

# **Total Synthesis of (+)-Suaveolindole: Establishment of its Absolute Configuration**

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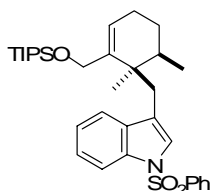
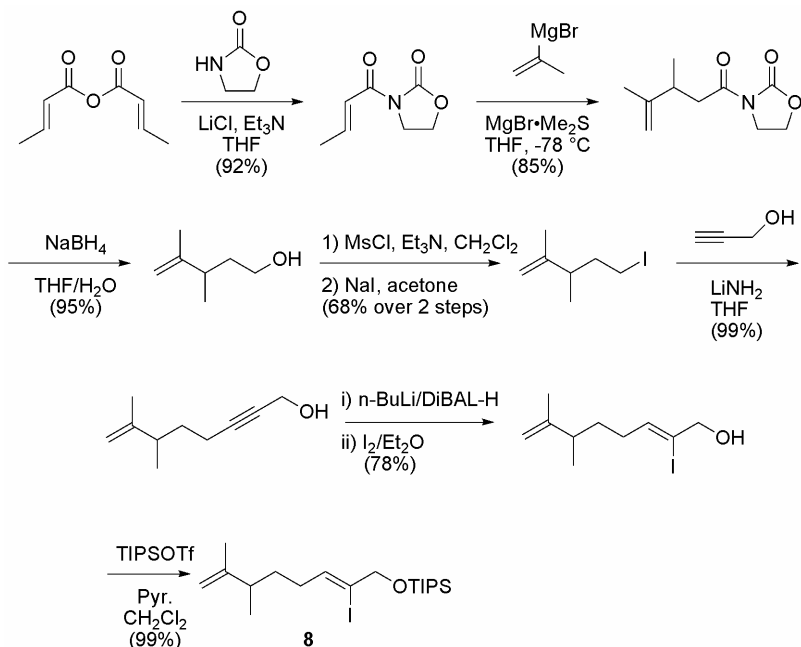


**General Information:**

**General Experimental Details:** Reactions involving air or moisture-sensitive reagents or intermediates were performed under argon or nitrogen atmosphere in glassware which had been oven dried, or flame-dried under high vacuum. Indicated reaction temperatures refer to those of the reaction bath, while room temperature (rt) is noted as 23 °C. Preparative reactions were stirred magnetically. Tetrahydrofuran (THF), diethyl ether (Et<sub>2</sub>O), methylene chloride (CH<sub>2</sub>Cl<sub>2</sub>), and toluene were obtained from a dry solvent system (activated alumina columns, positive pressure of argon). All other solvents were used as received in Sure/Seal bottles (Aldrich). Triethylamine (Et<sub>3</sub>N), diisopropylethylamine (*i*-Pr<sub>2</sub>NEt), pyridine, 2,6-lutidine, and chlorotrimethylsilane (TMSCl) were distilled from CaH<sub>2</sub> immediately prior to use. All other reagents were purchased from Aldrich at the highest commercial quality and used without further purification, with the exception of Pd(PPh<sub>3</sub>)<sub>4</sub>, and Pd<sub>2</sub>dba<sub>3</sub>, which were purchased from Strem and PhNTf<sub>2</sub> which was purchased from Oakwood Chemical.

**Instrumentation:** Optical rotations were measured on a JASCO DIP-370 digital polarimeter at rt. Concentration (c) in g/100 ml and solvent are given in parentheses. Infrared spectra were obtained on a Perkin-Elmer 1600 FT-IR spectrophotometer neat or as a film in CHCl<sub>3</sub> (NaCl plates). Absorption bands are noted in cm<sup>-1</sup>. <sup>1</sup>H- and <sup>13</sup>C NMR spectra were recorded on a Bruker AMX-400, a Bruker DRX-500, or a Bruker AVII+-600 spectrometer in CDCl<sub>3</sub>. Chemical shifts (δ-values) are reported in ppm with residual undeuterated CHCl<sub>3</sub> as the internal standard (referenced to 7.26 ppm for <sup>1</sup>H-NMR and 77.2 ppm for <sup>13</sup>C-NMR). Coupling constants (J) (H,H) are given in Hz, spectral splitting patterns are designated as singlet (s), doublet (d), triplet (t), quadruplet (q), quintet (qunit), multiplet or more overlapping signals (m), apparent (app), broad signal (br). Low resolution mass spectra (ionspray, a variation of electrospray) were acquired on a Perkin-Elmer Sciex API 100 spectrometer. Samples were introduced by direct infusion. High resolution mass spectra data was collected at the CUNY Mass Spectrometry Facility at Hunter College on an Agilent Technologies 6210 high resolution time-of-flight mass spectrometer attached to an Agilent Technologies 1200 HPLC system. The sample was ionized by simultaneous electrospray and atmospheric pressure chemical ionization with data being collected for both positive and negative ionization using Agilent's Multimode source. Flash chromatography (FC) was performed with E. Merck silica gel (60, particle size 0.040-0.063 mm) with loading approximately 50:1 (wt/wt) silica gel/crude residue. Preparative thin layer chromatography (TLC) was performed with Whatman Partisil Plates (20 x 20 cm, 60 Å, 200 μm).

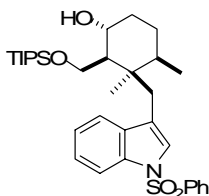


**Scheme 1.** Synthesis of Heck precursor **8**.

Chemical Formula:  $\text{C}_{33}\text{H}_{47}\text{NO}_3\text{SSi}$   
 Exact Mass: 565.30459  
 Molecular Weight: 565.88168

**Indole 10.** A solution of alkenyl iodide **8** (0.5 g, 1.15 mmol), indole stannane **9** (0.94 g, 1.7 mmol), and  $\text{Bu}_4\text{NCl}$  (0.35 g, 1.3 mmol) in DMF (4 mL) was degassed with Ar. After 15 min,  $\text{Ph}_3\text{As}$  (35 mg, 0.12 mmol) and  $\text{Pd}_2\text{dba}_3$  (26 mg, 0.029 mmol) were added in one portion and the reaction was immediately submersed in a 105 °C oil bath. After stirring for 3 h, the reaction was cooled to 23 °C and poured into  $\text{H}_2\text{O}$  (10 mL) and  $\text{Et}_2\text{O}$  (10 mL). The layers were partitioned, and the aqueous phase was extracted with  $\text{Et}_2\text{O}$  (2 × 20 mL). The combined organic extracts were washed with  $\text{H}_2\text{O}$  (5 × 10 mL), brine (10 mL), dried ( $\text{MgSO}_4$ ), filtered and concentrated. The crude residue was purified by flash column chromatography on silica gel (1% ethyl acetate – hexanes) to yield 0.36g (55%) of **10** as a 4:1 mixture of diastereomers; major isomer:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  7.99 (d,  $J$  = 8.1 Hz, 1H), 7.84 (d,  $J$  = 7.6 Hz, 2H), 7.54–7.49 (m, 2H), 7.42–7.39 (m, 3H), 7.29–7.19 (m, 2H), 5.84 (bs, 1H), 4.10 (d,  $J$  = 13 Hz, 1H), 4.00 (d,  $J$  = 13 Hz, 1H), 2.87 (d,  $J$  = 14.5 Hz, 1H), 2.71 (d,  $J$  = 14.5 Hz, 1H), 2.00–1.94 (m, 2H), 1.64–1.56 (m, 2H), 1.46–1.41 (m, 1H), 1.29–1.21 (m, 2H), 1.21 (s, 3H), 1.06–0.89 (m, 33H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz):  $\delta$  141.59, 138.47, 134.79, 133.56, 132.28, 129.10, 126.64, 124.45, 124.39, 123.53, 122.98, 120.84, 120.13, 113.57, 64.48, 40.12, 38.24, 29.76, 26.29, 25.67, 23.88, 18.09, 17.71, 16.15, 12.29, 11.96; HRMS (ESI,  $[\text{M}+\text{Na}]^+$ ) calculated for:  $\text{C}_{33}\text{H}_{47}\text{NO}_3\text{SSiNa}$  588.2938. Found: 588.2933.

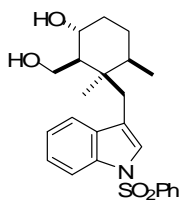




Chemical Formula:  $C_{33}H_{49}NO_4SSi$   
 Exact Mass: 583.31516  
 Molecular Weight: 583.89696

**Alcohol 11.** A solution of  $BH_3 \cdot THF$  (0.53 mL, 0.53 mmol, 1.0 M THF) was added dropwise to a solution of **10** (0.1 g, 0.18 mmol) in THF (0.5 mL) at 0 °C. The mixture was stirred for 3 h allowing it to warm to 23 °C. Excess borane was quenched by the slow addition of  $H_2O$  (36  $\mu$ L), and the organoborane was sequentially treated with  $NaBO_3 \cdot 4H_2O$  (0.14 g, 0.9 mmol) and  $H_2O$  (0.18 mL). The reaction mixture was stirred for 16 h at 23 °C, whereupon it was diluted with  $Et_2O$  (5 mL) and  $H_2O$  (5 mL). The layers were partitioned and the aqueous phase was

extracted with  $Et_2O$  ( $2 \times 5$  mL), the combined organic extracts were washed with brine (10 mL), dried ( $MgSO_4$ ), filtered and concentrated. The crude residue was purified by flash column chromatography on silica gel (10% ethyl acetate – hexanes) to yield 56 mg (56%) of **11** as a colorless oil:  $^1H$  NMR ( $CDCl_3$ , 400 MHz):  $\delta$  7.99 (d,  $J$  = 7.8 Hz, 1H), 7.88 (d,  $J$  = 7.8 Hz, 2H), 7.53–7.52 (m, 2H), 7.45–7.42 (m, 2H), 7.33–7.26 (m, 3H), 4.14–4.06 (m, 1H), 3.70 (d,  $J$  = 9.7 Hz, 1H), 3.52 (dd,  $J$  = 14.6, 9.7 Hz, 1H), 2.65 (d,  $J$  = 22 Hz, 1H), 2.52 (d,  $J$  = 22 Hz, 1H), 1.46–1.34 (m, 3H), 1.22–1.19 (m, 3H), 0.90–0.83 (m, 28H);  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz):  $\delta$  138.36, 134.73, 133.69, 131.93, 129.21, 126.71, 124.65, 124.62, 123.37, 120.31, 119.85, 113.71, 73.18, 70.57, 65.77, 56.49, 43.96, 40.43, 35.58, 28.71, 25.09, 23.37, 17.70, 17.64, 16.46, 11.31. HRMS (ESI,  $[M+Na]^+$ ) calculated for:  $C_{33}H_{49}NO_4SSiNa$  606.3043. Found: 606.3042.

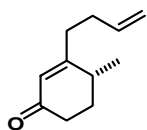
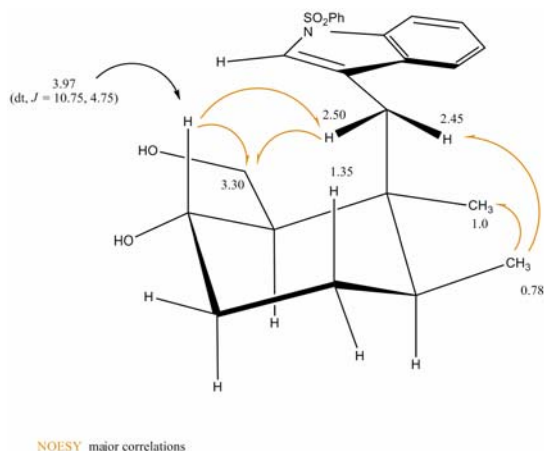


Chemical Formula:  $C_{24}H_{29}NO_4S$   
 Exact Mass: 427.18173  
 Molecular Weight: 427.55636

**Deprotection of 11:** A solution of **11** (10 mg, 0.017 mmol) in THF (100  $\mu$ L) and AcOH (4  $\mu$ L, 0.068 mmol) at 23 °C was treated with TBAF (34  $\mu$ L, 0.034 mmol, 1.0 M THF). After stirring for 16 h, the reaction was concentrated in vacuo and the crude residue was purified by preparative TLC (20% ethyl acetate – hexanes) to afford the title compound as a white foam (7.0 mg, 100% yield):  $^1H$  NMR ( $CDCl_3$ , 500 MHz):  $\delta$  7.95 (d,  $J$  = 8.2 Hz, 1H), 7.79 (d,  $J$  = 7.9 Hz, 2H), 7.47–7.42 (m, 2H), 7.36 (dd,  $J$  = 7.9, 7.8 Hz, 2H), 7.25–7.24 (m, 1H), 7.21–7.18 (m, 2H),

3.97 (dt,  $J$  = 10.75, 4.75 Hz, 1H), 3.44–3.40 (m, 1H), 3.30 (dd,  $J$  = 10.85, 8.3 Hz, 1H), 2.64 (bs, 1H), 2.50 (d,  $J$  = 15 Hz, 1H), 2.45 (d,  $J$  = 15 Hz, 1H), 2.02–1.98 (m, 1H), 1.52–1.31 (m, 5H), 1.25–1.17 (m, 3H), 1.15 (s, 3H), 0.78 (d,  $J$  = 6.9 Hz, 3H);  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz):  $\delta$  138.21, 134.95, 133.76, 132.16, 129.23, 128.34, 126.62, 124.97, 124.69, 123.34, 120.77, 119.85, 113.93, 73.55, 63.95, 57.91, 43.85, 41.06, 36.42, 29.69, 28.97, 24.95, 23.57, 16.49. HRMS (ESI,  $[M+Na]^+$ ) calculated for:  $C_{24}H_{29}NO_4SNa$  450.1709. Found: 450.1707.



Structural Determination of Deprotected **11**:

**Chemical Formula:** C<sub>11</sub>H<sub>16</sub>O  
**Exact Mass:** 164.1201  
**Molecular Weight:** 164.2441

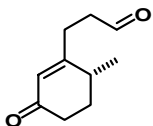
**(R)-3-(but-3-enyl)-4-methylcyclohex-2-enone (13).**<sup>1</sup>

A solution of 4-bromo-1-butene (1.0 mL, 10 mmol) in THF (4.6 mL) was added dropwise over a 20 min period to a stirring suspension of Mg turnings (240 mg, 10 mmol) in THF (2.0 mL). Following completion of the addition, the reaction mixture was stirred for 30 min at 23 °C and cannulated into a solution of (*R*)-**12**<sup>2</sup> in THF (17 mL) at –78 °C. After 20 min, the reaction was warmed to 23 °C and maintained at this temperature for 3 h. The reaction was quenched by cooling to 0 °C and cautiously adding H<sub>2</sub>O (10 mL). The layers were partitioned and the aqueous phase was extracted with Et<sub>2</sub>O (2 × 20 mL), the combined organic extracts were washed with brine (2 × 20 mL), dried (MgSO<sub>4</sub>), filtered and concentrated. The crude carbinol was then dissolved in CH<sub>2</sub>Cl<sub>2</sub> (6 mL) and added to a flask charged with PCC (3.9 g, 18.2 mmol) and CH<sub>2</sub>Cl<sub>2</sub> (18 mL) at 23 °C. The resulting reaction mixture was maintained at this temperature for 16 h, decanted, and the remaining polymeric material was thoroughly washed with Et<sub>2</sub>O (3 × 30 mL). The organic layers were combined, and sequentially washed with aqueous NaOH (2 × 20 mL, 5%), aqueous HCl (2 × 20 mL, 5%), saturated aqueous NaHCO<sub>3</sub> (2 × 20 mL) and brine (2 × 20 mL). The layers were dried (MgSO<sub>4</sub>), filtered, and concentrated. The crude residue was purified by flash column chromatography on silica gel (5→10% ethyl acetate – hexanes) to yield 0.7 g (47%) of (*R*)-**13** as a pale yellow oil: [α]<sub>D</sub><sup>22</sup> = +15° (c = 2.3, CHCl<sub>3</sub>); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 5.82–5.75 (m, 2H), 5.06–4.98 (m, 2H), 2.51–2.41 (m, 2H), 2.34–2.19 (m, 5H), 2.13–2.06 (m, 1H), 1.80–1.74 (m, 1H), 1.19 (d, J = 7.6 Hz, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 199.88, 169.53, 137.28, 125.42, 115.79, 34.99, 34.42, 33.32, 31.40, 30.37, 18.0; HRMS (ESI, [M+H]<sup>+</sup>) calculated for: C<sub>11</sub>H<sub>17</sub>O 165.1273. Found: 165.1272.

(1) Access to racemic **13** was accomplished by a Stork-Danheiser reaction of 3-ethoxy-6-methylcyclohex-2-enone and but-3-enylmagnesium bromide in 94% yield, see: Stork, G.; Danheiser, R. L. *J. Org. Chem.* **1973**, 38, 1775–1776.

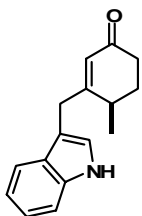
(2) (a) *Organic Syntheses*; Wiley & Sons: New York, 2005; Vol. 82, p 108. (b) Schreiber, S. L. *J. Am. Chem. Soc.* **1980**, 102, 6165–6166.





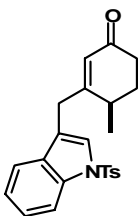
**Chemical Formula:** C<sub>10</sub>H<sub>14</sub>O<sub>2</sub>  
**Exact Mass:** 166.10  
**Molecular Weight:** 166.22

**(R)-3-(6-methyl-3-oxocyclohex-1-enyl)propanal (14).** A suspension of **13** (0.5 g, 3.0 mmol) and NaHCO<sub>3</sub> (12 mg, 0.15 mmol) in MeOH (30 mL) at -78 °C was treated with ozone until TLC indicated the reaction was complete. The reaction mixture was purged with argon for 10 min, and a solution of DMS (1.1 mL, 15 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.1 mL) was added. The reaction was maintained at 23 °C for 16 h, filtered through a pad of celite and concentrated. The crude residue (0.44 g, 88%) was dried in vacuo (450 mtorr) for 1 h and used without further purification:  $[\alpha]^{22}_D = +48^\circ$  (c = 1.3, CHCl<sub>3</sub>). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 9.82 (s, 1H), 5.72 (s, 1H), 2.71–2.67 (m, 2H), 2.63–2.43 (m, 4H), 2.35–2.29 (m, 1H), 2.15–2.08 (m, 1H), 1.82–1.76 (m, 1H), 1.21 (d, *J* = 6.9 Hz, 3 H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 200.21, 199.26, 167.72, 124.87, 41.0, 34.10, 33.49, 30.13, 27.29, 17.83; HRMS (ESI, [M+H]<sup>+</sup>) calculated for: C<sub>10</sub>H<sub>15</sub>O<sub>2</sub> 167.1066. Found 167.1063.



**Chemical Formula:** C<sub>16</sub>H<sub>17</sub>NO  
**Exact Mass:** 239.13101  
**Molecular Weight:** 239.31228

**(R)-3-((1H-indol-3-yl)methyl)-4-methylcyclohex-2-enone.** A mixture of 2-iodoaniline (6.4 g, 29.2 mmol), (*R*)-**14** (4.4 g, 26.5 mmol), and DABCO (8.9 g, 79.5 mmol) in DMF (130 mL) was degassed with Ar for 20 min. Pd(OAc)<sub>2</sub> (300 mg, 1.33 mmol) was added to the reaction, and the resulting reaction mixture was heated to 85 °C for 16 h. The reaction was cooled to 23 °C and was diluted with H<sub>2</sub>O (100 mL). The aqueous phase was extracted with Et<sub>2</sub>O (3 × 50 mL), and the combined organic phases were washed with brine (50 mL), dried (MgSO<sub>4</sub>), filtered and concentrated. The crude residue was purified by flash column chromatography on silica gel (30% ethyl acetate – hexanes) to yield 4.3 g (68%) of the title compound as a yellow foam:  $[\alpha]^{22}_D = +81^\circ$  (c = 0.9, CHCl<sub>3</sub>). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz): δ 8.03 (bs, 1H), 7.47 (d, *J* = 8.6 Hz, 1H), 7.27 (d, *J* = 9.5 Hz, 1H), 7.19 (dd, *J* = 15.3, 7.6 Hz, 1H), 7.09 (dd, *J* = 16.2, 7.6 Hz, 1H), 7.03 (s, 1H), 5.84 (s, 1H), 3.68 (s, 3H), 2.54–2.46 (m, 2H), 2.31 (ddd, *J* = 11.5, 11.5, 5.8, 1H), 2.11–2.04 (m, 1H), 1.81–1.75 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz): δ 200.28, 169.56, 136.69, 127.41, 125.78, 122.95, 122.16, 119.59, 118.71, 111.33, 111.16, 34.09, 32.57, 31.97, 30.22, 17.83; HRMS (ESI, [M+H]<sup>+</sup>) calculated for: C<sub>16</sub>H<sub>18</sub>NO 240.1382. Found: 240.1383.

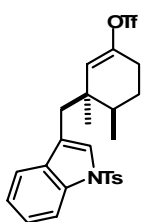


**Chemical Formula:** C<sub>23</sub>H<sub>23</sub>NO<sub>3</sub>S  
**Exact Mass:** 393.13986  
**Molecular Weight:** 393.49862

**(R)-4-methyl-3-((1-tosyl-1H-indol-3-yl)methyl)cyclohex-2-enone (15).** Tosyl chloride (0.87 g, 4.60 mmol) was added to a mixture of (*R*)-3-((1H-indol-3-yl)methyl)-4-methylcyclohex-2-enone (1.0 g, 4.18 mmol), and TBAB (0.14 g, 0.42 mmol) in NaOH (14 mL, 15% aq), and benzene (13 mL) at 23 °C. The reaction was maintained at this temperature for 2 h, and monitored by <sup>1</sup>H NMR analysis. Upon consumption of starting material the layers were partitioned, and the aqueous phase was extracted with Et<sub>2</sub>O (3 × 20 mL). The combined organic phases were washed with brine (50 mL), dried (MgSO<sub>4</sub>), filtered and concentrated. The crude residue was purified by flash column chromatography on silica gel (20→30% ethyl acetate – hexanes) to yield 1.5 g



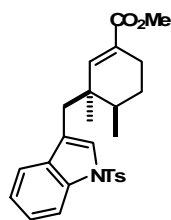
(91%) of **15** as a yellow foam:  $[\alpha]_D^{22} = +48^\circ$  ( $c = 2.5$ ,  $\text{CHCl}_3$ ).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.98 (d,  $J = 8.0$  Hz, 1H), 7.74 (d,  $J = 8.8$  Hz, 1H), 7.38 (s, 1H), 7.36–7.21 (m, 2H), 7.23–7.19 (m, 3H), 5.70 (s, 1H), 3.59 (s, 1H), 2.53–2.44 (m, 2H), 2.35–2.29 (m, 4H), 2.11–2.05 (m, 1H), 1.82–1.76 (m, 1H), 1.25 (d,  $J = 7.2$  Hz, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125MHz):  $\delta$  199.39, 166.72, 145.08, 135.4, 135.06, 130.48, 125.01, 124.54, 123.38, 119.48, 118.13, 113.91, 34.03, 31.63, 30.12, 21.60, 17.81; HRMS (ESI,  $[\text{M}+\text{H}]^+$ ) calculated for:  $\text{C}_{23}\text{H}_{24}\text{NO}_3\text{S}$  394.1471. Found: 394.1474.



**Chemical Formula:**  $\text{C}_{25}\text{H}_{26}\text{F}_3\text{NO}_5\text{S}_2$   
**Exact Mass:** 541.1204  
**Molecular Weight:** 541.6028

**(3*R*,4*R*)-3,4-dimethyl-3-((1-tosyl-1*H*-indol-3-yl)methyl)cyclohex-1-enyl trifluoromethanesulfonate (**16**).**

A suspension of CuI (0.87 g, 4.58 mmol), in  $\text{Et}_2\text{O}$  (38 mL) was cooled to  $-30^\circ\text{C}$  and treated dropwise with a solution of MeLi (7.4 mL, 9.17 mmol, 1.25 M in  $\text{Et}_2\text{O}$ ). After 10 min, the reaction mixture was warmed to  $0^\circ\text{C}$  resulting in a colorless, homogeneous solution. A solution of enone **15** (1.5 g, 3.82 mmol) in  $\text{Et}_2\text{O}$ -THF (6 mL–1.5 mL) was added over a 20 min period, resulting in an orange precipitate. After stirring for an additional 30 min, the reaction mixture was treated with a solution of  $\text{PhNTf}_2$  (3.14 g, 8.79 mmol) in THF (9 mL). After 20 min, saturated aqueous  $\text{NH}_4\text{Cl}$  was added, and the layers partitioned. The aqueous phase was extracted with  $\text{Et}_2\text{O}$  ( $2 \times 20$  mL), and the combined organic extracts were washed with brine ( $2 \times 20$  mL), dried ( $\text{MgSO}_4$ ), filtered and concentrated. The residue was purified by flash column chromatography on silica gel (5→10% ethyl acetate – hexanes) to yield (*R,R*)-**16** as a 9:1 mixture of diastereomers and an inseparable mixture<sup>3</sup> with  $\text{PhNHTf}$ :  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 600 MHz):  $\delta$  7.92 (d,  $J = 8.4$  Hz, 1H), 7.68 (d,  $J = 8.4$  Hz, 2H), 7.32 (d,  $J = 7.9$  Hz, 1H), 7.22 (dd,  $J = 7.6, 7.4$  Hz, 1H), 7.17–7.12 (m, 3H), 2.60 (d,  $J = 13.8$  Hz, 1H), 2.54 (d,  $J = 13.8$  Hz, 1H), 2.36–2.27 (m, 2H), 2.25 (s, 3H), 1.72–1.67 (m, 2H), 1.63–1.55 (m, 1H), 1.00 (d,  $J = 7.0$  Hz, 3H), 0.95 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150MHz):  $\delta$  148.19, 144.91, 135.16, 135.06, 131.86, 128.84, 127.10, 126.74, 125.06, 124.60, 123.18, 120.06, 118.43, 113.73, 39.01, 38.07, 30.67, 30.67, 27.35, 27.09, 26.11, 21.51, 15.22; HRMS (ESI,  $[\text{M}+\text{Na}]^+$ ) calculated for:  $\text{C}_{25}\text{H}_{26}\text{NO}_5\text{F}_3\text{S}_2\text{Na}$  564.1096. Found: 564.1096.



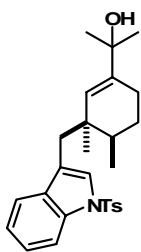
**Chemical Formula:**  $\text{C}_{26}\text{H}_{29}\text{NO}_4\text{S}$   
**Exact Mass:** 451.18  
**Molecular Weight:** 451.58

**(3*R*,4*R*)-Methyl 3,4-dimethyl-3-((1-tosyl-1*H*-indol-3-yl)methyl)cyclohex-1-enecarboxylate.** A mixture of enol triflate **16** (2.0 g, 3.8 mmol), *i*- $\text{Pr}_2\text{EtN}$  (3.3 mL, 19 mmol), MeOH (13 mL, 0.3 M), and DMF (20 mL) was purged with Ar for 5 min.  $\text{Pd}(\text{PPh}_3)_4$  (0.22 g, 0.19 mmol) was added and CO was bubbled through the reaction mixture for 30 min.  $\text{Et}_2\text{O}$  (20 mL) and  $\text{H}_2\text{O}$  (20 mL) were added, and the layers partitioned. The aqueous phase was extracted with  $\text{Et}_2\text{O}$  ( $2 \times 20$  mL), and the combined organic extracts were washed with water ( $5 \times 20$  mL), brine ( $2 \times 20$  mL), dried ( $\text{MgSO}_4$ ), filtered and concentrated. The residue was purified by flash column chromatography on silica gel (5→10% ethyl acetate – hexanes) to yield the title

<sup>3</sup> Note, that on small scale, recrystallization of **18** from 5% ethyl acetate was successful in removing majority of the  $\text{PhNHTf}$ .

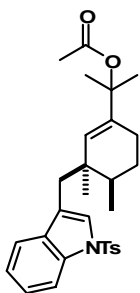


compound (0.76 g, 45% from **15**) as a white foam:  $[\alpha]_D^{22} = +40^\circ$  ( $c = 1.1$ ,  $\text{CHCl}_3$ ).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 600 MHz):  $\delta$  7.90 (d,  $J = 8.3$  Hz, 1H), 7.69 (d,  $J = 8.3$  Hz, 2H), 7.30 (d,  $J = 7.9$  Hz, 1H), 7.22–7.18 (m, 1H), 7.14 (d,  $J = 8.2$  Hz, 2H), 7.11–7.10 (m, 1H), 6.40 (s, 1H), 2.60 (dd,  $J = 22.3, 13.8$ , 2H), 2.38–2.35 (m, 1H), 2.26 (s, 3H), 2.18–2.12 (m, 1H), 1.67–1.61 (m, 1H), 1.58–1.51 (m, 2H), 1.00 (d,  $J = 7.6$  Hz, 3H), 0.91 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150MHz):  $\delta$  167.97, 147.84, 144.74, 135.17, 135.05, 132.16, 129.80, 129.12, 126.81, 125.06, 124.42, 122.98, 120.35, 119.19, 113.69, 51.62, 38.46, 38.17, 30.76, 26.75, 25.57, 24.27, 21.56, 16.0; HRMS (ESI,  $[\text{M}+\text{H}]^+$ ) calculated for:  $\text{C}_{26}\text{H}_{30}\text{NO}_4\text{S}$  452.1890. Found: 452.1887.



**Chemical Formula:**  $\text{C}_{27}\text{H}_{33}\text{NO}_3\text{S}$   
**Exact Mass:** 451.22  
**Molecular Weight:** 451.62

**2-((3R,4R)-3,4-dimethyl-3-((1-tosyl-1H-indol-3-yl)methyl)cyclohex-1-enyl)propan-2-ol.** A solution the above  $\alpha,\beta$ -unsaturated ester (0.76 g, 1.69 mmol) in THF (13 mL) at  $0^\circ\text{C}$  was treated with MeLi (4.0 mL, 5.0 mmol, 1.25 M in  $\text{Et}_2\text{O}$ ). After 10 min, the reaction was poured into half-saturated  $\text{NH}_4\text{Cl}$  (20 mL) and  $\text{Et}_2\text{O}$  (20 mL) and the layers partitioned. The aqueous phase was extracted with  $\text{Et}_2\text{O}$  ( $2 \times 10$  mL), and the combined organic extracts were washed with brine ( $2 \times 10$  mL), dried ( $\text{MgSO}_4$ ), filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10 $\rightarrow$ 20% ethyl acetate – hexanes) to yield the title compound (0.64 g, 84%) as a white foam:  $[\alpha]_D^{22} = +52^\circ$  ( $c = 1.1$ ,  $\text{CHCl}_3$ ).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.99 (d,  $J = 8.3$  Hz, 1H), 7.75 (d,  $J = 8.3$  Hz, 1H), 7.37 (d,  $J = 7.9$  Hz, 1H), 7.31 (s, 1H), 7.29–7.26 (m, 2H), 7.21–7.18 (m, 3H), 2.67 (d,  $J = 13.5$  Hz, 1H), 2.52 (d,  $J = 13.5$  Hz, 1H), 2.38 (s, 3H), 2.21–2.18 (m, 1H), 2.06–2.00 (m, 1H), 1.66–1.57 (m, 3H), 1.15 (s, 3H), 1.06–1.05 (m, 6H), 1.00 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150MHz):  $\delta$  144.92, 142.04, 135.44, 135.33, 132.70, 130.19, 129.97, 128.66, 127.62, 126.95, 124.85, 124.64, 122.84, 120.80, 120.49, 114.03, 72.77, 44.85, 39.04, 37.48, 31.33, 28.86, 28.78, 28.04, 27.94, 26.86, 24.65, 21.76, 16.10; HRMS (ESI,  $[\text{M}+\text{Na}]^+$ ) calculated for:  $\text{C}_{27}\text{H}_{33}\text{NO}_3\text{SNa}$  474.2073. Found: 474.2069.

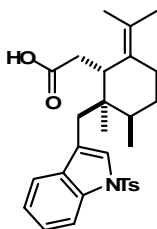


**Chemical Formula:**  $\text{C}_{29}\text{H}_{35}\text{NO}_4\text{S}$   
**Exact Mass:** 493.23  
**Molecular Weight:** 493.66

**2-((3R,4R)-3,4-dimethyl-3-((1-tosyl-1H-indol-3-yl)methyl)cyclohex-1-enyl)propan-2-yl acetate (17).** A mixture of the above tertiary allylic alcohol (0.5 g, 1.11 mmol),  $i\text{-Pr}_2\text{EtNH}$  (2.0 mL, 11.1 mmol),  $\text{Ac}_2\text{O}$  (0.63 mL, 6.65 mmol), and DMAP (13 mg, 0.11 mmol) in  $\text{CH}_2\text{Cl}_2$  (2 mL) was maintained at  $23^\circ\text{C}$  for 16 h.  $\text{Et}_2\text{O}$  (10 mL) and  $\text{H}_2\text{O}$  (10 mL) were added, and the layers partitioned. The aqueous phase was extracted with  $\text{Et}_2\text{O}$  ( $2 \times 10$  mL), and the combined organic extracts were washed with brine (20 mL), dried ( $\text{MgSO}_4$ ), filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (5% ethyl acetate – hexanes) to yield **17** (0.45 g, 83%) as a white foam:  $[\alpha]_D^{22} = +67^\circ$  ( $c = 1.1$ ,  $\text{CHCl}_3$ ).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz):  $\delta$  7.86 (d,  $J = 8.3$  Hz, 1H), 7.64–7.62 (d,  $J = 8.3$  Hz, 2H), 7.35 (d,  $J = 7.9$  Hz, 1H), 7.17–7.14 (m, 2H), 7.08–7.06 (m, 3H), 2.53 (d,  $J = 13.5$  Hz, 1H), 2.42 (d,  $J = 13.5$  Hz, 1H), 2.21 (s, 3H), 1.95–1.92 (m, 1H), 1.82 (s, 3H), 1.52–1.46 (m, 4H), 1.23 (s, 3H), 1.18–1.13 (m, 4H), 0.93 (d,  $J = 5.9$



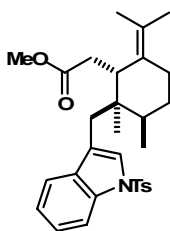
Hz, 3H), 0.85 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150MHz):  $\delta$  169.76, 144.62, 138.77, 135.36, 135.02, 132.49, 129.71, 126.74, 124.97, 124.25, 122.78, 120.75, 120.02, 113.52, 38.74, 37.39, 30.82, 27.44, 26.72, 26.52, 25.74, 24.03, 22.67, 22.13, 21.53, 15.90; HRMS (ESI,  $[\text{M}+\text{Na}]^+$ ) calculated for:  $\text{C}_{29}\text{H}_{35}\text{NO}_4\text{SNa}$  516.2179. Found: 516.2174.



**Chemical Formula:**  $\text{C}_{29}\text{H}_{35}\text{NO}_4\text{S}$   
**Exact Mass:** 493.23  
**Molecular Weight:** 493.66

**2-((1S,2R,3R)-2,3-dimethyl-6-(propan-2-ylidene)-2-((1-tosyl-1H-indol-3-yl)methyl)cyclohexyl)acetic acid (18).** A solution of **17** (0.45 g, 0.91 mmol) in THF (4.0 mL) was added dropwise over a 5 min period to a mixture of LiHMDS (3.0 mL, 3.0 mmol, 1.0 M THF) and TMSCl (0.35 mL, 2.74 mmol) in THF (5.0 mL) at  $-78^\circ\text{C}$ . The reaction mixture was warmed to  $23^\circ\text{C}$  over a 2 h period, and maintained at this temperature for 16 h.  $\text{Et}_2\text{O}$  (10 mL) and 2M HCl (10 mL) were added, and the layers partitioned. The aqueous phase was extracted with  $\text{Et}_2\text{O}$  ( $3 \times 5$  mL), and the combined organic extracts were washed with brine (10 mL), dried

( $\text{MgSO}_4$ ), filtered, and concentrated. The crude residue was purified by flash column chromatography on silica gel (10% ethyl acetate – hexanes with 1% AcOH) to yield 0.25g (56%) of the title compound as a white foam:  $[\alpha]_D^{22} = +89^\circ$  ( $c = 0.9$ ,  $\text{CHCl}_3$ ).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 600 MHz):  $\delta$  8.06 (d,  $J = 8.3$  Hz, 1H), 7.80 (d,  $J = 8.3$  Hz, 2H), 7.49–7.45 (m, 2H), 7.39–7.34 (m, 1H), 7.28–7.24 (m, 3H), 3.26–3.20 (m, 1H), 2.75 (d,  $J = 14.5$  Hz, 1H), 2.72–2.69 (m, 1H), 2.59 (d,  $J = 14.5$  Hz, 1H), 2.52–2.46 (m, 2H), 2.36 (s, 3H), 2.16–2.04 (m, 1H), 1.85–1.75 (m, 1H), 1.73 (s, 3H), 1.65–1.63 (m, 1H), 1.53–1.45 (m, 1H), 1.32 (s, 3H), 1.04 (d,  $J = 6.7$  Hz, 1H), 0.95 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150MHz):  $\delta$  182.06, 146.76, 137.13, 137.11, 135.03, 131.89, 131.84, 128.74, 128.02, 126.63, 126.41, 124.90, 122.38, 122.03, 115.83, 43.97, 42.50, 38.43, 36.14, 32.80, 28.24, 27.08, 26.71, 23.54, 22.82, 22.30, 17.89; HRMS (ESI,  $[\text{M}+\text{H}]^+$ ) calculated for:  $\text{C}_{29}\text{H}_{36}\text{NO}_4\text{S}$  494.2360. Found: 494.2365.



**Chemical Formula:**  $\text{C}_{30}\text{H}_{37}\text{NO}_4\text{S}$   
**Exact Mass:** 507.24433  
**Molecular Weight:** 507.68408

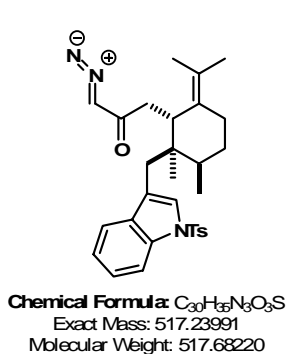
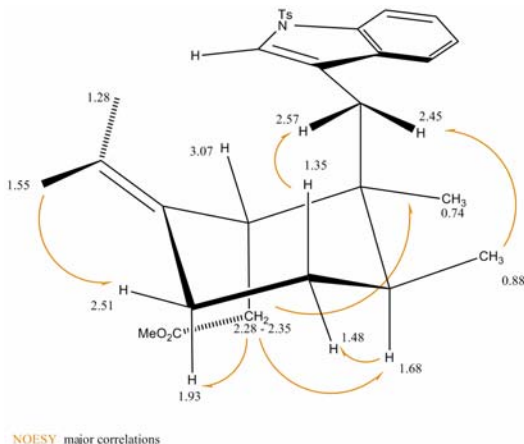
**Methyl 2,3-dimethyl-6-(propan-2-ylidene)-2-((1-tosyl-1H-indol-3-yl)methyl)cyclohexylacetate.** A solution of **18** (5.0 mg, 0.01 mmol) in MeOH (0.5 mL) was treated with  $\text{H}_2\text{SO}_4$  (15  $\mu\text{L}$ , catalytic) at  $23^\circ\text{C}$  and maintained for 16 h. The reaction mixture was poured into 10% aq. NaOH (1 mL) and extracted with  $\text{Et}_2\text{O}$  ( $3 \times 2$  mL). The combined organic extracts were washed with brine (5 mL), dried ( $\text{MgSO}_4$ ), filtered and concentrated. The crude residue was purified by preparative TLC (20% ethyl acetate – hexanes) to afford the title compound as a colorless oil (5 mg, 100% yield):  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 600 MHz):  $\delta$  7.89 (d,  $J = 8.3$  Hz, 1H), 7.69 (d,  $J = 8.3$  Hz, 2H), 7.34–7.32 (m, 2H), 7.14–1.11 (m, 3H), 3.49 (s, 3H), 3.08 (dd,  $J = 9.5, 5.9$  Hz, 1H), 2.58–2.53 (m, 3H), 2.44 (d,  $J = 14.6$  Hz, 1H), 2.35–2.29 (m, 2H), 2.25 (s, 3H), 1.94–1.89 (m, 1H), 1.71–1.66 (m, 1H), 1.57 (s, 3H), 1.49–1.47 (m, 4H), 1.36–1.27 (m, 4H), 6.80 (d,  $J = 6.8$  Hz, 3H), 0.74 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 150MHz):

$\delta$  173.72, 144.55, 135.17, 135.02, 132.99, 130.17, 129.77, 126.76, 125.53, 124.65, 124.28, 122.82, 120.37, 119.99, 113.74, 51.27, 42.05, 40.38, 36.32, 34.14, 30.77, 26.17, 25.09, 2



4.38, 21.53, 20.71, 20.26, 15.83; LRMS (ESI,  $[M+H]^+$ ) calculated for:  $C_{30}H_{38}NO_4S$  508. Found: 508.

### Structural Determination for the Methyl Ester Derivative of 18:



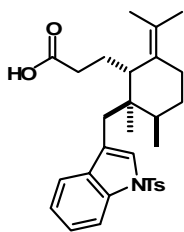
**1-diazo-3-((1S,2R,3R)-2,3-dimethyl-6-(propan-2-ylidene)-2-((1-tosyl-1H-indol-3-yl)methyl)cyclohexyl)propan-2-one.** To a solution of carboxylic acid **18** (108 mg, 0.22 mmol) in  $CH_2Cl_2$  (2 mL) at 0 °C, was added oxalyl chloride (0.55 mL, 1.1 mmol), followed by DMF (25  $\mu$ L). After stirring at 0 °C for 45 min, all solvent was removed in vacuo. The crude acid chloride was dried in vacuo (450 mtorr) for 2 h and used without any further purification.

*Note in the next step, diazomethane was dried with sodium metal for 15 min immediately prior to use. Diazomethane is toxic and explosive. Extreme caution should be exercised when preparing and handling diazomethane. All reactions should be carried out in a well-ventilated fume hood and behind a blast shield.*

The crude acid chloride was dissolved in THF (1 mL), cooled to 0 °C, and treated sequentially with *i*-Pr<sub>2</sub>EtNH (0.2 mL, 1.1 mmol) and diazomethane (6.6 mL, 2.2 mmol). The reaction mixture was warmed to 23 °C and maintained at this temperature for 30 min. The reaction mixture was poured into saturated aqueous  $NH_4Cl$  (5 mL) and extracted with  $Et_2O$  (3  $\times$  5 mL). The combined organic extracts were washed with brine (5 mL), dried ( $MgSO_4$ ), filtered, and concentrated. The crude residue was purified by flash column chromatography on silica gel (2 $\rightarrow$ 20% ethyl acetate – hexanes) to yield the title compound as a white foam (70 mg, 62%):  $^1H$  NMR ( $C_6D_6$ , 600 MHz):  $\delta$  8.54 (s,  $J$  = 8.3 Hz, 1H), 8.21 (d,  $J$  = 8.3 Hz, 2H), 8.03 (s, 1H), 7.61–7.59 (d,  $J$  = 1 Hz, 1H), 7.31 (dd,  $J$  = 7.4, 7.4 Hz, 1H), 7.11 (d,  $J$  = 8.0 Hz, 2H), 4.42 (s, 1H), 3.66–3.63 (m, 1H), 2.81–2.79 (m, 1H), 2.60 (d,  $J$  = 14.4 Hz, 1H), 2.33–2.30 (m, 1H), 2.00 (s, 3H), 1.79 (s, 3H), 1.70 (s, 3H), 1.66–1.51 (m, 3H), 1.07 (d,  $J$  = 6.4 Hz, 3H), 0.92 (s, 3H);  $^{13}C$  NMR ( $C_6D_6$ , 150MHz):  $\delta$  193.56, 144.33, 135.91, 135.74, 133.53, 130.73, 130.16, 127.22, 125.57, 124.62, 123.09, 120.28, 120.23, 114.35, 40.59, 36.70, 30.89, 26.16, 25.70, 24.61, 21.07, 21.04

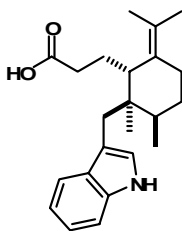


, 20.21, 15.83, 5.61; HRMS (ESI,  $[M+H]^+$ ) calculated for:  $C_{30}H_{36}N_3O_3S$  518.2471. Found: 518.2475. IR (NaCl) 2096  $cm^{-1}$ .



**Chemical Formula:**  $C_{30}H_{37}NO_4S$   
**Exact Mass:** 507.24  
**Molecular Weight:** 507.68

**3-((1S,2R,3R)-2,3-dimethyl-6-(propan-2-ylidene)-2-((1-tosyl-1H-indol-3-yl)methyl)cyclohexyl)propanoic acid.** The purified diazoketone from the preceding step is dissolved in THF (0.54 mL) and  $H_2O$  (0.05 mL). The reaction mixture is cooled to  $-25\text{ }^{\circ}C$  and protected from light. A solution of  $CF_3CO_2Ag$  (4.0 mg, 0.017 mmol) in  $Et_3N$  (60  $\mu L$ , 0.41 mmol) was added in one portion and the reaction was warmed to  $23\text{ }^{\circ}C$  over a two hour period. After stirring for 16 h, 2M HCl (1 mL) was added and the aqueous phase was extracted with  $Et_2O$  ( $3 \times 5$  mL). The combined organic extracts were washed with brine (2 mL), dried ( $MgSO_4$ ), filtered, and concentrated. The crude residue was purified by flash column chromatography on silica gel (5 $\rightarrow$ 50% ethyl acetate – hexanes) to yield the title compound as a white foam (50 mg, 74%):  $[\alpha]_D^{22} = +62^{\circ}$  ( $c = 1.1$ ,  $CHCl_3$ ).  $^1H$  NMR ( $CDCl_3$ , 600 MHz):  $\delta$  8.00 (d,  $J = 8.3$  Hz, 1H), 7.72 (d,  $J = 8.3$  Hz, 2H), 7.38 (d,  $J = 7.7$  Hz, 1H), 7.30–7.29 (m, 2H), 7.22–7.18 (m, 3H), 2.65 (d,  $J = 14.5$  Hz, 1H), 2.58–2.55 (m, 1H), 2.47 (d,  $J = 14.5$  Hz, 1H), 2.42–2.39 (m, 1H), 2.32 (s, 3H), 2.18–1.94 (m, 2H), 1.87–1.73 (m, 3H), 1.71–1.65 (m, 1H), 1.63 (s, 3H), 1.55–1.53 (m, 1H), 1.43–1.38 (m, 1H), 1.06 (s, 3H), 0.94 (d,  $J = 6.7$  Hz, 3H);  $^{13}C$  NMR ( $CDCl_3$ , 150MHz):  $\delta$  193.56<sup>4</sup>, 144.72, 135.24, 135.11, 133.15, 130.08, 129.66, 125.75, 124.36, 124.17, 122.85, 121.05, 120.06, 113.80, 44.04, 40.57, 36.17, 31.96, 30.89, 26.62, 25.23, 24.75, 22.69, 21.46, 20.79, 20.06, 15.84; HRMS (ESI,  $[M+Na]^+$ ) calculated for:  $C_{30}H_{37}NO_4SNa$  530.2335. Found: 530.2333. IR (NaCl) 1705  $cm^{-1}$ .



**Chemical Formula:**  $C_{23}H_{31}NO_2$   
**Exact Mass:** 353.24  
**Molecular Weight:** 353.50

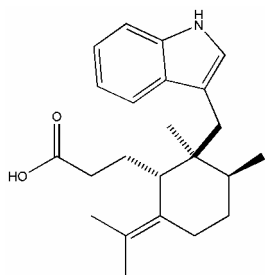
**Suaveolindole (1).** A 1.0 M stock solution of sodium naphthalenide was prepared by added Na (0.23 g, 10 mmol) to a solution of naphthalene (1.3 g, 10 mmol) in DME (10 mL) at  $23\text{ }^{\circ}C$ . After stirring for 90 m, the freshly prepared sodium naphthalenide (1.0 mL, 1.0 mmol, 1.0 M in DME) was transferred to a solution of the tosyl protected indole (52 mg, 0.10 mmol) in DME (1 mL) at  $23\text{ }^{\circ}C$ . The reaction mixture was maintained at this temperature for 20 min, poured into 2M HCl (1 mL) and extracted with  $Et_2O$  ( $3 \times 2$  mL). The combined organic extracts were washed with brine (5 mL), dried ( $MgSO_4$ ), filtered, and concentrated. The crude residue was purified by HPLC (2% IPA – hexanes;  $4.6 \times 250$  mm column, Silica  $5\mu M$ ;  $R_T = 6.6$  min) to afford suaveolindole (**1**, 33 mg, 94%) as a white foam:  $[\alpha]_D^{22} = +32^{\circ}$  ( $c = 1.0$ ,  $CD_3OD$ ).  $^1H$  NMR ( $CD_3OD$ , 600 MHz): 10.10 (bs, 0.4H), 7.43 (d,  $J = 7.9$  Hz, 1H), 7.28 (d,  $J = 8.1$  Hz, 1H), 7.02 (dd,  $J = 7.7, 7.3$  Hz, 1H), 6.99 (s, 1H), 6.94 (dd,  $J = 7.9, 7.1$  Hz, 1H), 2.72 (d,  $J = 14.5$ , 1H), 2.68–2.61 (m, 3H), 2.08–1.85 (m, 4 H), 1.80–1.72 (m, 2H), 1.70 (s, 3H), 1.56–1.46 (m, 2 H), 1.35 (s, 3H), 1.00 (m, 6H);  $^{13}C$  NMR ( $CD_3OD$ , 150 MHz):  $\delta$  178.22, 137.70, 132.35, 130.78, 126.23, 124.33, 121.80, 120.13, 119.16, 113.27, 111.92

<sup>4</sup> Visible when LB = 10

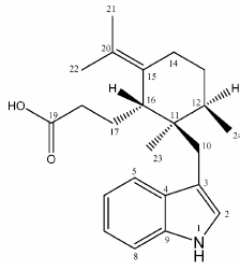


, 45.57, 41.86, 37.75, 33.11, 32.31, 27.89, 26.09, 25.91, 24.0, 21.67, 20.33, 16.39; HRMS (ESI,  $[M+H]^+$ ) calculated for:  $C_{23}H_{32}NO_2$  354.2427. Found: 354.2427.



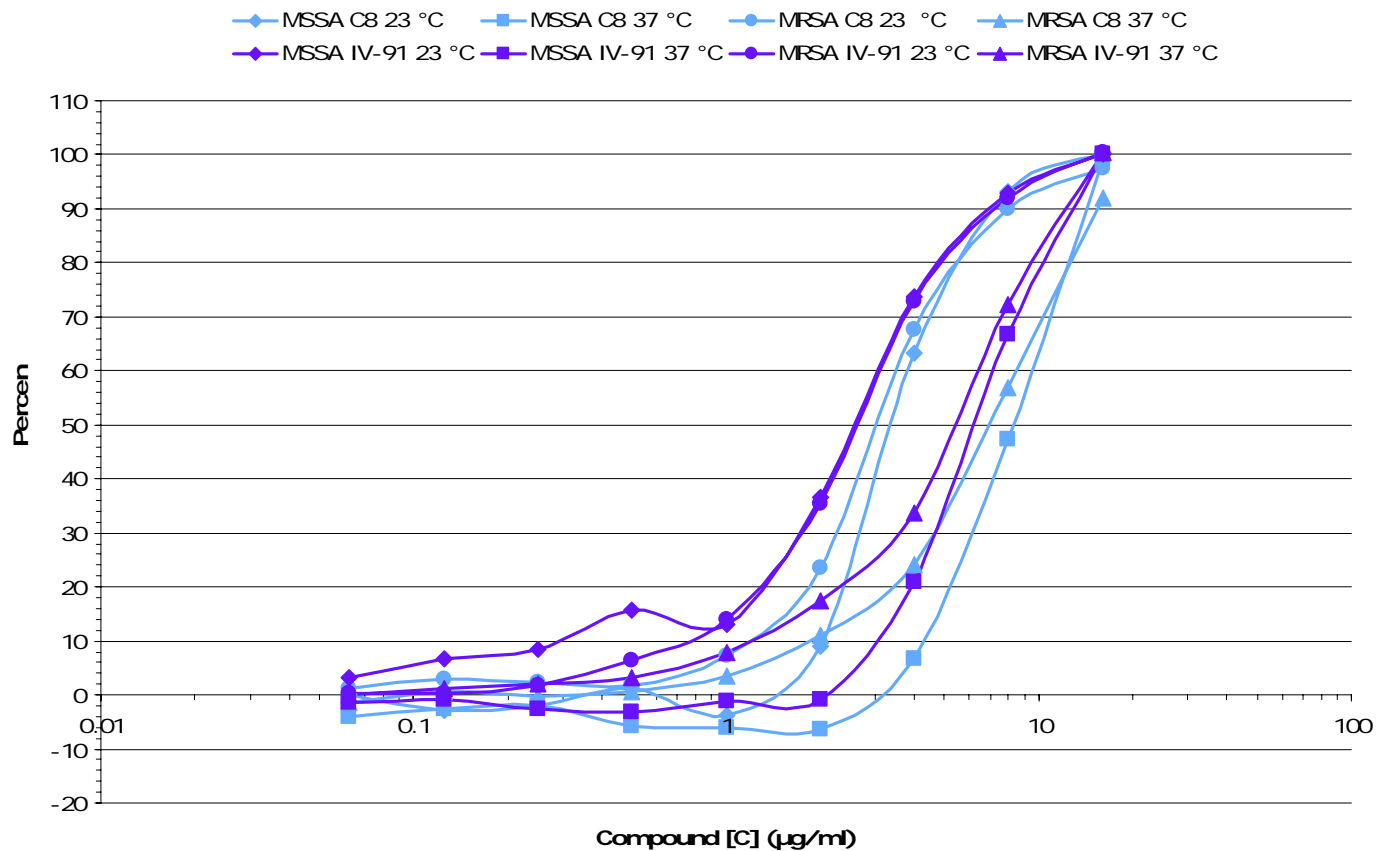


**EJV-IV-91**  
(synthetic)

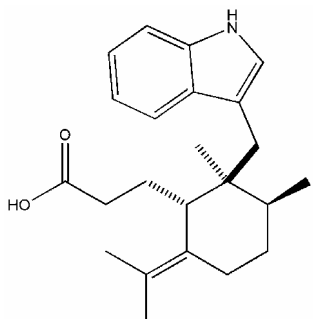


**C8 – (natural product)**

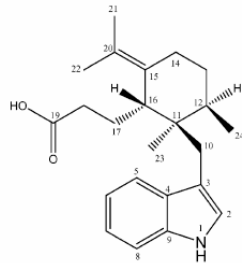
**C8 and C8 precursors MIC on *Staphylococcus aureus* (Suave4)**







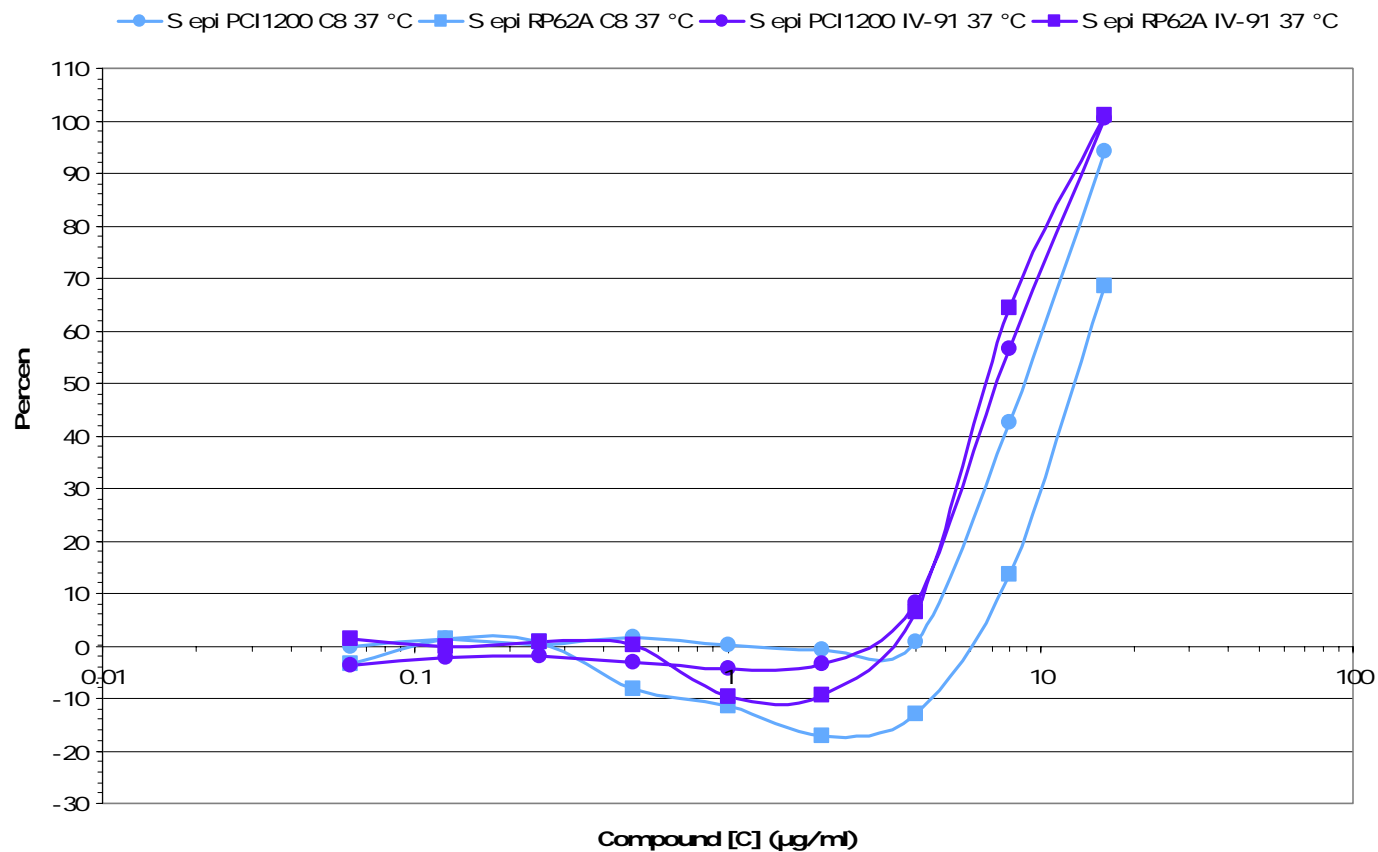
**EJV-IV-91**  
(synthetic)



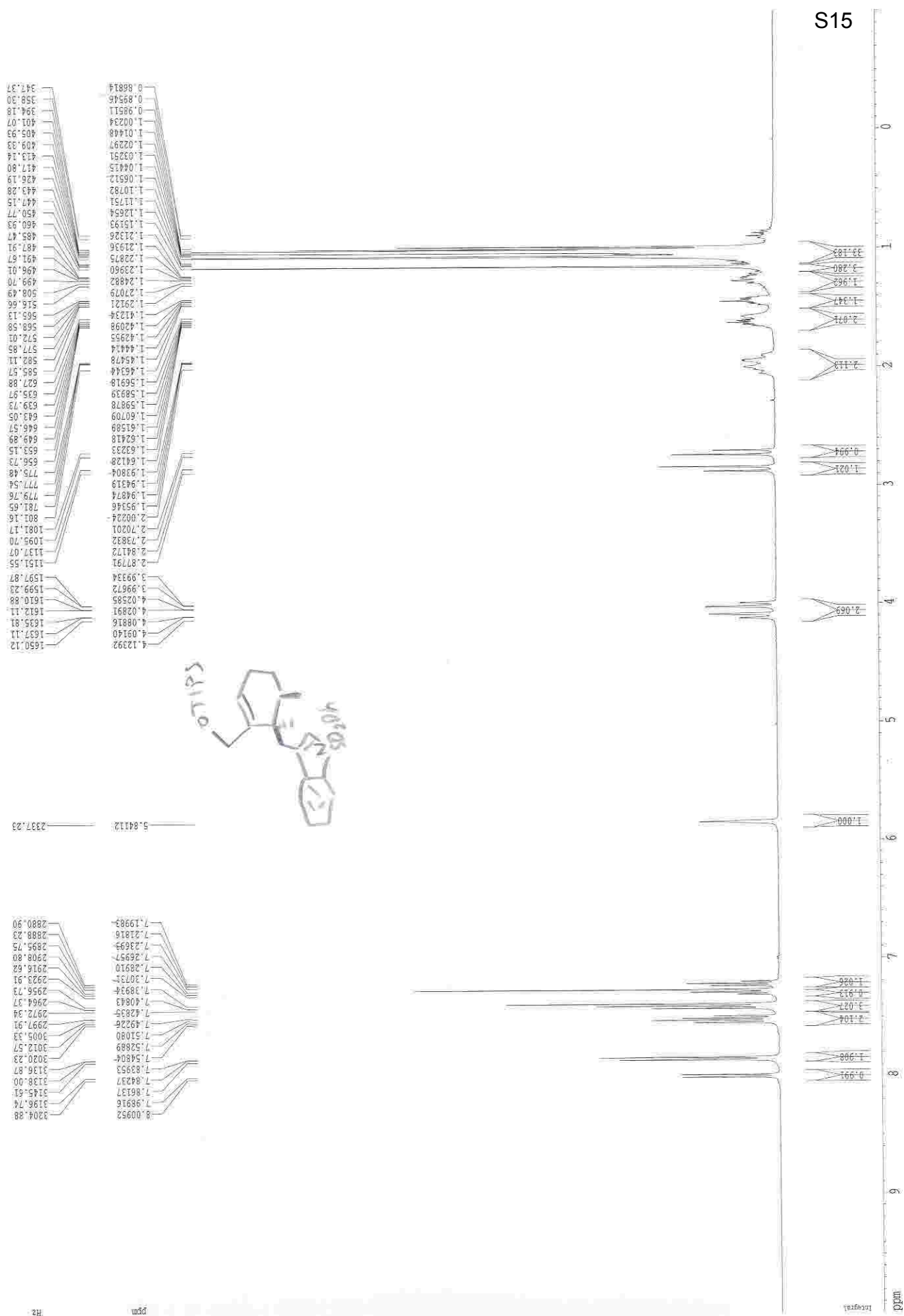
**C8 – (natural product)**

S **Suave4** 14

**C8 and C8 precursors MIC on *Staphylococcus epidermidis* (Suave4)**







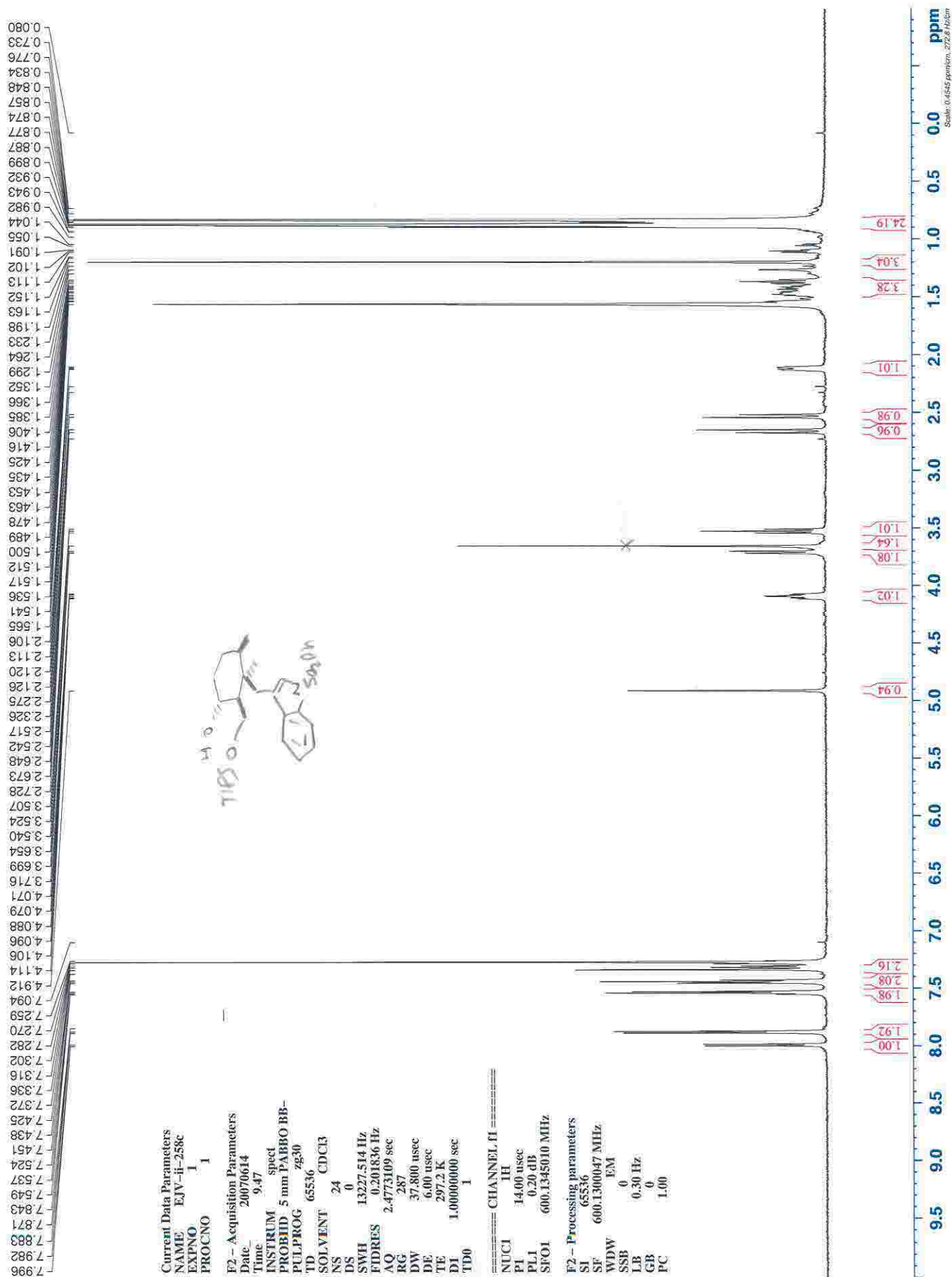


17807.08	141.60
17414.00	138.47
16950.93	134.79
16796.23	133.56
16635.67	132.28
16235.77	129.10
15927.05	126.05
15651.45	124.46
15643.53	124.39
15535.91	123.54
15466.64	122.99
15196.59	120.84
15107.31	120.13
14282.85	113.57
9720.03	77.29
9656.15	76.78
8109.19	64.48
5045.40	40.12
4808.97	38.24
3743.49	29.77
3507.30	26.30
3228.70	25.67
3004.15	23.89
2275.59	18.09
2228.34	17.72
2031.22	16.15
1545.51	12.29
1504.39	11.96

S16









11.31  
16.46  
17.65  
17.71  
23.37  
25.10  
28.71  
35.58  
40.43  
43.97  
56.49  
65.77  
70.58  
73.19  
76.80  
77.01  
77.22  
77.49

113.71  
119.86  
120.31  
123.37  
124.63  
124.66  
126.71  
129.22  
131.93  
133.69  
134.73  
138.36

# Current Data Parameters

NAME EJ4-ii-258c  
EXPNO 2  
PROCNO 1

## F2 - Acquisition Parameters

Date\_ 20070614  
Time 9.56  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 134144  
SOLVENT CDCl3  
NS 3000  
DS 0  
SWH 39062.500 Hz  
FIDRES 0.291198 Hz  
AQ 1.7170932 sec  
RG 2050  
DW 12.800 usec  
DE 6.00 usec  
TE 297.2 K  
D1 1.10000002 sec  
d11 0.03000000 sec  
DELTA 1.00000000 sec  
TD0 1

## CHANNEL f1

NUC1 13C  
P1 9.50 usec  
PL1 2.00 dB  
SFO1 150.9194083 MHz

## CHANNEL f2

CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL2 0.20 dB  
SFO2 600.1327006 MHz

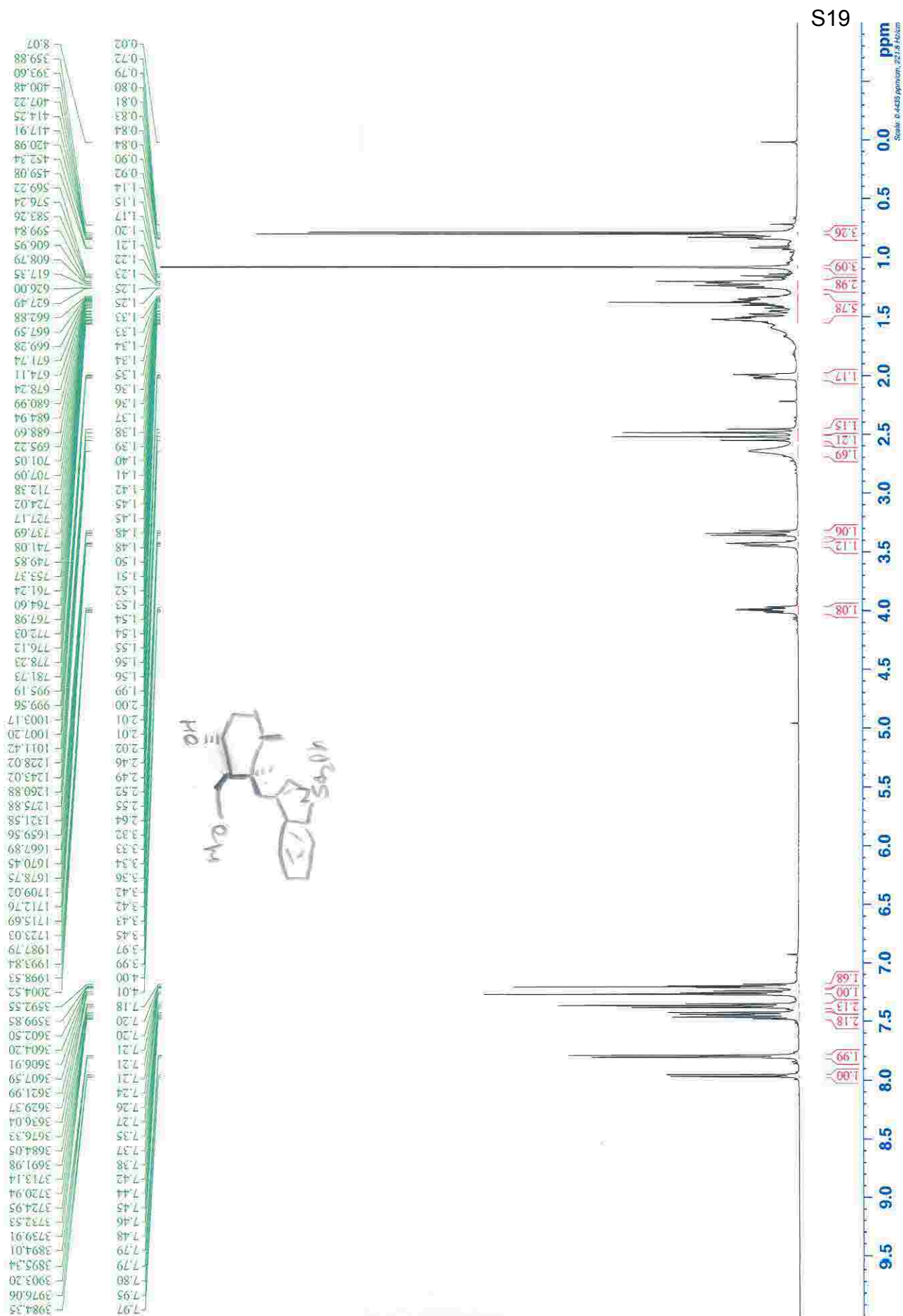
## F2 - Processing parameters

SI 131072  
SF 150.9028090 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Scale: 10.7 ppm/cm, 161.4 Hz/cm



Proton24  
. \* evelthui ejv-ii-154\_2D (1 1) CDCI3 24.0C September\_07,2006\_18:56 DRX 500MHz zg30 1H \*



16.49  
23.57  
24.95  
28.98  
29.70  
36.42  
41.07  
43.85  
57.91  
63.95  
73.55  
76.81  
77.02  
77.23

113.93  
119.86  
120.77  
123.34  
124.69  
124.98  
126.62  
128.34  
129.24  
132.16  
133.77  
134.95  
138.21

Current Data Parameters  
NAME EJV-ii-154b  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20070613  
Time 11.11

INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30

TD 134144  
SOLVENT CDCl3

NS 821  
DS 0

SWH 39062.500 Hz  
FIDRES 0.291195 Hz

RG 1.7170932 sec  
AQ 2050

DW 12.800 usec  
DE 6.00 usec

TE 297.2 K  
D1 1.10000002 sec

d11 0.030000000 sec  
DELTA 1.000000000 sec

TD0 1  
=====

CHANNEL f1  
NUC1 13C  
P1 9.50 usec  
PL1 2.00 dB  
SFO1 150.9194053 MHz

===== CHANNEL f2 =====  
CTDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL2 0.20 dB  
SFO2 600.1327006 MHz

F2 - Processing parameters  
SI 131072

SF 150.9028090 MHz

WDW EM

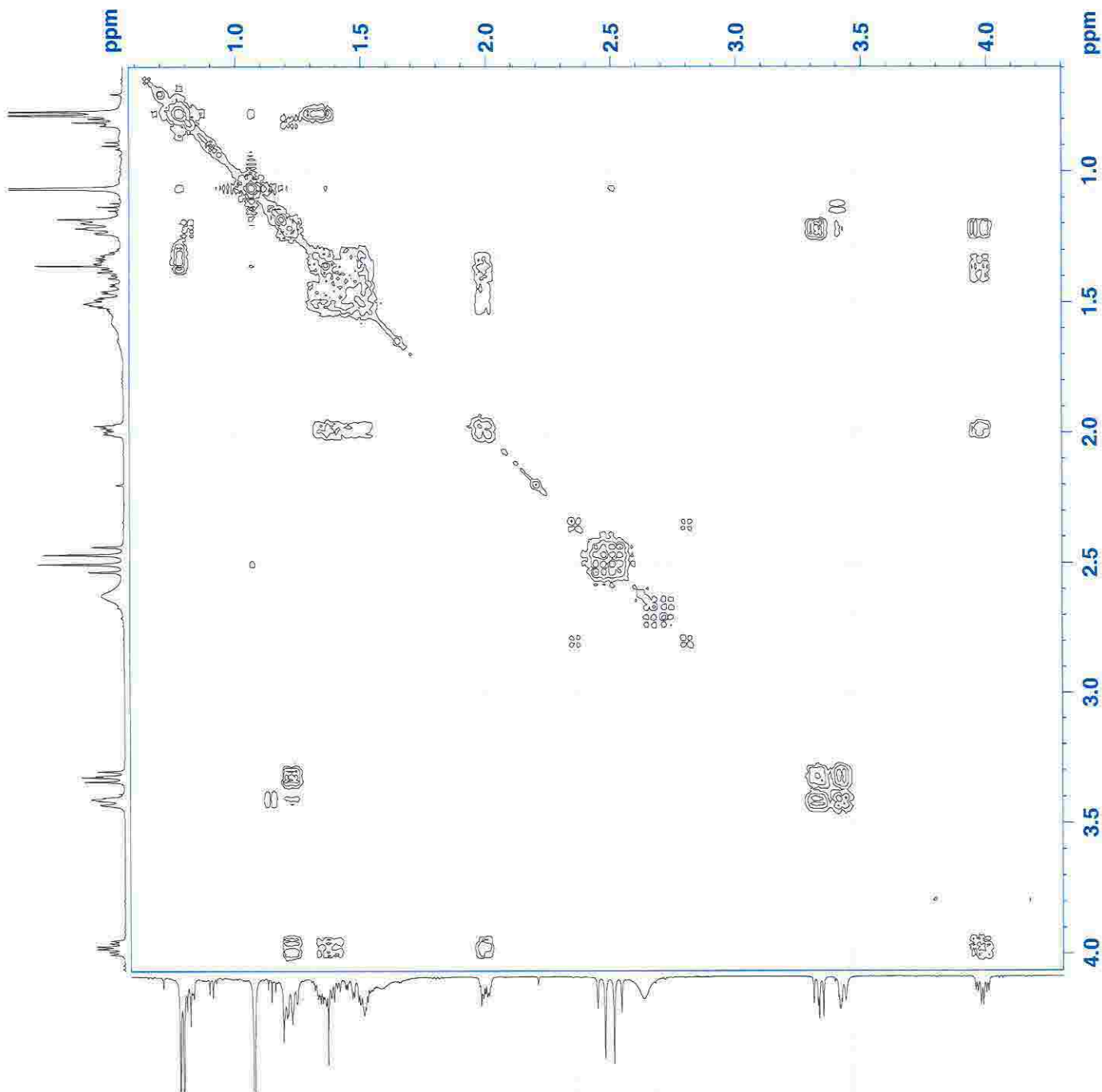
SSB 0

LB 1.00 Hz

GB 0

PC 0





Current Data Parameters  
NAME ejv-ii-154\_2D  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20060907  
Time 18:58  
INSTRUM spect  
PROBHD 5 mm QNP 1H/13  
PULPROG cosygpc  
TD 65536  
SOLVENT CDCl3  
NS 2  
DS 16  
SWH 4066.410 Hz  
FIDRES 1.956255 Hz  
AQ 0.2556404 sec  
RG 161.3  
DW 124.800 usec  
DE 6.00 usec  
TE 297.2 K  
D1 0.0000300 sec  
d13 0.83841843 sec  
d16 0.00000400 sec  
DL6 0.0002000 sec  
INQ 0.00024960 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 14.00 usec  
PL2 -1.00 dB  
SFO1 500.1322286 MHz

===== GRADIENT CHANNEL =====  
GPNAM1 SINE.100  
GPNAM2 SINE.100  
GPZ1 10.00 %  
GPZ2 10.00 %  
PL6 1000.00 usec

F1 - Acquisition parameters  
ND0 1  
TD 256  
SFO1 500.1322286 MHz  
FIDRES 15.690043 Hz  
SWH 8.011 kHz  
PULPROG QF

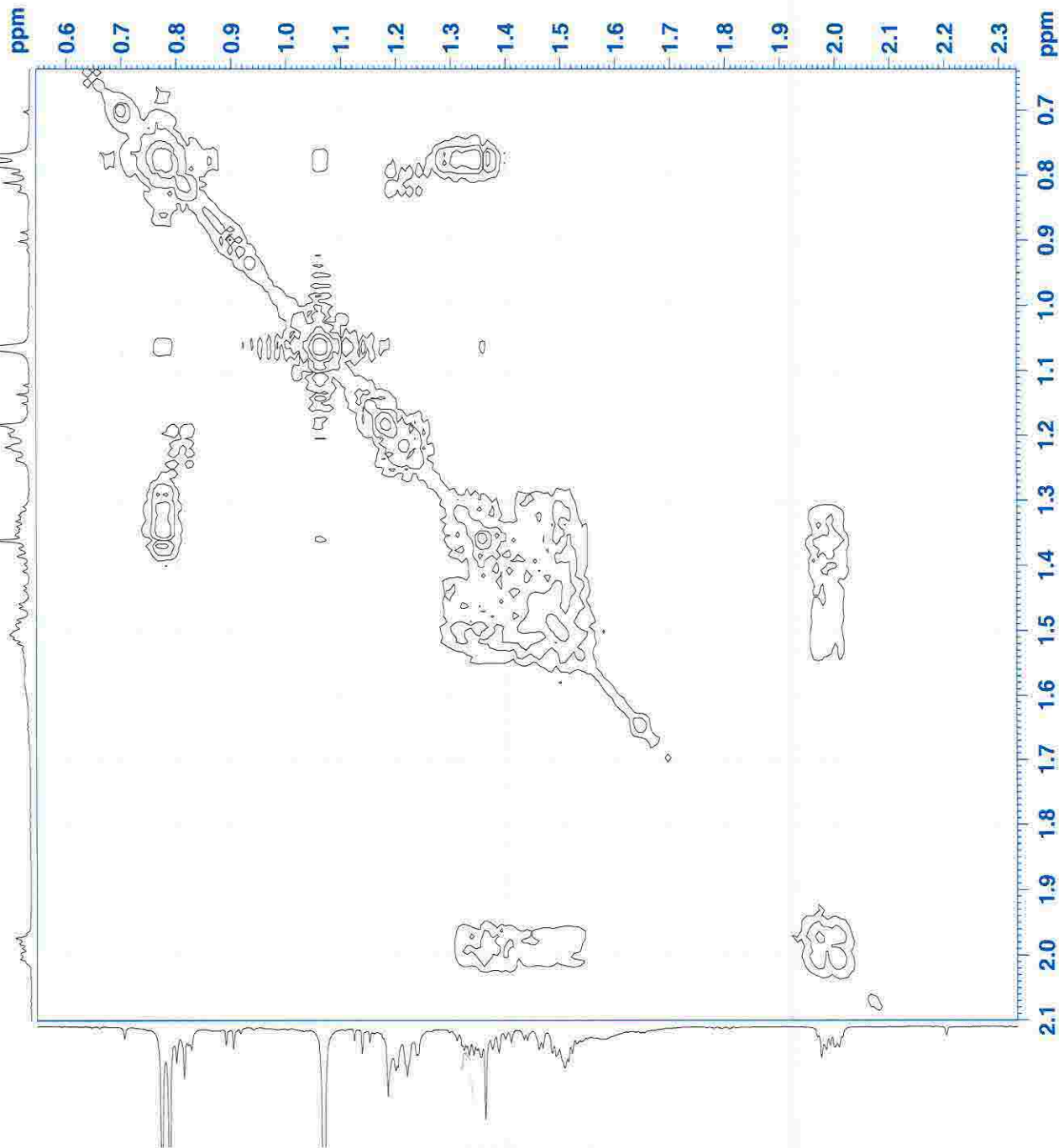
F2 - Processing parameters  
SI 1024  
SF 500.1300483 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

F1 - Processing parameters  
SI 1024  
SF 500.1300483 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0





NMR Analytical Core Facility  
Rockefeller Research Laboratories  
430 E 57th St  
New York, NY 10021



Current Data Parameters  
NAME ejv-ii-154\_2D  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20060907  
Time 18:58  
INSTRUM spect  
PROBHD 5 mm QNP 1H/13  
PULPROG cosygpqf  
TD 2048  
SOLVENT CDCI3  
NS 16  
DS 16  
SWH 4006.410 Hz  
FIDRES 1.956255 Hz  
AQ 0.2556404 sec  
RG 161.3  
DW 124.800 usec  
DE 6.00 usec  
TE 297.2 K  
d0 0.0000300 sec  
d13 0.89841843 sec  
d16 0.00000400 sec  
d16 0.00020000 sec  
IND 0.00024960 sec

CHANNEL f1  
NUC1 1H  
P1 14.00 usec  
PL1 14.00 dB  
P2 1.00 dB  
SF01 500.1322286 MHz

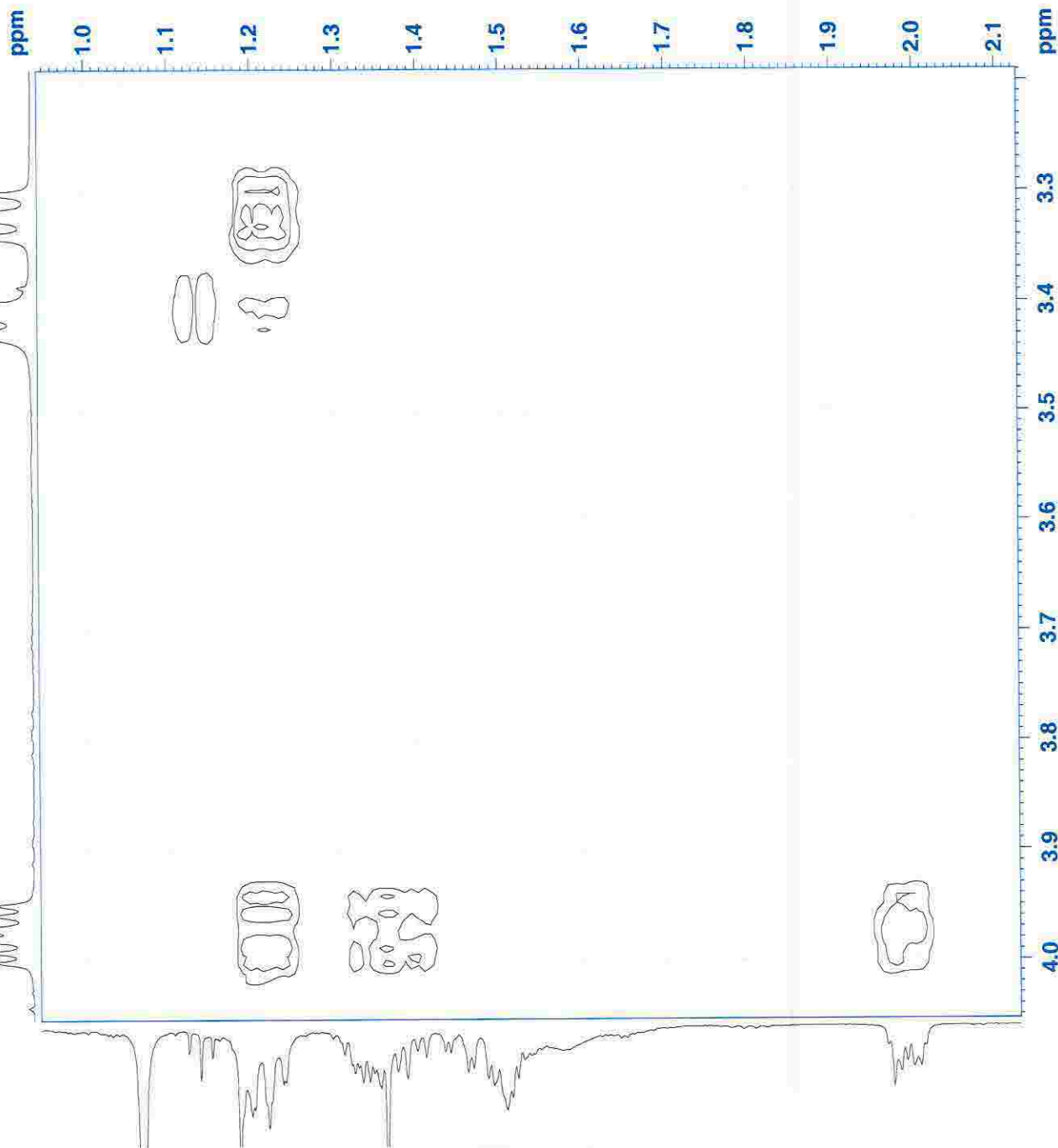
GRADIENT CHANNEL =====  
GPNAM1 SINE.100  
GPNAM2 SINE.100  
GPZ1 10.00 %  
GPZ2 10.00 %  
P16 1000.00 usec

F1 - Acquisition parameters  
ND0 1  
TD 256  
SF01 500.1322 MHz  
FIDRES 15.680040 Hz  
SW 8.011 ppm  
FREQDE QF

F2 - Processing parameters:  
SI 1024  
SF 500.1300483 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00

F1 - Processing parameters:  
SI 1024  
SF 500.1300483 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.00





Current Data Parameters  
 NAME 65v-ii-154\_2D  
 EXPNO 2  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20060907  
 Time 18.58  
 INSTRUM spect  
 PROBD 5 mm QNP 1H/13  
 PULPROG cosyg2d  
 TO 2046  
 SOLVENT CDCl3  
 NS 2  
 DS 16  
 SWH 4006.410 Hz  
 FIDRES 1.956255 Hz  
 AQ 0.2556404 sec  
 RG 161.3  
 DW 124.800 usec  
 DE 6.00 usec  
 TE 297.2 K  
 D1 0.00000300 sec  
 D11 0.89841843 sec  
 D16 0.00000400 sec  
 D17 0.00020000 sec  
 INO 0.00024960 sec

===== CHANNEL F1 =====  
 NUC1 1H  
 P1 14.00 usec  
 PL1 14.00 usec  
 PL2 -1.00 dB  
 SFO1 500.1322286 MHz

===== GRADIENT CHANNEL =====  
 GPNAM1 SINE.100  
 GPNAM2 SINE.100  
 GPZ1 10.00 %  
 GPZ2 10.00 %  
 P16 1000.00 usec

F1 - Acquisition parameters  
 NDQ 1  
 TD 256  
 FFO1 500.1322 MHz  
 FIDRES 15.65004 Hz  
 SW 8.011 ppm  
 SHF00 QF

F2 - Processing parameters  
 SI 1024  
 SF 500.1300483 MHz  
 WMW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0  
 FC 1.00

F1 - Processing parameters  
 SI 1024  
 SF 500.1300483 MHz  
 WMW SINE  
 SSB 0  
 LB 0.00 Hz  
 GB 0





Current Data Parameters  
NAME ejv-ii-154\_2D  
EXPNO 3  
PROCNO 1

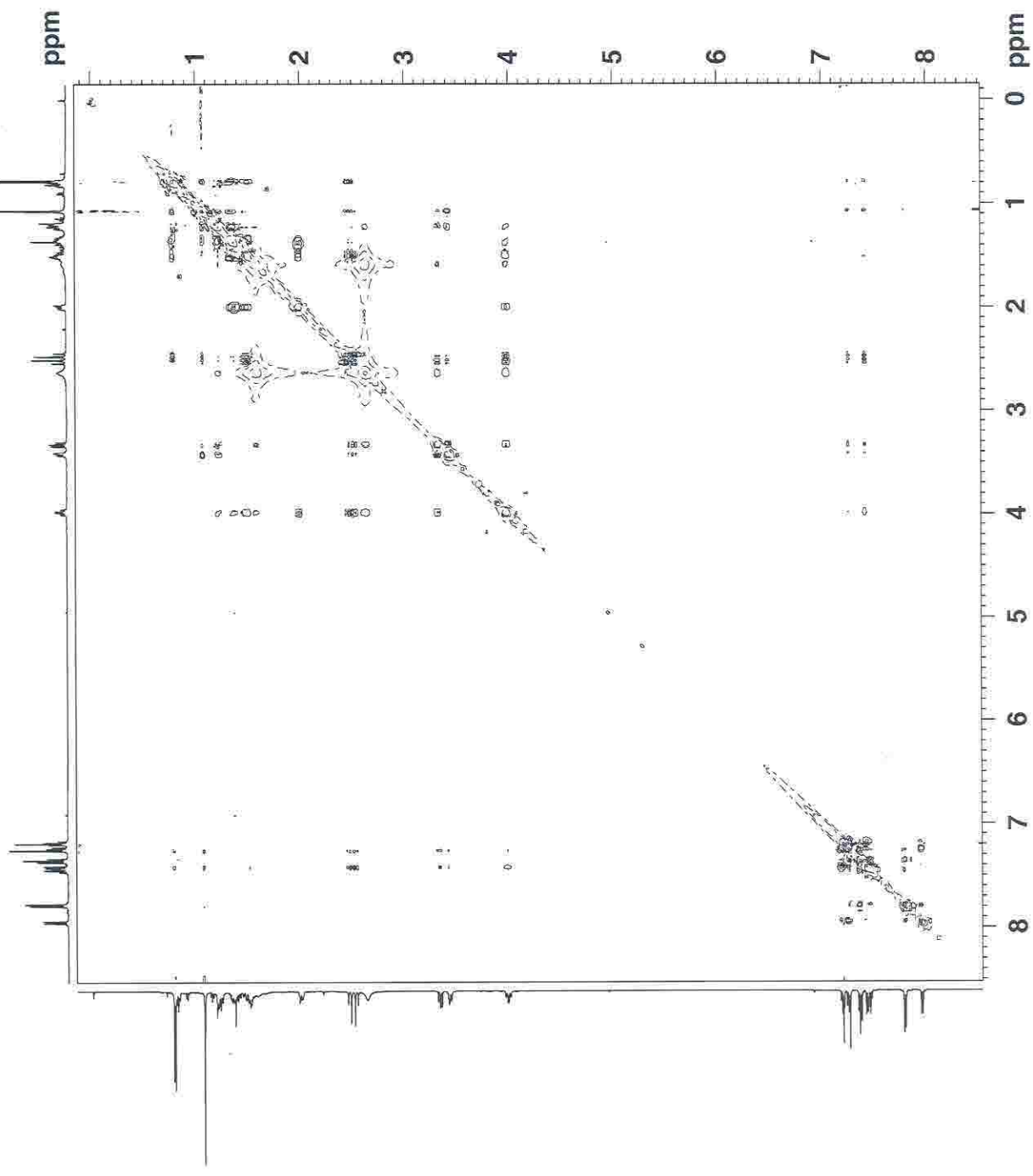
F2 - Acquisition Parameters  
Date\_ 20060907  
Time 19.11  
INSTRUM spect  
PROBHD 5 mm QNP 1H/13  
PULPROG noesyph  
TD 2048  
SOLVENT CDC13  
NS 64  
DS 4  
SWH 4340.278 Hz  
FIDRES 2.119276 Hz  
AQ 0.2359796 sec  
RG 143.7  
DW 115.200 usec  
DE 6.00 usec  
TE 297.2 K  
d0 0.0009737 sec  
D1 1.96436405 sec  
D8 0.44999999 sec  
IN0 0.00023040 sec  
ST1CNT 128

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 -1.00 dB  
SFO1 500.1321423 MHz

F1 - Acquisition parameters  
ND0 1  
TD 256  
SFO1 500.1321 MHz  
FIDRES 16.954210 Hz  
SW 8.678 ppm  
FMODE States-tpPI

F2 - Processing parameters  
SI 1024  
SF 500.1300483 MHz  
WDW QSINE  
SSB 2  
LB 0.00 Hz  
GB 0  
PC 1.00

F1 - Processing parameters  
SI 1024  
MC2 States-tpPI  
SF 500.1300483 MHz  
WDW QSINE  
SSB 2  
LB 0.00 Hz  
GB 0







Current Data Parameters  
 NAME ejv-ii-154\_2D  
 EXPNO 3  
 PROCNO 1

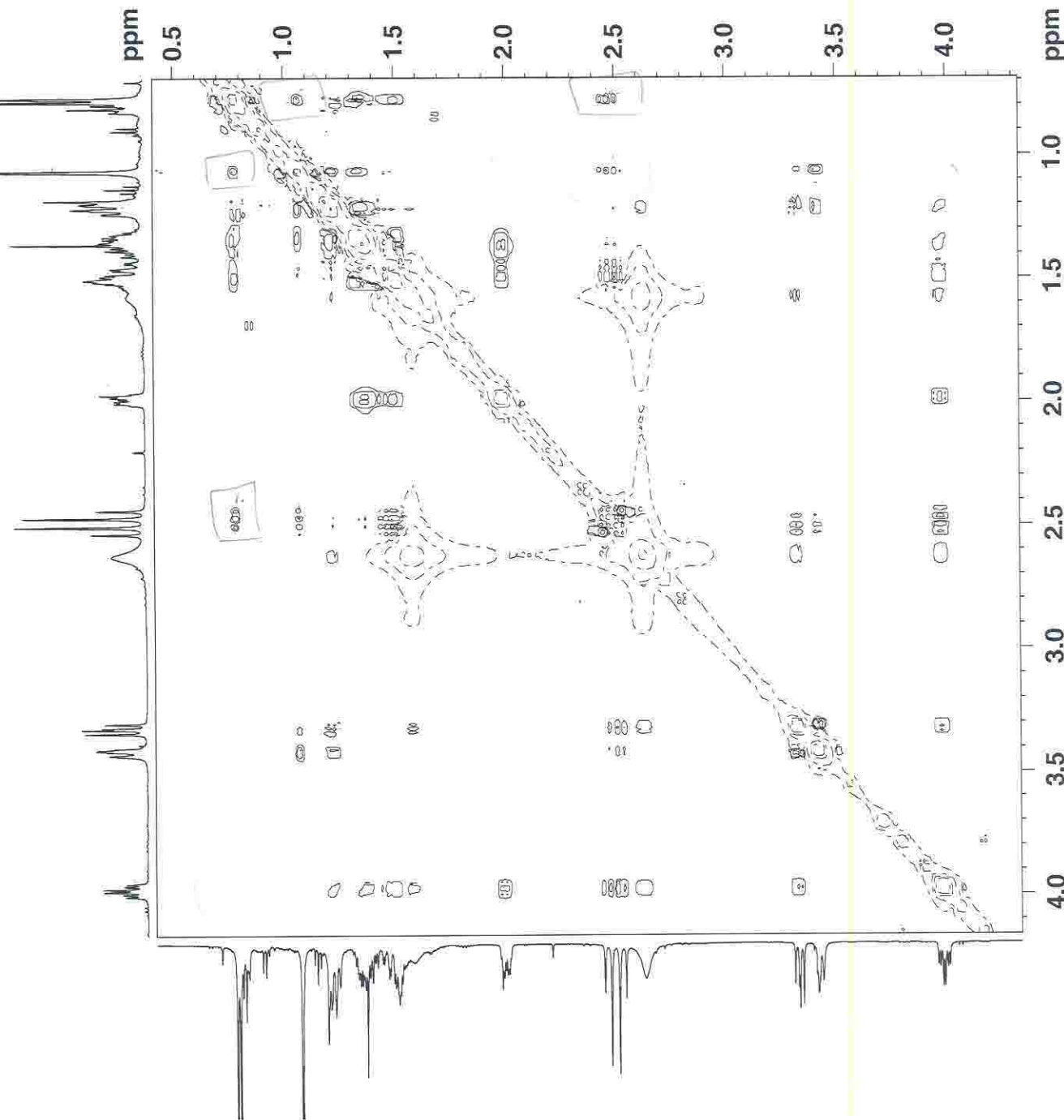
F2 - Acquisition Parameters  
 Date\_ 20060907  
 Time 19.11  
 INSTRUM spect  
 PROHD 5 mm QNP 1H/13  
 PULPROG noesyph  
 TD 2048  
 SOLVENT CDC13  
 NS 64  
 DS 4  
 SWH 4340.278 Hz  
 FIDRES 2.119276 Hz  
 AQ 0.2359796 sec  
 RG 143.7  
 DW 115.200 usec  
 DE 6.00 usec  
 TE 297.2 K  
 d0 0.0009737 sec  
 d1 1.96436405 sec  
 d8 0.44999999 sec  
 IN0 0.00023040 sec  
 STICNT 128

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 14.00 usec  
 PL1 -1.00 dB  
 SFO1 500.1321423 MHz

F1 - Acquisition parameters  
 ND0 1  
 TD 256  
 SFO1 500.1321 MHz  
 FIDRES 16.954210 Hz  
 SW 8.678 ppm  
 FMODE States-TPPI

F2 - Processing parameters  
 SI 1024  
 SF 500.1300483 MHz  
 WDW QSI  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 MC2 States-TPPI  
 SF 500.1300483 MHz  
 WDW QSI  
 SSB 2  
 LB 0.00 Hz  
 GB 0







Current Data Parameters  
 NAME ejv-ii-154\_2D  
 EXPNO 3  
 PROCNO 1

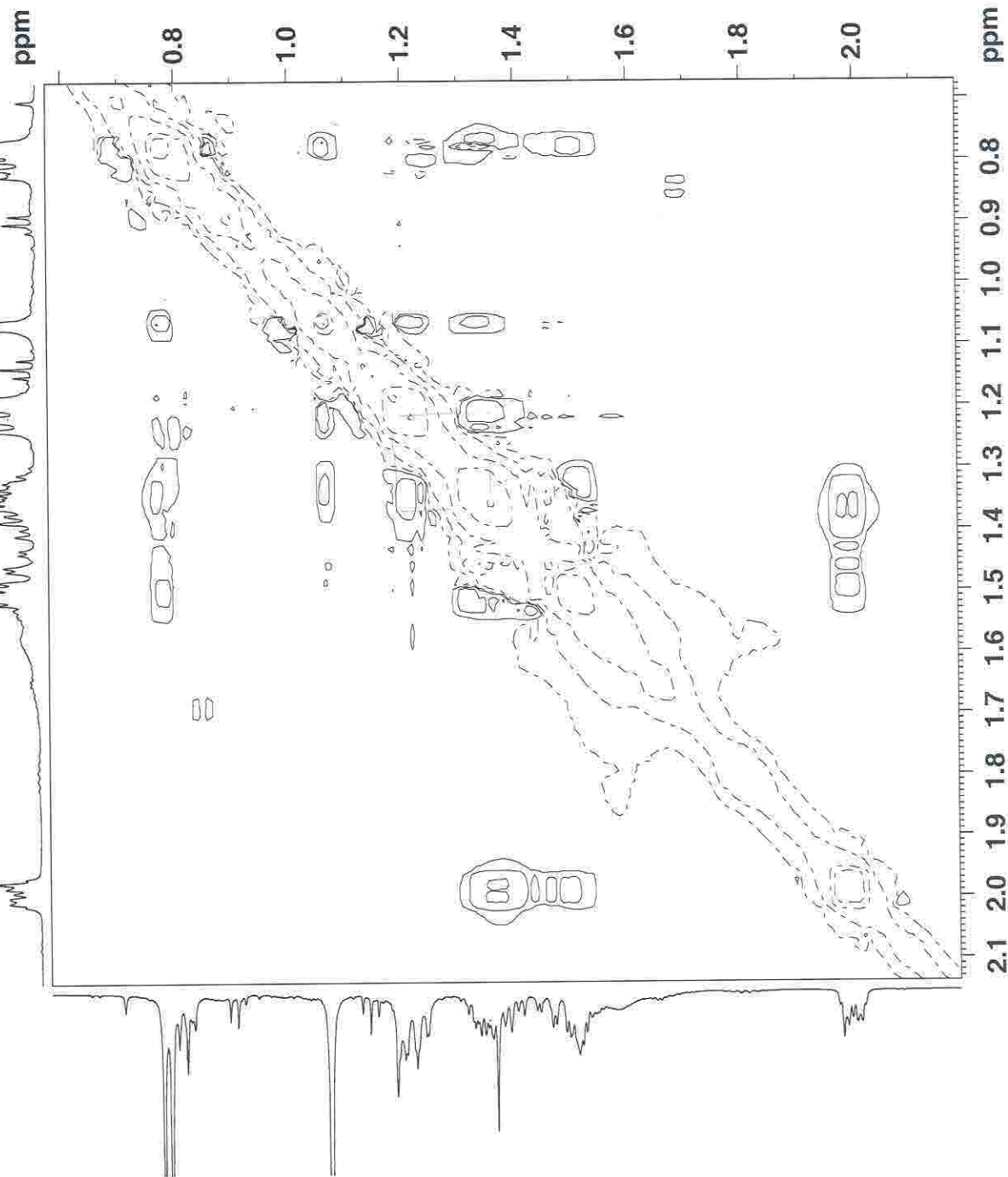
F2 - Acquisition Parameters  
 Date\_ 20060907  
 Time 19.11  
 INSTRUM spect  
 PROBHD 5 mm QNP 1H/13  
 PULPROG noesyph  
 TD 2048  
 SOLVENT CDCl3  
 NS 64  
 DS 4  
 SWH 4340.278 Hz  
 FIDRES 2.119276 Hz  
 AQ 0.2359796 sec  
 RG 143.7  
 DW 115.200 usec  
 DE 6.00 usec  
 TE 297.2 K  
 d0 0.0009737 sec  
 d1 1.96436405 sec  
 D8 0.44999999 sec  
 IN0 0.00023040 sec  
 STICNT 128

===== CHANNEL f1 =====  
 NUC1 1H  
 P1 14.00 usec  
 PL1 -1.00 dB  
 SFO1 500.1321423 MHz

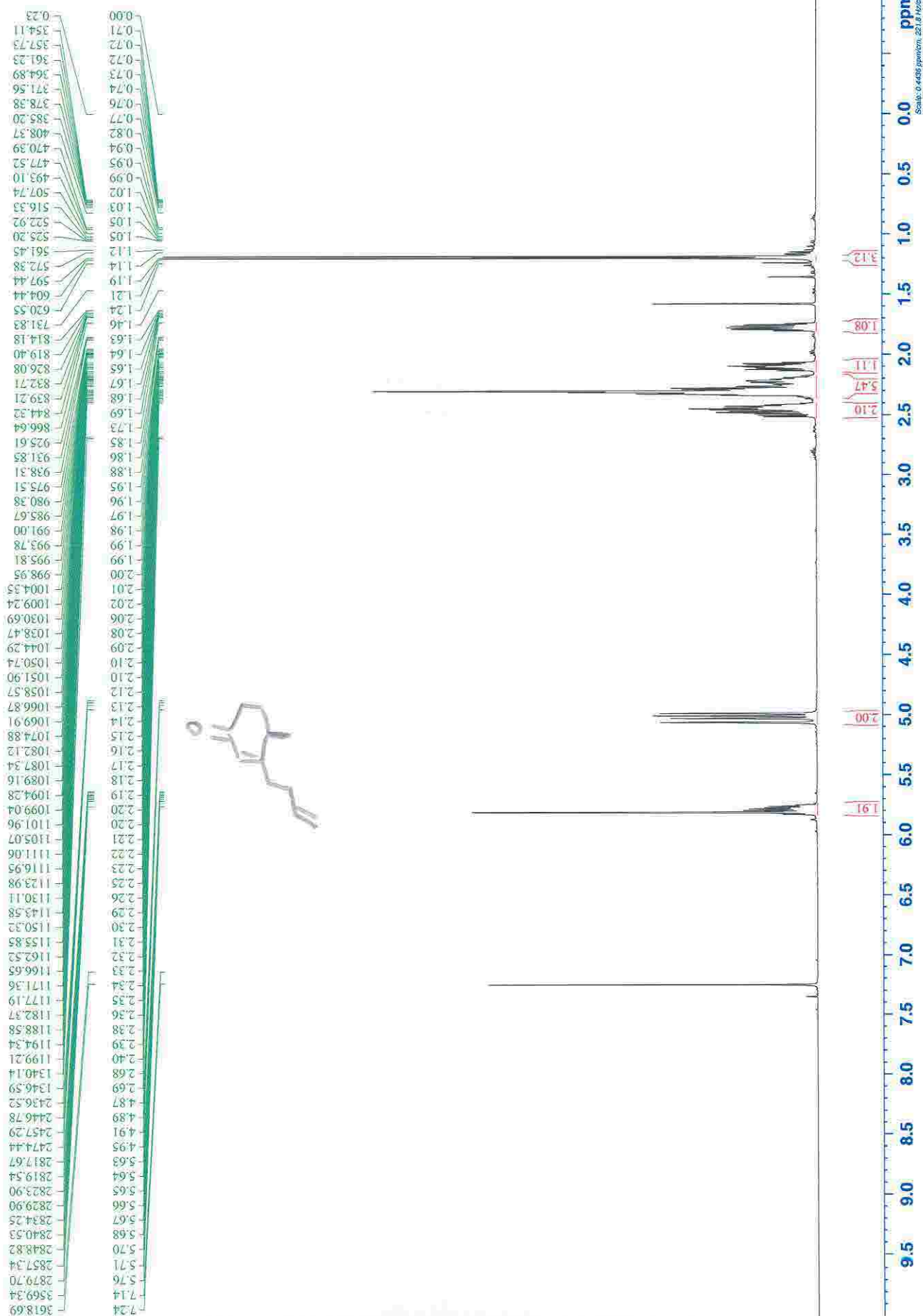
F1 - Acquisition parameters  
 ND0 1  
 TD 256  
 SFO1 500.1321 MHz  
 FIDRES 16.954210 Hz  
 SW 8.678 ppm  
 FMODE States-TPPI

F2 - Processing parameters  
 SI 1024  
 SF 500.1300483 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0  
 PC 1.00

F1 - Processing parameters  
 SI 1024  
 MC2 States-TPPI  
 SF 500.1300483 MHz  
 WDW QSINE  
 SSB 2  
 LB 0.00 Hz  
 GB 0

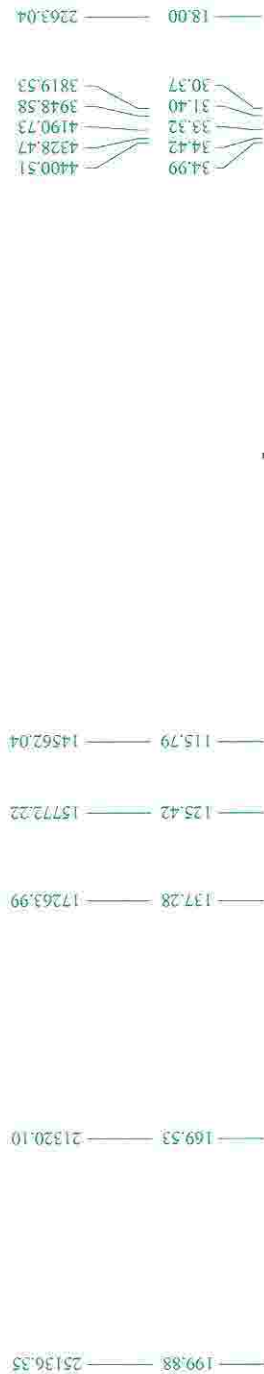








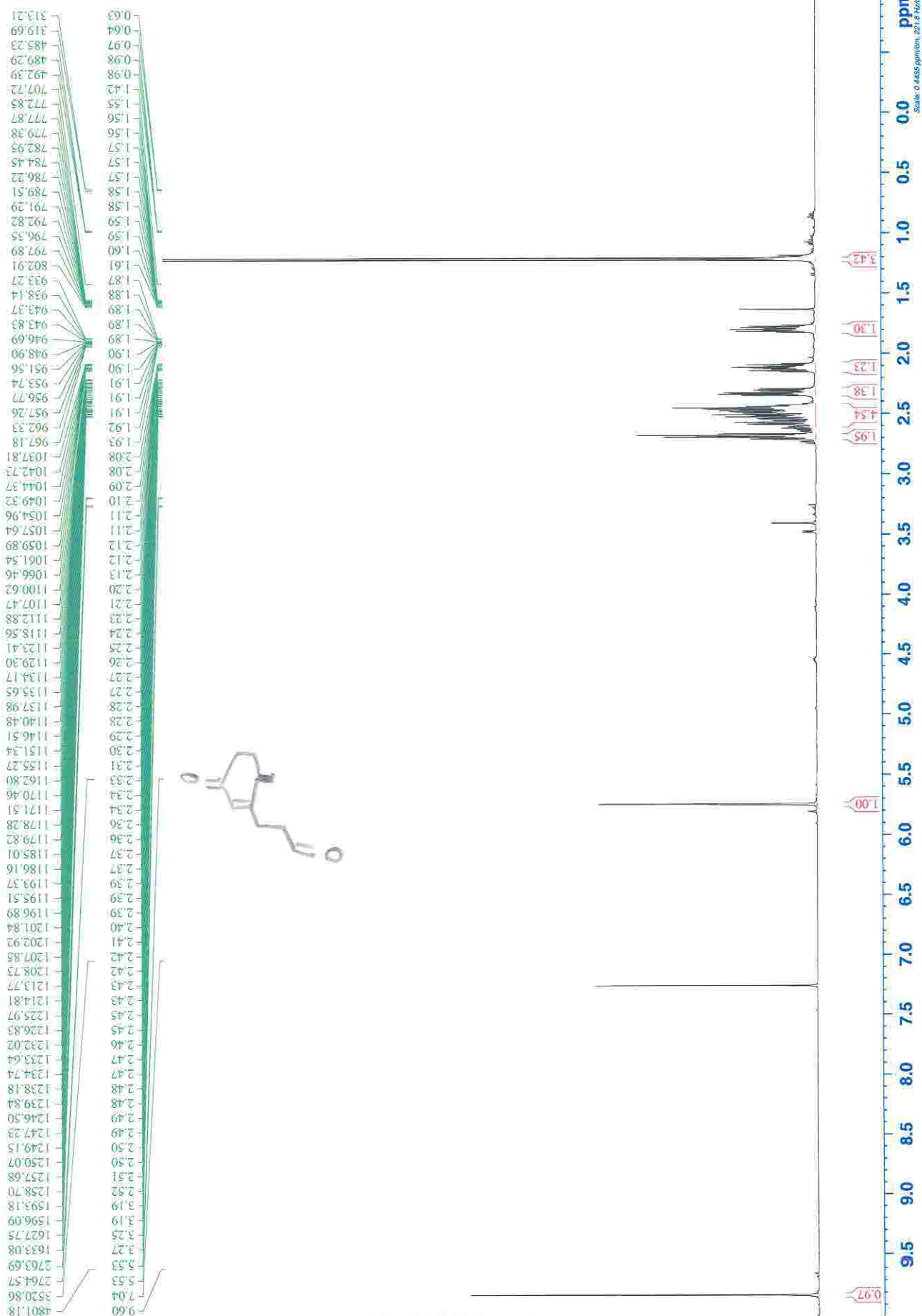
Carbon \* evelthui ejv-iii-240\_1H (1 1) CDCl3 24.0C February\_23,2007\_15:55 DRX 500MHz zgpg30 13C; 1H O2=4.000 \*



S28









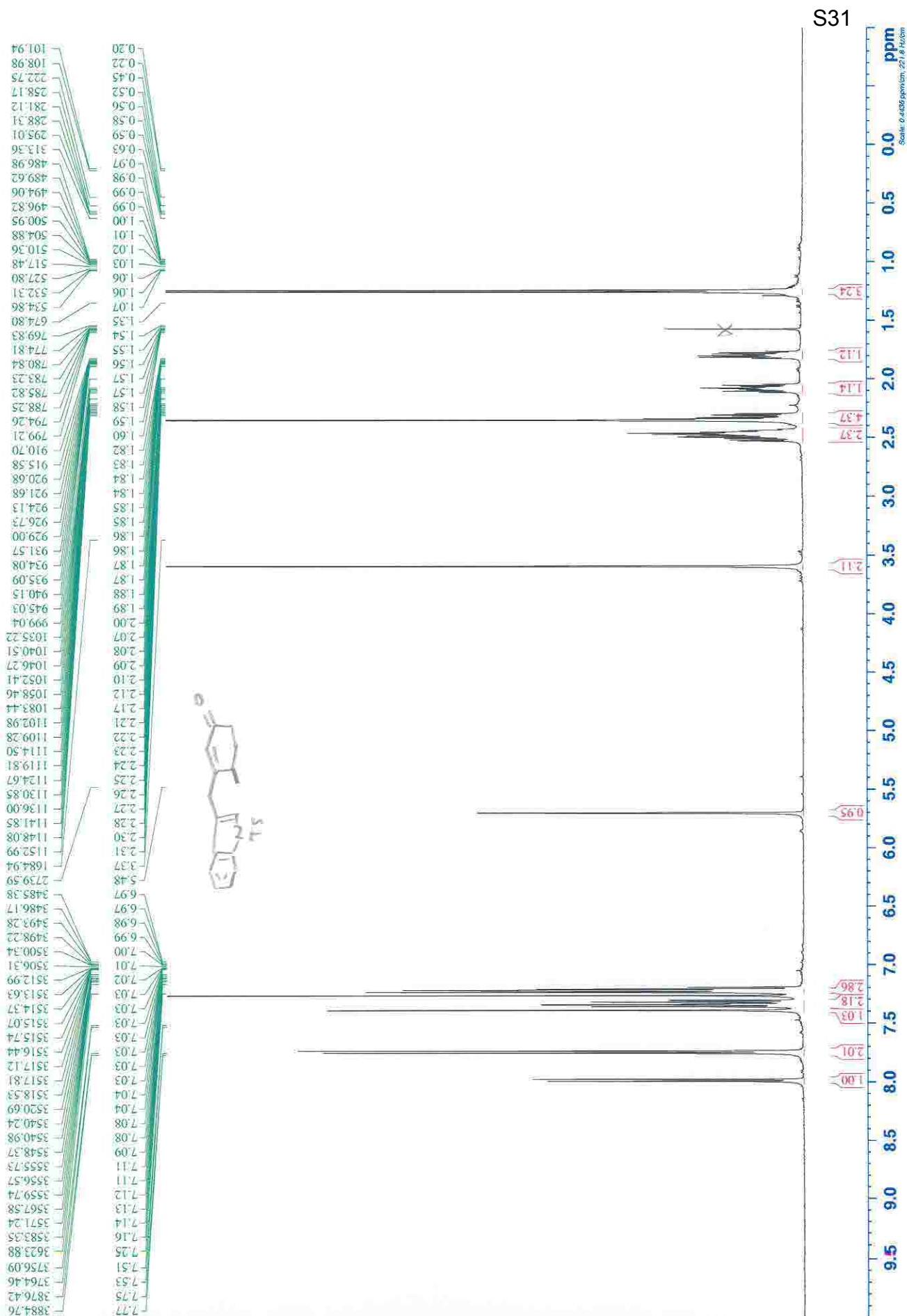
Carbon \* evelthui ejv-iii-222\_c13 (1 1) CDCl3 24.0C February\_14,2007\_10:27 DRX 500MHz zgpg30 13C; 1H O2=4.000 \*



S30

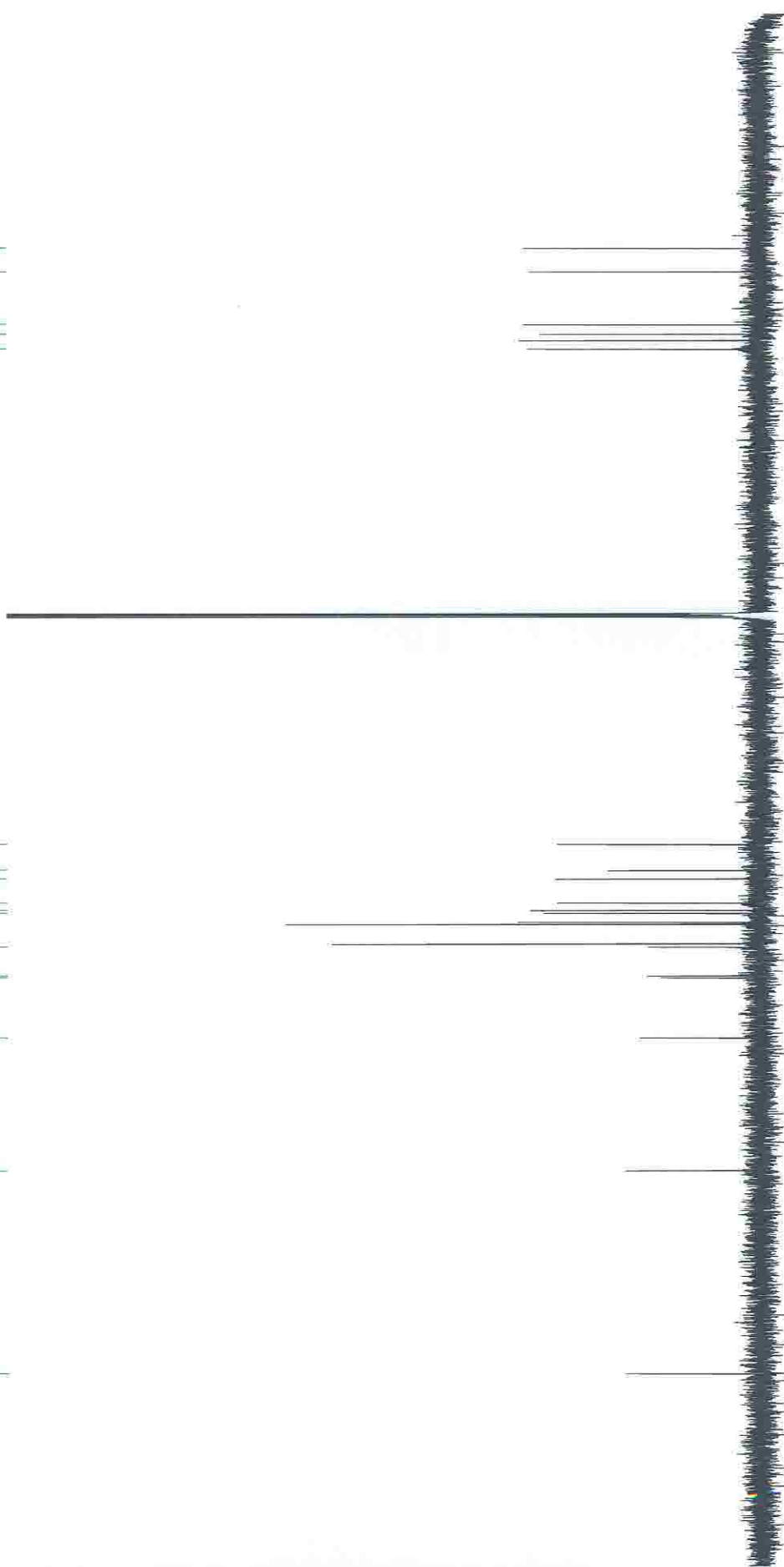
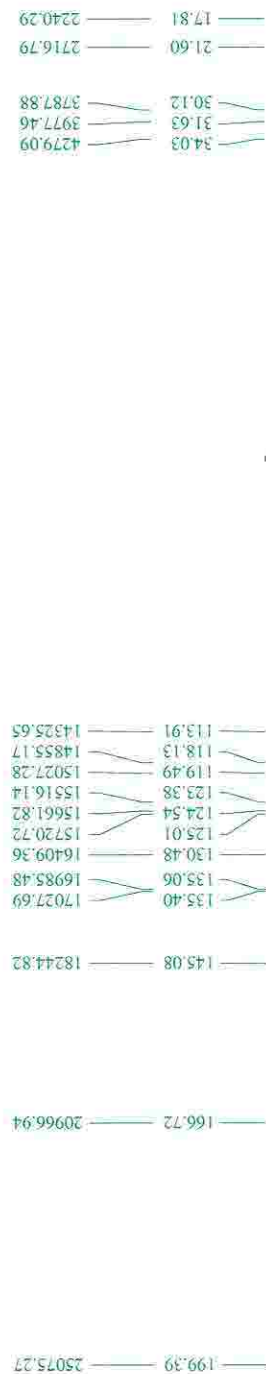








Carbon \* evelthui ejv-iii-228\_13C (1 1) CDCl3 24.0C February\_19,2007\_07:54 DRX 500MHz zgpg30 13C; 1H O2=4.000 \*



S32











NMR Analytical Core Facility  
Memorial Sloan-Kettering Cancer Center  
1275 York Ave.  
Box 357  
New York, NY 10021

## Current Data Parameters

EXPTNO 573v-30-f

PROCNO 1

P2-Acquisition Parameters

Date 20070416

Time 12:25

INSTRUM spect

PROBHD 5 mm TAIBO BB-

PULPROG zgpg30

TD 134144

SOLVENT CDCl3

NS 202

DS 0

SWH 30062.500 Hz

FIDRES 0.24588 Hz

AQ 1.716932 sec

RG 2050

DW 12.800 nsec

TE 297.2 K

D1 1.1000002 sec

d11 0.0300000 sec

DELTA 1.0000000 sec

TD 1

===== CHANNEL f1 =====

NUC1 13C

P1 9.50 nsec

PL1 2.00 dB

SFO1 150.914483 MHz

===== CHANNEL f2 =====

CTDPRG2 waltz16

NUC2 1H

P2 8.00 nsec

PL2 15.00 dB

PL13 15.00 dB

PL2 0.20 dB

SFO2 400.132706 MHz

P2-Processing parameters

SI 131072

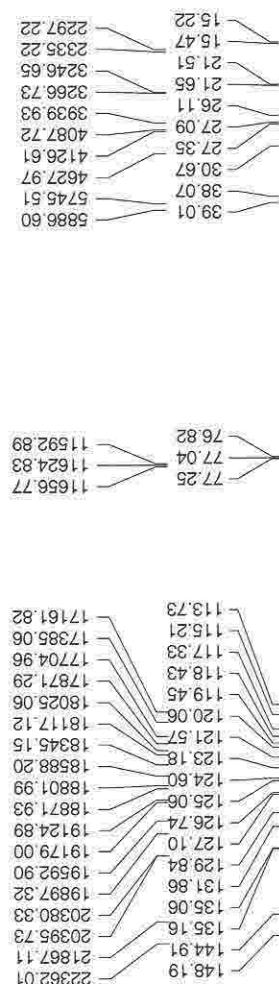
WDW EM

SSB 0

LB 1.00 Hz

GB 0

PC 1.40

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm  
Scale: 11.11 pps/cm, 107.7 Hz/cm



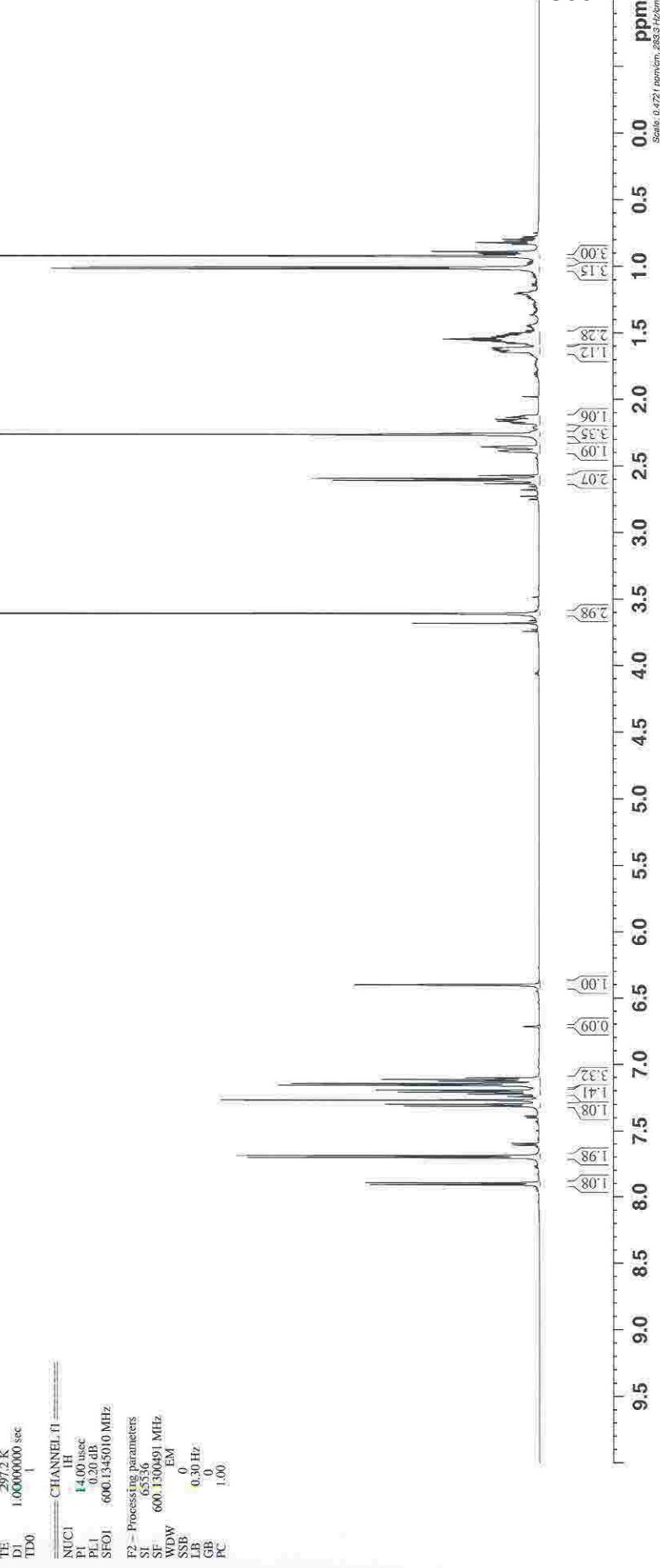
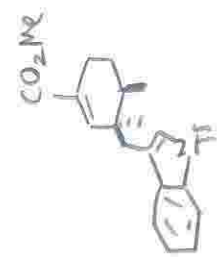
Proton \* evelthui ejv-iv-17-1H (1 1) CDCl3 24.0C March\_31\_2007\_13:48 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm \*

MEMORIAL SLOAN-KETTERING CANCER CENTER  
ESTABLISHED 1884  
NMR Analytical Core Facility  
Noninvasive Cancer Diagnostics  
470 E. 67th St.  
New York, NY 10021

4795.75  
4803.88  
4811.99  
4820.10  
4828.21  
4836.32  
4844.43  
4852.54  
4860.65  
4868.76  
4876.87  
4884.98  
4893.09  
4901.20  
4909.31  
4917.42  
4925.53  
4933.64  
4941.75  
4949.86  
4957.97  
4966.08  
4974.19  
4982.30  
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4998.52  
5006.63  
5014.74  
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5047.18  
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6425.87  
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6466.42  
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6506.97  
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6896.25  
6904.36  
6912.47  
6920.58  
6928.69  
6936.80  
6944.91  
6953.02  
6961.13  
6969.24  
6977.35  
6985.46  
6993.57  
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7009.79  
7017.90  
7026.01  
7034.12  
7042.23  
7050.34  
7058.45  
7066.56  
7074.67  
7082.78  
7090.89  
7099.00  
7107.11  
7115.22  
7123.33  
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7147.66  
7155.77  
7163.88  
7171.99  
7180.10  
7188.21  
7196.32  
7204.43  
7212.54  
7220.65  
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7236.87  
7244.98  
7253.09  
7261.20  
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7277.42  
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7301.75  
7309.86  
7317.97  
7326.08  
7334.19  
7342.30  
7350.41  
7358.52  
7366.63  
7374.74  
7382.85  
7390.96  
7399.07  
7407.18  
7415.29  
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7439.62  
7447.73  
7455.84  
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7504.50  
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7877.56  
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7910.00  
7918.11  
7926.22  
7934.33  
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7950.55  
7958.66  
7966.77  
7974.88  
7982.99  
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8429.04  
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8485.81  
8493.92  
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8542.58  
8550.69  
8558.80  
8566.91  
8575.02  
8583.13  
8591.24  
8599.35  
8607.46  
8615.57  
8623.68  
8631.79  
8639.90  
8648.01  
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8680.45  
8688.56  
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8761.55  
8769.66  
8777.77  
8785.88  
8793.99  
8802.10  
8810.21  
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8826.43  
8834.54  
8842.65  
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8858.87  
8866.98  
8875.09  
8883.20  
8891.31  
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8948.08  
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8980.52  
8988.63  
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9029.18  
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9061.62  
9069.73  
9077.84  
9085.95  
9094.06  
9102.17  
9110.28  
9118.39  
9126.50  
9134.61  
9142.72  
9150.83  
9158.94  
9167.05  
9175.16  
9183.27  
9191.38  
9199.49  
9207.60  
9215.71  
9223.82  
9231.93  
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9377.91  
9386.02  
9394.13  
9402.24  
9410.35  
9418.46  
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9450.90  
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9467.12  
9475.23  
9483.34  
9491.45  
9499.56  
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9523.89  
9532.00  
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9596.88  
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9629.32  
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9710.42  
9718.53  
9726.64  
9734.75  
9742.86  
9750.97  
9759.08  
9767.19  
9775.30  
9783.41  
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9799.63  
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9815.85  
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9864.51  
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9921.28  
9929.39  
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9945.61  
9953.72  
9961.83  
9969.94  
9978.05  
9986.16  
9994.27  
10002.38  
10010.49  
10018.60  
10026.71  
10034.82  
10042.93  
10051.04  
10059.15  
10067.26  
10075.37  
10083.48  
10091.59  
10099.70  
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10148.36  
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10213.24  
10221.35  
10229.46  
10237.57  
10245.68  
10253.79  
10261.90  
10270.01  
10278.12  
10286.23  
10294.34  
10302.45  
10310.56  
10318.67  
10326.78  
10334.89  
10343.00  
10351.11  
10359.22  
10367.33  
10375.44  
10383.55  
10391.66  
10400.00

Current Data Parameters  
NAME ejv-iv-17-1H  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20070331  
Time 13.50  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 24  
DS 0  
SWH 13227.514 Hz  
FIDRES 0.201836 Hz  
AQ 2.4773109 sec  
RG 71.8  
DW 57.800 usec  
DE 6.00 usec  
TE 297.2 K  
D 1.00000000 sec  
TD0 1

Channel f1  
NUC1 1H  
P1 14.00 usec  
PL1 0.20 dB  
SFO1 600.1345010 MHz  
F2 - Processing parameters  
SI 65536  
SF 600.130491 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



S35

ppm  
Scale: 0.471 ppm/cm, 265.3 Hz/cm



Carbon. \* evelthui eiv-iv-17-13C (1 1) CDCl3 24.0C March 31,2007 13:51 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm.



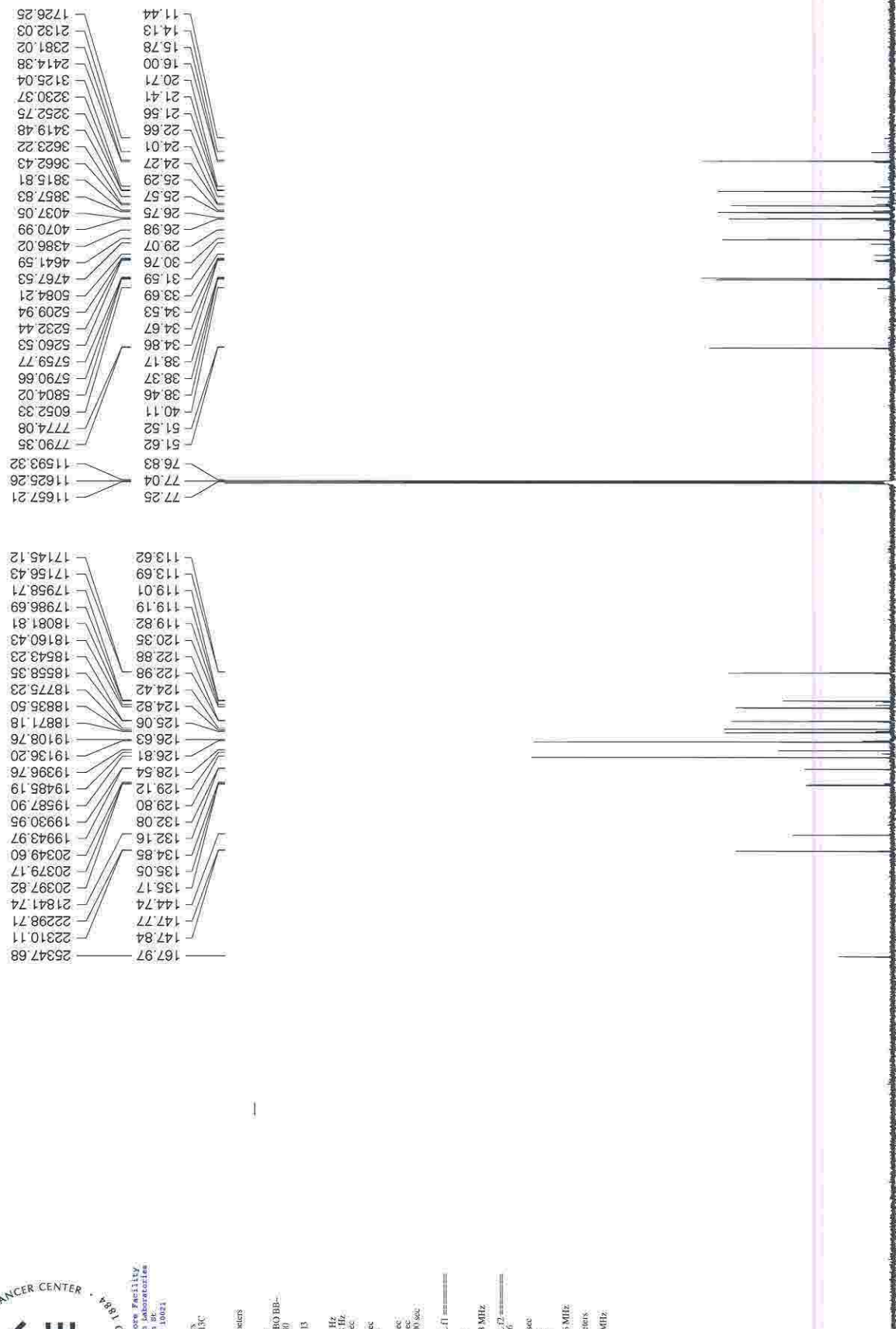
NMR Analytical Core Facility  
Rockefeller Research Laboratories  
430 E 67th St  
New York, NY 10021

Current Data Parameters		F2 - Acquisition Parameters	
NAME	Value	NAME	Value
EXPNO	9	INSTRUM	3 mm PABBO BB-
PROCNO	1	PULPROG	zgpg30
		TD	13444
		DO	4095
		DS	0
		SWH	39963.500 Hz
		FIDRES	1.7709198 Hz
		AQ	0.7019952 sec
		RG	327.680 usec
		RG2	12.880 usec
		DE	6.00 usec
		TE	297.2 K
		D1	1.1000000 sec
		d11	0.0500000 sec
		DELTA	1.0000000 sec

```
===== CHANNEL f1 =====
NUC1 13C
P1 9.50 usec
PL1 2.00 dB
SEQ1 150.9194083 MHz
```

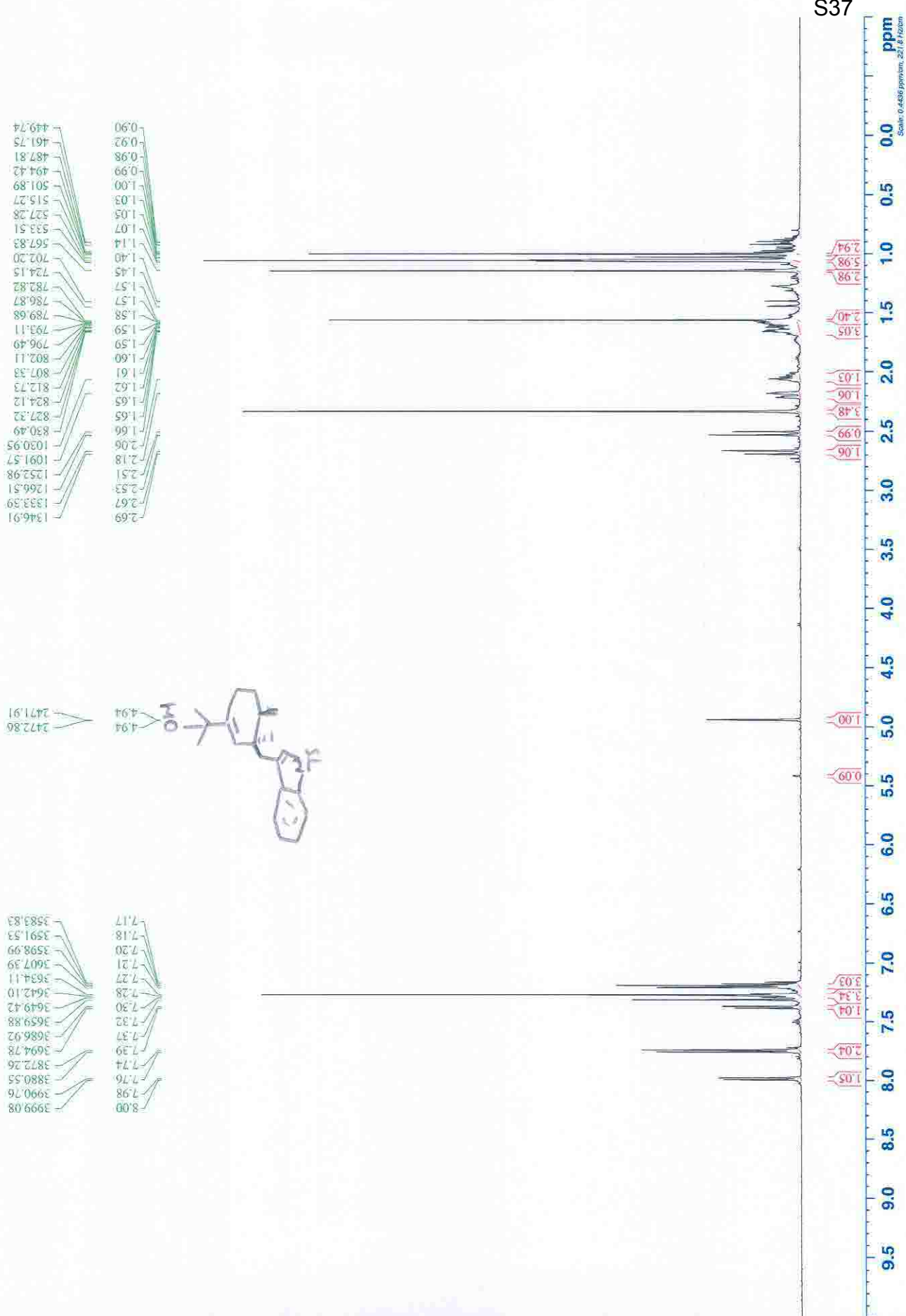
```
=====
CHANNEL f2
=====
CPDRG2
waltz16
    1H
    80.000 usec
    15.00 dB
    15.00 dB
    0.20 dB
```

F2 - Processing parameters	
SI	131072
SF	150.9027818 MHz
EM	0
WDW	1.00 Hz
SSB	0
LB	0
GB	0
PC	1.40



Scale: 11.11 ppm/cm, 167.4 Hz/cm









Mem Analytical Core Facility  
Rockefeller Research Laboratories  
430 E 71th St  
Box 208, NY 10021

## Current Data Parameters

NAME ejv-iv-18-13C

PROBHD 5mm PABIO BB-

PULPROG zgpg30

TD 327

SOLVENT CDCl3

NS 325

SWH 300.62500 Hz

FIDRES 0.291198 Hz

AQ 1.717093 sec

RG 327.5

RM 42.860 usec

DE 6.00 usec

TE 297.2 K

AQ 1.717093 sec

DELTA 1.0000000 sec

TD 327

CHN1 13C

PI 9.50 usec

PL 1.00 dB

SFO1 125.014083 MHz

CHN2 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO2 125.014083 MHz

CHN3 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO3 125.014083 MHz

CHN4 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO4 125.014083 MHz

CHN5 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO5 125.014083 MHz

CHN6 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO6 125.014083 MHz

CHN7 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO7 125.014083 MHz

CHN8 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO8 125.014083 MHz

CHN9 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO9 125.014083 MHz

CHN10 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO10 125.014083 MHz

CHN11 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO11 125.014083 MHz

CHN12 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO12 125.014083 MHz

CHN13 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO13 125.014083 MHz

CHN14 600.132706 MHz

PI 9.50 usec

PL 1.00 dB

SFO14 125.014083 MHz

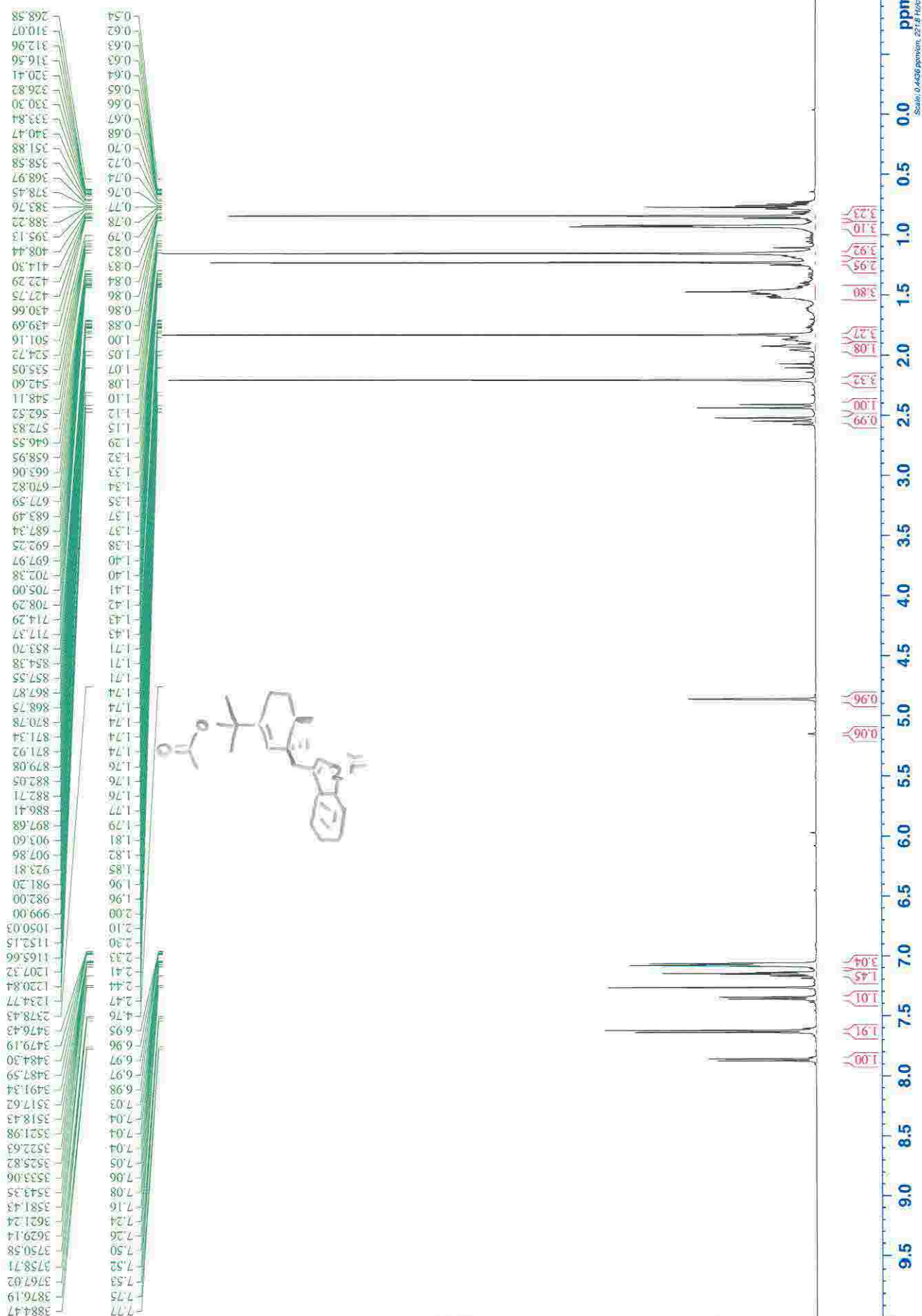
CHN15 600.132706 MHz

PI 9.50 usec

PL 1.00 dB



Proton24 \*. \* evelthui ejv-iii-294 (1 1) CDCl3 24.0C March\_20,2007\_10:38 DRX 500MHz zg30 1H \*.

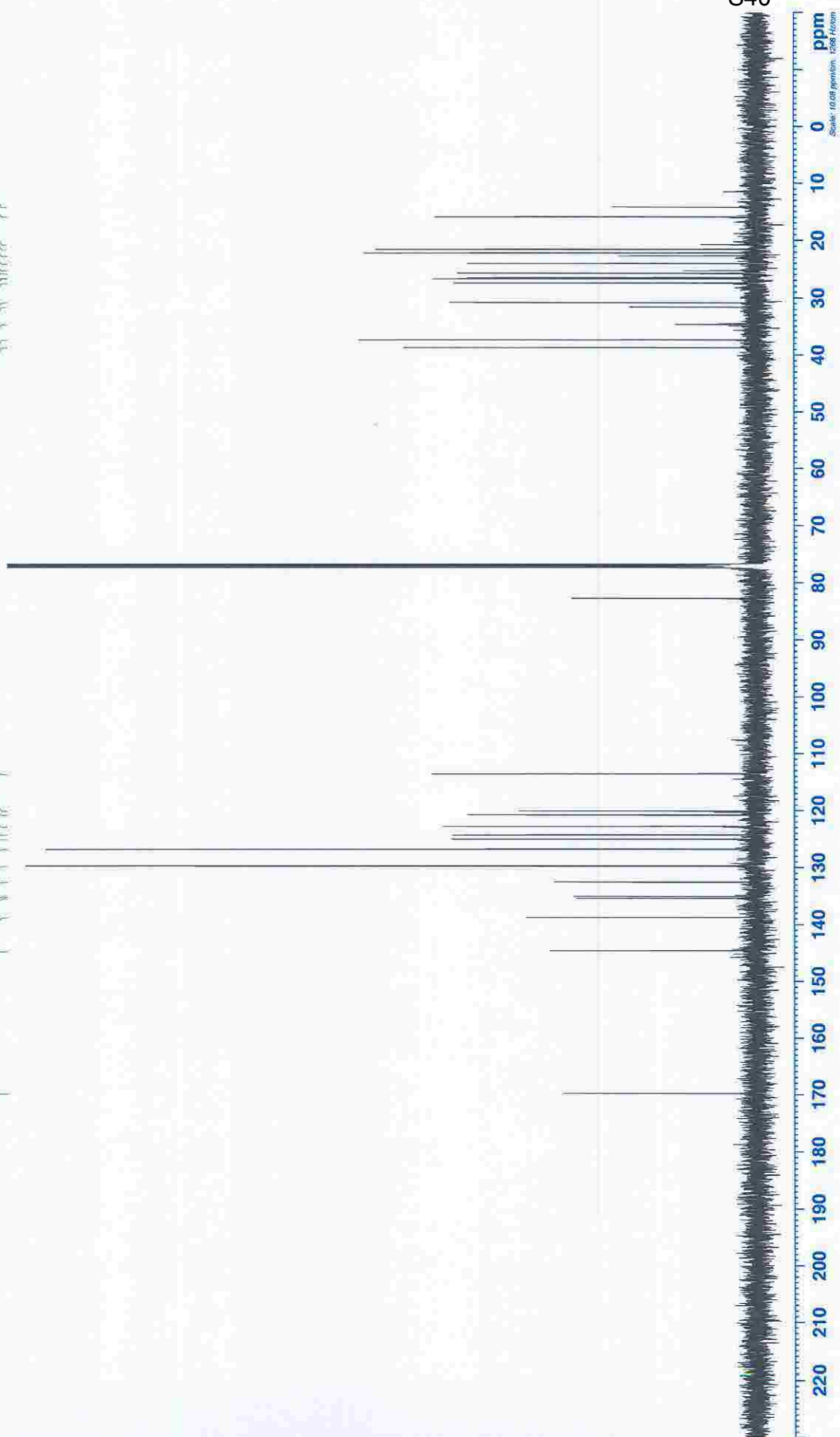




Carbon . \* evelthui ejv-iii-294-C13 (1 1) CDC13 24.0C March\_20,2007\_10:42 DRX 500MHz zgpg30 13C; 1H O2=4.000 \*.

169.76  
18187.68  
17451.45  
138.77  
135.37  
17023.27  
16979.94  
16662.21  
16312.51  
15938.97  
15716.39  
15624.98  
15440.10  
15185.14  
15092.26  
14276.43  
144.62  
132.49  
129.71  
126.74  
124.97  
124.25  
122.78  
120.75  
120.01  
113.52

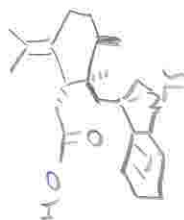
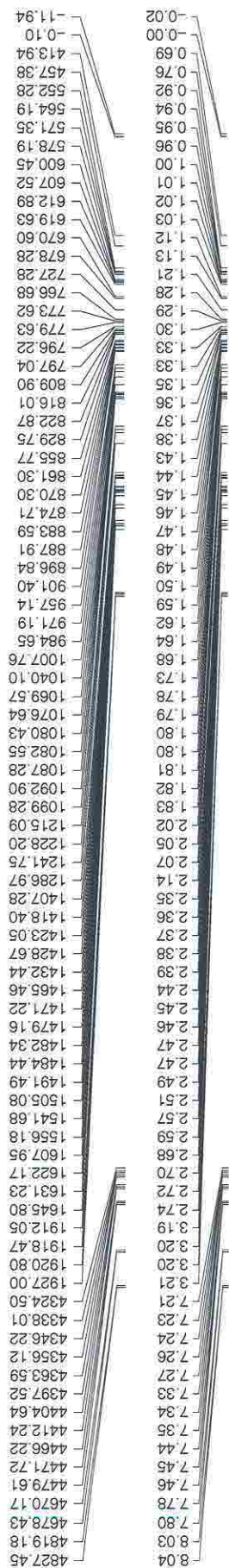
4872.19  
37.40  
34.68  
31.61  
30.82  
27.44  
26.72  
26.52  
25.75  
25.29  
24.03  
22.67  
21.54  
20.72  
15.90  
14.14  
3874  
4702.79  
4361.64  
3974.78  
3875.92  
3450.57  
3360.30  
3335.62  
3238.18  
3181.03  
3022.39  
2851.24  
2708.62  
2605.46  
1999.81  
1778.53



S40



Proton \* evelthui ejv-iv-87f (1 1) CDCl3 24.0C May\_31,2007\_09:06 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm \*



S41





Carbon-13 NMR Spectrum of 1,4-bis(4-ethoxyphenyl)benzene (BPEO) in CDCl<sub>3</sub>. The spectrum shows peaks for the aromatic carbons (120-140 ppm), the quaternary carbons (150-160 ppm), and the ethoxy carbons (16-18 ppm). The x-axis is labeled in ppm from 0 to 230.



MR Analytical Core Facility  
Woodward Hall, 6th Floor  
1275 York Ave  
New York, NY 10021

Current Data Parameters  
NAME: 1,4-bis(4-ethoxyphenyl)benzene  
EXPNO: 2  
PROCNO: 1

F2 - Acquisition Parameters  
Date\_ Time: 20070531 9:12

PROBHD: 5 mm PABBO BBI-  
PILPROG: zgpg30

TD: 65536  
AQ: 1.7176933 sec

RG: 2050  
DS: 0

SWH: 30062.500 Hz  
FIDRES: 0.0001000 Hz

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

RG: 2050  
AQ: 1.7176933 sec

181.69

146.72  
137.09  
137.07  
135.00  
131.88  
131.81  
128.89  
128.72  
127.98  
126.62  
126.38  
126.16  
124.88  
122.35  
122.01  
115.81  
115.59  
174.43.33  
174.76.16  
184.11.20  
184.62.23  
184.44.15  
190.37.95  
190.70.73  
191.07.21  
191.28.88  
193.12.88  
194.24.56  
194.49.21  
198.90.03  
199.01.78  
203.71.63  
206.84.89  
206.87.84  
221.40.94

79.28  
79.07  
78.86  
11963.50  
11931.57  
11899.61

43.96  
43.82  
43.25  
42.47  
38.40  
36.93  
36.05  
35.26  
53.21.33  
51.20.43  
49.43.73  
46.88.68  
42.54.66  
40.83.41  
26.66  
24.74  
23.57  
23.51  
35.48.23  
34.43.07  
33.71.06  
22.29  
33.63.49  
29.17.57  
27.16.54  
26.95.07  
24.39.33  
-0.14

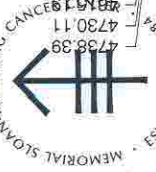
S42

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Scale: 1:111 ppm/cm 1677 Hz/cm



Proton \* evelthui ejv-iv-20-1H (1 1) CDC13 24.0C April\_02,2007\_16:17 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm \*



4738.39  
4730.11  
4606.86  
4404.16  
4396.24  
4392.44  
4382.28  
4326.11  
4318.65  
4314.55  
4311.52  
4289.24  
4281.19  
4274.07  
4266.55  
2170.62  
2145.41  
2093.41  
1854.65  
1848.78  
1845.18  
1839.51  
1722.41  
1553.15  
1538.54  
1520.92  
1477.93  
1463.49  
1415.72  
1406.02  
1392.34  
1386.21  
1378.46  
1372.78  
1348.27  
1260.71  
1184.87  
1166.14  
1152.08  
1138.87  
1025.44  
1021.26  
1019.03  
1013.03  
1008.66  
1006.21  
1002.14  
964.04  
932.19  
898.67  
890.78  
884.53  
817.17  
811.39  
806.83  
797.94  
793.47  
784.17  
782.31  
771.62  
771.00  
720.57  
713.27  
711.45  
706.36  
695.35  
596.95  
591.65  
554.30  
550.26  
535.11  
528.33  
519.00  
506.95  
472.01  
445.01  
372.84  
315.47

MEMORIAL SLOAN-KETTERING CANCER CENTER  
ESTABLISHED 1884  
NMR Analytical Core Facility  
Rockefeller University  
1230 York Ave.  
New York, NY 10021

Current Data Parameters  
NAME ejv-iv-20-1H  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20070402  
Time 16:19  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zg30  
TD 65536  
SOLVENT CDC13  
NS 24  
DS 0  
SWH 13227.514 Hz  
FIDRES 0.201836 Hz  
AQ 2.4773109 sec  
RG 287  
DW 37.800 usec  
DE 6.00 usec  
TE 297.2 K  
F1 1.00000000 sec  
TD0

===== CHANNEL f1 =====  
NUC1 1H  
P1 14.00 usec  
PL1 0.20 dB  
SFO1 600.1345010 MHz

F2 - Processing parameters  
SI 65536  
SF 600.1300531 MHz  
WDW EM  
SSB 0  
LB 0.30 Hz  
GB 0  
PC 1.00



S43





Carbon \* evelthui ejv-iv-20\_cosy (3 1) CDCl3 24.0C April\_02,2007\_17:33 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm \*

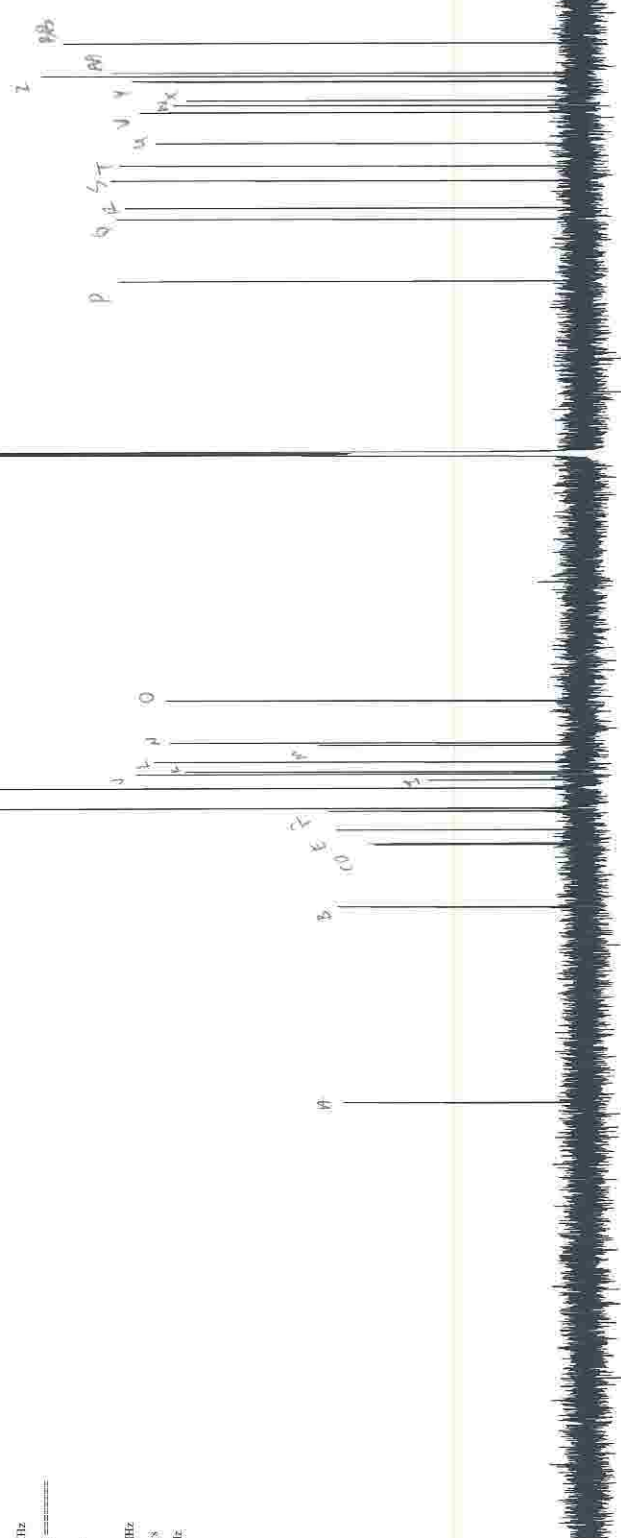
S44



MR Analytical Core Facility  
Department of Radiology  
400 E 71st St  
New York, NY 10021

Current Data Parameters  
NAME ejv-iv-20\_cosy  
EXPNO 3  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20070402  
Time 23:57  
INSTRUM spect  
PROBHD 5 mm TAIHO BBO-  
PULPROG zgpg30  
TD 134144  
SOLVENT DMSO  
NS 8000  
DS 0  
SWH 30062.500 Hz  
FIDRES 0.000180 Hz  
AQ 1.7176932 sec  
RG 2050  
DWT 12.800 usec  
DE 297.0 K  
TE 300.2 K  
D1 1.10000000 sec  
d11 0.03000000 sec  
DELTA 1.10000000 sec  
TIMO 1  
===== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 2.00 dB  
SFO1 100.6281500 MHz  
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 8.00 usec  
PL2 15.00 dB  
PL12 15.00 dB  
PL13 15.00 dB  
PL2 0.20 dB  
SFO2 500.1327006 MHz  
F2 - Processing parameters  
SI 32768  
SF 500.1327006 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

173.72  
26214.97  
144.55  
135.17  
20397.46  
20375.72  
20069.28  
19643.58  
1930.17  
19583.60  
1929.78  
126.77  
18943.41  
125.53  
18810.75  
18755.53  
18533.73  
122.82  
120.38  
119.99  
17162.85  
113.73  
76.79  
77.00  
11619.48  
11587.54  
77.21  
51.27  
63.45  
60.93  
54.80  
40.36  
36.32  
51.52  
34.14  
30.77  
46.43  
39.48  
26.17  
37.87  
25.10  
24.38  
32.49  
31.24  
20.71  
20.27  
15.83  
2389.52



230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm  
Scale: 11.11 ppm/cm, 1077 Hz/cm

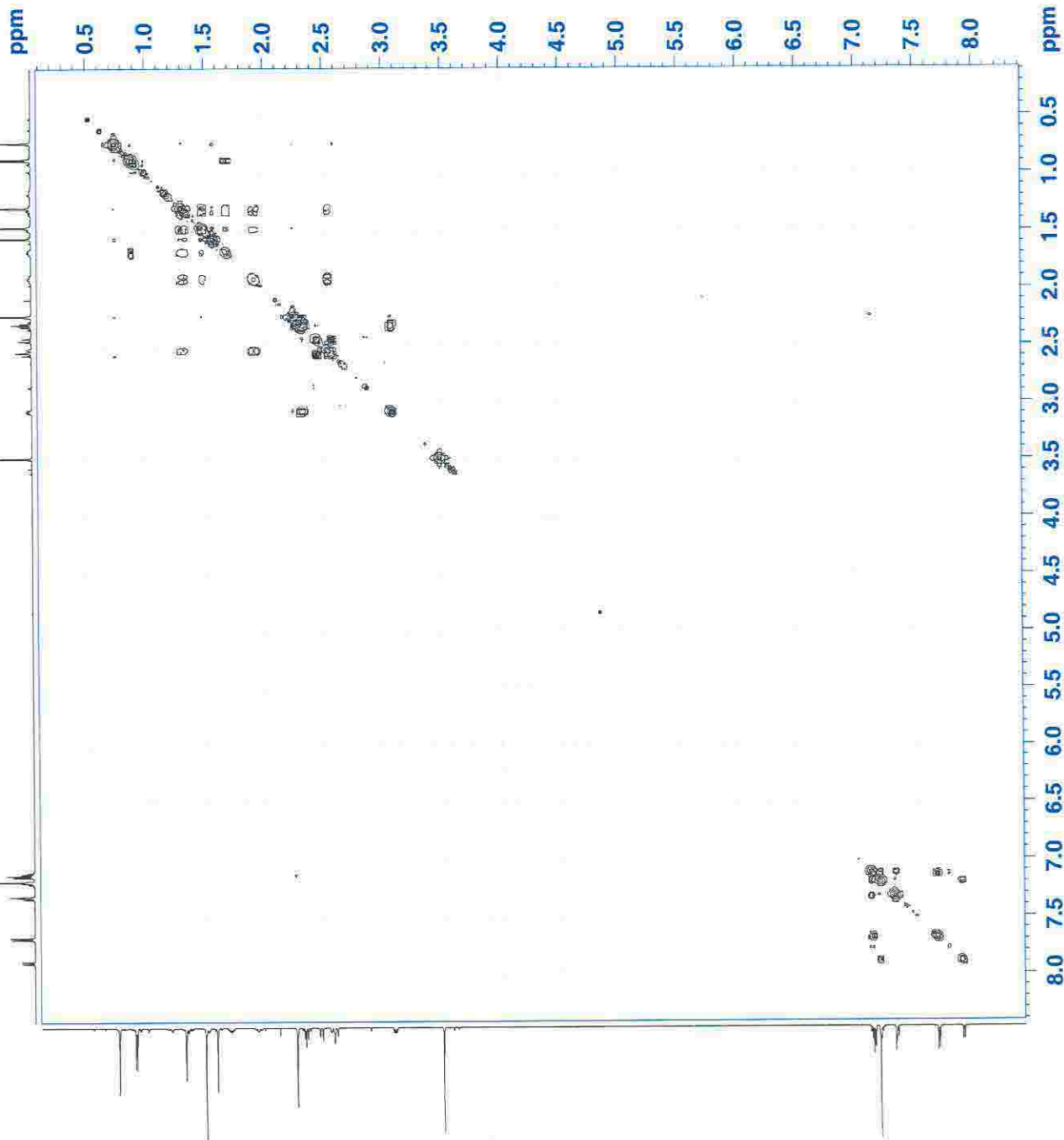


# Grad COSY

ui ejv-iv-20\_cosy (2 1) CDCI3 24.0C April\_02,2007\_17:08 Bruker AVII+ 600MHz RRL1326: cosygpqf : 1H 4.34C



NMR Analytical Core Facility  
Rockefeller Research Laboratories  
430 E 67th St  
New York, NY 10021



Current Data Parameters  
NAME ejv-iv-20\_cosy  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20070402  
Time 17.09  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG cosygpgqf  
TD 2048  
SOLVENT CDCI3  
NS 12  
DS 12  
SWH 5000.000 Hz  
FIDRES 2.441406 Hz  
AQ 0.2048500 sec  
RG 144  
DM 100.000 usec  
DE 5.00 usec  
TE 297.2 K  
d0 0.00000300 sec  
d1 1.40988600 sec  
d13 0.00000400 sec  
d16 0.00020000 sec  
TD 0.00020000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P0 14.00 usec  
PL 0.00 dB  
F1 0.20 MHz  
SFO1 600.1326033 MHz

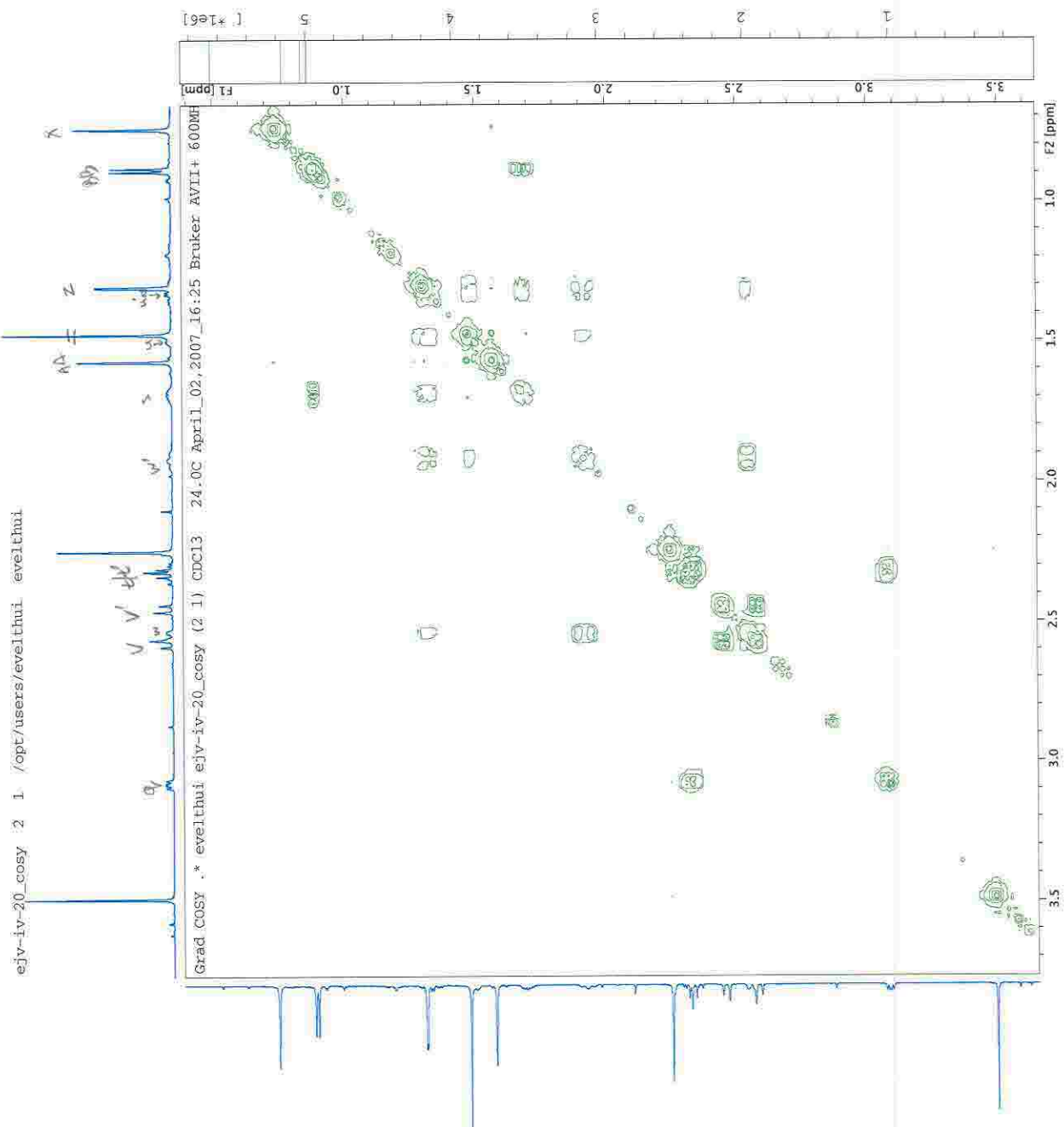
===== GRADIENT CHANNEL =====  
GPM1 SINE 100  
GPM2 SINE 100  
GP21 10.00 %  
GP22 10.00 %  
P16 1000.00 usec

F1 - Acquisition parameters  
ND0 1  
TD 128  
SFO1 600.1326 MHz  
FIDRES 39.062500 Hz  
SW 8.331 ppm  
FNAME Qf

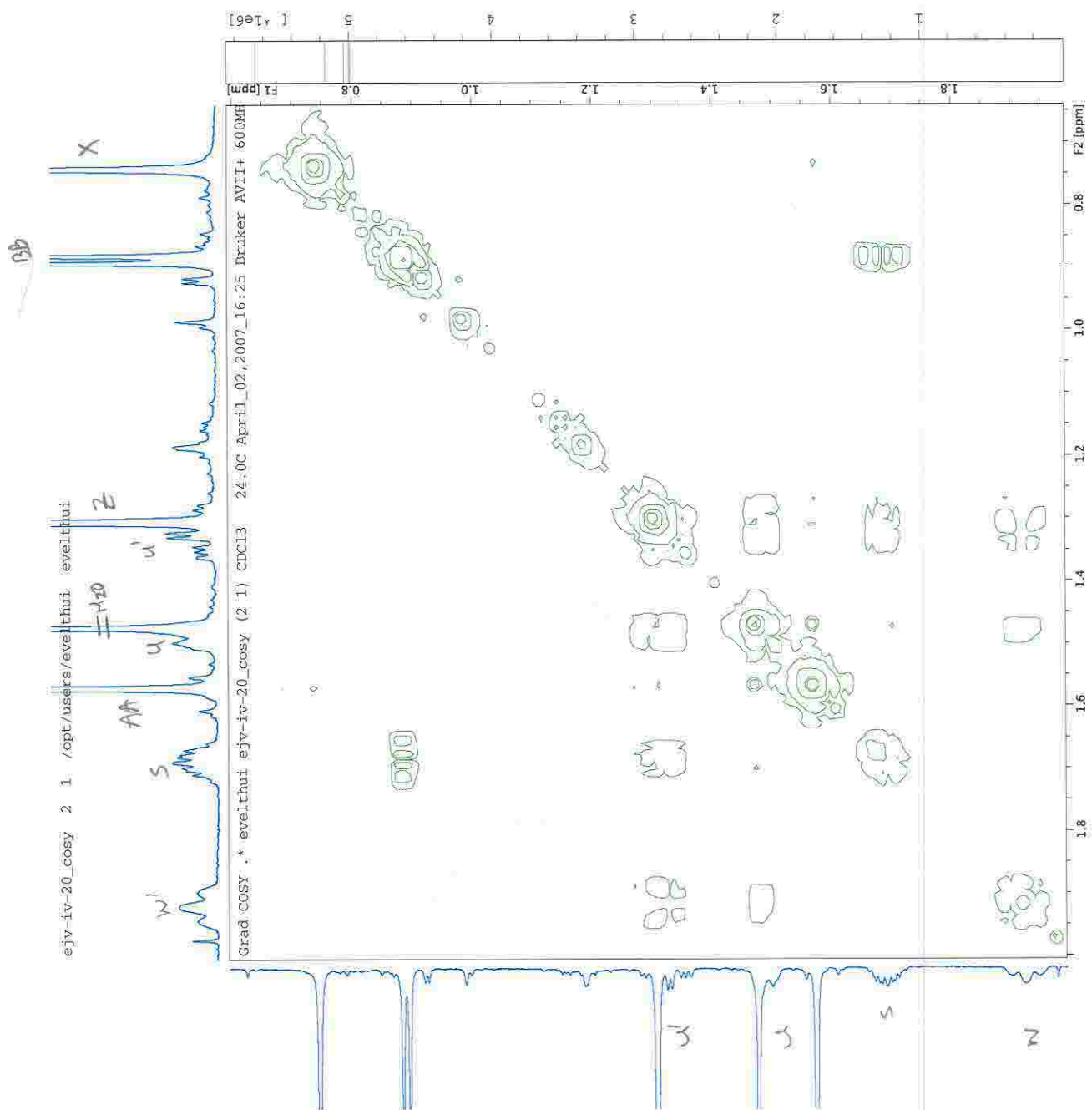
F2 - Processing parameters  
SI 1024  
SF 600.1300527 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0  
PC 1.40

F1 - Processing parameters  
SI 1024  
MC2 QF  
SF 600.1300527 MHz  
WDW SINE  
SSB 0  
LB 0.00 Hz  
GB 0





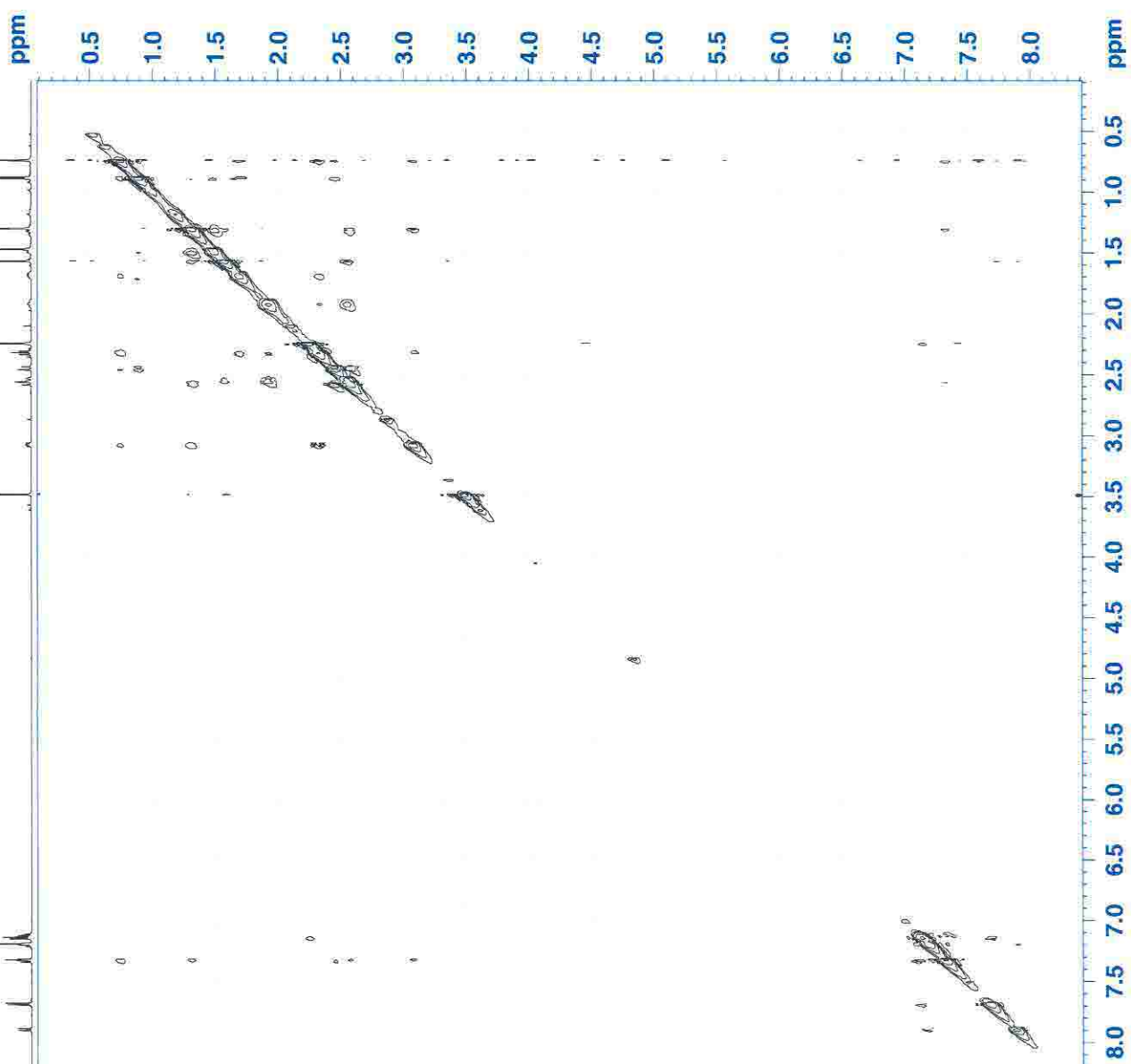






# NOESY

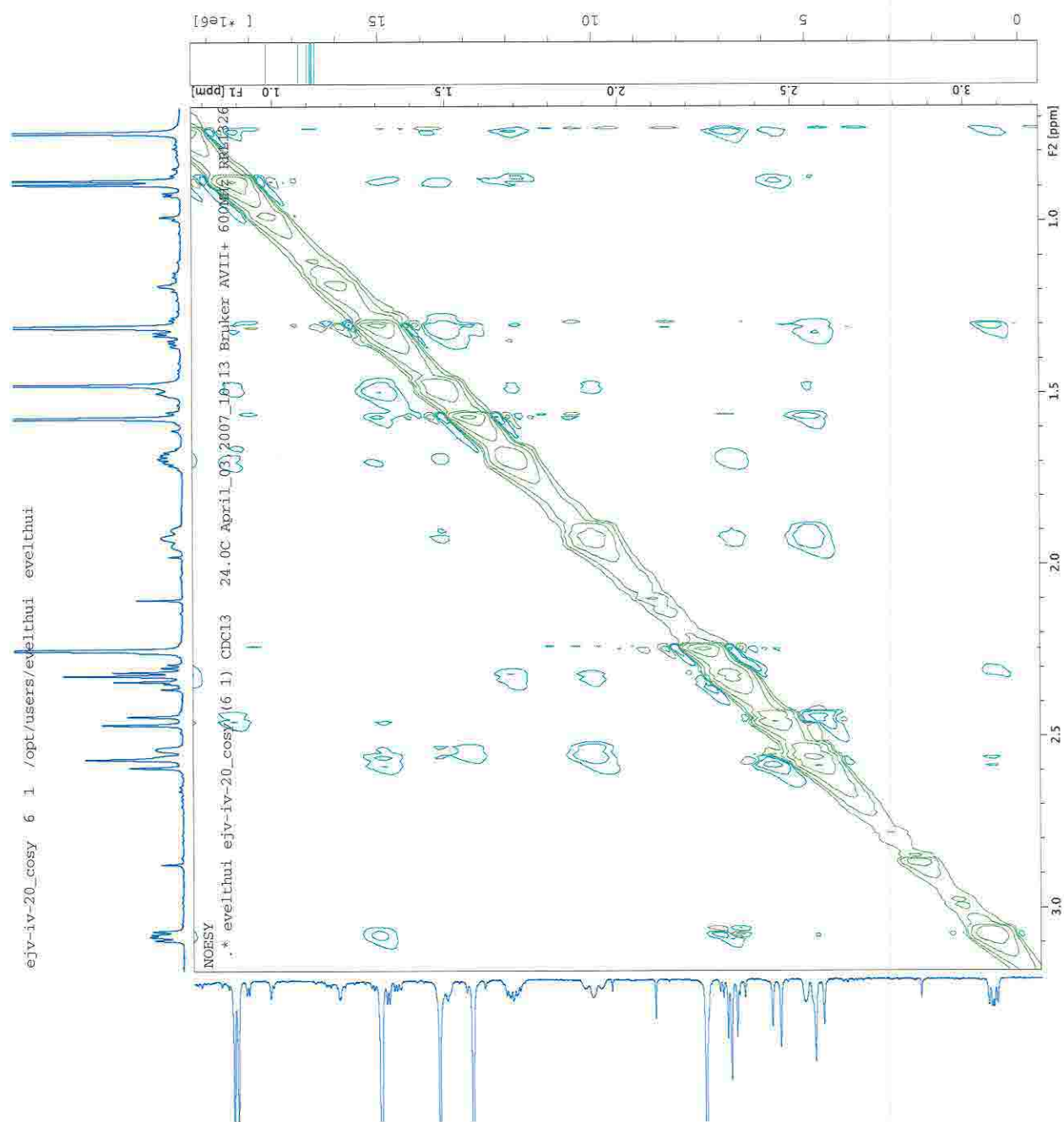
uij ejv-iv-20\_cosy (6 1) CDCI3 24.0C April\_03,2007\_10:13 Bruker AVII+ 600MHz RRL1326: noesyph : 1H 4.603



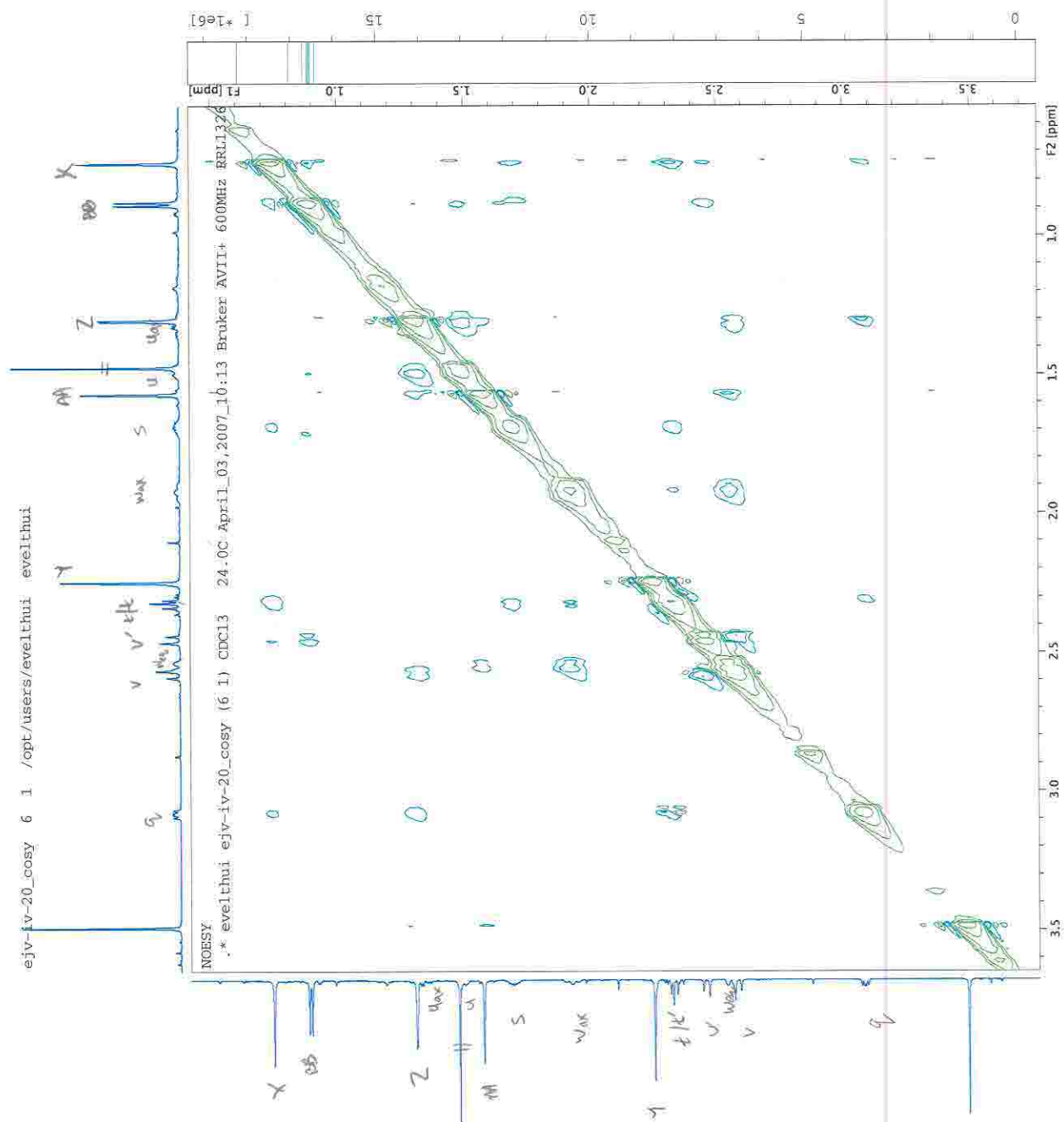
Current Data Parameters  
 NAME ejv-iv-20\_cosy  
 EXPNO 2  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20070403  
 Time 10:15  
 INSTRUM spect  
 PROBD 5 mm EBBQ BB-  
 PULPROG noesyph  
 TD 2048  
 SOLVENT CDCl3  
 NS 16  
 DS 16  
 SWH 5000.000 Hz  
 FIDRES 2.441406 Hz  
 AQC 0.2048500 sec  
 RG 181  
 DW 100.000 usec  
 DE 6.00 usec  
 TE 297.2 K  
 DO 0.00008217 sec  
 EI 1.9621604 sec  
 D8 0.30000001 sec  
 INO 0.00020000 sec  
 STICNT 128  
 ===== CHANNEL f1 =====  
 NUC1 1H  
 P1 14.00 usec  
 PL1 0.20 dB  
 SFO1 600.1326033 MHz  
 F1 - Acquisition parameters  
 ND0 1  
 TD 256  
 SFO1 600.1326 MHz  
 FIDRES 19.531250 Hz  
 SW 8.331 ppm  
 PRMODE States-TPPI  
 F2 - Processing parameters  
 SI 1024  
 SF 600.1300527 MHz  
 WDW QSI  
 SSF 2  
 GB 0.00 Hz  
 PC 1.00  
 F1 - Processing parameters  
 SI 1024  
 MC2 States-TPPI  
 SF 600.1300527 MHz  
 SSF 2  
 LB 0.00 Hz  
 GB 0



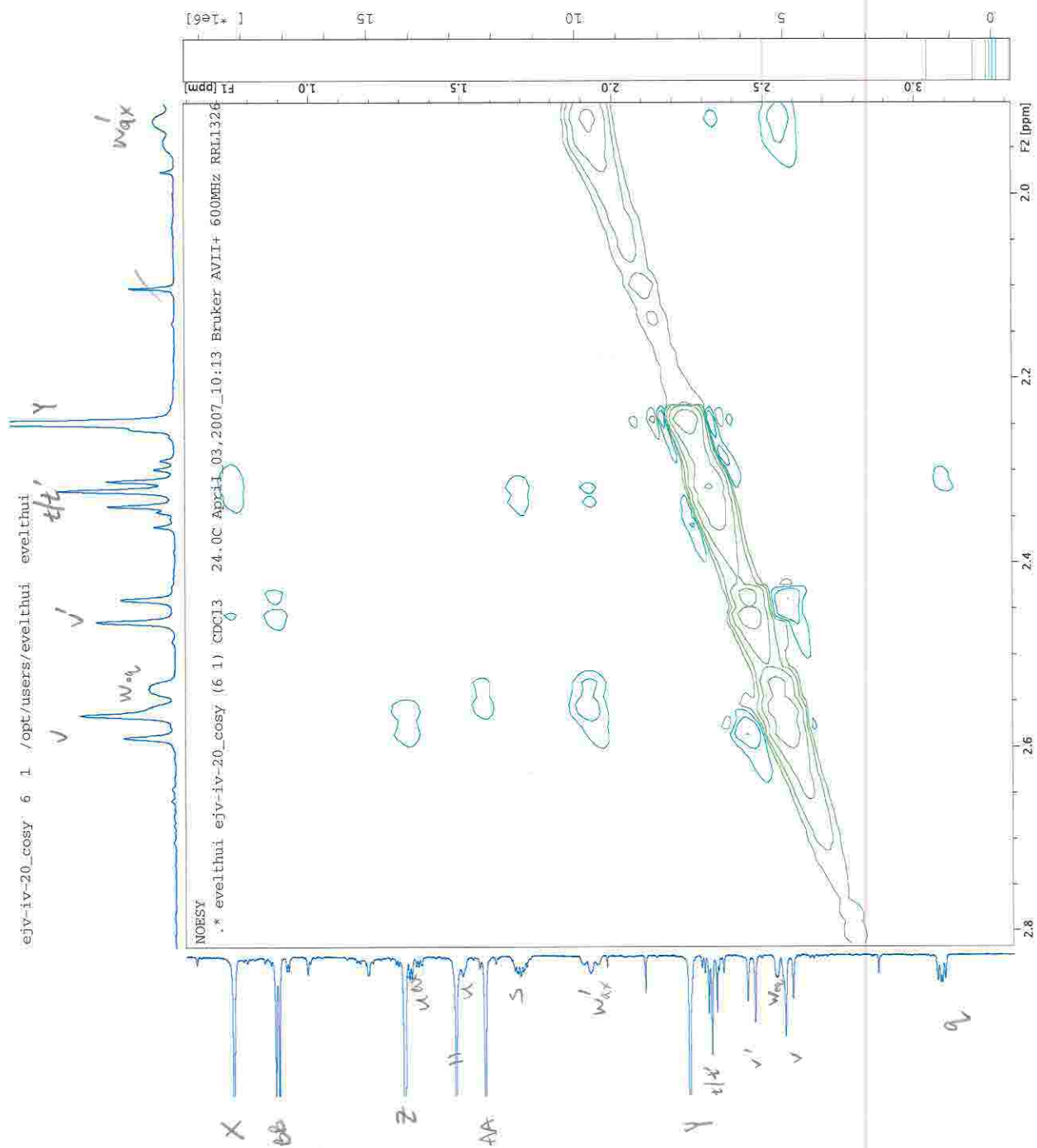
eiv-iv-20\_cosy 6 1 /opt/users/evelthui evelthui





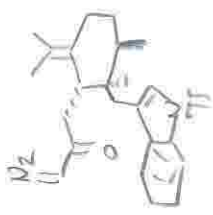
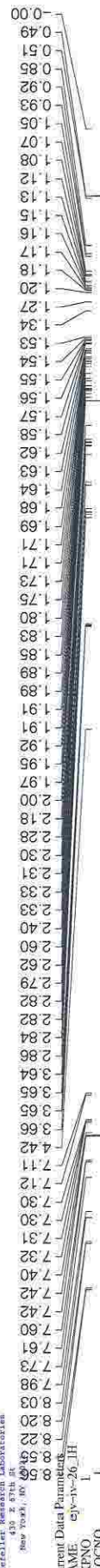
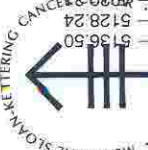








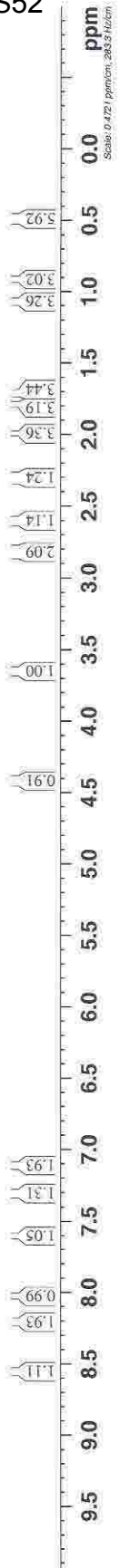
Proton \* evelthui ejv-iv-26\_1H (1 1) C6D6 24.0C April\_04\_2007\_17:28 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm \*



Current Data Parameters  
 Name: ejv-iv-26\_1H  
 EXPNO: 1  
 PROCNO: 1  
 F2 - Acquisition Parameters  
 Date\_ : 20070404  
 Time : 17:29  
 INSTRUM: spect  
 PROBHD: 5 mm PABBO BB-  
 PULPROG: zg30  
 TD: 65536  
 SOLVENT: C6D6  
 NS: 24  
 DS: 0  
 SWH: 13227.514 Hz  
 FIDRES: 0.201836 Hz  
 AQ: 2.4773109 sec  
 RG: 64  
 DW: 37.800 usec  
 DE: 6.00 usec  
 TE: 297.2 K  
 D1: 1.0000000 sec  
 TDO: 1

===== CHANNEL f1 =====  
 NUC1: 1H  
 PL1: 14.00 usec  
 PL1: 0.20 dB  
 SFO1: 600.1345010 MHz  
 F2 - Processing parameters  
 SI: 65536  
 SF: 600.1299099 MHz  
 EM: 0  
 WDW: 0  
 LB: 0.30 Hz  
 GB: 0  
 PC: 1.00

S52





Carbon \* evelthui ejv-iv-26\_13C (1 1) C6D6 24.0C April\_04\_2007\_17:31 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm \*



Current Data Parameters  
NAME: GY-N-13C  
EXPNO: 1  
PROCNO: 1  
F2-Acquisition Parameters  
Date\_ Time: 20070404 18:01  
INSTRUM: spect  
PULPROG: zgpg30  
TD: 131144  
SOLVENT: C6D6  
US: 0  
SWH: 39462.500 Hz  
FIDRES: 0.291198 Hz  
AQ: 1.717692 sec  
RG: 2050  
DW: 12.800 usec  
DE: 2.00 usec  
D1: 2.00 usec  
D11: 0.0000000 sec  
DELTA: 1.0000000 sec  
F2-Processing parameters  
SI: 131072  
SF: 150907758 MHz  
WDW: EM  
SSB: 0  
LB: 1.00 Hz  
GB: 0  
PC: 1.46

193.56  
29209.29  
144.33  
142.54  
136.04  
135.92  
135.75  
133.53  
130.73  
130.16  
130.01  
129.83  
129.73  
128.56  
128.31  
128.18  
128.02  
127.86  
127.58  
127.22  
125.57  
125.50  
124.62  
123.09  
120.28  
120.23  
114.35  
18142.45  
18150.60  
18574.87  
18805.38  
18937.81  
18949.04  
19154.02  
19197.46  
19252.30  
19295.02  
19319.24  
19343.46  
19362.99  
19399.52  
19577.16  
19591.27  
19618.96  
19641.19  
19727.10  
20150.02  
20484.57  
20509.99  
20528.79  
21509.71  
21779.78

53.15  
41.70  
40.60  
39.19  
37.41  
36.70  
31.30  
30.90  
30.45  
30.19  
29.05  
28.96  
27.97  
26.84  
26.17  
25.70  
24.73  
24.61  
21.07  
21.04  
20.21  
3049.79  
2418.43  
2389.16  
847.28  
334.36  
-2.22

S53





Proton  
 \*. evelthui ejv-iv-28f (1 1) CDCl3 24.0C April\_07.2007\_13:36 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm \*

4809.39  
 4801.12  
 4637.20  
 4629.01  
 4439.73  
 4431.95  
 4386.40  
 4374.92  
 4364.49  
 4332.11  
 4324.36  
 4318.96  
 4310.96

8.01  
 8.00  
 7.73  
 7.71  
 7.40  
 7.38  
 7.31  
 7.29  
 7.27  
 7.22  
 7.21  
 7.20  
 7.18

1604.59  
 1589.99  
 1544.46  
 1530.97  
 1496.41  
 1481.84  
 1454.89  
 1451.54  
 1442.89  
 1392.30  
 1386.16  
 1126.38  
 1119.56  
 1114.54  
 1102.96  
 1023.62  
 976.37  
 934.24  
 922.12  
 858.57  
 851.13  
 846.58  
 843.24  
 828.81  
 771.19  
 762.21  
 733.17  
 640.57  
 586.67  
 579.83  
 567.83  
 561.12  
 521.99

2.67  
 2.65  
 2.57  
 2.55  
 2.49  
 2.47  
 2.42  
 2.42  
 1.451.54  
 1.442.89  
 1.392.30  
 1.386.16  
 1.126.38  
 1.119.56  
 1.114.54  
 1.102.96  
 1.023.62  
 0.976.37  
 0.934.24  
 0.922.12  
 0.858.57  
 0.851.13  
 0.846.58  
 0.843.24  
 0.828.81  
 0.771.19  
 0.762.21  
 0.733.17  
 0.640.57  
 0.586.67  
 0.579.83  
 0.567.83  
 0.561.12  
 0.521.99



S54





Carbon \* evelthui ejv-iv-28f (2 1) CDCI3 24.0C April\_07,2007\_13:38 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm \*

S55

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm  
Scale: 11.11 ppm/cm, 1677 Hz/cm

1300.19  
2390.38  
3026.64  
20.80  
31.38.60  
32.39.01  
34.25.00  
37.34.71  
38.06.79  
40.17.48  
46.62.22  
48.23.10  
54.58.64  
61.23.10  
66.46.13  
69.13.86  
8.62  
15.84  
20.06  
20.80  
21.46  
22.70  
24.75  
25.23  
26.62  
30.90  
31.96  
36.17  
40.58  
44.04  
45.82

11652.53  
11620.62  
11588.68  
76.80  
77.01  
77.22

17164.60  
17172.85  
18117.04  
18267.30  
18538.63  
18737.58  
18765.56  
18975.85  
19118.53  
19122.00  
19629.96  
19629.96  
20092.63  
20389.29  
20409.16  
21838.44  
144.72  
135.25  
135.12  
133.15  
130.08  
129.76  
129.67  
126.72  
126.69  
125.75  
124.36  
124.17  
122.85  
121.05  
120.06  
113.80  
113.75

Current Data Parameters  
NAME gyl-iv-28f  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20070607  
Time 14:59:00  
INSTRUM spect  
PROBHD 5mm TAIABBO BB-  
PULPROG zgpg30  
SOLVENT CDCl3  
NS 5000  
DS 0  
SWH 3901.500 Hz  
FIDRES 0.291198 Hz  
AQ 1.7176932 sec  
RG 2050  
DE 1.000000 sec  
TE 297.2 K  
D1 1.1000002 sec  
D11 0.0500000 sec  
DELTA 1.0000000 sec  
TD0 1

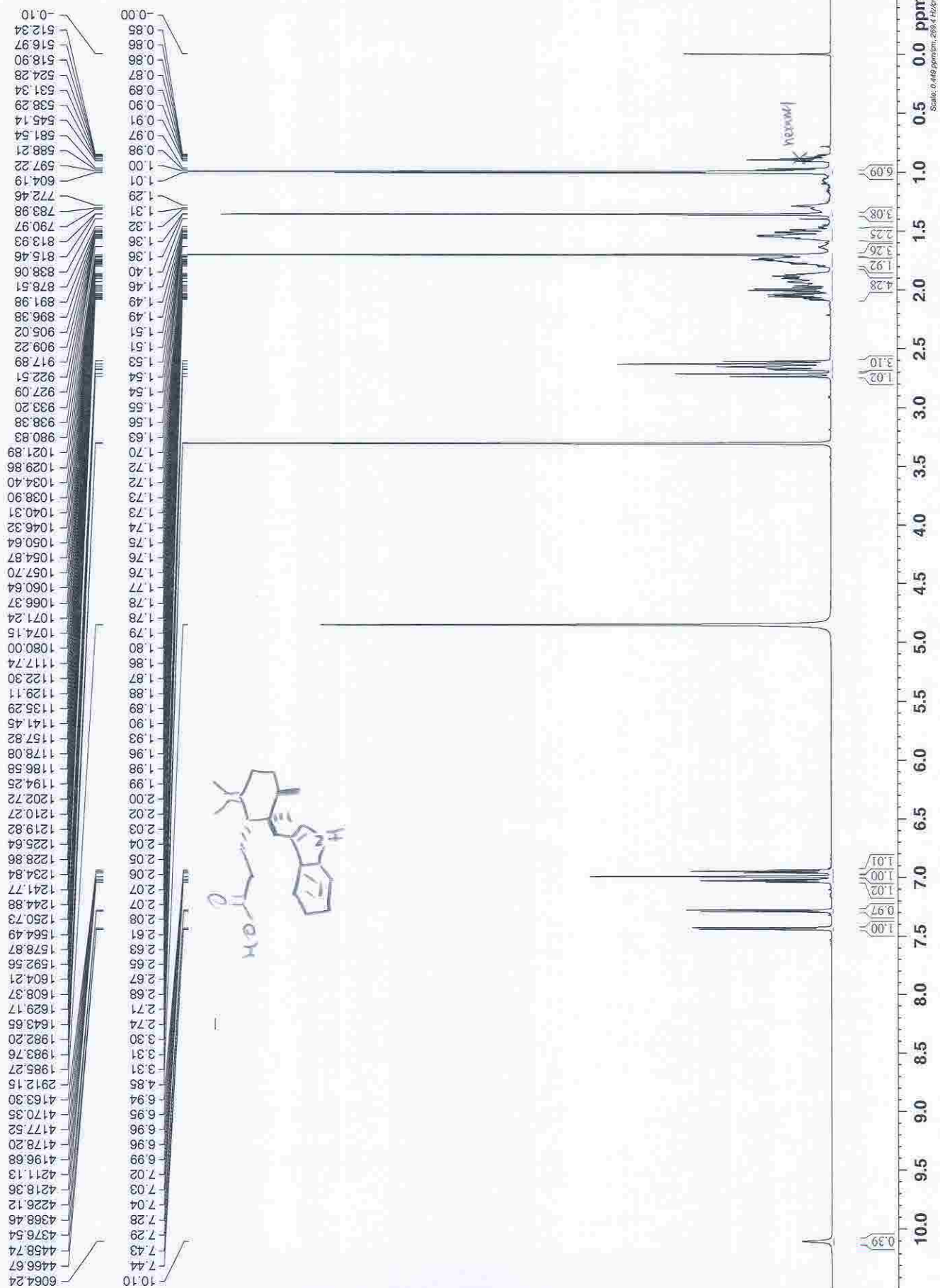
CHANNEL 1  
NUC1 13C  
P1 9.50 usec  
PL1 2.00 dB  
SFO1 100.626119 MHz

CHANNEL 2  
CDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL14 15.00 dB  
SFO2 400.147605 MHz

F2 - Processing parameters  
SI 32768  
SF 150.902690 MHz  
WDW EM  
SSB 0  
GB 0  
PC 1.40



Proton . \* evelthui ejv-iv-76-f (1 1) MeOD 24.0C May\_23,2007\_16:37 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm \*





Carbon \* evelthui ejv-iv-76\_13C (1 1) MeOD 24.0C May\_22,2007\_12:02 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm \*

S57

Scale 11.11 ppm/cm, 657-Hz/cm

2180.25  
2474.68  
3067.88  
3174.14  
3270.87  
3579.21  
3621.88  
3909.44  
3937.74  
3967.02  
4209.55  
4876.70  
4945.06  
4997.74  
5387.34  
5696.59  
6317.36  
6877.71  
7333.25  
7354.67  
7376.08  
7397.49  
7418.91  
7440.33  
7461.76  
49.45  
49.31  
49.16  
49.02  
48.88  
48.74  
48.60  
45.58  
41.86  
37.75  
35.70  
33.12  
32.77  
32.32  
27.90  
26.22  
26.09  
25.91  
24.00  
23.72  
21.68  
21.03  
20.33  
16.40  
14.45

16888.48  
16896.60  
17092.88  
17981.59  
18127.65  
18380.16  
18761.70  
18786.53  
19049.12  
19735.42  
19972.45  
20779.64  
137.70  
132.35  
130.78  
126.23  
124.49  
124.33  
121.80  
120.13  
119.16  
113.27  
111.97  
111.92

178.22  
26893.85



Memorial Sloan-Kettering Cancer Center  
1275 York Ave., Box 208  
New York, NY 10021

Current Data Parameters  
NAME: giv-iv-76\_13C  
EXPNO: 1  
PROCNO: 1

F2 - Acquisition Parameters  
Date\_ 20070522  
Time 12:02  
INSTRUM spect  
PROBHD 5mm PABBO HBI-  
PULPROG zgpg30  
SOLVENT MeOD  
NS 864  
DS 4  
SWH 30602.500 Hz  
FIDRES 0.29108 Hz  
AQ 1.7170932 sec  
RG 328.000  
RG 1.000 usec  
DE 6.00 usec  
TE 297.2 K  
D1 1.00000000 sec  
d11 0.03000000 sec  
DELTA 1.00000000 sec  
TD0 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.50 usec  
PL1 0.00 dB  
SFO1 100.628133 MHz

===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL12 15.00 dB  
PL13 15.00 dB  
PL14 15.00 dB  
PL15 15.00 dB  
SFO2 500.132706 MHz

F2 - Processing Parameters  
SI 32768  
SF 150.9025947 MHz  
WDW EM  
SSB 0  
GB 0  
EC 1.40



