Pd(II)-Catalyzed Cyclization of Unsaturated Peroxides for the Synthesis of 1,2-Dioxanes

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Supporting Information

Contents:

General. ^{1}H and ^{13}C NMR spectra were recorded at room temperature using Bruker DRX 400, CRYO500, or GN500 MHZ spectrometers, as indicated and were referenced to residual protio solvent ($CHCl_3$: δ 7.27 ppm). The data are reported as follows: chemical shifts are reported in ppm on the δ scale, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, qu = quintet, m = multiplet, app = apparent), coupling constants (Hz), and integration. Infrared (IR) spectra were obtained using a MIDAC Prospect FT-IR spectrometer. High resolution mass spectra were acquired on a VG Analytical 7070E or Fisons Autospec spectrometer, and were obtained from peak matching. Microanalyses were performed by Atlantic Microlabs, Norcross, GA. Melting points are uncorrected. Analytical thin layer chromatography was performed on EM reagents 0.25 mm silica gel 60-F plates. Liquid chromatography was performed using forced flow (flash chromatography) of the indicated solvent system on EM reagents silica gel (SiO_2) 60 (230–400) mesh. Analytical gas chromatography (GC) was performed on an Agilent 6850 series chromatograph, equipped with an Agilent 6850 series auto sampler and flame-ionization detector. A fused silica capillary column (30 m x 320 μm x 0.25 μm) wall-coated with DB-1 (J & W Scientific) was used with helium gas carrier (25 psi column head pressure). Unless otherwise noted all reactions were run under nitrogen atmosphere. Solvents were distilled from CaH_2 or filtered through alumina before use.[1]

Caution: Organic peroxides are potentially hazardous compounds and should be handled very carefully. Solutions of anhydrous hydrogen peroxide in organic solvents are potentially explosive. When handling organic peroxides, exposure to heat, light, or redox active metal salts should be avoided. Reactions were performed on small scale and behind a safety shield.[2,3] Solutions of ethereal hydrogen peroxide were prepared as needed on small scale and should not be stored or concentrated. Further, excess amounts of the ethereal hydrogen peroxide were immediately quenched with an aqueous solution of sodium bisulfite.

Preparation and Titration of Ethereal Hydrogen Peroxide: Solutions of ethereal hydrogen peroxide were prepared by extracting 50% aqueous hydrogen peroxides with diethyl ether as previously reported in the literature.[4] A representative example of its preparation is as follows:

A solution of 50% aqueous hydrogen peroxide (20 mL) was extracted with Et_2O (20 mL). The organic layer was separated, dried over $MgSO_4$, and filtered. Titration of ethereal hydrogen peroxide solutions was achieved with $KMnO_4$ as previously reported.[5] An aqueous solution of $KMnO_4$ was prepared by dissolving $KMnO_4$ (0.1231 mg, 0.7789 mmol) into H_2O (10.00 mL). to an aliquot of the ethereal hydrogen peroxide solution (0.100 mL) was added 5 N H_2SO_4 (10 mL). The resulting solution was titrated with the $KMnO_4$ solution until the pale pink color persisted. The procedure was repeated to ensure precision.

Balanced Equation:

$$2\ KMnO_4 + 5\ H_2O_2 + 4\ H_2SO_4 \rightarrow 2\ KHSO_4 + 2\ MnSO_4 + 8\ H_2O + 5\ O_2$$

Calculations for Titrations:

Trial 1: 3.11 mL of 0.07789 M $KMnO_4$ solution = 0.242 mmol $KMnO_4$

$$0.242\ \text{mmol}\ KMnO_4 \times \frac{5\ \text{mmol}\ H_2O_2}{2\ \text{mmol}\ KMnO_4} = 0.605\ \text{mmol}\ H_2O_2$$

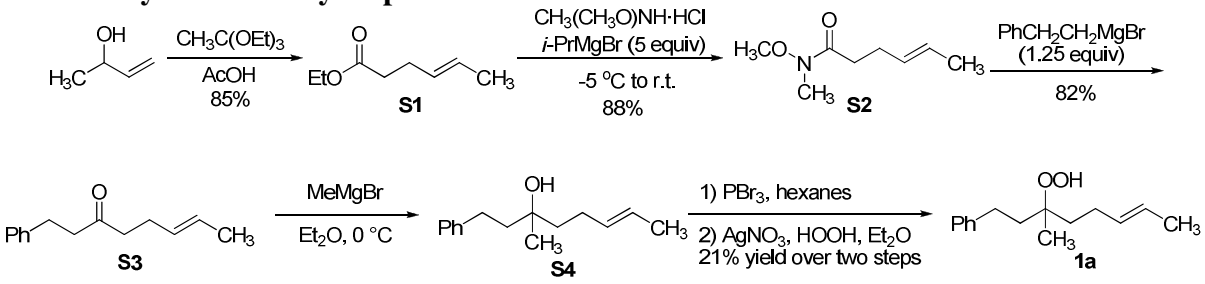
$$\frac{0.605\ \text{mmol}\ H_2O_2}{0.100\ \text{mL ethereal}\ H_2O_2} = 6.05\ \text{M}\ H_2O_2\ \text{in}\ Et_2O$$

Trial 2: 3.14 mL of 0.07789 M $KMnO_4$ solution = 0.245 mmol $KMnO_4$

$$0.245\ \text{mmol}\ KMnO_4 \times \frac{5\ \text{mmol}\ H_2O_2}{2\ \text{mmol}\ KMnO_4} = 0.613\ \text{mmol}\ H_2O_2$$

$$\frac{0.613\ \text{mmol}\ H_2O_2}{0.100\ \text{mL ethereal}\ H_2O_2} = 6.13\ \text{M}\ H_2O_2\ \text{in}\ Et_2O$$

I. Synthesis of Hydroperoxides



(*E*)-Ethyl hex-4-enoate (S1).[6] To a solution of but-3-en-2-ol (35 mL, 0.40 mol) and triethyl orthoacetate (110 mL, 0.61 mol) was added acetic acid (0.69 mL, 0.012 mol). The resulting solution was heated (140 °C) for 6 h with an air condenser and short-path distillation head affixed and then the temperature was raised (155 °C). After an additional 2 h, the solution was cooled and H_2O (100 mL) was added to the reaction mixture and the resulting mixture was extracted with Et_2O (3 × 100 mL). The organic layer was stirred with 1 N HCl for 2 h and the layers were separated. The organic layer was then washed with brine (100 mL), dried over

$MgSO_4$, and concentrated *in vacuo*. The resulting oil was distilled (boiling point: 93 °C at 45 torr) to give the ester (48 g, 85% yield, 95:5 *E*/*Z*). Characterization data matched those reported in the literature:[6] ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.25-5.47 (m, 2H), 4.10 (q, *J* = 7.2 Hz, 2H), 2.23-2.27 (m, 4H), 1.62 (d, *J* = 5.5 Hz, 3H), 1.25 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 173.4, 129.4, 126.3, 60.4, 34.5, 28.1, 18.0, 14.4.

(*E*)-*N*-Methoxy-*N*-methylhex-4-enamide (S2). To a cooled (–15 °C) slurry of $CH_3(CH_3O)NH{\cdot}HCl$ (16.3 g, 167 mmol) and the ester (19.0, 134 mmol) in THF (300 mL) was added *i*-PrMgBr (609 mL, 0.670 mol, 1.10 M in THF) over 1 h. After 3 h, NH_4Cl (300 mL) was added portionwise. The THF was removed *in vacuo* and ^{t}BuOMe (400 mL) was added. The organic layer was then washed with brine, dried over $MgSO_4$, and concentrated *in vacuo*. The resulting oil was purified by distillation to yield the amide (18.5 g, 88%). Characterization data matched those reported in the literature:[7] ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.40-5.56 (m, 2H), 3.68 (s, 3H), 3.18 (s, 3H), 2.40-2.52 (m, 2H), 2.23-2.34 (m, 2H), 1.64 (app d, *J* = 4.7 Hz, 3H).

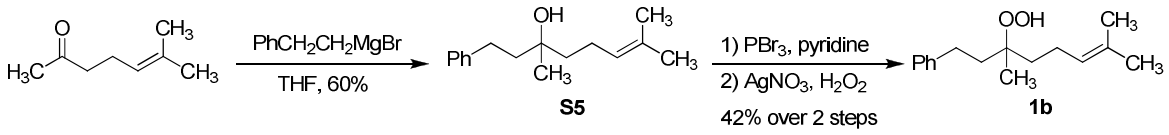
(*E*)-1-Phenyloct-6-en-3-one (S3). To a refluxing slurry of magnesium mesh (2.81 g, 116 mmol) and THF (100 mL) was added (2-bromoethyl)benzene (14.1 mL, 103 mmol) dropwise over 0.5 h. After stirring for an additional 0.5 h at reflux, the reaction mixture was cooled to ambient temperature and then added dropwise to a cooled (–15 °C) solution of the amide (13.0 g, 82.7 mmol) in THF (100 mL), maintaining reaction temperature below –5 °C. The reaction mixture was stirred at ambient temperature for 8 h and was then partitioned between NH_4Cl (200 mL) and Et_2O (200 mL). The organic layer was washed with brine (200 mL), dried over $MgSO_4$, and concentrated *in vacuo*. Purification by flash column chromatography (hexanes/EtOAc 95:5) provided the ketone (13.7 g, 82% yield) as a clear oil. Previous reports did not include characterization data:[8] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.30 (m, 2H), 7.08-7.20 (m, 3H), 5.27-5.50 (m, 2H), 2.88 (t, *J* = 7.4 Hz, 2H), 2.70 (t, *J* = 7.4 Hz, 2H), 2.42 (t, *J* = 7.3 Hz, 2H) 2.16-2.26 (m, 2H), 1.62 (d, *J* = 5.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.7, 141.4, 129.8, 128.7, 128.6, 126.3, 126.1, 44.6, 43.0, 30.0, 27.0, 18.1; HRMS (ESI) *m* / *z* calcd for $C_{14}H_{18}ONa$ $[M+Na]^+$ 225.1255, found 225.1251.

(*E*)-3-Methyl-1-phenyloct-6-en-3-ol (S4). A solution of the ketone (1.5 g, 7.2 mmol) in Et_2O (10 mL) was cooled to 0 °C and a solution of MeMgBr (3.0 M in Et_2O; 4.0 mL, 12 mmol) was added dropwise. The resulting solution was warmed to room temperature and stirred for 16 h. Upon completion of the reaction, as judged by TLC, saturated aqueous NH_4Cl (5 mL) was added slowly. The layers were separated and the aqueous layer was extracted with Et_2O (2 × 15 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated *in vacuo*. The residue was purified by flash chromatography (hexanes/EtOAc 9:1→8:2) to afford the alcohol **S4** (1.3 g, 83 % yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (m, 2H), 7.20 (m, 3H), 5.48 (m, 2H), 2.69 (m, 2H), 2.09 (m, 2H), 1.78 (m, 2H), 1.67 (m, 3H), 1.60 (m, 2H), 1.26 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.8, 131.5, 128.6, 128.5, 126.0, 125.4, 72.9, 44.1, 41.7, 30.5, 27.5, 27.1, 18.2; IR (thin film) 3394, 3026, 2933, 1602, 1494, 1375, 1115 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{15}H_{22}ONa$ $[M+Na]^+$ 241.1568, found 241.1568. Anal. Calcd for $C_{15}H_{22}O$: C, 82.52; H, 10.16. Found: C, 82.25; H, 10.21.

(*E*)-(3-Hydroperoxy-3-methyloct-6-enyl)benzene (1a). To a cooled (0 °C) solution of alcohol **S4** (2.42 g, 11.1 mmol) in dry hexanes (63 mL) was added PBr_3 (0.382 mL, 4.06 mmol). After 3 h, saturated aqueous $NaHCO_3$ (10 mL) was added to reaction mixture slowly followed by the

addition of EtOAc (30 mL). The organic layer was then washed with brine (15 mL), dried over $MgSO_4$, and concentrated *in vacuo*.

The resulting oil was dissolved in Et_2O (13 mL) and ethereal hydrogen peroxide (5.9 mL, 36 mmol; 6.1 M in Et_2O) was added to the solution. The reaction mixture was cooled (0 °C) and placed behind a blast shield without overhead light. $AgNO_3$ (1.8 g, 11 mmol) was then added through a Teflon tube in one portion, and the resulting slurry was allowed to warm to room temperature and stirred for 16 h. The reaction mixture was neutralized slowly with saturated aqueous $NaHCO_3$ (5 mL) and filtered through Celite. The organic layer was extracted, washed with saturated aqueous $NaHCO_3$ (2 × 5 mL), dried over $MgSO_4$, and concentrated *in vacuo*. The resulting oil was purified by flash column chromatography (hexanes/EtOAc 98:2) to yield hydroperoxide **1a** (0.54 g, 21% yield over two steps): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.06 (t, *J* = 7.6 Hz, 2H), 7.21 (m, 3H), 7.06 (s, 1H), 5.59 (m, 2H), 2.67 (app t, *J* = 8.4 Hz, 2H), 2.06 (m, 2H), 1.88 (ddd, *J* = 6.5, 9.5, 13.0 Hz, 2H), 1.67 (m, 5H), 1.26 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.8, 131.4, 128.7, 128.6, 126.0, 125.4, 84.5, 38.8, 36.6, 30.1, 27.0, 21.7, 18.1. IR (thin film) 3412, 3026, 2938, 1603, 1496, 1453, 1374 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{15}H_{22}O_2Na$ $[M+Na]^+$ 257.1518, found 257.1526. Anal. Calcd for $C_{15}H_{22}O_2$: C, 76.88; H, 9.46. Found: C, 76.71; H, 9.49.

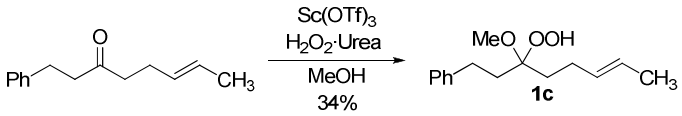


3,7-Dimethyl-1-phenyloct-6-en-3-ol (S5). To a refluxing slurry of magnesium mesh (5.13 g, 211 mmol) and THF (300 mL) was added (2-bromoethyl)benzene (26.1 mL, 192 mmol) dropwise over 0.5 h. After stirring for an additional 0.5 h at reflux, the reaction mixture was cooled to 0 °C and 6-methylhept-5-en-2-one (22.7 mL, 154 mmol) was added dropwise over 0.5 h. Saturated aqueous NH_4Cl (200 mL) was added and the resulting mixture was extracted with Et_2O (2 × 200 mL). The organic layers were combined, dried over $MgSO_4$ and concentrated *in vacuo*. The resulting yellow oil was purified by flash column chromatography (hexanes:EtOAc 8:2) to yield **S5** (20.9 g, 60% yield) as a clear oil. The characterization data matched those reported in the literature:[9] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.35 (m, 2H), 7.16-7.25 (m, 3H), 5.18 (t, *J* = 1.4 Hz, 1H), 2.68-2.78 (m, 2H), 2.04-2.16 (m, 2H), 1.78-1.85 (m, 2H), 1.74 (s, 3H), 1.68 (s, 3H), 1.50-1.60 (m, 2H), 1.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.9, 132.1, 128.7, 128.6, 126.0, 124.6, 72.9, 44.1, 42.0, 30.7, 27.1, 26.0, 23.0, 17.9.

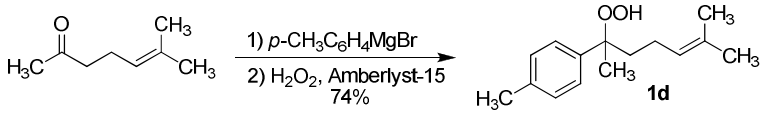
(3-Hydroperoxy-3,7-dimethyloct-6-enyl)benzene (1b). To a cooled (0 °C) solution of the alcohol (10.0 g, 43.0 mmol) in hexanes (70 mL) were added PBr_3 (1.75 mL, 18.1 mmol) and pyridine (1.50 mL, 18.6 mmol). After 3 h, saturated aqueous $NaHCO_3$ (50 mL) and EtOAc (50 mL) were added to the reaction mixture. The organic layer was then washed with saturated aqueous NH_4Cl (2 × 50 mL) and brine (50 mL) and was dried over $MgSO_4$. The organic layer was concentrated *in vacuo*.

The resulting oil was dissolved in Et_2O (70 mL) and ethereal hydrogen peroxide (71 mL, 430 mmol; 6.1 M in Et_2O) and $NaHCO_3$ (7.3 g, 87 mmol) were added to the solution. The reaction mixture was cooled (0 °C) and placed behind a blast shield. $AgNO_3$ (7.3 g, 43 mmol) was then added through a Teflon tube portion-wise and the resulting slurry was stirred for 3 h. The reaction mixture was washed with saturated aqueous $NaHCO_3$ (2 × 100 mL) and brine. The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The resulting oil was purified

by flash chromatography (hexanes/EtOAc 19:1) to yield hydroperoxide **1b** as a colorless oil (4.5 g, 42% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.25-7.32 (m, 2H), 7.17-7.25 (m, 3H), 5.20 (t, *J* = 1.4 Hz, 1H), 2.67-2.73 (m, 2H), 2.0 (q, *J* = 7.8 Hz, 2H), 1.85-1.97 (m, 3H), 1.60-1.74 (m, 1H), 1.74 (s, 3H), 1.68 (s, 3H), 1.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.9, 132.3, 128.7, 128.5, 126.0, 124.6, 84.5, 38.9, 36.8, 30.3, 25.9, 22.6, 21.7, 17.9; HRMS (ESI) *m* / *z* calcd for $C_{16}H_{24}O_2Na$ $[M+Na]^+$ 271.1674, found 271.1676.



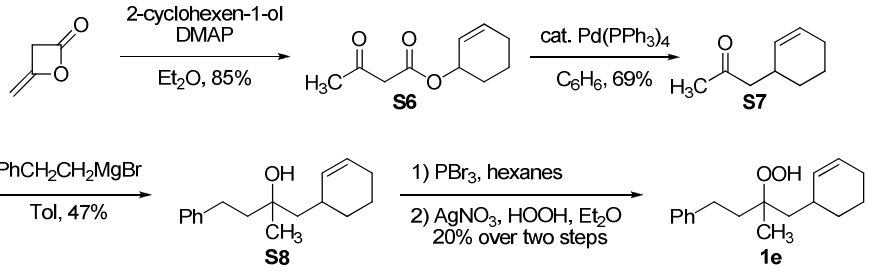
(*E*)-(3-Hydroperoxy-3-methoxyoct-6-enyl)benzene (1c). To a solution of the ketone (5.0 g, 25 mmol) in MeOH (480 mL) were added urea-hydrogen peroxide (9.3 g, 99 mmol) and $Sc(OTf)_3$ (0.61 g, 1.2 mmol). After stirring for 12 h, the reaction mixture was concentrated and dissolved in EtOAc (200 mL). The organic layer was washed with saturated aqueous $NaHCO_3$ (2 × 200 mL), dried over $MgSO_4$, and concentrated *in vacuo*. The resulting clear oil was purified by flash chromatography (hexanes/EtOAc 19:1) to yield peroxyacetal **1c** as a colorless oil (3.3 g, 34% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (s, 1H), 7.23-7.30 (m, 2H), 7.15-7.22 (m, 3H), 5.34-5.52 (m, 2H), 3.33 (s, 3H), 2.64-2.78 (m, 2H), 1.95-2.15 (m, 3H), 1.93-1.68 (m, 3H), 1.65 (d, *J* = 5.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.8, 130.4, 128.7, 128.4, 126.2, 125.7, 108.6, 48.8, 33.0, 30.7, 30.2, 26.9, 18.1; HRMS (ESI) *m* / *z* calcd for $C_{15}H_{22}O_3Na$ $[M+Na]^+$ 273.1467, found 273.1473. Anal. Calcd for $C_{15}H_{22}O_3$: C, 71.97; H, 8.86. Found: C, 71.99; H, 9.00.



1-(2-Hydroperoxy-6-methylhept-5-en-2-yl)-4-methylbenzene (1d).[10] To a refluxing slurry of magnesium mesh (7.02 g, 0.289 mol) and THF (550 mL) was added 4-bromotoluene (45.0 g, 0.263 mol) dropwise over 0.5 h. After stirring for an additional 0.5 h at reflux, the reaction mixture was cooled to 0 °C and the ketone (31.0 mL, 0.210 mol) was added dropwise over 0.5 h. Saturated aqueous NH_4Cl (200 mL) was added and the resulting mixture was extracted with Et_2O (2 × 200 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated *in vacuo*. The resulting yellow oil was purified by flash chromatography (hexanes/ Et_2O 95:5→85:15) to yield 6-methyl-2-*p*-tolylhept-5-en-2-ol (36.6 g, 80% yield) as a clear oil. Characterization data matched those reported in the literature:[11] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 6.8 Hz, 2H), 7.17 (d, *J* = 7.2 Hz, 2H), 5.13 (t, *J* = 6.9 Hz, 1H), 2.37 (s, 3H), 2.01 (s, 1H), 2.01-1.85 (m, 2H), 1.90-1.85 (m, 2H), 1.68 (s, 3H), 1.55 (s, 3H), 1.53 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.2, 136.2, 132.3, 129.0, 125.0, 124.5, 75.1, 43.9, 30.6, 25.9, 23.2, 21.2, 17.9; HRMS (ESI) *m* / *z* calcd for $C_{15}H_{22}ONa$ $[M+Na]^+$ 241.1568, found 241.1569.

To a cooled solution of 6-methyl-2-*p*-tolylhept-5-en-2-ol (11 g, 49 mmol) and ethereal hydrogen peroxide (82 mL, 0.50 mol; 6.1 M in Et_2O) in Et_2O (120 mL) was added Amberlyst-15 (5.0 g, 24 mmol; 4.7 meq/g). The resulting slurry was stirred for 18 h at ambient temperature and then filtered. The resulting solution was washed with saturated aqueous $NaHCO_3$ (2 × 100 mL), dried over $MgSO_4$, and concentrated *in vacuo*. The resulting oil was purified by flash chromatography (hexanes/EtOAc 9:1) to yield hydroperoxide **1d** as a colorless oil (8.5 g, 74% yield). The previous literature report does not provide NMR characterization data:[10] ^{1}H NMR

(400 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.2 Hz, 2H), 7.31 (s, 1H), 7.21 (d, *J* = 8.2 Hz, 2H), 5.10 (t, *J* = 6.9 Hz, 1H), 2.37 (s, 3H), 2.06-1.80 (m, 2H), 1.93-1.78 (m, 2H), 1.67 (s, 3H), 1.66 (s, 3H), 1.56 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.2, 137.2, 132.3, 129.4, 125.9, 124.3, 86.4, 39.7, 25.9, 23.0, 22.9, 21.3, 17.8; HRMS (ESI) *m* / *z* calcd for $C_{15}H_{22}O_2Na$ $[M+Na]^+$ 257.1518, found 257.1511.



Cyclohex-2-enyl 3-oxobutanoate (S6).[12] To a solution of diketene (1.4 g, 17 mmol) and 2-cyclohexen-1-ol (1.5 g, 15 mmol) in Et_2O (56 mL) was added *N*,*N*-dimethylaminopyridine (0.093 g, 0.76 mmol). After stirring the resulting solution for 0.5 h, saturated aqueous NH_4Cl (30 mL) was added. The organic layer was then washed with brine, dried over $MgSO_4$, and concentrated *in vacuo*. The resulting oil was purified by flash chromatography (hexanes/EtOAc 9:1) to yield the β-ketoester (2.3 g, 85% yield). Characterization data matched those reported in the literature:[12] δ ^{1}H NMR (500 MHz, $CDCl_3$) δ 12.15 (br s, 10% enol OH), 5.99 (ddt, *J* = 1.0, 4.4, 10.0 Hz, 1H), 5.73 (dd, *J* = 1.8, 10.0 Hz, 1H), 5.33 (br s, 1H), 4.99 (s, 10% enol CH), 3.45 (s, 2H), 2.28 (s, 3H), 2.10 (m, 1H), 2.00 (m, 1H), 1.89 (m, 1H), 1.81-1.69 (m, 2H), 1.65 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 200.9, 167.0, 133.6, 125.2, 69.4, 50.7, 30.3, 28.3, 25.0, 18.9.

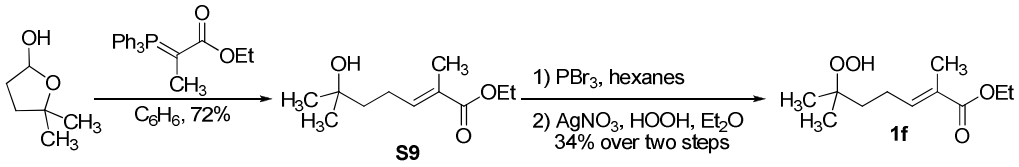
1-(Cyclohex-2-enyl)propan-2-one (S7).[13] To a solution of $Pd(PPh_3)_4$ (0.74 g, 0.64 mmol) in benzene (20 mL) was added the β-ketoester (2.3 g, 13 mmol). The solution was allowed to stir for 48 h, at which point another portion of $Pd(PPh_3)_4$ (0.10 g, 0.86 mmol) was added. After stirring another 48 h, the reaction was determined to be complete by TLC. The mixture was concentrated *in vacuo* and purified directly by flash chromatography (hexanes/Et_2O 95:5) to yield the desired unsaturated ketone (1.2 g, 75% yield). The characterization data matched those reported in the literature:[13] ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.70 (ddd, *J* = 3.5, 5.8, 10.2 Hz, 1H), 5.50 (ddd, *J* = 2.0, 4.2, 10.2 Hz, 1H), 2.64 (br m, 1H), 2.42 (t, *J* = 6.6 Hz, 2H), 2.15 (s, 3H), 1.98 (br m, 2H), 1.81 (m, 1H), 1.69 (m, 1H), 1.57 (m, 1H), 1.21 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 208.6, 130.6, 128.2, 50.3, 31.4, 30.7, 29.1, 25.2, 21.2.

1-(Cyclohex-2-enyl)-2-methyl-4-phenylbutan-2-ol (S8). To a refluxing slurry of magnesium strips (0.353 g, 14.7 mmol) and THF (27 mL) was added (2-bromoethyl)benzene (2.27 g, 12.3 mmol) dropwise over 1 h. The reaction mixture was then cooled to 0 °C and the ketone (1.22 g, 9.81 mmol) was added dropwise over 1 h. The mixture was then warmed to ambient temperature and stirred for 1.5 h. The solution was then heated to reflux for 2 h and allowed to cool to ambient temperature. Saturated aqueous NH_4Cl (10 mL) was then added and then the resulting mixture was extracted with Et_2O (2 × 30 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated *in vacuo*. The resulting yellow oil was purified by flash chromatography to yield the desired alcohol as a 50:50 mixture of diastereomers (1.10 g, 46% yield) as a colorless oil. The product was characterized as a mixture of diastereomers: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.8 Hz, 2H), 7.20 (m, 3H), 5.68 (m, 2H), 2.71 (m, 2H), 2.34 (br s, 1H), 1.98 (m, 2H), 1.88 (m, 1H), 1.82 (m, 2H), 1.72 (m, 1H), 1.64-1.52 (m, 3H), 1.36 (q, *J* =

10.2 Hz, 1H), 1.30 (d, *J* = 2.3 Hz, 3H), 1.23 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.8, 133.13, 133.07, 128.7, 128.6, 127.11, 127.07, 126.0, 73.50, 48.6, 45.0, 44.9, 31.42, 31.37, 31.30, 31.27, 27.5, 24.5, 25.2, 21.6, 21.5; IR (thin film) 3430, 3023, 2925, 1699, 1602, 1496 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{17}H_{24}ONa$ $[M+Na]^+$ 267.1725, found 257.1716.

4-(Cyclohex-2-enyl)-3-hydroperoxy-3-methylbutyl)benzene (1e). To a cooled (0 °C) solution of the alcohol (0.52 g, 2.1 mmol) in dry hexanes (12 mL) was added PBr_3 (66 μL, 0.70 mmol). After 3 h, saturated aqueous $NaHCO_3$ (5 mL) and EtOAc (10 mL) were added to the reaction mixture. The layers were separated and then the organic layer was washed with brine (10 mL), was dried over $MgSO_4$, and concentrated *in vacuo*.

The resulting oil was dissolved in Et_2O (40 mL) and ethereal hydrogen peroxide (1.6 mL, 0.010 mol; 6.1 M in Et_2O) was added to the solution. The reaction mixture was cooled (0 °C) and placed behind a blast shield without overhead light. $AgNO_3$ (0.51 g, 3.0 mmol) was then added through a Teflon tube in one portion and the resulting slurry was allowed to warm to room temperature and stirred for 16 h. Saturated aqueous $NaHCO_3$ (3 mL) was added to the solution and the mixture was filtered through a pad of Celite. The organic layer was extracted and washed again with saturated aqueous $NaHCO_3$ (2 × 3 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The resulting oil was purified by flash chromatography (hexanes/EtOAc 98:2) to yield hydroperoxide **1e** as a 50:50 mixture of diastereomers (0.10 g, 20% yield over two steps). The product was characterized as a mixture of diastereomers: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.6 Hz, 4H), 7.22 (m, 6H), 7.08 (s, 1H), 7.04 (s, 1H), 5.79 (m, 1H), 5.72 (m, 1H), 5.70 (m, 2H), 2.67 (m, 4H), 2.33 (m, 2H) 1.99 (m, 5H), 1.96-1.81 (m, 6H), 1.79-1.67 (m, 4H), 1.65-1.52 (m, 3H), 1.36 (m, 2H), 1.31 (s, 3H), 1.30 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.8, 142.7, 132.7, 132.5, 128.66, 128.64, 128.57, 128.55, 127.7, 127.6, 126.06, 125.99, 85.2, 85.1, 43.1, 43.0, 39.9, 39.3, 30.89, 30.85, 30.76, 30.75, 30.34, 30.27, 30.26, 30.25, 22.2, 21.7, 21.3, 21.1; IR (thin film) 3405, 3025, 2931, 1602, 1496 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{17}H_{24}O_2Na$ $[M+Na]^+$ 283.1674, found 283.1671.

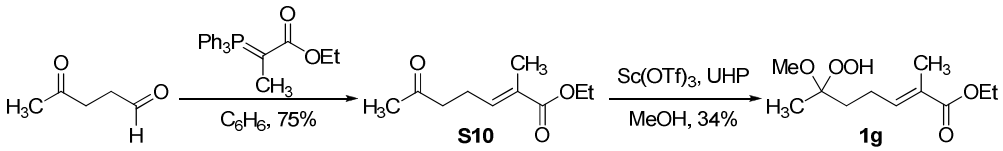


Ethyl (*E*)-6-hydroxy-2,6-dimethylhept-2-enoate (S9). The crude lactol (3.06 g, 26.3 mmol) synthesized by known procedures,[14] was dissolved in benzene (130 mL) and carbethoxyethylidenetriphenyl-phosphorane[15] (10.4 g, 28.9 mmol) was added. The resulting solution was heated to reflux for 2 h. The mixture was concentrated *in vacuo* until about 3 mL of solution remained. The slurry was purified by flash chromatography (hexanes/EtOAc 8:2) to afford the enone as a pale yellow oil (3.80 g, 72% yield). Characterization data matched those reported in the literature:[16] ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.78 (t, *J* = 7.6 Hz, 1H), 4.19 (q, *J* = 7.2 Hz, 2H), 2.24 (dt, *J* = 7.6, 8.6 Hz, 2H), 1.86 (s, 3H), 1.61 (m, 2H), 1.30 (t, *J* = 7.2 Hz, 3H), 1.26 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.4, 142.2, 128.1, 70.9, 60.7, 42.5, 29.5, 24.0, 14.5, 12.5.

(E)-Ethyl 6-hydroperoxy-2,6-dimethylhept-2-enoate (1f). To a cooled (0 °C) solution of the alcohol (1.5 g, 7.5 mmol) in dry hexanes (40 mL) was added PBr_3 (240 μL, 2.5 mmol). After 3

h, saturated aqueous $NaHCO_3$ (10 mL) and EtOAc (20 mL) were added to the reaction mixture. The organic layer was washed with brine (10 mL), dried over $MgSO_4$, and concentrated *in vacuo*.

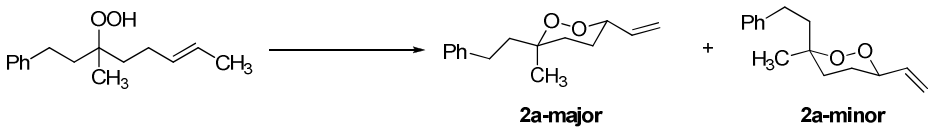
The resulting oil was dissolved in Et_2O (13 mL) and ethereal hydrogen peroxide (4.9 mL, 0.030 mol; 6.1 M in Et_2O) was added to the solution. The reaction mixture was cooled (0 °C) and placed behind a blast shield without overhead light. $AgNO_3$ (1.6 g, 9.4 mmol) was then added through a Teflon tube in one portion and the resulting slurry was allowed to warm to room temperature and stirred for 16 h. The reaction mixture was neutralized slowly with saturated aqueous $NaHCO_3$ (10 mL) and filtered through a pad of Celite. The organic layer was extracted and washed again with saturated aqueous $NaHCO_3$ (2 × 10 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The resulting oil was purified by flash chromatography (hexanes/EtOAc 95:5→90:10) to yield hydroperoxide **1f** (0.46 g, 34% yield over two steps). (A portion of the starting alcohol could be recovered by flushing the column with EtOAc and concentrating *in vacuo*.) Characterization data for hydroperoxide: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.77 (td, *J* = 1.2, 7.3 Hz, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 2.24 (q, *J* = 7.6 Hz, 2H), 1.86 (s, 3H), 1.71 (m, 2H), 1.30 (t, *J* = 7.2 Hz, 3H), 1.26 (s, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.5, 142.1, 128.2, 82.5, 60.7, 37.2, 24.1, 23.5, 14.5, 12.5; IR (thin film) 3402, 2981, 1708, 1691, 1646 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{11}H_{20}O_4Na$ $[M+Na]^+$ 239.1259, found 239.1259.



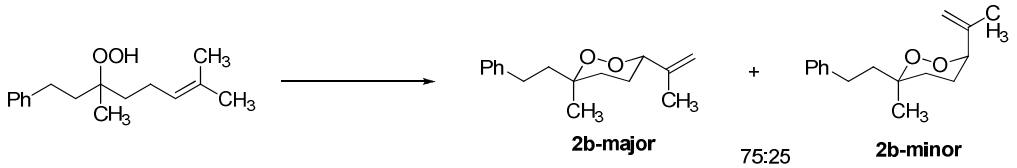
Ethyl (*E*)-2-methyl-6-oxohept-2-enoate (S10). 4-Oxopentanal (2.3 g, 23 mmol) synthesized by known procedures,[17] was dissolved in CH_2Cl_2 (110 mL) and carbethoxyethylidenetriphenylphosphorane[15] (8.3 g, 23 mmol) was added portionwise. The resulting mixture was stirred at ambient temperature for 16 h. The solution was then concentrated *in vacuo* until approximately 3 mL remained. The resulting slurry was purified by flash chromatography (hexanes/EtOAc 9:1) to afford the enone as a pale yellow oil (2.3 g, 75% yield). Characterization data matched those reported in literature:[18] ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.67 (tq, *J* = 1.3, 7.3 Hz, 1H), 4.19 (q, *J* = 7.0 Hz, 2H), 2.59 (t, *J* = 7.3 Hz, 2H), 2.44 (q, *J* = 7.3 Hz, 2H), 2.17 (s, 3H), 1.86 (d, *J* = 1.3 Hz, 3H), 1.29 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 207.5, 166.2, 140.2, 129.1, 60.7, 42.3, 30.2, 22.9, 14.5, 12.6.

Ethyl (*E*)-6-hydroperoxy-6-methoxy-2-methylhept-2-enoate (1g). To a solution of the ketone (1.4 g, 7.4 mmol) in MeOH (125 mL) was added scandium trifluoromethanesulfonate (0.37 g, 0.74 mmol) and urea hydrogen peroxide (5.2 g, 56 mmol).[19] The reaction mixture was allowed to stir for 2 h at ambient temperature. Saturated aqueous $NaHCO_3$ (30 mL) and CH_2Cl_2 (100 mL) were added to the mixture. The resulting slurry was filtered and extracted with saturated aqueous $NaHCO_3$ (2 × 20 mL). The organic layer was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by flash chromatography (hexanes: EtOAc 9:1→8:2), affording mixed peroxyacetal **1g** (0.58 g, 34% yield): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (s, 1H), 6.75 (tq, *J* = 1.7, 7.5 Hz, 1H), 4.20 (q, *J* = 6.9 Hz, 2H), 3.33 (s, 3H), 2.27 (m, 2H), 1.90-1.79 (m, 2H), 1.86 (s, 3H), 1.37 (s, 3H), 1.30 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.4, 141.2, 128.6, 106.9, 60.7, 49.2, 34.2, 23.7, 19.1, 14.5, 12.6; IR (thin film) 3392, 3025, 2931, 1708, 1691 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{11}H_{20}O_5Na$ $[M+Na]^+$ 255.1208, found 255.1204.

II. Synthesis of 1,2-Dioxanes

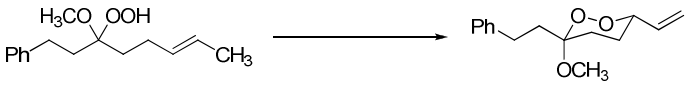


3-Methyl-3-phenethyl-6-vinyl-1,2-dioxane (2a). To a slurry of $Pd(OAc)_2$ (0.0072 g, 0.032 mmol), pyridine (11 μL, 0.13 mmol), benzoquinone (0.0070 g, 0.064 mmol), and Ag_2CO_3 (0.35 g, 1.3 mmol) in 1,4-dioxane (5.5 mL) was added a solution of hydroperoxide (0.15 g, 0.64 mmol) in 1,4-dioxane (2 mL). The reaction mixture was heated (80 °C) for 3 h and then concentrated. The resulting residue was purified by flash chromatography (hexanes/EtOAc 95:5) to yield **2a** as a mixture of diastereomers (0.050 g, 30% yield) in a 60:40 ratio. Characterization data for major diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (m, 3H), 7.20 (m, 2H), 5.83 (dddd, *J* = 6.0, 10.6, 16.9, 16.9 Hz, 1H), 5.34 (d, *J* = 17.5 Hz, 1H), 5.26 (d, *J* = 11.0 Hz, 1H), 4.53 (br m, 1H), 2.77 (td, *J* = 12.6, 4.6 Hz, 1H), 2.62 (td, *J* = 12.6, 4.8 Hz, 1H), 2.30 (td, *J* = 13.6, 4.5 Hz, 1H), 1.79 (m, 3H), 1.74 (m, 2H), 1.43 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.4, 135.6, 128.64, 128.59, 126.1, 118.4, 81.1, 79.8, 37.0, 32.5, 29.6, 25.7, 20.8. Characterization data for minor diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (m, 3H), 7.24 (d, *J* = 8.4 Hz, 2H), 5.80 (ddd, *J* = 6.1, 10.0, 16.3 Hz, 1H), 5.33 (d, *J* = 17.9 Hz, 1H), 5.25 (d, *J* = 10.8 Hz, 1H), 4.53 (br m, 1H), 2.69 (m, 2H), 1.85 (m, 4H), 1.74 (m, 2H), 1.22 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 142.9, 135.5, 128.60, 128.57, 126.0, 118.5, 81.4, 79.6, 42.5, 33.0, 30.0, 25.8, 24.3. Characterization for mixture of isomers: IR (thin film) 3085, 3025, 2940, 1643, 1602, 1494 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{15}H_{20}O_2Na$ $[M+Na]^+$ 255.1361, found 255.1362. Anal. Calcd for $C_{15}H_{20}O_2$: C, 77.55; H, 8.68. Found: C, 77.64; H, 8.79.



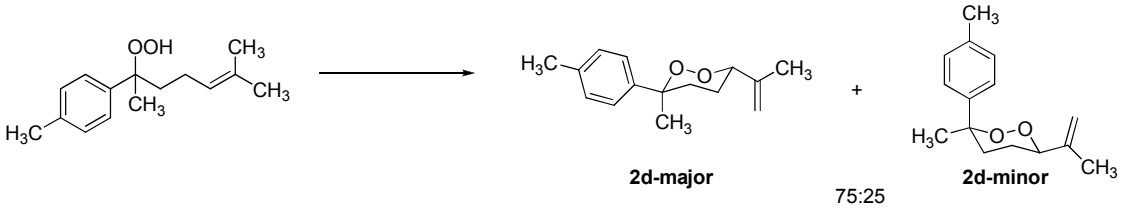
3-Methyl-3-phenethyl-6-(prop-1-en-2-yl)-1,2-dioxane (2b). To a slurry of $Pd(OAc)_2$ (0.0040 g, 0.018 mmol), pyridine (6.0 μL, 0.072 mmol), and benzoquinone (0.027 g, 0.25 mmol) in toluene (1 mL) was added a solution of hydroperoxide (0.51 g, 0.21 mmol) in toluene (1 mL). The reaction mixture was heated (70 °C) for 3 h and then concentrated. The resulting residue was purified by flash chromatography (hexanes/EtOAc 19:1) to yield **2b** as a mixture diastereomers (0.018 g, 35% yield) in a 75:25 ratio. Characterization data for major diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.5 Hz, 3H), 7.21 (d, *J* = 7.1 Hz, 2H), 5.01 (s, 1H), 4.97 (s, 1H), 4.45 (d, *J* = 10.7 Hz, 1H), 2.71 (m, 1H), 1.97 (m, 2H), 1.79-1.82 (m, 1H), 1.82 (s, 3H), 1.78 (m, 4H), 1.46 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.0, 142.3, 128.5, 128.4, 125.9, 113.0, 83.6, 79.7, 42.6, 32.9, 29.5, 24.5, 20.5, 19.6. Characterization data for minor diastereomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (t, *J* = 7.5 Hz, 3H), 7.21 (d, *J* = 7.1 Hz, 2H), 5.00 (s, 1H), 4.95 (s, 1H), 4.45 (d, *J* = 10.7 Hz, 1H), 2.79 (td, *J* = 4.4, 12.9 Hz, 1H), 2.63 (td, *J* = 5.9, 13.1 Hz, 1H), 2.34 (td, *J* = 4.4, 12.9 Hz, 1H), 1.96 (m, 1H), 1.81 (s, 3H), 1.78 (m,

4H), 1.23 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.0, 142.8, 128.5, 128.4, 125.8, 113.0, 83.2, 79.5, 36.8, 33.4, 29.9, 24.5, 24.2, 19.6. Characterization for both isomers: IR (thin film) 3054, 1550, 1474, 1194 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{16}H_{22}O_2Na$ $[M+Na]^+$ 269.1518, found 269.1528. Anal. Calcd for $C_{16}H_{22}O_2$: C, 78.01; H, 9.00. Found: C, 78.23; H, 8.81.



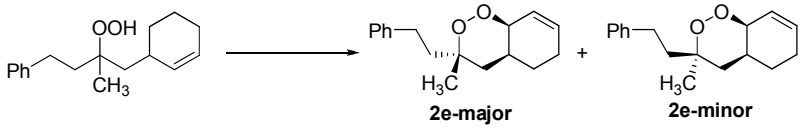
2c
>97:3 dr

3-Methoxy-3-phenethyl-6-vinyl-1,2-dioxane (2c). To a slurry of $Pd(OAc)_2$ (0.044 g, 0.20 mmol), pyridine (65 μL, 0.80 mmol), and benzoquinone (0.43 g, 4.0 mmol) in toluene (20 mL) was added a solution of the hydroperoxide (1.0 g, 4.0 mmol) in toluene (20 mL). The reaction mixture was heated (40 °C) for 5 h and then concentrated. The resulting residue was purified by flash chromatography (hexanes/EtOAc 19:1) to yield **2c** with a diastereomeric ratio of >97:3 as determined by ^{1}H NMR spectroscopy (0.34 g, 34% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (m, 2H), 7.19 (m, 3H), 6.15 (ddd, *J* = 4.7, 10.7, 17.2 Hz, 1H), 5.32 (dt, *J* = 17.2, 2.0 Hz, 1H), 5.27 (dt, *J* = 10.7, 2.0 Hz, 1H), 4.56 (m, 1H), 3.37 (s, 3H), 2.64 (m, 2H) 2.29 (m, 1H), 1.95 (m, 1H), 1.91-1.73 (m, 3H), 1.65 (dq, *J* = 4.7, 13.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.6, 136.9, 128.7, 128.4, 126.3, 117.1, 103.7, 79.5, 49.0, 34.3, 29.3, 26.4, 23.1; IR (thin film) 3120, 2980, 2318, 1612, 1485 cm^{-1}; (ESI) *m* / *z* calcd for $C_{15}H_{20}O_3Na$ $[M+Na]^+$ 271.1310, found 271.1311. Anal. Calcd for $C_{15}H_{20}O_3$: C, 72.55; H, 8.12. Found: C, 72.55; H, 8.08.



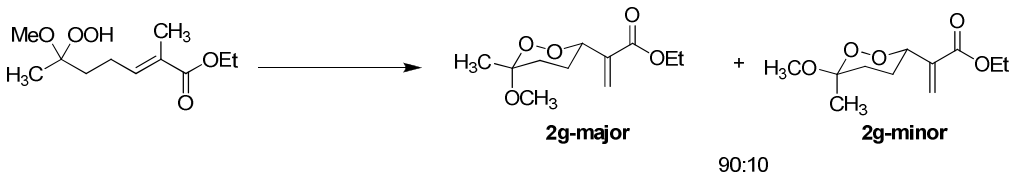
2d-major 75:25 **2d-minor**

3-Methyl-6-(prop-1-en-2-yl)-3-*p*-tolyl-1,2-dioxane (2d). To a slurry of $Pd(OAc)_2$ (0.021 g, 0.094 mmol), pyridine (0.030 mL, 0.37 mmol), and benzoquinone (0.47 g, 4.3 mmol) in toluene (7 mL) was added a solution of hydroperoxide (1.0 g, 4.3 mmol) in toluene (7 mL). The reaction mixture was heated (80 °C) for 3 h and then concentrated. The resulting residue was purified by flash chromatography (hexanes/EtOAc 19:1) to yield **2d** as a 75:25 mixture of diastereomers (0.030 g, 30% yield). Characterization data for major diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 (d, *J* = 7.4 Hz, 2H), 7.19 (d, *J* = 7.5 Hz, 2H), 5.06 (s, 1H), 5.01 (s, 1H), 4.50 (dd, *J* = 3.0, 10.2 Hz, 1H), 2.36 (s, 3H), 2.14 (m, 2H), 2.12-2.03 (m, 1H), 1.89 (m, 1H), 1.86 (s, 3H), 1.65 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.3, 142.4, 137.2, 129.2, 124.9, 113.1, 83.4, 81.1, 33.35, 24.6, 21.24, 19.9. Characterization data for minor diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.2 Hz, 2H), 7.19 (d, *J* = 7.5 Hz, 2H), 4.85 (s, 2H), 4.50 (dd, *J* = 3.8, 9.3 Hz, 1H), 2.56 (dt, *J* = 3.8, 13.8 Hz, 1H), 2.36 (s, 3H), 2.07 (m, 1H), 2.00 (m, 1H), 1.68 (m, 1H), 1.65 (s, 3H), 1.38 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.7, 141.1, 136.3, 129.1, 125.9, 113.5, 84.0, 82.4, 33.37, 30.5, 25.2, 21.22, 19.7. Characterization for mixture of isomers: HRMS (ESI) *m* / *z* calcd for $C_{15}H_{20}O_2Na$ $[M+Na]^+$ 255.1361, found 255.1364. Anal. Calcd for $C_{15}H_{20}O_2$: C, 77.55; H, 8.68. Found: C, 77.39; H, 8.66.

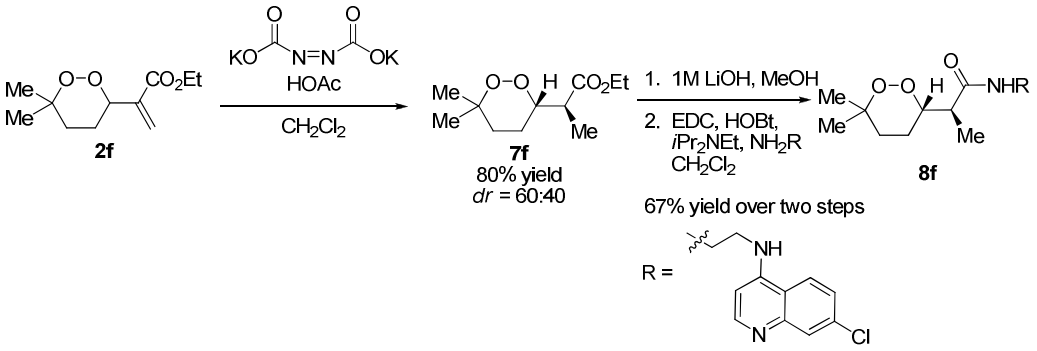


3-Methyl-3-phenethyl-3,4,4a,5,6,8a-hexahydrobenzo[c][1,2]dioxine (2e). To a slurry of $Pd(OAc)_2$ (0.0083 g, 0.037 mmol), pyridine (12 μL, 0.15 mmol), benzoquinone (0.0080 g, 0.074 mmol), and Ag_2CO_3 (0.41 g, 1.5 mmol) in 1,4-dioxane (3 mL) was added a solution of hydroperoxide (0.093 g, 0.74 mmol) in 1,4-dioxane (1.5 mL). The reaction mixture was heated (80 °C) for 3 h and then concentrated. The resulting residue was purified by flash chromatography (hexanes/EtOAc 95:5) to yield **2e** as a colorless oil (0.032 g, 35% yield) as a mixture of four diastereomers in a 48:30:16:6 ratio, where only the two major diastereomers shown above are fully characterized. Due to broad peaks in the ^{13}C NMR spectrum, the sample was heated to 50 °C during data acquisition to resolve peaks. Characterization data for major diastereomer: ^1H NMR (500 MHz, $CDCl_3$) δ 7.28 (m, 2H), 7.20 (m, 3H), 5.93 (m, 1H), 5.75 (m, 1H), 4.54 (m, 1H), 2.64 (m, 2H), 2.20 (m, 1H), 2.11 (m, 2H), 1.95 (m, 1H), 1.74 (m, 3H), 1.63 (m, 2H), 1.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$, 50 °C) δ 142.9, 132.1, 128.6, 128.6, 126.4, 126.0, 79.4, 76.6, 42.2, 35.3, 30.1, 29.8, 25.7, 22.90, 22.86. Characterization data for minor diastereomer: ^1H NMR (500 MHz, $CDCl_3$) δ 7.28 (m, 2H), 7.20 (m, 3H), 5.95 (m, 1H), 5.75 (m, 1H), 4.57 (m, 1H), 2.72 (m, 2H), 2.20 (m, 1H), 2.11 (m, 1H), 1.99 (m, 2H), 1.83 (m, 1H), 1.74 (m, 2H), 1.65 (m, 2H), 1.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.7, 132.1, 128.60, 128.57, 126.1, 126.0, 79.7, 76.8, 39.7, 34.8, 30.1, 29.0, 25.7, 24.8, 22.7. Characterization for mixture of all isomers: IR (thin film) 3027, 2923, 1650, 1602, 1496 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{17}H_{22}O_2Na$ $[M+Na]^+$ 281.1518, found 281.1520. Anal. Calcd for $C_{17}H_{22}O_2$: C, 79.03; H, 8.58. Found: C, 79.26; H, 8.68.

Ethyl (*E*)-3-(6,6-dimethyl-1,2-dioxan-3-yl)-2-methylacrylate (2f). To a slurry of $Pd(OAc)_2$ (0.018 g, 0.079 mmol), pyridine (26 μL, 0.32 mmol), and benzoquinone (0.17 g, 1.6 mmol) in 1,2-dichloroethane (15 mL) was added a solution of hydroperoxide (0.34 g, 1.6 mmol) in 1,2-dichloroethane (2 mL). The reaction mixture was heated (80 °C) for 3 h and then concentrated. The resulting residue was purified by flash chromatography (hexanes/EtOAc 98:2) to yield **2f** as a pale pink oil (0.11 g, 31% yield): ^1H NMR (500 MHz, $CDCl_3$) δ 6.32 (s, 1H), 5.81 (s, 1H), 4.90 (app d, *J* = 10.0 Hz, 1H), 4.24 (dq, *J* = 2.5, 7.1 Hz, 2H), 1.92 (m, 1H), 1.82-1.77 (m, 2H), 1.75-1.69 (m, 1H), 1.40 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.20 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.5, 139.5, 126.1, 78.5, 78.3, 61.2, 34.4, 27.3, 26.8, 22.9, 14.4; IR (thin film) 2977, 2937, 1722, 1631, 1450 cm^{-1}; HRMS (ESI) *m* / *z* calcd for $C_{11}H_{18}O_4Na$ $[M+Na]^+$ 237.1103, found 237.1097. Anal. Calcd for $C_{11}H_{18}O_4$: C, 61.66; H, 8.47. Found: C, 61.94; H, 8.65.



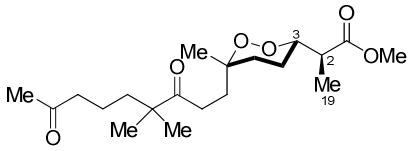
Ethyl (*E*)-3-(6-methoxy-6-methyl-1,2-dioxan-3-yl)-2-methylacrylate (2g). To a slurry of $Pd(OAc)_2$ (0.0072 g, 0.032 mmol), pyridine (11 μL, 0.13 mmol), and benzoquinone (0.070 g, 0.65 mmol) in 1,2-dichloroethane (5.5 mL) was added a solution of hydroperoxide (0.15 g, 0.65 mmol) in 1,2-dichloroethane (2 mL). The reaction mixture was heated (80 °C) for 3 h and then concentrated. The resulting residue was purified by flash chromatography (hexanes/acetone 98:2) to yield **2g** as a colorless oil (0.045 g, 30% yield) as a mixture of diastereomers in a 90:10 ratio where only the major diastereomer was fully characterized using NMR spectroscopy. Characterization for the major diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.36 (t, *J* = 1.1 Hz, 1H), 6.14 (t, *J* = 1.3 Hz, 1H), 4.93 (tt, *J* = 1.3, 6.5 Hz, 1H), 4.24 (m, 2H), 3.34 (s, 3H), 2.36 (m, 1H), 1.82 (app t, *J* = 5.9 Hz, 2H), 1.62 (m, 1H), 1.32 (t, *J* = 7.3 Hz, 3H), 1.30 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.1, 140.1, 125.7, 102.9, 77.6, 61.0, 49.5, 30.5, 23.7, 19.4, 14.4. Characterization for mixture of diastereomers: IR (thin film) 3025, 2931, 1714, 1666, 1496 cm^{-1}; (ESI) *m* / *z* calcd for $C_{11}H_{18}O_5Na$ $[M+Na]^+$ 253.1052, found 253.1044. Anal. Calcd for $C_{11}H_{18}O_5$: C, 57.38; H, 7.88. Found: C, 57.65; H, 8.04.



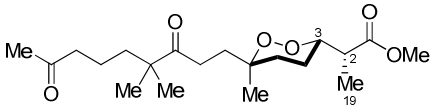
Ethyl-2-(6,6-dimethyl-1,2-dioxan-3-yl)propanoate (7f). To a solution of the endoperoxide (0.030 g, 0.14 mmol) in CH_2Cl_2 (3 mL) was added dipotassium azodicarboxylate[20] (0.82 g, 4.2 mmol) and the suspension was cooled to –78 °C. A solution of HOAc (0.084 g, 1.4 mmol) in CH_2Cl_2 (250 μL) was then added dropwise.[21] The mixture was stirred vigorously for 1.5 h at –78 °C, then warmed to ambient temperature. After 16 h, the reaction was confirmed to be complete by TLC. The solids were removed by filtration and the filtrate was concentrated *in vacuo*. The crude residue (60:40 mixture of diastereomers) was purified by flash chromatography (hexanes/EtOAc 98:2→95:5) to afford **7f** as separated diastereomers (major: 0.021 g, 70% yield; minor: 0.0050 g, 17% yield). Characterization of major diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.24 (m, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 2.56 (qu, *J* = 7.2 Hz, 1H), 1.69 (app d, *J* = 2.6 Hz, 4H), 1.34 (s, 3H), 1.27 (t, *J* = 7.1 Hz, 3H), 1.17 (s, 3H), 1.16 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.2, 81.9, 78.4, 60.9, 43.0, 34.1, 27.3, 23.2, 23.0, 14.4, 13.1; HRMS (ESI) *m* / *z* calcd for $C_{15}H_{20}O_4Na$ $[M+Na]^+$ 239.1259, found 239.1262. Characterization for minor diastereomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.16 (dq, *J* = 2.0, 7.2 Hz, 2H), 4.12 (m, 1H), 2.62 (m, 1H), 1.71 (m, 2H), 1.69 (m, 2H), 1.32 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.25 (d, *J* = 6.7 Hz, 3H), 1.20 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.0, 81.6, 78.4, 60.8, 43.2, 33.6, 26.8, 24.0, 23.4, 14.4, 13.9; HRMS (ESI) *m* / *z* calcd for $C_{15}H_{20}O_4Na$ $[M+Na]^+$ 239.1259, found 239.1266. Characterization for mixture of both isomers: IR (thin film) 2979, 2940, 1737 cm^{-1}. Anal. Calcd for $C_{11}H_{20}O_4$: C, 61.09; H, 9.32. Found: C, 60.83; H, 9.34.

Assignment of Relative Stereochemistry for Major and Minor Diastereomers of 7f. Shown below are the ^{1}H and ^{13}C NMR spectroscopic assignments for dicarperoxides B and C which

have been reported.[22] Given their structural similarity to **7f**, their assignment has been used to identify the major relative stereochemistry of the hydrogenation of **2f** to give **7f**. Both the ^{1}H and ^{13}C shifts have distinct correlations between the known compounds and the observed isomers of **7f**. The relative configurations of the dicarperoxides B and C were determined based on empirical rules developed by Capon and Macleod.[23]



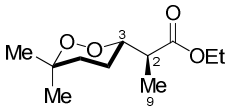
dicarperoxide B



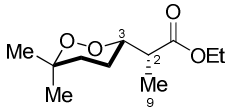
dicarperoxide C

	δ ^{1}H	δ ^{13}C
C-2	2.49	42.6
C-3	4.24	81.3
C-19	1.11	12.8

	δ ^{1}H	δ ^{13}C
C-2	2.68	42.7
C-3	4.11	81.2
C-19	1.25	13.6



major diastereomer **7f**



minor diastereomer **7f**

	δ ^{1}H	δ ^{13}C
C-2	2.56	43.0
C-3	4.24	81.9
C-9	1.16	13.1

	δ ^{1}H	δ ^{13}C
C-2	2.62	43.2
C-3	4.12	81.6
C-9	1.25	13.9

***N*-(2-(7-Chloroquinolin-4-ylamino)ethyl)-3-(6,6-dimethyl-1,2-dioxan-3-yl)-2-methyl-propanamide (8f).** To a solution of the major diastereomer of 1,2-dioxane **7f** (0.030 g, 0.14 mmol) in MeOH (0.6 mL) was added LiOH (1.0 M in H_2O, 0.60 mL, 0.60 mmol). The solution was stirred for 16 h at ambient temperature. The reaction mixture was concentrated *in vacuo* to remove the MeOH. The solution was diluted with Et_2O (5 mL) and 1 M HCl was added until the pH of the aqueous layer reached 2. The aqueous layer was extracted with Et_2O (2 × 5 mL), and the organic layers were combined, dried over $MgSO_4$, and concentrated *in vacuo* to afford the crude carboxylic acid as a colorless crystalline solid.

To a solution of the colorless solid dissolved in CH_2Cl_2 (4.9 mL) was added *N*-(7-chloroquinolin-4-yl)ethane-1,2-diamine[24] (0.031 g, 0.14 mmol), *N,N*-diisopropylethylamine (34 µL, 0.21 mmol), hydroxybenzotriazole (0.020 g, 0.15 mmol), and *N*-(3-dimethylaminopropyl)-*N*'-ethylcarbodiimide hydrochloride[25] (0.029 g, 0.15 mmol). The slurry was stirred at ambient temperature for 16 h. Water (2 mL) was added to the mixture and the solution was extracted with CH_2Cl_2 (2 × 5 mL). The organic layers were combined, dried over $MgSO_4$, and concentrated *in vacuo*. The residue was purified by flash chromatography on basic Al_2O_3 (EtOAc/MeOH 98:2) to afford **8f** as a white solid (0.036 g, 67% yield): Melting point range 174-176 °C; ^{1}H NMR (500 MHz, CD_3OD) δ 8.36 (d, J = 5.6 Hz, 1H), 8.02 (d, J = 9.0 Hz, 1H), 7.76 (d, J = 2.1 Hz, 1H), 7.38 (dd, 2.1, 9.0 Hz, 1H), 6.60 (d, J = 5.6 Hz, 1H), 5.49 (s, 1H), 4.09 (m, 1H), 3.60 (m, 1H), 3.48 (m, 2H), 2.37 (m, 1H), 1.75 (m, 1H), 1.70 (m, 1H), 1.60 (m, 2H), 1.26

(s, 3H), 1.11 (s, 3H), 1.06 (d, J = 7.0 Hz, 3H); ^{13}C NMR (126 MHz, CD_3OD) δ 177.9, 152.8, 152.2, 149.3, 136.5, 127.3, 126.2, 124.4, 118.6, 99.7, 83.3, 79.2, 45.1, 43.9, 39.1, 34.6, 27.1, 24.6, 23.2, 13.7; IR (thin film) 3336, 2973, 2468, 1637, 1579 cm^{-1}; HRMS (ESI) m / z calcd for $C_{20}H_{26}O_3N_3ClH$ $[M+H]^+$ 392.1741, found 392.1731. Anal. Calcd for $C_{20}H_{26}O_3N_3Cl$: C, 61.30; H, 6.69; N, 10.72. Found: C, 61.08; H, 6.89; N, 10.67.

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IV. Calculations for Determination of Yields by NMR

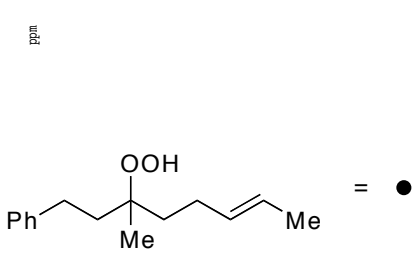
Initial Spectrum: Internal Standard: 9.000 / 9 protons = 1.000
Starting Hydroperoxide: 1.589 / 2 protons = 0.7945

Spectrum at t = 3 h: Internal Standard: 9.000 / 9 protons = 1.000
Starting Hydroperoxide: 0.0740 / 1 protons = 0.0740
Reduced Alcohol*: 0.3163 / 2 protons = 0.1581
Desired 1,2-Dioxane: 0.3205 / 1 proton = 0.3205
*Value obtained from overlapping signal: Reduced Alcohol = Total Signal – Starting Hydroperoxide = 0.3903 – 0.0740 = 0.3163

Calculated Yields:
Desired 1,2-Dioxane: 0.3205 / 0.7945 = 40.3%
Reduced Alcohol: 0.1581 / 0.7945 = 19.8%
Starting Hydroperoxide: 0.0740 / 0.7945 = 9.3%

V. Spectra for Determination of Yields by NMR

Initial Spectrum for t=0 h of starting hydroperoxide and internal standard in tol-d8



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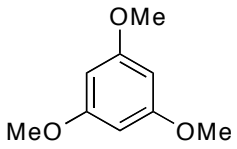

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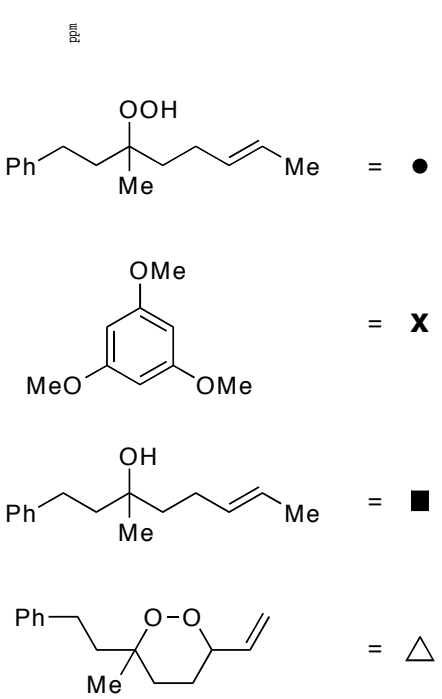
Spectrum of reaction mixture at t = 3h in tol-d8

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RG                  128
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HZCM          175.49562 Hz/cm
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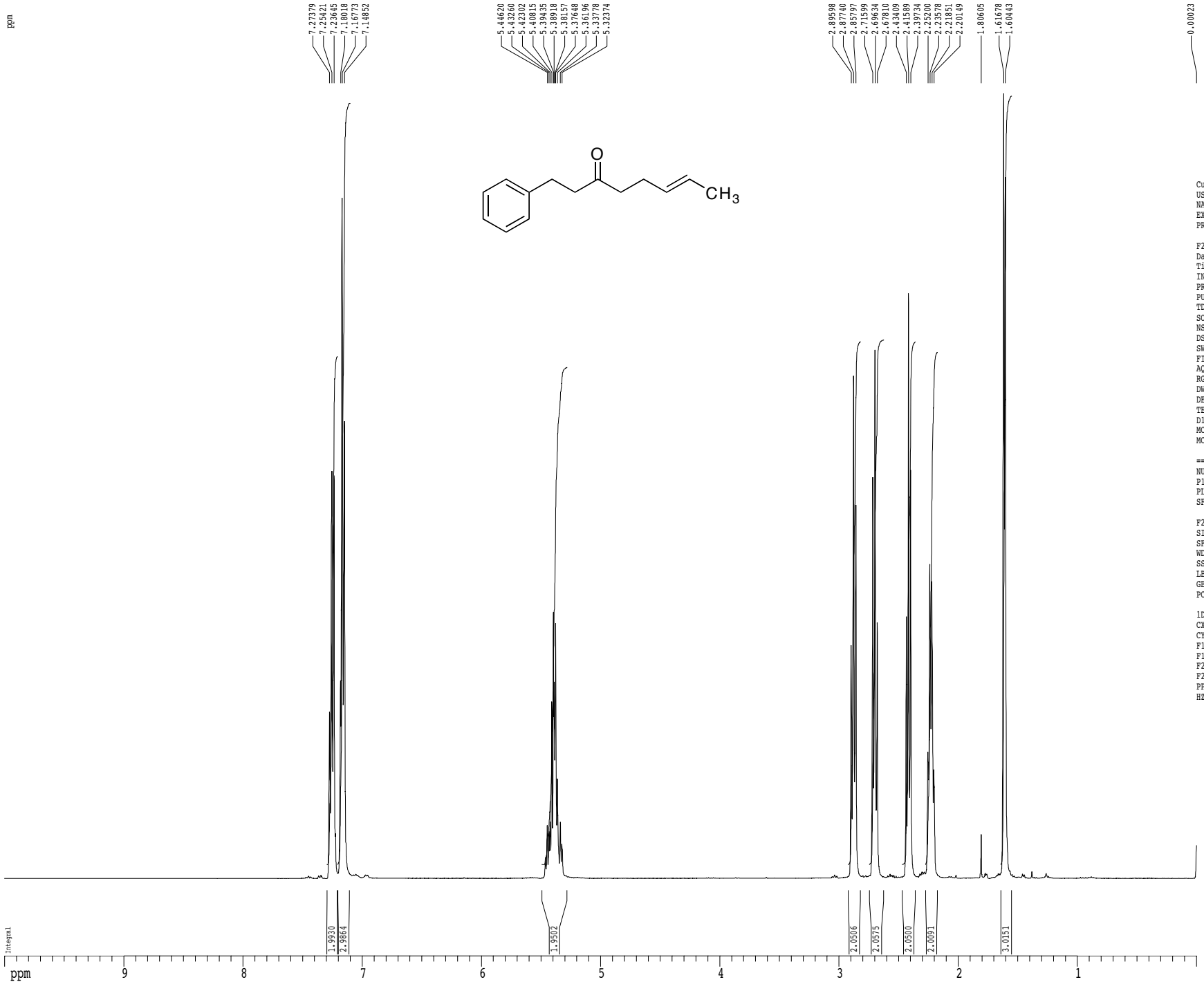
VI. Selected Spectra

1H spectrum

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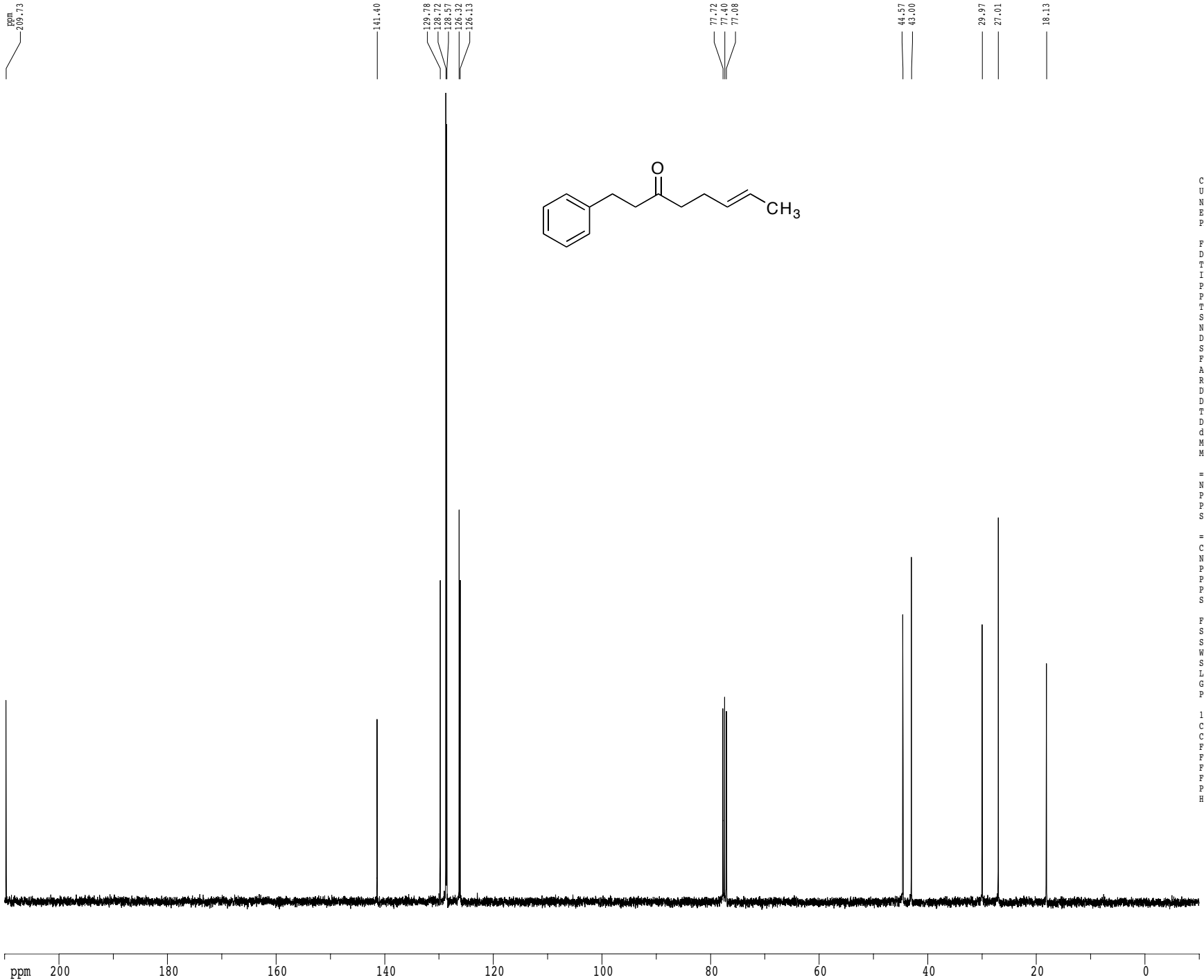
SF           400.1300358 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P               10.000 ppm
F1              4001.30 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM           0.43860 ppm/cm
HZCM          175.49564 Hz/cm
```

13C spectrum with 1H decoupling



Current Data Parameters
USER harris
NAME 4165-c1-cf3
EXPNO 2
PROCNO 1

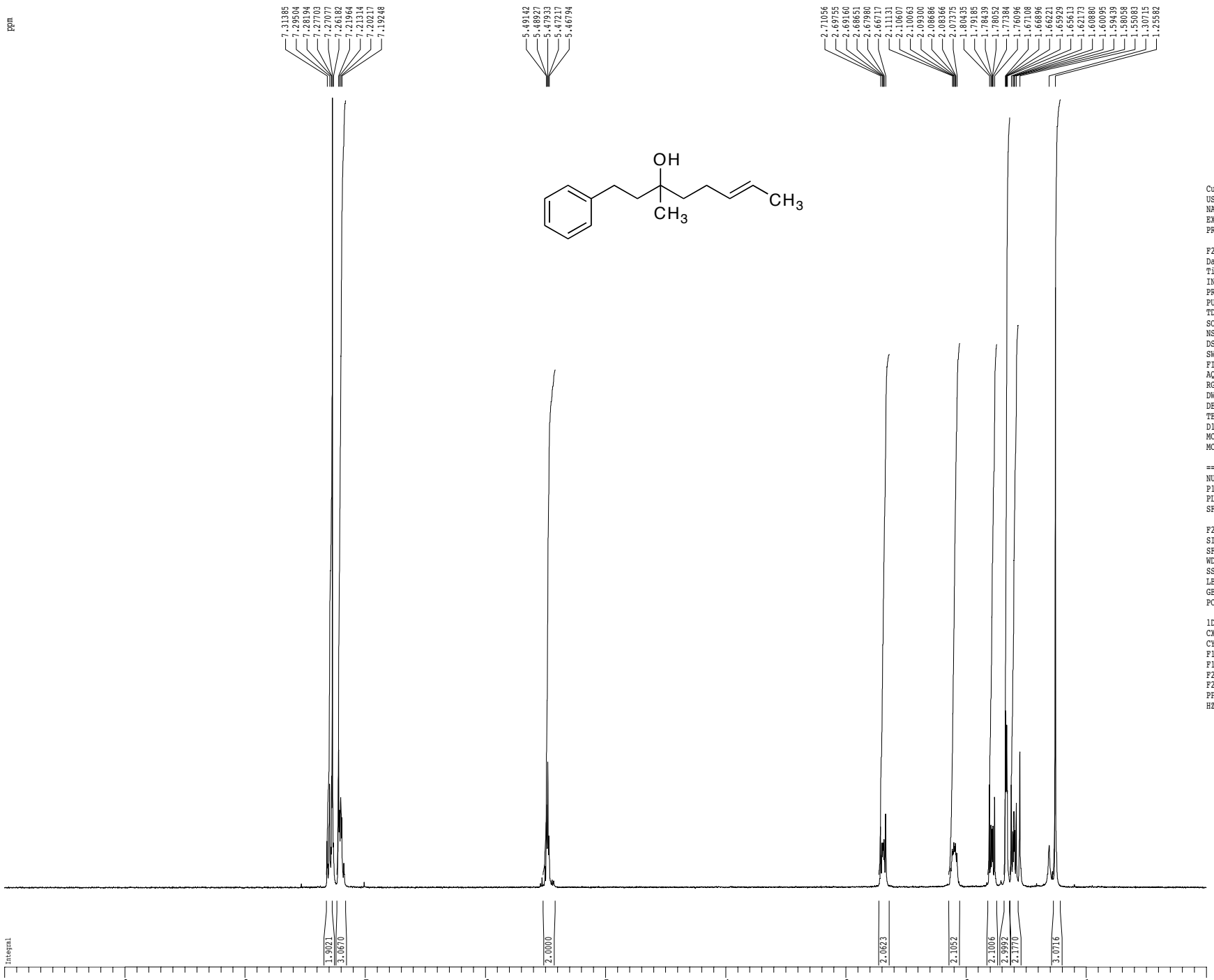
F2 - Acquisition Parameters
Date_ 20071112
Time 18.15
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 257
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 13004
DW 20.700 usec
DE 20.39 usec
TE 298.1 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 0.00 dB
SFO1 100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 210.000 ppm
F1 21128.68 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.64912 ppm/cm
HZCM 970.82477 Hz/cm



Current Data Parameters
USER shelli
NAME srw1166
EXPNO 9
PROCNO 1

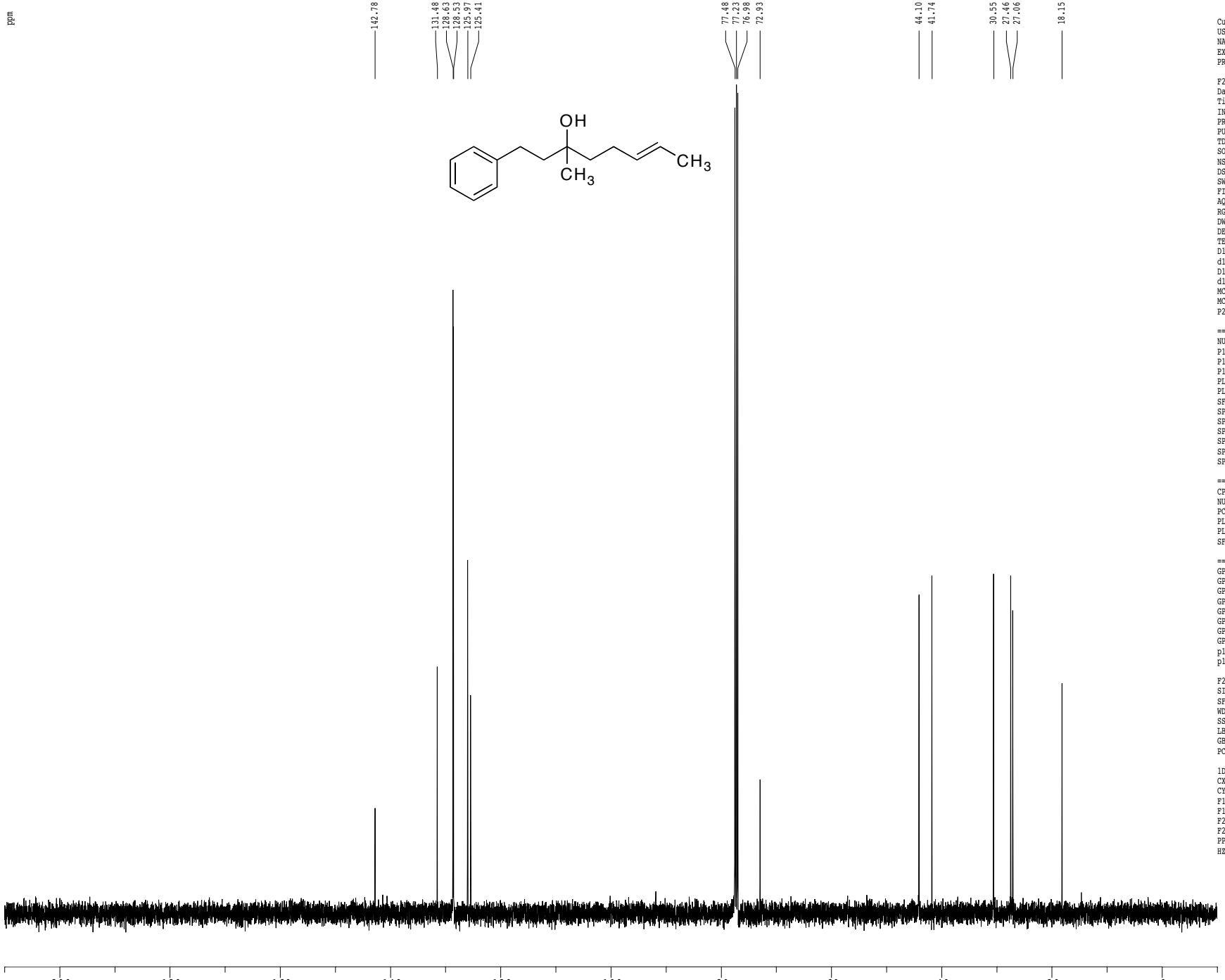
F2 - Acquisition Parameters
Date_ 20090402
Time 11.10
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 4401.409 Hz
FIDRES 0.067160 Hz
AQ 7.4449396 sec
RG 812.7
DW 113.600 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 175.49562 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw1166
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090401
Time 9.07
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 149
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

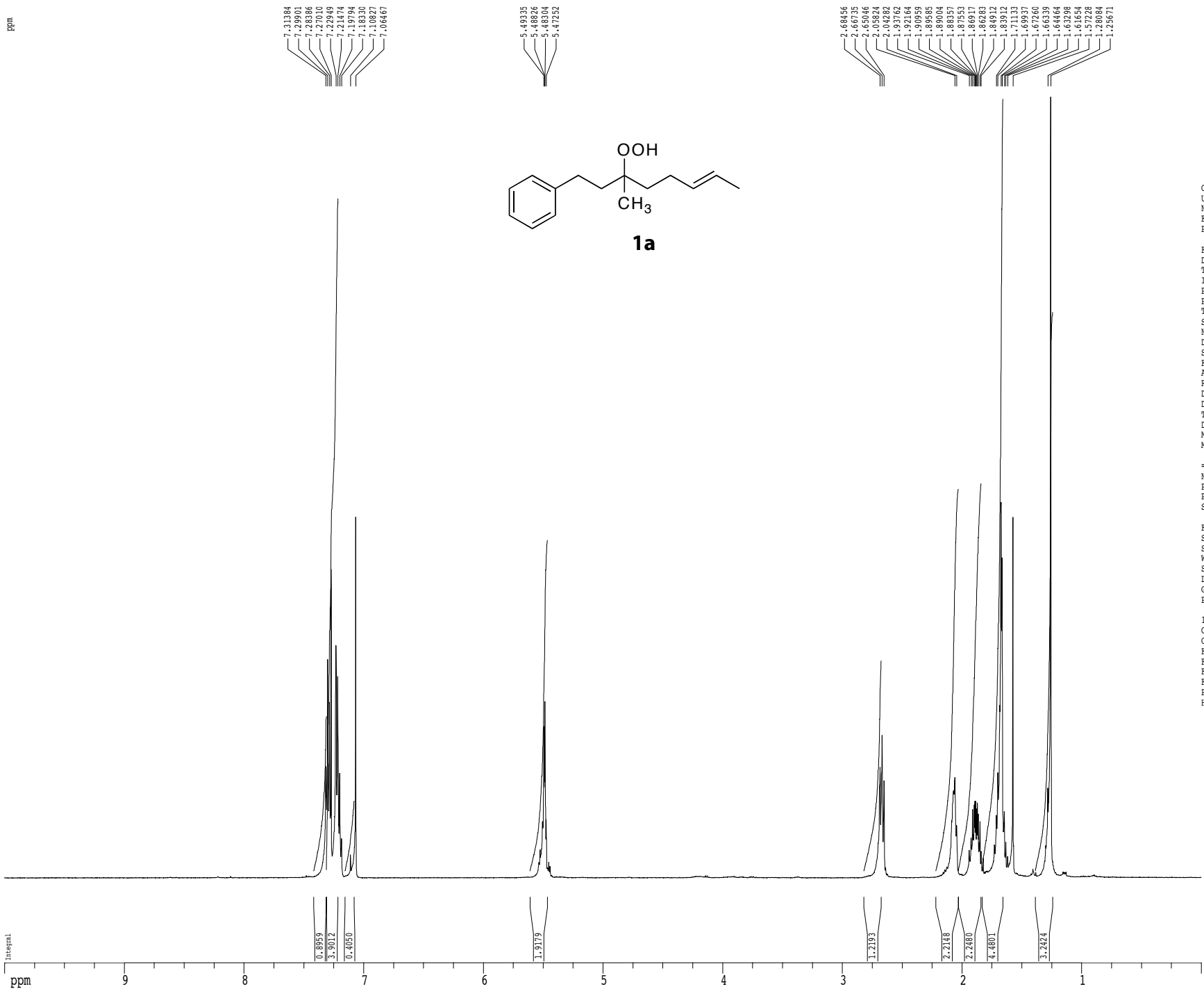
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7804002 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67065 Hz/cm

1H spectrum



Current Data Parameters
USER shelli
NAME srw1169
EXPNO 2
PROCNO 1

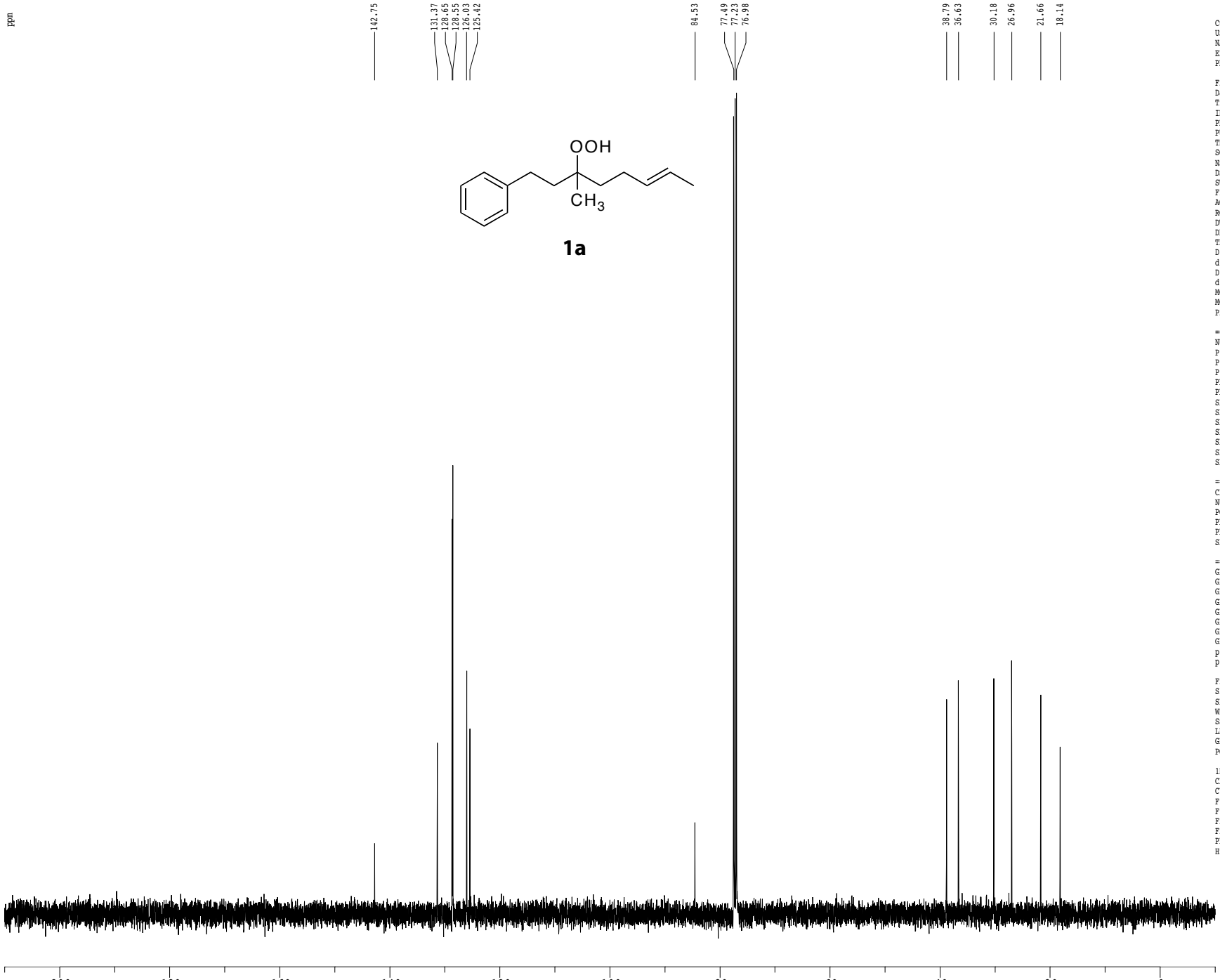
F2 - Acquisition Parameters
Date_ 20081028
Time 13.31
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200262 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw1169
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081028
Time 13.34
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 215
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

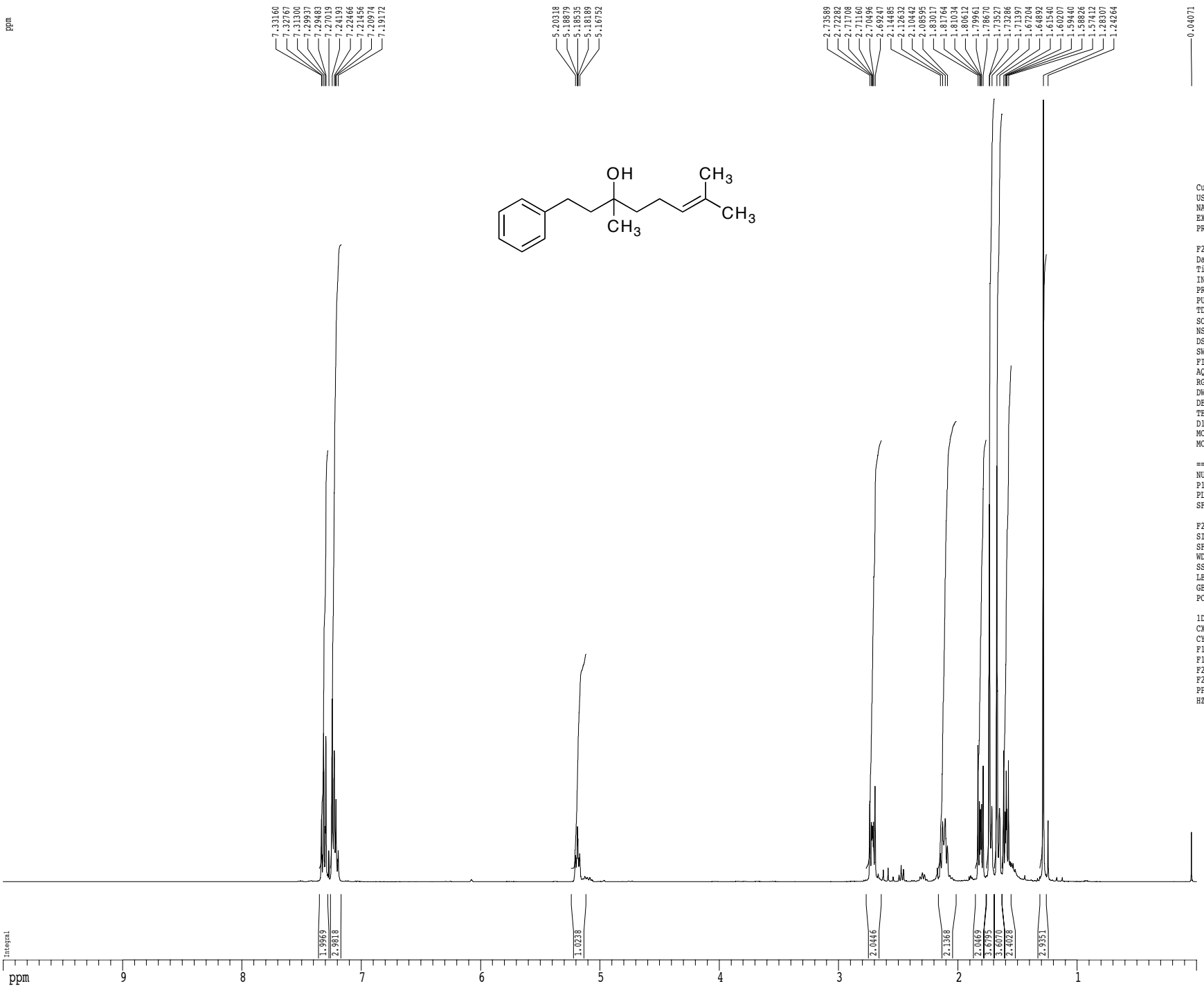
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803988 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67053 Hz/cm

1H spectrum



Current Data Parameters
USER harris
NAME 4103-d1-f4
EXPNO 1
PROCNO 1

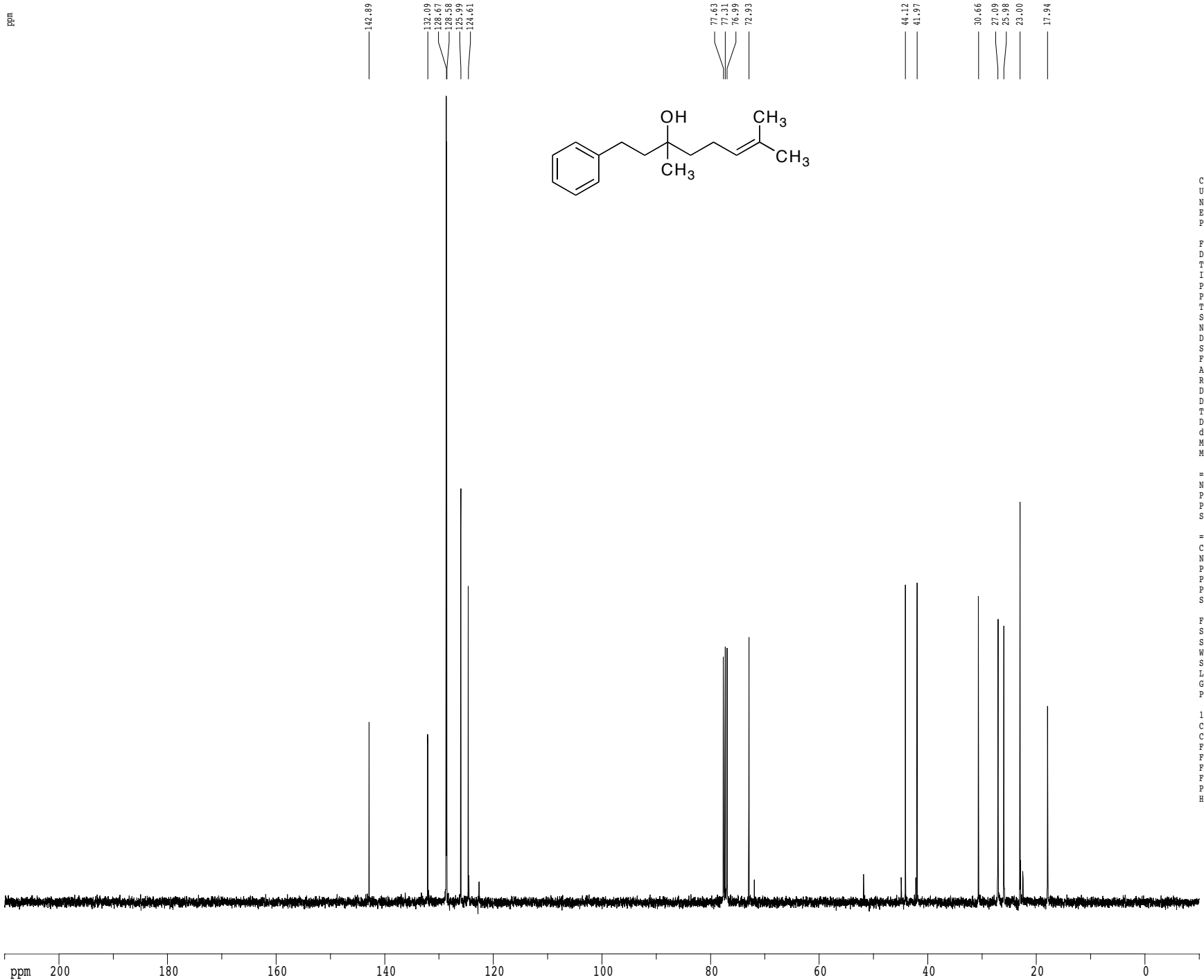
F2 - Acquisition Parameters
Date_ 20070807
Time 13.53
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 32
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 175.49562 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER harris
NAME 4124-c1-cf4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070910
Time 15.24
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 208
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 4597.6
DW 20.700 usec
DE 20.39 usec
TE 297.9 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

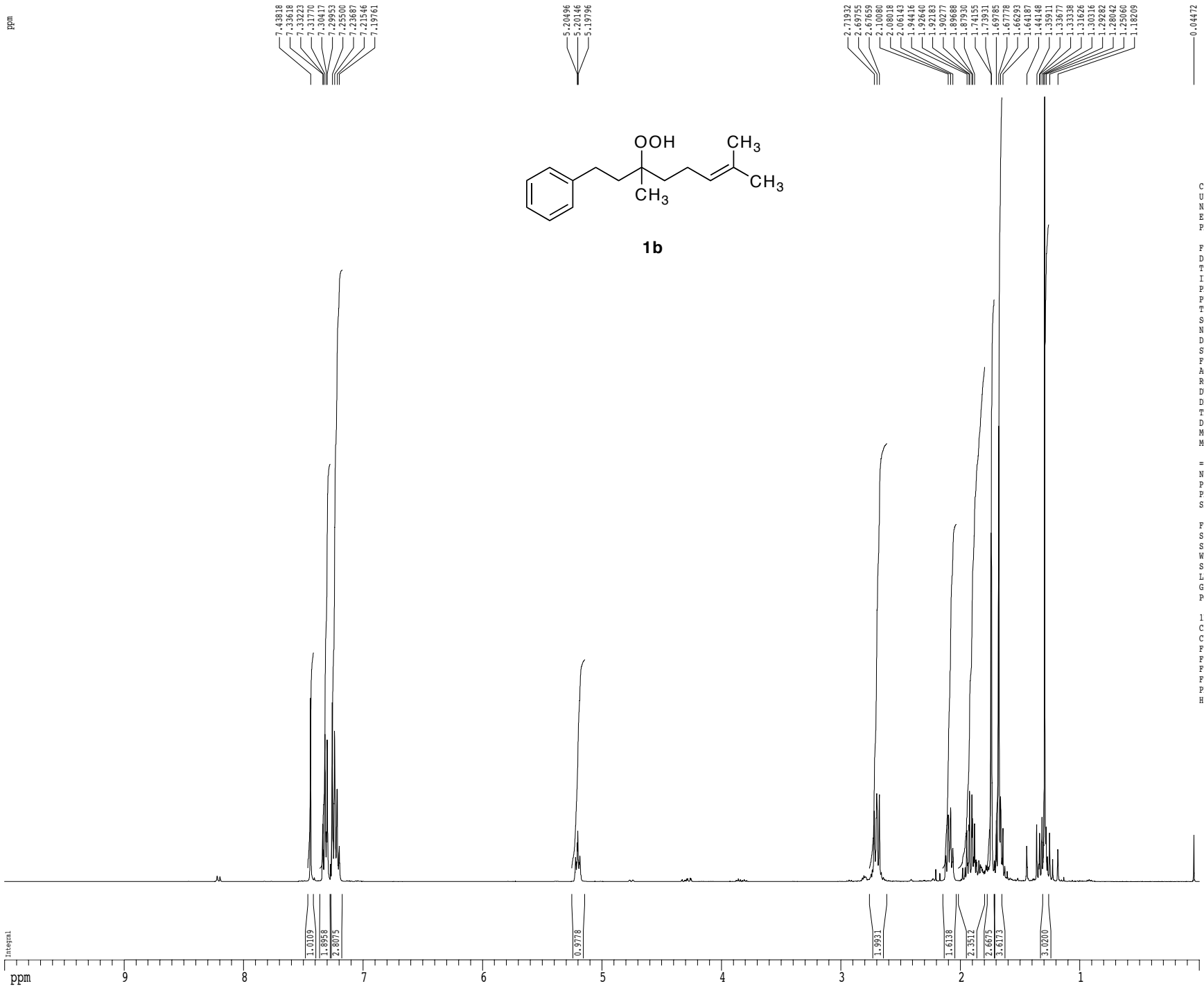
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 0.00 dB
SFO1 100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 210.000 ppm
F1 21128.68 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.64912 ppm/cm
HZCM 970.82477 Hz/cm

1H spectrum



Current Data Parameters
USER harris
NAME 4105-c2-cf1
EXPNO 1
PROCNO 1

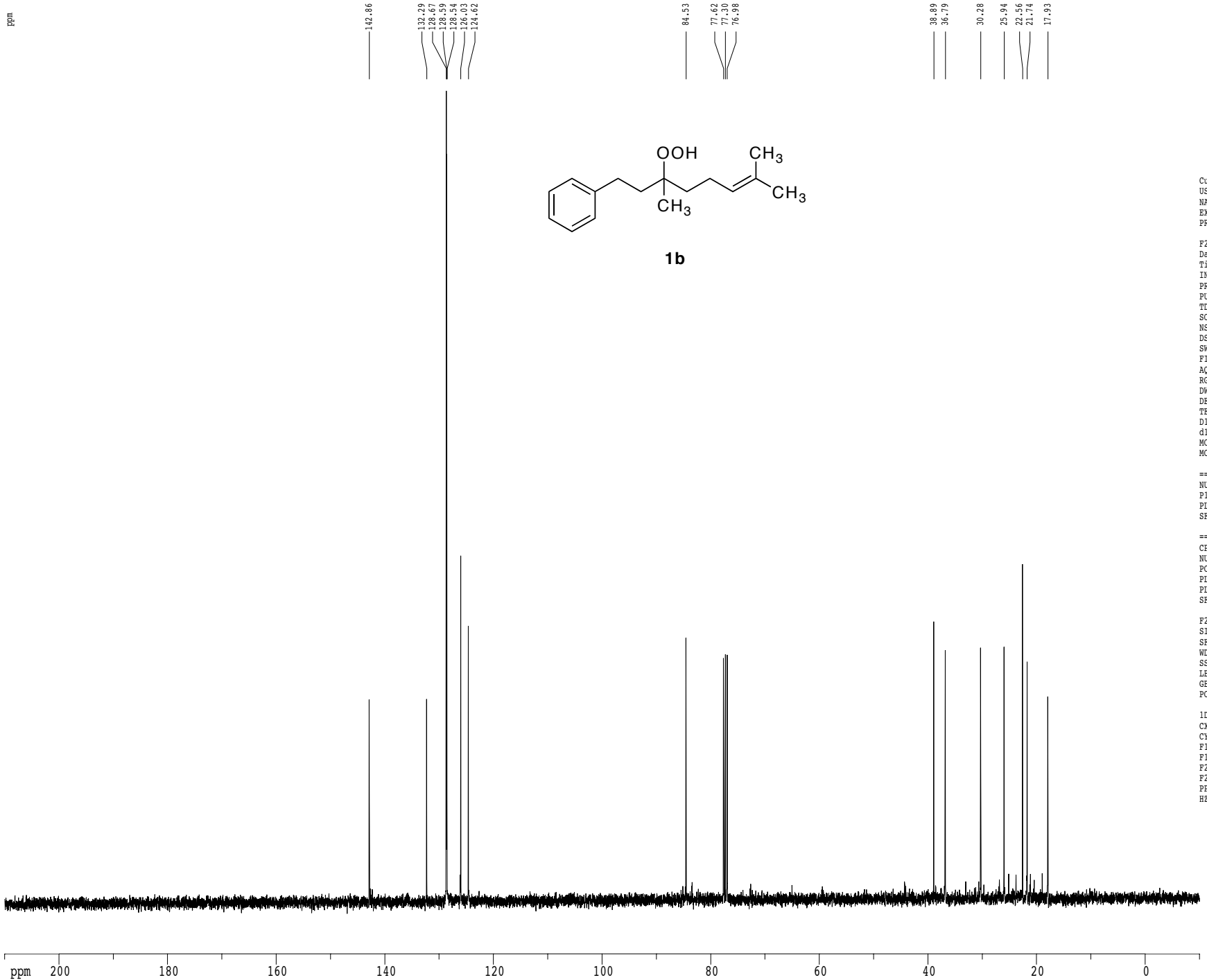
F2 - Acquisition Parameters
Date_ 20070808
Time 17.44
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 32
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

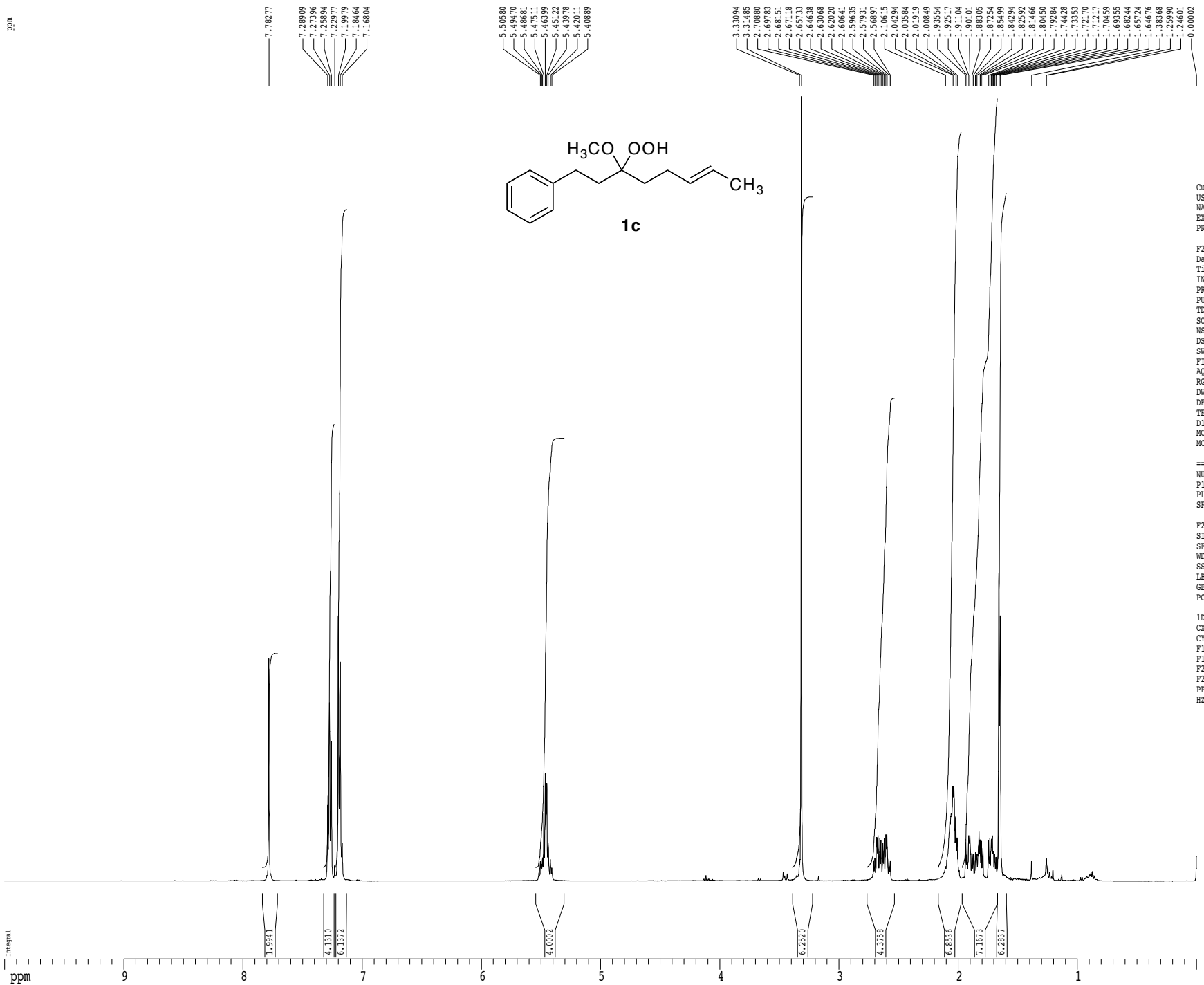
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 175.49562 Hz/cm

13C spectrum with 1H decoupling



1H spectrum



Current Data Parameters

USER	harris
NAME	4206-c1-cf2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080107
Time	11.32
INSTRUM	gn500
PROBHD	5 mm broadband
PULPROG	zg30
TD	81728
SOLVENT	CDCl3T
NS	8
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	32
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	12.00 usec
PL1	-3.00 dB
SFO1	499.8284988 MHz

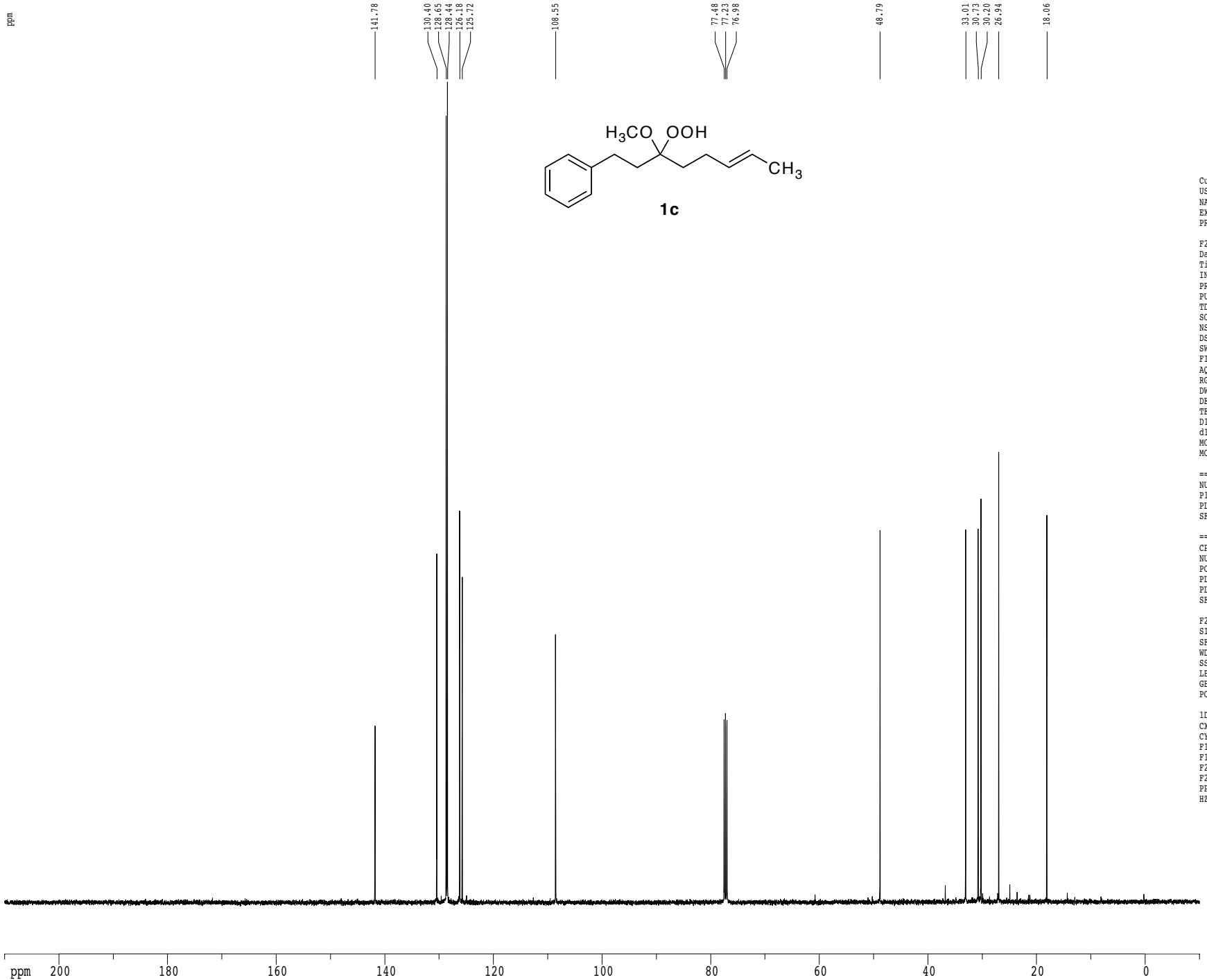
F2 - Processing parameters

SI	65536
SF	499.8250454 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	10.000 ppm
F1	4998.25 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.43860 ppm/cm
HZCM	219.22151 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER harris
NAME 4206-c1-cf2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080107
Time 11.38
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 365
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 3251
DW 16.500 usec
DE 4.50 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

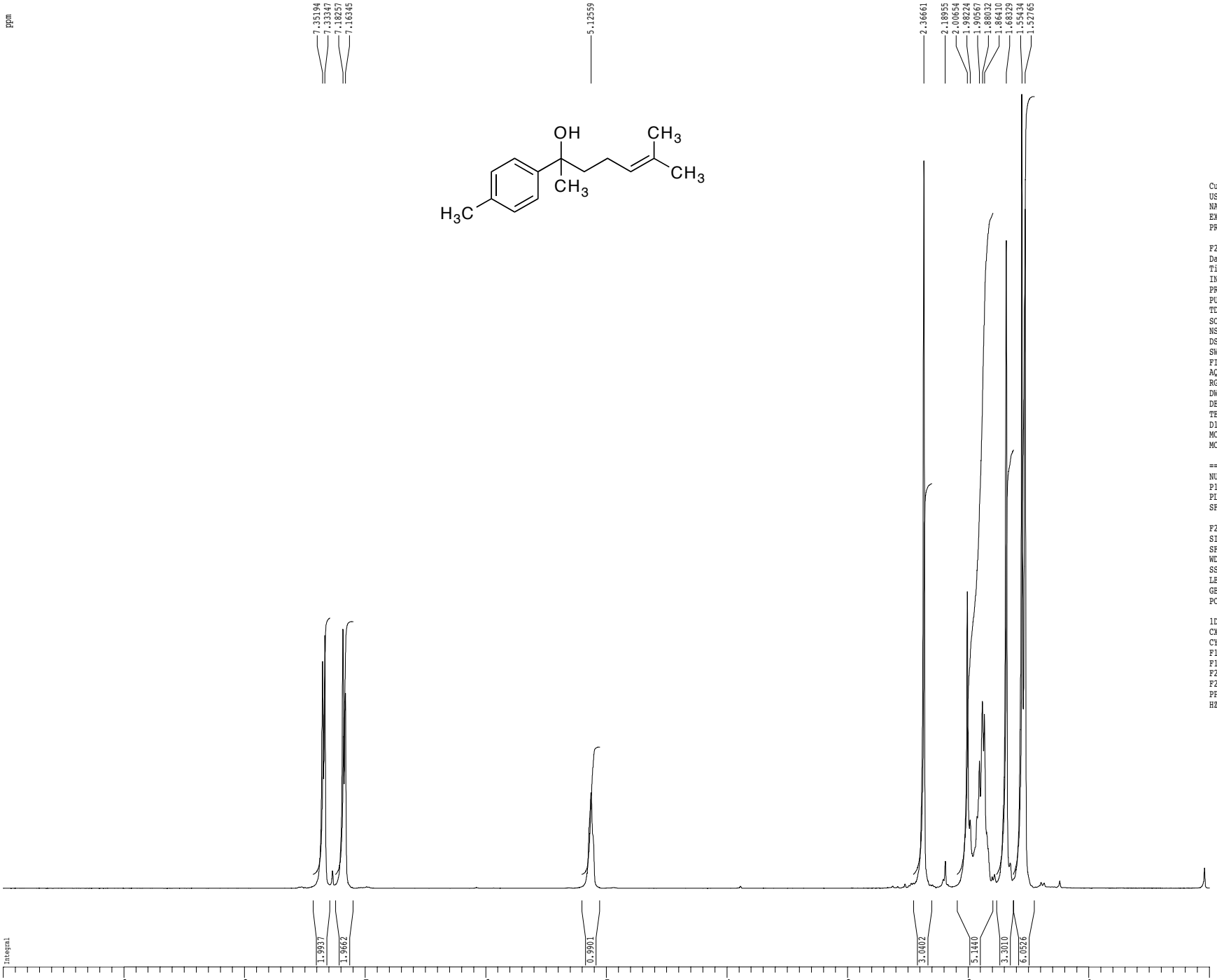
======== CHANNEL f1 ========
NUC1 13C
P1 7.08 usec
PL1 0.00 dB
SFO1 125.6949209 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 14.70 dB
SFO2 499.8274991 MHz

F2 - Processing parameters
SI 65536
SF 125.6810822 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26393.03 Hz
F2P -10.000 ppm
F2 -1256.81 Hz
PPMCM 9.64912 ppm/cm
HZCM 1212.71228 Hz/cm

1H spectrum



Current Data Parameters
USER harris
NAME 5079-d1-f2
EXPNO 1
PROCNO 1

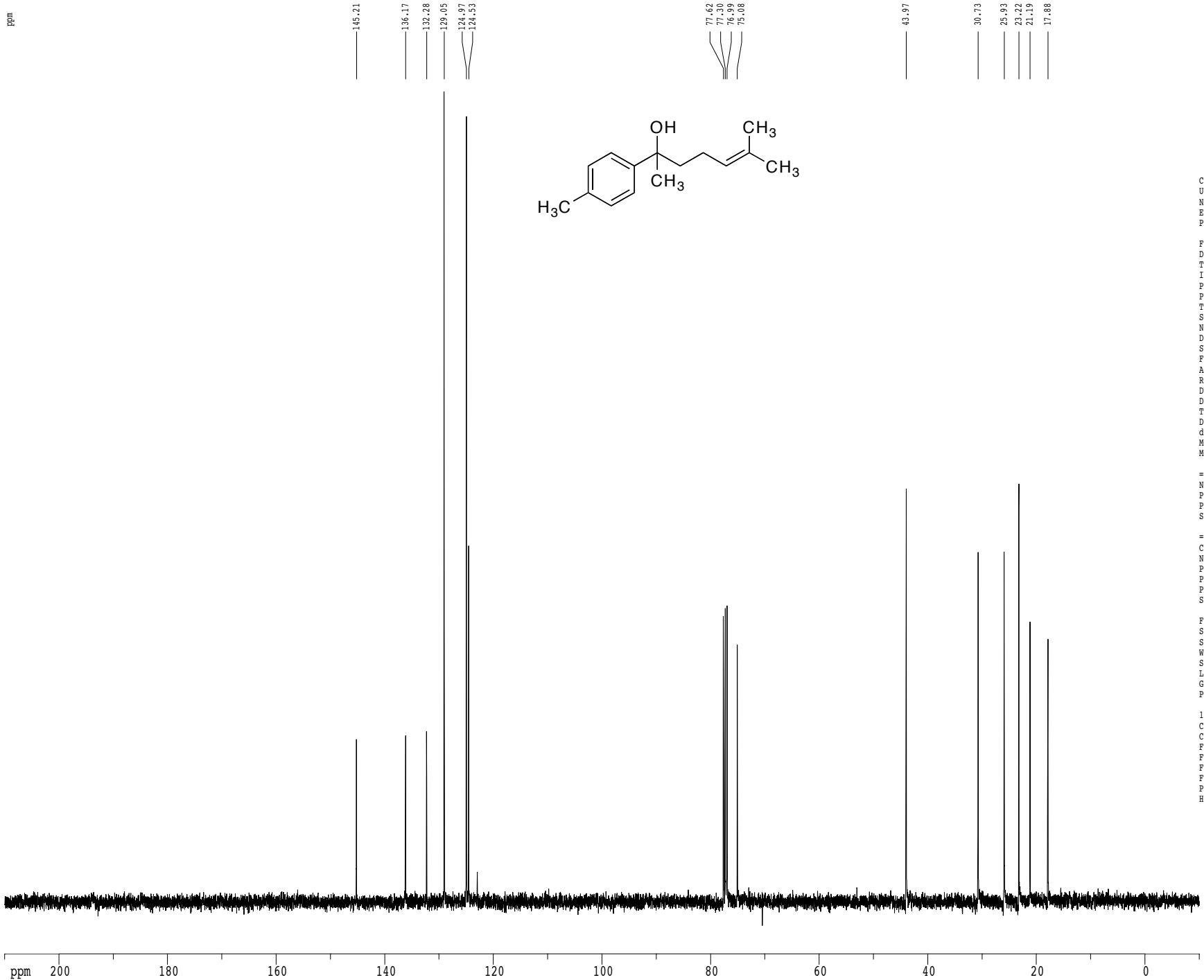
F2 - Acquisition Parameters
Date_ 20080307
Time 15.33
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 32
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 400.1300182 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 175.49562 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER harris
NAME 5079-d1-f2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080307
Time 15.35
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 141
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

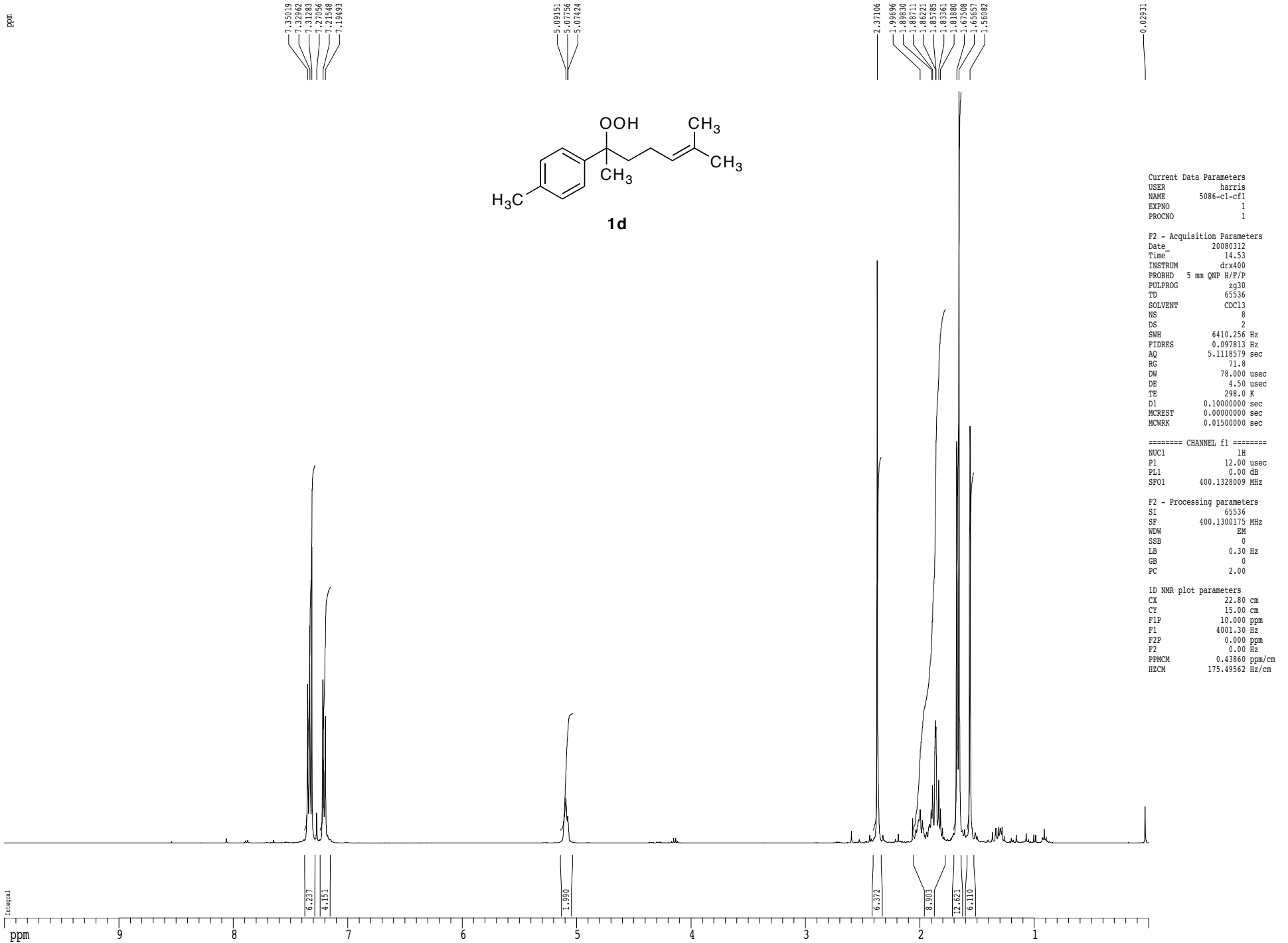
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 0.00 dB
SFO1 100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 210.000 ppm
F1 21128.68 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.64912 ppm/cm
HZCM 970.82477 Hz/cm

1H spectrum



Current Data Parameters

USER	harris
NAME	5086-c1-cf1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080312
Time	14.53
INSTRUM	drx400
PROBHD	5 mm QNP H/F/P
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	6410.256 Hz
FIDRES	0.097813 Hz
AQ	5.1118579 sec
RG	71.8
DW	78.000 usec
DE	4.50 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	12.00 usec
PL1	0.00 dB
SFO1	400.1328009 MHz

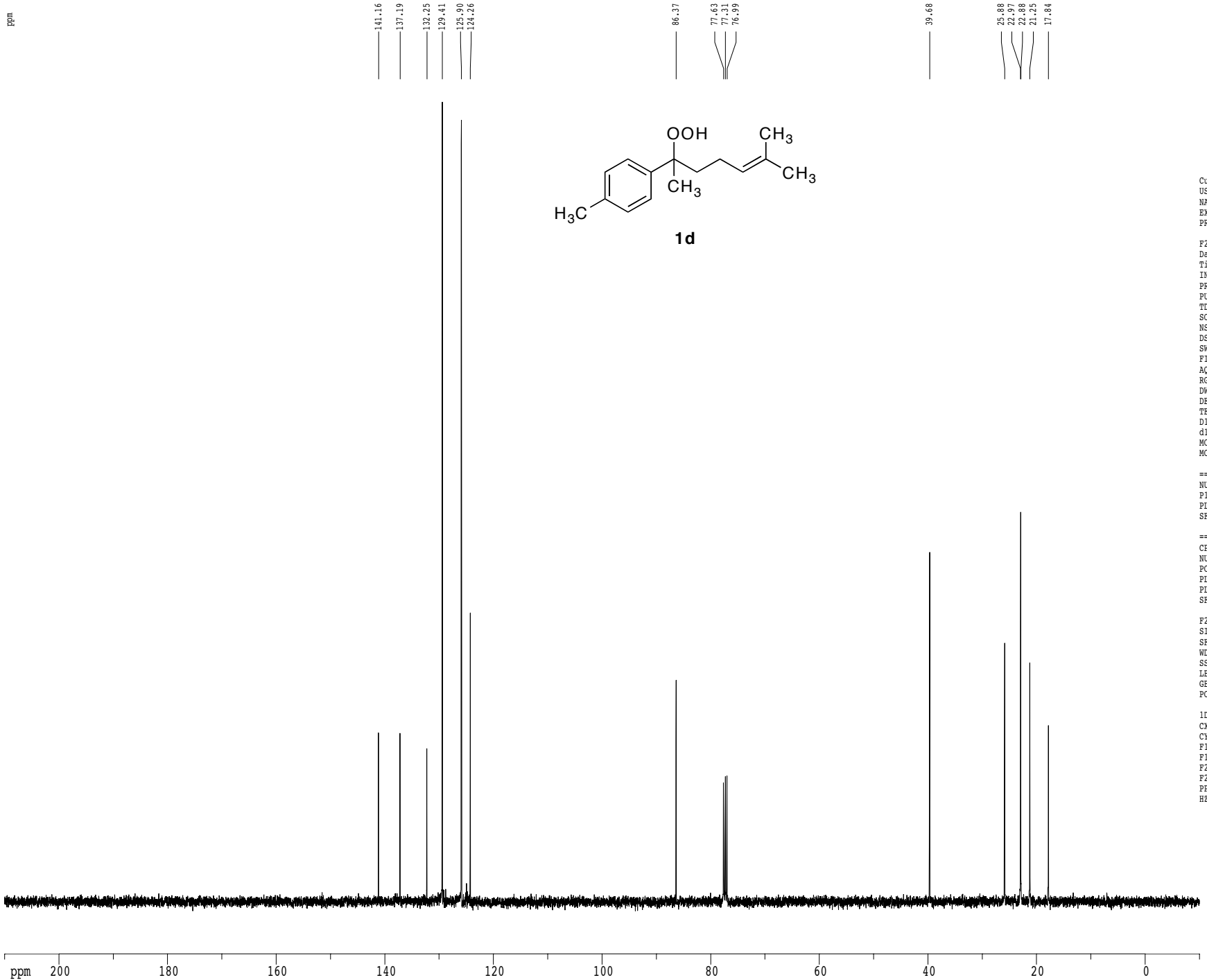
F2 - Processing parameters

SI	65536
SF	400.1300175 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.43860 ppm/cm
HZCM	175.49562 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER harris
NAME 5132-c1-cf1
EXPNO 2
PROCNO 1

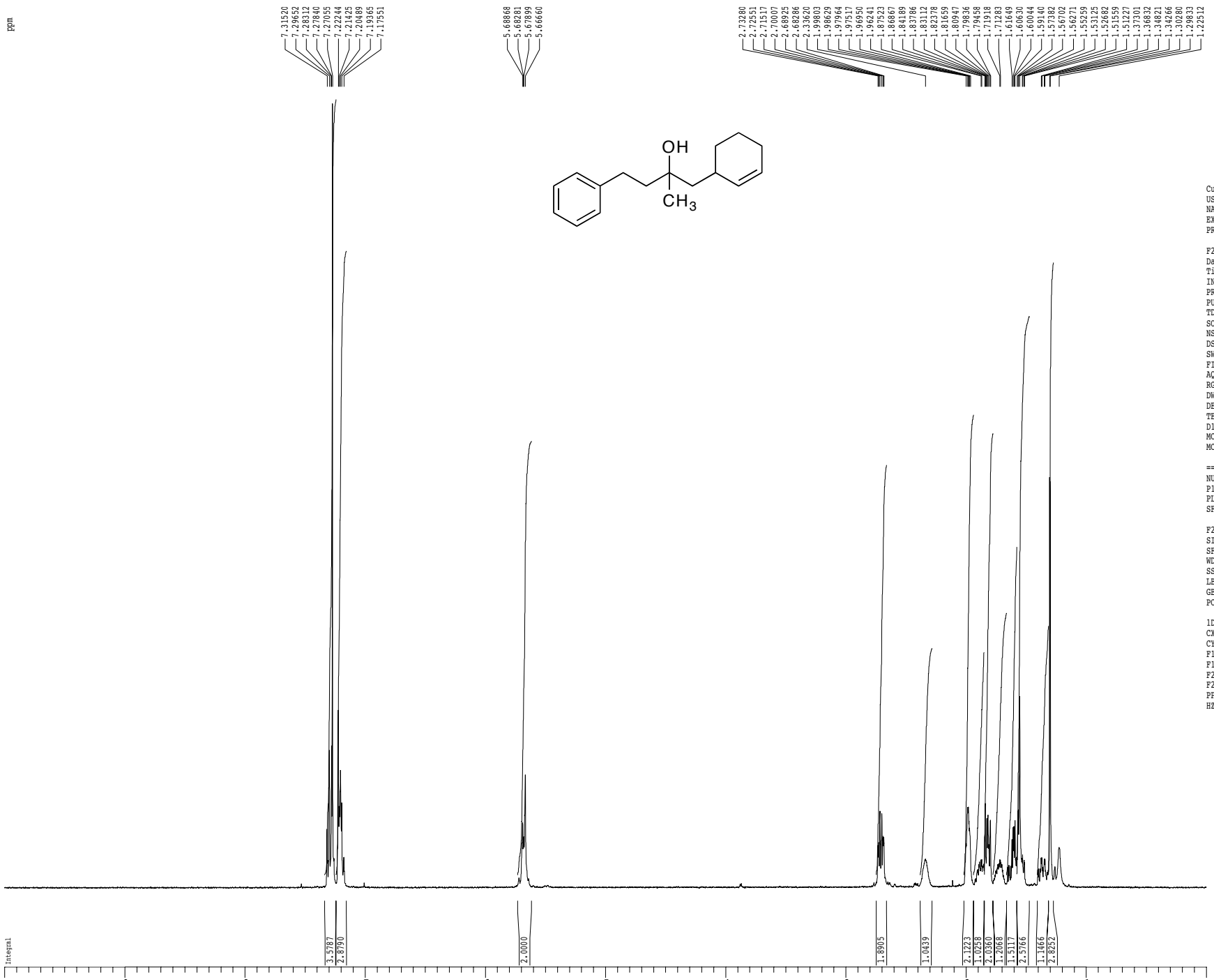
F2 - Acquisition Parameters
Date_ 20080501
Time 16.48
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 77
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 14596.5
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 0.00 dB
SFO1 100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 210.000 ppm
F1 21128.68 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.64912 ppm/cm
HZCM 970.82477 Hz/cm



Current Data Parameters
USER shelli
NAME srw2116
EXPNO 9
PROCNO 1

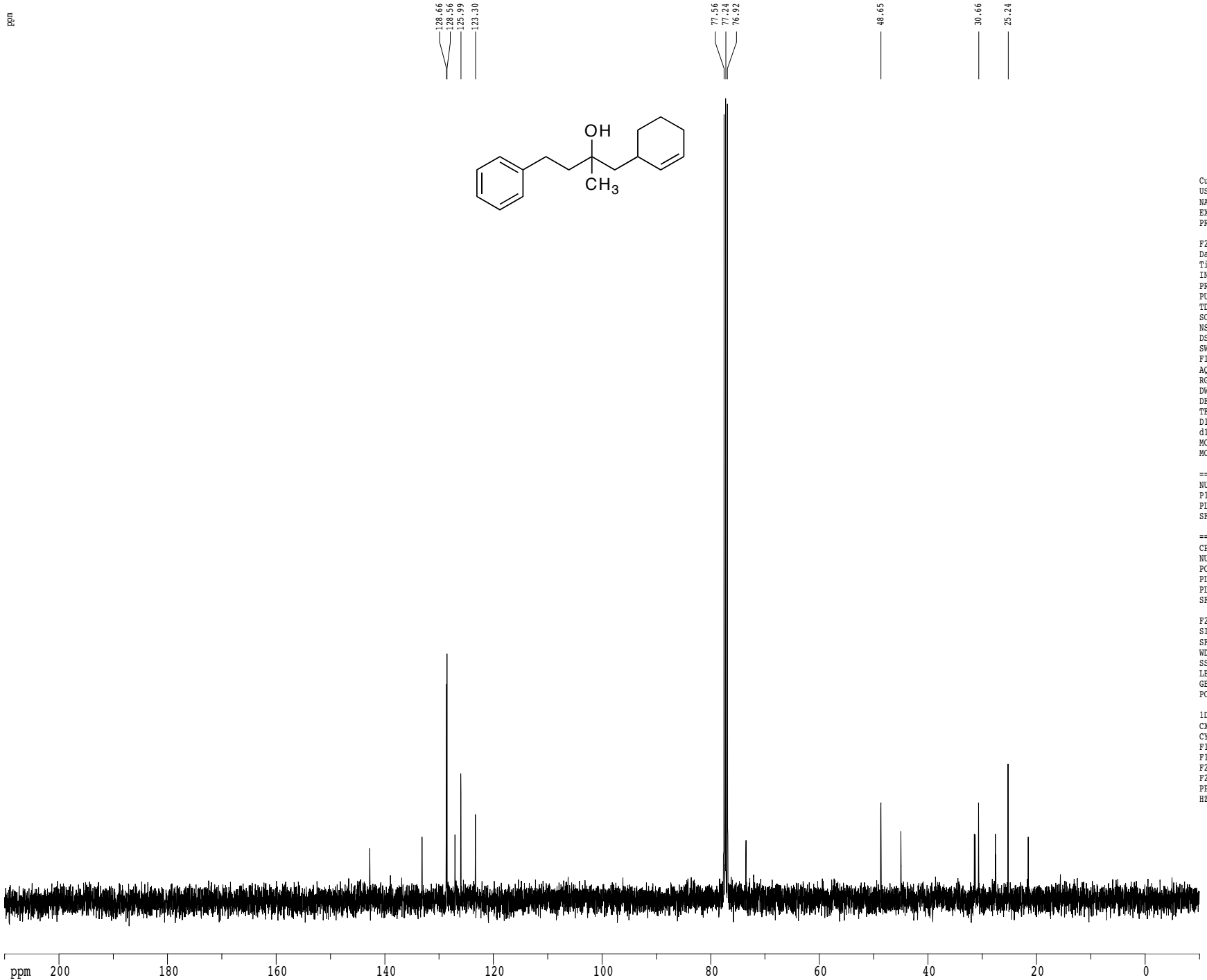
F2 - Acquisition Parameters
Date_ 20090319
Time 9.31
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 4401.409 Hz
FIDRES 0.067160 Hz
AQ 7.4449396 sec
RG 724.1
DW 113.600 usec
DE 4.50 usec
TE 298.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 175.49562 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw2116
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090318
Time 13.22
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 278
DS 4
SWH 24154.590 Hz
FIDRES 0.368570 Hz
AQ 1.3566452 sec
RG 9195.2
DW 20.700 usec
DE 20.39 usec
TE 298.0 K
D1 0.10000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

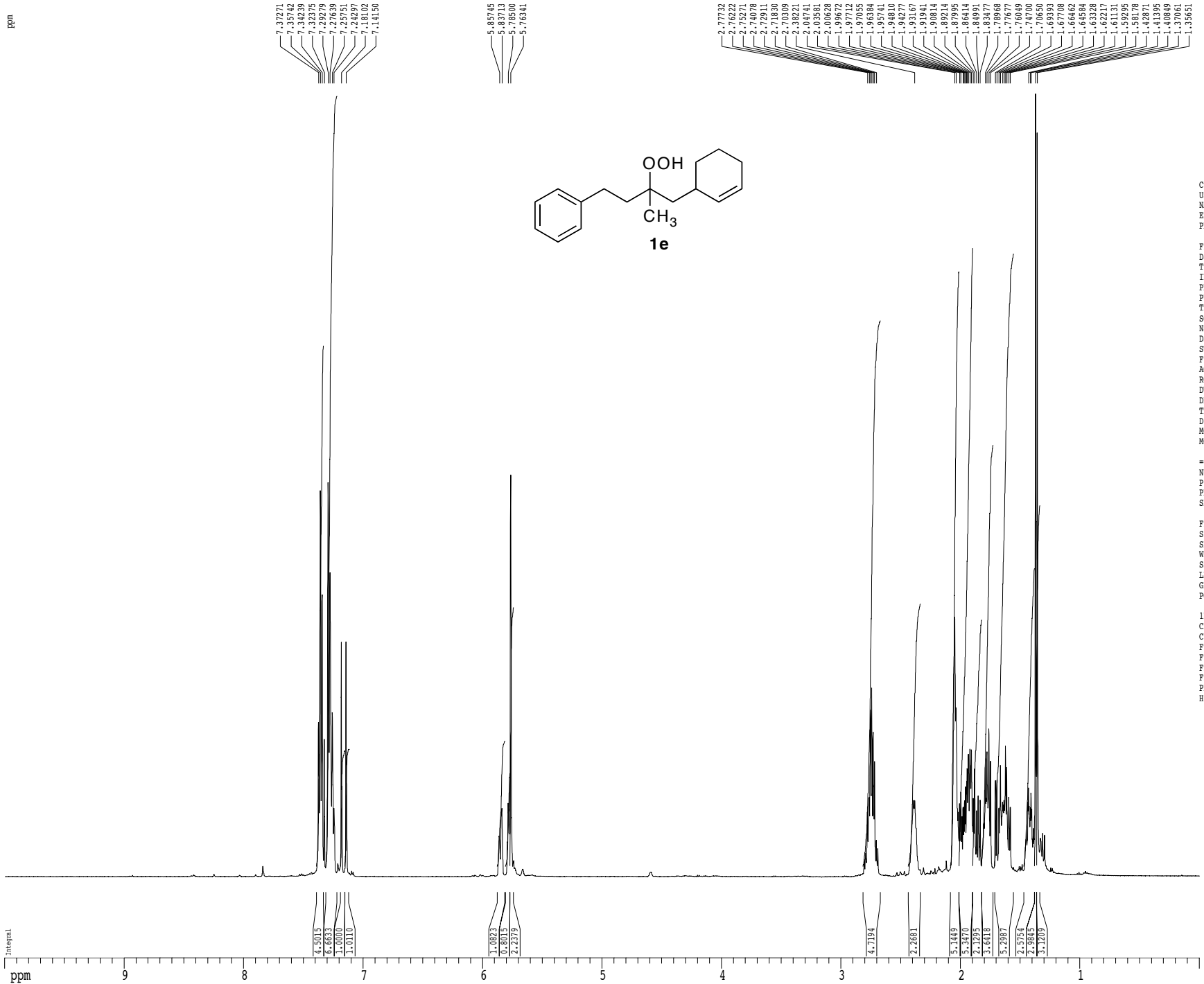
======== CHANNEL f1 ========
NUC1 13C
P1 10.75 usec
PL1 0.00 dB
SFO1 100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2 mlev16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 17.70 dB
SFO2 400.1328009 MHz

F2 - Processing parameters
SI 65536
SF 100.6127500 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.50 cm
F1P 210.000 ppm
F1 21128.68 Hz
F2P -10.000 ppm
F2 -1006.13 Hz
PPMCM 9.64912 ppm/cm
HZCM 970.82477 Hz/cm

1H spectrum



Current Data Parameters

USER	shelli
NAME	srw2127
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20090402
Time	17.32
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	8
DS	2
SWH	5482.456 Hz
FIDRES	0.067082 Hz
AQ	7.4536438 sec
RG	3.6
DW	91.200 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2225011 MHz

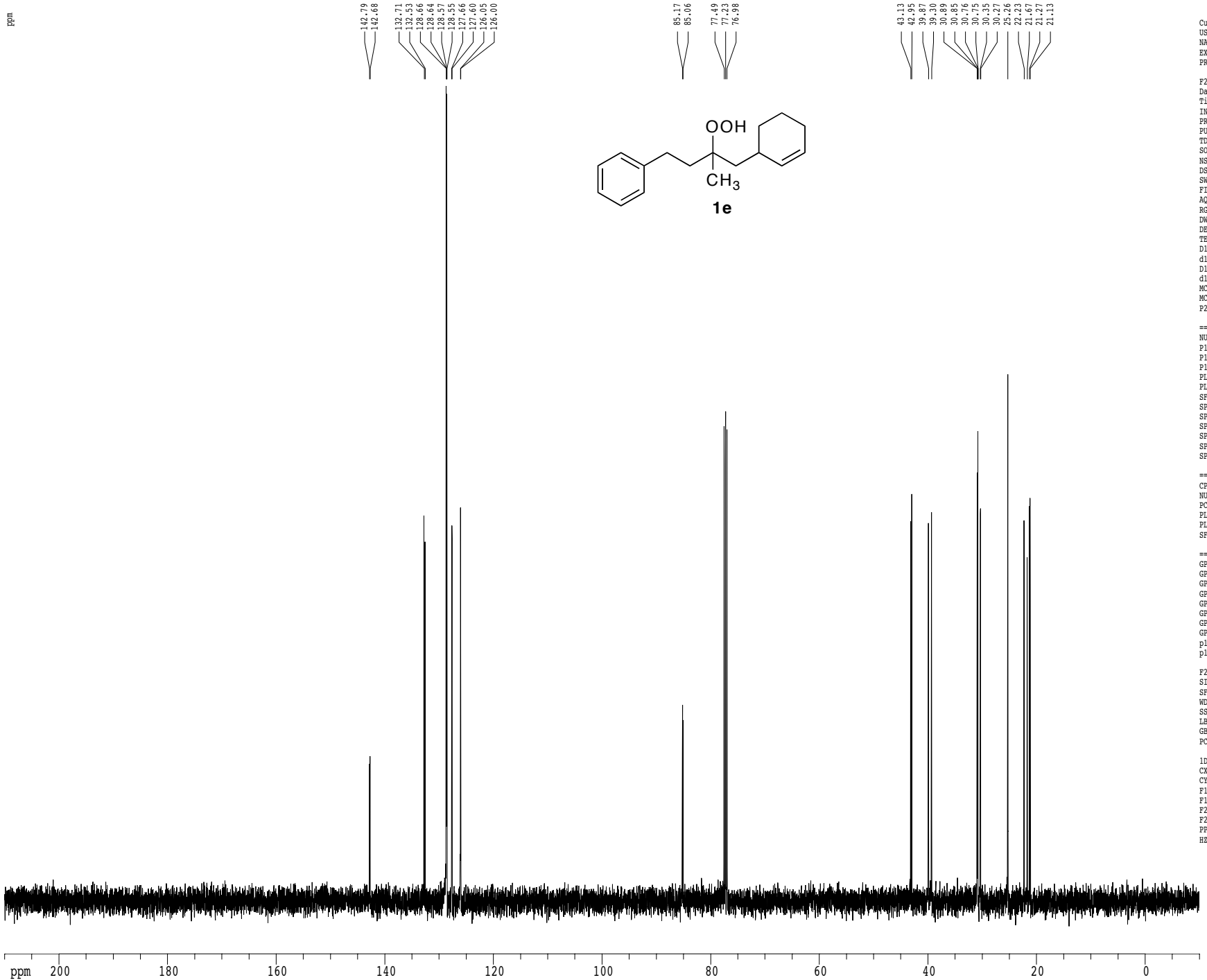
F2 - Processing parameters

SI	65536
SF	500.2200000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	10.000 ppm
F1	5002.20 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.43860 ppm/cm
HZCM	219.39474 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw2127
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090402
Time 17.34
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 81
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

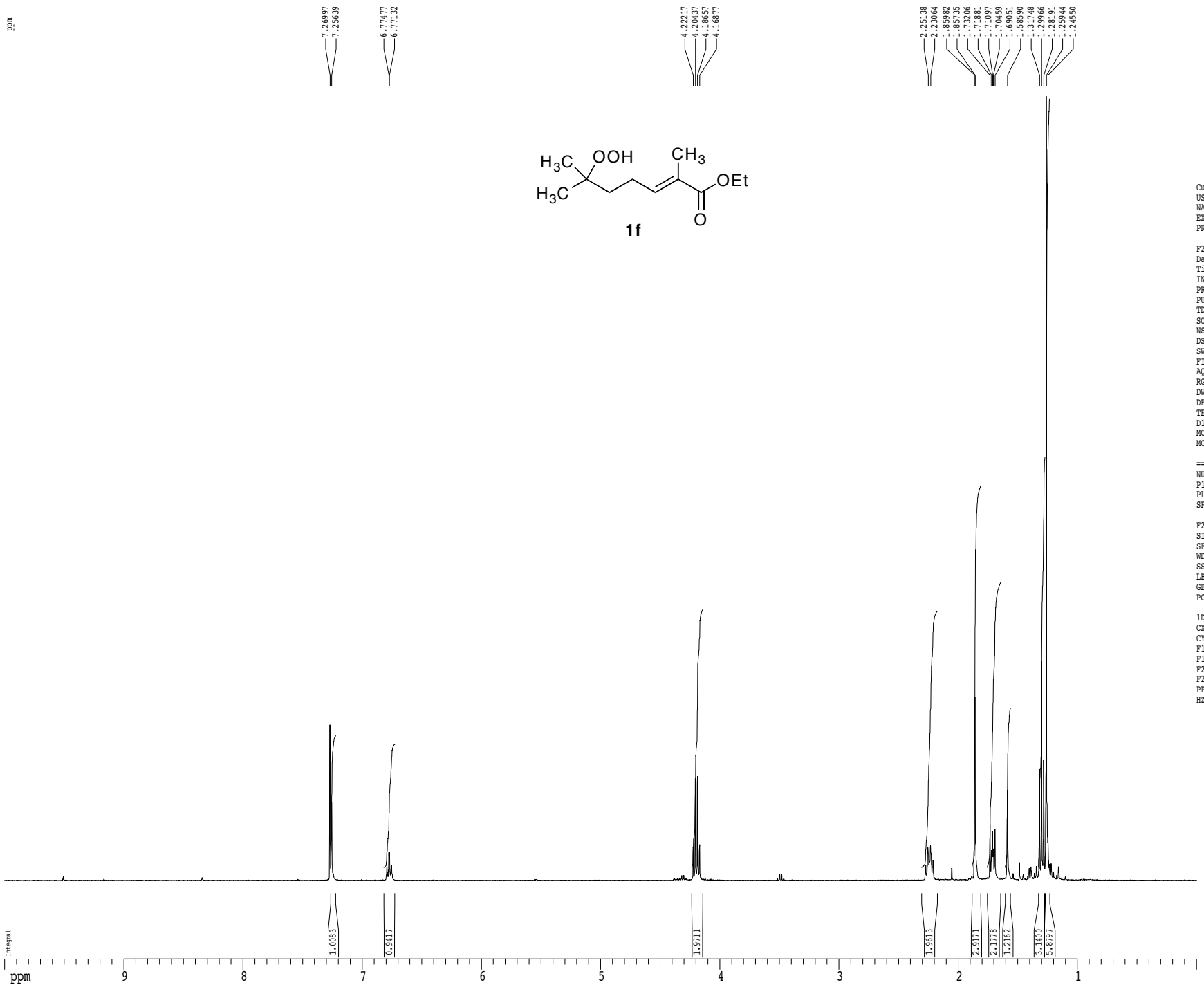
======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803998 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67065 Hz/cm



Current Data Parameters
USER shelli
NAME srw2098
EXPNO 1
PROCNO 1

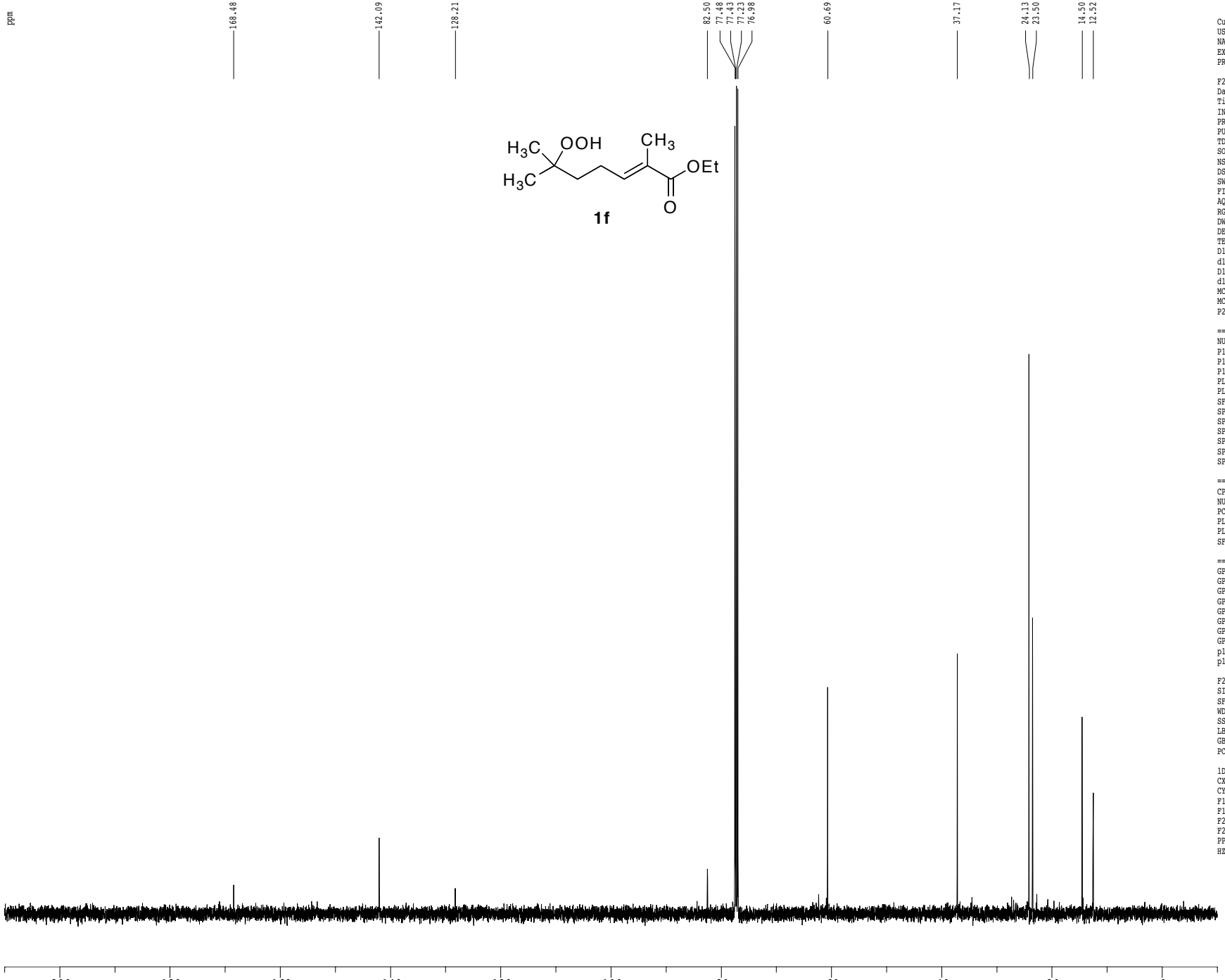
F2 - Acquisition Parameters
Date_ 20090306
Time 12.47
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 4401.409 Hz
FIDRES 0.067160 Hz
AQ 7.4449396 sec
RG 574.7
DW 113.600 usec
DE 4.50 usec
TE 298.5 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 65536
SF 400.1300178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 175.49562 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw1190
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081113
Time 14.31
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 425
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

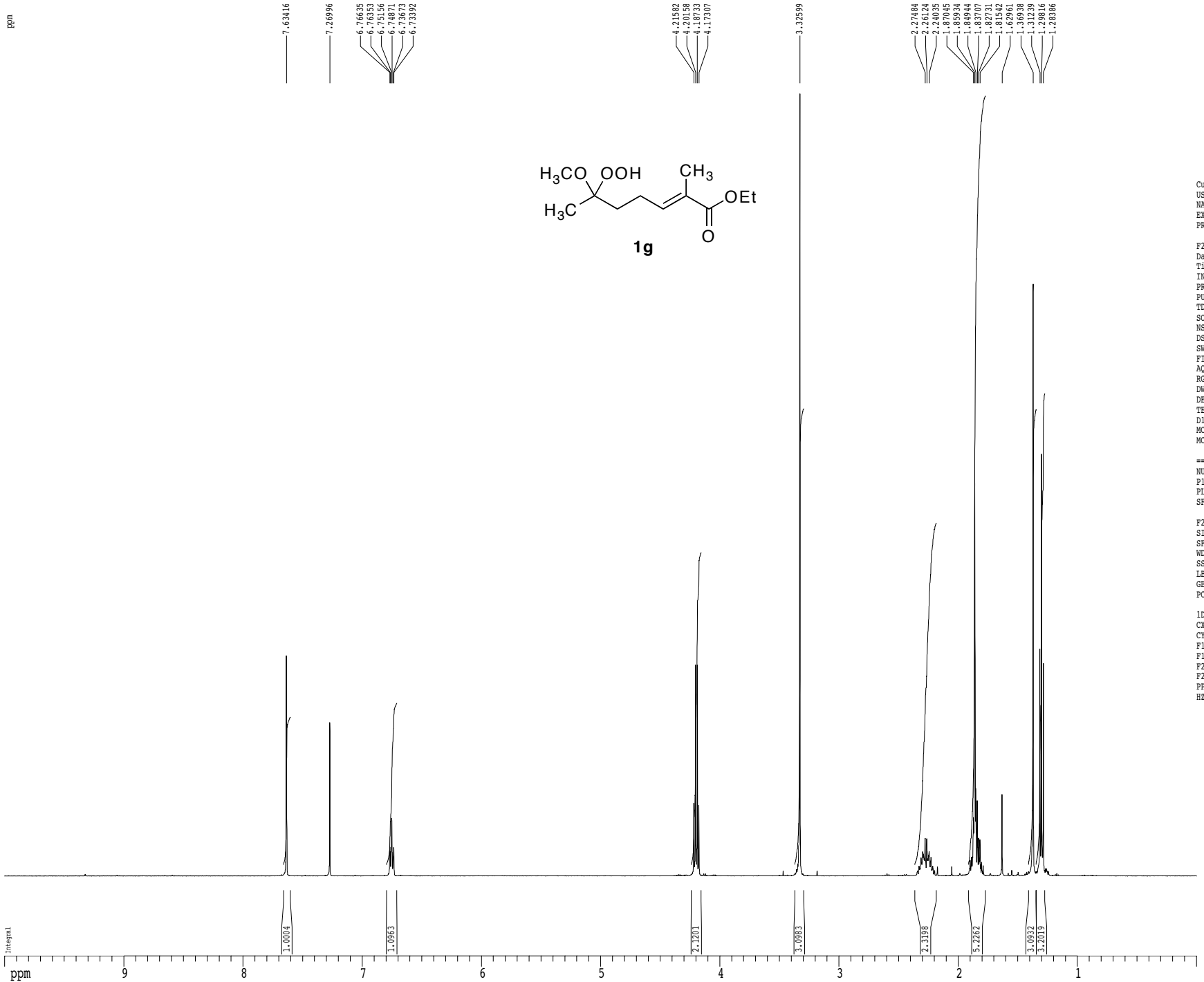
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 0.10

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67053 Hz/cm

1H spectrum



Current Data Parameters
USER shelli
NAME srw1193
EXPNO 5
PROCNO 1

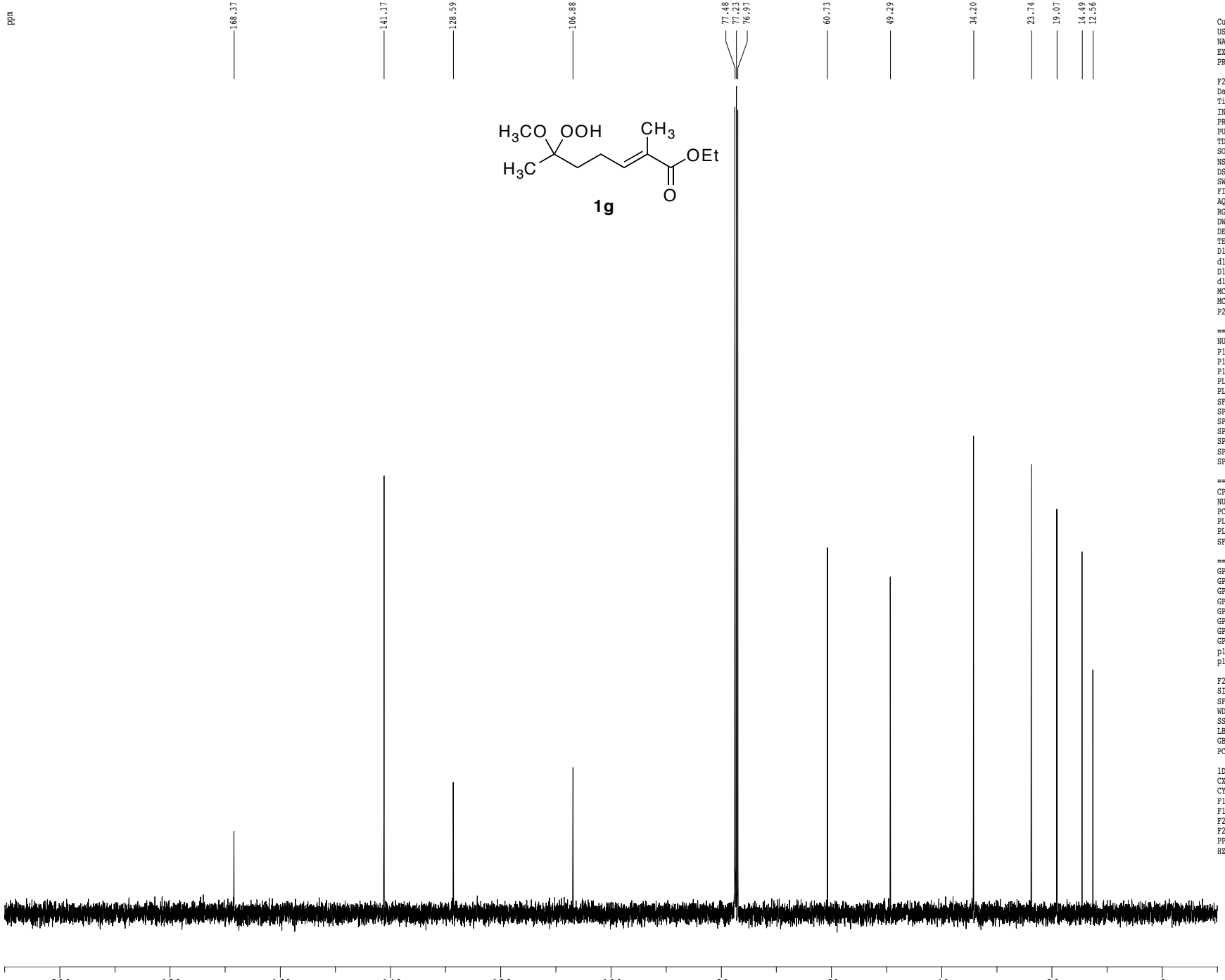
F2 - Acquisition Parameters
Date_ 20081119
Time 13.12
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200252 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw1193
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081119
Time 13.14
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 183
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

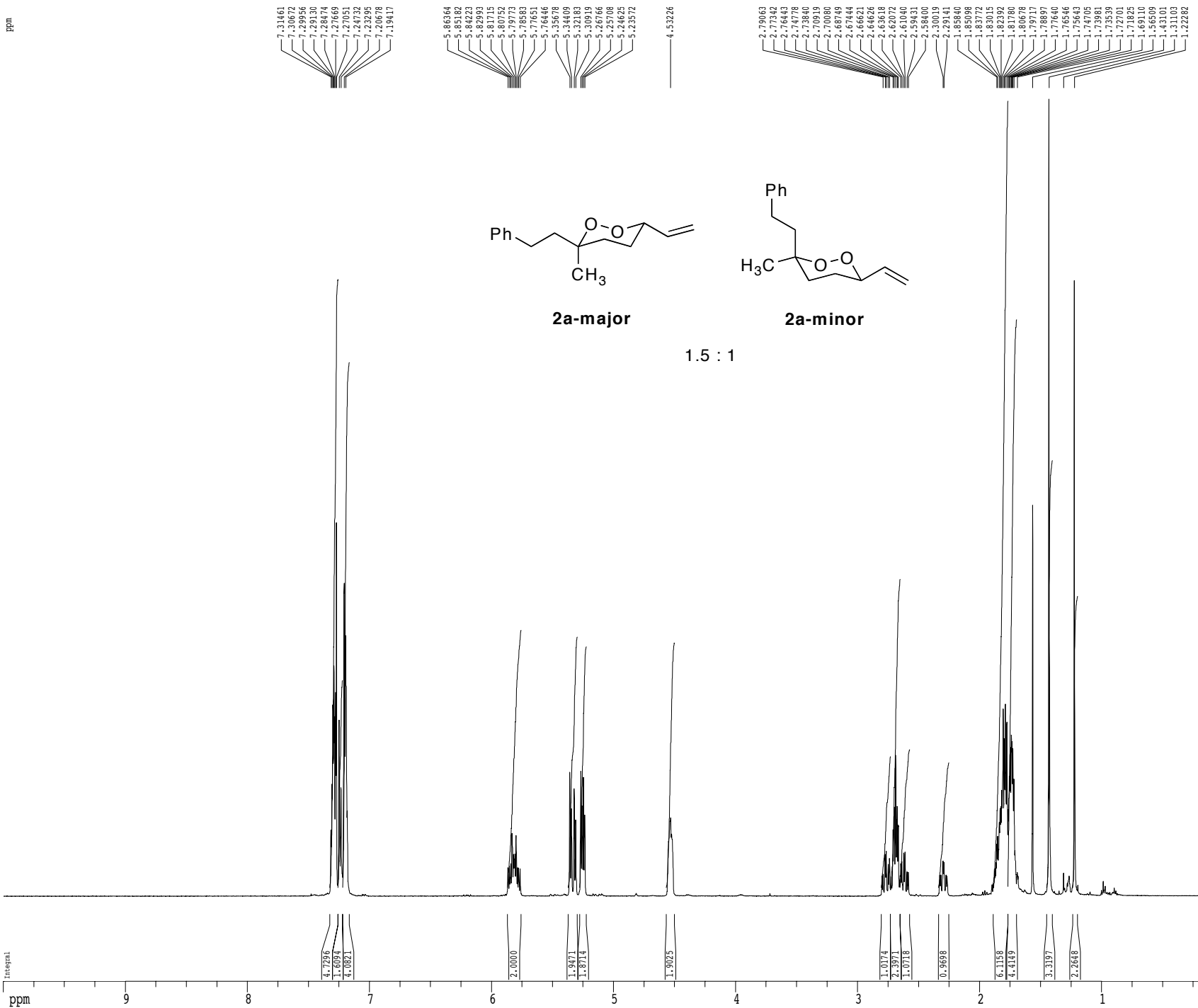
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803983 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67053 Hz/cm

1H spectrum



Current Data Parameters

USER	shelli
NAME	srw1171
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20081029
Time	17.00
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	8
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	5.7
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2235015 MHz

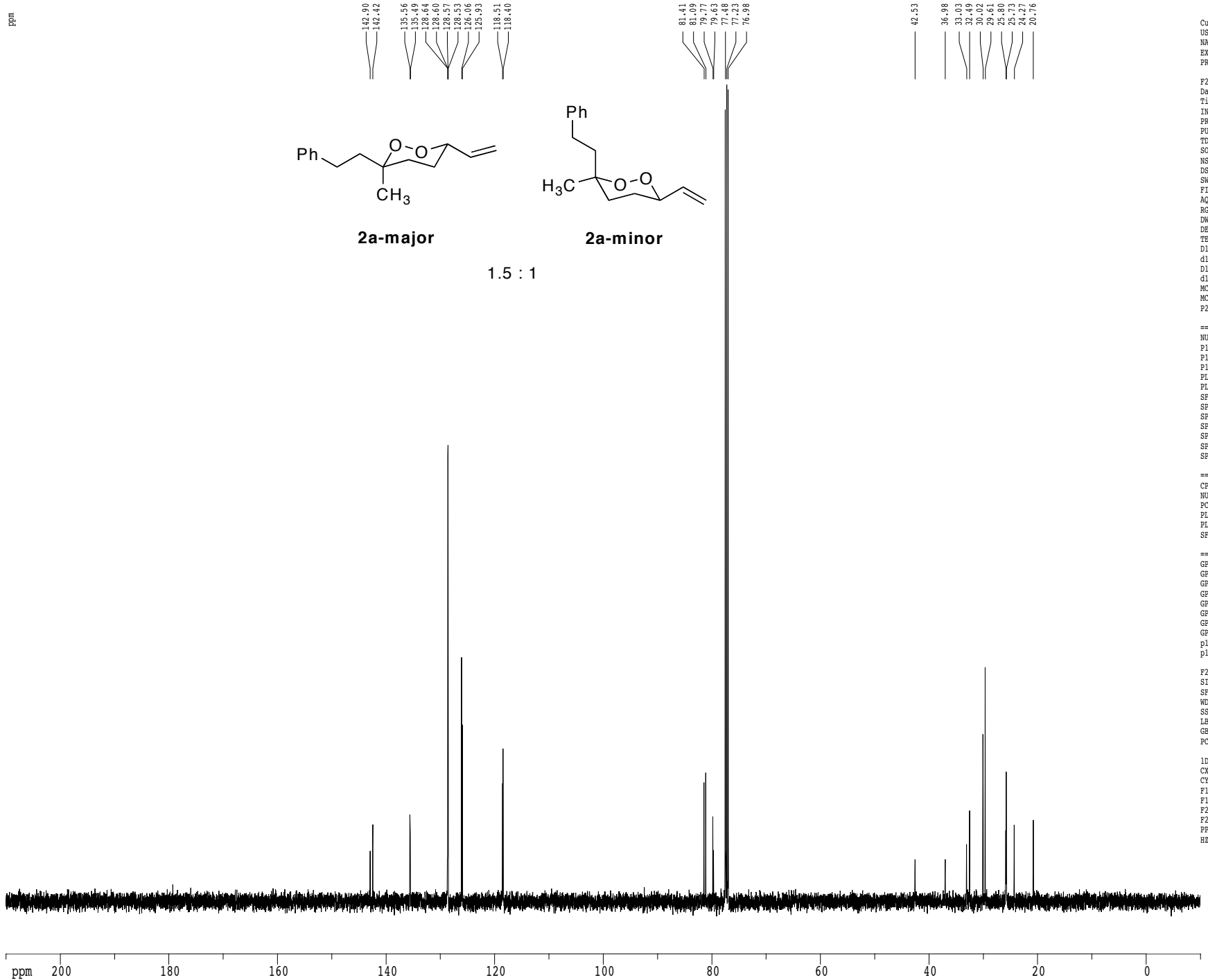
F2 - Processing parameters

SI	65536
SF	500.2200263 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	10.000 ppm
F1	5002.20 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.43860 ppm/cm
HZCM	219.39476 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw1171
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081029
Time 17.02
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 284
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

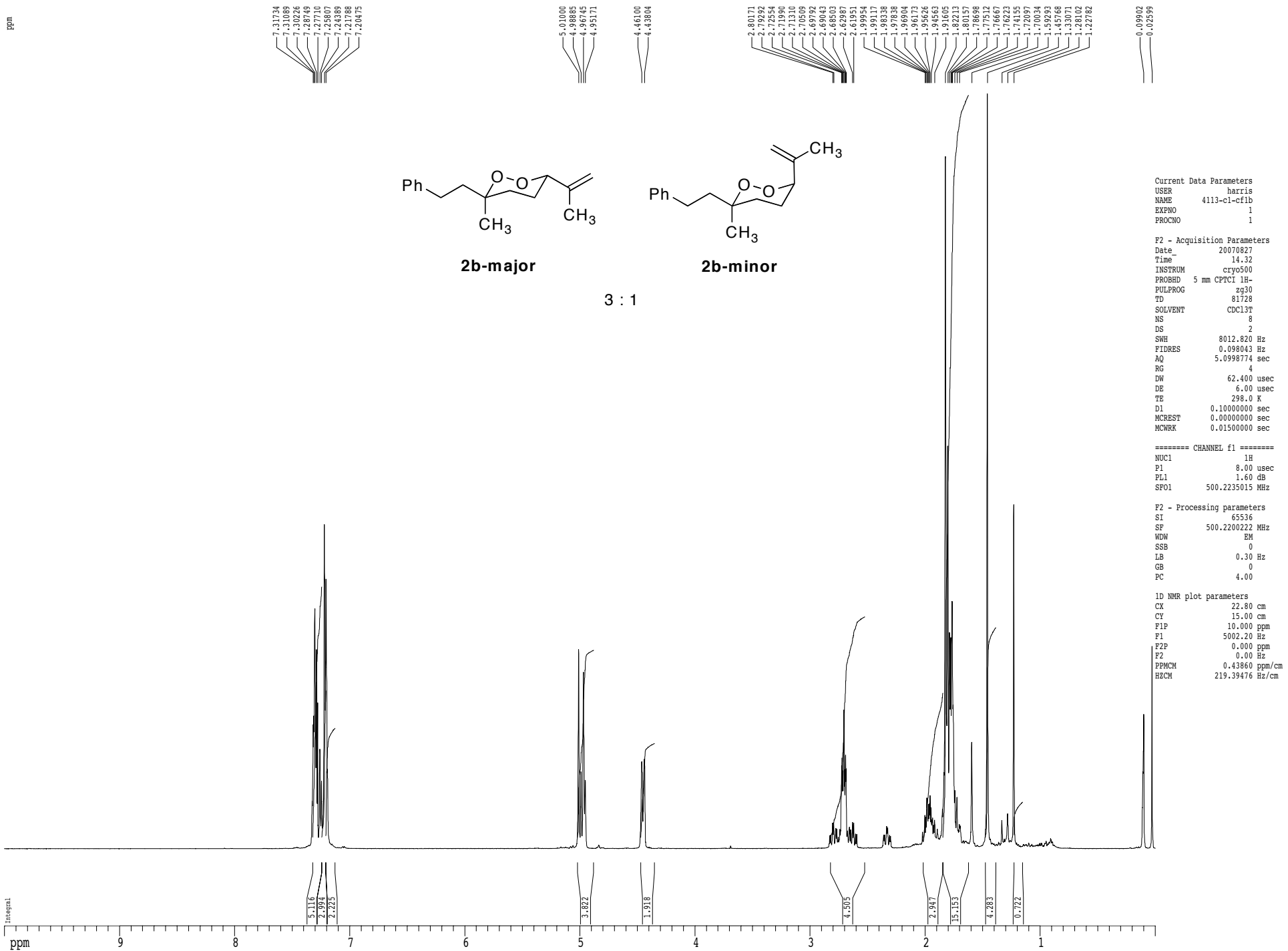
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803997 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67065 Hz/cm

1H spectrum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7.21788
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5.01000
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4.96745
4.95171
4.46100
4.43804
2.80171
2.79292
2.72554
2.71990
2.71310
2.70509
2.69792
2.69043
2.68503
2.62987
2.61951
1.99954
1.99117
1.98338
1.97838
1.96904
1.96173
1.95626
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1.82213
1.80157
1.78698
1.77512
1.76667
1.76223
1.74155
1.72097
1.70034
1.59293
1.45768
1.33071
1.28102
1.22782
0.09902
0.02599
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O
O
CH3
CH3
2b-major
Ph
O
O
CH3
CH3
2b-minor
3 : 1
Current Data Parameters
USER harris
NAME 4113-c1-cf1b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070827
Time 14.32
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 8.00 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200222 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm
Integral
5.116
2.994
2.225
3.822
1.918
4.505
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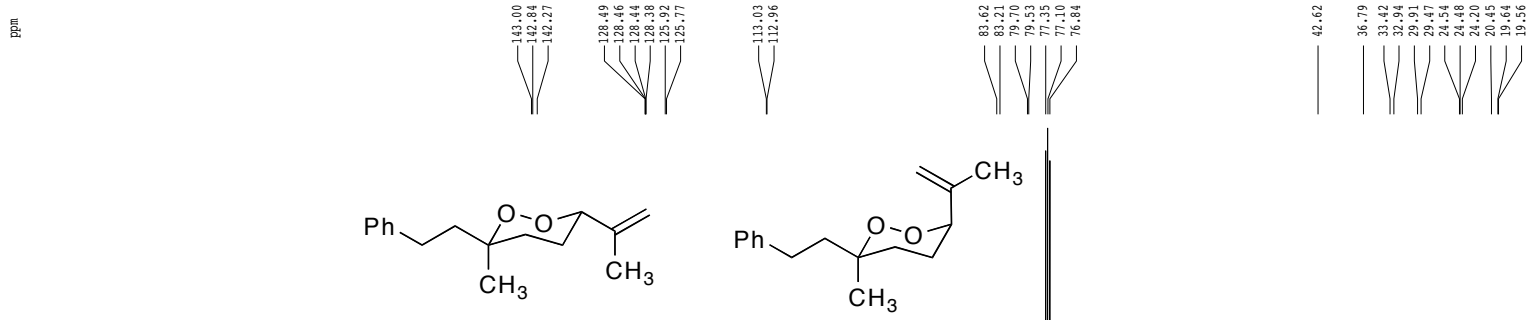
0.722
ppm
9
8
7
6
5
4
3
2
1

13C spectrum with 1H decoupling



2b-major

2b-minor

3 : 1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Current Data Parameters
USER              harris
NAME        4113-c1-cf1b
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_          20070827
Time              14.36
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG          zgdc30
TD                65418
SOLVENT           CDCl3
NS                  241
DS                    4
SWH           30303.031 Hz
FIDRES         0.463222 Hz
AQ             1.0794470 sec
RG                 8192
DW                16.500 usec
DE                 6.00 usec
TE                298.0 K
D1             0.25000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

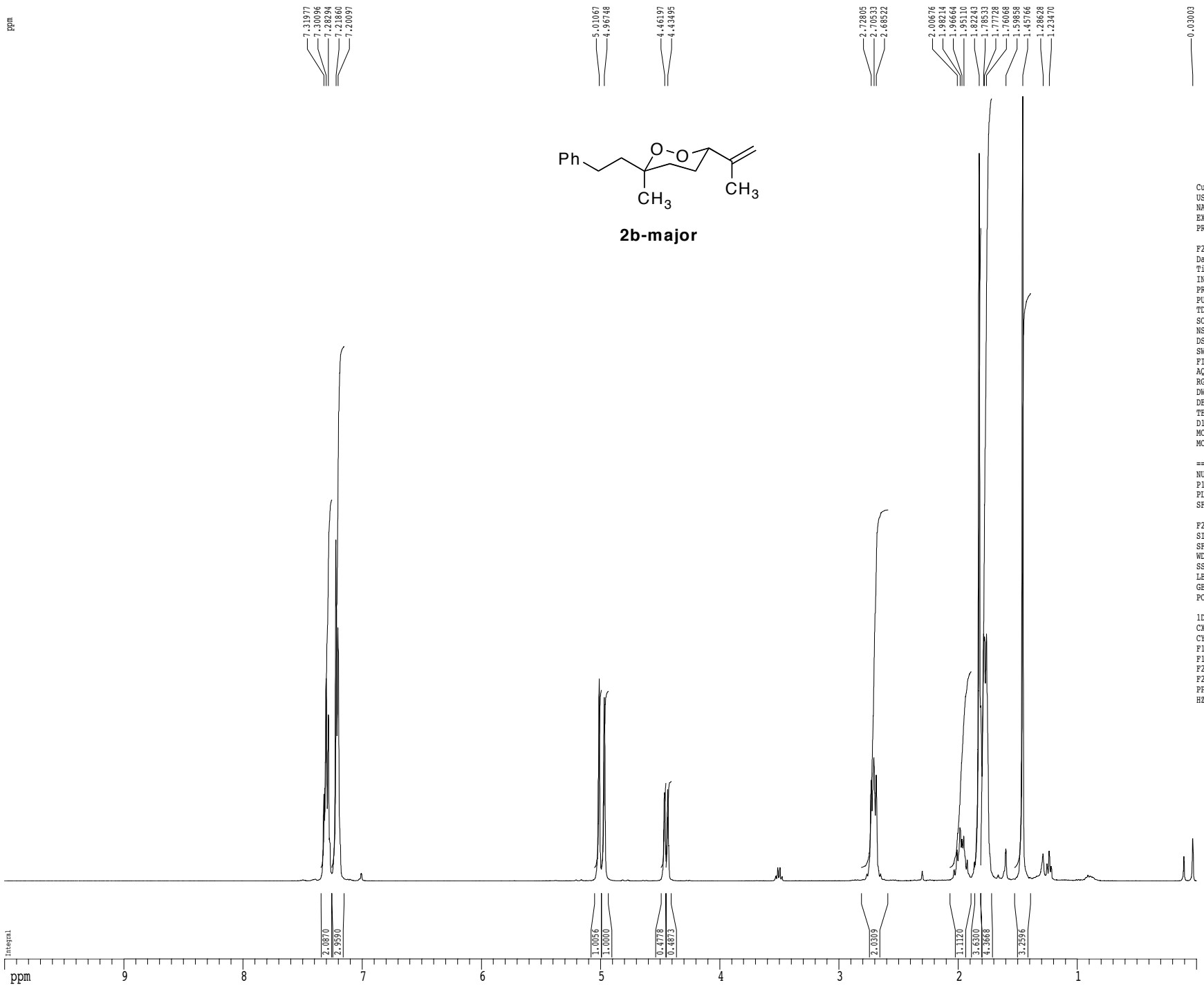
======== CHANNEL f1 ========
NUC1                13C
P1                15.00 usec
PL1               -1.00 dB
SFO1        125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12              23.54 dB
SFO2        500.2225011 MHz

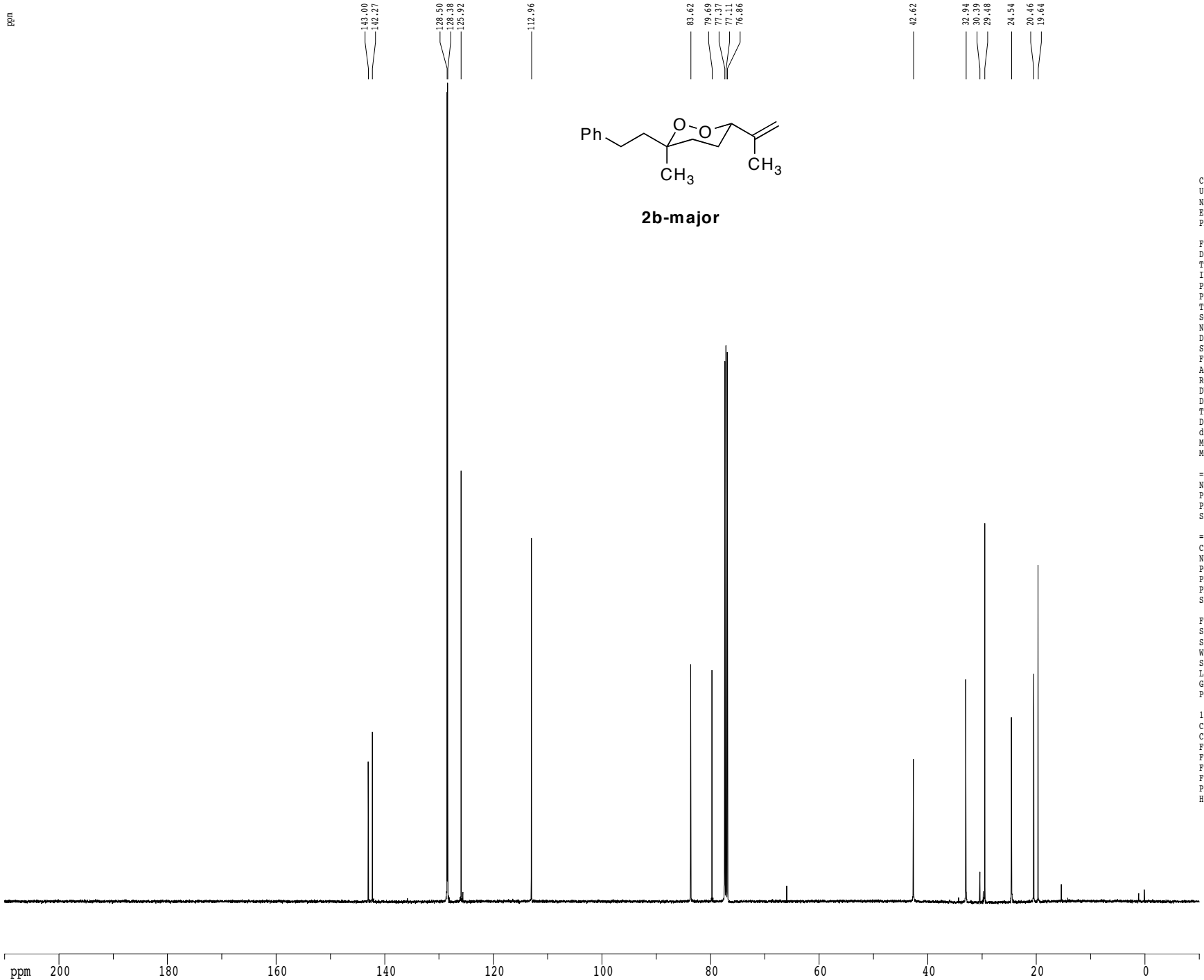
F2 - Processing parameters
SI                 65536
SF          125.7804182 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                15.65 cm
F1P              210.000 ppm
F1             26413.89 Hz
F2P              -10.000 ppm
F2             -1257.80 Hz
PPMCM           9.64912 ppm/cm
HZCM         1213.67078 Hz/cm
```

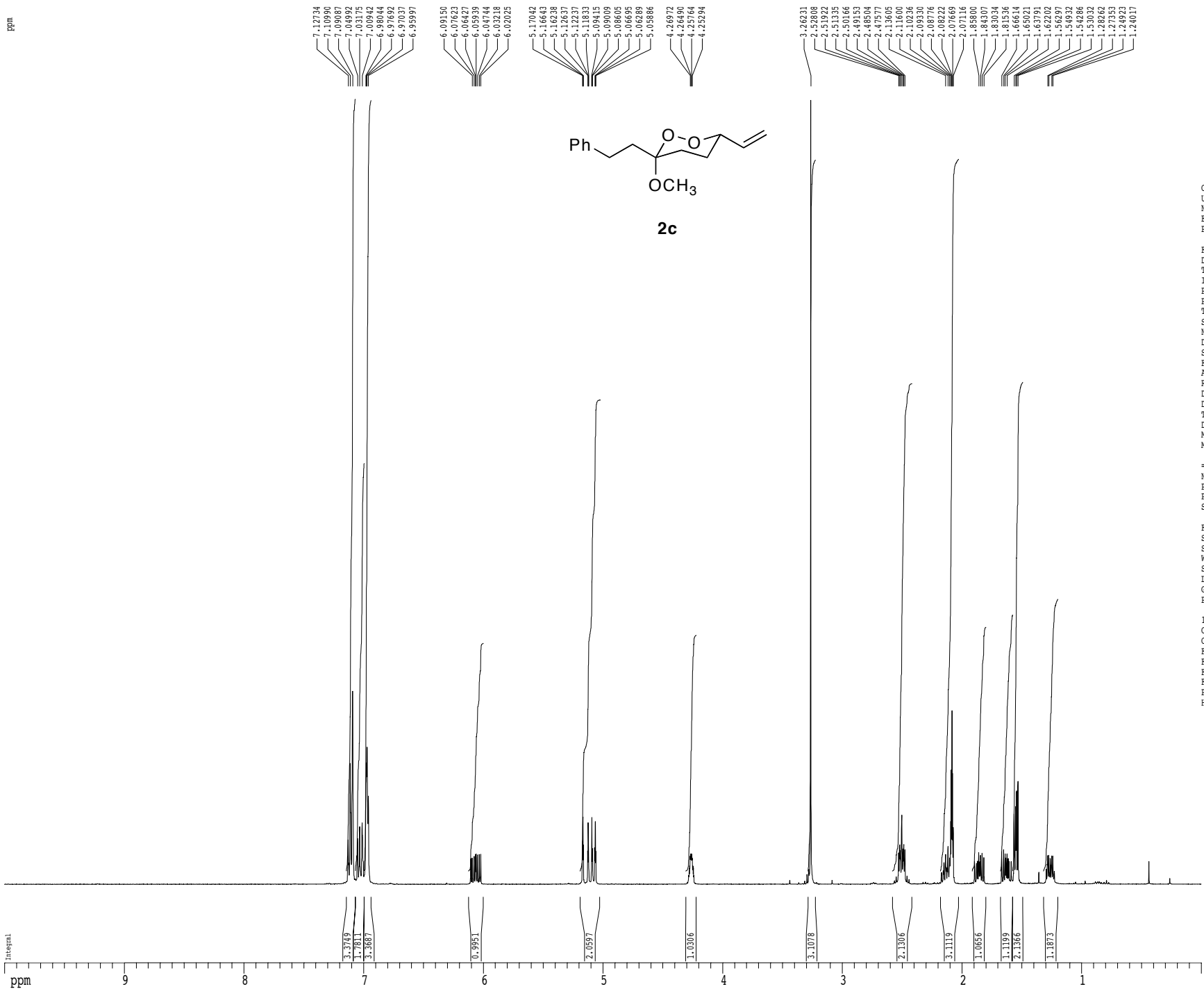
1H spectrum


ppm
7.31977
7.30096
7.28294
7.21860
7.20097
5.01067
4.96748
4.46197
4.43495
2.72805
2.70533
2.68522
2.00676
1.98214
1.96664
1.95110
1.82243
1.78533
1.77728
1.76068
1.59858
1.45766
1.28628
1.23470
0.03003
Ph
O
O
CH3
CH3
2b-major
Current Data Parameters
USER harris
NAME 4180-c2-cf1b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080213
Time 9.59
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 71.8
DW 78.000 usec
DE 4.50 usec
TE 297.9 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 175.49562 Hz/cm
Integral
2.0870
2.9590
1.0056
1.0000
0.4778
0.4873
2.0309
1.1120
3.6300
4.3668
3.2596
ppm
9
8
7
6
5
4
3
2
1

13C spectrum with 1H decoupling



1H spectrum



Current Data Parameters

USER	harris
NAME	5031-c2-cf1
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080205
Time	17.01
INSTRUM	drx400
PROBHD	5 mm QNP H/F/P
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	2
SWH	6410.256 Hz
FIDRES	0.097813 Hz
AQ	5.1118579 sec
RG	228.1
DW	78.000 usec
DE	4.50 usec
TE	298.1 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	12.00 usec
PL1	0.00 dB
SFO1	400.1328009 MHz

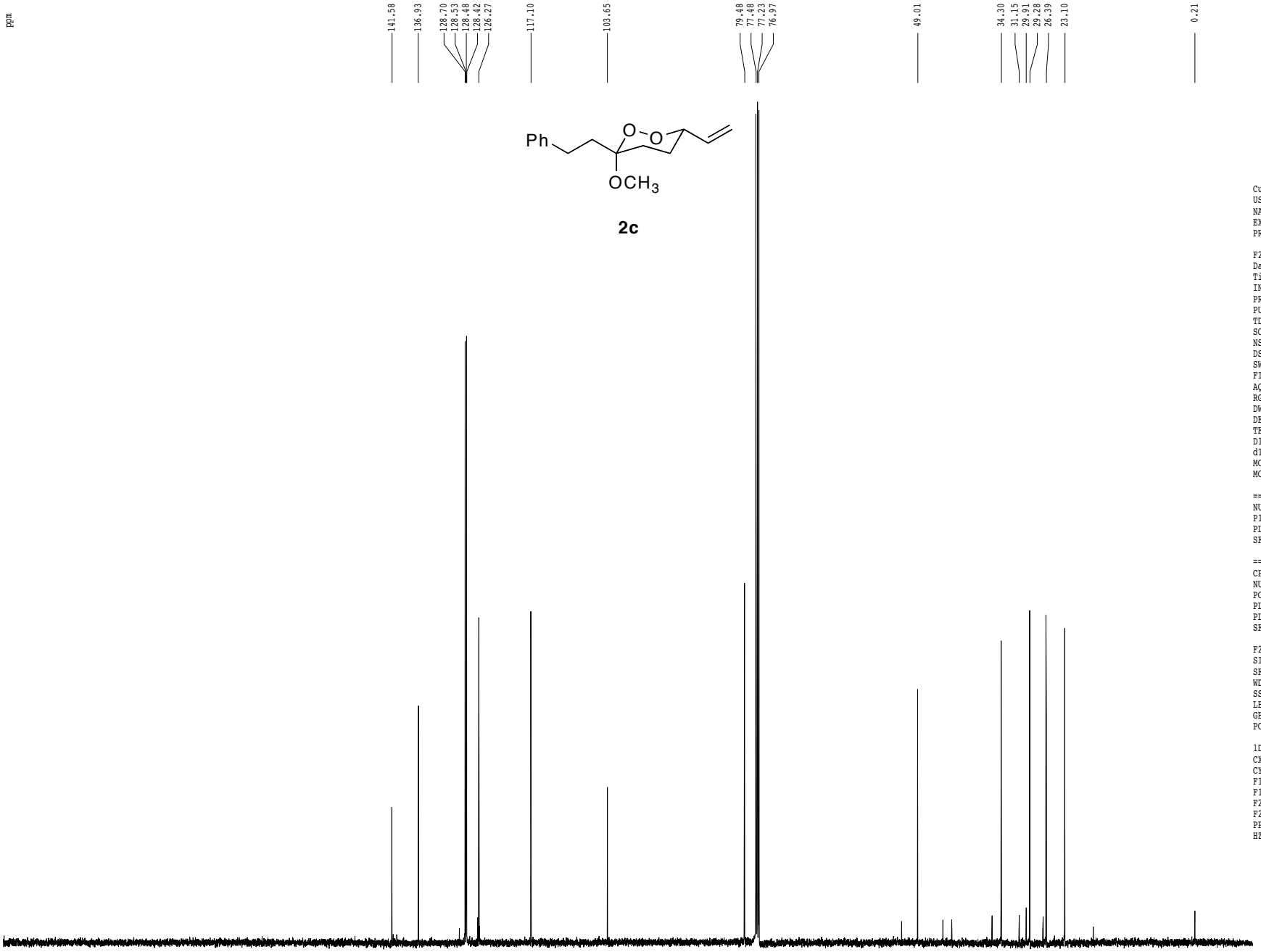
F2 - Processing parameters

SI	65536
SF	400.1300176 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.43860 ppm/cm
HZCM	175.49562 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER harris
NAME 5002-c1-cf2
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080108
Time 12.11
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 447
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 11585.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

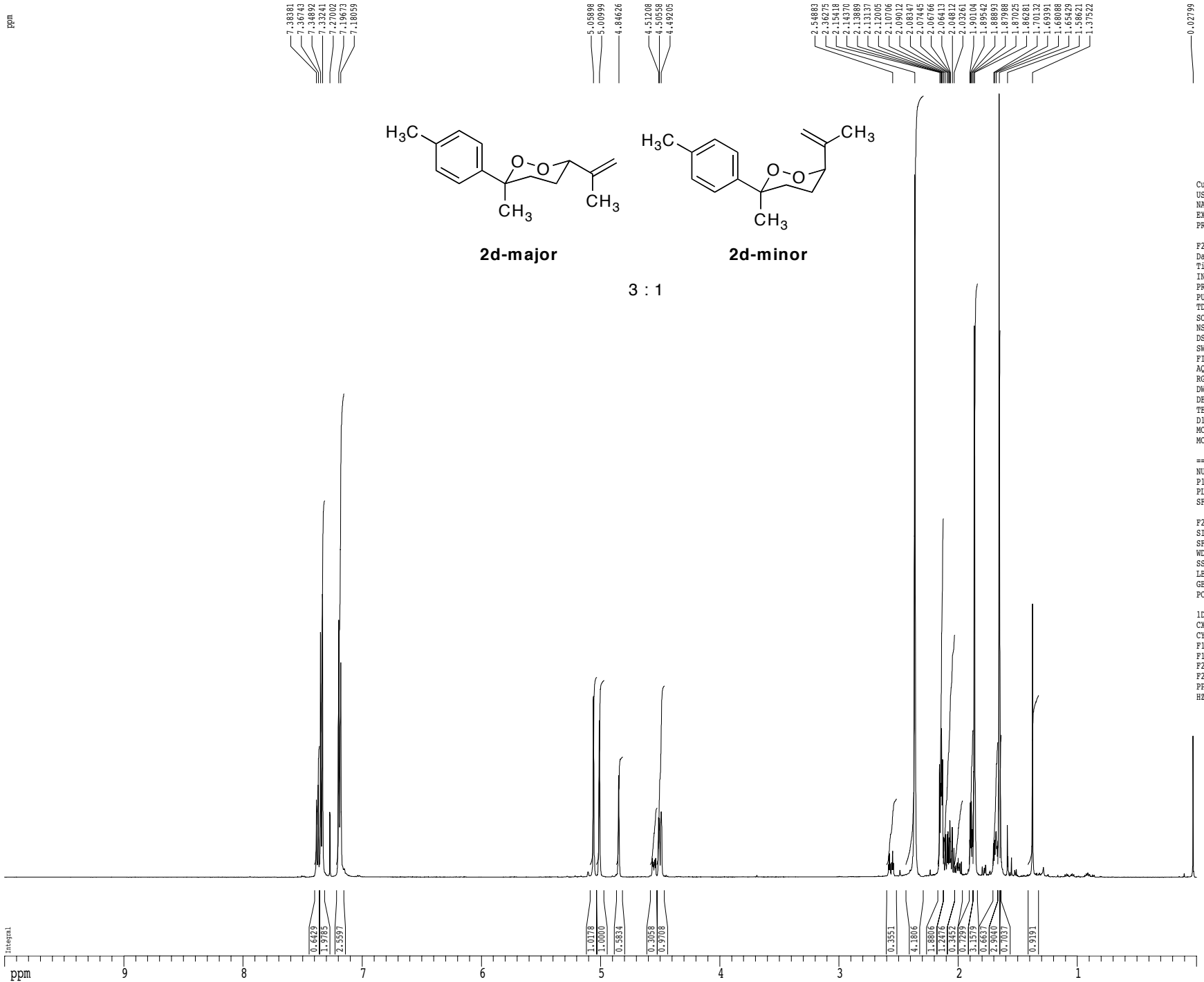
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 23.54 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804002 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67065 Hz/cm

1H spectrum




```
Current Data Parameters
USER              harris
NAME         5090-c1-cf1
EXPNO                  1
PROCNO                 1

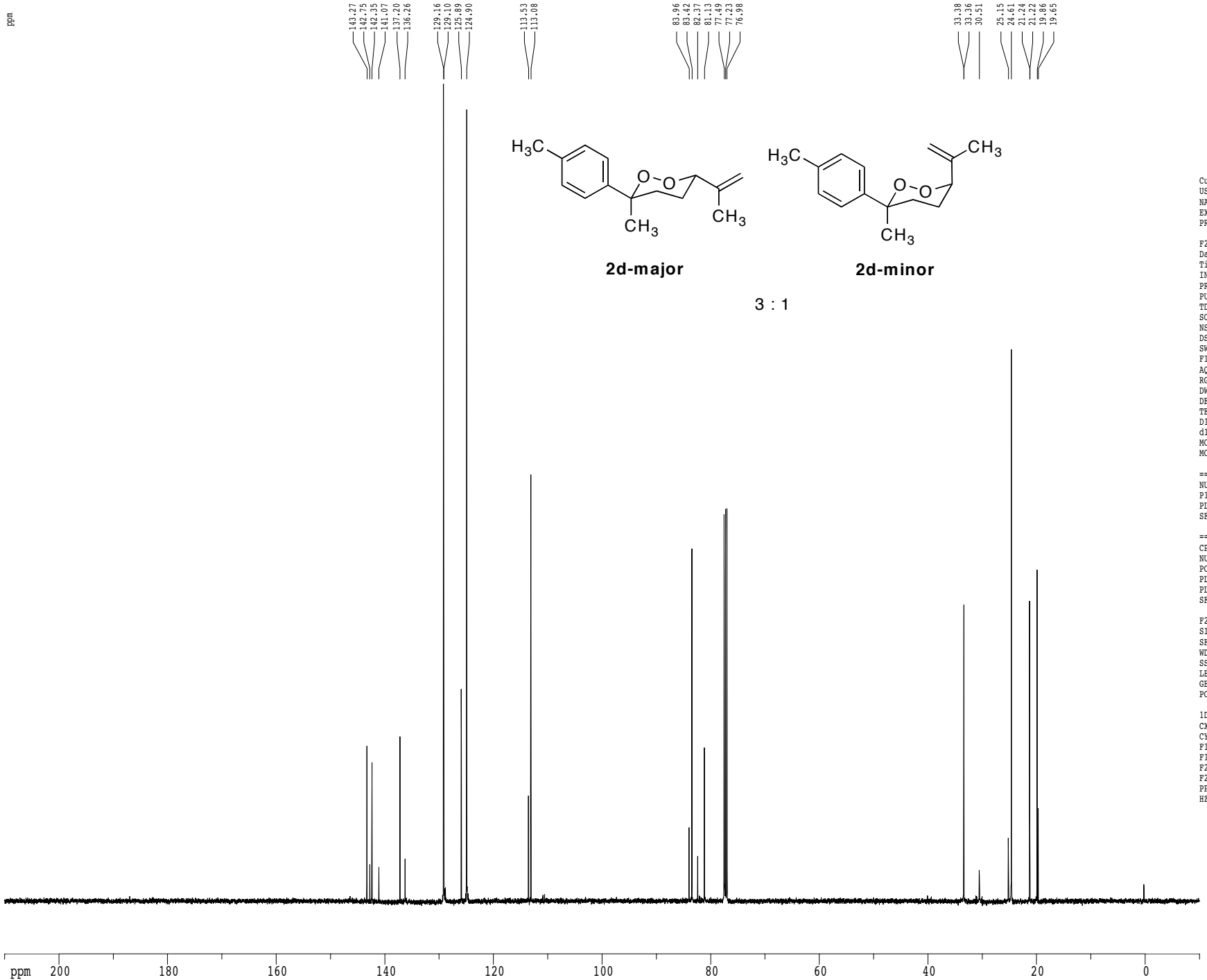
F2 - Acquisition Parameters
Date_           20080314
Time              11.43
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES             0.098043 Hz
AQ              5.0998774 sec
RG                   3.6
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.38 usec
PL1                1.60 dB
SFO1         500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF            500.2200258 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  4.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P               10.000 ppm
F1              5002.20 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM           0.43860 ppm/cm
HZCM          219.39476 Hz/cm
```

13C spectrum with 1H decoupling



Current Data Parameters
USER harris
NAME 5090-c1-cf1
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080314
Time 11.47
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 225
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 13004
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

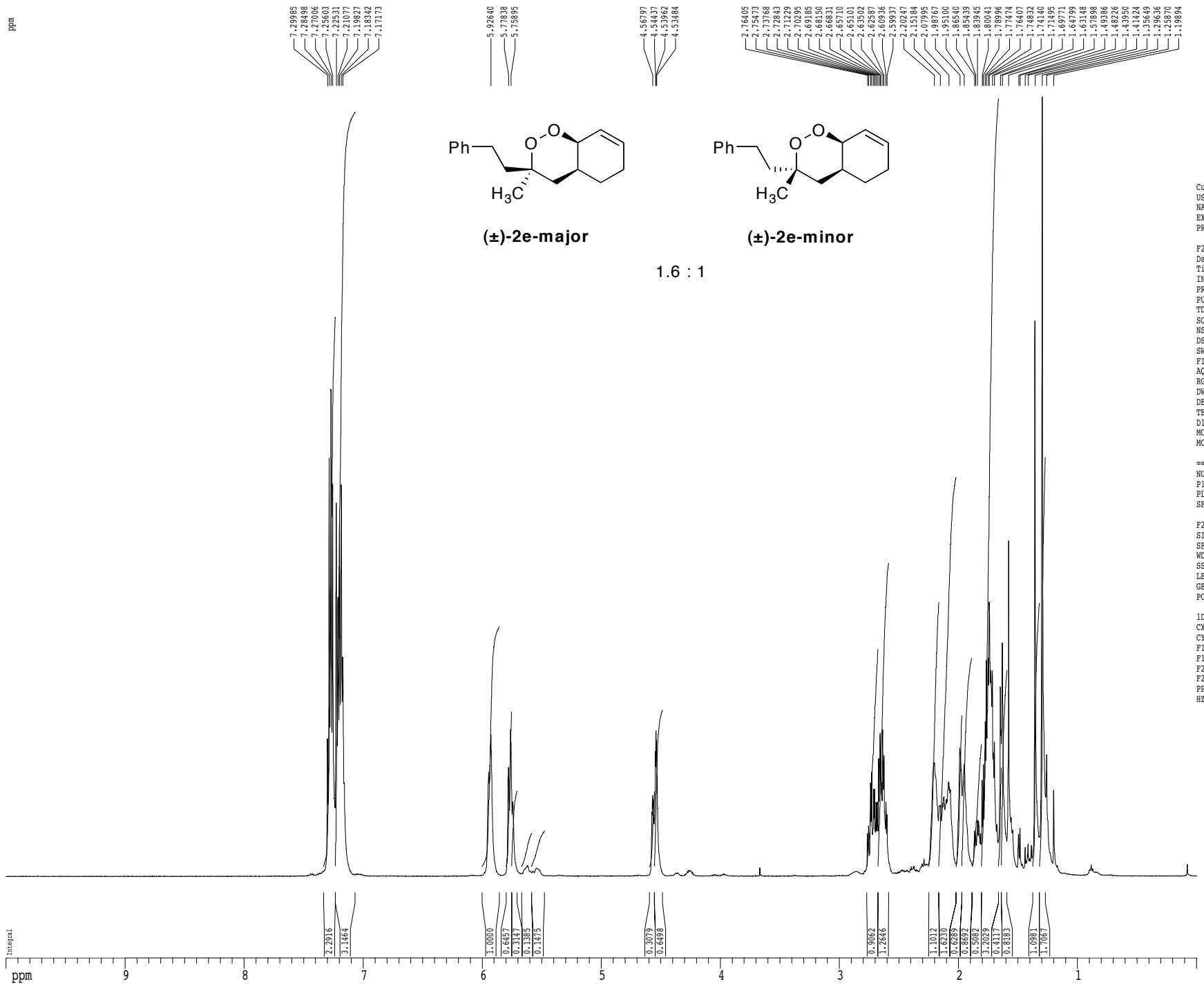
======== CHANNEL f1 ========
NUC1 13C
P1 14.75 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.80 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804034 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67065 Hz/cm

1H spectrum




```
Current Data Parameters
USER              shelli
NAME              srw1187
EXPNO                   2
PROCNO                  1

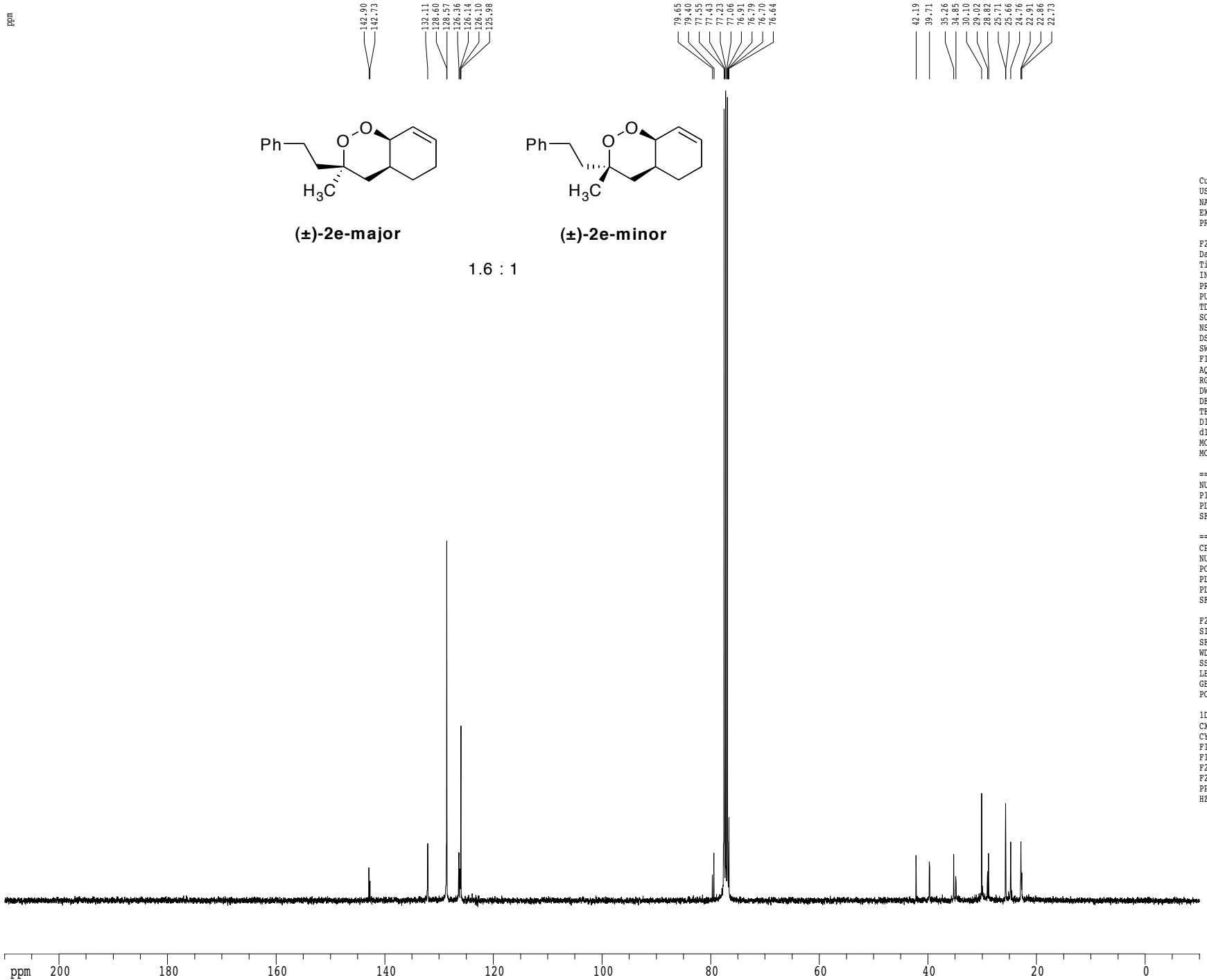
F2 - Acquisition Parameters
Date_           20081111
Time                9.01
INSTRUM          cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  81728
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                      4
DW                 62.400 usec
DE                  6.00 usec
TE                298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  7.50 usec
PL1                 1.60 dB
SFO1         500.2235015 MHz

F2 - Processing parameters
SI                  65536
SF           500.2200321 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   4.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.00 cm
F1P                10.000 ppm
F1                5002.20 Hz
F2P                 0.000 ppm
F2                  0.00 Hz
PPMCM            0.43860 ppm/cm
HZCM           219.39476 Hz/cm
```

13C spectrum with 1H decoupling

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Current Data Parameters
USER              shelli
NAME             srw2130
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090406
Time                21.18
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG           zgdc30
TD                 65536
SOLVENT            CD3OD
NS                 25866
DS                     4
SWH           24154.590 Hz
FIDRES         0.368570 Hz
AQ             1.3566452 sec
RG                9195.2
DW                20.700 usec
DE                20.39 usec
TE                323.0 K
D1            0.10000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

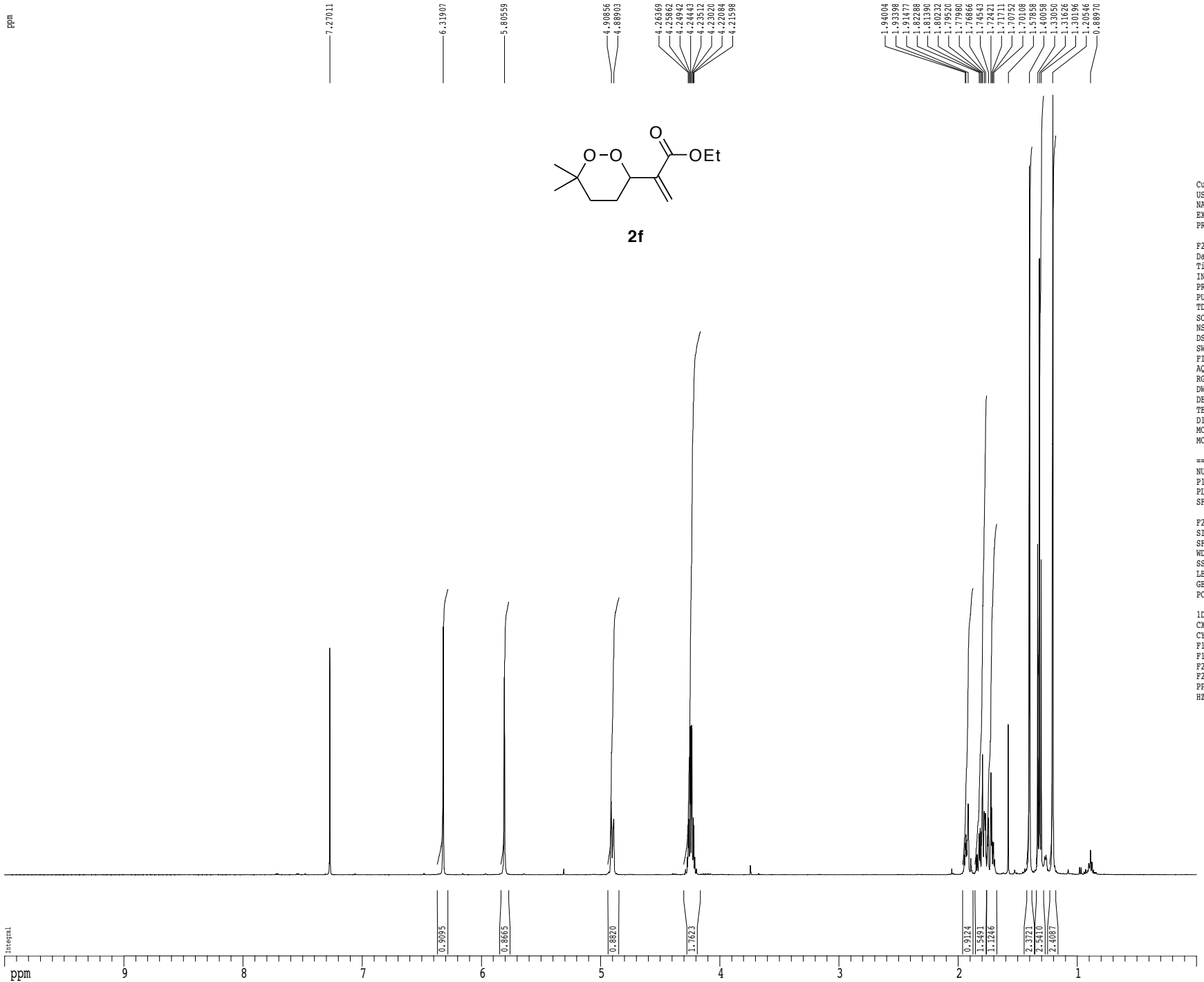
======== CHANNEL f1 ========
NUC1                13C
P1                10.75 usec
PL1                0.00 dB
SFO1        100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2           mlev16
NUC2                1H
PCPD2             90.00 usec
PL2                0.00 dB
PL12              17.70 dB
SFO2        400.1328009 MHz

F2 - Processing parameters
SI                 65536
SF          100.6127418 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  0.25

1D NMR plot parameters
CX                 22.80 cm
CY                 15.50 cm
F1P              210.000 ppm
F1             21128.68 Hz
F2P              -10.000 ppm
F2             -1006.13 Hz
PPMCM           9.64912 ppm/cm
HZCM          970.82471 Hz/cm
```

1H spectrum



Current Data Parameters
USER shelli
NAME srw1191
EXPNO 2
PROCNO 1

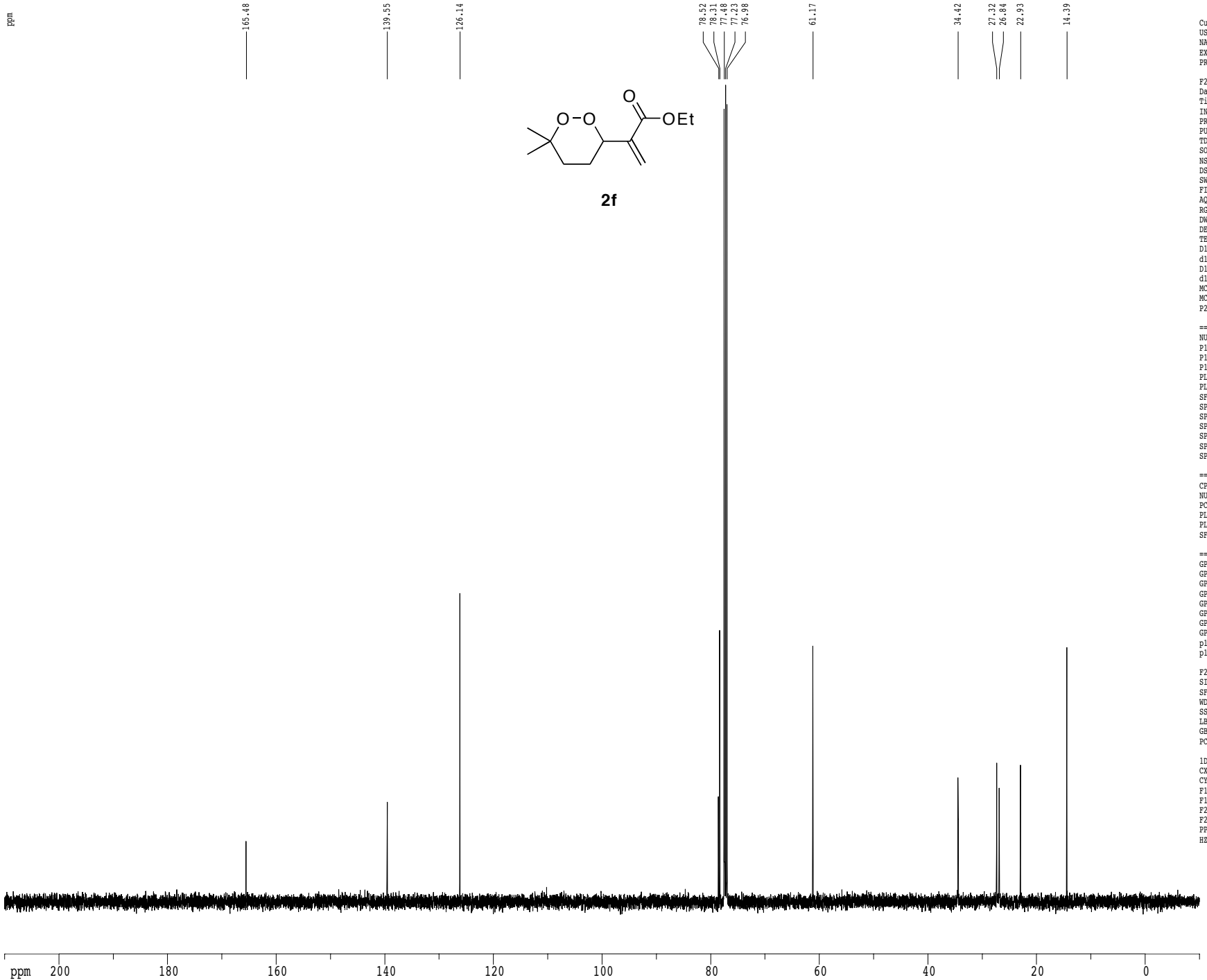
F2 - Acquisition Parameters
Date_ 20081114
Time 10.19
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200251 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw1191
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081114
Time 10.21
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 184
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

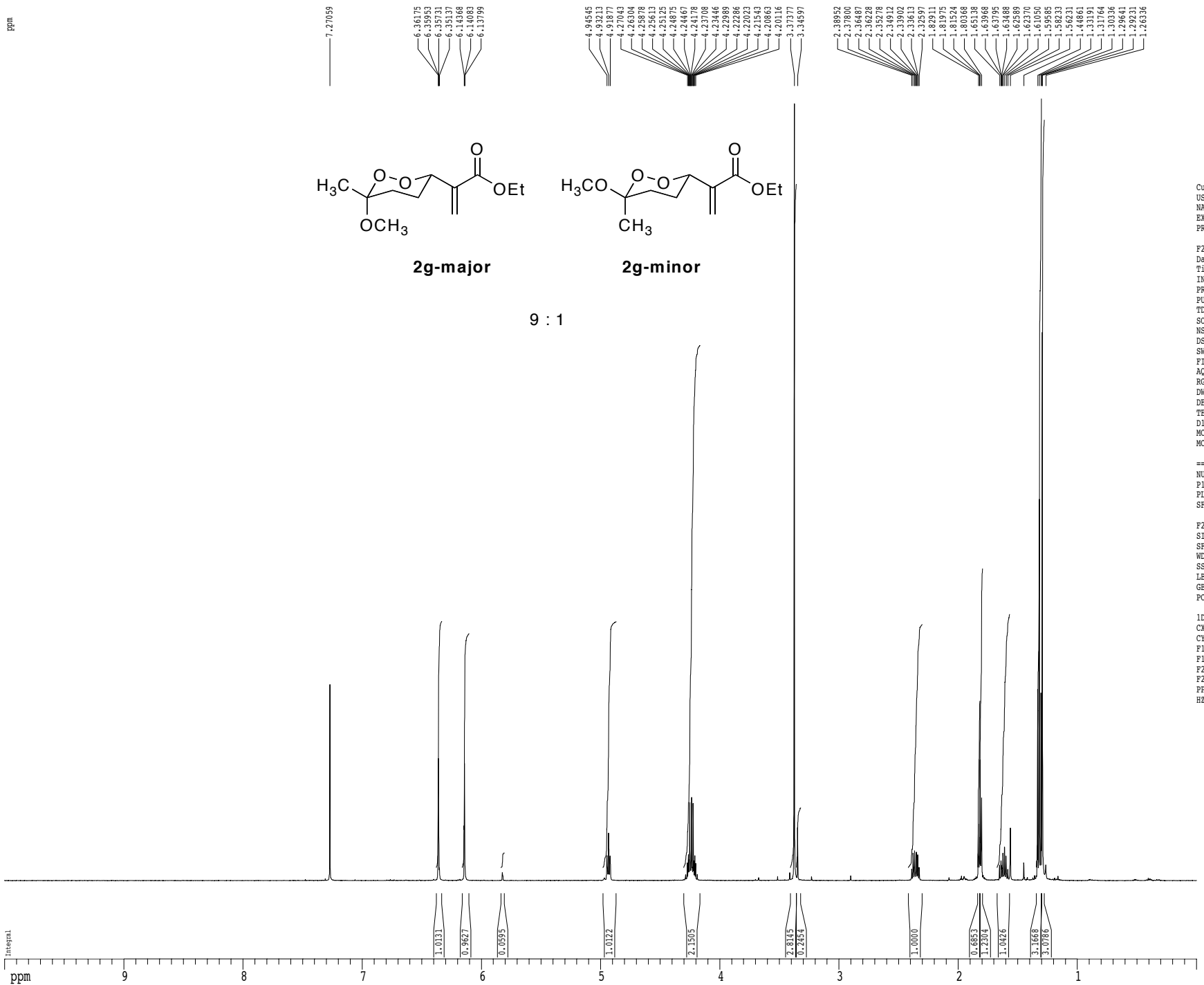
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803978 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67053 Hz/cm

1H spectrum




```
Current Data Parameters
USER              shelli
NAME             srw1196
EXPNO                  3
PROCNO                 1

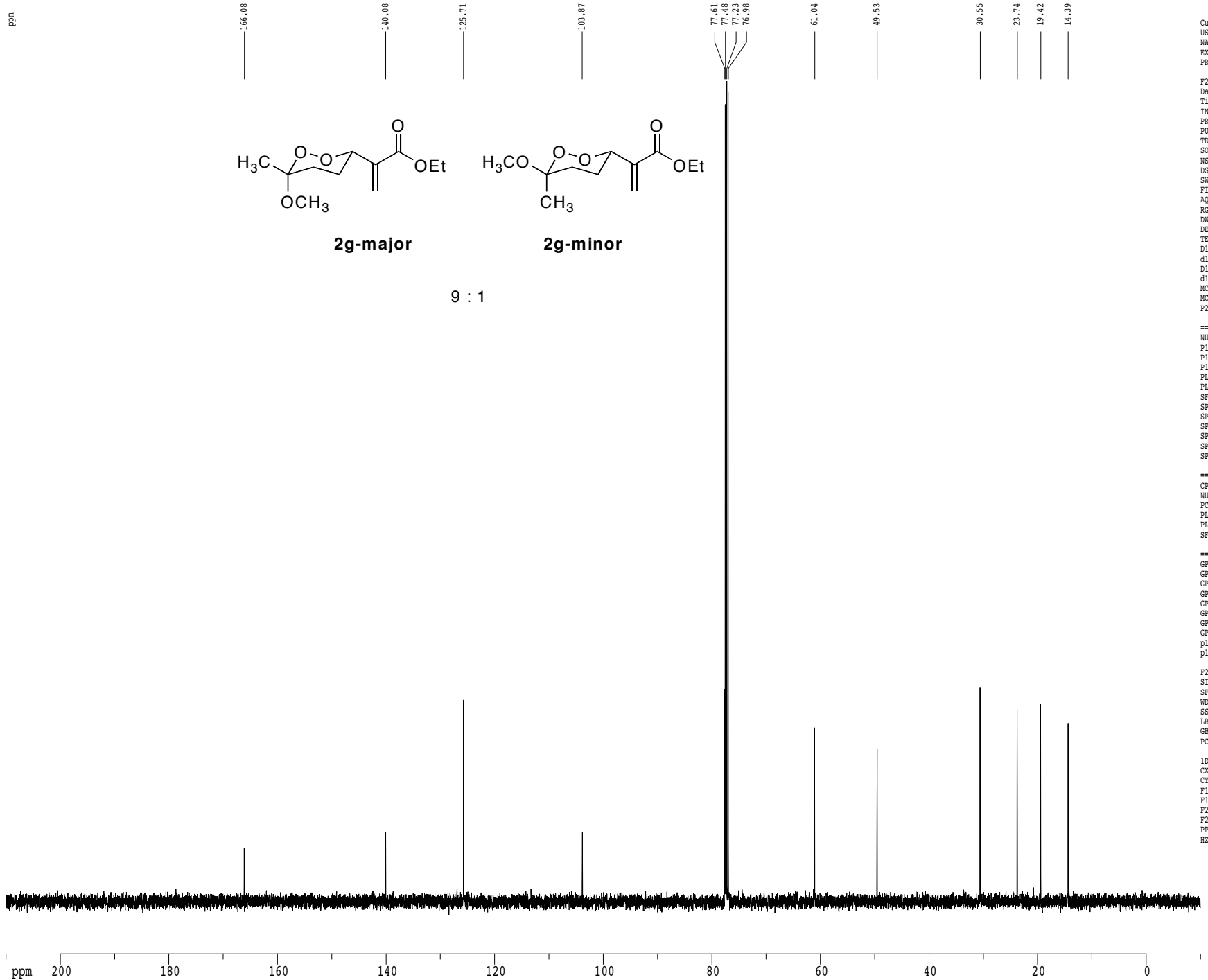
F2 - Acquisition Parameters
Date_           20081119
Time               17.10
INSTRUM            gn500
PROBHD   5 mm broadband
PULPROG             zg30
TD                 81728
SOLVENT             DMSO
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.098043 Hz
AQ              5.0998774 sec
RG                1149.4
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -3.00 dB
SFO1        499.7234980 MHz

F2 - Processing parameters
SI                 65536
SF          499.7200260 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00

1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P              10.000 ppm
F1              4997.20 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM          0.43860 ppm/cm
HZCM          219.17546 Hz/cm
```

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw1196
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081119
Time 17.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT C6D6
NS 201
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

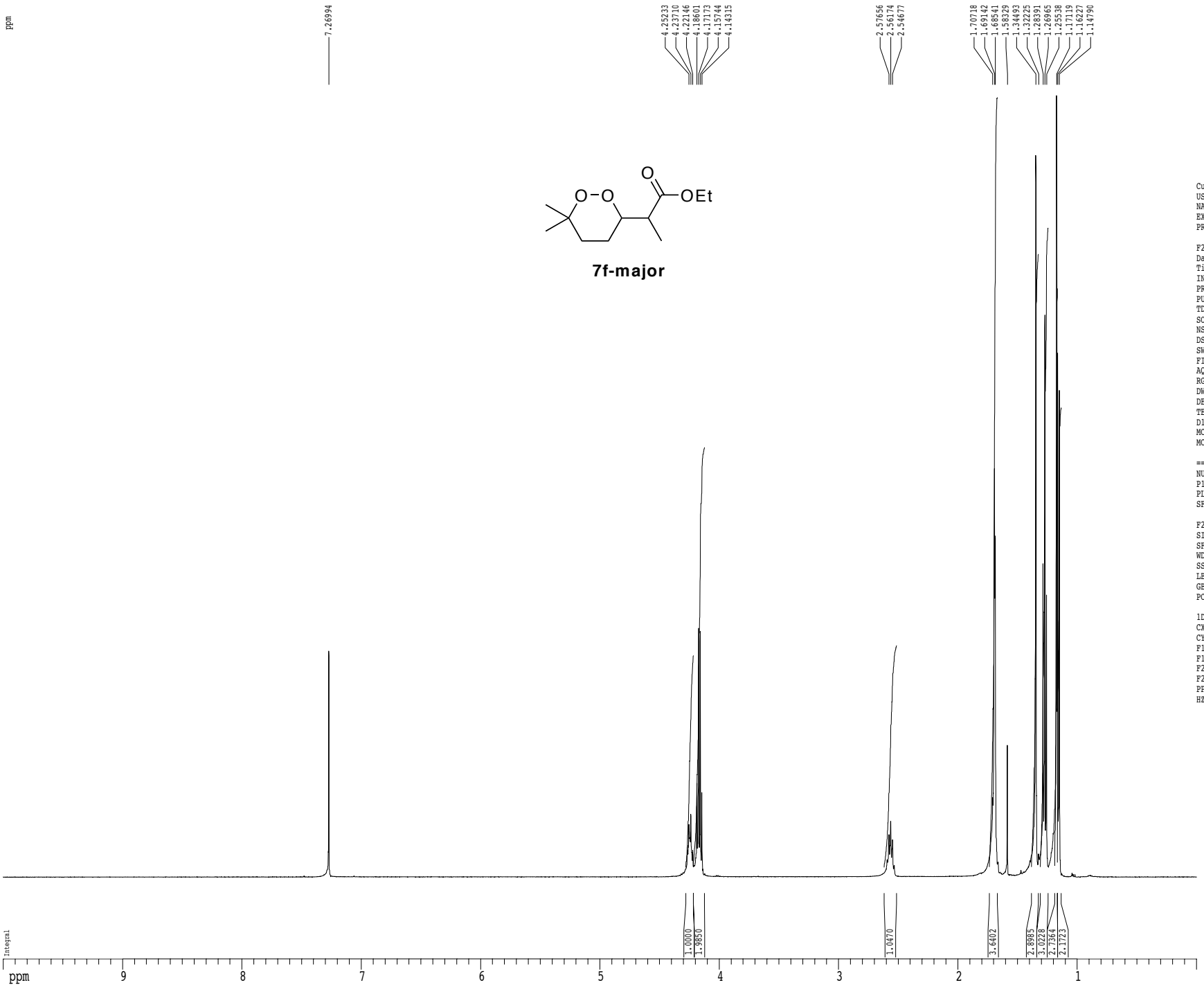
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803978 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67053 Hz/cm

1H spectrum



Current Data Parameters
USER shelli
NAME srw2014
EXPNO 2
PROCNO 1

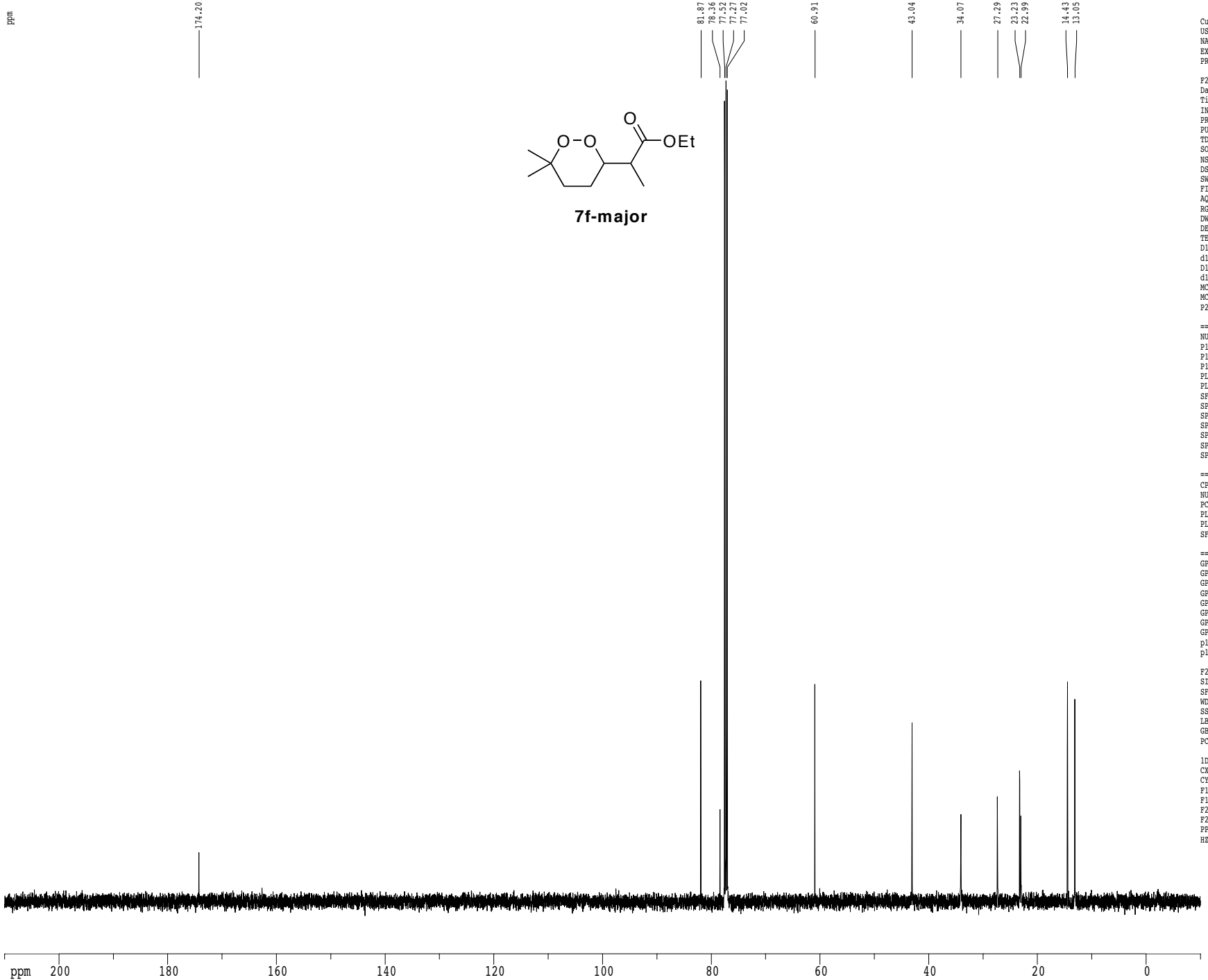
F2 - Acquisition Parameters
Date_ 20081217
Time 12.42
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200261 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39476 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling




```
Current Data Parameters
USER                shelli
NAME              srw2014
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20081217
Time                12.44
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                  65536
SOLVENT             CDCl3
NS                    249
DS                     16
SWH             30303.031 Hz
FIDRES          0.462388 Hz
AQ              1.0813940 sec
RG                 7298.2
DW                16.500 usec
DE                 6.00 usec
TE                 298.0 K
D1             0.25000000 sec
d11            0.03000000 sec
D16            0.00020000 sec
d17            0.00019600 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec
P2                 29.70 usec

======== CHANNEL f1 ========
NUC1                  13C
P1                 14.85 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                3.60 dB
SP2                3.60 dB
SPNAM1    Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1               0.00 Hz
SPOFF2               0.00 Hz

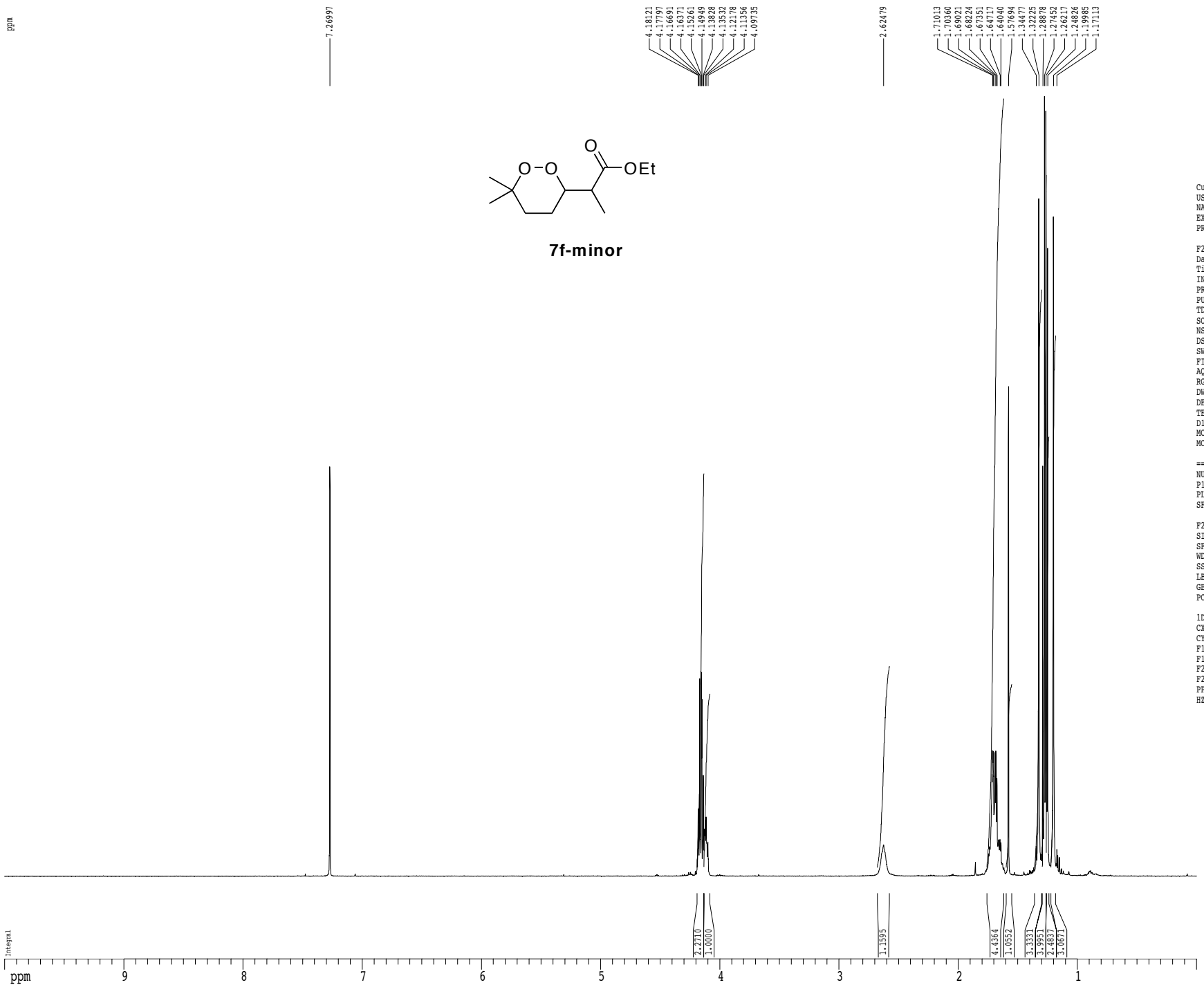
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2         500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1                0.00 %
GPX2                0.00 %
GPY1                0.00 %
GPY2                0.00 %
GPZ1               30.00 %
GPZ2               50.00 %
p15               500.00 usec
p16              1000.00 usec

F2 - Processing parameters
SI                  65536
SF           125.7803937 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P              210.000 ppm
F1             26413.88 Hz
F2P              -10.000 ppm
F2             -12257.80 Hz
PPMCM           9.64912 ppm/cm
HZCM         1213.67053 Hz/cm
```

1H spectrum



Current Data Parameters

USER	shelli
NAME	srw2027
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20090112
Time	9.57
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	8
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	4
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2235015 MHz

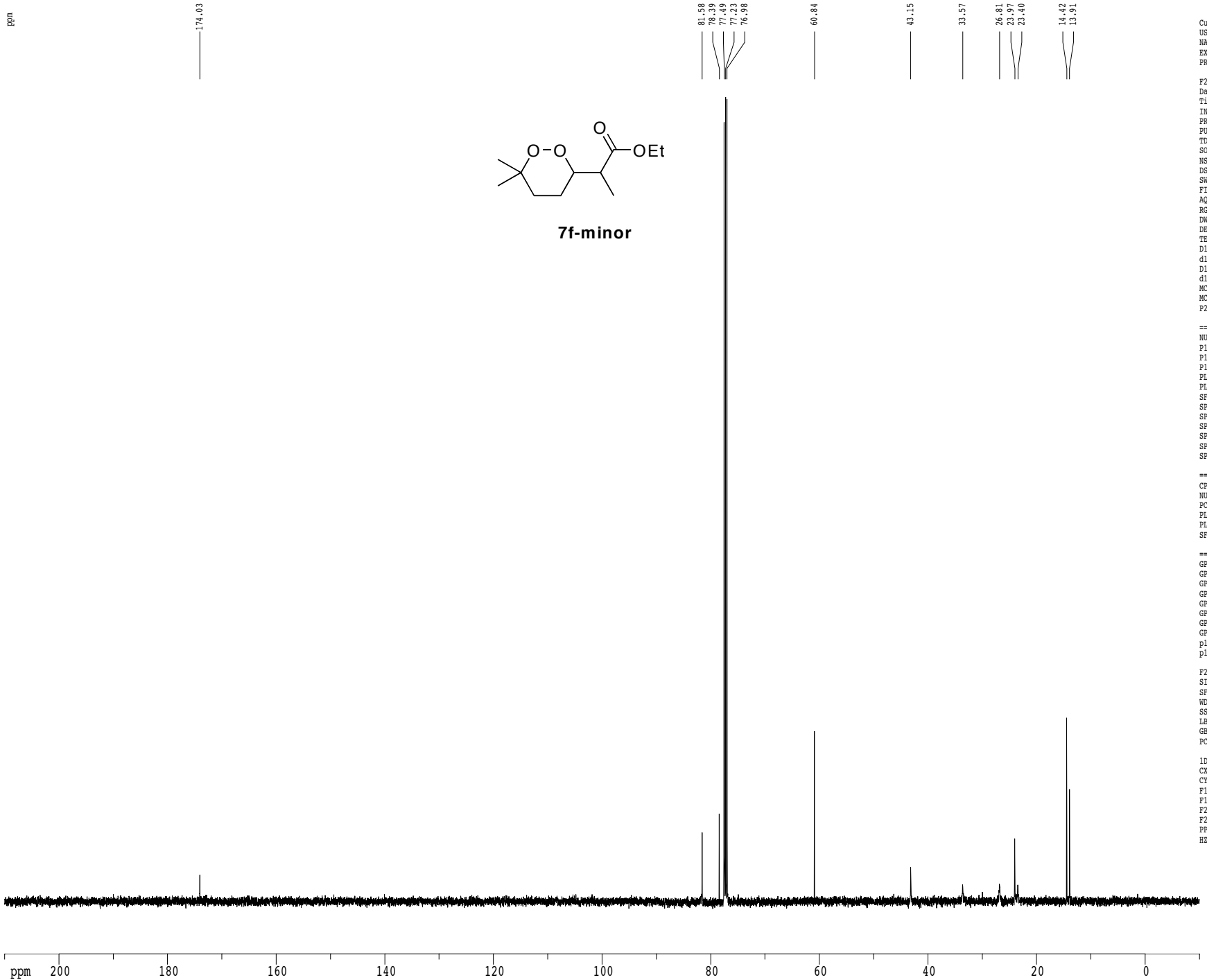
F2 - Processing parameters

SI	65536
SF	500.2200258 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	10.000 ppm
F1	5002.20 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.43860 ppm/cm
HZCM	219.39476 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER shelli
NAME srw2027
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090112
Time 10.00
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 480
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

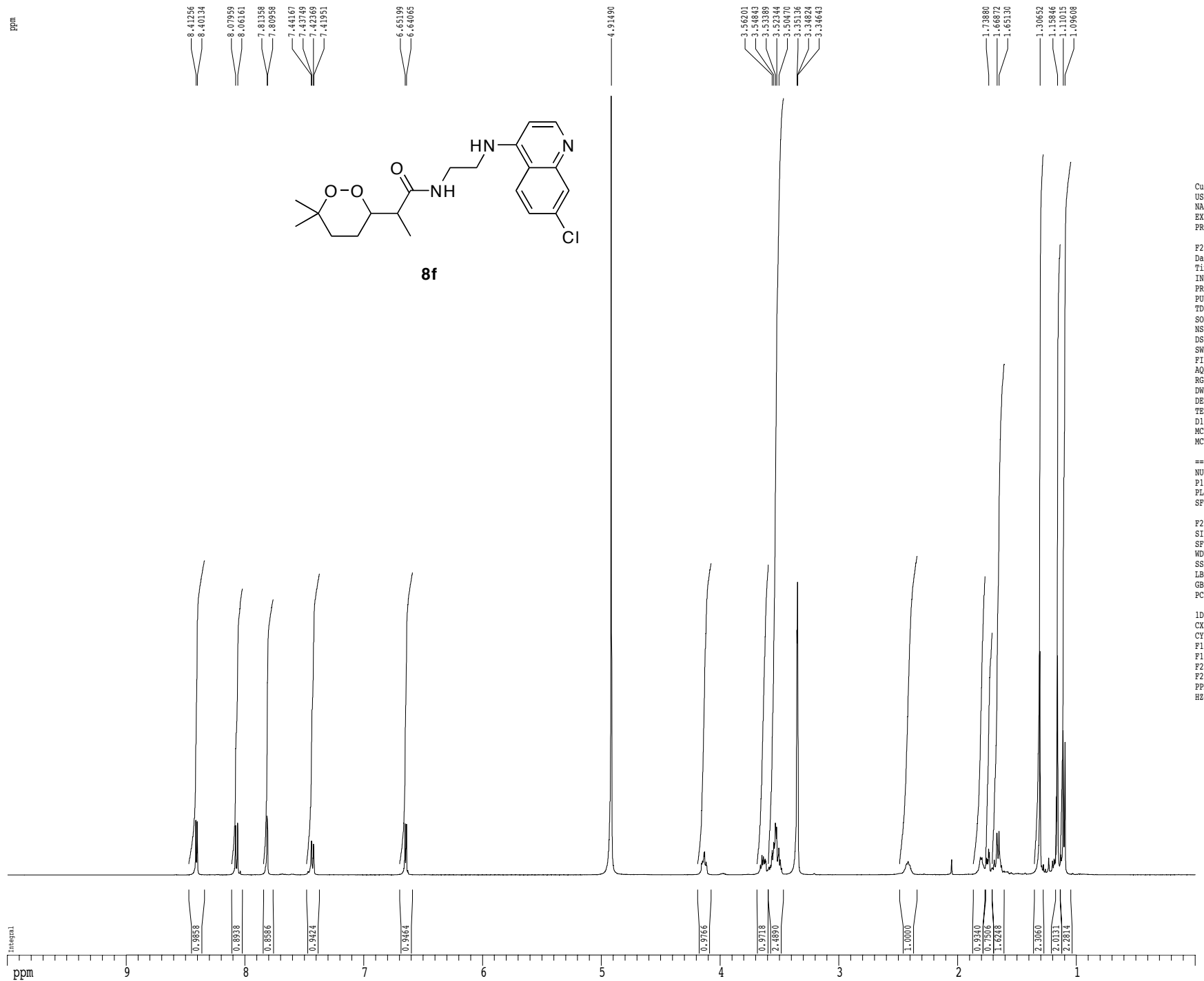
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803983 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 210.000 ppm
F1 26413.88 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 9.64912 ppm/cm
HZCM 1213.67053 Hz/cm

1H spectrum



Current Data Parameters
USER shelli
NAME srw2134
EXPNO 3
PROCNO 1

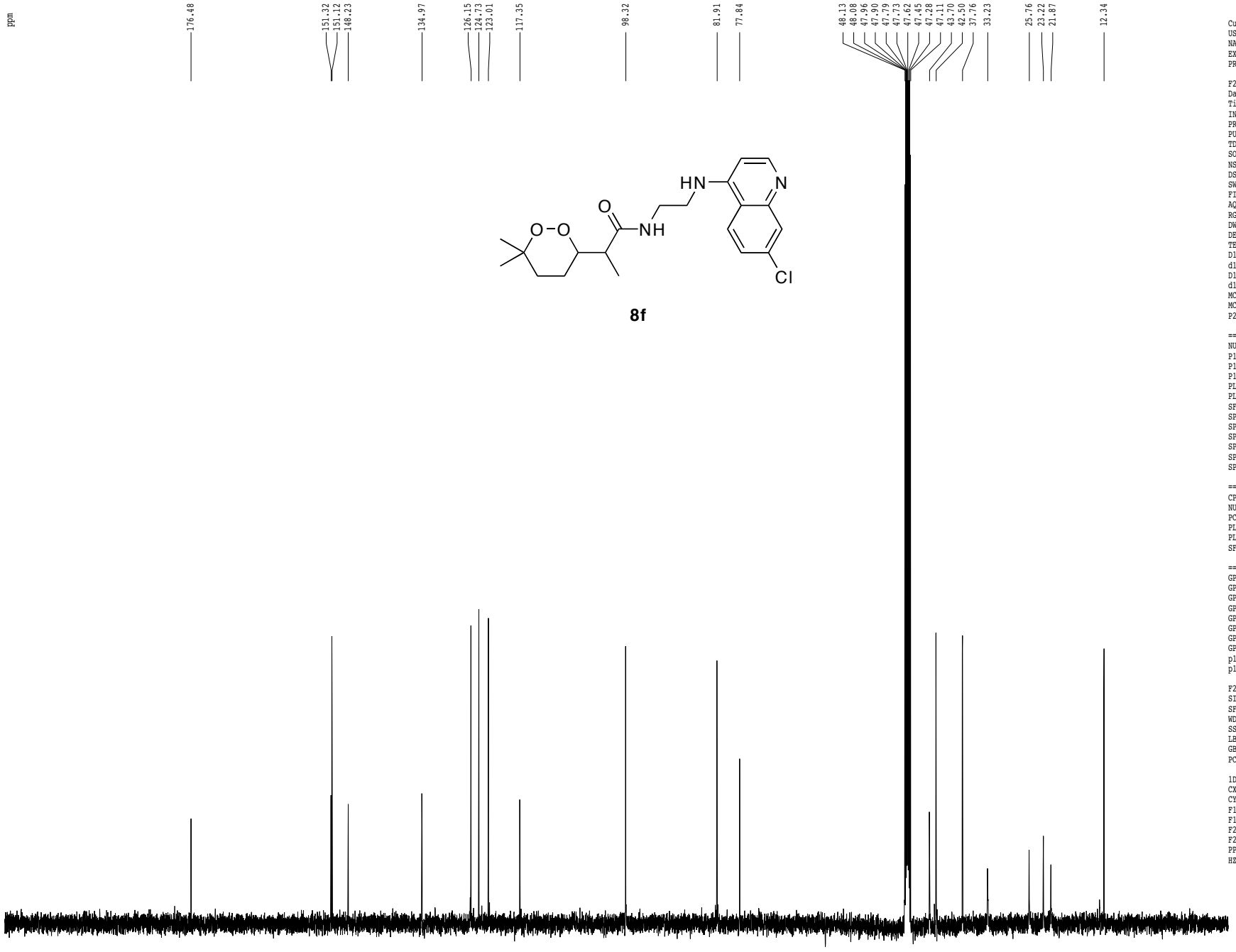
F2 - Acquisition Parameters
Date_ 20090408
Time 15.42
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 5482.456 Hz
FIDRES 0.067082 Hz
AQ 7.4537349 sec
RG 10.1
DW 91.200 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39474 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling




```
Current Data Parameters
USER                shelli
NAME               srw2134
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_            20090408
Time                15.44
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                   65536
SOLVENT              CDCl3
NS                     516
DS                      16
SWH              30303.031 Hz
FIDRES           0.462388 Hz
AQ               1.0814105 sec
RG                  7298.2
DW                  16.500 usec
DE                   6.00 usec
TE                  298.0 K
D1              0.25000000 sec
d11             0.03000000 sec
D16             0.00020000 sec
d17             0.00019600 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec
P2                  29.70 usec

======== CHANNEL f1 ========
NUC1                   13C
P1                  14.85 usec
P11                500.00 usec
P12               2000.00 usec
PL0                120.00 dB
PL1                 -1.00 dB
SFO1          125.7942548 MHz
SP1                  3.60 dB
SP2                  3.60 dB
SPNAM1    Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1               0.00 Hz
SPOFF2               0.00 Hz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                  1.60 dB
PL12                24.60 dB
SFO2          500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPNAM2           SINE.100
GPX1                 0.00 %
GPX2                 0.00 %
GPY1                 0.00 %
GPY2                 0.00 %
GPZ1                30.00 %
GPZ2                50.00 %
p15                500.00 usec
p16               1000.00 usec

F2 - Processing parameters
SI                   65536
SF            125.7804190 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.80 cm
CY                 100.00 cm
F1P               210.000 ppm
F1               26413.89 Hz
F2P               -10.000 ppm
F2               -12557.80 Hz
PPMCM             9.64912 ppm/cm
HZCM           1213.67078 Hz/cm
```