Simple and Direct sp^3 C-H Bond Arylation of Tetrahydroisoquinolines and Isochromans via DDQ Oxidation under Mild Conditions

Supporting Information

Wataru Muramatsu,[†]* Kimihiro Nakano[†] and Chao-Jun Li[‡]*

[†] Graduate School of biomedical Sciences, Nagasaki University, 1-14 Bunkyo-machi, Nagasaki, Nagasaki 852-8521, Japan

muramatu@nagasaki-u.ac.jp

[‡] Department of Chemistry, McGill University, 801 Sherbrooke Street West, Montreal, QC H3A 0B8, Canada

cj.li@mcgill.ca

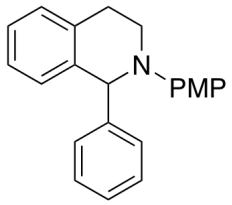
Contents

1. General

NMR spectra were recorded on a JEOL JNM-AL400 spectrometer operating at 400 MHz and 100 MHz for ^{1}H and ^{13}C acquisitions, respectively. Chemical shifts are reported in ppm with a solvent resonance as an internal standard (^{1}H-NMR; tetramethylsilane or chloroform as internal standards, indicating 0 or 7.26, respectively, ^{13}C-NMR; chloroform as internal standard, indicating 77.00). Data is reported as follows: s = singlet, br = broad, d = doublet, t = triplet, q = quartet, m = multiplet; coupling constants in Hz; integration. IR spectra were recorded with a SHIMADZU IRAffinity-1 spectrometer. Specific rotation was measured with a JASCO DIP-1000 digital polarimeter. MS spectra were recorded with a JEOL JMS-700N mass spectrometer with fast atom bombardment (FAB) and a double-focusing magnetic sector mass analyzer for MS and HRMS measurements. TLC analysis was performed on commercial glass plates bearing a 0.25 mm layer of Merck TLC Silica gel 60 F_{254}. Silica gel chromatography was carried out Merck Silica gel 60 (70-230 mesh).

2. Experimental details and characterization data for all compounds

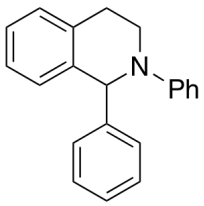
1-Phenyl-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (1)[1]



After stirring the mixture of 2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (95.7 mg, 0.4 mmol) and DDQ (99.9 mg, 0.44 mmol) in chlorobenzene (4.0 mL) in a vial at room temperature for 10 min under Ar atmosphere, phenylmagnesium bromide (1.0 M in THF, 8.0 mL, 0.80 mmol) was added to the suspension at 0 °C. After stirring vigorously for 3 hours at 0 °C, the reaction mixture was quenched with saturated aqueous $NaHCO_3$, and extracted with ethyl acetate. The organic layer was washed with water and brine, dried over $MgSO_4$, filtrated, and concentrated in *vacuo*. The residue was purified by SiO_2 column chromatography (*n*-hexane/ether = 100/0-100/5) to give 1-phenyl-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline **1** (120.2 mg, 95%) as pale yellow solid.

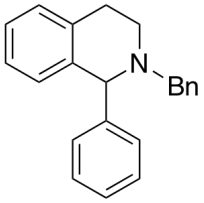
120.2 mg (95%). Pale yellow solid. R_f = 0.29 (AcOEt/*n*-Hexane, 1 : 10). M.p. 128-129 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.10 (m, 9H), 6.90-6.75 (m, 4H), 6.80 (s, 1H), 3.74 (s, 3H), 3.65-3.53 (m, 1H), 3.50-3.35 (m, 1H), 3.05-2.85 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.9, 144.4, 143.2, 137.7, 135.5, 128.4, 128.1, 128.1, 128.0 (2C), 126.8, 126.7 (2C), 126.0, 117.7 (2C), 114.5 (2C), 64.3, 55.6, 44.5, 28.1. IR (solid) 3022, 2918, 2822, 1606, 1506, 1450, 1032, 826 cm^{-1}. MS (FAB) *m/z* (rel intensity) 316 ($M+H^+$, 25), 315 (45), 307 (25), 289 (15), 238 (20), 154 (100), 136 (70), 107 (20). HRMS (FAB) Calcd for $C_{22}H_{22}NO$ ($M+H^+$): 316.1696, found, 316.1726. Anal Calcd for $C_{22}H_{21}NO$: C, 83.78; H, 6.71. Found: C, 83.70; H, 7.00.

1-Phenyl-2-phenyl-1,2,3,4-tetrahydroisoquinoline (2)[1-2]



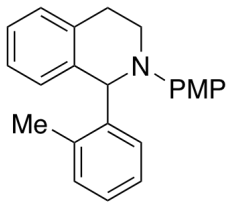
98.3 mg (86%). White solid. R_f = 0.27 (Et_2O/*n*-Hexane, 1 : 20). M.p. 62-64 °C (lit.[2] M.p. 55-56 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.10 (m, 11H), 6.85 (d, *J* = 8.8 Hz, 2H), 6.75 (t, *J* = 7.3 Hz, 1H), 5.83 (s, 1H), 3.80-3.67 (m, 1H), 3.55-3.45 (m, 1H), 3.00-2.85 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.5, 143.1, 137.9, 135.7, 129.1 (3C), 128.2 (2C), 128.1, 127.8, 127.3 (2C), 127.0, 126.8, 126.1, 117.4, 113.8, 62.8, 43.8, 28.0. IR (solid) 3020, 2916, 2853, 1593, 1503, 1381, 1250, 746 cm^{-1}. MS (FAB) *m/z* (rel intensity) 286 (M+H$^+$, 40), 285 (60), 208 (45), 154 (100), 136 (65), 107 (15). HRMS (FAB) Calcd for $C_{21}H_{20}N$ (M+H$^+$): 286.1590, found, 286.1574.

1-Benzyl-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3)



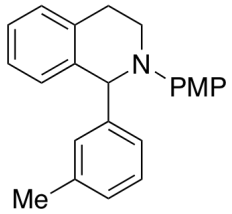
82.6 mg (69%). White solid. R_f = 0.48 (AcOEt/*n*-Hexane, 1 : 10). M.p. 106-109 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50-7.17 (m, 10H), 7.17-7.05 (m, 2H), 6.99 (t, *J* = 7.8 Hz, 1H), 6.72 (d, *J* = 7.8 Hz, 1H), 4.60 (s, 1H), 3.81 (d, *J* = 13.6 Hz, 1H), 3.24 (d, *J* = 13.6 Hz, 1H), 3.15-3.00 (m, 2H), 2.82-2.70 (m, 1H), 2.60-2.45 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.4, 139.6, 138.5, 134.8, 129.6 (2C), 128.8, 128.7 (2C), 128.4, 128.3 (2C), 128.1 (2C), 127.2, 126.8, 125.9, 125.6, 68.8, 58.8, 47.3, 29.2. IR (solid) 3027, 2930, 2808, 1598, 1489, 1450, 1256, 1123 cm^{-1}. MS (FAB) *m/z* (rel intensity) 300 (M+H$^+$, 40), 298 (60), 222 (95), 154 (30), 136 (20), 91 (100). HRMS (FAB) Calcd for $C_{22}H_{22}N$ (M+H$^+$): 300.1747, found, 300.1791.

1-(2-Tolyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (4)



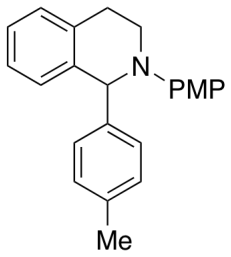
116.3 mg (88%). White solid. R_f = 0.40 (EtOAc/*n*-Hexane, 1 : 10). M.p. 124-126 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.05 (m, 5H), 7.05-6.97 (m, 1H), 6.97-6.90 (m, 1H), 6.92 (d, *J* = 8.9 Hz, 2H), 6.80-6.70 (m, 1H), 6.74 (d, *J* = 8.9 Hz, 2H), 5.65 (s, 1H), 3.72 (s, 3H), 3.52-3.35 (m, 2H), 2.87 (ddd, *J* = 16.4, 8.8, 5.9 Hz, 1H), 2.76 (dt, *J* = 16.4, 4.4 Hz, 1H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.1, 144.7, 142.2, 137.7, 136.9, 135.9, 130.5, 130.1, 128.9, 128.5, 126.9, 126.3, 125.9, 125.2, 121.3 (2C), 114.2 (2C), 61.5, 55.4, 46.0, 26.3, 19.6. IR (solid) 3015, 2928, 2833, 1580, 1504, 1240, 1194, 1128 cm^{-1}. MS (FAB) *m/z* (rel intensity) 330 ($M+H^+$, 45), 329 (100), 238 (60), 154 (25), 136 (20). HRMS (FAB) Calcd for $C_{23}H_{24}NO$ ($M+H^+$): 330.1852, found, 330.1860. Anal Calcd for $C_{23}H_{23}NO$: C, 83.85; H, 7.04. Found: C, 83.60; H, 7.38.

1-(3-Tolyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (5)



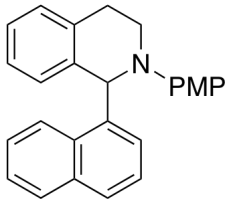
113.0 mg (86%). Colorless viscous oil. R_f = 0.39 (EtOAc/*n*-Hexane, 1 : 9). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22-7.05 (m, 5H), 7.05-6.90 (m, 3H), 6.85-6.75 (m, 4H), 5.62 (s, 1H), 3.74 (s, 3H), 3.59 (ddd, *J* = 11.7, 6.3, 5.6 Hz, 1H), 3.41 (ddd, *J* = 11.7, 6.8, 5.4 Hz, 1H), 3.05-2.85 (m, 2H), 2.25 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.7, 144.4, 143.4, 137.7, 137.6, 135.5, 128.6, 128.3, 128.1, 127.9, 127.6, 126.6, 126.0, 125.1, 117.4 (2C), 114.5 (2C), 64.2, 55.6, 44.5, 28.0, 21.5. IR (neat) 3020, 2907, 2830, 1603, 1508, 1240, 1180, 1036 cm^{-1}. MS (FAB) *m/z* (rel intensity) 330 ($M+H^+$, 55), 329 (100), 238 (65), 136 (10), 105 (5). HRMS (FAB) Calcd for $C_{23}H_{24}NO$ ($M+H^+$): 330.1852, found, 330.1819. Anal Calcd for $C_{23}H_{23}NO$: C, 83.85; H, 7.04. Found: C, 84.15; H, 7.13.

1-(4-Tolyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (6)



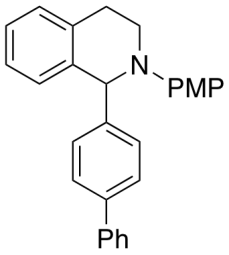
126.0 mg (96%). Colorless viscous oil. R_f = 0.45 (Et_2O/*n*-Hexane, 1 : 10). ^1H NMR (400 MHz, $CDCl_3$) δ 7.22-7.10 (m, 4H), 7.02 (s, 4H), 6.85-6.75 (m, 4H), 5.64 (s, 1H), 3.74 (s, 3H), 3.57 (ddd, *J* = 11.7, 6.6, 5.4 Hz, 1H), 3.40 (ddd, *J* = 11.7, 6.6, 5.4 Hz, 1H), 3.05-2.85 (m, 2H), 2.27 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8, 144.5, 140.2, 137.8, 136.3, 135.4, 128.7 (2C), 128.3, 128.1, 128.0, 126.6, 125.9 (2C), 117.7 (2C), 114.4 (2C), 64.0, 55.6, 44.4, 28.1, 21.0. IR (neat) 3022, 2926, 2841, 1665, 1510, 1258, 1175, 1130 cm^{-1}. MS (FAB) *m/z* (rel intensity) 330 ($M+H^+$, 65), 329 (100), 238 (75), 147 (20), 73 (50). HRMS (FAB) Calcd for $C_{23}H_{24}NO$ ($M+H^+$): 330.1852, found, 330.1845.

1-(1-Naphthyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (7)[1]



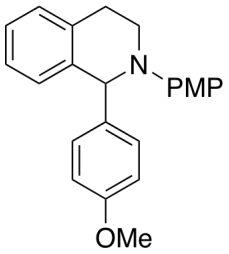
87.9 mg (60%). White solid. R_f = 0.31 (AcOEt/*n*-Hexane, 1 : 10). M.p. 172-174 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 8.11 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.84 (dd, *J* = 6.8, 2.4 Hz, 1H), 7.72 (d, *J* = 8.1 Hz, 1H), 7.50-7.35 (m, 2H), 7.30-7.10 (m, 5H), 7.02 (d, *J* = 8.1 Hz, 1H), 6.99 (d, *J* = 9.0 Hz, 2H), 6.76 (d, *J* = 9.0 Hz, 2H), 6.23 (s, 1H), 3.74 (s, 3H), 3.60-3.40 (m, 2H), 2.91 (dt, *J* = 16.6, 8.3 Hz, 1H), 2.76 (dt, *J* = 16.6, 3.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.0, 144.3, 139.9, 136.7, 136.1, 134.0, 132.3, 129.0, 128.7, 128.6, 128.2, 128.0, 126.6, 126.1, 126.0, 125.5, 124.8, 124.7, 120.9 (2C), 114.4 (2C), 60.8, 55.4, 45.9, 25.8. IR (solid) 3046, 2914, 2830, 1595, 1510, 1506, 1238, 1032 cm^{-1}. MS (FAB) *m/z* (rel intensity) 366 ($M+H^+$, 10), 365 (M^+, 20), 307 (25), 154 (100), 136 (70). HRMS (FAB) Calcd for $C_{26}H_{24}NO$ ($M+H^+$): 366.1852, found, 366.1800.

1-(4-Biphenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (8)[1]



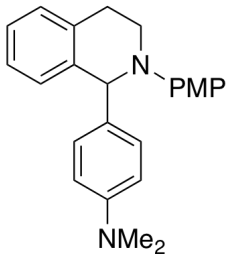
145.6 mg (93%). White solid. R_f = 0.31 (EtOAc/*n*-Hexane, 1 : 10). M.p. 130-132 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.4, 1.2 Hz, 2H), 7.50-7.35 (m, 4H), 7.35-7.28 (m, 1H), 7.28-7.15 (m, 6H), 6.90-6.78 (m, 4H), 5.71 (s, 1H), 3.75 (s, 3H), 3.62 (ddd, *J* = 11.7, 6.3, 5.4 Hz, 1H), 3.44 (ddd, *J* = 11.7, 6.8, 5.4 Hz, 1H), 3.10-2.90 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.9, 144.4, 142.3, 140.8, 139.6, 137.6, 135.5, 128.7 (2C), 128.5 (2C), 128.4, 128.1, 127.1, 127.0 (2C), 126.8, 126.7 (2C), 126.0, 117.7 (2C), 114.5 (2C), 64.1, 55.6, 44.5, 28.1. IR (solid) 3032, 2922, 2851, 1600, 1504, 1246, 1196, 1132 cm^{-1}. MS (FAB) *m/z* (rel intensity) 392 ($M+H^+$, 25), 391 (45), 238 (25), 154 (100), 136 (70). HRMS (FAB) Calcd for $C_{28}H_{26}NO$ ($M+H^+$): 392.2009, found, 392.1968.

1-(4-Methoxyphenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (9)[1]



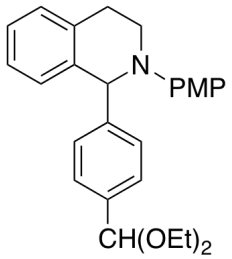
101.3 mg (73%). Colorless viscous oil. R_f = 0.24 (EtOAc/*n*-Hexane, 1 : 10). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.05 (m, 4H), 7.02 (d, *J* = 8.5 Hz, 2H), 6.85-6.74 (m, 4H), 6.72 (d, *J* = 8.5 Hz, 2H), 5.61 (s, 1H), 3.70 (s, 3H), 3.69 (s, 3H), 3.51 (ddd, *J* = 12.0, 6.8, 5.1 Hz, 1H), 3.36 (ddd, *J* = 12.0, 6.6, 5.4 Hz, 1H), 3.00-2.83 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.4, 152.9, 144.5, 137.8, 135.3, 135.2, 129.2 (2C), 128.4, 128.0, 126.5, 125.8, 118.2 (2C), 114.4 (2C), 113.2 (2C), 63.9, 55.5, 55.1, 44.3, 28.1. IR (neat) 2995, 2930, 2832, 1607, 1504, 1238, 1169, 1032 cm^{-1}. MS (FAB) *m/z* (rel intensity) 346 ($M+H^+$, 65), 345 (100), 344 (40), 238 (90), 136 (10). HRMS (FAB) Calcd for $C_{23}H_{24}NO_2$ ($M+H^+$): 346.1802, found, 346.1802.

1-(4-*N*,*N*-dimethylaminoaniline)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (10)



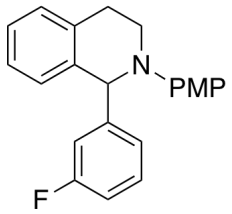
126.0 mg (88%). Pale yellow viscous oil. R_f = 0.14 (EtOAc/*n*-Hexane, 1 : 10). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.05 (m, 4H), 6.96 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 9.0 Hz, 2H), 6.78 (*J* = 9.0 Hz, 2H), 6.57 (d, *J* = 8.5 Hz, 2H), 5.61 (s, 1H), 3.73 (s, 3H), 3.54 (ddd, *J* = 12.2, 7.3, 5.4 Hz, 1H), 3.38 (ddd, *J* = 12.2, 6.1, 5.6 Hz, 1H), 3.05-2.85 (m, 2H), 2.87 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.7, 149.4, 144.7, 138.2, 135.4, 131.0, 128.9 (2C), 128.3, 128.1, 126.4, 125.8, 118.0 (2C), 114.4 (2C), 112.0 (2C), 63.8, 55.6, 44.0, 40.5 (2C), 28.0. IR (solid) 2990, 2903, 2830, 1611, 1506, 1441, 1346, 1240 cm^{-1}. MS (FAB) *m/z* (rel intensity) 359 ($M+H^+$, 40), 358 (M^+, 100), 238 (40), 154 (15), 136 (10). HRMS (FAB) Calcd for $C_{24}H_{27}N_2O$ ($M+H^+$): 359.2118, found, 359.2126.

1-(4-benzaldehyde dimethylacetal)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (11)



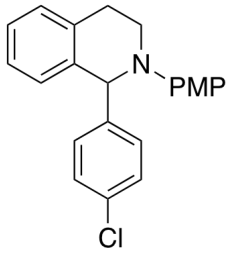
127.0 mg (76%). Colorless viscous oil. R_f = 0.24 (EtOAc/*n*-Hexane, 1 : 10). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.1 Hz, 2H), 7.25-7.10 (m, 6H), 6.85-6.75 (m, 4H), 5.65 (s, 1H), 5.43 (s, 1H), 3.74 (s, 3H), 3.65-3.35 (m, 6H), 3.00-2.85 (m, 2H), 1.20 (d, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8, 144.4, 143.4, 137.6, 137.6, 135.5, 128.3, 128.1, 127.9 (2C), 126.7, 126.3 (2C), 126.0, 117.5 (2C), 114.5 (2C), 101.5, 64.1, 61.1 (2C), 55.6, 44.6, 28.1, 15.2 (2C). IR (neat) 3020, 2972, 2880, 1508, 1454, 1240, 1111, 1049 cm^{-1}. MS (FAB) *m/z* (rel intensity) 418 ($M+H^+$, 40), 417 (M^+, 100), 372 (25), 238 (60), 136 (10). HRMS (FAB) Calcd for $C_{27}H_{32}NO_3$ ($M+H^+$): 418.2377, found, 418.2356. Anal Calcd for $C_{27}H_{31}NO_3$: C, 77.67; H, 7.48. Found: C, 77.46; H, 7.78.

1-(3-Fluorophenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (12)



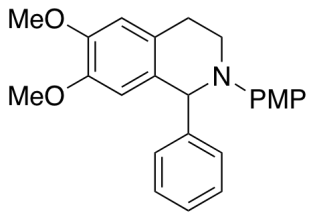
105.0 mg (79%). White solid. R_f = 0.36 (EtOAc/*n*-Hexane, 1 : 10). M.p. 87-89 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.10 (m, 5H), 7.00-6.75 (m, 7H), 5.62 (s, 1H), 3.74 (s, 3H), 3.56 (dt, *J* = 11.7, 5.6 Hz, 1H), 3.40 (ddd, *J* = 11.7, 7.1, 5.1 Hz, 1H), 3.00-2.85 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.8 (d, *J* = 245.8 Hz), 153.1, 146.2 (d, *J* = 6.6 Hz), 144.2, 137.1, 135.5, 129.4 (d, *J* = 8.3 Hz), 128.5, 128.0, 127.0, 126.1, 123.7 (d, *J* = 3.3 Hz), 117.8 (2C), 114.9 (d, *J* = 21.5 Hz), 114.5 (2C), 113.7 (d, *J* = 21.5 Hz), 64.0, 55.6, 44.8, 28.2. IR (solid) 3025, 2907, 2832, 1611, 1587, 1508, 1439, 1240 cm^{-1}. MS (FAB) *m/z* (rel intensity) 334 ($M+H^+$, 50), 333 (M^+, 100), 238 (60), 154 (25), 136 (25). HRMS (FAB) Calcd for $C_{22}H_{21}FNO$ ($M+H^+$): 334.1602, found, 334.1610. Anal Calcd for $C_{22}H_{20}FNO$: C, 79.26; H, 6.05. Found: C, 79.20; H, 5.88.

1-(4-Chlorophenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (13)



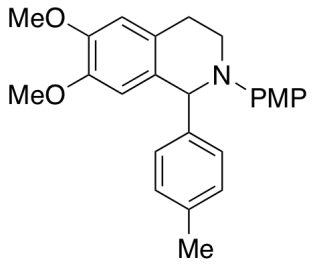
109.7 mg (78%). Colorless viscous oil. R_f = 0.30 (EtOAc/*n*-Hexane, 1 : 9). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22-7.00 (m, 8H), 6.80 (d, *J* = 1.5 Hz, 4H), 5.60 (s, 1H), 3.74 (s, 3H), 3.51 (ddd, *J* = 11.7, 6.6, 5.4 Hz, 1H), 3.38 (ddd, *J* = 11.7, 6.6, 5.4 Hz, 1H), 3.05-2.85 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.2, 144.2, 141.7, 137.2, 135.4, 132.6, 129.5 (2C), 128.5, 128.1 (2C), 128.0, 126.9, 126.1, 118.3 (2C), 114.5 (2C), 64.0, 55.6, 44.7, 28.2. IR (neat) 3025, 2930, 2832, 1508, 1487, 1240, 1090, 1036 cm^{-1}. MS (FAB) *m/z* (rel intensity) 350 ($M+H^+$, 60), 349 (100), 238 (75), 154 (20), 136 (20). HRMS (FAB) Calcd for $C_{22}H_{21}ClNO$ ($M+H^+$): 350.1306, found, 350.1227. Anal Calcd for $C_{22}H_{20}ClNO$: C, 75.53; H, 5.76. Found: C, 75.80; H, 5.78.

1-Phenyl-2-(4-methoxyphenyl)-1,2,3,4-tetrahydro-6,7-dimethoxyisoquinoline (14)



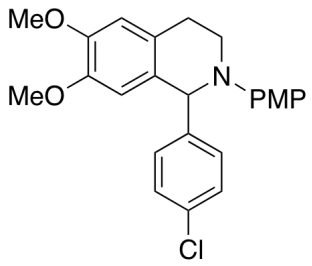
124.3 mg (83%). White solid. R_f = 0.24 (EtOAc/*n*-Hexane, 1 : 5). M.p. 112-114 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.05 (m, 5H), 6.85 (d, *J* = 9.2 Hz, 2H), 6.78 (d, *J* = 9.2 Hz, 2H), 6.65 (s, 1H), 6.59 (s, 1H), 5.58 (s, 1H), 3.87 (s, 3H), 3.79 (s, 3H), 3.74 (s, 3H), 3.50-3.30 (m, 2H), 2.90 (ddd, *J* = 15.9, 8.1, 5.9 Hz, 1H), 2.77 (dt, *J* = 15.9, 5.1 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.2, 147.8, 147.2, 144.6, 143.2, 129.0, 128.4 (2C), 127.9 (2C), 127.6, 126.8, 118.7 (2C), 114.4 (2C), 111.3, 111.2, 64.0, 56.0, 55.9, 55.6, 44.1, 27.2. IR (solid) 2905, 2828, 1609, 1506, 1449, 1373, 1236, 1118 cm^{-1}. MS (FAB) *m/z* (rel intensity) 376 ($M+H^+$, 45), 375 (M^+, 100), 298 (70), 154 (10), 136 (10). HRMS (FAB) Calcd for $C_{24}H_{26}NO_3$ ($M+H^+$): 376.1907, found, 376.1919.

1-(2-Tolyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydro-6,7-dimethoxyisoquinoline (15)



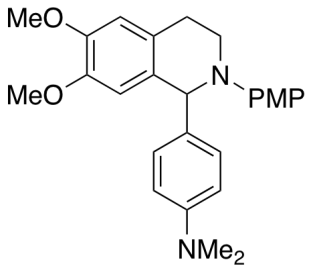
129.2 mg (83%). Pale yellow viscous oil. R_f = 0.21 (EtOAc/*n*-Hexane, 1 : 5). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.02 (d, *J* = 8.3 Hz, 2H), 6.99 (d, *J* = 8.3 Hz, 2H), 6.85 (d, *J* = 9.0 Hz, 2H), 6.78 (d, *J* = 9.0 Hz, 2H), 6.65 (s, 1H), 6.59 (s, 1H), 5.56 (s, 1H), 3.86 (s, 3H), 3.78 (s, 3H), 3.74 (s, 3H), 3.50-3.30 (m, 2H), 2.90 (ddd, *J* = 15.9, 8.3, 5.9 Hz, 1H), 2.75 (dt, *J* = 15.9, 5.1 Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.1, 147.8, 147.2, 144.6, 140.2, 136.4, 129.2, 128.6 (2C), 128.3 (2C), 127.5, 118.6 (2C), 114.4 (2C), 111.2, 111.2, 63.7, 56.0, 55.8, 55.6, 43.8, 27.1, 21.0. IR (neat) 2932, 2832, 1611, 1508, 1462, 1238, 1113, 1034 cm^{-1}. MS (FAB) *m/z* (rel intensity) 390 ($M+H^+$, 50), 389 (M^+, 95), 388 (40), 298 (100), 239 (15). HRMS (FAB) Calcd for $C_{25}H_{28}NO_3$ ($M+H^+$): 390.2064, found, 390.2073. Anal Calcd for $C_{25}H_{27}NO_3 \bullet 1H_2O$: C, 73.68; H, 7.17. Found: C, 73.79; H, 6.87.

1-(4-Chlorophenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydro-6,7-dimethoxyisoquinoline (16)



128.0 mg (78%). Pale yellow viscous oil. R_f = 0.18 (EtOAc/*n*-Hexane, 1 : 5). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.4 Hz, 2H), 7.02 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 9.0 Hz, 2H), 6.78 (d, *J* = 9.0 Hz, 2H), 6.66 (s, 1H), 6.52 (s, 1H), 5.52 (s, 1H), 3.87 (s, 3H), 3.78 (s, 3H), 3.74 (s, 3H), 3.45-3.30 (m, 2H), 2.90 (dt, *J* = 15.4, 6.8 Hz, 1H), 2.77 (dt, *J* = 15.4, 5.1 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.5, 148.0, 147.3, 144.4, 141.7, 132.6, 129.8 (2C), 128.5, 128.0 (2C), 127.5, 119.3 (2C), 114.4 (2C), 111.3, 111.1, 63.7, 56.0, 55.9, 55.5, 44.3, 27.3. IR (neat) 2934, 2832, 1611, 1506, 1462, 1240, 1115, 1034 cm^{-1}. MS (FAB) *m/z* (rel intensity) 410 ($M+H^+$, 40), 409 (M^+, 75), 298 (45), 154 (100), 136 (70). HRMS (FAB) Calcd for $C_{24}H_{25}ClNO_3$ ($M+H^+$): 410.1517, found, 410.1515. Anal Calcd for $C_{24}H_{24}ClNO_3$•1/4H_2O: C, 69.56; H, 5.96. Found: C, 69.70; H, 5.89.

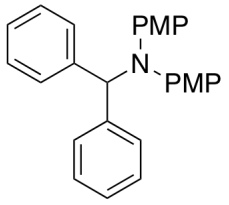
1-(4-Dimethylaminophenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydro-6,7-dimethoxyisoquinoline (17)



145.9 mg (87%). Yellow solid. R_f = 0.28 (EtOAc/*n*-Hexane, 3 : 7). M.P. 42-44 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.93 (d, *J* = 8.5 Hz, 2H), 6.87 (d, *J* = 9.0 Hz, 2H), 6.78 (d, *J* = 9.0 Hz, 2H), 6.70-6.50 (m, 4H), 5.54 (s, 1H), 3.86 (s, 3H), 3.78 (s, 3H), 3.74 (s, 3H), 3.50-3.30 (m, 2H), 3.00-2.80 (m, 1H), 2.89 (s, 6H), 2.72 (dt, *J* = 15.6, 4.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.0, 149.4, 147.6, 147.1, 144.8, 130.9, 129.5, 129.2 (2C), 127.4, 118.8 (2C), 118.1, 114.5, 114.3 (2C), 111.9 (2C), 111.2, 111.1, 63.3, 55.9, 55.8, 55.6, 43.3, 40.5 (2C), 26.9. IR (solid) 2934, 2832, 1611, 1506, 1462, 1348, 1240, 1113 cm^{-1}. MS (FAB) *m/z* (rel intensity) 419 ($M+H^+$, 50), 418 (M^+, 100),

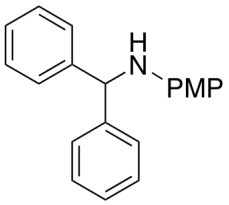
298 (45), 154 (40), 136 (30). HRMS (FAB) Calcd for $C_{26}H_{31}N_2O_3$ ($M+H^+$): 419.2329, found, 419.2327.

_N_-Di-(4-methoxyphenyl)-1,1-diphenylmethylamine (18)



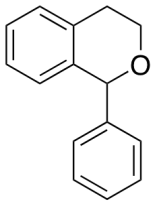
110.4 mg (70%). Pale yellow viscous oil. R_f = 0.28 (EtOAc/*n*-Hexane, 1 : 10). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.10 (m, 10H), 6.75 (d, *J* = 7.7 Hz, 4H), 6.65 (d, *J* = 7.7 Hz, 4H), 6.17 (s, 1H), 3.69 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.4 (2C), 141.7 (4C), 129.3 (4C), 128.0 (4C), 126.8 (2C), 124.5 (4C), 114.0 (4C), 69.5, 55.4 (2C). IR (neat) 3390, 3028, 2951, 2833, 1501, 1236, 1179, 1030 cm^{-1}. MS (FAB) *m/z* (rel intensity) 396 ($M+H^+$, 30), 395 (M^+, 70), 319 (30), 228 (100), 167 (65). HRMS (FAB) Calcd for $C_{27}H_{26}NO_2$ ($M+H^+$): 396.1958, found, 396.1928. Anal Calcd for $C_{27}H_{25}NO_2$•1/3H_2O: C, 80.77; H, 6.44. Found: C, 81.02; H, 6.38.

_N_-(4-Methoxyphenyl)-1,1-diphenylmethylamine (19)[3]



105.0 mg (91%). Pale yellow viscous oil. R_f = 0.35 (EtOAc/*n*-Hexane, 1 : 9). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45-7.20 (m, 10H), 6.70 (d, *J* = 8.8 Hz, 2H), 6.49 (d, *J* = 8.8 Hz, 2H), 5.41 (s, 1H), 3.97 (br s, 1H), 3.68 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.1, 143.2 (2C), 141.7, 128.7 (2C), 127.4 (4C), 127.3 (4C), 114.7 (2C), 114.6 (2C), 63.8, 55.7. IR (neat) 3391, 3026, 2930, 2832, 1508, 1240, 1177, 1028 cm^{-1}. MS (FAB) *m/z* (rel intensity) 290 ($M+H^+$, 30), 289 (100), 212 (15), 167 (70), 165 (15). HRMS (FAB) Calcd for $C_{20}H_{20}NO$ ($M+H^+$): 290.1539, found, 290.1521.

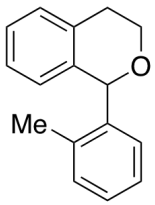
1-Phenylisochroman (20)[4]



After stirring the mixture of isochroman (53.7 mg, 0.4 mmol) and DDQ (99.9 mg, 0.44 mmol) in chlorobenzene (4.0 mL) in a vial at 80 °C for 1.5 h under Ar atmosphere, phenylmagnesium bromide (3.0 M in Et_2O, 2.7 mL, 0.80 mmol) was added to the suspension at 0 °C. After stirring vigorously for 3 hours at 0 °C, the reaction mixture was quenched with saturated aqueous $NaHCO_3$, and extracted with ethyl acetate. The organic layer was washed with water and brine, dried over $MgSO_4$, filtrated, and concentrated in *vacuo*. The residue was purified by SiO_2 column chromatography (*n*-hexane/ether = 100/0-100/5) to give 1-phenylisochroman **20** (71.1 mg, 85%) as white solid.

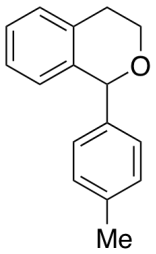
71.1 mg (85%). White solid. R_f = 0.30 (EtOAc/*n*-Hexane, 1 : 10). M.p. 87-89 °C (lit.[4] M.p. 87-89 °C). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.25 (m, 5H), 7.25-7.15 (m, 2H), 7.15-7.00 (m, 1H), 6.75 (d, *J* = 7.6 Hz, 1H), 5.73 (s, 1H), 4.20 (ddd, *J* = 11.4, 5.6, 3.7 Hz, 1H), 3.94 (ddd, *J* = 11.4, 9.5, 3.7 Hz, 1H), 3.15 (ddd, *J* = 16.4, 9.5, 5.6 Hz, 1H), 2.81 (dt, *J* = 16.4, 3.7 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.2, 137.4, 133.8, 128.9 (2C), 128.7, 128.4 (2C), 128.1, 126.9, 126.6, 125.9, 79.7, 63.9, 28.8. IR (solid) 3019, 2930, 2835, 1599, 1489, 1450, 1281, 1109 cm^{-1}. MS (FAB) *m/z* (rel intensity) 211 ($M+H^+$, 25), 209 (60), 154 (100), 136 (75), 91 (70). HRMS (FAB) Calcd for $C_{15}H_{15}O$ ($M+H^+$): 211.1117, found, 211.1116.

1-(2-Methylphenyl)-isochroman (21)



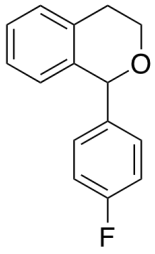
69.1 mg (77%). Colorless oil. R_f = 0.35 (EtOAc/*n*-Hexane, 1 : 10). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.00 (m, 7H), 6.69 (d, *J* = 7.8 Hz, 1H), 5.92 (s, 1H), 4.20 (ddd, *J* = 11.2, 5.6, 3.7 Hz, 1H), 3.93 (ddd, *J* = 11.2, 9.5, 3.9 Hz, 1H), 3.15 (ddd, *J* = 16.4, 9.5, 5.6 Hz, 1H), 2.81 (dt, *J* = 16.4, 3.7 Hz, 1H), 2.35 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.9, 137.5, 137.2, 133.9, 130.9, 129.9, 128.6, 128.0, 126.5, 126.3, 126.0, 125.7, 77.7, 64.1, 28.8, 19.4. IR (neat) 3021, 2924, 2853, 1489, 1450, 1373, 1271, 1082 cm^{-1}. MS *m/z* (rel intensity) 225 ($M+H^+$, 35), 224 (M^+, 15), 223 (50), 154 (100), 136 (70), 105 (60). HRMS Calcd for $C_{16}H_{17}O$ ($M+H^+$): 225.1274, found, 225.1314. Anal Calcd for $C_{16}H_{16}O$•1/10 H_2O: C, 84.99; H, 7.22. Found: C, 85.18; H, 7.57.

1-(4-Methylphenyl)-isochroman (22)



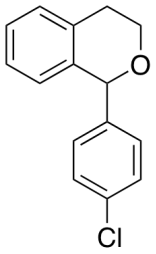
72.6 mg (81%). Colorless oil. R_f = 0.47 (EtOAc/*n*-Hexane, 1 : 10). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23-7.10 (m, 6H), 7.10-7.00 (m, 1H), 6.76 (d, *J* = 7.8 Hz, 1H), 5.70 (s, 1H), 4.18 (ddd, *J* = 11.2, 5.4, 3.9 Hz, 1H), 3.91 (ddd, *J* = 11.2, 9.4, 3.9 Hz, 1H), 3.12 (ddd, *J* = 16.4, 9.4, 5.4 Hz, 1H), 2.80 (dt, *J* = 16.4, 3.9 Hz, 1H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.3, 137.8, 137.5, 133.9, 129.1 (2C), 128.8 (2C), 128.7, 126.9, 126.5, 125.9, 79.4, 63.7, 28.8, 21.2. IR (neat) 3021, 2922, 2851, 1514, 1491, 1450, 1279, 1088 cm^{-1}. MS *m/z* (rel intensity) 225 ($M+H^+$, 40), 224 (M^+, 30), 223 (100), 207 (30), 133 (65), 105 (60). HRMS Calcd for $C_{16}H_{17}O$ ($M+H^+$): 225.1274, found, 225.1271. Anal Calcd for $C_{16}H_{16}O$: C, 85.68; H, 7.19. Found: C, 85.55; H, 7.07.

1-(4-Fluorophenyl)-isochroman (23)



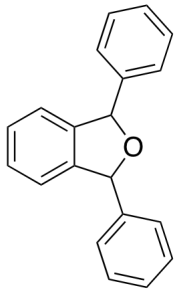
78.7 mg (86%). White solid. R_f = 0.27 (EtOAc/*n*-Hexane, 1 : 10). M.p. 94-98 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35-7.00 (m, 7H), 6.72 (d, *J* = 7.8 Hz, 1H), 5.72 (s, 1H), 4.18 (ddd, *J* = 11.2, 5.4, 3.7 Hz, 1H), 3.93 (ddd, *J* = 11.2, 9.5, 3.9 Hz, 1H), 3.14 (ddd, *J* = 16.4, 9.5, 5.4 Hz, 1H), 2.80 (dt, *J* = 16.4, 3.7 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6 (d, *J* = 246.6 Hz), 138.1 (d, *J* = 3.3 Hz), 137.1, 133.8, 130.6 (d, *J* = 8.3 Hz, 2C), 128.8, 126.8, 126.7, 126.0, 115.3 (d, *J* = 21.5 Hz, 2C), 78.9, 63.9, 28.8. IR (solid) 3063, 2928, 2862, 1603, 1508, 1449, 1281, 1092 cm^{-1}. MS *m/z* (rel intensity) 229 ($M+H^+$, 10), 228 (M^+, 5), 227 (15), 154 (100), 136 (70). HRMS Calcd for $C_{15}H_{14}FO$ ($M+H^+$): 229.1023, found, 229.1026. Anal Calcd for $C_{15}H_{13}FO$: C, 78.93; H, 5.74. Found: C, 79.06; H, 5.40.

1-(4-Chlorophenyl)-isochroman (24)



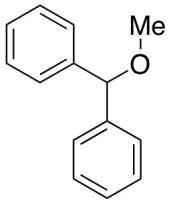
88.7 mg (91%). White solid. R_f = 0.31 (EtOAc/*n*-Hexane, 1 : 10). M.p. 60-62 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.10 (m, 6H), 7.10-7.00 (m, 1H), 6.71 (d, *J* = 7.8 Hz, 1H), 5.70 (s, 1H), 4.17 (ddd, *J* = 11.5, 5.6, 3.7 Hz, 1H), 3.92 (ddd, *J* = 11.5, 9.5, 3.9 Hz, 1H), 3.13 (ddd, *J* = 16.4, 9.5, 5.6 Hz, 1H), 2.80 (dt, *J* = 16.4, 3.7 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.7, 136.8, 133.9, 133.8, 130.2 (2C), 128.8, 128.6 (2C), 126.8, 126.7, 126.0, 78.8, 63.9, 28.7. IR (solid) 3020, 2926, 2853, 1597, 1489, 1450, 1279, 1088 cm^{-1}. MS *m/z* (rel intensity) 278 ($M+H^+$, 65), 243 (75), 154 (100), 136 (65), 105 (35). HRMS Calcd for $C_{15}H_{14}ClO$ ($M+H^+$): 245.0728, found, 245.0729. Anal Calcd for $C_{15}H_{13}ClO$: C, 73.62; H, 5.35. Found: C, 73.55; H, 5.48.

1,3-Diphenyl-1,3-dihydroisobenzofuran (25)[5]



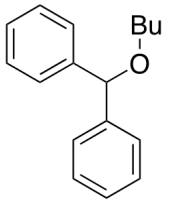
38.2 mg (35%). White solid. R_f = 0.51 (EtOAc/*n*-Hexane, 1 : 10). ^{1}H NMR (400 MHz, $CDCl_3$) two isomers (*trans*:*cis*=10:1) δ 7.40-7.20 (m, 12H), 7.15-7.00 (m, 2H), 6.44 (s, 20/11H), 6.21 (s, 2/11H). ^{13}C NMR (100 MHz, $CDCl_3$) two isomers (*trans*:*cis*=10:1) δ 142.5 (2/11C), 142.1 (20/11C), 141.8 (20/11C), 141.3 (2/11C), 128.6 (40/11C), 128.4 (4/11C), 128.3 (2/11C), 128.1 (20/11C), 127.9 (20/11C), 127.8 (2/11C), 126.9 (4C), 122.3 (20/11C), 122.2 (2/11C), 86.0 (20/11C), 85.5 (2/11C). IR (solid) 3026, 2924, 2853, 1659, 1597, 1493, 1281, 1011 cm^{-1}. MS *m/z* (rel intensity) 273 ($M+H^+$, 20), 271 (30), 154 (100), 136 (65), 107 (20). HRMS Calcd for $C_{20}H_{17}O$ ($M+H^+$): 273.1274, found, 273.1274. Anal Calcd for $C_{20}H_{16}O$: C, 88.20; H, 5.92. Found: C, 88.16; H, 6.04.

Diphenylmethyl methyl ether (26)[6]



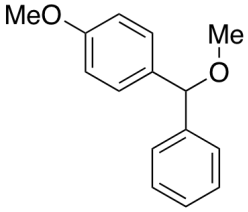
65.3 mg (82%). White solid. R_f = 0.57 (EtOAc/*n*-Hexane, 1 : 9). M.p. 49-50 °C (lit.[6a] M.P. 48-49). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.10 (m, 10H), 5.24 (s, 1H), 3.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.1 (2C), 128.4 (4C), 127.4 (2C), 126.9 (4C), 85.4, 57.0. IR (solid) 3030, 2926, 2822, 1597, 1481, 1454, 1190, 1094 cm^{-1}. MS *m/z* (rel intensity) 199 ($M+H^+$, 5), 198 (M^+, 10), 167 (75), 154 (100), 136 (65). HRMS Calcd for $C_{14}H_{15}O$ ($M+H^+$): 199.1117, found, 199.1131. Anal Calcd for $C_{14}H_{14}O$: C, 84.81; H, 7.12. Found: C, 84.91; H, 7.04.

Diphenylmethyl butyl ether (27)[7]



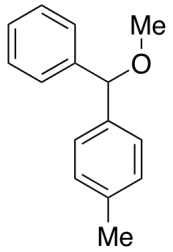
93.5 mg (97%). Colorless oil. R_f = 0.54 (EtOAc/*n*-Hexane, 1 : 19). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.20 (m, 10H), 5.33 (s, 1H), 3.45 (t, *J* = 6.5 Hz, 2H), 1.63 (quintet, 6.5 Hz, 2H), 1.42 (sextet, 7.3 Hz, 2H), 0.91 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.7 (2C), 128.3 (4C), 127.3 (2C), 126.9 (4C), 83.6, 68.9, 32.0, 19.5, 13.9. IR (neat) 3030, 2932, 2870, 1493, 1452, 1186, 1094, 1028 cm^{-1}. MS *m/z* (rel intensity) 241 ($M+H^+$, 5), 239 (10), 167 (100), 165 (30), 107 (30). HRMS Calcd for $C_{17}H_{21}O$ ($M+H^+$): 241.1587, found, 241.1589.

1-Methoxy-4-(methoxyphenylmethyl)benzene (28)



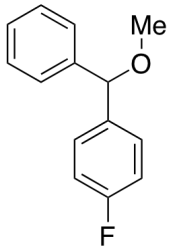
148.4 mg (82%). Colorless oil. R_f = 0.42 (EtOAc/*n*-Hexane, 1 : 9). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.15 (m, 7H), 6.85 (d, *J* = 8.5 Hz, 2H), 5.20 (s, 1H), 3.77 (s, 3H), 3.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.9, 142.3, 134.3, 128.3 (2C), 128.2 (2C), 127.3, 126.8 (2C), 113.8 (2C), 84.9, 56.9, 22.2. IR (neat) 3028, 2934, 2820, 1611, 1508, 1244, 1171, 1090 cm^{-1}. MS *m/z* (rel intensity) 229 ($M+H^+$, 10), 228 (M^+, 60), 197 (100), 151 (40), 121 (45). HRMS Calcd for $C_{15}H_{17}O_2$ ($M+H^+$): 229.1223, found, 229.1189.

4-(Methoxyphenylmethyl)toluene (29)[8]



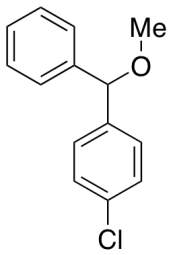
69.3 mg (82%). Colorless oil. R_f = 0.60 (EtOAc/*n*-Hexane, 1 : 9). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.00 (m, 9H), 5.21 (s, 1H), 3.37 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.3, 139.1, 137.1, 129.1 (3C), 128.3 (2C), 127.3, 126.9 (2C), 126.8 (2C), 85.3, 56.9, 21.1. IR (neat) 3026, 2924, 2820, 1512, 1452, 1192, 1092, 1020 cm^{-1}. MS *m/z* (rel intensity) 213 ($M+H^+$, 5), 212 (M^+, 10), 181 (100), 154 (35), 121 (50). HRMS Calcd for $C_{15}H_{17}O$ ($M+H^+$): 213.1274, found, 213.1194.

1-Fluoro-4-(methoxyphenylmethyl)benzene (30)[9]



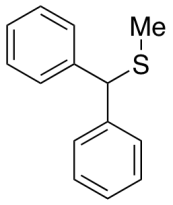
81.6 mg (94%). Colorless oil. R_f = 0.47 (EtOAc/*n*-Hexane, 1 : 19). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.20 (m, 7H), 7.00 (t, *J* = 8.8 Hz, 2H), 5.22 (s, 1H), 3.37 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.1 (d, *J* = 245.0 Hz), 141.8, 137.9 (d, *J* = 3.3 Hz), 128.5 (d, *J* = 7.4 Hz, 2C), 128.5 (2C), 127.6, 126.8 (2C), 115.2 (d, *J* = 21.5 Hz, 2C), 84.7, 57.0. IR (neat) 3030, 2932, 2822, 1603, 1506, 1452, 1221, 1090 cm^{-1}. MS *m/z* (rel intensity) 217 ($M+H^+$, 5), 216 (M^+, 10), 185 (100), 154 (80), 121 (60). HRMS Calcd for $C_{14}H_{14}FO$ ($M+H^+$): 217.1023, found, 217.1036.

1-Chloro-4-(methoxyphenylmethyl)benzene (31)[10]



90.6 mg (97%). White solid. R_f = 0.54 (EtOAc/*n*-Hexane, 1 : 9). M.p. 45-47 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.20 (m, 9H), 5.21 (s, 1H), 3.37 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.6, 140.7, 133.2, 129.0, 128.5, 128.5 (2C), 128.2 (2C), 127.7, 126.9 (2C), 84.7, 57.0. IR (solid) 3030, 2928, 2822, 1593, 1489, 1474, 1190, 1088 cm^{-1}. MS *m/z* (rel intensity) 233 ($M+H^+$, 10), 232 (M^+, 15), 201 (100), 154 (65), 121 (70). HRMS Calcd for $C_{14}H_{14}ClO$ ($M+H^+$): 233.0728, found, 233.0617.

Diphenylmethyl butyl sulfide (32)[11]



44.3 mg (52%). White solid. R_f = 0.43 (EtOAc/*n*-Hexane, 1 : 39). M.p. 30-31 °C {lit.[11a] 30-32 °C}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.20 (m, 10H), 5.05 (s, 1H), 1.98 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.2 (2C), 128.5 (4C), 128.3 (4C), 127.1 (2C), 56.1, 15.9. IR (solid) 3026, 2914, 1665, 1597, 1493, 1481, 1449, 1074 cm^{-1}. MS *m/z* (rel intensity) 215 ($M+H^+$, 1), 214 (M^+, 15), 167 (100), 154 (30), 137 (35). HRMS Calcd for $C_{14}H_{15}S$ ($M+H^+$): 215.0889, found, 215.0860.

Reference

1. Basle, O.; Li, C.-J. *Org. Lett.* **2008**, *10*, 3661-3663.

2. Hoeft, E.; Rieche, A.; Schultze, H. *Justus Liebigs Ann. Chem.* **1966**, *697*, 181-187.

3. (a) Grammaticakis, P. *Compt. Rend.* **1940**, *210*, 716-718. (b) Yu, A.; Wu, Y.; Cheng, B.; Wei, K.; Li, J. *Adv. Synth. Catal.* **2009**, *351*, 767-771.

4. Fujisaka, T.; Nojima, M.; Kusabayashi, S. *J. Org. Chem.* **1985**, *50*, 275-277.

5. (a) Li, G.; Zhou, S.; Su, G.; Liu, Y.; Wang, P. G. *J. Org. Chem.* **2007**, *72*, 9830-9833. (b) Smith, J. G.; McCall, R. B. *J. Org. Chem.* **1980**, *45*, 3982-3986.

6. (a) Markees, D. *J. Org. Chem.* **1958**, *23*, 1490-1492. (b) Phan, T. B.; Nolte, C.; Kobayashi, S.; Ofial, A. R.; Mayr, H. *J. Am. Chem. Soc.* **2009**, *131*, 11392-11401.

7. Bikard, Y.; Mezaache, R.; Weibel, J.-M.; Benkouider, A.; Sirlin, C.; Pale, P. *Tetrahedron* **2008**, *64*, 10224-10232.

8. Shi, M.; Okamoto, Y.; Takamuku, S. *Bull. Chem. Soc. Jpn.* **1990**, *63*, 2731-2733.

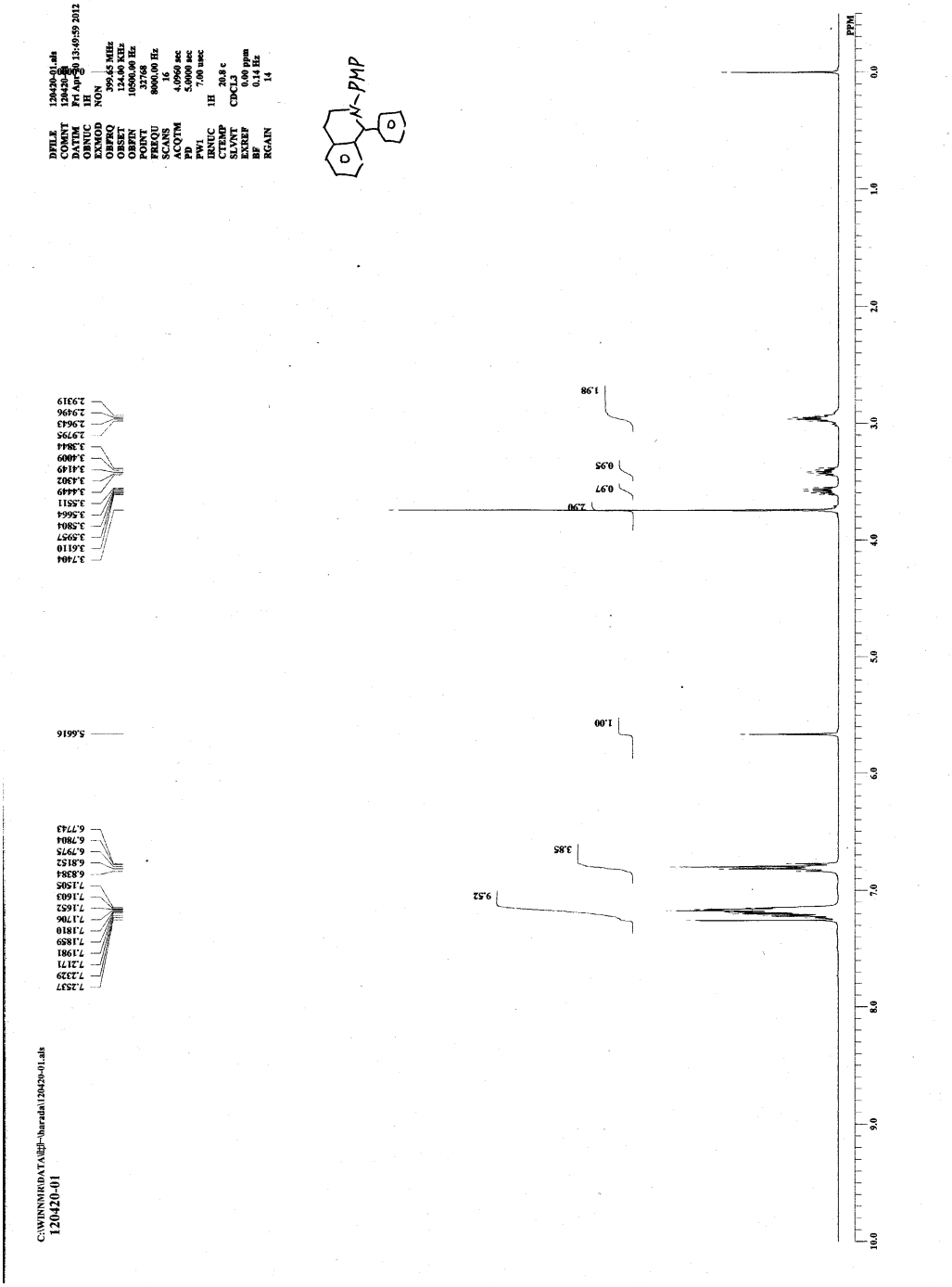
9. Matsumoto, K,; Ueoka, K.; Fujie, S.; Suga, S.; Yoshida, J. *Heterocycles* **2008**, *76*, 1103-1119.

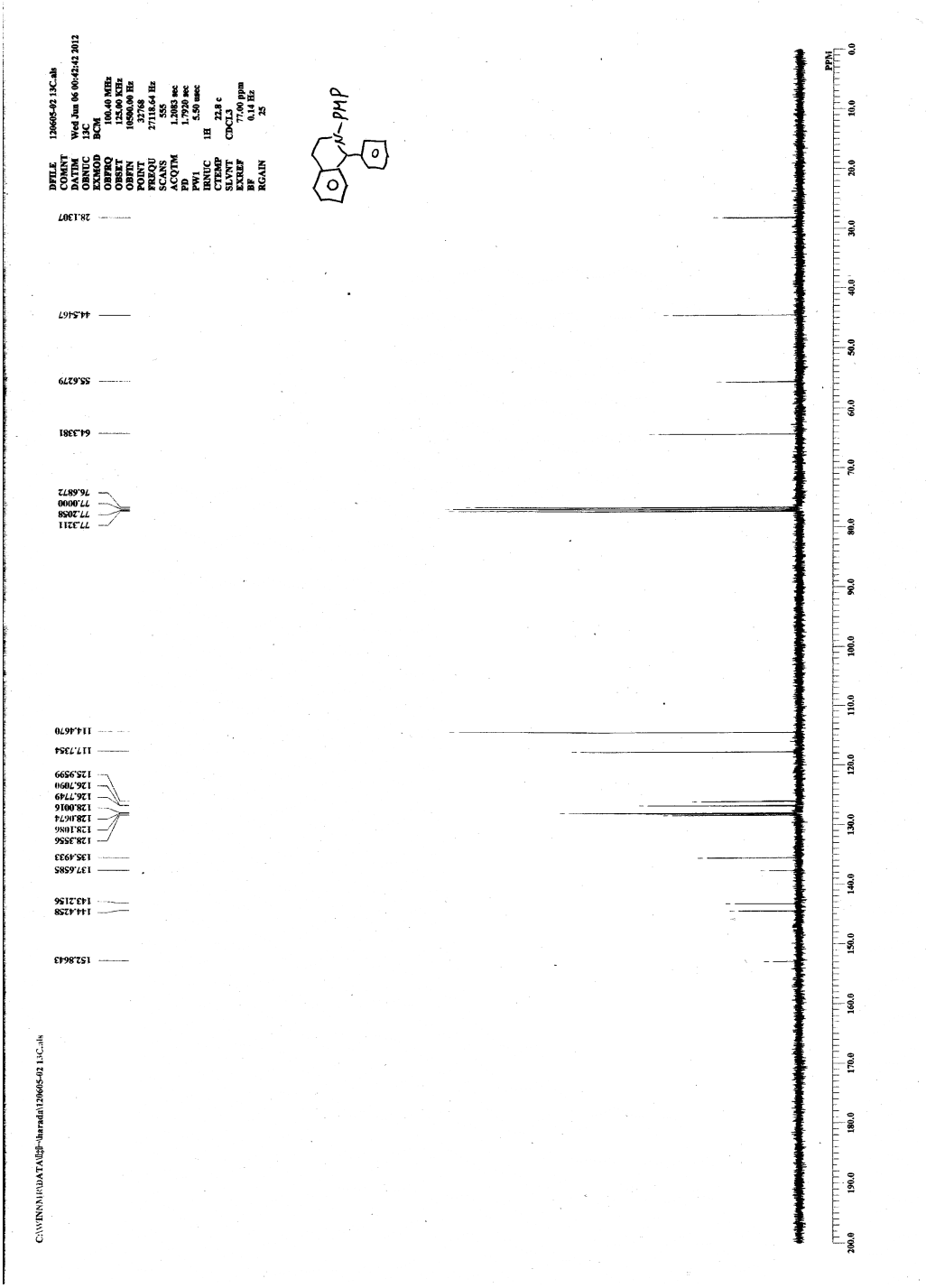
10. (a) Rutherford, K. G.; Mamer, O. A.; Prokipcak, J. M.; Jobin, R. A.. *Can. J. Chem.* **1966**, *44*, 2337-2339. (b) Chang, E. P.; Huang, R. L.; Lee, K. H. *J. Chem. Soc. B: Physical Organic* **1969**, 878-880.

11. (a) Lerch, U.; Moffatt, J. G. *J. Org. Chem.* **1971**, *36*, 3861-3869. (b) Ikehira, H.; Tanimoto, S.; Oida, T.; Okano, M. *J. Org. Chem.* **1983**, *48*, 1120-1122.

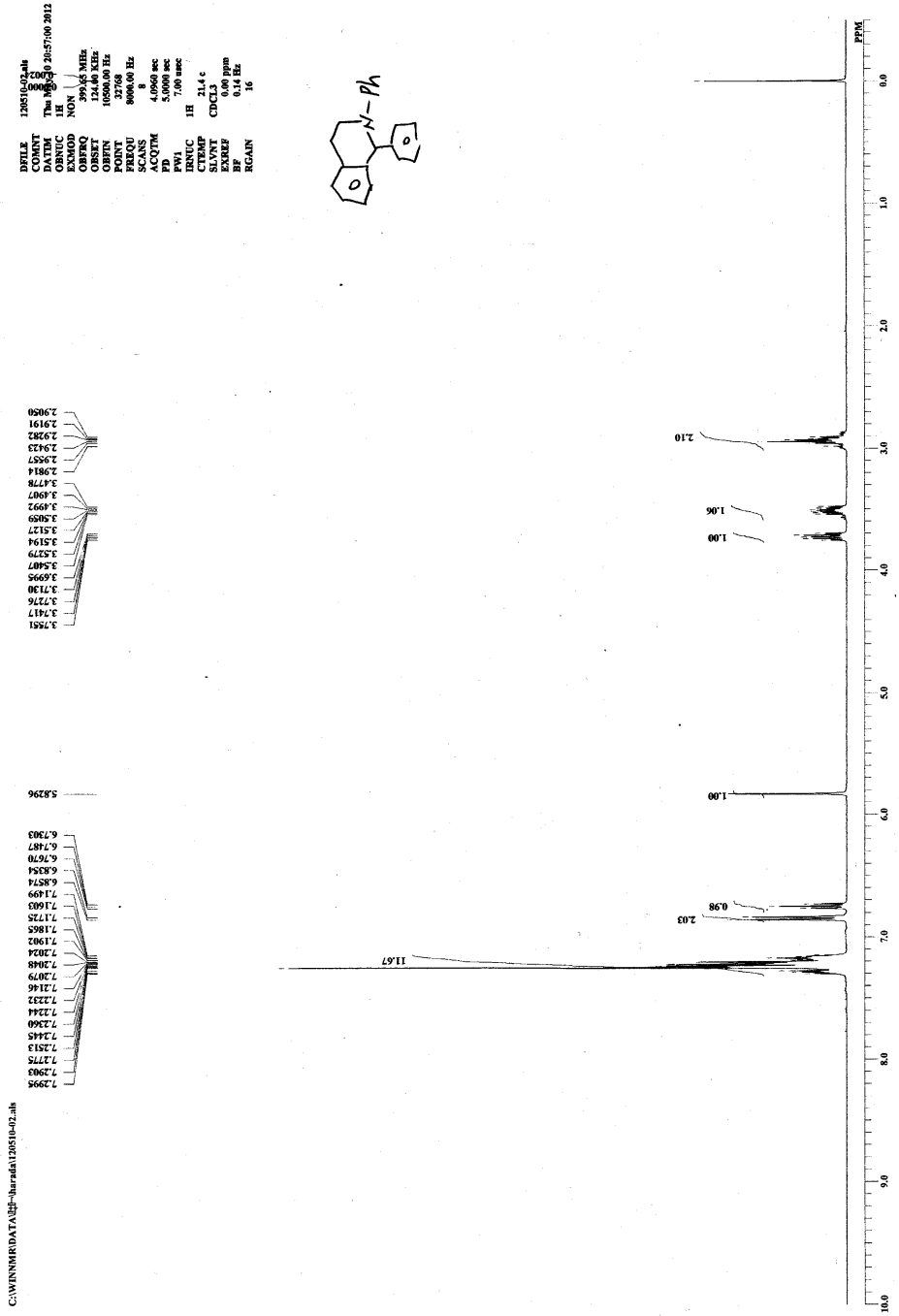
4. PDF file of copies of ^{1}H NMR and ^{13}C NMR spectra for new compounds

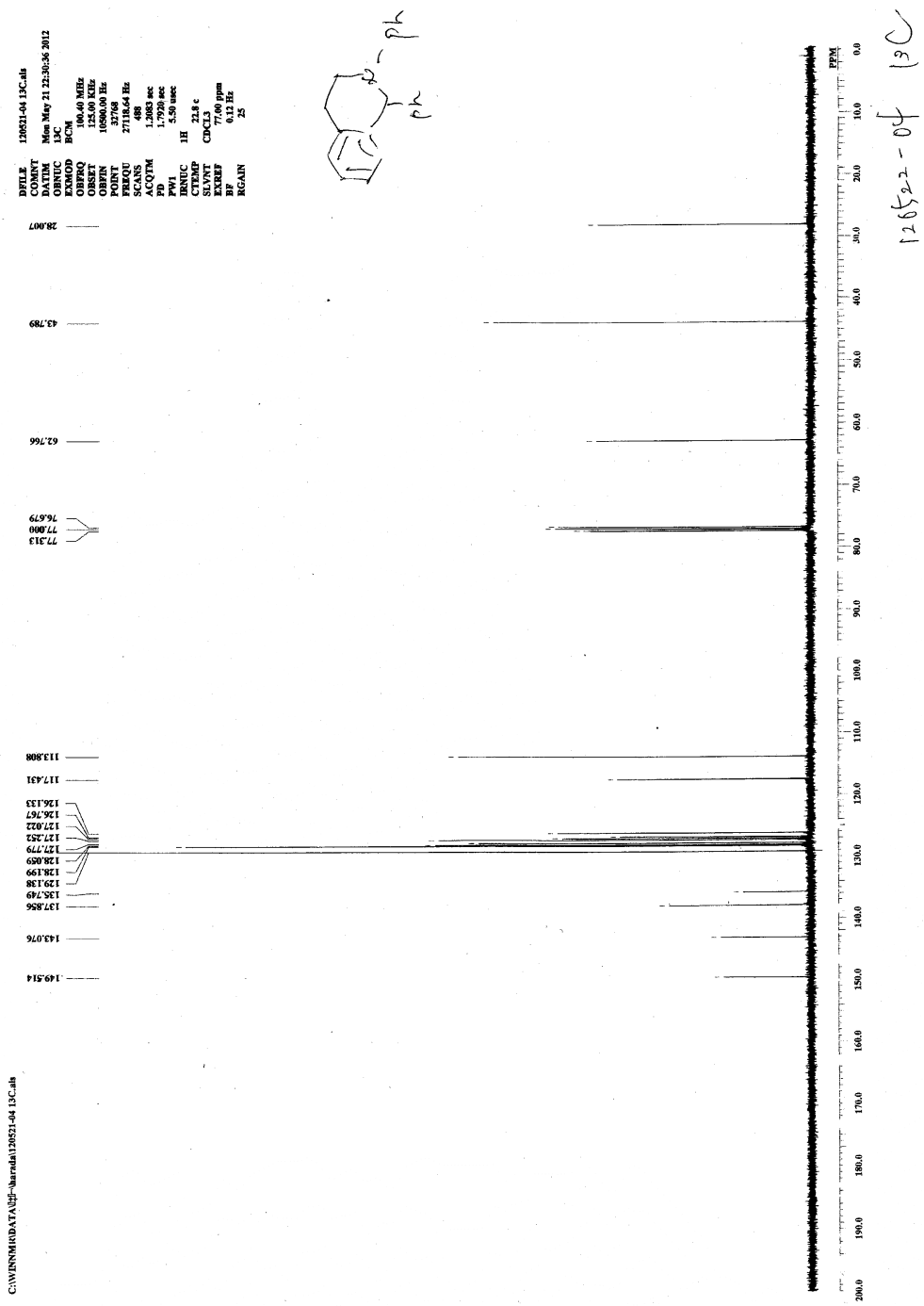
1-Phenyl-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (1)



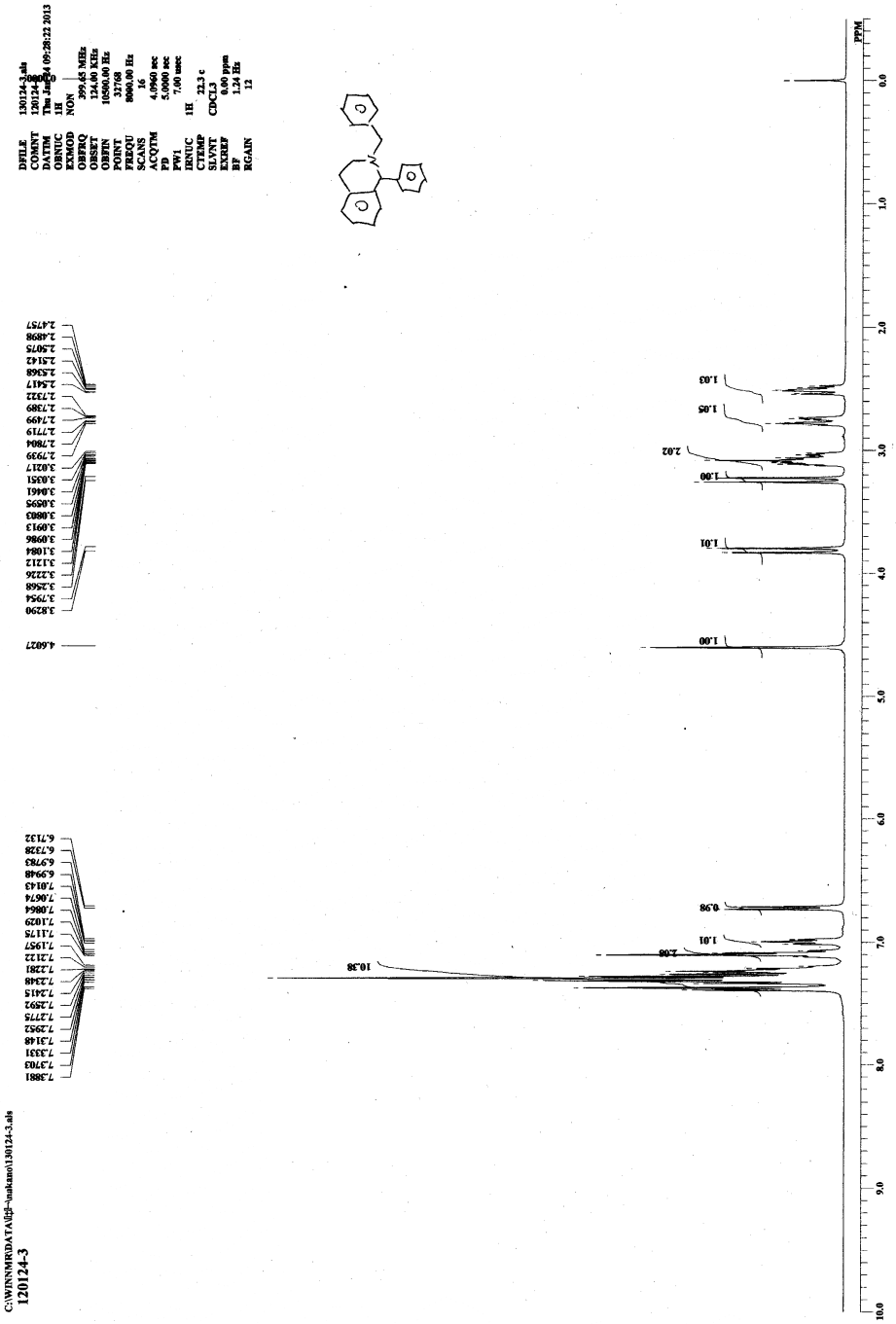

DFILE 120605-02 13C.als
COMNT
DATIM Wed Jun 06 00:42:42 2012
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 151.60 MHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 555
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H 22.8 c
CTEMP
SLVNT CDCL3
EXREF 77.00 ppm
RF 0.14 Hz
RGAIN 25
N-PMP
28.1307
44.5467
55.6279
64.3381
76.6872
77.0000
77.2058
77.3211
114.4670
117.7354
125.9599
126.7090
126.7749
128.0016
128.0674
128.1806
128.3556
135.4933
137.6585
143.2156
144.4258
152.8643
C:\WINNMR\DATA\data\120605-02 13C.als
PPM

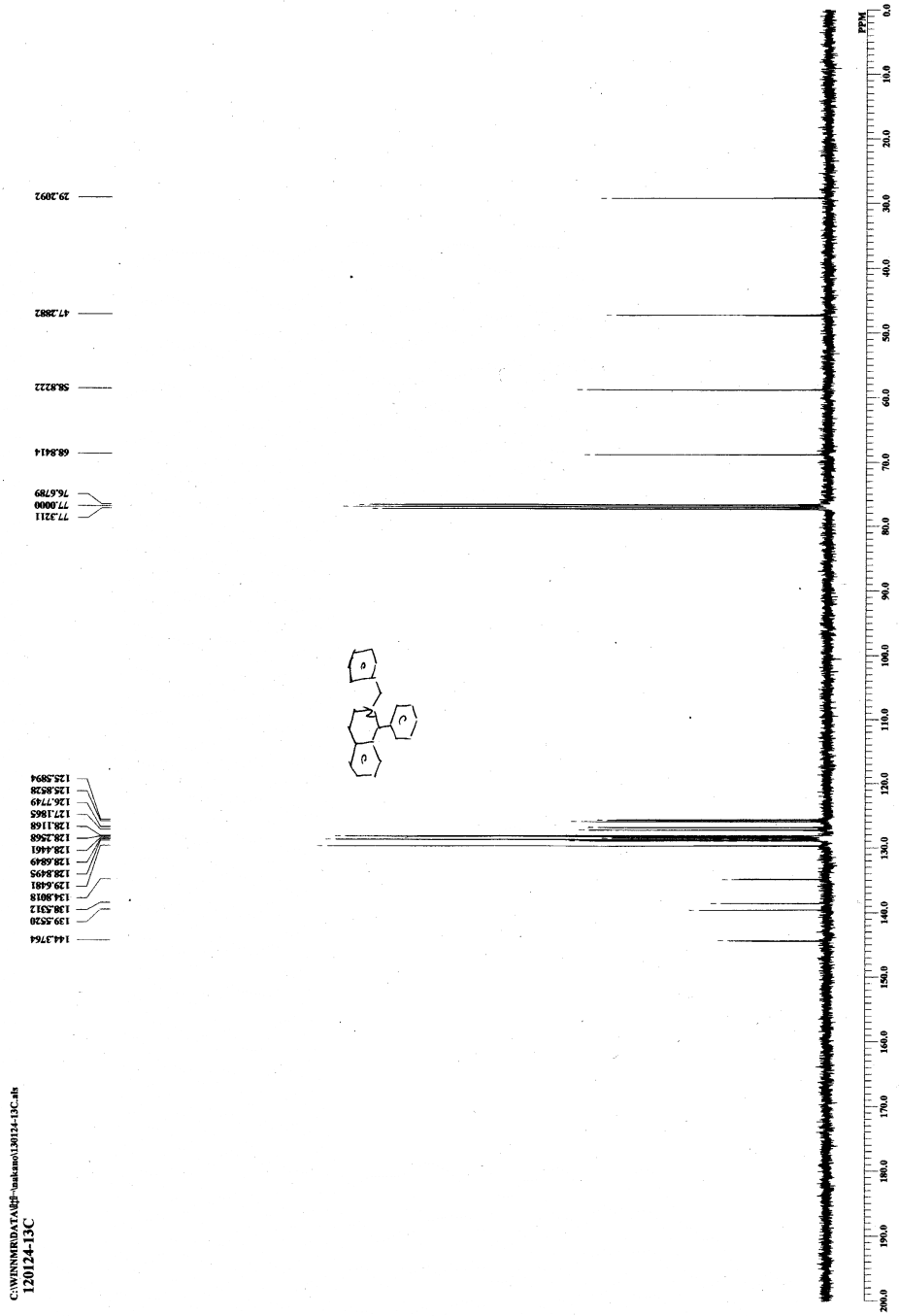
1-Phenyl-2-phenyl-1,2,3,4-tetrahydroisoquinoline (2)



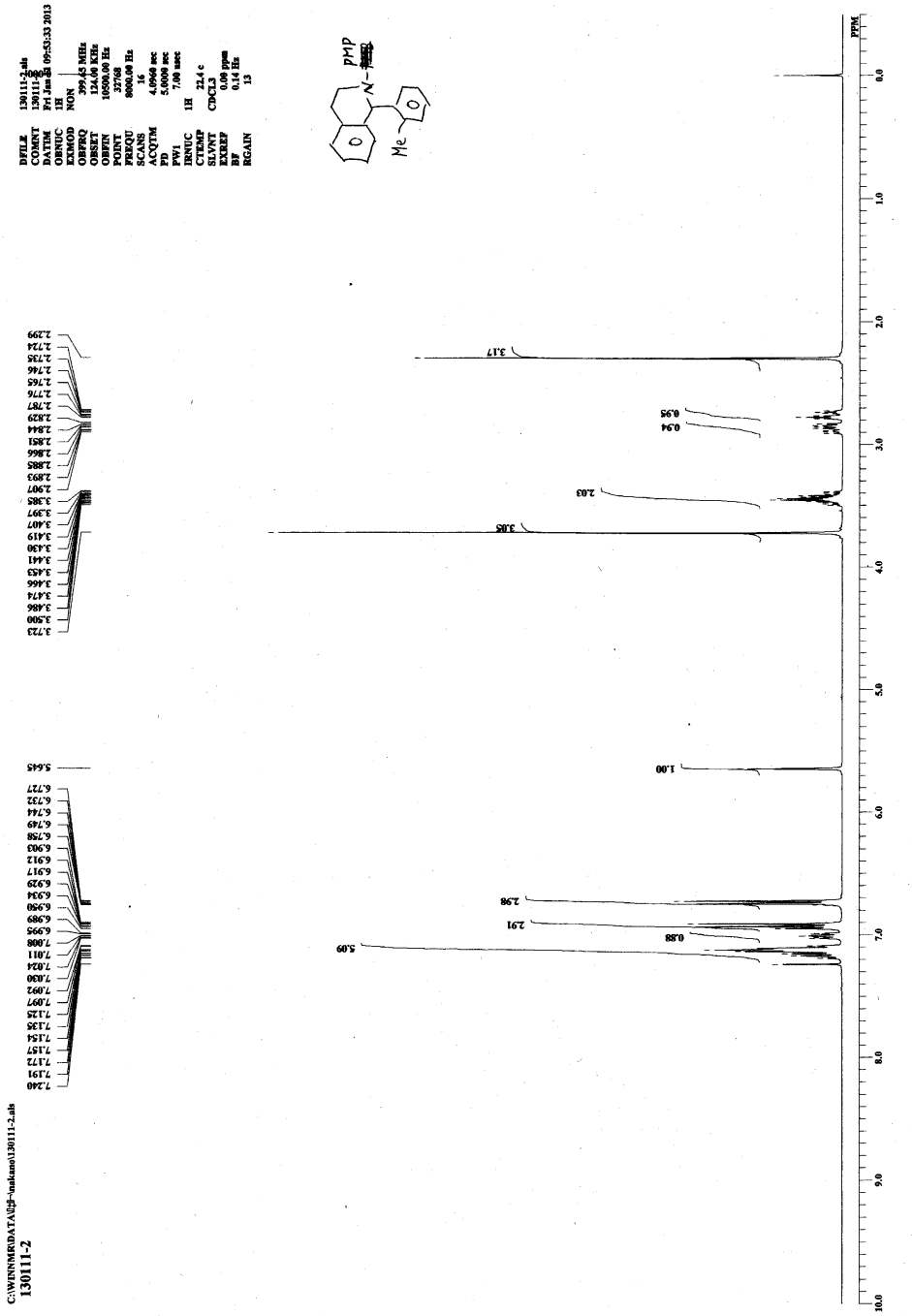

DFILE 120521-04 13C.als
COMNT
DATIM Mon May 21 22:30:36 2012
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10046.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 488
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
1H 22.0 c
IRNUC
CTEMP
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 25
C:\WINNMR\DATA\dpf-\harada\120521-04 13C.als
28.007
43.789
62.766
76.679
77.000
77.313
113.808
117.431
126.133
126.767
127.022
127.252
127.779
128.069
128.199
129.138
135.749
137.856
143.076
149.514
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
Ph
Ph
120502-04 13C

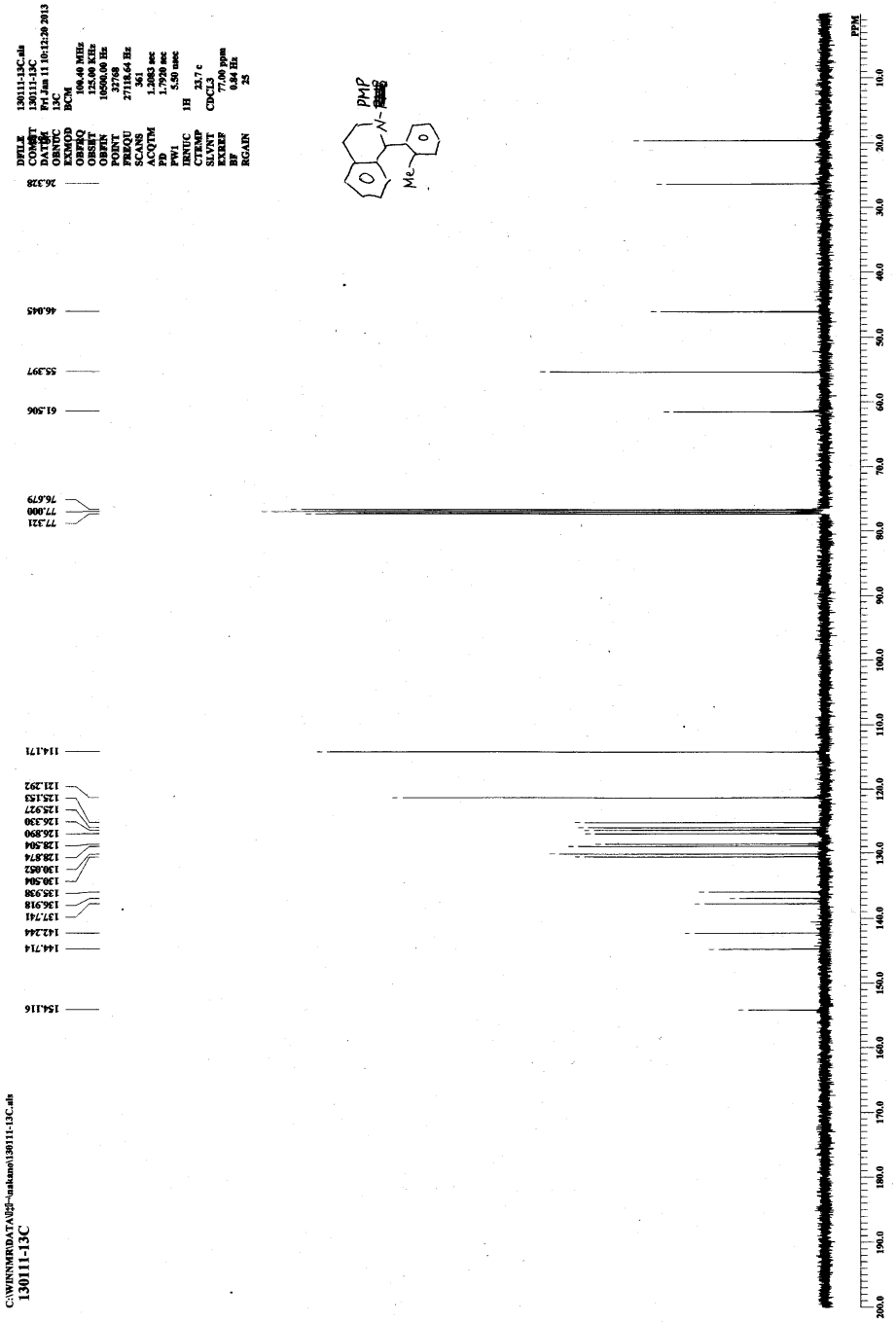
1-Benzyl-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3)



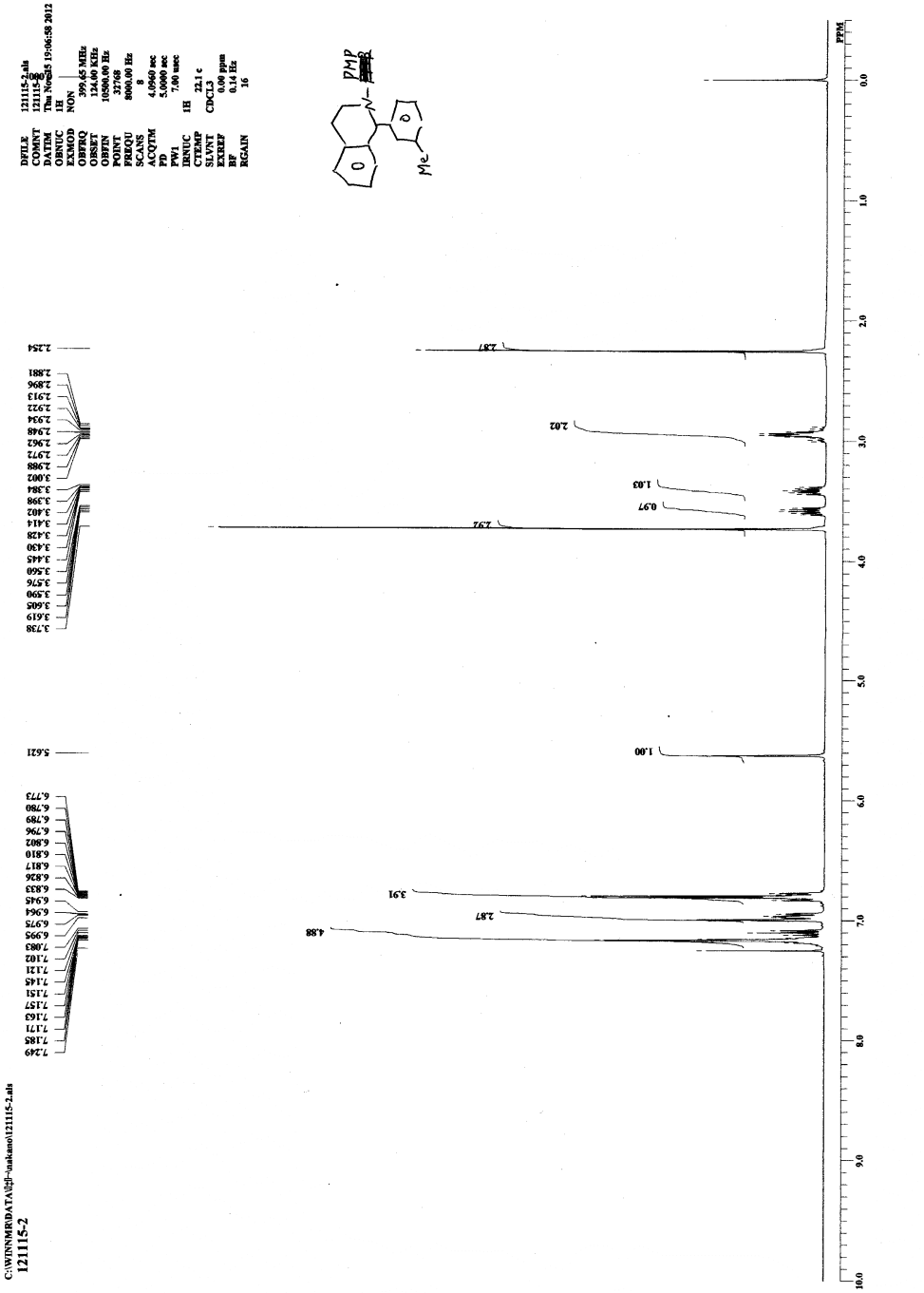

C:\WINNMR\DATA\tnh~\nakano\130124-13C.als
120124-13C
144.3764
139.3520
138.8512
134.8018
129.6481
128.8695
128.6849
128.4461
128.3568
128.1168
127.1865
126.7749
125.8528
125.5894
77.3211
77.0000
76.6789
68.8414
58.8222
47.3882
29.3092
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0

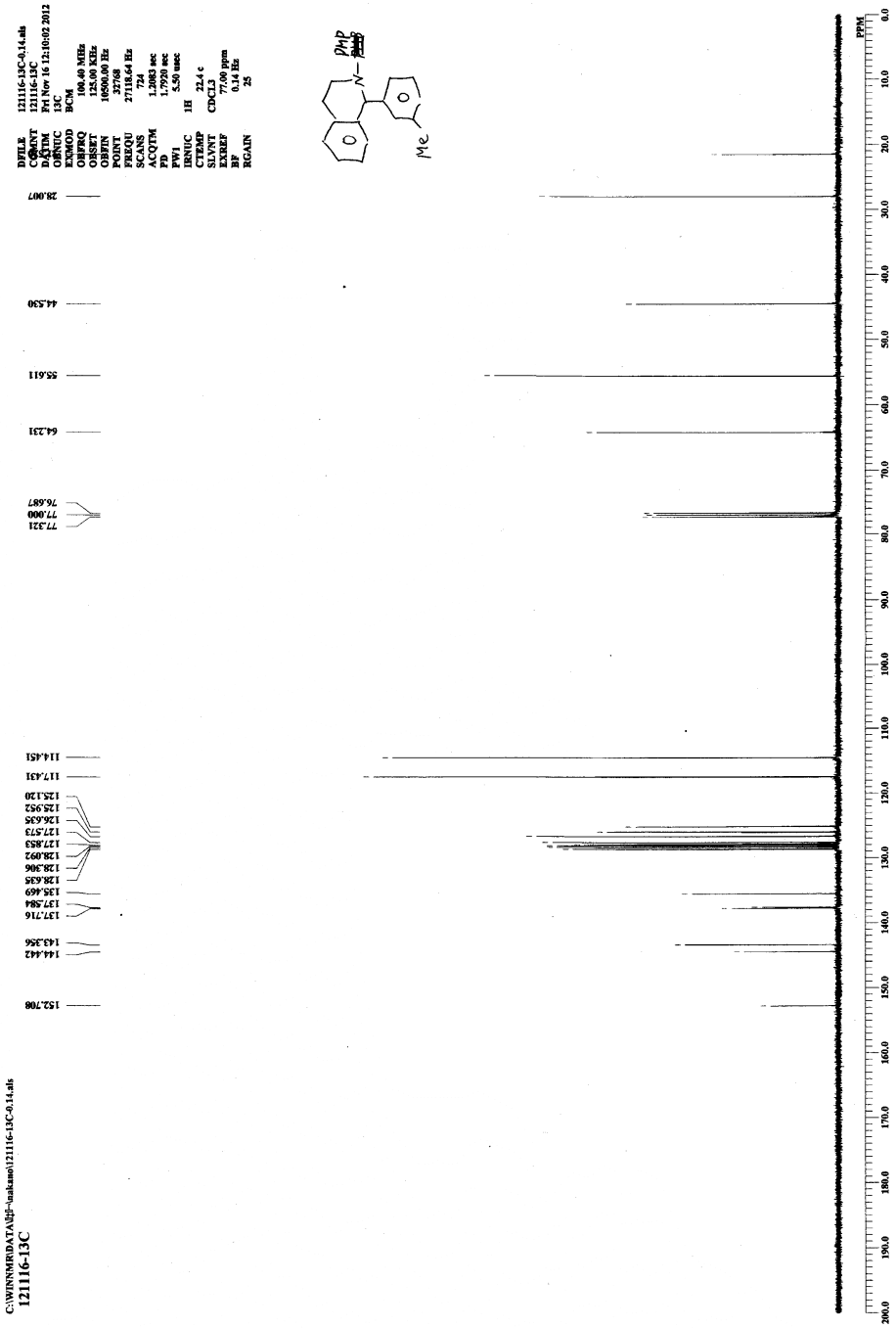
1-(2-Tolyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (4)



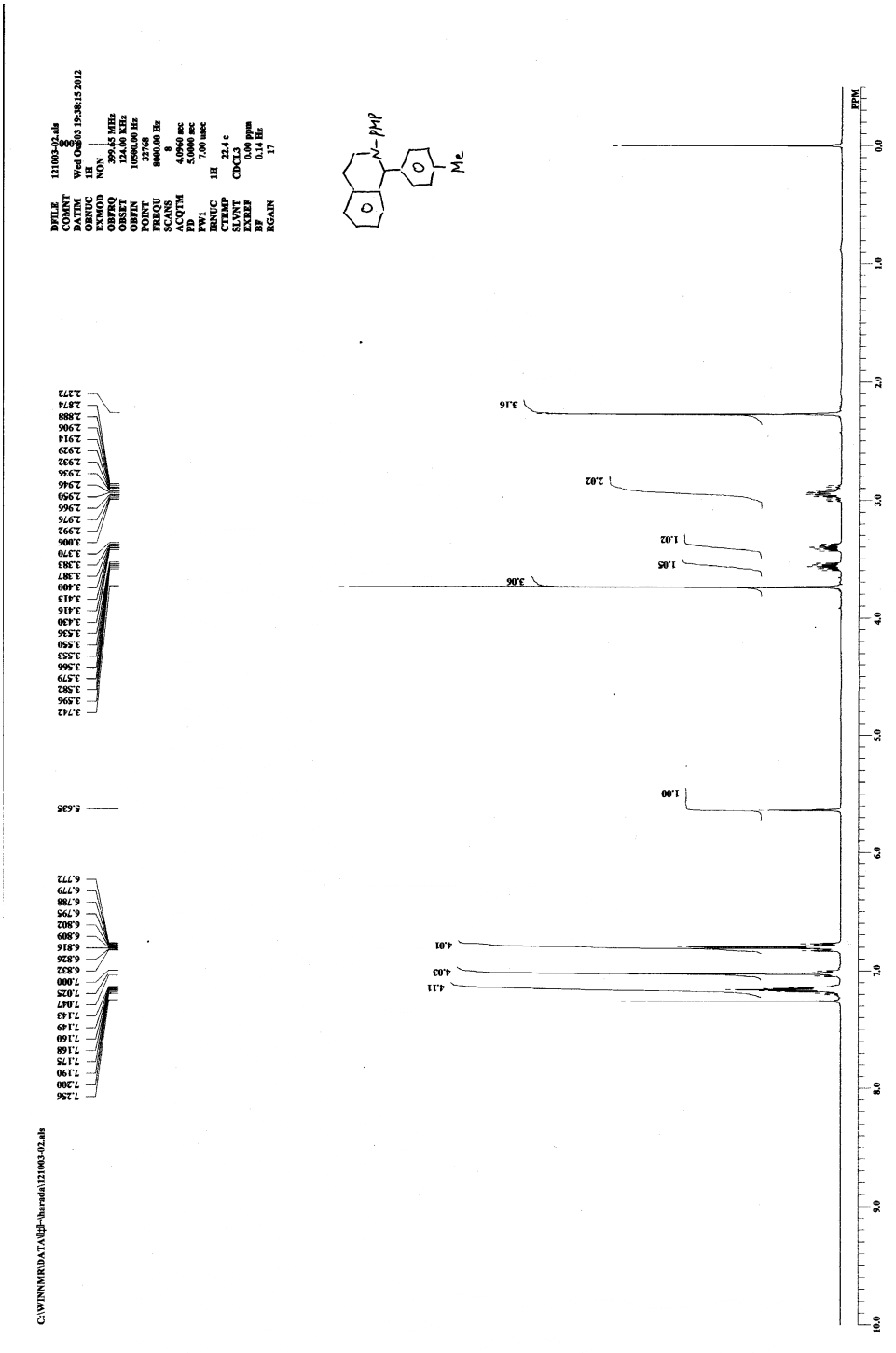

C:\WINNMR\DATA\@2*-nakano\130111-13C.als
130111-13C
DFILE 130111-13C.als
COMNT 130111-13C
DATIM Fri Jan 11 10:12:26 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 361
ACQTM 1.2090 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.44 Hz
RGAIN 25
PMP
Me
36.328
46.045
55.397
61.506
76.679
77.000
77.321
114.171
121.292
123.153
125.597
126.350
126.890
128.504
128.874
130.052
130.504
133.938
136.918
137.741
142.244
144.714
154.116
PPM
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0

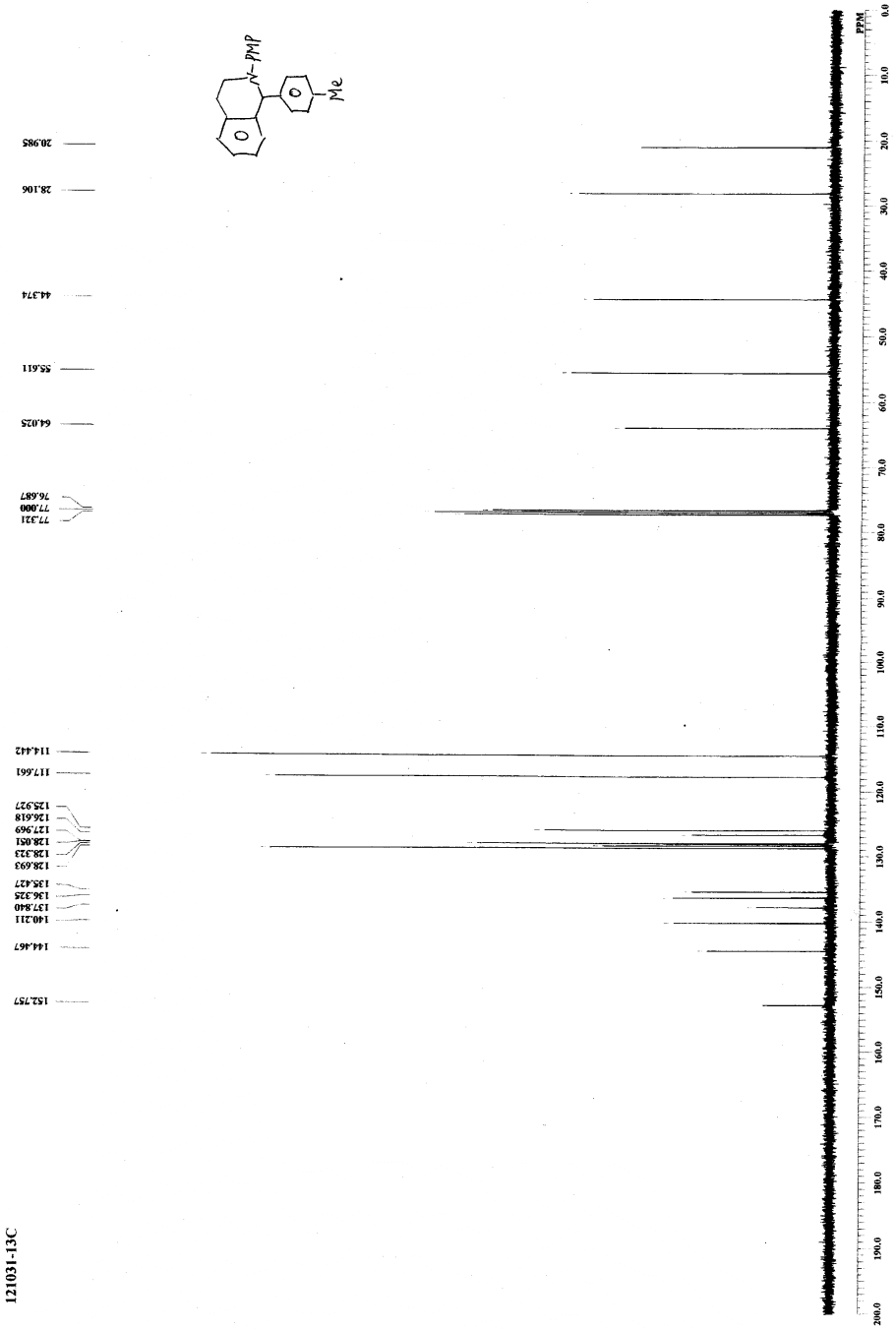
1-(3-Tolyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (5)



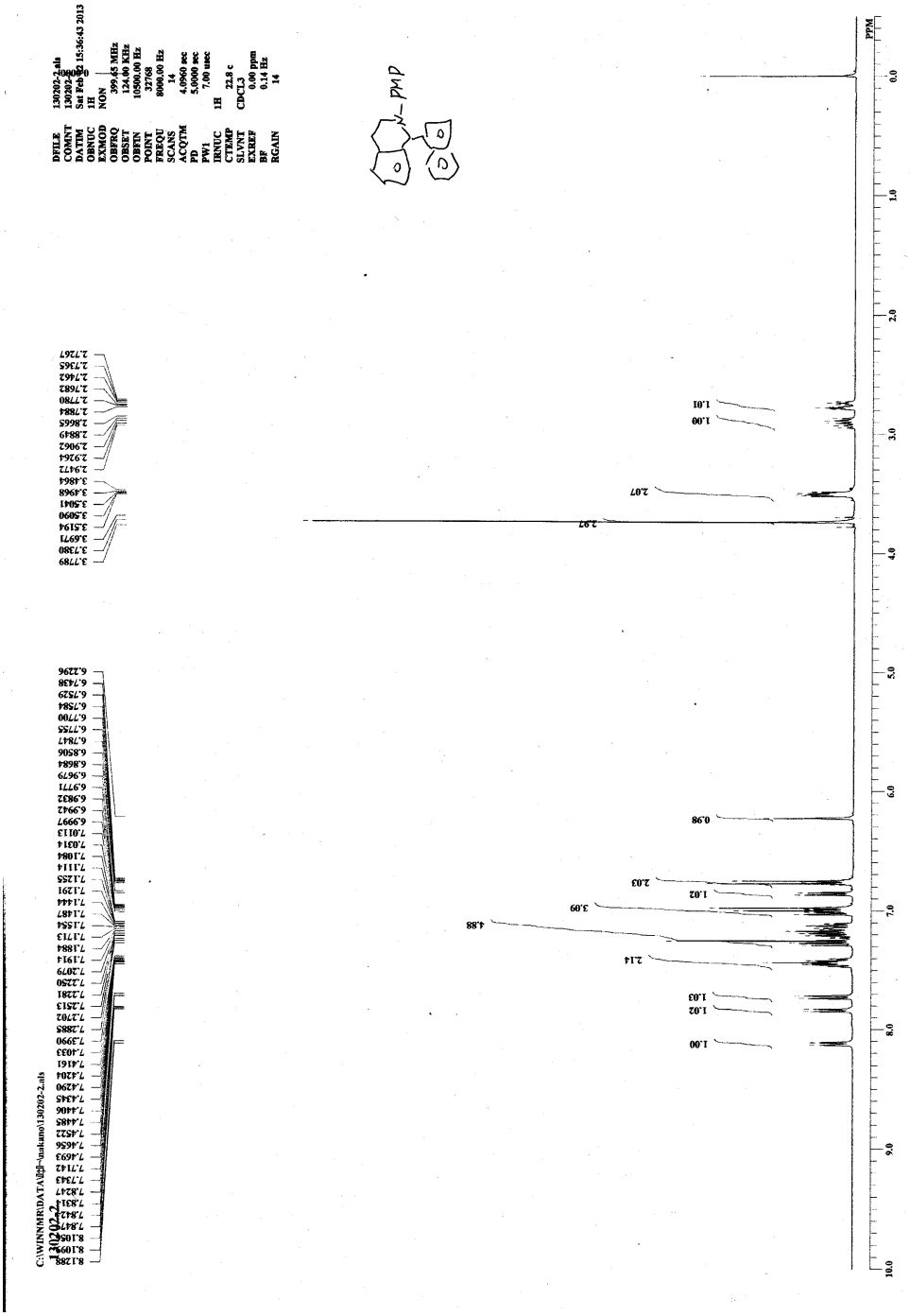

DFILE 121116-13C-0.14.als
COMNT 121116-13C
DATIM Fri Nov 16 12:10:02 2012
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 MHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 724
ACQTM 1.2083 sec
PD 1.7939 sec
PW1 5.50 usec
IRNUC 1H 22.4 c
CTEMP
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.14 Hz
RGAIN 25
28.007
44.530
55.611
64.231
76.687
77.000
77.321
114.451
117.431
125.120
125.952
126.635
127.573
127.853
128.092
128.366
128.635
135.469
137.584
137.716
143.356
144.442
152.708
Me
PPM
C:\WINNMRDATA\hjf-nakano\121116-13C-0.14.als
121116-13C

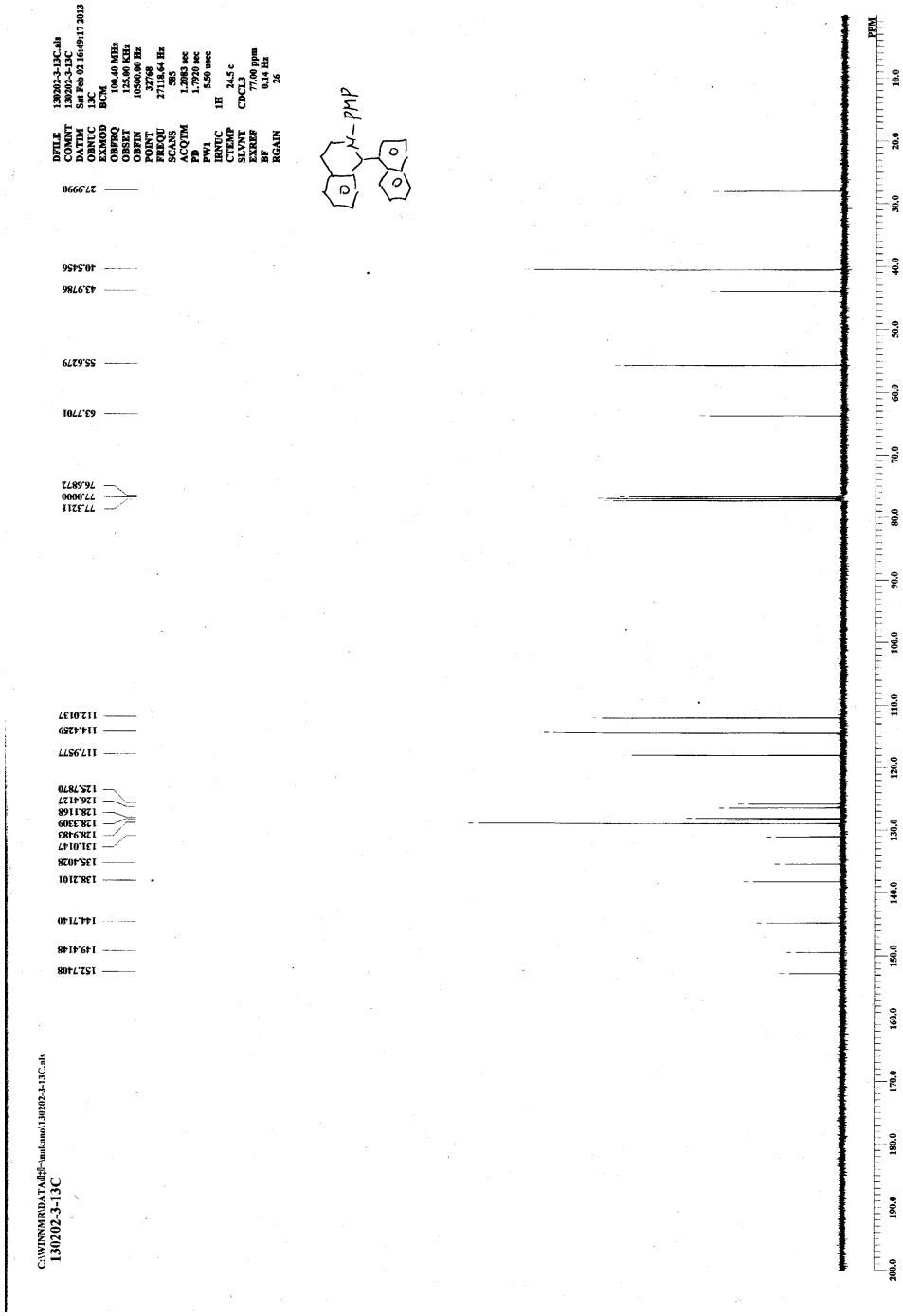
1-(4-Tolyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (6)



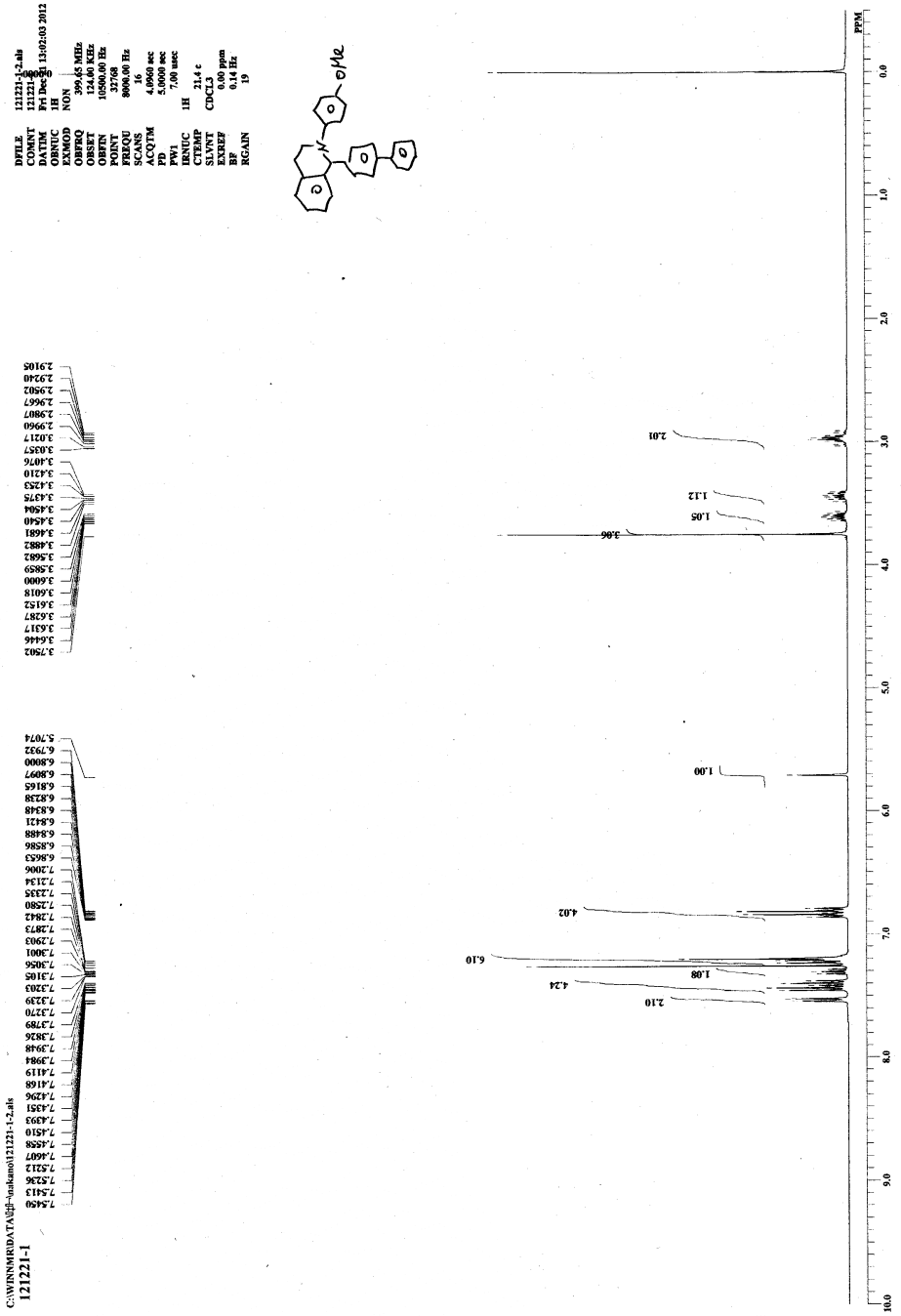

121031-13C
N-PMP
Me
20.985
28.106
44.374
55.611
64.025
76.687
77.000
77.321
114.442
117.661
125.927
126.618
127.969
128.051
128.323
128.693
135.427
136.325
137.840
140.311
144.467
152.757
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0

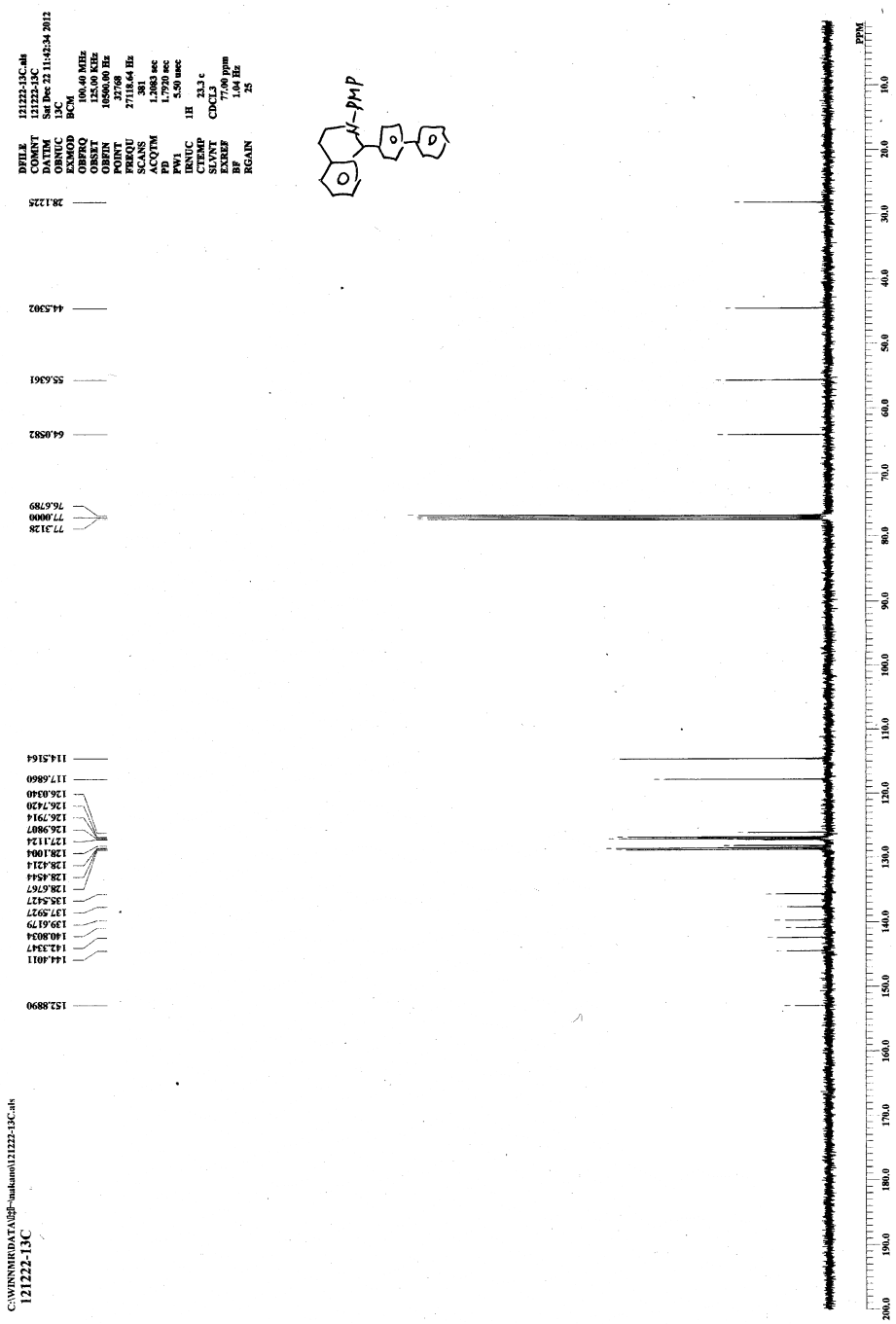
1-(1-Naphthyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (7)



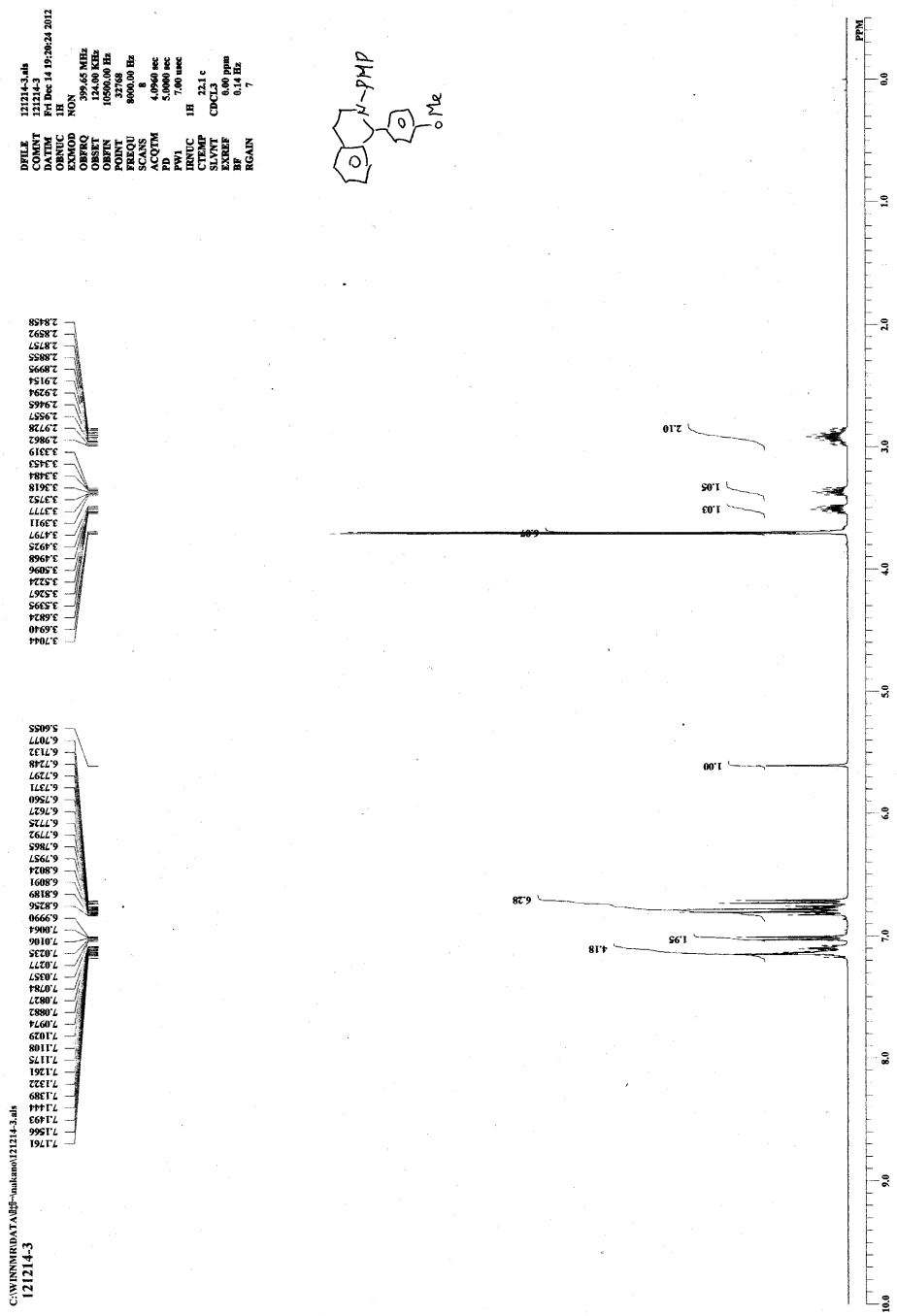

C:\WINNMR\DATA\data\imikano\130202-3-13C.als
130202-3-13C
DFILE 130202-3-13C.als
COMNT 130202-3-13C
DATIM Sat Feb 02 16:49:17 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.60 MHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 585
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.14 Hz
RGAIN 26
N-PMP
27.9990
40.5456
43.9786
55.6279
63.7701
76.6872
77.0000
77.3211
112.0137
114.4259
117.9577
125.7870
126.4127
128.1168
128.3309
128.9485
131.0147
135.4028
138.2101
144.7140
149.4148
152.7408
PPM

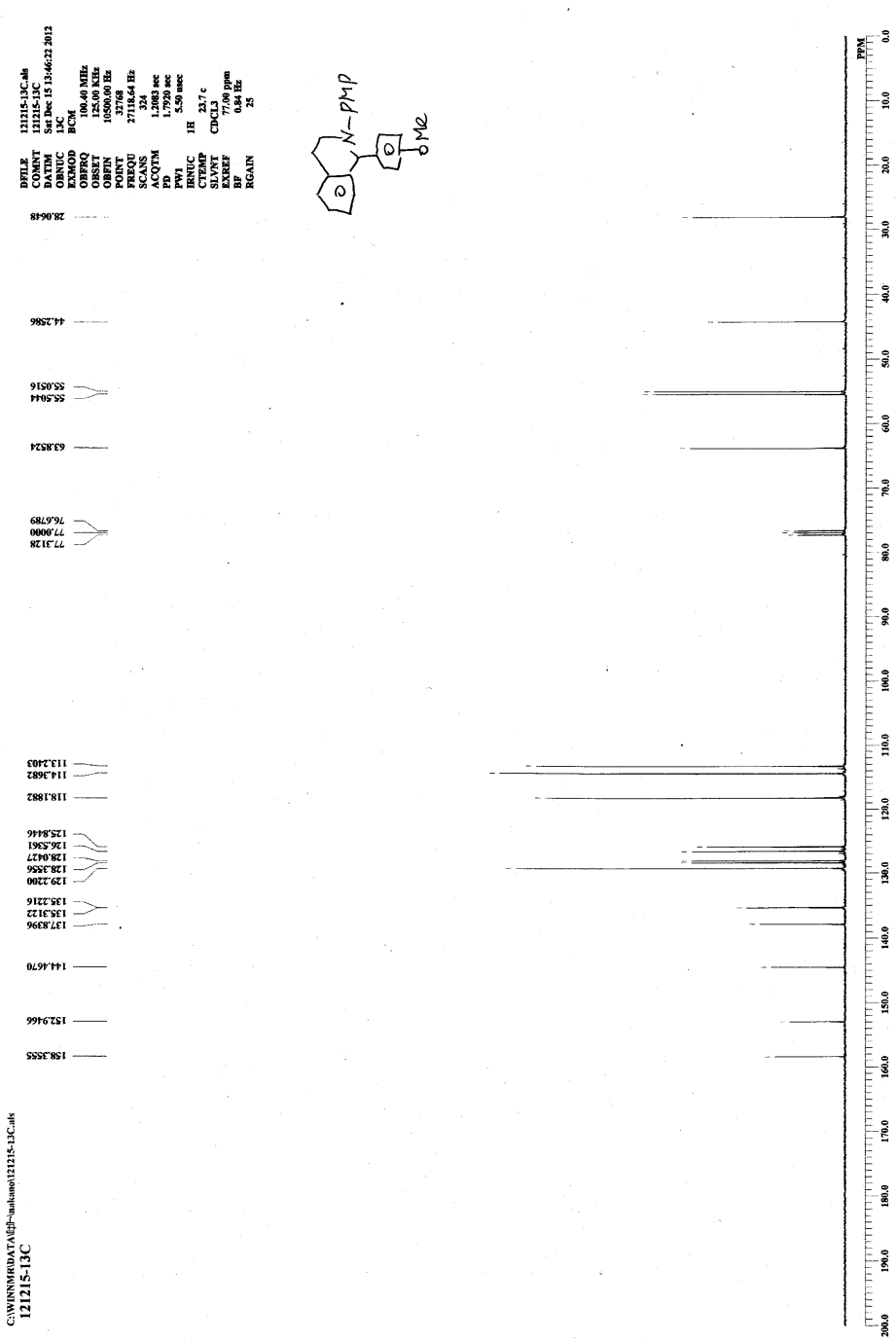
1-(4-Biphenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (8)



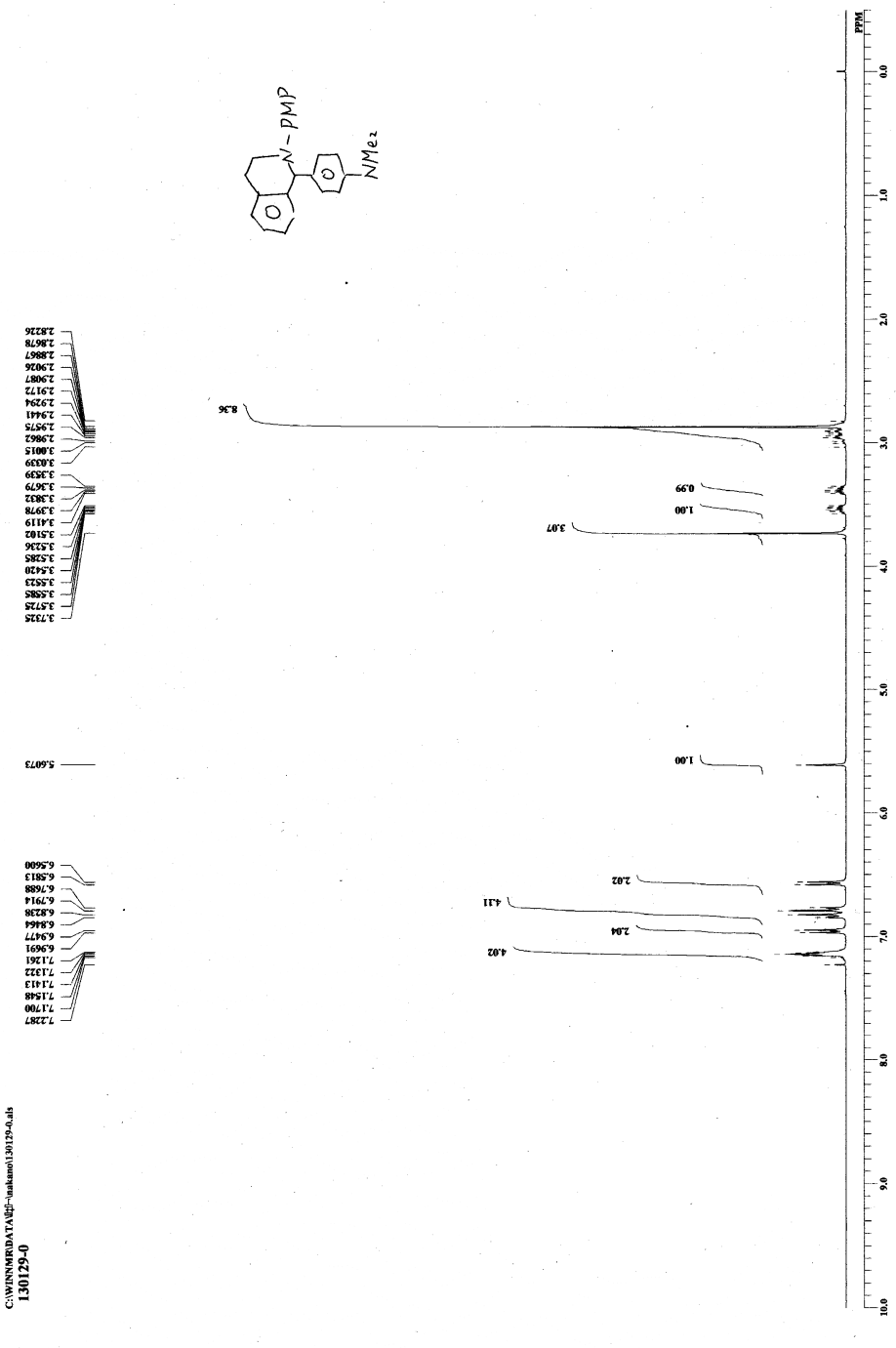

C:\WINNMR\DATA\dpf-tsukano\121222-13C.als
121222-13C
DFILE 121222-13C.als
COMNT 121222-13C
DATIM Sat Dec 22 11:42:34 2012
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 25.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 381
ACQTM 1.2083 sec
FD 1.7920 sec
PW1 5.50 usec
IRNUC 1H 23.3 c
CTEMP
SLVNT CDCl3
EXREF 77.00 ppm
BF 1.04 Hz
RGAIN 25
28.1225
44.5302
55.6361
64.0582
76.6789
77.0000
77.3128
114.5164
117.6860
126.0340
126.7420
126.7914
126.9807
127.1124
128.1064
128.4214
128.4544
128.6767
135.5427
137.5927
139.6179
140.8034
142.3347
144.4011
152.8890
PPM
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0

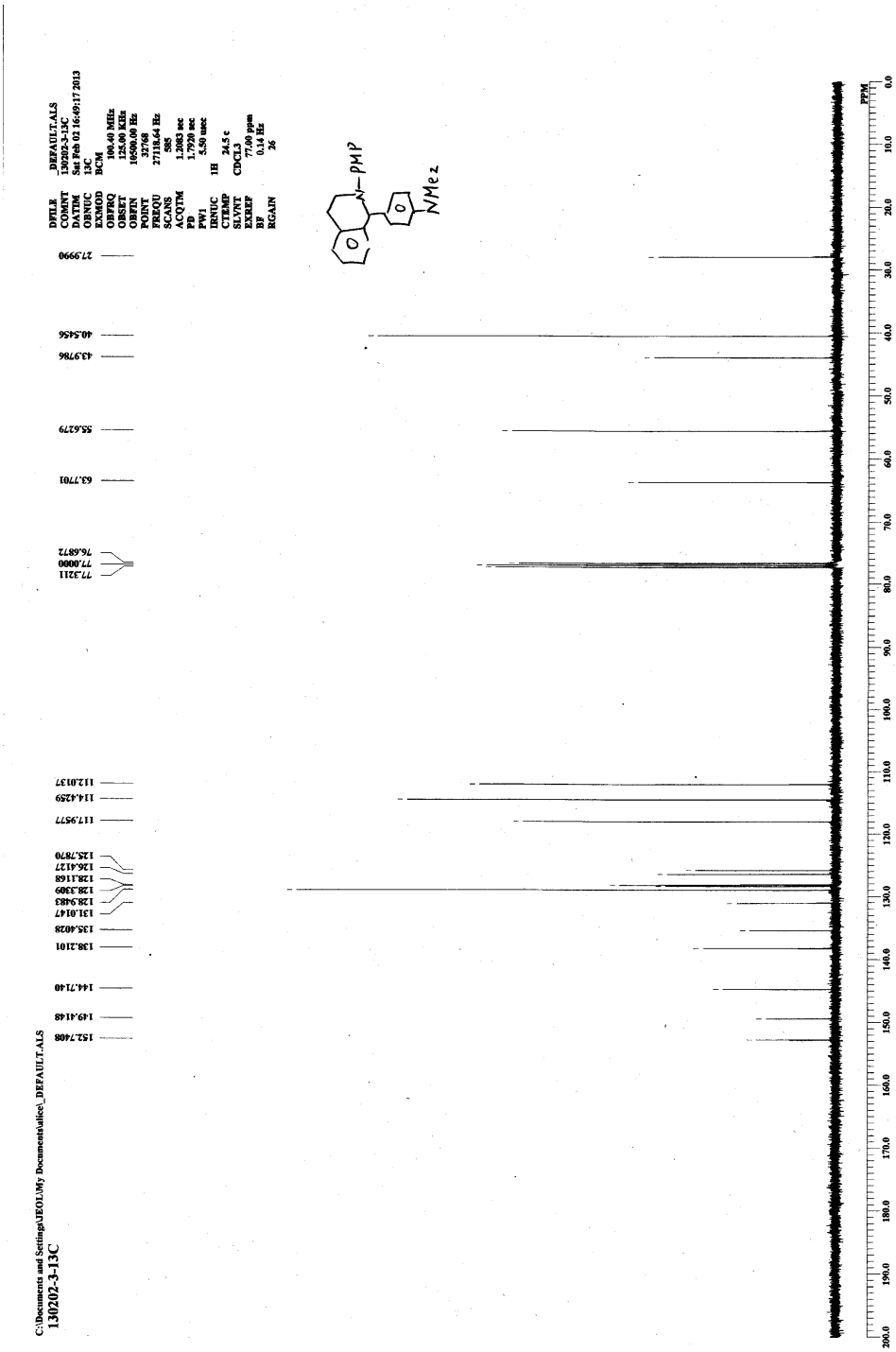
1-(4-Methoxyphenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (9)



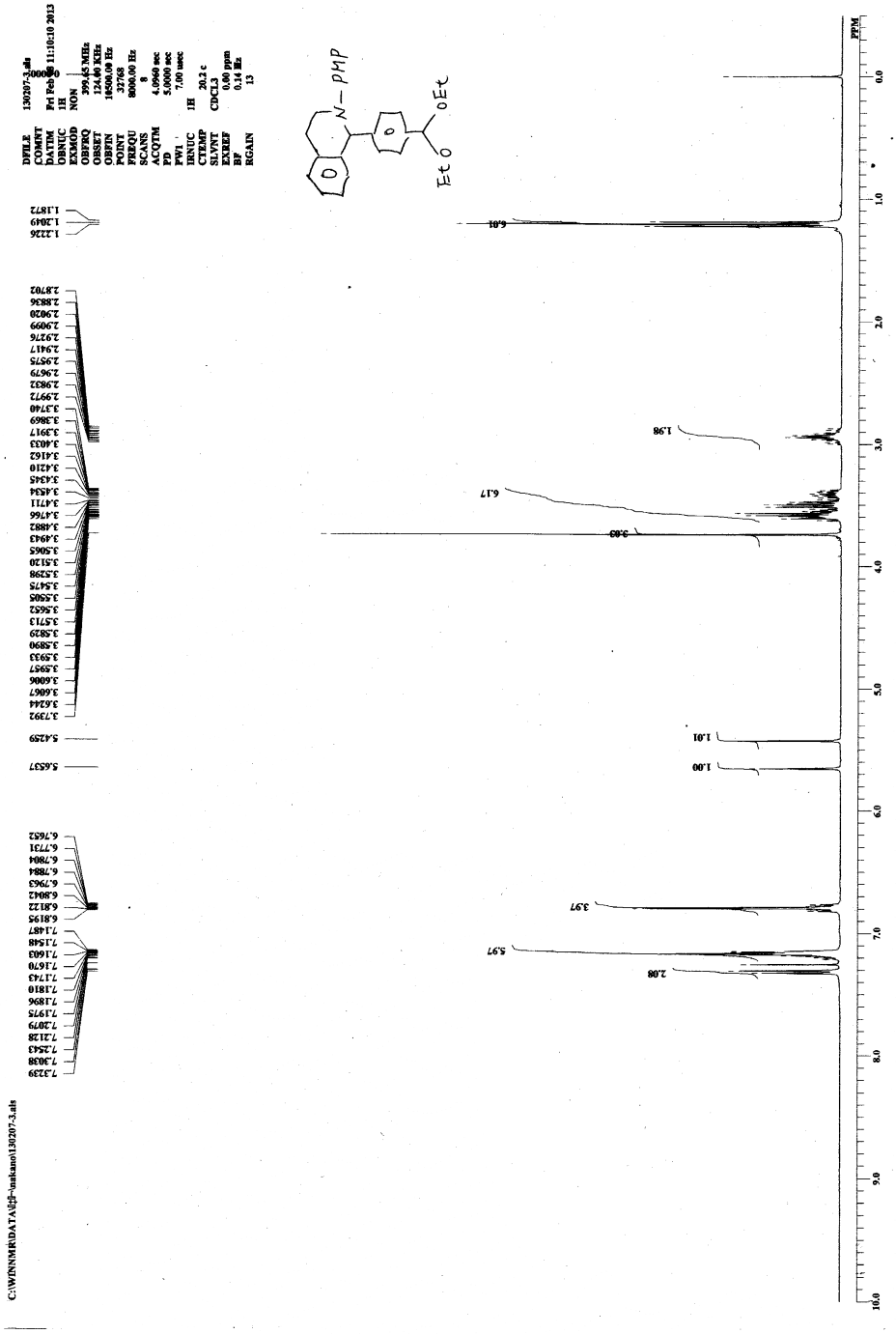

C:\WINNMR\DATA\郭-tsuhano\121215-13C.als
121215-13C
DFILE 121215-13C.als
COMNT 121215-13C
DATIM Sat Dec 15 13:46:22 2012
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 25.00 KHz
OBFIN 100400.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 324
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H 23.7 c
CTEMP
SLVNT CDCl3
EXREF 77.00 ppm
BF 0.84 Hz
RGAIN 25
28.0648
44.2586
55.0516
55.5044
63.8524
76.6789
77.0000
77.3128
113.2403
114.3682
118.1882
125.8446
126.5361
128.0427
128.3556
129.2280
135.2216
135.3122
137.8396
144.4670
152.9466
158.3555
N-PMP
OMe
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0

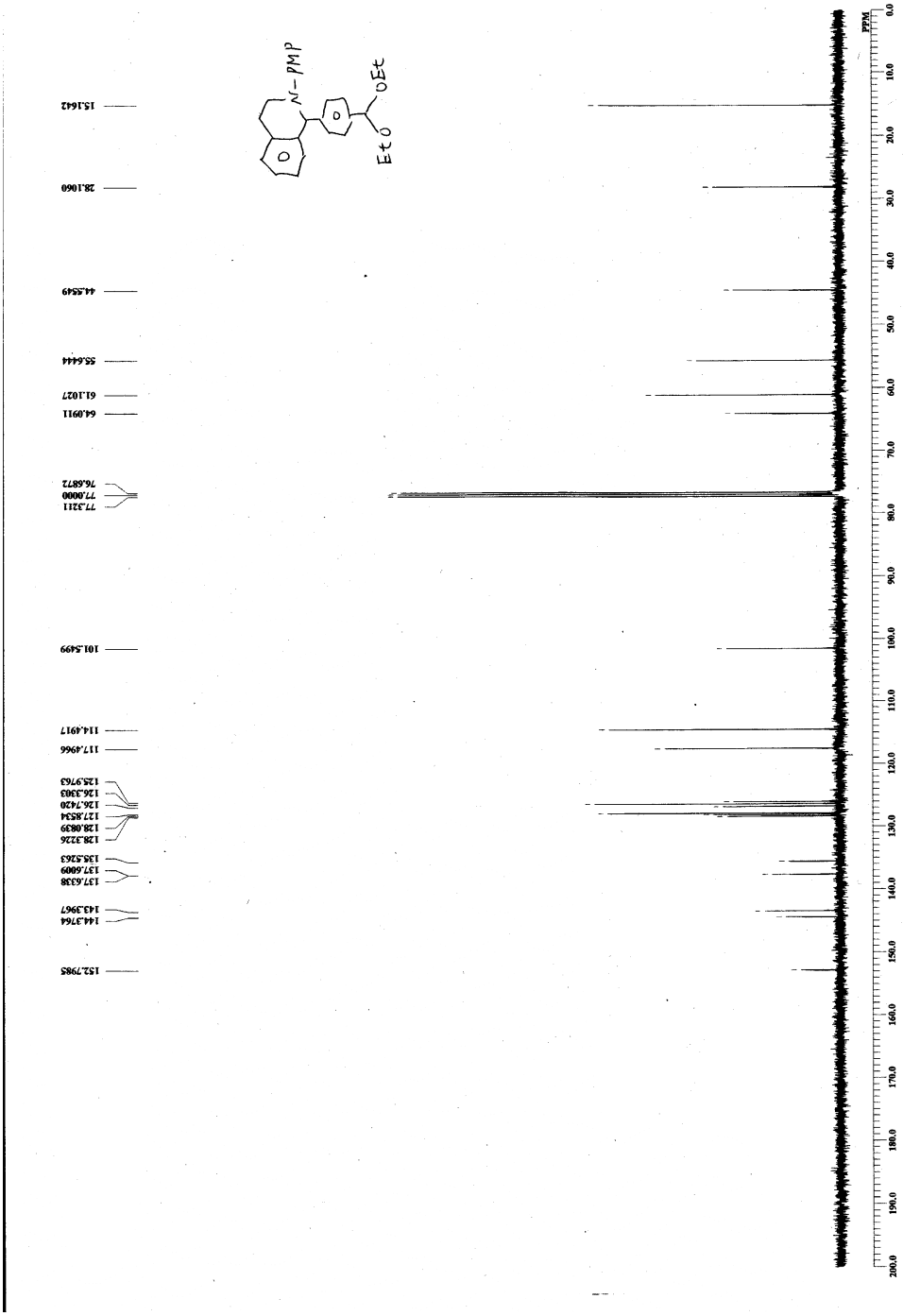
1-(4-*N*,*N*-dimethylaminoaniline)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (10)



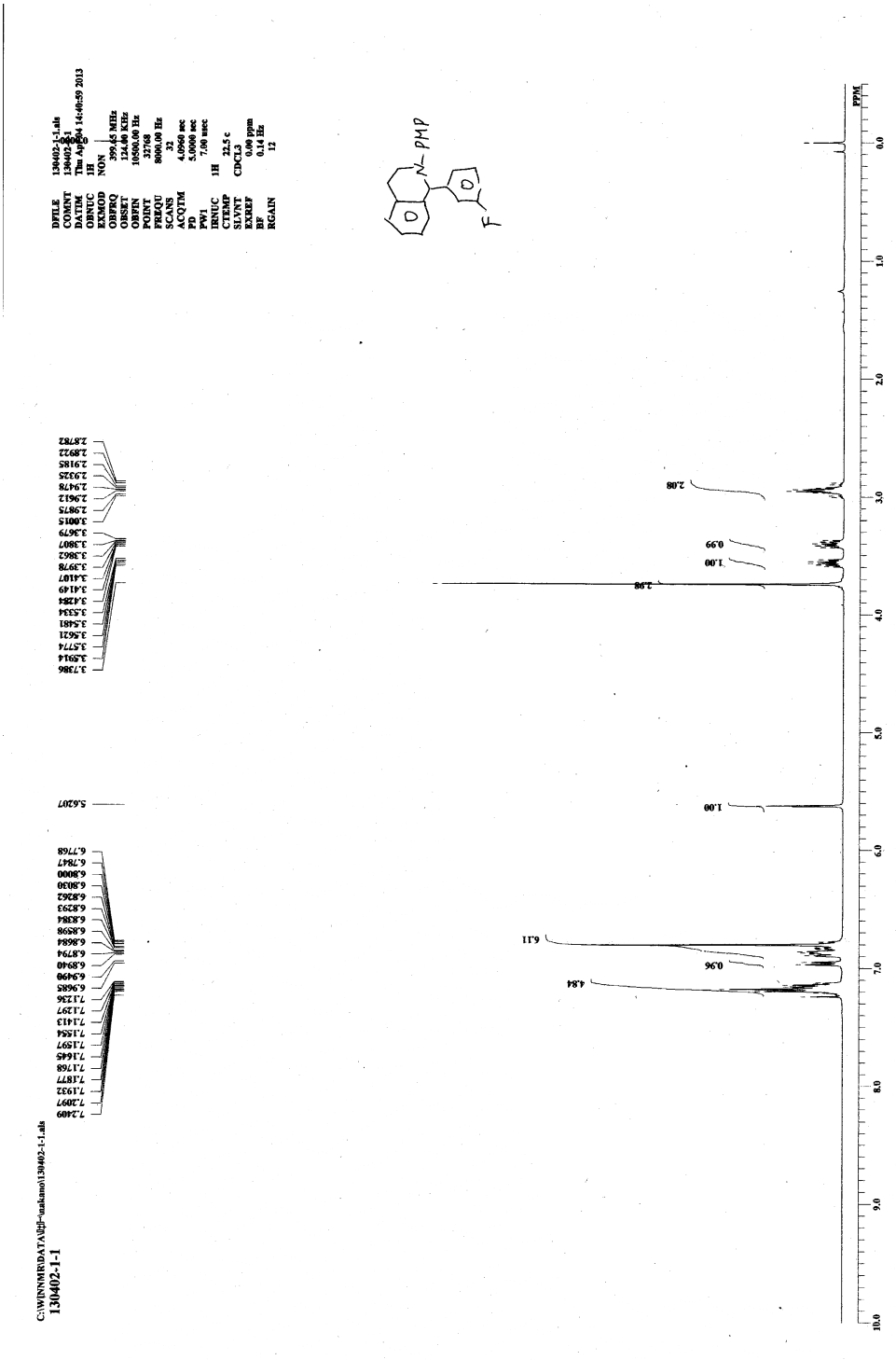

DFILE _DEFAULT.ALS
COMNT 130202-3-13C
DATIM Sat Feb 02 16:49:17 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
SWWID 125.00 KHz
ORSET 16500.00 Hz
OBFIN 32768
POINT 27118.64 Hz
FREQU 585
SCANS 1.2083 sec
ACQTM 1.7050 sec
PD 4.50 sec
PW1
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.14 Hz
RGAIN 26
27.9990
40.5456
43.9786
55.6279
63.7701
76.6872
77.3211
77.0000
112.0137
114.4259
117.9577
125.7870
126.4127
128.1168
128.3309
128.9483
131.0147
135.4028
138.2101
144.7140
149.4148
152.7408
PPM
C:\Documents and Settings\JEOL\My Documents\alice_DEFAULT.ALS
130202-3-13C

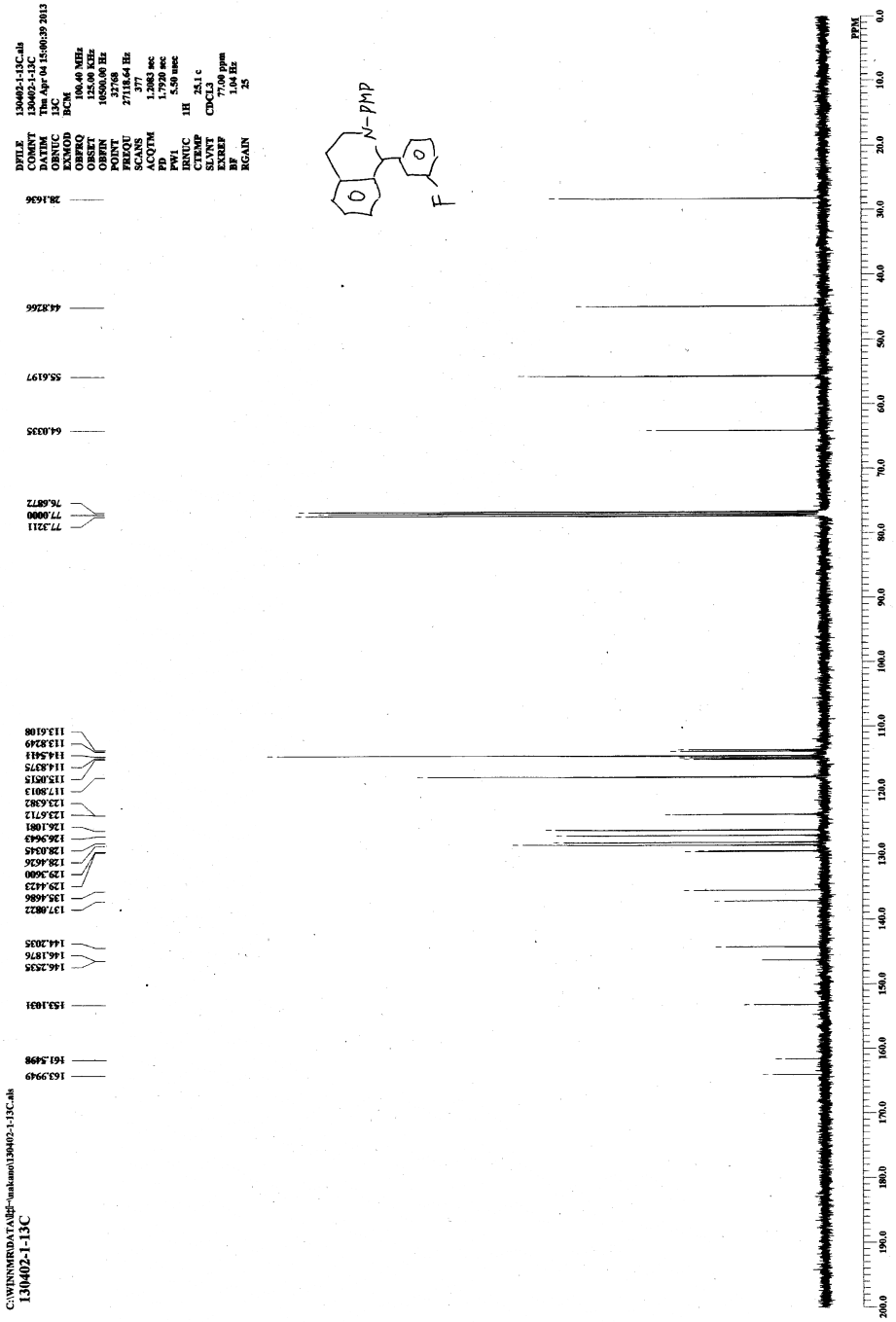
1-(4-benzaldehyde dimethylacetal)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (11)



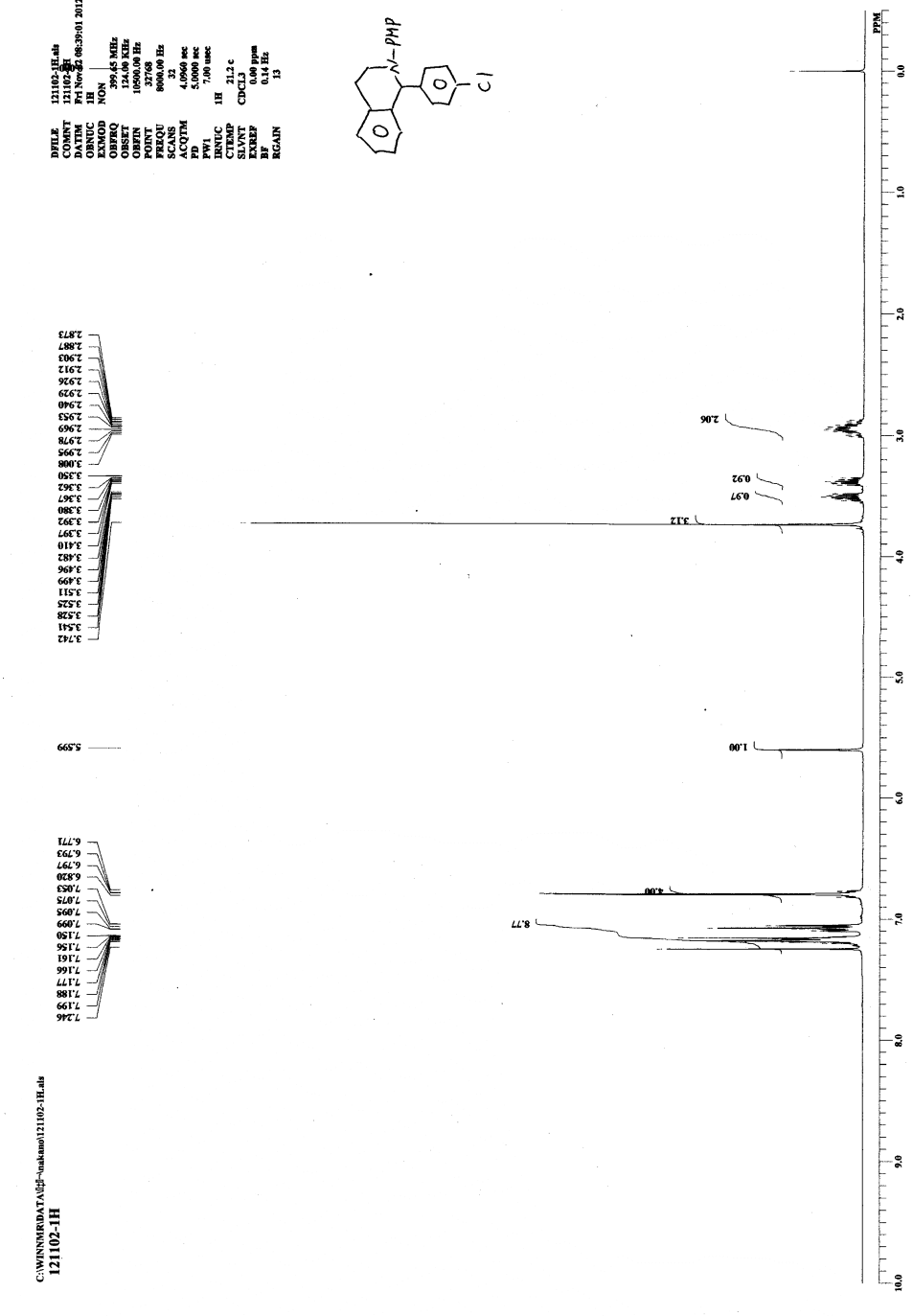

N-PMP
EtO
OEt
15.1642
28.1060
44.5549
55.6444
61.1027
64.0911
76.6872
77.0000
77.3211
101.5499
114.4917
117.4966
125.9763
126.3303
126.7420
127.8534
128.0839
128.3726
135.5763
137.6009
137.6338
143.3967
144.3764
152.7985
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0

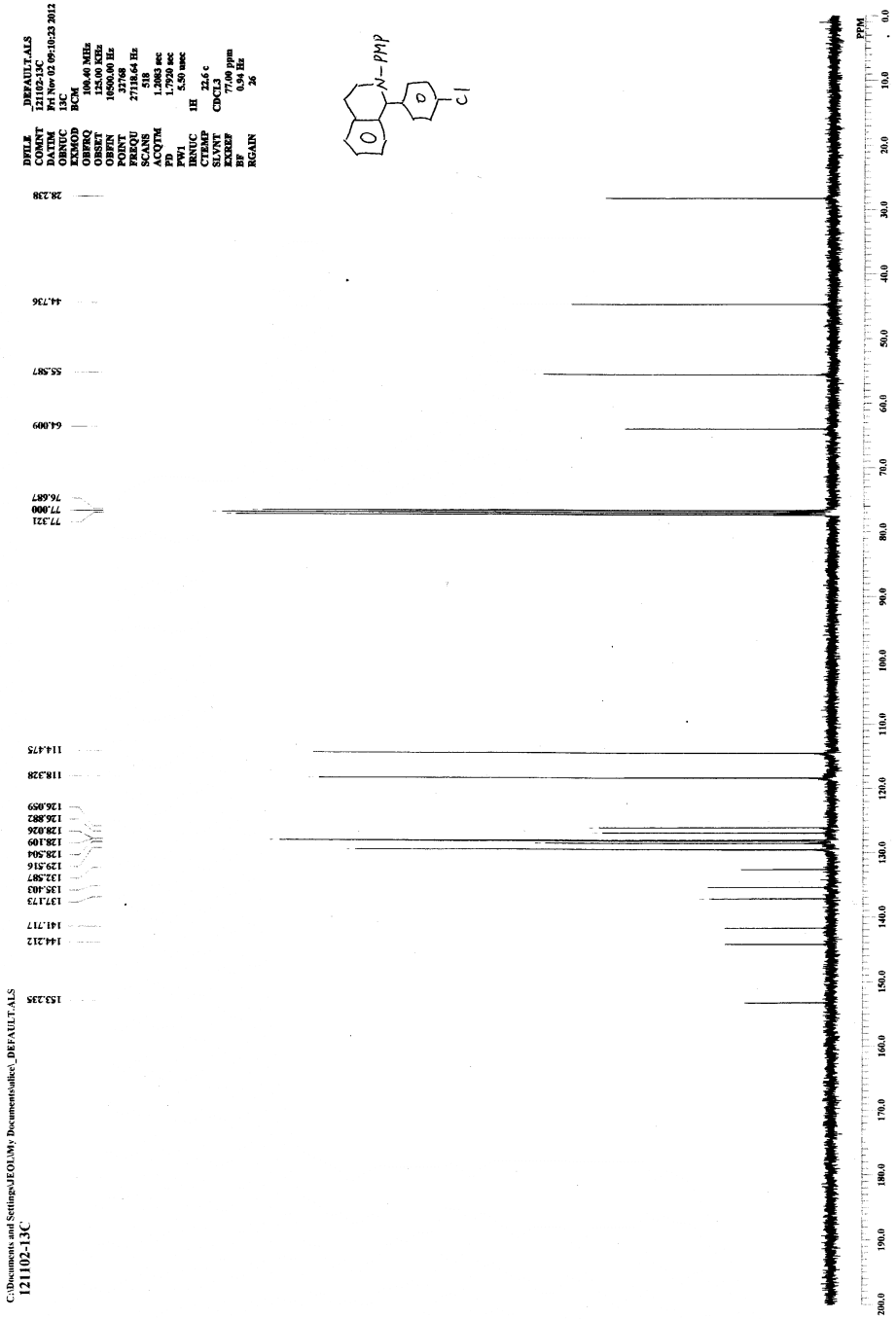
1-(3-Fluorophenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (12)



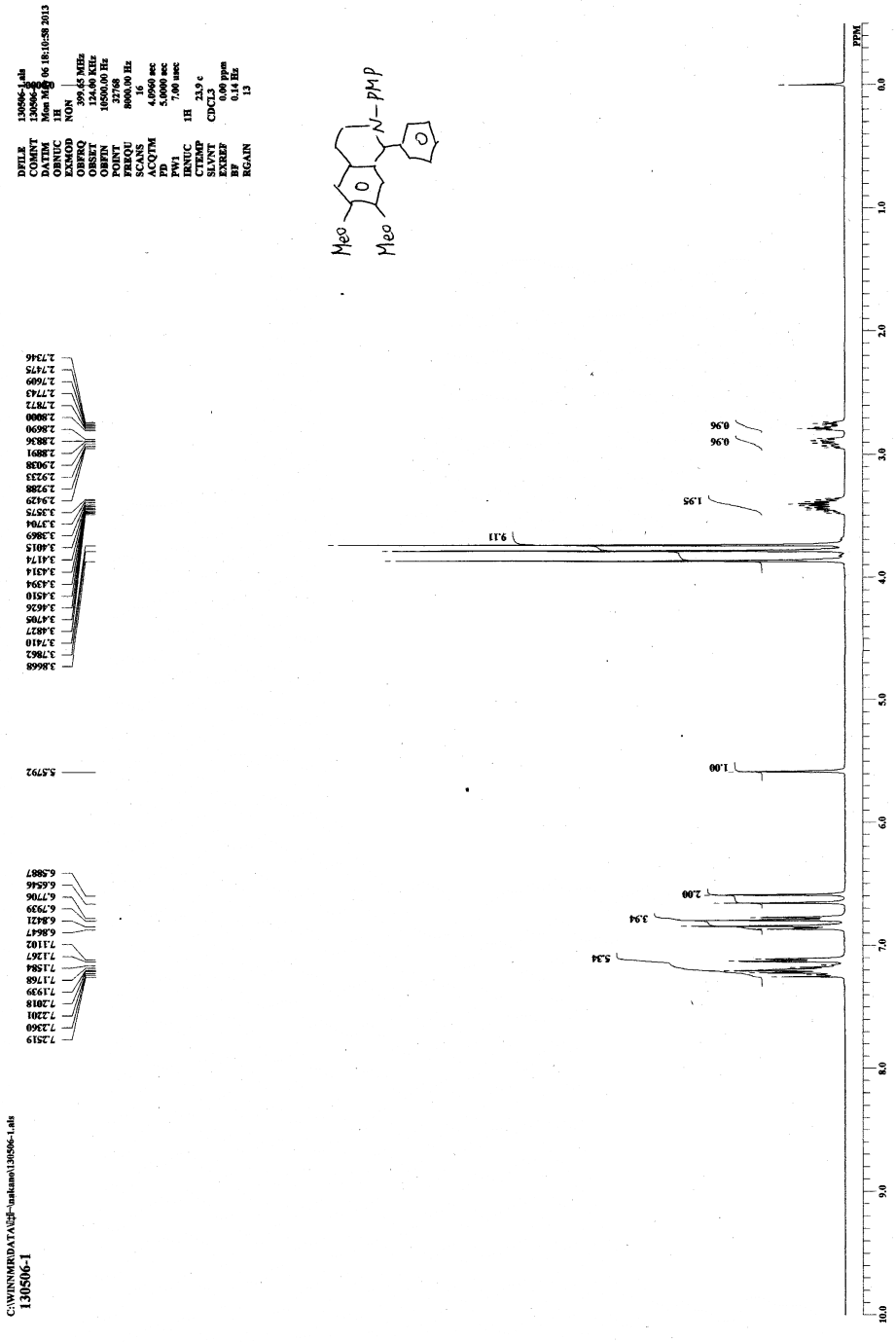

C:\WINNMR\DATA\用户\tsukano\130402-1-13C.als
130402-1-13C
DFILE 130402-1-13C.als
COMNT 130402-1-13C
DATIM Thu Apr 04 15:00:39 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 377
ACQTM 1.2083 sec
PD 1.7500 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 25.1 c
SLVNT CDCl3
EXREF 77.00 ppm
RF 1.04 Hz
RGAIN 25
28.1636
44.8766
55.6197
64.0335
76.6872
77.0000
77.3211
113.6108
113.8249
114.5611
114.8375
115.0515
117.8013
123.6382
123.6712
126.1081
126.9643
128.0545
128.4636
129.3600
129.4423
135.4686
137.0822
144.2035
146.1876
146.2535
153.1031
161.5498
163.9949
N-PMP
F
PPM

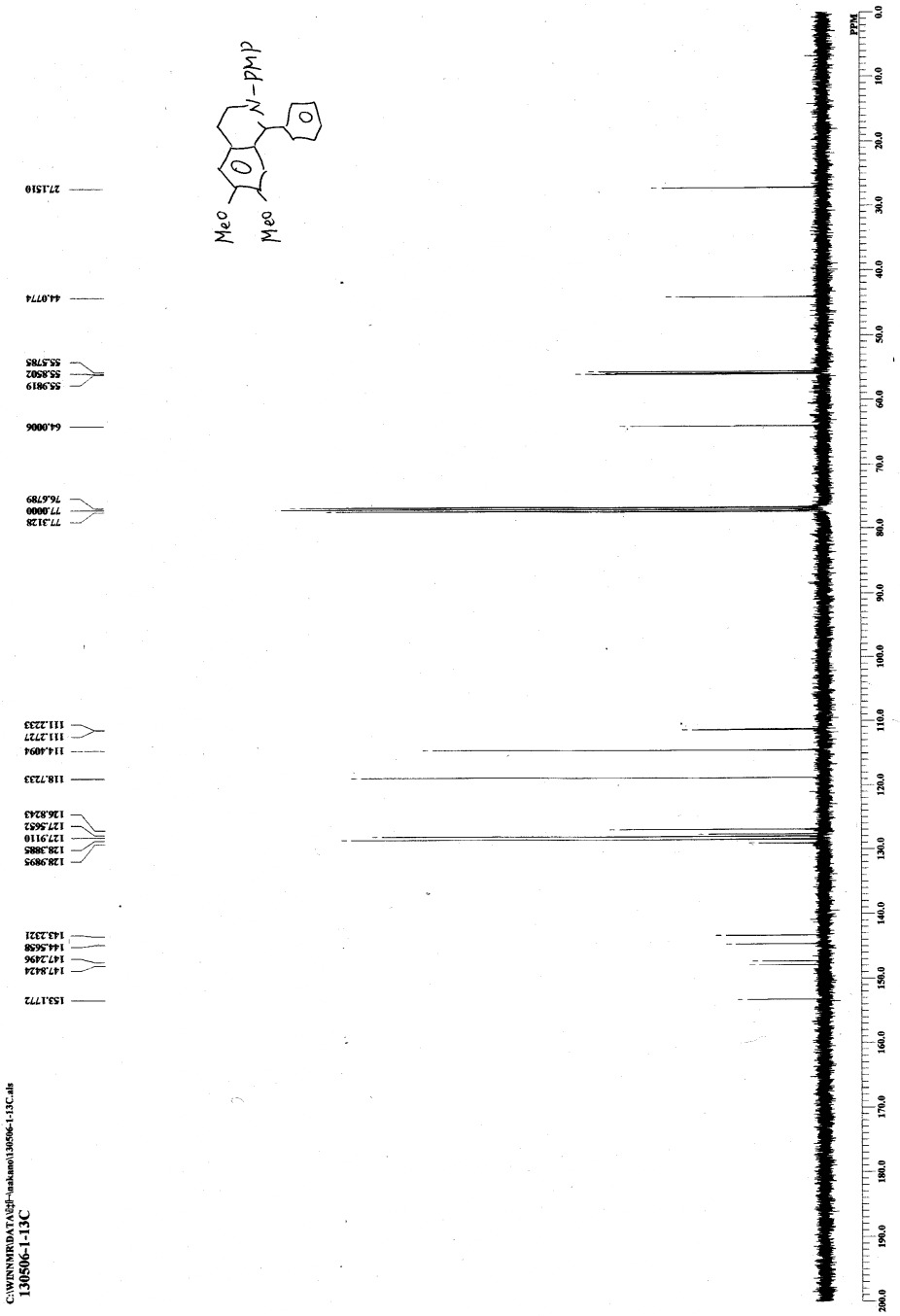
1-(4-Chlorophenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydroisoquinoline (13)



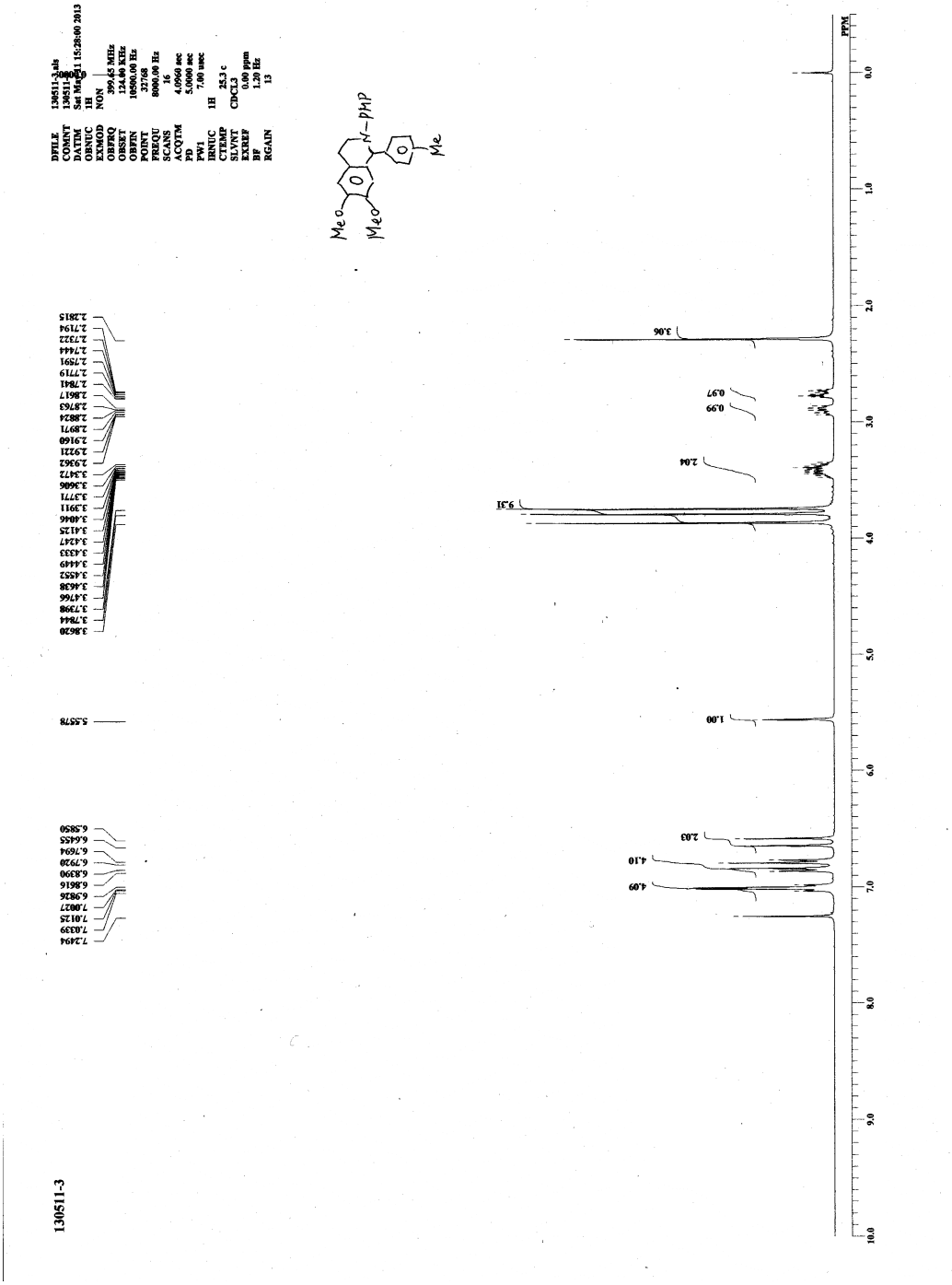

DFILE DEFAULT.ALS
COMNT 121102-13C
DATIM Fri Nov 02 09:10:23 2012
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.60 MHz
OBFIN 16500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 518
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H 22.6 c
CTEMP
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.94 Hz
RGAIN 26
N-PMP
Cl
28.238
44.736
55.587
64.009
76.687
77.000
77.321
114.475
118.328
126.059
126.882
128.026
128.109
128.504
129.516
132.587
135.403
137.173
141.717
144.212
153.385
PPM
C:\Documents and Settings\JEOL\My Documents\salke_DEFAULT.ALS
121102-13C

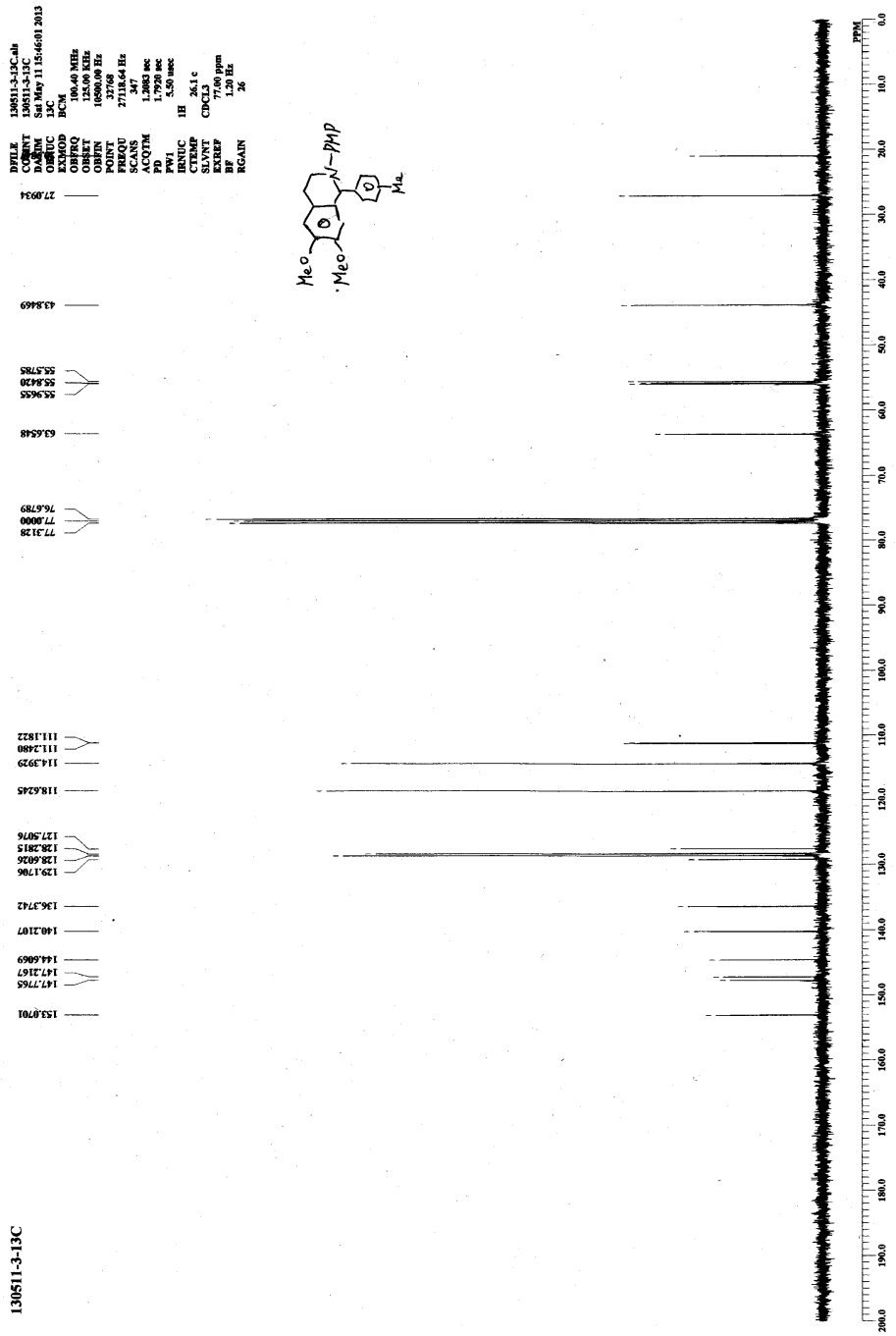
1-Phenyl-2-(4-methoxyphenyl)-1,2,3,4-tetrahydro-6,7-dimethoxyisoquinoline (14)



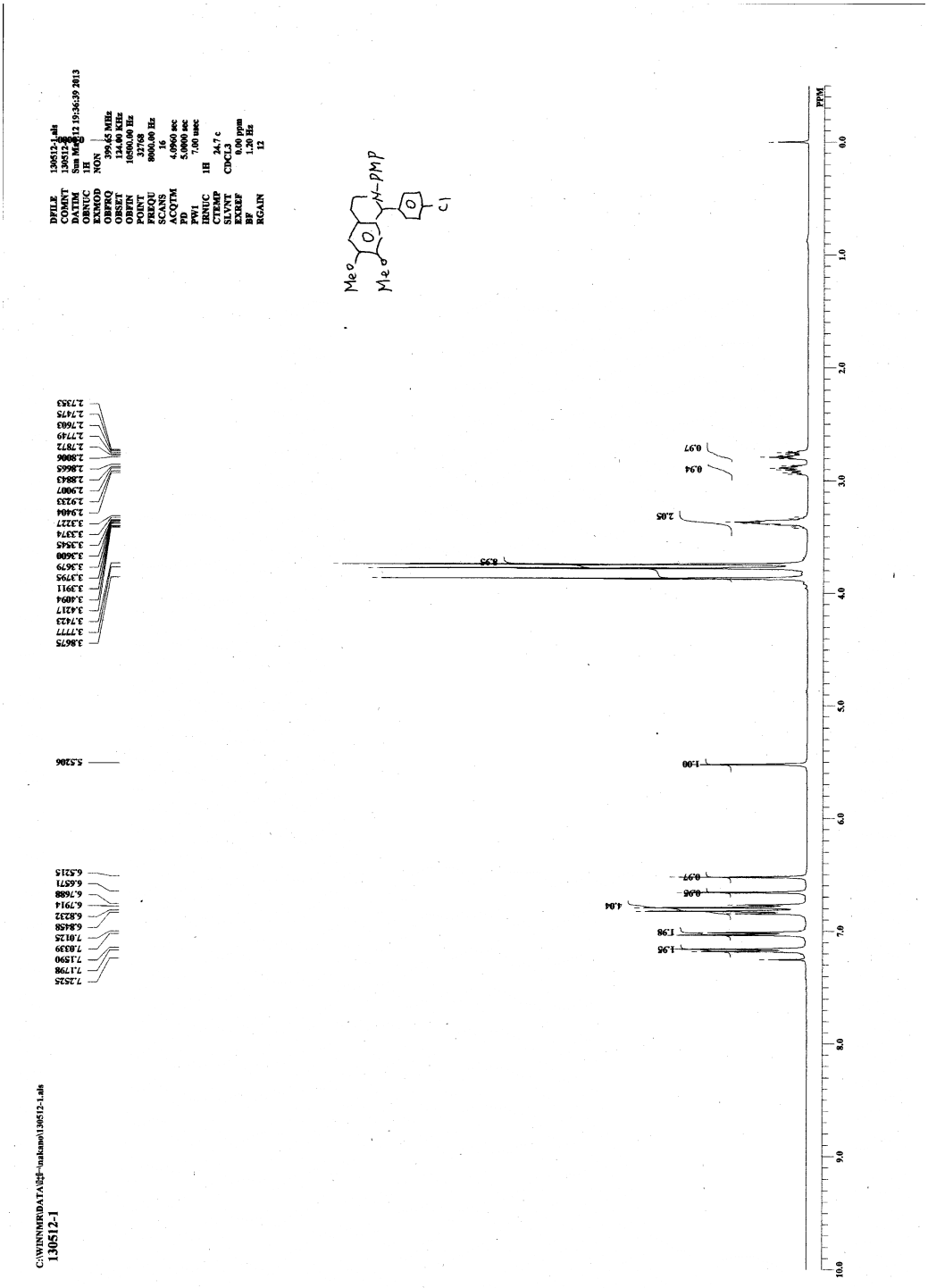

N-PMP
MeO
MeO
27.1510
44.0774
55.5785
55.8502
55.9819
64.0006
76.6789
77.0000
77.3128
111.2333
111.2727
114.4094
118.7233
136.8243
127.5652
127.9110
128.3885
128.9895
143.3321
144.5658
147.2496
147.8424
153.1772
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0
C:\WINNMR\DATA\伊-nakano\130506-1-13C.als
130506-1-13C

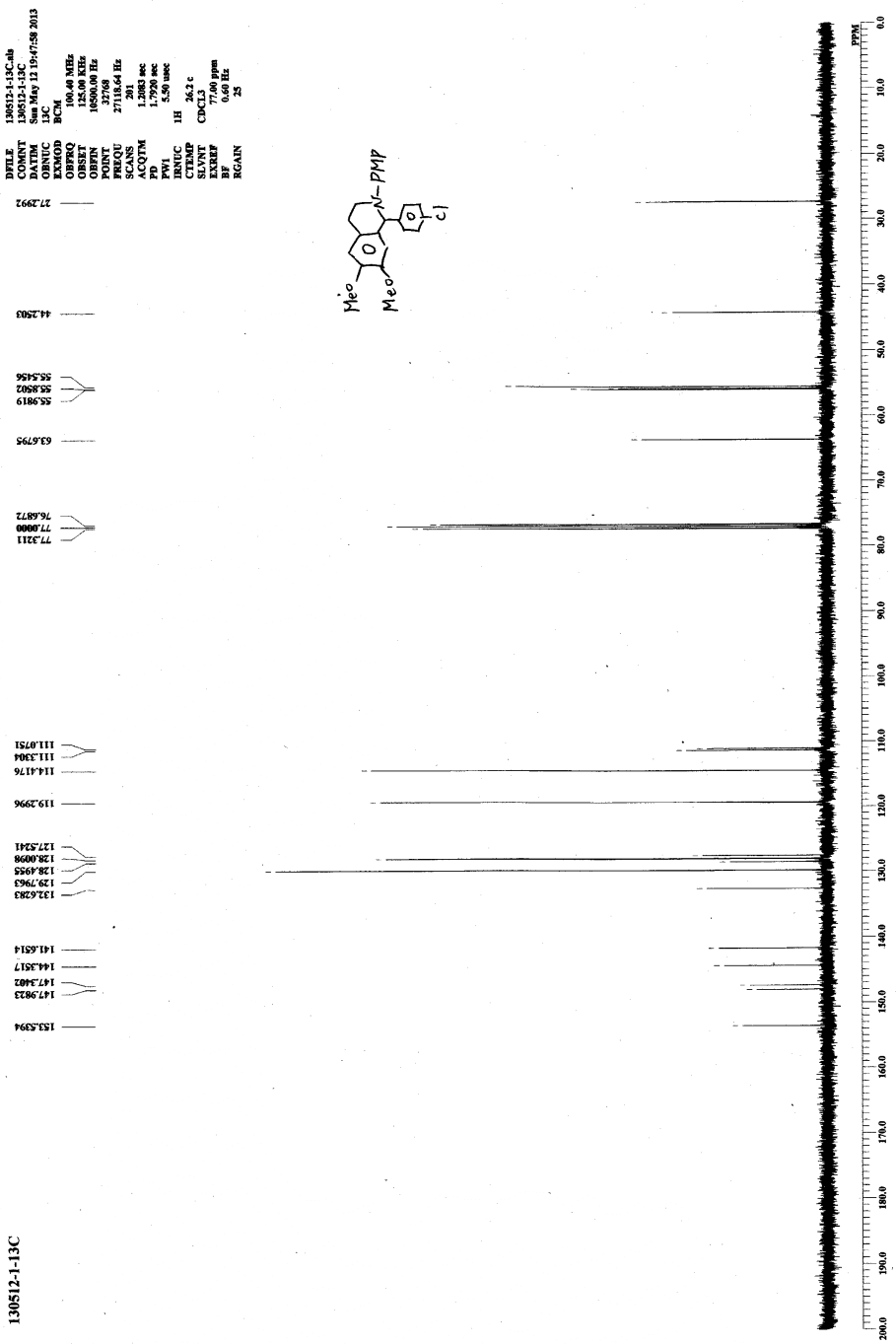
1-(2-Tolyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydro-6,7-dimethoxyisoquinoline (15)



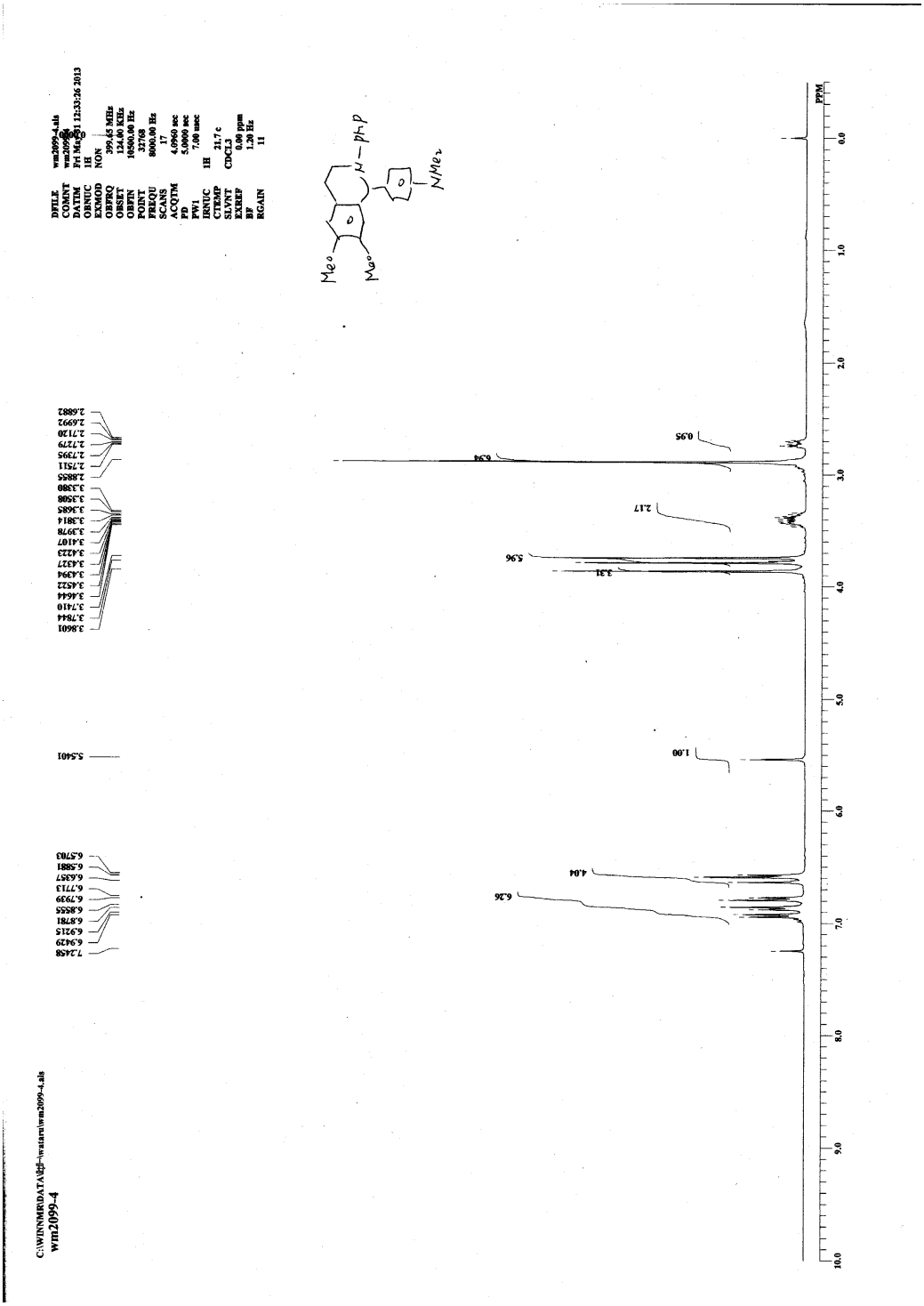

130511-3-13C
DFILE 130511-3-13C.als
COMNT 130511-3-13C
DATIM Sat May 11 15:46:01 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 MHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 347
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H 26.1 c
CTEMP
SLVNT CDCl3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 26
27.0934
43.8469
55.5785
55.8420
55.9655
63.6548
76.6789
77.0000
77.3128
111.1822
111.2480
114.3929
118.6245
127.5076
128.2815
128.6026
129.1706
136.3742
140.2107
144.6069
147.2167
147.7765
153.0701
Me0
Me0
N-PMP
Me
PPM

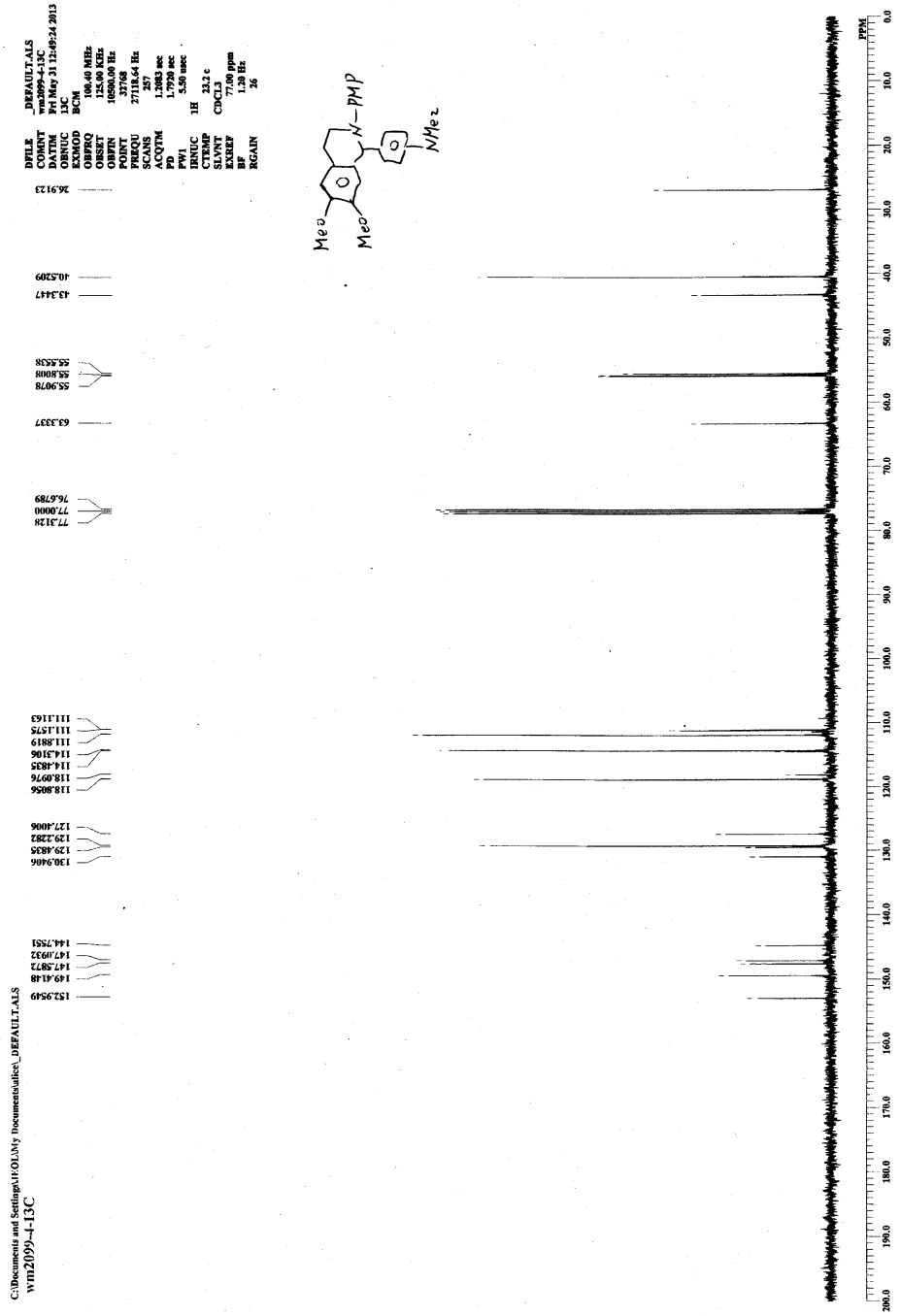
1-(4-Chlorophenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydro-6,7-dimethoxyisoquinoline (16)



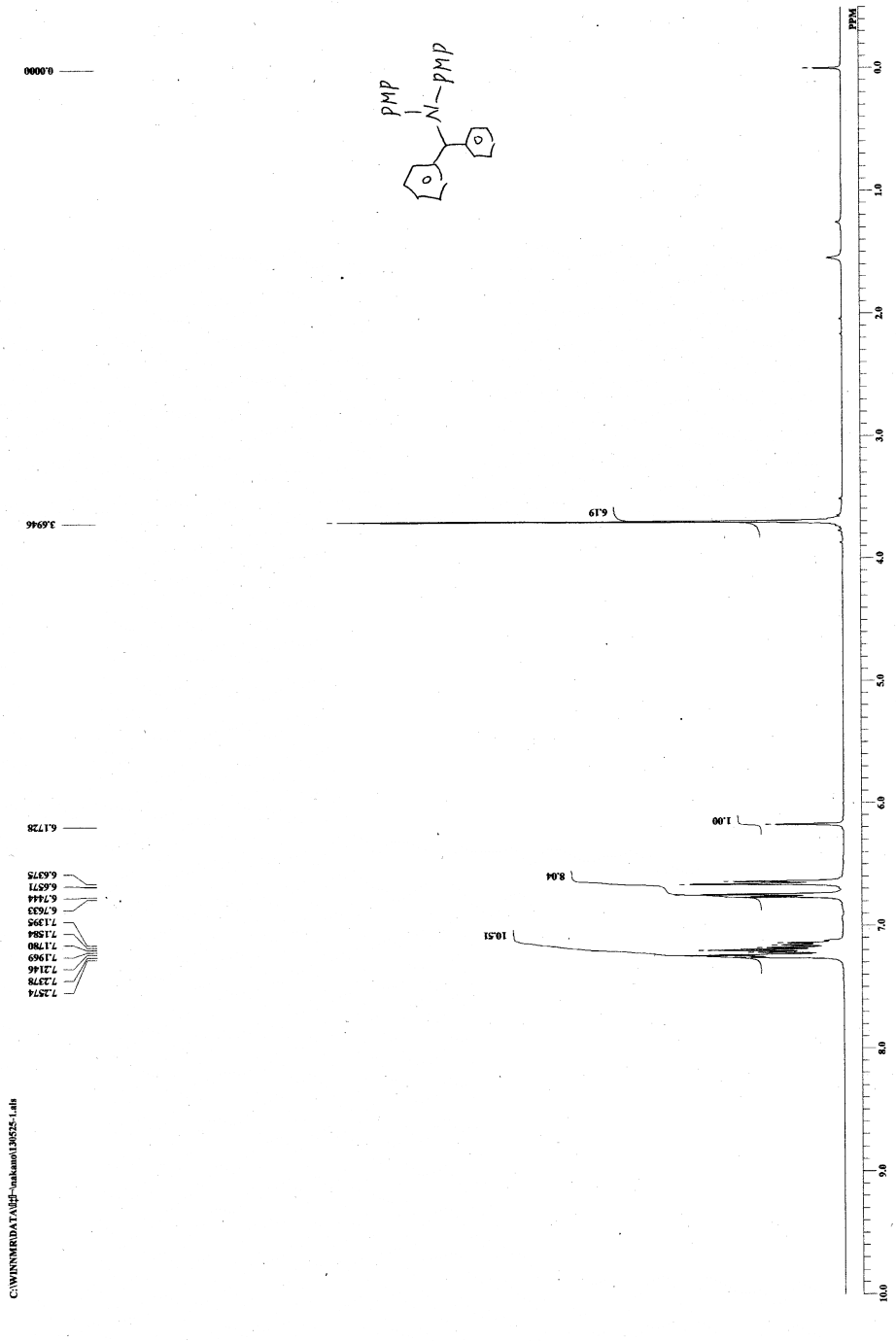

130512-1-13C
DPFILE 130512-1-13C.als
COMNT 130512-1-13C
DATIM Sun May 12 19:47:58 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 281
ACQTM 1.2883 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 26.2 c
SLVNT CDCl3
EXREF 77.00 ppm
RG 0.60 Hz
RGAIN 25
PMP
MeO
MeO
Cl
PPM

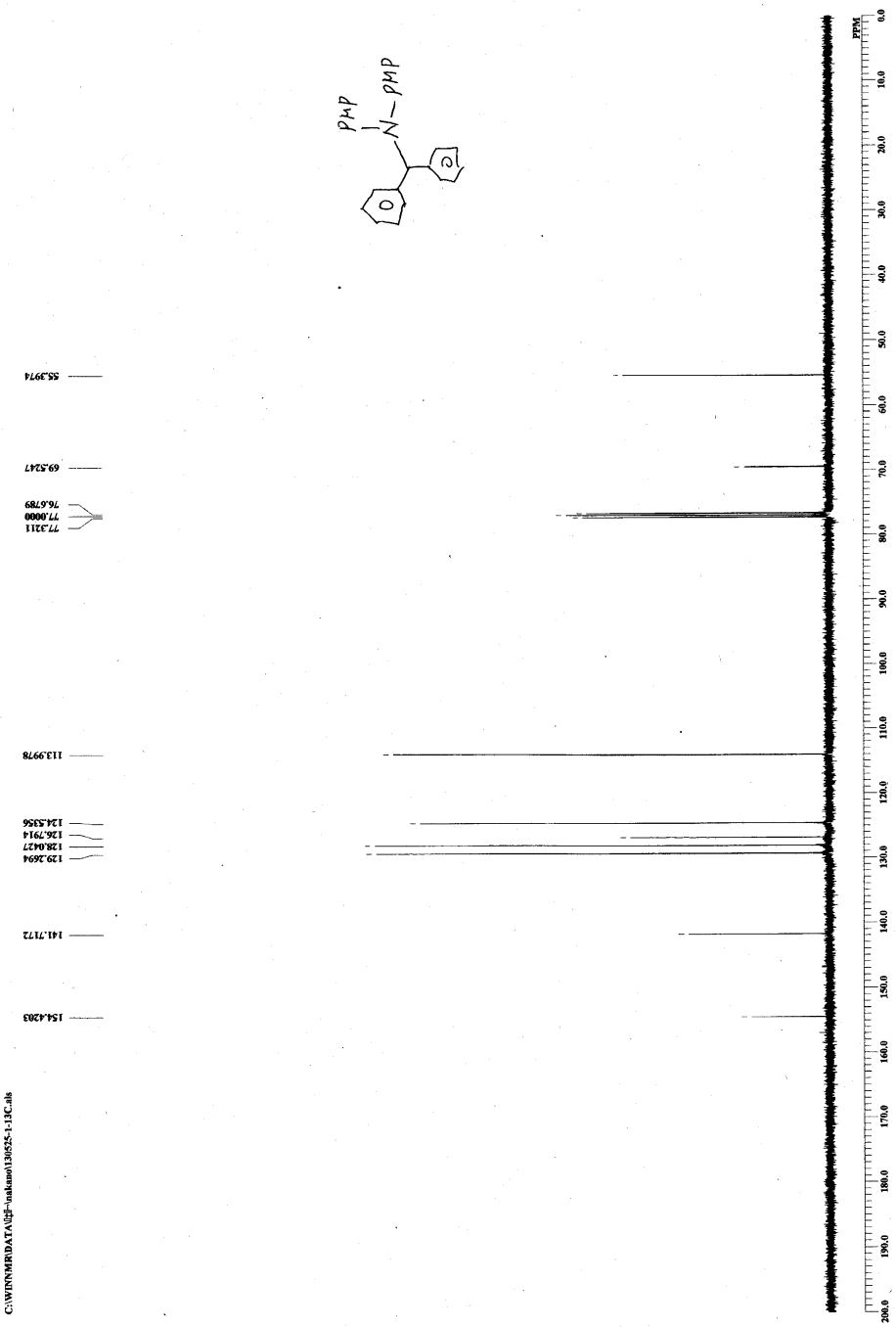
1-(4-Dimethylaminophenyl)-2-(4-methoxyphenyl)-1,2,3,4-tetrahydro-6,7-dimethoxyisoquinoline (17)



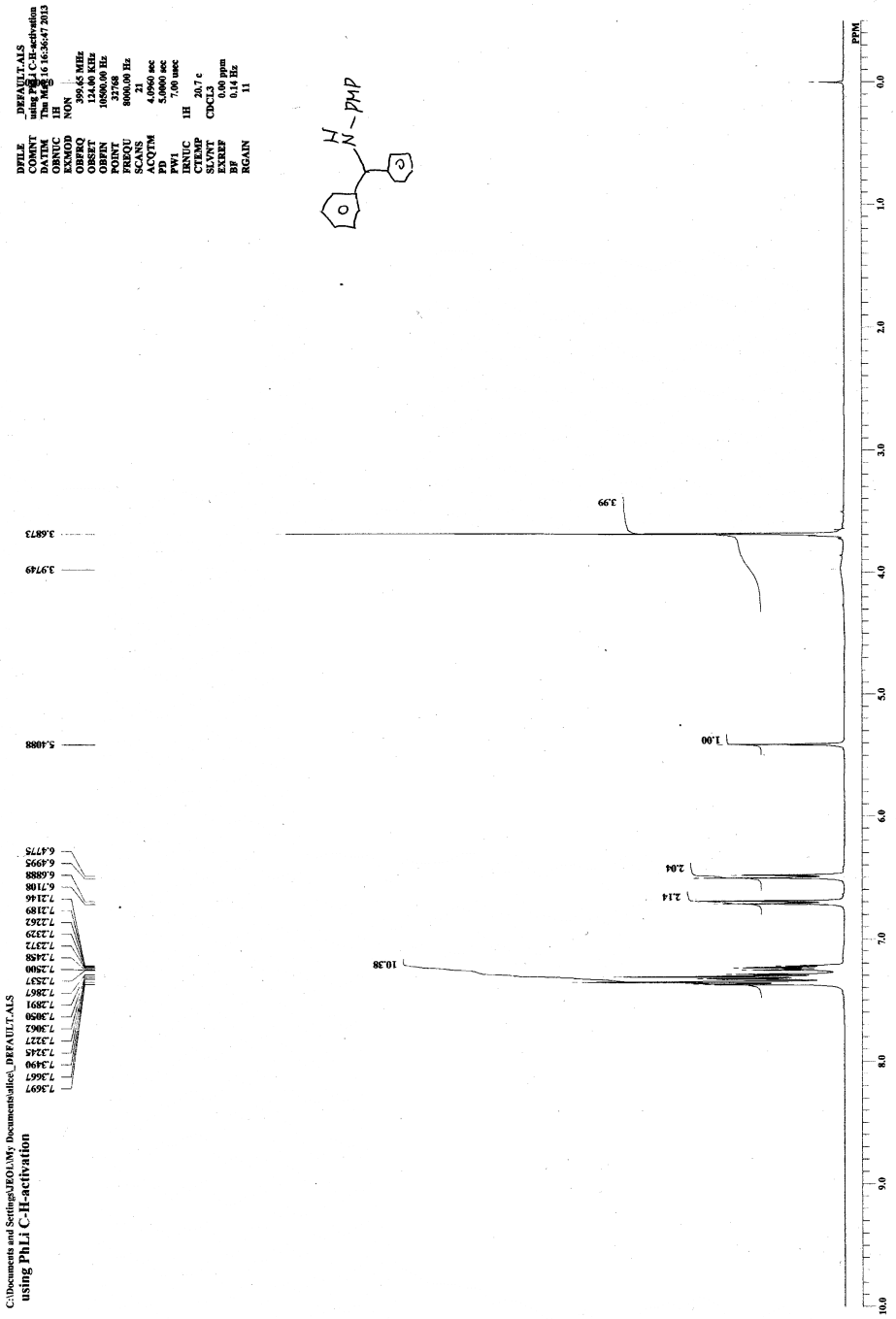

C:\Documents and Settings\HOUJM\My Documents\data\s_DEFAULT.ALS
wm2099-4-13C
DFILE _DEFAULT.ALS
COMMT wm2099-4-13C
DATIM Fri May 31 12:09:24 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 100660.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 257
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
INUC 1H 23.2 c
CTEMP
SLVNT CDCl3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 26
36.9123
40.5209
43.3447
55.3538
55.4008
55.5078
63.3337
76.6789
77.0000
77.3128
111.1163
111.1575
111.8819
114.5196
114.4835
118.0976
118.8056
127.4006
129.2282
129.4835
130.9406
144.7551
147.0932
147.5872
149.4148
152.9549
N-PMP
MeO
MeO
NMe2
PPM
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0

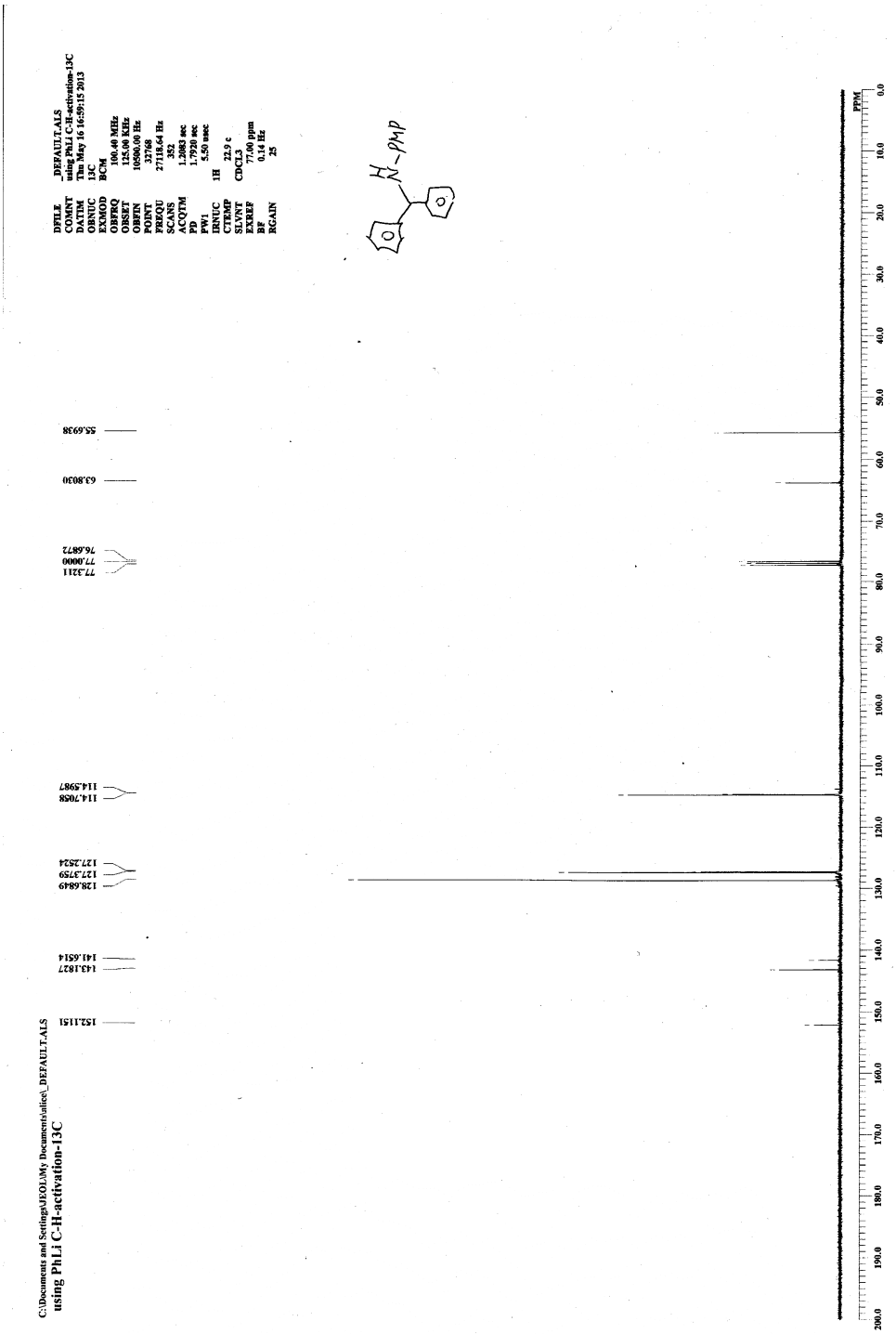
***N*-Di-(4-methoxyphenyl)-1,1-diphenylmethylamine (18)**



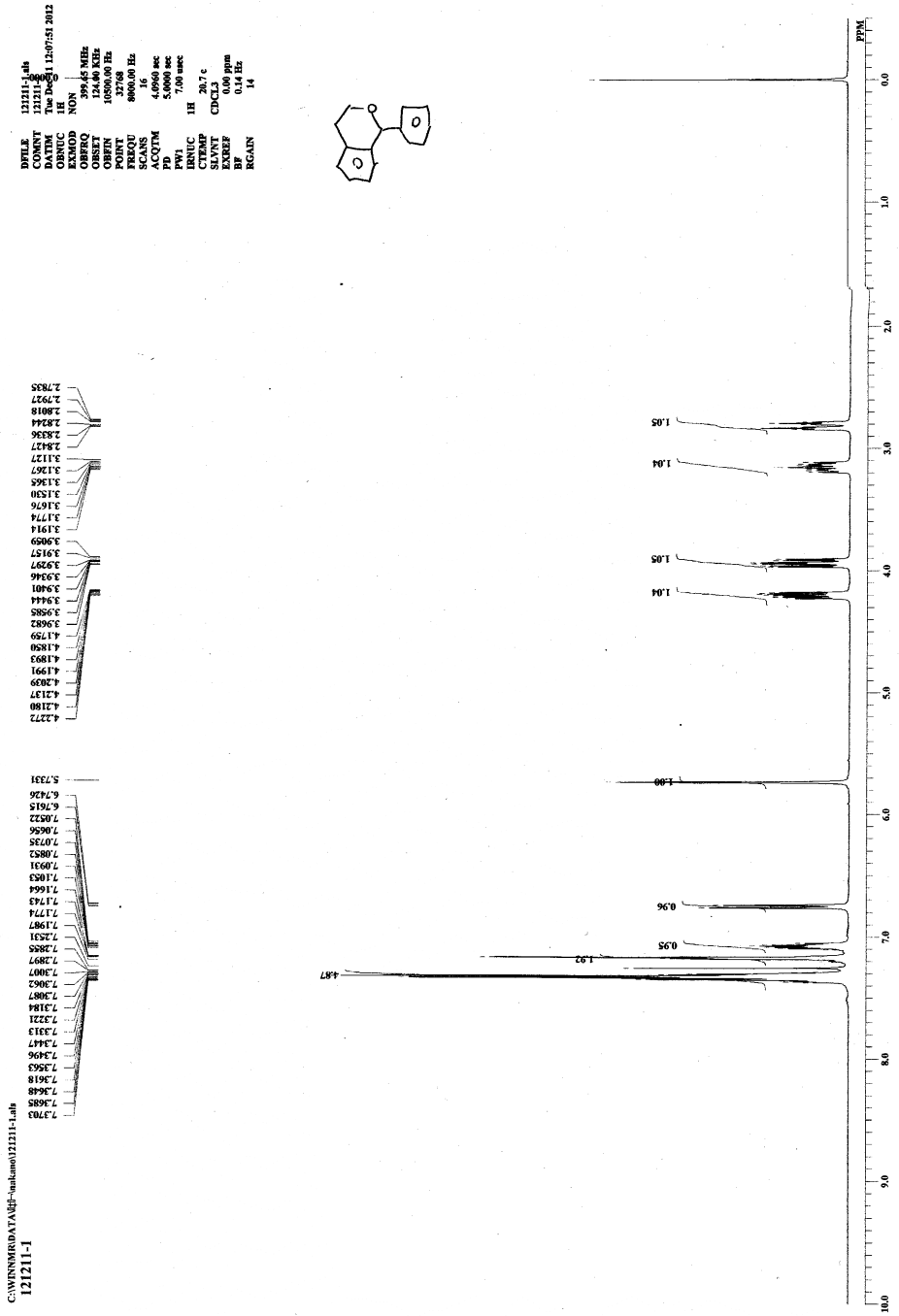

PMP
N
PMP
O
2
55.3974
69.5247
76.6789
77.0000
77.3211
113.9978
124.3356
126.7914
128.0427
129.3694
141.7172
154.4203
C:\WINNMR\DATA\dgf=nakano\130525-1-13C.als
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0

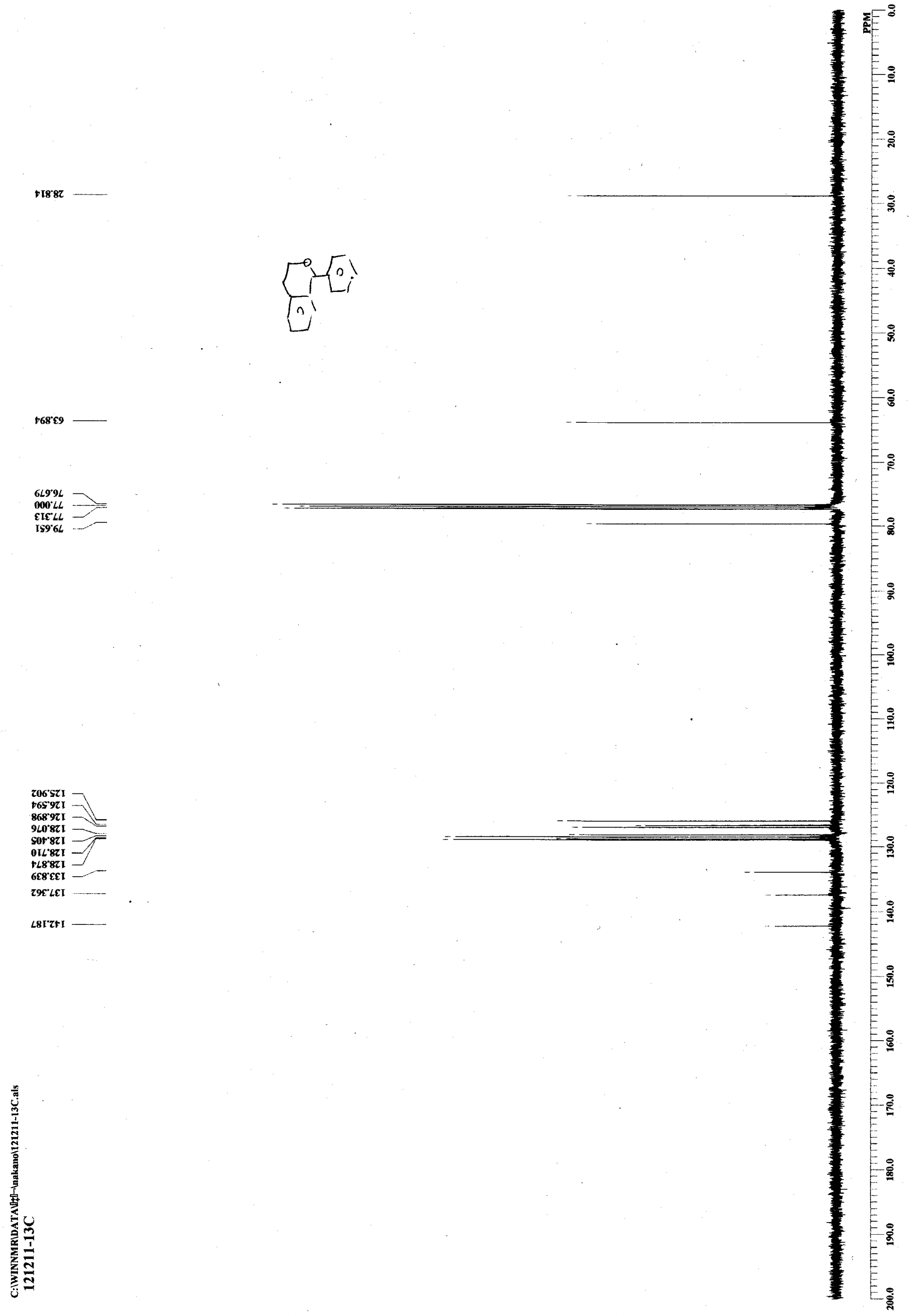
N-(4-Methoxyphenyl)-1,1-diphenylmethylamine (19)



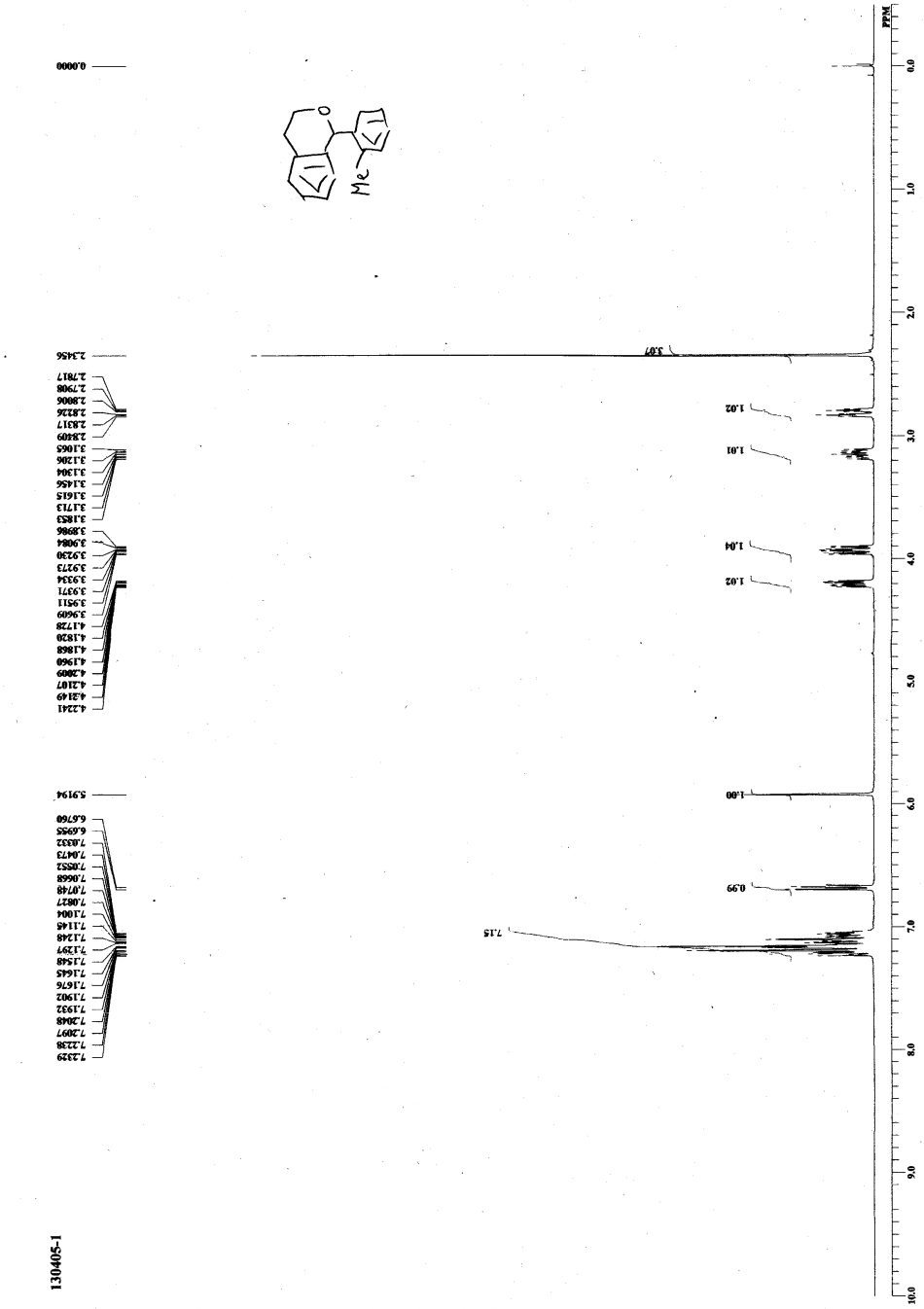

C:\Documents and Settings\JEOL\My Documents\alice_DEFAULT.ALS
using PhLi C-H-activation-13C
152.1151
143.1827
141.6514
128.6849
127.3759
127.2524
114.7058
114.3987
77.3211
77.0000
76.6872
63.8030
55.6938
DFILE DEFAULT.ALS
COMNT using PhLi C-H-activation-13C
DATIM Thu May 16 16:09:15 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10080.00 Hz
POINT 32504
FREQU 27118.64 Hz
SCANS 352
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
DRNUC 1H
CTEMP 22.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.14 Hz
RGAIN 25
N-PMP
PPM
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0

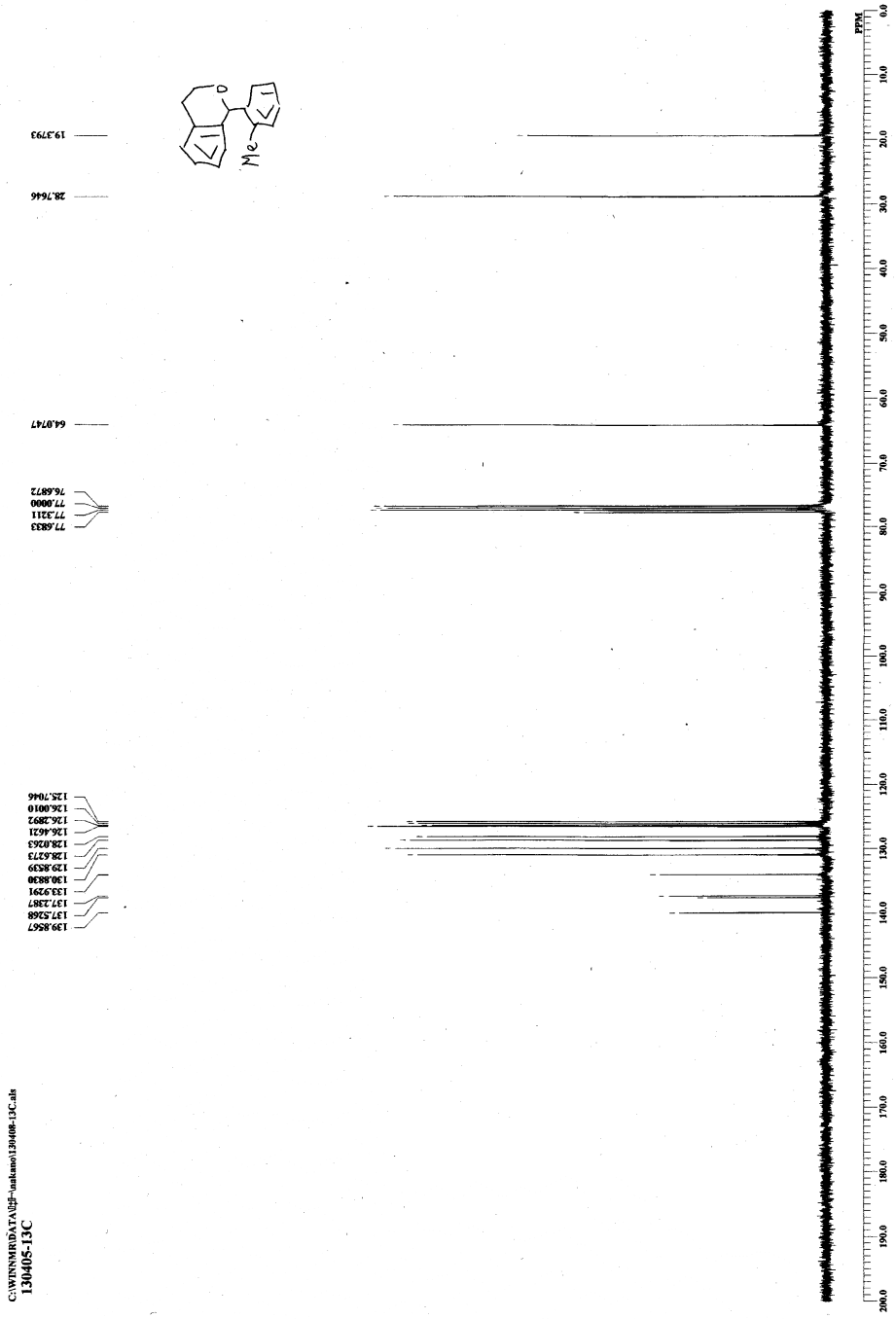
1-Phenylisochroman (20)



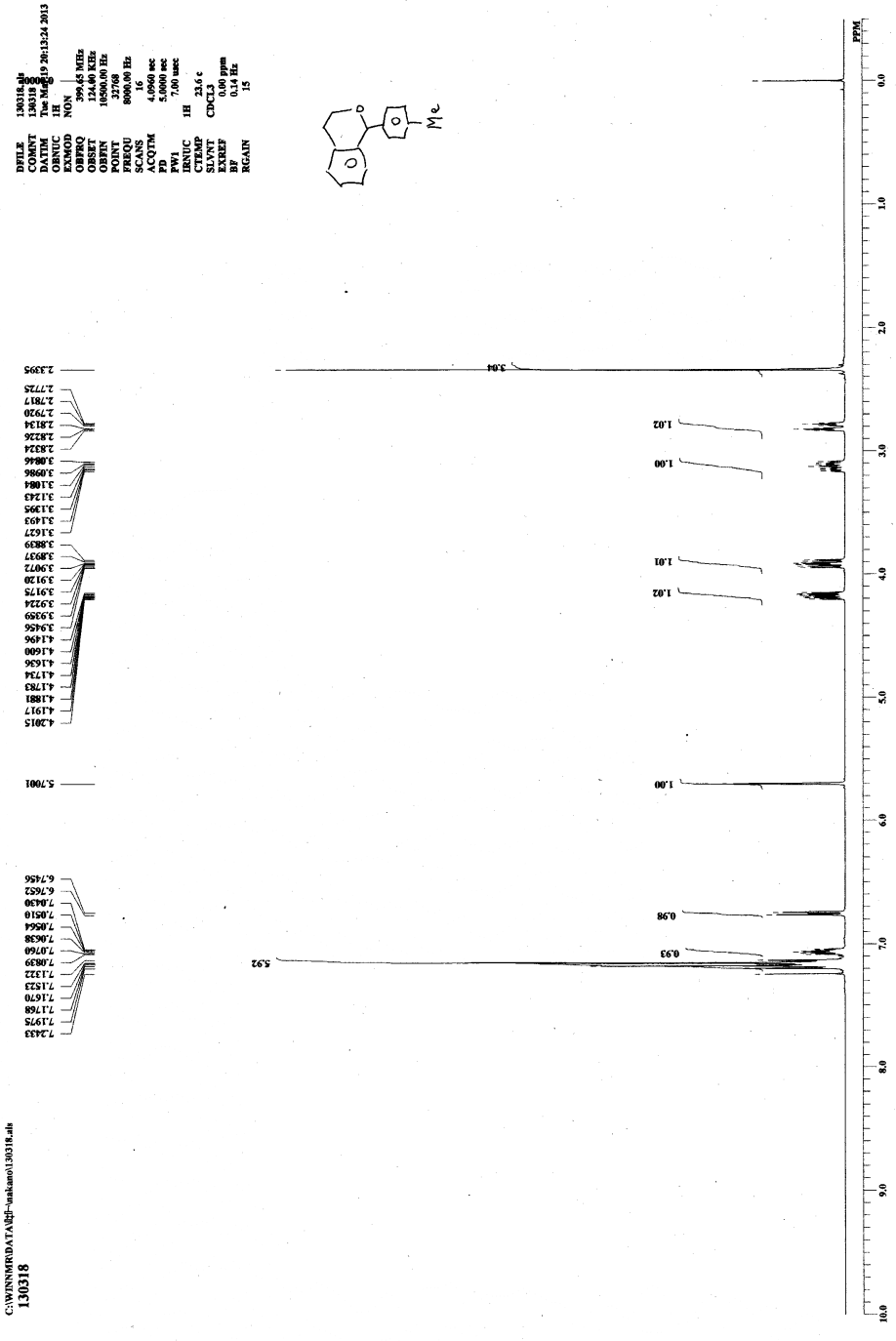

121211-13C
28.814
63.894
76.679
77.000
77.313
79.651
125.902
126.594
126.898
128.076
128.405
128.710
128.874
133.839
137.362
142.187
PPM

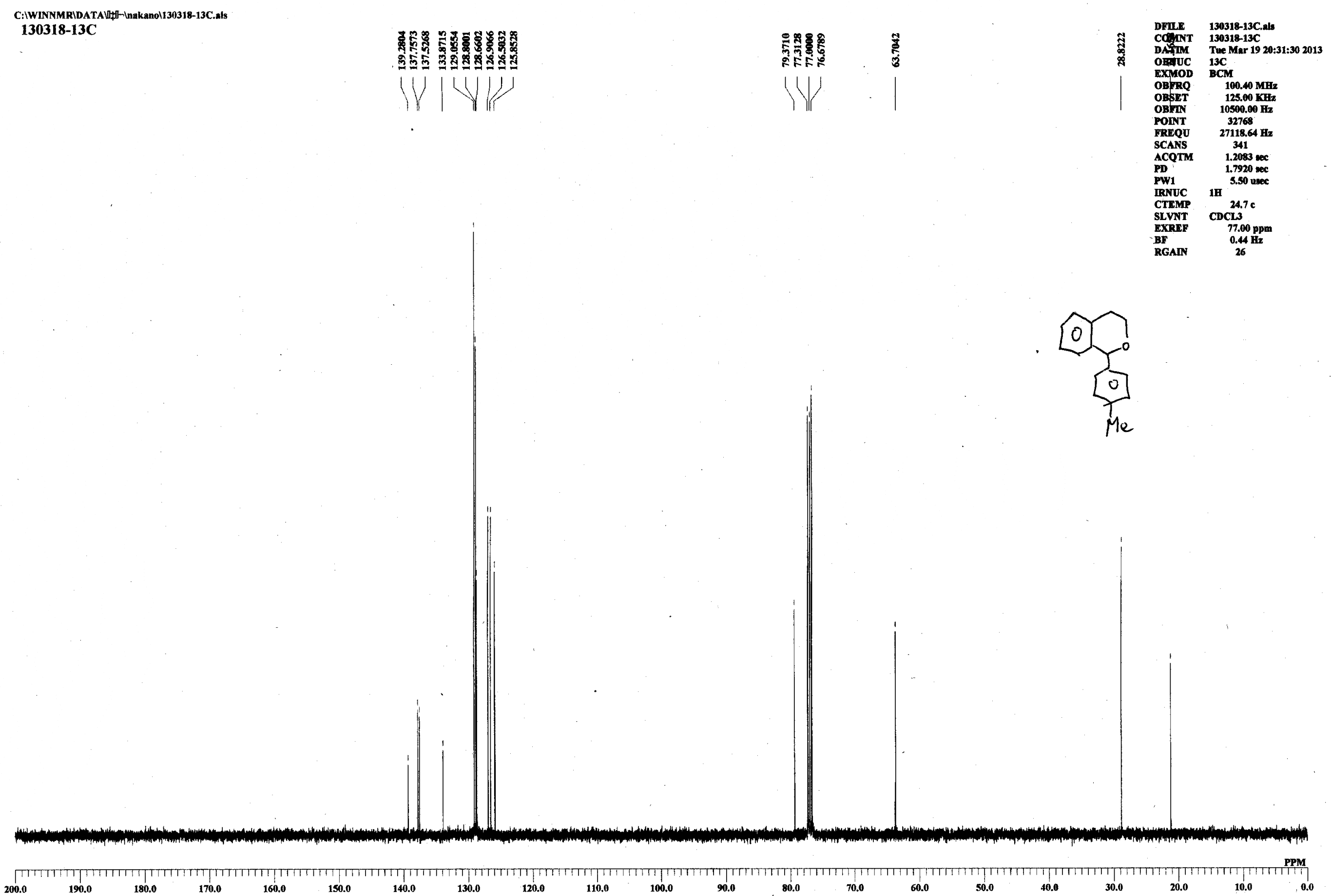
1-(2-Methylphenyl)-isochroman (21)



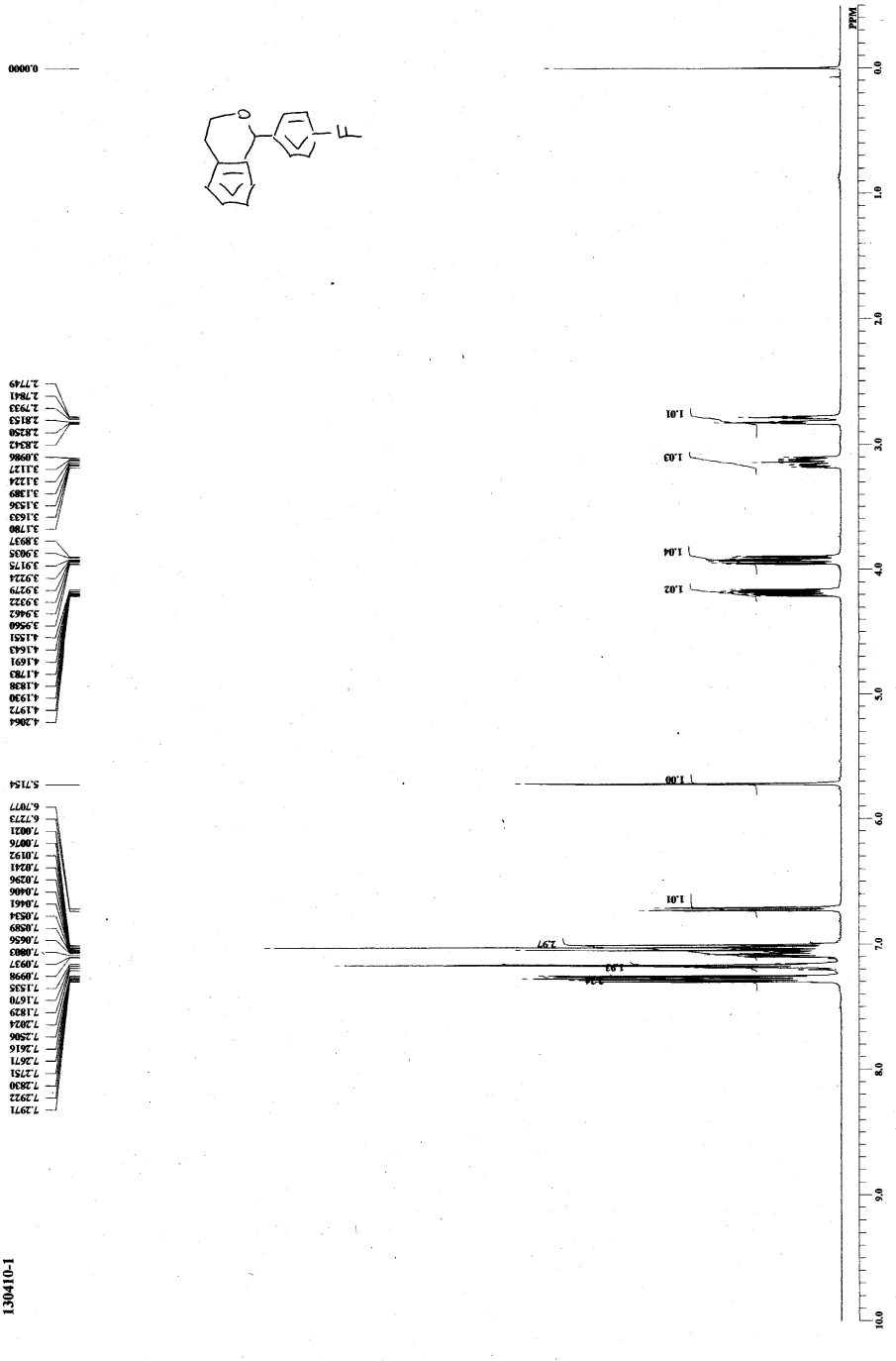

Me
19.3793
28.7646
64.0747
76.6872
77.0000
77.3211
77.6833
125.7046
126.0019
126.2892
126.6621
128.0263
128.6373
129.8539
130.8830
133.9291
137.2387
137.5268
139.8567
PPM
C:\WINNMR\DATA\tjf~tankano\130408-13C.als
130405-13C

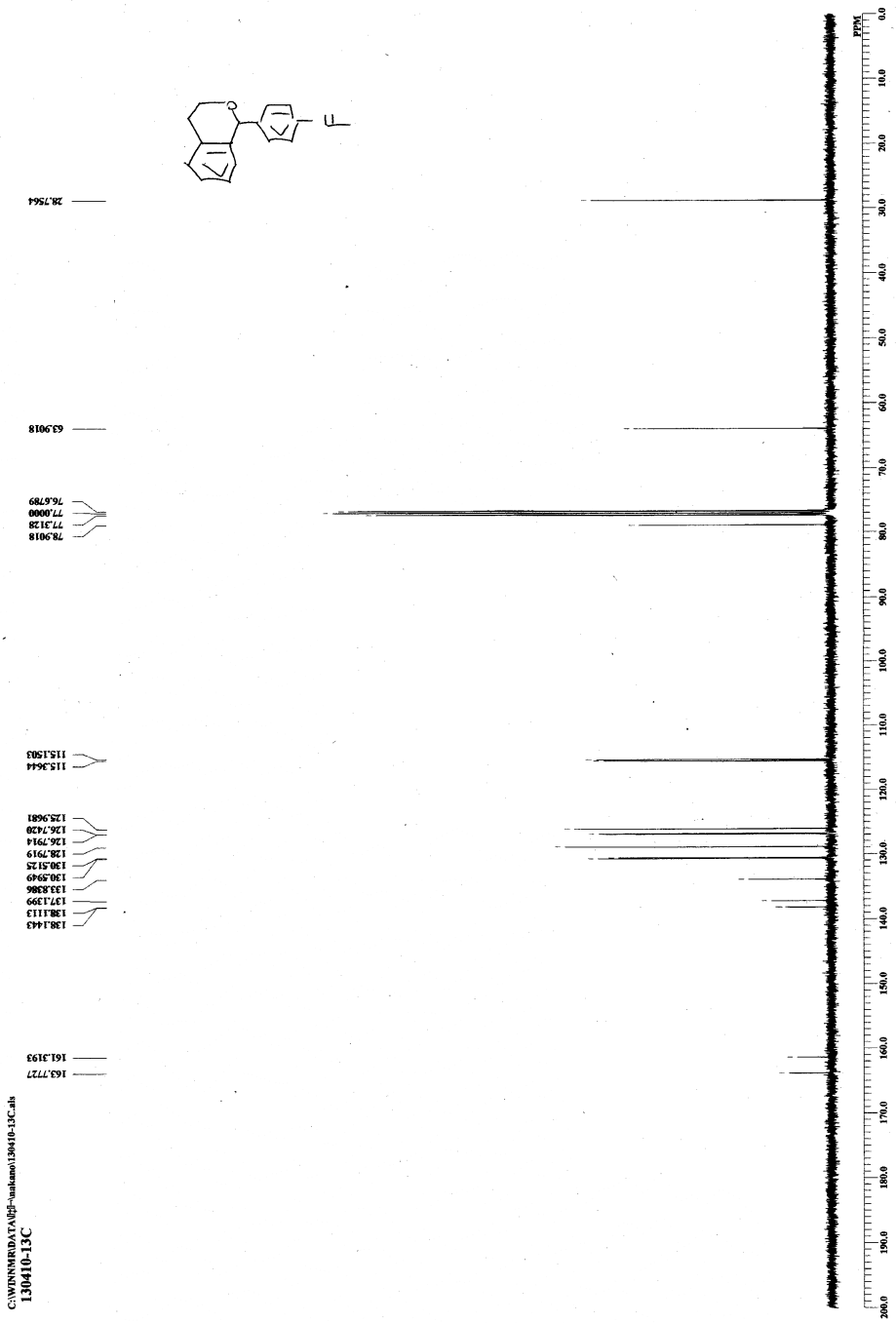
1-(4-Methylphenyl)-isochroman (22)



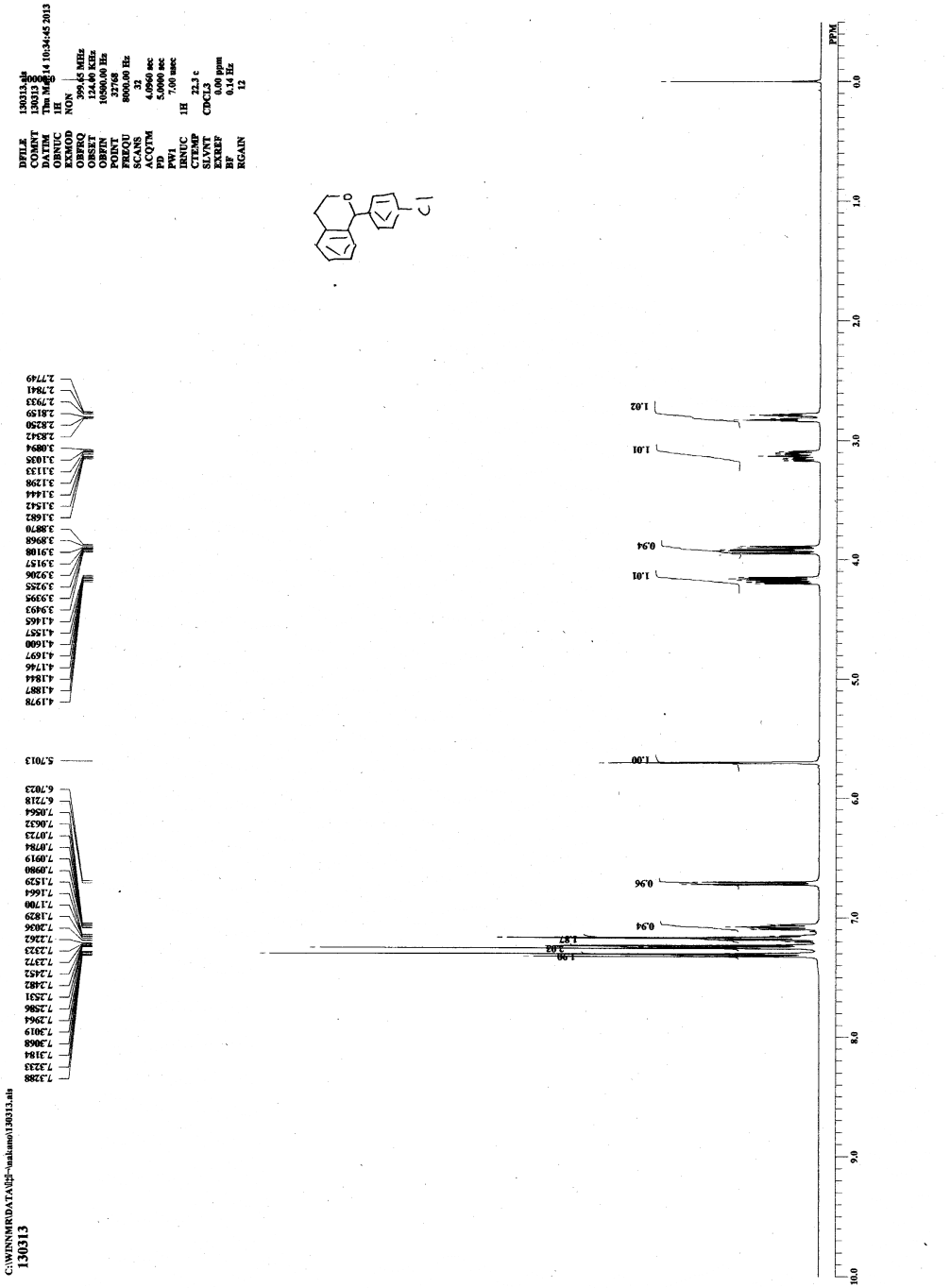

C:\WINNMR\DATA\...\nakano\130318-13C.als
130318-13C
139.2804
137.7573
137.5268
133.8715
129.0554
128.8001
128.6602
126.9066
126.5032
125.8528
79.3710
77.3128
77.0000
76.6789
63.7042
28.8222
DFILE 130318-13C.als
COMNT 130318-13C
DATIM Tue Mar 19 20:31:30 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 341
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 24.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.44 Hz
RGAIN 26
Me
PPM
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0

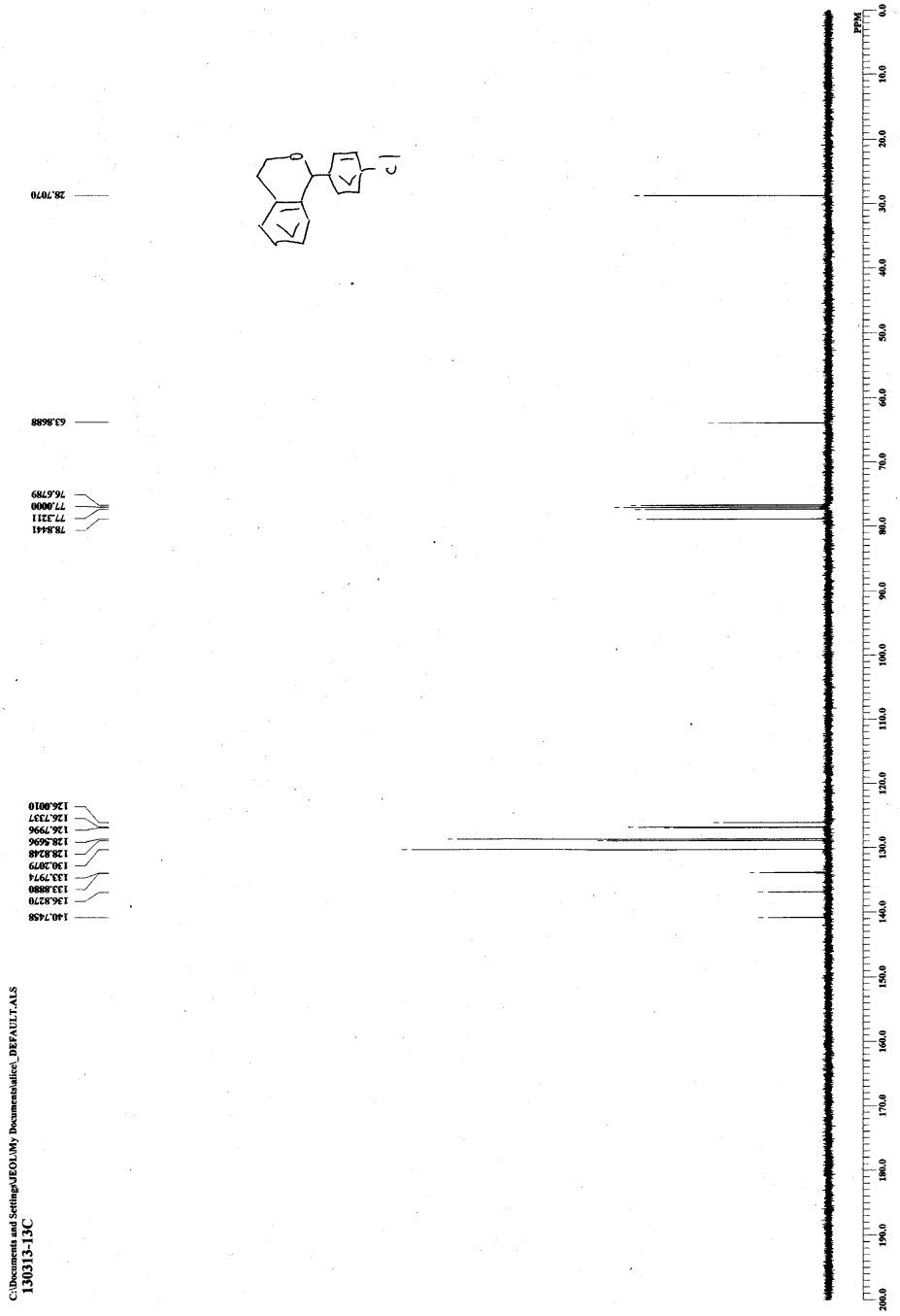
1-(4-Fluorophenyl)-isochroman (23)



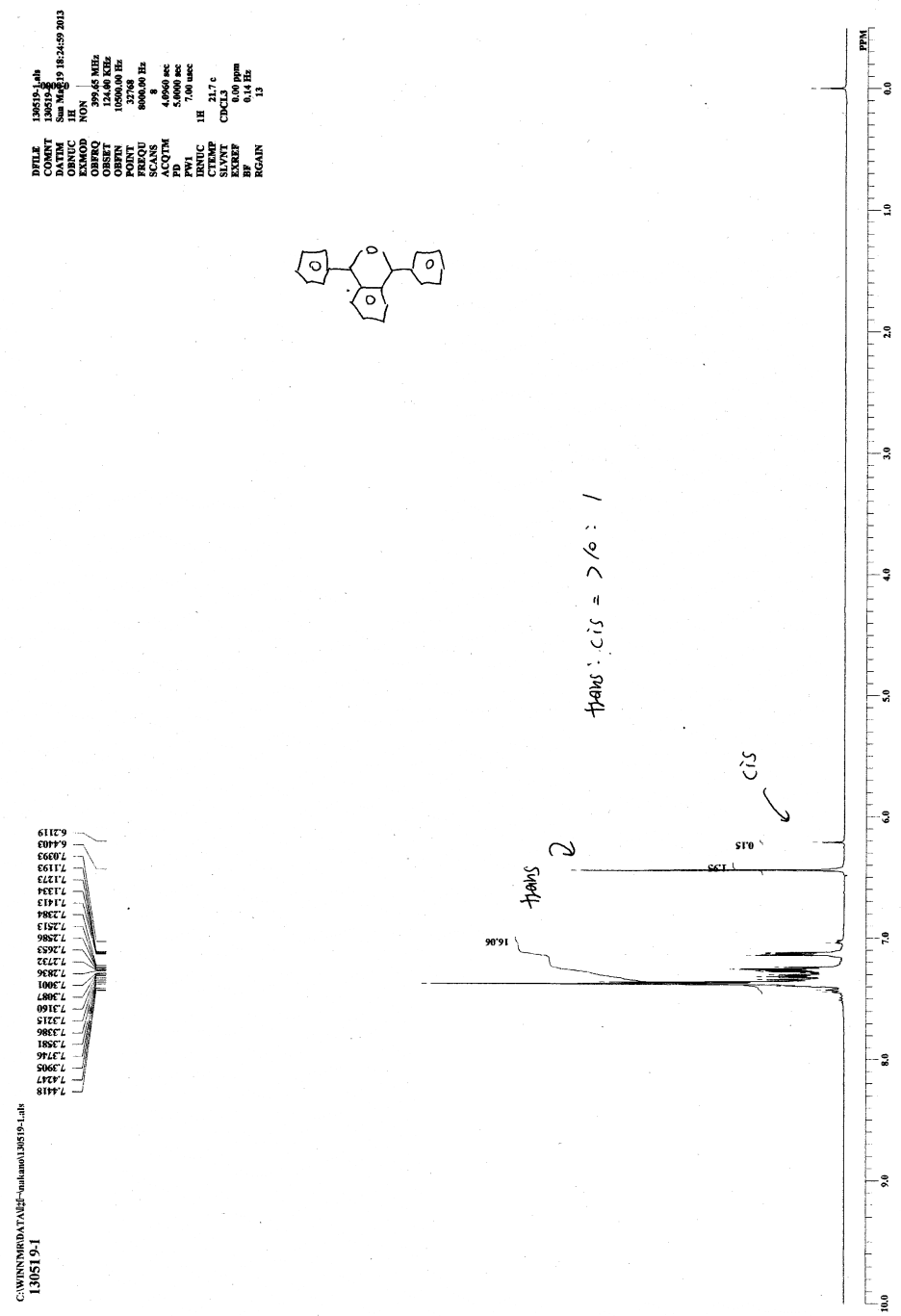

C:\WINNMRDATA\hjf-nakano\130410-13C.als
130410-13C
163.7727
161.3193
138.1443
138.1113
137.1399
133.3386
130.5949
130.5125
128.7919
126.7914
126.7420
125.9681
115.3644
115.1503
78.9018
77.3128
77.0000
76.6789
63.9018
28.7564
PPM

1-(4-Chlorophenyl)-isochroman (24)

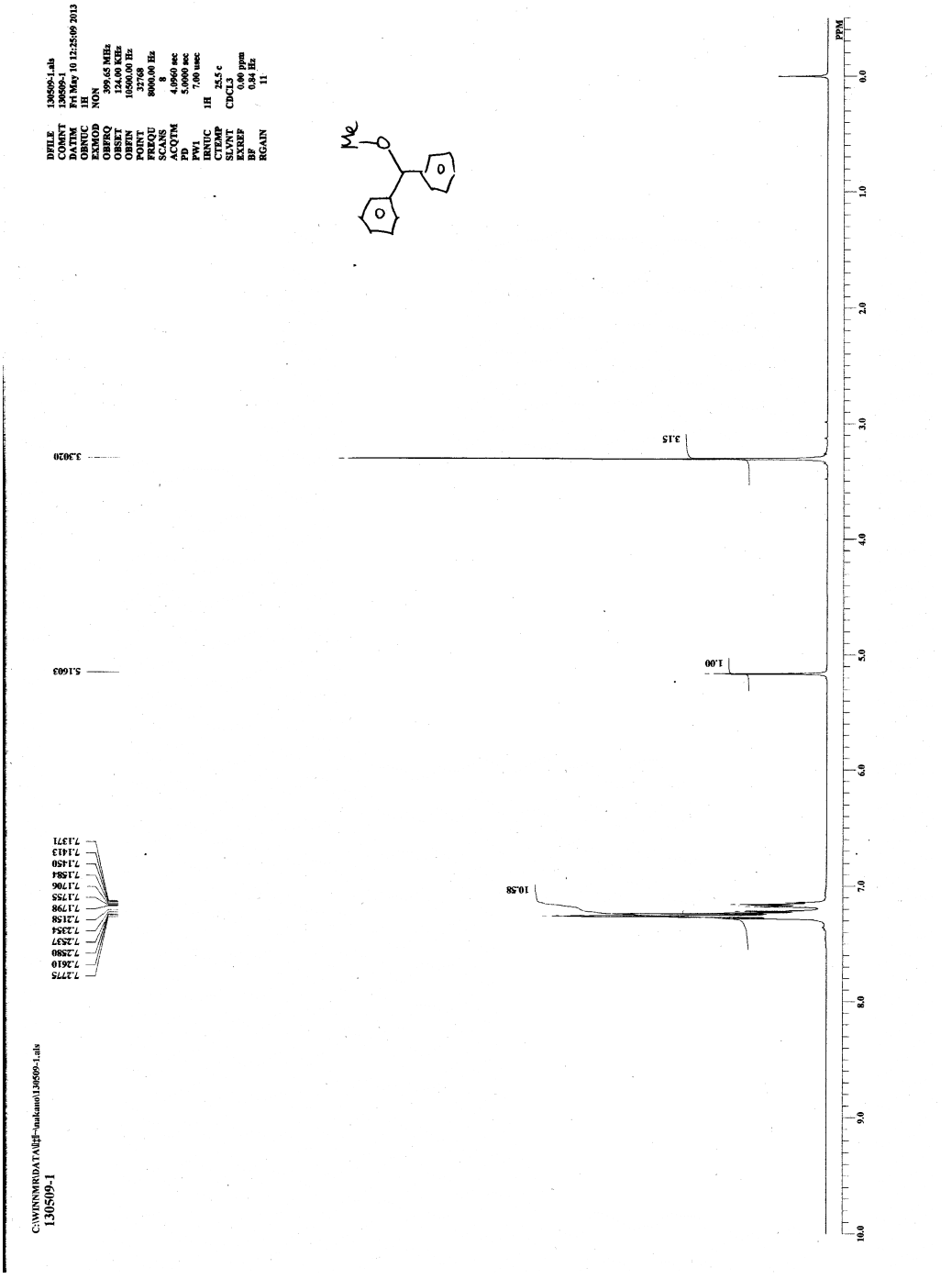


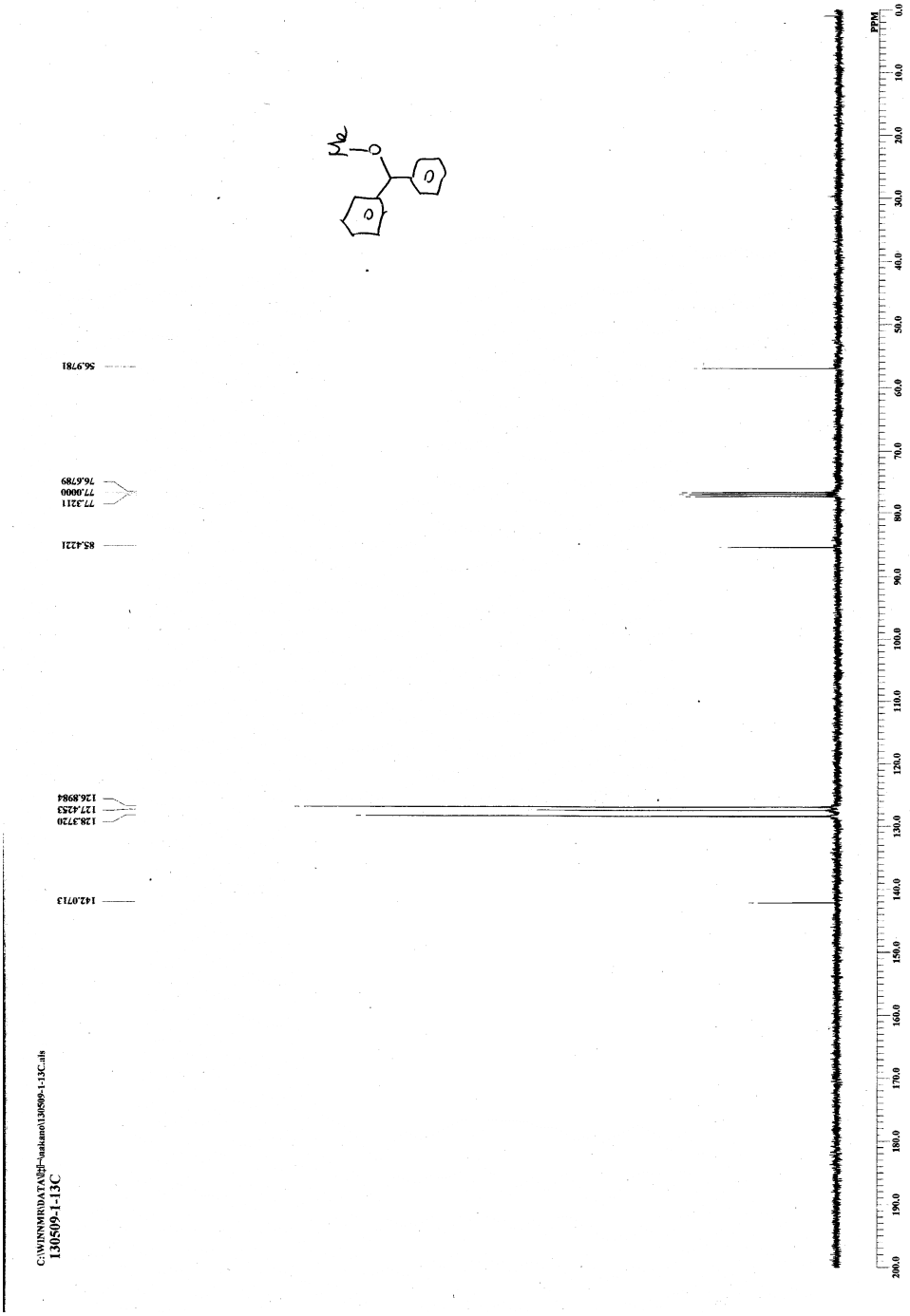

C:\Documents and Settings\JEOL\My Documents\alice_DEFAULT.ALS
130313-13C
28.7070
63.8688
76.6789
77.0000
77.3211
78.8441
126.0010
126.7337
126.7996
128.5696
128.8248
130.3079
133.7974
133.8880
136.8270
140.7458
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0

1,3-Diphenyl-1,3-dihydroisobenzofuran (25)

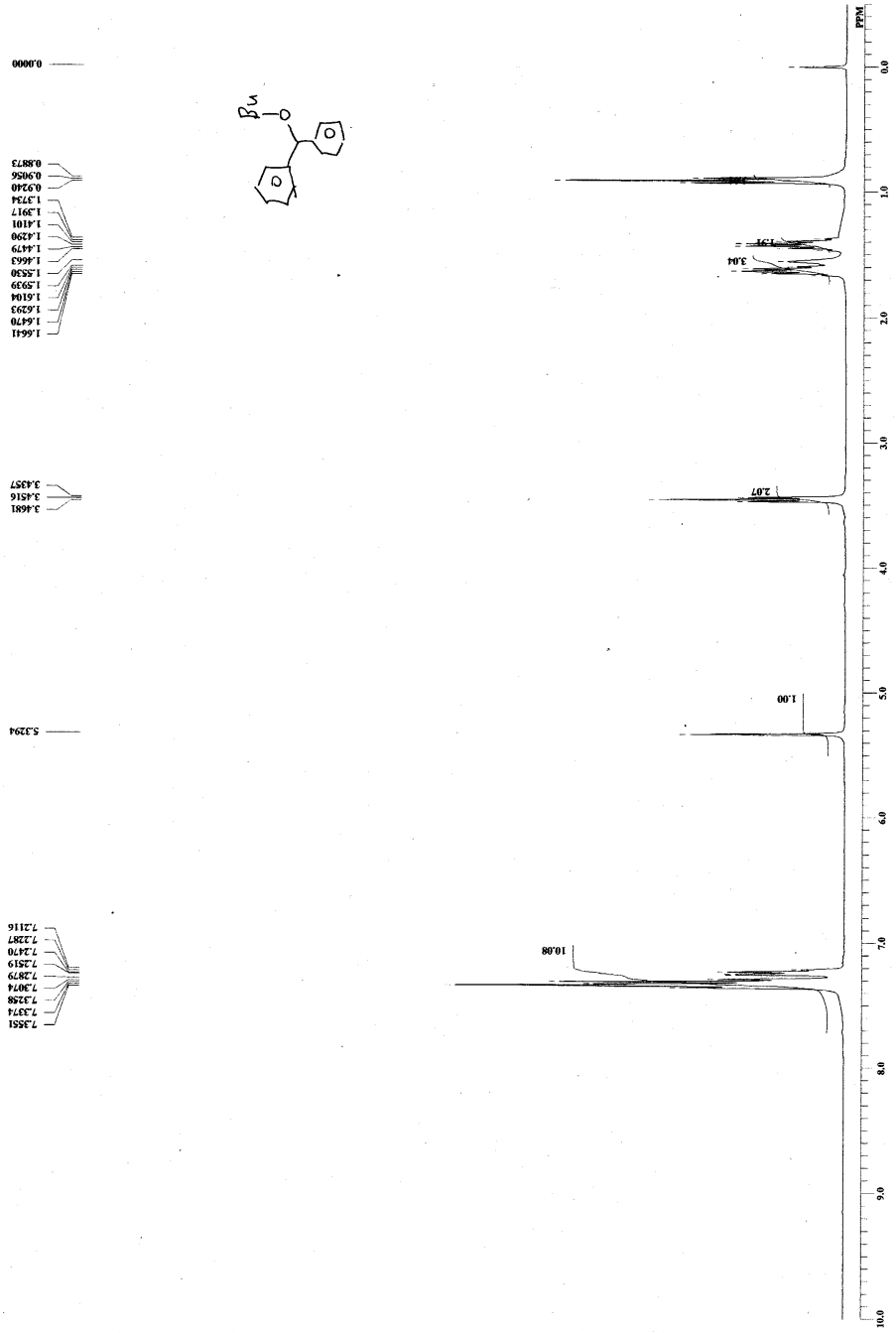


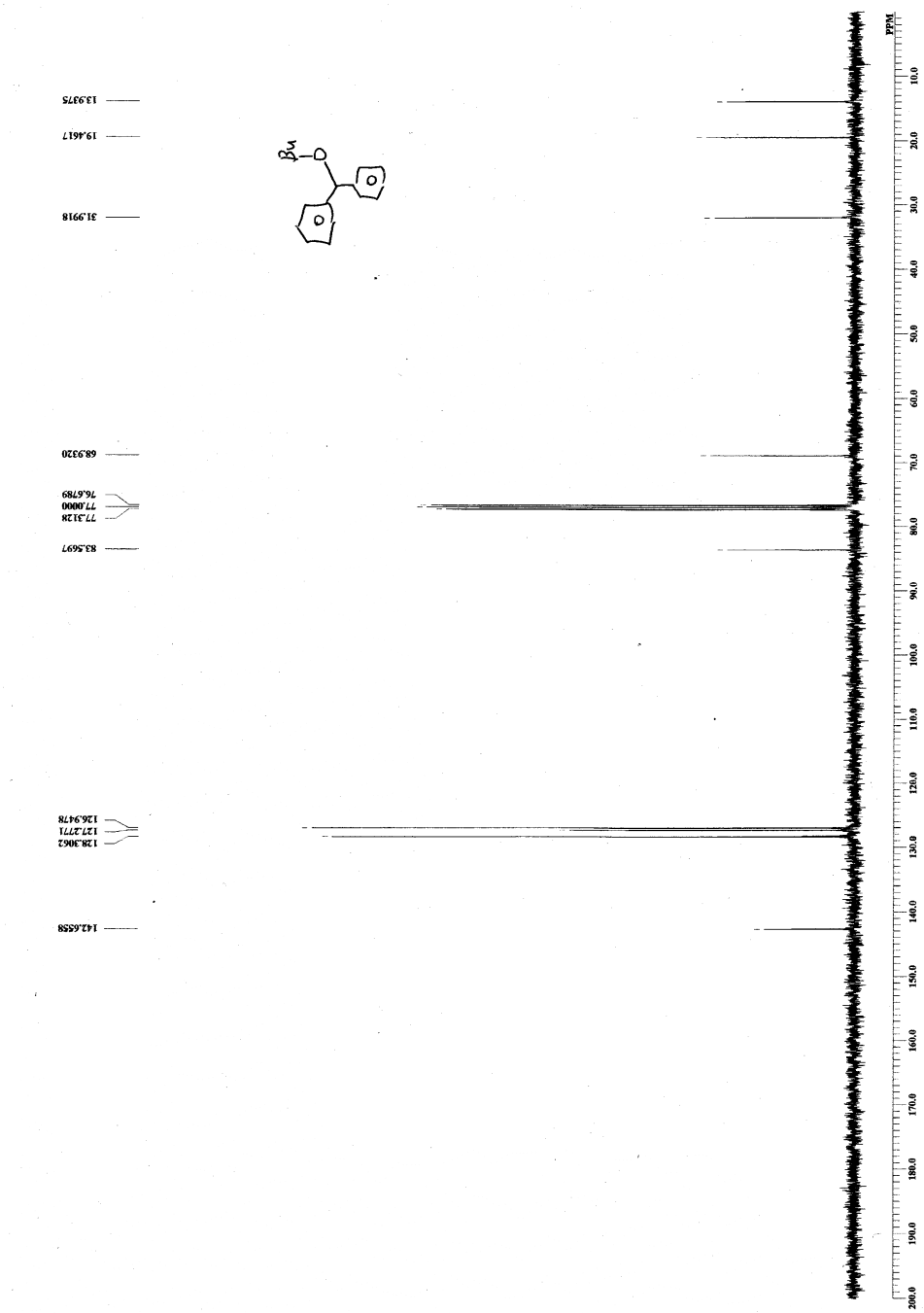
Diphenylmethyl methyl ether (26)



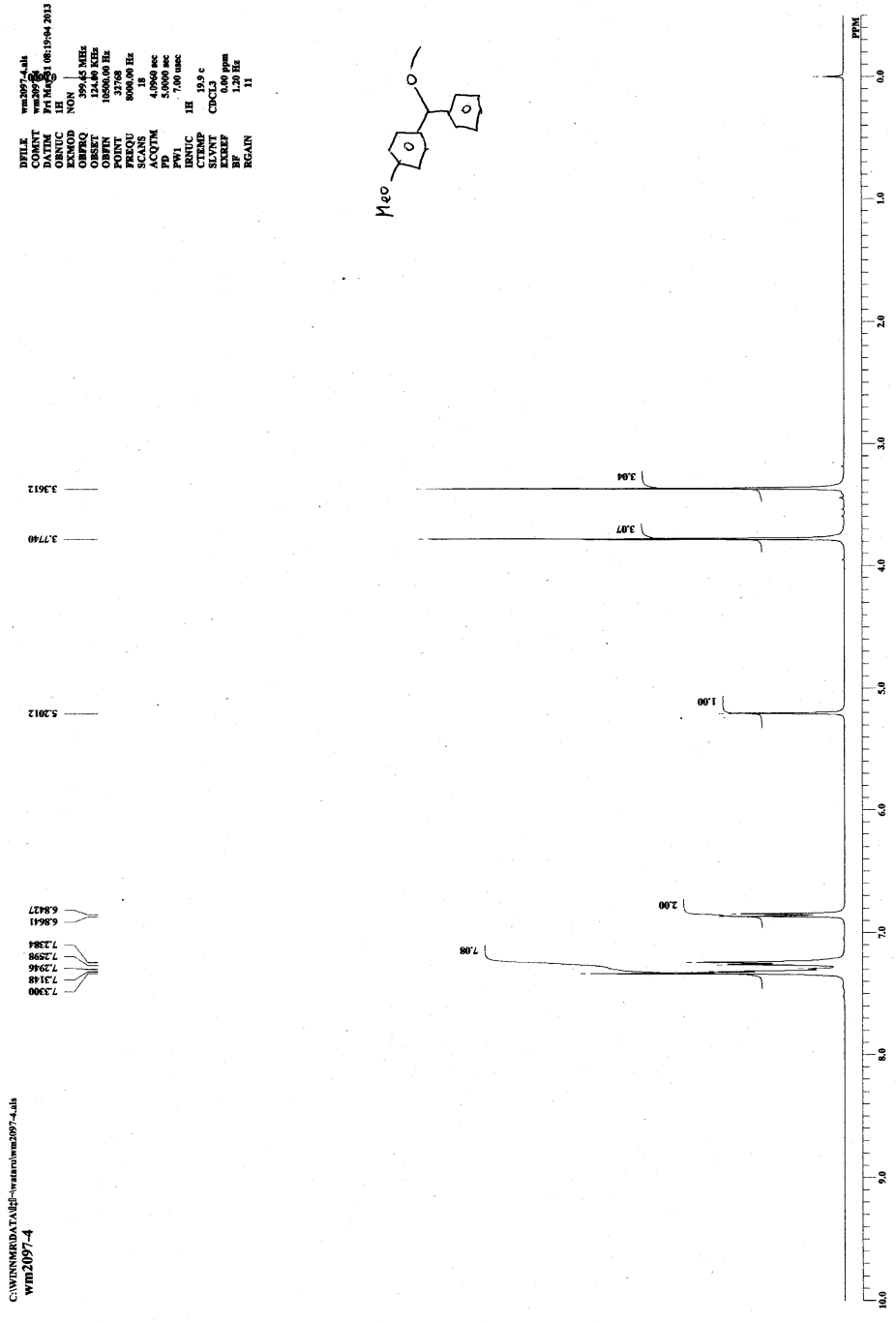

C:\WINNMRDATA\tjf~nakano\130509-1-13C.als
130509-1-13C
142.0713
128.3720
127.4253
126.8984
85.4221
77.3211
77.0000
76.6789
56.9781
PPM

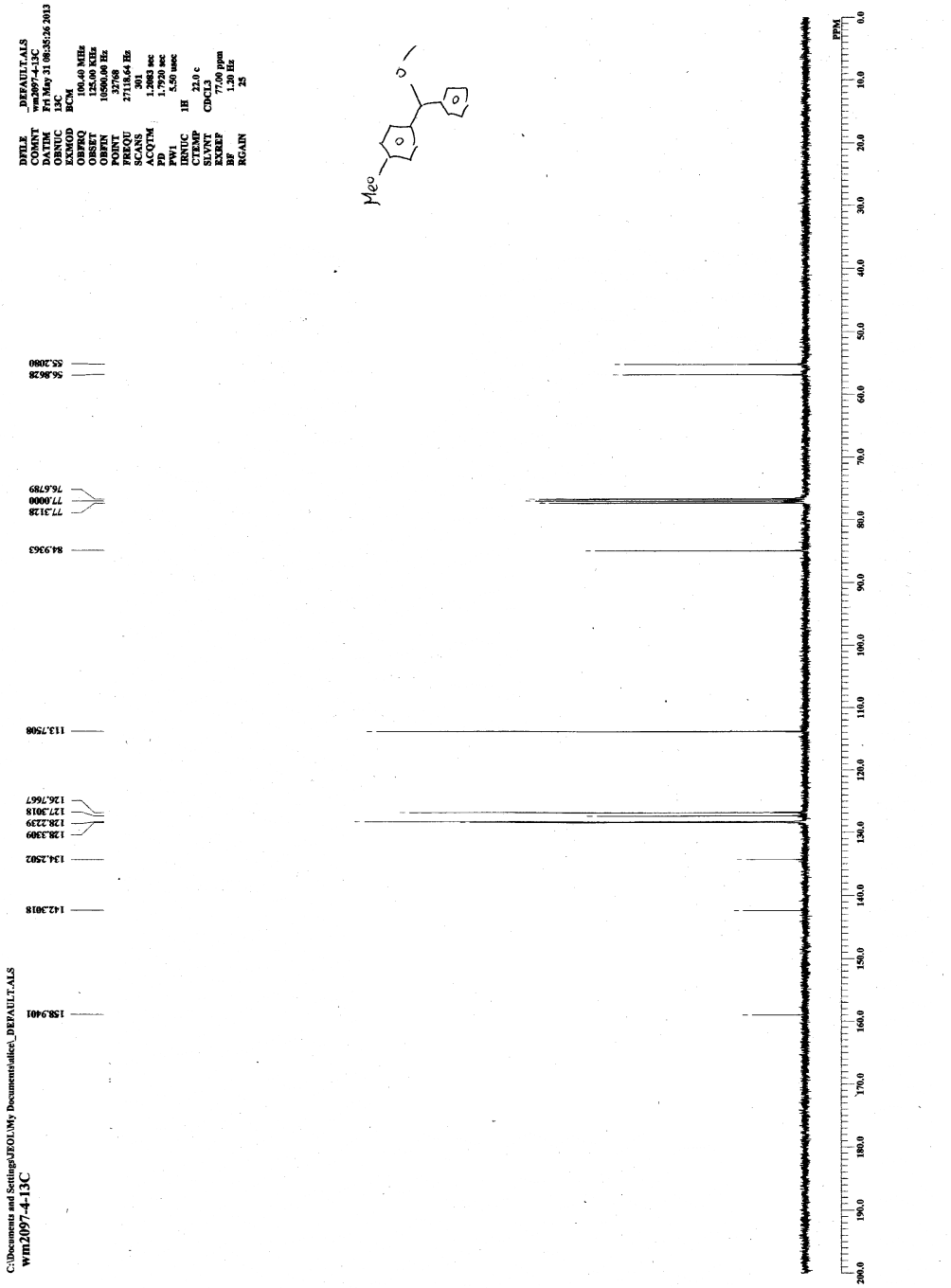
Diphenylmethyl butyl ether (27)



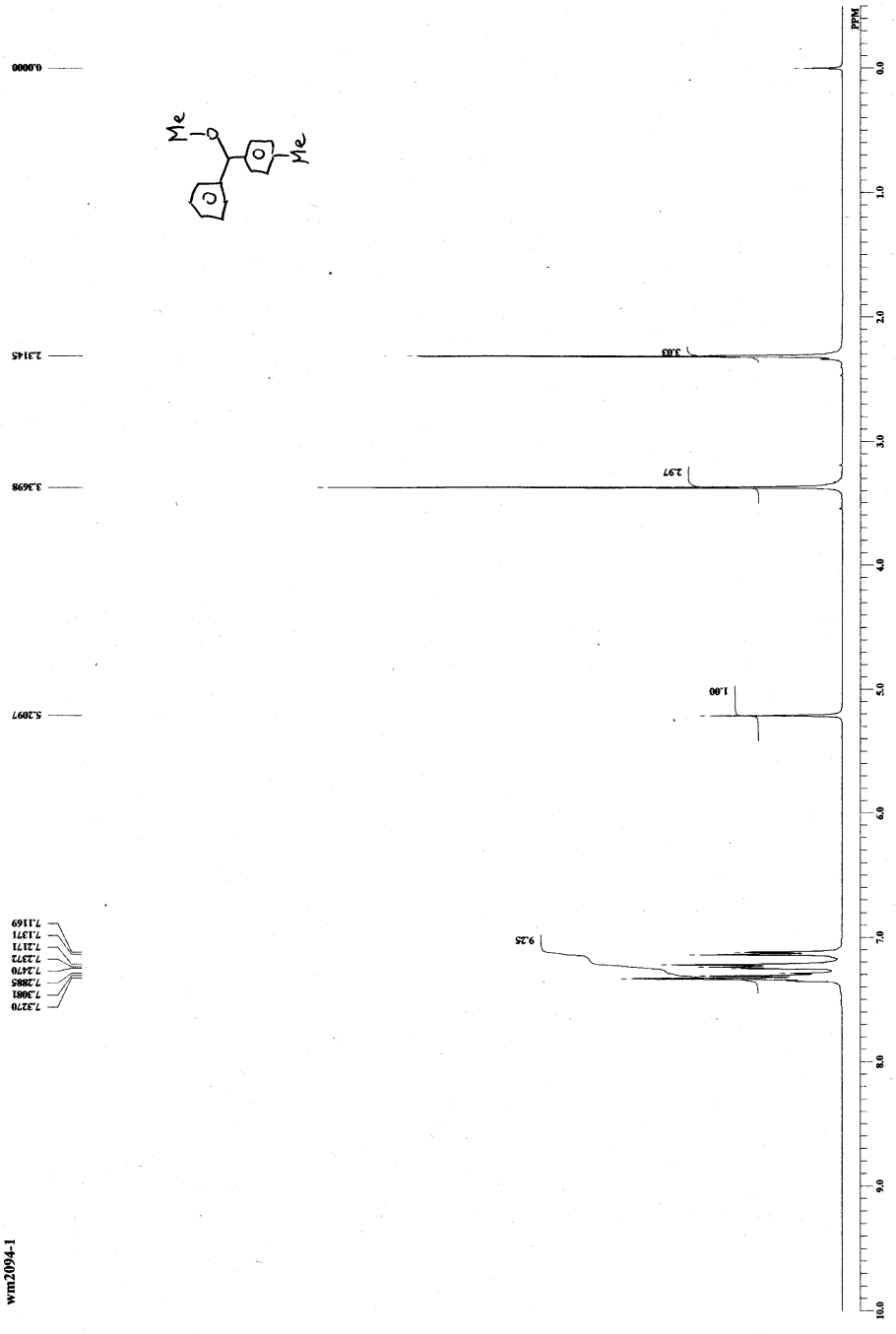

13.9375
19.4617
31.9918
68.9320
76.6789
77.0000
77.3128
83.5697
126.9478
127.2771
128.3062
141.6558
PPM

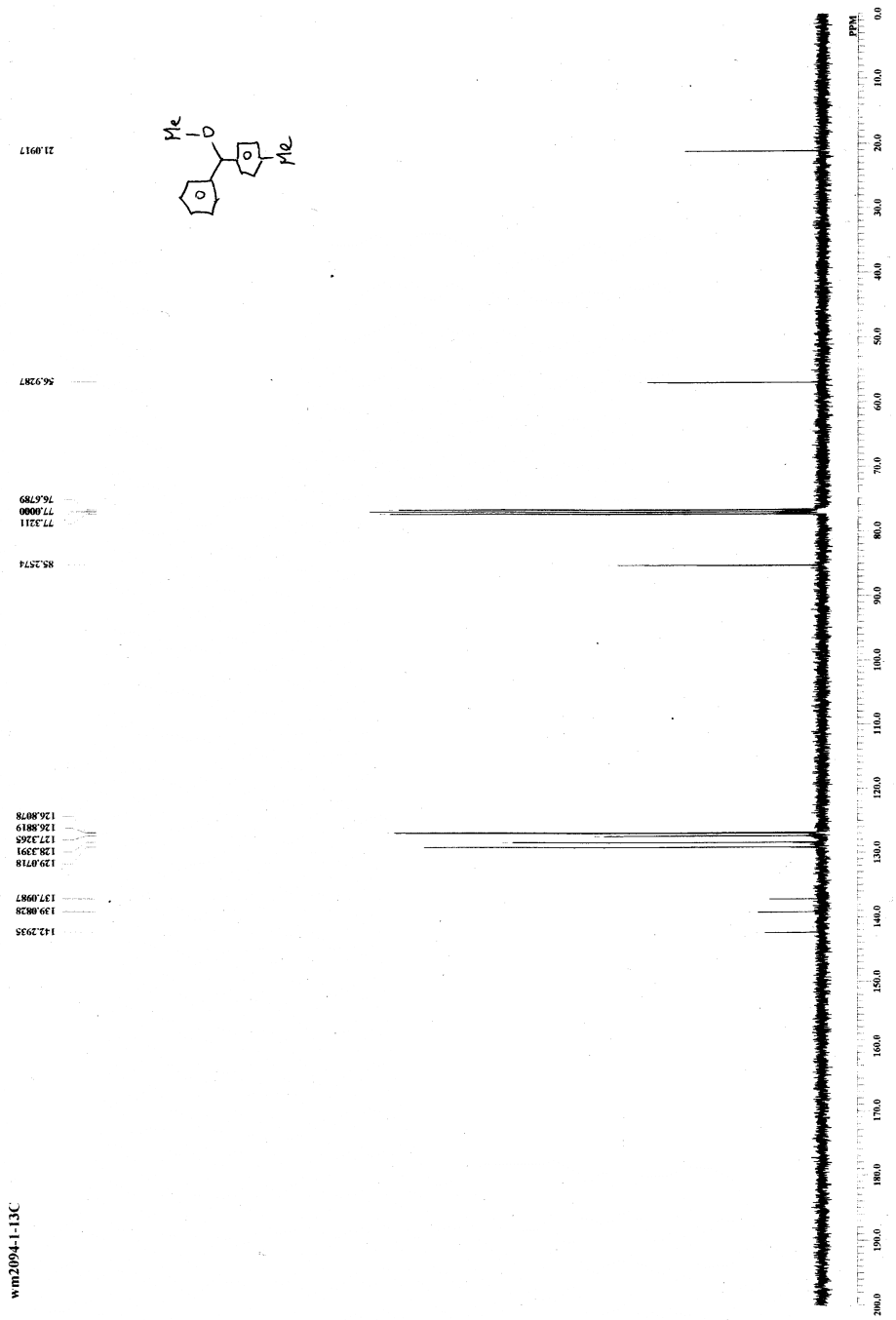
1-Methoxy-4-(methoxyphenylmethyl)benzene (28)



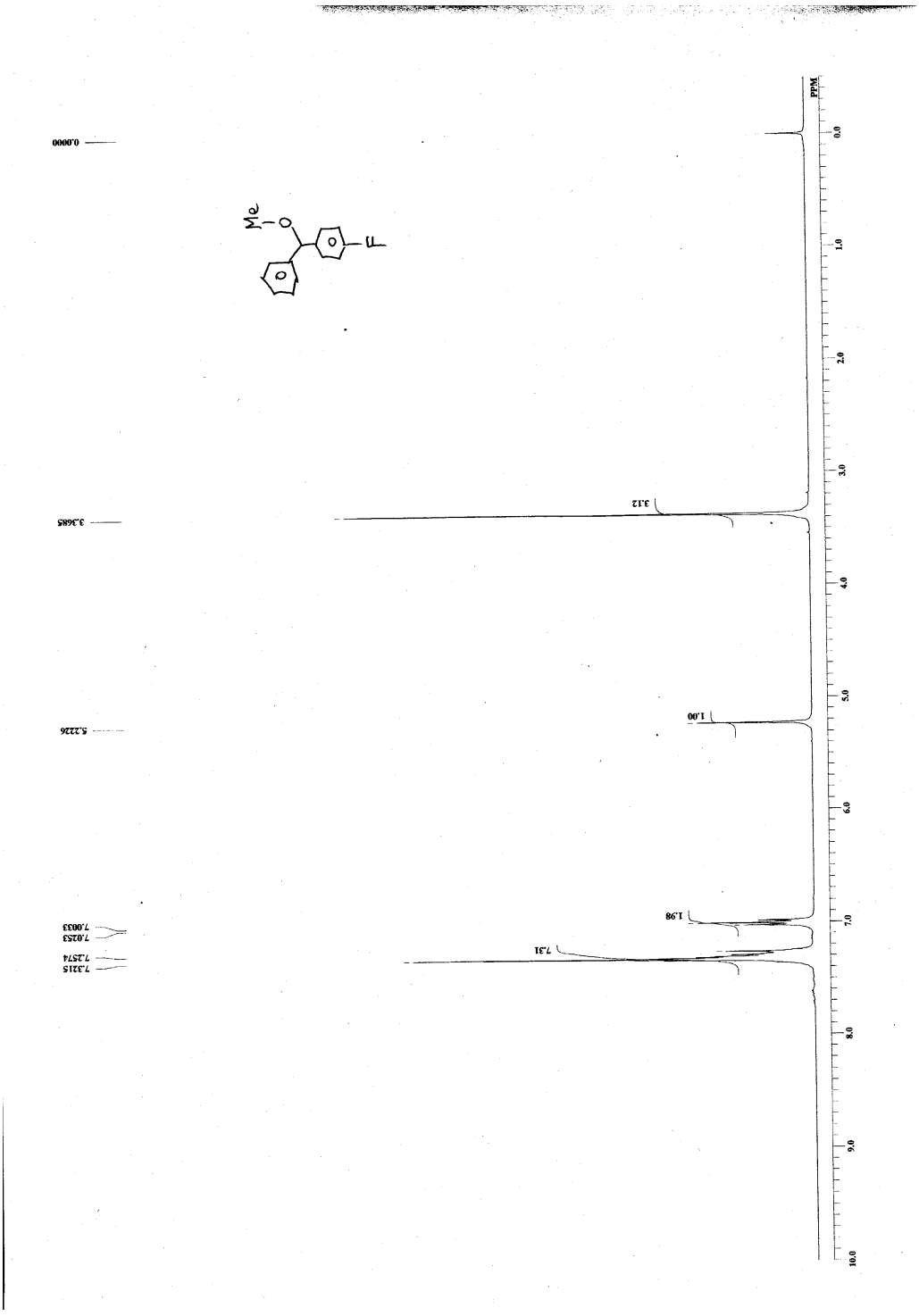

DFILE DEFAULT.ALS
COMNT wm2097-4-13C
DATIM Fri May 31 08:35:26 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.60 MHz
OBSET 125.00 KHz
OBFIN 16500.66 Hz
POINT 32768
FREQU 27110.64 Hz
SCANS 301
ACQTM 1.2083 sec
PD 1.7930 sec
PW1 5.50 usec
INTUC 1H 22.0 c
CTEMP
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 25
MeO
55.2080
56.8628
76.6789
77.0000
77.3128
84.9363
113.7508
126.7667
127.3018
128.2229
128.3309
134.2502
142.3018
158.9401
PPM
C:\Documents and Settings\JEOL\My Documents\alice_DEFAULT.ALS
wm2097-4-13C

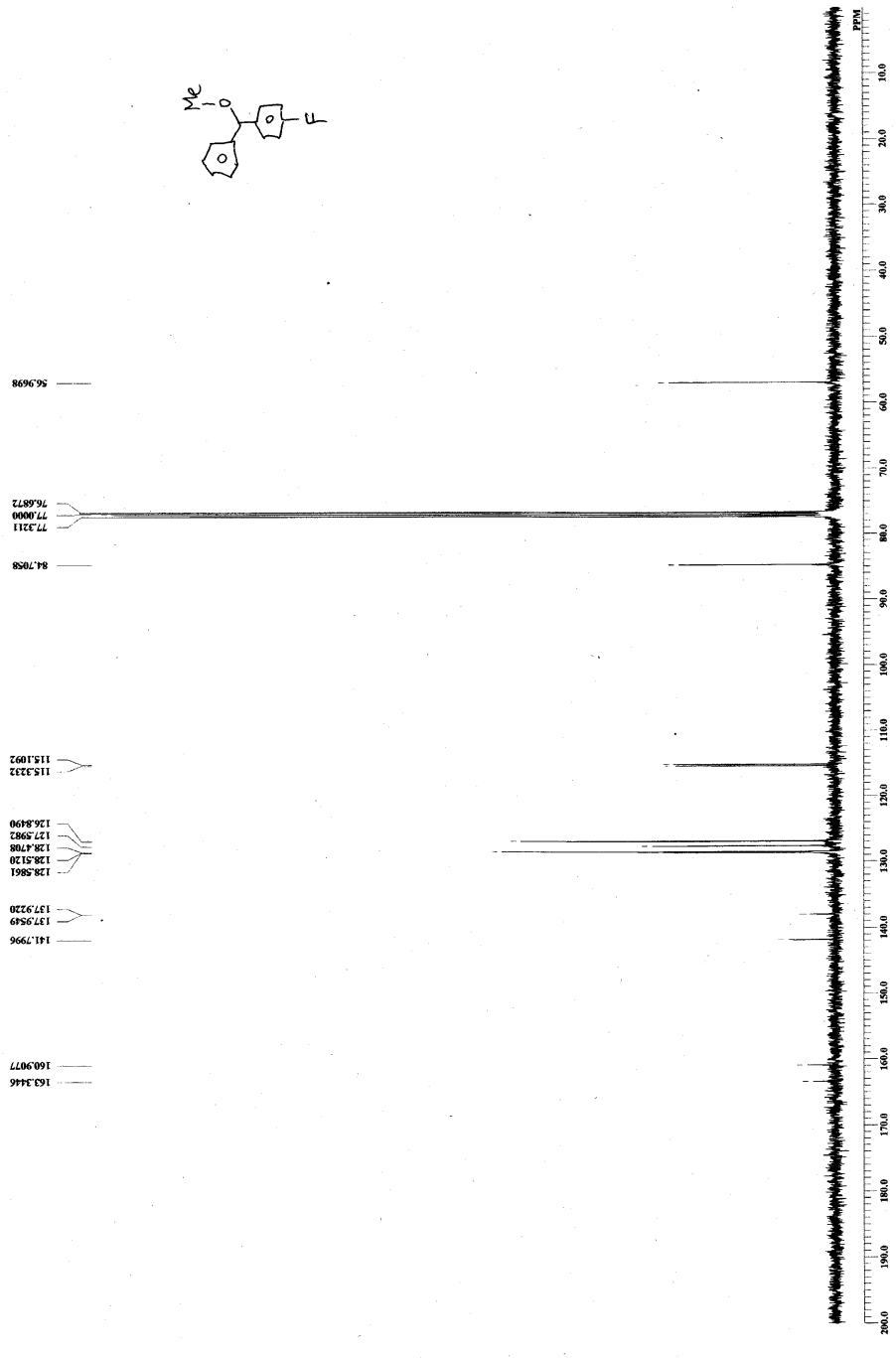
4-(Methoxyphenylmethyl)toluene (29)



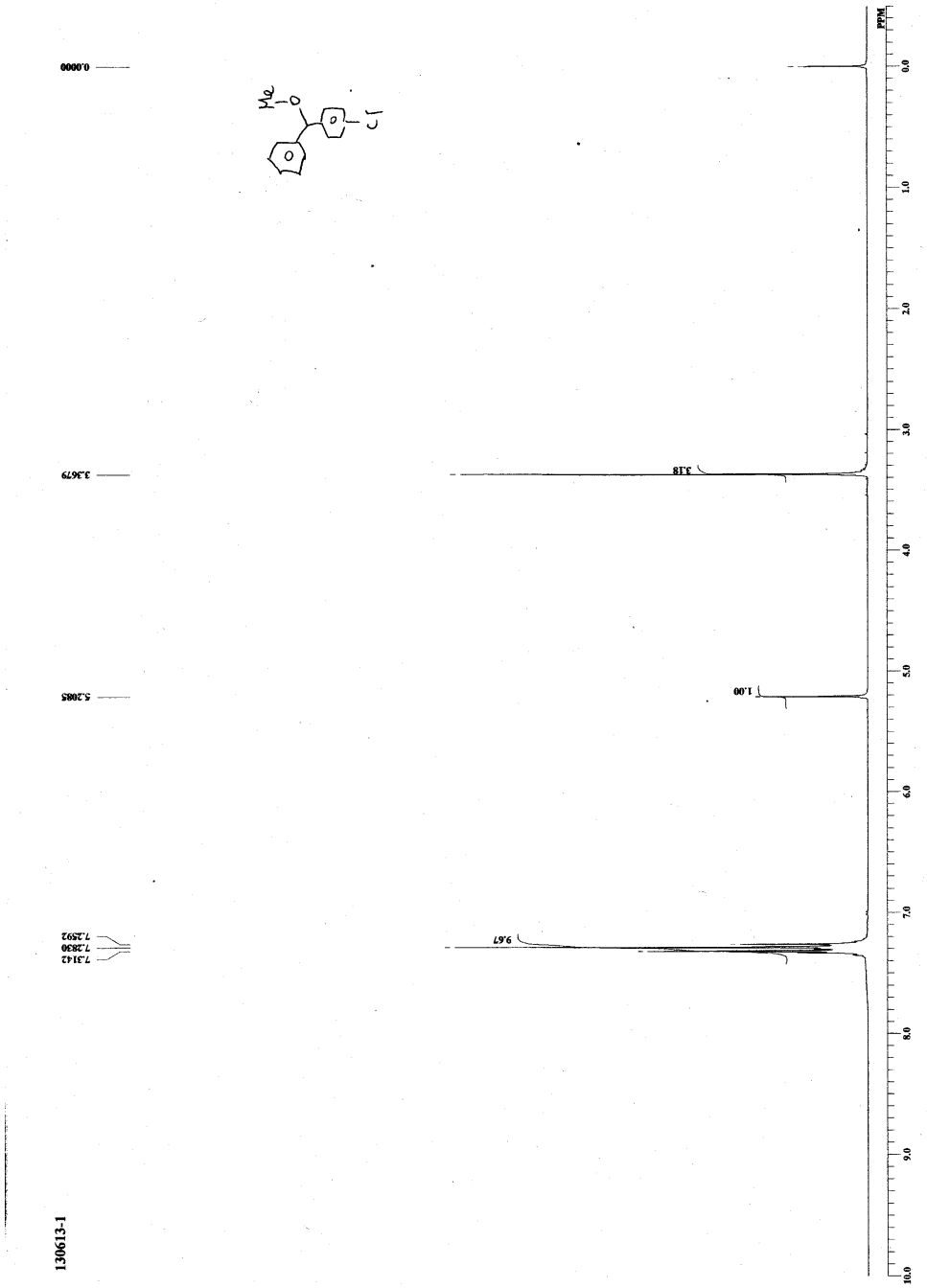

wm2094-1-13C
142.2935
139.0838
137.0987
129.0718
128.3391
127.3265
126.8819
126.8078
85.3574
77.3211
77.0000
76.6789
56.9287
21.0917
Me
O
Me
PPM
0.0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0

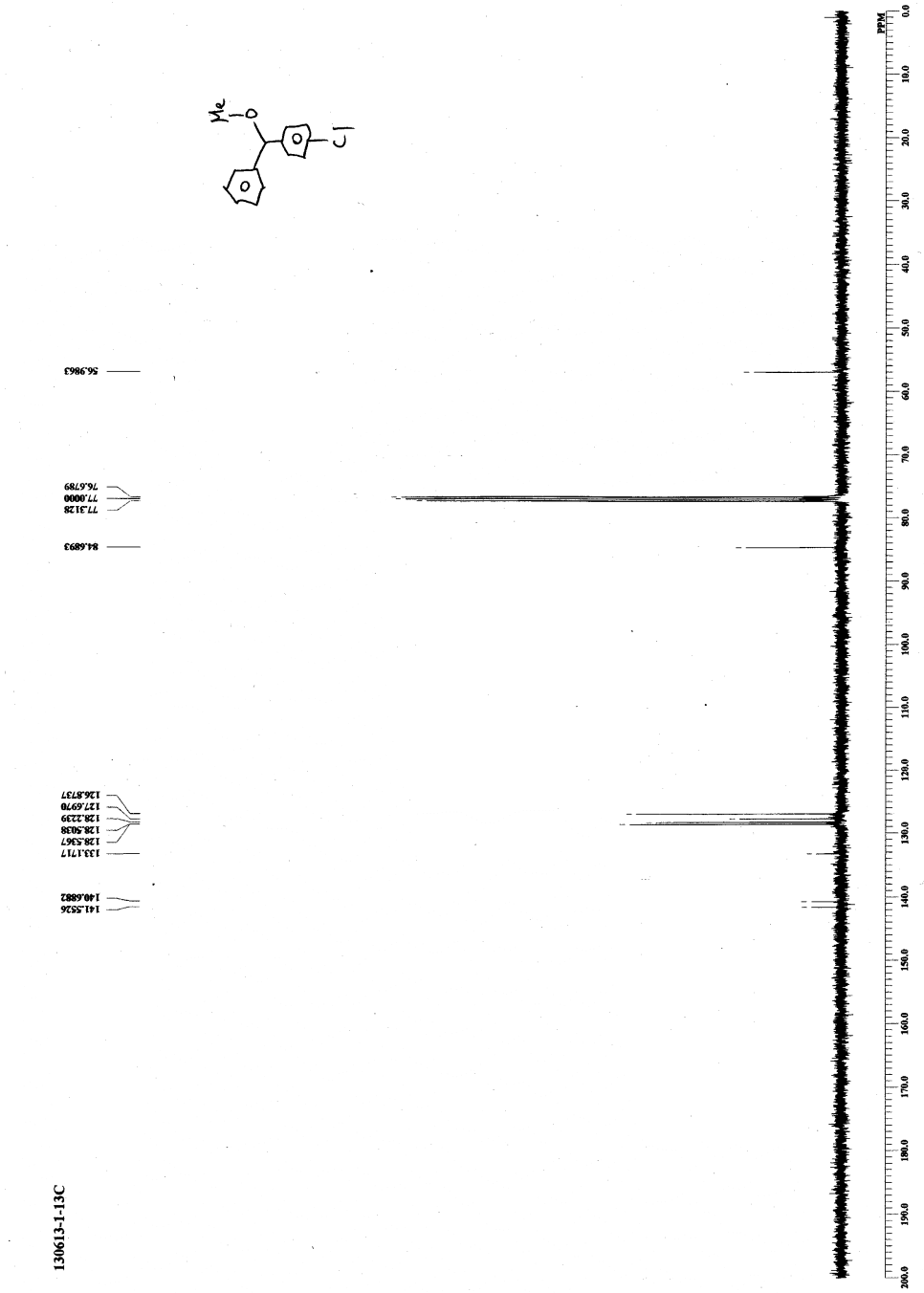
1-Fluoro-4-(methoxyphenylmethyl)benzene (30)



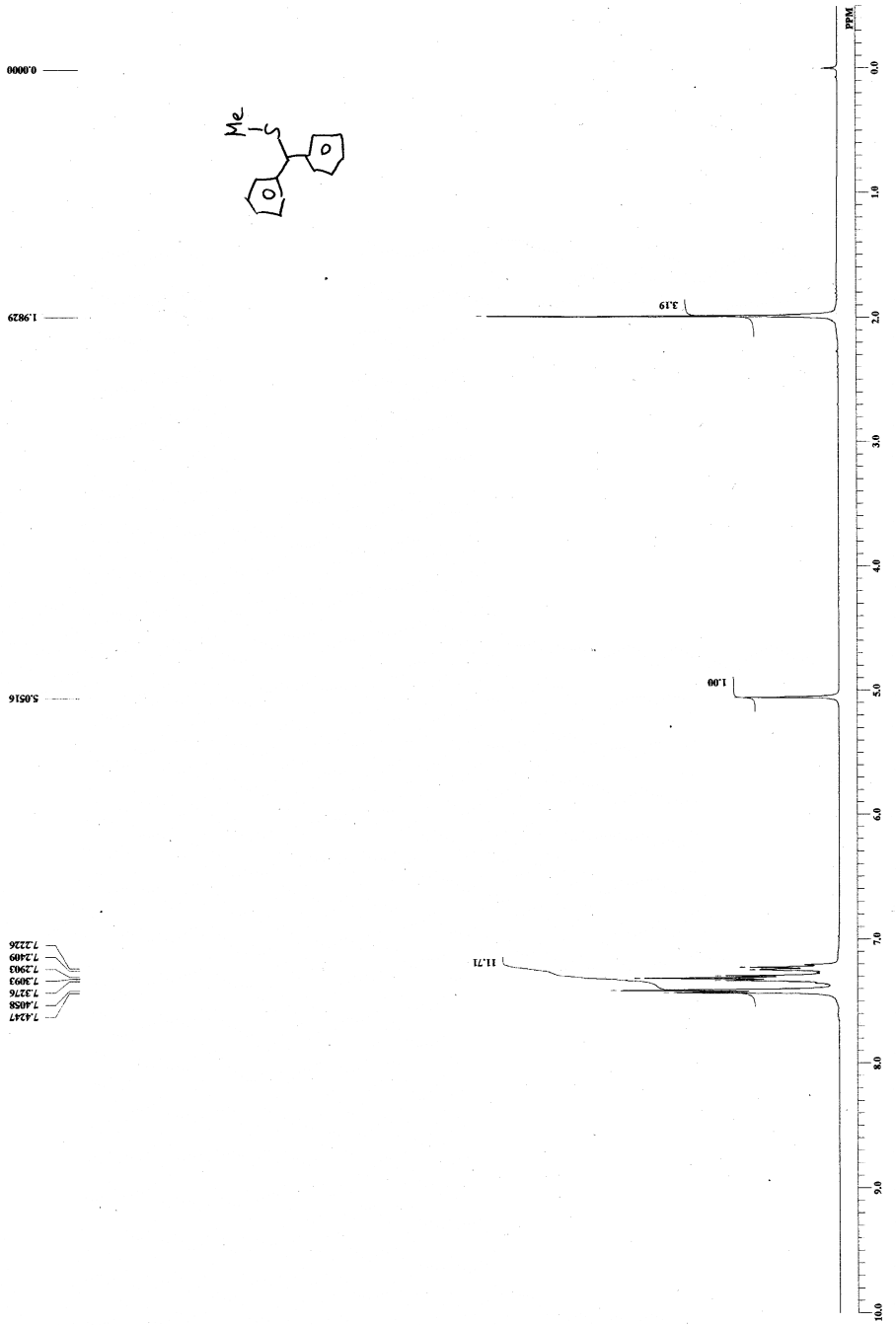

56.9698
76.6872
77.0000
77.3211
84.7058
115.1092
115.3232
126.8490
127.5982
128.4708
128.5120
128.5861
137.9220
137.9549
141.7996
160.9077
163.3446
PPM
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0

1-Chloro-4-(methoxyphenylmethyl)benzene (31)




130613-1-13C
56.9863
76.6789
77.0000
77.3128
84.6893
126.8737
127.6970
128.2239
128.5367
133.1717
140.6882
141.5526
PPM

Diphenylmethyl butyl sulfide (32)



15.8640
56.0807
77.3128
77.0000
76.6789
128.5038
128.2815
127.1124
141.2151
Me
O
O
PPM
10.0
20.0
30.0
40.0
50.0
60.0
70.0
80.0
90.0
100.0
110.0
120.0
130.0
140.0
150.0
160.0
170.0
180.0
190.0
200.0