# Supplementary Information

## Diastereo- and Enantioselective Reductive Amination of Cycloaliphatic Ketones by Preformed Chiral Palladium Complexes

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**General:**

All reactions and manipulations were carried out by using Schlenk-type techniques. Flash column chromatography was performed on silica gel (70-230 mesh). $^{1}$H NMR, $^{13}$C NMR and $^{31}$P NMR spectra were obtained on a JEOL GX-300, 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. All chemical shifts ($\delta$) are reported in ppm and coupling constants (*J*) are reported in Hz to apparent peak multiplications. 2D NOESY, DEPT 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 and the characteristic absorption frequencies are reported in $cm^{-1}$. Optical rotations were performed on a Perkin-Elmer 343 spectropolarimeter (589 nm). 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 (EI mode at 70 eV). Elemental compositions were calculated within an uncertainty of 5 ppm by using the program installed in the computer system. The enantiomeric excess were determined by GC-MS analyses employing a Hewlett Packard 5890 (series II) instrument coupled with a JEOL JMS-AX505-A GC/MS-EI and Agilent Technologies 6890N coupled with JMS-GC/MS at 70 eV instruments employing a chiral capillary column Cyclodex-β (0.32 mm x 0.32 mm x 50 m) and 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. 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. Diethyl ether and benzene were distilled from sodium-benzophenone and chloroform was distilled from $P_2O_5$ under nitrogen. Other solvents were HPLC grade. The $Pd(MeCN)_2Br_2$, Pd[(*R*)-BINAP]$Cl_2$ (**1b**), Pd[(*R*)-BINAP]$Br_2$ (**1c**), Pd[(*S*)-BINAP]$Br_2$ (**1d**), Pd[(*R*)-Tol-BINAP]$Br_2$ (**1e**), Pd[(*S*)-Tol-BINAP]$Br_2$ (**1f**) and Pd[(*S*,*S*)-CHIRAPHOS]$Br_2$ (**1g**) complexes were prepared according to the literature procedures.[1-3]

Crystal structure of Pd[(*R*)-Tol-BINAP]$Br_2$ (**1e**):

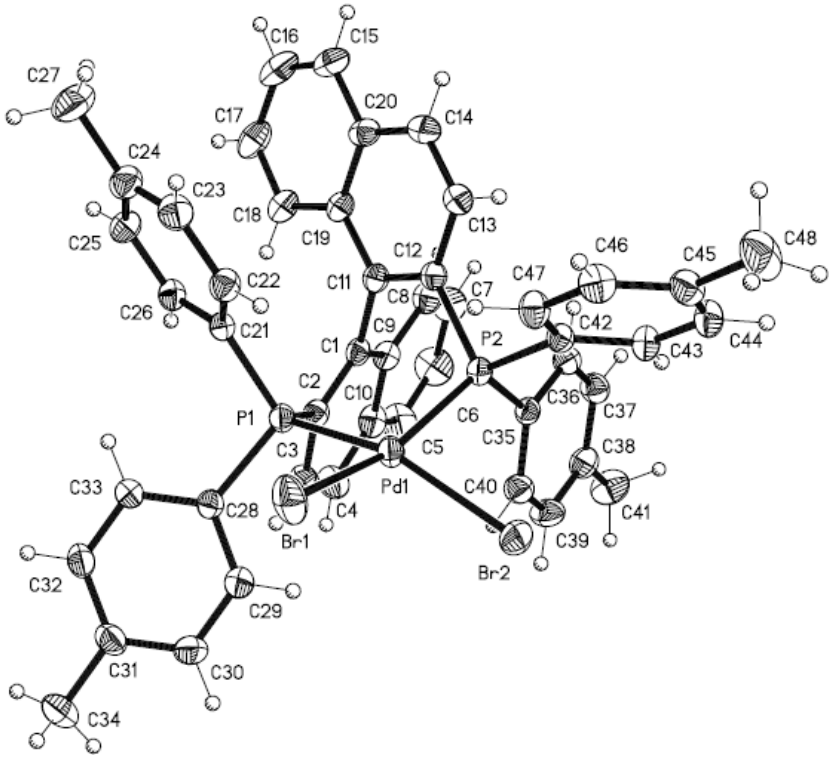
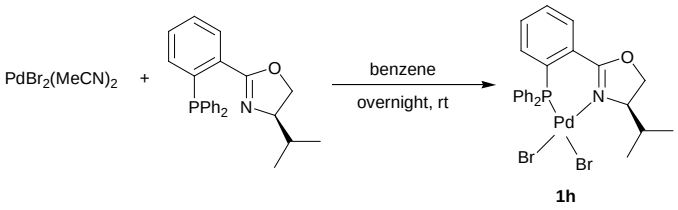



**Figure S1**. X-Ray diffraction structure of catalyst Pd[(*R*)-Tol-BINAP]$Br_2$ (**1e**). Selected bond lengths (Å): Pd(1)–Br(1) 2.4918(5); Pd(1)–Br(2) 2.4898(5); Pd(1)–P(1) 2.2473(1); Pd(1)–P(2) 2.2613(9); selected angles ($^o$): P(1)-Pd(1)-Br(2) 158.08(3); P(2)-Pd(1)-Br(1) 159.00(3); P(1)-Pd(1)-P(2) 94.14(3); P(1)-Pd(1)-Br(1) 90.27(3); P(2)-Pd(1)-Br(2) 88.80(3); Br(2)-Pd(1)-Br(1) 94.733(1).

**Preparation of complex 1h:**




This complex was synthesized by modified method described for the synthesis of $PdCl_2$[(*S*)-PˆN] reported in the literature.[4] In a Schlenk tube, $PdBr_2(MeCN)_2$ (174 mg, 0.5 mmol) was suspended in 10 mL of benzene. (*R*)-2-(2-(diphenylphosphino)phenyl)-4-isopropyl-1,3-oxazoline (186.5 mg, 0.5 mmol) was added. The suspension was stirred at room temperature for overnight. The yellow precipitate was collected by filtration, washed several times with diethyl ether and dried in vacuum. The 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 orange

crystals (yield: 82%). The presence of crystallization solvent in the product was ascertained by $^1$H NMR spectroscopy. $^1$H NMR (300 MHz, $CDCl_3$) δ 8.16-8.12 (m, 1H, Ar**H**), 7.75-7.29 (m, 12H, Ar**H**), 6.98-6.92 (m, 1H, Ar**H**), 5.71-5.68 (m, 1H, -C**H**N), 4.51 (t, 1H, *J* = 9.3 Hz, -C**H**$_2$O), 4.36 (q, 1H, *J* = 4.4 Hz, -C**H**$_2$O), 2.60-2.54 (m, 1H, C**H**$(CH_3)_2$), 0.78 (d, 3H, *J* = 7.1 Hz, -CH(C**H**$_3)_2$), -0.03 (d, 3H, *J* = 7.1 Hz, -CH(C**H**$_3)_2$); $^{31}$P NMR (121 MHz, $CDCl_3$) δ 26.7 (s, 1P, {(R)-P^N}); IR (dissolution in $CHCl_3$, $cm^{-1}$) 1624, 1570, 1247, 1100; MS ($FAB^+$) *m/z*: 638 $[M + 1]^+$, 560 (100), 478 (13), 402 (18); HRMS (FAB) *m/z* calcd for $C_{24}H_{25}Br_2NOP_2Pd$ $[M +1]^+$ 637.9075, found 637.9076; $[\alpha]^{20}_D$ -258.5 (c 0.4, acetone).

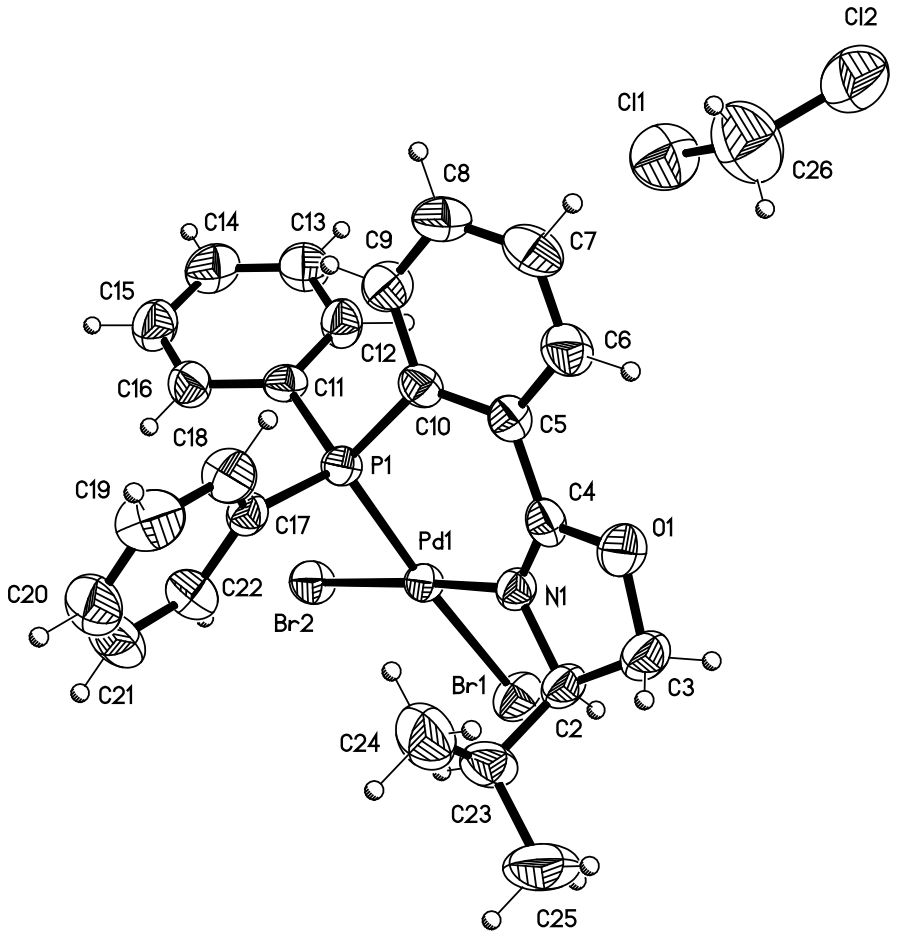
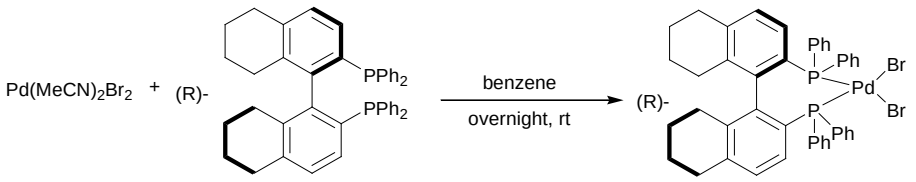


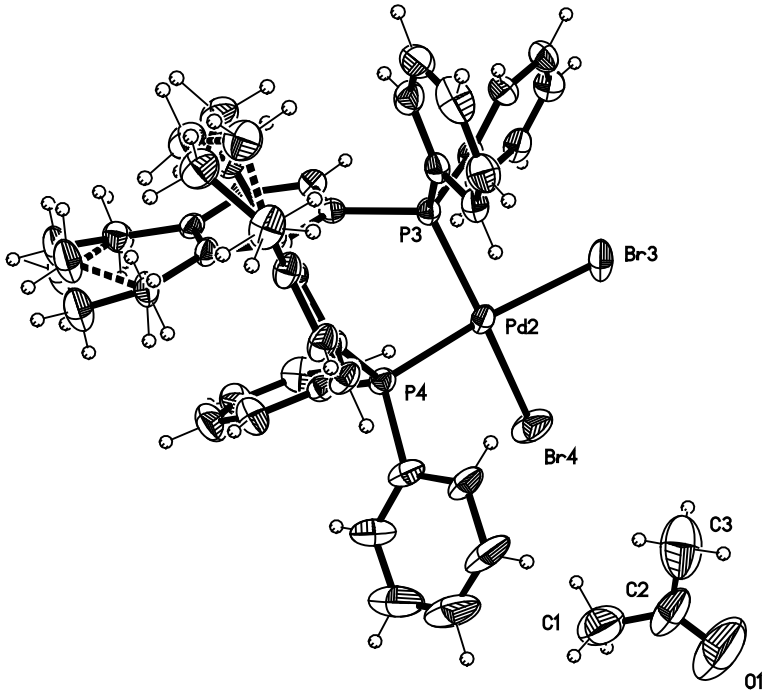

**Figure S2**. X-Ray diffraction structure of catalyst Pd[(*R*)-PHOX]$Br_2$.$CH_2Cl_2$(**1h**); selected bond lengths (Å): Pd(1)–Br(1) 2.511(1); Pd(1)–Br(2) 2.414(1); Pd(1)–P(1) 2.223(1); Pd(1)–N(1) 2.048(3); selected angles ($^o$): N(1)-Pd(1)-P(1) 88.40(9); N(1)-Pd(1)-Br(2) 174.42(9); P(1)-Pd(1)-Br(2) 89.79(3); N(1)-Pd(1)-Br(1) 92.61(9); P(1)-Pd(1)-Br(1) 175.06(3); Br(2)-Pd(1)-Br(1) 89.645(1).

**Preparation of complex 1i:**

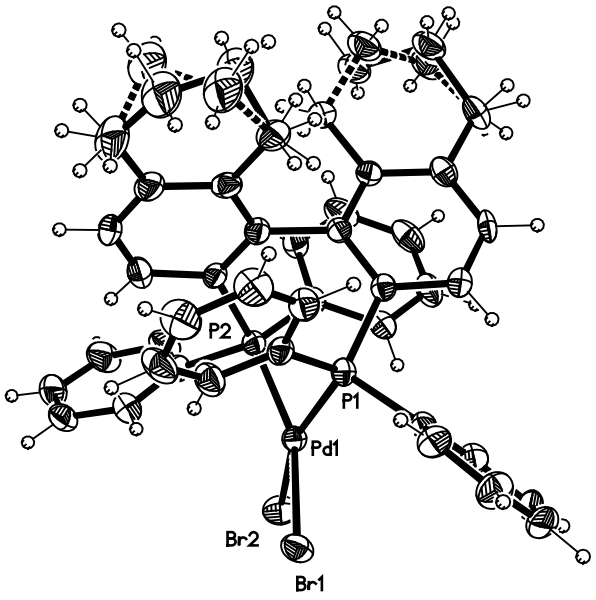



**1i**

Complex **1i** was prepared by modified method described for the synthesis of Pd[(*R*)-BINAP]$Cl_2$ reported in the literature.[2] In a Schlenk tube, [$(MeCN)_2$]$PdBr_2$ (174 mg, 0.5 mmol) and (*R*)-($H_8$)-BINAP (315 mg, 0.5 mmol) were suspended in 10 mL of benzene. The suspension was stirred at room temperature for overnigth. The yellow-orange precipitate was collected by filtration, washed several times with diethyl ether and dried in vacuum. Yield: 75%. $^{31}$P NMR (121 MHz, $CDCl_3$) δ 24.40 (s, 2P, BINAP); $^1$H NMR (300 MHz, $CDCl_3$) δ 7.87-7.74 (m, 8H, Ar**H**), 7.36-7.18 (m, 12H, Ar**H**), 6.99-6.93 (m, 2H, Ar**H**), 6.65 (d, 2H, J = 8.1 Hz, Ar**H**), 2.48-2.40 (m, 2H, -C**H**$_2$), 2.23-2.18 (m, 2H, -C**H**$_2$), 1.85-1.77 (m, 2H, -C**H**$_2$), 1.51-1.20 (m, 10H, -C**H**$_2$); FAB MS (positive ion mode): *m/z*: 815 [$M^+$ - Br]; HRMS-FAB (*m/z*): calcd for $C_{44}H_{40}BrP_2Pd$ [M –Br]$^+$ 815.0823, found: 815.0827; $[\alpha]^{20}_D$ +364 (c 0.2, $CHCl_3$).




**(a)**

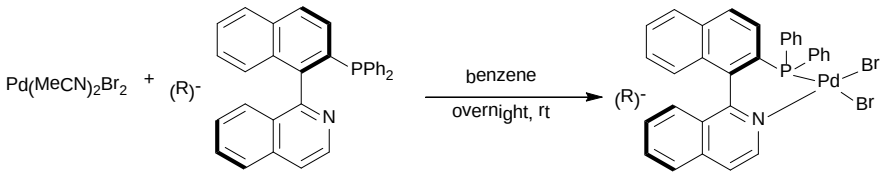



**(b)**

**Figure S3**. X-Ray diffraction structure of catalyst Pd[(*R*)-$H_8$-BINAP]$Br_2$.$COMe_2$ (**1h**); selected bond lengths (Å): **(a)** Pd(2)–Br(3) 2.4778(9); Pd(2)–Br(4) 2.4784(9); Pd(2)–P(3) 2.2731(1); Pd(2)–P(4) 2.2586(1); selected angles (°): P(4)-Pd(1)-Br(3) 169.15(5); P(3)-Pd(1)-Br(4) 163.97(5); P(4)-Pd(1)-P(3) 92.94(6); P(3)-Pd(2)-Br(3) 90.46(5); P(4)-Pd(2)-Br(4) 89.76(5); Br(3)-Pd(2)-Br(4) 89.81(4).

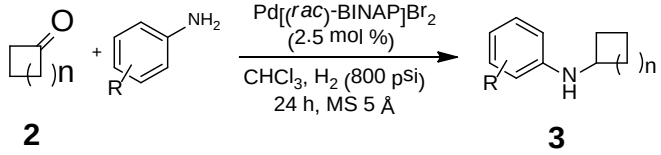
**(b)** Pd(1)–Br(1) 2.4842(8); Pd(1)–Br(2) 2.4948(8); Pd(1)–P(1) 2.2536(1); Pd(1)–P(2) 2.2632(1); selected angles (°): P(1)-Pd(1)-Br(2) 163.04(4); P(2)-Pd(1)-Br(1) 160.57(5); P(1)-Pd(1)-P(2) 93.00(6); P(1)-Pd(1)-Br(1) 88.95(5); P(2)-Pd(1)-Br(2) 90.66(5); Br(2)-Pd(1)-Br(1) 93.08(3).

**Preparation of complex 1j:**



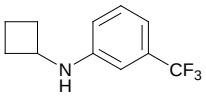

In a Schlenk tube, $[(MeCN)_2]PdBr_2$ (174 mg, 0.5 mmol) and (*R*)-QUINAP (219.5 mg, 0.5 mmol) were suspended in 10 mL of benzene. The suspension was stirred at room temperature for overnight. The yellow precipitate was collected by filtration, washed several times with diethyl ether and dried in vacuum. Yield: 68%. $^1$H NMR (300 MHz, $CDCl_3$) δ 9.57 (d, 1H, *J* = 8.0 Hz, Ar**H**), 8.05 (d, 2H, *J* = 8.0 Hz, Ar**H**), 7.95 (d, 1H, *J* = 8.4 Hz, Ar**H**), 7.61 (d, 1H, *J* = 8.5 Hz, Ar**H**), 7.59-7.52 (m, 5H, Ar**H**), 7.41-7.37 (m, 4H, Ar**H**), 7.31-7.19 (m, 4H, Ar**H**), 6.99-6.91 (m, 3H, Ar**H**), 6.81 (d, 1H, *J* = 8.4 Hz, Ar**H**); $^{31}$P NMR (121 MHz, $CDCl_3$) δ 35.43 (s, 1P, BINAP); FAB MS (positive ion mode): *m/z*: 706 [$M^+$]; HRMS-FAB (*m/z*): calcd for $C_{44}H_{40}BrP_2Pd$ [$M^+$] 706.9204, found: 706.9209; ; $[\alpha]^{20}_D$ +468 (c 0.21, $CHCl_3$).

**General procedure for reductive amination of cyclic ketones.**



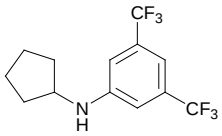

1.0 mmol of cycloaliphatic ketone and 1.3 mmol of aniline derivative were added to a stirred solution of 0.025 mmol of Pd[(*rac*)-BINAP]$Br_2$, **1k**, in 10 mL of dry chloroform (in a Schlenk tube) and stirred for 10 minutes. The solution was transferred to a 45 ml stainless steel autoclave (PARR) previously purged with vacum-$N_2$ and containing 50 mg of MS 5Å. Subsequently, the reaction was taken to the desired pressure (800 psi $H_2$), stirred in an oil bath at 80°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 corresponding product.



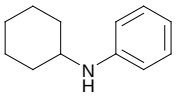
**3a**

***N-cyclobutyl-3-(trifluoromethyl)benzenamine*** **(3a)**. Prepared according to the general procedure from 2-cyclobutanone (75 μL, 1.0 mmol), *m*-trifluoromethyl aniline (162 μL, 1.3 mmol) and Pd[(*rac*)-BINAP]$Br_2$, **1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (95%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.14 (t, 2H, *J* = 7.8 Hz, Ar**H**), 6.82 (d, 1H, *J* = 7.5 Hz, Ar**H**), 6.64 (s, 1H, Ar**H**), 6.57 (d, 1H, *J* = 8.1 Hz, Ar**H**), 3.89-3.78 (m, 2H, -N**H**CH + -NHC**H**), 2.39-2.30 (m, 2H, -C$\mathbf{H}_2$), 1.80-1.67 (m, 4H, -C$\mathbf{H}_2$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 147.3, 131.5 (q, *J* = 31.6 Hz), 129.6, 124.5 (d, *J* = 272.0 Hz), 115.8, 113.5 (q, *J* = 3.9 Hz), 108.9 (q, *J* = 3.9 Hz), 48.7 (-NH**C**H), 31.0 (-2**C**$H_2$), 15.2 (-**C**$H_2$); IR (film, $cm^{-1}$) 3418, 2926, 2855, 1615, 1517; MS (EI) *m/z*: 215 ($M^+$), 187 (100), 166 (29), 145 (21); HRMS (EI) *m/z* calcd for $C_{11}H_{12}NF_3$ ($M^+$) 215.0922, found 215.0922.



**3b**

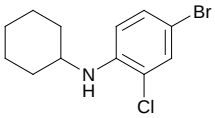
***N-cyclopentyl-3,5-bis(trifluoromethyl)benzenamine*** **(3b).** Prepared according to the general procedure from cyclopentanone (88 μL, 1.0 mmol), 3,5-bis(trifluoromethyl)aniline (203 μL, 1.3 mmol) and Pd[(*rac*)-BINAP]$Br_2$, **1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (93%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.14 (s, 1H, Ar**H**), 6.95 (s, 2H, Ar**H**), 4.14 (bs, 1H, -N**H**CH), 3.87 – 3.83 (m, 1H, -NHC**H**), 2.14-2.05 (m, 2H, -C$\mathbf{H}_2$), 1.82-1.67 (m, 4H, -C$\mathbf{H}_2$), 1.57-1.48 (m, 2H, -C$\mathbf{H}_2$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 148.4, 132.3 (q = 32.4 Hz), 123.6 (q = 272.5 Hz), 112.0 (q = 3.0 Hz), 109.5 (q = 3.9 Hz), 54.5 (-NH**C**H), 33.2 (-**C**$H_2$), 23.9 (**C**$H_2$); IR (film, $cm^{-1}$) 3414, 2925, 2848, 1615, 1517; MS (EI) *m/z*: 297 ($M^+$), 78 (13), 268 (100), 255 (16), 242 (11), 213 (14); HRMS (EI) *m/z* calcd for $C_{13}H_{13}NF_6$ ($M^+$) 297.0957, found 297.0952.



**3c**

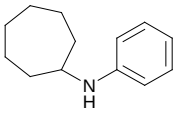
***N-cyclohexylbenzenamine*** **(3c)**. Prepared according to the general procedure from cyclohexanone (103 μL, 1.0 mmol), 3,5-bis(trifluoromethyl)aniline (203 μL, 1.3 mmol) and Pd[(*rac*)-BINAP]$Br_2$, **1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (89%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.17-7.10 (m, 2H, Ar**H**), 6.67-6.55 (m, 3H, Ar**H**), 3.48 (bs, 1H, -N**H**CH), 3.28-3.19 (m, 1H, - NHC**H**), 2.08-2.01 (m, 2H, -C$\mathbf{H}_2$), 1.79-1.72 (m, 2H, -C$\mathbf{H}_2$), 1.67-1.61 (m, 1H, -C$\mathbf{H}_2$), 1.43-1.07 (m, 5H, -C$\mathbf{H}_2$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 147.3, 116.7, 113.1, 51.6 (-NH**C**H), 33.4 (-**C**$H_2$), 25.9 (-**C**$H_2$), 25.0 (-**C**$H_2$); EM-IE (70 eV)

*m/z*: 175 ($M^+$), 146 (8), 132 (100), 119 (22); HRMS (EI) *m/z* calcd for $C_{12}H_{17}N_1$ ($M^+$) 175.1361, found 175.1356.



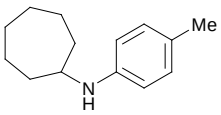
**3d**

***4-bromo-2-chloro-N-cyclohexylbenzenamine*** **(3d)**. Prepared according to the general procedure from cyclohexanone (103 μL, 1.0 mmol), 4-bromo-2-cloroaniline (266.5 mg, 1.3 mmol) and Pd[(*rac*)-BINAP]$Br_2$, **1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (74%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.38 (s, 1H, Ar**H**), 7.22 (dd, 1H, *J* = 8.7 Hz, Ar**H**), 6.55 (s, 1H, Ar**H**), 4.23 (d, 1H, *J* = 6.60 Hz, -N**H**CH), 3.33-3.24 (m, 2H, -NHC**H**), 2.07-1.84 (m, 2H, -C$\mathbf{H}_2$), 1.82-1.78 (m, 2H, -C$\mathbf{H}_2$), 1.71-1.64 (m, 1H, -C$\mathbf{H}_2$), 1.48-1.17 (m, 5H, -C$\mathbf{H}_2$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 142.3, 131.4, 130.4, 119.5, 112.6, 106.7, 51.5 (-NH**C**H), 33.0 (-**C**$H_2$), 25.8 (**C**$H_2$), 24.8 (**C**$H_2$); IR (film, $cm^{-1}$) 3415, 2927, 2850, 1615, 1517; MS (EI) *m/z*: 287 ($M^+$), 187 (100), 166 (29), 145 (21); HRMS (EI) *m/z* calcd for $C_{12}H_{15}NBrCl$ ($M^+$) 287.0076, found 287.0070.



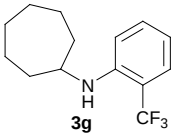
**3e**

***N-phenylcycloheptanamine*** **(3e)**. Prepared according to the general procedure from cycloheptanone (126 μL, 1.0 mmol), aniline (120 μL, 1.3 mmol) and Pd[(*rac*)-BINAP]$Br_2$, **1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (92%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.24-7.18 (m, 2H, Ar**H**), 6.73-6.69 (m, 1H, Ar**H**), 6.65-6.58 (m, 2H, Ar**H**), 3.63 (bs, 1H, -N**H**CH), 3.54-3.47 (m, 1H, - NHC**H**), 2.09-2.01 (m, 2H, -C$\mathbf{H}_2$), 1.76-1.45 (m, 10H, -C$\mathbf{H}_2$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 147.4, 29.4, 116.9, 113.3, 53.7 (-NH**C**H), 39.9 (-**C**$H_2$), 28.5 (-**C**$H_2$), 24.5 (-**C**$H_2$); IR (film, $cm^{-1}$) 3404, 2925, 2854, 1602, 1503; MS (EI) *m/z*: 189 ($M^+$), 146 (14), 132 (100), 120 (38); HRMS (EI) *m/z* calcd for $C_{13}H_{19}N_1$($M^+$) 189.1517, found 189.1520.



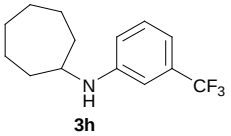
**3f**

***N-p-tolylcycloheptanamine*** **(3f)**. Prepared according to the general procedure from cycloheptanone (126 μL, 1.0 mmol), *p*-toluidine (139 mg, 1.3 mmol) and Pd[(*rac*)-BINAP], $Br_2$**1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (91%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 6.97 (d, 2H, *J* = 7.9 Hz, Ar**H**), 6.51 (d, 2H, *J* = 8.2 Hz, Ar**H**), 3.76 (bs, 1H, -N**H**CH), 3.45-3.38 (m, 1H, - NHC**H**), 2.23 (s, 3H, -**Me**), 2.03-1.95 (m, 2H, -C$\mathbf{H}_2$), 1.70-1.39 (m, 10H, -C$\mathbf{H}_2$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 144.6, 129.8, 126.5, 113.9, 54.4 (-NH**C**H), 34.7 (-**C**$H_2$), 28.4 (-**C**$H_2$), 24.4 (-**C**$H_2$), 20.4 (-**Me**); IR (film, $cm^{-1}$) 3400, 2924, 2855, 1617, 1517; MS (EI) *m/z*: 203 ($M^+$), 160 (5), 146 (25), 68 (100); HRMS (EI) *m/z* calcd for $C_{14}H_{21}N_1$($M^+$) 203.1674, found 203.1678.



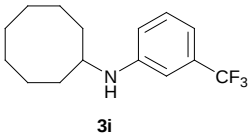
3g

***N-(2-(trifluoromethyl)phenyl)cycloheptanamine*** **(3g)**. Prepared according to the general procedure from cycloheptanone (118 μL, 1.0 mmol), *o*-trifluoromethyl aniline (163 μL, 1.3 mmol) and Pd[(*rac*)-BINAP]$Br_2$, **1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (86%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.45 (dd, 1H, *J* = 7.96, 1.20 Hz, Ar**H**), 7.37 (t, 1H, *J* = 7.81 Hz, Ar**H**), 6.71-6.66 (m, 2H, Ar**H**), 4.32 (bs, 1H, -N**H**CH), 3.59-3.56 (m, 1H, -NHC**H**), 2.07-2.00 (m, 2H, -C$\mathbf{H}_2$), 1.76-1.55 (m, 10H, -C$\mathbf{H}_2$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 144.8, 132.9, 126.7 (q =5.5 Hz), 125.3 (d = 272.1 Hz), 115.1, 113.1 (q = 29.0 Hz), 53.3 (-NH**C**H), 34.5 (-**C**$H_2$), 28.2 (**C**$H_2$), 24.2 (**C**$H_2$); IR (film, $cm^{-1}$) 3414, 2925, 2849, 1615, 1517; MS (EI) *m/z*: 257 ($M^+$, 83), 238 (13), 228 (14), 214 (24), 200 (100); HRMS (EI) *m/z* calcd for $C_{14}H_{18}NF_3$ ($M^+$) 257.1391, found 257.1389.



3h

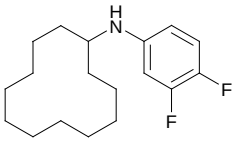
***N-(3-(trifluoromethyl)phenyl)cycloheptanamine*** **(3h)**. Prepared according to the general procedure from cycloheptanone (118 μL, 1.0 mmol), *m*-trifluomethylaniline (162 μL, 1.3 mmol) and Pd[(*rac*)-BINAP]$Br_2$, **1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (88%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.25 (t, 1H, *J* = 7.9 Hz, Ar**H**), 6.91 (d, 1H, *J* = 7.7 Hz, Ar**H**), 6.76 (s, 1H, Ar**H**), 6.68 (dd, 1H, *J* = 8.3, 2.2 Hz, Ar**H**), 3.84 (bs, 1H, -N**H**CH), 3.53-3.46 (m, 1H, - NHC**H**), 2.05-1.97 (m, 2H, -C$\mathbf{H}_2$), 1.74-1.50 (m, 10H, -C$\mathbf{H}_2$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 147.5, 131.6 (q, *J* = 32.4 Hz), 129.7, 124.5 (d, *J* = 272.2 Hz), 116.0, 113.1 (q, *J* = 3.4 Hz), 109.3 (q, *J* = 3.4 Hz), 53.6 (-NH**C**H), 34.7 (-**C**$H_2$), 28.4 (-**C**$H_2$), 24.3 (-**C**$H_2$); IR (film, $cm^{-1}$) 3425, 2929, 2857, 1614, 1515; MS (EI) *m/z*: 257 ($M^+$), 228 (3), 214 (24), 200 (100), 187 (19). HRMS (EI) *m/z* calcd for $C_{14}H_{18}NF_3$ ($M^+$) 257.1391, found 257.1388.



3i

***N-(3-(trifluoromethyl)phenyl)cyclooctanamine*** **(3i)**. Prepared according to the general procedure from cyclooctanone (132 μL, 1.0 mmol), *m*-trifluoromethyl aniline (162 μL, 1.3 mmol) and Pd[(*rac*)-BINAP]$Br_2$, **1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (88%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.13 (dd, 1H, *J* = 7.8 Hz, Ar**H**), 6.78 (d, 1H, *J* = 7.8 Hz, Ar**H**), 6.64 (s, 1H, Ar**H**), 6.57 (dd, 1H, *J* = 8.1, 2.0 Hz, Ar**H**), 3.70 (bs, 1H, -N**H**CH), 3.45-3.38 (m, 1H, -NHC**H**), 1.83-1.76 (m, 2H, -C$\mathbf{H}_2$), 1.66-1.44 (m, 12H, -C$\mathbf{H}_2$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 147.4, 131.5 (q, *J* = 31.5 Hz), 129.6, 124.0 (d, *J* = 272.0 Hz), 115.9, 112.9 (q, *J* = 3.8 Hz), 109.2 (q, *J* = 3.8 Hz), 52.4 (-NH**C**H), 32.3 (-**C**$H_2$), 27.1 (-**C**$H_2$), 25.8 (-**C**$H_2$), 23.9 (-**C**$H_2$); IR (film, $cm^{-1}$) 3416, 2926, 2850, 1614, 1515; MS (EI) *m/z*: 271 ($M^+$), 252 (5),

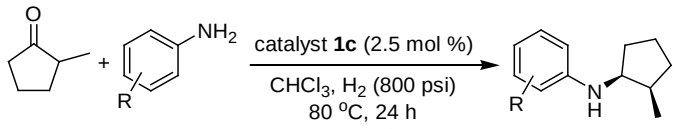
228 (8), 214 (7), 200 (100), 187 (31); HRMS (EI) *m/z* calcd for $C_{15}H_{20}F_3N$ ($M^+$) 271.1548, found 271.1550.



**3j**

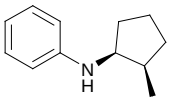
***N-(3,4-difluorophenyl)cyclododecanamine* (3j)**. Prepared according to the general procedure from cyclododecanone (182 mg, 1.0 mmol), 3,4-difluoromethyl aniline (130 μL, 1.3 mmol) and Pd[(*rac*)-BINAP]$Br_2$, **1k**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (81%). $^1H$ NMR (500 MHz, $CDCl_3$) δ 6.92 (ddd, 1H, $J_{H-H}$ = 9.0, $J_{H-F}$ = 9.0, 1.5 Hz, Ar**H**), 6.35 (ddd, 1H, $J_{H-H}$ = 9.5, $J_{H-F}$ = 6.5, 3.0 Hz, Ar**H**), 6.23-6.19 (m, 1H, Ar**H**), 3.39-3.35 (m,2H, -N**H**CH + -NHC**H**), 1.636-1.57 (m, 1H, -C$\mathbf{H}_2$), 1.49-1.24 (m, 21H, -C$\mathbf{H}_2$). $^1H$ NMR (125 MHz, $CDCl_3$) δ 150.9 (dd = 243.7, 13.7 Hz), 144.8 (d = 8.7Hz), 142.5 (dd = 233.7, 12.5 Hz), 117.3 (d = 12.5 Hz), 108.0 (d = 12.5 Hz), 101.3 (d = 25.0 Hz), 50.2 (-NH**C**H), 40.3 (-**C**$H_2$), 29.5 (-**C**$H_2$), 24.3 (-**C**$H_2$), 24.0 (-**C**$H_2$), 23.2 (-**C**$H_2$), 23.1(-**C**$H_2$), 21.1 (-**C**$H_2$); IR (film, $cm^{-1}$) 3475, 2955, 2873, 1614, 1518; MS (EI) *m/z*: 295 ($M^+$, 100), 266 (3), 252 (8), 238 (4), 224 (8), 168 (71); HRMS (EI) *m/z* calcd for $C_{18}H_{27}F_2N$ ($M^+$) 295.2112, found 295.2115.

**General procedure for asymmetric reductive amination of 2-methylcyclopentanone.**



*rac*-**4** **5**

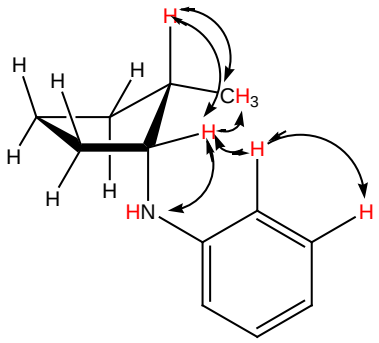
1.0 mmol of *rac*-**4** and 1.3 mmol of aniline derivative were added to a stirred solution of 0.025 mmol of preformed chiral palladium complex in 10 mL of dry chloroform (in a Schlenk tube) and stirred under nitrogen atmosphere for 10 minutes. The solution was transferred to a 45 ml stainless steel autoclave (PARR) previously purged with vacum-$N_2$. Subsequently, the reaction was taken to the desired pressure (900 psi $H_2$), stirred in an oil bath at 80$^o$C for 24 h. At the end of this period, the gas was liberated. The solution was analyzed by NMR to quantify the remaining substrate and d.r. respectively, 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 corresponding product.



**5a**

***cis-N*-(phenyl)-2-methylcyclopentaneamine (5a)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), aniline (120 μL, 1.3

mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as light yellow oil (90%). $^1H$ NMR (500 MHz, $CDCl_3$) δ 7.16-7.12 (m, 2H, Ar**H**), 6.64 (t, 1H, *J* = 7.8 Hz, Ar**H**), 6.60 (dd, 2H, *J* = 1.0, 7.8 Hz, Ar**H**), 3.72 (m, 1H, -NHC**H**), 3.56 (bs, 1H, -N**H**CH), 2.31-2.22 (m, 1H, -C**H**$CH_3$), 2.02-1.36 (m, 6H, -C$\mathbf{H}_2$), 0.88 (d, 3H, *J* = 7.0 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (125 MHz, $CDCl_3$) δ 148.2, 129.1, 116.6, 112.9, 57.2 (-NH**C**H), 35.8 (-**C**H$CH_3$), 32.0 (-**C**$H_2$), 31.5 (-**C**$H_2$), 21.2 (-**C**$H_2$), 14.3 (-CHC$\mathbf{H}_3$); IR (film, $cm^{-1}$) 3412, 2957, 2871, 1603, 1507; MS (EI) *m/z*: 175 ($M^+$), 146 (19), 132 (100), 119 (21); HRMS (EI) *m/z* calcd for $C_{12}H_{17}N(M^+)$ 175.1361, found 175.1364; $[\alpha]^{20}_D$ +9.2 (c 0.5, $CHCl_3$); ee = 98% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 31.3 min (minor), $t_R$ = 32.2 min (major)]. The relative stereochemistry was assigned by NOE analysis.




Interactions observed by NOE.

With Pd/C the selectivity *cis*/*trans* was 62/38.

With catalyst Pd[(*R*)-BINAP]$Cl_2$, **1b**: $[\alpha]^{20}_D$ +7.8 (c 0.48, $CHCl_3$); ee = 92% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 33.1 min (minor), $t_R$ = 34.3 min (major)].

With catalyst Pd[(*S*)-BINAP]$Br_2$, **1d**: $[\alpha]^{20}_D$ -11.2 (c 0.5, $CHCl_3$); ee = 95% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 31.4 min (major), $t_R$ = 32.3 min (minor)].

With catalyst Pd[(*R*)-Tol-BINAP]$Br_2$, **1e**: $[\alpha]^{20}_D$ +8.1 (c 0.5, $CHCl_3$); ee = 89% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 32.2 min (minor), $t_R$ = 33.3 min (major)].

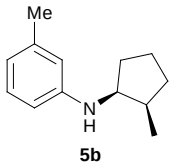
With catalyst Pd[(*S*)-Tol-BINAP]$Br_2$, **1f**: $[\alpha]^{20}_D$ -10.9 (c 0.51, $CHCl_3$); ee = 93% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 33.2 min (major), $t_R$ = 34.3 min (minor)].

With catalyst Pd[(*S*,*S*)-CHIRAPHOS]$Br_2$, **1g**: $[\alpha]^{20}_D$ +8.6 (c 0.49, $CHCl_3$); ee = 96% by GC-MS (EI) [chiral column: Cyclodex-β; conditions $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1°/min; $t_R$ = 33.4 min (minor), $t_R$ = 34.5 min (major)].

With catalyst Pd[(*R*)-PHOX]$Br_2$, **1h**: $[\alpha]^{20}_D$ -7.2 (c 0.53, $CHCl_3$); ee = 87% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1°/min; $t_R$ = 33.1 min (minor), $t_R$ = 34.2 min (major)].

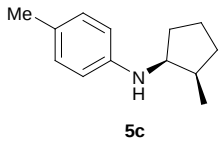
With catalyst Pd[(*R*)-$H_8$BINAP]$Br_2$, **1i**: $[\alpha]^{20}_D$ +9.0 (c 0.5, $CHCl_3$); ee = 98% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1°/min; $t_R$ = 31.3 min (minor), $t_R$ = 32.2 min (major)].

With catalyst Pd[(*R*)-QUINAP]$Br_2$, **1b**: $[\alpha]^{20}_D$ -8.5 (c 0.51, $CHCl_3$); ee = 89% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1°/min; $t_R$ = 32.4 min (minor), $t_R$ = 33.8 min (major)].



**5b**

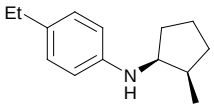
***cis*-*N*-(3-methylphenyl)-2-methylcyclopentaneamine (5b)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), *m*-toluidine (140 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 °C for 24 h, to provide the title compound as light yellow oil (90%). $^1$H NMR (300 MHz, $CDCl_3$) δ 7.10 (t, 1H, *J* = 7.9 Hz, Ar**H**), 6.55-6.47 (m, 3H, Ar**H**), 3.77 (m, 1H, -NHC**H**), 3.63 (bs, 1H, -N**H**CH), 2.37-2.27 (m, 4H, -C**H**$CH_3$ + -Me), 2.10-1.39 (m, 6H, -C$\mathbf{H}_2$), 0.93 (d, 3H, *J* = 6.8 Hz, -CHC$\mathbf{H}_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 148.3, 139.0, 129.2, 117.8, 113.8, 110.2, 57.2 (-NH**C**H), 35.8 (-**C**HMe), 32.1 (-**C**$H_2$), 31.6 (-**C**$H_2$), 21.8 (-**C**$H_3$), 21.3 (-**C**$H_2$), 14.5 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3410, 2955, 2868, 1604, 1511; MS (EI) *m/z*: 189 ($M^+$), 160 (12), 146 (100), 133 (22); HRMS (EI) *m/z* calcd for $C_{13}H_{19}N(M^+)$ 189.1517, found 189.1515; $[\alpha]^{20}_D$ +8.2 (c 0.5, $CHCl_3$); ee = 93% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1°/min; $t_R$ = 30.2 min (minor), $t_R$ = 31.2 min (major)].



**5c**

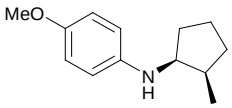
***cis*-*N*-(4-methylphenyl)-2-methylcyclopentaneamine (5c)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), *p*-toluidine (139 mg, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 °C for 24 h, to provide the title compound as yellow oil (91%). $^1$H NMR (500 MHz, $CDCl_3$) δ 6.95 (d, 2H, *J* = 8.0 Hz, Ar**H**), 6.53 (d, 2H, *J* = 8.5 Hz, Ar**H**), 3.69 (m, 1H, -NHC**H**), 3.35 (bs, 1H, -N**H**CH), 2.29-2.21 (m, 4H, -C**H**$CH_3$ + -Me), 2.00-1.35 (m, 6H, -C$\mathbf{H}_2$), 0.87 (d, 3H, *J* = 7.0 Hz, -CHC$\mathbf{H}_3$); $^{13}$C

NMR (125 MHz, $CDCl_3$) δ 145.9, 129.6, 125.8, 113.1, 57.5 (-NH**C**H), 35.7 (-**C**HCH$_3$), 31.9 (-**C**H$_2$), 31.5 (-**C**H$_2$), 21.1 (-**C**H$_2$), 20.3 (-**C**H$_3$), 14.3 (-CH**C**H$_3$); IR (film, cm$^{-1}$) 3409, 2955, 2868, 1618, 1518; MS (EI) *m/z*: 189 ($M^+$), 160 (17), 146 (100), 133 (37); HRMS (EI) *m/z* calcd for $C_{13}H_{19}N(M^+)$ 189.1517, found 189.1521; $[\alpha]^{20}_D$ +5.2 (c 0.5, $CHCl_3$); ee = 95% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1°/min; $t_R$ = 39.0 min (minor), $t_R$ = 40.2 min (major)].



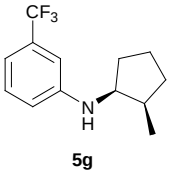
**5d**

***cis-N*-(4-ethylphenyl)-2-methylcyclopentaneamine (5d)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), *p*-ethylaniline (160 μL, 1.3 mmol) and Pd[(*R*)-BINAP]Br$_2$, **1c**, (22 mg, 0.025 mmol) at 80 °C for 24 h, to provide the title compound as yellow oil (86%). $^1$H NMR (300 MHz, $CDCl_3$) δ 7.00 (d, 2H, *J* = 7.9 Hz, Ar**H**), 6.57 (d, 2H, *J* = 7.9 Hz, Ar**H**), 3.73-3.56 (m, 2H, -NHC**H** + -N**H**CH), 2.53 (q, 2H, *J* = 7.5 Hz, -C**H**$_2$CH$_3$), 2.33-2.22 (m, 1H, -C**H**CH$_3$), 1.99-1.39 (m, 6H, -C**H**$_2$), 1.19 (t, 3H, *J* = 7.5 Hz, -CH$_2$C**H**$_3$), 0.88 (d, 3H, *J* = 7.1 Hz, -CHC**H**$_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 146.1, 132.6, 128.5, 113.1, 57.6 (-NH**C**H), 35.8 (-**C**HCH$_3$), 32.0 (-**C**H$_2$), 31.5 (-**C**H$_2$), 27.9 (-**C**H$_2$CH$_3$), 21.2 (-**C**H$_2$), 16.9 (-CH$_2$**C**H$_3$), 14.4 (-CH**C**H$_3$); IR (film, cm$^{-1}$) 3413, 2958, 2869, 1616, 1517; MS (EI) *m/z*: 203 ($M^+$), 174 (11), 160 (65), 147 (21), 106 (100); HRMS (EI) *m/z* calcd for $C_{14}H_{21}N(M^+)$ 203.1674, found 203.1674; $[\alpha]^{20}_D$ +5.9 (c 0.17, $CHCl_3$); ee = 83% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1°/min; $t_R$ = 51.8 min (minor), $t_R$ = 53.1 min (major)].



**5f**

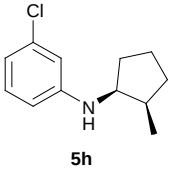
***cis-N*-(4-methoxyphenyl)-2-methylcyclopentaneamine (5f)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), *p*-anisidine (160 mg, 1.3 mmol) and Pd[(*R*)-BINAP]Br$_2$, **1c**, (22 mg, 0.025 mmol) at 80 °C for 24 h, to provide the title compound as yellow oil (78%). $^1$H NMR (300 MHz, $CDCl_3$) δ 6.76 (d, 2H, *J* = 8.8 Hz, Ar**H**), 6.60 (d, 2H, *J* = 8.8 Hz, Ar**H**), 3.74 (s, 4H, -OMe), 3.66 (m, 1H, -NHC**H**), 3.21 (bs, 1H, -N**H**CH), 2.30-2.21 (m, 1H, -C**H**CH$_3$), 1.99-1.34 (m, 6H, -C**H**$_2$), 0.88 (d, 3H, *J* = 6.9 Hz, -CHC**H**$_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 142.3, 114.9, 114.4, 111.7, 58.3 (-NH**C**H), 55.9 (-O**C**H$_3$), 35.7 (-**C**HCH$_3$), 32.0 (-**C**H$_2$), 31.3 (-**C**H$_2$), 21.2 (-**C**H$_2$), 14.3 (-CH**C**H$_3$); IR (film, cm$^{-1}$) 3398, 2954, 2869, 1615, 1512; MS (EI) *m/z*: 205 ($M^+$), 190 (10), 176 (18), 162 (100), 149 (38); HRMS (EI) *m/z* calcd for $C_{13}H_{19}NO(M^+)$ 205.1473, found 205.1467; $[\alpha]^{20}_D$ +8.4 (c 0.5, $CHCl_3$); ee = 96% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1 °/min; $t_R$ = 54.6 min (minor), $t_R$ = 56.2 min (major)].



5g

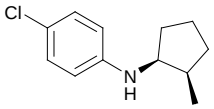
***cis*-*N*-(3-trifluoromethylphenyl)-2-methylcyclopentaneamine (5g)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), *m*-trifluomethylaniline (162 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (80%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.22 (t, 1H, *J* = 7.7 Hz, Ar**H**), 6.88 (d, 1H, *J* = 7.7 Hz, Ar**H**), 6.81 (s, 1H, Ar**H**), 6.74 (dd, 1H, *J* = 2.0, 7.7 Hz, Ar**H**), 3.90 (bs, 1H, -N**H**CH), 3.78-3.71 (m, 1H, -NHC**H**), 2.36-2.22 (m, 1H, -C**H**$CH_3$), 2.05-1.38 (m, 6H, -C$\mathbf{H}_2$), 0.89 (d, 3H, *J* = 7.1 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 148.3, 131.5 (q, *J* = 31.3 Hz), 129.6, 124.5 (d, *J* = 272.2 Hz), 115.9, 113.1 (q, *J* = 3.6 Hz), 109.0 (q, *J* = 3.6 Hz), 57.2 (-NH**C**H), 35.8 (-**C**H$CH_3$), 32.0 (-**C**$H_2$), 31.5 (-**C**$H_2$), 21.2 (-**C**$H_2$), 14.4 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3436, 2960, 2873, 1615, 1517; MS (EI) *m/z*: 243 ($M^+$), 214 (16), 200 (100), 187 (15); HRMS (EI) *m/z* calcd for $C_{13}H_{16}NF_3$($M^+$) 243.1235, found 243.1239; $[\alpha]^{20}_D$ +7.2 (c 0.58, $CHCl_3$); ee = 98% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 17.2 min (minor), $t_R$ = 17.6 min (major)].

With Pd[(*S*)-BINAP]$Br_2$, **1d**, $[\alpha]^{20}_D$ -10.9 (c 0.55, $CHCl_3$).



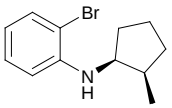
5h

***cis*-*N*-(3-chlorophenyl)-2-methylcyclopentaneamine (5h)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), *m*-chloroaniline (136 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (86%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.05 (t, 1H, *J* = 7.8 Hz, Ar**H**), 6.63 (s, 1H, Ar**H**), 6.60-6.58 (m, 1H, Ar**H**), 6.47 (dd, 1H, *J* = 1.8, 7.8 Hz, Ar**H**), 3.83 (bs, 1H, -N**H**CH), 3.73-3.66 (m, 1H, -NHC**H**), 2.35-2.21 (m, 1H, -C**H**$CH_3$), 2.05-1.34 (m, 6H, -C$\mathbf{H}_2$), 0.88 (d, 3H, *J* = 7.2 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 149.1, 134.9, 130.0, 116.5, 112.4, 111.3, 57.1 (-NH**C**H), 35.6 (-**C**H$CH_3$), 31.8 (-**C**$H_2$), 31.4 (-**C**$H_2$), 21.1 (-**C**$H_2$), 14.3 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3423, 2957, 2870, 1598, 1501; MS (EI) *m/z*: 209 ($M^+$), 180 (21), 166 (100), 153 (22); HRMS (EI) *m/z* calcd for $C_{12}H_{16}NCl$($M^+$) 209.0971, found 209.0969; $[\alpha]^{20}_D$ +7.9 (c 0.58, $CHCl_3$); ee = 91% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 39.8 min (minor), $t_R$ = 41.0 min (major)].



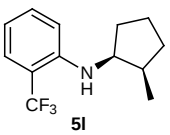
5i

***cis*-*N*-(4-chlorophenyl)-2-methylcyclopentaneamine (5i)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), *p*-chloroaniline (165 mg, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as light yellow oil (88%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.09 (d, 2H, *J* = 8.7 Hz, Ar**H**), 6.53 (d, 2H, *J* = 8.7 Hz, Ar**H**), 3.90 (bs, 1H, -N**H**CH), 3.71-3.65 (m, 1H, -NHC**H**), 2.37-2.22 (m, 1H, -C**H**$CH_3$), 2.03-1.34 (m, 6H, -C$\mathbf{H}_2$), 0.87 (d, 3H, *J* = 7.2 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 146.6, 128.9, 121.2, 114.0, 57.4 (-NH**C**H), 35.6 (-**C**$HCH_3$), 31.9 (-**C**$H_2$), 31.4 (-**C**$H_2$), 21.1 (-**C**$H_2$), 14.3 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3421, 2957, 2870, 1599, 1499; MS (EI) *m/z*: 209 ($M^+$), 180 (16), 166 (100), 153 (27); HRMS (EI) *m/z* calcd for $C_{12}H_{16}NCl(M^+)$ 209.0971, found 209.0968; $[\alpha]^{20}_D$ +10.4 (c 0.5, $CHCl_3$); ee = 84% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 32.3 min (minor), $t_R$ = 33.7 min (major)].



5j

***cis*-*N*-(2-bromophenyl)-2-methylcyclopentaneamine (5j)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), *o*-bromoaniline (147 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (75%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.40 (d, 1H, *J* = 8.1 Hz, Ar**H**), 7.15 (td, 1H, *J* = 1.5, 8.1 Hz, Ar**H**), 6.70 (dd, 1H, *J* = 1.3, 8.2 Hz, Ar**H**), 6.53 (td, 1H, *J* = 1.5, 8.0 Hz, Ar**H**), 4.13 (bs, 1H, -N**H**CH), 3.80-3.73 (m, 1H, -NHC**H**), 2.37-2.21 (m, 1H, -C**H**$CH_3$), 2.07-1.41 (m, 6H, -C$\mathbf{H}_2$), 0.93 (d, 3H, *J* = 6.9 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 144.6, 132.3, 128.3, 117.2, 112.0, 109.8, 57.4 (-NH**C**H), 35.9 (-**C**$HCH_3$), 31.9 (-**C**$H_2$), 31.5 (-**C**$H_2$), 21.3 (-**C**$H_2$), 14.4 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3423, 2957, 2870, 1598, 1501; MS (EI) *m/z*: 253 ($M^+$), 224 (19), 210 (100), 197 (21); HRMS (EI) *m/z* calcd for $C_{12}H_{16}NBr(M^+)$ 253.0466, found 253.0467; $[\alpha]^{20}_D$ -2.2 (c 0.23, $CHCl_3$); ee = 18% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 32.3 min (minor), $t_R$ = 32.5 min (major)].

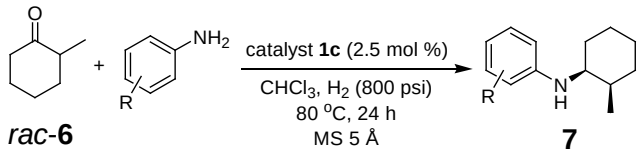


5l

***cis*-*N*-(2-trifluoromethylphenyl)-2-methylcyclopentaneamine (5l)**. Prepared according to the general procedure from 2-methylcyclopentanone (106 μL, 1.0 mmol), *o*-trifluomethylaniline (163 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as light yellow oil (69%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.37 (dd, 1H, *J* = 7.96, 1.20 Hz, Ar**H**), 7.28 (t, 1H, *J* = 7.81 Hz, Ar**H**), 6.84-6.76 (m, 2H, Ar**H**), 3.90 (bs, 1H, -N**H**CH), 3.79-3.72 (m, 1H, -NHC**H**), 2.36-2.22 (m, 1H, -C**H**$CH_3$), 2.05-1.38 (m,

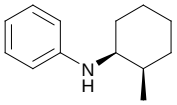
6H, -C**H**$_2$), 0.90 (d, 3H, *J* = 7.8 Hz, -CHC**H**$_3$); IR (film, cm$^{-1}$) 3440, 2960, 2877, 1615, 1516; MS (EI) *m/z*: 243 (M$^+$), 214 (16), 200 (100); HRMS (EI) *m/z* calcd for $C_{13}H_{16}NF_3$(M$^+$) 243.1235, found 243.1239; ee = 95% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^o$C; $T_{det}$ = 280 $^o$C; $T_{initial}$ = 30 $^o$C for 3 min; $T_{final}$ = 200 $^o$C; rate = 1$^o$/min; $t_R$ = 37.7 min (minor), $t_R$ = 38.2 min (major)].

**General procedure for asymmetric reductive amination of 2-methylcyclohexanone.**




*rac*-**6** **7**

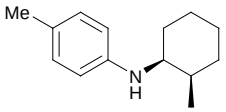
The procedure was the same as 2-methylcyclopentanone.




**7a**

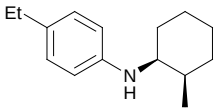
***cis*-*N*-(phenyl)-2-methylcyclohexaneamine (7a)**. Prepared according to the general procedure from 2-methylcyclohexanone (121 μL, 1.0 mmol), aniline (120 μL, 1.3 mmol) and Pd[(*R*)-BINAP]Br$_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^o$C for 24 h, to provide the title compound as colorless oil (91%). %). $^1$H NMR (300 MHz, $CDCl_3$) δ 7.18-7.13 (m, 2H, Ar**H**), 6.67-6.56 (m, 3H, Ar**H**), 3.64 (bs, 1H, -N**H**CH), 3.52-3.47 (m, 1H, -NHC**H**), 2.06-1.98 (m, 1H, -C**H**CH$_3$), 1.80-1.35 (m, 8H, -C**H**$_2$), 0.92 (d, 3H, *J* = 6.9 Hz, -CHC**H**$_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 147.6, 129.2, 116.5, 113.1, 53.1 (-NH**C**H), 33.0 (-**C**HCH$_3$), 30.3 (-**C**H$_2$), 28.5 (-**C**H$_2$), 22.9 (-**C**H$_2$), 22.7 (-**C**H$_2$), 15.4 (-CH**C**H$_3$); IR (film, cm$^{-1}$) 3415, 2926, 2854, 1601, 1504; MS (EI) *m/z*: 189 (M$^+$), 160 (4), 146 (27), 132 (100); HRMS (EI) *m/z* calcd for $C_{13}H_{19}N$(M$^+$) 189.1517, found 189.1520; $[\alpha]^{20}_D$ -13.3 (c 0.18, $CHCl_3$); ee = 79% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $t_{inj}$ = 280$^o$; $t_{initial}$ = 30$^o$ for 3 min; rate = 1.2$^o$/min; $t_R$ = 39.3 min (minor), $t_R$ = 40.3 min (major)]. The relative stereochemistry of the product was assigned by comparison of $^1$H NMR spectral data reported in the literatura for non chiral compound.[5]

With Pd[(*R*)-Tol-BINAP]Br$_2$, **1e**, $[\alpha]^{20}_D$ -11.5 (c 0.53, $CHCl_3$); ee = 73% by GC-MS (EI) [chiral column: Cyclodex-β; conditions $T_{inj}$ = 280 $^o$C; $T_{det}$ = 280 $^o$C; $T_{initial}$ = 30 $^o$C for 3 min; $T_{final}$ = 200 $^o$C; rate = 1.2$^o$/min; $t_R$ = 39.4 min (minor), $t_R$ = 40.3 min (major)].



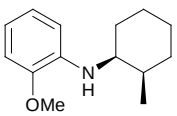
**7b**

***cis*-*N*-(4-methylphenyl)-2-methylcyclohexaneamine (7b)**. Prepared according to the general procedure from 2-methylcyclohexanone (121 μL, 1.0 mmol), *p*-toluidine (139 mg, 1.3 mmol) and Pd[(*S*)-BINAP]$Br_2$, **1d**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as light yellow oil (91%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 6.98 (d, 2H, *J* = 8.2 Hz, Ar**H**), 6.55 (d, 1H, *J* = 8.5 Hz, Ar**H**), 3.49-3.38 (m, 2H, -N**H**CH + -NHC**H**), 2.24 (s, 3H, -C**H**$_3$), 2.06-2.00 (m, 1H, -C**H**$CH_3$), 1.80-1.35 (m, 8H, -C**H**$_2$), 0.92 (d, 3H, *J* = 7.1 Hz, -CHC**H**$_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 145.5, 129.8, 125.8, 113.4, 53.5 (-NH**C**H), 33.1 (-**C**$HCH_3$), 30.5 (-**C**$H_2$), 28.7 (-**C**$H_2$), 23.0 (-2**C**$H_2$), 20.4 (-**C**$H_3$), 15.5 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3412, 2924, 2855, 1617, 1518; MS (EI) *m/z*: 203 ($M^+$), 174 (9), 160 (22), 146 (100); HRMS (EI) *m/z* calcd for $C_{14}H_{21}N(M^+)$ 203.1670, found 203.1674; $[\alpha]^{20}_D$ +7.6 (c 0.5, $CHCl_3$); ee = 75% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1.0$^o$/min; $t_R$ = 138.6 min (minor), $t_R$ = 139.9 min (major)].



**7c**

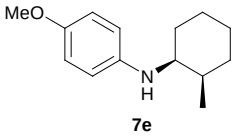
***cis*-*N*-(4-ethylphenyl)-2-methylcyclohexaneamine (7c)**. Prepared according to the general procedure from 2-methylcyclohexanone (121 μL, 1.0 mmol), *p*-ethylaniline (160 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as yellow oil (74%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.02 (d, 2H, *J* = 8.4 Hz, Ar**H**), 6.58 (d, 1H, *J* = 8.7 Hz, Ar**H**), 3.51-3.47 (m, 2H, -N**H**CH + -NHC**H**), 2.56 (q, 2H, *J* = 7.6 Hz, -C**H**$_2CH_3$), 2.08-1.99 (m, 1H, -C**H**$CH_3$), 1.80-1.31 (m, 8H, -C**H**$_2$), 1.2 (t, 3H, *J* = 7.5 Hz, -$CH_2$C**H**$_3$), 0.94 (d, 3H, *J* = 6.9 Hz, -CHC**H**$_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 145.6, 132.4, 128.5, 113.2, 53.4 (-NH**C**H), 33.1 (-**C**$HCH_3$), 30.3 (-**C**$H_2$), 28.6 (-**C**$H_2$), 27.8 (-**C**$H_2CH_3$), 22.9 (-**C**$H_2$), 22.7 (-**C**$H_2$), 15.8 (-**C**$H_2CH_3$), 15.5 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3418, 2926, 2855, 1615, 1517; MS (EI) *m/z*: 217 ($M^+$), 188 (5), 174 (29), 160 (100); HRMS (EI) *m/z* calcd for $C_{15}H_{23}N(M^+)$ 217.1830, found 217.1832; $[\alpha]^{20}_D$ -8.8 (c 0.42, $CHCl_3$); ee = 62% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $t_{inj}$ = 280$^o$; $t_{initial}$ = 30$^o$ for 3 min; rate = 1.2$^o$/min; $t_R$ = 49.8 min (minor), $t_R$ = 51.4 min (major)].



**7d**

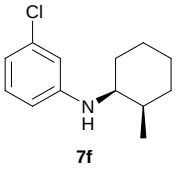
***cis*-*N*-(2-methoxyphenyl)-2-methylcyclohexaneamine (7d)**. Prepared according to the general procedure from 2-methylcyclohexanone (121 μL, 1.0 mmol), *o*-anisidine (150 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (83%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 6.87 (td, 1H, *J* = 1.3, 7.15 Hz, Ar**H**), 6.79 (d, 1H, *J* = 7.7 Hz, Ar**H**), 6.66-6.61 (m, 2H, Ar**H**), 4.34 (bs, 1H, -N**H**CH), 3.87 (s, 3H, -OMe), 3.55-3.51 (m, 1H, -NHC**H**), 2.10-2.00 (m, 1H, -C**H**$CH_3$), 1.76-1.38 (m, 8H, -C**H**$_2$), 0.95 (d, 3H, *J* = 6.8 Hz, -CHC**H**$_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 146.9, 137.8, 121.3, 115.5, 110.2,

109.6, 55.6 (-O**C**$H_3$), 53.0 (-NH**C**H), 33.3 (-**C**HCH$_3$), 30.5 (-**C**$H_2$), 28.8 (-**C**$H_2$), 23.2 (-**C**$H_2$), 22.9 (-**C**$H_2$), 15.7 (-CH**C**$H_3$); IR (film, cm$^{-1}$) 3421, 2926, 2855, 1594, 1498; MS (EI) *m/z*: 219 ($M^+$), 190 (4), 176 (28), 162 (100); HRMS (EI) *m/z* calcd for $C_{14}H_{21}NO(M^+)$ 219.1623, found 219.1619; $[\alpha]^{20}_D$ -7.8 (c 0.54, $CHCl_3$); ee = 98% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1.0°/min; $t_R$ = 156.1 min (minor), $t_R$ = 157.5 min (major)].



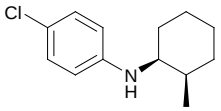
**7e**

***cis-N*-(4-methoxyphenyl)-2-methylcyclohexaneamine (7e)**. Prepared according to the general procedure from 2-methylcyclohexanone (121 μL, 1.0 mmol), *p*-anisidine (160 mg, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 °C for 24 h, to provide the title compound as light yellow oil (85%). $^1$H NMR (300 MHz, $CDCl_3$) δ 6.76 (d, 2H, *J* = 8.7 Hz, Ar**H**), 6.58 (d, 2H, *J* = 8.7 Hz, Ar**H**), 4.14 (bs, 1H, -N**H**CH), 3.74 (s, 3H, -OMe), 3.43-3.38 (m, 1H, -NHC**H**), 2.06-1.97 (m, 1H, -C**H**CH$_3$), 1.65-1.35 (m, 8H, -C**H**$_2$), 0.91 (d, 3H, *J* = 7.2 Hz, -CHC**H**$_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 151.6, 141.9, 114.9, 114.6, 55.8 (-O**C**$H_3$), 54.2 (-NH**C**H), 33.0 (-**C**HCH$_3$), 30.4 (-**C**$H_2$), 28.5 (-**C**$H_2$), 22.9 (-**C**$H_2$), 22.8 (-**C**$H_2$), 15.2 (-CH**C**$H_3$); IR (film, cm$^{-1}$) 3393, 2925, 2853, 1617, 1512; MS (EI) *m/z*: 219 ($M^+$), 190 (4), 176 (22), 162 (100); HRMS (EI) *m/z* calcd for $C_{14}H_{21}NO(M^+)$ 219.1623, found 219.1625; $[\alpha]^{20}_D$ -9.3 (c 0.4, $CHCl_3$); ee = >99% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1.0°/min; $t_R$ = 143.8 min (minor), $t_R$ = 144.9 min (major)].



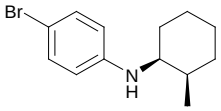
**7f**

***cis-N*-(3-chlorophenyl)-2-methylcyclohexaneamine (7f)**. Prepared according to the general procedure from 2-methylcyclohexanone (121 μL, 1.0 mmol), *m*-chloroaniline (136 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 °C for 24 h, to provide the title compound as colorless oil (90%). $^1$H NMR (300 MHz, $CDCl_3$) δ 7.07-7.00 (m, 1H, Ar**H**), 6.62-6.57 (m, 2H, Ar**H**), 6.46 (dd, 1H, *J* = 1.0, 8.3 Hz Ar**H**), 3.70 (bs, 1H, -N**H**CH), 3.48-3.44 (m, 1H, -NHC**H**), 2.04-1.96 (m, 1H, -C**H**CH$_3$), 1.83-1.35 (m, 8H, -C**H**$_2$), 0.91 (d, 3H, *J* = 6.6 Hz, -CHC**H**$_3$); $^{13}$C NMR (75 MHz, $CDCl_3$) δ 148.8, 134.9, 130.0, 116.2, 112.4, 111.3, 53.0 (-NH**C**H), 32.9 (-**C**HCH$_3$), 30.2 (-**C**$H_2$), 28.5 (-**C**$H_2$), 22.9 (-**C**$H_2$), 22.6 (-**C**$H_2$), 15.6 (-CH**C**$H_3$); IR (film, cm$^{-1}$) 3425, 2927, 2855, 1597, 1502; MS (EI) *m/z*: 223 ($M^+$), 194 (5), 180 (32), 166 (100); HRMS (EI) *m/z* calcd for $C_{13}H_{18}NCl(M^+)$ 223.1128, found 223.1123; $[\alpha]^{20}_D$ -9.5 (c 0.46, $CHCl_3$); ee = 93% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 °C; $T_{det}$ = 280 °C; $T_{initial}$ = 30 °C for 3 min; $T_{final}$ = 200 °C; rate = 1.2°/min; $t_R$ = 28.4 min (minor), $t_R$ = 29.7 min (major)].



7g

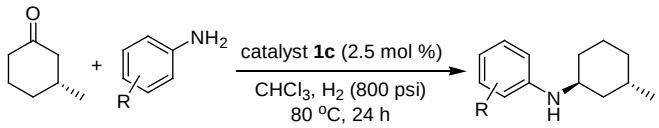
***cis*-*N*-(4-chlorophenyl)-2-methylcyclohexaneamine (7g)**. Prepared according to the general procedure from 2-methylcyclohexanone (121 μL, 1.0 mmol), *p*-chloroaniline (165 mg, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (82%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.09 (d, 2H, *J* = 8.8 Hz, Ar**H**), 6.51 (d, 2H, *J* = 8.8 Hz, Ar**H**), 3.59 (bs, 1H, -N**H**CH), 3.46-3.41 (m, 1H, -NHC**H**), 2.04-1.97 (m, 1H, -C**H**$CH_3$), 1.75-1.36 (m, 8H, -C$\mathbf{H}_2$), 0.90 (d, 3H, *J* = 7.2 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 146.4, 129.1, 121.0, 114.2, 53.4 (-NH**C**H), 33.1 (-**C**$HCH_3$), 30.3 (-**C**$H_2$), 28.6 (-**C**$H_2$), 23.0 (-**C**$H_2$), 22.8 (-**C**$H_2$), 15.5 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3423, 2927, 2856, 1599, 1500; MS (EI) *m/z*: 223 ($M^+$), 194 (6), 180 (37), 166 (100); HRMS (EI) *m/z* calcd for $C_{13}H_{18}NCl(M^+)$ 223.1128, found 223.1135; $[\alpha]^{20}_D$ -6.4 (c 0.5, $CHCl_3$); ee = 89% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1.0$^o$/min; $t_R$ = 157.1 min (minor), $t_R$ = 157.8 min (major)].



7h

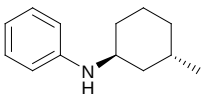
***cis*-*N*-(4-bromophenyl)-2-methylcyclohexaneamine (7h)**.Prepared according to the general procedure from 2-methylcyclohexanone (121 μL, 1.0 mmol), *p*-bromoaniline (222 mg, 1.3 mmol) and Pd[(*S*)-BINAP]$Br_2$, **1d**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (71%). $^1H$ NMR (300 MHz, $CDCl_3$) δ 7.21 (d, 2H, *J* = 8.8 Hz, Ar**H**), 6.48 (d, 2H, *J* = 8.8 Hz, Ar**H**), 3.67 (bs, 1H, -N**H**CH), 3.46-3.41 (m, 1H, -NHC**H**), 2.02-1.97 (m, 1H, -C**H**$CH_3$), 1.77-1.22 (m, 8H, -C$\mathbf{H}_2$), 0.90 (d, 3H, *J* = 7.2 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (75 MHz, $CDCl_3$) δ 146.8, 132.0, 114.7, 108.0, 53.4 (-NH**C**H), 33.0 (-**C**$HCH_3$), 30.3 (-**C**$H_2$), 28.6 (-**C**$H_2$), 23.0 (-**C**$H_2$), 22.8 (-**C**$H_2$), 15.5 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3434, 2926, 2854, 1600, 1515; MS (EI) *m/z*: 267 ($M^+$), 240 (2), 226 (19), 212 (100); HRMS (EI) *m/z* calcd for $C_{13}H_{18}NBr(M^+)$ 267.0623, found 267.0631; $[\alpha]^{20}_D$ +6.5 (c 0.52, $CHCl_3$); ee = 83% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1.0$^o$/min; $t_R$ = 167.3 min (minor), $t_R$ = 168.0 min (major)].

**General procedure for asymmetric reductive amination of (*R*)-3-methylcyclohexanone.**




*R*-**8** **9**

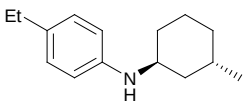
The procedure was the same as 2-methylcyclopentanone.



9a

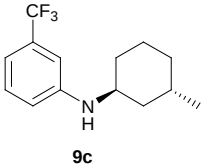
***trans*-*N*-(phenyl)-3-methylcyclohexaneamine (9a)**. Prepared according to the general procedure from (*R*)-(+)-3-methylcyclohexanone (122 μL, 1.0 mmol), aniline (120 μL, 1.3 mmol) and Pd[(*S*)-BINAP]$Br_2$, **1d**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (87%). $^1H$ NMR (500 MHz, $CDCl_3$) δ 7.17-7.14 (m, 2H, Ar**H**), 6.68 (t, 1H, *J* = 7.2 Hz, Ar**H**), 6.63 (d, 2H, *J* = 8.0 Hz, Ar**H**), 4.21 (bs, 1H, -N**H**CH), 3.68-3.64 (m, 1H, -NHC**H**), 1.78-1.50 (m, 7H, -C**H**$CH_3$ + -C$\mathbf{H}_2$), 1.38-1.33 (m, 1H, -C$\mathbf{H}_2$), 1.09-1.01 (m, 1H, -C$\mathbf{H}_2$), 0.91 (d, 3H, *J* = 6.5 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (125 MHz, $CDCl_3$) δ 146.8, 129.2, 117.2, 113.6, 48.1 (-NH**C**H), 38.6 (-**C**$H_2$), 33.8 (-**C**$H_2$), 30.3 (-**C**$H_2$), 27.1 (-**C**$HCH_3$), 21.5 (-CH**C**$H_3$), 20.5 (-**C**$H_2$); IR (film, $cm^{-1}$) 3415, 2923, 2850, 1602, 1504; MS (EI) *m/z*: 189 ($M^+$), 174 (8), 160 (5), 146 (100), 132 (87); HRMS (EI) *m/z* calcd for $C_{13}H_{19}N(M^+)$ 189.1517, found 189.1514; $[\alpha]^{20}_D$ -13.3 (c 0.48, $CHCl_3$); ee = 91% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 132.7 min (major), $t_R$ = 133.2 min (minor)].

The relative configuration was confirmed by NOE experiment and compared with a similar reported compound.[6]



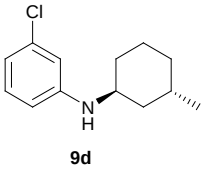
9b

***trans*-*N*-(*p*-ethylphenyl)-3-methylcyclohexaneamine (9b)**. Prepared according to the general procedure from (*R*)-(+)-3-methylcyclohexanone (122 μL, 1.0 mmol), *p*-ethylaniline (160 μL, 1.3 mmol) and Pd[(*S*)-BINAP]$Br_2$, **1e**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as light yellow oil (90%). $^1H$ NMR (500 MHz, $CDCl_3$) δ 6.99 (d, 2H, *J* = 8.5 Hz, Ar**H**), 6.54 (d, 2H, *J* = 8.5 Hz, Ar**H**), 4.02 (bs, 1H, -N**H**CH), 3.65-3.62 (m, 1H, -NHC**H**), 2.52 (q, 2H, *J* = 7.5 Hz, -C$\mathbf{H}_2CH_3$), 1.76-1.49 (m, 7H, -C**H**$CH_3$ + -C$\mathbf{H}_2$), 1.36-1.30 (m, 1H, -C$\mathbf{H}_2$), 1.18 (t, 3H, *J* = 7.5 Hz, -C$\mathbf{H}_2CH_3$), 1.07-1.00 (m, 1H, -C$\mathbf{H}_2$), 0.90 (d, 3H, *J* = 6.5 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (125 MHz, $CDCl_3$) δ 145.3, 132.6, 128.5, 113.2, 47.8 (-NH**C**H), 38.9 (-**C**$H_2$), 33.9 (-**C**$H_2$), 30.5 (-**C**$H_2$), 27.8 (-**C**$H_2CH_3$), 27.1 (-**C**$HCH_3$), 21.6 (-CH**C**$H_3$), 20.5 (-**C**$H_2$), 15.8 (-$CH_2$**C**$H_3$); IR (film, $cm^{-1}$) 3411, 2924, 2864, 1615, 1517; MS (EI) *m/z*: 217 ($M^+$), 202 (9), 173 (3), 160 (100), 146 (82); HRMS (EI) *m/z* calcd for $C_{15}H_{23}N(M^+)$ 217.1830, found 217.1829; $[\alpha]^{20}_D$ -8.4 (c 0.5, $CHCl_3$); ee = 95% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 149.8 min (major), $t_R$ = 150.5 min (minor)].



9c

***trans*-*N*-(3-trifluoromethylphenyl)-3-methylcyclohexaneamine (9c)**. Prepared according to the general procedure from (*R*)-(+)-3-methylcyclohexanone (122 μL, 1.0 mmol), *m*-trifluoromethyl aniline (162 μL, 1.3 mmol) and Pd[(*S*)-BINAP]$Br_2$, **1d**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (84%). $^1H$ NMR (500 MHz, $CDCl_3$) δ 7.21 (t, 1H, *J* = 8.0 Hz, Ar**H**), 6.87 (d, 1H, *J* = 7.5 Hz, Ar**H**), 6.77 (s, 1H, Ar**H**), 6.70 (d, 1H, *J* = 8.0 Hz, Ar**H**), 4.06 (bs, 1H, -N**H**CH), 3.70-3.66 (m, 1H, -NHC**H**), 1.76-1.45 (m, 7H, -C**H**$CH_3$ + -C$\mathbf{H}_2$), 1.38-1.32 (m, 1H, -C$\mathbf{H}_2$), 1.09-1.01 (m, 1H, -C$\mathbf{H}_2$), 0.92 (d, 3H, *J* = 7.0 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (125 MHz, $CDCl_3$) δ 147.4, 131.6 (q, *J* = 31.4 Hz), 129.6, 124.6 (d, *J* = 271.0 Hz), 115.9, 113.1 (q, *J* = 3.7 Hz), 109.1 (q, *J* = 3.8 Hz), 47.6 (-NH**C**H), 38.6 (-**C**$H_2$), 33.8 (-**C**$H_2$), 30.2 (-**C**$H_2$), 27.1 (-**C**$HCH_3$), 21.6 (-CH**C**$H_3$), 20.4 (-**C**$H_2$); IR (film, $cm^{-1}$) 3443, 2926, 2857, 1614, 1515; MS (EI) *m/z*: 257 ($M^+$), 242 (7), 214 (100), 228 (2), 200 (74); HRMS (EI) *m/z* calcd for $C_{14}H_{18}NF_3(M^+)$ 257.1391, found 257.1389; $[\alpha]^{20}_D$ -13.1 (c 0.48, $CHCl_3$); ee = >99% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 135.8 min (major)].

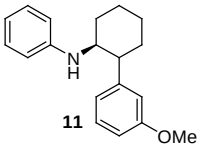


9d

***trans*-*N*-(3-chlorophenyl)-3-methylcyclohexaneamine (9d)**. Prepared according to the general procedure from (*R*)-(+)-3-methylcyclohexanone (122 μL, 1.0 mmol), *m*-chloroaniline (136 μL, 1.3 mmol) and Pd[(*S*)-BINAP]$Br_2$, **1d**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (89%). $^1H$ NMR (500 MHz, $CDCl_3$) δ 7.02 (t, 1H, *J* = 8.2 Hz, Ar**H**), 6.62 (d, 1H, *J* = 8.0 Hz, Ar**H**), 6.54 (s, 1H, Ar**H**), 6.43 (d, 1H, *J* = 8.5 Hz, Ar**H**), 3.90 (bs, 1H, -N**H**CH), 3.63-3.60 (m, 1H, -NHC**H**), 1.72-1.43 (m, 7H, -C**H**$CH_3$ + -C$\mathbf{H}_2$), 1.35-1.29 (m, 1H, -C$\mathbf{H}_2$), 1.07-0.99 (m, 1H, -C$\mathbf{H}_2$), 0.91 (d, 3H, *J* = 6.5 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (125 MHz, $CDCl_3$) δ 148.4, 135.0, 130.1, 116.5, 112.5, 111.3, 47.6 (-NH**C**H), 38.6 (-**C**$H_2$), 33.8 (-**C**$H_2$), 30.2 (-**C**$H_2$), 27.1 (-**C**$HCH_3$), 21.5 (-CH**C**$H_3$), 20.4 (-**C**$H_2$); IR (film, $cm^{-1}$) 3425, 2924, 2861, 1598, 1500; MS (EI) *m/z*: 223 ($M^+$), 208 (7), 180 (100), 166 (73), 153 (17); HRMS (EI) *m/z* calcd for $C_{13}H_{18}NCl(M^+)$ 223.1128, found 223.1125; $[\alpha]^{20}_D$ -12.7 (c 0.47, $CHCl_3$); $[\alpha]^{20}_D$ -20.3 (c 0.52, benzene); ee = >99% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 189.4 min (major)].

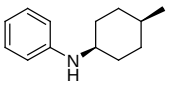
**General procedure for asymmetric reductive amination of other substituted rings.**

The procedure was the same as 2-methylcyclopentanone.



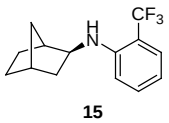
11

***cis-N-[2-(3-methoxyphenyl)cyclohexyl]benzenamine* (11)**. Prepared according to the general procedure from 2-(3-methoxyphenyl)cyclohexanone (185 μL, 1.0 mmol), aniline (120 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as light yellow oil (77%). $^1H$ NMR (500 MHz, $CDCl_3$) δ 7.16 (t, 1H, *J* = 7.5 Hz, Ar**H**), 7.03 (dd, 2H, *J* = 8.5 Hz, Ar**H**), 6.84 (dt, 1H, *J* = 8.5, 1.0 Hz, Ar**H**), 6.80 (t, 1H, *J* = 8.5 Hz, Ar**H**), 6.68 (dd, 1H, *J* = 8.0, 2.5 Hz, Ar**H**), 6.56 (tt, 1H, *J* = 7.5, 1.0 Hz, Ar**H**), 6.41(d, 2H, *J* = 8.0 Hz, Ar**H**), 3.82-3.79 (m, 1H, -NHC**H**), 3.70 (s, 3H, -OMe), 3.66 (bs, 1H, -N**H**CH), 2.98-2.92 (ddd, 1H, *J* = 11.5, 4.0, 4.0 Hz, -C**H**ArH), 2.12-2.08 (m, 1H, -C$\mathbf{H}_2$), 1.92-1.82 (m, 3H, -C$\mathbf{H}_2$), 1.61-1.42 (m, 4H, -C$\mathbf{H}_2$) ; $^{13}C$ NMR (125 MHz, $CDCl_3$) δ 159.5, 147.8, 145.4, 129.1, 128.9, 119.9, 116.6, 113.7, 113.2, 111.3, 55.0 (-OMe), 53.2 (-NH**C**H), 46.1 (-C**H**ArH), 30.1 (-**C**$H_2$), 25.87 (-**C**$H_2$), 25.83 (-**C**$H_2$), 20.3 (-**C**$H_2$); IR (film, $cm^{-1}$) 3424, 2926, 2861, 1598, 1502; MS (EI) *m/z*: 281 ($M^+$), 252 (3), 238 (18), 224 (3), 159 (19), 132 (100); HRMS (EI) *m/z* calcd for $C_{19}H_{23}ON$ ($M^+$) 281.1780, found 281.1784; $[\alpha]^{20}_D$ -36.6 (c 0.21, $CHCl_3$); ee = 80% by GC-MS (EI) [chiral column: Cyclodex-β; conditions: $T_{inj}$ = 280 $^oC$; $T_{det}$ = 280 $^oC$; $T_{initial}$ = 30 $^oC$ for 3 min; $T_{final}$ = 200 $^oC$; rate = 1$^o$/min; $t_R$ = 147.0 min (minor), $t_R$ = 148.4 min (major)].



13

***cis-N-(4-methylcyclohexyl)*aniline (13)**. Prepared according to the general procedure from 4-methylcyclohexanone (123 μL, 1.0 mmol), aniline (120 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as light yellow oil (74%). $^1H$ NMR (500 MHz, $CDCl_3$) δ 7.15 (dd, 2H, *J* = 8.5 Hz, Ar**H**), 6.65 (t, 1H, *J* = 8.0 Hz, Ar**H**), 6.61-6.58 (m, 2H, Ar**H**), 3.73 (bs, 1H, -N**H**CH), 3.56-3.52 (m, 1H, -NHC**H**), 1.77-1.72 (m, 2H, -C$\mathbf{H}_2$), 1.66-1.60 (m, 2H, -C$\mathbf{H}_2$), 1.57-1.51 (m, 3H, -C**H**$CH_3$+ -C$\mathbf{H}_2$), 0.92 (d, 3H, *J* = 6.5 Hz, -CHC$\mathbf{H}_3$); $^{13}C$ NMR (125 MHz, $CDCl_3$) δ 147.3, 129.2, 116.7, 113.1, 48.1 (-NH**C**H), 30.8 (-CH**C**$H_3$), 29.7 (-2**C**$H_2$), 29.2 (-2**C**$H_2$), 21.3 (-CH**C**$H_3$); IR (film, $cm^{-1}$) 3425, 2924, 2860, 1601, 1514; MS (EI) *m/z*: 189 ($M^+$), 157 (3), 144 (6), 132 (100), 118 (14), 106 (11); HRMS (EI) *m/z* calcd for $C_{13}H_{19}N(M^+)$ 189.1517, found 189.1515.



15

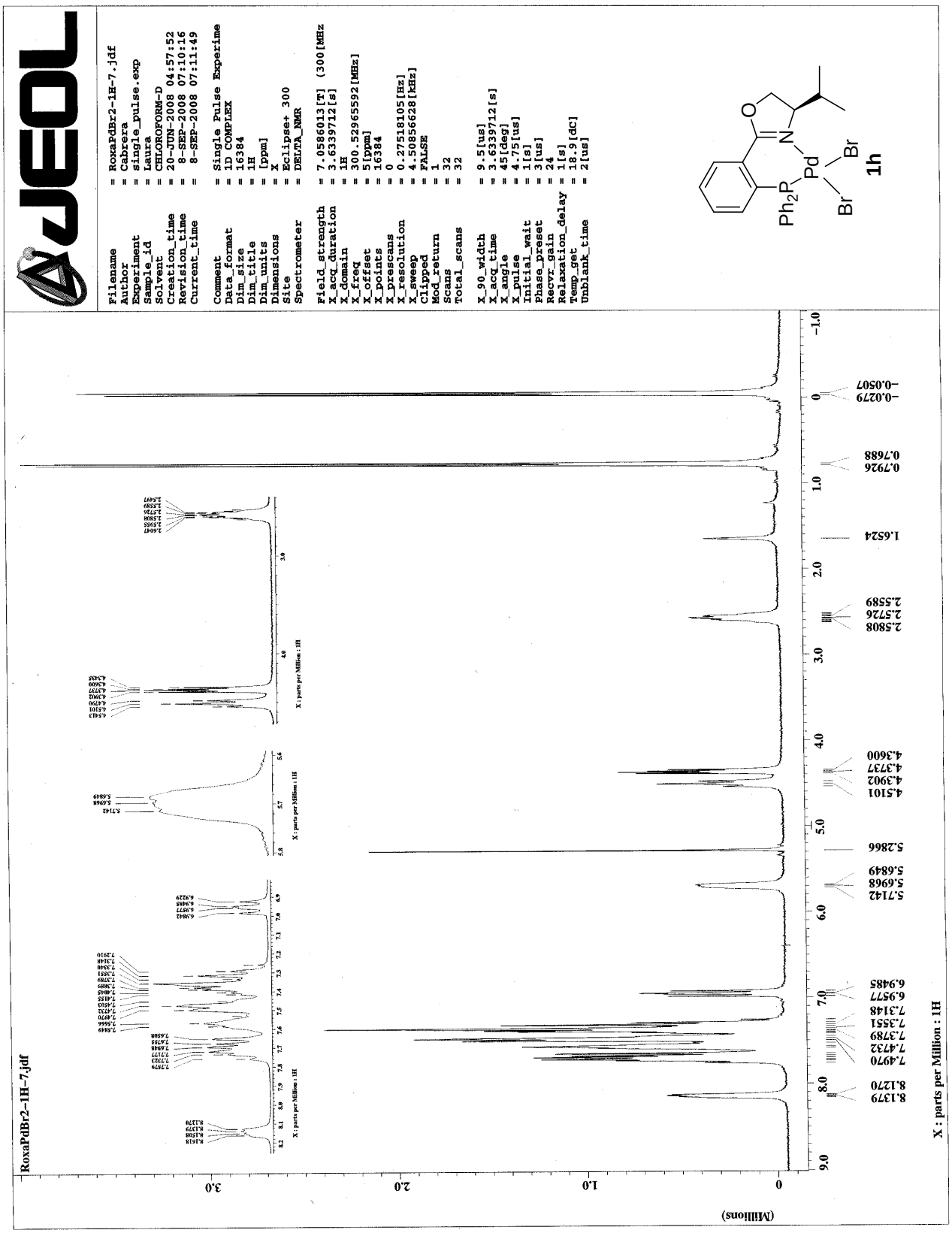
***N-(2-(trifluoromethyl)phenyl)bicyclo[2.2.1]heptan-2-amine* (15)**. Prepared according to the general procedure from norcamphor (110 mg, 1.0 mmol), *o*-trifluoromethyl aniline (163 μL, 1.3 mmol) and Pd[(*R*)-BINAP]$Br_2$, **1c**, (22 mg, 0.025 mmol) at 80 $^oC$ for 24 h, to provide the title compound as colorless oil (74%). $^1H$ NMR (500 MHz, $CDCl_3$) δ 7.41 (d, 1H, *J* = 8.0 Hz, Ar**H**), 7.32 (t, 1H, *J* = 7.5 Hz, Ar**H**), 6.71(d, 1H, *J* = 8.5 Hz, Ar**H**), 6.67 (t, 1H, *J* = 7.5 Hz, Ar**H**), 4.51 (bs, 1H, -N**H**CH), 3.76-3.73 (m, 1H, -NHC**H**), 2.55-2.53 (m, 1H, -C**H**), 2.32-2.26 (m,

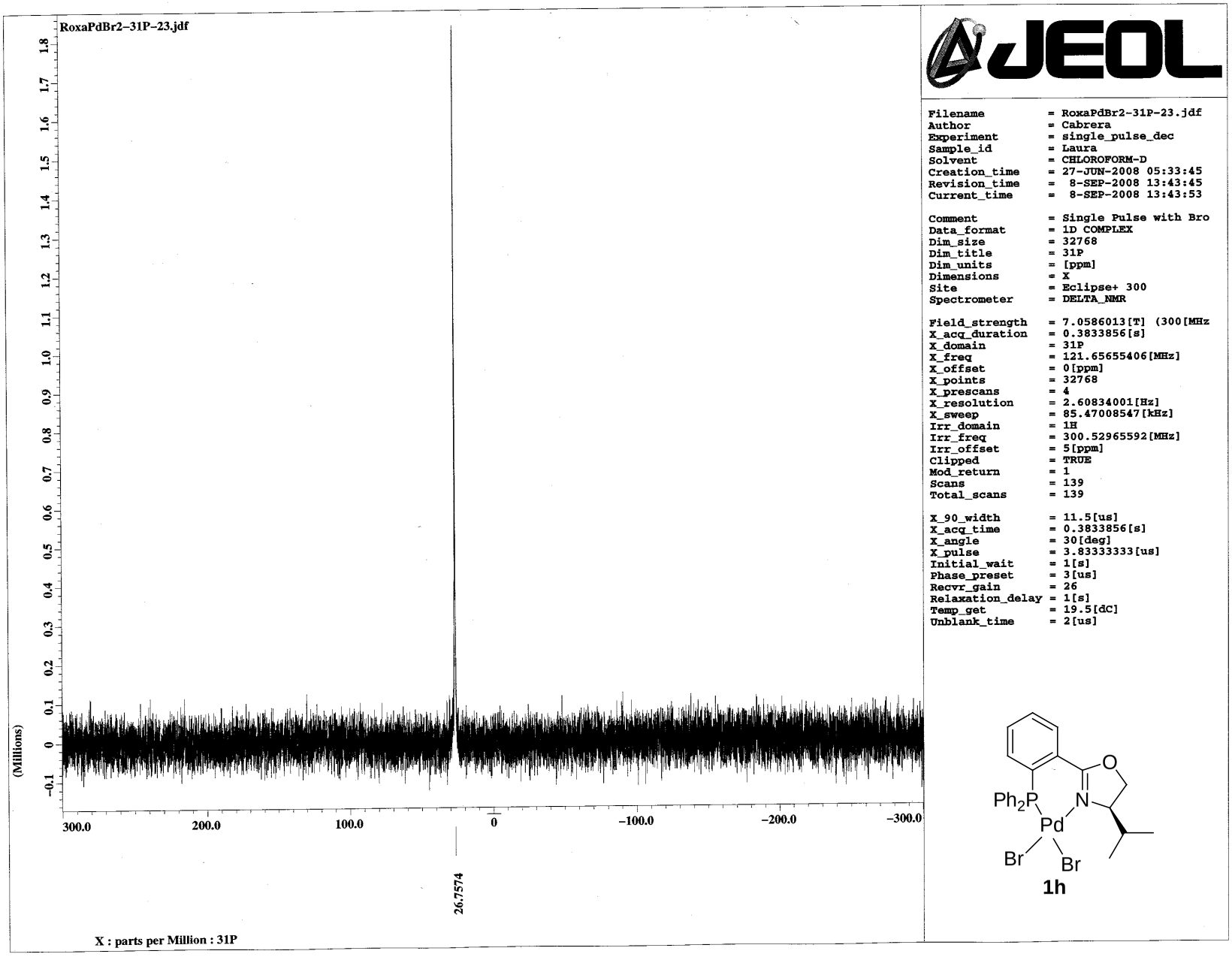
1H, -C**H**), 2.20-2.14 (m, 1H, -C**H**$_2$), 1.68-1.55 (m, 2H, -C**H**$_2$), 1.50-1.47 (m, 1H, -C**H**$_2$), 1.42-1.35 (m, 2H, -C**H**$_2$), 1.30-1.16 (m, 1H, -C**H**$_2$), 0.84-0.80 (m, 1H, -C**H**$_2$) ; $^{13}$C NMR (125 MHz, $CDCl_3$) δ 145.6, 132.9, 126.8 (q, *J* = 5.3 Hz), 119.8 (d = 272.1 Hz), 115.3, 113.2 (q, *J* = 29.4 Hz), 112.6, 54.2 (-NH**C**H), 39.7 (-**C**H), 38.9 (-**C**H$_2$), 38.1 (-**C**H$_2$), 36.8 (-**C**H), 29.7 (-**C**H$_2$), 21.2 (-**C**H$_2$); IR (film, cm$^{-1}$) 3476, 2956, 2873, 1615, 1519; MS (EI) *m/z*: 255 ($M^+$, 100), 226 (23), 214 (12), 200 (29), 187 (57), 174 (21); HRMS (EI) *m/z* calcd for $C_{14}H_{16}NF_3$($M^+$) 255.1235, found 235.1239; $[\alpha]^{20}_D$ -15.7 (c 0.50, $CHCl_3$); ee = 87% by HPLC (Daicel Chiracel OD-H, eluent hexane-iPrOH = 99 1, flow rate = 1mL min, $t_R$ = 6.58 min (major), $t_R$ = 7.58 min (minor)].

**References:**


1. M. A. Andrews, T. C. T. Chang, C. W. F. Cheng, T. J. Emge, K. P. Kelly, T. F. Koetzle, *J. Am. Chem. Soc*., 1984, **106**, 5913-5920.
2. F. Ozawa, A. Kubo, Y. Matsumoto, T. Hayashi, *Organometallics*, 1993, **12**, 4188-4196.
3. L. Rubio-Pérez, F. J. Pérez-Flores, P. Sharma, A. Cabrera, *Org. Lett*., 2009, **11**, 265-268.
4. A. J. Blacker, M. L. Clarke, M. S. Loft, M. F. Mahon, M. E. Humphries, J. M. J. Williams, *Chem. A. Eur. J.*, 2000, **6**, 353-360.
5. T. Suwa, E. Sugiyama, I. Shibata, A. Baba, *Synthesis*, 2000, 789-800.
6. J. Zhou, B. List, *J. Am. Chem. Soc*., 2007, **129**, 7498-7499.

**Scanned spectra of NMR and GC-MS (EI) or HPLC for all compounds:**

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0.5
0.4
0.3
0.2
0.1
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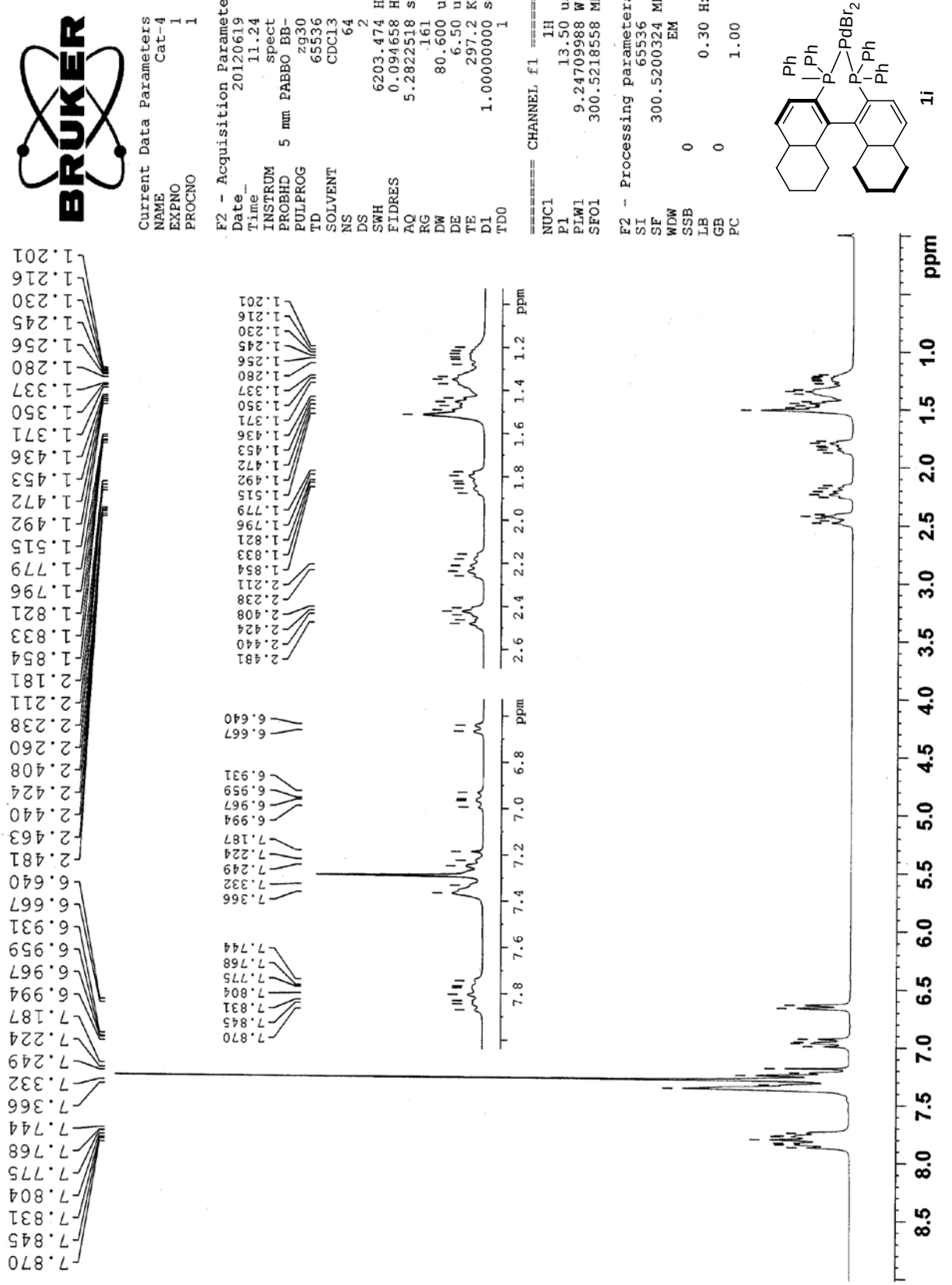
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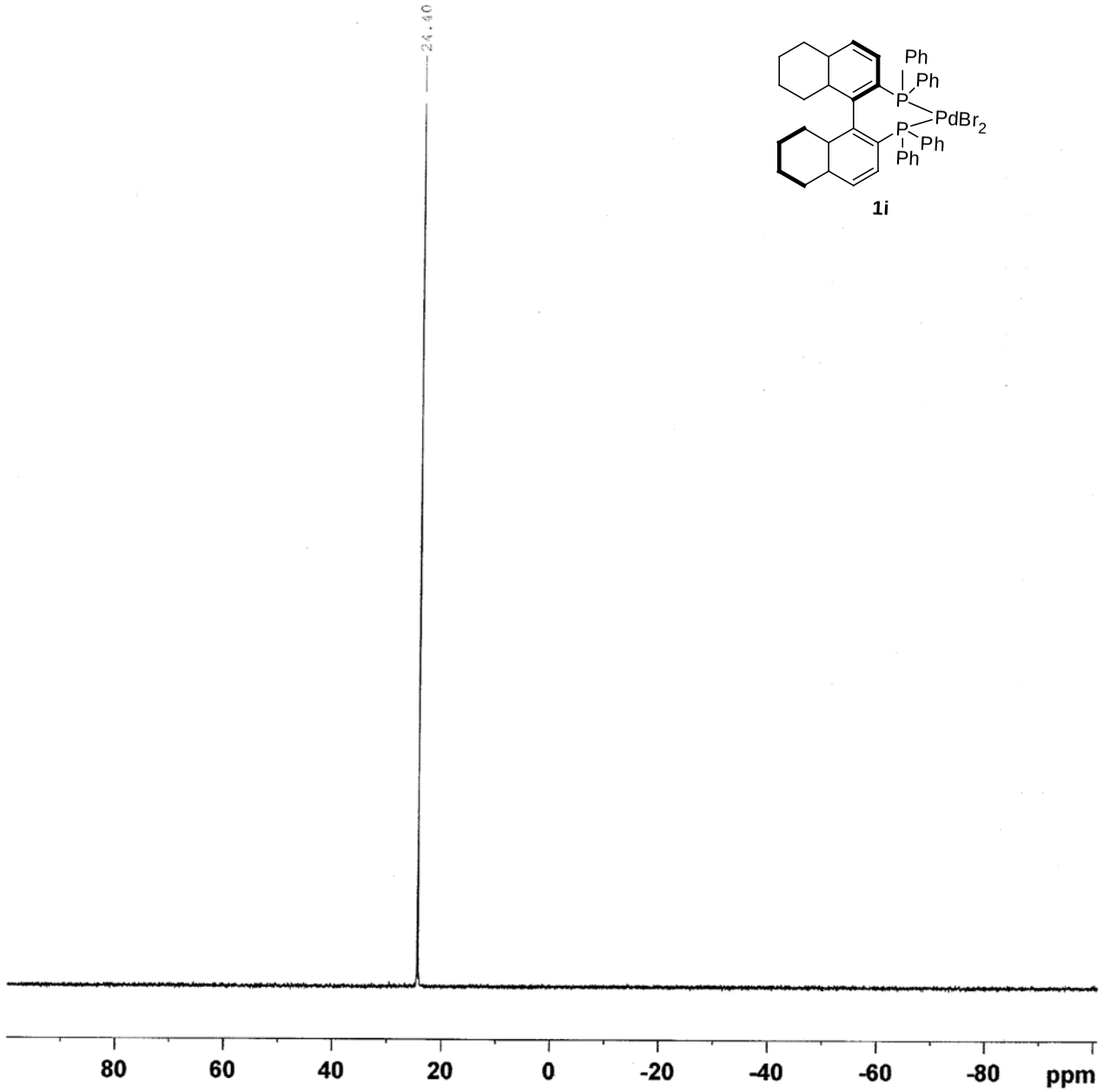





**1i**

BRUKER

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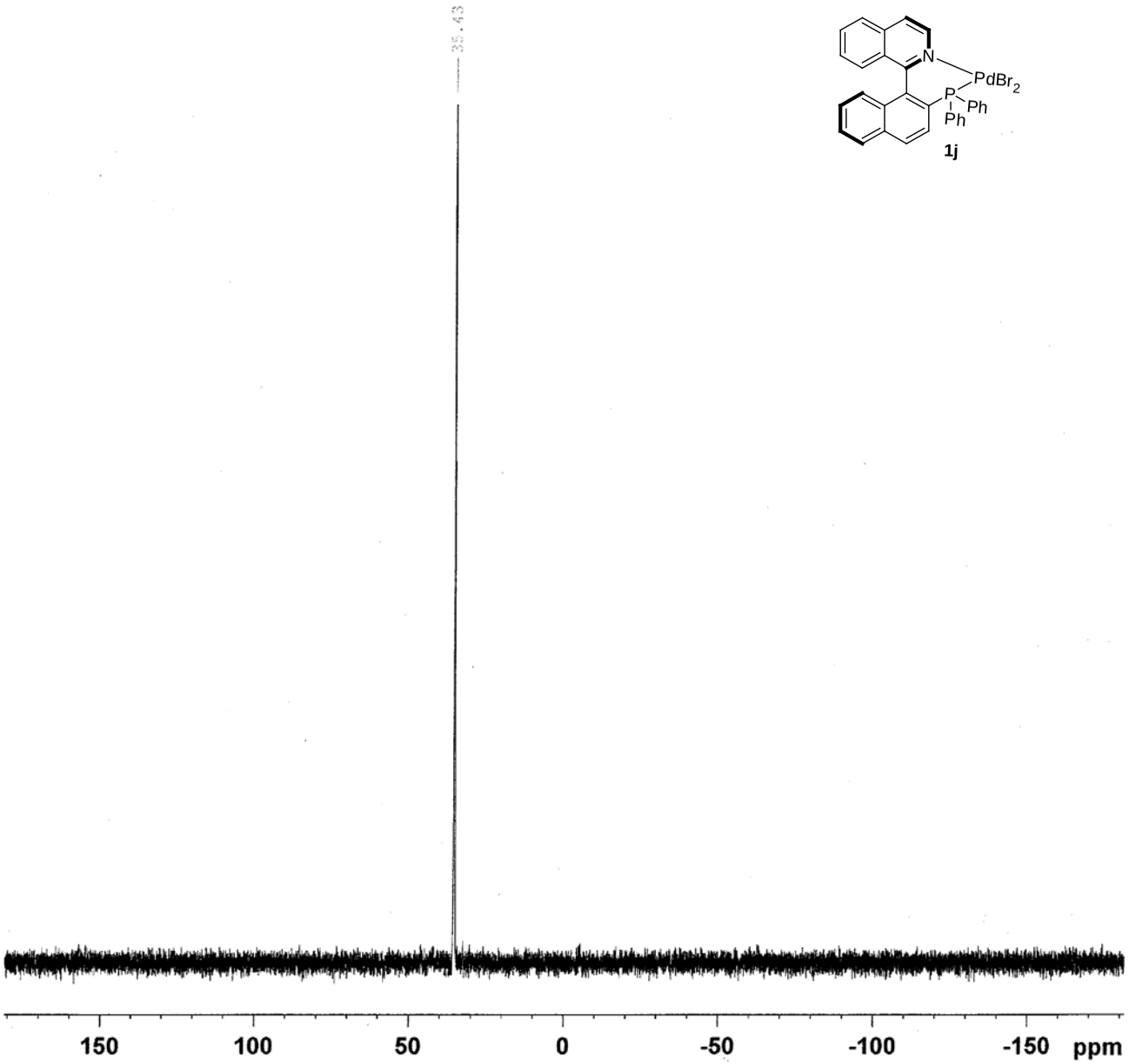





**1j**

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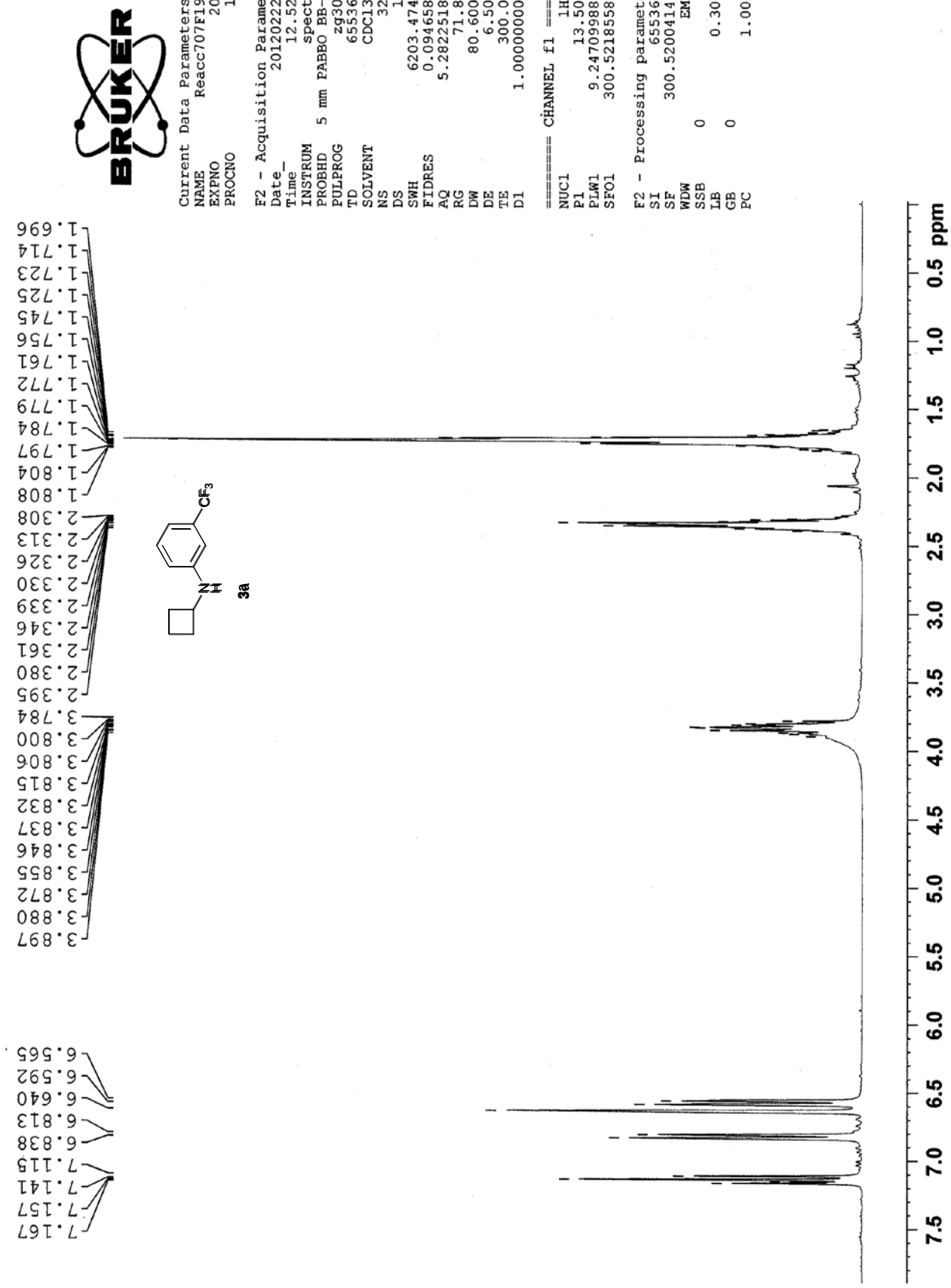
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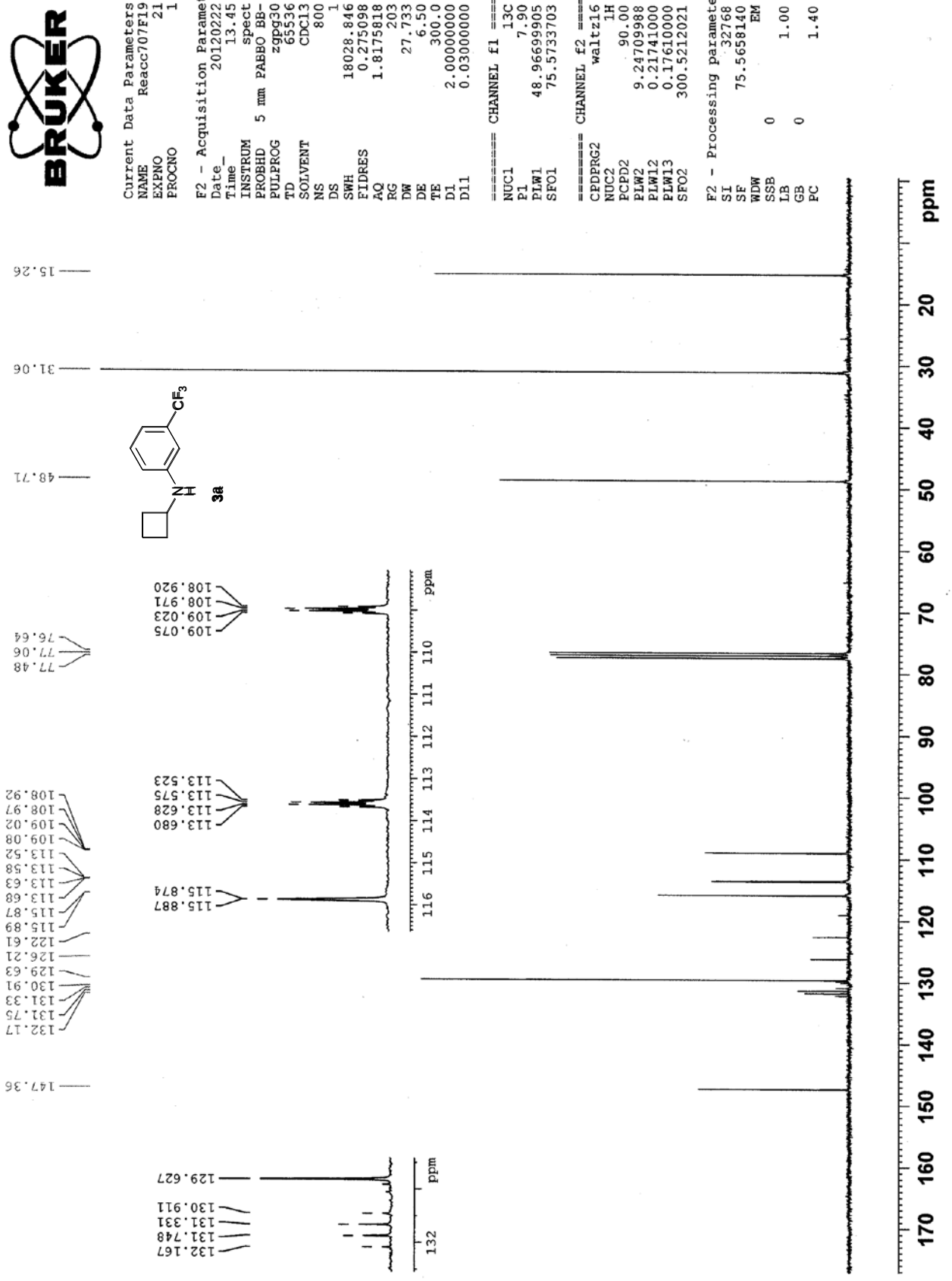
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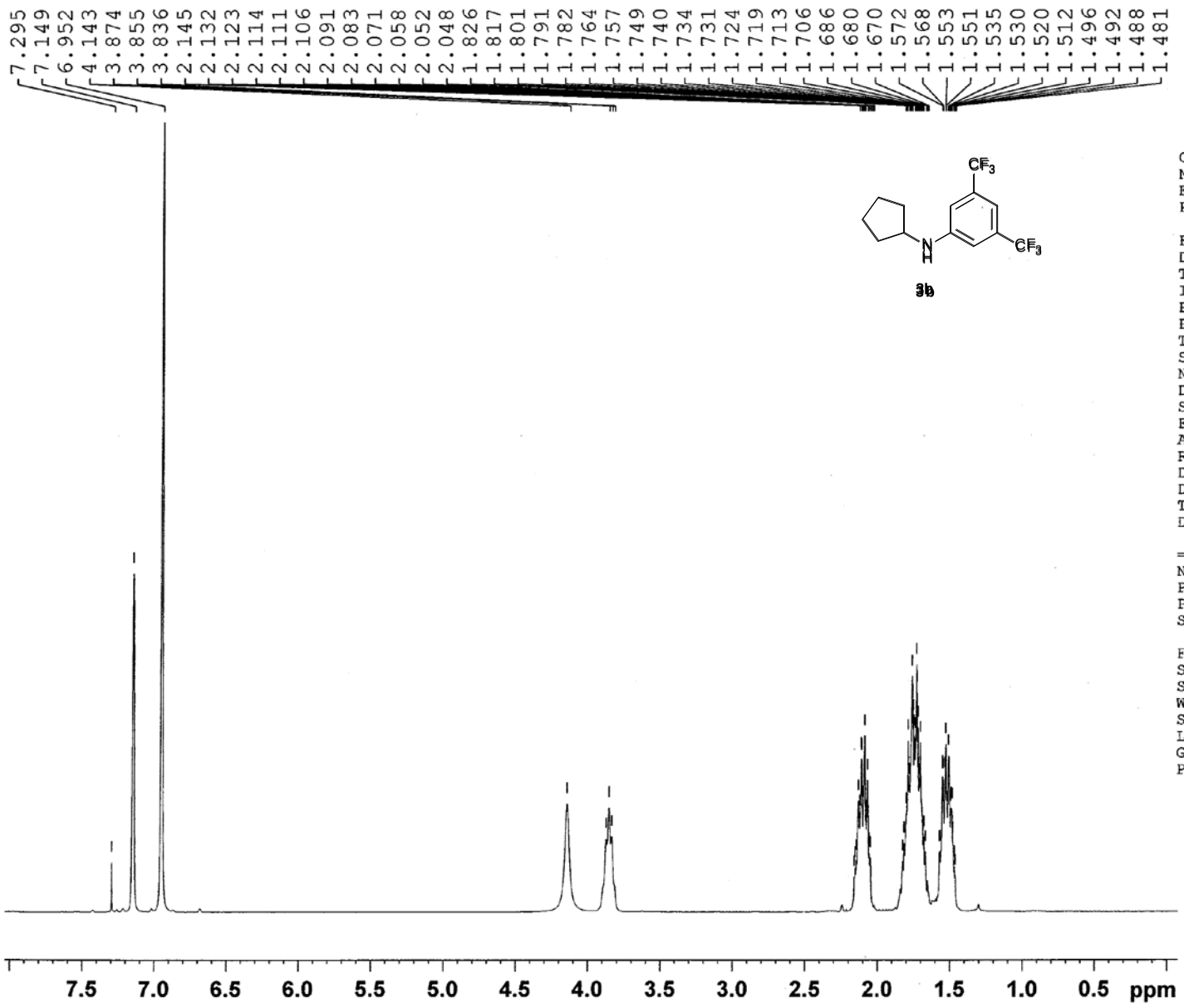


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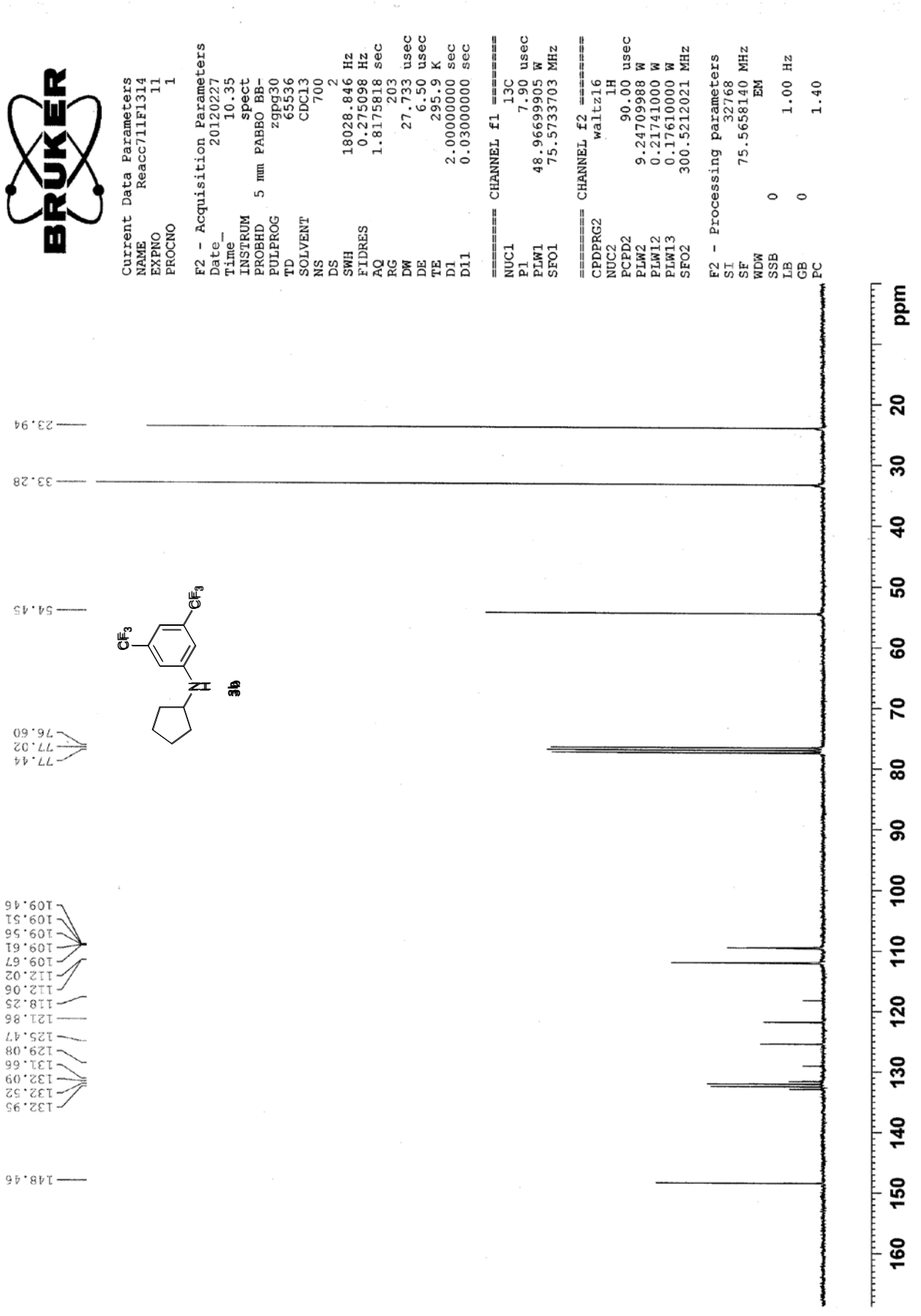
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Instituto de Quimica UNAM NZ
Dr. A. Cabrera/Laura R.
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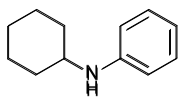

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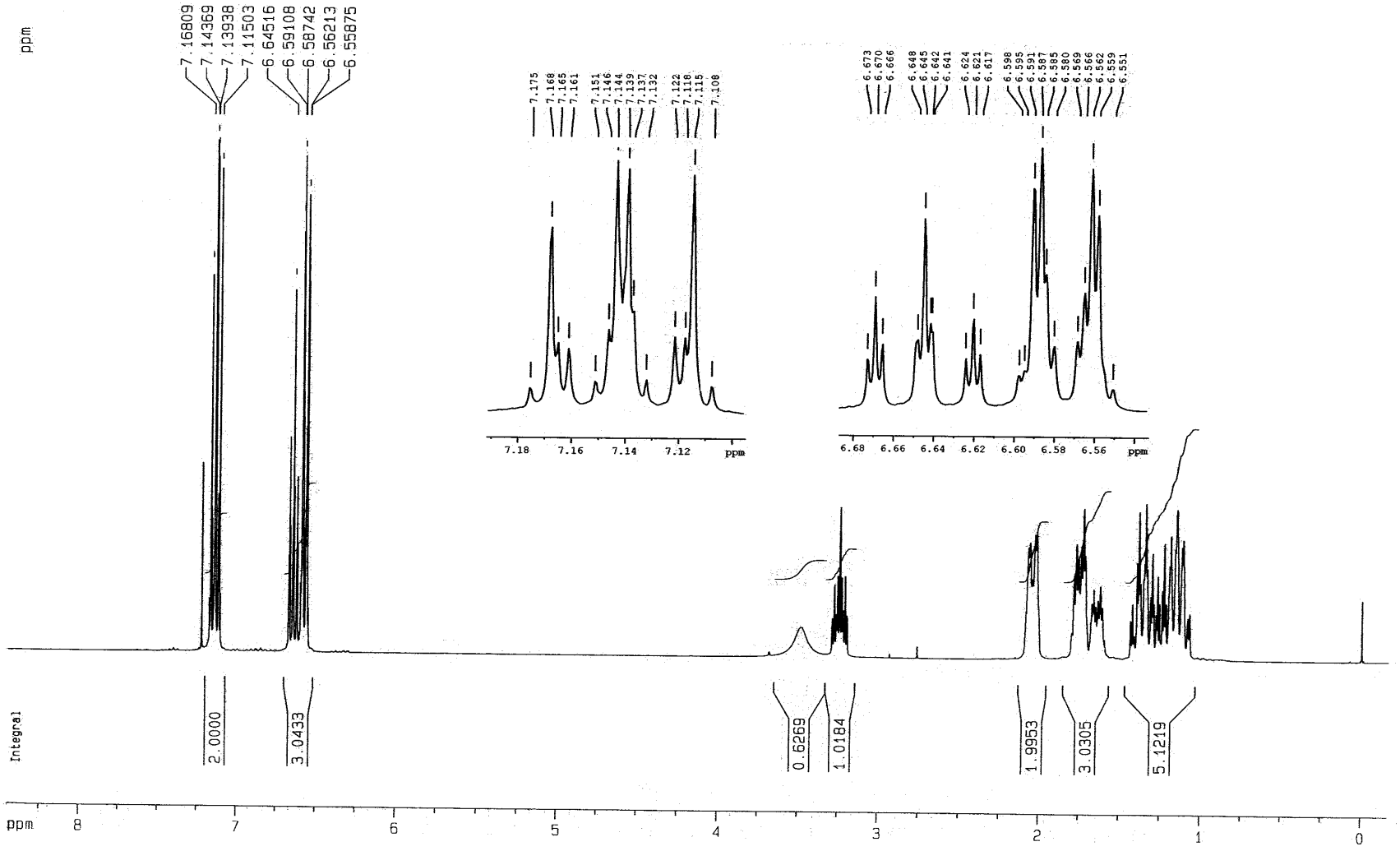

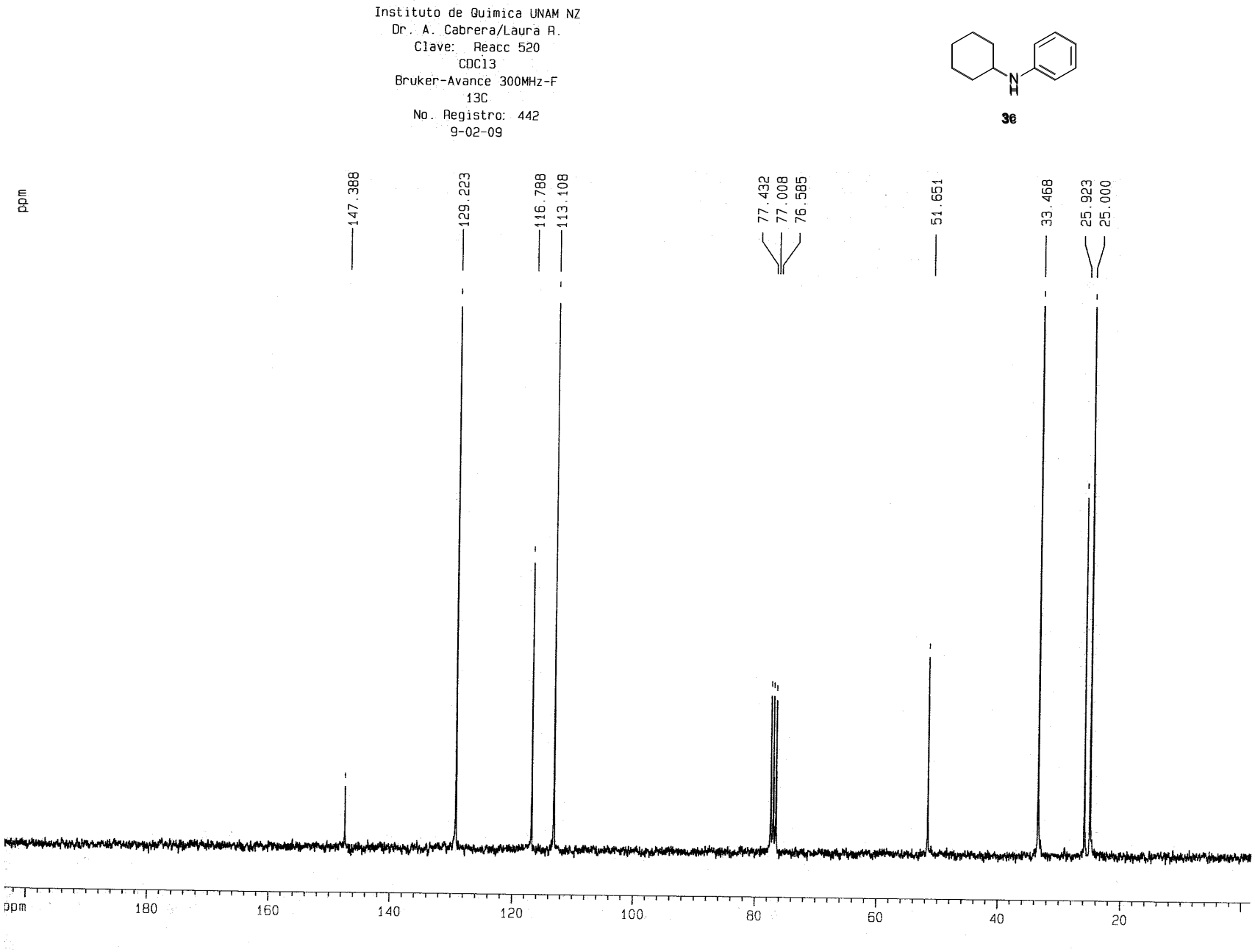
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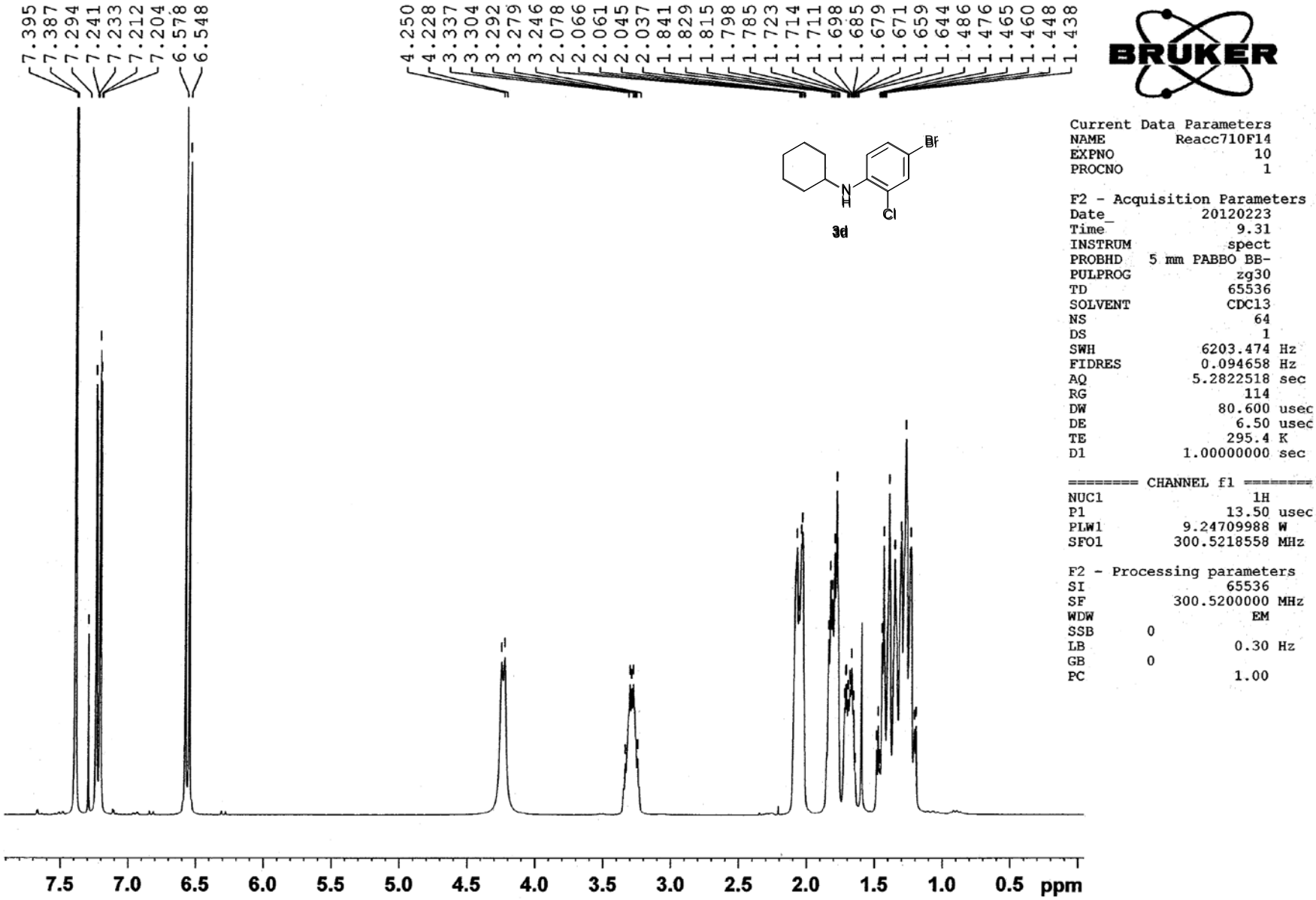
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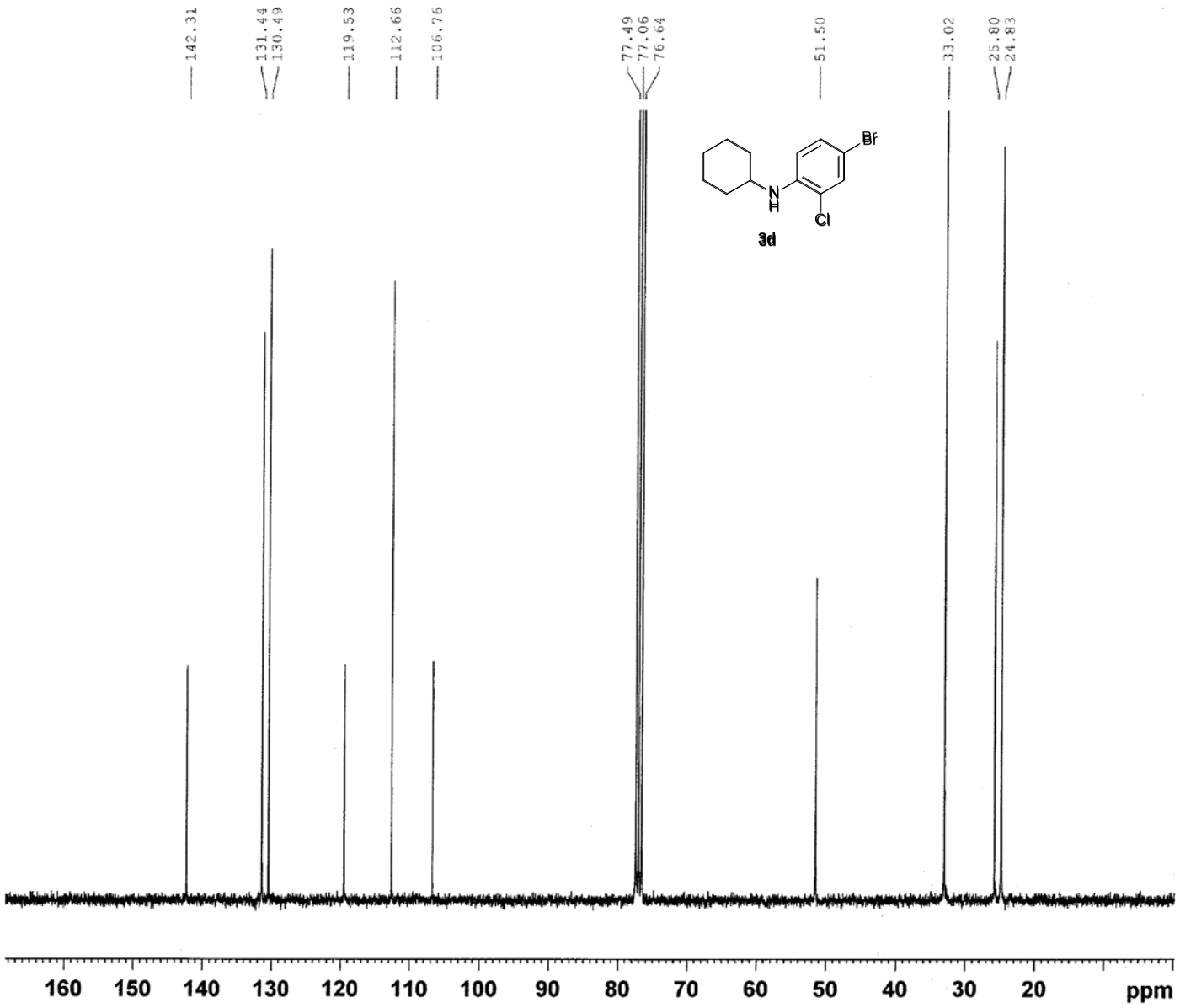
7.212
7.204
6.578
6.548
4.250
4.228
3.337
3.304
3.292
3.279
3.246
2.078
2.066
2.061
2.045
2.037
1.841
1.829
1.815
1.798
1.785
1.723
1.714
1.711
1.698
1.685
1.679
1.671
1.659
1.644
1.486
1.476
1.465
1.460
1.448
1.438
BRUKER
Br
N
H
Cl
3d
Current Data Parameters
NAME Reacc710F14
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120223
Time 9.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 1
SWH 6203.474 Hz
FIDRES 0.094658 Hz
AQ 5.2822518 sec
RG 114
DW 80.600 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PLW1 9.24709988 W
SFO1 300.5218558 MHz
F2 - Processing parameters
SI 65536
SF 300.5200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm




BRUKER

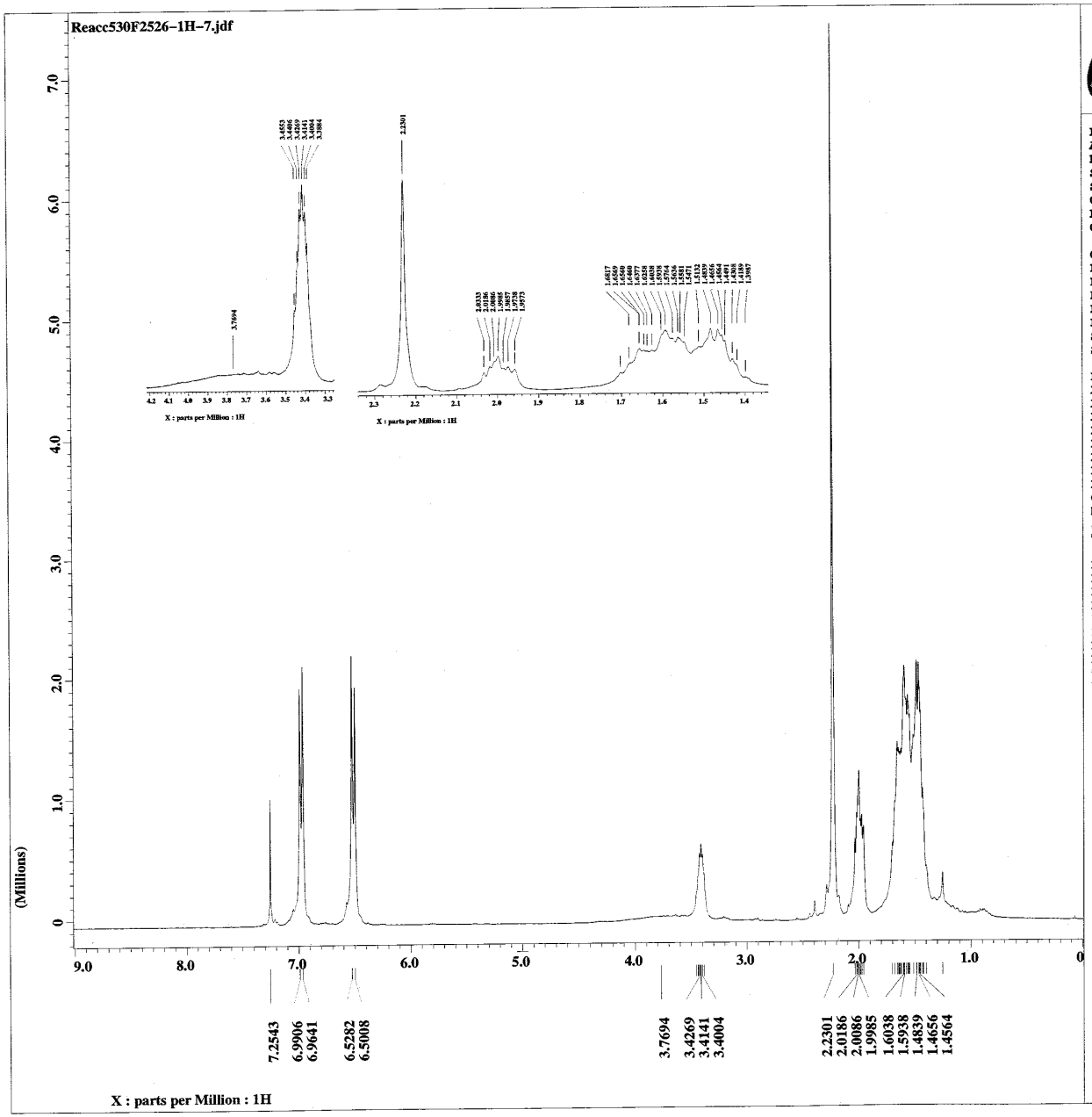
Current Data Parameters
NAME Reacc710F14
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120223
Time 10.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 1
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PLW1 48.96699905 W
SFO1 75.5733703 MHz

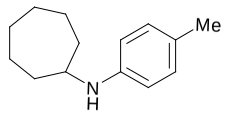
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 9.24709988 W
PLW12 0.21741000 W
PLW13 0.17610000 W
SFO2 300.5212021 MHz

F2 - Processing parameters
SI 32768
SF 75.5658140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

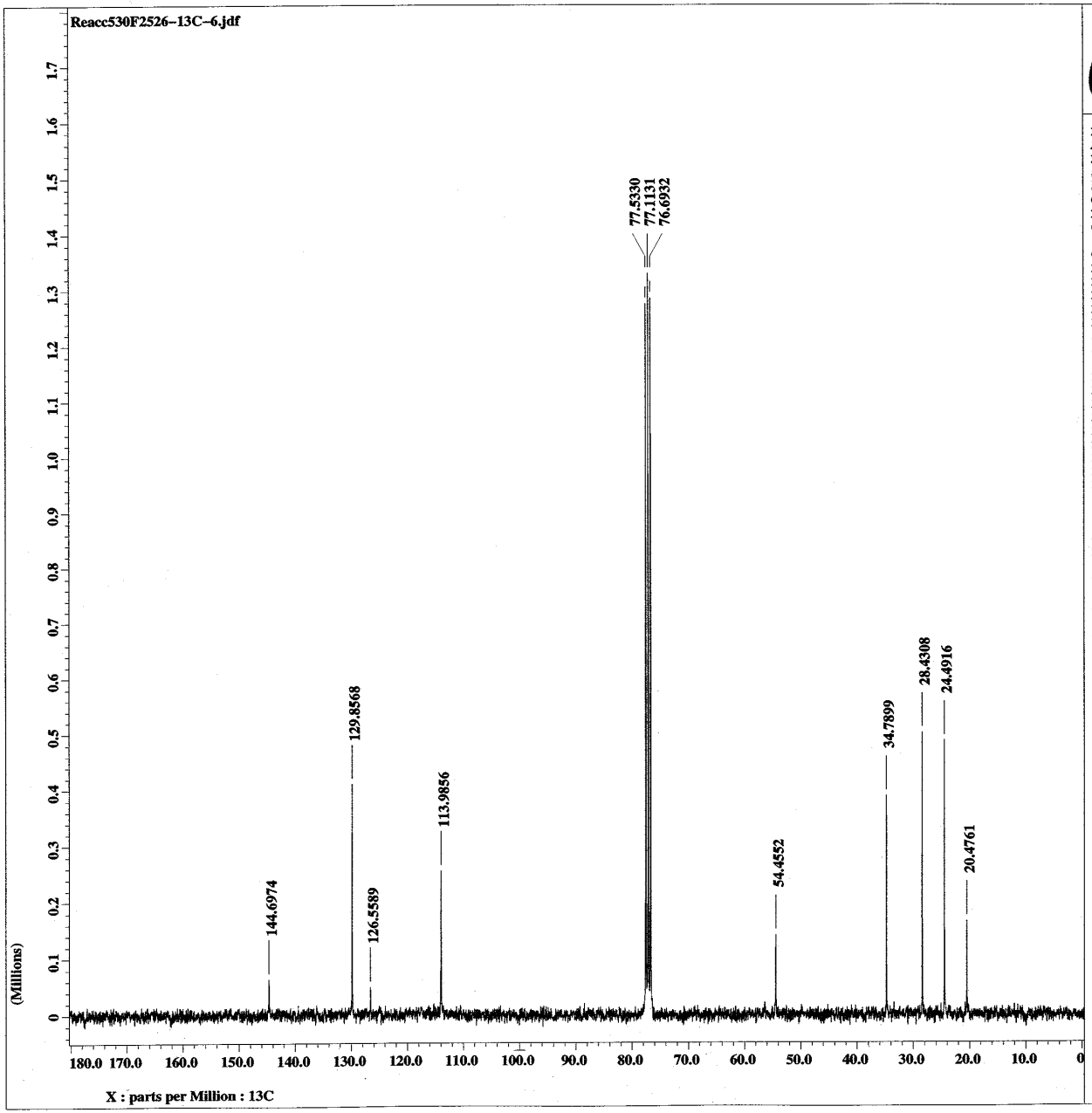



JEOL

| | |
|---|---|
| Filename | = Reacc530F2526-1H-7.jd |
| Author | = Cabrera |
| Experiment | = single_pulse.exp |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 22-DEC-2008 08:03:03 |
| Revision_time | = 5-MAR-2009 16:16:13 |
| Current_time | = 5-MAR-2009 16:16:42 |
| 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 | = 20 |
| Relaxation_delay | = 1[s] |
| Temp_get | = 19.3[dC] |
| Unblank_time | = 2[us] |

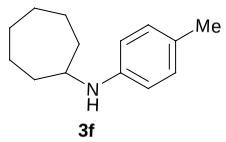



3f

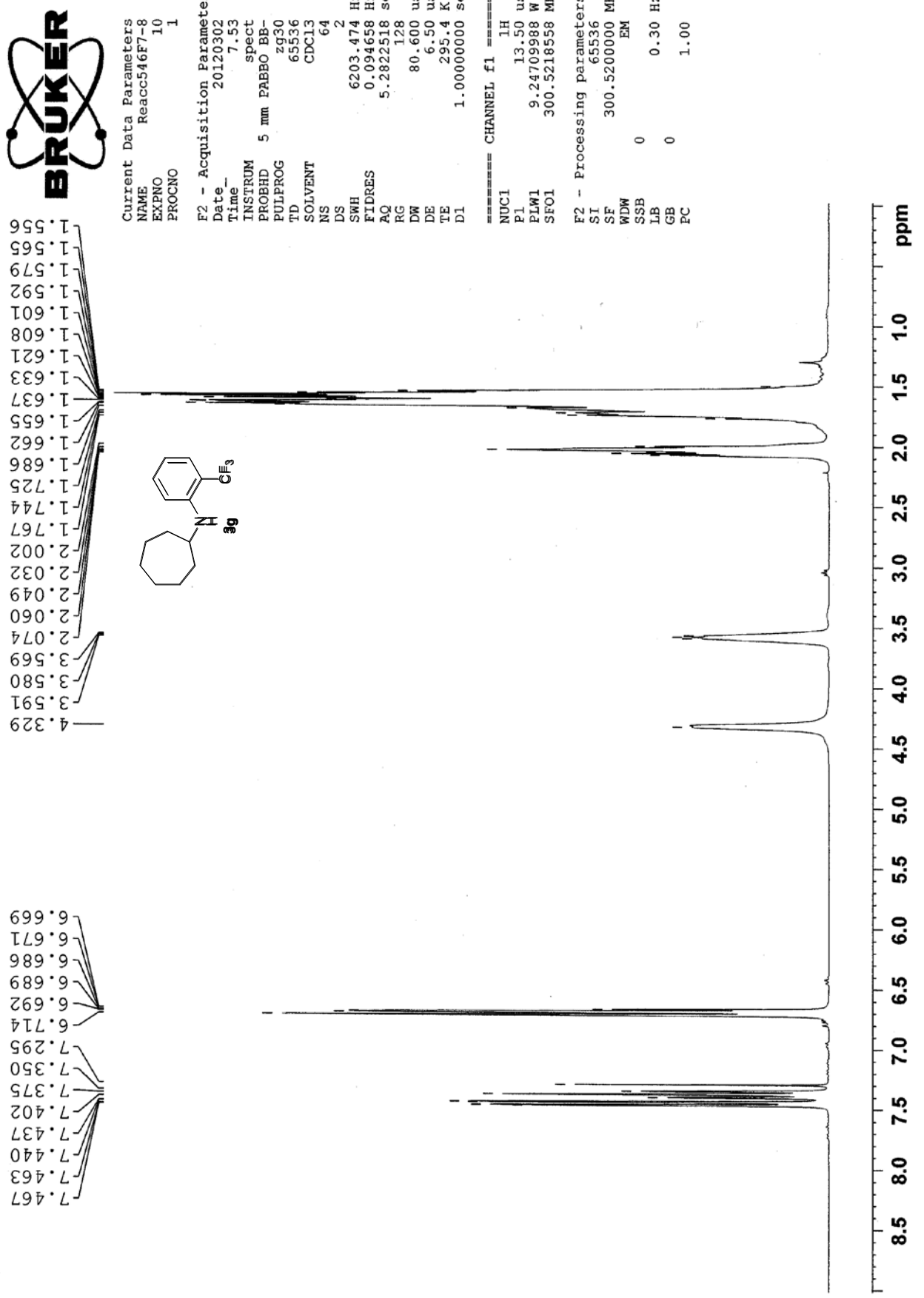


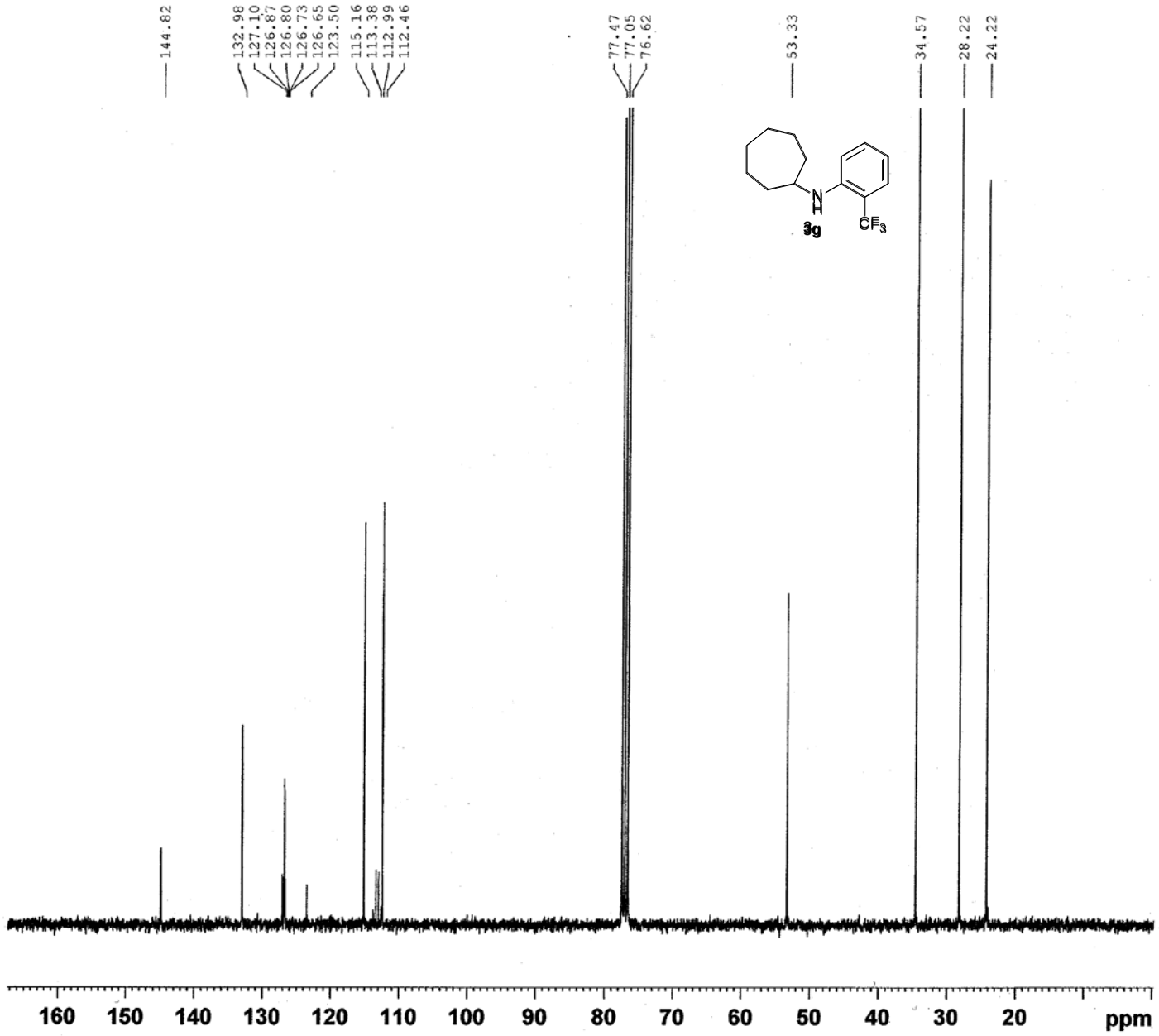

JEOL

| | |
|---|---|
| Filename | = Reacc530F2526-13C-6.j |
| Author | = Cabrera |
| Experiment | = single_pulse_dec |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 22-DEC-2008 08:50:02 |
| Revision_time | = 5-MAR-2009 16:56:22 |
| Current_time | = 5-MAR-2009 16:56:48 |
| 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 | = 943 |
| Total_scans | = 943 |
| 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] |




3f


Current Data Parameters
NAME Reacc546F7-8
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120302
Time 7.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 6203.474 Hz
FIDRES 0.094658 Hz
AQ 5.2822518 sec
RG 128
DW 80.600 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PLW1 9.24709988 W
SFO1 300.5218558 MHz
F2 - Processing parameters
SI 65536
SF 300.5200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3g
4.329
3.591
3.580
3.569
2.074
2.060
2.049
2.032
2.002
1.767
1.744
1.725
1.686
1.662
1.655
1.637
1.633
1.621
1.608
1.601
1.592
1.579
1.565
1.556
7.467
7.463
7.440
7.437
7.402
7.375
7.350
7.295
6.714
6.692
6.689
6.686
6.671
6.669
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm




BRUKER

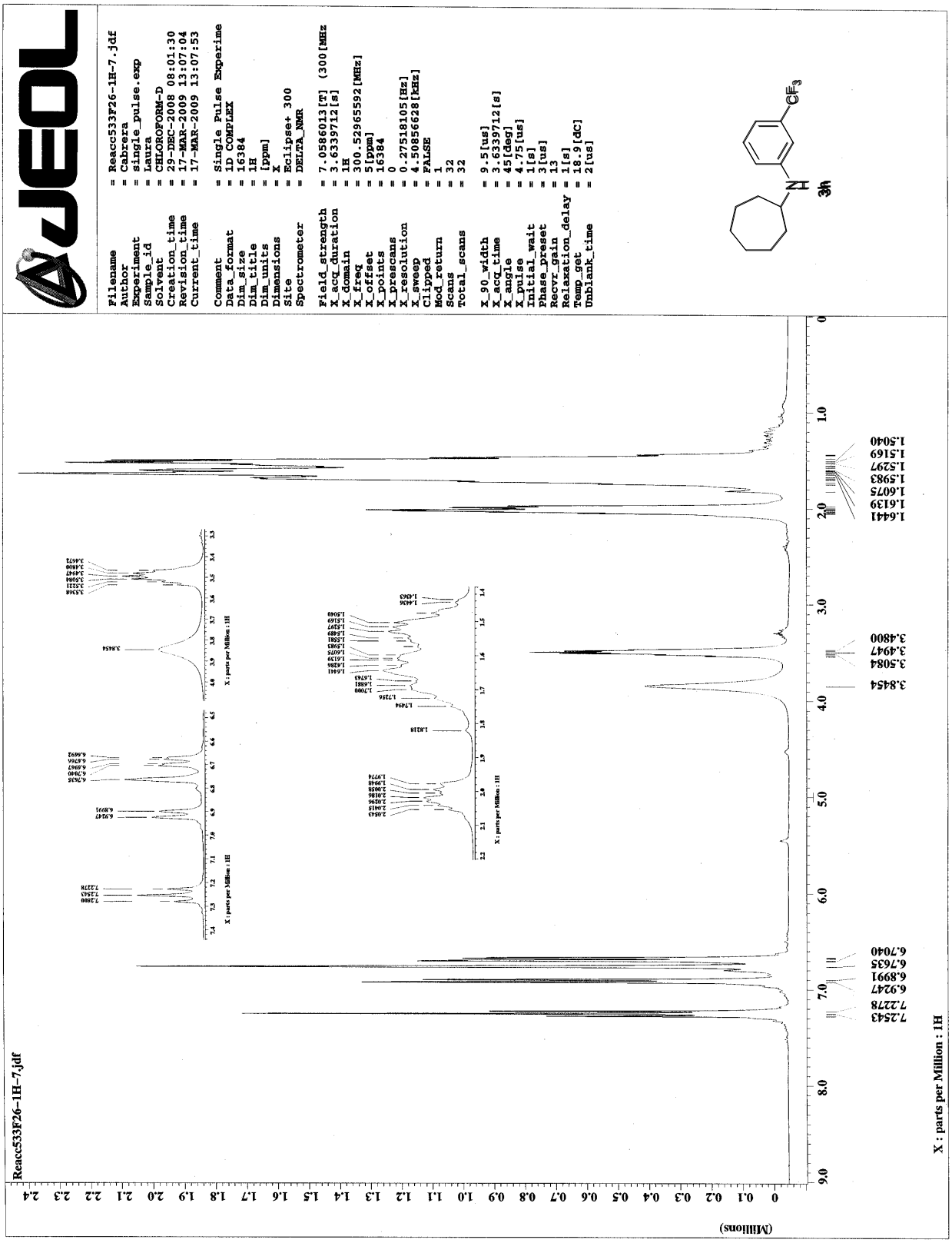
Current Data Parameters
NAME Reacc546F7-8
EXPNO 11
PROCNO 1

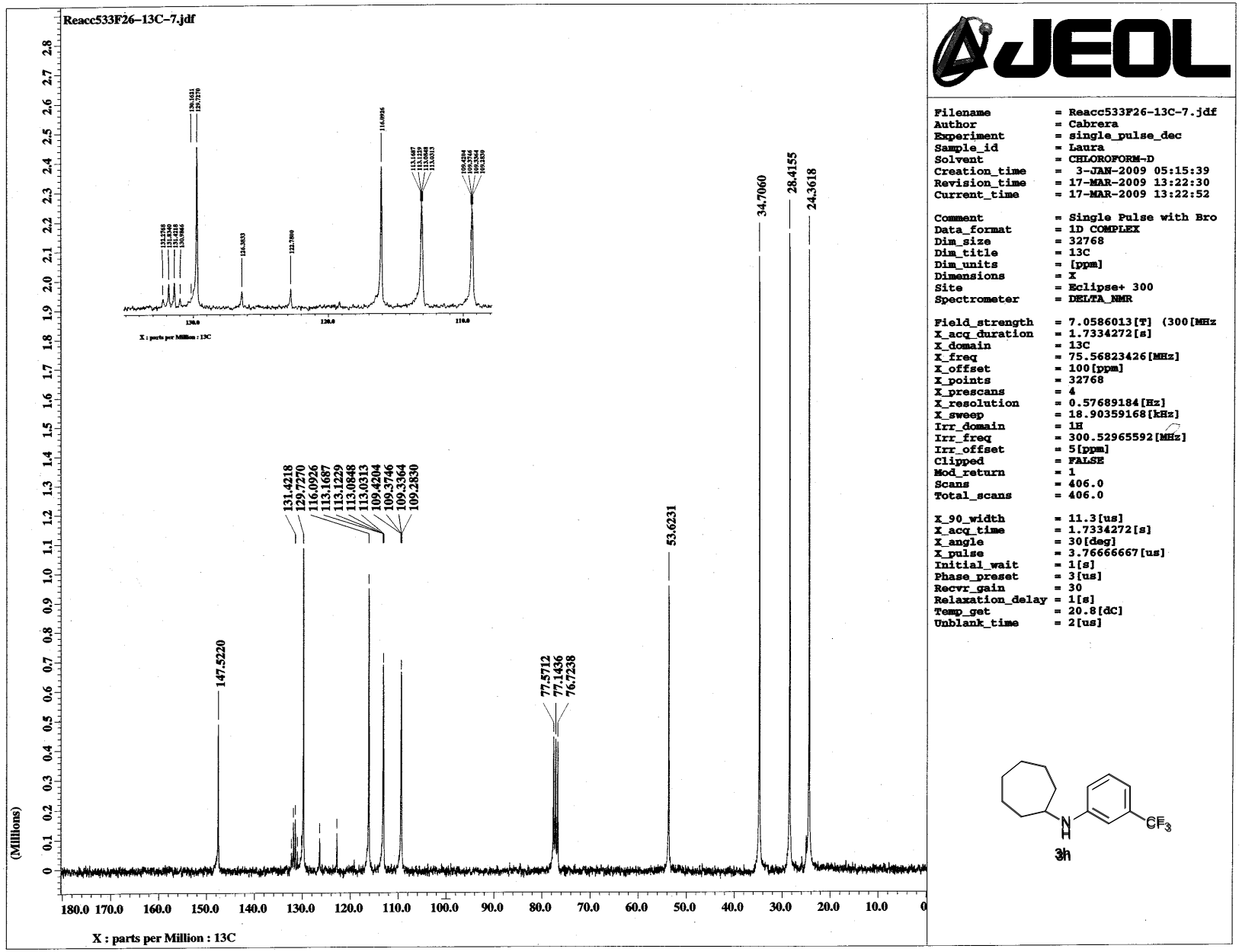
F2 - Acquisition Parameters
Date_ 20120302
Time 8.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 800
DS 1
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec

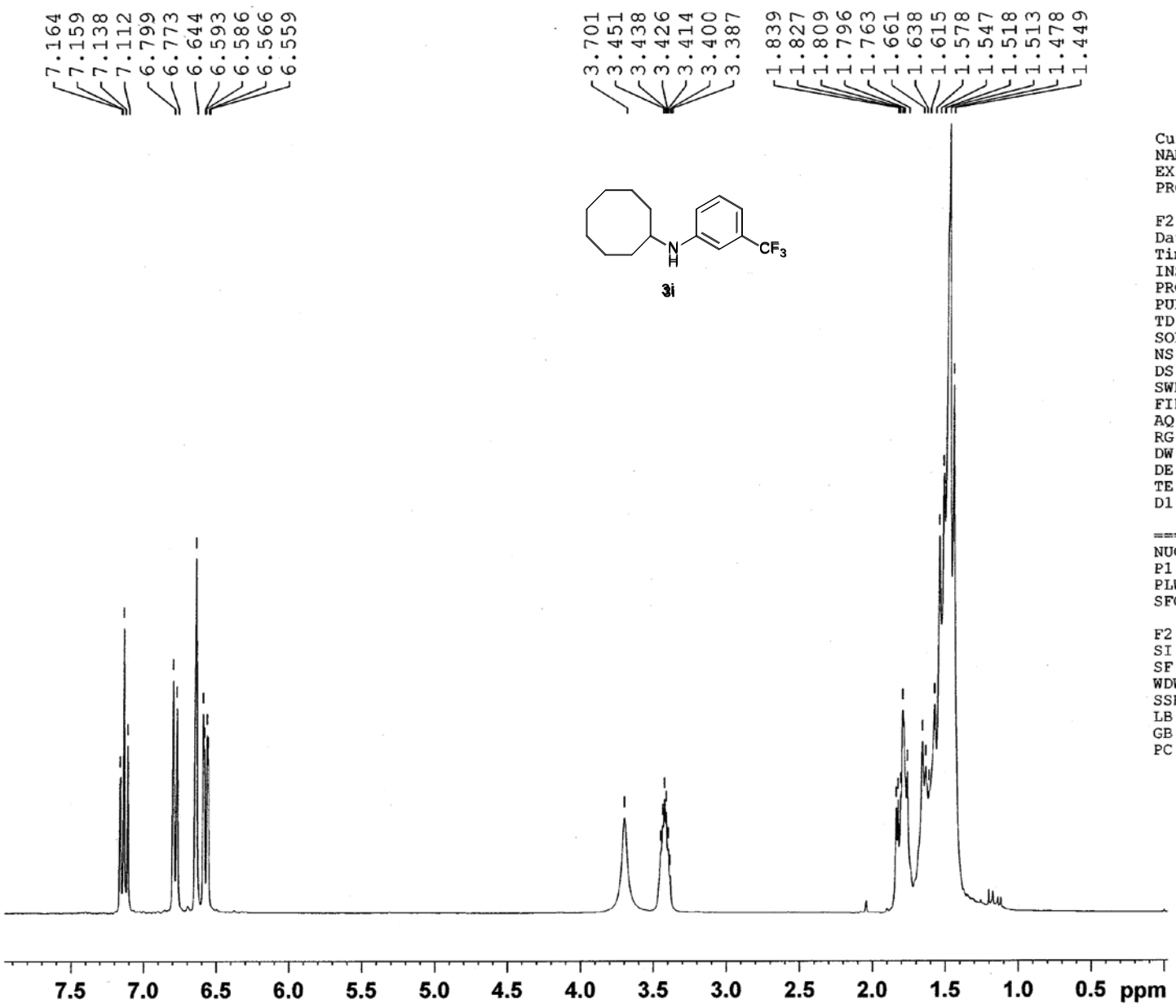
======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PLW1 48.96699905 W
SFO1 75.5733703 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 9.24709988 W
PLW12 0.21741000 W
PLW13 0.17610000 W
SFO2 300.5212021 MHz

F2 - Processing parameters
SI 32768
SF 75.5658140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


JEOL
Filename = Reacc533F26-1H-7.jdf
Author = Cabrera
Experiment = single_pulse.exp
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 29-DEC-2008 08:01:30
Revision_time = 17-MAR-2009 13:07:04
Current_time = 17-MAR-2009 13:07:53
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 = 13
Relaxation_delay = 1[s]
Temp_get = 18.9[dC]
Unblank_time = 2[us]
CF3
N
H
3h
Reacc533F26-1H-7.jdf
(Millions)
X : parts per Million : 1H
7.2543
7.2278
6.9247
6.8991
6.7635
6.7040
3.8454
3.5084
3.4947
3.4800
1.6441
1.6139
1.6075
1.5983
1.5297
1.5169
1.5040


Reacc533F26-13C-7.jdf
(Millions)
X : parts per Million : 13C
147.5220
131.4218
129.7270
116.0926
113.1687
113.1229
113.0848
113.0313
109.4204
109.3746
109.3364
109.2830
77.5712
77.1436
76.7238
53.6231
34.7060
28.4155
24.3618
Filename = Reacc533F26-13C-7.jdf
Author = Cabrera
Experiment = single_pulse_dec
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 3-JAN-2009 05:15:39
Revision_time = 17-MAR-2009 13:22:30
Current_time = 17-MAR-2009 13:22:52
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 = 406.0
Total_scans = 406.0
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]
N
H
CF3
3h



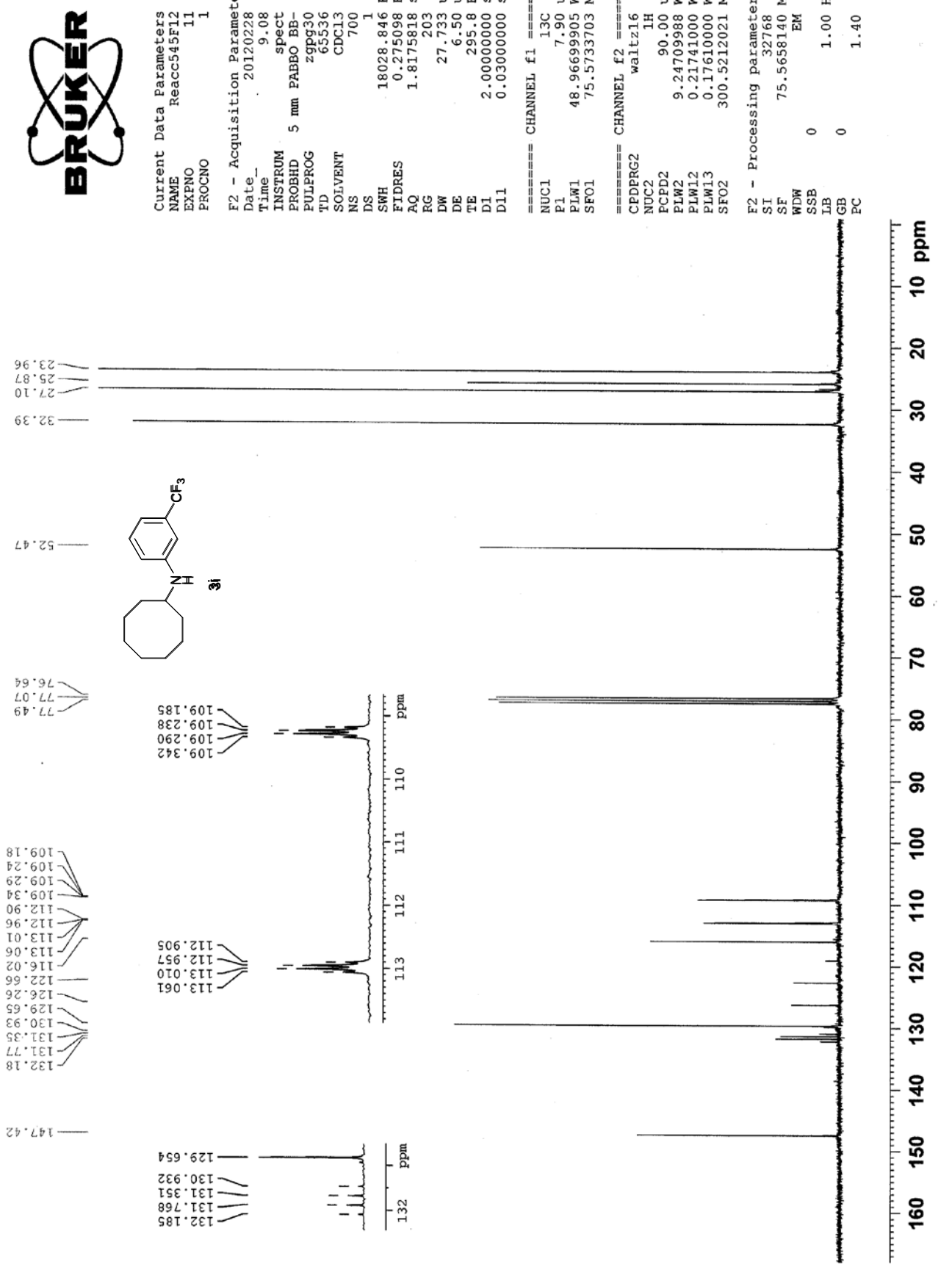

BRUKER

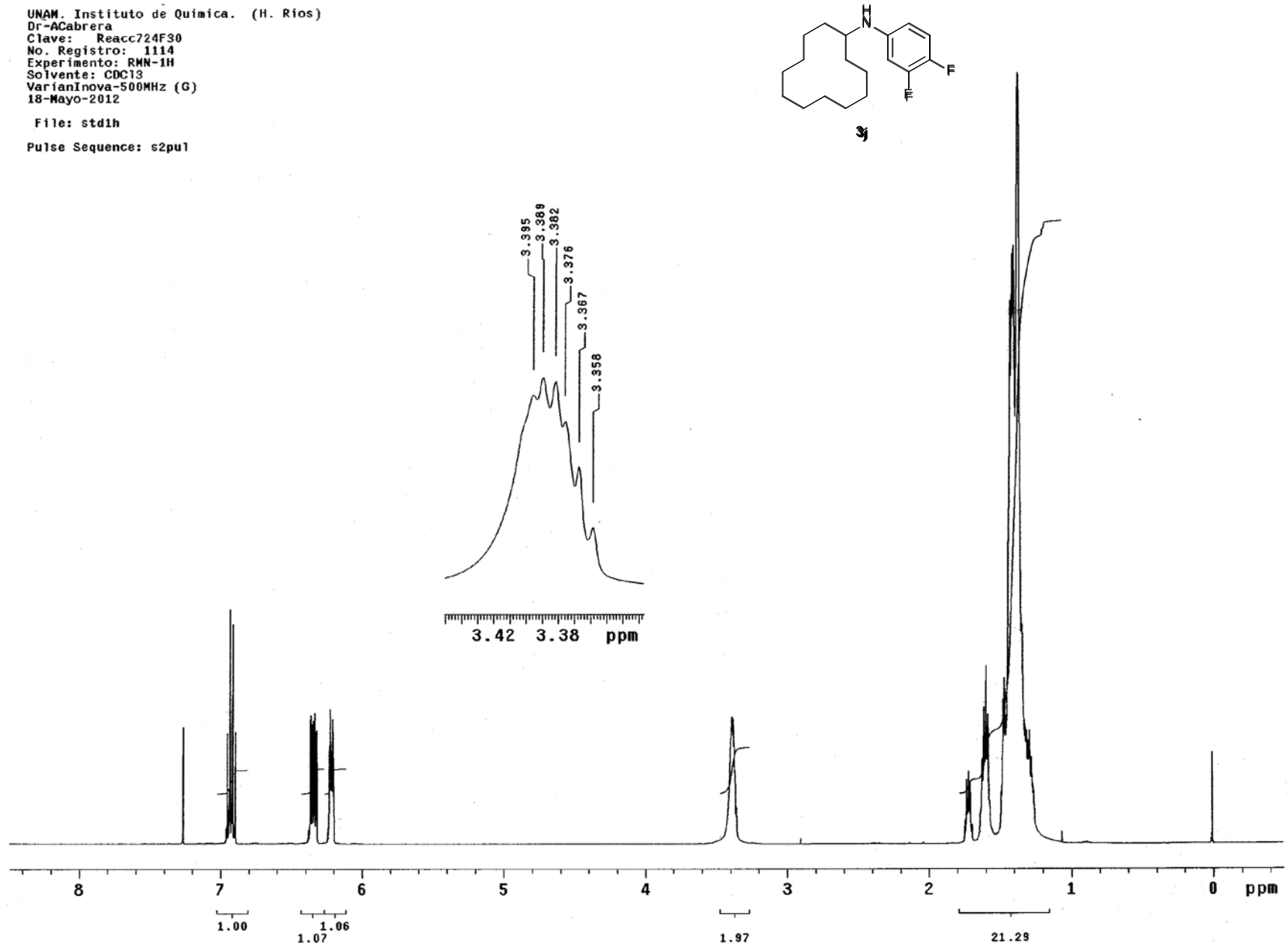
Current Data Parameters
NAME Reacc545F12
EXPNO 10
PROCNO 1

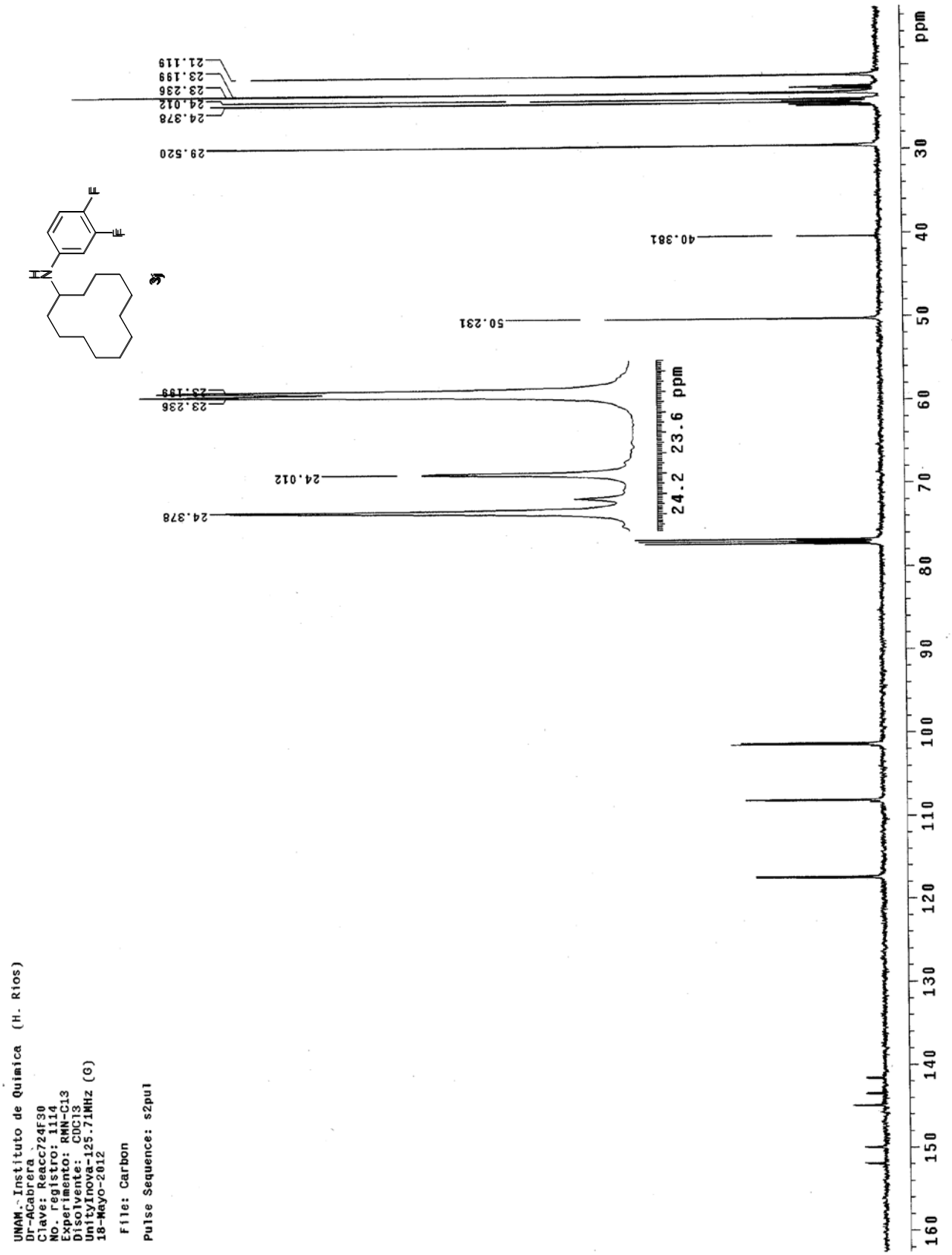
F2 - Acquisition Parameters
Date_ 20120228
Time 8.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 1
SWH 6203.474 Hz
FIDRES 0.094658 Hz
AQ 5.2822518 sec
RG 57
DW 80.600 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec

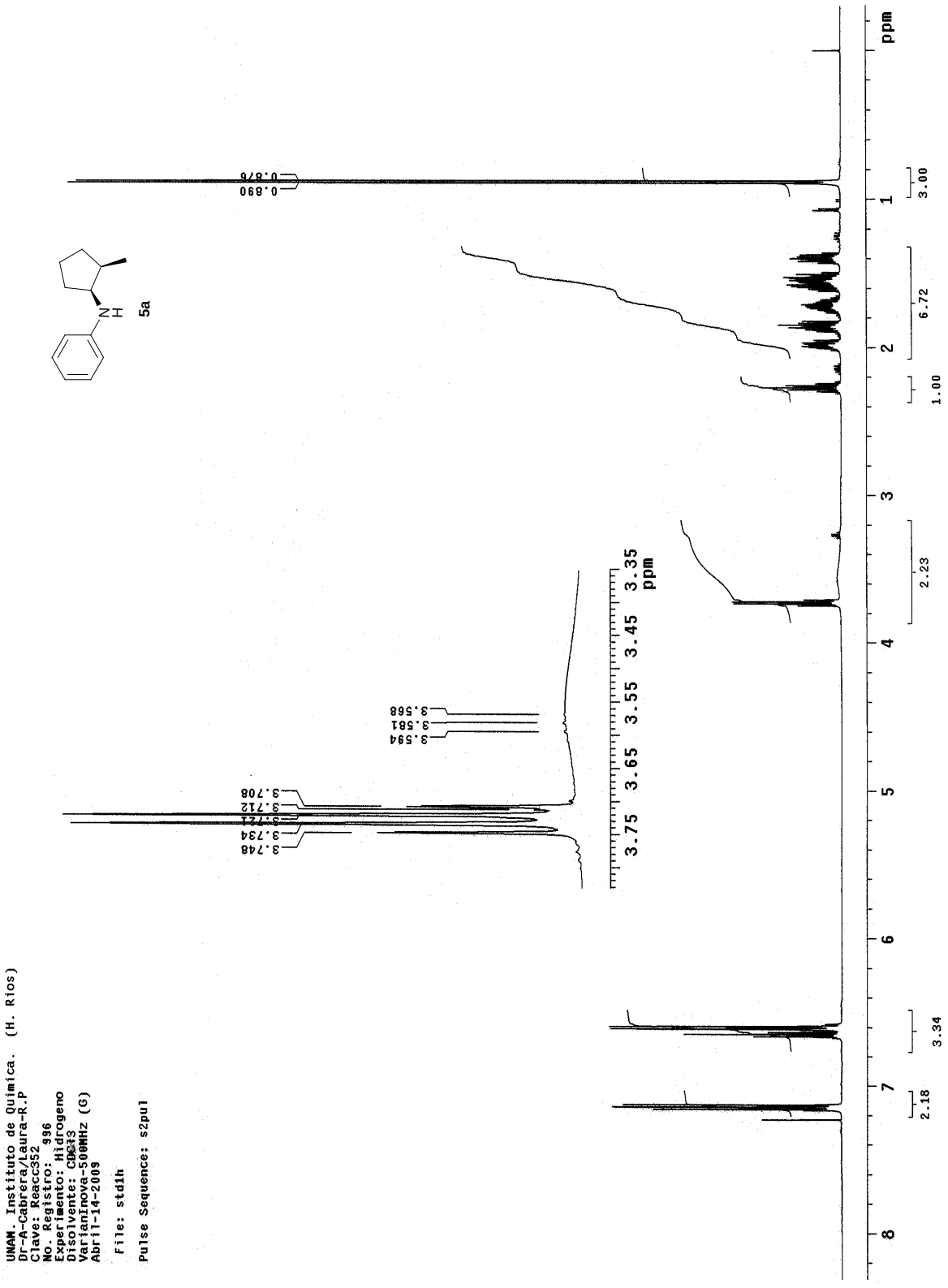
======== CHANNEL f1 ========
NUC1 1H
P1 13.50 usec
PLW1 9.24709988 W
SFO1 300.5218558 MHz

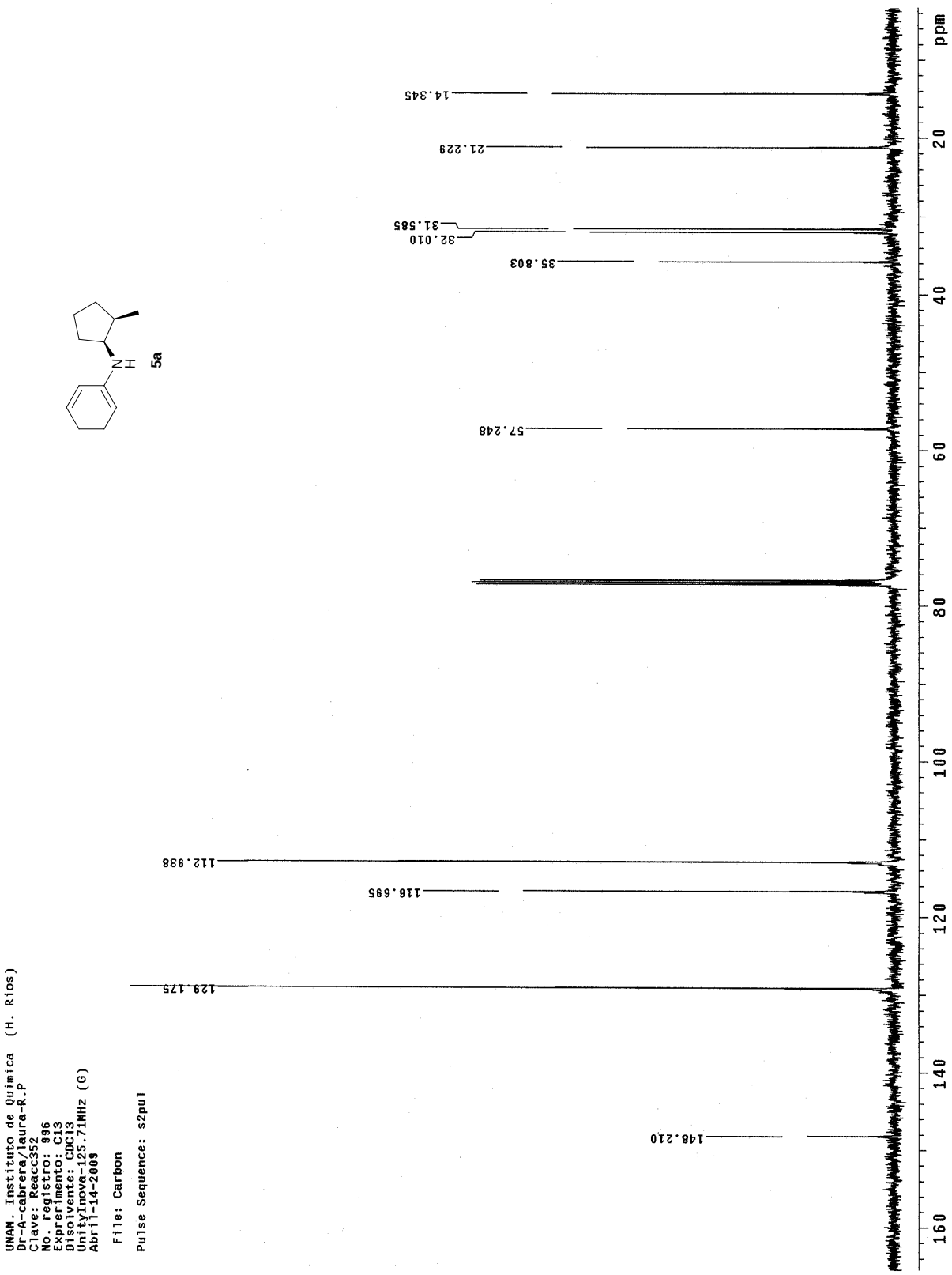
F2 - Processing parameters
SI 65536
SF 300.5200407 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

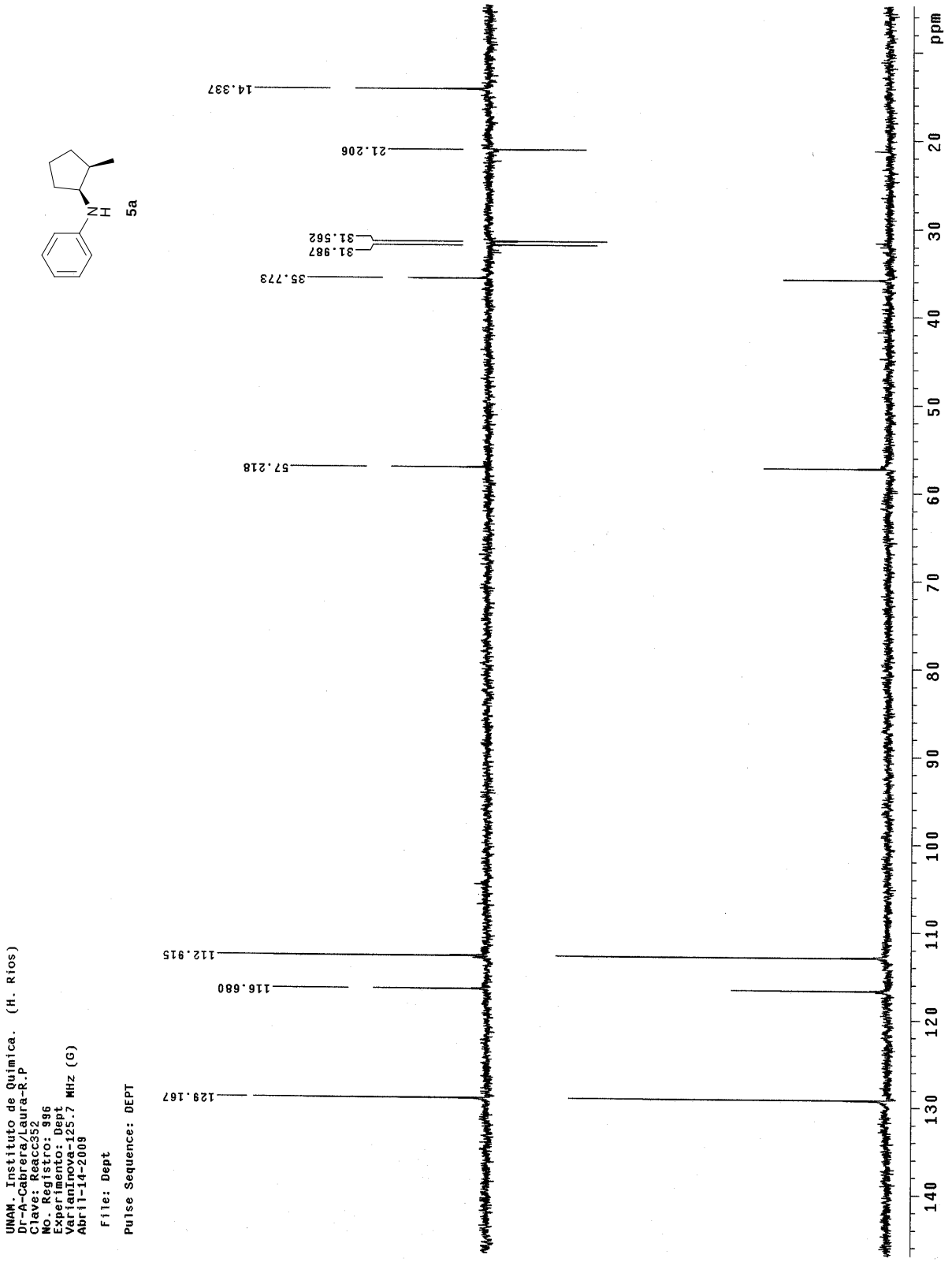

Current Data Parameters
NAME Reacc545F12
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120228
Time_ 9.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 700
DS 1
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 203
DW 27.733 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.90 usec
PLW1 48.96699905 W
SFO1 75.5733703 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 9.24709988 W
PLW12 0.21741000 W
PLW13 0.17610000 W
SFO2 300.5212021 MHz
F2 - Processing parameters
SI 32768
SF 75.5658140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
3i
ppm

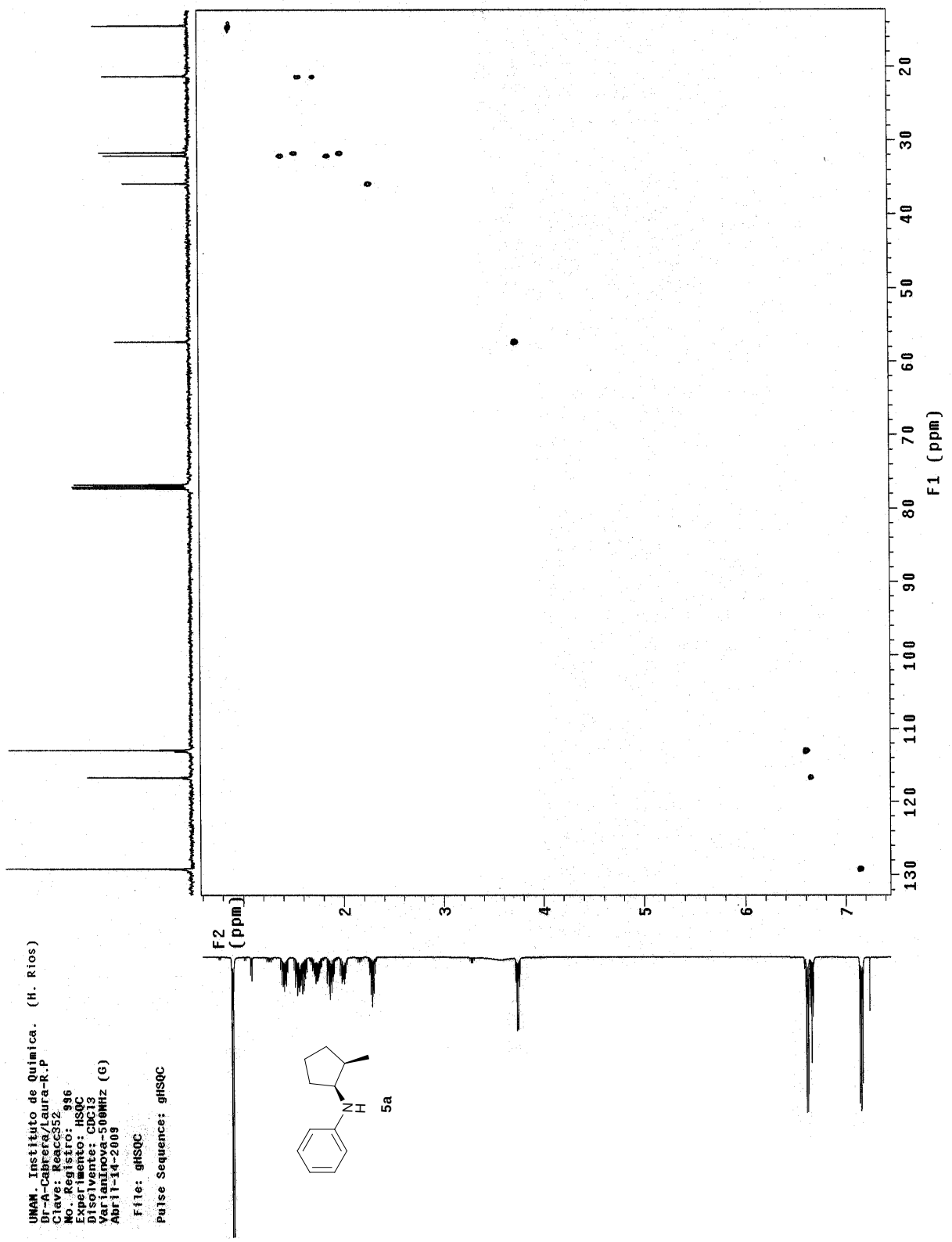

UNAM. Instituto de Quimica. (H. Rios)
Dr-ACabrera
Clave: Reacc724F30
No. Registro: 1114
Experimento: RMN-1H
Solvente: CDCl3
VarianInova-500MHz (G)
18-Mayo-2012
File: std1h
Pulse Sequence: s2pul
3j
3.395
3.389
3.382
3.376
3.367
3.358
3.42 3.38 ppm
8 7 6 5 4 3 2 1 0 ppm
1.00
1.07
1.06
1.97
21.29

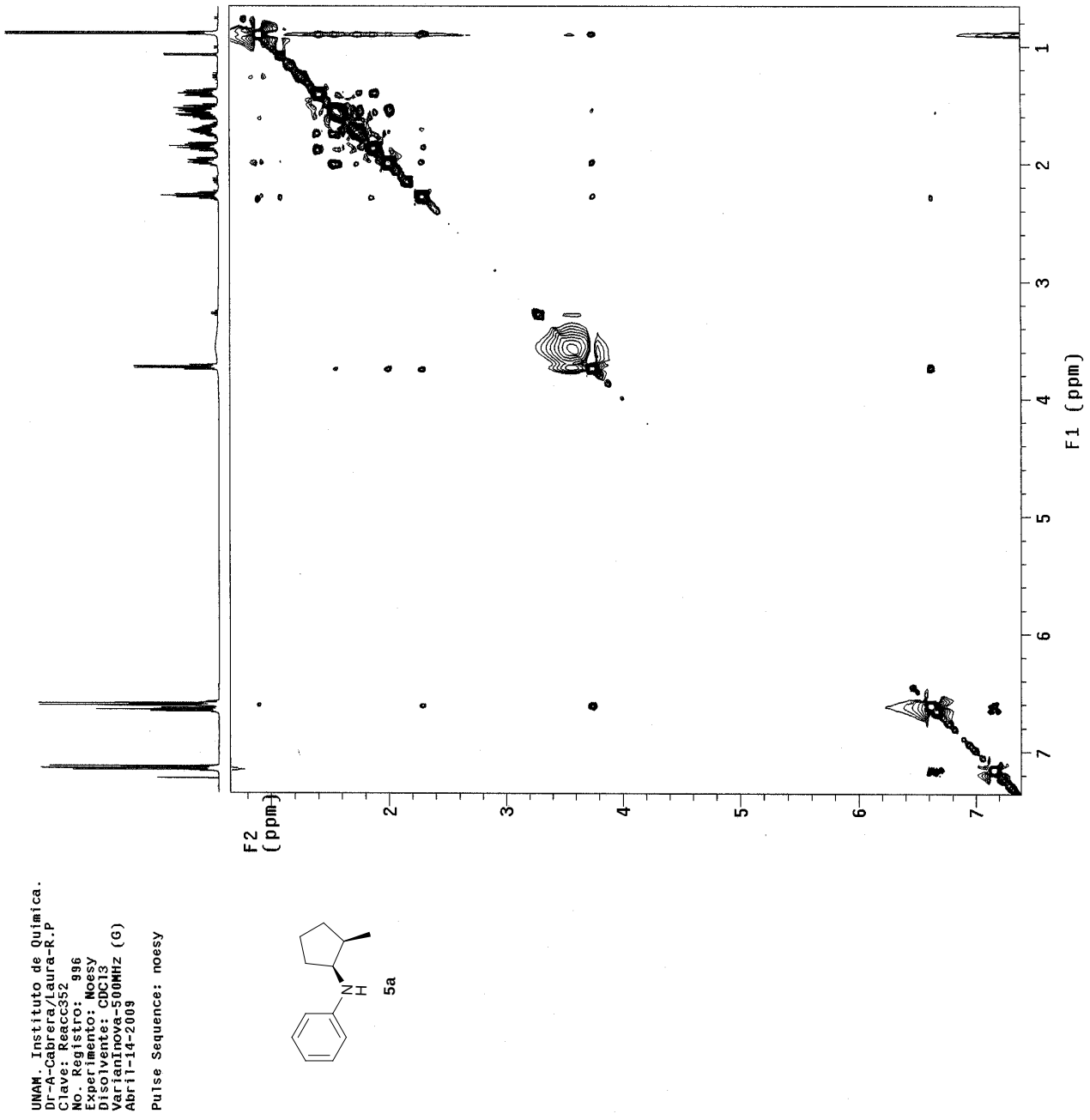

UNAM.-Instituto de Quimica (H. Rios)
Dr-ACabrera
Clave: Reacc724F30
No. registro: 1114
Experimento: RMN-C13
Disolvente: CDCl3
UnityInova-125.7iMHz (G)
18-Mayo-2012
File: Carbon
Pulse Sequence: s2pul
21.119
23.199
23.236
24.012
24.378
29.520
40.381
50.231
23.188
23.236
24.012
24.378
24.2 23.6 ppm
F
F
N
H
3j
ppm


UNAM. Instituto de Quimica. (H. Rios)
Dr-A-Cabrera/Laura-R.P
Clave: Reacc352 996
No. Registro:
Experimento: Hidrogeno
Disolvente: CDCl3
VarianInova-500MHz (G)
Abril-14-2009
File: stdih
Pulse Sequence: s2pul
5a
0.890
0.876
3.748
3.734
3.721
3.712
3.708
3.594
3.581
3.568
3.75 3.65 3.55 3.45 3.35
ppm
2.23
6.72
1.00
3.00
2.18
3.34
ppm


UNAM. Instituto de Quimica (H. Rios)
Dr-A-cabrera/laura-R.P
Clave: Reacc352
No. registro: 996
Experimento: C13
Disolvente: CDCl3
Unityinova-125.71MHz (G)
Abril-14-2009
File: Carbon
Pulse Sequence: s2pul
5a
148.210
129.175
112.938
116.695
57.248
35.803
32.010
31.585
21.229
14.345
ppm

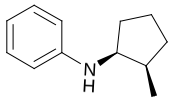

UNAM. Instituto de Quimica. (H. Rios)
Dr-A-Cabrera/Laura-R.P
Clave: Reacc352
No. Registro: 996
Experimento: Dept
VarianInova-125.7 MHz (G)
Abril-14-2009
File: Dept
Pulse Sequence: DEPT
5a
14.337
21.206
31.562
31.987
35.773
57.218
112.915
116.680
129.167
ppm


UNAM. Instituto de Quimica. (H. Rios)
Dr-A-Cabrera/Laura-R.P
Clave: Reacc352
No. Registro: 986
Experimento: HSQC
Disolvente: CDCl3
VarianInova-500MHz (G)
Abril-14-2009
File: gHSQC
Pulse Sequence: gHSQC
F2 (ppm)
F1 (ppm)
5a


UNAM. Instituto de Quimica.
Dr-A-Cabrera/Laura-R.P
Clave: Reacc352
No. Registro: 996
Experimento: Noesy
Disolvente: CDCl3
VarianInova-500MHz (G)
Abril-14-2009
Pulse Sequence: noesy
F2 (ppm)
F1 (ppm)
5a

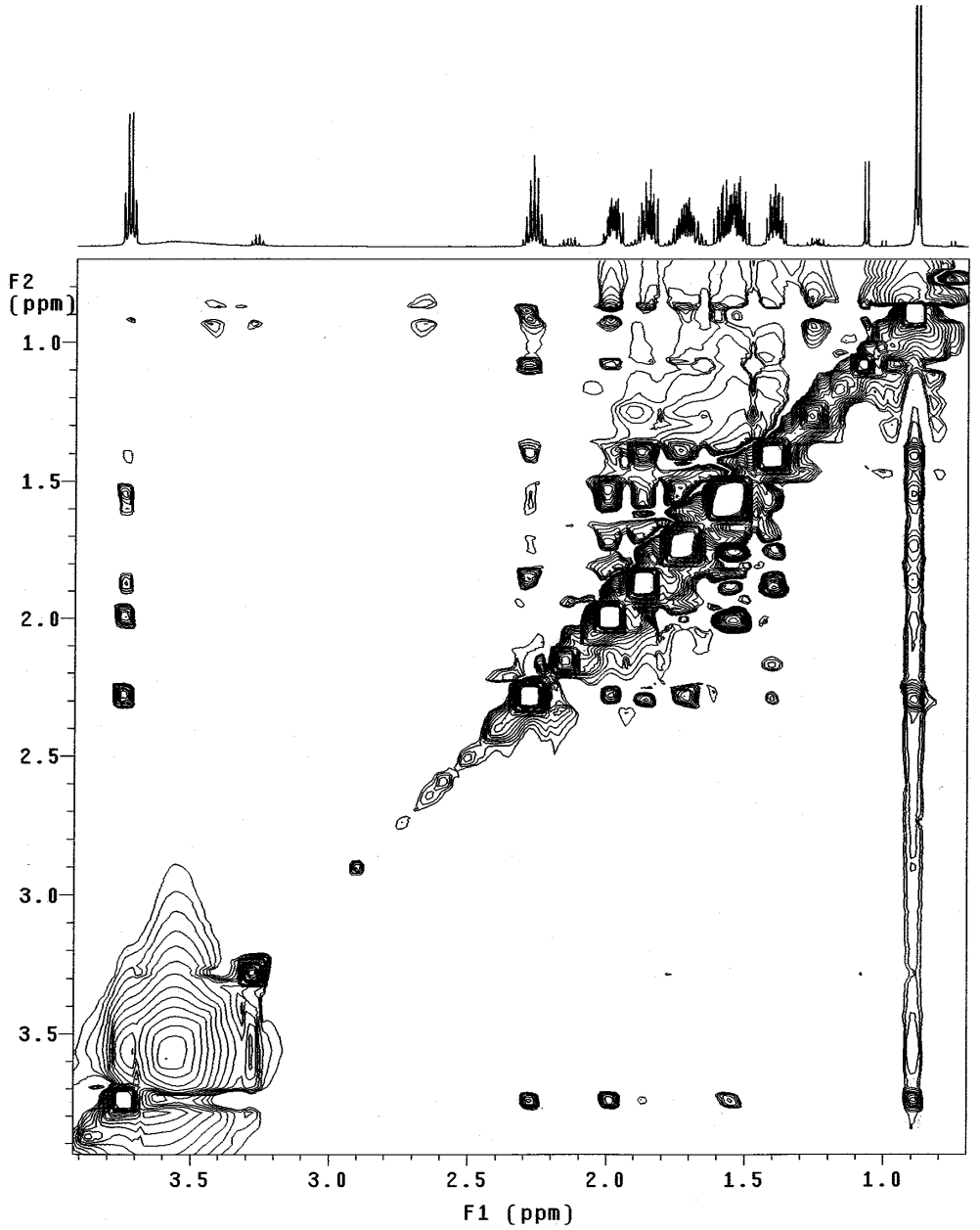
UNAM. Instituto de Quimica.
Dr-A-Cabrera/Laura-R.P
Clave: Reacc352
No. Registro: 996
Experimento: Noesy
Disolvente: CDCl3
VarianInova-500MHz (G)
Abril-14-2009

Pulse Sequence: noesy

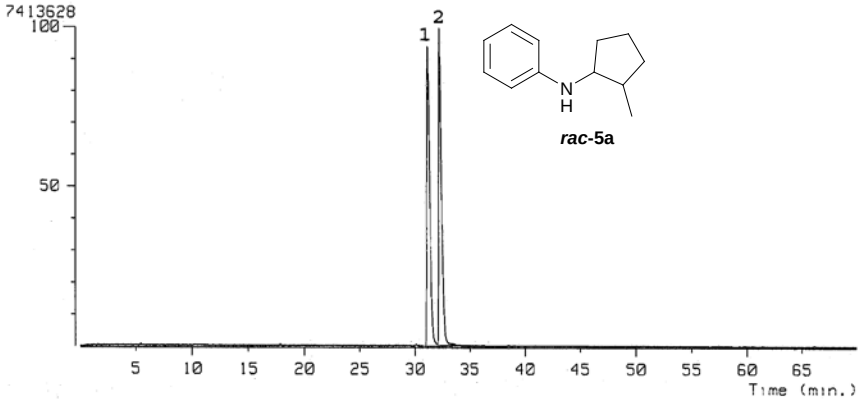


5a

NOE-Expansions



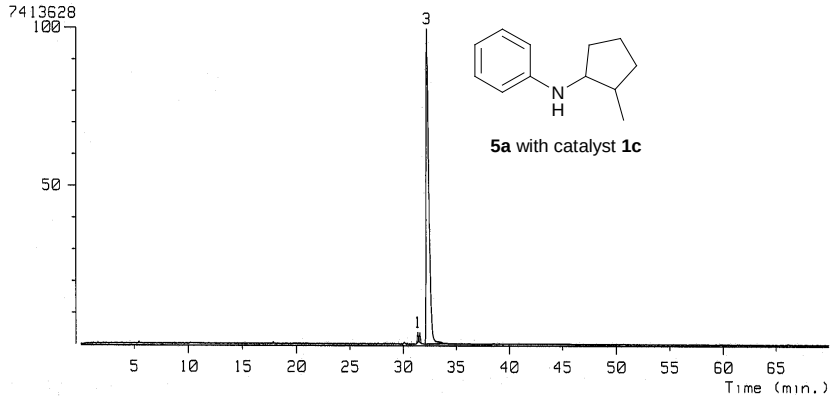
[ TIC ]
Data : Dr-Cabrera-Armando-962 Date : 16-Feb-120 16:27
Sample: 405 G reacc 321-rac AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650






***rac*-5a**

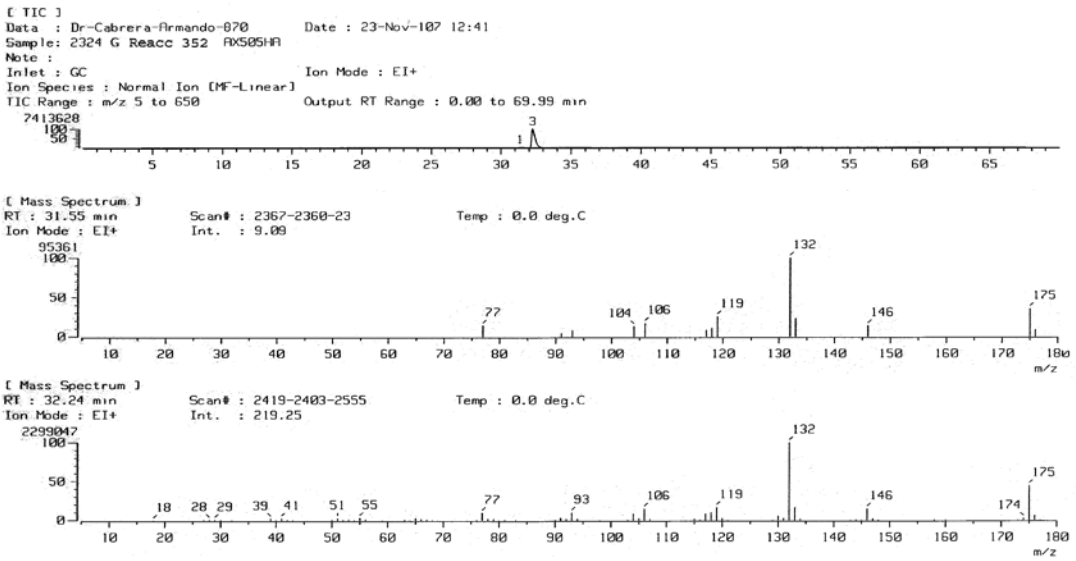
[ TIC ]
Data : Dr-Cabrera-Armando-870 Date : 23-Nov-107 12:41
Sample: 2324 G Reacc 352 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650

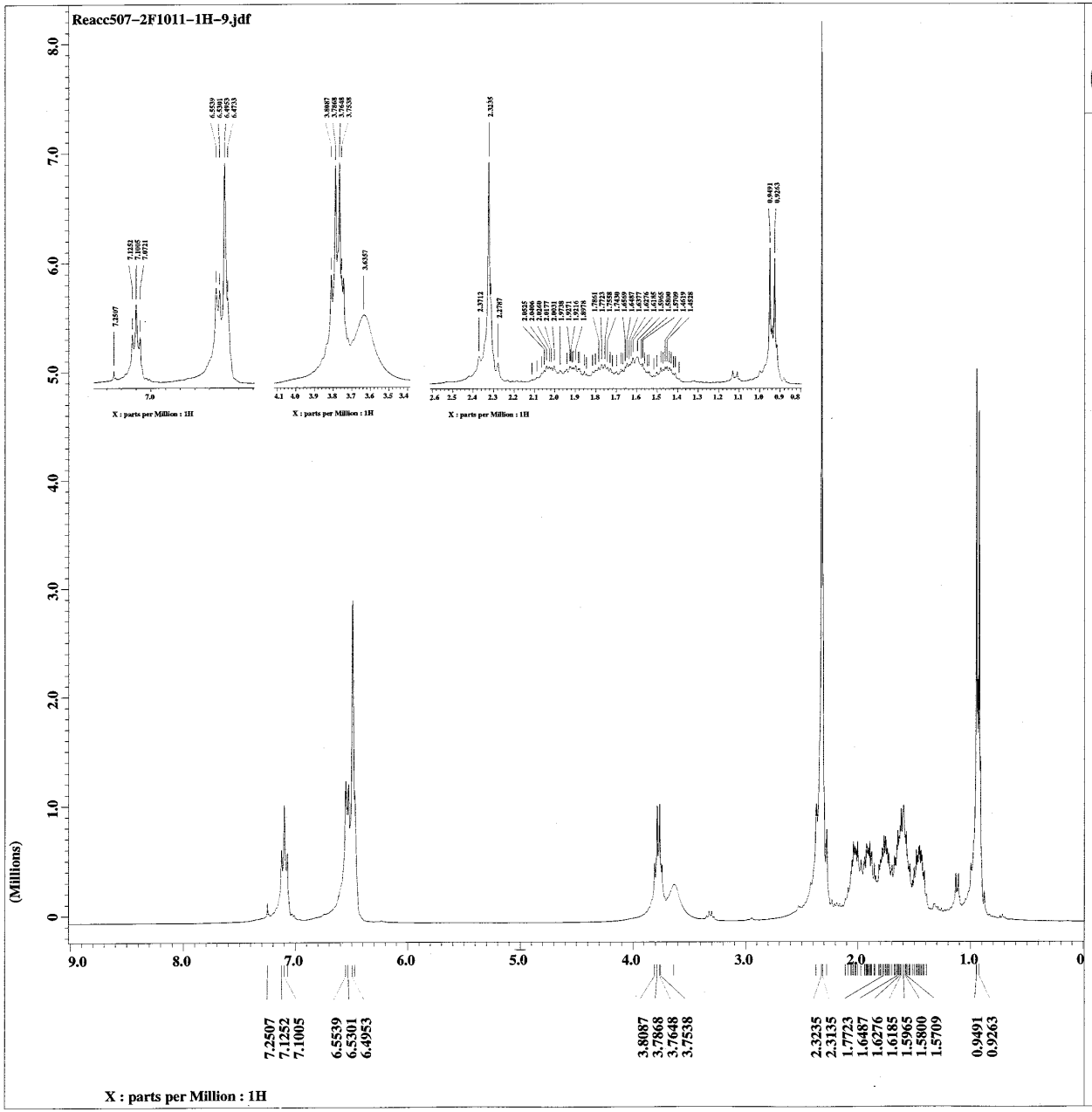





**5a** with catalyst **1c**

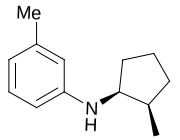
| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 31.35 | 135.68 | 1.06 | 22.01 | 2.95 | 5.79 | BB |
| 2 | 31.55 | 135.08 | 1.05 | 20.51 | 2.75 | 6.18 | BB |
| 3 | 32.24 | 12542.16 | 97.89 | 704.79 | 94.31 | 16.71 | BB |



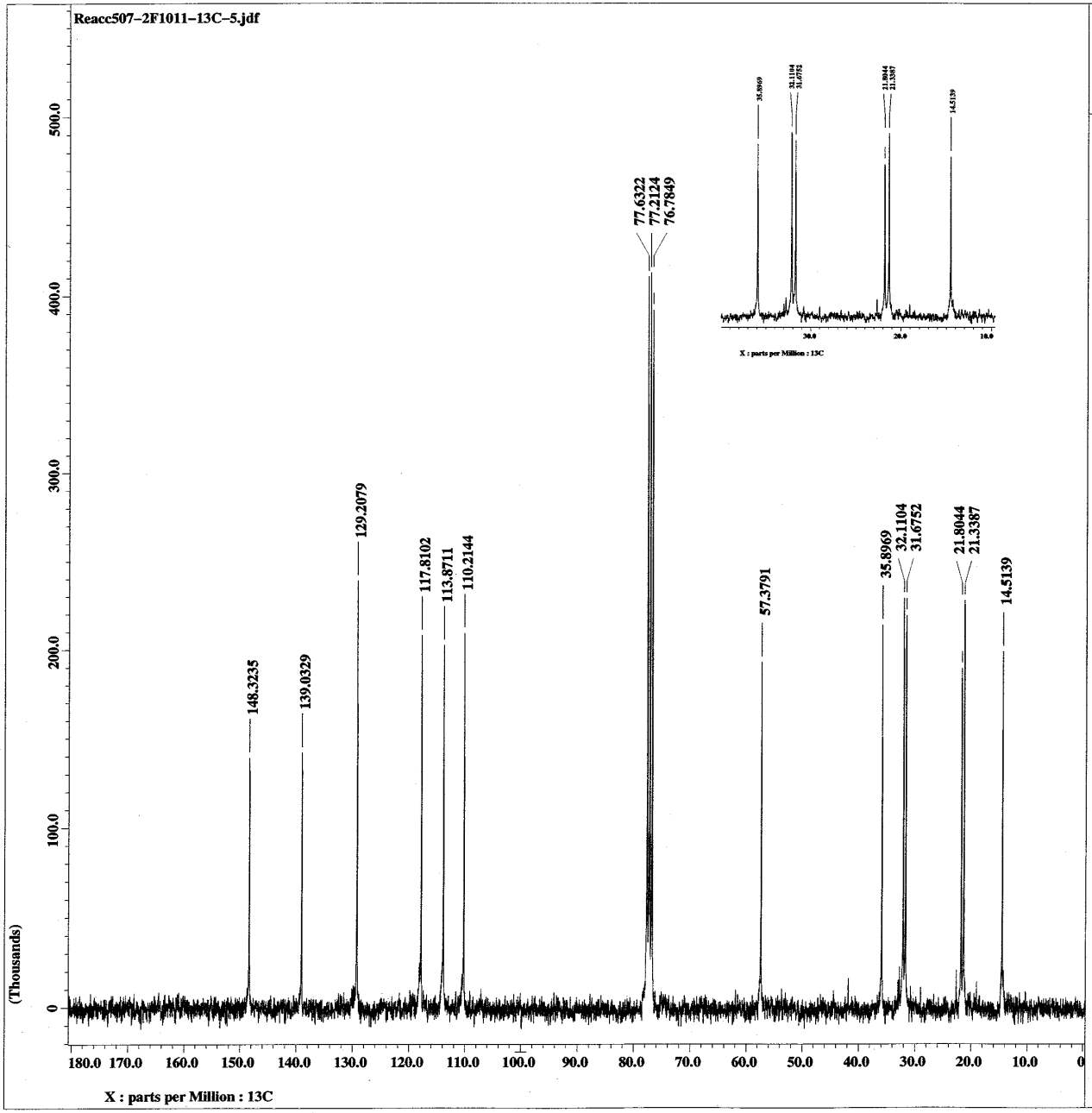



JEOL

| | |
|---|---|
| Filename | = Reacc507-2F1011-1H-9. |
| Author | = Cabrera |
| Experiment | = single_pulse.exp |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 30-AUG-2008 01:58:16 |
| Revision_time | = 11-NOV-2008 09:10:14 |
| Current_time | = 11-NOV-2008 09:12:23 |
| 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 | = 15 |
| Relaxation_delay | = 1[s] |
| Temp_get | = 19.6[dC] |
| Unblank_time | = 2[us] |




5b

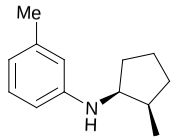






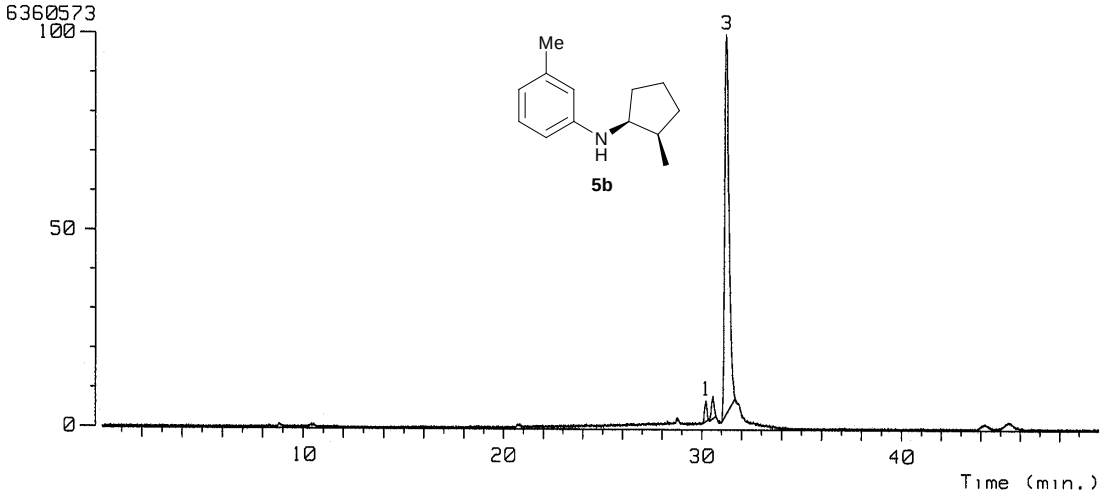
JEOL

| | |
|---|---|
| Filename | = Reacc507-2F1011-13C-5 |
| Author | = Cabrera |
| Experiment | = single_pulse_dec |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 30-AUG-2008 01:55:08 |
| Revision_time | = 11-NOV-2008 09:03:55 |
| Current_time | = 11-NOV-2008 09:04:48 |
| 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 | = 2626 |
| Total_scans | = 2626 |
| 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] |

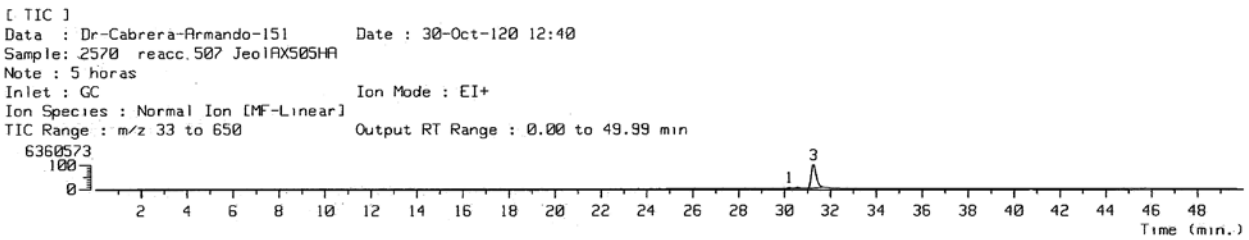


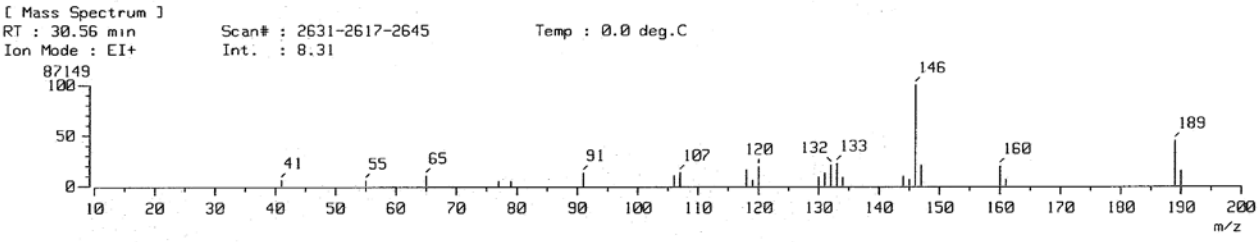

5b

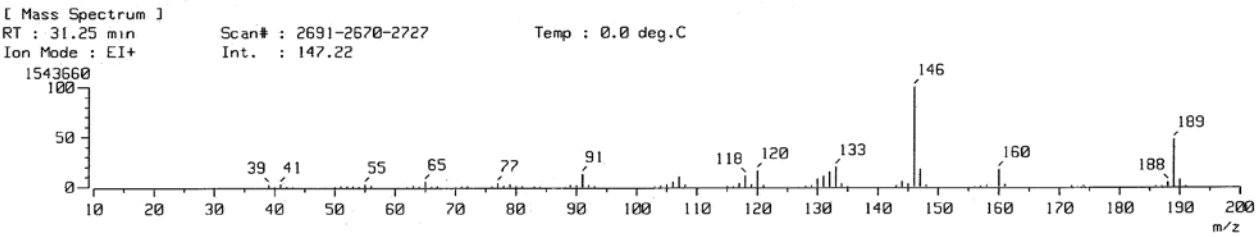
[ TIC ]
Data : Dr-Cabrera-Armando-151 Date : 30-Oct-120 12:40
Sample: 2570 reacc 507 JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 33 to 650

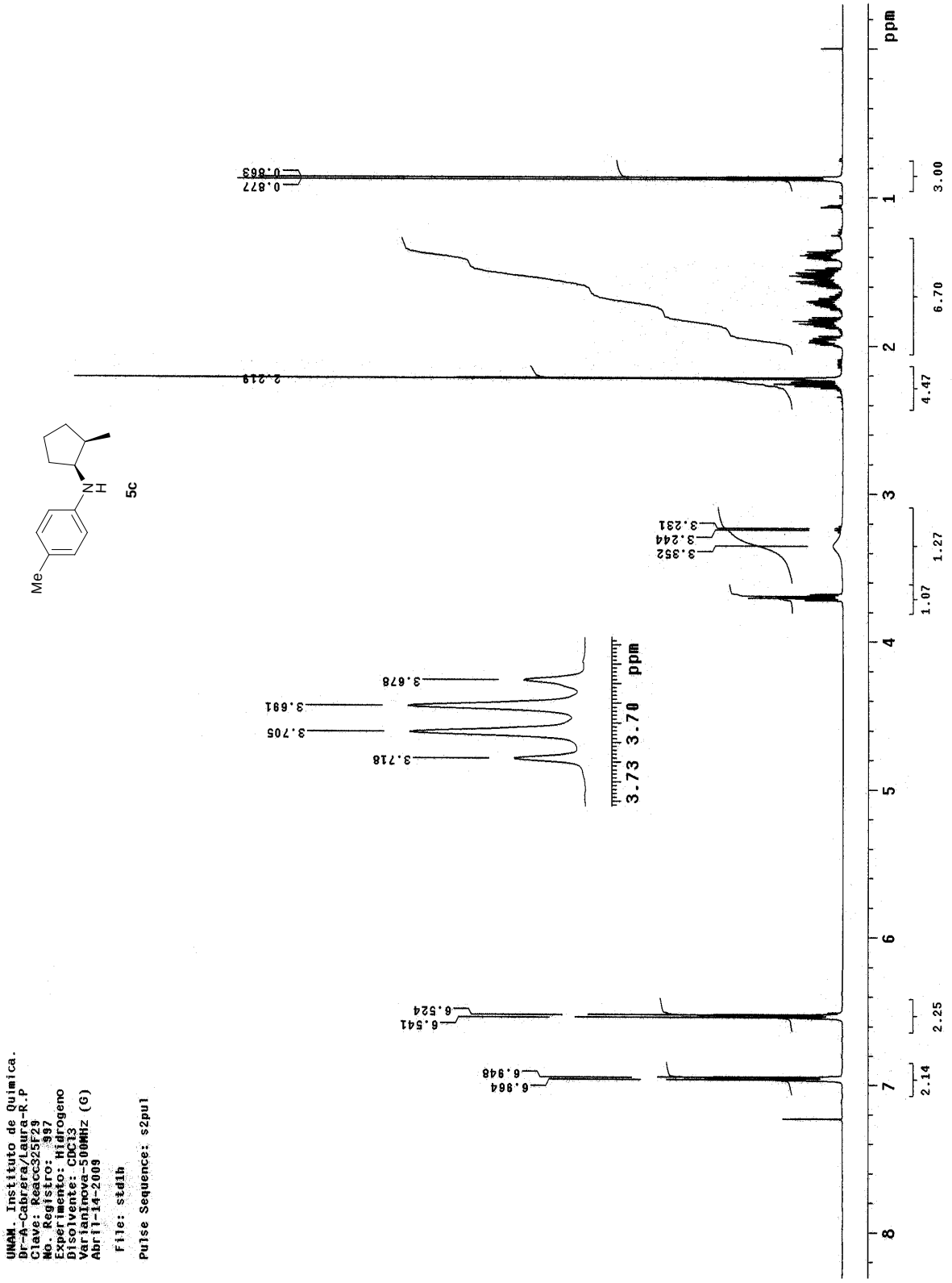


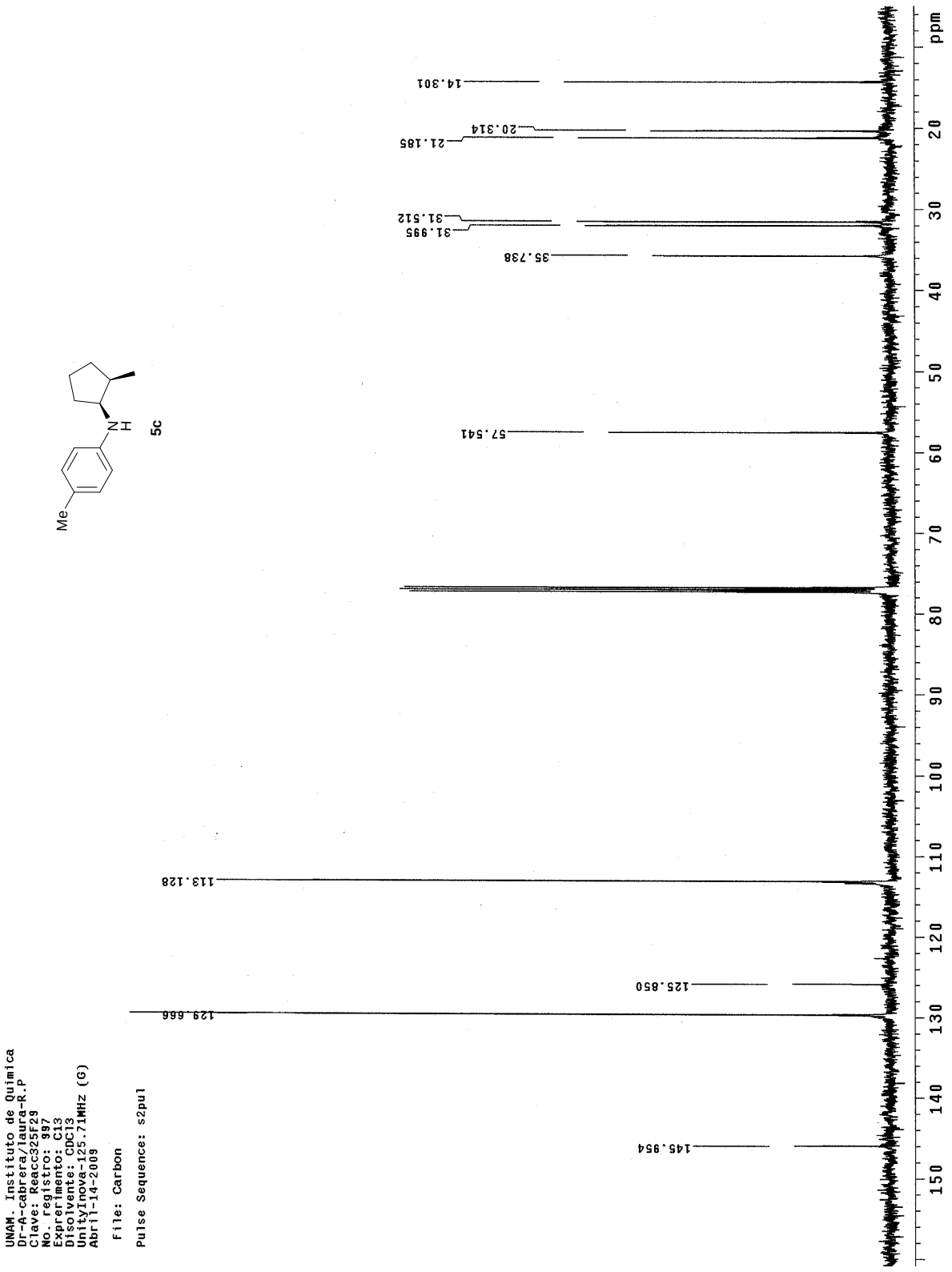

| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 30.20 | 255.25 | 2.70 | 31.90 | 4.91 | 7.51 | BB |
| 2 | 30.56 | 317.29 | 3.36 | 34.27 | 5.28 | 8.69 | BB |
| 3 | 31.25 | 8876.89 | 93.94 | 583.43 | 89.81 | 14.29 | BB |



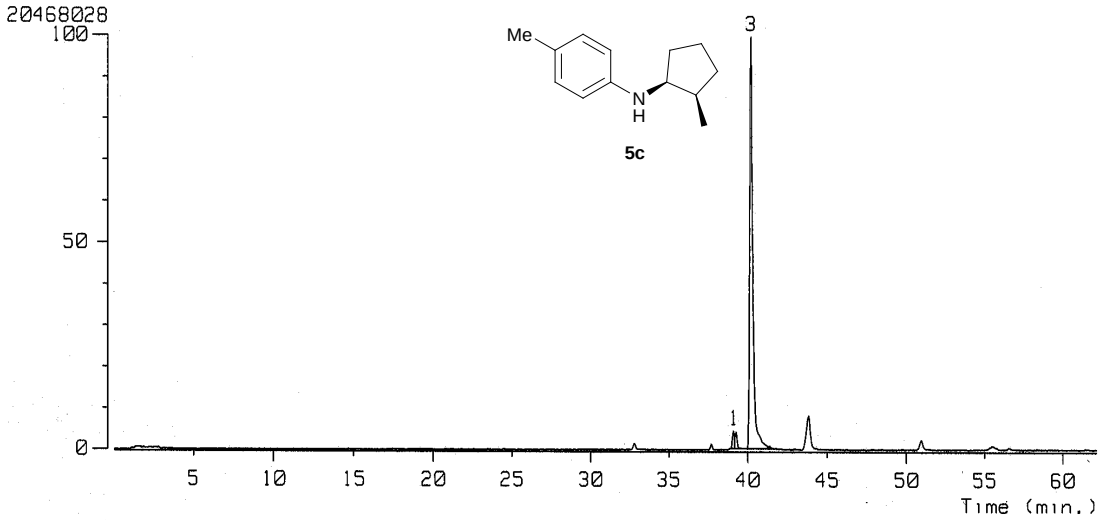



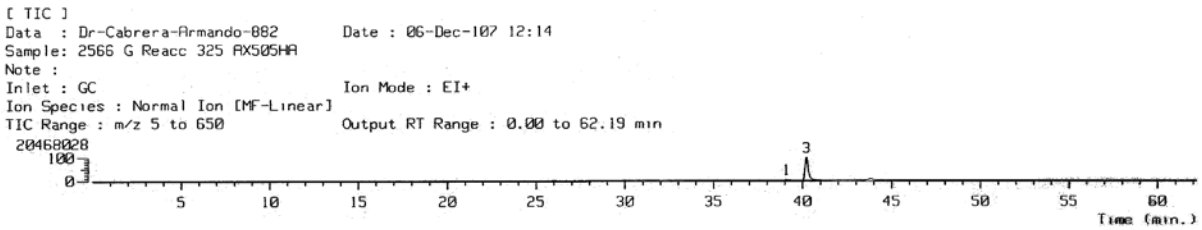

UNAM. Instituto de Quimica.
Dr-A-Cabrera/Laura-R.P
Clave: Reacc325F29
No. Registro: 997
Experimento: Hidrogeno
Disolvente: CDCl3
VarianInova-500MHz (G)
Abril-14-2009
File: std1h
Pulse Sequence: s2pul
5c
Me
N
H
0.863
0.877
2.219
3.244
3.352
3.281
3.678
3.681
3.705
3.718
3.73 3.70 ppm
6.524
6.541
6.848
6.864
ppm
1.07
1.27
4.47
6.70
3.00
2.14
2.25

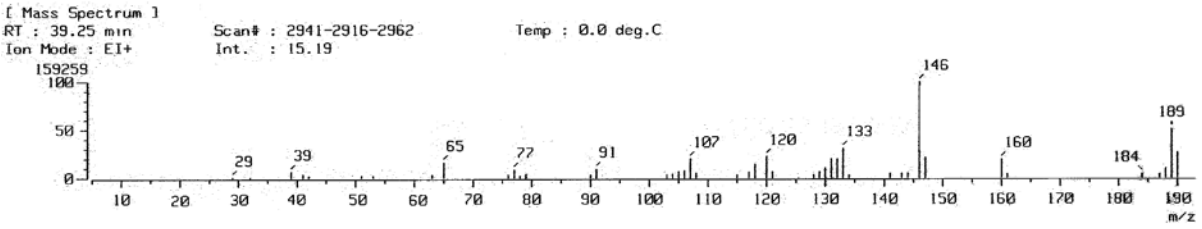

UNAM. Instituto de Quimica
Dr-A-cabrera/laura-R.P
Clave: Reacc325F29
No. registro: 997
Experimento: C13
Disolvente: CDCl3
UnityInova-125.71MHz (G)
Abril-14-2009
File: Carbon
Pulse Sequence: s2pul
5c
Me
N
H
145.954
129.666
125.850
113.128
57.541
35.738
31.995
31.512
21.185
20.314
14.301
ppm

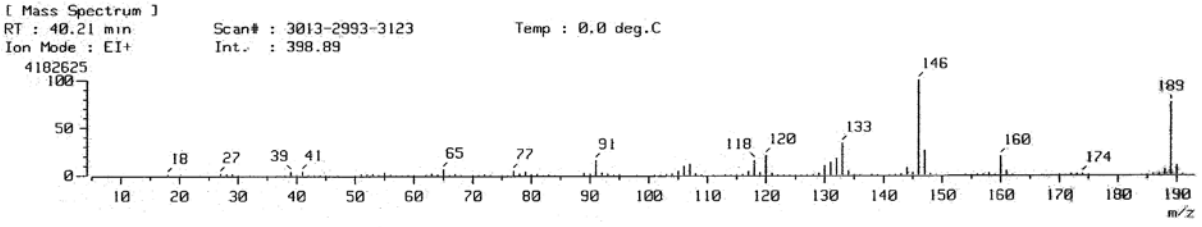
[ TIC ]
Data : Dr-Cabrera-Armando-882 Date : 06-Dec-107 12:14
Sample: 2566 G Reacc 325 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650

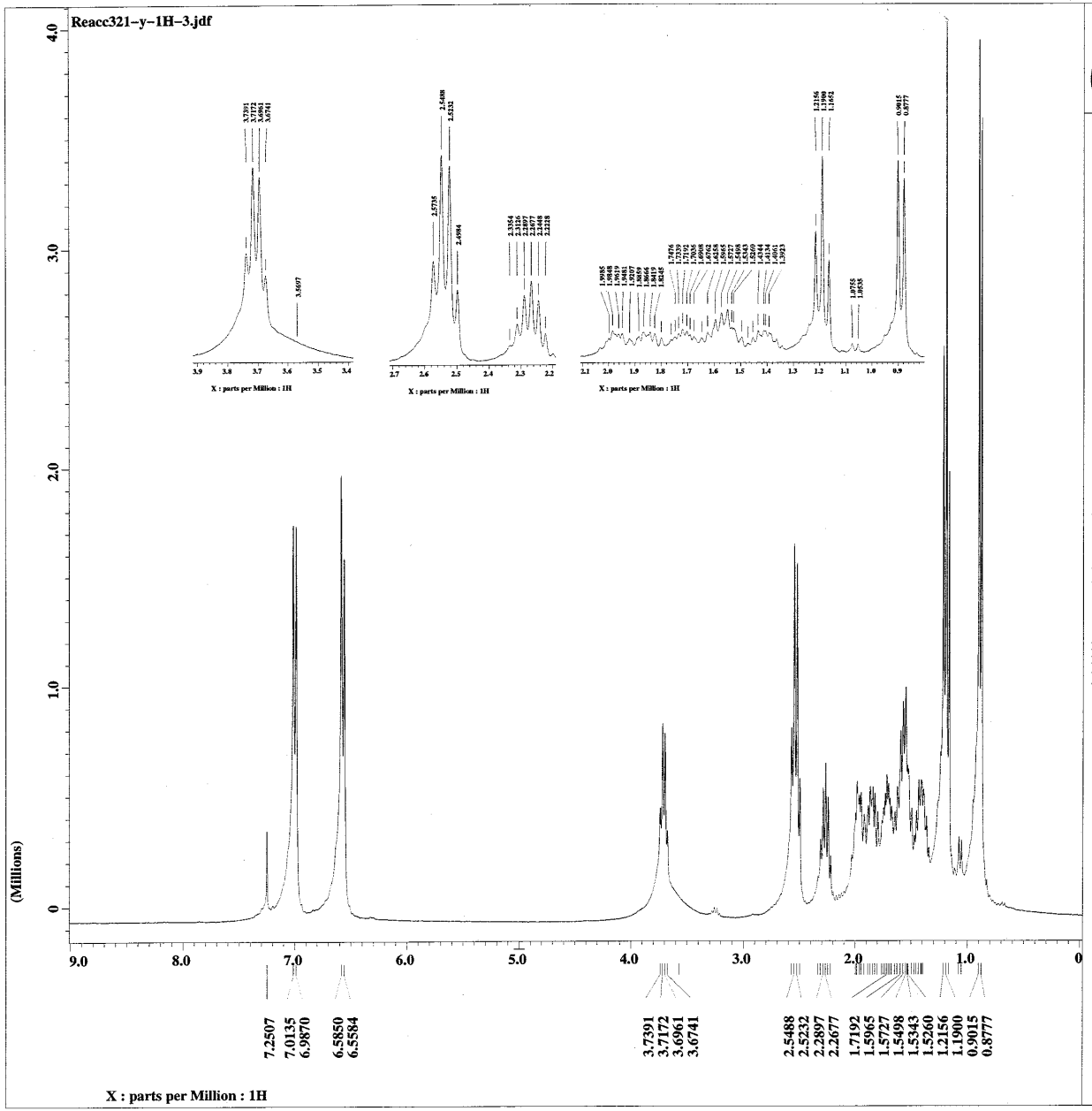



| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 39.09 | 683.64 | 2.42 | 80.41 | 3.83 | 7.98 | BV |
| 2 | 39.25 | 651.28 | 2.30 | 76.03 | 3.62 | 8.04 | VB |
| 3 | 40.21 | 26923.40 | 95.28 | 1941.51 | 92.54 | 13.02 | BB |



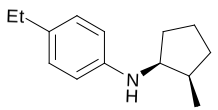



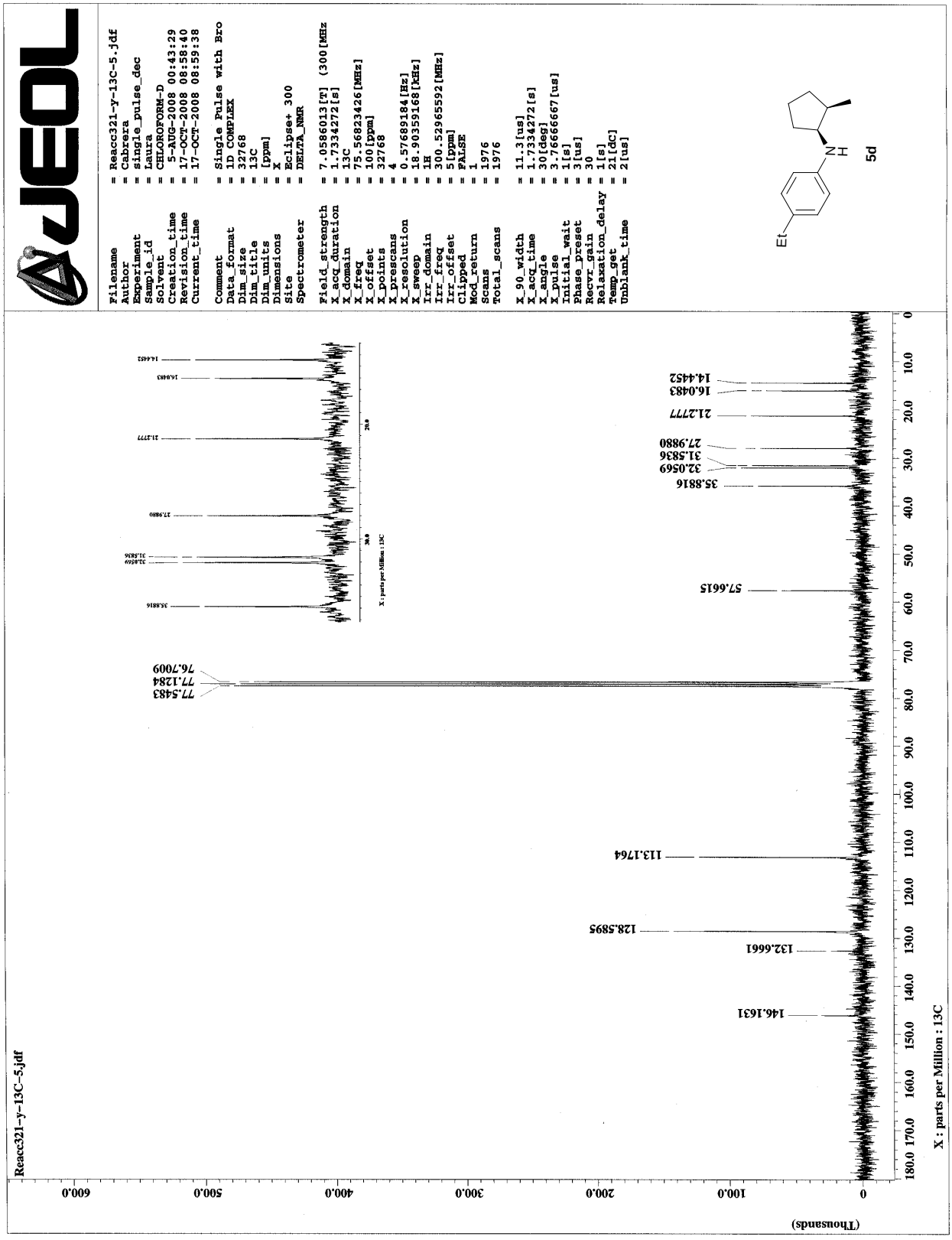



JEOL

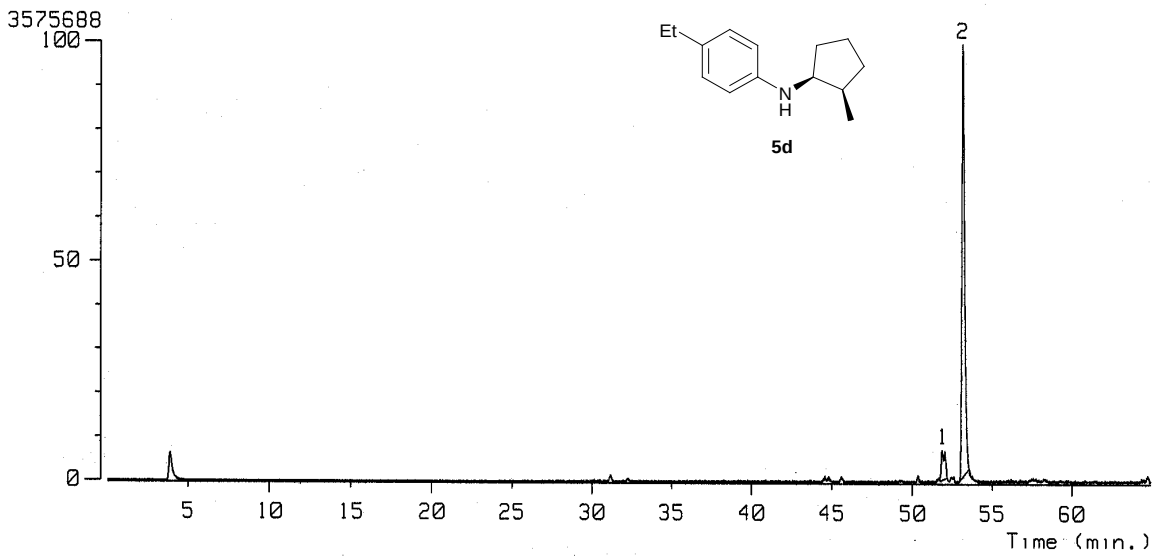
| | |
|---|---|
| Filename | = Reacc321-y-1H-3.jdf |
| Author | = Cabrera |
| Experiment | = single_pulse.exp |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 4-AUG-2008 23:12:15 |
| Revision_time | = 17-OCT-2008 07:42:20 |
| Current_time | = 17-OCT-2008 07:47:01 |
| Comment | = 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 | = 19.5[dC] |
| Unblank_time | = 2[us] |




5d

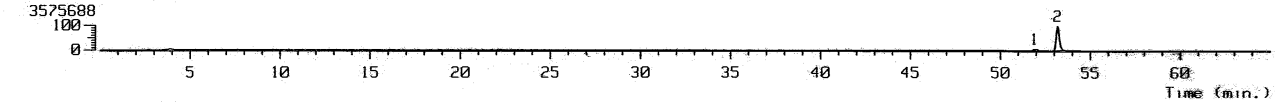

JEOL
Filename = Reacc321-y-13C-5.jdf
Author = Cabrera
Experiment = single_pulse_dec
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 5-AUG-2008 00:43:29
Revision_time = 17-OCT-2008 08:58:40
Current_time = 17-OCT-2008 08:59:38
Comment = Single Pulse with Bro
Data_format = 1D COMPLEX
Dim_size = 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 = 1976
Total_scans = 1976
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[dC]
Unblank_time = 2[us]
Et
N
H
5d
Reacc321-y-13C-5.jdf
(Thousands)
X : parts per Million : 13C
146.1631
132.6661
128.5895
113.1764
77.5483
77.1284
76.7009
57.6615
35.8816
32.0569
31.5836
27.9880
21.2777
16.0483
14.4452

[ TIC ]
Data : Dr-Cabrera-Armando-137 Date : 17-Oct-120 10:08
Sample: 2435 G2 Reac 321 y JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 33 to 650

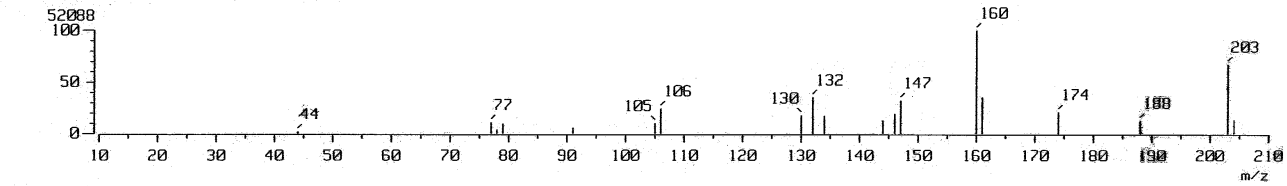



| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 51.86 | 376.07 | 8.32 | 22.97 | 6.39 | 15.37 | BB |
| 2 | 53.16 | 4143.46 | 91.68 | 336.27 | 93.61 | 11.57 | BB |

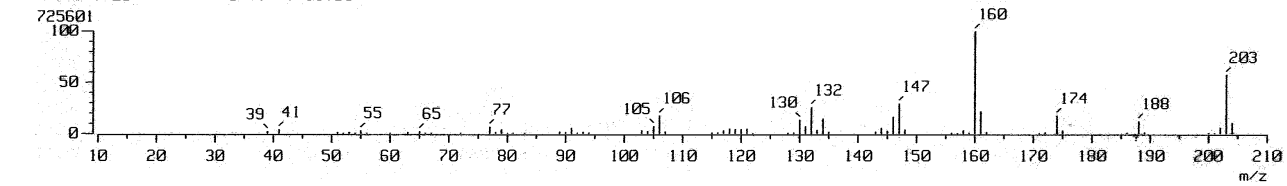
[ TIC ]
Data : Dr-Cabrera-Armando-137 Date : 17-Oct-120 10:08
Sample: 2435 G2 Reac 321 y JeolAX505HA
Note : 5 horas
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 33 to 650 Output RT Range : 0.00 to 65.00 min

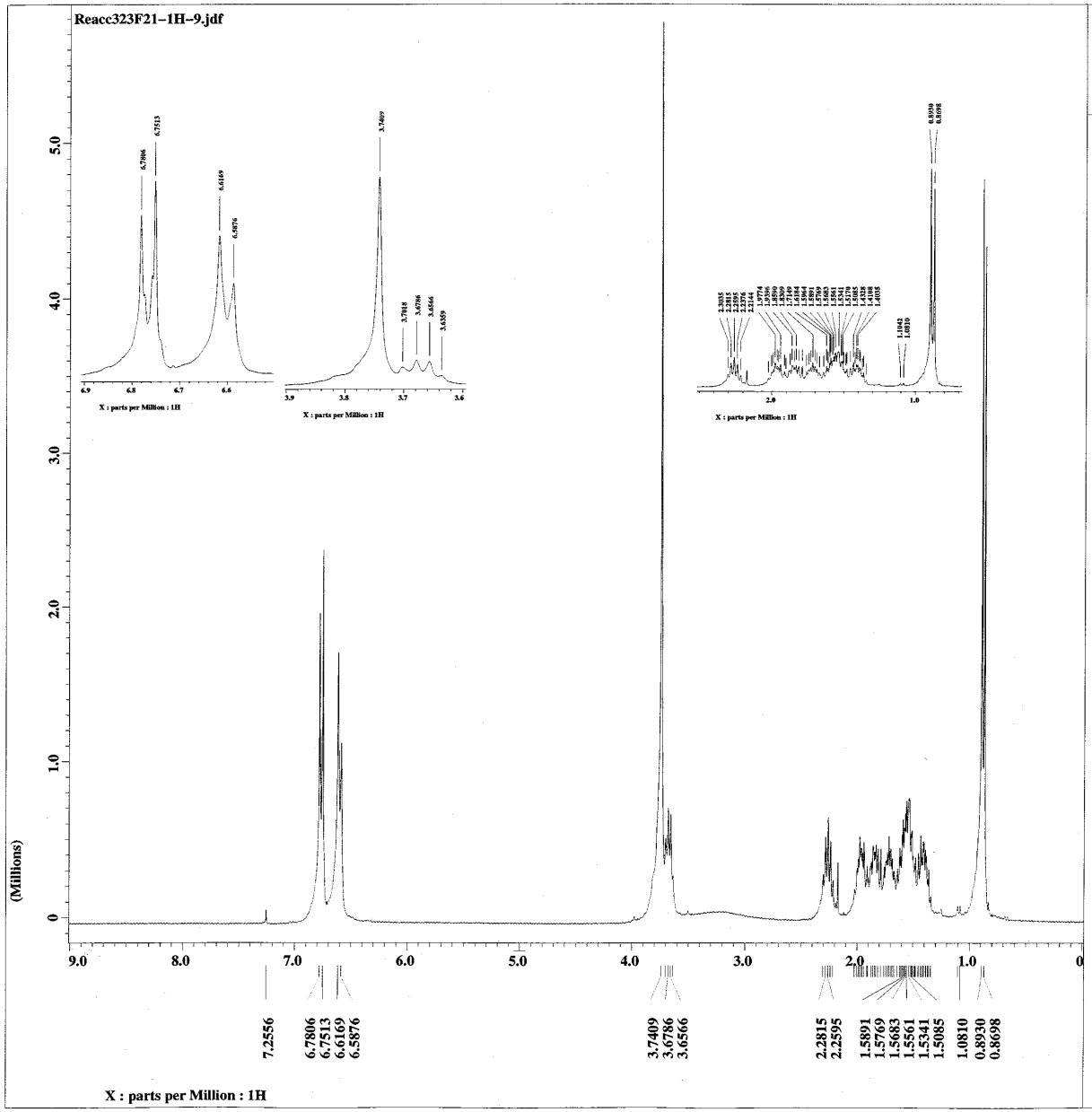



[ Mass Spectrum ]
RT : 51.86 min Scan# : 4465-4453-4491 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 4.97



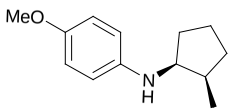

[ Mass Spectrum ]
RT : 53.16 min Scan# : 4577-4557-4609 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 69.20



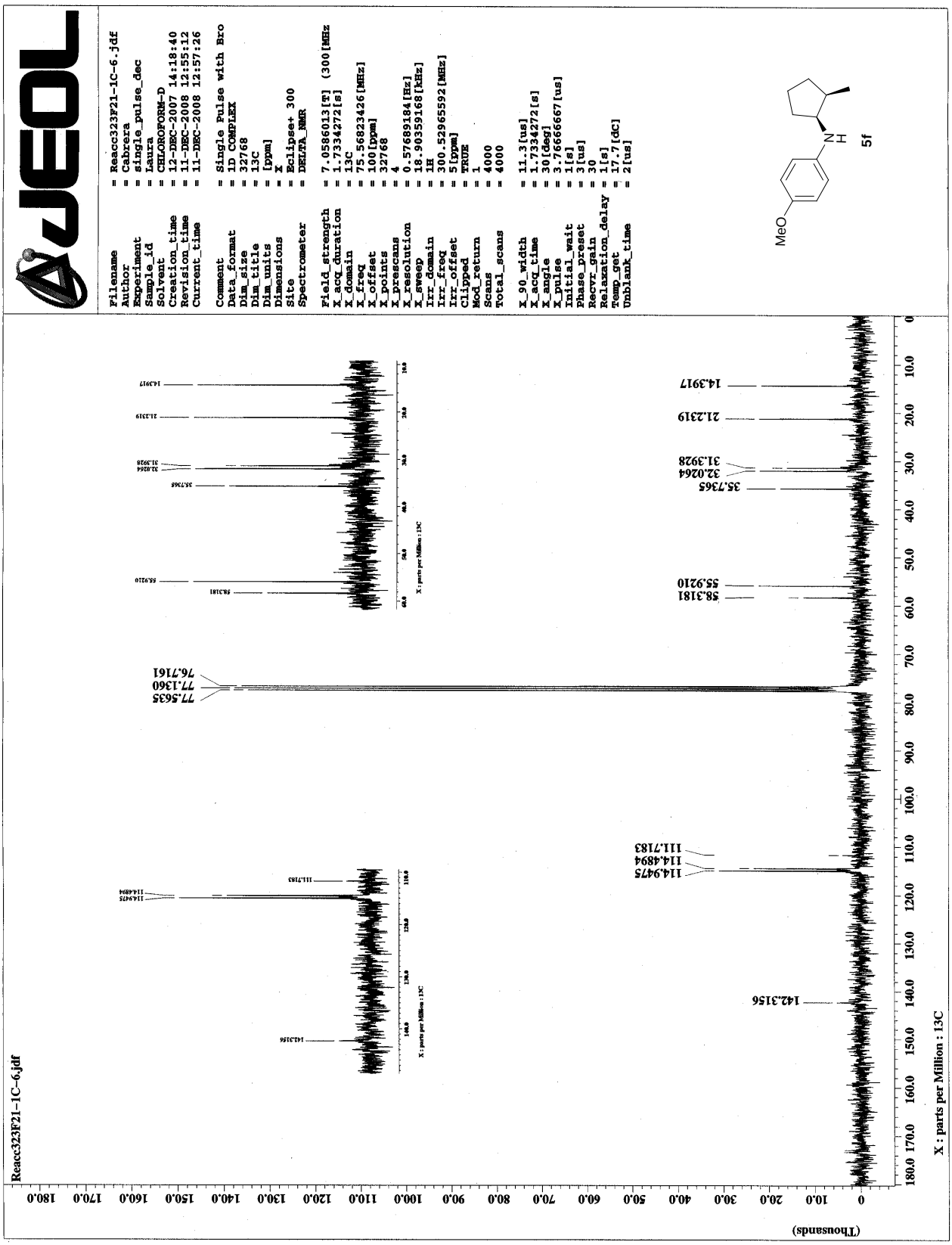



JEOL

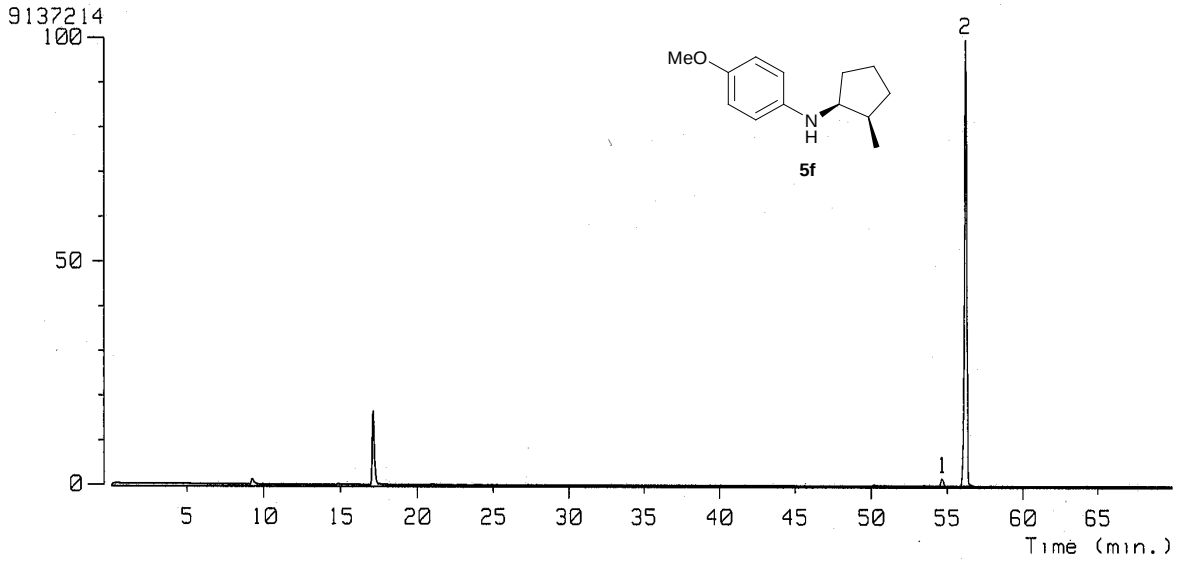
| | |
|---|---|
| Filename | = Reacc323F21-1H-9.jdf |
| Author | = Cabrera |
| Experiment | = single_pulse.exp |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 12-DEC-2007 10:54:12 |
| Revision_time | = 11-DEC-2008 12:52:37 |
| Current_time | = 11-DEC-2008 12:53:37 |
| Comment | = 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 | = 2.7246592[s] |
| X_domain | = 1H |
| X_freq | = 300.52965592[MHz] |
| X_offset | = 5[ppm] |
| X_points | = 16384 |
| X_prescans | = 0 |
| X_resolution | = 0.36701838[Hz] |
| X_sweep | = 6.0132291[kHz] |
| Clipped | = TRUE |
| Mod_return | = 1 |
| Scans | = 32 |
| Total_scans | = 32 |
| X_90_width | = 9.5[us] |
| X_acq_time | = 2.7246592[s] |
| X_angle | = 45[deg] |
| X_pulse | = 4.75[us] |
| Initial_wait | = 1[s] |
| Phase_preset | = 3[us] |
| Recvr_gain | = 22 |
| Relaxation_delay | = 4[s] |
| Temp_get | = 15.8[dC] |
| Unblank_time | = 2[us] |




5f

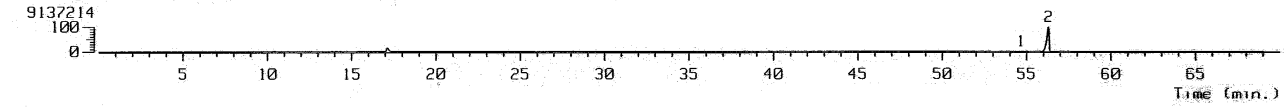

Reacc323F21-1C-6.jdf
Filename = Reacc323F21-1C-6.jdf
Author = Cabrera
Experiment = single_pulse_dec
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 12-DEC-2007 14:18:40
Revision_time = 11-DEC-2008 12:55:12
Current_time = 11-DEC-2008 12:57:26
Comment = Single Pulse with Bro
Data_format = 1D COMPLEX
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 = 1.73342272[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 = TRUE
Mod_return = 1
Scans = 4000
Total_scans = 4000
X_90_width = 11.3[us]
X_acq_time = 1.73342272[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 = 17.7[dC]
Unblank_time = 2[us]
MeO
5f
142.3156
114.9475
114.4894
111.7183
77.5635
77.1360
76.7161
58.3181
55.9210
35.7365
32.0264
31.3928
21.2319
14.3917
(Thousands)
X : parts per Million : 13C

[ TIC ]
Data : Dr-Cabrera-Armando-878 Date : 28-Nov-107 11:49
Sample: 2389 G Reacc 323 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650

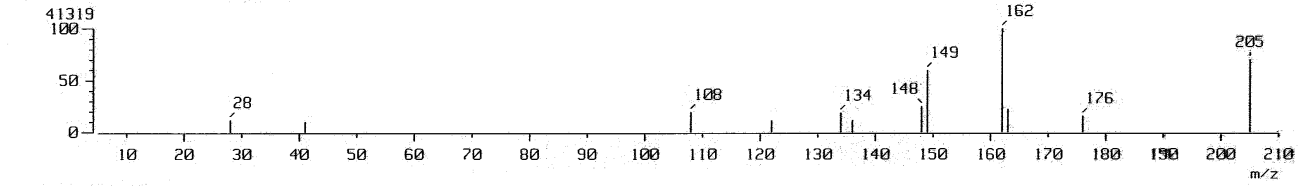



| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 54.66 | 181.96 | 2.02 | 14.44 | 1.63 | 11.83 | BB |
| 2 | 56.28 | 8829.38 | 97.98 | 871.36 | 98.37 | 9.51 | BB |

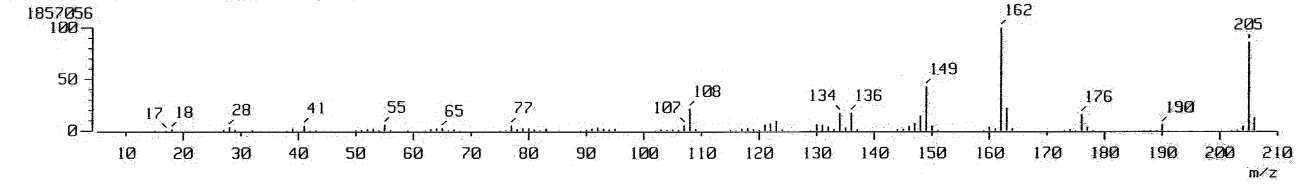
[ TIC ]
Data : Dr-Cabrera-Armando-878 Date : 28-Nov-107 11:49
Sample: 2389 G Reacc 323 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 5 to 650 Output RT Range : 0.00 to 69.99 min

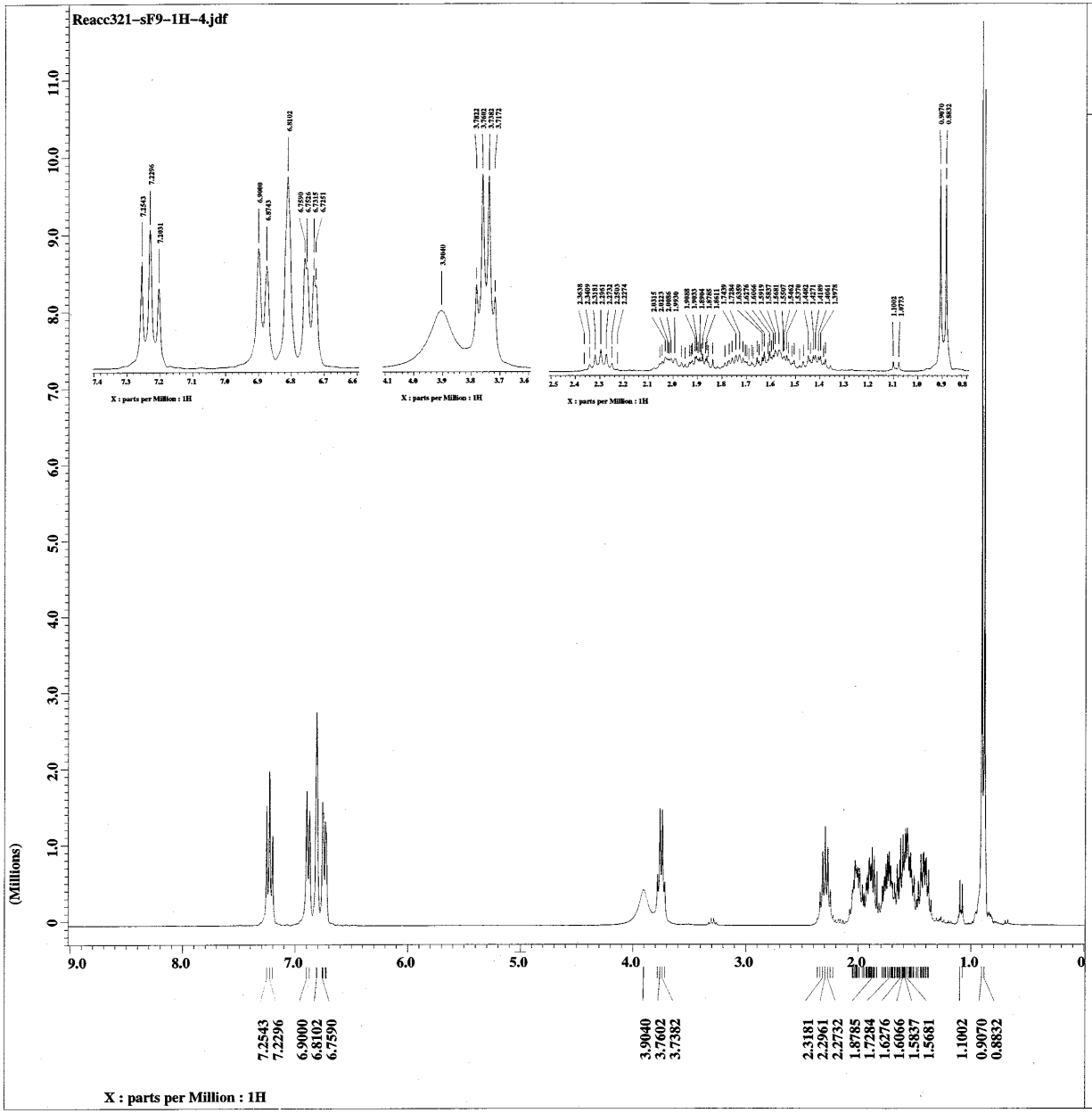



[ Mass Spectrum ]
RT : 54.66 min Scan# : 4100-4085-4121 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 3.94

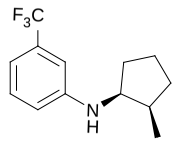



[ Mass Spectrum ]
RT : 56.28 min Scan# : 4222-4188-4266 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 177.10

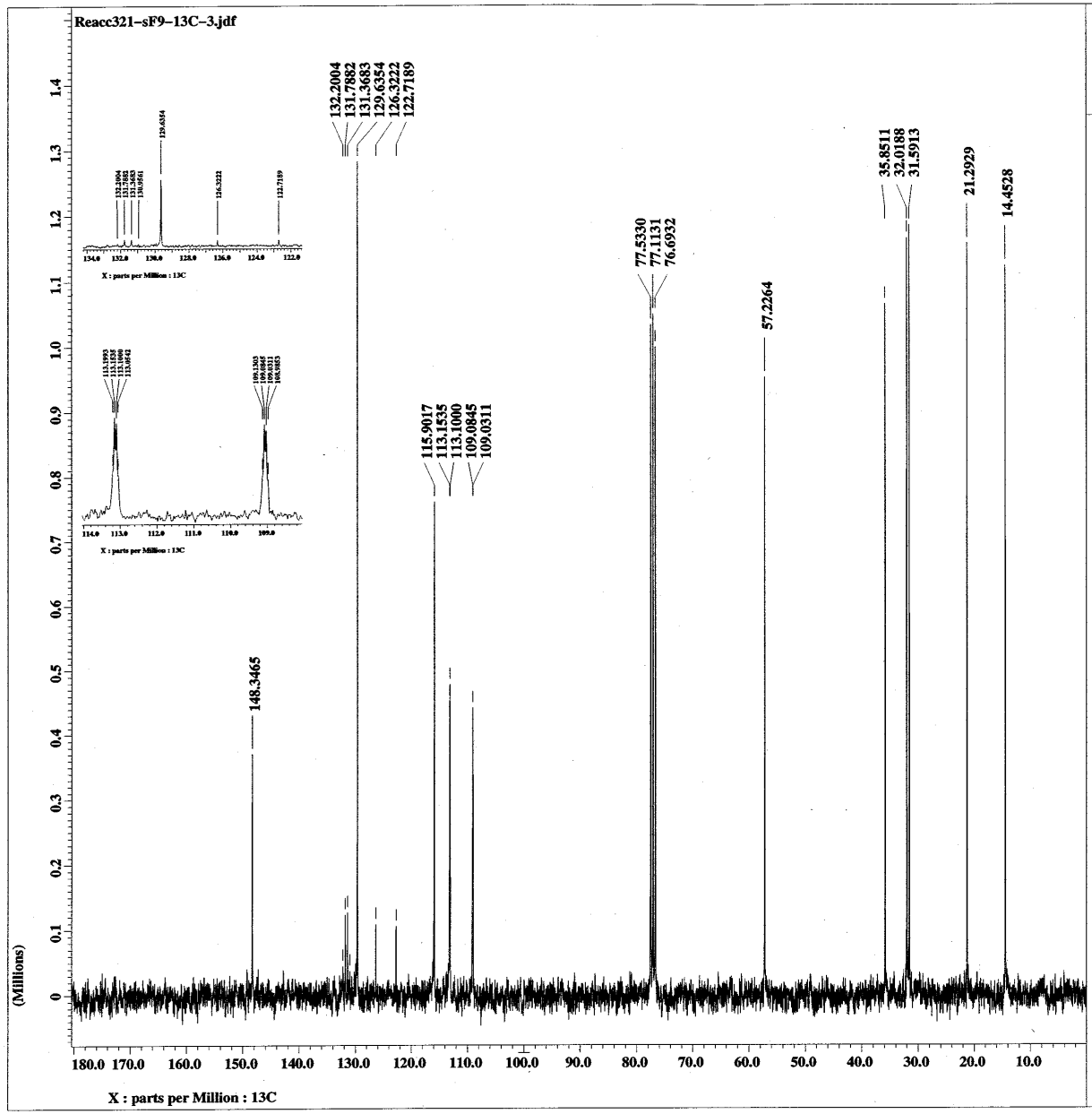





| | |
|---|---|
| Filename | = Reacc321-sF9-1H-4.jdf |
| Author | = Cabrera |
| Experiment | = single_pulse.exp |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 8-AUG-2008 08:02:11 |
| Revision_time | = 20-OCT-2008 16:16:52 |
| Current_time | = 20-OCT-2008 16:17:56 |
| Comment | = 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 | = 19.5[dC] |
| Unblank_time | = 2[us] |

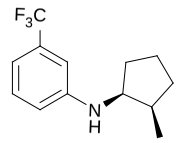



5g



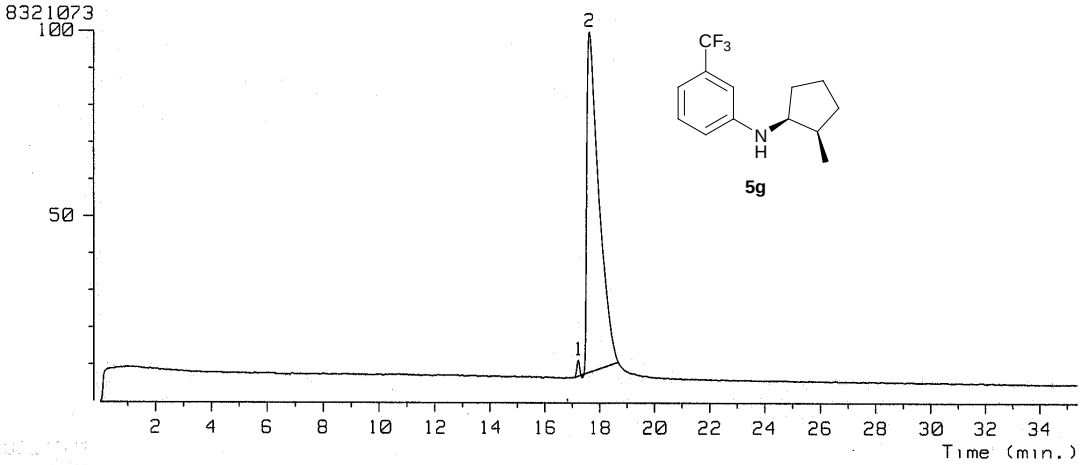

JEOL

| | |
|---|---|
| Filename | = Reacc321-sF9-13C-3.jd |
| Author | = Cabrera |
| Experiment | = single_pulse_dec |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 8-AUG-2008 08:13:29 |
| Revision_time | = 20-OCT-2008 16:25:04 |
| Current_time | = 20-OCT-2008 16:25:52 |
| 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 | = 221 |
| Total_scans | = 221 |
| 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 | = 21.3[dC] |
| Unblank_time | = 2[us] |



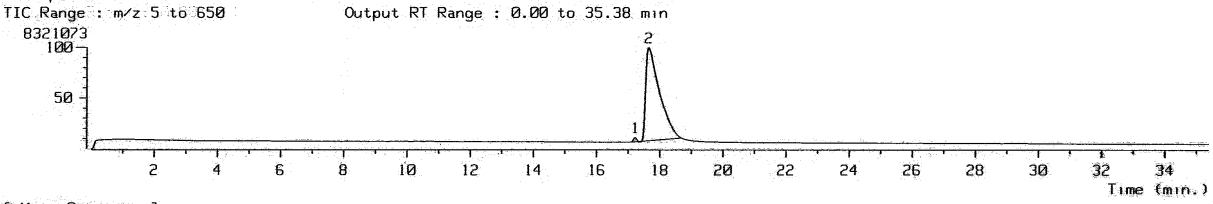

5g

[ TIC ]
Data : Dr-Cabrera-Armando-505 Date : 26-Mar-120 10:16
Sample: 857 G Rx-321-s AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650

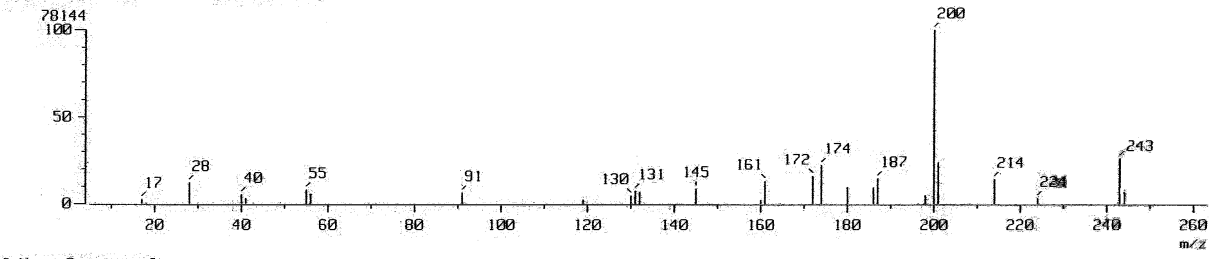



| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 17.22 | 211.29 | 0.95 | 33.69 | 4.41 | 5.89 | BV |
| 2 | 17.64 | 22132.42 | 99.05 | 729.55 | 95.59 | 28.49 | VB |

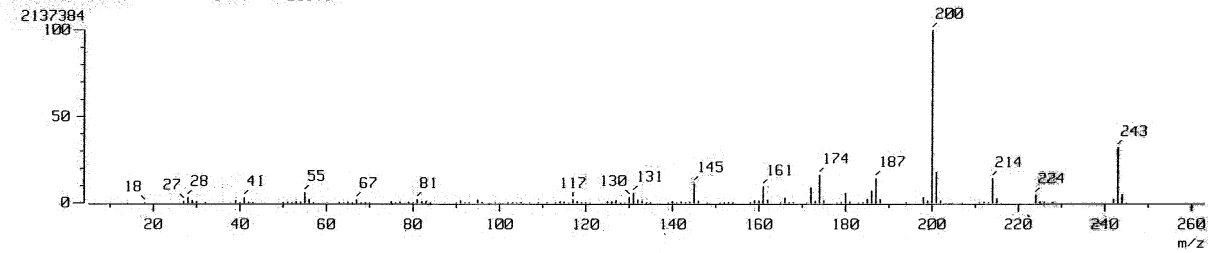
[ TIC ]
Data : Dr-Cabrera-Armando-505 Date : 26-Mar-120 10:16
Sample: 857 G Rx-321-s AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 5 to 650 Output RT Range : 0.00 to 35.38 min




[ Mass Spectrum ]
RT : 17.22 min Scan# : 1292-1283-1402 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 7.45




[ Mass Spectrum ]
RT : 17.64 min Scan# : 1324-1283-1402 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 203.84



INSTITUTO DE QUIMICA, UNAM/ EHS

Dr. A. Cabrera/ Laura R. P.

Clave. Reacc514

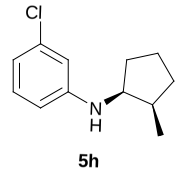
Disolvente: CDCl3

Hidrogeno-1

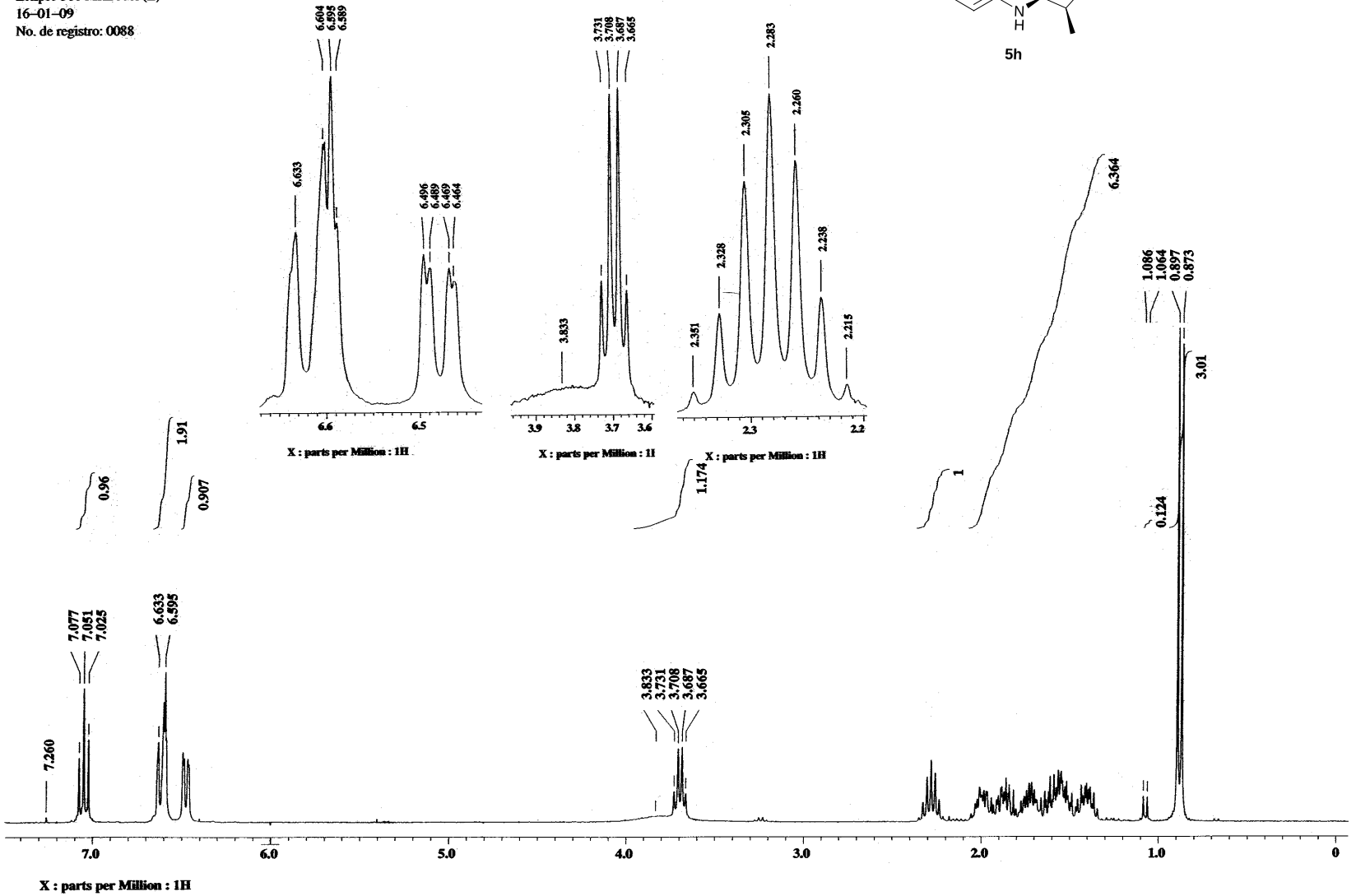
Eclipse 300 MHz Jeol (E)

16-01-09

No. de registro: 0088




5h



INSTITUTO DE QUIMICA, UNAM/ EHS

Dr. A. Cabrera/ Laura R. P.

Clave: Reacc514–2

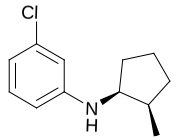
Disolvente: CDCl3

Carbono–13

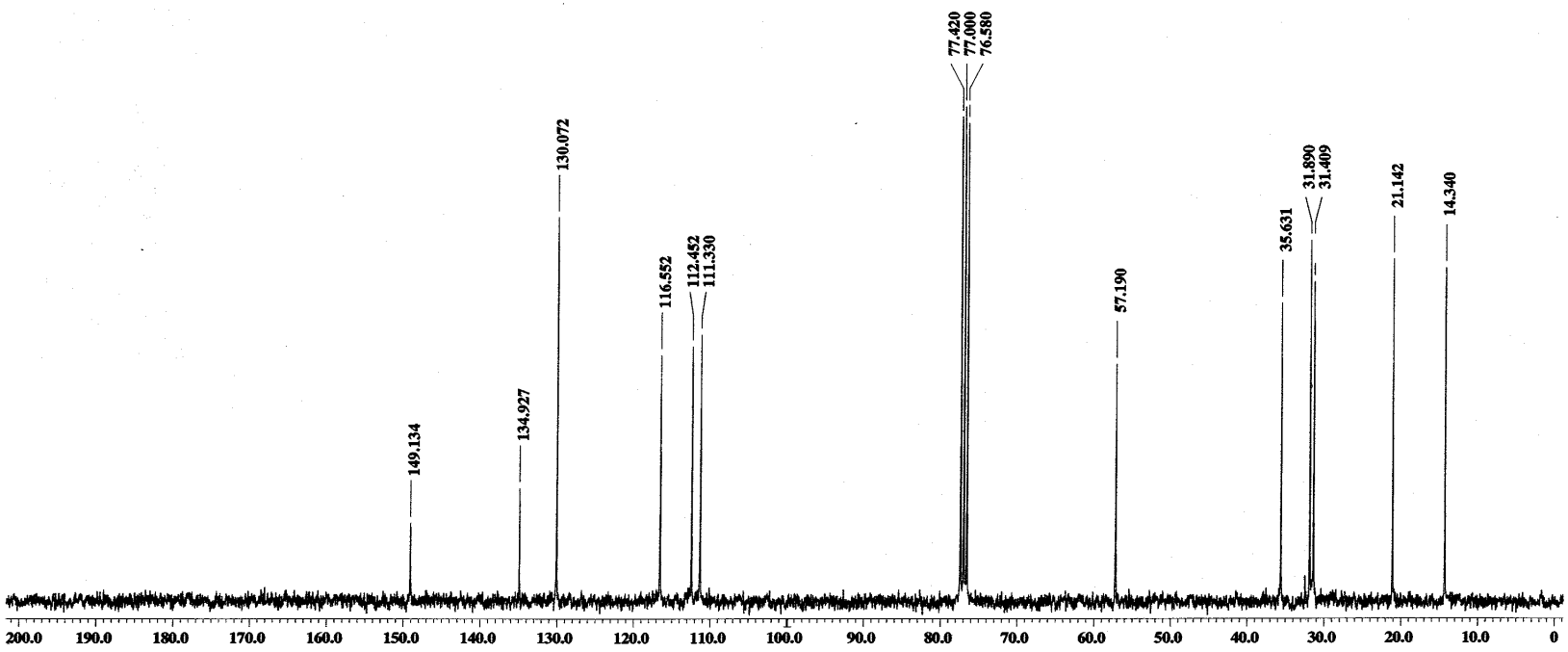
Eclipse 300 MHz Jeol (E)

26–01–09

No. de registro: 0261

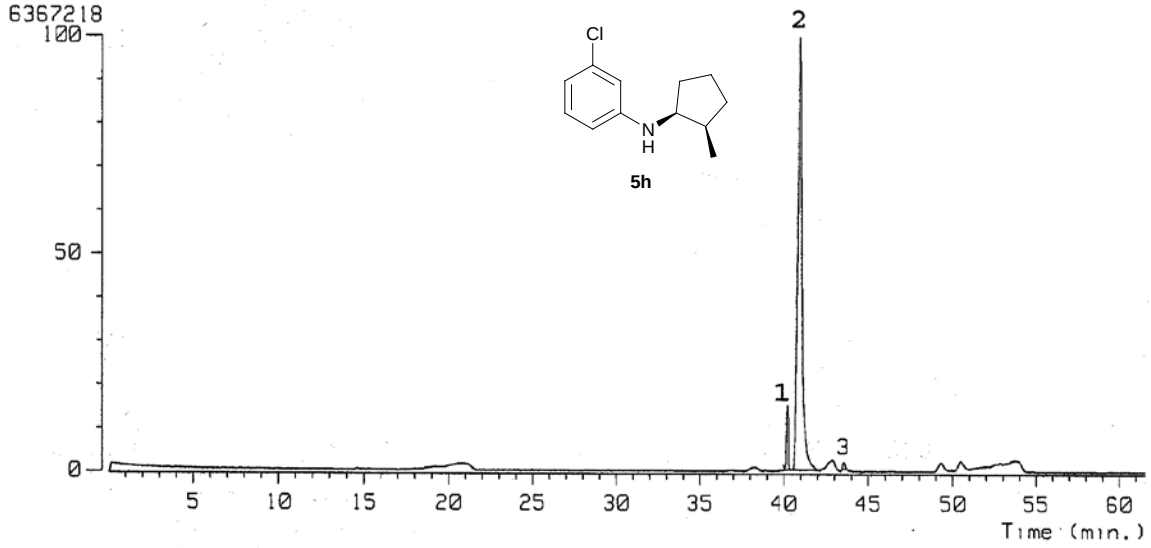



5h




X : parts per Million : 13C

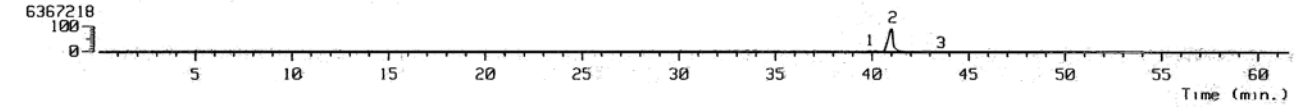
[ TIC ]
Data : Dr-Cabrera-Armando-922 Date : 14-Jan-120 12:05
Sample: 57 G reacc 514 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650



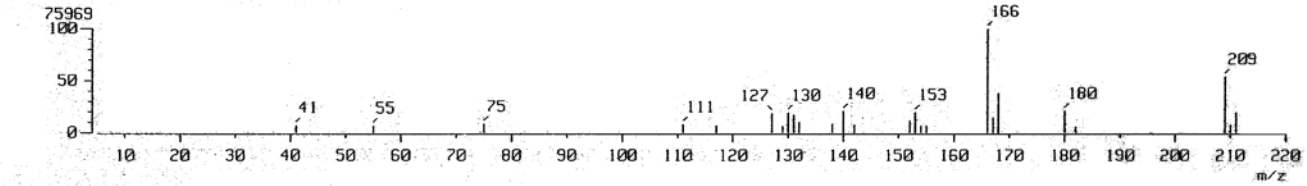

5h

| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 40.17 | 474.94 | 4.23 | 55.11 | 8.27 | 16.16 | VB |
| 2 | 41.00 | 10678.02 | 95.09 | 602.60 | 90.43 | 16.64 | BB |
| 3 | 43.55 | 6.68 | 0.68 | 8.67 | 1.30 | 8.30 | BB |

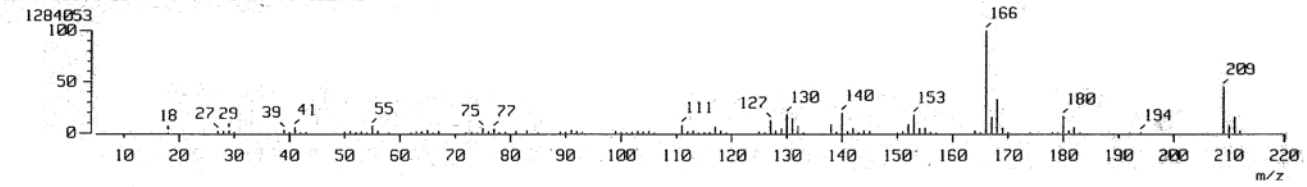
[ TIC ]
Data : Dr-Cabrera-Armando-922 Date : 14-Jan-120 12:05
Sample: 57 G reacc 514 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 5 to 650 Output RT Range : 0.00 to 61.56 min

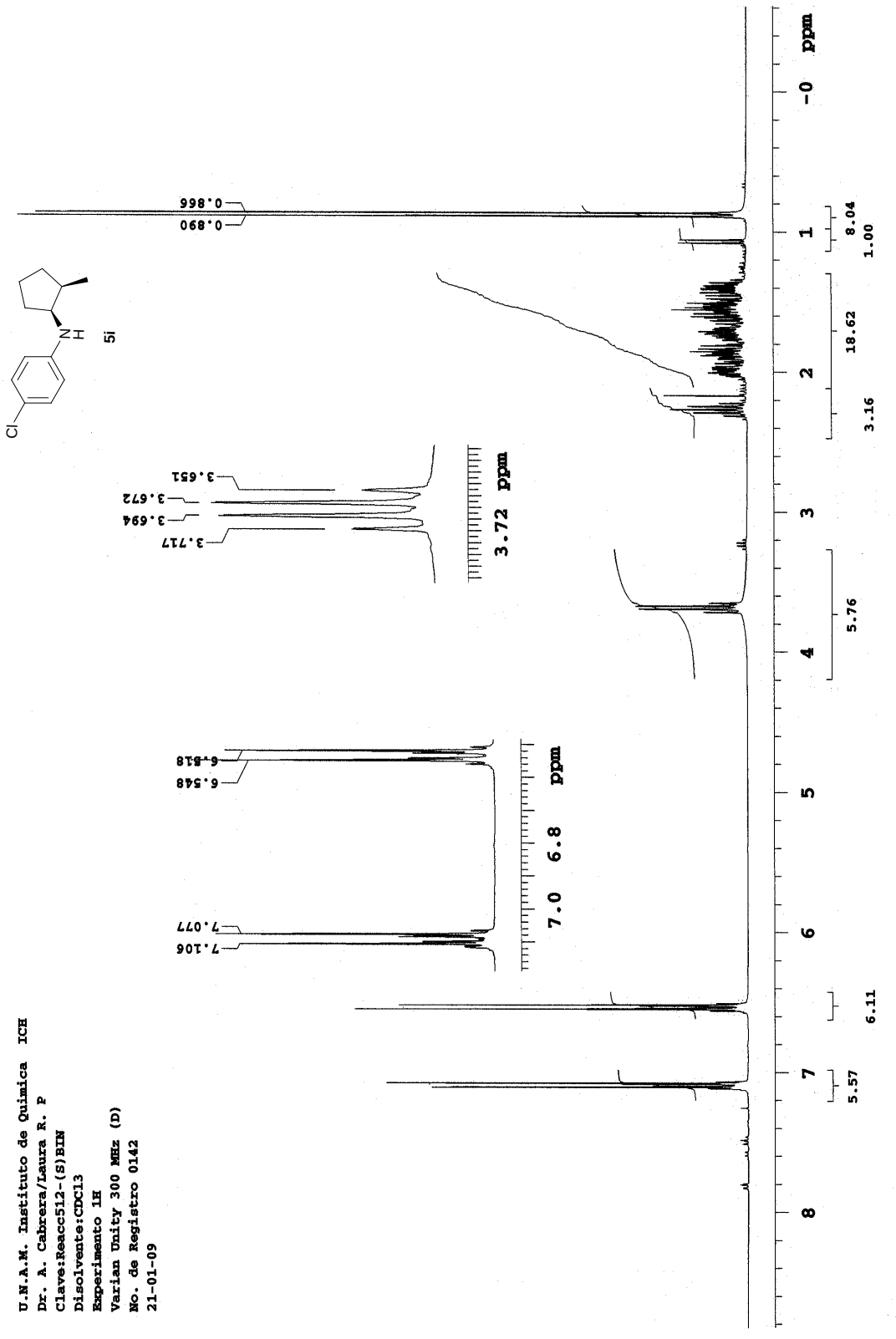


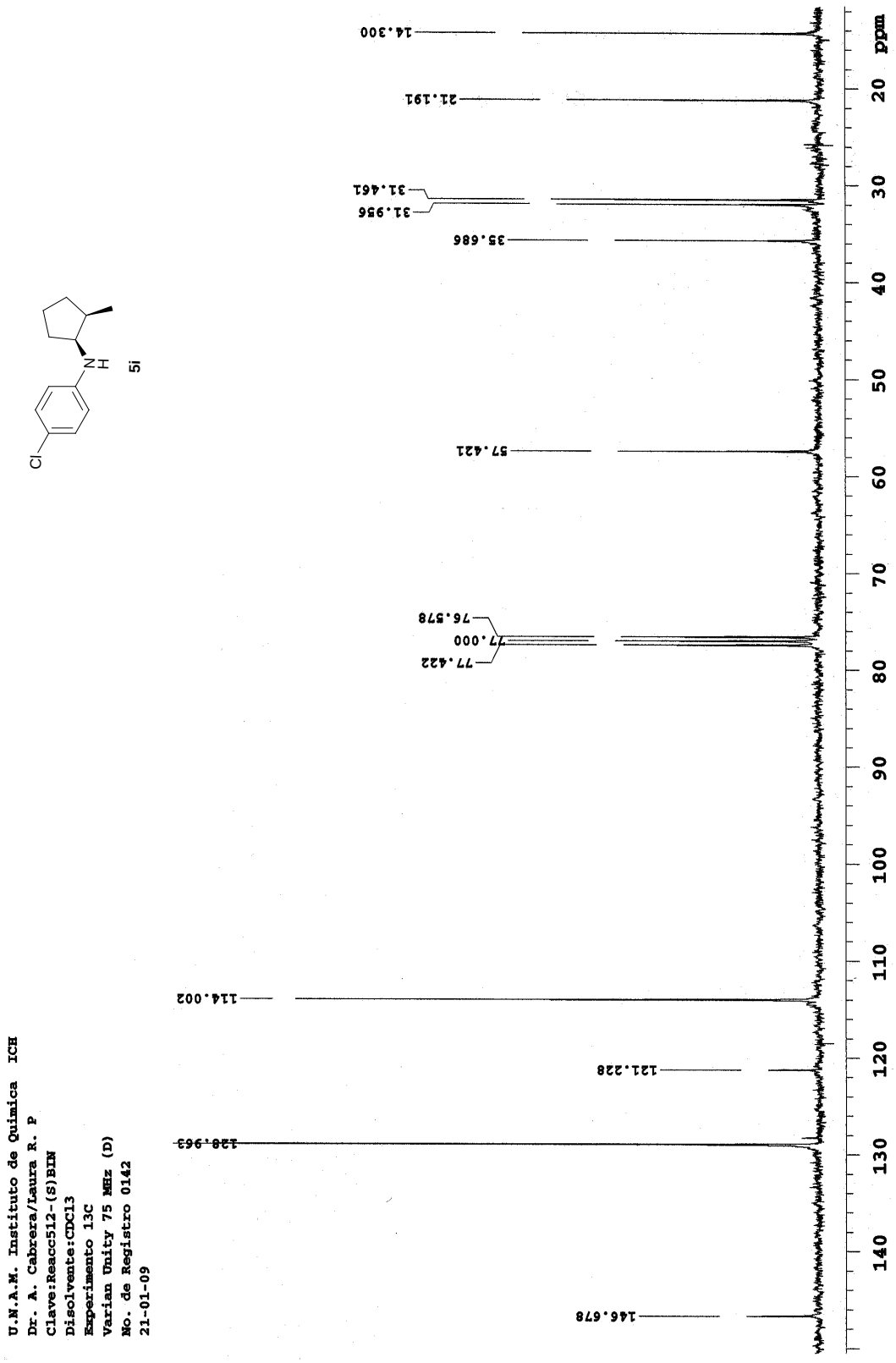

[ Mass Spectrum ]
RT : 40.17 min Scan# : 3010-2977-302 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 7.24



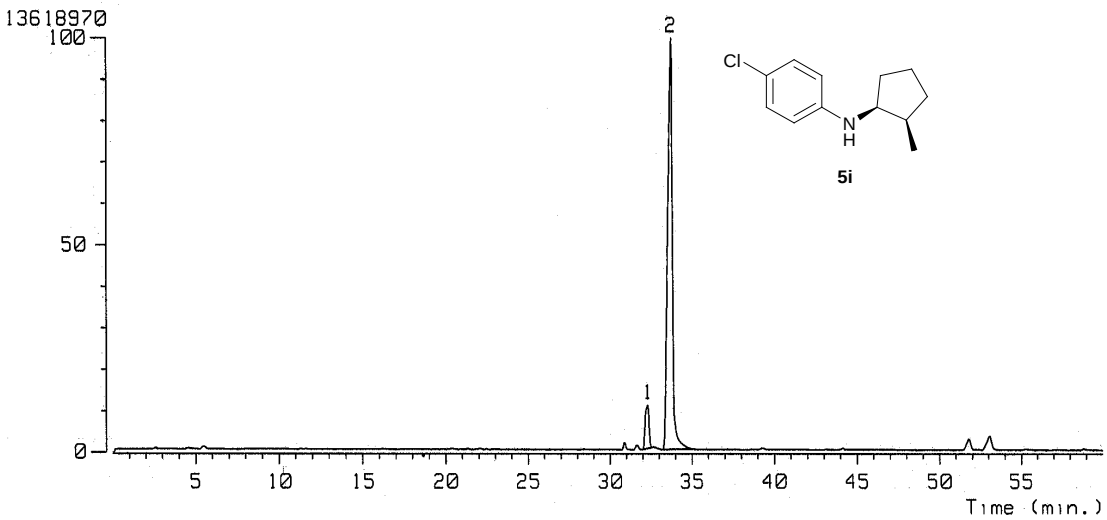

[ Mass Spectrum ]
RT : 41.00 min Scan# : 3072-3035-3154 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 122.46




U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc512-(S)BIN
Disolvente:CDCl3
Experimento 1H
Varian Unity 300 MHz (D)
No. de Registro 0142
21-01-09
5i
Cl
N
H
0.890
0.866
3.717
3.694
3.672
3.651
3.72 ppm
6.548
6.518
7.106
7.077
7.0 6.8 ppm
ppm
5.57
6.11
5.76
3.16
18.62
1.00
8.04

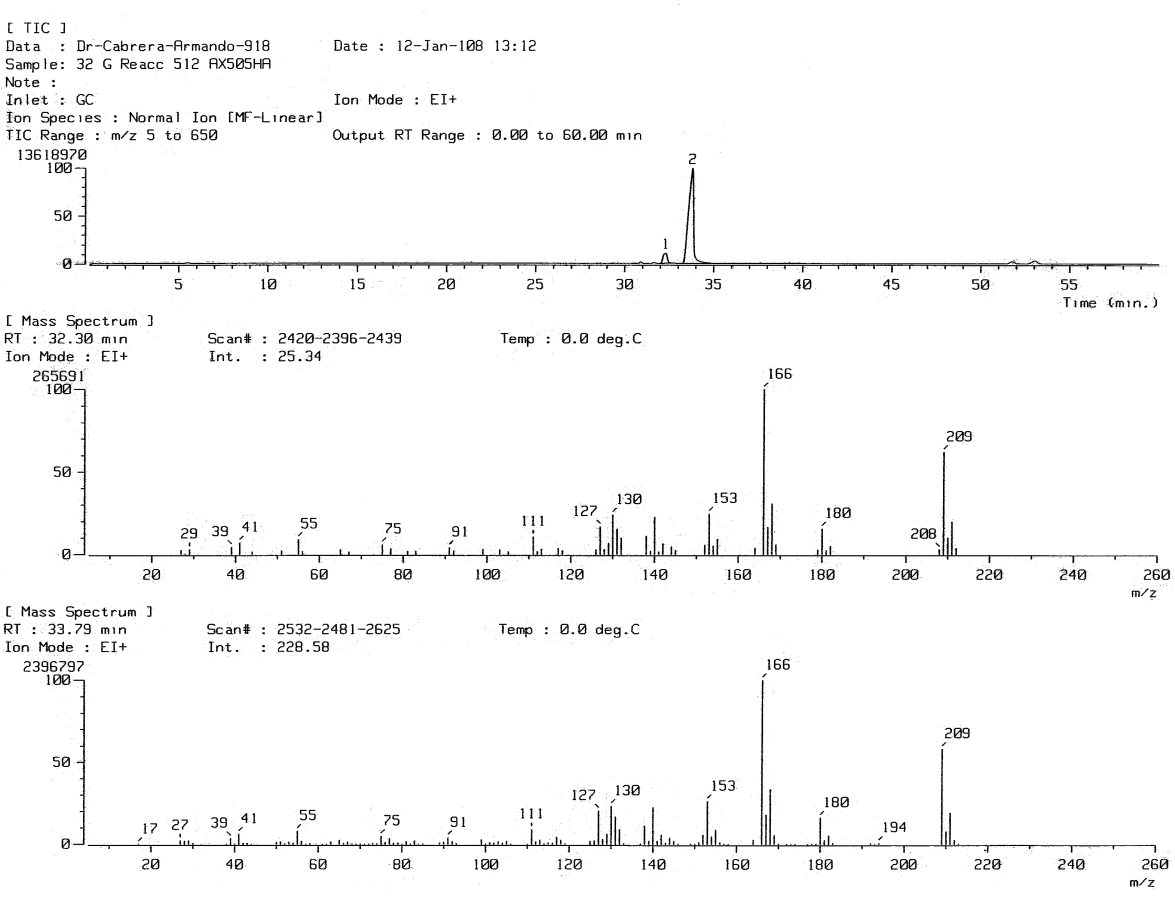

U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc512-(S)BIN
Disolvente:CDCl3
Experimento 13C
Varian Unity 75 MHz (D)
No. de Registro 0142
21-01-09
5i
Cl
N
H
146.678
138.963
121.228
114.002
77.422
77.000
76.578
57.421
35.686
31.956
31.461
21.191
14.300
ppm

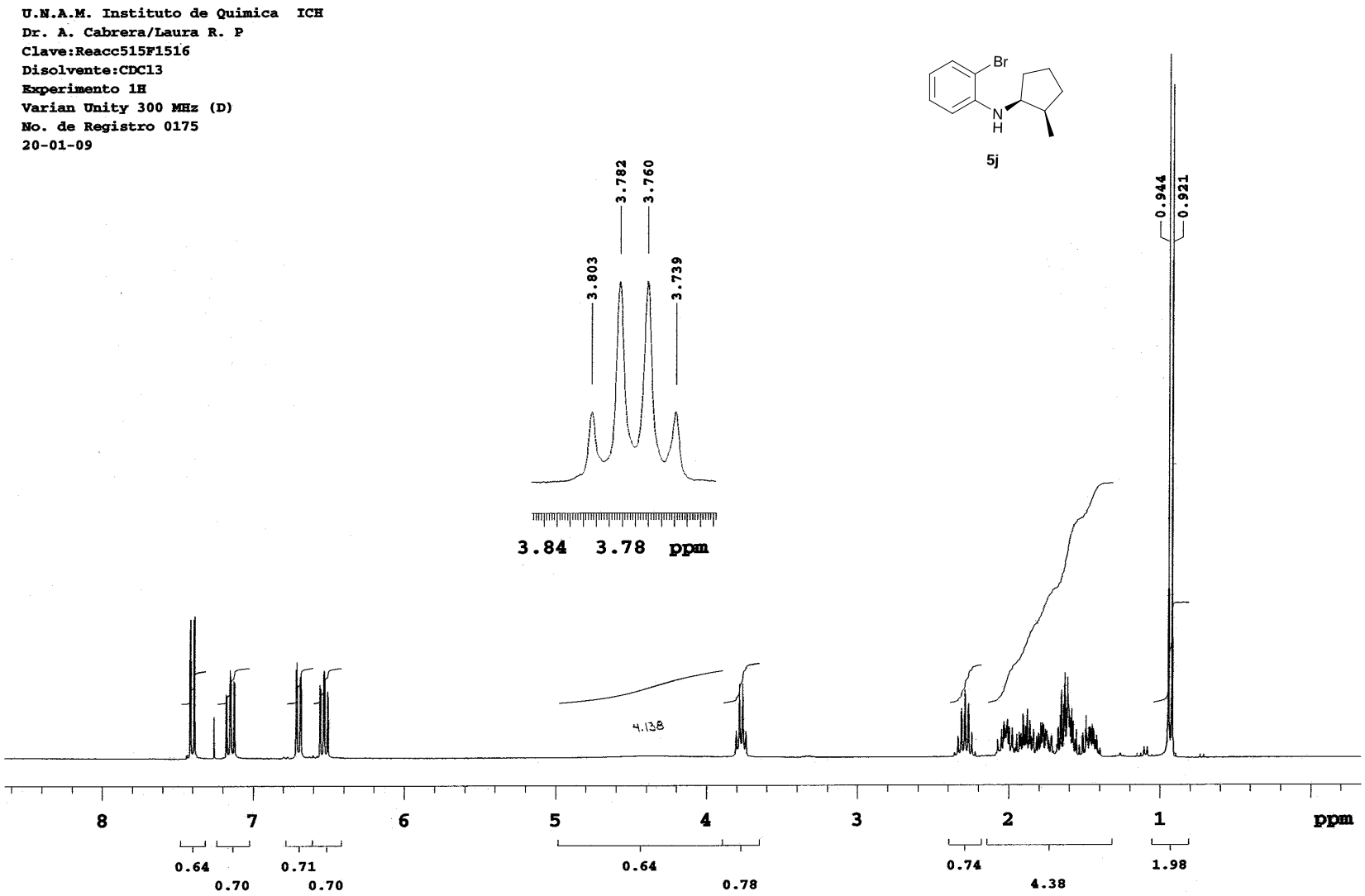
[ TIC ]
Data : Dr-Cabrera-Armando-918 Date : 12-Jan-108 13:12
Sample: 32 G Reacc 512 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650



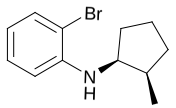

5i

| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 32.30 | 2149.23 | 7.73 | 134.85 | 9.47 | 14.97 | BB |
| 2 | 33.79 | 25647.76 | 92.27 | 1288.72 | 90.53 | 18.69 | BB |



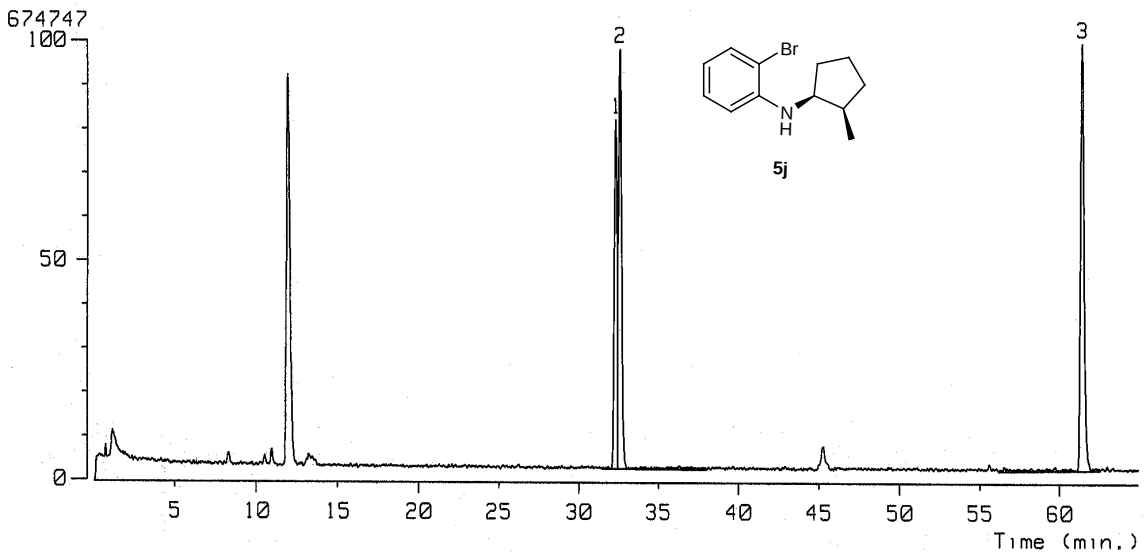

U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc515F1516
Disolvente:CDCl3
Experimento 1H
Varian Unity 300 MHz (D)
No. de Registro 0175
20-01-09
Br
N
H
5j
3.803
3.782
3.760
3.739
3.84 3.78 ppm
0.944
0.921
4.138
8
7
6
5
4
3
2
1
ppm
0.64
0.70
0.71
0.70
0.64
0.78
0.74
4.38
1.98

U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc515F1516
Disolvente:CDCl3
Experimento 13C
Varian Unity 75 MHz (D)
No. de Registro 0175
20-01-09



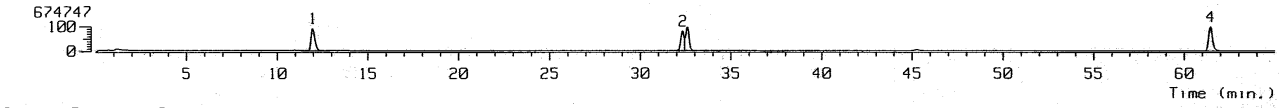

5j

[ TIC ]
Data : Dr-Cabrera-Armando-924 Date : 15-Jan-120 14:48
Sample: 88 G reacc 515 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650



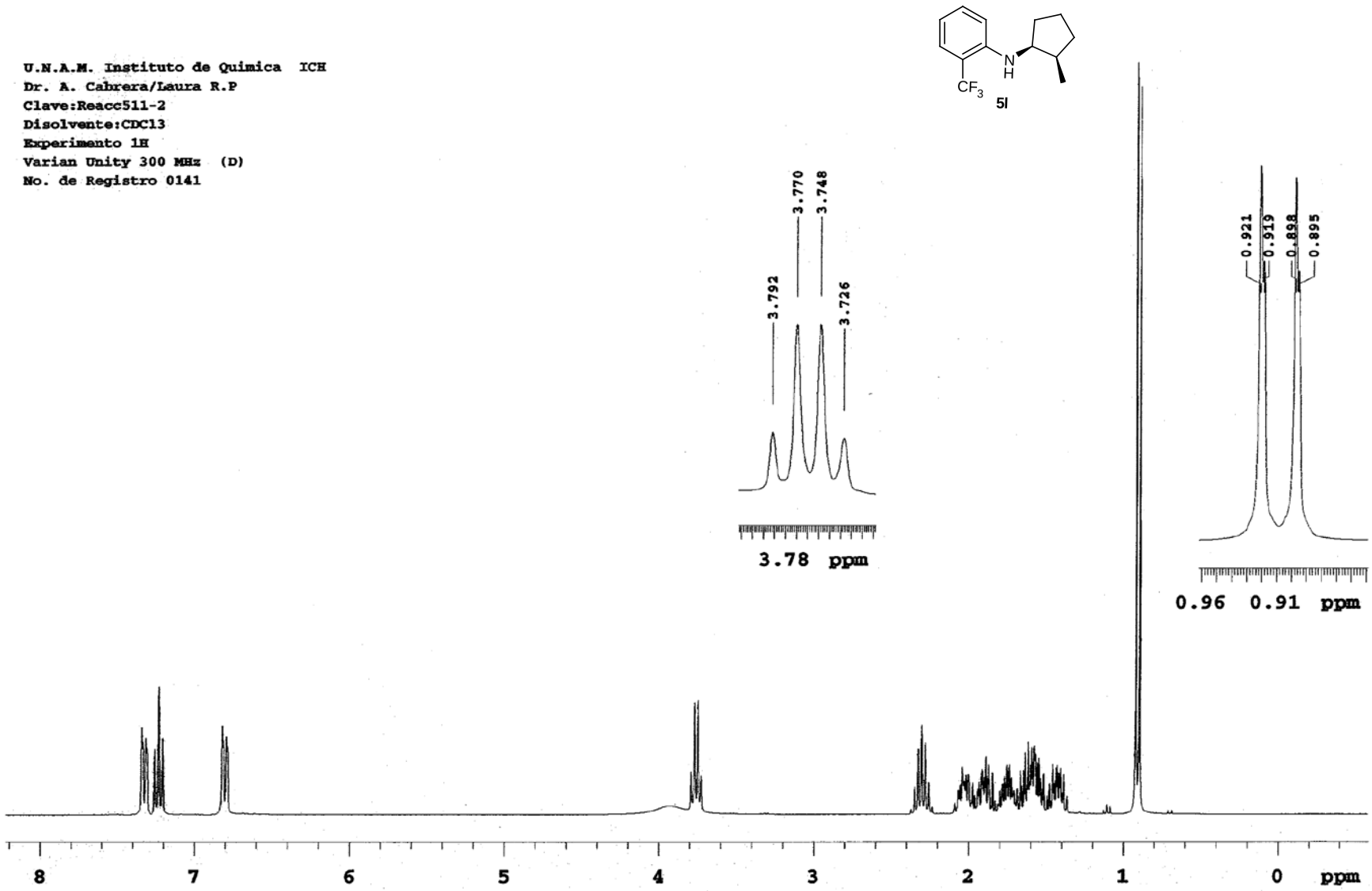

| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 32.32 | 631.92 | 24.50 | 51.16 | 29.19 | 11.60 | BV |
| 2 | 32.59 | 902.94 | 35.01 | 61.51 | 35.09 | 13.78 | VB |
| 3 | 61.44 | 1044.29 | 40.49 | 62.61 | 35.72 | 15.66 | BB |

[ TIC ]
Data : Dr-Cabrera-Armando-924 Date : 15-Jan-120 14:48
Sample: 88 G reacc 515 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 5 to 650 Output RT Range : 0.00 to 64.99 min

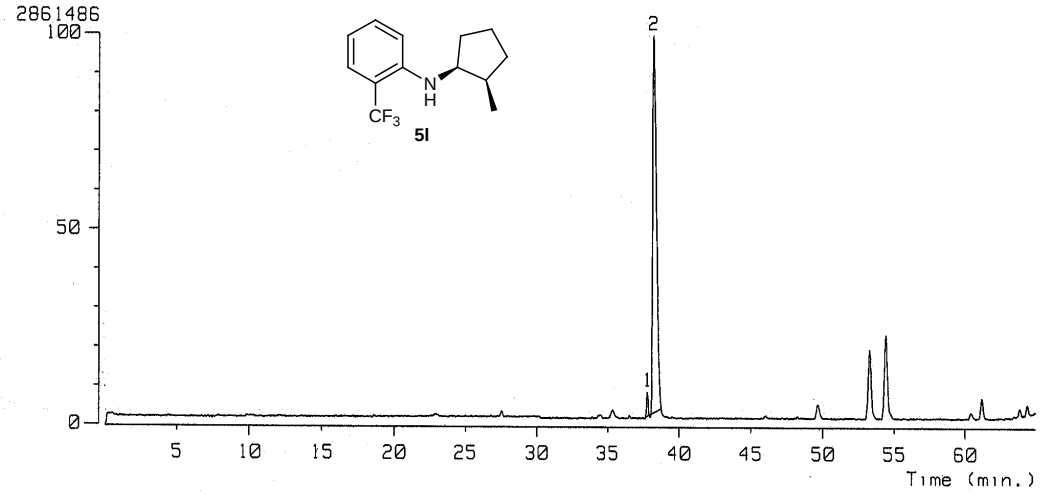



[ Mass Spectrum ]
RT : 32.32 min Scan# : 2422-2356-2846 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 7.14


[ Mass Spectrum ]
RT : 32.59 min Scan# : 2442-2356-2846 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 7.86

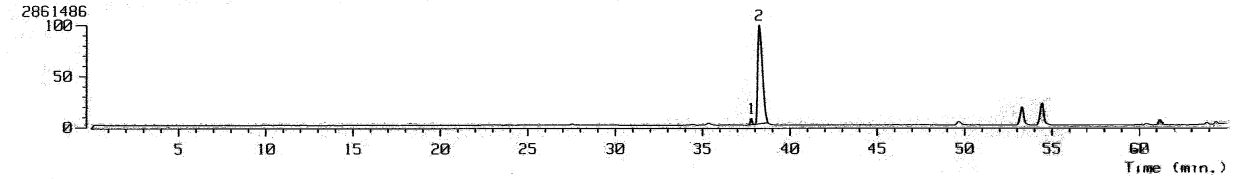

U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R.P
Clave:Reacc511-2
Disolvente:CDCl3
Experimento 1H
Varian Unity 300 MHz (D)
No. de Registro 0141
5l
3.792
3.770
3.748
3.726
3.78 ppm
0.921
0.919
0.898
0.895
0.96 0.91 ppm
8
7
6
5
4
3
2
1
0
ppm

[ TIC ]
Data : Dr-Cabrera-Armando-916 Date : 09-Jan-108 07:58
Sample: 31 G Reacc 511 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650

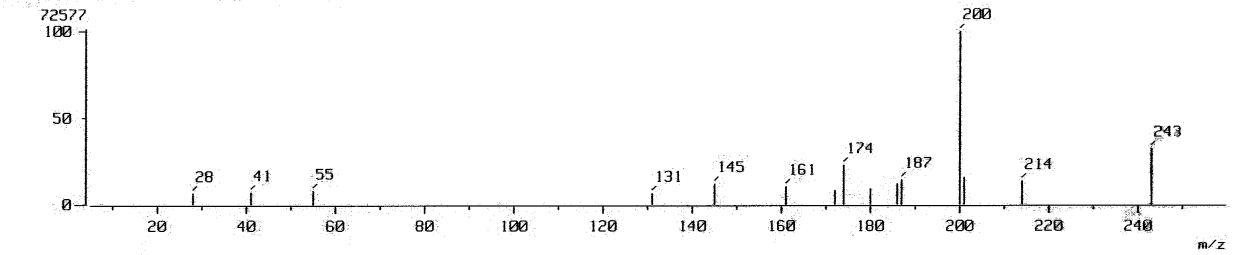



| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 37.77 | 112.96 | 2.27 | 16.64 | 5.94 | 6.37 | BV |
| 2 | 38.21 | 4858.82 | 97.73 | 263.57 | 94.06 | 17.31 | VB |

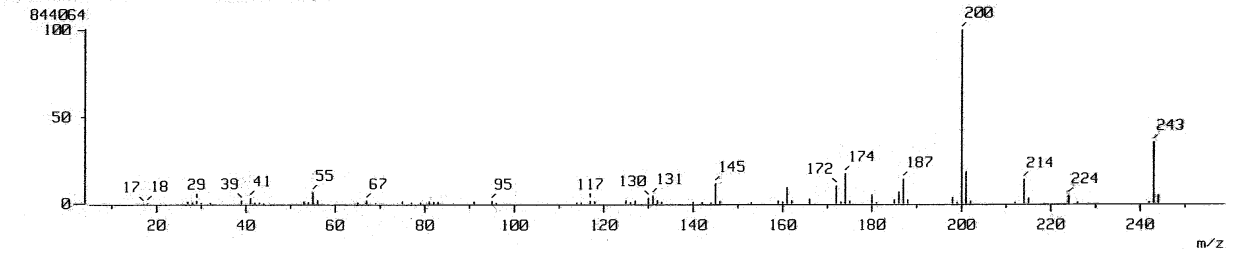
[ TIC ]
Data : Dr-Cabrera-Armando-916 Date : 09-Jan-108 07:58
Sample: 31 G Reacc 511 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 5 to 650 Output RT Range : 0.00 to 64.99 min



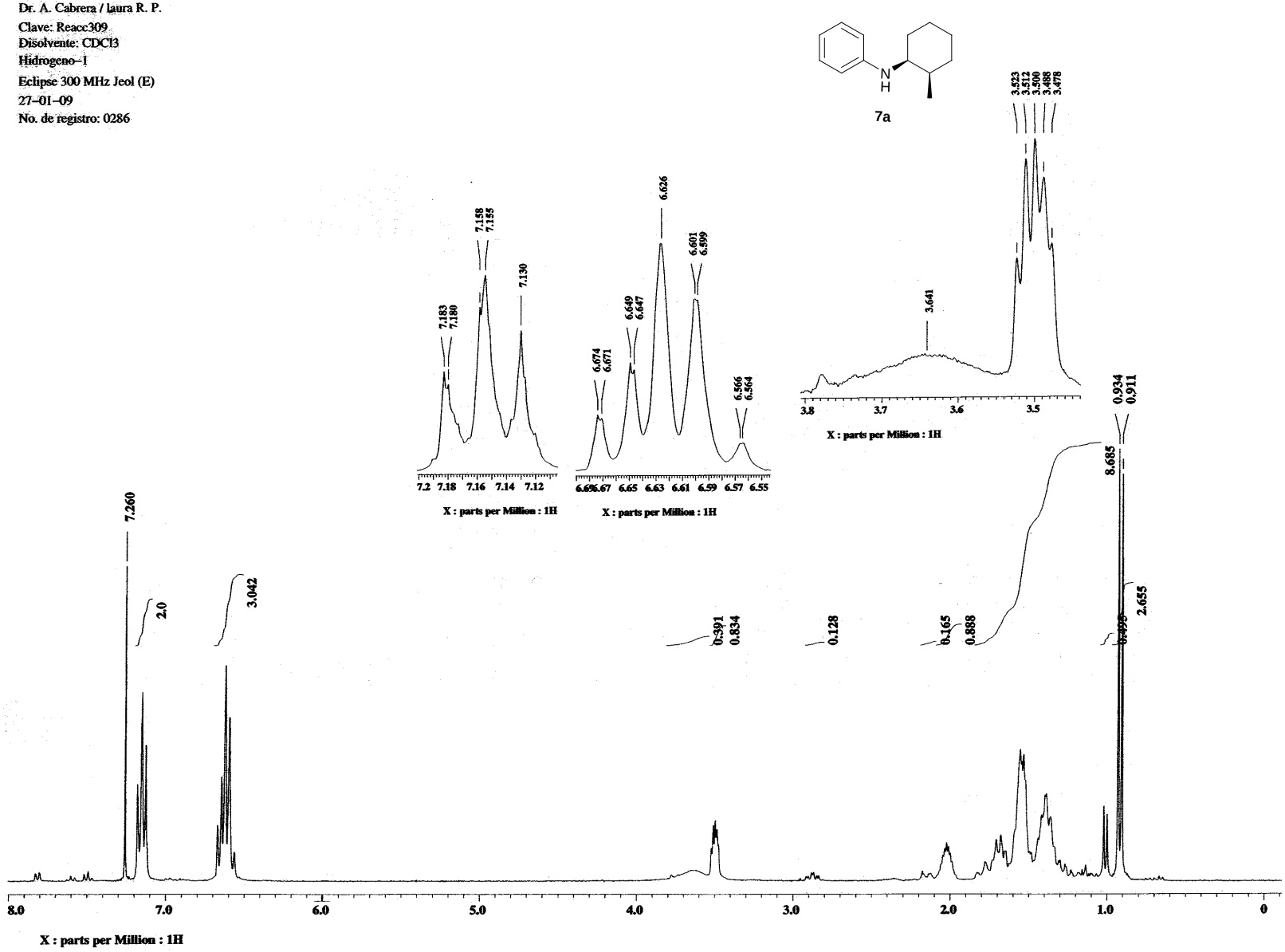

[ Mass Spectrum ]
RT : 37.77 min Scan# : 2830-2821-2903 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 6.82




[ Mass Spectrum ]
RT : 38.21 min Scan# : 2863-2821-2903 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 79.91



INSTITUTO DE QUIMICA, UNAM/ EHS
Dr. A. Cabrera / Laura R. P.
Clave: Reacc309
Disolvente: CDCl3
Hidrogeno-1
Eclipse 300 MHz Jeol (E)
27-01-09
No. de registro: 0286



INSTITUTO DE QUIMICA, UNAM/ EHS

Dr. A. Cabrera/ Laura R. P.

Clave: Reacc309

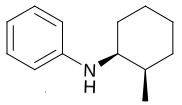
Disolvente: CDCl3

Carbono–13

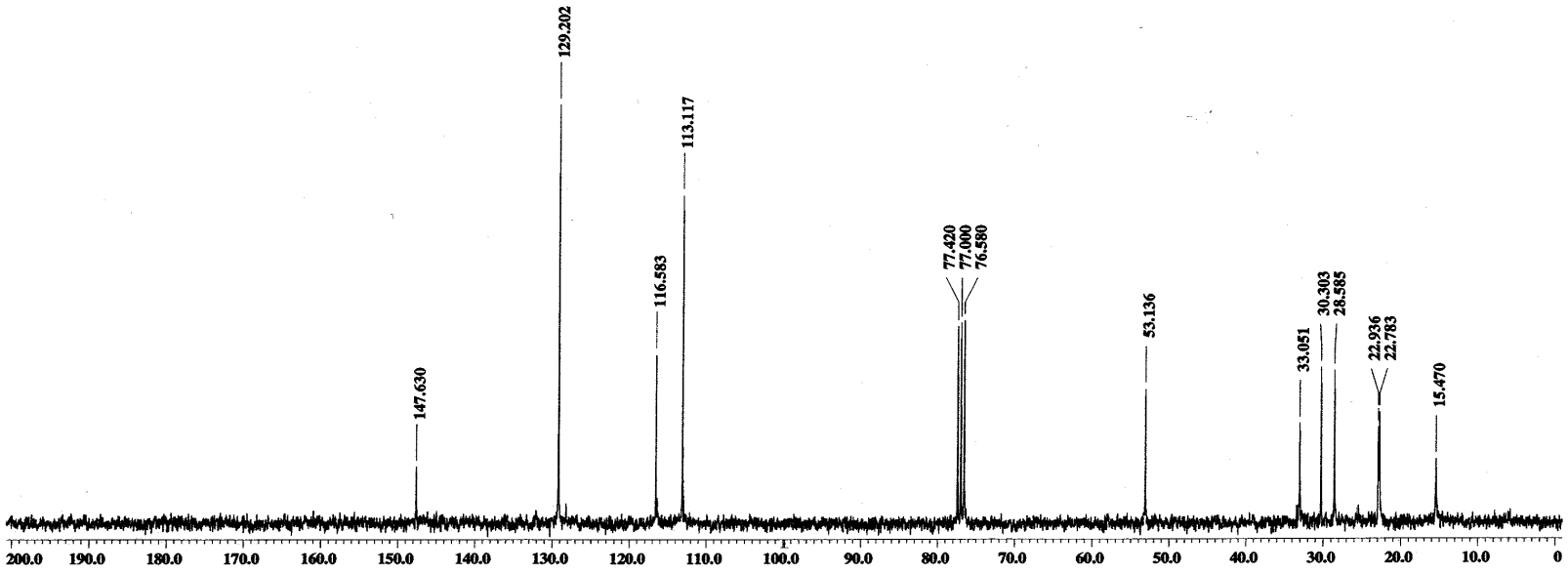
Eclipse 300 MHz Jeol (E)

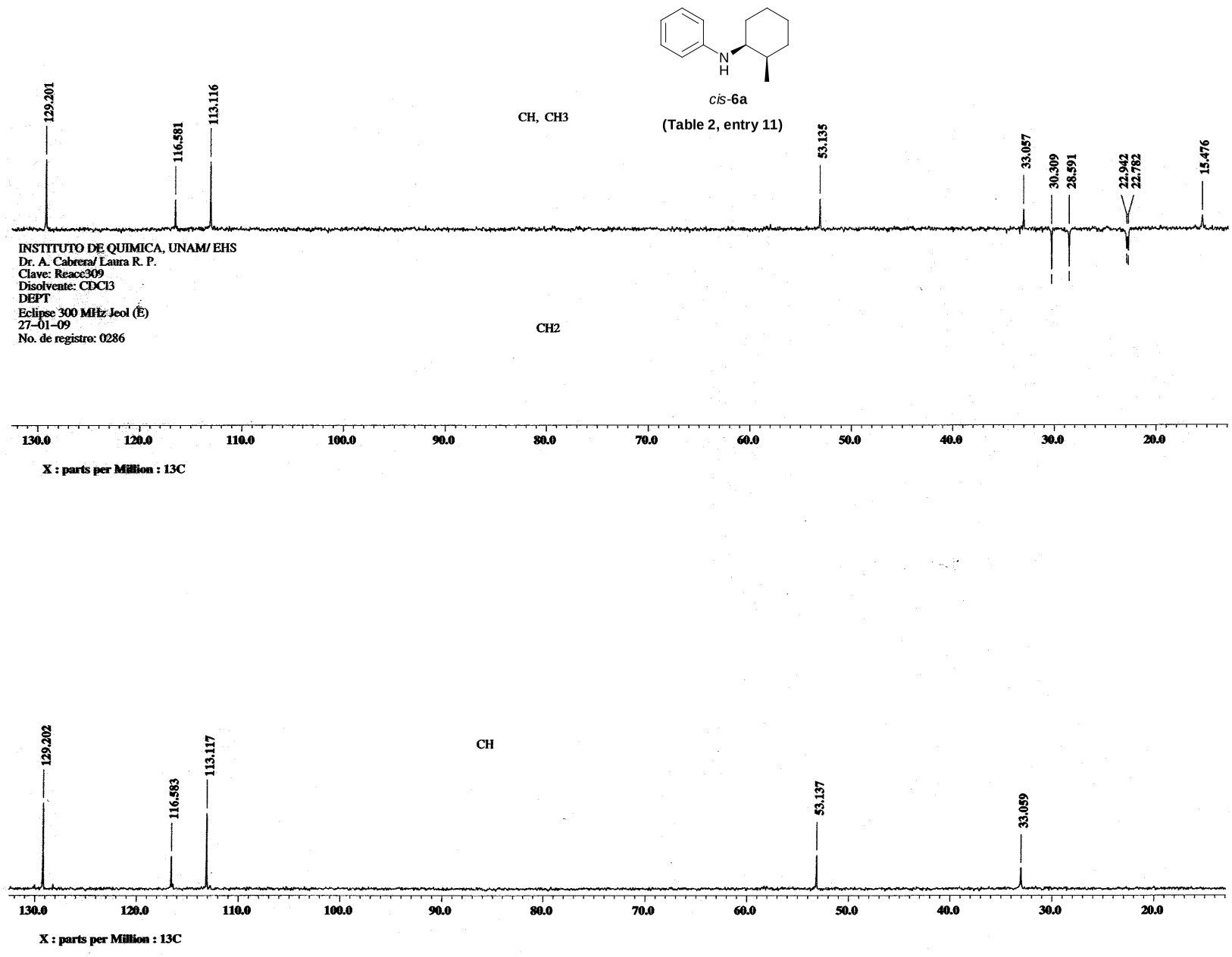
27–01–09

No. de registro: 0286

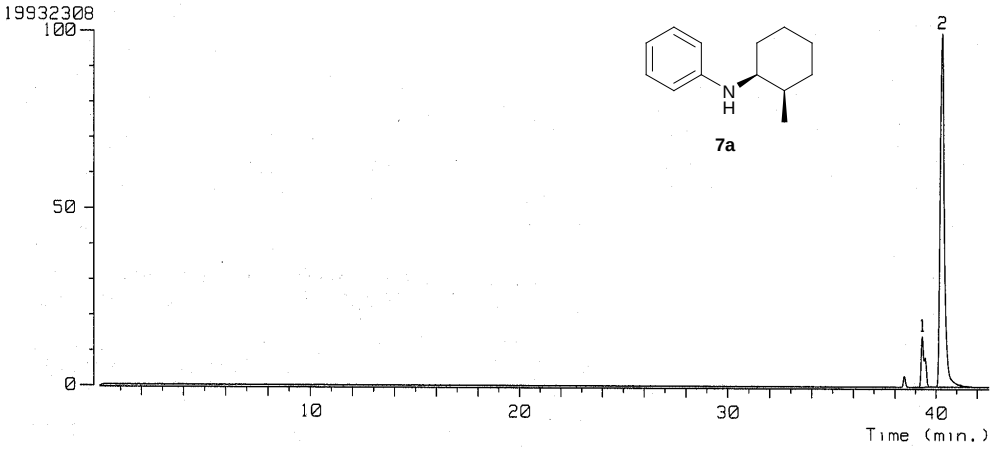



7a



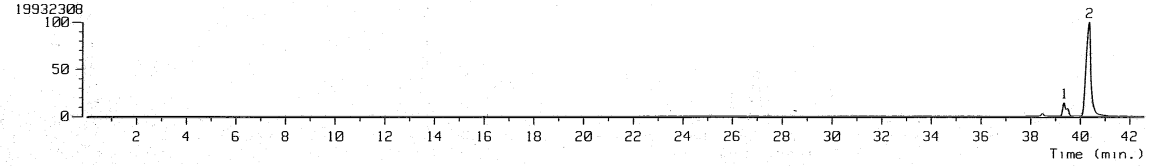

cis-6a
(Table 2, entry 11)
CH, CH3
CH2
129.201
116.581
113.116
53.135
33.057
30.309
28.591
22.942
22.782
15.476
INSTITUTO DE QUIMICA, UNAM/ EHS
Dr. A. Cabrera/ Laura R. P.
Clave: Reacc309
Disolvente: CDCl3
DEPT
Eclipse 300 MHz Jeol (E)
27-01-09
No. de registro: 0286
130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0
X : parts per Million : 13C
CH
129.202
116.583
113.117
53.137
33.059
130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0
X : parts per Million : 13C

[ TIC ]
Data : Dr-Dr-Cabrera-Armando-841 Date : 23-Oct-107 15:37
Sample: 2118 G reacc 309 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650

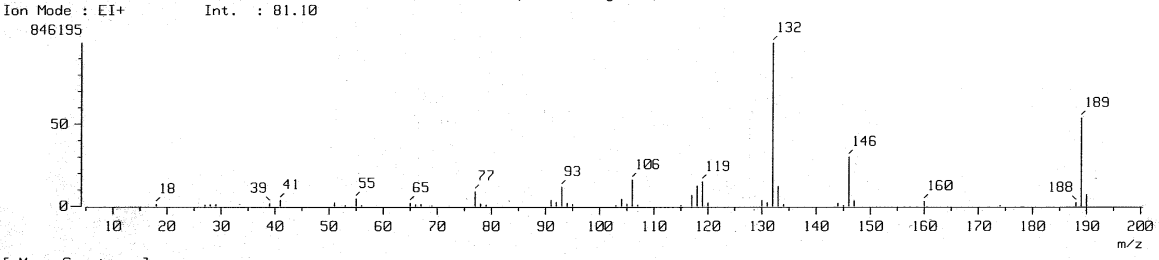



| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 39.34 | 3215.02 | 10.67 | 270.85 | 12.52 | 11.15 | BB |
| 2 | 40.35 | 26924.89 | 89.33 | 1892.75 | 87.48 | 13.36 | BB |

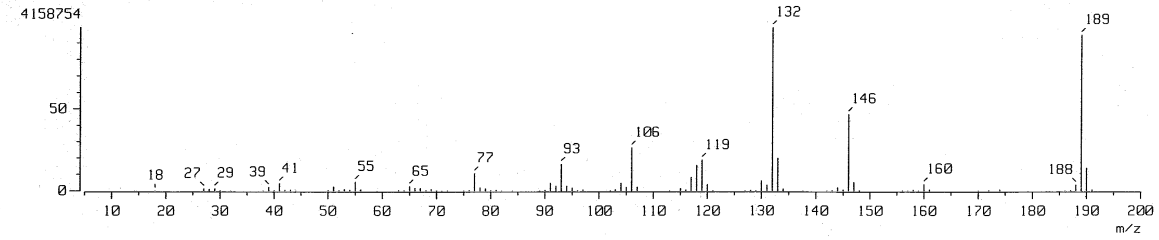
[ TIC ]
Data : Dr-Dr-Cabrera-Armando-841 Date : 23-Oct-107 15:37
Sample: 2118 G reacc 309 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 5 to 650 Output RT Range : 0.00 to 42.58 min

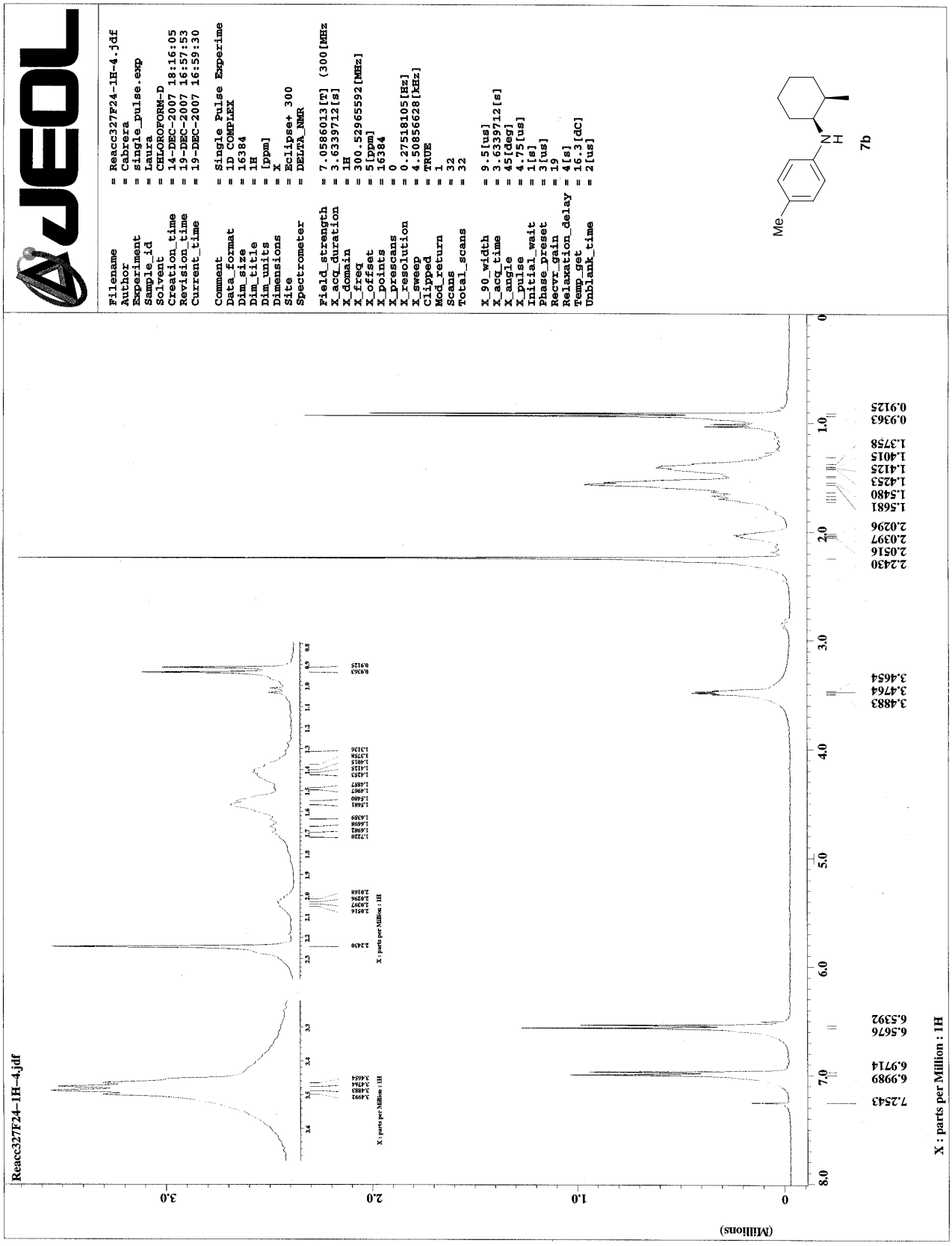


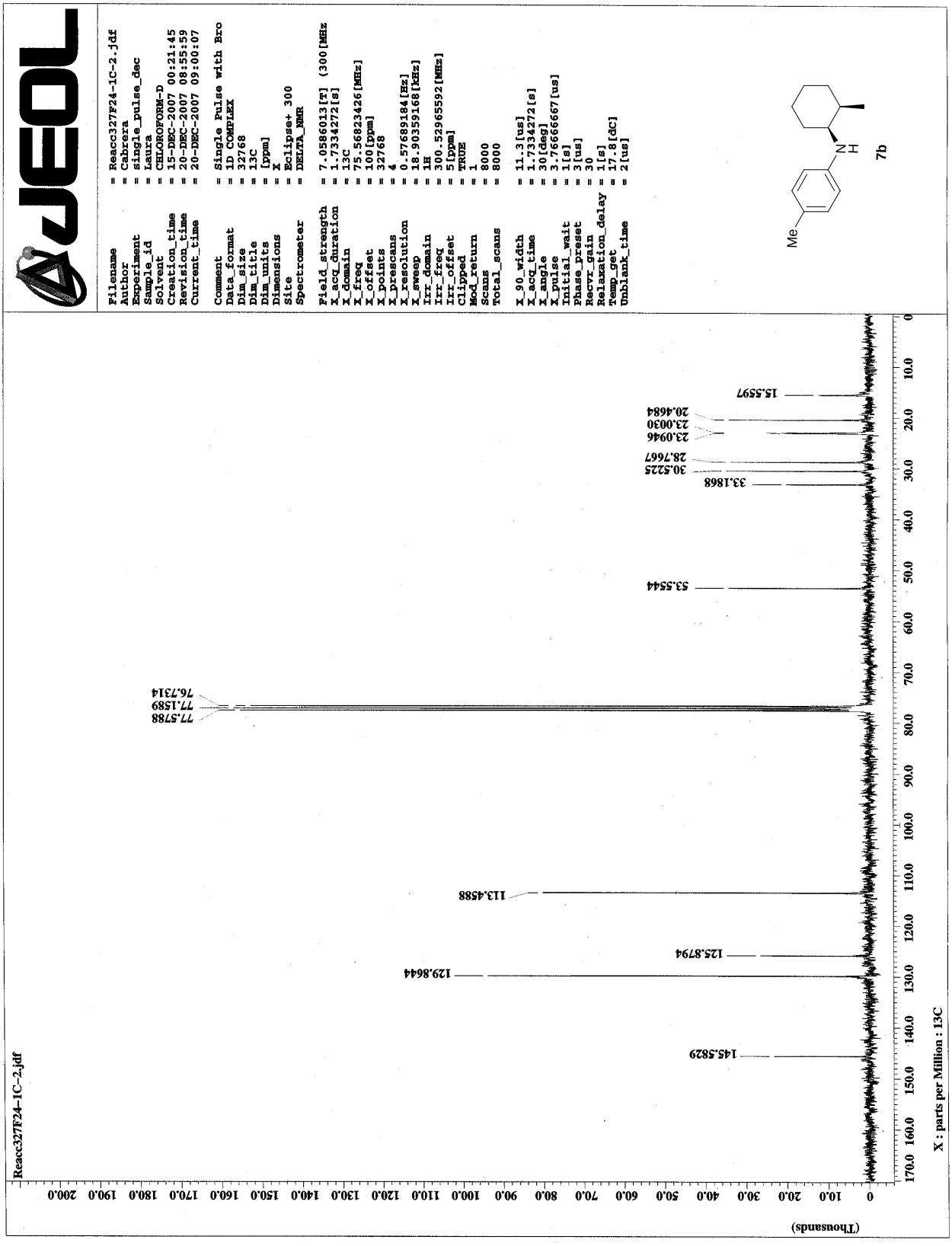

[ Mass Spectrum ]
RT : 39.34 min Scan# : 2951-2938-2980 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 81.10




[ Mass Spectrum ]
RT : 40.35 min Scan# : 3027-2998-3125 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 398.54



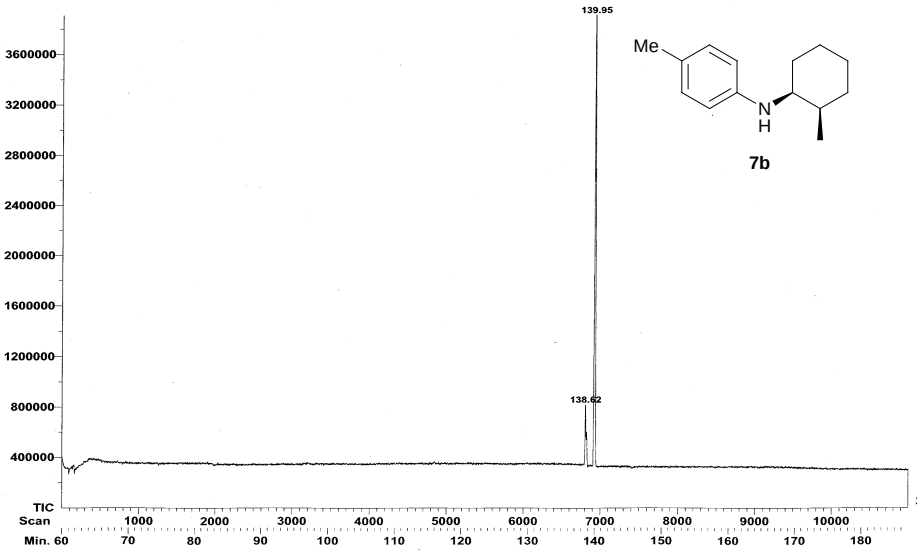

Filename = Reacc327F24-1H-4.jdf
Author = Cabrera
Experiment = single_pulse.exp
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 14-DEC-2007 18:16:05
Revision_time = 19-DEC-2007 16:57:53
Current_time = 19-DEC-2007 16:59:30
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
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 = 4.5085628[kHz]
Clipped = TRUE
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 = 16.3[dC]
Unblank_time = 2[us]
Reacc327F24-1H-4.jdf
(Millions)
X : parts per Million : 1H
7.2543
6.9989
6.9714
6.5676
6.5392
3.4883
3.4764
3.4654
2.2430
2.0516
2.0397
2.0296
1.5681
1.5480
1.4253
1.4125
1.4015
1.3758
0.9363
0.9125
Me
N
H
7b


JEOL
Filename = Reacc327F24-1C-2.jdf
Author = Cabrera
Experiment = single_pulse_dec
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 15-DEC-2007 00:21:45
Revision_time = 20-DEC-2007 08:55:59
Current_time = 20-DEC-2007 09:00:07
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 = TRUE
Mod_return = 1
Scans = 8000
Total_scans = 8000
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 = 17.8[dC]
Unblank_time = 2[us]
Me
N
H
7b
Reacc327F24-1C-2.jdf
(Thousands)
X : parts per Million : 13C
145.5829
129.8644
125.8794
113.4588
77.5788
77.1589
76.7314
53.5544
33.1868
30.5225
28.7667
23.0946
23.0030
20.4684
15.5597

File: 1236-Reac327-2
Sample: Dr-Cabrera
Instrument: JEOL GCmate
Inlet: GC

Date Run: 06-02-2009 (Time Run: 08:50:34)

Ionization mode: EI+







7b

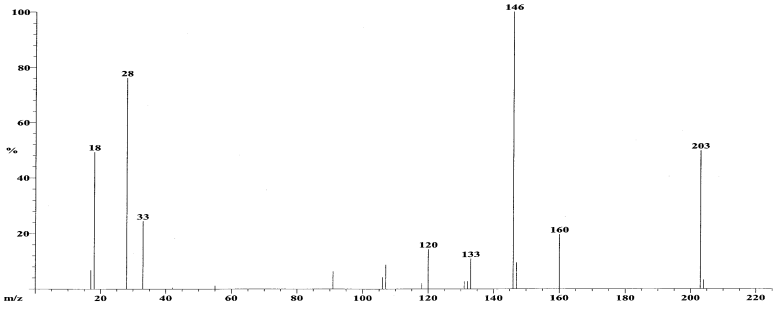
File: 1236-Reac327-2
Sample: Dr-Cabrera
Instrument: JEOL GCmate
Inlet: GC

Date Run: 06-02-2009 (Time Run: 08:50:34)

Ionization mode: EI+

Scan: 6805 R.T.: 138.62
Base: m/z 146; 5.1%FS TIC: 838064

#Ions: 19



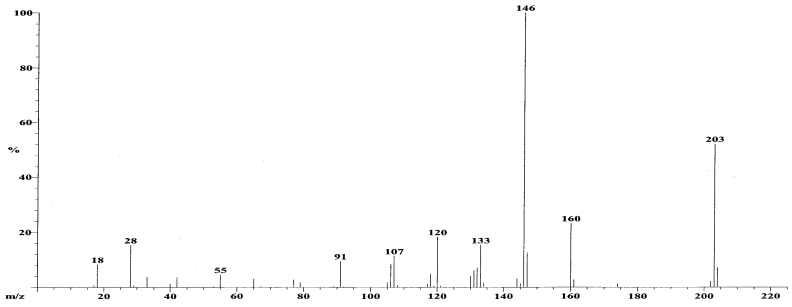

File: 1236-Reac327-2
Sample: Dr-Cabrera
Instrument: JEOL GCmate
Inlet: GC

Date Run: 06-02-2009 (Time Run: 08:50:34)

Ionization mode: EI+

Scan: 6921 R.T.: 139.95
Base: m/z 146; 27%FS TIC: 3925680

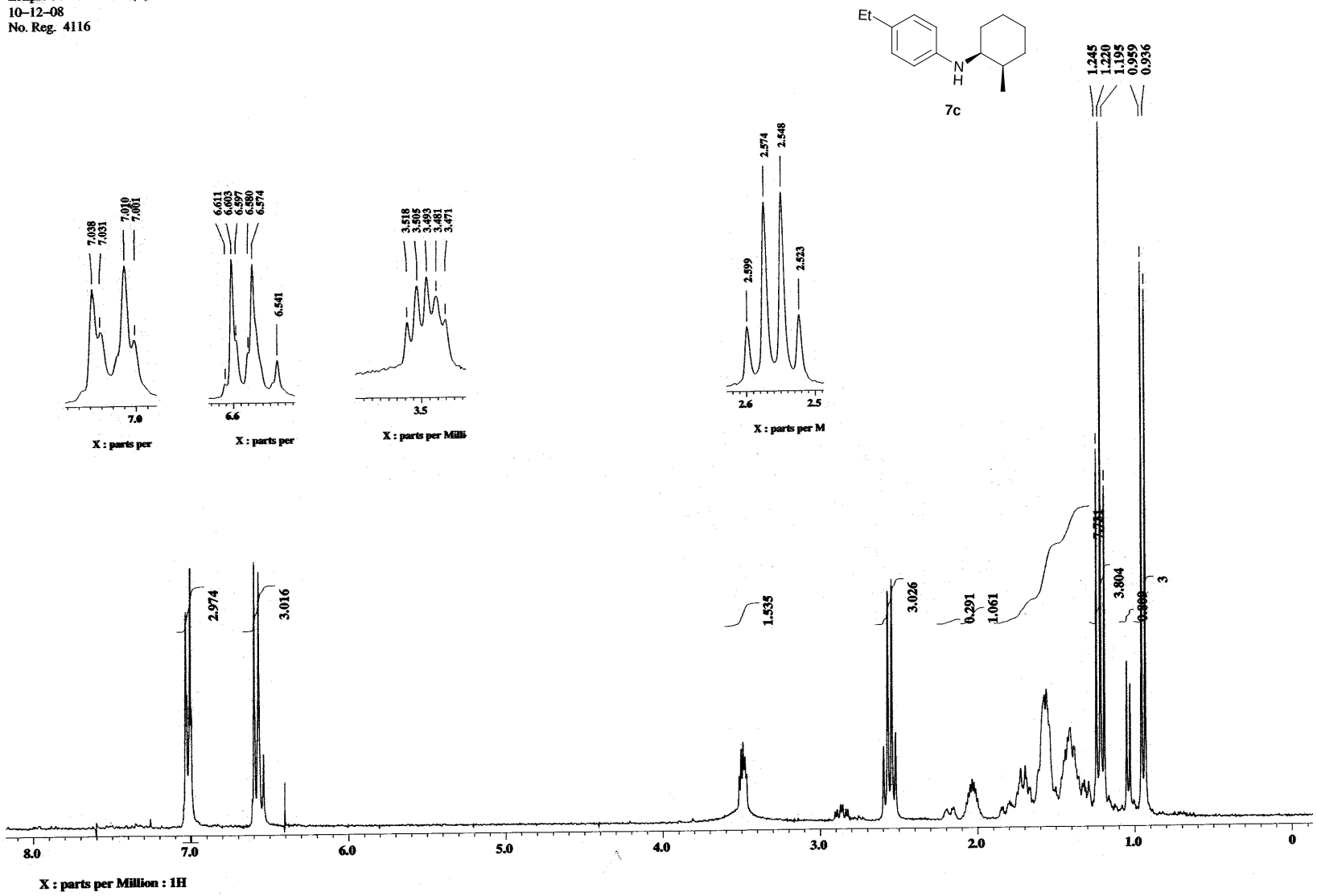
#Ions: 43



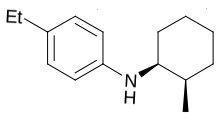

## Peak Table

| Peak Label | RIC | Scan Range | Baseline at | Total Area | Background | Peak Area |
|---|---|---|---|---|---|---|
| 138.62 | TIC | 6793-6829 | 6784-6788 | 14388850 | 8469663 | 5919187 |
| 139.95 | TIC | 6905-6941 | 6895-6899 | 49880378 | 8273759 | 41606619 |

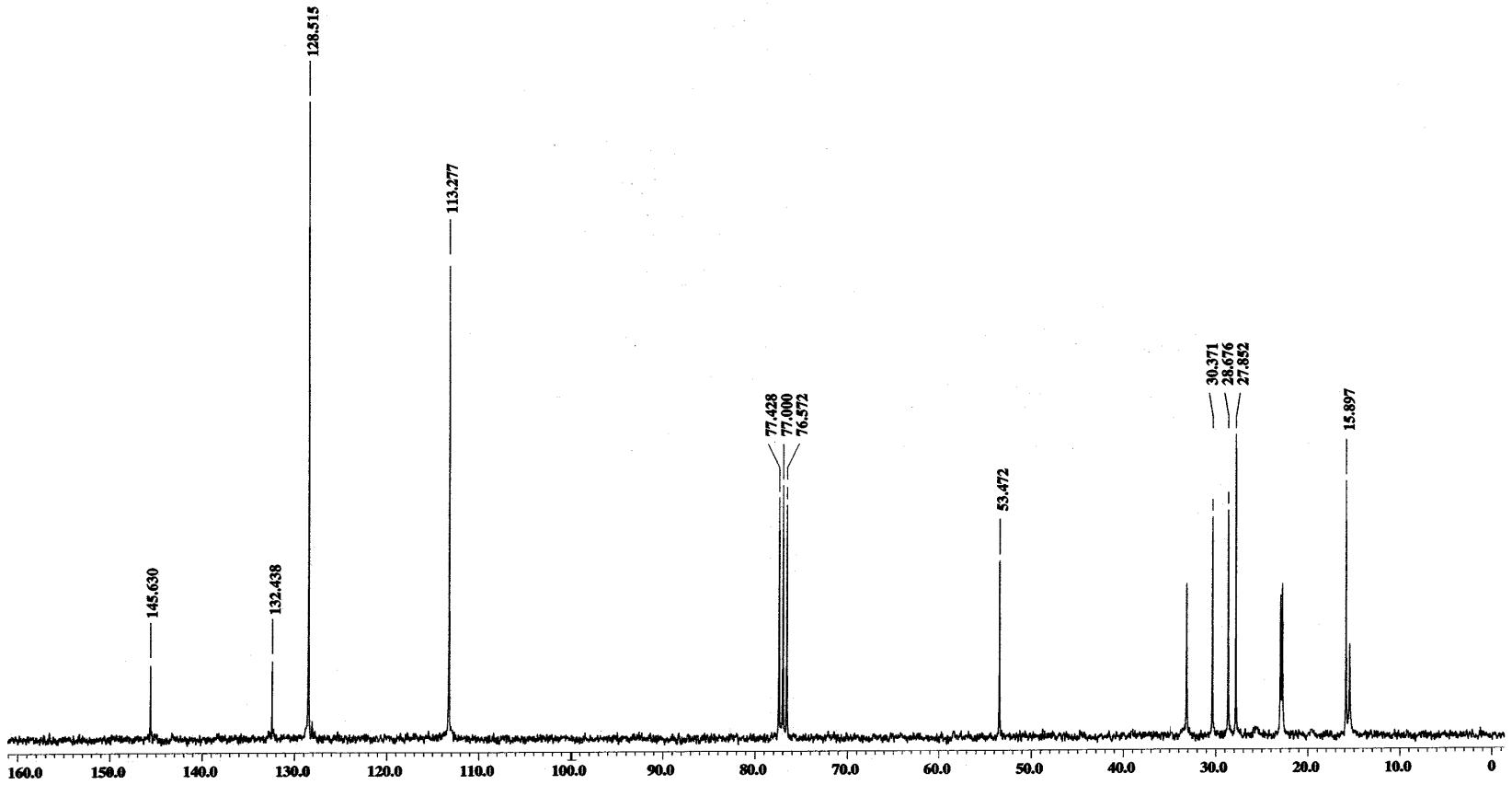
UNAM, INSTITUTO DE QUIMICA, apg
Dr. Armando Cabrera / Laura R. P.
Clave: Reacc. 507–Et
Disolvente: CDCl3
1H
Eclipse 300 MHz Jeol (E)
10–12–08
No. Reg. 4116



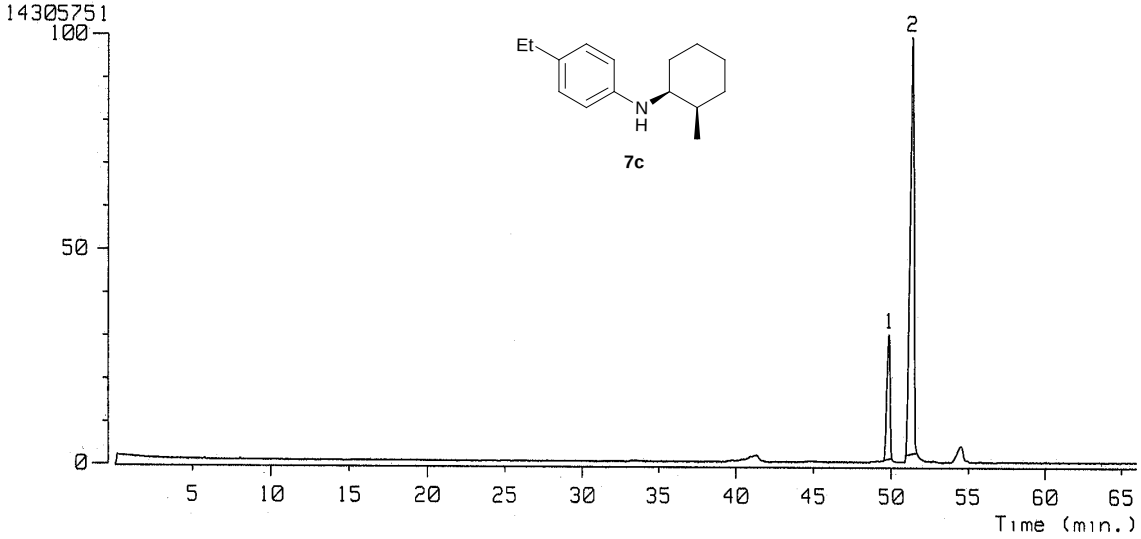
UNAM, INSTITUTO DE QUIMICA, apg
Dr. Armando Cabrera / Laura R. P.
Clave: Reacc. 507-Et
Disolvente: CDCl3
13C
Eclipse 300 MHz Jeol (E)
10-12-08
No. Reg. 4116



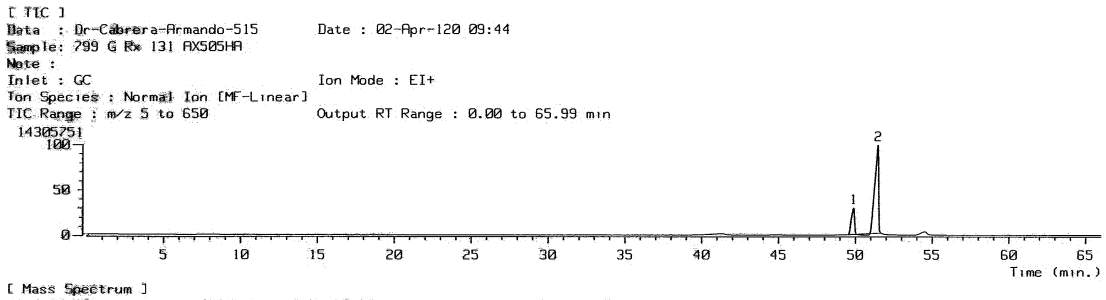

7c

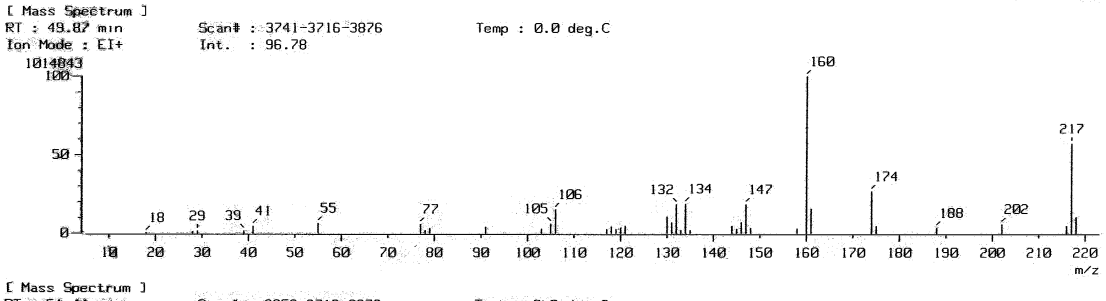


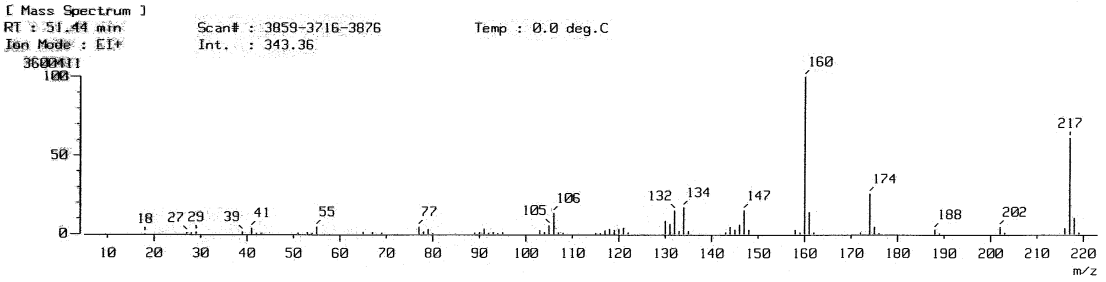
[ TIC ]
Data : Dr-Cabrera-Armando-515 Date : 02-Apr-120 09:44
Sample: 799 G Rx 131 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650

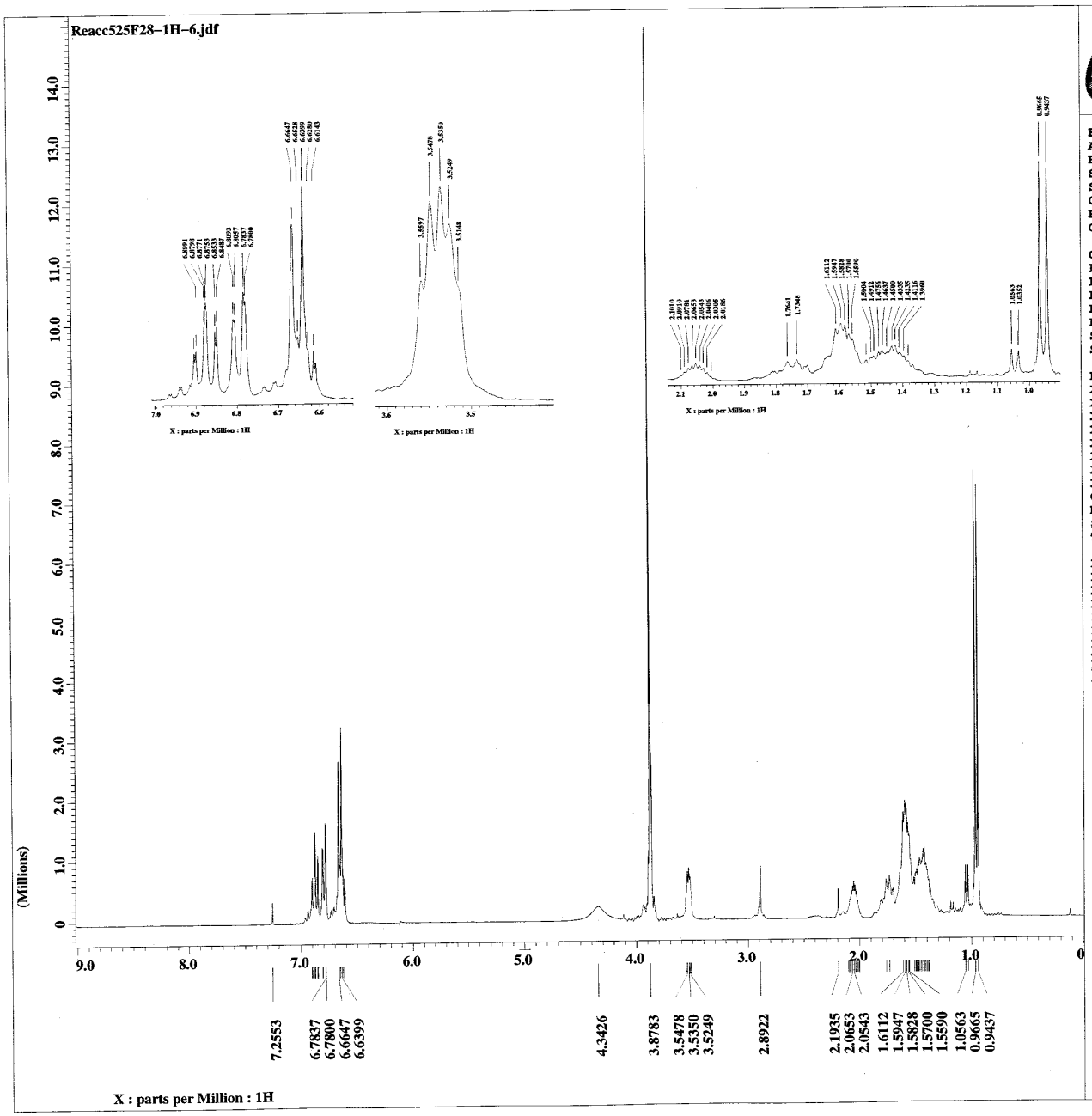



| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 49.87 | 5331.06 | 19.06 | 396.18 | 23.05 | 12.64 | BV |
| 2 | 51.44 | 22645.32 | 80.94 | 1322.93 | 76.95 | 16.07 | VB |



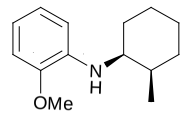



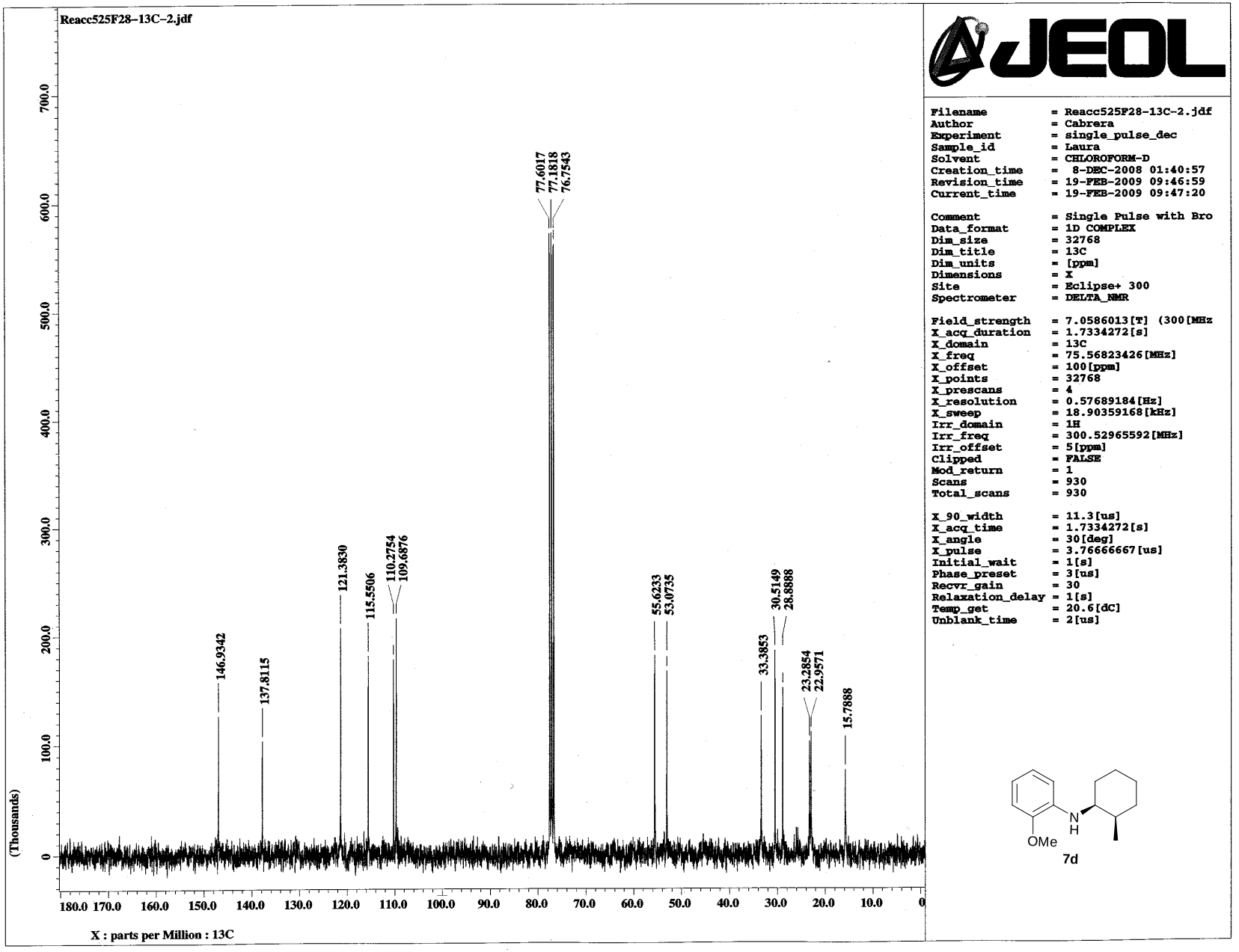


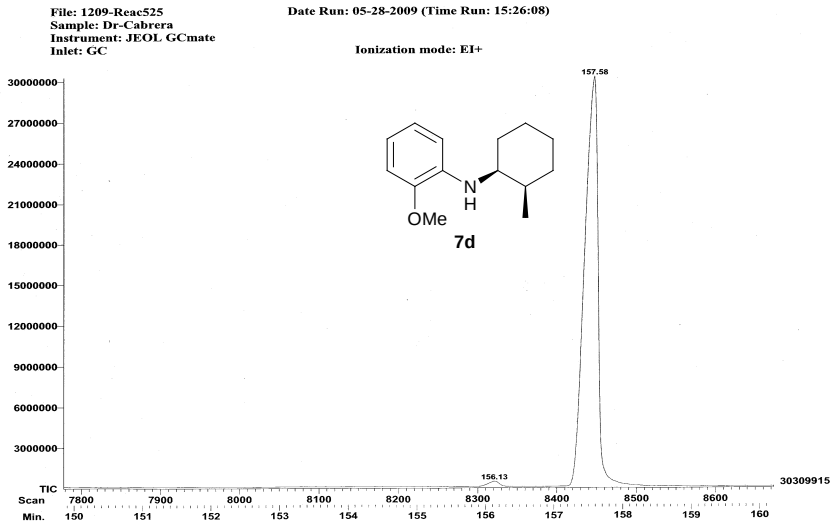

JEOL

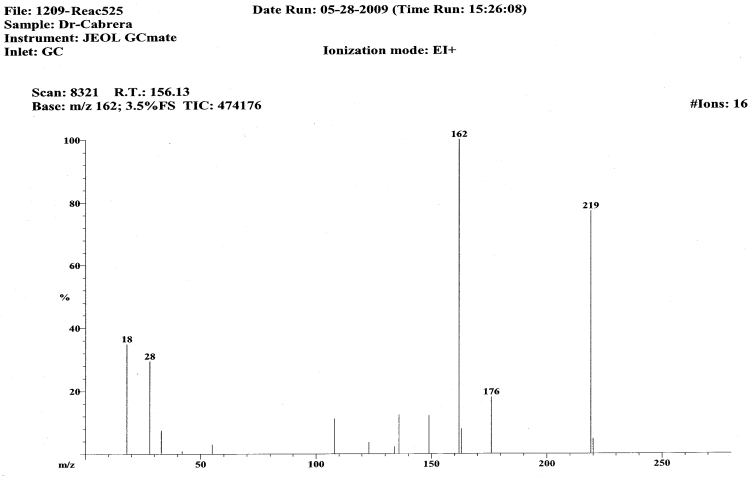
| | |
|---|---|
| Filename | = Reacc525F28-1H-6.jdf |
| Author | = Cabrera |
| Experiment | = single_pulse.exp |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 8-DEC-2008 00:57:24 |
| Revision_time | = 19-FEB-2009 09:19:13 |
| Current_time | = 19-FEB-2009 09:19:30 |
| Comment | = 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 | = 3 |
| Total_scans | = 3 |
| 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] |

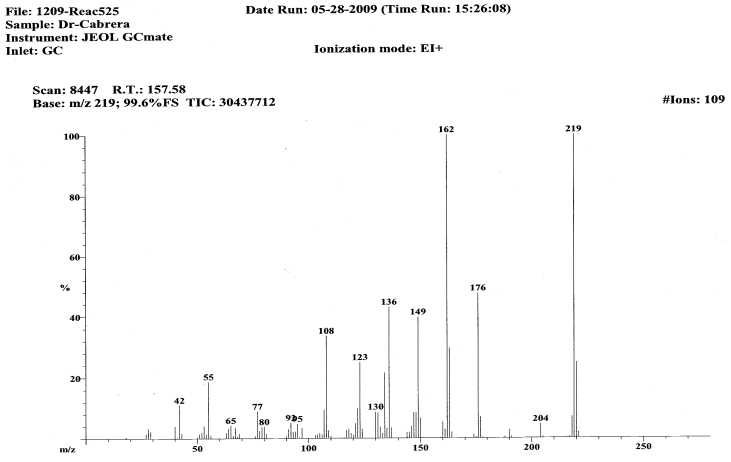



7d


Reacc525F28-13C-2.jdf
(Thousands)
700.0
600.0
500.0
400.0
300.0
200.0
100.0
0
77.6017
77.1818
76.7543
146.9342
137.8115
121.3830
115.5506
110.2754
109.6876
55.6233
53.0735
33.3853
30.5149
28.8888
23.2854
22.9571
15.7888
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0
X : parts per Million : 13C
JEOL
Filename = Reacc525F28-13C-2.jdf
Author = Cabrera
Experiment = single_pulse_dec
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 8-DEC-2008 01:40:57
Revision_time = 19-FEB-2009 09:46:59
Current_time = 19-FEB-2009 09:47:20
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 = 930
Total_scans = 930
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]
N
H
OMe
7d



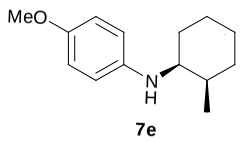




## Peak Table

| Peak Label | RIC | Scan Range | Baseline at | Total Area | Background | Peak Area |
|---|---|---|---|---|---|---|
| 156.13 | TIC | 8301-8337 | 8339-8343 | 6642961 | 2632589 | 4010372 |
| 157.58 | TIC | 8421-8457 | 8469-8473 | 397293413 | 12381400 | 384912013 |

INSTITUTO DE QUIMICA, UNAM / EHS
Dr. A. Cabrera/ Laura R. P.
Clave: reacc341
disolvente: CDCl3
eclipse 300 MHz Jeol (E)
10–03–08
No. de registro: 0957
Hidrogeno–1




7e

INSTITUTO DE QUIMICA, UNAM / EHS

Dr. A. Cabrera/ Laura R. P.

Clave: Reacc341

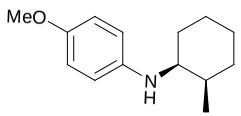
Disolvente: CDCl3

Carbono–13

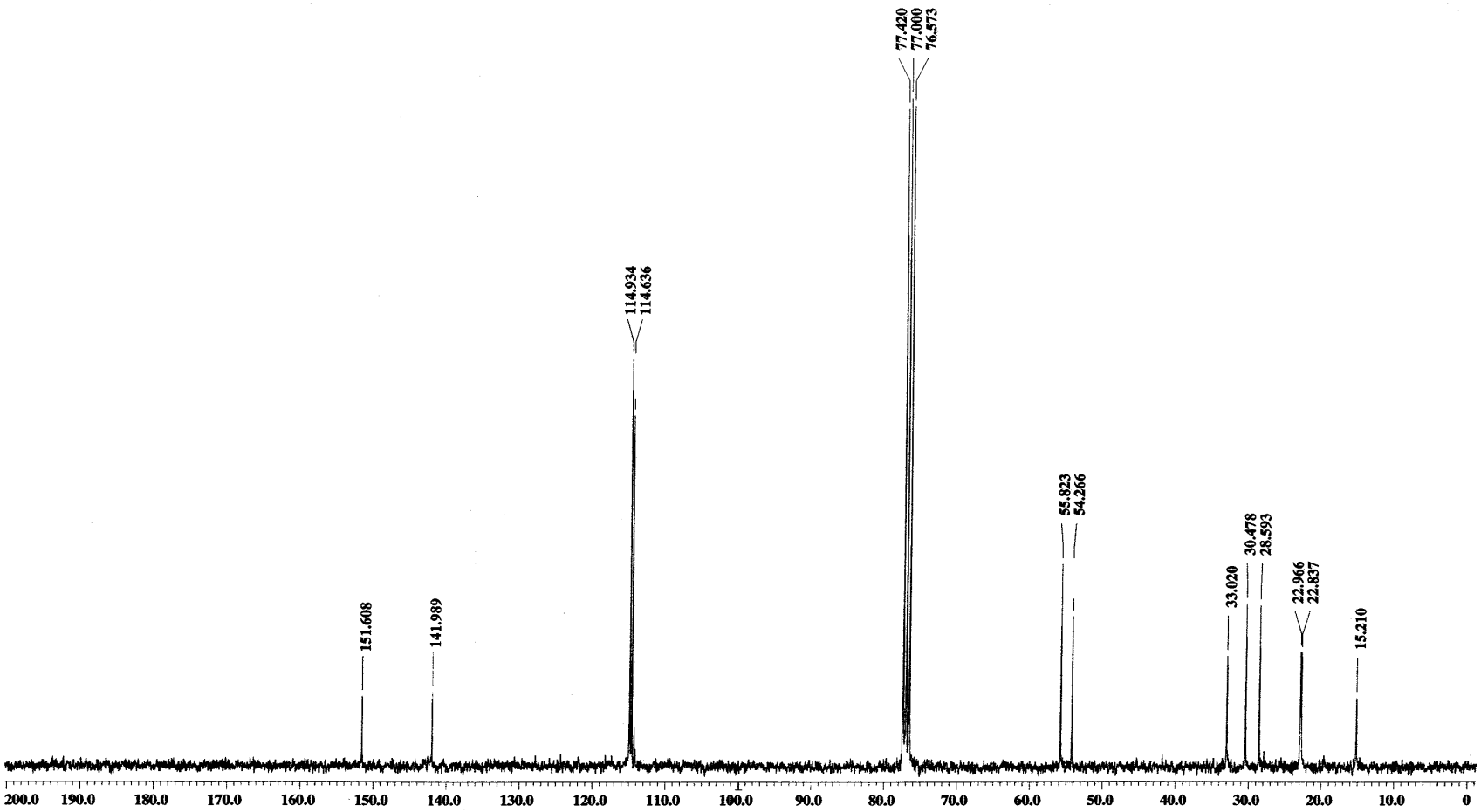
Eclipse 300 MHz Jeol (E)

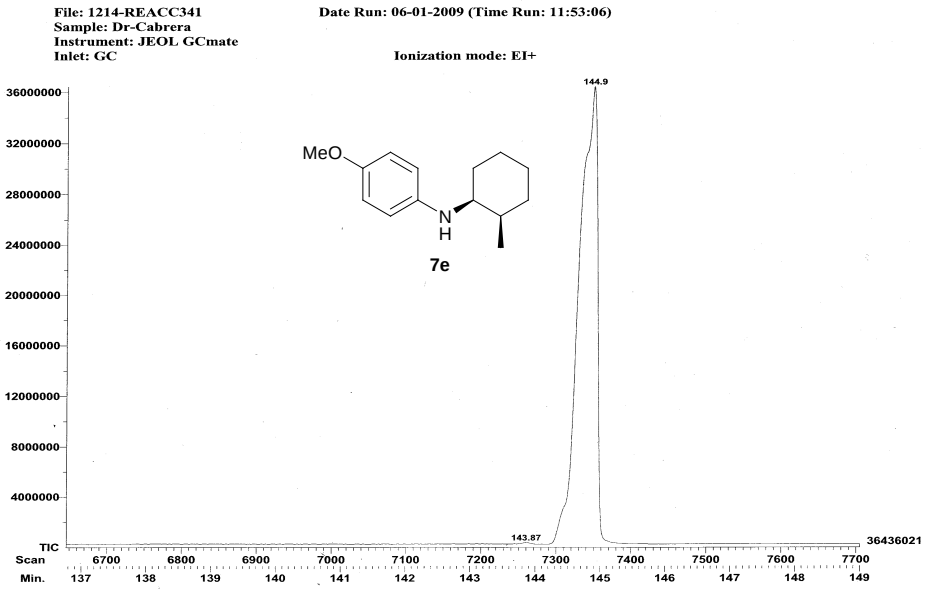
10–03–08

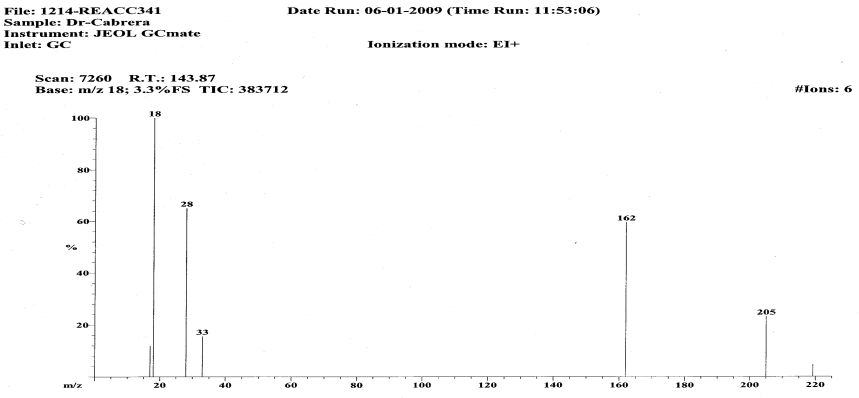
No. de registro: 0957

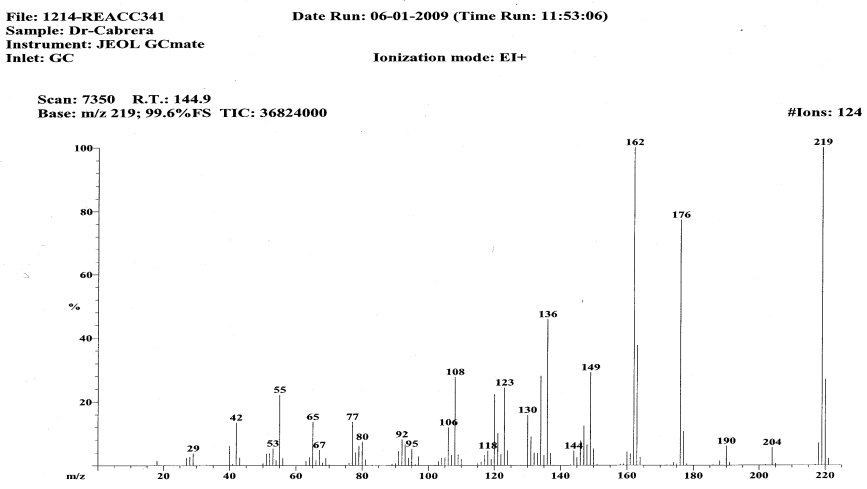



7e



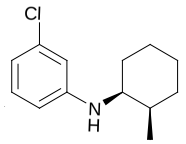


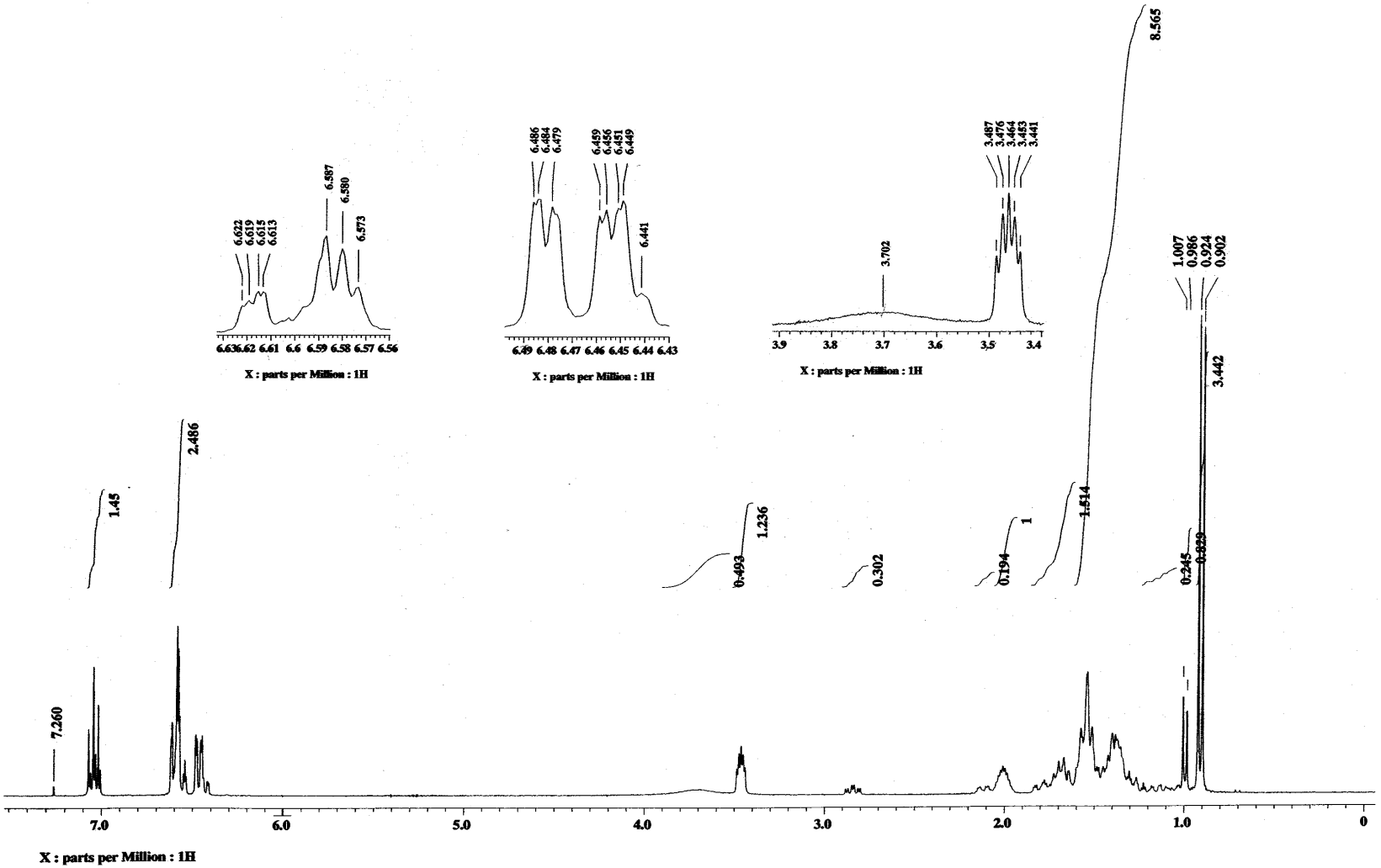




| Peak Label | RIC | Scan Range | Baseline at | Total Area | Background | Peak Area |
|---|---|---|---|---|---|---|
| 143.87 | TIC | 7247-7283 | 7235-7239 | 7461277 | 6447096 | 1014181 |
| 144.9 | TIC | 7324-7360 | 7372-7376 | 637084535 | 10078603 | 627005932 |

INSTITUTO DE QUIMICA, UNAM/ EHS
Dr. A. Cabrera/ Laura R. P.
Clave: Reacc519F22
Disolvente: CDCl3
Hidrogeno–1
Eclipse 300 MHz Jeol (E)
30–01–09
No. de registro: 0343




7f



INSTITUTO DE QUIMICA, UNAM/ EHS

Dr. A. Cabrera/ Laura R. P.

Clave: Reacc519F22

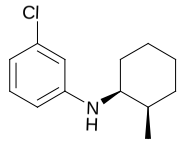
Disolvente: CDCl3

Carbono–13

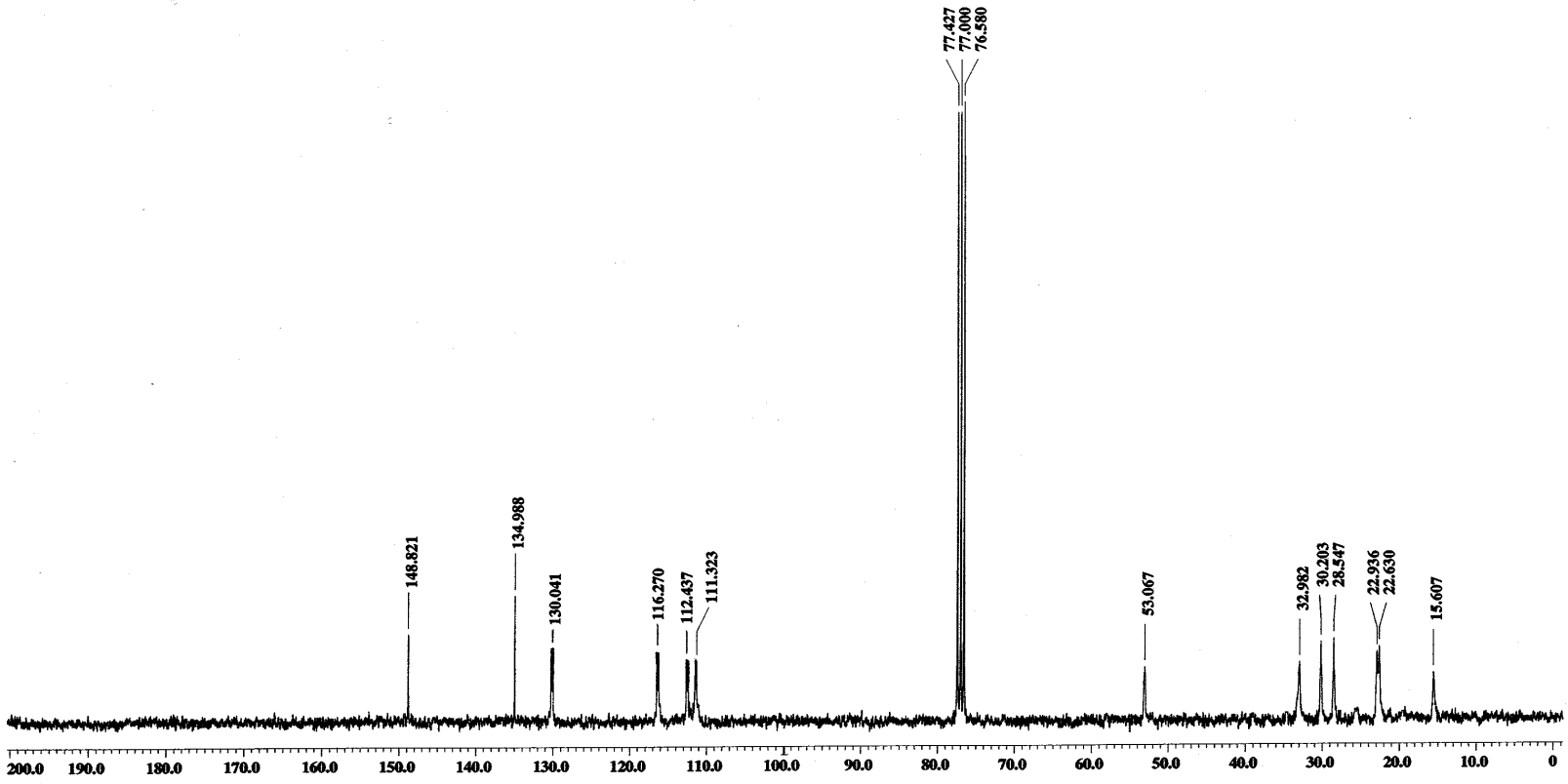
Eclipse 300 MHz Jeol (E)

30–01–09

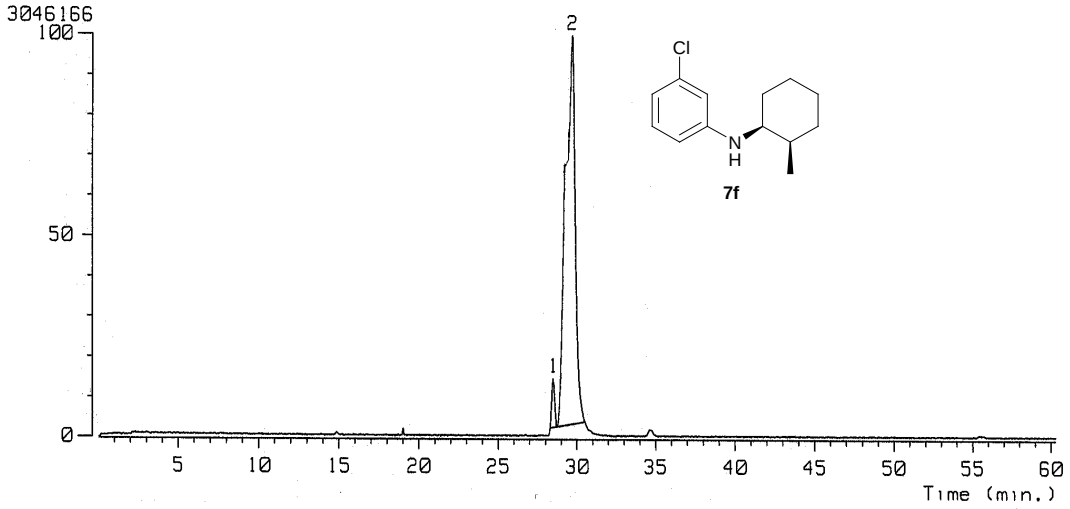
No. de registro: 0343




7f



[ TIC ]
Data : Dr-Cabrera-Armando-944 Date : 29-Jan-120 15:20
Sample: 219 G reacc 519 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion TIC Range : m/z 5 to 650



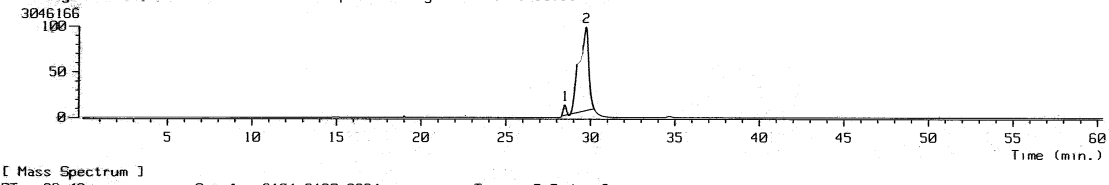




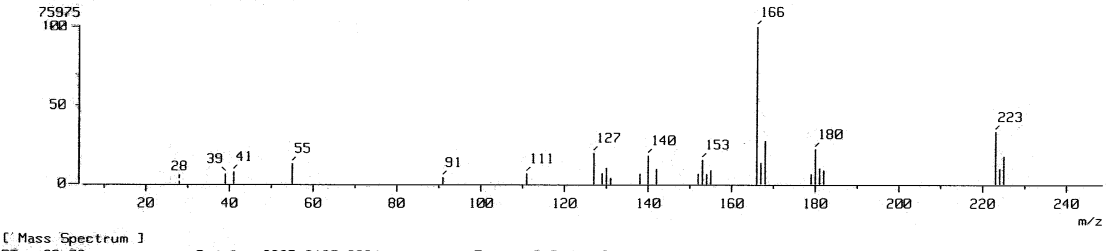
7f

| No. | RT[min] | Area | Area% | Height | Height% | Width[sec] | INTEG |
|---|---|---|---|---|---|---|---|
| 1 | 28.48 | 443.20 | 3.40 | 35.05 | 11.12 | 11.87 | BV |
| 2 | 29.72 | 12604.04 | 96.60 | 280.19 | 88.88 | 42.24 | VB |

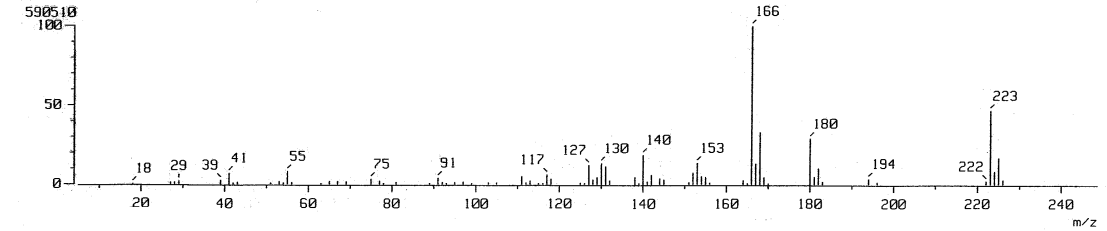
[ TIC ]
Data : Dr-Cabrera-Armando-944 Date : 29-Jan-120 15:20
Sample: 219 G reacc 519 AX505HA
Note :
Inlet : GC Ion Mode : EI+
Ion Species : Normal Ion [MF-Linear]
TIC Range : m/z 5 to 650 Output RT Range : 0.00 to 60.31 min

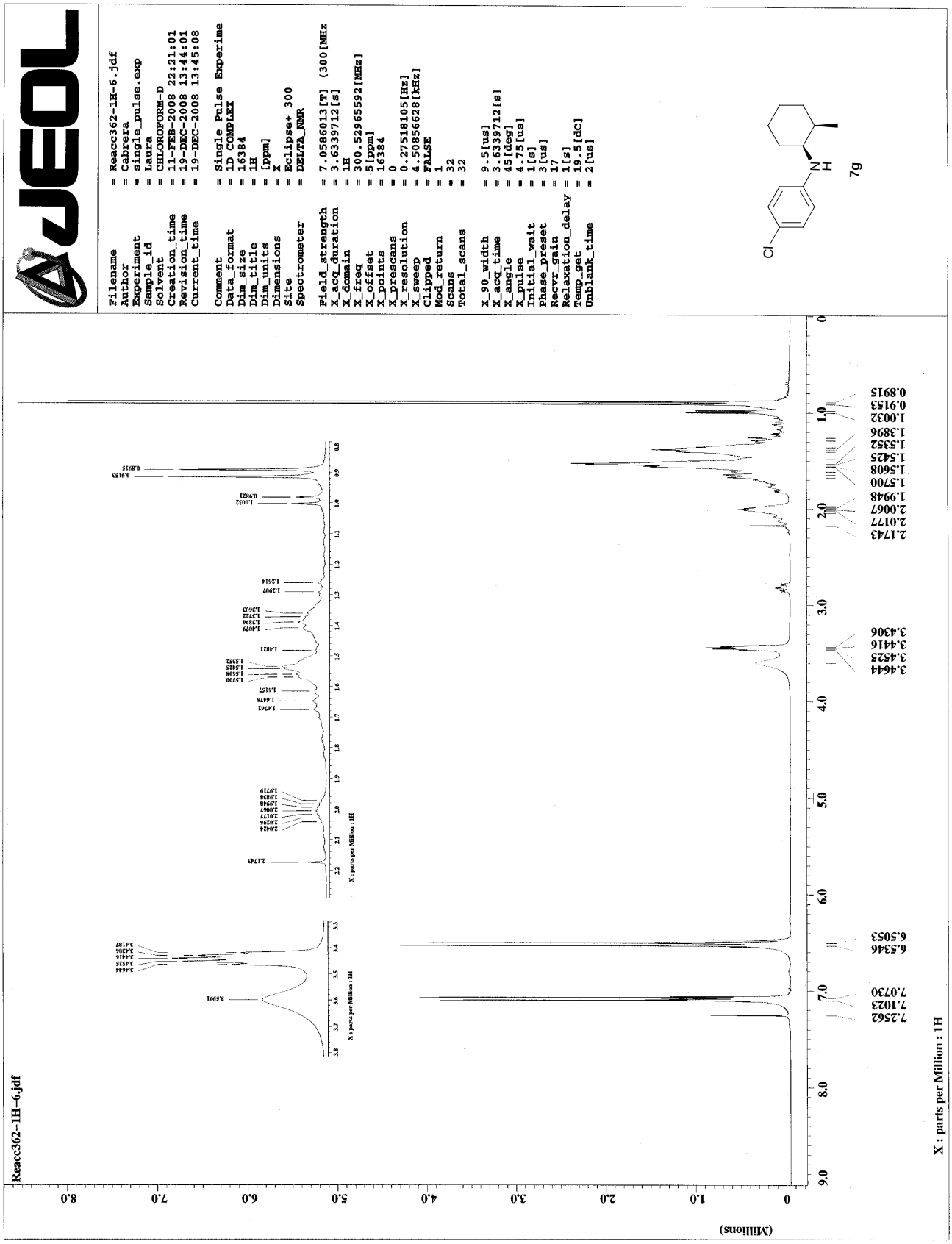


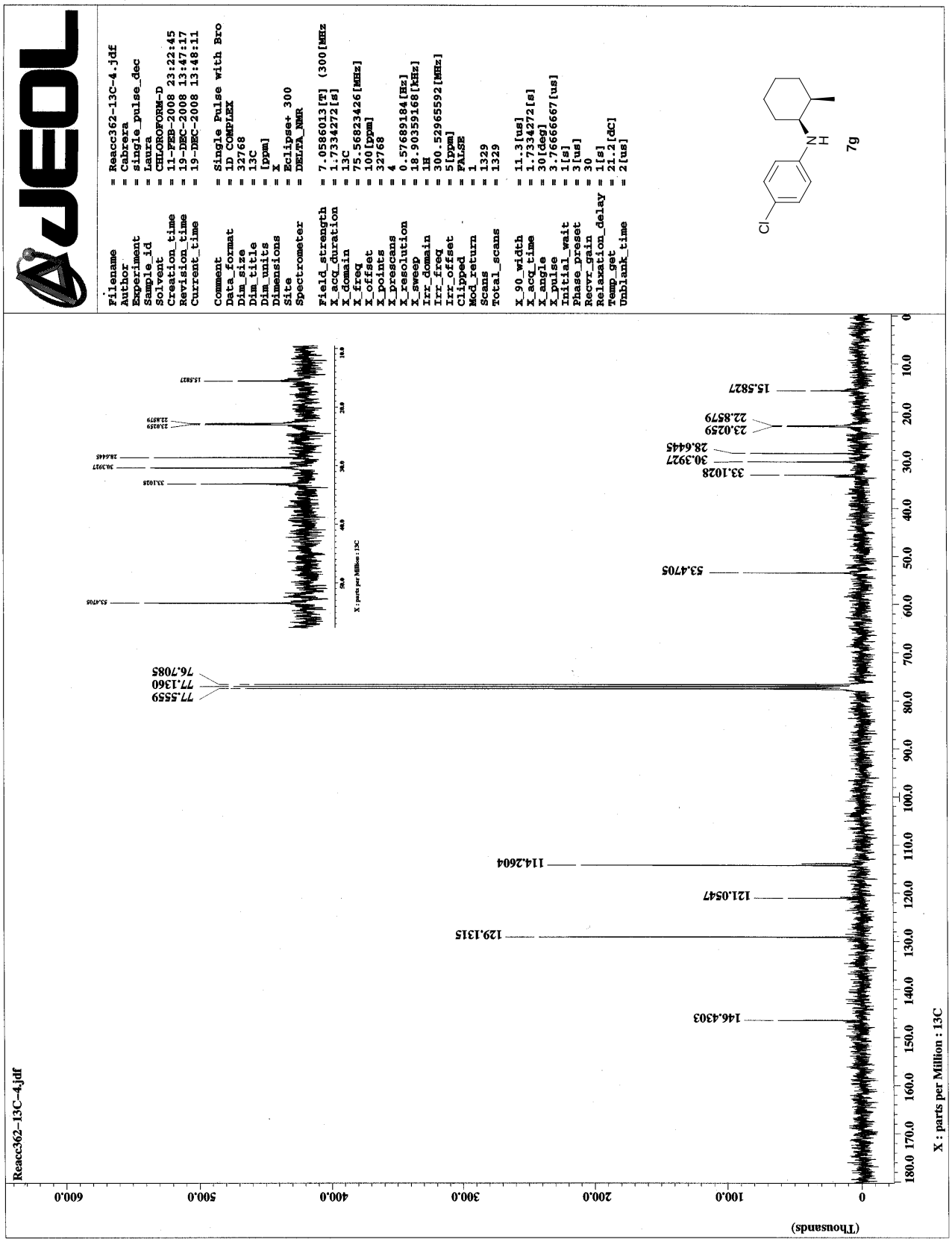

[ Mass Spectrum ]
RT : 28.48 min Scan# : 2134-2120-2264 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 7.19

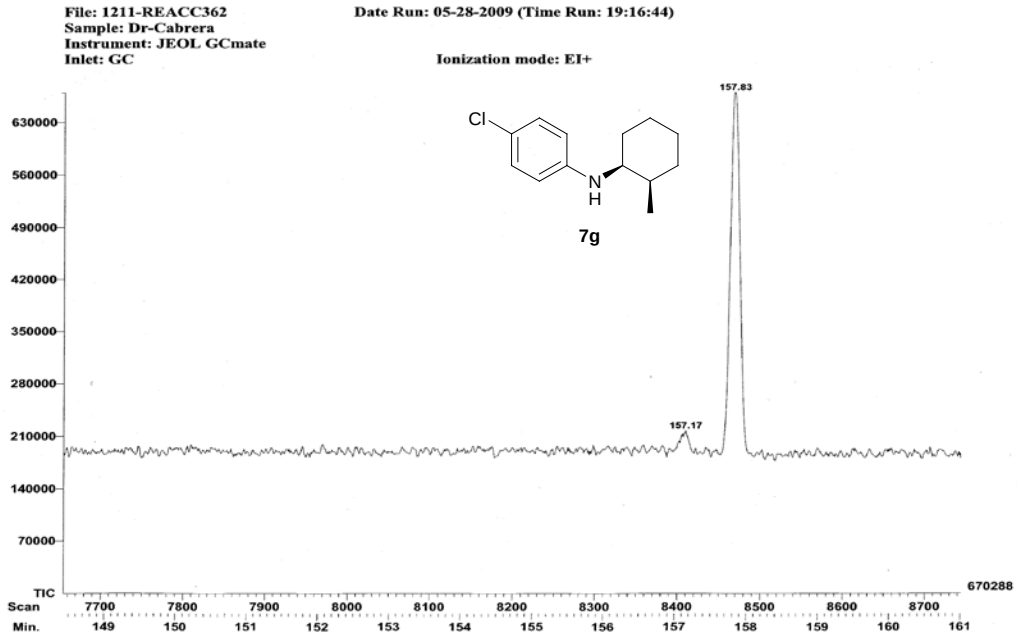


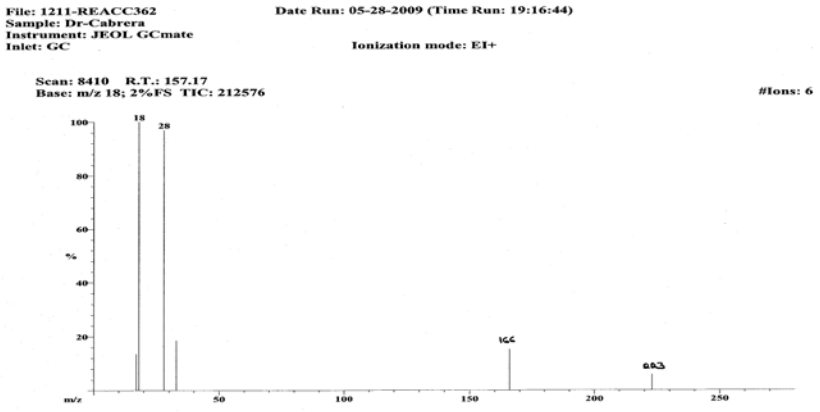

[ Mass Spectrum ]
RT : 29.72 min Scan# : 2227-2120-2264 Temp : 0.0 deg.C
Ion Mode : EI+ Int. : 56.32

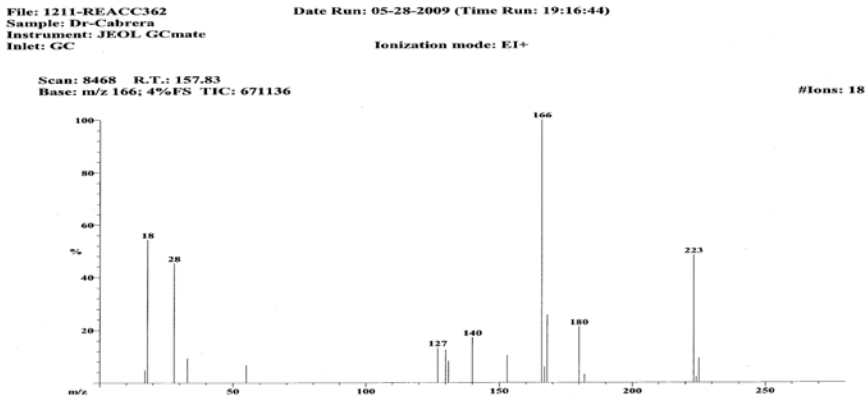



JEOL
Filename = Reacc362-1H-6.jdf
Author = Cabrera
Experiment = single_pulse.exp
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 11-FEB-2008 22:21:01
Revision_time = 19-DEC-2008 13:44:01
Current_time = 19-DEC-2008 13:45:08
Comment = Single Pulse Experime
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = Eclipse+ 300
Spectrometer = DELTA_NMR
Field_strength = 7.0586013[T] (300[MHz
X_acq_duration = 3.63397712[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 = 3.63397712[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 = 19.5[dC]
Unblank_time = 2[us]
Cl
N
H
7g
Reacc362-1H-6.jdf
(Millions)
X : parts per Million : 1H
7.2562
7.1023
7.0730
6.5346
6.5053
3.4644
3.4525
3.4416
3.4306
2.1743
2.0177
2.0067
1.9948
1.5700
1.5608
1.5425
1.5352
1.3896
1.0032
0.9153
0.8915


JEOL
Filename = Reacc362-13C-4.jdf
Author = Cabrera
Experiment = single_pulse_dec
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 11-FEB-2008 23:22:45
Revision_time = 19-DEC-2008 13:47:17
Current_time = 19-DEC-2008 13:48:11
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 = PAUSE
Mod_return = 1
Scans = 1329
Total_scans = 1329
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 = 21.2[dC]
Unblank_time = 2[us]
Cl
N
H
7g
Reacc362-13C-4.jdf
(Thousands)
X : parts per Million : 13C
146.4303
129.1315
121.0547
114.2604
77.5559
77.1360
76.7085
53.4705
33.1028
30.3927
28.6445
23.0259
22.8579
15.5827

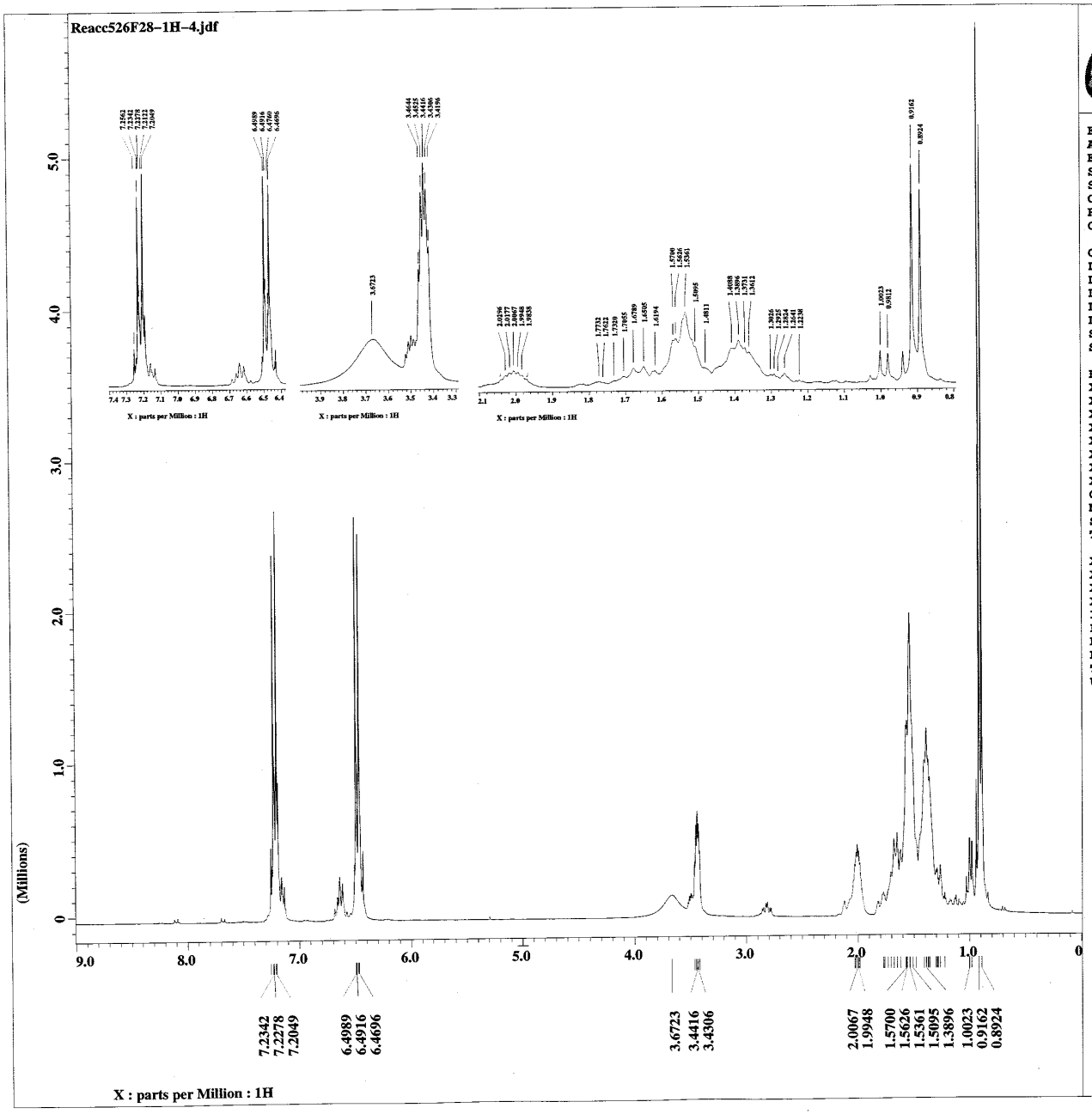


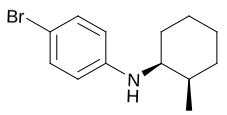




**Peak Table**

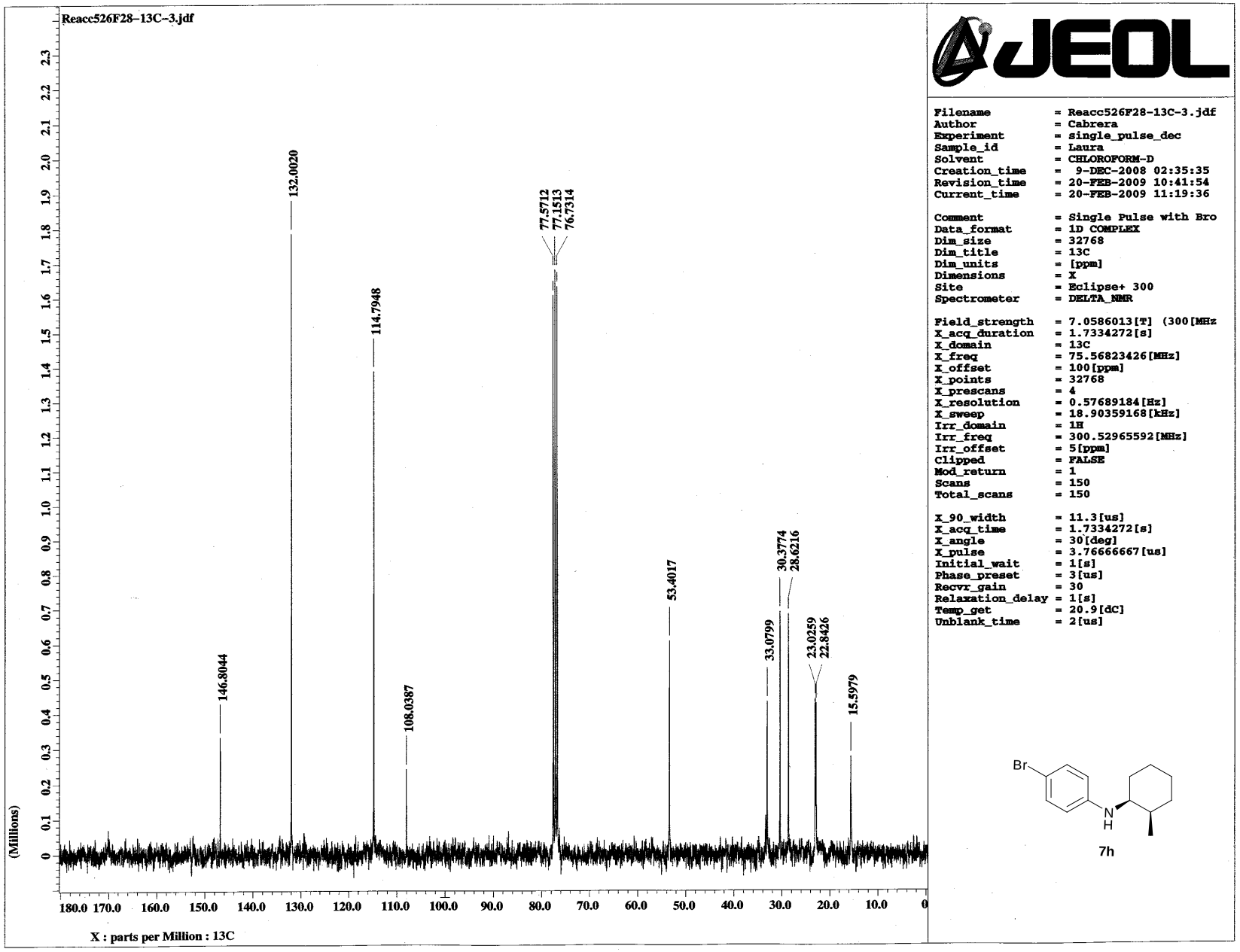
| Peak Label | RIC | Scan Range | Baseline at | Total Area | Background | Peak Area |
|---|---|---|---|---|---|---|
| 157.17 | TIC | 8397-8423 | 8385-8389 | 3629883 | 3379220 | 250663 |
| 157.83 | TIC | 8455-8481 | 8490-8494 | 7812256 | 3305930 | 4506326 |

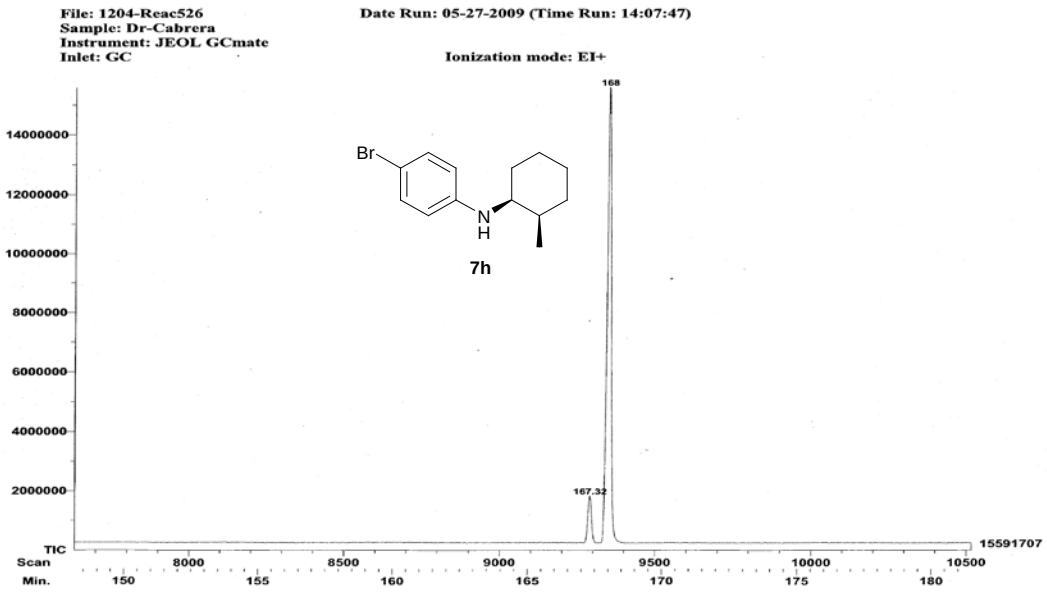


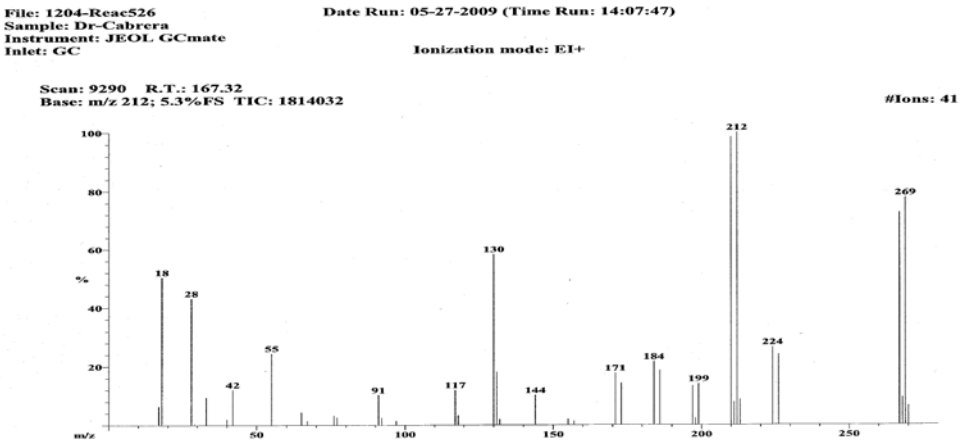

| | |
|---|---|
| Filename | = Reacc526F28-1H-4.jdf |
| Author | = Cabrera |
| Experiment | = single_pulse.exp |
| Sample_id | = Laura |
| Solvent | = CHLOROFORM-D |
| Creation_time | = 9-DEC-2008 02:27:29 |
| Revision_time | = 20-FEB-2009 11:34:52 |
| Current_time | = 20-FEB-2009 11:36:25 |
| Comment | = 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 | = 1 |
| 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] |

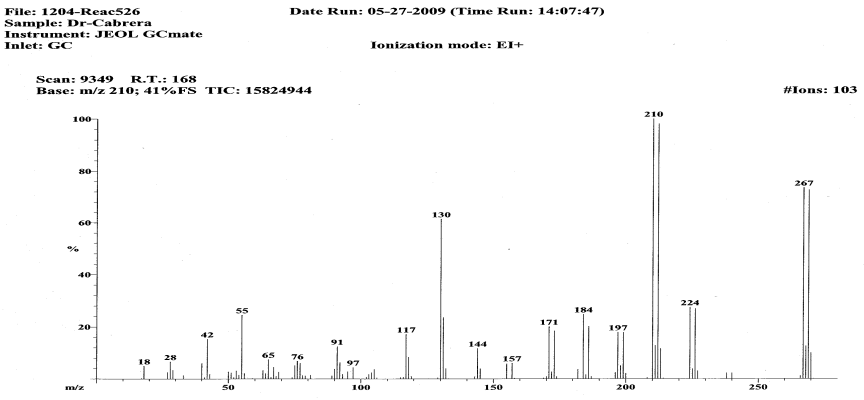



7h


Reacc526F28-13C-3.jdf
(Millions)
X : parts per Million : 13C
146.8044
132.0020
114.7948
108.0387
77.5712
77.1513
76.7314
53.4017
33.0799
30.3774
28.6216
23.0259
22.8426
15.5979
Filename = Reacc526F28-13C-3.jdf
Author = Cabrera
Experiment = single_pulse_dec
Sample_id = Laura
Solvent = CHLOROFORM-D
Creation_time = 9-DEC-2008 02:35:35
Revision_time = 20-FEB-2009 10:41:54
Current_time = 20-FEB-2009 11:19:36
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 = 150
Total_scans = 150
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]
Br
N
H
7h

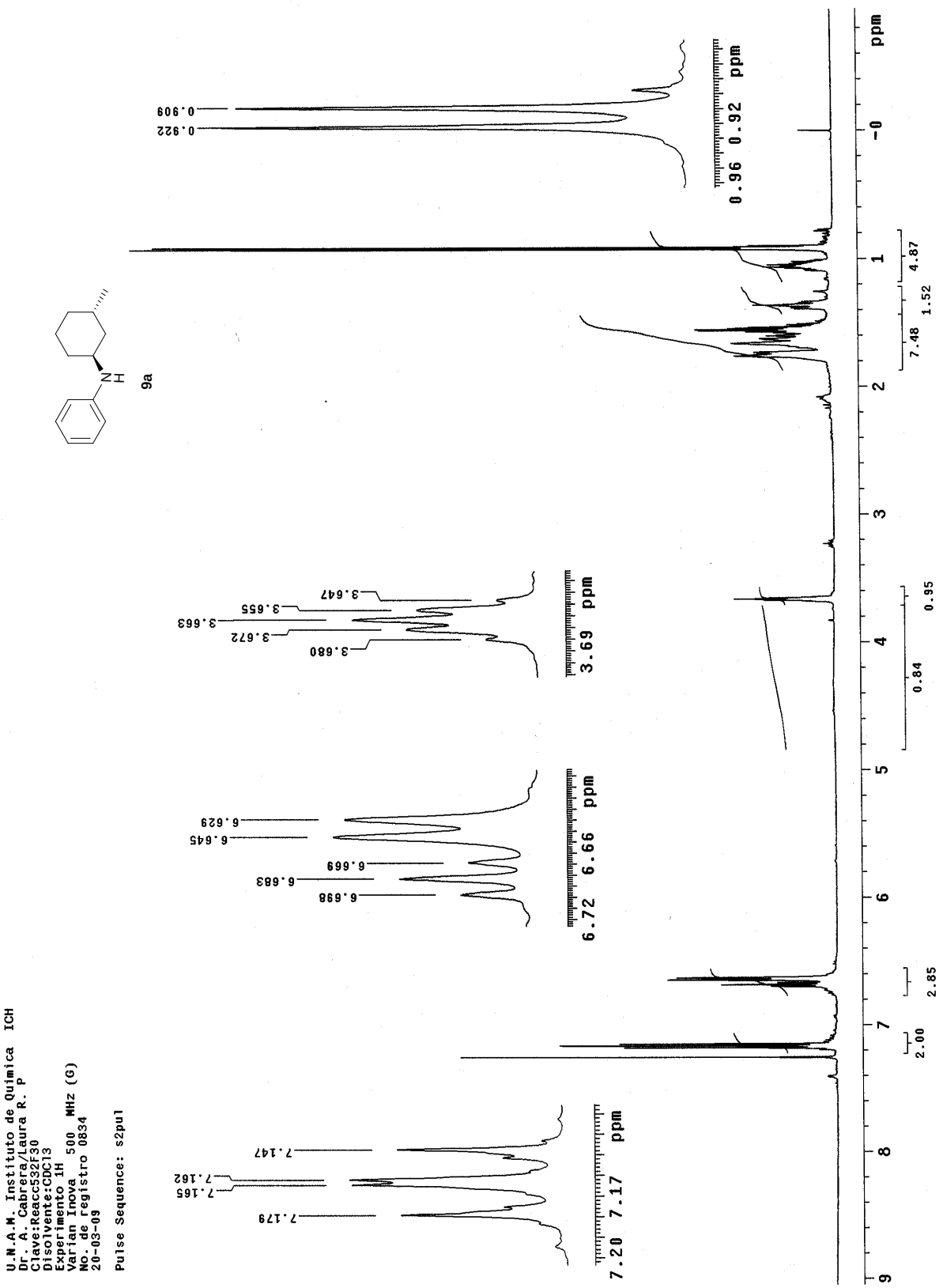


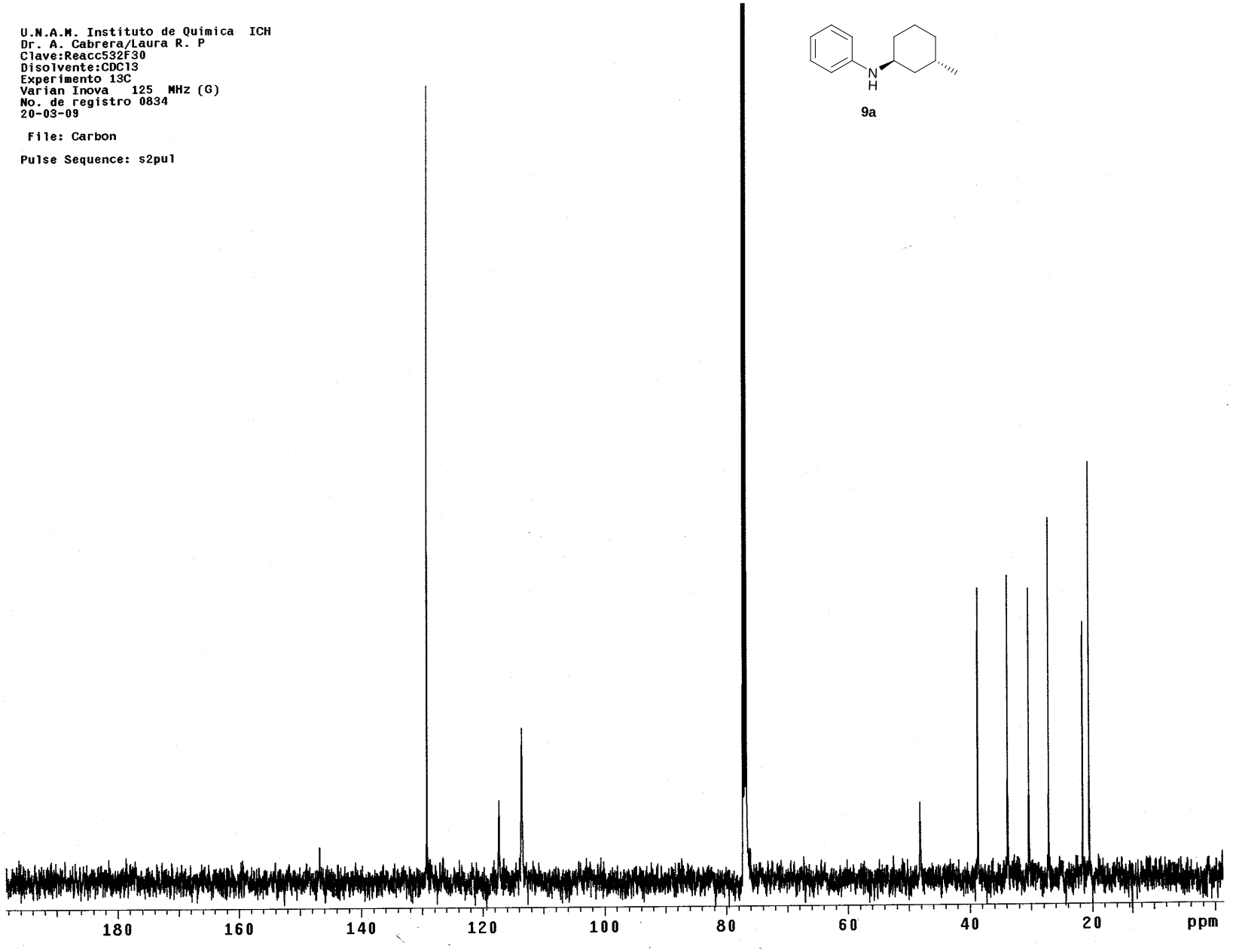



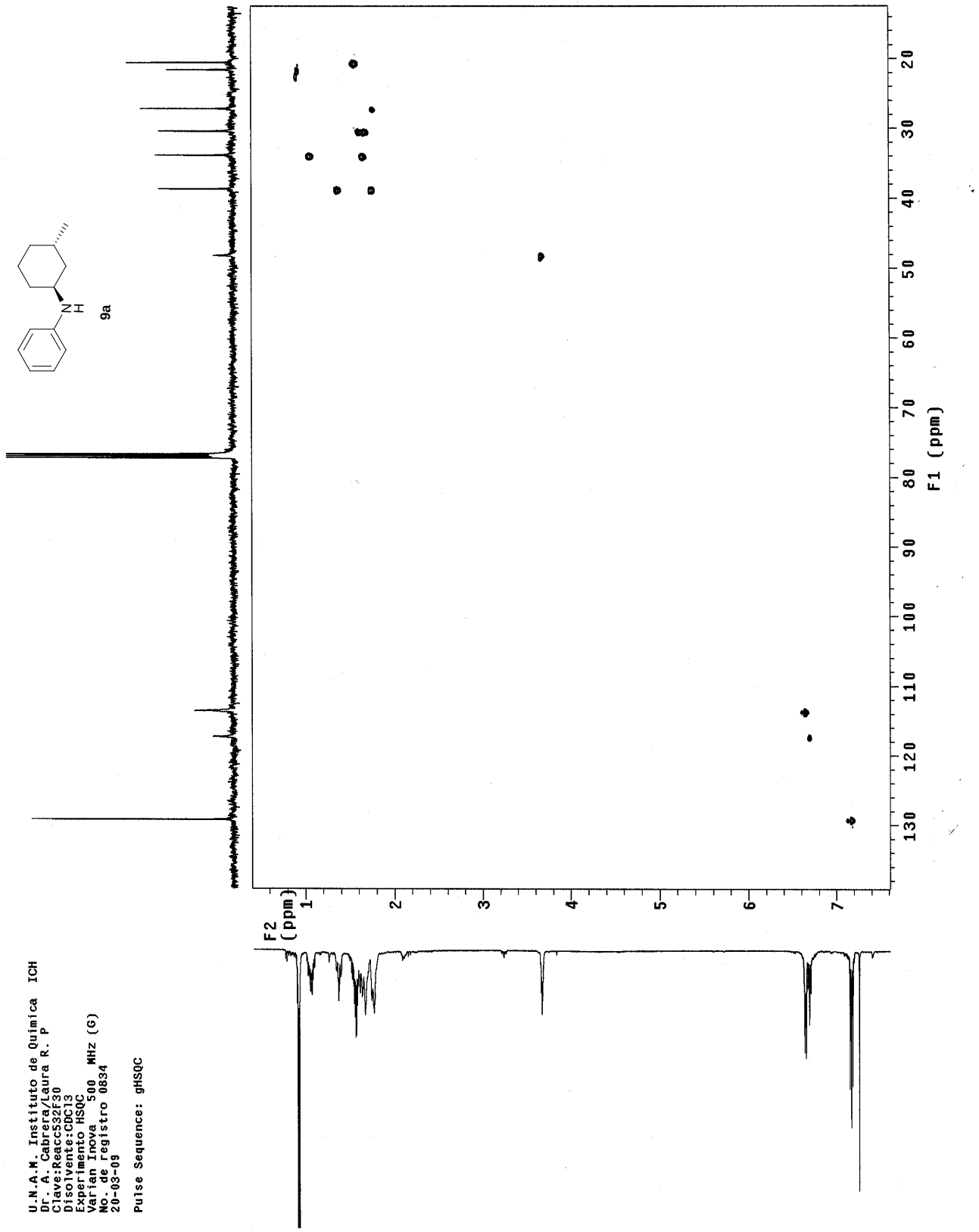



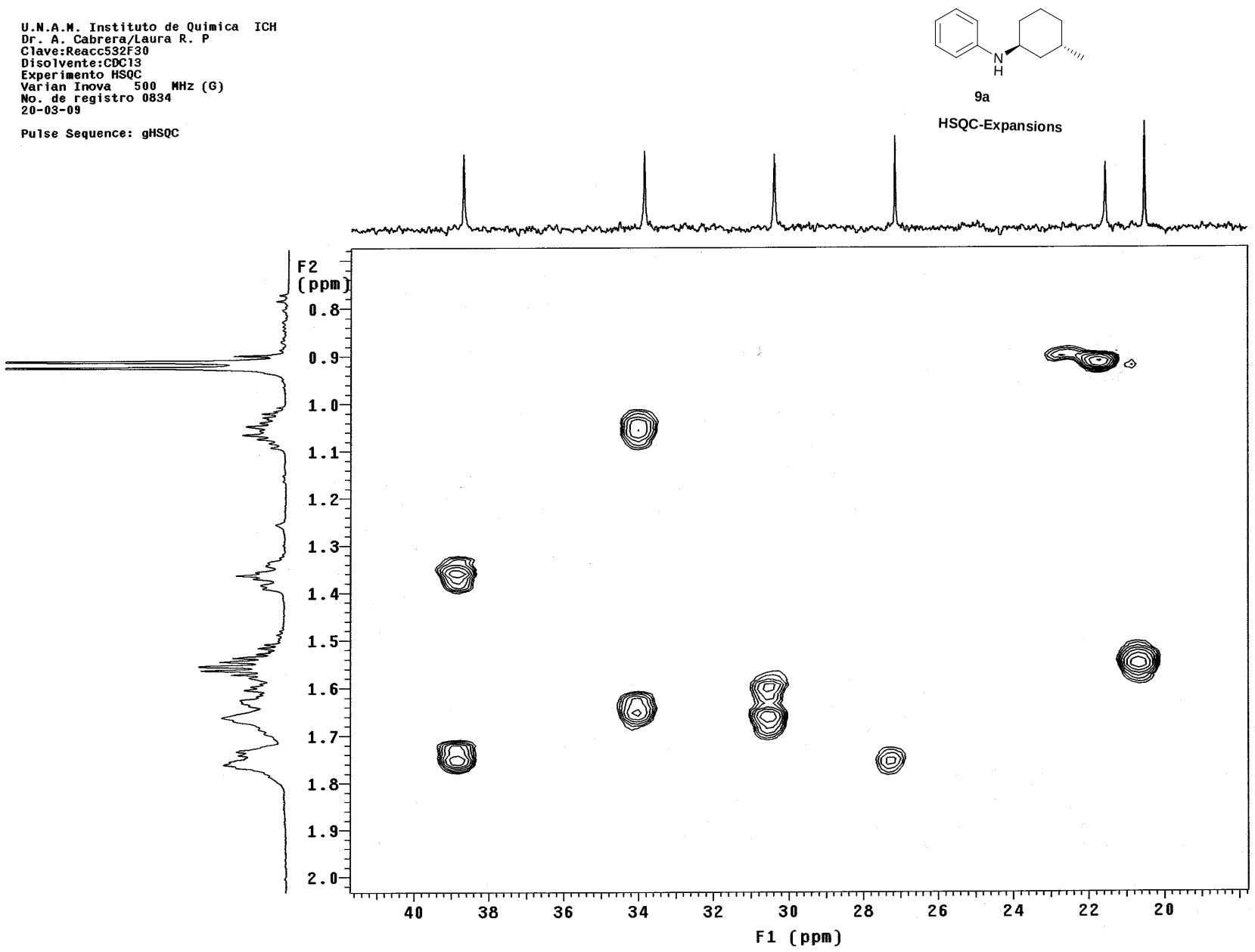

**Peak Table**

| Peak Label | RIC | Scan Range | Baseline at | Total Area | Background | Peak Area |
|---|---|---|---|---|---|---|
| 157.17 | TIC | 8397-8423 | 8385-8389 | 3629883 | 3379220 | 250663 |
| 157.83 | TIC | 8455-8481 | 8490-8494 | 7812256 | 3305930 | 4506326 |


U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc532F30
Disolvente:CDCl3
Experimento 1H
Varian Inova 500 MHz (G)
No. de registro 0834
20-03-09
Pulse Sequence: s2pul
9a
7.179
7.165
7.162
7.147
7.20 7.17 ppm
6.698
6.683
6.669
6.645
6.629
6.72 6.66 ppm
3.680
3.672
3.663
3.655
3.647
3.69 ppm
0.922
0.909
0.96 0.92 ppm
ppm
2.00
2.85
0.84
0.95
7.48
1.52
4.87

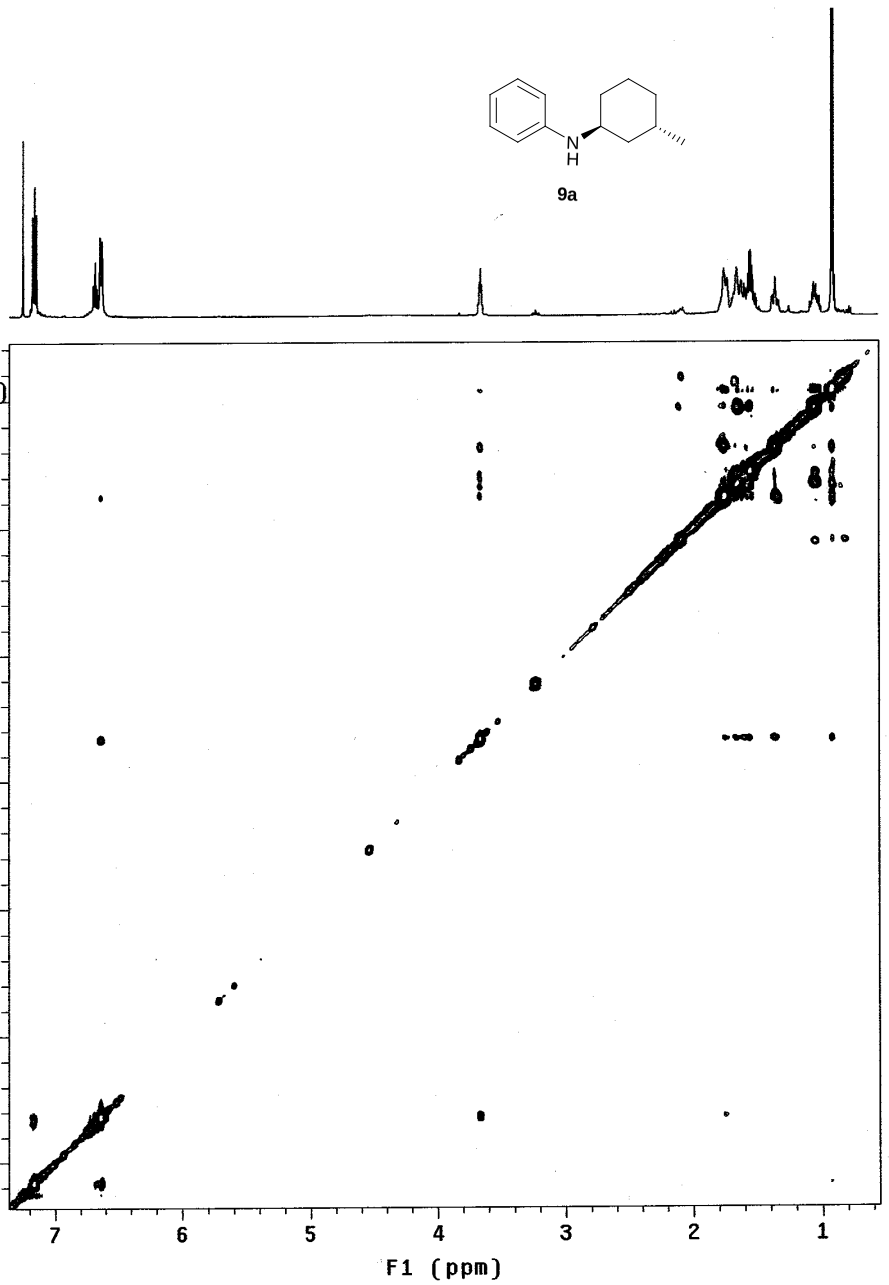

U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc532F30
Disolvente:CDCl3
Experimento 13C
Varian Inova 125 MHz (G)
No. de registro 0834
20-03-09
File: Carbon
Pulse Sequence: s2pul
N
H
9a
180
160
140
120
100
80
60
40
20
ppm


U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc532F30
Disolvente:CDCl3
Experimento HSQC
Varian Inova 500 MHz (G)
No. de registro 0834
20-03-09
Pulse Sequence: gHSQC
9a
F2 (ppm)
F1 (ppm)


U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc532F30
Disolvente:CDCl3
Experimento HSQC
Varian Inova 500 MHz (G)
No. de registro 0834
20-03-09
Pulse Sequence: gHSQC
9a
HSQC-Expansions
F2 (ppm)
0.8
0.9
1.0
1.1
1.2
1.3
1.4
1.5
1.6
1.7
1.8
1.9
2.0
40
38
36
34
32
30
28
26
24
22
20
F1 (ppm)

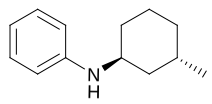
U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc532F30
Disolvente:CDCl3
Experimento NOESY
Varian Inova 500 MHz (G)
No. de registro 0834
20-03-09

Pulse Sequence: noesy



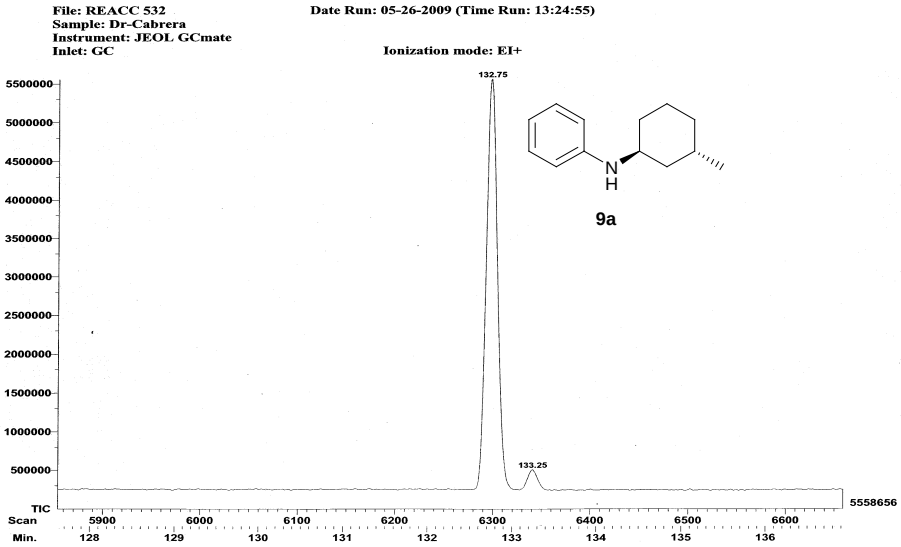
U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura R. P
Clave:Reacc532F30
Disolvente:CDCl3
Experimento NOESY
Varian Inova 500 MHz (G)
No. de registro 0834
20-03-09

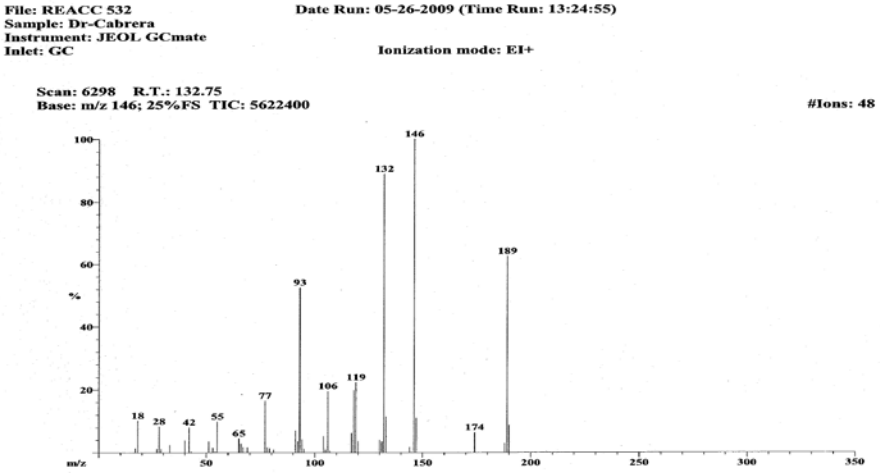
Pulse Sequence: noesy

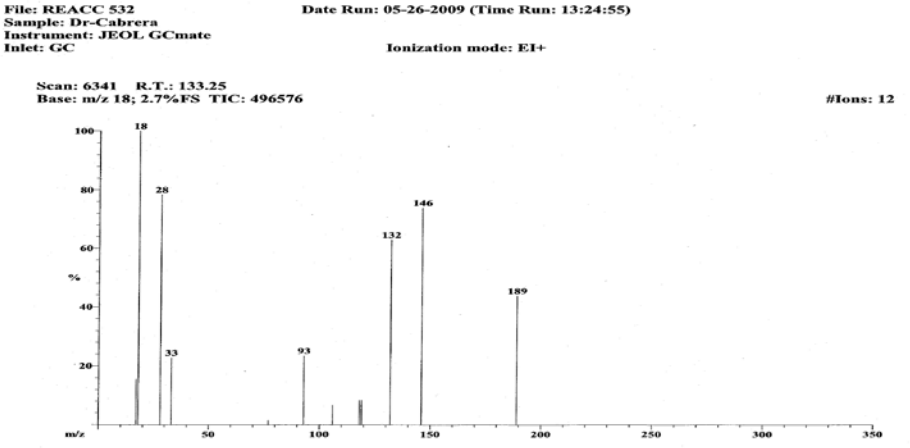



9a

NOESY-Expansions

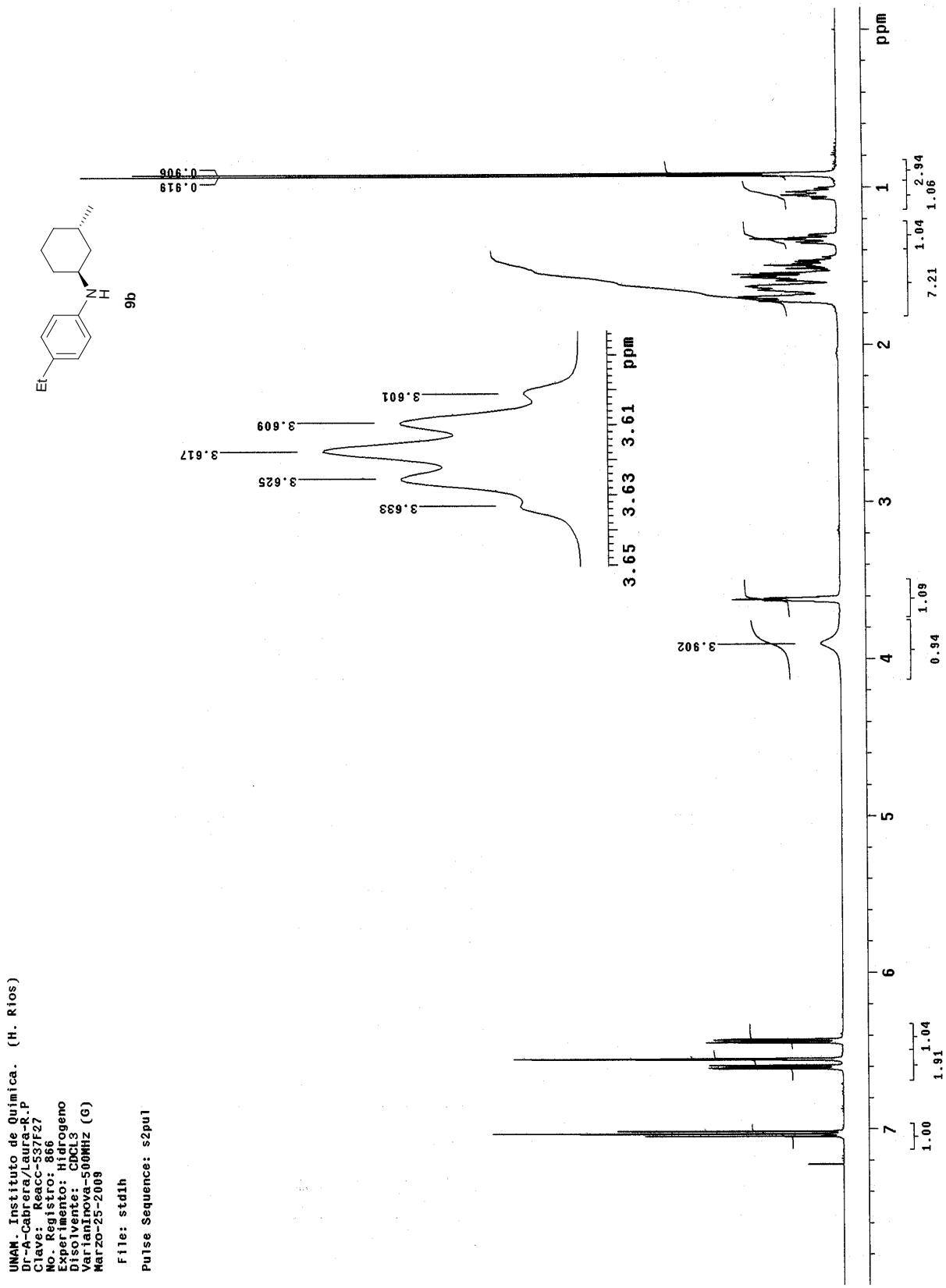


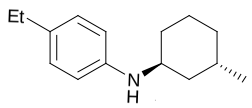




**Peak Table**

| Peak Label | RIC | Scan Range | Baseline at | Total Area | Background | Peak Area |
|---|---|---|---|---|---|---|
| 132.75 | TIC | 6286-6310 | 6274-6278 | 51692778 | 4175630 | 47517148 |
| 133.25 | TIC | 6329-6353 | 6360-6364 | 6197332 | 4020182 | 2177150 |


UNAM. Instituto de Quimica. (H. Rios)
Dr-A-Cabrera/Laura-R.P
Clave: Reacc-537F27
No. Registro: 866
Experimento: Hidrogeno
Disolvente: CDCL3
VarianInova-500MHz (G)
Marzo-25-2009
File: std1h
Pulse Sequence: s2pul
9b
Et
N
H
0.919
0.906
3.633
3.625
3.617
3.609
3.601
3.65 3.63 3.61 ppm
3.902
ppm
1.00
1.04
1.91
1.09
0.94
7.21
1.04
1.06
2.94

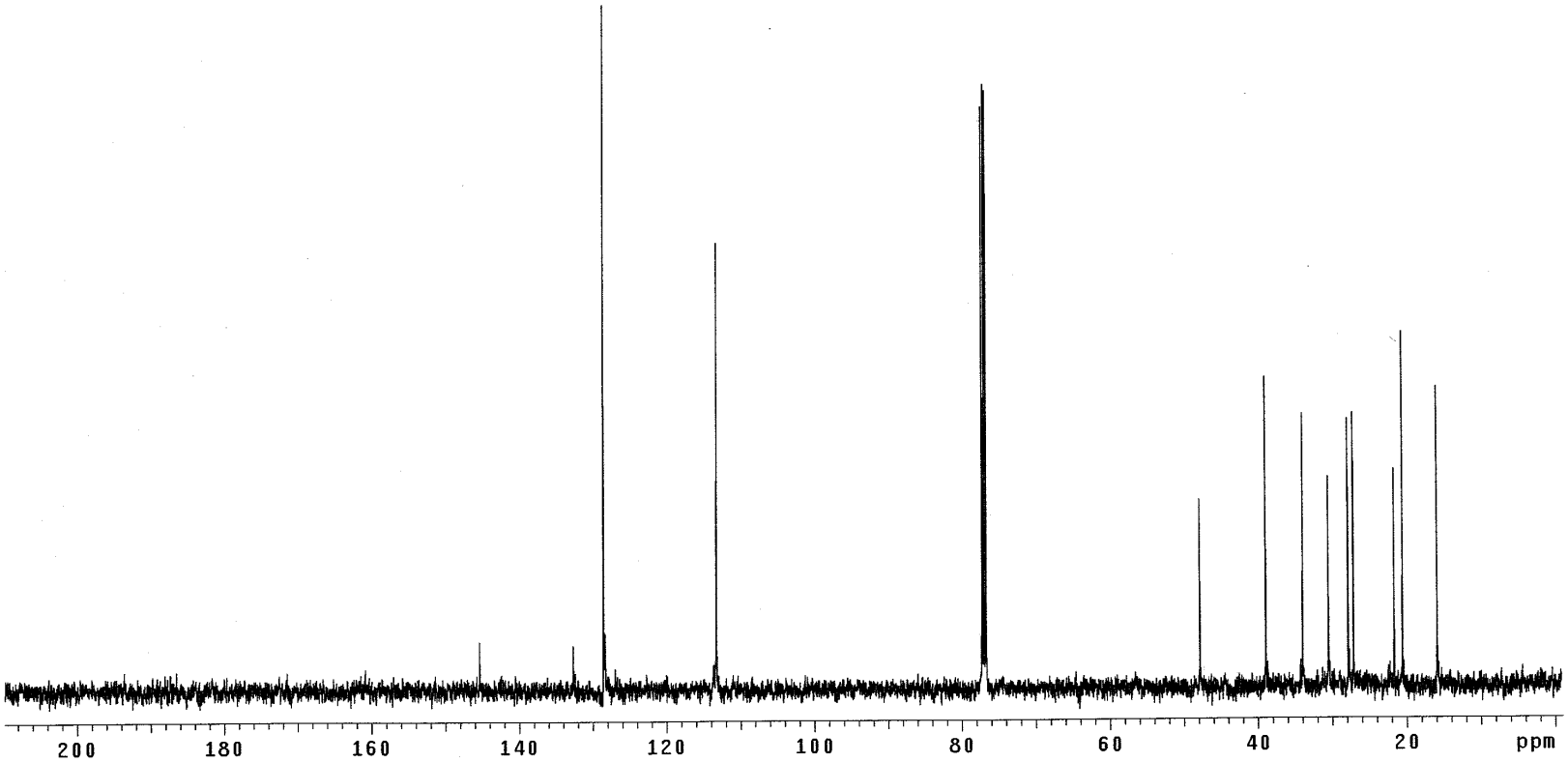
U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera/Laura RP
Clave : Reacc 538F2627
Disolvente: CDCl3
Experimento : 13C
Varian Inova 125 MHz
No. de Registro 1039
23-04-09

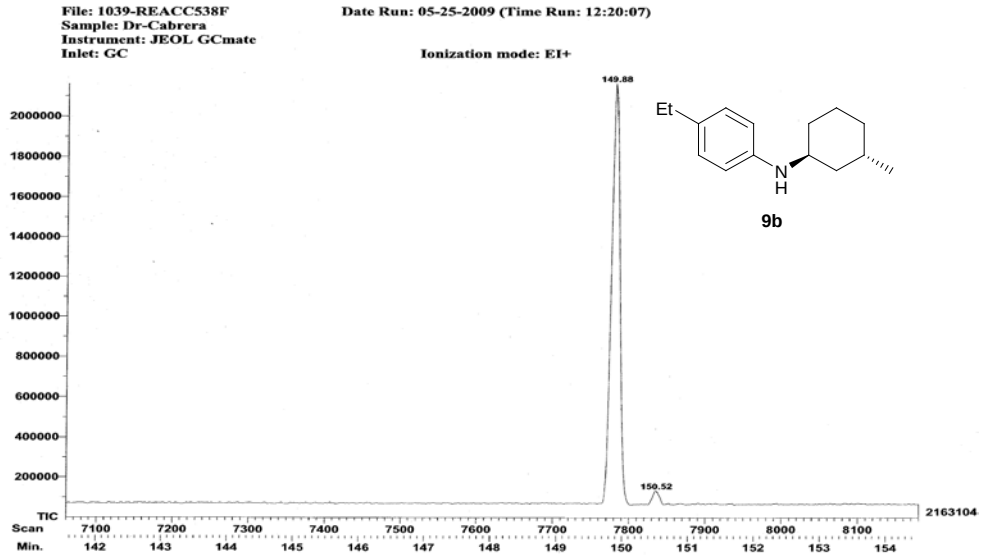


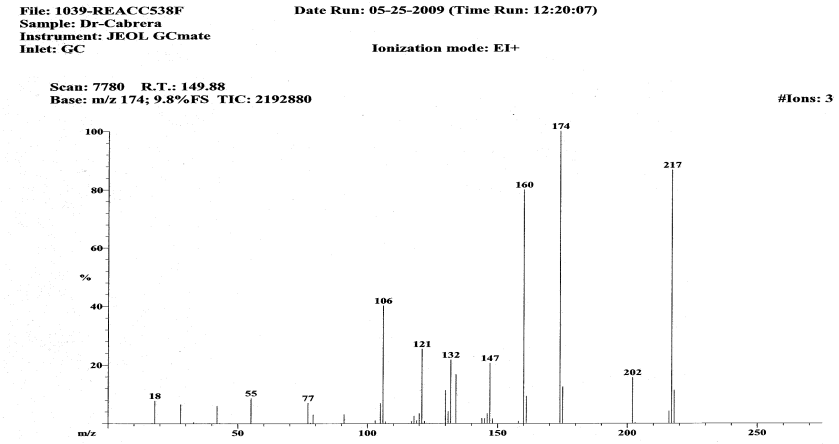

**9b**

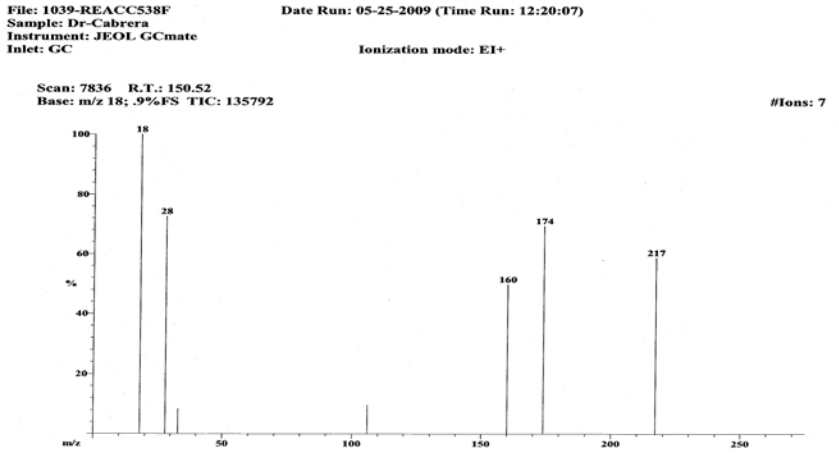
File: Carbon

Pulse Sequence: s2pul



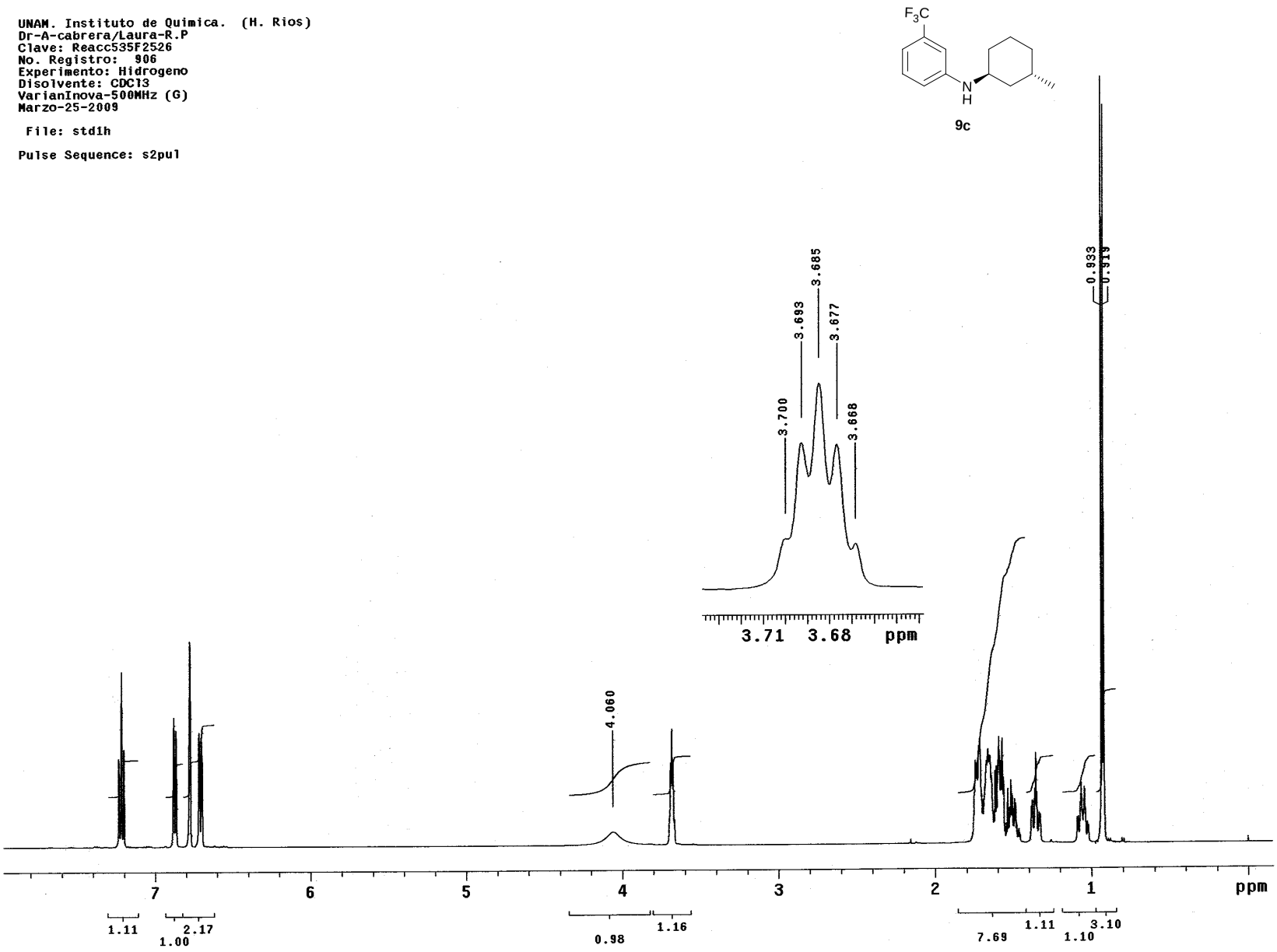


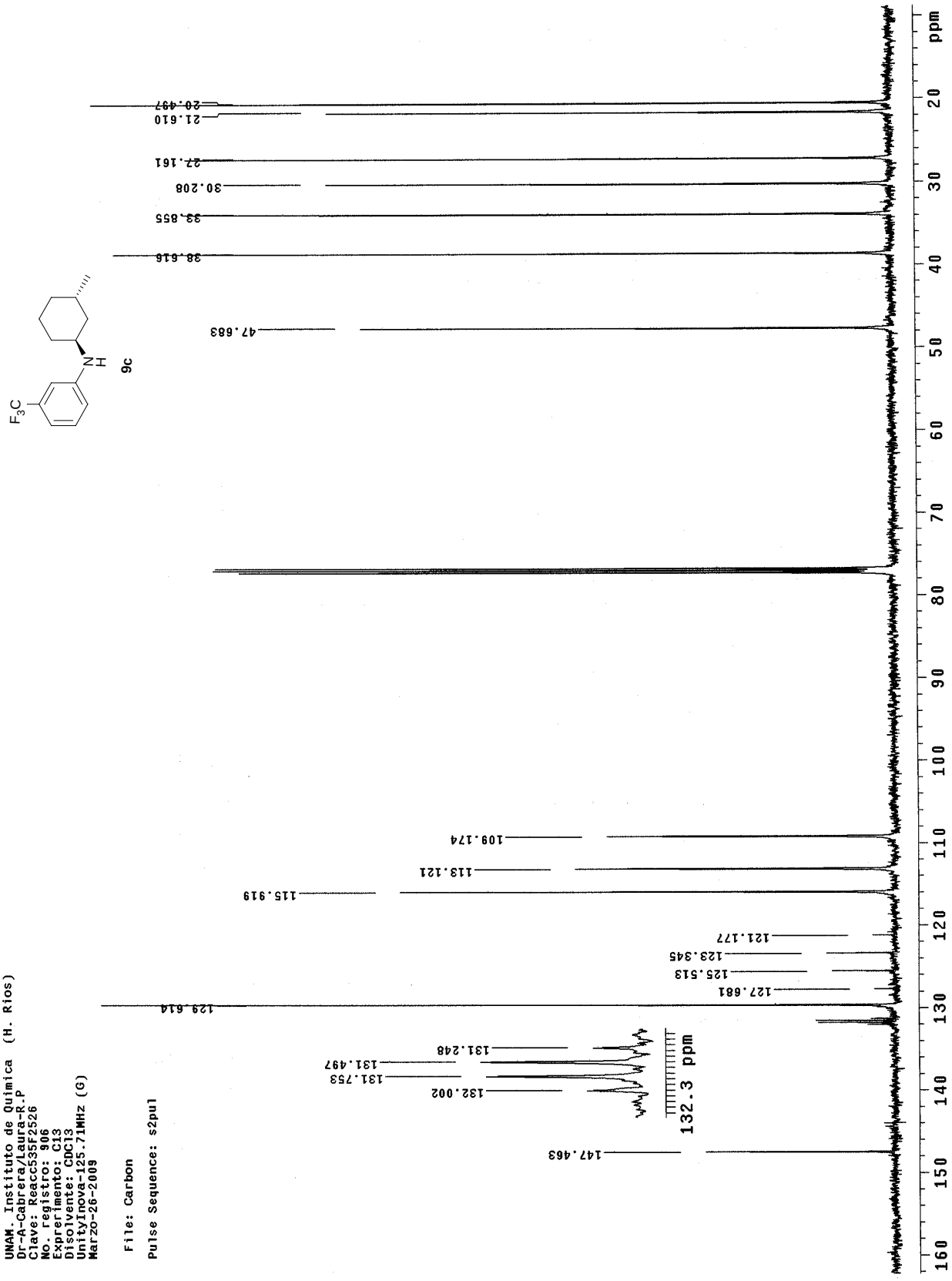




**Peak Table**

| Peak Label | RIC | Scan Range | Baseline at | Total Area | Background | Peak Area |
|---|---|---|---|---|---|---|
| 149.88 | TIC | 7768-7792 | 7801-7805 | 19318740 | 1071222 | 18247518 |
| 150.52 | TIC | 7824-7848 | 7856-7860 | 1571026 | 1086492 | 484534 |

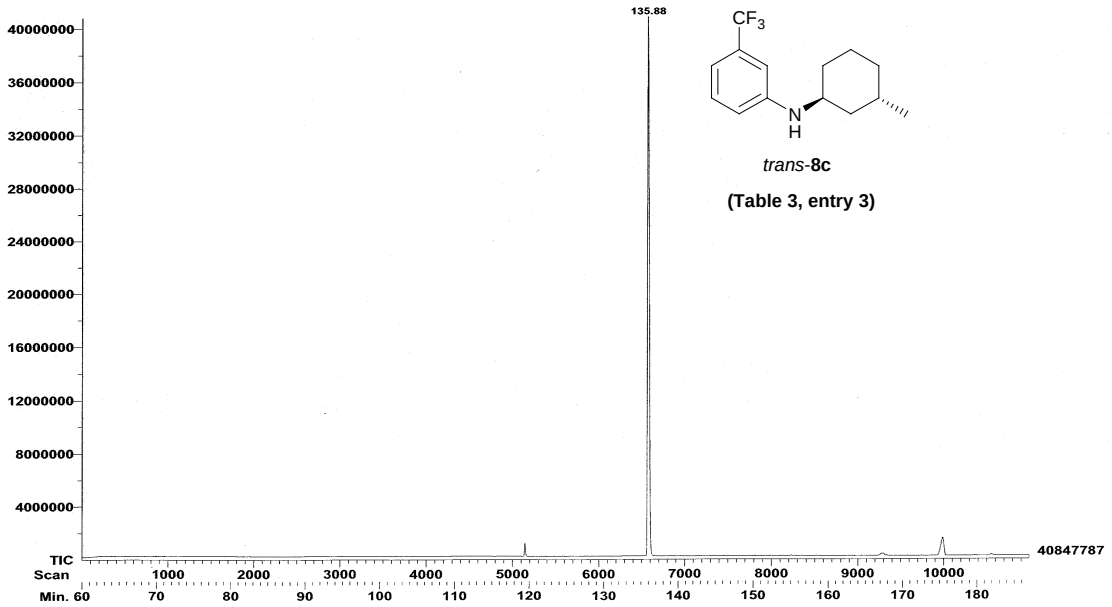

UNAM. Instituto de Quimica. (H. Rios)
Dr-A-cabrera/Laura-R.P
Clave: Reacc535F2526
No. Registro: 906
Experimento: Hidrogeno
Disolvente: CDCl3
VarianInova-500MHz (G)
Marzo-25-2009
File: std1h
Pulse Sequence: s2pul
F3C
N
H
9c
3.700
3.693
3.685
3.677
3.668
3.71 3.68 ppm
0.933
4.060
7
6
5
4
3
2
1
ppm
1.11
2.17
1.00
0.98
1.16
7.69
1.11
1.10
3.10


UNAM. Instituto de Quimica (H. Rios)
Dr-A-Cabrera/Laura-R.P
Clave: Reacc535F2526
No. registro: 906
Experimento: C13
Disolvente: CDCl3
UnityInova-125.71MHz (G)
Marzo-26-2009
File: Carbon
Pulse Sequence: s2pul
9c
F3C
NH
20.497
21.610
27.161
30.208
33.855
38.616
47.683
109.174
113.121
115.919
121.177
123.345
125.513
127.681
129.614
131.248
131.497
131.753
132.002
147.463
132.3 ppm
ppm

File: 1183-reac-535
Sample: Dr-Cabrera
Instrument: JEOL GCmate
Inlet: GC

Date Run: 05-25-2009 (Time Run: 19:05:23)

Ionization mode: EI+




*trans*-**8c**

**(Table 3, entry 3)**

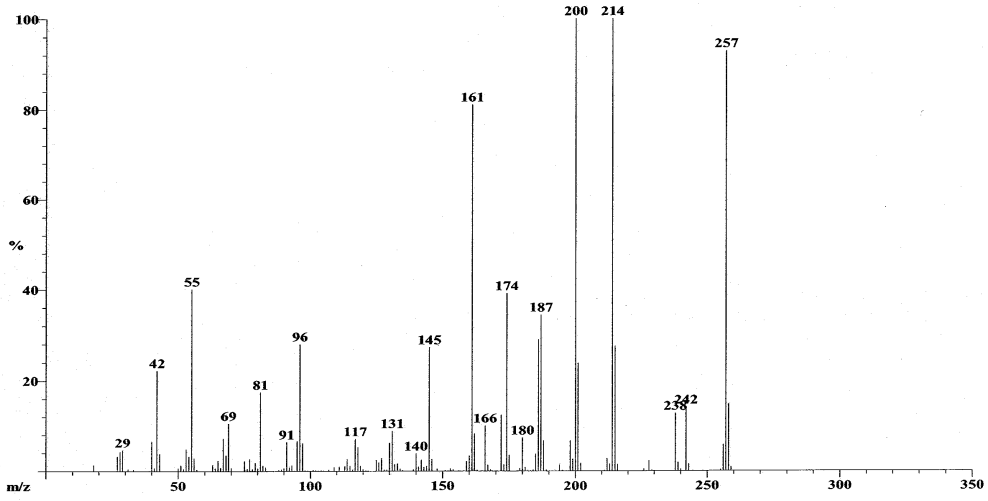
File: 1183-reac-535
Sample: Dr-Cabrera
Instrument: JEOL GCmate
Inlet: GC

Date Run: 05-25-2009 (Time Run: 19:05:23)

Ionization mode: EI+

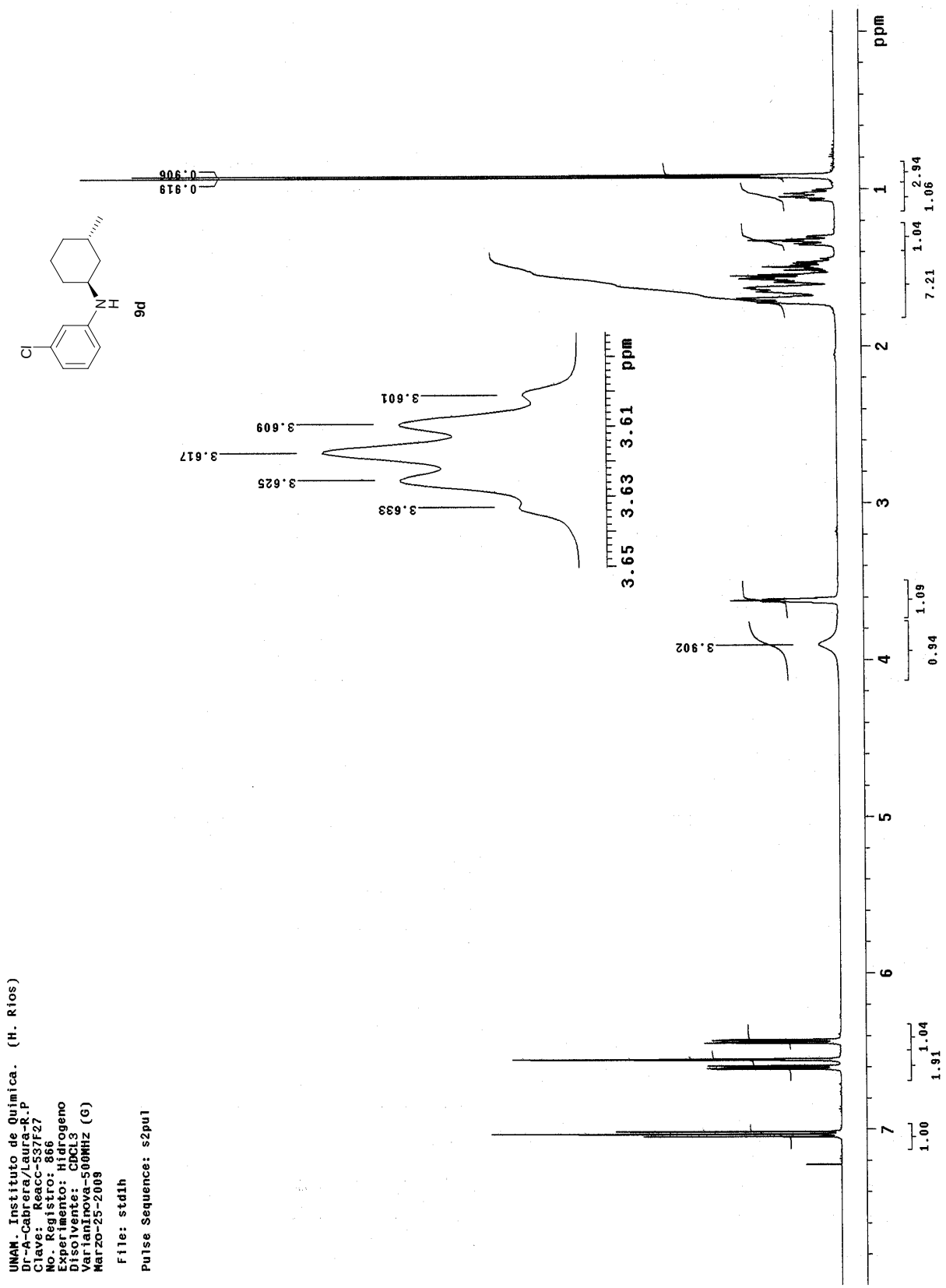
Scan: 6569 R.T.: 135.88
Base: m/z 214; 99.6%FS TIC: 40759472

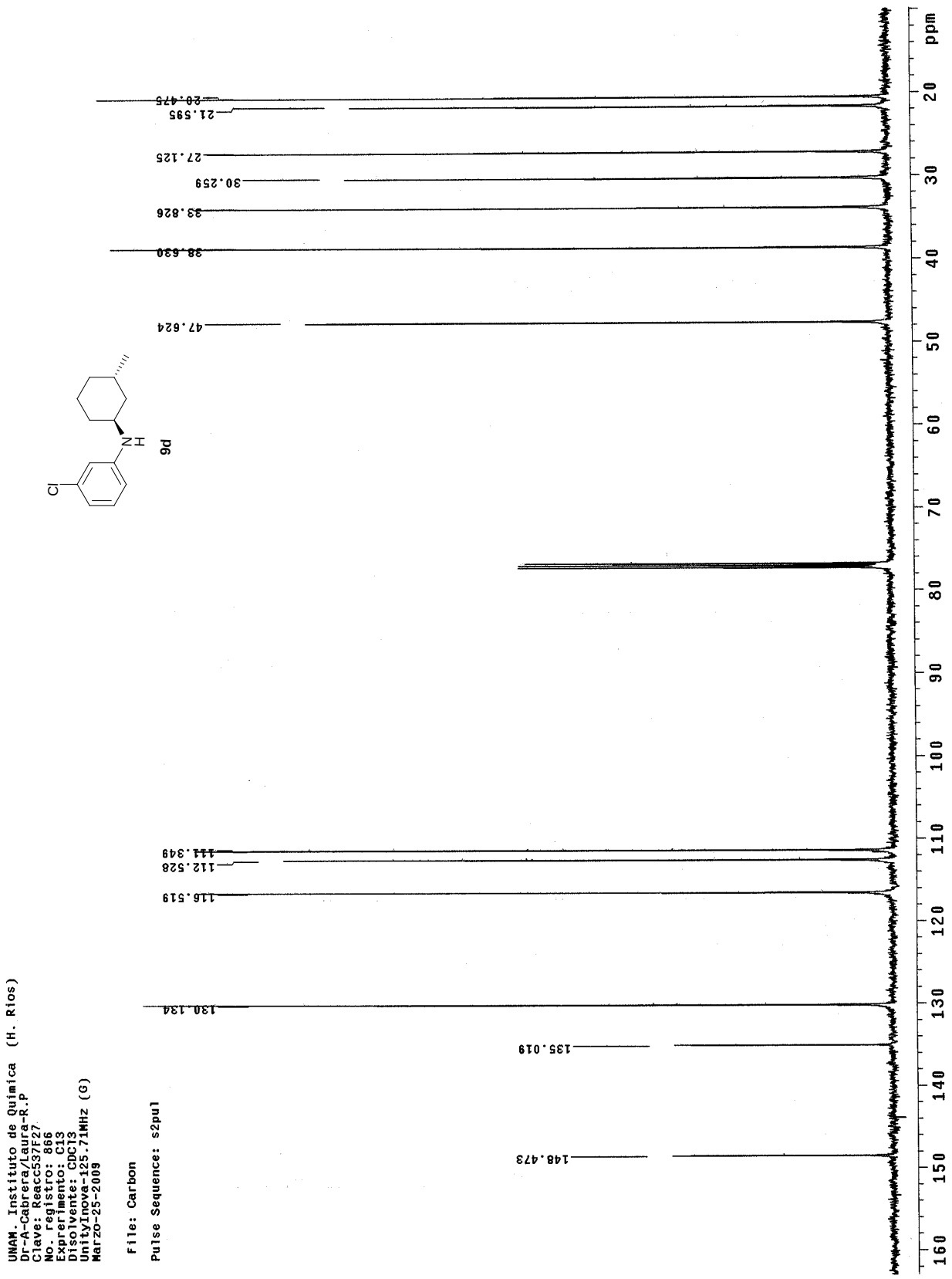
#Ions: 163




**Peak Table**

| Peak Label | RIC | Scan Range | Baseline at | Total Area | Background | Peak Area |
|---|---|---|---|---|---|---|
| 135.88 | TIC | 6557-6581 | 6547-6551 | 478247767 | 4150451 | 474097316 |

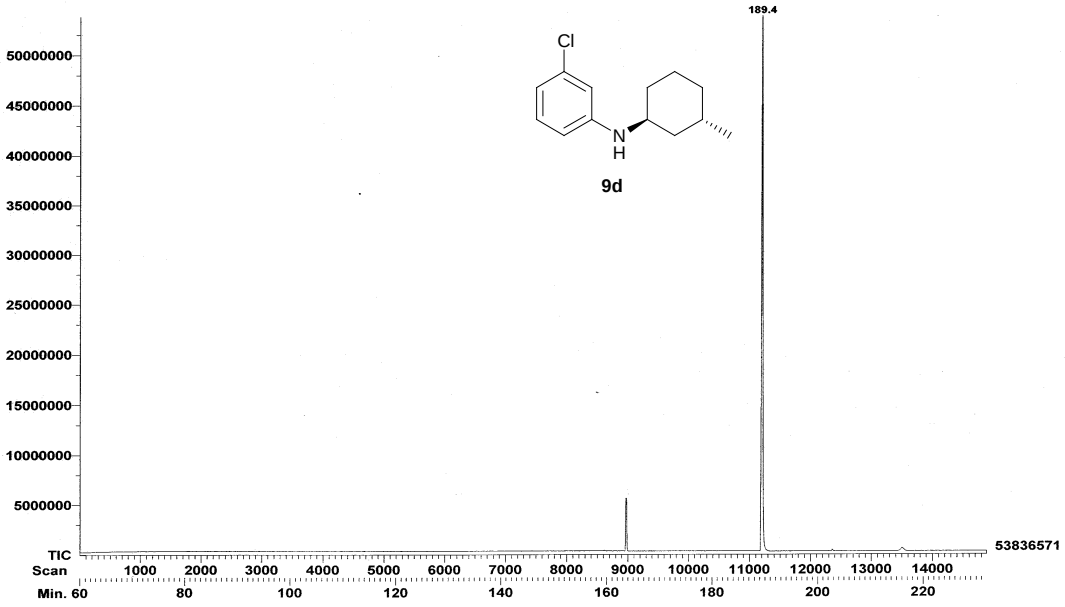

UNAM. Instituto de Quimica. (H. Rios)
Dr-A-Cabrera/Laura-R.P
Clave: Reacc-537F27
No. Registro: 866
Experimento: Hidrogeno
Disolvente: CDCL3
VarianInova-500MHz (G)
Marzo-25-2009
File: std1h
Pulse Sequence: s2pul
p6
Cl
NH
0.919
0.906
3.902
3.633
3.625
3.617
3.609
3.601
3.65 3.63 3.61 ppm
ppm
1.00
1.04
1.91
1.09
0.94
1.04
7.21
2.94
1.06


UNAM. Instituto de Quimica (H. Rios)
Dr-A-Cabrera/Laura-R.P
Clave: Reacc537F27
No. registro: 866
Experimento: C13
Disolvente: CDCl3
UnityInova-125.71MHz (G)
Marzo-25-2009
File: Carbon
Pulse Sequence: s2pul
p6d
Cl
N
H
148.473
135.019
130.134
116.519
112.528
111.349
47.624
38.630
33.826
30.259
27.125
21.595
20.475
ppm

File: REACC-537
Sample: Dr-Cabrera
Instrument: JEOL GCmate
Inlet: GC

Date Run: 05-26-2009 (Time Run: 09:02:42)

Ionization mode: EI+



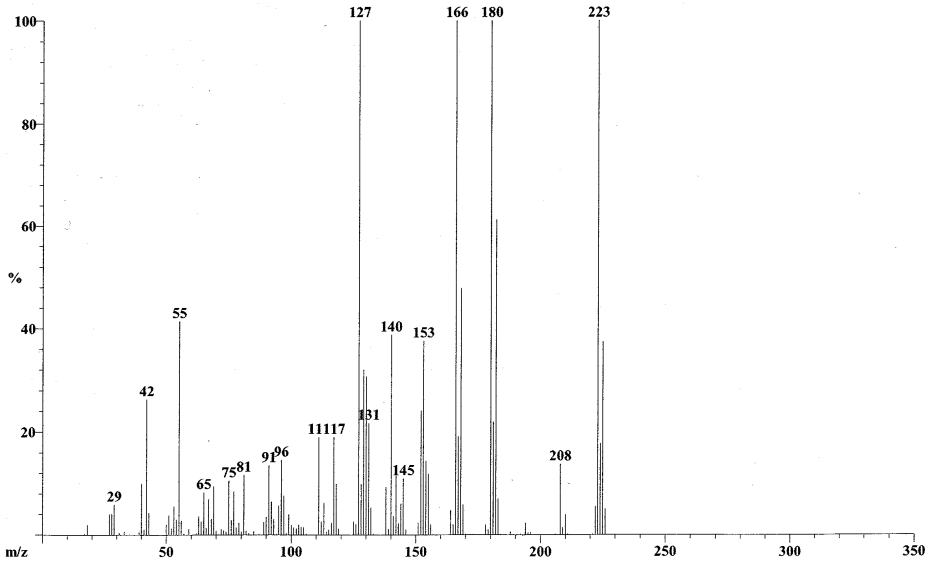

File: REACC-537
Sample: Dr-Cabrera
Instrument: JEOL GCmate
Inlet: GC

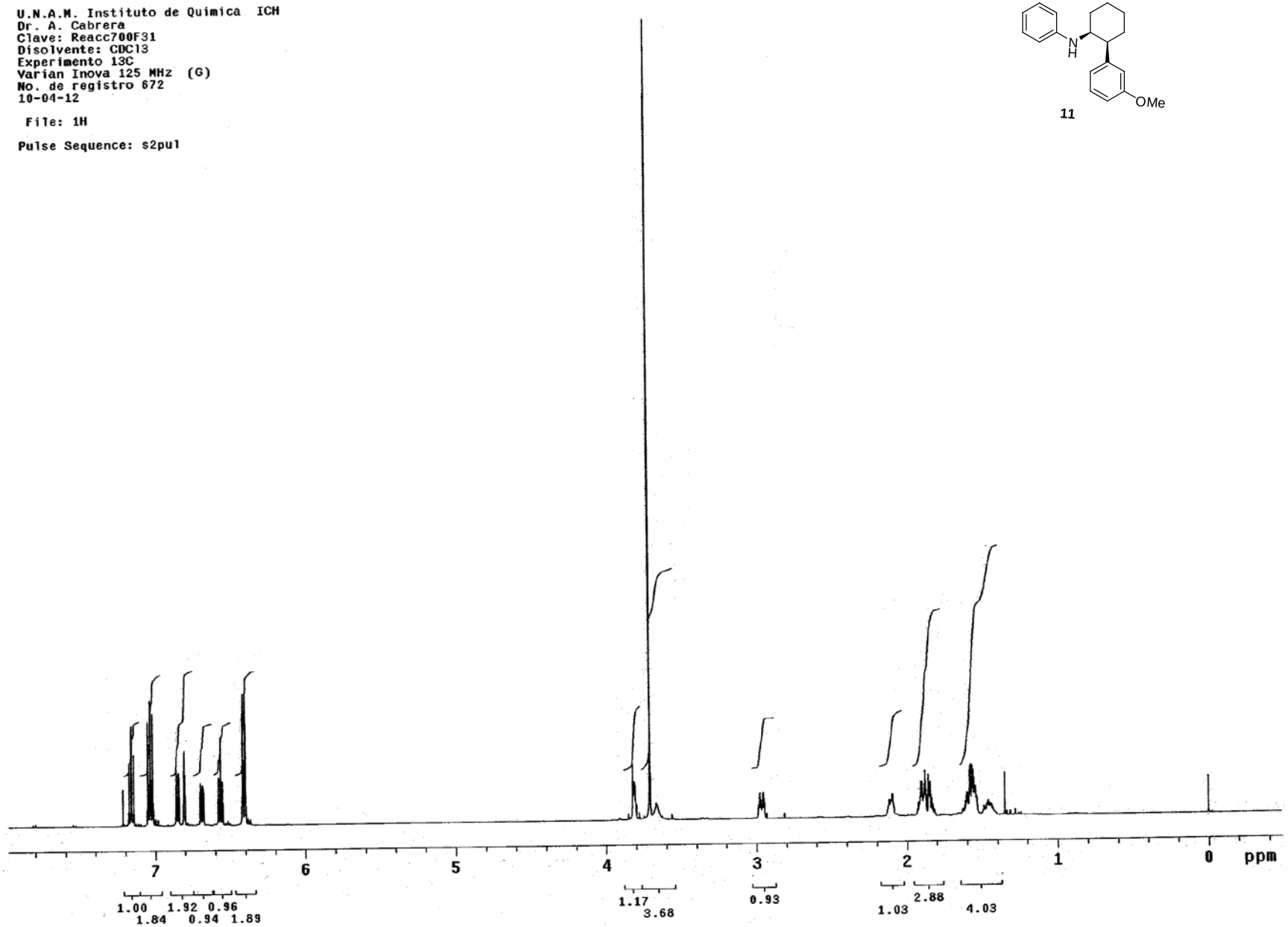
Date Run: 05-26-2009 (Time Run: 09:02:42)

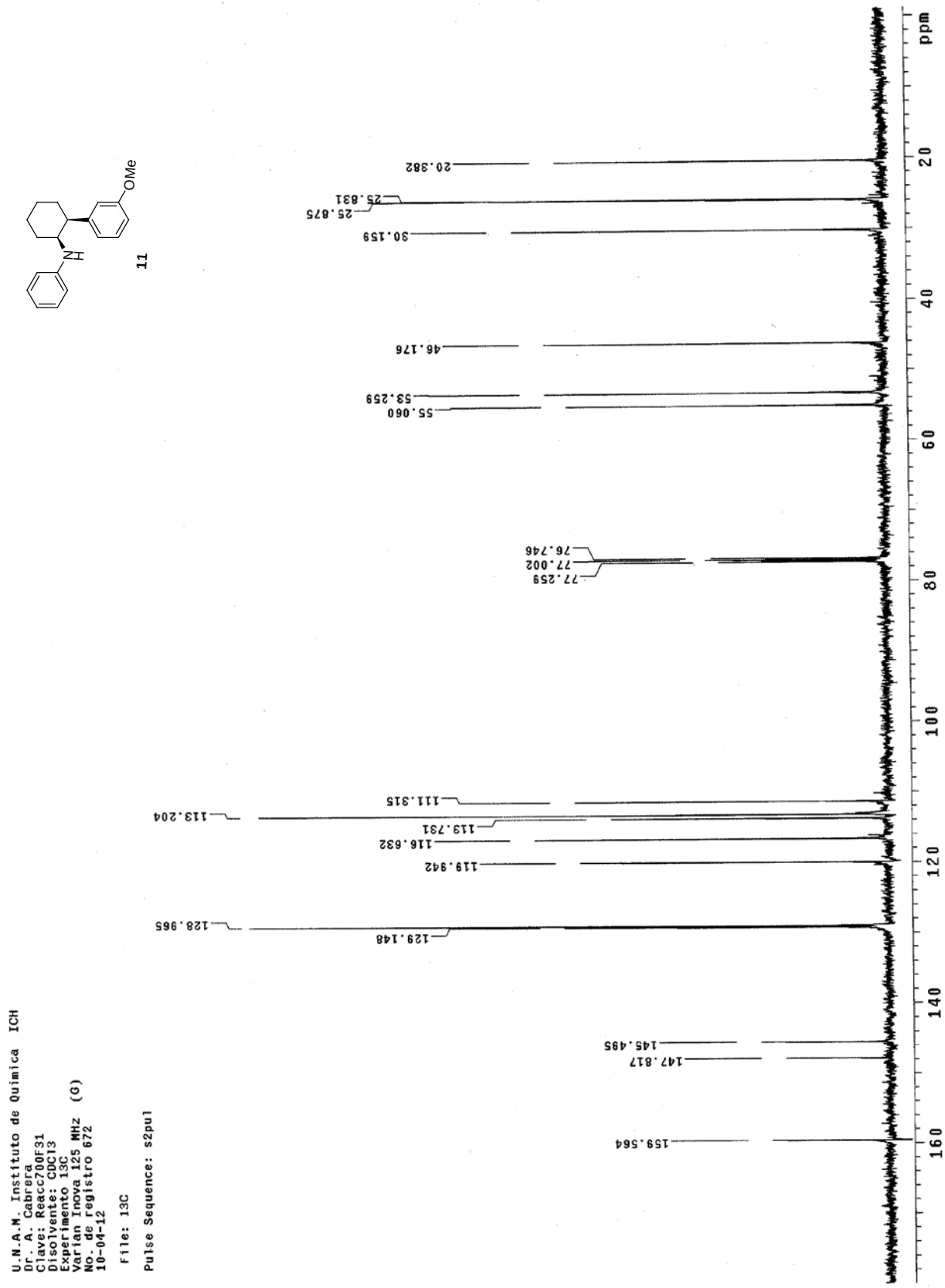
Ionization mode: EI+

Scan: 11201 R.T.: 189.4
Base: m/z 223; 99.6%FS TIC: 53914192

#Ions: 153

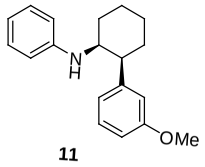



U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera
Clave: Reacc700F31
Disolvente: CDCl3
Experimento 13C
Varian Inova 125 MHz (G)
No. de registro 672
10-04-12
File: 1H
Pulse Sequence: s2pul
11
OMe
7
6
5
4
3
2
1
0
ppm
1.00
1.84
1.92
0.94
0.96
1.89
1.17
3.68
0.93
1.03
2.88
4.03


U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera
Clave: Reacc700F31
Disolvente: CDCl3
Experimento 13C
Varian Inova 125 MHz (G)
No. de registro 672
10-04-12
File: 13C
Pulse Sequence: s2pul
159.564
147.817
145.495
129.148
128.965
119.942
116.632
113.731
113.204
111.315
77.259
77.002
76.746
55.060
53.259
46.176
30.159
25.875
25.831
20.382
11
OMe
H
N
ppm
20
40
60
80
100
120
140
160

U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera
Clave: Reacc700F31
Disolvente: CDCl3
Experimento NOESY
Varian Inova 500 MHz (G)
No. de registro 672
10-04-12

Pulse Sequence: noesy

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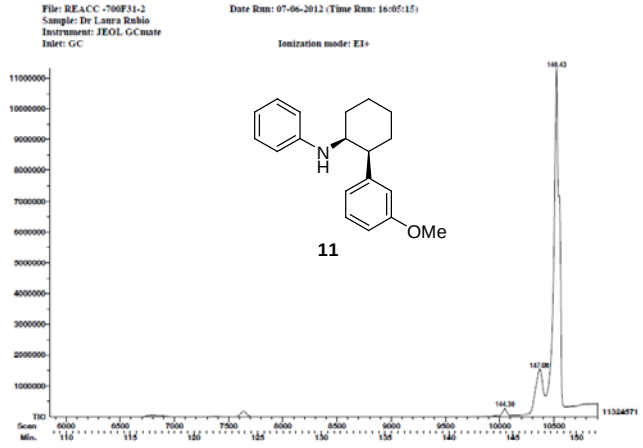
*Instituto de Química (UNAM)*



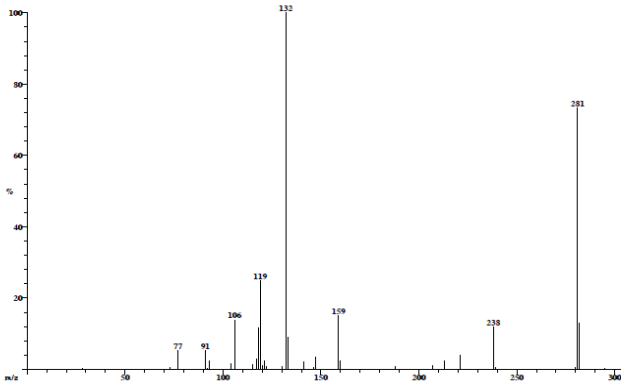

**11**

File: REACC -700F31-2
Sample: Dr Laura Rubio
Instrument: JEOL GCmate
Inlet: GC

Date Run: 07-06-2012 (Time Run: 16:05:15)

Ionization mode: EI+

Scan: 10366 R.T.: 147.08
Base: m/z 132; 11.3%FS TIC: 1557808

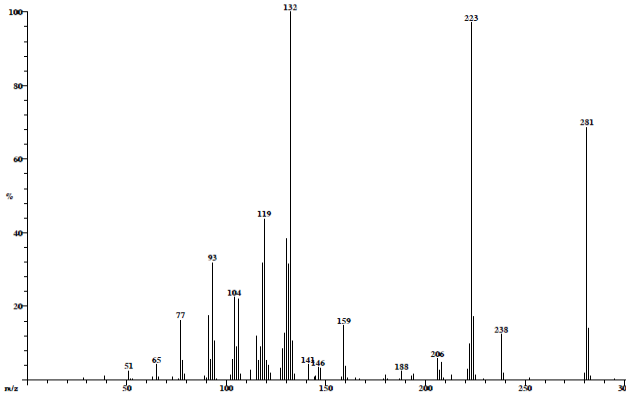



File: REACC -700F31-2
Sample: Dr Laura Rubio
Instrument: JEOL GCmate
Inlet: GC

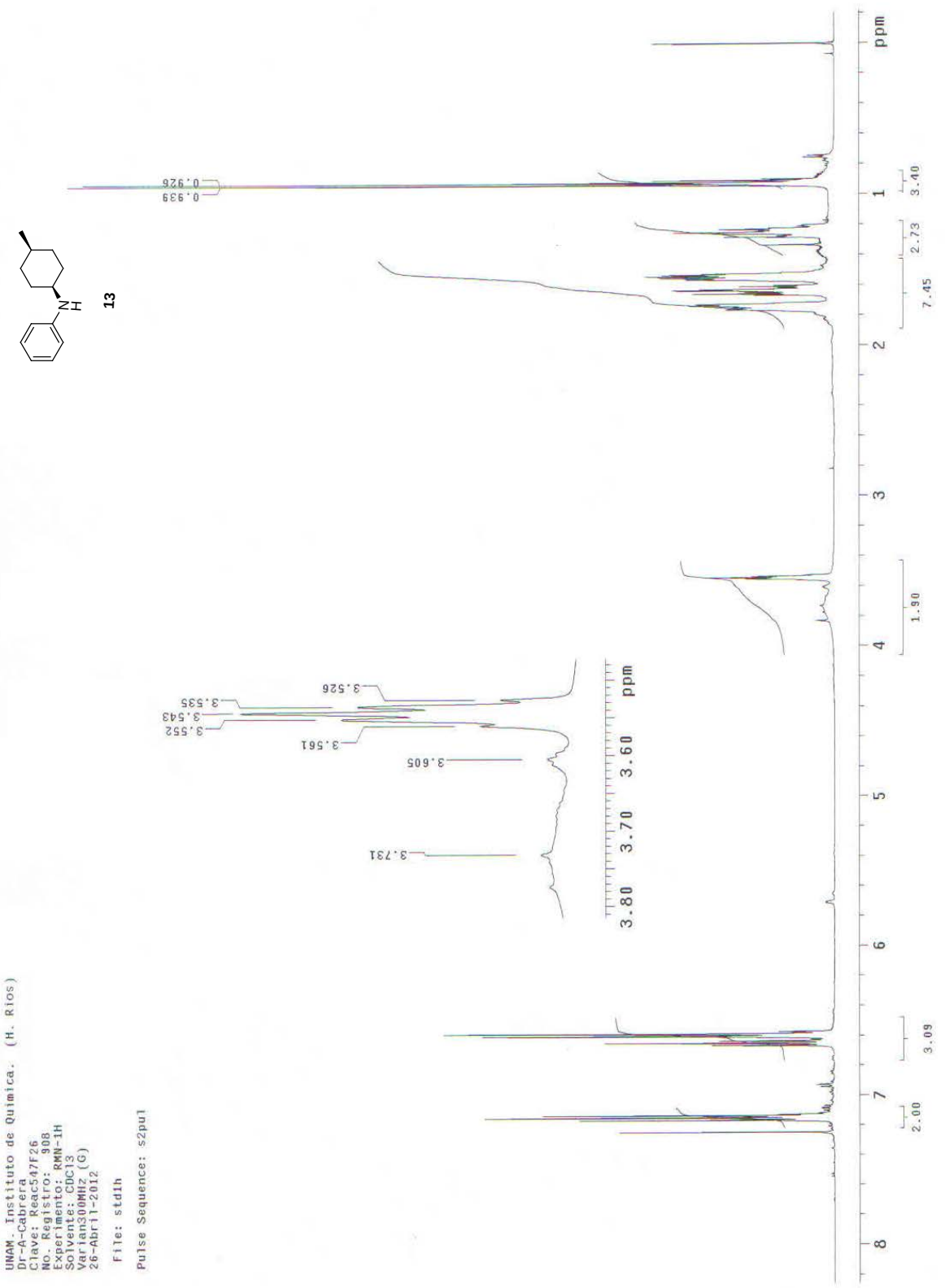
Date Run: 07-06-2012 (Time Run: 16:05:15)

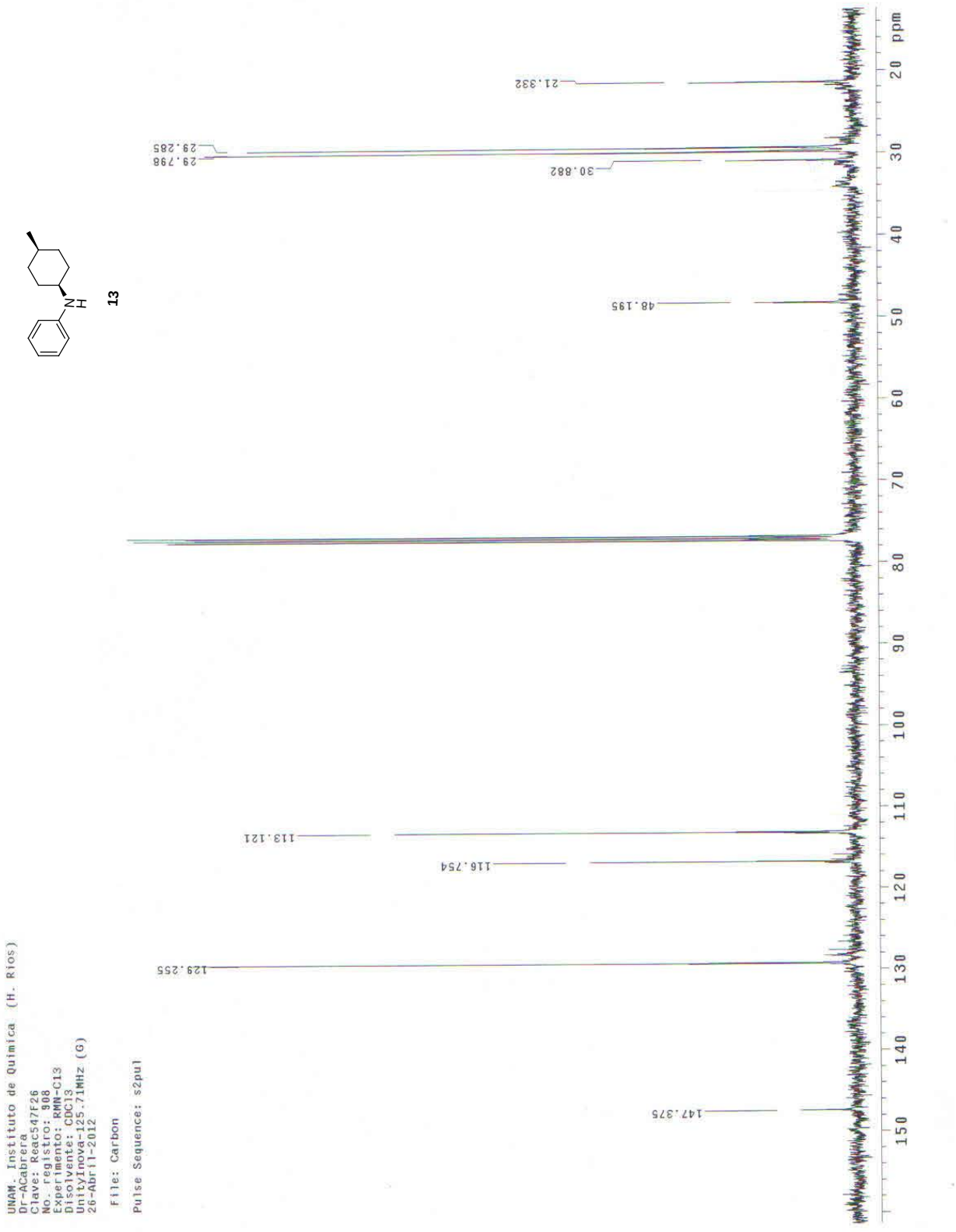
Ionization mode: EI+

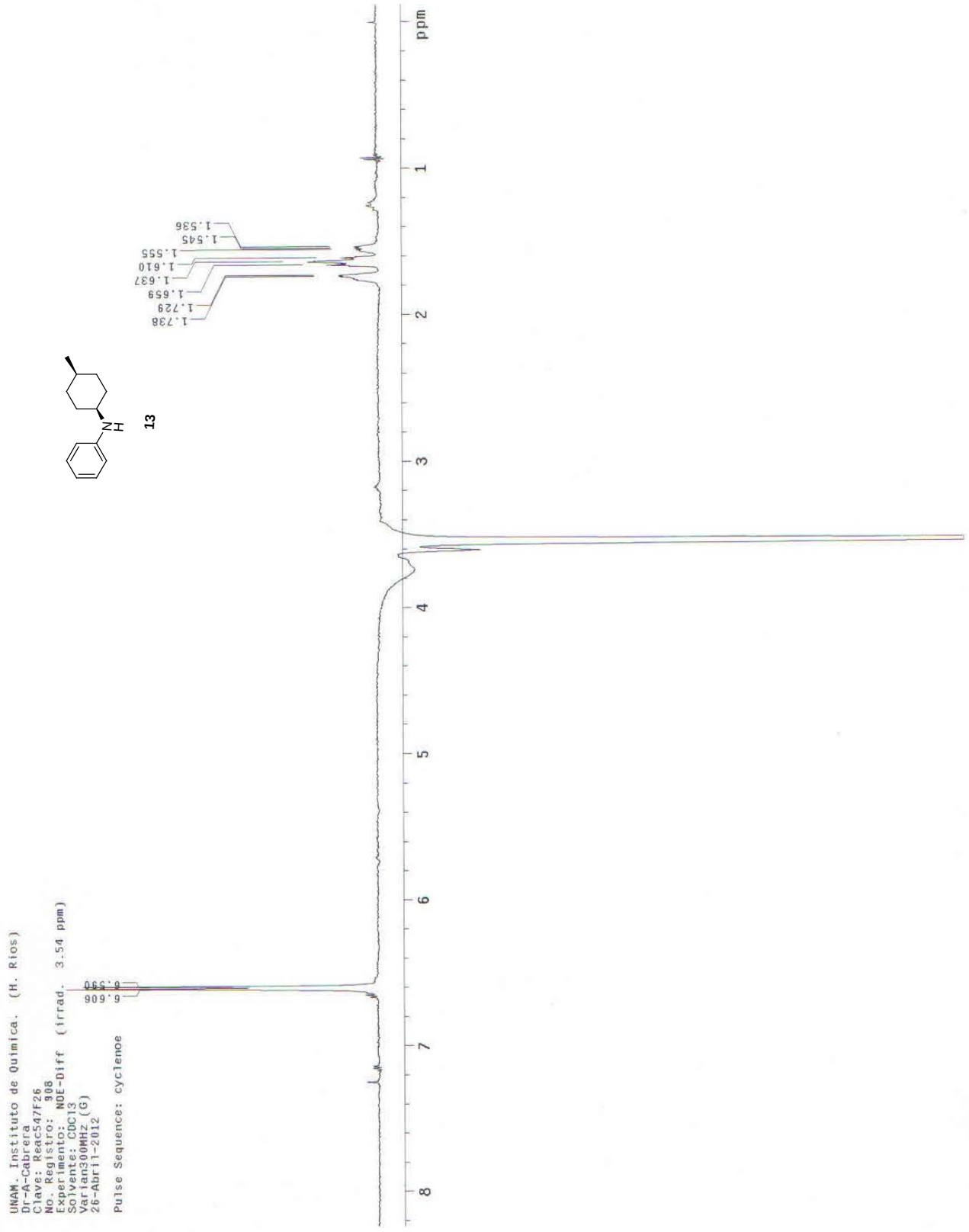
Scan: 10525 R.T.: 148.43
Base: m/z 132; 35%FS TIC: 11463152

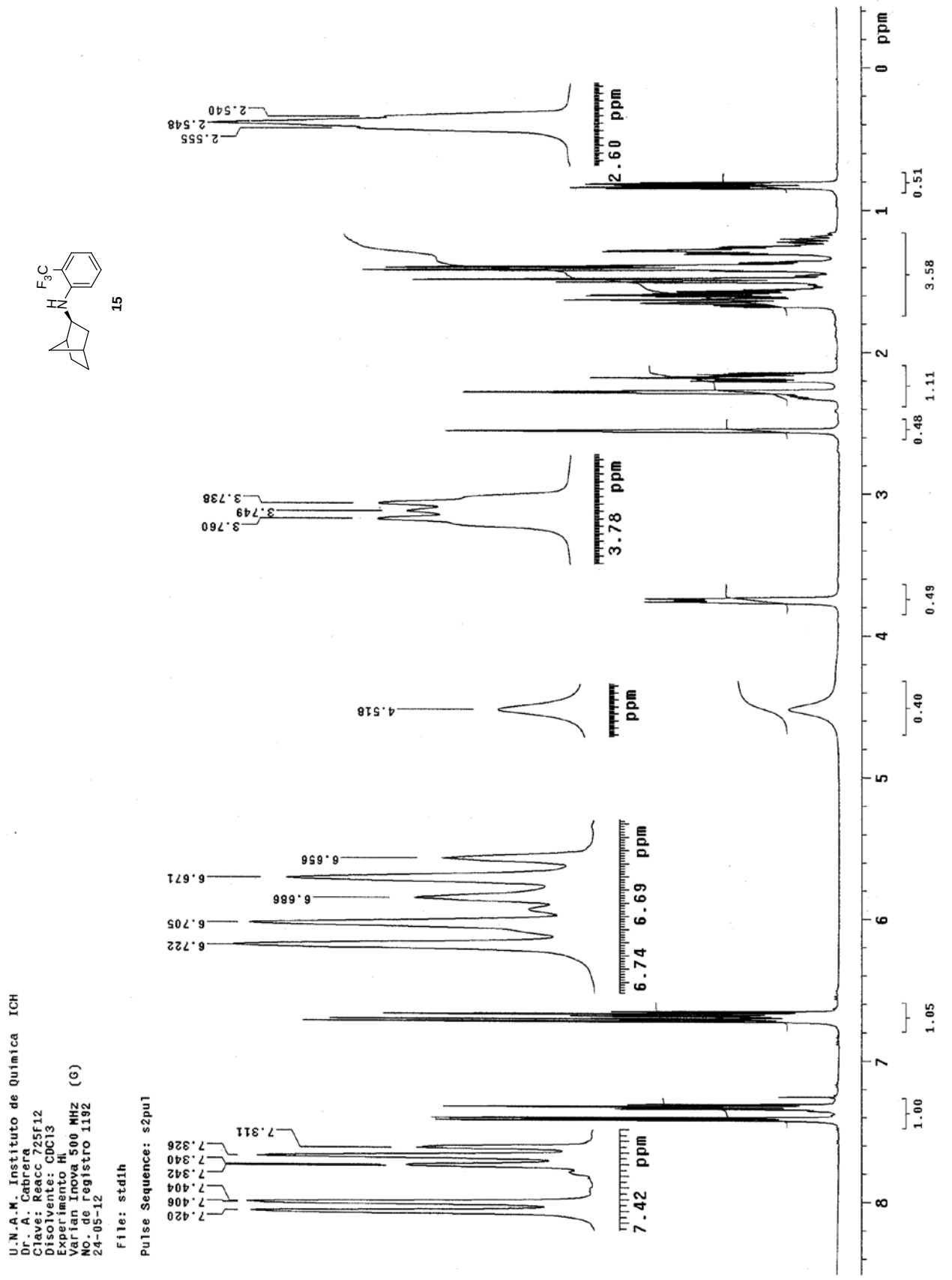


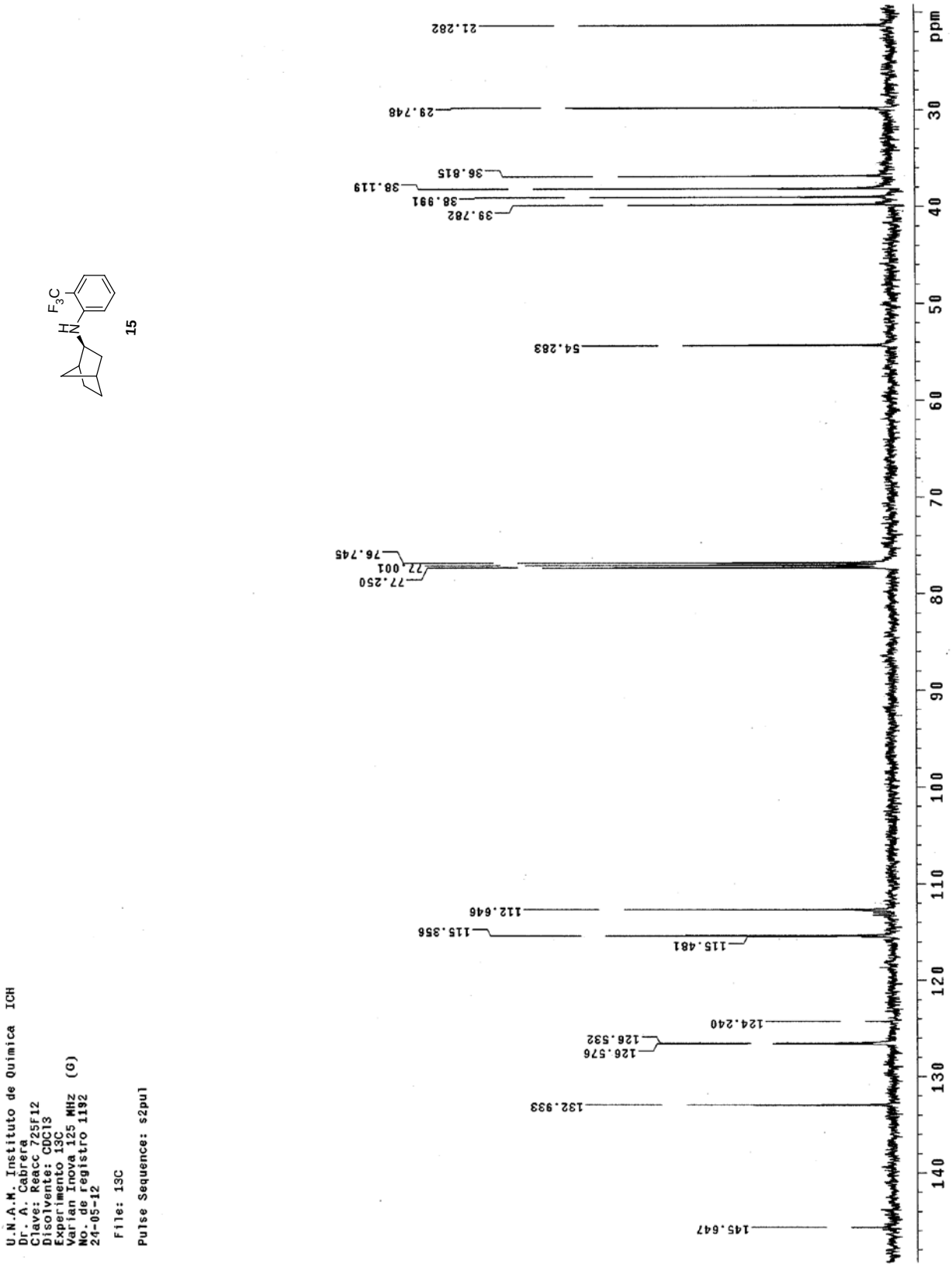

| File: REACC -700F31-2 | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Peak Table | | | | | | | | | | | | |
| Peak | Compound Name | Peak | RIC | Scan Range | Baseline at | Total Area | Background | Peak Area | | Percentage | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | Total Area |
| 147.88 | | 147.88 | TIC | 10452-10465 | 10455 | 16561208 | 15102734 | 1458474 | | 9.80298967 | | 14877849 |
| 148.4 | | 148.4 | TIC | 10518-10531 | 10518, 10532 | 141140616 | 127721241 | 13419375 | | 90.1970103 | | |
| 148.43 | | 148.43 | TIC | 0-0 | 0 | 0 | 0 | 0 | | 0 | | |
| | | | | | | | | | | | | |
| | | | | | | | | | | | | Total Percent |
| | | | | | | | | | | | | 100 |


UNAM. Instituto de Quimica. (H. Rios)
Dr-A-Cabrera
Clave: Reac547F26
No. Registro: 908
Experimento: RMN-1H
Solvente: CDCl3
Varian300MHz (G)
26-Abril-2012
File: std1h
Pulse Sequence: s2pul
13
0.926
0.939
3.731
3.605
3.561
3.552
3.543
3.535
3.526
3.80
3.70
3.60
ppm
2.00
3.09
1.90
7.45
2.73
3.40
ppm


UNAM. Instituto de Quimica (H. Rios)
Dr-ACabrera
Clave: Reac547F26
No. registro: 908
Experimento: RMN-C13
Disolvente: CDCl3
UnityInova-125.71MHz (G)
26-Abril-2012
File: Carbon
Pulse Sequence: s2pul
147.375
129.255
116.754
113.121
48.195
30.882
29.798
29.285
21.332
ppm
13

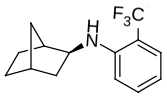

UNAM. Instituto de Quimica. (H. Rios)
Dr-A-Cabrera
Clave: Reac547F26
No. Registro: 908
Experimento: NOE-Diff (irrad. 3.54 ppm)
Solvente: CDCl3
Varian300MHz (G)
26-Abril-2012
Pulse Sequence: cyclenoe
6.606
0.390
1.738
1.729
1.659
1.637
1.610
1.555
1.545
1.536
ppm
1
2
3
4
5
6
7
8
13


U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera
Clave: Reacc 725F12
Disolvente: CDCl3
Experimento H1
Varian Inova 500 MHz (G)
No. de registro 1192
24-05-12
File: std1h
Pulse Sequence: s2pul
15
7.420
7.406
7.404
7.342
7.340
7.326
7.311
6.722
6.705
6.686
6.671
6.656
4.518
3.760
3.749
3.738
2.555
2.548
2.540
7.42 ppm
6.74 6.69 ppm
ppm
3.78 ppm
2.60 ppm
1.00
1.05
0.40
0.49
0.48
1.11
3.58
0.51
ppm

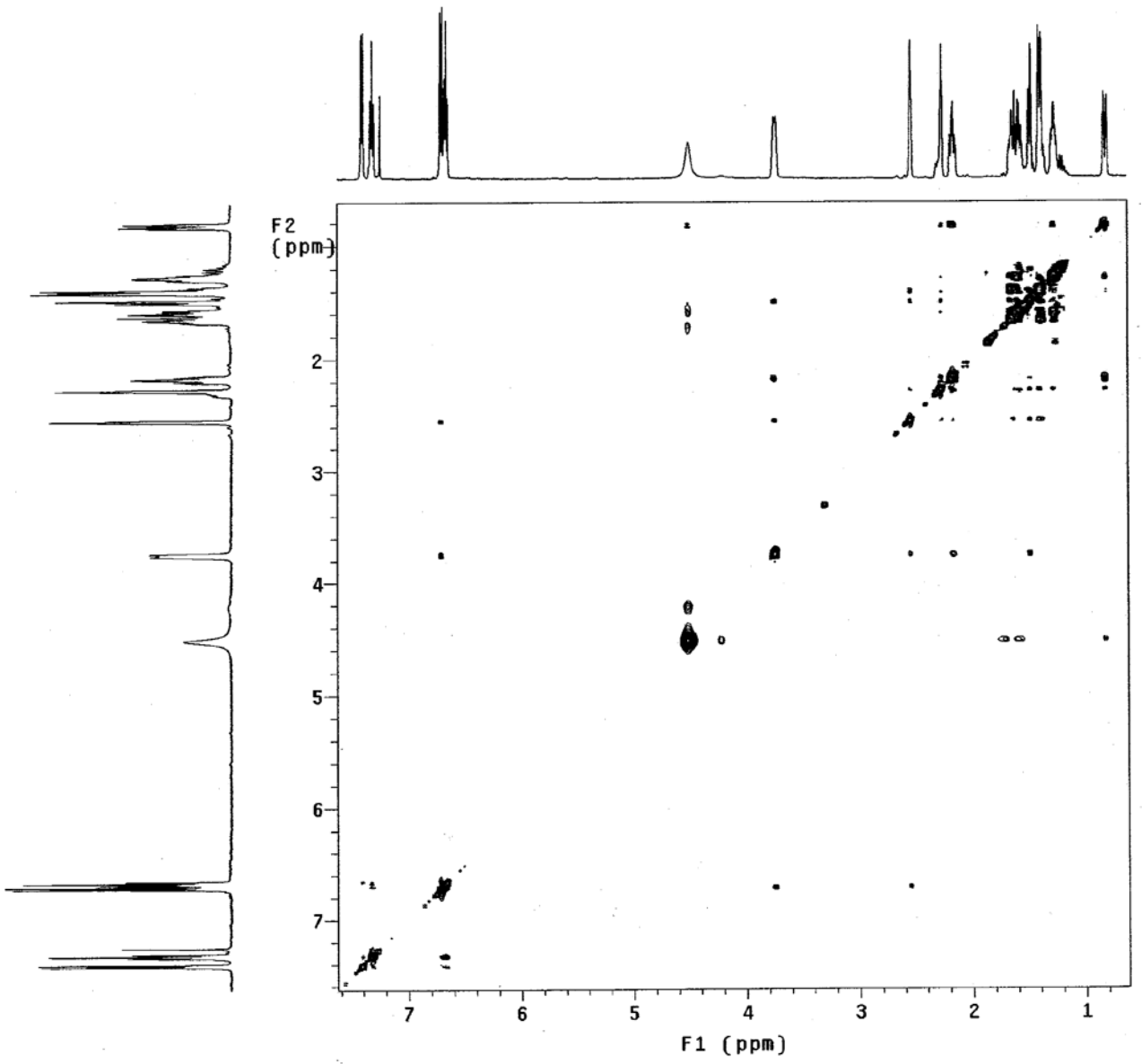

U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera
Clave: Reacc 725F12
Disolvente: CDCl3
Experimento 13C
Varian Inova 125 MHz (G)
No. de registro 1192
24-05-12
File: 13C
Pulse Sequence: s2pul
15
21.282
29.748
36.815
38.119
38.991
39.782
54.283
76.745
77.001
77.250
112.646
115.356
115.481
124.240
126.532
126.576
132.933
145.647
ppm

U.N.A.M. Instituto de Quimica ICH
Dr. A. Cabrera
Clave: Reacc 725F12
Disolvente: CDCl3
Experimento NOESY
Varian Inova 500 MHz (G)
No. de registro 1192
11-06-12

Pulse Sequence: noesy

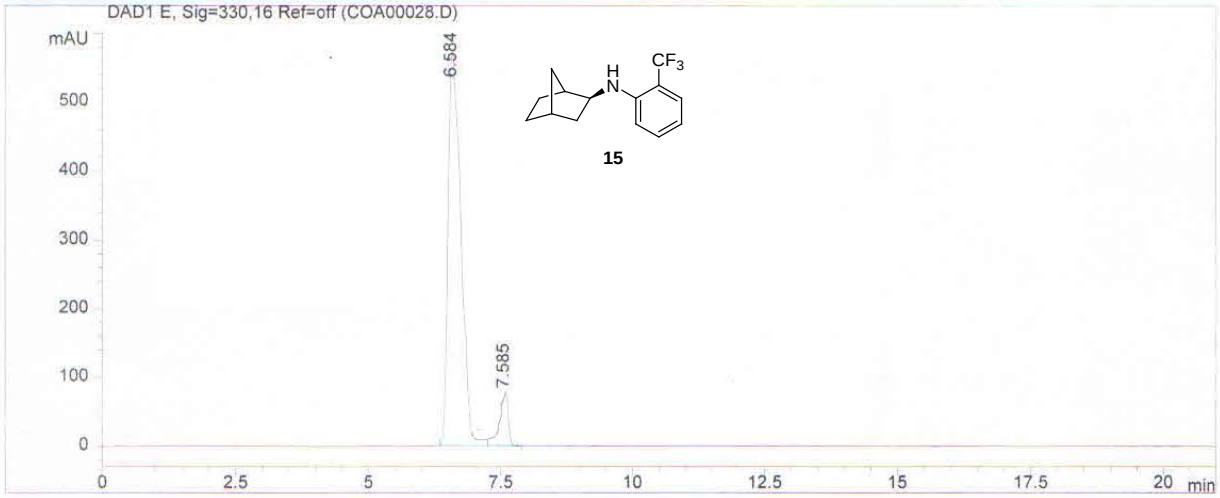



15



Data File C:\HPCHEM\1\DATA\COA00028.D Sample Name: Reacc733F9Quiral
Chiralpak IA Hex/iPrOH/ 99/1 flujo 0.5 mL/min

```
==========================================================================
Injection Date  : 01/06/12 8:38:50 PM
Sample Name     : Reacc725F9Quiral                    Vial :   1
Acq. Operator   : Erendira Garcia
Method          : C:\HPCHEM\1\METHODS\QUIRAL.M
Last changed    : 01/06/12 7:11:45 PM by Erendira Garcia
                  (modified after loading)
==========================================================================
```




**15**

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

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: DAD1 E, Sig=330,16 Ref=off
 Results obtained with enhanced integrator!

Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.584 BV    0.2941 1.23782e4   568.26147  93.2883
   2   7.585 VP    0.1931  890.56598   76.88500   6.7117

Totals :                  1.32688e4   645.14648

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

ee = 86.57 ~87%