Supporting Information

Stable Pre-formed Chiral Palladium Catalysts for the One-Pot Asymmetric Reductive Amination of Ketones

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General: All reactions and manipulations were carried out under nitrogen atmosphere by using Schlenk-type techniques. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR spectra were obtained on a JEOL GX300 Bruker-Avance 300, Varian Unity 300 (300, 75 and 121 MHz respectively), and Varian Inova Plus 500 (500 for ^{1}H and 125 MHz for ^{13}C) spectrometers using TMS as the internal reference in $CDCl_3$ as solvent at 25°C. All chemical shifts are reported in ppm (δ). Coupling constants (*J*) are reported in Hz to apparent peak multiplications. 2D NOESY and $^{1}H/^{13}C$ HSQC sequences were used for help the assignments of the ^{1}H and ^{13}C spectra. IR spectra were recorded on a Nicolet FTIR Magna 750 spectrophotometer. Optical rotations were measured on a Perkin-Elmer 343 spectropolarimeter. Mass spectra were obtained using a JEOL JMS-SX102A instrument with *m*-nitrobenzyl alcohol as the matrix (FAB^{+} mode), and a JEOL JMS-AX505-A GC/MS-EI at 70 eV. Elemental compositions were calculated within an uncertainty of 5 ppm by using the program installed in the computer system. Elemental analyses for some compounds were obtained on a Elemental Analyzer CE-440. GC-MS analyses were conducted on a Hewlett Packard 5890 (series II) instrument coupled with a JEOL JMS-AX505-A GC/MS-EI at 70 eV instrument equipped with a FID detector and a chiral capillary column Cyclodex-β (0.32 mm x 0.32 mm x 50 m) using He as a carrier gas. HPLC analyses were performed on a Hewlett Packard 1100 system with UV-DAD. Separations were achieved on a Daicel Chiracel OD-H (25 x 4.6mm) column. Flash column chromatography was performed on silica gel (70-230 mesh). X-ray determination was collected on a Bruker SMART APEX CCD area diffractometer by the ω-scan method.

Materials: All reagents were obtained from commercial suppliers and used without further purification. Molecular sieves (5 Å) were activated by flame under vacuum and stored at 200 ^{o}C. Diethyl ether and benzene were distilled from sodium-benzophenone under nitrogen. Chloroform ($CHCl_3$) was distilled from P_2O_5 under nitrogen. All other solvents were HPLC grade. $PdBr_2$ (palladium dibromide), BINAP [(*rac*)-2,2´-Bis(diphenylphosphine)-1,1´-binaphthyl], (*R*)-BINAP [(+)-2,2´-Bis(diphenylphosphine)-1,1´-binaphthyl], (*S*)-BINAP [(-)-2,2´-Bis(diphenylphosphine)-1,1´-binaphthyl], (*R*)-Tol-BINAP [(+)-2,2´-Bis(di-*p*-tolylphosphine)-1,1´- binaphthyl] and (*S*,*S*)-CHIRAPHOS [(2S,3S)-Bis(diphenylphosphino)butane] were purchased from Strem Chemical Co. The

$[(MeCN)_2]PdBr_2$ complex was prepared similar to $[(MeCN)_2]PdCl_2$ according to the previous published procedure.[1]

General procedure for [(*rac*)-BINAP]PdBr$_2$ (1a), [(*R*)-BINAP]PdBr$_2$ (1b), [(*S*)-BINAP]PdBr$_2$ (1c), **[(*R*)-Tol-BINAP]PdBr$_2$ (1d)** and **[(*S*,*S*)-CHIRAPHOS]PdBr$_2$ (1e) complexes**: These complexes were prepared by modified method described for the synthesis of [(R)-BINAP]$PdCl_2$ reported in the literature.[2] In a Schlenk tube, $[(MeCN)_2]PdBr_2$ (174 mg, 0.5 mmol) was suspended in 10 mL of benzene. Chiral diphosphine (0.5 mmol) was added. The suspension was stirred at room temperature for 24 h. The yellow-orange (**1a-1d)** or pinkish (**1e**) precipitate was collected by filtration, washed several times with diethyl ether and dried in vacuum. Each complex was pure enough for further purposes, but it can be crystallized by the slow diffusion of diethyl ether into a concentrated solution of the solid in a mixture of dichloromethane:acetone (1:1) to obtain red crystals for **1a-1d** and yellow crystals for **1e**.

[(*R*)-BINAP]PdBr$_2$ (1b): Prepared according to the general procedure from $[(MeCN)_2]PdBr_2$ (174 mg, 0.5 mmol) and [(R)-BINAP] (311 mg, 0.5 mmol) at room temperature for overnight, to provide the title compound as yellow solid (85%); ^{31}P NMR (121 MHz, $CDCl_3$) δ 25.57 (s, 2P, BINAP); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80-6.59 (m, 32H, Ar**H**); FAB MS (positive ion mode): *m/z*: 809 [M^+ - Br]; HRMS-FAB (*m/z*): calcd for $C_{44}H_{32}BrP_2Pd$ [M –Br]$^+$ 809.0177, found: 809.01850; Anal. Calcd. for $C_{44}H_{32}Br_2P_2Pd$: C, 59.45; H, 3.63. Found: C, 59.35; H 3.60; $[\alpha]^{20}_D$ +630 (c 0.18, acetone).

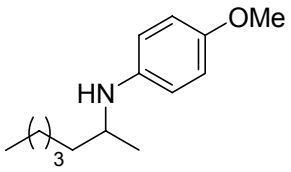
[(*S*)-BINAP]PdBr$_2$ (1c): $[\alpha]^{20}_D$ -635 (c 0.18, acetone).

[(*R*)-Tol-BINAP]PdBr$_2$ (1d): Prepared according to the general procedure from $[(MeCN)_2]PdBr_2$ (174 mg, 0.5 mmol) and [(R)-Tol-BINAP] (472 mg, 0.5 mmol) at room temperature for 24 h, to provide the title compound as orange solid (88%); ^{31}P NMR (121 MHz, $CDCl_3$) δ 28.35 (s, 2P, Tol-BINAP); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75-7.68 (m, 4H, Ar**H**), 7.56 – 7.11 (m, 18H, Ar**H**), 6.75 (d, 2H, J = 7.15 Hz, Ar**H**), 6.44 (d, 2H, J = 8.5 Hz, Ar**H**), 2.36 (s, 6H, -C**H**$_3$), 1.98 (s, 6H, -C**H**$_3$); FAB MS (positive ion mode): *m/z*: 865

[M^+ - Br]; Anal. Calcd. for $C_{48}H_{40}Br_2P_2Pd$: C, 61.01; H, 4.27. Found: C, 60.11; H 4.25; $[\alpha]^{20}_D$ +641.1 (c 0.18, acetone).

[(*S*,*S*)-CHIRAPHOS]PdBr$_2$ (1e): Prepared according to the general procedure from $[(MeCN)_2]PdBr_2$ (174 mg, 0.5 mmol) and [(S,S)-CHIRAPHOS] (213 mg, 0.5 mmol) at room temperature for 24 h, to provide the title compound as pinkish solid (78%); ^{31}P NMR (121 MHz, $CDCl_3$) δ 64.3 (s, 2P, BINAP); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 – 7.23 (m, 20H, Ar**H**), 2.41 (m, 2H, -C**H**CH_3), 1.06 (dd, 6H, *J* = 4.9, 7.9 Hz, -CHC**H**$_3$); FAB MS (positive ion mode): *m/z*: 613 [M^+ - Br]; Anal. Calcd. for $C_{28}H_{28}Br_2P_2Pd$: C, 48.55; H, 4.07. Found: C, 48.29; H 4.05; $[\alpha]^{20}_D$ +113.75 (c 0.2, CH_3CN/CH_2Cl_2).

General procedure for asymmetric reductive amination of alkyl ketones: 1.0 mmol of the carbonyl compound, 1.5 mmol of aniline derivative were added to a stirred solution of 0.025 mmol of chiral palladium complex in 10 mL of dry $CHCl_3$ (in a Schlenk tube) and stirred under nitrogen atmosphere for 10 minutes. The solution was transferred to a 45 ml stainless steel autoclave (PARR), which contains 150 mg of activated molecular sieves 5Å previously purged with vacum-N_2. Subsequently, the reaction was taken to the desired pressure (800 psi H_2), stirred in an oil bath at 70°C for 24 h. At the end of this period, the gas was liberated. The solution was analyzed by GC-MS to quantify the remaining substrate, and was later concentrated under reduced pressure, affording a crude residue, which was purified by column chromatography over silica gel (70-230 mesh), and eluted with hexane-ethyl acetate (99/1) to isolate the product.

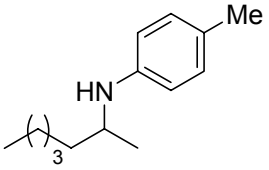


4a

***(-)-N*-(4-methoxyphenyl)-[1-(methyl)-hexyl]amine (4a) (Table 1):** Prepared according to the general procedure from 2-heptanone (140 μL, 1.0 mmol), *p*-anisidine (185 mg, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 ^{o}C for 24 h, to provide the title compound as a yellow oil (78%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.77 (d, 2H, *J* = 9.0

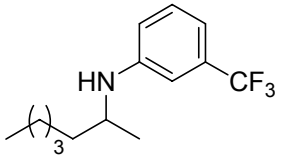
Hz, Ar**H**), 6.55 (d, 2H, J = 8.7 Hz, Ar**H**), 3.74 (s, 3H, -OC**H**$_3$), 3.36 (sext, 1H, J = 6.0 Hz, -C**H**CH$_3$), 2.99 (bs, 1H, -N**H**CH), 1.57-1.28 (m, 8H, -CH$_2$), 1.14 (d, 3H, J = 6.3 Hz, -CHC**H**$_3$), 0.89 (t, 3H, J = 6.6 Hz, -CH$_2$C**H**$_3$); ^{13}C NMR (75 MHz, CDCl$_3$) δ 151.7, 141.9, 114.9, 114.6, 55.8, 49.5, 37.1, 31.9, 25.8, 22.6, 20.7, 14.0; IR(neat) 3405, 2959, 2929, 1618, 1518, 1181, 807 cm^{-1}; EIMS (70 eV) *m/z* 221 (M$^+$); HRMS-EI *m/*z calcd for $C_{14}H_{23}ON$ (M$^+$) 221.1780, found 221.1775; $[\alpha]^{20}_D$ -2.0 (c 0.4, CHCl$_3$); 76% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 95/5, flow rate = 1 mL/min, t_R = 3.1 min (major), t_R = 4.5 min (minor).

With [(*R*)-Tol-BINAP]PdBr$_2$ (**1d**): 77% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 98/2, flow 1 mL/min, t_r = 4.2 min (minor), t_r = 7.4 min (major). [(*S*,*S*)-CHIRAPHOS]PdBr$_2$ (**1e**): 14% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 98/2, flow 1 mL/min, t_R = 4.2 min (minor), t_R = 7.5 min (major).



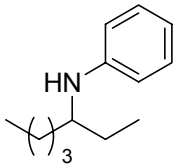
4b

***(-)-N*-(4-methylphenyl)-[1-(methyl)-hexyl]amine (4b) (Table 2, entry 1):** Prepared according to the general procedure from 2-heptanone (140 μL, 1.0 mmol), *p*-toluidine (161 mg, 1.5 mmol) and [(*R*)-BINAP]PdBr$_2$, **1b**, (22 mg, 0.025 mmol) at 70 ^{o}C for 24 h, to provide the title compound as yellow oil (84%); ^{1}H NMR (300 MHz, CDCl$_3$) δ 6.99 (d, 2H, J = 7.9 Hz, Ar**H**), 6.52 (d, 2H, J = 8.2 Hz, Ar**H**), 3.43 (sext, 1H, J = 6.0 Hz, -C**H**CH$_3$), 3.28 (bs, 1H, -N**H**CH), 2.25 (s, 3H, -CH$_3$), 1.59-1.30 (m, 8H, -C**H**$_2$), 1.17 (d, 3H, J = 6.3 Hz, -CHC**H**$_3$), 0.91 (t, 3H, J = 6.7 Hz, -CH$_2$C**H**$_3$); ^{13}C NMR (75 MHz, CDCl$_3$) δ 145.5, 129.8, 126.0, 113.4, 48.8, 37.3, 32.0, 25.9, 22.7, 20.9, 20.4, 14.1; IR(neat) 3402, 2958, 2927, 1618, 1181, 806 cm^{-1}; EIMS (70 eV) *m/z* 205 (M$^+$); Anal. Calcd (%) for $C_{14}H_{23}N$ (205.1830): C, 82.13; H, 11.49; N, 6.39. Found: C, 82.11; H, 11.50; N, 6.38; HRMS-EI *m/*z calcd for $C_{14}H_{23}N$ (M$^+$) 205.1830, found 205.1830; $[\alpha]^{20}_D$ -2.0 (c 0.4, CHCl$_3$); 73% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 3.1 min (minor), t_R = 3.4 min (major).



4c

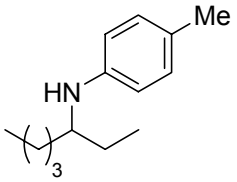
***(-)-N*-(3-trifluoromethylphenyl)-[1-(methyl)-hexyl]amine (4c) (Table 2, entry 2):** Prepared according to the general procedure from 2-heptanone (140 μL, 1.0 mmol), *m*-trifluoromethyl aniline (180 μL, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as colorless oil (51%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.21 (d, 1H, *J* = 7.9 Hz, Ar**H**), 6.87 (d, 1H, *J* = 8.2 Hz, Ar**H**), 6.75 (s, 1H, Ar**H**), 6.69 (d, 1H, *J* = 8.2 Hz, Ar**H**), 3.65 (bs, 1H, -N**H**CH), 3.47 (sext, 1H, *J* = 6.3 Hz, -C**H**CH_3), 1.57-1.28 (m, 8H, -C$\mathbf{H}_2$), 1.18 (d, 3H, *J* = 6.3 Hz, -CHC$\mathbf{H}_3$), 0.89 (t, 3H, *J* = 6.6 Hz, -CH_2C$\mathbf{H}_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.8, 131.6 (Cq, *J* = 32.3Hz), 129.7, 124.4 (Cd, *J* = 272.3 Hz), 115.9, 113.1(Cq, *J* = 4.0Hz), 109.1(Cq, *J* = 4.0Hz), 48.5, 37.1, 31.9, 25.8, 22.7, 20.6, 14.1; IR(neat) 3426, 2961, 2931, 1614, 1517, 1162, 857 cm^{-1}; EIMS (70 eV) *m*/z 259 (M^+); HRMS-EI *m/*z calcd for $C_{14}H_{20}NF_3$ (M^+) 259.1548, found 259.1545; $[\alpha]^{20}_D$ -1.6 (c 0.4, $CHCl_3$); 95% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 3.0 min (minor), t_R = 3.6 min (major).



4d

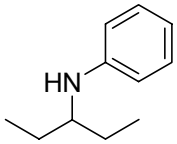
***(+)-N*-phenyl-[1-(ethyl)-pentyl]amine (4d) (Table 2, entry 3):** Prepared according to the general procedure from 3-heptanone (140 μL, 1.0 mmol), aniline (0.13 μL, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as colorless oil (86%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.15 (td, 2H, *J* = 8.7, 1.5 Hz, Ar**H**), 6.64 (tt, 1H, *J* = 1.2, 7.2 Hz, Ar**H**), 6.57 (dd, 1H, *J* = 1.2, 8.5 Hz, Ar**H**), 3.46 (bs, 1H, -N**H**CH), 3.28 (quint, 1H, *J* = 6.0 Hz, -C**H**CH_2), 1.74 – 1.26 (m, 8H, C$\mathbf{H}_2$), 0.92 (t, 3H, *J* = 7.5 Hz, -CH_2C$\mathbf{H}_3$), 0.89 (t, 3H, *J* = 7.2 Hz, -CH_2C$\mathbf{H}_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.1, 129.2, 116.4, 112.8, 54.0, 34.0, 28.1, 27.2, 22.8, 14.0, 10.0; IR(neat) 3405, 2959, 2929, 1602, 1504, 1179, 865 cm^{-1}; EIMS (70 eV) *m/z* 191 (M^+); $[\alpha]^{20}_D$ +2.72 (c 0.44,

$CHCl_3$); 49% ee by GC-MS (EI) (column: Ciclodex-β, flow rate = 1 grade/min, t_R = 28.4 min (minor), t_R = 28.7 min (major).



4e

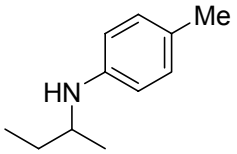
***(-)-N*-(4-methylphenyl)-[1-(ethyl)-pentyl]-amine (4e) (Table 2, entry 4):** Prepared according to the general procedure from 3-heptanone (140 μL, 1.0 mmol), *p*-toluidine (161 mg, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1c**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as colorless oil (82%); ^1H NMR (300 MHz, $CDCl_3$) δ 6.97 (d, 2H, *J* = 8.5 Hz, Ar**H**), 6.50 (d, 1H, *J* = 8.4 Hz, Ar**H**), 3.31 (bs, 1H, -N**H**CH), 3.25 (quint, 1H, *J* = 6.0 Hz, -C**H**CH_2), 3.04 (s, 3H, -C$\mathbf{H_3}$), 1.63 – 1.27 (m, 8H, -C$\mathbf{H_2}$), 0.92 (t, 3H, *J* = 7.2 Hz, -CH_2C$\mathbf{H_3}$), 0.90 (t, 3H, *J* = 7.2 Hz, -CH_2C$\mathbf{H_3}$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.9, 129.7, 125.6, 113.0, 54.3, 34.1, 28.1, 27.2, 22.8, 20.3, 14.0, 10.0; IR(neat) 3404, 2959, 2929, 1618, 1518, 1151, 806 cm^{-1}; EIMS (70 eV) *m/z* 205 (M^+); HRMS-EI *m/z* calcd for $C_{14}H_{23}N$ 205.1830 (M^+), found 205.1833; $[\alpha]^{20}_D$ -1.5 (c 0.54, $CHCl_3$); 59% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 3.0 min (minor), t_R = 3.4 min (major).



4f

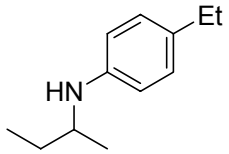
***N*-phenyl-[1-(ethyl)-propyl]amine (4f) (Table 2, entry 5):** Prepared according to the general procedure from 3-pentanone (100 μL, 1.0 mmol), aniline (130 μL, 1.5 mmol) and [(*rac*)-BINAP]$PdBr_2$, **1a**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as light yellow oil (77%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.17 (td, 2H, *J* = 8.5, 1.1 Hz, Ar**H**), 6.61 (t, 1H, *J* = 8.2 Hz, Ar**H**), 6.59 (d, 2H, *J* = 8.5 Hz, Ar**H**), 3.44 (bs, 1H, -N**H**CH), 3.25 (quint, 1H, *J* = 5.8 Hz, -C**H**CH_2), 1.65 – 1.45 (m, 4H, -C$\mathbf{H_2}$), 0.94 (t, 6H, *J* = 7.4 Hz, -CH_2C$\mathbf{H_3}$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.3, 129.3, 116.6, 113.0, 55.4, 26.8,

10.2; IR(neat) 3403, 2963, 2930, 1602, 1505, 1179, 865 cm^{-1}; EIMS (70 eV) *m/z* 163 (M^+); HRMS-EI *m/z* calcd for $C_{11}H_{17}N$ (M^+) 163.1361, found 163.1360.



4g

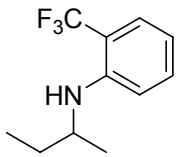
***(-)-N-sec*-butyl-(*p*-tolyl)amine (4g) (Table 2, entry 6):** Prepared according to the general procedure from 2-butanone (90 μL, 1.0 mmol), *p*-toluidine (161 mg, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1c**, (22 mg, 0.025 mmol) at 70 °C for 24 h, to provide the title compound as yellow oil (77%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.97 (d, 2H, *J* = 8.2 Hz, Ar**H**), 6.51 (d, 2H, *J* = 8.5 Hz, Ar**H**), 3.36 (sext, 1H, *J* = 6.4 Hz, -C**H**CH_3), 3.26 (bs, 1H, -N**H**CH), 2.23 (s, 3H, C**H**$_3$), 1.64 – 1.55 (m, 1H, -C**H$_2$**CH_3), 1.50 – 1.39 (m, 1H, -C**H$_2$**CH_3), 1.16 (d, 3H, *J* = 6.3 Hz, -CHC**H**$_3$), 0.94 (t, 3H, *J* = 7.4 Hz, -CH_2C**H**$_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.5, 129.8, 126.0, 113.4, 50.1, 29.7, 20.4, 20.3, 10.4; IR(neat) 3399, 2964, 2924, 1618, 1518, 1160, 807 cm^{-1}; EIMS (70 eV) *m/z* 163 (M^+); $[\alpha]^{20}_D$ -1.0 (c 0.44, $CHCl_3$); >99% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 95/5, flow rate = 1 mL/min, t_R = 5.0 min (major), t_R = 5.9 min (minor).



4h

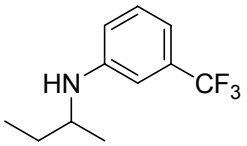
***(-)-N-sec*-butyl-(4-ethylphenyl)amine (4h) (Table 2, entry 7):** Prepared according to the general procedure from 2-butanone (90 μL, 1.0 mmol), *p*-ethyl aniline (190 μL, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 °C for 24 h, to provide the title compound as yellow oil (77%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.03 (d, 2H, *J* = 8.5 Hz, Ar**H**), 6.55 (d, 2H, *J* = 8.5 Hz, Ar**H**), 3.44 – 3.33 (m, 2H, -N**H**CH + -C**H**CH_3), 2.55 (q, 2H, *J* = 7.5 Hz, -C**H$_2$**CH_3), 1.66 – 1.55 (m, 1H, -C**H$_2$**CH_3), 1.52 – 1.40 (m, 1H, -C**H$_2$**CH_3), 1.22 (d, 3H, *J* = 7.6 Hz, -CH_2C**H$_3$**), 1.18 (d, 3H, *J* = 6.3 Hz, -CH_2C**H$_3$**); ^{13}C NMR (75 MHz,

$CDCl_3$) δ 145.7, 132.7, 128.6, 113.3, 50.1, 29.7, 28.0, 20.4, 16.0, 10.5; IR(neat) 3402, 2963, 2927, 1616, 1518, 1158, 819 cm^{-1}; EIMS (70 eV) *m/z* 177 (M^+); $[\alpha]^{20}_D$ -2.80 (c 0.5, $CHCl_3$); 92% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 90/10, flow rate = 1 mL/min, t_R = 3.2 min (minor), t_R = 3.7 min (major).



4i

***(+)-N-sec*-butyl-(2-trifluoromethylphenyl)amine (4i) (Table 2, entry 8):** Prepared according to the general procedure from 2-butanone (90 μL, 1.0 mmol), *o*-trifluoromethyl aniline (180 μL, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1c**, (22 mg, 0.025 mmol) at 70 °C for 24 h, to provide the title compound as colorless oil (76%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (d, 1H, *J* = 7.7 Hz, Ar**H**), 7.33 (t, 1H, *J* = 7.9 Hz, Ar**H**), 6.71 (d, 1H, *J* = 8.5 Hz, Ar**H**), 6.65 (t, 1H, *J* = 7.6 Hz, Ar**H**), 4.13 (bs, 1H, -N**H**CH), 3.48 (sext, 1H, *J* = 6.5 Hz, -C**H**CH_3), 1.53 – 1.51 (m, 2H, -C**H$_2$**CH_3), 1.20 (d, 3H, *J* = 6.3 Hz, -CHC**H$_3$**), 0.96 (t, 3H, *J* = 7.4 Hz, -CH_2C**H$_3$**); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.3, 133.0, 126.8 (Cq, *J* = 5.3 Hz), 123.5, 115.2, 113.2 (Cq, *J* = 29.4 Hz), 112.3, 49.7, 29.5, 20.1, 10.2; IR(neat) 3468, 2969, 2929, 1615, 1586, 1168, 941 cm^{-1}; EIMS (70 eV) *m/z* 217 (M^+); $[\alpha]^{20}_D$ +3.33 (c 0.36, $CHCl_3$); 82% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 3.8 min (major), t_R = 4.6 min (minor).

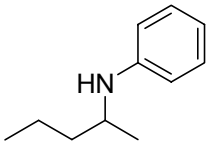


4j

***(S)-N-sec*-butyl-(3-trifluoromethylphenyl)amine (4j) (Table 2, entry 9):** Prepared according to the general procedure from 2-butanone (90 μL, 1.0 mmol), *m*-trifluoromethyl (180 μL, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 °C for 24 h, to provide the title compound as colorless oil (71%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23 (t,

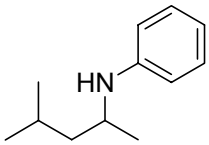
1H, J = 7.9 Hz, Ar**H**), 6.87 (d, 1H, J = 7.4 Hz, Ar**H**), 6.76 (s, 1H, Ar**H**), 6.70 (d, 1H, J = 8.2 Hz, Ar**H**), 3.65 (bs, 1H, -N**H**CH), 3.42 (sext, 1H, J = 6.0 Hz, -C**H**CH_3), 1.64 – 1.45 (m, 2H, -C**H_2**CH_3), 1.18 (d, 3H, J = 6.0 Hz, -CHC**H_3**), 0.96 (t, 3H, J = 7.4 Hz, -CH_2C**H_3**); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.9, 131.5 (Cq, J = 31.7Hz), 129.7, 122.7, 115.9, 113.1 (Cq, J = 4.0 Hz), 109.1 (Cq, J = 4.0 Hz), 49.8, 29.6, 20.1, 15.8; IR(neat) 3425, 2970, 2932, 1614, 1517, 1163, 859 cm^{-1}; EIMS (70 eV) *m/z* 217 (M^+); HRMS-EI *m/*z calcd for $C_{11}H_{14}NF_3$ (M^+) 217.1078, found 217.1083; $[\alpha]^{20}_D$ -3.01 (c 0.53, $CHCl_3$); 75% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 3.0 min (minor), t_R = 3.8 min (major).

To determine the absolute configuration of this compound was derivatized by hydrogenolysis in presence of Pd/C and salt formation with HCl in methanol to obtain 2-butylamine hydrochloride. The obtained ammonium salt (2-butylamine hydrochloride) has optical rotation $[\alpha]^{20}_D$ –4.8 (c 0.25, EtOH). This was compared with the optical rotation of reported 2-butylamine hydrochloride.[4]



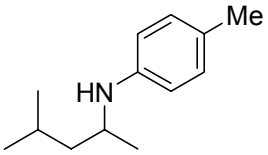
4k

***(-)-N*-phenyl-[1-(methyl)-butyl]amine (4k) (Table 2, entry 10):** Prepared according to the general procedure from 3-penten-2-one (100 μL, 1.0 mmol), aniline (130 μL, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1c**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (78%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.02 (d, 2H, J = 8.2 Hz, Ar**H**), 6.55 (d, 2H, J = 8.2 Hz, Ar**H**), 3.48 (quint, 1H, J = 6.0 Hz, -C**H**CH_3), 3.31 (bs, 1H, -N**H**CH), 1.62 – 1.39 (m, 4H, -C**H_2**CH_3), 1.20 (d, 3H, J = 6.3 Hz, -CH_2C**H_3**), 0.97 (t, 3H, J = 7.0 Hz, -CH_2C**H_3**); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.6, 129.9, 126.0, 113.4, 48.6, 39.6, 20.9, 20.5, 19.4, 14.3; IR(neat) 3383, 2959, 2924, 1616, 1513, 1172, 806 cm^{-1}; EIMS (70 eV) *m/z* 177 (M^+); HRMS-EI *m/*z calcd for $C_{12}H_{19}N$ (M^+) 177.1517, found 177.1518; $[\alpha]^{20}_D$ -3.40 (c 0.47, $CHCl_3$); 10% ee by CG-MS (column: Ciclodex-β, flow rate = 1 grade/min, t_R = 20.9 min (major), t_R = 21.3 min (minor).



4l

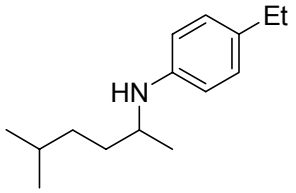
***(-)-N*-phenyl-[1-(methyl)-3-(methyl)-butyl]amine (4l) (Table 2, entry 11):** Prepared according to the general procedure from 4-methyl-2-pentanone (120 μL, 1.0 mmol), aniline (130 μL, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1c**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (83%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.17 (t, 2H, *J* = 7.4 Hz, Ar**H**), 6.66 (t, 1H, *J* = 7.4 Hz, Ar**H**), 6.58 (d, 2H, *J* = 7.6 Hz, Ar**H**), 3.53 (sext, 1H, *J* = 6.3 Hz, -C**H**CH_3), 3.39 (bs, 1H, -N**H**CH), 1.80 – 1.71 (m, 1H, -C**H**$_2CH_3$), 1.52 – 1.43 (m, 1H, -C**H**CH), 1.31 – 1.23 (m, 1H, -C**H$_2$**CH), 1.16 (d, 3H, *J* = 6.3 Hz, -CHC**H**$_3$), 0.95 (d, 3H, *J* = 6.6 Hz, -CHC**H**$_3$), 0.91 (d, 3H, *J* = 6.3 Hz, -CHC**H**$_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.8, 129.3, 116.8, 113.0, 47.0, 46.5, 25.1, 23.0, 22.6, 21.1; IR(neat) 3402, 2958, 2927, 1602, 1504, 1160, 866 cm^{-1}; EIMS (70 eV) *m/z* 177 (M^+); HRMS-EI *m/*z calcd for $C_{12}H_{19}N$ (M^+) 177.1517, found 177.1511; $[\alpha]^{20}_D$ -1.27 (c 0.47, $CHCl_3$); 51% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 4.0 min (major), t_R = 4.7 min (minor).



4m

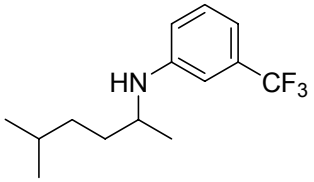
***(-)-N*-(4-methylphenyl-[1-(methyl)-3-(methyl)-butyl]amine (4m) (Table 2, entry 12):** Prepared according to the general procedure from 4-methyl-2-pentanone (120 μL, 1.0 mmol), *p*-toluidine (161 mg, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1c**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (73%); ^1H NMR (300 MHz, $CDCl_3$) δ 6.97 (d, 2H, *J* = 8.7 Hz, Ar**H**), 6.51 (d, 2H, *J* = 8.4 Hz, Ar**H**), 3.49 (sext, 1H, *J* = 6.6 Hz, -C**H**CH_3), 3.26 (bs, 1H, -N**H**CH), 2.23 (s, 3H, C**H**$_3$) 1.81 – 1.68 (m, 1H, -C**H**$_2CH_3$), 1.50 – 1.41 (m, 1H, -C**H**CH), 1.28 – 1.19 (m, 1H, -C**H$_2$**CH), 1.14 (d, 3H, *J* = 6.3 Hz, -CHC**H**$_3$), 0.93 (d, 3H, *J* = 6.6 Hz, -CHC**H**$_3$), 0.90 (d, 3H, *J* = 6.3 Hz, -CHC**H**$_3$); ^{13}C NMR

(75 MHz, $CDCl_3$) δ 145.4, 129.7, 125.9, 113.2, 46.9, 46.8, 25.0, 22.9, 22.6, 22.5, 21.0; IR(neat) 3397, 2957, 2924, 1617, 1517, 1162, 880 cm^{-1}; EIMS (70 eV) *m/z* 191 (M^+); $[\alpha]^{20}_D$ -4.25 (c 0.40, $CHCl_3$); 90% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 95/5, flow rate = 1 mL/min, t_R = 4.9 min (major), t_R = 5.4 min (minor).



4n

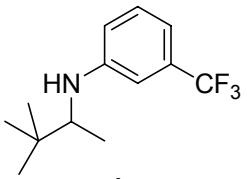
***(-)-N*-(4-ethylphenyl)-[1-(methyl)-4-(methyl)-pentyl]amine (4n) (Table 2, entry 13):** Prepared according to the general procedure from 5-methyl-2-hexanone (125 μL, 1.0 mmol), *p*-ethyl aniline (190 μL, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1c**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (80%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.00 (d, 2H, *J* = 8.5 Hz, Ar**H**), 6.52 (d, 2H, *J* = 8.5 Hz, Ar**H**), 3.44 – 3.33 (m, 2H, -C**H**CH_3 + -N**H**), 2.54 (q, 2H, *J* = 7.6 Hz, C**H**$_2CH_3$), 1.64 – 1.24 (m, 5H, -C**H**$(CH_3)_2$ + -C**H**$_2CH_3$), 1.19 (t, 3H, *J* = 7.6 Hz, -CH_2C**H**$_3$), 1.16 (d, 3H, *J* = 6.3 Hz, -CHC**H**$_3$), 0.89 (d, 3H, *J* = 6.6 Hz, -CHC**H**$_3$), 0.88 (d, 3H, *J* = 6.6 Hz, -CHC**H**$_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.7, 132.6, 128.6, 113.2, 49.1, 35.4, 35.1, 28.2, 27.9, 22.8, 22.7, 20.9, 16.0; IR(neat) 3403, 2959, 2928, 1616, 1518, 1158, 818 cm^{-1}; EIMS (70 eV) *m/z* 219 (M^+); HRMS-EI *m/z* calcd for $C_{15}H_{25}N$ (M^+) 219.1987, found 219.1984; $[\alpha]^{20}_D$ -0.70 (c 0.43, $CHCl_3$); 83% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 95/5, flow rate = 1 mL/min, t_R = 3.6 min (major), t_R = 4.9 min (minor).



4o

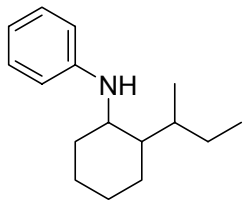
***(-)-N*-(3-trifluoromethylphenyl)-[1-(methyl)-4-(methyl)-pentyl]amine (4o) (Table 2, entry 14):** Prepared according to the general procedure from 5-methyl-2-hexanone (125 μL, 1.0 mmol), *m*-trifluoromethyl aniline (180 μL, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1c**,

(22 mg, 0.025 mmol) at 70 ^{o}C for 24 h, to provide the title compound as colorless oil (71%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25 (t, 1H, *J* = 7.7 Hz, Ar**H**), 6.90 (d, 1H, *J* = 7.7 Hz, Ar**H**), 6.79 (s, 2H, 1H, Ar**H**), 6.71 (d, 1H, *J* = 7.9 Hz, Ar**H**), 3.63 (bs, 1H, -N**H**CH), 3.47 (sext, 1H, *J* = 6.3 Hz, -C**H**CH_3), 1.64 – 1.25 (m, 5H, -C**H**$(CH_3)_2$ + -C$\mathbf{H}_2CH_3$), 1.20 (d, 3H, *J* = 6.3 Hz, -CHC$\mathbf{H}_3$), 0.93 (d, 3H, *J* = 6.6 Hz, -CHC$\mathbf{H}_3$), 0.92 (d, 3H, *J* = 6.6 Hz, -CHC$\mathbf{H}_3$); IR(neat) 3425, 2960, 2931, 1614, 1516, 1164, 858 cm^{-1}; EIMS (70 eV) *m/z* 259 (M^+); HRMS-EI *m/*z calcd for $C_{14}H_{20}NF_3$ (M^+) 259.1548, found 259.1545; $[\alpha]^{20}_D$ -2.08 (c 0.24, $CHCl_3$); 82% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 90/10, flow rate = 1 mL/min, t_R = 3.8 min (major), t_R = 5.1 min (minor).



4p

***(-)-N*-(3-trifluoromethylphenyl)-[1-(methyl)-2,2-(dimethyl)-propyl]-amine (4p) (Table 2, entry 15):** Prepared according to the general procedure from 3,3-dimethyl-2-butanone (90 μL, 1.0 mmol), *m*-trifluoromethyl aniline (180 μL, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 ^{o}C for 24 h, to provide the title compound as colorless oil (89%); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 (t, 1H, *J* = 7.9 Hz, Ar**H**), 6.85 (d, 1H, *J* = 7.4 Hz, Ar**H**), 6.77 (s, 1H, Ar**H**), 6.71 (d, 1H, *J* = 7.9 Hz, Ar**H**), 3.63 (bs, 1H, N**H**CH), 3.26 (q, 1H, *J* = 6.0 Hz, -C**H**CH_3), 1.10 (d, 3H, *J* = 6.3 Hz, -CHC$\mathbf{H}_3$), 0.97 (s, 9H, *J* = 6.6 Hz, -C(C$\mathbf{H}_3)_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.6, 131.6 (Cq, *J* = 31.7Hz), 129.7, 122.7, 115.8, 112.9 (Cq, *J* = 4.0Hz), 109.0 (Cq, *J* = 4.0Hz), 57.1, 34.9, 26.5, 15.8; IR(neat) 3430, 2966, 1614, 1519, 1162, 855 cm^{-1}; EIMS (70 eV) *m/z* 245 (M^+); HRMS-EI *m/*z calcd for $C_{13}H_{18}NF_3$ (M^+) 245.1391, found 245.1397; $[\alpha]^{20}_D$ -24.72 (c 0.55, $CHCl_3$); 96% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 3.0 min (minor), t_R = 3.4 min (major).



4q

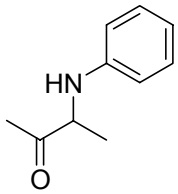
***(-)-N*-(phenyl)-[2-*sec*-butylcyclohexyl]amine (4q) (Table 2, entry 16):** Prepared according to the general procedure from 2-*sec*-butylcyclohexanone (Mixture of diastereomers with a slight 10% of diastereomeric excess, 170 μL, 1.0 mmol), aniline (130 μL, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as colorless oil (80%); First diastereomer: ^1H NMR (500 MHz, $CDCl_3$) δ 7.14 (td, 2H, *J* = 2.0, 7.5 Hz, Ar**H**), 6.62 (td, 1H, *J* = 2.0, 7.5 Hz, Ar**H**), 6.58 (d, 2H, *J* = 2.0, 7.5 Hz, Ar**H**), 3.77 (m, 1H, -C**H**NH), 3.71 (bs, 1H, -CHN**H**), 2.04 – 2.03 (m, 2H, cyclohexyl), 1.83 – 1.74 (m, 2H, cyclohexyl), 1.55 – 1.08 (m, 7H, cyclohexyl and butyl), 0.87 (d, 3H, *J* = 6.5 Hz, -CHC$\mathbf{H}_3$), 0.79 (t, 3H, *J* = 6.5 Hz, -CH_2C$\mathbf{H}_3$). ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.7, 129.3, 116.3, 112.8, 48.5, 45.1, 35.4, 29.5, 26.4, 25.9, 25.0, 20.3, 16.6, 10.6; Second diastereomer: ^1H NMR (500 MHz, $CDCl_3$) δ 7.14 (td, 2H, *J* = 2.0, 7.5 Hz, Ar**H**), 6.62 (td, 1H, *J* = 2.0, 7.5 Hz, Ar**H**), 6.58 (d, 2H, *J* = 2.0, 7.5 Hz, Ar**H**), 3.77 (m, 1H, -C**H**NH), 3.71 (bs, 1H, -CHN**H**), 2.02 – 2.00 (m, 2H, cyclohexyl), 1.83 – 1.74 (m, 2H, cyclohexyl), 1.55 – 1.08 (m, 7H, cyclohexyl and butyl), 0.85 (d, 3H, *J* = 6.5 Hz, -CHC$\mathbf{H}_3$), 0.82 (t, 3H, *J* = 6.5 Hz, -CH_2C$\mathbf{H}_3$). ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.7, 129.3, 116.3, 112.8, 48.3, 44.8, 35.8, 29.5, 26.3, 26.2, 24.9, 20.3, 16.1, 10.5; IR(neat) 3429, 3051, 2926, 1601, 1154, 860 cm^{-1}; EIMS (70 eV) *m/z* 231 (M^+); HRMS-EI *m/*z calcd for $C_{16}H_{25}N$ (M^+) 231.1987, found 231.1991; $[\alpha]^{20}_D$ -36.2 (c 0.16, $CHCl_3$); 53 and 66% de by GC-MS (EI) [column: Ciclodex-β, flow rate = 1.2 grade/min, t_R = 55.7 min (minor), t_R = 56.1 min (major) and t_R = 57.5 min (minor), t_R = 57.8 min (major) respectively].

Notes:

1. The injection of 2-*sec*-butylcyclohexanone by GC-MS employing a non chiral column was detected two peaks with t_R = 26.7 min (major) and t_R = 26.8 min (minor) with a slight diastereomeric excess of 10%. When this substrate was aminated with aniline using [(*rac*)-BINAP]$PdBr_2$ (**1a**), two pairs of diastereomers of the desired product (**4n**) were detected by

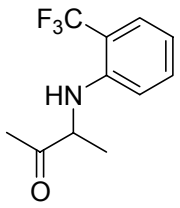
GC-MS (EI) [column: Ciclodex-β, flow rate = 1.2 grade/min, t_R = 55.7 min (major), t_R = 56.0 min (major) and t_R = 57.5 min (minor), t_R = 57.7 min (minor) respectively] with the same intensity. See S80.

2. When [(*S*)-BINAP]$PdBr_2$ (**1c**) complex was used: $[\alpha]^{20}_D$ +33.0 (c 0.16, $CHCl_3$); 57 and 69% of diastereomeric excess was detected by GC-MS (EI) [column: Ciclodex-β, flow rate = 1.2 grade/min, t_R = 55.7 min (minor), t_R = 56.0 min (major) and t_R = 57.6 min (minor), t_R = 57.8 min (major) respectively]. These values are opposite to [(*R*)-BINAP]$PdBr_2$ (**1b**) was used. See S82.



4r

***(-)-N*-(phenyl)-[1-(methyl)-2-(one)-propyl]amine (4r) (Table 2, entry 17):** Prepared according to the general procedure from 2,3-butanedione (90 μL, 1.0 mmol), aniline (130 μL, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1c**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (85%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.18 (td, 2H, *J* = 7.4, 1.6 Hz, Ar**H**), 6.72 (t, 1H, *J* = 7.4 Hz, Ar**H**), 6.56 (d, 2H, *J* = 7.4 Hz, ArH), 4.39 (bs, 1H, -N**H**CH), 4.06 (q, 1H, *J* = 7.3 Hz, -C**H**CH_3), 2.21 (s, 3H, -COC$\mathbf{H}_3$), 1.41 (d, 3H, *J* = 6.8 Hz, -CHC$\mathbf{H}_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 210.4 (-CO), 146.5, 129.5, 118.0, 113.0, 58.6, 25.8, 18.0; IR(neat) 3391, 2977, 2930, 1712(CO), 1602, 1505, 1177, 872 cm^{-1}; EIMS (70 eV) *m/z* 163 (M^+); $[\alpha]^{20}_D$ -1.62 (c 0.43, $CHCl_3$); 20% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 90/10, flow rate = 1 mL/min, t_R = 8.6 min (major), t_R = 10.8 min (minor).

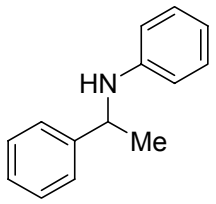


4s

***(-)-N*-(2-trifluoromethylphenyl)-[1-(methyl)-2-(one)-propyl]amine (4s) (Table 2, entry 18):** Prepared according to the general procedure from 2,3-butanedione (90 μL, 1.0 mmol), *o*-trifluoromethyl aniline (180 μL, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as colorless oil (83%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.46 (d, 1H, *J* = 7.4 Hz, Ar**H**), 7.33 (t, 1H, *J* = 7.7 Hz, Ar**H**), 6.73 (t, 1H, *J* = 7.7 Hz, Ar**H**), 6.54 (d, 1H, *J* = 8.2 Hz, Ar**H**), 5.12 (bs, 1H, -N**H**CH), 4.10 (q, 1H, *J* = 6.7 Hz, -C**H**CH_3), 2.19 (s, 3H, -CH_3), 1.44 (d, 3H, *J* = 7.1 Hz, -CHC**H**$_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.1 (-CO), 143.9, 133.3, 127.0 (Cq, *J* = 5.6 Hz), 123.3, 119.9, 116.7, 114.0 (Cq, *J* = 29.4 Hz), 58.2, 20.3, 17.7; IR(neat) 3423, 2985, 2929, 1721(CO), 1614, 1520, 1145, 752 cm^{-1}; EIMS (70 eV) *m/z* 231 (M^+); $[\alpha]^{20}_D$ -1.0 (c 0.4, $CHCl_3$); 2% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 99/1, flow rate = 1 mL/min, t_R = 6.4 min (minor), t_R = 6.8 min (major).

General procedure for asymmetric reductive amination of aryl ketones: 1.0 mmol of the acetophenone derivative, 1.5 mmol of aniline derivative were added to a stirred solution of 0.025 mmol of chiral palladium complex in 10 mL of dry $CHCl_3$ (in a Schlenk tube) and stirred for 10 minutes. The solution was transferred to a 45 ml stainless steel autoclave (PARR) that contained 150 mg of molecular sieves 5Å previously purged with vacum-N_2. Subsequently, the reaction was taken to the desired pressure (800 psi H_2), stirred in an oil bath at 70^oC for 24 h. At the end of this period, the gas was liberated. The solution was analyzed by GC-MS to quantify the remaining substrate, and was later concentrated under reduced pressure, affording a crude residue, which was purified by column chromatography over silica gel (70-230 mesh), and eluted with hexane-ethyl acetate (99/1) to isolate the product.

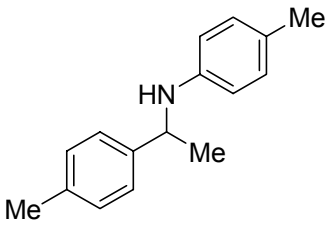
Absolute configurations of known compounds were assigned by comparison of optical rotations to literature values.



6a

***(R)-(-)-N*-[1-(phenyl)-ethyl]aniline (6a) (Table 3, entry 1):** Prepared according to the general procedure from acetophenone (110 μL, 1 mmol), aniline (130 μL, 1.5 mmol) and [(*S*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (64%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.40 – 7.21 (m, 5H, Ar**H**), 7.11 (dd, 2H, *J* = 8.7 Hz, Ar**H**), 6.66 (t, 1H, *J* = 7.8 Hz, Ar**H**), 6.53 (d, 2H, *J* = 7.8 Hz, Ar**H**), 4.51 (q, 1H, *J* = 6.6 Hz, -C**H**CH_3), 4.04 (bs, 1H, -N**H**CH), 1.54 (d, 3H, *J* = 7.0 Hz, -CHC**H**$_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.2, 145.1, 129.0, 128.6, 126.8, 125.8, 117.2, 113.2, 53.4, 24.9; EIMS (70 eV) *m/z* 197 (M^+); $[\alpha]^{20}_D$ -3.6 (c 0.5, $CHCl_3$); 43% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 5.9 min (minor), t_R = 6.9 min (major).

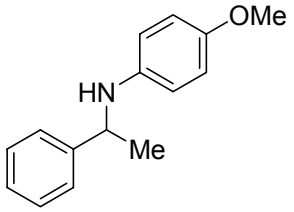
The absolute configuration was determined by comparison with the reported literature as (*R*) with $[\alpha]^{20}_D$ -3.9 (c 1.0, $CHCl_3$) and 81% ee.[3]



6b

***(+)-N*-(4-tolyl)-[1-(4-methylphenyl)-ethyl]amine (6b) (Table 3, entry 2):** Prepared according to the general procedure from *p*-methyl acetophenone (130 μL, 1 mmol), *p*-toluidine (160.5 mg, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1c**, (22 mg, 0.025mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (67%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.28 (d, 2H, *J* = 8.1 Hz, ArH), 7.15 (d, 2H, *J* = 7.8 Hz, ArH), 6.93 (d, 2H, *J* = 8.1 Hz, ArH), 6.43 (d, 2H, *J* = 8.4 Hz, ArH), 4.46 (q, 1H, *J* = 6.6 Hz, -C**H**CH_3), 3.89 (bs, 1H, N**H**CH), 2.34 (s, 3H, -CH_3), 2.21 (s, 3H, -CH_3), 1.51 (d, 3H, *J* = 6.7 Hz, -CHC**H**$_3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.0, 142.3, 136.2, 129.5, 129.2, 126.2, 125.7, 113.4, 53.3, 24.9, 21.0,

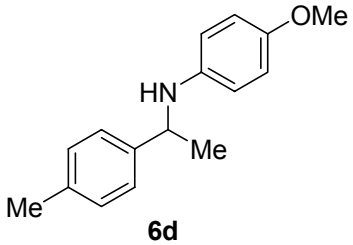
20.3; IR(neat) 3409, 2966, 2921, 1618, 1519, 1140, 808 cm^{-1}; EIMS (70 eV) *m/z* 225 (M^+); Anal. Calcd for $C_{16}H_{19}N$ (225.1517): C, 85.28; H, 8.50; N, 6.22. Found: C, 85.27; H, 8.46; N, 6.25; HRMS-EI *m/z* calcd for $C_{16}H_{19}N$ (M^+) 225.1517, found 225.1515; $[\alpha]^{20}_D$ +10.18 (c 0.54, $CHCl_3$); 35% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 5.2 min (major), t_R = 6.0 min (minor).



6c

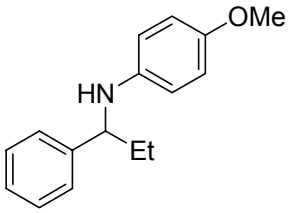
***(R)-(+)-N*-(4-methoxyphenyl)-[1-(phenyl)-ethyl]amine (6c) (Table 3, entry 3):** Prepared according to the general procedure from acetophenone (110 μL, 1.0 mmol), *p*-anisidine (184.5 mg, 1.5 mmol) and [(*R*)-Tol-BINAP]$PdBr_2$, **1d**, (23.6 mg, 0.025mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (65%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 4H, Ar**H**), 7.25 (d, 2H, *J* = 8.0 Hz, Ar**H**), 6.71 (d, 2H, *J* = 8.8 Hz, Ar**H**), 6.49 (d, 2H, *J* = 8.8 Hz, Ar**H**), 4.43 (q, 1H, *J* = 6.6 Hz, -C**H**CH_3), 3.49 (bs, 1H, -N**H**CH), 3.70 (s, 3H, -OC$\mathbf{H_3}$), 1.51 (d, 3H, *J* = 6.6 Hz, -CHC$\mathbf{H_3}$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.9, 145.6, 141.6, 128.7, 126.9, 126.0, 114.8, 114.6, 55.8, 54.3, 25.2; EIMS (70 eV) *m/z* 227 (M^+); Anal. Calcd for $C_{15}H_{17}NO$ (227.1310): C, 79.26; H, 7.54; N, 6.16. Found: C, 79.25; H, 7.56; N, 6.20; HRMS-EI *m/z* calcd for $C_{15}H_{17}NO$ (M^+) 227.1310, found 227.1314; $[\alpha]^{20}$ = +5.6 (c 0.4, $CHCl_3$); 35% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 6.9 min (major), t_R = 7.6 min (minor).

The absolute configuration was determined by comparison with the reported literature as (*R*) with $[\alpha]^{20}_D$ +6.0 (c 0.3, $CHCl_3$) and 21% ee.[5]



6d

***(+)-N*-(4-methoxyphenyl)-[1-(4-tolyl)-ethyl]amine (6d) (Table 3, entry 4):** Prepared according to the general procedure from *p*-methyl acetophenone (130 μL, 1 mmol), *p*-anisidine (184.5 mg, 1.5 mmol) and [(*R*)-BINAP]$PdBr_2$, **1b**, (22 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (53%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.25 (d, 2H, *J* = 7.7 Hz, Ar**H**), 7.12 (d, 2H, *J* = 8.2 Hz, Ar**H**), 6.69 (d, 2H, *J* = 8.8 Hz, Ar**H**), 6.47 (d, 2H, *J* = 8.8 Hz, Ar**H**), 4.39 (q, 1H, *J* = 6.6 Hz, -C**H**CH_3), 3.71 (bs, 1H, N**H**CH), 3.69 (s, 3H, -OC$\mathbf{H_3}$), 2.32 (s, 3H, -C$\mathbf{H_3}$), 1.48 (d, 3H, *J* = 6.6 Hz, -CHC$\mathbf{H_3}$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.9, 142.5, 141.7, 136.4, 129.3, 125.8, 114.8, 114.6, 55.8, 25.2, 21.1; EIMS (70 eV) *m/z* 241 (M^+); $[\alpha]^{20}_D$ +6.73 (c 0.22, $CHCl_3$); 38% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 23.3 min (major), t_R = 25.2 min (minor).



6e

***(+)-N*-(4-methoxyphenyl)-[1-(phenyl)-propyl]amine (6e) (Table 3, entry 5):** Prepared according to the general procedure from propiophenone (130 μL, 1.0 mmol), *p*-anisidine (184.5 mg, 1.5 mmol) and [(*R*)-Tol-BINAP]$PdBr_2$, **1d**, (23.6 mg, 0.025 mmol) at 70 ^oC for 24 h, to provide the title compound as yellow oil (57%); ^1H NMR (300 MHz, $CDCl_3$) δ 7.33 – 7.21 (m, 5H, Ar**H**), 6.68 (d, 2H, *J* = 8.8 Hz, Ar**H**), 6.47 (d, 2H, *J* = 8.8 Hz, Ar**H**), 4.15 (t, 1H, *J* = 6.6 Hz, -C**H**CH_3), 3.82 (bs, 1H, N**H**CH), 3.69 (s, 3H, -OC$\mathbf{H_3}$), 1.81 (sext, 2H, *J* = 7.4 Hz, -C$\mathbf{H_2}CH_3$), 0.94 (t, 3H, *J* = 7.4 Hz, -CH_2C$\mathbf{H_3}$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.9, 144.2, 141.9, 128.5, 126.9, 126.6, 114.8, 114.5, 60.6, 55.8, 31.8, 10.9; IR(neat) 3402, 2963, 2932, 1614, 1513, 1178, 819 cm^{-1}; EIMS (70 eV) *m/z* 241 (M^+); $[\alpha]^{20}_D$ +9.44

(c 0.54, $CHCl_3$); 34% ee by HPLC (column: Daicel Chiracel OD-H; eluent hexane/^{i}PrOH = 92/8, flow rate = 1 mL/min, t_R = 5.6 min (major), t_R = 6.0 min (minor).

References

1. Andrews, M. A.; Chang, T. C. T.; Cheng, C. W. F.; Emge. T. J.; Kelly, K. P.; Koetzle, T. F. *J. Am. Chem. Soc*. **1984,** *106*, 5913.

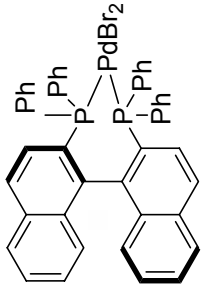
2. Ozawa, F. Kubo, A.; Matsumoto, Y. Hayashi, T. *Organometallics* **1993,** *12*, 4188.

3. Cheemala, M. N.; Knochel, P. *Org. Lett*. **2007,** *9*, 3089.

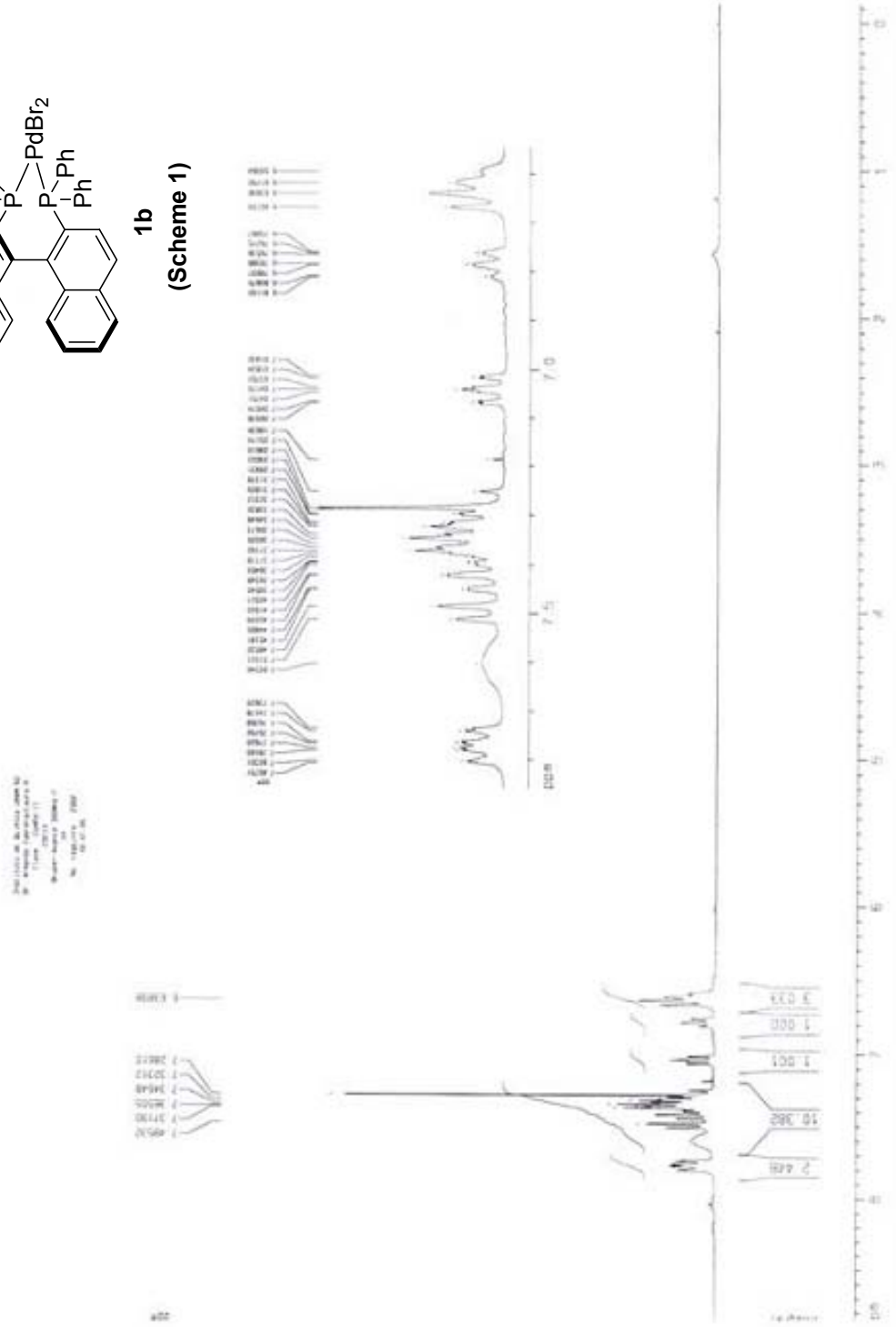
4. (a) Pallavicini, M.; Bolchi, C.; Fumagalli, L.; Valoti, E., Villa, L. *Tetrahedron: Asymmetry* **2002,** *13*, 2277. (b) Grishina, G.V.; Luk´yanenko, E. R.; Borisenko, A. A. Russ. *J. Org. Chem*. **2005,** *41*, 807. (c) Andrés, C.; Nieto, J.; Pedrosa, R.; Villamañán, N. *J. Org. Chem*. **1996**, *61*, 4130.

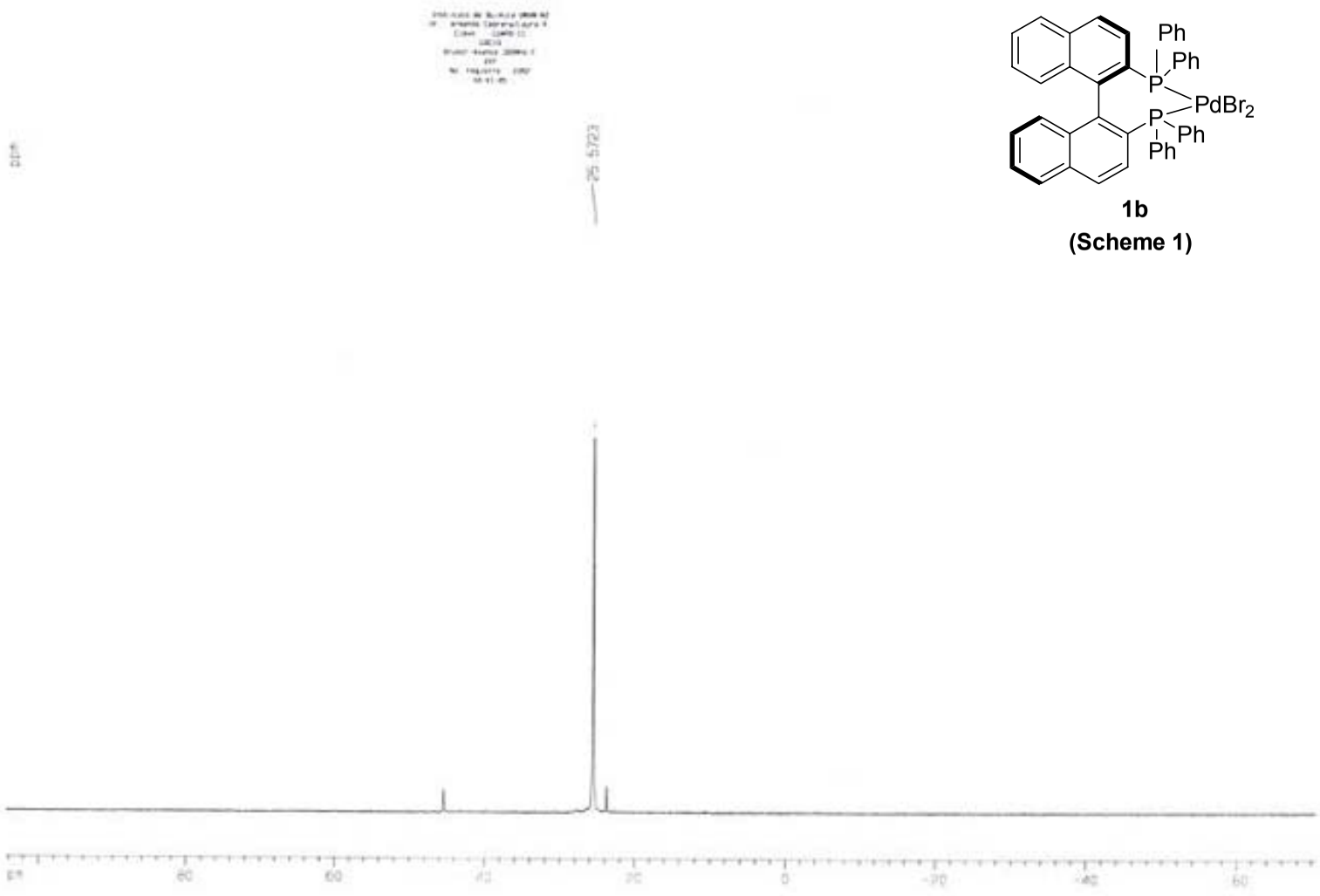
5. Tagashira, J.; Imao, D.; Yamamoto, T.; Ohta, T.; Furukawa, I.; Ito, Y. *Tetrahedron: Asymmetry*, **2005,** *16*, 2307.

Copies of NMR, GC-MS (EI) or HPLC for all compounds.

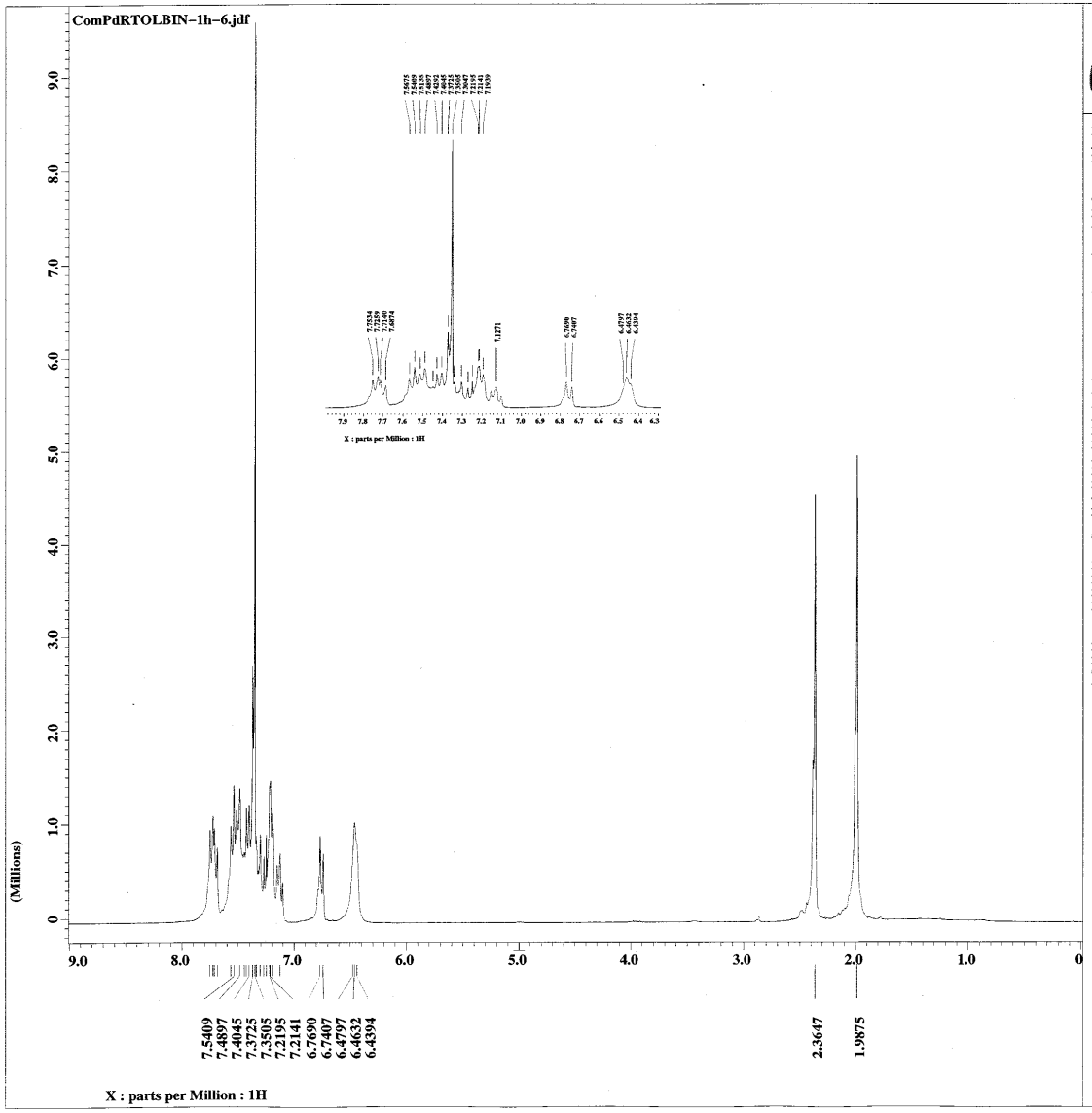


1b
(Scheme 1)





1b
(Scheme 1)



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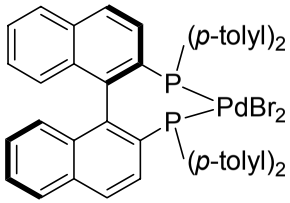
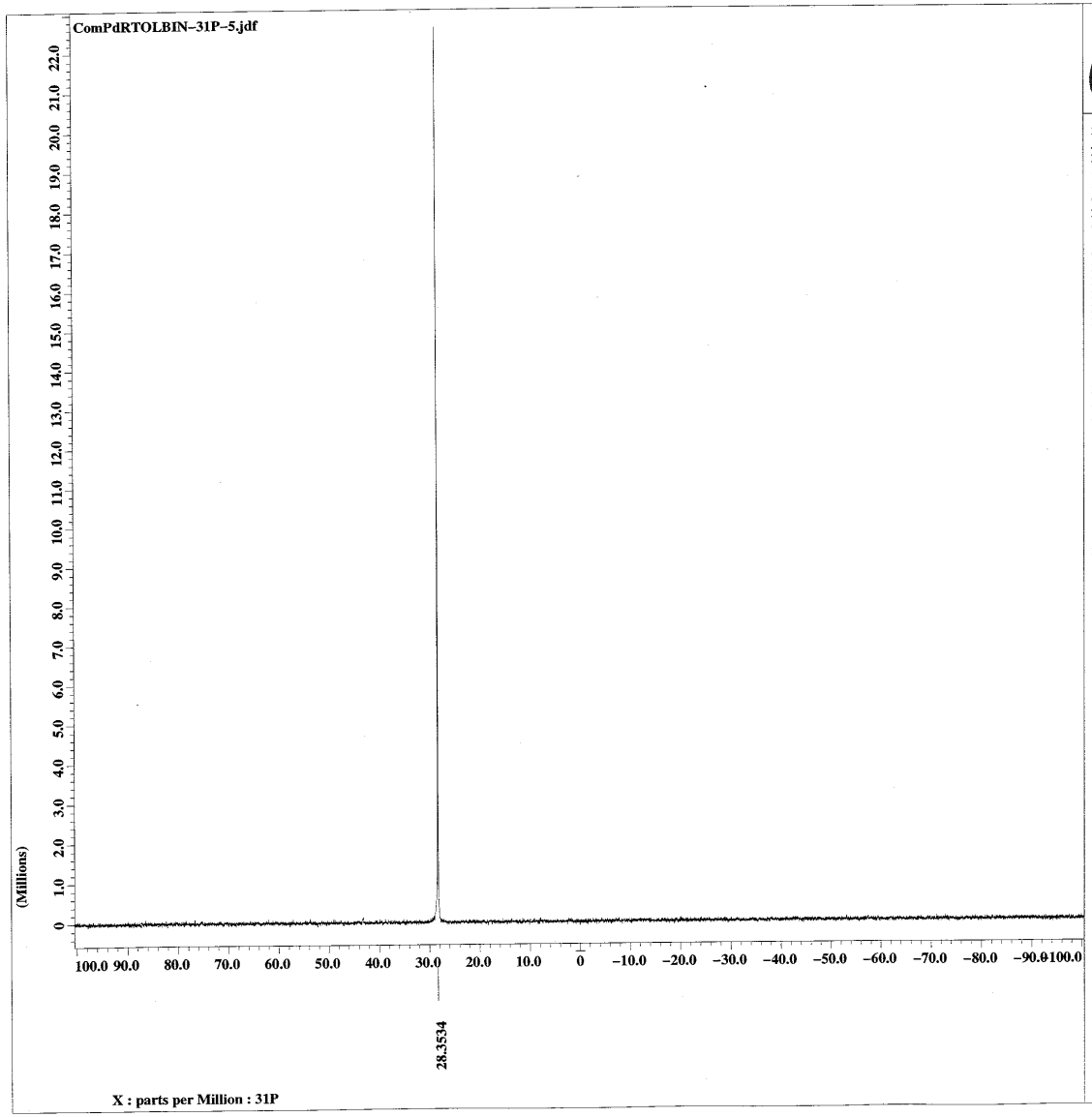


1d

(Scheme 1)



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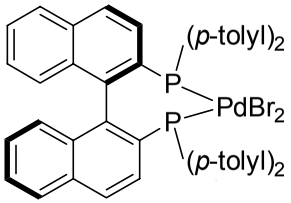
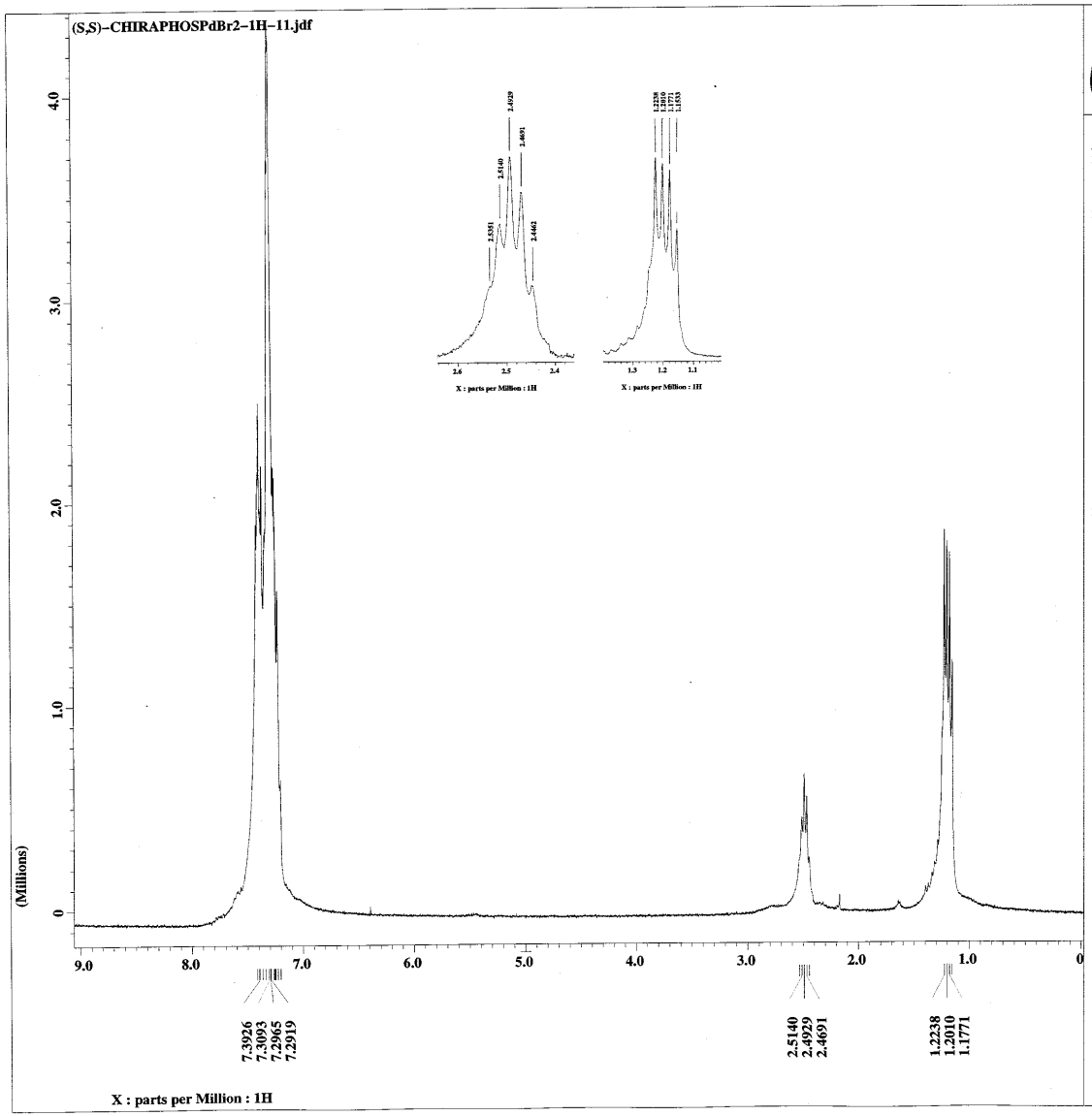


1d
(Scheme 1)



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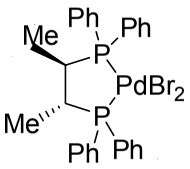
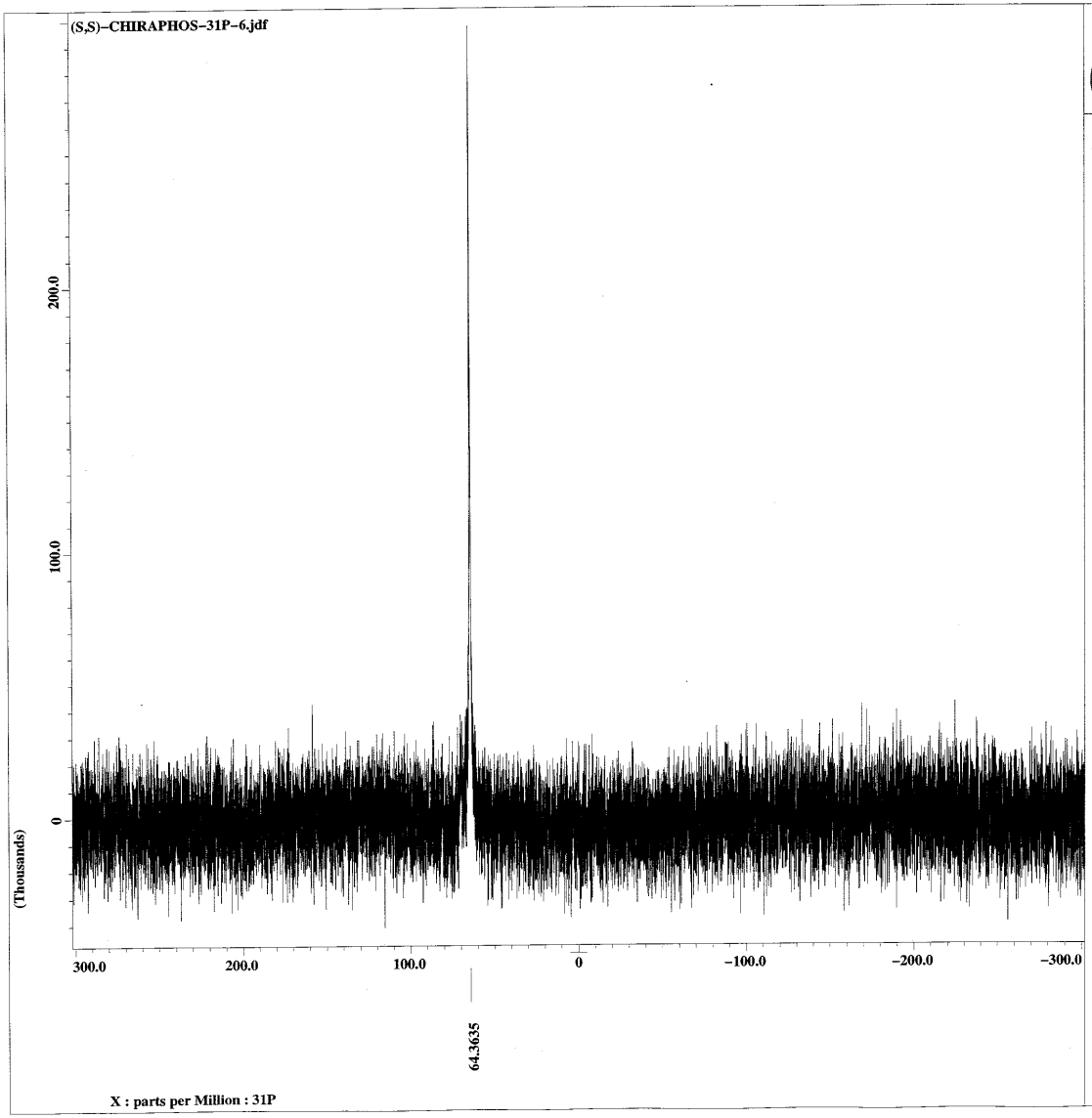


1e
(Scheme 1)



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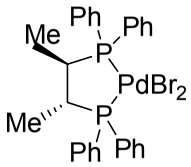
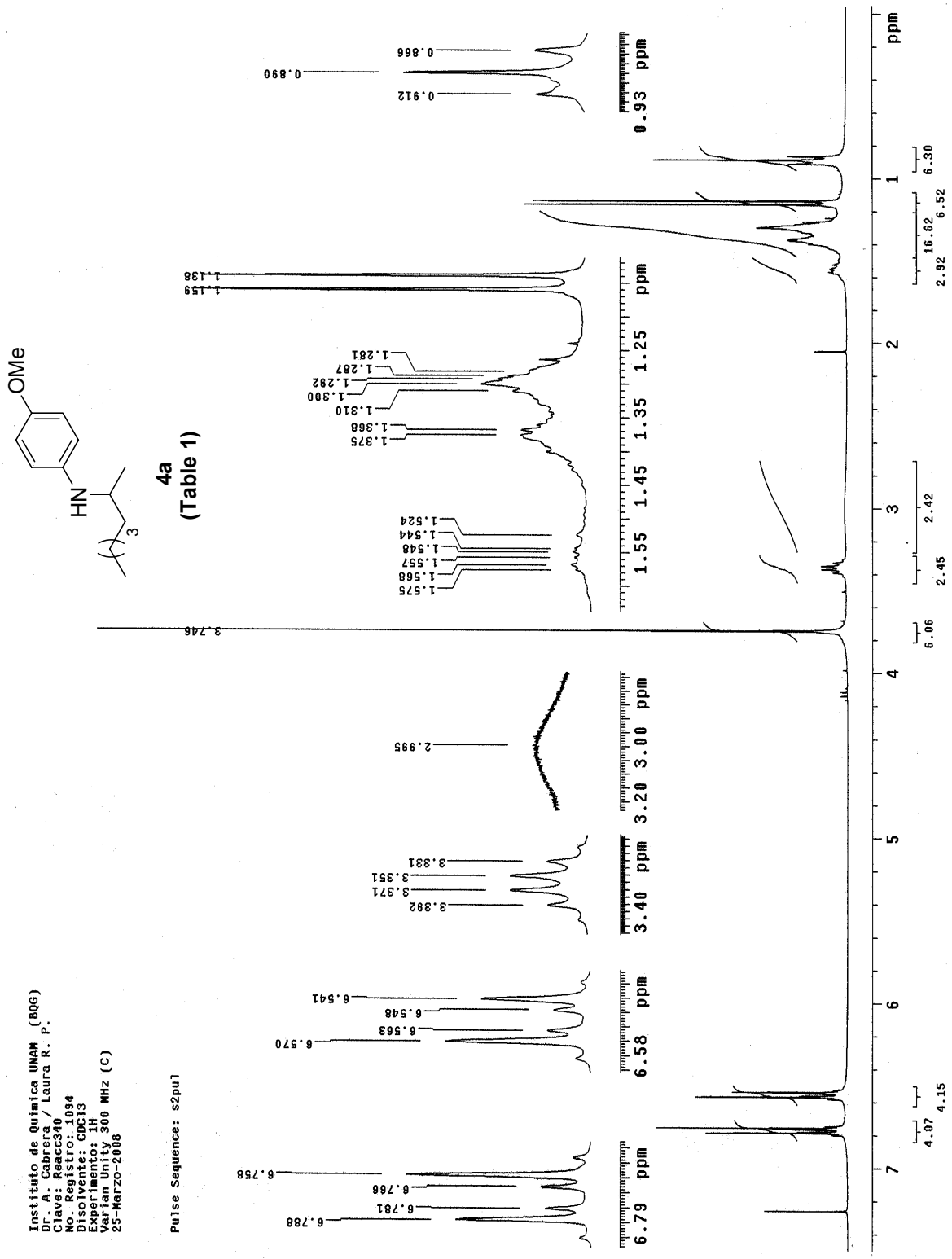
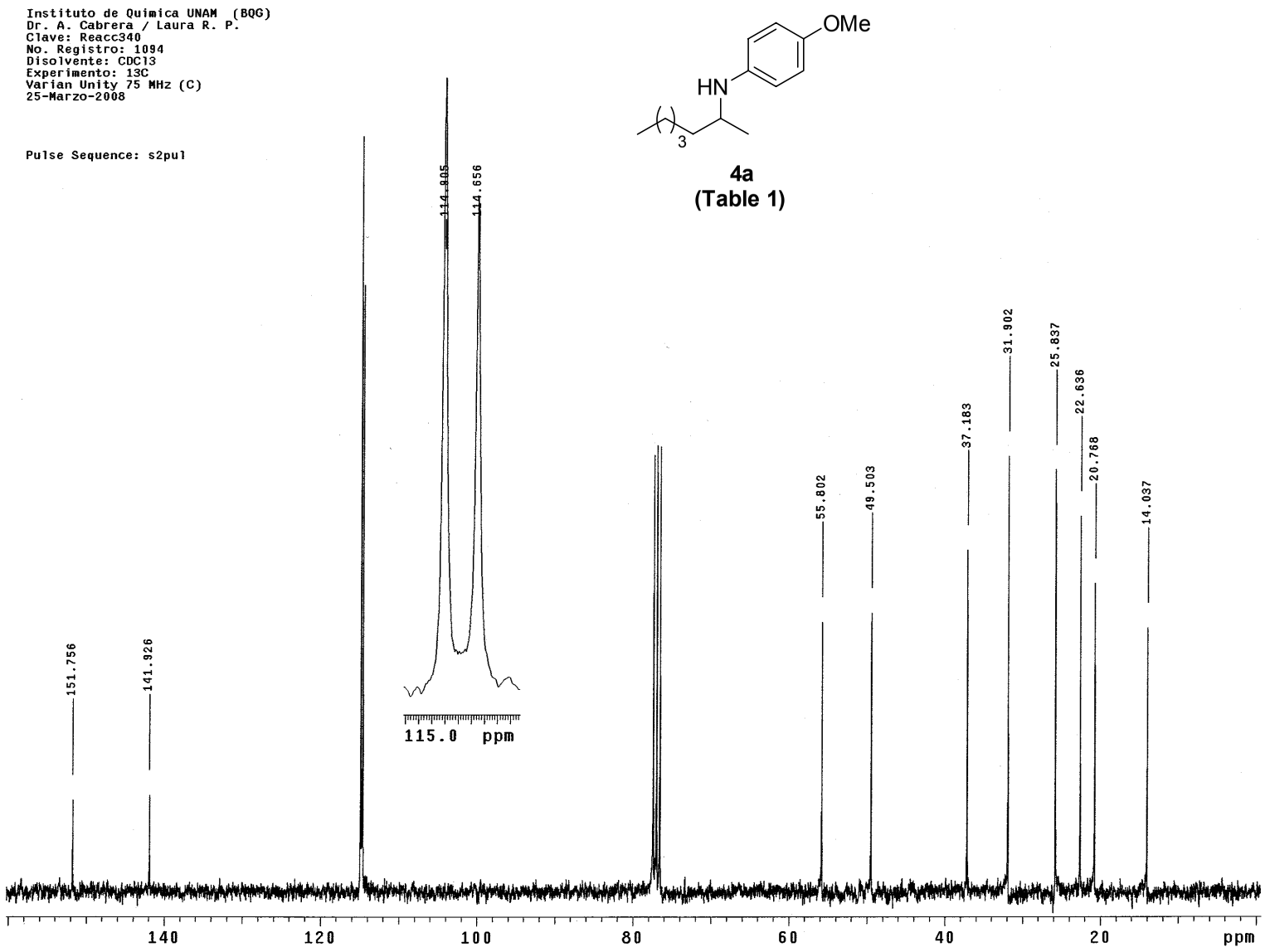
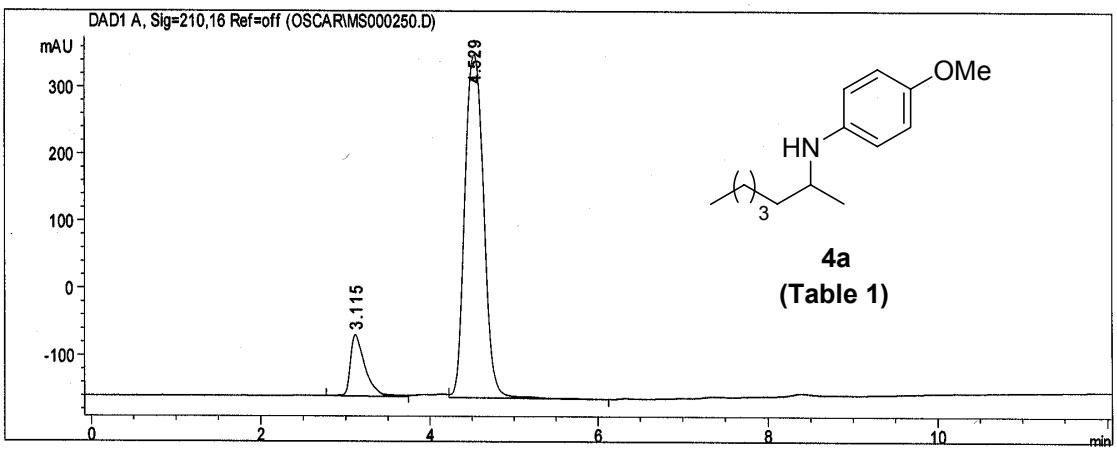


1e
(Scheme 1)


Instituto de Quimica UNAM (BQG)
Dr. A. Cabrera / Laura R. P.
Clave: Reacc340
No. Registro: 1094
Disolvente: CDCl3
Experimento: 1H
Varian Unity 300 MHz (C)
25-Marzo-2008
Pulse Sequence: s2pul
OMe
HN
3
4a
(Table 1)
6.788
6.781
6.766
6.758
6.570
6.563
6.548
6.541
3.392
3.371
3.351
3.331
2.995
3.246
1.575
1.568
1.557
1.548
1.544
1.524
1.375
1.368
1.310
1.300
1.292
1.287
1.281
1.159
1.138
0.890
0.866
0.912
6.79 ppm
6.58 ppm
3.40 ppm
3.20 3.00 ppm
1.55 1.45 1.35 1.25 ppm
0.93 ppm
ppm
4.07
4.15
6.06
2.45
2.42
2.92
16.62
6.52
6.30


Instituto de Quimica UNAM (BQG)
Dr. A. Cabrera / Laura R. P.
Clave: Reacc340
No. Registro: 1094
Disolvente: CDCl3
Experimento: 13C
Varian Unity 75 MHz (C)
25-Marzo-2008
Pulse Sequence: s2pul
OMe
HN
3
4a
(Table 1)
151.756
141.926
114.805
114.656
115.0 ppm
55.802
49.503
37.183
31.902
25.837
22.636
20.768
14.037
140
120
100
80
60
40
20
ppm

Reacc340
080616-coa-01



Data File C:\HPCHEM\1\DATA\OSCAR\MS000250.D Sample Name: Reacc340
HPLC IQ 19/06/08 10:23:33 AM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 95/5
flujo 1 ml/min
UV 210

==========

Injection Date : 18/06/08 3:48:25 PM
Sample Name : Reacc340 Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 18/06/08 3:38:29 PM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 19/06/08 10:08:54 AM by carmen
(modified after loading)

==========

Area Percent Report

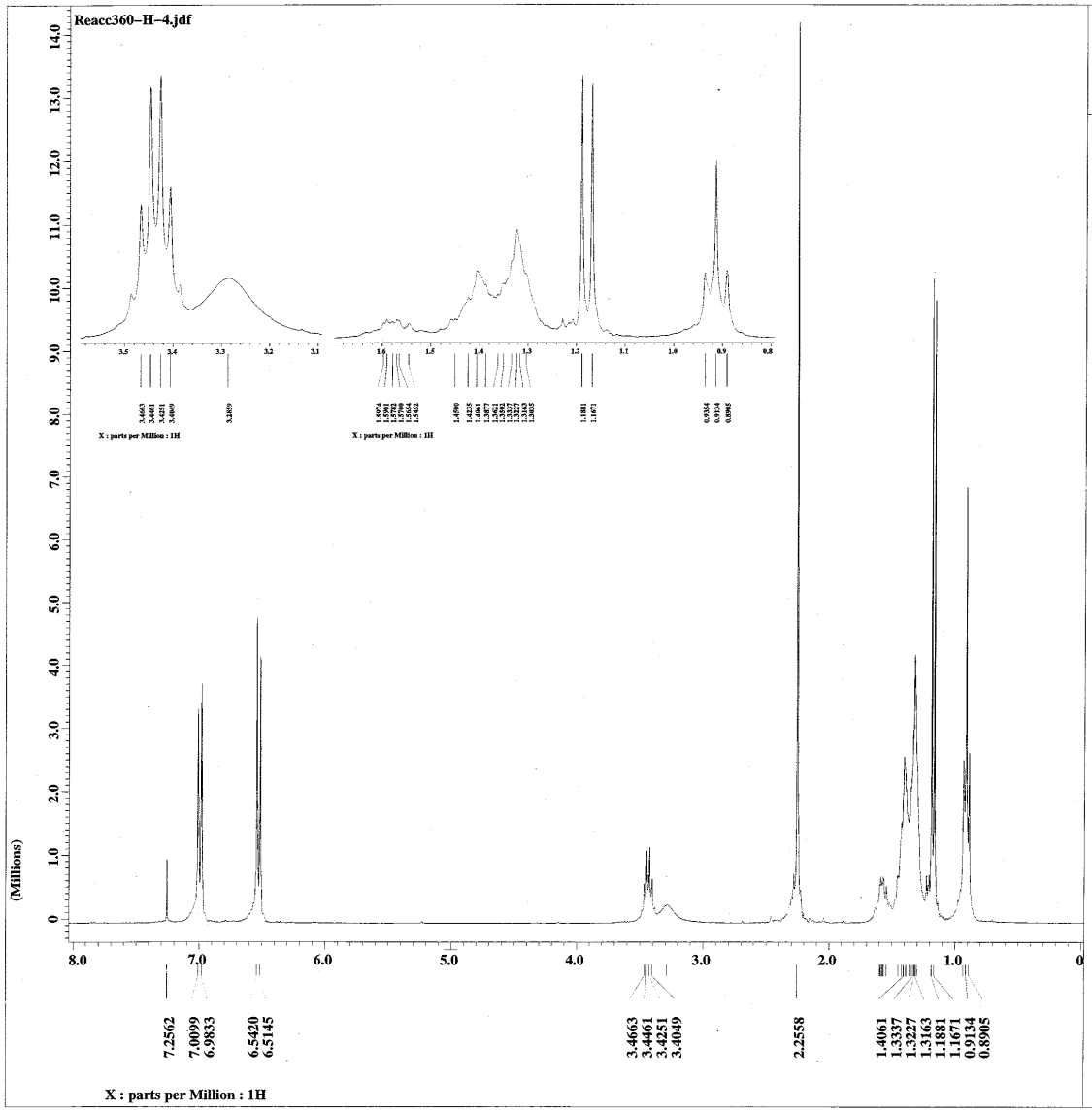
==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

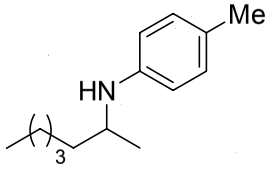
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.115	BV	0.1834	1095.69629	91.72173	12.0154
2	4.529	VP	0.2551	8023.39697	511.09534	87.9846
Totals :				9119.09326	602.81706	

% ee = 75.96

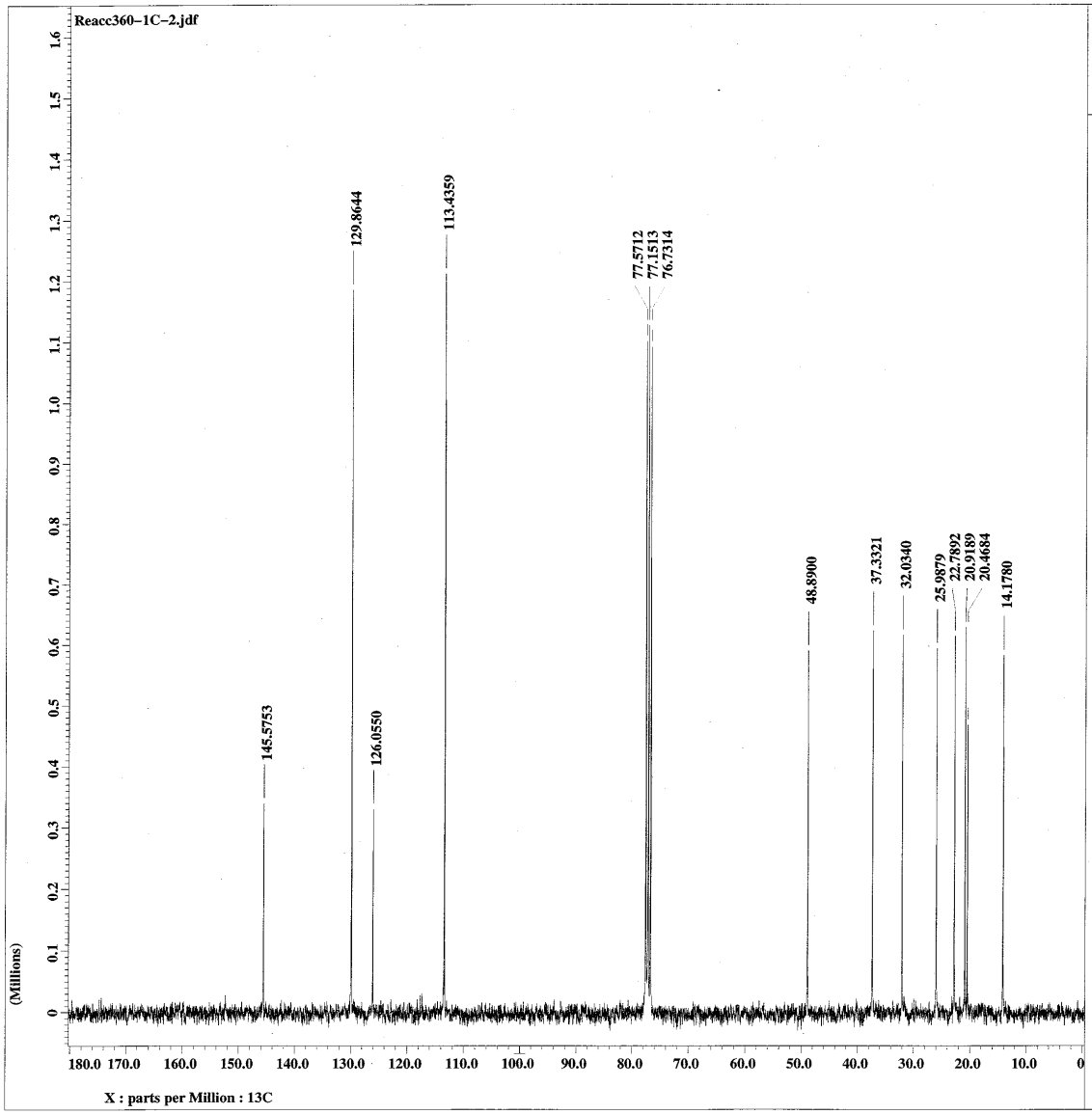


JEOL

Filename	= Reacc360-H-4.jdf
Author	= Cabrera
Experiment	= single_pulse.exp
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 22-JAN-2008 00:19:58
Revision_time	= 3-APR-2008 17:08:43
Current_time	= 3-APR-2008 17:10:06
Comment	= Single Pulse Experime
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 32
Total_scans	= 32
X_90_width	= 9.5[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 4.75[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 16
Relaxation_delay	= 4[s]
Temp_get	= 20.3[dC]
Unblank_time	= 2[us]

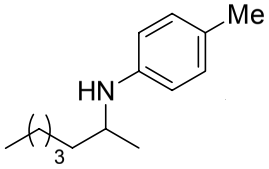


4b
(Table 2, entry 1)

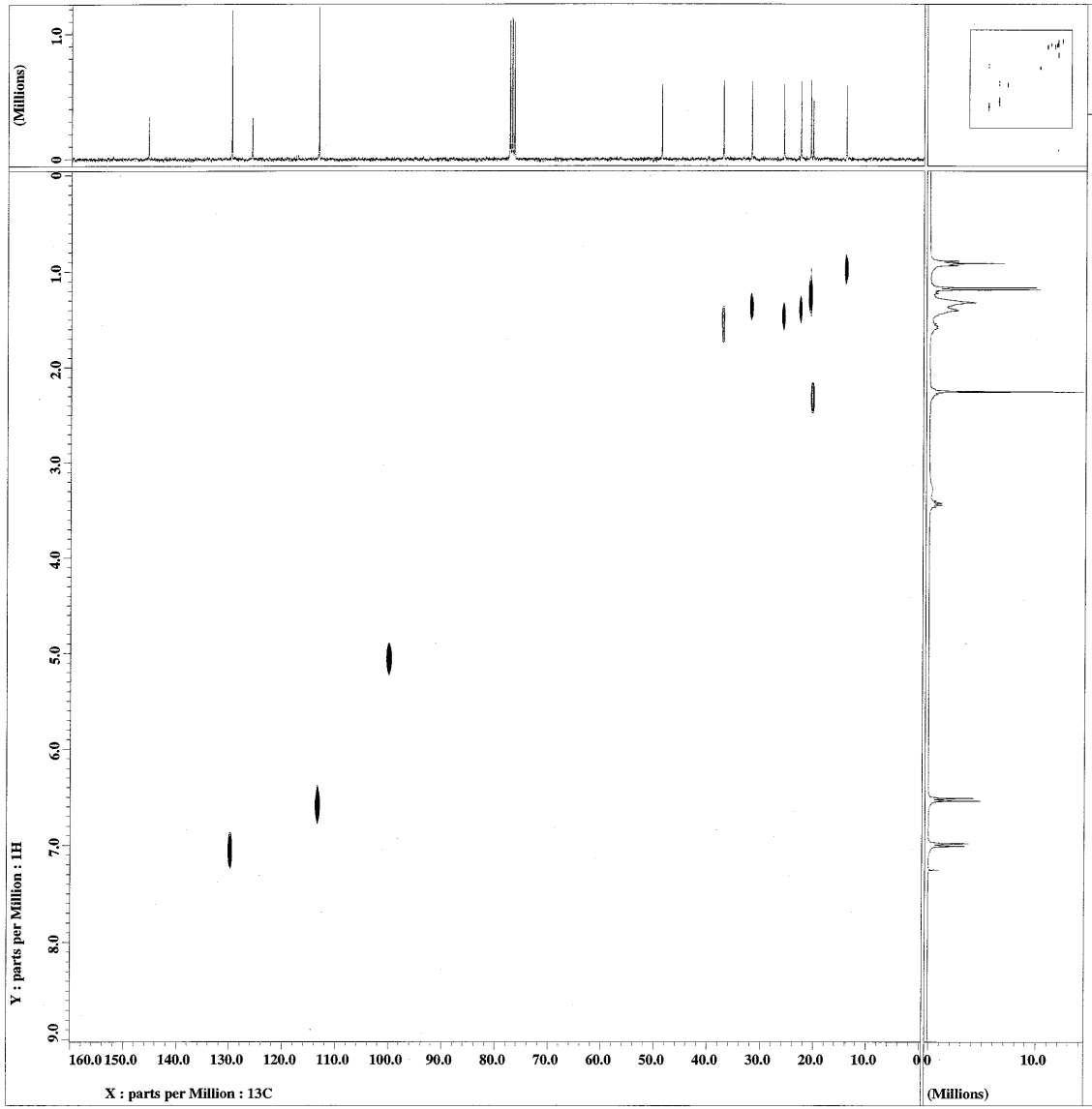


JEOL

```
Filename          = Reacc360-1C-2.jdf
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 22-JAN-2008 00:39:54
Revision_time     =  3-APR-2008 17:23:50
Current_time      =  3-APR-2008 17:24:24

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 411
Total_scans       = 411

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 21.8[dC]
Unblank_time      = 2[us]
```

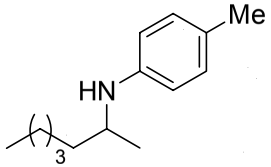


4b
(Table 2, entry 1)



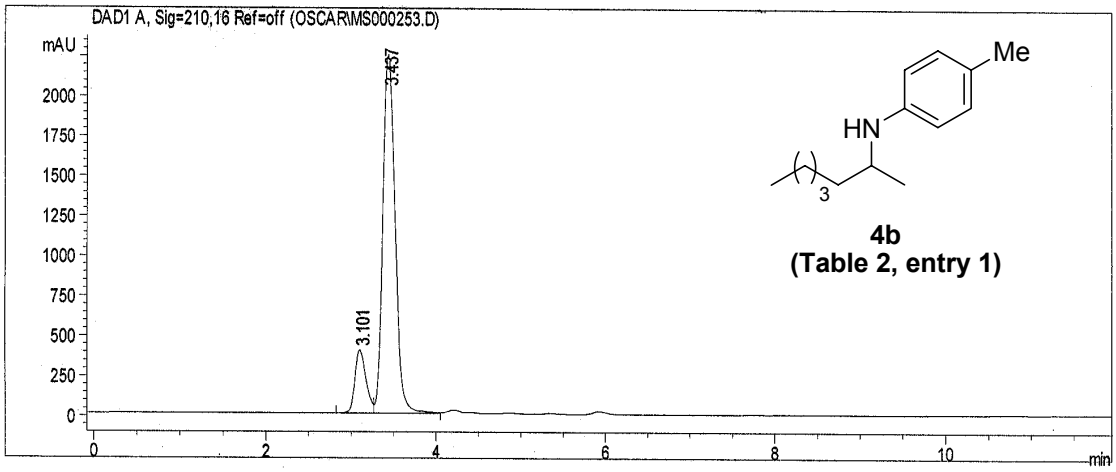
JEOL

Filename	= Reacc360-chshf-2.jdf
Author	= Cabrera
Experiment	= chshf.exp
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 22-JAN-2008 01:12:21
Revision_time	= 3-APR-2008 17:56:21
Current_time	= 3-APR-2008 17:57:52
Comment	= Heteronuclear Shift C
Data_format	= 2D REAL REAL
Dim_size	= 1024, 242
Dim_title	= 13C 1H
Dim_units	= [ppm] [ppm]
Dimensions	= X Y
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 54.1696[ms]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 1024
X_prescans	= 4
X_resolution	= 18.46053875[Hz]
X_sweep	= 18.90359168[kHz]
Y_domain	= 1H
Y_freq	= 300.52965592[MHz]
Y_offset	= 5[ppm]
Y_points	= 121
Y_prescans	= 0
Y_resolution	= 37.26087831[Hz]
Y_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 10
Total_scans	= 1210
X_acq_time	= 54.1696[ms]
X_pulse	= 11.3[us]
Y_acq_time	= 28.3904[ms]
Y_pulse	= 37[us]
Initial_wait	= 1[s]
J_constant	= 140[Hz]
Phase_preset	= 3[us]
Recvr_gain	= 30
Relaxation_delay	= 1.5[s]
T1	= 1[us]
Temp_get	= 20.5[dC]
Unblank_time	= 2[us]



4b
(Table 2, entry 1)

Reacc390
080619-coa-02



Data File C:\HPCHEM\1\DATA\OSCAR\MS000253.D Sample Name: Reacc390
HPLC IQ 20/06/08 12:10:17 PM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

==========

Injection Date : 20/06/08 11:10:51 AM
Sample Name : Reacc390 Vial : 1
Acq. Operator : carmen
Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 20/06/08 9:08:55 AM by carmen
(modified after loading)

==========

Area Percent Report

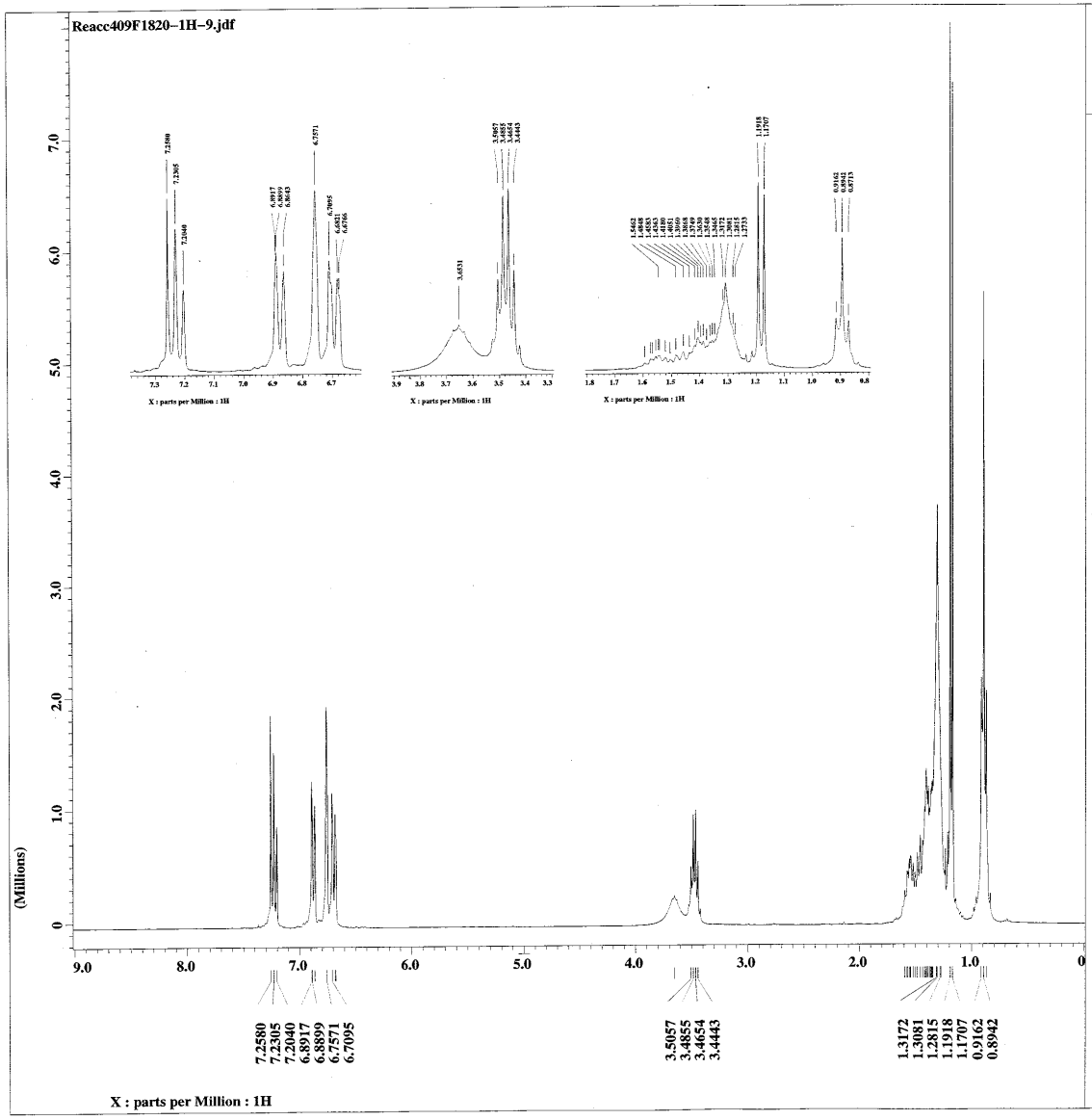
==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

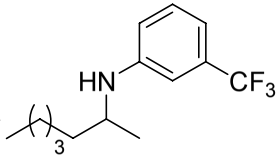
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.101	BV	0.1371	3566.70728	395.91531	13.6440
2	3.437	VV	0.1612	2.25745e4	2246.77417	86.3560
Totals :				2.61412e4	2642.68948	

ee = 72.71%



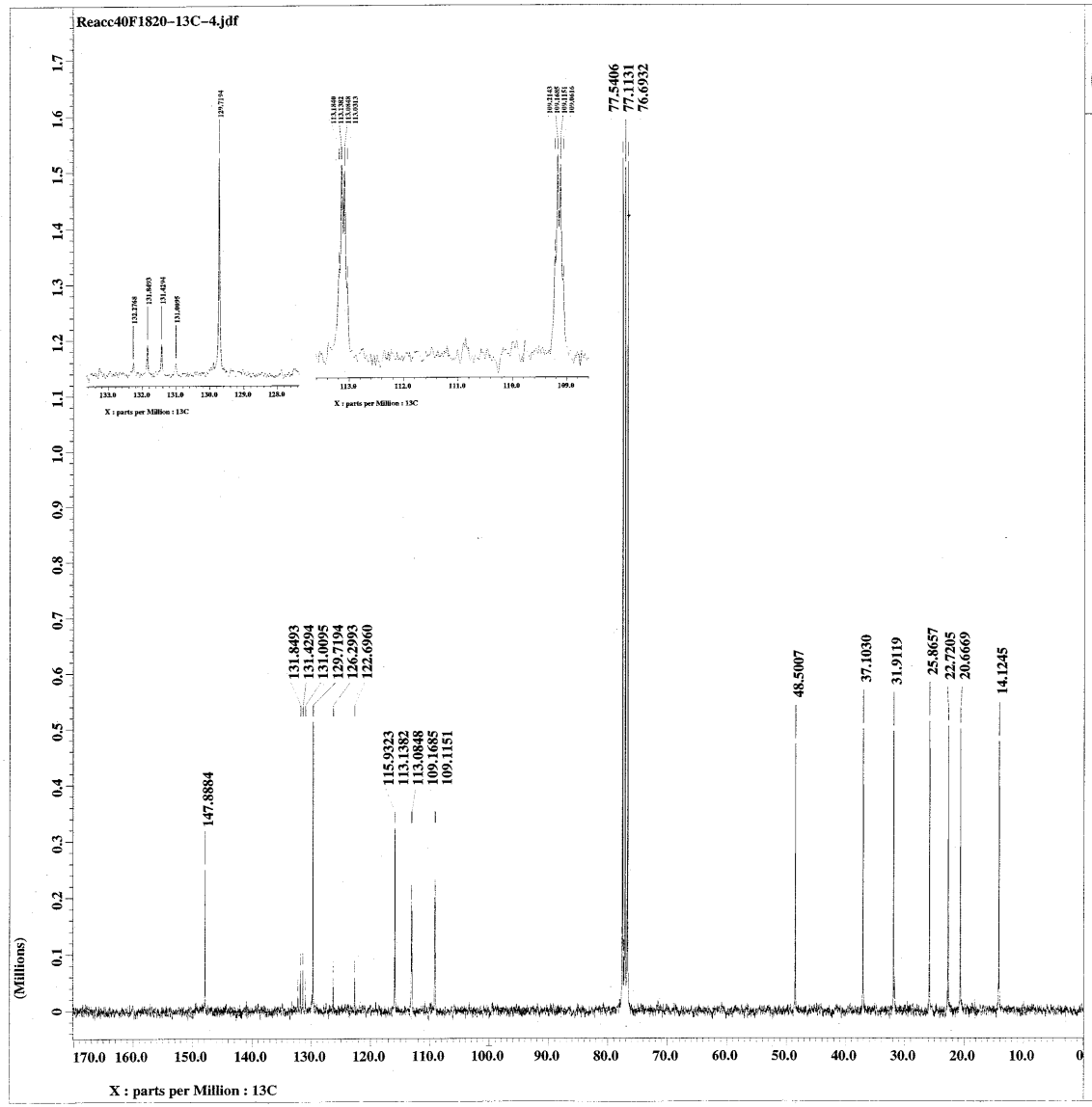
JEOL

```
Filename          = Reacc409F1820-1H-9.jd
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 16-MAR-2008 14:08:53
Revision_time     =  2-SEP-2008 07:20:49
Current_time      =  2-SEP-2008 07:22:11

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 16
Relaxation_delay  = 1[s]
Temp_get          = 16.3[dC]
Unblank_time      = 2[us]
```



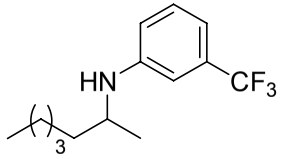
4c

(Table 2, entry 2)



JEOL

Filename	= Reacc40F1820-13C-4.jd
Author	= Cabrera
Experiment	= single_pulse_dec
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 16-MAR-2008 15:21:29
Revision_time	= 28-MAY-2008 09:09:50
Current_time	= 28-MAY-2008 09:10:14
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1265
Total_scans	= 1265
X_90_width	= 11.3[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 3.76666667[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 30
Relaxation_delay	= 1[s]
Temp_get	= 18.2[dC]
Unblank_time	= 2[us]



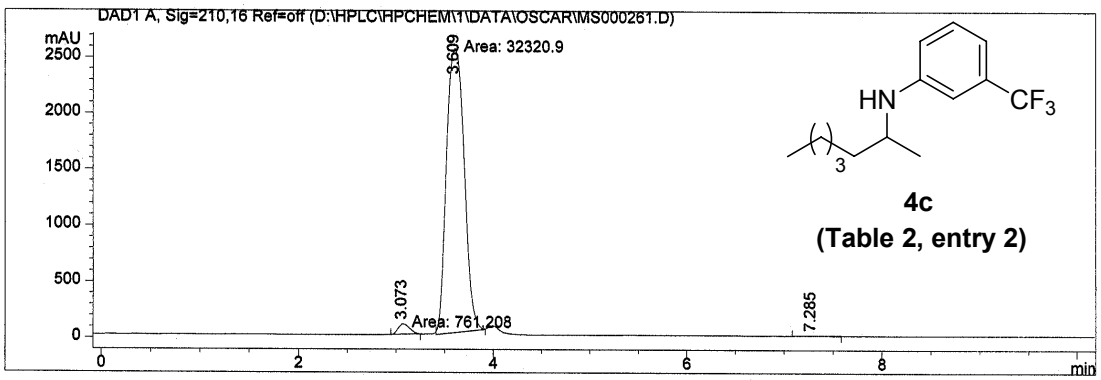
4c

(Table 2, entry 2)

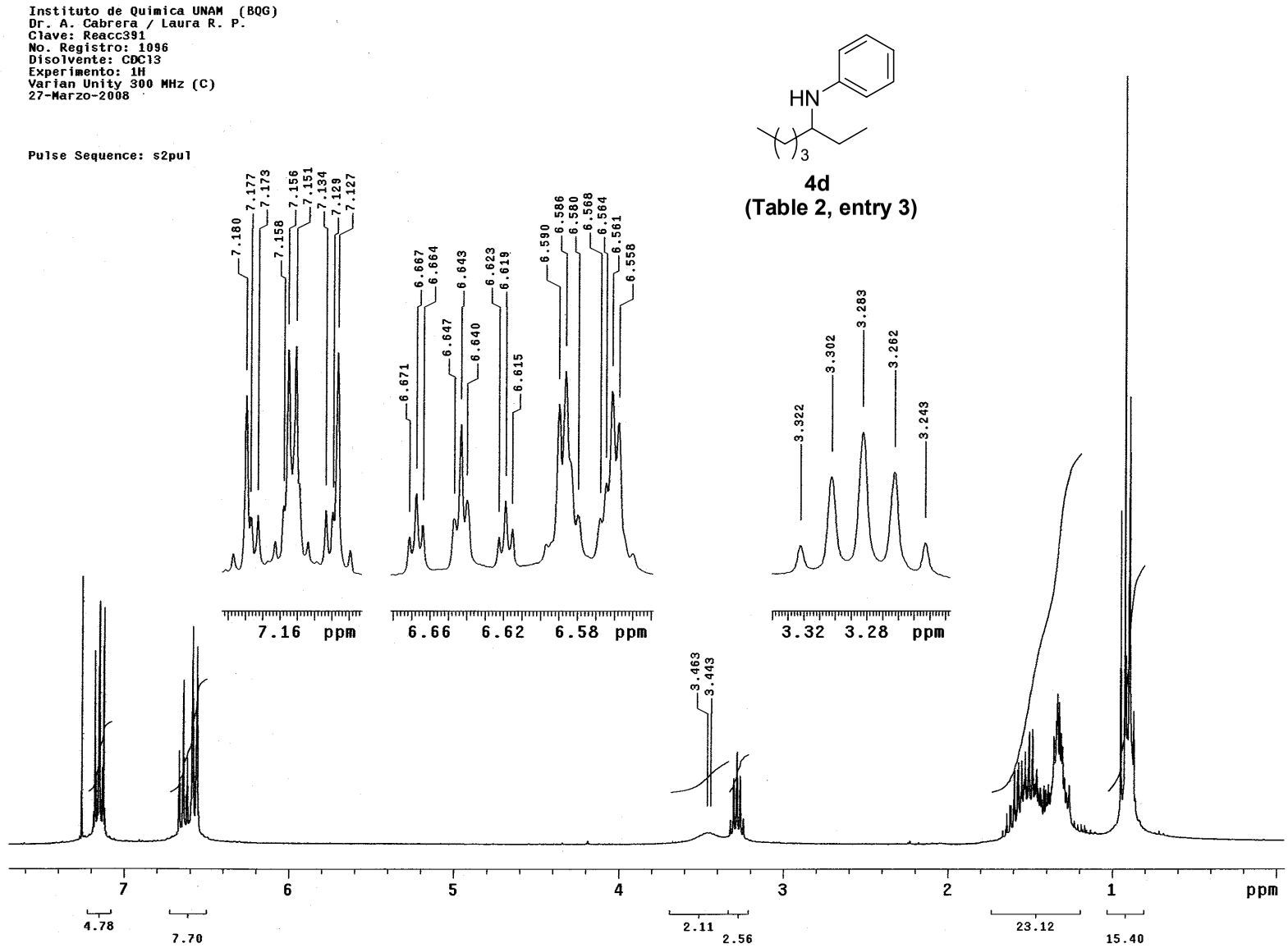
Data File D:\HPLC\HPCHEM\1\DATA\OSCAR\MS000261.D Sample Name: Reacc409

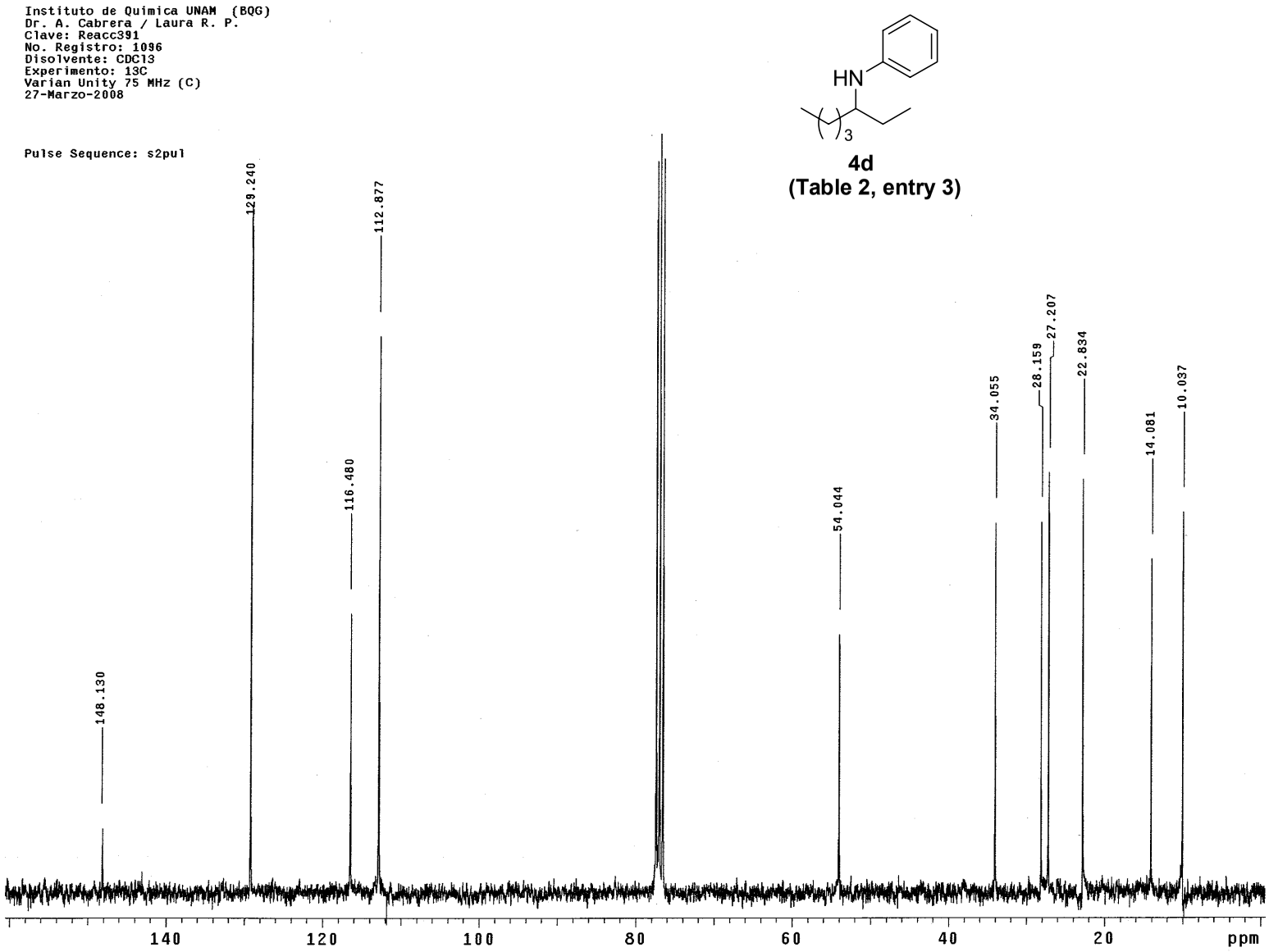
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

```
==========================================================================
Injection Date  : 20/06/08 4:26:23 PM
Sample Name     : Reacc409                              Vial :   1
Acq. Operator   : carmen
Acq. Method     : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed    : 20/06/08 3:40:09 PM by carmen
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed    : 12/09/08 12:43:24 PM by 428
                  (modified after loading)
para Le legadec
==========================================================================
```

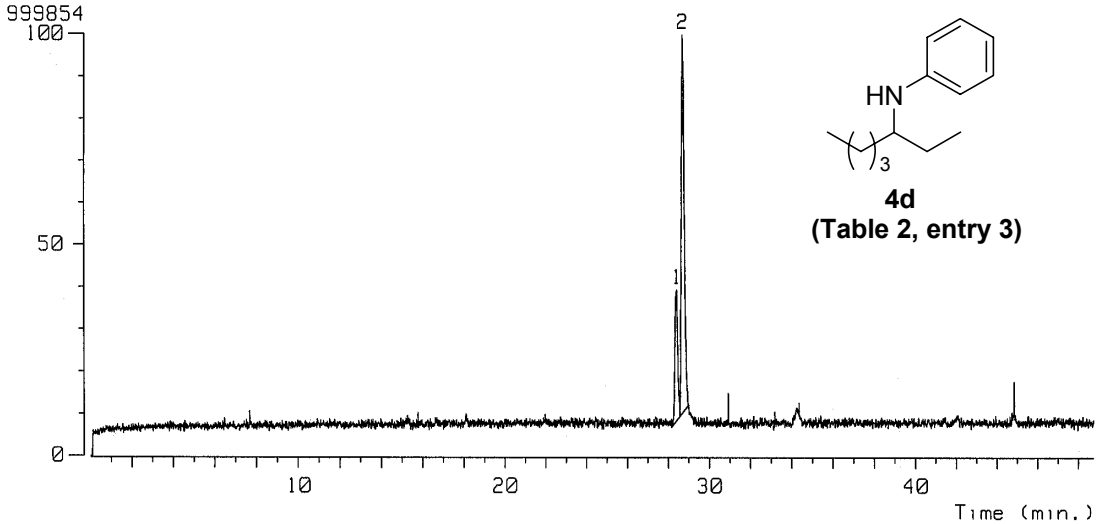



```
==========================================================================
                              Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: DAD1 A, Sig=210,16 Ref=off
 Results obtained with enhanced integrator!

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.073 MM    0.1349  761.20813   94.03625   2.2999
   2   3.609 MM    0.2120 3.23209e4  2541.08276  97.6527
   3   7.285 BP    0.1814   15.70433    1.33349   0.0474

Totals :                  3.30978e4  2636.45250

==========================================================================
```


Instituto de Quimica UNAM (BQG)
Dr. A. Cabrera / Laura R. P.
Clave: Reacc391
No. Registro: 1096
Disolvente: CDCl3
Experimento: 1H
Varian Unity 300 MHz (C)
27-Marzo-2008
Pulse Sequence: s2pul
HN
3
4d
(Table 2, entry 3)
7.180
7.177
7.173
7.158
7.156
7.151
7.134
7.129
7.127
6.671
6.667
6.664
6.647
6.643
6.640
6.623
6.619
6.615
6.590
6.586
6.580
6.568
6.564
6.561
6.558
3.322
3.302
3.283
3.262
3.243
3.463
3.443
7.16 ppm
6.66 6.62 6.58 ppm
3.32 3.28 ppm
7 6 5 4 3 2 1 ppm
4.78
7.70
2.11
2.56
23.12
15.40

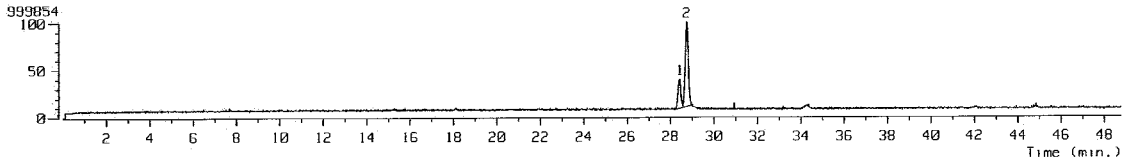

Instituto de Quimica UNAM (BQG)
Dr. A. Cabrera / Laura R. P.
Clave: Reacc391
No. Registro: 1096
Disolvente: CDCl3
Experimento: 13C
Varian Unity 75 MHz (C)
27-Marzo-2008
Pulse Sequence: s2pul
HN
3
4d
(Table 2, entry 3)
148.130
129.240
116.480
112.877
54.044
34.055
28.159
27.207
22.834
14.081
10.037
140
120
100
80
60
40
20
ppm

[TIC]
Data : Dr-Cabrera-Armando-021 Date : 11-Mar-120 15:50
Sample: 595 G Reacc 391 JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 10 to 650

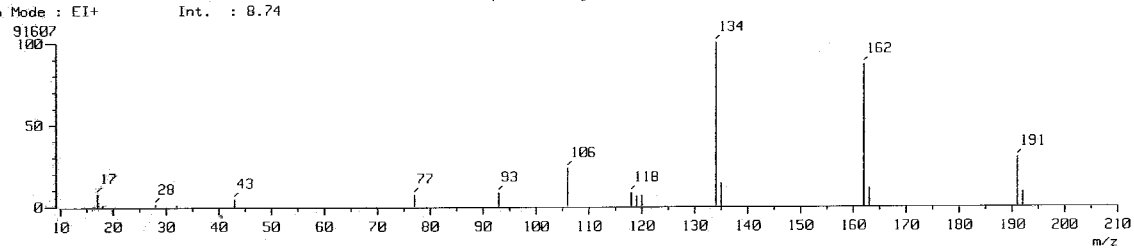


4d
(Table 2, entry 3)

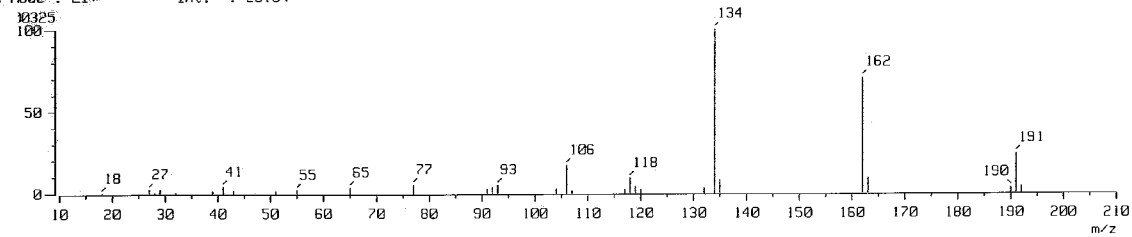
No.	RT[min]	Area	Area%	Height	Height%	Width[sec]	INTEG
1	28.42	280.66	25.22	29.44	25.64	8.95	BV
2	28.73	831.97	74.78	85.35	74.36	9.15	VB

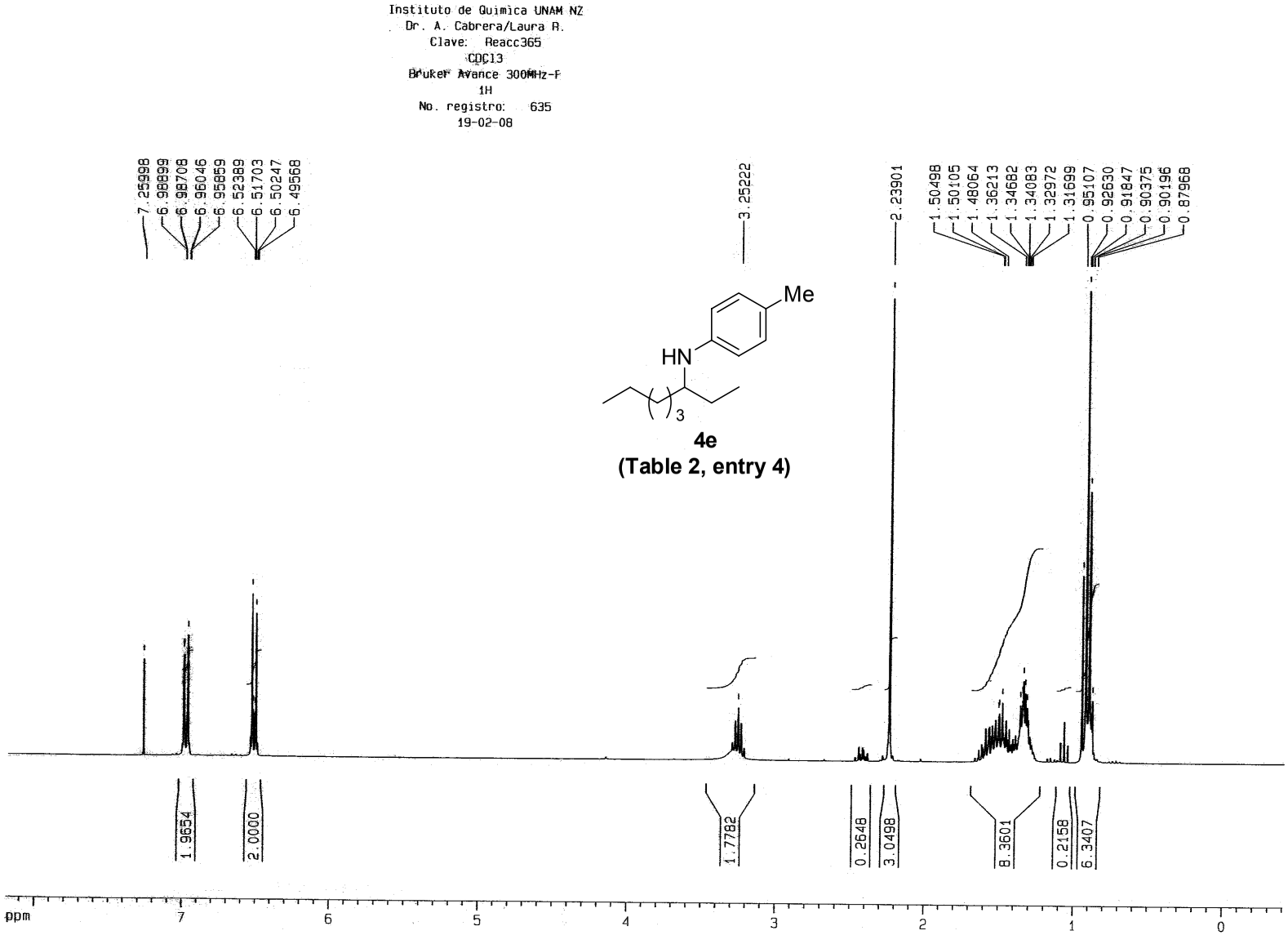


[Mass Spectrum]
RT : 28.42 min Scan# : 2207-2186-2250 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 8.74

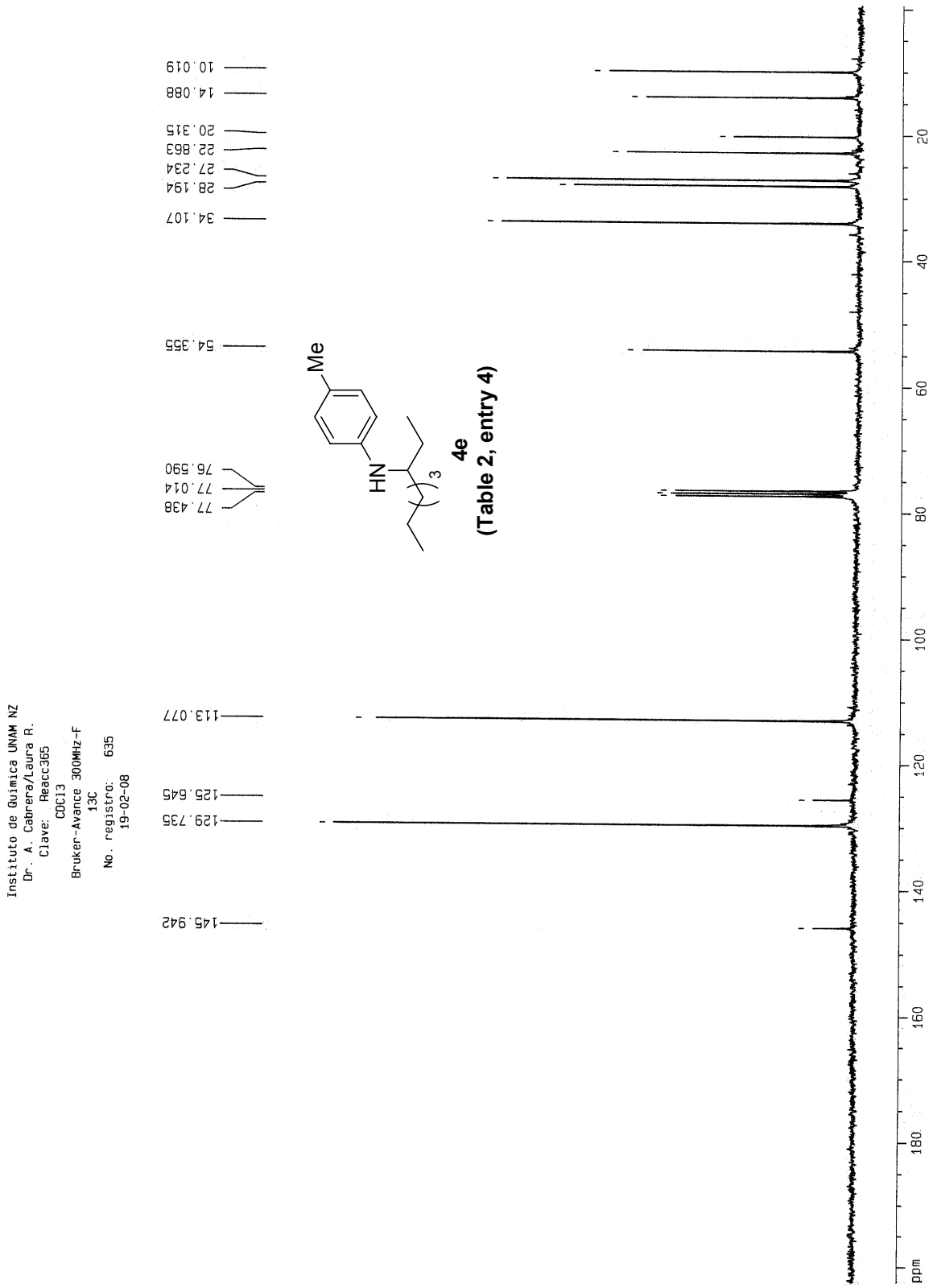


[Mass Spectrum]
RT : 28.73 min Scan# : 2231-2186-2250 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 28.64

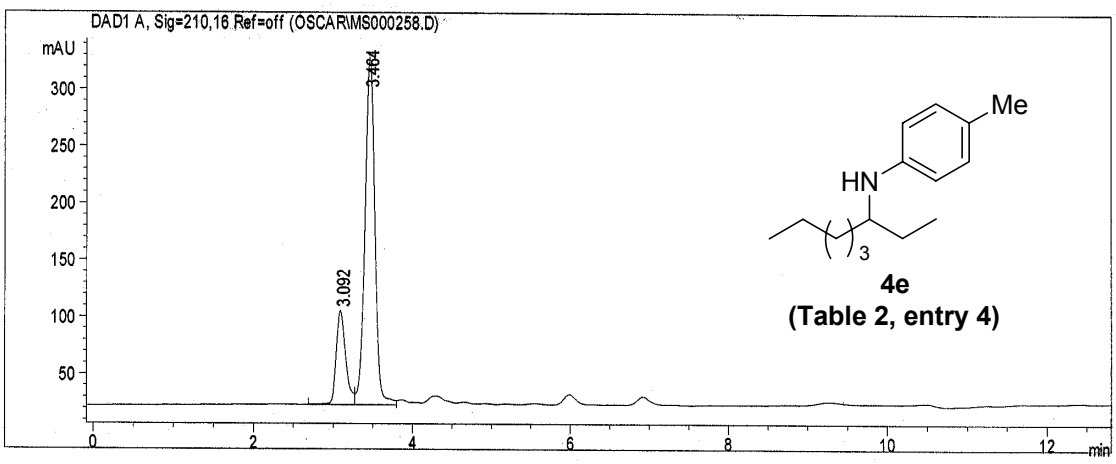




4e
(Table 2, entry 4)


Instituto de Quimica UNAM NZ
Dr. A. Cabrera/Laura R.
Clave: Reacc365
CDCl3
Bruker-Avance 300MHz-F
13C
No. registro: 635
19-02-08
145.942
129.735
125.845
113.077
77.438
77.014
76.590
54.355
34.107
28.194
27.234
22.863
20.315
14.088
10.019
Me
HN
3
4e
(Table 2, entry 4)
ppm
20
40
60
80
100
120
140
160
180

Reacc365
080619-coa-06



Data File C:\HPCHEM\1\DATA\OSCAR\MS000258.D Sample Name: Reacc365
HPLC IQ 20/06/08 2:44:03 PM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

==========

Injection Date : 20/06/08 12:21:49 PM
Sample Name : Reacc365 Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 20/06/08 9:08:55 AM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 20/06/08 12:36:04 PM by carmen
(modified after loading)
para Le legadec

==========

Area Percent Report

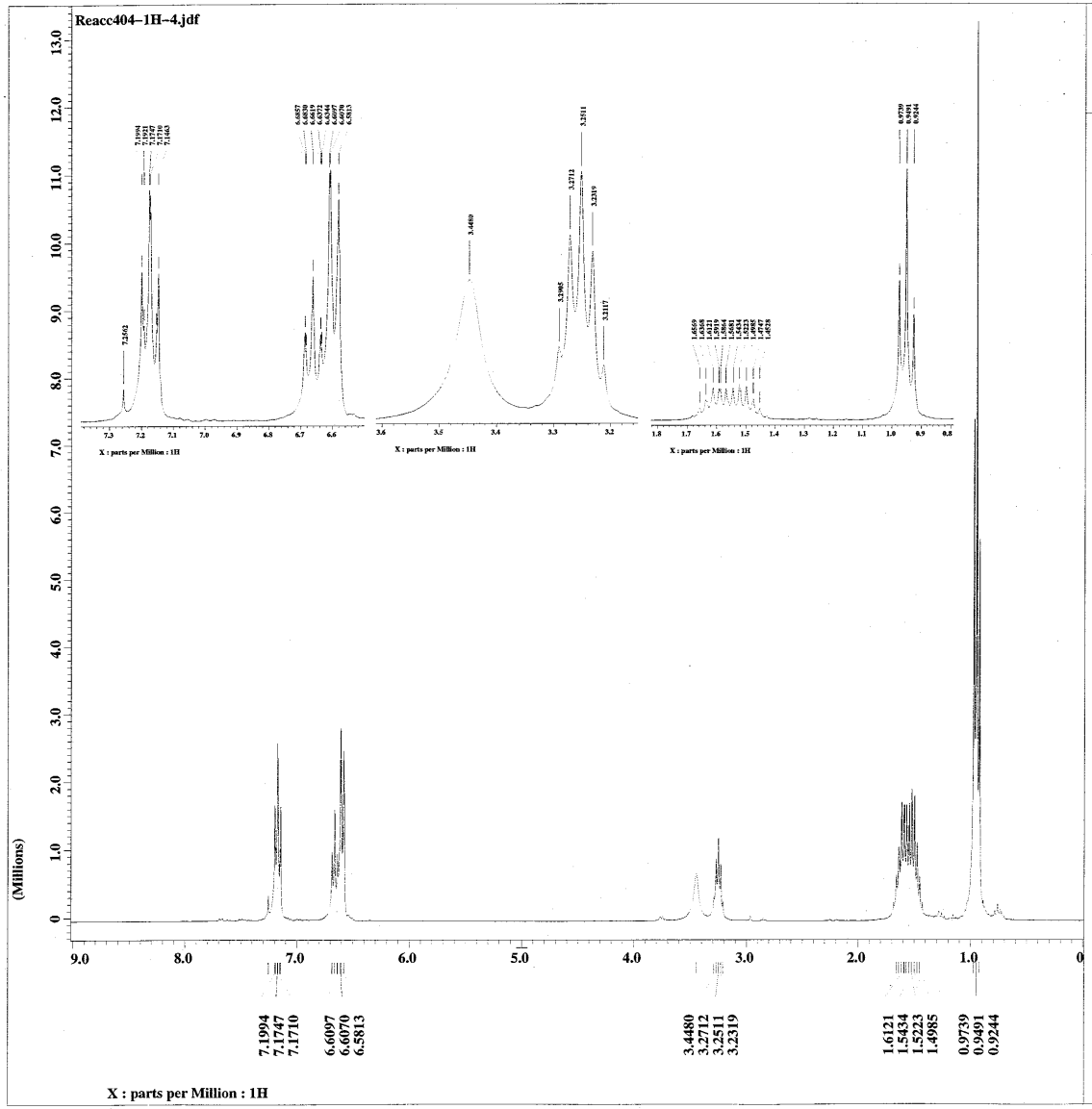
==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

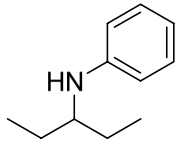
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.092	BV	0.1243	682.65735	82.69392	20.4483
2	3.464	VV	0.1375	2655.80273	305.39322	79.5517
Totals :				3338.46008	388.08714	

ee = 59.10%

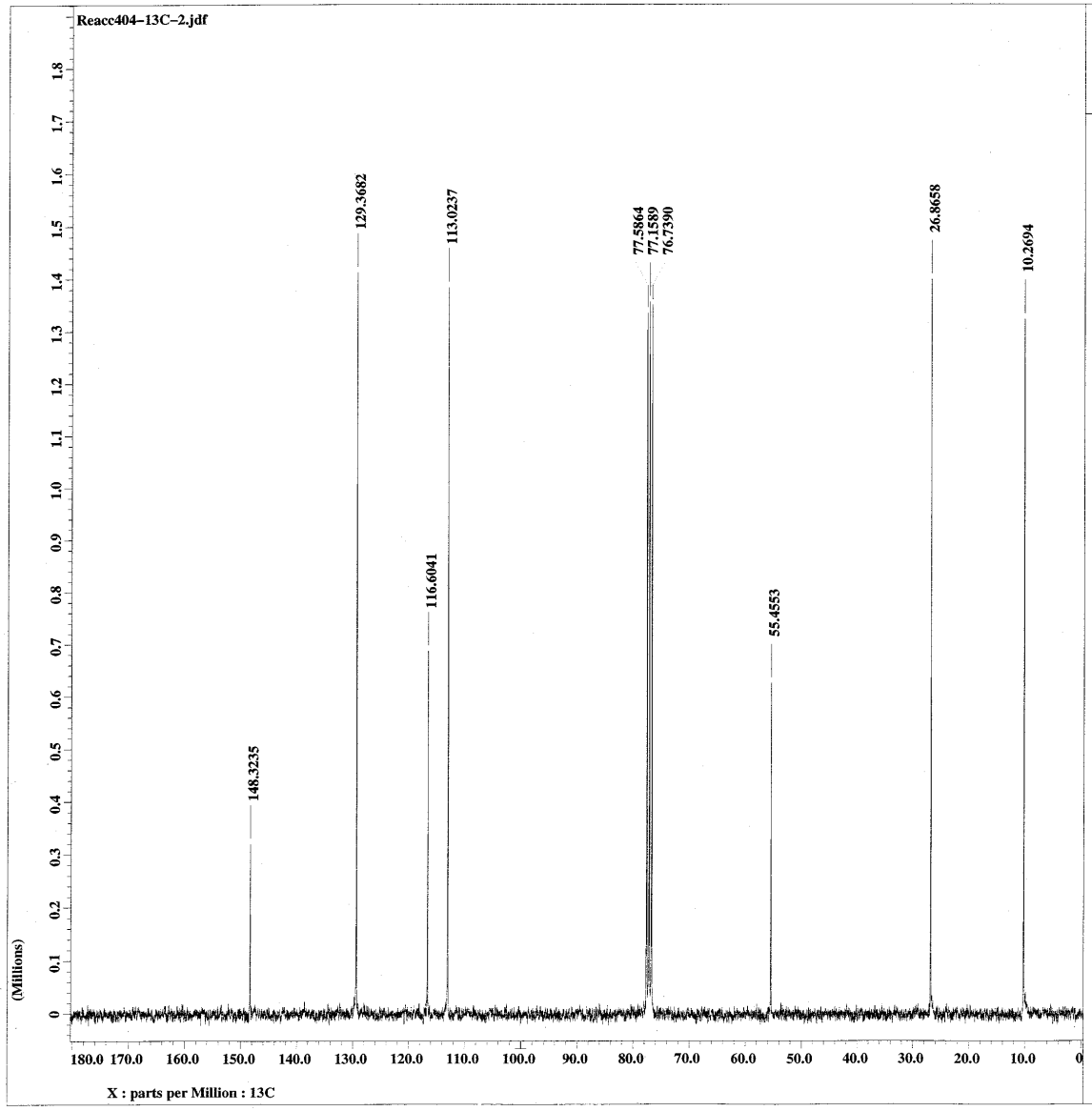


JEOL

```
Filename          = Reacc404-1H-4.jdf
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 26-FEB-2008 13:33:15
Revision_time     =  9-MAY-2008 07:48:27
Current_time      =  9-MAY-2008 07:49:02

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 16
Relaxation_delay  = 1[s]
Temp_get          = 17.7[dC]
Unblank_time      = 2[us]
```

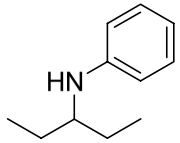


4f
(Table 2, entry 5)

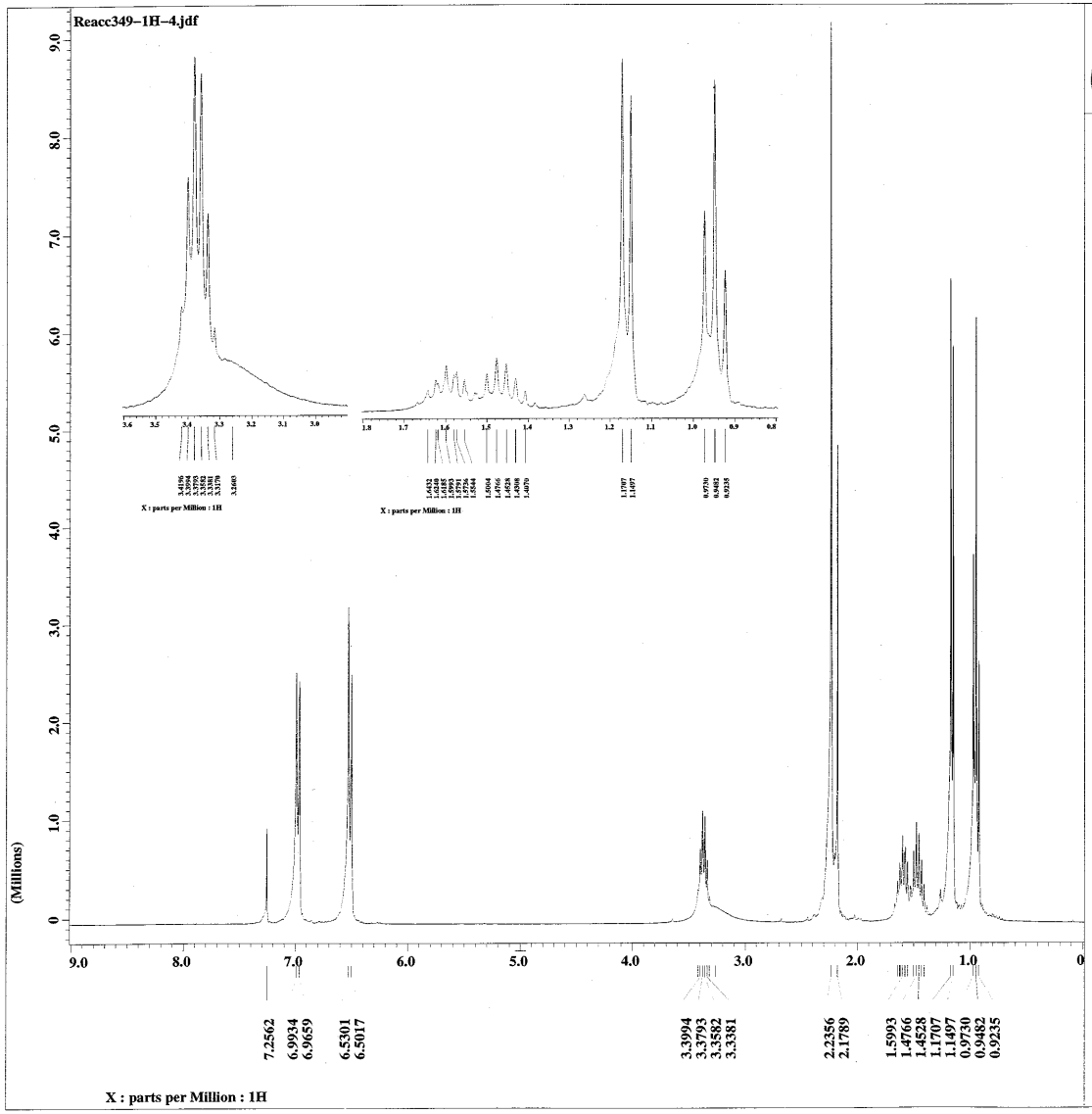


JEOL

```
Filename          = Reacc404-13C-2.jdf
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 26-FEB-2008 14:01:02
Revision_time     =  9-MAY-2008 07:44:13
Current_time      =  9-MAY-2008 07:44:33

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 584
Total_scans       = 584

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 19.3[dC]
Unblank_time      = 2[us]
```

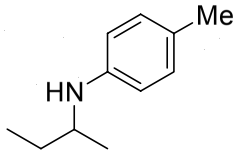


4f
(Table 2, entry 5)

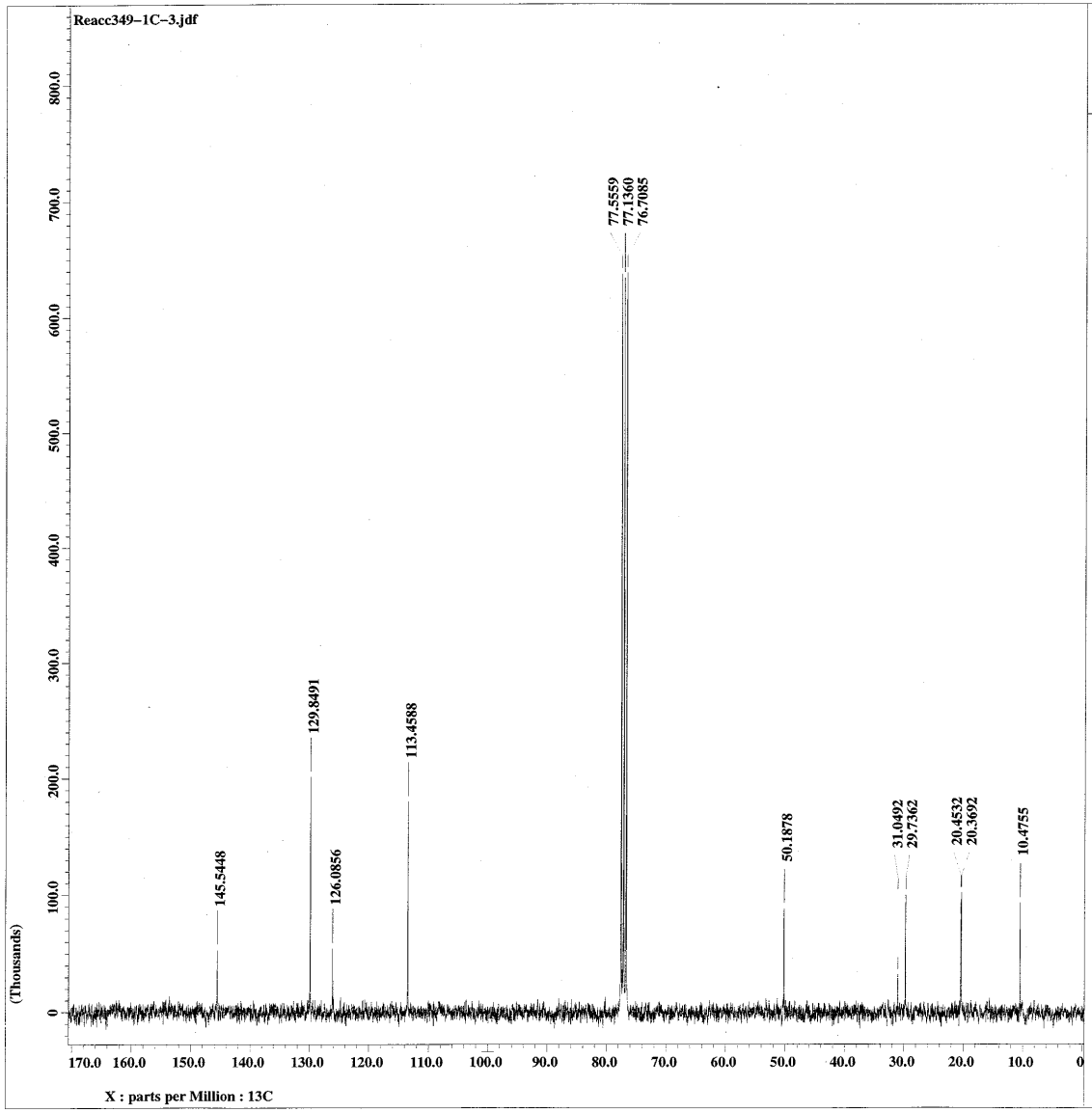


JEOL

Filename	= Reacc349-1H-4.jdf
Author	= Cabrera
Experiment	= single_pulse.exp
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 22-JAN-2008 15:47:34
Revision_time	= 4-APR-2008 08:40:51
Current_time	= 4-APR-2008 08:44:17
Comment	= Single Pulse Experime
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 32
Total_scans	= 32
X_90_width	= 9.5[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 4.75[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 19
Relaxation_delay	= 4[s]
Temp_get	= 18[dC]
Unblank_time	= 2[us]

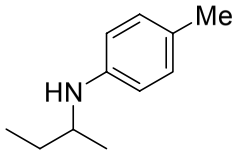


4g
(Table 2, entry 6)



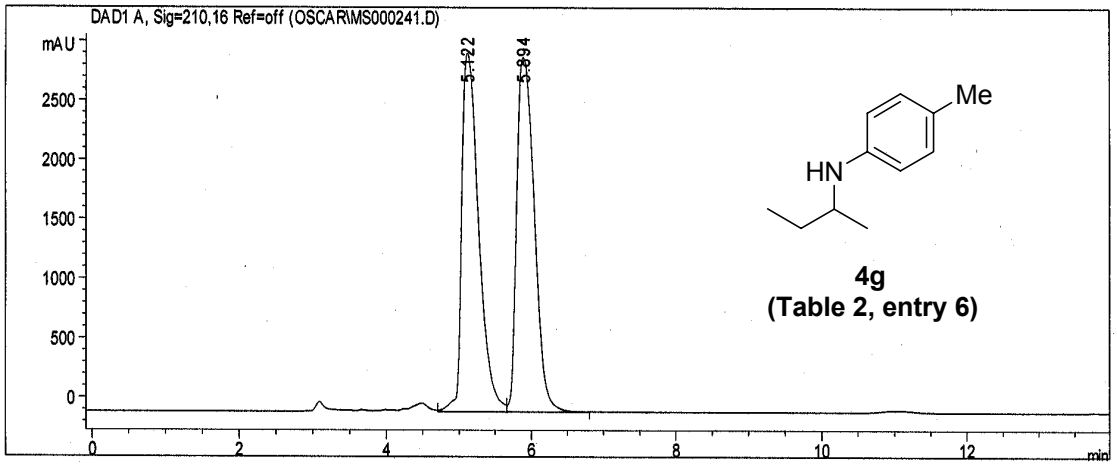
JEOL

Filename	= Reacc349-1C-3.jdf
Author	= Cabrera
Experiment	= single_pulse_dec
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 22-JAN-2008 17:03:17
Revision_time	= 4-APR-2008 09:47:27
Current_time	= 4-APR-2008 09:47:41
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1563
Total_scans	= 1563
X_90_width	= 11.3[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 3.76666667[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 30
Relaxation_delay	= 1[s]
Temp_get	= 20[dC]
Unblank_time	= 2[us]



4g
(Table 2, entry 6)

Reacc349 rac
080617-coa07



Data File C:\HPCHEM\1\DATA\OSCAR\MS000241.D Sample Name: Reacc331rac
HPLC IQ 09/09/08 5:50:01 PM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 95/5
flujo 1 ml/min
UV 254

==========

Injection Date : 09/09/08 12:24:12 PM
Sample Name : Reacc349rac Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 10/09/08 9:44:12 AM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 10/09/08 9:02:34 AM by carmen
(modified after loading)

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

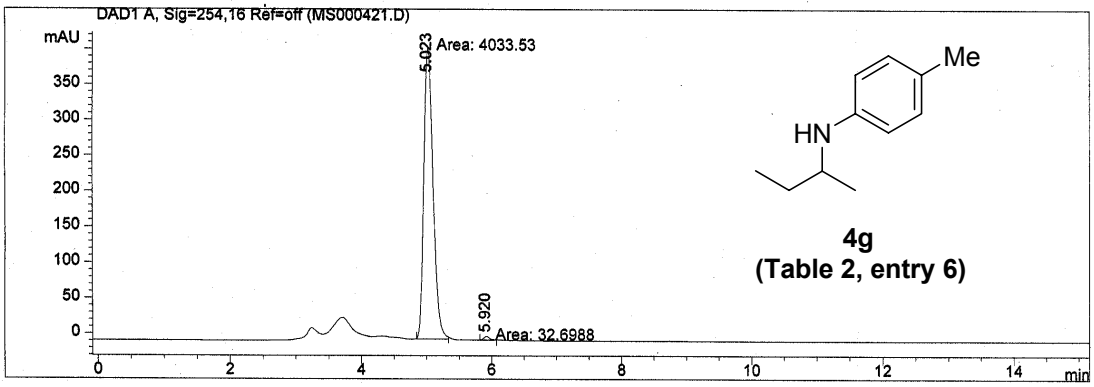
Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.122	VV	0.2500	4.91991e4	3021.12646	49.2589
2	5.894	VV	0.2743	5.06794e4	2981.29468	50.7411
Totals :				9.98785e4	6002.42114	

Data File C:\HPCHEM\1\DATA\MS000421.D Sample Name: ~~Reacc 431F24~~ Reacc349
080825-coa-07

Chiralcel OD 100 5 250x 4.6 mm
hexano/isopropanol 95/5
flujo 1 ml/min
UV 254 nm

==========
Injection Date : 11/09/08 12:13:20 PM
Sample Name : Reacc349 Vial : 1
Acq. Operator : 428
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 11/09/08 11:09:31 AM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 08/09/08 7:24:29 PM by carmen
para Le legadec
==========



==========
Area Percent Report
==========

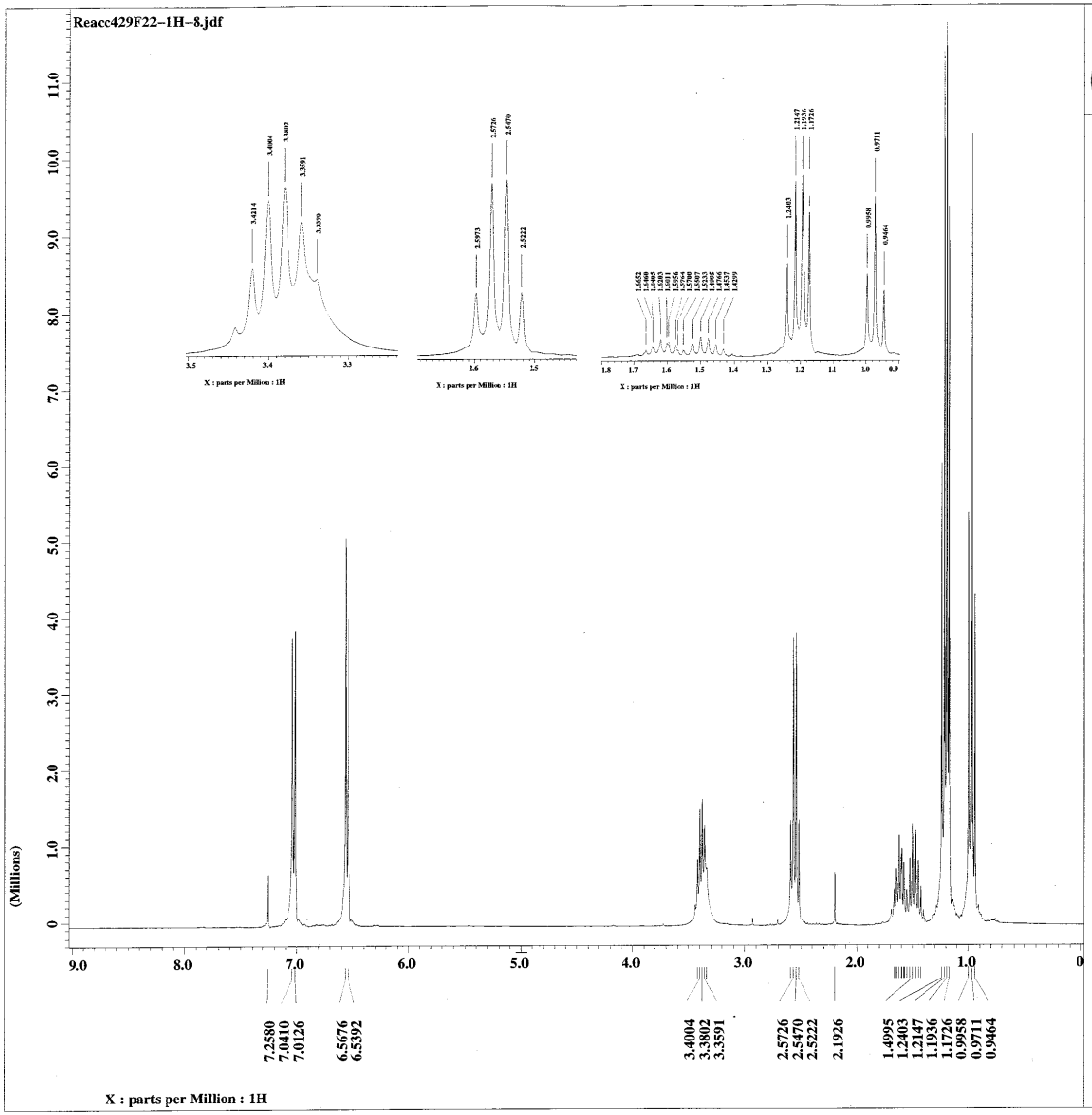
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.023	MM	0.1634	4033.52783	411.43063	99.1958
2	5.920	MM	0.1086	32.69881	5.01843	0.8042

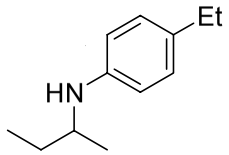
Totals : 4066.22664 416.44907

==========
*** End of Report ***

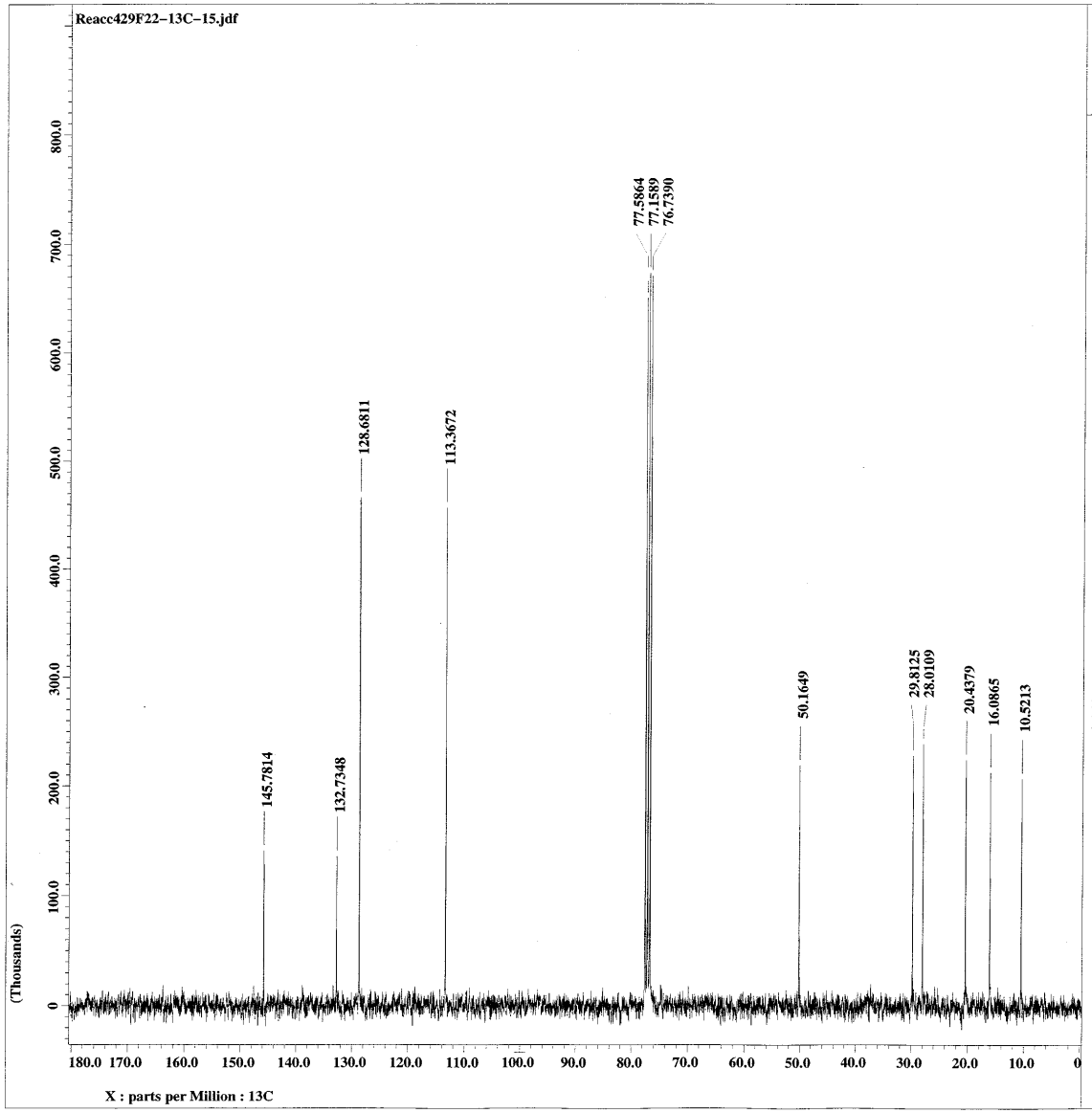


JEOL

```
Filename          = Reacc429F22-1H-8.jdf
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 31-MAY-2008 00:09:51
Revision_time     =  1-SEP-2008 14:26:37
Current_time      =  1-SEP-2008 14:27:53

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 16
Relaxation_delay  = 1[s]
Temp_get          = 18.9[dC]
Unblank_time      = 2[us]
```

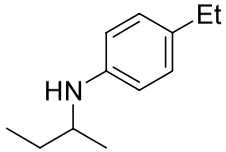


4h
(Table 2, entry 7)



JEOL

```
Filename          = Reacc429F22-13C-15.jd
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 31-MAY-2008 01:14:54
Revision_time     =  1-SEP-2008 13:08:59
Current_time      =  1-SEP-2008 13:09:15

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 964
Total_scans       = 964

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 20.6[dC]
Unblank_time      = 2[us]
```



4h
(Table 2, entry 7)

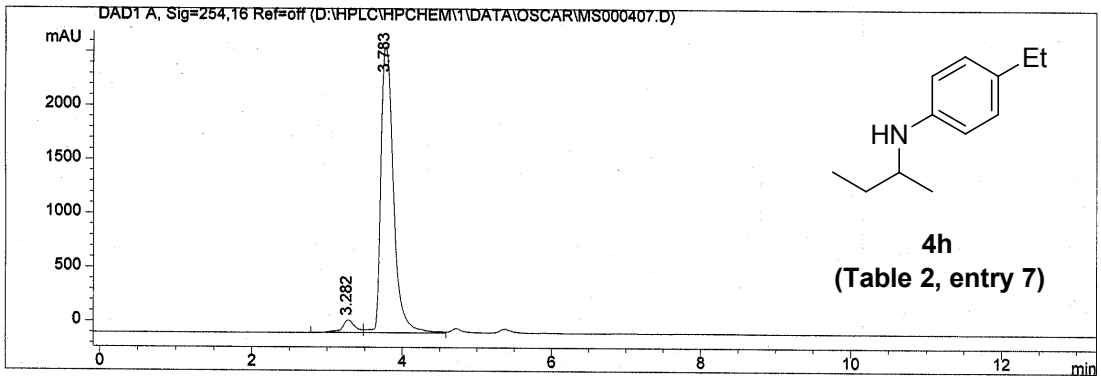
Data File D:\HPLC\HPCHEM\1\DATA\OSCAR\MS000407.D Sample Name: Reacc 429F23
080825-coa-03

Chiralcel OD 100 5 250x 4.6 mm
hexano/isopropanol 90/10
flujo 1 ml/min
UV 254 nm

==========

Injection Date : 04/09/08 1:10:41 PM
Sample Name : Reacc 429F23 Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 04/09/08 11:49:21 AM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 11/09/08 11:09:31 AM by carmen
(modified after loading)
para Le legadec

==========



==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

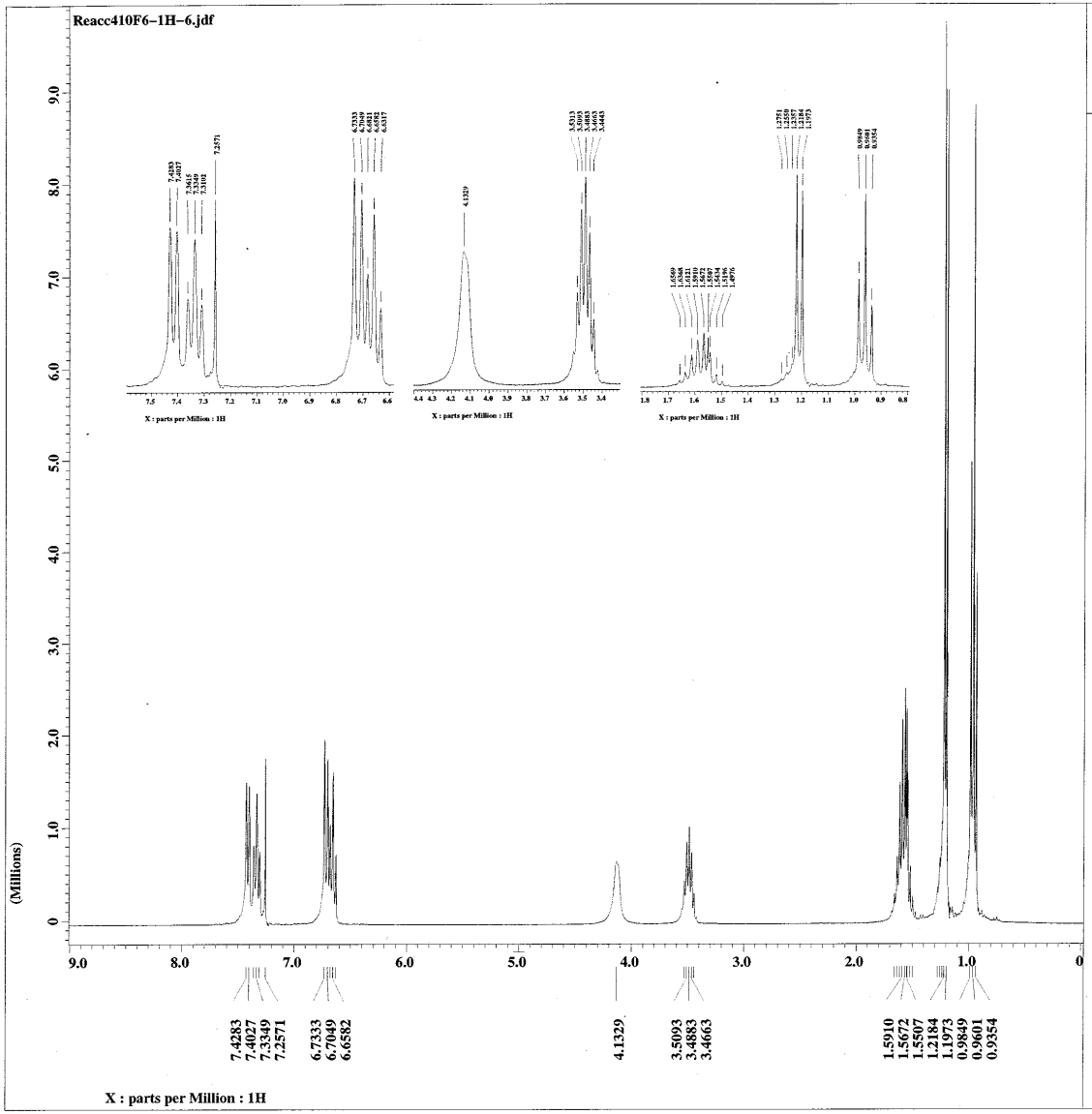
Signal 1: DAD1 A, Sig=254,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.282	BV	0.1674	1359.57544	117.16486	4.1915
2	3.783	VV	0.1846	3.10769e4	2654.95972	95.8085

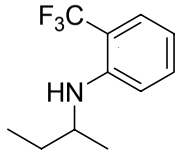
Totals : 3.24365e4 2772.12458 ee= 91.61 ~92%

==========

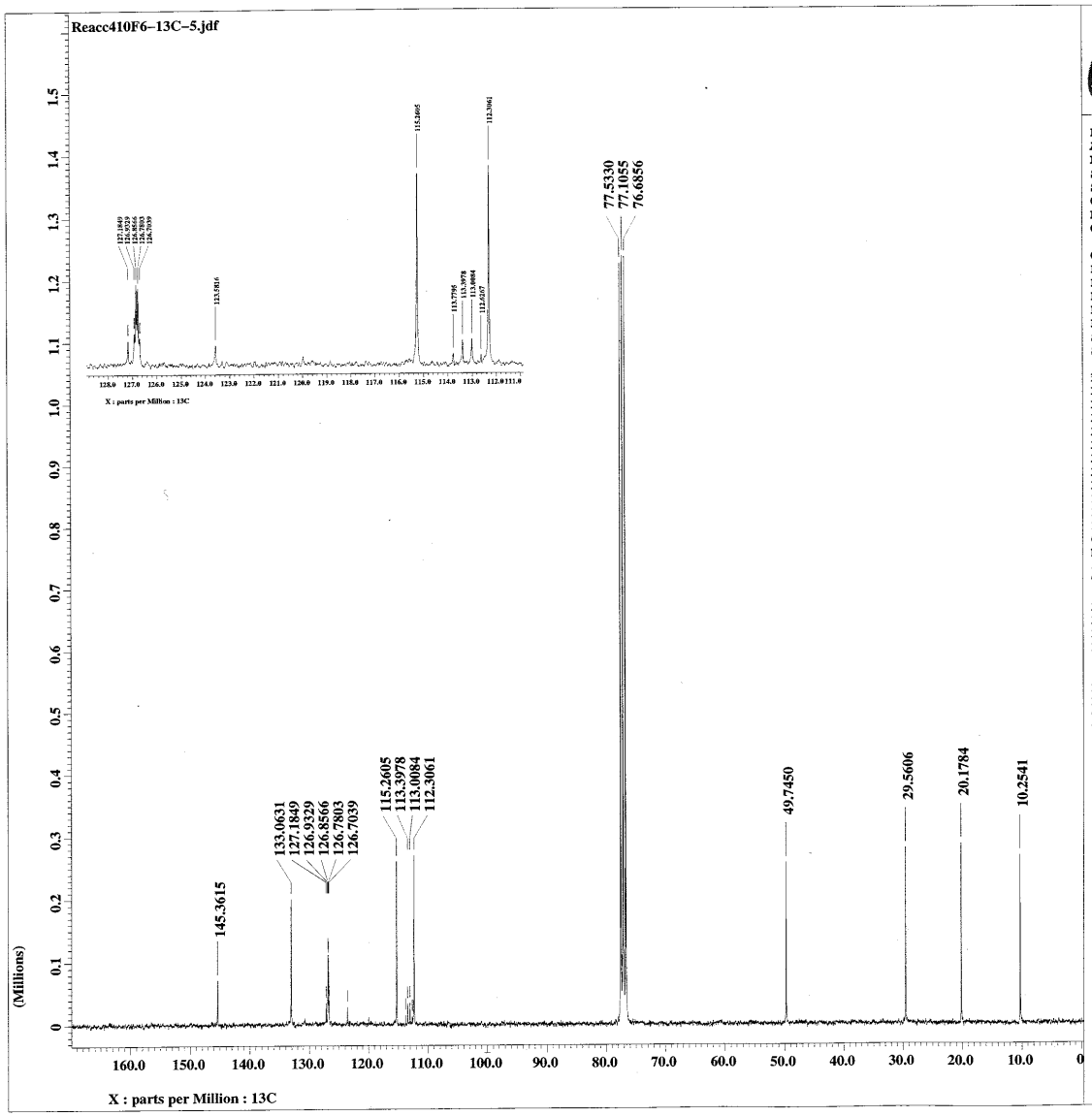
*** End of Report ***




```
Filename          = Reacc410F6-1H-6.jdf
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 11-APR-2008 09:48:29
Revision_time     =  1-SEP-2008 14:07:21
Current_time      =  1-SEP-2008 14:08:04

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 20
Relaxation_delay  = 1[s]
Temp_get          = 19.4[dC]
Unblank_time      = 2[us]
```

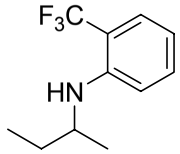


4i
(Table 2, entry 8)



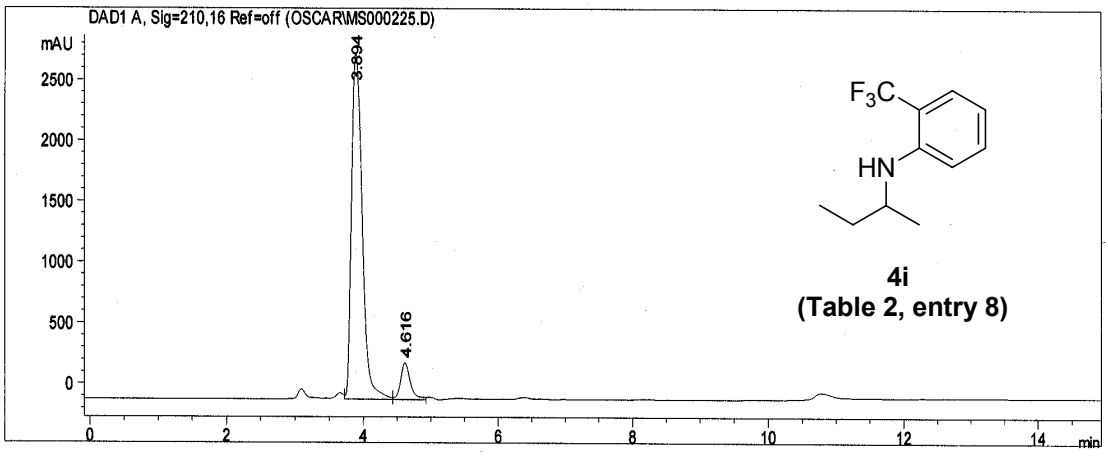
JEOL

Filename	= Reacc410F6-13C-5.jdf
Author	= Cabrera
Experiment	= single_pulse_dec
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 11-APR-2008 15:08:40
Revision_time	= 1-SEP-2008 14:09:19
Current_time	= 1-SEP-2008 14:12:37
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 7000
Total_scans	= 7000
X_90_width	= 11.3[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 3.76666667[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 30
Relaxation_delay	= 1[s]
Temp_get	= 20.9[dC]
Unblank_time	= 2[us]



4i
(Table 2, entry 8)

Reacc410F21
080616-coa-10



Data File C:\HPCHEM\1\DATA\OSCAR\MS000225.D Sample Name: Reacc410F21
HPLC IQ 18/06/08 3:50:52 PM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

==========

Injection Date : 16/06/08 3:32:37 PM
Sample Name : Reacc410F21 Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 16/06/08 3:24:23 PM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 18/06/08 3:38:29 PM by carmen
(modified after loading)
para Cabrera Armando

==========

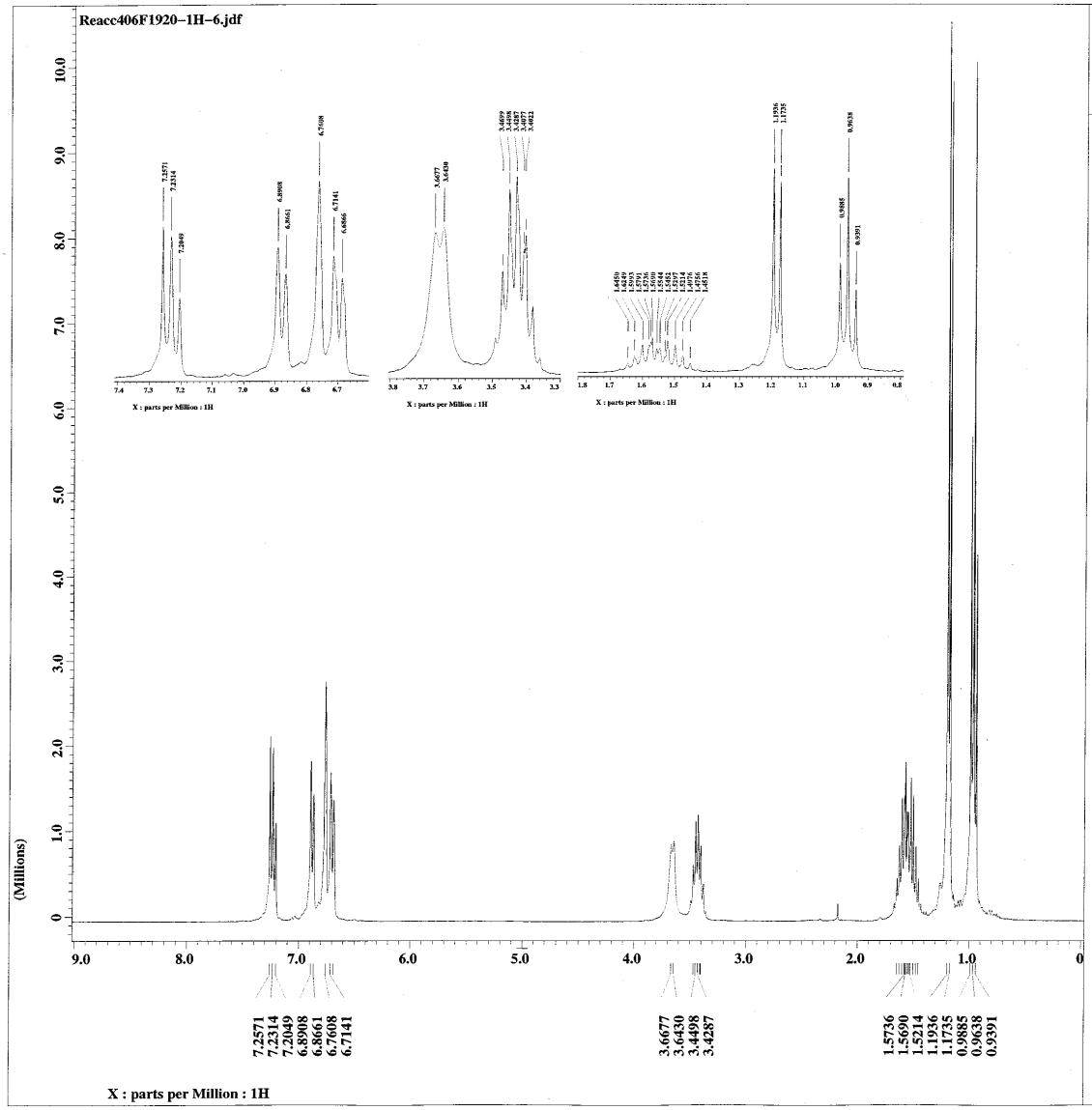
Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

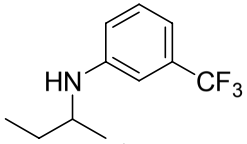
Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.894	VV	0.1715	3.12233e4	2856.57617	91.2217
2	4.616	VV	0.1480	3004.63037	302.16672	8.7783
Totals :				3.42279e4	3158.74289	

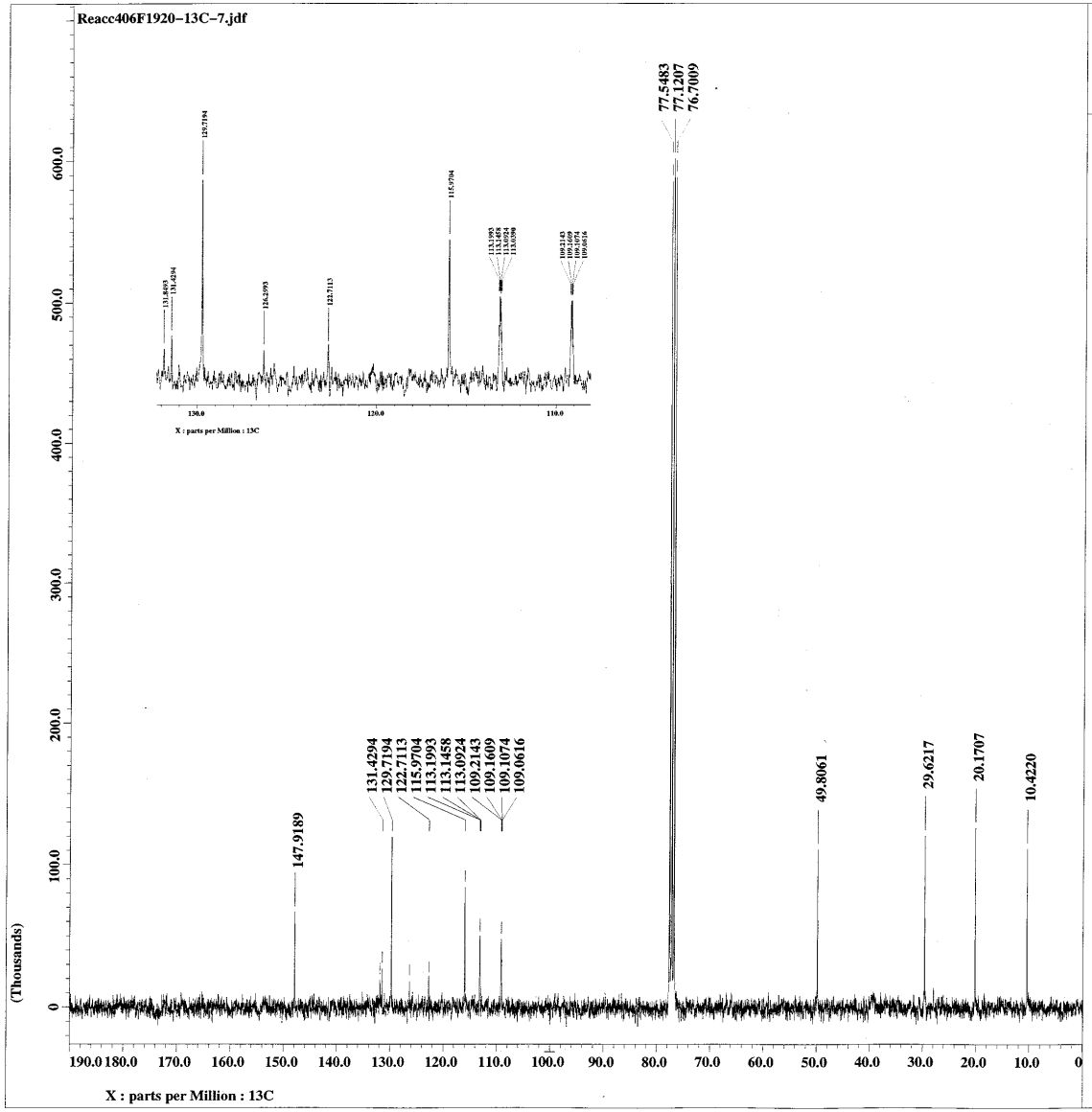


JEOL

```
Filename          = Reacc406F1920-1H-6.jd
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     =  9-MAR-2008 20:11:54
Revision_time     =  2-SEP-2008 07:09:21
Current_time      =  2-SEP-2008 07:11:18

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 18
Relaxation_delay  = 1[s]
Temp_get          = 16.2[dC]
Unblank_time      = 2[us]
```

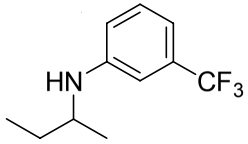


4j
(Table 2, entry 9)



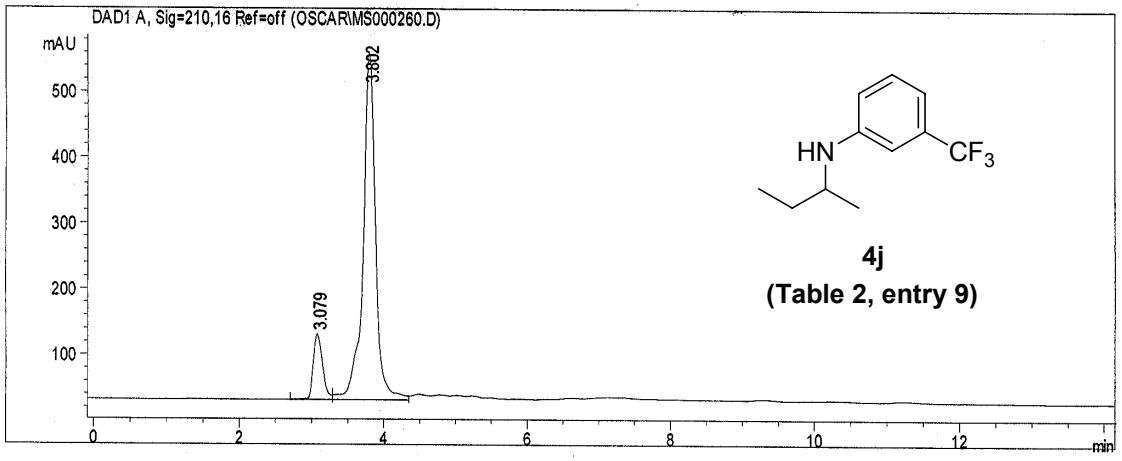
JEOL

Filename	= Reacc406F1920-13C-7.j
Author	= Cabrera
Experiment	= single_pulse_dec
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 9-MAR-2008 22:13:18
Revision_time	= 2-SEP-2008 07:14:55
Current_time	= 2-SEP-2008 07:15:38
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 2639
Total_scans	= 2639
X_90_width	= 11.3[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 3.76666667[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 30
Relaxation_delay	= 1[s]
Temp_get	= 18.4[dC]
Unblank_time	= 2[us]



4j
(Table 2, entry 9)

Reacc406F21
080619-coa-08



Data File C:\HPCHEM\1\DATA\OSCAR\MS000260.D Sample Name: Reacc406F21
HPLC IQ 20/06/08 5:22:37 PM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

==========

Injection Date : 20/06/08 4:11:22 PM
Sample Name : Reacc406F21 Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 20/06/08 3:40:09 PM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 20/06/08 5:08:36 PM by carmen
(modified after loading)
para Le legadec

==========

Area Percent Report

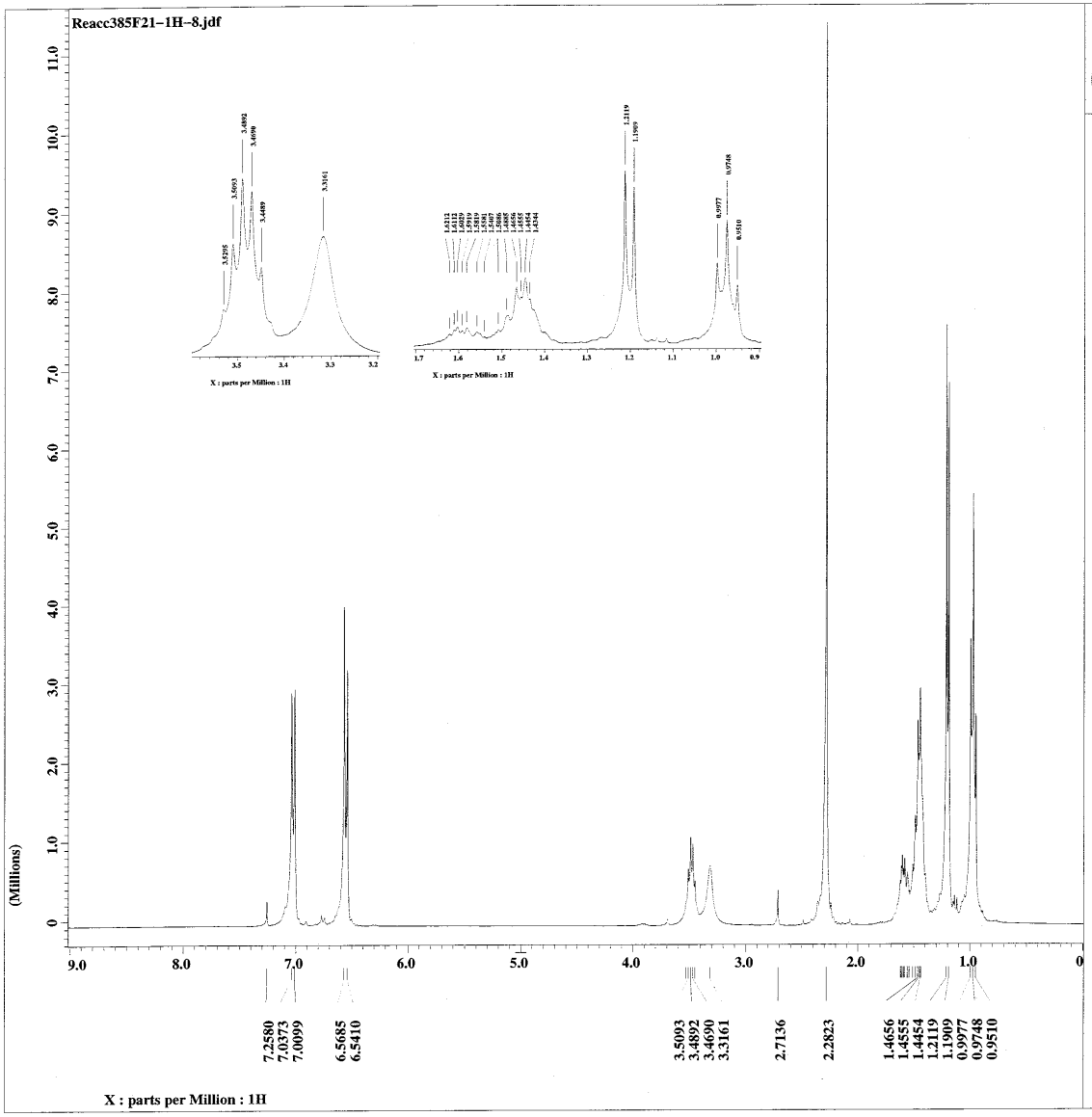
==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

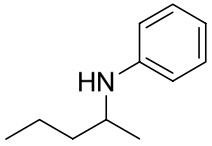
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.079	BV	0.1435	892.61469	100.62816	12.2826
2	3.802	VV	0.1802	6374.69727	530.28589	87.7174
Totals :				7267.31195	630.91405	

ee = 75.43%

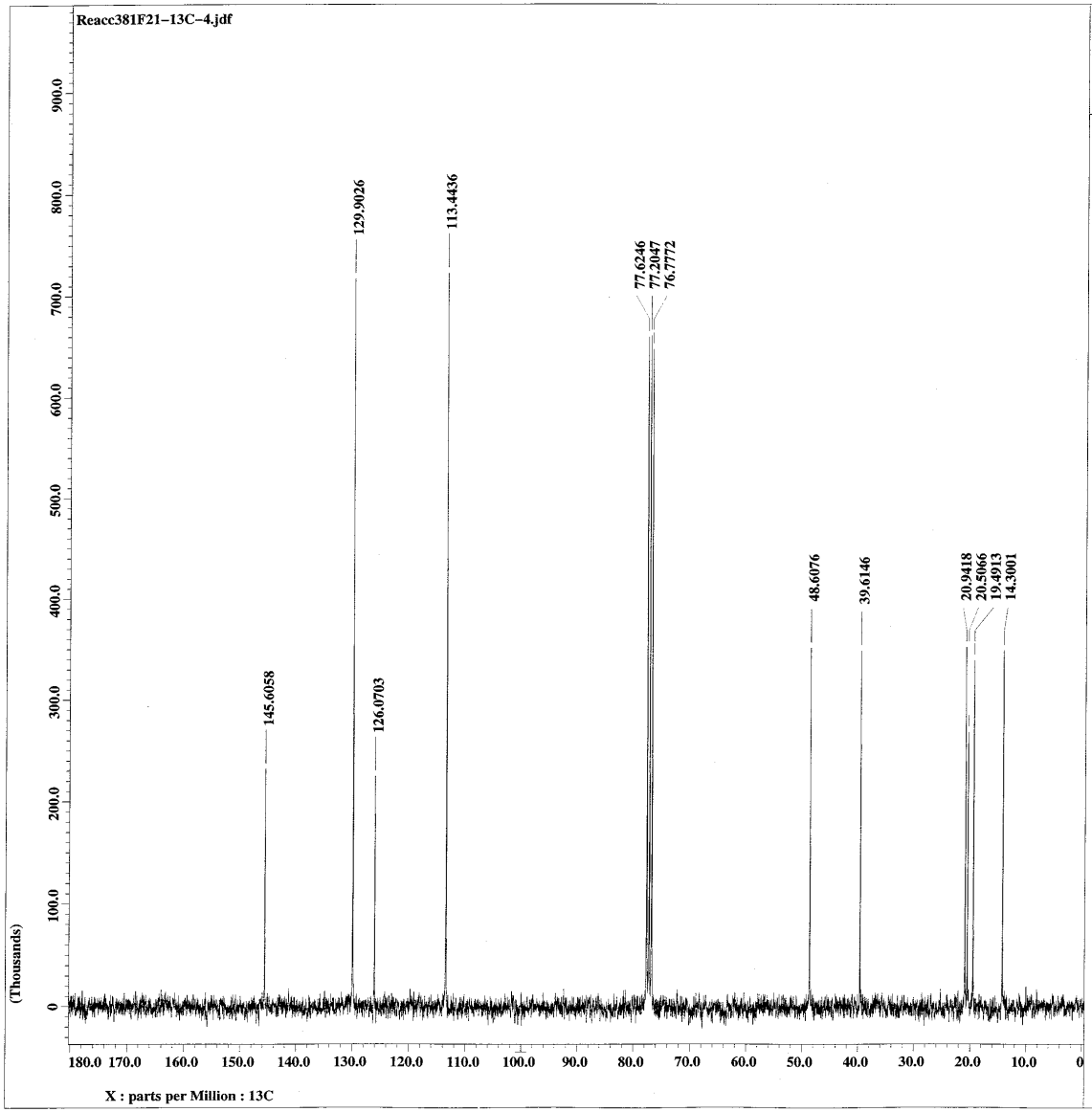


JEOL

```
Filename          = Reacc385F21-1H-8.jdf
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 25-MAR-2008 13:00:30
Revision_time     =  2-SEP-2008 07:06:13
Current_time      =  2-SEP-2008 07:08:46

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 14
Relaxation_delay  = 1[s]
Temp_get          = 17.5[dC]
Unblank_time      = 2[us]
```

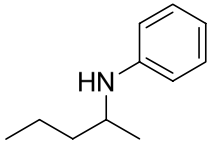


4k
(Table 2, entry 10)



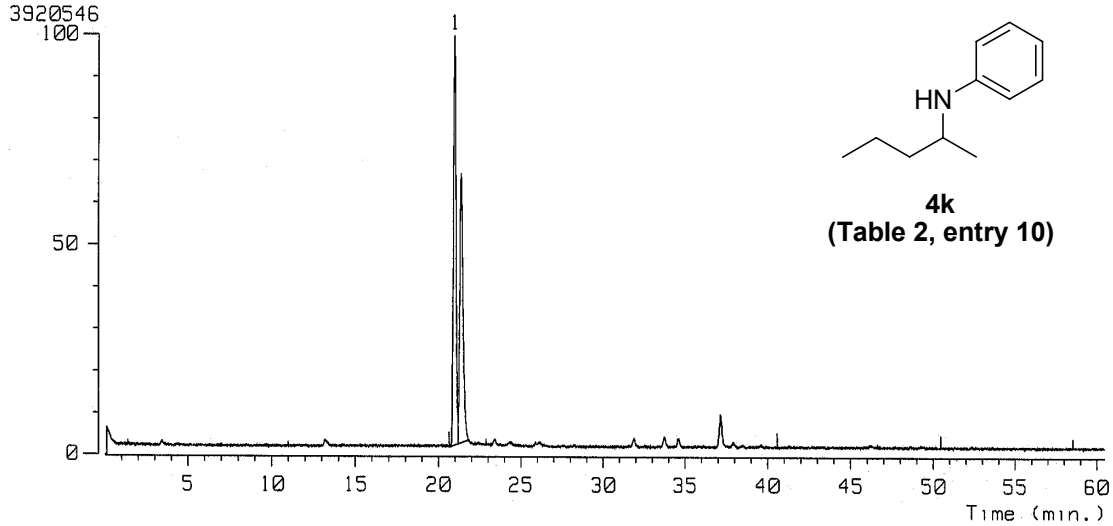
JEOL

```
Filename          = Reacc381F21-13C-4.jdf
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 25-MAR-2008 13:58:11
Revision_time     =  2-SEP-2008 07:07:19
Current_time      =  2-SEP-2008 07:07:46

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T]  (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1150
Total_scans       = 1150

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 19.4[dC]
Unblank_time      = 2[us]
```



4k
(Table 2, entry 10)

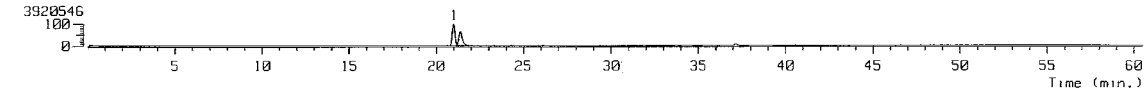
[TIC]
Data : Dr-Cabrera-Armando-004 Date : 04-Mar-108 14:46
Sample: 453 G Reacc 385 JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 10 to 650



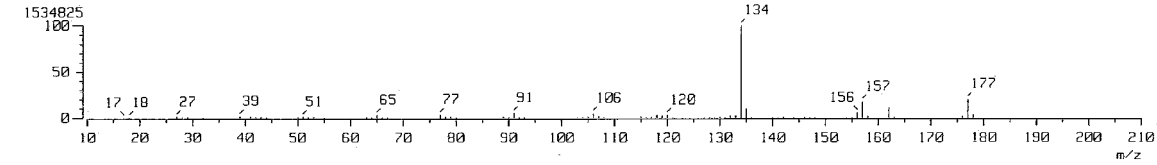
4k
(Table 2, entry 10)

No.	RT[min]	Area	Area%	Height	Height%	Width[sec]	INTEG
1	20.99	3966.79	54.78	364.66	60.35	10.21	BV
2	21.36	3274.27	45.22	239.55	39.65	12.83	VB

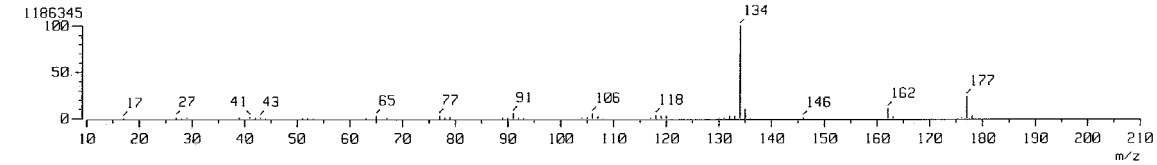
[TIC]
Data : Dr-Cabrera-Armando-004 Date : 04-Mar-108 14:46
Sample: 453 G Reacc 385 JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 10 to 650 Output RT Range : 0.00 to 60.49 min

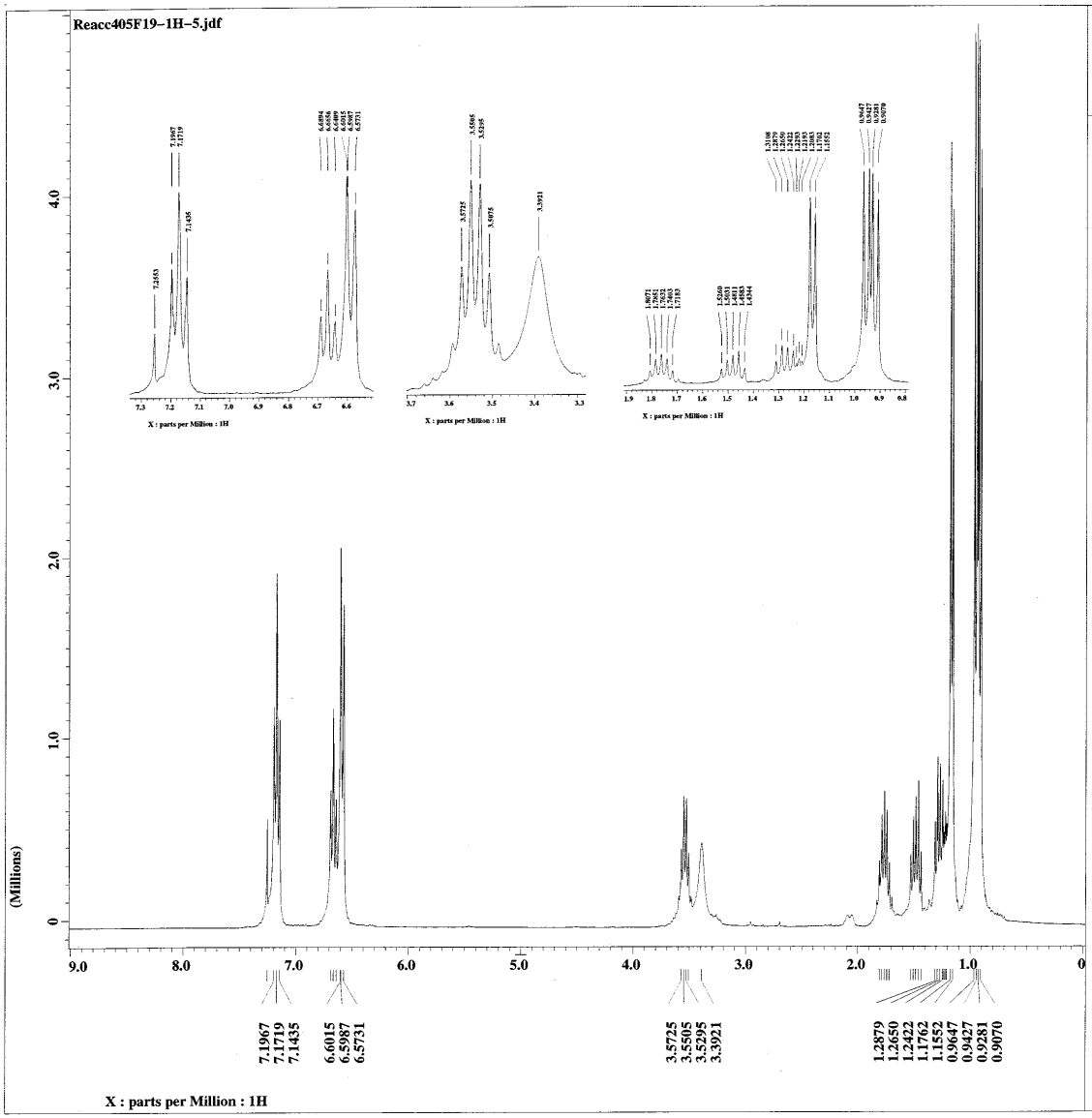


[Mass Spectrum]
RT : 20.99 min Scan# : 1630-1603-1692 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 146.37



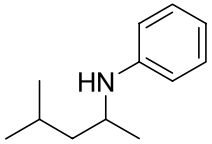
[Mass Spectrum]
RT : 21.36 min Scan# : 1659-1603-1692 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 113.14



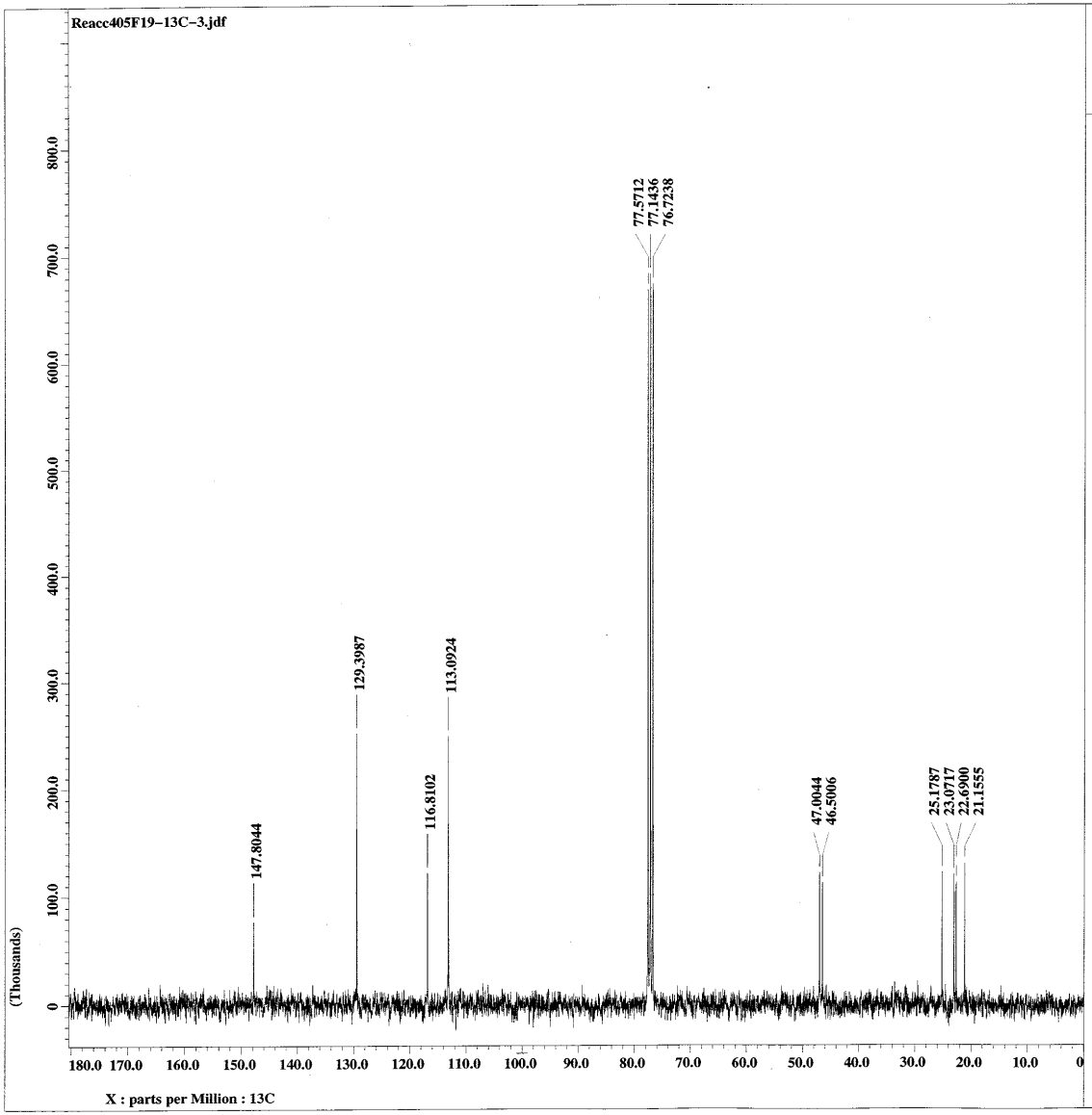


JEOL

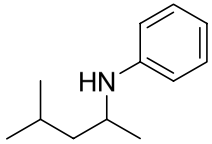
Filename	= Reacc405F19-1H-5.jdf
Author	= Cabrera
Experiment	= single_pulse.exp
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 7-MAR-2008 18:26:22
Revision_time	= 1-SEP-2008 13:50:08
Current_time	= 1-SEP-2008 13:51:51
Comment	= Single Pulse Experime
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 32
Total_scans	= 32
X_90_width	= 9.5[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 4.75[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 17
Relaxation_delay	= 1[s]
Temp_get	= 15.8[dC]
Unblank_time	= 2[us]



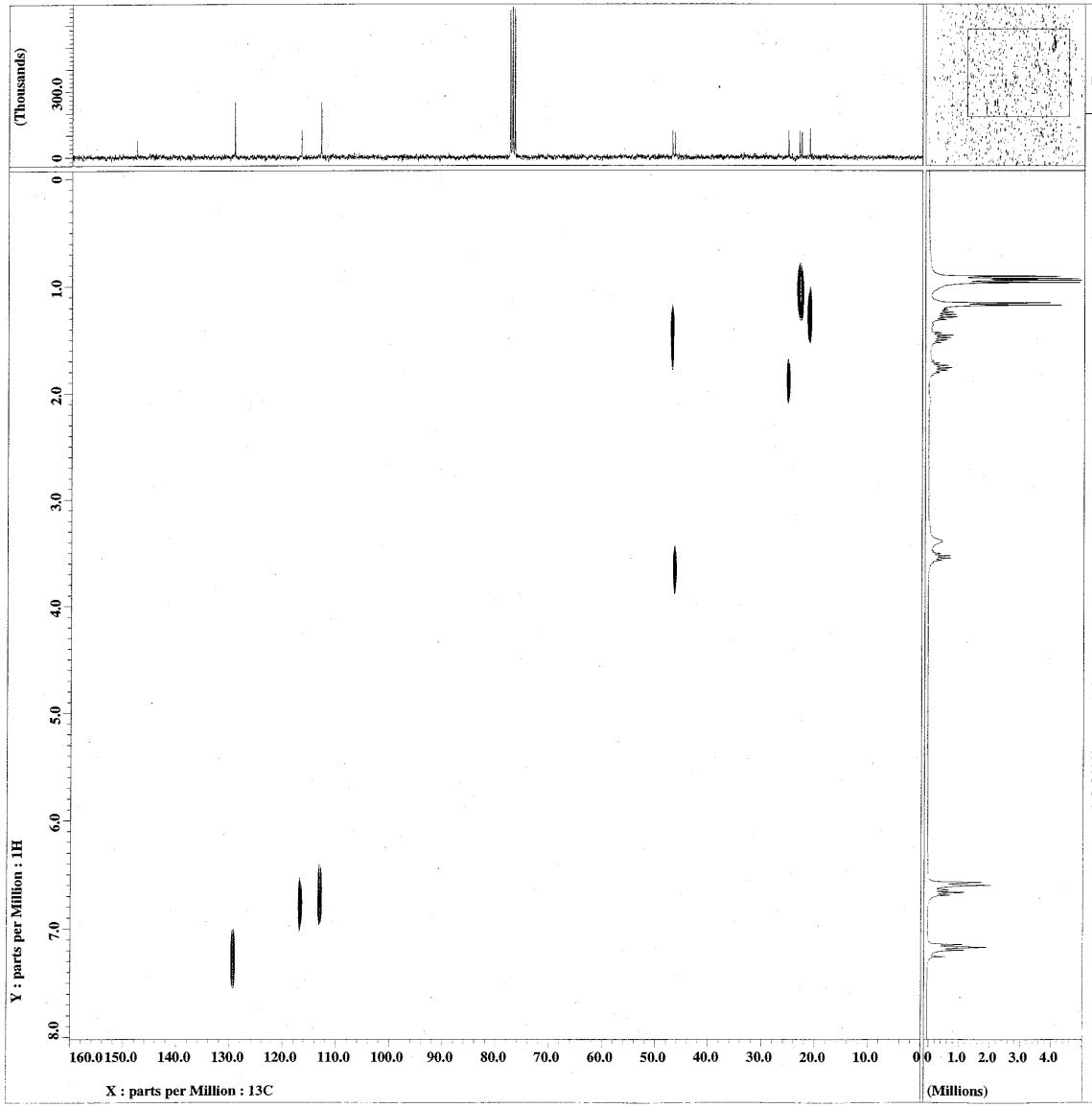
4l
(Table 2, entry 11)




```
Filename          = Reacc405F19-13C-3.jdf
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     =  7-MAR-2008 19:42:00
Revision_time     =  1-SEP-2008 13:55:19
Current_time      =  1-SEP-2008 13:55:35

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1008
Total_scans       = 1008

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 18[dC]
Unblank_time      = 2[us]
```

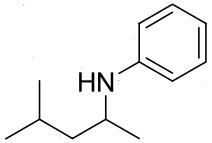


4l
(Table 2, entry 11)



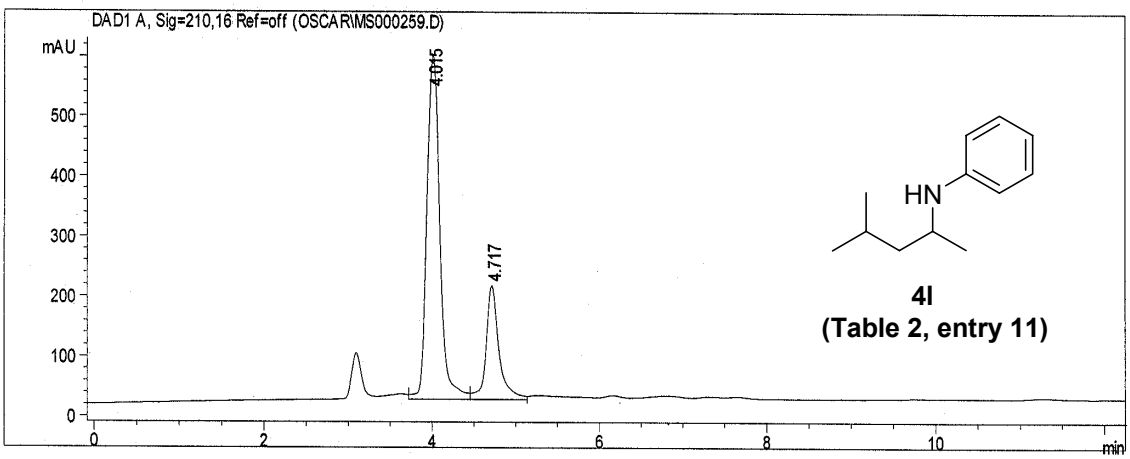
JEOL

```
Filename          = Reacc405F19-chshf-2.j
Author            = Cabrera
Experiment        = chshf.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     =  8-MAR-2008 21:59:17
Revision_time     = 20-MAY-2008 15:42:16
Current_time      = 20-MAY-2008 15:44:49

Comment           = Heteronuclear Shift C
Data_format       = 2D REAL REAL
Dim_size          = 1024, 152
Dim_title         = 13C 1H
Dim_units         = [ppm] [ppm]
Dimensions        = X Y
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 54.1696[ms]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 1024
X_prescans        = 4
X_resolution      = 18.46053875[Hz]
X_sweep           = 18.90359168[kHz]
Y_domain          = 1H
Y_freq            = 300.52965592[MHz]
Y_offset          = 5[ppm]
Y_points          = 76
Y_prescans        = 0
Y_resolution      = 59.32324047[Hz]
Y_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 14
Total_scans       = 1064

X_acq_time        = 54.1696[ms]
X_pulse           = 11.3[us]
Y_acq_time        = 28.3904[ms]
Y_pulse           = 37[us]
Initial_wait      = 1[s]
J_constant        = 140[Hz]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1.5[s]
T1                = 1[us]
Temp_get          = 15.5[dC]
Unblank_time      = 2[us]
```



4l
(Table 2, entry 11)

Reacc405
080619-coa-07



Data File C:\HPCHEM\1\DATA\OSCAR\MS000259.D Sample Name: Reacc405
HPLC IQ 20/06/08 4:18:56 PM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

==========

Injection Date : 20/06/08 3:57:57 PM
Sample Name : Reacc405 Vial : 1
Acq. Operator : carmen
Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 20/06/08 3:40:09 PM by carmen
(modified after loading)
para Le legadec

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

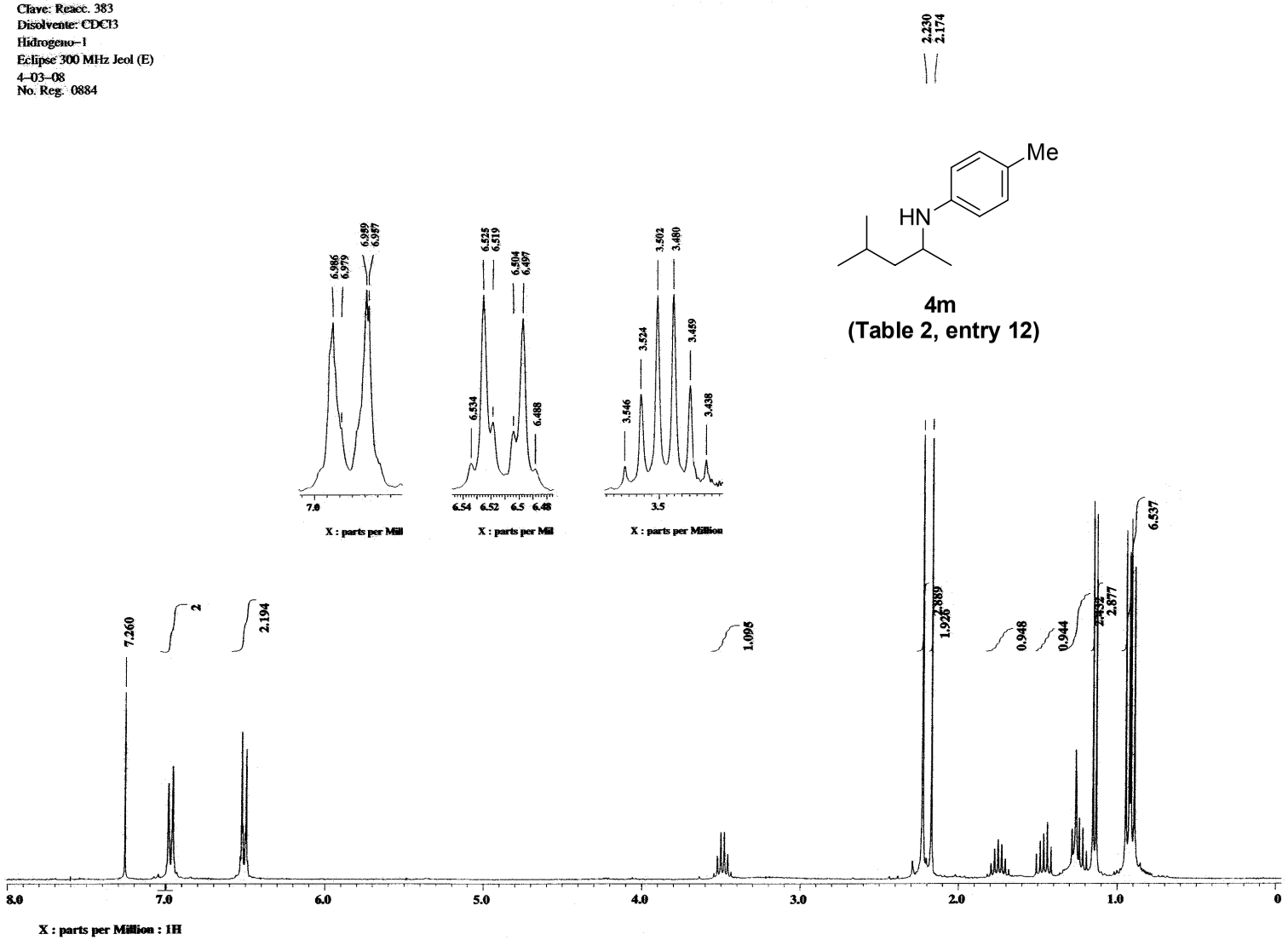
Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.015	VV	0.1700	6197.39795	574.00317	75.3456
2	4.717	VV	0.1564	2027.89673	190.14859	24.6544
Totals :				8225.29468	764.15176	

ee = 50.69%

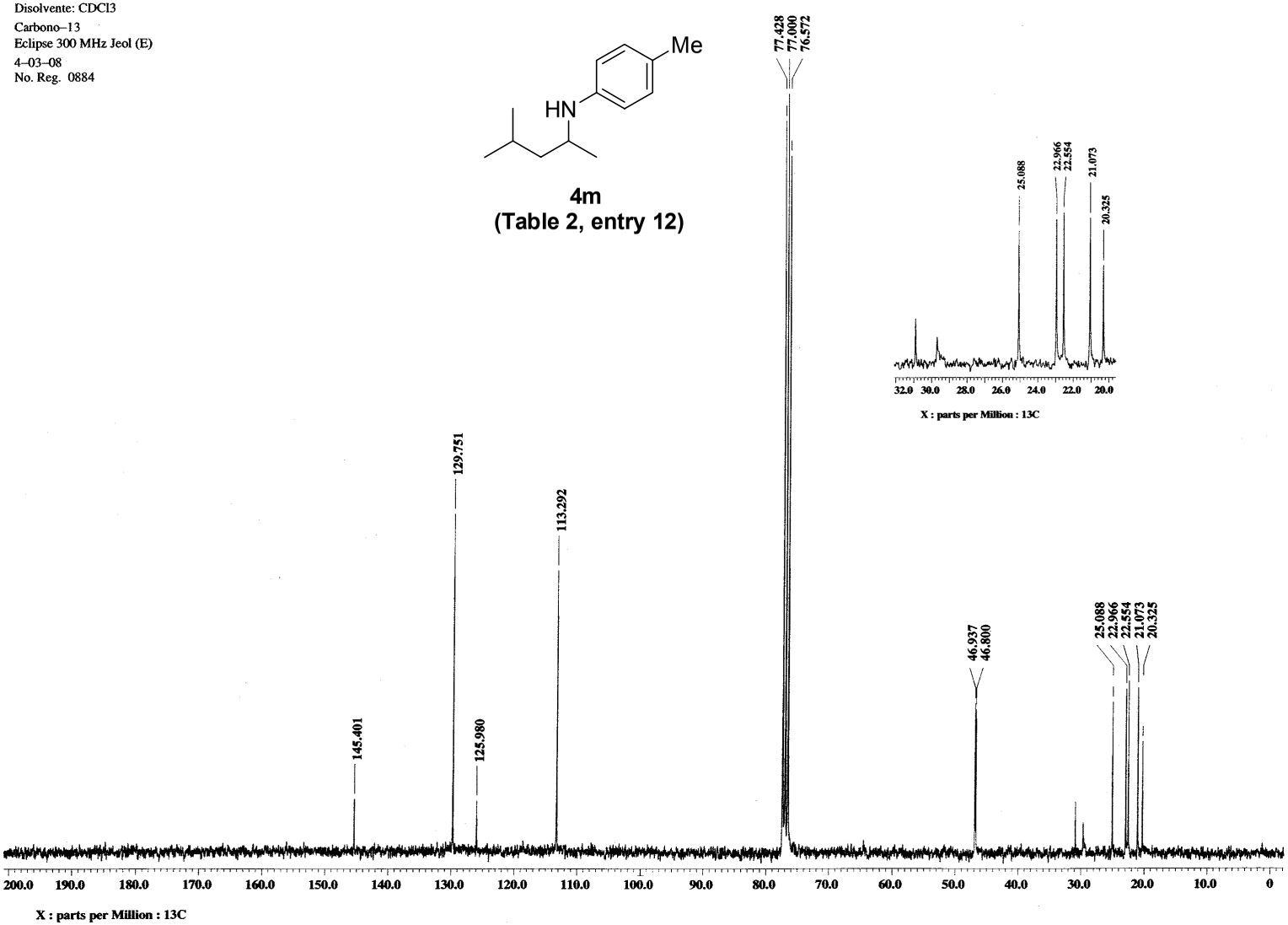
UNAM, INSTITUTO DE QUIMICA, apg
Dr. Armando Carbrera / Laura R. P.
Clave: Reacc. 383
Disolvente: CDCl3
Hidrogeno-1
Eclipse 300 MHz Jeol (E)
4-03-08
No. Reg. 0884

4m
(Table 2, entry 12)



UNAM, INSTITUTO DE QUIMICA, apg
Dr. Armando Cabrera / Laura R. P.
Clave: Reacc. 383
Disolvente: CDCl3
Carbono–13
Eclipse 300 MHz Jeol (E)
4–03–08
No. Reg. 0884

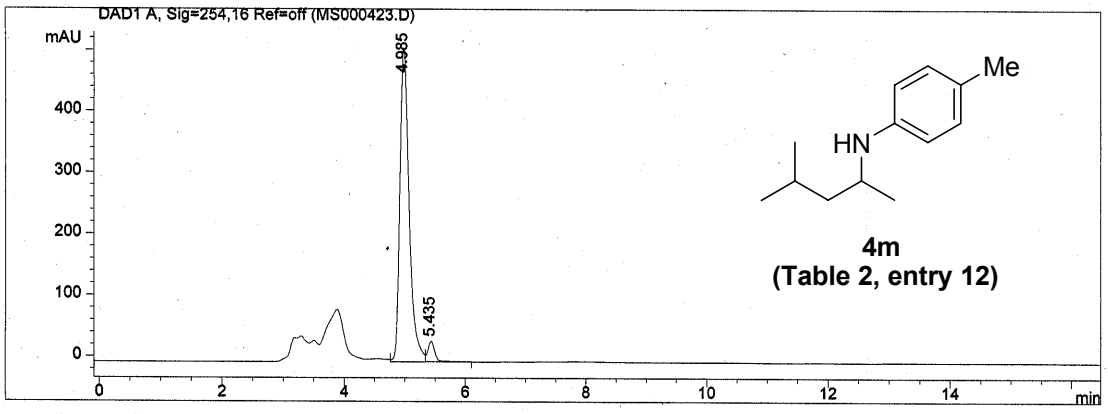
4m
(Table 2, entry 12)



Data File C:\HPCHEM\1\DATA\MS000423.D Sample Name: Reacc 432F24
080825-coa-06

Chiralcel OD 100 5 250x 4.6 mm
hexano/isopropanol 95/5
flujo 1 ml/min
UV 254 nm

==========
Injection Date : 11/09/08 1:34:42 PM
Sample Name : Reacc 432F24 Vial : 1
Acq. Operator : 428
Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 11/09/08 12:35:40 PM by 428
(modified after loading)
para Le legadec
==========



==========
Area Percent Report
==========

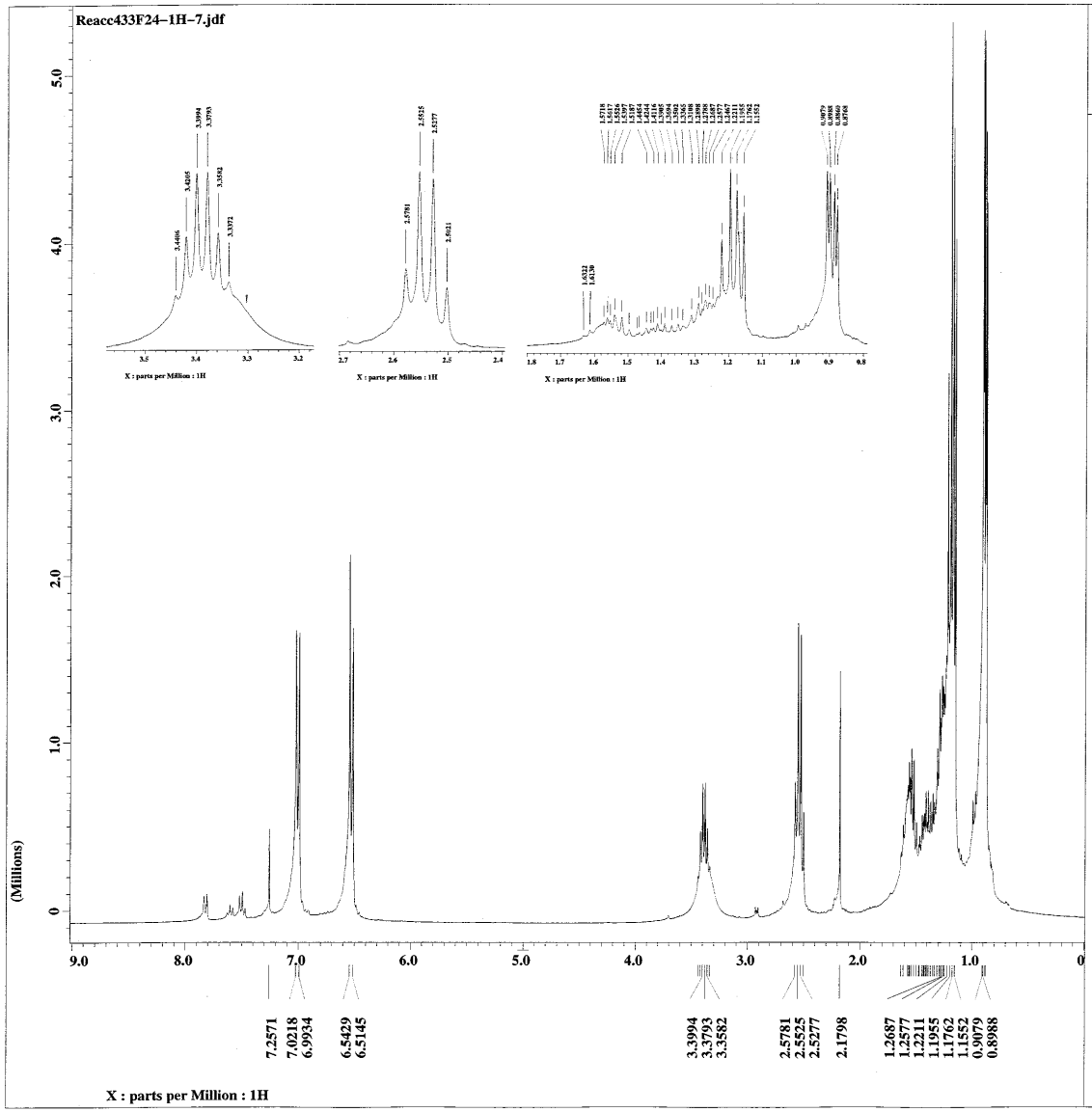
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.985	VV	0.1598	5433.41309	511.96649	95.1338
2	5.435	VB	0.1191	277.92603	34.10902	4.8662

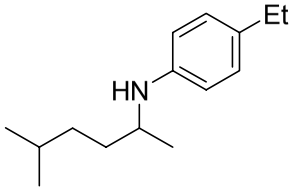
Totals : 5711.33911 546.07551

==========
*** End of Report ***

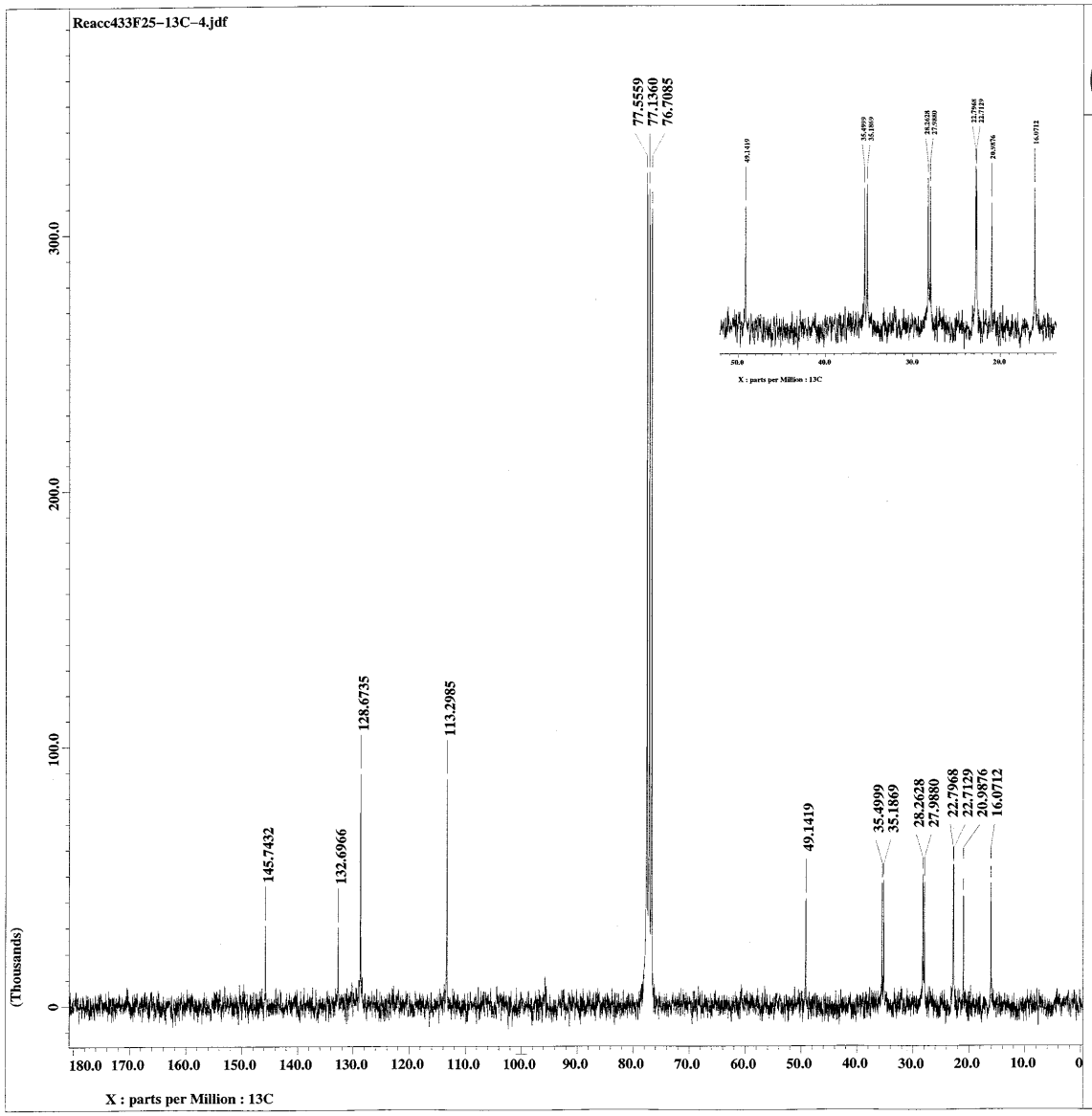


JEOL

```
Filename          = Reacc433F24-1H-7.jdf
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 11-JUN-2008 09:15:09
Revision_time     =  1-SEP-2008 14:18:01
Current_time      =  1-SEP-2008 14:18:34

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 64
Total_scans       = 64

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 18
Relaxation_delay  = 1[s]
Temp_get          = 19.5[dC]
Unblank_time      = 2[us]
```

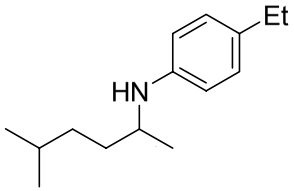


4n
(Table 2, entry 13)



JEOL

```
Filename          = Reacc433F25-13C-4.jdf
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 11-JUN-2008 09:09:17
Revision_time     =  1-SEP-2008 14:14:01
Current_time      =  1-SEP-2008 14:14:38

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 5000
Total_scans       = 5000

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 20.4[dC]
Unblank_time      = 2[us]
```

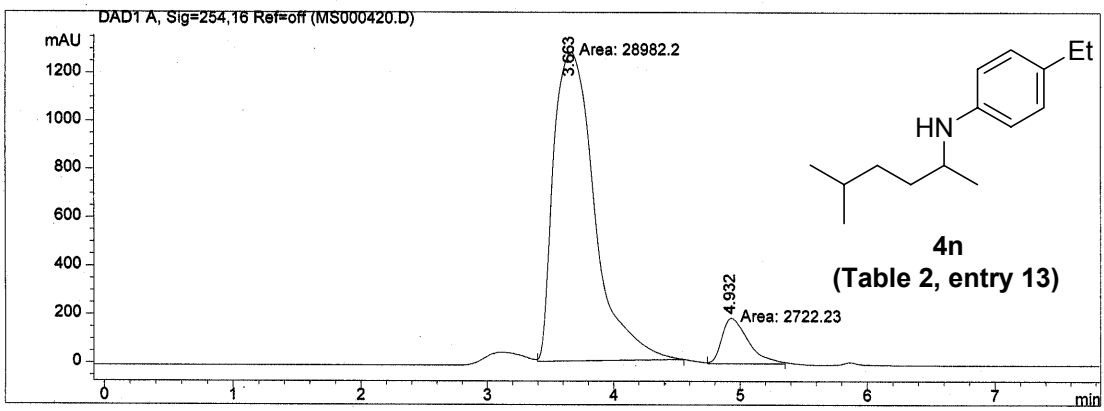


4n
(Table 2, entry 13)

Data File C:\HPCHEM\1\DATA\MS000420.D Sample Name: Reacc 433f25
080825-coa-04

Chiralcel OD 100 5 250x 4.6 mm
hexano/isopropanol 95/5
flujo 1 ml/min
UV 254 nm

==========
Injection Date : 11/09/08 12:02:30 PM
Sample Name : Reacc 433f25 Vial : 1
Acq. Operator : 428
Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 11/09/08 11:09:31 AM by carmen
(modified after loading)
para Le legadec
==========



==========
Area Percent Report
==========

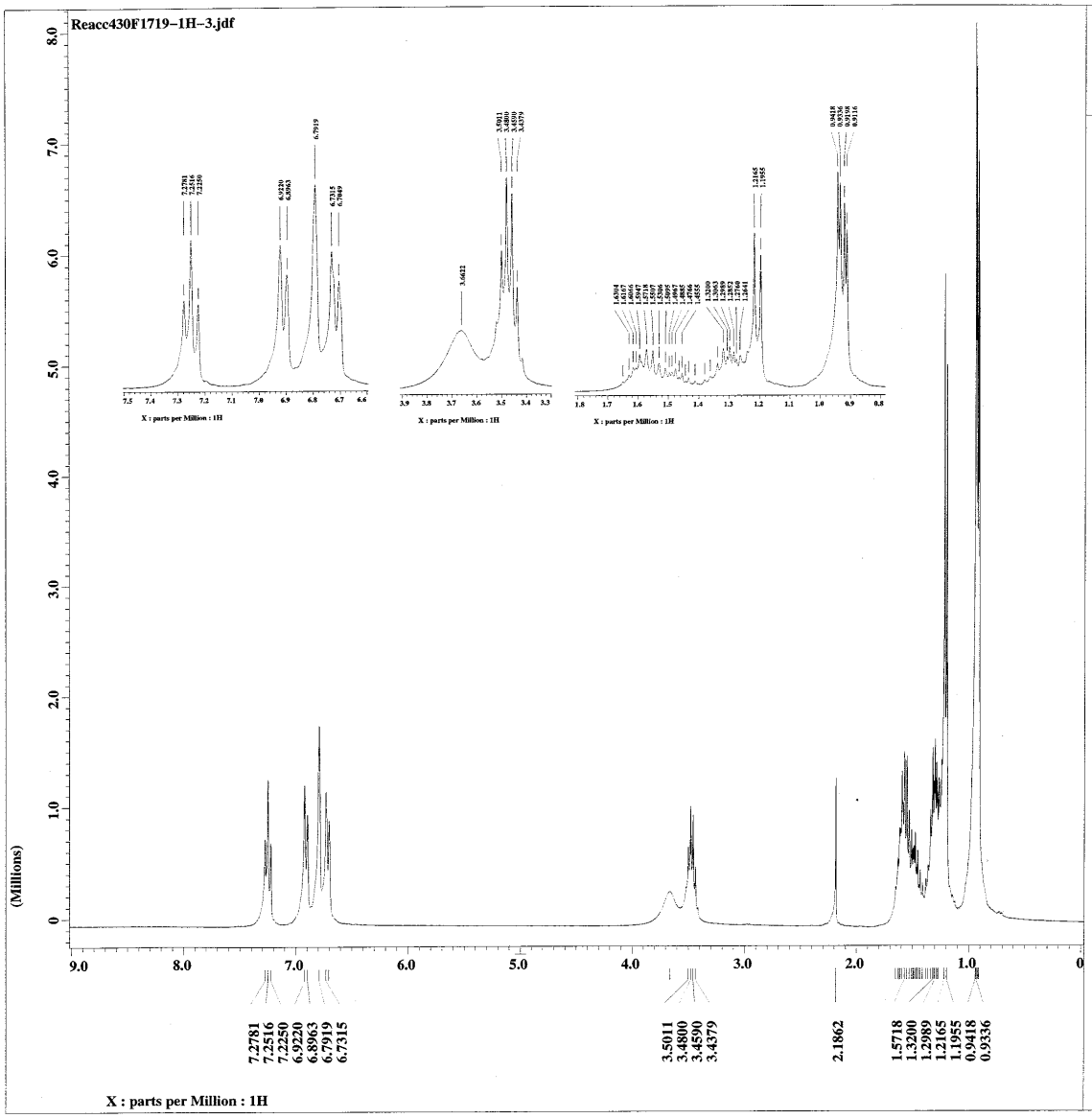
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=254,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.663	MM	0.3768	2.89822e4	1281.97351	91.4137
2	4.932	MM	0.2406	2722.23267	188.55191	8.5863

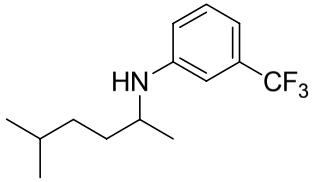
Totals : 3.17044e4 1470.52542

==========
*** End of Report ***



JEOL

Filename	= Reacc430F1719-1H-3.jd
Author	= Cabrera
Experiment	= single_pulse.exp
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 21-JUN-2008 23:07:13
Revision_time	= 3-SEP-2008 07:25:33
Current_time	= 3-SEP-2008 07:30:39
Comment	= Single Pulse Experime
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 32
Total_scans	= 32
X_90_width	= 9.5[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 4.75[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 19
Relaxation_delay	= 1[s]
Temp_get	= 19.2[dC]
Unblank_time	= 2[us]



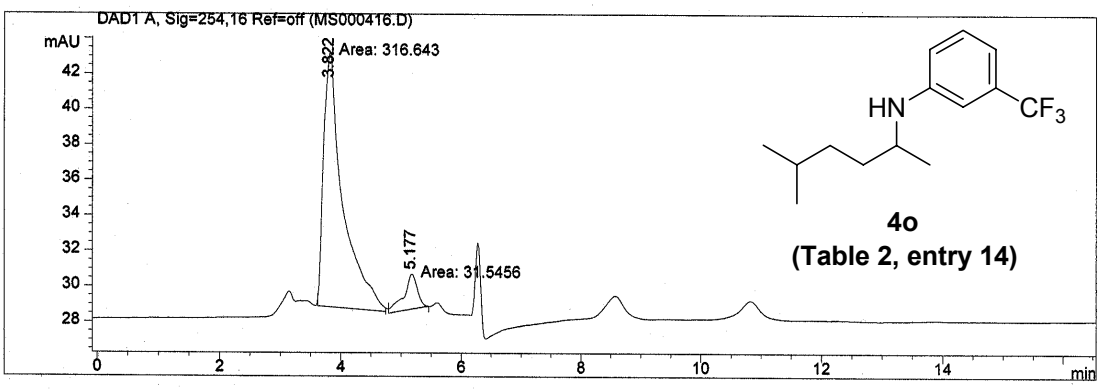
4o

(Table 2, entry 14)

Data File C:\HPCHEM\1\DATA\MS000416.D Sample Name: Reacc 430
080825-coa-05

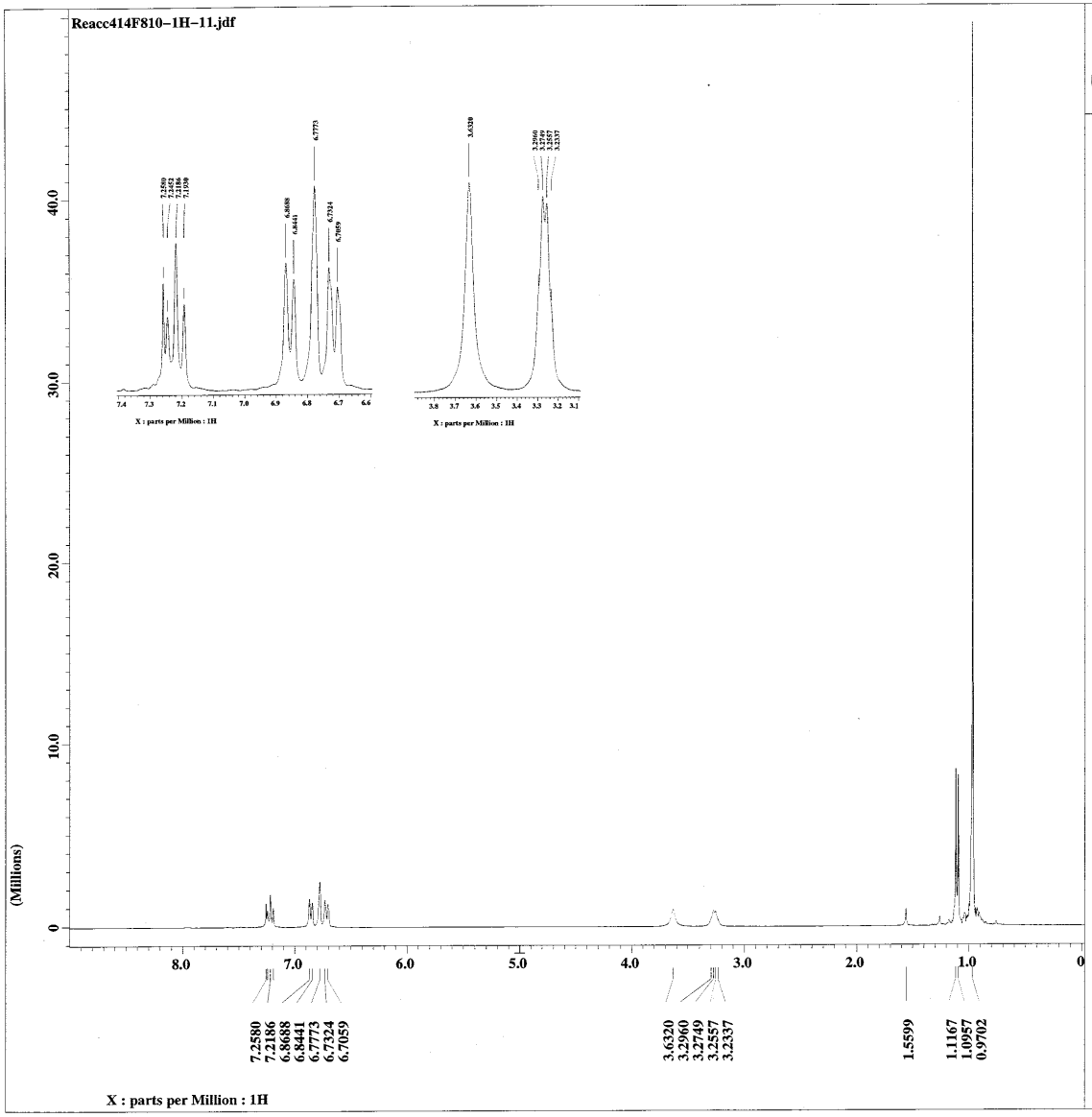
Chiralcel OD 100 5 250x 4.6 mm
hexano/isopropanol 90/10
flujo 1 ml/min
UV 254 nm

```
==========================================================================
Injection Date  : 08/09/08 1:01:11 PM
Sample Name     : Reacc 430                          Vial :   1
Acq. Operator   : carmen
Acq. Method     : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed    : 08/09/08 10:22:21 AM by carmen
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed    : 12/09/08 12:43:24 PM by 428
                  (modified after loading)
para Le legadec
==========================================================================
```



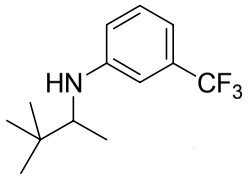

```
==========================================================================
                              Area Percent Report
==========================================================================

Sorted By              :      Signal
Multiplier             :      1.0000
Dilution               :      1.0000

Signal 1: DAD1 A, Sig=254,16 Ref=off
 Results obtained with enhanced integrator!
```

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.773	Fsho	0.0000	0.00000	12.74802	0.0000
2	3.822	MM	0.3657	316.64301	14.43213	90.9401
3	4.224	Rsho	0.0000	0.00000	3.26390	0.0000
4	4.455	Rsho	0.0000	0.00000	1.68889	0.0000
5	5.013	Fsho	0.0000	0.00000	5.51193e-1	0.0000
6	5.177	MM	0.2628	31.54560	2.00050	9.0599

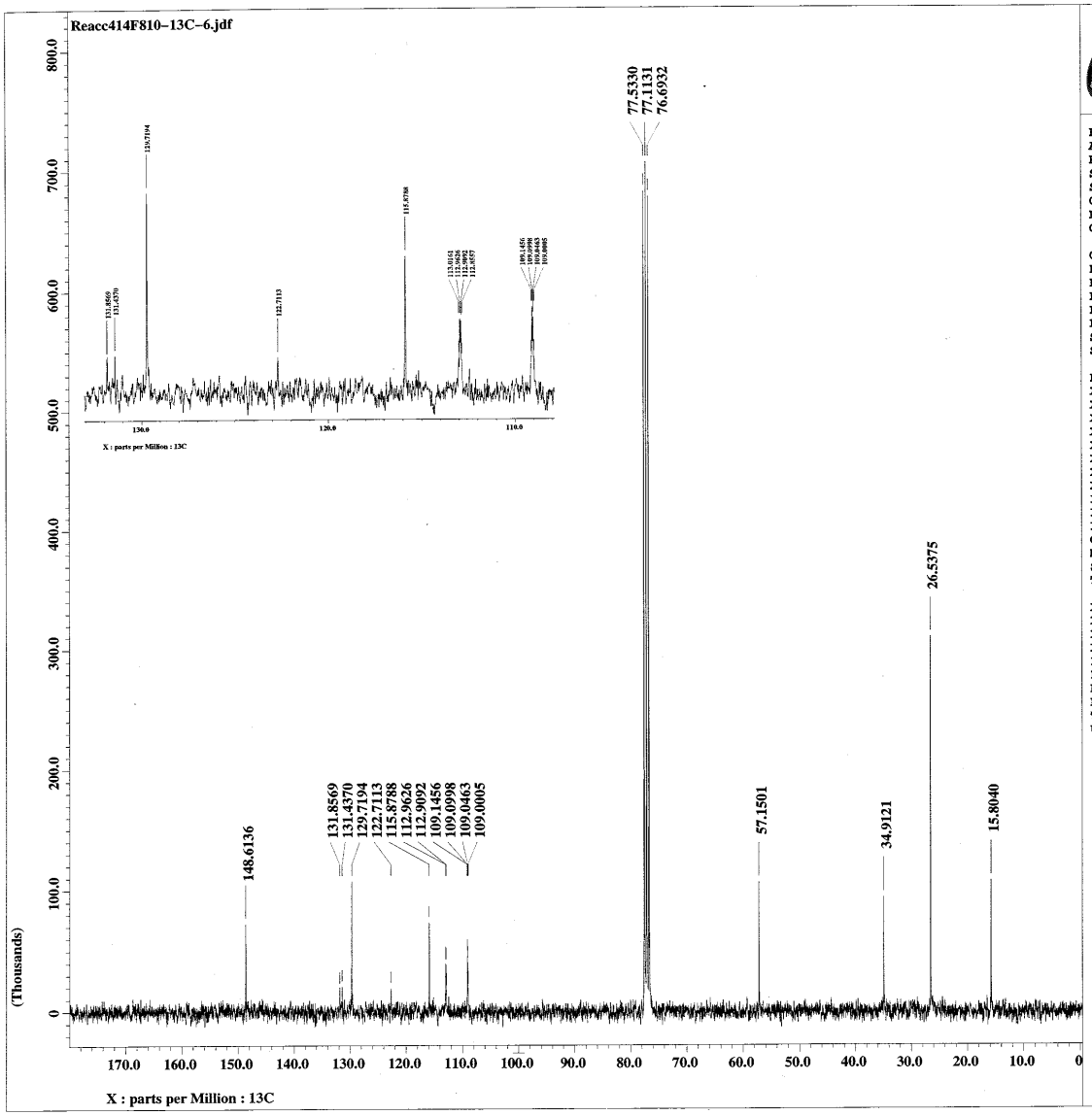


JEOL

```
Filename          = Reacc414F810-1H-11.jd
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 24-MAR-2008 14:16:34
Revision_time     =  2-SEP-2008 07:32:07
Current_time      =  2-SEP-2008 07:33:31

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 18
Relaxation_delay  = 1[s]
Temp_get          = 18[dC]
Unblank_time      = 2[us]
```

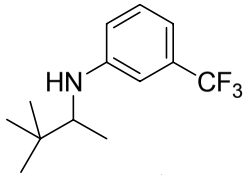


4p
(Table 2, entry 15)



JEOL

```
Filename          = Reacc414F810-13C-6.jd
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 24-MAR-2008 16:07:01
Revision_time     =  2-SEP-2008 07:29:50
Current_time      =  2-SEP-2008 07:30:43

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 2375
Total_scans       = 2375

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 19.9[dC]
Unblank_time      = 2[us]
```

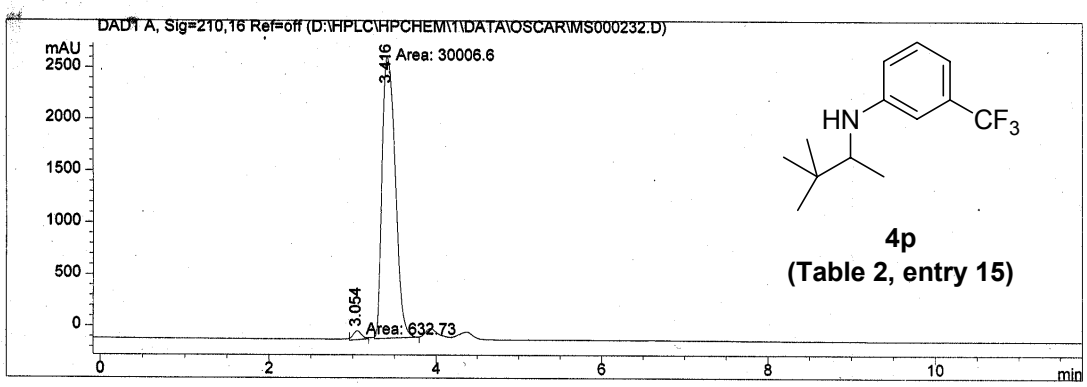


4p
(Table 2, entry 15)

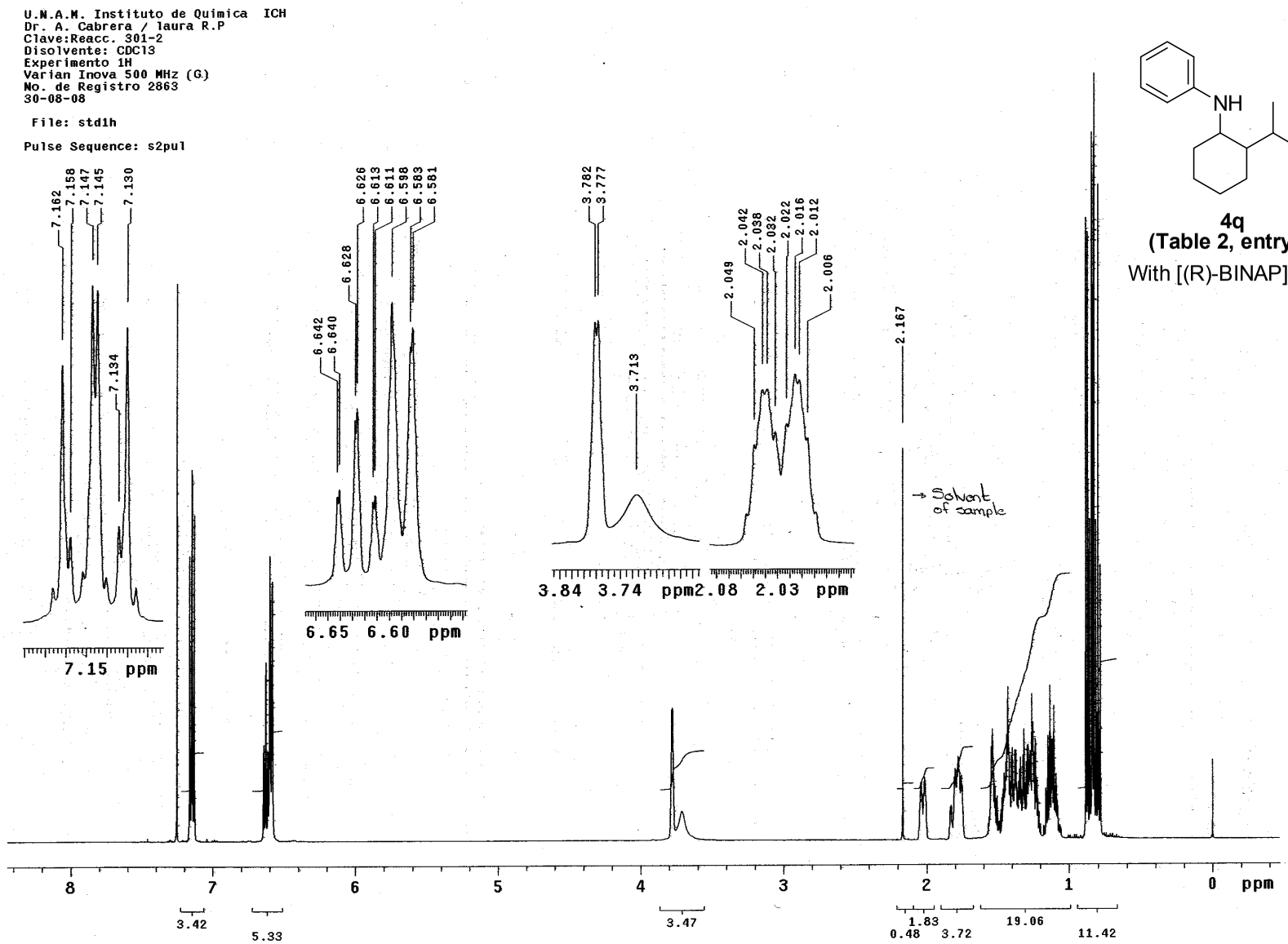
Data File D:\HPLC\HPCHEM\1\DATA\OSCAR\MS000232.D Sample Name: Reacc414F812

Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

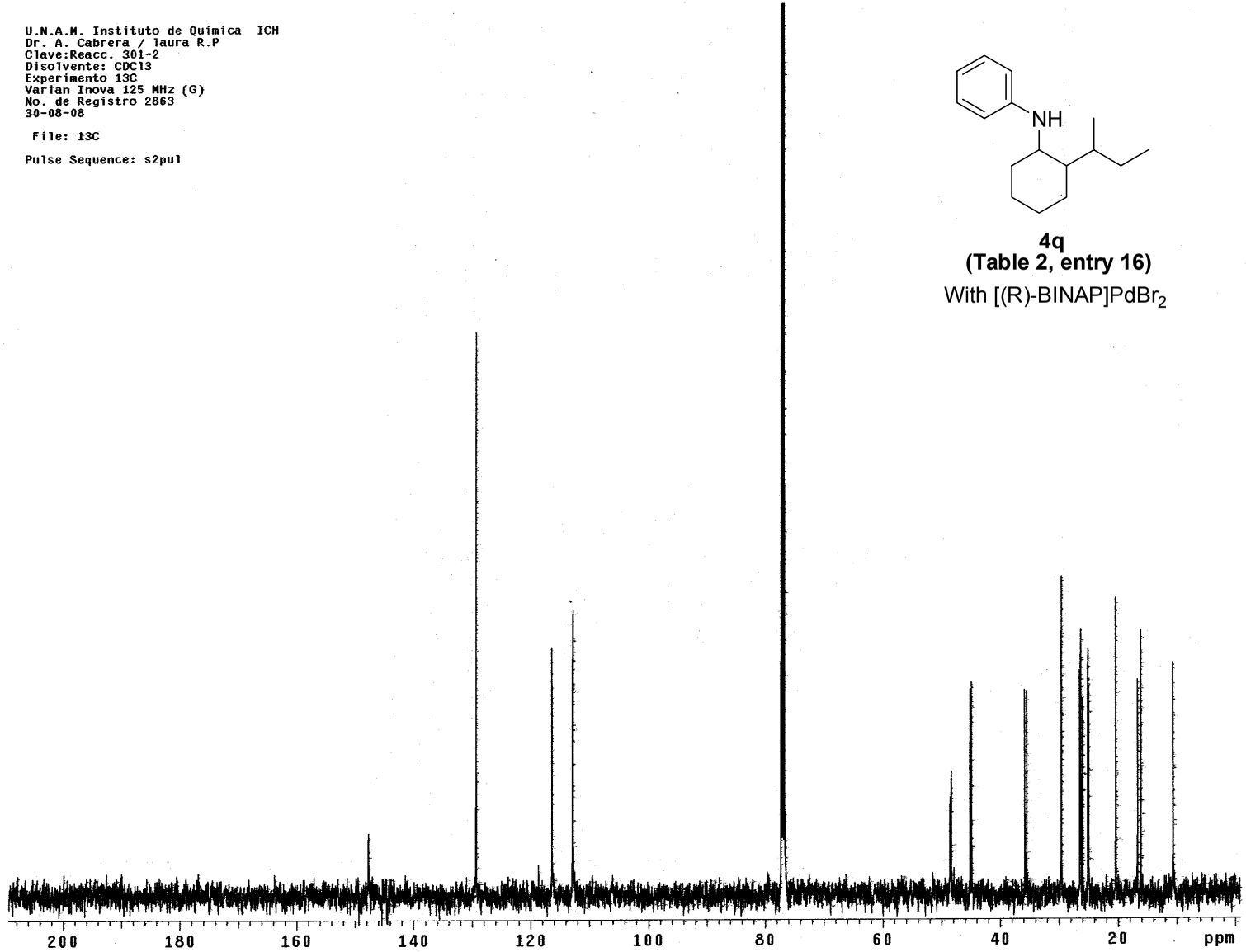
```
==========================================================================
Injection Date  : 17/06/08 10:15:40 AM
Sample Name     : Reacc414F812                          Vial :   1
Acq. Operator   : carmen
Acq. Method     : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed    : 17/06/08 9:44:12 AM by carmen
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed    : 12/09/08 12:43:24 PM by 428
                  (modified after loading)
para Le legadec
==========================================================================
```




```
==========================================================================
                              Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: DAD1 A, Sig=210,16 Ref=off
 Results obtained with enhanced integrator!

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   3.054 MM    0.1197  632.72961   88.13225   2.0651
   2   3.416 MM    0.1834 3.00066e4  2726.47974  97.9349

Totals :                   3.06393e4  2814.61198

==========================================================================
                              *** End of Report ***
```



4q
(Table 2, entry 16)
With [(R)-BINAP]$PdBr_2$

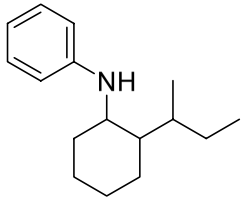


4q
(Table 2, entry 16)
With [(R)-BINAP]$PdBr_2$

U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera / laura R.P
Clave:Reacc. 301-2
Disolvente: CDCl3
Experimento 13C
Varian Inova 125 MHz (G)
No. de Registro 2863
30-08-08

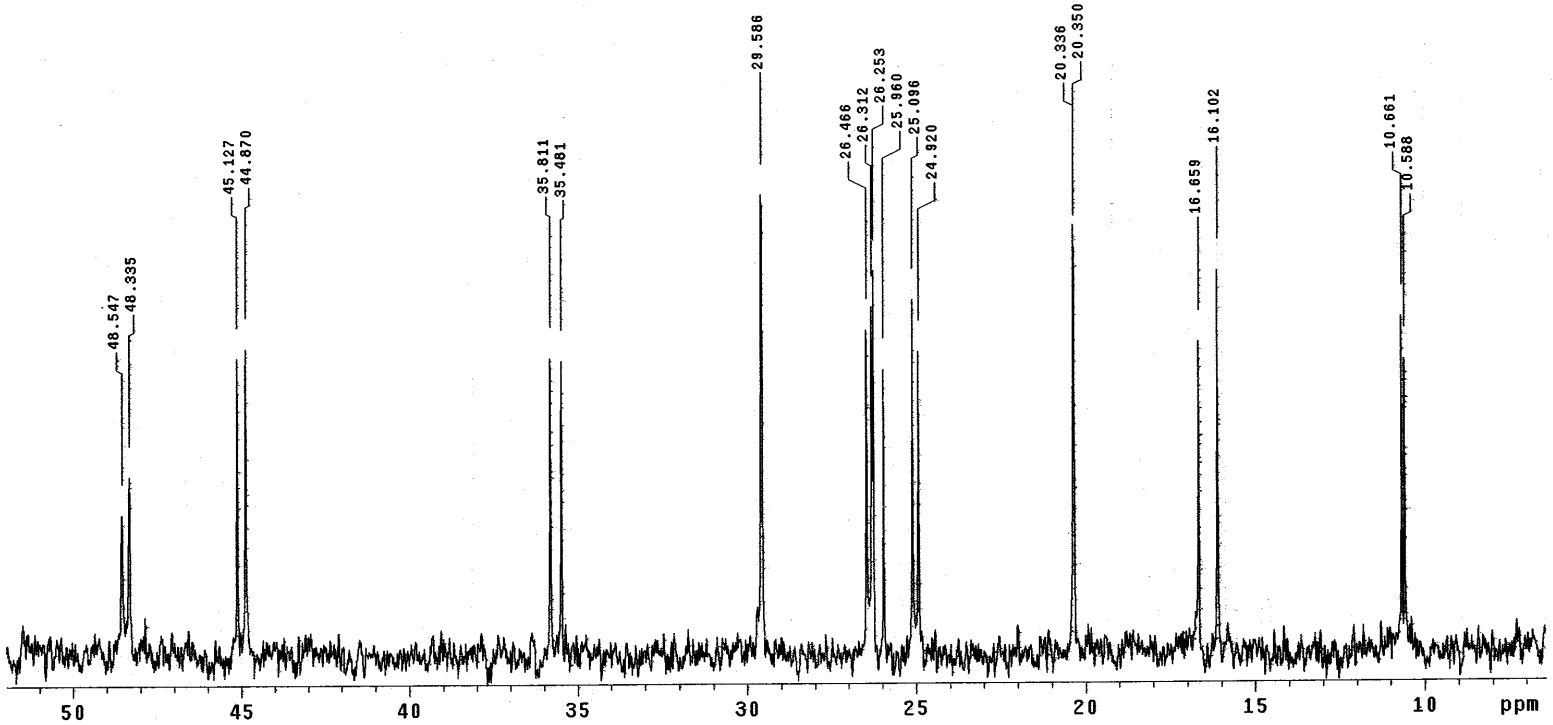
File: 13C

Pulse Sequence: s2pul



4q
(Table 2, entry 16)
With [(R)-BINAP]$PdBr_2$

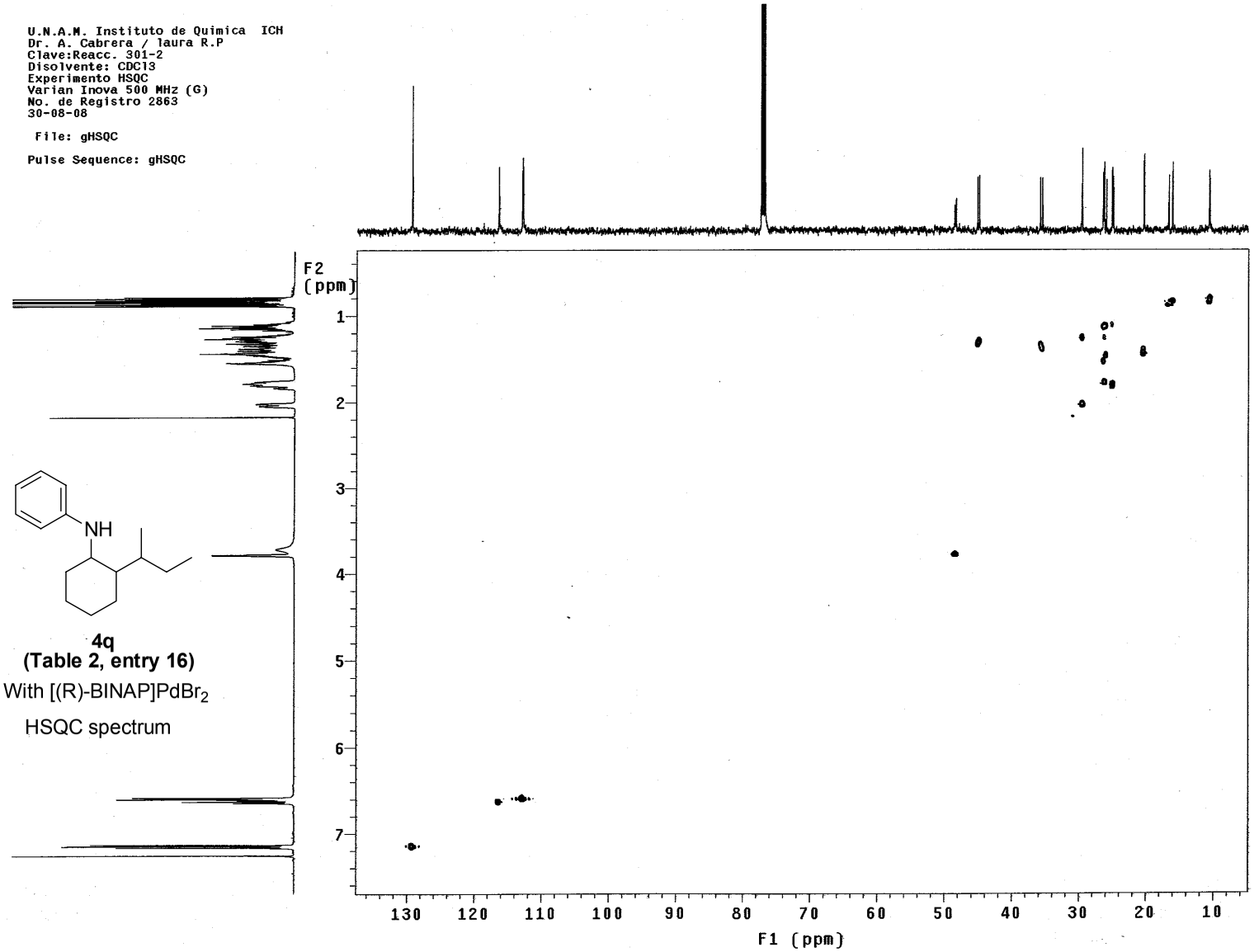
Expansions of ^{13}C NMR



U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera / laura R.P
Clave:Reacc. 301-2
Disolvente: CDCl3
Experimento HSQC
Varian Inova 500 MHz (G)
No. de Registro 2863
30-08-08

File: gHSQC

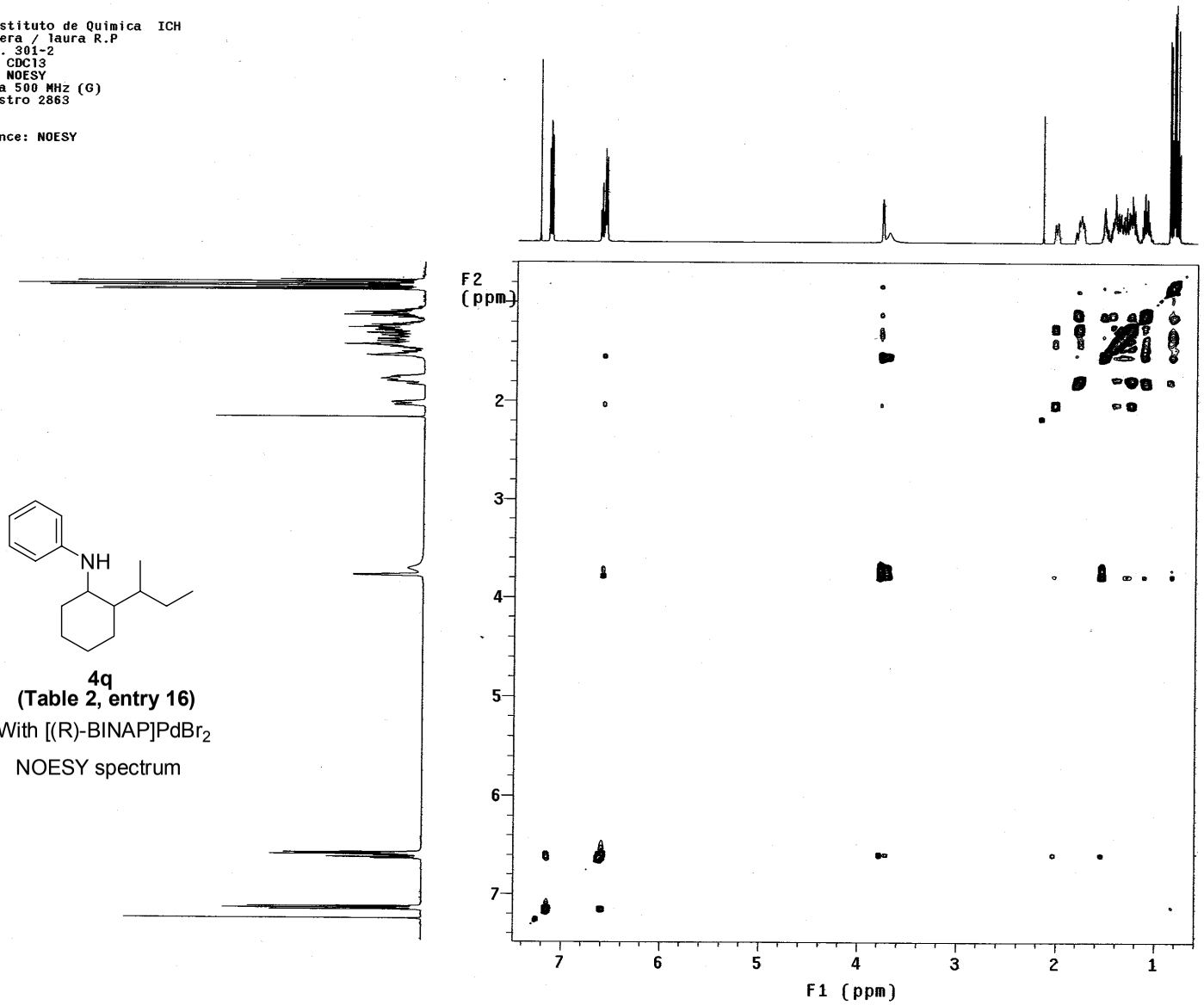
Pulse Sequence: gHSQC



4q
(Table 2, entry 16)
With [(R)-BINAP]$PdBr_2$
HSQC spectrum

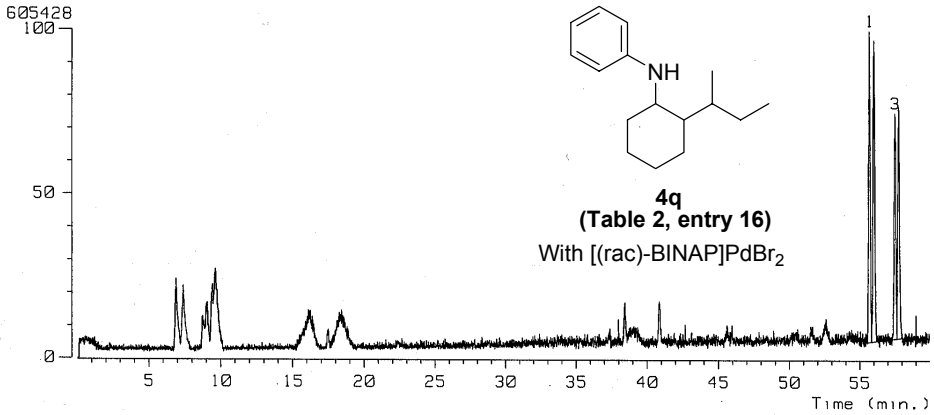
U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera / laura R.P
Clave:Reacc. 301-2
Disolvente: CDCl3
Experimento NOESY
Varian Inova 500 MHz (G)
No. de Registro 2863
30-08-08

Pulse Sequence: NOESY



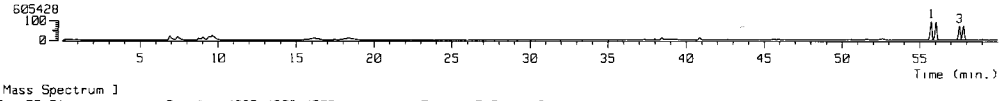
4q
(Table 2, entry 16)
With [(R)-BINAP]$PdBr_2$
NOESY spectrum

[TIC]
Data : Dr-Cabrera-Armando-091 Date : 06-Aug-120 09:34
Sample: 1742 G Reacc 306 JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 10 to 650

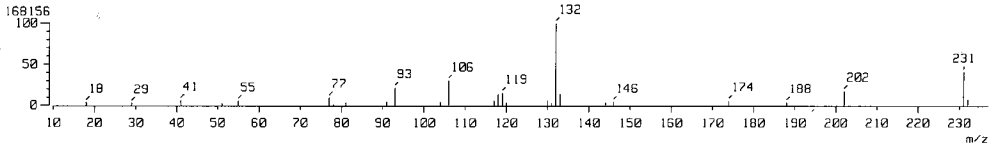


No.	RT[min]	Area	Area%	Height	Height%	Width[sec]	INTEG
1	55.71	436.93	27.30	54.64	29.08	7.51	BV
2	56.05	457.54	28.59	52.97	28.19	8.11	VV
3	57.53	346.95	21.68	39.64	21.09	8.22	VV
4	57.79	358.96	22.43	40.68	21.64	8.29	VB

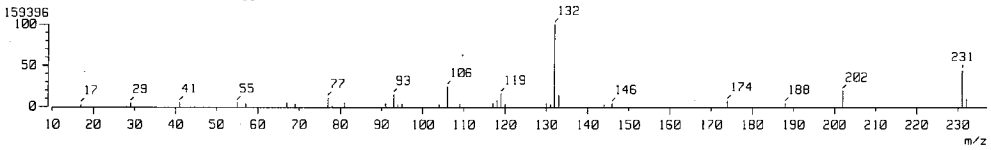
[TIC]
Data : Dr-Cabrera-Armando-091 Date : 06-Aug-120 09:34
Sample: 1742 G Reacc 306 JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 10 to 650 Output RT Range : 0.00 to 59.99 min



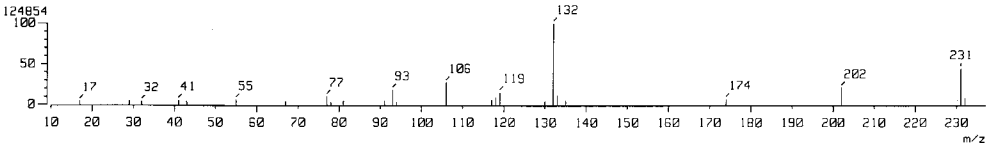
[Mass Spectrum]
RT : 55.71 min Scan# : 4325-4309-4505 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 16.04



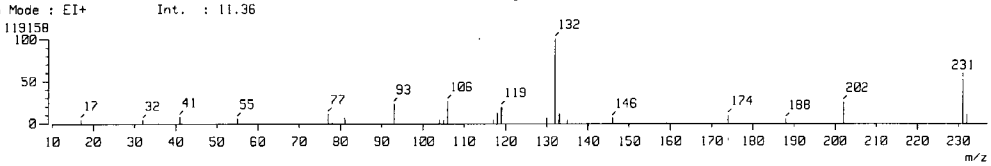
[Mass Spectrum]
RT : 56.05 min Scan# : 4351-4309-4505 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 15.20



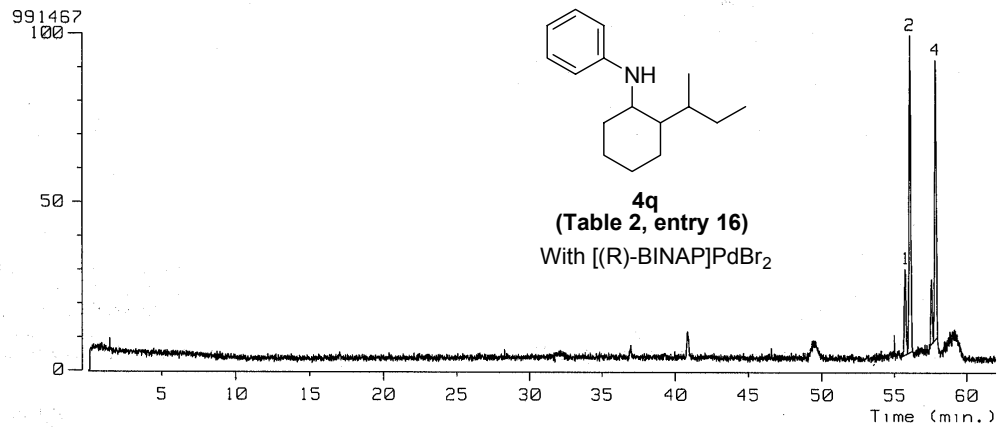
[Mass Spectrum]
RT : 57.53 min Scan# : 4466-4309-4505 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 11.91



[Mass Spectrum]
RT : 57.79 min Scan# : 4486-4309-4505 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 11.36

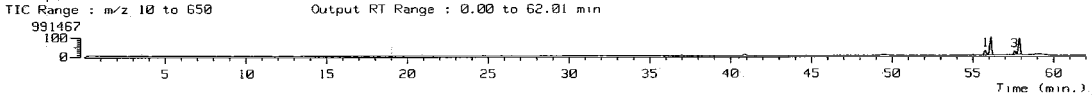


[TIC]
Data : Dr-Cabrera-Armando-095 Date : 07-Aug-120 11:43
Sample: 1747 G Reacc 301 JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 10 to 650

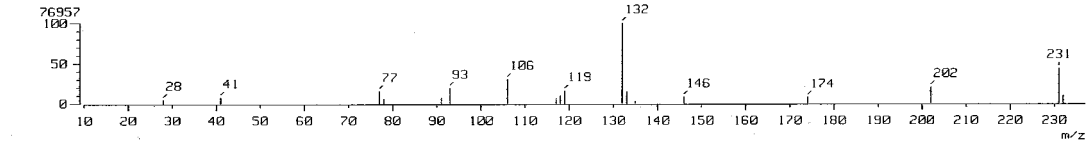


No.	RT[min]	Area	Area%	Height	Height%	Width[sec]	INTEG
1	55.75	218.04	13.06	24.56	11.67	8.34	BV
2	56.10	714.49	42.79	89.17	42.38	7.52	VB
3	57.57	126.39	7.57	18.18	8.64	6.53	BV
4	57.86	611.02	36.59	78.51	37.31	7.31	VB

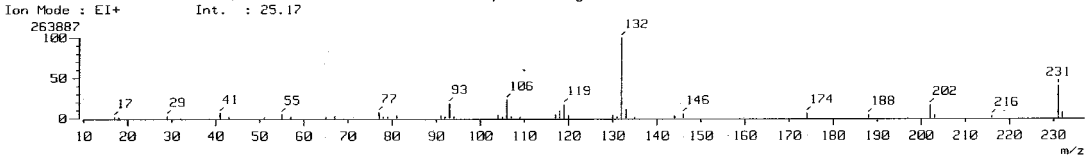
[TIC]
Data : Dr-Cabrera-Armando-095 Date : 07-Aug-120 11:43
Sample: 1747 G Reacc 301 JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 10 to 650 Output RT Range : 0.00 to 62.01 min



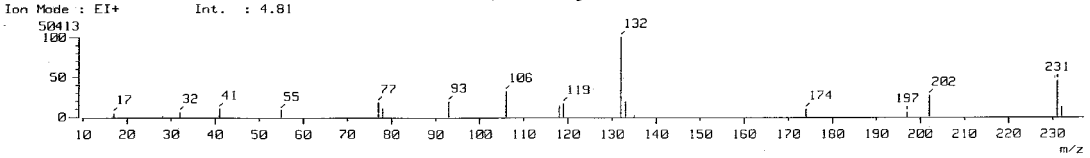
[Mass Spectrum]
RT : 55.75 min Scan# : 4328-4299-4367 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 7.34



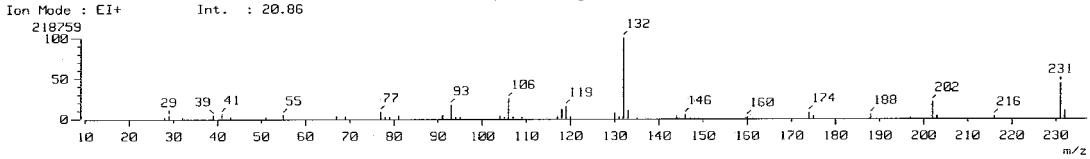
[Mass Spectrum]
RT : 56.10 min Scan# : 4355-4299-4367 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 25.17



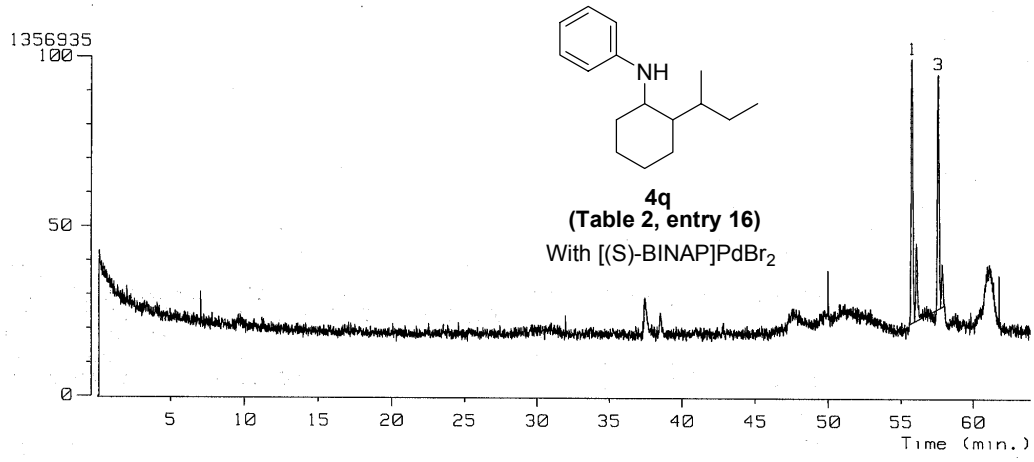
[Mass Spectrum]
RT : 57.57 min Scan# : 4469-4461-4502 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 4.81



[Mass Spectrum]
RT : 57.86 min Scan# : 4492-4461-4502 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 20.86

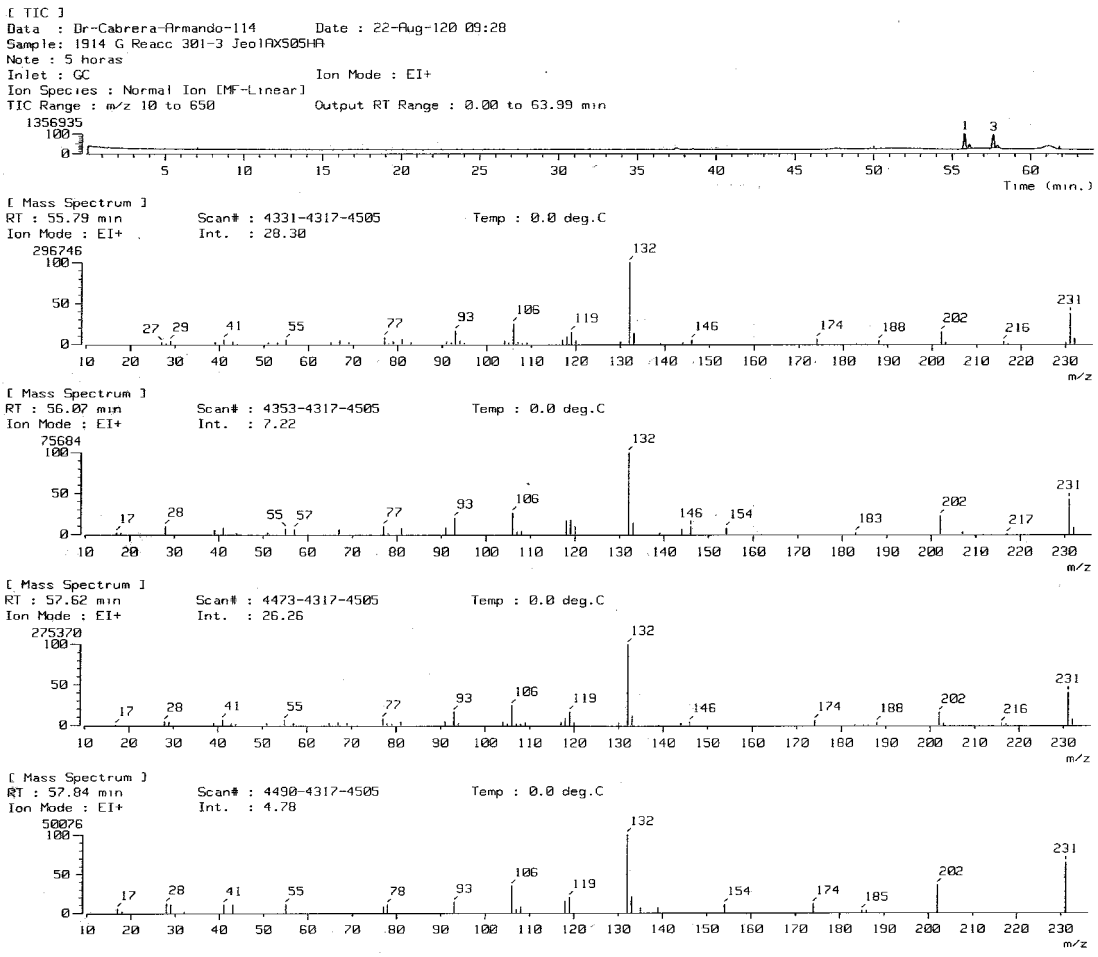


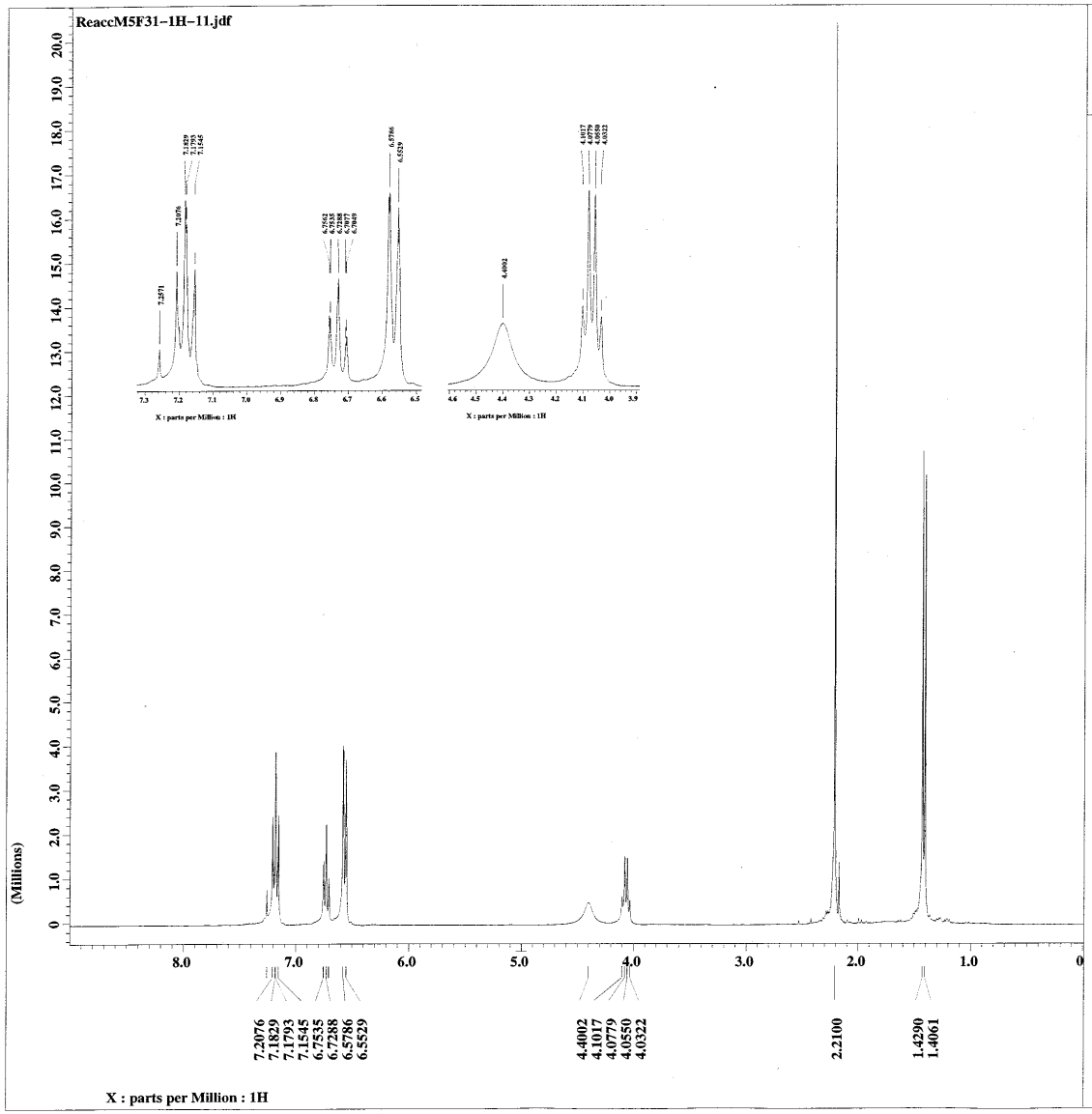
[TIC]
Data : Dr-Cabrera-Armando-114 Date : 22-Aug-120 09:28
Sample: 1914 G Reacc 301-3 JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 10 to 650



4q
(Table 2, entry 16)
With [(S)-BINAP]$PdBr_2$

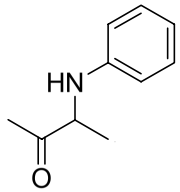
No.	RT[min]	Area	Area%	Height	Height%	Width[sec]	INTEG
1	55.79	854.25	44.10	100.62	42.65	7.97	BV
2	56.07	234.92	12.13	29.48	12.49	7.48	VV
3	57.62	716.21	36.97	89.52	37.94	7.51	VV
4	57.84	131.84	6.81	16.32	6.92	7.59	VB



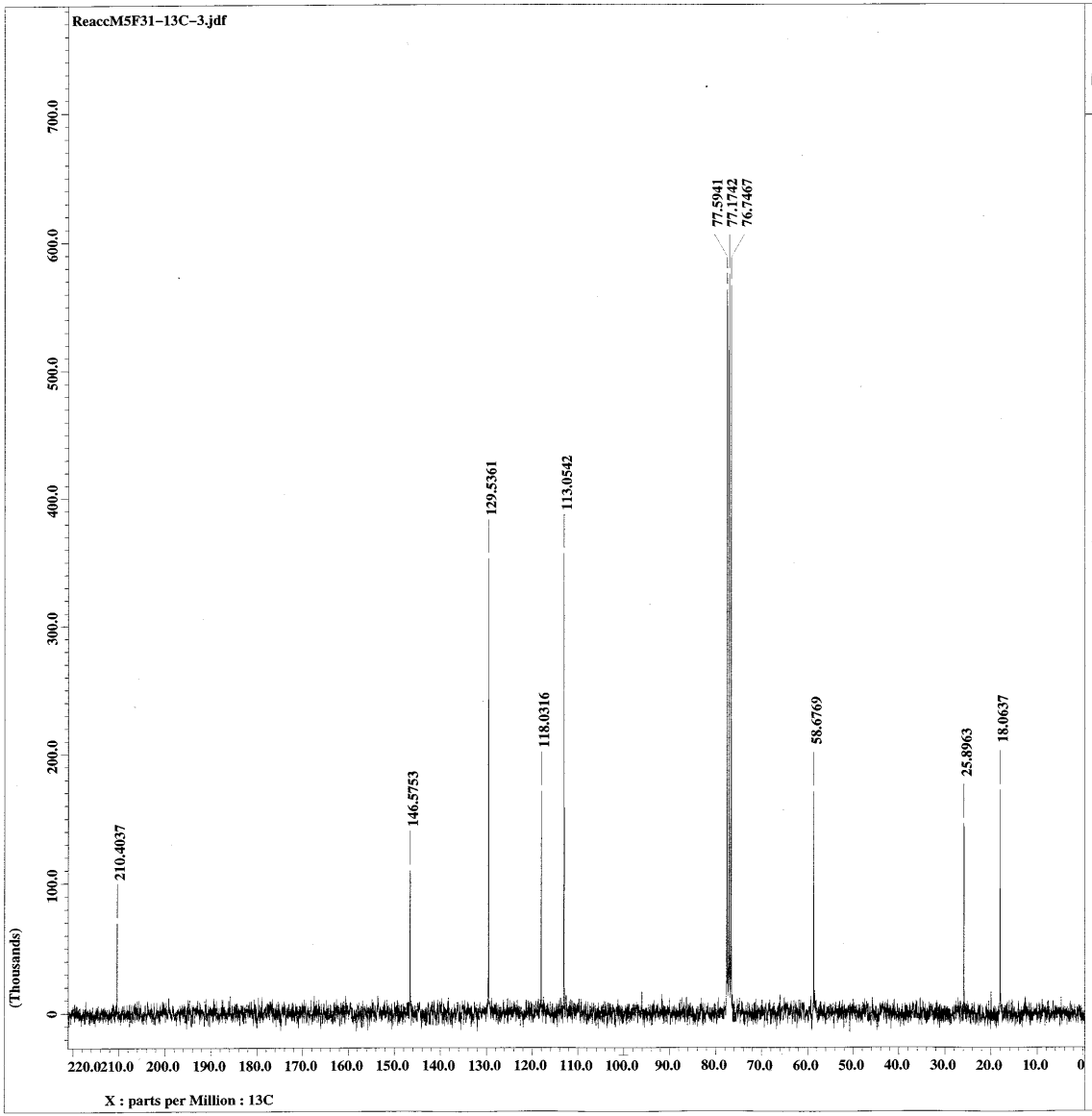


JEOL

```
Filename          = ReaccM5F31-1H-11.jdf
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     =  5-JUN-2008 22:42:16
Revision_time     =  1-SEP-2008 13:13:24
Current_time      =  1-SEP-2008 13:13:57

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 17
Relaxation_delay  = 1[s]
Temp_get          = 18.9[dC]
Unblank_time      = 2[us]
```

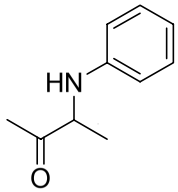


4r
(Table 2, entry 17)



JEOL

```
Filename          = ReaccM5F31-13C-3.jdf
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     =  4-JUN-2008 03:11:18
Revision_time     =  1-SEP-2008 13:15:15
Current_time      =  1-SEP-2008 13:15:24

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1725
Total_scans       = 1725

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 20.8[dC]
Unblank_time      = 2[us]
```

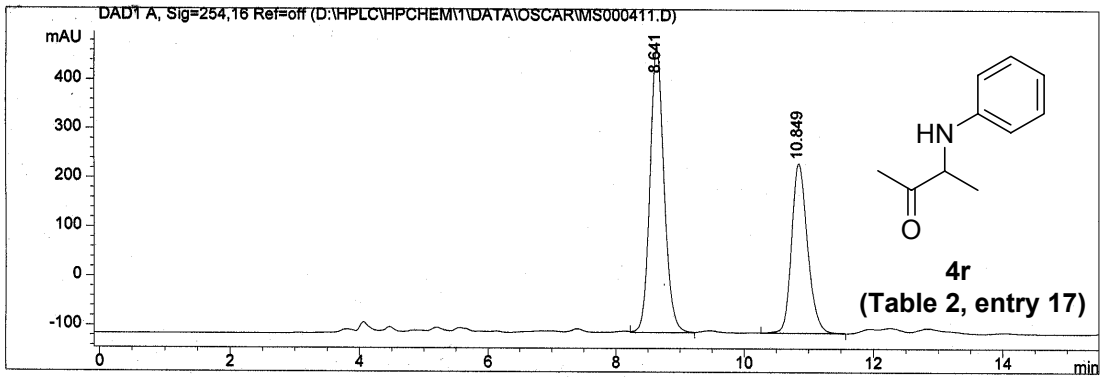


4r
(Table 2, entry 17)

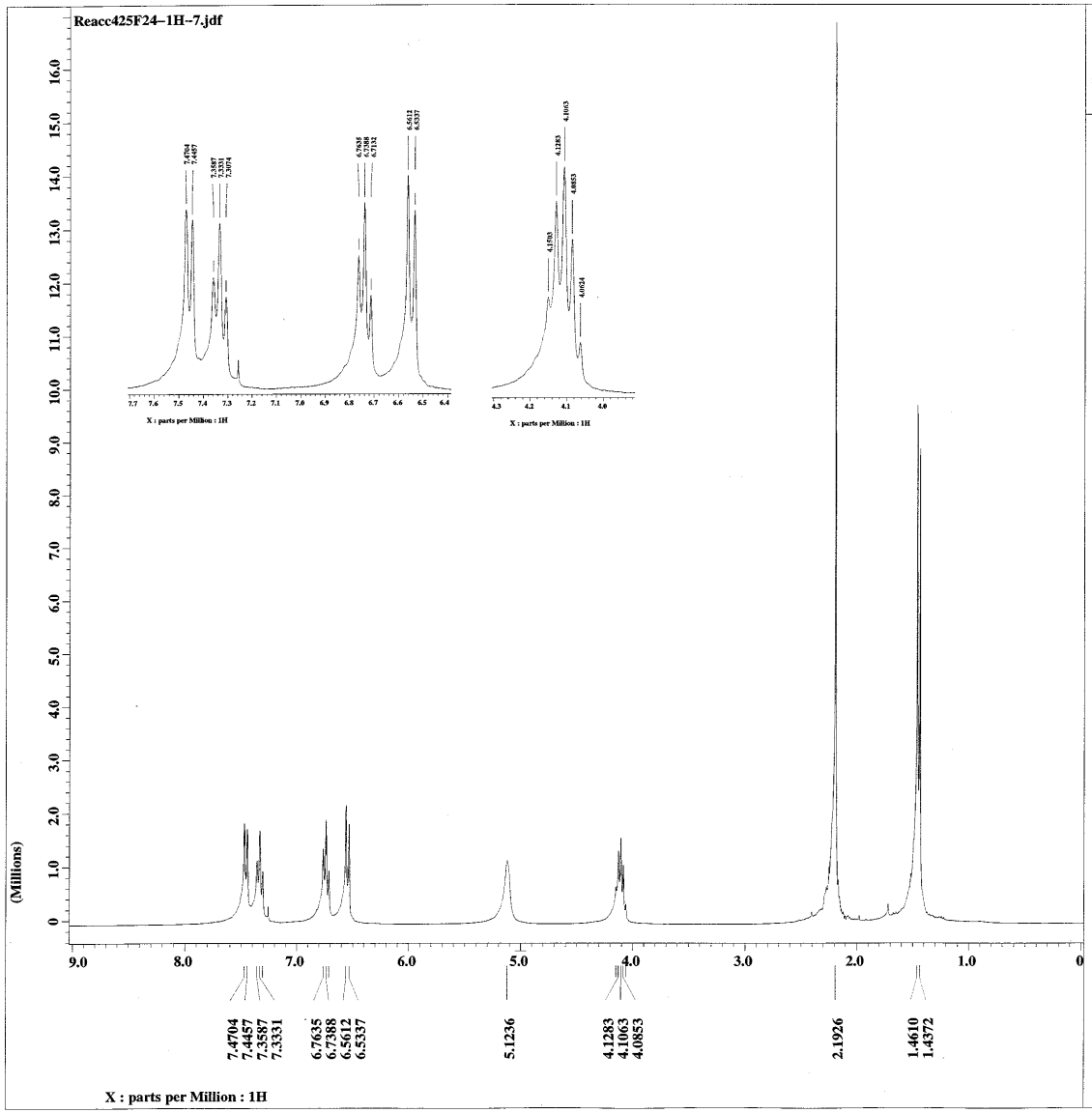
Data File D:\HPLC\HPCHEM\1\DATA\OSCAR\MS000411.D Sample Name: Reacc H5F31
080825-coa-01

Chiralcel OD 100 5 250x 4.6 mm
hexano/isopropanol 90/10
flujo 1 ml/min
UV 254 nm

```
==========================================================================
Injection Date  : 04/09/08 6:16:33 PM
Sample Name     : Reacc H5F31                          Vial :   1
Acq. Operator   : carmen
Acq. Method     : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed    : 04/09/08 5:41:33 PM by carmen
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed    : 11/09/08 11:09:31 AM by carmen
                  (modified after loading)
para Le legadec
==========================================================================
```

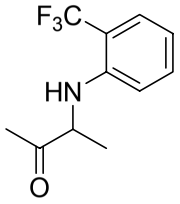



```
==========================================================================
                              Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: DAD1 A, Sig=254,16 Ref=off
 Results obtained with enhanced integrator!

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.641 VV    0.2346 8898.23145  581.41614  59.9473
   2  10.849 VP    0.2608 5945.19336  345.46396  40.0527

Totals :                  1.48434e4   926.88010      ee = 19.89 ~ 20%

==========================================================================
                           *** End of Report ***
```



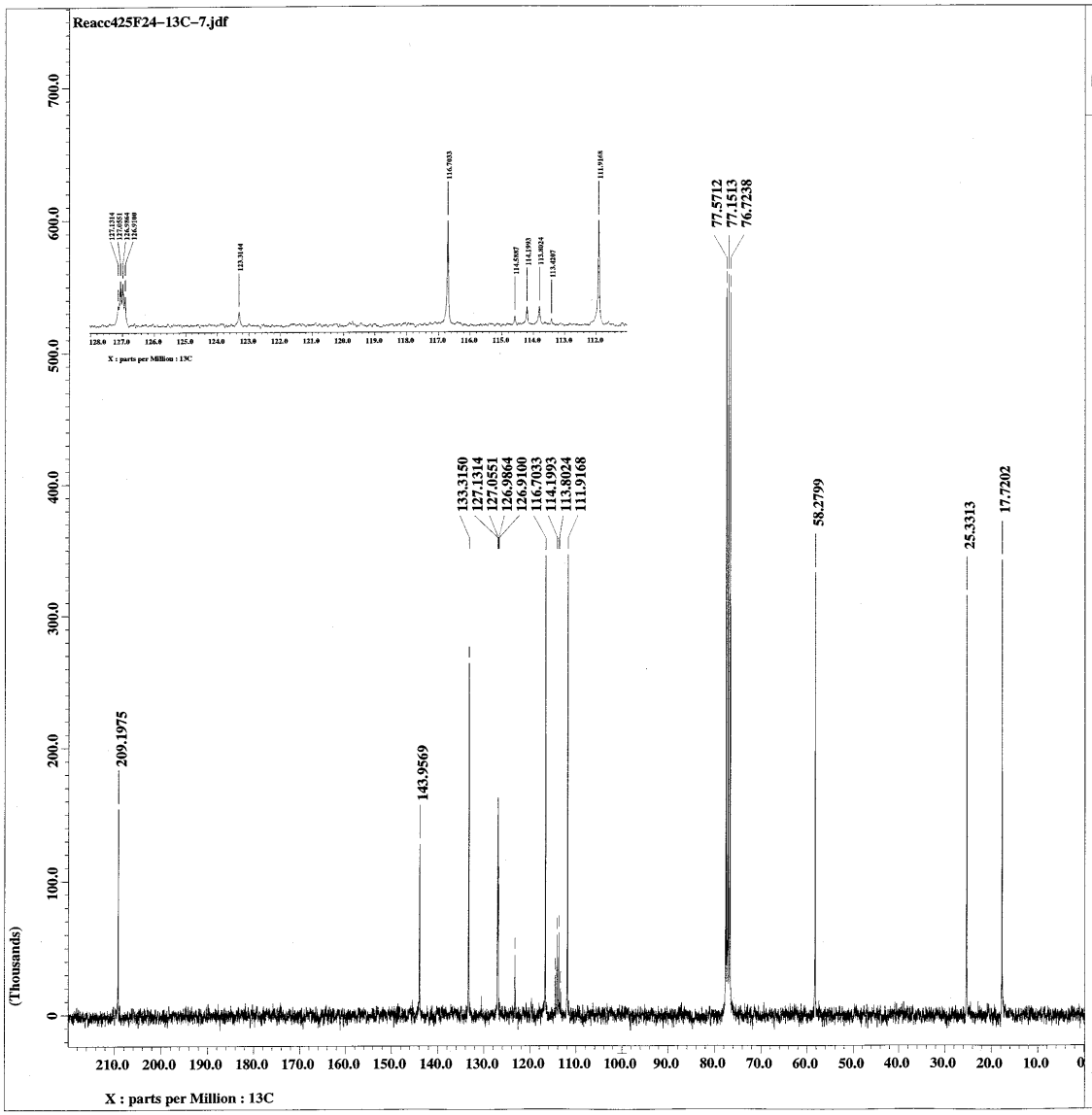
JEOL

```
Filename          = Reacc425F24-1H-7.jdf
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 15-APR-2008 06:04:36
Revision_time     =  2-SEP-2008 07:42:40
Current_time      =  2-SEP-2008 07:46:17

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 16
Relaxation_delay  = 1[s]
Temp_get          = 19.2[dC]
Unblank_time      = 2[us]
```



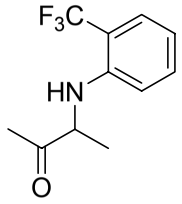
4s

Table 2, entry 18



JEOL

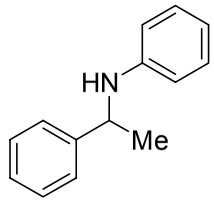
Filename	= Reacc425F24-13C-7.jdf
Author	= Cabrera
Experiment	= single_pulse_dec
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 18-APR-2008 01:39:30
Revision_time	= 3-SEP-2008 07:54:41
Current_time	= 3-SEP-2008 07:57:26
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 3000
Total_scans	= 3000
X_90_width	= 11.3[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 3.76666667[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 30
Relaxation_delay	= 1[s]
Temp_get	= 20.7[dC]
Unblank_time	= 2[us]



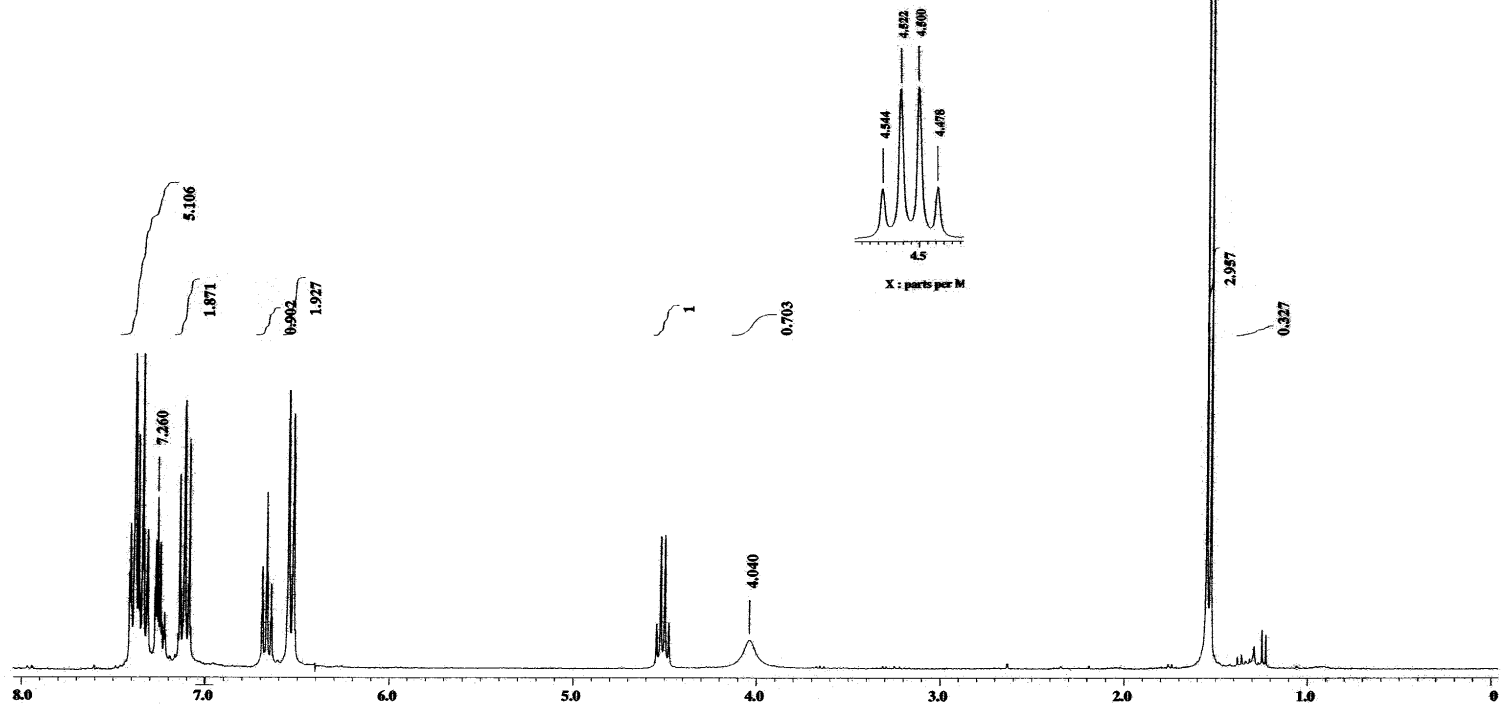
4s

Table 2, entry 18

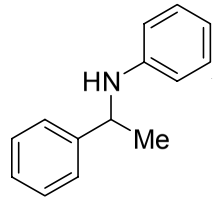
UNAM, INSTITUTO DE QUIMICA, apg
Dr. Armando Cabrera / Laura R.P.
Clave: Reacc. 332 F24
Disolvente: CDCl3
Hidrogeno-1
Eclipse 300 MHz Jeol (E)
20-02-08



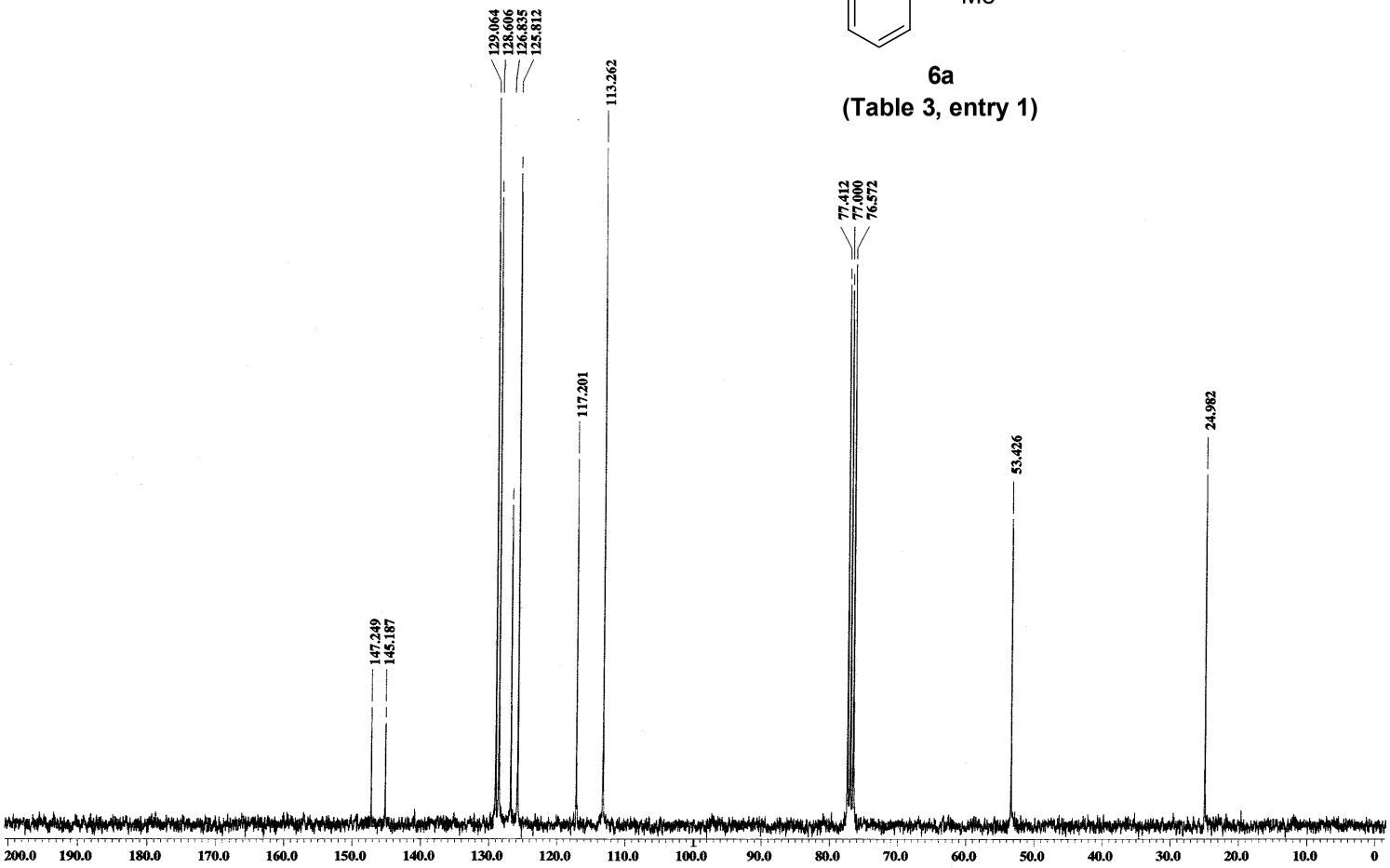
6a
(Table 3, entry 1)



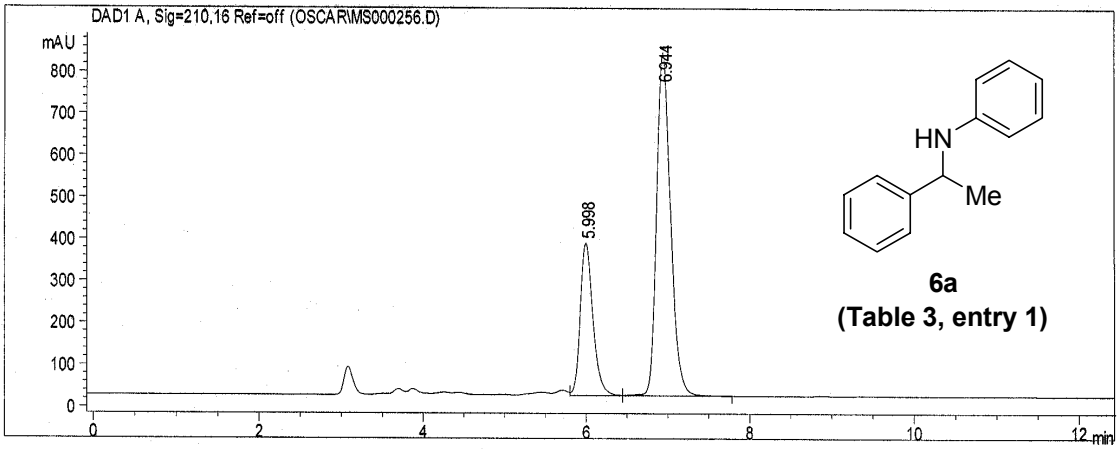
UNAM, INSTITUTO DE QUIMICA,
Dr. Armando Cabrera / Laura R.P.
Clave: Reacc. 332 F24
Disolvente: CDCl3
Carbono–13
Eclipse 300 MHz Jeol (E)
20–02–08
No. Reg. 0634



6a
(Table 3, entry 1)



Reacc332
080619-coa-04



Data File C:\HPCHEM\1\DATA\OSCAR\MS000256.D Sample Name: Reacc332
HPLC IQ 20/06/08 2:39:48 PM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

==========

Injection Date : 20/06/08 11:53:01 AM
Sample Name : Reacc332 Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 20/06/08 9:08:55 AM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 20/06/08 12:36:04 PM by carmen
(modified after loading)
para Le legadec

==========

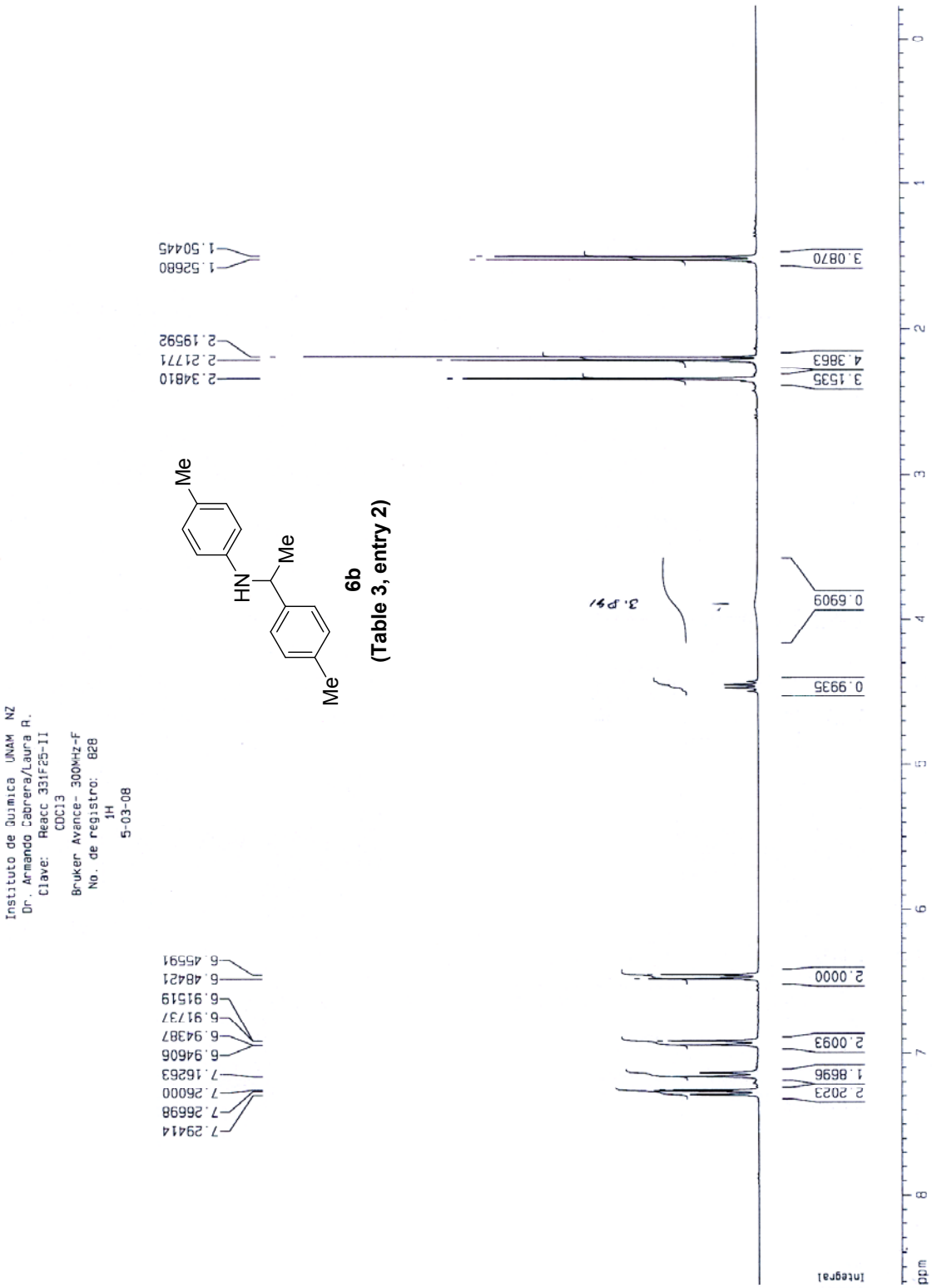
Area Percent Report

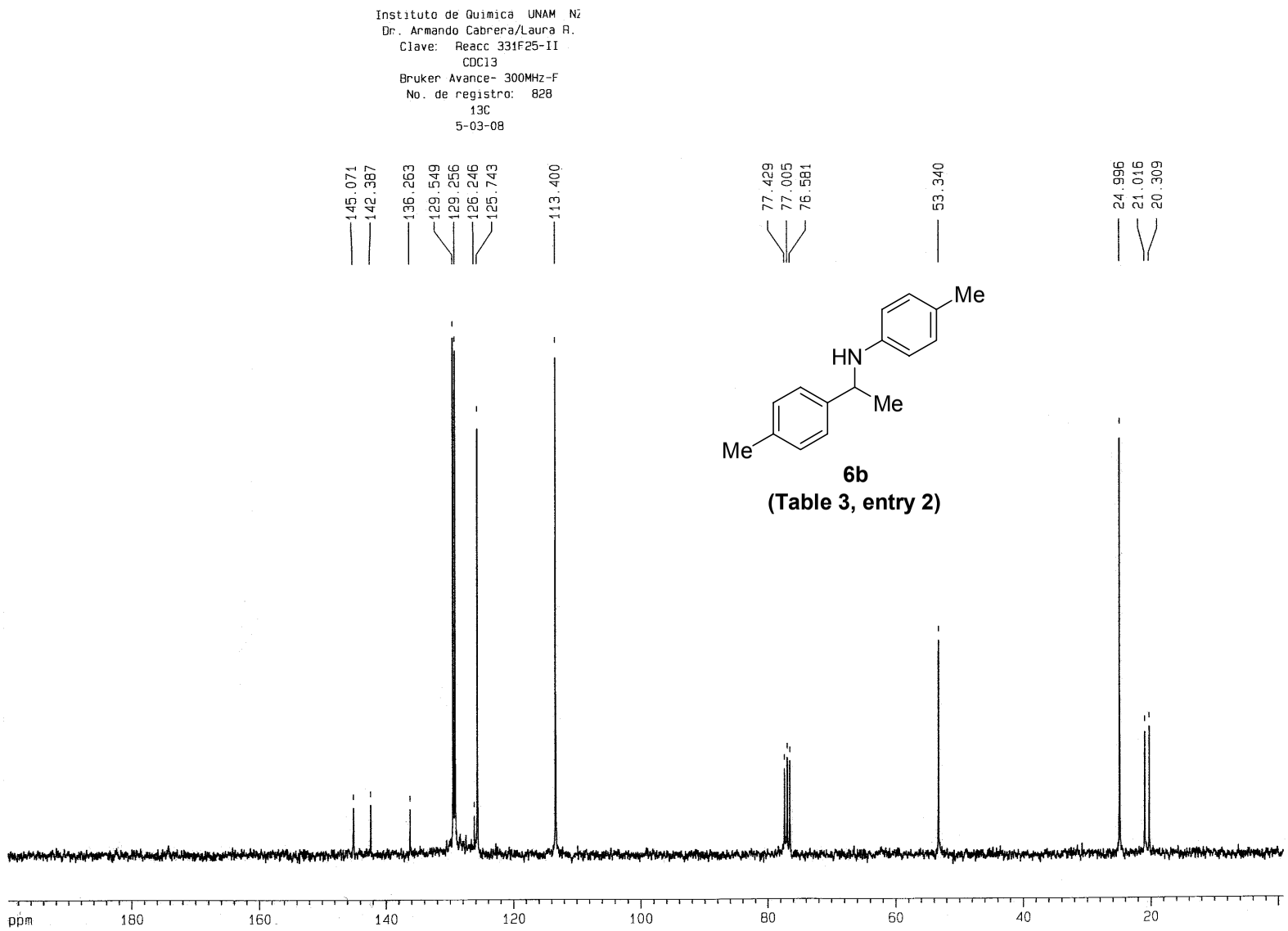
==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

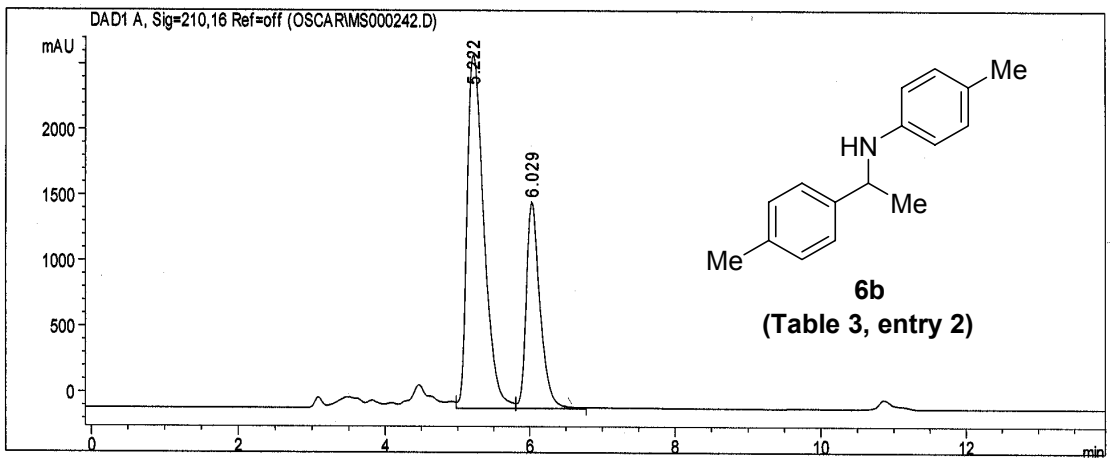
Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.998	VV	0.1615	3923.38159	364.84668	28.6245
2	6.944	VP	0.1830	9782.97656	821.06763	71.3755
Totals :				1.37064e4	1185.91431	


Instituto de Quimica UNAM NZ
Dr. Armando Cabrera/Laura R.
Clave: Reacc 331F25-II
CDCl3
Bruker Avance- 300MHz-F
No. de registro: 828
1H
5-03-08
Me
Me
HN
Me
6b
(Table 3, entry 2)
1.50445
1.52680
2.19592
2.21771
2.34810
6.45591
6.48421
6.91519
6.91737
6.94387
6.94606
7.16263
7.26000
7.26698
7.29414
3.0870
4.3863
3.1535
0.6909
0.9935
2.0000
2.0093
1.8696
2.2023
Integral
ppm


Instituto de Quimica UNAM N2
Dr. Armando Cabrera/Laura R.
Clave: Reacc 331F25-II
CDCl3
Bruker Avance- 300MHz-F
No. de registro: 828
13C
5-03-08
145.071
142.387
136.263
129.549
129.256
126.246
125.743
113.400
77.429
77.005
76.581
53.340
24.996
21.016
20.309
Me
HN
Me
Me
6b
(Table 3, entry 2)
ppm
180
160
140
120
100
80
60
40
20

Reacc331-quiral
080617-coa-08



Data File C:\HPCHEM\1\DATA\OSCAR\MS000242.D Sample Name: Reacc331-quiral
HPLC IQ 18/06/08 5:59:19 PM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
Injection Date : 17/06/08 12:40:26 PM
Sample Name : Reacc331-quiral Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 17/06/08 9:44:12 AM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 18/06/08 4:02:34 PM by carmen
(modified after loading)

==========

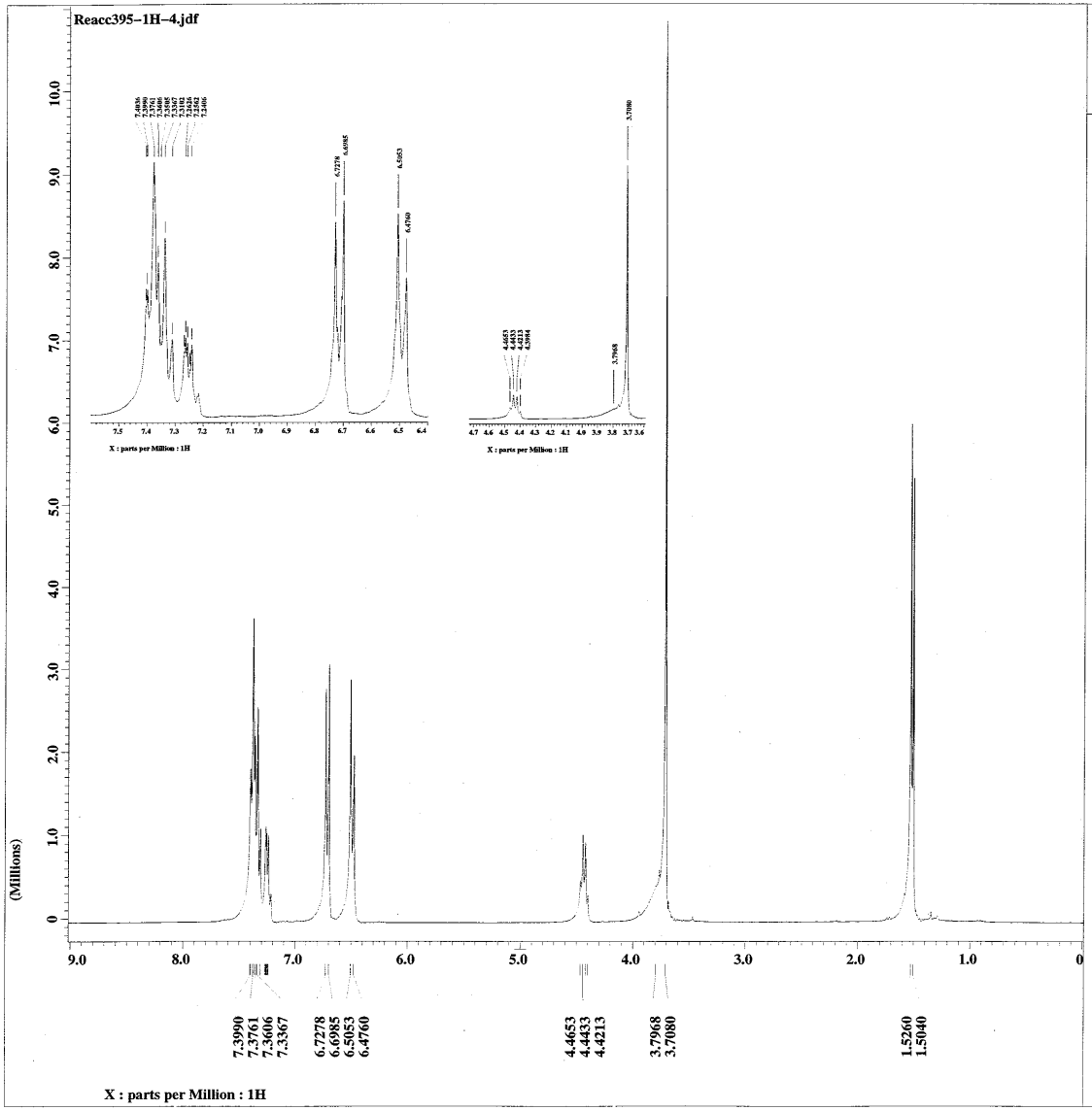
Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

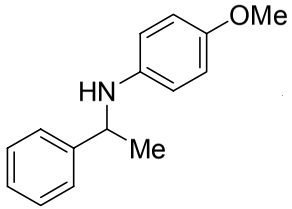
Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.222	VV	0.2482	4.34641e4	2694.71753	67.4740
2	6.029	VV	0.1993	2.09520e4	1574.06262	32.5260
Totals :				6.44161e4	4268.78015	

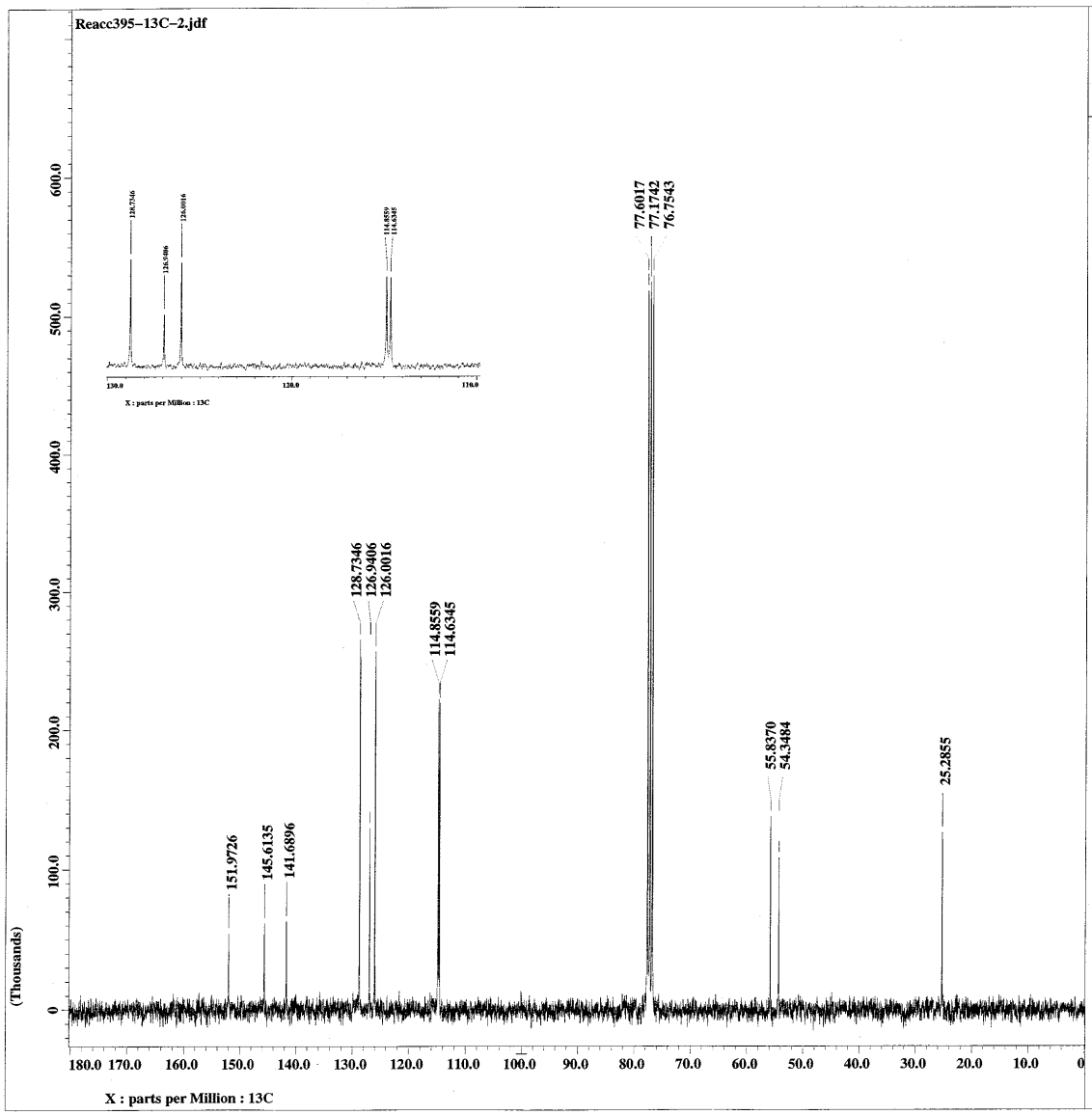


JEOL

```
Filename          = Reacc395-1H-4.jdf
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     =  4-FEB-2008 16:48:38
Revision_time     = 17-APR-2008 10:35:42
Current_time      = 17-APR-2008 10:37:46

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 17
Relaxation_delay  = 1[s]
Temp_get          = 17.4[dC]
Unblank_time      = 2[us]
```

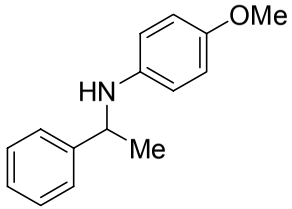


6c
(Table 3, entry 3)



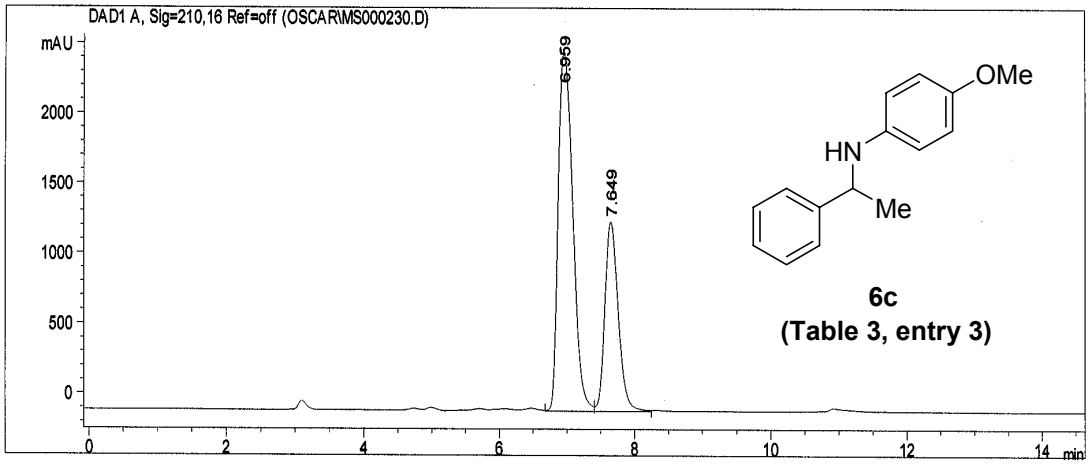
JEOL

Filename	= Reacc395-13C-2.jdf
Author	= Cabrera
Experiment	= single_pulse_dec
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 4-FEB-2008 18:09:13
Revision_time	= 17-APR-2008 11:53:25
Current_time	= 17-APR-2008 11:54:44
Comment	= Single Pulse with Bro
Data_format	= 1D COMPLEX
Dim_size	= 32768
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 1.7334272[s]
X_domain	= 13C
X_freq	= 75.56823426[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.57689184[Hz]
X_sweep	= 18.90359168[kHz]
Irr_domain	= 1H
Irr_freq	= 300.52965592[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 1743
Total_scans	= 1743
X_90_width	= 11.3[us]
X_acq_time	= 1.7334272[s]
X_angle	= 30[deg]
X_pulse	= 3.76666667[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 30
Relaxation_delay	= 1[s]
Temp_get	= 19.1[dC]
Unblank_time	= 2[us]



6c
(Table 3, entry 3)

Reacc395
080616-coa-07



Data File C:\HPCHEM\1\DATA\OSCAR\MS000230.D Sample Name: Reacc395
HPLC IQ 18/06/08 4:34:31 PM carmen
Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

==========

Injection Date : 16/06/08 4:57:00 PM
Sample Name : Reacc395 Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 16/06/08 3:24:23 PM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 18/06/08 4:02:34 PM by carmen
(modified after loading)
para Le legadec

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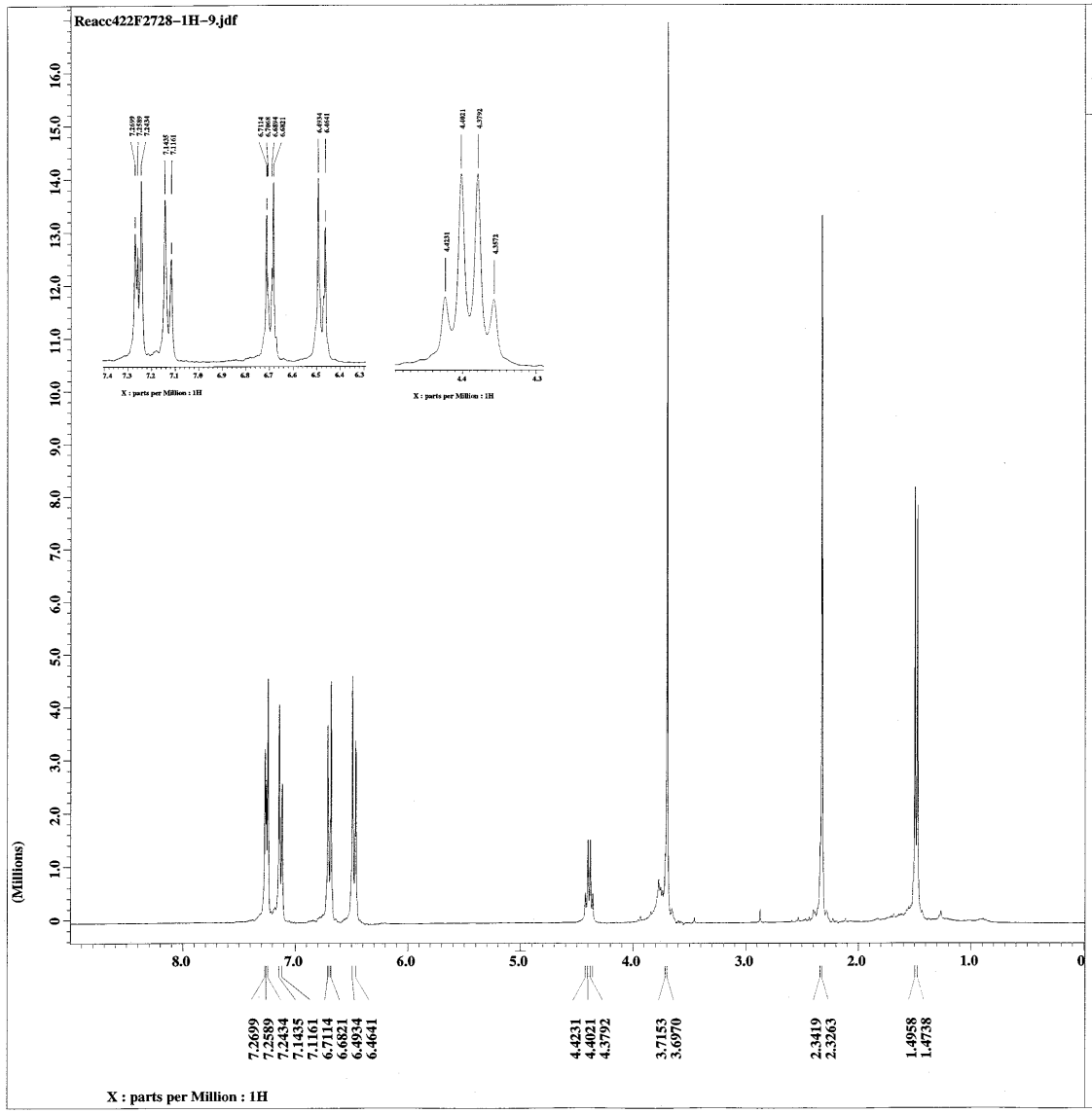
Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

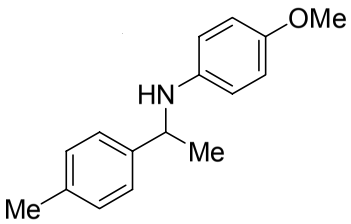
Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.959	VV	0.2437	3.84775e4	2554.97192	67.4206
2	7.649	VV	0.2128	1.85934e4	1349.13049	32.5794
Totals :				5.70709e4	3904.10242	

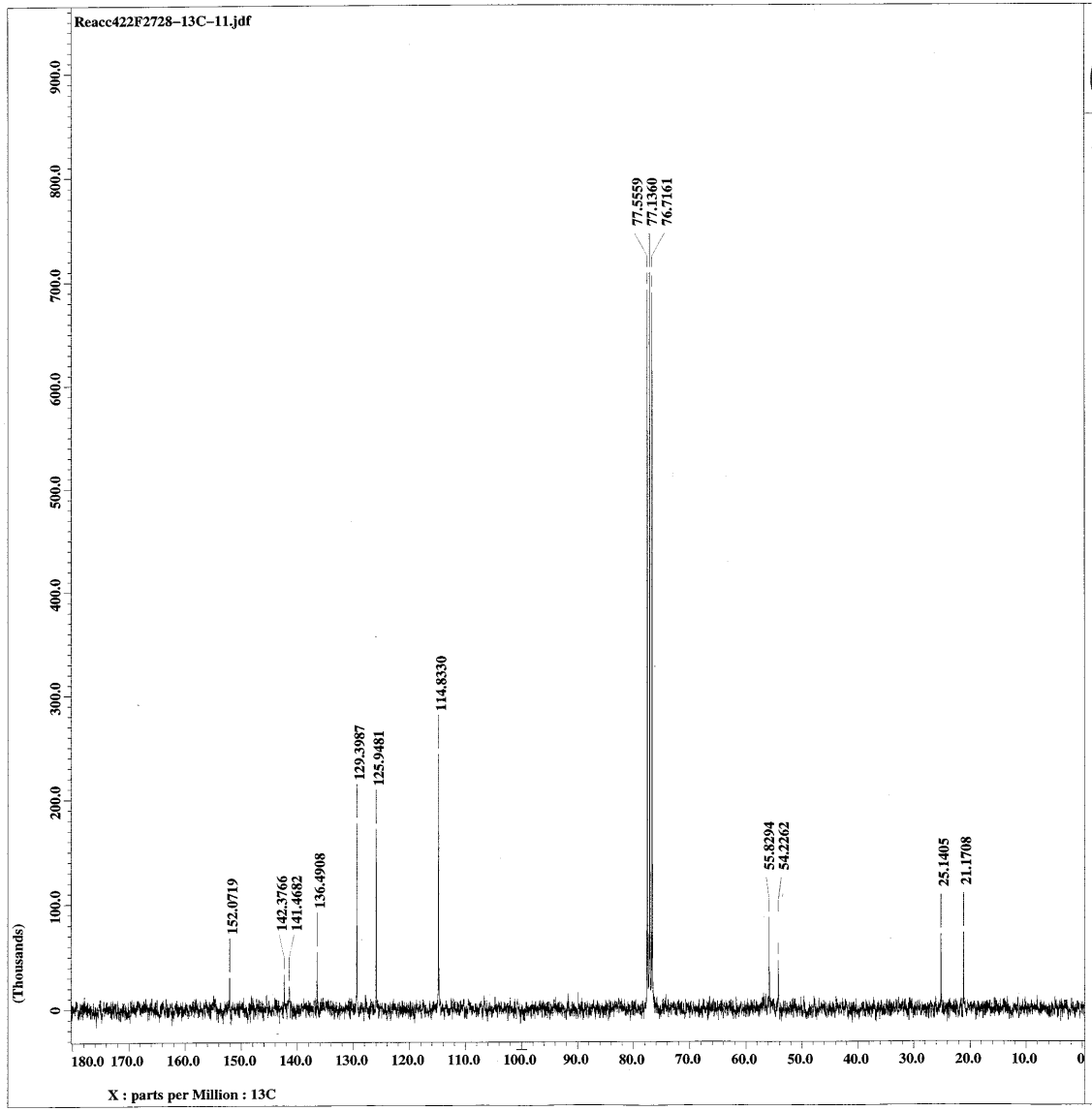


JEOL

```
Filename          = Reacc422F2728-1H-9.jd
Author            = Cabrera
Experiment        = single_pulse.exp
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 12-APR-2008 21:56:32
Revision_time     =  2-SEP-2008 07:51:11
Current_time      =  2-SEP-2008 07:52:52

Comment           = Single Pulse Experime
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 3.6339712[s]
X_domain          = 1H
X_freq            = 300.52965592[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 0
X_resolution      = 0.27518105[Hz]
X_sweep           = 4.50856628[kHz]
Clipped           = FALSE
Mod_return        = 1
Scans             = 32
Total_scans       = 32

X_90_width        = 9.5[us]
X_acq_time        = 3.6339712[s]
X_angle           = 45[deg]
X_pulse           = 4.75[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 20
Relaxation_delay  = 1[s]
Temp_get          = 19[dC]
Unblank_time      = 2[us]
```

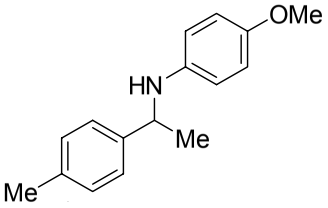


6d
(Table 3, entry 4)



JEOL

```
Filename          = Reacc422F2728-13C-11.
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 18-APR-2008 09:56:56
Revision_time     =  2-SEP-2008 07:41:14
Current_time      =  2-SEP-2008 07:41:35

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 1685
Total_scans       = 1685

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 21.1[dC]
Unblank_time      = 2[us]
```



6d
(Table 3, entry 4)

Data File C:\HPCHEM\1\DATA\OSCAR\MS000288.D Sample Name: Reacc 422

080630-coa-06
Chiralcel OD 250x 4.6 mm
hexano/isopropanol 98/2
flujo 1 ml/min
UV 230

==========

Injection Date : 30/06/08 6:54:03 PM
Sample Name : Reacc 422 Vial : 1
Acq. Operator : carmen
Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 30/06/08 6:53:35 PM by carmen
(modified after loading)
para Le legadec

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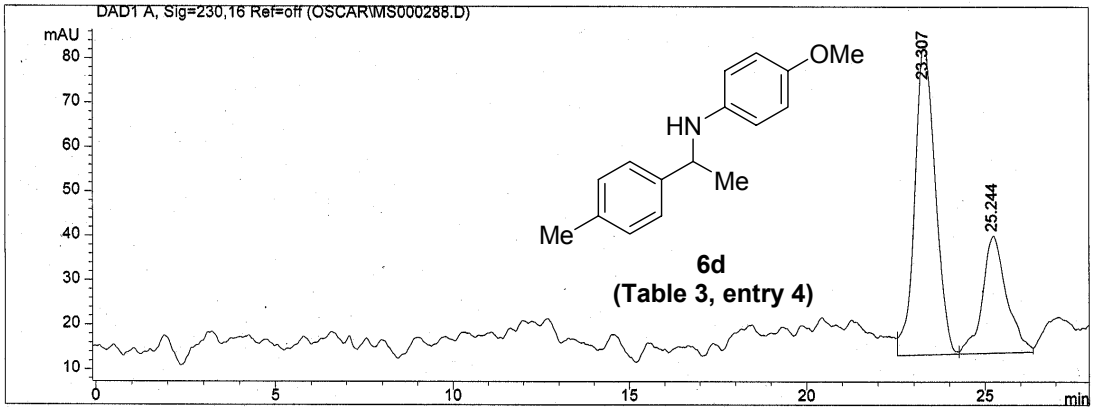


==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

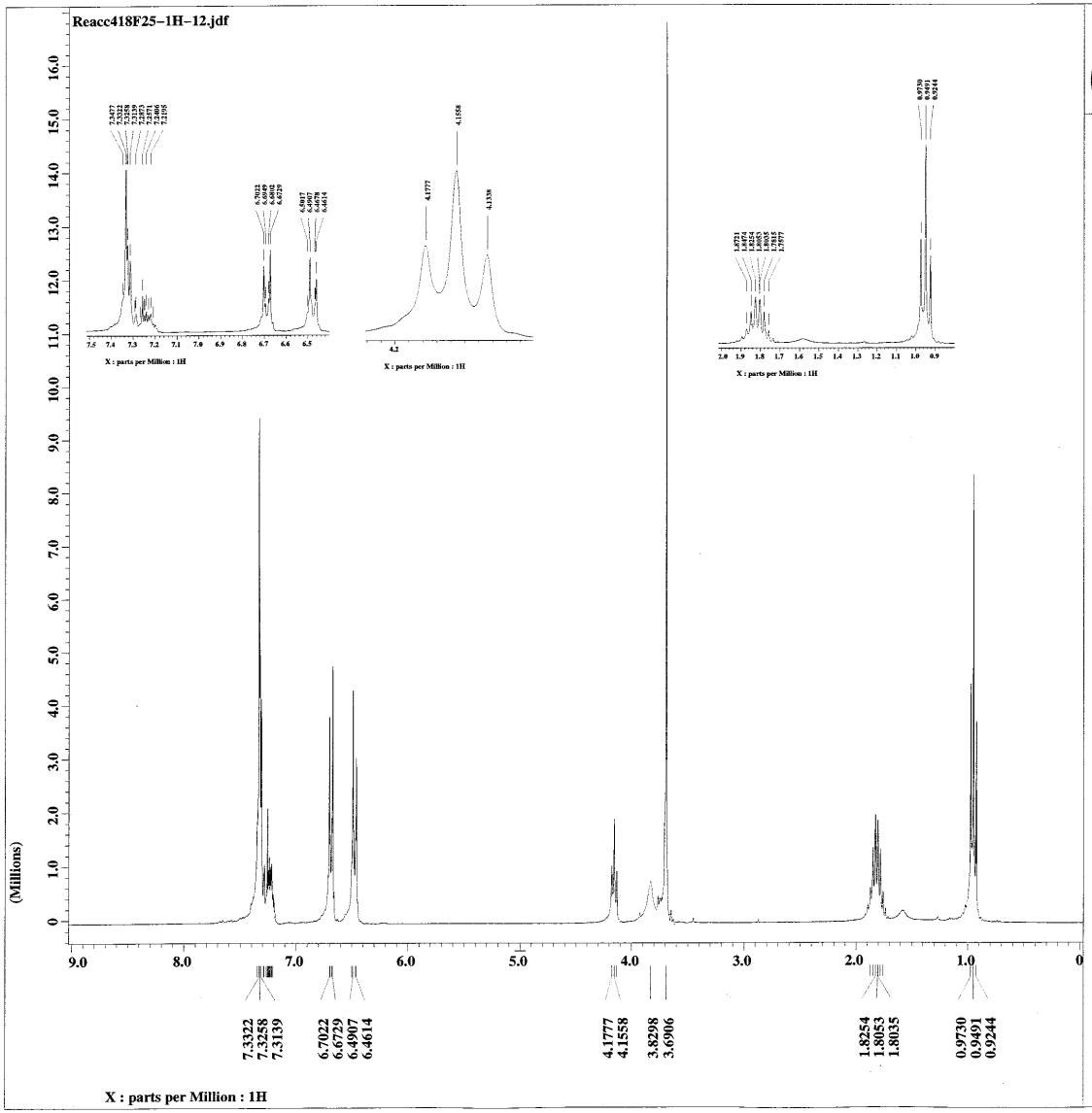
Signal 1: DAD1 A, Sig=230,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.307	BV	0.6129	2792.58521	69.48521	69.2117
2	25.244	VV	0.6864	1242.25806	26.33048	30.7883

Totals : 4034.84326 95.81569

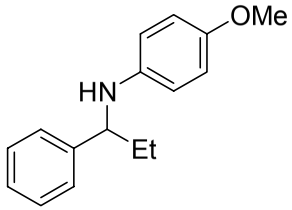
==========

*** End of Report ***

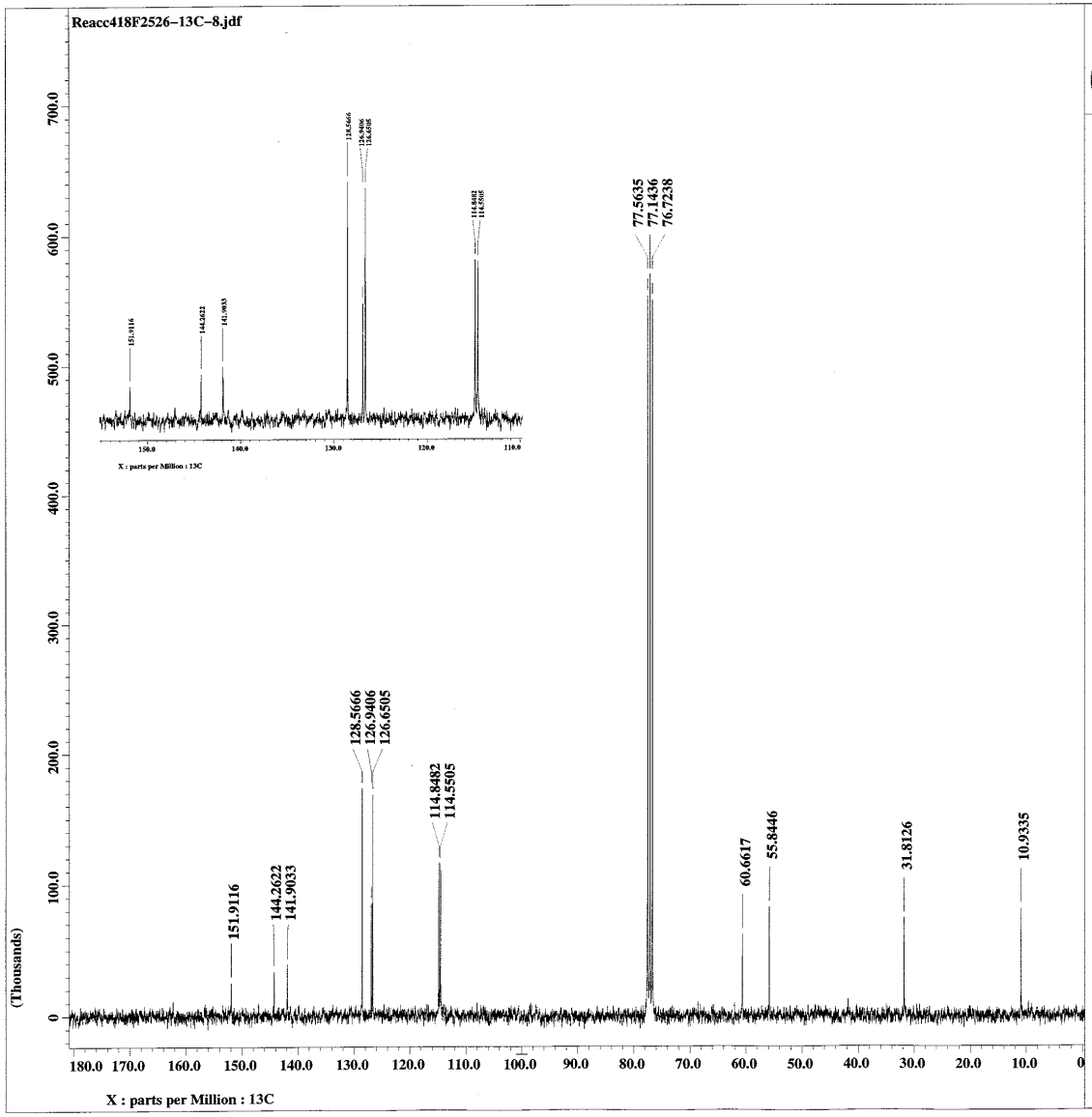


JEOL

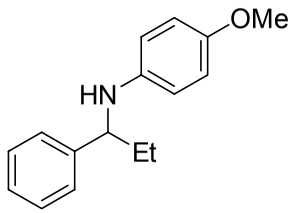
Filename	= Reacc418F25-1H-12.jdf
Author	= Cabrera
Experiment	= single_pulse.exp
Sample_id	= Laura
Solvent	= CHLOROFORM-D
Creation_time	= 31-MAR-2008 03:43:41
Revision_time	= 2-SEP-2008 07:37:17
Current_time	= 2-SEP-2008 07:39:23
Comment	= Single Pulse Experime
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= Eclipse+ 300
Spectrometer	= DELTA_NMR
Field_strength	= 7.0586013[T] (300[MHz
X_acq_duration	= 3.6339712[s]
X_domain	= 1H
X_freq	= 300.52965592[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 0
X_resolution	= 0.27518105[Hz]
X_sweep	= 4.50856628[kHz]
Clipped	= FALSE
Mod_return	= 1
Scans	= 32
Total_scans	= 32
X_90_width	= 9.5[us]
X_acq_time	= 3.6339712[s]
X_angle	= 45[deg]
X_pulse	= 4.75[us]
Initial_wait	= 1[s]
Phase_preset	= 3[us]
Recvr_gain	= 20
Relaxation_delay	= 1[s]
Temp_get	= 18.9[dC]
Unblank_time	= 2[us]



6e
(Table 3, entry 5)

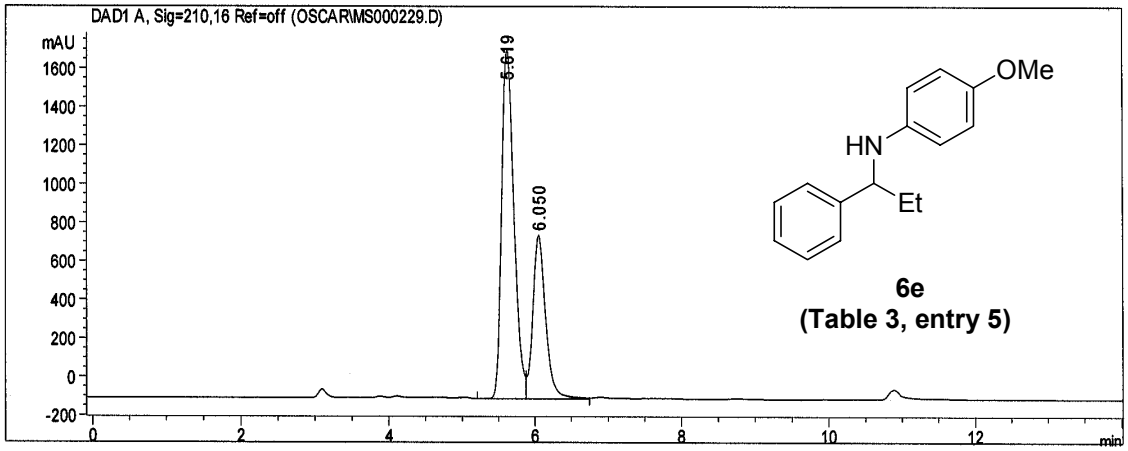



```
Filename          = Reacc418F2526-13C-8.j
Author            = Cabrera
Experiment        = single_pulse_dec
Sample_id         = Laura
Solvent           = CHLOROFORM-D
Creation_time     = 31-MAR-2008 09:36:14
Revision_time     =  3-SEP-2008 07:51:42
Current_time      =  3-SEP-2008 07:52:44

Comment           = Single Pulse with Bro
Data_format       = 1D COMPLEX
Dim_size          = 32768
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = Eclipse+ 300
Spectrometer      = DELTA_NMR

Field_strength    = 7.0586013[T] (300[MHz
X_acq_duration    = 1.7334272[s]
X_domain          = 13C
X_freq            = 75.56823426[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.57689184[Hz]
X_sweep           = 18.90359168[kHz]
Irr_domain        = 1H
Irr_freq          = 300.52965592[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 3000
Total_scans       = 3000

X_90_width        = 11.3[us]
X_acq_time        = 1.7334272[s]
X_angle           = 30[deg]
X_pulse           = 3.76666667[us]
Initial_wait      = 1[s]
Phase_preset      = 3[us]
Recvr_gain        = 30
Relaxation_delay  = 1[s]
Temp_get          = 20.8[dC]
Unblank_time      = 2[us]
```



6e
(Table 3, entry 5)

Reacc418F27
080616-coa-06



Data File C:\HPCHEM\1\DATA\OSCAR\MS000229.D Sample Name: Reacc418F27
HPLC IQ 18/06/08 4:29:26 PM carmen

Chiralcel OD 25x 4.6 mm
hexano/isopropanol 92/8
flujo 1 ml/min
UV 210

==========

Injection Date : 16/06/08 4:42:14 PM
Sample Name : Reacc418F27 Vial : 1
Acq. Operator : carmen
Acq. Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 16/06/08 3:24:23 PM by carmen
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed : 18/06/08 4:02:34 PM by carmen
(modified after loading)

==========

Area Percent Report

==========

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=210,16 Ref=off
Results obtained with enhanced integrator!

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.619	VV	0.1852	2.12812e4	1809.30591	67.1939
2	6.050	VV	0.1864	1.03901e4	851.18237	32.8061
Totals :				3.16713e4	2660.48828	