

Secondary Amine Formation from Reductive Amination of Carbonyl Compounds Promoted by Lewis Acid Using $\text{InCl}_3/\text{Et}_3\text{SiH}$ System

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General Experimental

All reagents and solvents for reactions were used as received. AR grade methanol was used directly as reaction solvent. Reactions were monitored by thin layer chromatography (TLC) using silica gel 60 precoated glass plates with 0.25 mm thickness. Flash column chromatography was performed on E. Merck silica gel 60 (230–400 mesh ASTM) using ethyl acetate/*n*-hexane as eluting solvents.

^1H -NMR (300 or 400 MHz) and ^{13}C -NMR (75 or 100 MHz) spectra were recorded in CDCl_3 , with tetramethylsilane (TMS) as internal standard at ambient temperature. Mass spectra were recorded with a Finnigan MAT 95 mass spectrometer for both low resolution and high resolution mass spectra. Infrared absorption spectra were recorded as a solution in CH_2Cl_2 with a Bio-Rad FTS 165 Fourier Transform Spectrophotometer.

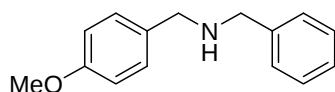
Typical Procedure for Lewis Acid Promoted Reductive Amination Catalyzed by $\text{InCl}_3/\text{Et}_3\text{SiH}$ System

To a stirred solution of carbonyl substrate (0.5 mmol) in methanol (1.0 mL) was added the corresponding amine (0.5 mmol) at room temperature. The mixture was stirred for 1 h. $\text{Zn}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ (93 mg, 0.25 mmol) and Et_3SiH (0.15 mL, 1.0 mmol) were added, followed by InCl_3 (33 mg, 0.15 mmol). The reaction was allowed to proceed with stirring at room temperature and is monitored by TLC. Upon completion, the mixture was quenched by saturated K_2CO_3

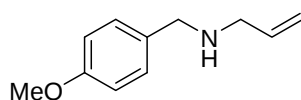
solution (1 mL). The mixture was then extracted with ethyl acetate (5 mL $\times$ 3). The combined organic layer was washed with brine (5 mL) and dried over anhydrous Na₂SO₄. The crude product was purified by flash column chromatography to afford the desired product.

Products **3k**, **3u**, **3v**, **3w** and **3y** were isolated as their HCl salt. The crude product was subjected to HCl in ether solution (5 mL). The corresponding HCl salt was then washed with hexane (5 mL $\times$ 3) and finally dried in vacuo.

Characterization of Compounds 3a–z

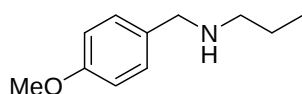


Compound **3a**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, R_f = 0.62; ¹H NMR (300 MHz, CDCl₃) δ 7.33–7.31 (m, 4H), 7.29–7.22 (m, 3H), 6.88–6.84 (m, 2H), 3.78 (s, 5H), 3.73 (s, 2H), 1.61 (s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 158.9, 140.6, 132.7, 129.6, 128.7, 128.5, 127.2, 114.1, 55.5, 53.3, 52.8; IR (CH₂Cl₂) 3295, 3037, 2941, 2837, 1606, 1513, 1455, 1301 cm⁻¹; LRMS (EI) 227 (M⁺, 50), 226 (52), 136 (52), 121 (100); HRMS (EI) calcd for C₁₅H₁₇NO (M⁺) 227.1310, found 227.1301.

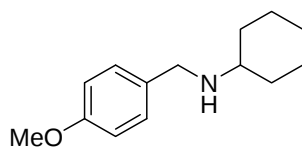


Compound **3b**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, R_f = 0.21; ¹H NMR (400

MHz, CDCl₃) δ 7.24 (d, J = 8.6 Hz, 2H), 6.86 (d, J = 8.6 Hz, 2H), 5.96–5.89 (m, 1H), 5.18 (dd, J = 17.1, 1.3 Hz, 1H), 5.10 (dd, J = 10.2, 1.3 Hz, 1H), 3.79 (s, 3H), 3.72 (s, 2H), 3.26 (d, J = 4.8 Hz, 2H), 1.59 (br s, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 158.9, 137.1, 132.7, 129.6, 116.2, 114.1, 55.5, 52.9, 52.0; IR (CH₂Cl₂) 3310, 3014, 2918, 2844, 1617, 1513 cm⁻¹; LRMS (EI) 177 (M⁺, 17), 176 (35), 121 (100); HRMS (EI) calcd for C₁₁H₁₅NO (M⁺) 177.1154, found 177.1139.

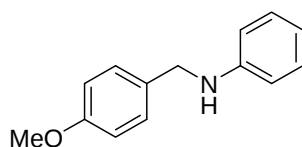


Compound **3c**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, R_f = 0.62; ¹H NMR (400 MHz, CDCl₃) δ 7.23 (d, J = 8.6 Hz, 2H), 6.86 (d, J = 8.6 Hz, 2H), 3.79 (s, 3H), 3.71 (s, 2H), 2.58 (t, J = 7.3 Hz, 2H), 1.51 (sextet, J = 7.3 Hz, 2H), 1.40 (br s, 1H), 0.91 (t, J = 7.3 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 158.5, 132.7, 129.2, 113.7, 55.2, 53.4, 51.2, 23.2, 11.8; IR (CH₂Cl₂) 3411, 2961, 2826, 2244, 1617, 1505, 1449 cm⁻¹; LRMS (EI) 179 (M⁺, 11), 150 (23), 121 (100); HRMS (EI) calcd for C₁₁H₁₇NO (M⁺) 179.1310, found 179.1307.

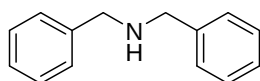


Compound **3d**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, R_f = 0.21; ¹H NMR (300 MHz, CDCl₃) δ 7.24 (d, J = 8.5 Hz, 2H), 6.85 (d, J = 8.6 Hz, 2H), 3.78 (s, 3H), 3.73 (s, 2H), 2.50–2.43 (m, 1H), 1.92–1.87 (br d, 2H), 1.74–1.70 (m, 2H), 1.62–1.60 (m, 1H), 1.22–1.04 (m,

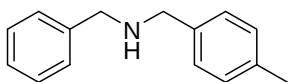
6H); ^{13}C NMR (75 MHz, CDCl_3) δ 158.4, 133.0, 129.1, 113.7, 56.0, 55.1, 50.3, 33.5, 26.1, 25.0; IR (CH_2Cl_2) 3433, 2932, 2855, 1613, 1512, 1454 cm^{-1} ; LRMS (EI) 219 (M^+ , 28), 176 (17), 121 (100); HRMS (EI) calcd for $\text{C}_{14}\text{H}_{21}\text{NO}$ (M^+) 219.1623, found 219.1622.



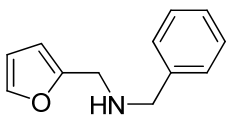
Compound **3e**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, R_f = 0.62; ^1H NMR (300 MHz, CDCl_3) δ 7.26 (d, J = 8.6 Hz, 2H), 1.15 (t, J = 7.9 Hz, 2H), 6.86 (d, J = 8.6 Hz, 2H), 6.69 (t, J = 7.3 Hz, 1H), 6.61 (d, J = 8.6 Hz, 2H), 4.22 (s, 2H), 3.91 (br s, 1H), 3.77 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 158.8, 148.2, 131.4, 129.2, 128.7, 117.4, 114.0, 112.8, 55.2, 47.7; IR (CH_2Cl_2) 3420, 3035, 2929, 2832, 2244, 1599, 1499, 1455 cm^{-1} ; LRMS (EI) 213 (M^+ , 34), 166 (29), 135 (60), 121 (100); HRMS (EI) calcd for $\text{C}_{14}\text{H}_{15}\text{NO}$ (M^+) 213.1154, found 213.1152.



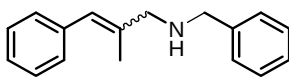
Compound **3f**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, R_f = 0.62; ^1H NMR (300 MHz, CDCl_3) δ 7.33–7.28 (m, 8H), 7.26–7.21 (m, 2H), 3.79 (s, 4H); ^{13}C NMR (75 MHz, CDCl_3) δ 140.2, 128.4, 128.1, 127.0, 53.1; IR (CH_2Cl_2) 3426, 3029, 2923, 2826, 1650, 1493, 1455 cm^{-1} ; LRMS (EI) 197 (M^+ , 37), 106 (86), 91 (100); HRMS (EI) calcd for $\text{C}_{14}\text{H}_{15}\text{N}$ (M^+) 197.1204, found 197.1194.



Compound **3g**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, $R_f = 0.62$; ^1H NMR (400 MHz, CDCl_3) δ 7.33–7.28 (m, 4H), 7.25–7.20 (m, 3H), 7.12 (d, $J = 7.9$ Hz, 2H), 3.77 (s, 2H), 3.75 (s, 2H), 2.32 (s, 3H), 1.88 (br s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 140.3, 137.1, 136.4, 129.0, 128.3, 128.1, 128.1, 126.9, 53.0, 52.8, 21.0; IR (CH_2Cl_2) 3320, 3034, 2961, 2867, 1505, 1455 cm^{-1} ; LRMS (EI) 211 (M^+ , 55), 210 (49), 120 (78), 106 (100); HRMS (EI) calcd for $\text{C}_{15}\text{H}_{17}\text{N}$ (M^+) 211.1361, found 211.1353.



Compound **3h**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, $R_f = 0.12$; ^1H NMR (400 MHz, CDCl_3) δ 7.36 (s, 1H), 7.35–7.26 (m, 4H), 7.27–7.23 (m, 1H), 6.31 (dd, $J = 3.0, 1.9$ Hz, 1H), 6.17 (d, $J = 3.1$ Hz, 1H), 3.78 (s, 4H), 1.79 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 153.8, 141.8, 139.8, 128.4, 128.2, 127.0, 110.1, 107.0, 52.7, 45.3; IR (CH_2Cl_2) 3328, 3041, 2832, 1599, 1493, 1449 cm^{-1} ; LRMS (EI) 187 (M^+ , 38), 106 (40), 96 (73), 91 (100); HRMS (EI) calcd for $\text{C}_{12}\text{H}_{13}\text{N}$ (M^+) 187.0997, found 187.0988.

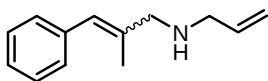


Compound **3i**: Isolated as mixture of *Z/E* isomers (ratio = 1:6.7); Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, $R_f = 0.52$;

For *Z*-isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.33–7.22 (m, 10H), 6.43 (s, 1H), 3.70 (s, 2H), 3.39 (s, 2H), 1.97 (s, 3H), 1.74 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 140.1, 137.6, 137.0, 128.6, 128.3, 128.1, 128.0, 126.9, 126.2, 126.0, 53.3, 49.7, 23.0.

For *E*-isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.33–7.22 (m, 10H), 6.46 (s, H), 3.81 (s, 2H), 3.33 (s, 2H), 1.91 (s, 3H), 1.74 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 140.2, 137.9, 136.7, 128.8, 128.4, 128.1, 128.0, 126.9, 126.1, 126.0, 57.4, , 52.9, 16.6.

IR (CH_2Cl_2) 3345, 3029, 2917, 1487, 1449 cm^{-1} ; LRMS (EI) 237 (M^+ , 18), 222 (29), 146 (41), 91 (100); HRMS (EI) calcd for $\text{C}_{17}\text{H}_{19}\text{N}$ (M^+) 237.1518, found 237.1509.

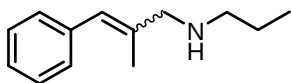


Compound **3j**: As mixture of *Z/E* isomers (ratio = 1:8.3); Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, R_f = 0.15;

For *Z*-isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.33–7.30 (m, 2H), 7.27–7.25 (m, 2H), 7.21–7.18 (m, 1H), 6.44 (s, 1H), 5.86–5.81 (m, 1H), 5.11 (d, J = 10.2 Hz, 1H), 5.04 (d, J = 10.2 Hz, 1H), 3.37 (s, 2H), 3.17 (d, J = 5.8 Hz, 2H), 1.96 (s, 3H), 1.42 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 137.2, 136.8, 128.6, 128.0, 126.2, 125.8, 115.9, 52.0, 49.9, 22.9.

For *E*-isomer: ^1H NMR (400 MHz, CDCl_3) δ 7.33–7.30 (m, 2H), 7.27–7.25 (m, 2H), 7.21–7.18 (m, 1H), 6.44 (s, 1H), 5.99–5.90 (m, 1H), 5.21 (d, J = 17.1 Hz, 1H), 5.11 (d, J = 10.2 Hz, 1H), 3.32 (s, 2H), 3.28 (d, J = 6.0 Hz, 2H), 1.90 (s, 3H), 1.42 (br s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 138.0, 136.9, 128.8, 128.0, 126.1, 125.8, 115.9, 57.5, 51.6, 16.6.

IR (CH₂Cl₂) 3355, 3033, 2977, 2923, 2819, 1638, 1590 cm⁻¹; LRMS (EI) 187 (M⁺, 31), 172 (100), 153 (22); HRMS (EI) calcd for C₁₃H₁₇N (M⁺) 187.1361, found 187.1358.

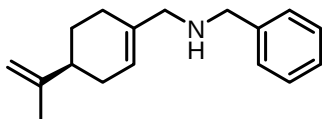


Compound **3k**: Isolated as a mixture of *Z/E* isomer (ratio = 1:7.7);

For *Z*-isomer: ¹H NMR (400 MHz, CDCl₃) δ 7.34–7.26 (m, 4H_c), 6.44 (br s, 1H_c), 3.38 (s, 2H), 2.51 (t, *J* = 7.2 Hz, 2H), 1.95 (s, 3H), 1.74 (br s, 1H), 1.46 (sextet, *J* = 7.3 Hz, 2H), 0.87 (t, *J* = 7.3 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 137.7, 137.2, 128.6, 128.0, 127.9, 126.2, 51.3, 50.3, 23.0, 22.8, 11.7.

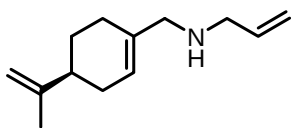
For *E*-isomer: ¹H NMR (400 MHz, CDCl₃) δ 7.34–7.26 (m, 4H), 6.44 (br s, 1H), 3.33 (s, 2H), 2.60 (t, *J* = 7.2 Hz, 2H), 1.89 (s, 3H), 1.74 (br s, 1H), 1.55 (sextet, *J* = 7.3 Hz, 2H), 0.92 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 138.0, 136.9, 128.8, 128.0, 126.1, 125.6, 58.0, 51.0, 23.1, 16.6, 11.8.

IR (CH₂Cl₂) 3425, 2940, 2772, 1723, 1578, 1455 cm⁻¹; LRMS (EI) 189 (M⁺, 3), 167 (47), 149 (100); HRMS (EI) calcd for C₁₃H₁₉N (M⁺) 189.1518, found 189.1520.

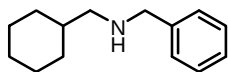


Compound **3l**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, *R_f* = 0.39; ¹H NMR (400 MHz, CDCl₃) δ 7.32–7.30 (m, 4H), 7.25–7.22 (m, 1H), 5.61 (s, 1H), 4.71 (s, 2H), 3.73 (s, 2H),

3.14 (s, 2H), 2.16–2.11 (m, 4H), 2.10–2.07 (m, 2H), 1.85–1.81 (m, 1H), 1.73 (s, 3H), 1.52–1.44 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.8, 140.3, 135.6, 128.2, 128.1, 126.9, 122.4, 108.5, 55.0, 52.9, 41.1, 30.5, 27.6, 27.3, 20.7; IR (CH_2Cl_2) 3320, 3029, 2917, 1646, 1449 cm^{-1} ; LRMS (EI) 241 (M^+ , 37), 172 (34), 91 (100); HRMS (EI) calcd for $\text{C}_{17}\text{H}_{23}\text{N}$ (M^+) 241.1831, found 241.1819.

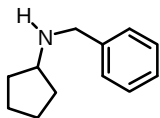


Compound **3m**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, R_f = 0.16; ^1H NMR (400 MHz, CDCl_3) δ 5.96–5.86 (m, 1H), 5.60 (br s, 1H), 5.17 (dt, J = 17.1, 1.6 Hz, 1H), 5.08 (dd, J = 10.2, 1.4 Hz, 1H), 4.71 (s, 2H), 3.20 (d, J = 5.7 Hz, 2H), 3.13 (s, 2H), 2.17–2.06 (m, 4H), 1.99–1.94 (m, 1H), 1.88–1.82 (m, 1H), 1.74 (s, 3H), 1.53–1.43 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 149.9, 136.9, 135.7, 122.2, 115.7, 108.5, 55.1, 51.7, 41.2, 30.5, 27.6, 27.4, 20.7; IR (CH_2Cl_2) 3351, 3085, 2929, 1646, 1455 cm^{-1} ; LRMS (EI) 191 (M^+ , 24), 153 (75), 136 (59), 122 (100); HRMS (EI) calcd for $\text{C}_{13}\text{H}_{21}\text{N}$ (M^+) 191.1674, found 191.1659.

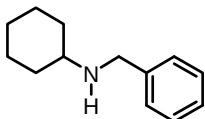


Compound **3n**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, R_f = 0.31; ^1H NMR (300 MHz, CDCl_3) δ 7.40 (d, J = 4.3 Hz, 4H), 7.36–7.30 (m, 1H), 3.85 (s, 2H), 2.54 (d, J = 6.6 Hz, 2H), 1.86–1.77 (m, 5H), 1.60–1.51 (m, 2H), 1.39–1.21 (m, 3H), 1.08–1.01 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 141.0, 128.6, 128.3, 127.1, 56.5, 54.4, 38.3, 31.8, 27.0, 26.4; IR (CH_2Cl_2) 3501, 3029,

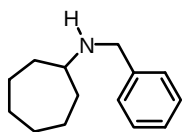
2923, 2846, 1449 cm^{-1} ; LRMS (EI) 203 (M^+ , 5), 120 (100); HRMS (EI) calcd for $\text{C}_{14}\text{H}_{21}\text{N}$ (M^+) 203.1674, found 203.1661.



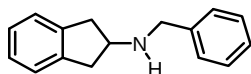
Compound **3o**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, R_f = 0.23; ^1H NMR (300 MHz, CDCl_3) δ 7.31–7.30 (m, 4H), 7.27–7.21 (m, 1H), 3.76 (s, 2H), 3.11 (quintet, J = 6.6 Hz, 1H), 1.90–1.79 (m, 2H), 1.76–1.64 (m, 2H), 1.55–1.46 (m, 3H), 1.42–1.31 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 141.0, 128.6, 128.4, 127.1, 59.1, 52.7, 33.1, 24.4; IR (CH_2Cl_2) 3431, 2956, 2860, 1640, 1455 cm^{-1} ; LRMS (EI) 175 (M^+ , 20), 146 (85), 91 (100); HRMS (EI) calcd for $\text{C}_{12}\text{H}_{17}\text{N}$ (M^+) 175.1361, found 175.1363.



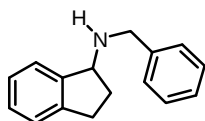
Compound **3p**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, R_f = 0.27; ^1H NMR (300 MHz, CDCl_3) δ 7.31–7.30 (m, 4H), 7.27–7.20 (m, 1H), 3.80 (s, 2H), 2.51–2.45 (m, 1H), 1.92–1.89 (br m, 2H), 1.71–1.74 (br m, 2H), 1.62–1.60 (br m, 1H), 1.44 (br s, 1H), 1.31–1.05 (m, 5H); ^{13}C NMR (75 MHz, CDCl_3) δ 140.9, 128.3, 128.0, 126.7, 56.1, 51.0, 33.5, 26.1, 24.9; IR (CH_2Cl_2) 3431, 3034, 2945, 2860, 1644, 1467 cm^{-1} ; LRMS (EI) 189 (M^+ , 28), 146 (91), 91 (100); HRMS (EI) calcd for $\text{C}_{13}\text{H}_{19}\text{N}$ (M^+) 189.1518, found 189.1509.



Compound **3q**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, $R_f = 0.23$; ^1H NMR (300 MHz, CDCl_3) δ 7.31–7.29 (m, 4H), 7.26–7.21 (m, 1H), 3.76 (s, 2H), 2.70–2.66 (br m, 1H), 1.89–1.81 (br m, 2H), 1.70–1.62 (br m, 2H), 1.55–1.50 (br m, 4H), 1.47–1.40 (m, 4H); ^{13}C NMR (75 MHz, CDCl_3) δ 140.6, 128.3, 128.1, 126.7, 58.2, 51.4, 34.6, 28.2, 24.3; IR (CH_2Cl_2) 3331, 2930, 2856, 1459 cm^{-1} ; LRMS (EI) 203 (M^+ , 45), 160 (24), 146 (100); HRMS (EI) calcd for $\text{C}_{14}\text{H}_{21}\text{N}$ (M^+) 203.1674, found 203.1670.

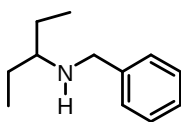


Compound **3r**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, $R_f = 0.62$; ^1H NMR (300 MHz, CDCl_3) δ 7.33–7.29 (m, 4H), 7.27–7.22 (m, 1H), 7.20–7.11 (m, 4H), 3.85 (s, 2H), 3.67 (quintet, $J = 6.8$ Hz, 1H), 3.17 (dd, $J = 15.5, 7.1$ Hz, 2H), 2.81 (dd, $J = 15.5, 7.1$ Hz, 2H), 1.70 (br s, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 141.7, 140.3, 128.4, 128.2, 127.0, 126.4, 124.7, 59.0, 52.3, 40.0; IR (CH_2Cl_2) 3295, 3031, 2837, 1496, 1454 cm^{-1} ; LRMS (EI) 223 (M^+ , 100), 132 (33); HRMS (EI) calcd for $\text{C}_{16}\text{H}_{17}\text{N}$ (M^+) 223.1361, found 223.1358.

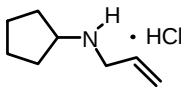


Compound **3s**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, $R_f = 0.62$; ^1H NMR (400 MHz, CDCl_3) δ 7.38–7.29 (m, 5H), 7.24–7.17 (m, 4H), 4.28 (t, $J = 6.6$ Hz, 1H), 3.88 (AB system,

2H), 3.00 (ddd, $J = 15.9, 8.6, 4.7$ Hz, 1H), 2.79 (quintet, $J = 7.8$ Hz, 1H), 2.44–2.36 (m, 1H), 2.16–1.96 (br s, 1H), 1.92–1.83 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ 145.1, 143.6, 140.4, 128.3, 128.1, 127.3, 126.8, 126.1, 124.7, 124.0, 62.6, 51.3, 33.4, 30.3; IR (CH_2Cl_2) 3429, 2958, 1712, 1457 cm^{-1} ; LRMS (EI) 223 (M^+ , 69), 222 (100), 132 (98), 117 (62); HRMS (EI) calcd for $\text{C}_{16}\text{H}_{16}\text{N}$ ($\text{M}^+ - 1$) 222.1282, found 222.1280.

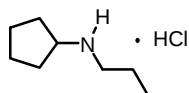


Compound **3t**: Analytical TLC (silica gel 60), 50 % EtOAc in *n*-hexane, $R_f = 0.27$; ^1H NMR (400 MHz, CDCl_3) δ 7.34–7.29 (m, 4H), 7.25–7.21 (m, 1H), 3.75 (s, 2H), 2.43 (quintet, $J = 5.9$ Hz, 1H), 1.60 (br s, 1H), 1.50–1.41 (m, 4H), 0.89 (t, $J = 7.5$ Hz, 6H); ^{13}C NMR (75 MHz, CDCl_3) δ 141.3, 128.7, 128.5, 127.1, 59.7, 51.5, 26.0, 10.2; IR (CH_2Cl_2) 3350, 2969, 2853, 1449 cm^{-1} ; LRMS (EI) 148 ($\text{M}^+ - \text{C}_2\text{H}_5$, 53), 91 (100); HRMS (EI) calcd for $\text{C}_{10}\text{H}_{14}\text{N}$ ($\text{M}^+ - \text{C}_2\text{H}_5$) 148.1226, found 148.1120.

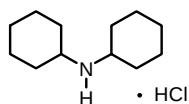


Compound **3u**: The compound was isolated as its HCl salt. ^1H NMR (300 MHz, CDCl_3) δ 9.48 (br s, 2H), 6.20–6.07 (m, 1H), 5.50 (d, $J = 17.1$ Hz, 1H), 5.44 (d, $J = 10.3$ Hz, 1H), 3.64–3.58 (m, 2H), 3.49–3.44 (m, 1H), 2.09–2.04 (br m, 2H), 2.00–1.84 (br m, 4H), 1.70–1.50 (br m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 128.0, 123.5, 57.9, 48.6, 29.4, 23.7; IR (CH_2Cl_2) 3384, 2970, 2791, 1675, 1449

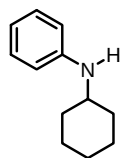
cm⁻¹; LRMS (EI) 126 (M⁺+H, 100), 125 (47); HRMS (EI) calcd for C₈H₁₅N (M⁺) 125.1204, found 125.1202.



Compound **3v**: The compound was isolated as its HCl salt. ¹H NMR (300 MHz, CDCl₃) δ 3.62–3.53 (m, 1H), 3.23–3.07 (br s, 1H), 3.05 (t, *J* = 7.7 Hz, 2H), 2.25–2.10 (br m, 2H), 1.95–1.75 (br m, 6H), 1.70–1.64 (m, 2H), 1.03 (t, *J* = 7.3 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 60.6, 60.4, 49.5, 49.4, 29.7, 23.8, 19.7, 10.9; IR (CH₂Cl₂) 3116, 2974, 2871, 1593, 1455 cm⁻¹; LRMS (EI) 127 (M⁺, 15), 98 (100); HRMS (EI) calcd for C₈H₁₇N (M⁺) 127.1361, found 127.1362.

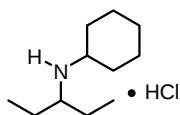


Compound **3w**: ¹H NMR (300 MHz, CDCl₃) δ 3.06 (t, *J* = 11.1 Hz, 2H), 2.20 (d, *J* = 11.1 Hz, 4H), 1.84 (br s, 4H), 1.72–1.65 (br m, 6H), 1.24 (br s, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 53.7, 29.3, 24.9, 24.8; IR (CH₂Cl₂) 3429, 2946, 2867, 1455 cm⁻¹; LRMS (EI) 181 (M⁺, 8), 149 (40), 138 (100); HRMS (EI) calcd for C₁₂H₂₃N (M⁺) 181.1831, found 181.1841.

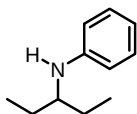


Compound **3x**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, *R_f* = 0.66; ¹H NMR (400

MHz, CDCl₃) δ 7.16–7.11 (m, 2H), 6.64 (t, J = 7.3 Hz, 1H), 6.57 (d, J = 7.7 Hz, 2H), 3.50–3.40 (br s, 1H), 3.27–3.20 (m, 1H), 2.04 (dd, J = 12.9, 3.2 Hz, 2H), 1.74 (dt, J = 13.4, 3.8 Hz, 2H), 1.63 (dt, J = 13.4, 3.8 Hz, 1H), 1.38–1.31 (m, 2H), 1.25–1.22 (m, 1H), 1.19–1.11 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 147.4, 129.2, 116.8, 113.1, 51.6, 33.5, 25.9, 25.0; IR (CH₂Cl₂) 3417, 2934, 2856, 1602, 1504 cm⁻¹; LRMS (EI) 175 (M⁺, 45), 132 (100); HRMS (EI) calcd for C₁₂H₁₇N (M⁺) 175.1361, found 175.1360.



Compound **3y**: ¹H NMR (300 MHz, CDCl₃) δ 6.64 (br s, 1H), 3.15–3.08 (br m, 2H), 2.15–2.11 (m, 3H), 1.89–1.86 (m, 3H), 1.81 (quintet, J = 7.3 Hz, 4H), 1.68–1.54 (m, 4H), 1.03 (t, J = 7.3 Hz, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 58.8, 56.4, 24.7, 24.6, 24.2, 22.6, 9.13; IR (CH₂Cl₂) 3301, 2946, 2861, 1594, 1449 cm⁻¹; LRMS (EI) 140 (M⁺–C₂H₅, 100), 126 (12); HRMS (EI) calcd for C₉H₁₈N (M⁺–C₂H₅) 140.1439, found 140.1457.



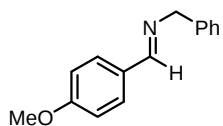
Compound **3z**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, R_f = 0.77; ¹H NMR (400 MHz, CDCl₃) δ 7.23 (t, J = 5.9 Hz, 2H), 6.63 (t, J = 7.3 Hz, 1H), 6.56 (d, J = 7.7 Hz, 2H), 3.49–3.40 (br s, 1H), 3.22 (quintet, J = 6.0 Hz, 1H), 1.58 (heptet, J = 7.4 Hz, 2H), 1.47 (heptet, J = 7.4 Hz, 2H), 0.92 (t, J = 7.6 Hz, 6H); ¹³C NMR (75 MHz, CDCl₃) δ 148.2, 129.2, 116.5, 112.9, 55.4,

26.8, 10.1; IR (CH₂Cl₂) 3417, 2963, 2929, 1594, 1499, 1302 cm⁻¹; LRMS (EI) 163 (M⁺, 15), 134 (100), 132 (15); HRMS (EI) calcd for C₁₁H₁₇N (M⁺) 163.1361, found 163.1364.

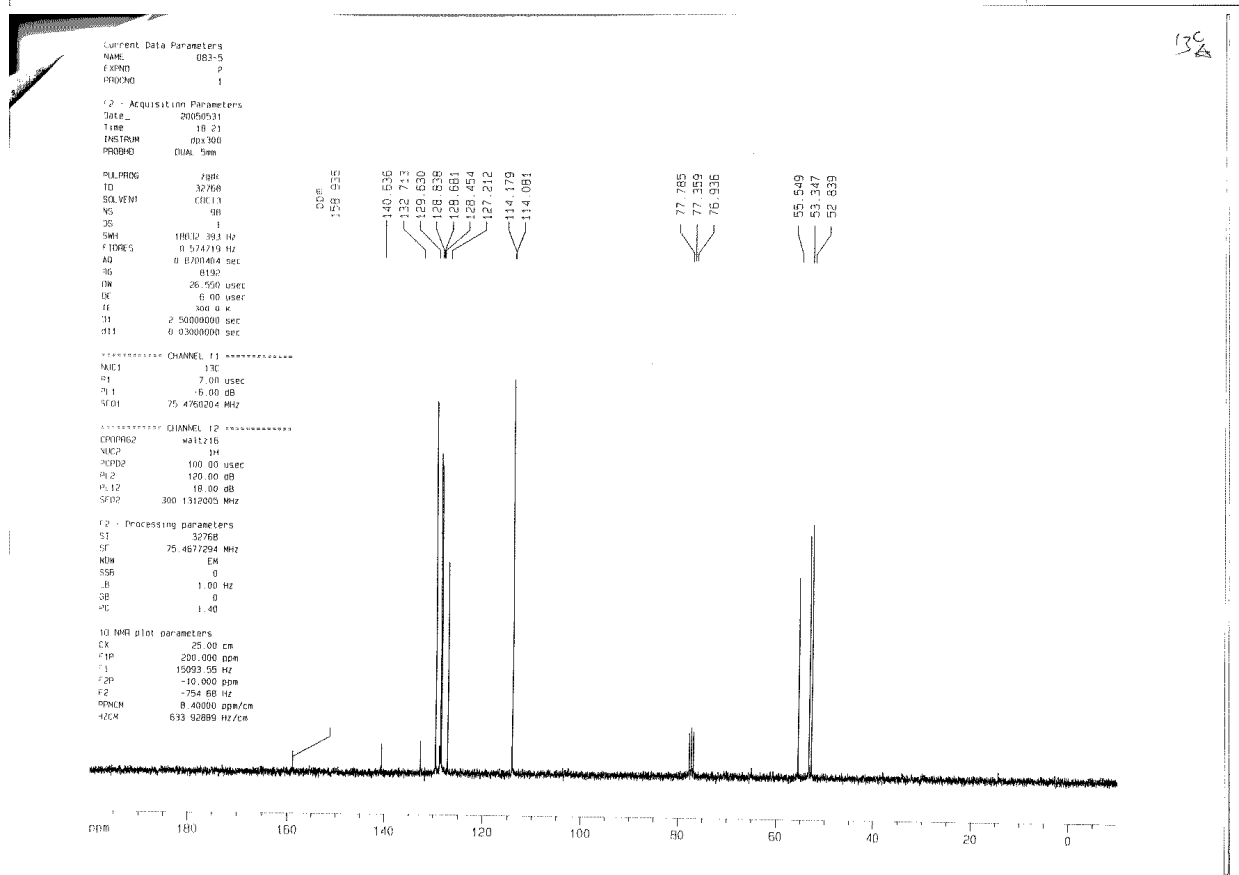
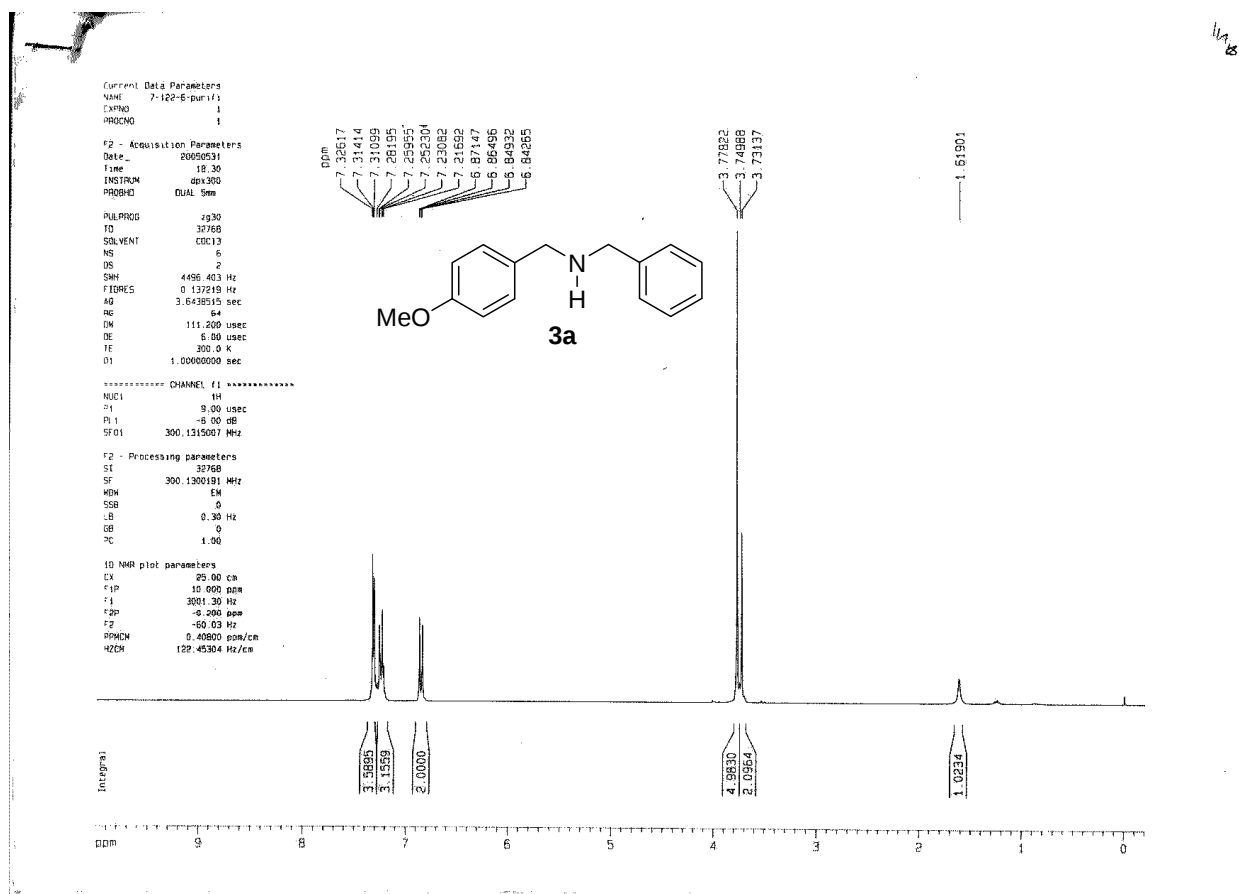
Procedure for the Preparation of Imine 4

To a stirred solution of 4-methoxybenzaldehyde (3.00 g, 22.0 mmol) and benzylamine (2.4 mL, 22.0 mmol) in CH₂Cl₂ (80 mL) was added Na₂SO₄ (6.30 g, 44.0 mmol) at room temperature. The reaction was allowed to stir overnight and was monitored by TLC. When the reaction was completed, the mixture was filtered and concentrated. The crude product was purified by flash column chromatography (5 % ethyl acetate in *n*-hexane) to afford **4** (4.91 g, 21.8 mmol, 99 % yield) as a yellow oil.

Characterization of Compound 4



Compound **4**: Analytical TLC (silica gel 60), 20 % EtOAc in *n*-hexane, *R_f* = 0.57; ¹H NMR (400 MHz, CDCl₃) δ 8.33 (s, 1H), 7.72 (d, *J* = 8.8 Hz, 2H), 7.34–7.32 (m, 3H), 7.26–7.25 (m, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 4.79 (s, 2H), 3.84 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 162.1, 161.3, 139.6, 129.8, 129.2, 128.5, 128.0, 127.0, 114.0, 65.0, 55.4; IR (CH₂Cl₂) 2856, 1613, 1517 cm⁻¹; LRMS (EI) 225 (M⁺, 22), 224 (21), 134 (5); HRMS (EI) calcd for C₁₅H₁₅NO (M⁺) 225.1154, found 225.1145.



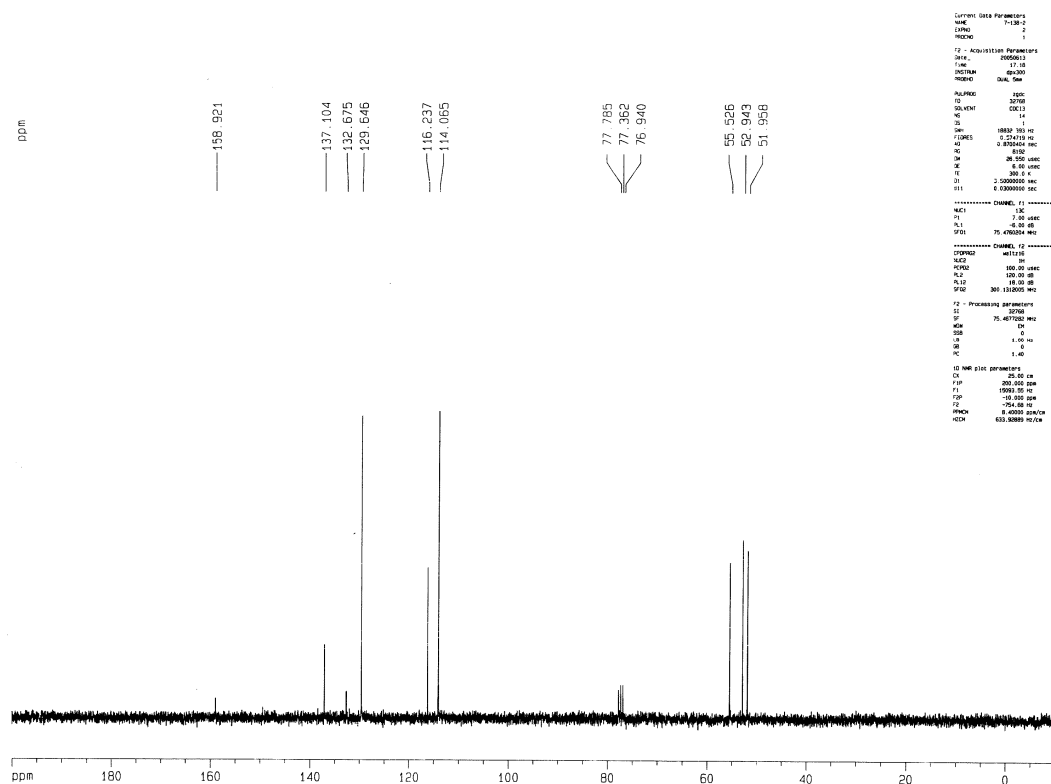
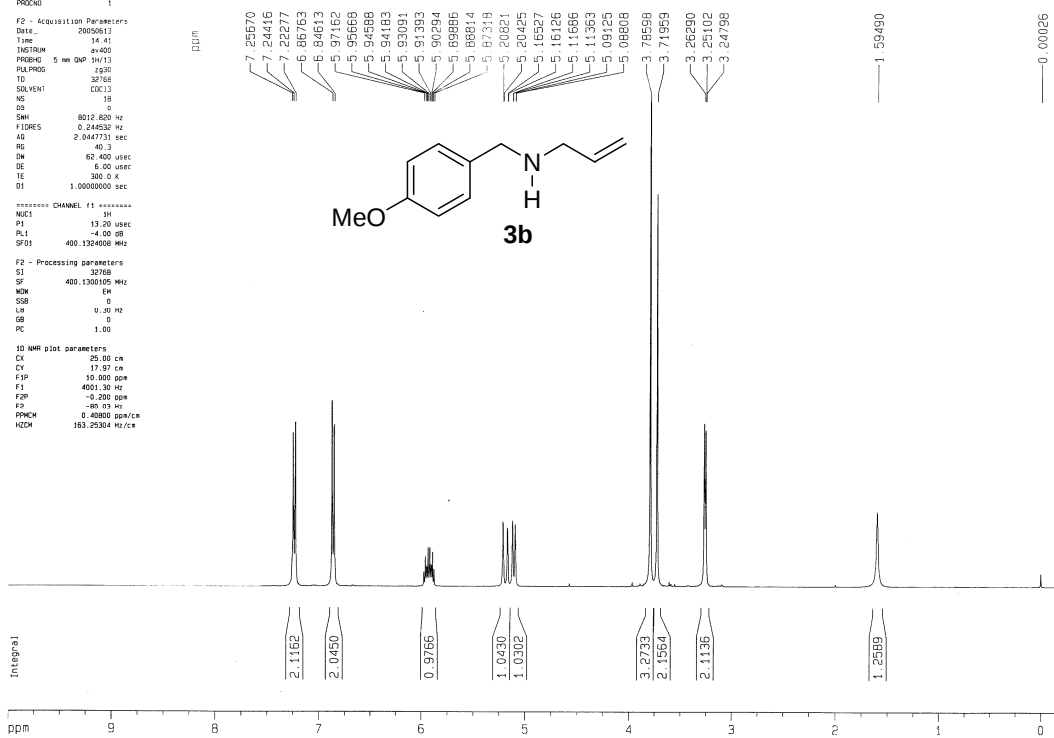
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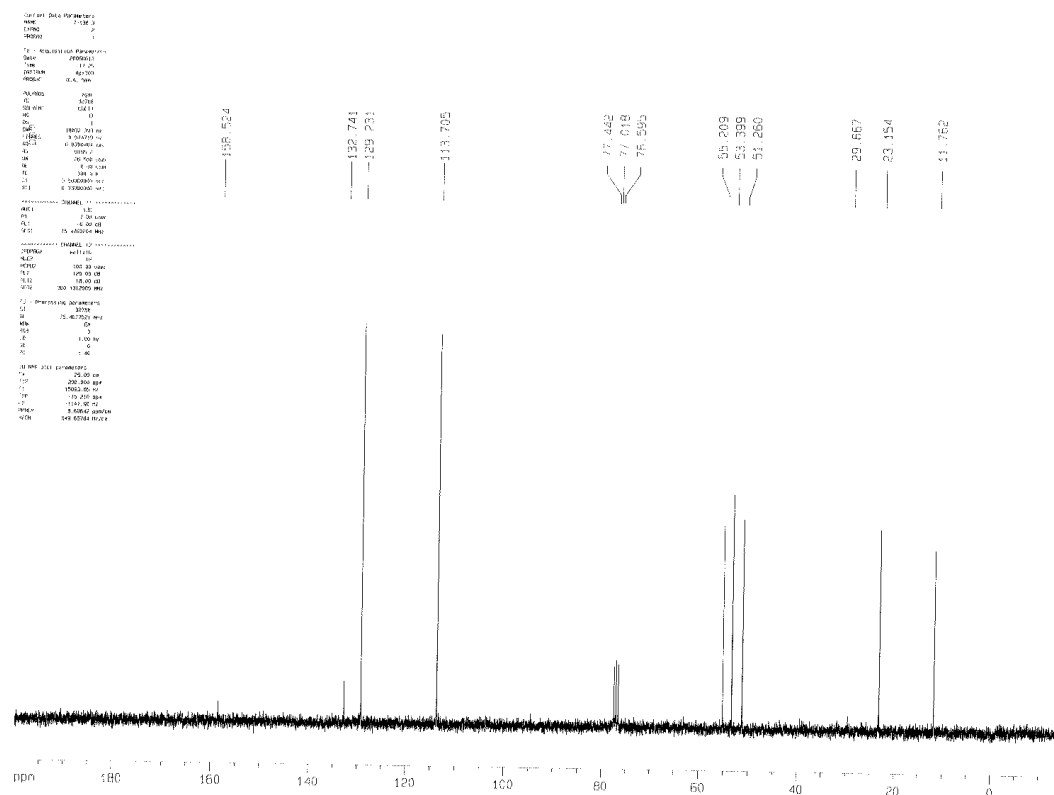
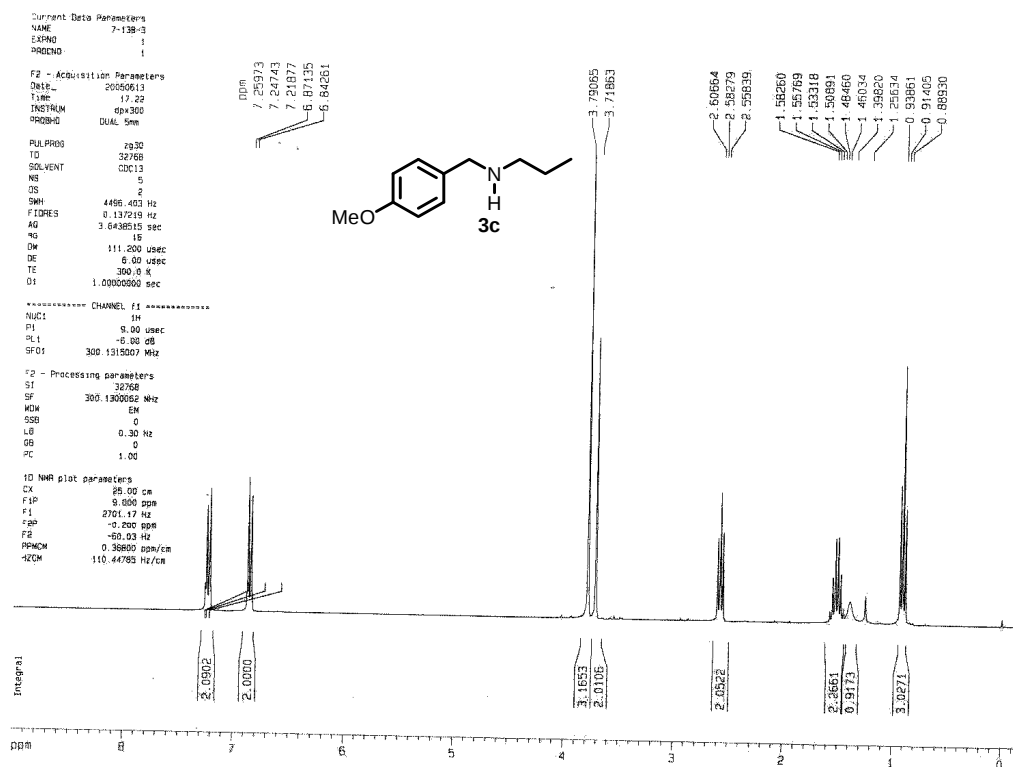
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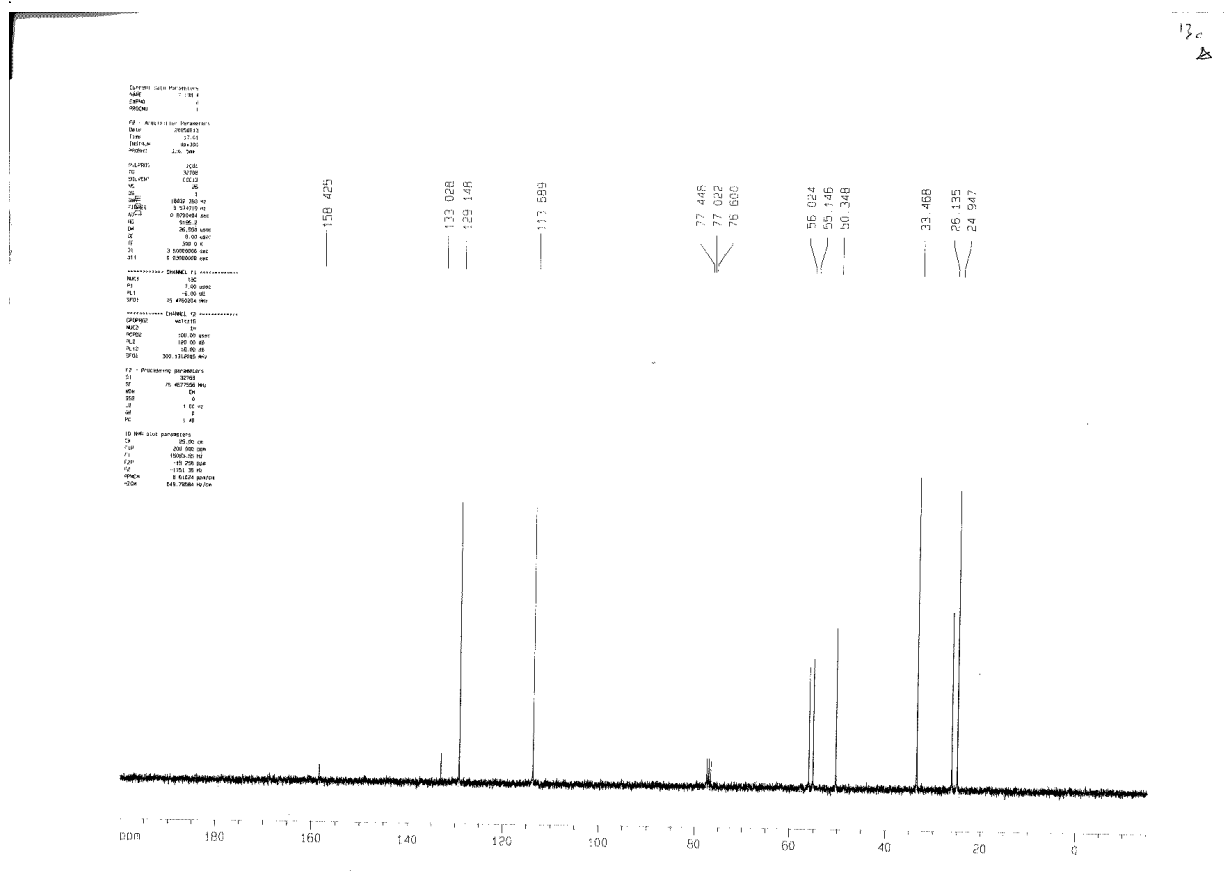
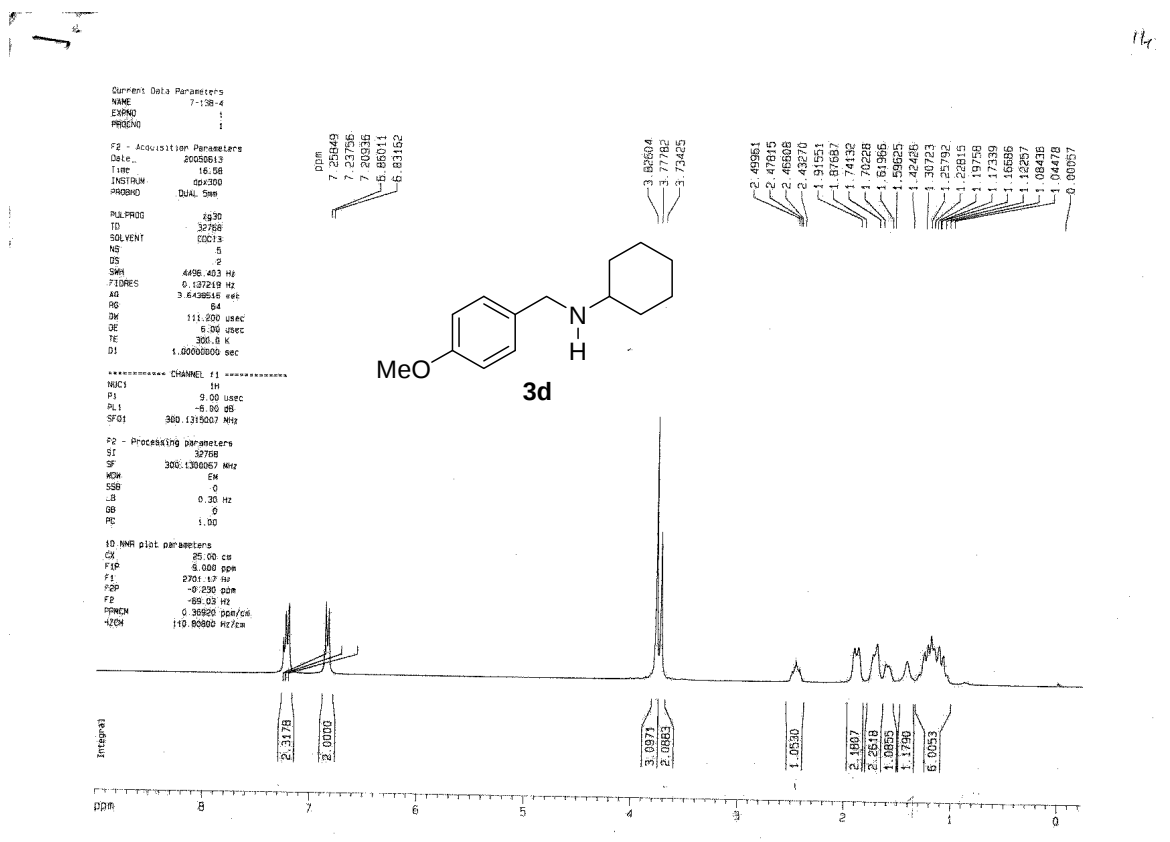
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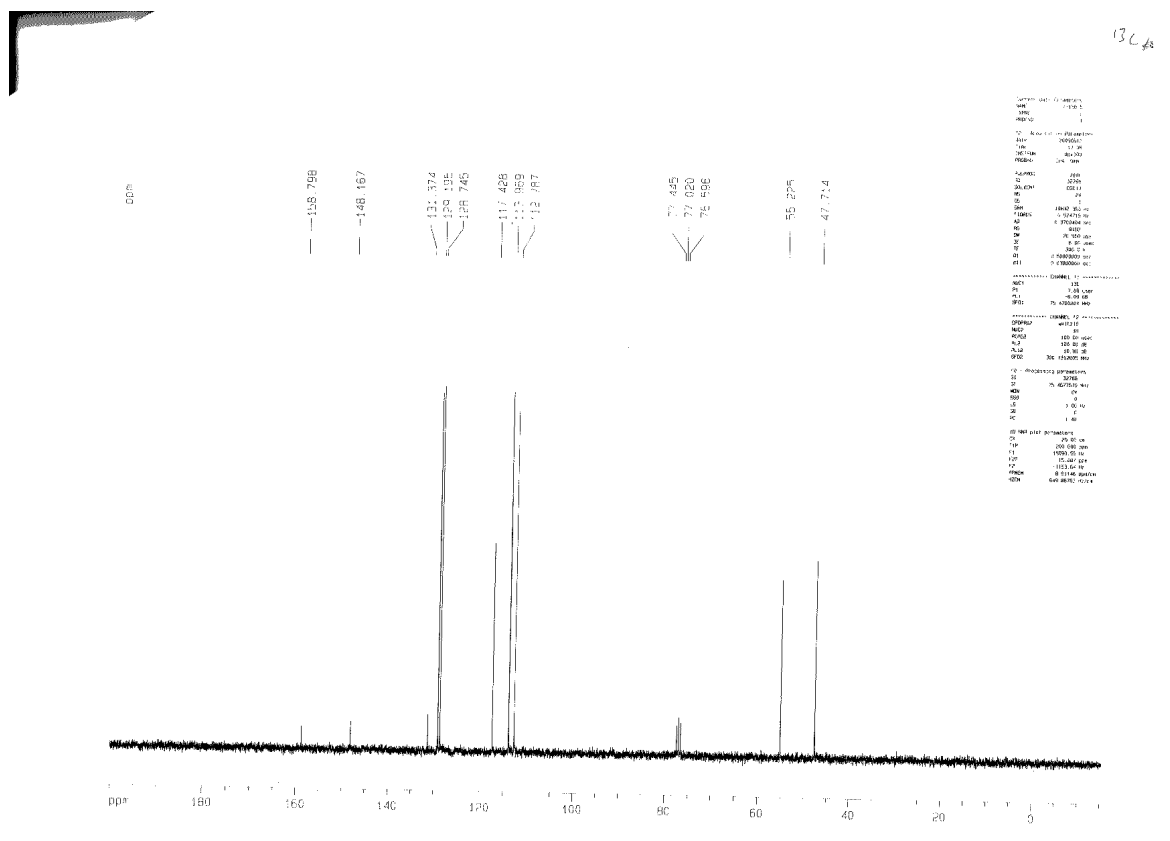
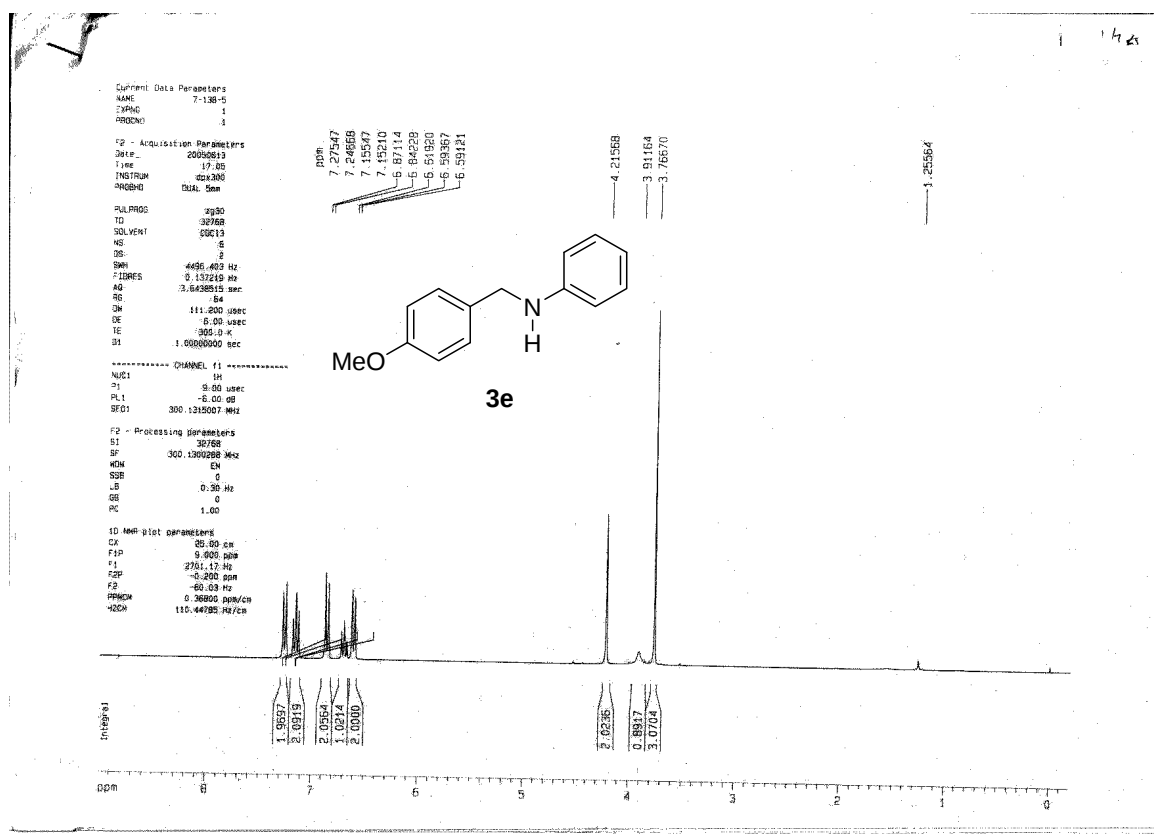
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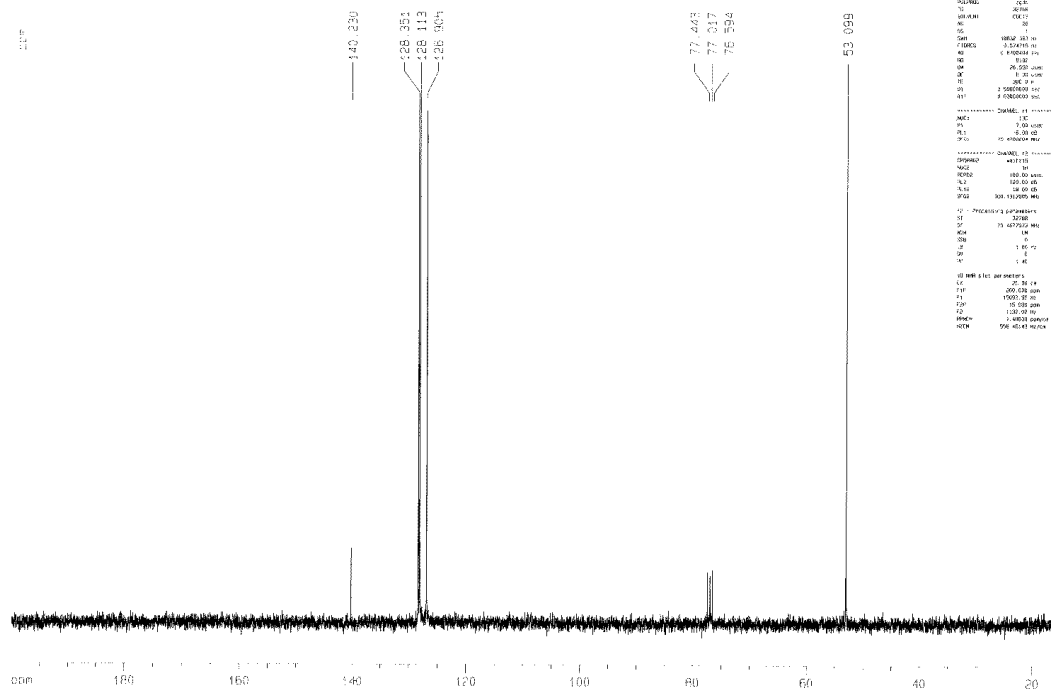
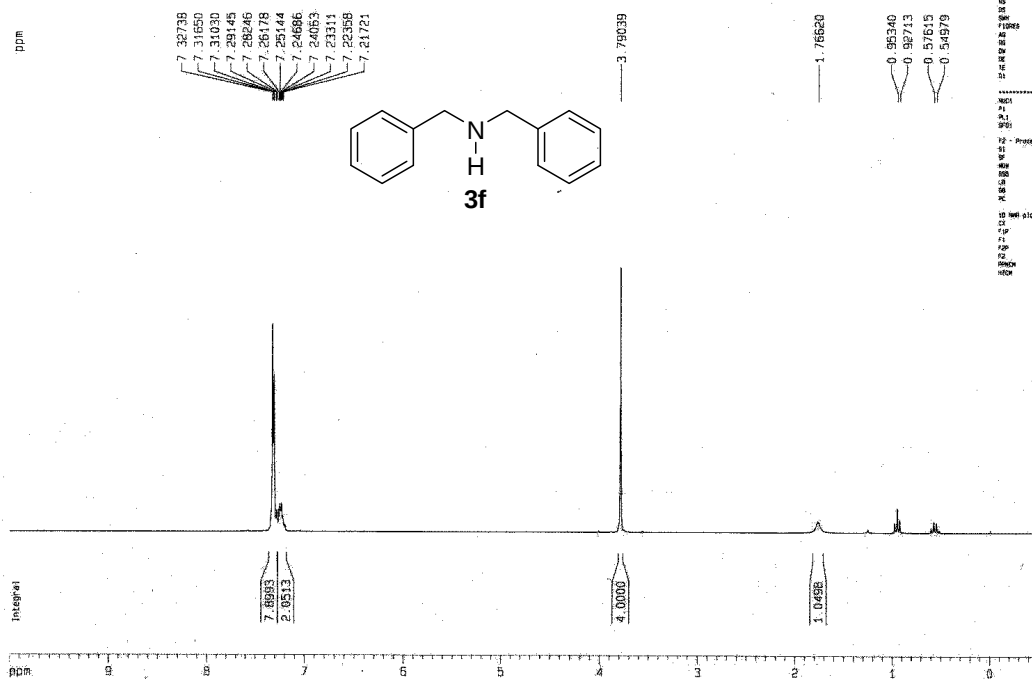
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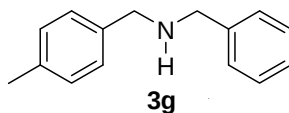
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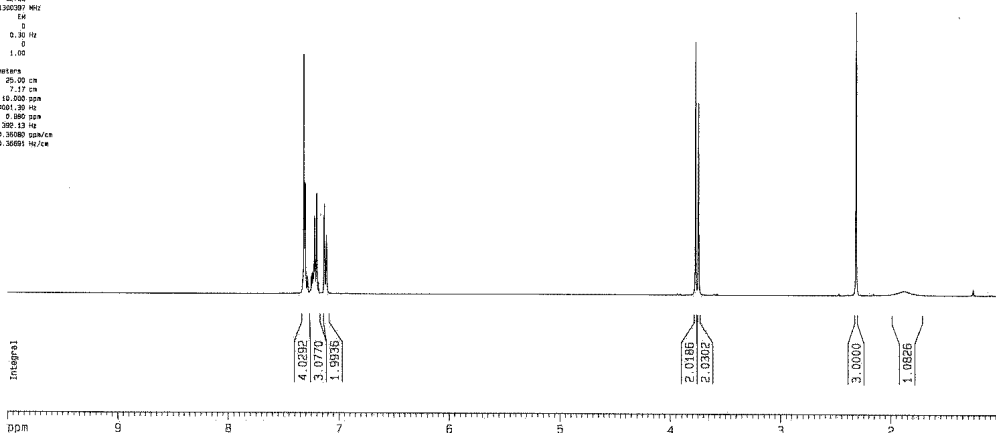


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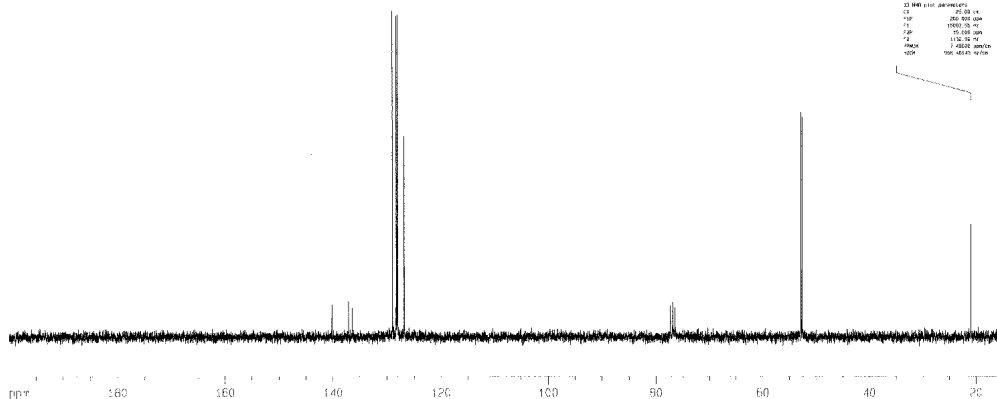


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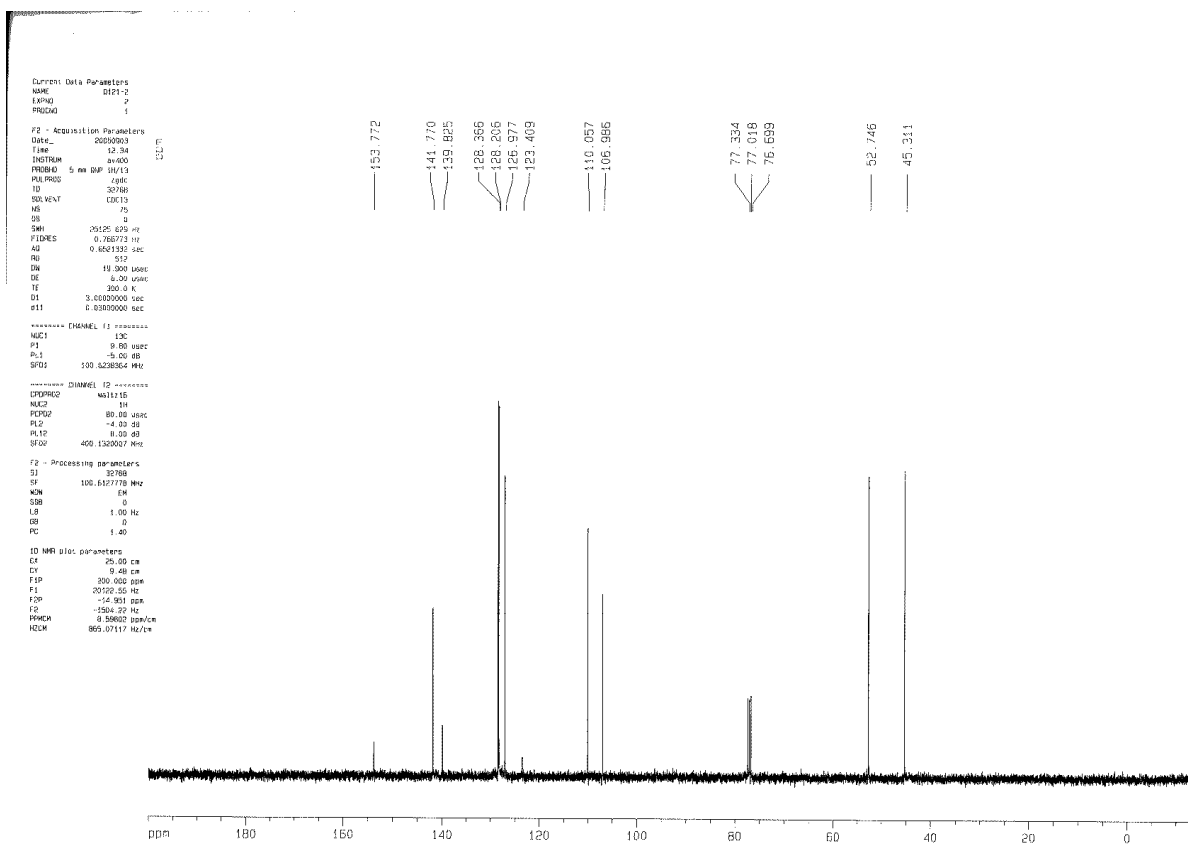
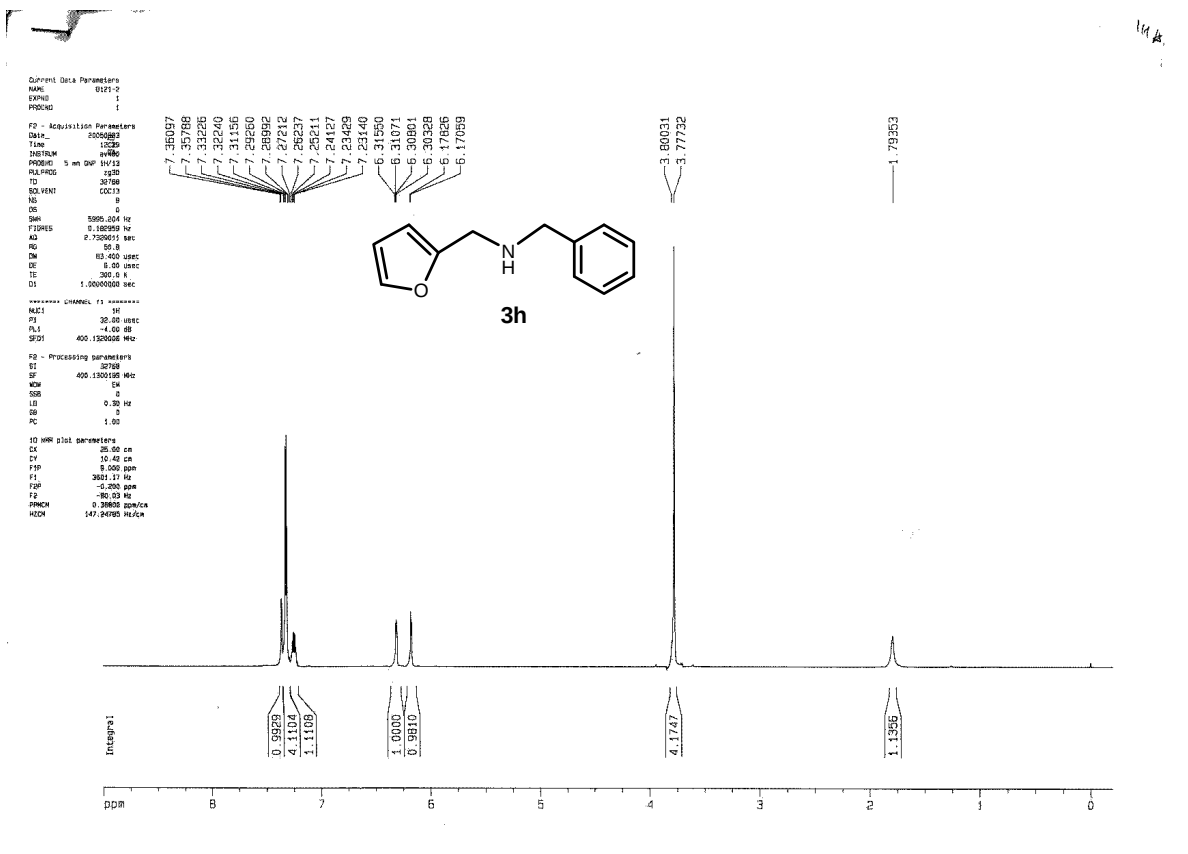
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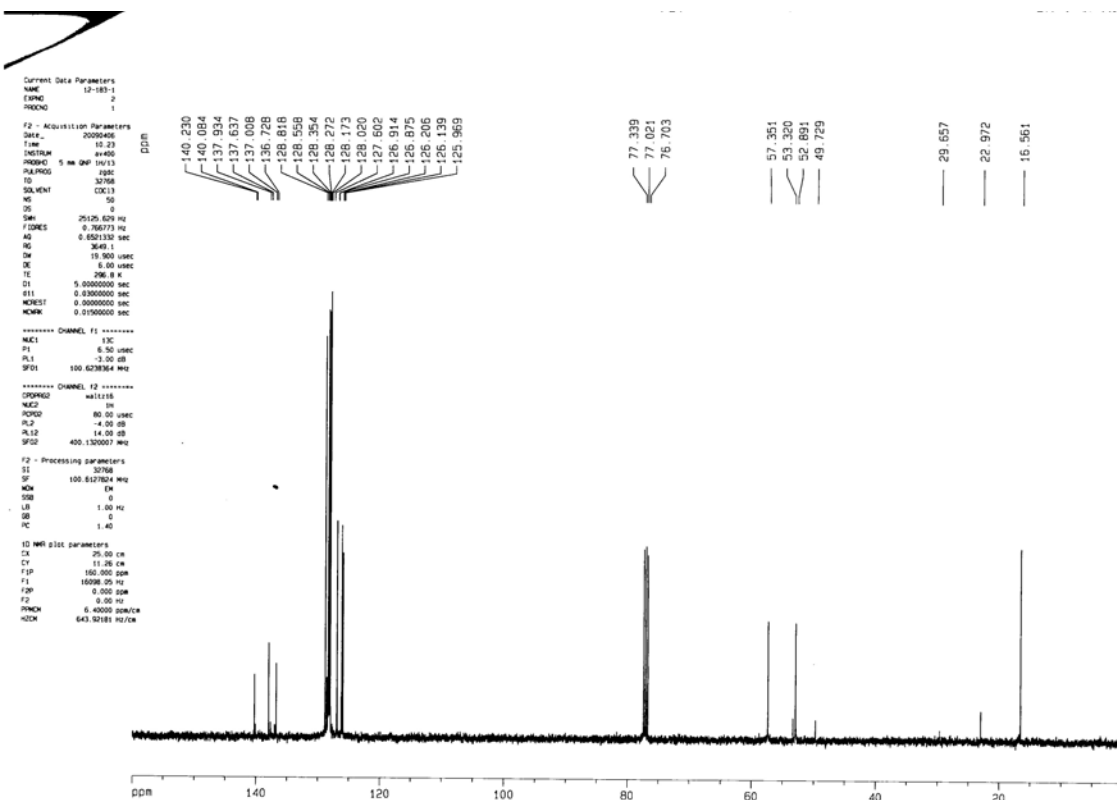
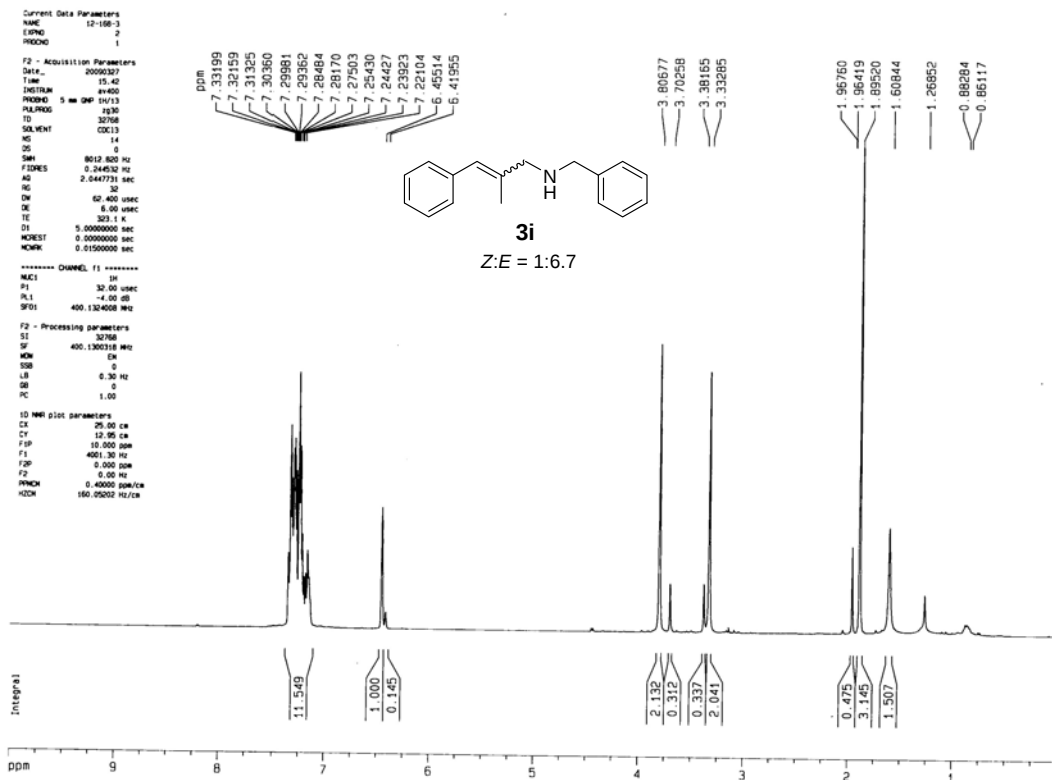
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TE 300.2 K
D1 1.00000000 sec

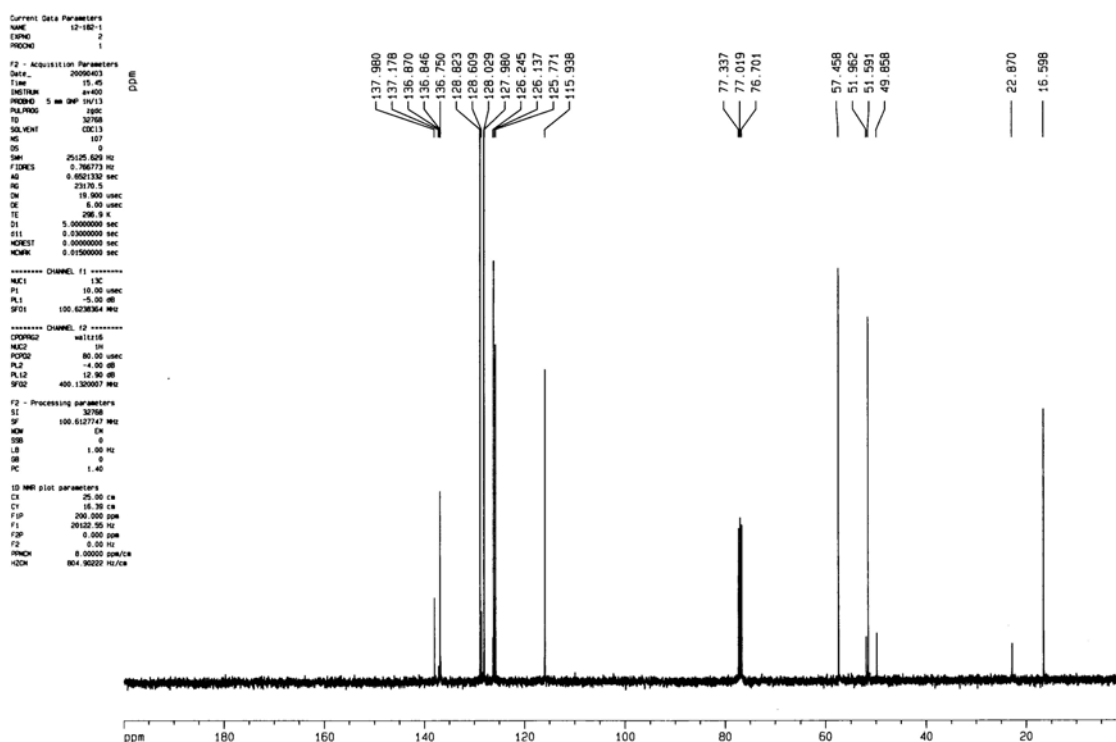
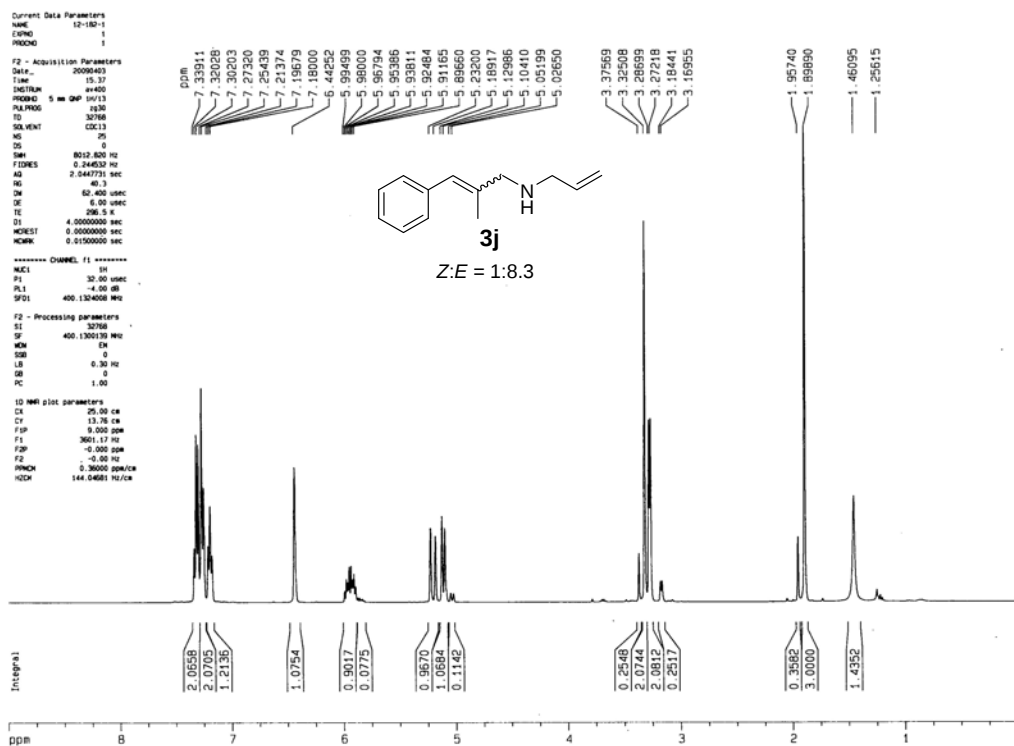
===== CHANNEL f2 =====
NAME 13C
P1 13.00 usec
PL1 0.00 dB
SFO2 100.6250000 MHz

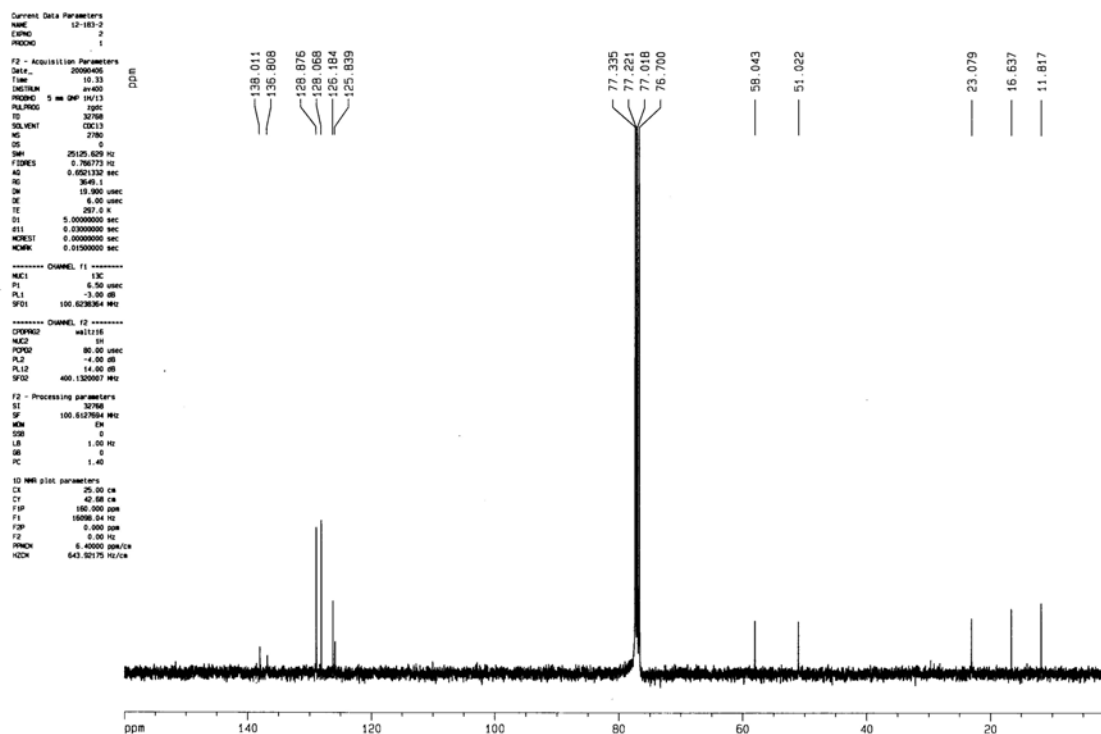
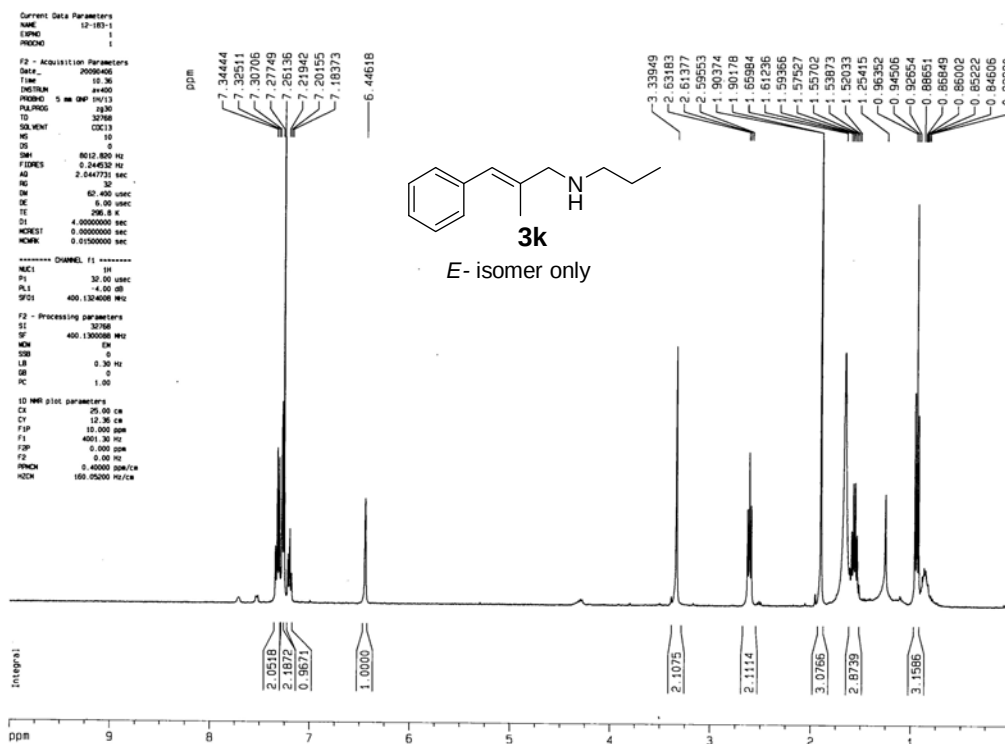
F2 - Processing Parameters
SI 32768
SF 100.6250000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
SI 25.00 cm
CY 7.17 cm
FID 10.000 cm
F1 400.130 MHz
F2 100.625 MHz
F3 0.000 cm
F4 0.000 cm
F5 0.000 cm
F6 0.000 cm
F7 0.000 cm
F8 0.000 cm
F9 0.000 cm
F10 0.000 cm
F11 0.000 cm
F12 0.000 cm
F13 0.000 cm
F14 0.000 cm
F15 0.000 cm
F16 0.000 cm
F17 0.000 cm
F18 0.000 cm
F19 0.000 cm
F20 0.000 cm
F21 0.000 cm
F22 0.000 cm
F23 0.000 cm
F24 0.000 cm
F25 0.000 cm
F26 0.000 cm
F27 0.000 cm
F28 0.000 cm
F29 0.000 cm
F30 0.000 cm
F31 0.000 cm
F32 0.000 cm
F33 0.000 cm
F34 0.000 cm
F35 0.000 cm
F36 0.000 cm
F37 0.000 cm
F38 0.000 cm
F39 0.000 cm
F40 0.000 cm
F41 0.000 cm
F42 0.000 cm
F43 0.000 cm
F44 0.000 cm
F45 0.000 cm
F46 0.000 cm
F47 0.000 cm
F48 0.000 cm
F49 0.000 cm
F50 0.000 cm
F51 0.000 cm
F52 0.000 cm
F53 0.000 cm
F54 0.000 cm
F55 0.000 cm
F56 0.000 cm
F57 0.000 cm
F58 0.000 cm
F59 0.000 cm
F60 0.000 cm
F61 0.000 cm
F62 0.000 cm
F63 0.000 cm
F64 0.000 cm
F65 0.000 cm
F66 0.000 cm
F67 0.000 cm
F68 0.000 cm
F69 0.000 cm
F70 0.000 cm
F71 0.000 cm
F72 0.000 cm
F73 0.000 cm
F74 0.000 cm
F75 0.000 cm
F76 0.000 cm
F77 0.000 cm
F78 0.000 cm
F79 0.000 cm
F80 0.000 cm
F81 0.000 cm
F82 0.000 cm
F83 0.000 cm
F84 0.000 cm
F85 0.000 cm
F86 0.000 cm
F87 0.000 cm
F88 0.000 cm
F89 0.000 cm
F90 0.000 cm
F91 0.000 cm
F92 0.000 cm
F93 0.000 cm
F94 0.000 cm
F95 0.000 cm
F96 0.000 cm
F97 0.000 cm
F98 0.000 cm
F99 0.000 cm
F100 0.000 cm









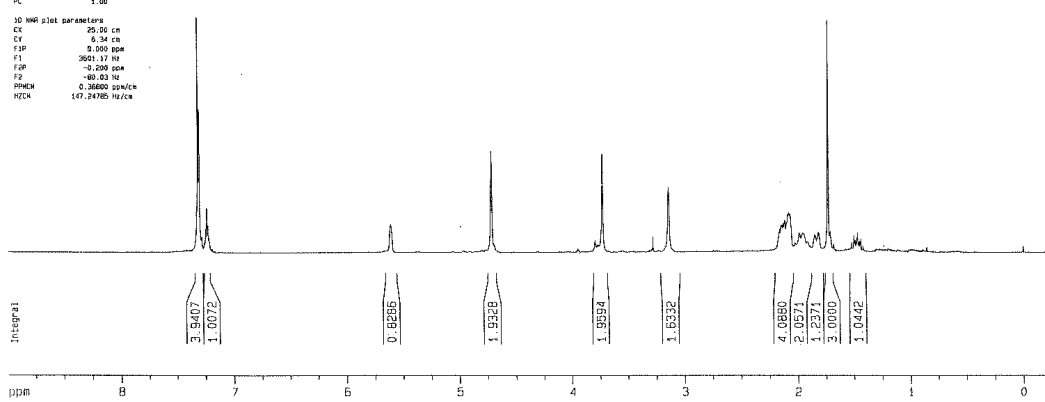
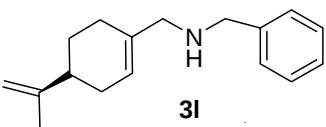
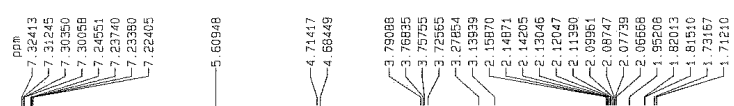
Current Data Parameters
NAME 6-48-B-3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050909
Time 19:37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 11
DS 0
SWH 8012.500 Hz
FIDRES 0.244530 Hz
AQ 2.044733 sec
RG 38.2
OW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 32.00 usec
PL1 -4.00 dB
SFO1 400.1304000 MHz

F2 - Processing parameters
SI 32768
SF 400.1304000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 25.00 cm
CY 6.34 cm
FSP 0.000 gpm
FI 3601.17 Hz
FPI -0.200 usec
F2 -49.83 Hz
PRCH 0.36600 gpm/cm
HCKA 147.24780 Hz/cm



Current Data Parameters
NAME 6-48-B-3
EXPNO 1
PROCNO 1

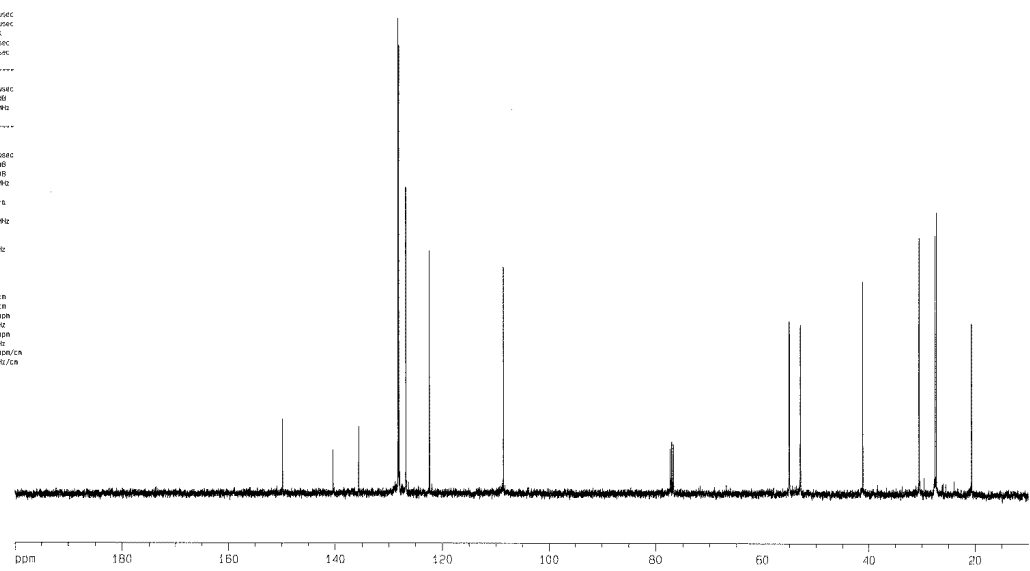
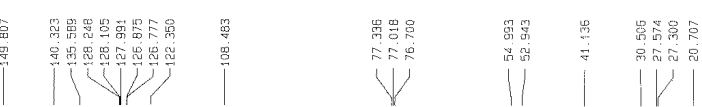
F2 - Acquisition Parameters
Date_ 20050909
Time 19:37
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 11
DS 0
SWH 75129.500 Hz
FIDRES 0.156773 Hz
AQ 2.044733 sec
RG 38.2
OW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec

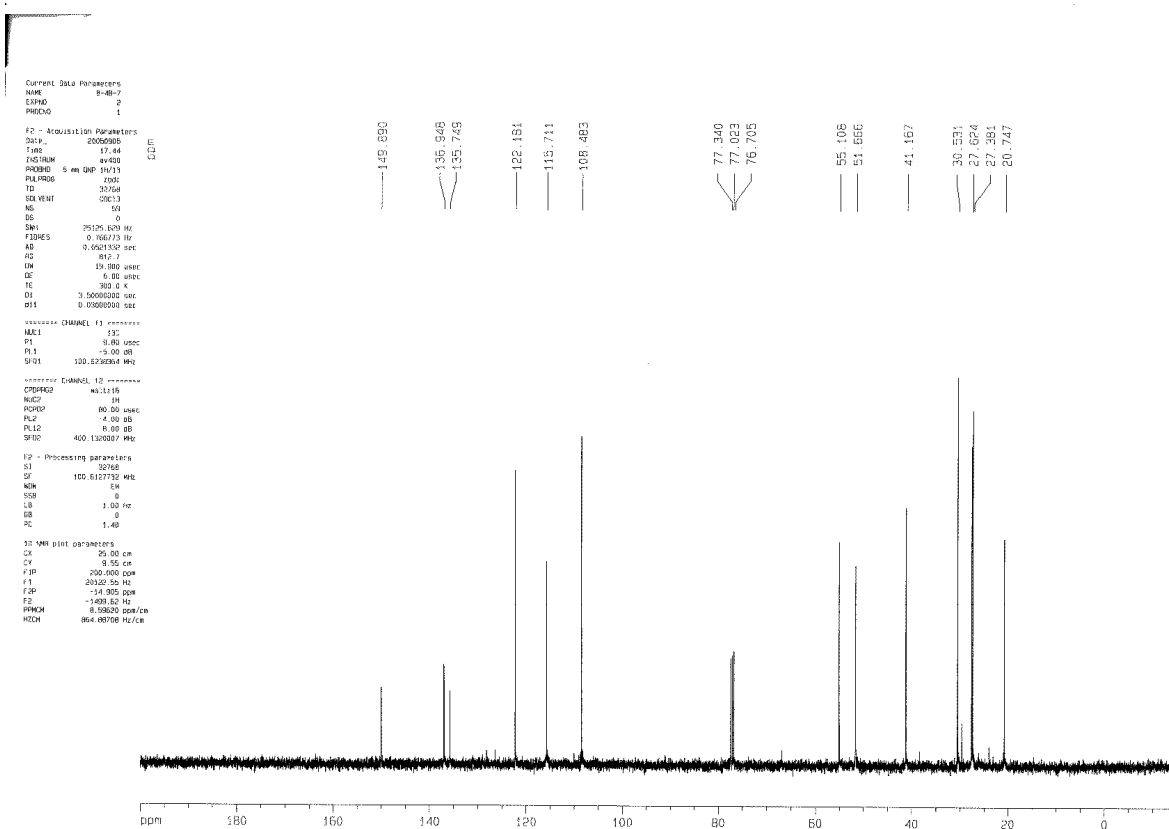
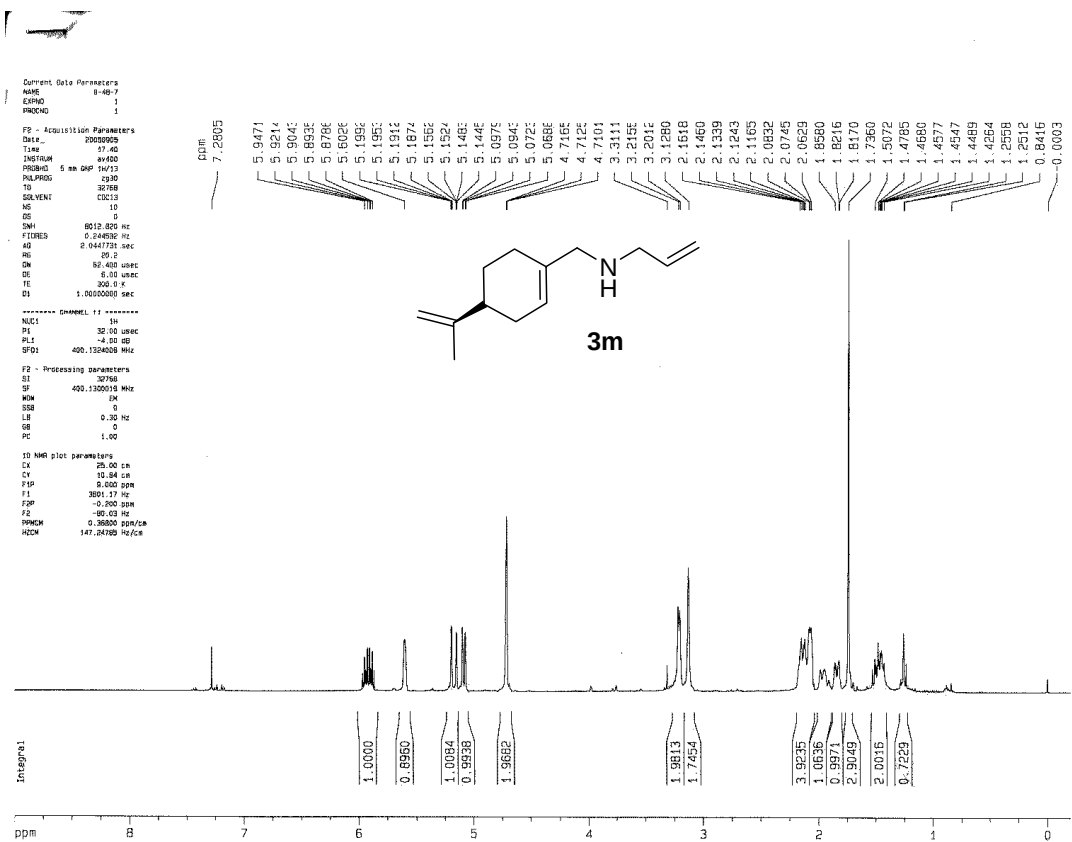
===== CHANNEL f1 =====
NUC1 13C
P1 9.00 usec
PL1 -5.00 dB
SFO1 100.6280000 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 9.00 dB
SFO2 400.1304000 MHz

F2 - Processing parameters
SI 32768
SF 100.6280000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 25.00 cm
CY 12.04 cm
FSP 0.000 gpm
FI 20420.16 Hz
FPI 0.200 usec
F2 1008.13 Hz
PRCH 7.60800 gpm/cm
HCKA 807.7 Hz/cm





Current Data Parameters
NAME 0117-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050830
Time 17:40
INSTRUM dpx300
PROBHD QNP 5mm

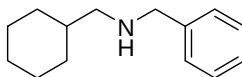
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 10
DS 2
SWH 7485.030 Hz
FIDRES 0.230425 Hz
AQ 2.1089594 sec
RG 50.8
SM 66.800 usec
DE 0.00 usec
TE 300.2 K
D1 1.0000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 8.00 usec
PL1 -6.00 dB
SFO1 300.1355007 MHz

F2 - Processing parameters
SI 32768
SF 300.1299662 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
RC 1.00

1D NMR plot parameters
CX 25.00 cm
FIP 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCH 0.42900 ppm/cm
HZCM 126.08458 Hz/cm

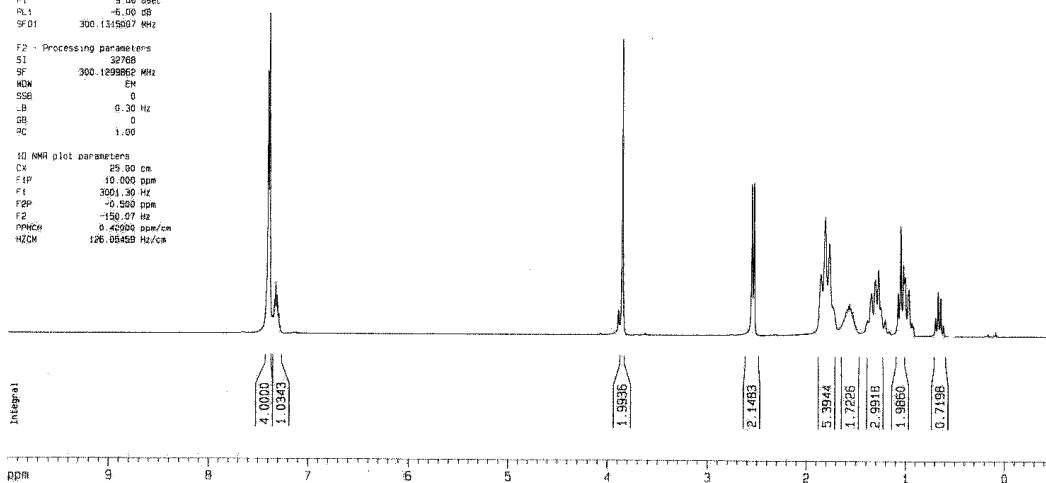
7.404013
7.390032
7.361566
7.355543
7.341152
7.327771
7.314622
7.300113



3n

3.99290
3.85279

2.55673
2.53459
1.85786
1.81629
1.77238
1.60362
1.59241
1.58100
1.56976
1.55841
1.54990
1.53321
1.5075
1.31319
1.27800
1.25346
1.08004
1.05371
1.02660
1.00948
0.97148
0.67199
0.64556



Current Data Parameters
NAME 0117-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050830
Time 17:47
INSTRUM dpx300
PROBHD QNP 5mm

PULPROG zgpg
TD 32768
SOLVENT CDCl₃
NS 217
DS 1
SWH 18832.303 Hz
FIDRES 0.574719 Hz
AQ 0.8700484 sec
RG 8192
SM 26.050 usec
DE 9.00 usec
TE 300.2 K
D1 2.5000000 sec
D11 0.0300000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SFO1 75.4769204 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 300.1312605 MHz

F2 - Processing parameters
SI 32768
SF 75.4672868 MHz
WDW EK
SSB 0
LB 1.00 Hz
GB 0
RC 1.40

1D NMR plot parameters
CX 25.00 cm
FIP 238.000 ppm
F1 17307.36 Hz
F2P -13.000 ppm
F2 -754.68 Hz
PPMCH 9.50000 ppm/cm
HZCM 724.49023 Hz/cm

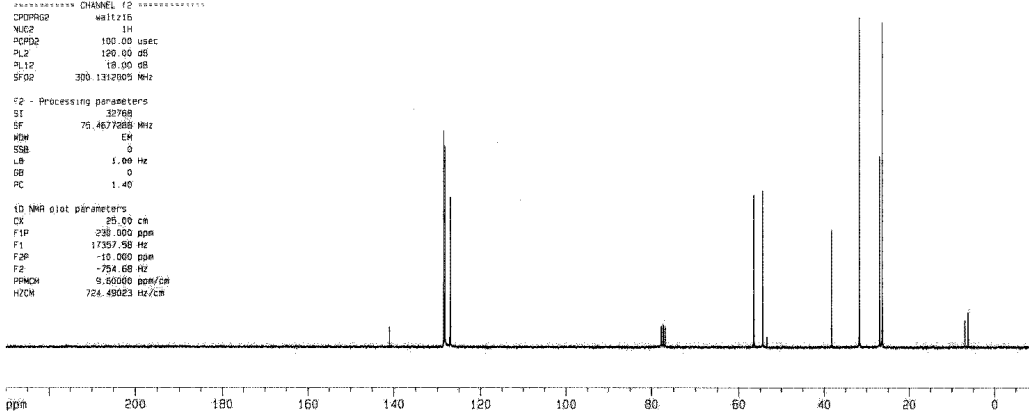
141.000
128.666
128.610
128.429
128.334
127.075

77.786
77.382
76.938

55.528
54.464

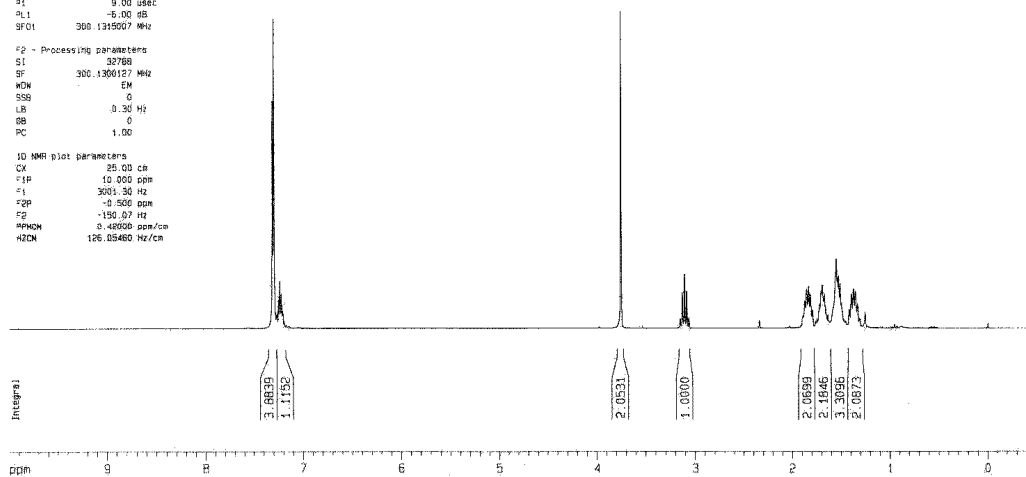
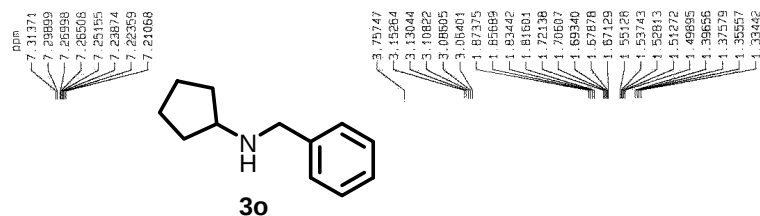
38.299
31.772
27.006
26.385

5.970
6.220



Current Data Parameters
 NAME: 007-1-pure
 -KNO: 1
 -PROBHD: 1
 #2 - Acquisition Parameters
 Date_: 20050623
 Time: 14.12
 INSTRUM: spect
 PROBHD: QNP 5mm
 PULPROG: zgpg30
 TO: 32768
 SOLVENT: CDCl3
 NS: 14
 DS: 2
 SWH: 7485.030 Hz
 FIDRES: 0.226425 Hz
 AQ: 2.1689524 sec
 RG: 71.8
 DW: 66.800 usec
 DE: 6.00 usec
 TE: 300.0 K
 D1: 1.00000000 sec

===== CHANNEL f1 =====
 NUC1: 1H
 P1: 9.00 usec
 PL1: -5.00 dB
 SFO1: 300.1315007 MHz
 #2 - Processing parameters
 SI: 32768
 SF: 300.1300127 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00
 #3 NMR plot parameters
 CY: 25.00 cm
 FIP: 10.000 ppm
 FI: 3001.30 Hz
 FOP: -0.300 ppm
 FQ: -150.07 Hz
 RPPHOM: 0.48000 ppm/cm
 RICH: 126.05460 Hz/cm



Current Data Parameters
NAME DB7-1-pure
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050623
Time 14.13
INSTRUM gpc300
PROBHD DUAL 5mm

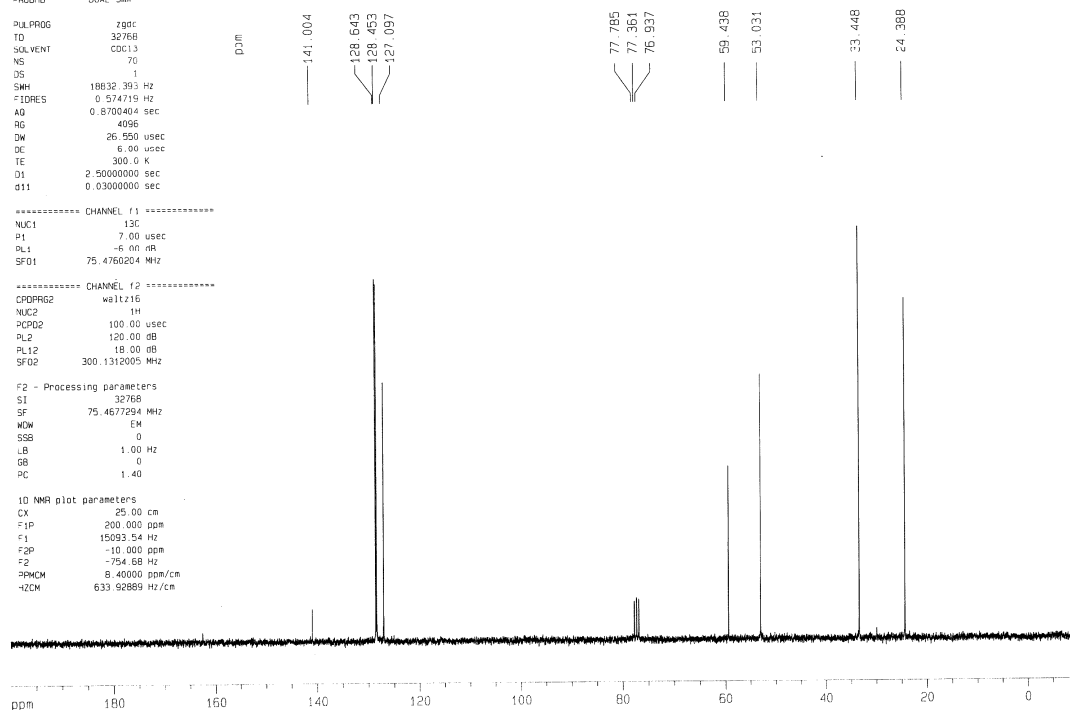
PULPROG zgpgc
TD 32768
SOLVENT CDCl3
NS 70
DS 1
SWH 18632.383 Hz
FIDRES 0.574719 Hz
AQ 0.8700404 sec
RG 4096
DM 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.5000000 sec
d11 0.0300000 sec

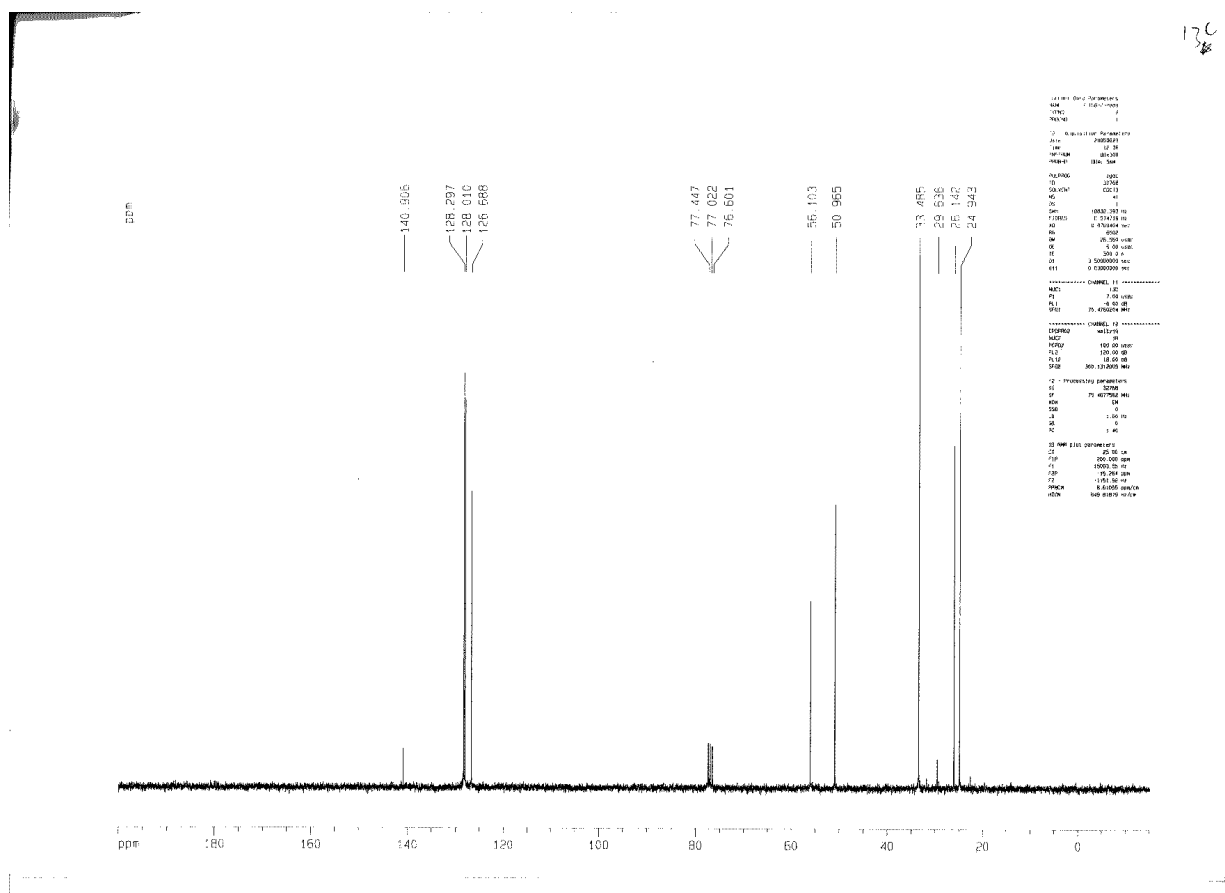
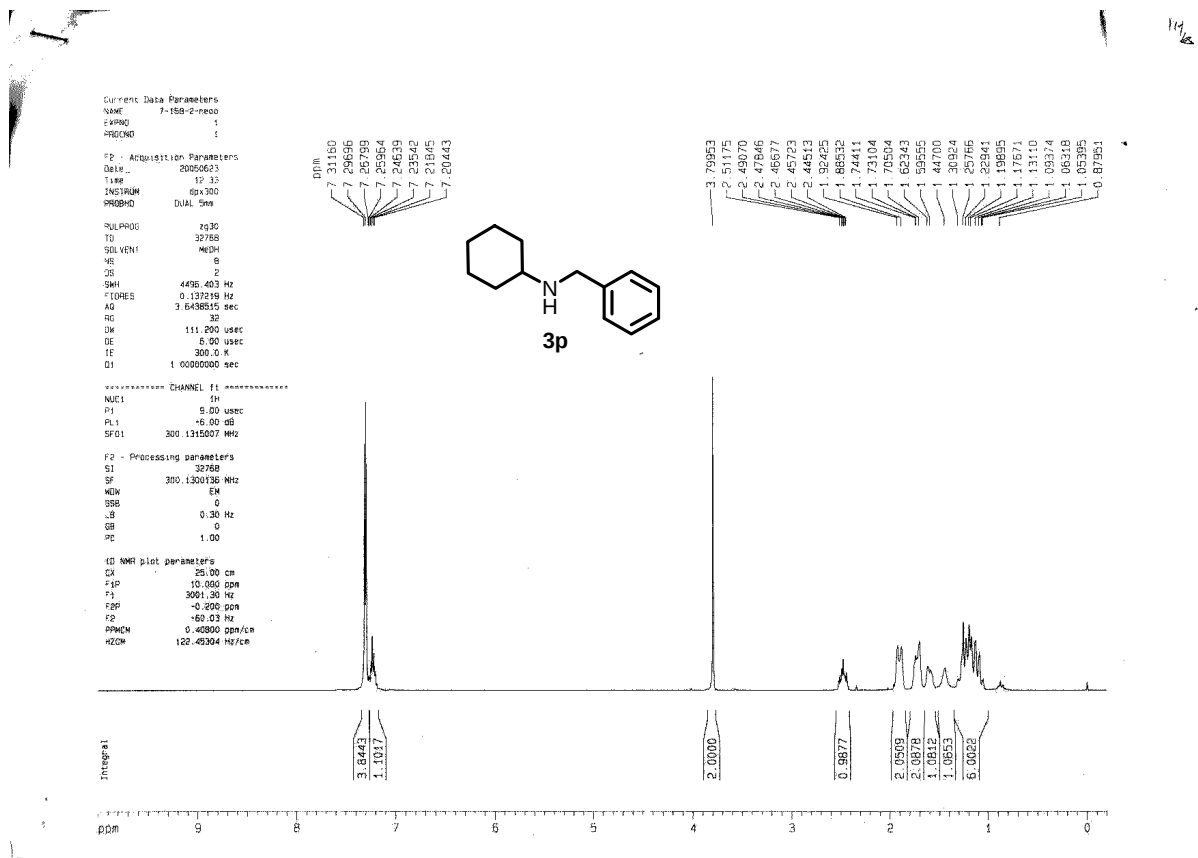
***** CHANNEL f1 *****
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SFO1 75.4760204 MHz

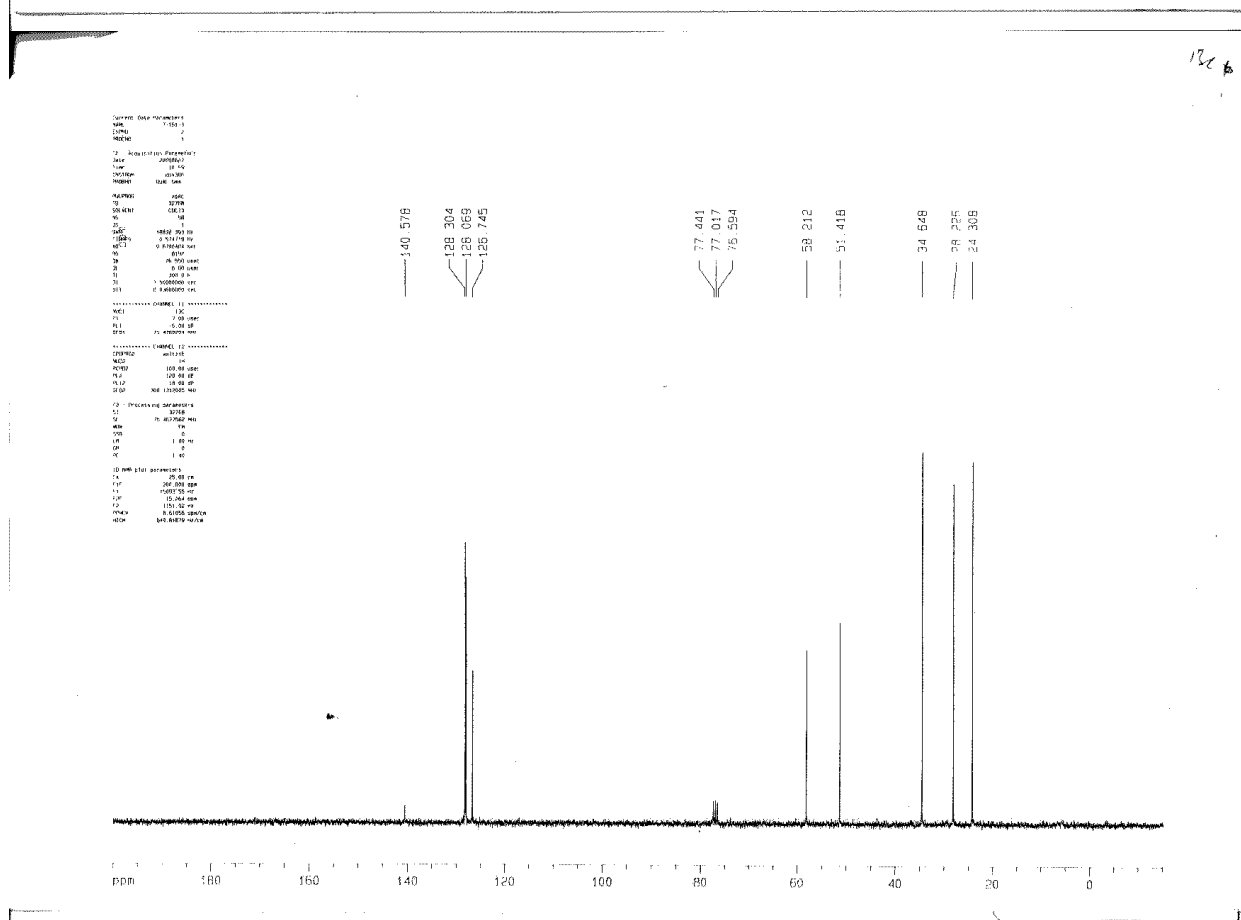
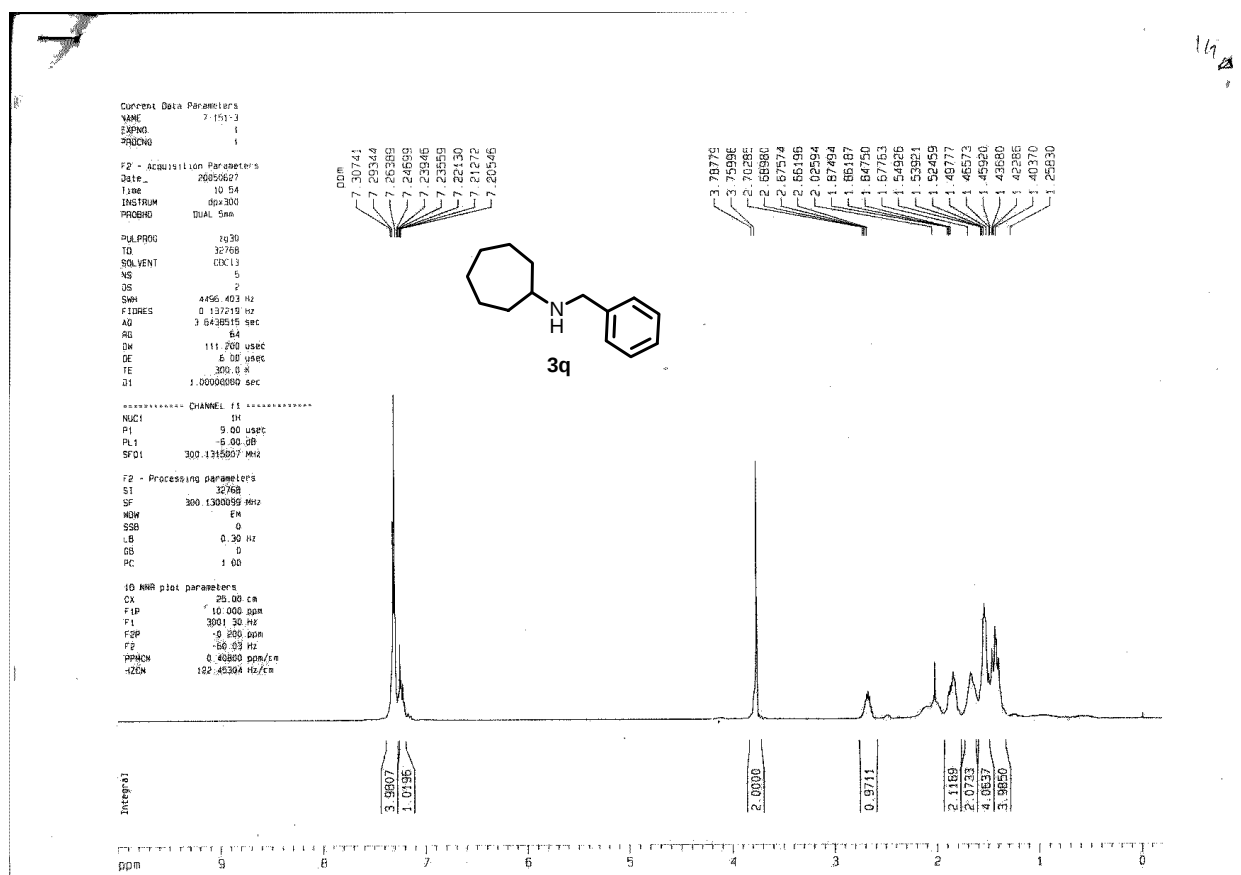
***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677294 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 25.00 cm
F1P 200.000 ppm
F1 15093.54 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PCMD 8.40000 ppm/cm
HZCM 633.92889 Hz/cm







Current Data Parameters
NAME 7-158-4
EXPNO 1
PROCNO 1

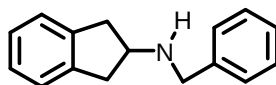
F2 - Acquisition Parameters
Date_ 20050623
Time 14.44
INSTRUM dpx300
PROBHD QNP 5mm

PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 4496.403 Hz
FIDRES 0.137219 Hz
AQ 3.6429615 sec
RG 645.1
DM 111.200 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

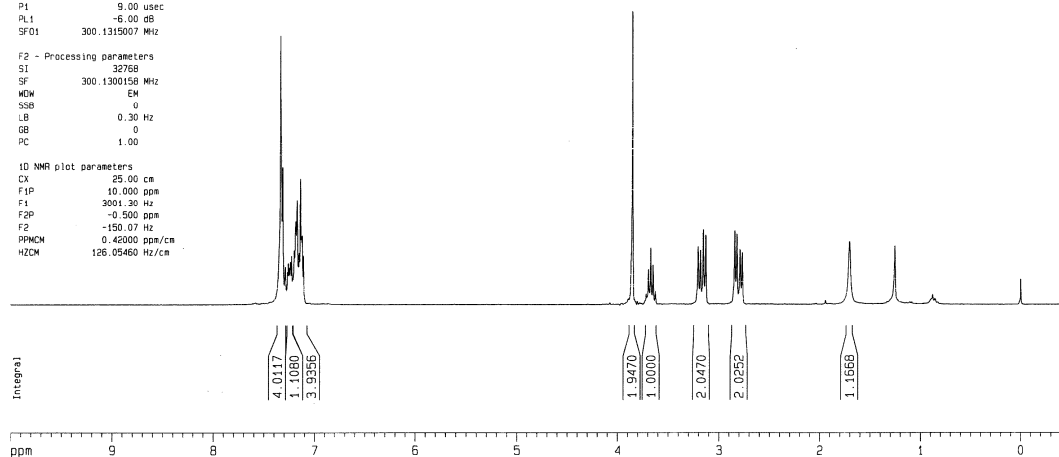
===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 300.1315007 MHz

F2 - Processing parameters
SI 32768
SF 300.1300158 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

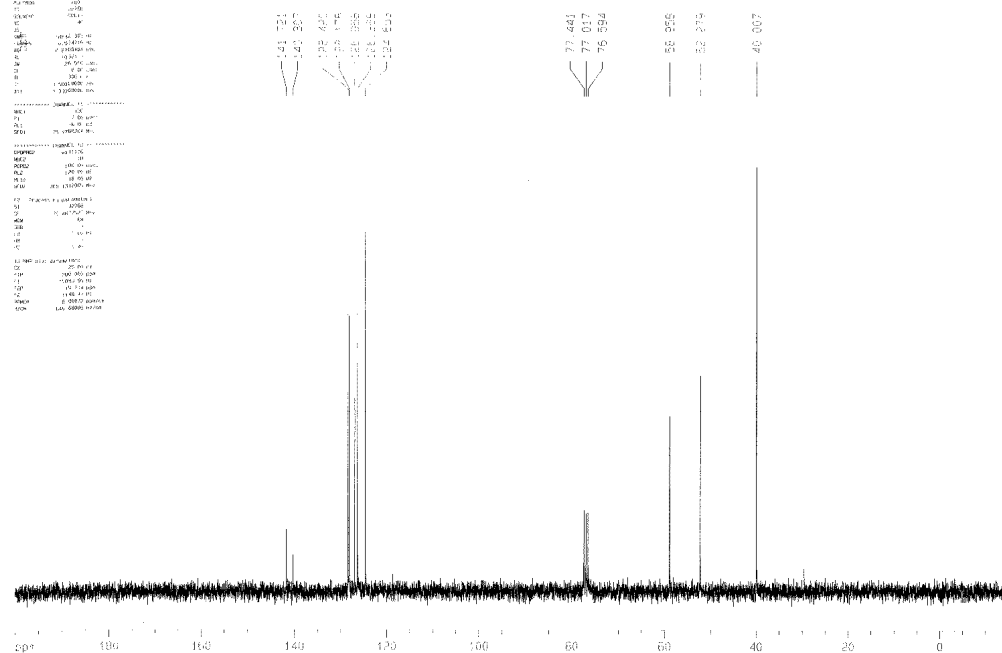
1D NMR plot parameters
CX 25.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.42000 ppm/cm
HZCM 126.05460 Hz/cm

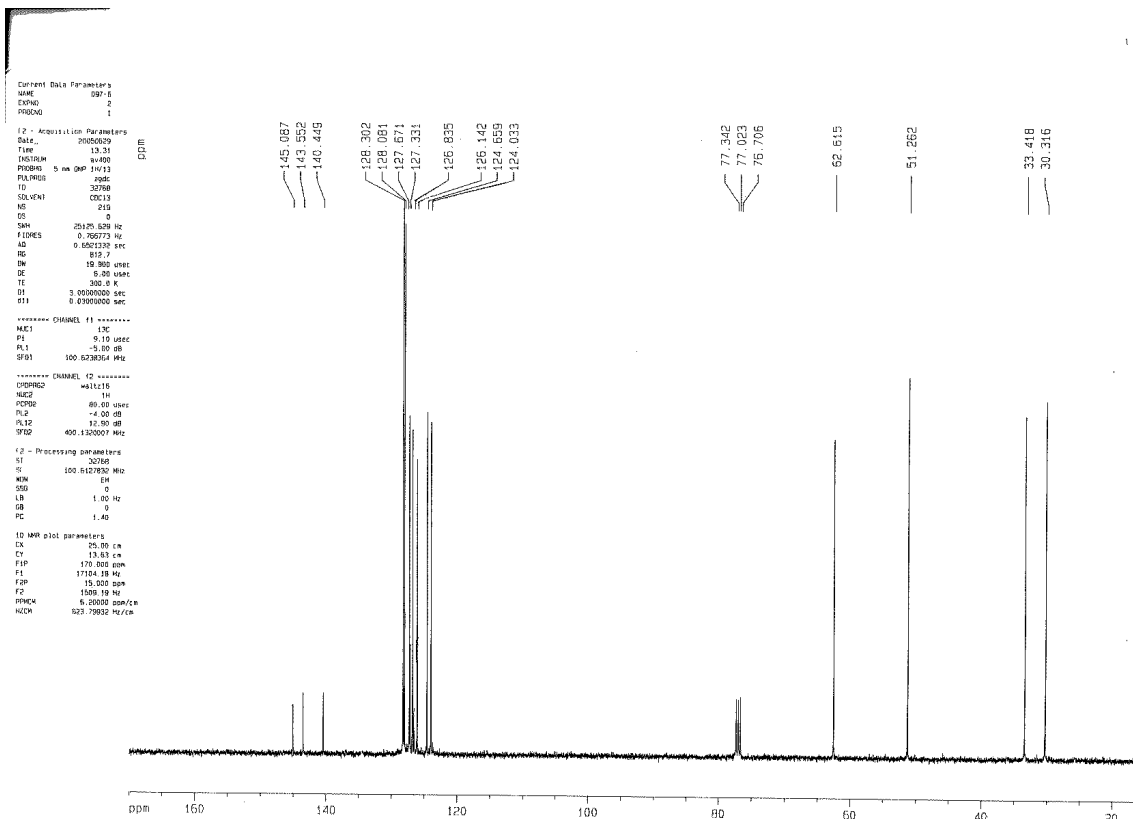
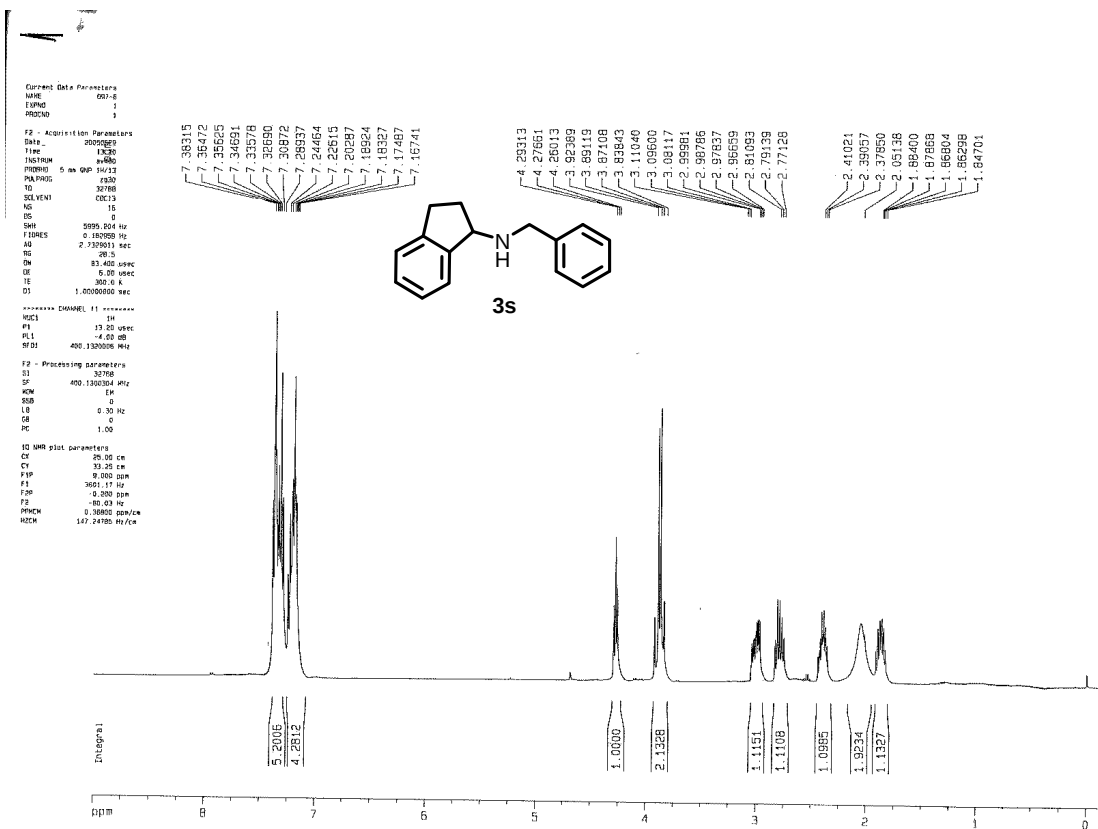


3r



Current Data Parameters
NAME 7-158-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20050623
Time 14.44
INSTRUM dpx300
PROBHD QNP 5mm
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 2
SWH 4496.403 Hz
FIDRES 0.137219 Hz
AQ 3.6429615 sec
RG 645.1
DM 111.200 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec
===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 300.1315007 MHz
F2 - Processing parameters
SI 32768
SF 300.1300158 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.42000 ppm/cm
HZCM 126.05460 Hz/cm





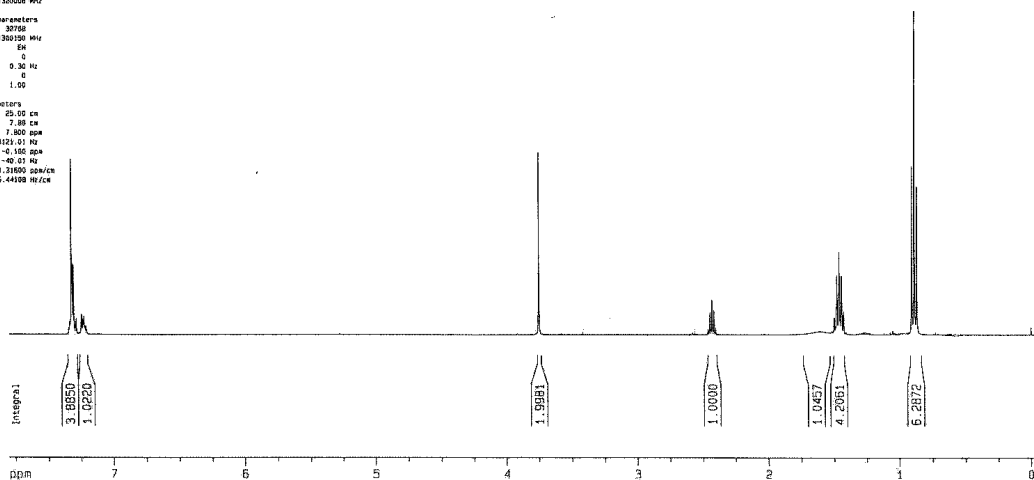
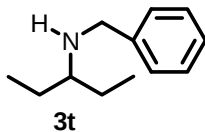
Current Data Parameters
NAME 087-7-pure
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050823
Time 17.36
INSTRUM av600
PROBHD 5 mm QNP 14133
PULPROG zgpg30
SOLVENT CDCl3
NS 350
DS 4
SWH 5885.254 Hz
FIDRES 0.192959 Hz
AQ 2.130011 sec
RG 35.9
OR 89.460 usec
DE 6.00 usec
TE 300.2 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 -4.00 dB
SFO1 400.130000 MHz

F2 - Processing parameters
SI 32768
SF 400.130000 MHz
WDW EN
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 25.00 cm
CY 7.00 cm
FIP 7.800 ppm
FI 3121.01 Hz
FOP -0.160 ppm
F2 -40.01 Hz
FNUC 0.31850 ppm/cx
HZCM 126.44108 Hz/cx



Current Data Parameters
NAME 087-7-pure
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050823
Time 14.28
INSTRUM mxt300
PROBHD DUAL 5mm

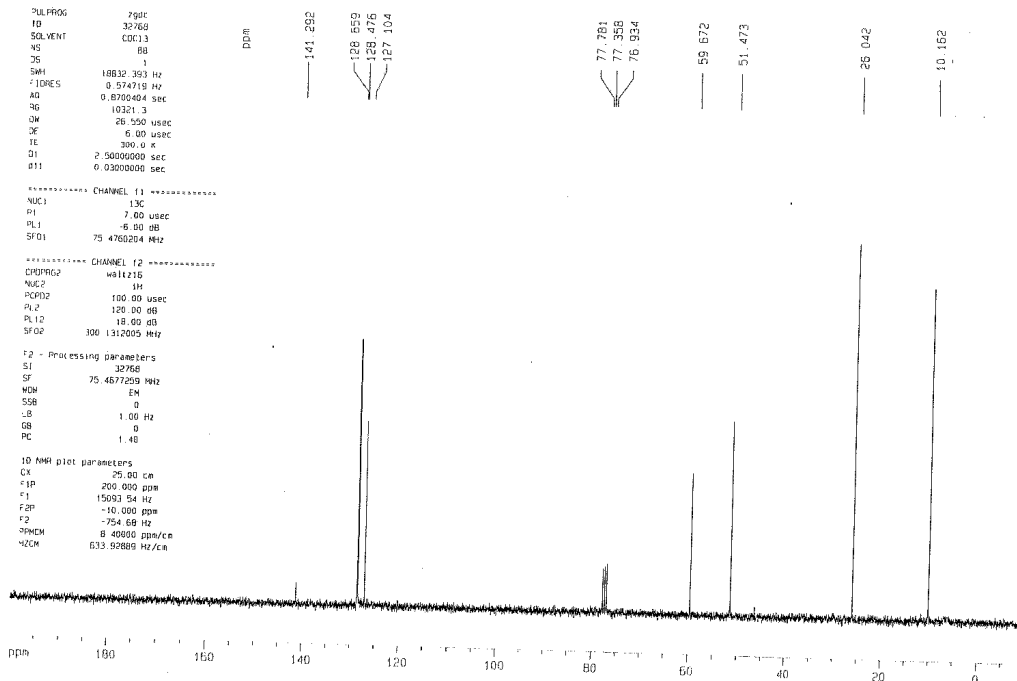
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 60
DS 1
SWH 18832.393 Hz
FIDRES 0.574718 Hz
AQ 0.8700404 sec
RG 10321.3
OR 26.550 usec
DE 6.00 usec
TE 300.0 K
D1 2.5000000 sec
D11 0.0300000 sec

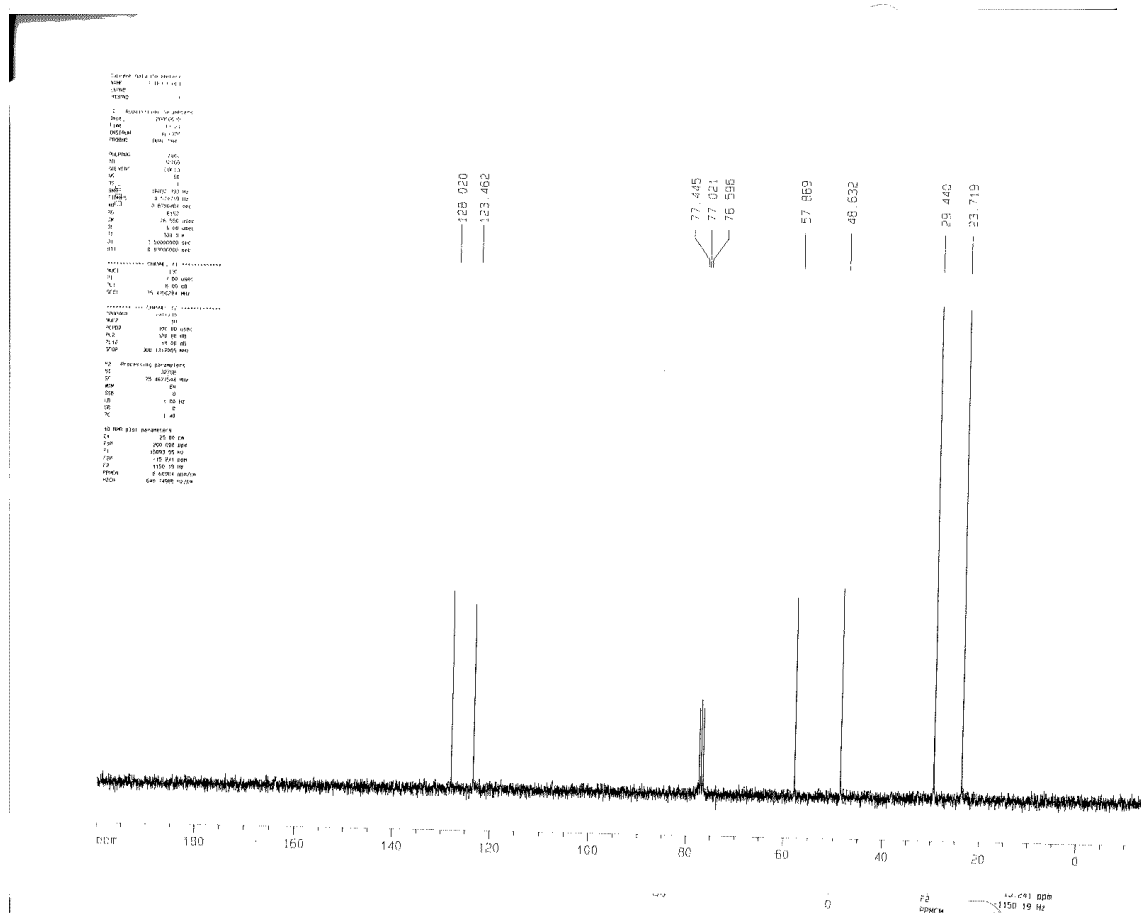
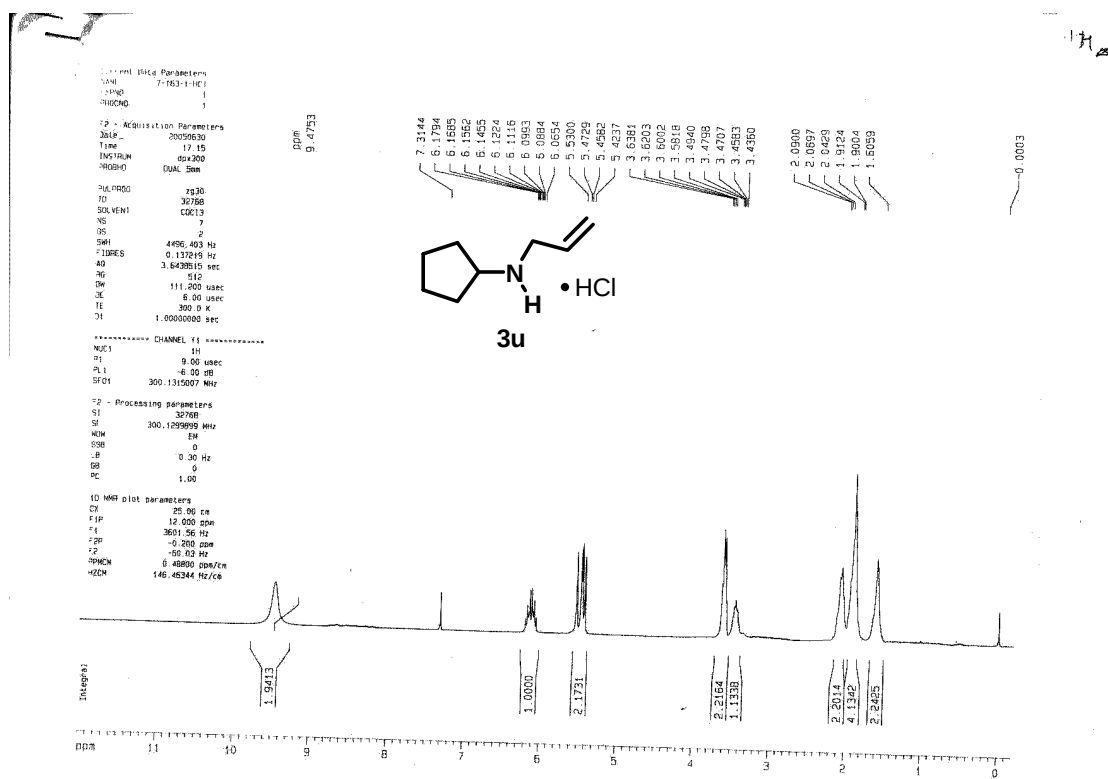
===== CHANNEL f1 =====
NUC1 13C
P1 7.00 usec
PL1 -6.00 dB
SFO1 75.476204 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4577259 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 25.00 cm
FIP 200.000 ppm
FI 15003.54 Hz
FOP -10.000 ppm
F2 -754.68 Hz
FNUC 9.40800 ppm/cx
HZCM 633.52885 Hz/cx





Current Data Parameters
NAME 168-2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090704
Time 17.14
INSTRUM dpx300
PROBHD QNP1 5mm

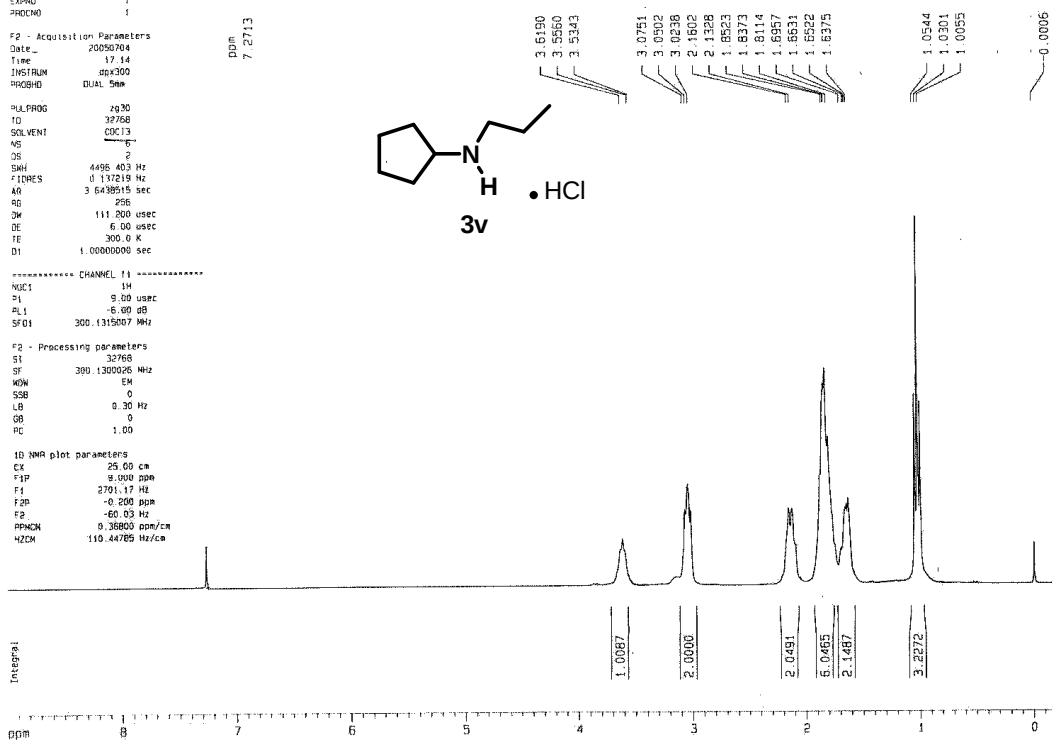
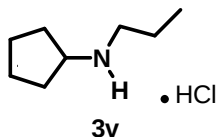
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 1
DS 2
SWH 4496.403 Hz
FIDRES 0.117219 Hz
AQ 3.6439515 sec
RG 256
DM 111.200 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

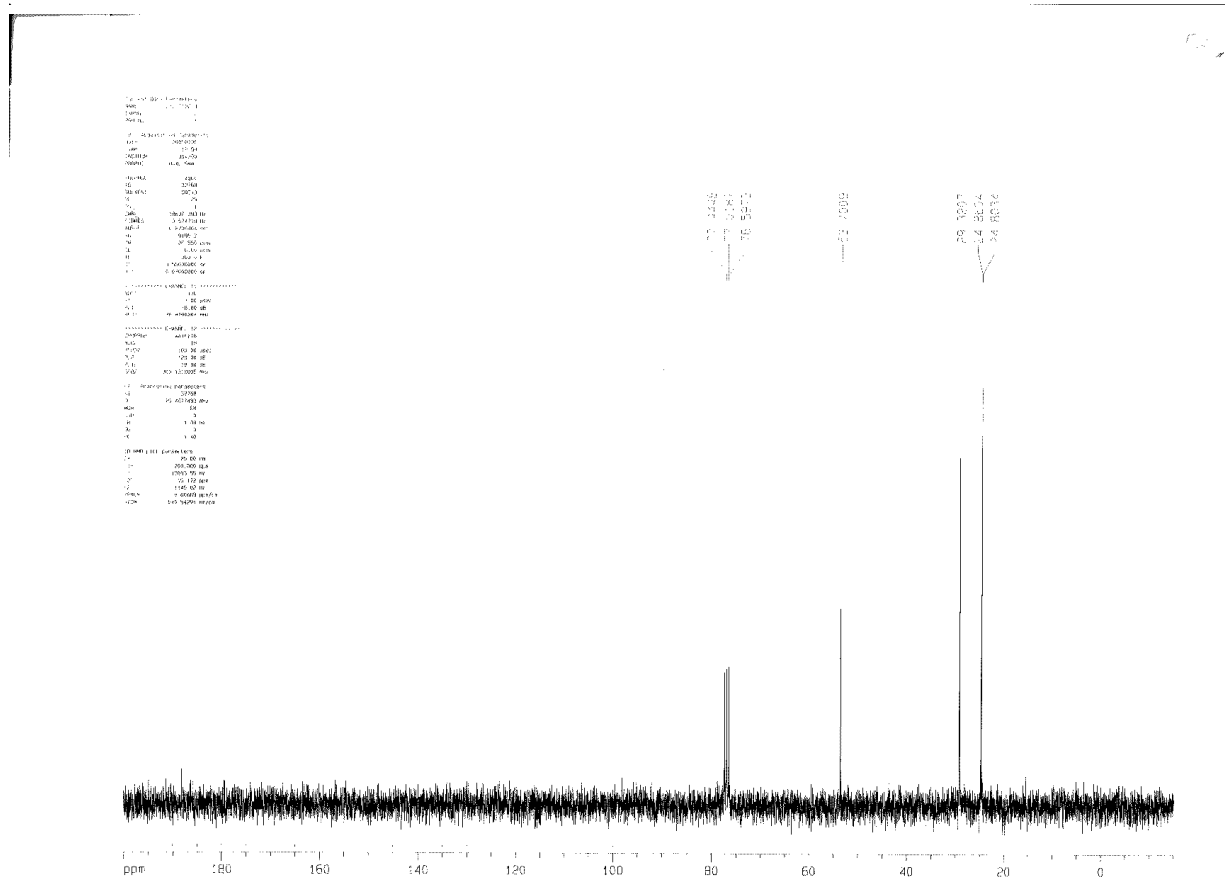
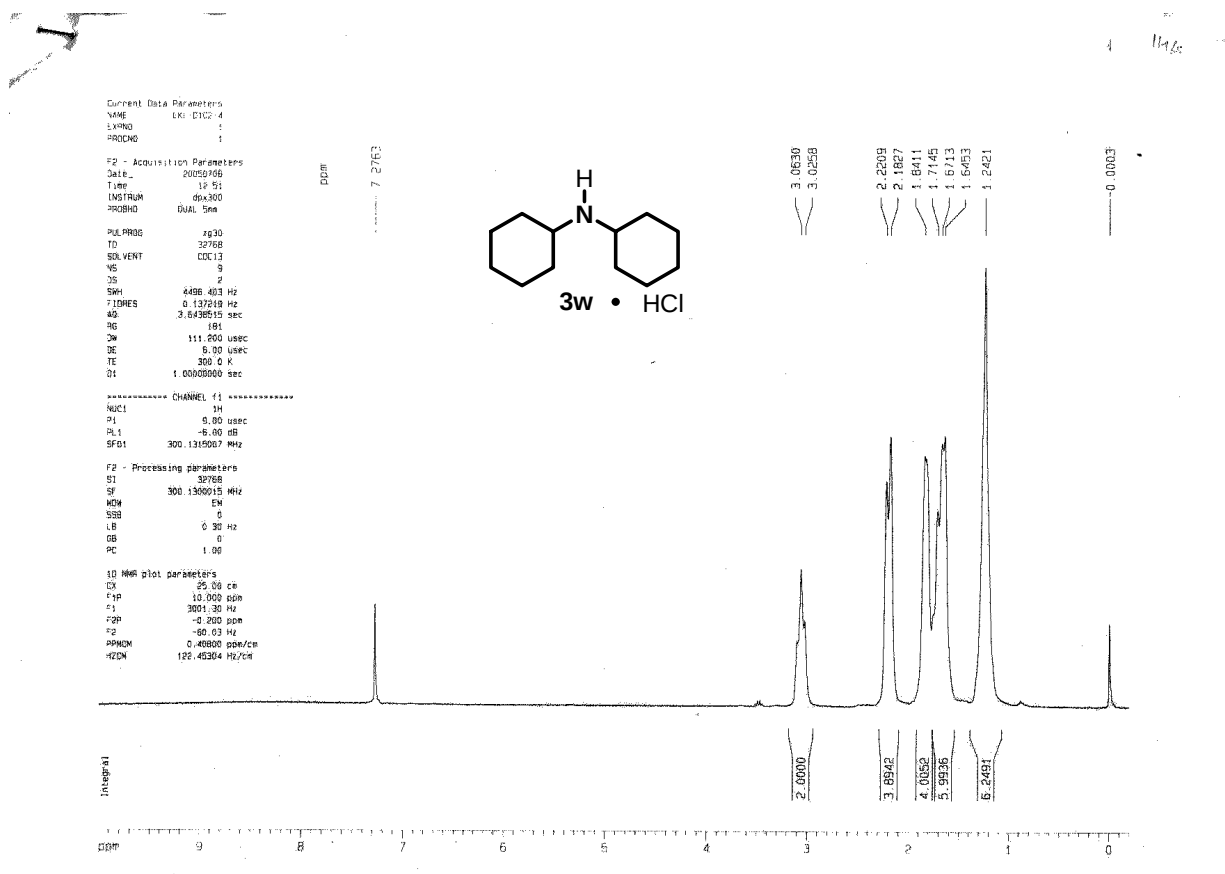
----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 300.1315067 MHz

F2 - Processing parameters
SI 32768
SF 300.1300026 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 25.00 cm
F1P 0.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPHCK 0.38800 ppm/cm
HZCM 110.44789 Hz/cm

DDM
7.2713





Current Data Parameters
NAME LAL-0142-3
EXPNO 1
PROCNO 1

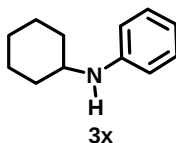
F2 - Acquisition Parameters
Date_ 20050701
Time 15:28
INSTRUM spect
PROBHD 5 mm BBO
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 4
DS 0
SWH 8022.820 Hz
FIDRES 0.244532 Hz
AQ 0.0467711 sec
RG 698.1
EN 62.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.20 usec
PL1 -4.00 dB
SFO1 400.132428 MHz

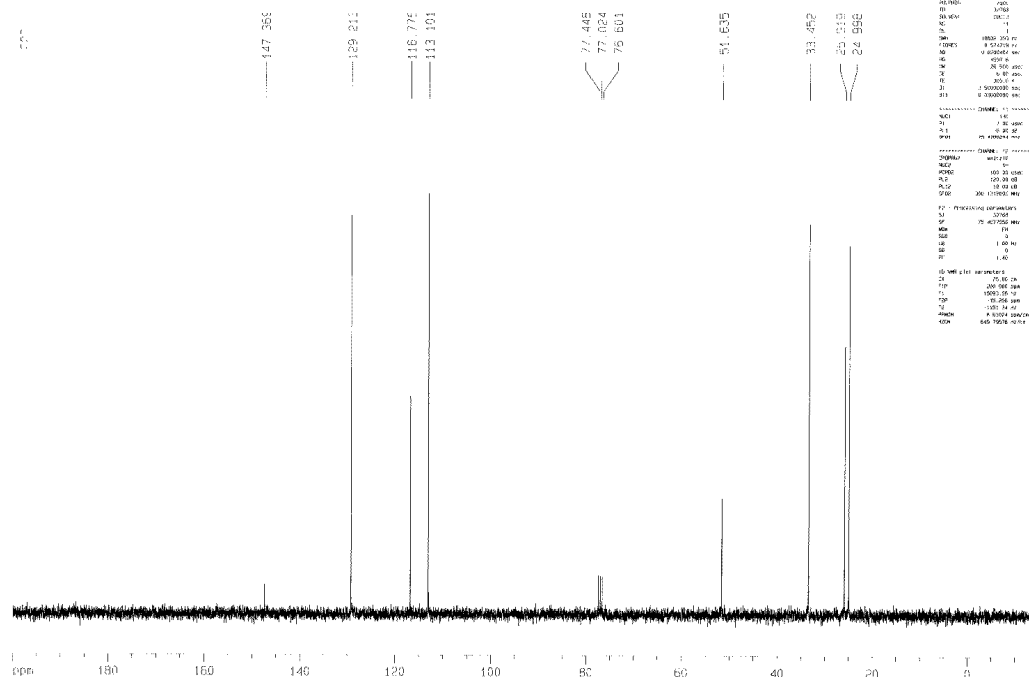
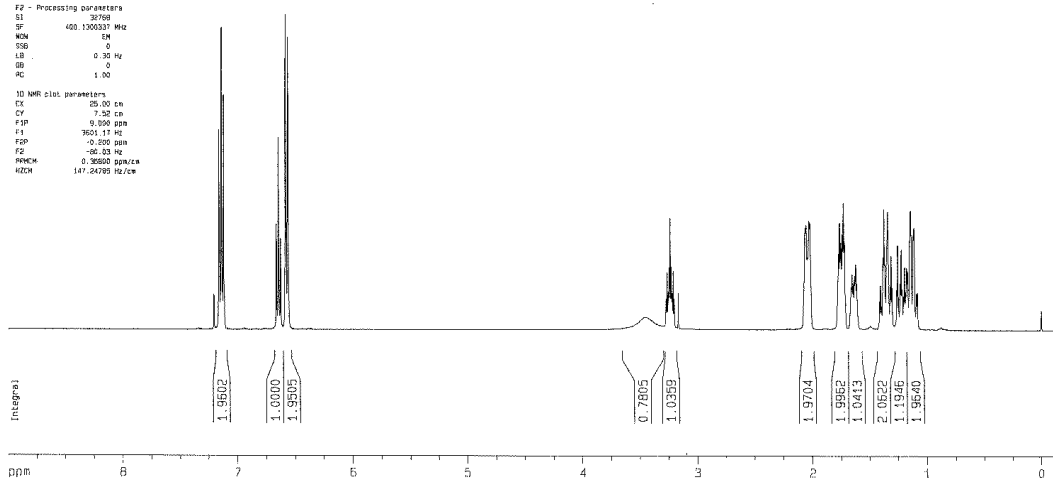
F2 - Processing parameters
SI 32768
SF 400.1300329 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
MC 1.50

1D NMR list parameters
CX 25.00 cm
CY 7.50 cm
FIR 5.300 gpm
F1 3601.11 Hz
F2 0.00000000 Hz
F3 -86.23 Hz
F4 0.39500000 gpm/Hz
F5 147.24750 Hz/cm

ppm
7.1573
7.1525
7.1389
7.1362
7.1221
7.1177
7.1124
6.6607
6.6424
6.6241
6.5788
6.5595



3.4423
3.2621
3.2524
3.2460
3.2365
3.2273
3.2207
3.2112
3.1671
2.0659
2.0549
2.0456
2.0307
2.0227
1.7764
1.7629
1.7530
1.7390
1.7293
1.7206
1.3814
1.3730
1.3651
1.3403
1.3102
1.2544
1.2243
1.1447
1.1193
1.1135
0.0003



Current Data Parameters
NAME LAL-0142-3
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050701
Time 15:45
INSTRUM spect
PROBHD 5 mm BBO
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 4
DS 0
SWH 8022.820 Hz
FIDRES 0.244532 Hz
AQ 0.0467711 sec
RG 698.1
EN 62.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 0.00 dB
SFO1 100.628150 MHz

F2 - Processing parameters
SI 32768
SF 100.628150 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
MC 1.50

1D NMR list parameters
CX 25.00 cm
CY 7.50 cm
FIR 5.300 gpm
F1 3601.11 Hz
F2 0.00000000 Hz
F3 -86.23 Hz
F4 0.39500000 gpm/Hz
F5 147.24750 Hz/cm

Current Data Parameters
NAME 7-103-11
F2PROG 1
PROCNO 1

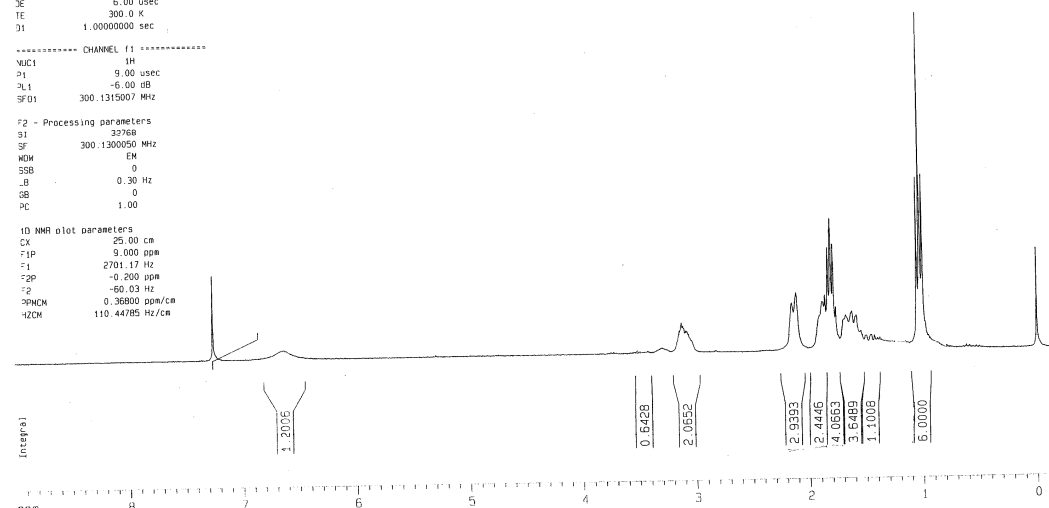
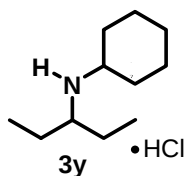
F2 - Acquisition Parameters
Date 20050704
Time 16:51
INSTRUM gpc300
PROBHD DUAL 5mm

PULPROG zg30
ID 32768
SOLVENT CDCl3
VS 7
JG 2
SMH 4496.403 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 256
JW 111.200 usec
ZC 6.00 usec
TE 300.0 K
SI 1.0000000 sec

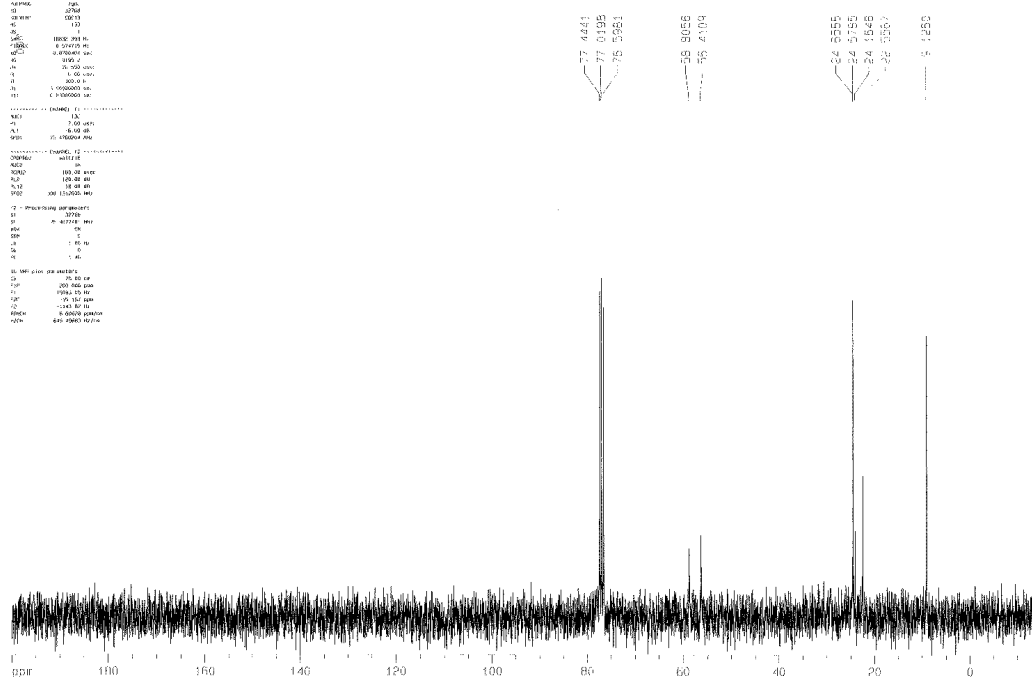
----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 300.1315007 MHz

F2 - Processing parameters
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 25.00 cm
C1P 9.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
SFOCM 0.36800 ppm/cm
HZCM 110.44785 Hz/cm



Current Data Parameters
NAME 7-103-11
F2PROG 1
PROCNO 1
Date 20050704
Time 16:51
INSTRUM gpc300
PROBHD DUAL 5mm
PULPROG zg30
ID 32768
SOLVENT CDCl3
VS 7
JG 2
SMH 4496.403 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 256
JW 111.200 usec
ZC 6.00 usec
TE 300.0 K
SI 1.0000000 sec
----- CHANNEL f1 -----
NUC1 1H
P1 9.00 usec
PL1 -6.00 dB
SFO1 300.1315007 MHz
F2 - Processing parameters
SI 32768
SF 300.1300050 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
C1P 9.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
SFOCM 0.36800 ppm/cm
HZCM 110.44785 Hz/cm



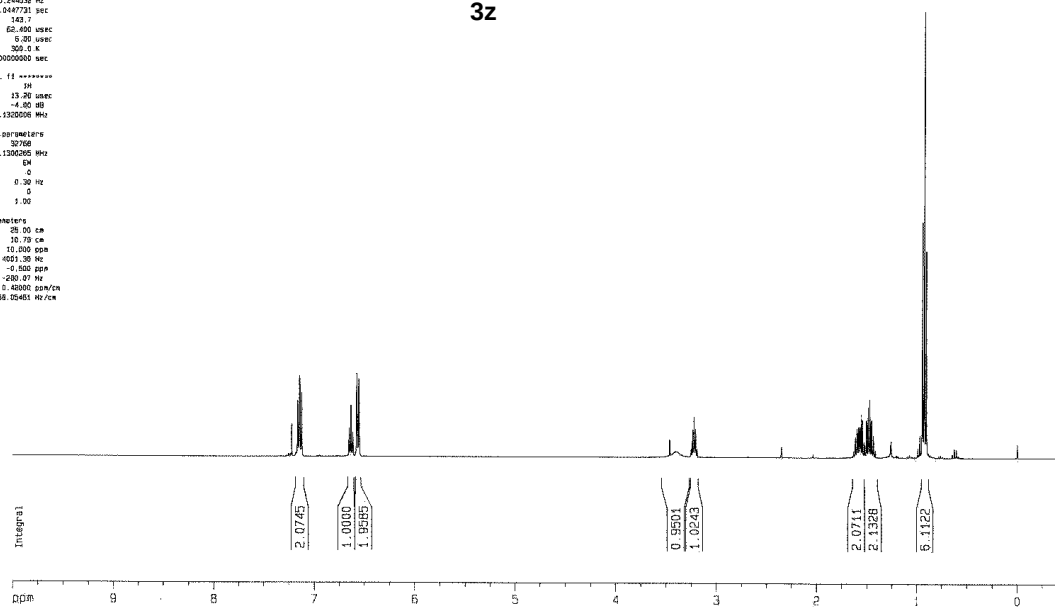
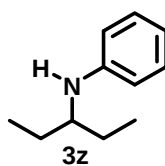
Current Data Parameters
NAME D102-10
EXPNO 1
PROCNO 1

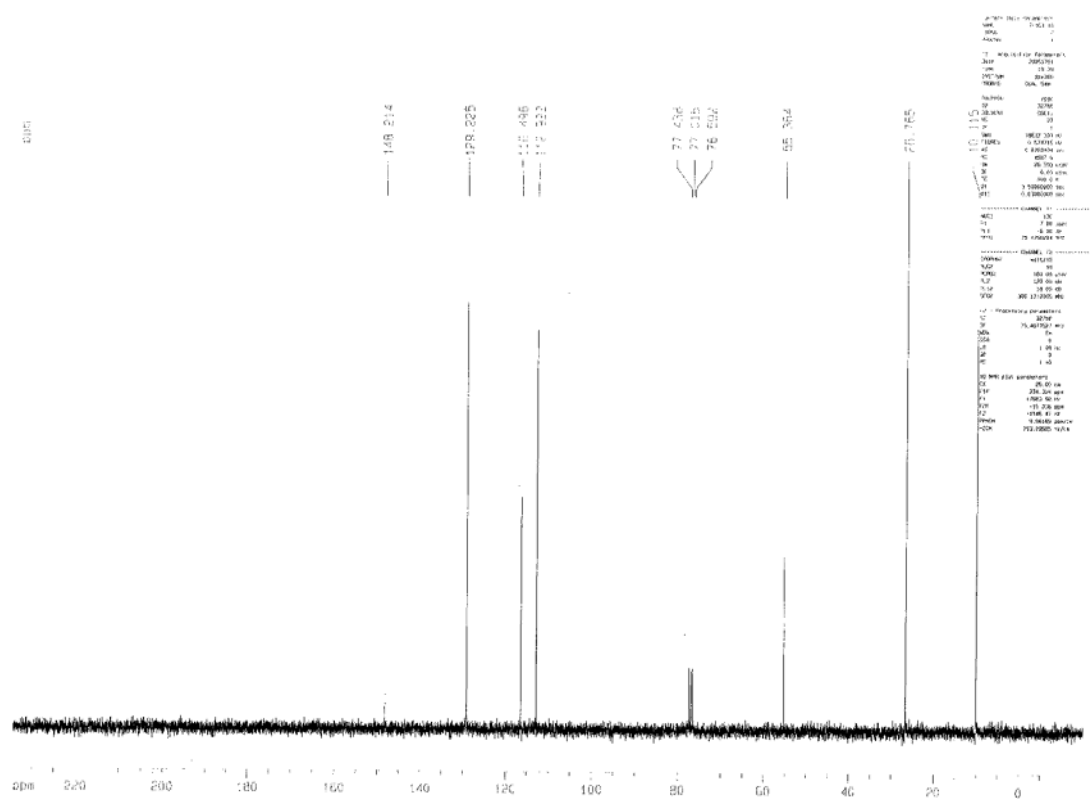
F2 - Acquisition Parameters
Date_ 20050830
Time 1830
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 16
DS 4
SWH 8012.820 Hz
FIDRES 0.244032 Hz
AQ 2.2447731 sec
RG 142.7
DA 62.400 usec
DE 9.20 usec
TE 300.2 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.20 usec
PL1 -4.00 dB
SFO1 400.1300000 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

13 NMR list parameters
CX 25.00 cm
CY 10.73 cm
F1P 10.000 um
F1 4001.30 Hz
F2P -0.000 ppm
F2 -280.07 Hz
PNUC1 0.42000 ppm/CN
H2CN 156.25401 Hz/CN



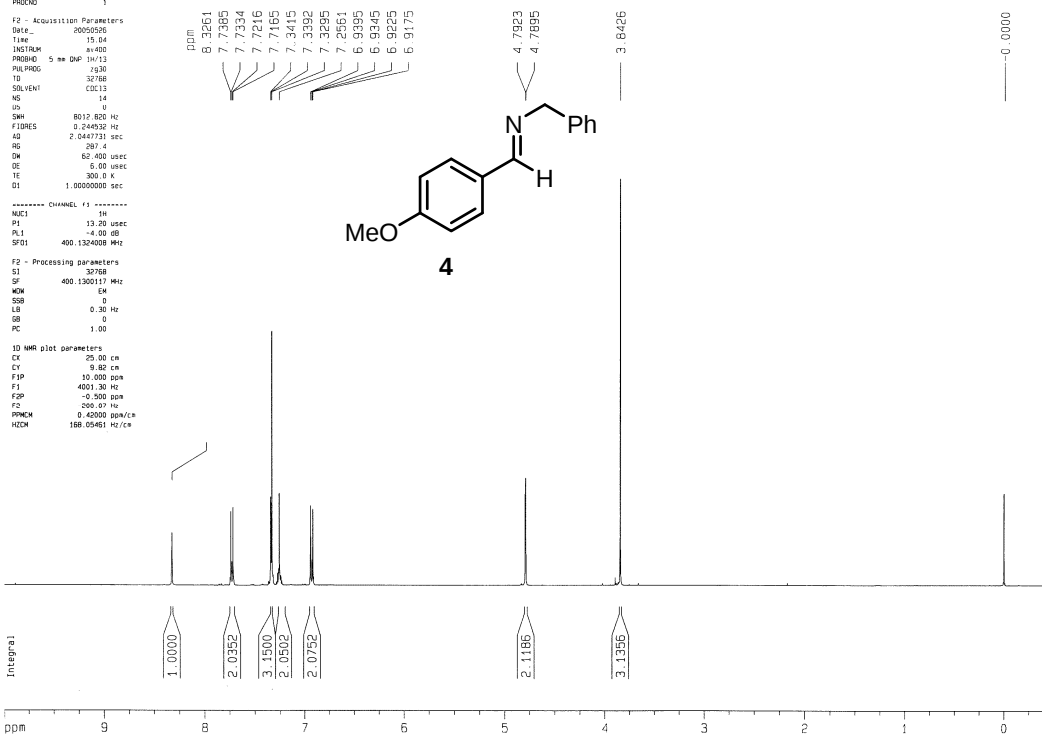


Current Data Parameters
 NAME 7-121-crude
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20050506
 Time 15.04
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TO 32768
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 8012.820 Hz
 FIDRES 0.244020 Hz
 AQ 2.0447731 sec
 RG 267.4
 CW 662.400 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.0000000 sec

***** CHANNEL f3 *****
 NUC1 13C
 P1 13.00 usec
 PL1 -4.00 dB
 SFO1 400.1324008 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300117 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 25.00 cm
 CY 9.80 cm
 FIP 10.000 ppm
 F1 4001.30 Hz
 F2P -5.300 ppm
 F2 200.07 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 168.05461 Hz/cm



Current Data Parameters
 NAME 7-121-crude
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20050506
 Time 15.07
 INSTRUM av400
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TO 32768
 SOLVENT CDCl3
 NS 115
 DS 4
 SWH 25125.620 Hz
 FIDRES 0.768773 Hz
 AQ 0.021132 sec
 RG 1024
 CW 19.900 usec
 DE 6.00 usec
 TE 300.2 K
 D1 3.5000000 sec
 D11 0.0300000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 9.10 usec
 PL1 -5.00 dB
 SFO1 100.628364 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -4.00 dB
 PL12 12.00 dB
 SFO2 400.1300007 MHz

F2 - Processing parameters
 SI 32768
 SF 100.617350 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 25.00 cm
 CY 11.31 cm
 FIP 200.000 ppm
 F1 20122.55 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 PPMCM 8.40000 ppm/cm
 HZCM 845.14697 Hz/cm

