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

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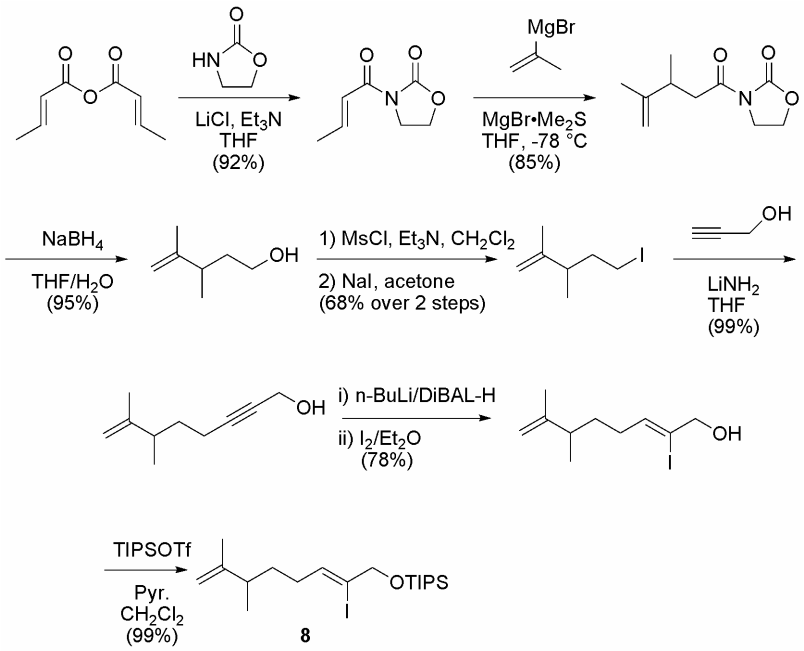
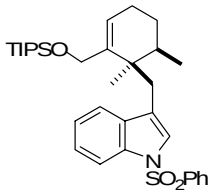
General Information:

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_2O), methylene chloride (CH_2Cl_2), 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_3N), diisopropylethylamine (*i*-Pr_2NEt), pyridine, 2,6-lutidine, and chlorotrimethylsilane (TMSCl) were distilled from CaH_2 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 $Pd(PPh_3)_4$, and Pd_2dba_3, which were purchased from Strem and $PhNTf_2$ 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 $CHCl_3$ (NaCl plates). Absorption bands are noted in cm^{-1}. ^1H- and ^{13}C NMR spectra were recorded on a Bruker AMX-400, a Bruker DRX-500, or a Bruker AVII+-600 spectrometer in $CDCl_3$. Chemical shifts (δ-values) are reported in ppm with residual undeuterated $CHCl_3$ as the internal standard (referenced to 7.26 ppm for 1H-NMR and 77.2 ppm for 13C-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).

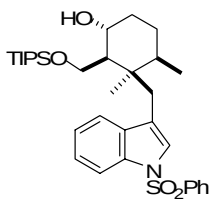
Scheme 1. Synthesis of Heck precursor **8**.





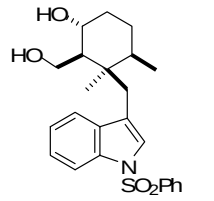
Chemical Formula: $C_{33}H_{47}NO_3SSi$
Exact Mass: 565.30459
Molecular Weight: 565.88168

Indole 10. A solution of alkenyl iodide **8** (0.5 g, 1.15 mmol), indole stannane **9** (0.94 g, 1.7 mmol), and Bu_4NCl (0.35 g, 1.3 mmol) in DMF (4 mL) was degassed with Ar. After 15 min, Ph_3As (35 mg, 0.12 mmol) and Pd_2dba_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 H_2O (10 mL) and Et_2O (10 mL). The layers were partitioned, and the aqueous phase was extracted with Et_2O (2 × 20 mL). The combined organic extracts were washed with H_2O (5 × 10 mL), brine (10 mL), dried ($MgSO_4$), 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}H NMR ($CDCl_3$, 400 MHz): δ 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}C NMR ($CDCl_3$, 125 MHz): δ 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, $[M+Na]^+$) calculated for: $C_{33}H_{47}NO_3SSiNa$ 588.2938. Found: 588.2933.



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

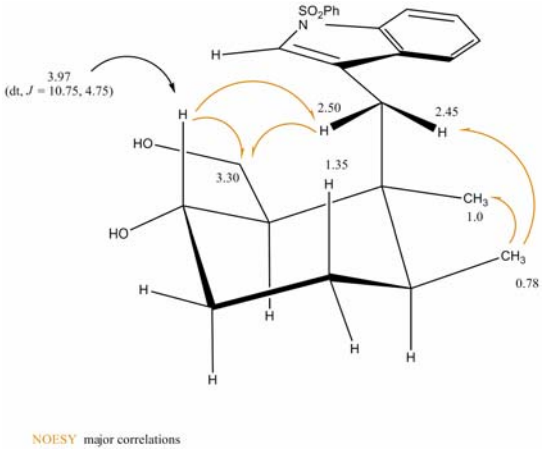
Alcohol 11. A solution of BH_3•THF (0.53 mL, 0.53 mmol, 1.0 M THF) was added dropwise to a solution of **10** (0.1 g, 0.18 mmol) in THF (0.5 mL) at 0 °C. The mixture was stirred for 3 h allowing it to warm to 23 °C. Excess borane was quenched by the slow addition of H_2O (36 μL), and the oraganoborane was sequentially treated with $NaBO_3$•$4H_2O$ (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 × 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: ^{1}H NMR ($CDCl_3$, 400 MHz): δ 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): δ 138.36134.73, 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.

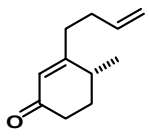


Chemical Formula: $C_{24}H_{29}NO_4S$
Exact Mass: 427.18173
Molecular Weight: 427.55636

Deprotection of 11: A solution of **11** (10 mg, 0.017 mmol) in THF (100 μL) and AcOH (4 μL, 0.068 mmol) at 23 °C was treated with TBAF (34 μL, 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): ^{1}H NMR ($CDCl_3$, 500 MHz): δ 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): δ 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: $C_{11}H_{16}O$
Exact Mass: 164.1201
Molecular Weight: 164.2441

(*R*)-3-(but-3-enyl)-4-methylcyclohex-2-enone (13).[1]

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**[2] 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_2O (10 mL). The layers were partitioned and the aqueous phase was extracted with Et_2O (2 × 20 mL), the combined organic extracts were washed with brine (2 × 20 mL), dried ($MgSO_4$), filtered and concentrated. The crude carbinol was then dissolved in CH_2Cl_2 (6 mL) and added to a flask charged with PCC (3.9 g, 18.2 mmol) and CH_2Cl_2 (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_2O (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 $NaHCO_3$ (2 × 20 mL) and brine (2 × 20 mL). The layers were dried ($MgSO_4$), 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: $[\alpha]^{22}_D$ = + 15° (c = 2.3, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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, 1 H), 1.19 (d, *J* = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 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, $[M+H]^+$) calculated for: $C_{11}H_{17}O$ 165.1273. Found: 165.1272.

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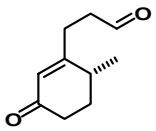
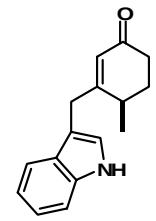
(1) Access to racemic **13** was accomplished by a Stork-Danheiser reaction of 3-ethoxy-6-methylcyclohex-2-enone and but-3-enylmagnesium bromide in 94% yield, see: Stork, G.; Danheiser, R. L. *J. Org. Chem.* **1973**, *38*, 1775–1776.

(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.



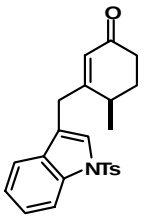
Chemical Formula: $C_{10}H_{14}O_2$
Exact Mass: 166.10
Molecular Weight: 166.22

(*R*)-3-(6-methyl-3-oxocyclohex-1-enyl)propanal (14). A suspension of **13** (0.5 g, 3.0 mmol) and $NaHCO_3$ (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_2Cl_2 (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° (c = 1.3, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 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, $[M+H]^+$) calculated for: $C_{10}H_{15}O_2$ 167.1066. Found 167.1063.



Chemical Formula: $C_{16}H_{17}NO$
Exact Mass: 239.13101
Molecular Weight: 239.31228

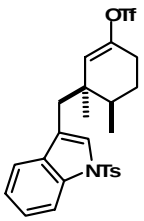
(*R*)-3-((1H-indol-3-yl)methyl)-4-methylcyclohex-2-enone. A mixture of 2-iodoaniline (6.4 g, 29.2 mmol), (*R*)-**14** (4.4 g, 26.5 mmol), and DABCO (8.9 g, 79.5 mmol) in DMF (130 mL) was degassed with Ar for 20 min. $Pd(OAc)_2$ (300 mg, 1.33 mmol) was added to the reaction, and the resulting reaction mixture was heated to 85 °C for 16 h. The reaction was cooled to 23 °C and was diluted with H_2O (100 mL). The aqueous phase was extracted with Et_2O (3 × 50 mL), and the combined organic phases were washed with brine (50 mL), dried ($MgSO_4$), 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° (c = 0.9, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 125MHz): 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, $[M+H]^+$) calculated for: $C_{16}H_{18}NO$ 240.1382. Found: 240.1383.



Chemical Formula: $C_{23}H_{23}NO_3S$
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 ^{1}H NMR analysis. Upon consumption of starting material the layers were partitioned, and the aqueous phase was extracted with Et_2O (3 × 20 mL). The combined organic phases were washed with brine (50 mL), dried ($MgSO_4$), 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

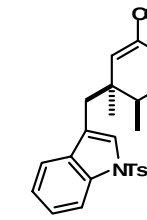
(91%) of **15** as a yellow foam: $[\alpha]^{22}_{D}$ = + 48° (c = 2.5, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 500 MHz): δ 7.98 (d, *J* = 8.0 Hz, 1H), 7.74 (d, *J* = 8.8 Hz, 1H), 7.38 (s, 1H), 7.36–7.21 (m, 2H), 7.23–7.19 (m, 3H), 5.70 (s, 1H), 3.59 (s, 1H), 2.53–2.44 (m, 2H), 2.35–2.29 (m, 4H), 2.11–2.05 (m, 1H), 1.82–1.76 (m, 1H), 1.25 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125MHz):δ 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, $[M+H]^+$) calculated for: $C_{23}H_{24}NO_3S$ 394.1471. Found: 394.1474.



Chemical Formula: $C_{25}H_{26}F_3NO_5S_2$
Exact Mass: 541.1204
Molecular Weight: 541.6028

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

A suspension of CuI (0.87 g, 4.58 mmol), in Et_2O (38 mL) was cooled to –30 °C and treated dropwise with a solution of MeLi (7.4 mL, 9.17 mmol, 1.25 M in Et_2O). After 10 min, the reaction mixture was warmed to 0 °C resulting in a colorless, homogeneous solution. A solution of enone **15** (1.5 g, 3.82 mmol) in Et_2O-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 $PhNTf_2$ (3.14 g, 8.79 mmol) in THF (9 mL). After 20 min, saturated aqueous NH_4Cl was added, and the layers partitioned. The aqueous phase was extracted with Et_2O (2 × 20 mL), and the combined organic extracts were washed with brine (2 × 20 mL), dried ($MgSO_4$), 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[3] with PhNHTf: ^{1}H NMR ($CDCl_3$, 600 MHz): δ 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, 1 H), 7.17–7.12 (m, 3H), 2.60 (d, *J* = 13.8 Hz, 1H), 2.54 (d, *J* = 13.8 Hz, 1 H), 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}C NMR ($CDCl_3$, 150MHz): δ 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, $[M+Na]^+$) calculated for: $C_{25}H_{26}NO_5F_3S_2Na$ 564.1096. Found: 564.1096.

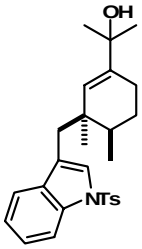


Chemical Formula: $C_{26}H_{29}NO_4S$
Exact Mass: 451.18
Molecular Weight: 451.58

(3*R*,4*R*)-Methyl 3,4-dimethyl-3-((1-tosyl-1H-indol-3-yl)methyl)cyclohex-1-enecarboxylate. A mixture of enol triflate **16** (2.0 g, 3.8 mmol), *i*-Pr_2EtN (3.3 mL, 19 mmol), MeOH (13 mL, 0.3 M), and DMF (20 mL) was purged with Ar for 5 min. $Pd(PPh_3)_4$ (0.22 g, 0.19 mmol) was added and CO was bubbled through the reaction mixture for 30 min. Et_2O (20 mL) and H_2O (20 mL) were added, and the layers partitioned. The aqueous phase was extracted with Et_2O (2 × 20 mL), and the combined organic extracts were washed with water (5 × 20 mL), brine (2 × 20 mL), dried ($MgSO_4$), filtered and concentrated. The residue was purified by flash column chromatography on silica gel (5→10% ethyl acetate – hexanes) to yield the title

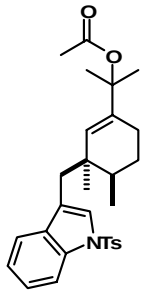
[3] Note, that on small scale, recrystallization of **18** from 5% ethyl acetate was successful in removing majority of the PhNHTf.

compound (0.76 g, 45% from **15**) as a white foam: $[\alpha]^{22}_D$ = + 40° (c = 1.1, $CHCl_3$). ^1H NMR ($CDCl_3$, 600 MHz): δ 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}C NMR ($CDCl_3$, 150MHz):
δ 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, 1 6.0; HRMS (ESI, $[M+H]^+$) calculated for: $C_{26}H_{30}NO_4S$ 452.1890. Found: 452.1887.



Chemical Formula: $C_{27}H_{33}NO_3S$
Exact Mass: 451.22
Molecular Weight: 451.62

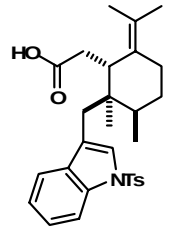
2-((3*R*,4*R*)-3,4-dimethyl-3-((1-tosyl-1H-indol-3-yl)methyl)cyclohex-1-enyl)propan-2-ol. A solution the above α,β-unsaturated ester (0.76 g, 1.69 mmol) in THF (13 mL) at 0 °C was treated with MeLi (4.0 mL, 5.0 mmol, 1.25 M in Et_2O). After 10 min, the reaction was poured into half-saturated NH_4Cl (20 mL) and Et_2O (20 mL) and the layers partitioned. The aqueous phase was extracted with Et_2O (2 × 10 mL), and the combined organic extracts were washed with brine (2 × 10 mL), dried ($MgSO_4$), filtered, and concentrated. The residue was purified by flash column chromatography on silica gel (10→20% ethyl acetate – hexanes) to yield the title compound (0.64 g, 84%) as a white foam: $[\alpha]^{22}_D$ = + 52° (c = 1.1, $CHCl_3$). ^1H NMR ($CDCl_3$, 500 MHz): δ 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}C NMR ($CDCl_3$, 150MHz):
δ 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, $[M+Na]^+$) calculated for: $C_{27}H_{33}NO_3SNa$ 474.2073. Found: 474.2069.



Chemical Formula: $C_{29}H_{35}NO_4S$
Exact Mass: 493.23
Molecular Weight: 493.66

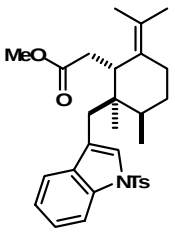
2-((3*R*,4*R*)-3,4-dimethyl-3-((1-tosyl-1H-indol-3-yl)methyl)cyclohex-1-enyl)propan-2-yl acetate (17). A mixture of the above tertiary allylic alcohol (0.5 g, 1.11 mmol), *i*-Pr_2EtNH (2.0 mL, 11.1 mmol), Ac_2O (0.63 mL, 6.65 mmol), and DMAP (13 mg, 0.11 mmol) in CH_2Cl_2 (2 ml) was maintained at 23 °C for 16 h. Et_2O (10 mL) and H_2O (10 mL) were added, and the layers partitioned. The aqueous phase was extracted with Et_2O (2 × 10 mL), and the combined organic extracts were washed with brine (20 mL), dried ($MgSO_4$), 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: $[\alpha]^{22}_D$ = + 67° (c = 1.1, $CHCl_3$). ^1H NMR ($CDCl_3$, 500 MHz): δ 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}C NMR ($CDCl_3$, 150MHz): δ 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, $[M+Na]^+$) calculated for: $C_{29}H_{35}NO_4SNa$ 516.2179. Found: 516.2174.



Chemical Formula: $C_{29}H_{35}NO_4S$
Exact Mass: 493.23
Molecular Weight: 493.66

2-((1*S*,2*R*,3*R*)-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 °C. The reaction mixture was warmed to 23 °C over a 2 h period, and maintained at this temperature for 16 h. Et_2O (10 mL) and 2M HCl (10 mL) were added, and the layers partitioned. The aqueous phase was extracted with Et_2O (3 × 5 mL), and 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 with 1% AcOH) to yield 0.25g (56%) of the title compound as a white foam: $[\alpha]^{22}_D$ = + 89° (c = 0.9, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 600 MHz): δ 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, 1 H), 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}C NMR ($CDCl_3$, 150MHz):δ 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, 27.0 8, 26.71, 23.54, 22.82, 22.30, 17.89; HRMS (ESI, $[M+H]^+$) calculated for: $C_{29}H_{36}NO_4S$ 494.2360. Found: 494.2365.



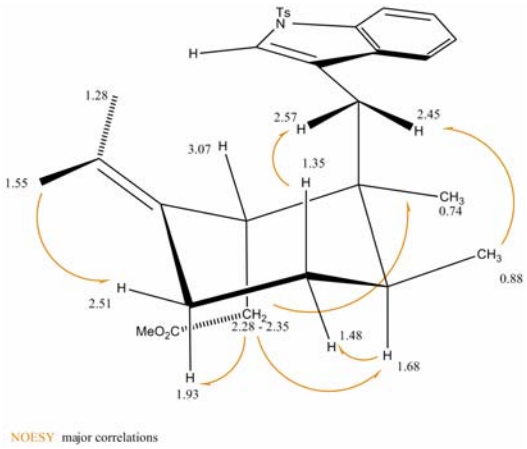
Chemical Formula: $C_{30}H_{37}NO_4S$
Exact Mass: 507.24433
Molecular Weight: 507.68408

Methyl 2,3-dimethyl-6-(propan-2-ylidene)-2-((1-tosyl-1H-indol-3-yl)methyl)cyclohexyl)acetate. A solution of **18** (5.0 mg, 0.01 mmol) in MeOH (0.5 mL) was treated with H_2SO_4 (15 μL, catalytic) at 23 °C and maintained for 16 h. The reaction mixture was poured into 10% aq. NaOH (1 mL) and extracted with Et_2O (3 × 2 mL). The combined organic extracts were washed with brine (5 mL), dried ($MgSO_4$), 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}H NMR ($CDCl_3$, 600 MHz): δ 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, 3 H), 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, 2 H), 2.25 (s, 3 H), 1.94–1.89 (m, 1 H), 1.71–1.66 (m, 1 H), 1.57 (s, 3 H), 1.49–1.47 (m, 4 H), 1.36–1.27 (m, 4 H), 6.80 (d, *J* = 6.8 Hz, 3 H), 0.74 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150MHz):

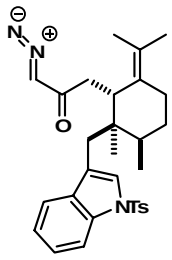
δ 173.72, 144.55, 135.17, 135.02, 132.99, 130.17, 129.77, 126.76, 125.53, 124.65, 124.28 , 122.82, 120.37, 119.99, 113.74, 51.27, 42.05, 40.38, 36.32, 34.14, 30.77, 26.17, 25.09, 2

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:



NOESY major correlations



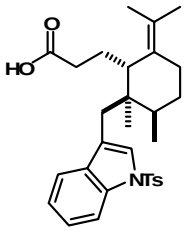
Chemical Formula: $C_{30}H_{35}N_3O_3S$
Exact Mass: 517.23991
Molecular Weight: 517.68220

1-diazo-3-((1*S*,2*R*,3*R*)-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 μl). 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.

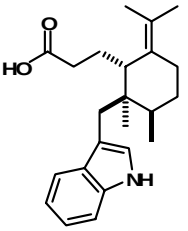
The crude acid chloride was dissolved in THF (1 mL), cooled to 0 °C, and treated sequentially with *i*-Pr_2EtNH (0.2 mL, 1.1 mmol) and diazomethane (6.6 mL, 2.2 mmol). The reaction mixture was warmed to 23 °C and maintained at this temperature for 30 min. The reaction mixture was poured into saturated aqueous NH_4Cl (5 mL) and extracted with Et_2O (3 × 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→20% ethyl acetate – hexanes) to yield the title compound as a white foam (70 mg, 62%): ^{1}H NMR (C_6D_6, 600 MHz): δ 8.54 (δ, *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):δ 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-((1*S*,2*R*,3*R*)-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 °C and protected from light. A solution of CF_3CO_2Ag (4.0 mg, 0.017 mmol) in Et_3N (60 μL, 0.41 mmol) was added in one portion and the reaction was warmed to 23 °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 × 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→50% ethyl acetate – hexanes) to yield the title compound as a white foam (50 mg, 74%): $[\alpha]^{22}_D$ = + 62° (c = 1.1, $CHCl_3$). ^{1}H NMR ($CDCl_3$, 600 MHz): δ 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):δ 193.56[4], 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, 24.7 5, 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}.

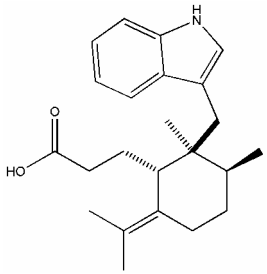


Chemical Formula: $C_{23}H_{31}NO_2$
Exact Mass: 353.24
Molecular Weight: 353.50

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 °C. After stirring for 90 m, the freshly prepared sodium napthalenide (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 °C. The reaction mixture was maintained at this temperature for 20 min, poured into 2M HCl (1 mL) and extracted with Et_2O (3 × 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 × 250 mm column, Silica 5μM; R_T = 6.6 min) to afford suaveolindole (**1,** 33 mg, 94%) as a white foam: $[\alpha]^{22}_D$ = + 32° (c = 1.0, CD_3OD). ^{1}H 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):
δ 178.22, 137.70, 132.35, 130.78, 126.23, 124.33, 121.80, 120.13, 119.16, 113.27, 111.92

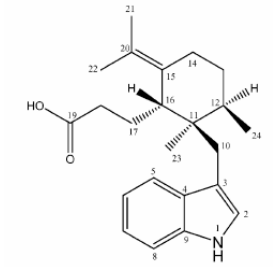
[4] Visible when LB = 10

, 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

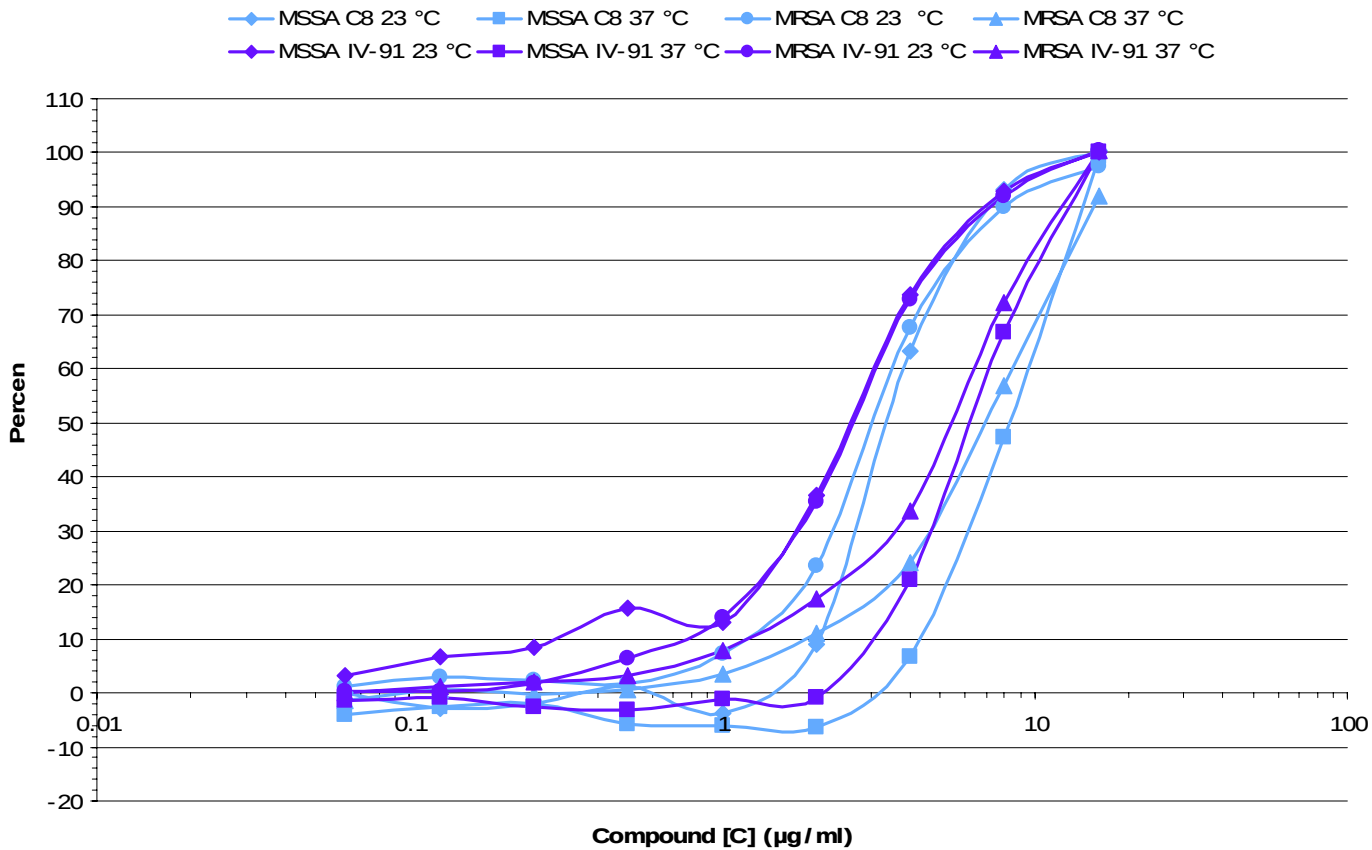
(synthetic)

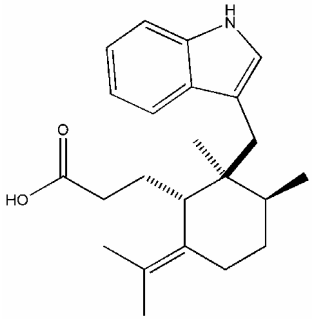


C8 – (natural product)

S **Suave4** 13

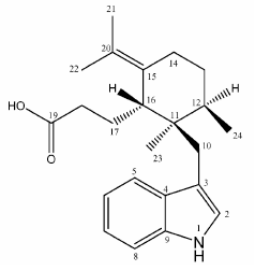
C8 and C8 precursors MIC on *Staphylococcus aureus* (Suave4)





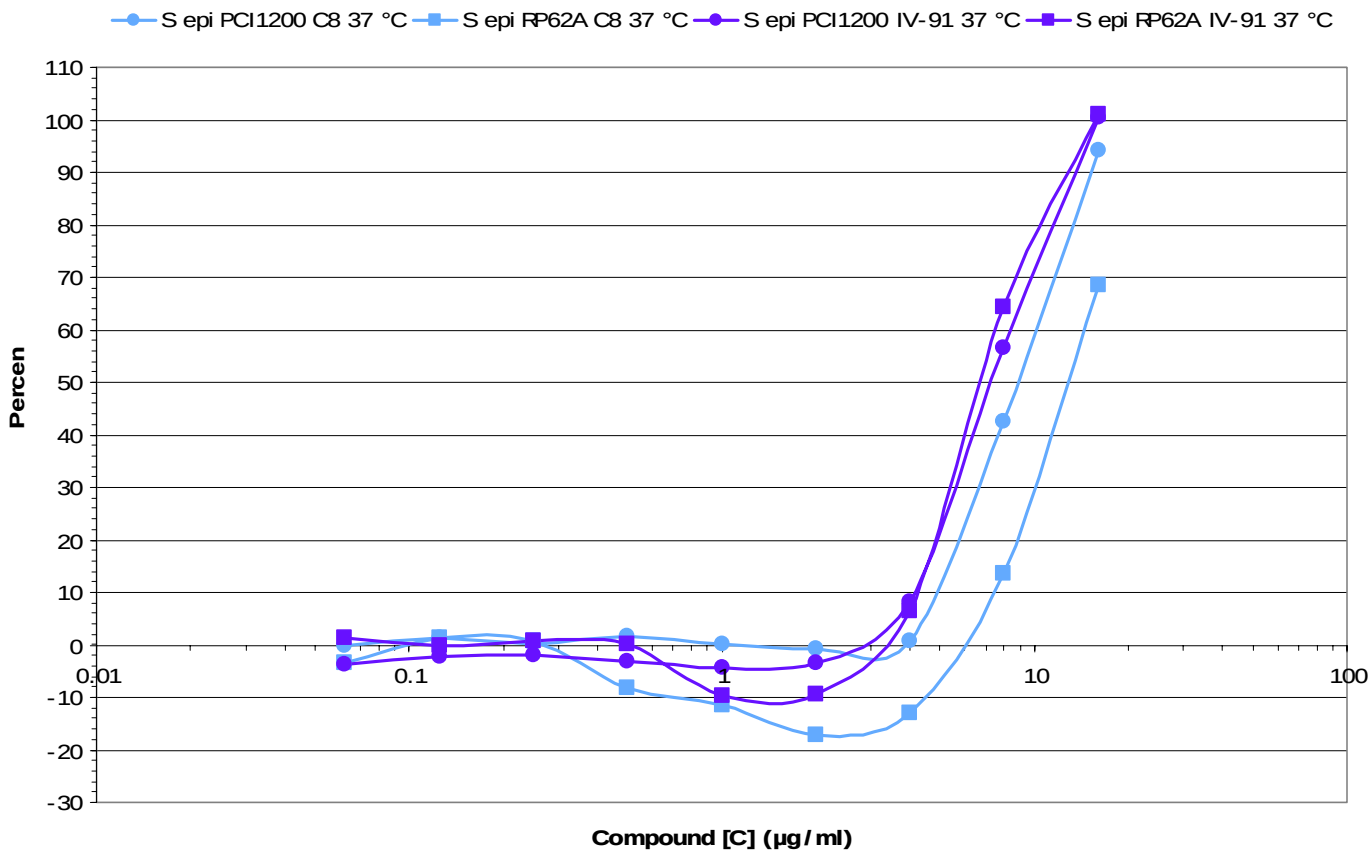
EJV-IV-91

(synthetic)

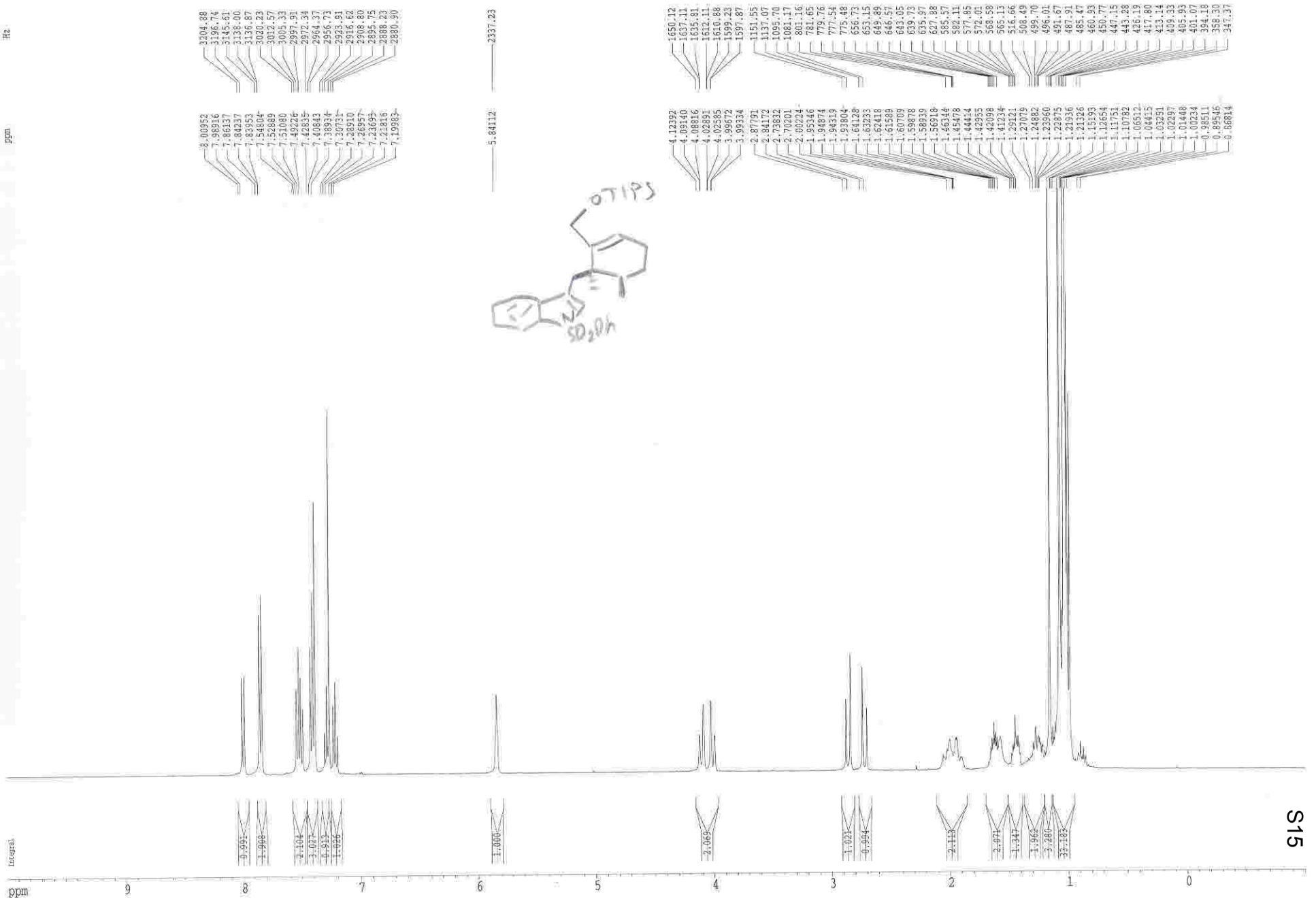


C8 – (natural product)

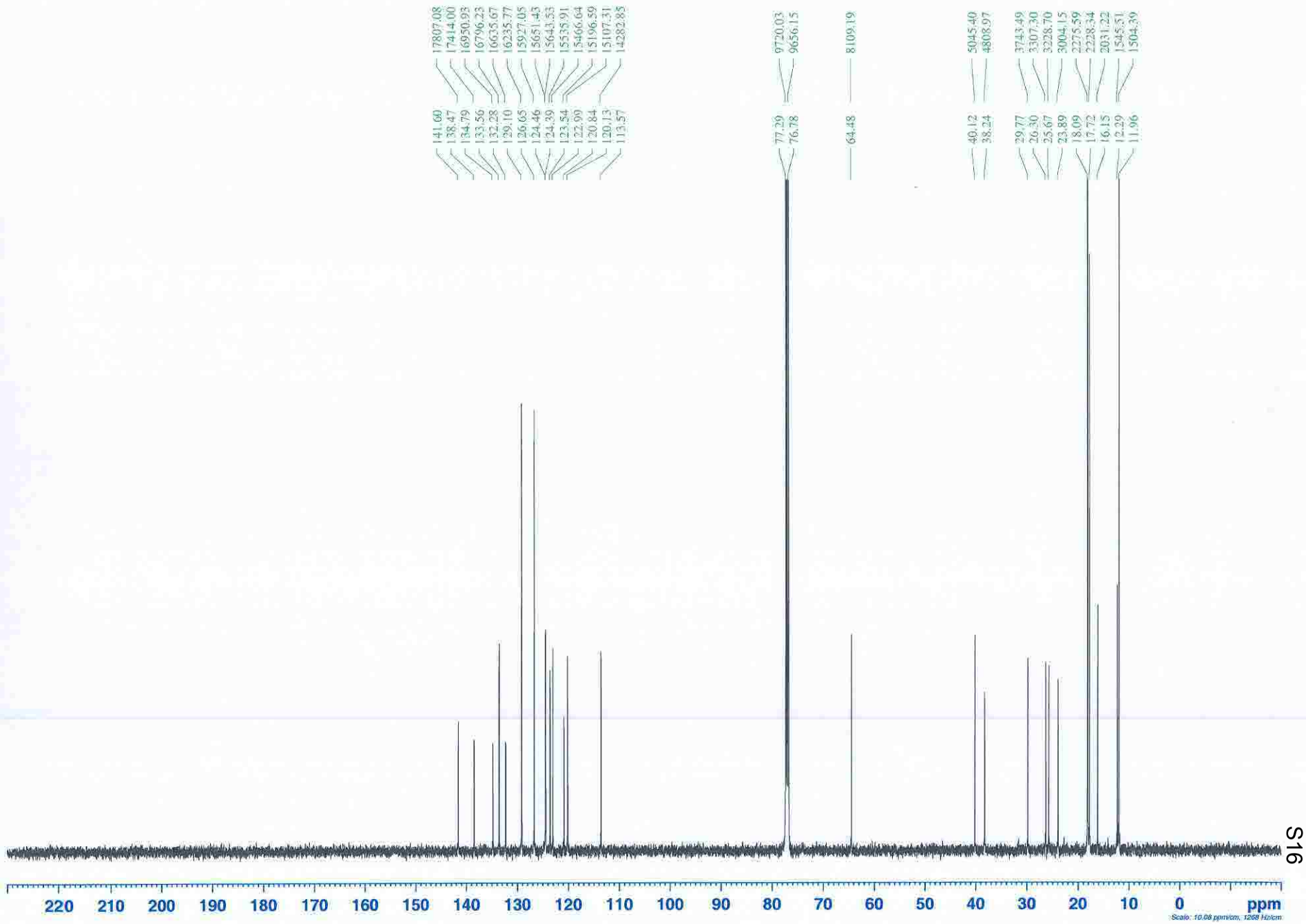
C8 and C8 precursors MIC on *Staphylococcus epidermidis* (Suave4)

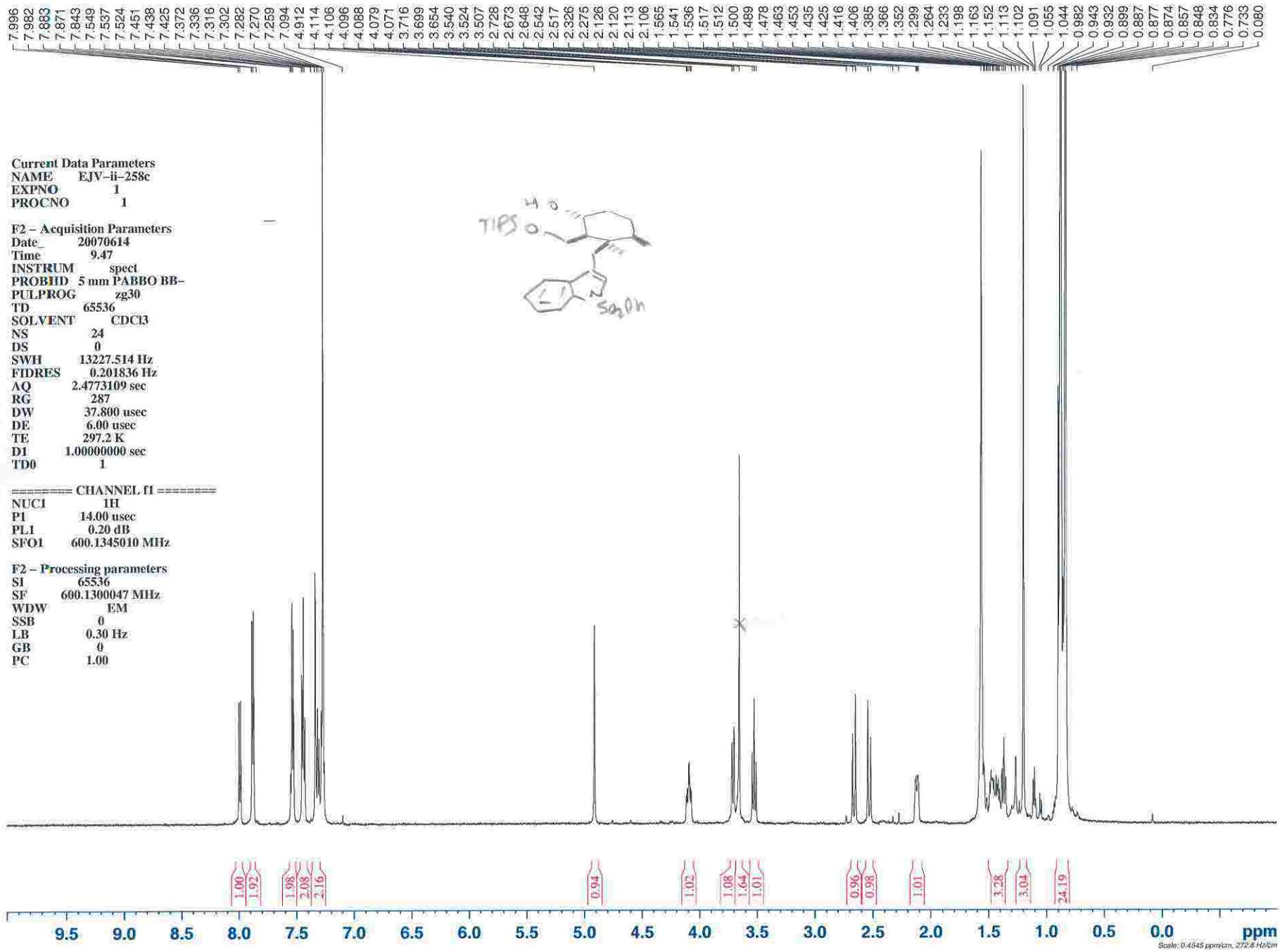


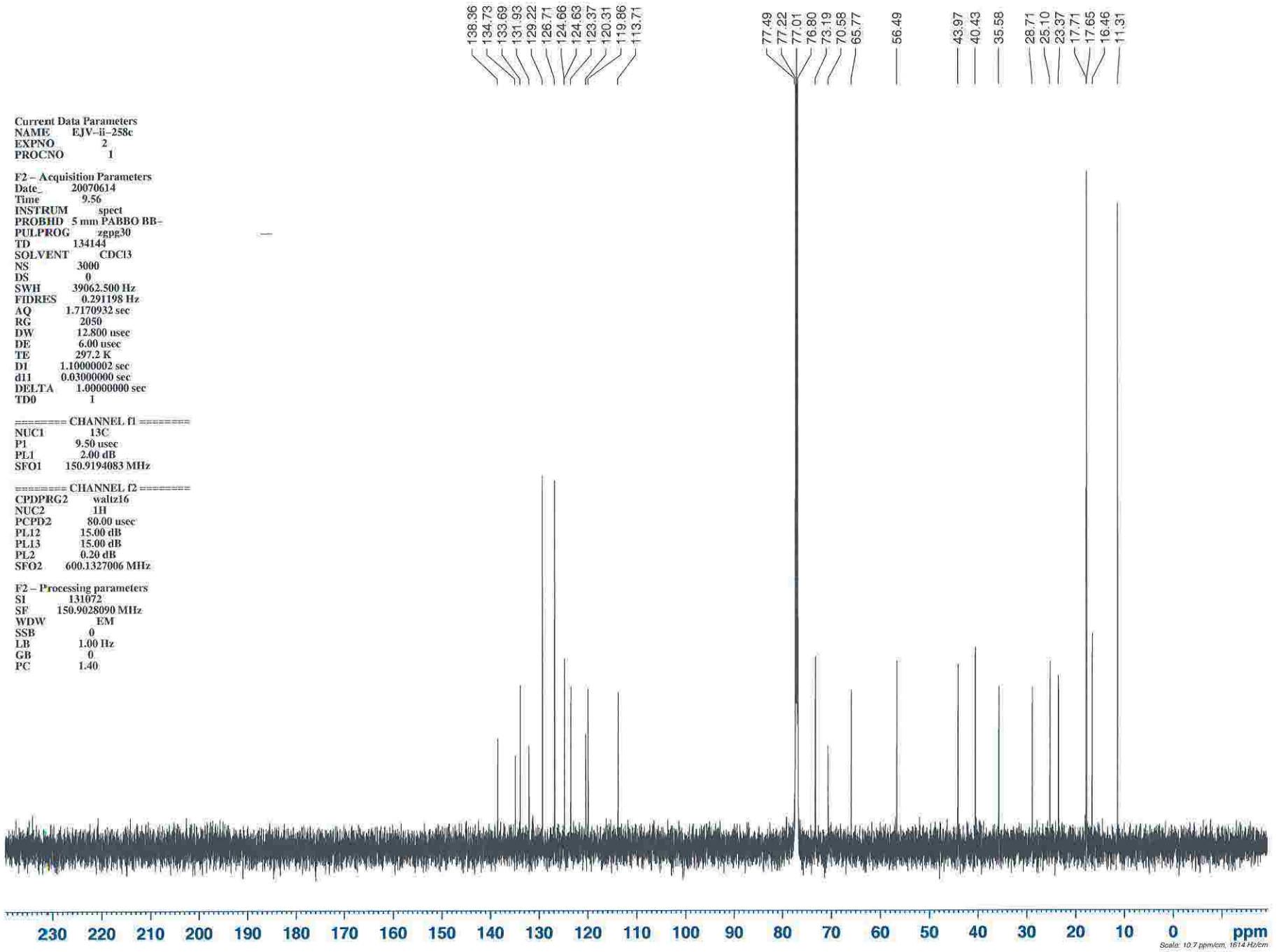
Proton .* evelthui ejv-ii-151f (1 1) CDCl3 25.0C September_07,2006_08:50 AMX 400MHz zg30 1H *.



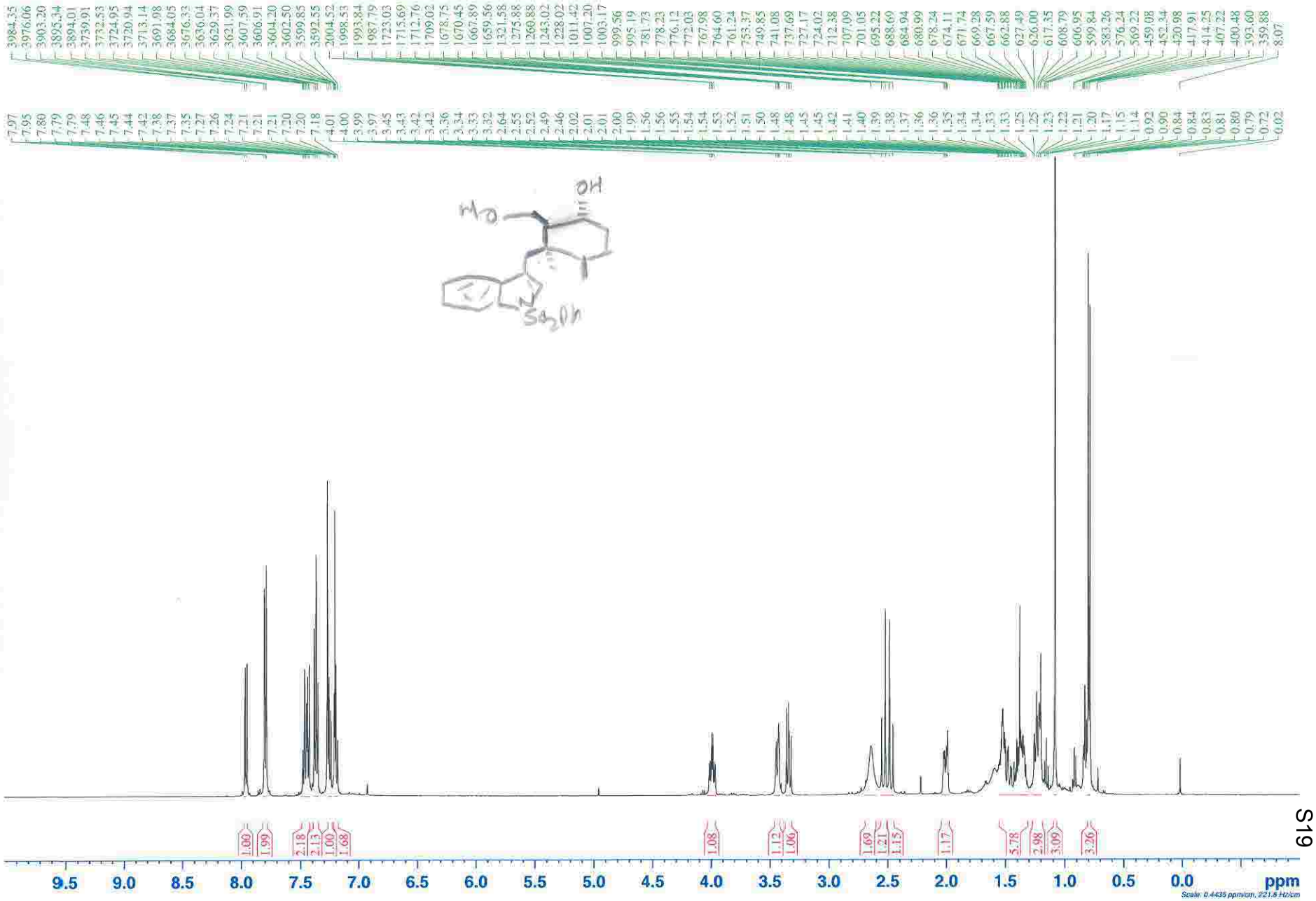
Carbon .* evelthui ejv-ii-151_c13 (1 1) CDCl3 24.0C September_09,2006_11:07 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.

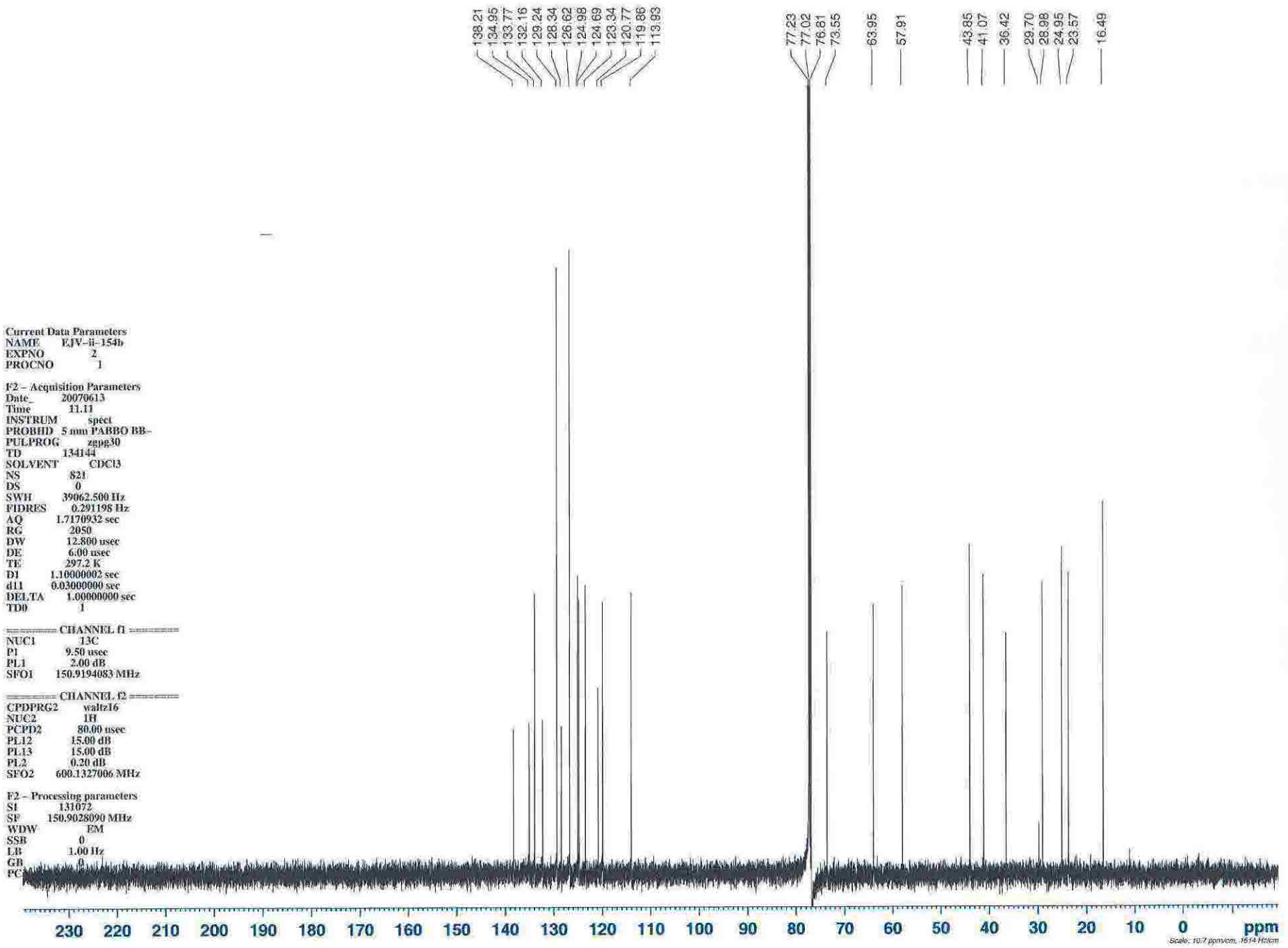


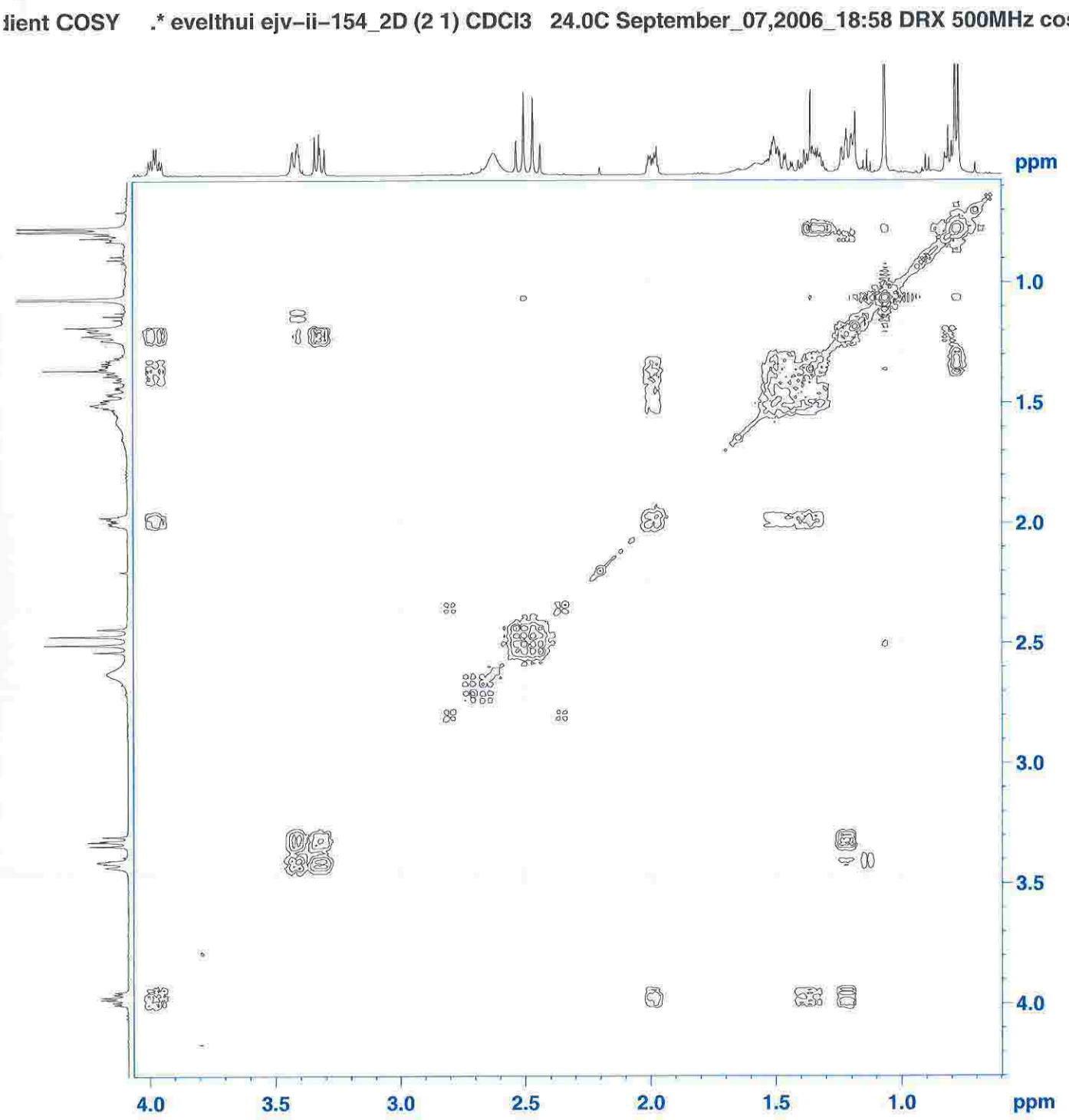

7.996
7.982
7.883
7.871
7.843
7.549
7.537
7.524
7.451
7.438
7.425
7.372
7.336
7.316
7.302
7.282
7.270
7.259
7.094
4.912
4.114
4.106
4.096
4.088
4.079
4.071
3.716
3.699
3.654
3.540
3.524
3.507
2.728
2.673
2.648
2.542
2.517
2.326
2.275
2.126
2.120
2.113
2.106
1.565
1.541
1.536
1.517
1.512
1.500
1.489
1.478
1.463
1.453
1.435
1.425
1.416
1.406
1.385
1.366
1.352
1.299
1.264
1.233
1.198
1.163
1.152
1.113
1.102
1.091
1.055
1.044
0.982
0.943
0.932
0.899
0.887
0.877
0.874
0.857
0.848
0.834
0.776
0.733
0.080
Current Data Parameters
NAME EJV–ii–258c
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20070614
Time 9.47
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 287
DW 37.800 usec
DE 6.00 usec
TE 297.2 K
D1 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 600.1300047 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TIPS
SO2Ph
1.00
1.92
1.98
2.08
2.16
0.94
1.02
1.08
1.64
1.01
0.96
0.98
1.01
3.28
3.04
24.19
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4545 ppm/cm, 272.8 Hz/cm


138.36
134.73
133.69
131.93
129.22
126.71
124.66
124.63
123.37
120.31
119.86
113.71
77.49
77.22
77.01
76.80
73.19
70.58
65.77
56.49
43.97
40.43
35.58
28.71
25.10
23.37
17.71
17.65
16.46
11.31
Current Data Parameters
NAME EJV-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, 1614 Hz/cm

Proton24 .* evelthui ejv-ii-154_2D (1 1) CDCl3 24.0C September_07,2006_18:56 DRX 500MHz zg30 1H *.




138.21
134.95
133.77
132.16
129.24
128.34
126.62
124.98
124.69
123.34
120.77
119.86
113.93
77.23
77.02
76.81
73.55
63.95
57.91
43.85
41.07
36.42
29.70
28.98
24.95
23.57
16.49
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 0.291198 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
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, 1614 Hz/cm



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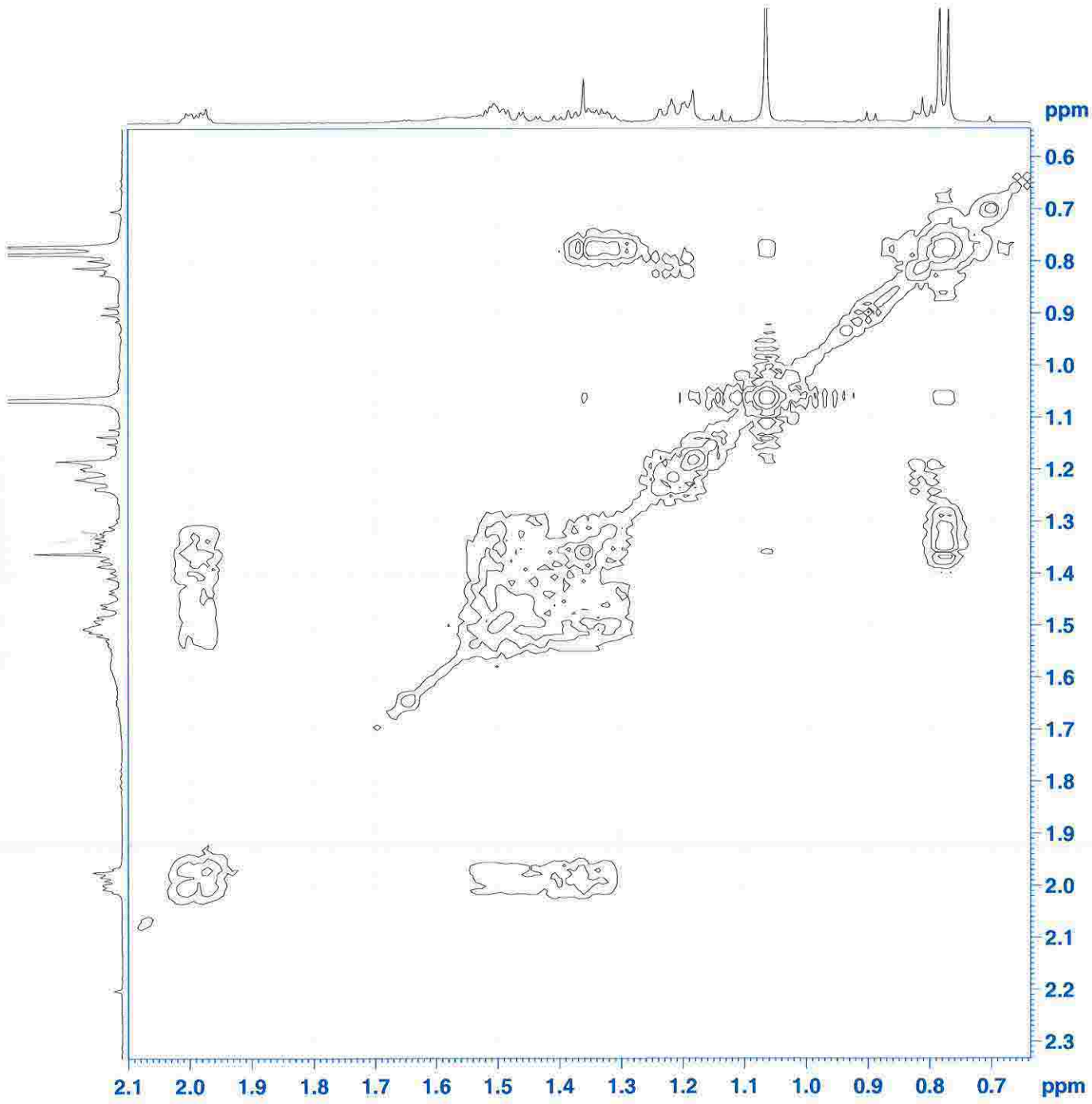
NMR Analytical Core Facility
Rockefeller Research Laboratories
430 E 67th St
New York, NY 10021

```
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                      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
d0                      0.00000300 sec
D1                      0.89841843 sec
d13                     0.00000400 sec
D16                     0.00020000 sec
IN0                     0.00024960 sec

======== CHANNEL f1 ========
NUC1                           1H
P0                         14.00 usec
P1                         14.00 usec
PL1                        -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
ND0                              1
TD                             256
SFO1                 500.1322 MHz
FIDRES              15.650040 Hz
SW                       8.011 ppm
FnMODE                        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
MC2                           QF
SF                   500.1300483 MHz
WDW                         SINE
SSB                            0
LB                          0.00 Hz
GB                             0
```



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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	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
d0	0.00000300	sec
D1	0.89841843	sec
d13	0.00000400	sec
D16	0.00020000	sec
IN0	0.00024960	sec

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

NUC1	1H	
P0	14.00	usec
P1	14.00	usec
PL1	-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

ND0	1	
TD	256	
SFO1	500.1322	MHz
FIDRES	15.650040	Hz
SW	8.011	ppm
FnMODE	QF	

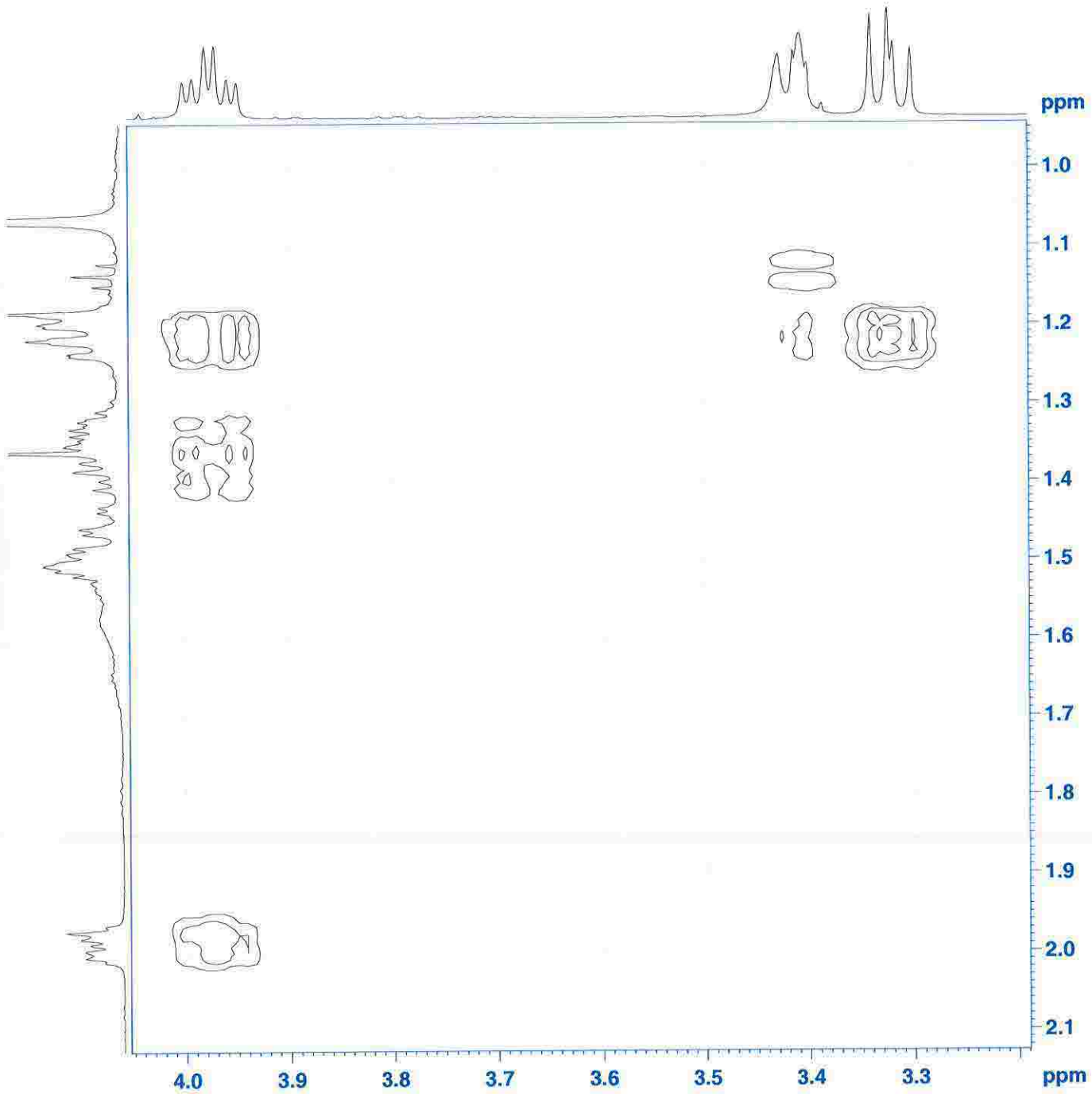
F2 - Processing parameters

SI	1024	
SF	500.1300483	MHz
WDW	SINE	
SSB	0	
LB	0.00	Hz
GB	0	
PC	1.00	

F1 - Processing parameters

SI	1024	
MC2	QF	
SF	500.1300483	MHz
WDW	SINE	
SSB	0	
LB	0.00	Hz
GB	0	

lient COSY .* evelthui ejv-ii-154_2D (2 1) CDCl3 24.0C September_07,2006_18:58 DRX 500MHz cosygpqf 1



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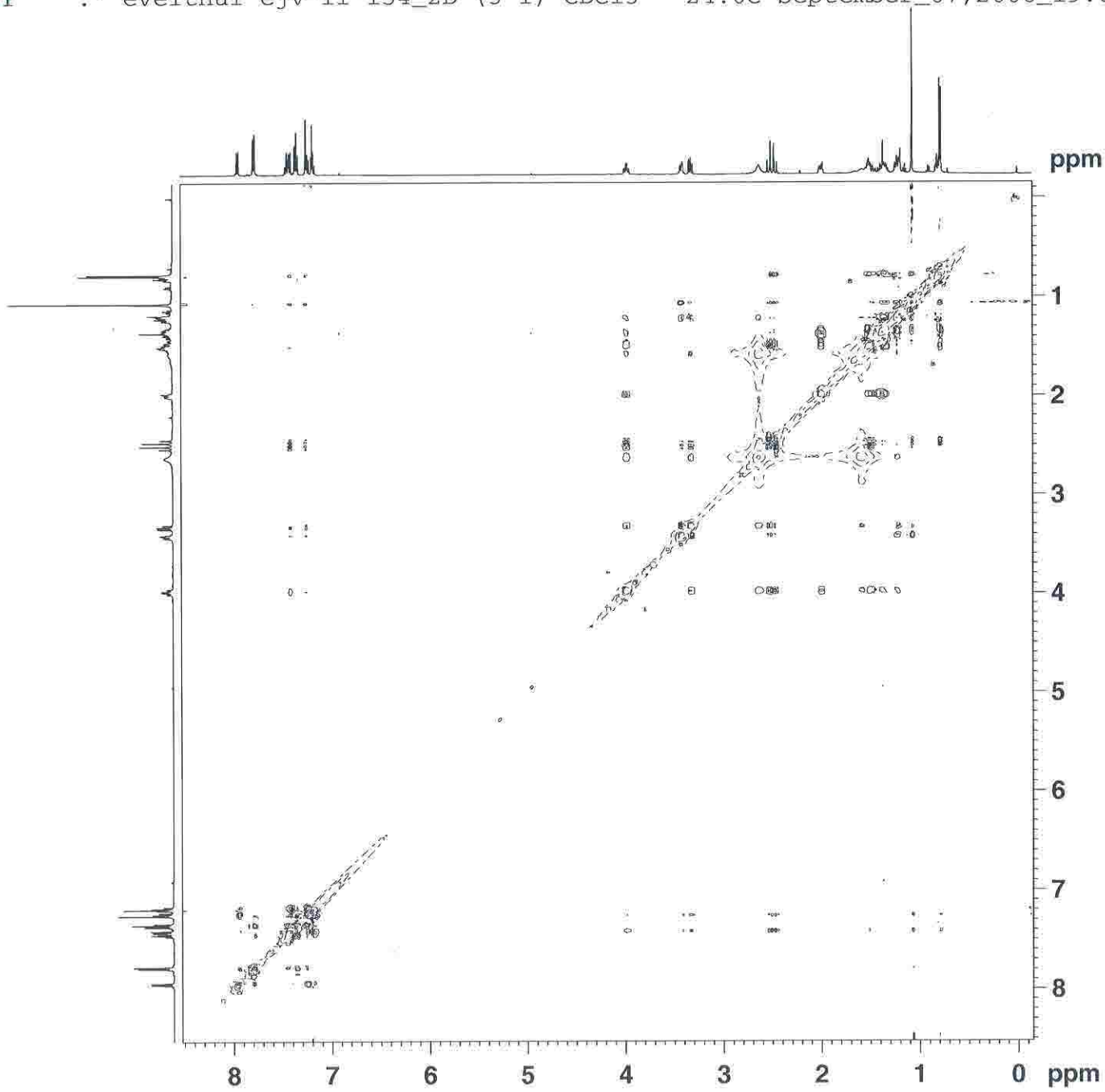
New York, NY 10021


```
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                     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
d0                    0.00000300 sec
D1                    0.89841843 sec
d13                   0.00000400 sec
D16                   0.00020000 sec
IN0                   0.00024960 sec

======== CHANNEL f1 ========
NUC1                          1H
P0                         14.00 usec
P1                         14.00 usec
PL1                        -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
ND0                              1
TD                             256
SFO1                 500.1322 MHz
FIDRES                15.650040 Hz
SW                        8.011 ppm
FnMODE                       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
MC2                          QF
SF                 500.1300483 MHz
WDW                         SINE
SSB                            0
LB                          0.00 Hz
GB                             0
```

NOESY .* evelthui ejv-ii-154_2D (3 1) CDCl3 24.0C September_07,2006_19:08



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	0.00009737 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
FnMODE	States-TPPI

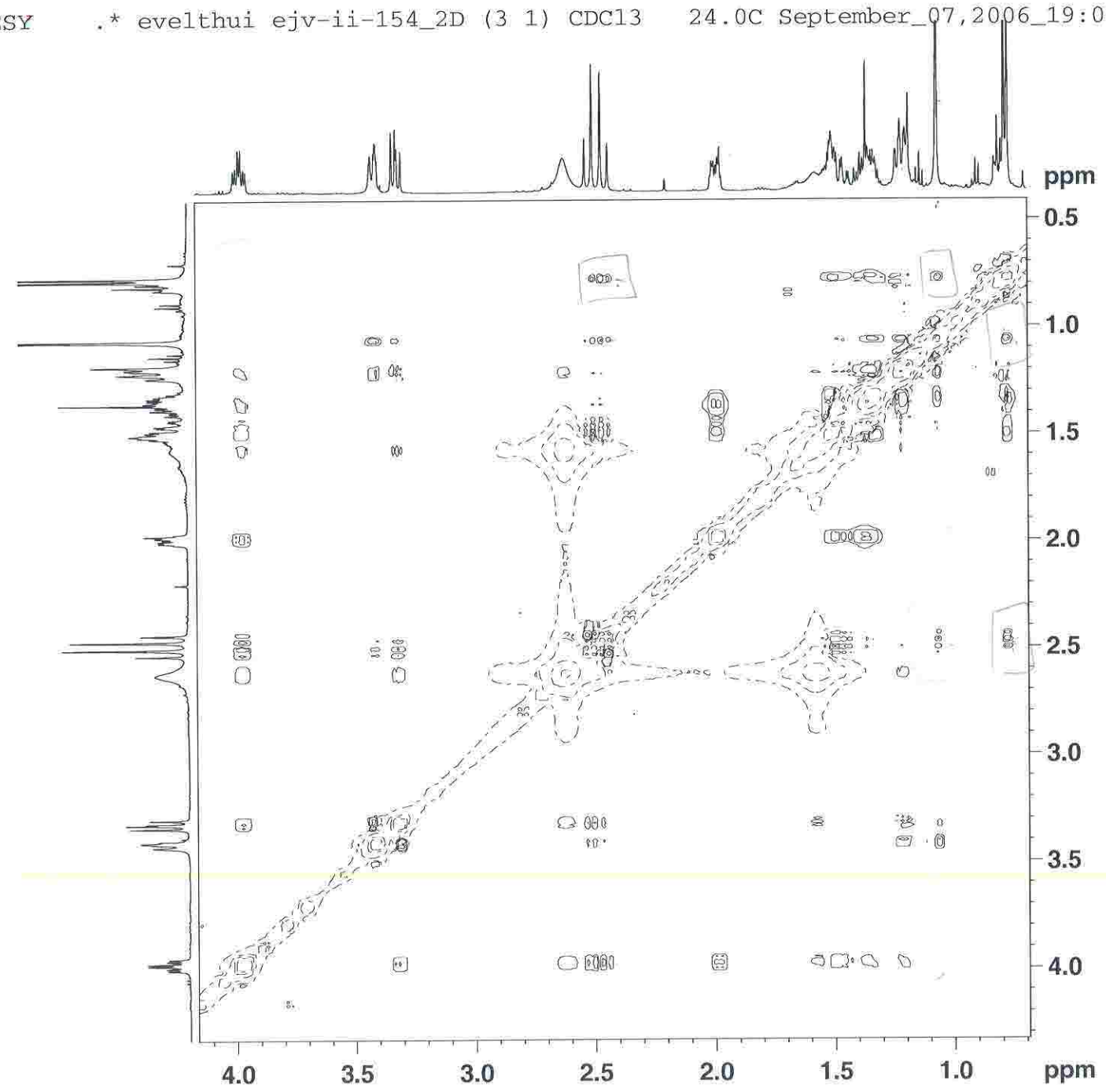
F2 - Processing parameters

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

NOESY .* evelthui ejv-ii-154_2D (3 1) CDCl3 24.0C September_07,2006_19:08



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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 0.00009737 sec
D1 1.96436405 sec
D8 0.44999999 sec
IN0 0.00023040 sec
ST1CNT 128

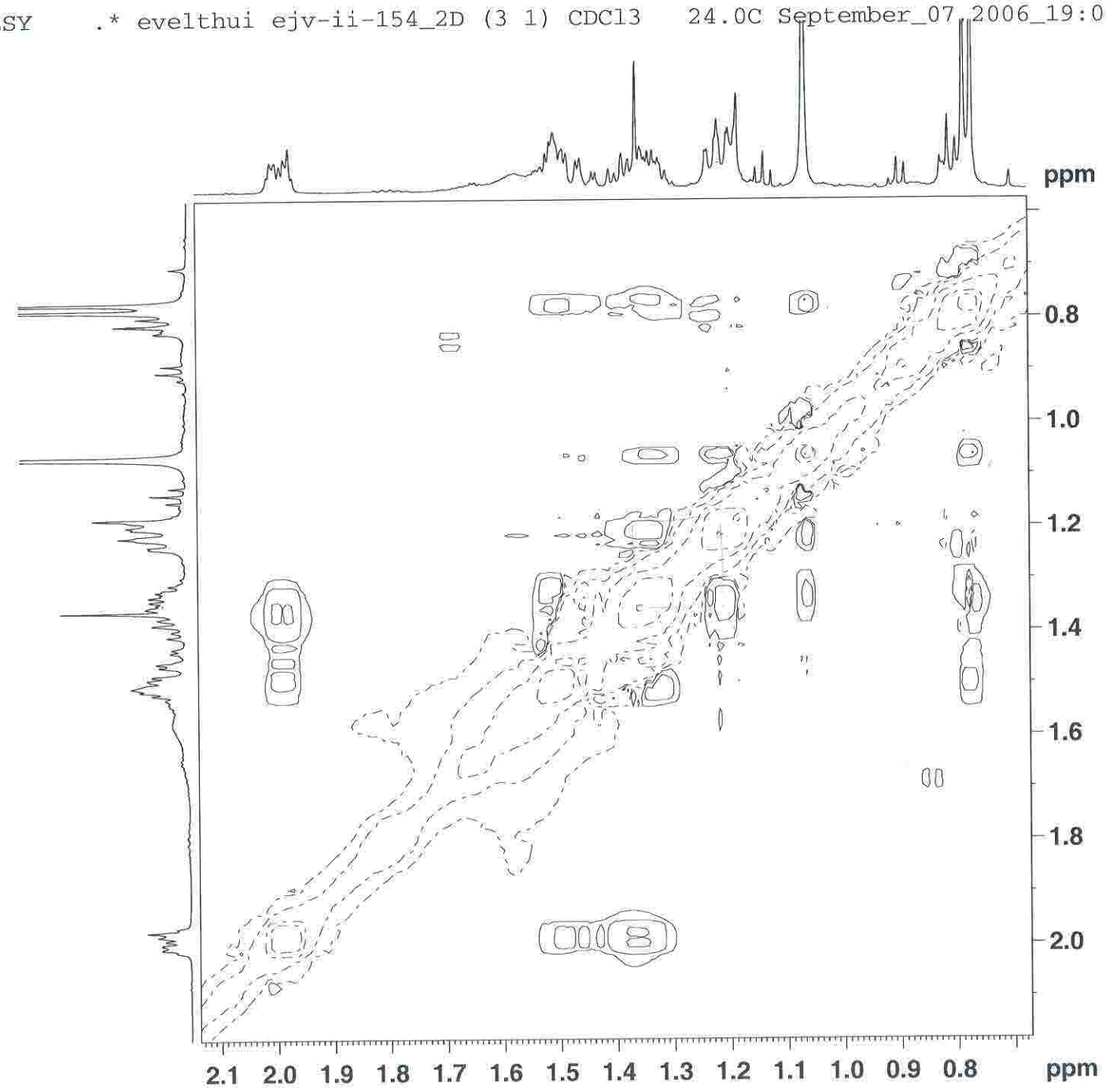
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
SFO1 500.1321423 MHz

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

NOESY .* evelthui ejv-ii-154_2D (3 1) CDCl3 24.0C September_07 2006_19:08



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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	0.00009737 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
FnMODE	States-TPPI

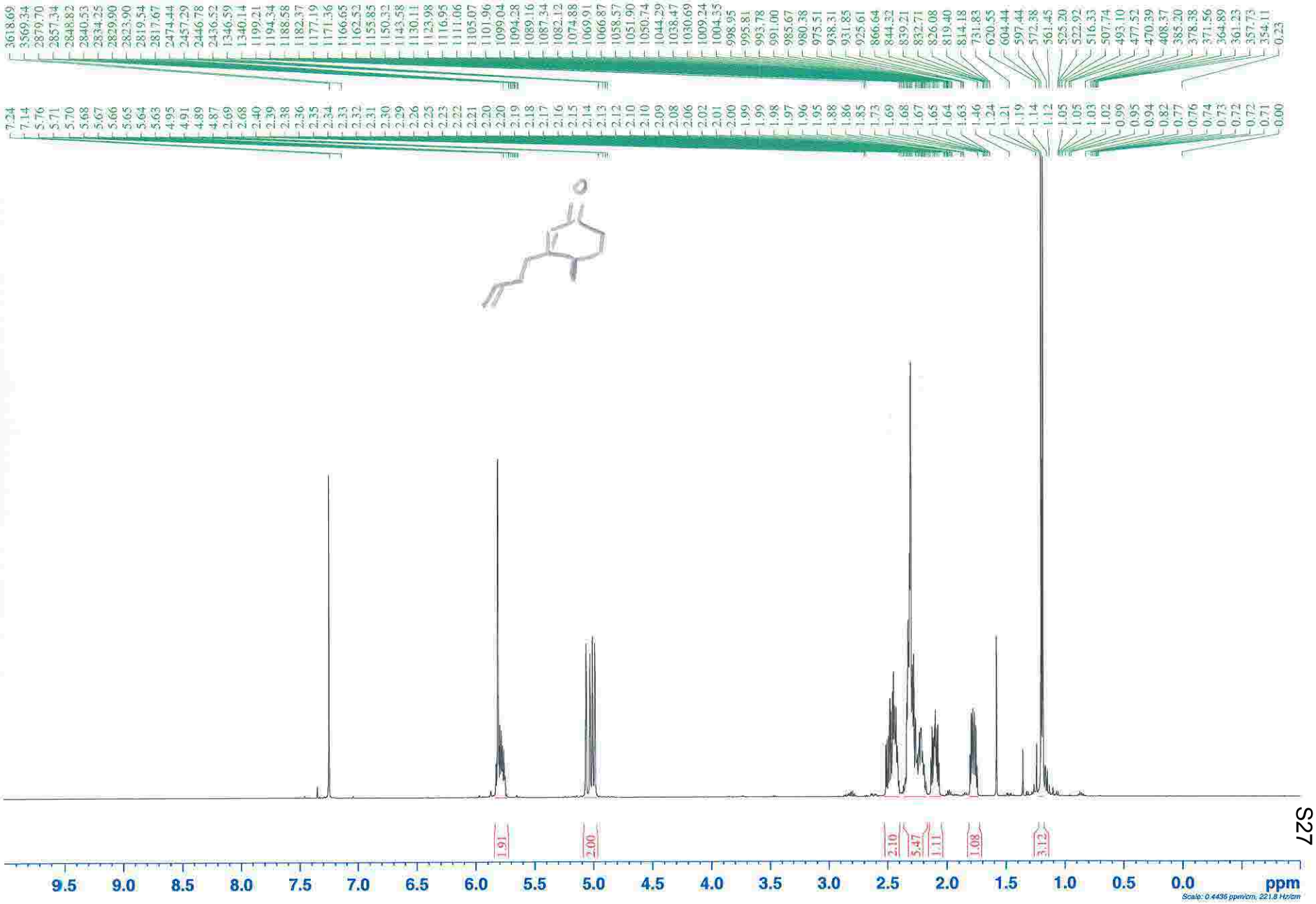
F2 - Processing parameters

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

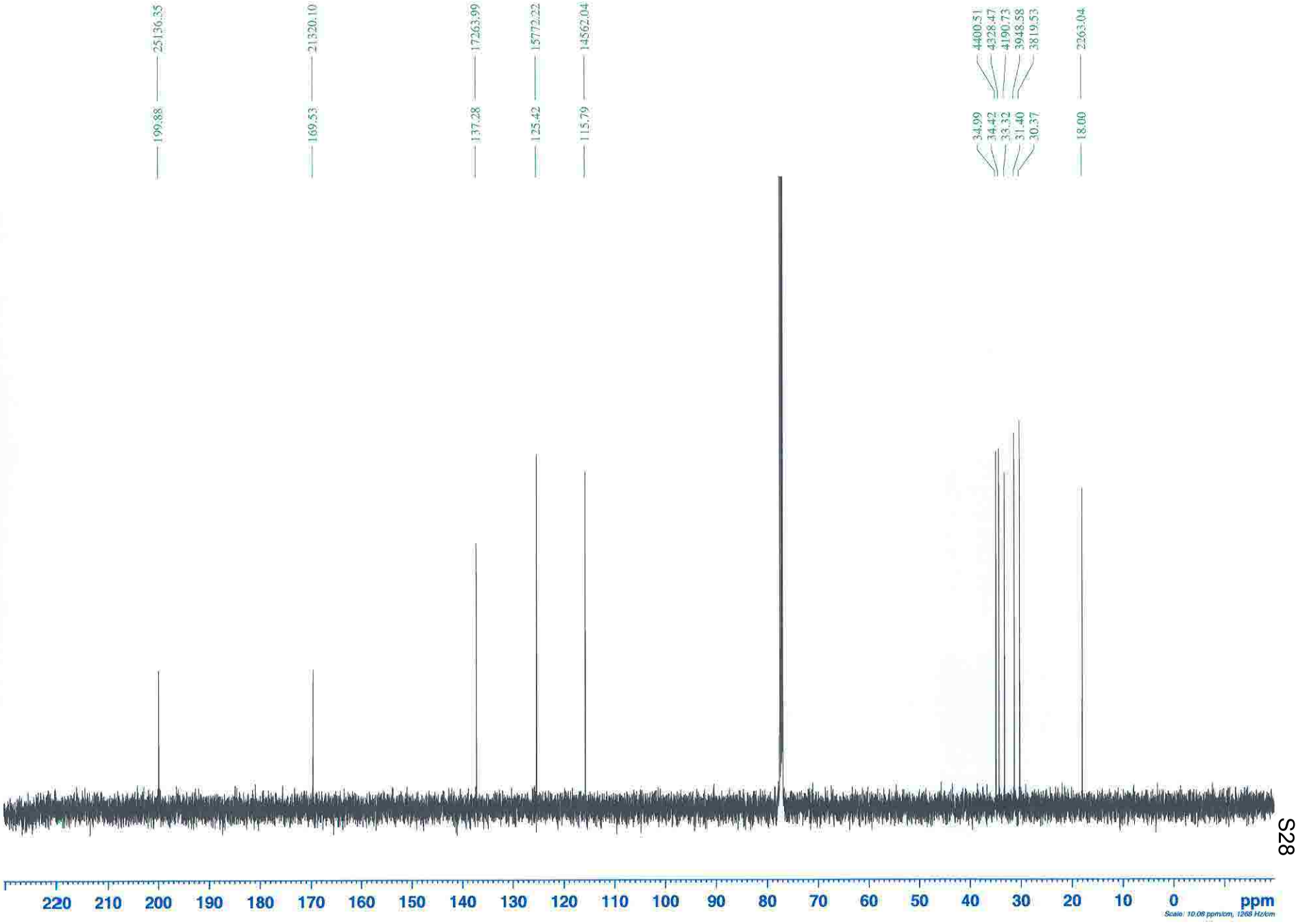
F1 - Processing parameters

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

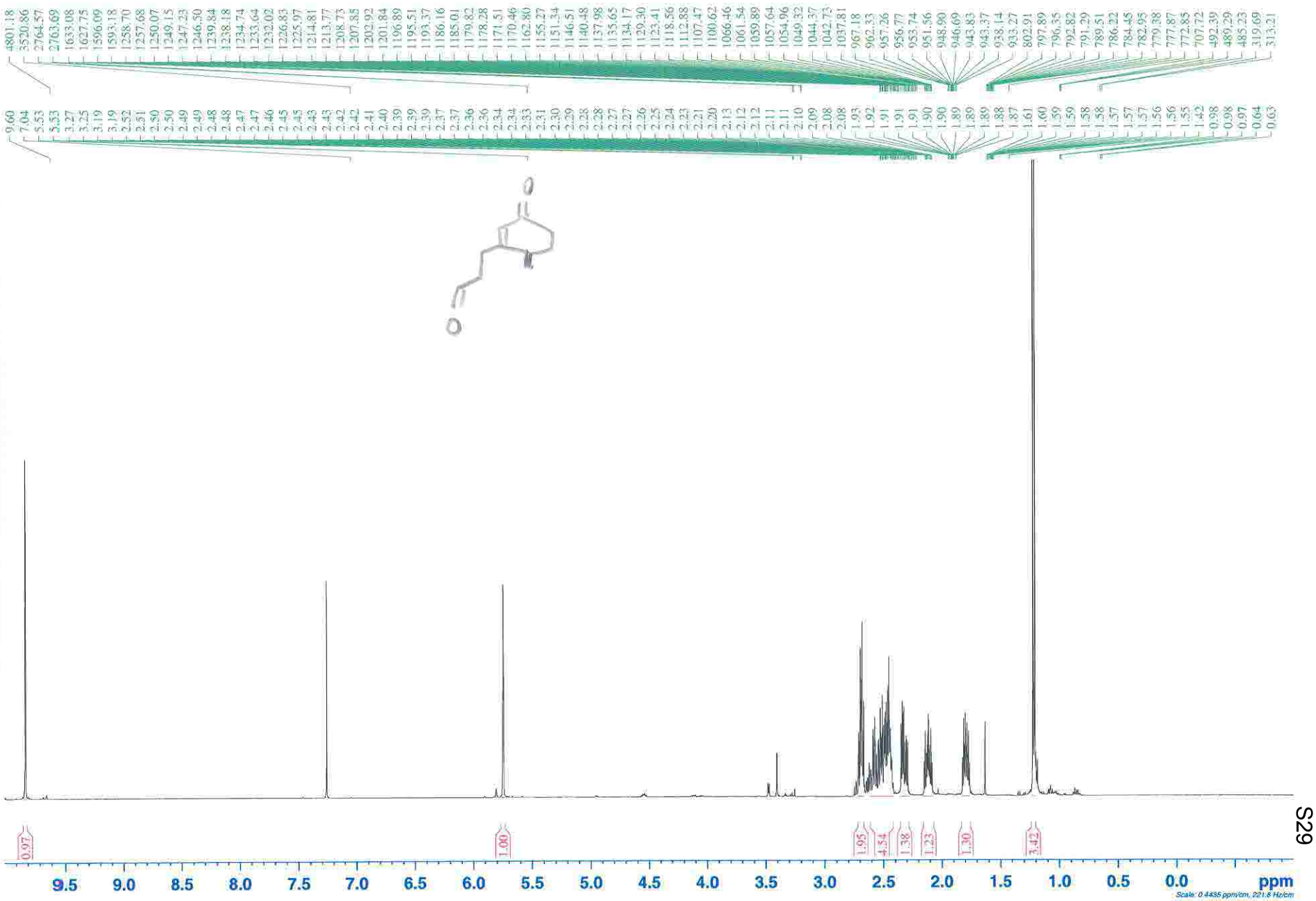
Proton24 .* evelthui ejv-iii-240 (1 1) CDCl3 24.0C February_23,2007_15:50 DRX 500MHz zg30 1H *.



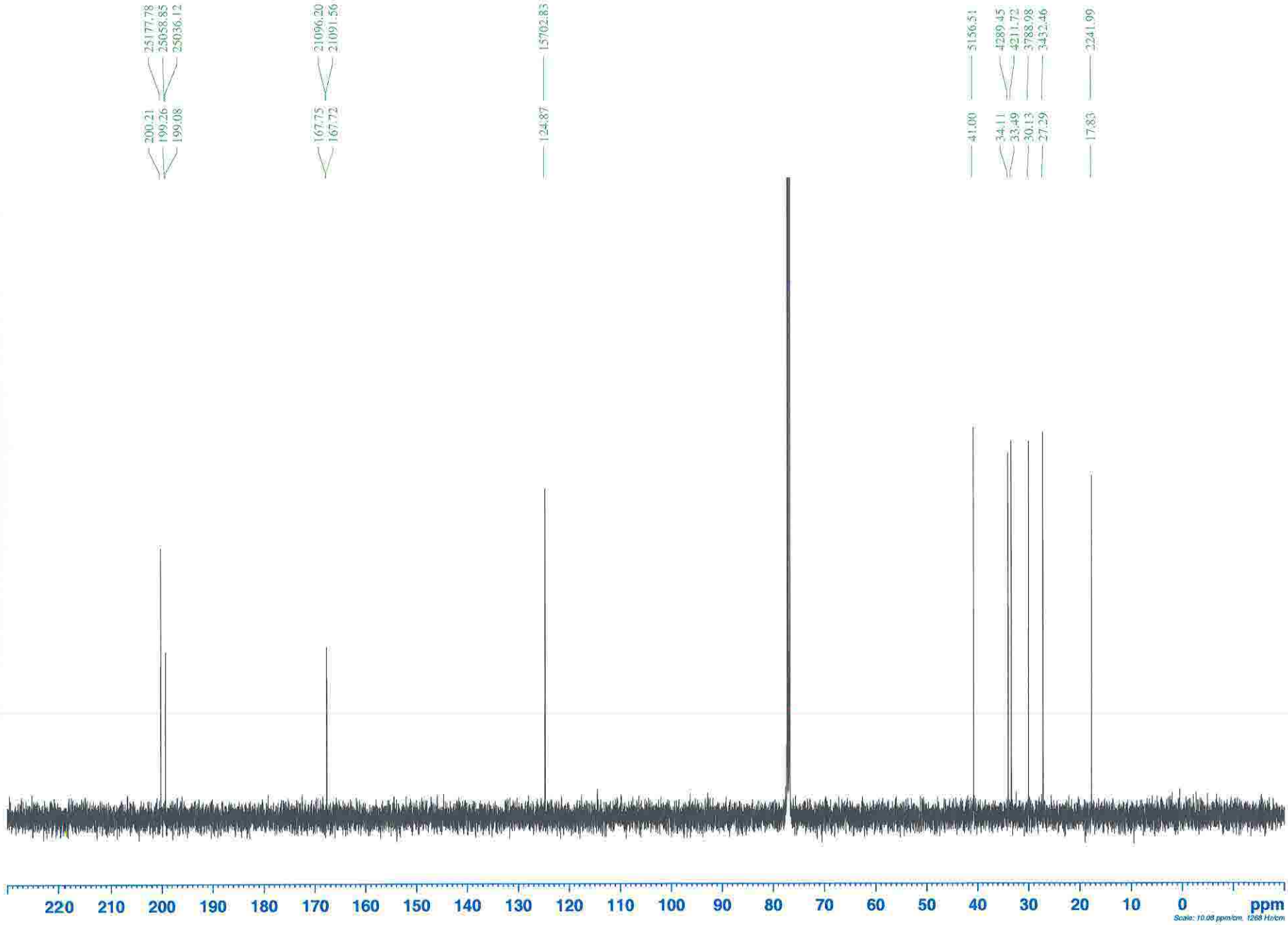
Carbon .* evelthui ejv-iii-240_1H (1 1) CDCl3 24.0C February_23,2007_15:55 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.



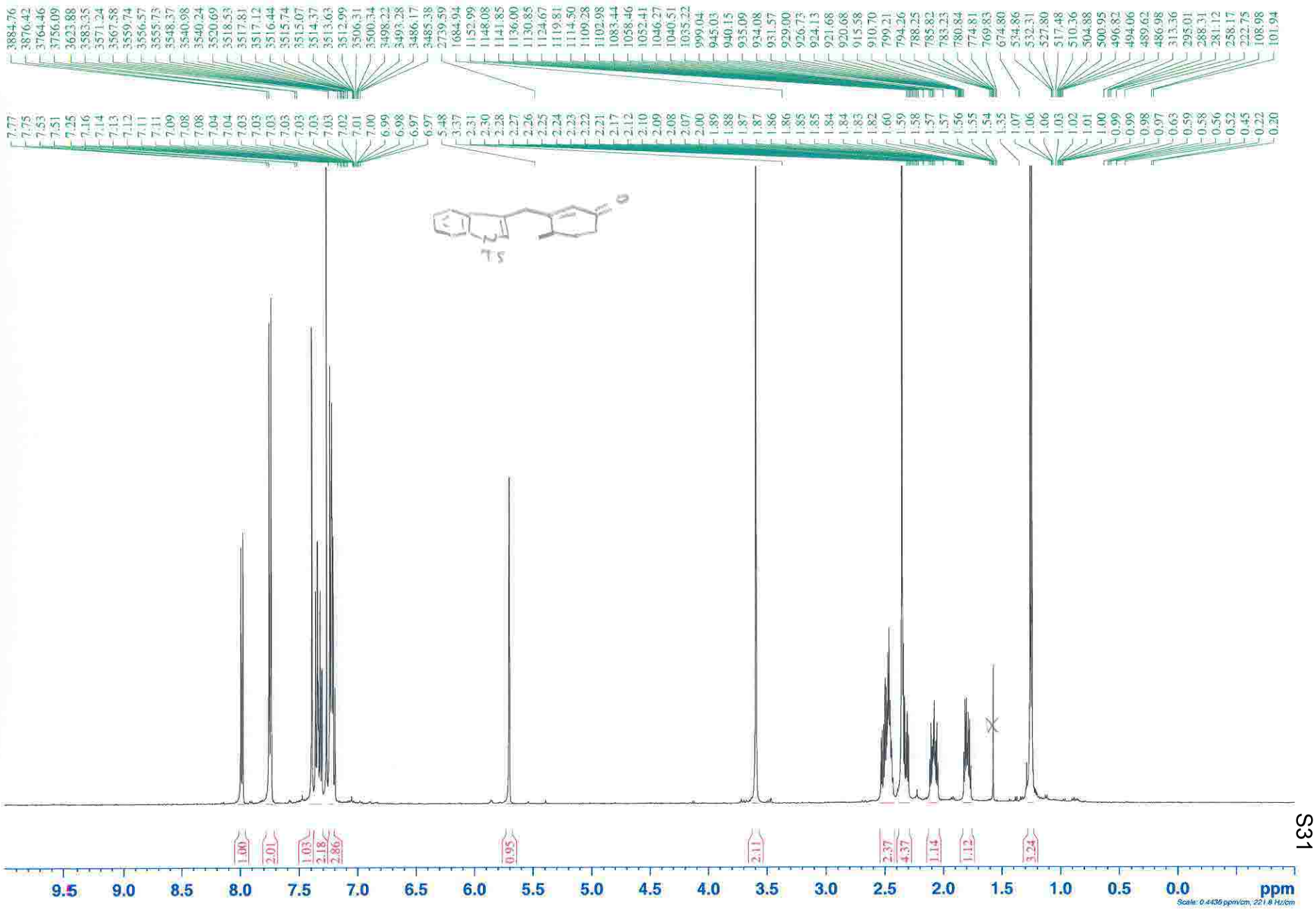
Proton24 .* evelthui ejv-iii-222_1H (1 1) CDCl3 24.0C February_14,2007_10:20 DRX 500MHz zg30 1H *.



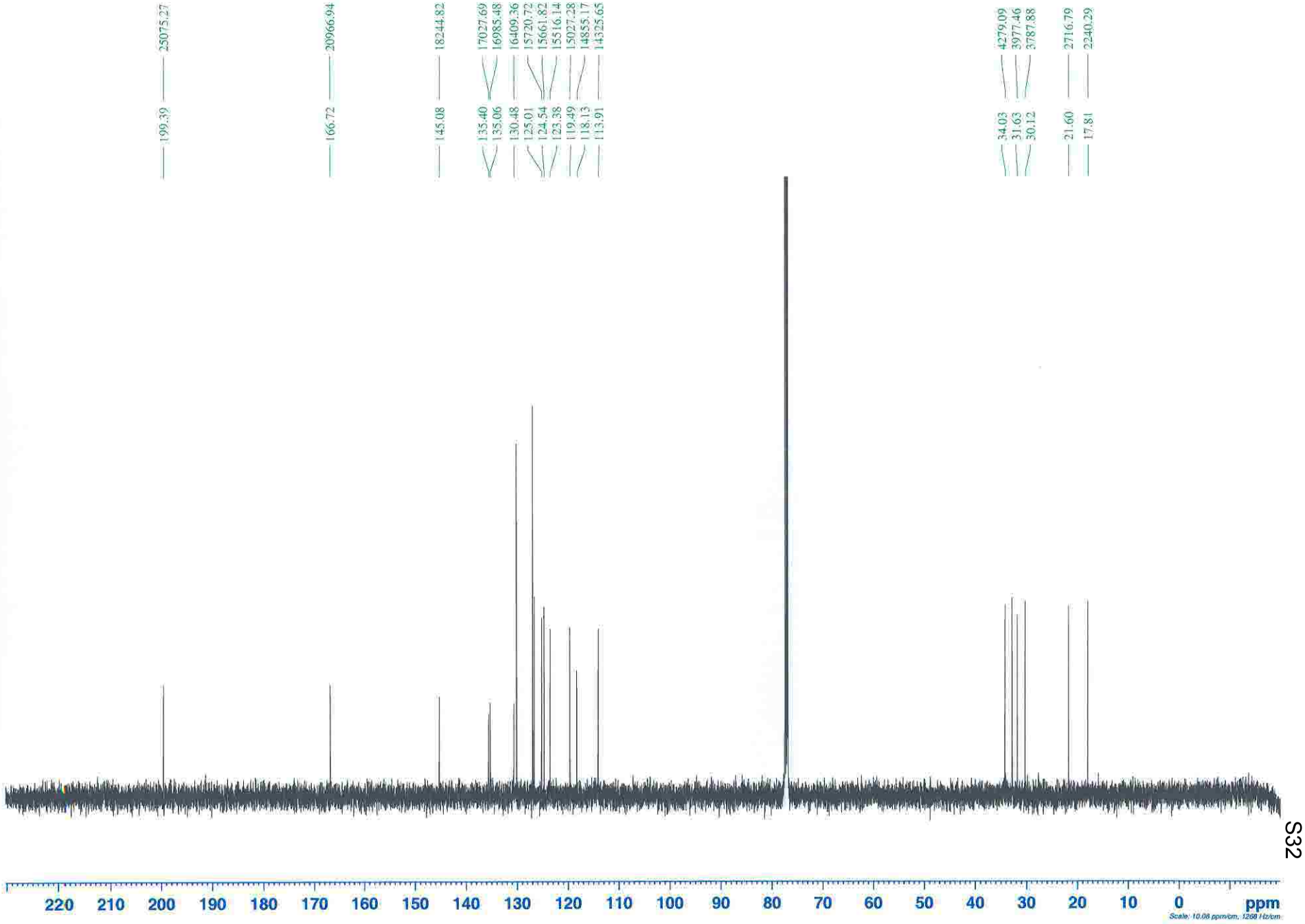
Carbon .* evelthui ejv-iii-222_c13 (1 1) CDCl3 24.0C February_14,2007_10:27 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.



Proton24 .* evelthui ejv-iii-228_1H (1 1) CDCl3 24.0C February_19,2007_07:50 DRX 500MHz zg30 1H *.

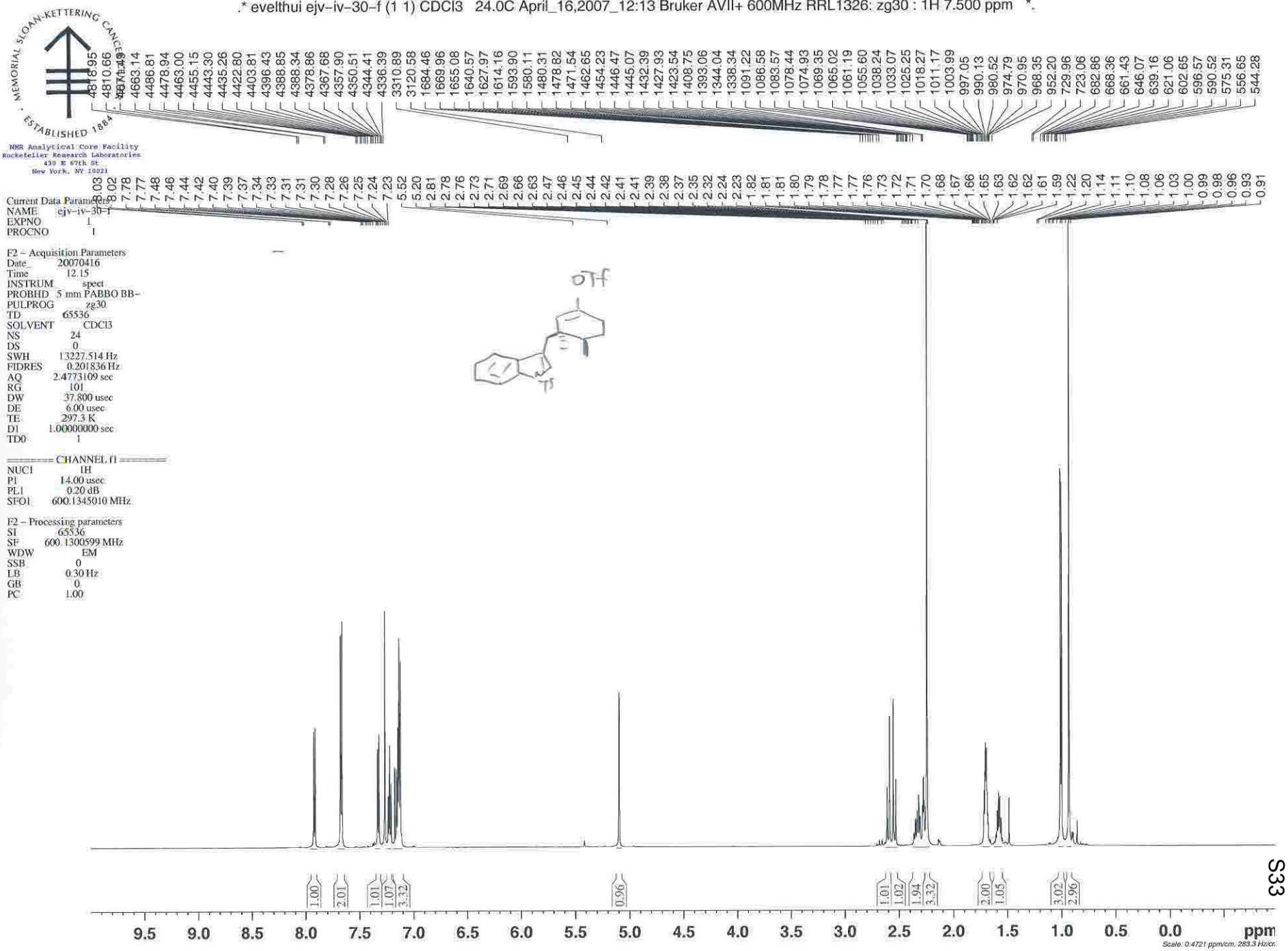


Carbon .* evelthui ejv–iii–228_13C (1 1) CDCl3 24.0C February_19,2007_07:54 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.



Proton

.* evelthui ejv-iv-30-f (1 1) CDCl3 24.0C April_16,2007_12:13 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm *.



Current Data Parameters
NAME ejv-iv-30-f
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20070416
Time 12.15
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 101
DW 37.800 usec
DE 6.00 usec
TE 297.3 K
D1 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 600.1300599 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Carbon .* evelthui ejv-iv-30-f (2 1) CDCl3 24.0C April_16,2007_12:15 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.

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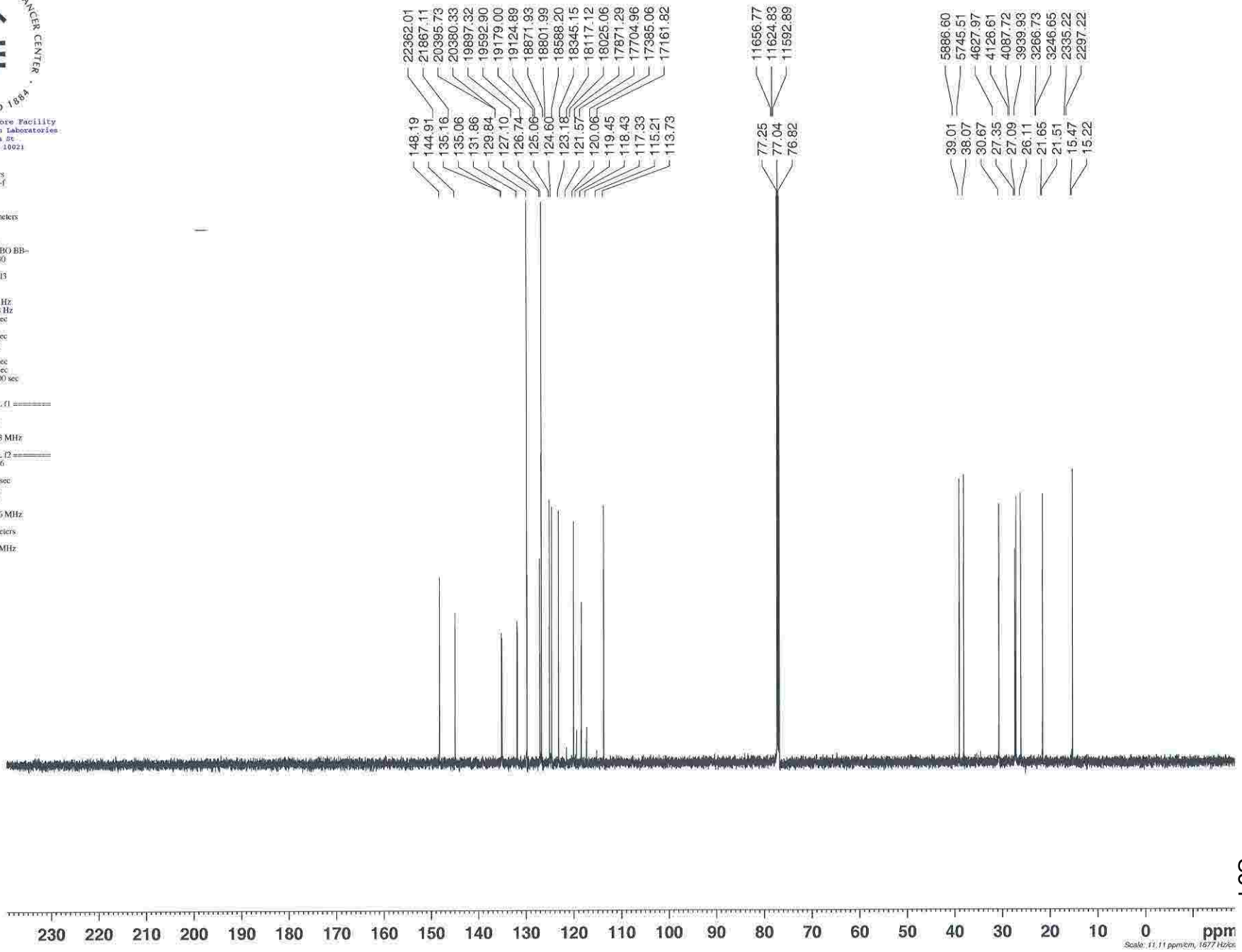
Current Data Parameters
NAME ejv-iv-30-f
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070416
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 134144
SOLVENT CDCl3
NS 202
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



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 *.
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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 37.800 usec
DE 6.00 usec
TE 297.2 K
D1 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 600.1300491 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
CO2Me
1.08
1.98
1.08
1.41
3.32
0.09
1.00
2.98
2.07
1.09
3.35
1.06
1.12
2.28
3.15
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4721 ppm/cm, 283.3 Hz/cm

Carbon .* evelthui ejv-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 *.

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Current Data Parameters
NAME ejv-iv-17-13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070331
Time 14.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 134144
SOLVENT CDCl3
NS 405
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 150.9027818 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

25347.68 22310.11 22298.71 21841.74 20397.82 20379.17 20349.60 19943.97 19930.95 19587.90 19485.19 19396.76 19136.20 19108.76 18871.18 18835.50 18775.23 18558.35 18543.23 18160.43 18081.81 17986.69 17958.71 17156.43 17145.12

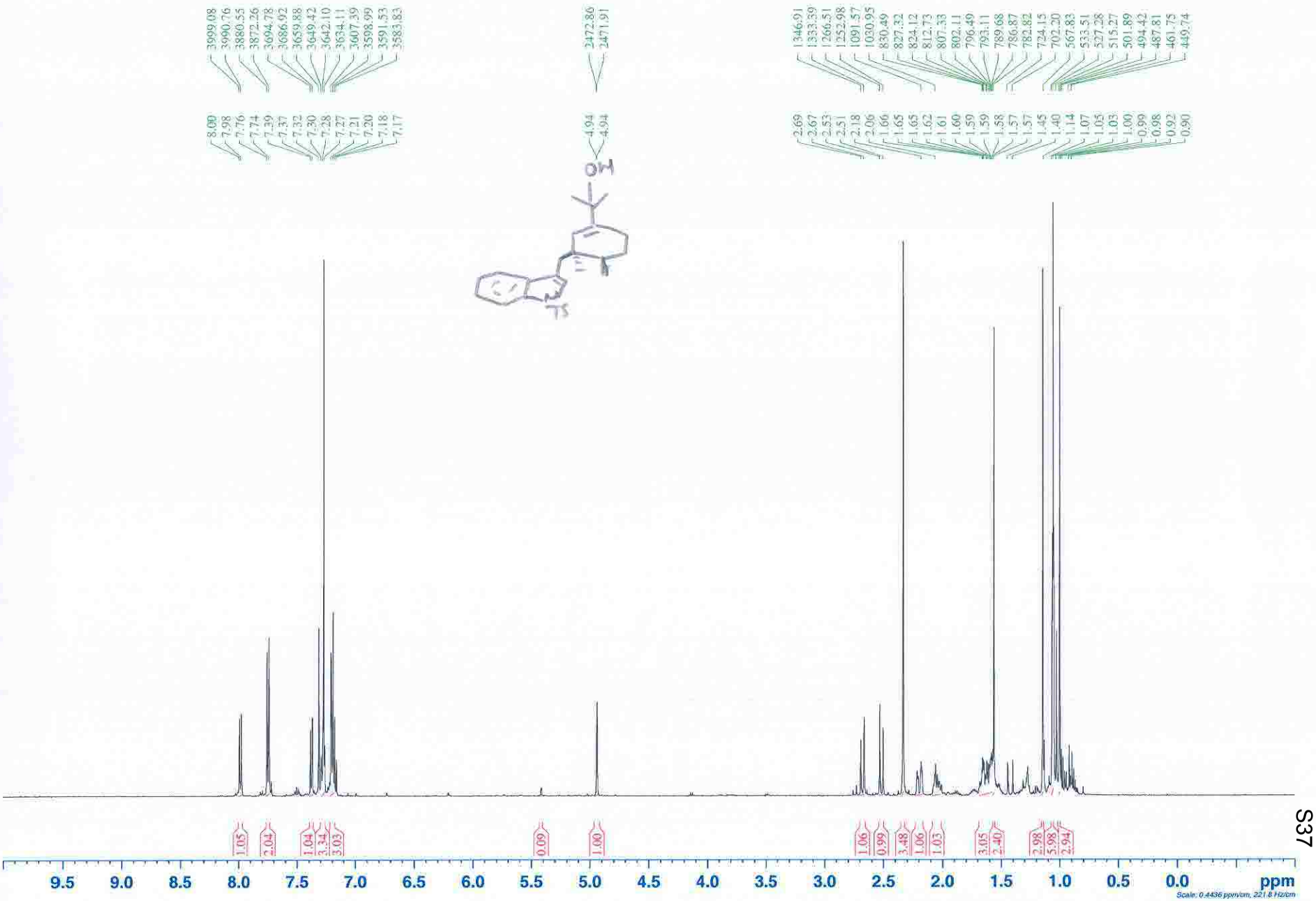
11657.21 11625.26 11593.32 7790.35 7774.08 6052.33 5804.02 5790.66 5759.77 5260.53 5232.44 5209.94 5084.21 4767.53 4641.59 4386.02 4070.99 4037.05 3857.83 3815.81 3662.43 3623.22 3419.48 3252.75 3230.37 3125.04 2414.38 2381.02 2132.03 1726.25

167.97 147.84 147.77 144.74 135.17 135.05 134.85 132.16 132.08 129.80 129.12 128.54 126.81 126.63 125.06 124.82 124.42 122.98 122.88 120.35 119.82 119.19 119.01 113.69 113.62

77.25 77.04 76.83 51.62 51.52 40.11 38.46 38.37 38.17 34.86 34.67 34.53 33.69 31.59 30.76 29.07 26.98 26.75 25.57 25.29 24.27 24.01 22.66 21.56 21.41 20.71 16.00 15.78 14.13 11.44

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

Proton24 .* evelthui ejv-iii-293 (1 1) CDCl3 24.0C March_15,2007_11:38 DRX 500MHz zg30 1H *.



Carbon .* evelthui ejv-iv-18-13C (1 1) CDCl3 24.0C April_01,2007_12:16 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.

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Current Data Parameters
NAME ejv-iv-18-13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070401
Time 12.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 134144
SOLVENT CDCl3
NS 325
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 150.9027745 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

21869.27
21434.09
20439.20
20422.67
20025.09
19645.38
19612.35
19416.42
19258.48
19157.41
18840.79
18808.95
18536.45
18228.96
18182.64
17206.92

11688.85
11656.93
11624.99
10980.05

6768.62
5890.79
5655.33
4728.64
4355.45
4343.03
4231.69
4216.24
4054.54
3719.79
3297.37
3283.89
2430.03

144.92
142.04
135.45
135.34
132.70
130.19
129.97
128.67
127.62
126.95
124.85
124.64
122.84
120.80
120.49
114.03

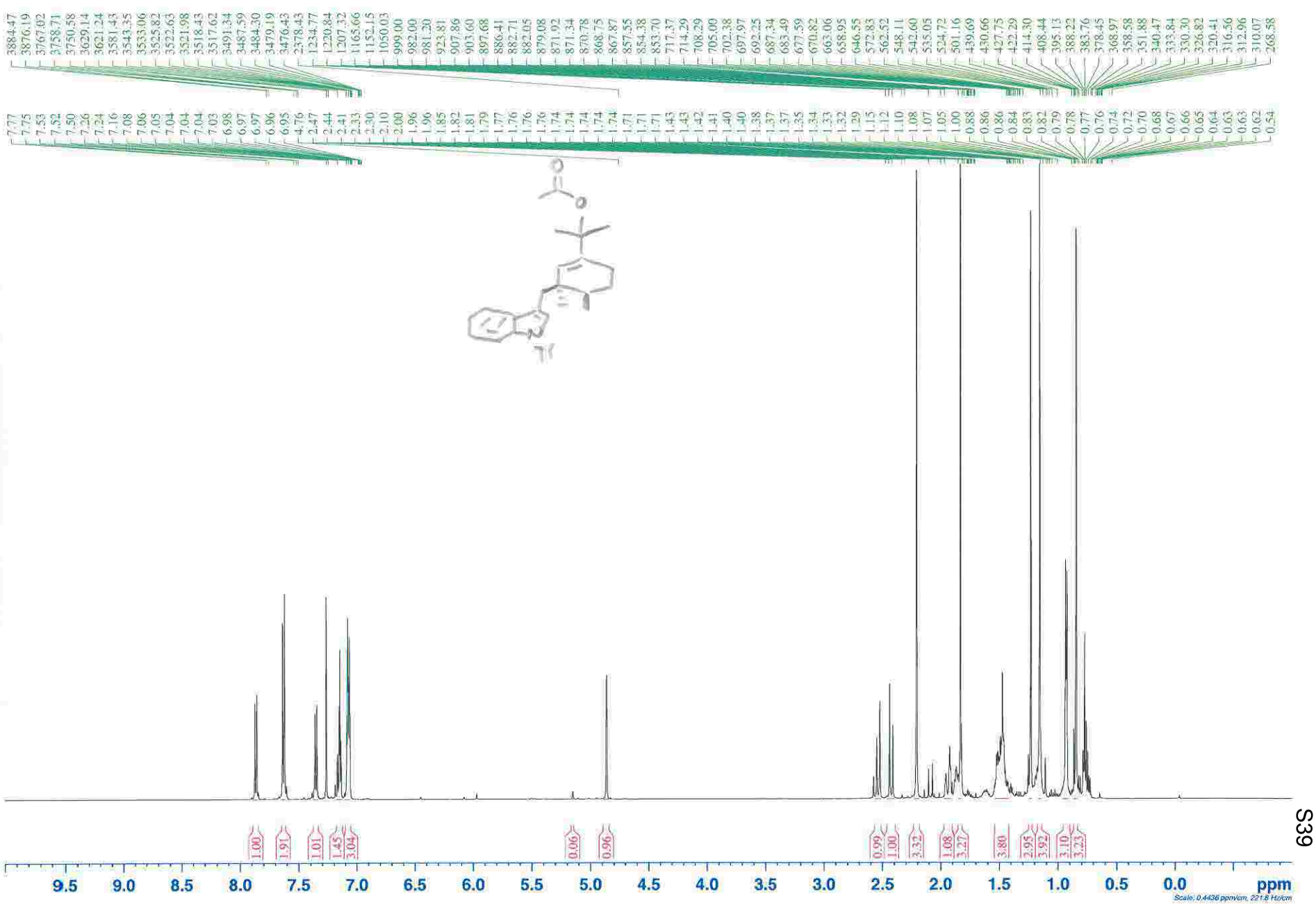
77.46
77.25
77.04
72.76

44.85
39.04
37.48
31.34
28.86
28.78
28.04
27.94
26.87
24.65
21.85
21.76
16.10

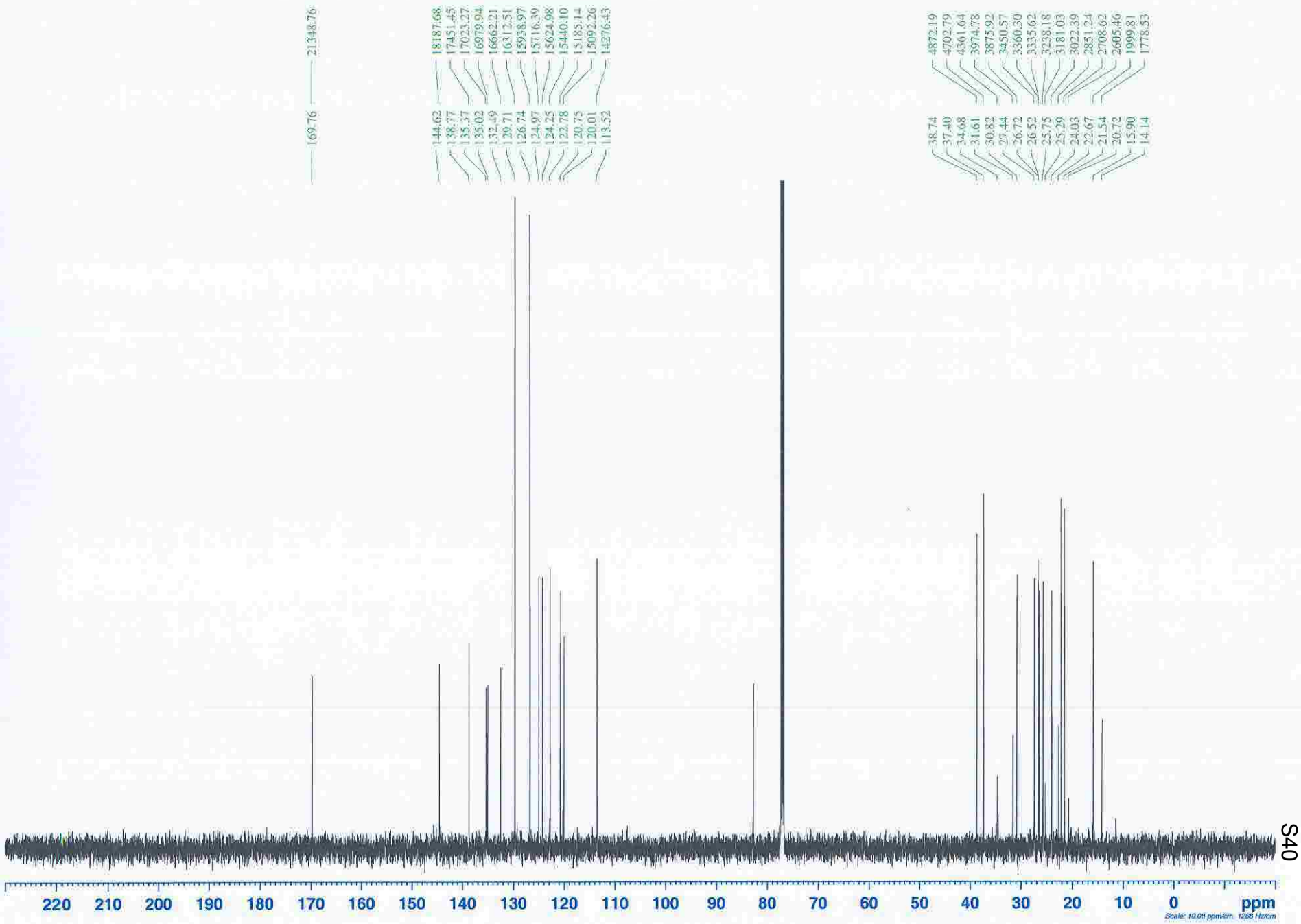
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Scale: 11.11 ppm/cm, 1677 Hz/cm

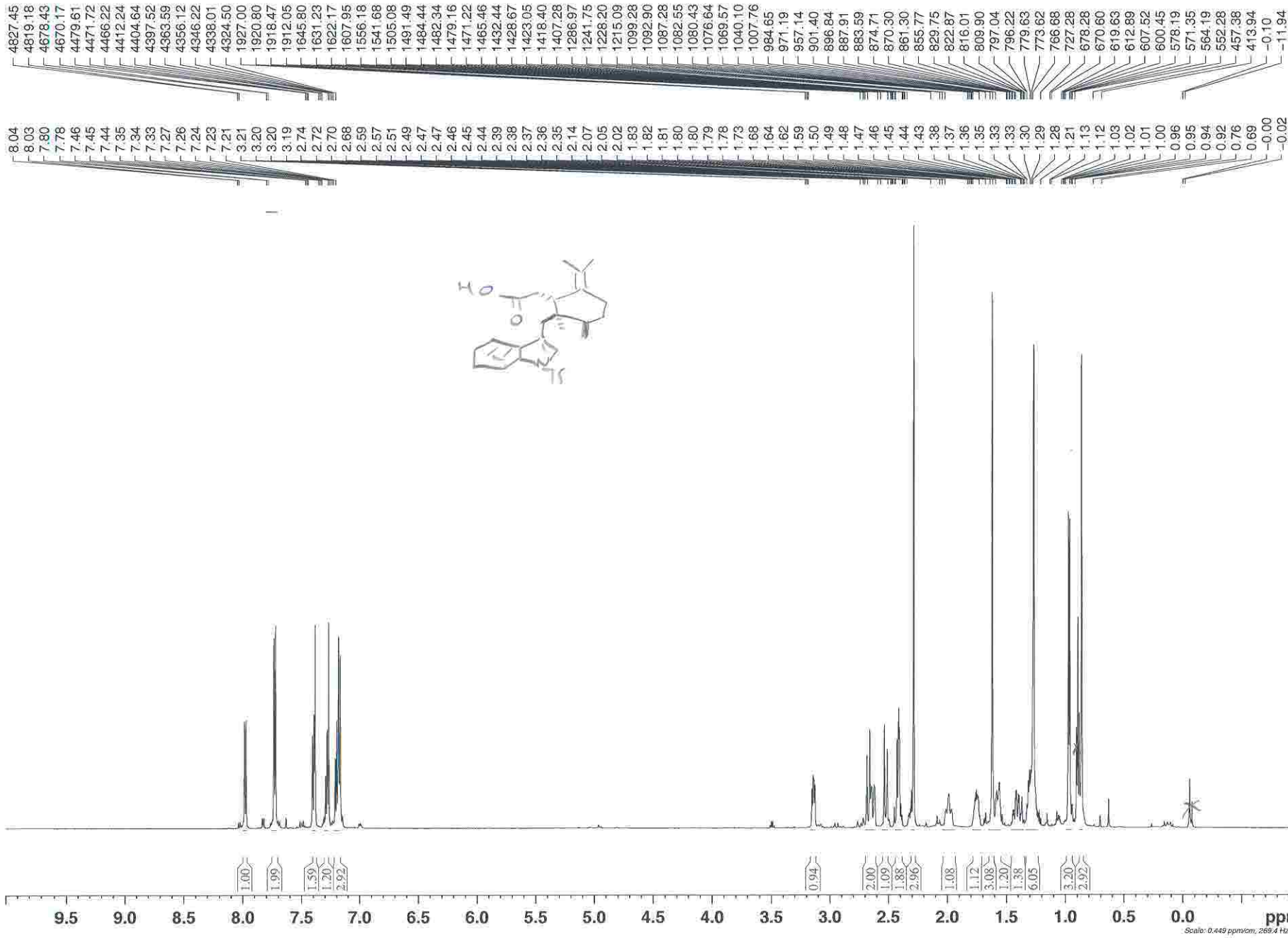
Proton24 .* evelthui ejv-iii-294 (1 1) CDCl3 24.0C March_20,2007_10:38 DRX 500MHz zg30 1H *.



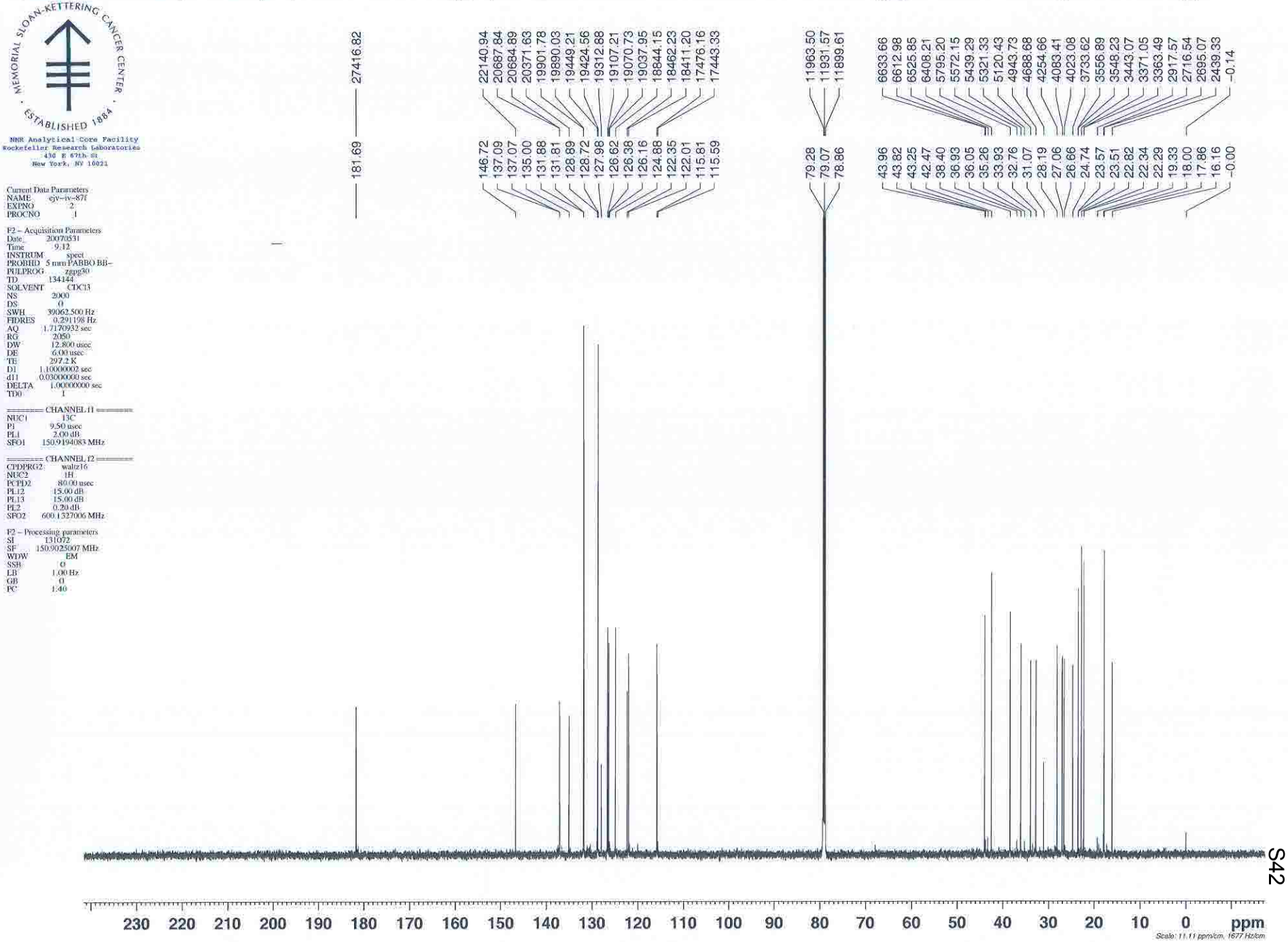
Carbon .* evelthui ejv-iii-294-C13 (1 1) CDCl3 24.0C March_20,2007_10:42 DRX 500MHz zgpg30 13C; 1H O2=4.000 *.

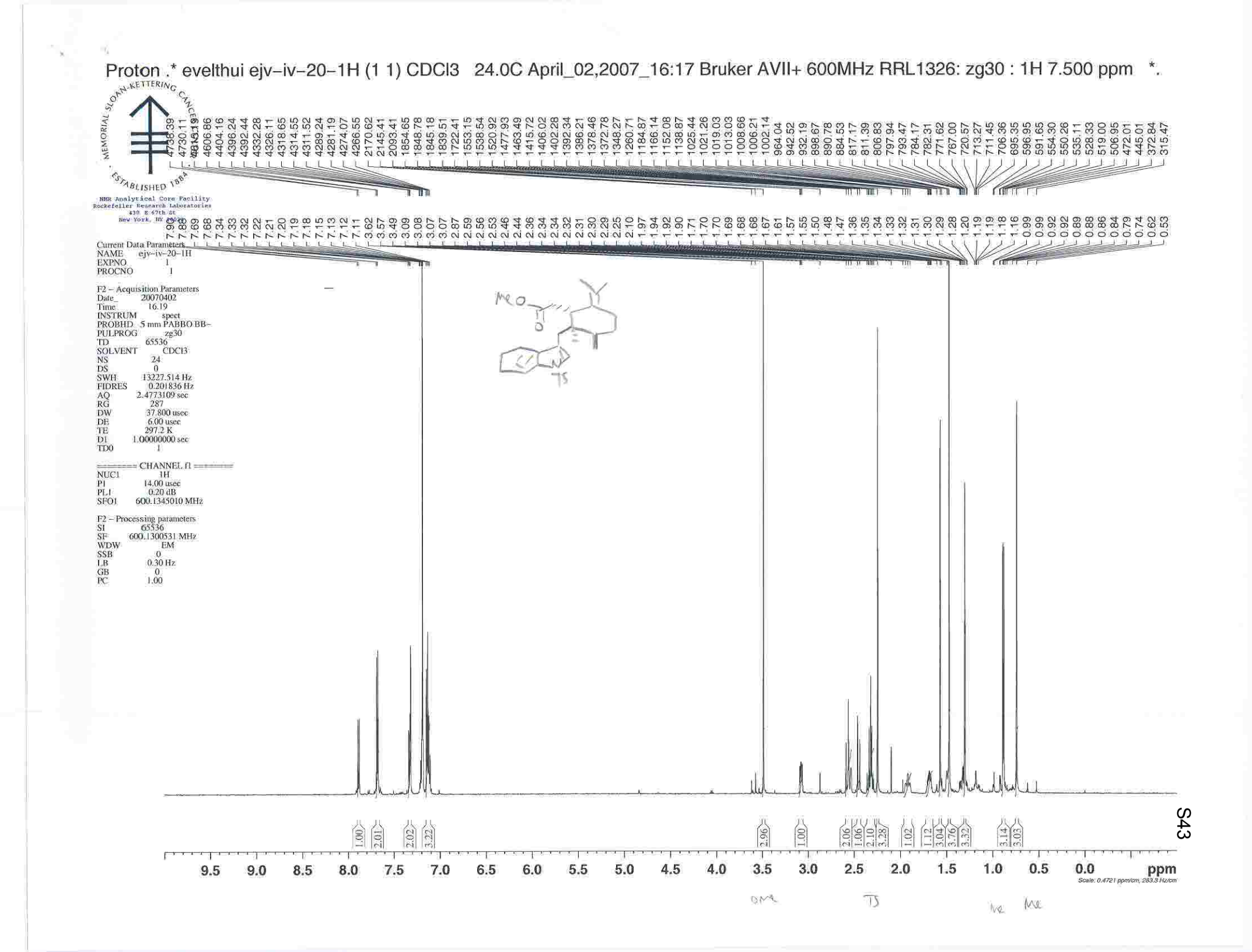


Proton .* evelthui ejv-iv-87f (1 1) CDCl3 24.0C May_31,2007_09:06 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm *.



Carbon .* evelthui ejv-iv-87f (2 1) CDCl3 24.0C May_31,2007_09:08 Bruker AVII+ 600MHz RRL1326: zgpg30 : 13C 110.000 ppm; 1H 4.500 ppm *.




Proton .* evelthui ejv-iv-20-1H (1 1) CDCl3 24.0C April_02,2007_16:17 Bruker AVII+ 600MHz RRL1326: zg30 : 1H 7.500 ppm *.
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430 E 67th St
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 CDCl3
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
D1 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 600.1300531 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
TS
1.00
2.01
2.02
3.22
2.96
1.00
2.06
1.06
2.10
3.28
1.02
1.12
3.04
3.76
3.32
3.14
3.03
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.4721 ppm/cm, 283.3 Hz/cm
DMF
TS
Me
Me

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 *.

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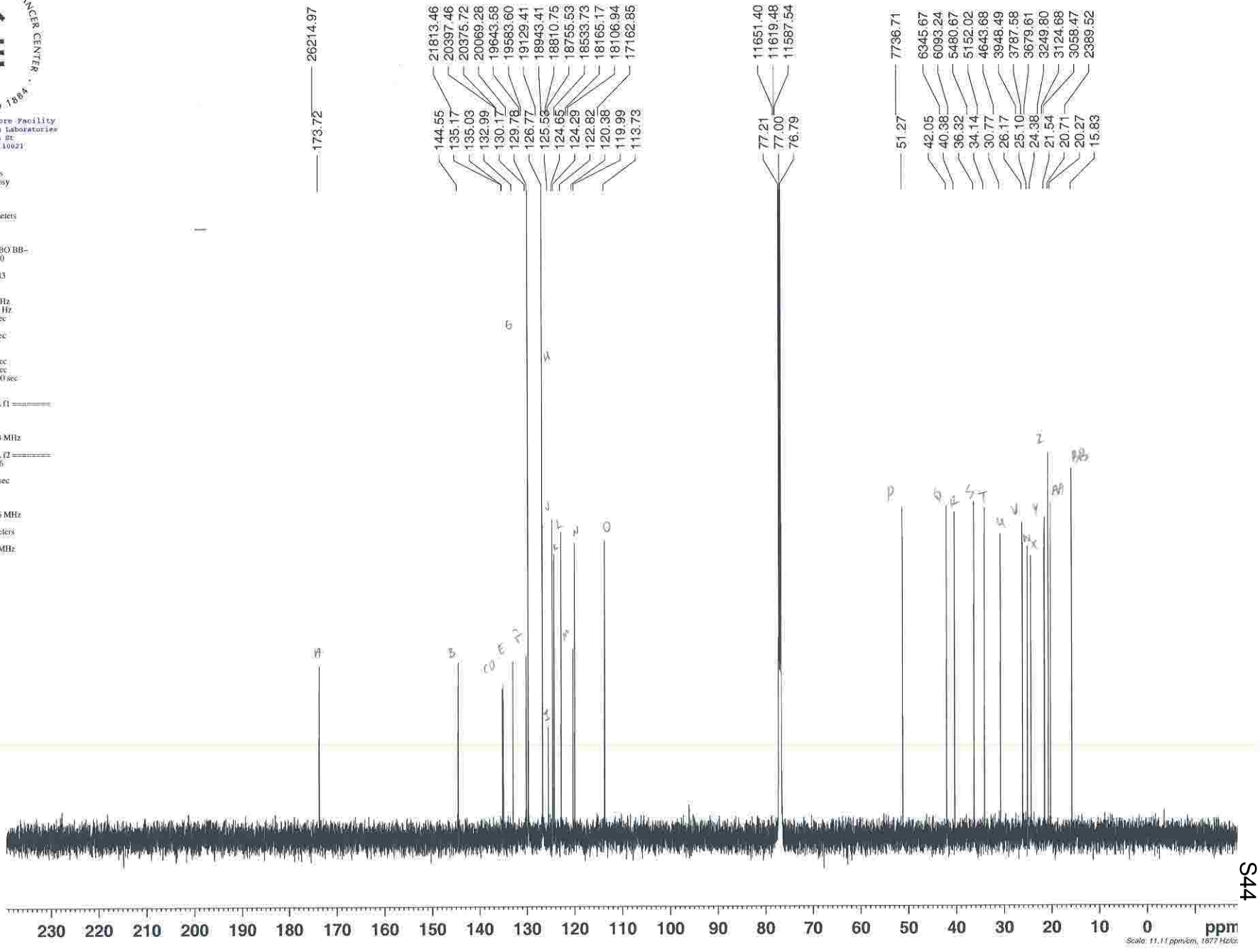
Current Data Parameters
NAME ejv-iv-20_cosy
EXPNO 3
PROCNO 1

F2 – Acquisition Parameters
Date 20070402
Time 23.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 134144
SOLVENT CDCl3
NS 8000
DS 0
SWH 39062.500 Hz
FIDRES 0.291108 Hz
AQ 1.7170932 sec
RG 2050
DW 12.800 usec
DE 6.00 usec
TE 297.0 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

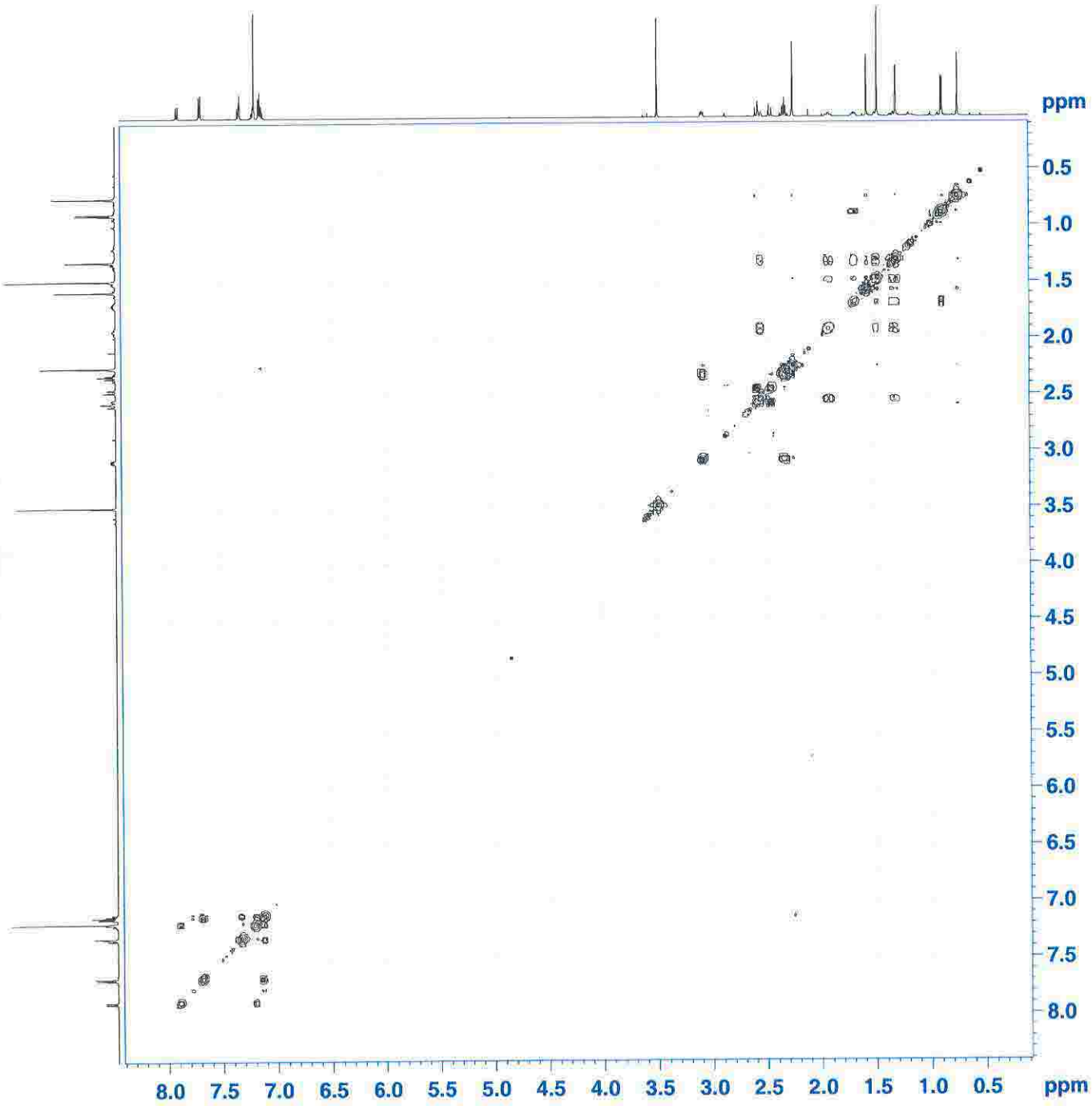
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 15.00 dB
PL13 15.00 dB
PL2 0.20 dB
SFO2 600.1327006 MHz

F2 – Processing parameters
SI 131072
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Grad COSY

ui ejv–iv–20_cosy (2 1) CDCl3 24.0C April_02,2007_17:08 Bruker AVII+ 600MHz RRL1326: cosygpqf : 1H 4.34(



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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	cosygpqf
TD	2048
SOLVENT	CDCl3
NS	2
DS	16
SWH	5000.000 Hz
FIDRES	2.441406 Hz
AQ	0.2048500 sec
RG	144
DW	100.000 usec
DE	6.00 usec
TE	297.2 K
d0	0.00000300 sec
D1	1.40988600 sec
d13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00020000 sec

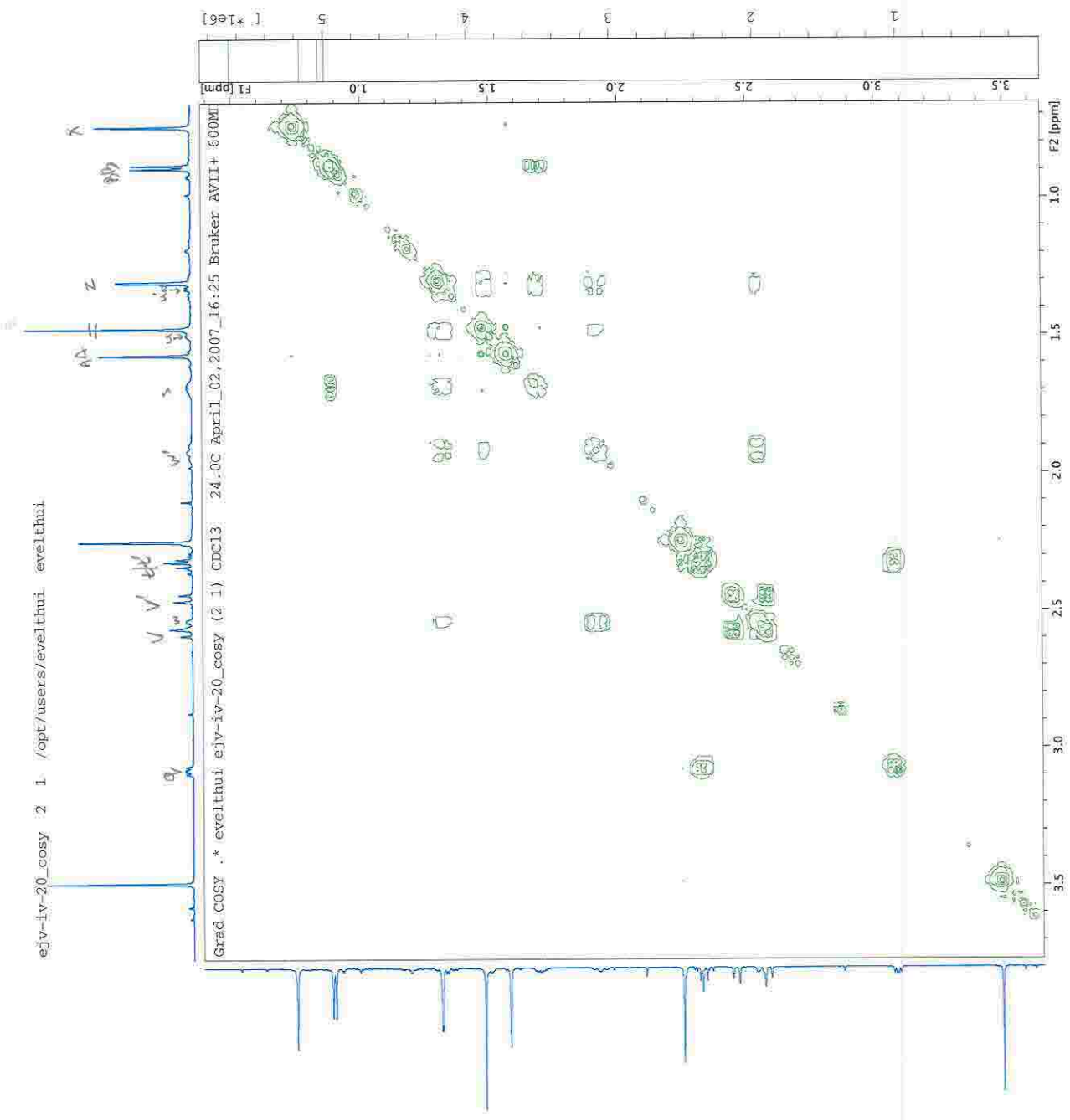
======== CHANNEL f1 ========	
NUC1	1H
P0	14.00 usec
P1	14.00 usec
PL1	0.20 dB
SFO1	600.1326033 MHz

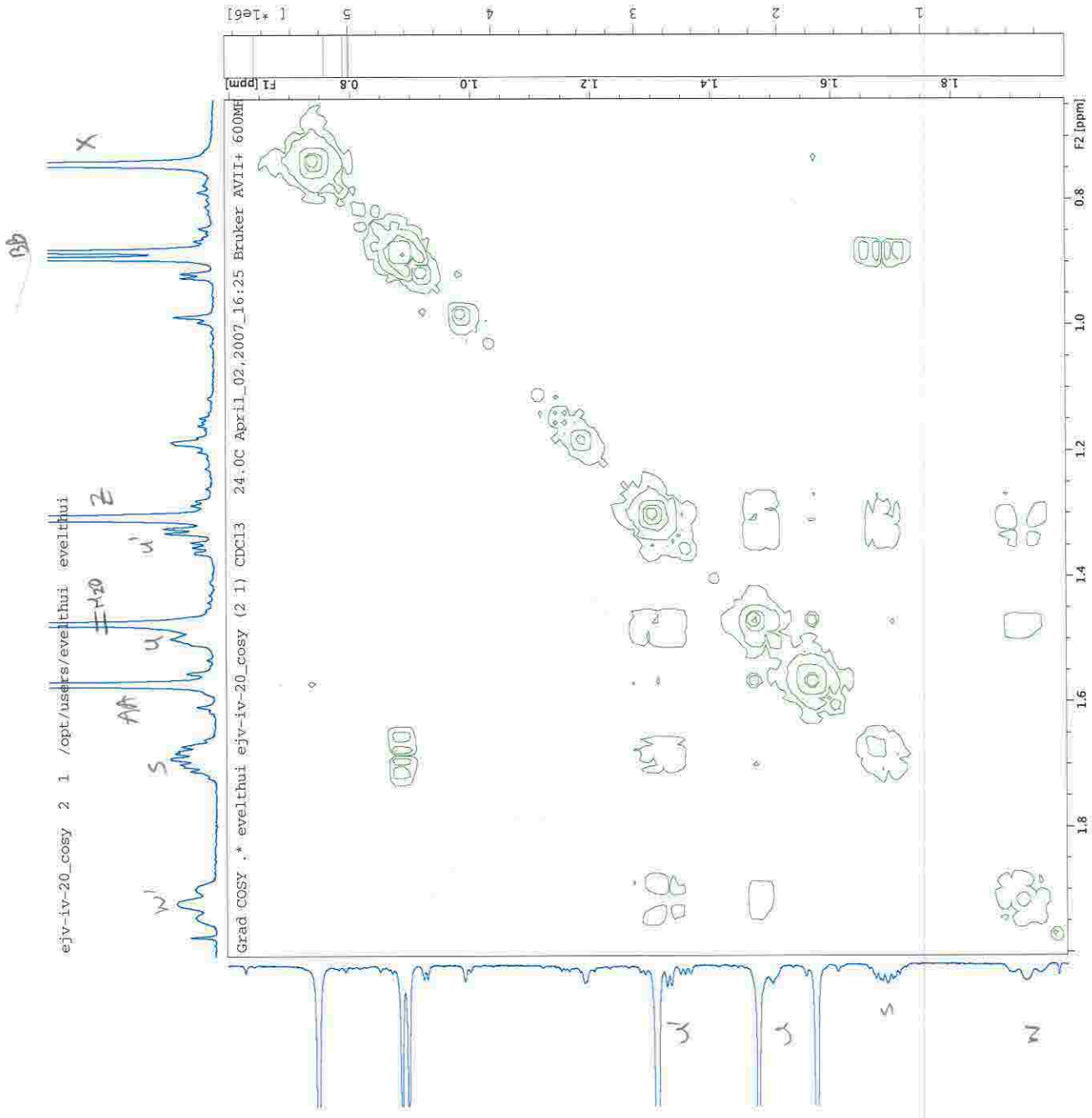
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPZ1	10.00 %
GPZ2	10.00 %
P16	1000.00 usec

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

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

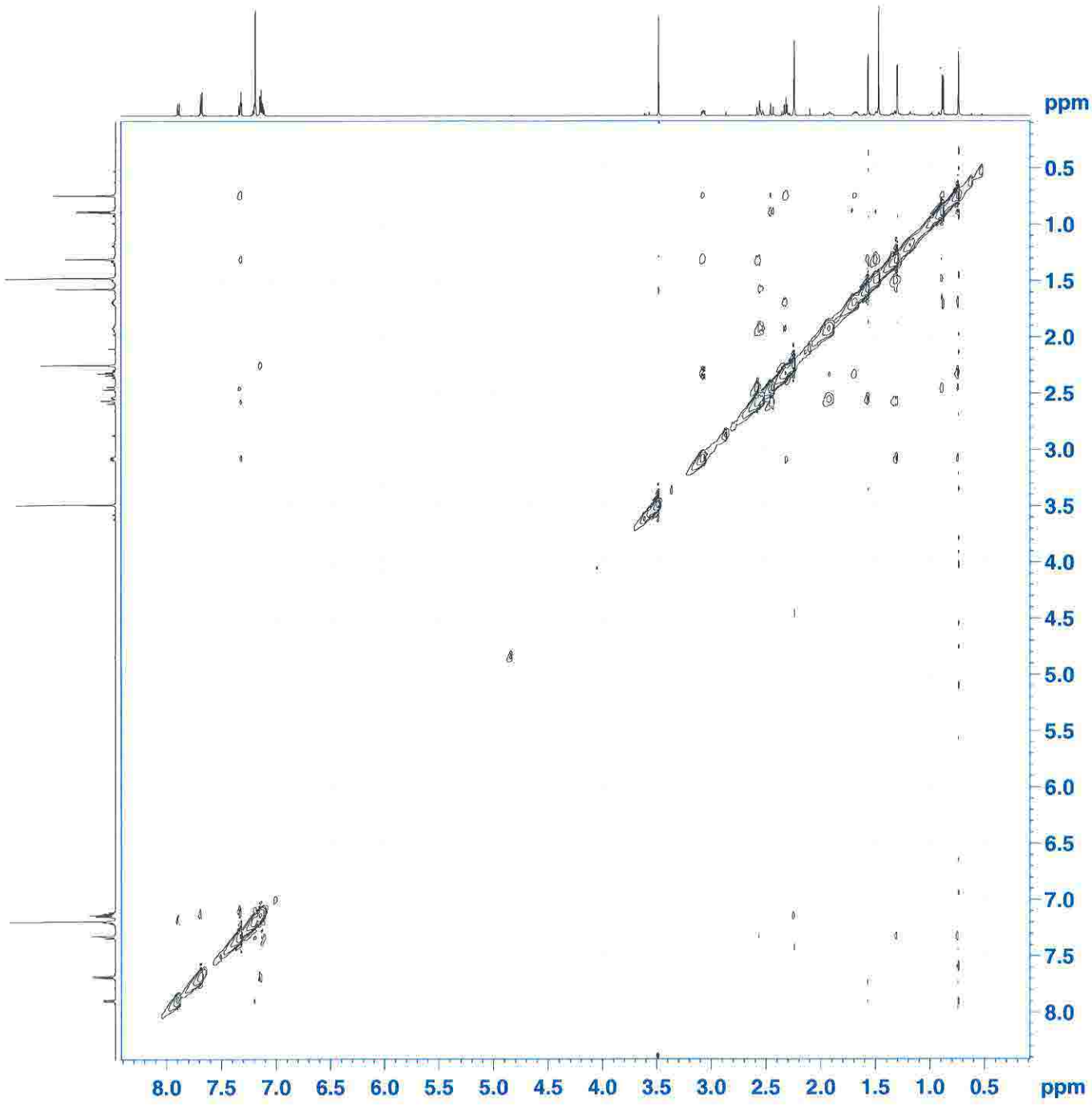
F1 - Processing parameters	
SI	1024
MC2	QF
SF	600.1300527 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0


ejv-iv-20_cosy 2 1 /opt/users/evelthui evelthui
Grad COSY .* evelthui ejv-iv-20_cosy (2 1) CDCl3 24.0C April_02,2007_16:25 Bruker AVII+ 600MH
F1 [ppm]
F2 [ppm]
[*1e6]


ejv-iv-20_cosy 2 1 /opt/users/evelthui evelthui
Grad COSY .* evelthui ejv-iv-20_cosy (2 1) CDCl3 24.0C April_02,2007_16:25 Bruker AVII+ 600MF
F1 [ppm]
F2 [ppm]
H-20

NOESY

ui ejv-iv-20_cosy (6 1) CDCl3 24.0C April_03,2007_10:13 Bruker AVII+ 600MHz RRL1326: noesyph : 1H 4.603



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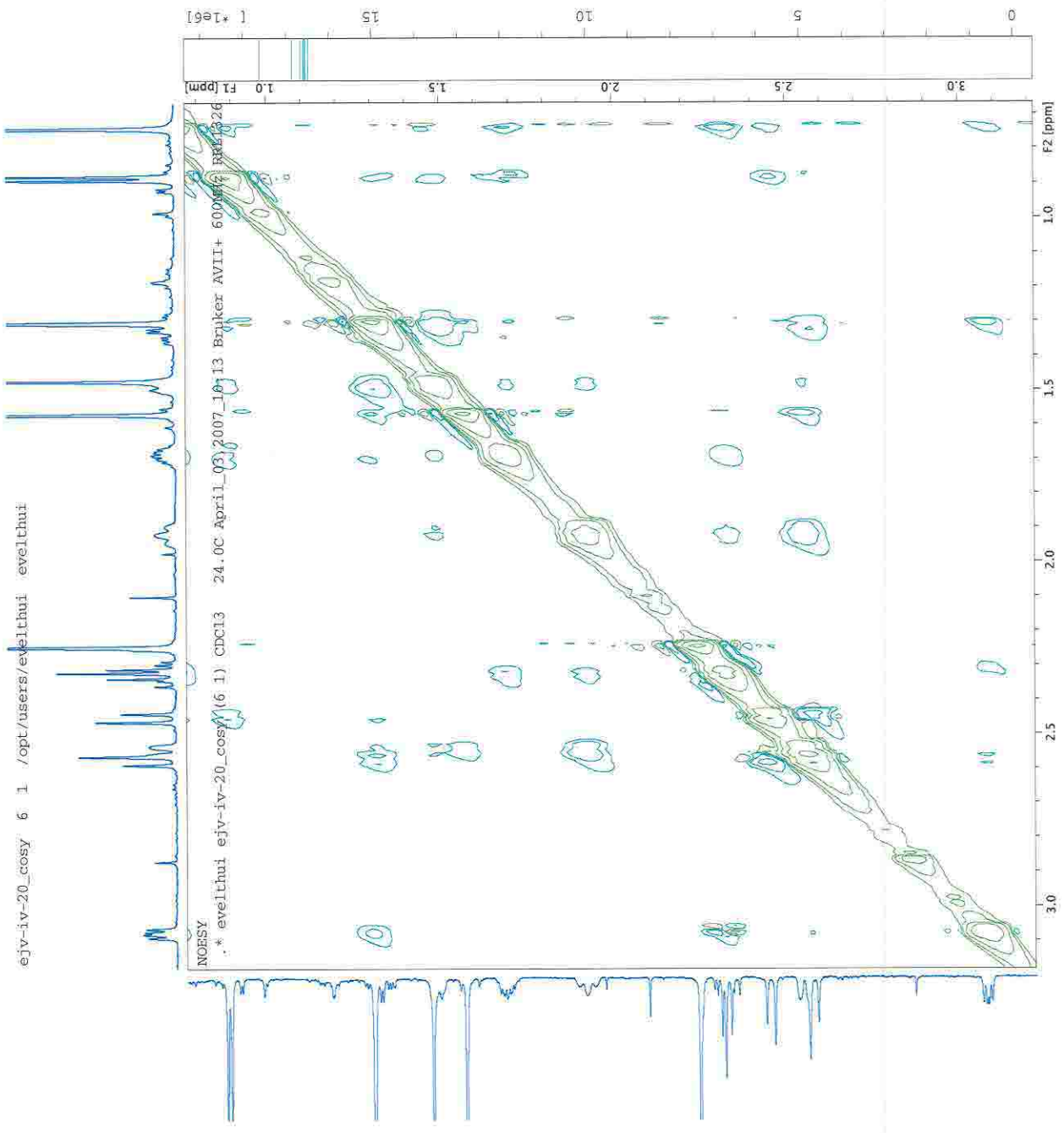
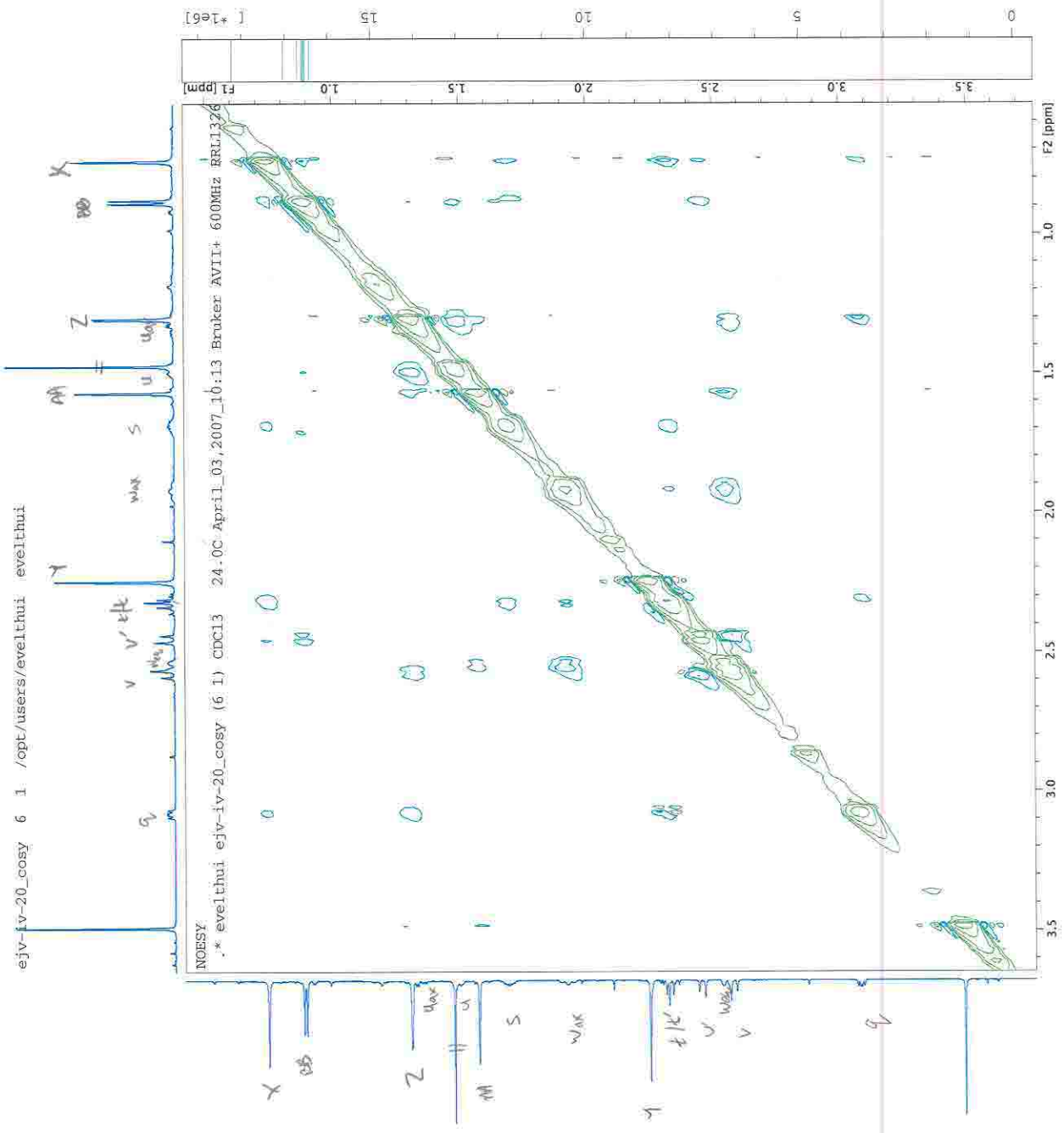
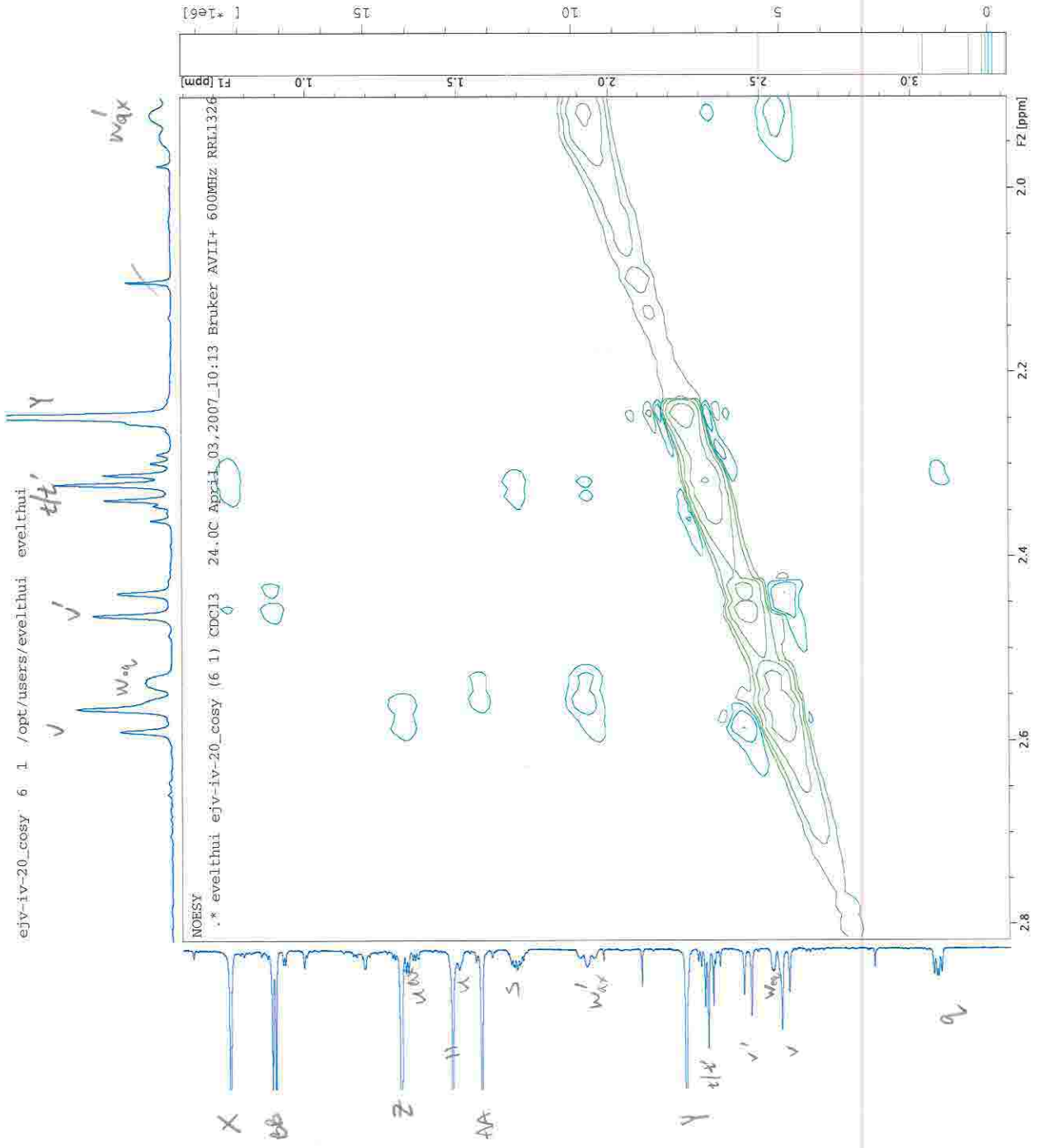
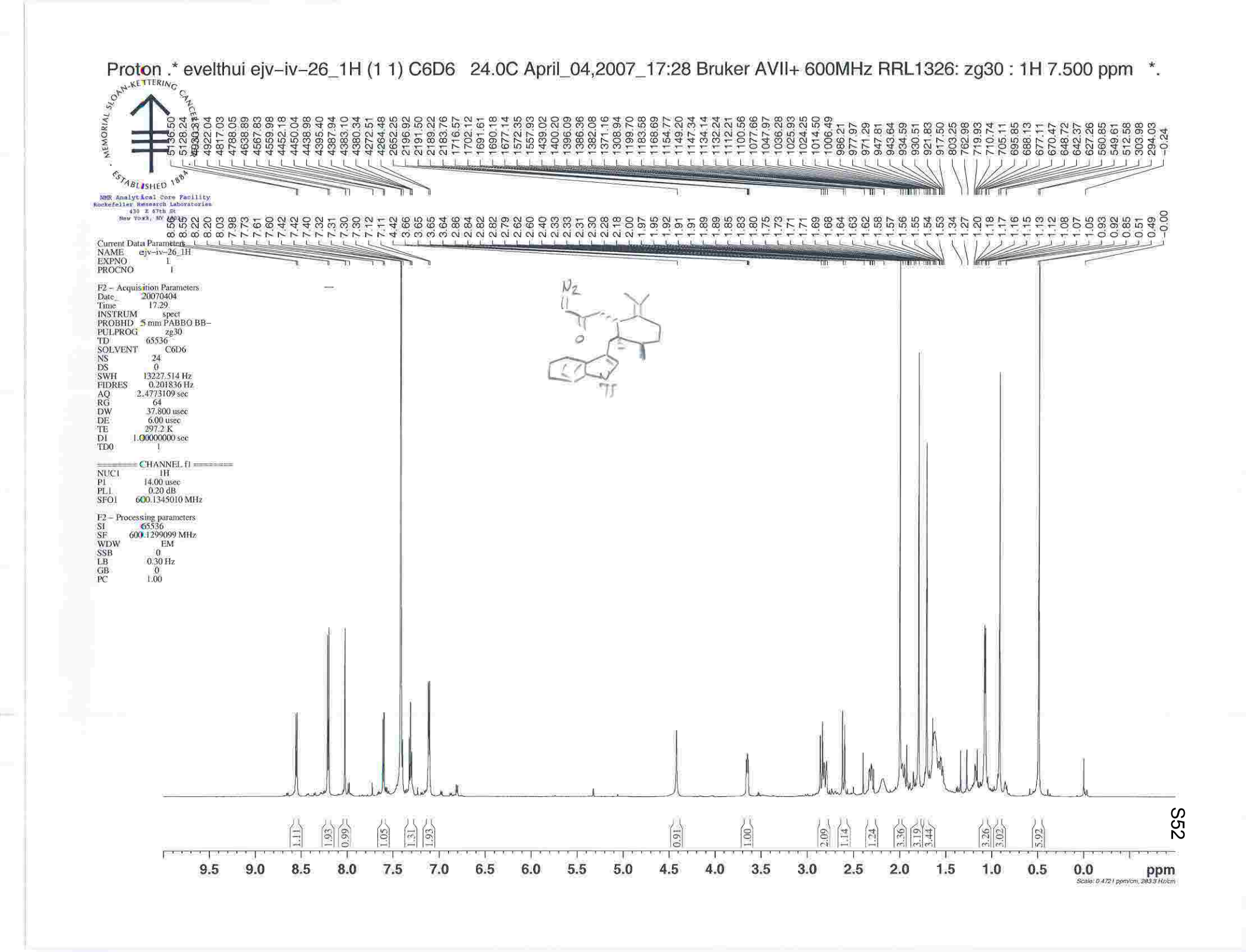
```
Current Data Parameters
NAME      ejv-iv-20_cosy
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070403
Time               10.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          noesyph
TD                  2048
SOLVENT             CDCl3
NS                     16
DS                     16
SWH          5000.000 Hz
FIDRES       2.441406 Hz
AQ           0.2048500 sec
RG                   181
DW           100.000 usec
DE             6.00 usec
TE              297.2 K
d0           0.00008217 sec
D1           1.96231604 sec
D8           0.30000001 sec
IN0          0.00020000 sec
ST1CNT              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
FnMODE     States-TPPI

F2 - Processing parameters
SI                  1024
SF          600.1300527 MHz
WDW                QSINE
SSB                    2
LB              0.00 Hz
GB                     0
PC                  1.00

F1 - Processing parameters
SI                  1024
MC2        States-TPPI
SF          600.1300527 MHz
WDW                QSINE
SSB                    2
LB              0.00 Hz
GB                     0
```

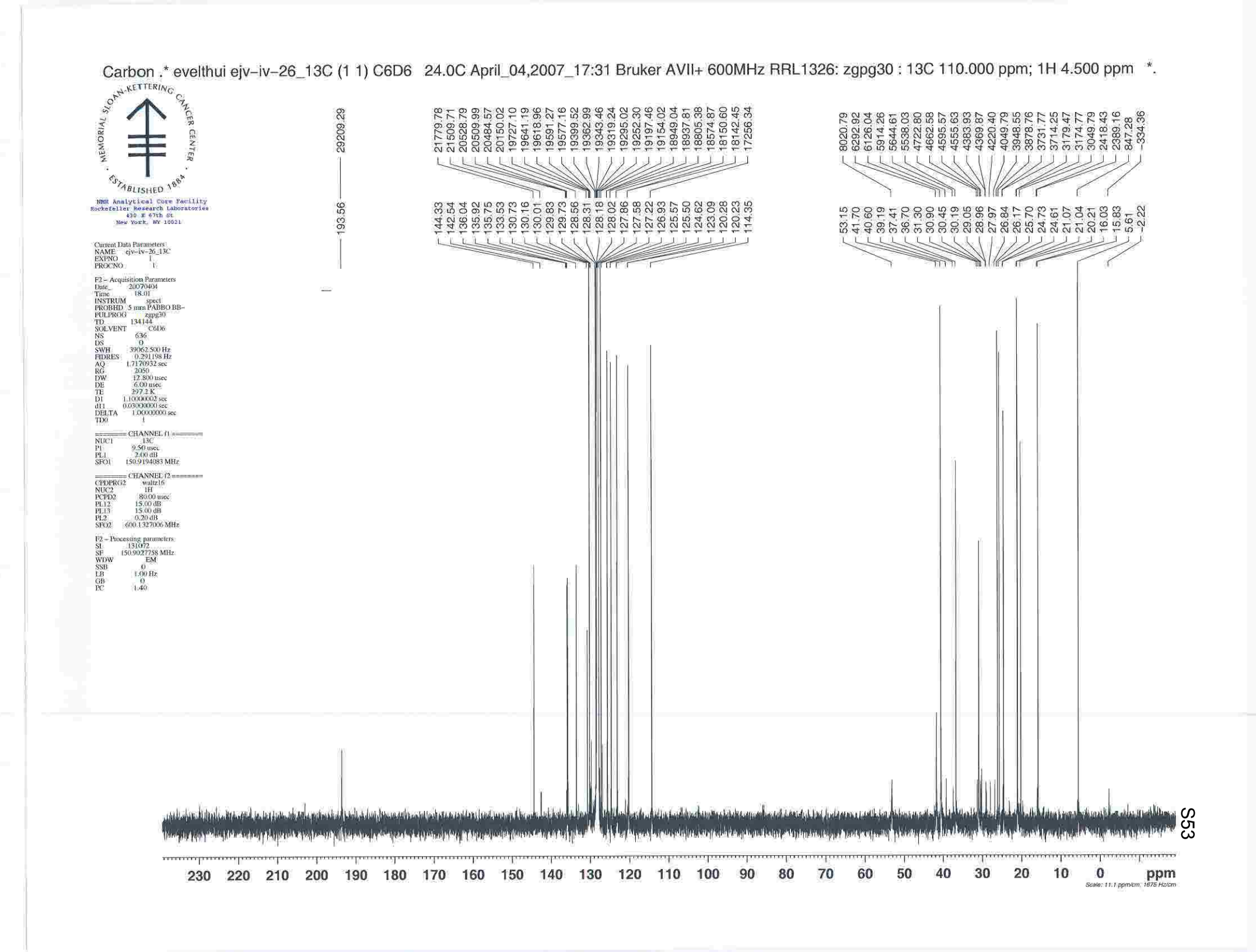

ejv-iv-20_cosy 6 1 /opt/users/evelthui evelthui
NOESY
.* evelthui ejv-iv-20_cosy (6 1) CDCl3 24.0C April_03 2007_10_13 Bruker AVII+ 600MHz RMA326
F1 [ppm]
F2 [ppm]


ejv-iv-20_cosy 6 1 /opt/users/evelthui evelthui
NOESY
.* evelthui ejv-iv-20_cosy (6 1) CDCl3 24.0C April_03,2007_10:13 Bruker AVII+ 600MHz RRL1326
F1 [ppm]
F2 [ppm]


ejv-iv-20_cosy 6 1 /opt/users/evelthui evelthui
NOESY
.* evelthui ejv-iv-20_cosy (6 1) CDCl3 24.0C April 03,2007_10:13 Bruker AVII+ 600MHz RRI1326
F1 [ppm]
F2 [ppm]
[*1e6]


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 *.
MEMORIAL SLOAN-KETTERING CANCER CENTER
ESTABLISHED 1884
NMR Analytical Core Facility
Rockefeller Research Laboratories
430 E 67th St
New York, NY 10021
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 1.00000000 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 600.1299099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N2
O
1.11
1.93
0.99
1.05
1.31
1.93
0.91
1.00
2.09
1.14
1.24
3.36
3.19
3.44
3.26
3.02
5.92
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.472 ppm/cm, 283.3 Hz/cm

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 *.



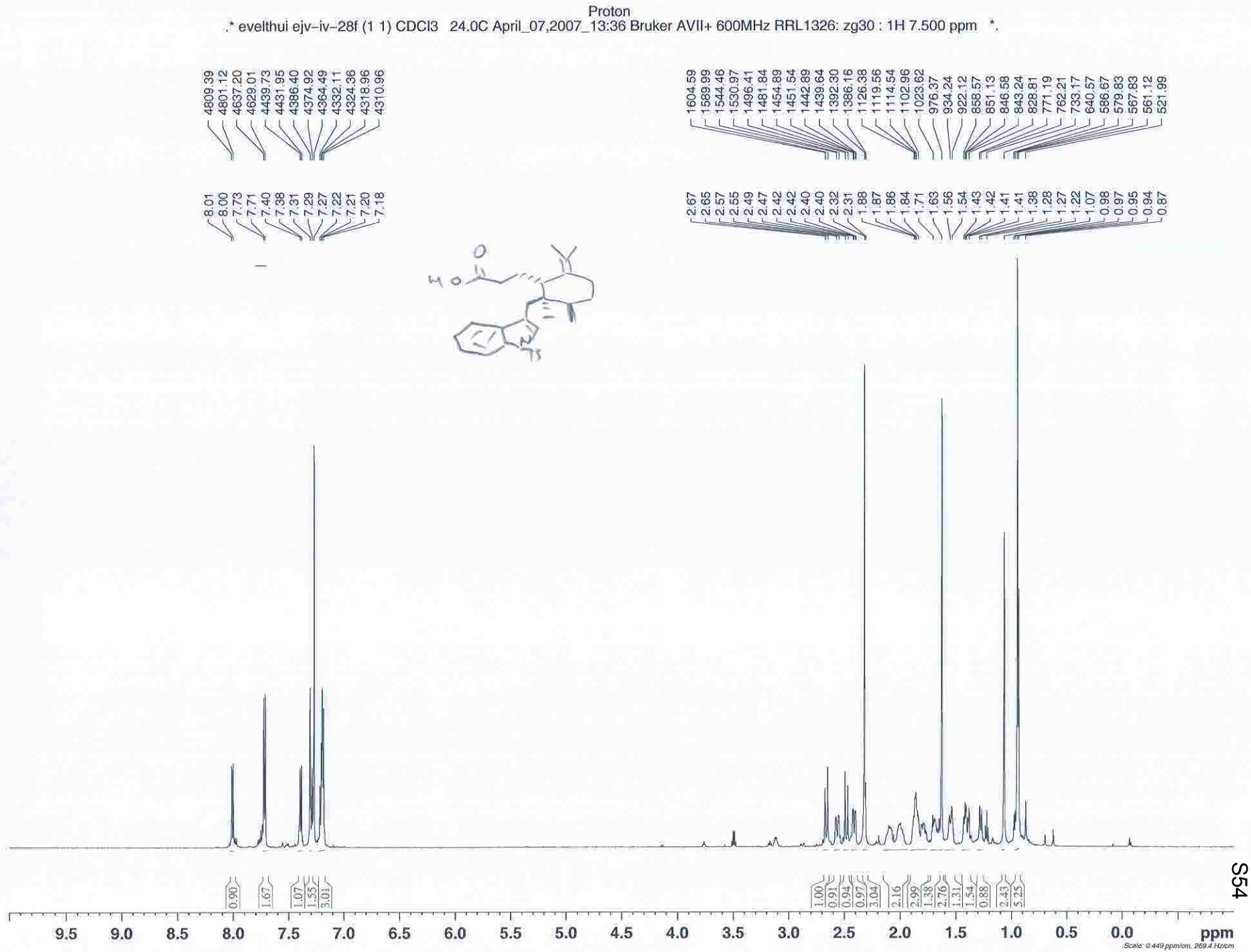
Current Data Parameters
NAME ejv-iv-26_13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070404
Time 18.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 134144
SOLVENT C6D6
NS 636
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 150.9027758 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


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
1604.59
1589.99
1544.46
1530.97
1496.41
1481.84
1454.89
1451.54
1442.89
1439.64
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
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
2.67
2.65
2.57
2.55
2.49
2.47
2.42
2.42
2.40
2.40
2.32
2.31
1.88
1.87
1.86
1.84
1.71
1.63
1.56
1.54
1.43
1.42
1.41
1.41
1.38
1.28
1.27
1.22
1.07
0.98
0.97
0.95
0.94
0.87
0.90
1.67
1.07
1.55
3.01
1.00
0.91
0.94
0.97
3.04
2.16
2.99
1.38
2.76
1.31
1.54
0.88
2.43
5.25
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
Scale: 0.449 ppm/cm, 269.4 Hz/cm

Carbon .* evelthui ejv-iv-28f (2 1) CDCl3 24.0C April_07,2007_13:38 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
NAME ejv-iv-28f
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070407
Time 14.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 134144
SOLVENT CDCl3
NS 5000
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

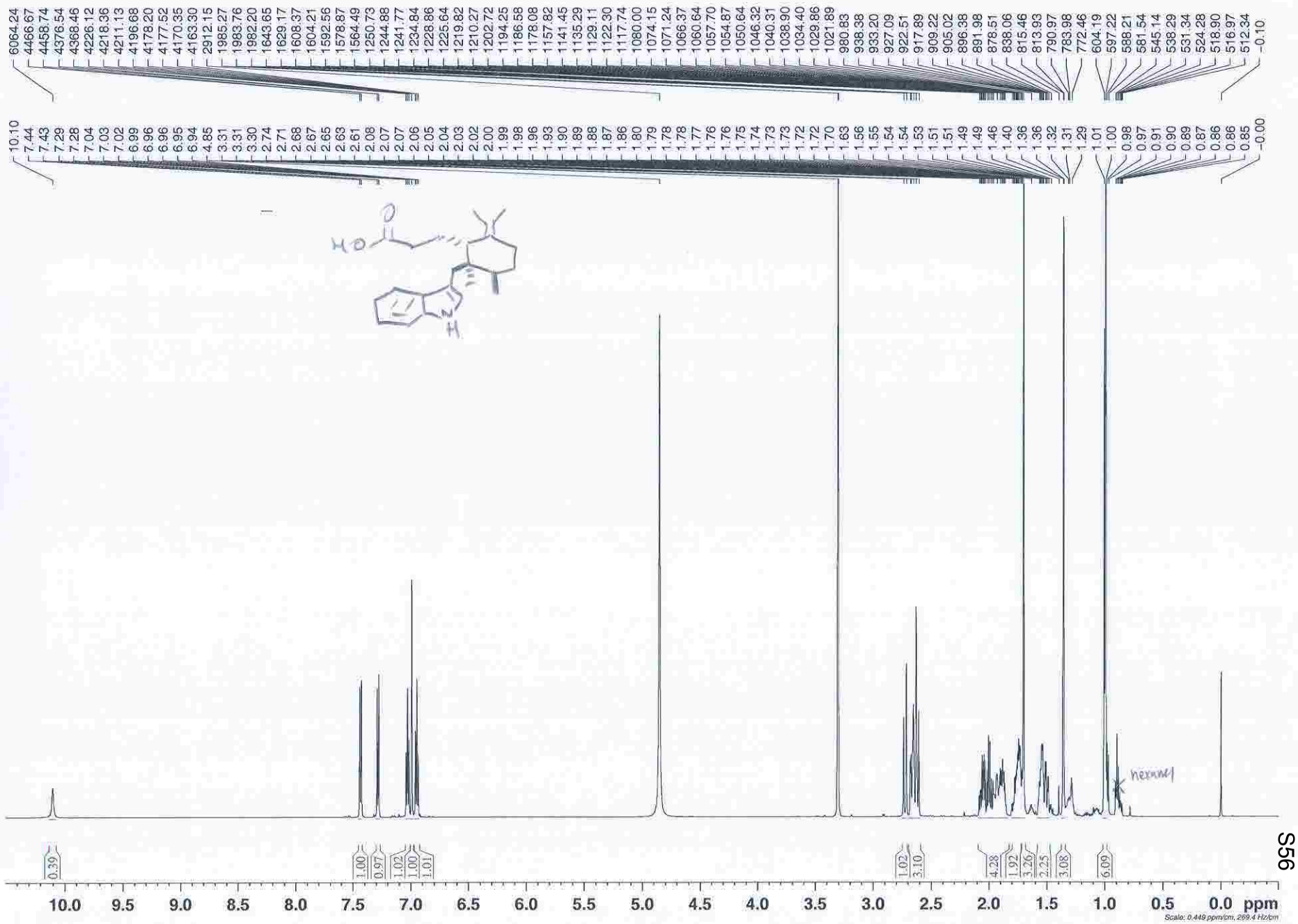
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 15.00 dB
PL13 15.00 dB
PL2 0.20 dB
SFO2 600.1327006 MHz

F2 - Processing parameters
SI 131072
SF 150.9028090 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

21838.44
20409.16
20389.29
20092.63
19629.96
19581.06
19566.85
19122.00
19118.53
18975.85
18765.56
18737.58
18538.63
18267.30
18117.04
17172.85
17164.60
11652.53
11620.62
11588.68
6913.86
6646.13
6123.10
5458.64
4823.10
4662.22
4017.48
3806.79
3734.71
3425.00
3239.01
3138.60
3026.64
2390.38
1300.19
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
77.22
77.01
76.80
45.82
44.04
40.58
36.17
31.96
30.90
26.62
25.23
24.75
22.70
21.46
20.80
20.06
15.84
8.62

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

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 *.

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

Current Data Parameters
NAME ejv-iv-76_13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070522
Time 12.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 134144
SOLVENT MeOD
NS 844
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 150.9025947 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

26893.85
178.22

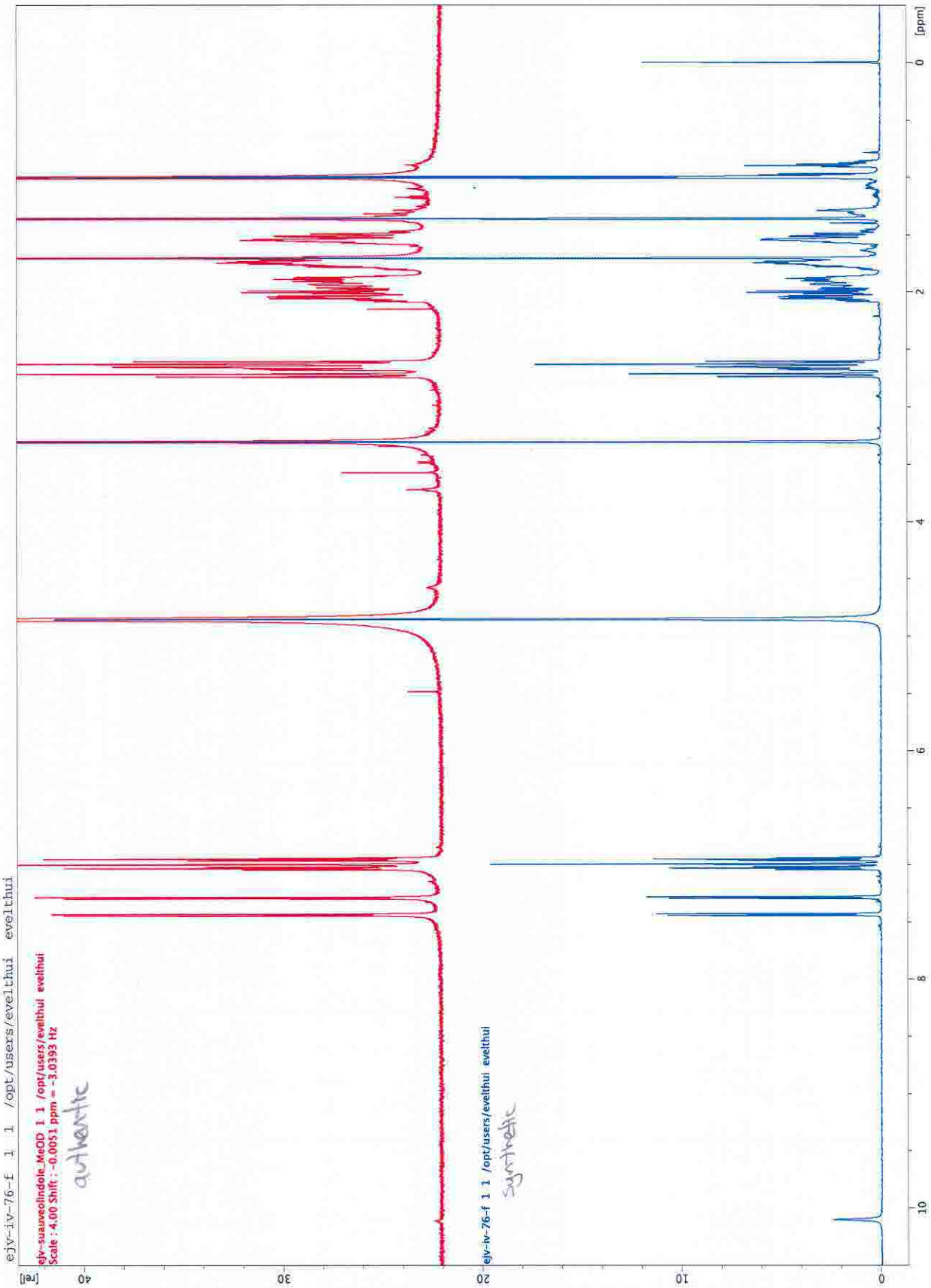
20779.64
19972.45
19735.42
19049.12
18786.53
18761.70
18380.16
18127.65
17981.59
17092.88
16896.60
16888.48

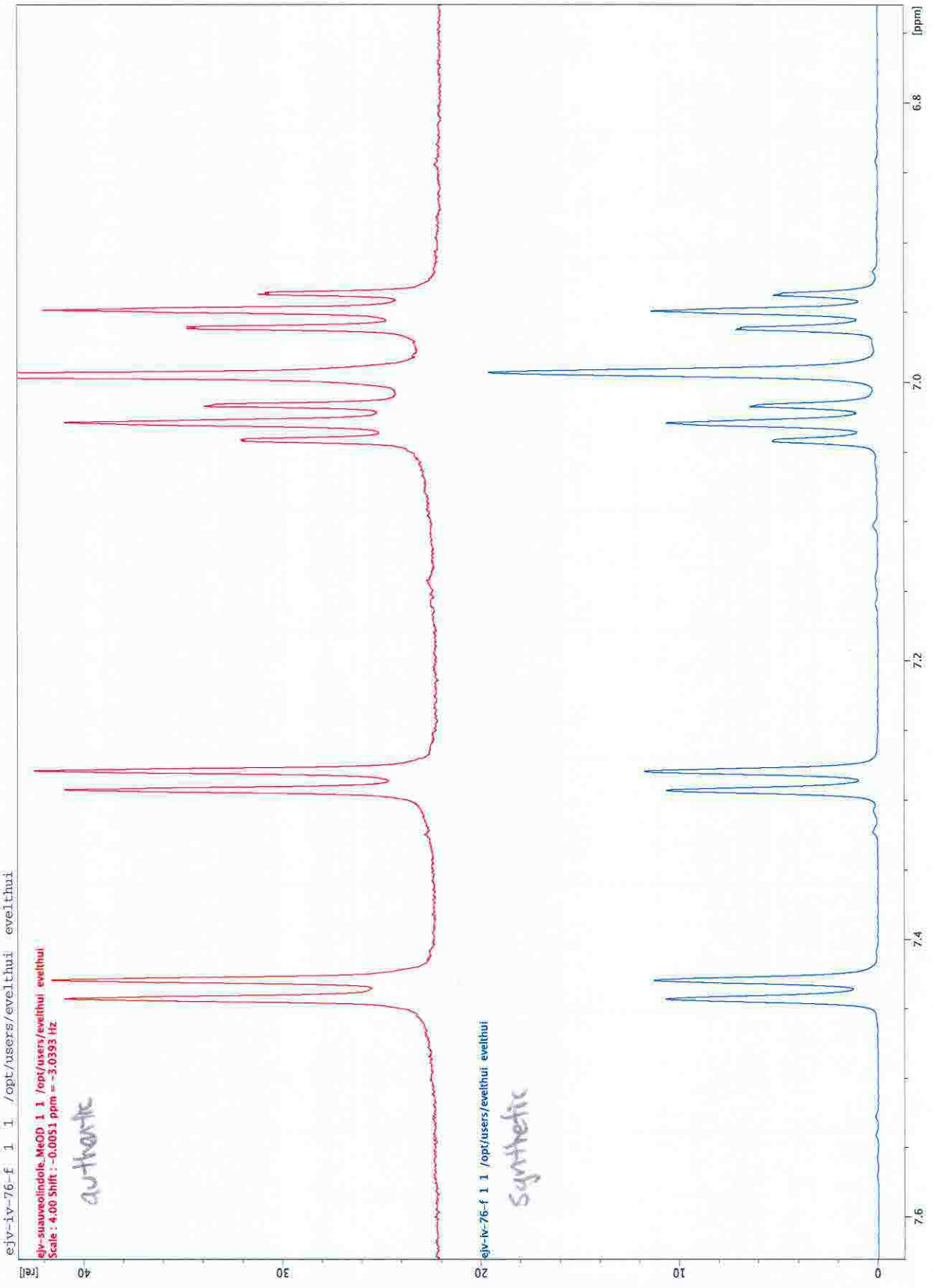
137.70
132.35
130.78
126.23
124.49
124.33
121.80
120.13
119.16
113.27
111.97
111.92

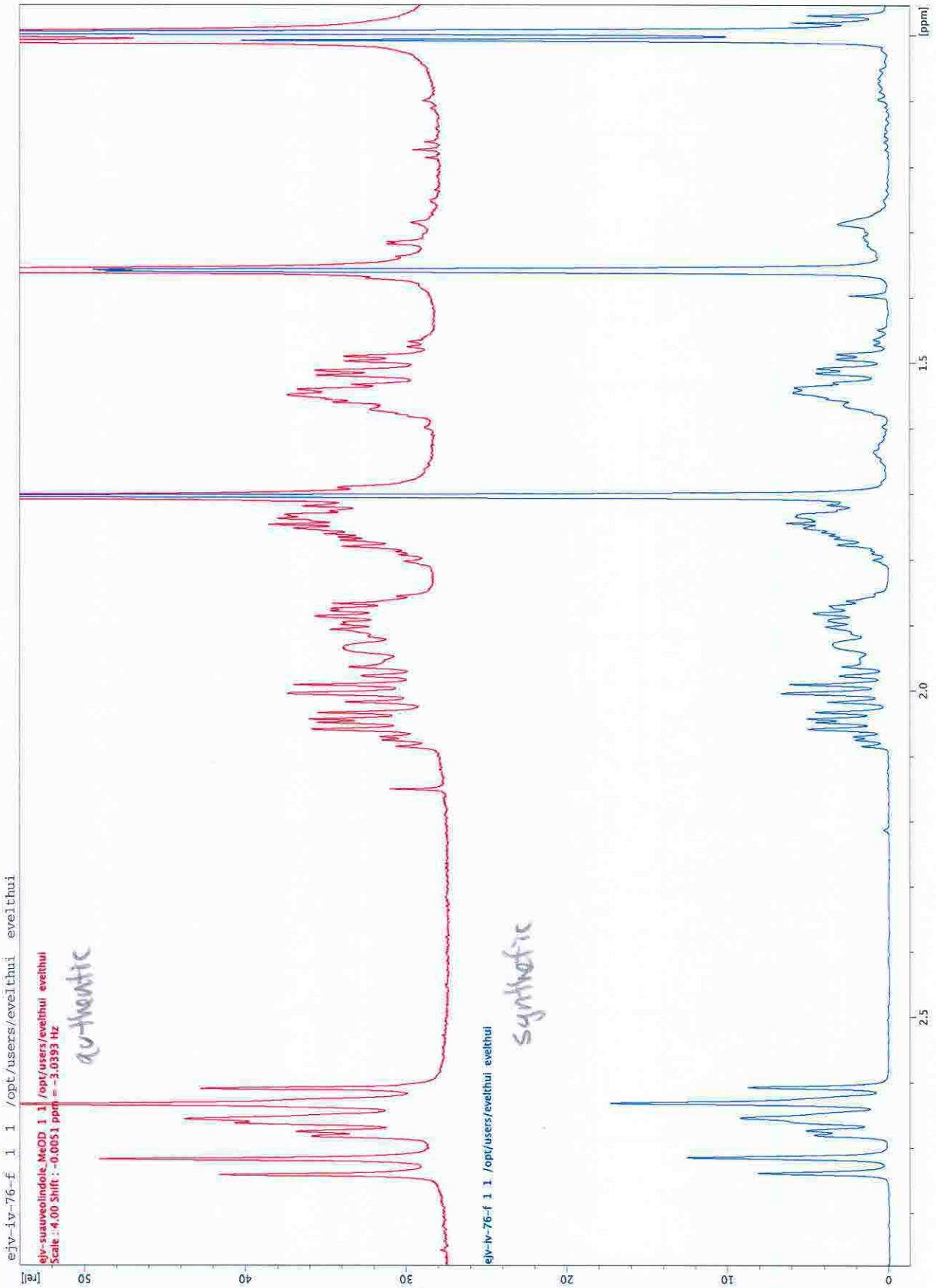
7461.76
7440.33
7418.91
7397.49
7376.08
7354.67
7333.25
6877.71
6317.36
5696.59
5387.34
4997.74
4945.06
4876.70
4209.55
3957.02
3937.74
3909.44
3621.88
3579.21
3270.87
3174.14
3067.88
2474.68
2180.25

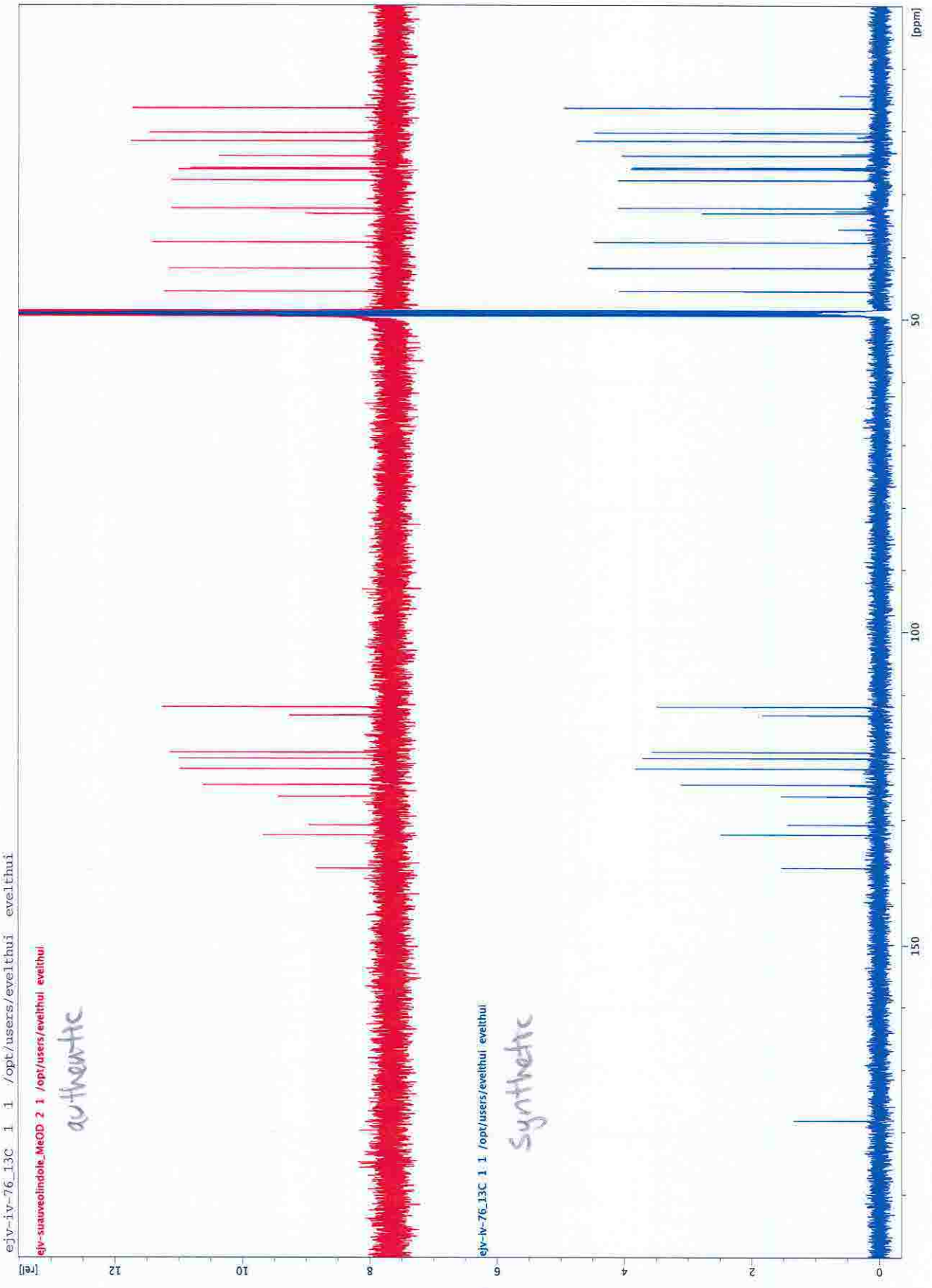
49.45
49.31
49.16
49.02
48.88
48.74
48.60
45.58
41.86
37.75
35.70
33.12
32.77
32.32
27.90
26.22
26.09
25.91
24.00
23.72
21.68
21.03
20.33
16.40
14.45

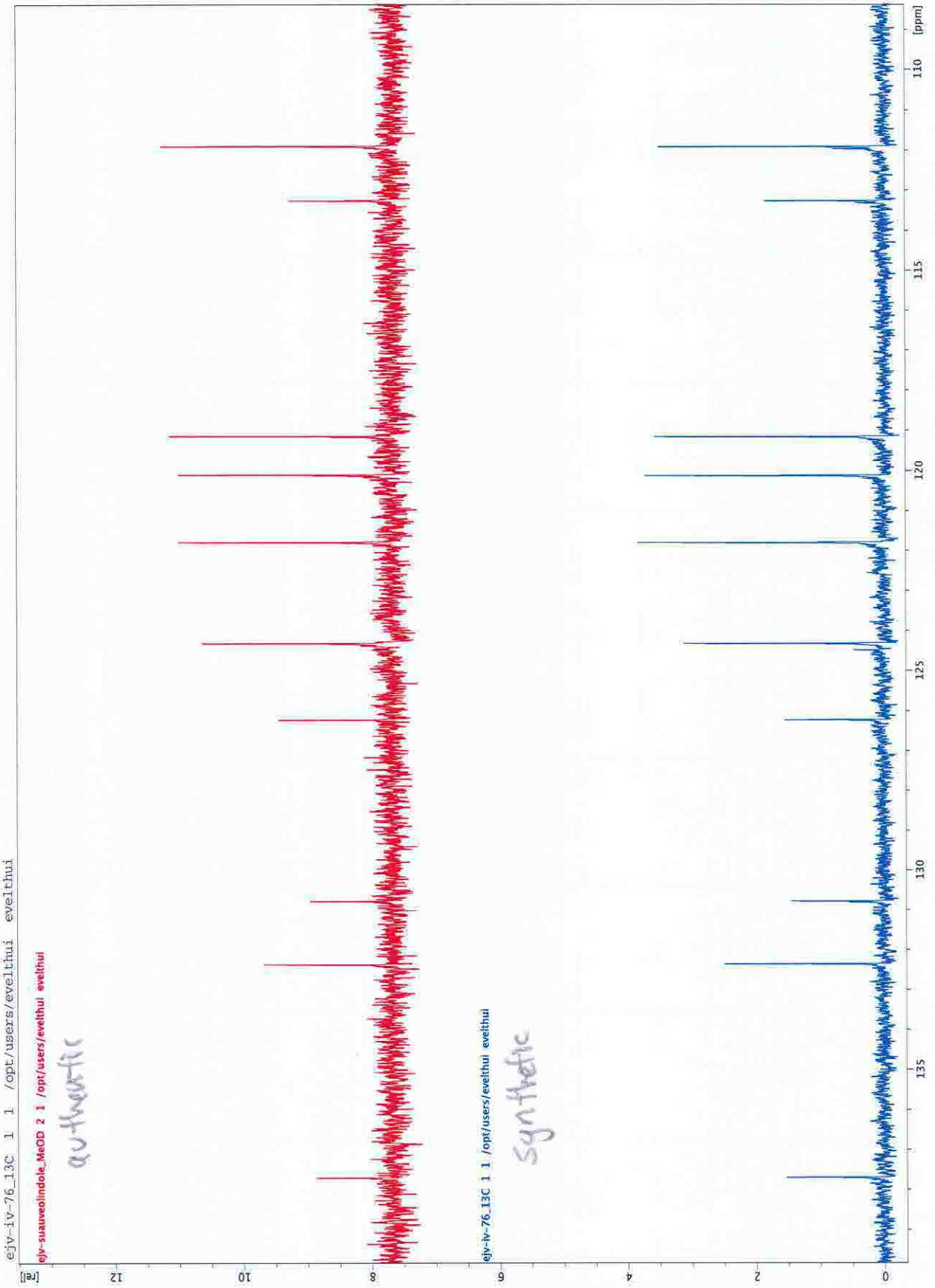
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Scale. 11.11 ppm/cm, 1677 Hz/cm

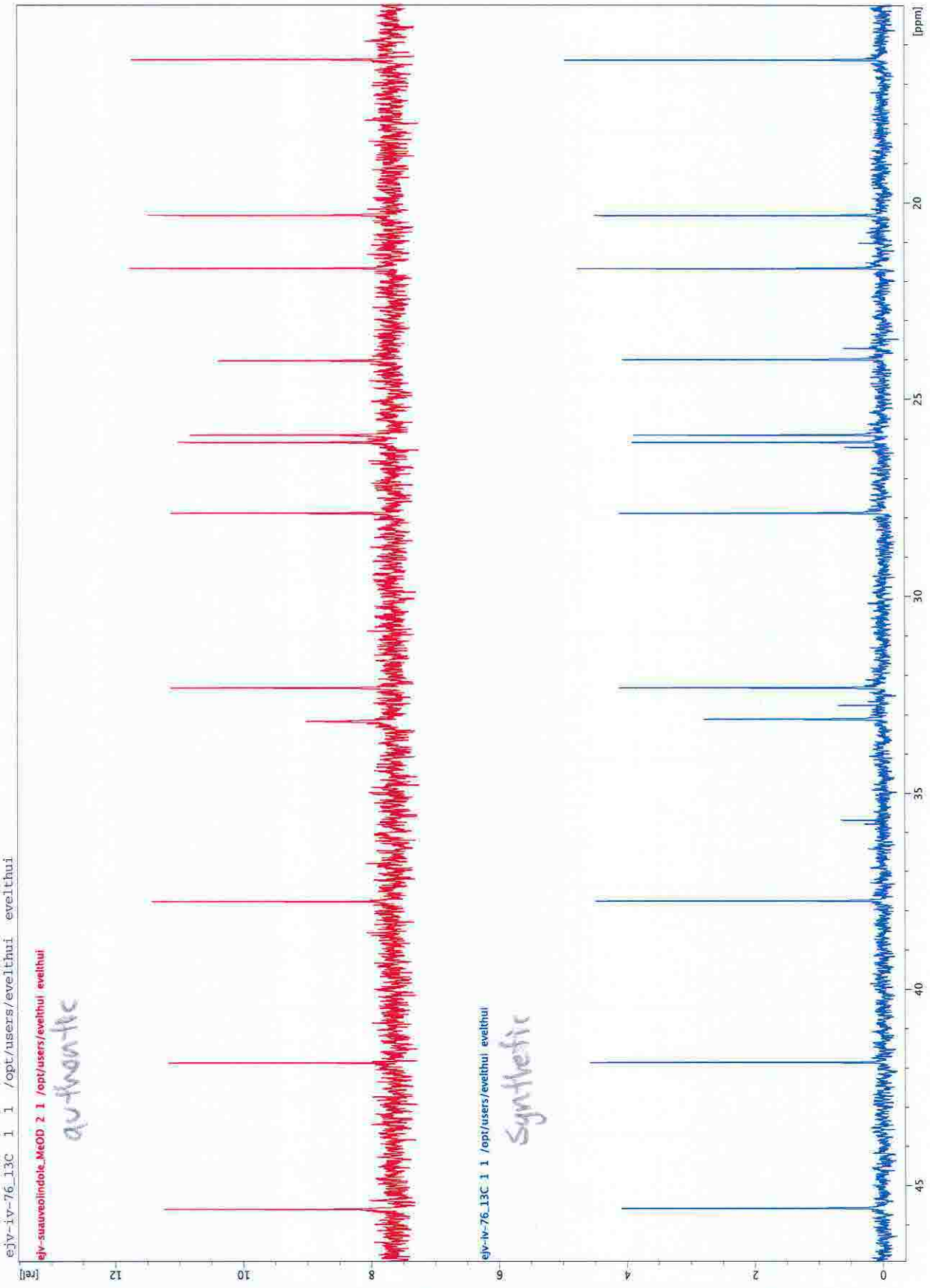

ejv-iv-76-f 1 1 /opt/users/evelthui evelthui
ejv-suauveolindole_MeOD 1 1 /opt/users/evelthui evelthui
Scale : 4.00 Shift : -0.0051 ppm = -3.0393 Hz
authentic
ejv-iv-76-f 1 1 /opt/users/evelthui evelthui
synthetic
[rel]
40
30
20
10
0
[ppm]
0
2
4
6
8
10


ejv-iv-76-f 1 1 /opt/users/evelthui evelthui
ejv-suauveolindole_MeOD 1 1 /opt/users/evelthui evelthui
Scale : 4.00 Shift : -0.0051 ppm = -3.0393 Hz
authentic
ejv-iv-76-f 1 1 /opt/users/evelthui evelthui
synthetic
[rel]
40
30
20
10
0
7.6
7.4
7.2
7.0
6.8
[ppm]


ejv-iv-76-f 1 1 /opt/users/evelthui evelthui
ejv-suauveolindole_MeOD 1 1 /opt/users/evelthui evelthui
Scale : 4.00 Shift : -0.0051 ppm = -3.0393 Hz
authentic
ejv-iv-76-f 1 1 /opt/users/evelthui evelthui
synthetic
[rel]
50
40
30
20
10
0
2.5
2.0
1.5
[ppm]


ejv-iv-76_13C 1 1 /opt/users/evelthui evelthui
ejv-suauveolindole_MeOD 2 1 /opt/users/evelthui evelthui
authentic
ejv-iv-76_13C 1 1 /opt/users/evelthui evelthui
synthetic
[rel]
[ppm]


ejv-iv-76_13C 1 1 /opt/users/evelthui evelthui
ejv-suauveolindole_MeOD 2 1 /opt/users/evelthui evelthui
authentic
ejv-iv-76_13C 1 1 /opt/users/evelthui evelthui
synthetic
[rel]
[ppm]


ejv-iv-76_13C 1 1 /opt/users/evelthui evelthui
ejv--suauveolindole_MeOD 2 1 /opt/users/evelthui evelthui
authentic
ejv-iv-76_13C 1 1 /opt/users/evelthui evelthui
Synthetic
[rel]
[ppm]