

## Supporting Information

### Synthesis of Lactams by Radical Substitution Reaction of $\alpha,\beta$ -Unsaturated Acyl Radicals at Amine Nitrogen

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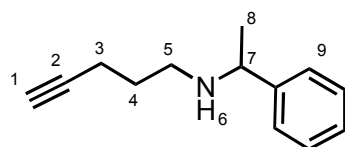
**General information.**  $^1\text{H}$  NMR spectra were recorded with a JEOL JMN-ECP500 (500 MHz) spectrometer in  $\text{CDCl}_3$ . Chemical shifts are reported in parts per million ( $\delta$ ) downfield from internal TMS at 0.00.  $^{13}\text{C}$  NMR spectra were recorded with a JEOL JMN-ECP500 (125 MHz) spectrometer and referenced to the solvent peak at 77.00 ppm. For  $^1\text{H}$ -Sn or  $^{13}\text{C}$ -Sn coupling constants, the central signals are normally associated with two close pairs of satellites corresponding to both  $^{117}\text{Sn}$  and  $^{119}\text{Sn}$  isotopes, and average values of the two different coupling constants are reported. Infrared spectra were obtained on a JASCO FT/IR-4100 spectrometer; absorptions are reported in reciprocal centimeters. Both conventional and high resolution mass spectra were recorded with a JEOL MS700 spectrometer. Products were purified by flash chromatography on silica gel (nacalai tesque inc. Silica Gel 60, 230-400 mesh). Optical rotations were obtained on JASCO DIP-370 Digital Polarimeter at a wavelength of 589 nm (sodium D line). A single crystal suitable for X-ray crystallography was sealed in glass capillary. All measurements were performed on a Rigaku RAXIS Rapid diffractometer equipped with an imaging plate detector. The frame data were processed using the Rigaku PROCESS-AUTO program,<sup>1</sup> and the reflection data were corrected for absorption with an ABSCOR program.<sup>2</sup> The structure were solved by direct method and refined on  $F^2$  by full-matrix least-squares method by using SHELX97.<sup>3</sup> Anisotropic refinement was applied to all non hydrogen atoms. Hydrogen atoms were found in the final difference Fourier map and have been isotropically refined.

**Typical procedure for stannylcarbonylation of *N*-phenylethyl-pentynylamine (**1b**).**

A magnetic stirring bar, AIBN (16.5 mg, 0.1 mmol), benzene (50 mL),  $\text{Bu}_3\text{SnH}$  (194.6 mg, 0.67 mmol), and *N*-(4-pentynyl)-*N*-(1-phenylethyl)amine (**1b**) (93.1 mg, 0.50 mmol) were placed in a 100-mL stainless autoclave. The autoclave was closed, purged three times with

carbon monoxide, pressurized with 78 atm of CO and then heated 90 °C for 4 h. Excess CO was discharged at room temperature. The solvent was removed under reduced pressure. The residue was purified by flash chromatography on silica gel (gradient from hexane to hexane/EtOAc = 1/1) to give **(Z)-2a** (141.8 mg, 71%,  $R_f$  = 0.63) and **(E)-2a** (6.7 mg, 3%,  $R_f$  = 0.075)  $R_f$  values were with hexane/EtOAc = 2/1.

#### Preparation of *N*-(4-Pentynyl)-*N*-(1-phenylethyl)amine (**1b**).

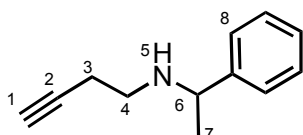


A mixture of 4-pent-1-ynyl methanesulfonate (20 mmol, 3.5 g) and (R)-(+)-1-phenylethylamine (60 mmol, 7.3 g) in acetonitrile (20mL) was stirred for 7.5 h under reflux. After cooling to

room temperature, saturated  $\text{Na}_2\text{CO}_3$  aqueous solution and AcOEt were added to the mixture. The layers were separated and the aqueous layer was extracted with AcOEt (3 x 30 mL). The combined AcOEt extracts were washed with brine. The organic layer was dried over  $\text{K}_2\text{CO}_3$ , filtered, and concentrated. The crude product was flash chromatographed on  $\text{SiO}_2$  (AcOEt) and distilled under reduce pressure (bp = 50-52 °C/0.5 mmHg) to give 2.2 g (59%) of *N*-4-pentynyl-*N*-(1-phenylethyl)amine (**1b**) as a colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.25 (bs, 1H, H-6), 1.34 (d,  $J$  = 6.9 Hz, 3H, H-8), 1.66 (quint,  $J$  = 6.9 Hz, 2H, H-4), 1.91 (t,  $J$  = 2.3 Hz, 1H, H-1), 2.14-2.30 (m, 2H, H-3), 2.46-2.55 (m, 1H, H-5), 2.57-2.66 (m, 1H, H-5), 3.76 (q,  $J$  = 6.4 Hz, 1H, H-7), 7.20-7.35 (m, 5H, H-9);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 16.44, 24.59, 29.07, 46.66, 58.34, 68.69, 84.25, 126.63, 126.94, 128.50, 145.90; IR (neat) 1369, 1450, 1492, 1602, 2116, 2840, 2863, 2960, 3026, 3061, 3300; MS (EI)  $m/z$  (rel intensity) 186 ( $\text{M}^+$ -H, 15), 172 (98), 134 (13), 105 (100), 77 (19); HRMS (EI)  $m/z$  calcd for  $\text{C}_{13}\text{H}_{18}\text{N}$  ( $\text{M}^+$ -H) 186.1283, found 186.1283.

*N*-Phenylethyl- $\omega$ -alkynylamines **1a**, **1c**, **1d**, **1e**, **1j**, **1k**, **1m**, **1n** were prepared by a similar procedure from the racemic phenylethyl amine and the corresponding methansulfonate.

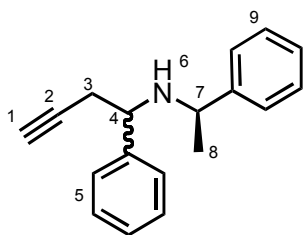
***N*-(3-Butynyl)-*N*-(1-phenylethyl)amine (**1e**).**



Colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.36 (d,  $J = 7.4$  Hz, 3H, H-7), 1.59 (bs, 1H, H-5), 1.98 (t,  $J = 2.3$  Hz, 1H, H-1), 2.27-2.40 (m, 2H, H-3), 2.56-2.70 (m, 2H, H-4), 3.80 (q,  $J =$

6.4 Hz, 1H, H-6), 7.20-7.36 (m, 5H, H-8);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 19.74, 24.62, 45.83, 57.81, 69.70, 82.68, 126.67, 126.69, 128.56, 145.56; IR (neat) 1451, 1492, 1602, 2116, 2840, 2925, 2962, 3025, 3061, 3300; MS (EI)  $m/z$  (rel intensity) 173 ( $\text{M}^+$ , 4), 158 (21), 134 (100), 129 (52), 105 (100), 77 (36); HRMS (EI)  $m/z$  calcd for  $\text{C}_{12}\text{H}_{18}\text{N}$  ( $\text{M}^+$ ) 173.1205, found 173.1196.

**Preparation of *N*-(1-Phenyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1f**, **1g**).**



Benzaldehyde (50 mmol, 5.1 mL) was cooled to 0 °C and (R)-(+)-1-phenylethylamine (50 mmol, 6.3 mL) was added dropwise. The mixture was then allowed to warm to room temperature and stirred for 1 h. Alumina Activated 200 (5 g)

was added to the mixture. This was then filtered. The alumina was washed with THF, and the filtrate was concentrated to afford benzylidene-(1-phenyl-ethyl)-amine quantitatively. Zinc powder (150 mmol, 9.8 g) was subsequently washed with 2 N HCl,  $\text{H}_2\text{O}$ , MeOH, and THF (x2). The zinc was then suspended in THF and a THF solution of benzylidene-(1-phenyl-ethyl)-amine was added. 3-Bromo-propyne was added dropwise to this mixture at 0 °C and stirred for 30 min. Then the mixture was allowed to warm to room

temperature and stirred for 12 h. Water and Et<sub>2</sub>O were added, and the reaction mixture was filtered through Celite. The aqueous phase was extracted with AcOEt (30 mL x 3) and the combined organic layers were washed with brine, dried over K<sub>2</sub>CO<sub>3</sub>, and concentrated under vacuum. The crude product was purified by vacuum distillation (bp = 118 °C/0.5 mmHg) to give 10.1g (81%) of the **1f** and **1g** as a diastereomeric mixture (dr = 51/49). To a ether solution of the diastereomeric mixture (5.5 g) was added 2 N HCl (30 mL) and the mixture was then cooled to 0 °C for 4 h. The white solid was afforded. This solid was filtered, then treated with NaOHaq. The mixture was extracted with ether and the combined organic phase was washed with water and brine, dried over K<sub>2</sub>CO<sub>3</sub>, and concentrated under vacuum to afford (S,R)- *N*-(1-phenyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1f**) in 53% yield, (93.8% de). The filtrate from the recrystallization process was treated with NaOHaq and extracted with Et<sub>2</sub>O. The organic phase was washed with water and brine and then dried over K<sub>2</sub>CO<sub>3</sub>, filtered, and evaporated under vacuum to afford (R,R)- *N*-(1-phenyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1g**) in 43% yield, (90%de). This acid/base treatment was repeated two times to afford (R,R)- *N*-(1-phenyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1g**) in 99.8%de. Diastereomeric excess was determined by HPLC. HPLC conditions; Chiracel OD-H; hexane/2-propanol 99.9/0.1; flow rate 1.0 mL/min; column temperature 20 °C; UV detector 254 nm; retention time for (R,R)- *N*-(1-phenyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1g**) 10.6 min, retention time for (S,R)- *N*-(1-phenyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1f**) 14.2 min.

**(S,R)- *N*-(1-Phenyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1f**)**<sup>5</sup>

Colorless oil. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 1.37 (d, *J* = 6.4 Hz, 3H, H-8), 1.87 (bs, 1H, H-6), 1.99 (t, *J* = 2.3 Hz, 1H, H-1), 2.57 (ddd, *J* = 8.7, 6.0, 2.3 Hz, 1H, H-3), 2.64 (ddd, *J* = 9.2, 6.4, 2.3 Hz, 1H, H-3), 3.79 (q, *J* = 6.4 Hz, 1H, H-7), 3.89 (t, *J* = 6.0 Hz, 1H, H-4), 7.20-7.40 (m,

10H, H-5,9);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 23.15, 27.09, 54.90, 58.30, 70.88, 81.57, 126.74, 127.10, 127.22, 127.47, 128.50, 128.56, 143.00, 145.85; IR (neat) 1453, 1492, 1602, 1738, 2117, 2863, 2923, 2962, 3026, 3061, 3002, 3025, 3061, 3083, 3300; MS (EI)  $m/z$  (rel intensity) 249 ( $\text{M}^+$ , 0.40), 210 (81), 128 (14), 105 (100), 77 (20); HRMS (EI)  $m/z$  calcd for  $\text{C}_{18}\text{H}_{19}\text{N}$  ( $\text{M}^+$ ) 249.1518, found 249.1516.

**(R,R)- *N*-(1-Phenyl-3-butynyl)-*N*-(1-phenylethyl)amine (1g)**

Colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.30 (d,  $J$  = 6.4 Hz, 3H, H-8), 2.02 (t,  $J$  = 2.8 Hz, 1H, H-1), 2.10 (bs, 1H, H-6), 2.41-2.49 (m, 2H, H-3), 3.51 (q,  $J$  = 6.9 Hz, 1H, H-7), 3.55 (t,  $J$  = 6.4 Hz, 1H, H-4), 7.17-7.36 (m, 10H, H-5,9);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 25.03, 28.52, 55.00, 58.30, 70.49, 81.73, 126.72, 126.95, 127.24, 127.50, 128.53(b), 142.82, 145.40. IR (neat) 1453, 1492, 1602, 1737, 2117, 2862, 2925, 2965, 3026, 3061, 3082, 3300; MS (EI)  $m/z$  (rel intensity) 249 ( $\text{M}^+$ , 0.35), 210 (93), 128 (14), 105 (100), 77 (19); HRMS (EI)  $m/z$  calcd for  $\text{C}_{18}\text{H}_{19}\text{N}$  ( $\text{M}^+$ ) 249.1518, found 249.1510.

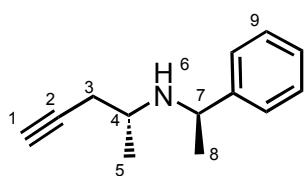
The structure of **1g** was determined as a HCl salt by X-ray analysis. To ether solution of **1g** was added 2 N HCl and filtered. The obtained white solid was recrystallized with acetonitrile.

***N*-(1-Methyl-3-butynyl)-*N*-(1-phenylethyl)amine (1h, 1i).**

A similar procedure was used for *N*-(1-methyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1h**, **1i**), in which acetaldehyde was used in place of benzaldehyde. The reaction mixture obtained was distilled under reduced pressure (bp = 65-67 °C/0.5 mmHg). Two diastereomers were separated by flash chromatography on  $\text{SiO}_2$  (Hexane/AcOEt =10/1). HPLC conditions: Chiracel OD-H; hexane/2-propanol 99.9/0.1; flow rate 0.8 mL/min; column temperature

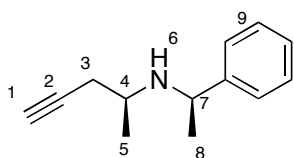
20 °C; UV detector 254 nm; retention time for (R,R)-*N*-(1-methyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1h**) 11.7 min in 99.9% dr, and retention time for retention time for (S,R)-*N*-(1-methyl-3-butynyl)-*N*-(1-phenylethyl)amine (**1i**) 13.8 min in 99.3% dr.

**(R,R)-*N*-(1-Methyl-3-butynyl)-*N*-(1-phenylethyl)amine (1h)**



Colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.10 (d,  $J = 6.4$  Hz, 3H, H-5), 1.34 (d,  $J = 6.4$  Hz, 3H, H-8), 1.48-1.58 (bs, 1H, H-6), 2.00 (t,  $J = 2.7$  Hz, 1H, H-1), 2.19 (ddd,  $J = 8.7, 6.4, 2.7$  Hz, 1H, H-3), 2.23 (ddd,  $J = 8.2, 6.0, 2.7$  Hz, 1H, H-3), 2.68 (sext,  $J = 6.4$  Hz, 1H, H-4), 3.90 (q,  $J = 6.4$  Hz, 1H, H-7), 7.20-7.35 (m, 5H, H-9);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 19.70, 24.95, 27.11, 48.68, 54.99, 70.33, 81.95, 126.62, 126.97, 128.53, 145.69; IR (neat) 1375, 1451, 1493, 1603, 2116, 2866, 2926, 2963, 3025, 3062, 3302; MS (EI)  $m/z$  (rel intensity) 186 ( $\text{M}^+ - \text{H}$ , 8), 148 (45), 105 (100), 77 (15); HRMS (EI)  $m/z$  calcd for  $\text{C}_{13}\text{H}_{16}\text{N}$  ( $\text{M}^+ - \text{H}$ ) 186.1283, found 186.1277.

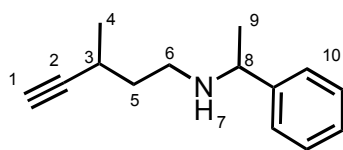
**(S,R)-*N*-(1-Methyl-3-butynyl)-*N*-(1-phenylethyl)amine (1i)**



Colorless oil.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.08 (d,  $J = 6.4$  Hz, 3H, H-5), 1.32 (d,  $J = 6.4$  Hz, 3H, H-8), 1.36-1.44 (bs, 1H, H-6), 1.99 (t,  $J = 2.8$  Hz, 1H, H-1), 2.16-2.26 (m, 1H, H-3), 2.39 (ddd,  $J = 8.7, 6.4, 2.7$  Hz, 1H, H-3), 2.65-2.74 (m, 1H, H-4), 3.89 (q,  $J = 6.5$  Hz, 1H, H-7), 7.20-7.36 (m, 5H, H-9);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 21.21, 24.93, 25.28, 48.49, 55.04, 70.48, 81.50, 126.68, 126.98, 128.55, 145.89; IR (neat) 1374, 1452, 1492, 1603, 1810, 1878, 1952, 2116, 2865, 2926, 2965, 3025, 3062, 3303; MS (EI)  $m/z$  (rel intensity) 186 ( $\text{M}^+ - \text{H}$ , 4), 148 (50), 105 (100),

77 (16); HRMS (EI)  $m/z$  calcd for  $C_{13}H_{16}N$  ( $M^+ - H$ ) 186.1283, found 186.1275. The structure of **1i** was determined as a HCl salt by X-ray analysis. To ether solution of **1i** was added 2 N HCl and filtered. The obtained white solid was recrystallized with  $CHCl_3$ .

***N*-(3-Methyl-4-pentynyl)-*N*-(1-phenylethyl)amine (**1j**)**<sup>5</sup>

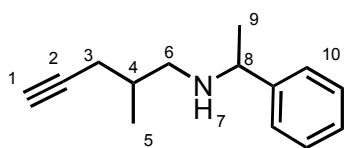


Colorless oil. bp = 60-64 °C/0.5 mmHg. dr = 50/50:  $^1H$  NMR

(500 MHz,  $CDCl_3$ )  $\delta$  1.14 (d,  $J$  = 6.9 Hz, 3H, H-4), 1.16 (d,  $J$  = 6.9 Hz, 3H, H-4), 1.24 (bs, 2H, H-7), 1.34 (d,  $J$  = 6.4 Hz,

6H, H-9), 1.58 (q,  $J$  = 7.3 Hz, 4H, H-5), 1.99 (t,  $J$  = 1.8 Hz, 1H, H-1), 2.00 (t,  $J$  = 1.8 Hz, 1H, H-1), 2.44-2.70 (m, 6H, H-3,6), 3.76 (q,  $J$  = 6.9 Hz, 2H, H-8), 7.20-7.35 (m, 10H, H-10);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ ) 21.01, 21.25, 23.71, 23.82, 24.50, 24.57, 37.16, 37.22, 45.53, 45.70, 58.42 (b), 68.54, 68.65, 88.63, 88.82, 126.63 (b), 126.90 (b), 128.46 (b), 145.86, 145.98, IR (neat) 1372, 1452, 1493, 1603, 1810, 1949, 2111, 2967, 3026, 3061, 3303; MS (EI)  $m/z$  (rel intensity) 200 ( $M^+ - H$ , 16), 186 (98), 158 (9), 105 (100), 97 (16), 77 (19); HRMS (EI)  $m/z$  calcd for  $C_{14}H_{18}N$  ( $M^+ - H$ ) 200.1440, found 200.1441.

***N*-(2-Methyl-4-pentynyl)-*N*-(1-phenylethyl)amine (**1k**)**<sup>5</sup>



Colorless oil. bp = 107-110 °C/3 mmHg. dr = 51/49:  $^1H$  NMR

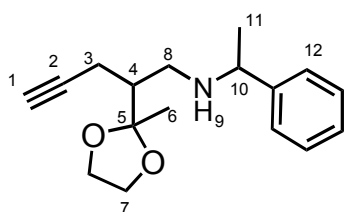
(500 MHz,  $CDCl_3$ )  $\delta$  0.97 (d,  $J$  = 6.5 Hz, 3H, H-5), 0.98 (d,  $J$  = 6.9 Hz, 3H, H-5), 1.24 (bs, 2H, H-7), 1.33 (d,  $J$  = 6.4 Hz, 3H,

H-9), 1.34 (d,  $J$  = 6.5 Hz, 3H, H-9), 1.73 (m, 2H, H-4), 1.92 (t,  $J$  = 2.8 Hz, 1H, H-1), 1.93 (t,  $J$  = 2.8 Hz, 1H, H-1), 2.06-2.56 (m, 8H, H-3,6), 3.735 (q,  $J$  = 6.4 Hz, 1H, H-8), 3.7340 (q,  $J$  = 6.4 Hz, 1H, H-8), 7.20-7.35 (m, 10H, H-10);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ ) 17.92, 17.94, 23.71, 23.91, 24.69, 24.72, 58.37, 58.52, 69.42 (b), 83.13, 83.16, 126.67 (b), 126.88 (b), 128.46 (b), 146.04, 146.14, IR (neat) 1452, 1493, 1541, 1602, 1646, 1947, 2116, 2310, 2348, 2370,



2832, 2925, 2960, 3026, 3061, 3083, 3304; MS (EI)  $m/z$  (rel intensity) 200 ( $M^+-H$ , 22), 186 (100), 105 (35), 77 (10); HRMS (EI)  $m/z$  calcd for  $C_{14}H_{18}N$  ( $M^+-H$ ) 200.1440, found 200.1431.

**{2-(2-Methyl-[1,3]dioxolan-2-yl)-4-pentynyl}-(1-phenylethyl)amine (1I) <sup>5</sup>**

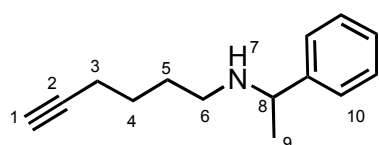


2-(2-Methyl-[1,3]dioxolan-2-yl)-pent-4-ynal was prepared from ethyl-3-oxobutanoate in 4 steps by propargylation of ethyl 3-oxobutanoate, acetal protection of ketone, reduction with lithium aluminum hydride, and oxidation with PCC in 35%

over four steps. The obtained aldehyde (7.7 mmol) was treated with (R)-(+)-1-phenylethylamine (7.7 mmol) at 0 °C for 10 min and then stirred for 1 h at room temperature to give the corresponding imine. After the addition of  $Et_2O$  and water to the mixture, the aqueous layer was extracted with  $Et_2O$  (30 mL x 3), the combined organic layer was washed with brine, dried over  $Na_2SO_4$ , filtered, and concentrated under reduced pressure. To a MeOH (10 mL) solution of the imine was added  $NaBH_4$  (23.1 mmol) portionwise at 0 °C and stirred for 12 h at room temperature. Water and  $Et_2O$  were added to the mixture. The layers were separated and the aqueous layer was extracted with  $Et_2O$  (3 x 30 mL). The combined  $Et_2O$  extracts were washed with brine. The organic layer was dried over  $K_2CO_3$ , filtered, and concentrated. The crude product was flash chromatographed on  $SiO_2$  (hexane/ $AcOEt$  = 1/1) and distilled under reduce pressure (bp = 103-105 °C/0.5 mmHg) to give 1.3 g (62%) of the desired amine (1I) as a colorless oil. dr = 50/50:  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  1.19 (s, 3H, H-6), 1.21 (s, 3H, H-6), 1.339 (d,  $J$  = 6.9 Hz, 3H, H-11), 1.344 (d,  $J$  = 6.9 Hz, 3H, H-11), 1.86-2.06 (m, 6H, H-1,4,9), 2.12-2.30 (m, 2H, H-3 or 8), 2.42-2.53 (m, 2H, H-3 or 8), 2.56-2.76 (m, 4H, H-3 or 8), 3.65-3.80 (m, 2H, H-10), 3.84-4.00

(m, 8H, H-7), 7.17-7.50 (m, 10H, H-12);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 18.09, 18.28, 20.97, 21.16, 24.57, 24.63, 45.64, 45.88, 47.71, 47.81, 58.45, 58.56, 64.52, 64.64, 69.34(b), 83.37, 83.50, 111.16, 111.32, 126.66(b), 126.71(b), 126.78(b), 128.38(b), 145.93, 146.07, IR (neat) 1375, 1450, 1493, 1603, 2116, 2883, 2924, 2979, 3025, 3060, 3294; MS (EI)  $m/z$  (rel intensity) 272 ( $\text{M}^+ - \text{H}$ , 20), 258 (58), 228 (28), 186 (28), 134 (23), 118 (38), 105 (100), 77 (17); HRMS (EI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{22}\text{N}_1\text{O}_2$  ( $\text{M}^+ - \text{H}$ ) 272.1650, found 272.1653.

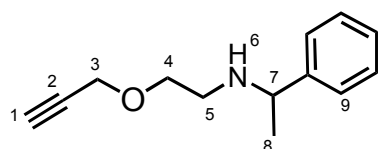
***N*-(5-Hexynyl)-*N*-(1-phenylethyl)amine (1m).**



Colorless oil. bp = 62-63 °C/0.5 mmHg.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.18 (bs, 1H, H-7), 1.34 (d,  $J$  = 6.4 Hz, 3H, H-9), 1.47-1.62 (m, 4H, H-4,5), 1.92 (t,  $J$  = 2.3 Hz, 1H, H-1),

2.10-2.22 (m, 2H, H-3), 2.38-2.46 (m, 1H, H-6), 2.47-2.55 (m, 1H, H-6), 3.74 (q,  $J$  = 6.9 Hz, 1H, H-8), 7.20-7.35 (m, 5H, H-10);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 18.41, 24.59, 26.35, 29.47, 47.32, 58.44, 68.55, 84.43, 126.60, 126.99, 128.47, 145.96, IR (neat) 1369, 1451, 1493, 1603, 2116, 2862, 2935, 3025, 3061, 3303; MS (EI)  $m/z$  (rel intensity) 200 ( $\text{M}^+ - \text{H}$ , 5), 186 (55), 148 (12), 134 (11), 105 (100), 79 (16), 77 (15); HRMS (EI)  $m/z$  calcd for  $\text{C}_{14}\text{H}_{18}\text{N}$  ( $\text{M}^+ - \text{H}$ ) 200.1440, found 200.1436.

***N*-(3-Butyloxyethyl)-*N*-(1-phenylethyl)amine (1n).**

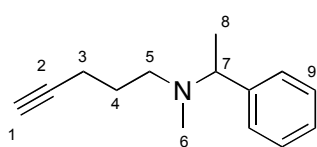


Colorless oil. bp = 78-80 °C/0.5 mmHg.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.35 (d,  $J$  = 6.9 Hz, 3H, H-8), 1.70 (bs, 1H, H-6), 2.41 (t,  $J$  = 2.3 Hz, 1H, H-1), 2.62 (ddd,  $J$  = 10.1, 6.4, 3.7

Hz, 1H, H-5), 2.70 (ddd,  $J$  = 10.5, 6.4, 3.7 Hz, 1H, H-5), 3.56 (ddd,  $J$  = 9.6, 6.9, 3.7 Hz, 1H, H-4), 3.62 (ddd,  $J$  = 9.2, 6.4, 3.7 Hz, 1H, H-4), 3.77 (q,  $J$  = 6.4 Hz, 1H, H-7), 4.12 (dd,  $J$  =

15.6, 2.3 Hz, 1H, H-3), 4.16 (dd,  $J$  = 15.6, 2.3 Hz, 1H, H-3), 7.20-7.34 (m, 5H, H-9);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 24.55, 47.15, 58.30, 69.61, 74.59, 74.63, 79.86, 126.71, 126.97, 128.51, 145.62; IR (neat) 1351, 1451, 1493, 1603, 2116, 2862, 2926, 3026, 3061, 3290; MS (EI)  $m/z$  (rel intensity) 203 ( $\text{M}^+$ , 7), 188 (37), 149 (16), 134 (100), 105 (100), 77 (21); HRMS (EI)  $m/z$  calcd for  $\text{C}_{13}\text{H}_{16}\text{N}$  ( $\text{M}^+$ ) 203.1310, found 203.1302.

***N*-Methyl-*N*-(1-phenylethyl)-4-pentynyl-1-amine (**1o**).**



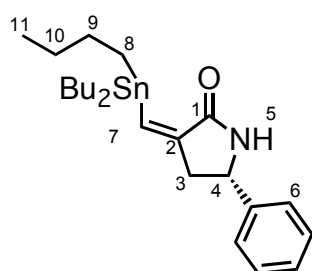
Colorless oil:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  1.35 (d,  $J$  = 6.9 Hz, 3H, H-8), 1.67 (quint,  $J$  = 7.3 Hz, 2H, H-4), 1.90 (t,  $J$  = 2.3 Hz, 1H, H-1), 2.11-2.25 (m, 5H, H-3, 6), 2.36 (dt,  $J$  = 12.8, 6.9 Hz, 1H, H-5), 2.49 (dt,  $J$  = 12.4, 7.4 Hz, 1H, H-5), 3.56 (q,  $J$  = 6.9 Hz, 1H, H-7), 7.20-7.35 (m, 5H, H-9);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ) 16.28, 18.36, 26.44, 38.41, 53.04, 63.49, 68.25, 84.65, 126.76, 127.75, 128.17, 144.12; IR (neat) 1452, 1492, 1602, 2117, 2791, 2841, 2972, 3027, 3060, 3305; MS (EI)  $m/z$  (rel intensity) 200 ( $\text{M}^+ - \text{H}$ , 6), 186 (74), 172 (25), 148 (20), 105 (100), 77 (15); HRMS (EI)  $m/z$  calcd for  $\text{C}_{14}\text{H}_{18}\text{N}$  ( $\text{M}^+ - \text{H}$ ) 200.1440, found 200.1436.

**Typical procedure for stannylcarbonylation of *N*-phenylethyl-pentynylamine (**1b**).**

A magnetic stirring bar, AIBN (16.5 mg, 0.1 mmol), benzene (50 mL),  $\text{Bu}_3\text{SnH}$  (194.6 mg, 0.67 mmol), and *N*-(4-pentynyl)-*N*-(1-phenylethyl)amine (**1b**) (93.1 mg, 0.50 mmol) were placed in a 100-mL stainless autoclave. The autoclave was closed, purged three times with carbon monoxide, pressurized with 78 atm of CO and then heated 90 °C for 4 h. Excess CO was discharged at room temperature. The solvent was removed under reduced pressure. The residue was purified by flash chromatography on silica gel (gradient from hexane to hexane/EtOAc = 1/1) to give (**Z**)-**2a** (141.8 mg, 71%,  $R_f$  = 0.63) and (**E**)-**2a** (6.7 mg, 3%,  $R_f$  =

0.075)  $R_f$  values were with hexane/EtOAc = 2/1. The spectral data of these compounds, as well as those of 3-[(tributylstannanyl)methylene]pyrrolidin-2-one (**2e**) and 3-[(tributylstannanyl)methylene]azepan-2-one (**2l**), were identical with the data we previously reported.<sup>4</sup>

**(S)-5-Phenyl-3-[(tributylstannanyl)methylene]-2-pyrrolidinone (2f).**



*E* isomer: ( $R_f$  = 0.55, hexane/EtOAc = 1/1), colorless oil.  $^1\text{H}$

NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  0.75-1.00 (m, 15H, H-8, 11), 1.20-1.34

(m, 6H, H-10), 1.38-1.52 (m, 6H, H-9), 2.58-2.68 (m, 1H, H-3),

3.20-3.30 (m, 1H, H-3), 4.72 (q,  $J$  = 3.7 Hz 1H, H-4), 5.96 (bs, 1H,

H-5), 7.20 (s, H-7), 7.23-7.40 (m, 5H, H-6);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  9.69 (C-8), 13.60

(C-11), 27.22 (C-10), 29.07 (C-9), 39.20 (C-3), 54.32 (C-4), 125.78 (C-6), 127.98 (C-6),

128.94 (C-6), 134.84 (C-7), 142.9 (C-6), 146.23 (C-2), 169.12 (C-1); IR (neat) 1685, 1623,

EIMS,  $m/z$  (rel intensity) 406 ( $\text{M}^+$ - $\text{C}_4\text{H}_9$ , 100), 292 (59); HRMS calcd for  $\text{C}_{19}\text{H}_{28}\text{NOSn}$

( $\text{M}^+$ - $\text{C}_4\text{H}_9$ ) 406.1193, found 406.1195.

*Z* isomer: ( $R_f$  = 0.80, hexane/EtOAc = 1/1), colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$

0.78-0.96 (m, 15H, H-8, 11), 1.18-1.34 (m, 6H, H-10), 1.34-1.54 (m, 6H, H-9), 2.70 (ddd,  $J$  =

17.0, 4.6, 2.3 Hz, 1H, H-3), 3.32 (ddd,  $J$  = 17.0, 4.6, 2.3 Hz, 1H, H-3), 4.74 (q,  $J$  = 3.7 Hz, 1H,

H-4), 6.56 (s,  $J$   $^1\text{H}$ -Sn = 60.5 Hz, 1H, H-7), 7.22-7.40 (m, 5H, H-6), 8.00 (bs, 1H, H-5);  $^{13}\text{C}$

NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  11.49 ( $J$   $^{13}\text{C}$ -Sn = 354.1 Hz, C-8), 13.75 (C-11), 27.31 ( $J$   $^{13}\text{C}$ -Sn =

56.6 Hz, C-10), 29.20 ( $J$   $^{13}\text{C}$ -Sn = 20.2 Hz, C-9), 39.52 (C-3), 54.53 (C-4), 125.63 (C-2 or 6

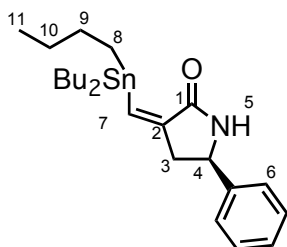
or 7), 127.62 (C-2 or 6 or 7), 128.74 (C-2 or 6 or 7), 138.56 (C-2 or 6 or 7), 143.16 (C-2 or 6

or 7), 144.31 (C-2 or 6 or 7), 171.96 (C-1); IR (neat) 1689, 1621; EIMS,  $m/z$  (rel intensity)

406 ( $\text{M}^+$ - $\text{C}_4\text{H}_9$ , 100), 292 (59); HRMS calcd for  $\text{C}_{19}\text{H}_{28}\text{NOSn}$  ( $\text{M}^+$ - $\text{C}_4\text{H}_9$ ) 406.1193, found

406.1195.

**(R)-5-Phenyl-3-[(tributylstannanyl)methylene]-2-pyrrolidinone (2g).**

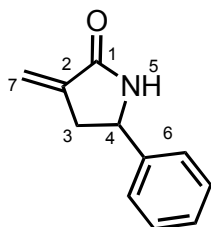


*E* isomer: colorless oil.  $^1\text{H}$ -NMR,  $^{13}\text{C}$ -NMR, IR and EIMS were identical with those of (S)-5-phenyl-3-[(tributylstannanyl)methylene]-2-pyrrolidinone (**2f**); HRMS calcd for  $\text{C}_{19}\text{H}_{28}\text{NOSn}$  ( $\text{M}^+ - \text{C}_4\text{H}_9$ ) 406.1193, found

406.1196.

*Z* isomer: colorless oil.  $^1\text{H}$ -NMR,  $^{13}\text{C}$ -NMR, IR and EIMS were identical with those of (S)-5-phenyl-3-[(tributylstannanyl)methylene]-2-pyrrolidinone (**2f**); HRMS calcd for  $\text{C}_{19}\text{H}_{28}\text{NOSn}$  ( $\text{M}^+ - \text{C}_4\text{H}_9$ ) 406.1193, found 406.1197.

**3-Methylene-5-phenyl-2-pyrrolidine (3g)**

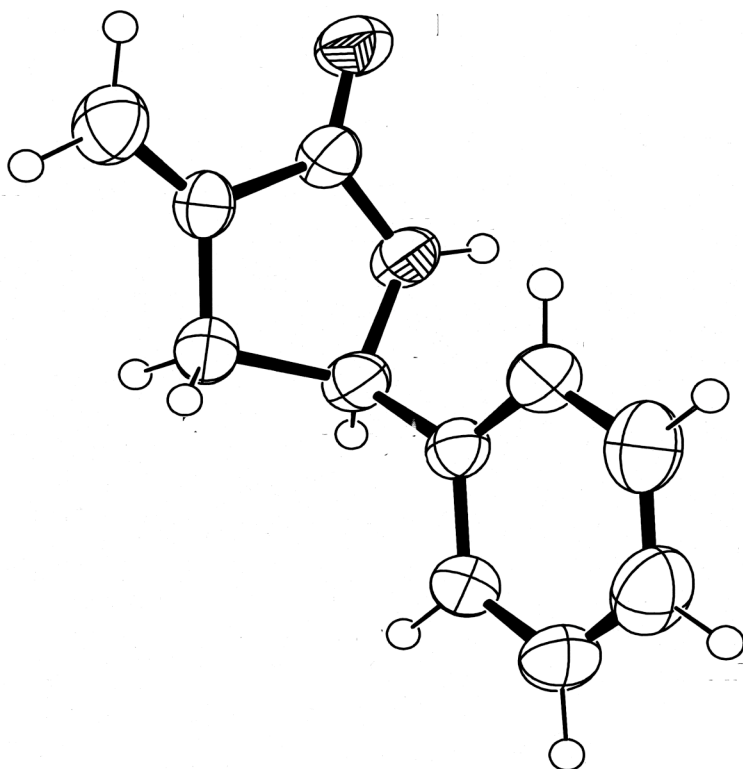


Protodestannylation of **2g** leading to 3-methylene-5-phenyl-2-pyrrolidine (**3g**) was carried out. The optical yield was estimated by HPLC analysis using a chiral column.

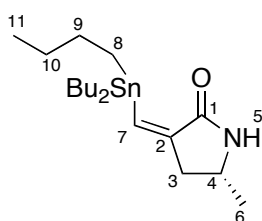
( $R_f$  = 0.15, hexane/EtOAc = 1/1), white solid.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  2.60-2.69 (m, 1H, H-3), 3.22-3.34 (m, 1H, H-3), 4.73 (m, 1H, H-4), 5.34 (s, 1H, H-7), 6.00 (s, 1H, H-7), 7.22-7.38 (m, 5H, H-6), 7.49 (bs, 1H, H-5);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  36.71 (C-3), 54.78 (C-4), 116.32 (C-2 or 6 or 7), 125.58 (C-2 or 6 or 7), 127.82 (C-2 or 6 or 7), 128.83 (C-2 or 6 or 7), 138.87 (C-2 or 6 or 7), 142.67 (C-2 or 6 or 7), 171.01 (C-1); IR (KBr) 1656, 1591; EIMS,  $m/z$  (rel intensity) 173 ( $\text{M}^+$ , 100), 144 (27), 104 (37); HRMS calcd for  $\text{C}_{11}\text{H}_{11}\text{NO}$  ( $\text{M}^+$ ) 173.0840, found 173.0843; HPLC conditions; Chiralcel OD-H; hexane/2-propanol 95/5; flow rate 1.0 mL/min; column temperature 20 °C; UV detector 254 nm; retention time for

racemate 26.5, 31.4 min, retention time for (R)-isomer (**3g**) 26.5 min in 95% ee, retention time for (S)-isomer (**3f**) 31.4 min in 98% ee. m.p. 169-170 °C,  $[\alpha]_D^{18}$  -10.0 (c 0.88, CHCl<sub>3</sub>). It should be noted that R isomer has been already known.<sup>6</sup> However, there is discrepancy in spectral and physical data between their data and our data. This led us to examine X-ray analysis of **3g**, which well supported the structure of **3g**. Crystallographic data: C<sub>11</sub>H<sub>11</sub>N<sub>1</sub>O<sub>1</sub>; *M* = 173.21, orthorhombic, space group *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 8.179(4) Å, *b* = 9.368(5) Å, *c* = 12.062(7) Å,  $\alpha = 90^\circ$ ,  $\beta = 90^\circ$ ,  $\gamma = 90^\circ$ , *V* = 924.2(7) Å<sup>3</sup>, *Z* = 4, *D*<sub>calcd</sub> = 1.245 g/cm<sup>3</sup>, *T* = 296 K,  $\mu(\text{Mo K}\alpha) = 0.080 \text{ mm}^{-1}$ , 9103 reflections measured, 2118 unique (*R*<sub>int</sub> = 0.0299), *R*<sub>1</sub> = 0.0342, *wR*<sub>2</sub> = 0.0900, GOF = 1.023.

ORTEP drawing of **3g**.



**(R)-5-Methyl-3-[(tributylstannanyl)methylene]-2-pyrrolidinone (2h).**



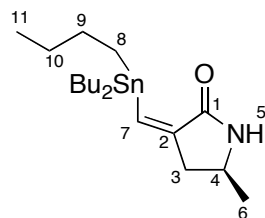
*E* isomer: (*R*<sub>f</sub> = 0.13, hexane/EtOAc = 2/1), colorless oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz)  $\delta$  0.80-1.08 (m, 15H, H-8, 11), 1.24 (d, *J* = 6.4 Hz,

3H, H-6), 1.25-1.40 (m, 6H, H-10), 1.41-1.60 (m, 6H, H-9), 2.32 (ddd,  $J = 16.5, 4.1, 2.7$  Hz, 1H, H-3), 2.95 (ddd,  $J = 16.5, 7.3, 2.3$  Hz, 1H, H-3), 3.72-3.84 (m, 1H, H-4), 6.82 (bs, 1H, H-5), 7.09 (s,  $J^1\text{H-Sn} = 61.9$  Hz, H-7);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  9.78 ( $J^{13}\text{C-Sn} = 361.8$  Hz, C-8), 13.73 (C-11), 23.21 (C-6), 27.36 ( $J^{13}\text{C-Sn} = 57.6$  Hz, C-10), 29.20 ( $J^{13}\text{C-Sn} = 20.2$  Hz, C-9), 37.21 (C-3), 46.33 (C-4), 133.71 (C-7), 147.34 (C-2), 168.92 (C-1); IR (neat) 1691, 1622, EIMS,  $m/z$  (rel intensity) 344 ( $\text{M}^+ - \text{C}_4\text{H}_9$ , 100), 230 (53), 148 (9); 105 (10); HRMS calcd for  $\text{C}_{14}\text{H}_{26}\text{ONSn}$  ( $\text{M}^+ - \text{C}_4\text{H}_9$ ) 344.1037, found 344.1034.

Z isomer: ( $R_f = 0.31$ , hexane/EtOAc = 2/1), colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  0.82-0.99 (m, 15H, H-8, 11), 1.23 (d,  $J = 6.4$  Hz, 3H, H-6), 1.24-1.33 (m, 6H, H-10), 1.39-1.57 (m, 6H, H-9), 2.33 (ddd,  $J = 17.0, 4.1, 2.3$  Hz, 1H, H-3), 3.00 (ddd,  $J = 17.0, 7.8, 2.3$  Hz, 1H, H-3), 3.68-3.80 (m, 1H, H-4), 6.47 (s,  $J^1\text{H-Sn} = 61.9$  Hz, H-7), 7.64 (bs, 1H, H-5);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  11.54 (C-8), 13.74 (C-11), 23.02 (C-6), 27.38 (C-10), 29.27 (C-9), 37.64 (C-3), 46.52 (C-4), 137.22 (C-7), 145.46 (C-2), 171.52 (C-1); IR (neat) 1693, 1624, EIMS,  $m/z$  (rel intensity) 344 ( $\text{M}^+ - \text{C}_4\text{H}_9$ , 29), 288 (14), 230 (44), 149 (17); HRMS calcd for  $\text{C}_{14}\text{H}_{26}\text{ONSn}$  ( $\text{M}^+ - \text{C}_4\text{H}_9$ ) 344.1037, found 344.1038.

**(S)-5-Methyl-3-[(tributylstannanyl)methylene]-2-pyrrolidinone (2i).**

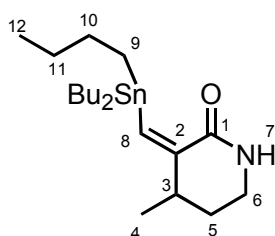
*E* isomer: colorless oil.  $^1\text{H}$ -NMR,  $^{13}\text{C}$ -NMR, IR and EIMS were identical with those of (R)-5-methyl-3-[(tributylstannanyl)methylene]-2-pyrrolidinone (**2h**); HRMS calcd for  $\text{C}_{14}\text{H}_{26}\text{ONSn}$  ( $\text{M}^+ - \text{C}_4\text{H}_9$ ) 344.1037, found 344.1037.



Z isomer: colorless oil.  $^1\text{H}$ -NMR,  $^{13}\text{C}$ -NMR, IR and EIMS were identical with those of (R)-5-methyl-3-[(tributylstannanyl)methylene]-2-pyrrolidinone (**2h**); HRMS calcd for

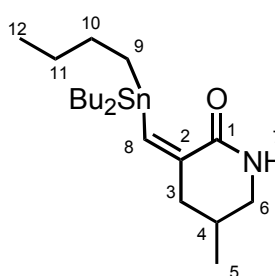
C<sub>14</sub>H<sub>26</sub>ONSn (M<sup>+</sup>-C<sub>4</sub>H<sub>9</sub>) 344.1037, found 344.1037.

**4-Methyl-3-[t(ributylstannanyl)methylene]-2-piperidinone (2j).**



(R<sub>f</sub> = 0.75, hexane/EtOAc = 2/1), colorless oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 0.76-0.94 (m, 15H, H-9,12), 1.18 (d, *J* = 6.9 Hz, 3H, H-4), 1.22-1.33 (m, 6H, H-11), 1.40-1.51 (m, 6H, H-10), 1.56-1.64 (m, 1H, H-5), 1.87-1.95 (m, 1H, H-5), 2.61-2.71 (m, 1H, H-3), 3.30-3.44 (m, 1H, H-6), 6.52 (s, *J* <sup>1</sup>H-Sn = 70.1 Hz, 1H, H-8), 7.65 (bs, 1H, H-7); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 12.10 (*J* <sup>13</sup>C-Sn = 359.9 Hz, C-9), 13.90 (C-12), 19.71 (C-4), 27.56 (*J* <sup>13</sup>C-Sn = 31.7 Hz, C-11), 29.43 (C-10), 30.72 (C-3or5), 35.87 (C-3or5), 40.05 (C-6), 143.84 (C-8), 148.37 (C-2), 167.15 (C-1); IR (neat) 1661, 1582; EIMS, *m/z* (rel intensity) 358 (M<sup>+</sup>- C<sub>4</sub>H<sub>9</sub>, 65), 269 (44), 203 (22); HRMS calcd for C<sub>15</sub>H<sub>28</sub>NOSn (M<sup>+</sup>-C<sub>4</sub>H<sub>9</sub>) 358.1192, found 358.1194.

**5-Methyl-3-[(tributylstannanyl)methylene]-2-piperidinone (2k).**



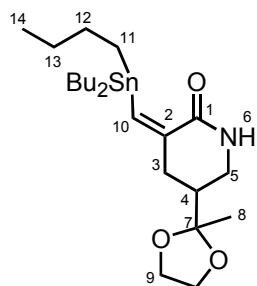
*E* isomer: (R<sub>f</sub> = 0.10, hexane/EtOAc = 1/1), colorless oil. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz) δ 0.82-0.98 (m, 15H, H-9,12), 1.02 (d, *J* = 6.5 Hz, 3H, H-5), 1.22-1.36 (m, 6H, H-11), 1.40-1.56 (m, 6H, H-10), 2.02-2.14 (m, 1H, H-3), 2.22-2.34 (m, 1H, H-3), 2.50-2.62 (m, 1H, H-4), 3.00-3.10 (m, 1H, H-6), 3.30-3.40 (m, 1H, H-6), 5.93 (bs, 1H, H-7), 7.44 (s, *J* <sup>1</sup>H-Sn = 60.5 Hz, 1H, H-8); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz) δ 10.20 (C-9), 13.73 (C-12), 18.38 (C-5), 27.38 (C-11), 29.23 (C-10), 29.67 (C-4), 40.52 (C-3), 49.46 (C-6), 141.83 (C-8), 143.56 (C-2), 164.72 (C-1); IR (neat) 1661, 1588; EIMS, *m/z* (rel intensity) 358 (M<sup>+</sup>- C<sub>4</sub>H<sub>9</sub>, 30), 244 (14); HRMS calcd for C<sub>15</sub>H<sub>28</sub>NOSn



( $M^+ - C_4H_9$ ) 358.1193, found 358.1192.

Z isomer: ( $R_f$  = 0.55, hexane/EtOAc = 1/1), colorless oil.  $^1H$  NMR ( $CDCl_3$ , 500 MHz)  $\delta$  0.76-0.93 (m, 15H, H-9,12), 0.99 (d,  $J$  = 6.9 Hz, 3H, H-5), 1.21-1.34 (m, 6H, H-11), 1.38-1.54 (m, 6H, H-10), 1.98-2.12 (m, 1H, H-3), 2.26-2.38 (m, 1H, H-3), 2.63-2.74 (m, 1H, H-4), 2.95-3.06 (m, 1H, H-6), 3.26-3.37 (m, 1H, H-6), 6.45 (s,  $J$   $^1H-Sn$  = 70.1 Hz, 1H, H-8), 7.50 (bs, 1H, H-7);  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz)  $\delta$  11.98 ( $J$   $^{13}C-Sn$  = 352.2 Hz, C-9), 13.88 (C-12), 18.22 (C-5), 27.56 (C-11), 29.38 (C-4or10), 29.45 (C-4or10), 41.47 (C-3), 49.37 (C-6), 142.36 (C-8), 146.34 (C-2), 166.97 (C-1); IR (neat) 1661, 1588; EIMS,  $m/z$  (rel intensity) 358 ( $M^+ - Bu$ , 45), 244 (19); HRMS calcd for  $C_{15}H_{28}NOSn$  ( $M^+ - C_4H_9$ ) 358.1193, found 358.1193.

**5-(2-Methyl-[1,3]dioxolan-2-yl)-3-[(tributylstannanyl)methylene]-2-piperidinone (2I).**

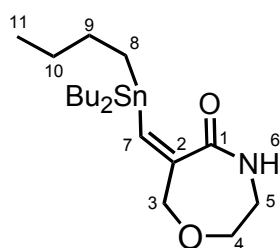


E isomer: ( $R_f$  = 0.08, hexane/EtOAc = 1/1), colorless oil.  $^1H$  NMR ( $CDCl_3$ , 500 MHz)  $\delta$  0.84-1.00 (m, 15H, H-11,14), 1.25-1.35 (m, 9H, H-8,13), 1.45-1.54 (m, 6H, H-12), 2.10-2.26 (m, 1H, H-4), 2.40-2.52 (m, 1H, H-3), 2.66-2.76 (m, 1H, H-3), 3.26-3.34 (m, 1H, H-5), 3.40-3.47 (m, 1H, H-5), 3.85-4.02 (m, 4H, H-9), 5.83 (bs, 1H, H-6), 7.44 (s,  $J$   $^1H-Sn$  = 59.1 Hz, 1H, H-10);  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz)  $\delta$  10.17 (C-11), 13.77 (C-14), 21.80 (C-8), 29.23 (C-12), 33.53 (C-3), 43.32 (C-4or5), 43.41 (C-4or5), 65.02 (C-9), 109.56 (C-7), 142.14 (C-10), 143.36 (C-2), 164.65 (C-1); IR (neat) 1663, 1592; EIMS,  $m/z$  (rel intensity) 430 ( $M^+ - C_4H_9$ , 9), 386 (14); HRMS calcd for  $C_{18}H_{32}NO_3Sn$  ( $M^+ - C_4H_9$ ) 430.1405, found 430.1407.

Z isomer: ( $R_f$  = 0.73, hexane/EtOAc = 1/1), colorless oil.  $^1H$  NMR ( $CDCl_3$ , 500 MHz)  $\delta$  0.76-0.92 (m, 15H, H-11,14), 1.22-1.34 (m, 9H, H-8,13), 1.38-1.52 (m, 6H, H-12), 2.12-2.22 (m, 1H, H-4), 2.47-2.58 (m, 1H, H-3), 2.76-2.84 (m, 1H, H-3), 3.22-3.30 (m, 1H, H-5),

3.38-3.46 (m, 1H, H-5), 3.84-4.00 (m, 4H, H-9), 6.50 (s,  $J$   $^1\text{H-Sn}$  = 68.7 Hz, 1H, H-10) 7.40 (bs, 1H, H-6);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  11.94 ( $J$   $^{13}\text{C-Sn}$  = 360.4 Hz, C-11), 13.89 (C-14), 21.72 (C-8), 27.55 ( $J$   $^{13}\text{C-Sn}$  = 56.6 Hz, C-12), 29.43 ( $J$   $^{13}\text{C-Sn}$  = 19.2 Hz, C-13), 34.44 (C-3), 42.98 (C-4or5), 43.27 (C-4or5), 64.95 (C-9), 109.65 (C-10), 141.89 (C-7), 146.83 (C-2), 166.85 (C-1); IR (neat) 1662, 1588; EIMS,  $m/z$  (rel intensity) 430 ( $\text{M}^+ - \text{C}_4\text{H}_9$ , 15), 272 (4); HRMS calcd for  $\text{C}_{18}\text{H}_{32}\text{NO}_3\text{Sn}$  ( $\text{M}^+ - \text{C}_4\text{H}_9$ ) 430.1405, found 430.1404.

**6-[(Tributylstannanyl)methylene]-[1,4]oxazepan-5-one (2n).**



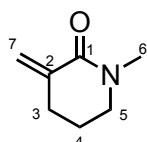
*E* isomer: ( $R_f$  = 0.15, hexane/EtOAc = 1/1), colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  0.82-1.08 (m, 15H, H-8,11), 1.20-1.34 (m, 6H, H-10), 1.40-1.56 (m, 6H, H-9), 3.31 (q,  $J$  = 4.0 Hz, 2H, H-5), 3.78 (t,  $J$  = 4.6 Hz, 2H, H-4), 4.14 (s, 2H, H-3), 6.48 (bs, 1H, H-6), 7.12 (s,  $J$   $^1\text{H-Sn}$  = 52.7 Hz, 1H, H-7);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  10.53 (C-8), 13.74 (C-11), 27.33 (C-10), 29.11 (C-9), 44.20 (C-3), 70.15 (C-5), 71.31 (C-4), 145.84 (C-7), 150.96 (C-2), 174.63 (C-1); IR (neat) 1653, 1579; EIMS,  $m/z$  (rel intensity) 360 ( $\text{M}^+ - \text{C}_4\text{H}_9$ , 8), 246 (4); HRMS calcd for  $\text{C}_{12}\text{H}_{26}\text{NO}_2\text{Sn}$  ( $\text{M}^+ - \text{C}_4\text{H}_9$ ) 360.0985, found 360.0986.

*Z* isomer: ( $R_f$  = 0.58, hexane/EtOAc = 1/1), colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  0.80-0.92 (m, 15H, H-8,11), 1.20-1.32 (m, 6H, H-10), 1.38-1.54 (m, 6H, H-9), 3.32 (q,  $J$  = 4.6 Hz, 2H, H-5), 3.79 (t,  $J$  = 4.6 Hz, 2H, H-4), 4.25 (s, 2H, H-3), 6.73 (s,  $J$   $^1\text{H-Sn}$  = 60.1 Hz, 1H, H-7), 7.47 (bs, 1H, H-6);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  11.28 ( $J$   $^{13}\text{C-Sn}$  = 353.7 Hz, C-8), 13.84 (C-11), 27.45 ( $J$   $^{13}\text{C-Sn}$  = 58.5 Hz, C-10), 29.26 ( $J$   $^{13}\text{C-Sn}$  = 20.2 Hz, C-9), 43.80 (C-3), 70.06 (C-5), 72.74 (C-4), 149.64 (C-7), 150.13 (C-2), 174.58 (C-1); IR (neat) 1650, 1591; EIMS,  $m/z$  (rel intensity) 360 ( $\text{M}^+ - \text{C}_4\text{H}_9$ , 8), 246 (3); HRMS calcd for  $\text{C}_{14}\text{H}_{26}\text{NO}_2\text{Sn}$  ( $\text{M}^+ - \text{C}_4\text{H}_9$ ) 360.0985, found 360.0985.

**Procedure for the carbonylative  $S_N1$  reaction coupled with the subsequent protodestannylation of *N*-methyl-*N*-(1-phenylethyl)-4-pentynyl-1-amine (**1o**).**

A magnetic stirring bar, AIBN (31.6 mg, 0.19 mmol), benzene (100 mL),  $\text{Bu}_3\text{SnH}$  (457.1 mg, 1.57 mmol), and *N*-methyl-*N*-(1-phenylethyl)-4-pentynyl-1-amine (**1o**) (225.4 mg, 1.12 mmol) were placed in a 200-mL stainless autoclave. The autoclave was closed, purged three times with carbon monoxide, pressurized with 93 atm of CO and then heated 90 °C for 4 h. Excess CO was discharged at room temperature. The solvent was removed under reduced pressure. To the residue dissolved in methanol (7 mL) was added dropwise TMSCl (1.5 mL) at room temperature and the reaction mixture was stirred for 10 min. The solvent was removed under reduced pressure. The residue was purified by flash chromatography on silica gel to give 1-methyl-3-methylene-piperidin-2-one (**2o**) (120.9 mg, 86%).

**1-Methyl-3-methylene-piperidin-2-one (**2o**)**



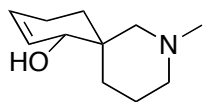
Colorless oil. ( $R_f$  = 0.2, Hexane/EtOAc = 1/1),  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  1.88 (quint,  $J$  = 6.0 Hz, 2H, H-4), 2.55 (t,  $J$  = 6.4 Hz, 2H, H-3), 3.01 (s, 3H, H-6), 3.37 (t,  $J$  = 6.0 Hz, 2H, H-5), 5.24 (s, 1H, H-7), 6.17 (s, 1H, H-7);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  23.07 (C-4), 30.11 (C-3), 35.09 (C-5), 50.30 (C-6), 120.90 (C-2), 137.82 (C-7), 164.35 (C-1); IR (neat) 1657, 1613; EIMS,  $m/z$  (rel intensity) 125 ( $\text{M}^+$ , 100), 96 (17), 82 (18), 69 (24), 54 (100); HRMS calcd for  $\text{C}_7\text{H}_{11}\text{NO}$  ( $\text{M}^+$ ) 125.0841, found 125.0848

**Procedure for the synthesis of ( $\pm$ )-sibirine from 1-methyl-3-methylene-piperidin-2-one (**2o**)**

A mixture of 1-methyl-3-methylene-piperidin-2-one (130 mg, 1 mmol) and 1-(trimethylsilyloxy)-1,3-butadiene (4 mL,  $E/Z$  = 94/6) was stirred for 24 h at reflux. After the

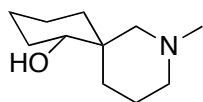
remained diene was removed, the residue was purified by flash chromatography on silica gel (hexane/EtOAc = 2/1) to give **4** (303.1 mg,  $R_f$  = 0.3). Further purification using preparative HPLC gave the desired spirocyclic compound **4** (132.6 mg, 0.5 mmol, 50%). The spirocyclic compound **4** (0.5 mmol) was added to a suspension of  $\text{LiAlH}_4$  (19 mg, 1.0 equiv) in ether (5 mL). The mixture was stirred for 1 h. The reaction was then quenched with water and the resulting mixture extracted with ether. The organic layer was treated with acid/base workup and the desired compound **5** was obtained. (60.8 mg, 0.34 mmol, 68%). The compound **5** (60.8 mg, 0.34 mmol) was added to the suspension of  $\text{Pd}(\text{OH})_2/\text{C}$  (3 mg) in methanol (3 mL). The mixture was stirred for 20 h at room temperature under hydrogen atmosphere. The reaction mixture was filtered through Celite. After removal of the solvent, the residue was dissolved with ether. The ethereal solution of the mixture was treated with acid/base workup to give the desired alkaloid ( $\pm$ )-sibirine (37.4 mg, 60%).

#### 7-Hydroxy-2-azaspiro[5,5]undec-8-ene (**5**)



Colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  0.96-1.10 (m, 1H), 1.20-1.40 (m, 3H), 1.48-1.62 (m, 1H), 1.85-2.20 (m, 6H), 2.20-2.30 (m, 3H), 2.45-2.60 (m, 1H), 2.60-2.80 (m, 1H), 4.15-4.30 (m, 1H), 5.60-5.70 (m, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  22.07, 22.78, 27.34, 30.76, 35.79, 46.73, 56.57, 67.83, 75.82, 126.88, 129.67; IR (neat) 1451, 1650, 2789, 2849, 2936, 3402; EIMS,  $m/z$  (rel intensity) 181 ( $\text{M}^+$ , 46), 138 (20), 112 (33), 91 (17), 70 (17), 58 (100); HRMS calcd for  $\text{C}_{11}\text{H}_{19}\text{NO}$  ( $\text{M}^+$ ) 181.1467, found 181.1464.

#### ( $\pm$ )-Sibirine (**6**)



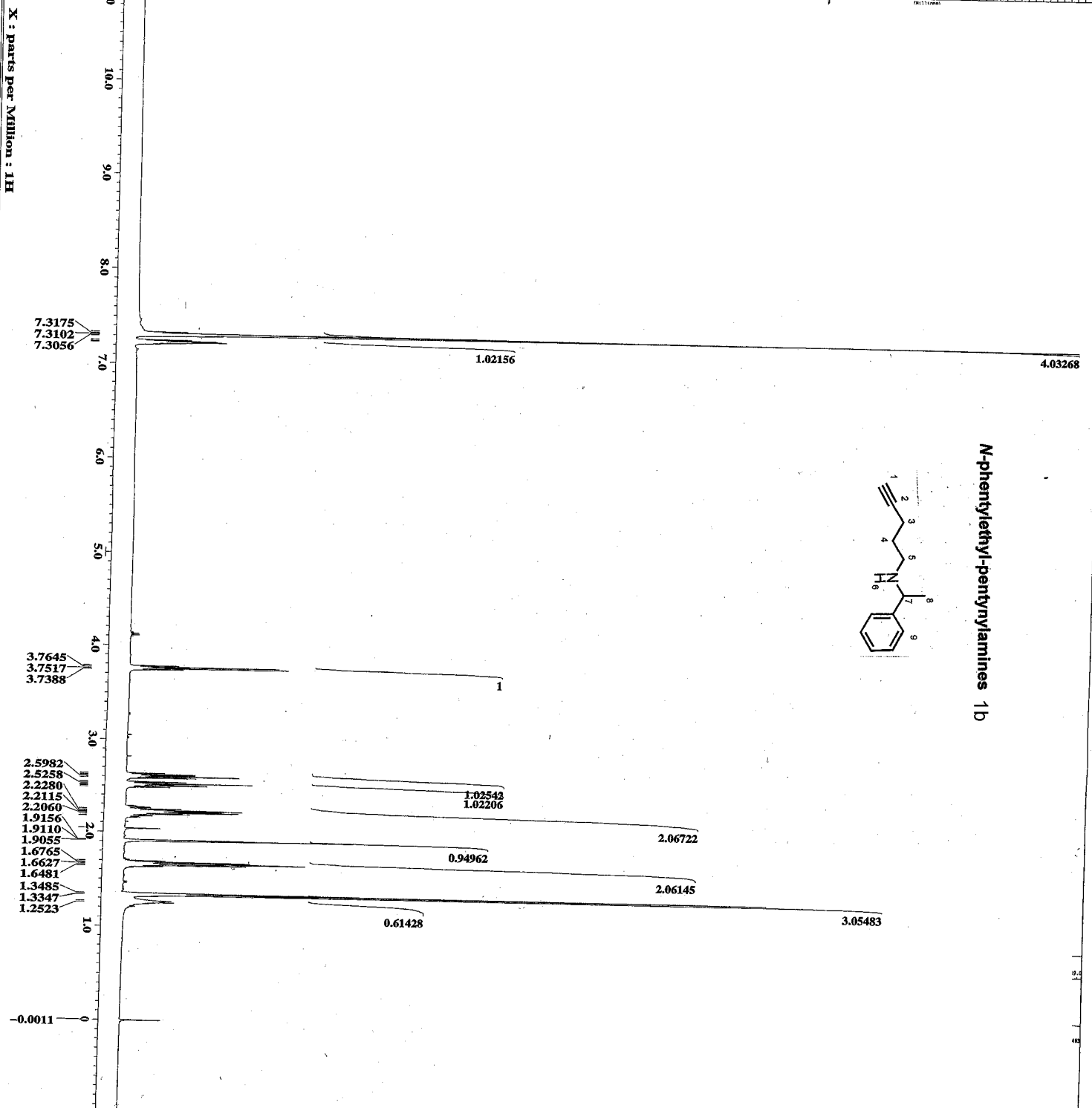
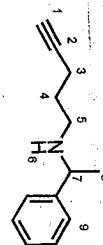
Colorless oil.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz)  $\delta$  0.80-1.00 (m, 2H), 1.10-1.28 (m, 2H), 1.32-1.40 (m, 2H), 1.46-1.55 (m, 2H), 1.66-1.76 (m, 2H), 1.82-1.92 (m, 2H), 2.08-2.16 (m, 2H), 2.10 (s, 3H), 2.57 (d,  $J$  = 11.0 Hz, 1H), 2.74-2.84 (m, 1H), 3.59 (dd,  $J$  = 3.7, 11.5 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz)  $\delta$  20.33, 23.08, 24.35, 27.66, 29.44, 37.05, 37.15, 46.46, 56.36, 69.95, 80.51; IR (neat) 1450, 2788, 2859, 2931, 3406; EIMS,  $m/z$  (rel intensity) 183 ( $\text{M}^+$ , 19), 155 (9), 140 (25), 98 (22), 71 (28), 57 (100); HRMS calcd for  $\text{C}_{11}\text{H}_{21}\text{NO}$  ( $\text{M}^+$ ) 183.1623, found 183.1623.

These data are identical with those reported for (-)-sibirine.<sup>6</sup>

#### References

- 1 PROCESS-AUTO. *Automatic Data Acquisition and Processing Package for Imaging Plate Diffractometer*, Rigaku Corporation: Tokyo, Japan, 1998.
- 2 Higashi, T. *ABSCOR, Empirical Absorption Correction based on Fourier Series Approximation*; Rigaku Corporation: Tokyo, Japan, 1998.
- 3 Sheldrick, G. M. *SHELX97, Program for Crystal Structure Determination*; University of Göttingen: Göttingen, Germany, 1997.
- 4 Tojino, M.; Uenoyama, Y.; Fukuyama, T.; Ryu, I. *Chem. Commun.* **2004**, 2482.
- 5 For these compounds, some <sup>13</sup>C-NMR signal corresponding to the respective diastereomers are not well resolved due to overlapping.
- 6 (a) Dembele, Y. A.; Belaud, C.; Villieras, J. *Tetrahedron Asymmetry*, **1992**, 3, 511. (b) Frenandes, R. A.; Yamamoto, Y. *J. Org. Chem.* **2004**, 69, 3562.
- 7 Koreeda, M.; Wang, Y.; Zhang, L. *Org. Lett.* **2002**, 4, 3329.

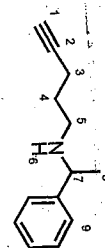
*N*-phenylethyl-pentylamines 1b



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# N-phenylethyl-pent-1-ynylamines 1b



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68.6898

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46.6616

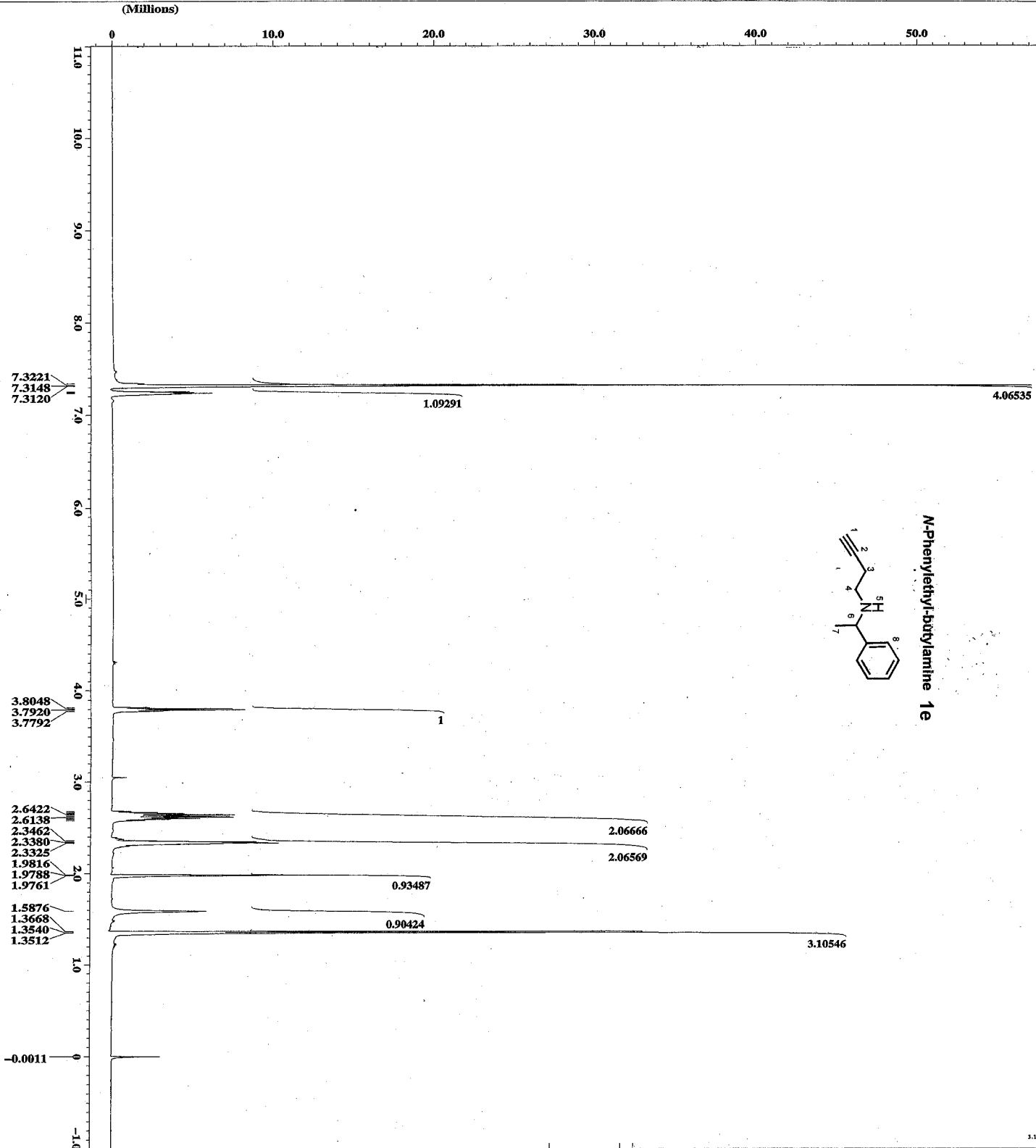
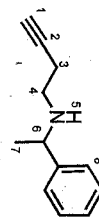
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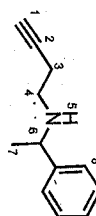
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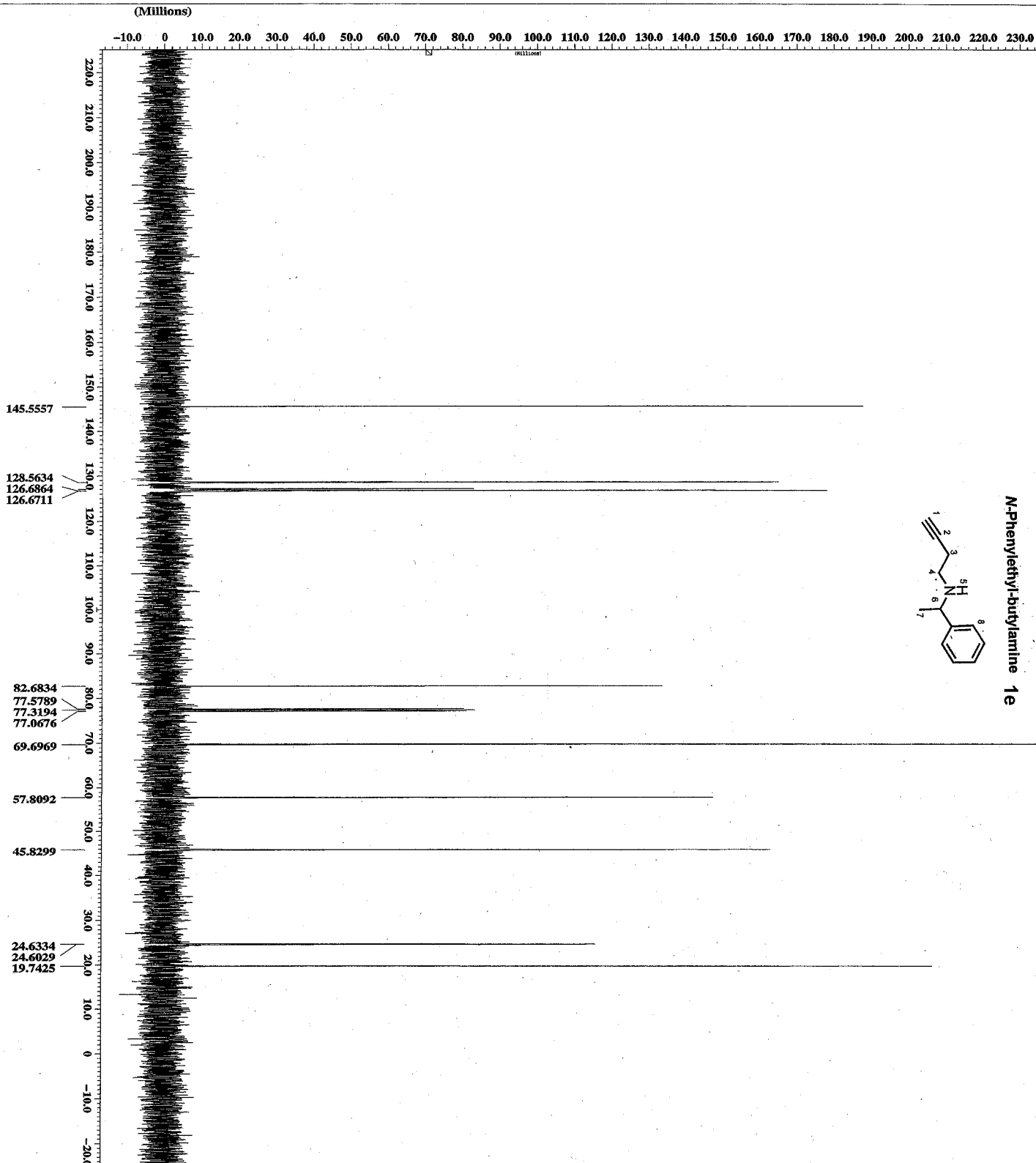
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N-Phenylethyl-butylamine 1e



X : parts per Million : 13C

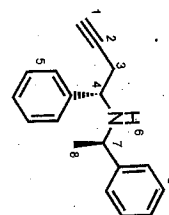


**JEOL**

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## Preparation of (1-Phenyl-but-3-ynyl)-(1-phenyl-ethyl)-amine 1f



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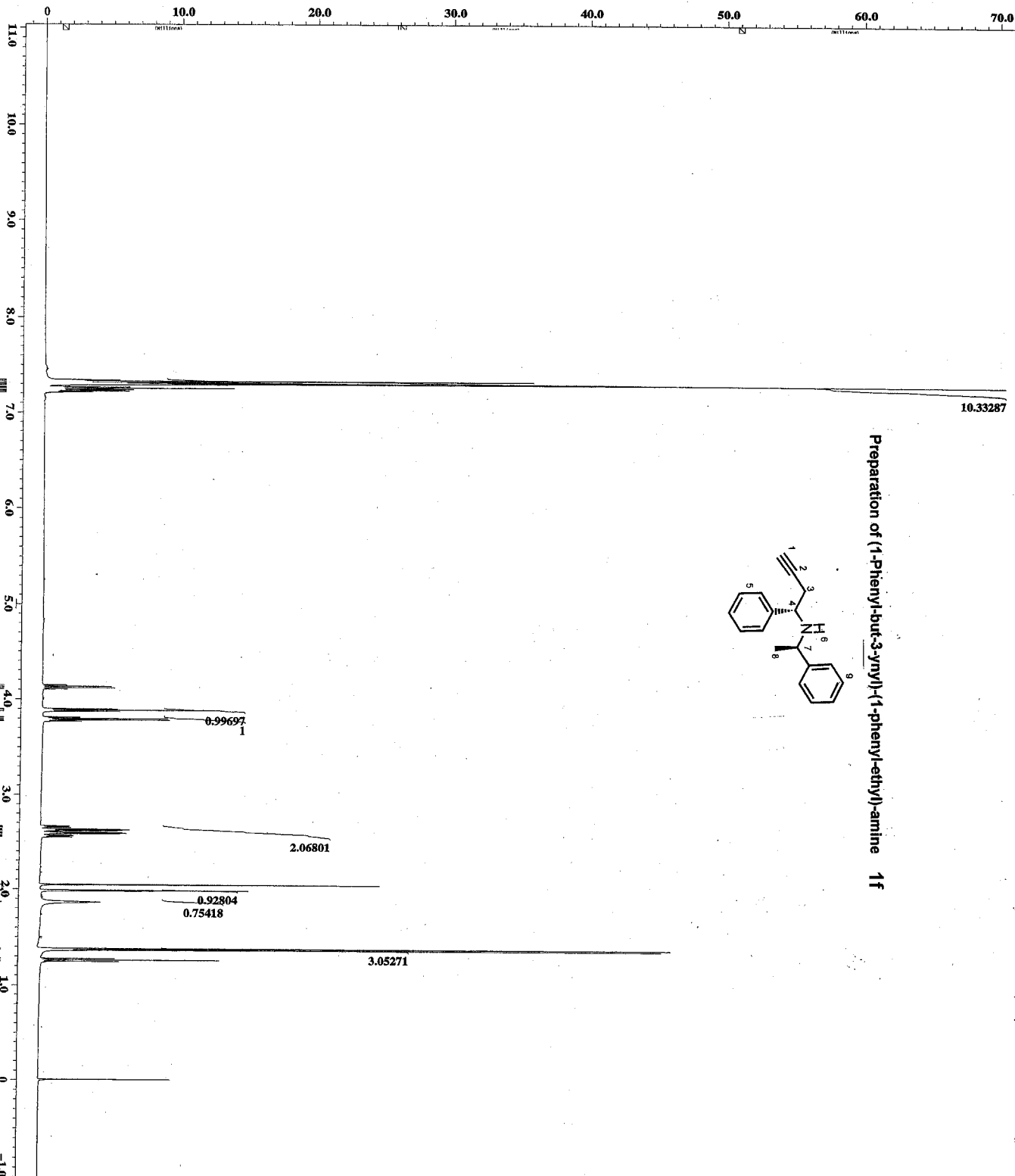
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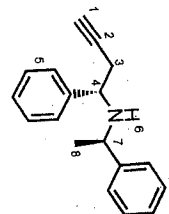
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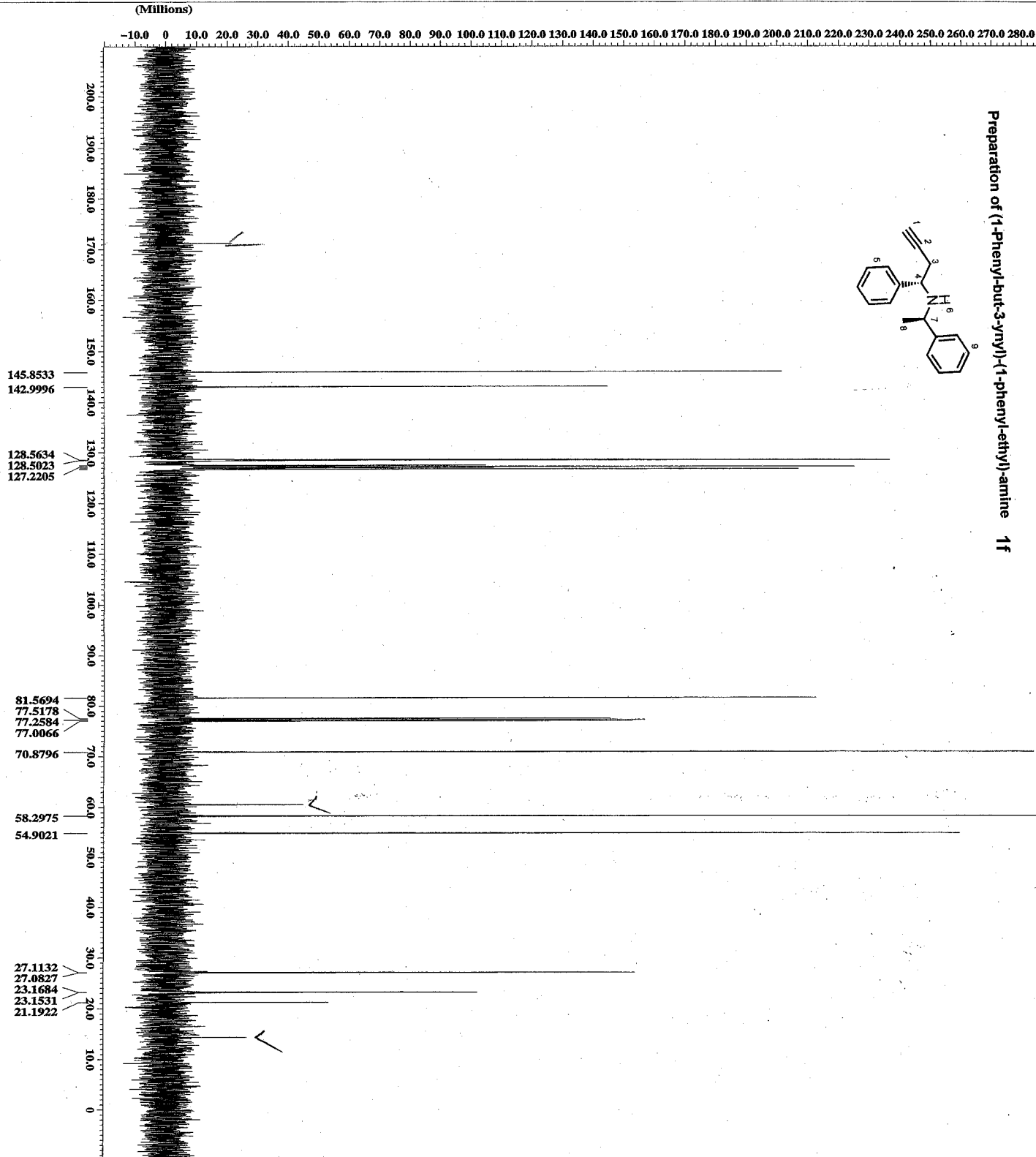
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Preparation of (1-Phenyl-but-3-ynyl)-(1-phenylethyl)-amine 1f

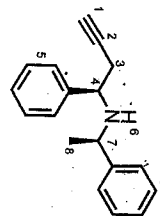


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Preparation of (1-Phenyl-but-3-ynyl)-(1-phenyl-ethyl)-amine 1g



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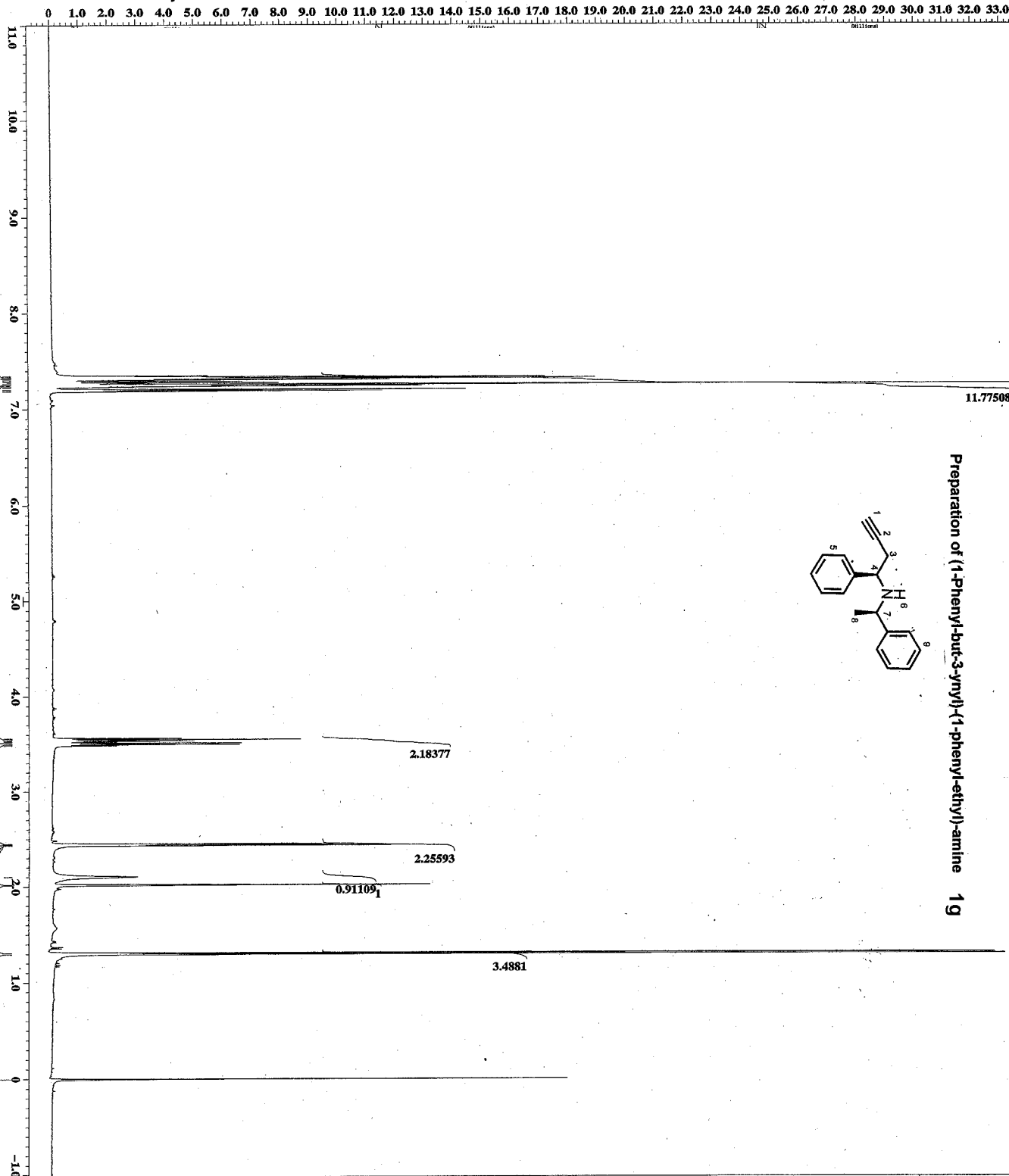
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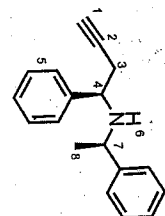
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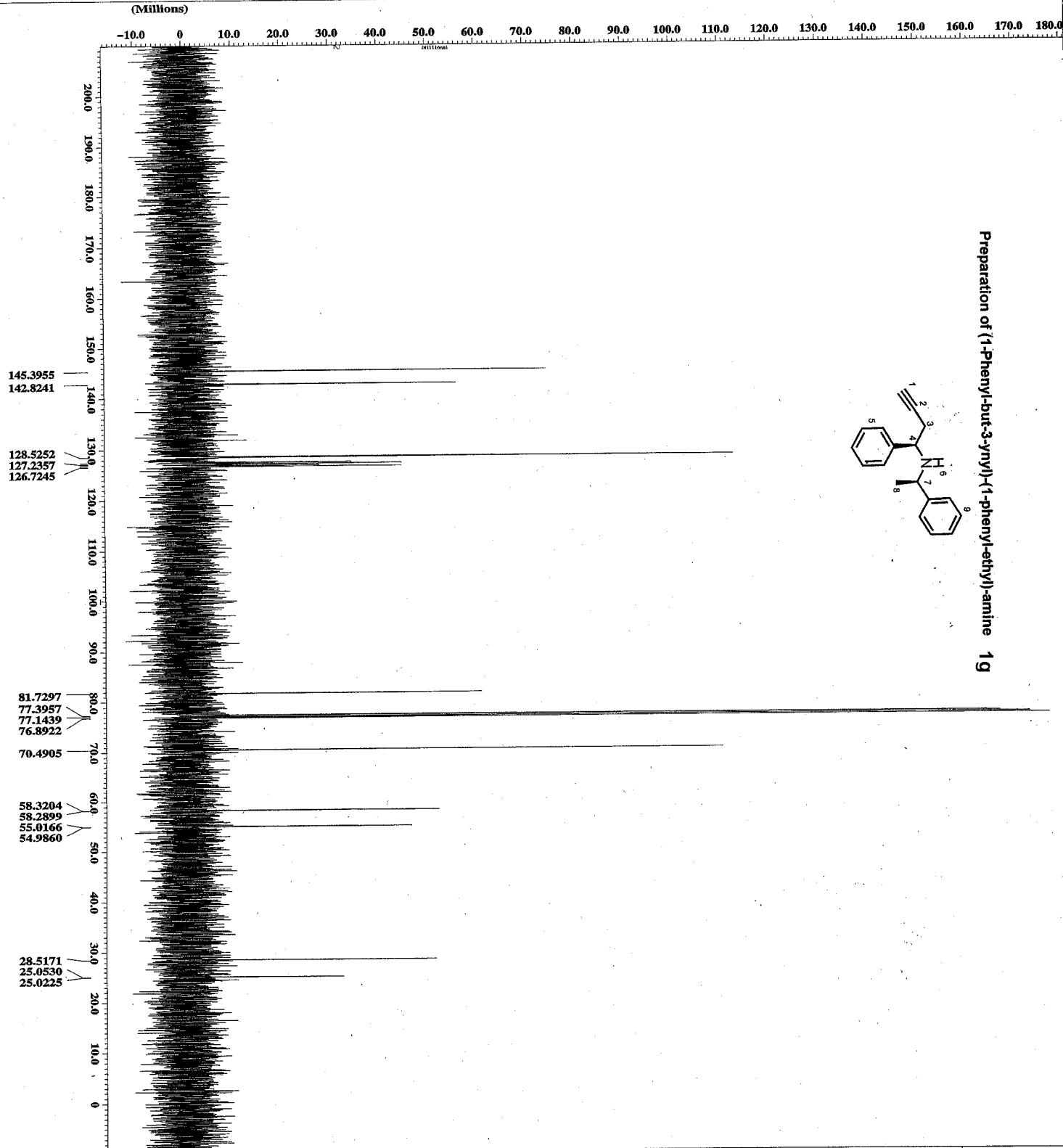
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Creation Date = 15-APR-2005 21:49:34  
Revision Date = 15-APR-2005 23:34:20  
Spec Site = KCP500  
Spec Type = 1H NMR  
Date Acquired = 15-APR-2005  
Dimensions = 1H  
Dim Title = 1H  
Dim Size = 16384  
Dim Units = [ppm]  
Mod return = 1  
X domain = 1H  
X offset = 51.0ppm  
X freq = 500.16241602(MHz)  
X sweep = 0.50750751(MHz)  
SOLVENT = CDCl3  
Spin set = 18(Hz)  
Temp set = 21.91dc  
Recvr gain = 25  
Field strength = 11.74737912  
Filter\_name = 500KHZ1H1  
Filter\_width = 5.75119306(MHz)

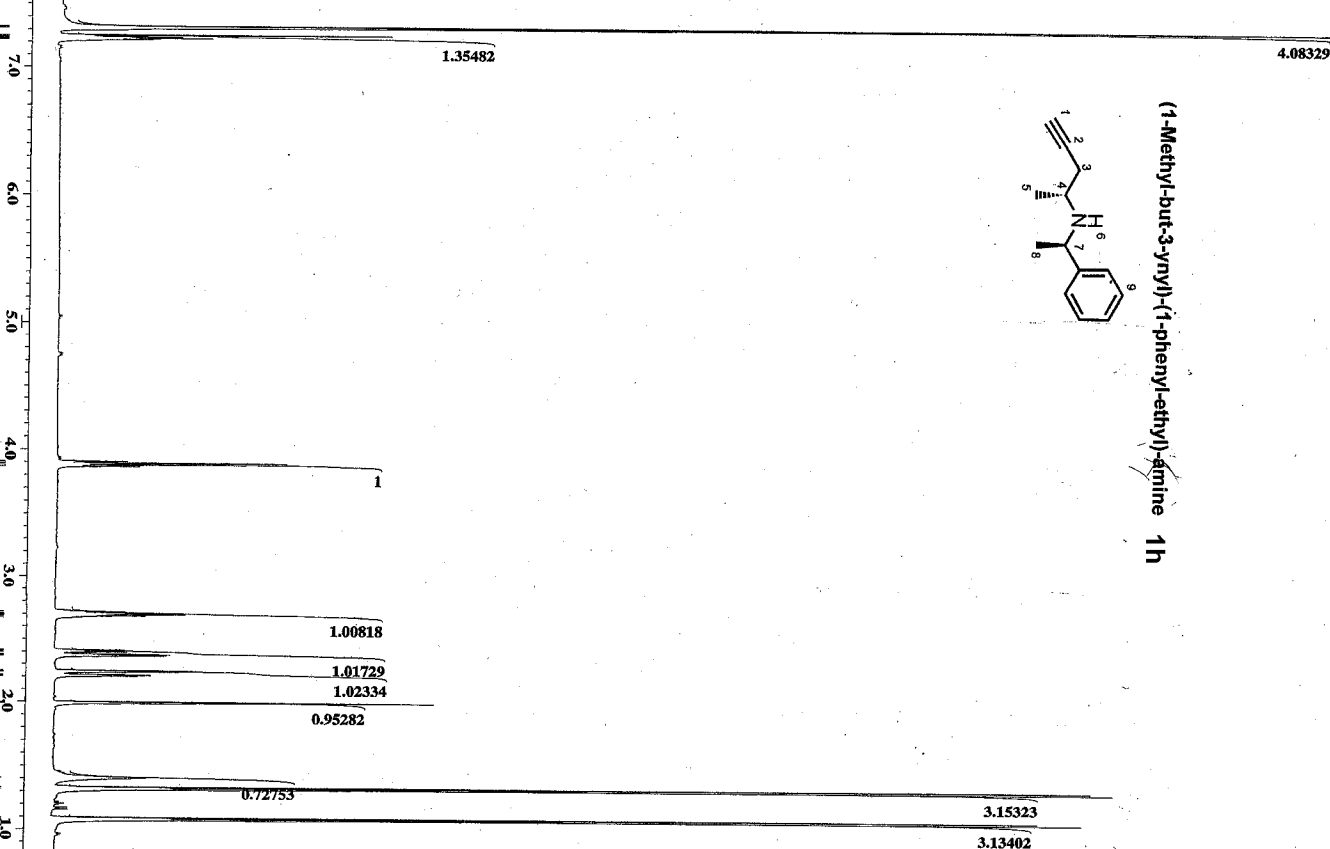
Preparation of (1-Phenyl-but-3-ynyl)-(1-phenyl-ethyl)-amine 1g

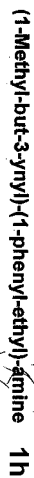


X : parts per Million : 13C



ACQUISITION PARAMETERS  
 File Name = 1d\_13c\_spectrum.1300  
 Sample ID = S8649791  
 Content = Single Pulse with Broad  
 Creation Date = 14-SEP-2005 21:57:10  
 Revision Date = 15-SEP-2005 21:39:34  
 Spec Site = KCP300  
 Spec Type = DELTA\_NMR  
 Data Format = 1D\_COMPLEX  
 Dimensions = 1  
 Num Fids = 130  
 Num Ss = 130  
 Num Files = 130  
 Scans = 53  
 Mod Return = 1  
 X Domain = 130  
 X Offset = 100 [ppm]  
 X Range = 12.7747 [ppm]  
 X Resol = 11.4465088 [Hz]  
 Solvent = CHLOROFORM-D  
 Spin Gat = 11 [Hz]  
 Temp Gat = 22.9 [C]  
 Recv Gain = 30.747379 [V]  
 Pulse Length = 12.00 [ppm]  
 Filter Mode = HETZSCHMIDT  
 Filter Width = 15.7206221 [Hz]

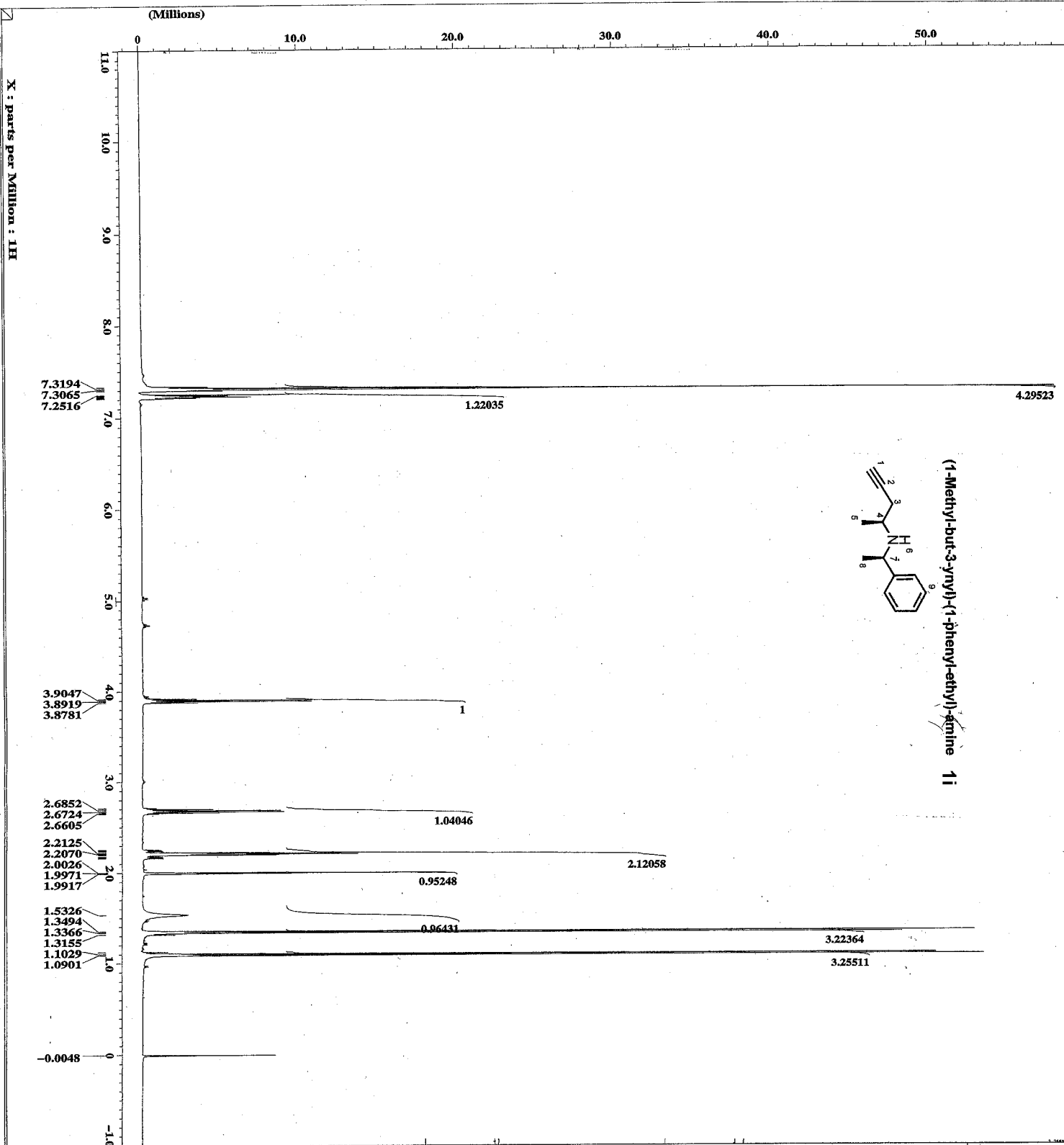
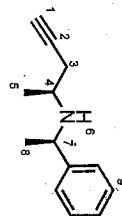




**JEOL**

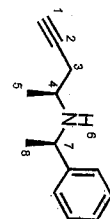
----- ACQUISITION PARAMETERS -----  
 File Name = 1d\_spectrum\_4618  
 Author ID = 11  
 Experiment = Single Pulse Experiment  
 Content = 13-APR-2005 13:56:28  
 Creation Date = 13-APR-2005 13:56:28  
 Revision Date = 14-APR-2005 15:40:50  
 Spec Name = ECP500  
 Spec Type = 1H NMR  
 Data Format = ID CONTEX  
 Dimensions = 1  
 Data Type = 1H  
 Data Size = 16384  
 Data Rate = 1000  
 Scans = 8  
 Mod. return = 1  
 X. domain = 1H  
 X. offset = 0.0000000000000000  
 X. sweep = 0.0000000000000000  
 Solvent = CHLOROFORM-D  
 Spin. get = 14[Hz]  
 Temp. get = 22.3[degC]  
 Acq. gain = 23.747379[Hz]  
 Filter mode = HETEROMODE  
 Filter width = 3.7511936[Hz]

## (1-Methyl-but-3-ynyl)-(1-phenylethyl)-amine 11





## (1-Methyl-but-3-ynyl)-(1-phenylethyl)-amine 11



X : parts per Million : 13C

145.6930  
128.5329  
126.9687  
126.6253

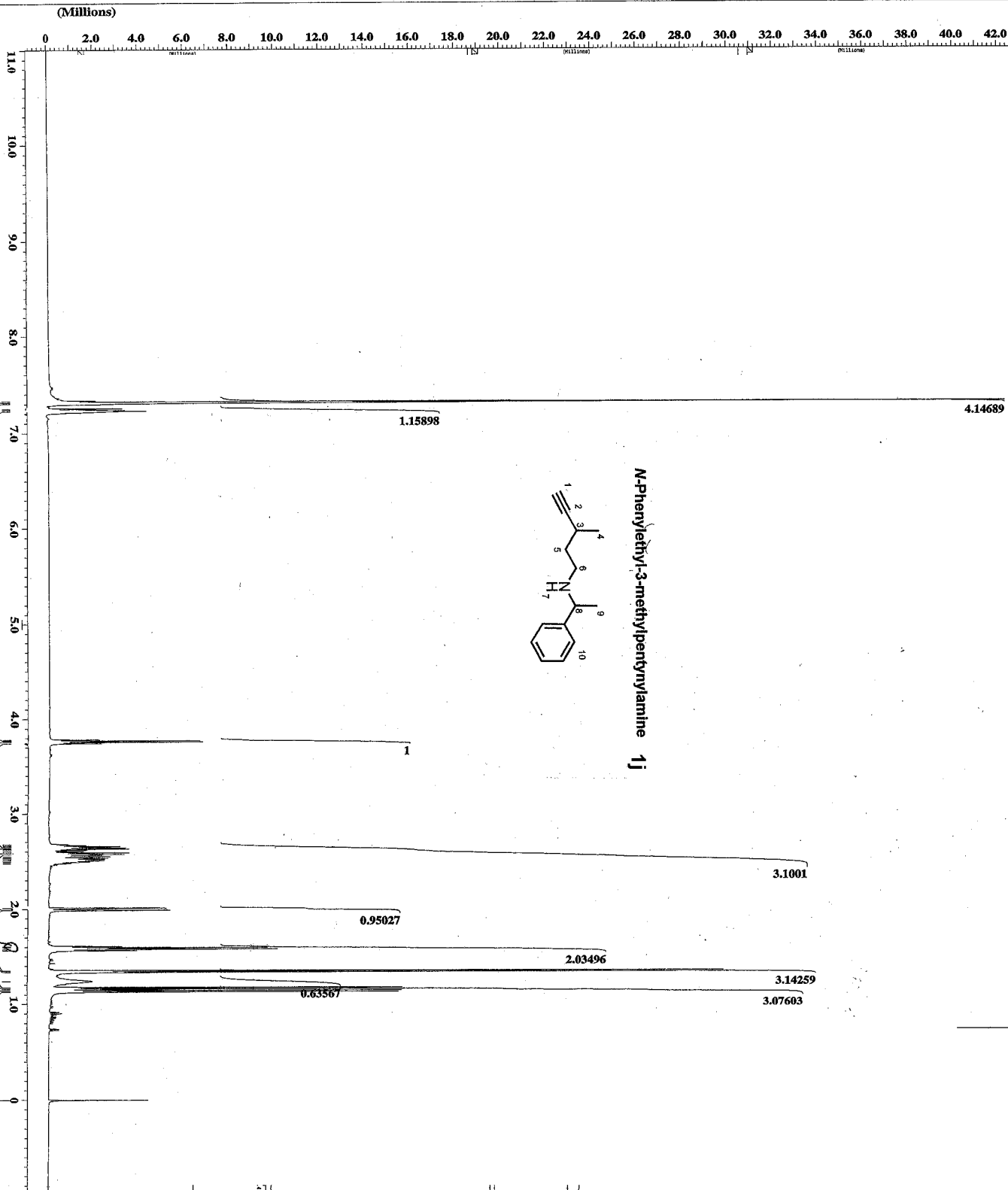
81.9509  
77.4644  
77.2126  
76.9532  
70.3302

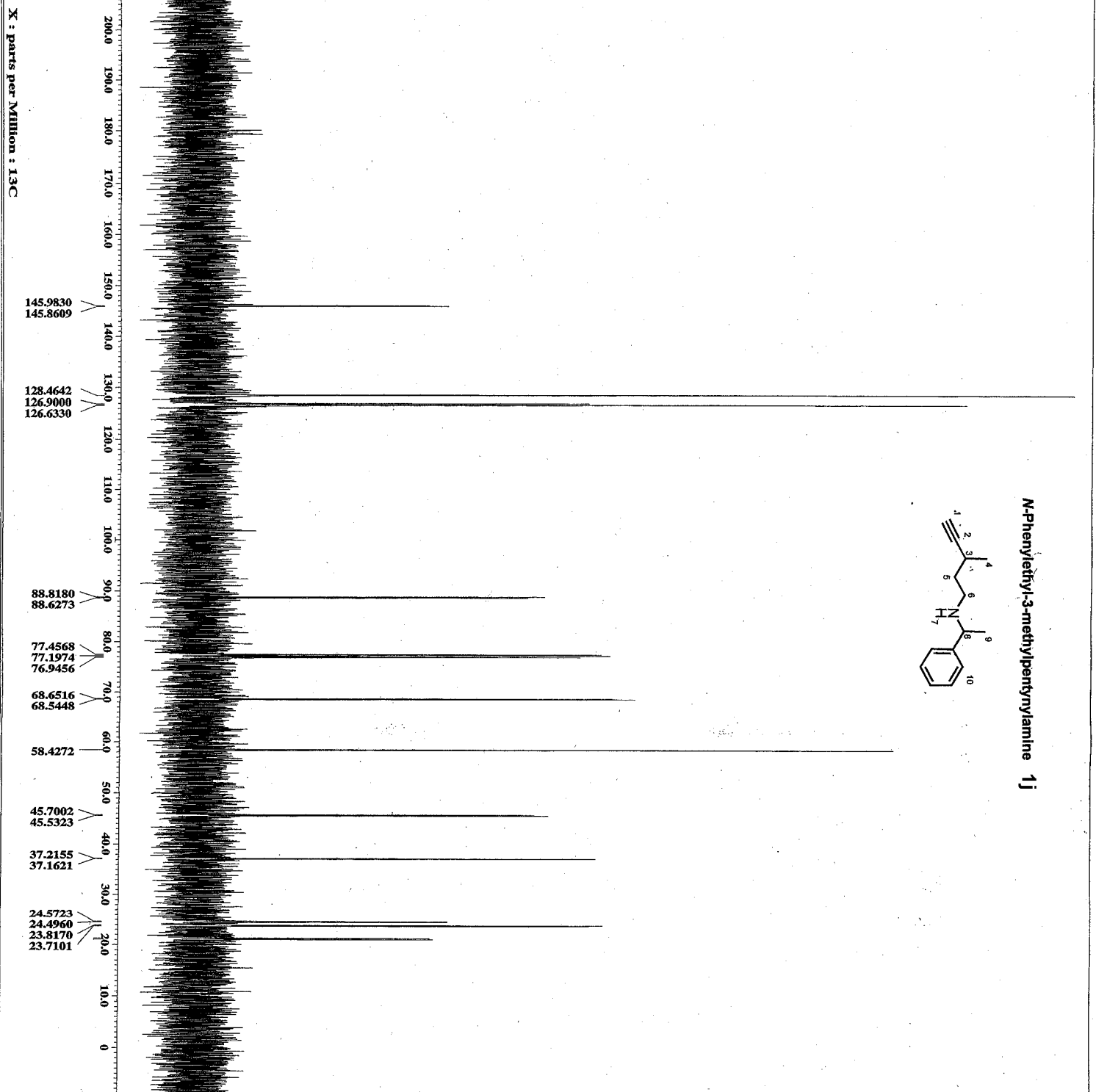
54.9860  
48.6759

27.1056  
24.9615  
24.9386  
19.7043

----- ACQUISITION PARAMETERS -----  
File Name = 1d\_13c\_spectrum.111  
Author = syon  
Sample ID = syon  
Content = Single Pulse with Broad  
Creation Date = 11-NOV-2004 18:14:57  
Revision Date = 11-NOV-2004 17:09:26  
Spec Site = ECP500  
Spec Type = DELTA\_NMR  
Pulse Program = zgpg30  
Dimensions = 13C  
D1a Title = 13C  
D1a Size = 32768  
D1a Units = [ppm]  
D1a Mod = 45  
X Domain = 13C  
X Offset = 100 [ppm]  
X Freq = 125.7778547 [MHz]  
X Sweep = 31.44654086 [Hz]  
SOLVENT = CDCl3  
SOLV\_PPM = 77.00  
Temp Set = 22.8 [deg]  
Recvr Gain = 30  
Field Strength = 11.7473579 [T]  
P1 Pulsar Mode = BOUTERBROWER  
Filter Width = 15.72066221 [Hz]

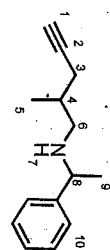
----- ACQUISITION PARAMETERS -----  
 File Name = ID\_Spectrum\_4631  
 Author ID =  
 Date =  
 Content = Sample Pulse Experiment  
 Creation Date = 13-APR-2005 17:07:41  
 Revision Date = 14-APR-2005 18:52:19  
 Spec Site = ECP500  
 Spec Type = DELTA NMR  
 Data Format = ID COMPLEX  
 Dimensions = X  
 Dim Title = 1H  
 Dim Size = 16384  
 Num Nuclei = 1  
 Name = 1H  
 Mod\_return = 1  
 X\_domain = 1H  
 X\_offset = 5.0ppm  
 X\_freq = 400.146021MHz  
 X\_gamma = 201.150711Hz  
 Solvent = CHLOROFORM-D  
 Spia\_get = 17Hz  
 Temp\_get = 22.4[C]  
 Recvz\_gain = 21.747379Hz  
 Recvz\_offset = 17.450Hz  
 Filter\_width = 3.7513936KHz





Author	John R. Stone	Acquisition Parameters	----
File Name	id_161_spectrum.1283		
Sample ID	a		
Content	Single Pulse with Broad		
Creation Date	13-MAR-2005 17:27:46		
Revision Date	14-MAR-2005 19:11:22		
Spec Site	KCP500		
Spec Format	= DATA_BNR		
Data Format	= ID_COMPLEX		
File Size	= 11K		
Din Size	= 32768		
Din Units	= [psec]		
Scans	= 67		
X_Center	= 1.000		
X_Offset	= 100 [psec]		
F_Freq	= 135.77787547 [MHz]		
F_sweep	= 3.44654088 [Hz/s]		
Solvent	= CHLOROFORM-D		
Temp Set	= 32.0 [C]		
Recovery Gain	= 30.1 [dB]		
Field Strength	= 11.7473579 [G]		
Pulse Code	= BIRDHOMER-2		
Pulse Width	= 17.2066522 [psec]		

## N-Phenylethyl-2-methylpentylamine 1k



X : parts per Million : 1H

7.3175  
7.3084  
7.2342

1.04746

3.93815

3.7462  
3.7416  
3.7288

1

2.4259  
2.4140  
2.3820  
2.3032  
1.9330  
1.9275  
1.9257  
1.9202

0.58408  
0.47819

0.47617

0.95887

0.8977

0.97827

1.46299

1.3384  
1.3292

0.9747  
0.9618

0.73025

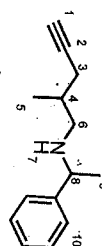
2.95368

2.95032

0.0017

ACQUISITION PARAMETERS  
File Name = 1d\_spectrum\_4634  
Sample ID = 1  
Content = Single Pulse Experiment  
Creation Date = 13-Apr-2005 17:37:56  
Revision Date = 14-Apr-2005 19:20:34  
Spec Site = ECP500  
Spec Type = 1H NMR  
Data Format = ID COMPLEX  
Dimensions = 1  
Dim 1 Size = 16384  
Dim 2 Size = 16384  
Dim 3 Size = 16384  
Dim 4 Size = 16384  
Mod Return = 1  
X Domain = 1H  
X Offset = 500.13241602 [Hz]  
X Freq = 500.13241602 [MHz]  
X 1 Freq = 500.13241602 [MHz]  
Solvent = CDCl3  
Spin Set = 16 [Hz]  
Temp Set = 22.3 [C]  
Recycle Delay = 19.473579 [s]  
NS = 128  
DS = 4  
SFO = 1  
Filter Width = 3.7511936 [Hz]

**N-Phenylethyl-2-methylpentylamine 1k**



X : parts per Million : 13C

146.1356  
146.0364

128.4566  
126.8771  
126.6711

83.1565  
83.1260  
77.4568  
77.2050  
76.9608

69.4223

58.5188  
58.3738  
58.3586  
53.0327  
52.9641

33.2020  
33.1105

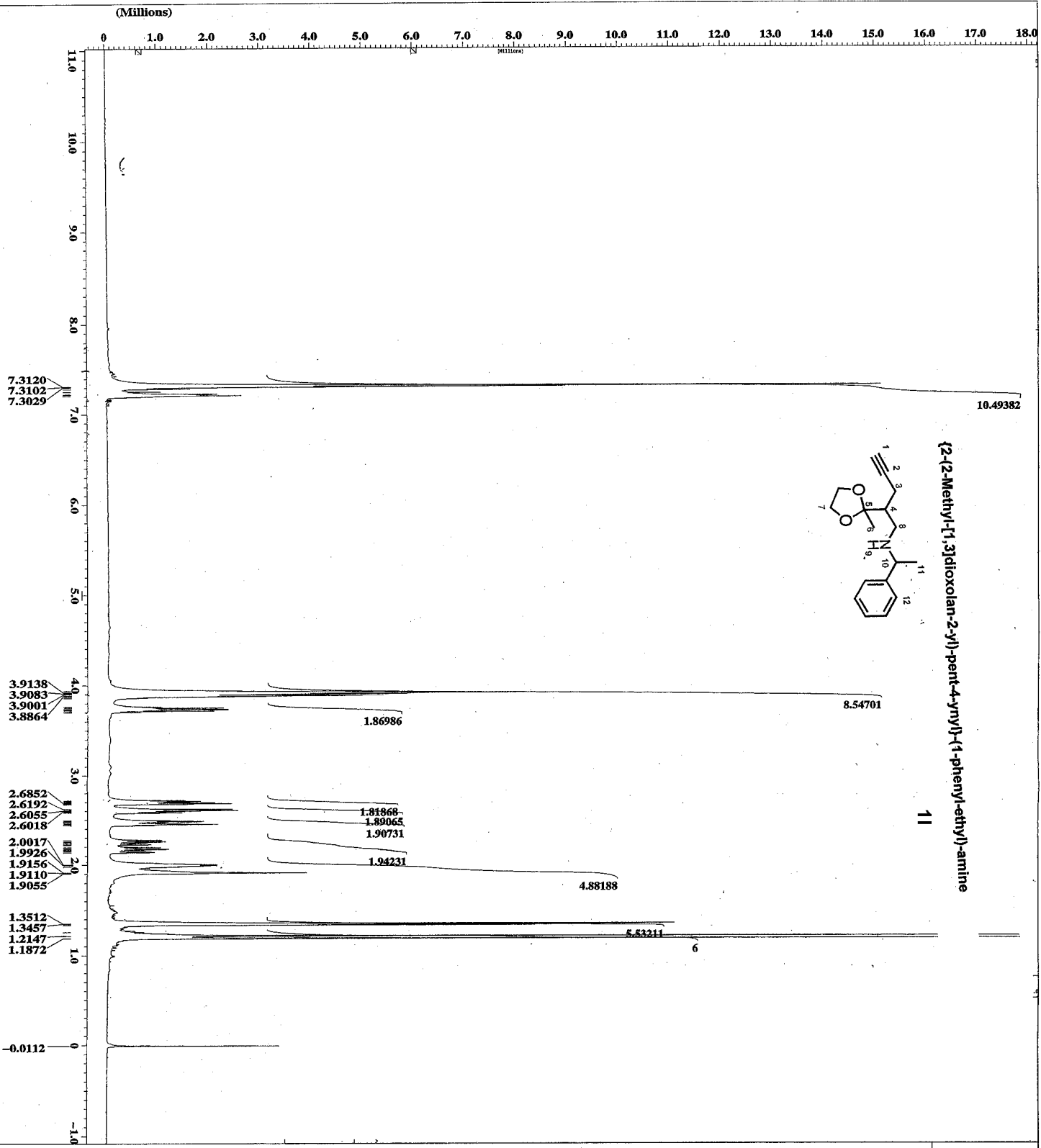
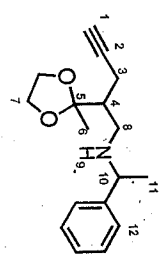
24.6868  
23.9085  
23.7101  
17.9418  
17.9189

----- ACQUISITION PARAMETERS -----  
File Name = 1d\_13c\_spectrum.1284  
Author =  
Sample ID = 1  
Content = Single Pulse with Broad  
Creation Date = 13-Apr-2005 17:45:14  
Revision Date = 14-Apr-2005 19:26:53  
Spec File = ECP500  
Spec Type = 1D NMR  
Pulse Program = zgpg30  
Dimensions = 13C  
Dia File = 13C  
Dia Size = 32768  
Dia Units = [ppm]  
Spectrum = 1  
X offset = 13C  
X offset = 106 [ppm]  
X freq = 125.7778547 [MHz]  
X sweep = 31.44654081 [kHz]  
SOLVENT = CDCl3  
SOLVENT-D =  
SOLVENT-D = 13C  
Temp. set = 23.2 [degC]  
Pulpr. gain = 30  
Field strength = 11.7473579 [T]  
Pulpr. mode = BUREAU  
Pulpr. width = 15.7206521 [kHz]

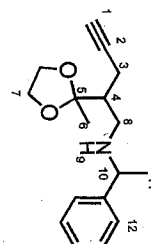
**JEOL**

----- ACQUISITION PARAMETERS -----  
 File Name = 1d\_spectrum\_4635  
 Author =  
 Computer ID =  
 Comment =  
 Experiment = Simple Pulse Experiment  
 Creation Date = 13-Apr-2005 18:05:20  
 Revision Date = 14-Apr-2005 19:50:06  
 Spec Site = ECP500  
 Spec Type = 1H NMR  
 Data Format = ID COMPLEX  
 Dimensions = 2  
 Dia Title =  
 Dia Size = 16384  
 Dia Units =  
 Scans = 8  
 Mod Return = 1  
 X Domain = 1H  
 X Offset = 5.000  
 X Freq = 500.145020 MHz  
 X Pulse = 7.0015072511 Hz  
 Solvent = CHLOROFORM-D  
 SpIn\_yet = 13 Hz  
 Temp\_yet = 22.51 (C)  
 Recvry\_gain = 21.743579 (V)  
 Filter\_gain = 1.000000  
 Filter\_width = 3.75119361 (Hz)

## {2-(2-Methyl-[1,3]dioxolan-2-yl)-pent-4-ynyl-(1-phenyl-ethyl)-amine



(2-(2-Methyl-(1,3)dioxolan-2-yl)-pent-4-ynyl)-(1-phenyl-ethyl)-amine 11



X: Parts per Million : 13C

146.0745  
145.9296

128.3803  
126.7093  
126.6559

111.3193  
111.1591

83.4998  
83.3701  
77.4873  
77.2202  
76.9761

69.3383  
64.6763  
64.6382  
64.6076  
64.5237  
58.5646  
58.4501

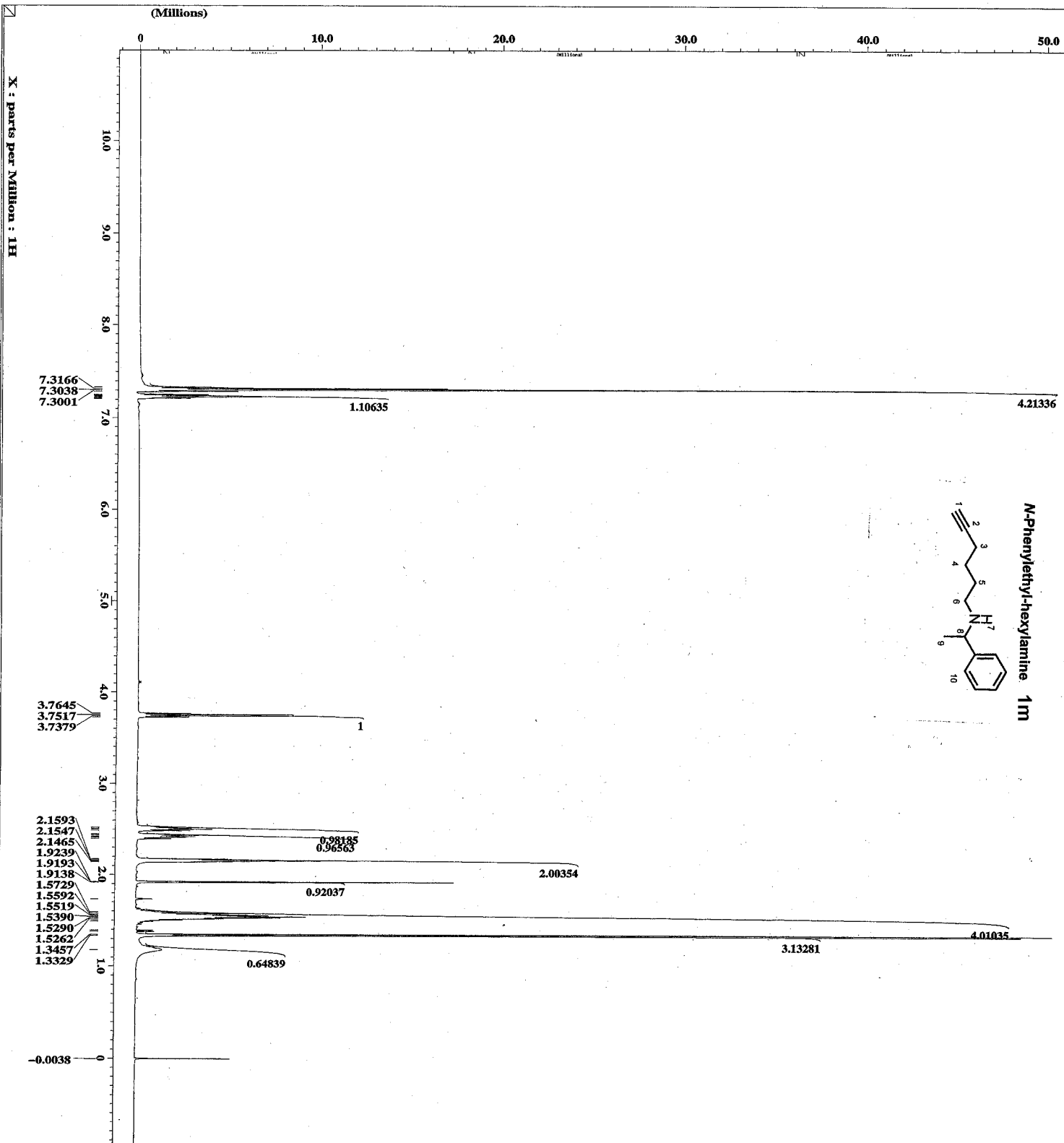
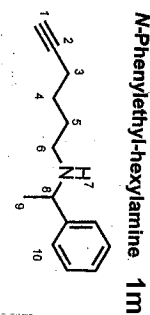
47.8061  
45.8757  
45.6391

24.6258  
24.6029  
24.5723  
18.2775  
18.0944

--- ACQUISITION PARAMETERS ---  
File Name = 14\_13c\_spectrum.1285  
Sample ID = S8713071  
Content = Single Pulse with Broad  
Creation Date = 11-APR-2005 18:11:50  
Revision Date = 14-APR-2005 19:53:34  
Spec File = K2P80  
Spec Type = 13C  
Data Format = ID COMPLEX  
Dimensions = 130  
F2 (Hz) = 125.777547 [MHz]  
D1a Size = 32768  
D1a Units = [ppm]  
Scans = 87  
Mod Return = 1  
X (Contam) = 13C [ppm]  
X (Contam) = 130 [ppm]  
X (Freq) = 125.777547 [MHz]  
X (Sweep) = 31.445408 [Hz]  
Solvent = CHLOROFORM-D  
Spin Set = 16 [Hz]  
Temp Set = 30 [C]  
Acq Start = 11:47:59.191  
Field Strength = 11.747357 [T]  
Filter Mode = SFTZSWHWH  
Filter Width = 15.7206221 [Hz]

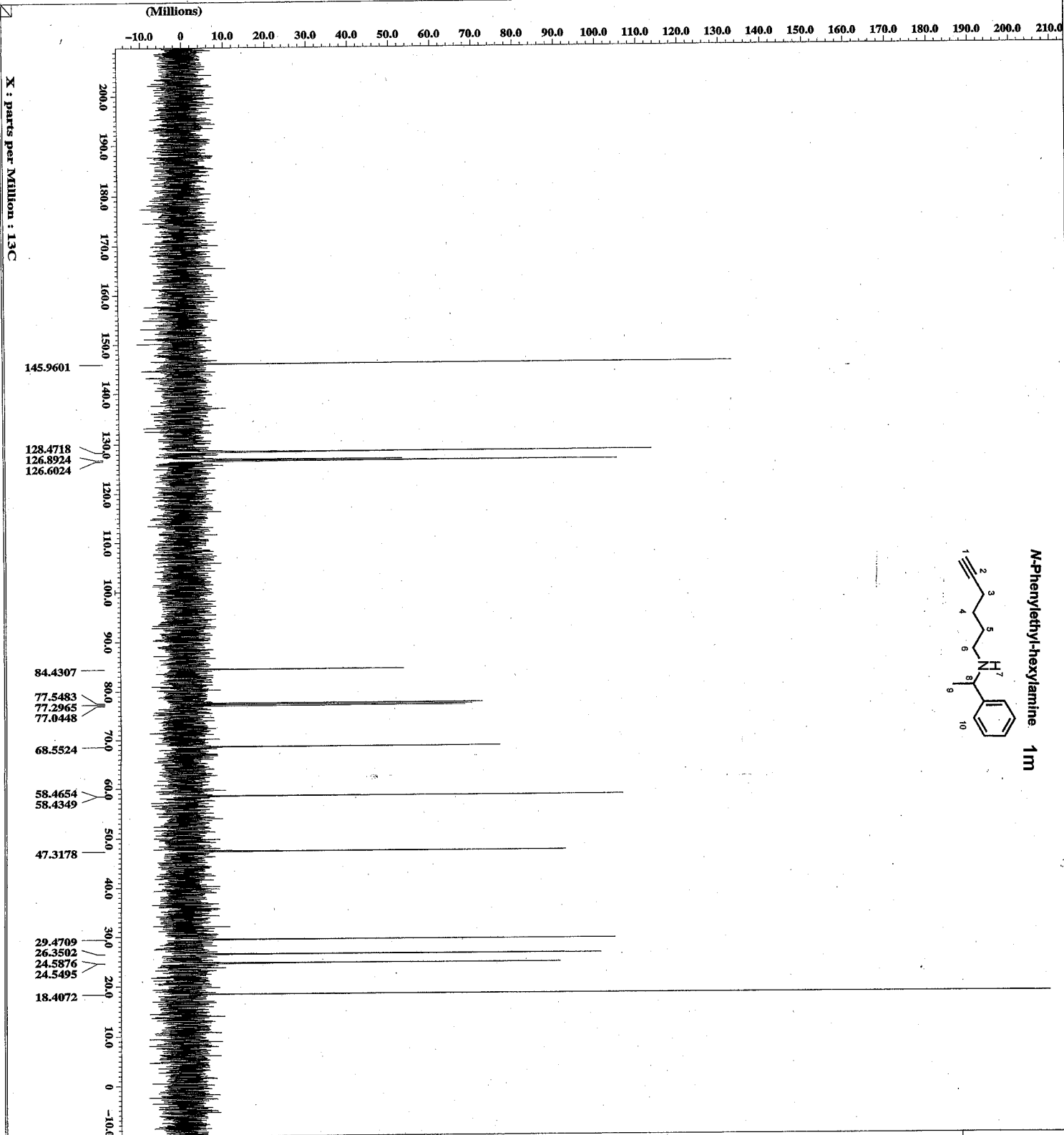
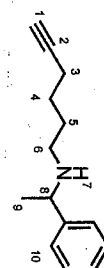


----- ACQUISITION PARAMETERS -----  
 File Name = 14\_Spectrum\_4626  
 Author =  
 Sample ID = 1  
 Content = Single Pulse Experiment  
 Creation Date = 11-APR-2005 14:20:08  
 Revision Date = 14-APR-2005 16:02:16  
 Spec Site = MCP500  
 Spec Type = 1D 1H NMR  
 Data Format = 1D COMPLEX  
 Data Size = 11  
 Data Title = 16364  
 Data Units = [ppm]  
 Scans = 1  
 Rod Return = 1  
 X Offset = 51ppm  
 X Sweep = 500.16241602 [MHz]  
 Solvent = 7.50750751 [Hz]  
 Solvent = CHLOROFORM-D  
 Spin Rate = 17 [Hz]  
 Recv Gain = 19.8 [dB]  
 Field Strength = 11.743579 [T]  
 Filter Mode = BRYANOMATH  
 Filter Width = 3.75119936 [kHz]



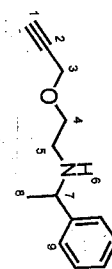


N-Phenylethyl-hexylamine 1m



--- ACQUISITION PARAMETERS ---  
 File Name = 1c\_13c\_spectrum.1280  
 Sample ID = 1  
 Content = Single pulse with broad  
 Creation Date = 13-APR-2005 14:24:13  
 Revision Date = 14-APR-2005 16:07:32  
 Spec Name = 1c\_13c  
 Spec Type = 13C  
 Data Format = ID COMPLEX  
 Dimensions = 130  
 Data Size = 13768  
 Data Units = [ppm]  
 Scans = 37  
 Mod return = 1  
 X domain = 130 [ppm]  
 X offset = 125.777547 [Hz]  
 X sweep = 31.4465408 [Hz]  
 Solvent = CHLOROFORM-D  
 Spill set = 17 [Hz]  
 Temp set = 25.1 [C]  
 Field strength = 125.767919 [MHz]  
 Filter mode = BPPZMCNCR  
 Filter width = 15.7206221 [Hz]

N-Phenylethyl-butyl-oxyethylamine 1n



X : parts per Million : 1H

7.3148  
7.3065  
7.2222

1.07623

3.8205

4.1429  
4.1337  
4.1291  
3.7727  
3.7599  
3.6087  
3.5758

0.9834

1.99619

2.6806  
2.6339  
2.4177  
2.4131  
2.4085

0.9654  
0.96089

0.91041

1.7030

0.84112

1.3613  
1.3476

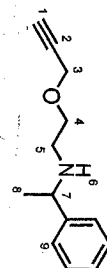
2.97677

-0.0084

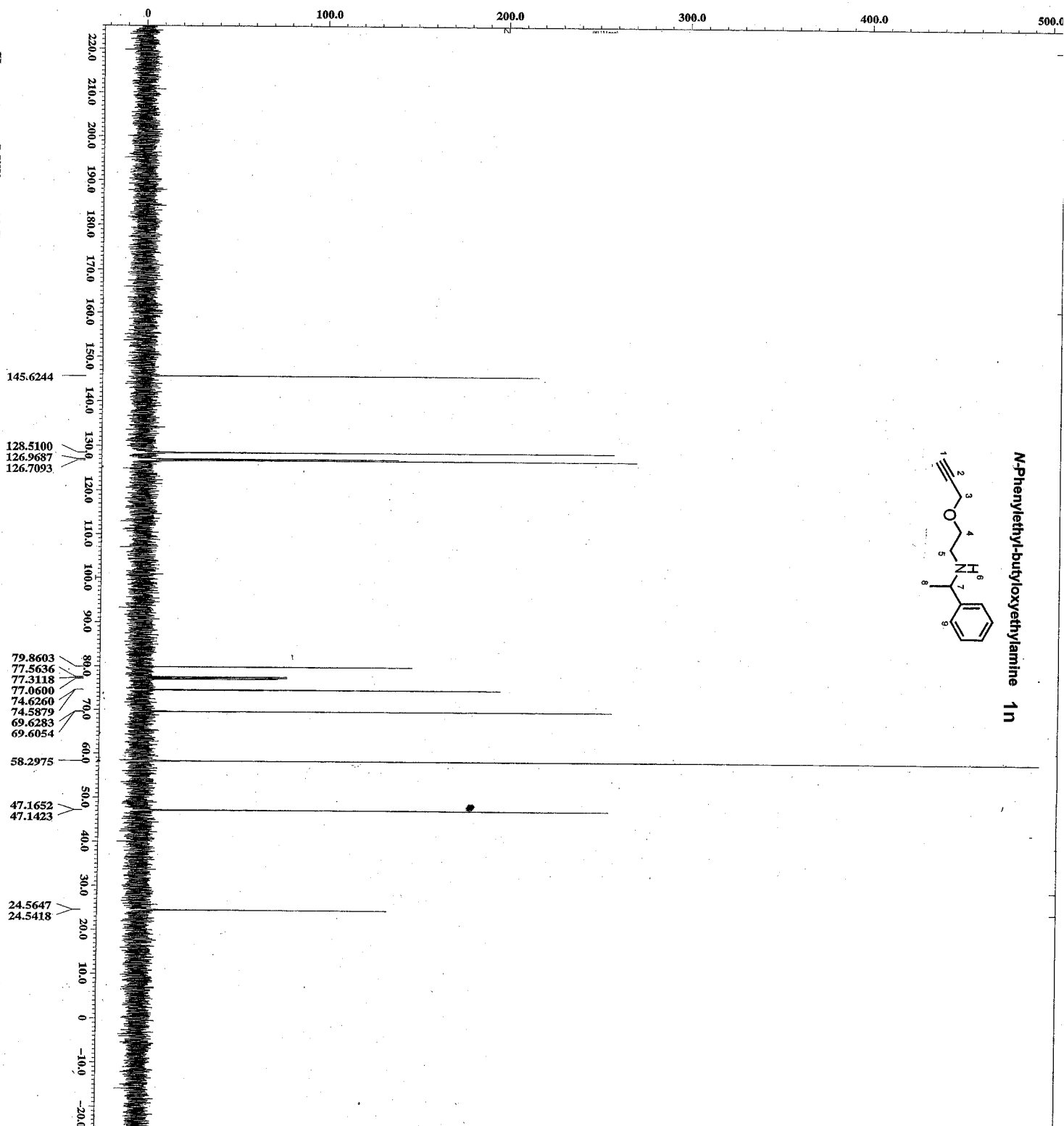
**JEOL**

ACQUISITION PARAMETERS  
File Name = ID Spectrum.2497  
Author =  
Sample ID = 002  
Content = Single Pulse Experiment  
Creation Date = 3-7PM-2005 22:23:36  
Revision Date = 4-PM-2005 21:48:58  
Spec Site = KCP500  
Spec Type = DEPTX 1H  
Data Format = ID COMPLEX  
Pulse Program = zgpg30  
D1m Units = [ppm]  
D1m Sls = 16384  
D1m Sls = [ppm]  
Scans = 4  
Mod Return = 1  
X Gain = 1H  
X Offset = 100MHz  
X Freq = 500.16241601MHz  
X Sweep = 7.507507511kHz  
Solvent = CHLOROFORM-D  
Spin Jet = 16Hz  
Recycle Delay = 21.4[sec]  
Field Strength = 11.747379T  
Field Mode = HYPERCROSS  
Filter Width = 3.7519361kHz

N-Phenylethyl-butyl-oxyethylamine 1n



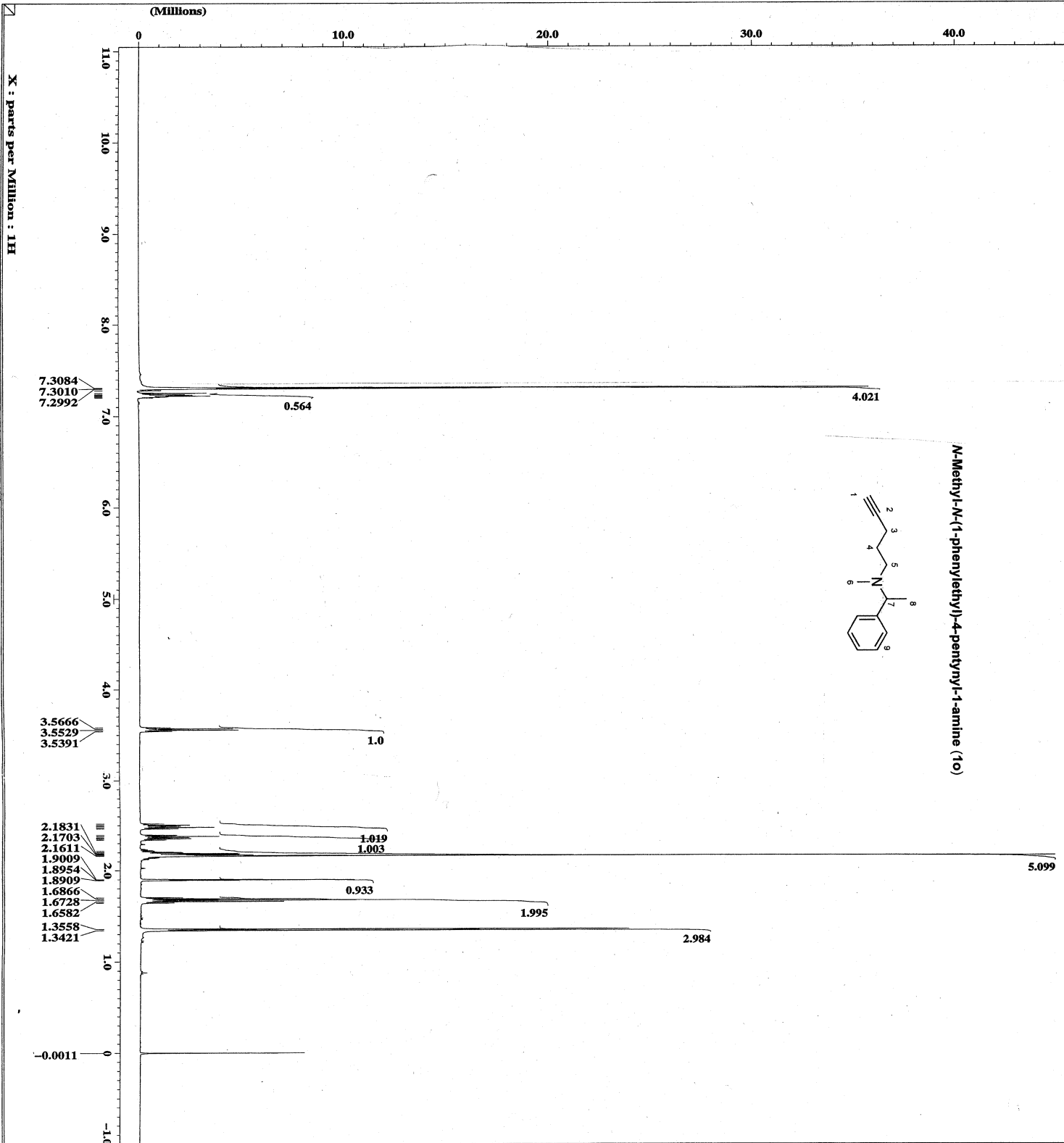
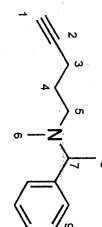
X : parts per Million : 13C



**JEOL**

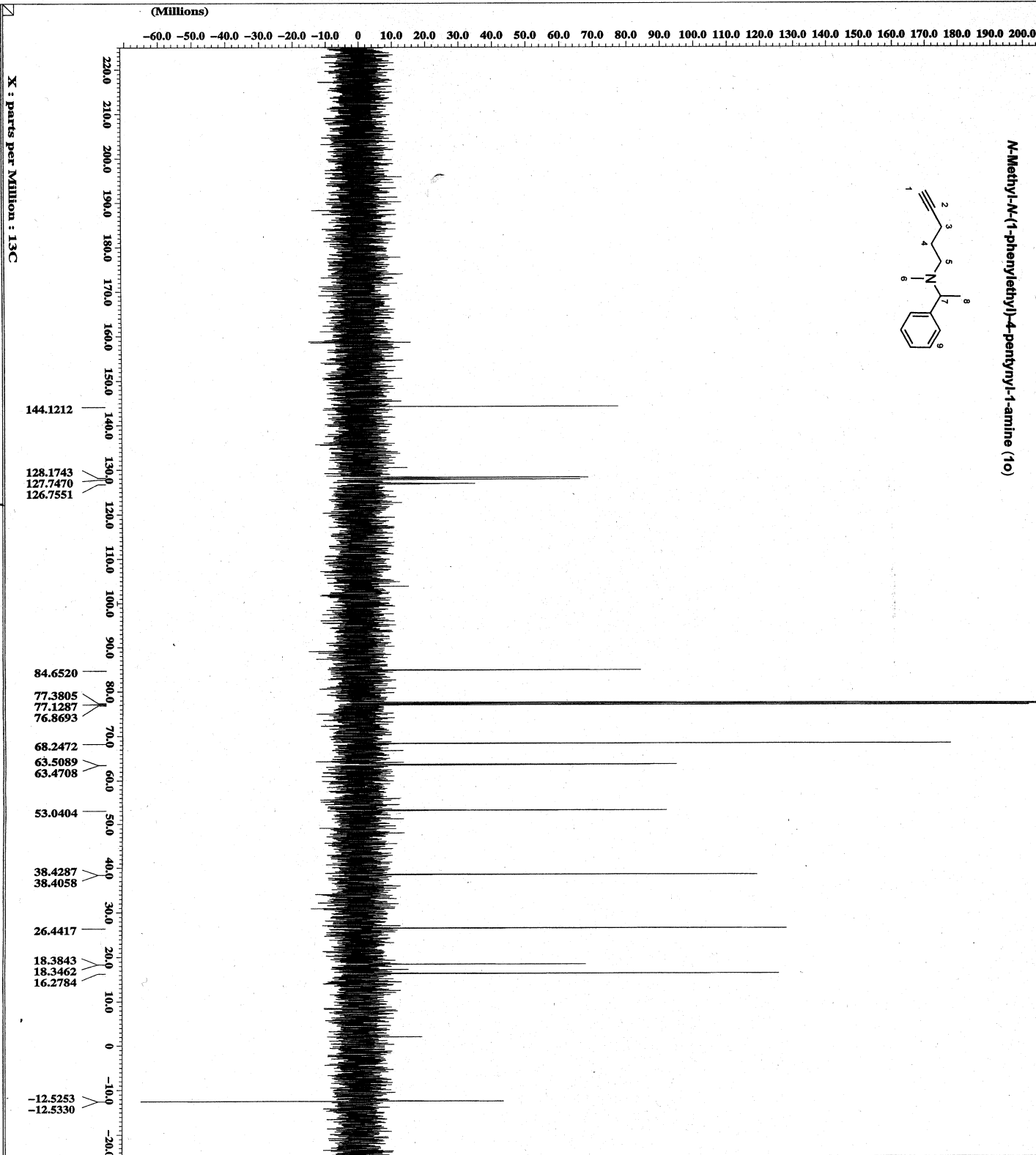
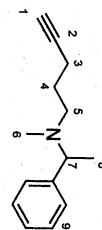
----- ACQUISITION PARAMETERS -----  
 File Name = 1d\_13c\_spectrum.828  
 Author =  
 Sample ID = 002  
 Content = Single Pulse with Broad  
 Creation Date = 3-FEB-2005 22:36:01  
 Revision Date = 4-FEB-2005 22:07:47  
 Spec Site = ECE500  
 Spec Type = DEPTA NMR  
 Spec Name = 13C NMR  
 Dimensions = 13C  
 Dim Title =  
 Dim Size = 32768  
 Dim Units = [ppm]  
 Scans = 43  
 X Domain = 13C  
 X Offset = 100 [ppm]  
 X Freq = 125.7778547 [MHz]  
 X Sweep = 31.44654088 [kHz]  
 Solvent = CDCl3  
 Temp Set = 29.2 [C]  
 Recv Gain = 30  
 Field Strength = 11.743579 [T]  
 Filter Mode = HYPERCROSS  
 Filter Width = 15.7206221 [kHz]

## N-Methyl-N-(1-phenylethyl)-4-pentenyl-1-amine (10)



----- ACQUISITION PARAMETERS -----  
 File Name = 1d\_spectrum.369  
 Author ID = phenylethyl-amine  
 Content = Single Pulse Experiment  
 Creation Date = 21-JUN-2006 10:57:18  
 Revision Date = 22-JUN-2006 20:38:21  
 Spec Site = KCP300  
 Spec Type = 1H  
 Data Format = ID COMPLEX  
 Dimensions = 1H  
 Num Fids = 16384  
 Num Spts = 16384  
 Num Dpts = 16384  
 Scans = 4  
 Mod Return = 1  
 X Domain = 1H  
 X Offset = 50.16241602 [MHz]  
 X Sweep = 7.50750751 [Hz]  
 Solvent = CHLOROFORM-D  
 Spin Gate = 17 [Hz]  
 Temp Set = 22.8 [C]  
 Temp Unit = C  
 Field Strength = 11.747379 [T]  
 Filter Mode = BUTTERWORTH  
 Filter Width = 3.7511936 [Hz]

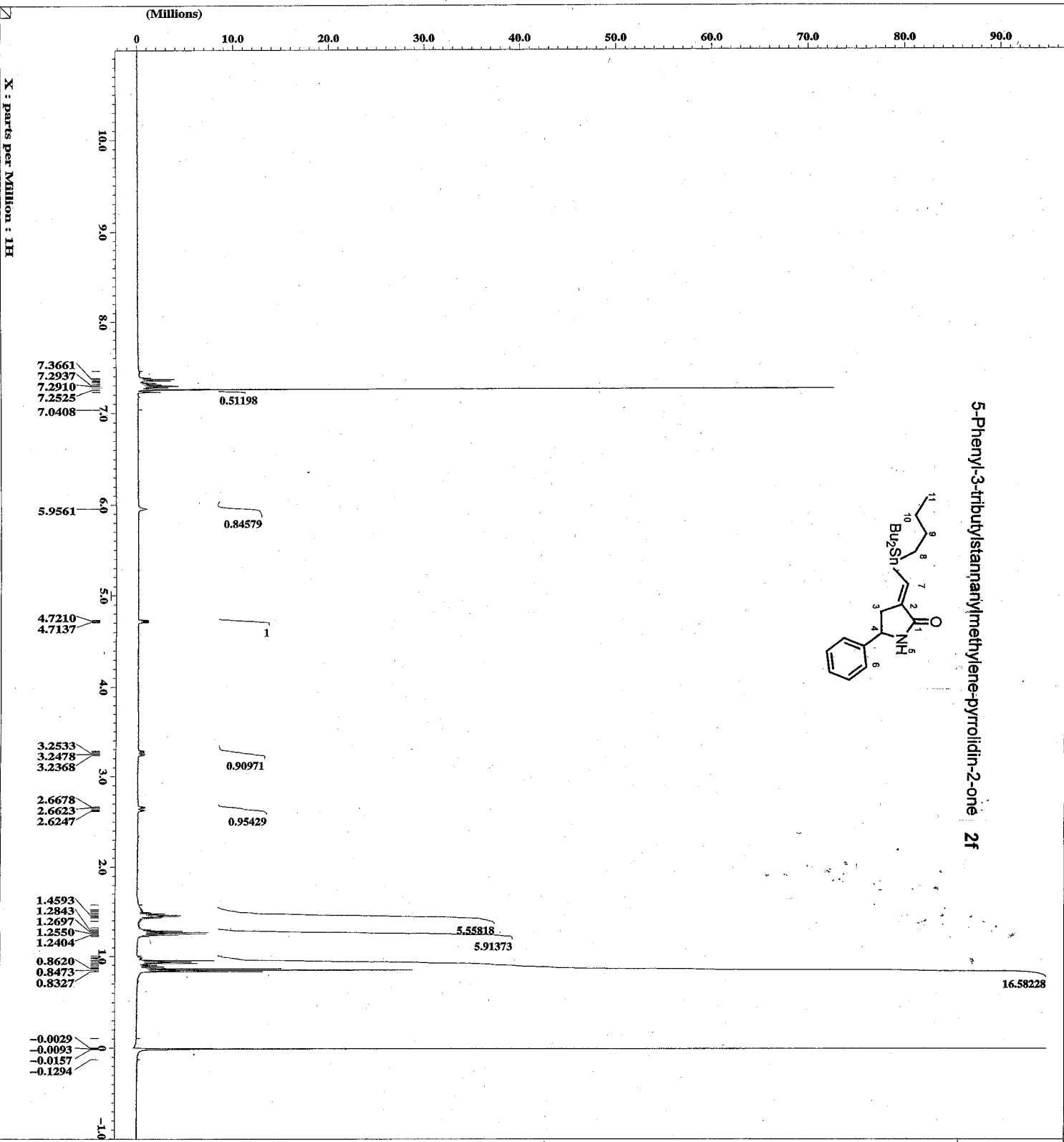
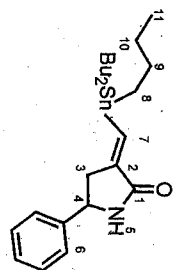
**N-Methyl-N-(1-phenylethyl)-4-pentenyl-1-amine (10)**



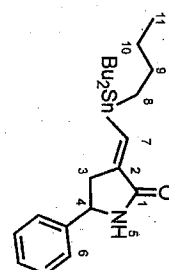
**JEOL**

----- ACQUISITION PARAMETERS -----  
 File Name = 1d\_13c\_spectrum\_306  
 Author = phylip@jeol.ac.uk  
 Sample ID = N-Methyl-N-(1-phenylethyl)-4-pentenyl-1-amine  
 Sample Name = N-Methyl-N-(1-phenylethyl)-4-pentenyl-1-amine  
 Creation Date = 21-JUN-2006 11:05:06  
 Revision Date = 22-JUN-2006 20:44:58  
 Spec Site = ECP500  
 Spec Type = 1D NMR  
 Data Format = ID COMPLEX  
 Dimensions = X  
 Dim 1 Title = 13C  
 Dim 1 Size = 32768  
 Dim 1 Units = ppm  
 Scans = 87  
 Mod. return = 1  
 X.domain = 13C  
 X.offset = 100 [ppm]  
 X.range = 128.1743-12.5253 [ppm]  
 X.steps = 314455408 [Hz]  
 Solvent = CHLOROFORM-D  
 Sp1n. get = 14 [Hz]  
 Temp. get = 23.4 [C]  
 Nuc1z. get = 13  
 Nuc1z. length = 747379 [Hz]  
 Filter mode = BUTTERWORTH  
 Filter width = 15.72065221 [Hz]

5-Phenyl-3-tributylstannanylmethylene-pyrrolidin-2-one 2f



ACQUISITION PARAMETERS  
 File Name = 1d\_spectrum\_4285  
 Author = 6-14-17  
 Date = 18-MAR-2005 10:41:42  
 Content = 18-MAR-2005 10:41:42  
 Creation Date = 18-MAR-2005 10:41:42  
 Revision Date = 18-MAR-2005 10:41:42  
 Spec Site = KCP500  
 Spec Type = 1D NMR  
 Data Format = 1D COMPLEX  
 Dimensions = 1H  
 Data Title = 16384  
 Data Size = 16384  
 Data Units = 8  
 Mod Return = 1  
 X Domain = 1H  
 X Offset = 500.16344602 [Hz]  
 X Range = 7.3661025 [Hz]  
 X Resol = 0.0001025 [Hz]  
 Solvent = CHLOROFORM-D  
 Spin Set = 16 [Hz]  
 Temp Set = 21.8 [degC]  
 Recv Gain = 29.747379 [V]  
 File Length = 11.743 [sec]  
 File Name = 1d\_spectrum\_4285  
 Filter Width = 3.7511936 [Hz]



**5-Phenyl-3-tributylstannanylmethylene-pyrrolidin-2-one 2f**



(Millions)

## 10.0

**20.0**

**30.0**

## 40.0

**50.0**

## 60.0

**70.0**

200.0  
190.0  
180.0  
170.0  
160.0  
150.0  
140.0  
130.0  
120.0  
110.0  
100.0  
90.0  
80.0  
70.0  
60.0  
50.0  
40.0  
30.0  
20.0  
10.0  
0

169.2243

**146.3340**

142.9996

134.9498

134.9040

100 0441

129.0441  
128.0827

125.8776

77.3576

77.1058

76.8464

54.4519

54.3985

**29.1657**

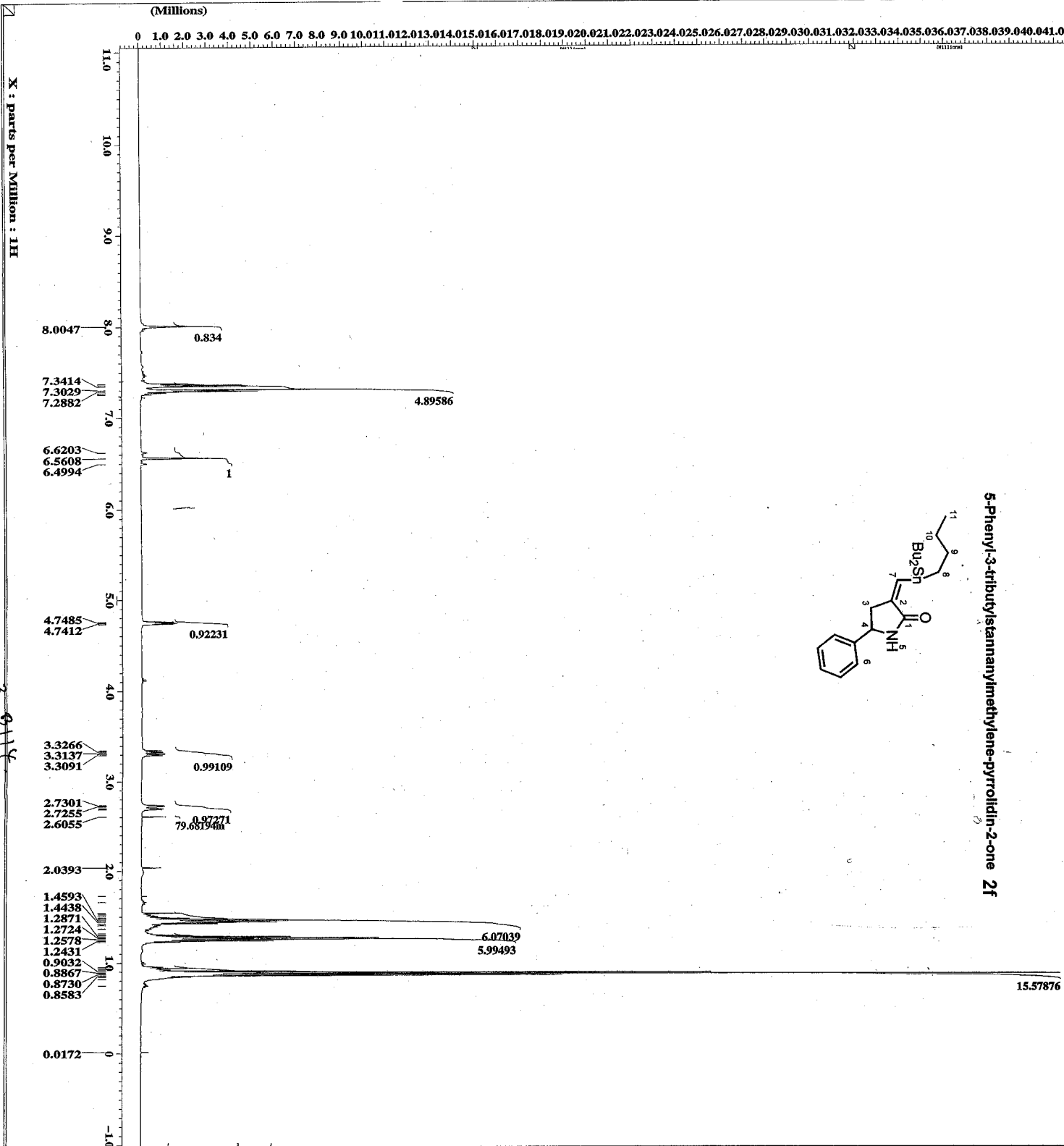
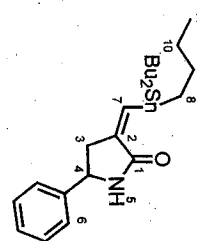
27.4718

27.3192

13.7070

9 7925

5-Phenyl-3-tributylstannanylmethylene-pyrrolidin-2-one 2f

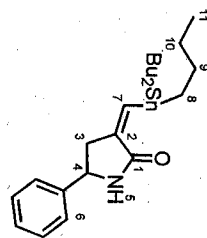


JEOL

----- ACQUISITION PARAMETERS -----  
 File Name = 1d\_spectrum.308  
 Author =  
 Sample ID = RD-019 EC3-10  
 Concentr = 5.000 g/100 ml  
 Creation Date = 29-JUN-2004 00:23:18  
 Revision Date = 29-JUN-2004 21:08:13  
 Spec Site = ECP500  
 Spec Type = 1H NMR  
 Spec Name = 1D CPMAS  
 Dimensions = 1H  
 Dia Title =  
 Dia Size = 16384  
 Dia Units = [ppm]  
 Mod Return = 1  
 X Domain = 1H  
 X Offset = 5.1 [ppm]  
 X Freq = 500.15241602 [MHz]  
 X Resolp = 7.00750751 [Hz]  
 X Resolw = 7.00750751 [Hz]  
 Spin Set = 16 [Hz]  
 Temp Set = 22.8 [deg]  
 Recv Gain = 13.747379 [v]  
 Field Strength = 11.747379 [v]  
 Filter Width = 3.7511936 [MHz]



5-Phenyl-3-tributylstannanylmethylene-pyrrolidin-2-one 2f



X : parts per Million : 13C

172.1238

144.4722  
143.3201  
138.7267  
138.6809

128.9068  
128.3955  
127.7775  
125.7937

77.4110  
77.1592  
76.8998

54.7114  
54.6580

39.6800

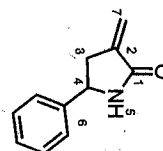
29.3641  
29.2801  
27.4642

13.9131  
13.0890  
11.6469  
10.2735

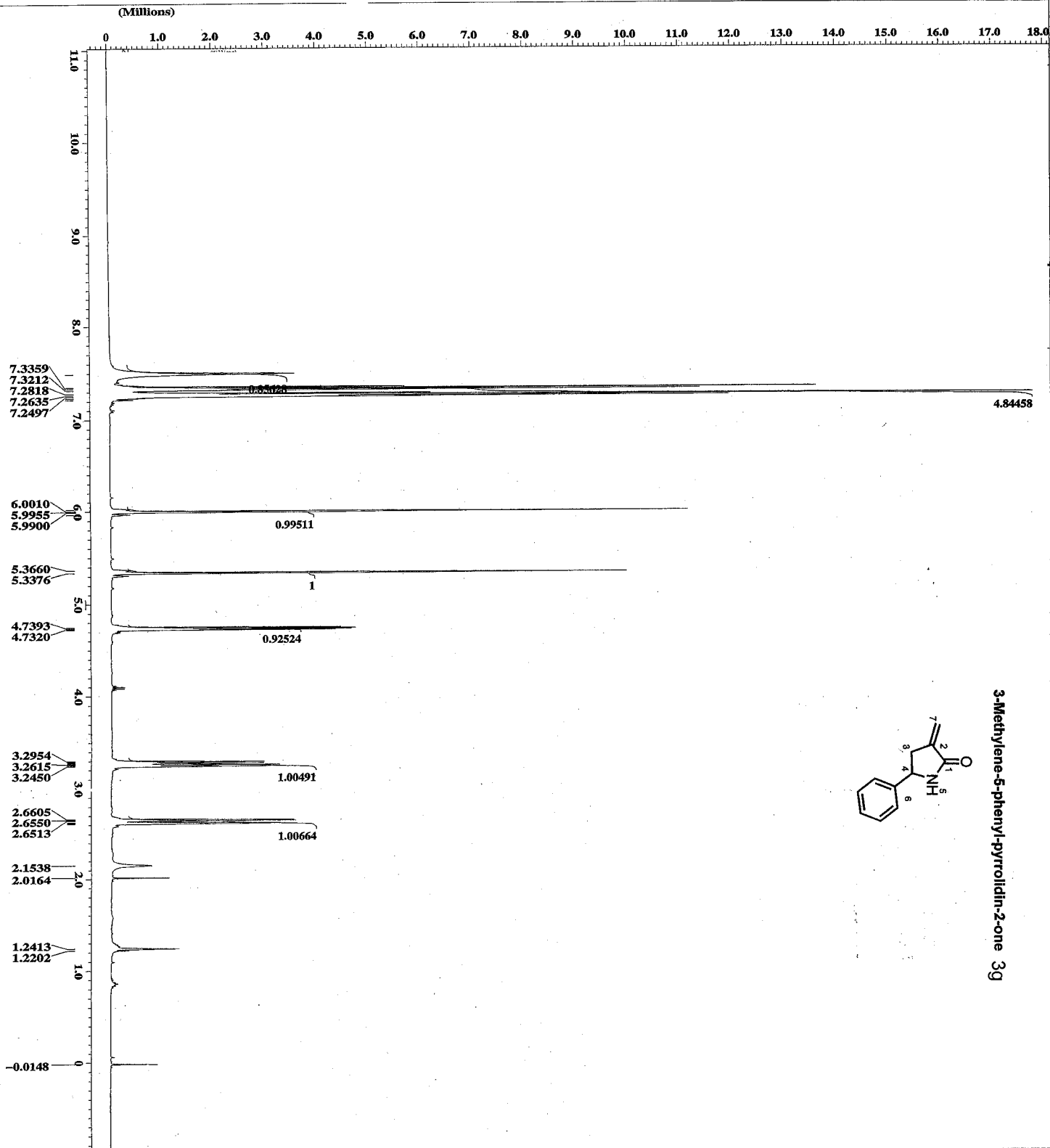
ACQUISITION PARAMETERS  
File Name = 1d\_13c\_spectrum\_63  
Author ID = npi-019 4-3-10  
Sample Name = Single pulse with broad  
Compound = 2f-2004 00:28:56  
Creation Date = 25-JUN-2004 21:09:40  
Revision Date = 25-JUN-2004 21:09:40  
Spec Site = ECP500  
Spec Type = 1D NMR  
Data Format = ID COMPLEX  
Dimensions = 1  
Dim Title = 13C  
Dim Size = 32768  
F2 Labels = 13C  
Scans = 150  
Mod return = 1  
X.domain = 13C  
X.offset = 100 [ppm]  
X.freq = 125.7777347 [MHz]  
X.res = 14.46581 [Hz]  
Solvent = CDCl3  
Solvant = CDCl3  
Solv get = 16 [Hz]  
Temp.get = 23.5 [C]  
Recv.get = 14  
Pulse.prog = zgpg30  
Pulse.length = 11.747357 [s]  
Pulse.power = 15.72065221 [dB]  
Filter.width = 15.72065221 [Hz]

JEOL

# 3-Methylene-5-phenyl-pyrrolidin-2-one 3g



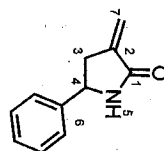
X : parts per Million : 1H



===== ACQUISITION PARAMETERS =====  
 File Name = 1d\_spectra.186  
 Author =  
 Sample ID = R01-020 2 fr-12  
 Content = Single Pulse Experiment  
 Creation Date = 30-JUN-2004 22:12:38  
 Revision Date = 1-JUN-2004 18:42:06  
 Spec Site = ECP500  
 Spec Type = 1D NMR  
 Data Format = ID COMPLEX  
 Dimensions = 1  
 Num Fids = 18  
 Num Sols = 1684  
 Num Dets = 8  
 Scans = 8  
 Mod. return = 1  
 X. offset = 10  
 X. freq = 500.162416021MHz  
 X. sweep = 7.507507511Hz  
 Solvent = CHLOROFORM-D  
 Spin. set = 15Hz  
 Temp. set = 12.31C  
 Recycle delay = 17  
 Field strength = 11.74735791G  
 Filter. mode = HUYENHOREN  
 Filter. width = 3.75119361Hz

**JEOL**

3-Methylene-5-phenyl-pyrrolidin-2-one 3g



X : parts per Million : 13C

(Millions)

-1.0 0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0 22.0

200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0

171.1929

142.8470

139.0472

129.0136

127.9988

125.7631

116.5002

116.4467

77.4339

77.1821

76.9227

54.9937

54.9479

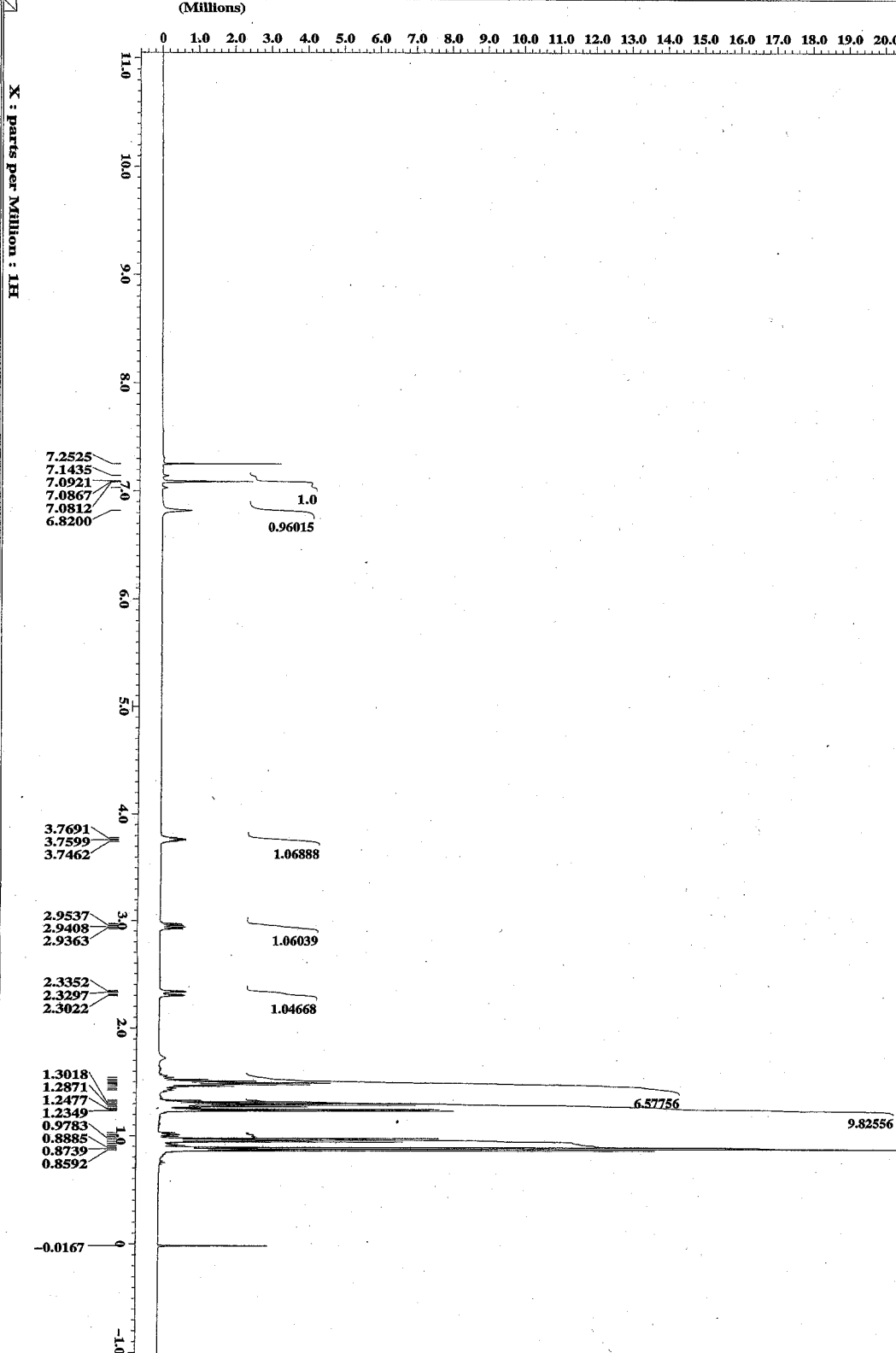
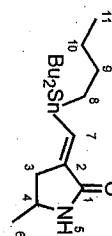
36.8874

--- ACQUISITION PARAMETERS ---  
 File Name = 10\_13c\_spectrum\_80  
 Sample ID = R01-010 2 f29-12  
 Content = Single Pulse with Broad  
 Creation Date = 30-JUN-2004 22:16:51  
 Acquisition Date = 1-JUN-2004 18:44:15  
 Spec Site = ECP300  
 Spec Type = DELTA\_NMR  
 Data Format = ID COMPLEX  
 Dimensions = 1D  
 F2 = 125.7631 MHz  
 F1 = 125.7631 MHz  
 Diam Size = 32768  
 Diam Units = [ppm]  
 Scans = 113.0  
 Mod\_return = 1  
 X\_offset = 100 [ppm]  
 X\_freq = 125.77787547 [MHz]  
 X\_sweep = 31.44654088 [kHz]  
 Solvent = CHLOROFORM-D  
 Spin\_set = 14 [Hz]  
 Temp\_set = 125 [C]  
 Field Strength = 11.7473579 [T]  
 Filter mode = BUTTERWORTH  
 Filter width = 15.7206221 [kHz]

JEOL

----- ACQUISITION PARAMETERS -----  
 File Name = 1d\_spectrum.4805  
 Author =  
 Sample ID = 0600  
 Sample Name = 5-Methyl-3-tributylstannanylmethylene-pyrrolidin-2-one 2h  
 Experiment = Single Pulse Experiment  
 Creation Date = 19-MAR-2005 16:55:38  
 Revision Date = 20-MAR-2005 18:44:08  
 Spec Site = ECP500  
 Spec Type = 1D NMR  
 Data Format = 1D COMPLEX  
 Dimensions = 1  
 Dia File =  
 Dia Size = 16384  
 Dia Units = [ppm]  
 Mod Return = 1  
 X Domain = 1H  
 X Offset = 5 [ppm]  
 X Freq = 500.15241602 [MHz]  
 X Sweep = 7.50756751 [Hz]  
 X Sweep = 7.50756751 [Hz]  
 Soln Set = 14 [Hz]  
 Temp Set = 22.2 [deg]  
 Recv Gain = 21  
 Field Strength = 11.7473579 [T]  
 Filter Scale = 10000000  
 Filter Width = 5.7513956 [kHz]

## 5-Methyl-3-tributylstannanylmethylene-pyrrolidin-2-one 2h

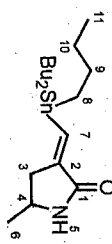


```

----- ACQUISITION PARAMETERS -----
File Name      = 1d_13c_spectrum.265
Sample ID      = RM2-084_f13-44
Content        = Single Pulse with Broad
Creation Date   = 10-MAR-2004 22:21:53
Revision Date   = 11-MAR-2004 15:49:13
Spec Site      = ECP500

Spec Type      = DELTA NMR
Data Format     = 1D COMPLEX
Dimensions     = 1
Num Fids      = 1368
Dim 1 Size    = 1368
Dim 2 Size    = 1368
Dim 3 Size    = 1368
Dim 4 Size    = 1368
Dim 5 Size    = 1368
Dim 6 Size    = 1368
Dim 7 Size    = 1368
Dim 8 Size    = 1368
Dim 9 Size    = 1368
Dim 10 Size   = 1368
Dim 11 Size   = 1368
Dim 12 Size   = 1368
Dim 13 Size   = 1368
Mod. return    = 1
X_domain       = 130
X_offset       = 130
X_resolution   = 130
X_sweep        = 130
Solvent        = CH2OPOPOH-D
Spin. proc     = 10 Hz
Temp. set      = 24.1 (C)
Acq. gain      = 19
Acq. length    = 13.747376 (s)
Filter mode     = HETCOR2DPR
Filter width    = 15.7206221 (Hz)
  
```

5-Methyl-3-tributylstannanylmethylene-pyrrolidin-2-one 2h



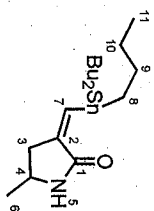
X : parts per Million : 13C

168.9191  
147.3411  
133.7061  
77.3500  
77.0905  
76.8387

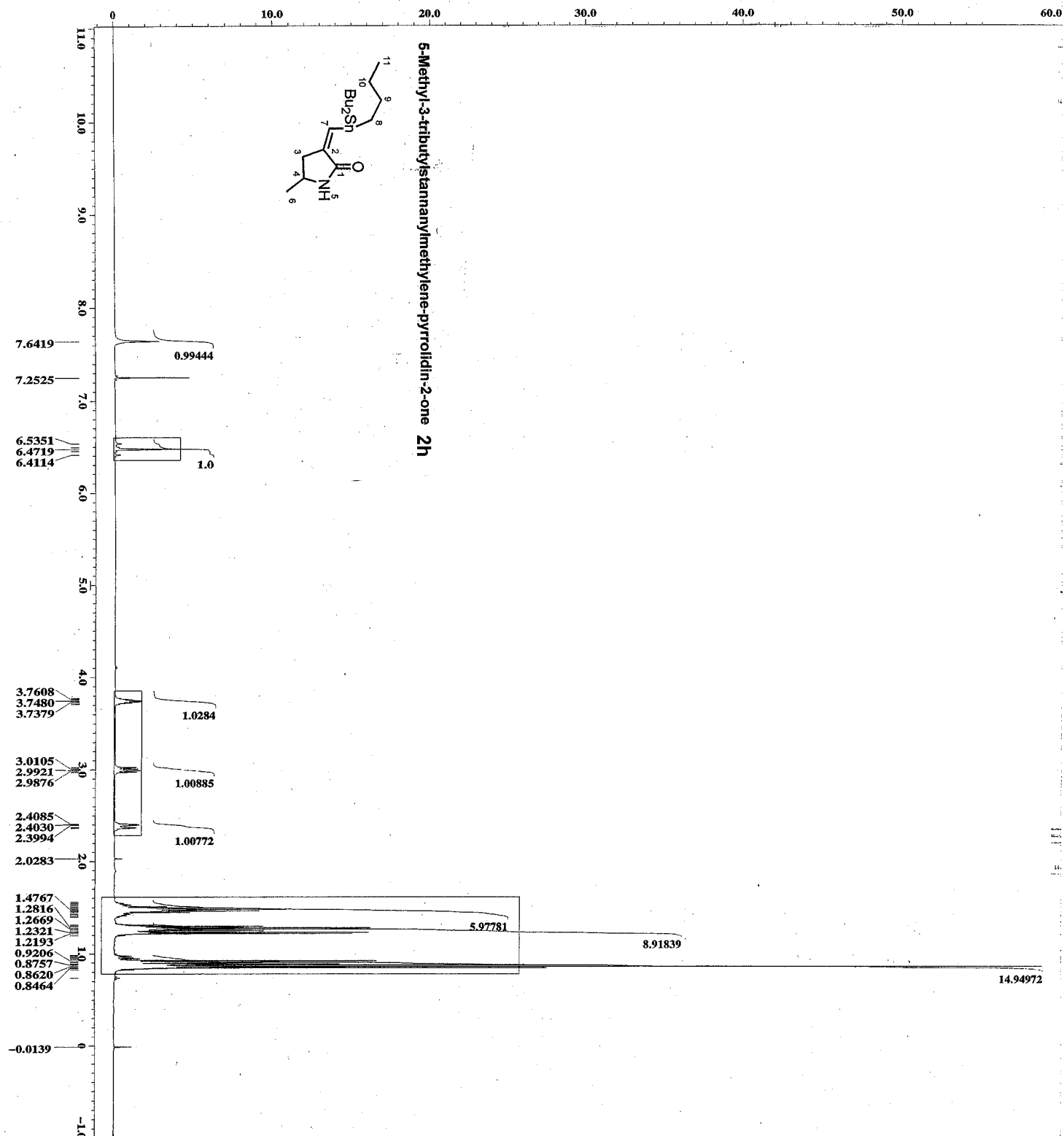
46.3335  
37.2078  
29.2038  
27.3573  
23.2142  
13.7299  
9.7775

--- ACQUISITION PARAMETERS ---  
 File Name = ID\_spectrum.1075  
 Author =  
 Sample ID = RM2-084\_f15-27\_HPLC  
 Content = Single Pulse Experiment  
 Creation Date = 24-MAR-2004 14:53:17  
 Acquisition Date = 25-MAR-2004 08:37:16  
 Spec Date = RM2500  
 Spec Type = DEPTA NMR  
 Data Format = 1D COMPLEX  
 Dimensions = X  
 Num Files = 16384  
 File Size = 16384  
 Num Data = 16384  
 Scans = 8  
 Mod\_return = 1  
 X\_domain = 1H  
 X\_offset = 100MHz  
 X\_freq = 500.136416021MHz  
 X\_resol = 7.507507511Hz  
 Solvent = CHLOROFORM-D  
 SpIn\_gac = 10Hz  
 Temp\_gac = 23.2(C)  
 Temp\_mon = 23.2(C)  
 Field\_strength = 12.747357917  
 Filter\_mode = HETERONORM  
 Filter\_width = 3.751193616Hz

5-Methyl-3-tributylstannanylmethylene-pyrrolidin-2-one 2h

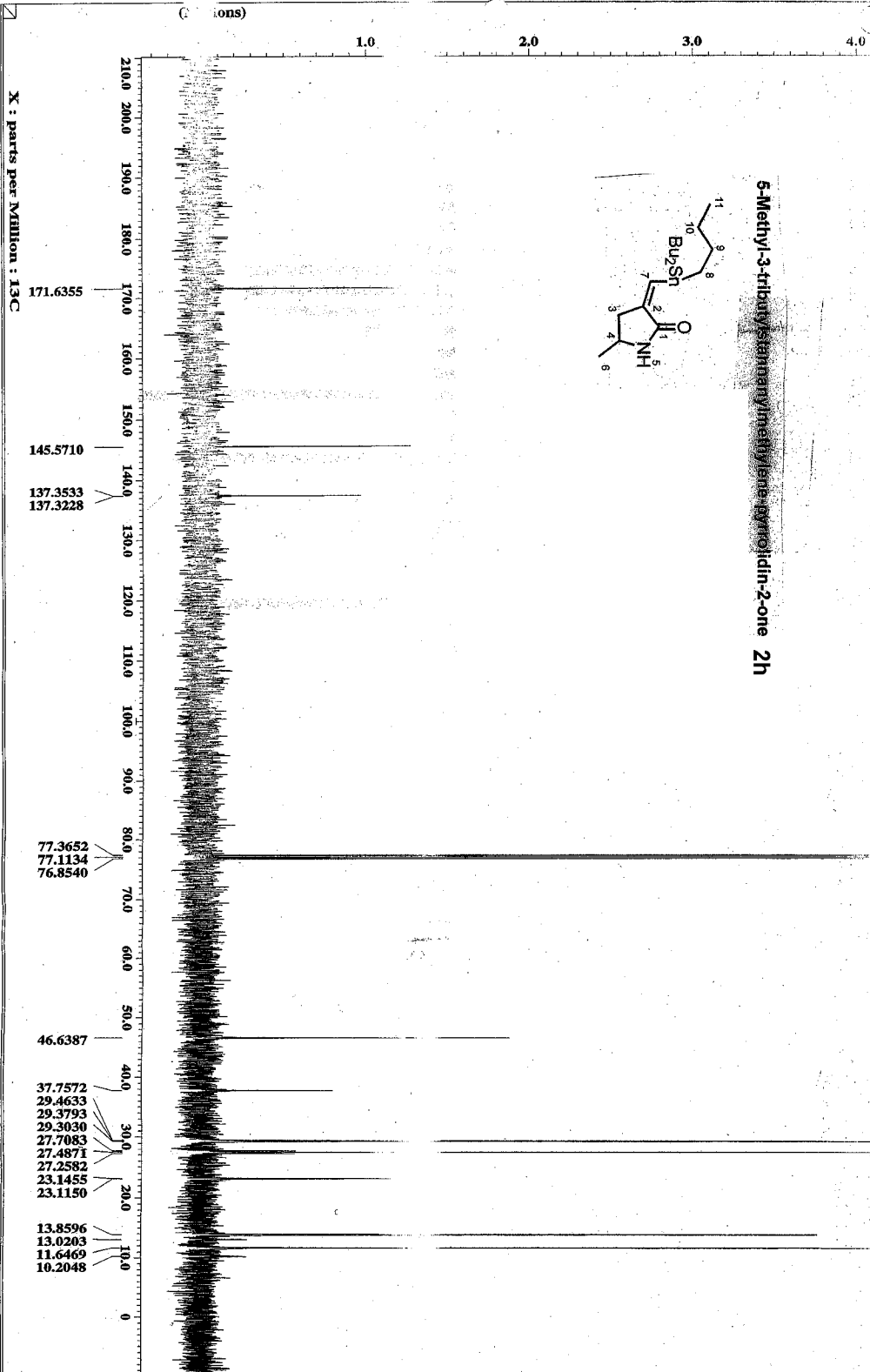
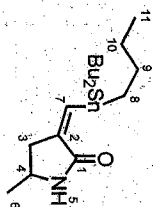


X : parts per Million : 1H

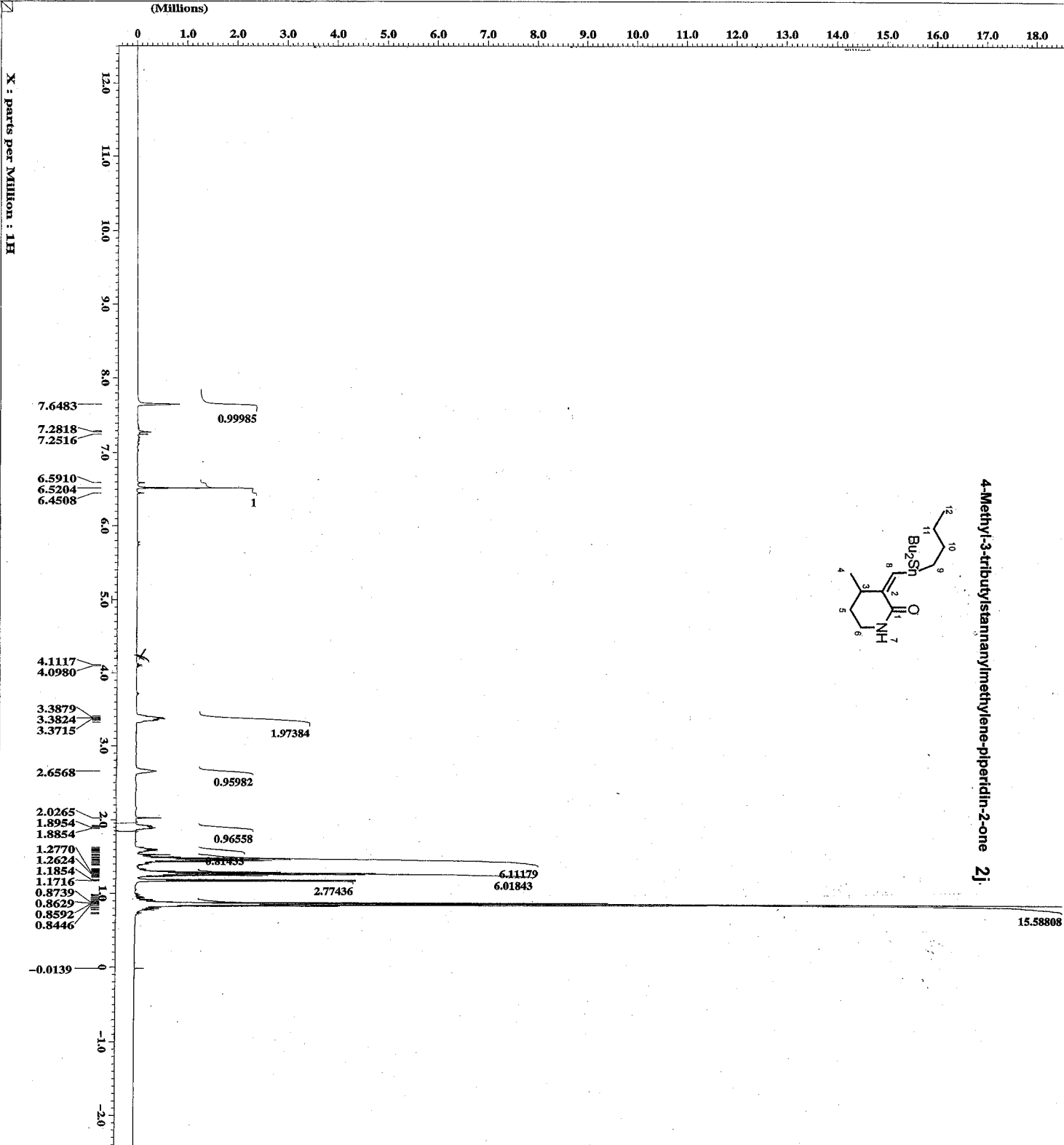
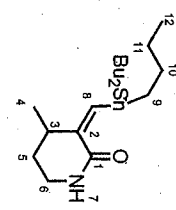


ACQUISITION PARAMETERS  
 File Name = id\_13c\_spectrum\_172  
 Author ID =  
 Sample ID = 001-016 #47-17  
 Content = Single Pulse 5th Broad  
 Creation Date = 25-MAY-2004 21:13:15  
 Revision Date = 26-MAY-2004 17:07:10  
 Spec Site = ECP500  
 Spec Type = 1D NMR  
 Data Format = ID COMPLEX  
 Dimensions = 1  
 Dia Title = 13C  
 Dia Size = 12764  
 Dia Units = 19cm  
 Mod Return = 13C  
 X Domain = 100 [ppm]  
 X Offset = 3.77787547 [Hz]  
 X Freq = 125.760481 [MHz]  
 Solvent = CDCl3  
 Spn. ref = 44 [Hz]  
 Tmco. ref = 23.2 [Hz]  
 Recv. gain = 11.743359 [V]  
 Field strength = 125.760481 [MHz]  
 Filter width = 15.72066221 [Hz]

5-Methyl-3-tributylstannylmethylene-pyrrolidin-2-one 2h



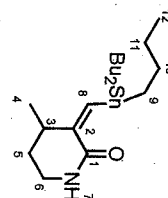
## 4-Methyl-3-tributylstannanylmethylene-piperidin-2-one 2j



--- ACQUISITION PARAMETERS ---  
 File Name = 10\_spectra.373  
 Author =  
 Sample ID = 4-4-11  
 Content = Single Pulse Experiment  
 Creation Date = 21-Feb-2005 21:58:08  
 Revision Date = 22-Feb-2005 21:44:26  
 Spec Site = ECP500  
 Spec Type = DEVEL NMR  
 Data Format = ID COMPLEX  
 Data File = 10  
 Dim File = 16384  
 Dim Size = [ppm]  
 Scans = 4  
 Mod Return = 1  
 X Offset = 10  
 X Freq = 500.16241602 [MHz]  
 X Sweep = 7.50750751 [Hz]  
 Solvent = CHLOROFORM-D  
 Spin Set = 13 [Hz]  
 Recycle Del = 11.3 [Sec]  
 Field Strength = 11.743579 [T]  
 Filter Mode = HETEROGENE  
 Filter Width = 3.7511936 [Hz]



4-Methyl-3-tributylstannanylmethylene-piperidin-2-one 2j



X : parts per Million : 13C

167.1490

148.3712  
143.8694  
143.8313

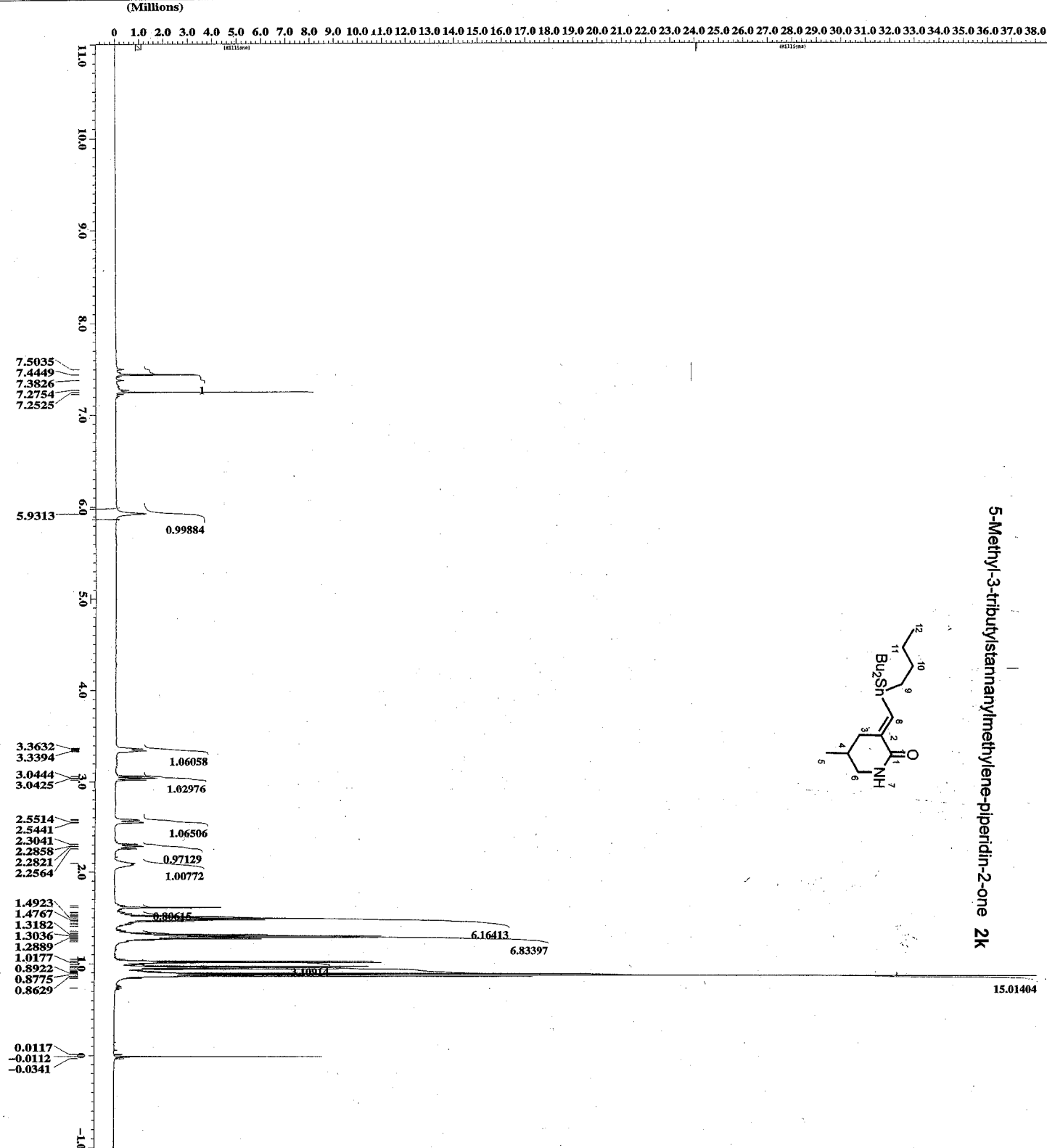
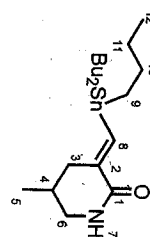
77.3728  
77.1211  
76.8693

40.0539  
35.8726  
30.7222  
29.5167  
29.4327  
29.3564  
27.5634  
27.3116  
19.7120  
13.8978  
13.5697  
13.5010  
12.1047  
10.7084  
10.6397

ACQUISITION PARAMETERS  
File Name = 1d\_13c\_spectrum.1011  
Author =  
Sample ID = 424-11  
Conc = Single Pulse v4.1b Broad  
Creation Date = 21-FEB-2005 21:02:02  
Revision Date = 22-FEB-2005 21:49:24  
Spec Site = ECF500  
Spec Type = 1D NMR  
Data Format = 1D COMPLEX  
Dimensions = X  
Dim Title = 13C  
Dim Size = 32768  
Dim Units = [ppm]  
Mod Return = 103  
X Domain = 13C  
X Offset = 100 [ppm]  
X Freq = 125.77787547 [MHz]  
X Sweep = 31.44654088 [Hz]  
X Channel = 13C NMR-D  
Solv Int = 13 [Hz]  
Temp Set = 21.9 [degC]  
Recvr Gain = 14  
Field Strength = 11.7473579 [T]  
Filter Mode = HETCOR/NOESY  
Filter Width = 15.72062 [kHz]

JEOL

5-Methyl-3-tributylstannanylmethylene-piperidin-2-one 2k



15.01404

**JEOL**

----- ACQUISITION PARAMETERS -----  
 File Name = 1d spectrum.1200  
 Author =  
 Sample ID = R01-001 427-33 BRIC  
 Compound = Single Pulse Experiment  
 Creation Date = 3-MAR-2004 10:00:59  
 Revision Date = 10-APR-2004 05:14:52  
 Spec file = E03500  
 Spec type = DEPTA\_NMR  
 NMR type = 1D  
 Dimensions = 1H  
 Dim 1 file = 16384  
 Dim 2 file = 16384  
 Dim 3 file = 16384  
 Dim 4 file = 16384  
 Dim 5 file = 16384  
 Dim 6 file = 16384  
 Dim 7 file = 16384  
 Dim 8 file = 16384  
 Dim 9 file = 16384  
 Dim 10 file = 16384  
 Dim 11 file = 16384  
 Dim 12 file = 16384  
 X offset = 500.16241602 (Hz)  
 X freq = 500.16241602 (Hz)  
 X sweep = 7.50750751 (Hz)  
 Solvent = CDCl3  
 Temp deg = 23.1 (C)  
 Recv gain = 23.1 (C)  
 Field strength = 11.7473579 (T)  
 Filter mode = HYPERNOISE  
 Filter width = 3.7511936 (Hz)



77.3347  
77.0829  
76.8311

**49.4618**

40.5193

29.6693  
29.2267  
27.3802

18.3767

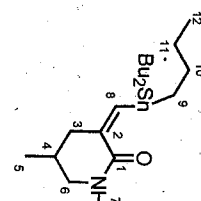
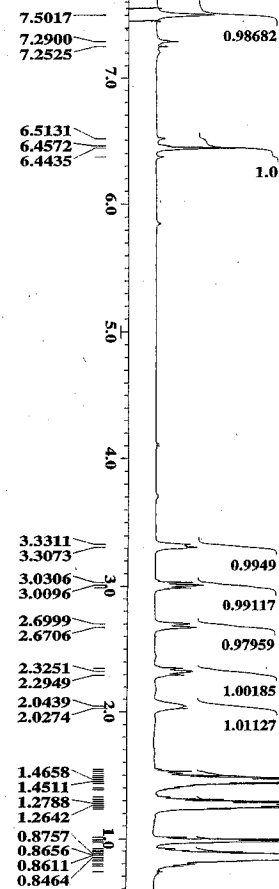
13.7299  
13.6384  
10.1972

0.0644

FILE NAME	ACQUISITION PARAMETERS
AUTHOR ID	AD 130, spectrum 362
CONTENT	BD-01, HPGC
CREATION DATE	Single Pulse with Broad
CREATION DATE	= 11-APR-2004 06:58:50
REVISION DATE	= 11-APR-2004 04:34:36
SPEC SLICE	REC500
SPEC TYPE	DETAILED
DATA FORMAT	X
PARAMETERS	X
DATA SLICE	32766
DATA UNITS	[ppm]
MOD RETURN	15000
MOD RETURN	1
MOD RETURN	10 [ppm]
MOD RETURN	= 123.77787547 [Hz]
MOD RETURN	= 31.44654088 [Hz]
MOD RETURN	= CHROMOPORM-D
MOD RETURN	10 [Hz]
MOD RETURN	= 30
MOD RETURN	= 11.747579 [Hz]
MOD RETURN	= BUTTERWORTH
MOD RETURN	= 5.72066221 [Hz]

0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0 29.0 30.0 31.0

**X : parts per Million : 10**



**5-Methyl-3-tributylstannanylmethylene-piperidin-2-one 2K**

15.76164

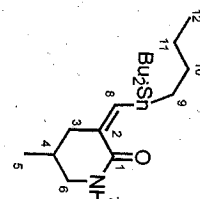
```

=====
File Name          = 14 spectrum.1165
Sample ID          = BH-01-#5-12
Content            = Single Pulse Experiment
Creation Date      = 6-MAR-2004 19:06:46
Spec Slice        = 825900
Revision Number    = 8-MAR-2004 14:07:17
=====
Spec Type          = 1D NMR
Data Format         = 1D COMPLEX
Dimensions         = 16384
Dir Size           = 16384
Data Units        = [nm]
Scan              = 8
Mod. Return        = 1
X offset           = 0
X Freq             = 500.1634602 [MHz]
X sweep           = 1.807507511 [kHz]
Solvent            = CHLOROFORM-D
Temp. Set          = 12.1 [deg]
Recvr. gain        = 11.7473579 [V]
Field strength     = 8.975959 [T]
Pulse mode         = BPPHPPHPPHPPH
Filter mode        = 3.75119396 [kHz]
=====

```



5-Methyl-3-tributylstannanylmethylene-piperidin-2-one 2k



166.9735

146.3645  
146.3340  
142.3587

77.3652  
77.1134  
76.8540

49.3703

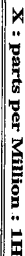
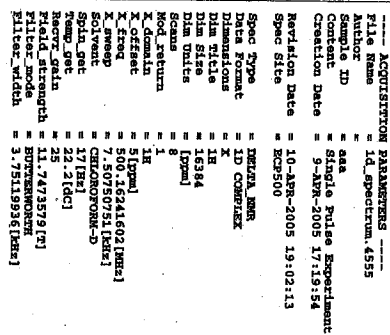
41.4731

29.4480  
29.3793  
27.5634  
27.3345  
18.2241  
13.8825  
13.4476  
13.3789  
11.9750  
10.5787  
10.5100

===== ACQUISITION PARAMETERS =====  
File Name = 1d\_13c\_spectrum\_347  
Author =  
Sample ID = RUI-001 f45-12  
Content = Single Pulse with Broad  
Creation Date = 7-APR-2004 19:11:01  
Revision Date = 8-APR-2004 14:09:29  
Spec Site = KCP500  
Spec Type = DELTA HR  
Data Format = ID COMPLEX  
Dimensions = 130  
F2 = 125.77781547 [MHz]  
Dim Size = 32768  
Dim Units = [ppm]  
Scans = 110  
Mod\_return = 1  
X\_domain = 130 [ppm]  
X\_offset = 125.77781547 [MHz]  
X\_freq = 31.44540861 [kHz]  
X\_sweep = CHLOROFORM-D  
Solvent =  
Splt\_gct = 7 [Hz]  
Temp\_get = 23.1 [deg]  
Acq\_date = 20040407  
Field\_strength = 11.747379 [T]  
Filter\_mode = BUTTERWORTH  
Filter\_width = 15.7206221 [kHz]

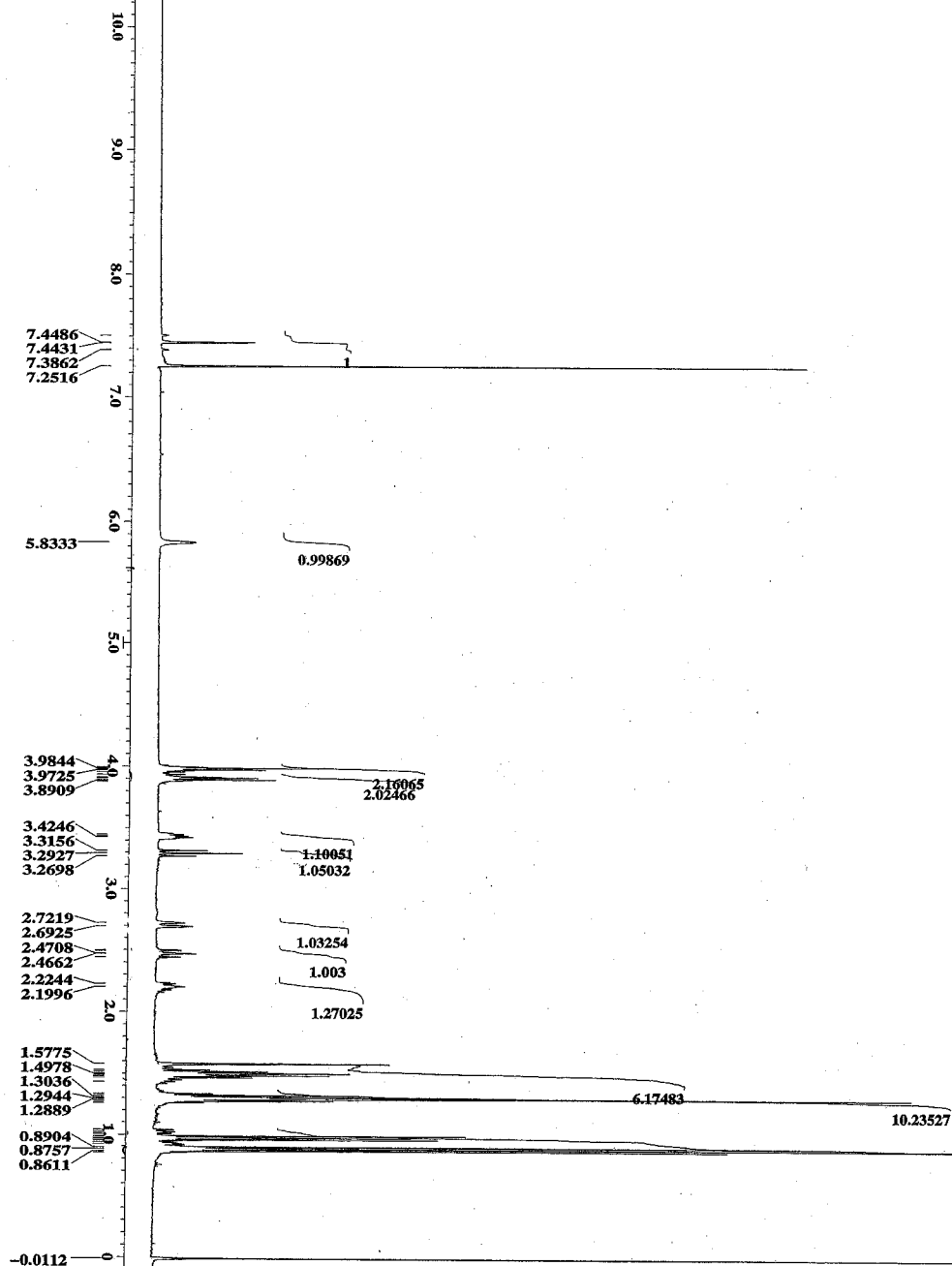
**JEOL**

16.85927

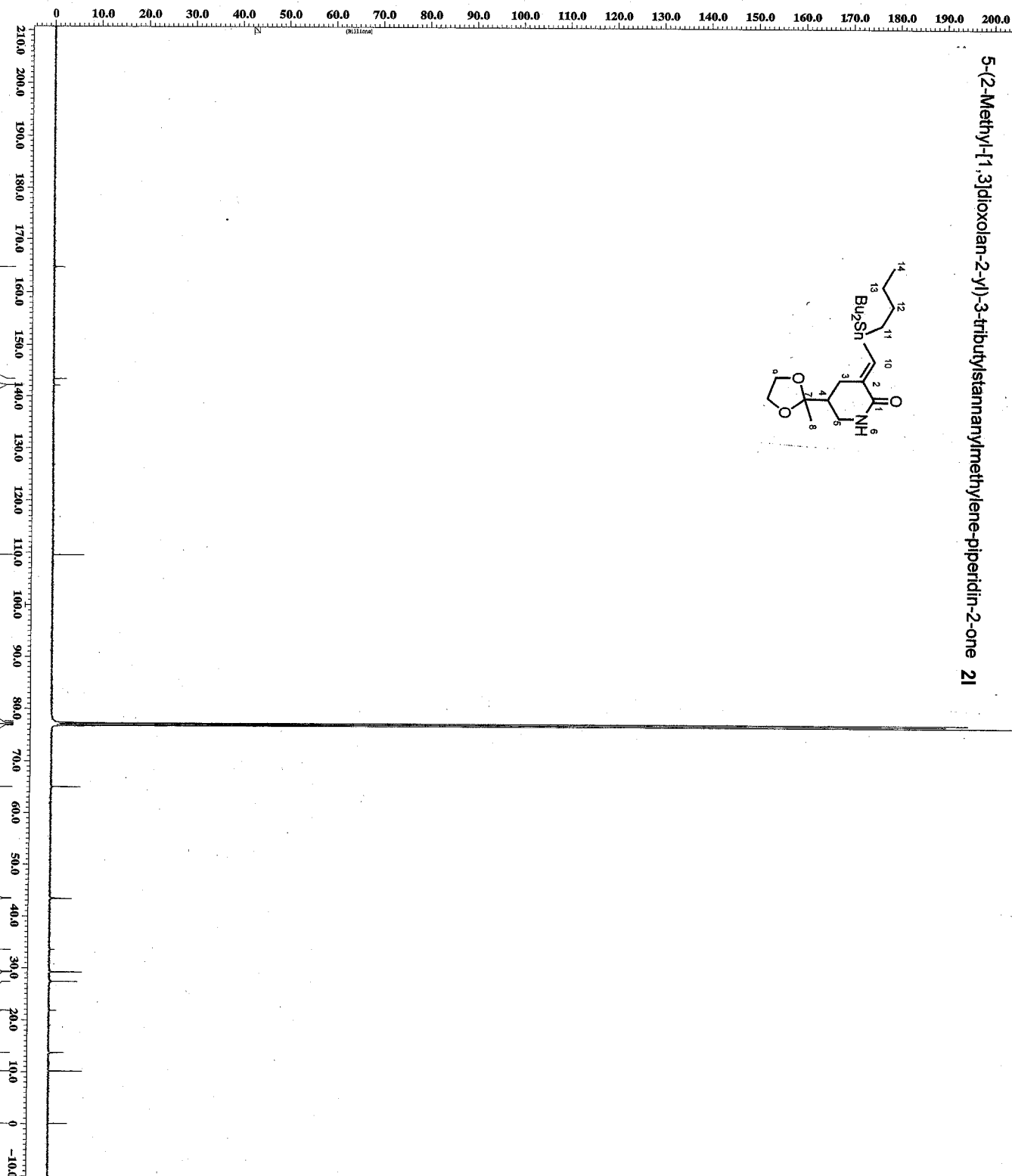
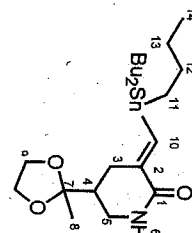


(Millions)

0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0 22.0 23.0 24.0 25.0 26.0 27.0 28.0 29.0 30.0 31.0 32.0 33.0 34.0 35.0 36.0



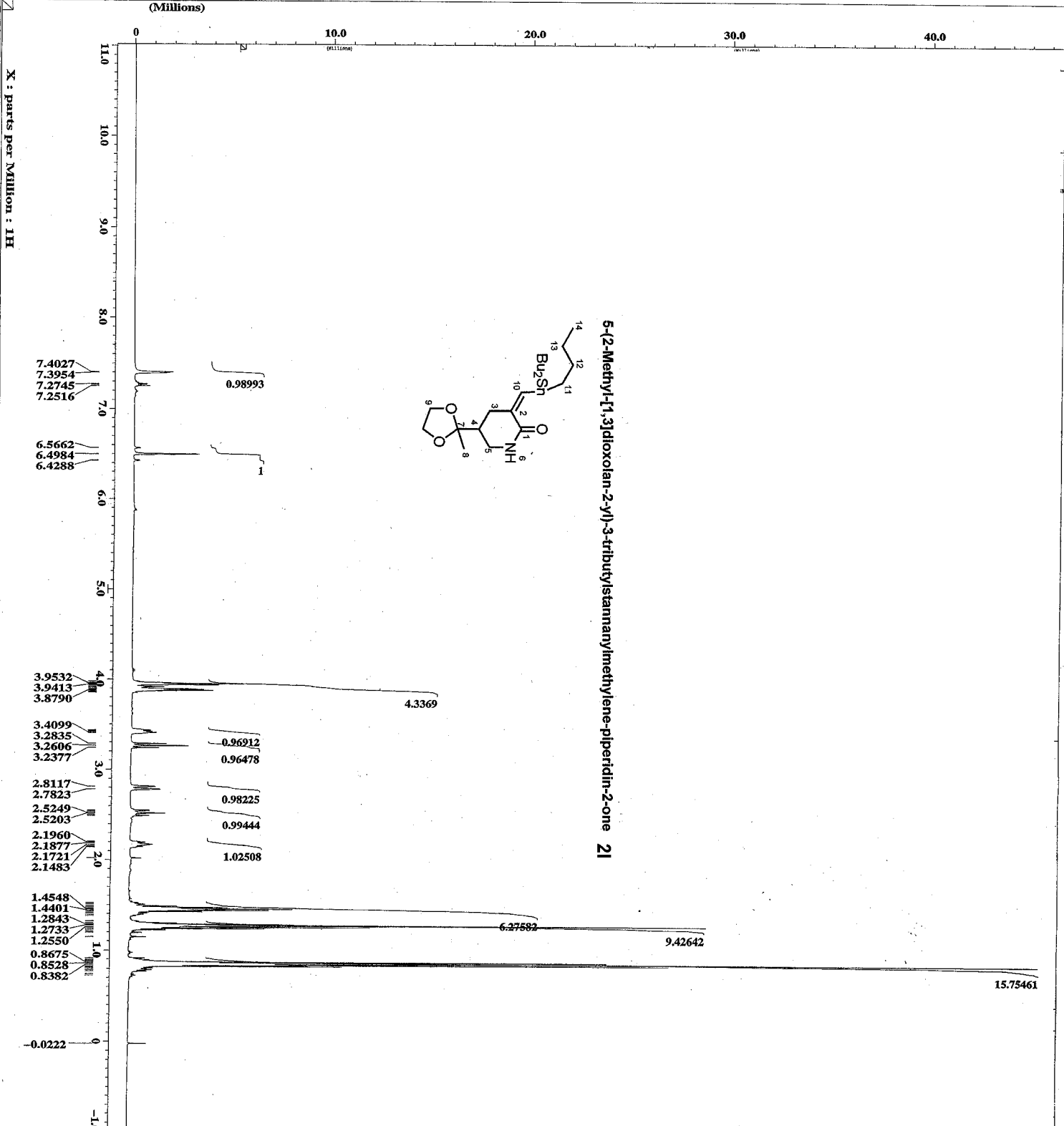
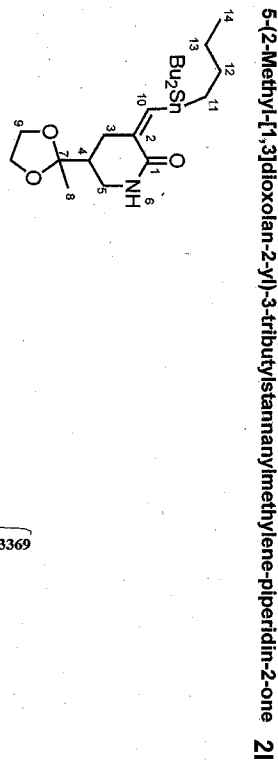
5-(2-Methyl-1,3-dioxolan-2-yl)-3-tributylstannanylmethylene-piperidin-2-one 21



**JEOL**

ACQUISITION PARAMETERS  
 File Name = 1d\_13c\_spectrum.1262  
 Author = f-actyl-8-  
 Sample ID = Sample 13c with Broad  
 Content = 11-Apr-2005 10:02:08  
 Revision Date = 12-Apr-2005 11:40:39  
 Spec Site = BCP500  
 Spec Type = DELTA 500  
 Data Format = ID COMPLEX  
 Dimensions = 1  
 Data File = 13c  
 Data Size = 32768  
 Data Units = 59034  
 Scans = 1  
 Mod. Return = 13c  
 X. Domain = 100 [ppm]  
 X. Offset = 128.7781847 [Hz]  
 X. Freq = 125.7614508 [MHz]  
 Solvent = CHLOROFORM-D  
 Spin. Jet = 15 [Hz]  
 Temp. Jet = 23.6 [deg]  
 Recv. Gain = 30  
 F1 Acq. Length = 11.7473579 [s]  
 F2 Acq. Length = 11.7473579 [s]  
 Filter Width = 15.72066221 [Hz]

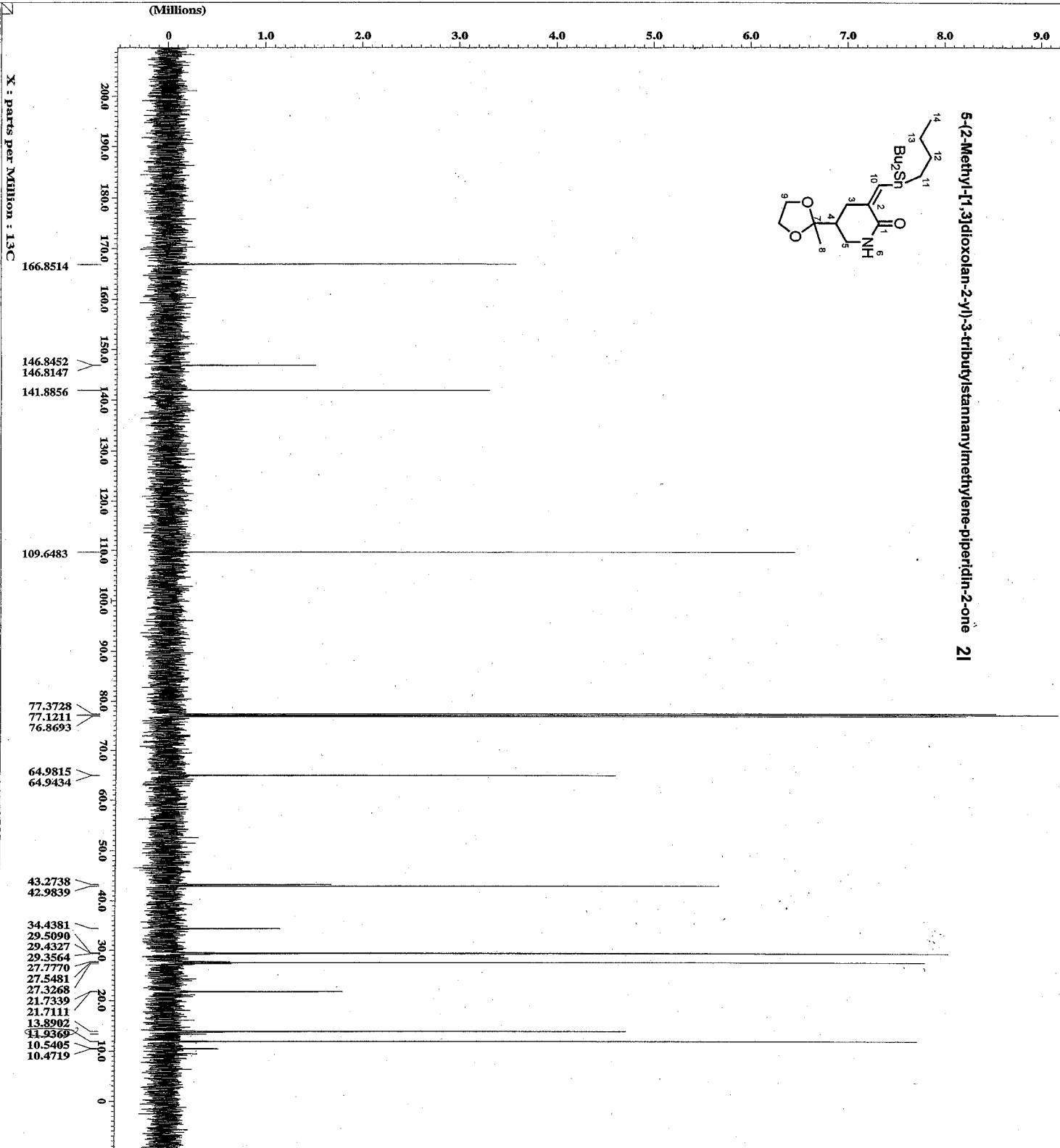
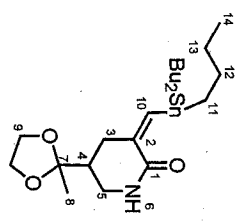
---- ACQUISITION PARAMETERS ----  
 File Name = 1d\_spectrum\_2005  
 Author =  
 Sample ID = 16-16  
 Content = Single Pulse Experiment  
 Creation Date = 10-FEB-2005 15:04:28  
 Experiment Date = 11-FEB-2005 14:37:08  
 Spec Date = 11-FEB-2005 14:37:08  
 Spec Site = HCP500  
 Spec Type = 1D NMR  
 Data Format = 1D COMPLEX  
 Dimensions = 1  
 F2 = 500.1364199 MHz  
 F1 = 163.84 MHz  
 Dim Size = 16384  
 Dim Units = [ppm]  
 Scans = 8  
 Mod\_return = 1  
 X\_domain = 1H  
 X\_offset = 0.000000  
 X\_freq = 500.1364199 MHz  
 X\_sweep = 7.5075075 kHz  
 Solvent = CHLOROFORM-D  
 Spin\_get = 18 Hz  
 Spin\_loss = 22.1 Hz  
 Temp\_get = 300.2 K  
 Temp\_loss = 300.2 K  
 Field\_strength = 11.713791 T  
 Filter\_mode = PULPROG  
 Filter\_width = 3.7511936 Hz







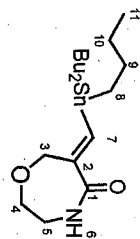
5-(2-Methyl-[1,3]dioxolan-2-yl)-3-tributylstannanylmethylene-piperidin-2-one 21



ACQUISITION PARAMETERS  
File Name = 10\_13c\_spectrum.942  
Sample ID = 5-16  
Content = Single Pulse with Broad  
Creation Date = 10-FEB-2005 15:09:21  
Revision Date = 11-FEB-2005 14:41:45  
Spec Site = MCP500  
Spec Type = DEPTA 13C  
Data Format = 1D COMPLEX  
Pulse Program = zgpg30  
Pulse Length = 13C  
Pulse Size = 12768  
Pulse Width = 132  
Scans = 132  
Modulation = 13C  
X offset = 106.19941  
X freq = 125.77787547 (MHz)  
X sweep = 31.44654086 (Hz)  
Solvent = CHLOROFORM-D  
Spin set = 18 (Hz)  
Recy set = 15 (Hz)  
Recy gain = 11.7473579 (x)  
Field strength = 11.7473579 (T)  
Filter mode = HUYENKORNE  
Filter width = 15.72665221 (Hz)



6-Tributylstannanylmethylene-[1,4]oxazepan-5-one 2n



X : parts per Million : 13C

200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0

(Millions)

174.6341

150.9578  
145.8609  
145.8228

77.3576  
77.0982  
76.8464  
71.3069  
70.1547

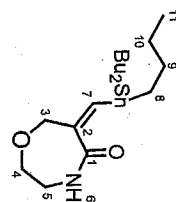
44.2047

29.1123  
27.3345

13.7376  
10.5329

ACQUISITION PARAMETERS  
File Name = 1d\_13c\_spectrum\_893  
Author =  
Sample ID = 427-34  
Date = 8-FEB-2005  
Pulse with Broad  
Creation Date = 7-FEB-2005 21:33:01  
Revision Date = 8-FEB-2005 21:02:39  
Spec Site = ECP500  
Spec Type =  
Data Format = 1D COMPLEX  
Dimensions = 13C  
Dim File = 13C  
Dim Size = 32768  
Dim Units = 100  
Mod Return = 1  
X Domain = 13C  
X Offset = 100 [ppm]  
X Freq = 125.77787547 [MHz]  
X Sweep = 21.44634084 [Hz]  
X Relax = 3.00 [sec]  
SOLV = CDCl3  
SOLV get = 13 [Hz]  
Temp get = 21.8 [deg]  
Recvr Gain = 21  
Field Strength = 11.7473579 [T]  
Filter Acq = 1072000000 [Hz]  
Filter Width = 15.7266221 [Hz]

# 6-Tributylstannanymethylene-[1,4]oxazepan-5-one 2n



7.4705  
7.2974  
7.2516

0.91057

6.7898  
6.7284  
6.6679

1.0

4.2473

1.9788

3.7938  
3.7856

1.93817

3.3430  
3.3339  
3.3247

1.94211

2.0173

1.2715  
1.2569  
0.9114  
0.8950  
0.8794  
0.8684  
0.8537  
0.8391

6.15589

6.23913

-0.0258

15.51968

**JEOL**

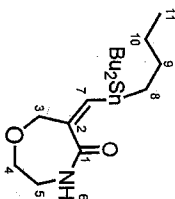
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--- ACQUISITION PARAMETERS ---
File Name      = ID_spectrum.2644
Author ID      = 6114-15
Sample ID      = 5-FEB-2005 20:15:02
Creation Date   = 5-FEB-2005 20:15:02
Revision Date  = 6-FEB-2005 19:41:53
Spec Site      = EPR500

Spec Type      = DELTA RMS
Data Format     = ID COMPLEX
Dimensions     = 1H
Data Title     = 1H
Data Size      = 1.6384
Data Units     = (ppm)
Status         = 1
Mod Return     = 1H
X Domain       = 1H
X Offset       = 500.16041602 (Hz)
X Freq         = 500.137051 (MHz)
Solvent        = CHLOROFORM-D
Spin Set       = 0 (Hz)
Temp Set       = 21.2 (C)
Recvr Gain     = 15.747379 (uV)
Pulse Program  = SPMRNO2DPRN
Filter Width    = 3.7511936 (kHz)
  
```

----- ACQUISITION PARAMETERS -----  
 File Name = 10\_13c\_spectrum.665  
 Author =  
 Sample ID = f14-18  
 Content = Single Pulse with Broad  
 Creation Date = 5-FEB-2005 20:20:27  
 Revision Date = 6-FEB-2005 19:49:09  
 Spec Site = KCP500  
 Spec Type = DELTA\_NMR  
 Data Format = 2D CORTEX  
 Dimensions = 13C  
 Num F2s = 1  
 Num F1s = 1  
 Dia Size = 32768  
 Dia Units = [pxm]  
 Scans = 148  
 Mod Return = 1  
 X\_Coalt = 130 [ppm]  
 X\_Coalt = 125.77787547 [MHz]  
 X\_Freq = 125.77787547 [MHz]  
 X\_Sweep = 31.44654088 [Hz]  
 Solvent = CHLOROFORM-D  
 Spin Set = 0 [Hz]  
 Recycle = 12.3 [sec]  
 Field Strength = 11.743579 [T]  
 Filter Mode = HETEROMODE  
 Filter Width = 15.7206221 [kHz]

6-Tributylstannanylmethylene-[1,4]oxazepan-5-one 2n



174.5807

150.1338  
149.6378

77.3881  
77.1287  
76.8769  
72.7414  
70.0632

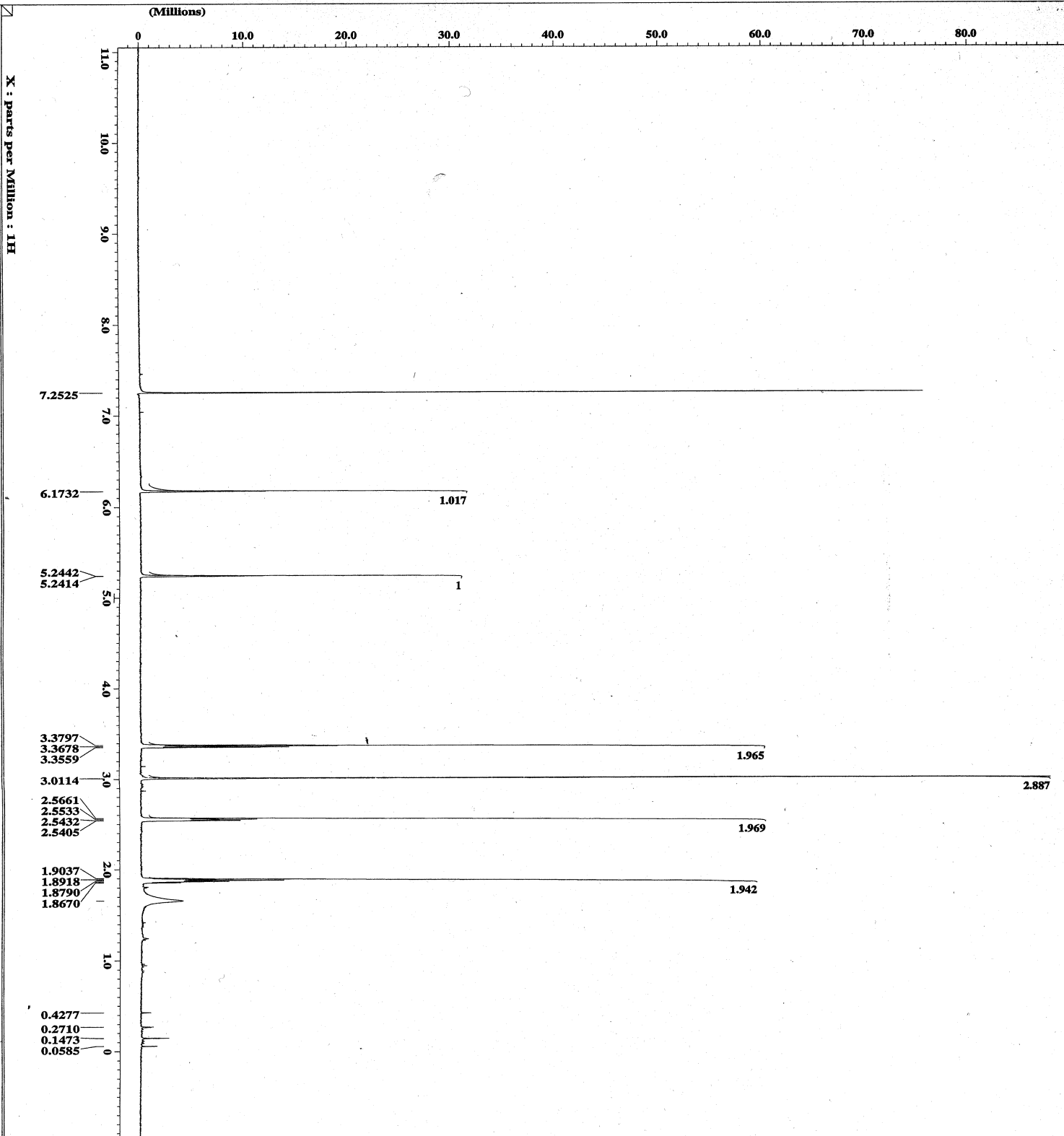
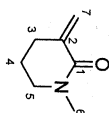
43.8003

29.3488  
29.2649  
27.4489

13.8444  
12.7228  
11.2807  
9.8462

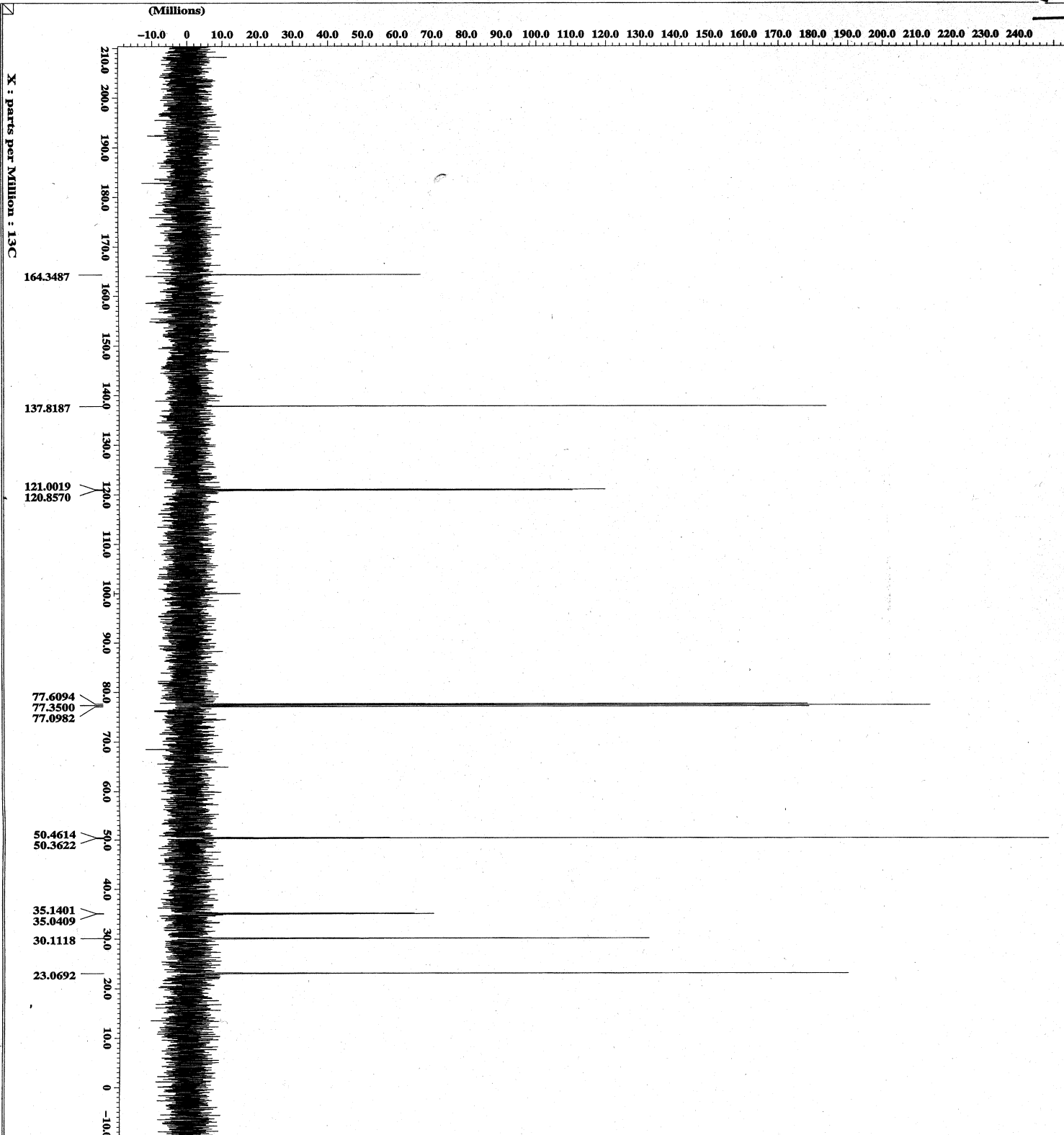
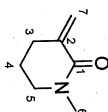
--- ACQUISITION PARAMETERS ---  
 File Name = 1d\_spectrum.483  
 Sample ID = G000  
 Content = Single Pulse Experiment  
 Creation Date = 26-JUN-2006 18:56:40  
 Revolution Date = 26-JUN-2006 04:42:37  
 Spec file = 262508  
 Spec Type = 1H  
 Data Format = ID COMPLEX  
 Dimensions = 1  
 Dim 1 Size = 16384  
 Dim Units = [ppm]  
 Scans = 8  
 Mod Return = 1H  
 X (ppm) = 11.74379 [Hz]  
 X (Hz) = 500.16241602 [MHz]  
 X Sweep = 7.50750751 [Hz]  
 Solvent = CHLOROFORM-D  
 Spin Set = 15 [Hz]  
 Temp Set = 30 [C]  
 Field Strength = 11.74379 [T]  
 Filter Mode = BUTTERWORTH  
 Filter Width = 3.7511936 [Hz]

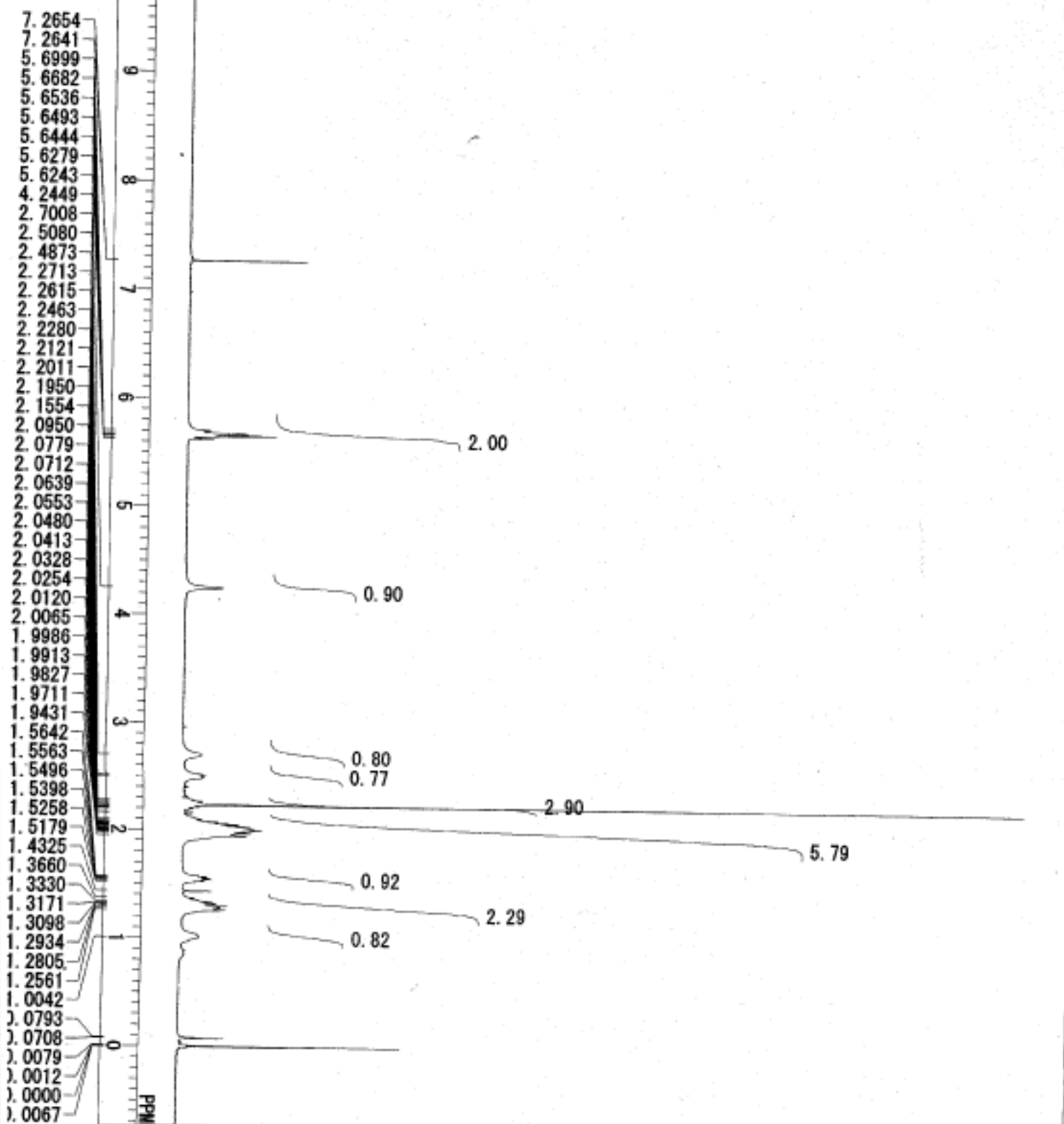
1-Methyl-3-methylene-piperidin-2-one (2o)



--- ACQUISITION PARAMETERS ---  
 File Name = 1d\_13c\_spectrum.561  
 Author ID = 6861148  
 Sample ID = 14320  
 Creation Date = 7-AUG-2006 13:12:43  
 Revision Date = 8-AUG-2006 23:41:59  
 Spec Site = KCP500  
 Spec Type = DELTA NMR  
 Data Format = ID COMPLEX  
 Dimensions = X  
 Dim 1 Size = 13C  
 Dim 2 Size = 32768  
 Dim 3 Size = 17280  
 Scans = 59  
 Mod. return = 1  
 X domain = 13C  
 X offset = 100 (ppm) [7547 (Hz)]  
 X sweep = 31.4454088 (Hz)  
 Solvent = CHLOROFORM-D  
 Spin gate = 16 (Hz)  
 Temp. gate = 24.3 (deg)  
 Recycle delay = 30.747379 (s)  
 F2 delay length = BUTTERMERE  
 Filter mode = BUTTERMERE  
 Filter width = 15.7206221 (kHz)

1-Methyl-3-methylene-piperidin-2-one (2o)





DE FILE C:\WINNMR\98\*COMMON\*\_DEFAULT.AL  
 COUNT Sat Sep 02 16:39:55 2006  
 DATIM 1H  
 OENUC  
 EXMOD NON  
 OBFRO 399.65 MHz  
 OBFRO 124.00 KHz  
 OBFIN 10500.00 Hz  
 POINT 32768  
 FREQ 7992.01 Hz  
 SCANS 8  
 ACQTM 4.1001 sec  
 PD 2.9010 sec  
 PNT 5.80 usec  
 IROUC 1H  
 CTEMP 21.8 c  
 SLVNT CDCL3  
 EXREF 0.00 ppm  
 BF 0.12 Hz  
 RGAIN 15



7-Hydroxy-2-azaspiro[5.5]undecan-8-one (5)



C:\WINNR98XCOMMONY\_DEFAULT.AL

DE FILE  
 COUNT  
 DATIN Sat Sep 02 16:49:18 2006  
 GENUC 13C  
 EXMOD BOM  
 OFTRO 100.40 MHz  
 OFSET 125.00 KHz  
 OFFIN 10500.00 Hz  
 POINT 32768  
 FREQU 27210.88 Hz  
 SCANS 73  
 ACQTM 1.2042 sec  
 PD 1.7940 sec  
 PFI 5.50 usec  
 IRNUC 1H  
 CTMP 22.3 °C  
 SLVIT CDCL3  
 EXREF 77.00 ppm  
 BF 1.20 Hz  
 RGAIN 23

29.6702  
 26.8781

77.3222  
 77.0000  
 76.6778  
 75.8187  
 37.8306

56.5713

16.7328

35.7874

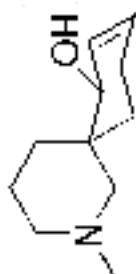
30.7649

27.3449

22.7768

22.0663

PPM



7-Hydroxy-2-azaspiro[5.5]undec-8-ene (5)



Wavelength	254 nm
Wavelength	280 nm
Wavelength	310 nm
Wavelength	330 nm
Wavelength	350 nm
Wavelength	370 nm
Wavelength	390 nm
Wavelength	410 nm
Wavelength	430 nm
Wavelength	450 nm
Wavelength	470 nm
Wavelength	490 nm
Wavelength	510 nm
Wavelength	530 nm
Wavelength	550 nm
Wavelength	570 nm
Wavelength	590 nm
Wavelength	610 nm
Wavelength	630 nm
Wavelength	650 nm
Wavelength	670 nm
Wavelength	690 nm
Wavelength	710 nm
Wavelength	730 nm
Wavelength	750 nm
Wavelength	770 nm
Wavelength	790 nm
Wavelength	810 nm
Wavelength	830 nm
Wavelength	850 nm
Wavelength	870 nm
Wavelength	890 nm
Wavelength	910 nm
Wavelength	930 nm
Wavelength	950 nm
Wavelength	970 nm
Wavelength	990 nm
Wavelength	1010 nm
Wavelength	1030 nm
Wavelength	1050 nm
Wavelength	1070 nm
Wavelength	1090 nm
Wavelength	1110 nm
Wavelength	1130 nm
Wavelength	1150 nm
Wavelength	1170 nm
Wavelength	1190 nm
Wavelength	1210 nm
Wavelength	1230 nm
Wavelength	1250 nm
Wavelength	1270 nm
Wavelength	1290 nm
Wavelength	1310 nm
Wavelength	1330 nm
Wavelength	1350 nm
Wavelength	1370 nm
Wavelength	1390 nm
Wavelength	1410 nm
Wavelength	1430 nm
Wavelength	1450 nm
Wavelength	1470 nm
Wavelength	1490 nm
Wavelength	1510 nm
Wavelength	1530 nm
Wavelength	1550 nm
Wavelength	1570 nm
Wavelength	1590 nm
Wavelength	1610 nm
Wavelength	1630 nm
Wavelength	1650 nm
Wavelength	1670 nm
Wavelength	1690 nm
Wavelength	1710 nm
Wavelength	1730 nm
Wavelength	1750 nm
Wavelength	1770 nm
Wavelength	1790 nm
Wavelength	1810 nm
Wavelength	1830 nm
Wavelength	1850 nm
Wavelength	1870 nm
Wavelength	1890 nm
Wavelength	1910 nm
Wavelength	1930 nm
Wavelength	1950 nm
Wavelength	1970 nm
Wavelength	1990 nm
Wavelength	2010 nm
Wavelength	2030 nm
Wavelength	2050 nm
Wavelength	2070 nm
Wavelength	2090 nm
Wavelength	2110 nm
Wavelength	2130 nm
Wavelength	2150 nm
Wavelength	2170 nm
Wavelength	2190 nm
Wavelength	2210 nm
Wavelength	2230 nm
Wavelength	2250 nm
Wavelength	2270 nm
Wavelength	2290 nm
Wavelength	2310 nm
Wavelength	2330 nm
Wavelength	2350 nm
Wavelength	2370 nm
Wavelength	2390 nm
Wavelength	2410 nm
Wavelength	2430 nm
Wavelength	2450 nm
Wavelength	2470 nm
Wavelength	2490 nm
Wavelength	2510 nm
Wavelength	2530 nm
Wavelength	2550 nm
Wavelength	2570 nm
Wavelength	2590 nm
Wavelength	2610 nm
Wavelength	2630 nm
Wavelength	2650 nm
Wavelength	2670 nm
Wavelength	2690 nm
Wavelength	2710 nm
Wavelength	2730 nm
Wavelength	2750 nm
Wavelength	2770 nm
Wavelength	2790 nm
Wavelength	2810 nm
Wavelength	2830 nm
Wavelength	2850 nm
Wavelength	2870 nm
Wavelength	2890 nm
Wavelength	2910 nm
Wavelength	2930 nm
Wavelength	2950 nm
Wavelength	2970 nm
Wavelength	2990 nm
Wavelength	3010 nm
Wavelength	3030 nm
Wavelength	3050 nm
Wavelength	3070 nm
Wavelength	3090 nm
Wavelength	3110 nm
Wavelength	3130 nm
Wavelength	3150 nm
Wavelength	3170 nm
Wavelength	3190 nm
Wavelength	3210 nm
Wavelength	3230 nm
Wavelength	3250 nm
Wavelength	3270 nm
Wavelength	3290 nm
Wavelength	3310 nm
Wavelength	3330 nm
Wavelength	3350 nm
Wavelength	3370 nm
Wavelength	3390 nm
Wavelength	3410 nm
Wavelength	3430 nm</

