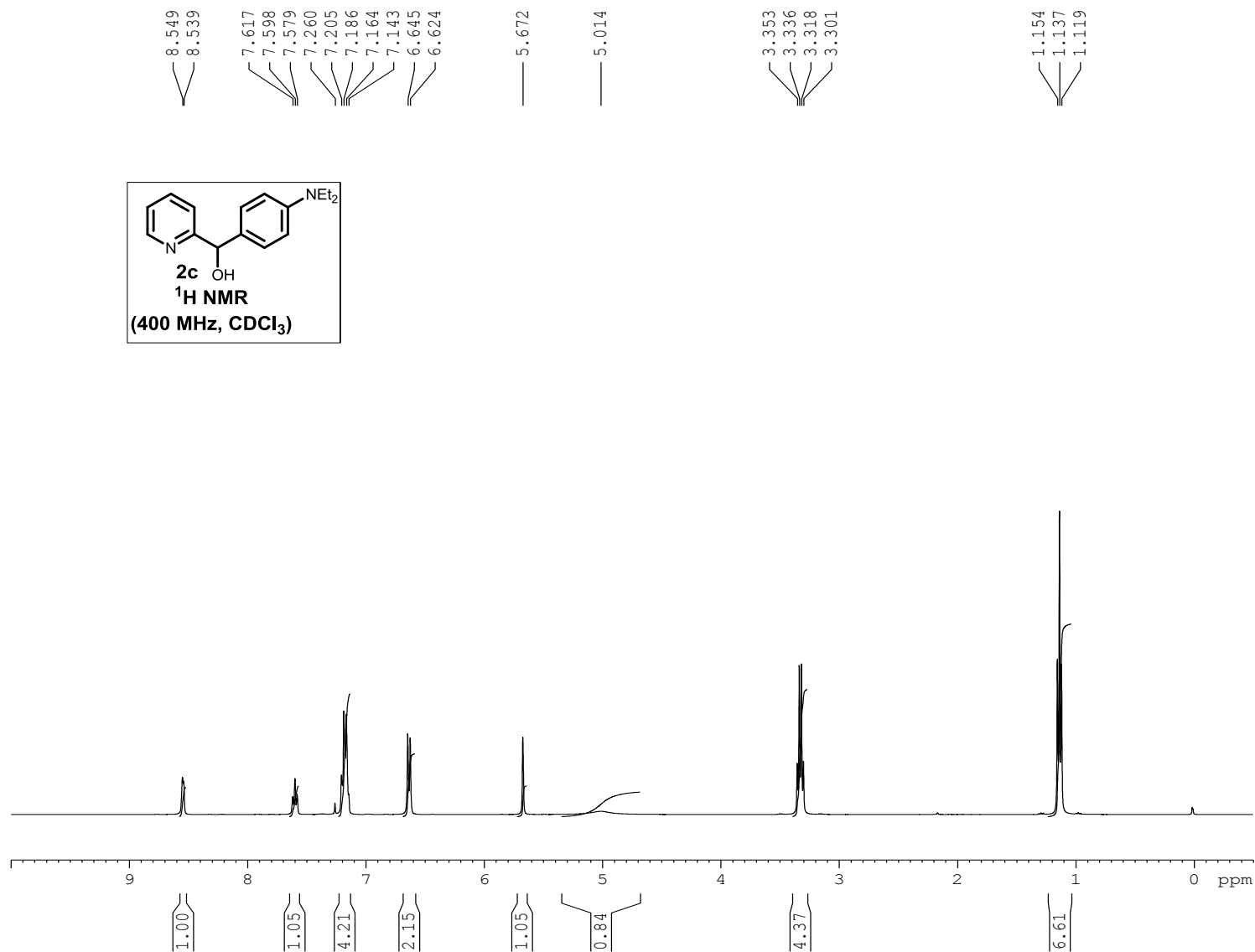
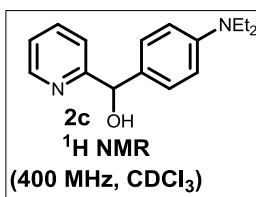
Current Data Parameters  
NAME AHK-71B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100914  
Time\_ 11.35  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 2050  
DW 20.800 usec  
DE 6.00 usec  
TE 296.7 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

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



Current Data Parameters  
NAME AHK-I-72B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100916  
Time\_ 10.37  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 161  
DW 60.800 usec  
DE 6.00 usec  
TE 296.4 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300028 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



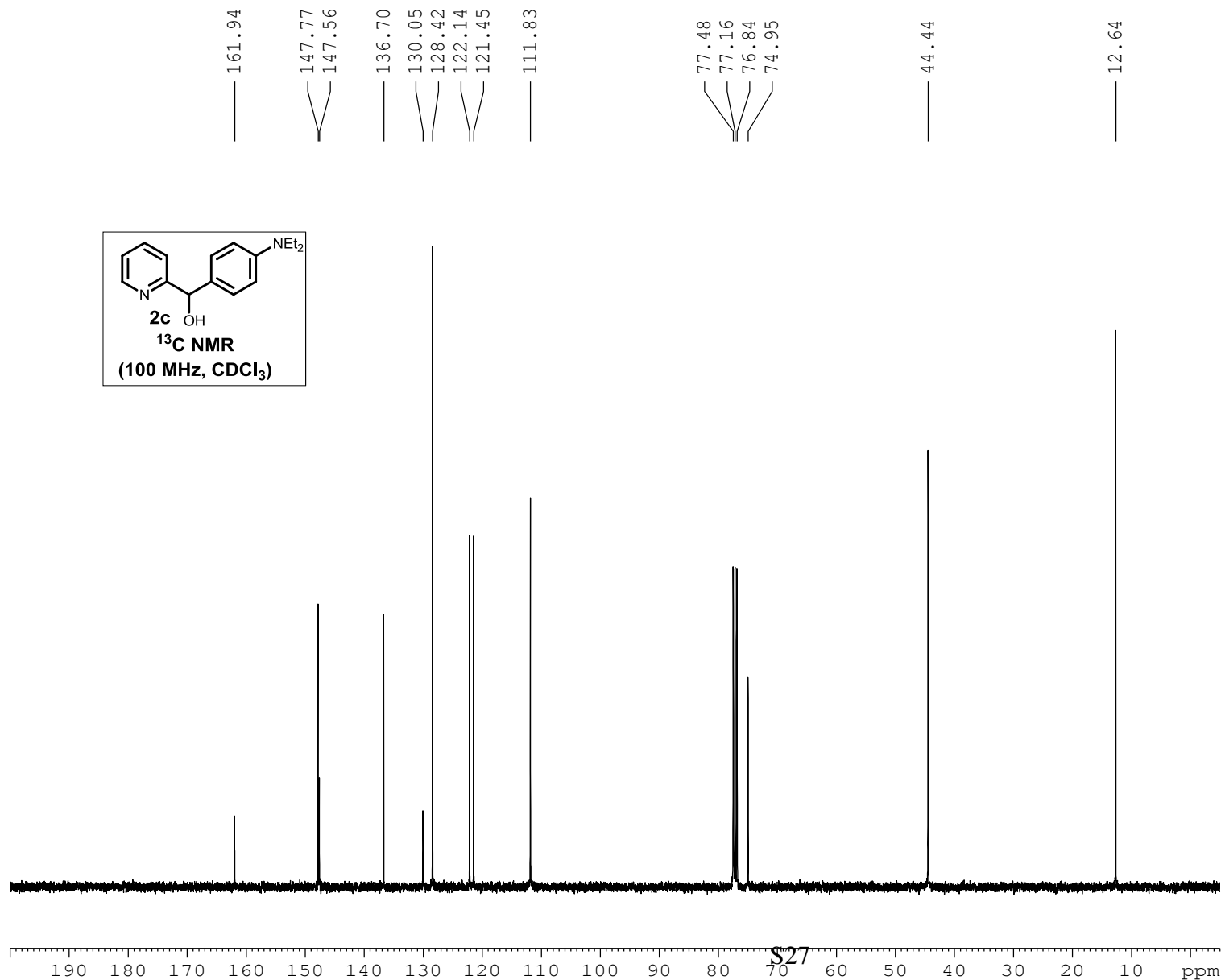
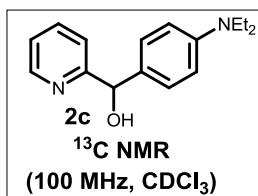
Current Data Parameters  
NAME AHK-I-72B  
EXPNO 2  
PROCNO 1

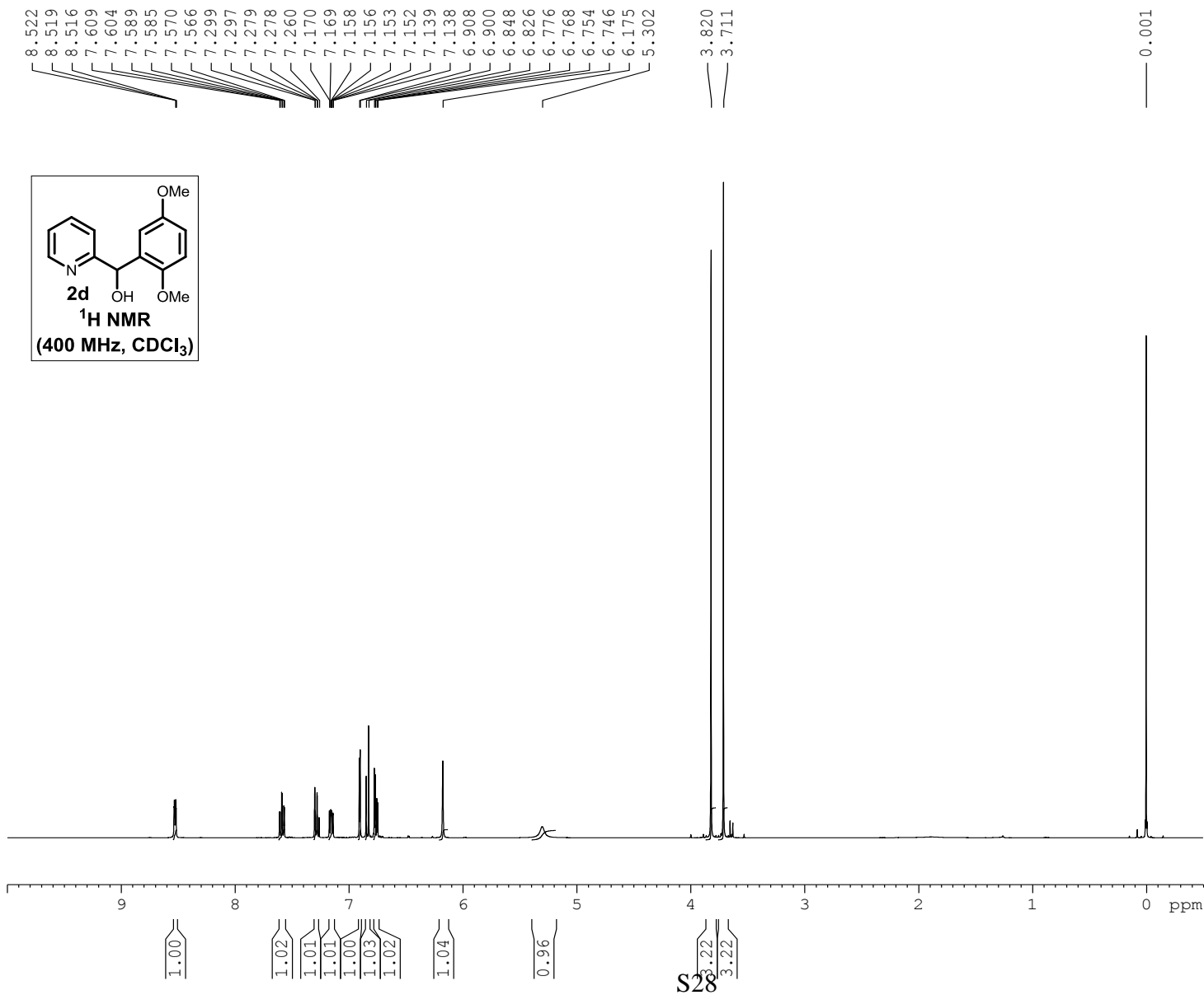
F2 - Acquisition Parameters  
Date\_ 20100916  
Time 10.38  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 778  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 57  
DW 20.800 usec  
DE 6.00 usec  
TE 296.8 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127613 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



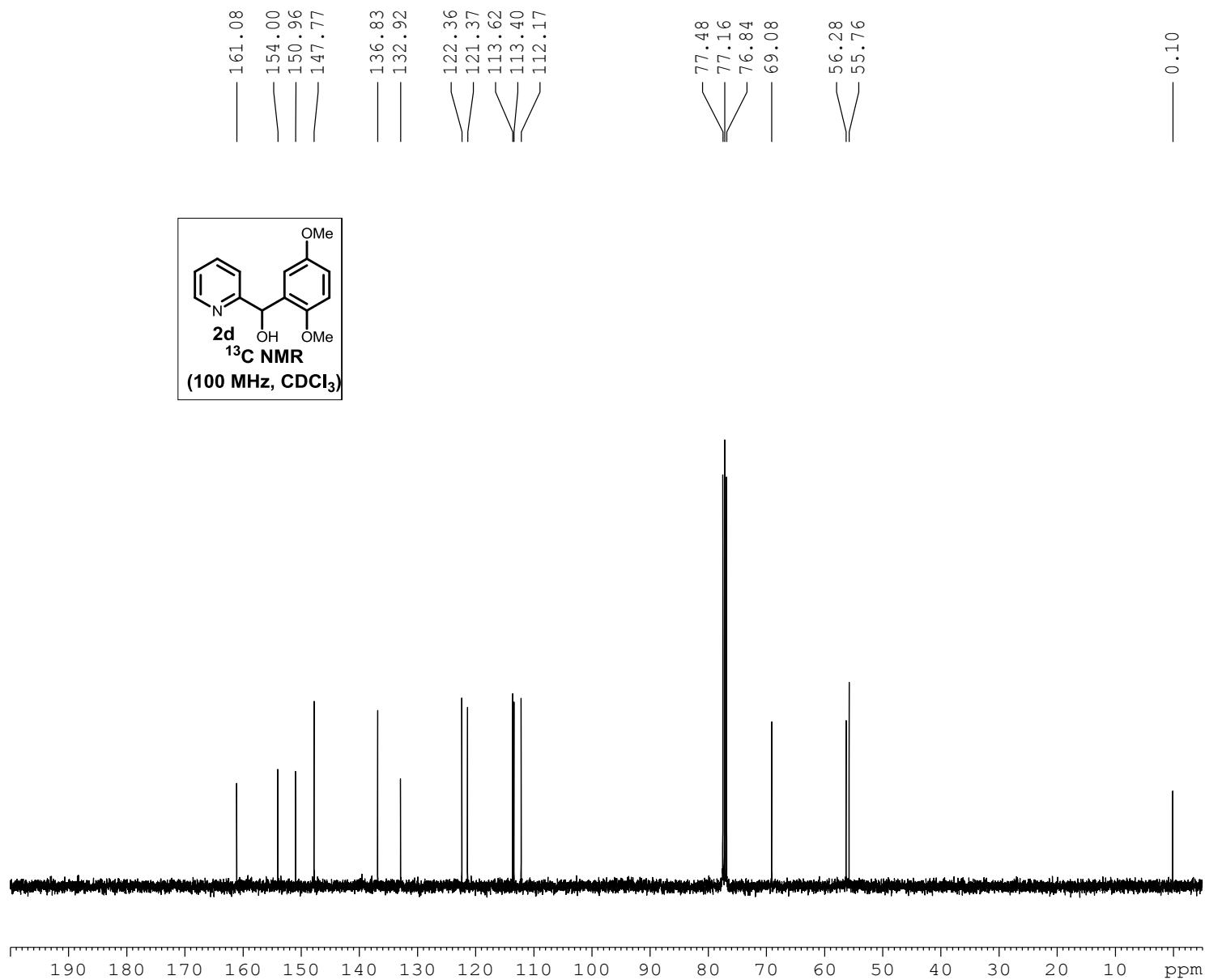


Current Data Parameters  
NAME AHK-I-141-B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110623  
Time\_ 12.15  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 128  
DW 60.800 usec  
DE 6.00 usec  
TE 295.8 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300038 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



Current Data Parameters  
NAME AHK-I-141-B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110623  
Time\_ 12.20  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl<sub>3</sub>  
NS 92  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1150  
DW 20.800 usec  
DE 6.00 usec  
TE 296.4 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127582 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

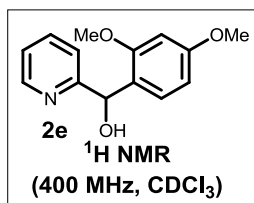
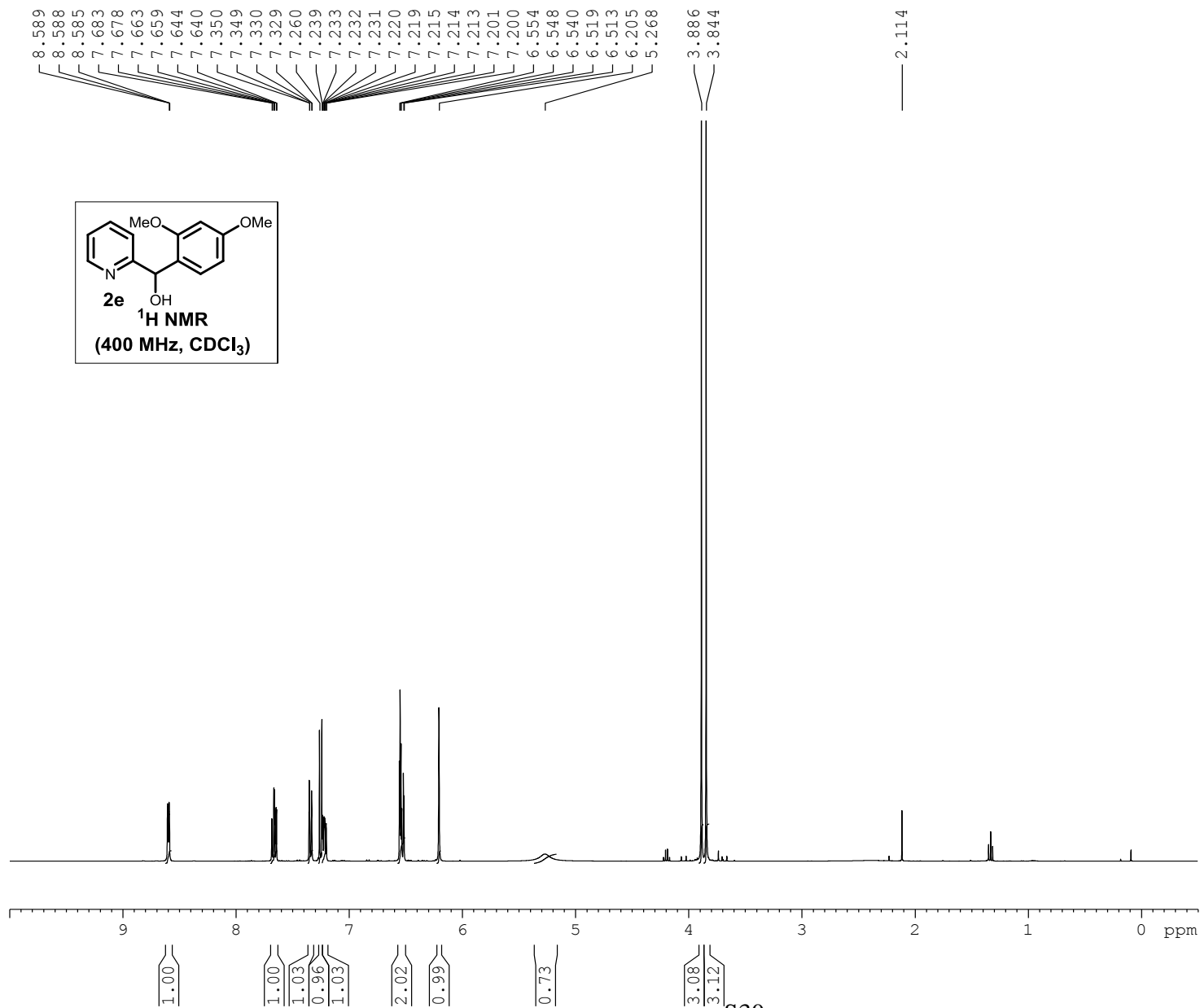


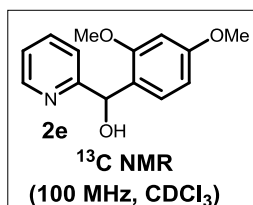
Current Data Parameters  
NAME AHK-I-64  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100819  
Time 12.22  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 57  
DW 60.800 usec  
DE 6.00 usec  
TE 294.6 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1299664 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





161.63  
160.35  
157.79  
147.70  
136.62  
128.63  
124.26  
122.07  
121.15  
104.60  
98.58  
77.16  
69.11  
55.47  
55.31

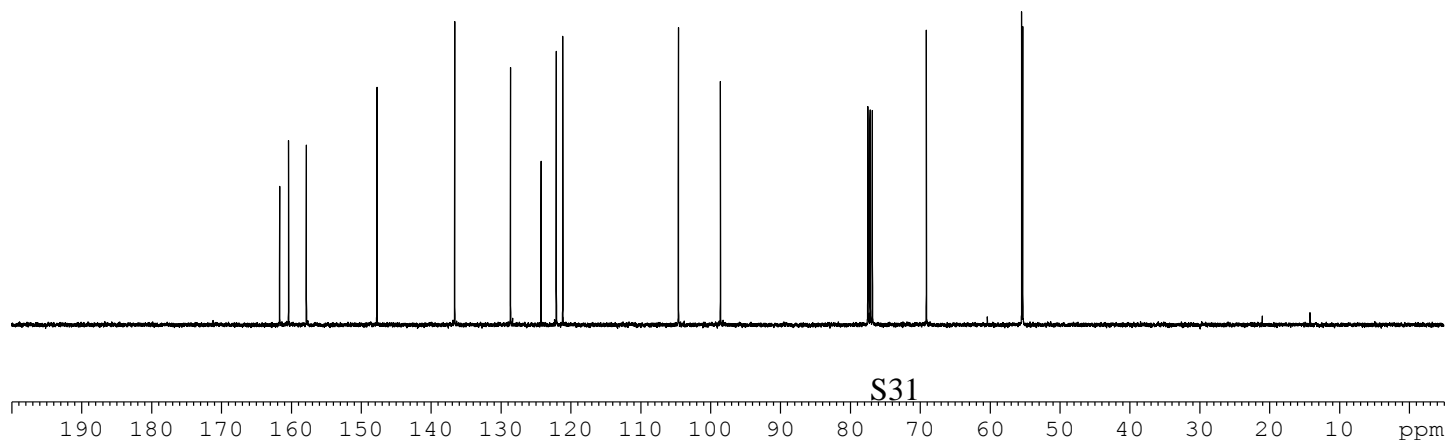
Current Data Parameters  
NAME AHK-I-64  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100819  
Time\_ 12.38  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 812  
DW 20.800 usec  
DE 6.00 usec  
TE 295.2 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127694 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



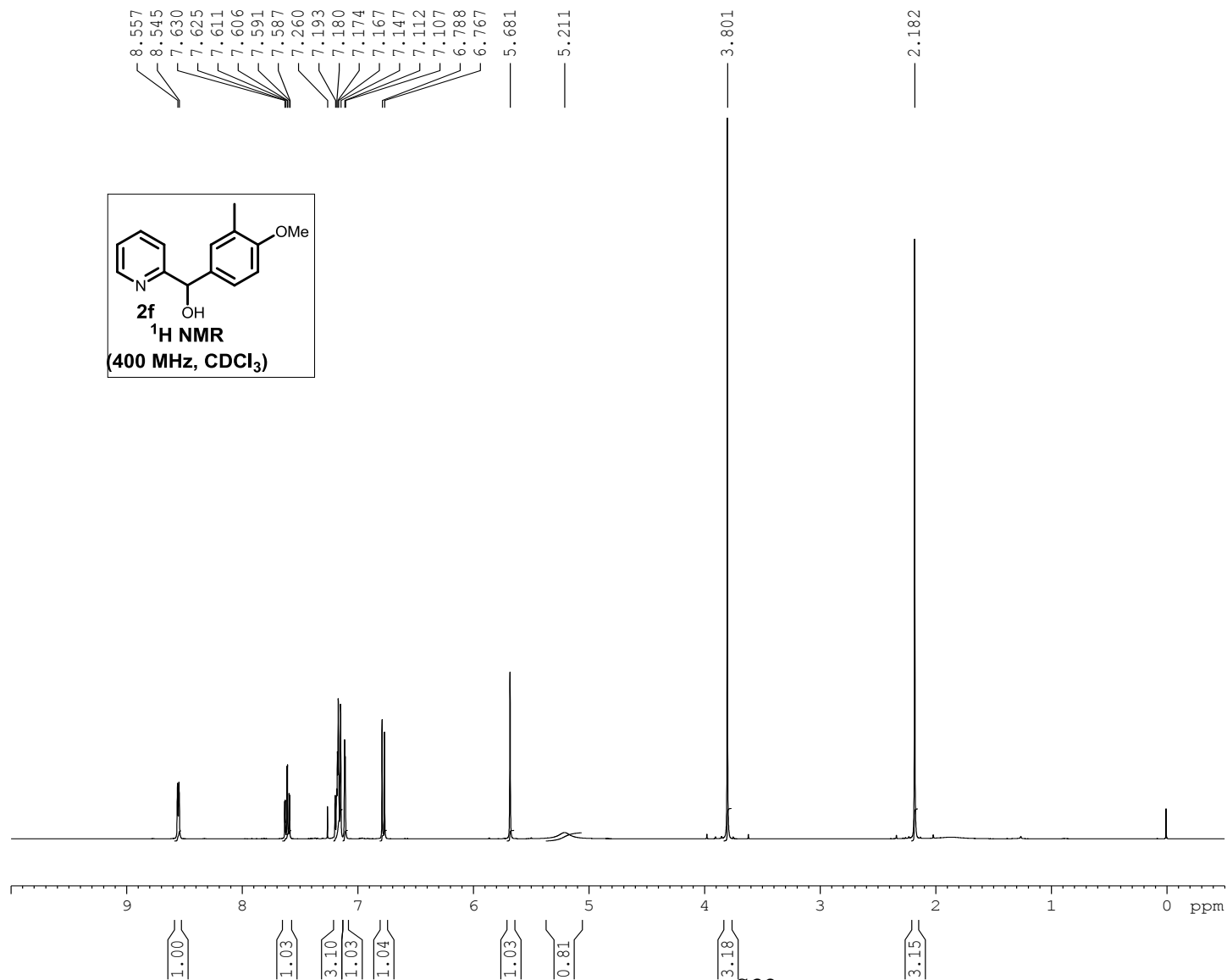
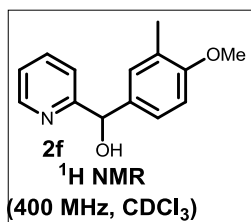


Current Data Parameters  
NAME AHK-I-102B  
EXNO 1  
PROCNO 1

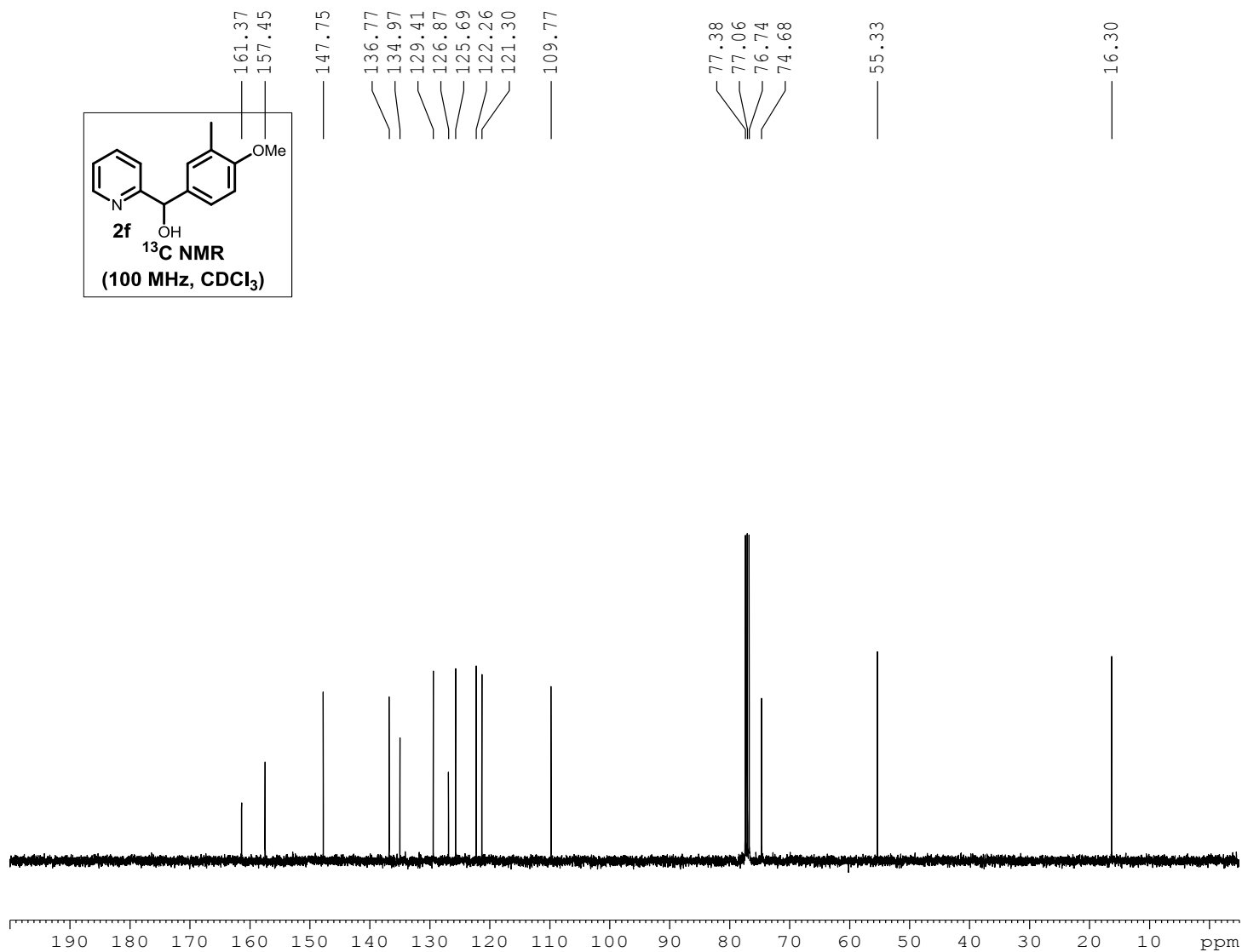
F2 - Acquisition Parameters  
Date\_ 20110314  
Time\_ 11.50  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 128  
DW 60.800 usec  
DE 6.00 usec  
TE 294.6 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300038 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00







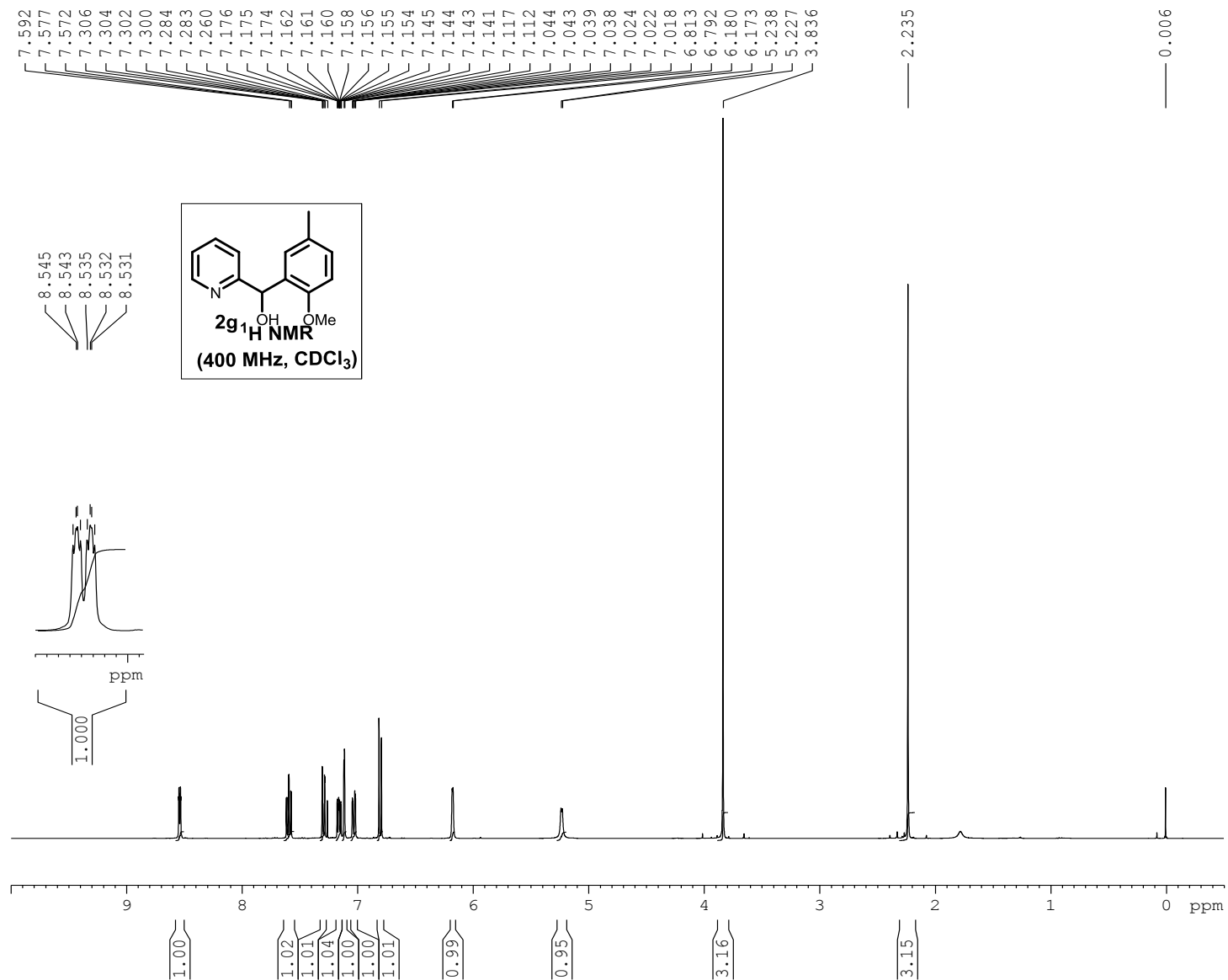
Current Data Parameters  
NAME AHK-I-102B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110314  
Time\_ 12.02  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 113  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 50.8  
DW 20.800 usec  
DE 6.00 usec  
TE 295.2 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

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

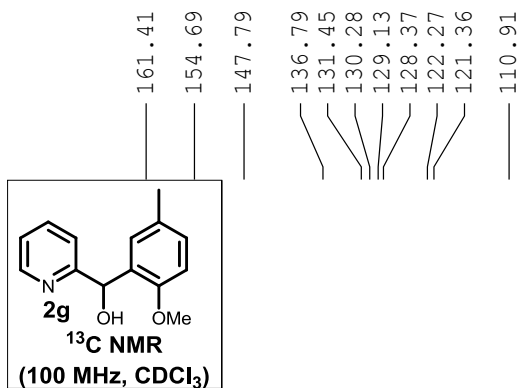


Current Data Parameters  
NAME AHK-I-103-A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110311  
Time 11.29  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 144  
DW 60.800 usec  
DE 6.00 usec  
TE 295.2 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300040 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



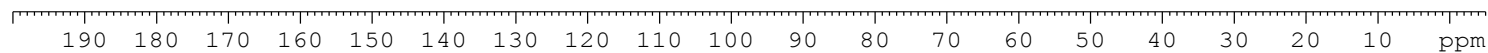
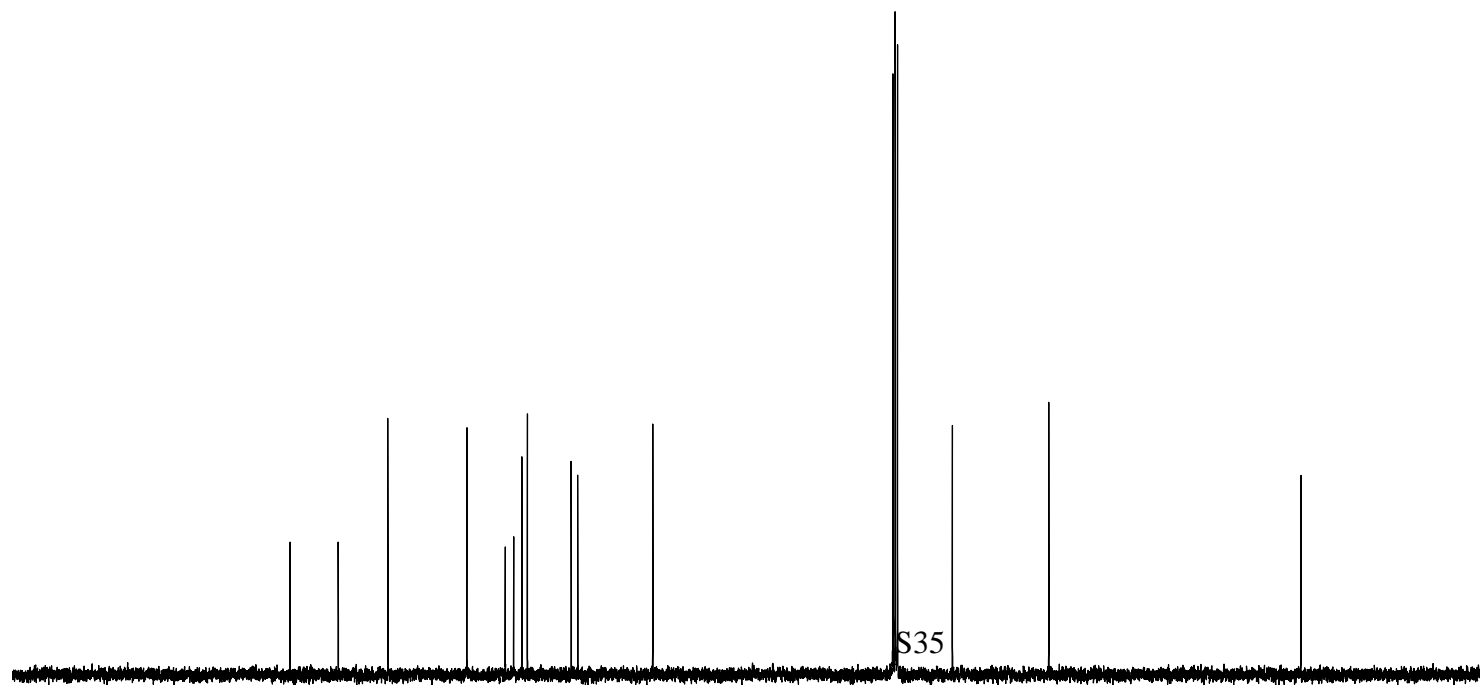
Current Data Parameters  
NAME AHK-I-103-A  
EXPNO 2  
PROCNO 1

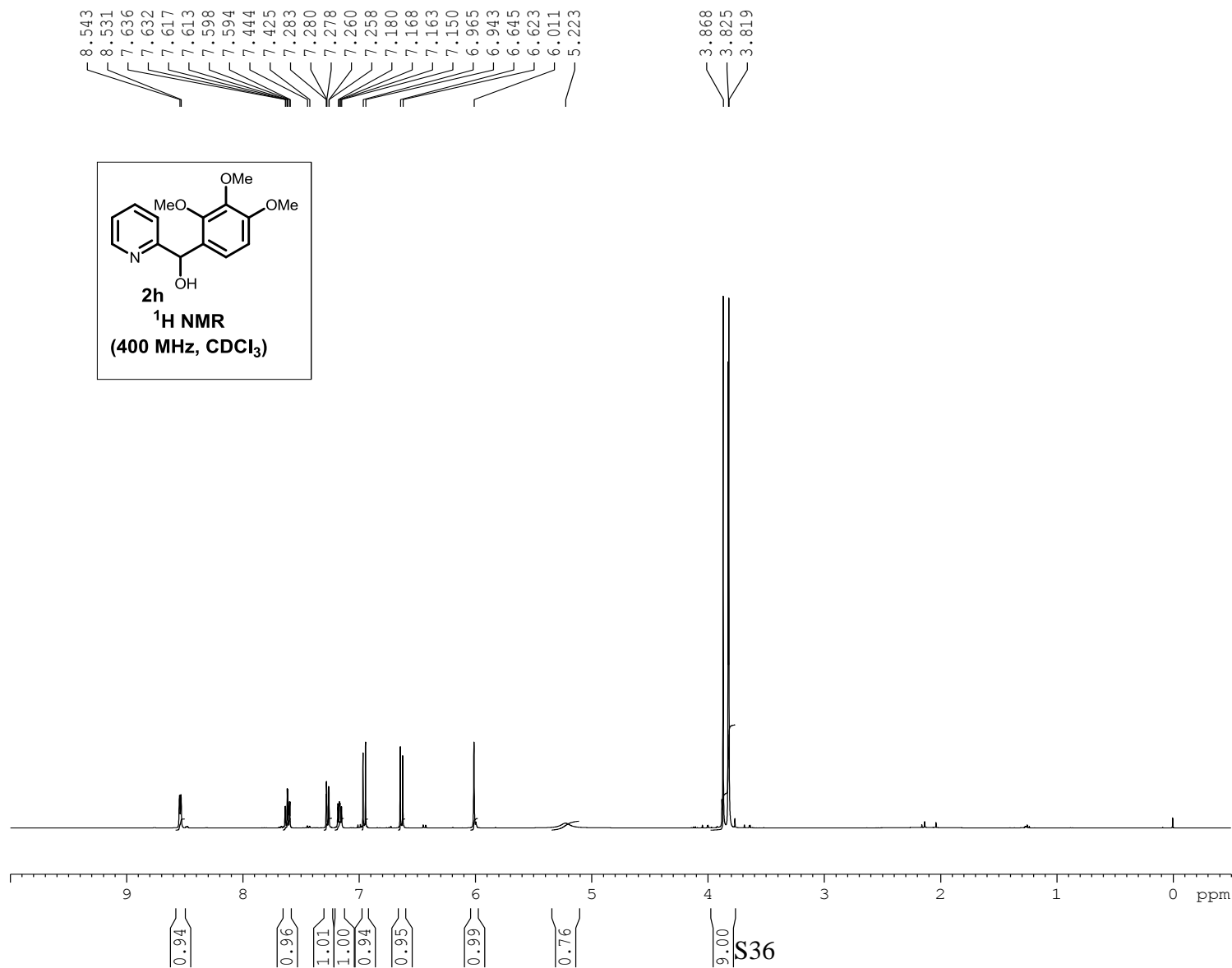
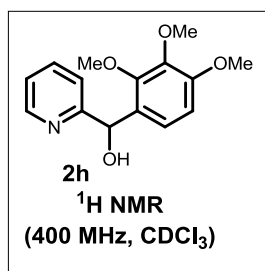
F2 - Acquisition Parameters  
Date\_ 20110311  
Time 11.38  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 167  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 2050  
DW 20.800 usec  
DE 6.00 usec  
TE 295.7 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127572 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



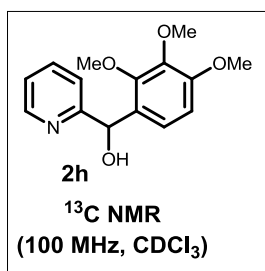


Current Data Parameters  
NAME AHK-I-63  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100819  
Time 12.46  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 80.6  
DW 60.800 usec  
DE 6.00 usec  
TE 294.8 K  
D1 1.00000000 sec  
TDO 1

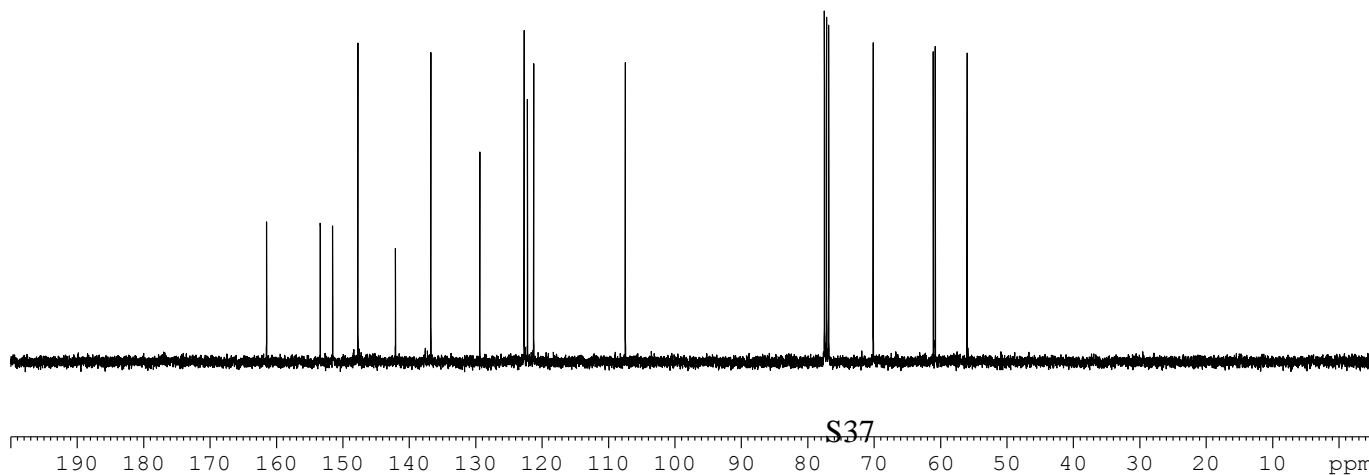
===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1299948 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



161.48  
153.38  
151.52  
147.72  
142.07  
136.74  
129.36  
122.68  
122.18  
121.25  
107.45

77.47  
77.15  
76.83  
70.12  
61.07  
60.76  
55.97



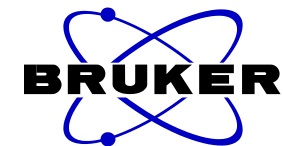
Current Data Parameters  
NAME AHK-I-63  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100819  
Time\_ 12.49  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 89  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 50.8  
DW 20.800 usec  
DE 6.00 usec  
TE 295.4 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127653 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

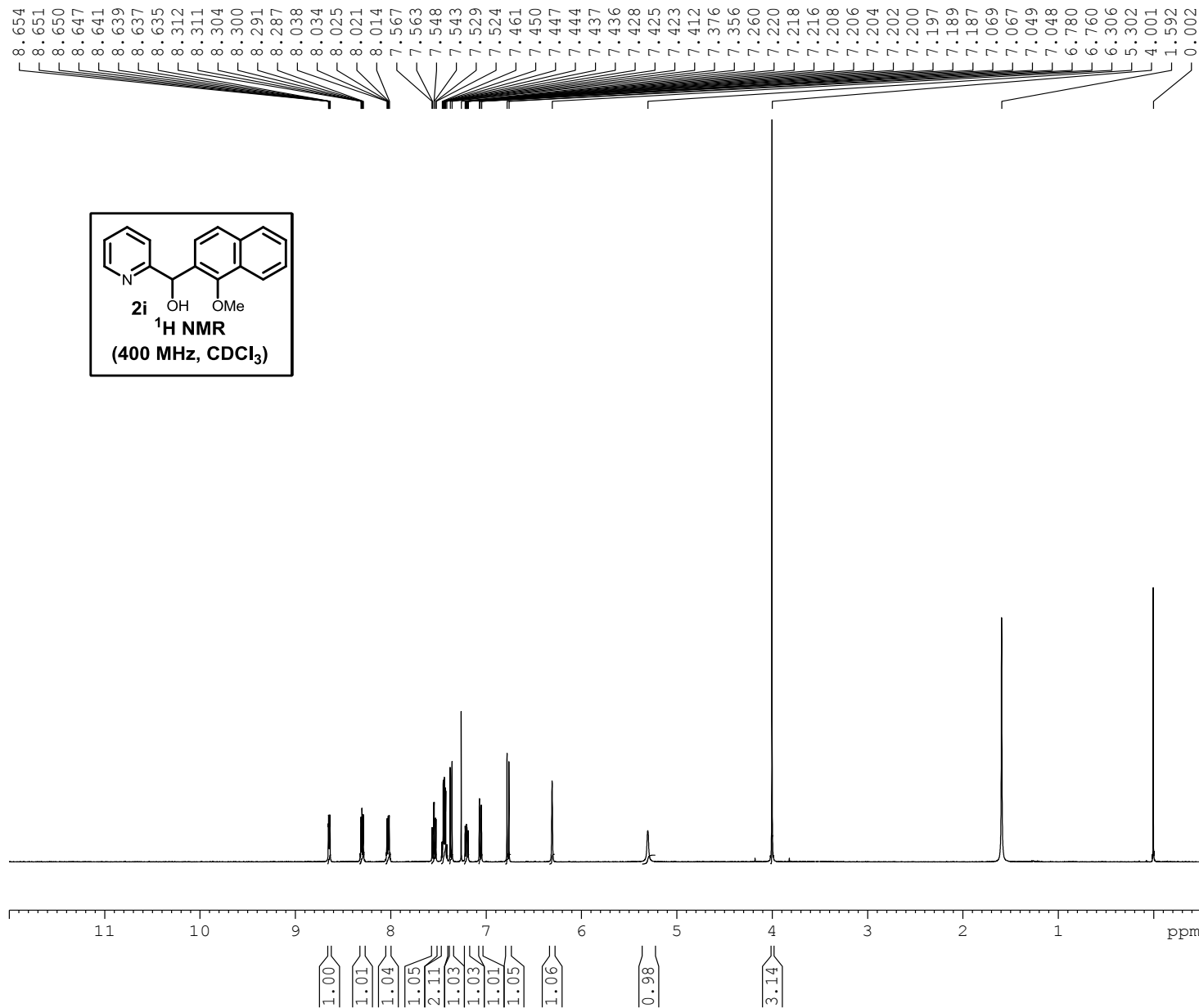


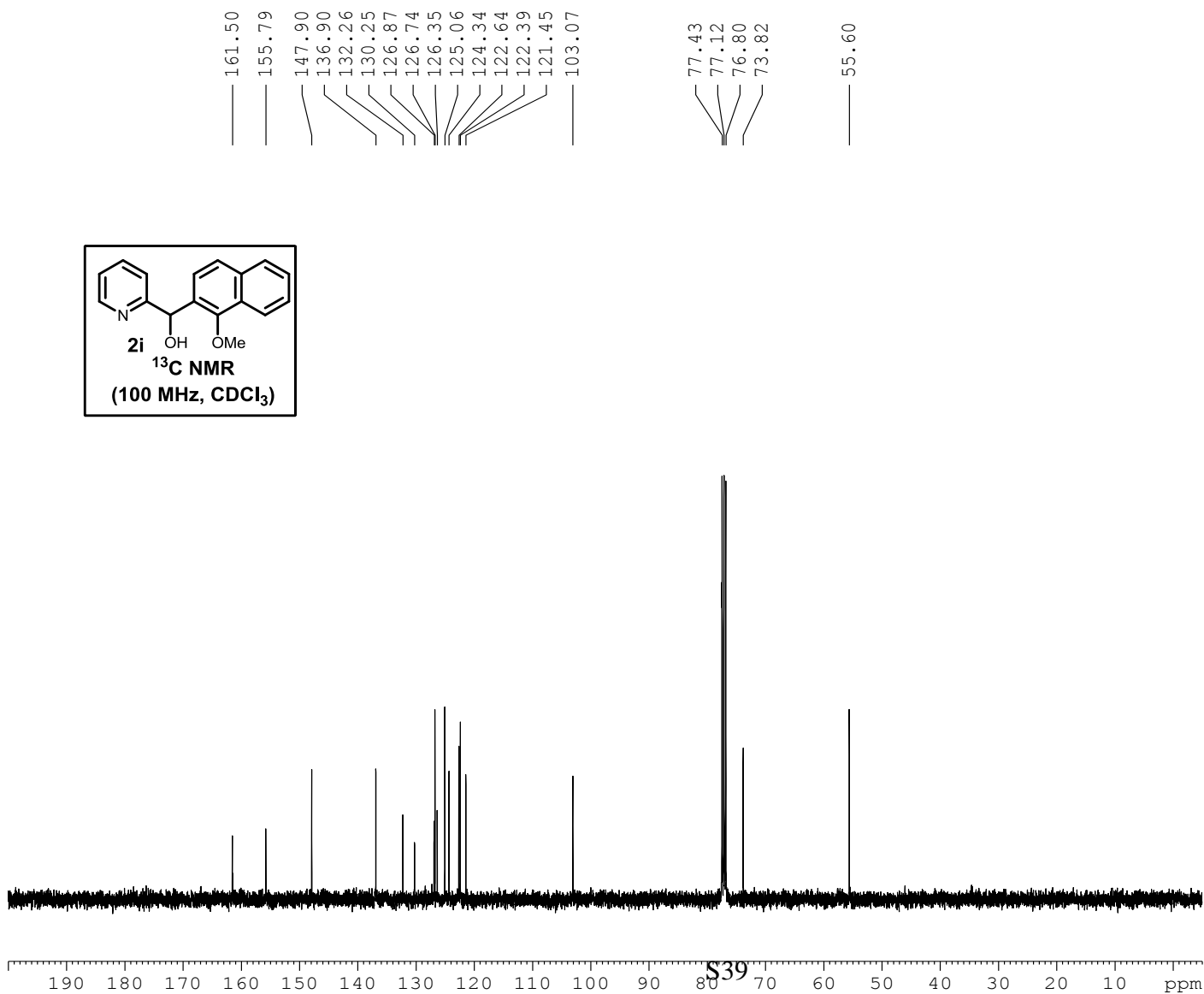
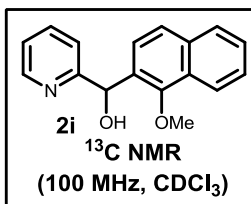
Current Data Parameters  
NAME AHK-I-69 D  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111003  
Time\_ 10.29  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 256  
DW 60.800 usec  
DE 6.00 usec  
TE 297.3 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300041 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





```
Current Data Parameters
NAME          AHK-I-69B
EXPNO         2
PROCNO        1

F2 - Acquisition Parameters
Date_         20100913
Time_         11.05
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            1150
DW            20.800 usec
DE            6.00 usec
TE            296.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0           1

===== CHANNEL f1 =====
NUC1           13C
P1             9.50 usec
PL1            -0.60 dB
SFO1           100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         90.00 usec
PL12          15.60 dB
PL13          15.60 dB
PL2           -0.90 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI             32768
SF            100.6127600 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```



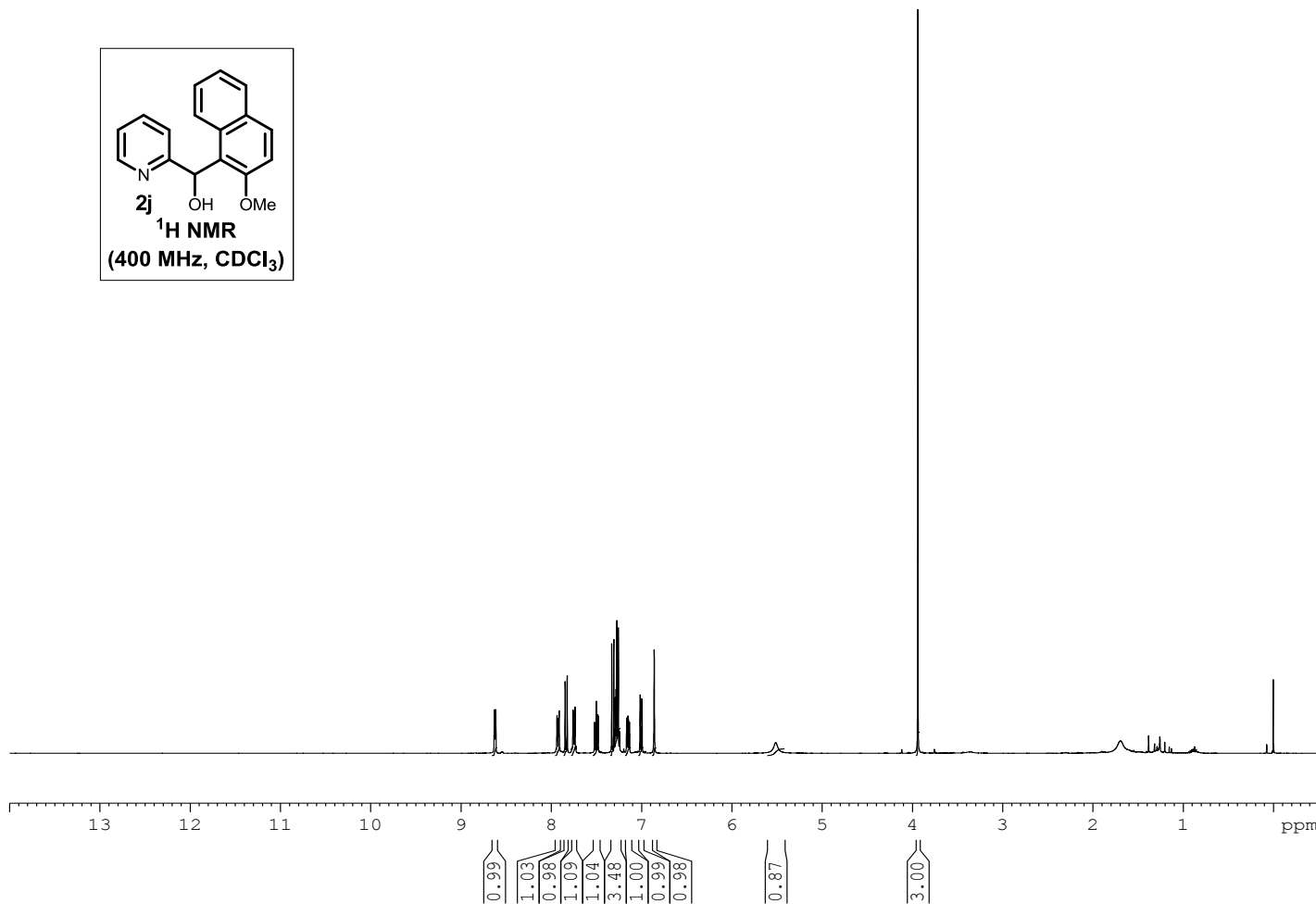
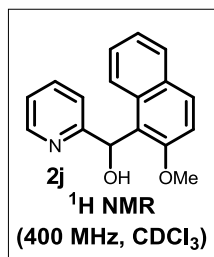
Current Data Parameters  
NAME AHK-I-31-A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100517  
Time 12.58  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.250967 Hz  
AQ 1.9923444 sec  
RG 406  
DW 60.800 usec  
DE 6.00 usec  
TE 296.4 K  
D1 2.0000000 sec  
TD0 1

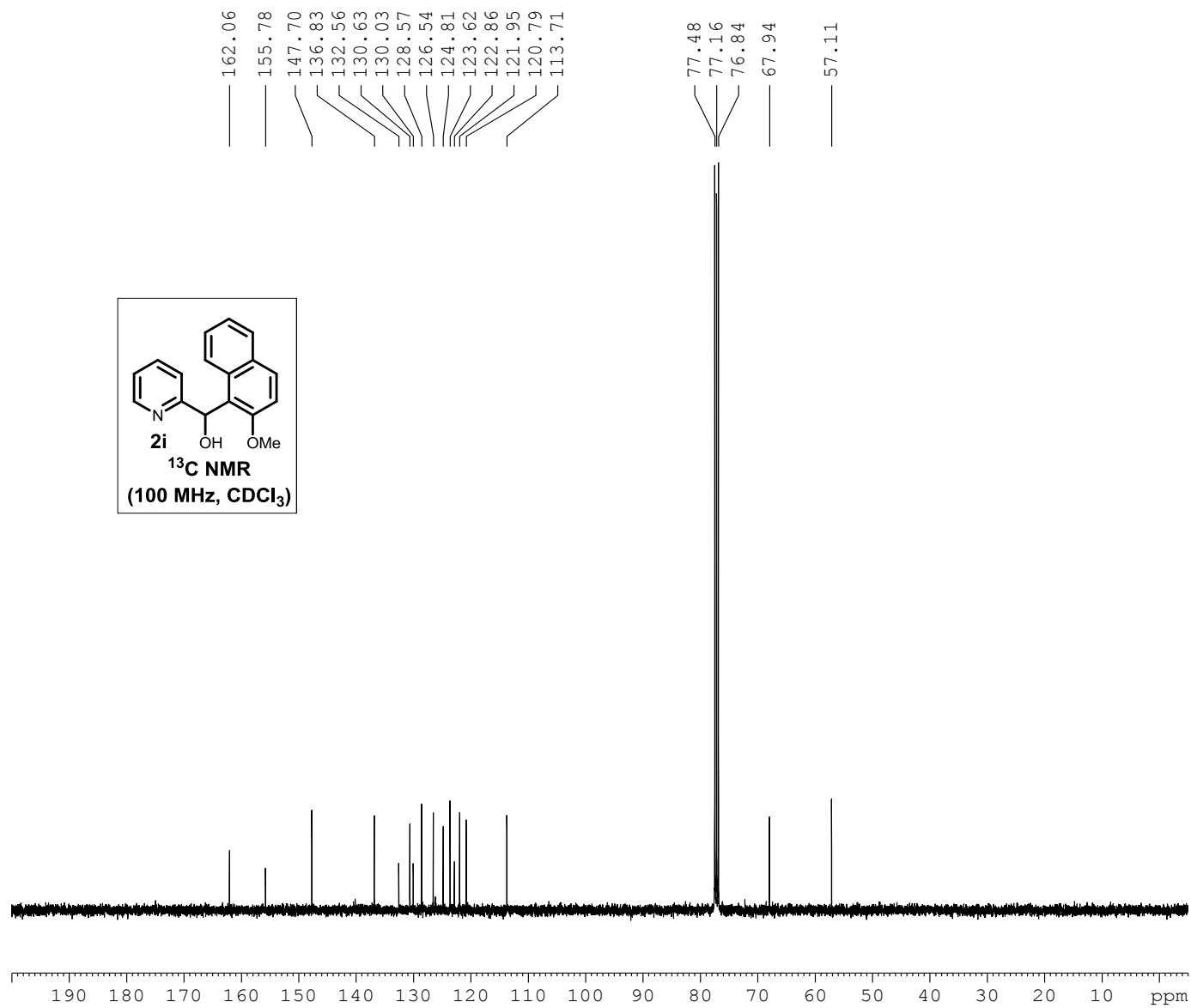
==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300067 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

8.630  
8.627  
8.624  
8.618  
8.615  
8.612  
7.935  
7.929  
7.920  
7.918  
7.912  
7.911  
7.844  
7.822  
7.758  
7.755  
7.750  
7.741  
7.735  
7.519  
7.515  
7.500  
7.496  
7.481  
7.477  
7.328  
7.306  
7.301  
7.289  
7.284  
7.279  
7.272  
7.263  
7.260  
7.256  
7.254  
7.243  
7.164  
7.162  
7.160  
7.150  
7.147  
7.144  
7.133  
7.131  
7.129  
7.129  
7.014  
7.012  
6.994  
6.992  
6.857  
5.510  
3.937







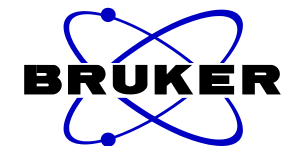
Current Data Parameters  
NAME AHK-I-31-A  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100517  
Time 13.29  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 512  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1030  
DW 20.800 usec  
DE 6.00 usec  
TE 296.9 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127550 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

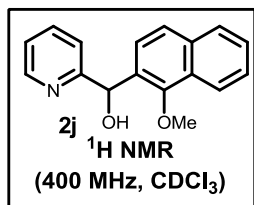
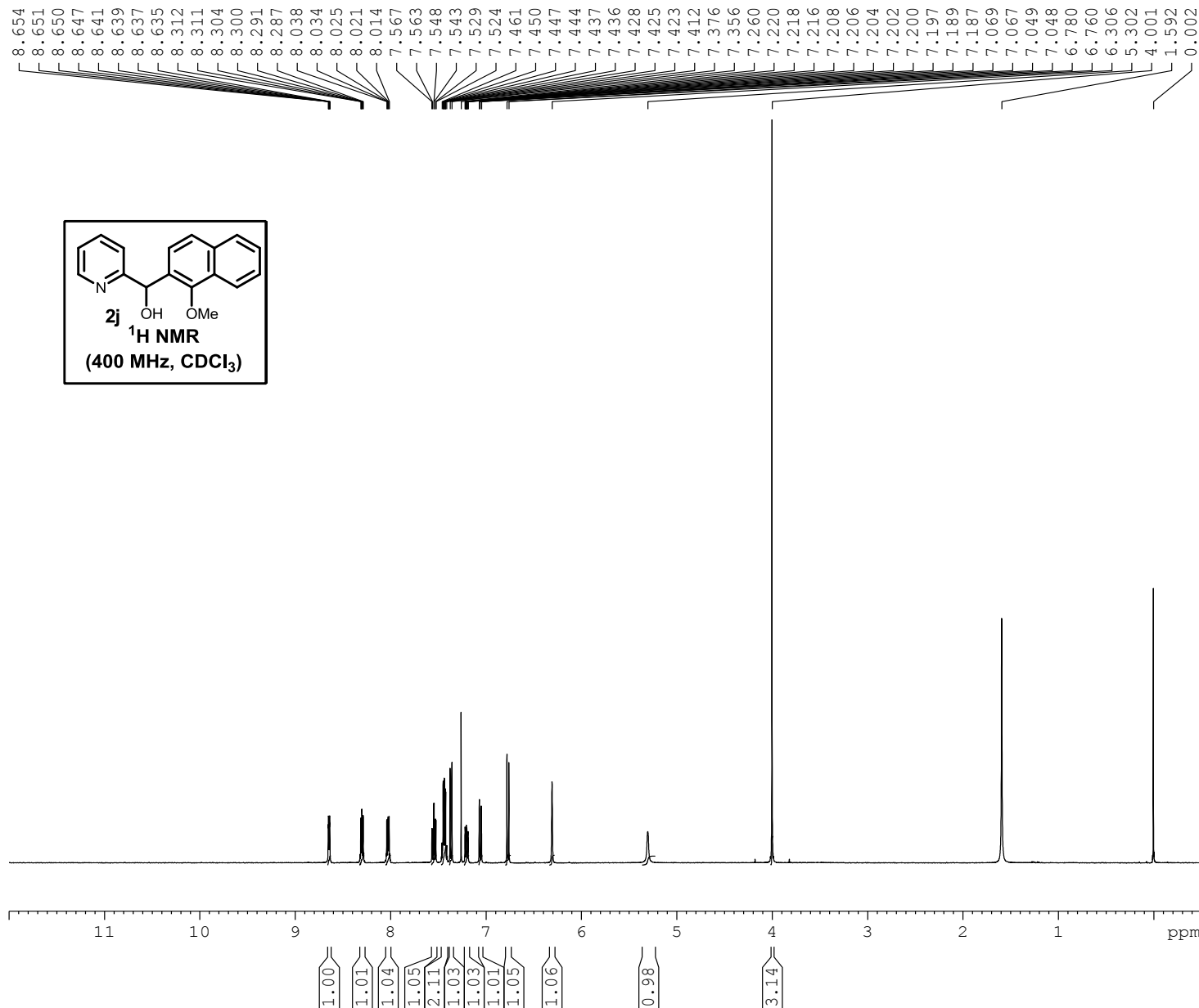


Current Data Parameters  
NAME AHK-I-69 D  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20111003  
Time\_ 10.29  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 256  
DW 60.800 usec  
DE 6.00 usec  
TE 297.3 K  
D1 1.0000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300041 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





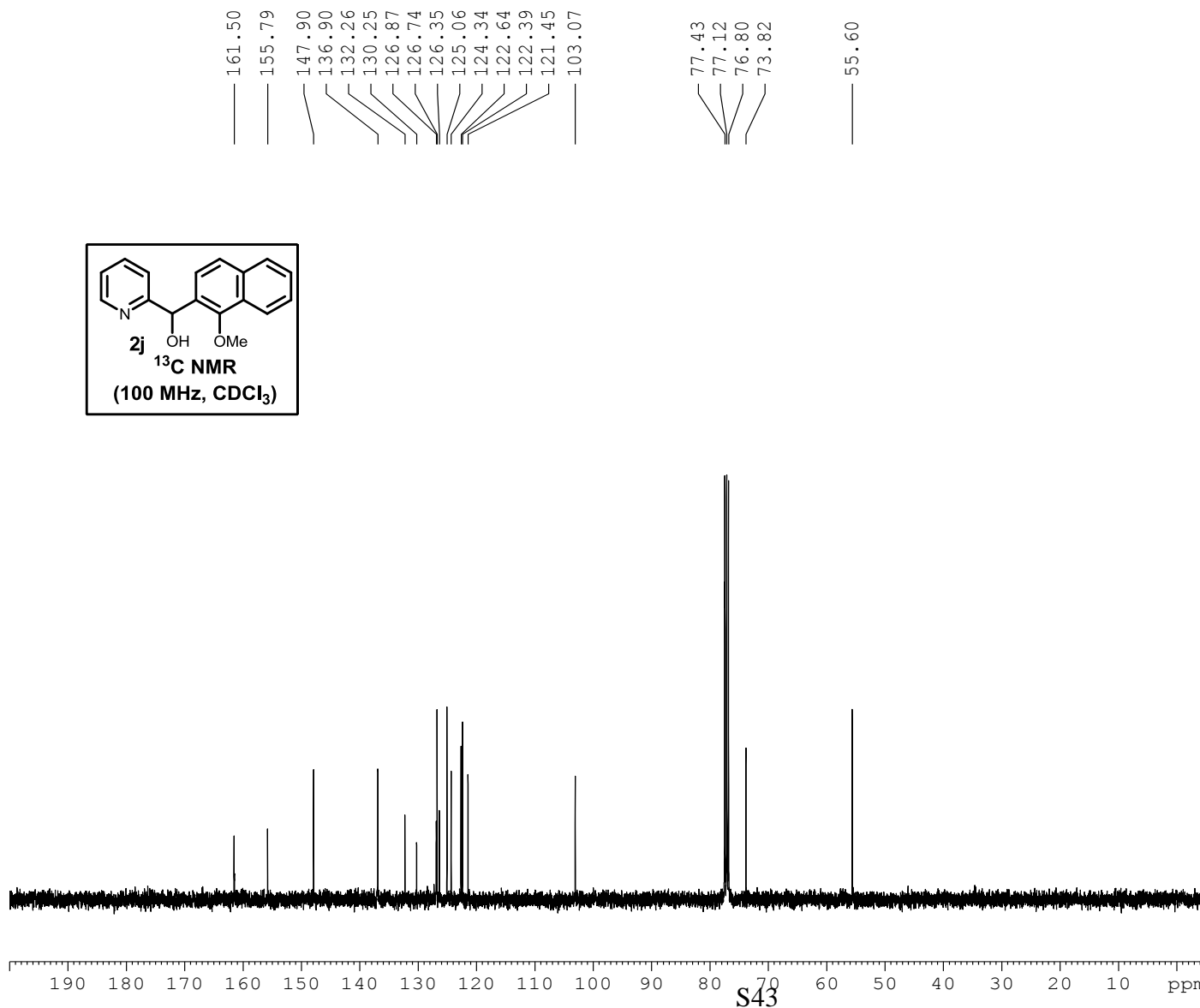
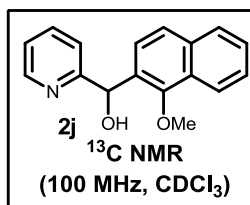
```
Current Data Parameters
NAME          AHK-I-69B
EXPNO         2
PROCNO        1

F2 - Acquisition Parameters
Date_         20100913
Time_         11.05
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            256
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            1150
DW            20.800 usec
DE            6.00 usec
TE            296.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TDO           1

===== CHANNEL f1 =====
NUC1           13C
P1             9.50 usec
PL1            -0.60 dB
SFO1           100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2           1H
PCPD2         90.00 usec
PL12          15.60 dB
PL13          15.60 dB
PL2           -0.90 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI             32768
SF            100.6127600 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```



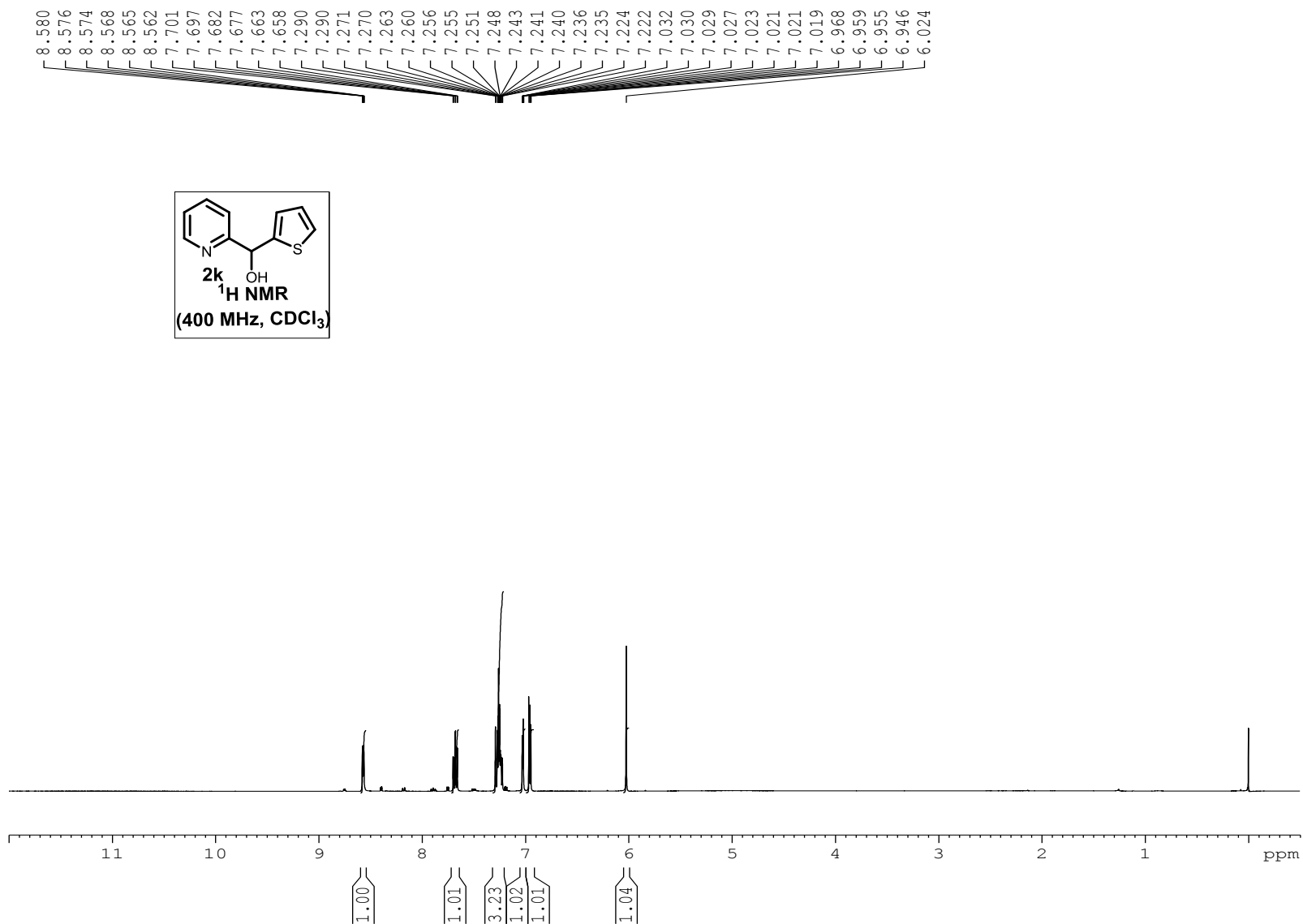


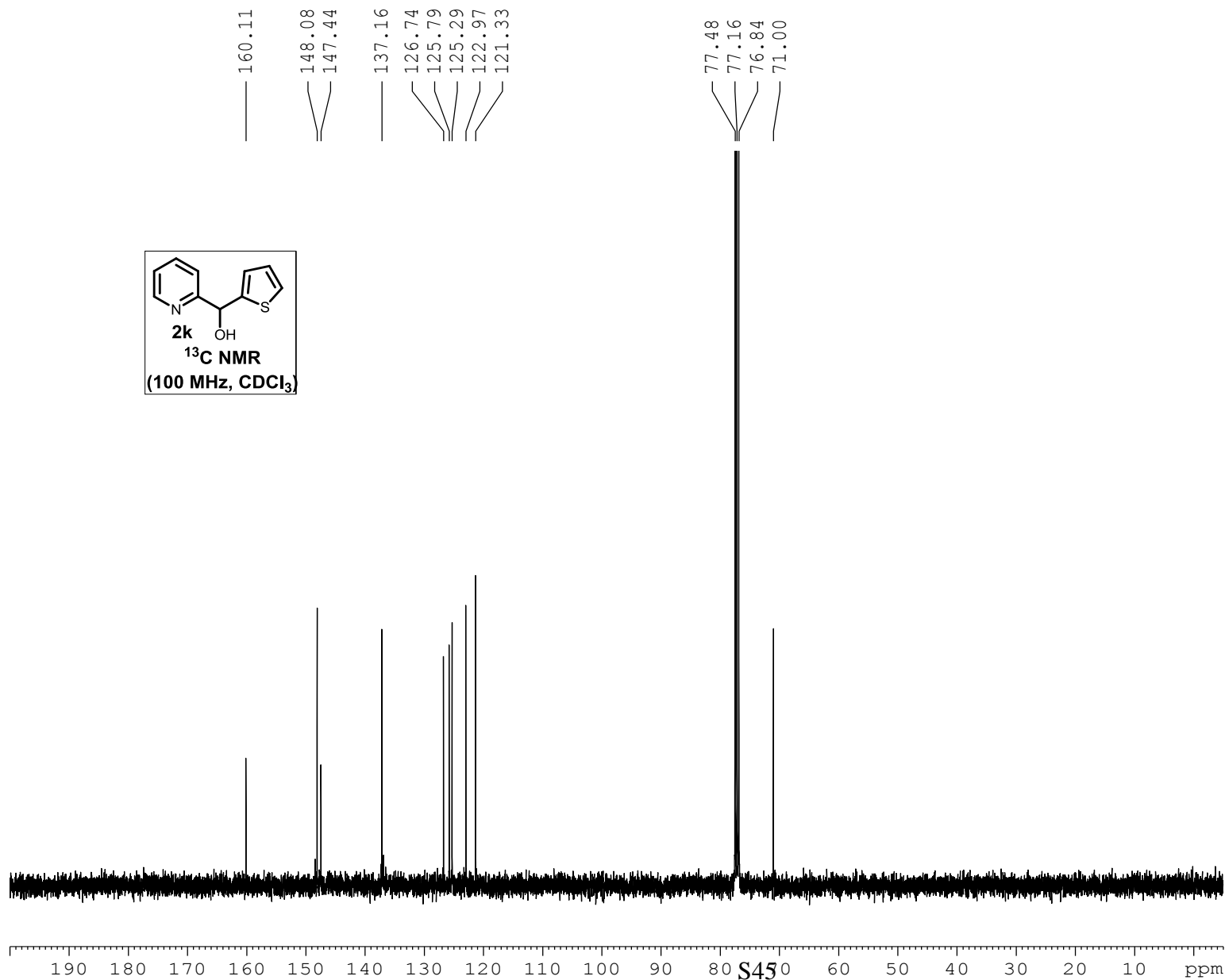
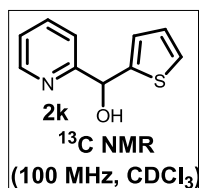
Current Data Parameters  
NAME AHK-I-98  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110227  
Time 19.33  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 228  
DW 60.800 usec  
DE 6.00 usec  
TE 294.9 K  
D1 1.00000000 sec  
TD0 1

----- CHANNEL f1 -----  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300041 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





Current Data Parameters  
NAME AHK-I-90 A  
EXPNO 2  
PROCNO 1

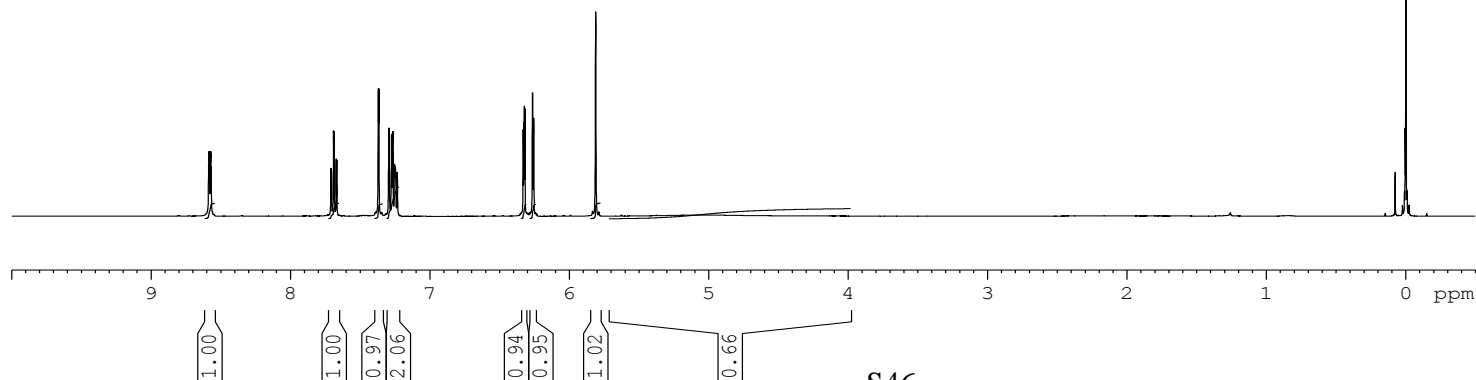
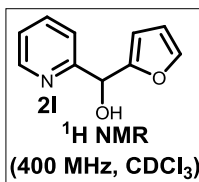
F2 - Acquisition Parameters  
Date\_ 20110311  
Time 12.07  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 2050  
DW 20.800 usec  
DE 6.00 usec  
TE 295.5 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127553 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

8.585  
8.582  
8.573  
8.570  
8.567  
7.708  
7.704  
7.689  
7.685  
7.670  
7.666  
7.369  
7.367  
7.365  
7.363  
7.292  
7.292  
7.273  
7.272  
7.263  
7.252  
7.246  
7.233  
7.233  
6.329  
6.325  
6.321  
6.316  
6.262  
6.254  
5.809  
5.056



-0.000

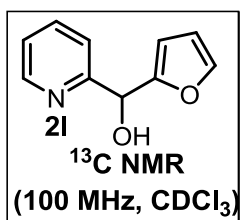


Current Data Parameters  
NAME AHK-I-160 A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110711  
Time\_ 10.16  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 181  
DW 60.800 usec  
DE 6.00 usec  
TE 295.9 K  
D1 1.00000000 sec  
TD0 1

----- CHANNEL f1 -----  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300027 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



158.16  
155.28  
148.15  
142.81  
137.05  
123.04  
121.44  
110.37  
107.73

77.48  
77.16  
76.84  
68.79

0.11



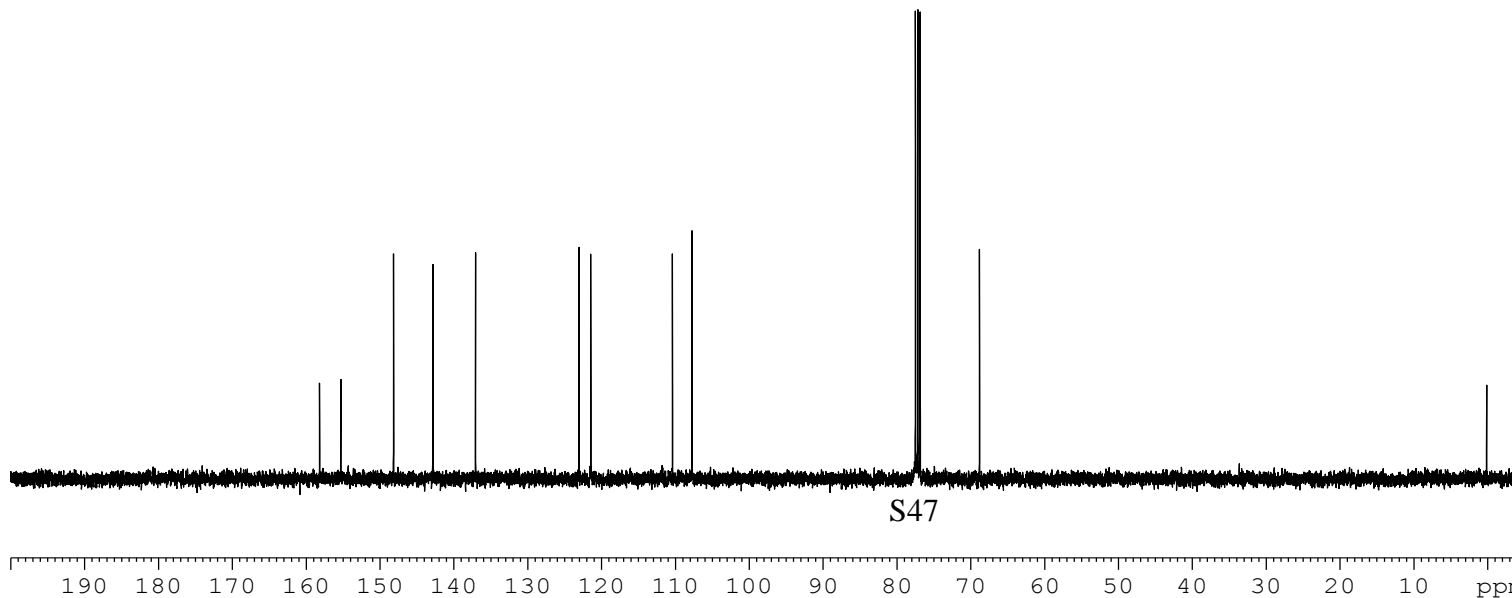
Current Data Parameters  
NAME AHK-I-160 A  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110711  
Time\_ 10.20  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 83  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 812  
DW 20.800 usec  
DE 6.00 usec  
TE 296.5 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127566 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40





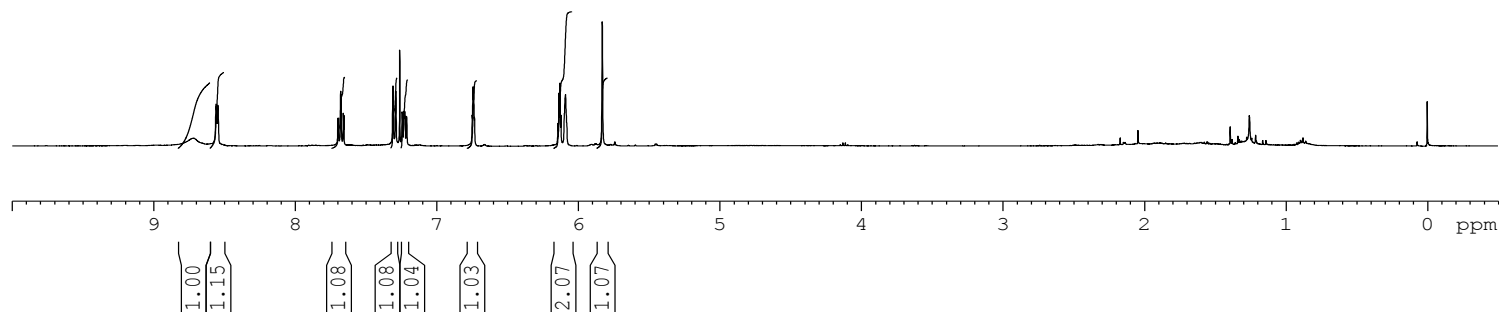
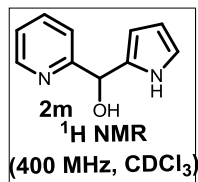
Current Data Parameters  
NAME AHK-I-33-B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100524  
Time\_ 16.44  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.250967 Hz  
AQ 1.9923444 sec  
RG 406  
DW 60.800 usec  
DE 6.00 usec  
TE 295.0 K  
D1 2.00000000 sec  
TD0 1

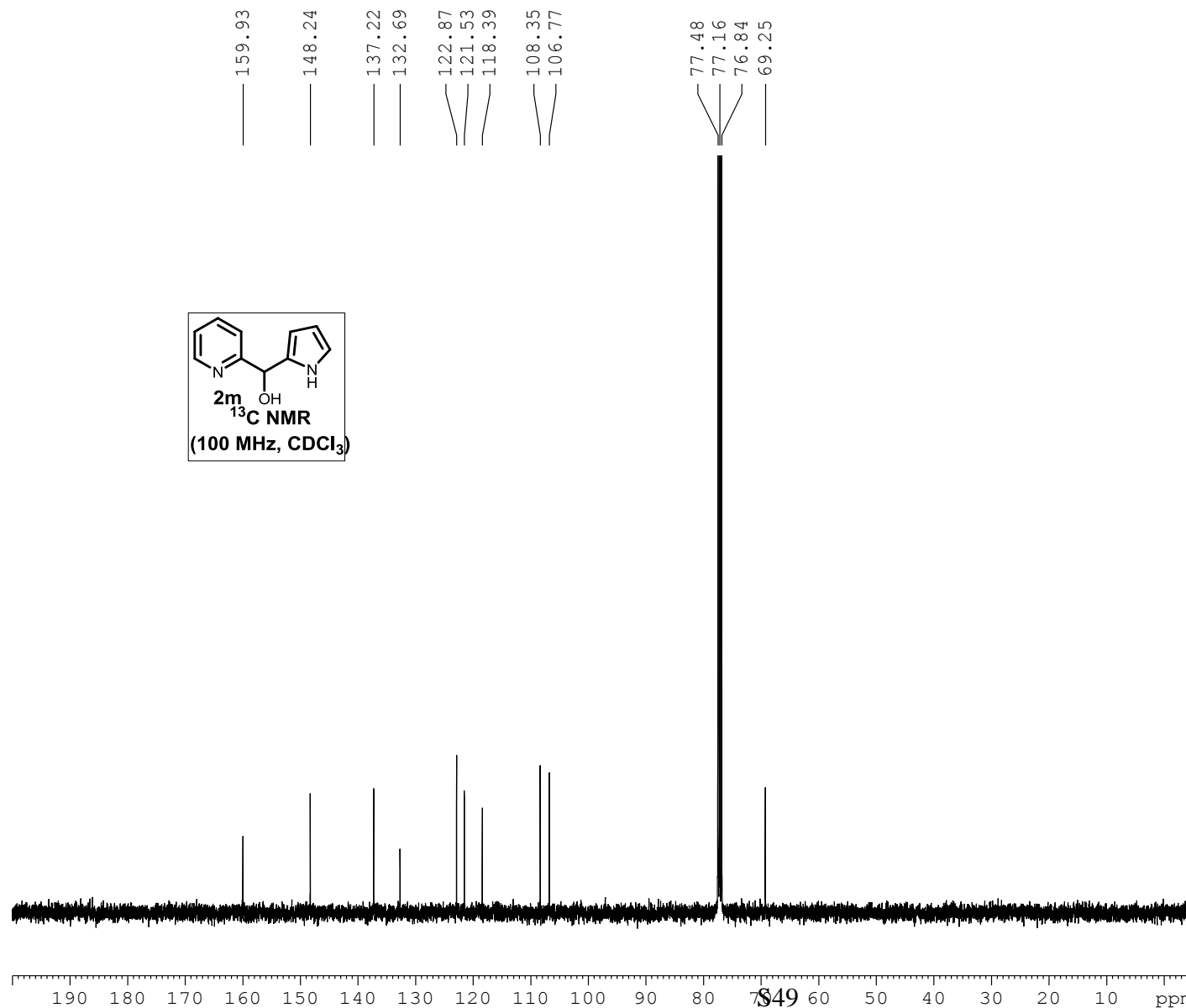
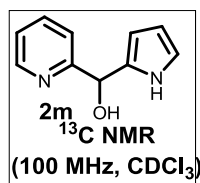
==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300043 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

8.715  
8.557  
8.545  
7.697  
7.693  
7.678  
7.674  
7.659  
7.654  
7.307  
7.288  
7.260  
7.243  
7.241  
7.231  
7.228  
7.224  
7.212  
7.210  
6.748  
6.741  
6.738  
6.731  
6.143  
6.136  
6.128  
6.121  
6.097  
6.088  
5.829







Current Data Parameters  
NAME AHK-I-33-B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100524  
Time\_ 16.46  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 512  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1030  
DW 20.800 usec  
DE 6.00 usec  
TE 295.2 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127554 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



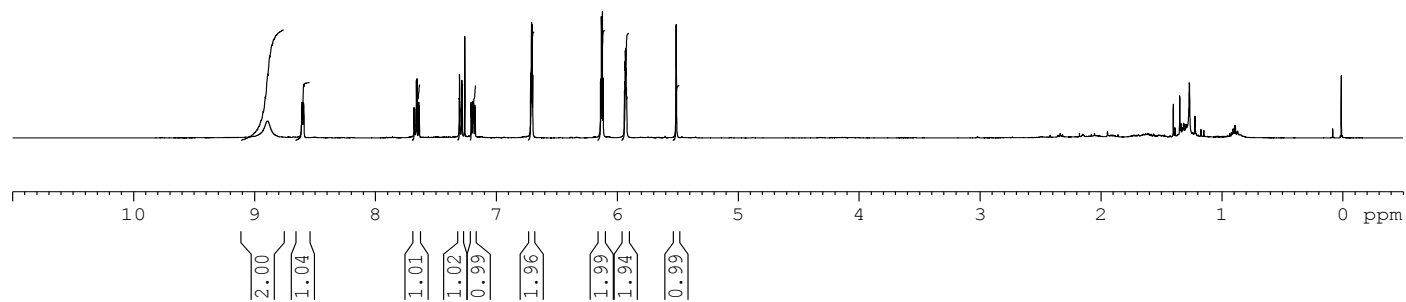
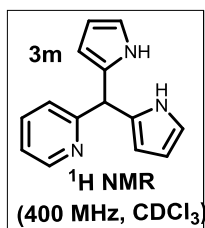
Current Data Parameters  
NAME AHK-I-33-A  
EXPNO 1  
PROCNO 1

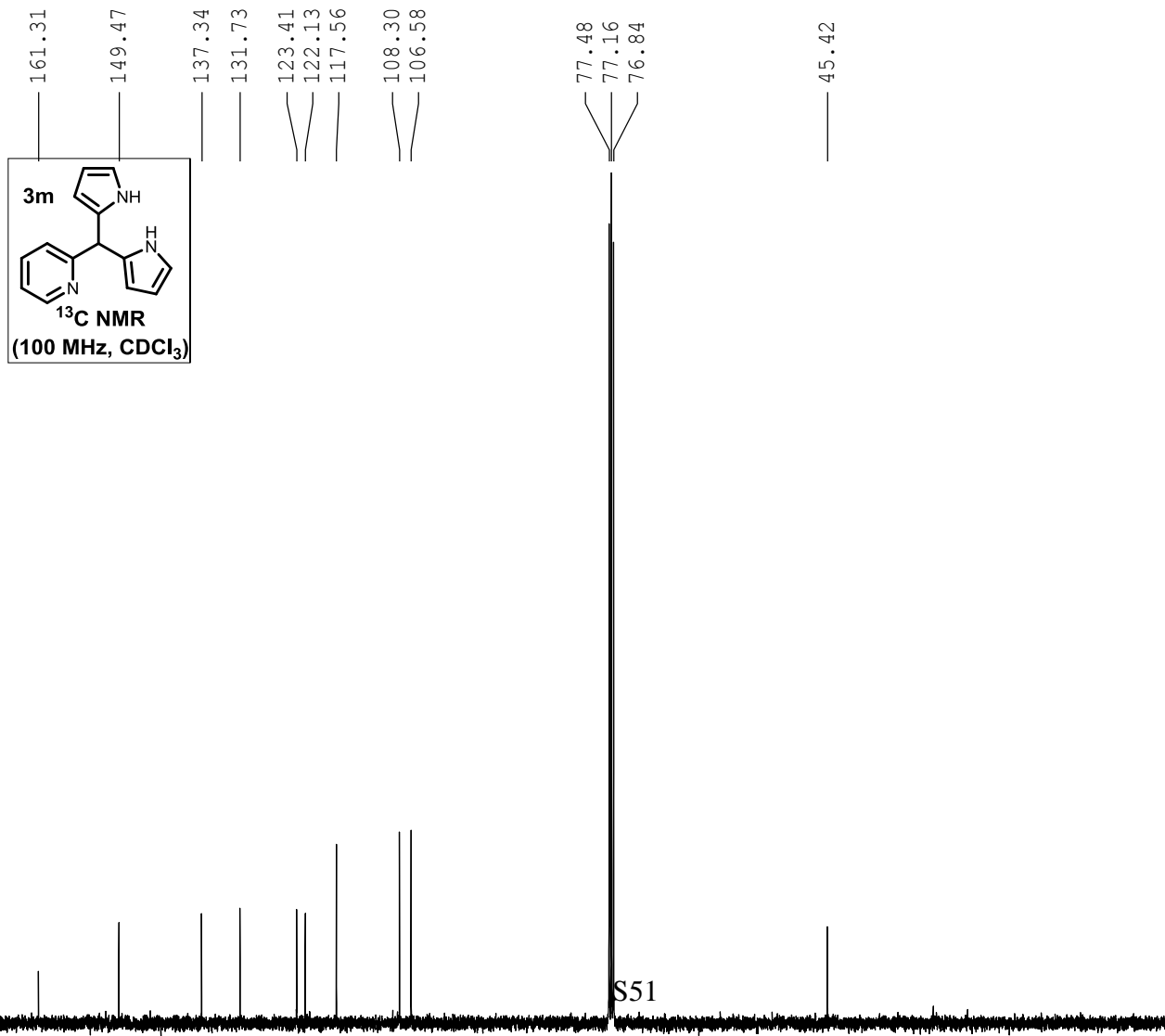
F2 - Acquisition Parameters  
Date\_ 20100524  
Time 12.26  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 32768  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.250967 Hz  
AQ 1.9923444 sec  
RG 362  
DM 60.800 usec  
DE 6.00 usec  
TE 295.4 K  
D1 2.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300043 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

8.595  
7.679  
7.674  
7.660  
7.655  
7.641  
7.636  
7.305  
7.303  
7.301  
7.286  
7.283  
7.281  
7.260  
7.207  
7.205  
7.195  
7.192  
7.189  
7.186  
7.176  
7.174  
6.715  
6.711  
6.708  
6.705  
6.702  
6.698  
6.137  
6.130  
6.122  
6.115  
5.939  
5.938  
5.936  
5.934  
5.932  
5.930  
5.928  
5.926  
5.924  
5.921  
5.920  
5.511





Current Data Parameters  
NAME AHK-I-33-A  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20100524  
Time\_ 12.32  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 434  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 50.8  
DW 20.800 usec  
DE 6.00 usec  
TE 295.8 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

=====  
CHANNEL f1  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

=====  
CHANNEL f2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127561 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



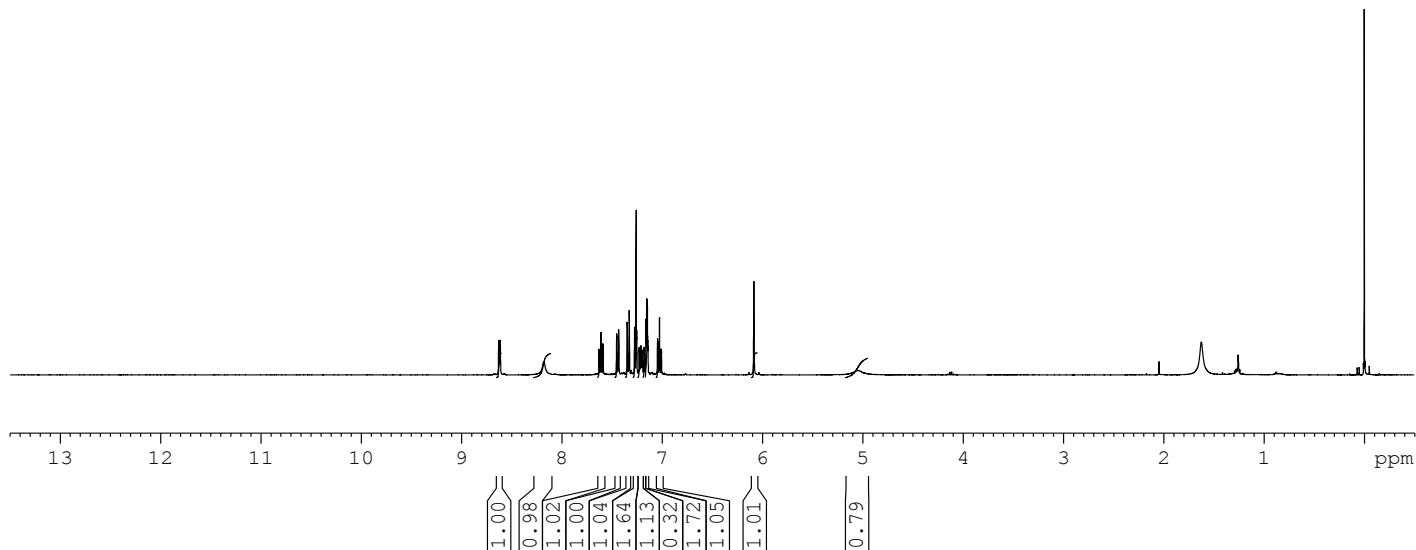
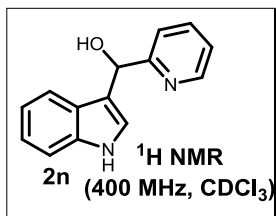
Current Data Parameters  
NAME AHK-I-145 B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110616  
Time 11.32  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 512  
DW 60.800 usec  
DE 6.00 usec  
TE 296.0 K  
D1 1.0000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300048 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

8.629  
8.626  
8.623  
8.617  
8.614  
8.611  
8.179  
7.628  
7.624  
7.609  
7.605  
7.590  
7.586  
7.452  
7.450  
7.432  
7.430  
7.348  
7.347  
7.328  
7.326  
7.324  
7.273  
7.271  
7.258  
7.253  
7.252  
7.228  
7.215  
7.214  
7.213  
7.209  
7.198  
7.197  
7.180  
7.177  
7.162  
7.159  
7.156  
7.152  
7.146  
7.142  
7.139  
7.045  
7.043  
7.027  
7.025  
7.023  
7.008  
7.005  
6.084  
5.058





Current Data Parameters  
NAME AHK-I-145 B  
EXPNO 1  
PROCNO 1

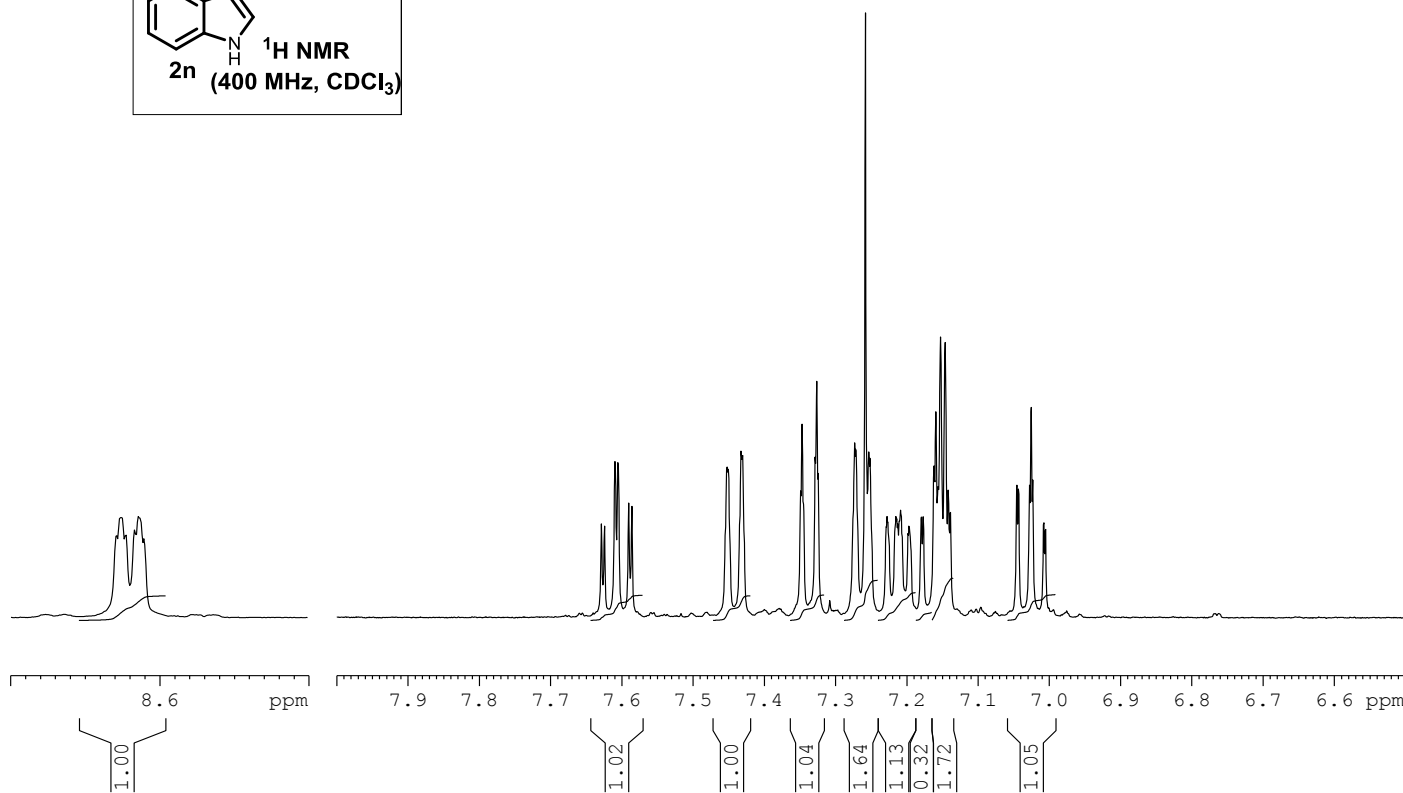
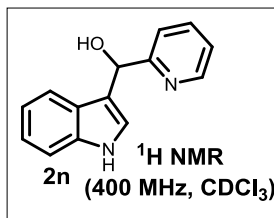
F2 - Acquisition Parameters  
Date\_ 20110616  
Time\_ 11.32  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 512  
DW 60.800 usec  
DE 6.00 usec  
TE 296.0 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

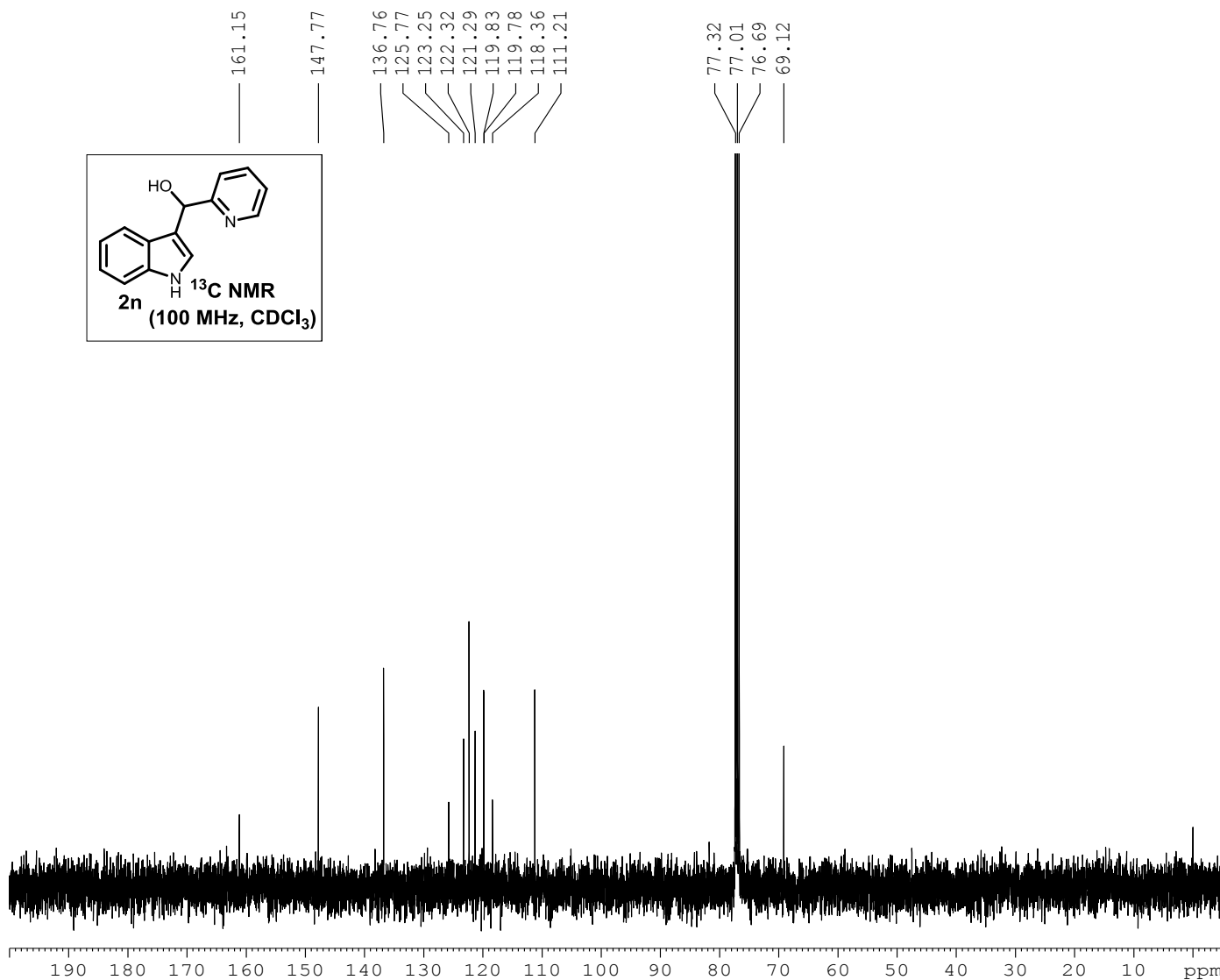
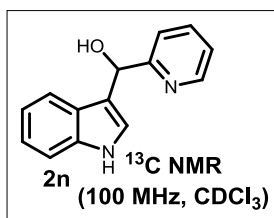
F2 - Processing parameters  
SI 32768  
SF 400.1300048 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

8.629  
8.626  
8.623  
8.617  
8.614  
8.611

7.628  
7.624  
7.620  
7.609  
7.605  
7.590  
7.586  
7.452  
7.450  
7.432  
7.430  
7.348  
7.347  
7.328  
7.326  
7.324  
7.273  
7.271  
7.258  
7.253  
7.252  
7.228  
7.215  
7.214  
7.213  
7.209  
7.198  
7.197  
7.180  
7.177  
7.162  
7.159  
7.156  
7.152  
7.146  
7.142  
7.139  
7.045  
7.043  
7.027  
7.025  
7.023  
7.008  
7.005



S53



Current Data Parameters  
NAME AHK-I-145 B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110616  
Time\_ 11.36  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 57  
DW 20.800 usec  
DE 6.00 usec  
TE 296.7 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 <sup>13</sup>C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 <sup>1</sup>H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

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

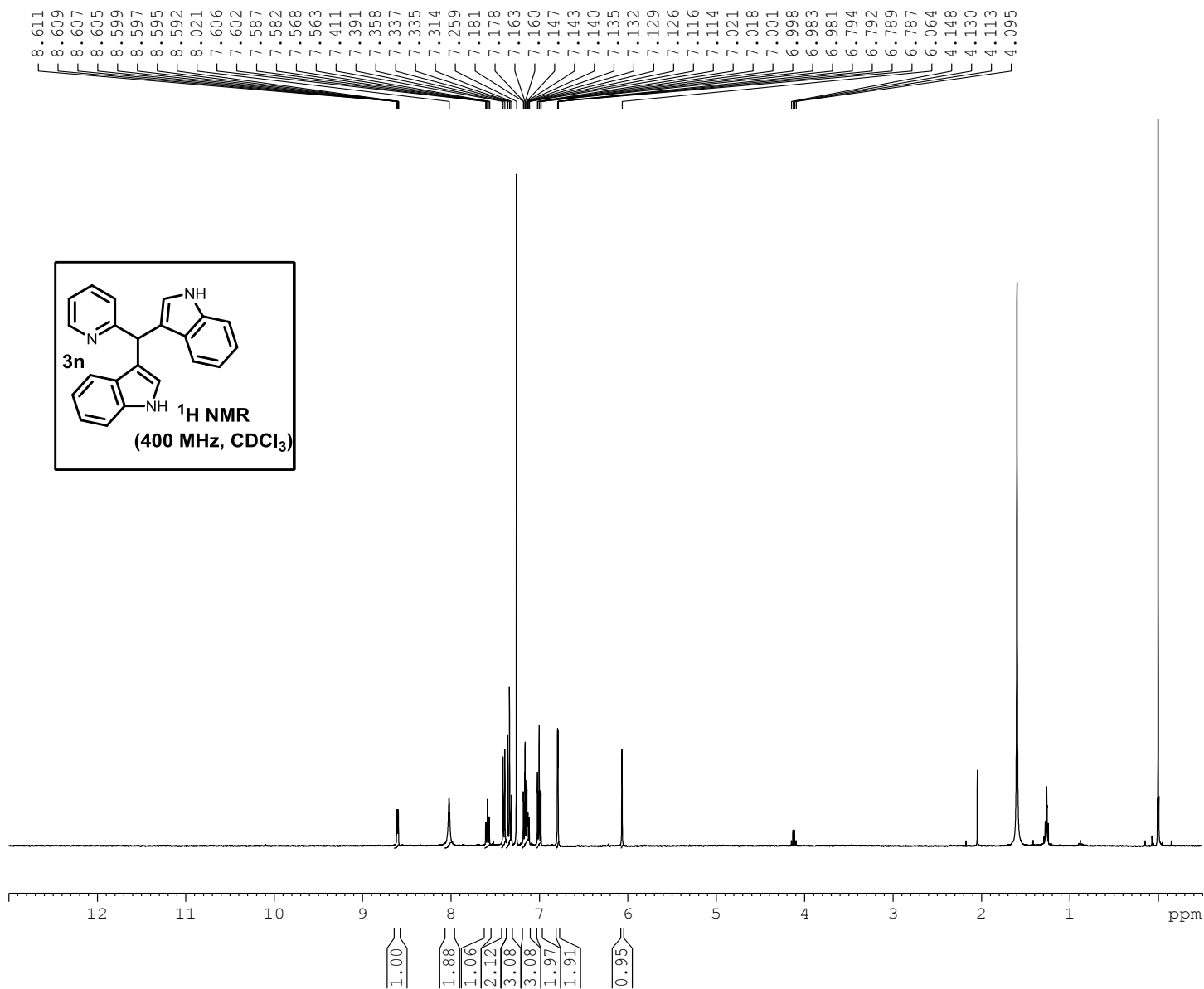


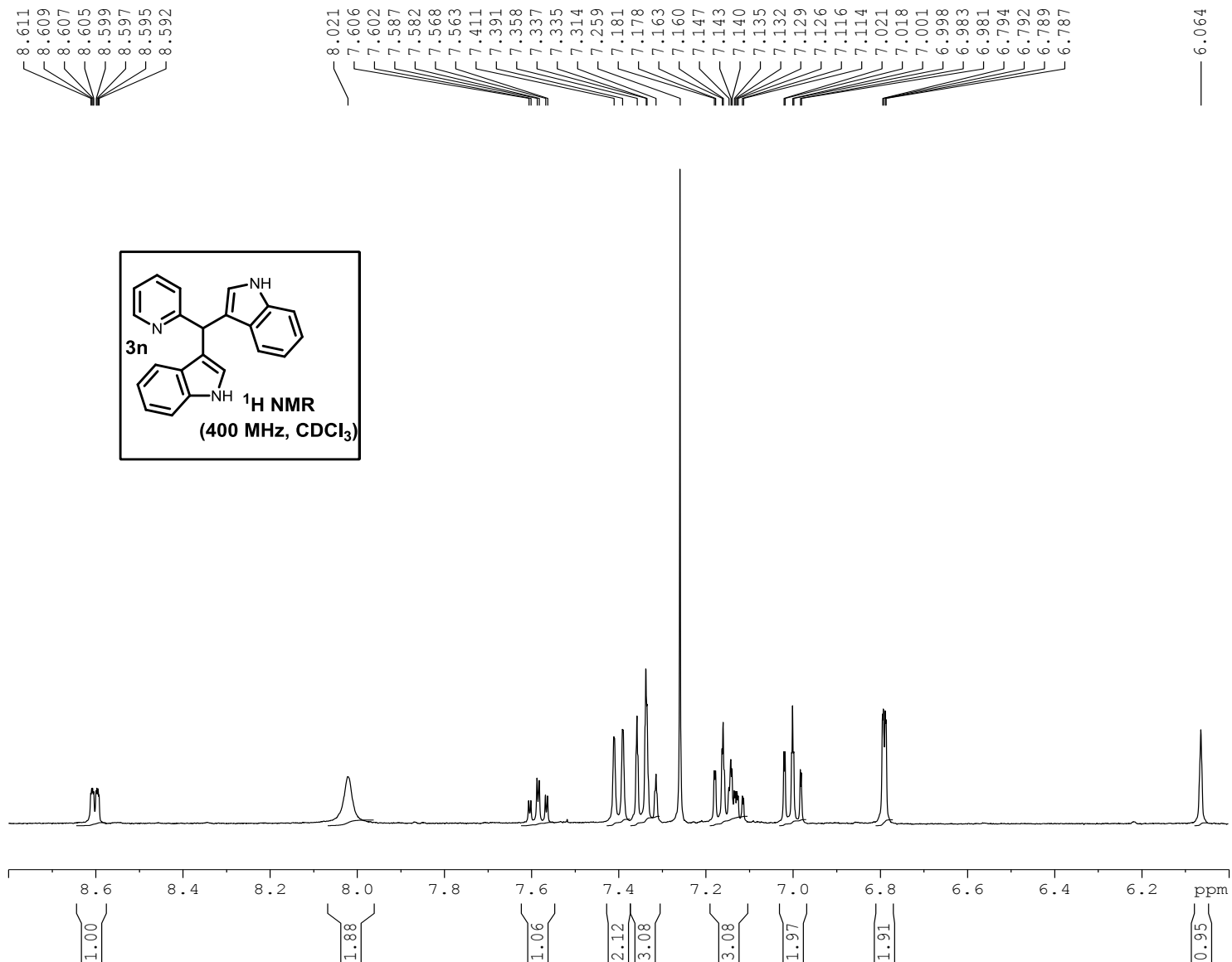
Current Data Parameters  
NAME AHK-I-145A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110616  
Time\_ 14.28  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 575  
DW 60.800 usec  
DE 6.00 usec  
TE 295.5 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300044 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





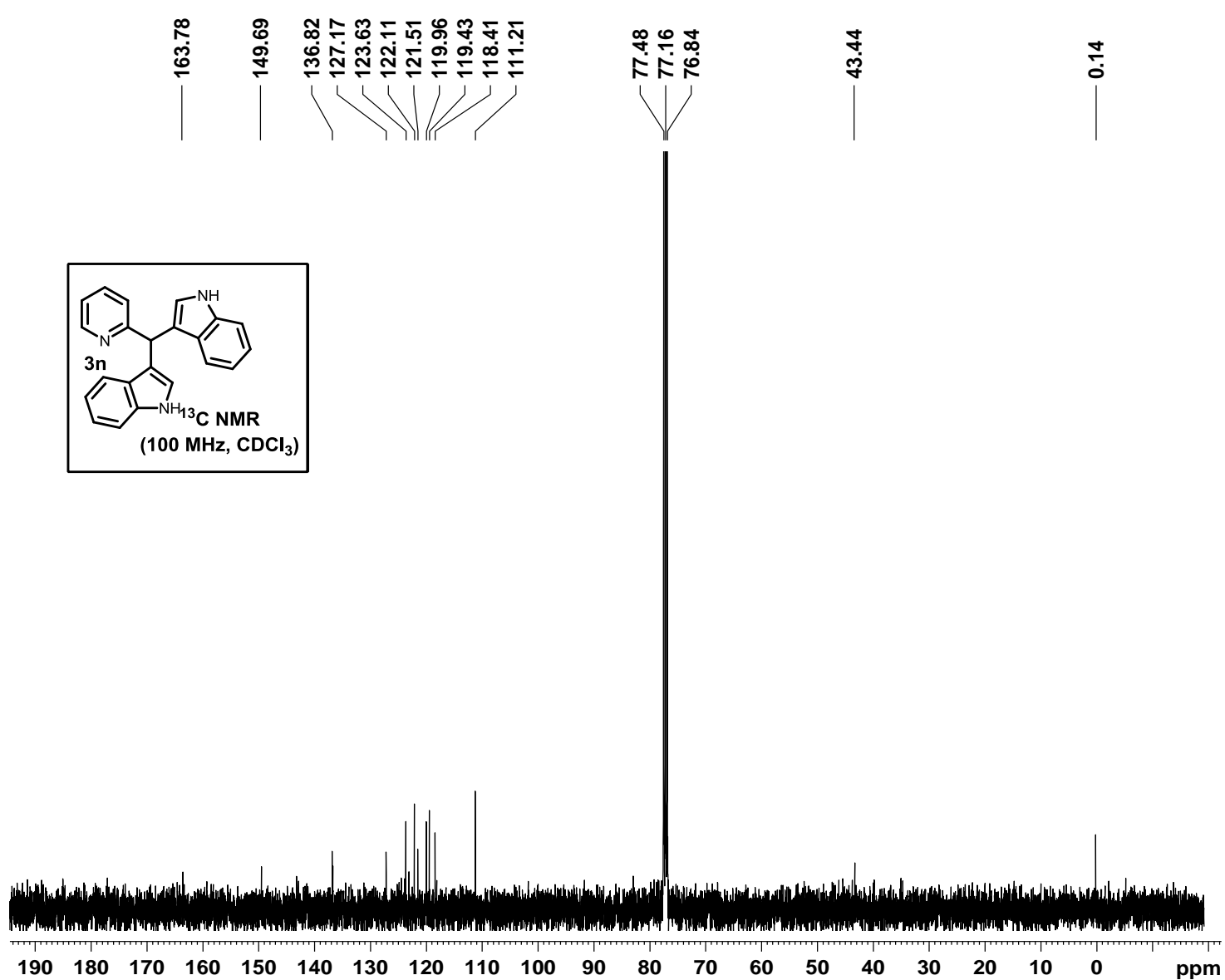
Current Data Parameters  
NAME AHK-I-145A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110616  
Time\_ 14.28  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 575  
DW 60.800 usec  
DE 6.00 usec  
TE 295.5 K  
D1 1.0000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300044 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





Current Data Parameters  
NAME AHK-I-145A  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110616  
Time 14.40  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 416  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1030  
DW 20.800 usec  
DE 6.00 usec  
TE 296.1 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.89999998 sec  
TD0 1

=====  
CHANNEL f1  
=====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

=====  
CHANNEL f2  
=====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127534 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

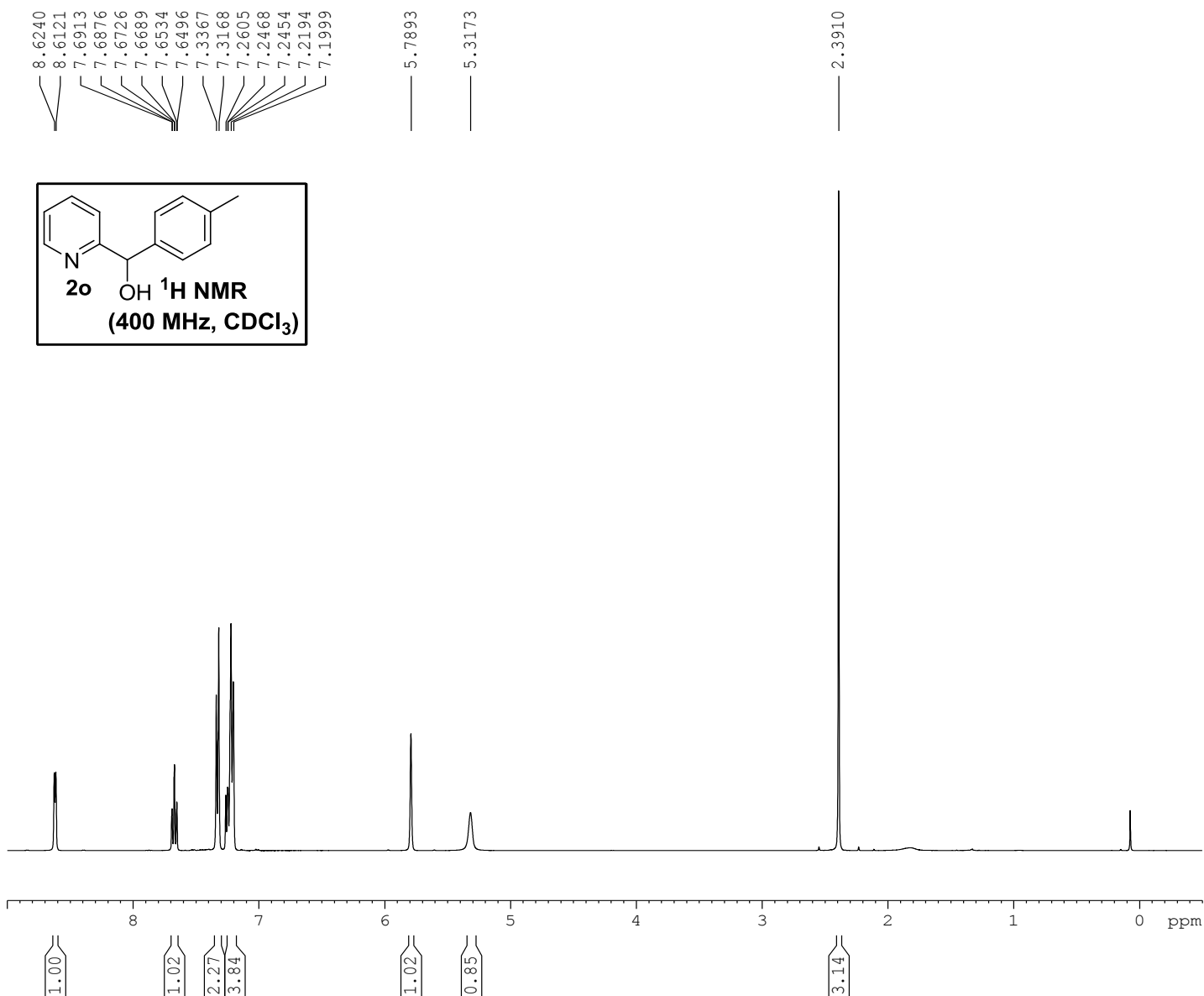


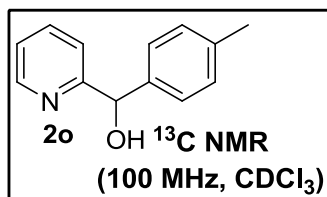
Current Data Parameters  
NAME AHK-I-243B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120214  
Time\_ 11.16  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 181  
DW 60.800 usec  
DE 6.00 usec  
TE 294.6 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1299784 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

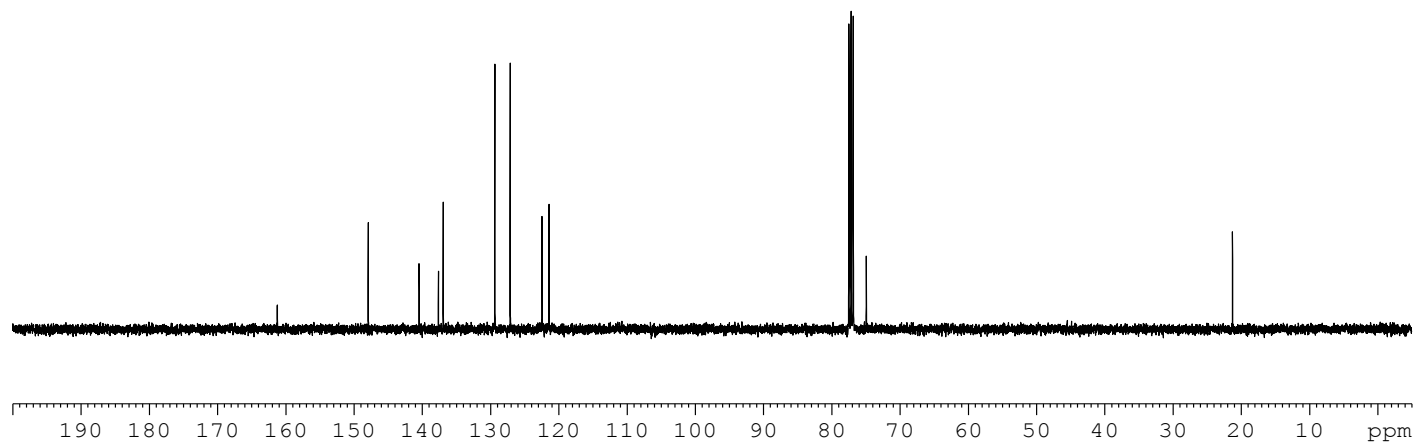




161.24  
147.91  
140.45  
137.62  
136.91  
129.37  
127.12  
122.45  
121.42

77.48  
77.16  
76.84  
74.92

21.26



Current Data Parameters  
NAME AHK-I-243B  
EXPNO 2  
PROCNO 1

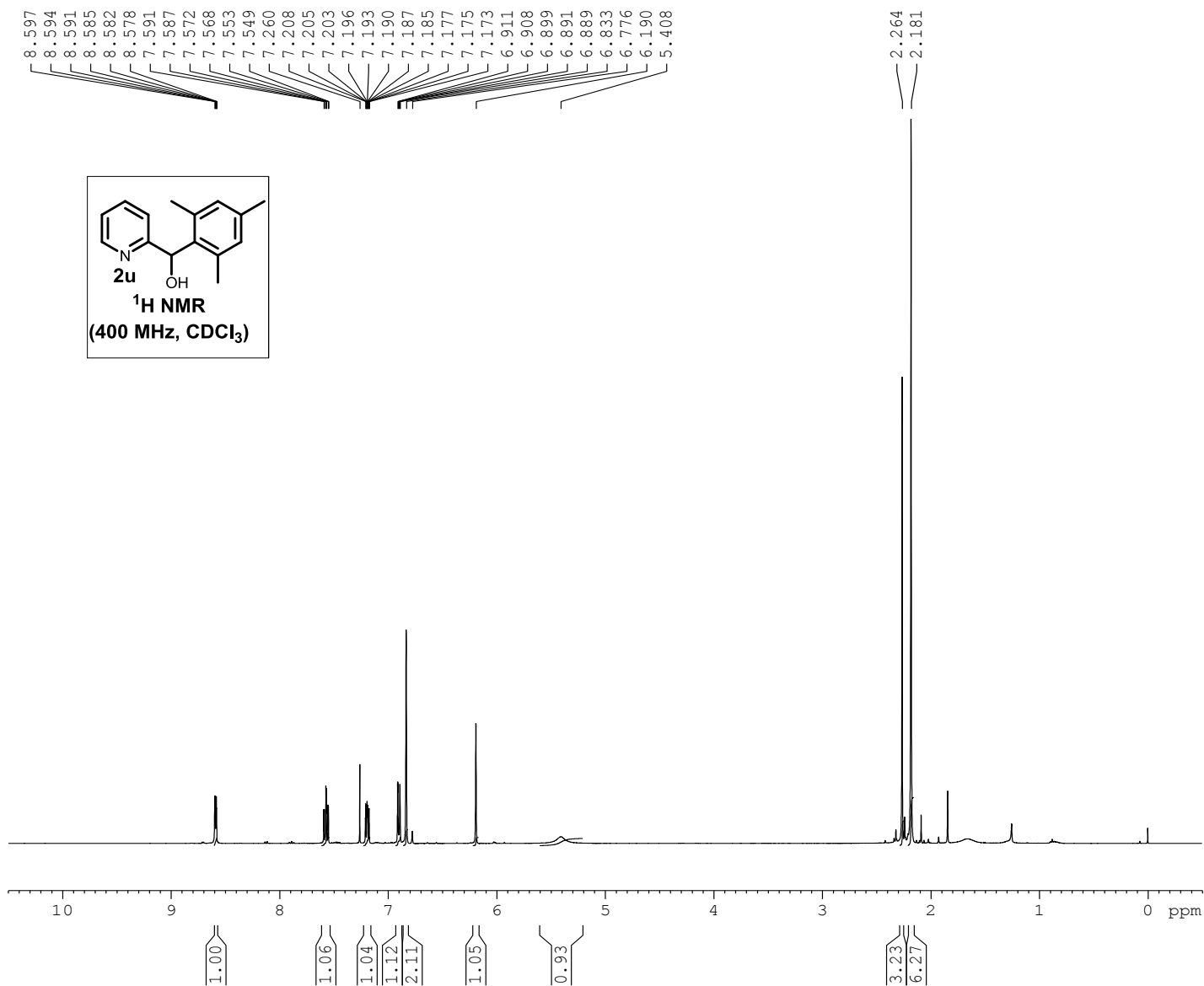
F2 - Acquisition Parameters  
Date\_ 20120214  
Time 11.20  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 127  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1150  
DW 20.800 usec  
DE 6.00 usec  
TE 295.1 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TDO 1

=====  
CHANNEL f1  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

=====  
CHANNEL f2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127575 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

S59

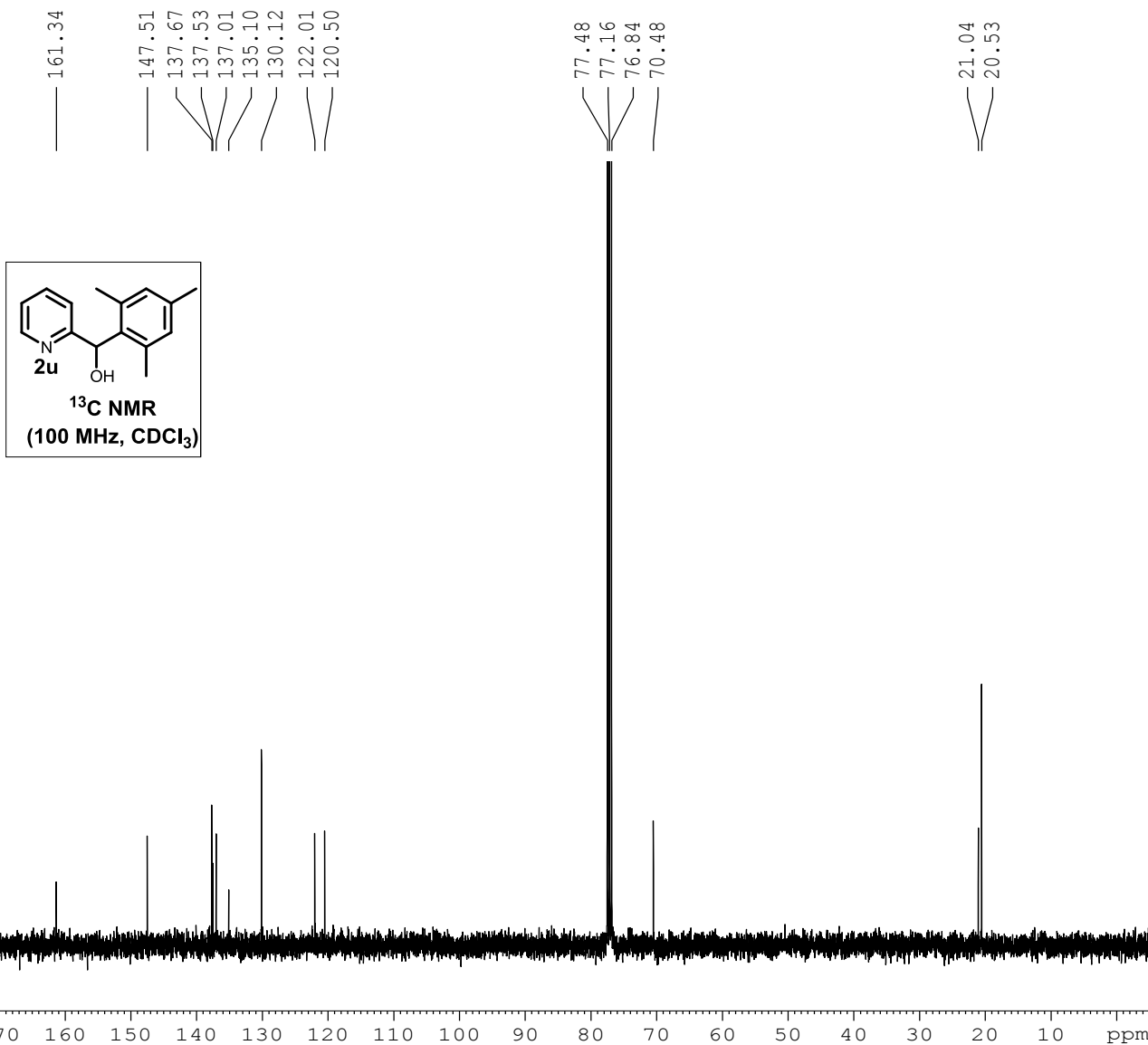


Current Data Parameters  
NAME AHK-I-105B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110429  
Time 14.23  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 12  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 228  
DW 60.800 usec  
DE 6.00 usec  
TE 291.0 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300040 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



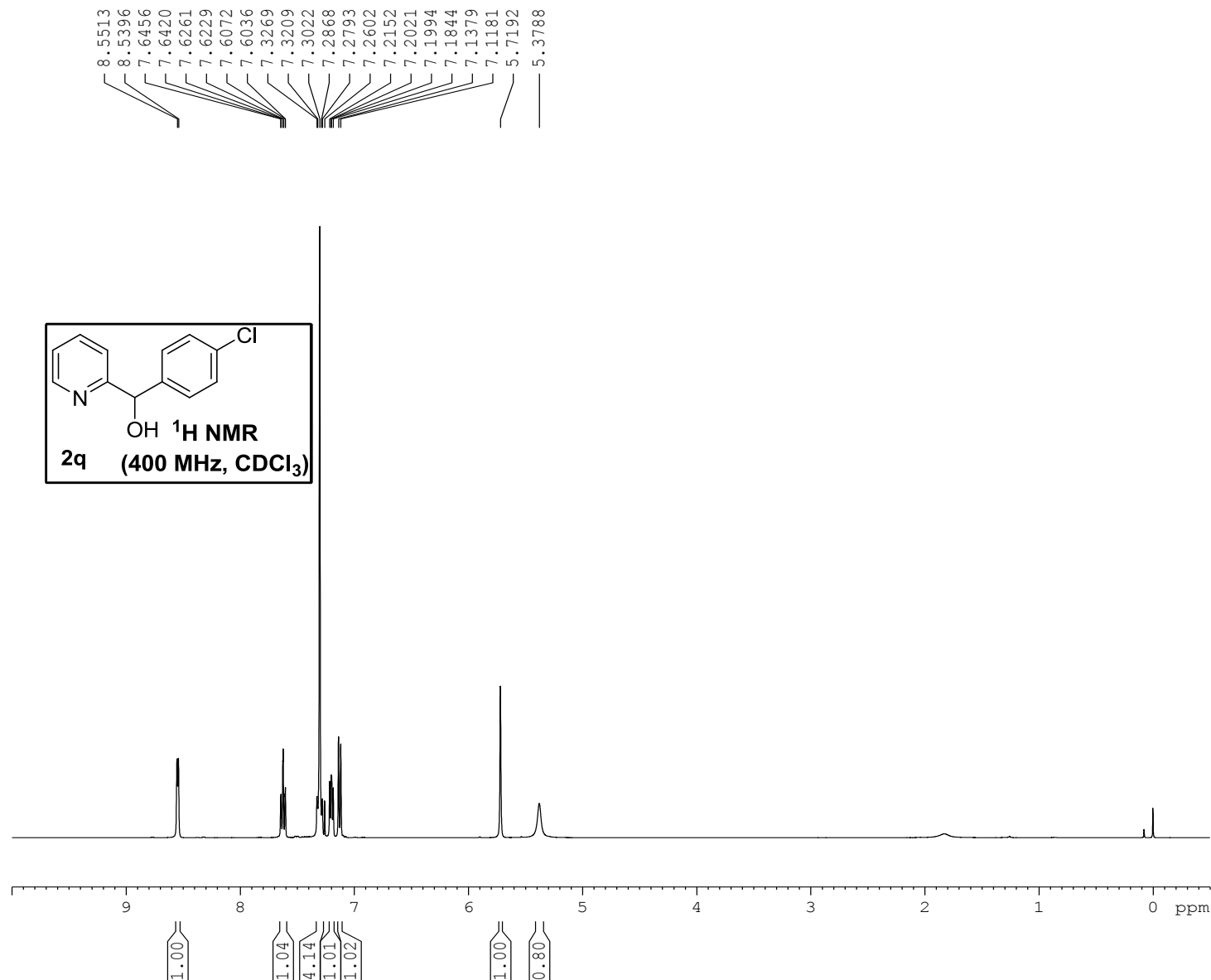
Current Data Parameters  
NAME AHK-I-105B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110429  
Time\_ 14.25  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 107  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 912  
DW 20.800 usec  
DE 6.00 usec  
TE 291.6 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TDO 1

=====  
CHANNEL f1  
=====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

=====  
CHANNEL f2  
=====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127557 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

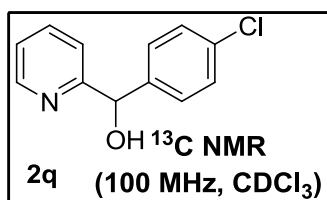


Current Data Parameters  
NAME AHK-I-241B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120214  
Time 11.29  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 181  
DW 60.800 usec  
DE 6.00 usec  
TE 294.5 K  
D1 1.00000000 sec  
TD0 1

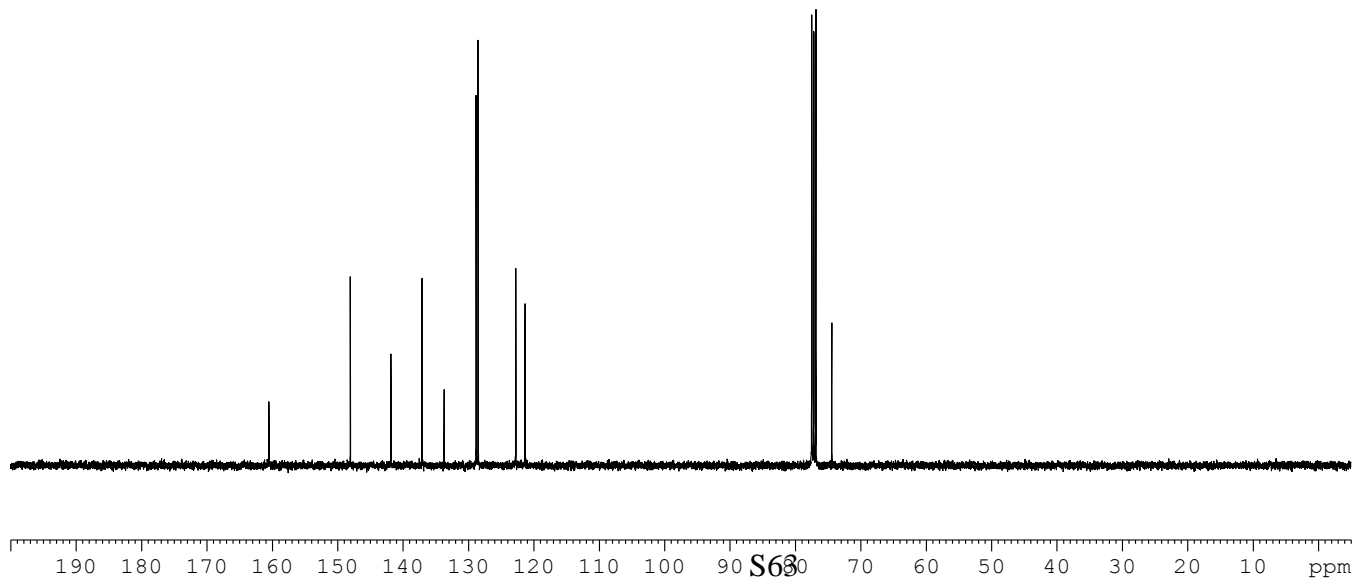
===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300037 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
FC 1.00



160.52  
148.06  
141.84  
137.10  
133.70  
128.83  
128.51  
122.75  
121.34

77.48  
77.16  
76.84  
74.42



Current Data Parameters  
NAME AHK-I-241B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120214  
Time 11.45  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1150  
DW 20.800 usec  
DE 6.00 usec  
TE 294.6 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.8999999 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127581 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



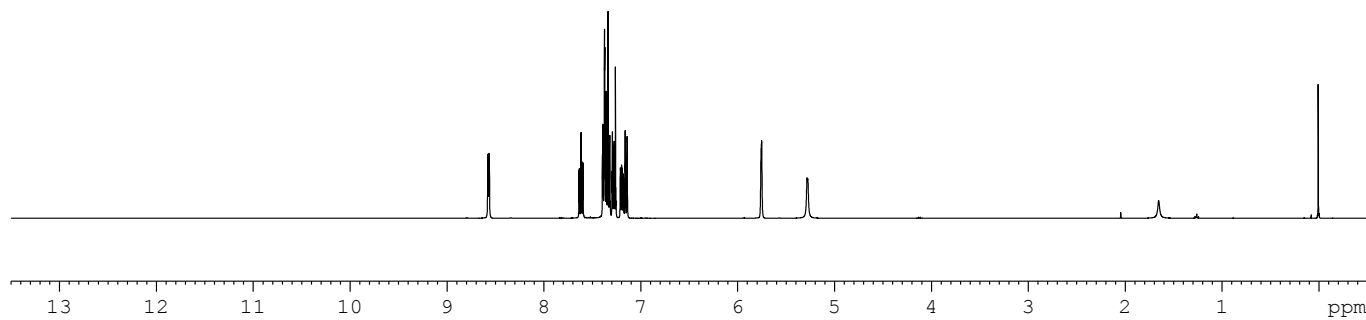
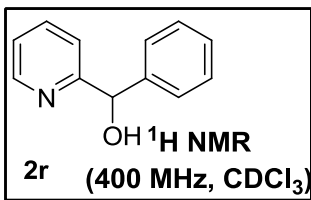
Current Data Parameters  
NAME AHK-I-300-C  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120706  
Time\_ 10.13  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 181  
DW 60.800 usec  
DE 6.00 usec  
TE 297.9 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 11.42 usec  
PL1 -3.00 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300031 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

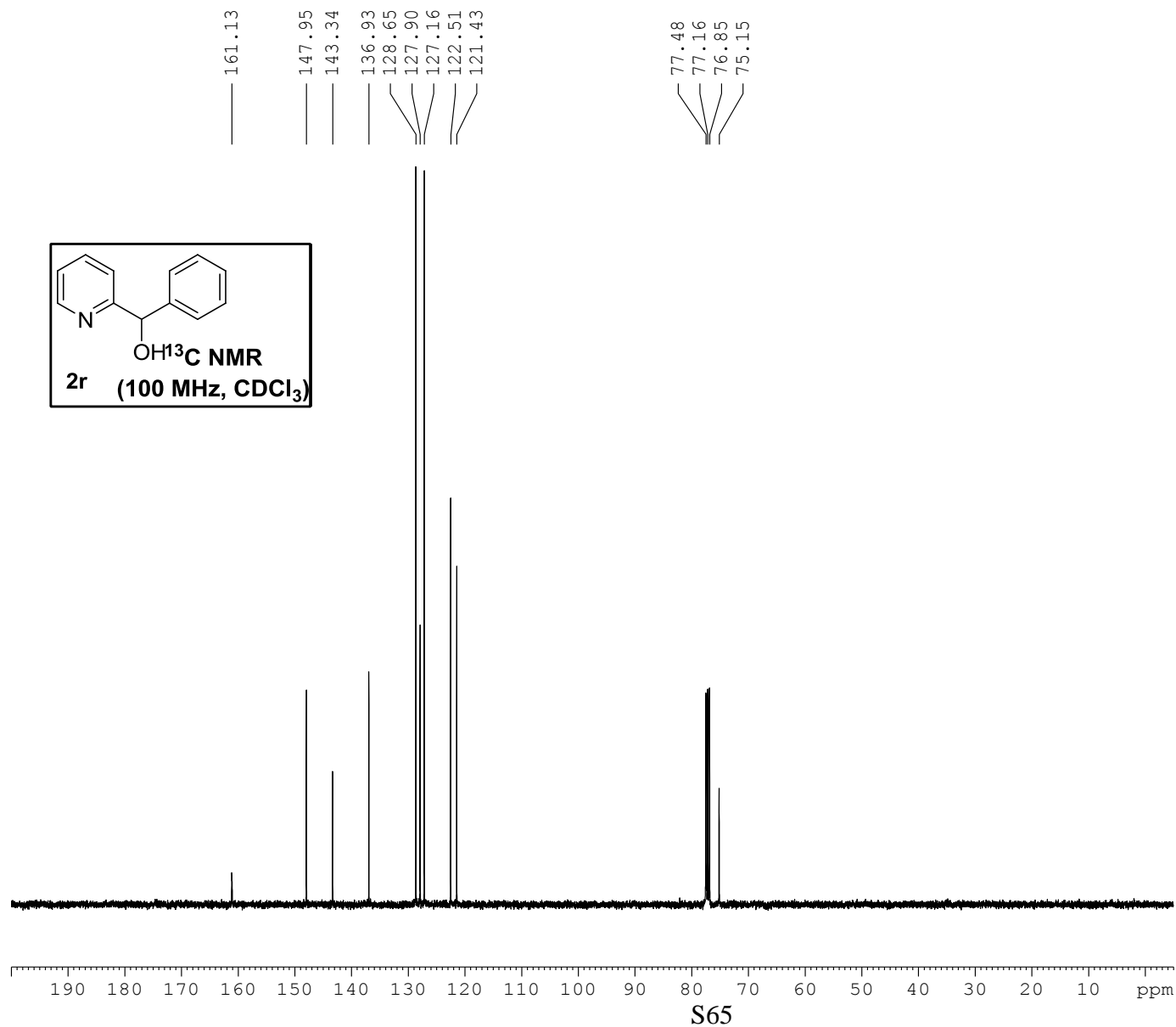
8.5766  
8.5734  
8.5705  
8.5645  
8.5611  
8.5584  
7.6374  
7.6331  
7.6182  
7.6139  
7.5989  
7.5946  
7.3948  
7.3905  
7.3855  
7.3734  
7.3705  
7.3661  
7.3576  
7.3549  
7.3500  
7.3373  
7.3332  
7.3223  
7.3182  
7.3139  
7.2960  
7.2920  
7.2877  
7.2809  
7.2741  
7.2675  
7.2601  
7.2569  
7.2530  
7.2103  
7.2091  
7.1967  
7.1956  
7.1927  
7.1916  
7.1905  
7.1780  
7.1769  
7.1605  
7.1591  
7.1408  
7.1395  
5.7512  
5.2807



1.00  
1.02  
4.05  
1.26  
1.01  
1.00  
0.99  
0.84

S64





Current Data Parameters  
NAME AHK-I-300B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120702  
Time 12.46  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 28.5  
DW 20.800 usec  
DE 6.00 usec  
TE 298.9 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.15 usec  
PL1 0.00 dB  
SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 14.90 dB  
PL13 14.90 dB  
PL2 -3.00 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127589 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

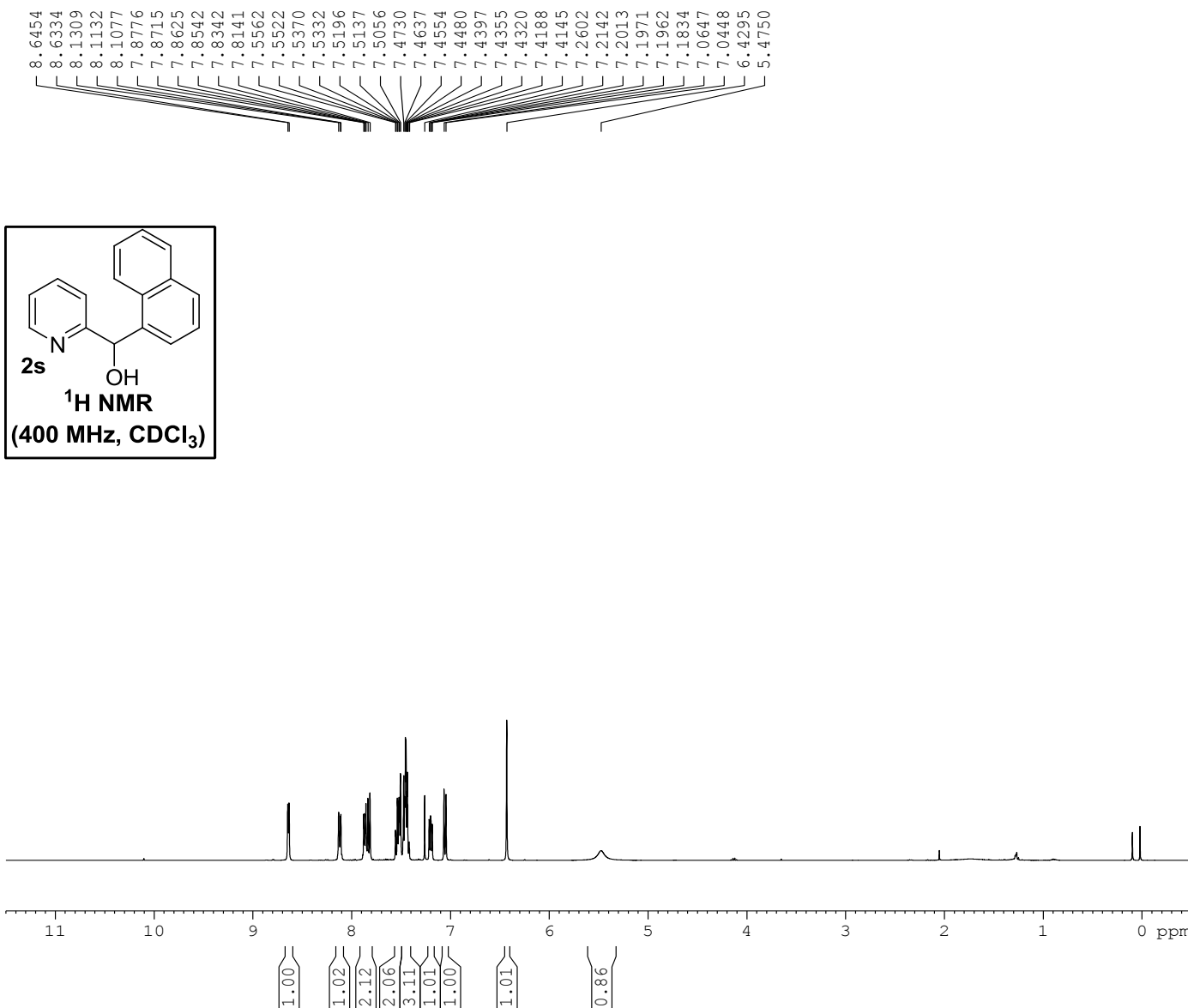


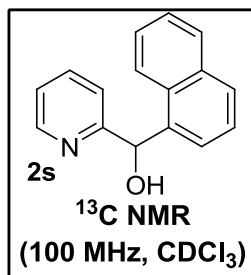
Current Data Parameters  
NAME AHK-I-246B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120222  
Time 10.50  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 203  
DW 60.800 usec  
DE 6.00 usec  
TE 297.7 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

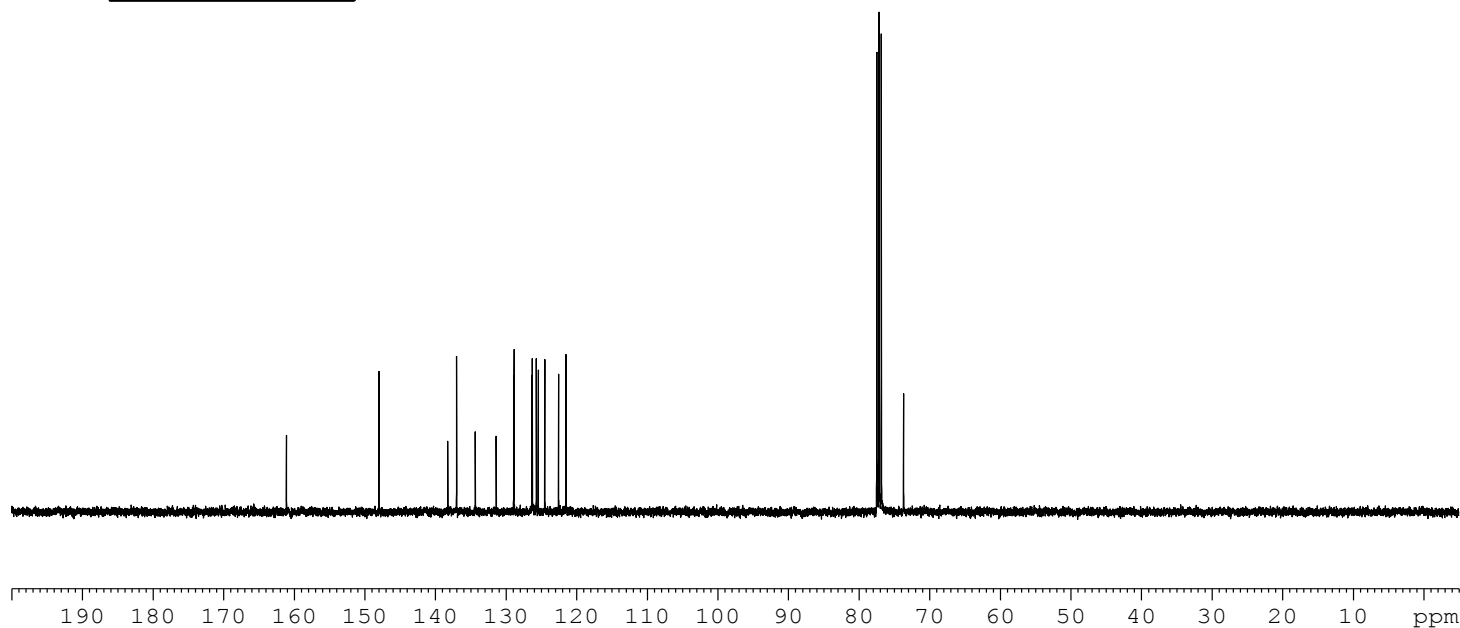
F2 - Processing parameters  
SI 32768  
SF 400.1300036 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





161.09  
147.99  
138.25  
136.99  
134.35  
131.39  
128.92  
128.86  
126.35  
126.28  
125.71  
125.43  
124.51  
122.55  
121.49

77.47  
77.15  
76.84  
73.68



Current Data Parameters  
NAME AHK-I-246B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120222  
Time 11.06  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1150  
DW 20.800 usec  
DE 6.00 usec  
TE 298.0 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

=====  
CHANNEL f1  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

=====  
CHANNEL f2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127574 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



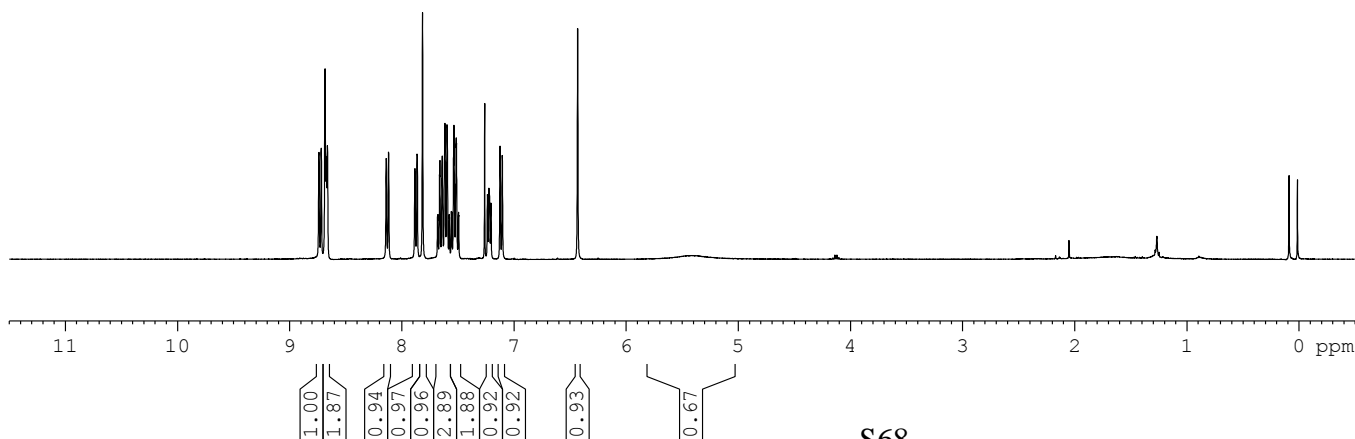
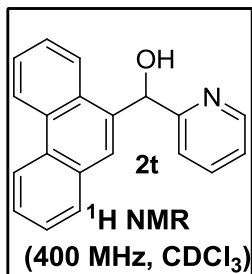
Current Data Parameters  
NAME AHK-I-245B  
EXPNO 1  
PROCNO 1

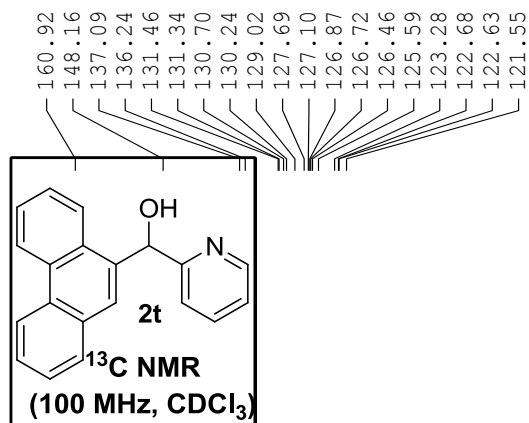
F2 - Acquisition Parameters  
Date\_ 20120222  
Time\_ 11.22  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 287  
DW 60.800 usec  
DE 6.00 usec  
TE 297.6 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300037 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

8.7376  
8.7169  
8.6828  
8.6720  
8.6617  
8.1375  
8.1169  
7.8823  
7.8632  
7.8140  
7.6770  
7.6624  
7.6596  
7.6422  
7.6383  
7.6167  
7.6137  
7.5957  
7.5762  
7.5572  
7.5531  
7.5377  
7.5332  
7.5186  
7.5143  
7.5116  
7.4936  
7.2602  
7.2348  
7.2219  
7.2172  
7.2041  
7.1228  
7.1029  
6.4310  
5.4312





Current Data Parameters  
NAME AHK-I-245B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120222  
Time\_ 11.40  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1150  
DW 20.800 usec  
DE 6.00 usec  
TE 298.0 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

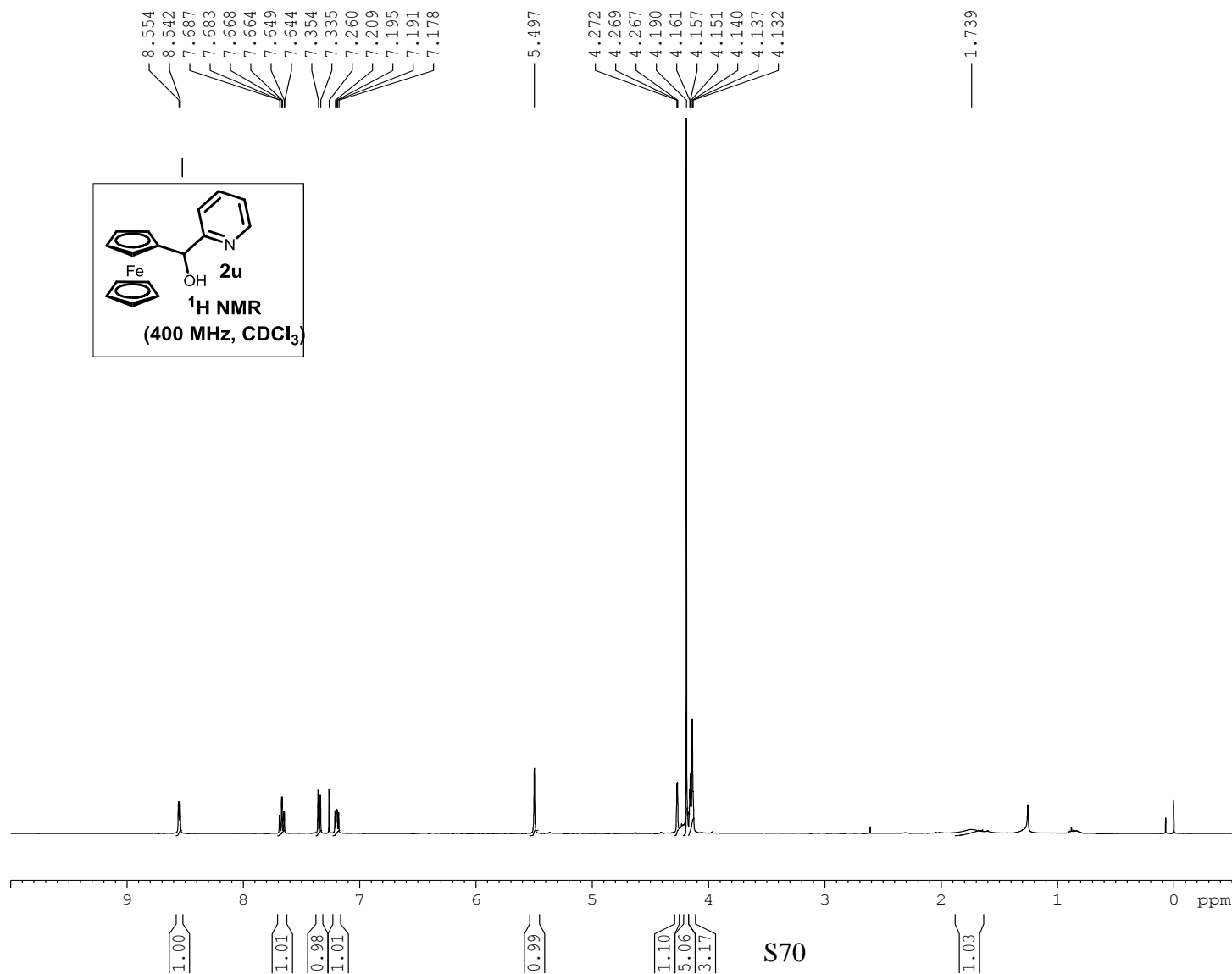
===== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127547 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

S69

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



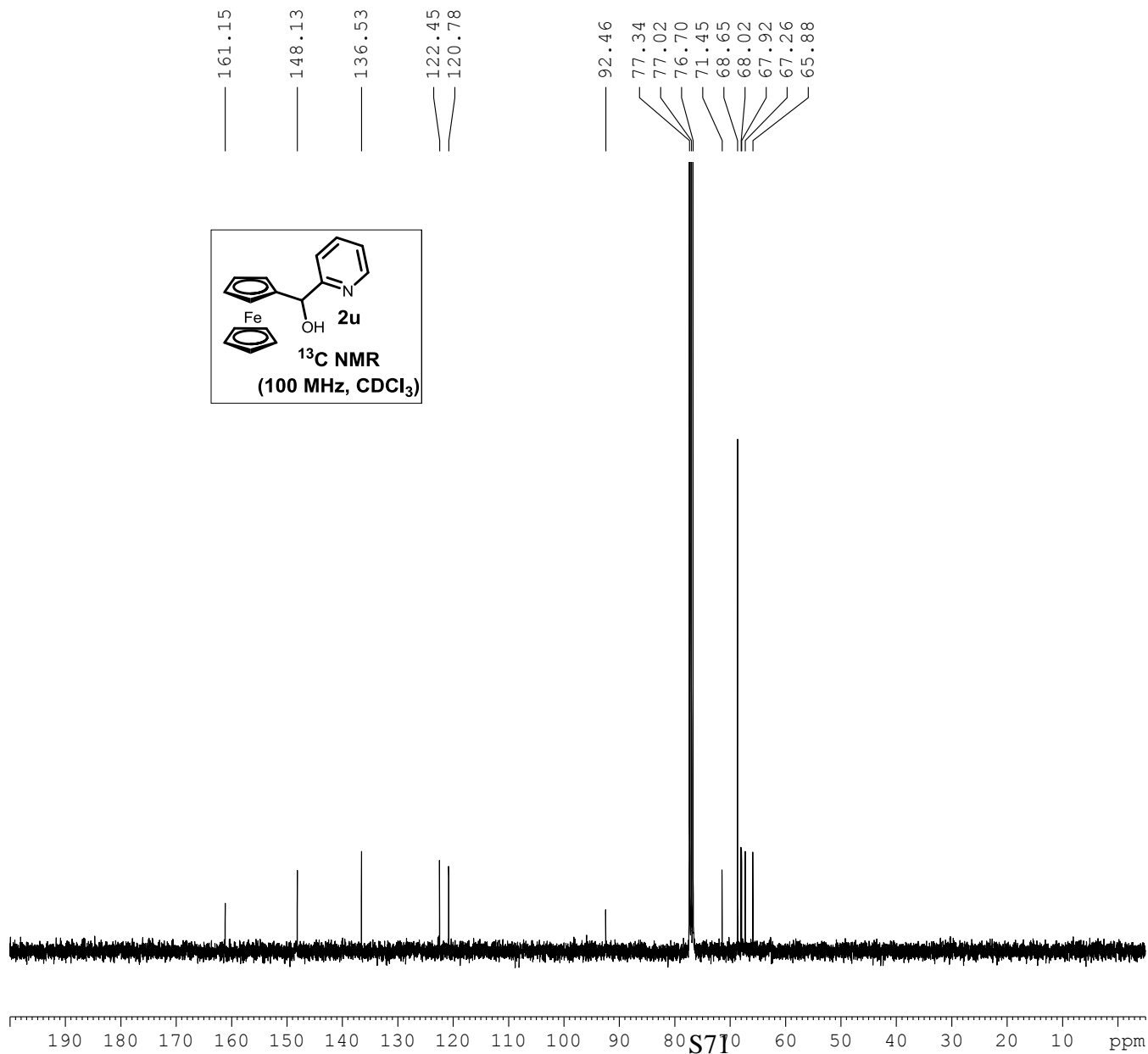
Current Data Parameters  
NAME AHK-I-121 A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110401  
Time\_ 12.13  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 228  
DW 60.800 usec  
DE 6.00 usec  
TE 295.3 K  
D1 1.00000000 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300039 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

S70



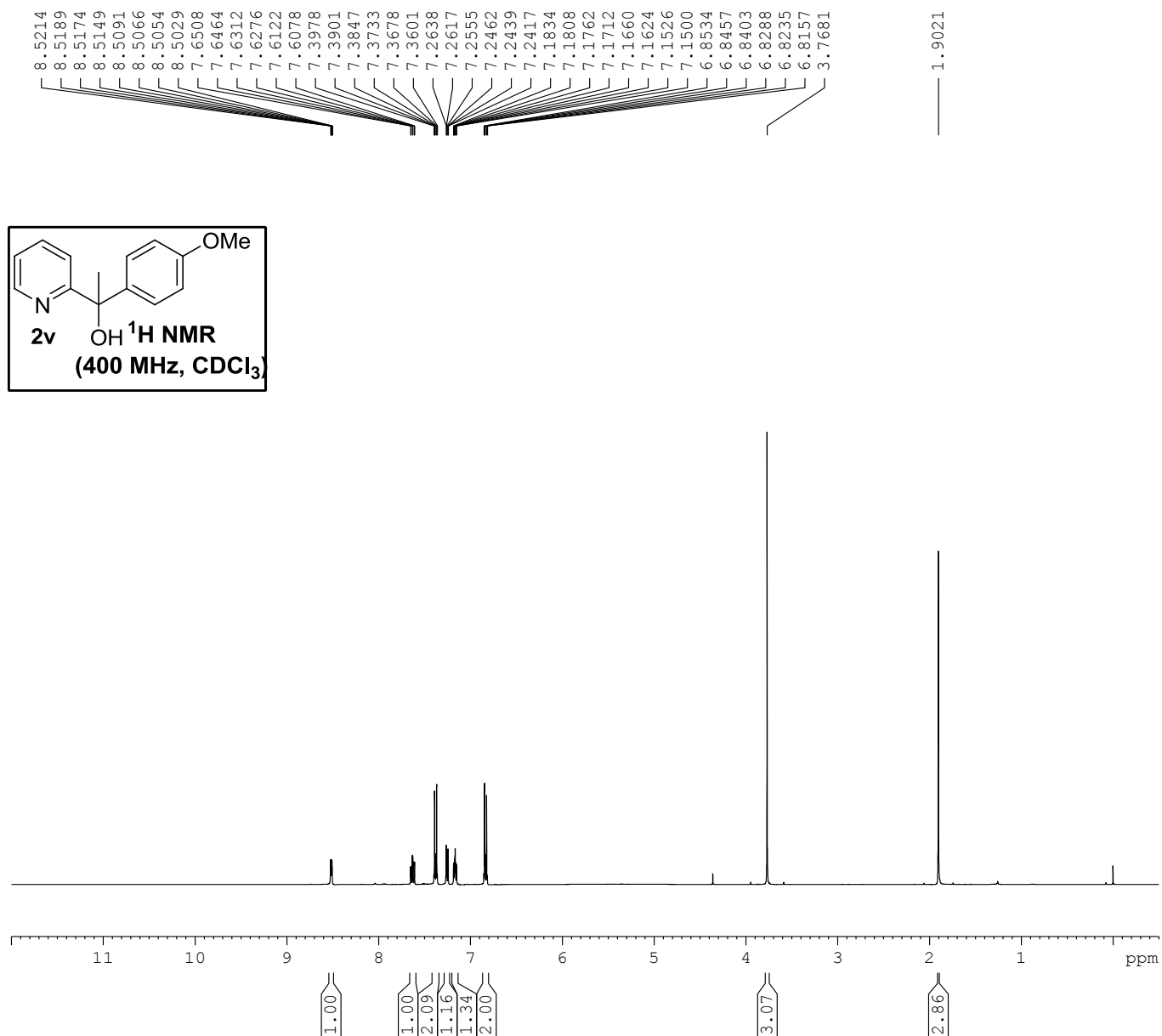
Current Data Parameters  
NAME AHK-I-121 A  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110401  
Time\_ 12.23  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 278  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 50.8  
DW 20.800 usec  
DE 6.00 usec  
TE 295.8 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

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



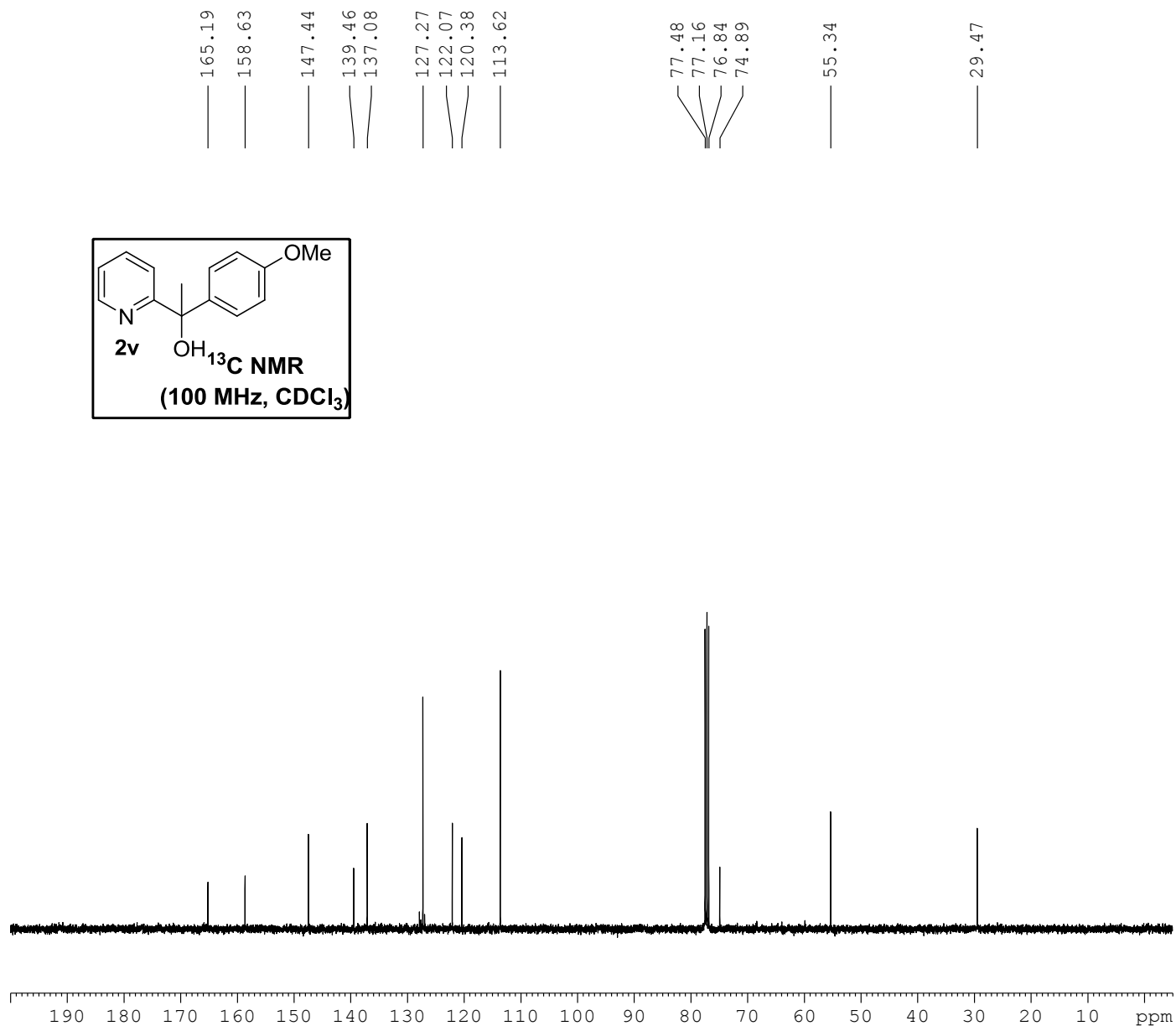
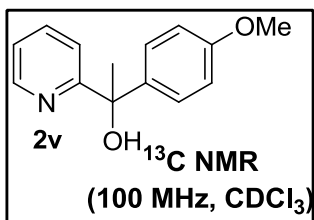
Current Data Parameters  
NAME AHK-I-249C  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120411  
Time\_ 11.45  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 161  
DW 60.800 usec  
DE 6.00 usec  
TE 294.2 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300057 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





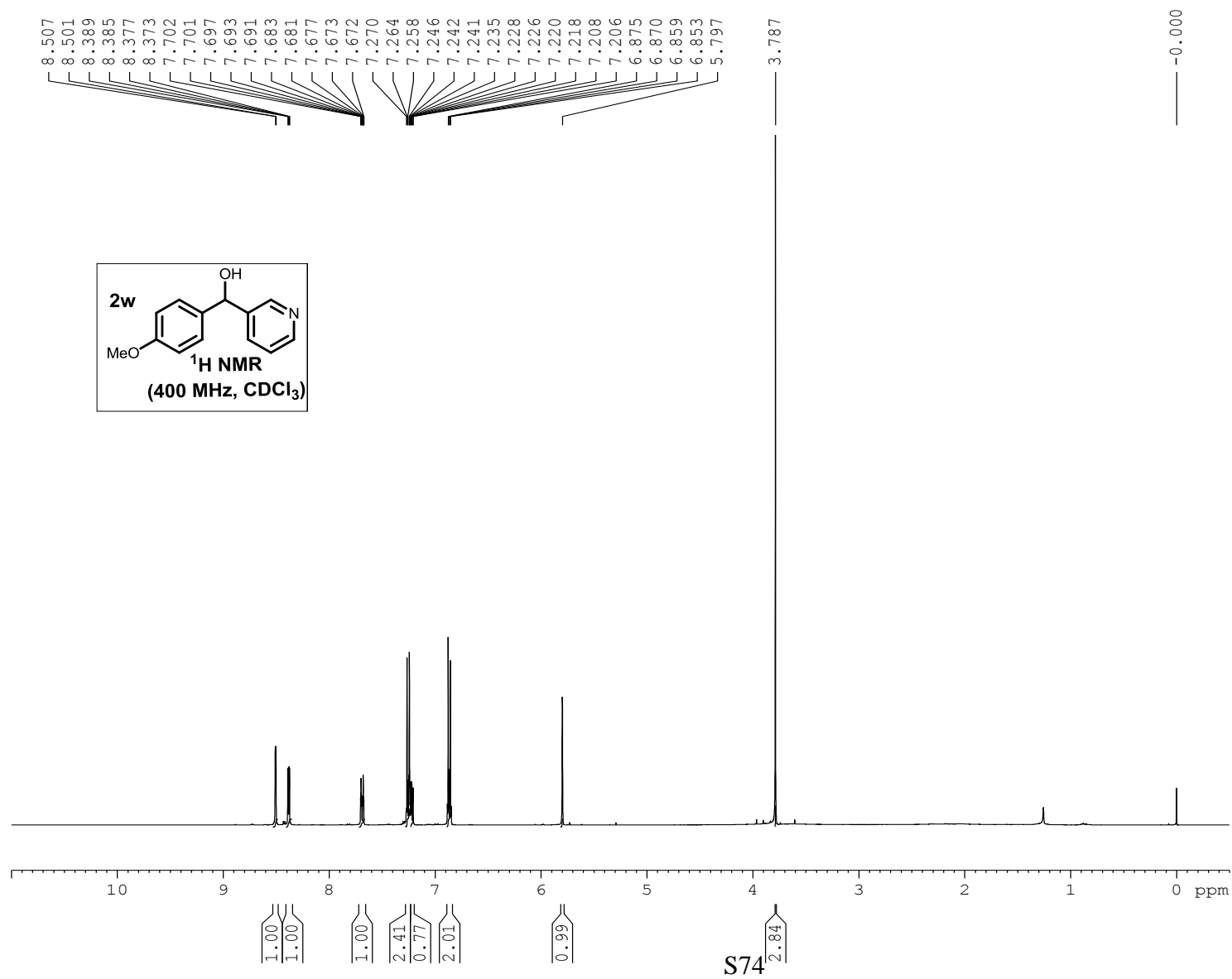
Current Data Parameters  
NAME AHK-I-249C  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120411  
Time 11.51  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 126  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1030  
DW 20.800 usec  
DE 6.00 usec  
TE 294.9 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TDO 1

=====  
CHANNEL f1  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

=====  
CHANNEL f2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127575 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



Current Data Parameters  
NAME AHK-I-117 A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110330  
Time 17.19  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 181  
DW 60.800 usec  
DE 6.00 usec  
TE 295.4 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300023 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



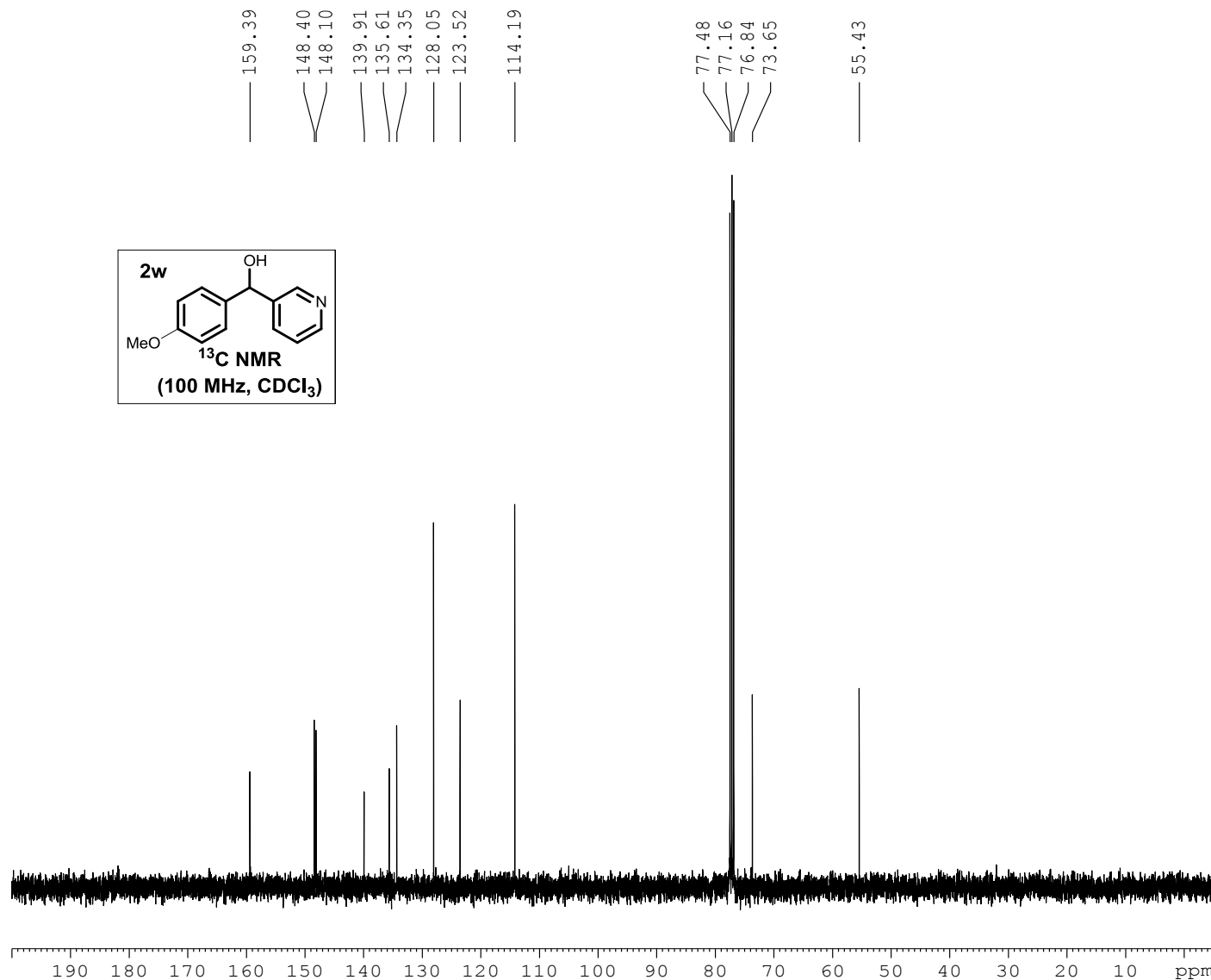
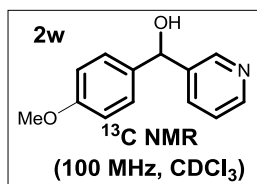
Current Data Parameters  
NAME AHK-I-117 A  
EXPNO 2  
PROCNO 1

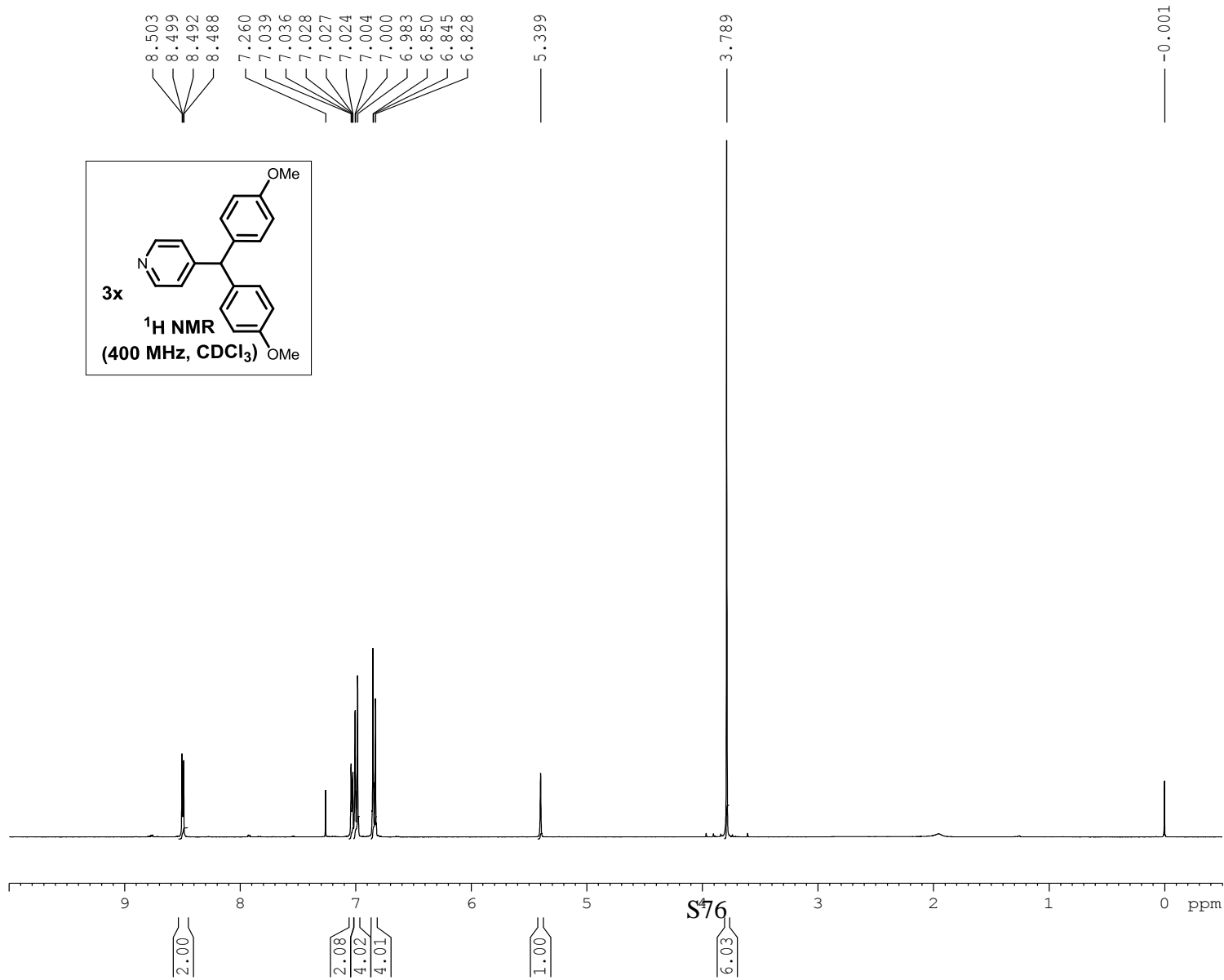
F2 - Acquisition Parameters  
Date\_ 20110330  
Time 17.24  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 64  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 50.8  
DW 20.800 usec  
DE 6.00 usec  
TE 296.0 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127560 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



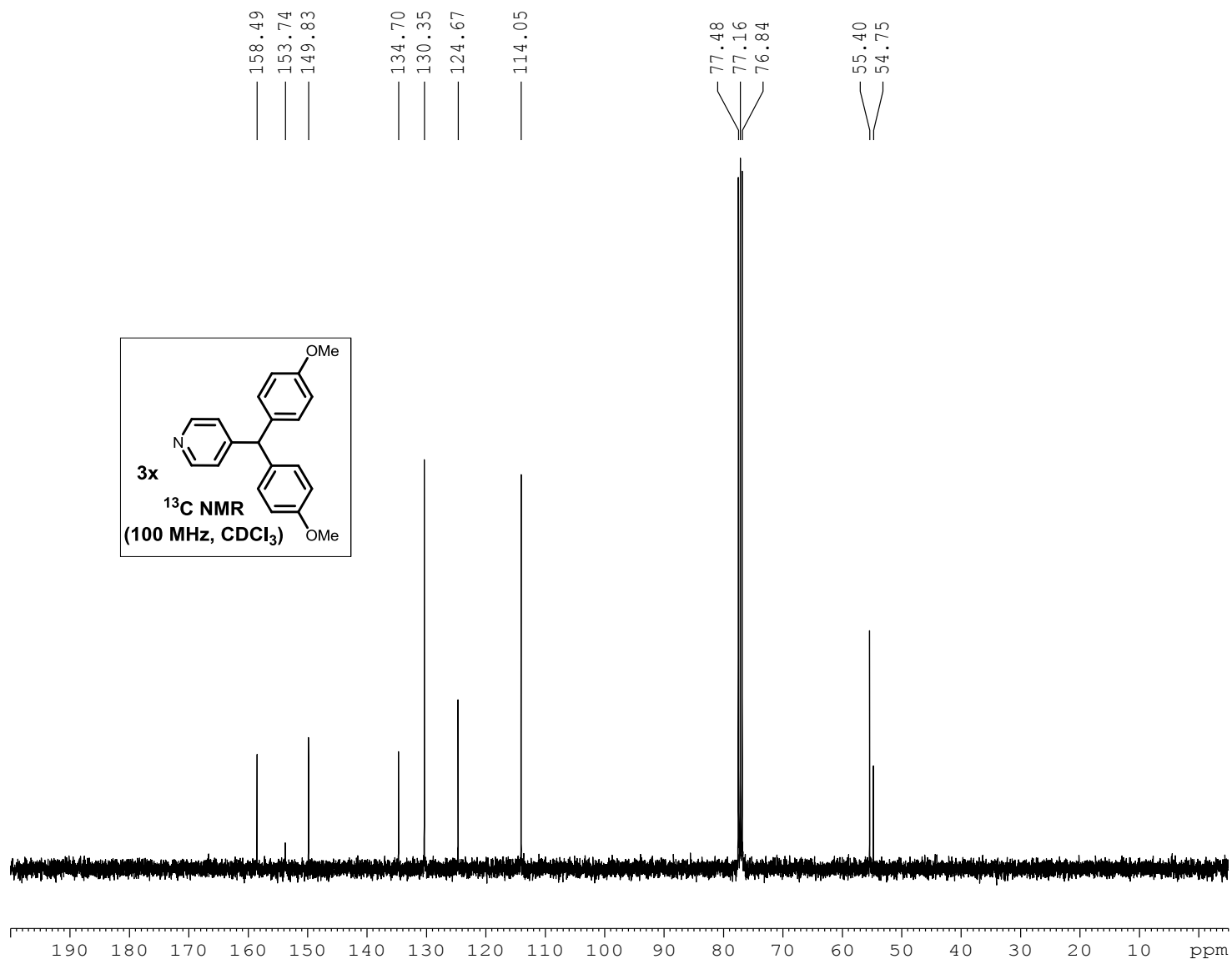


Current Data Parameters  
NAME AHK-I-118A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110324  
Time\_ 11.15  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 256  
DW 60.800 usec  
DE 6.00 usec  
TE 294.5 K  
D1 1.0000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300038 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
FC 1.00



```
Current Data Parameters
NAME      AHK-I-118A
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20110324
Time_     11.18
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        256
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        50.8
DW        20.800 usec
DE        6.00 usec
TE        295.1 K
D1        2.00000000 sec
d11       0.03000000 sec
DELTA    1.89999998 sec
TD0       1

===== CHANNEL f1 =====
NUC1      13C
P1        9.50 usec
PL1       -0.60 dB
SFO1      100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL12      15.60 dB
PL13      15.60 dB
PL2       -0.90 dB
SFO2      400.1316005 MHz

F2 - Processing parameters
SI        32768
SF        100.6127545 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
```



Current Data Parameters  
NAME AHK-I-273A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120427  
Time 12.19  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 80.6  
DW 60.800 usec  
DE 6.00 usec  
TE 298.1 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 11.42 usec  
PL1 -3.00 dB  
SFO1 400.1324710 MHz

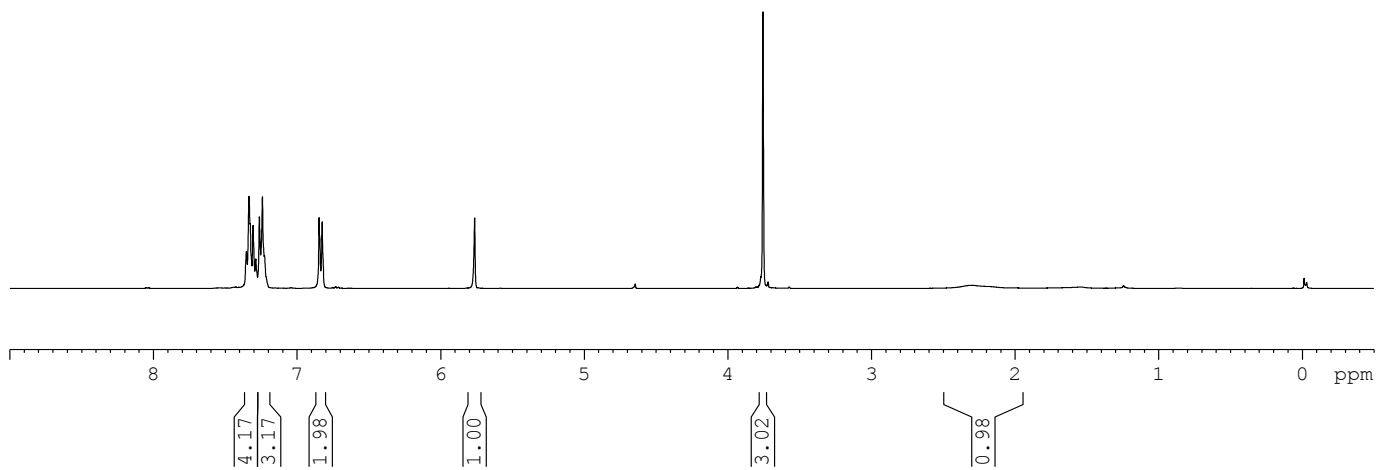
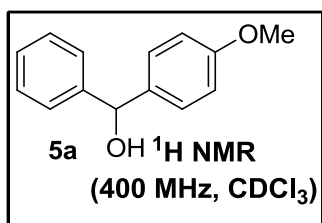
F2 - Processing parameters  
SI 32768  
SF 400.1300202 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

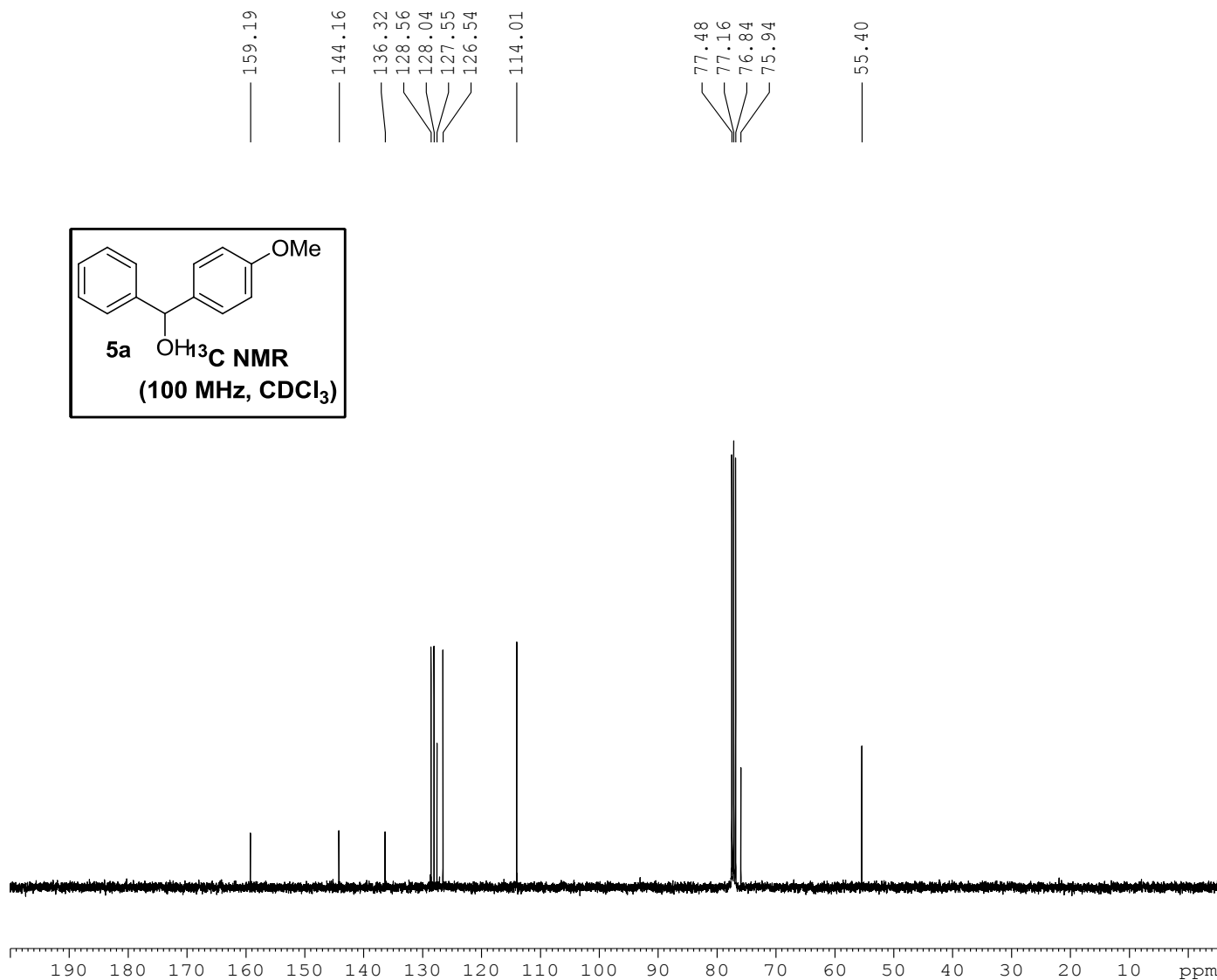
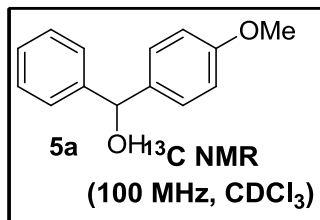
7.3507  
7.3321  
7.3240  
7.3044  
7.2848  
7.2601  
7.2387  
7.2242  
6.8457  
6.8242

5.7629

3.7551

2.3093





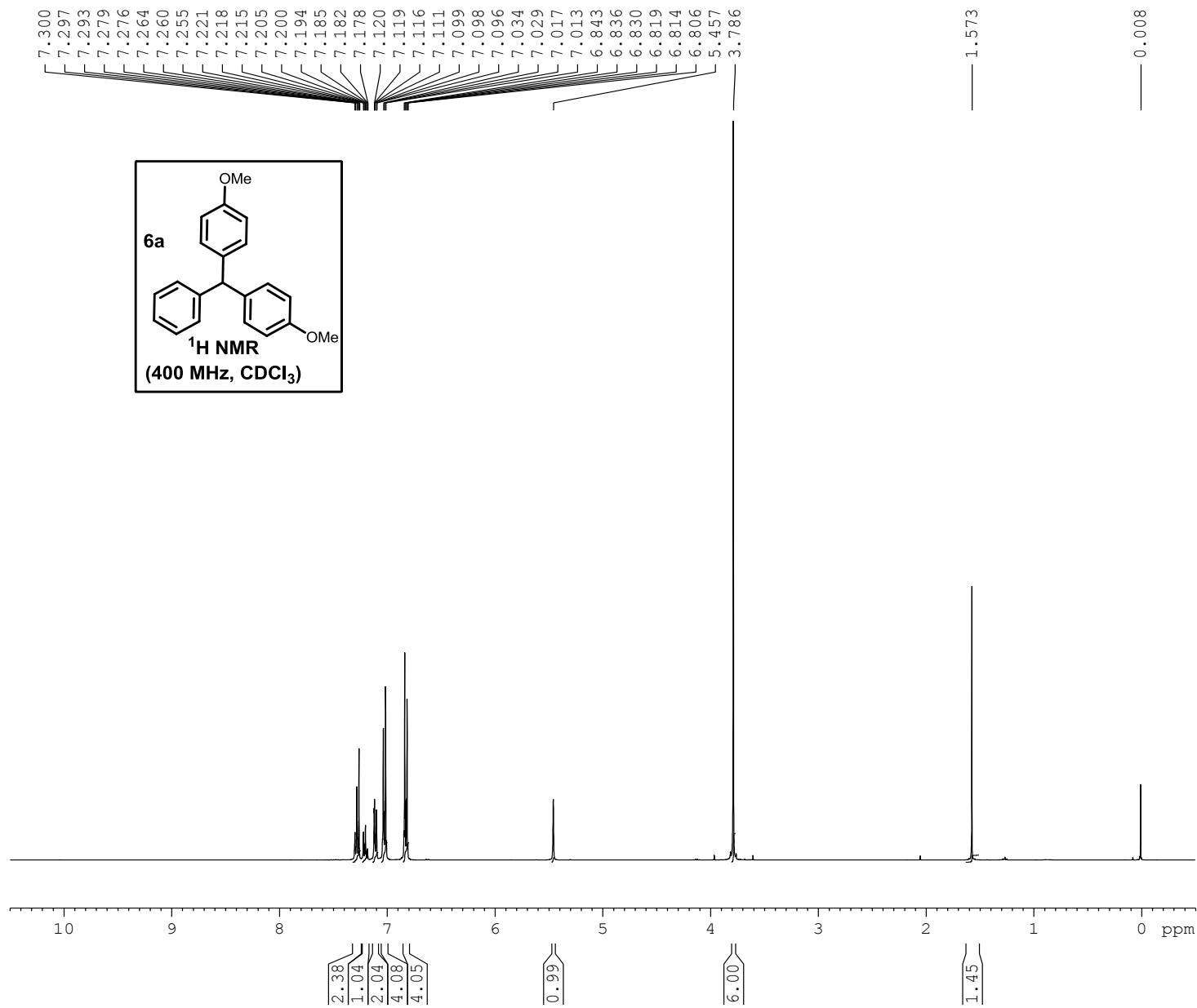
Current Data Parameters  
NAME AHK-I-273  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120427  
Time\_ 16.56  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 183  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 36  
DW 20.800 usec  
DE 6.00 usec  
TE 298.2 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.15 usec  
PL1 0.00 dB  
SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 14.90 dB  
PL13 14.90 dB  
PL2 -3.00 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127561 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



Current Data Parameters  
NAME AHK-I-120 A  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110331  
Time 12.13  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 256  
DW 60.800 usec  
DE 6.00 usec  
TE 294.2 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300040 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

S80





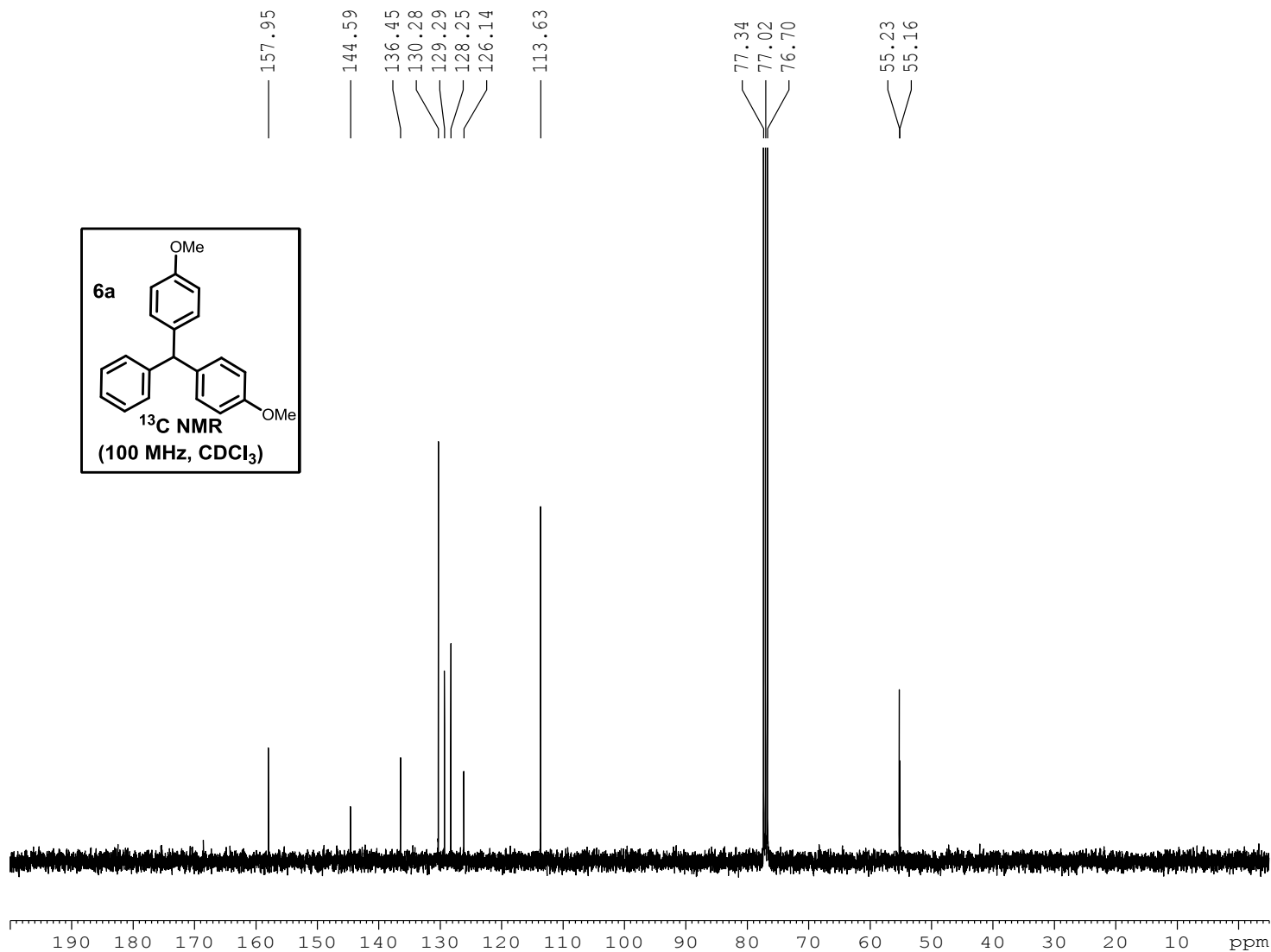
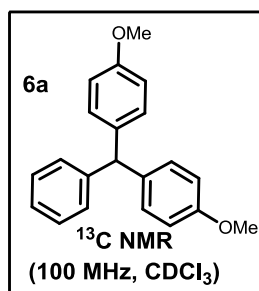
```
Current Data Parameters
NAME      AHK-I-120 A
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20110331
Time      12.22
INSTRUM   spect
PROBHD    5 mm BBO BB-1H
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         166
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG          50.8
DW         20.800 usec
DE         6.00 usec
TE         294.8 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
TD0        1

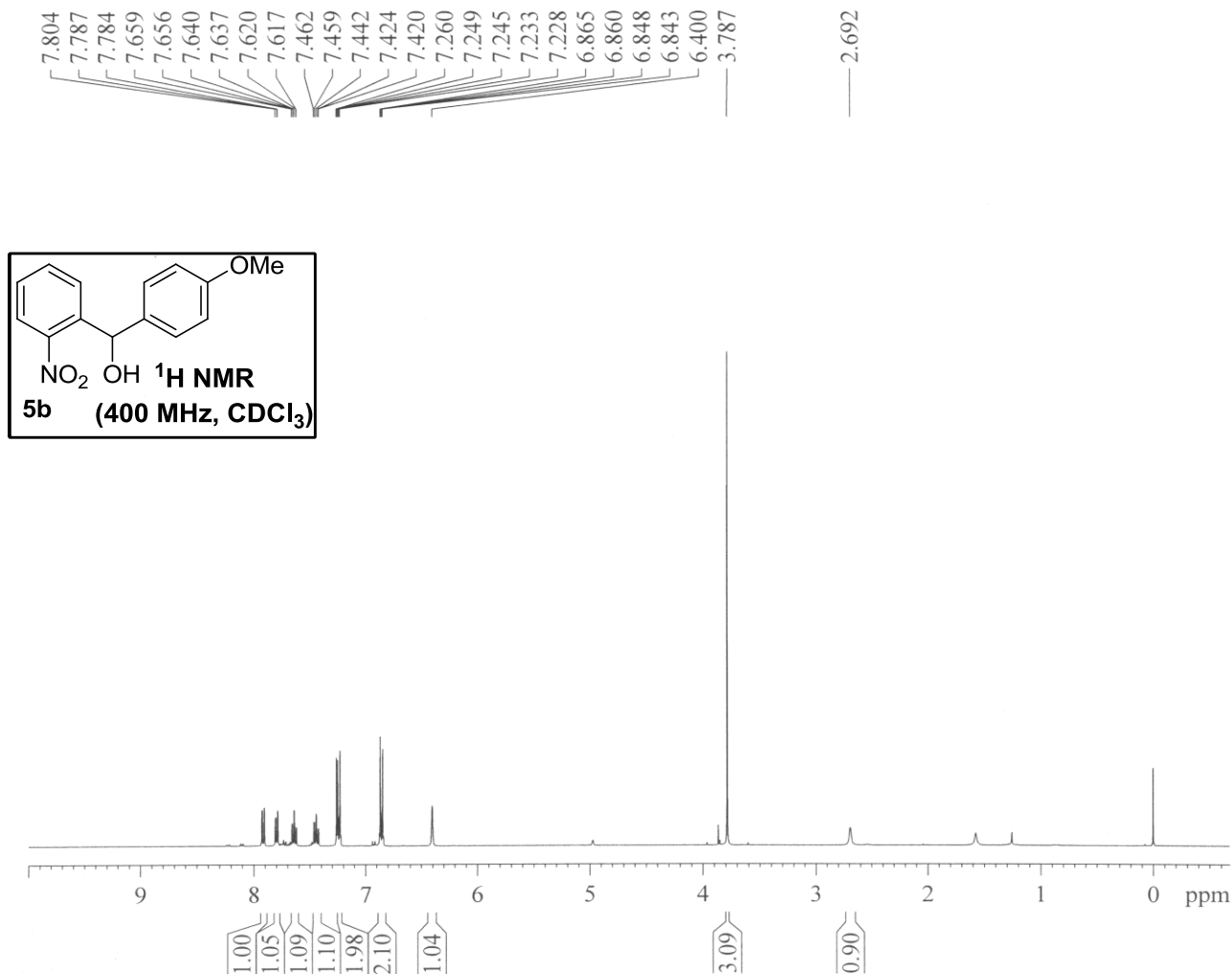
===== CHANNEL f1 =====
NUC1       13C
P1         9.50 usec
PL1        -0.60 dB
SFO1       100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      90.00 usec
PL12       15.60 dB
PL13       15.60 dB
PL2        -0.90 dB
SFO2       400.1316005 MHz

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



PROTON CDC13 {D:\GV} KOPAL 1



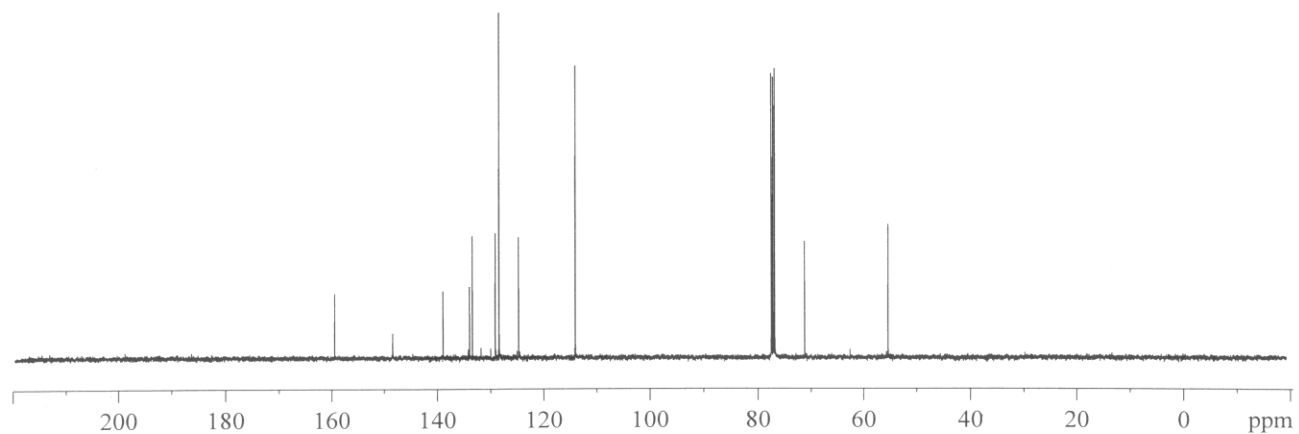
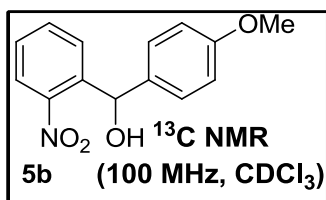
Current Data Parameters  
NAME AHK-I-253E  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120724  
Time\_ 11.24  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 144  
DW 60.800 usec  
DE 6.00 usec  
TE 297.1 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.42 usec  
PL1 -3.00 dB  
SF01 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300029 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00

C13CPD CDC13 {D:\CRR} KOPAL 1



Current Data Parameters  
NAME AHK-I-253D  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120723  
Time 10.32  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 137  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 32  
DW 20.800 usec  
DE 6.00 usec  
TE 298.0 K  
D1 2.0000000 sec  
d11 0.0300000 sec  
DELTA 1.89999998 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.15 usec  
PL1 0.00 dB  
SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 14.90 dB  
PL13 14.90 dB  
PL2 -3.00 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127565 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

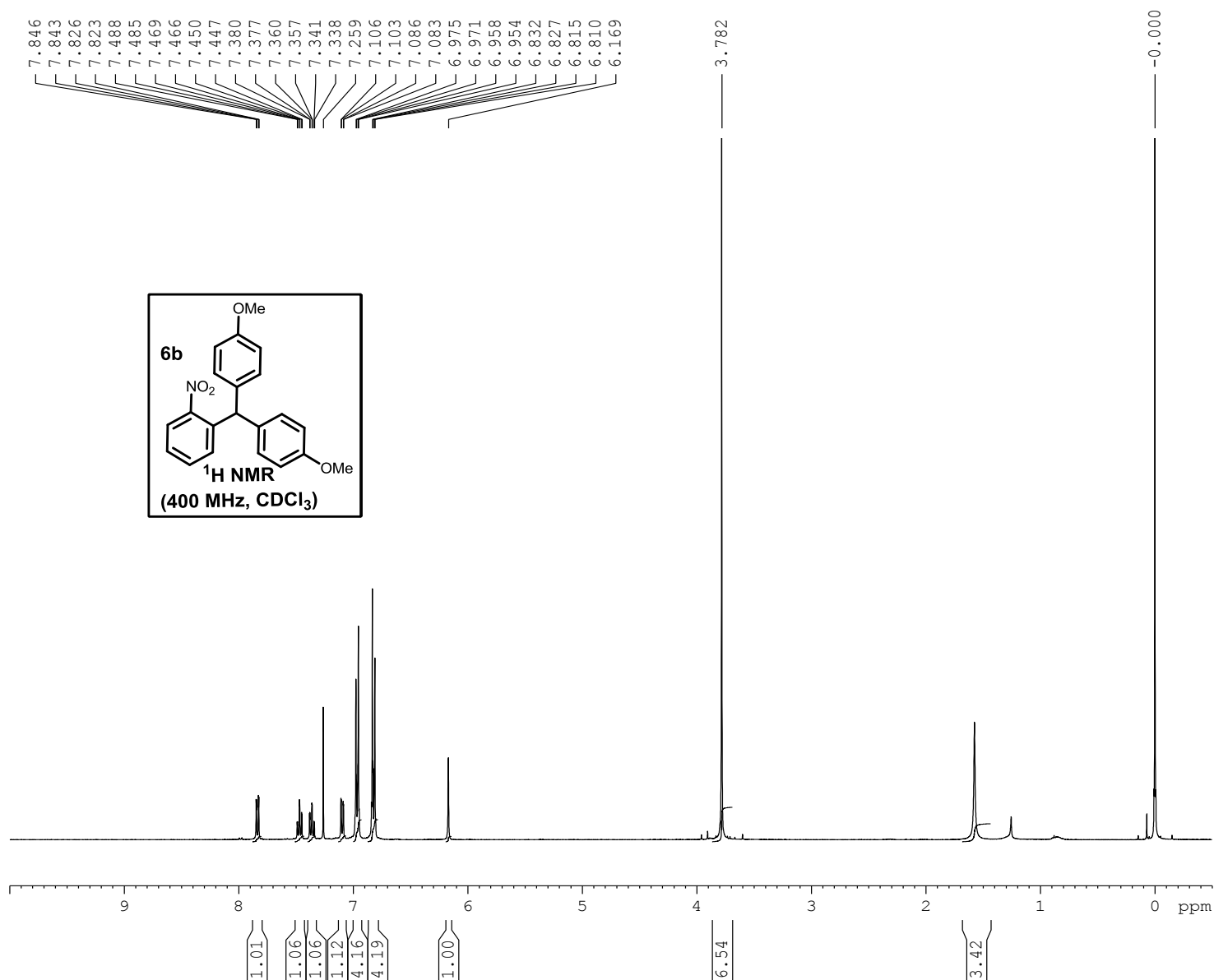


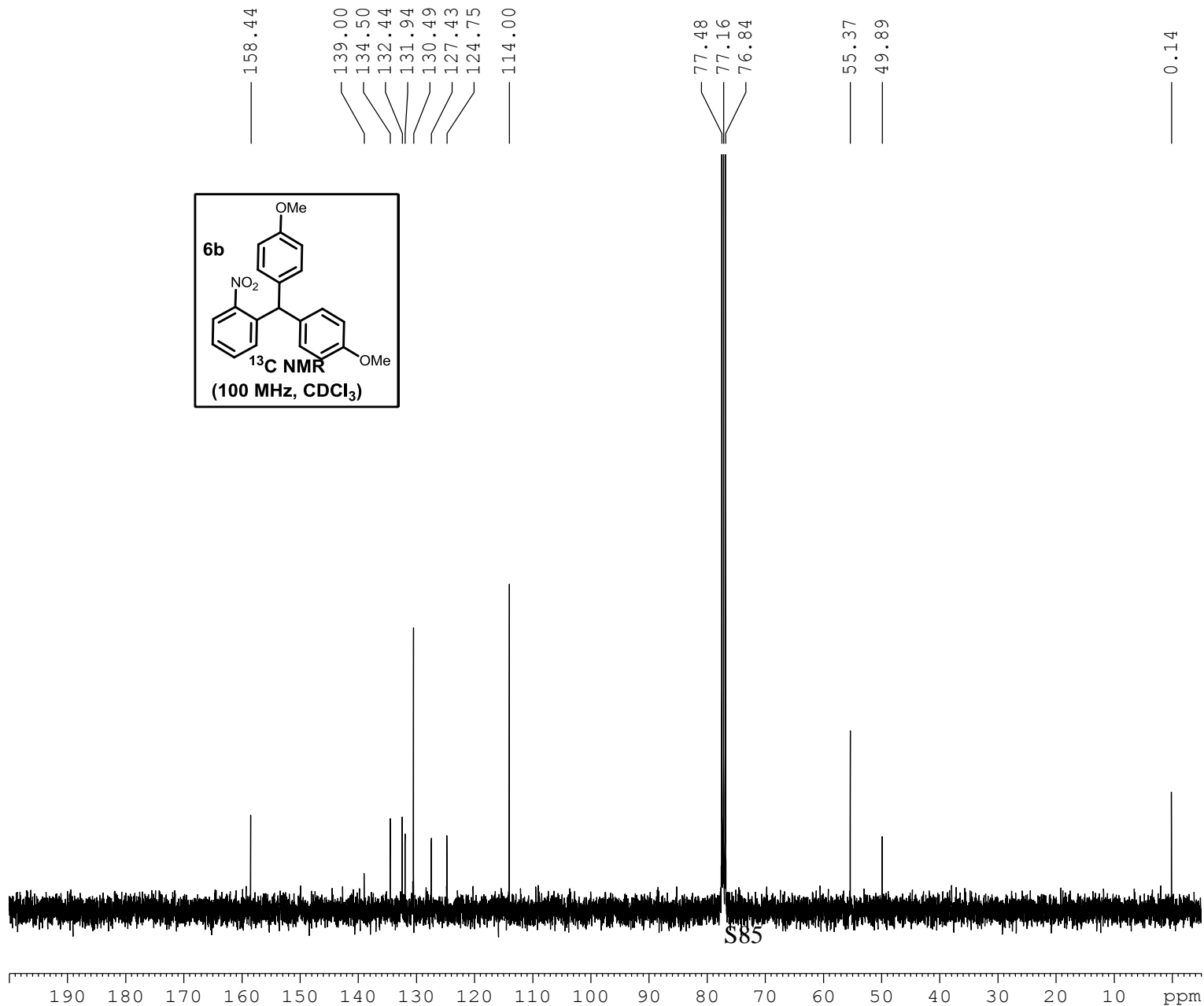
Current Data Parameters  
NAME AHK-I-126 B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110516  
Time 11.44  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 287  
DW 60.800 usec  
DE 6.00 usec  
TE 295.3 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300040 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
EC 1.00





Current Data Parameters  
NAME AHK-I-126 B  
EXPNO 2  
PROCNO 1

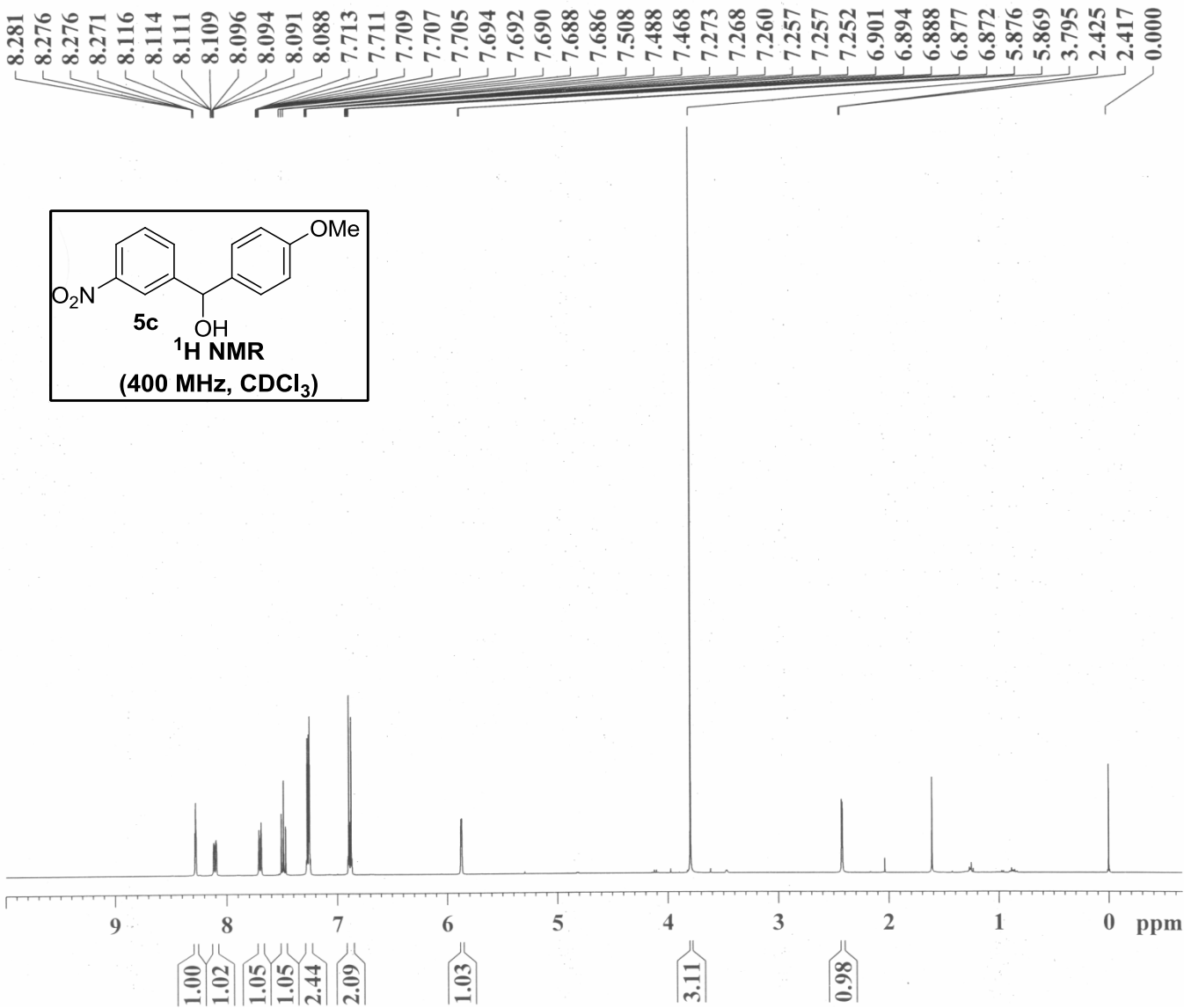
F2 - Acquisition Parameters  
Date\_ 20110516  
Time 12.00  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 50.8  
DW 20.800 usec  
DE 6.00 usec  
TE 295.8 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127534 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

PROTON CDCl3 {D:\CRR} crr 1

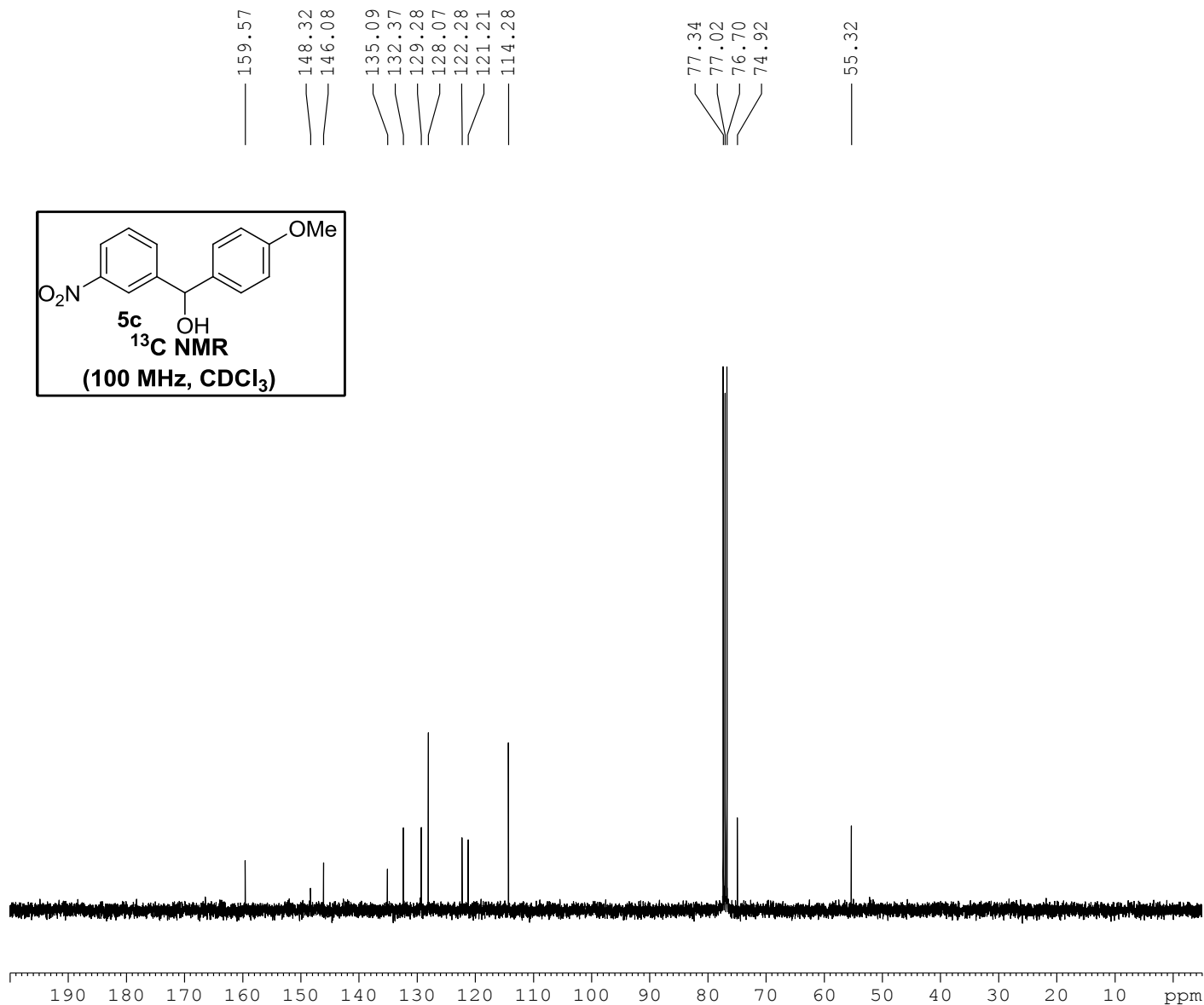
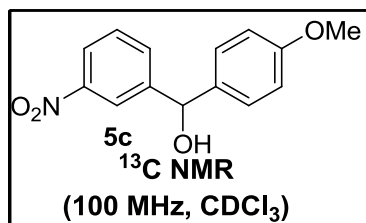


Current Data Parameters  
NAME AHK-I-267B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120413  
Time\_ 22.59  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 256  
DW 60.800 usec  
DE 6.00 usec  
TE 295.1 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300041 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



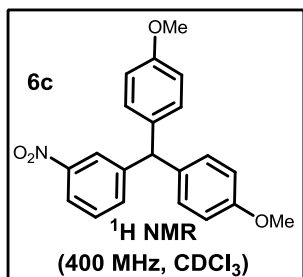
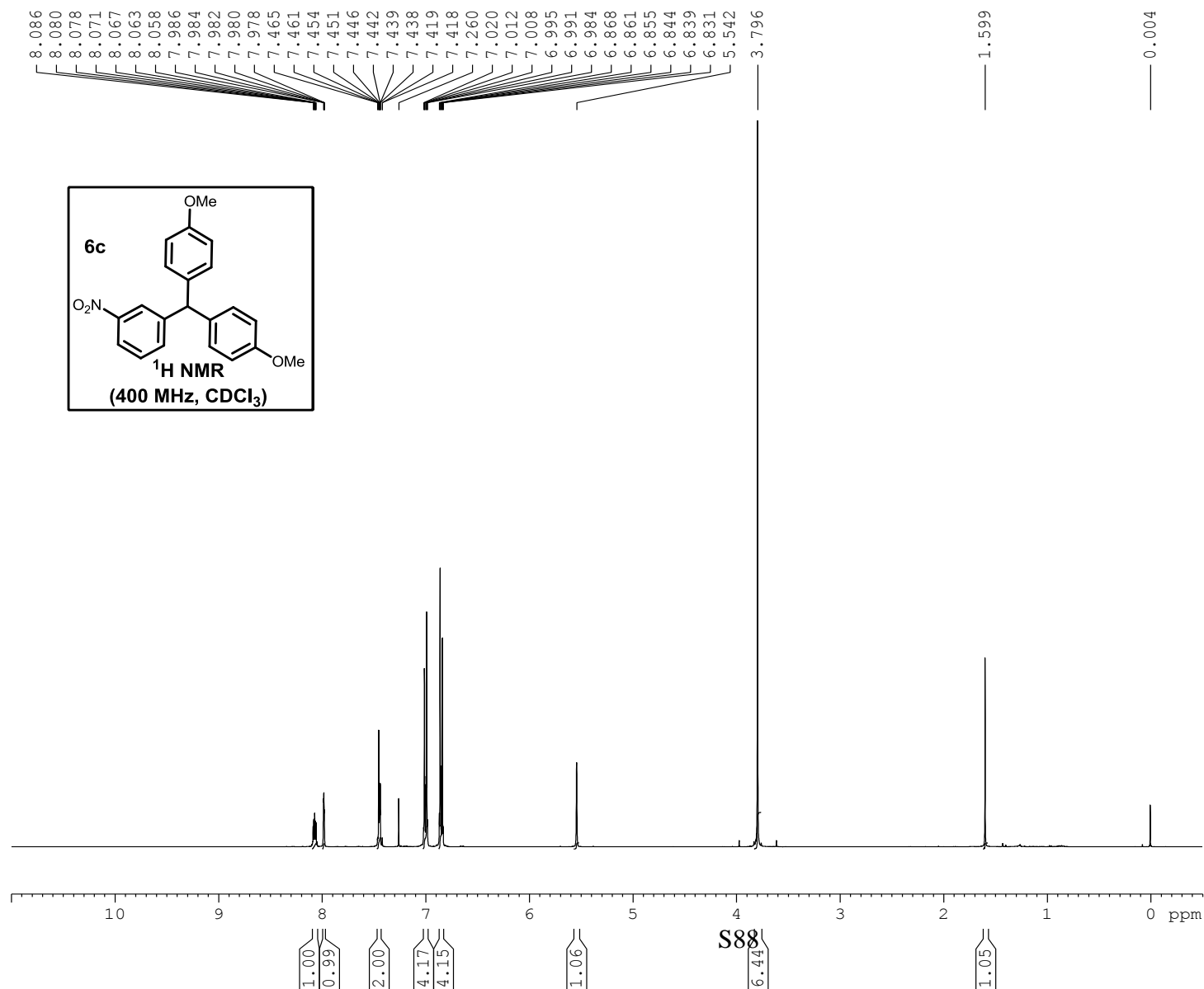
Current Data Parameters  
NAME AHK-I-267D  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120417  
Time\_ 11.45  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 118  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 2050  
DW 20.800 usec  
DE 6.00 usec  
TE 295.4 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

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



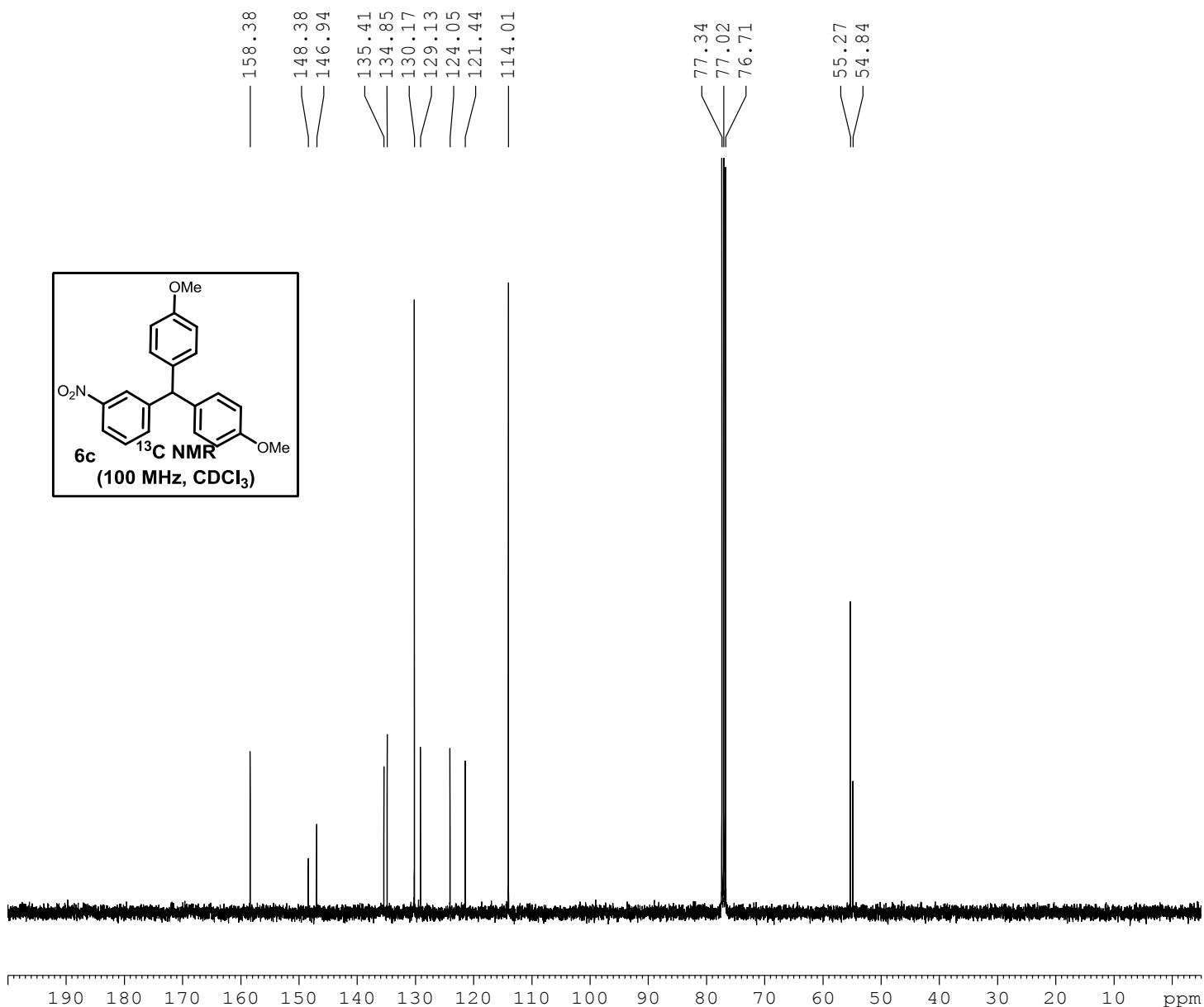
Current Data Parameters  
NAME AHK-I-116-E  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110406  
Time\_ 11.52  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 181  
DW 60.800 usec  
DE 6.00 usec  
TE 294.4 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300038 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





Current Data Parameters  
NAME AHK-I-116-E2  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110407  
Time\_ 10.38  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 258  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 50.8  
DW 20.800 usec  
DE 6.00 usec  
TE 295.4 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

=====  
CHANNEL f1  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

=====  
CHANNEL f2  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

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

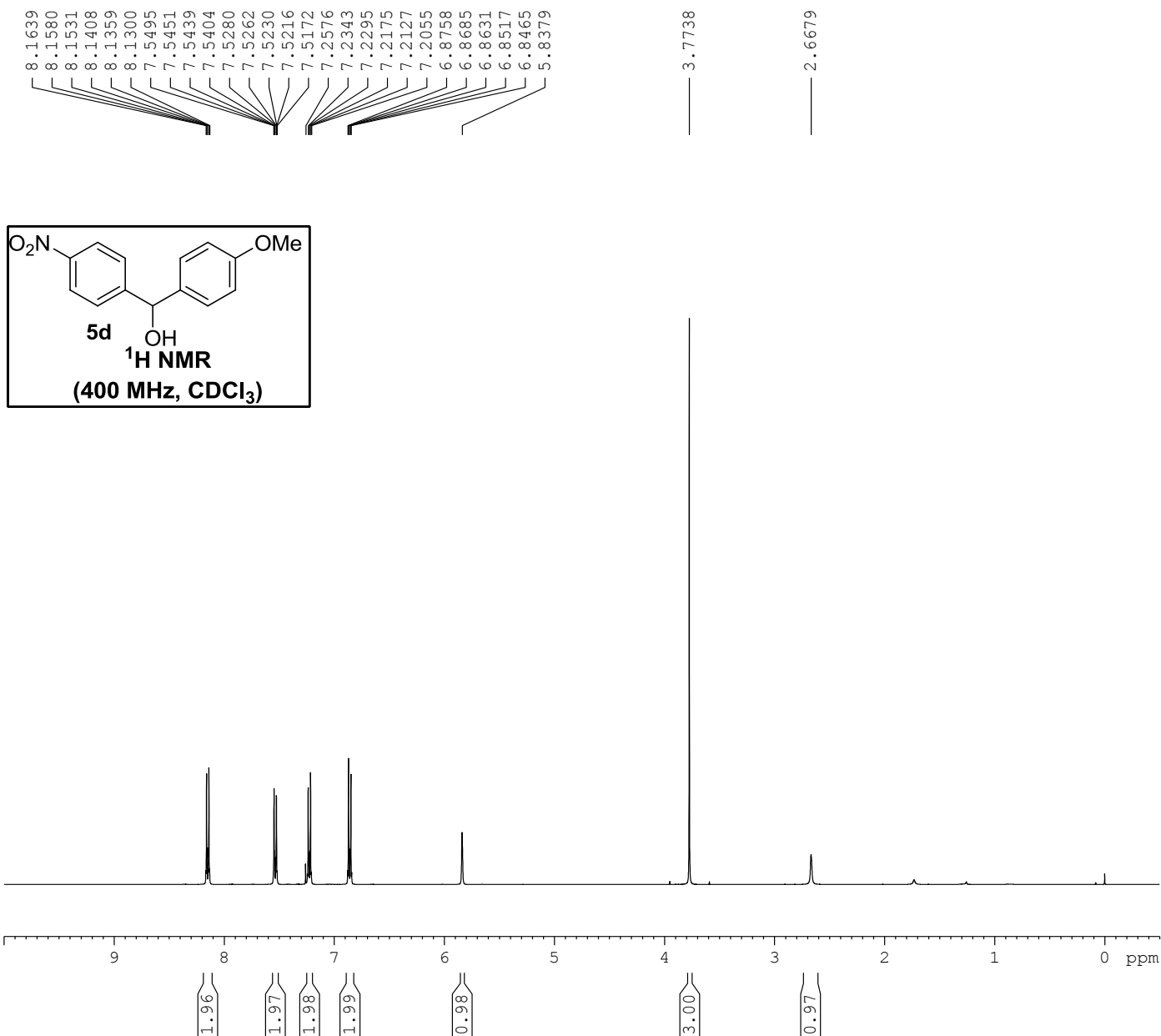


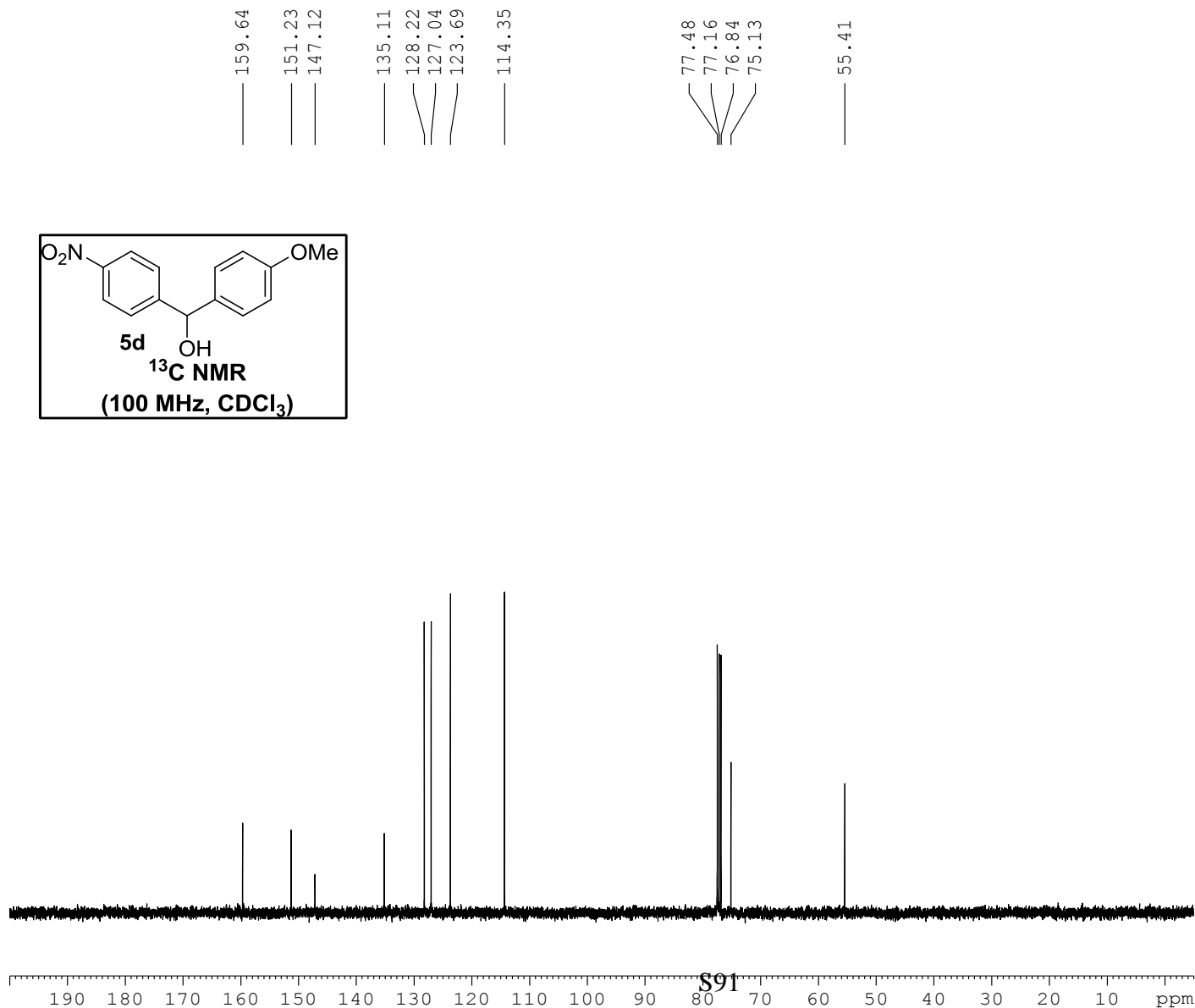
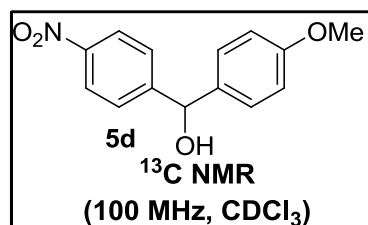
Current Data Parameters  
NAME AHK-I-252B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120411  
Time 11.58  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 128  
DW 60.800 usec  
DE 6.00 usec  
TE 294.7 K  
D1 1.00000000 sec  
TD0 1

=====  
CHANNEL f1  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300046 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





Current Data Parameters  
NAME AHK-I-252B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120411  
Time 12.00  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 38  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1030  
DW 20.800 usec  
DE 6.00 usec  
TE 295.1 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127597 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

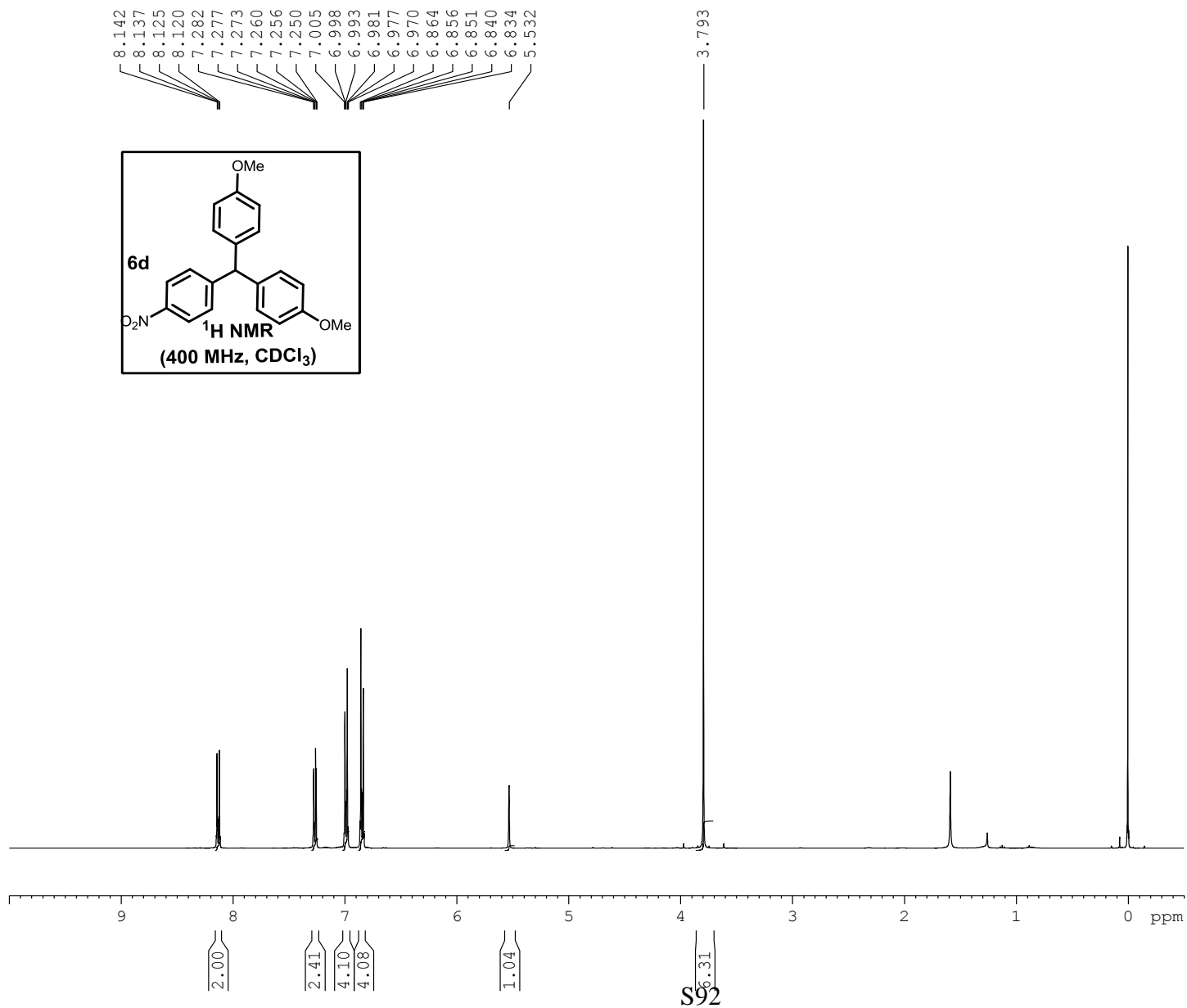


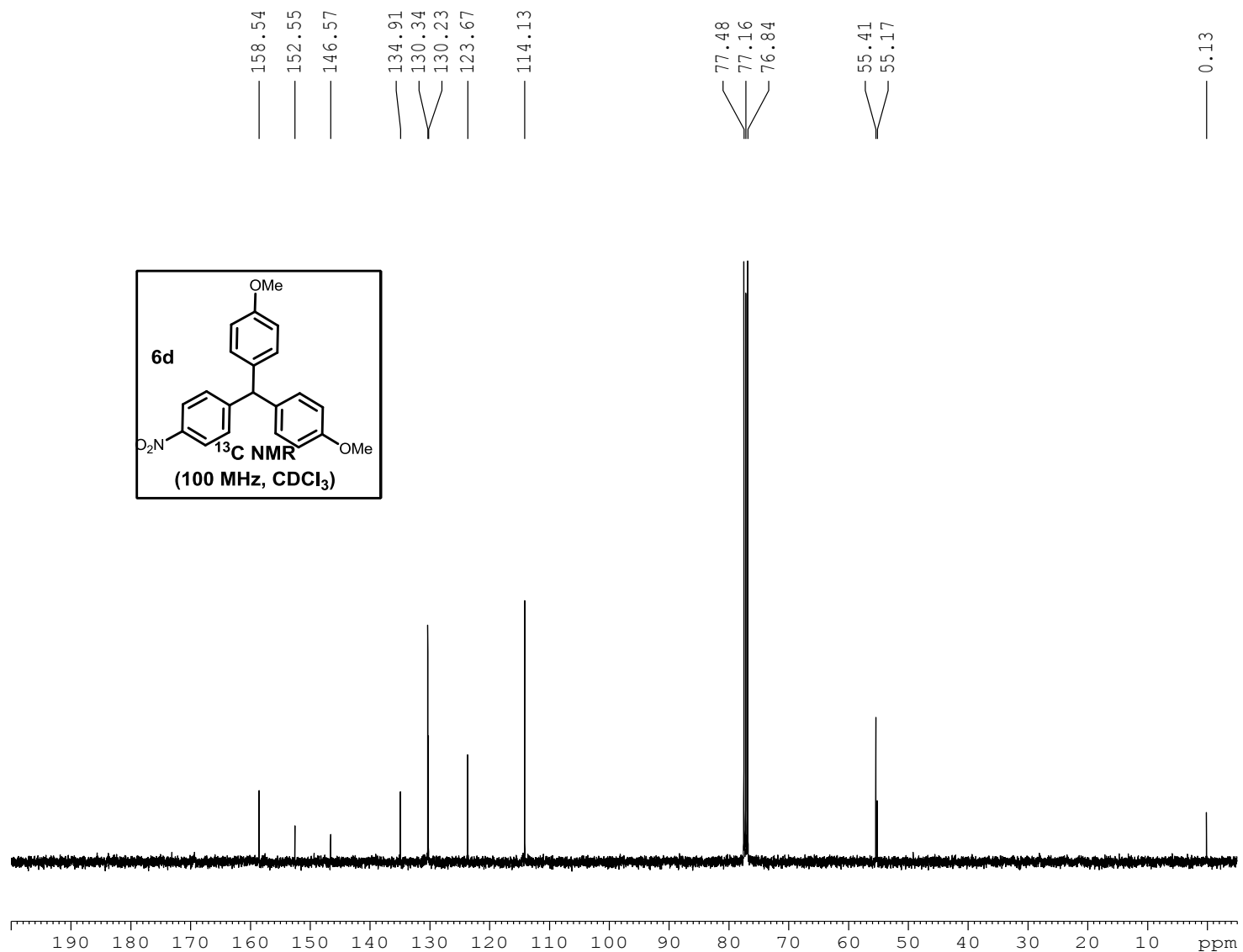
Current Data Parameters  
NAME AHK-I-127 B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110516  
Time\_ 12.59  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 256  
DW 60.800 usec  
DE 6.00 usec  
TE 295.1 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300038 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





Current Data Parameters  
NAME AHK-I-127 B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20110516  
Time\_ 13.15  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 1030  
DW 20.800 usec  
DE 6.00 usec  
TE 295.8 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

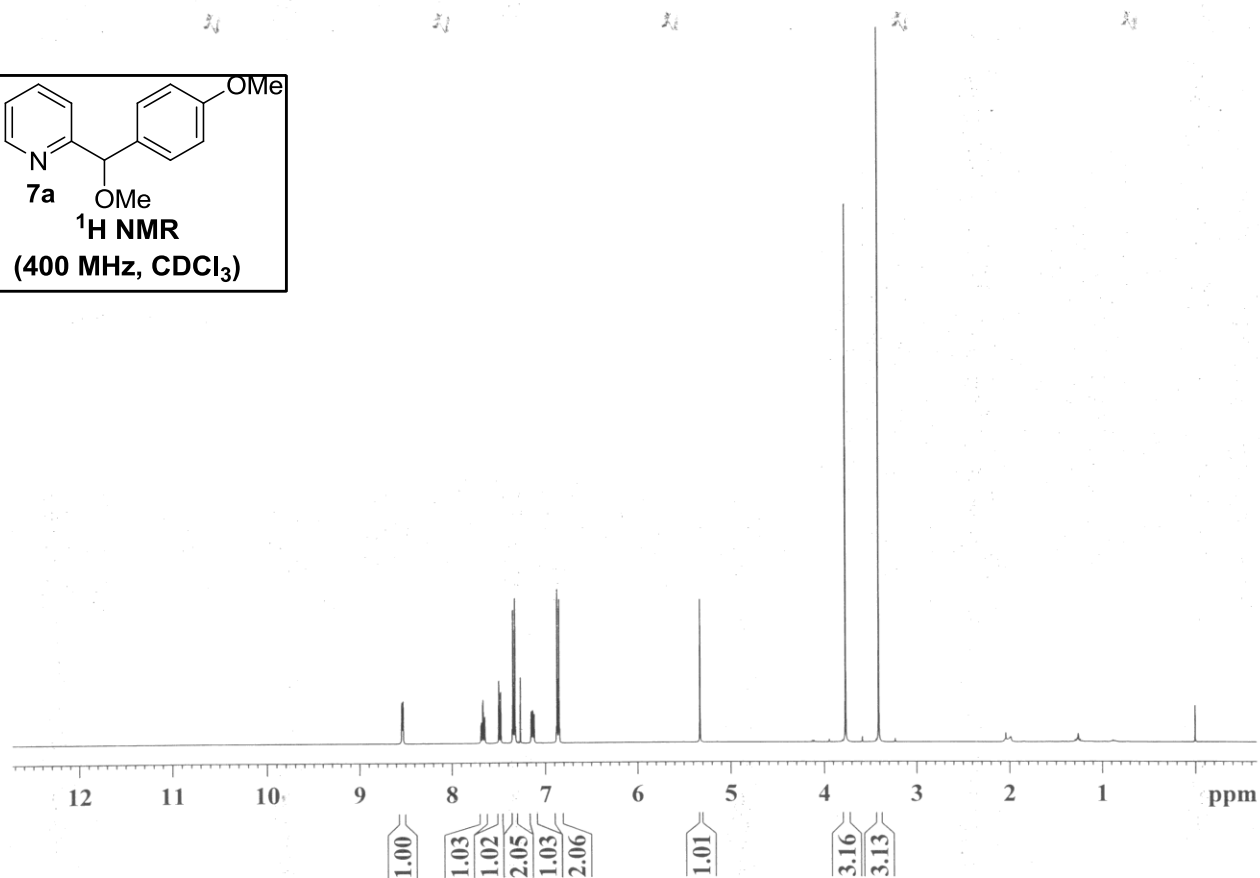
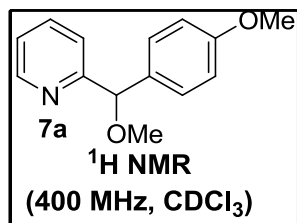
===== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127543 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

PROTON CDCl3 {D:\CRR} crr 1

8.533  
8.527  
8.525  
8.523  
8.521  
7.687  
7.683  
7.667  
7.663  
7.648  
7.644  
7.495  
7.475  
7.352  
7.344  
7.339  
7.328  
7.323  
7.316  
7.263  
7.150  
7.147  
7.138  
7.135  
7.132  
7.129  
7.119  
7.116  
6.877  
6.870  
6.864  
6.853  
6.848  
6.841  
5.325  
3.765  
3.407

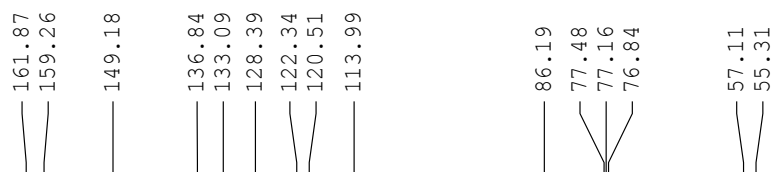
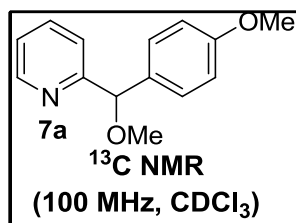


Current Data Parameters  
NAME AHK-I-263B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120413  
Time\_ 22.54  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 144  
DW 60.800 usec  
DE 6.00 usec  
TE 295.0 K  
D1 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300026 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



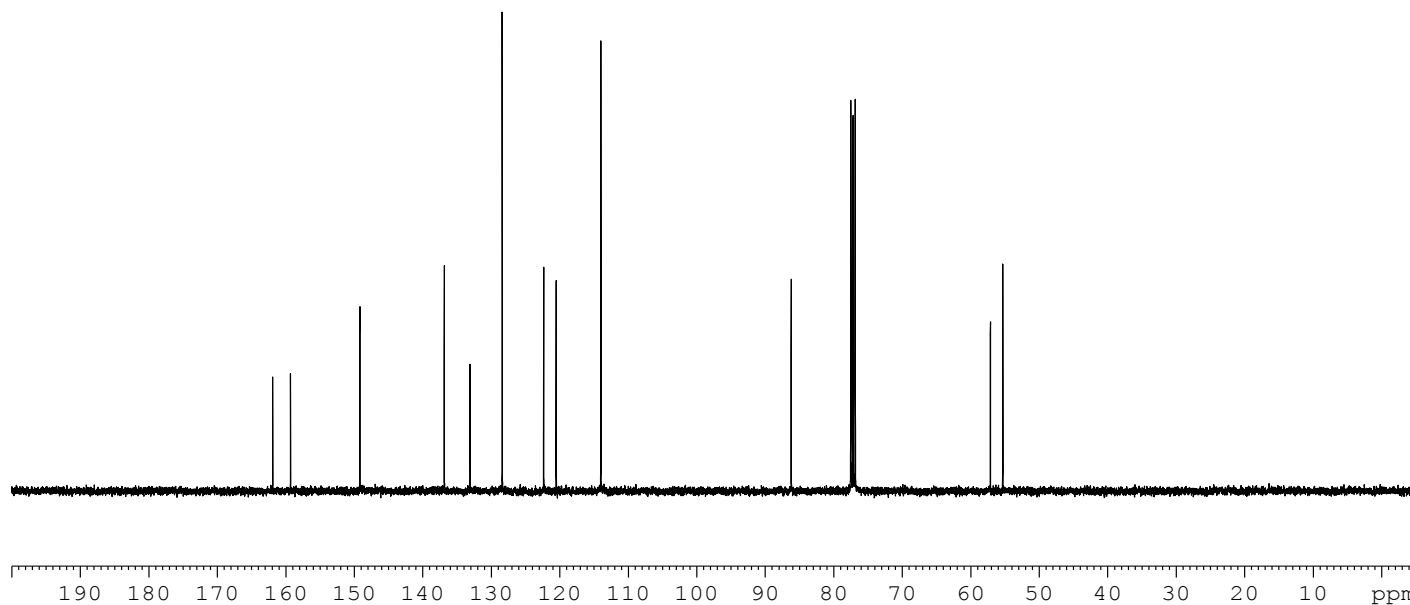
Current Data Parameters  
NAME AHK-263B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date 20120416  
Time 16.04  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 50.8  
DE 20.800 usec  
TE 6.00 usec  
D1 297.7 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

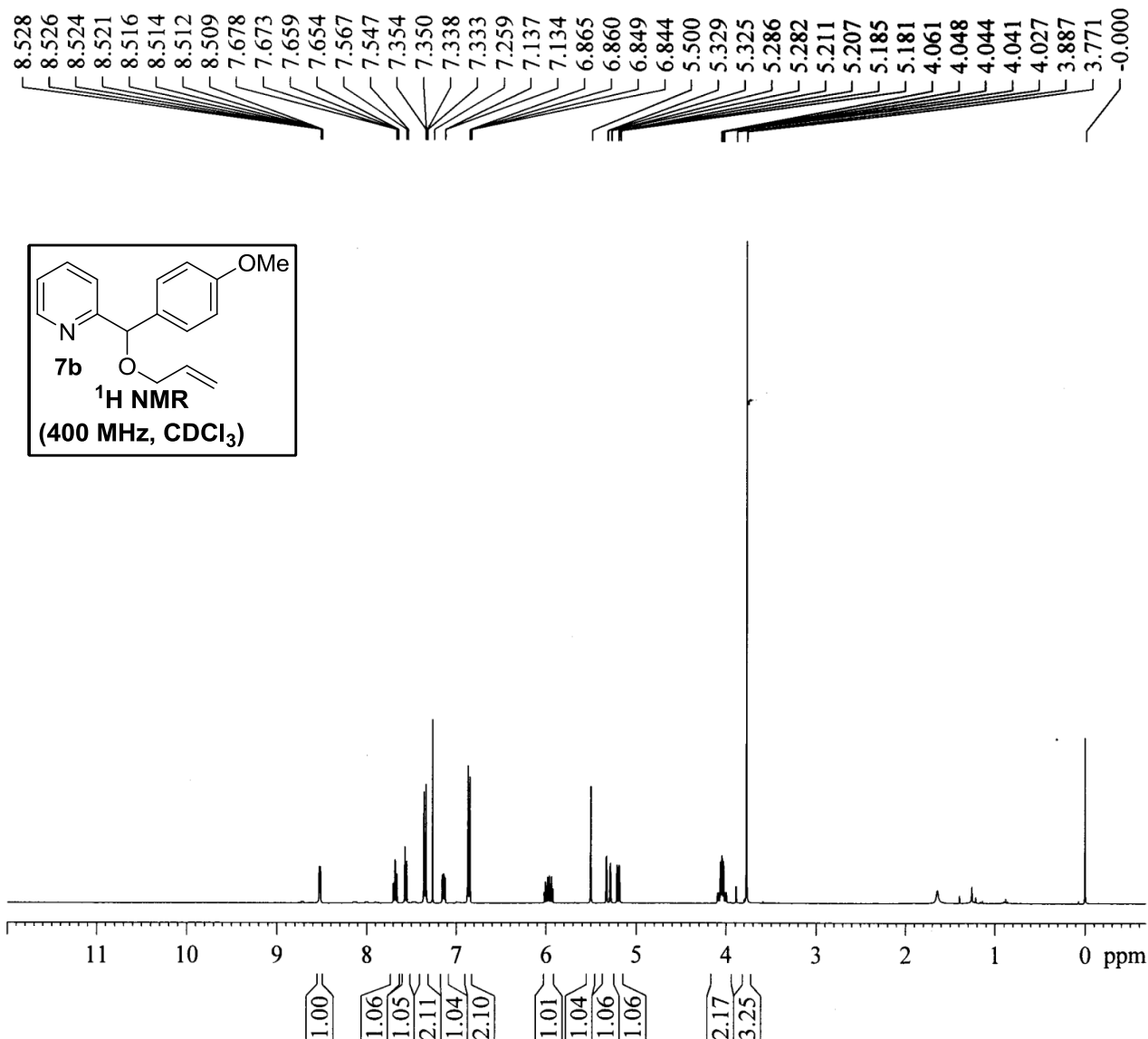
==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127585 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



PROTON CDC13 {D:\CRR} KOPAL 1



Current Data Parameters  
NAME AHK-I-265E  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120724  
Time 16.01  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 144  
DW 60.800 usec  
DE 6.00 usec  
TE 297.6 K  
D1 1.00000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 11.42 usec  
PL1 -3.00 dB  
SFO1 400.1324710 MHz

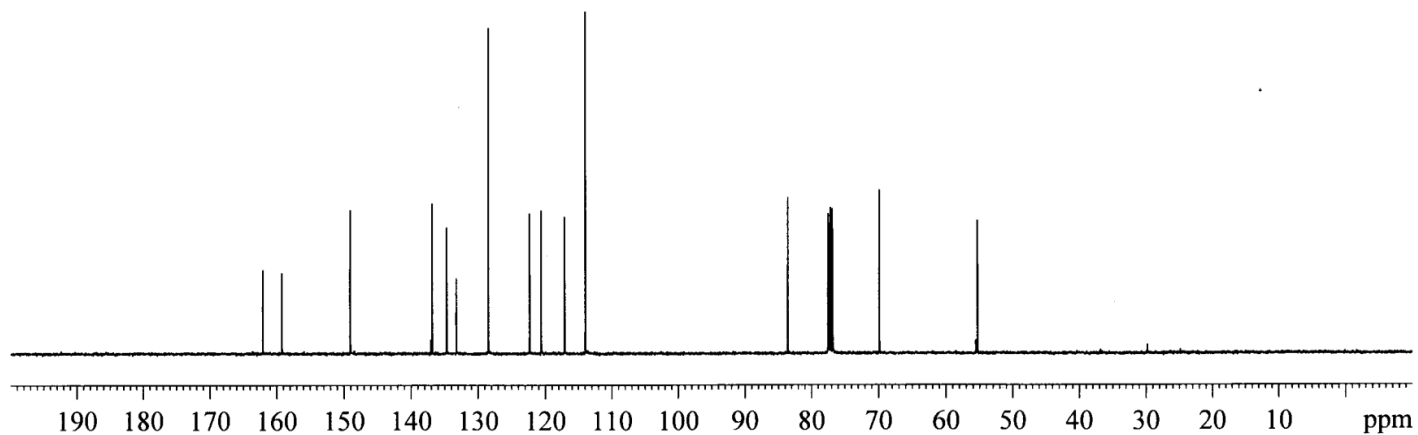
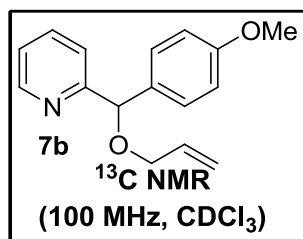
F2 - Processing parameters  
SI 32768  
SF 400.1300034 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



C13CPD CDC13 {D:\CRR} KOPAL 1

162.07  
159.20  
149.05  
136.84  
134.67  
133.23  
128.40  
122.29  
120.54  
117.07  
113.94

83.53  
77.48  
77.16  
76.84  
69.84  
55.26



Current Data Parameters  
NAME AHK-I-265D  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120724  
Time 11.50  
INSTRUM spect  
PROBHD 5 mm DUL 13C-1  
PULPROG zgpg30  
TD 65536  
SOLVENT CDC13  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 28.5  
DW 20.800 usec  
DE 6.00 usec  
TE 297.7 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.15 usec  
PL1 0.00 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 14.90 dB  
PL13 14.90 dB  
PL2 -3.00 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127616 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

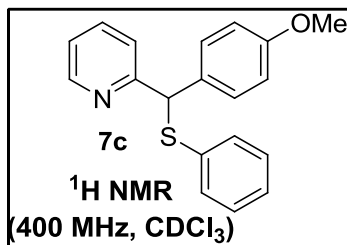
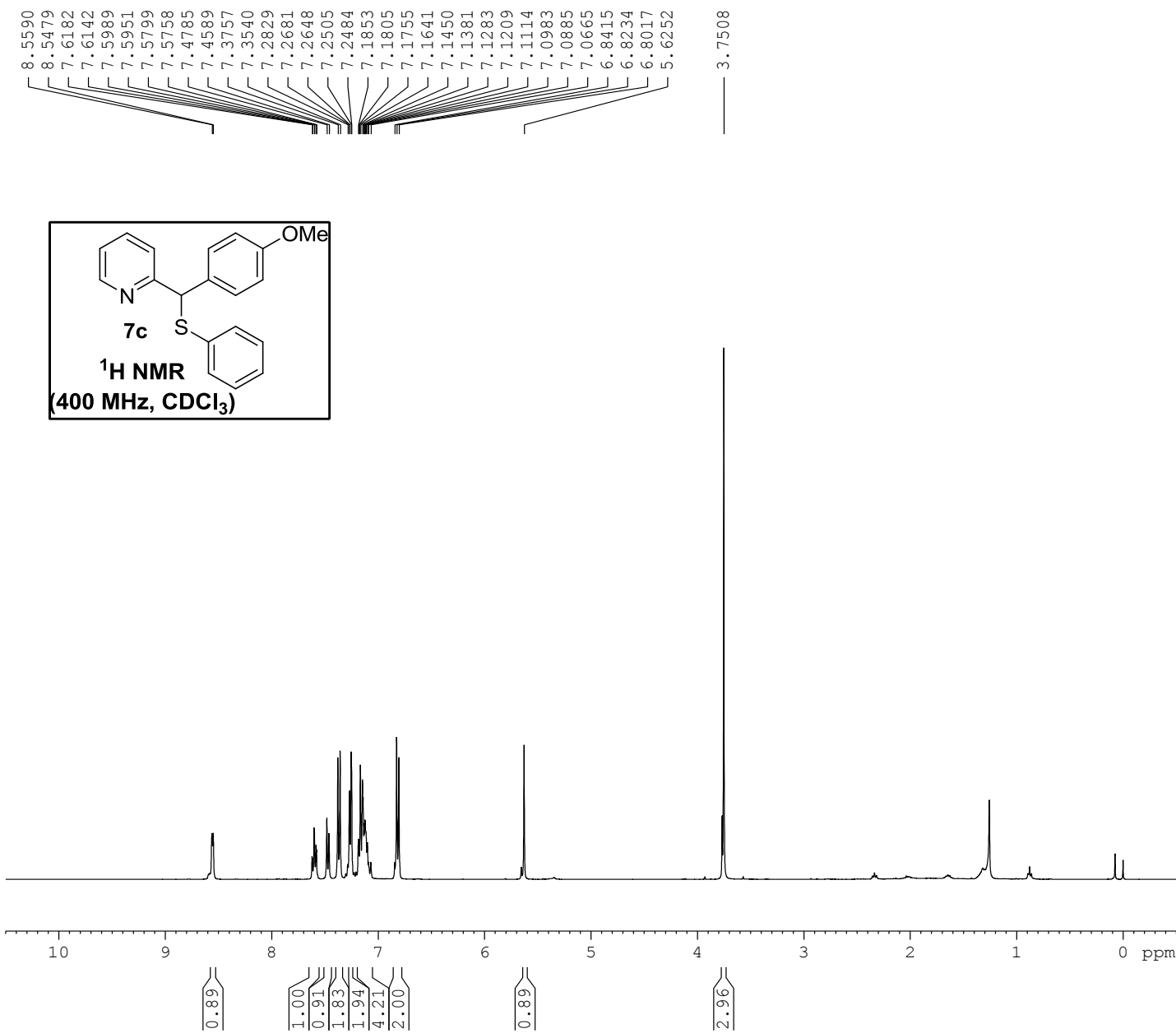


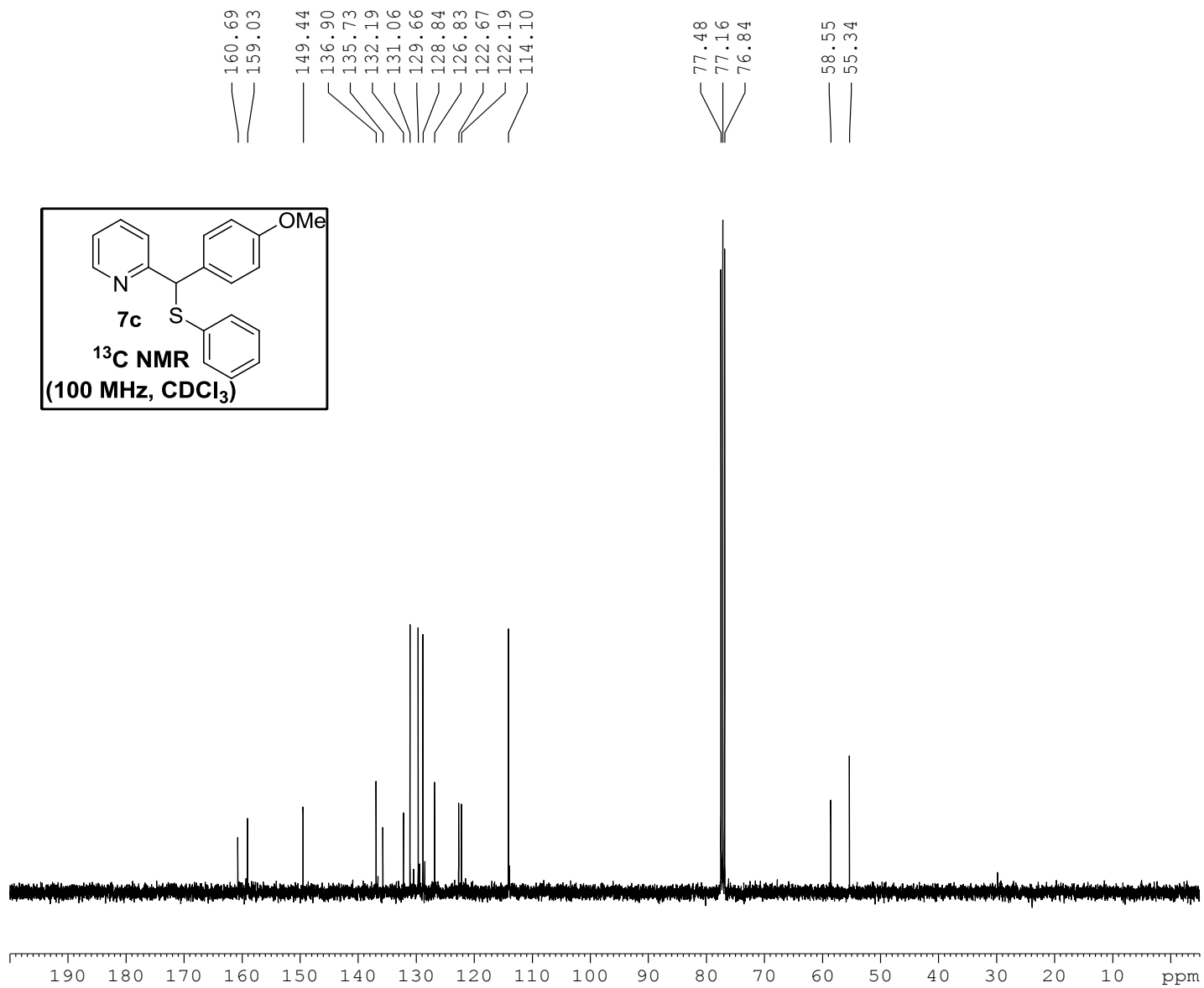
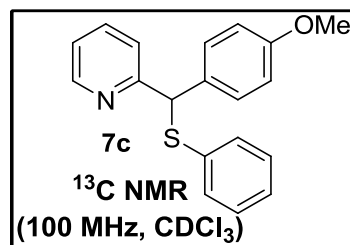
Current Data Parameters  
NAME AHK-I-248B  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20120302  
Time\_ 12.46  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 16  
DS 2  
SWH 8223.685 Hz  
FIDRES 0.125483 Hz  
AQ 3.9846387 sec  
RG 181  
DW 60.800 usec  
DE 6.00 usec  
TE 297.5 K  
D1 1.00000000 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -0.90 dB  
SFO1 400.1324710 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1300068 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00





Current Data Parameters  
NAME AHK-I-248B  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date 20120302  
Time 12.51  
INSTRUM spect  
PROBHD 5 mm BBO BB-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 181  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631988 sec  
RG 50.8  
DW 20.800 usec  
DE 6.00 usec  
TE 298.0 K  
D1 2.00000000 sec  
d11 0.03000000 sec  
DELTA 1.89999998 sec  
TD0 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 -0.60 dB  
SFO1 100.6228298 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PL12 15.60 dB  
PL13 15.60 dB  
PL2 -0.90 dB  
SFO2 400.1316005 MHz

F2 - Processing parameters  
SI 32768  
SF 100.6127569 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40