

## Supporting Information

**Gold-Catalyzed One-Step Practical Synthesis of Oxetan-3-ones from Readily Available Propargylic Alcohols**

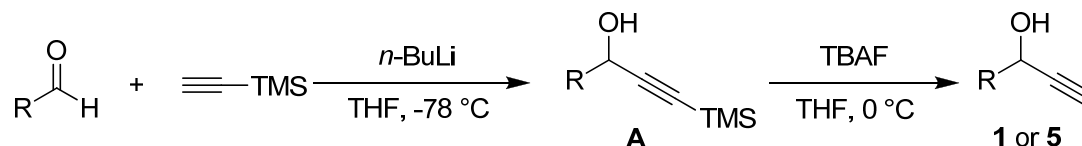
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**General.** Ethyl acetate (ACS grade), hexanes (ACS grade) and diethyl ether (ACS grade) were purchased from Fisher Scientific and used without further purification. Anhydrous 1,2-dichloroethane (HPLC grade) was purified by distillation over calcium hydride. Anhydrous tetrahydrofuran was distilled from sodium benzophenone ketyl. Commercially available reagents were used without further purification. Reactions were monitored by thin layer chromatography (TLC) using silicycle pre-coated silica gel plates. Flash column chromatography was performed over silicycle silica gel (230-400 mesh).  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded on a Varian 500 MHz UNITY INOVA spectrometer and a Varian 400 MHz UNITY INOVA spectrometer using residue solvent peaks as internal standards. Infrared spectra were recorded with a Perkin Elmer FT-IR spectrum 2000 spectrometer and are reported in reciprocal centimeter ( $\text{cm}^{-1}$ ). Mass spectra were recorded with Micromass QTOF2 Quadrupole/Time-of-Flight Tandem mass spectrometer using electron spray ionization.

**General procedure A: preparation of secondary propargyl alcohols 1 and 5**

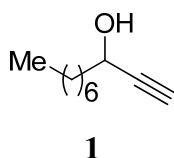


A solution of *n*-butyllithium in hexanes (1.6 M, 5.3 mL, 8.5 mmol) was added dropwise to a solution of (trimethylsilyl)acetylene (8.0 mmol) in anhydrous THF (20 mL) at -78 °C. After 15 min, a solution of an aldehyde (5.3 mmol) in anhydrous THF (20 mL) was added dropwise to the reaction mixture, and the resulting mixture was further stirred at the same temperature for 3 h. The reaction was quenched with saturated  $\text{NH}_4\text{Cl}$  solution, and the mixture was extracted four times with  $\text{Et}_2\text{O}$ . The combined organic layers were washed with brine, dried with anhydrous  $\text{MgSO}_4$ , and the solvents were evaporated under vacuum. The oily residue was purified by flash silica gel column chromatography (hexanes/ $\text{EtOAc}$ ) to yield propargylic alcohol A.

A solution of tetrabutylammonium fluoride (1.0 M, 4.8 mL, 4.8 mmol) was added dropwise to a solution of propargylic alcohol A (4.0 mmol) in THF (35 mL) at 0 °C and

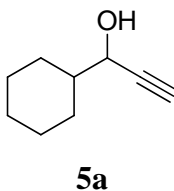
the mixture was stirred at 0 °C for 15 min. After addition of an aqueous NH<sub>4</sub>Cl solution, the mixture was extracted three times with Et<sub>2</sub>O. The combined organic layers were washed with brine, dried with anhydrous MgSO<sub>4</sub>, and the solvents was evaporated under vacuum. The oily residue was purified by flash silica gel column chromatography (hexanes/EtOAc ) to yield propargylic alcohols **1** or **5**.

### Dec-1-yn-3-ol (**1**)



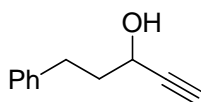
Alcohol **1** was prepared in 77% yield (2 steps) by following General Procedure A. This compound is known and the spectroscopic data match those reported.<sup>1</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 4.40 – 4.35 (m, 1H), 2.46 (d, 1H, *J* = 2.4 Hz), 1.78 (d, 1H, *J* = 6.0 Hz), 1.75 – 1.67 (m, 2H), 1.49 – 1.41 (m, 2H), 1.30 – 1.25 (m, 8H), 0.88 (t, 3H, *J* = 6.8 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 85.0, 72.8, 62.4, 37.7, 31.8, 29.2, 29.1, 25.0, 22.6, 14.1.

### 1-Cyclohexylprop-2-yn-1-ol (**5a**)

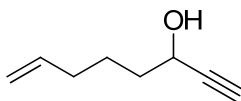


Alcohol **5a** was prepared in 73% yield (2 steps) by following General Procedure A. This compound is known and the spectroscopic data match those reported.<sup>1</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 4.19 – 4.14 (m, 1H), 2.46 (d, 1H, *J* = 2.0 Hz), 1.88 – 1.75 (m, 5H), 1.70 – 1.64 (m, 1H), 1.61 – 1.51 (m, 1H), 1.31 – 1.02 (m, 5H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 83.9, 73.6, 67.0, 43.9, 28.4, 27.9, 26.3, 25.8, 25.7.

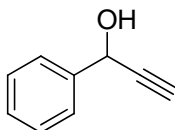
### 5-Phenylpent-1-yn-3-ol (**5b**)

**5b**

Alcohol **5b** was prepared in 69% yield (2 steps) by following General Procedure A. This compound is known and the spectroscopic data match those reported.<sup>1</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32 – 7.29 (m, 2H), 7.23 – 7.19 (m, 3H), 4.40 – 4.36 (m, 1H), 2.82 (t, 2H,  $J$  = 8.0 Hz), 2.52 (d, 1H,  $J$  = 2.4 Hz), 2.10 – 1.98 (m, 2H), 1.88 (d, 1H,  $J$  = 5.2 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  141.1, 128.5, 128.4, 126.0, 84.6, 73.3, 61.5, 39.0, 31.2.

**Oct-7-en-1-yn-3-ol (5c)****5c**

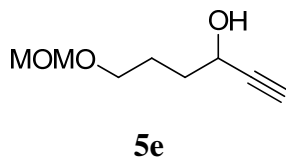
Alcohol **5c** was prepared in 63% yield (2 steps) by following General Procedure A. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  5.84 – 5.76 (m, 1H), 5.05 – 4.95 (m, 2H), 4.40 – 4.36 (m, 1H), 2.47 (d, 1H,  $J$  = 2.0 Hz), 2.12 – 2.07 (m, 2H), 1.93 – 1.90 (bs, 1H), 1.78 – 1.69 (m, 2H), 1.60 – 1.53 (m, 2H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  138.3, 114.9, 84.8, 73.0, 62.1, 37.0, 33.2, 24.2; IR (neat): 3432 (bs), 3302, 2935, 2862, 2114, 1643, 1439, 910; MS (ES<sup>+</sup>) Calculated for [C<sub>8</sub>H<sub>13</sub>O]<sup>+</sup>: 125.1; Found: 125.1.

**1-Phenylprop-2-yn-1-ol (5d)****5d**

Alcohol **5d** was prepared in 73% yield (2 steps) by following General Procedure A. This compound is known and the spectroscopic data match those reported.<sup>1</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.56 (d, 2H,  $J$  = 6.0 Hz), 7.42 – 7.33 (m, 3H), 5.48 (dd, 1H,  $J$  = 5.2 Hz,  $J$

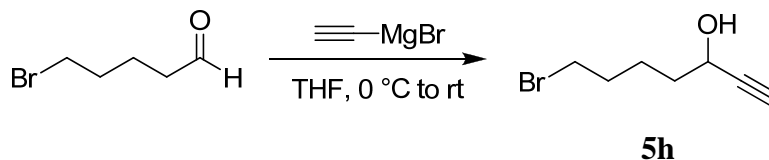
= 1.6 Hz), 2.68 (d, 1H,  $J$  = 1.6 Hz), 2.37 (d, 1H,  $J$  = 5.2 Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  140.0, 128.7, 128.6, 126.6, 83.5, 74.9, 64.4.

### 6-Methoxymethoxyhex-1-yn-3-ol (**5e**)



Alcohol **5e** was prepared in 70% yield (2 steps) by following General Procedure A.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.63 (s, 2H), 4.46 – 4.42 (m, 1H), 3.63 – 3.54 (m, 2H), 3.37 (s, 3H), 2.56 (d, 1H,  $J$  = 6.0 Hz), 2.47 (d, 1H,  $J$  = 2.4 Hz), 1.88 – 1.75 (m, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  96.1, 84.8, 72.7, 67.2, 61.6, 55.1, 34.5, 25.2; IR (neat): 3429(bs), 3290, 2939, 2114, 1732, 1446, 1041; Calculated for  $[\text{C}_8\text{H}_{14}\text{NaO}_3]^+$ : 181.1; Found: 181.1.

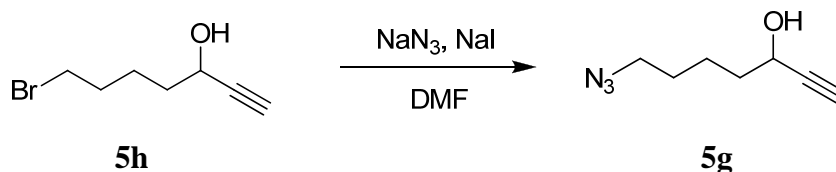
### 7-Bromohept-1-yn-3-ol (**5h**)



A solution of ethynylmagnesium bromide (24 mL) in THF (0.5 M, 12.0 mmol) was added dropwise over 5 min to a stirred solution of 5-bromopentanal (8.0 mmol) in anhydrous THF (20 mL) at 0 °C under  $\text{N}_2$ . The mixture was allowed to warm to room temperature and stirred for 30 min. Ether (80 mL) and saturated aqueous ammonium chloride solution (80 mL) were added, and the organic layer was then separated. The aqueous layer was extracted with ether (2 x 80 mL). The combined organic layers were then washed with brine (100 mL), dried and concentrated *in vacuo* to afford alcohol **5h** (90%) as a pale yellow oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.38 (dt, 1H,  $J$  = 6.4 Hz,  $J$  = 2.0 Hz), 3.41 (t, 2H,  $J$  = 6.8 Hz), 2.48 (d, 1H,  $J$  = 2.4 Hz), 2.06 (bs, 1H), 1.94 – 1.86 (m, 2H), 1.77 – 1.71 (m, 2H), 1.65 – 1.58 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  84.5, 73.2,

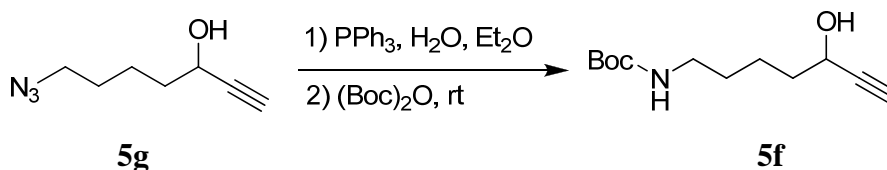
61.9, 36.5, 33.4, 32.3, 23.7; IR (neat): 3385(bs), 3294, 2939, 2866, 2114, 1624, 1442, 1254, 1053, 1011, 652; MS (ES<sup>+</sup>) Calculated for [C<sub>7</sub>H<sub>11</sub>BrNaO]<sup>+</sup>: 213.0; Found: 213.0.

### 7-Azidohept-1-yn-3-ol (**5g**)



To a solution of **5h** (1.15 g, 6.0 mmol) in DMF (20 mL) was added NaN<sub>3</sub> (1.18 g, 18 mmol) and NaI (0.60 g, 3.0 mmol) at room temperature. The resulting mixture was heated at 80 °C for 12 h and cooled to room temperature before the addition of water (20 mL) and diethyl ether (30 mL). The two layers were separated, and the aqueous layer was extracted with Et<sub>2</sub>O (3 × 50 mL). The combined organic layers were washed with water (2 × 20 mL), brine (20 mL), dried with MgSO<sub>4</sub>, and concentrated. The crude product was purified with flash silica gel column chromatography (eluent: hexanes : ethyl acetate = 5:1) to get **5g** (0.55 g) in 60% yield. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 4.40 (dt, 1H, *J* = 6.4 Hz, *J* = 2.0 Hz), 3.30 (t, 2H, *J* = 6.8 Hz), 2.48 (d, 1H, *J* = 2.0 Hz), 1.83 – 1.52 (m, 7H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 84.6, 73.1, 61.8, 51.2, 36.9, 28.4, 22.2; IR (neat): 3386(bs), 3294, 2943, 2866, 2102, 1458, 1265, 1022, 660; MS (ES<sup>+</sup>) Calculated for [C<sub>7</sub>H<sub>11</sub>N<sub>3</sub>NaO]<sup>+</sup>: 176.1; Found: 176.1.

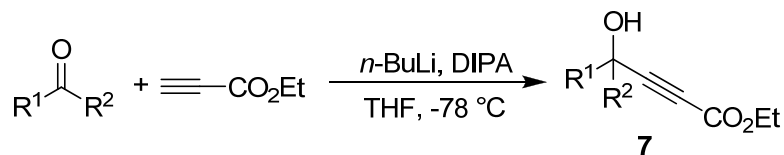
### *tert*-Butyl (5-hydroxyhept-6-ynyl)carbamate (**5f**)



To a solution of **5g** (0.400 g, 2.6 mmol) in Et<sub>2</sub>O (30 mL) was added PPh<sub>3</sub> (0.818 g, 3.1 mmol) and H<sub>2</sub>O (1.9 mL) at room temperature. The resulting mixture was stirred for 12 h. The reaction mixture was then treated with (Boc)<sub>2</sub>O (4.19 g, 19.2 mmol) and stirred overnight. Water was then added, and the resulting two layers were separated. The

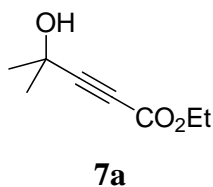
aqueous layer was extracted with Et<sub>2</sub>O (3 × 30 mL), and the combined organic layers were washed with brine (30 mL), dried with MgSO<sub>4</sub>, and concentrated. The crude product was purified with flash silica gel column chromatography (eluent: hexanes : ethyl acetate = 3:1) to give **5f** (0.396 g) in 67% yield. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 4.66 (bs, 1H), 4.34 (dt, 1H, *J* = 6.5 Hz, *J* = 2.0 Hz), 3.10 (s, 2H), 2.75 (bs, 1H), 2.43 (d, 1H, *J* = 2.5 Hz), 1.73 – 1.68 (m, 2H), 1.51 – 1.44 (m, 4H), 1.41 (s, 9H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 156.1, 84.9, 79.1, 72.7, 61.8, 40.2, 37.0, 29.6, 28.3, 22.1; IR (neat): 3352(bs), 3305(bs), 2939, 2870, 2110, 1693, 1527, 1169; MS (ES<sup>+</sup>) Calculated for [C<sub>12</sub>H<sub>21</sub>NNaO<sub>3</sub>]<sup>+</sup>: 250.1; Found: 250.1.

### General procedure B: preparation of tertiary propargylic alcohols **7**



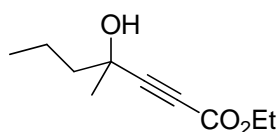
A solution of *n*-butyllithium in hexanes (1.6 M, 33.0 mmol) was added dropwise to a solution of freshly distilled diisopropylamine (33.0 mmol) in dried THF (30 mL) at 0 °C. The solution was stirred for 1 h at 0 °C, then cooled to -78 °C. Ethyl propiolate (31.3 mmol) in dried THF (10 mL) was then added dropwise to the reaction mixture. After 1 h at the same temperature, ketone (62.6 mmol) was added, and the resulting mixture was stirred at -78 °C for 3 h. The reaction was quenched with saturated NH<sub>4</sub>Cl solution, and the mixture was extracted four times with Et<sub>2</sub>O. The combined organic layers were washed with brine, dried with anhydrous MgSO<sub>4</sub>, and the solvents evaporated to dryness. The oily residue was purified by flash silica gel column chromatography (hexanes/EtOAc) to yield propargylic alcohol **7**.

### 4-Hydroxy-4-methylpent-2-ynoic acid ethyl ester (**7a**)



Alcohol **7a** was prepared in 90% yield (2 steps) by following General Procedure B. This compound is known and the spectroscopic data match those reported.<sup>2</sup> <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 4.22 (q, 2H, *J* = 7.0 Hz), 2.55 (s, 1H), 1.55 (s, 6H), 1.30 (t, 3H, *J* = 7.3 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 153.6, 91.0, 74.0, 64.9, 62.1, 30.5, 13.9; IR (neat): 3413(bs), 2985, 2237, 1705, 1612, 1450, 1373, 1257, 1026, 949; MS (ES<sup>+</sup>) Calculated for [C<sub>8</sub>H<sub>12</sub>NaO<sub>3</sub>]<sup>+</sup>: 179.1; Found: 179.1.

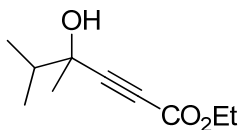
#### 4-Hydroxy-4-methylhept-2-ynoic acid ethyl ester (**7b**)



**7b**

Alcohol **7b** was prepared in 87% yield (2 steps) by following General Procedure B. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 4.21 (q, 2H, *J* = 7.0 Hz), 2.32 (bs, 1H), 1.72 – 1.62 (m, 2H), 1.57 – 1.44 (m, 5H), 1.29 (t, 3H, *J* = 7.0 Hz), 0.94 (t, 3H, *J* = 7.0 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 153.6, 90.4, 75.1, 67.9, 62.0, 45.0, 28.9, 17.7, 14.0, 13.9; IR (neat): 3421(bs), 2966, 2885, 2233, 1709, 1462, 1369, 1250, 1030, 752; MS (ES<sup>+</sup>) Calculated for [C<sub>10</sub>H<sub>16</sub>NaO<sub>3</sub>]<sup>+</sup>: 207.1; Found: 207.1.

#### 4-Hydroxy-4,5-dimethylhex-2-ynoic acid ethyl ester (**7c**)



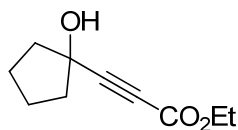
**7c**

Alcohol **7c** was prepared in 88% yield (2 steps) by following General Procedure B. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 4.21 (q, 2H, *J* = 7.2 Hz), 2.23 (bs, 1H), 1.86 (heptet, 1H, *J* = 7.2 Hz), 1.48 (s, 3H), 1.30 (t, 3H, *J* = 7.2 Hz), 1.04 (d, 3H, *J* = 7.2 Hz), 1.02 (d, 3H, *J* = 6.8 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 153.6, 89.9, 75.9, 71.5, 62.1, 38.6, 26.3, 17.6,



17.2, 14.0; IR (neat): 3406(bs), 2935, 2862, 2233, 1712, 1458, 1250, 1026; MS ( $\text{ES}^+$ )  
Calculated for  $[\text{C}_{10}\text{H}_{16}\text{NaO}_3]^+$ : 207.1; Found: 207.1.

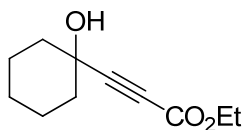
**(1-Hydroxycyclopentyl)propynoic acid ethyl ester (7d)**



**7d**

Alcohol **7d** was prepared in 91% yield (2 steps) by following General Procedure B. This compound is known and the spectroscopic data match those reported.<sup>2</sup>  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.23 (q, 2H,  $J = 7.2$  Hz), 2.06 – 1.94 (m, 5H), 1.90 – 1.74 (m, 4H), 1.31 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.6, 90.3, 75.2, 74.0, 62.1, 42.1, 23.5, 14.0.

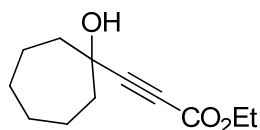
**(1-Hydroxycyclohexyl)propynoic acid ethyl ester (7e)**



**7e**

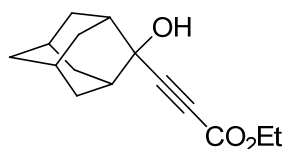
Alcohol **7e** was prepared in 90% yield (2 steps) by following General Procedure B. This compound is known and the spectroscopic data match those reported.<sup>2</sup>  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.23 (q, 2H,  $J = 7.2$  Hz), 2.40 (bs, 1H), 2.00 – 1.94 (m, 2H), 1.74 – 1.51 (m, 8H), 1.31 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.6, 90.3, 76.0, 68.4, 62.1, 39.0, 24.9, 22.8, 14.0.

**(1-Hydroxycycloheptyl)propynoic acid ethyl ester (7f)**

**7f**

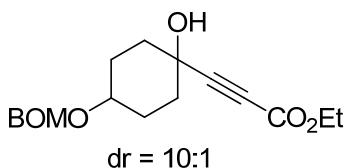
Alcohol **7f** was prepared in 82% yield (2 steps) by following General Procedure B.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.23 (q, 2H,  $J = 7.2$  Hz), 2.10 – 2.03 (m, 3H), 1.92 – 1.86 (m, 2H), 1.69 – 1.55 (m, 8H), 1.31 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.7, 91.6, 75.1, 71.5, 62.1, 42.2, 28.1, 21.9, 14.0; IR (neat): 3406(bs), 2935, 2862, 2233, 1712, 1458, 1250, 1026; Calculated for  $[\text{C}_{12}\text{H}_{18}\text{NaO}_3]^+$ : 233.1; Found: 233.1.

**(2-Hydroxyadamantan-2-yl)propynoic acid ethyl ester (7g)**

**7g**

Alcohol **7g** was prepared in 85% yield (2 steps) by following General Procedure B.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  4.23 (q, 2H,  $J = 7.0$  Hz), 2.25 (s, 1H), 2.16 (d, 2H,  $J = 13.0$  Hz), 2.09 (d, 2H,  $J = 13.0$  Hz), 2.02 (bs, 2H), 1.85 – 1.74 (m, 4H), 1.70 (s, 2H), 1.59 – 1.55 (m, 2H), 1.30 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.7, 91.0, 72.5, 62.0, 38.1, 37.3, 35.2, 31.2, 26.6, 26.5, 14.0; IR (neat): 3429(bs), 2920, 2854, 2225, 1701, 1250, 1022; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_{15}\text{H}_{20}\text{NaO}_3]^+$ : 271.1; Found: 271.2.

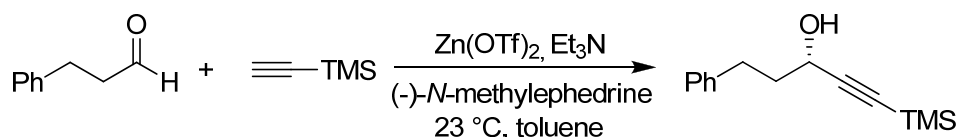
**(4-Benzyloxymethoxy-1-hydroxycyclohexyl)propynoic acid ethyl ester (7h)**

**7h**

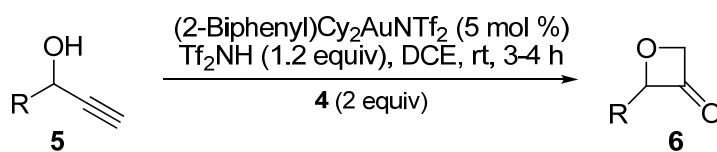
Alcohol **7h** (dr: 10/1) was prepared in 78% yield (2 steps) by following General Procedure B.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.39 – 7.27 (m, 5H), 4.80 (s, 2H), 4.62 (s,

2H), 4.23 (q, 2H,  $J = 7.2$  Hz), 3.76 (bs, 1H), 2.33 (bs, 1H), 2.03 – 1.95 (m, 2H), 1.87 – 1.76 (m, 6H), 1.31 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.5, 137.8, 128.4, 127.8, 127.7, 92.7, 89.8, 75.6, 71.4, 69.5, 67.3, 62.1, 35.0, 27.6, 14.0; IR (neat): 3417(bs), 2943, 2889, 2229, 1709, 1454, 1369, 1250, 1041, 744; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_{19}\text{H}_{24}\text{NaO}_5]^+$ : 355.2; Found: 355.2.

### Procedure for the preparation of chiral 5-phenyl-pent-1-yn-3-ol



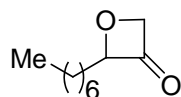
This chiral alcohol was prepared in 36% overall yield by reacting (trimethylsilyl)acetylene with 3-phenyl-propionaldehyde (according to Carreira's method<sup>3</sup>) and subsequently removing TMS group. This compound is known and the spectroscopic data match those reported.<sup>4</sup> Its enantiomeric excess (80%) was determined by chiral HPLC [Chiralpak IA Column, 2/98 *i*PrOH /hexane, 0.7 mL/min, 254 nm; TR = 27.83 min (major), 26.62 min (minor)].  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.32 – 7.19 (m, 5H), 4.41 – 4.36 (m, 1H), 2.82 (t, 2H,  $J = 8.0$  Hz), 2.52 (d, 1H,  $J = 2.0$  Hz), 2.10 – 2.02 (m, 2H), 1.99 (d, 1H,  $J = 5.6$  Hz).



**General procedure C:** 3-Methoxycarbonyl-5-bromopyridine *N*-oxide (**4**, 0.60 mmol),  $\text{Tf}_2\text{NH}$  (1.8 mL, 0.20 M in DCE), and (2-biphenyl) $\text{Cy}_2\text{PAuNTf}_2$  (12.5 mg, 0.015 mmol) were added sequentially to a solution of secondary propargyl alcohol **1** or **5** (0.30 mmol) in DCE (4.2 mL) at room temperature. The reaction mixture was stirred at rt and the progress of the reaction was monitored by TLC. The reaction typically took 3 – 4 h. Upon completion, the reaction was treated with saturated aqueous  $\text{NaHCO}_3$  (15 mL), and the resulting solution was extracted with DCM ( $2 \times 30$  mL). The combined organic layers

were dried with  $\text{MgSO}_4$ . The mixture was concentrated and the residue was purified by silica gel flash chromatography (eluent: hexanes/ethyl acetate) to afford desired products **2** or **6**.

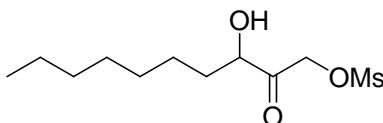
### 2-Heptyloxetan-3-one (**2**)



**2**

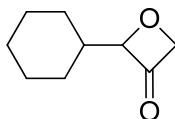
Compound **2** was prepared in 71% yield using **4** as oxidant according to General Procedure C. The reaction time was 3 h.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.56 – 5.45 (m, 1H), 5.28 (d, 1H,  $J = 15.0$  Hz), 5.22 (dd, 1H,  $J = 4.5, 15.0$  Hz), 1.88 – 1.77 (m, 2H), 1.52 – 1.40 (m, 2H), 1.39 – 1.22 (m, 8H), 0.88 (t, 3H,  $J = 7.0$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  203.6, 104.0, 88.6, 31.7, 31.3, 29.2, 29.0, 24.1, 22.6, 14.0; IR (neat): 2931, 2862, 1824, 1462, 1261, 1034, 960, 802; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_{10}\text{H}_{18}\text{NaO}_2]^+$ : 193.1; Found: 193.1.

### Methanesulfonic acid 3-hydroxy-2-oxodecyl ester **3**

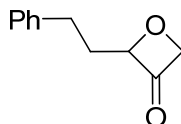


**3**

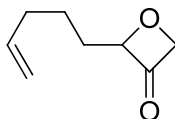
Compound **3** was isolated in 9.2% yield (**4** as oxidant, 1.2 equiv  $\text{MsOH}$  as the acid and  $\text{PPh}_3\text{AuNTf}_2$  as the gold catalyst) according to General Procedure C (entry 7, table 1). The reaction time was 6 h.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.05 (dd, 2H,  $J = 36.0$  Hz,  $J = 17.0$  Hz), 4.33 – 4.30 (m, 1H), 3.20 (s, 3H), 2.84 (d, 1H,  $J = 5.0$  Hz), 1.83 – 1.76 (m, 1H), 1.65 – 1.56 (m, 1H), 1.47 – 1.27 (m, 10H), 0.88 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  204.9, 75.3, 69.8, 38.9, 33.8, 31.7, 29.2, 29.0, 24.6, 22.6, 14.0; IR (neat): 3464(bs), 2924, 2854, 2256, 1732, 1335, 1173, 1026, 733; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_{11}\text{H}_{22}\text{NaO}_5\text{S}]^+$ : 289.1; Found: 289.1.

**2-Cyclohexyloxetan-3-one (6a)****6a**

Compound **6a** was prepared in 73% yield using **4** as oxidant according to General Procedure C. The reaction time was 3 h.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.24 – 5.21 (m, 2H), 5.14 (dd, 1H,  $J = 15.0$  Hz,  $J = 4.5$  Hz), 1.87 – 1.66 (m, 6H), 1.29 – 1.07 (m, 5H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  203.5, 107.8, 88.7, 40.1, 27.5, 27.4, 26.1, 25.5, 25.4; IR (neat): 2931, 2854, 1824, 1446, 1045, 960; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_9\text{H}_{14}\text{KO}_2]^+$ : 193.1; Found: 193.1.

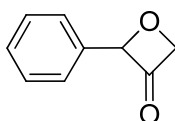
**2-Phenethyloxetan-3-one (6b)****6b**

Compound **6b** was prepared in 80% yield using **4** as oxidant according to General Procedure C. The reaction time was 4 h.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.32 – 7.29 (m, 2H), 7.23 – 7.20 (m, 3H), 5.48 – 5.44 (m, 1H), 5.31 (d, 1H,  $J = 15.0$  Hz), 5.26 (dd, 1H,  $J = 3.5, 15.0$  Hz), 2.85 – 2.75 (m, 2H), 2.23 – 2.10 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  203.1, 140.3, 128.5, 128.4, 126.3, 102.8, 88.8, 32.8, 30.1; IR (neat): 2924, 2866, 1817, 1454, 1068, 957, 698; Calculated for  $[\text{C}_{11}\text{H}_{12}\text{NaO}_2]^+$ : 199.1; Found: 199.1.

**2-Pent-4-enyl-oxetan-3-one (6c)****6c**

Compound **6c** was prepared in 65% yield using **4** as oxidant according to General Procedure C. The reaction time was 3 h.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.82 – 5.74 (m, 1H), 5.47 (dd, 1H,  $J = 10.5$  Hz,  $J = 6.5$  Hz), 5.28 (d, 1H,  $J = 15.0$  Hz), 5.22 (dd, 1H,  $J = 4.0$ , 15.0 Hz), 5.04 – 4.97 (m, 2H), 2.13 – 2.08 (m, 2H), 1.88 – 1.81 (m, 2H), 1.63 – 1.50 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  203.3, 137.8, 115.2, 103.7, 88.7, 33.2, 30.6, 23.3; IR (neat): 2931, 2862, 1817, 1439, 1049, 957; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_8\text{H}_{12}\text{KO}_2]^+$ : 179.1; Found: 179.1.

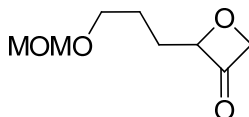
### 2-Phenyloxetan-3-one (**6d**)



**6d**

Compound **6d** was prepared in 57% yield using 2-bromopyridine *N*-oxide as oxidant and MsOH (1.2 equiv) as additive according to General Procedure C. The reaction time was 4 h.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.45 – 7.36 (m, 5H), 6.39 (d, 1H,  $J = 4.4$  Hz), 5.53 (d, 1H,  $J = 14.8$  Hz), 5.47 (dd, 1H,  $J = 4.4$ , 14.8 Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  199.3, 130.4, 128.9, 128.7, 125.2, 104.3, 90.1; IR (neat): 2931, 2854, 2364, 1828, 1732, 1643, 1404, 957, 698; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_9\text{H}_9\text{O}_2]^+$ : 149.1; Found: 149.0.

### 2-(3-Methoxymethoxypropyl)oxetan-3-one (**6e**)

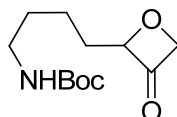


**6e**

Compound **6e** was prepared in 65% yield using **4** as oxidant according to General Procedure C. The reaction time was 3 h.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.50 (dd, 1H,  $J = 10.8$  Hz,  $J = 6.4$  Hz), 5.29 (d, 1H,  $J = 15.2$  Hz), 5.24 (dd, 1H,  $J = 4.0$ , 15.2 Hz), 4.61 (s, 2H), 3.56 (t, 2H,  $J = 6.4$  Hz), 3.35 (s, 3H), 1.96 (dd, 2H,  $J = 14.4$  Hz,  $J = 6.8$  Hz), 1.82 – 1.71 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  203.0, 103.5, 96.4, 88.8, 66.9, 55.2, 28.2,

24.4; IR (neat): 2935, 1824, 1439, 1041, 957; Calculated for  $[\text{C}_8\text{H}_{15}\text{O}_4]^+$ : 175.1; Found: 175.1.

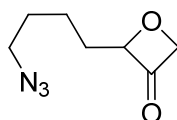
**[4-(3-Oxooxetan-2-yl)butyl]carbamic acid tert-butyl ester (6f)**



**6f**

Compound **6f** was prepared in 60% yield using **4** as oxidant at  $-20\text{ }^\circ\text{C}$  according to General Procedure C. The reaction time was 16 h.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.48 – 5.43 (m, 1H), 5.28 (d, 1H,  $J = 14.8$  Hz), 5.24 (dd, 1H,  $J = 4.0, 14.8$  Hz), 4.54 (bs, 1H), 3.17 – 3.10 (m, 2H), 1.88 – 1.82 (m, 2H), 1.55 – 1.46 (m, 4H), 1.43 (s, 9H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  203.1, 155.9, 103.6, 88.8, 79.1, 40.2, 30.8, 29.8, 28.4, 21.4; IR (neat): 3356(bs), 2978, 2935, 2866, 1817, 1701, 1527, 1250, 1173, 957; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_{12}\text{H}_{21}\text{NNaO}_4]^+$ : 266.1; Found: 266.2.

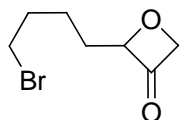
**2-(4-Azidobutyl)oxetan-3-one (6g)**



**6g**

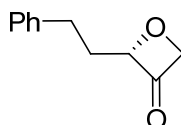
Compound **6g** was prepared in 81% yield using **4** as oxidant according to General Procedure C. The reaction time was 3 h.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.50 – 5.44 (m, 1H), 5.30 (d, 1H,  $J = 15.2$  Hz), 5.22 (dd, 1H,  $J = 4.4, 15.2$  Hz), 3.29 (t, 2H,  $J = 6.4$  Hz), 1.90 – 1.83 (m, 2H), 1.68 – 1.49 (m, 4H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  202.9, 103.4, 88.9, 51.1, 30.7, 28.5, 21.5; IR (neat): 2939, 2870, 2102, 1817, 1261, 960; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_7\text{H}_{11}\text{N}_3\text{NaO}_2]^+$ : 192.1; Found: 192.1.

**2-(4-Bromo-butyl)-oxetan-3-one (6h)**

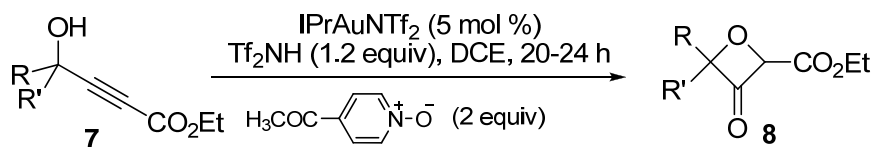
**6h**

Compound **6h** was prepared in 70% yield using **4** as oxidant according to General Procedure C. The reaction time was 3 h.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.49 – 5.45 (m, 1H), 5.30 (d, 1H,  $J = 14.8$  Hz), 5.23 (dd, 1H,  $J = 4.4, 14.8$  Hz), 3.41 (t, 2H,  $J = 6.4$  Hz), 1.95 – 1.82 (m, 4H), 1.71 – 1.56 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  202.9, 103.4, 88.9, 33.0, 32.3, 30.3, 22.9; IR (neat): 2935, 2870, 1817, 1439, 1273, 1030, 957; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_7\text{H}_{11}\text{BrNaO}_2]^+$ : 229.0; Found: 229.0.

### (2S)-2-phenethyloxetan-3-one



This chiral compound was prepared in 83% yield using **4** as oxidant according to General Procedure C. The reaction time was 4 h.  $[\alpha]_{\text{D}}^{20} = -39.5^\circ$  ( $c = 0.50$ ,  $\text{CHCl}_3$ ). 81% ee [determined by HPLC: Chiralpak IA Column, 1/99 *i*PrOH /hexane, 0.6 mL/min, 254 nm; TR = 15.09 min (major), 16.59 min (minor)].  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.32 – 7.19 (m, 5H), 5.49 – 5.45 (m, 1H), 5.48 – 5.44 (m, 1H), 5.31 (d, 1H,  $J = 15.0$  Hz), 2.84 – 2.76 (m, 2H), 2.24 – 2.10 (m, 2H).

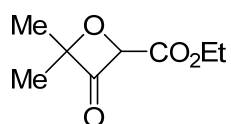


**General Procedure D:** 4-Acetylpyridine *N*-oxide (0.60 mmol),  $\text{Tf}_2\text{NH}$  (1.8 mL, 0.20 M in DCE), and  $\text{IPrAuNTf}_2$  (13.9 mg, 0.015 mmol) were added sequentially to a solution of tertiary propargyl alcohol **7** (0.30 mmol) in DCE (1.2 mL) at room temperature. The



reaction mixture was stirred at 40 °C or 60 °C, and the progress of the reaction was monitored by TLC. The reaction typically took 20 – 24 h. Upon completion, the reaction was treated with saturated aqueous NaHCO<sub>3</sub> (15 mL), and the resulting solution was extracted with DCM (2 × 30 mL). The combined organic layers were dried with MgSO<sub>4</sub>. The mixture was concentrated and the residue was purified by silica gel flash chromatography (eluent: hexanes/ethyl acetate) to afford desired products **8**.

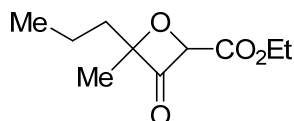
#### 4,4-Dimethyl-3-oxooxetane-2-carboxylic acid ethyl ester (**8a**)



**8a**

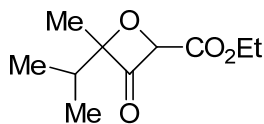
Compound **8a** was prepared in 82% yield according to General Procedure D. The reaction time was 20 h. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 5.73 (s, 1H), 4.36 – 4.23 (m, 2H), 1.59 (s, 3H), 1.56 (s, 3H), 1.32 (t, 3H, *J* = 7.2 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 197.9, 165.1, 108.6, 93.6, 62.2, 22.9, 22.5, 14.1; IR (neat): 2985, 2931, 1836, 1747, 1466, 1369, 1215, 1030; MS (ES<sup>+</sup>) Calculated for [C<sub>8</sub>H<sub>12</sub>NaO<sub>4</sub>]<sup>+</sup>: 195.1; Found: 195.1.

#### 4-Methyl-3-oxo-4-propyl-oxetane-2-carboxylic acid ethyl ester (**8b**)

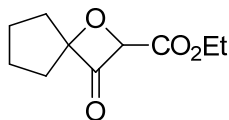


**8b**

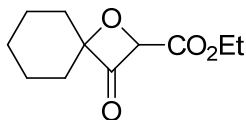
Compound **8b** (dr: 1.8/1) was prepared in 78% yield according to General Procedure D. The reaction time was 24 h. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 5.70 (s, 0.4H), 5.63 (s, 0.6H), 4.33 – 4.23 (m, 2H), 1.84 – 1.76 (m, 2H), 1.63 – 1.53 (m, 4H), 1.44 – 1.30 (m, 4H), 0.97 – 0.92 (m, 3H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 198.4, 198.2, 165.1, 164.6, 111.7, 111.0, 93.9, 93.2, 62.2, 62.1, 38.8, 38.2, 21.1, 21.0, 17.0, 16.8, 14.2, 14.1; IR (neat): 2970, 1824, 1743, 1462, 1200, 1014; Calculated for [C<sub>10</sub>H<sub>16</sub>NaO<sub>4</sub>]<sup>+</sup>: 223.1; Found: 223.1.

**4-Isopropyl-4-methyl-3-oxo-oxetane-2-carboxylic acid ethyl ester (8c)****8c**

Compound **8c** (dr: 2/1) was prepared in 72% yield according to General Procedure D. The reaction time was 24 h.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.70 (s, 0.33H), 5.60 (s, 0.67H), 4.36 – 4.18 (m, 2H), 2.22 (heptet, 0.33H,  $J = 8.5$  Hz), 2.09 (heptet, 0.67H,  $J = 8.5$  Hz), 1.53 (s, 2H), 1.48 (s, 1H), 1.35 – 1.26 (m, 3H), 1.06 – 0.98 (m, 6H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  198.4, 198.3, 165.1, 164.5, 114.3, 113.4, 93.9, 92.9, 62.2, 62.0, 34.0, 33.1, 17.8, 17.3, 16.7, 16.6, 16.5, 16.3, 14.2, 14.0; IR (neat): 2978, 1820, 1743, 1458, 1203, 1014; Calculated for  $[\text{C}_{10}\text{H}_{16}\text{NaO}_4]^+$ : 223.1; Found: 223.1.

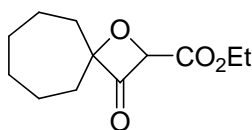
**3-Oxo-1-oxa-spiro[3.4]octane-2-carboxylic acid ethyl ester (8d)****8d**

Compound **8d** was prepared in 80% yield according to General Procedure D. The reaction time was 20 h.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.65 (s, 1H), 4.34 – 4.24 (m, 2H), 2.38 – 2.31 (m, 1H), 2.16 (t, 2H,  $J = 7.6$  Hz), 2.13 – 2.05 (m, 1H), 1.86 – 1.80 (m, 2H), 1.71 – 1.61 (m, 2H), 1.30 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  199.5, 165.0, 117.4, 94.1, 62.1, 36.4, 36.2, 24.8, 24.7, 14.1; IR (neat): 2966, 2877, 1828, 1747, 1446, 1203, 1022, 987; Calculated for  $[\text{C}_{10}\text{H}_{14}\text{NaO}_4]^+$ : 221.1; Found: 221.1.

**3-Oxo-1-oxa-spiro[3.5]nonane-2-carboxylic acid ethyl ester (8e)****8e**

Compound **8e** was prepared in 83% yield according to General Procedure D. The reaction time was 20 h.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.65 (s, 1H), 4.34 – 4.21 (m, 2H), 2.05 – 1.97 (m, 2H), 1.94 – 1.80 (m, 2H), 1.78 – 1.68 (m, 2H), 1.67 – 1.54 (m, 2H), 1.50 – 1.34 (m, 2H), 1.30 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  198.1, 165.2, 110.7, 92.7, 62.1, 32.6, 32.1, 24.6, 22.2, 22.0, 14.1; IR (neat): 2943, 2862, 1824, 1747, 1446, 1196, 1007; Calculated for  $[\text{C}_{11}\text{H}_{16}\text{NaO}_4]^+$ : 235.1; Found: 235.1.

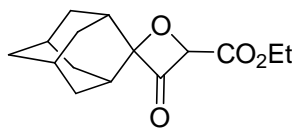
### 3-Oxo-1-oxa-spiro[3.6]decane-2-carboxylic acid ethyl ester (**8f**)



**8f**

Compound **8f** was prepared in 75% yield according to General Procedure D. The reaction time was 24 h.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  5.65 (s, 1H), 4.35 – 4.21 (m, 2H), 2.26 – 2.20 (m, 1H), 2.16 – 2.02 (m, 3H), 1.76 – 1.52 (m, 8H), 1.31 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  198.6, 165.3, 114.0, 93.0, 62.1, 35.1, 34.6, 28.9, 28.8, 22.0, 14.1; IR (neat): 2931, 2862, 1824, 1747, 1454, 1308, 1265, 1196, 1011, 960; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_{12}\text{H}_{18}\text{NaO}_4]^+$ : 249.1; Found: 249.1.

### Compound **8g**

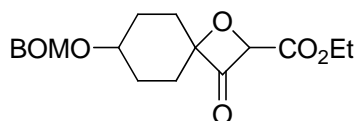


**8g**

Compound **8g** was prepared in 92% yield according to General Procedure D. The reaction time was 22 h.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.61 (s, 1H), 4.33 – 4.21 (m, 2H), 2.42 (s, 1H), 2.35 (s, 1H), 2.16 (d, 1H,  $J = 12.0$  Hz), 2.07 (d, 1H,  $J = 12.0$  Hz), 1.98 (t, 2H,  $J = 14.0$  Hz), 1.85 – 1.76 (m, 4H), 1.72 (s, 2H), 1.68 – 1.63 (m, 2H), 1.30 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  197.8, 165.4, 114.6, 92.3, 62.0, 36.3, 34.2, 34.1,

33.9, 33.6, 32.1, 32.0, 26.4, 26.1, 14.1; IR (neat): 2916, 2854, 1817, 1747, 1446, 1308, 1196, 1030, 984; MS ( $\text{ES}^+$ ) Calculated for  $[\text{C}_{15}\text{H}_{21}\text{O}_4]^+$ : 265.1; Found: 265.2.

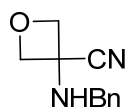
### 7-Benzyloxymethoxy-3-oxo-1-oxa-spiro[3.5]nonane-2-carboxylic acid ethyl ester (8h)



**8h**

Compound **8h** (dr: 10/1) was prepared in 74% yield according to General Procedure D. The reaction time was 24 h. Major isomer:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.38 – 7.28 (m, 5H), 5.69 (s, 1H), 4.81 (s, 2H), 4.62 (s, 2H), 4.33 – 4.25 (m, 2H), 3.75 – 3.71 (m, 1H), 2.28 – 2.14 (m, 2H), 1.96 – 1.82 (m, 6H), 1.32 (t, 3H,  $J = 7.2$  Hz);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  198.0, 165.1, 137.8, 128.4, 127.8, 127.7, 109.6, 93.0, 92.9, 71.5, 69.5, 62.2, 29.0, 28.5, 27.4, 27.3, 14.1; IR (neat): 2943, 1805, 1743, 1454, 1377, 1269, 1192, 1038; Calculated for  $[\text{C}_{19}\text{H}_{24}\text{NaO}_6]^+$ : 371.1; Found: 371.1.

### Preparation of 3-(benzylamino)oxetane-3-carbonitrile (9)

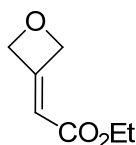


**9**

3-Methoxy-5-bromopyridine *N*-oxide (1.0 mmol),  $\text{TiF}_2\text{NH}$  (3.0 mL, 0.20 M in DCE), and (2-biphenyl) $\text{Cy}_2\text{PAuNTf}_2$  (20.7 mg, 0.025 mmol) were added sequentially to a solution of propargyl alcohol (28.0 mg, 0.50 mmol) in DCE (7.0 mL) at room temperature. The reaction mixture was stirred at rt for 0.5 h ( $^1\text{H}$  NMR monitoring of this reaction progress was carried out under the same conditions except that  $\text{CD}_2\text{Cl}_2$  instead of DCE was used solvent). Then, benzylamine (73 mg, 0.73 mmol) and trimethylacetone nitrile (79 mg, 0.73 mmol) were added at rt. After stirring for 8 h, the mixture was concentrated and the residue was purified by chromatography on silica gel (eluent: hexanes/ethyl acetate) to afford the desired product. This compound is known and the spectroscopic data match

those reported.<sup>5</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.41 – 7.29 (m, 5H), 4.85 (d, 2H, *J* = 6.8 Hz), 4.47 (d, 2H, *J* = 6.8 Hz), 3.86 (s, 2H), 1.93 (bs, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 137.6, 128.7, 128.5, 127.9, 118.8, 79.2, 53.8, 49.5; IR (neat): 3313(bs), 2954, 2877, 2229, 1454, 1219, 987, 744, 698; MS (ES<sup>+</sup>) Calculated for [C<sub>11</sub>H<sub>12</sub>N<sub>2</sub>NaO]<sup>+</sup>: 211.1; Found: 211.1.

### Preparation of ethyl 2-(oxetan-3-ylidene)acetate



**10**

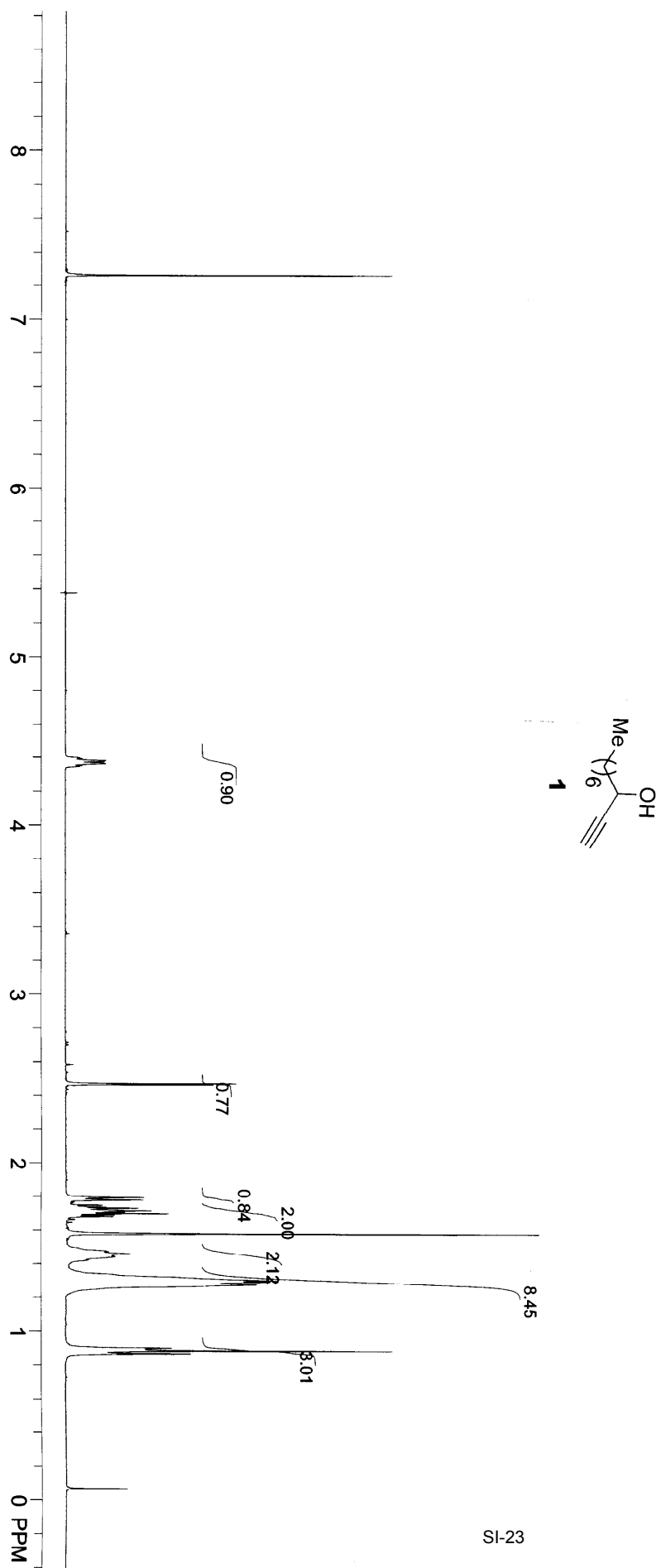
3-Methoxy-5-bromopyridine *N*-oxide (1.0 mmol), Tf<sub>2</sub>NH (3.0 mL, 0.20 M in DCE), and (2-biphenyl)Cy<sub>2</sub>PAuNTf<sub>2</sub> (20.7 mg, 0.025 mmol) were added sequentially to a solution of propargyl alcohol (28.0 mg, 0.50 mmol) in DCE (7.0 mL) at room temperature. The reaction mixture was stirred at rt for 0.5 h. Then, the reaction was treated with saturated aqueous NaHCO<sub>3</sub> (5 mL) and the resulting solution was extracted with DCM (1 × 10 mL) and the combined organic layers were dried with MgSO<sub>4</sub>. The mixture was filtrated and the residue was used directly for the next step.

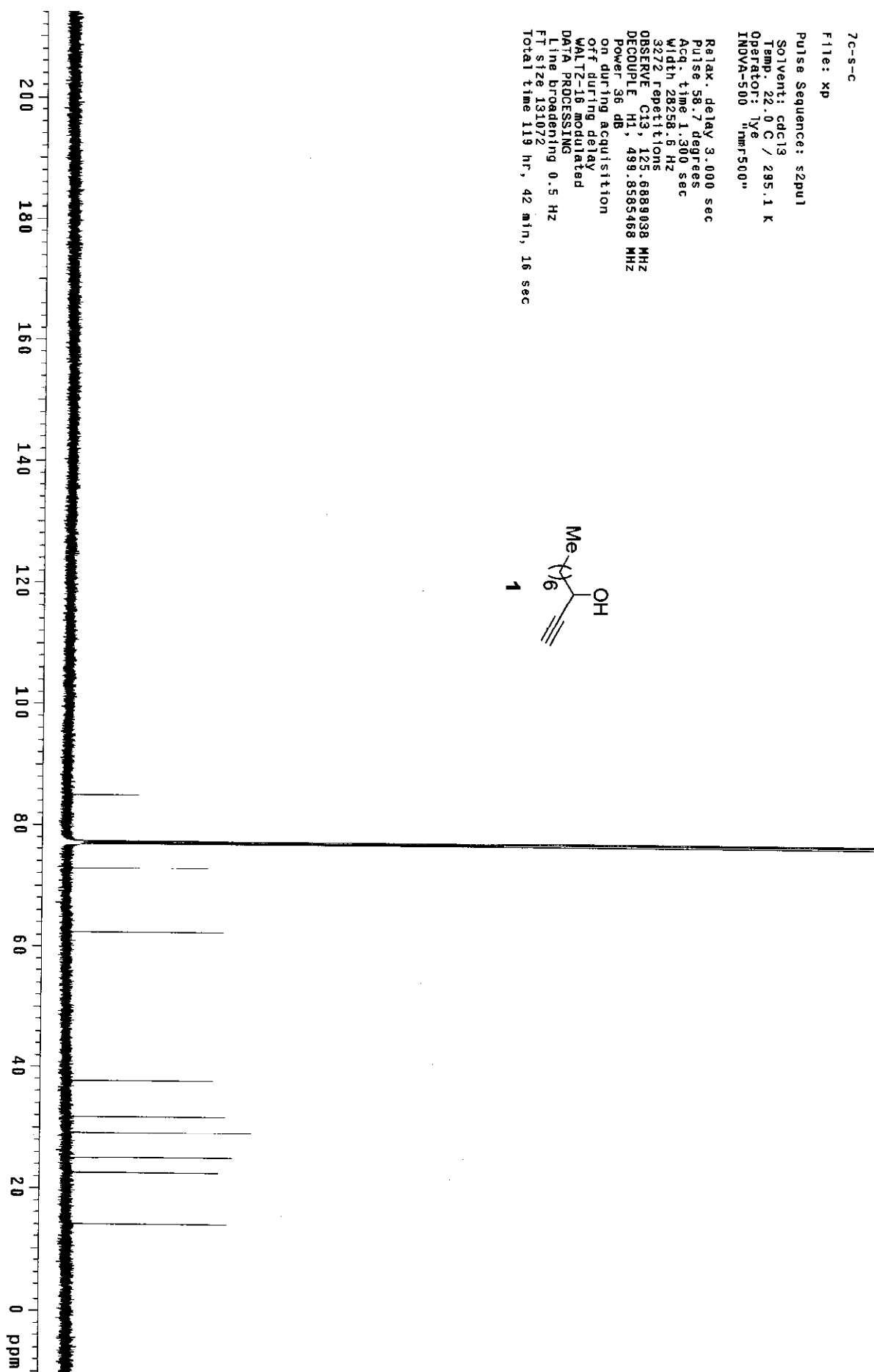
To the above solution was added carboethoxymethylene triphenylphosphorane (192 mg, 0.55 mmol, 1.5 equiv) at rt. After stirring for 5 h, the mixture was concentrated and the residue was purified by silica gel flash chromatography (eluent: hexanes/ethyl acetate) to afford the desired product. This compound is known and the spectroscopic data match those reported.<sup>6</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 5.63 – 5.61 (m, 1H), 5.50 – 5.48 (m, 2H), 5.30 – 5.28 (m, 2H), 4.15 (q, 2H, *J* = 7.2 Hz), 1.26 (t, 3H, *J* = 7.2 Hz); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 165.2, 159.1, 111.1, 81.0, 78.4, 60.3, 14.2; IR (neat): 2985, 2931, 2862, 1724, 1446, 1342, 1261, 1207, 1099, 1034, 960, 864, 833; MS (ES<sup>+</sup>) Calculated for [C<sub>7</sub>H<sub>10</sub>NaO<sub>3</sub>]<sup>+</sup>: 165.1; Found: 165.1.

### Reference:

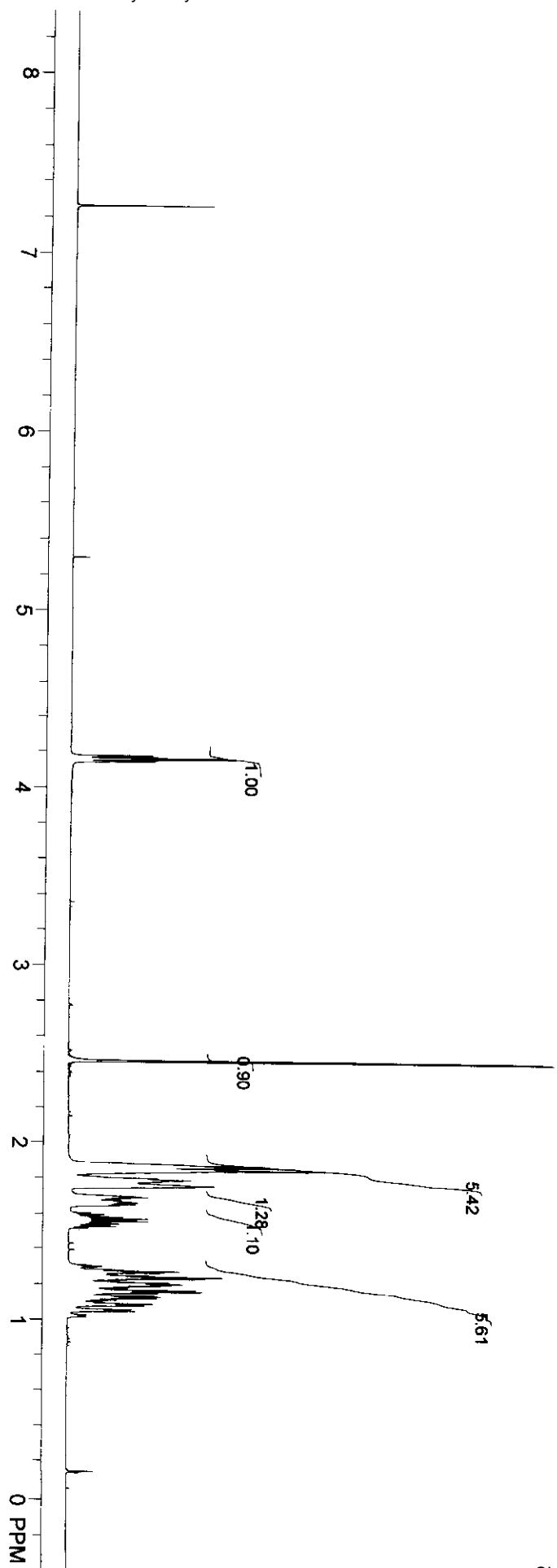
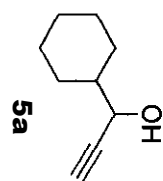
1. Cheng, X.; Jiang, X.; Yu, Y.; Ma, S. *J. Org. Chem.* **2008**, *73*, 8960–8965.

2. Zhang, G.; Zhang, L. *J. Am. Chem. Soc.* **2008**, *130*, 12598–12599.
3. Frantz, D. E.; Fässler, R.; Carreira, E. M. *J. Am. Chem. Soc.* **2000**, *122*, 1806–1807.
4. Clark, D. A.; Kulkarni, A. A.; Kalbarczyk, K.; Schertzer, B.; Diver, S. T. *J. Am. Chem. Soc.* **2006**, *128*, 15632–12636.
5. Kozikowski, A. P.; Fauq, A. H. *Synlett* **1991**, 783-784.
6. Wuitschik, G.; Rogers-Evans, M.; Müller, K.; Fischer, H.; Wagner, B.; Schuler, F.; Polonchuk, L.; Carreira, E. M. *Angew. Chem., Int. Ed.* **2006**, *45*, 7736-7739.









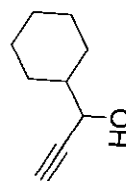
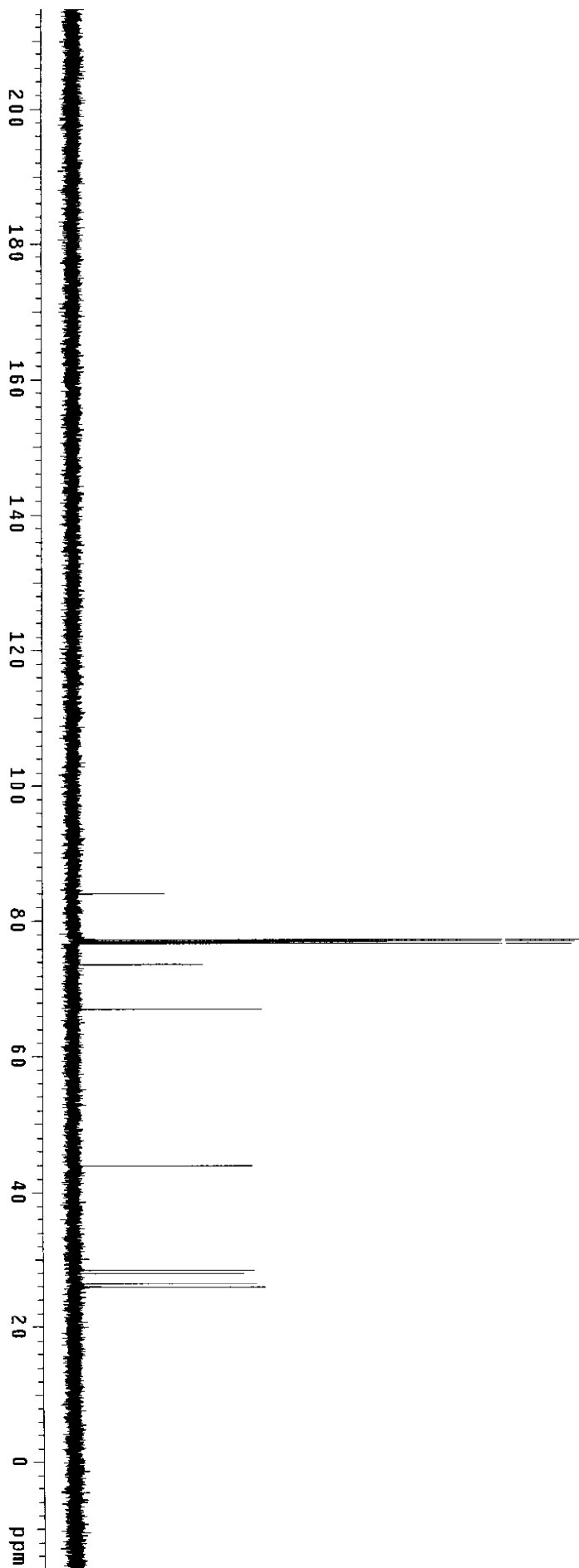
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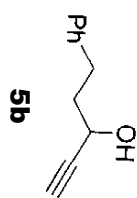
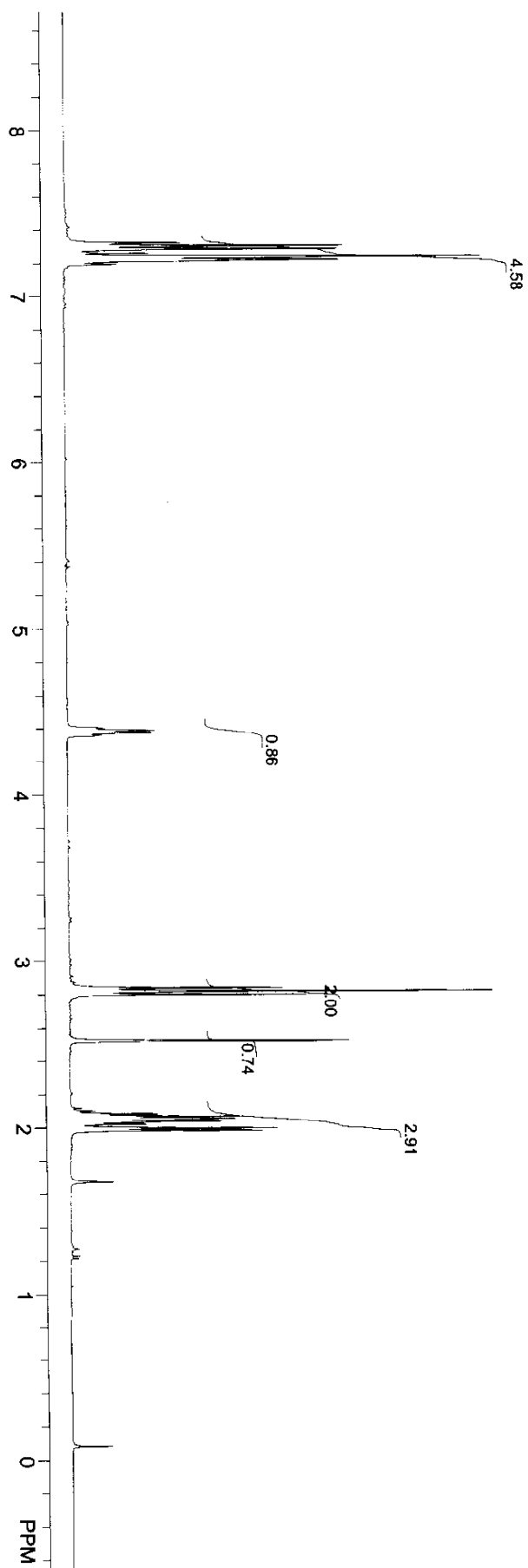
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Archive directory:  
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Sample directory:

File: CARBON

Pulse Sequence: s2pul  
Solvent: cdcl3  
Temp. 21.9 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec  
Pulse 58.7 degrees  
Acq. time 1.300 sec  
Width 40000.0 Hz  
104 repetitions  
OBSERVE C13, 125.6889847 MHz  
DECOUPLE H1, 499.8588575 MHz  
Power 36 dB  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 0.5 Hz  
FT size 131072  
Total time 0 min

**5a**



he-1-10-c

Data Collected on:  
nmr500-1nov4500  
Archive directory:  
/export/home/vnmr1/vnmrsys/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 22.0 C / 295.1 K

Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 4000.0 Hz

88 repetitions

OBSERVE C13, 125.6883884 MHz

DECUPLE H1, 499.8588575 MHz

Power 36 dB

on during acquisition

off during delay

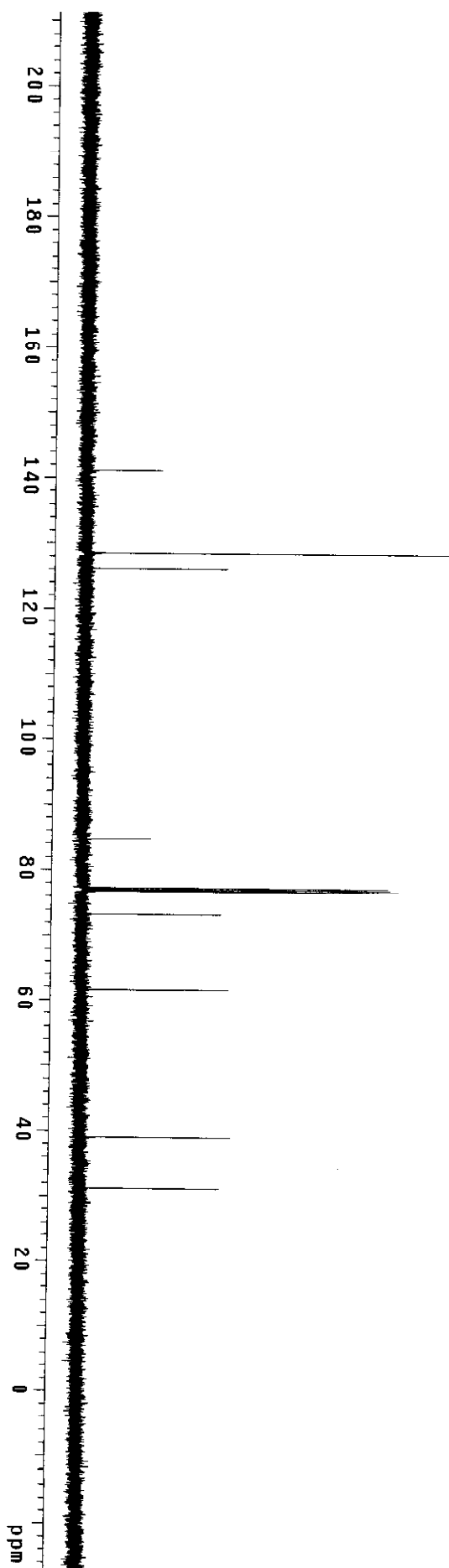
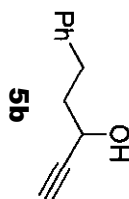
WALTZ-16 modulated

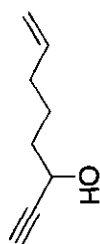
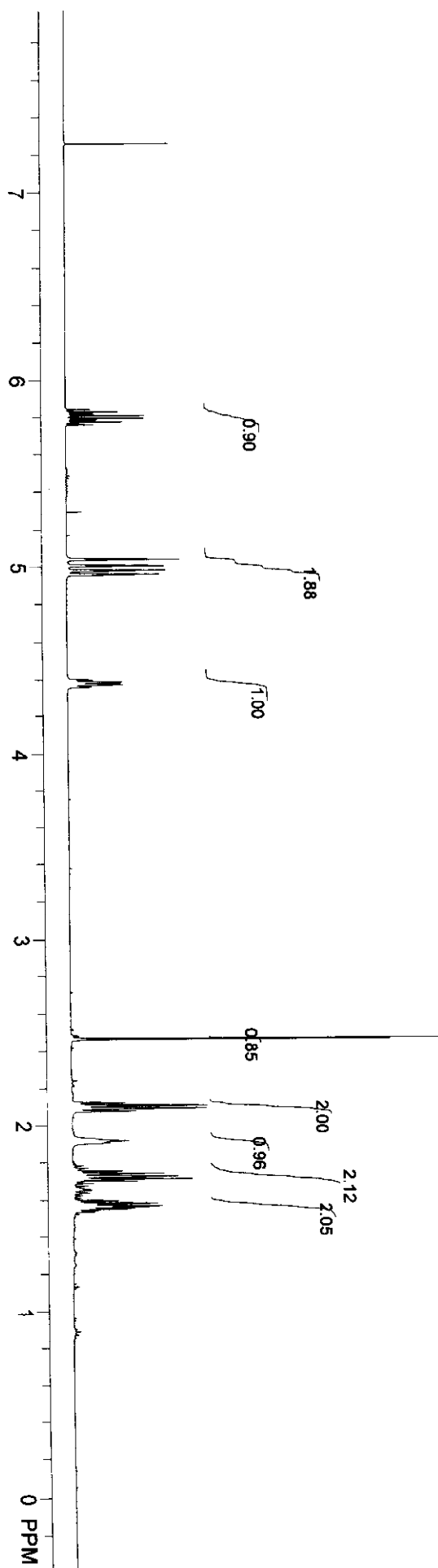
DATA PROCESSING

Line broadening 0.5 Hz

F1 size 131072

Total time 0 min



**5c**

v1w-3-266-c

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmrsys/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pul  
Solvent: cdcl3

Temp. 22.0 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 4000.0 Hz

124 repetitions

OBSERVE C13, 125.6889835 MHz

DECUPLE H1, 499.8588575 MHz

Power 36 dB

on during acquisition

off during delay

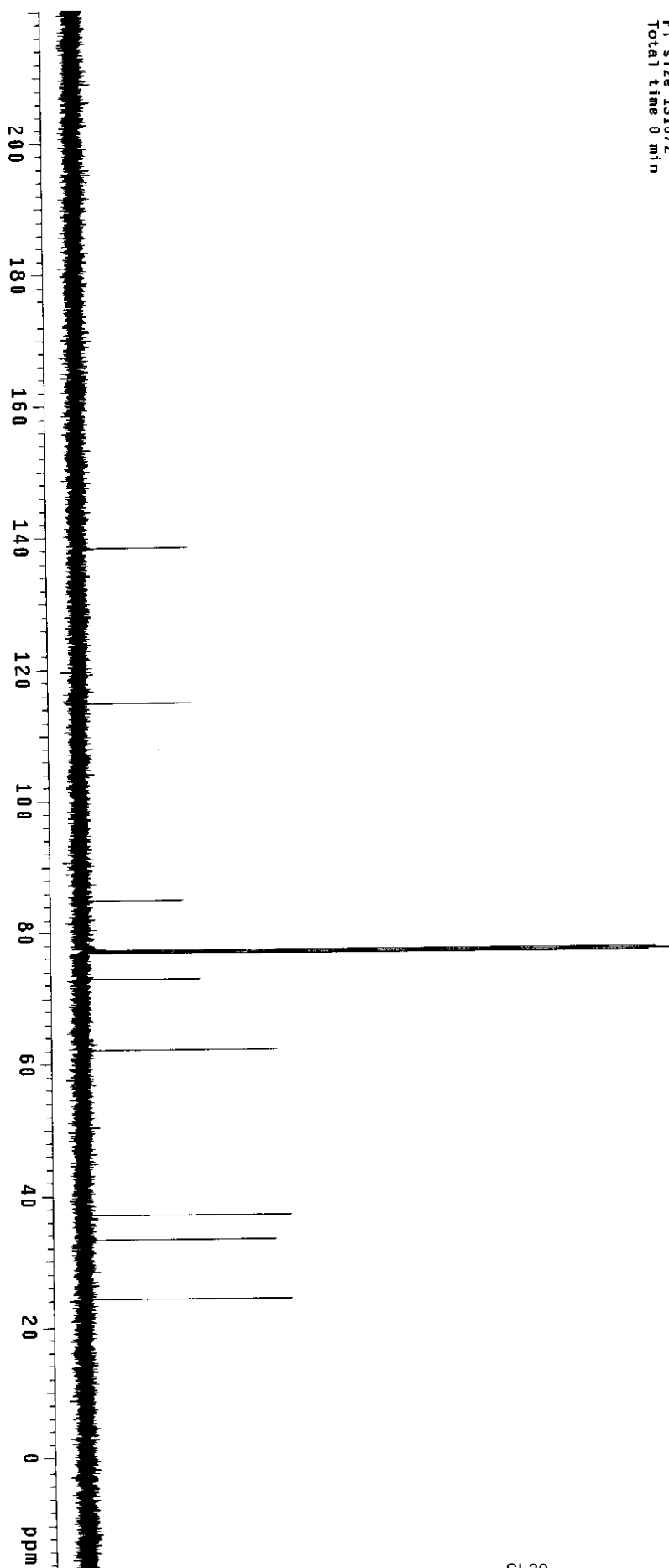
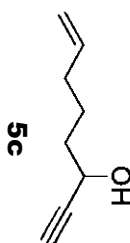
WALTZ-16 modulated

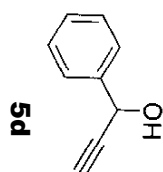
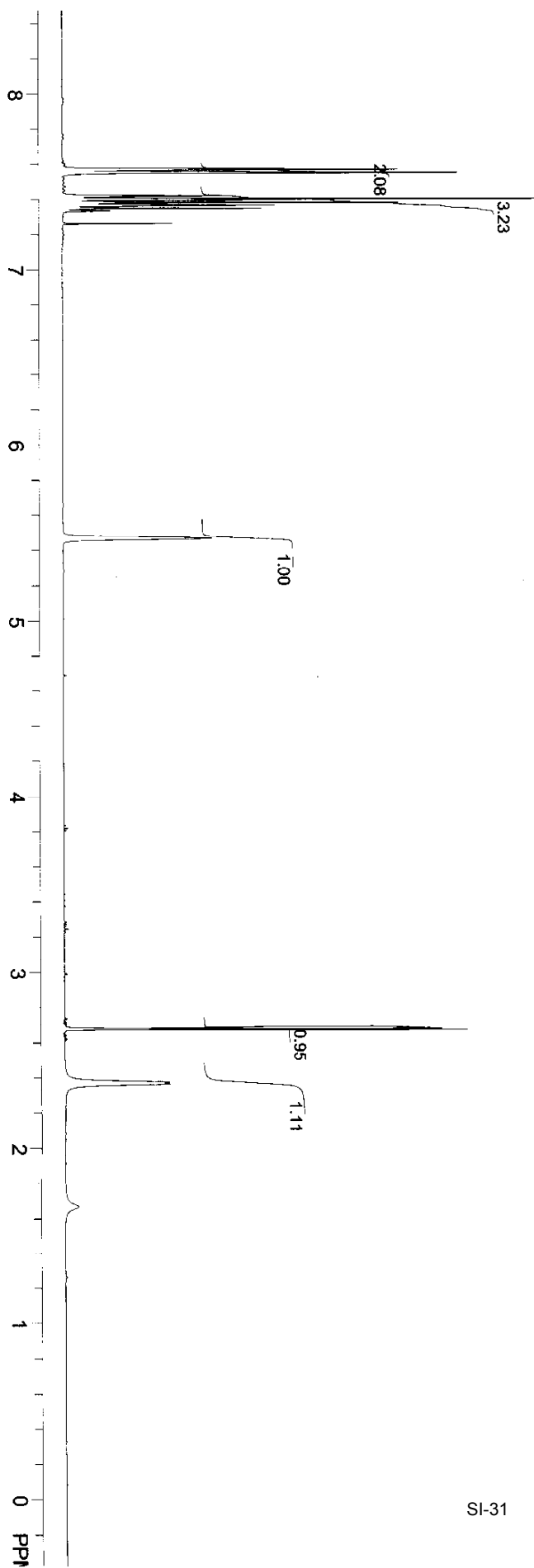
DATA PROCESSING

Line broadening 0.5 Hz

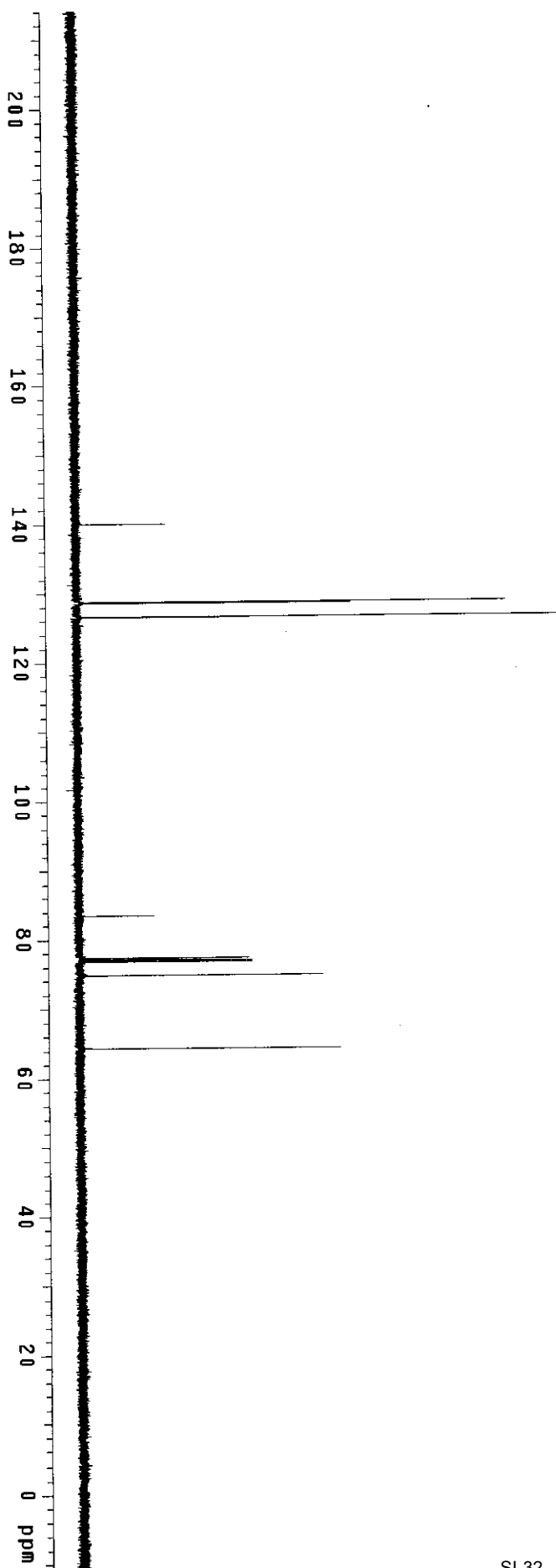
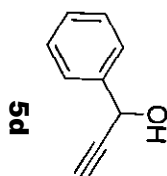
Fit size 131072

Total time 0 min

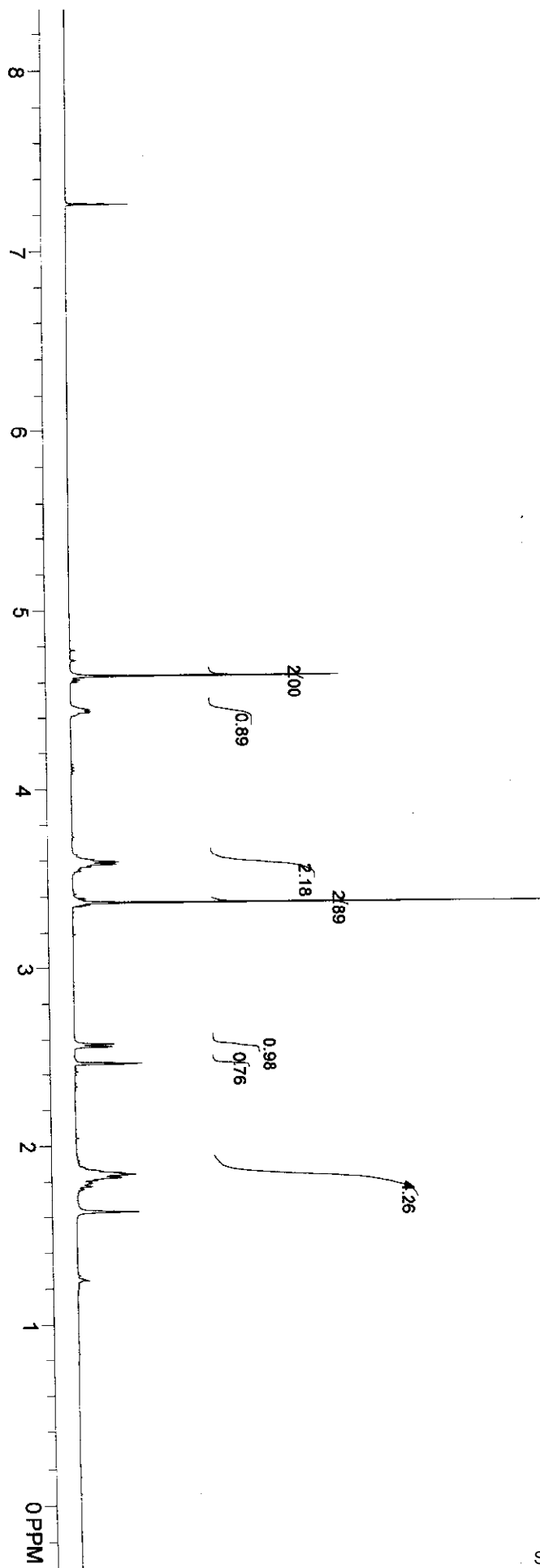
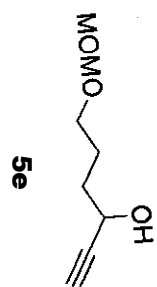


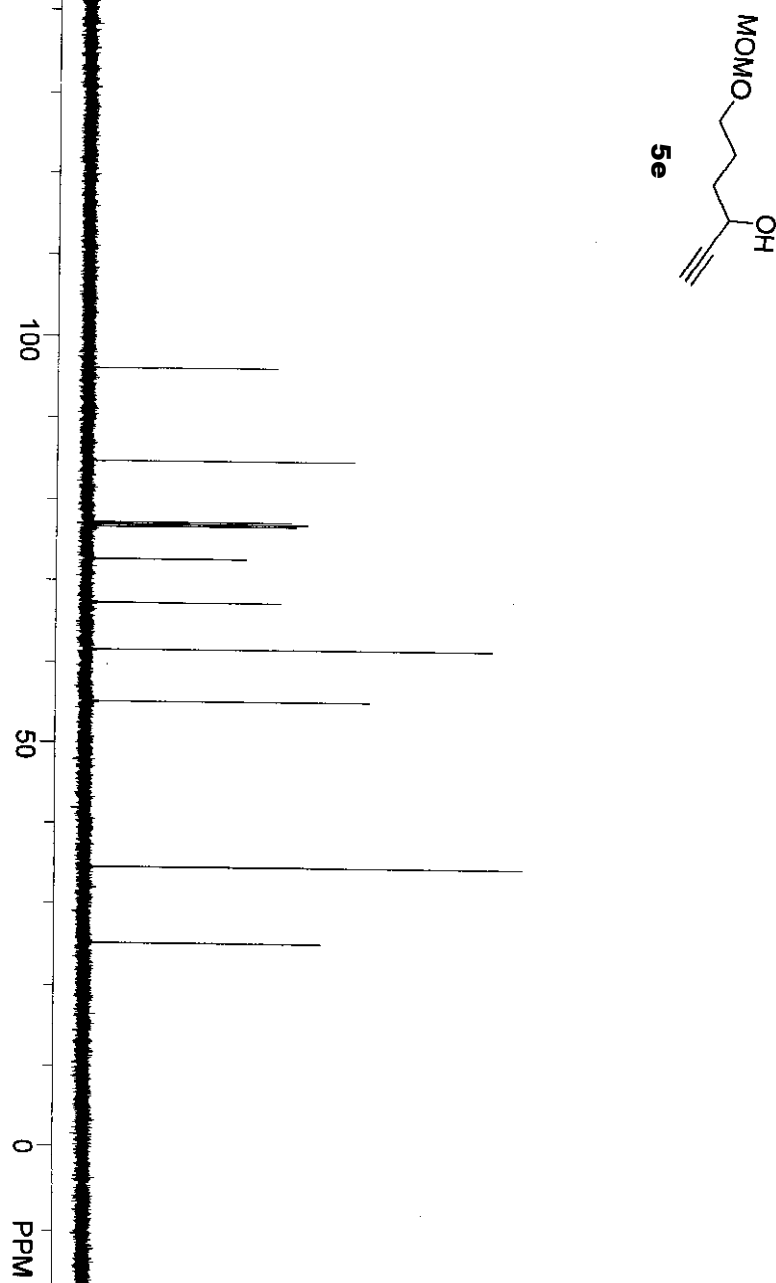


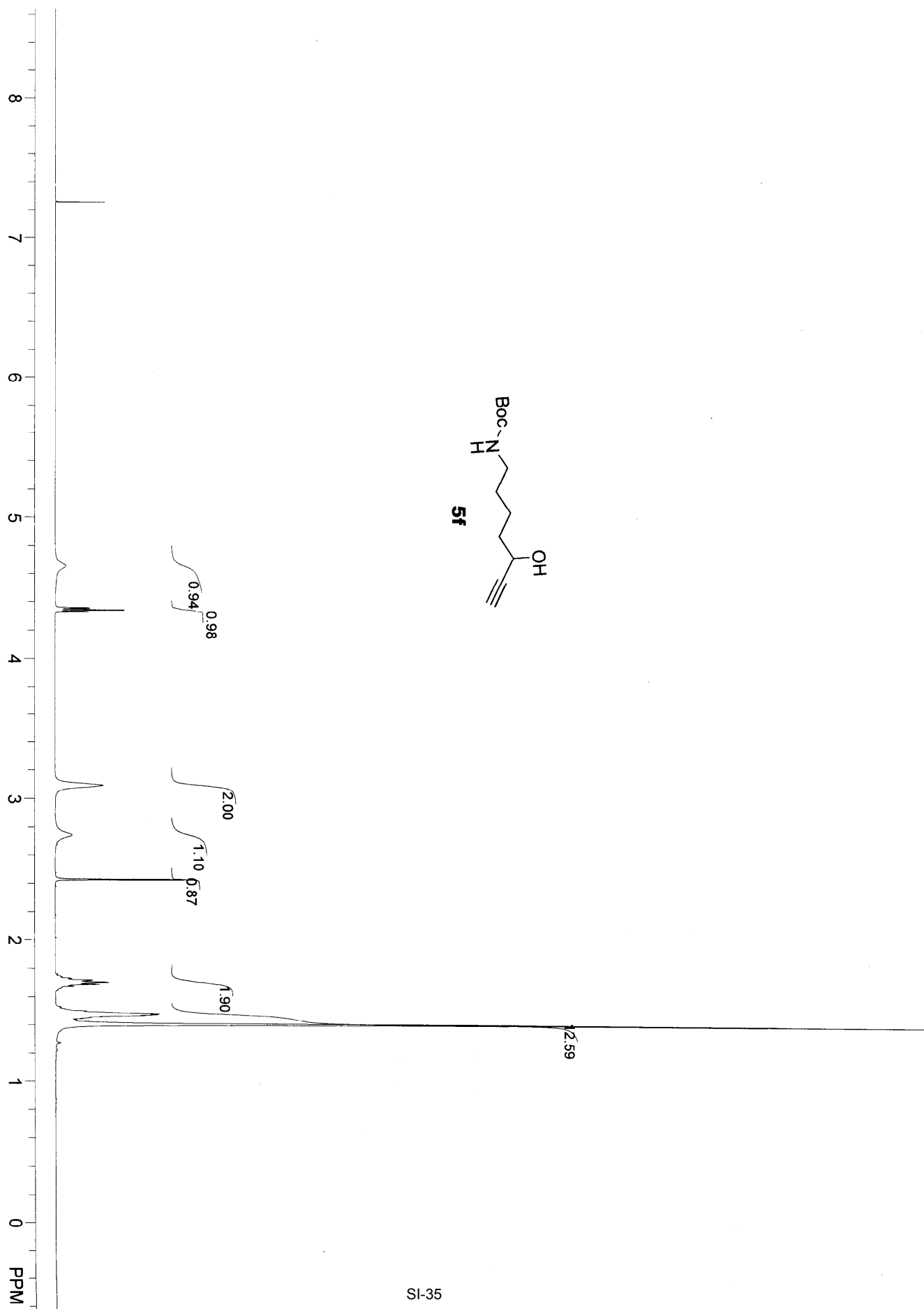
Y1w-3-238-Ph-S-C  
File: xp  
Pulse Sequence: szpu1  
Solvent: cdcl3  
Temp: 22.0 C / 295.1 K  
Operator: lye  
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Relax. delay 3.000 sec  
Pulse 58.7 degrees  
Acq. time 1.300 sec  
Width 28258.6 Hz  
48 repetitions  
OBSERVE C13, 125.6889032 MHz  
DECOUPLE H1, 499.8383466 MHz  
Power 36 dB  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 0.5 Hz  
FT size 131072  
Total time 11 hr, 58 min, 14 sec











y1w-3-296-NBOC-S-C

Data Collected on:  
nm500-inova500  
Archive directory:  
/export/home/vnmr1/vnmrSYS/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pu1

Solvent: cdcl3

Temp. 22.0 C / 295.1 K

Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 4000.0 Hz

246 repetitions

OBSERVE C13, 125.6889890 MHz

DECUPLE H1, 499.8588575 MHz

Power 36 dB

on during acquisition

off during delay

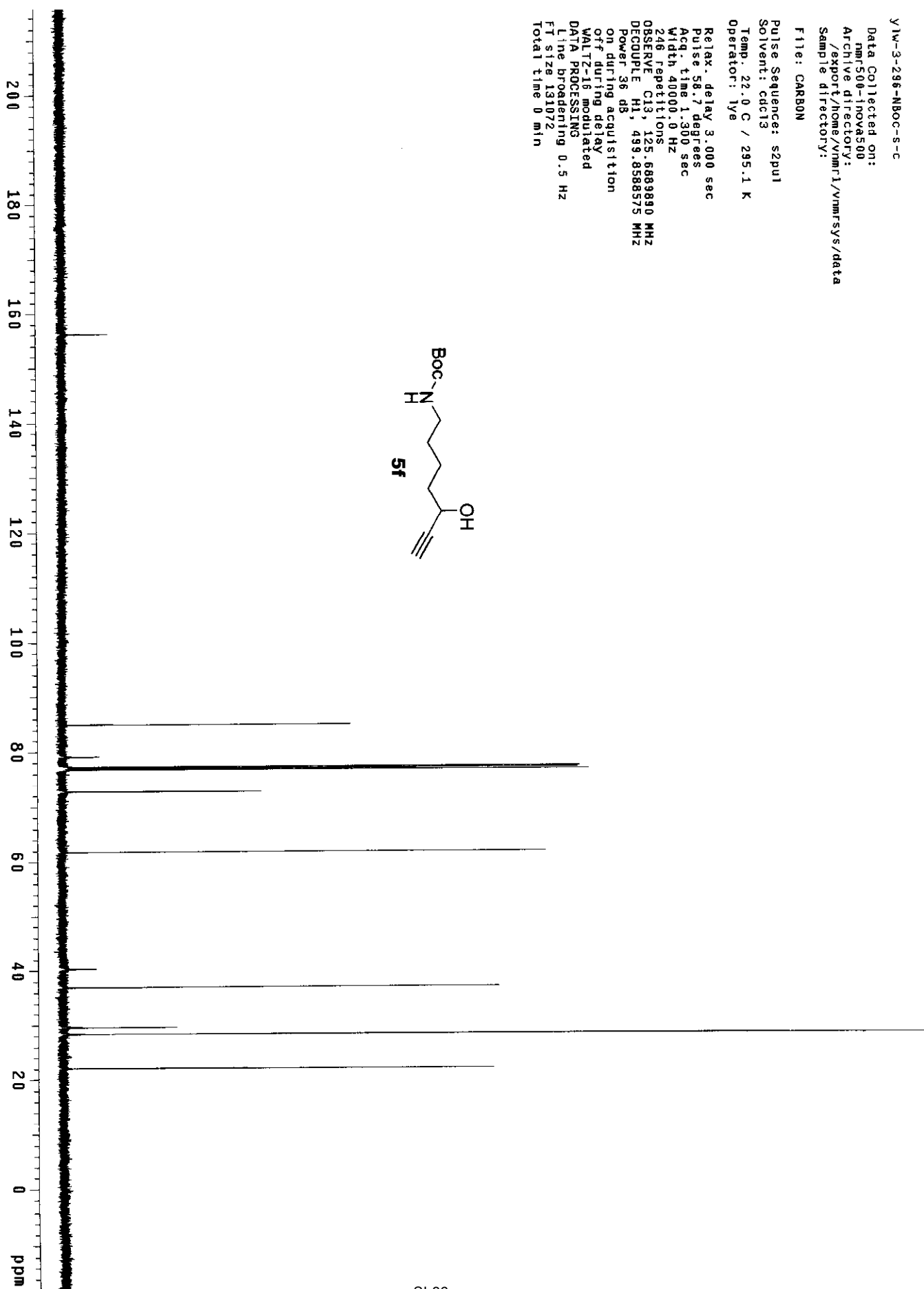
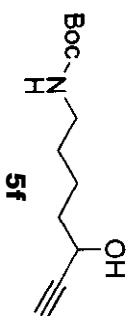
WALTZ-16 modulated

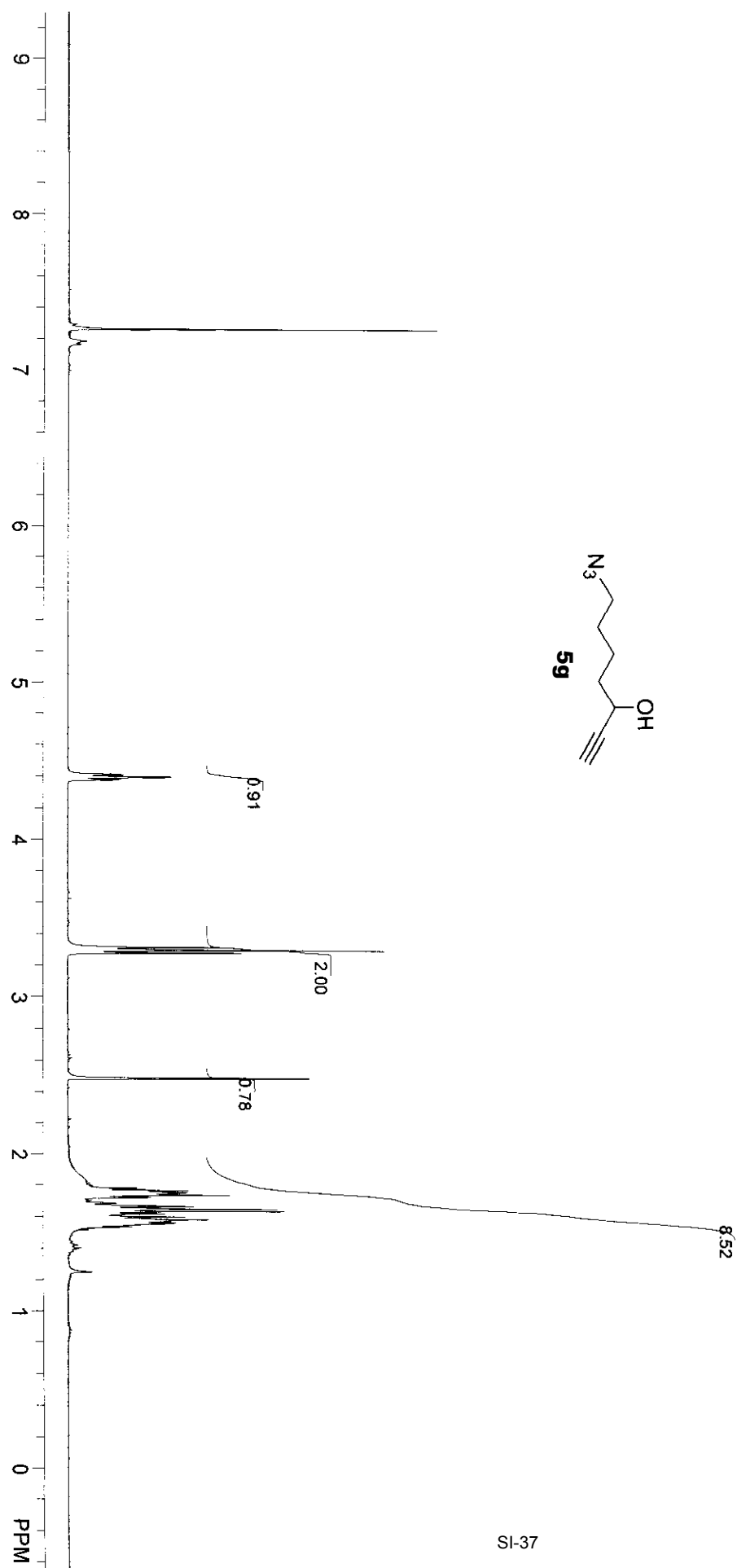
DATA PROCESSING

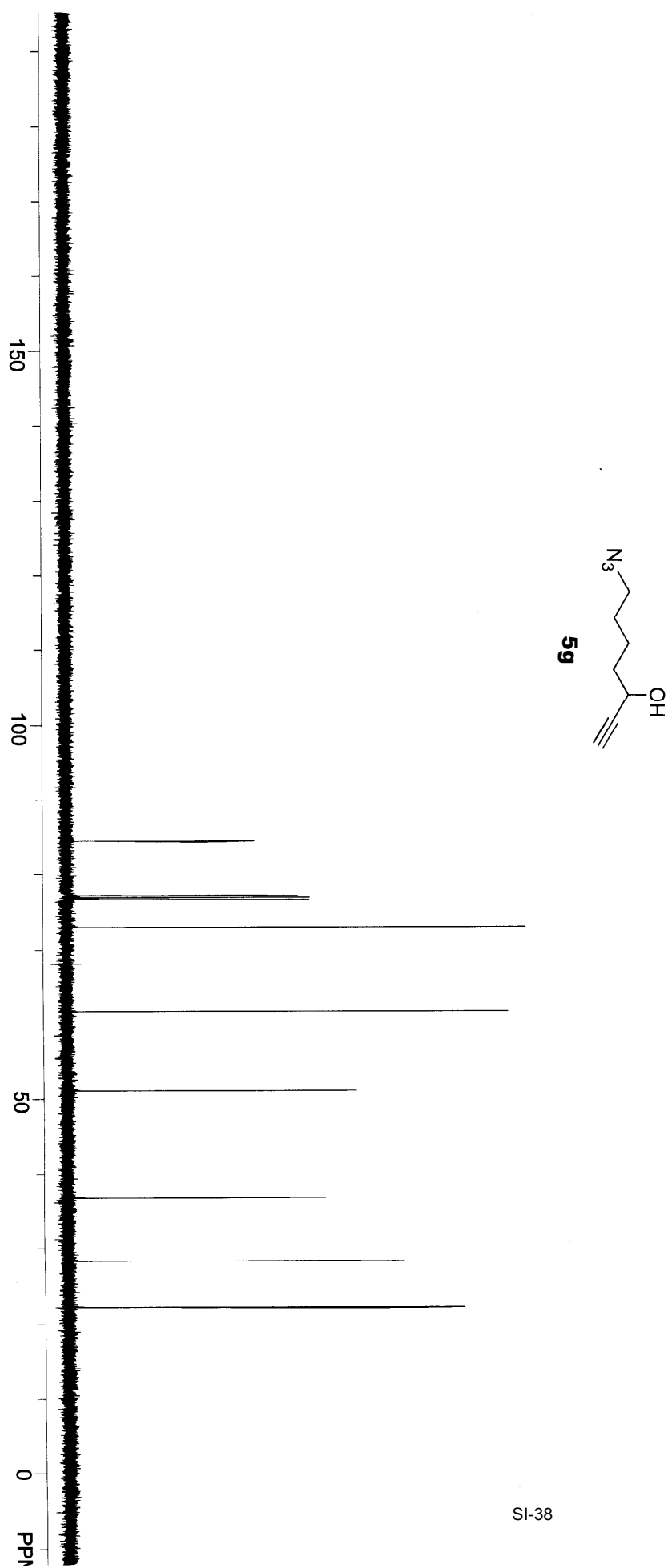
Line broadening 0.5 Hz

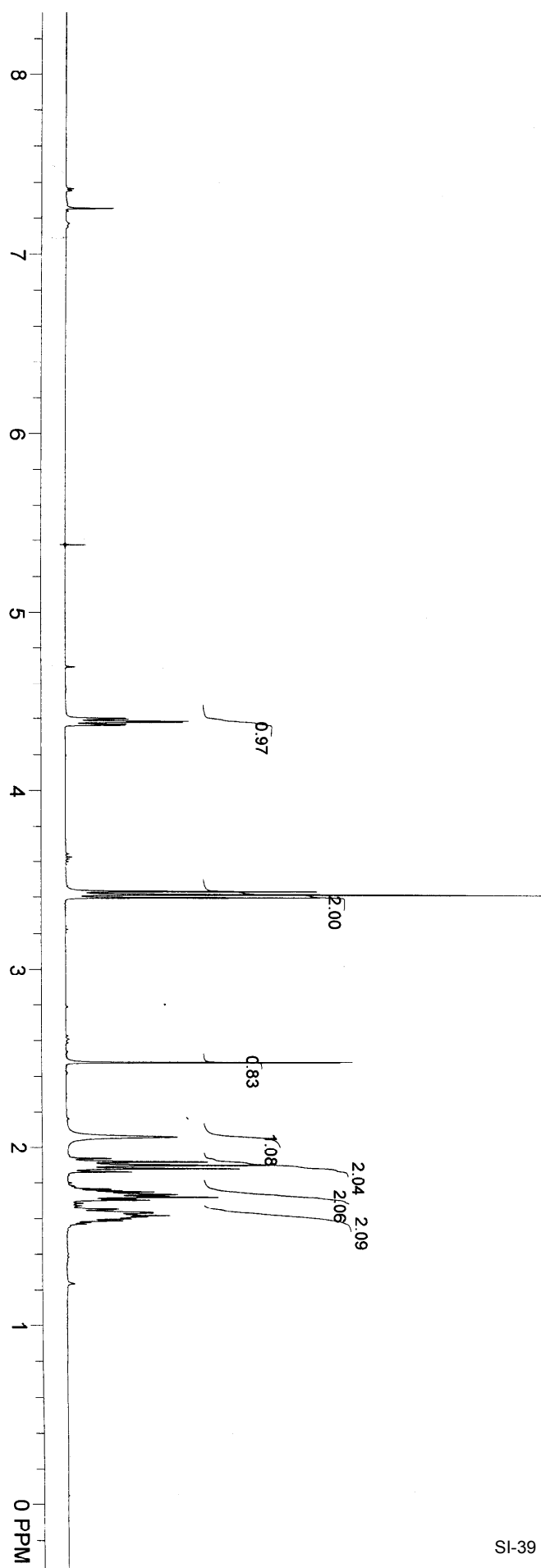
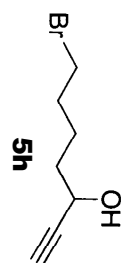
FT size 131072

Total time 0 min









y1w-3-302-Br-s

Data Collected on:  
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Archive directory:  
/export/home/vnmr1/vnmrSYS/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pul  
Solvent: cdcl3  
Temp. 21.9 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec  
Pulse 58.7 degrees  
Acq. time 1.300 sec  
Width 40000.0 Hz

88 repetitions

OBSERVE C13, 125.6888864 MHz  
DECUPLE H1, 499.8588575 MHz

Power 36 db

on during acquisition

off during delay

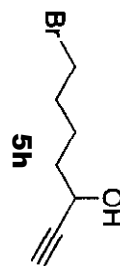
WALTZ-16 modulated

DATA PROCESSING

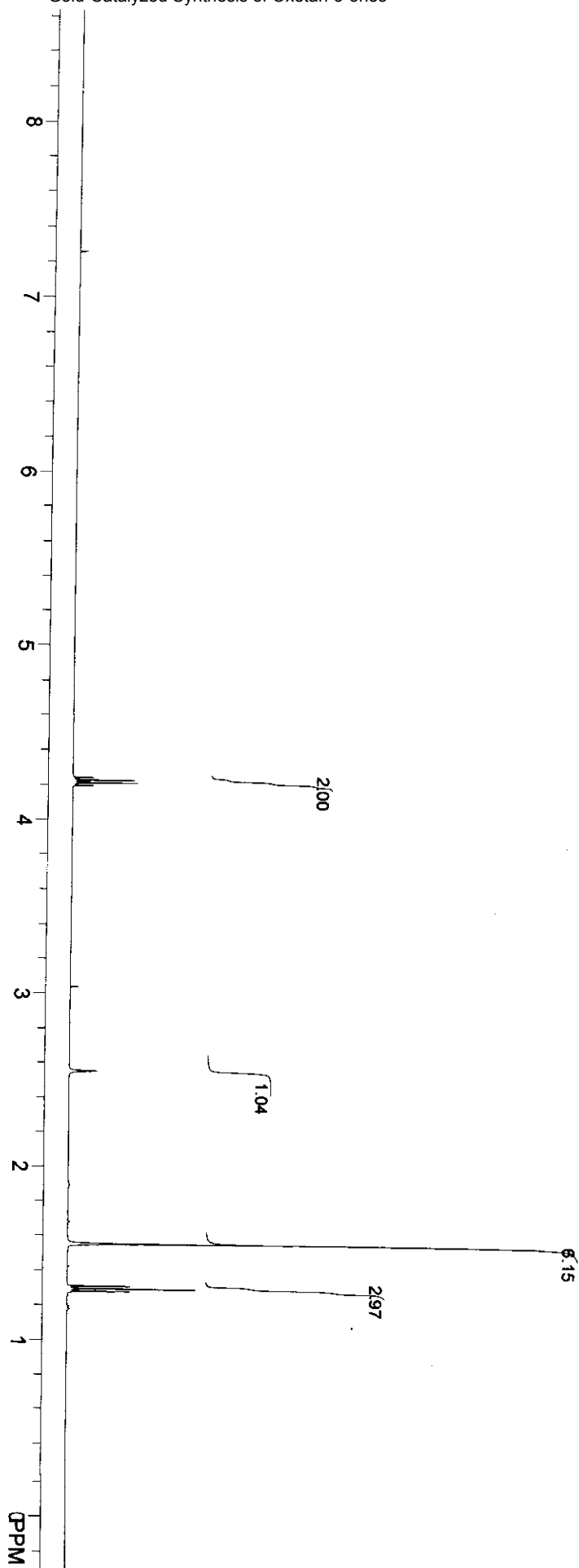
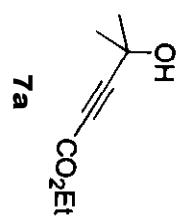
line broadening 0.5 Hz

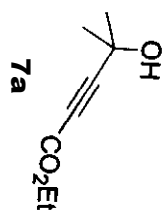
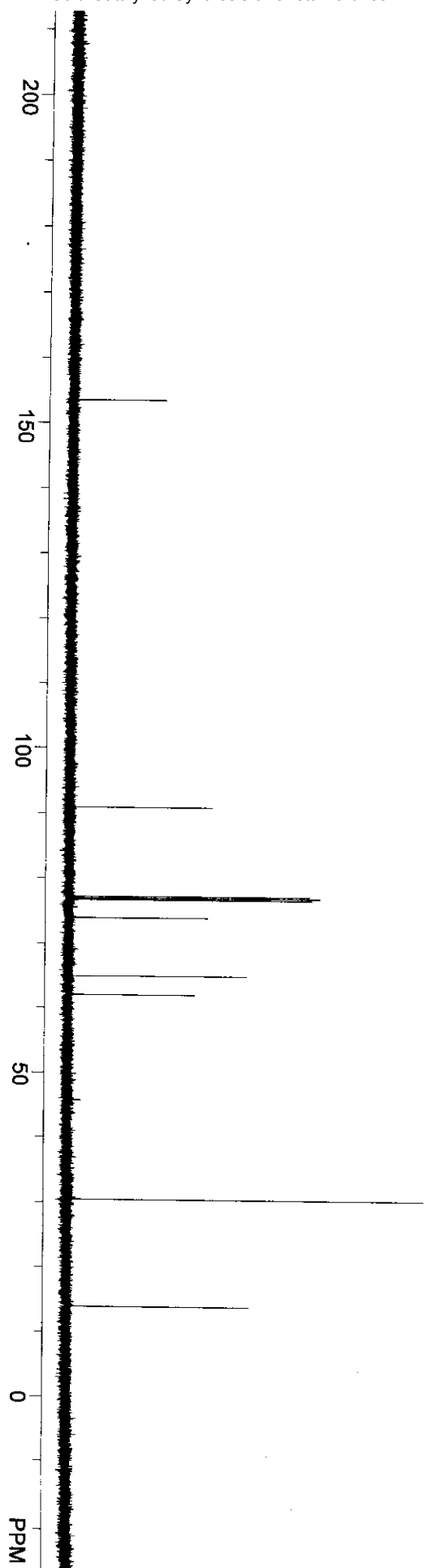
FT size 131072

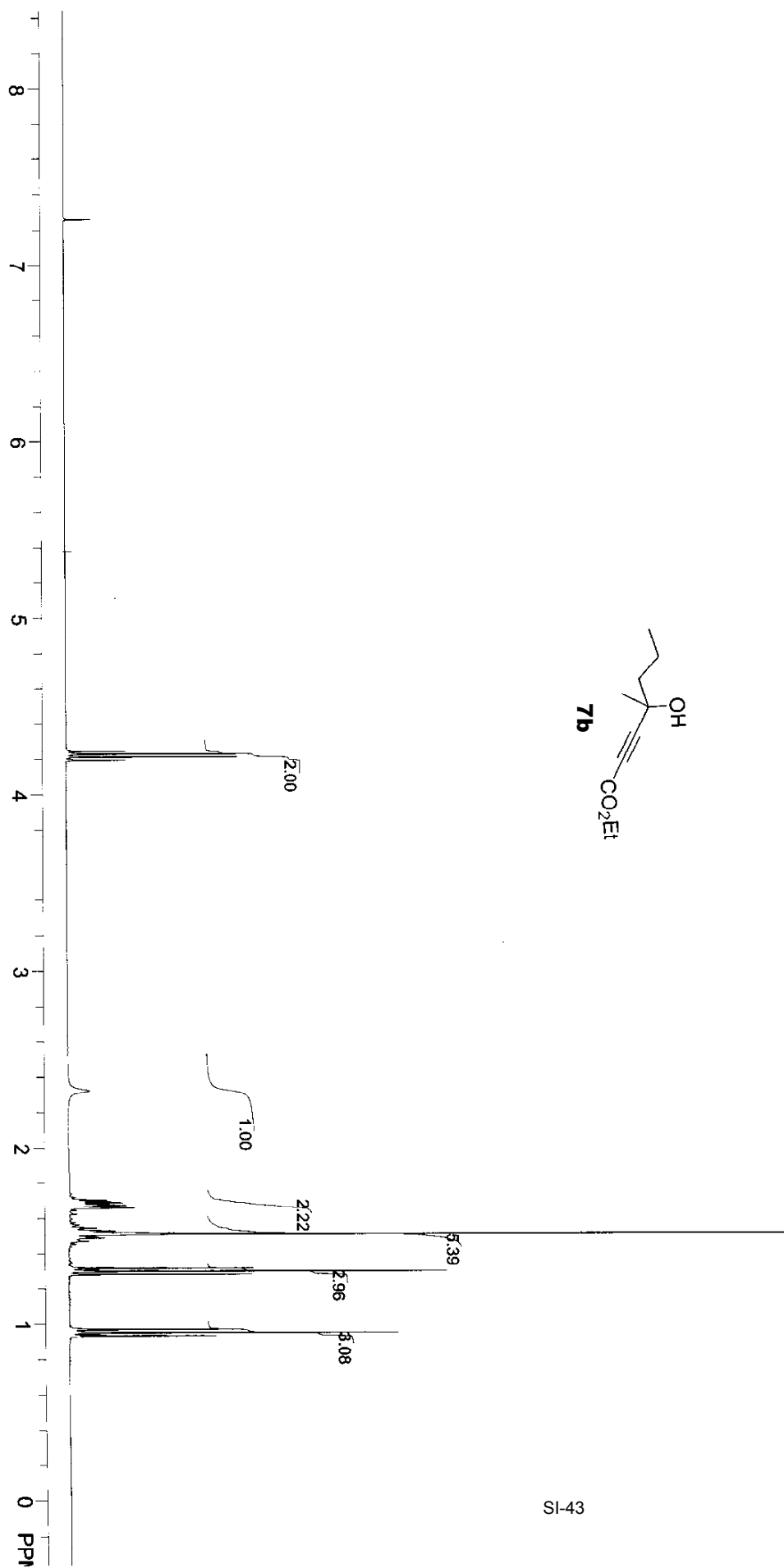
Total time 0 min











he-1-19-c

Data Collected on:  
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Archive directory:  
/export/home/vnmr1/vnmr-sys/data  
Sample directory:

File: CARBON

Pulse Sequence: szpul

Solvent: cdcl3

Temp. 22.0 C / 295.1 K

Operator: iye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 40000.0 Hz

146 repetitions

OBSERVE C13, 125.6889860 MHz

DECOUPLE H1, 499.8588575 MHz

Power 36 dB

on during acquisition

off during delay

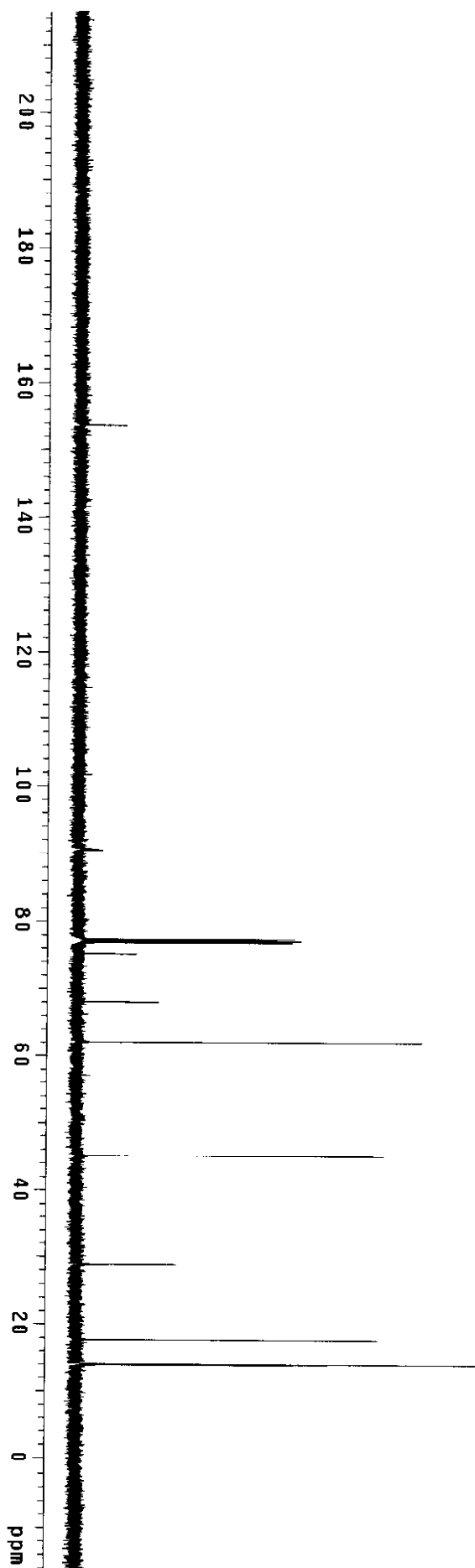
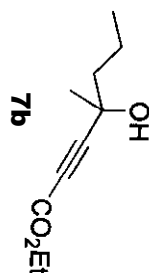
WALTZ-16 modulated

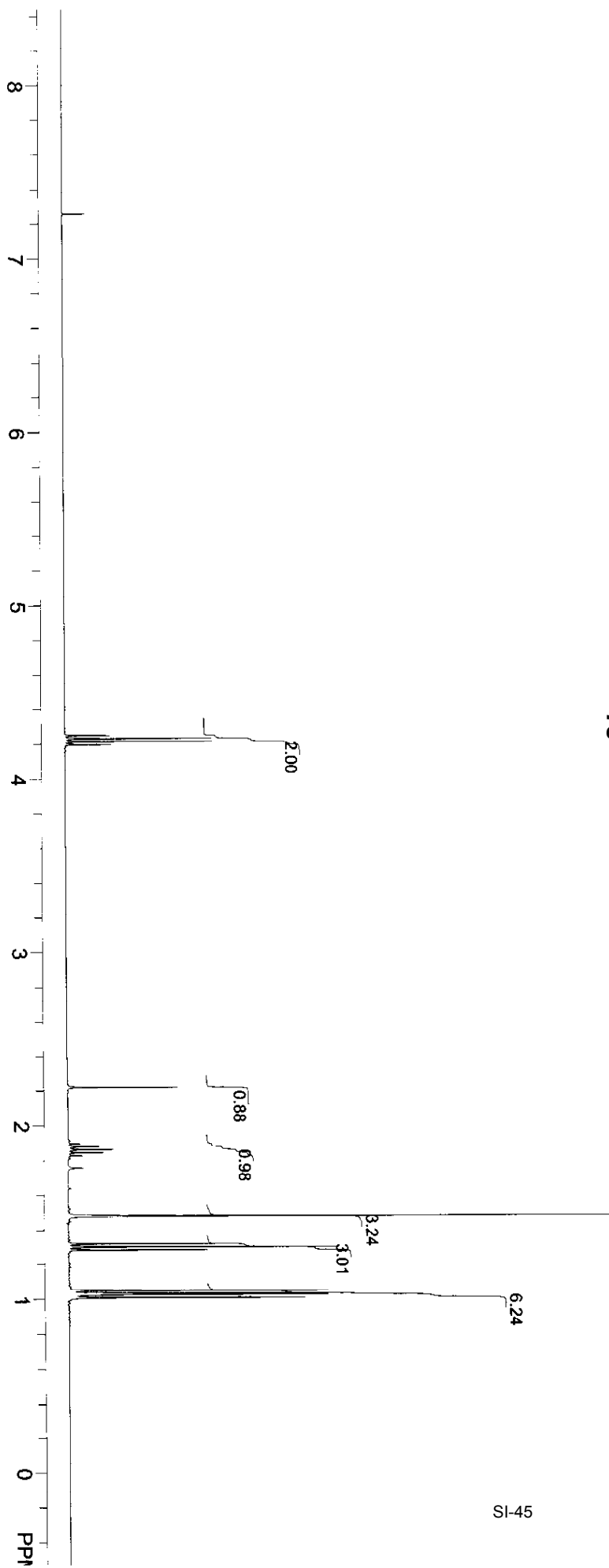
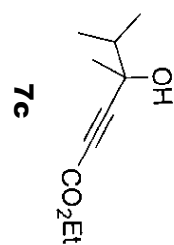
DATA PROCESSING

Line broadening 0.5 Hz

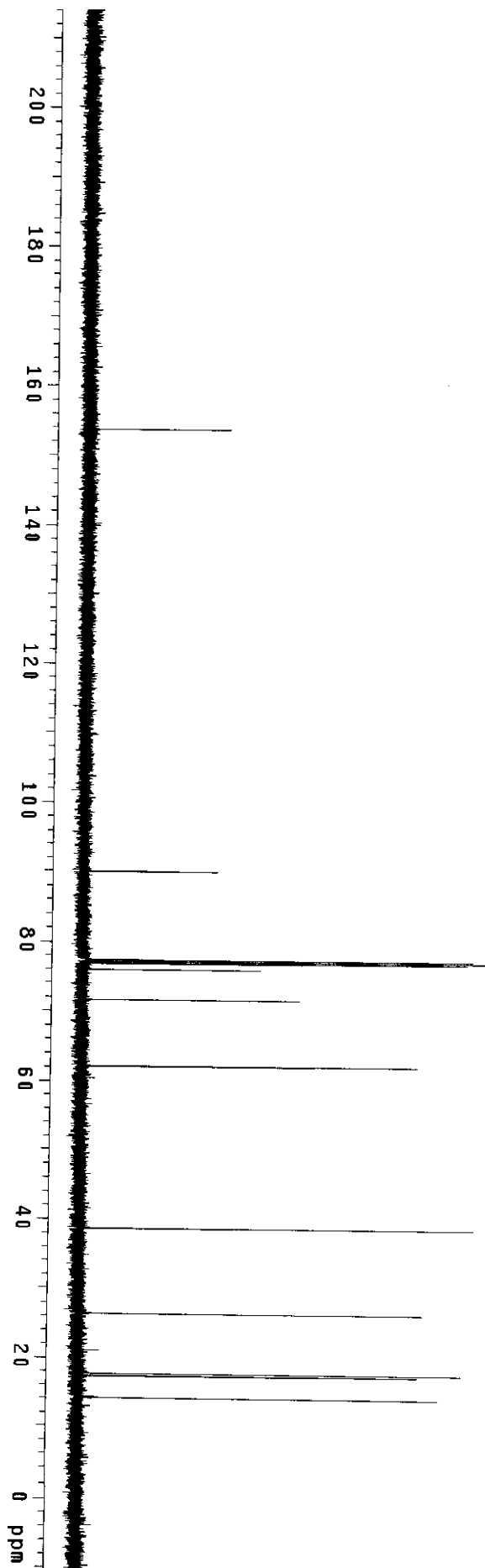
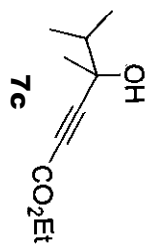
FT size 131072

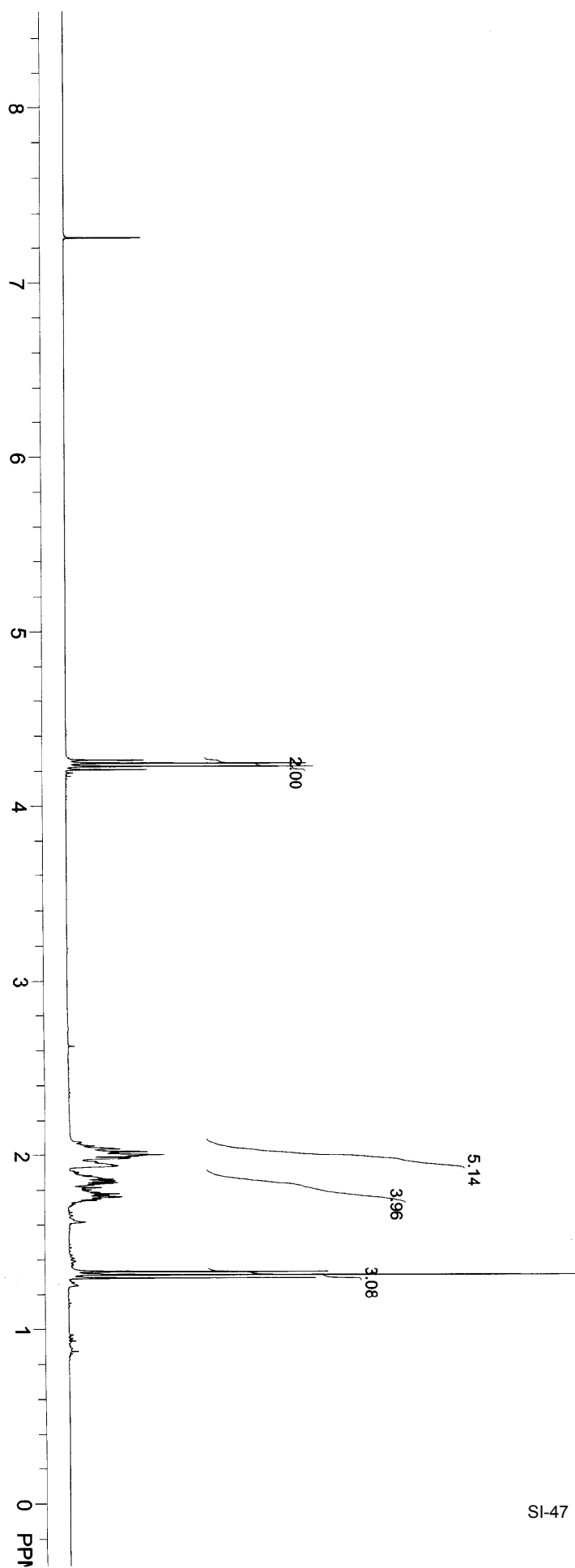
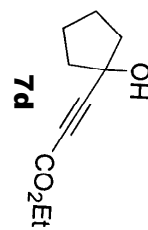
Total time 0 min





he-1-104-s-c  
File: xp  
Pulse Sequence: s2pu1  
Solvent: cdcl3  
Temp: 22.0 C / 295.1 K  
Operator: lye  
INOVA-500 "nmr500"  
Relax. delay 3.000 sec  
Pulse 58.7 degrees  
Acq. time 1.300 sec  
Width 28258.6 Hz  
74 Repetitions  
OBSERVE C13, 125.688032 MHz  
DECUPLE H1, 499.8585468 MHz  
Power 36 dB  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 0.5 Hz  
FI size 131072  
Total time 119 hr, 42 min, 16 sec





y1w-3-256-5membered-s-c

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Temp: 22.0 C / 295.1 K

Operator: lye

INOVA-500 "nmr500"

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.500 sec

Width 28258.6 Hz

156 repetitions

OBSERVE C13, 125.6889047 MHz

DECOUPLE H1, 499.8585468 MHz

Power 36 dB

on during acquisition

off during delay

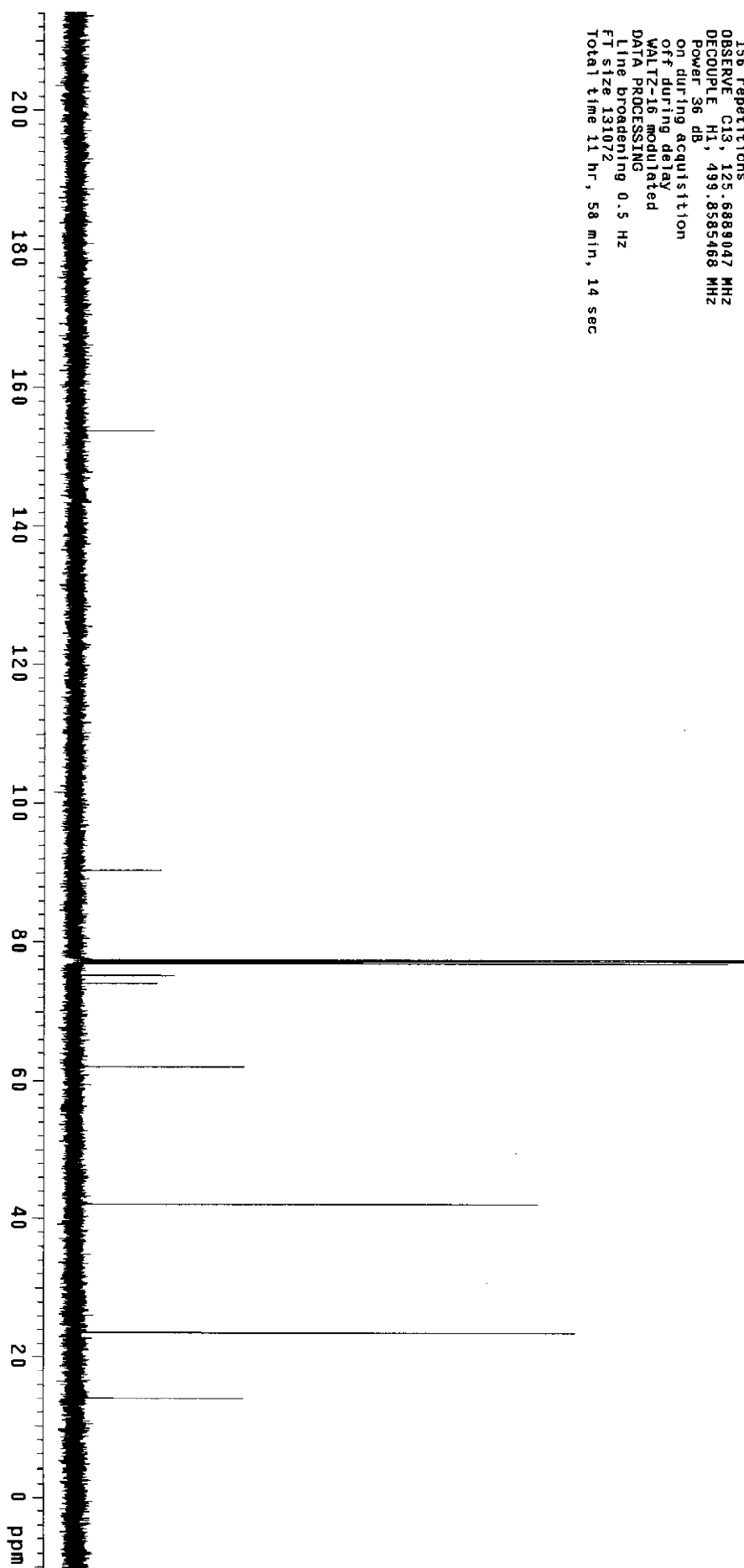
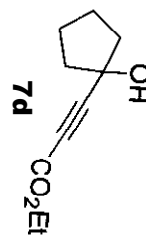
WALTZ-16 modulated

DATA PROCESSING

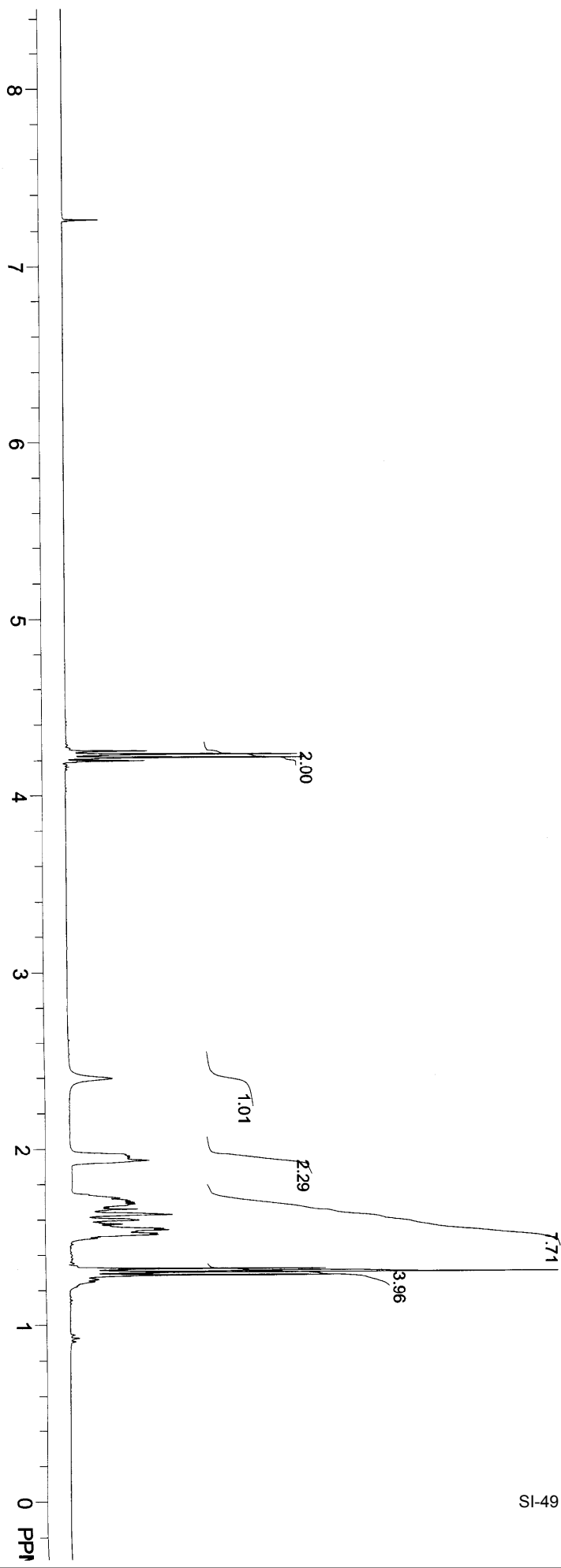
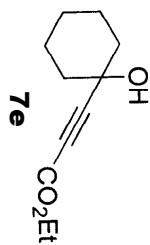
Line broadening 0.5 Hz

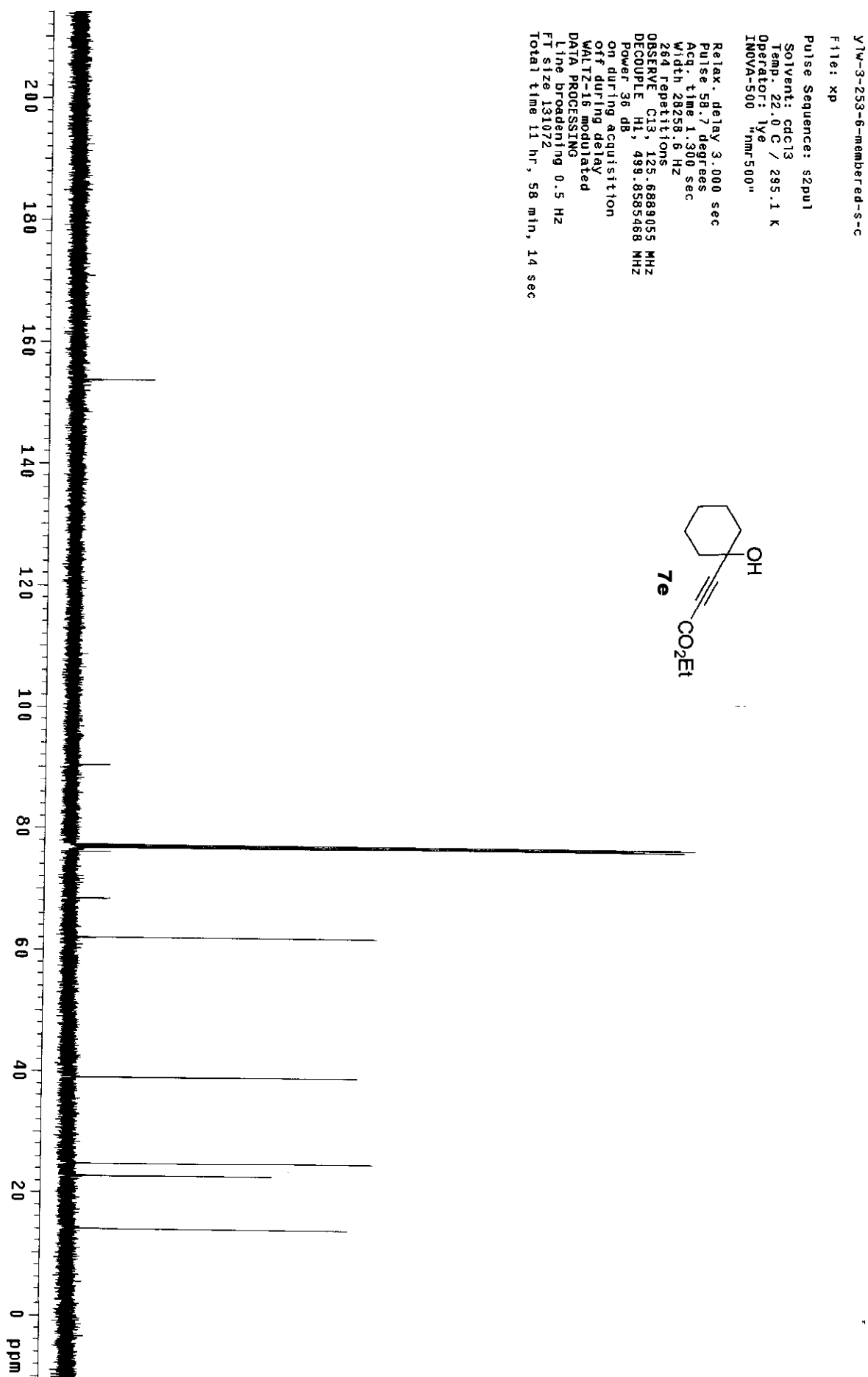
FT size 131072

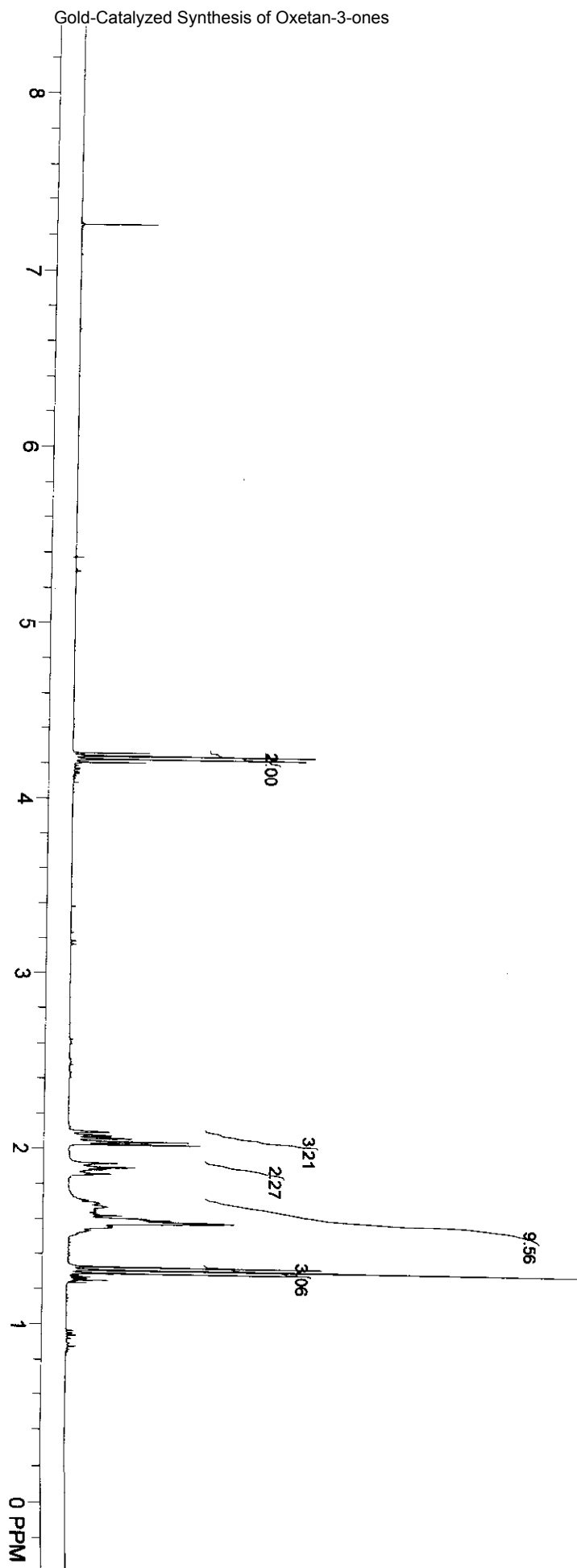
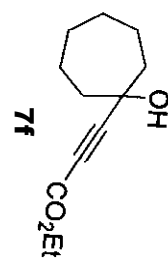
Total time 11 hr, 58 min, 14 sec











y1w-3-253-Ph-S-C

File: xp

Pulse Sequence: szpul

Solvent: cdcl3

Temp: 22.0 C / 295.1 K

Operator: lye

INOVA-500 "nmr 500"

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 28258.6 Hz

44 repetitions

OBSERVE C13, 125.6889032 MHz

DECOUPLE H1, 499.8585468 MHz

Power 36 dB

on during acquisition

off during delay

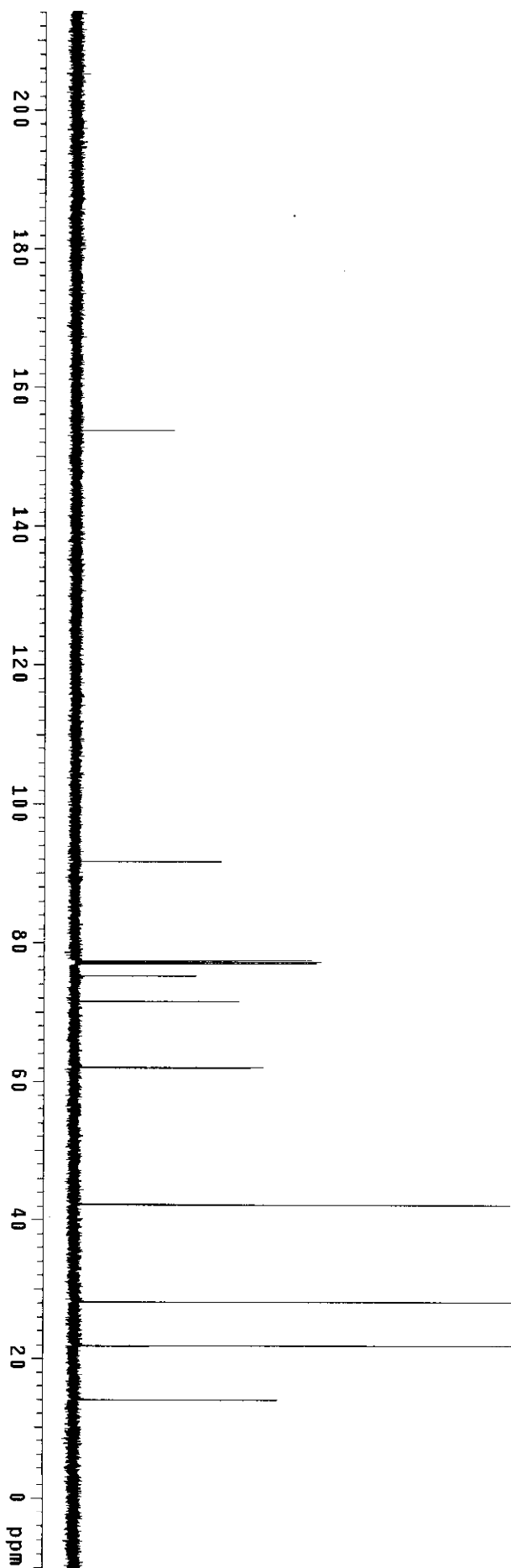
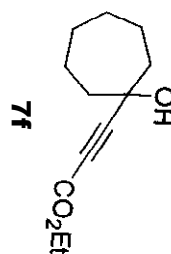
WALTZ-16 modulated

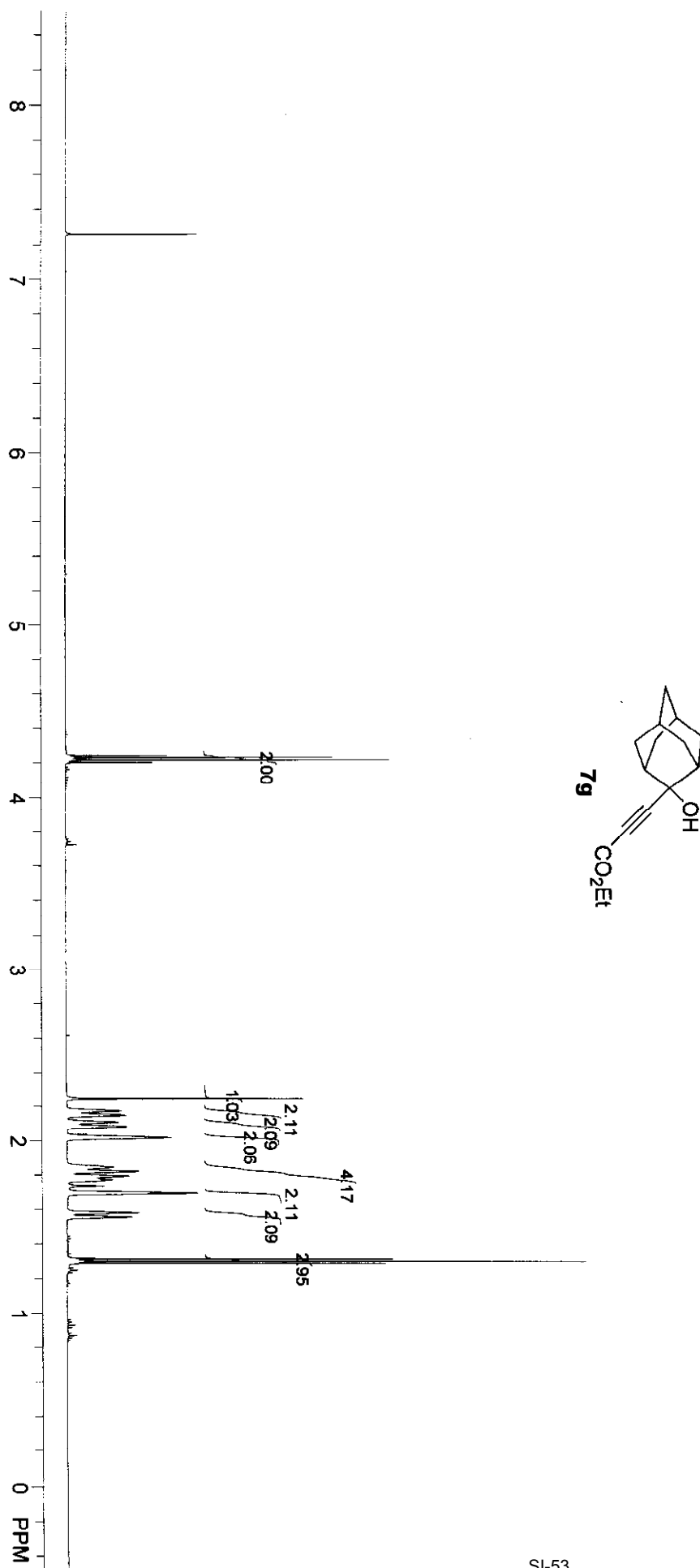
DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 11 hr, 58 min, 14 sec





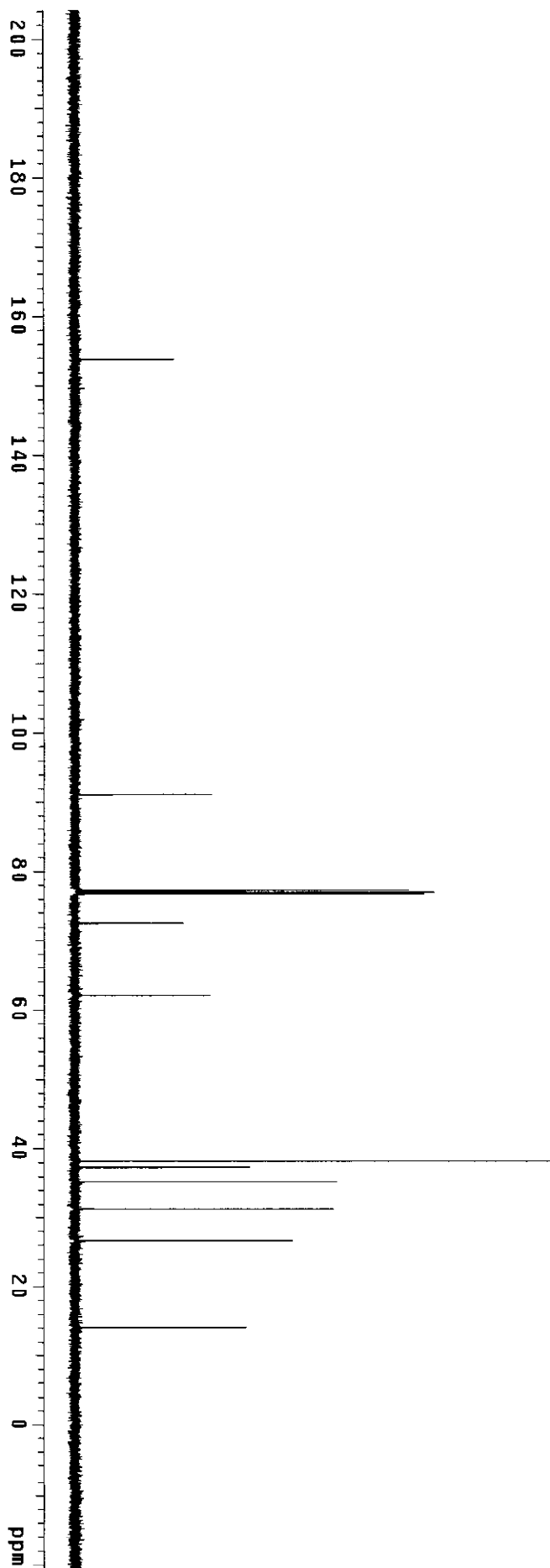
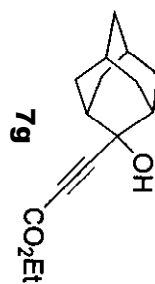
Y1w-3-285-2-s-c

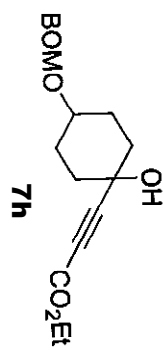
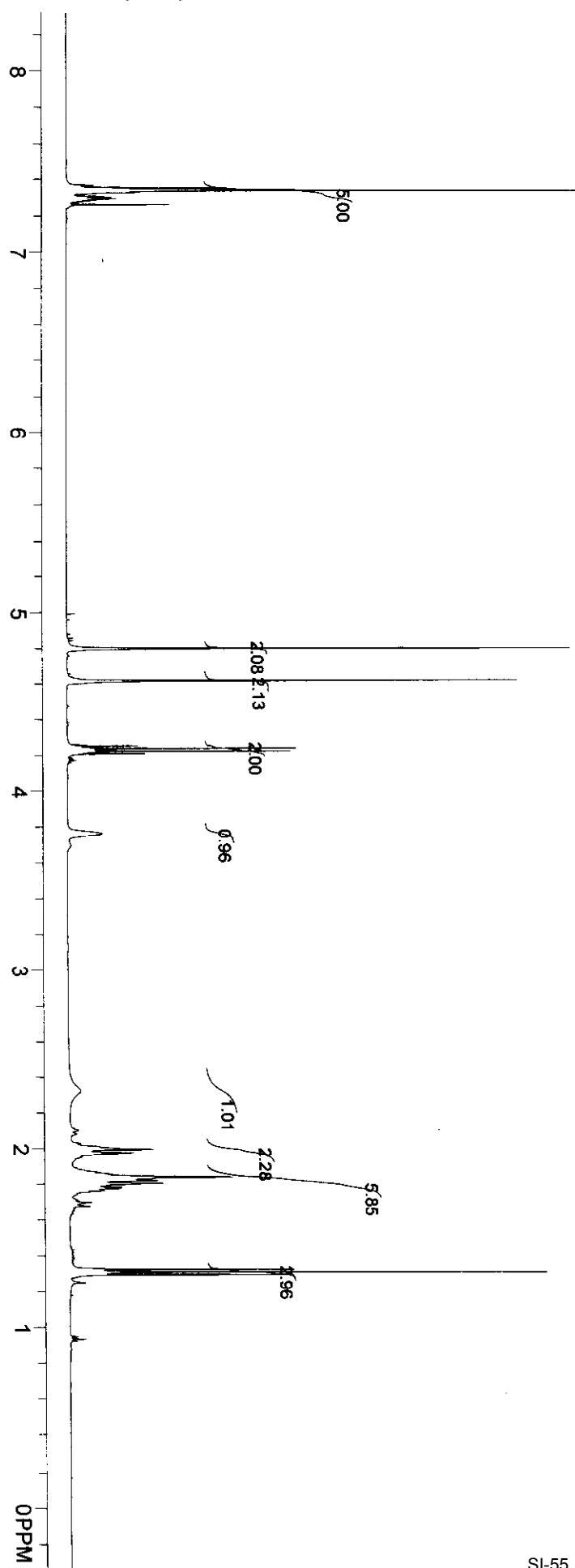
Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmrsys/data  
Sample directory:

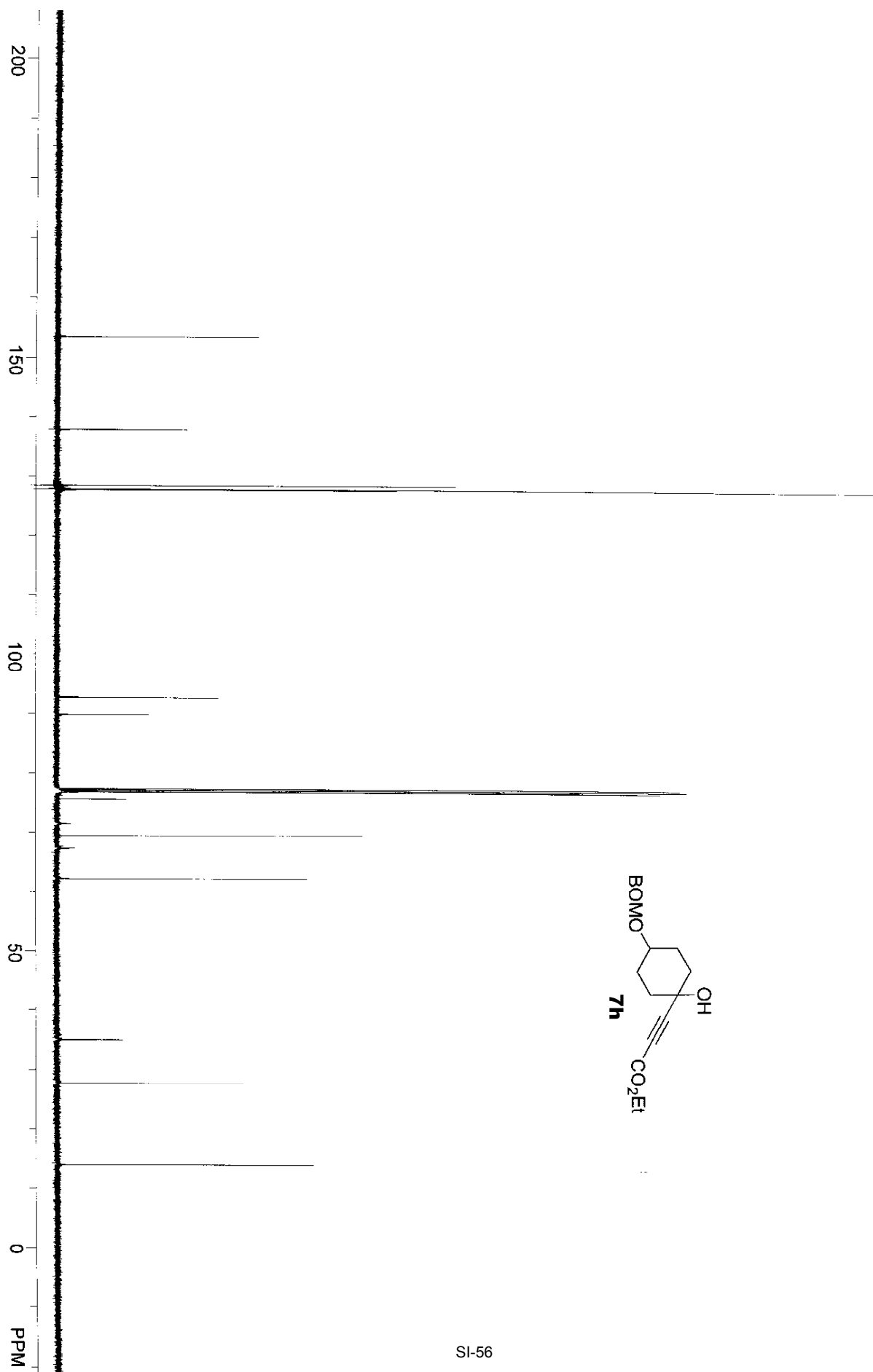
File: CARBON

Pulse Sequence: s2pu1  
Solvent: cdcl3  
Temp. 22.0 C / 295.1 K  
Operator: lye

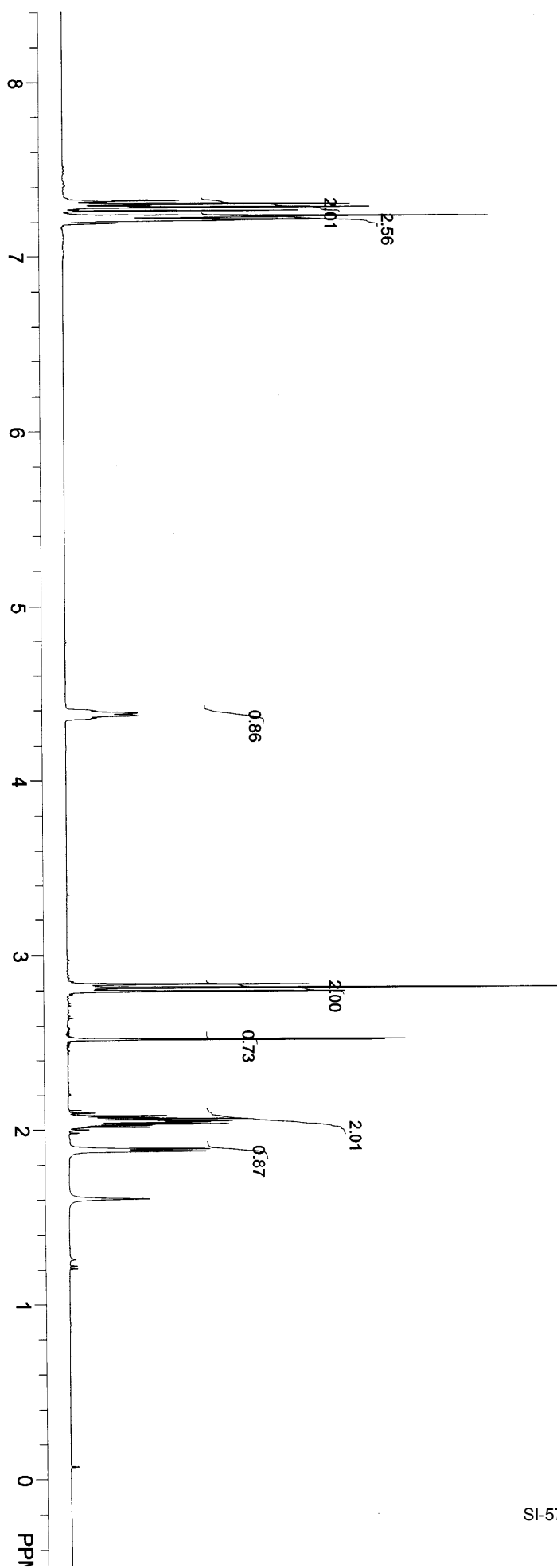
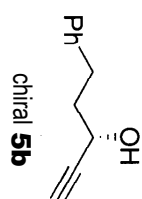
Relax. delay 3.000 sec  
Pulse 58.7 degrees  
Acq. time 1.300 sec  
Width 40000.0 Hz  
106 repetitions  
OBSERVE C13, 125.6889847 MHz  
DECUPLE H1, 499.858575 MHz  
Power 36 db  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 0.5 Hz  
FI size 131072  
Total time 0 min



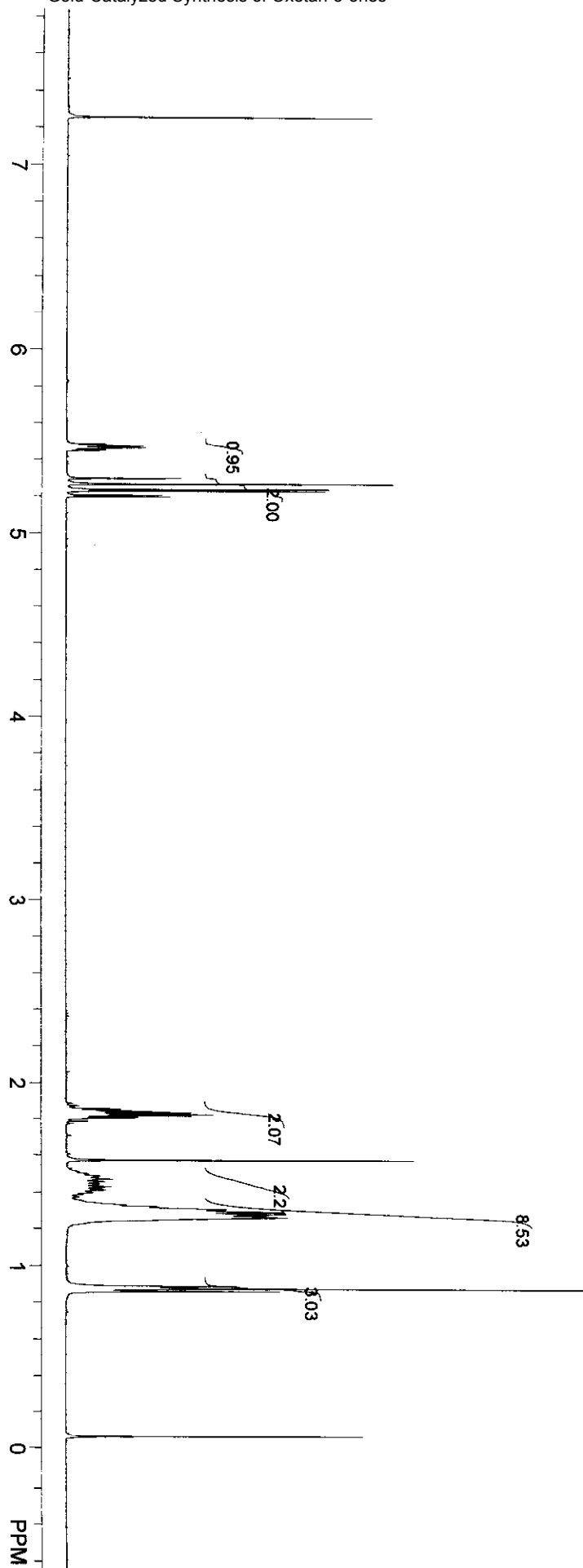
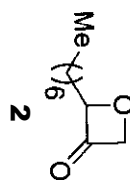








## Gold-Catalyzed Synthesis of Oxetan-3-ones



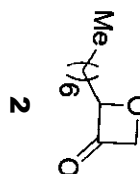
y1w-3-259-c1

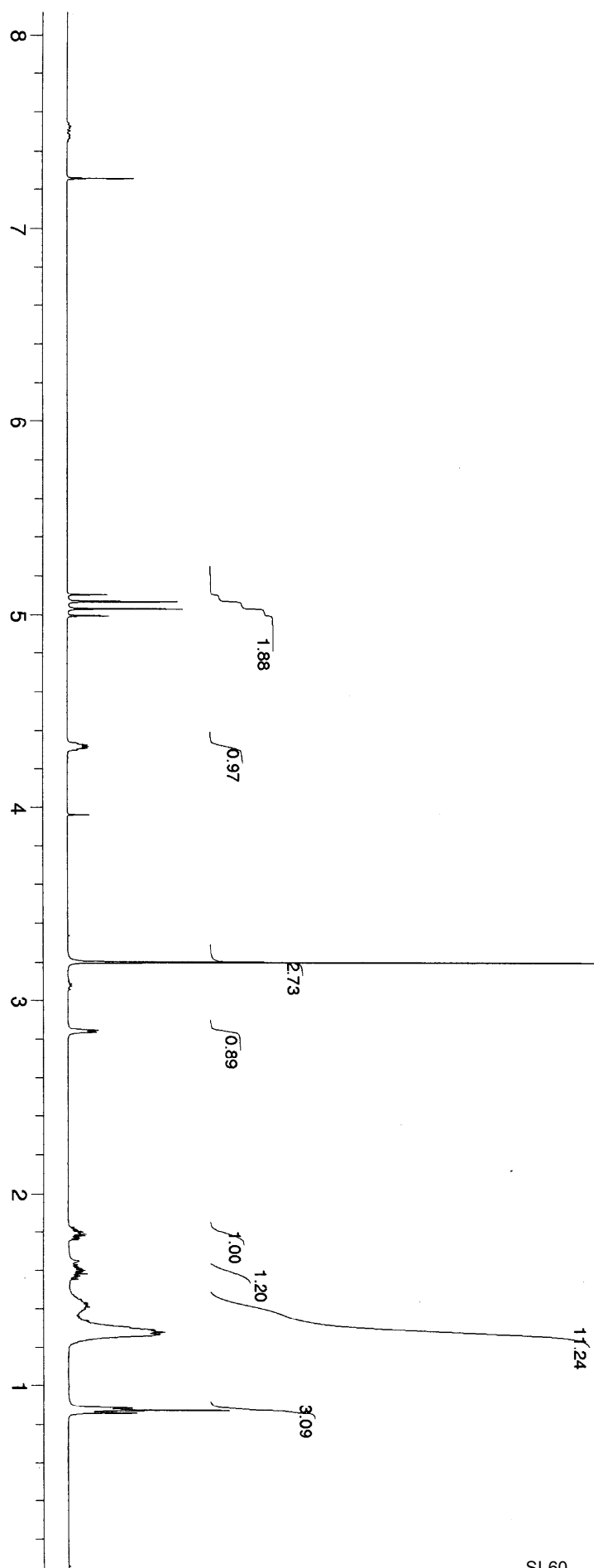
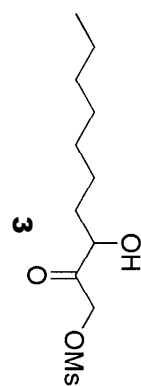
Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmrSYS/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pul  
Solvent: cdcl3  
Temp: 22.0 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec  
Pulse 58.7 degrees  
Acq. time 1.300 sec  
Width 40000.0 Hz  
232 repetitions  
OBSERVE C13, 125.6889829 MHz  
DECUPLE H1, 499.8588575 MHz  
Power 36 db  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 0.5 Hz  
F1 size 131072  
Total time 0 min





v1w-4-21-c

File: xp

Pulse Sequence: s2pu1

Solvent: cdcl3

Temp: 22.0 C / 295.1 K

Operator: lye

INNOVA-500

"nmr500"

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 28258.6 Hz

202 repetitions

OBSERVE C13, 125.6889060 MHz

DECOUPLE H1, 499.8585468 MHz

Power 36 db

on during acquisition

off during delay

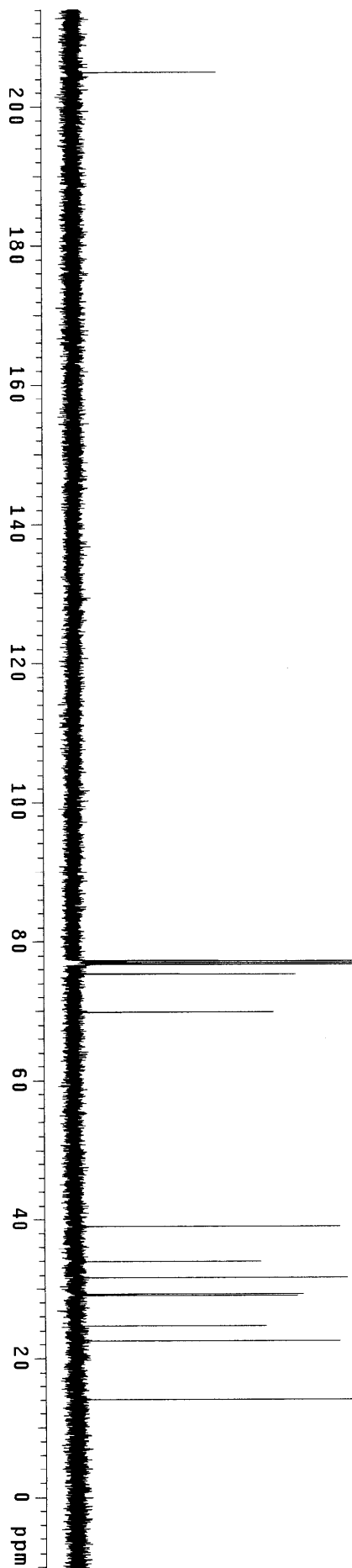
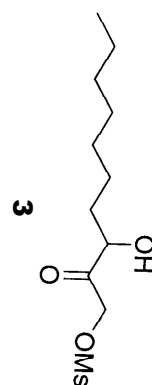
WALTZ-16 modulated

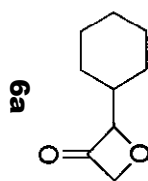
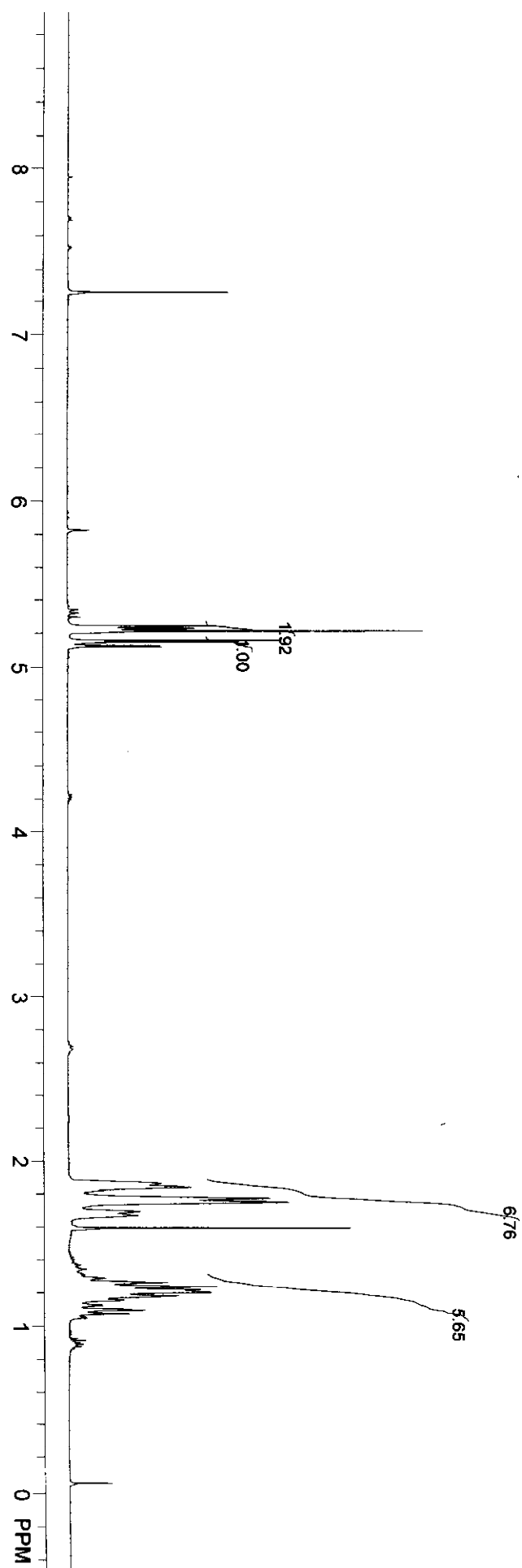
DATA PROCESSING

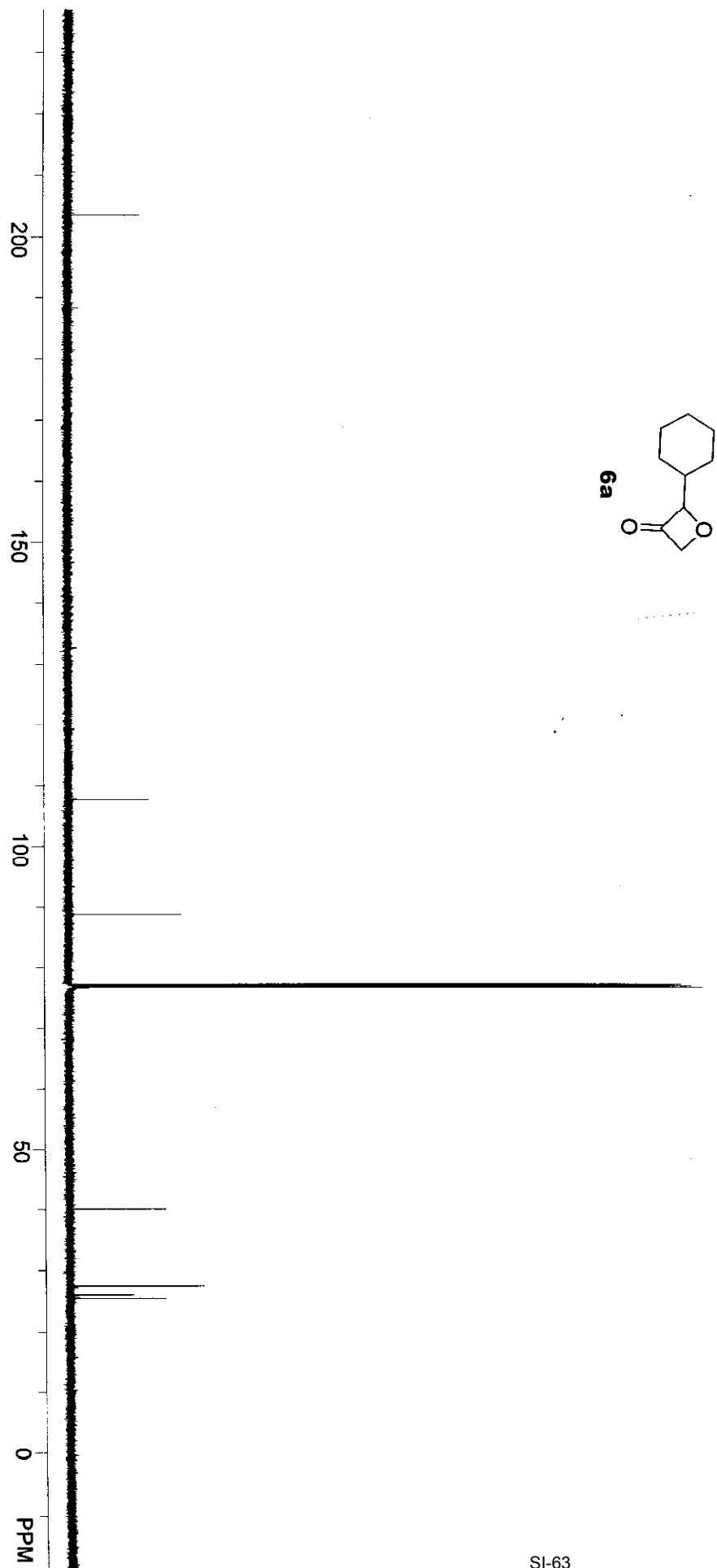
Line broadening 0.5 Hz

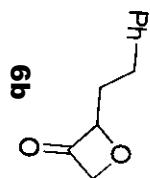
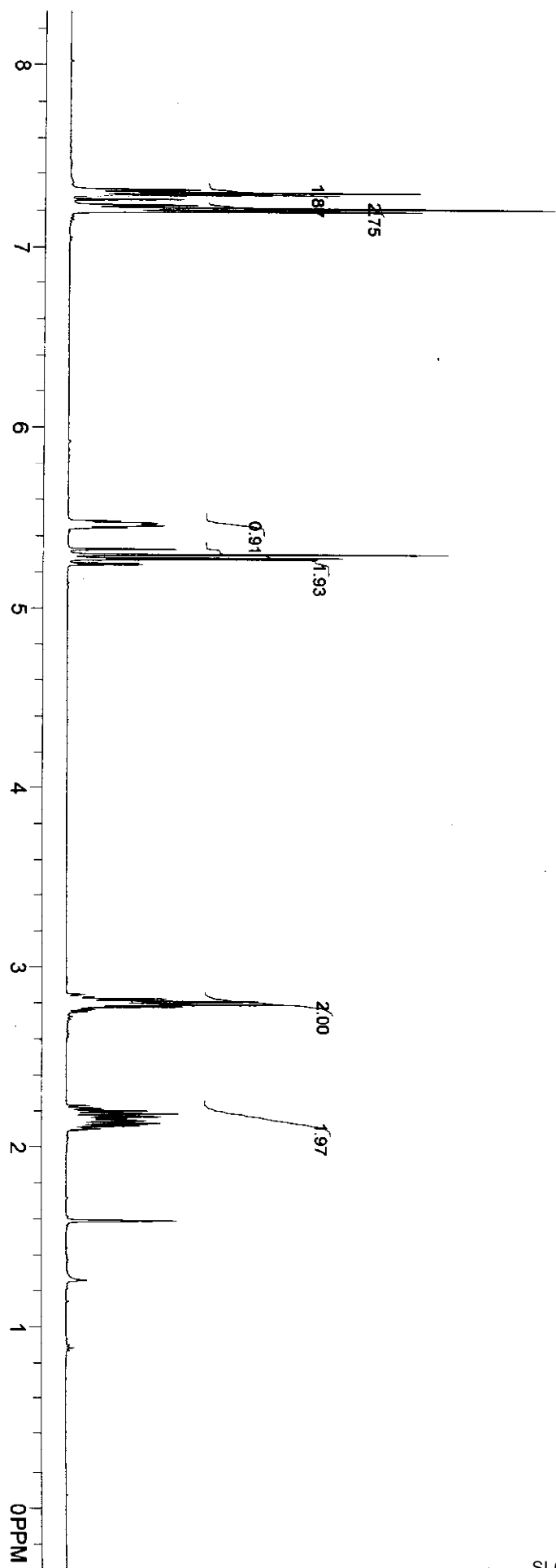
FT size 131072

Total time 119 hr, 42 min, 16 sec











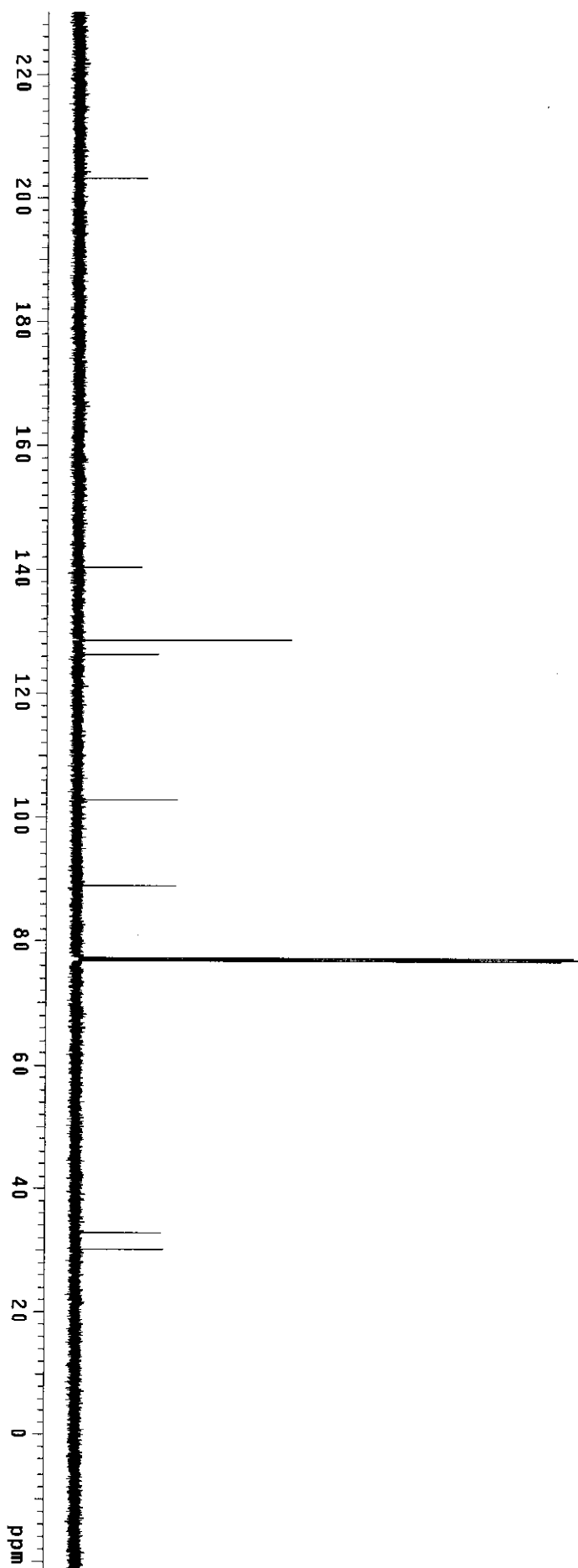
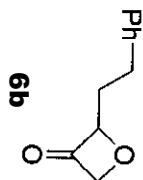
Ylw-3-261-c

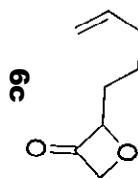
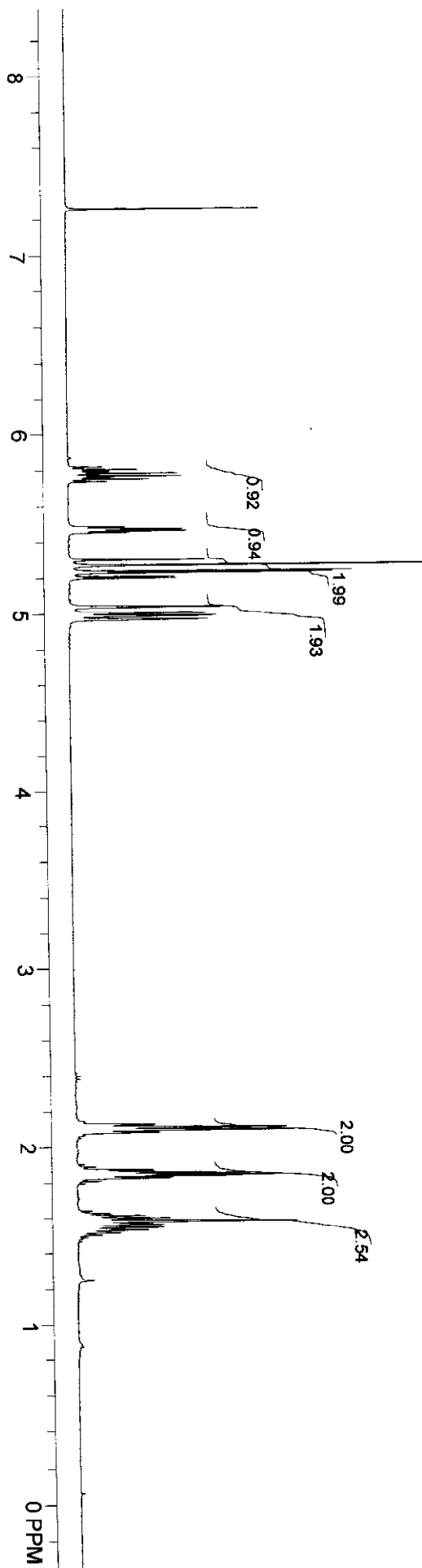
Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmrSYS/data  
Sample directory:

File: CARBON

Pulse Sequence: szpu1  
Solvent: cdc13  
Temp: 22.0 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec  
Pulse 58.7 degrees  
Acq. time 1.300 sec  
Width 40000.0 Hz  
196 repetitions  
OBSERVE C13, 125.6889847 MHz  
DECOUPLE H1, 499.8588575 MHz  
Power 35 dB  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 0.5 Hz  
FT size 131072  
Total time 0 min





y1w-3-267-c

Data Collected On:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmr/sys/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pul  
Solvent: cdcl3

Temp. 22.0 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec  
Pulse 58.7 degrees

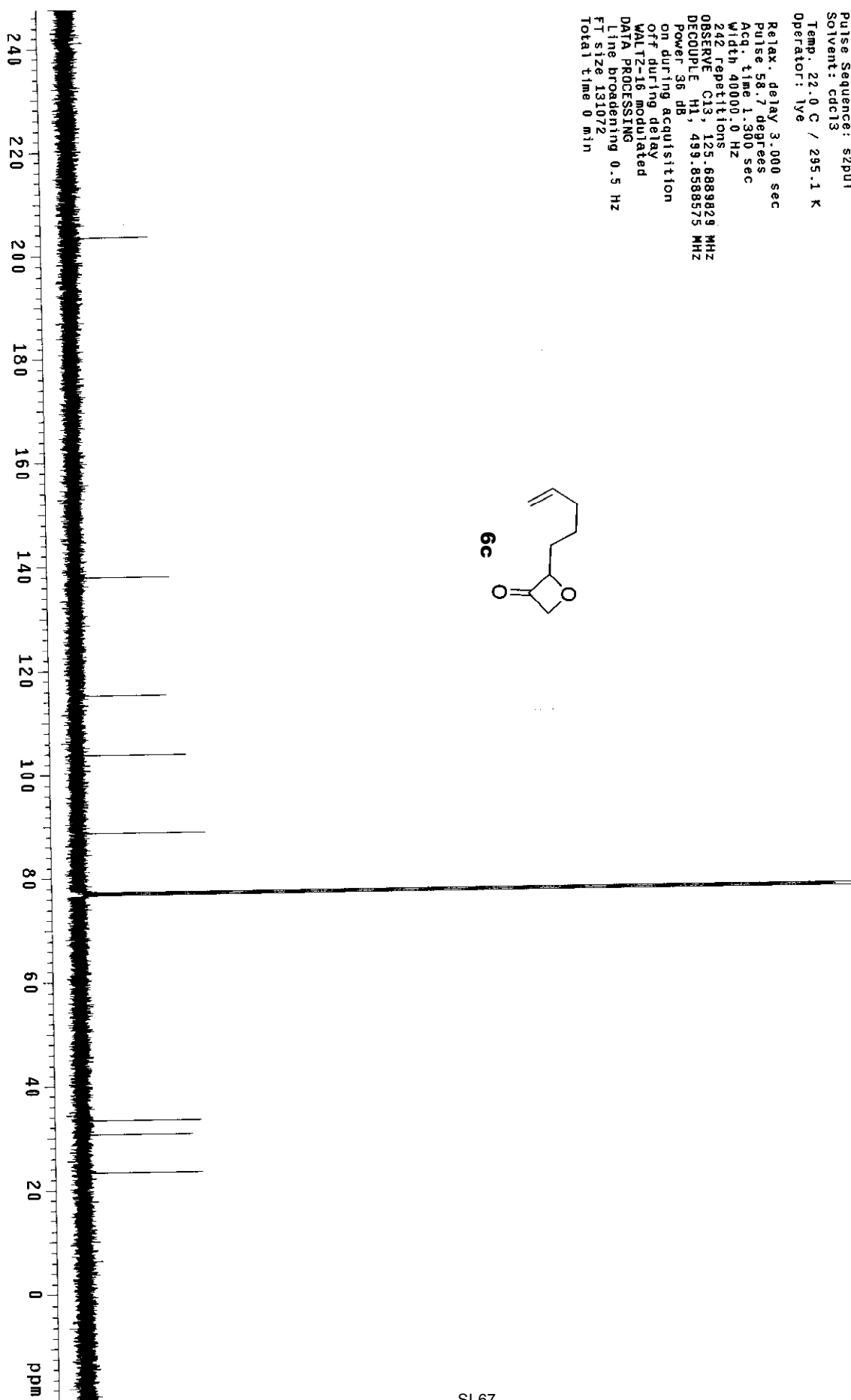
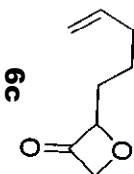
Acq. time 1.300 sec  
Width 40000.0 Hz

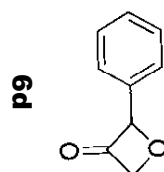
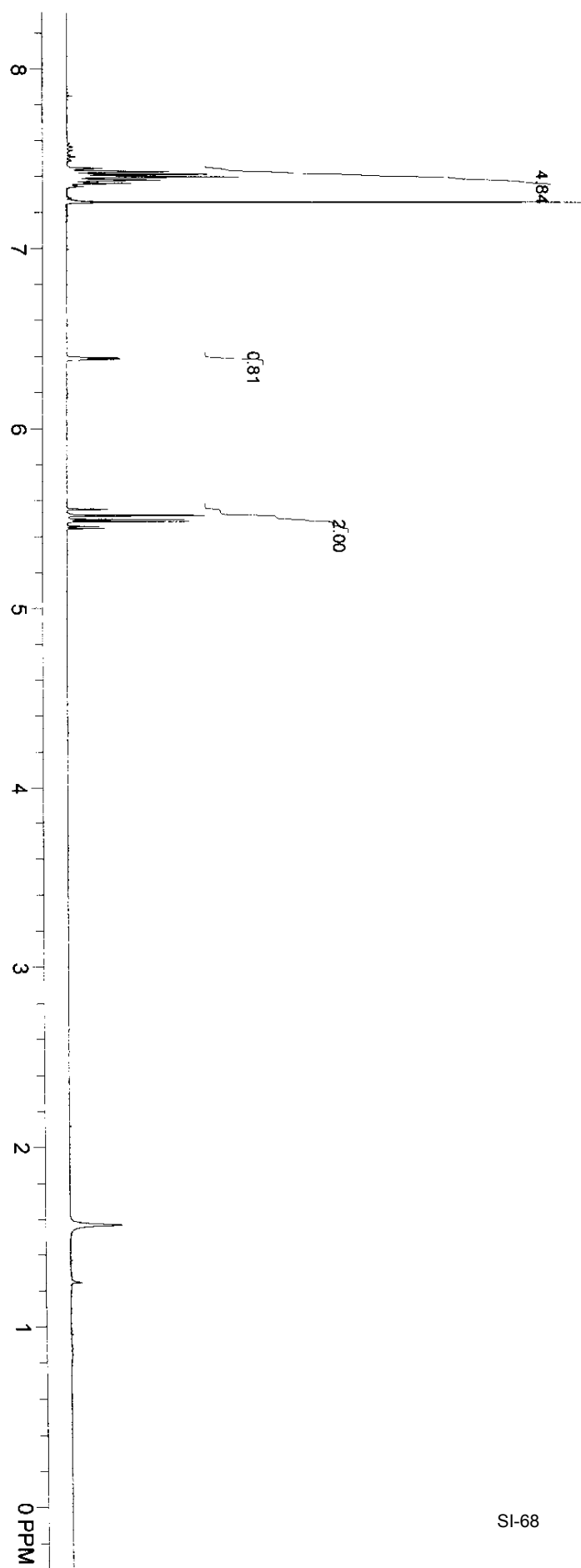
242 repetitions  
OBSERVE C13, 125.6889829 MHz  
DECUPLE H1, 499.8588575 MHz

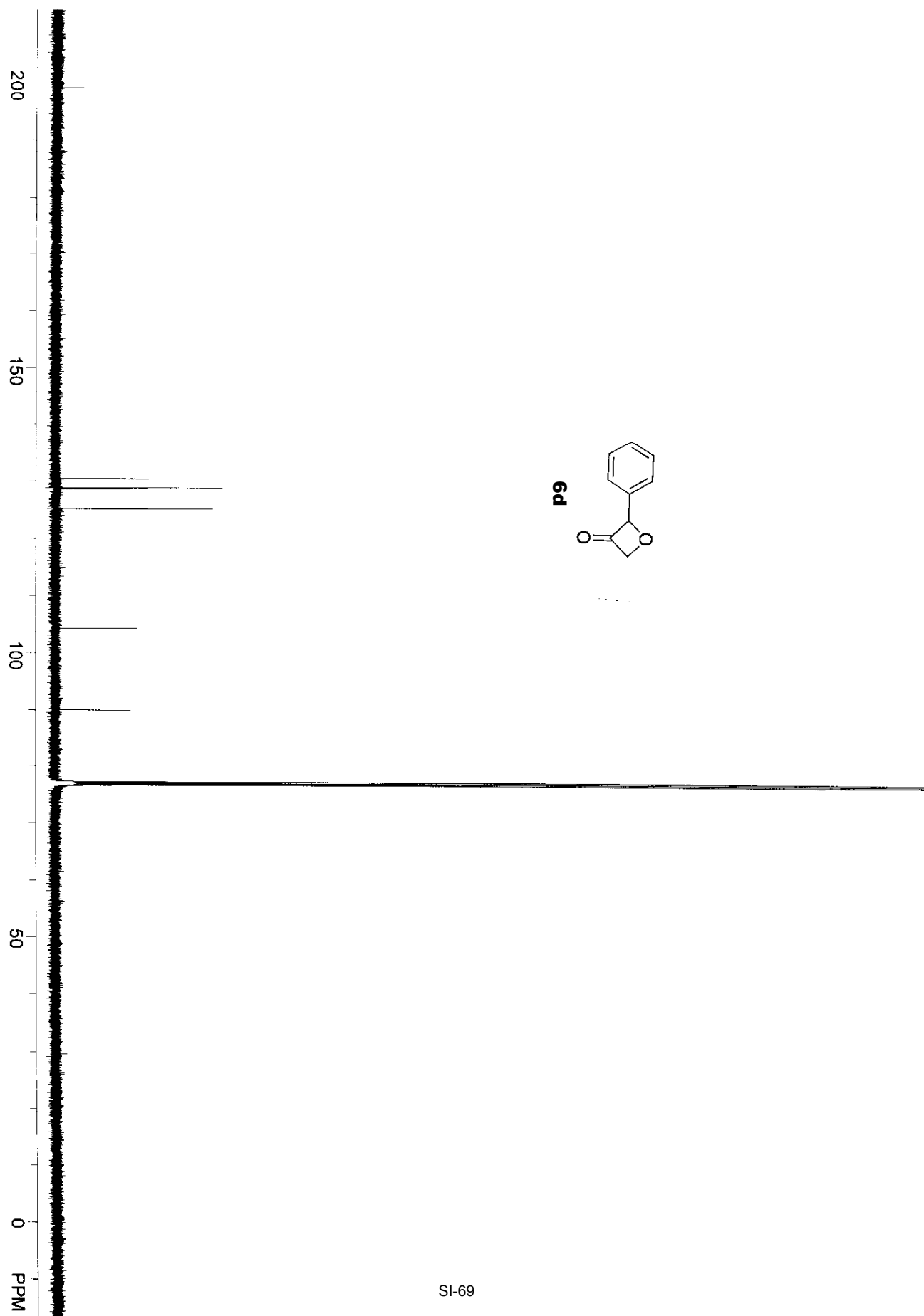
Power 36 db  
on during acquisition

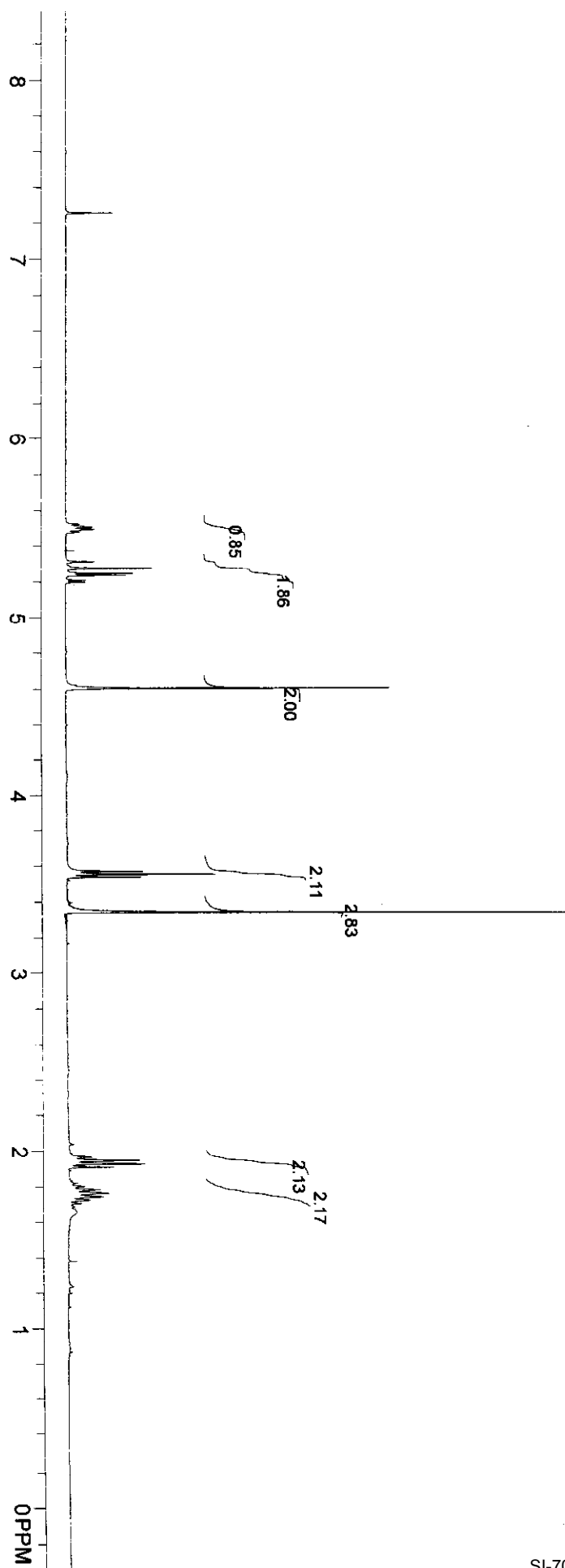
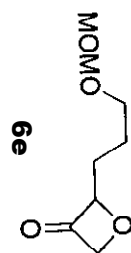
off during delay  
WALTZ-16 modulated

DATA PROCESSING  
Line broadening 0.5 Hz  
FI size 131072  
Total time 0 min









Y1W-3-285-C

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmr/sys/data  
Sample directory:

File: CARBON

Pulse Sequence: szpul  
Solvent: cdcl3

Temp. 22.0 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec  
Pulse 58.7 degrees  
Acq. time 1.300 sec  
Width 40000.0 Hz

144 repetitions

OBSERVE C13, 125.6889829 MHz  
DECOUPLE H1, 499.8588575 MHz

Power 36 dB

on during acquisition

off during delay

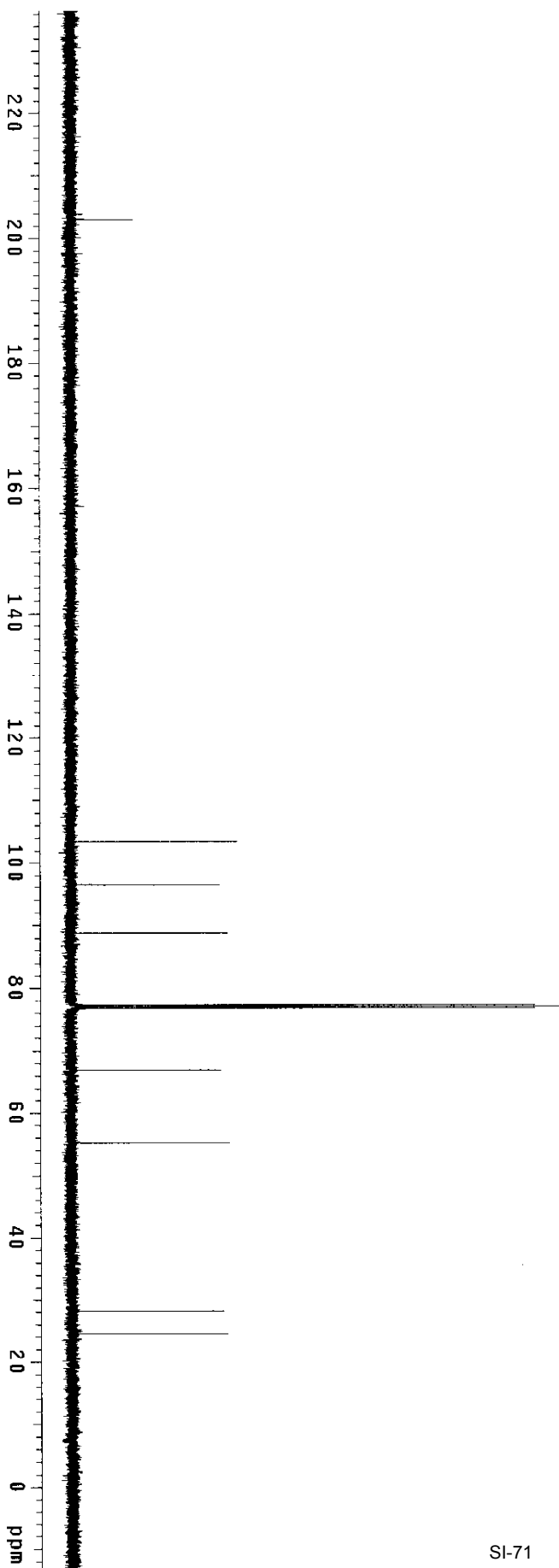
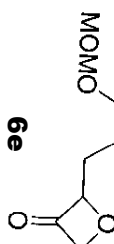
WALTZ-16 modulated

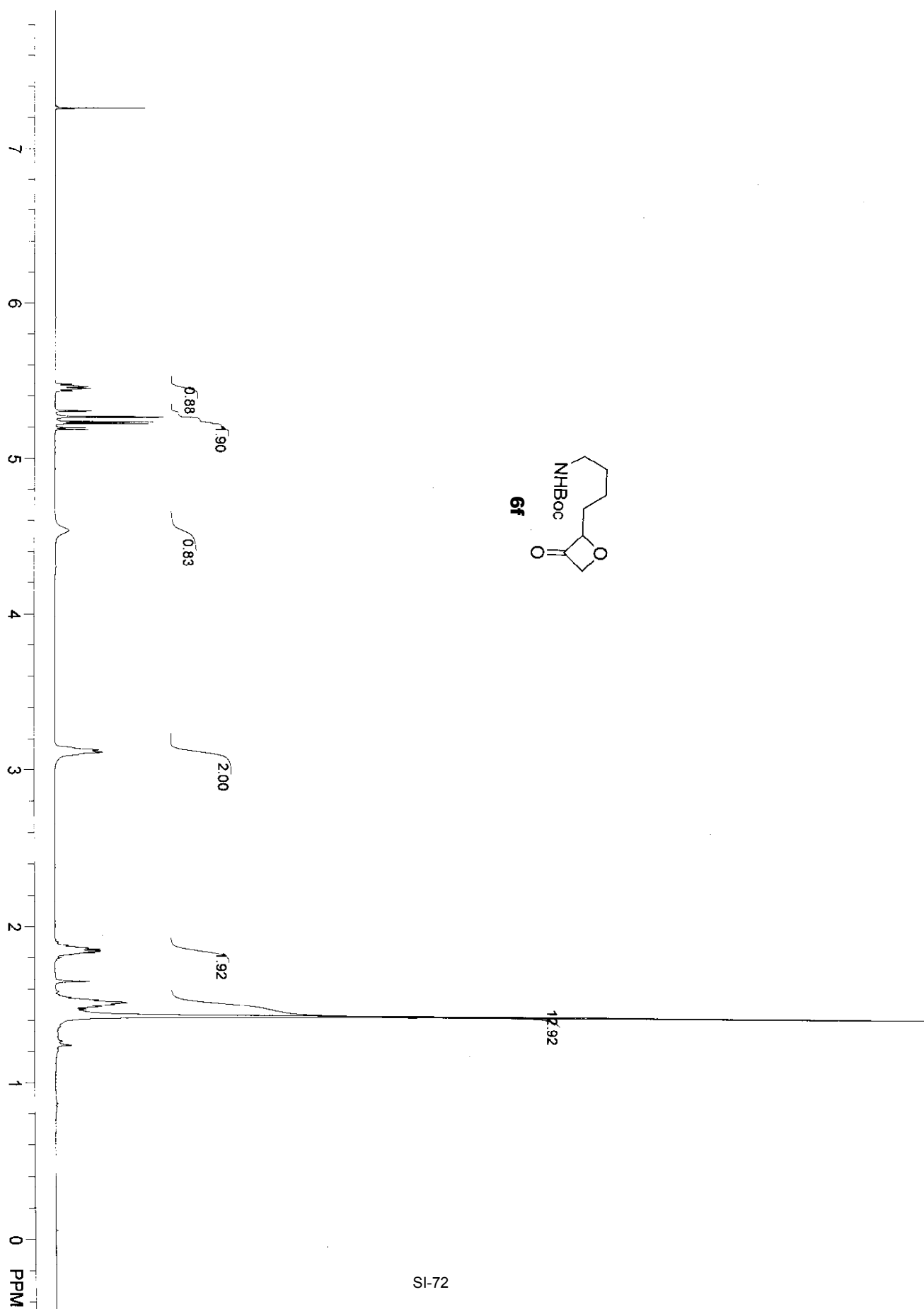
DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 0 min







y1w-3-296-c

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmr500/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pu1  
Solvent: cdcl3

Temp: 22.0 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec  
Pulse 58.7 degrees

Acq. time 1.300 sec  
Width 40000.0 Hz

528 repetitions

OBSERVE C13, 125.6889835 MHz  
DECOUPLE H1, 499.858575 MHz

Power 36 dB

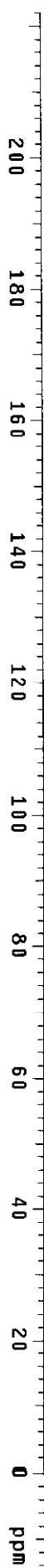
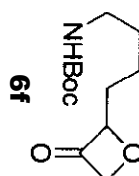
on during acquisition  
off during delay

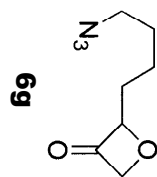
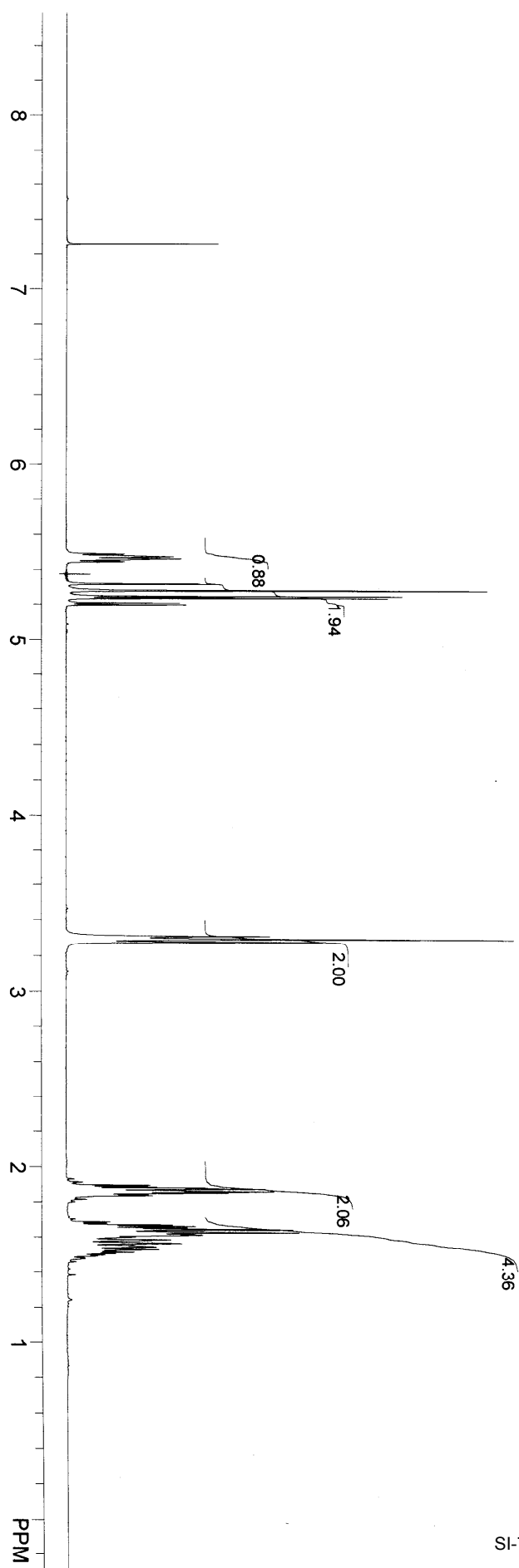
WALTZ-16 modulated

Line broadening 0.5 Hz

FT size 131072

Total time 0 min





y1w-3-303-N3-c

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmr/sys/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pu1

Solvent: cdcl3

Temp. 22.0 C / 295.1 K

Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 40000.0 Hz

52 repetitions

OBSERVE C13, 125.6889854 MHz

DECOUPLE H1, 499.858575 MHz

Power 36 dB

on during acquisition

off during delay

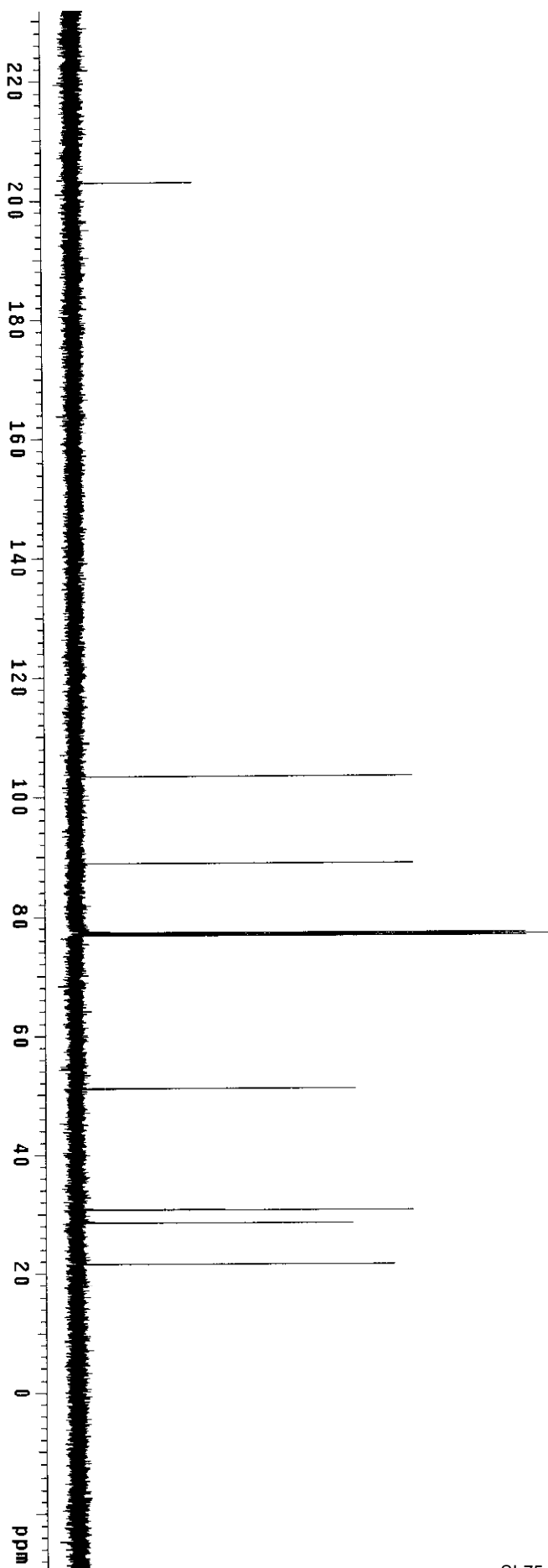
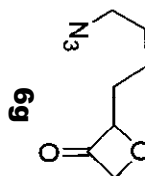
WALTZ-16 modulated

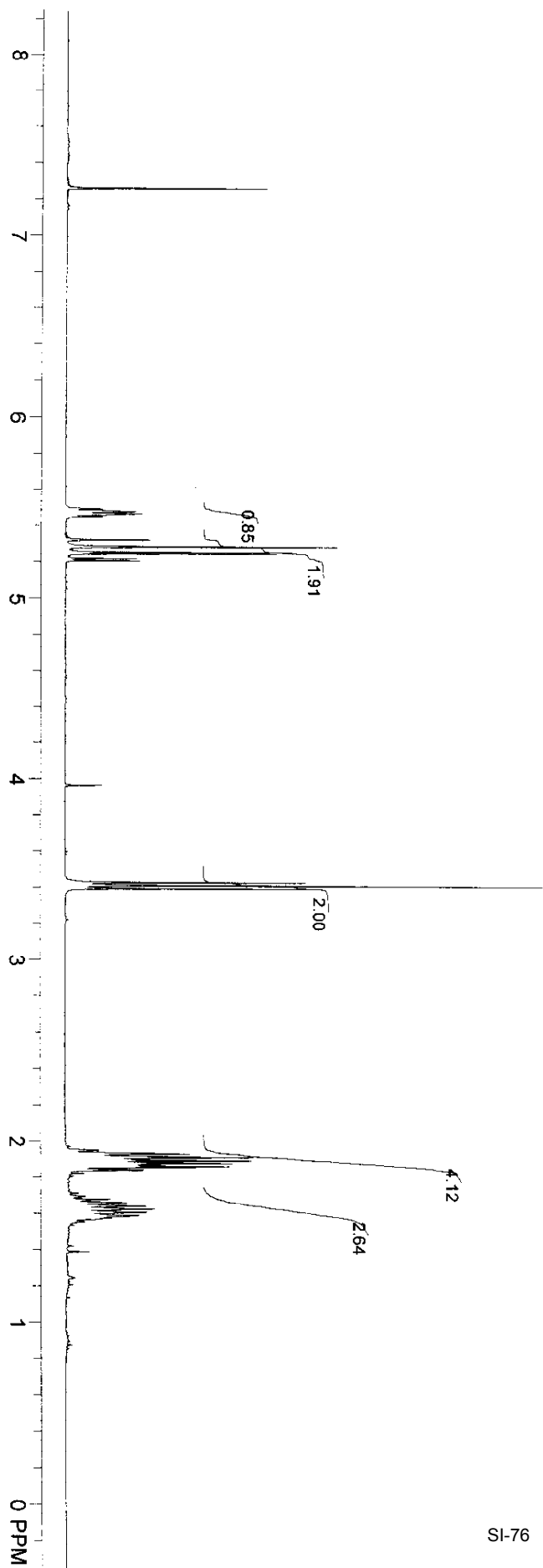
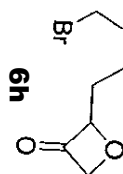
DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 0 min





y1w-3-302-Br-C

Data Collected on: nmr500-1prova500  
Archive directory: /export/home/vnmr1/vnmrsys/data  
Sample directory:

File: CARBON

Pulse Sequence: szpul  
Solvent: cdcl3

Temp. 22.0 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec  
Pulse 58.7 degrees

Acq. time 1.300 sec  
Width 40000.0 Hz

458 Repetitions

OBSERVE C13, 125.6889835 MHz  
DECUPLE H1, 499.858575 MHz

Power 36 dB

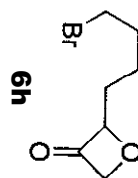
on during acquisition  
off during delay

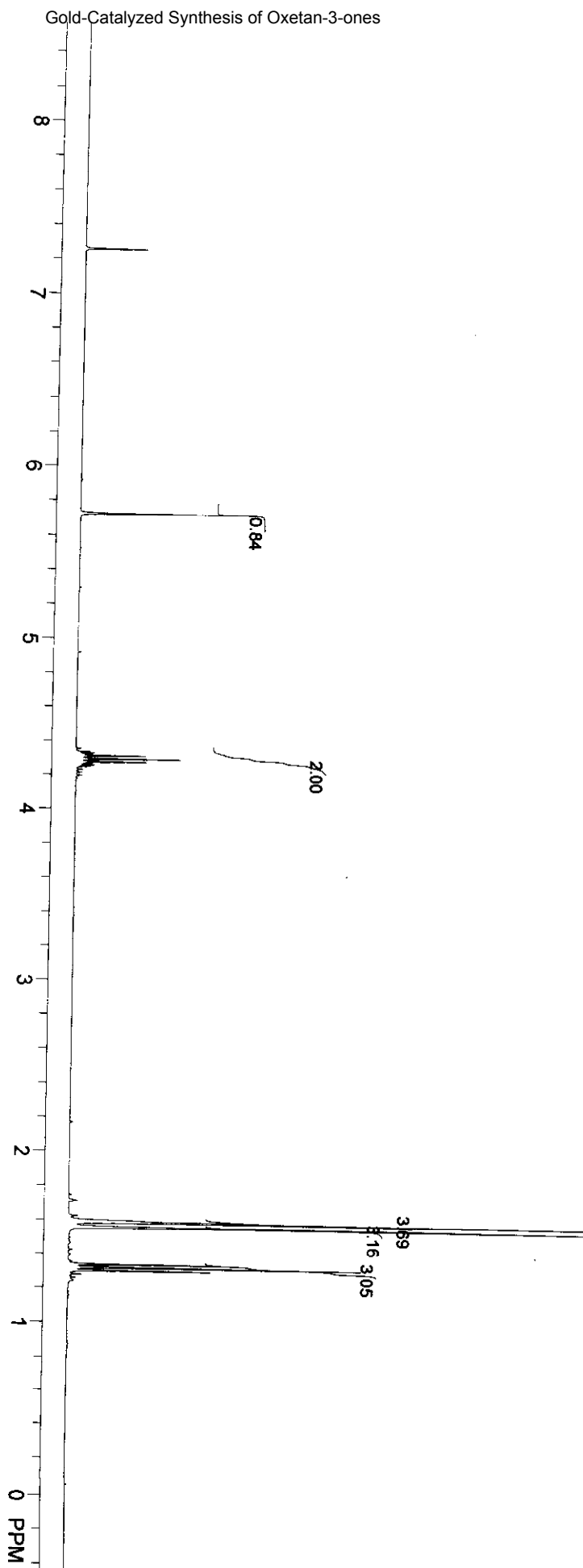
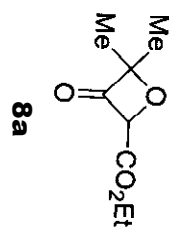
WALTZ-16 modulated

DATA PROCESSING  
Line broadening 0.5 Hz

F1 size 131072

Total time 0 min





y1w-3-269-c

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmr.sys/data  
Sample directory:

File: CARBON

Pulse Sequence: szpu1  
Solvent: cdcl3

Temp. 22.0 C / 295.1 K

Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 40000.0 Hz

290 repetitions

OBSERVE C13, 125.6889829 MHz

DECOUPLE H1, 499.8588575 MHz

Power 36 dB

on during acquisition

off during delay

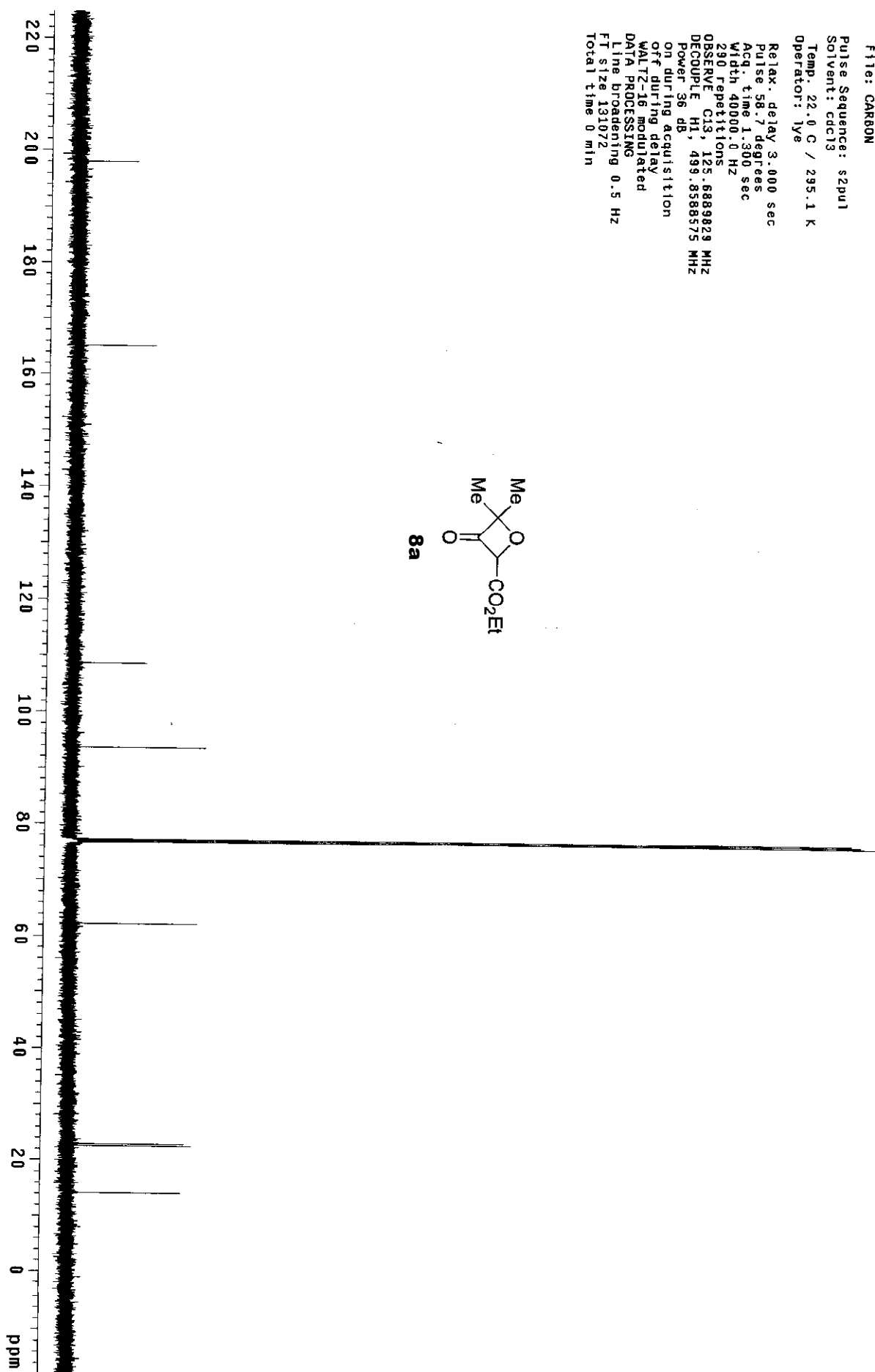
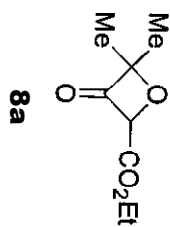
WALTZ-16 modulated

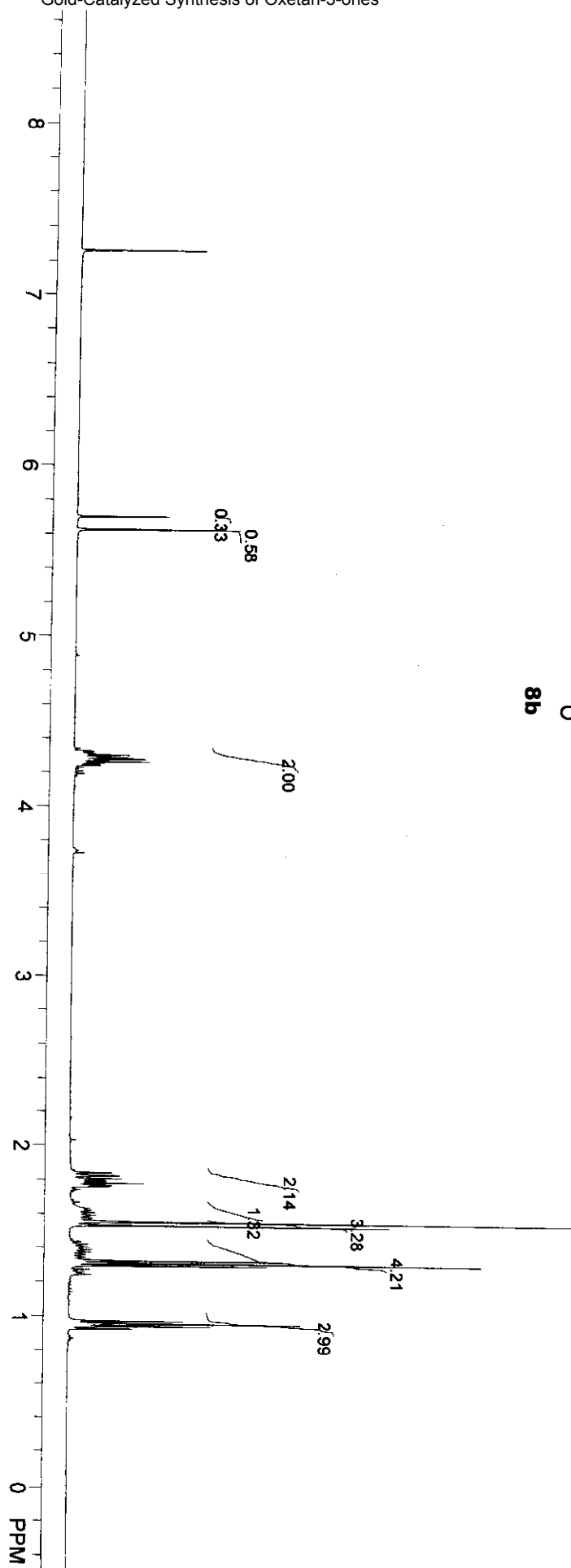
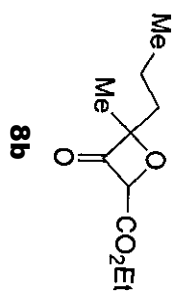
DATA PROCESSING

Line broadening 0.5 Hz

F1 size 131072

Total time 0 min







Y1w-3-260-c

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmr/sys/data  
Sample directory:

File: CARBON

Pulse Sequence: szpul

Solvent: cdcl3

Temp. 22.0 C / 295.1 K

Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 40000.0 Hz

378 repetitions

OBSERVE C13, 125.6889829 MHz

DECUPLE H1, 499.8558575 MHz

Power 36 dB

on during acquisition

off during delay

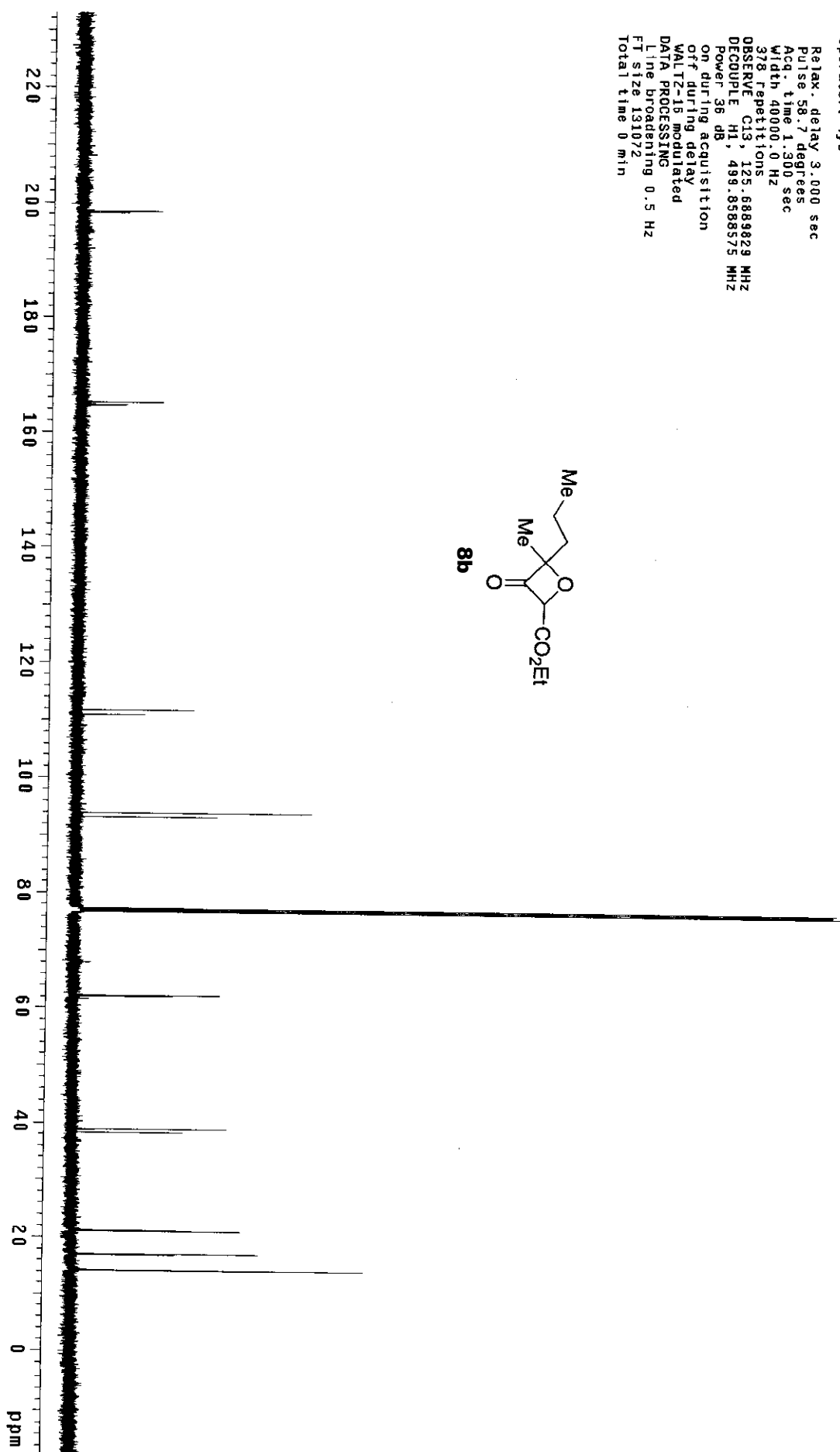
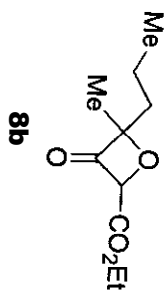
WALTZ-16 modulated

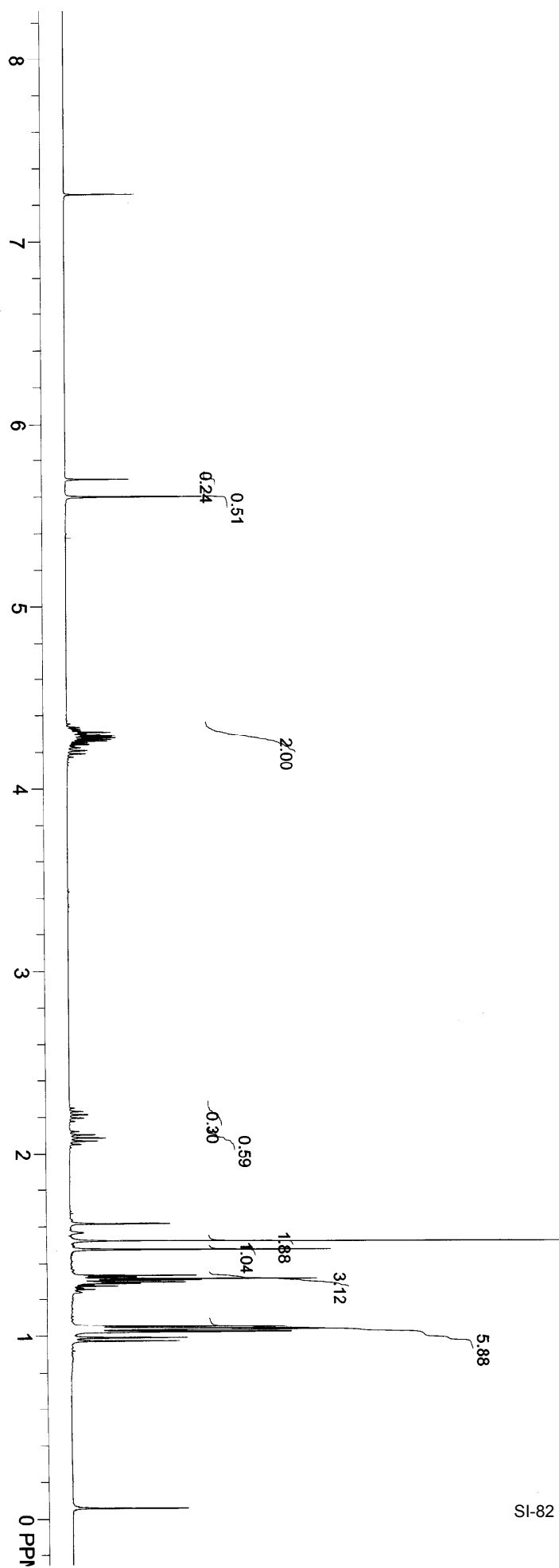
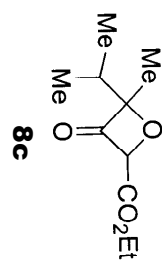
DATA PROCESSING

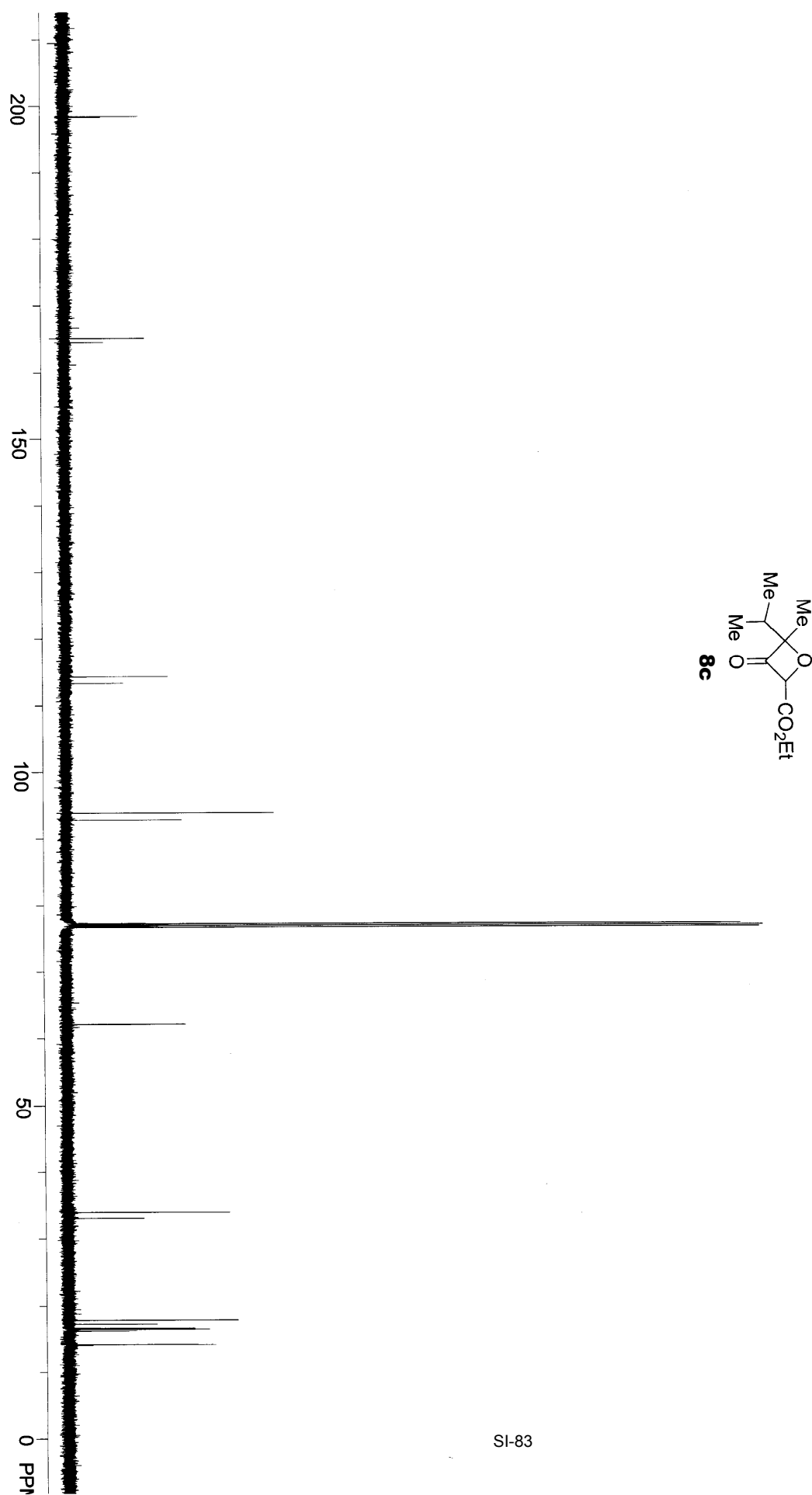
Line broadening 0.5 Hz

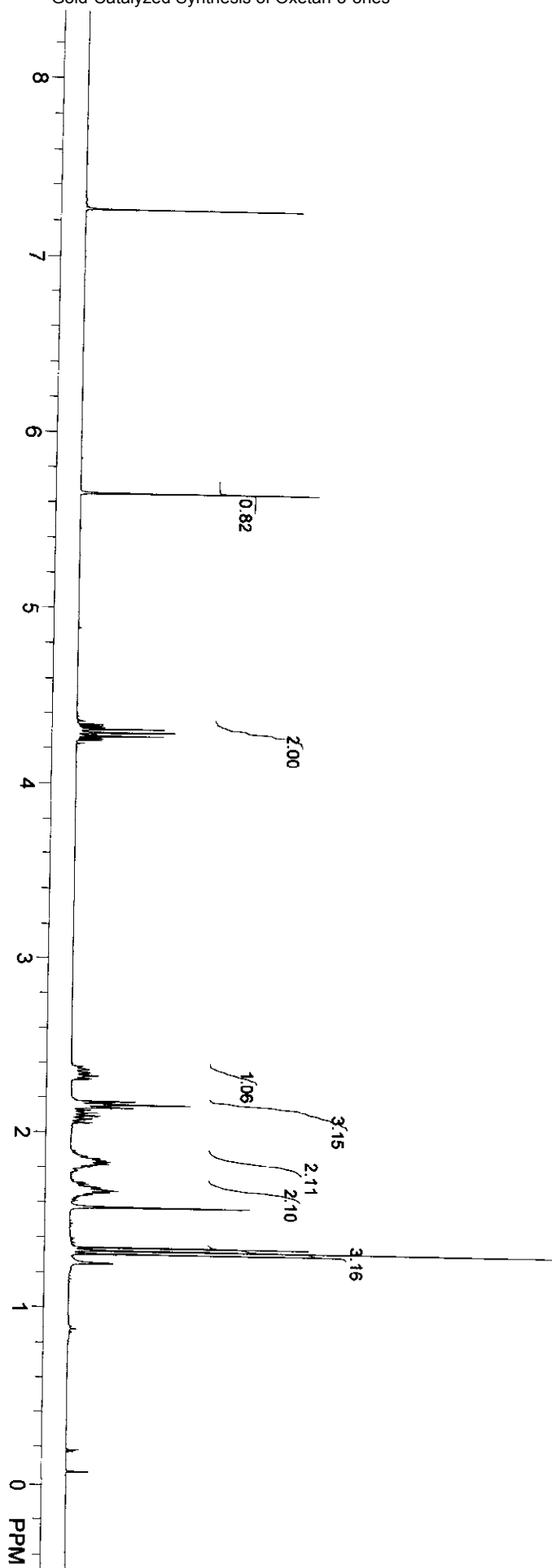
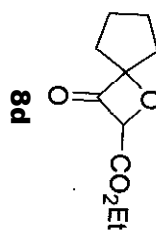
F1 size 181072

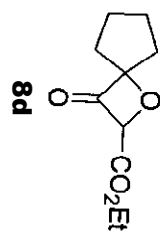
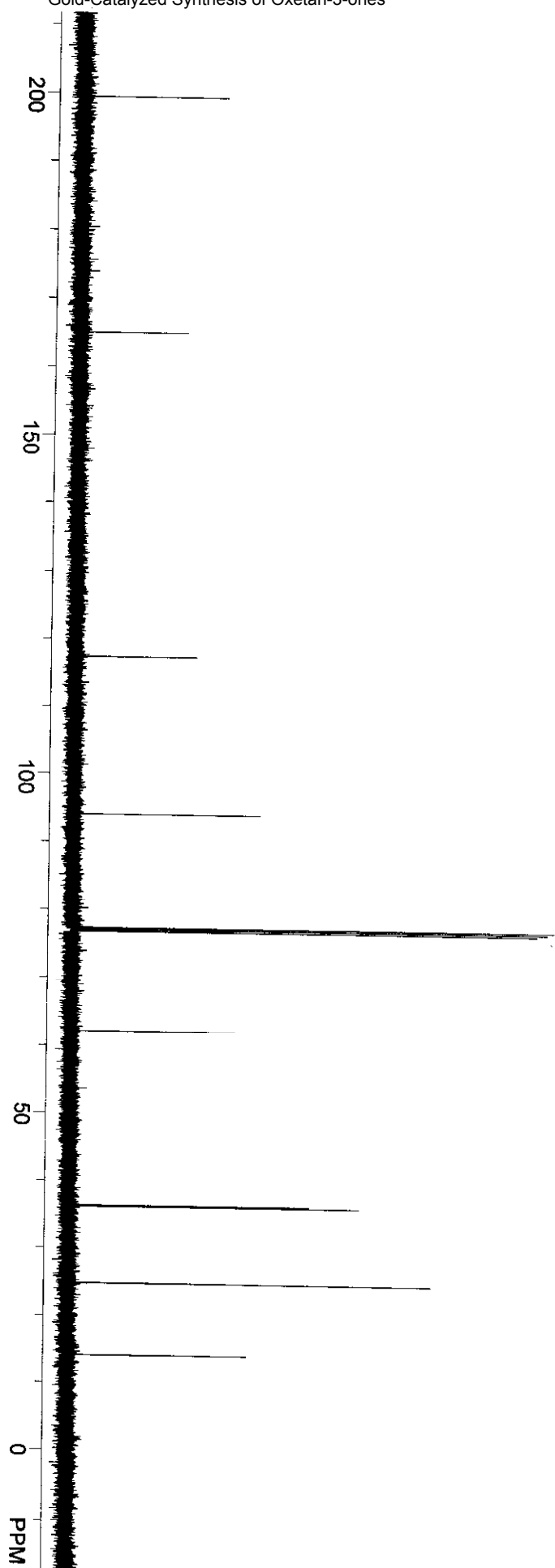
Total time 0 min

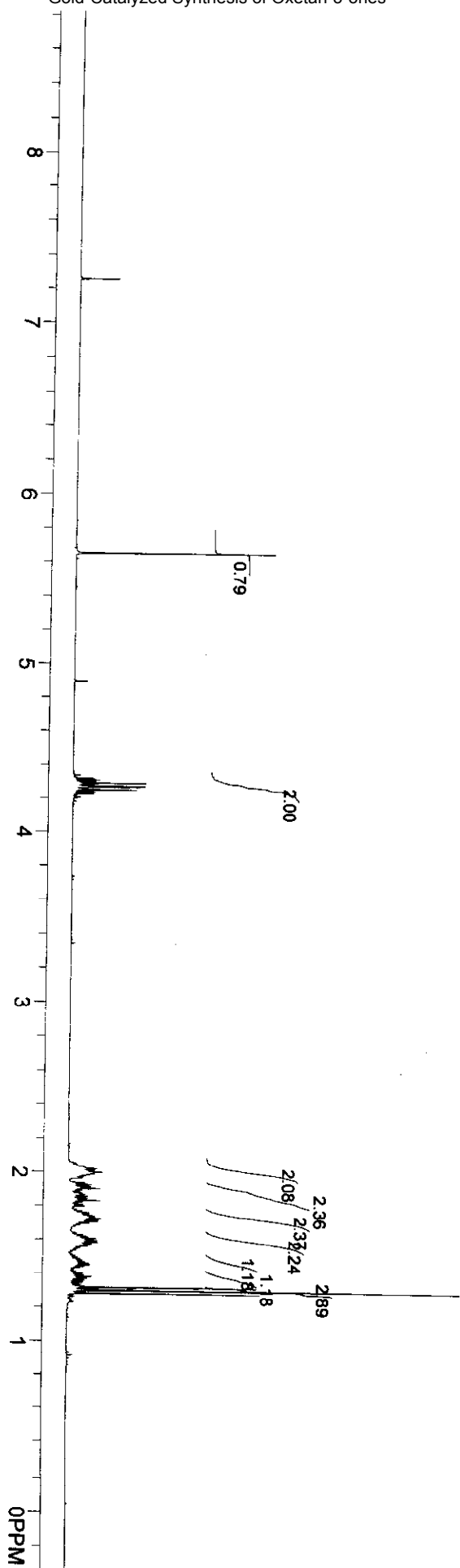
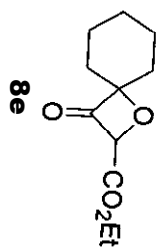












Ylw-3-274-c

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmr/sys/data  
Sample directory:

File: CARBON

Pulse Sequence: zgpg30

Solvent: cdcl3

Temp. 22.0 C / 295.1 K

Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 40000.0 Hz

160 repetitions

OBSERVE C13, 125.6889847 MHz

DECUPLE H1, 499.856575 MHz

Power 36 dB

on during acquisition

off during delay

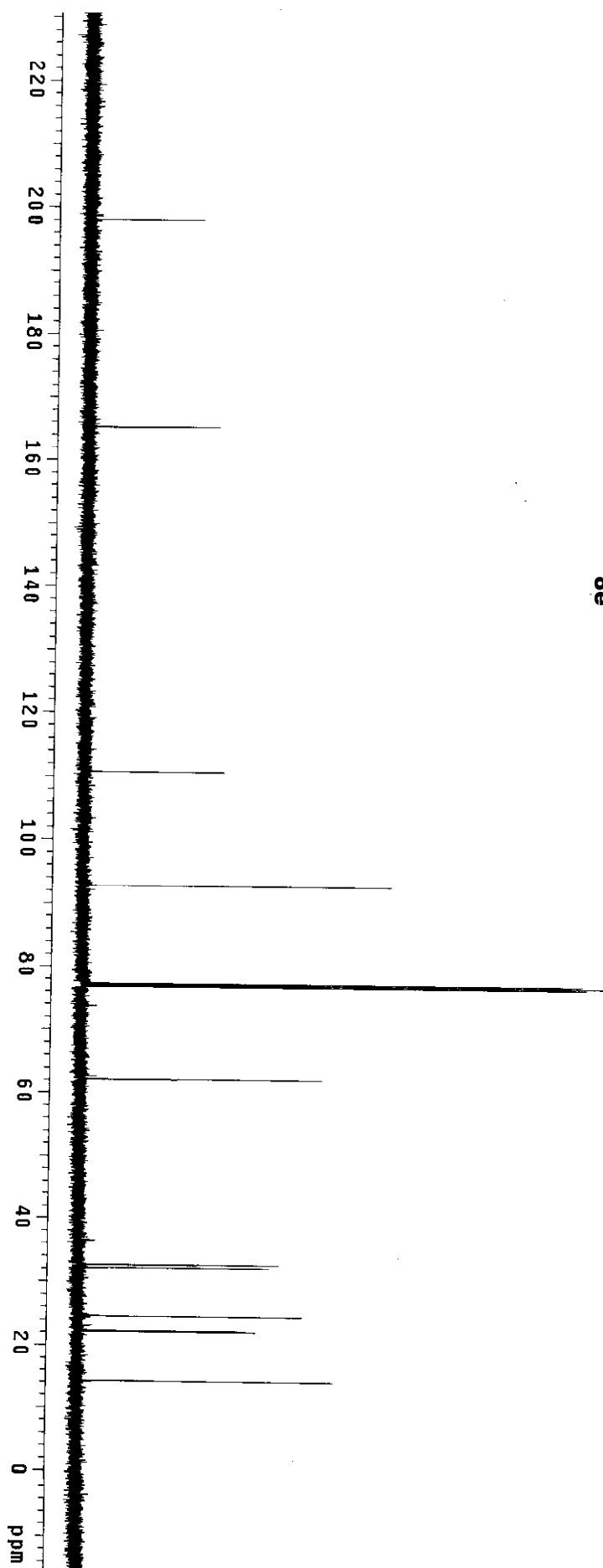
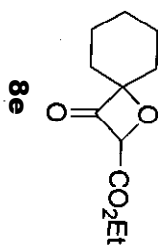
WALTZ-16 modulated

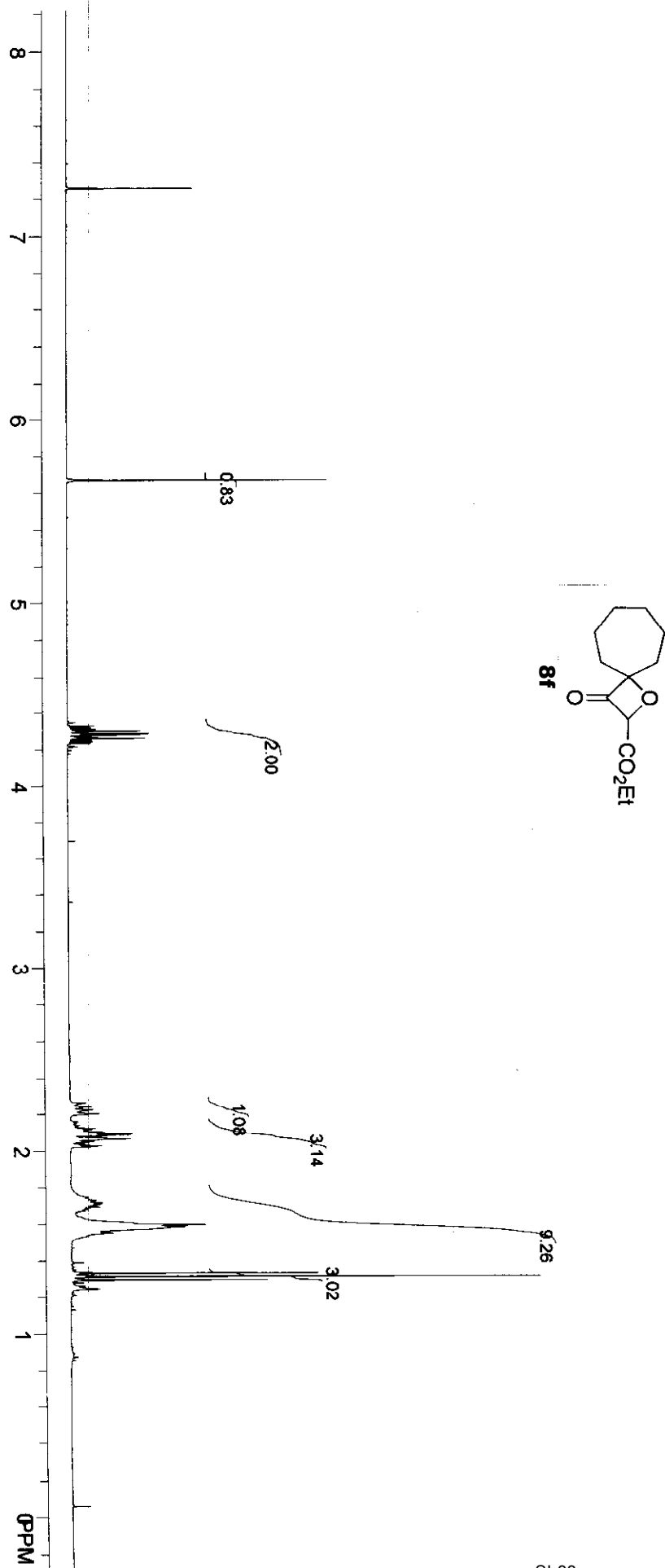
DATA PROCESSING

Line broadening 0.5 Hz

F1 size 131072

Total time 0 min







y1w-3-254-c

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmrsys/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pul  
Solvent: cdcl3

Temp. 22.0 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 4000.0 Hz

1618 repetitions

OBSERVE G13, 125.6889829 MHz

DECOUPLE H1, 499.8588575 MHz

Power 36 dB

on during acquisition

off during delay

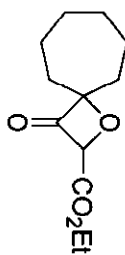
WALTZ-16 modulated

DATA PROCESSING

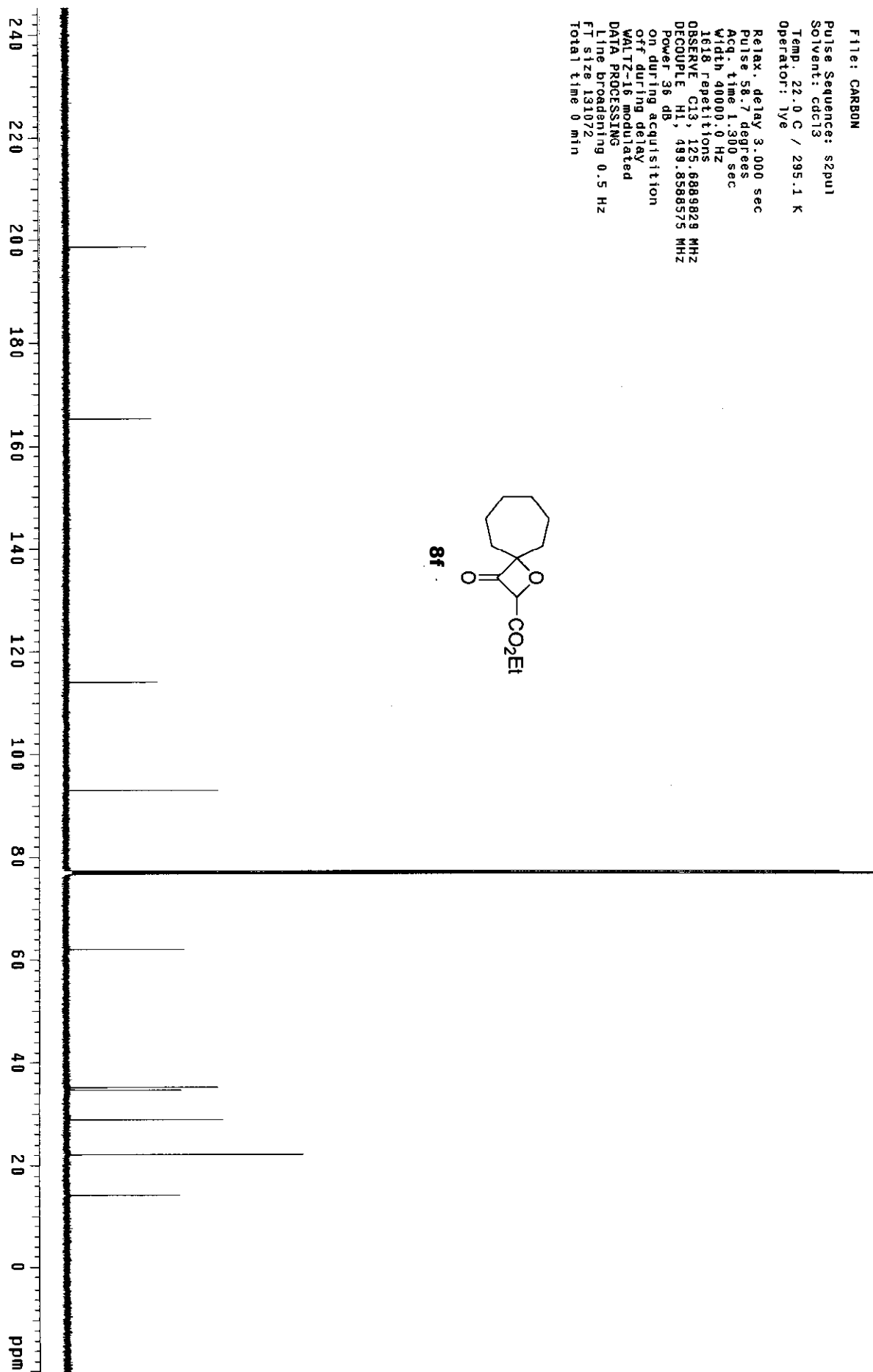
Line broadening 0.5 Hz

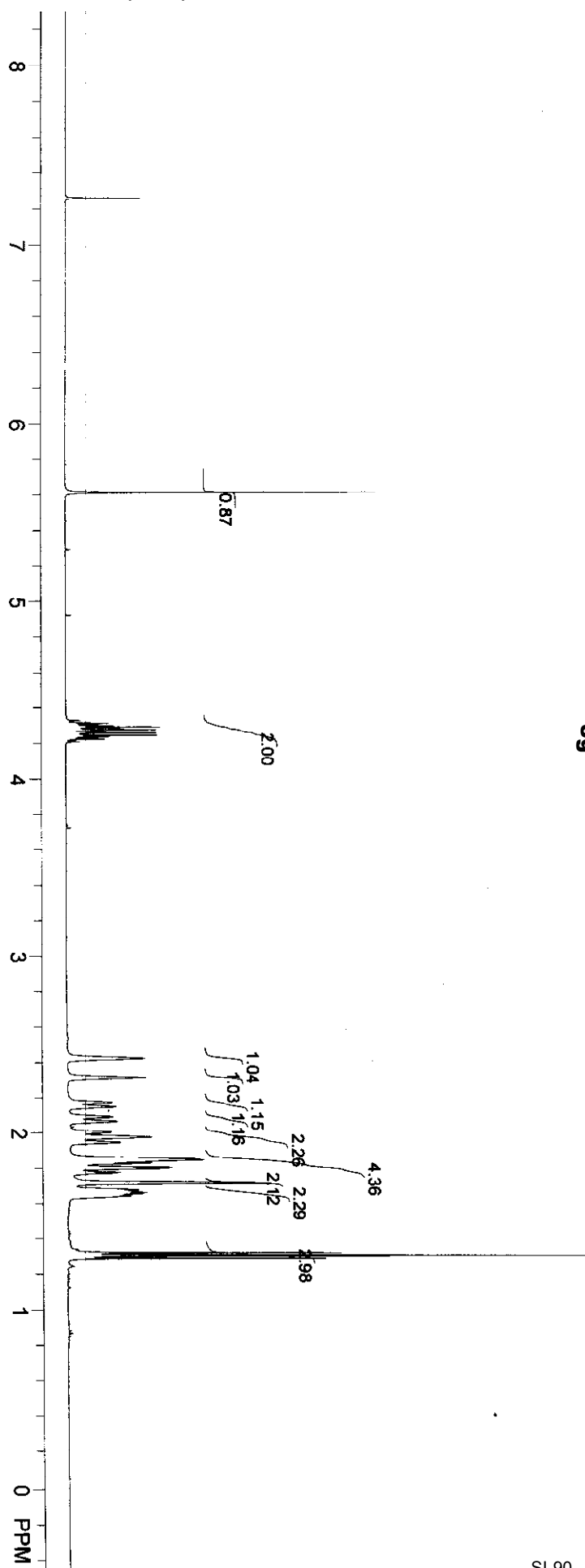
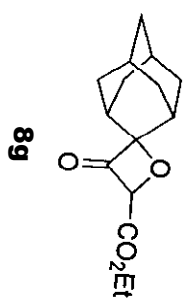
FT size 131072

Total time 0 min



8f





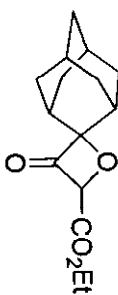
y1w-3-286-2-c

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmr500/data  
Sample directory:

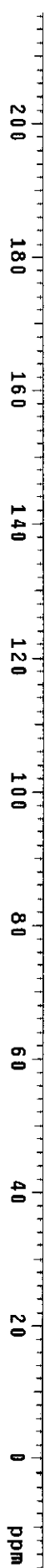
File: CARBON

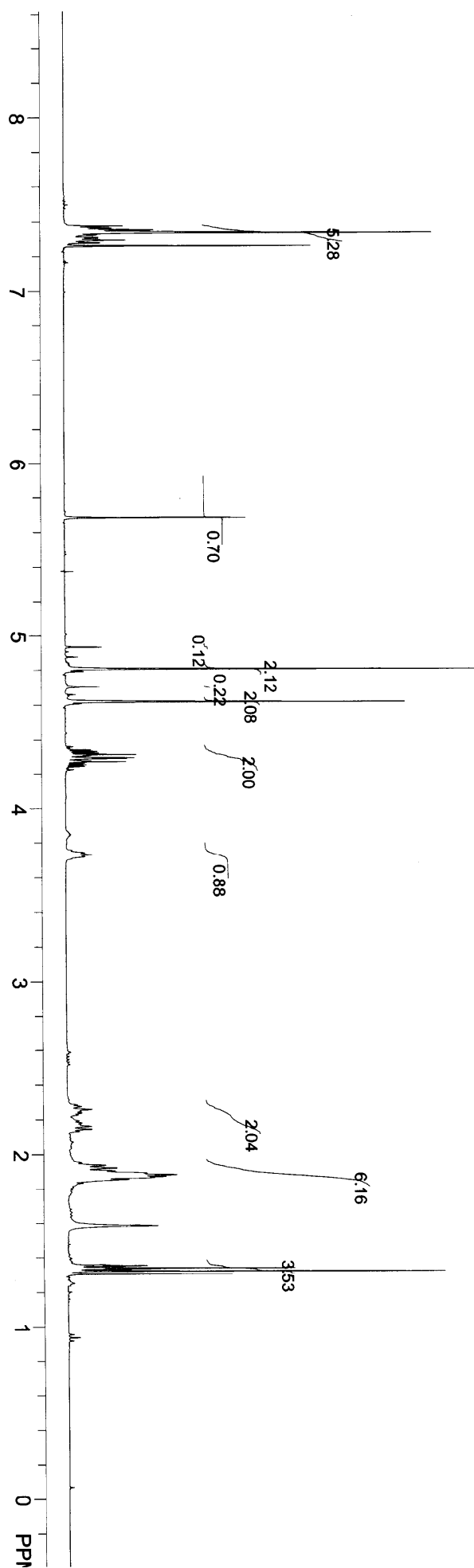
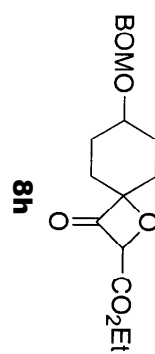
Pulse Sequence: s2pul  
Solvent: cdcl3  
Temp: 22.0 C / 295.1 K  
Operator: lye

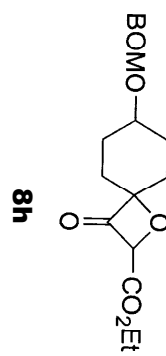
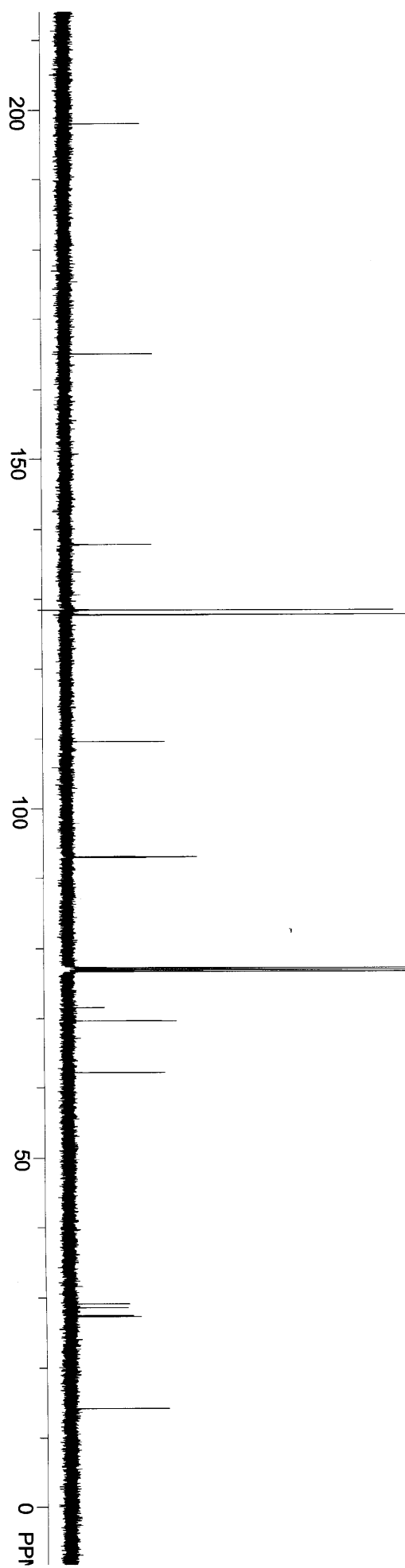
Relax. delay 3.000 sec  
Pulse 58.7 degrees  
Acq. time 1.300 sec  
Width 40000.0 Hz  
124 repetitions  
OBSERVE C13, 125.5889847 MHz  
DECUPLE H1, 499.8588575 MHz  
Power 36 db  
on during acquisition  
off during delay  
WALTZ-16 modulated  
DATA PROCESSING  
line broadening 0.5 Hz  
FT size 131072  
Total time 0 min

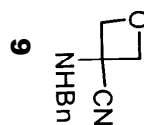
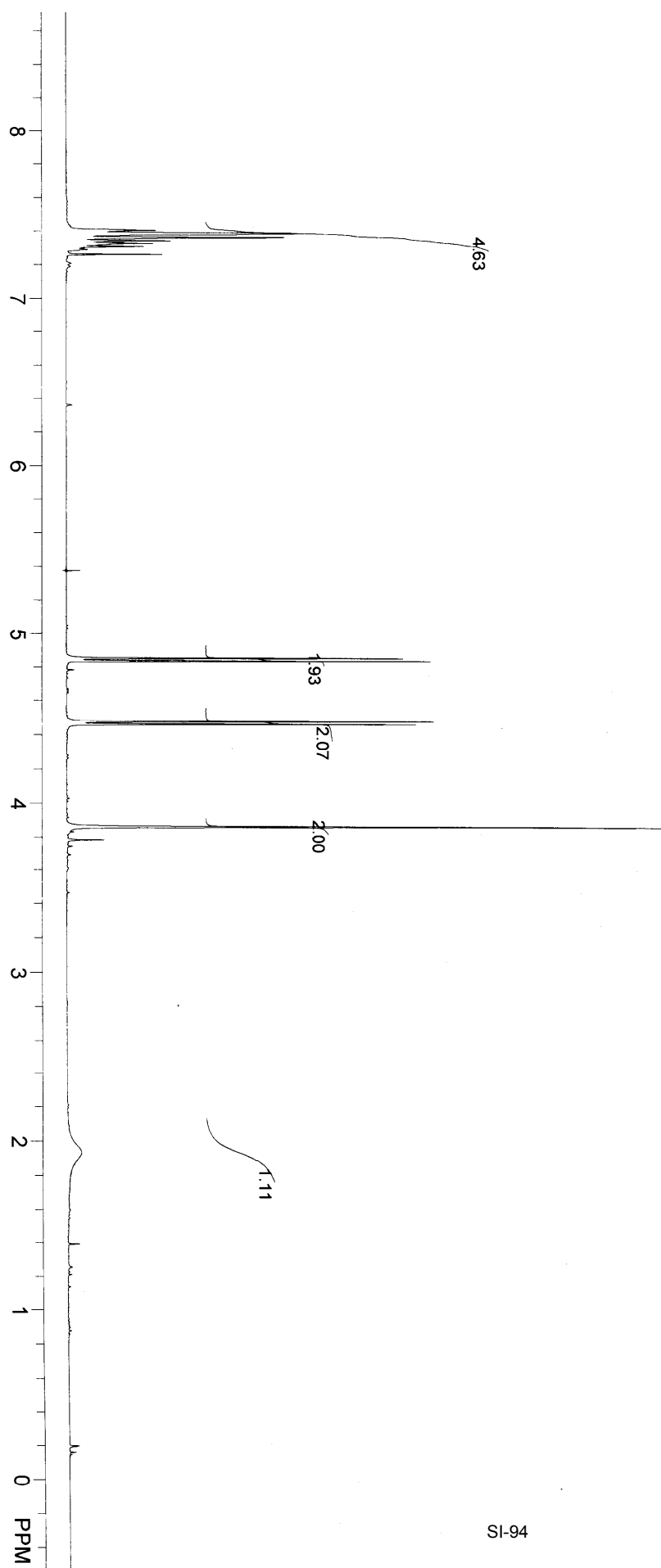


8g









Ylw-3-304-CN-C

Data Collected on:  
nm1500-inova500  
Archive directory:  
/export/home/vnmr1/vnmrSYS/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pul

Solvent: cdcl3

Temp. 22.0 C / 295.1 K

Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 4000.0 Hz

124 repetitions

OBSERVE C13, 125.6889860 MHz

DECOUPLE H1, 499.8588575 MHz

Power 36 db

on during acquisition

off during delay

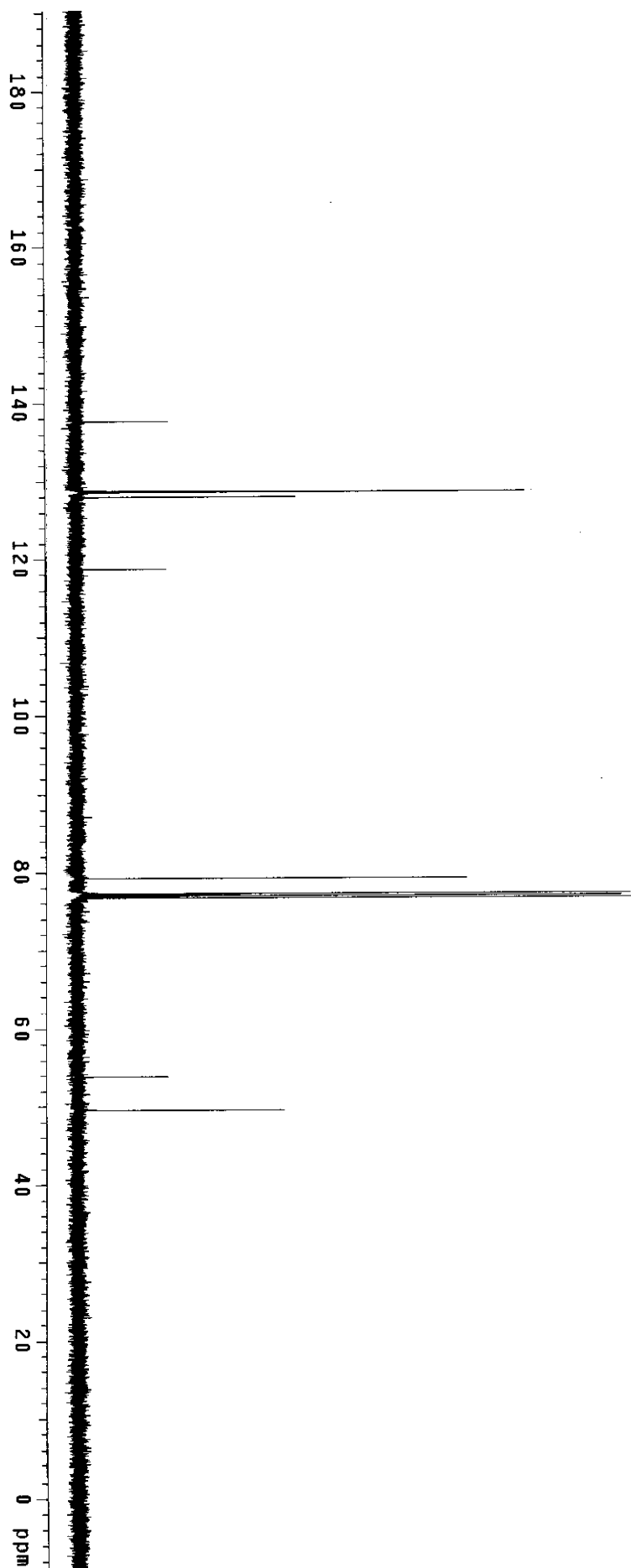
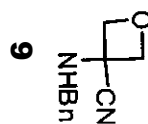
WALTZ-16 modulated

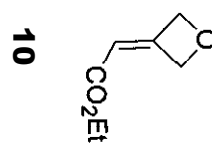
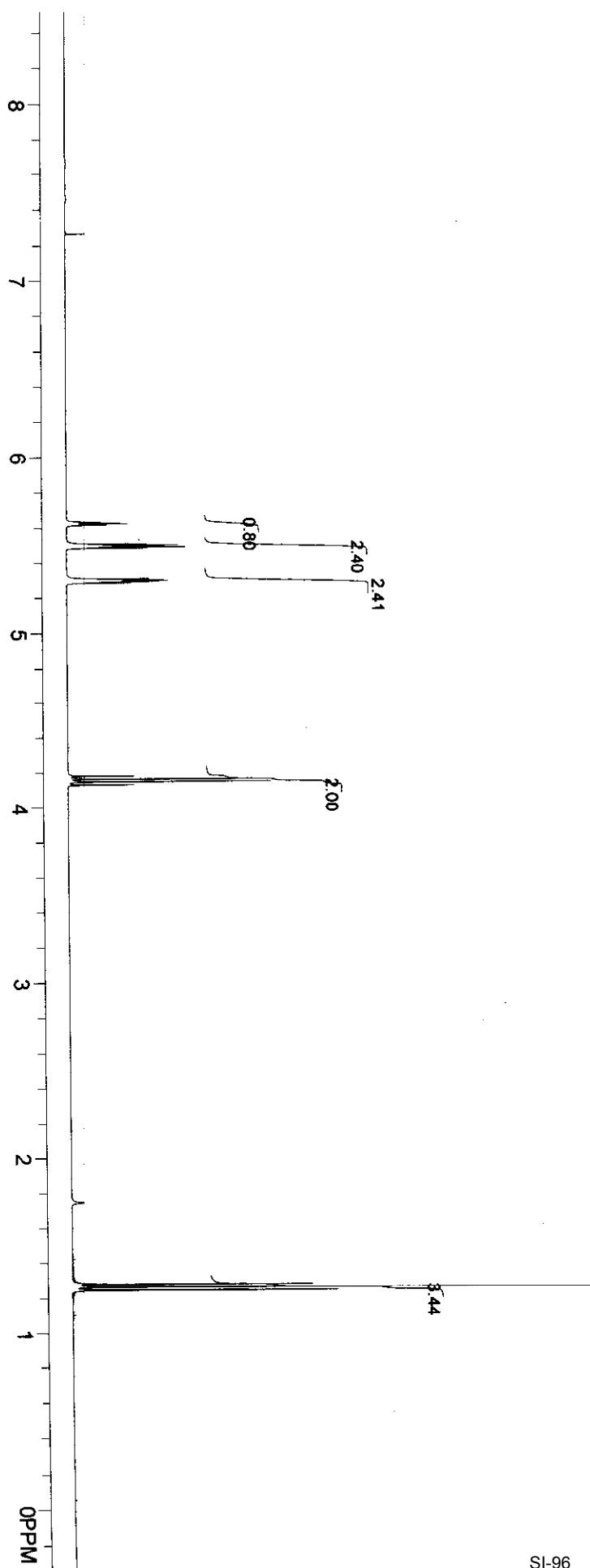
DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 0 min







y1w-3-272-c

Data Collected on:  
nmr500-inova500  
Archive directory:  
/export/home/vnmr1/vnmr500/data  
Sample directory:

File: CARBON

Pulse Sequence: s2pu1  
Solvent: cdcl3

Temp. 22.0 C / 295.1 K  
Operator: lye

Relax. delay 3.000 sec

Pulse 58.7 degrees

Acq. time 1.300 sec

Width 40000.0 Hz

56 repetitions

OBSERVE C13, 125.6888660 MHz

DECOUPLE H1, 499.8588575 MHz

Power 36 db

on during acquisition

off during delay

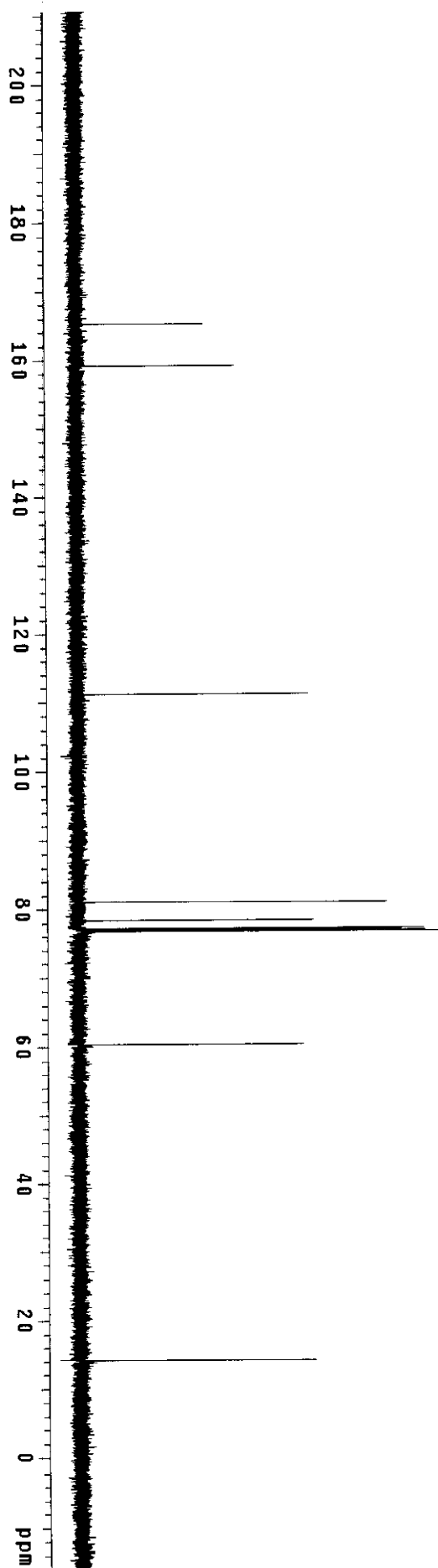
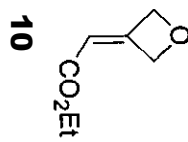
WALTZ-16 modulated

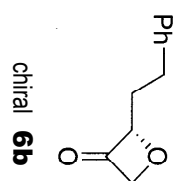
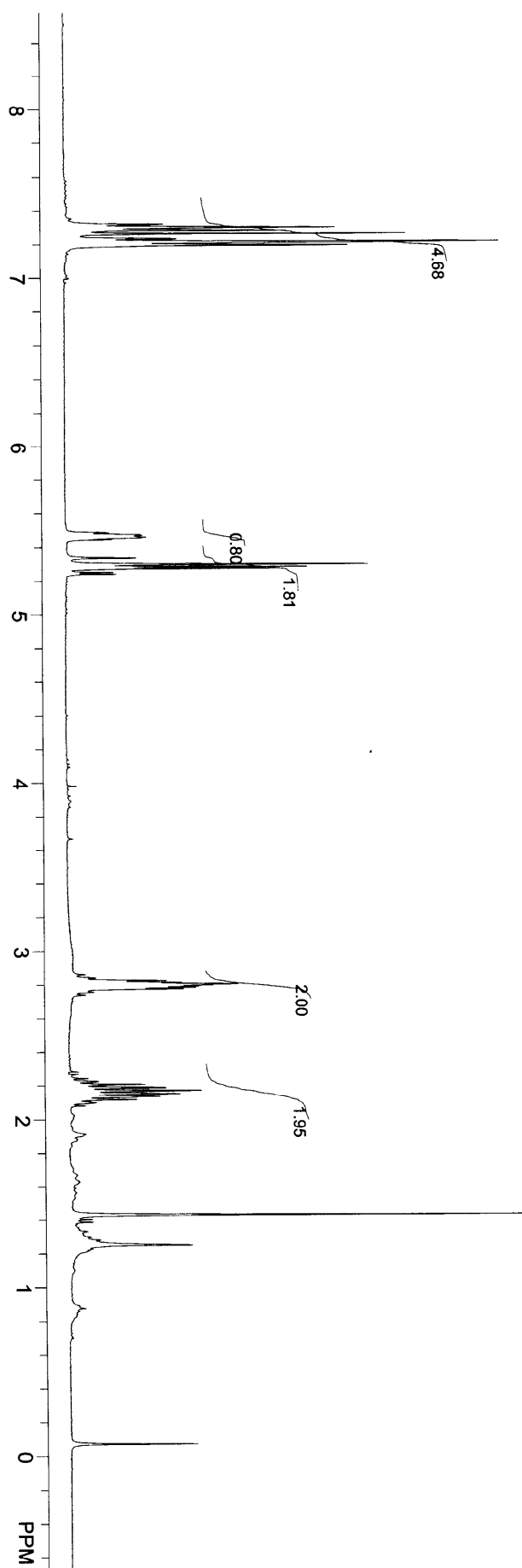
DATA PROCESSING

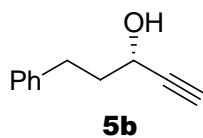
Line broadening 0.5 Hz

FT size 131072

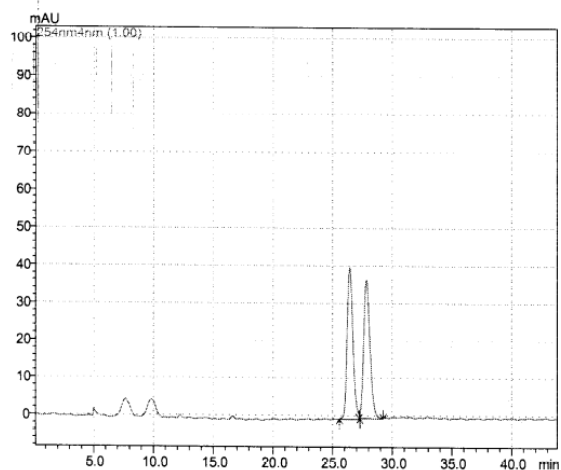
Total time 0 min







&lt;Chromatogram&gt;

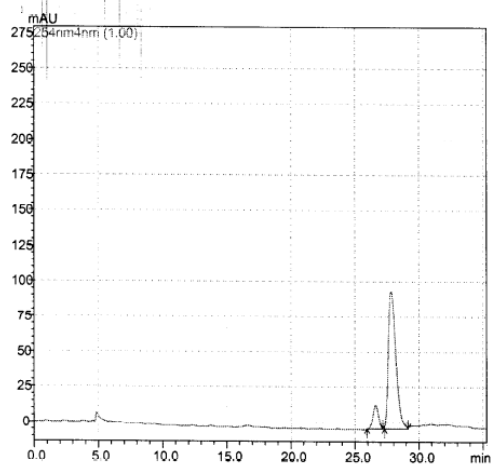


PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Area %
1	26.441	1382759	50.028
2	27.840	1381195	49.972
Total		2763954	100.000

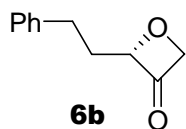
&lt;Chromatogram&gt;



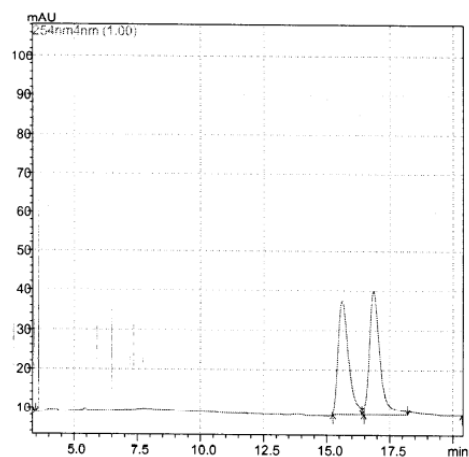
PeakTable

PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Area %
1	26.615	422444	9.865
2	27.830	3859983	90.135
Total		4282427	100.000



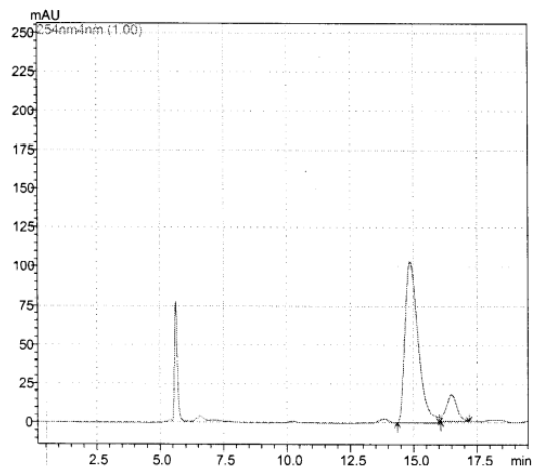
## &lt;Chromatogram&gt;



PeakTable

Peak#	Ret. Time	Area	Area %
1	14.934	724498	49.678
2	16.305	733876	50.322
Total		1458374	100.000

## &lt;Chromatogram&gt;



PeakTable

Peak#	Ret. Time	Area	Area %
1	15.090	1377288	90.354
2	16.590	147039	9.646
Total		1524327	100.000