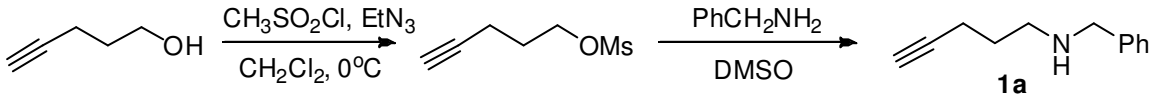
Supporting Information

Synthesis of Cyclic α-CN and α-CF_3 N-Heterocycles through Tandem Nucleophilic Additions

Junbin Han, Bo Xu and Gerald B. Hammond**

General considerations: ^{1}H, ^{13}C and ^{19}F NMR spectra were recorded at 400, 100 and 370 MHz respectively, using $CDCl_3$ as a solvent. The chemical shifts are reported in δ (ppm) values relative to $CHCl_3$ (δ 7.26 ppm for ^{1}H NMR and δ 77.0 ppm for ^{13}C NMR) and $CFCl_3$ (δ 0 ppm for ^{19}F NMR), multiplicities are indicated by s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (hextet), m (multiplet) and br (broad). Coupling constants, *J*, are reported in Hertz. Coupling constants are reported in hertz (Hz). All air and/or moisture sensitive reactions were carried out under argon atmosphere. Solvents (tetrahydrofuran, ether, dichloromethane and DMF) were chemically dried using a commercial solvent purification system. All other reagents and solvents were employed without further purification. The products were purified using a commercial flash chromatography system or a regular glass column. TLC was developed on Merck silica gel 60 F254 aluminum sheets. HRMS obtained at the CREAM Mass Spectrometry Facility, University of Louisville, The microwave reactor (CEM, model Discovery) was used in microwave condition.

General procedure for preparation of aminoalkyne 1

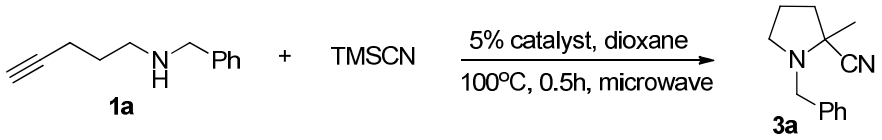


1a

N-Benzyl-4-pentyn-1-amine (**1a**). 4-pentyn-1-ol (25 mmol, 2.5 g) was dissolved in CH_2Cl_2 100 mL, and the solution was cooled down to -10°C, then methanesulfonyl chloride (2.15 mL, 28 mmol) and triethylamine (5.5 mL, 40mmol) are introduced by syringe successively. The mixture is stirred for 30 min at -10°C, and then it is poured onto ice-water (100 mL) during stirring. The organic layer is washed successively with 1 M hydrochloric acid solution (15 mL), saturated aqueous sodium bicarbonate solution (15ml), and brine (15 mL). The organic layer is dried over magnesium sulfate, filtered, and concentrated to crude 4-hexyn-1-yl methanesulfonate, which was used directly in the next step. Dimethyl sulfoxide (20 mL) and benzylamine (5.4 g, 50 mmol) are added to the above mentioned crude product. The resulting solution is heated in an oil bath at 47-53°C for 5 hr and then the reaction mixture is allowed to cool to room temperature. The reaction solution is poured into a separatory funnel containing 100 mL of aqueous

1% sodium hydroxide solution and the resulting mixture is extracted with ether (3×100 mL). The combined ether extracts are washed with brine (50 mL), dried over magnesium sulfate, and concentrated in a rotary evaporator under vacuum. The residue is purified by flash chromatography (1:1 hexane-ether containing 1% triethylamine as the eluent) to give a colorless liquid **1a** (3.6 g, 75%, 2 steps). The spectroscopic data are consisting with reference.[1]

General procedure for preparation of 3



1-Benzyl-2-methylpyrrolidine-2-carbonitrile (3a). N-Benzyl-4-pentyn-1-amine **1a** (43 mg, 0.25 mmol), trimethylsilyl cyanide (99 mg, 1.0 mmol), dioxane (1 mL) and water (4.5 mg, 1.0 equiv) were charged into a microwave tube reactor, and CuBr (1.8 mg, 5% equiv) was added to the mixture under stirring. After all the starting materials were dissolved, the microwave reactor was flushed with argon. Then the reaction mixture was heated to 100°C under microwave irradiation for 0.5 h. The resulting reaction mixture was directly concentrated in a rotary evaporator and purified by silica gel flash chromatography (4:1 hexane:ether) to give a yellow liquid **3a** (48 mg, 95%). IR (neat): 2978, 2943, 2810, 2216, 1455, 1369, 1159, 740, 700cm-1; ^{1}H NMR (400 MHz, $CDCl_3$): 1.42 (s, 3H), 1.62-1.81 (m, 3H), 2.21-2.39 (m, 2H), 2.90 (td, *J* = 8.4 Hz, 3.2 Hz, 1H), 3.22 (d, *J* = 13.2 Hz, 1H), 3.88 (d, *J* = 13.2 Hz, 1H), 7.11-7.22 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 19.9, 23.7, 38.9, 51.6, 54.5, 61.4, 120.0, 127.2, 128.4, 128.5, 138.5; ESI-HRMS (ESI+): m/z calcd. for $C_{13}H_{16}N_2$(M^+-CN) 174.1282, found 174.1277.

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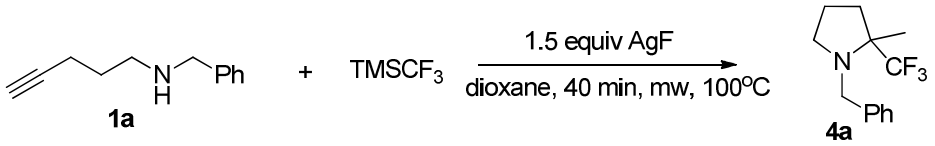
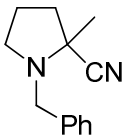
[1] H. Arnold, L. E. Overman, M. J. Sharp, and M. C. Witschel, Org. Synth. coll. vol. 9, p.46 (1998).

General procedure for preparation of 4.



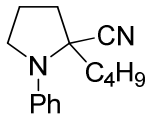
1-Benzyl-2-methyl-2-(trifluoromethyl)pyrrolidine (4a). N-Benzyl-4-pentyn-1-amine **1a** (43 mg, 0.25 mmol), trifluoromethyltrimethyl silane (71 mg, 0.5 mmol), dioxane (1 mL) and water (4.5 mg, 1.0 equiv) were charged into a microwave tube reactor, and AgF (48 mg, 1.5 equiv) was added to the mixture under stirring. After all the starting materials were dissolved, the microwave reactor was flushed with argon. Then the reaction mixture was heated to 100°C under microwave irradiation for 40 min. The resulting reaction mixture was directly concentrated in a rotary evaporator and purified by flash chromatography (20:1 hexane:ether) to give a yellow liquid **4a** (56 mg, 92%). IR(neat): 2946, 2843, 1460, 1470, 1144, 763, 690cm-1; 1H NMR (400 MHz, $CDCl_3$): δ 1.07 (s, 3H), 1.28-1.38 (m, 3H). 1.98-2.12 (m, 3H), 2.23-2.32 (m, 3H), 2.58 (t, J = 6.4 Hz, 1H), 3.35 (d, J = 13.6 Hz, 1H), 3.71 (d, J = 13.6 Hz, 1H), 6.98-7.05 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 17.6, 22.3, 35.7, 51.9, 53.4, 67.5 (q, J = 23.2 Hz), 126.7, 128.4 (q, J = 285 Hz), 128.0, 128.2, 140.3; ^{19}F NMR (370 MHz, $CDCl_3$): major isomer: δ -77.87; HRMS (ESI+): m/z calcd. for $C_{13}H_{16}F_3N$ (M^+- H) 242.1157, found 242.1153.

1-benzyl-2-methylpyrrolidine-2-carbonitrile (3a).



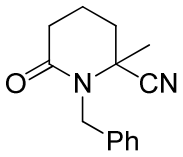
IR (neat): 2978, 2943, 2810, 2216, 1455, 1369, 1159, 740, 700cm-1; ^{1}H NMR (400 MHz, $CDCl_3$): 1.42 (s, 3H), 1.62-1.81 (m, 3H), 2.21-2.39 (m, 2H), 2.90 (td, J = 8.4 Hz, 3.2 Hz, 1H), 3.22 (d, J = 13.2 Hz, 1H), 3.88 (d, J = 13.2 Hz, 1H), 7.11-7.22 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): 19.9, 23.7, 38.9, 51.6, 54.5, 61.4, 120.0, 127.2, 128.4, 128.5, 138.5; HRMS (ESI+): m/z calcd. for $C_{13}H_{16}N_2$(M^+-CN) 174.1282, found 174.1277.

1-benzyl-2-methylpyrrolidine-2-carbonitrile (3b).



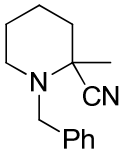
IR (neat): 2957, 2870, 2223, 1709, 1600, 1342, 1190, 996, 749 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.83 (t, *J* = 7.2 Hz, 3H), 1.18-1.32 (m, 3H), 1.39-1.60 (m, 2H), 1.94-2.16 (m, 3H), 2.22-2.31 (m, 1H), 2.47-2.54 (m, 1H), 3.33-3.40 (m, 1H), 3.50 (q, *J* = 14.8 Hz, 7.2 Hz, 1H), 6.75-6.85 (m, 3H), 7.17-7.25 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): d 13.8, 22.5, 22.8, 26.5, 35.6, 39.9, 50.7, 60.8, 115.2, 118.5, 121.3, 129.0, 144.5; HRMS (ESI+): m/z calcd. for $C_{13}H_{16}N_2$ (M^+-CN) 202.1596, found 202.1588.

1-benzyl-2-methyl-6-oxopiperidine-2-carbonitrile (3c).



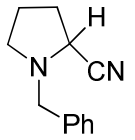
IR (neat): 2919, 1652, 1393, 1289, 731, 699cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.50 (s, 3H), 1.77-2.15 (m, 3H), 2.28-2.33 (m, 1H), 2.45-2.51 (m, 1H), 2.59-2.69 (m, 1H), 4.27 (d, *J* = 16 Hz, 1H), 5.26 (d, *J*=16Hz, 1H), 7.19-7.29 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): d 18.1, 27.8, 32.0, 36.8, 47.8, 57.4, 120.5, 126.9, 127.2, 128.6, 138.0, 170.1; HRMS (ESI+): m/z calcd. for $C_{14}H_{16}N_2O$(M^+- CN) 202.1232, found 202.1155.

1-benzyl-2-methylpiperidine-2-carbonitrile (3d)



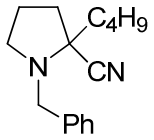
IR (neat): 2942, 2805, 1451, 1377, 1124, 737, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 1.45 (s, 3H), 1.42-1.61 (m, 3H), 1.80 (d, *J* = 10 Hz, 1H), 2.04-2.11(m, 1H), 2.60 (d, *J* = 12.4 Hz, 1H), 3.00 (d, *J* = 13.6 Hz, 1H), 3.99 (d, *J* = 13.6 Hz, 1H), 7.10-7.19 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 22.0, 25.2, 26.8, 38.5, 49.4, 55.8, 57.9, 119.4, 127.0, 128.3, 128.4, 138.8; HRMS (ESI+): m/z calcd. for $C_{14}H_{18}N_2$(M^+-CN) 188.1439, found 188.1433.

1-benzylpyrrolidine-2-carbonitrile (3e)



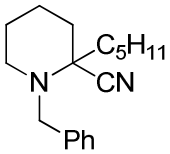
IR (neat):2906, 2 870, 1494, 1453, 1125, 744, 700 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.50 (s, 1H), 1.80-1.96 (m,2H), 2.01-2.14 (m, 2H), 2.52 (dd, *J* = 16.8 Hz, 8.8 Hz, 1H), 2.87 (td, *J* = 9.2 Hz, 4 Hz, 1H), 3.60 (d, *J* = 13.2 Hz, 1H), 3.85 (d, *J* = 12.8 Hz, 1H), 7.16-7.28 (m, 5H); ^{13}C NMR (100 MHz, CDCl3): δ 21.8, 29.5, 51.2, 53.2, 56.5, 118.0, 127.5, 128.5, 128.8, 137.6; HRMS (ESI+): m/z calcd. for $C_{12}H_{14}N_2(M^+$-CN) 160.1126, found 160.1120.

1-benzyl-2-butylpyrrolidine-2-carbonitrile (3f).



IR (neat):2957, 2871, 1690, 1454, 734, 700 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.87 (t, *J* = 7.2 Hz, 7H), 1.26-1.56 (m, 3H), 1.61-1.95 (m, 4H), 2.16-2.26 (m, 1H), 2.33 (q, *J* = 9.2 Hz, 1H), 2.91 (t, *J* = 8.8 Hz, 1H), 3.26 (d, *J* = 13.2 Hz, 1H), 3.97 (d, *J* = 12.8 Hz, 1H), 7.25 - 7.58 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.9, 20.1, 22.8, 26.7, 36.2, 36.5, 51.6, 54.5, 65.8, 119.9, 127.1, 128.3, 128.5, 138,6; HRMS (ESI+): m/z calcd. for $C_{16}H_{22}N_2$ (M^+-CN) 216.1752, found 216.1745.

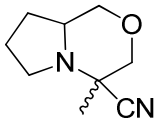
1-benzyl-2-pentylpiperidine-2-carbonitrile (3g).



IR (neat):2929, 2859, 1690, 1455, 1363, 1145, 737, 700cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.92 (t, *J*=6Hz, 3H), 1.28-1.60 (m, 7H), 1.75-2.04 (m, 4H), 2.26-2.33 (m, 1H), 2.42 (dd, *J* = 18 Hz, 9.2Hz, 1H), 2.94 (td, *J* = 8.4 Hz, 1.2Hz, 1H), 3.35 (d, *J* = 12.8 Hz, 1H), 4.06 (d, *J* = 12.8 Hz, 1H), 7.22-7.39 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 20.2, 22.5, 24.5,

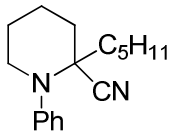
29.4, 31.6, 36.2, 36.8, 51.7, 54.6, 119.9, 127.2, 128.4, 128.5, 138.6; HRMS (ESI+): m/z calcd. for $C_{18}H_{26}N_2$ (M^+-CN) 244.2065, found 244.2060.

4-methylhexahydro-1H-pyrrolo[2,1-c][1,4]oxazine-4-carbonitrile (3h)



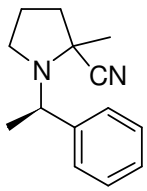
IR (neat): 2900, 2830, 1730, 1600, 1423, 1210, 925, 730 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$), major isomer: δ 1.30 (s, 3H), 1.52-1.79 (m, 4H), 2.31 (dd, *J* = 16.8 Hz, 8 Hz, 1H), 2.45-2.49 (m, 1H), 3.02-3.17 (m, 3H), 3.81 (d, *J* = 11.6 Hz, 1H) 3.97 (dd, *J* = 10.8 Hz, 3.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): ^{13}C NMR (100 MHz, $CDCl_3$), major isomer: δ 20.0, 21.0, 25.7, 47.3, 57.8, 58.0, 72.0, 73.2, 118.1; HRMS (ESI+): m/z calcd. for $C_9H_{14}N_2O$ (M^+-CN) 140.1075, found 140.1071.

2-pentyl-1-phenylpiperidine-2-carbonitrile (3i)



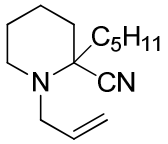
IR (neat): 2956, 2859, 1705, 1342, 1191, 750, 695cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 0.79 (t, *J*=6.4Hz, 3H), 1.16-1.36 (m, 7H), 1.42-1.59 (m, 2H),1.94-2.17 (m, 3H), 2.21-2.28 (m, 1H), 2.47-2.53 (m, 1H), 3.33-3.38 (m, 1H), 3.46-3.52 (m, 1H), 6.76 (t, *J*=7.2Hz, 1H), 6.80-6.84 (m, 2H), 7.17-7.23 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): 14.0, 22.5, 22.8, 24.4, 29.0, 31.5, 35.8, 39.9, 50.7, 60.8, 115.2, 118.5, 121.3, 129.0, 144.5; HRMS (ESI+): m/z calcd for $C_{17}H_{24}N_2$ (M^+-CN) 230.1909, found 230.1903.

1-isopropyl-2-methylpyrrolidine-2-carbonitrile (3k).



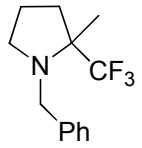
IR (neat): 2942, 2805, 1451, 1377, 1124, 737, 699cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$), major isomer: δ 0.95 (s, 3H), 1.40 (d, *J* = 6.4 Hz, 3H), 2.28-2.33 (m, 1H), 2.45-2.51 (m, 1H), 2.59-2.69 (m, 1H), 4.27 (d, *J* = 16 Hz, 1H), 5.26 (d, *J* = 16 Hz, 1H), 7.19-7.29 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.1, 27.8, 32.0, 36.8, 47.8, 57.4, 120.5, 126.9, 127.2, 128.6, 138.0, 170.1; HRMS (ESI+) (ESI^{+}): m/z calcd. for $C_{14}H_{18}N_2$ (M^{+}- CN) 188.1439, found 188.1436.

1-allyl-2-pentylpiperidine-2-carbonitrile (3l).



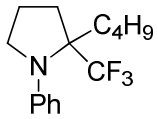
IR (neat): 2955, 2859, 1643, 1452, 1161, 993, 921cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.82 (t, *J*=6Hz, 3H), 1.18-1.50 (m, 1H), 1.72-1.86 (m,1H), 2.28-2.35 (m, 1H), 2.28-2.34 (m,1H), 2.80 (dd, *J* = 13.2 Hz, 8 Hz, 1H), 3.13 (t, *J* = 7.6 Hz, 1H), 3.39 (dd, *J* = 13.2 Hz, 4Hz, 1H), 5.06 (d, *J* = 10.4 Hz, 1H), 5.20 (d, *J* = 16.8 Hz, 1H), 5.72-5.82 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 20.0, 22.5, 24.6, 29.3, 31.6, 36.3, 36.8, 51.7, 53.3, 65.8, 117.3, 119.7, 135.3; HRMS (ESI+): m/z calcd. for $C_{14}H_{24}N_2$ (M^{+}- CN) 194.1909, found 194.1903.

1-benzyl-2-methyl-2-(trifluoromethyl)pyrrolidine (4a).



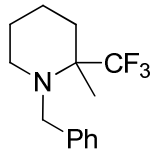
IR (neat): 2946, 2843, 1460, 1470, 1144, 763, 690 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.07 (s, 3H), 1.28-1.38 (m, 3H). 1.98-2.12 (m, 3H), 2.23-2.32 (m, 3H), 2.58 (t, *J* = 6.4 Hz, 1H), 3.35 (d, *J* = 13.6 Hz, 1H), 3.71 (d, *J* = 13.6 Hz, 1H), 6.98-7.05 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 17.6, 22.3, 35.7, 35.7 (d, *J* = 1.5 Hz), 51.9, 53.4, 67.5 (q, *J* = 23.2 Hz), 126.7, 128.4 (q, *J* = 285 Hz), 128.0, 128.2, 140.3; ^{19}F NMR (370 MHz, $CDCl_3$): major isomer: δ -77.87; HRMS (ESI+): m/z calcd. for $C_{13}H_{16}F_3N$ (M^{+}-H) 242.1157, found 242.1153.

2-butyl-1-phenyl-2-(trifluoromethyl)pyrrolidine (4b).



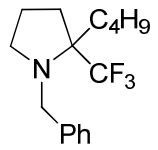
IR (neat): 2960, 2874, 1720, 1601, 1504, 1329, 1151, 751, 694 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.82 (t, *J* = 6.8 Hz, 3H), 1.06-1.32 (m, 4H), 1.73-1.84 (m, 1H), 1.84-1.96 (m, 1H), 1.98-2.15 (m, 2H), 2.23-2.39 (m, 2H), 3.42-3.55 (m, 2H), 6.79 (t, *J*=7.6Hz, 1H), 6.96 (d, *J*=8.4Hz, 1H), 7.22 (t, *J* = 8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.8, 22.1, 22.7, 24.4, 31.1, 34.8 (d, *J*=6.4Hz), 68.9 (q, *J* = 24.8 Hz), 116.0 (q, *J* = 2.4 Hz), 128.1 (q, *J* = 288.9 Hz), 128.7, 146.1; ^{19}F NMR (370 MHz, $CDCl_3$): major isomer: δ -73.20; HRMS (ESI+): m/z calcd. for $C_{15}H_{20}F_3N$ (M^+- H)270.1470, found 270.1465.

1-benzyl-2-methyl-2-(trifluoromethyl)piperidine (4d)



IR (neat): 2943, 2853, 2857, 1454, 1369, 1253, 1139, 725, 697 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.28 (s, 3H), 1.35-1.59 (m, 5H), 1.89-1.98 (m, 1H), 2.26-2.45 (m, 1H), 2.55-2.64 (m, 1H), 3.48 (d, *J* = 14.8 Hz, 1H), 3.99 (d, *J* = 14.8 Hz, 1H), 7.14-7.27 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.5 25.7 (d, *J* = 6 Hz), 32.8, 46.5, 54.2, 59.1 (q, *J* = 20.9 Hz), 126.6, 127.8, 128.2, 129.4 (q, *J* = 295.1 Hz), 140.9; ^{19}F NMR (370 MHz, $CDCl_3$): major isomer: δ -67.82; HRMS (ESI+): m/z calcd. for $C_{14}H_{18}F_3N$ (M^+- H) 256.1313, found 256.1310.

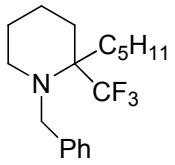
1-benzyl-2-butyl-2-(trifluoromethyl)pyrrolidine (4f).



IR (neat): 2958, 2874,1455, 1372, 1286, 1143, 731, 698 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.80 (t, *J*= 6.4Hz, 3H), 1.18- 1.36 (m, 4H), 1.50-1.63 (m, 4H), 1.75-1.80 (m, 1H), 1.90-1.96 (m, 1H), 2.58 (t, *J* = 6.4 Hz, 1H), 3.64 (d, *J* = 14.4 Hz, 1H), 3.78 (d,

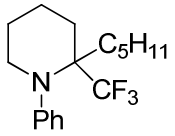
J=13.6Hz, 1H), 7.04-7.18 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.1 (q, *J* = 21.7 Hz), 22.2, 23.3, 25.5, 30.8, 31.2, 51.5, 52.1 (t, *J* = 11 Hz), 67.5 (q, *J* = 23.2 Hz), 126.7 (d, *J* = 43.4 Hz), 127.9 (d, *J* = 27.9 Hz), 128.3 (d, *J* = 29.4 Hz), 129.0 (q, *J* = 290 Hz), 140.4; ^{19}F NMR (370 MHz, $CDCl_3$): major isomer: δ -73.95; HRMS (ESI+): m/z calcd. for $C_{16}H_{22}F_3N$ (M^+-H) 284.1626, found 284.1622.

1-benzyl-2-pentyl-2-(trifluoromethyl)piperidine (4g)



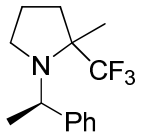
IR (neat): 2955, 2932, 2857, 1453, 1360, 1142, 740, 698 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.91 (t, *J* = 6 Hz, 3H), 1.33-1.56 (m, 8H), 1.64-1.83 (m, 4H), 1.88-1.98 (m, 1H), 2.06-2.15 (m, 1H), 2.74 (t, *J* = 6.8 Hz, 2H), 3.81 (d, *J*=13.6Hz, 1H), 3.94 (d, *J* = 13.6 Hz, 1H), 7.22-7.32 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 22.2, 22.6, 23.3, 30.0, 31.1, 31.2, 31.8, 51.5, 52.1 (d, *J* = 1.6 Hz), 67.6 (q, *J* = 23.2 Hz), 126.7, 127.9, 128.2, 129.0 (q, *J* = 290.5 Hz), 140.4; ^{19}F NMR (370 MHz, $CDCl_3$), major isomer: δ -73.95; HRMS (ESI+): m/z calcd. for $C_{18}H_{26}F_3N$ (M^+-H): 312.1939, found 312.1934.

2-pentyl-1-phenyl-2-(trifluoromethyl)piperidine (4i).



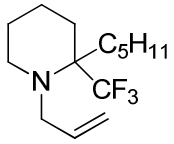
IR (neat): 2957, 2930, 1720, 1560, 1504, 1330, 1152, 751, 695 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.83(t, *J* = 6.8 Hz, 3H), 1.06-1.30 (m, 8H), 1.73-1.83 (m, 1H), 1.94-2.13 (m, 2H), 2.23-2.38 (m, 2H), 3.39-3.53 (m, 2H), 6.82 (t, *J* = 7.6 Hz, 1H), 6.95 (d, *J* = 8.4 Hz, 2H), 7.22 (t, *J* = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 13.9, 22.1, 22.2, 22.5, 29.3, 31.4, 31.5, 34.7 (d, *J* = 1.5 Hz), 52.3, 68.9 (q, *J* = 25.6 Hz), 116.0 (t, *J* = 2.3 Hz), 118.6, 128.1 (q, *J* = 288.9 Hz), 128.7, 146.1; ^{19}F NMR (370 MHz, $CDCl_3$): major isomer: δ -73.20; HRMS (ESI+): m/z calcd for $C_{15}H_{20}F_3N$ (M^+- H)298.1783, found 298.1778.

2-methyl-1-((R)-1-phenylethyl)-2-(trifluoromethyl)pyrrolidine (4i).



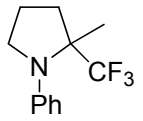
IR (neat): 2981, 2361, 1700, 1456, 1383, 1166, 699cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.23 (s, 3H), 1.40 (d, *J* = 6.4 Hz, 3H), 1.52-1.80 (m, 2H), 1.79-1.90 (m, 2H), 2.20-2.29 (m, 2H), 2.68-2.74 (m,1H), 3.10-3.14 (m, 1H), 3.84-3.87 (m ,1H), 7.09-7.38 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.8, 23.5, 38.7, 51.5, 54.4, 61.3, 119.9, 125.2, 127.1, 128.3 (q, *J* = 290 Hz), 128.9, 138.4; ^{19}F NMR (370 MHz, $CDCl_3$): major isomer: δ -74.33; HRMS (ESI+): m/z calcd. for $C_{14}H_{24}F_3N$ (M^+- H) 256.1313, found 256.1310.

1-allyl-2-pentyl-2-(trifluoromethyl)piperidine (4l)



IR (neat): 2957, 2858, 1457, 1144, 918, 699 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.752 (t, *J* = 6 Hz, 3H), 1.11-1.22 (m, 6H), 1.42-1.50 (m, 2H), 1.52-1.74 (m, 3H), 1.84-1.92 (m ,1H), 2.64-2.72 (m, 1H), 2.72-2.80 (m, 1H), 3.07-3.27 (m, 2H), 4.90 (d, *J* = 18 Hz, 1H), 5.07 (dd, *J* = 17.2 Hz, 1.6 Hz), 5.59-5.70 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.0, 22.1, 22.6, 23.0, 29.8, 30.8, 31.2, 31.7, 50.7, 51.6, 67.2 (q, *J* = 23.1 Hz), 115.4, 129.0 (q, *J* = 290.9 Hz), 137.3; ^{19}F NMR (370 MHz, $CDCl_3$), major isomer: δ -74.13; HRMS (ESI+): m/z calcd. for $C_{14}H_{24}F_3N$ (M^++ H): 264.1939, found 264.1935.

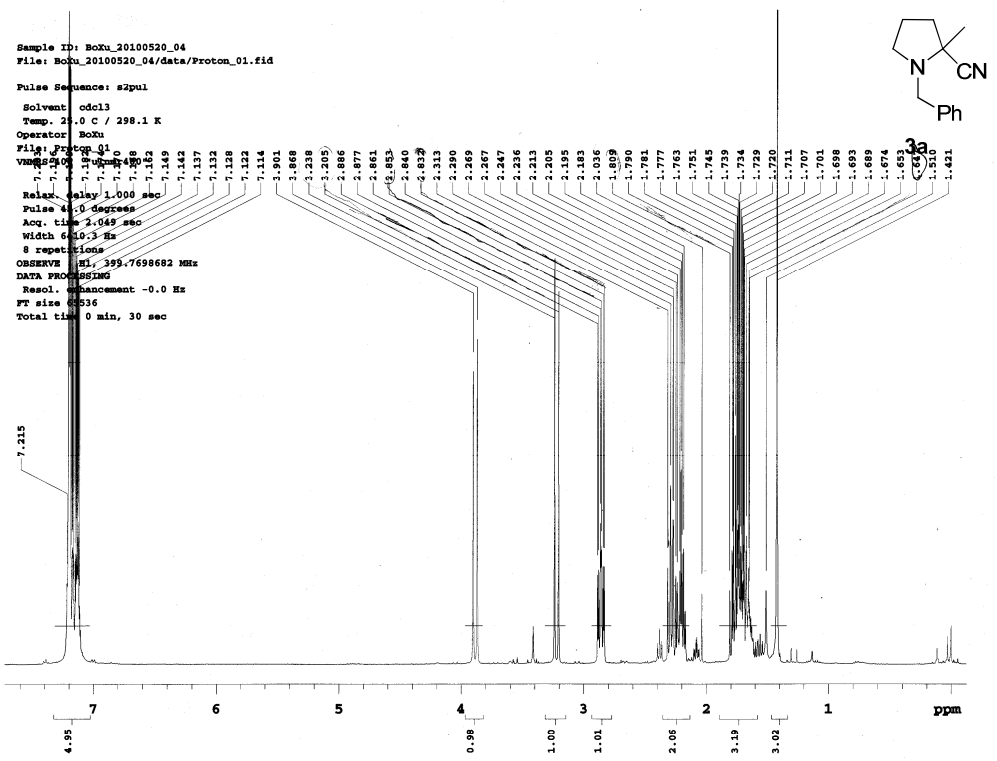
2-methyl-1-phenyl-2-(trifluoromethyl)pyrrolidine (4n).

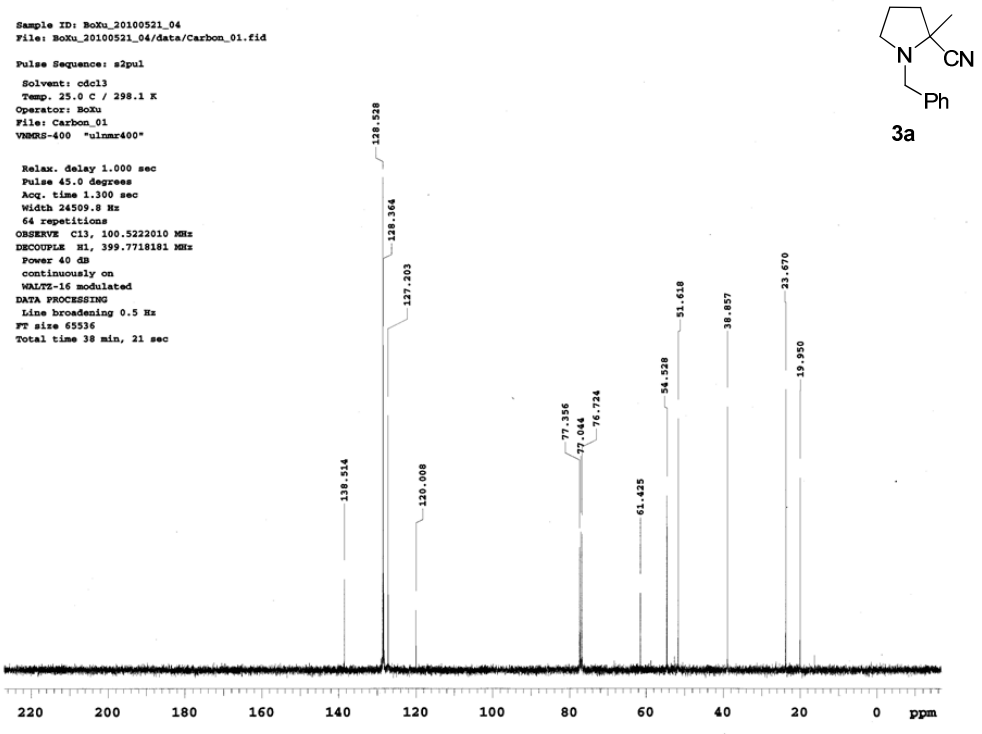


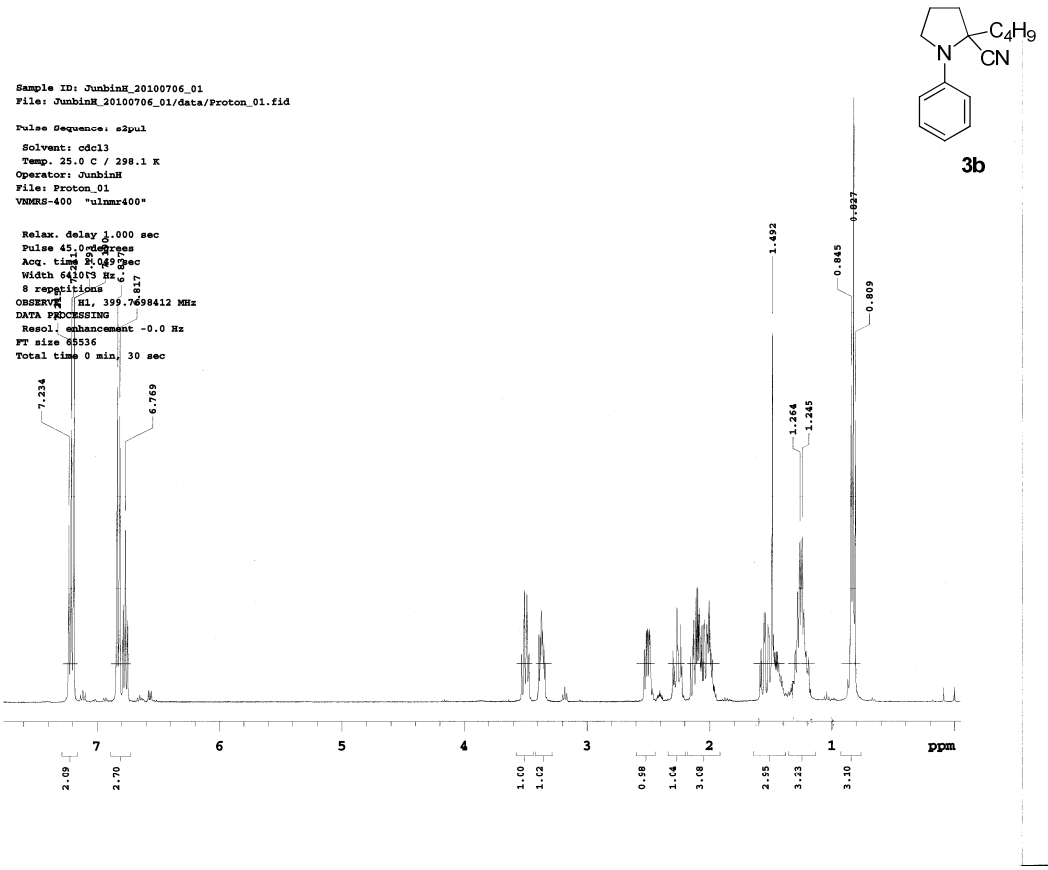
IR (neat): 2984, 2922, 2851, 1601, 1504, 1319, 1154, 751, 695 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.24 (s, 3H), 1.55-1.60 (m, 2H), 1.73-1.86 (m, 1H), 2.19-2.28 (m, 1H), 3.14-3.28 (m, 2H), 6.58 (t, *J* = 7.6 Hz, 1H), 6.73 (d, *J* = 8.4 Hz, 1H), 6.93-6.99 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.1, 22.1 (d, *J* = 1.6 Hz), 38.7 (d, *J* = 1.5 Hz), 51.7, 65.8 (q, *J*

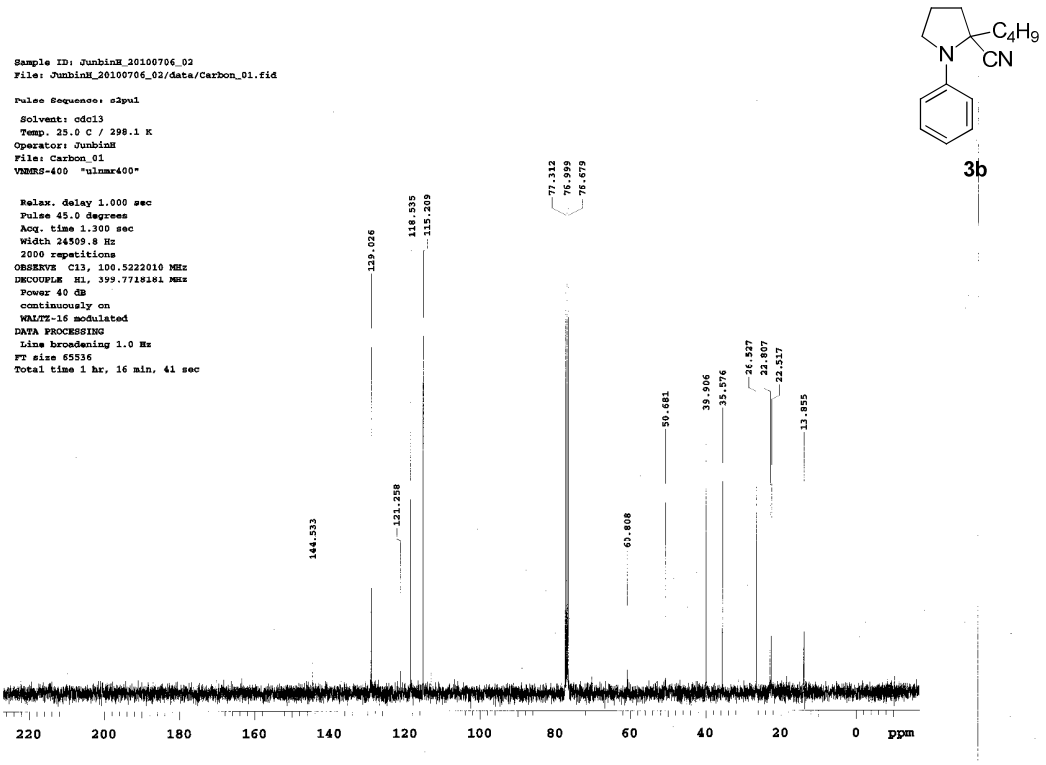
= 27.1 Hz), 117.3 (q, *J* = 3.1 Hz), 127.8 (q, *J* = 286.6 Hz), 128.6, 145.6; ^{19}F NMR (370 MHz, $CDCl_3$): major isomer: δ -74.69; HRMS (ESI+): m/z calcd. for $C_{12}H_{14}F_3N$ (M^+-H) 228.1000, found 228.0996.

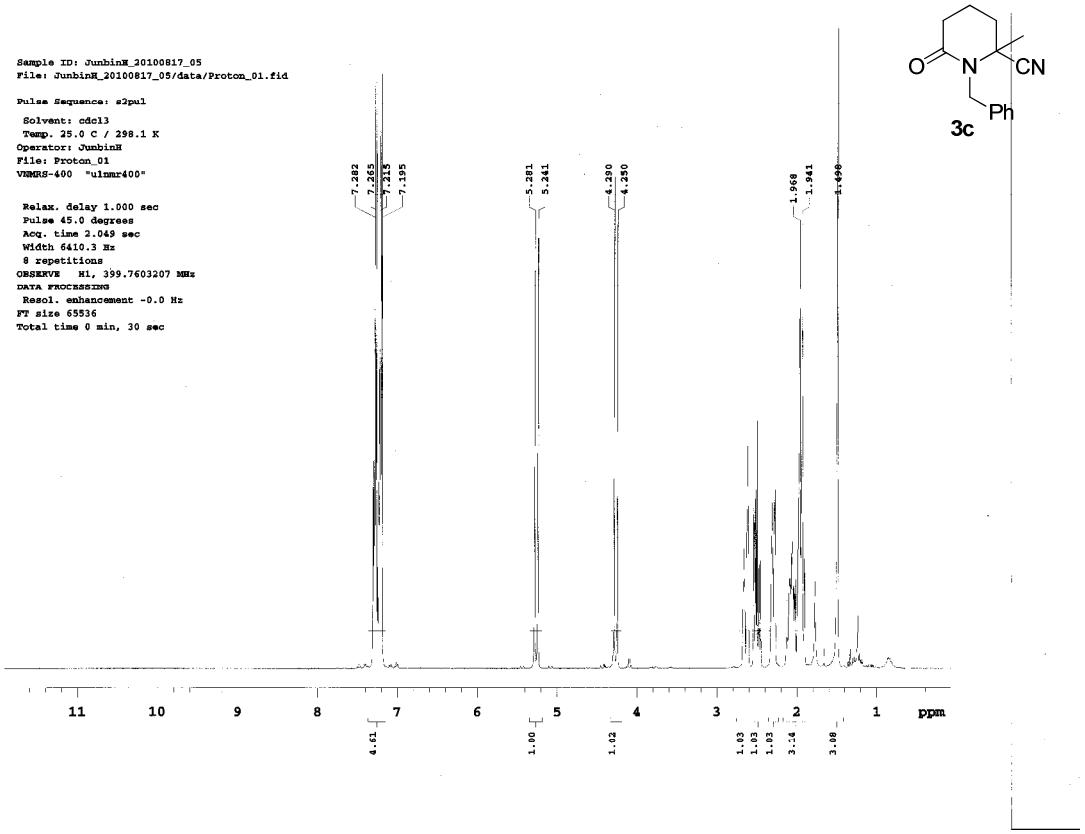
^{1}H and ^{13}C NMR spectra of compounds 3 and 4

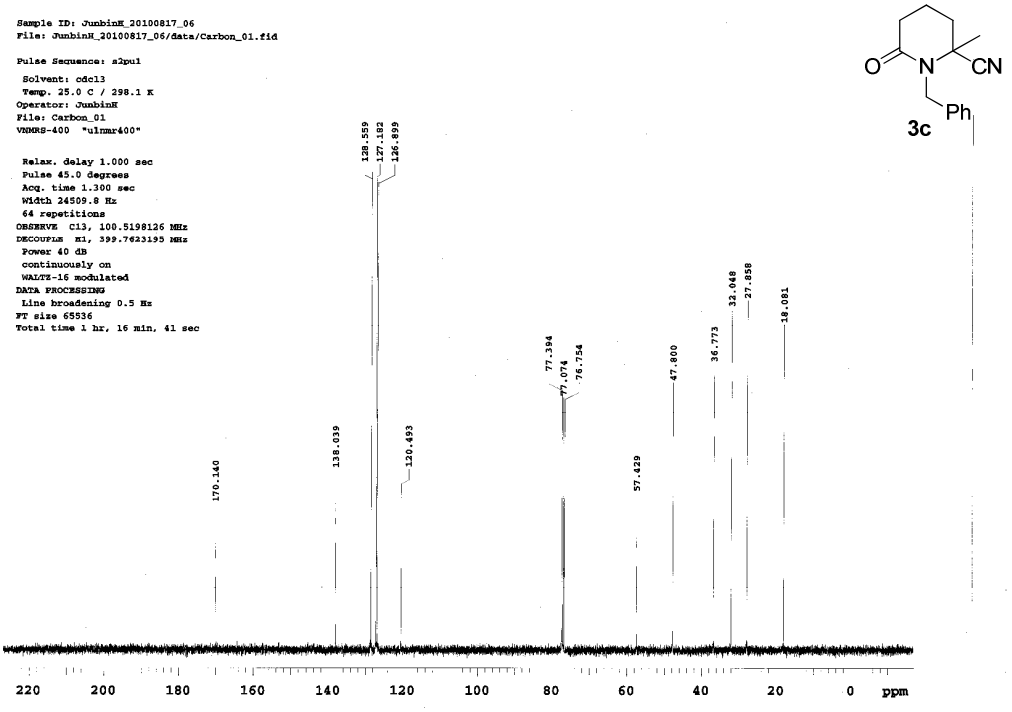

Sample ID: BoXu_20100520_04
File: BoXu_20100520_04/data/Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: BoXu
File: Proton_01
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6610.3 Hz
8 repetitions
OBSERVE H1, 399.7698682 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec
7.215
7 6 5 4 3 2 1 ppm
4.95 0.98 1.00 1.01 2.06 3.19 3.02
N CN Ph
3a

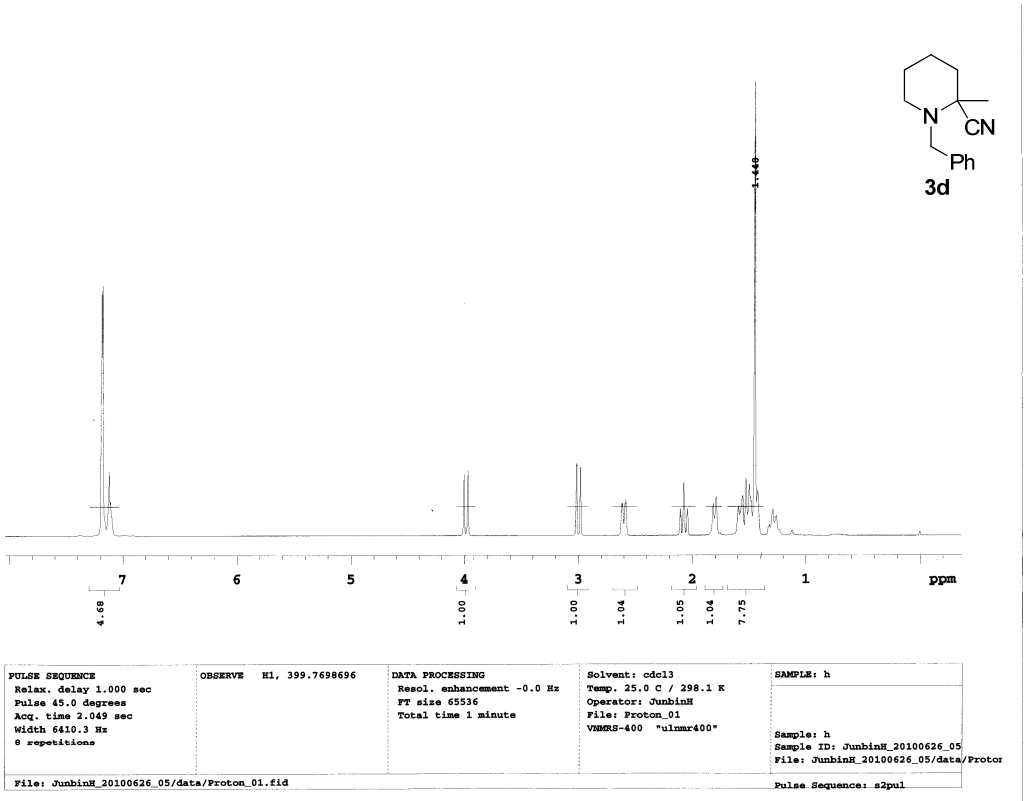

Sample ID: BoXu_20100521_04
File: BoXu_20100521_04/data/Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: BoXu
File: Carbon_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
64 repetitions
OBSERVE C13, 100.5222010 MHz
DECOUPLE H1, 399.7718181 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 min, 21 sec
128.528
128.364
127.203
77.356
77.044
76.724
54.528
51.618
38.857
23.670
19.950
61.425
138.514
120.008
220 200 180 160 140 120 100 80 60 40 20 0 ppm
N CN Ph
3a

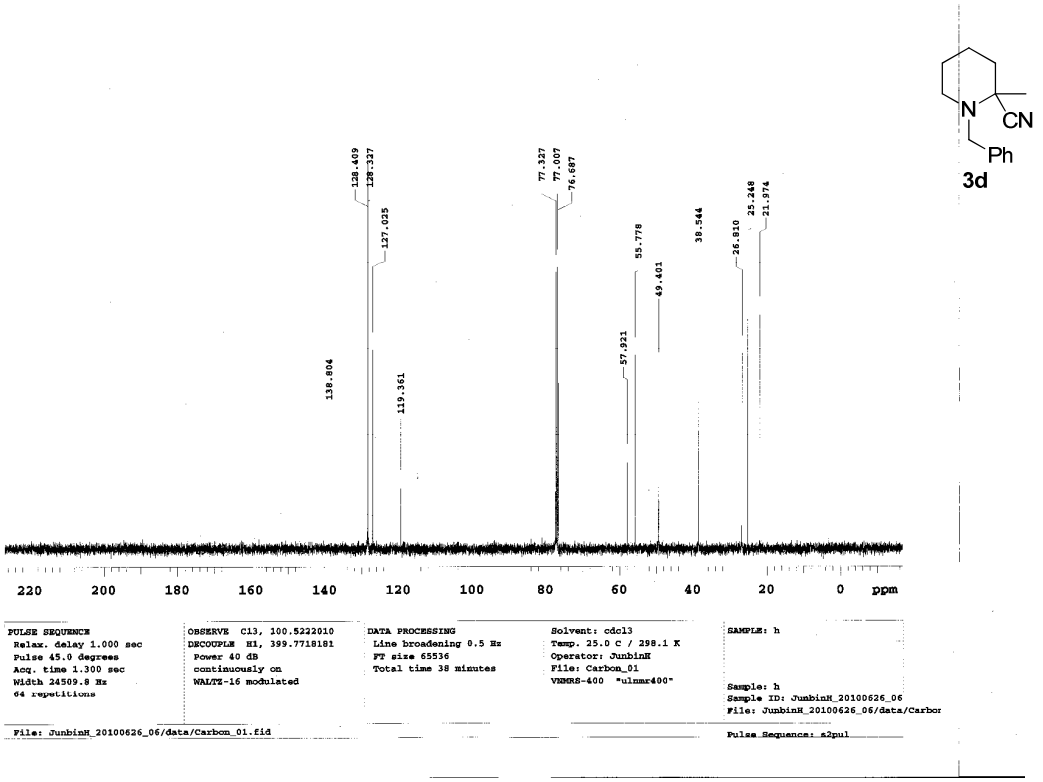

Sample ID: JunbinH_20100706_01
File: JunbinH_20100706_01/data/Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Proton_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7698412 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec
7.234
6.769
1.492
0.845
0.827
0.809
1.264
1.245
7
6
5
4
3
2
1
ppm
2.09
2.70
1.00
1.02
0.58
1.04
3.08
2.55
3.23
3.10
3b

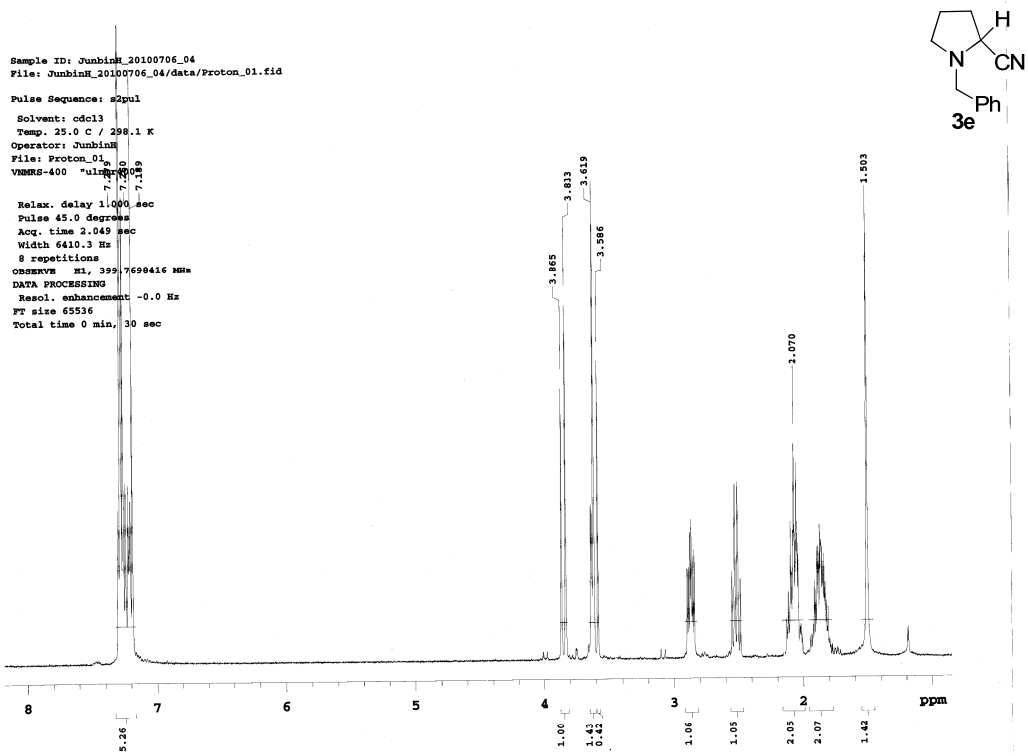

Sample ID: JunbinH_20100706_02
File: JunbinH_20100706_02/data/Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Carbon_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
2000 repetitions
OBSERVE C13, 100.5222010 MHz
DECOUPLE H1, 399.7718181 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 16 min, 41 sec
144.533
129.026
121.258
118.535
115.209
77.312
76.999
76.679
63.808
50.681
39.906
35.576
26.527
22.807
22.517
13.855
220
200
180
160
140
120
100
80
60
40
20
0
ppm
3b

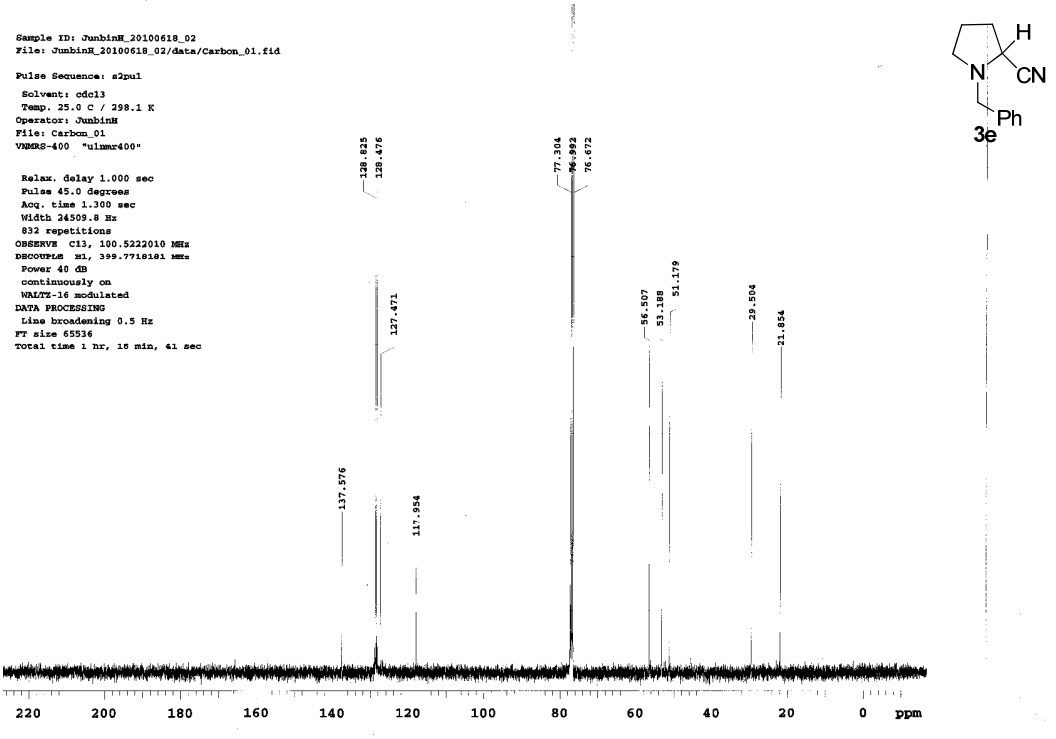

Sample ID: JunbinH_20100817_05
File: JunbinH_20100817_05/data/Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Proton_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7603207 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec
7.282
7.265
7.215
7.195
5.281
5.241
4.290
4.250
1.968
1.941
1.490
11 10 9 8 7 6 5 4 3 2 1 ppm
4.61
1.00
1.02
1.03
1.03
1.03
3.14
3.08
O
N
CN
Ph
3c

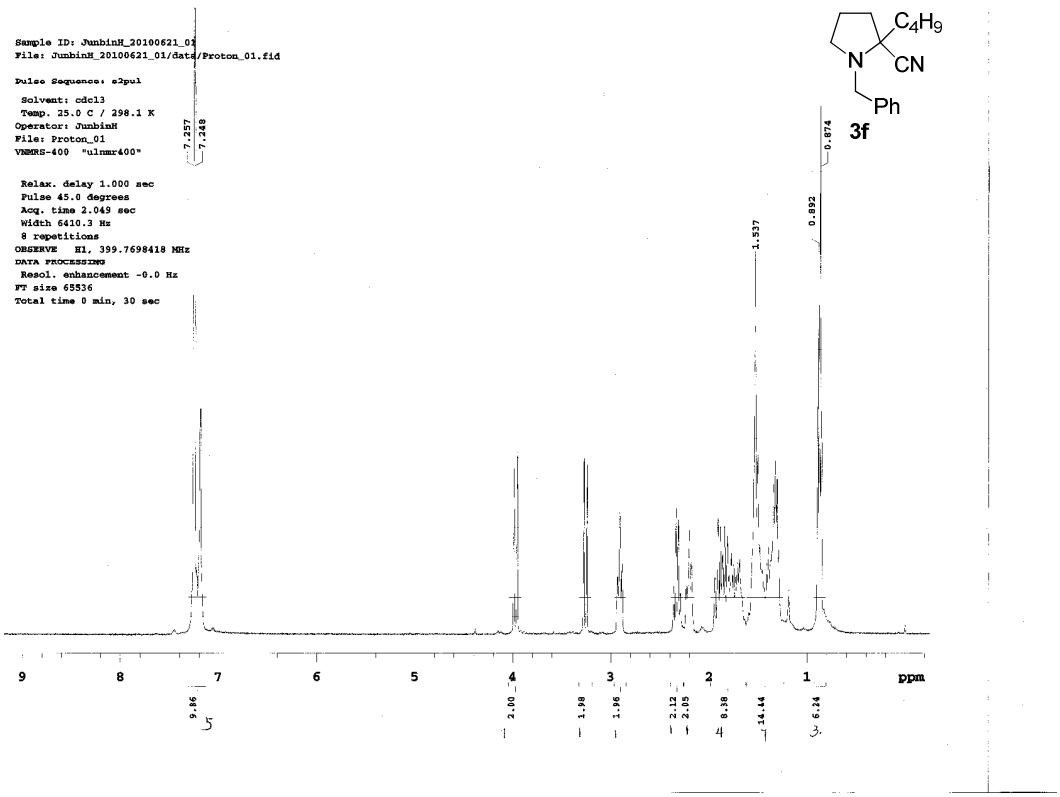

Sample ID: JunbinH_20100817_06
File: JunbinH_20100817_06/data/Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Carbon_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
64 repetitions
OBSERVE C13, 100.5198126 MHz
DECOUPLE H1, 399.7623195 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 41 sec
128.559
127.182
126.899
170.140
138.039
120.493
77.394
77.074
76.754
57.429
47.800
36.773
32.048
27.858
18.081
220 200 180 160 140 120 100 80 60 40 20 0 ppm
O
N
CN
Ph
3c

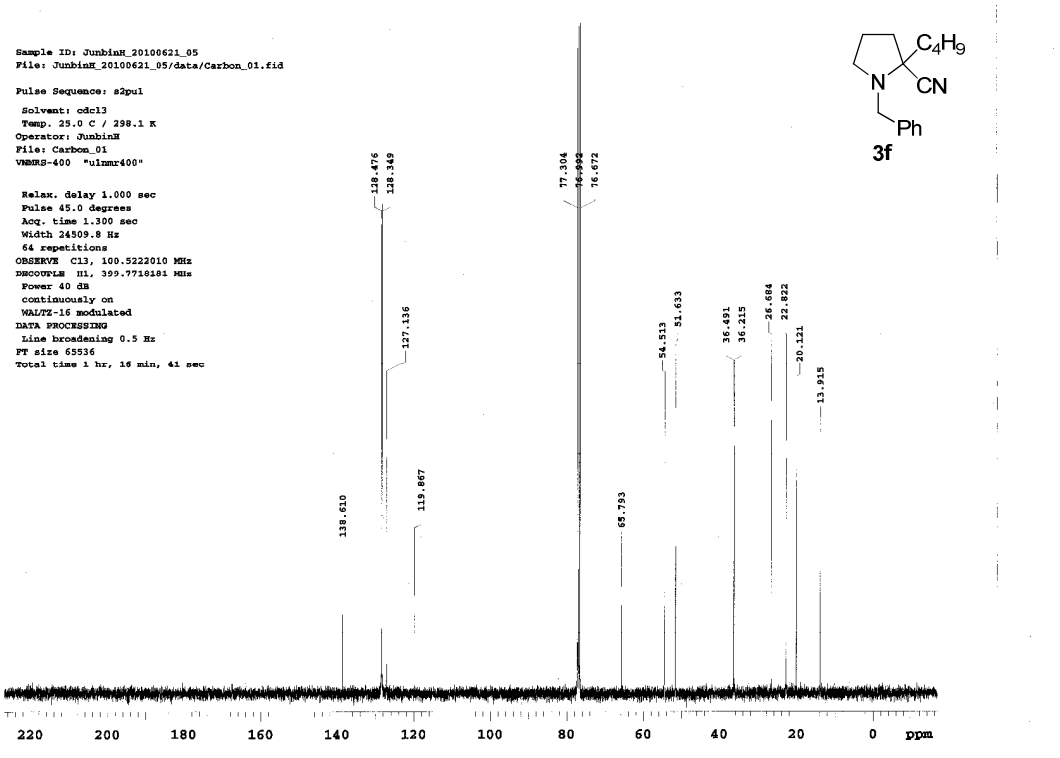

N
CN
Ph
3d
1.448
7
6
5
4
3
2
1
ppm
4.68
1.00
1.00
1.04
1.05
1.04
7.75
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7698696
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 1 minute
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Proton_01
VNMRS-400 "ulnmr400"
SAMPLE: h
Sample: h
Sample ID: JunbinH_20100626_05
File: JunbinH_20100626_05/data/Proton
File: JunbinH_20100626_05/data/Proton_01.fid
Pulse Sequence: s2pul

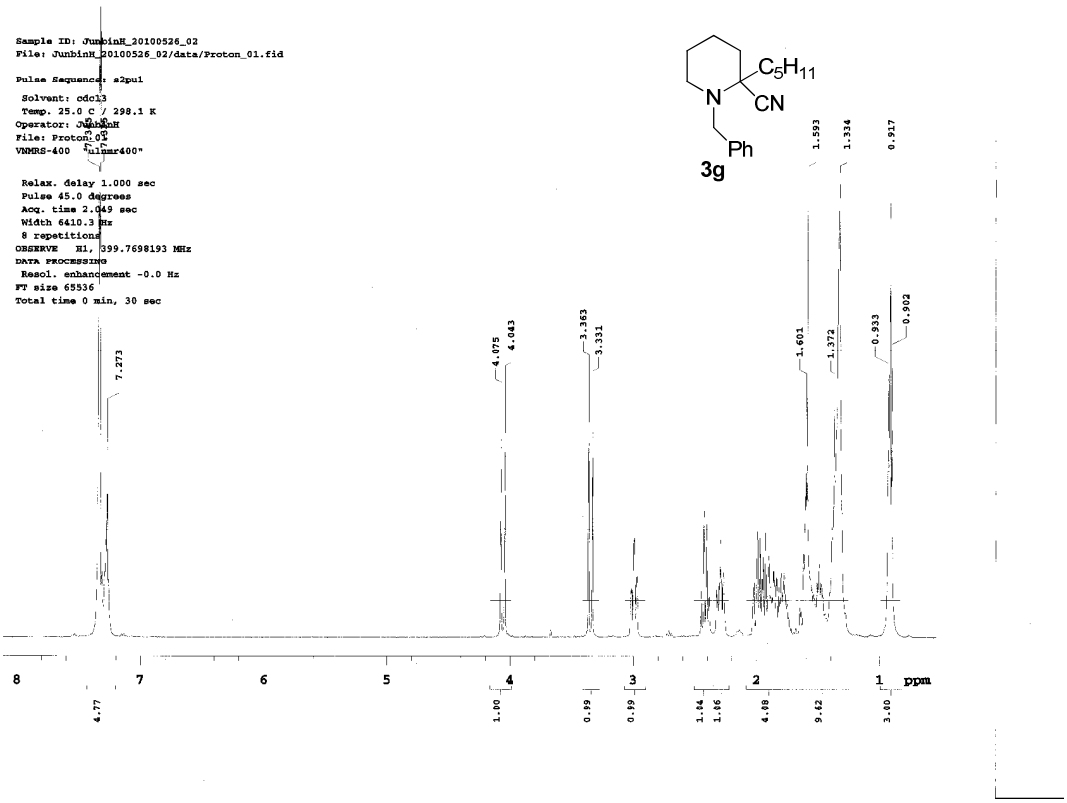

N
CN
Ph
3d
138.804
129.409
128.337
127.025
119.361
77.327
77.007
76.687
57.921
55.778
49.401
39.544
26.810
25.248
21.974
220
200
180
160
140
120
100
80
60
40
20
0
ppm
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
64 repetitions
OBSERVE C13, 100.5222010
DECOUPLE H1, 399.7718181
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 minutes
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Carbon_01
VNMRS-400 "ulnmr400"
SAMPLE: h
Sample: h
Sample ID: JunbinH_20100626_06
File: JunbinH_20100626_06/data/Carbon
File: JunbinH_20100626_06/data/Carbon_01.fid
Pulse Sequence: s2pul

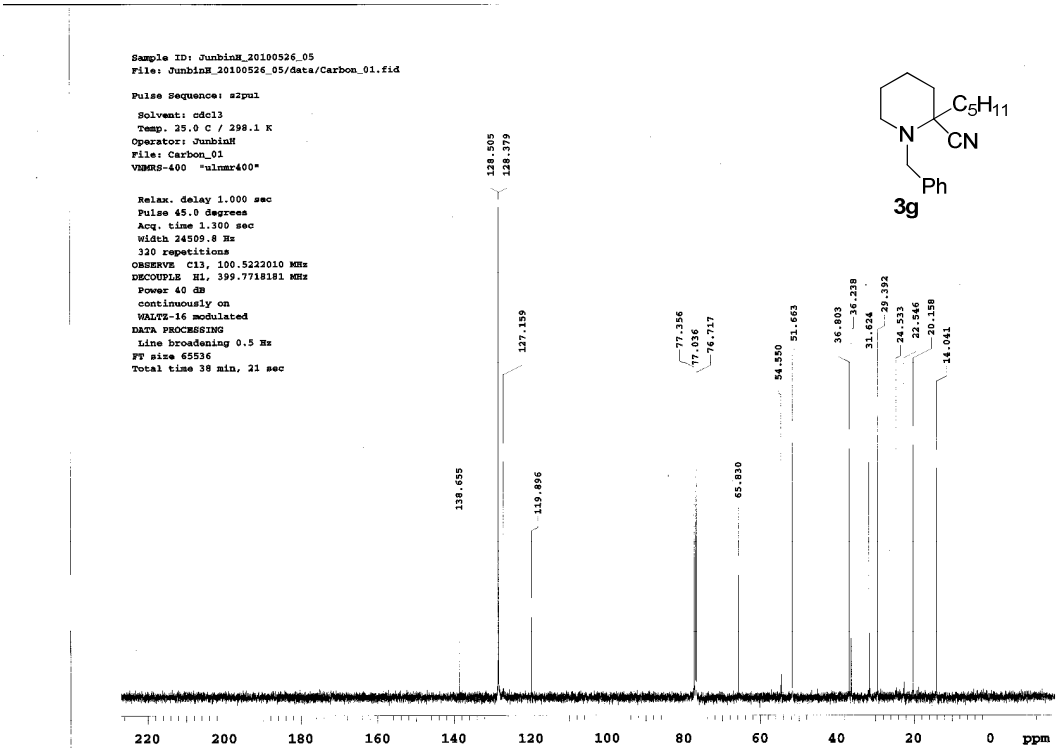

Sample ID: JunbinH_20100706_04
File: JunbinH_20100706_04/data/Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Proton_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7698416 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec
7.299
7.280
7.259
7.189
3.865
3.833
3.619
3.586
2.070
1.503
8
7
6
5
4
3
2
ppm
5.26
1.00
1.43
0.42
1.06
1.05
2.05
2.07
1.42
H
N
CN
Ph
3e

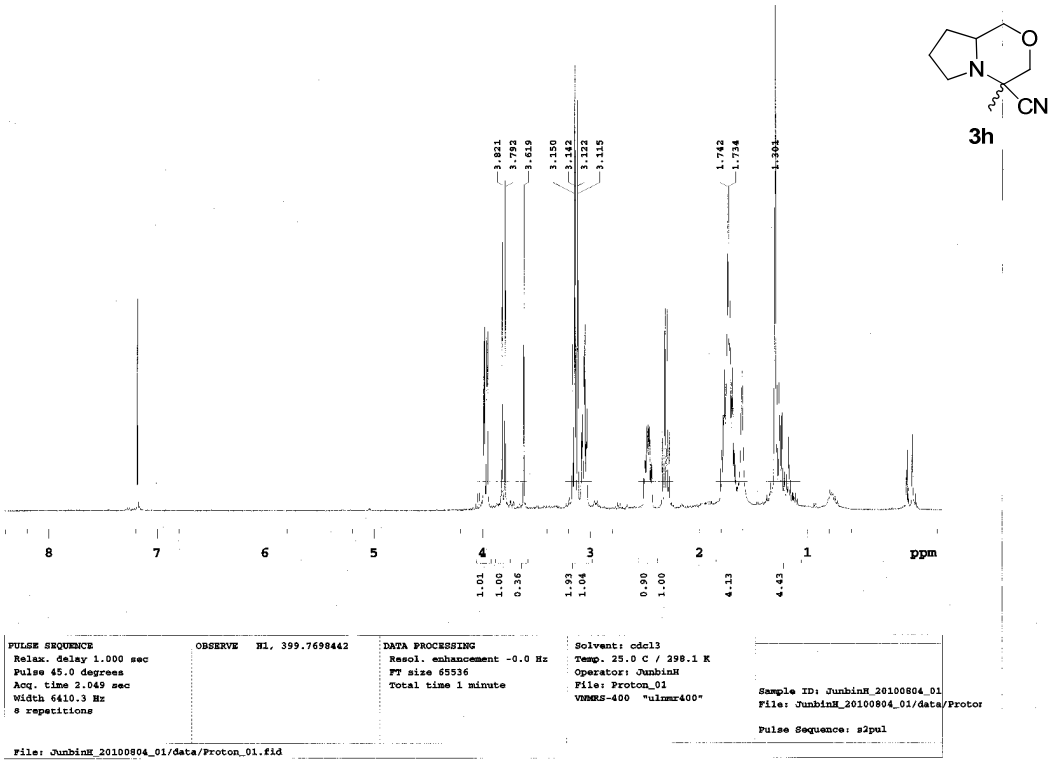

Sample ID: JunbinH_20100618_02
File: JunbinH_20100618_02/data/Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Carbon_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
832 repetitions
OBSERVE C13, 100.5222010 MHz
DECOUPLE H1, 399.7718181 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 15 min, 41 sec
128.825
128.476
127.471
137.576
117.954
77.304
76.992
76.672
56.507
53.188
51.179
29.504
21.854
220
200
180
160
140
120
100
80
60
40
20
0
ppm
H
N
CN
Ph
3e

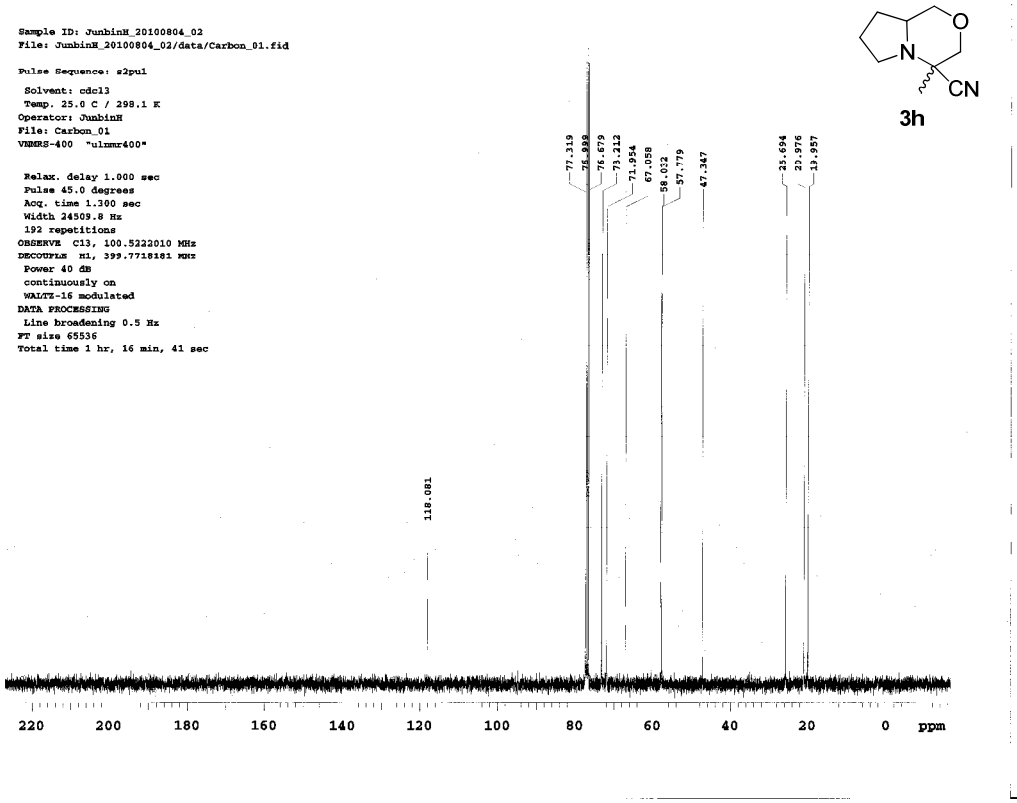

Sample ID: JunbinH_20100621_01
File: JunbinH_20100621_01/data/Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Proton_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7698418 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec
7.257
7.248
1.537
0.892
0.874
9.16
2.00
1.98
1.96
2.12
2.05
8.38
14.44
6.24
ppm
C4H9
N
CN
Ph
3f

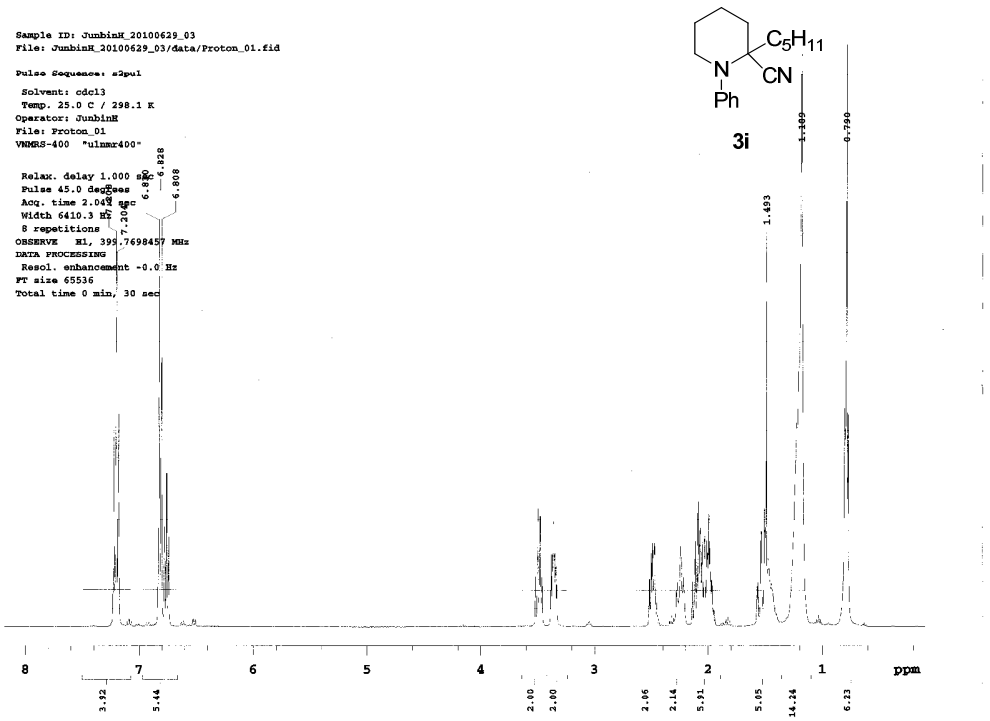

Sample ID: JunbinH_20100621_05
File: JunbinH_20100621_05/data/Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Carbon_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
64 repetitions
OBSERVE C13, 100.5222010 MHz
DECOUPLE H1, 399.7716181 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 41 sec
138.476
128.349
127.136
119.867
138.610
77.304
76.992
76.672
63.793
54.513
51.633
36.491
36.215
26.684
22.822
20.121
13.915
ppm
C4H9
N
CN
Ph
3f

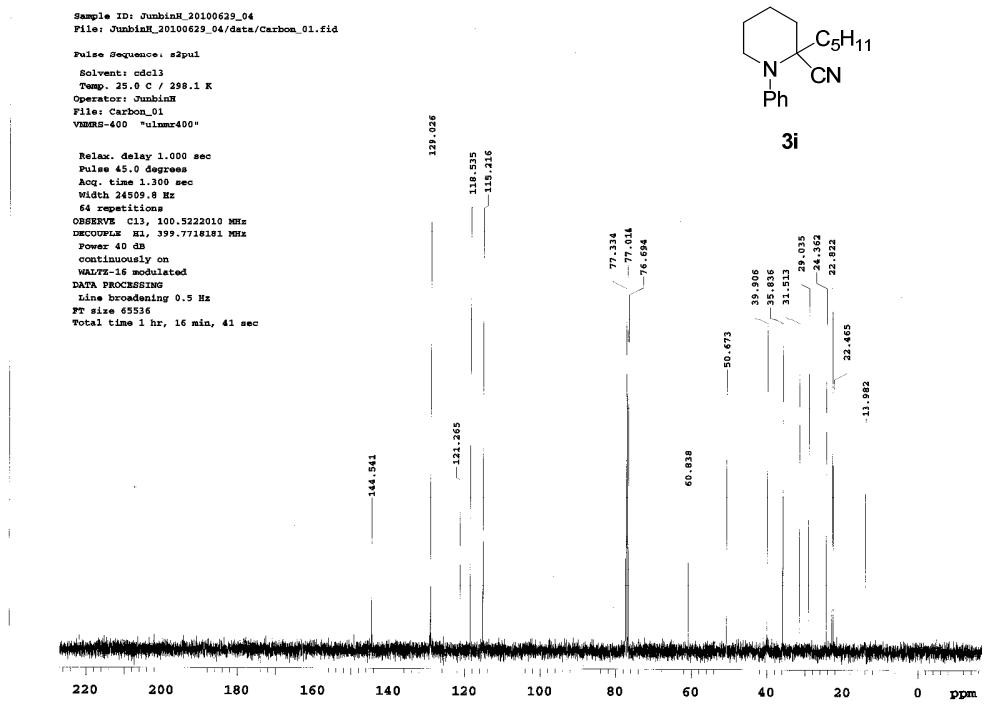

Sample ID: JunbinH_20100526_02
File: JunbinH_20100526_02/data/Proton_01.fid
Pulse Sequence: s2pu1
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Proton_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7698193 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec
7.273
4.075
4.043
3.363
3.331
1.601
1.372
1.593
1.334
0.933
0.902
0.917
ppm
4.77
1.00
0.99
0.99
1.84
1.86
4.08
9.42
3.40
C5H11
N
CN
Ph
3g

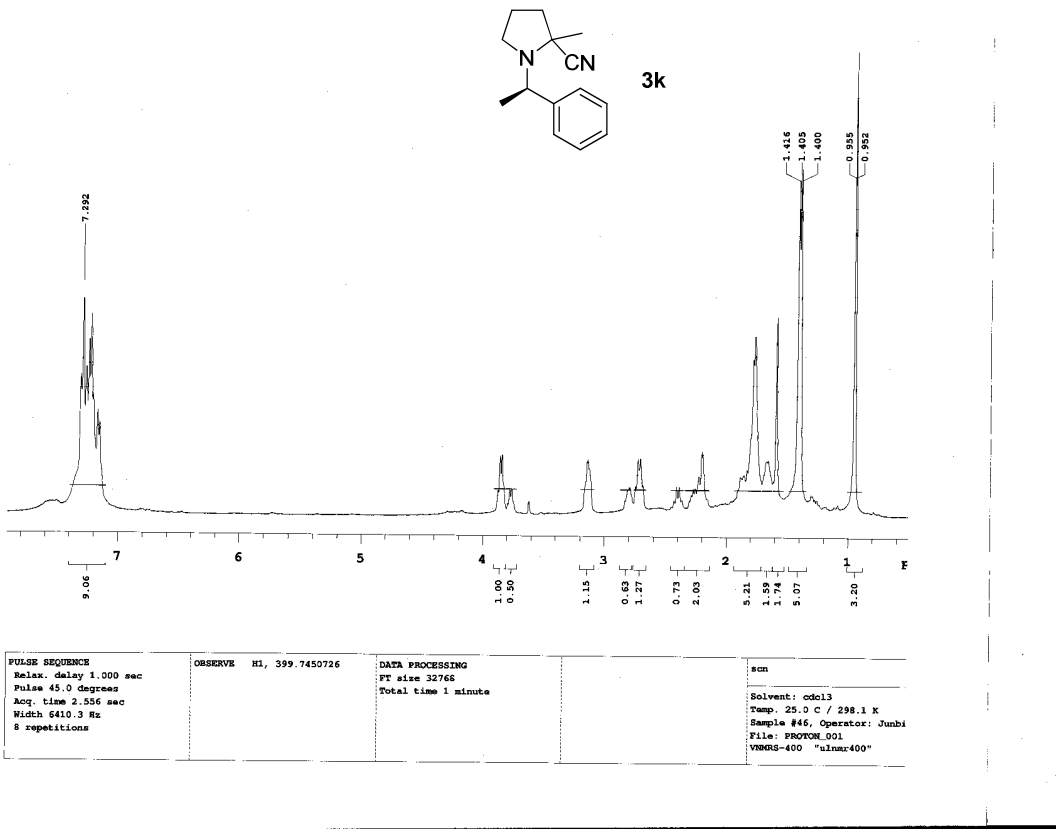

Sample ID: JunbinH_20100526_05
File: JunbinH_20100526_05/data/Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Carbon_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
320 repetitions
OBSERVE C13, 100.5222010 MHz
DECOUPLE H1, 399.7718181 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 min, 21 sec
128.505
128.379
127.159
119.896
138.655
77.356
77.036
76.717
65.830
54.550
51.663
36.803
36.238
31.624
29.392
24.533
22.546
20.158
14.041
ppm
C5H11
N
CN
Ph
3g

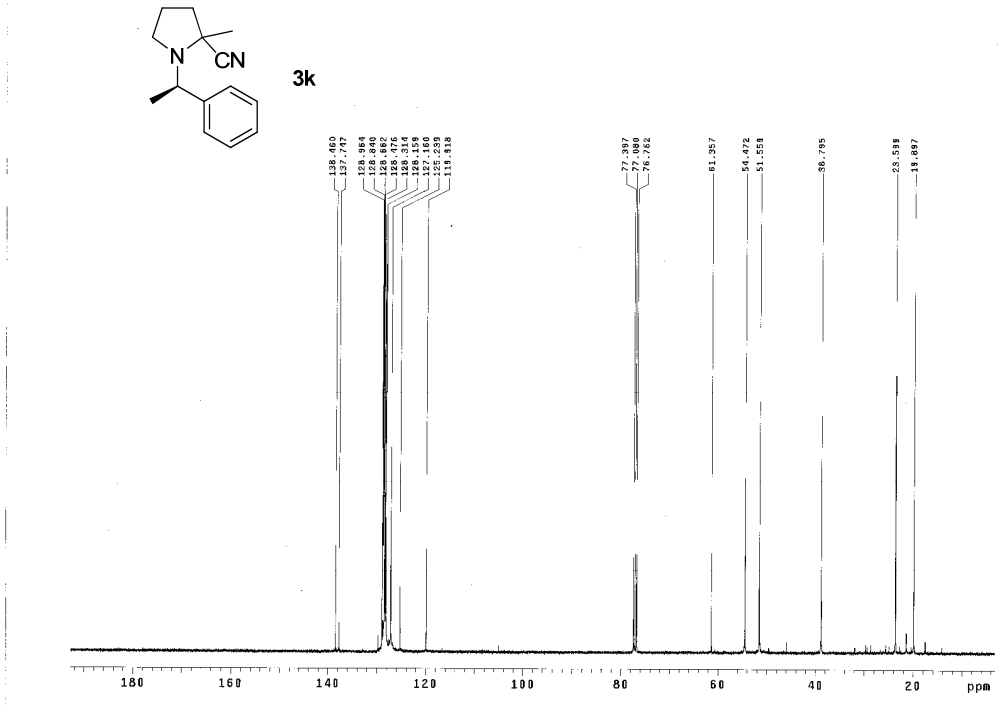

3h
3.821
3.792
3.619
3.150
3.142
3.122
3.115
1.742
1.734
1.304
8
7
6
5
4
3
2
1
ppm
1.01
1.00
0.36
1.93
1.04
0.90
1.00
4.13
4.43
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7698442
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 1 minute
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Proton_01
VNMRS-400 "ulnmr400"
Sample ID: JunbinH_20100804_01
File: JunbinH_20100804_01/data/Proton_01.fid
Pulse Sequence: s2pul
File: JunbinH_20100804_01/data/Proton_01.fid

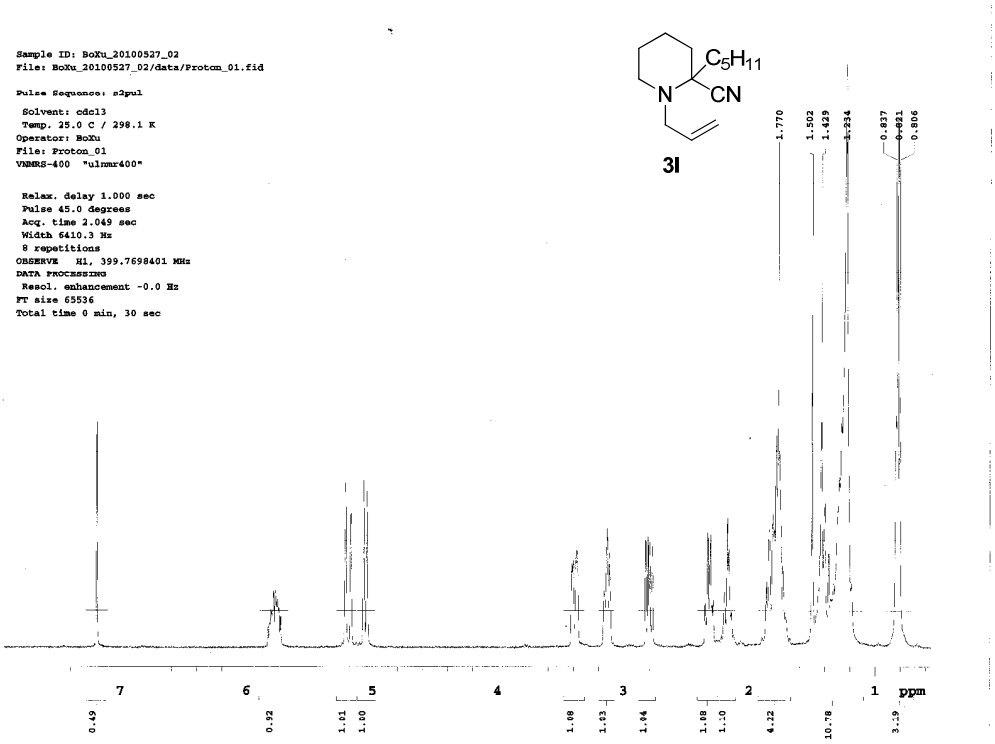

Sample ID: JunbinH_20100804_02
File: JunbinH_20100804_02/data/Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Carbon_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
192 repetitions
OBSERVE C13, 100.5222010 MHz
DECOUPLE H1, 399.7718181 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 41 sec
3h
77.319
76.999
76.679
73.212
71.954
67.058
58.012
57.779
47.347
35.694
25.976
23.957
118.081
220
200
180
160
140
120
100
80
60
40
20
0
ppm

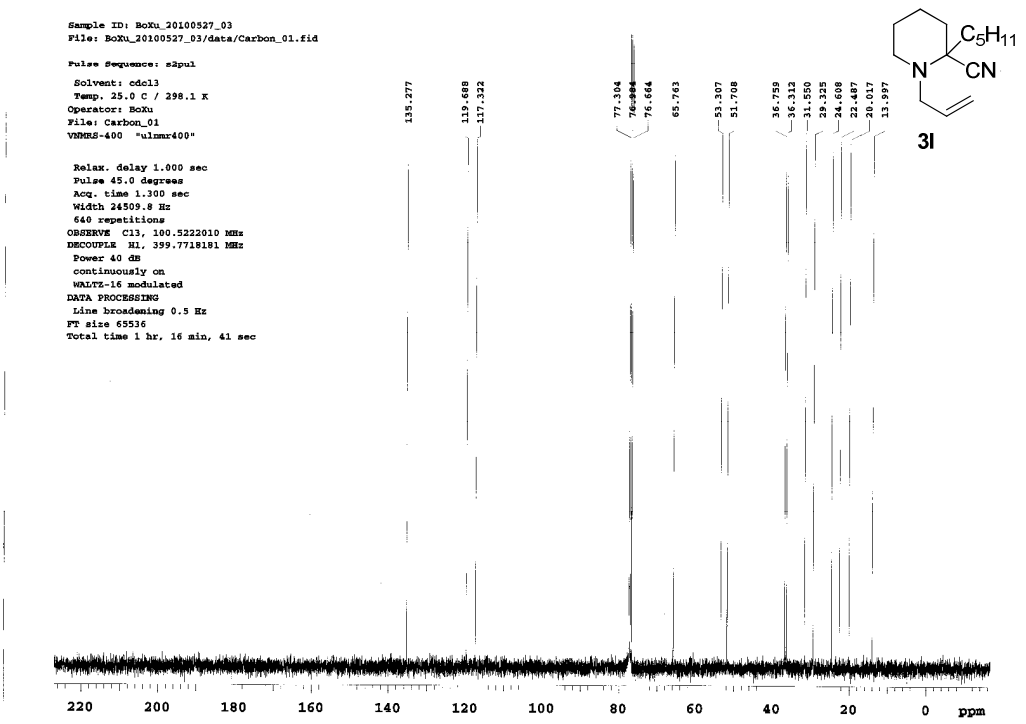

Sample ID: JunbinH_20100629_03
File: JunbinH_20100629_03/data/Proton_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Proton_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.048 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7698457 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec
C5H11
N
CN
Ph
3i
ppm

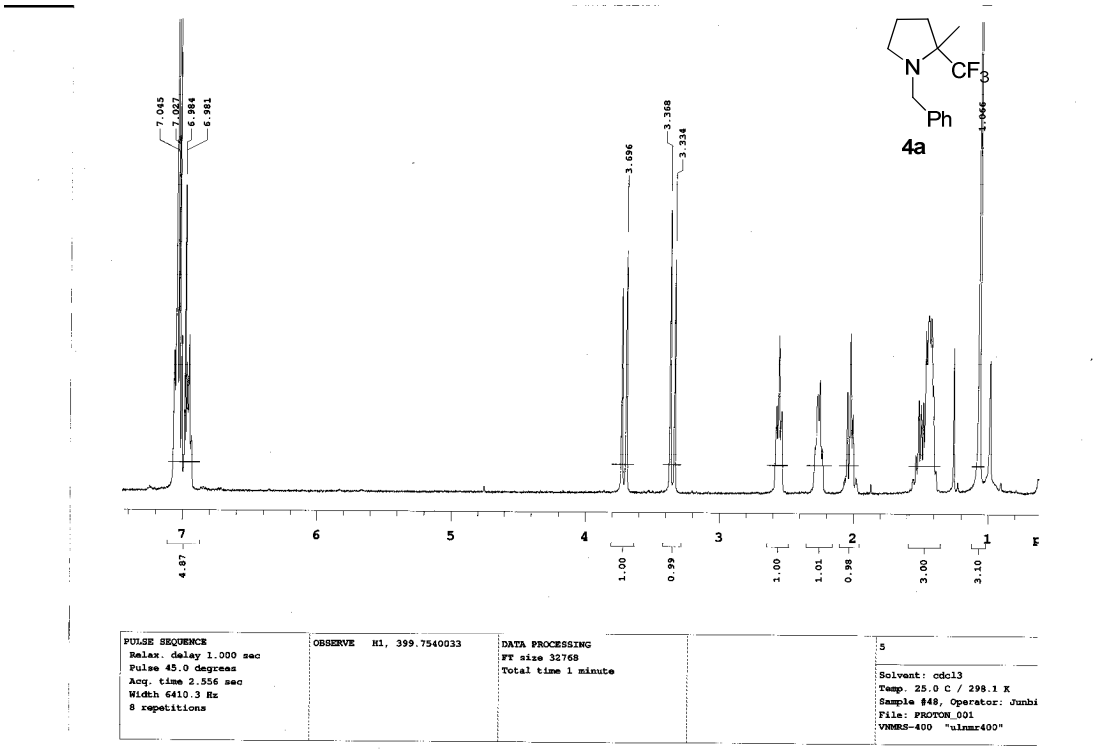

Sample ID: JunbinH_20100629_04
File: JunbinH_20100629_04/data/Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: JunbinH
File: Carbon_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
64 repetitions
OBSERVE C13, 100.5222010 MHz
DECOUPLE H1, 399.7718181 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 41 sec
C5H11
N
CN
Ph
3i
ppm

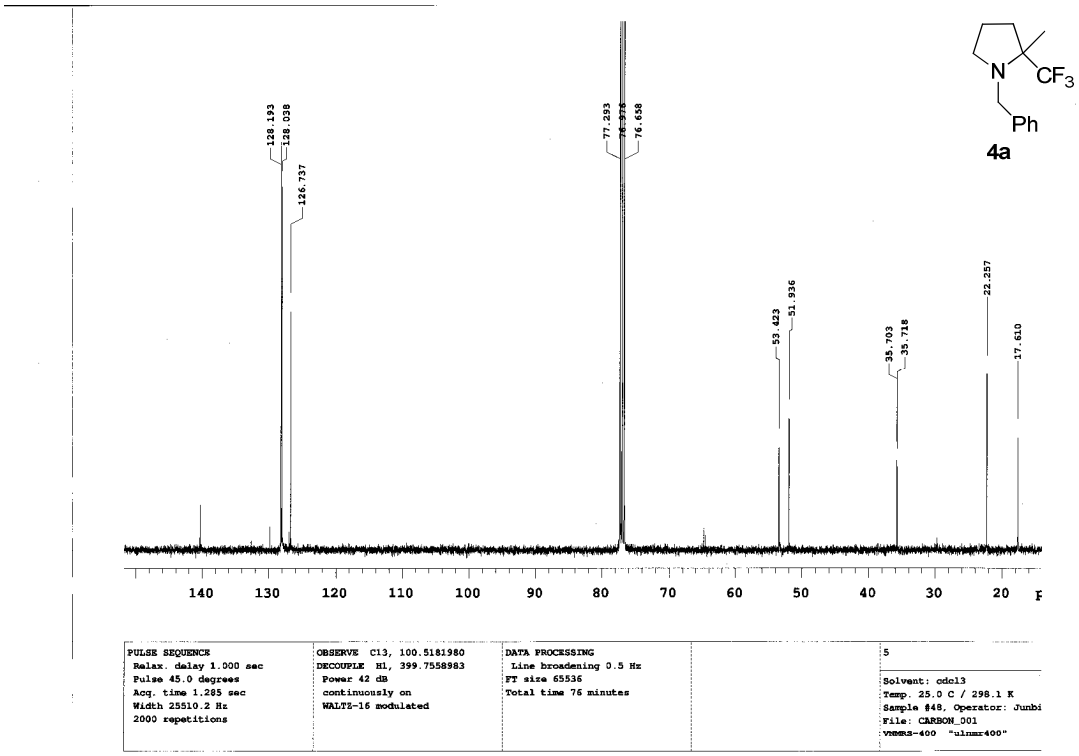

3k
7.292
1.416
1.405
1.400
0.955
0.952
9.06
1.00
0.50
1.15
0.63
1.27
0.73
2.03
5.21
1.59
1.74
5.07
3.20
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7450726
DATA PROCESSING
FT size 32768
Total time 1 minute
scn
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #46, Operator: Junbi
File: PROTON_001
VNMRS-400 "ulnar400"

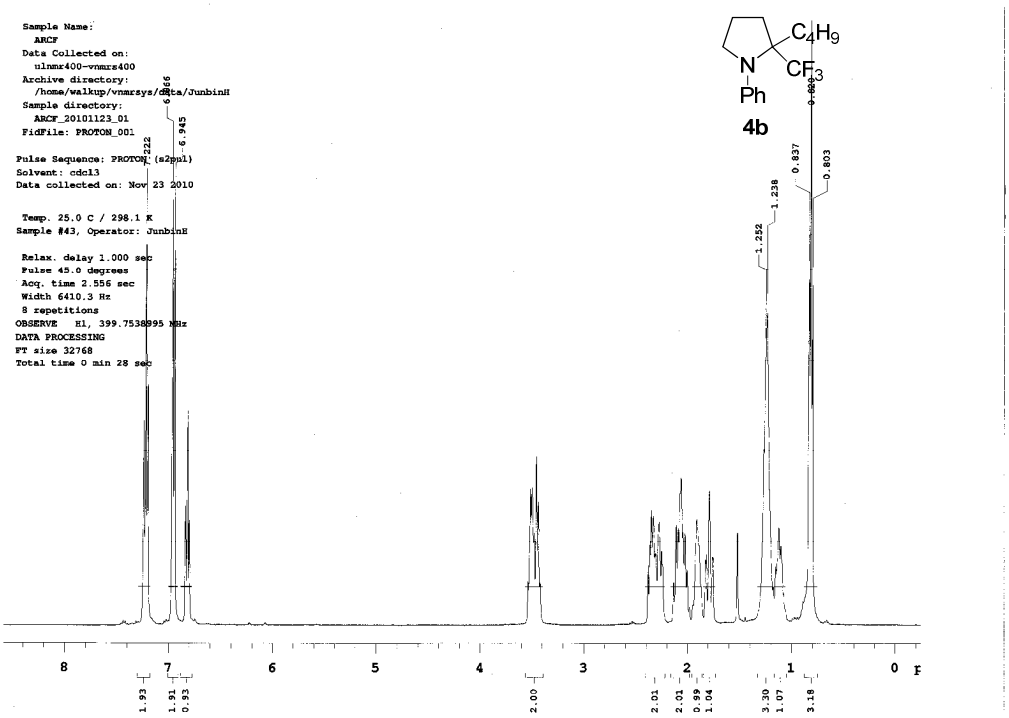

3k
138.460
137.747
128.984
128.680
128.582
128.476
128.314
128.158
127.160
125.239
118.918
77.387
77.080
76.762
61.357
54.472
51.558
36.795
23.598
19.897
180
160
140
120
100
80
60
40
20
ppm

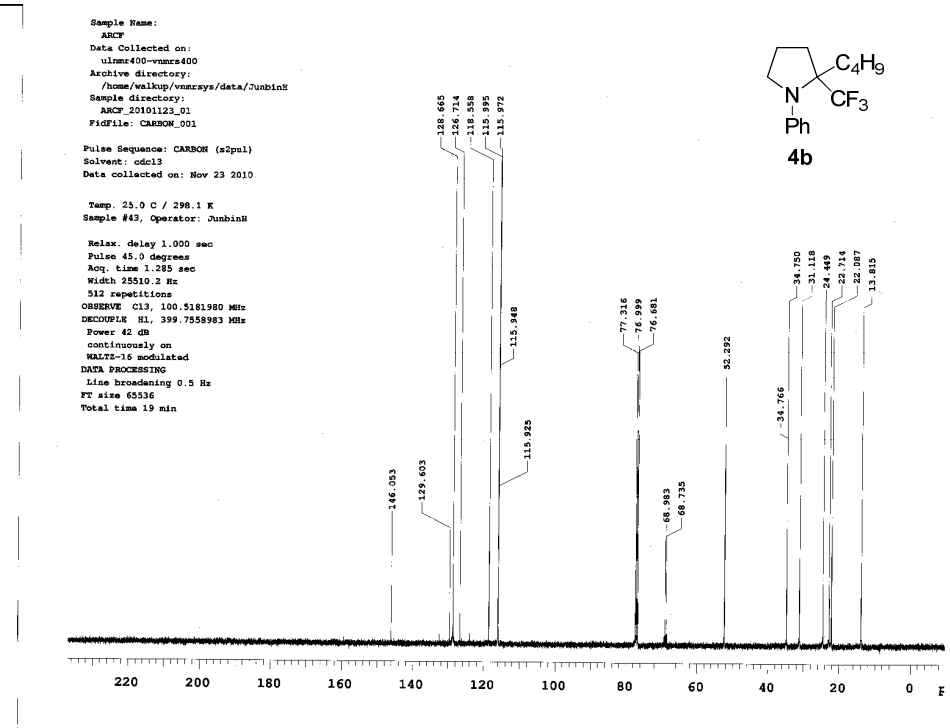

Sample ID: BoXu_20100527_02
File: BoXu_20100527_02/data/Protom_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: BoXu
File: Proton_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7698401 MHz
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 0 min, 30 sec
3l
1.770
1.502
1.429
1.334
0.837
0.821
0.806
7
6
5
4
3
2
1
ppm
0.49
0.92
1.01
1.00
1.08
1.03
1.04
1.08
1.00
4.22
10.78
3.19

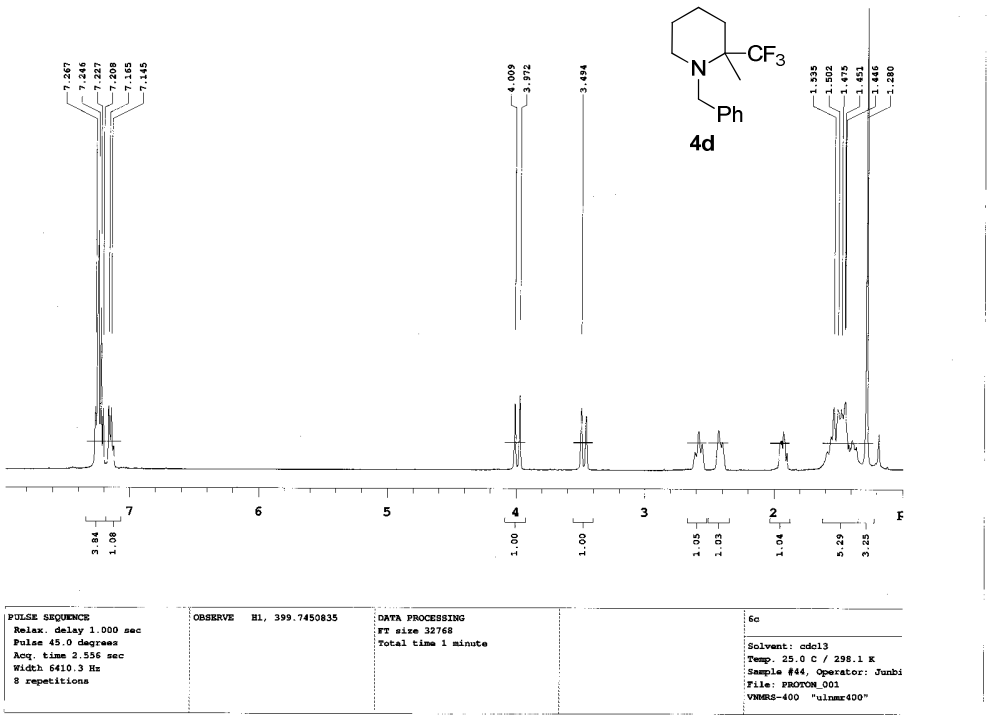

Sample ID: BoXu_20100527_03
File: BoXu_20100527_03/data/Carbon_01.fid
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: BoXu
File: Carbon_01
VNMRS-400 "ulnmr400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
640 repetitions
OBSERVE C13, 100.5222010 MHz
DECOUPLE H1, 399.7718181 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min, 41 sec
3l
135.277
119.688
117.322
77.304
76.664
65.763
53.307
51.708
36.759
36.312
31.550
29.325
24.608
22.487
20.017
13.997
220
200
180
160
140
120
100
80
60
40
20
0
ppm

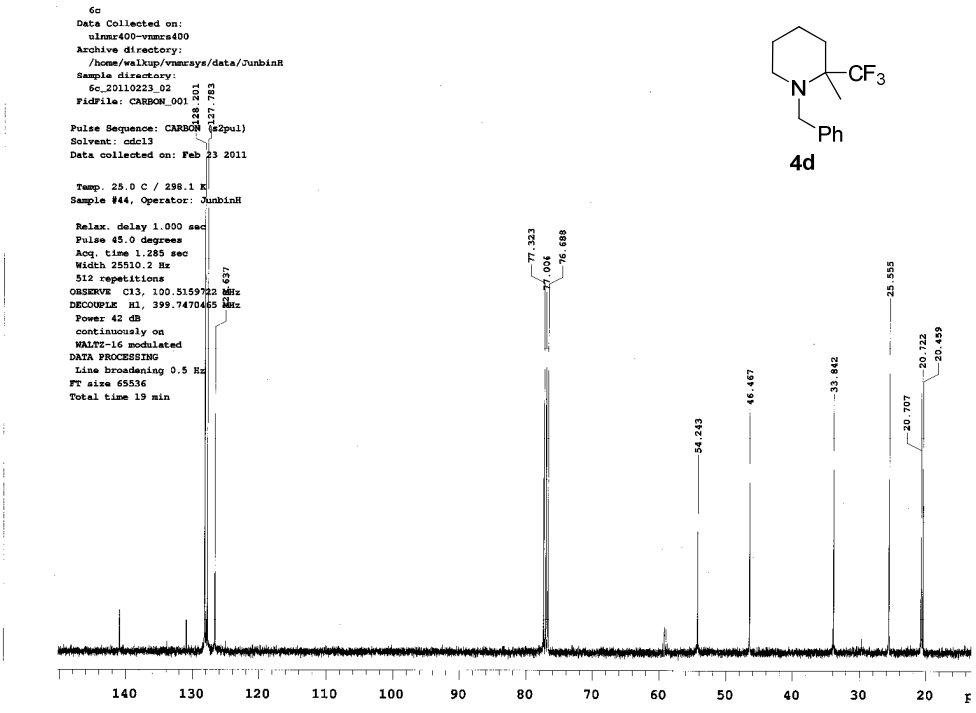

7.045
7.027
6.984
6.981
3.696
3.368
3.334
1.046
4.87
1.00
0.99
1.00
1.01
0.98
3.00
3.10
N
CF3
Ph
4a
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7540033
DATA PROCESSING
FT size 32768
Total time 1 minute
5
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #48, Operator: Junbi
File: PROTON_001
VNMRS-400 "ulnmr400"

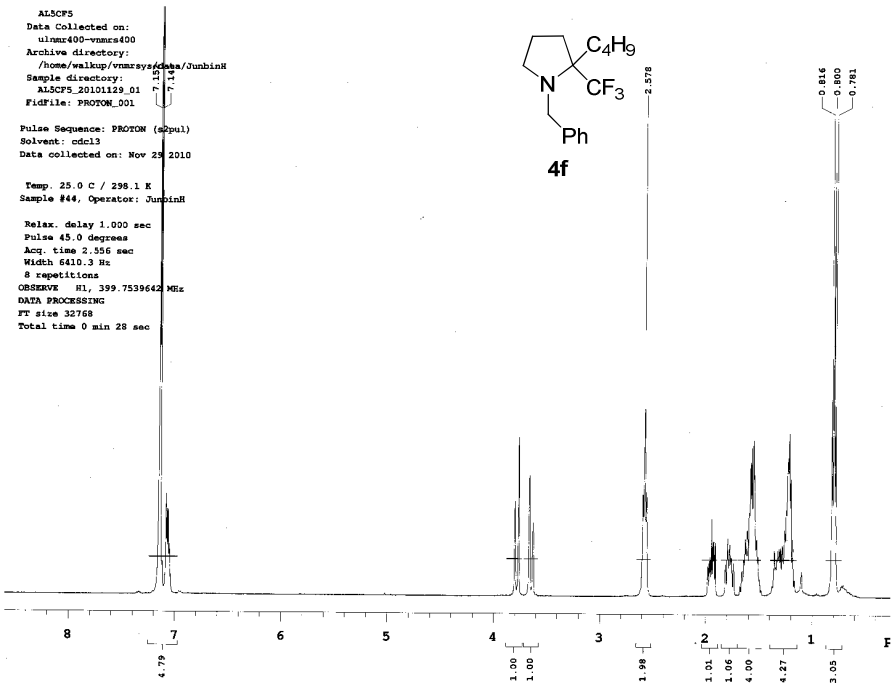

128.193
128.038
126.737
77.293
76.976
76.658
53.423
51.936
35.703
35.718
22.257
17.610
N
CF3
Ph
4a
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
2000 repetitions
OBSERVE C13, 100.5181980
DECOUPLE H1, 399.7558983
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 76 minutes
5
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #48, Operator: Junbi
File: CARBON_001
VNMRS-400 "ulnmr400"

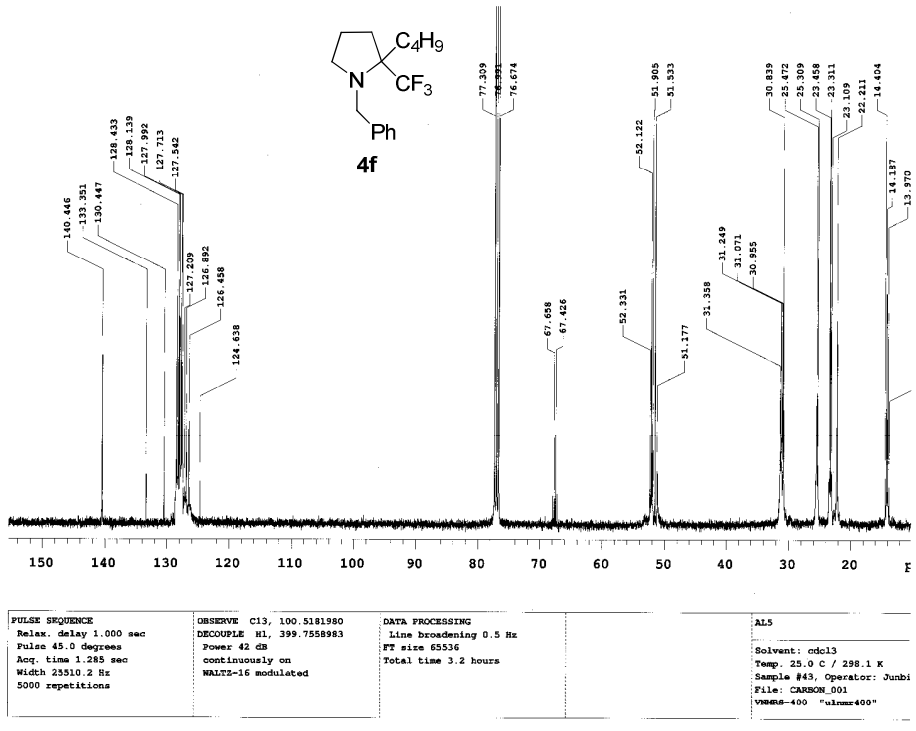

Sample Name:
ARCF
Data Collected on:
ulnmr400-vnmrs400
Archive directory:
/home/walkup/vnmrsys/data/JunbinH
Sample directory:
ARCF_20101123_01
FidFile: PROTON_001
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Nov 23 2010
Temp. 25.0 C / 298.1 K
Sample #43, Operator: JunbinH
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7538995 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec
4b
7.222
7.166
6.945
1.252
1.238
0.837
0.803
1.93
1.91
0.93
2.00
2.01
2.01
0.99
1.04
3.30
1.07
3.18


Sample Name:
ARCF
Data Collected on:
ulnmr400-vnmrs400
Archive directory:
/home/walkup/vnmrsys/data/JunbinH
Sample directory:
ARCF_20101123_01
FidFile: CARBON_001
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Nov 23 2010
Temp. 25.0 C / 298.1 K
Sample #43, Operator: JunbinH
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
512 repetitions
OBSERVE C13, 100.5181980 MHz
DECOUPLE H1, 399.7558983 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min
4b
128.665
126.714
118.558
115.995
115.972
115.948
115.925
146.053
129.603
77.316
76.999
76.681
68.983
68.735
52.292
34.766
34.750
31.118
24.449
22.714
22.087
13.815

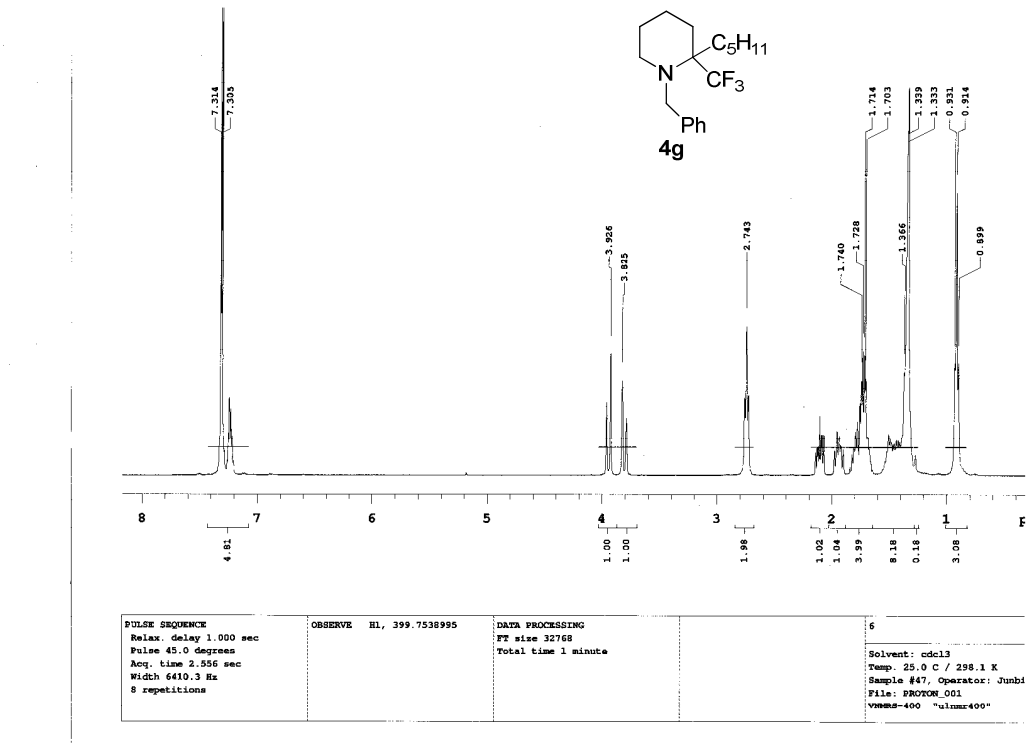

7.267
7.246
7.227
7.208
7.165
7.145
4.009
3.972
3.494
1.535
1.502
1.475
1.451
1.446
1.280
CF3
N
Ph
4d
7
6
5
4
3
2
3.84
1.08
1.00
1.00
1.05
1.03
1.04
5.29
3.25
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7450835
DATA PROCESSING
FT size 32768
Total time 1 minute
6c
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #44, Operator: Junbi
File: PROTON_001
VNMRS-400 "ulnmr400"

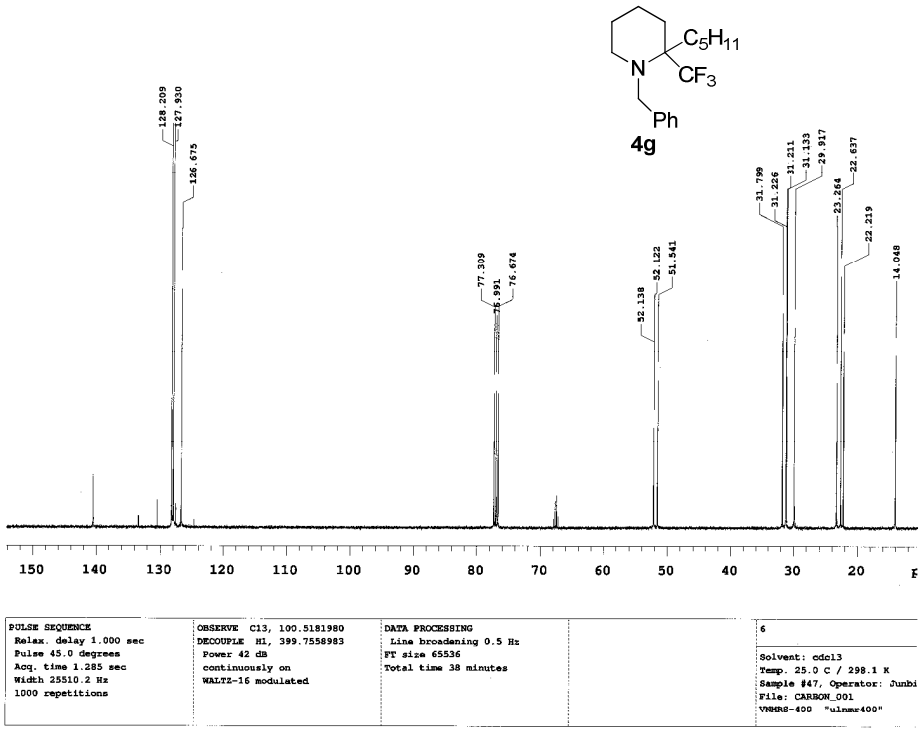

6c
Data Collected on:
ulnmr400-vnmrs400
Archive directory:
/home/walkup/vnmrsys/data/JunbinH
Sample directory:
6c_20110223_02
FidFile: CARBON_001
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Feb 23 2011
Temp. 25.0 C / 298.1 K
Sample #44, Operator: JunbinH
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
512 repetitions
OBSERVE C13, 100.5159723 MHz
DECOUPLE H1, 399.7470465 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 19 min
128.201
127.783
77.323
77.006
76.688
54.243
46.467
33.842
25.555
20.722
20.459
20.707
CF3
N
Ph
4d
140
130
120
110
100
90
80
70
60
50
40
30
20

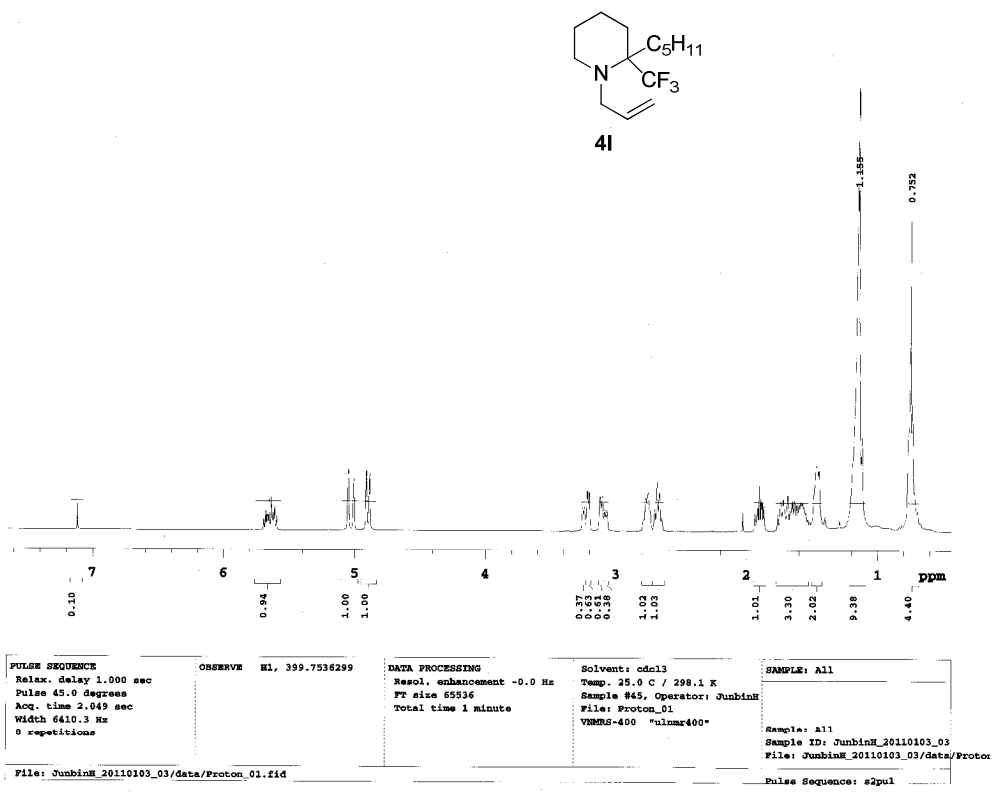


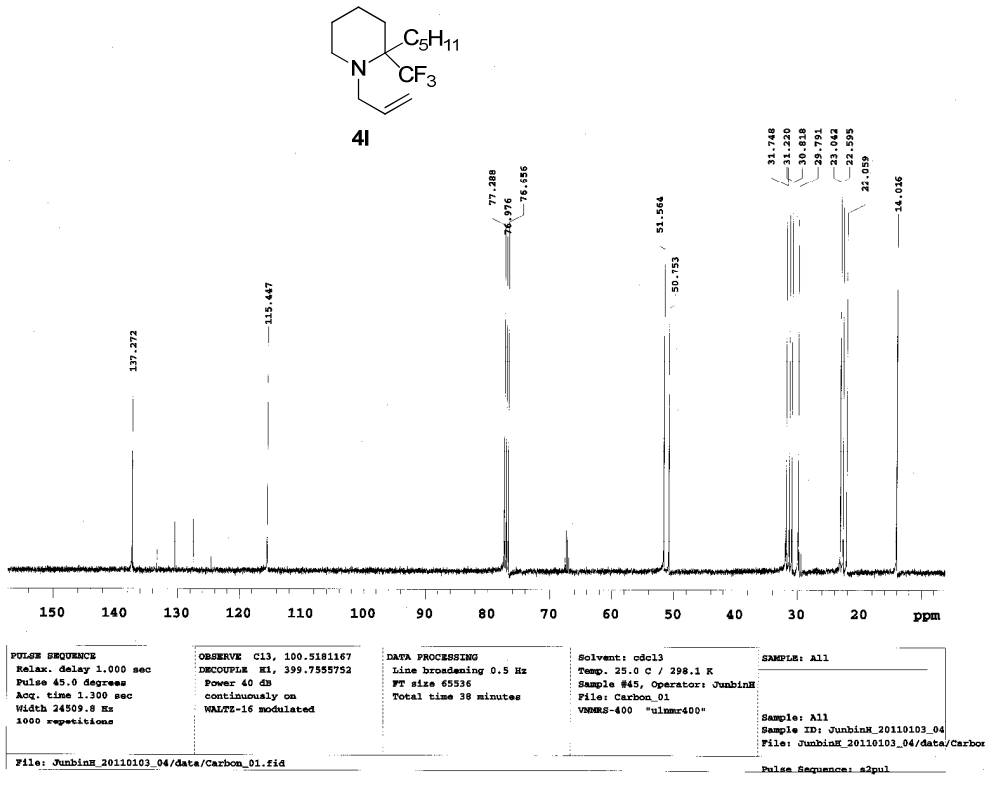


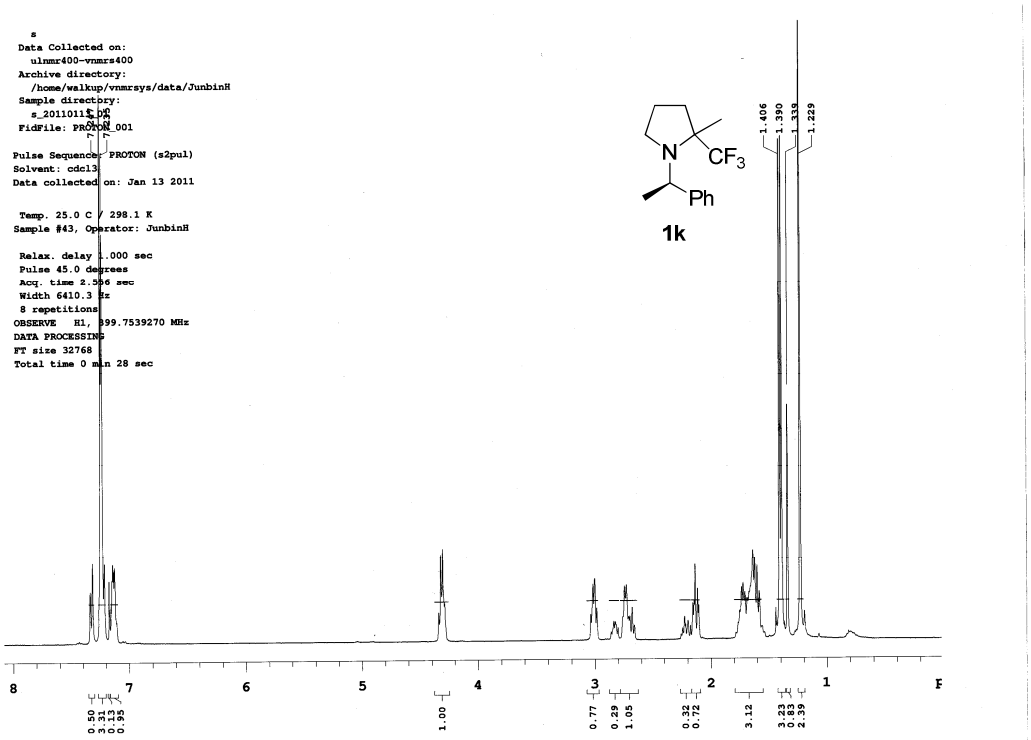
PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 1.285 sec Width 25510.2 Hz 5000 repetitions	OBSERVE C13, 100.5181980 DECOUPLE H1, 399.7558983 Power 42 dB continuously on WALTZ-16 modulated	DATA PROCESSING Line broadening 0.5 Hz FT size 65536 Total time 3.2 hours		AL5 Solvent: cdcl3 Temp. 25.0 C / 298.1 K Sample #43, Operator: Junbi File: CARBON_001 VNMRS-400 "ulnmr400"

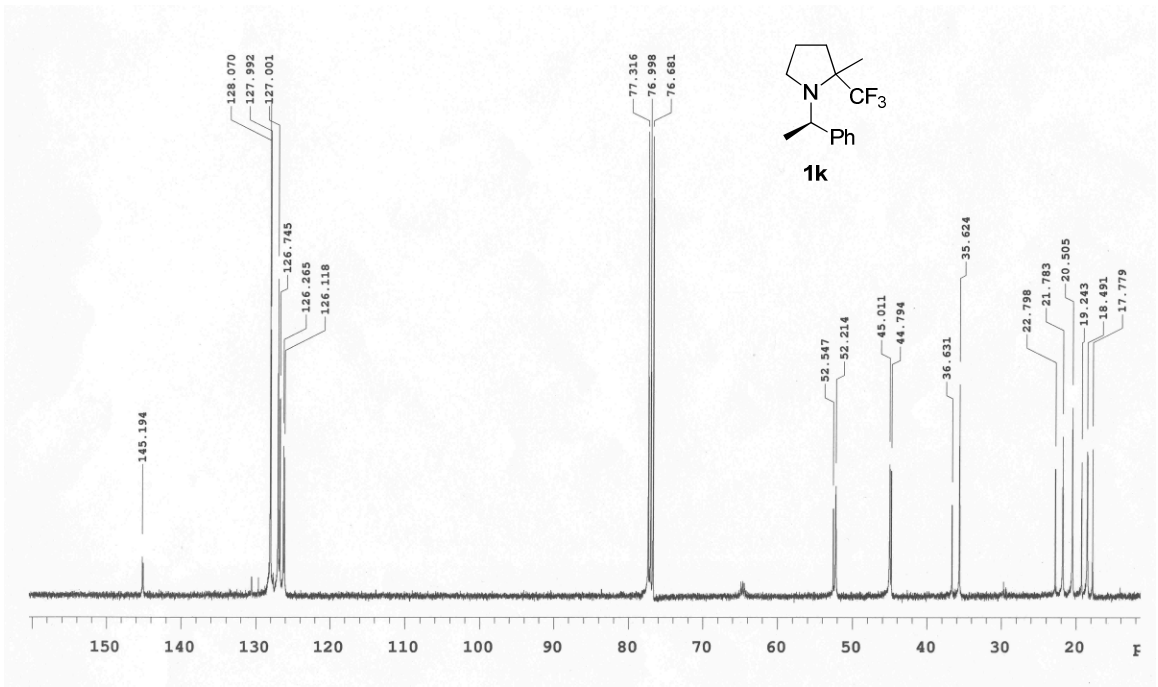

C_5H_{11}
N
CF_3
Ph
4g
7.314
7.305
3.926
3.825
2.743
1.740
1.728
1.714
1.703
1.366
1.339
1.333
0.931
0.914
0.899
8
7
6
5
4
3
2
1
4.81
1.00
1.00
1.98
1.02
1.04
3.99
8.18
0.18
3.08
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7538995
DATA PROCESSING
FT size 32768
Total time 1 minute
6
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #47, Operator: Junbi
File: PROTON_001
VNMRS-400 "ulnmr400"

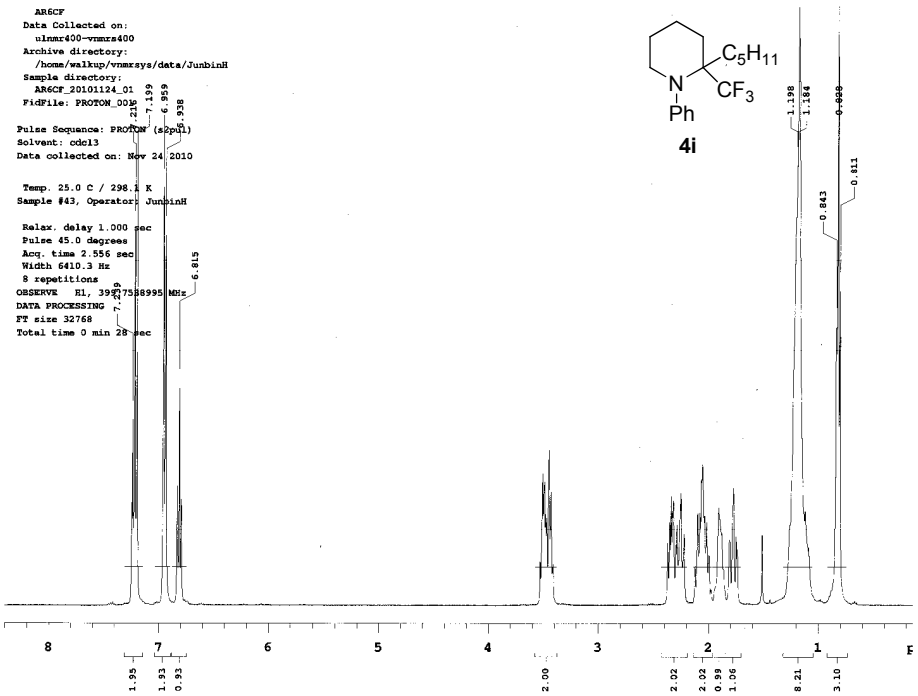

C_5H_{11}
N
CF_3
Ph
4g
128.209
127.930
126.675
77.309
76.991
76.674
52.138
52.122
51.541
31.799
31.226
31.211
31.133
29.917
23.264
22.637
22.219
14.048
150
140
130
120
110
100
90
80
70
60
50
40
30
20
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
1000 repetitions
OBSERVE C13, 100.5181980
DECOUPLE H1, 399.7558983
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 minutes
6
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #47, Operator: Junbi
File: CARBON_001
VNMRS-400 "ulnmr400"

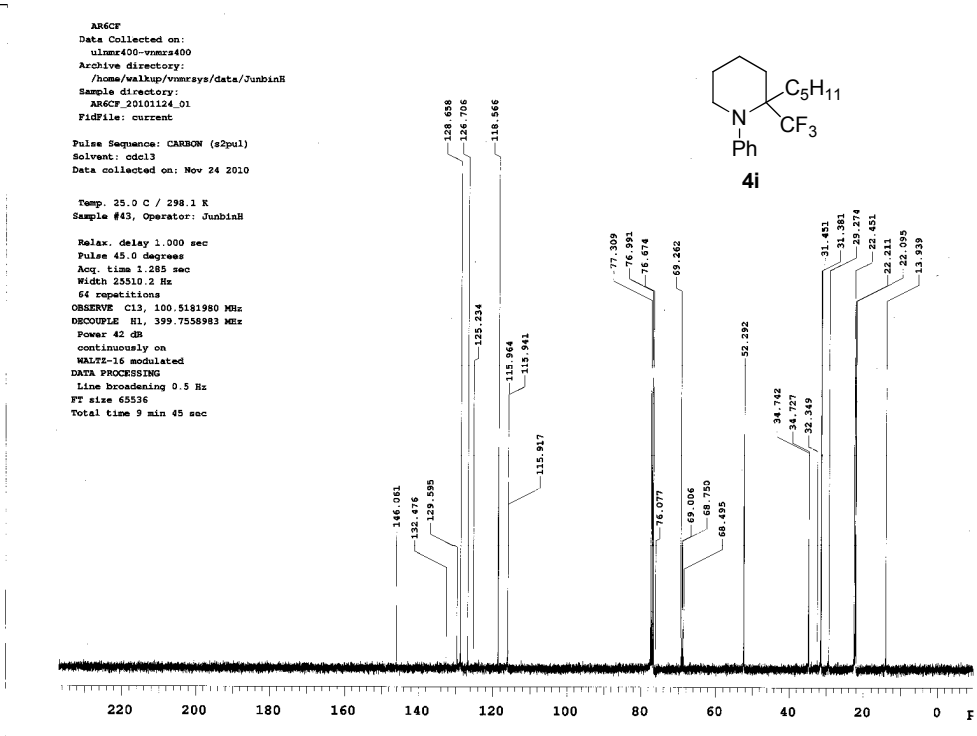

4l
1.455
0.752
7
6
5
4
3
2
1
ppm
0.10
0.94
1.00
1.00
0.37
0.63
0.61
0.38
1.02
1.03
1.01
3.30
2.02
9.38
4.40
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7536299
DATA PROCESSING
Resol. enhancement -0.0 Hz
FT size 65536
Total time 1 minute
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #45, Operator: JunbinH
File: Proton_01
VNMRS-400 "ulnmr400"
SAMPLE: All
Sample: All
Sample ID: JunbinH_20110103_03
File: JunbinH_20110103_03/data/Proton
Pulse Sequence: s2pul
File: JunbinH_20110103_03/data/Proton_01.fid

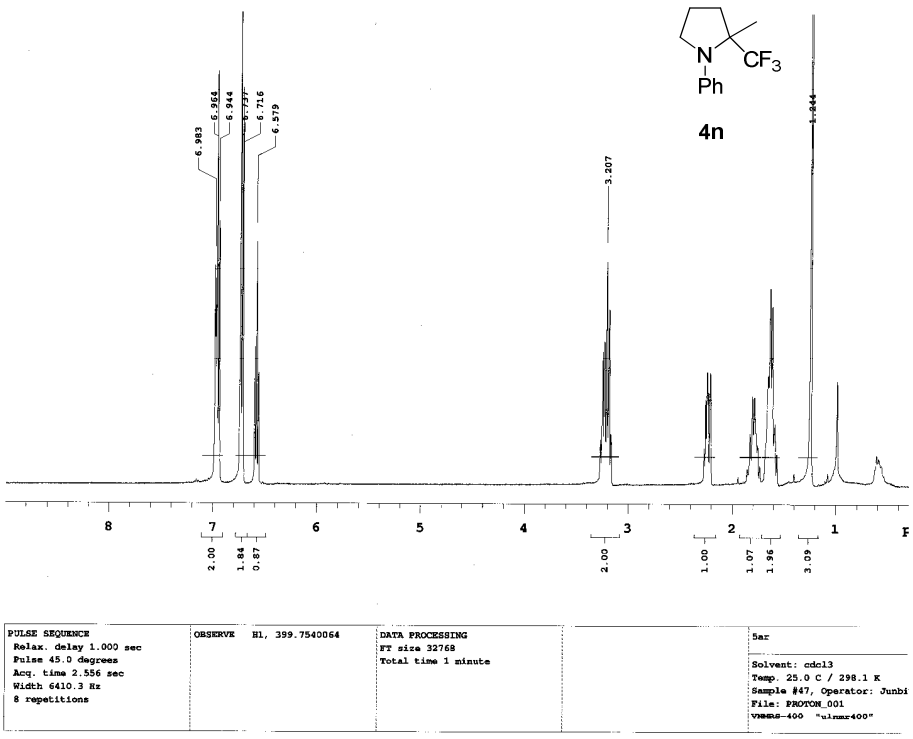

4l
137.272
115.447
77.388
76.976
76.556
51.564
50.753
31.748
31.220
30.818
29.791
23.042
22.595
22.059
14.016
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
1000 repetitions
OBSERVE C13, 100.5181167
DECOUPLE H1, 399.7555752
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 minutes
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #45, Operator: JunbinH
File: Carbon_01
VNMRS-400 "ulnmr400"
SAMPLE: All
Sample: All
Sample ID: JunbinH_20110103_04
File: JunbinH_20110103_04/data/Carbon
Pulse Sequence: s2pul
File: JunbinH_20110103_04/data/Carbon_01.fid

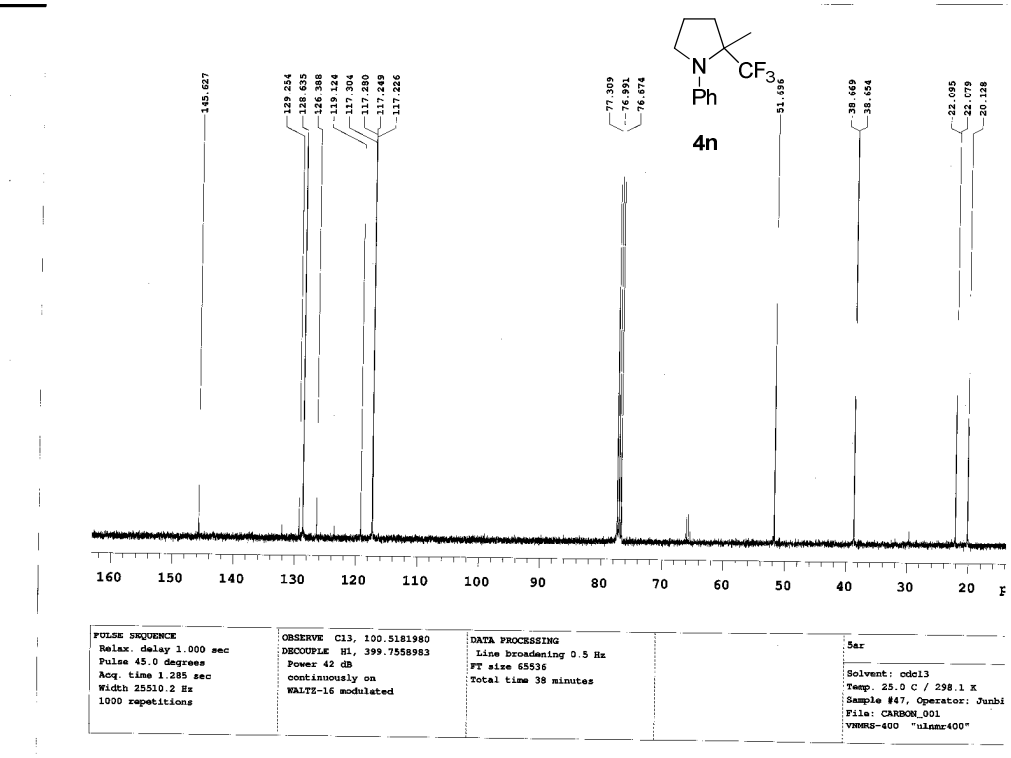

Data Collected on:
ulnmr400-vnmrs400
Archive directory:
/home/walkup/vnmrsys/data/JunbinH
Sample directory:
FidFile: PROTON_001
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Jan 13 2011
Temp. 25.0 C / 298.1 K
Sample #43, Operator: JunbinH
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7539270 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec
N
CF3
Ph
1k
1.406
1.390
1.339
1.229
8
7
6
5
4
3
2
1
0.50
3.15
3.13
0.95
1.00
0.77
0.29
1.05
0.32
0.72
3.12
3.23
0.83
2.39


128.070
127.992
127.001
126.745
126.265
126.118
145.194
77.316
76.998
76.681
52.547
52.214
45.011
44.794
36.631
35.624
22.798
21.783
20.505
19.243
18.491
17.779
N
CF3
Ph
1k
150
140
130
120
110
100
90
80
70
60
50
40
30
20


AR6CF
Data Collected on:
ulnmr400-vnmrs400
Archive directory:
/home/walkup/vnmrsys/data/JunbinH
Sample directory:
AR6CF_20101124_01
FidFile: PROTON_001
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Nov 24 2010
Temp. 25.0 C / 298.1 K
Sample #43, Operator: JunbinH
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7558995 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 28 sec
7.219
7.199
6.959
6.938
6.815
1.198
1.184
0.888
0.843
0.811
1.95
1.93
0.93
2.00
2.02
2.02
0.99
1.06
8.21
3.10
4i


AR6CF
Data Collected on:
ulnmr400-vnmrs400
Archive directory:
/home/walkup/vnmrsys/data/JunbinH
Sample directory:
AR6CF_20101124_01
FidFile: current
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Nov 24 2010
Temp. 25.0 C / 298.1 K
Sample #43, Operator: JunbinH
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
64 repetitions
OBSERVE C13, 100.5181980 MHz
DECOUPLE H1, 399.7558983 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 9 min 45 sec
128.658
126.706
118.566
125.234
115.964
115.941
115.917
146.061
132.476
129.595
77.309
76.991
76.674
76.077
69.262
69.006
68.750
68.495
52.292
34.742
34.727
32.349
31.451
31.381
29.274
22.451
22.211
22.095
13.939
4i


N
CF3
Ph
4n
6.983
6.964
6.944
6.737
6.716
6.579
3.207
1.844
8
7
6
5
4
3
2
1
2.00
1.84
0.87
2.00
1.00
1.07
1.96
3.09
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.556 sec
Width 6410.3 Hz
8 repetitions
OBSERVE H1, 399.7540064
DATA PROCESSING
FT size 32768
Total time 1 minute
5ar
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #47, Operator: Junbi
File: PROTON_001
VNMRS-400 "ulnmr400"


N
CF3
Ph
4n
145.627
129.254
128.635
126.388
119.124
117.304
117.280
117.249
117.226
77.309
76.991
76.674
51.696
38.669
38.454
22.095
22.079
20.128
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
1000 repetitions
OBSERVE C13, 100.5181980
DECOUPLE H1, 399.7558983
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 38 minutes
5ar
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Sample #47, Operator: Junbi
File: CARBON_001
VNMRS-400 "ulnmr400"