Supporting Information

Enantiospecific Intramolecular Heck Reactions of Secondary Benzylic Ethers

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I. General Procedures

All reactions were carried out under an atmosphere of N_2, or Ar when noted. All glassware was oven- or flame-dried prior to use. Tetrahydrofuran (THF), diethyl ether (Et_2O), dichloromethane (CH_2Cl_2), and toluene (PhMe) were degassed with Ar and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; LaRoche Chemicals; activated under a flow of argon at 350 ºC for 12 h) to remove H_2O. All other solvents utilized were purchased "anhydrous" commercially, or purified as described. ^{1}H NMR spectra were recorded on Bruker DRX-400 (400 MHz ^{1}H, 100 MHz ^{13}C, 376.5 MHz ^{19}F), GN-500 (500 MHz ^{1}H, 125.7 MHz ^{13}C), or CRYO-500 (500 MHz ^{1}H, 125.7 MHz ^{13}C) spectrometers. Proton chemical shifts are reported in ppm (δ) relative to internal tetramethylsilane (TMS, δ 0.00). Data are reported as follows: chemical shift (multiplicity [singlet (s), broad singlet (br s), doublet (d), doublet of doublets (dd), triplet (t), doublet of triplets (dt), doublet of doublet of triplets (ddt), triplet of triplets (tt), quartet (q), quintet (quin), apparent doublet (ad), apparent triplet (at), multiplet (m)], coupling constants [Hz], integration). Carbon chemical shifts are reported in ppm (δ) relative to TMS with the respective solvent resonance as the internal standard ($CDCl_3$, δ 77.16 ppm). Unless otherwise indicated, NMR data were collected at 25 ºC. Infrared (IR) spectra were obtained on a Thermo Scientific Nicolet iS5 spectrometer with an iD5 ATR tip (neat) and are reported in terms of frequency of absorption (cm $^{-1}$). Analytical thin-layer chromatography (TLC) was performed using Silica Gel 60 F_{254} precoated plates (0.25 mm thickness). Visualization was accomplished by irradiation with a UV lamp and/or staining with $KMnO_4$, ceric ammonium molybdate (CAM), or *p*-anisaldehyde (PAA) solutions. Flash chromatography was performed using Silica Gel 60 (170-400 mesh) from Fisher Scientific. Melting points (m.p.) were obtained using a Mel-Temp melting point apparatus and are uncorrected. Optical rotations were measured on a Rudolph Research Analytical Autopol IV Automatic Polarimeter. SFC determinations of enantiopurity were performed on a Berger Analytical instrument using a Daicel™ Chiralpak® column (OD-H, OJ-H, or AD-H; 100 bar, 50 °C, 215 nm). High resolution mass spectrometry was performed by the University of California, Irvine Mass Spectrometry Center.

Bis(tricyclohexylphosphine)nickel(II) chloride was purchased from Strem, stored in a glovebox under an atmosphere of N_2, and used as received. All other reagents were purchased commercially and used as received.

Where noted, silver nitrate impregnated silica gel was used to separate cyclization products from alkene byproducts, which was prepared as follows.[1] To a 1 L round bottom flask was added $AgNO_3$ (15 g) followed by H_2O (5 mL) and CH_3CN (100 mL), with the exclusion of light. The resulting solution was agitated for 15-20 min. Silica gel (100 mL) was added and agitated for 15-20 min. The solvent was removed in vacuo, the silica gel was dried overnight at reduced pressure (~ 1 torr), and then stored in the dark.

II. PREPARATION OF METHYLMAGNESIUM IODIDE

Under an argon atmosphere, dry Et_2O (10 mL) was added to magnesium turnings (1.1 g, 45 mmol) in a 3-neck flask equipped with a reflux condenser and Schlenk filtration apparatus. Iodomethane (1.9 mL, 31 mmol) was then added slowly (over 30 min), so as to maintain a gentle reflux. The mixture was stirred for 2 h at room temperature then filtered through the fritted Schlenk filter into a Schlenk flask under an argon-atmosphere. The Schlenk flask was sealed and removed from the rest of the apparatus.

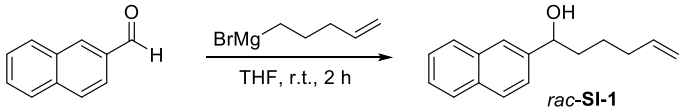
The resulting Grignard reagent titrated with LiCl and iodine and was typically between 2.5 and 3.0 M.[2] The Grignard reagent could be stored (sealed, under argon) for at least 4 weeks without detrimental effects. For satisfactory yields, the Grignard reagent must be prepared from the alkyl iodide in Et_2O at a concentration of at least 2.0 M.

III. SYNTHESIS AND CHARACTERIZATION OF SUBSTRATES FOR TABLE 2

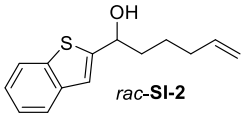
i. Racemic substrates for entries 1–4

Racemic benzylic alcohols were prepared by Grignard addition to the corresponding aldehydes. Alkylation was performed according to the same procedure utilized for enantioenriched substrates (General Procedures G and H).

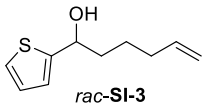
General Procedure A. *Grignard addition to aldehydes.*



***rac*-SI-1.** In a flame-dried round-bottom flask, to a solution of 2-naphthaldehyde (3.12 g, 20.0 mmol, 1.00 equiv) in THF (30 mL) was added at 0 °C pent-4-en-1-ylmagnesium bromide (1.8 M in THF, 17 mL, 30 mmol, 1.5 equiv). After stirring at room temperature for 2 h, saturated ammonium chloride (25 mL) was added at 0 °C and the reaction was extracted with EtOAc (3 x 25 mL). The combined organic layers were washed with brine (1 x 40 mL), dried over $MgSO_4$, and concentrated in vacuo. The product was purified by column flash chromatography (10% EtOAc/hexanes) to afford the title compound as a white solid (4.30 g, 19.0 mmol, 95%). Analytical data is consistent with the values listed for (*R*)-**SI-1** (vide infra).

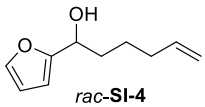


***rac*-SI-2.** Using representative procedure A outlined above, the following amounts of reagents were used: thianaphthene-2-carboxaldehyde (1.05 g, 6.48 mmol, 1.00 equiv), pent-4-en-1-ylmagnesium bromide (1.8 M in THF, 5.4 mL, 9.7 mmol, 1.5 equiv). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a white solid (1.47 g, 6.34 mmol, 98%). Analytical data is consistent with the values listed for (*R*)-**SI-2** (vide infra).



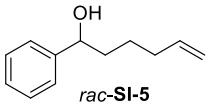
rac-**SI-3**

***rac*-SI-3.** Using representative procedure A outlined above, the following amounts of reagents were used: 2-thiophenecarboxaldehyde (0.56 mL, 6.0 mmol, 1.0 equiv), pent-4-en-1-ylmagnesium bromide (1.8 M in THF, 5.0 mL, 9.0 mmol, 1.5 equiv). The product was purified by flash column chromatography (20% Et_2O/hexanes) to afford the title compound as a clear, colorless oil (1.09 g, 6.00 mmol, quantitative). Analytical data is consistent with the values listed for (*R*)-**SI-3** (vide infra).



rac-**SI-4**

***rac*-SI-4.** Using representative procedure A outlined above, the following amounts of reagents were used: furfural (0.83 mL, 10 mmol, 1.0 equiv), pent-4-en-1-ylmagnesium bromide (1.8 M in THF, 8.3 mL, 15 mmol, 1.5 equiv). The product was purified by flash column chromatography (20% Et_2O/hexanes) to afford the title compound as a clear, colorless oil (1.47 g, 8.85 mmol, 86%). Analytical data is consistent with the values listed for (*R*)-**SI-4** (vide infra).



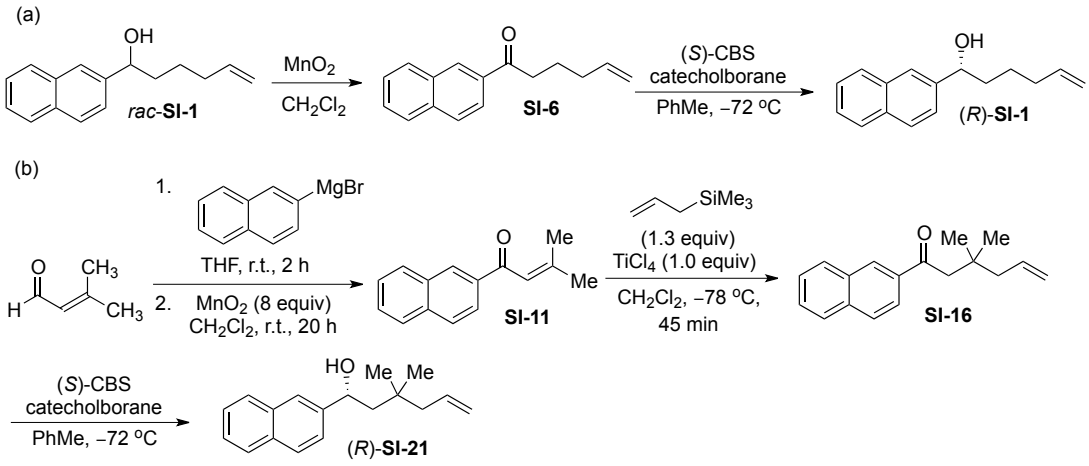
rac-**SI-5**

***rac*-SI-5.** Using representative procedure A outlined above, the following amounts of reagents were used: benzaldehyde (0.71 mL, 7.0 mmol, 1.0 equiv), pent-4-en-1-ylmagnesium bromide (1.8 M in THF, 5.8 mL, 11 mmol, 1.5 equiv). The product was purified by flash column chromatography (15% Et_2O/hexanes) to afford the title compound as a clear, colorless oil (1.14 g, 6.44 mmol, 92%). Analytical data is consistent with the values listed for (*R*)-**SI-5** (vide infra).

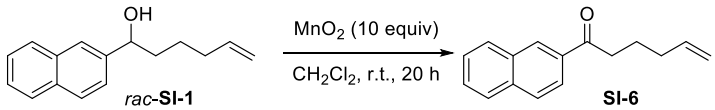
ii. Enantioenriched alcohols for 1–9

Enantioenriched benzylic alcohols were prepared from the racemic benzylic alcohols by oxidation, enantioselective CBS reduction (Table 2, entries 1–4; Scheme **SI-1a**). Others were prepared by Grignard addition into 3-methyl-2-butenal, oxidation, Hosomi–Sakurai allylation and enantioselective CBS reduction (Table 2, entries 5–9; Scheme **SI-1b**).

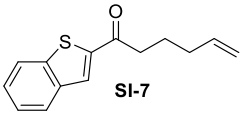
Scheme SI-1. Representative synthetic routes to enantioenriched alcohols



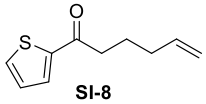
General Procedure B: *Oxidation of benzylic alcohols*



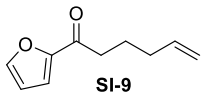
SI-6. The product was prepared according to a modified procedure reported by Wipf.[3] To a solution of ***rac*-SI-1** (1.18 g, 5.20 mmol, 1.00 equiv) in wet CH_2Cl_2 (120 mL) was added in a single portion MnO_2 (4.52 g, 52.0 mmol, 10.0 equiv). The reaction was allowed to stir overnight at room temperature. The resulting slurry was filtered through celite, and the celite was washed with Et_2O. Solvent was removed in vacuo to afford the title compound as a yellow solid (1.08 g, 4.82 mmol, 93%). **TLC R_f** = 0.5 (10% EtOAc/hexanes, UV active); **m.p.** = 36–37 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.44 (s, 1H), 8.02 (dd, *J* = 8.7, 1.7 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.85 (t, *J* = 8.1 Hz 2H), 7.60–7.50 (m, 2H), 5.85 (ddt, *J* = 17.0, 10.2, 3.2 Hz, 1H), 5.07 (ad, *J* = 17.0 Hz, 1H), 5.02 (ad, *J* = 10.2 Hz, 1H), 3.08 (t, *J* = 7.7 Hz, 2H), 2.19 (q, *J* = 7.1 Hz, 2H), 1.90 (quin, *J* = 7.7 Hz 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 200.2, 138.2, 135.6, 134.4, 132.6, 129.7, 129.6, 128.5, 128.4, 127.8, 126.8, 124.0, 115.4, 37.8, 33.3, 23.5; **IR** (neat) 3058, 2829, 1678 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{16}H_{16}OH$ $(M + H)^+$ 225.1279, found 225.1278.



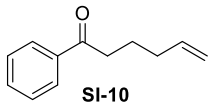
SI-7. Using representative procedure B outlined above, the following amounts of reagents were used: Alcohol ***rac*-SI-2** (0.72 g, 3.1 mmol, 1.0 equiv), MnO_2 (2.68 g, 31.0 mmol, 10.0 equiv), CH_2Cl_2 (40 mL). Further purification after celite plug was unnecessary. The title compound was isolated as a white solid (0.664 g, 2.88 mmol, 93%). **TLC R_f** = 0.6 (10% EtOAc/hexanes, UV active); **m.p.** = 75–76 ^oC; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.87 (t, *J* = 8.7 Hz, 2H), 7.46 (t, *J* = 7.1 Hz, 1H), 7.40 (t, *J* = 7.3 Hz, 1H), 5.83 (ddt, *J* = 16.9, 10.1, 3.4 Hz, 1H), 5.06 (d, *J* = 17.1 Hz, 1H), 5.02 (d, *J* = 10.1 Hz, 1H), 3.01 (t, *J* = 7.3 Hz, 2H), 2.18 (q, *J* = 7.1 Hz, 2H), 1.90 (quin, *J* = 7.2 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 194.9, 144.0, 142.6, 139.3, 138.0, 128.9, 127.5, 126.0, 125.1, 123.1, 115.7, 38.5, 33.3, 23.8; **IR** (neat) 3078, 2892, 1657 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{14}H_{14}OSH$ $(M + H)^+$ 231.0844, found 231.0847.



SI-8. Using representative procedure B outlined above, the following amounts of reagents were used: Alcohol ***rac*-SI-3** (0.73 g, 4.0 mmol, 1.0 equiv), MnO_2 (3.50 g, 40.0 mmol, 10.0 equiv), CH_2Cl_2 (50 mL). Further purification after celite plug was unnecessary. The title compound was isolated as a clear oil (0.69 g, 3.8 mmol, 95%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.71 (dd, *J* = 3.7, 1.0 Hz, 1H), 7.62 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.13 (dd, *J* = 4.9, 3.9 Hz, 1H), 5.82 (ddt, *J* = 17.1, 10.2, 3.3 Hz, 1H), 5.05 (dd, *J* = 17.1, 1.6 Hz, 1H), 5.00 (d, *J* = 10.2 Hz, 1H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.16 (q, *J* = 7.2 Hz, 2H), 1.86 (quin, *J* = 7.3 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 193.3, 144.6, 138.0, 133.5, 131.8, 128.2, 115.5, 38.6, 33.3, 23.8; **IR** (neat) 3076, 2931, 1658 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{10}H_{12}OSH$ $(M + H)^+$ 181.0687, found 181.0693.

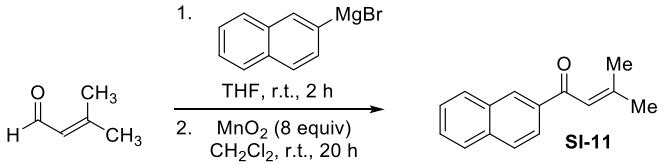


SI-9. Using representative procedure B outlined above, the following amounts of reagents were used: Alcohol ***rac*-SI-4** (0.70 g, 4.2 mmol, 1.0 equiv), MnO_2 (3.7 g, 42 mmol, 10 equiv), CH_2Cl_2 (50 mL). Further purification after celite plug was unnecessary. The title compound was isolated as a clear oil (0.63 g, 3.9 mmol, 92%). **TLC R_f** = 0.6 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.18 (d, *J* = 3.4, Hz, 1H), 6.53 (dd, *J* = 3.4, 1.6 Hz, 1H), 5.81 (ddt, *J* = 17.1, 10.1, 3.4 Hz, 1H), 5.04 (dd, *J* = 17.1, 1.6 Hz, 1H), 5.00 (d, *J* = 10.1 Hz, 1H), 2.83 (t, *J* = 7.6 Hz, 2H), 2.14 (q, *J* = 7.3 Hz, 2H), 1.83 (quin, *J* = 7.3 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 189.7, 152.9, 146.4, 138.0, 117.0, 115.5, 112.3, 37.7, 33.3, 23.4; **IR** (neat) 3076, 2933, 1646, 1569 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{10}H_{12}O_2H$ $(M + H)^+$ 165.0916, found 165.0920.

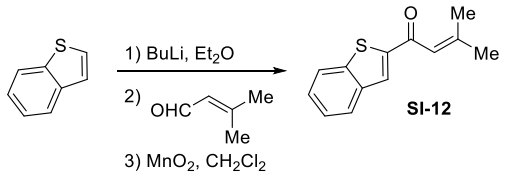


SI-10. Using representative procedure B outlined above, the following amounts of reagents were used: Alcohol **SI-5** (1.67 g, 9.50 mmol, 1.00 equiv), MnO_2 (8.26 g, 95.0 mmol, 10.0 equiv), CH_2Cl_2 (80 mL). Further purification after celite plug was unnecessary. The title compound was isolated as a clear oil (1.57 g, 9.00 mmol, 95%). Analytical data is consistent with literature values.[4] **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.1 Hz, 2H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.46 (t, *J* = 7.8 Hz, 2H), 5.83 (ddt, *J* = 17.0, 10.3, 6.7 Hz, 1H), 5.05 (d, *J* = 17.0 Hz, 1H), 5.00 (d, *J* = 10.3 Hz, 1H), 2.98 (t, *J* = 7.5 Hz, 2H), 2.16 (dd, *J* = 14.4, 7.2, Hz, 2H), 1.86 (tt, *J* = 14.4, 7.2 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 200.4, 138.2, 137.2, 133.1, 128.7, 128.2, 115.4, 37.8, 33.3, 23.4.

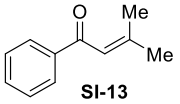
General Procedure C. *Preparation of α,β-unsaturated ketones.*



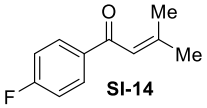
SI-11. In a flame-dried round-bottom flask, to a solution of 3-methyl-2-butenal (1.93 mL, 20.0 mmol, 1.00 equiv) in THF (10 mL) was cooled to 0 °C. 2-Naphthylmagnesium bromide (1.0 M in THF, 30 mL, 30 mmol, 1.5 equiv) was added. After stirring at room temperature for 2 h, saturated ammonium chloride (25 mL) was added at 0 °C and the reaction was extracted with EtOAc (3 x 25 mL). The combined organic layers were washed with brine (1 x 40 mL), dried over $MgSO_4$, and concentrated in vacuo. The unpurified product was redissolved in 125 mL of CH_2Cl_2 in a 250 mL round bottom flask. Solid MnO_2 (13.91 g, 160.0 mmol, 8.000 equiv) was added in a single portion at room temperature. The reaction was allowed to stir open to air for 20 h before it was filtered through celite. The celite was washed with Et_2O and the solvents were removed in vacuo. The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford a thick oil which was further purified by trituration with pentane to afford the title compound as a yellow solid (2.52 g, 12.0 mmol, 60%). **TLC R_f** = 0.4 (10% EtOAc/hexanes, UV active); **m.p.** = 52 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.43 (s, 1H), 8.03 (d, *J* = 8.3 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 7.87 (at, *J* = 9.9 Hz, 2H), 7.57 (t, *J* = 7.5 Hz, 1H), 7.53 (t, *J* = 7.5 Hz, 1H), 6.90 (s, 1H), 2.25 (s, 3H), 2.06 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 191.5, 156.7, 136.7, 135.4, 132.7, 129.60, 129.58, 128.4, 128.2, 127.9, 126.7, 124.5, 121.4, 28.2, 21.4; **IR** (neat) 2057, 2909, 1655, 1610cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{15}H_{14}ONa$ $(M + Na)^+$ 233.0942, found 233.0951.



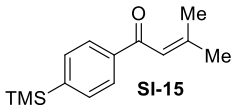
SI-12. Using representative procedure C outlined above, the following amounts of reagents were used: 3-methyl-2-butenal (3.80 mL, 39.4 mmol, 1.06 equiv) in THF (30 mL), 2-lithiobenzothiophene[5] (1.5 M, 25 mL, 37 mmol, 1.0 equiv) CH_2Cl_2 (100 mL), and manganese dioxide (12.4 g, 142 mmol, 3.84 equiv). The product was purified by flash column chromatography (10% EtOAc/ hexanes) to afford the title compound as a yellow solid (5.11 g, 23.6 mmol, 63%). **TLC R_f** = 0.6 (15% EtOAc/hexanes, UV active); **m.p.** = 56–58 ^oC; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.88 (d, *J* = 8.2 Hz, 2H), 7.23 (m, 2H), 6.82 (quintet, *J* = 1.3 Hz, 1H), 2.30 (d, *J* = 1.1 Hz, 3H), 2.07 (d, *J* = 1.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 184.8, 158.5, 146.8, 142.6, 139.7, 127.9, 127.2, 126.0, 125.1, 123.1, 120.2, 28.4, 21.5; **IR** (neat) 3055, 2975, 1644, 1604, 1513, 1257, 1157 cm^{-1}; **HRMS** (TOF MS ES+) *m/z* calcd for $C_{13}H_{12}OSNa$ $(M + Na)^+$ 239.0507, found 239.0513.



SI-13. Using representative procedure C outlined above, the following amounts of reagents were used: 3-methyl-2-butenal (1.93 mL, 20.0 mmol, 1.00 equiv) in THF (15 mL), phenylmagnesium bromide (2.0 M, 15 mL, 30 mmol, 1.5 equiv), CH_2Cl_2 (125 mL), and manganese dioxide (13.91 g, 160.0 mmol, 8.000 equiv). The product was purified by flash column chromatography (5% EtOAc/ hexanes) to afford the title compound as a pale yellow solid (2.02 g, 12.5 mmol, 83%). Analytical data is consistent with literature values.[6] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.2 Hz, 2H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 6.75 (s, 1H), 2.22 (s, 3H), 2.02 (s, 3H).

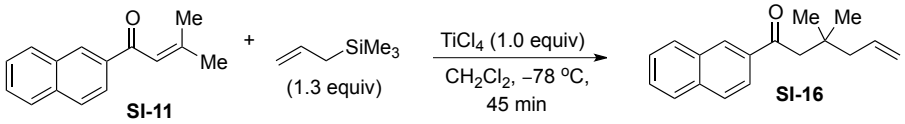


SI-14. Using representative procedure C outlined above, the following amounts of reagents were used: 3-methyl-2-butenal (1.05 mL, 10.5 mmol, 1.00 equiv) in THF (15 mL), 4-fluorophenylmagnesium bromide (1.5 M, 7.0 mL, 10.5 mmol, 1.00 equiv), CH_2Cl_2 (50 mL), and manganese dioxide (4.47 g, 51.4 mmol, 4.90 equiv). The product was purified by flash column chromatography (10% EtOAc/ hexanes) to afford the title compound as a pale yellow oil (1.46 g, 8.19 mmol, 78%). **TLC R_f** = 0.6 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.98–7.92 (m, 2H), 7.11 (t, *J* = 8.7 Hz, 2H), 6.71 (br s, 1H), 2.21 (s, 3H), 2.02 (s, 3H); **^{13}C NMR** ($CDCl_3$, 125 MHz) δ 190.0, 165.3 (d, *J* = 253 Hz), 157.1, 135.6 (d, *J* = 3 Hz), 130.8 (d, *J* = 9 Hz), 120.9 (d, *J* = 1 Hz), 115.5 (d, *J* = 22 Hz), 28.1, 21.2; **IR** (neat) 2977, 1661, 1613, 1597, 1233, 1010, 822 cm-1; [1]; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{11}H_{11}FOH$ $(M + H)^+$ 179.0872, found 179.0876.

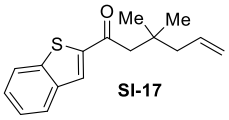


SI-15. Using representative procedure C outlined above, the following amounts of reagents were used: 3-methyl-2-butenal (1.10 mL, 11.4 mmol, 1.19 equiv) in THF (15 mL), 1-bromo-4-trimethylsilylphenylmagnesium bromide (1.4 M, 7.0 mL, 9.6 mmol, 1.0 equiv), CH_2Cl_2 (50 mL), and manganese dioxide (3.40 g, 39.1 mmol, 4.07 equiv). The product was purified by flash column chromatography (10% EtOAc/ hexanes) to afford the title compound as a pale yellow oil (1.33 g, 5.72 mmol, 60%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.9 Hz, 2H), 7.62 (d, *J* = 7.8 Hz, 2H), 6.76 (br s, 1H), 2.23 (s, 3H), 2.03 (s, 3H), 0.3 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 191.8, 156.8, 146.3, 139.6, 133.6, 127.4, 121.5, 28.2, 21.4, -1.1; **IR** (neat) 2955, 1661, 1613, 1246, 821 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{14}H_{20}OSiH$ $(M + H)^+$ 233.1362, found 233.1360.

General Procedure D. *Hosomi–Sakurai reaction of benzylic enones*

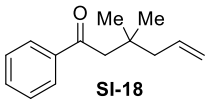


SI-16. The product was prepared according to a modified procedure reported by Coates and co-workers.[7] To a flame-dried 50 mL round bottom flask was added **SI-11** (2.04 g, 9.71 mmol, 1.00 equiv) dissolved in 20 mL CH_2Cl_2. Under an inert atmosphere was added neat titanium tetrachloride (1.20 mL, 10.7 mmol, 1.10 equiv) dropwise over 5 min at –78 °C. The reaction was allowed to stir at this temperature for 10 min before dropwise addition of allyltrimethylsilane (2.05 mL, 12.6 mmol, 1.30 equiv). The reaction was allowed to stir for an additional 5 min at –78 °C before it was placed in a room temperature water bath and stirred for 30 min. The reaction was cooled to 0 °C in an ice water bath and quenched with 2.25 M HCl. The reaction was diluted with Et_2O (80 mL) and washed with saturated $NaHCO_3$ (2 x 25 mL) and brine (1 x 30 mL). The organic layer was dried with $MgSO_4$, filtered, and concentrated in vacuo to afford the title compound as a thick colorless oil (2.43 g, 9.63 mmol, 99%). **TLC R_f** = 0.4 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.43 (s, 1H), 8.01 (d, *J* = 8.2 Hz, 1H), 7.97 (d, *J* = 8.1 Hz, 1H), 7.87 (at, *J* = 7.7 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.55 (t, *J* = 7.2 Hz, 1H), 5.88 (ddt, *J* = 17.2, 10.1, 4.9 Hz, 1H), 5.09–5.02 (m, 2H), 2.99 (s, 2H), 2.22 (d, *J* = 7.3 Hz, 2H), 1.08 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 200.4, 136.1, 135.5, 135.2, 132.6, 129.8, 129.7, 128.48, 128.45, 127.9, 126.8, 124.2, 117.9, 47.9, 46.9, 34.4, 27.7; **IR** (neat) 3060, 2957, 1682, 1596 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{18}H_{20}OH$ $(M + H)^+$ 253.1592, found 253.1582.



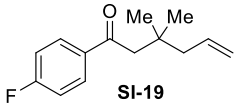
SI-17

SI-17. Using representative procedure D outlined above, the following amounts of reagents were used: **SI-12** (4.52 g, 20.9 mmol, 1.00 equiv), titanium tetrachloride (2.60 mL, 23.7 mmol, 1.13 equiv), allyltrimethylsilane (4.60 mL, 28.9 mmol, 1.38 equiv). The product was purified by flash column chromatography (25% EtOAc/hexanes) to afford the title compound as a yellow oil (1.73 g, 6.69 mmol, 32%). **TLC R_f** = 0.7 (15% EtOAc/hexanes, UV active); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.93–7.84 (m, 3H), 7.44 (m, 2H), 5.89 (quintet, *J* = 17.7, 10.4, 7.5 Hz, 1H), 5.13–5.04 (m, 2H), 2.88 (s, 2H), 2.21 (d, *J* = 7.5 Hz, 2H), 1.10 (s, 6H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ 197.8, 146.0, 142.9, 139.5, 135.1, 129.2, 127.6, 126.2, 125.2, 123.2, 118.2, 49.0, 47.1, 34.7, 27.7 (2C); **IR** (neat) 3072, 2957, 1651, 1514, 1153 cm^{-1}; **HRMS** (TOF MS ES+) *m/z* calcd for $C_{16}H_{18}OSNa$ $(M + Na)^+$ 281.0976, found 281.0974.



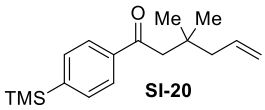
SI-18

SI-18. Using representative procedure D outlined above, the following amounts of reagents were used: **SI-13** (1.98 g, 12.3 mmol, 1.00 equiv), titanium tetrachloride (1.4 mL, 12 mmol, 1.0 equiv), allyltrimethylsilane (2.54 mL, 16.0 mmol, 1.30 equiv). The product was purified by flash column chromatography (1–3% EtOAc/hexanes) to afford the title compound as a colorless oil (1.50 g, 7.40 mmol, 60%). **TLC R_f** = 0.5 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.6 Hz, 2H), 7.54 (t, *J* = 7.5 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 5.85 (ddt, *J* = 17.2, 10.2, 4.2 Hz, 1H), 5.08–5.00 (m, 2H), 2.86 (s, 2H), 2.18 (d, *J* = 7.3 Hz, 2H), 1.05 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 200.4, 138.7, 135.2, 132.8, 128.6, 128.2, 117.8, 47.8, 46.9, 34.2, 27.7; **IR** (neat) 3077, 2957, 1689, 1673 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{14}H_{18}OH$ $(M + H)^+$ 203.1436, found 203.1432.



SI-19

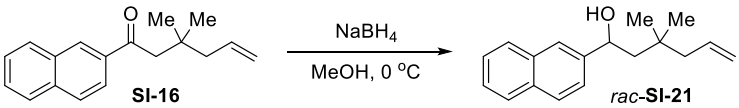
SI-19. Using representative procedure D outlined above, the following amounts of reagents were used: **SI-14** (1.35 g, 7.55 mmol, 1.00 equiv), titanium tetrachloride (0.91 mL, 8.8 mmol, 1.2 equiv), allyltrimethylsilane (1.60 mL, 10.1 mmol, 1.34 equiv). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (1.21 g, 5.49 mmol, 73%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.99–7.92 (m, 2H), 7.15–7.07 (m, 2H), 5.85 (ddt, *J* = 17.7, 10.2, 7.5 Hz, 1H), 5.09–5.00 (m, 2H), 2.83 (s, 2H), 2.18 (d, *J* = 7.6 Hz, 2H), 1.05 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 198.8, 165.7 (d, *J* = 254 Hz), 135.22 (d, *J* = 3 Hz), 135.18, 130.9 (d, *J* = 9 Hz), 118.0, 115.7 (d, *J* = 22 Hz), 47.8, 47.0, 34.3, 27.7; **IR** (neat) 2958, 1673, 1596, 1225, 1155 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{14}H_{17}FOH$ $(M + H)^+$ 221.1342, found 221.1341.



SI-20

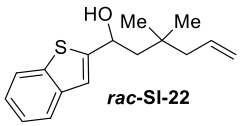
SI-20. Using representative procedure D outlined above, the following amounts of reagents were used: **SI-15** (1.23 g, 5.28 mmol, 1.00 equiv), titanium tetrachloride (0.65 mL, 5.9 mmol, 1.1 equiv), allyltrimethylsilane (1.10 mL, 6.92 mmol, 1.31 equiv). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a colorless oil (1.13 g, 4.12 mmol, 78%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (ad, *J* = 8.4 Hz, 2H), 7.61 (ad, *J* = 8.4 Hz, 2H), 5.86 (ddt, *J* = 18.2, 10.3, 7.4 Hz, 1H), 5.10–5.01 (m, 2H), 2.86 (s, 2H), 2.19 (d, *J* = 7.6 Hz, 2H), 1.06 (s, 6H), 0.30 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 200.7, 146.9, 138.9, 135.3, 133.7, 127.3, 117.9, 47.9, 47.0, 34.3, 27.8, -1.1; **IR** (neat) 2955, 1687, 1387, 1224, 835 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{17}H_{26}OSiH$ $(M + H)^+$ 275.1831, found 275.1837.

General Procedure E. *Reduction of benzylic ketones with $NaBH_4$*



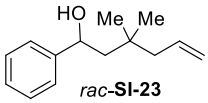
SI-16 *rac*-**SI-21**

***rac*-SI-21.** The product was prepared according to a modified procedure reported by Wang and Franzén.[8] A round bottom flask containing **SI-16** (1.51 g, 6.00 mmol, 1.00 equiv) dissolved in MeOH (10 mL) was cooled to 0 °C in an ice water bath. Sodium borohydride (0.36 g, 9.6 mmol, 1.6 equiv) was added in a single portion and the reaction was stirred for 30 min at 0 °C. The reaction was warmed to room temperature and stirred for an additional 1 h, after which time it was quenched with water. The reaction was extracted with Et_2O (3 x 25 mL) and washed with brine (1 x 40 mL). The combined organic layers were dried with $MgSO_4$, filtered and dried in vacuo. The residue was purified by flash column chromatography (5–10% EtOAc/hexanes) to afford the title compound as a thick, colorless oil (1.25 g, 4.93 mmol, 82%). The analytical data is consistent with the values listed for (*R*)-**SI-21** (vide infra).

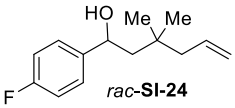


rac-**SI-22**

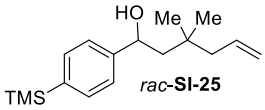
***rac*-SI-22.** Using representative procedure E outlined above, the following amounts of reagents were used: **SI-17** (0.67 g, 2.6 mmol, 1.0 equiv), MeOH (10 mL), sodium borohydride (0.15 g, 4.0 mmol, 1.5 equiv). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a pale yellow oil (0.58 g, 2.2 mmol, 85%). The analytical data is consistent with the values listed for (*R*)-**SI-22** (vide infra).



***rac*-SI-23.** Using representative procedure E outlined above, the following amounts of reagents were used: **SI-18** (0.73 g, 3.6 mmol, 1.0 equiv), MeOH (8 mL), sodium borohydride (0.33 g, 8.7 mmol, 2.4 equiv). The product was purified by flash column chromatography (8–15% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (0.64 g, 3.1 mmol, 86%). The analytical data is consistent with the values listed for (*R*)-**SI-23** (vide infra).

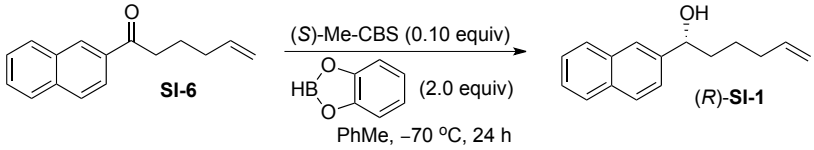


***rac*-SI-24.** Using representative procedure E outlined above, the following amounts of reagents were used: **SI-19** (0.50 g, 2.3 mmol, 1.0 equiv), MeOH (10 mL), sodium borohydride (0.30 g, 7.9 mmol, 3.4 equiv). The product was purified by flash column chromatography (3–10% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (0.37 g, 1.7 mmol, 73%). The analytical data is consistent with the values listed for (*R*)-**SI-24** (vide infra).



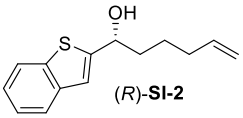
***rac*-SI-25.** Using representative procedure E outlined above, the following amounts of reagents were used: **SI-20** (0.49 g, 1.8 mmol, 1.0 equiv), MeOH (10 mL), sodium borohydride (0.33 g, 8.7 mmol, 4.8 equiv). The product was purified by flash column chromatography (3–10% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (0.35 g, 1.3 mmol, 86%). The analytical data is consistent with the values listed for (*R*)-**SI-25** (vide infra).

General Procedure F. *Enantioselective reduction of benzylic ketones*



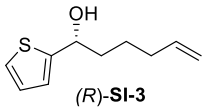
(*R*)-SI-1. The product was prepared according to a modified procedure reported by Okamura.[9] In a glovebox, (*S*)-Me-CBS (103 mg, 0.454 mmol, 0.100 equiv) was added to a flame-dried round bottom flask equipped with a stir bar. The flask was capped with a septum and removed from the box. Ketone **SI-6** (1.02 g, 4.54 mmol, 1.00 equiv) was added to the flask as a solution in PhMe (20 mL). The reaction was then cooled to –70 °C and catecholborane (0.97 mL, 9.1 mmol, 2.0 equiv) was added dropwise via syringe. After stirring for 24 h at –70 °C, the reaction was warmed to ambient temperature and quenched with water. Sat. NH_4Cl was added to the reaction flask and the mixture was extracted with EtOAc (3 x 30 mL). The combined organics were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The product was purified by flash column chromatography (8–12% EtOAc/hexanes) to afford the title compound as a white solid (0.95 g, 4.3 mmol, 94%, 93% ee). The solid was recrystallized from hexanes to improve enantiomeric excess (99% ee). **TLC R_f** = 0.2 (10% EtOAc/hexanes, UV active); **m.p.** = 58–60 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.88–7.80 (m, 3H), 7.78 (s, 1H), 7.52–7.43 (m, 3H), 5.78 (ddt, *J* = 17.3, 10.1, 3.3 Hz, 1H), 4.99 (dd, *J* = 17.1, 1.3 Hz, 1H), 4.94 (d, *J* = 10.3 Hz, 1H), 4.89–4.82 (m, 1H), 2.09 (q, *J* = 7.1 Hz, 2H), 1.96–1.77 (m, 3H), 1.62–1.50 (m, 1H), 1.46–1.36 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 142.3, 138.7, 133.4, 133.1, 128.5, 128.1, 127.8, 126.3, 126.0, 124.7, 124.2, 114.9, 74.8, 38.5, 33.7, 25.2; **IR** (neat) 3279, 3056, 2932 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{16}H_{18}ONH_4$ $(M + NH_4)^+$ 244.1701, found 244.1701; **$[\alpha]^{26}_D$** +36 (*c* 3.6, $CHCl_3$); **SFC** analysis (OD-H, 8% IPA, 2.5 mL/min) indicated 99% ee: t_R (major) = 7.2 minutes, t_R (minor) = 6.5 minutes.

E.J. Corey's model for stereoselectivity of CBS reductions was used to assign the absolute configuration of benzylic alcohols prepared from this method. [10]



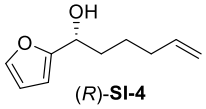
(*R*)-SI-2. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-7** (0.64 g, 2.8 mmol, 1.0 equiv), (*S*)-Me-CBS (63 mg, 0.23 mmol, 0.10 equiv), catecholborane (0.59 mL, 5.5 mmol, 2.0 equiv), and PhMe (20 mL). After extraction with Et_2O, the combined organics were removed in vacuo, with much care taken to avoid heating. Heating the mixture under vacuum prior to column chromatography causes irreversible complexation of the desired product with boron reagents present from the reaction. The remaining solvent (mostly PhMe) was removed by running the mixture through a plug of silica. Flash column chromatography (8–15% EtOAc/hexanes) afforded the title compound as a white solid (0.39 g, 1.7 mmol, 60%, 93% ee). The solid was recrystallized from hexanes to improve enantiomeric excess (98% ee). **TLC R_f** = 0.2 (10% EtOAc/hexanes, UV active); **m.p.** = 34–35 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.8 Hz, 1H), 7.72 (d, *J* = 8.2 Hz, 1H), 7.39–7.28 (m, 2H), 7.19 (s, 1H), 5.80 (ddt, *J* = 17.3, 10.1, 3.2 Hz, 1H), 5.05–4.94 (m, 3H), 2.12 (q, *J* = 7.0 Hz, 2H), 2.08–2.05 (m, 1H), 1.99–1.85 (m, 2H), 1.64–1.56 (m, 1H) 1.53–1.42 (m, 1H); **^{13}C**

NMR (125 MHz, $CDCl_3$) δ 149.5, 139.6, 139.4, 138.5, 124.4, 124.3, 123.6, 122.7, 120.3, 115.1, 71.0, 38.5, 33.6, 25.0; **IR** (neat) 3289, 3074, 2932 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{14}H_{16}OSNH_4$ $(M + NH_4)^+$ 250.1266, found 250.1260; $[\alpha]^{27}_D$ +19 (*c* 0.3, $CHCl_3$); **SFC** analysis (OD-H, 15% IPA, 2.5 mL/min) indicated 98% ee: t_R (major) = 7.2 minutes, t_R (minor) = 6.6 minutes.



(*R*)-**SI-3**

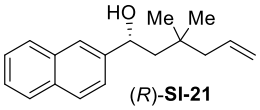
(*R*)-SI-3. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-8** (0.45 g, 2.5 mmol, 1.0 equiv), (*S*)-Me-CBS (57 mg, 0.25 mmol, 0.10 equiv), catecholborane (0.53 mL, 5.0 mmol, 2.0 equiv), and PhMe (15 mL). After extraction with Et_2O, the combined organics were removed in vacuo, with much care taken to avoid heating. Heating the mixture under vacuum prior to column chromatography causes irreversible complexation of the desired product with boron reagents present from the reaction. The remaining solvent (mostly PhMe) was removed by running the mixture through a plug of silica. Flash column chromatography (10% EtOAc/hexanes) afforded the title compound as a clear oil (0.40 g, 2.2 mmol, 88%, 96% ee). **TLC R_f** = 0.3 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.24 (dd, *J* = 4.3, 1.6 Hz, 1H), 6.99–6.94 (m, 2H), 5.79 (ddt, *J* = 17.3, 10.1, 3.3 Hz, 1H), 5.01 (add, *J* = 17.1, 1.5 Hz, 1H), 4.96 (d, *J* = 10.3, Hz, 1H), 4.92 (dd, *J* = 6.9, 3.9 Hz, 1H), 2.10 (q, *J* = 7.1 Hz, 2H), 2.05–2.00 (m, 1H), 1.94–1.78 (m, 2H), 1.60–1.51 (m, 1H), 1.48–1.38 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 148.9, 138.6, 126.7, 124.7, 123.9, 115.0, 70.3, 38.8, 33.6, 25.2; **IR** (neat) 3342, 3074, 2934, 2859 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{10}H_{14}SOH$ $(M + H)^+$ 183.0844, found 183.0844; $[\alpha]^{26}_D$ +21 (*c* 1.0, $CHCl_3$); **SFC** analysis (OJ-H, 3% IPA, 2.5 mL/min) indicated 96% ee: t_R (major) = 6.0 minutes, t_R (minor) = 5.1 minutes.



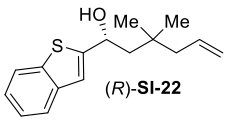
(*R*)-**SI-4**

(*R*)-SI-4. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-9** (0.52 g, 2.8 mmol, 1.0 equiv), (*S*)-Me-CBS (64 mg, 0.28 mmol, 0.10 equiv), catecholborane (0.60 mL, 5.6 mmol, 2.0 equiv), and PhMe (20 mL). After extraction with Et_2O, the combined organics were removed in vacuo, with much care taken to avoid heating. Heating the mixture under vacuum prior to column chromatography causes irreversible complexation of the desired product with boron reagents present from the reaction. The remaining solvent (mostly PhMe) was removed by running the mixture through a plug of silica. Flash column chromatography (10% Et_2O/hexanes) afforded the title compound as a clear oil (0.37 g, 2.2 mmol, 80%). Enantiomeric excess could not be determined for the title compound using SFC and chiral GC instrumentation. **TLC R_f** = 0.3 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37 (s, 1H), 6.33 (dd, *J* = 1.7, 1.1 Hz, 1H), 6.23 (d, *J* = 2.9 Hz, 1H), 5.80 (ddt, *J* = 17.1, 10.2, 3.3 Hz, 1H), 5.01 (add, *J* = 17.1, 1.5 Hz, 1H), 4.96 (d, *J* = 10.2, Hz, 1H), 4.68 (t, *J* = 7.0 Hz, 1H), 2.10 (q, *J* = 7.0 Hz, 2H), 1.94 (br s, 1H), 1.90–1.83 (m, 2H), 1.55 (sep, *J* = 7.2 Hz, 1H), 1.42 (sep, *J* = 7.3 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.9, 142.0, 138.6, 114.9, 110.3, 106.0, 67.8, 35.1, 33.6, 24.9; **IR** (neat) 3348, 3077,

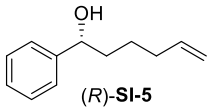
2932, 2861 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{10}H_{14}O_2NH_4$ $(M + NH_4)^+$ 184.1338, found 184.1329; $[\alpha]^{24}_D$ +14 (*c* 1.0, $CHCl_3$).



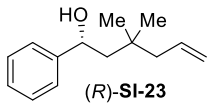
(*R*)-SI-21. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-16** (0.698 g, 2.77 mmol, 1.00 equiv), (*S*)-Me-CBS (63 mg, 0.28 mmol, 0.10 equiv), catecholborane (0.59 mL, 5.5 mmol, 2.0 equiv), and PhMe (20 mL). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a clear oil (0.295 g, 1.16 mmol, 42%, 88% ee). **TLC R_f** = 0.6 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.80 (d, *J* = 8.4 Hz, 3H), 7.75 (s, 1H), 7.49–7.41 (m, 3H), 5.85 (ddt, *J* = 17.1, 10.1, 4.8 Hz, 1H), 5.07–4.97 (m, 3H), 2.15–2.04 (m, 2H), 1.90–1.85 (br s, 1H), 1.82 (dd, *J* = 14.7, 8.7 Hz, 1H), 1.66 (dd, *J* = 14.6, 3.3 Hz, 1H), 1.02 (s, 3H), 0.98 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.0, 135.7, 133.5, 133.0, 128.5, 128.0, 127.8, 126.3, 125.9, 124.27, 124.25, 117.3, 72.3, 50.7, 47.5, 33.5, 27.9, 27.8; **IR** (neat) 3392, 3057, 2956, 1601 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{18}H_{22}ONH_4$ $(M + NH_4)^+$ 272.2014, found 272.2009; $[\alpha]^{23}_D$ +112 (*c* 0.5, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 2.5 mL/min) indicated 88% ee: t_R (major) = 11.5 minutes, t_R (minor) = 10.1 minutes.



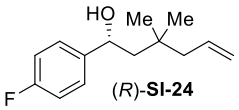
(*R*)-SI-22. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-17** (0.500 g, 1.94 mmol, 1.00 equiv), (*S*)-Me-CBS (57 mg, 0.25 mmol, 0.13 equiv), catecholborane (0.40 mL, 3.8 mmol, 2.0 equiv), and PhMe (20 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a clear oil (0.280 g, 1.02 mmol, 56%, 85% ee). **TLC R_f** = 0.5 (15% EtOAc/hexanes, UV active); **m.p**. = 54–56 °C; **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.84–7.80 (m, 1H), 7.74–7.70 (m, 1H), 7.33 (tdd, *J* = 14.6, 7.1, 1.4 Hz, 2H), 7.18 (s, 1H), 5.88 (, *J* = 16.8, 10.4, 7.4 Hz, 2H), 5.19 (dd, *J* = 7.7, 3.0 Hz, 1H), 5.11–5.03 (m, 2H), 2.13 (ddt, *J* = 7.4, 3.4, 1.3 Hz, 2H), 2.04 (s, 1H), 1.89 (d, *J* = 8.2, 1H), 1.85 (d, *J* = 3.8 Hz, 1H), 1.05 (s, 3H), 1.02 (s, 3H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ 151.5, 139.7, 139.5, 135.6, 124.5, 124.4, 123.7, 122.7, 119.7, 117.6, 68.7, 50.8, 47.5, 33.6, 27.8, 27.7; **IR** (neat) 3403, 3071, 2956, 1458, 1436, 1366, 913 cm^{-1}; **HRMS** (TOF MS ES+) *m/z* calcd for $C_{16}H_{20}OSNa$ $(M + Na)^+$ 283.1133, found 283.1122. $[\alpha]^{30}_D$ +25 (*c* 1.1, $CHCl_3$); **SFC** analysis (OD-H, 15% IPA, 2.5 mL/min) indicated 85% ee: tR (major) = 6.3 minutes, t_R (minor) = 5.8 minutes.



(*R*)-SI-5. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-10** (0.435 g, 2.50 mmol, 1.00 equiv), (*S*)-Me-CBS (57 mg, 0.25 mmol, 0.10 equiv), catecholborane (0.53 mL, 5.0 mmol, 2.0 equiv), and PhMe (15 mL). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a clear oil (0.396 g, 2.26 mmol, 90%). Analytical data is consistent with literature values.[11] **TLC R_f** = 0.3 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.38–7.31 (m, 4H), 7.30–7.25 (m, 1H), 5.78 (ddt, *J* = 17.0, 10.3, 6.6 Hz, 1H), 4.99 (d, *J* = 17.0 Hz, 1H), 4.94 (d, *J* = 10.3 Hz, 1H), 4.70–4.64 (m, 1H), 2.08 (dd, *J* = 14.2, 7.1 Hz, 2H), 1.86–1.76 (m, 2H), 1.76–1.68 (m, 1H), 1.57–1.48 (m, 1H), 1.43–1.33 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.9, 138.7, 128.6, 127.7, 126.0, 114.8, 74.7, 38.6, 33.7, 25.2; **$[\alpha]^{23}_D$** +38 (*c* 1.1, $CHCl_3$).



(*R*)-SI-23. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-18** (0.42 g, 2.1 mmol, 1.0 equiv), (*S*)-Me-CBS (47 mg, 0.21 mmol, 0.10 equiv), catecholborane (0.44 mL, 4.1 mmol, 2.0 equiv), and PhMe (20 mL). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a clear oil (0.207 g, 1.01 mmol, 50%, 92% ee). Analytical data is consistent with literature values.[12] **TLC R_f** = 0.4 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 4.3 Hz, 4H), 7.29–7.23 (m, 1H), 5.85 (ddt, *J* = 17.2, 10.1, 4.9 Hz, 1H), 5.08–4.99 (m, 2H), 4.85 (dt, *J* = 8.6, 3.2 Hz, 1H), 2.13–2.03 (m, 2H), 1.80–1.72 (m, 2H), 1.59 (dd, *J* = 14.7, 3.3 Hz, 1H), 1.00 (s, 3H), 0.97 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.6, 135.7, 128.7, 127.5, 125.8, 117.3, 72.3, 50.9, 47.4, 33.5, 27.82, 27.79; **IR** (neat) 3392, 2956, 1366 cm^{-1}; **$[\alpha]^{27}_D$** +55 (*c* 1.2, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 2.5 mL/min) indicated 92% ee: t_R (major) = 1.8 minutes, t_R (minor) = 2.1 minutes.



(*R*)-**SI-24**

(*R*)-SI-24. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-19** (0.632 g, 2.87 mmol, 1.00 equiv), (*S*)-Me-CBS (90 mg, 0.39 mmol, 0.17 equiv), catecholborane (0.62 mL, 5.8 mmol, 2.5 equiv), and PhMe (20 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a clear oil (0.466 g, 2.10 mmol, 73%, 90% ee). **TLC R_f** = 0.3 (5% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.45–7.37 (m, 2H), 7.13 (at, *J* = 8.8 Hz, 2H), 5.97 (ddt, *J* = 17.6, 10.3, 7.4 Hz, 1H), 5.22–5.10 (m, 2H), 4.93 (dd, *J* = 8.5, 3.5 Hz, 1H), 2.19 (add, *J* = 7.4, 3.9 Hz, 1H), 2.15 (s, 1H), 1.85 (dd, *J* = 14.7, 8.6 Hz, 1H), 1.71 (dd, *J* = 14.7, 3.5 Hz, 1H), 1.11 (s, 3H), 1.08 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 162.2 (d, *J* = 245 Hz), 142.5 (d, *J* = 3 Hz), 135.7, 127.6 (d, *J* = 8 Hz), 117.4, 115.5 (d, *J* = 21 Hz), 71.6, 51.0, 47.5, 33.5, 27.91, 27.88; **IR** (neat) 3394, 2957, 1638, 1604, 1508, 1155 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{14}H_{19}FOH$ $(M + H)^+$ 223.1498, found 223.1499; **$[\alpha]^{27}_D$** +45 (*c* 2.0, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 90% ee: t_R (major) = 9.5 minutes, t_R (minor) = 10.3 minutes.

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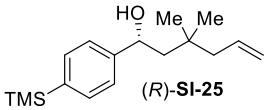
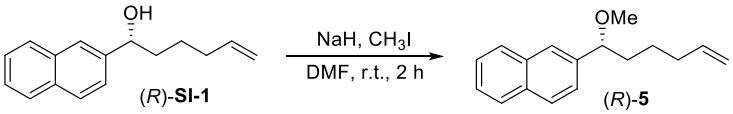


(*R*)-**SI-25**

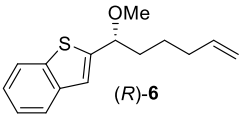
(*R*)-SI-25. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-20** (0.612 g, 2.25 mmol, 1.00 equiv), (*S*)-Me-CBS (63 mg, 0.28 mmol, 0.12 equiv), catecholborane (0.50 mL, 4.5 mmol, 2.0 equiv), and PhMe (20 mL). The product was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a clear oil (0.283 g, 1.02 mmol, 46%, 92% ee). **TLC R_f** = 0.3 (5% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 7.7 Hz, 2H), 5.84 (ddt, *J* = 17.6, 10.2, 7.5 Hz, 1H), 5.06–4.98 (m, 2H), 4.85–4.77 (m, 1H), 2.13–2.03 (m, 2H), 1.79–1.70 (m, 1H), 1.73 (ad, *J* = 8.8 Hz, 1H), 1.57 (dd, *J* = 14.8, 2.8 Hz, 1H), 1.00 (s, 3H), 0.97 (s, 3H), 0.25 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$ δ 147.2, 139.5, 135.7, 133.7, 125.1, 117.2, 72.2, 50.8, 47.4, 33.4, 27.8, 27.7, -1.0; **IR** (neat) 3388, 2955, 1638, 1600, 1386, 1248, 912 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{17}H_{28}OSiH$ $(M + H)^+$ 277.1988, found 277.1990; **$[\alpha]^{27}_D$** +38 (*c* 1.4, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 92% ee: t_R (major) = 1.9 minutes, t_R (minor) = 2.3 minutes.

iii. Alkylations of enantioenriched alcohols for entries 1–9

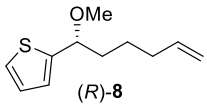
General Procedure G. *Methylation of benzylic alcohols.*



(*R*)-5. In a glovebox, NaH (42 mg, 1.8 mmol, 1.6 equiv) was added to a flame-dried round bottom flask equipped with a stir bar. The flask was removed from the glovebox, and anhydrous DMF (2 mL) was added. To this slurry was added a solution of alcohol (*R*)-**SI-1** (0.244 g, 1.10 mmol, 1.00 equiv) in DMF (4 mL) at 0 °C. The solution was warmed to room temperature over 30 min, then cooled to 0 °C and neat methyl iodide (0.14 mL, 2.2 mmol, 2.0 equiv) was added. The reaction was warmed to ambient temperature and stirred an additional 1.5 h. The reaction was quenched at 0 °C with 1M HCl and extracted with EtOAc (3 x 15 mL). The combined organic extracts were washed with water (1 x 50 mL) and brine (1 x 50 mL), dried over $MgSO_4$, and concentrated in vacuo. The product was purified by flash column chromatography (2–7% Et_2O/hexanes) to afford the title compound as a colorless oil (0.210 g, 0.890 mmol, 81%, 99% ee). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.88–7.80 (m, 3H), 7.70 (s, 1H), 7.51–7.41 (m, 3H), 5.76 (ddt, *J* = 17.1, 10.2, 3.2 Hz, 1H), 4.97 (ad, *J* = 17.1, Hz, 1H), 4.92 (d, *J* = 10.2 Hz, 1H), 4.25 (t, *J* = 6.9 Hz, 1H), 3.24 (s, 3H) 2.06 (q, *J* = 7.1 Hz, 2H), 1.95–1.86 (m, 1H), 1.77–1.68 (m, 1H), 1.58–1.48 (m, 1H) 1.41–1.31 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 139.9, 138.8, 133.3, 133.2, 128.5, 128.0, 127.9, 126.2, 126.0, 125.9, 124.6, 114.8, 84.2, 56.9, 37.6, 33.8, 25.3; **IR** (neat) 3057, 2859, 1601, 1098 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{17}H_{20}O$ $(M)^+$ 240.1514, found 240.1523; **$[\alpha]^{25}_D$** +82 (*c* 1.5, $CHCl_3$); **SFC** analysis (OD-H, 4% IPA, 2.5 mL/min) indicated 99% ee: t_R (major) = 5.3 minutes, t_R (minor) = 5.0 minutes.

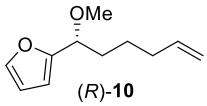


(*R*)-6. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**SI-2** (76 mg, 0.33 mmol, 1.0 equiv), NaH (13 mg, 0.52 mmol, 1.6 equiv), methyl iodide (0.030 mL, 0.42 mmol, 1.3 equiv) and DMF (4 mL). The product was purified by flash column chromatography (2–10% EtOAc/hexanes) to afford the title compound as a colorless oil (52 mg, 0.21 mmol, 64%, 97% ee). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.0 Hz, 1H), 7.72 (d, *J* = 7.3 Hz, 1H), 7.33 (at, *J* = 7.5 Hz, 1H), 7.29 (at, *J* = 7.3 Hz, 1H), 7.18 (s, 1H), 5.77 (ddt, *J* = 17.1, 10.3, 3.3 Hz, 1H), 4.99 (ad, *J* = 17.1 Hz, 1H), 4.94 (ad, *J* = 10.2 Hz, 1H), 4.43 (t, *J* = 6.7 Hz, 1H), 3.31 (s, 3H), 2.07 (q, *J* = 7.0 Hz, 2H), 2.01–1.91 (m, 1H), 1.84–1.75 (m, 1H), 1.59–1.49 (m, 1H) 1.46–1.35 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 147.4, 139.8, 139.5, 138.6, 124.30, 124.26, 123.4, 122.7, 122.0, 114.9, 80.1, 56.9, 37.5, 33.6, 25.1; **IR** (neat) 3060, 2928, 2820, 1458 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{15}H_{18}OS$ $(M)^+$ 246.1078, found 246.1075; **$[\alpha]^{24}_D$** +60 (*c* 3.0, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 97% ee: t_R (major) = 3.6 minutes, t_R (minor) = 3.2 minutes.



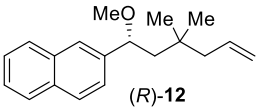
(*R*)-**8**

(*R*)-8. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**SI-3** (0.219 g, 1.20 mmol, 1.00 equiv), NaH (46 mg, 1.9 mmol, 1.6 equiv), methyl iodide (0.15 mL, 2.4 mmol, 2.0 equiv) and DMF (6 mL). The product was purified by flash column chromatography (2–10% EtOAc/hexanes) to afford the title compound as a colorless oil (0.178 g, 0.910 mmol, 76%, 97% ee). **TLC R_f** = 0.8 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.27 (d, *J* = 5.1 Hz, 1H), 6.98–6.94 (m, 2H), 5.79 (ddt, *J* = 17.1, 10.4, 3.3 Hz, 1H), 4.99 (adq, *J* = 17.1, 1.5 Hz, 1H), 4.94 (d, *J* = 10.2, Hz, 1H), 4.36 (t, *J* = 7.0 Hz, 1H), 3.25 (s, 3H), 2.06 (q, *J* = 7.2 Hz, 2H), 1.97–1.88 (m, 1H), 1.79–1.70 (m, 1H), 1.56–1.46 (m, 1H), 1.43–1.32 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 146.3, 138.7, 126.4, 125.4, 125.0, 114.8, 79.4, 56.6, 37.9, 33.6, 25.2; **IR** (neat) 3075, 2935, 1440 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{11}H_{16}OS$ $(M)^+$ 196.0922, found 196.0932; **$[\alpha]^{27}_D$** +70 (*c* 1.0, $CHCl_3$); **SFC** analysis (OD-H, 1% IPA, 2.5 mL/min) indicated 97% ee: t_R (major) = 3.4 minutes, t_R (minor) = 3.6 minutes.

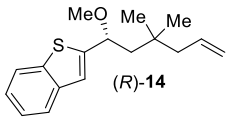


(*R*)-**10**

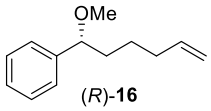
(*R*)-10. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**SI-4** (0.20 g, 1.2 mmol, 1.0 equiv), NaH (46 mg, 1.9 mmol, 1.6 equiv), methyl iodide (0.15 mL, 2.4 mmol, 2.0 equiv) and DMF (6 mL). The product was purified by flash column chromatography (2–10% EtOAc/hexanes) to afford the title compound as a colorless oil (0.168 g, 0.936 mmol, 78%, 93% ee). **TLC R_f** = 0.8 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.39 (s, 1H), 6.34 (dd, *J* = 2.8, 1.7 Hz, 1H), 6.26 (d, *J* = 3.1 Hz, 1H), 5.78 (ddt, *J* = 17.1, 10.3, 3.1 Hz, 1H), 4.99 (adq, *J* = 17.1, 1.5 Hz, 1H), 4.94 (d, *J* = 10.2, Hz, 1H), 4.16 (t, *J* = 7.0 Hz, 1H), 3.25, (s, 3H), 2.06 (q, *J* = 7.0 Hz, 2H), 1.95–1.86 (m, 1H), 1.85–1.76 (m, 1H), 1.54–1.43 (m, 1H), 1.40–1.28 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 154.5, 142.3, 138.7, 114.8, 110.0, 108.0, 76.6, 56.5, 33.6 (2C), 25.0; **IR** (neat) 2979, 2820, 1641, 1504 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{11}H_{16}O_2$ $(M)^+$ 180.1150, found 180.1145; **$[\alpha]^{26}_D$** +54 (*c* 1.3, $CHCl_3$); **GC** analysis: 93% ee (CYCLODEX B, inlet temp 220 °C, flow rate 5.3781 mL/min, initial temp 55 °C, hold 2 min, ramp 10 °C/min up to 180 °C, hold 3 min, ramp 40 °C/min up to 230 °C, hold 1 min, t_{R1} = 33.72 min, t_{R2} = 33.75.



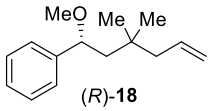
(*R*)-12. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**SI-17** (0.255 g, 1.00 mmol, 1.00 equiv), NaH (48 mg, 2.0 mmol, 2.0 equiv), methyl iodide (0.081 mL, 1.3 mmol, 1.3 equiv) and DMF (4 mL). The product was purified by flash column chromatography (2–5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.239 g, 0.890 mmol, 89%, 88% ee). **TLC R_f** = 0.8 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86–7.80 (m, 3H), 7.71 (s, 1H), 7.50–7.41 (m, 3H), 5.85 (ddt, *J* = 16.9, 9.9, 4.9 Hz, 1H), 5.05–4.98 (m, 2H), 4.38 (dd, *J* = 8.9, 3.1 Hz, 1H), 3.19 (s, 3H), 2.12–2.02 (m, 2H), 1.87 (dd, *J* = 14.8, 8.6 Hz, 1H), 1.53 (dd, *J* = 14.8, 3.1 Hz, 1H), 0.99 (s, 3H), 0.96 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.4, 135.8, 133.4, 133.1, 128.5, 127.94, 127.85, 126.2, 125.8, 125.4, 124.7, 117.2, 81.8, 56.4, 50.0, 47.5, 33.5, 27.78, 27.75; **IR** (neat) 3056, 2925, 1601, 1154 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{19}H_{24}O$ $(M)^+$ 268.1827, found 268.1837; **$[\alpha]^{23}_D$** +69 (*c* 1.3, $CHCl_3$); **SFC** analysis (OJ-H, 4% IPA, 2.5 mL/min) indicated 88% ee: t_R (major) = 4.1 minutes, t_R (minor) = 3.8 minutes.



(*R*)-14. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**SI-22** (0.190 g, 0.730 mmol, 1.00 equiv), NaH (36 mg, 1.5 mmol, 2.1 equiv), methyl iodide (0.10 mL, 1.1 mmol, 1.5 equiv) and THF (3 mL). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a pale yellow oil (0.191 g, 0.696 mmol, 95%). **TLC R_f** = 0.9 (15% EtOAc/hexanes, UV active); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.86–7.82 (m, 1H), 7.76–7.72 (m, 1H), 7.34 (tdd, *J* = 14.8, 7.1, 1.4 Hz, 2H), 7.19 (s, 1H), 5.87 (m, 2H), 5.10–5.02 (m, 2H), 4.60 (dd, *J* = 8.5, 3.5 Hz, 1H), 3.30 (s, 3H), 2.09 (ddt, *J* = 7.5, 3.2, 1.1 Hz, 2H), 1.98, (dd, *J* = 14.7, 8.4 Hz, 1H), 1.70 (dd, *J* = 14.7, 3.4 Hz, 1H), 1.02 (s, 3H), 0.99 (s, 3H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ 149.2, 139.9, 139.6, 135.7, 124.4, 124.3, 123.5, 122.8, 121.2, 117.4, 77.8, 56.6, 50.1, 47.6, 33.6, 27.8, 27.7; **IR** (neat) 3071, 2955, 1458, 1438, 1098, 912 cm^{-1}; **HRMS** (TOF MS ES+) *m/z* calcd for $C_{17}H_{22}OSNa$ $(M + Na)^+$ 297.1289, found 297.1288; **$[\alpha]^{29}_D$** +60 (*c* 1.5, $CHCl_3$).

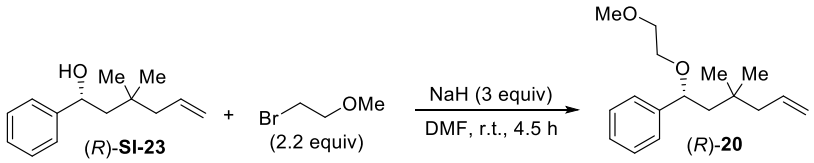


(*R*)-16. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**SI-5** (0.264 g, 1.50 mmol, 1.00 equiv), NaH (43 mg, 1.8 mmol, 1.2 equiv), methyl iodide (0.13 mL, 2.1 mmol, 1.4 equiv) and DMF (3 mL). The product was purified by flash column chromatography (2–5% Et_2O/hexanes) to afford the title compound as a colorless oil (0.257 g, 1.35 mmol, 90%, 93% ee). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.34 (t, *J* = 7.6 Hz, 2H), 7.28 (d, *J* = 7.1 Hz, 3H), 5.77 (ddt, *J* = 17.0, 10.3, 6.7 Hz, 1H), 4.98 (d, *J* = 17.0 Hz, 1H), 4.92 (d, *J* = 10.3 Hz, 1H), 4.09 (t, *J* = 6.9 Hz, 1H), 3.20 (s, 3H), 2.04 (dd, *J* = 14.2, 6.7 Hz, 2H), 1.86–1.77 (m, 1H), 1.68–1.58 (m, 1H), 1.55–1.45 (m, 1H), 1.39–1.29 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 142.5, 138.8, 128.5, 127.6, 126.8, 114.7, 84.1, 56.8, 37.8, 33.8, 25.3; **IR** (neat) 3056, 2925, 1601, 1154 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{13}H_{18}ONH_4$ $(M + NH_4)^+$ 208.1701, found 208.1706; **$[\alpha]^{23}_D$** +77 (*c* 0.7, $CHCl_3$); **SFC** analysis (OD-H, 5% IPA, 2.5 mL/min) indicated 93% ee: t_R (major) = 1.9 minutes, t_R (minor) = 2.1 minutes.

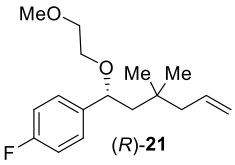


(*R*)-18. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol (*R*)-**SI-23** (0.125 g, 0.610 mmol, 1.00 equiv), NaH (21 mg, 0.85 mmol, 1.4 equiv), methyl iodide (0.046 mL, 0.73 mmol, 1.2 equiv) and DMF (3 mL). The product was purified by flash column chromatography (2–5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.116 g, 0.530 mmol, 87%, 94% ee). **TLC R_f** = 0.8 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.6 Hz, 2H), 7.30–7.231 (m, 3H), 5.84 (m, 1H), 5.05–4.97 (m, 2H), 4.22 (dd, *J* = 8.7, 2.3 Hz, 1H), 3.15 (s, 3H), 2.09–1.99 (m, 2H), 1.78 (dd, *J* = 14.8, 8.7 Hz, 1H), 1.45 (dd, *J* = 14.8, 2.3 Hz, 1H), 0.96 (s, 3H), 0.94 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ144.0, 135.8, 128.5, 127.4, 126.6, 117.1, 81.7, 56.3, 50.2, 47.5, 33.5, 27.7; **IR** (neat) 3056, 2925, 1601, 1154 cm^{-1}; **HRMS** (TOF MS EI+) *m* / *z* calcd for $C_{15}H_{22}O$ $(M)^+$ 218.1671, found 218.1667; **$[\alpha]^{23}_D$** +88 (*c* 1.0, $CHCl_3$); **SFC** analysis (OD-H, 1% IPA, 2.5 mL/min) indicated 94% ee: t_R (major) = 3.0 minutes, t_R (minor) = 3.2 minutes.

General Procedure H. *Alkylation of benzylic alcohol with 2-bromoethyl methyl ether*

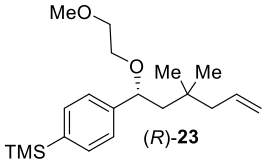


(*R*)-20. Alkylation of alcohols was performed according to a modified procedure reported by Lin.[13] Alcohol (*R*)-**SI-23** (0.183 g, 0.900 mmol, 1.00 equiv) was dissolved in DMF (3.5 mL) and added to a slurry of NaH (65 mg, 2.7 mmol, 3.0 equiv) in DMF (1.3 mL) at 0 ^{o}C. The reaction mixture was stirred for 30 min at ambient temperature, and a solution of bromoethyl methyl ether (0.10 mL, 1.0 mmol, 1.1 equiv) in DMF (4.2 mL) was slowly added over 30 min at 0 ^{o}C. The reaction was stirred for an additional 1 h at ambient temperature, after which a second portion of bromoethyl methyl ether (0.10 mL, 1.1 mmol, 1.1 equiv) in DMF (4.2 mL) was slowly added over 30 min at 0 ^{o}C. After stirring for 2 h, saturated aqueous NH_4Cl (15 mL) and EtOAc (20 mL) were added at 0 ^{o}C. The layers were separated and the aqueous phase was extracted with EtOAc (3 x 15 mL). The combined organic layers were washed with brine (20 mL), dried over Na_2SO_4, and concentrated in vacuo. The crude mixture was purified by flash column chromatography (5–10% EtOAc/hexanes) to afford the title compound as a colorless oil (0.185 g, 0.704 mmol, 78%, 92% ee). **TLC R_f** = 0.6 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.36–7.22 (m, 5H), 5.84 (ddt, *J* = 17.4, 9.8, 5.1 Hz, 1H), 5.04–4.97 (m, 2H), 4.39 (dd, *J* = 8.8, 2.6 Hz, 1H), 3.54–3.45 (m, 2H), 3.45–3.39 (m, 1H), 3.39–3.35 (m, 1H), 3.34 (s, 3H), 2.11–2.00 (m, 2H), 1.84 (dd, *J* = 14.6, 8.9 Hz, 1H), 1.44 (dd, *J* = 14.6, 2.2 Hz, 1H), 0.97 (s, 3H), 0.94 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.2, 136.0, 128.5, 127.4, 126.6, 117.0, 80.4, 72.2, 67.8, 59.0, 50.1, 47.4, 33.5, 27.8, 27.7; **IR** (neat) 2955, 2871, 1097 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{17}H_{26}O_2NH_4$ $(M + NH_4)^+$ 280.2277, found 280.2282; **$[\alpha]^{27}_D$** +66 (*c* 0.9, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 2.5 mL/min) indicated 92% ee: t_R (major) = 1.8 minutes, t_R (minor) = 2.1 minutes.



(*R*)-**21**

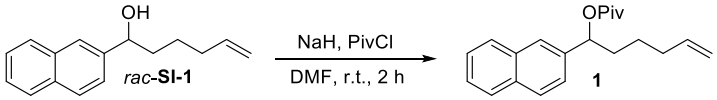
(*R*)-21. Using representative procedure H outlined above, the following amounts of reagents were used: alcohol (*R*)-**SI-24** (0.377 g, 1.70 mmol, 1.00 equiv), NaH (100 mg, 4.08 mmol, 2.40 equiv), bromoethyl methyl ether (0.50 mL, 5.3 mmol, 3.1 equiv) and DMF (8 mL). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.252 g, 0.899 mmol, 53%). Enantiomeric excess could not be determined for the title compound using SFC and chiral GC instrumentation. **TLC R_f** = 0.6 (5% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.28–7.22 (m, 2H), 7.01 (at, *J* = 8.8 Hz, 2H), 5.86 (ddt, *J* = 17.6, 10.4, 7.5 Hz, 1H), 5.04–4.97 (m, 2H), 4.37 (dd, *J* = 8.7, 2.8 Hz, 1H), 3.53–3.31 (m, 4H), 3.34 (s, 3H), 2.10–1.99 (m, 2H), 1.82 (dd, *J* = 14.7, 8.8 Hz, 1H) 1.42 (dd, *J* = 14.7, 3.0 Hz, 1H), 0.96 (s, 3H), 0.93 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 162.0 (d, *J* = 245 Hz), 139.8 (d, *J* = 3 Hz), 135.8, 128.0 (d, *J* = 8 Hz), 117.0, 115.3 (d, *J* = 21 Hz), 79.7, 72.1, 67.7, 58.9, 50.0, 47.3, 33.4, 27.7, 27.6; **IR** (neat) 2955, 2871, 1638, 1603, 1507, 1220, 1092, 912 cm$^-$1; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{17}H_{25}FO_2NH_4$ $(M + NH_4)^+$ 298.2182, found 298.2185; **$[\alpha]^{27}_D$** +54 (*c* 1.5, $CHCl_3$).



(*R*)-**23**

(*R*)-23. Using representative procedure H outlined above, the following amounts of reagents were used: alcohol (*R*)-**SI-25** (0.197 g, 0.712 mmol, 1.00 equiv), NaH (52 mg, 2.2 mmol, 3.1 equiv), bromoethyl methyl ether (0.30 mL, 3.2 mmol, 4.5 equiv) and DMF (8 mL). The product was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.128 g, 0.382 mmol, 54%). Enantiomeric excess could not be determined for the title compound using SFC and chiral GC instrumentation. **TLC R_f** = 0.6 (5% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 7.7 Hz, 2H), 5.86 (ddt, *J* = 17.9, 10.3, 7.5 Hz, 1H), 5.00 (m, 2H), 4.39 (dd, *J* = 9.2, 2.4 Hz, 1H), 3.53–3.35 (m, 4H), 3.35 (s, 3H), 3.36 (s, 3H), 2.11–2.01 (m, 2H), 1.83 (dd, *J* = 14.8, 9.2 Hz, 1H), 1.42 (dd, *J* = 14.8, 2.6 Hz, 1H), 0.98 (s, 3H), 0.95 (s, 3H), 0.25 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.7, 139.2, 135.9, 133.5, 125.8, 116.9, 80.3, 72.2, 67.8, 58.9, 50.1, 47.3, 33.5, 27.7, 27.6, -1.0; **IR** (neat) 2954, 1637, 1600, 1248, 1132, 911 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{20}H_{34}O_2SiNH_4$ $(M + NH_4)^+$ 352.2672, found 352.2670; **$[\alpha]^{29}_D$** +48 (*c* 1.4, $CHCl_3$).

iv. Esterification to provide benzylic pivalate 1



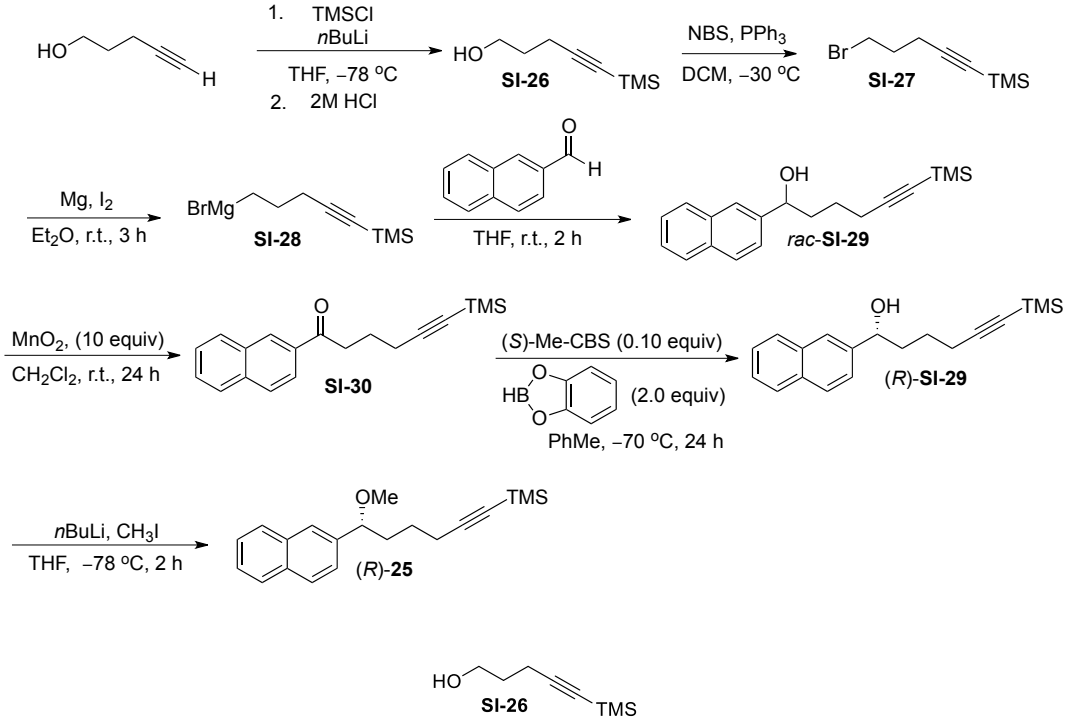
1. In a glovebox, a round bottom flask was equipped with a stir bar, and charged with NaH (84 mg, 3.5 mmol, 1.4 equiv). The flask was removed from the glovebox, and DMF (5 ml) was added, followed by a solution of *rac*-**SI-1** (0.566 g, 2.50 mmol, 1.00 equiv) in DMF (8 mL). The reaction flask was cooled to 0 ^{o}C in an ice bath and pivaloyl chloride (0.30 mL, 2.5 mmol, 1.00 equiv) was slowly added over 15 min. The reaction was allowed to warm to room temperature and was stirred for 2 h before quenching with sat. NH_4Cl (15 mL). The mixture was extracted with EtOAc (3 x 10 mL) and the combined organics were washed with brine (1 x 25 mL), dried over $MgSO_4$, filtered and concentrated in vacuo. The residue was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (0.656 g, 2.20 mmol, 88%). **TLC R_f** = 0.5 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85–7.78 (m, 3H), 7.75 (s, 1H), 7.50–7.41 (m, 3H), 5.86 (t, *J* = 6.8 Hz, 1H), 5.75 (ddt, *J* = 16.9, 9.9, 3.4 Hz, 1H), 4.99 (d, *J* = 17.2, Hz, 1H), 4.94 (d, *J* = 10.0 Hz, 1H), 2.12–1.94 (m, 3H), 1.91–1.81 (m, 1H), 1.55–1.33 (m, 2H), 1.22 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 117.8, 138.7, 138.4, 133.3, 133.1, 128.4, 128.2, 127.8, 126.3, 126.1, 125.5, 124.2, 115.0, 75.8, 39.0, 36.0, 33.5, 27.3, 24.9; **IR** (neat) 2976, 1721, 1153 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{21}H_{26}O_2Na$ $(M + Na)^+$ 333.1830, found 333.1821.

IV. Synthesis and characterization of substrates for Schemes 2–4

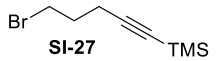
i. Alkyne 21 (Scheme 2)

Alkyne (*R*)-**25** was prepared by Grignard addition of **SI-28** into 2-naphthaldehyde. The resultant alcohol *rac*-**SI-29** was oxidized and the ketone underwent enantioselective reduction and was alkylated.

Scheme SI-2. Synthesis of alkyne (*R*)-21 for Scheme 2.

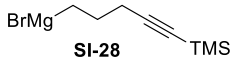


SI-26. The TMS protected alkyne was prepared according to a modified procedure reported by Ramesh.[14] A flame-dried round bottom flask equipped with a stir bar was charged with 4-pentyn-1-ol (1.86 mL, 20.0 mmol, 1.00 equiv) and THF (50 mL) and cooled to –78 °C. *n*-Butyllithium (29 mL, 1.5 M in hexanes, 44 mmol, 2.2 equiv) was slowly added to the flask and stirred for 1 h at –78 °C. TMSCl (7.6 mL, 60 mmol, 3.0 equiv) was added and the reaction was allowed to warm slowly to ambient temperature overnight. The reaction was cooled to 0 °C and quenched with 2M HCl and stirred at ambient temperature until the *bis*-silylated product was no longer present by TLC. The reaction mixture was then extracted with Et_2O (3 x 40 mL), and the combined organic layers were washed with brine, $NaHCO_3$ and with brine once more before drying over $MgSO_4$, filtration and concentration in vacuo. The product was purified by flash column chromatography (10–30% EtOAc/hexanes) to afford the title compound as a clear oil (2.64 g, 16.9 mmol, 84%). The analytical data is consistent with literature values.[13] **TLC R_f =** 0.3 (10% EtOAc/Hexanes stain with $KMnO_4$); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.76 (q, *J* = 5.9 Hz, 2H), 2.36 (t, *J* = 6.9 Hz, 2H), 1.78 (quin, *J* = 6.9 Hz, 2H), 1.59 (at, *J* = 5.9 Hz, 1H), 0.15 (s, 9H).



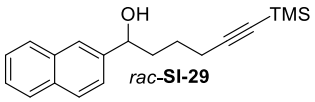
SI-27

SI-27. The bromide was prepared according to a modified procedure reported by Steliou.[15] A flame-dried round bottom flask equipped with a stir bar and septum was charged with anhydrous CH_2Cl_2 (40 mL) and alcohol **SI-26** (2.64 g, 16.8 mmol, 1.00 equiv). Triphenylphosphine (5.3 g, 20 mmol, 1.2 equiv) was added in a single portion and the flask was cooled to –30 °C before addition of NBS (3.30 g, 18.5 mmol, 1.10 equiv) in a single portion. The reaction was then allowed to warm to ambient temperature over 5 h. Et_2O (150 mL) was added to the reaction flask and the mixture was extracted with sat. $NaHCO_3$ (2 x 40 mL), washed with brine (1 x 50 mL), dried over $MgSO_4$ and concentrated in vacuo. A stir bar and 300 mL of hexanes were added to the remaining liquid and stirred for 15 min. The solution was filtered through celite and concentrated in vacuo. The product was purified by flash column chromatography (100% petroleum ether) to afford a colorless oil (2.33 g, 10.6 mmol, 63%). **TLC R_f** = 0.5 (100% petroleum ether, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 3.51 (t, J = 6.5 Hz, 2H), 2.42 (t, J = 6.5 Hz, 2H), 2.05 (quin, J = 6.6 Hz, 2H), 0.15 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 105.2, 85.9, 32.5, 31.6, 18.7, 0.2 (3C); **IR** (neat) 2959, 2177, 1248 cm^{-1}.



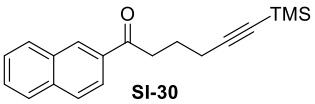
SI-28

SI-28. The Grignard reagent was prepared according to a modified procedure reported by Waldman.[16] A flame-dried 50 mL round bottom flask was equipped with a stir bar and charged with magnesium turnings (0.331 g, 13.8 mmol, 1.30 equiv), Et_2O (8 mL) and a catalytic amount (1 mg) of iodine. Approximately 20% of the volume of neat **SI-27** (2.32 g, 10.6 mmol, 1.00 equiv) was added in one portion. The reaction was heated briefly with a heat gun to reflux to initiate the reaction, and cooled to 0 °C after color change from red/pink to a colorless solution indicated initiation of the Grignard reagent. The remainder of the bromide **SI-27** was added dropwise over 30 minutes. The reaction was warmed to ambient temperature and stirred for 2.5 h. The resultant Grignard reagent was titrated with iodine and LiCl (0.50 M).[2]



rac-**SI-29**

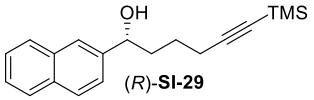
***rac*-SI-29.** Using representative procedure A outlined above, the following amounts of reagents were used: 2-naphthaldehyde (0.610 g, 3.91 mmol, 1.00 equiv), **SI-28** (0.50 M in Et_2O, 8.0 mL, 4.0 mmol, 1.0 equiv). The product was purified by flash column chromatography (5–10% EtOAc/hexanes) to afford the title compound as a white solid (1.03 g, 3.48 mmol, 89%). Analytical data is consistent with the values listed for (*R*)-**SI-29** (vide infra).



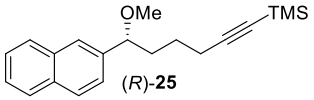
SI-30

SI-30. Using representative procedure B outlined above, the following amounts of reagents were used: Alcohol *rac*-**SI-29** (1.270 g, 4.480 mmol, 1.000 equiv), MnO_2 (3.89 g, 44.8 mmol, 10.0 equiv), CH_2Cl_2 (90 mL). Further purification after celite plug was unnecessary. The title compound was isolated as a white solid (1.28 g, 4.40 mmol, 98%). **TLC R_f** = 0.6 (10% EtOAc/hexanes, UV active,); **m.p.** = 65 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.50 (s, 1H), 8.05 (d, J = 8.4 Hz, 1H), 7.97 (d, J = 8.1 Hz, 1H), 7.90 (d, J = 9.3 Hz, 1H), 7.88 (d, J = 9.3 Hz, 1H), 7.61

(t, J = 7.0 Hz, 1H), 7.56 (t, J = 7.7 Hz, 1H), 3.25 (t, J = 7.5 Hz, 2H), 2.41 (t, J = 7.0 Hz, 2H), 2.03 (quin, J = 7.1 Hz, 2H), 0.16 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 199.9, 135.7, 134.4, 132.7, 129.8, 129.7, 128.59, 128.57, 127.9, 126.9, 124.0, 106.7, 85.7, 37.6, 23.3, 19.6, 0.3; **IR** (neat) 3059, 2957, 2230, 1680 cm^{-1}; **HRMS** (TOF MS CI+) m / z calcd for $C_{19}H_{22}OSiH$ (M + H)$^+$ 295.1518, found 295.1519.



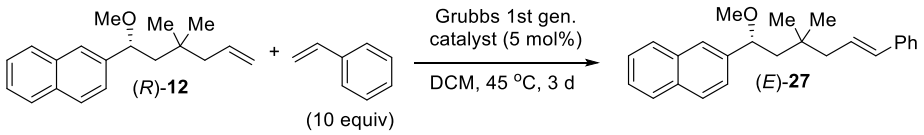
(*R*)-SI-29. Using representative procedure F outlined above, the following amounts of reagents were used: ketone **SI-30** (0.84 g, 3.0 mmol, 1.0 equiv), (*S*)-Me-CBS (68 mg, 0.30 mmol, 0.10 equiv), catecholborane (0.64 mL, 6.0 mmol, 2.0 equiv), and PhMe (25 mL). The product was purified by flash column chromatography (8–15% EtOAc/hexanes) to afford the title compound as a white solid (0.73 g, 2.6 mmol, 87%, 93% ee). **TLC R_f** = 0.2 (10% EtOAc/hexanes, UV active); **m.p.** = 48–49 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.87–7.81 (m, 3H), 7.79 (s, 1H), 7.51–7.45 (m, 3H), 4.92–4.87 (m, 1H), 2.33–2.22 (m, 2H), 2.03–1.90 (m, 3H), 1.73–1.63 (m, 1H), 1.61–1.51 (m, 1H), 0.14 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 142.0, 133.4, 133.1, 128.5, 128.1, 127.8, 126.3, 126.0, 124.8, 124.2, 107.2, 85.1, 74.3, 38.0, 24.9, 19.8, 0.3; **IR** (neat) 3266, 2941, 2173 cm^{-1}; **HRMS** (TOF MS ES+) m / z calcd for $C_{19}H_{24}OSiNa$ (M + Na)$^+$ 319.1494, found 319.1498; **$[\alpha]^{24}_D$** +69 (c 2.8, $CHCl_3$); **SFC** analysis (OD-H, 12% IPA, 2.5 mL/min) indicated 93% ee: t_R (major) = 11.6 minutes, t_R (minor) = 9.5 minutes.



(*R*)-25. A flame-dried round bottom flask equipped with a stir bar and septum was charged with (*R*)-**SI-29** (0.565 g, 2.00 mmol, 1.00 equiv) and anhydrous THF (15 mL). The flask was then cooled to –78 °C and *n*-butyllithium was slowly added (1.7 mL, 1.5 M in hexanes, 2.5 mmol, 1.3 equiv). The reaction was stirred for 45 min before the addition of methyl iodide (0.80 mL, 13 mmol, 5.0 equiv). The reaction was then allowed to warm to ambient temperature overnight before quenching with 1 M HCl at 0 °C. The mixture was extracted with Et_2O (3 x 20 mL) and the combined organic layers were washed with brine, dried over $MgSO_4$, filtered, and concentrated in vacuo. The product was purified by flash column chromatography (8% EtOAc/hexanes) to afford the title compound as a colorless oil (0.565 g, 1.80 mmol, 91%, 84% ee). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.88–7.79 (m, 3H), 7.72 (s, 1H), 7.51–7.41 (m, 3H), 4.29 (t, J = 6.7 Hz, 1H), 3.24 (s, 3H), 2.29–2.18 (m, 2H), 2.02–1.93 (m, 1H), 1.89–1.80 (m, 1H), 1.69–1.58 (m, 1H), 1.55–1.45 (s, 1H), 0.13 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 139.7, 133.3, 133.2, 128.5, 128.0, 127.9, 126.3, 126.0, 125.9, 124.5, 107.2, 84.7, 83.7, 56.8, 37.1, 24.9, 19.9, 0.3; **IR** (neat) 2955, 2172, 1602 cm^{-1}; **HRMS** (TOF MS CI+) m / z calcd for $C_{20}H_{26}OSiNH_4$ (M + NH_4)$^+$ 328.2097, found 328.2091; **$[\alpha]^{24}_D$** +55 (c 1.1, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 2.5 mL/min) indicated 84% ee: t_R (major) = 3.6 minutes, t_R (minor) = 3.9 minutes.

ii. Alkenes (*E*)-27 and (*Z*)-27 (Scheme 3)

Preparation of E 1,2-disubstituted olefin by cross-metathesis.

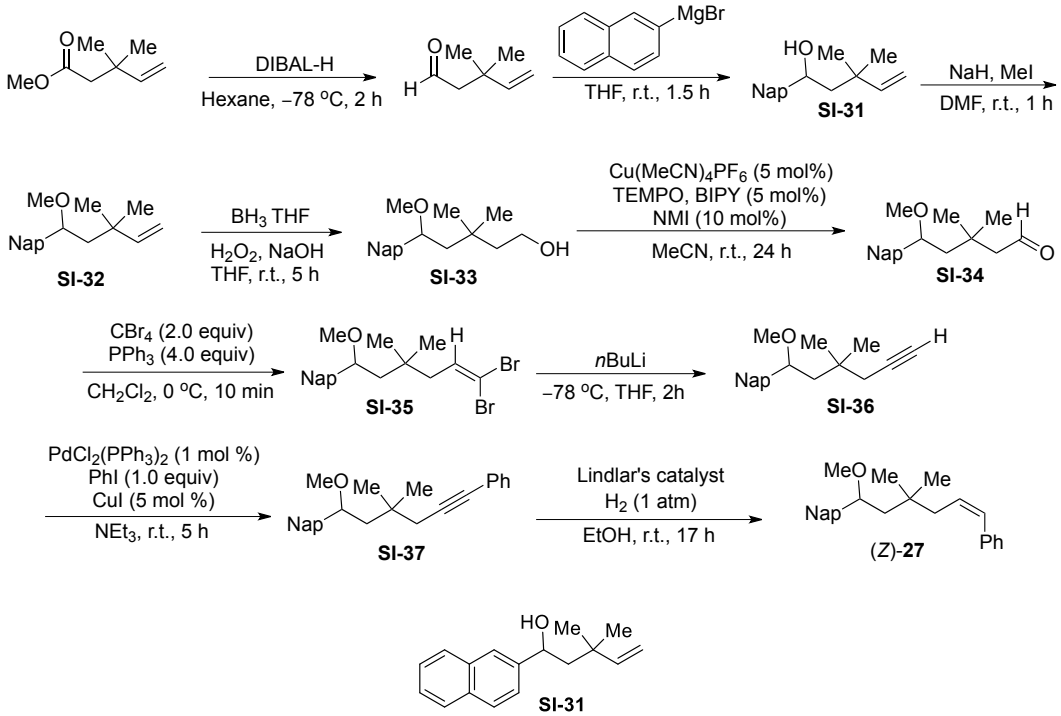


(*E*)-23. The title compound was prepared according to a modified procedure reported by Grubbs.[17] In a glovebox, a flame-dried bomb flask was charged with a stir bar, (*R*)-**12** (0.178 g, 0.660 mmol, 1.00 equiv), and bis(tricyclohexylphosphine)benzylidine ruthenium(IV) dichloride (27 mg, 0.053 mmol, 0.050 equiv). The flask was removed from the glovebox, and anhydrous CH_2Cl_2 (25 mL) and styrene (0.76 mL, 6.6 mmol, 10 equiv) were added. The flask was sealed and heated to reflux over three days. The flask was then cooled to ambient temperature, and the solvent was removed in vacuo. The residue was purified by flash column chromatography to afford the title compound as a colorless oil (102 mg, 0297 mmol, 45%, 89% ee). **TLC R_f** = 0.9 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86–7.77 (m, 3H), 7.71 (s, 1H), 7.51–7.41 (m, 3H), 7.33 (d, *J* = 7.5 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.18 (t, *J* = 7.3 Hz, 1H), 6.37 (d, *J* = 15.8 Hz, 1H), 6.27 (dt, *J* = 15.8, 7.4 Hz, 1H), 4.42 (dd, *J* = 8.6, 2.7 Hz, 1H), 3.20 (s, 3H), 2.27–2.16 (m, 2H), 1.93 (dd, *J* = 14.7, 8.8 Hz, 1H), 1.58 (dd, *J* = 14.8, 2.7 Hz, 1H), 1.06 (s, 3H), 1.02 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.3, 138.0, 113.4, 133.1, 132.4, 128.6, 128.5, 127.94, 127.86, 127.85, 127.0, 126.22, 126.15, 125.8, 125.5, 124.7, 81.9, 56.5, 50.2, 46.7, 34.3, 27.9 (2C); **IR** (neat) 3024, 3055, 2954, 1599 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{25}H_{28}O$ $(M)^+$ 344.2140, found 344.2131. **$[\alpha]^{24}_D$** +74 (*c* 2.6, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 2.5 mL/min) indicated 89% ee: t_R (major) = 7.3 minutes, t_R (minor) = 8.3 minutes.

Preparation of Z 1,2-disubstituted olefin.

(*Z*)-**27** was prepared from 3,3-dimethyl-4-pentenoate by the sequence outlined in Scheme SI-3.

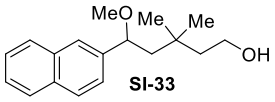
Scheme SI-3. Synthesis of (*Z*)-27 for Scheme 3.



SI-31. The title compound was prepared according to a modified procedure reported by Roush.[18] To a solution of methyl 3,3-dimethyl-4-pentenoate (12 mL, 76 mmol, 1.0 equiv) in hexane (200 mL) was added at –78 °C, neat DIBAL-H (16.2 mL, 91.1 mmol, 1.2 equiv) dropwise over 45 min. The reaction was stirred for 1 h at this temperature and quenched with MeOH. A saturated solution of Rochelle's salt (100 mL) was added and the biphasic layer was stirred overnight. The layers were separated and the aqueous phase was extracted with Et_2O (3 x 50 mL. The combined organics were dried ($MgSO_4$), filtered, and concentrated in vacuo. Vacuum distillation afforded the desired product (26%, as measured by ^{1}H NMR) as a 2:1 mixture with the over-reduced, primary alcohol. This mixture was then dissolved in THF (40 mL) and was added to a solution of 2-naphthylmagnesium bromide. After stirring for 1 h at room temperature, the reaction was quenched with sat. NH_4Cl. The layers were separated, and the aqueous phase was extracted with (4 x 40 mL EtOAc). The combined organics were dried ($MgSO_4$), filtered, and concentrated in vacuo. The residue was purified by flash column chromatography (3% EtOAc/hexanes) to afford the title compound as a white solid (3.92 g, 16.3 mmol, 22%). **TLC R_f** = 0.3 (10% EtOAc/hexanes, UV active); **m.p.** = 66 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.84–7.78 (m, 3H), 7.77 (s, 1H), 7.49–7.41 (m, 3H), 6.02 (dd, *J* = 17.5, 10.9 Hz, 1H), 5.13–5.03 (m, 2H), 4.96 (d, *J* = 9.2 Hz, 1H), 2.23 (d, *J* = 1.8 Hz, 1H), 1.93 (dd, *J* = 14.7, 9.2 Hz, 1H), 1.75 (dd, *J* = 14.7, 1.8 Hz, 1H), 1.16 (s, 3H), 1.11 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 148.8, 143.0, 133.4, 132.9, 128.3, 128.0, 127.7, 126.1, 125.8, 124.2, 111.6, 72.4, 52.3, 36.8, 28.8, 26.0; **IR** (neat) 3397, 3056, 2926 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{17}H_{20}ONa$ $(M + Na)^+$ 263.1412, found 263.1413.

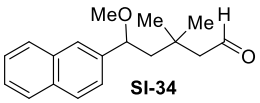
SI-32

SI-32. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol **SI-31** (3.70 g, 15.2 mmol, 1.00 equiv), NaH (0.511 g, 21.3 mmol, 1.4 equiv), methyl iodide (1.52 mL, 24.3 mmol, 1.60 equiv) and DMF (30 mL). The product was purified by flash column chromatography (2–5% EtOAc/hexanes) to afford the title compound as a colorless oil (3.73 g, 14.7 mmol, 97%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85–7.79 (m, 3H), 7.69 (s, 1H), 7.50–7.41 (m, 3H), 5.89 (dd, *J* = 17.5, 10.8 Hz, 1H), 5.01–4.94 (m, 2H), 4.27 (dd, *J* = 8.2, 2.8 Hz, 1H), 3.16 (s, 3H), 1.95 (dd, *J* = 14.6, 8.2 Hz, 1H), 1.64 (dd, *J* = 14.6, 3.0 Hz, 1H), 1.13 (s, 3H), 1.03 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 148.5, 141.1, 133.3, 133.0, 128.4, 127.9, 127.8, 126.1, 125.7, 125.4, 124.7, 110.4, 81.8, 56.3, 50.9, 36.8, 28.1, 26.9; **IR** (neat) 3056, 2926, 1601 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{18}H_{22}ONa$ $(M + Na)^+$ 277.1568, found 277.1575.



SI-33

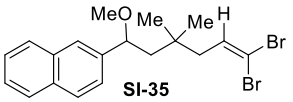
SI-33. The title compound was prepared according to a modified procedure reported by Kumar.[19] In a 500 mL round bottom flask, a solution of **SI-32** (3.70 g, 14.7 mmol, 1.00 equiv) in THF (60 mL) was cooled ot 0 °C and borane THF complex (29.4 mL, 1M in THF, 29.4 mmol, 2.0 equiv) was slowly added. The solution was stirred at ambient temperature for 3 h. The solution was cooled to 0 °C and 1M NaOH (17.2 mL, 103 mmol, 7.03 equiv) was added over 20 minutes. To this mixture was added 30% H_2O_2 (30.0 mL, 294 mmol, 20.0 equiv). The reaction was stirred another 2 h before diluting with EtOAc and extraction of the aqueous layer with EtOAc (3 x 30 mL). The combined organics were washed with water and brine, dried ($MgSO_4$), filtered, and concentrated in vacuo. The residue was purified by flash column chromatography (15% EtOAc/hexanes) to afford the title compound as a thick colorless oil (2.50 g, 9.18 mmol, 62%). **TLC R_f** = 0.2 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.87–7.79 (m, 3H), 7.71 (s, 1H), 7.51–7.40 (m, 3H), 4.43 (d, *J* = 9.4 Hz, 1H), 3.83–3.71 (m, 2H), 3.22 (s, 3H), 2.54 (br s, 1H), 2.11 (dd, *J* = 15.1, 9.5 Hz, 1H), 1.86 (quintet, *J* = 7.3 Hz, 1H), 1.53 (dt, *J* = 14.2, 5.6 Hz, 1H), 1.46 (dd, *J* = 15.1, 1.6 Hz, 1H), 1.07 (s, 3H), 0.99 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 140.7, 133.3, 133.1, 128.5, 127.85, 127.78, 126.2, 125.9, 125.2, 124.4, 81.8, 59.9, 56.3, 49.9, 43.8, 32.6, 28.9, 28.8; **IR** (neat) 3353, 2928, 975 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{18}H_{24}O_2Na$ $(M + Na)^+$ 295.1674, found 295.1663.



SI-34

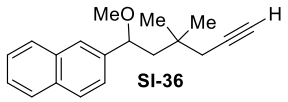
SI-34. The title compound was prepared according to a modified procedure reported by Stahl.[20] A solution of **SI-33** (2.48 g, 9.10 mmol, 1.00 equiv) in dry MeCN (50 mL) was prepared in a 100 mL round bottom flask. To this solution was added tetrakis(acetonitrile)copper(I)hexafluorophosphate (170 mg, 0.455 mmol, 0.0500 equiv), 2,2'-dipyridyl (71 mg, 0.46 mmol, 0.050 equiv), 2,2,6,6-tetramethylpiperidinooxy (71 mg, 0.46 mmol, 0.050 equiv), and 1-methylimidazole (0.07 mL, 0.9 mmol, 0.1 equiv). The reaction was stirred open to air for 24 h. The solvent was removed in vacuo and the residue purified by flash

column chromatography (5% EtOAc/hexanes) to afford the title compound as a slightly orange oil (2.09 g, 7.72 mmol, 85%). **TLC R_f** = 0.4 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 9.88 (at, *J* = 2.7 Hz, 1H), 7.87–7.79 (m, 3H), 7.71 (s, 1H), 7.51–7.39 (m, 3H), 4.41 (dd, *J* = 9.4, 1.8 Hz, 1H), 3.18 (s, 3H), 2.50 (dd, *J* = 15.0, 1.8 Hz, 1H), 2.36 (dd, *J* = 15.0, 2.6 Hz, 1H), 2.06 (dd, *J* = 15.0, 9.7 Hz, 1H), 1.63–1.55 (m, 1H), 1.20 (s, 3H), 1.14 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 203.7, 140.4, 133.3, 133.1, 128.6, 127.9, 127.8, 126.3, 125.9, 125.3, 124.4, 81.5, 56.3, 54.8, 50.4, 33.3, 29.2, 28.3; **IR** (neat) 2956, 1716, 1101 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{18}H_{22}O_2Na$ $(M + Na)^+$ 293.1518, found 293.1509.



SI-35

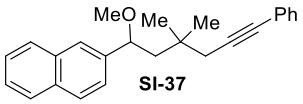
SI-35. The title compound was prepared according to a modified procedure reported by Hoppe.[21] A solution of triphenylphosphine (5.88 g, 22.4 mmol, 4.00 equiv) in CH_2Cl_2 (20 mL) was cooled to 0 °C in an ice bath. A solution of carbon tetrabromide (3.77 g, 11.1 mmol, 2.00 equiv) in CH_2Cl_2 (5 mL) was added (solution turns clear orange). After 2 minutes of stirring, a solution of **SI-34** (1.52 g, 5.60 mmol, 1.00 equiv) in CH_2Cl_2 (5 mL) was added. The reaction was monitored by TLC for disappearance of **SI-34**, and the reaction was complete after 10 min. The reaction mixture was run through a large plug of silica and eluted with CH_2Cl_2. The solvent was then removed in vacuo to afford the title compound as a thick pale yellow oil (2.28 g, 5.36 mmol, 96%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.87–7.81 (m, 3H), 7.71 (s, 1H), 7.51–7.41 (m, 3H), 6.50 (t, *J* = 7.5 Hz, 1H), 4.41 (dd, *J* = 9.1, 2.0 Hz, 1H), 3.19 (s, 3H), 2.20–2.10 (m, 2H), 1.91 (dd, *J* = 15.0, 9.1 Hz, 1H), 1.52 (dd, *J* = 15.0, 2.0 Hz, 1H), 1.05 (s, 3H), 1.00 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 140.8, 136.5, 133.3, 133.1, 128.6, 127.9, 127.8, 126.2, 125.9, 125.4, 124.5, 89.4, 81.6, 56.4, 50.0, 45.7, 34.3, 28.0, 27.7; **IR** (neat) 2955, 2925, 818, 779 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{19}H_{22}Br_2ONa$ $(M + Na)^+$ 446.9935, found 446.9930.



SI-36

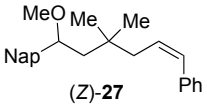
SI-36. The title compound was prepared according to a modified procedure reported by Hoppe.[17] In a 100 mL round bottom flask, a solution of **SI-35** (2.25 g, 5.28 mmol, 1.00 equiv) in THF (30 mL) was cooled to –78 °C. To this solution was added *n*-butyllithium (4.30 mL, 2.50 M in hexanes, 10.6 mmol, 2.00 equiv) dropwise over 20 min. The reaction was stirred at this temperature for 1 h before warming to ambient temperature and stirring for another 1 h. The reaction was quenched with MeOH, followed by H_2O. The aqueous phase was extracted with Et_2O (3 x 30 mL) and washed with brine, dried ($MgSO_4$), filtered, and concentrated in vacuo. The product was purified by flash column chromatography (3% Et_2O/hexanes) to afford the title compound as a clear, colorless oil (1.01 g, 3.79 mmol, 72%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.87–7.79 (m, 3H), 7.72 (s, 1H), 7.51–7.42 (m, 3H), 4.40 (dd, *J* = 9.2, 2.2 Hz, 1H), 3.19 (s, 3H), 2.25 (dd, *J* = 16.8, 2.1 Hz, 1H), 2.20 (dd, *J* = 16.5, 2.1 Hz, 1H), 2.00 (br s, 1H), 1.95 (dd, *J* = 14.6, 9.2 Hz, 1H), 1.65 (dd, *J* = 14.2, 2.2 Hz, 1H), 1.10 (s, 3H), 1.08 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 140.9, 133.3, 133.1, 128.4, 127.9, 127.8, 126.2, 125.8, 125.3, 124.6, 82.8, 81.8, 70.1, 56.4, 49.2, 33.5, 32.3, 27.7,

27.4;**IR** (neat) 3303, 2957, 2927 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{19}H_{22}ONa$ $(M + Na)^+$ 289.1568, found 289.1568.



SI-37

SI-37. The title compound was prepared according to a modified procedure reported by Hoppe.[17] In a glovebox, a flame-dried 50 mL round bottom flaks was equipped with a stir bar and charged with **SI-36** (1.00 g, 3.75 mmol, 1.00 equiv), bis(triphenylphosphine)palladium(II) dichloride (26 mg, 0.038 mmol, 0.010 equiv), copper(I) iodide (36 mg, 0.18 mmol, 0.050 equiv), iodobenzene (0.42 mL, 3.8 mmol, 1.0 equiv), and Et_3N (30 mL). The reaction was stirred in the glovebox at ambient temperature for 5 h. The round bottom flask was removed from the glovebox and quenched with 1M HCl (50 mL). The organic layer was separated and the aqueous phase was extracted with Et_2O (3 x 30 mL). The combined organics were washed with brine, dried (Na_2SO_4), filtered, and concentrated in vacuo. The residue was purified by flash column chromatography (4% Et_2O/hexanes) to afford the title compound as a clear, colorless oil (1.19 g, 3.67 mmol, 98%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86–7.77 (m, 3H), 7.74 (s, 1H), 7.53–7.42 (m, 3H), 7.42–7.35 (m, 2H), 7.32–7.22 (m, 3H), 4.40 (dd, *J* = 8.9, 2.5 Hz, 1H), 3.21 (s, 3H), 2.48 (d, *J* = 16.5 Hz, 1H), 2.39 (d, *J* = 16.5 Hz, 1H), 1.98 (dd, *J* = 14.6, 8.9 Hz, 1H), 1.75 (dd, *J* = 14.6, 2.5 Hz, 1H), 1.14 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.0, 133.3, 133.1, 131.6, 128.4, 128.2, 127.9, 127.8, 127.5, 126.1, 125.8, 125.4, 124.6, 124.2, 88.7, 82.5, 81.9, 56.4, 49.5, 34.1, 33.2, 27.9, 27.8; **IR** (neat) 3054, 2956, 2926 cm^{-1}; **HRMS** (TOF MS ES+) *m / z* calcd for $C_{25}H_{26}ONa$ $(M + Na)^+$ 365.1881, found 365.1870.



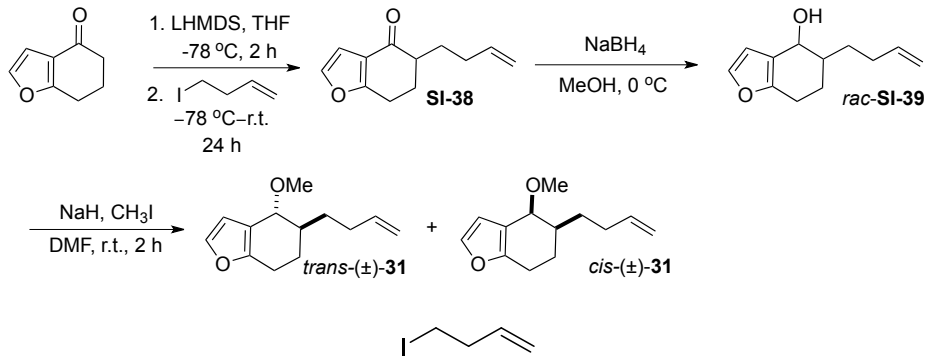
(*Z*)-**27**

(Z)-23. A 25 mL round bottom flask was charged with a stir bar and a solution of alkyne **SI-32** (200 mg, 0.620 mmol, 1.00 equiv) in EtOH (12 mL). The reaction flask was thoroughly purged with argon from a balloon and Lindlar's catalyst (69 mg, 0.020 mmol, 0.032 equiv with regard to palladium) was added neat. The argon atmosphere was exchanged with H_2 and the reaction was allowed to stir for 17 h, refilling the balloon with additional H_2 as necessary. At the end of the reaction, the H_2 atmosphere was exchanged with argon, and the reaction solution was filtered through a plug of celite, rinsing with 25% Et_2O/hexanes. The organics were concentrated in vacuo and the residue was purified by flash column chromatography on silver nitrate impregnated silica (1–5% Et_2O/hexanes with a final flush of 25% Et_2O/hexanes). The product was isolated as a clear oil (120 mg, 0.370 mmol, 60%, 10:1 Z:E). **TLC R_f** = 0.4 (5% Et_2O/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86–7.76 (m, 3H), 7.61 (s, 1H), 7.51–7.41 (m, 2H), 7.38 (d, *J* = 8.1 Hz, 1H), 7.35–7.25 (m, 4H), 7.25–7.18 (m, 1H), 6.52 (d, *J* = 12.0 Hz, 1H), 5.79 (dt, *J* = 14.4, 7.4 Hz, 1H), 4.27 (dd, *J* = 8.6, 2.6 Hz, 1H), 3.09 (s, 3H), 2.42 (dd, *J* = 14.4, 7.4 Hz, 1H), 2.34 (dd, *J* = 14.4, 7.4 Hz, 1H), 1.89 (dd, *J* = 14.7, 8.6 Hz, 1H), 1.56 (dd, *J* = 14.7, 2.6 Hz, 1H), 1.01 (s, 3H), 0.99 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.2, 138.0, 133.4, 133.1, 130.3, 129.6, 129.0, 128.5, 128.2, 127.9, 127.8, 126.6, 126.2, 125.8, 125.5, 124.7, 81.7, 56.3, 49.9, 40.7, 33.9, 28.1, 27.9.

iii. Dihydrobenzofurans *cis*-27 and *trans*-27 (Scheme 4)

cis and *trans*-**31** were prepared by a-alkylation of 6,7-dihydro-4(5H)-benzofuranone, followed by reduction and methylation of the diastereomeric mixture of alcohol *rac*-**SI-39**. Separation of the diastereomers by silica gel chromatography afforded *cis* and *trans*-**31.**

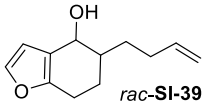
Scheme SI-4. Synthesis of substrates for Scheme 4.



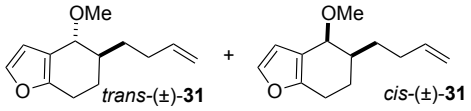
4-iodo-1-butene. The iodide was prepared according to a modified procedure reported by Evans.[22] To a stirred solution of NaI (4.50 g, 30.0 mmol, 2.00 equiv) in acetone (50 mL) was added 4-bromobutene (1.52 mL, 15.0 mmol, 1.00 equiv) and the reaction mixture heated at reflux for 45 min. After removing most of the solvent in vacuo at ambient temperature (the compound is extremely volatile, bp 128–130 °C), the remaining liquid was run through a plug of silica (100% pentane) and concentrated in vacuo (care was taken not to raise the temperature of the bath or leave under vacuum for longer than necessary) to afford the title compound as a colorless liquid (1.05 g, 5.70 mmol, 38%). The product must be stored in the dark to avoid rapid decomposition, as evident by a color change to pink/ orange. The analytical data is consistent with literature values.[23] **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.46–5.41 (m, 1H), 5.18–5.07 (m, 2H), 3.19 (t, 2H, J = 7.2), 2.62 (q, 2H, J = 7.0).

SI-38. The ketone was prepared according to a modified procedure reported by Li.[24] In a glovebox, LHMDS (1.36 g, 8.11 mmol, 1.00 equiv) was added to a flame-dried round bottom flask equipped with a stir bar and septum. The flask was removed from the glovebox, 10 mL of anhydrous THF (10 mL) was added, and the reaction cooled to –78 °C in a dry ice/acetone bath. 6,7-Dihydro-4(5H)-benzofuranone (1.10 g, 8.11 mmol, 1.00 equiv) was added as a solution in THF (10 mL). The mixture was allowed to stir for 1 h at this temperature before slow addition of 4-iodo-1-butene (1.49 g, 8.19 mmol, 1.01 equiv). The reaction was allowed to warm to room temperature over night and was quenched with sat. NH_4Cl. The mixture was extracted with Et_2O (3 x 25 mL) and the combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash column chromatography (5% EtOAc/hexanes) to afford the title compound as a slightly yellow oil (0.771 g, 4.01 mmol, 50%). **TLC R_f** = 0.4 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.31 (d, J = 1.1 Hz, 1H), 6.66 (d, J = 1.4 Hz, 1H), 5.83 (ddt, J = 17.1, 10.5, 3.1 Hz,

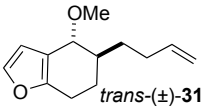
1H), 5.06 (ad, J = 17.1, 1.1 Hz, 1H), 4.99 (d, J = 10.3 Hz, 1H), 2.84 (dt, J = 17.4, 5.4 Hz, 1H), 2.86 (ddd, J = 17.2, 8.7, 5.5 Hz, 1H), 2.45–3.78 (m, 1H), 2.32–1.90 (m, 5H), 1.56–1.47 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 196.6, 166.3, 142.8, 138.3, 120.8, 115.2, 106.9, 45.9, 31.5, 28.4, 27.8, 22.5; **IR** (neat) 2939, 1681, 1600 cm^{-1}; **HRMS** (TOF MS CI+) m / z calcd for $C_{12}H_{14}O_2H$ $(M + H)^+$ 191.1072, found 191.1074.



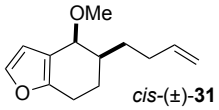
***rac*-SI-39.** Using representative procedure E outlined above, the following amounts of reagents were used: ketone **SI-38** (0.227 g, 1.19 mmol, 1.00 equiv), MeOH (8 mL), sodium borohydride (0.108 g, 2.86 mmol, 2.40 equiv). The product was purified by flash column chromatography (8–15% EtOAc/hexanes) to afford a 1:1 mixture of diastereomers as a clear, colorless oil (0.224 g, 1.17 mmol, 98%). The diastereomeric ratio was determined based on integration of the benzylic methines in the ^{1}H NMR spectrum. The diastereomers were not assigned cis and trans configurations, but were carried forward as a mixture. **TLC R_f** = 0.2 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.26 (d, J = 1.8 Hz, 1H), 6.39 (dd, J = 9.5, 1.5 Hz, 1H), 5.84 (ddt, J = 17.0, 10.5, 3.8 Hz, 1H), 5.05 (adq, J = 17.0, 1.5 Hz, 1H), 4.97 (d, J = 10.0 Hz, 1H), 4.57 and 4.36 (s, 1H), 2.72–2.50 (m, 2H), 2.27–2.15 (m, 1H), 2.15–2.03 (m, 1H), 1.79–1.43 (m, 5H), 1.38–1.24 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.2, 152.0, 141.5, 141.4, 138.9, 138.8, 120.3, 120.0, 114.84, 114.83, 109.8, 109.4, 69.5, 64.7, 42.5, 39.7, 31.7, 31.5, 30.7, 30.2, 24.7, 23.7, 23.4, 21.5; **IR** (neat) 3327, 3075, 2928 cm^{-1}; **HRMS** (TOF MS CI+) m / z calcd for $C_{12}H_{16}O_2H$ $(M + H)^+$ 193.1228, found 193.1224.



***trans*- and *cis*-(±)-31.** Using representative procedure G outlined above, the following amounts of reagents were used: alcohol *rac*-**SI-39** (0.202 g, 1.09 mmol, 1.00 equiv), NaH (39 mg, 1.6 mmol, 1.5 equiv), methyl iodide (0.090 mL, 1.4 mmol, 1.3 equiv) and DMF (8 mL). The products were purified and separated by flash column chromatography (1–5% Et_2O/hexanes) to afford the title compounds as colorless oils: *trans*-(±)-**27** (92 mg, 0.44 mmol, 41%) and *cis*-(±)-**27** (86 mg, 0.42 mmol, 39%).



***trans*-(±)-31.** **TLC R_f** = 0.2 (5% Et_2O/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.27 (s, 1H), 6.36 (s, 1H), 5.81 (ddt, J = 17.1, 10.4, 3.6 Hz, 1H), 5.03 (ad, J = 17.1 Hz, 1H), 4.96 (d, J = 10.3 Hz, 1H), 4.01 (d, J = 4.0 Hz, 1H), 3.30 (s, 3H), 2.62–2.50 (m, 2H), 2.23–2.05 (m, 3H), 2.02–1.94 (m, 1H), 1.70–1.62 (m, 1H), 1.53–1.44 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 152.5, 141.0, 138.7, 116.7, 114.8, 110.5, 77.2, 56.1, 37.2, 31.8, 29.4, 23.4, 20.4; **IR** (neat) 2975, 2929, 2818 cm^{-1}; **HRMS** (TOF MS CI+) m / z calcd for $C_{12}H_{16}O_2H$ $(M)^+$ 206.1307, found 206.1298.



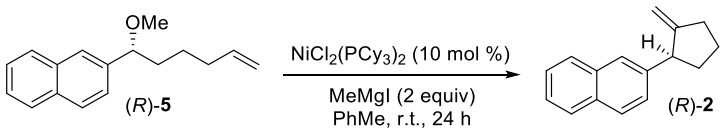
cis-(±)-**31**

***cis*-(±)-31. TLC R_f** = 0.3 (5% Et_2O/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.26 (s, 1H), 6.35 (s, 1H), 5.84 (ddt, *J* = 17.1, 10.4, 3.4 Hz, 1H), 5.04 (ad, *J* = 17.1 Hz, 1H), 4.96 (d, *J* = 10.3 Hz, 1H), 4.06 (ad, *J* = 1.8 Hz, 1H), 3.38 (s, 3H), 2.69 (dd, *J* = 17.1, 5.8 Hz, 1H), 2.54 (ddd, *J* = 17.1, 10.6, 6.4 Hz, 1H), 2.21–2.08 (m, 2H), 1.93–1.81 (m, 1H), 1.77–1.64 (m, 3H), 1.51–1.40 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 153.3, 140.7, 139.1, 117.8, 114.6, 110.8, 77.3, 56.7, 39.2, 31.6, 30.4, 24.2, 23.2.

Relative stereochemical configurations for *trans*-(±)-**31** and *cis*-(±)-**31** were assigned based on the coupling constants of the protons of the benzylic methines.[25] For *trans*-(±)-**31,** *J* = 4.0 Hz, and for *cis*-(±)-**31**, *J* = 1.8 Hz.

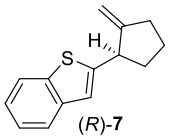
V. NICKEL-CATALYZED HECK CYCLIZATIONS OF BENZYLIC ETHERS AND CHARACTERIZATION DATA FOR PRODUCTS

***General procedure I**. Heck cyclization of benzylic ethers*



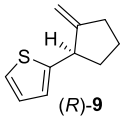
(*R*)-2. In a glovebox, a flame fried 7 mL vial equipped with a stir bar was charged with (*R*)-**5** (47 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). The reaction was stirred 24 h before removing the vial from the glovebox, opening to atmosphere, quenching with isopropanol, and eluted through a silica gel plug (30% Et_2O/hexanes). The combined organics were concentrated in vacuo, internal standard was added (PhTMS, 17.2 μL, 0.100 mmol, 0.500 equiv), and ^{1}H NMR yield was collected. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (31 mg, 0.15 mmol, 74%, 99% ee). **TLC R_f** = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.79 (t, *J* = 9.3 Hz, 3H), 7.66 (s, 1H), 7.44 (t, *J* = 6.8 Hz, 1H), 7.41 (t, *J* = 7.0 Hz, 1H), 7.33 (d, *J* = 8.2 Hz, 1H), 5.01 (s, 1H), 4.56 (s, 1H), 3.72 (at, *J* = 8.2 Hz, 1H), 2.63–2.49 (m, 2H), 2.26–2.18 (m, 1H), 1.96–1.81 (m, 2H), 1.78–1.66 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.2, 142.5, 133.6, 132.3, 128.1, 127.72, 127.67, 126.88, 126.87, 126.0, 125.3, 107.7, 51.6, 36.6, 33.7, 25.0; **IR** (neat) 3070, 2954, 2865, 1600 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{16}H_{16}H$ $(M + H)^+$ 209.1330, found 209.1326; **$[\alpha]^{24}_D$** –126 (*c* 1.4, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 99% ee: t_R (major) = 6.2 minutes, t_R (minor) = 5.8 minutes.

Table 2, entry 2. Using representative procedure I outlined above, the following amounts of reagents were used: (*R*)-**5** (0.24 g, 1.0 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (69 mg, 0.10 mmol, 0.10 equiv), PhMe (3.0 mL) and methylmagnesium iodide (1.0 mL, 2.0 M in Et_2O, 2.0 mmol, 2.0 equiv). The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (149 mg, 0.73 mmol, 73%, 89% ee).



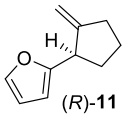
(*R*)-7. Using representative procedure I outlined above, the following amounts of reagents were used: (*R*)-**6** (49 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (35 mg, 0.16 mmol, 81%, 97% ee). **TLC R_f** = 0.6 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.30 (t, *J* = 7.3 Hz, 1H), 7.24 (t, *J* = 7.3 Hz, 1H), 7.09 (s, 1H), 5.07 (s, 1H), 4.88 (s, 1H), 3.93 (at, *J* = 7.3 Hz, 1H), 2.55–2.47 (m, 2H), 2.30–2.21 (m, 1H), 1.95–1.85 (m, 2H), 1.77–1.67 (m, 1H); **^{13}C**

NMR (125 MHz, $CDCl_3$) δ 154.8, 149.7, 140.1, 139.6, 124.1, 123.6, 122.9, 122.4, 120.9, 108.4, 46.8, 37.0, 32.9, 24.9; **IR** (neat) 3069, 2955, 2866, 1436 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{14}H_{14}SH$ $(M + H)^+$ 215.0892, found 215.0893; $[\alpha]^{26}_D$ –162 (*c* 1.3, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 97% ee: t_R (major) = 8.1 minutes, t_R (minor) = 7.1 minutes.



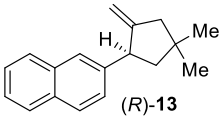
(*R*)-**9**

(*R*)-9. Using representative procedure I outlined above, the following amounts of reagents were used: (*R*)-**8** (37 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). Care must be taken during workup as the product is relatively volatile. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (24 mg, 0.15 mmol, 73%, 97% ee). **TLC R_f** = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.15 (d, *J* = 5.0 Hz, 1H), 6.94 (t, *J* = 4.9 Hz, 1H), 6.86 (d, *J* = 2.9 Hz, 1H), 5.01 (s, 1H), 4.80 (s, 1H), 3.86 (at, *J* = 7.3 Hz, 1H), 2.55–2.45 (m, 2H), 2.28–2.19 (m, 1H), 1.91–1.78 (m, 2H), 1.74–1.62 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.5, 148.6, 126.6, 124.3, 123.4, 107.7, 46.1, 37.3, 32.7, 24.6; **IR** (neat) 3072, 2957, 1440 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{10}H_{12}SH$ $(M + H)^+$ 165.0738, found 165.0739; $[\alpha]^{27}_D$ –164 (*c* 1.0, $CHCl_3$); **SFC** analysis (OJ-H, 2% IPA, 2.5 mL/min) indicated 97% ee: t_R (major) = 3.4 minutes, t_R (minor) = 3.6 minutes.



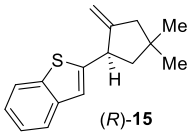
(*R*)-**11**

(*R*)-11. Using representative procedure I outlined above, the following amounts of reagents were used: (*R*)-**10** (36 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). Care must be taken while working up the product as it relatively volatile. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless and aromatic oil (22 mg, 0.15 mmol, 75%, 93% ee). **TLC R_f** = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (s, 1H), 6.29 (s, 1H), 6.05 (d, *J* = 2.6 Hz, 1H), 5.00 (s, 1H), 4.83 (s, 1H), 3.67 (t, *J* = 8.3 Hz, 1H), 2.44 (at, *J* = 6.6 Hz, 2H), 2.13–2.04 (m, 1H), 1.96–1.79 (m, 2H), 1.72–1.61 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 157.7, 153.3, 141.3, 110.1, 107.4, 105.0, 43.9, 32.92, 32.91, 24.9; **IR** (neat) 3075, 2959, 2870 cm^{-1}; **HRMS** (TOF MS CI+) *m / z* calcd for $C_{10}H_{12}OH$ $(M + H)^+$ 149.0966, found 149.0971; $[\alpha]^{24}_D$ –133 (*c* 0.7, $CHCl_3$); **GC** analysis: 93% ee (CYCLODEX B, inlet temp 220 °C, flow rate 5.3781 mL/min, initial temp 55 °C, hold 2 min, ramp 10 °C/min up to 180 °C, hold 3 min, ramp 40 °C/min up to 230 °C, hold 1 min, t_{R1} = 15.29 min, t_{R2} = 15.48.



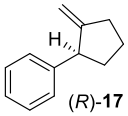
(*R*)-**13**

(*R*)-13. Using representative procedure I outlined above, the following amounts of reagents were used: (*R*)-**12** (54 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a white solid (45 mg, 0.19 mmol, 95%, 88% ee). **TLC R_f** = 0.8 (100% pentane, UV active, stain with $KMnO_4$); **m.p.** 84–86 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.83–7.73 (m, 3H), 7.66 (s, 1H), 7.47–7.38 (m, 2H), 7.33 (dd, *J* = 8.6, 1.1 Hz, 1H), 5.00 (s, 1H), 4.58 (s, 1H), 3.94 (tt, *J* = 10.5, 2.5 Hz, 1H), 2.44 (ad, *J* = 16.3 Hz, 1H), 2.34 (d, *J* = 16.3 Hz, 1H), 1.99 (dd, *J* = 12.3, 8.3 Hz, 1H), 1.76 (at, *J* = 12.3 Hz, 1H), 1.17 (s, 3H), 1.09 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.6, 142.9, 133.6, 132.3, 128.1, 127.73, 127.66, 126.8, 126.7, 126.0, 125.3, 108.7, 51.0, 50.1, 49.2, 37.8, 29.3, 27.6; **IR** (neat) 3073, 2948, 2861, 1599 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{18}H_{20}H$ $(M + H)^+$ 237.1643, found 237.1650; **$[\alpha]^{26}_D$** –106 (*c* 1.7, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 88% ee: t_R (major) = 4.2 minutes, t_R (minor) = 3.7 minutes.



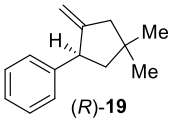
(*R*)-**15**

(*R*)-15. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**14** (55 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), and methylmagnesium iodide (0.16 mL, 2.5 M in Et2O, 0.40 mmol, 2.0 equiv). The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (36 mg, 0.15 mmol, 74%, 84% ee). **TLC R_f** = 0.9 (100% pentane, UV active); **^{1}H NMR** ($CDCl_3$, 400 MHz) δ 7.75 (d, *J* = 7.9 Hz, 1H), 7.66 (d, *J* = 7.8 Hz, 1H), 7.27 (m, 2H), 7.10 (s, 1H), 5.06 (m, 1H), 4.87 (m, 1H), 4.17–4.09 (m, 1H), 2.40 (dq, *J* = 15.8, 2.5 Hz, 1H), 2.28 (dd, *J* = 15.8, 1.8 Hz, 1H), 2.08 (ddd, *J* = 12.5, 8.0, 1.9 Hz, 1H), 1.81 (dd, *J* = 12.4, 10.8 Hz, 1H), 1.15 (s, 3H), 1.06 (s, 3H); **^{13}C NMR** ($CDCl_3$, 100 MHz) δ 154.9, 150.2, 140.2, 139.6, 124.2, 123.7, 123.0, 122.5, 120.7, 109.3, 51.1, 48.4, 45.5, 37.9, 29.2, 27.5; **IR** (neat) 3058, 2952, 2865, 1655, 1457, 885 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{16}H_{18}SH$ $(M + H)^+$ 243.1207, found 243.1206; **$[\alpha]^{27}_D$** –94 (*c* 1.7, $CHCl_3$); **SFC** analysis (OJ-H, 20% hexanes, 3.0 mL/min) indicated 84% ee: tR (major) = 6.3 minutes, t_R (minor) = 5.5 minutes.



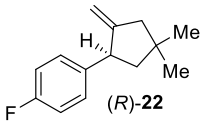
(*R*)-**17**

(*R*)-17. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**16** (38 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.15 mL, 2.7 M in Et_2O, 0.40 mmol, 2.0 equiv) and magnesium iodide (56 mg, 0.20 mmol, 1.00 equiv). Care must be taken during workup as the product is relatively volatile. Yield as determined by ^{1}H NMR with PhTMS as internal standard was 64%. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (12 mg, 0.073 mmol, 36%, 93% ee). **TLC R_f** = 0.8 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.3 Hz, 2H), 7.24–7.16 (m, 3H), 4.97 (s, 1H), 4.53 (s, 1H), 3.55 (t, *J* = 7.9 Hz, 1H), 2.58–2.44 (m, 2H), 2.21 (m, 1H), 1.91–1.83 (m, 1H), 1.82–1.71 (m, 1H), 1.71–1.64 (m, 1H), 1.06 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.7, 145.1, 128.41, 128.39, 126.1, 107.4, 51.4, 36.6, 33.6, 29.9, 24.9; **IR** (neat) 3028, 2952, 1601 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{12}H_{14}H$ $(M + H)^+$ 159.1174, found 159.1168; **$[\alpha]^{25}_D$** –93 (*c* 0.5, $CHCl_3$); **SFC** analysis (AD-H, 10% IPA, 2.5 mL/min) indicated 93% ee: t_R (major) = 1.7 minutes, t_R (minor) = 1.9 minutes.



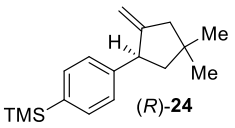
(*R*)-**19**

(*R*)-19. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**20** (52 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv) and magnesium iodide (56 mg, 0.20 mmol, 1.00 equiv). Care must be taken during workup as the product is relatively volatile. Yield as determined by ^{1}H NMR with PhTMS as internal standard was 81%. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (25 mg, 0.13 mmol, 67%, 92% ee). **TLC R_f** = 0.8 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.6 Hz, 2H), 7.24–7.15 (m, 3H), 4.96 (s, 1H), 4.55 (s, 1H), 3.76 (tt, *J* = 10.6, 2.2 Hz, 1H), 2.38 (ad, *J* = 15.9 Hz, 1H), 2.29 (d, *J* = 15.9 Hz, 1H), 1.94 (dd, *J* = 12.5, 8.3 Hz, 1H), 1.76 (at, *J* = 12.0 Hz, 1H), 1.14 (s, 3H), 1.06 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.8, 145.6, 128.4, 128.3, 126.1, 108.3, 51.1, 49.9, 49.2, 37.7, 29.3, 27.6; **IR** (neat) 3027, 3063, 2952, 1602 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{14}H_{18}H$ $(M + H)^+$ 187.1487, found 184.1479; **$[\alpha]^{25}_D$** –126 (*c* 1.0, $CHCl_3$); **SFC** analysis (OJ-H, 4% IPA, 2.5 mL/min) indicated 92% ee: t_R (major) = 1.7 minutes, t_R (minor) = 1.6 minutes.



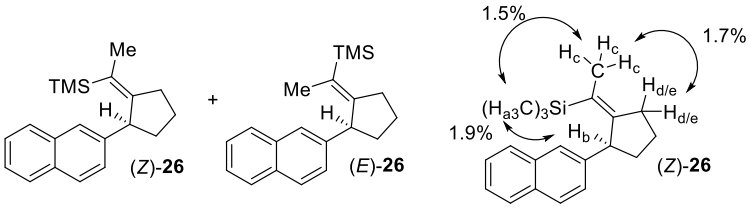
(*R*)-**22**

(*R*)-22. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**21** (56 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv) and magnesium iodide (56 mg, 0.20 mmol, 1.00 equiv). Care must be taken during workup as the product is relatively volatile. Yield as determined by ^{1}H NMR with PhTMS as internal standard was 77%. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a colorless solid (23 mg, 0.11 mmol, 57%, 90% ee). **TLC R_f** = 0.6 (100% pentane, UV active, stain with $KMnO_4$); **m.p.** 32–35 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.25–7.19 (m, 2H), 7.03 (at, *J* = 8.7 Hz, 2H), 5.02 (s, 1H), 4.59 (s, 1H), 3.81 (tt, *J* = 8.6, 2.2 Hz, 1H), 2.46–2.31 (m, 2H), 1.99 (ddd, *J* = 12.2, 8.1, 1.2 Hz, 1H), 1.67 (at, *J* = 11.9 Hz, 1H), 1.20 (s, 3H), 1.11 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 161.3 (d, *J* = 243 Hz), 156.6, 141.0 (d, *J* = 3 Hz), 129.5 (d, *J* = 8 Hz), 115.0 (d, *J* = 21 Hz), 108.3, 51.1, 49.1, 48.9, 37.5, 29.2, 27.5, ; **IR** (neat) 2952, 1653, 1603, 1508, 1463, 1223, 815 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{14}H_{17}F$ $(M)^+$ 204.1314, found 204.1316; **$[\alpha]^{27}_D$** –90 (*c* 1.4, $CHCl_3$); **SFC** analysis (OJ-H, 4% IPA, 2.0 mL/min) indicated 90% ee: t_R (major) = 2.1 minutes, t_R (minor) = 2.3 minutes.

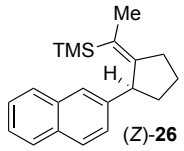


(*R*)-**24**

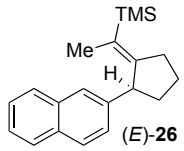
(*R*)-24. Using representative procedure I outlined above with the exception of adding magnesium iodide in the glovebox and heating the sealed reaction vial outside of the glovebox at 65 °C, the following amounts of reagents were used: (*R*)-**23** (67 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv) and magnesium iodide (56 mg, 0.20 mmol, 1.00 equiv). The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (44 mg, 0.17 mmol, 85%, 92% ee). **TLC R_f** = 0.3 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 4.97 (s, 1H), 4.57 (s, 1H), 3.78–3.72 (m, 1H), 2.38 (ad, *J* = 15.7 Hz, 1H), 2.28 (ad, *J* = 15.7 Hz, 1H), 1.93 (dd, *J* = 12.2, 8.3 Hz, 1H), 1.66 (t, *J* = 11.9 Hz, 1H), 1.13 (s, 3H), 1.05 (s, 3H), 0.25 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 156.5, 146.2, 137.5, 133.4, 127.6, 108.3, 50.9, 49.7, 49.2, 37.6, 29.2, 27.4, –1.0; **IR** (neat) 3067, 2952, 1653, 1600, 1247, 1109, 835 cm^{-1}; **HRMS** (TOF MS CI+) *m/z* calcd for $C_{17}H_{26}SiH$ $(M + H)^+$ 259.1882, found 259.1877; **$[\alpha]^{27}_D$** –109 (*c* 1.6, $CHCl_3$); **SFC** analysis (OJ-H, 3% hexanes, 2.0 mL/min) indicated 92% ee: t_R (major) = 2.6 minutes, t_R (minor) = 2.2 minutes



26. Using representative procedure I outlined above with the exception of heating the sealed reaction vial outside of the glovebox at 60 °C for 48 h, the following amounts of reagents were used: **(*R*)-25** (62 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). The product was purified by flash column chromatography (100% pentane) to afford the title compound as a 2:1 Z:E mixture of stereoisomers (62% by ^{1}H NMR with PhTMS as internal standard). The benzylic methane was integrated to determine the ratio of stereoisomers. Flash column chromatography with silver-impregnated silica gel (100% pentane) was used to separate the stereoisomers to afford (*Z*)-**26** (20 mg, 0.068 mmol, 34%) and (*E*)-**26** (10 mg, 0.034 mmol, 17%). The olefin geometry was assigned based on the nOe correlations shown for (*Z*)-**26** (see section X for relevant spectra).

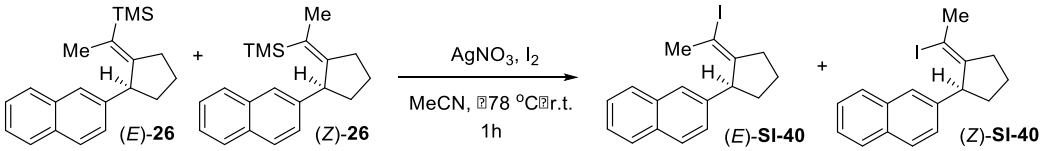


(*Z*)-26. TLC R_f = 0.8 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.8 Hz, 1H), 7.75 (d, *J* = 7.8 Hz, 2H), 7.47–7.37 (m, 3H), 7.29 (d, *J* = 7.3 Hz, 1H), 4.11 (d, *J* = 7.1 Hz, 1H), 2.58 (d, *J* = 17.2, 9.2 Hz, 1H), 2.48–2.38 (m, 1H), 2.12–2.01 (m, 1H), 1.94–1.86 (m, 1H), 1.83 (s, 3H), 1.71–1.63 (m, 1H), 1.57–1.45 (m, 1H), –0.13 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.0, 144.6, 133.4, 132.0, 128.3, 127.80, 127.75, 127.6, 127.2, 126.0, 125.9, 125.2, 49.6, 37.1, 32.5, 22.1, 19.2, –0.3; **IR** (neat) 3052, 2952, 1600 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{20}H_{26}Si$ $(M)^+$ 294.1804, found 294.1798. **$[\alpha]^{24}_D$** –258 (*c* 0.8, $CHCl_3$).

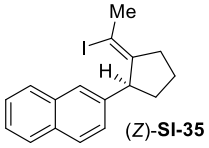


(*E*)-26. TLC R_f = 0.8 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.7 Hz, 1H), 7.75 (d, *J* = 7.7 Hz, 2H), 7.53 (s, 1H), 7.46–7.37 (m, 2H), 7.30 (d, *J* = 8.3 Hz, 1H), 4.07 (br s, 1H), 2.62–2.50 (m, 2H), 2.23–2.15 (m, 1H), 1.80–1.61 (m, 3H), 1.42 (s, 3H), 0.17 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 155.5, 143.9, 133.7, 132.0, 128.0, 127.9, 127.71, 127.68, 126.8, 125.9, 125.5, 125.1, 49.6, 37.1, 34.9, 25.2, 19.4, –0.2; **IR** (neat) 3053, 2951, 1599 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{20}H_{26}SiH$ $(M + H)^+$ 295.1882, found 295.1887. **$[\alpha]^{24}_D$** –88 (*c* 0.3, $CHCl_3$).

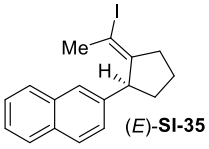
Enantiomers of **26** could not be separated by SFC. Derivatives of **26** were prepared to determine the enantiospecificity of the alkyne insertion/Kumada coupling of **25**.



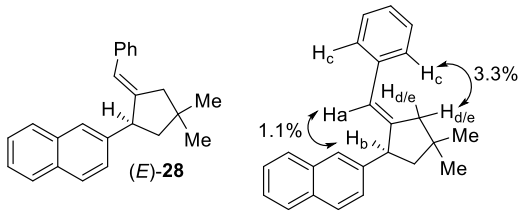
SI-40. The title compound was prepared according to a modified procedure reported by Xi.[26] Under an atmosphere of nitrogen, $AgNO_3$ (73 mg, 0.43 mmol, 1.2 equiv) was added to a solution of **(*Z*)-26** and **(*E*)-26** as a 2:1 mixture (110 mg, 0.360 mmol, 1.00 equiv) in MeCN (10 mL) at –78 °C. The solution was stirred for 15 min before addition of I_2 (119 mg, 0.470 mmol, 1.30 equiv). The reaction was allowed to warm to 0 °C in an ice water bath over 45 min and quenched with water. The mixture was extracted with Et_2O and the combined organics were washed with brine, dried with $MgSO_4$, filtered, and concentrated in vacuo. Flash column chromatography afforded (*Z*)-**SI-40** as a clear, colorless oil (46 mg, 0.12 mmol, 50%, 89% ee) and (*E*)-**SI-40** (24 mg, .069 mmol, 58%).



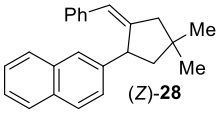
(*Z*)-SI-40. TLC R_f = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.83–7.74 (m, 3H), 7.52 (s, 1H), 7.49–7.39 (m, 2H), 7.28 (d, *J* = 8.6 Hz, 1H), 4.06 (at, *J* = 5.1 Hz, 1H), 2.70–2.59 (m, 1H), 2.58–2.49 (m, 1H), 2.37 (ddd, *J* = 15.1, 12.5, 7.4 Hz, 1H), 2.20 (s, 3H), 2.01–1.93 (m, 1H), 1.82–1.64 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 150.6, 142.2, 133.6, 132.2, 128.4, 127.8, 127.7, 126.23, 126.17, 125.53, 125.50, 92.3, 49.2, 42.7, 39.6, 31.1, 23.9; **IR** (neat) 3051, 2953, 1600, 849 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{17}H_{17}I$ $(M)^+$ 348.0375, found 348.0375. **$[\alpha]^{23}_D$** –217 (*c* 2.3, $CHCl_3$); **SFC** analysis (OJ-H, 10% IPA, 2.5 mL/min) indicated 89% ee: t_R (major) = 7.1 minutes, t_R (minor) = 8.6 minutes.



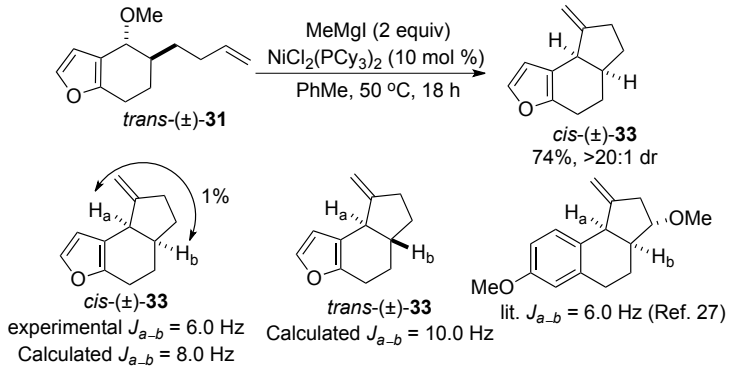
(*E*)-SI-40. TLC R_f = 0.8 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.79 (t, *J* = 8.0 Hz, 3H), 7.53 (s, 1H), 7.46–7.38 (m, 2H), 7.30 (d, *J* = 8.3 Hz, 1H), 3.98 (d, *J* = 7.7 Hz, 1H), 2.68–2.51 (m, 5H), 2.27–2.16 (m, 1H), 1.92–1.77; **^{13}C NMR** (125 MHz, $CDCl_3$) δ 150.0, 142.0, 133.6, 132.1, 128.1, 127.8, 127.7, 126.9, 126.1, 125.9, 125.3, 91.1, 56.9, 36.6, 32.5, 31.1, 25.4; **IR** (neat) 2956, 1600, 904 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{17}H_{17}INH_4$ $(M + NH_4)^+$ 366.0719, found 366.0709. **$[\alpha]^{23}_D$** –9.0 (*c* 0.8, $CHCl_3$); **SFC** analysis (AD-H, 7% IPA, 2.5 mL/min) indicated 89% ee: t_R (major) = 6.1 minutes, t_R (minor) = 6.8 minutes.



(*E*)-28. Using representative procedure I outlined above, the following amounts of reagents were used: (*E*)-**27** (69 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a white solid (50 mg, 0.16 mmol, 80%, 89% ee). The olefin geometry was assigned based on the nOe correlations indicated above (see section X for relevant spectra). **TLC R_f** = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **m.p.** = 104–105 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.79 (t, *J* = 8.8 Hz, 3H), 7.72 (s, 1H), 7.49–7.40 (m, 2H), 7.36 (d, *J* = 8.4 Hz, 1H), 7.28 (t, *J* = 7.7 Hz, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.14 (t, *J* = 7.0 Hz, 1H), 5.98 (s, 1H), 4.16 (t, *J* = 9.5 Hz, 1H), 2.72 (d, *J* = 16.8 Hz, 1H), 2.66 (d, *J* = 16.8 Hz, 1H), 2.00 (dd, *J* = 12.1, 8.4 Hz, 1H), 1.79 (at, *J* = 12.1 Hz, 1H), 1.25 (s, 3H), 1.09 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 150.2, 143.1, 138.8, 133.6, 132.4, 128.3, 128.2, 127.8, 127.7, 127.2, 127.0, 126.1 (2C), 125.4, 125.1, 52.2, 50.4, 47.8, 38.8, 29.6, 27.8; **IR** (neat) 3053, 2951, 2865, 1600 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{24}H_{24}NH_4$ $(M + NH_4)^+$ 330.2222, found 330.2213; **$[\alpha]^{23}_D$** +133 (*c* 1.0, $CHCl_3$); **SFC** analysis (AS-H, 4% IPA, 2.5 mL/min) indicated 89% ee: t_R (major) = 5.4 minutes, t_R (minor) = 6.0 minutes.



(*Z*)-28. Using representative procedure I outlined above, the following amounts of reagents were used: (Z)-**27** (10:1 Z:E) (69 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL) and methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). The product was purified by flash column chromatography with silver-impregnated silica gel (0–2%% Et_2O/pentane) to afford the title compound as a white solid (38 mg, 0.12 mmol, 60%, 9:1 Z:E). **TLC R_f** = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **m.p.** = 86–92 °C; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.77–7.67 (m, 3H), 7.59 (s, 1H), 7.43–7.34 (m, 2H), 7.32 (d, *J* = 8.7 Hz, 1H), 7.12–7.07 (m2H), 7.04 (t, *J* = 7.7 Hz, 2H), 6.96 (t, *J* = 6.8 Hz, 1H), 5.62 (s, 1H), 4.31 (t, *J* = 8.4 Hz, 1H), 2.75 (d, *J* = 14.7 Hz, 1H), 2.38 (d, *J* = 14.7 Hz, 1H), 2.25 (m1H), 1.71 (dd, *J* = 12.7, 8.4 Hz, 1H), 1.09 (s, 3H), 1.04 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 147.6, 143.5, 137.4, 133.9, 132.0, 128.51, 128.48, 128.0, 127.8, 127.7, 126.3, 126.0, 125.9, 125.2, 125.1, 124.8, 53.2, 52.9, 46.9, 37.6, 28.9, 27.3.



***trans*-(±)-33.** Using representative procedure I outlined above with the exception of heating the sealed reaction vial outside of the glovebox at 50 °C, the following amounts of reagents were used: *trans*-(±)-**31** (41 mg, 0.20 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (14 mg, 0.020 mmol, 0.10 equiv), PhMe (1.6 mL), methylmagnesium iodide (0.16 mL, 2.5 M in Et_2O, 0.40 mmol, 2.0 equiv). Care must be taken during workup as the product is relatively volatile. The product was purified by flash column chromatography with silver-impregnated silica gel (100% pentane) to afford the title compound as a clear, colorless oil (26 mg, 0.15 mmol, 74%, >20:1 dr). **TLC R_f** = 0.7 (100% pentane, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.26 (s, 1H), 6.34 (d, J = 1.1 Hz, 1H), 4.96 (s, 1H), 4.93 (s, 1H), 3.36 (d, J = 6.0 Hz, 1H), 2.58 (t, J = 6.3 Hz, 2H), 2.43 (t, J = 7.7 Hz, 2H), 2.39–2.31 (m, 1H), 1.88–1.66 (m, 3H), 1.61–1.51 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 154.9, 149.9, 140.6, 117.8, 110.6, 106.8, 42.8, 38.8, 30.9, 28.2, 25.4, 21.3; **IR** (neat) 2931, 2850, 1505 cm^{-1}; **HRMS** (TOF MS CI+) m / z calcd for $C_{12}H_{14}OH$ $(M + H)^+$ 175.1123, found 175.1132.

NOESY and 1D nOe experiments show a strong nOe correlation between H_a and H_b, consistent with a cis orientation (see section X for relevant spectra). The J-coupling between the protons indicated is consistent with literature values for structurally similar compounds with a cis relationship.[27] The experimentally measured J-coupling constants are consistent with computational calculations. All calculations were performed with Guassian 09.[28] The geometries of **33** were optimized with the B3LYP functional using the 6–31G(d,p) basis set.[29] J-coupling constants were obtained with the B3LYP functional using the 6–31+G(d,p) basis set with the SMD solvation model for chloroform.[30]

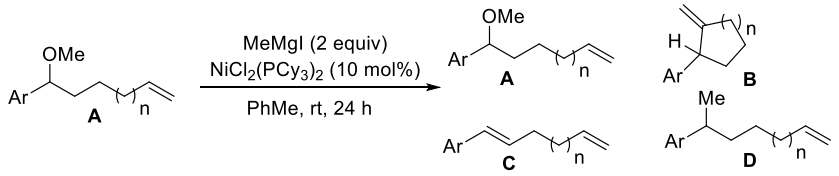
VI. ADDITIONAL INFORMATION REGARDING SUBSTRATE SCOPE

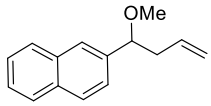
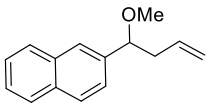
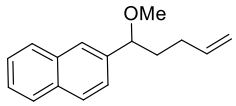
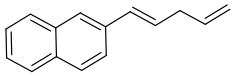
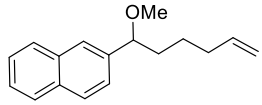
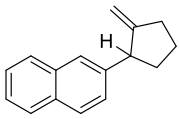
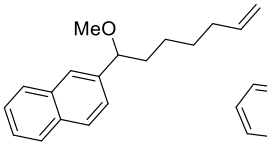
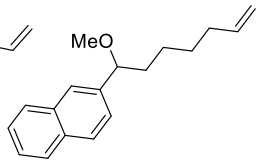
Representative examples of other substrates are presented to provide an idea of the scope and limitations with respect to formation of different ring sizes, functional group compatibility, and olefin substitution patterns.

i. Tether length.

The length of the alkane tether was varied to determine if alternate rings sizes could be formed in the Heck cyclization. When the tether length is too small, we observe only recovered starting material when the substrate is subjected to cyclization conditions (Table SI-1, entry 1). Interestingly, no β-hydrogen elimination to form a conjugated styrenyl diene is detected by ^{1}H NMR spectroscopy. When the length of the alkane tether is such that 4-exo or 5-endo cyclization could occur, we observe high conversion to the elimination product (entry 2). The Kumada cross-coupling product is also observed in modest yields for the first time using $NiCl_2(PCy_3)_2$ as the precatalyst. When the tether is the appropriate length to form 6-membered rings in the Heck cyclization, only recovered starting material is observed.

Table SI-1 Tether length.



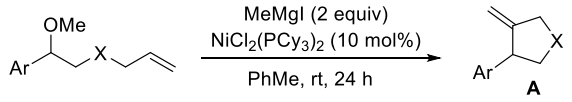
Entry	Substrate	Major Product	yield **A** (%)[a]	yield **B** (%)[a]	yield **C** (%)[a]	yield **D** (%)[a]
1			99	< 5	< 5	< 5
2			< 5	< 5	61	36
3			< 5	81	15	< 5
4			99	< 5	< 5	< 5

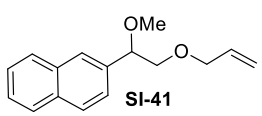
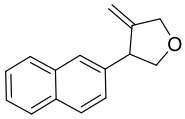
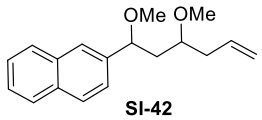
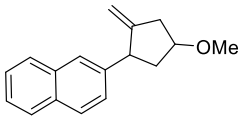
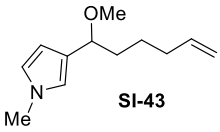
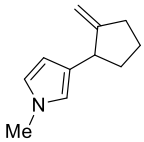
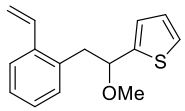
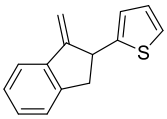
[a]Determined by ^{1}H NMR using an internal standard ($PhSiMe_3$).

ii. Functional group and substituent pattern compatibility

We illustrate representative examples of substituent patterns that provide low yields or no desired Heck product. Heck cyclization of substrates containing oxygenation in the tether failed to produce the desired tetrahydrofuran (Table SI-2, entry 1). 1,3-Diol derivatives (entry 2) provide low yields of the desired product. Significant decomposition stems from formation of an allylic ether byproduct that further reacts to a variety of products under the reaction conditions. Whereas substrates containing furan and thiophene moieties performed well under the reaction conditions, pyrrole **SI-43** decomposed under the conditions for the Heck cyclization (entry 3). Subjection of thiophene **SI-44** to cyclization conditions afforded the desired substituted indane in low yield. Elimination to form a stilbene was competitive with formation of desired product (~ 2:1 elimination : cyclization was observed).

Table SI-2. Representative examples of substituent patterns



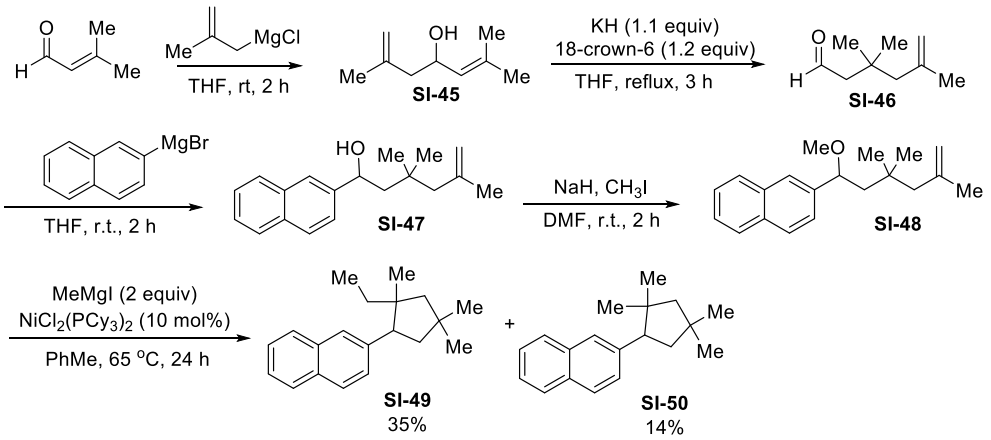
Entry	Substrate	Heck Product	yield **A** (%)[a]
1	 **SI-41**		< 5
2	 **SI-42**		23
3	 **SI-43**		< 5
4	 **SI-44**		18

[a]Determined by ^{1}H NMR using an internal standard ($PhSiMe_3$).

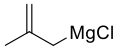
iii. Heck cyclization of 1,1-disubstituted olefin SI-48

A 1,1-disubstituted olefin (**SI-48**) was prepared as a test for formation of a quaternary center during the cyclization reaction. The 1,1-disubstituted olefin **SI-48** was prepared by Grignard addition into 3-methylcrotonaldehyde. Oxy-Cope rearrangement followed by Girgnard addition into the resultant aldehyde afforded alcohol **SI-47**, which upon methylation formed the 1,1-disubstituted olefin **SI-48**.

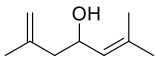
Scheme SI-5. Preparation and Heck cyclization of 1,1-disubstituted olefin SI-48



Cyclization of 1,1-disubstituted olefin **SI-48** was successful in providing products containing a quaternary stereocenter. However, two major products were formed: **SI-48**, from a Heck-Kumada sequence similar to that shown in Scheme 2, and **SI-50**, from a reductive Heck reaction. Unfortunately these products were inseparable; a combined yield of 49% was obtained.

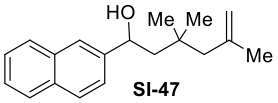


Methallylmagnesium chloride. The Grignard reagent was prepared according to a modified procedure by Moore.[31] A flame-dried 100 mL round-bottom flask was equipped with a stir bar and charged with magnesium turnings (0.91 g, 38 mmol, 1.3 equiv), THF (25 mL) and a catalytic amount of iodine. Neat methallyl chloride (2.5 mL, 30 mmol, 1.0 equiv) was added in one portion. The reaction flask was equipped with a reflux condenser and heated to reflux for 2 h, cooled to ambient temperature and titrated to 0.7 M.



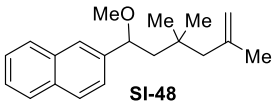
SI-45

SI-45. Using representative procedure A outlined above, the following amounts of reagents were used: 3-methyl-2-butenal (1.45 mL, 15.0 mmol, 1.00 equiv), methallylmagnesium chloride (0.7 M in THF, 29 mL, 20 mmol, 1.3 equiv). The product was purified by flash column chromatography (10% EtOAc/hexanes) to afford the title compound as a clear, colorless oil (1.76 g, 12.6 mmol, 84%). **TLC R_f** = 0.2 (10% EtOAc/hexanes, stained with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.19 (d, *J* = 8.7 Hz, 1H), 4.87 (s, 1H), 4.81 (s, 1H), 4.52–4.46 (m, 1H), 2.24 (dd, *J* = 13.8, 5.0 Hz, 1H), 2.16 (dd, *J* = 13.8, 4.4 Hz, 1H), 1.78 (s, 3H), 1.73, (s, 3H), 1.71 (s, 3H), 1.67 (br s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 142.6, 135.4, 127.5, 113.6, 66.1, 46.4, 25.9, 22.6, 18.3; **IR** (neat) 3346, 2876, 1698, 1600, 1365 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_9H_{16}ONH_4$ $(M + NH_4)^+$ 158.1545, found 158.1551.

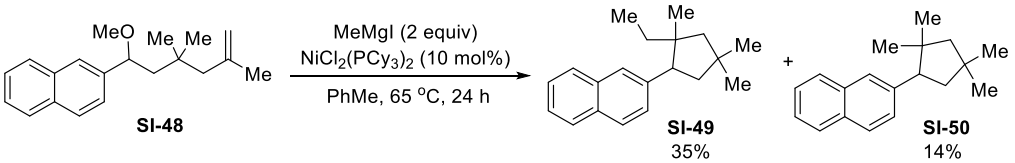


SI-47

SI-47. The alcohol was prepared according to a modified procedure by Maynard.[32] In a glovebox, a flame dried 50 mL round bottom flask equipped with a stir bar and septum was charged with potassium hydride (0.607 g, 15.1 mmol, 1.3 equiv). A separate 25 mL round bottom flask was charged with 18-crown-6 ether (3.60 g, 13.6 mmol, 1.17 equiv). Both flasks was removed from the glovebox and a solution of alcohol **SI-45** (1.633 g, 11.64 mmol, 1.000 equiv) and 18-crown-6 ether in THF (20 mL) was prepared. This mixture was added to the KH suspended in THF (5 mL) at 0 ^oC. The reaction was warmed to reflux and stirred for 3 h. Upon cooling, the reaction was quenched by pouring the contents of the reaction flask into a 250 mL round bottom flask containing 40 mL of sat. NH_4Cl. The mixture was extracted with ether (3 x 20 mL) and the combined organic layers were washed with brine (2 x 40 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. This crude mixture of aldehyde **SI-46** was then used immediately in the next step. The aldehyde was redissolved in anhydrous THF, and added to a solution of 2-naphthylmagnesium bromide (15 mL, 1.0 M in THF, 15 mmol, 1.3 equiv) at 0 ^oC. The reaction was allowed to stir for 1.5 h before it was quenched with sat. NH_4Cl. The mixture was once again extracted with ether (3 x 25 mL) and the combined organic layers were washed with brine (1 x 40 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The product was purified by flash column chromatography (2–8% EtOAc/hexanes) to afford the title compound as a thick oil (0.877 g, 3.27 mmol, 28% over two steps). **TLC R_f** = 0.3 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85–7.80 (m, 3H), 7.78 (s, 1H), 7.50–7.43 (m, 3H), 5.03 (d, *J* = 8.3 Hz, 1H), 4.87 (s, 1H), 4.68 (s, 1H), 2.09 (dd, *J* = 15.5, 12.8 Hz, 2H), 1.88 (dd, *J* = 14.5, 8.3 Hz, 1H), 1.83 (s, 1H), 1.79 (s, 3H), 1.73 (dd, *J* = 14.5, 3.2 Hz, 1H), 1.07 (s, 3H), 1.03 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 144.0, 143.6, 133.5, 133.0, 128.5, 128.1, 127.8, 126.3, 125.9, 124.34, 124.33, 114.7, 72.4, 51.6, 50.9, 34.2, 28.3, 28.2, 25.7; **IR** (neat) 3346, 2876, 1698, 1600, 1365 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{19}H_{24}ONa$ $(M + Na)^+$ 291.1725, found 291.1714.



SI-48. Using representative procedure G outlined above, the following amounts of reagents were used: alcohol **(*R*)-SI-42** (0.241 g, 0.900 mmol, 1.00 equiv), NaH (48 mg, 2.0 mmol, 2.2 equiv), methyl iodide (0.073 mL, 1.2 mmol, 1.3 equiv) and DMF (4 mL). The product was purified by flash column chromatography (2–5% EtOAc/hexanes) to afford the title compound as a colorless oil (0.19 g, 0.67 mmol, 75%). **TLC R_f** = 0.7 (10% EtOAc/hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.87–7.80 (m, 3H), 7.71 (s, 1H), 7.50–7.42 (m, 3H), 4.85 (s, 1H), 4.67 (s, 1H), 4.39 (dd, *J* = 8.5, 2.9 Hz, 1H), 3.18 (s, 3H), 2.06 (dd, *J* = 19.6, 13.2 Hz, 2H), 1.92 (dd, *J* = 14.8, 8.5 Hz, 1H), 1.78 (s, 3H), 1.58 (dd, *J* = 14.8, 3.1 Hz, 1H), 1.04 (s, 3H), 0.99 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 143.7, 141.4, 133.4, 133.1, 128.5, 127.94, 127.85, 126.2, 125.8, 125.5, 124.8, 114.6, 81.9, 56.4, 50.91, 50.89, 34.2, 28.2, 28.1, 25.1; **IR** (neat) 3055, 1602 cm^{-1}; **HRMS** (TOF MS CI+) *m* / *z* calcd for $C_{20}H_{26}O$ $(M)^+$ 282.1984, found 282.1988.

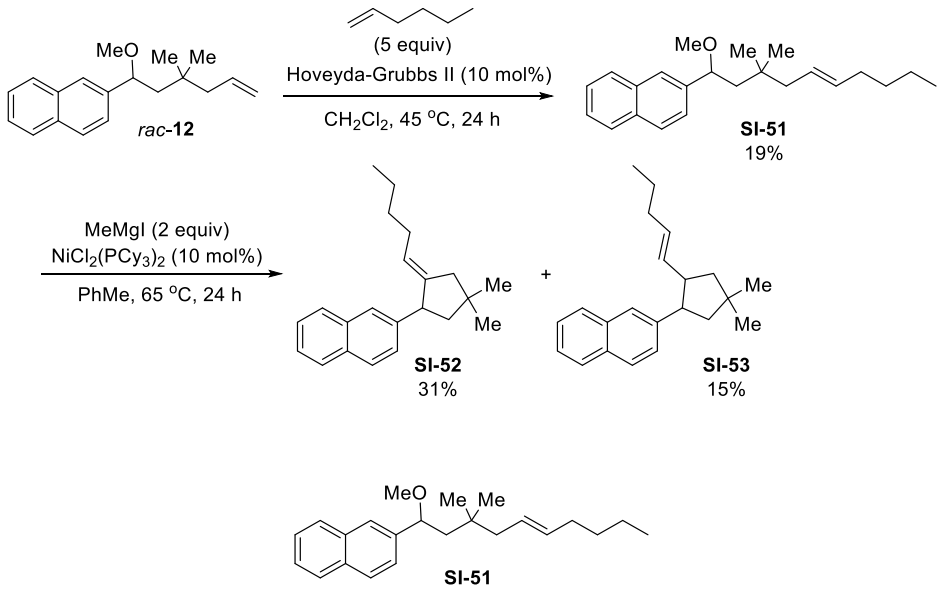


SI-49 and SI-50. Using representative procedure I outlined above, with the exception of heating to 65 °C, the following amounts of reagents were used: **SI-48** (27 mg, 0.10 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (7 mg, 0.010 mmol, 0.10 equiv), PhMe (0.8 mL) and methylmagnesium iodide (0.07 mL, 2.7 M in Et_2O, 0.20 mmol, 2.0 equiv). The products were purified by flash column chromatography on silver impregnated silica gel (100% pentane) to afford a 3.5:1 mixture of desired product **SI-49** and reduced product **SI-50** (13 mg, 0.049 mmol, 49%). Annoted ^{1}H NMR spectrum is provided in section X.

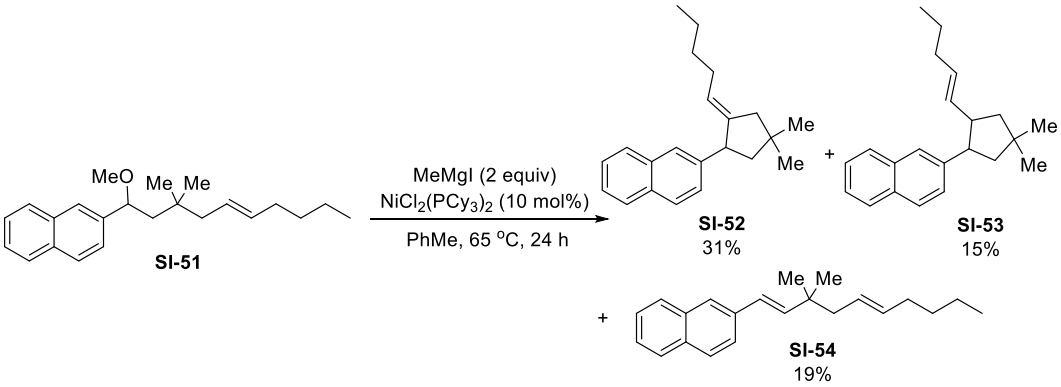
iv. Heck cyclization of a 1,2-dialkyl substituted olefin

The Heck cyclization of 1,2-disubstituted olefin **SI-51** was expected to provide a facile route to stereodefined trisubstituted olefins. Heck cyclization occurred smoothly, however, an unselective β-hydride elimination step resulted in an inseperable 2:1 mixture of olefin isomers (**SI-52** and **SI-53**, 46% combined yield).

Scheme SI-6. Preparation and Heck cyclization of 1,2-disubstituted olefin SI-51



SI-51. The title compound was prepared according to a modified procedure reported by Grubbs.[33] In a glovebox, a flame-dried bomb flask was charged with a stir bar, *rac*-**12** (0.336 g, 1.25 mmol, 1.00 equiv), and (1,3-Bis-(2,4,6-trimethylphenyl)-2-imidazolidinylidene)dichloro(o-isopropoxyphenylmethylene)ruthenium (78 mg, 0.13 mmol, 0.10 equiv). The flask was removed from the glovebox, and anhydrous CH_2Cl_2 (25 mL) and 1-hexene (0.78 mL, 6.3 mmol, 5.0 equiv) was added. The flask was sealed and heated to reflux 15 h. The flask was then cooled to ambient temperature, and the solvent was removed in vacuo. The residue was purified by flash column chromatography on silver impregnated silica gel to afford the title compound as a colorless oil (77 mg, 0.24 mmol, 19%). **TLC R_f** = 0.6 (100% hexanes, UV active); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.86–7.78 (m, 3H), 7.70 (s, 1H), 7.50–7.40 (m, 3H), 5.47–5.35 (m, 2H), 4.37 (dd, *J* = 8.6, 2.8 Hz, 1H), 3.18 (s, 3H), 2.03–1.93 (m, 4H), 1.85 (dd, *J* = 14.7, 8.6 Hz, 1H), 1.52 (dd, *J* = 14.7, 2.8 Hz, 1H), 1.36–1.22 (m, 4H), 0.96 (s, 3H), 0.94 (s, 3H), 0.86 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.5, 133.4, 133.1, 128.5, 127.9, 127.8, 126.8, 126.2, 125.8, 125.4, 124.8, 81.9, 56.4, 50.0, 46.2, 33.7, 32.6, 32.0, 27.83, 27.77, 22.4, 14.1; **IR** (neat) 3055, 1602 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{23}H_{32}ONa$ $(M + Na)^+$ 347.2351, found 347.2337.



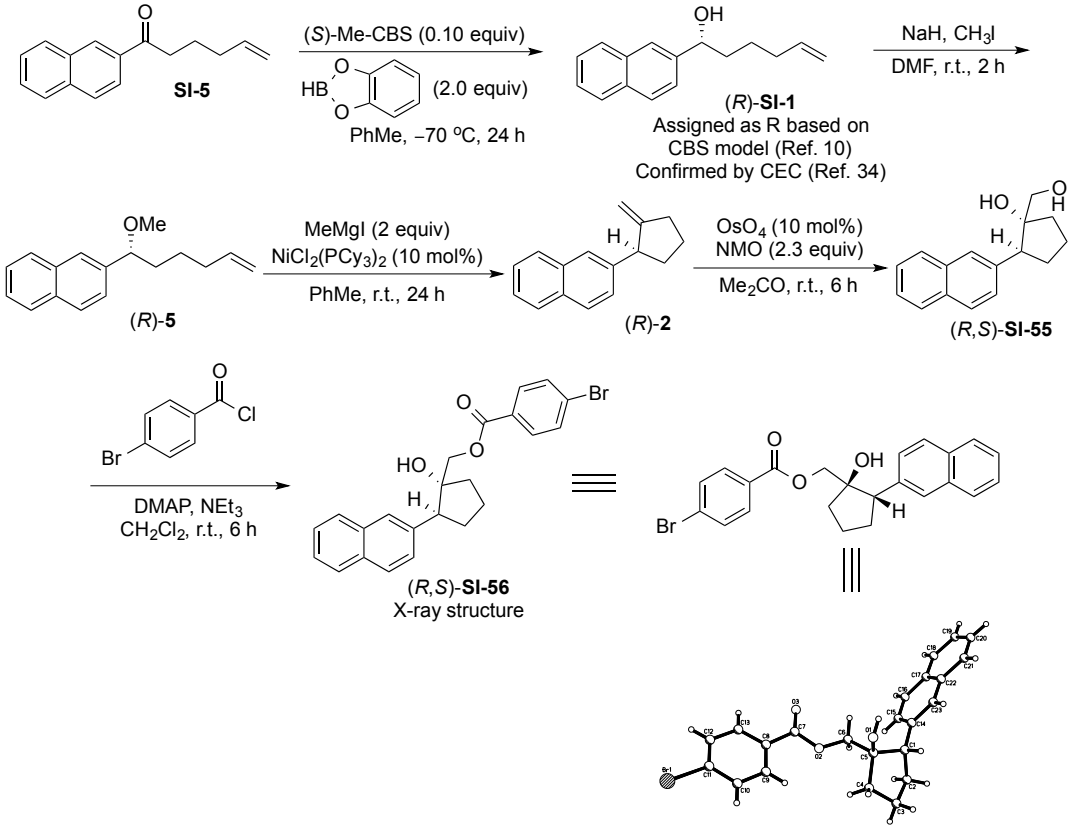
SI-52, SI-53, and SI-54. Using representative procedure I outlined above, with the exception of heating to 65 ^{o}C, the following amounts of reagents were used: **SI-51** (33 mg, 0.10 mmol, 1.0 equiv), bis(tricyclohexylphosphine)nickel(II) dichloride (7 mg, 0.010 mmol, 0.10 equiv), PhMe (0.8 mL) and methylmagnesium iodide (0.07 mL, 2.7 M in Et_2O, 0.20 mmol, 2.0 equiv). After workup, phenyltrimethylsilane was added (8.6 µL, 0.050 mmol, 0.50 equiv) and a ^{1}H NMR spectrum was collected to determine yields of products. Recovered **SI-51** (15%), **SI-52** (31%), **SI-53** (15%), **SI-54** (19%). See section X for annotated ^{1}H NMR spectrum.

VII. STEREOCHEMICAL PROOFS

i. Overview of stereochemical assignments

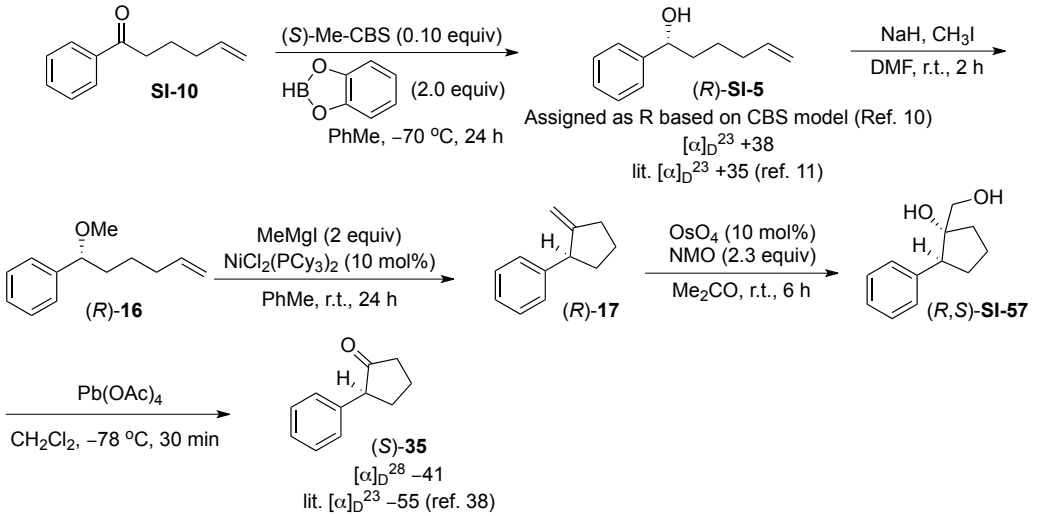
The stereochemical course of the Heck cyclization is demonstrated for three examples. Those experiments are summarized below. In all examples, we confirm that the Heck cyclization proceeds with inversion. For full experimental details of synthesis of starting materials and Heck cyclization reactions, see sections III and V, respectively. Full experimental details and characterization data for the derivatives synthesized to assign absolute configuration of products are provided below.

Scheme SI-7. Stereochemical course of the Heck cyclization.



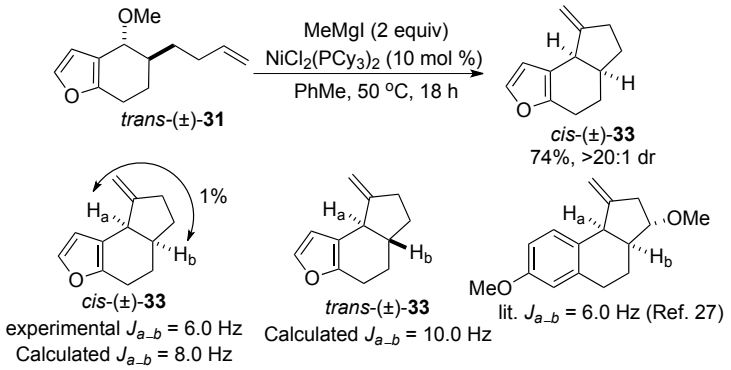
Enantioenriched alcohol (*R*)-**SI-1** was prepared by enantioselective CBS reduction (vida supra). Absolute configuration was assigned based on the accepted model for selectivity in CBS reductions[9] and confirmed by Competing Enantioselective Conversion (CEC) (vifa infra). Conversion to methyl ether (*R*)-**5**, followed by stereospecific Heck cyclization produced (*R*)-**2**. Dihydroxylation of the olefin and conversion of the primary alcohol to the 4-bromobenzoate afforded (*R*,*S*)-**SI-56**, the absolute configuration of which was determined by X-ray crystallography. See section IX for crystallographic data. This product corresponds to net inversion in the Heck cyclization.

Scheme SI-8. Stereochemical course of the Heck cyclization



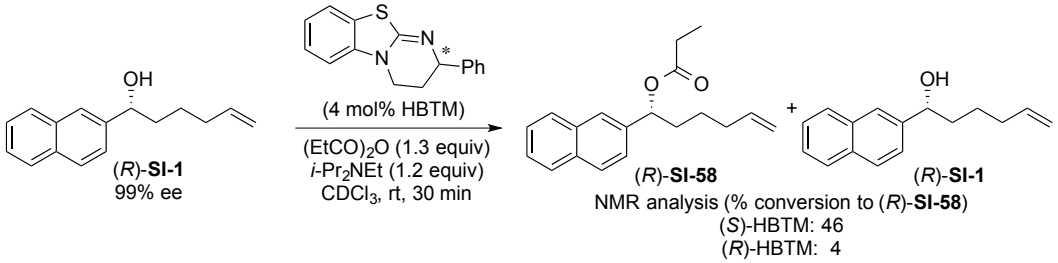
Enantioenriched alcohol (*R*)-**SI-5** was prepared by enantioselective CBS reduction (vida infra). Absolute configuration was assigned based on the accepted model for selectivity in CBS reductions[10] and confirmed by comparison of the optical rotation to literature value.[11] Conversion to methyl ether (*R*)-**16**, followed by stereospecific Heck-cyclization produced (*R*)-**17**. Dihydroxylation of the olefin followed by oxidative cleavage of the diol (*R*,*S*)-**SI-57** with $Pb(OAc)_4$ afforded α-aryl cyclopentanone (*S*)-**35**, the stereochemistry of which was determined by comparison of the optical rotation to the literature value.[34] This product corresponds to net inversion in the Heck cyclization.

Scheme SI-9. Stereochemical course of the Heck cyclization



NOESY and 1D nOe experiments show a strong nOe correlation between H_a and H_b, consistent with a cis orientation (see section X for relevant spectra). The J-coupling between the protons indicated is consistent with literature values for structurally similar compounds with a cis relationship.[27] The experimentally measured J-coupling constants are consistent with computational calculations. This product corresponds to net inversion in the Heck cyclization.

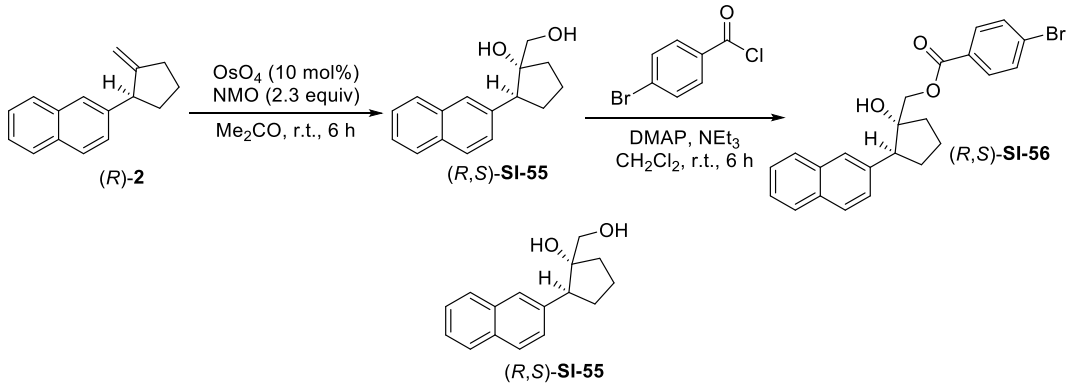
Scheme SI-10. Confirmation of absolute configuration of alcohol (*R*)-SI-1



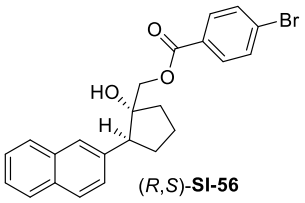
The competing enantioselective conversion experiment was performed according to the procedure outlined by Rychnovsky.[35] To a 7 mL vial under N_2 atmosphere was added alcohol (*R*)-**SI-1** (11 mg, 0.050 mmol, 1.0 equiv), *i*-Pr_2NEt (11 μL, 0.060 mmol, 1.2 equiv), HBTM catalyst (0.20 mL, 0.010 M solution in $CDCl_3$, 0.0020 mmol, 0.040 equiv) and $CDCl_3$ (0.7 mL). The mixture was allowed to stir for 5 min before addition of propionic anhydride (8 μL, 0.07 mmol, 1.3 equiv). The reaction was stirred for 30 min and transferred to a NMR tube for ^1H NMR analysis. Percent conversion was measured for two reactions, one run with (*S*)-HBTM catalyst (46% conversion) and one with (*R*)-HBTM catalyst (4% conversion). Based on the pneumonic described by Rychnovsky, we assigned alcohol (*R*)-**SI-1** as the *S* enantiomer. This assignment is consistent with E.J. Corey's stereochemical model for CBS reductions.

ii. Synthesis of hydroxyester (*R*,*S*)-SI-56

Hydroxyester (*R*,*S*)-**SI-56** was prepared from methylenecyclopentane (*R*)-**2** by dihydroxylation and esterification.



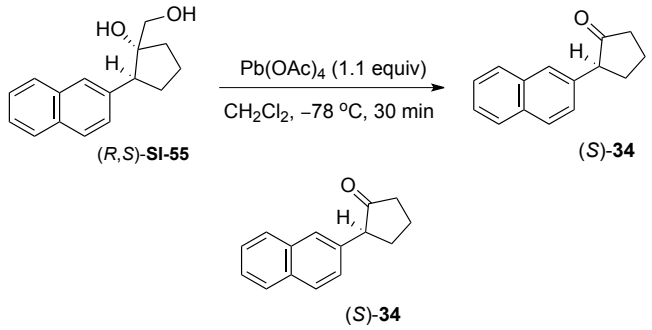
(*R*,*S*)-SI-55. The title compound was prepared according to a modified procedure reported by Gaikwad.[36] A 20 mL scintillation vial charged with a stir bar and (*R*)-**2** (90 mg, 0.44 mmol, 1.0 equiv) and acetone (4 mL). To this solution was added *N*-methylmorpholine-*N*-oxide (119 mg, 1.01 mmol, 2.30 equiv) and OsO_4 (0.28 mL, 4% in H_2O, 0.044 mmol, 0.10 equiv). The reaction vessel was sealed and allowed to stir at ambient temperature until TLC indicated the reaction had proceeded to conversion (5 h). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The diol was purified by flash column chromatography on silica gel (25% EtOAc/hexanes) to afford the title compound as a white, amorphous solid (88 mg, 0.36 mmol, 83%, >20:1 dr). **TLC R_f** = 0.3 (40% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.83–7.71 (m, 3H), 7.64 (s, 1H), 7.48–7.40 (m, 2H), 7.38 (d, *J* = 8.6 Hz, 1H), 3.32 (t, *J* = 8.2 Hz, 1H), 3.29–3.23 (m, 1H), 3.23–3.15 (m, 1H), 2.86 (s, 1H), 2.33–2.23 (m, 1H), 2.11–1.77 (m, 5H), 1.74 (br s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 138.8, 133.5, 132.4, 128.1, 127.8, 127.7, 126.8, 126.29, 126.26, 125.7, 84.0, 66.9, 55.2, 36.2, 30.6, 22.1; **IR** (neat) 3371, 2954, 2874 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{16}H_{18}O_2Na$ $(M + Na)^+$ 265.1205, found 265.1204; **$[\alpha]^{27}_D$** –29 (*c* 0.6, $CHCl_3$).



(*R*,*S*)-SI-56. The title compound was prepared according to a modified procedure reported by Hassner.[37] Under an atmosphere of N_2, a 20 mL scintillation vial charged with a stir bar and (*R*,*S*)-**SI-55** (40 mg, 0.17 mmol, 1.0 equiv) and dry CH_2Cl_2 (4 mL). To this solution was added 4-(dimethylamino)pyridine (2 mg, 0.02 mmol, 0.1 equiv), 4-bromobenzoylchloride (47 mg, 0.21 mmol, 1.3 equiv), and triethylamine (0.03 mL, 0.4 mmol, 2 equiv). The reaction was allowed to stir at ambient temperature until TLC indicated the reaction had proceeded to conversion (4 h). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The product was purified by flash column chromatography on silica gel (15% EtOAc/hexanes) to afford the title compound as a white solid (62 mg, 0.15 mmol, 88%, 99% ee). A single crystal for X-ray crystallography was grown **TLC** $\mathbf{R_f}$ = 0.2 (20% EtOAc/hexanes, UV active, stain with $KMnO_4$); **m.p.** = 138 °C; ^{1}**H NMR** (500 MHz, $CDCl_3$) δ 7.78–7.69 (m, 3H), 7.68 (s, 1H), 7.52 (d, *J* = 8.6 Hz, 2H), 7.47–7.39 (m, 3H), 7.37 (d, *J* = 8.6 Hz, 2H), 4.11 (d, *J* = 11.7 Hz, 1H), 4.02 (d, *J* = 11.7 Hz, 1H), 3.45 (t, *J* = 8.5 Hz, 1H), 2.58 (br s, 1H), 2.39–2.30 (m, 1H), 2.22–2.12 (m, 1H), 2.09–1.92 (m, 4H); ^{13}**C NMR** (125 MHz, $CDCl_3$) δ 165.9, 138.0, 133.5, 132.6, 131.6, 131.0, 128.5, 128.3, 128.1, 127.7, 127.6, 126.6, 126.5, 126.2, 125.7, 82.9, 69.3, 55.5, 36.7, 30.2, 21.8; **IR** (neat) 3487, 2956, 1717 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{23}H_{21}O_3BrNa$ $(M + Na)^+$ 447.0572, found 447.0581; $\mathbf{[\alpha]^{29}_D}$ –68 (*c* 2.8, $CHCl_3$); **SFC** analysis (Whelk-O (R,R), 10% IPA, 2.5 mL/min) indicated 99% ee: t_R (major) = 24.6 minutes, t_R (minor) = 23.5 minutes.

Single crystals suitable for X-ray crystallographic analysis were grown by slow diffusion of pentane into a solution of (*R*,*S*)-**SI-56** in EtOAc at ambient temperature. See Section IX for crystallographic data.

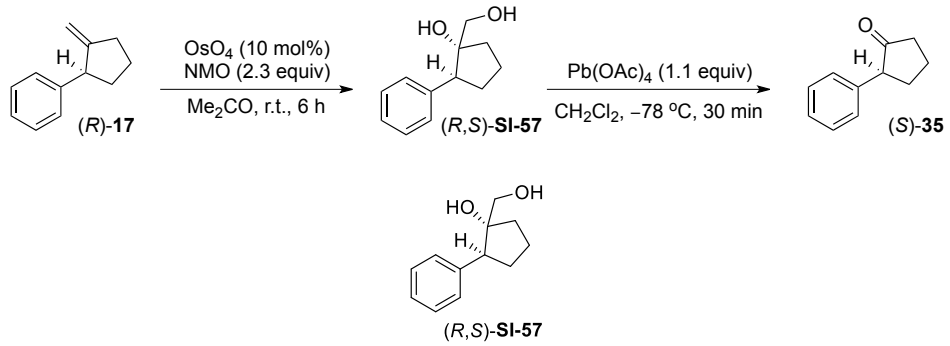
iii. Synthesis of ketone (*S*)-34 via oxidative cleavage



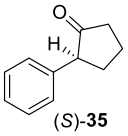
(*S*)-34. The title compound was prepared according to a modified procedure reported by Wicha.[38] A 20 mL scintillation vial charged with a stir bar and (*R*,*S*)-**SI-55** (36 mg, 0.15 mmol, 1.0 equiv) and CH_2Cl_2 (4 mL). The reaction was cooled to –78 °C to avoid epimerization of the benzylic stereocenter. To this solution was added $Pb(OAc)_4$ (73 mg, 0.17 mmol, 1.1 equiv) in a single portion. The reaction was allowed to stir at –78 °C until TLC indicated the reaction had proceeded to conversion (30 min). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The ketone was purified by flash column chromatography on silica gel (10% EtOAc/hexanes) to afford the title compound as a white solid (20 mg, 0.095 mmol, 63%, 98% ee). Analytical data is consistent with literature values.[34] **TLC** $\mathbf{R_f}$ = 0.2 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.85–7.76 (m, 3H), 7.64 (s, 1H), 7.49–7.41 (m, 2H), 7.31 (d, *J* = 8.6 Hz, 1H), 3.50 (at, *J* = 9.7 Hz, 1H), 2.61–2.54 (m, 1H), 2.52 (dd, *J* = 19.1, 8.6 Hz, 1H), 2.41–3.30 (m, 1H), 2.28–2.17 (m, 2H), 2.05–1.93 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 218.3, 136.0, 133.6, 132.6, 128.4, 127.82, 127.75, 126.9, 126.5, 126.2, 125.8, 55.6, 38.7, 31.9, 21.1; $\mathbf{[\alpha]^{24}_D}$ –37 (*c* 0.9, $CHCl_3$); **SFC** analysis (AS-H, 15% IPA, 2.5 mL/min) indicated 98% ee: t_R (major) = 3.9 minutes, t_R (minor) = 3.0 minutes.

iv. Synthesis of ketone (*S*)-35 via oxidative cleavage

The ketone (*S*)-**35** was prepared from the methylenecyclopentane (*R*)-**17**. Dihydroxylation of (*R*)-**17** followed by oxidative cleavage with $Pb(OAc)_4$ afforded the ketone (*S*)-**35**.



(*R*,*S*)-SI-55. The title compound was prepared according to a modified procedure reported by Gaikwad.[36] A 20 mL scintillation vial charged with a stir bar and (*R*)-**17** (55 mg, 0.45 mmol, 1.0 equiv) and acetone (4 mL). To this solution was added *N*-methylmorpholine-*N*-oxide (122 mg, 1.03 mmol, 2.30 equiv) and OsO_4 (0.28 mL, 4% in H_2O, 0.045 mmol, 0.10 equiv). The reaction vessel was sealed and allowed to stir at ambient temperature until TLC indicated the reaction had proceeded to conversion (5 h). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The diol was purified by flash column chromatography on silica gel (25% EtOAc/hexanes) to afford the title compound as clear oil (48 mg, 0.25 mmol, 56%, 7:1 dr). **TLC R_f** = 0.4 (40% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33–7.28 (m, 2H), 7.27–7.20 (m, 3H), 3.30 (d, *J* = 11.5 Hz, 1H), 3.24–3.13 (m, 2H), 2.75 (br s, 1H), 2.28–2.18 (m, 1H), 2.00–1.90 (m, 2H), 1.90–1.78 (m, 3H), 1.55 (br s, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 141.3, 128.7, 128.0, 126.9, 83.8, 66.9, 55.2, 36.0, 30.5, 22.0; **IR** (neat) 3376, 2954, 2874 cm^{-1}; **HRMS** (TOF MS ES+) *m* / *z* calcd for $C_{12}H_{16}O_2Na$ $(M + Na)^+$ 215.1048, found 215.1044.



(*S*)-**35**

(*S*)-35. The title compound was prepared according to a modified procedure reported by Wicha.[38] A 20 mL scintillation vial charged with a stir bar and (*R*,*S*)-**SI-57** (35 mg, 0.18 mmol, 1.0 equiv) and CH_2Cl_2 (4 mL). The reaction was cooled to –78 °C to avoid epimerization of the benzylic stereocenter. To this solution was added $Pb(OAc)_4$ (89 mg, 0.20 mmol, 1.1 equiv) in a single portion. The reaction was allowed to stir at –78 °C until TLC indicated the reaction had proceeded to conversion (30 min). The reaction was diluted with EtOAc (5 mL) and quenched with sat. NH_4Cl (4 mL) and the layers were separated. The aqueous phase was extracted with EtOAc (3 x 2 mL) and the combined organic layers were washed with brine (1 x 4 mL), dried over $MgSO_4$ filtered and concentrated in vacuo. The ketone was purified by flash column chromatography on silica gel (10% EtOAc/hexanes) to afford the title compound as a white, amorphous solid (20 mg, 0.13 mmol, 72%, 76% ee). Analytical data is consistent with literature values.[34] **TLC R_f** = 0.2 (10% EtOAc/hexanes, UV active, stain with $KMnO_4$); **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.1 Hz, 2H), 7.28–7.22 (m, 1H), 7.19 (d, *J* = 7.1 Hz, 2H), 3.37–3.29 (m, 1H), 2.57–2.43 (m, 2H), 2.35–2.24 (m, 1H), 2.21–2.07 (m, 2H), 1.99–1.89 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 218.3, 138.6, 128.7, 128.3, 127.0, 55.5, 38.6, 31.9, 21.0; **$[\alpha]^{28}_D$** –41 (*c* 0.3, $CHCl_3$); **SFC** analysis (OD-H, 10% IPA, 2.5 mL/min) indicated 76% ee: t_R (major) = 3.4 minutes, t_R (minor) = 3.2 minutes.

VIII. References and Notes

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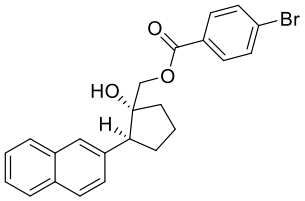
IX. CRYSTALLOGRAPHIC DATA



(*R*,*S*)-**SI-56**
Scheme SI-7

i. X-ray Data Collection, Structure Solution and Refinement for (*R*,*S*)-SI-56.

CCDC 1003298 contains the supplementary crystallographic data for this structure. These data can be obtained via www.ccdc.ac.uk/conts/retrieving.html. The crystal was obtained by slow evaporation from ethyl acetate over pentane.

A colorless crystal of approximate dimensions 0.293 x 0.205 x 0.111 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (15 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The diffraction symmetry was *mmm* and the systematic absences were consistent with the orthorhombic space group $P2_12_12_1$ that was later determined to be correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] for neutral atoms were used throughout the analysis. Hydrogen atoms were included using a riding model.

At convergence, wR2 = 0.0536 and Goof = 1.054 for 247 variables refined against 4644 data (0.73 Å), R1 = 0.0223 for those 4412 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6].

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2. SAINT Version 8.34a, Bruker AXS, Inc.; Madison, WI 2013.

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Definitions:

$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] / \Sigma[w(F_o^2)^2]\ ]^{1/2}$

$R1 = \Sigma||F_o|-|F_c|| / \Sigma|F_o|$

$Goof = S = [\Sigma[w(F_o^2-F_c^2)^2] / (n-p)]^{1/2}$ where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

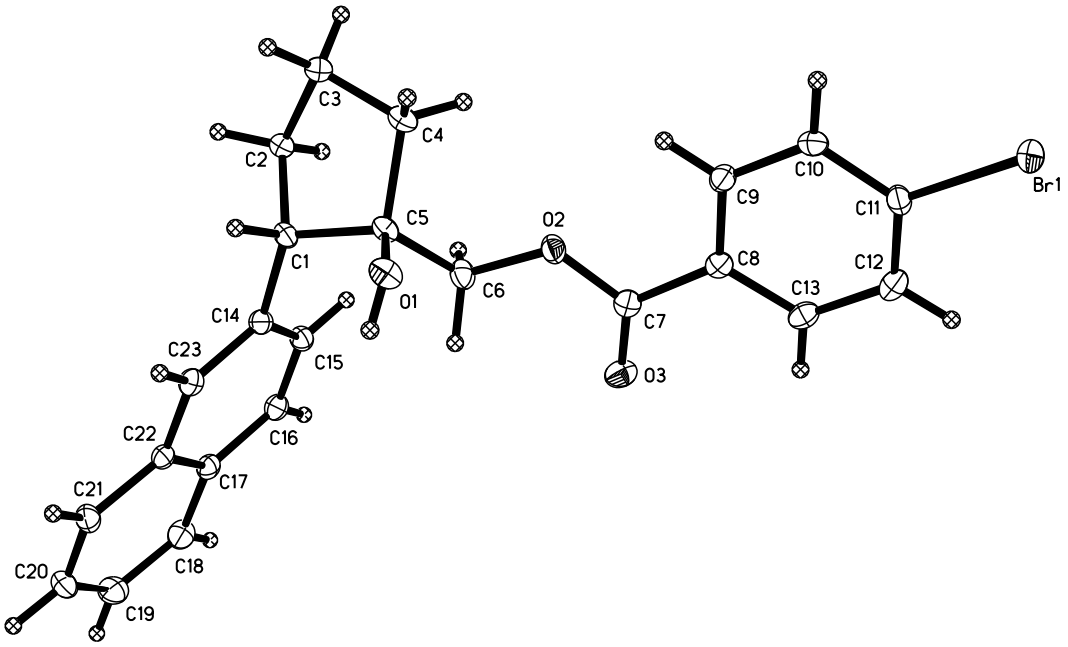


Table 1. Crystal data and structure refinement for erj16.

Identification code	erj16 (Michael Harris)	
Empirical formula	$C_{23}H_{21}BrO_3$	
Formula weight	425.31	
Temperature	88(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 7.9672(4) Å	α= 90°.
	b = 9.3042(4) Å	β= 90°.
	c = 24.7018(11) Å	γ = 90°.
Volume	1831.11(15) Å^3	
Z	4	
Density (calculated)	1.543 Mg/m^3	
Absorption coefficient	2.266 mm^{-1}	
F(000)	872	
Crystal color	colorless	

Crystal size	0.293 x 0.205 x 0.111 mm^3
Theta range for data collection	1.649 to 29.026°
Index ranges	$-10 \leq h \leq 10$, $-12 \leq k \leq 12$, $-32 \leq l \leq 32$
Reflections collected	22622
Independent reflections	4644 [R(int) = 0.0251]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Numerical
Max. and min. transmission	0.8205 and 0.6133
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4644 / 0 / 247
Goodness-of-fit on F^2	1.054
Final R indices [I>2sigma(I) = 4412 data]	R1 = 0.0223, wR2 = 0.0528
R indices (all data, 0.73 Å)	R1 = 0.0245, wR2 = 0.0536
Absolute structure parameter	-0.007(2)
Largest diff. peak and hole	0.597 and -0.299 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for erj16. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	12231(1)	12759(1)	9891(1)	21(1)
O(1)	9393(2)	5010(2)	8236(1)	17(1)
O(2)	8218(2)	7849(2)	8350(1)	17(1)
O(3)	8638(2)	9449(2)	7682(1)	19(1)
C(1)	6594(3)	4163(2)	7945(1)	13(1)
C(2)	4933(3)	4160(2)	8260(1)	15(1)
C(3)	5549(3)	4243(2)	8848(1)	19(1)
C(4)	7006(3)	5317(2)	8826(1)	19(1)
C(5)	7648(3)	5352(2)	8234(1)	13(1)
C(6)	7414(3)	6842(2)	7985(1)	16(1)
C(7)	8726(3)	9114(2)	8156(1)	14(1)
C(8)	9453(3)	10031(2)	8591(1)	14(1)
C(9)	9538(3)	9552(2)	9125(1)	15(1)
C(10)	10340(3)	10367(2)	9517(1)	15(1)
C(11)	11033(3)	11682(2)	9366(1)	15(1)
C(12)	10900(2)	12208(3)	8842(1)	18(1)
C(13)	10105(3)	11377(2)	8455(1)	16(1)
C(14)	6411(3)	4202(2)	7334(1)	13(1)
C(15)	5344(3)	5226(2)	7076(1)	14(1)
C(16)	5088(3)	5208(2)	6529(1)	15(1)
C(17)	5859(3)	4150(2)	6198(1)	14(1)
C(18)	5577(3)	4084(3)	5629(1)	19(1)
C(19)	6305(3)	3026(3)	5325(1)	22(1)
C(20)	7352(3)	1985(2)	5571(1)	20(1)
C(21)	7666(3)	2027(2)	6119(1)	17(1)
C(22)	6920(2)	3113(2)	6443(1)	13(1)
C(23)	7176(3)	3179(2)	7016(1)	14(1)

Table 3. Bond lengths [Å] and angles [°] for erj16.

Br(1)-C(11)	1.895(2)
O(1)-C(5)	1.426(3)
O(2)-C(7)	1.334(3)
O(2)-C(6)	1.450(2)
O(3)-C(7)	1.212(3)
C(1)-C(14)	1.515(3)
C(1)-C(2)	1.535(3)
C(1)-C(5)	1.562(3)
C(2)-C(3)	1.535(3)
C(3)-C(4)	1.533(3)
C(4)-C(5)	1.551(3)
C(5)-C(6)	1.527(3)
C(7)-C(8)	1.490(3)
C(8)-C(9)	1.394(3)
C(8)-C(13)	1.397(3)
C(9)-C(10)	1.387(3)
C(10)-C(11)	1.393(3)
C(11)-C(12)	1.388(3)
C(12)-C(13)	1.384(3)
C(14)-C(23)	1.377(3)
C(14)-C(15)	1.427(3)
C(15)-C(16)	1.367(3)
C(16)-C(17)	1.420(3)
C(17)-C(22)	1.419(3)
C(17)-C(18)	1.425(3)
C(18)-C(19)	1.366(3)
C(19)-C(20)	1.415(3)
C(20)-C(21)	1.375(3)
C(21)-C(22)	1.420(3)
C(22)-C(23)	1.430(3)
C(7)-O(2)-C(6)	118.63(16)
C(14)-C(1)-C(2)	114.94(18)
C(14)-C(1)-C(5)	119.27(17)

C(2)-C(1)-C(5)	103.50(16)
C(1)-C(2)-C(3)	101.76(18)
C(4)-C(3)-C(2)	104.03(17)
C(3)-C(4)-C(5)	107.22(17)
O(1)-C(5)-C(6)	108.87(17)
O(1)-C(5)-C(4)	108.19(17)
C(6)-C(5)-C(4)	110.96(17)
O(1)-C(5)-C(1)	111.64(16)
C(6)-C(5)-C(1)	113.18(16)
C(4)-C(5)-C(1)	103.83(16)
O(2)-C(6)-C(5)	106.40(16)
O(3)-C(7)-O(2)	123.9(2)
O(3)-C(7)-C(8)	124.8(2)
O(2)-C(7)-C(8)	111.27(18)
C(9)-C(8)-C(13)	119.7(2)
C(9)-C(8)-C(7)	121.2(2)
C(13)-C(8)-C(7)	119.0(2)
C(10)-C(9)-C(8)	120.6(2)
C(9)-C(10)-C(11)	118.4(2)
C(12)-C(11)-C(10)	121.9(2)
C(12)-C(11)-Br(1)	119.28(17)
C(10)-C(11)-Br(1)	118.77(17)
C(13)-C(12)-C(11)	118.9(2)
C(12)-C(13)-C(8)	120.3(2)
C(23)-C(14)-C(15)	117.97(19)
C(23)-C(14)-C(1)	120.63(19)
C(15)-C(14)-C(1)	121.22(19)
C(16)-C(15)-C(14)	121.6(2)
C(15)-C(16)-C(17)	120.9(2)
C(22)-C(17)-C(16)	118.89(19)
C(22)-C(17)-C(18)	119.1(2)
C(16)-C(17)-C(18)	122.0(2)
C(19)-C(18)-C(17)	120.3(2)
C(18)-C(19)-C(20)	120.5(2)
C(21)-C(20)-C(19)	120.7(2)
C(20)-C(21)-C(22)	119.9(2)

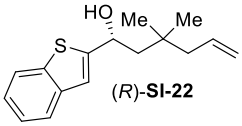
C(17)-C(22)-C(21)	119.47(19)
C(17)-C(22)-C(23)	118.60(19)
C(21)-C(22)-C(23)	121.92(19)
C(14)-C(23)-C(22)	122.1(2)

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for erj16. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	21(1)	21(1)	20(1)	-4(1)	1(1)	-8(1)
O(1)	14(1)	22(1)	15(1)	-3(1)	-2(1)	3(1)
O(2)	24(1)	13(1)	13(1)	-1(1)	-2(1)	-2(1)
O(3)	16(1)	25(1)	16(1)	4(1)	-2(1)	-4(1)
C(1)	15(1)	12(1)	12(1)	-2(1)	0(1)	2(1)
C(2)	18(1)	15(1)	12(1)	-1(1)	2(1)	-2(1)
C(3)	28(1)	16(1)	13(1)	0(1)	3(1)	-3(1)
C(4)	25(1)	21(1)	12(1)	-3(1)	1(1)	-3(1)
C(5)	13(1)	15(1)	11(1)	-2(1)	-1(1)	1(1)
C(6)	20(1)	15(1)	15(1)	-2(1)	-4(1)	-1(1)
C(7)	10(1)	15(1)	18(1)	0(1)	0(1)	2(1)
C(8)	11(1)	13(1)	16(1)	1(1)	2(1)	2(1)
C(9)	16(1)	13(1)	17(1)	0(1)	3(1)	-1(1)
C(10)	15(1)	16(1)	13(1)	1(1)	3(1)	1(1)
C(11)	12(1)	15(1)	18(1)	-5(1)	1(1)	-1(1)
C(12)	15(1)	14(1)	24(1)	2(1)	2(1)	-1(1)
C(13)	14(1)	16(1)	17(1)	4(1)	1(1)	1(1)
C(14)	13(1)	15(1)	12(1)	-1(1)	-1(1)	-2(1)
C(15)	14(1)	14(1)	16(1)	-2(1)	1(1)	0(1)
C(16)	14(1)	14(1)	17(1)	1(1)	-1(1)	-1(1)
C(17)	14(1)	14(1)	16(1)	0(1)	-1(1)	-3(1)
C(18)	21(1)	19(1)	17(1)	2(1)	-4(1)	-3(1)
C(19)	27(1)	26(1)	13(1)	-2(1)	-1(1)	-6(1)
C(20)	22(1)	19(1)	18(1)	-6(1)	4(1)	-2(1)
C(21)	15(1)	15(1)	20(1)	-4(1)	1(1)	-1(1)
C(22)	11(1)	13(1)	16(1)	-1(1)	1(1)	-3(1)
C(23)	12(1)	14(1)	16(1)	0(1)	-1(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj16.

	x	y	z	U(eq)
H(1A)	7156	3230	8031	16
H(2A)	4288	3268	8193	18
H(2B)	4231	5001	8165	18
H(3A)	4647	4591	9091	23
H(3B)	5937	3292	8976	23
H(4A)	7917	5012	9074	23
H(4B)	6619	6284	8938	23
H(6A)	7940	6885	7622	20
H(6B)	6205	7068	7948	20
H(9A)	9043	8659	9221	18
H(10A)	10414	10037	9881	18
H(12A)	11346	13124	8752	21
H(13A)	10003	11724	8095	19
H(15A)	4799	5935	7289	17
H(16A)	4385	5914	6368	18
H(18A)	4879	4779	5459	23
H(19A)	6107	2988	4946	26
H(20A)	7842	1250	5357	24
H(21A)	8382	1331	6279	20
H(23A)	7897	2495	7182	17
H(1)	9750(40)	4920(30)	7940(12)	21



(*R*)-**SI-22**

ii. X-ray Data Collection, Structure Solution and Refinement for (*R*)-SI-22.

CCDC 1003301 contains the supplementary crystallographic data for this structure. These data can be obtained via www.ccdc.ac.uk/conts/retrieving.html. The crystal was obtained by slow evaporation from ethyl acetate over pentane.

A colorless crystal of approximate dimensions 0.062 x 0.120 x 0.329 mm was mounted on a glass fiber and transferred to a Bruker SMART APEX II diffractometer. The APEX2[1] program package was used to determine the unit-cell parameters and for data collection (120 sec/frame scan time for a sphere of diffraction data). The raw frame data was processed using SAINT[2] and SADABS[3] to yield the reflection data file. Subsequent calculations were carried out using the SHELXTL[4] program. The systematic absences were consistent with the trigonal space groups $P3_1$ and $P3_2$. It was later determined that space group $P3_1$ was correct.

The structure was solved by direct methods and refined on F^2 by full-matrix least-squares techniques. The analytical scattering factors[5] for neutral atoms were used throughout the analysis. Hydrogen atom H(1) was located from a difference-Fourier map and refined (x,y,z and U_{iso}) with d(O-H) = 0.85Å. The remaining hydrogen atoms were included using a riding model. Carbon atoms C(12), C(13) and C(14) were disordered and included using multiple components with partial site-occupancy-factors (0.62:0.38).

At convergence, wR2 = 0.0960 and Goof = 1.039 for 196 variables refined against 3419 data (0.74Å), R1 = 0.0383 for those 3258 data with $I > 2.0\sigma(I)$. The absolute structure was assigned by refinement of the Flack parameter[6].

References.

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9. Sheldrick, G. M. SADABS, Version 2012/1, Bruker AXS, Inc.; Madison, WI 2012.

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12. Flack, H. D., Parsons, S., Wagner, T. Acta. Cryst., B69, 249-259, 2013

Definitions:

$wR2 = [\Sigma[w(F_o^2-F_c^2)^2] / \Sigma[w(F_o^2)^2]\ ]^{1/2}$

$R1 = \Sigma||F_o|-|F_c|| / \Sigma|F_o|$

$Goof = S = [\Sigma[w(F_o^2-F_c^2)^2] / (n-p)]^{1/2}$ where n is the number of reflections and p is the total number of parameters refined.

The thermal ellipsoid plot is shown at the 50% probability level.

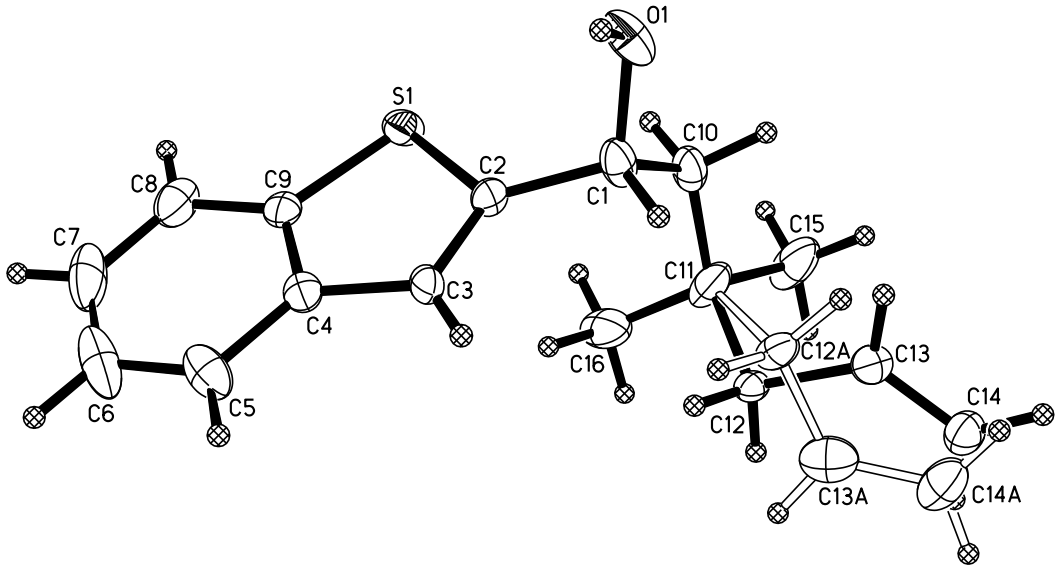


Table 1. Crystal data and structure refinement for erj15.

Identification code	erj15 (Mikhail Konev)	
Empirical formula	$C_{16}H_{20}OS$	
Formula weight	260.38	
Temperature	133(2) K	
Wavelength	0.71073 Å	
Crystal system	Trigonal	
Space group	$P3_1$	
Unit cell dimensions	a = 13.4769(8) Å	α= 90°.
	b = 13.4769(8) Å	β= 90°.
	c = 6.8167(4) Å	γ = 120°.
Volume	1072.22(14) $Å^3$	
Z	3	
Density (calculated)	1.210 Mg/m^3	
Absorption coefficient	0.213 mm^{-1}	
F(000)	420	
Crystal color	colorless	
Crystal size	0.329 x 0.120 x 0.062 mm^3	
Theta range for data collection	1.745 to 28.670°	

Index ranges	$-18 \leq h \leq 17$, $-17 \leq k \leq 17$, $-9 \leq l \leq 9$
Reflections collected	12748
Independent reflections	3419 [R(int) = 0.0230]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Numerical
Max. and min. transmission	1.0000 and 0.9139
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3419 / 2 / 196
Goodness-of-fit on F^2	1.039
Final R indices [I>2sigma(I) = 3258 data]	R1 = 0.0383, wR2 = 0.0935
R indices (all data, 0.74Å)	R1 = 0.0410, wR2 = 0.0960
Absolute structure parameter	0.02(2)
Largest diff. peak and hole	0.436 and -0.314 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for erj15. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	7101(1)	-1544(1)	2156(1)	24(1)
O(1)	9767(2)	457(2)	1758(3)	31(1)
C(1)	8870(2)	712(2)	1372(4)	26(1)
C(2)	7821(2)	-343(2)	652(4)	18(1)
C(3)	7341(2)	-502(2)	-1192(4)	21(1)
C(4)	6330(2)	-1637(2)	-1378(4)	20(1)
C(5)	5595(3)	-2117(3)	-2987(4)	31(1)
C(6)	4679(3)	-3219(3)	-2855(6)	43(1)
C(7)	4482(3)	-3858(3)	-1157(7)	44(1)
C(8)	5184(3)	-3414(3)	460(5)	33(1)
C(9)	6112(2)	-2292(2)	331(4)	21(1)
C(10)	8751(3)	1254(2)	3270(4)	27(1)
C(11)	7978(4)	1797(3)	3275(5)	39(1)
C(12)	8083(5)	2583(4)	1603(7)	17(1)
C(13)	9261(4)	3618(4)	1533(6)	22(1)
C(14)	9469(6)	4683(5)	1533(8)	26(1)
C(12A)	8673(9)	2719(7)	1334(11)	23(2)
C(13A)	8261(9)	3546(8)	1236(13)	36(2)
C(14A)	8926(12)	4666(9)	1432(15)	37(2)
C(15)	8229(4)	2481(3)	5197(5)	50(1)
C(16)	6711(3)	896(3)	3229(6)	41(1)

Table 3. Bond lengths [Å] and angles [°] for erj15.

S(1)-C(9)	1.731(3)
S(1)-C(2)	1.745(3)
O(1)-C(1)	1.438(4)
C(1)-C(2)	1.500(4)
C(1)-C(10)	1.533(4)
C(2)-C(3)	1.381(4)
C(3)-C(4)	1.459(4)
C(4)-C(5)	1.401(4)
C(4)-C(9)	1.401(4)
C(5)-C(6)	1.380(5)
C(6)-C(7)	1.387(6)
C(7)-C(8)	1.379(5)
C(8)-C(9)	1.403(4)
C(10)-C(11)	1.544(5)
C(11)-C(12)	1.513(5)
C(11)-C(16)	1.522(6)
C(11)-C(15)	1.539(4)
C(11)-C(12A)	1.734(9)
C(12)-C(13)	1.502(6)
C(13)-C(14)	1.318(7)
C(12A)-C(13A)	1.475(12)
C(13A)-C(14A)	1.322(15)
C(9)-S(1)-C(2)	91.55(13)
O(1)-C(1)-C(2)	109.8(2)
O(1)-C(1)-C(10)	105.1(2)
C(2)-C(1)-C(10)	117.0(2)
C(3)-C(2)-C(1)	126.3(2)
C(3)-C(2)-S(1)	113.0(2)
C(1)-C(2)-S(1)	120.71(19)
C(2)-C(3)-C(4)	111.2(2)
C(5)-C(4)-C(9)	118.9(3)
C(5)-C(4)-C(3)	128.7(3)
C(9)-C(4)-C(3)	112.3(2)

C(6)-C(5)-C(4)	119.4(3)
C(5)-C(6)-C(7)	120.9(3)
C(8)-C(7)-C(6)	121.4(3)
C(7)-C(8)-C(9)	117.8(3)
C(4)-C(9)-C(8)	121.6(3)
C(4)-C(9)-S(1)	111.9(2)
C(8)-C(9)-S(1)	126.5(2)
C(1)-C(10)-C(11)	118.9(2)
C(12)-C(11)-C(16)	102.6(3)
C(12)-C(11)-C(15)	107.9(3)
C(16)-C(11)-C(15)	108.5(3)
C(12)-C(11)-C(10)	118.7(3)
C(16)-C(11)-C(10)	112.0(2)
C(15)-C(11)-C(10)	106.7(3)
C(16)-C(11)-C(12A)	123.0(4)
C(15)-C(11)-C(12A)	110.0(4)
C(10)-C(11)-C(12A)	95.0(4)
C(13)-C(12)-C(11)	111.1(4)
C(14)-C(13)-C(12)	124.2(5)
C(13A)-C(12A)-C(11)	107.6(7)
C(14A)-C(13A)-C(12A)	124.2(10)

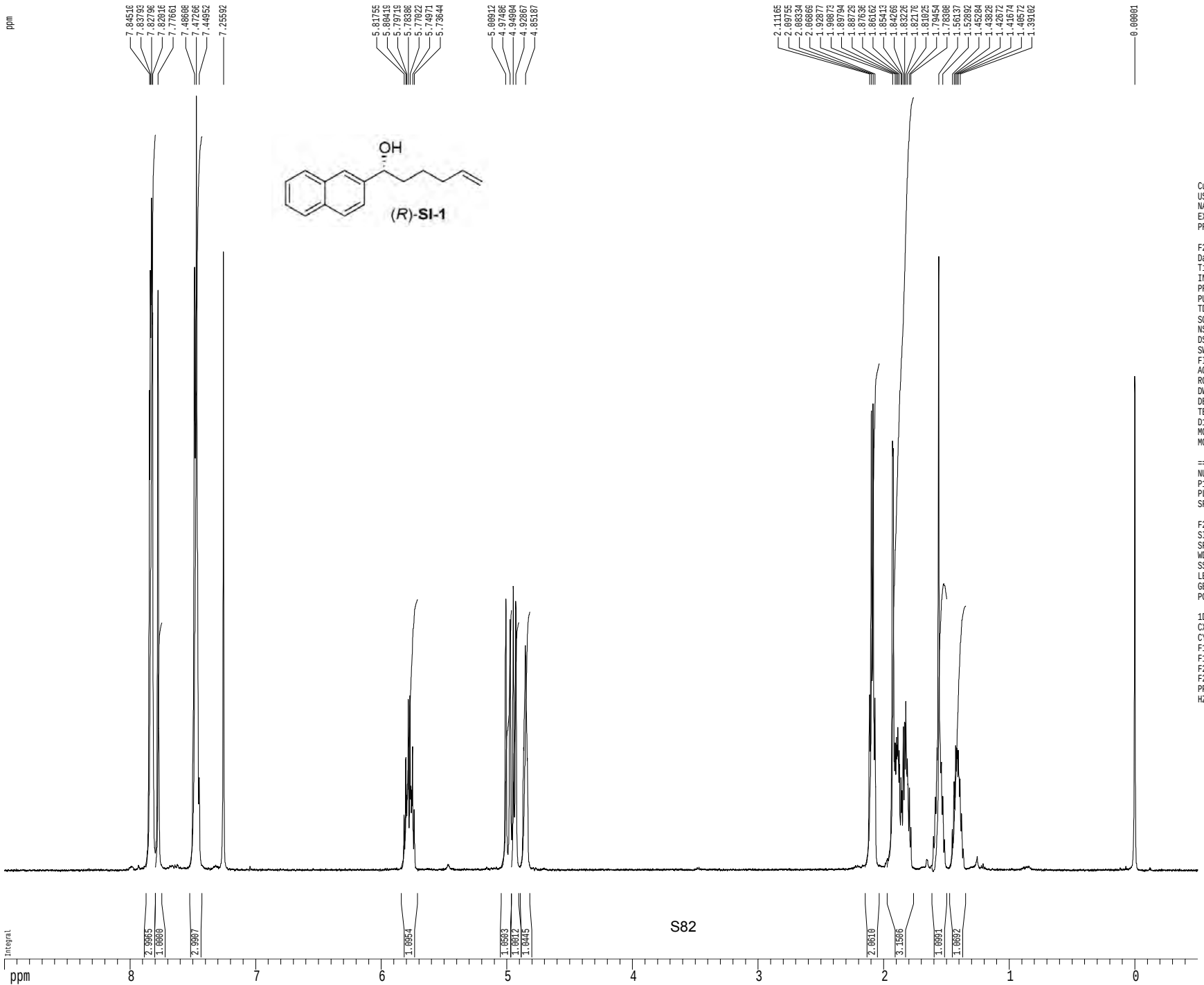
Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for erj15. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	18(1)	22(1)	27(1)	8(1)	-1(1)	7(1)
O(1)	20(1)	44(1)	19(1)	0(1)	-1(1)	8(1)
C(1)	25(1)	20(1)	19(1)	3(1)	-3(1)	2(1)
C(2)	19(1)	16(1)	16(1)	5(1)	2(1)	6(1)
C(3)	20(1)	19(1)	27(1)	-8(1)	-6(1)	11(1)
C(4)	18(1)	22(1)	24(1)	-5(1)	1(1)	12(1)
C(5)	24(1)	44(2)	24(1)	-12(1)	-2(1)	17(1)
C(6)	23(2)	49(2)	44(2)	-28(2)	-2(1)	9(1)
C(7)	21(2)	27(2)	72(3)	-19(2)	1(2)	3(1)
C(8)	20(1)	21(1)	56(2)	2(1)	6(1)	9(1)
C(9)	15(1)	19(1)	30(1)	-1(1)	0(1)	11(1)
C(10)	32(2)	20(1)	21(1)	0(1)	-9(1)	6(1)
C(11)	72(3)	21(1)	26(2)	-6(1)	-20(2)	26(2)
C(12)	14(2)	17(2)	22(2)	3(2)	2(2)	9(2)
C(13)	19(2)	23(2)	23(2)	4(2)	4(2)	11(2)
C(14)	27(3)	22(2)	26(2)	3(2)	1(2)	11(2)
C(12A)	32(5)	22(4)	20(4)	3(3)	0(3)	17(4)
C(13A)	42(5)	47(5)	30(4)	2(4)	1(4)	29(4)
C(14A)	53(7)	27(5)	34(5)	0(3)	2(5)	23(5)
C(15)	97(3)	30(2)	30(2)	-12(1)	-24(2)	36(2)
C(16)	59(2)	44(2)	41(2)	-18(2)	-20(2)	41(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for erj15.

	x	y	z	U(eq)
H(1)	9960(40)	290(40)	670(40)	67(16)
H(1A)	9137	1304	312	31
H(3A)	7630	55	-2208	25
H(5A)	5726	-1687	-4159	37
H(6A)	4176	-3544	-3942	52
H(7A)	3850	-4618	-1107	53
H(8A)	5044	-3854	1622	40
H(10A)	9529	1854	3672	33
H(10B)	8463	656	4299	33
H(12A)	7922	2160	347	20
H(12B)	7509	2829	1771	20
H(13A)	9897	3497	1485	26
H(14A)	8852	4833	1580	31
H(14B)	10237	5300	1486	31
H(12C)	9513	3126	1544	28
H(12D)	8501	2283	91	28
H(13B)	7467	3255	1020	44
H(14C)	9725	4985	1650	45
H(14D)	8605	5153	1354	45
H(15A)	8053	1963	6316	76
H(15B)	7753	2841	5263	76
H(15C)	9040	3074	5237	76
H(16A)	6535	363	4326	62
H(16B)	6532	473	1988	62
H(16C)	6251	1271	3345	62

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-285-1HNMR
EXPNO 1
PROCNO 1

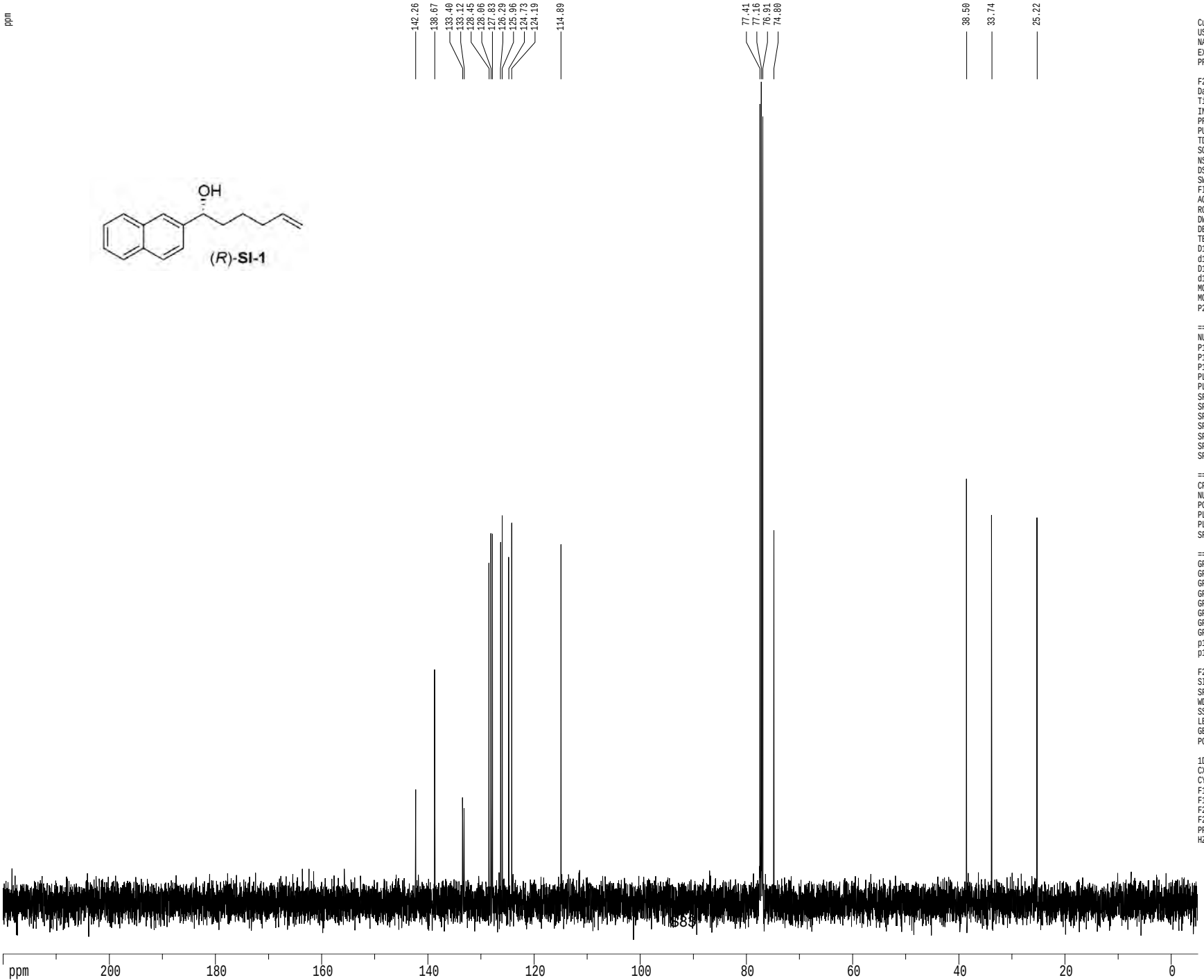
F2 - Acquisition Parameters
Date_ 20131209
Time 18.39
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SF01 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200326 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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

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Current Data Parameters
USER              mharri
NAME     MRH-V-285-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131209
Time              18.43
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   76
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG               9195.2
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

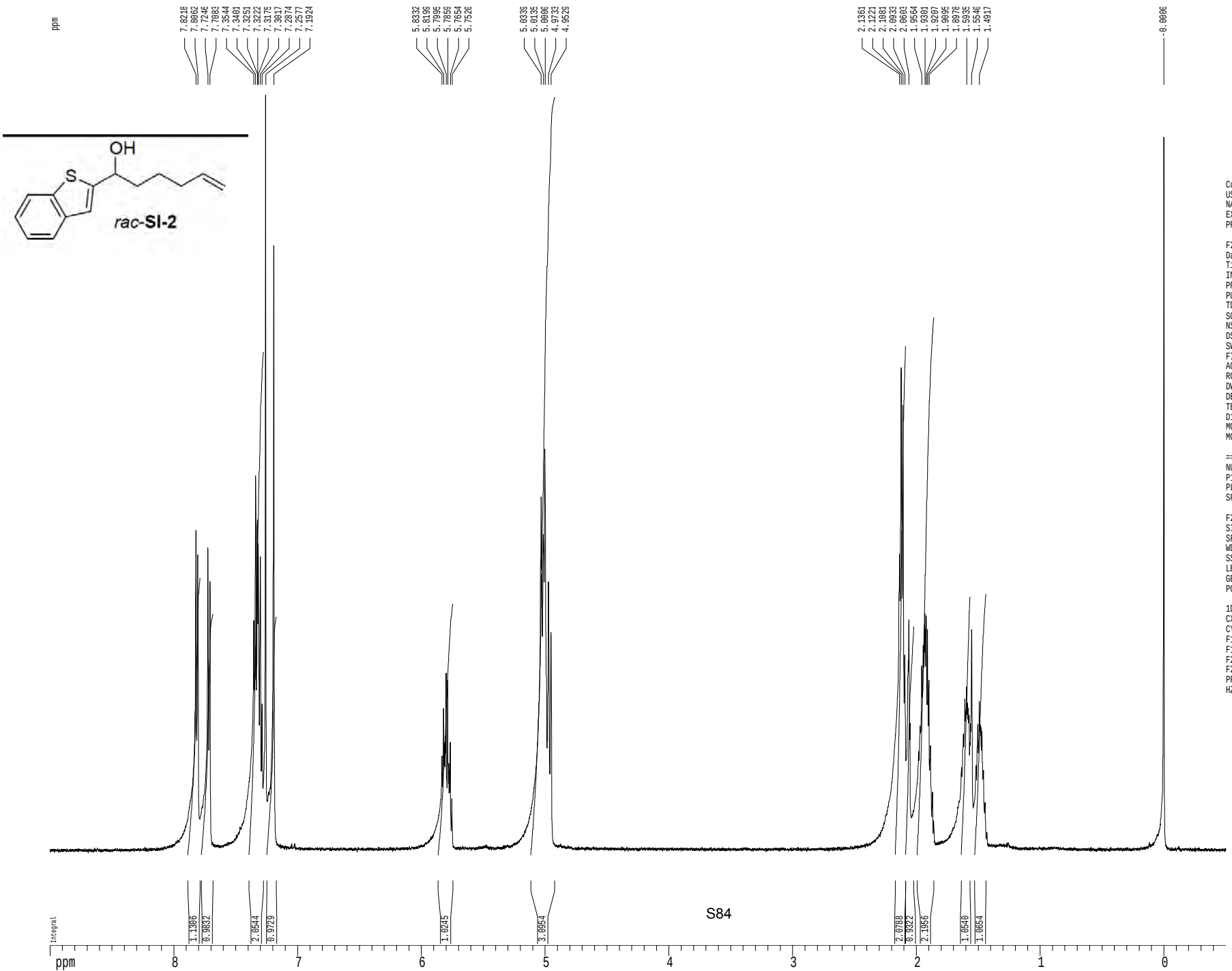
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804085 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            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-205-1HNMR
EXPNO 1
PROCNO 1

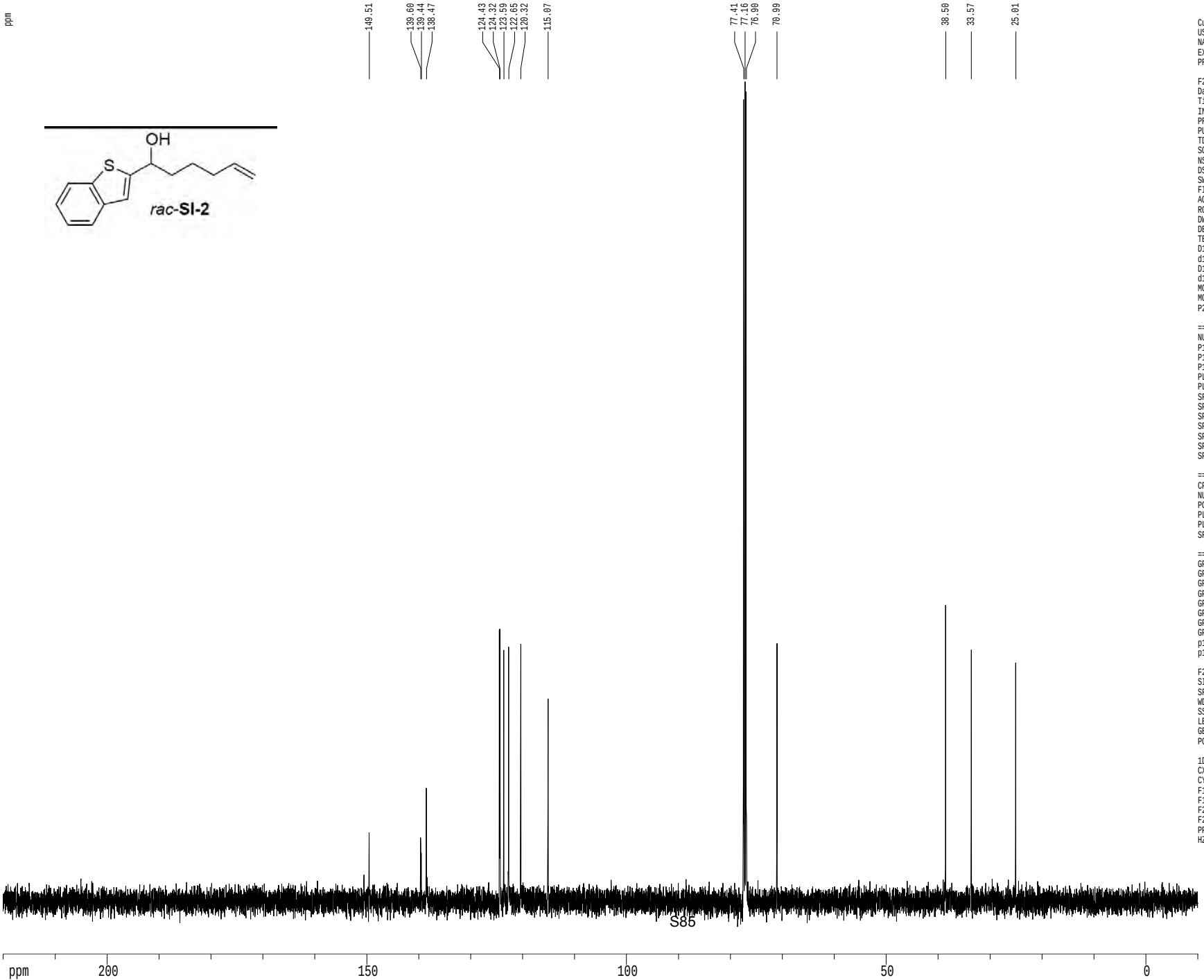
F2 - Acquisition Parameters
Date_ 20130918
Time 17.11
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 9
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200315 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-205-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130918
Time              17.13
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  161
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

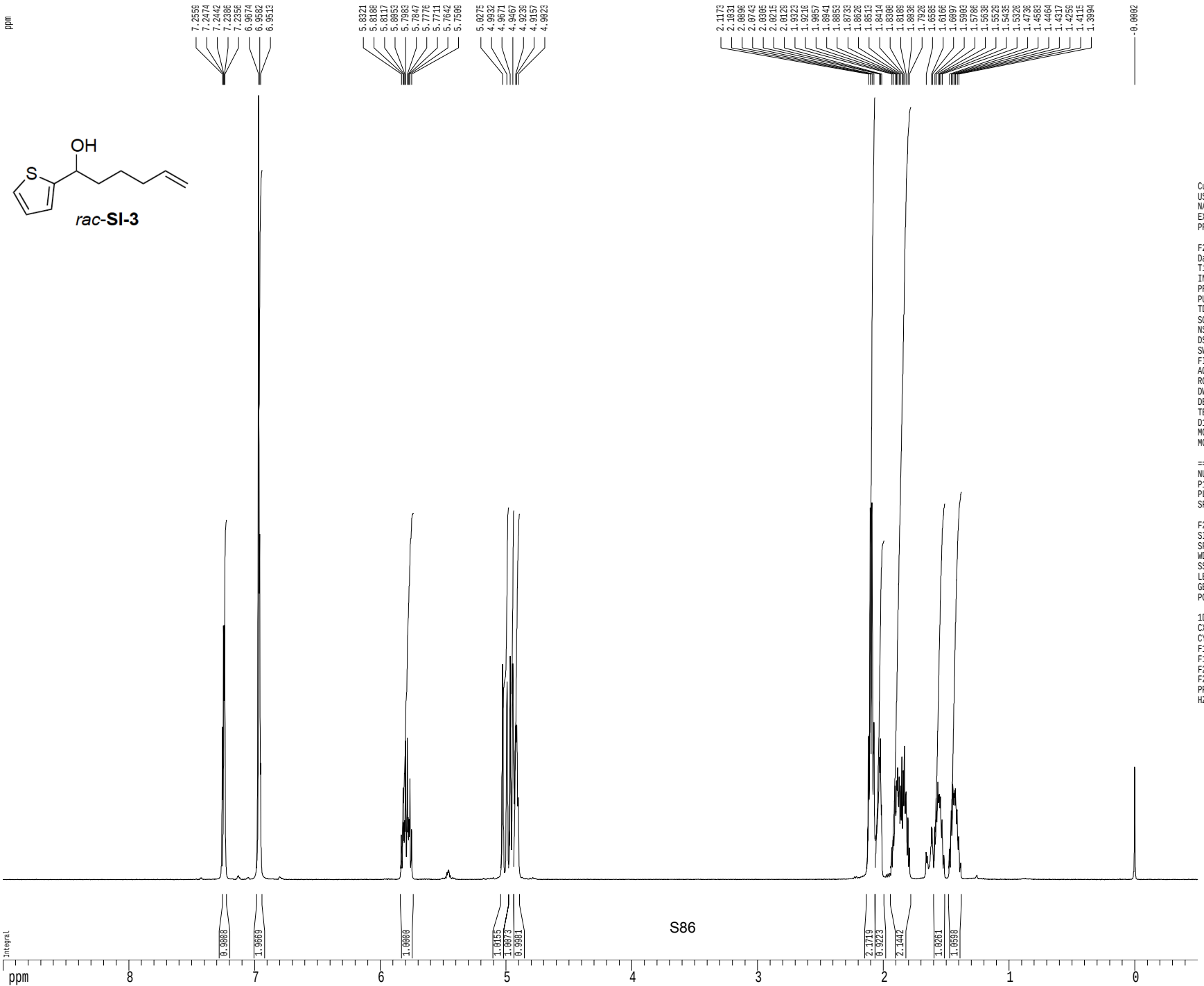
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804085 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM         10.08772 ppm/cm
HZCM        1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-294-1HNMR
EXPNO 1
PROCNO 1

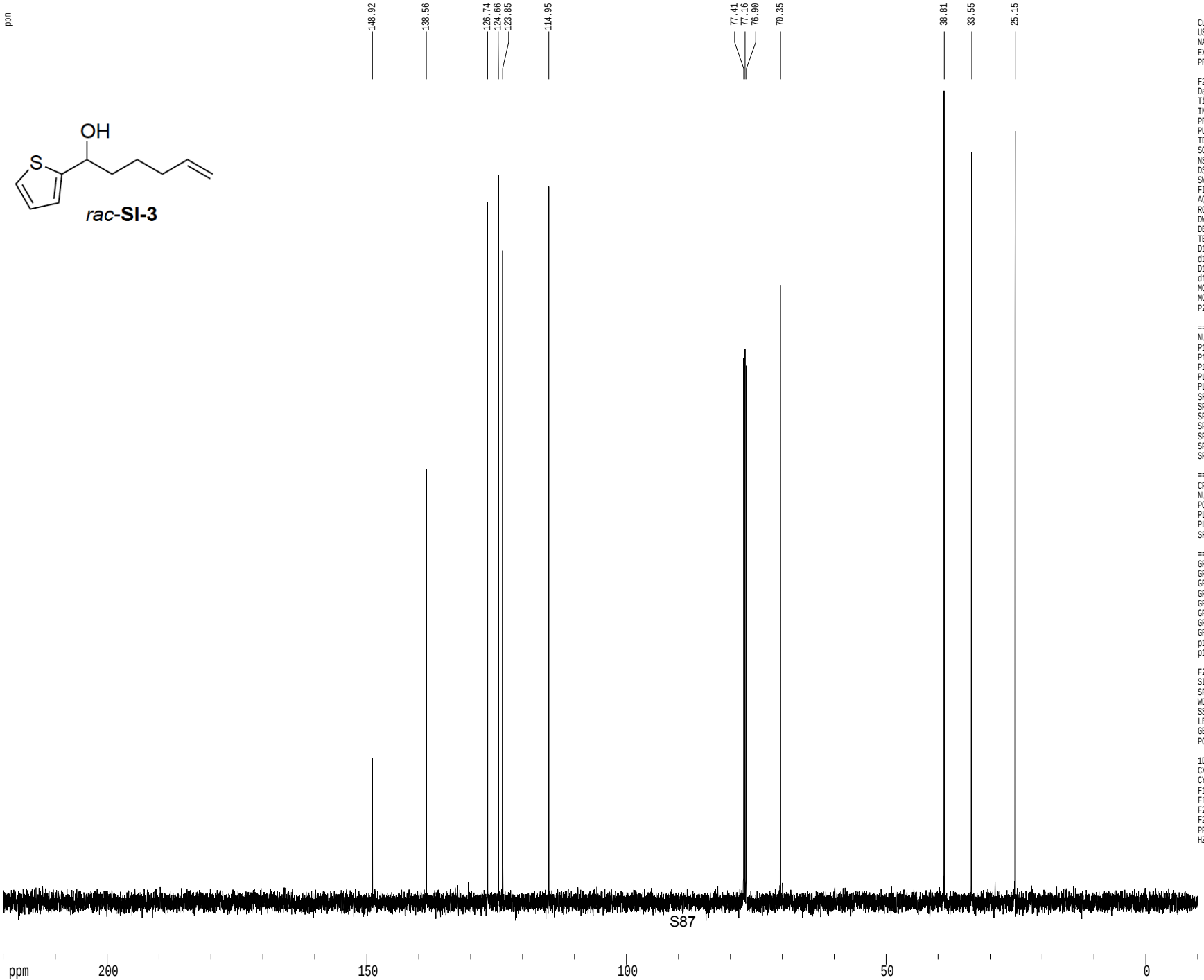
F2 - Acquisition Parameters
Date_ 20131209
Time 15.54
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200327 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-294-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131209
Time              15.56
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   61
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               31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1               3.20 dB
SP2               3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1            0.00 Hz
SPOFF2            0.00 Hz

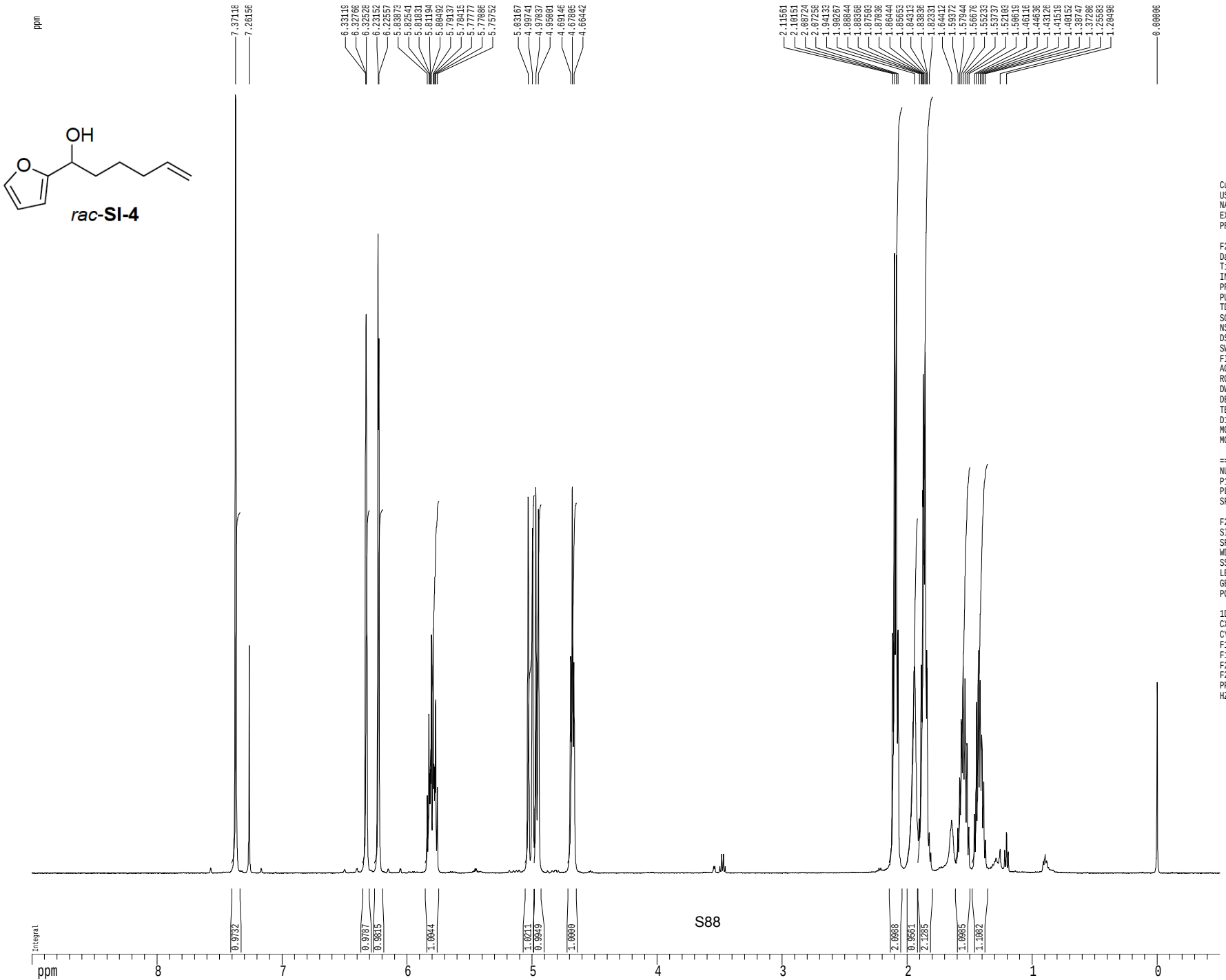
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2           100.00 usec
PL2               1.60 dB
PL12             24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804108 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM         10.08772 ppm/cm
HZCM        1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-290-1HNMR
EXPNO 1
PROCNO 1

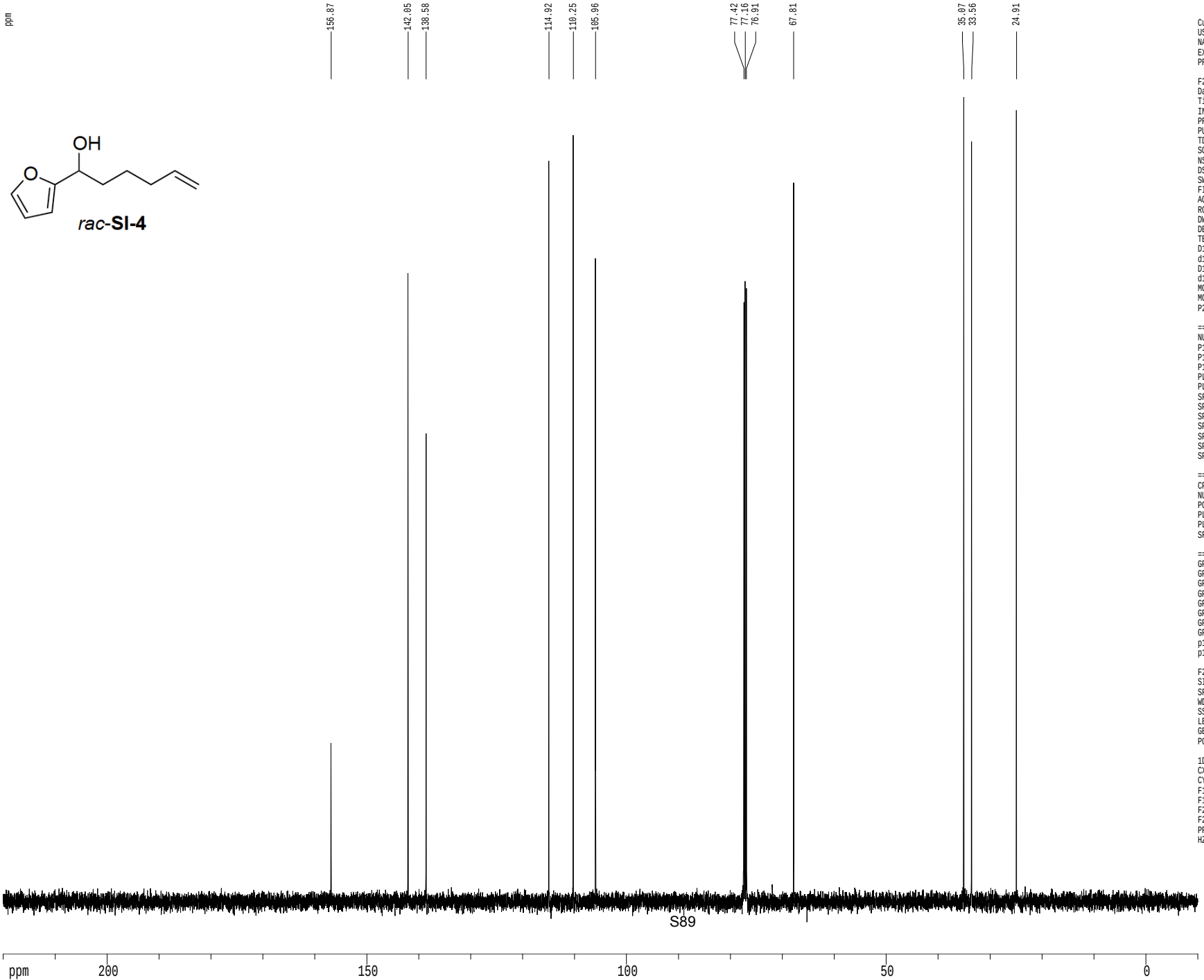
F2 - Acquisition Parameters
Date_ 20131209
Time 18.51
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200298 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-290-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131209
Time              18.53
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   83
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG               3649.1
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

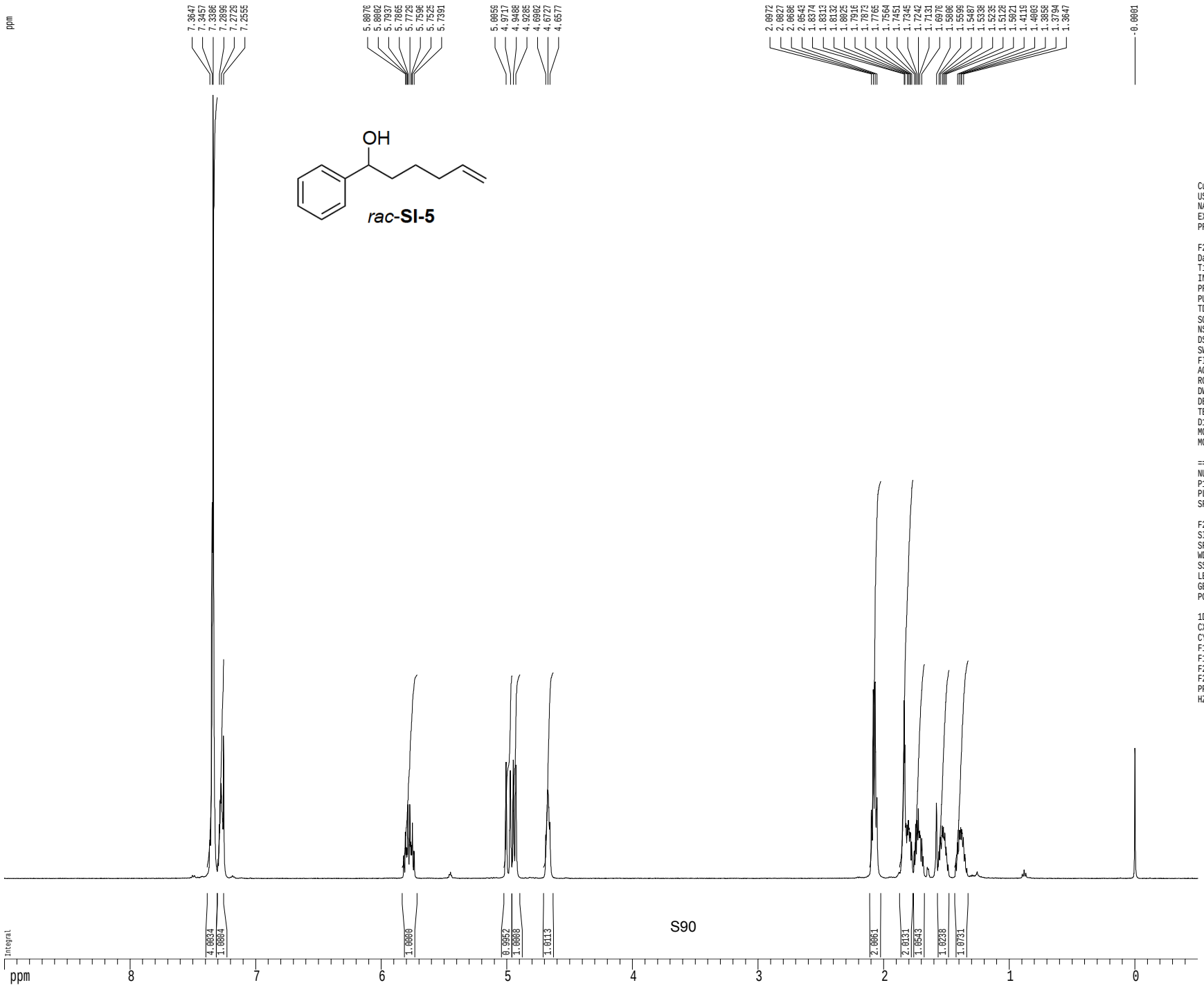
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804094 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             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER                mharri
NAME      MRH-VI-156-1HNMR
EXPNO                    1
PROCNO                   1

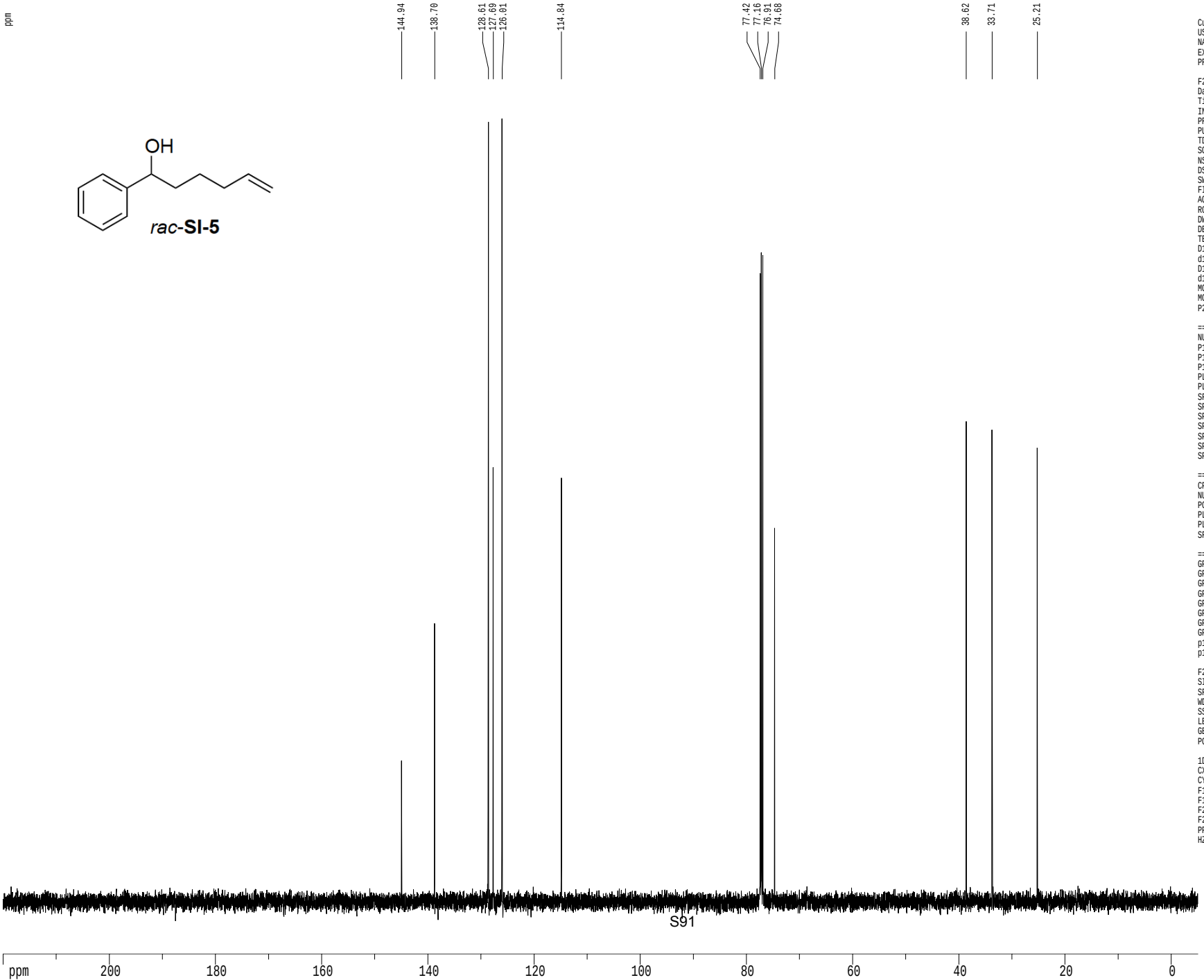
F2 - Acquisition Parameters
Date_            20140430
Time               15.48
INSTRUM          cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT            CDCl3
NS                     1
DS                     2
SWH             8012.820 Hz
FIDRES          0.098043 Hz
AQ             5.0998774 sec
RG                   6.3
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
SF01        500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF          500.2200351 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                9.000 ppm
F1               4501.98 Hz
F2P               -0.500 ppm
F2               -250.11 Hz
PPMCM            0.41667 ppm/cm
HZCM           208.42502 Hz/cm
```

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-156-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140430
Time              15.52
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  109
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG                13004
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

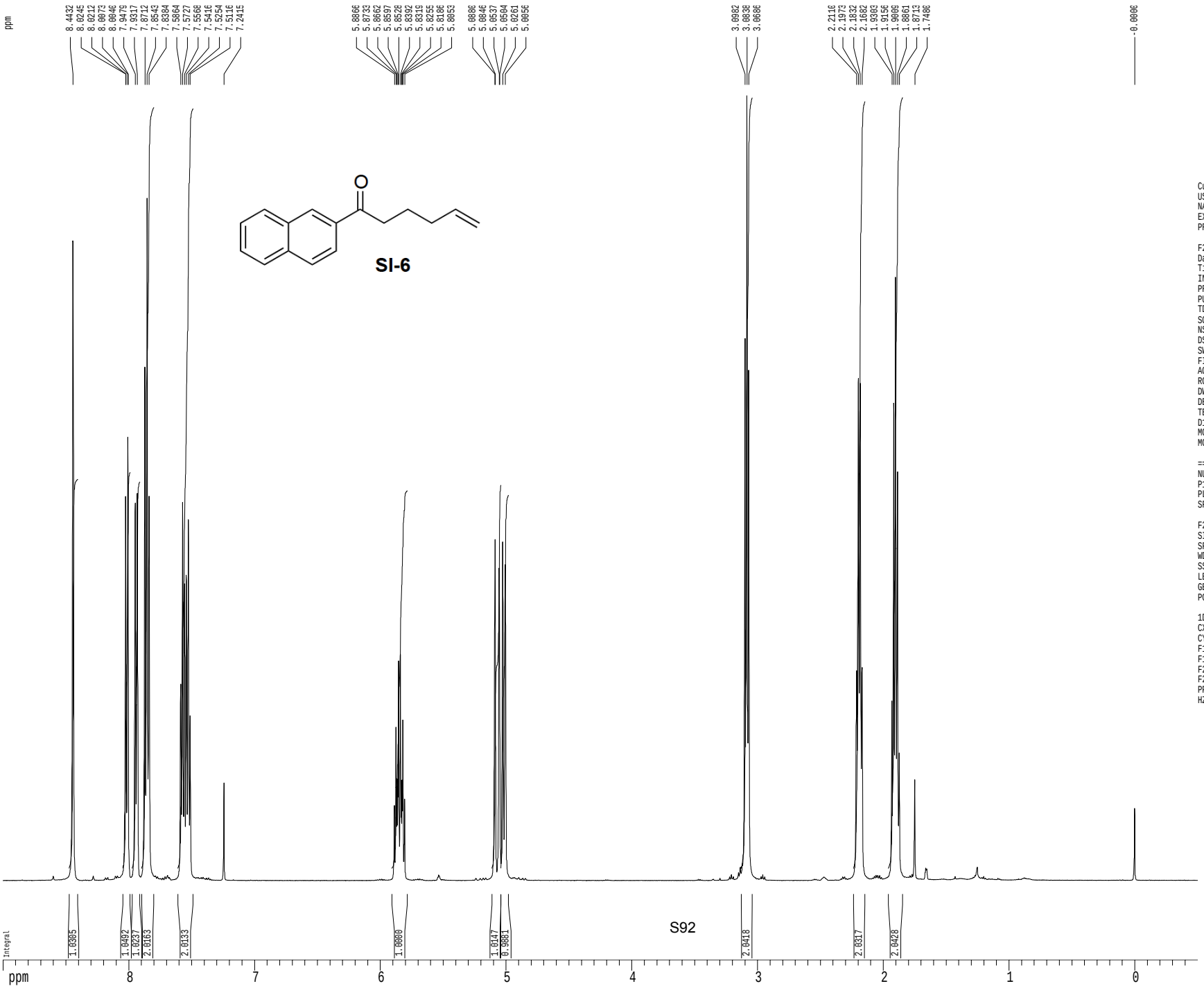
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804094 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             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-284-1HNMR
EXPNO 1
PROCNO 1

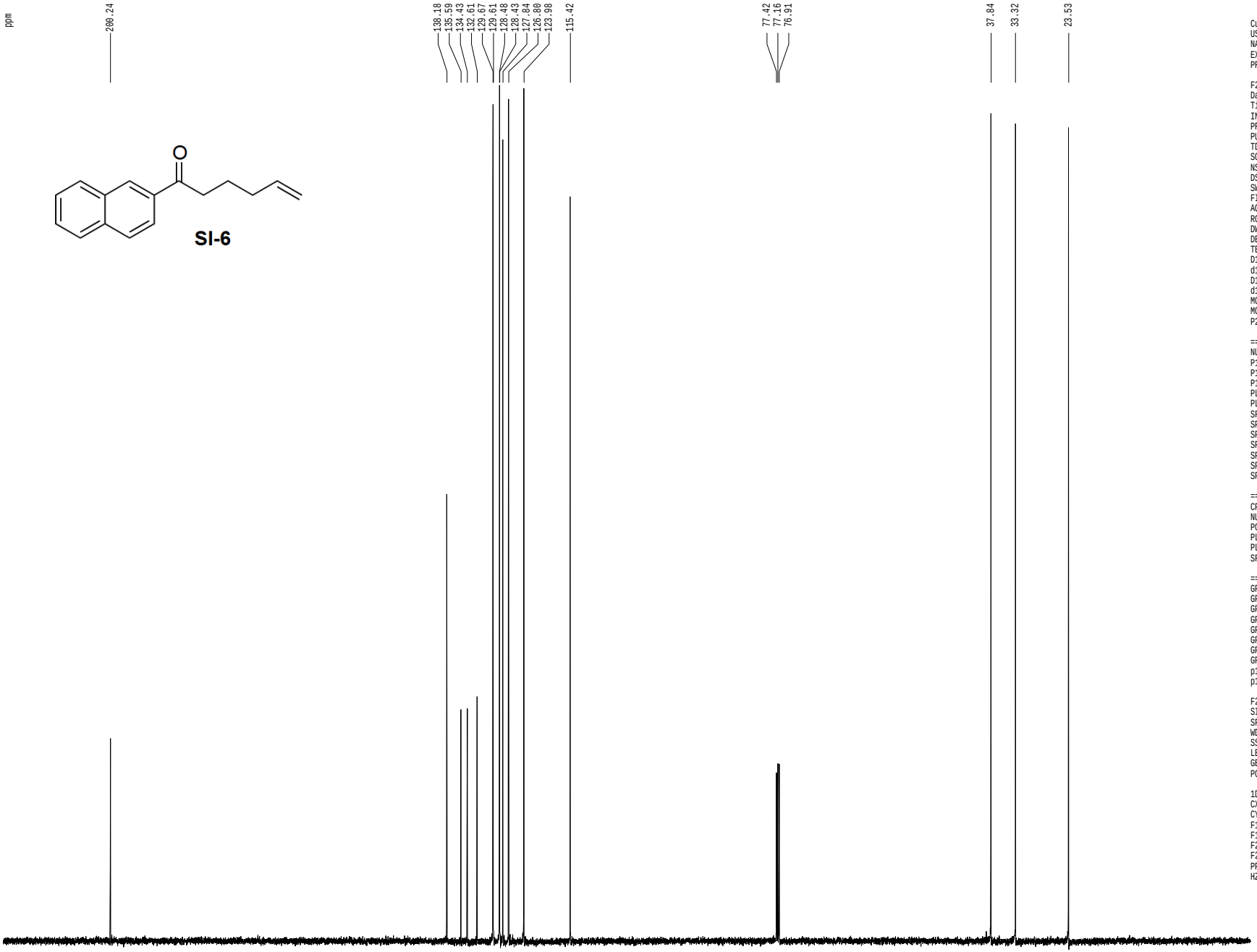
F2 - Acquisition Parameters
Date_ 20131025
Time 17.23
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 5
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 3.6
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200399 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-284-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131025
Time              17.26
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   91
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG               5160.6
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

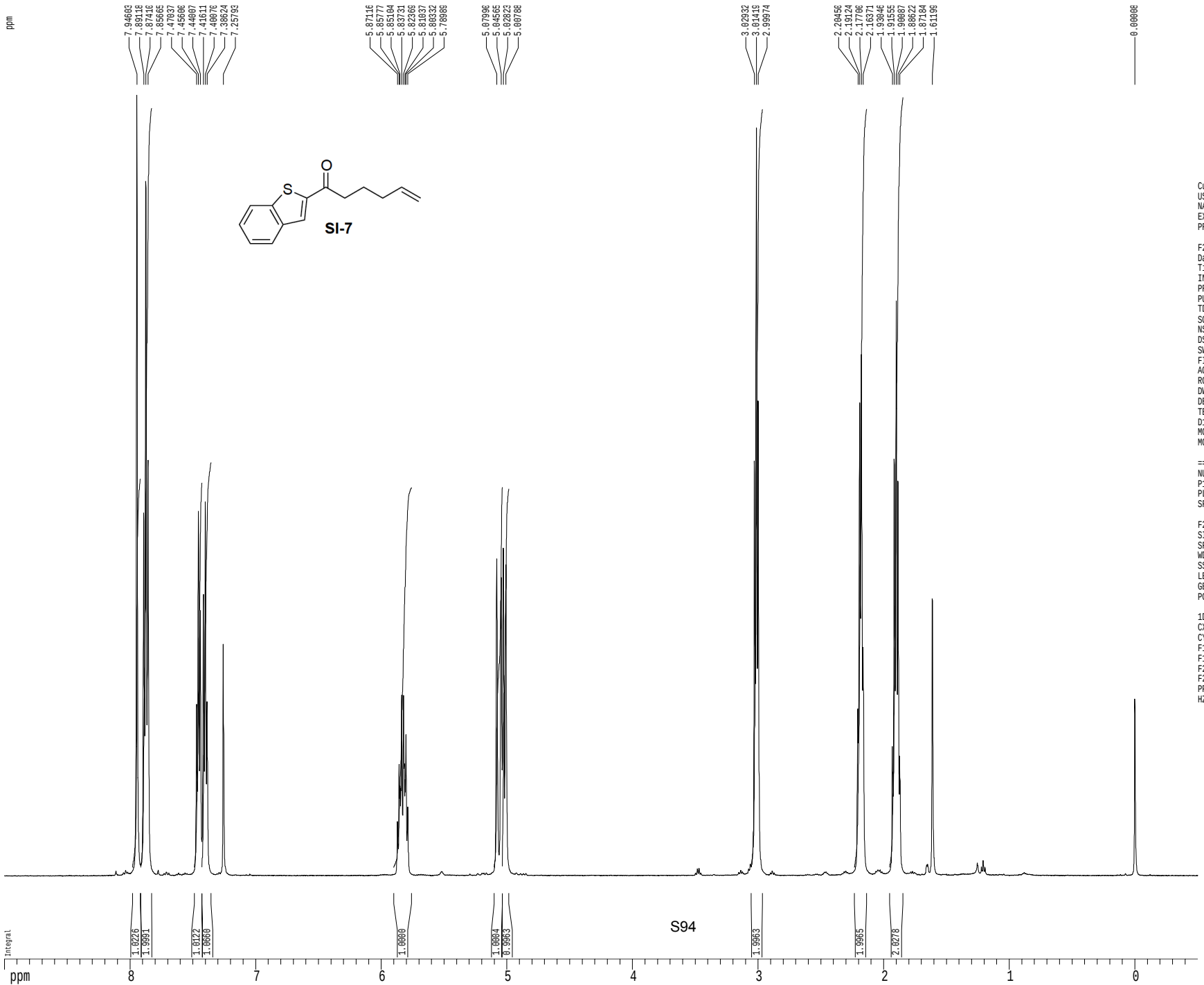
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804182 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             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83765 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-266-1HNMR
EXPNO 1
PROCNO 1

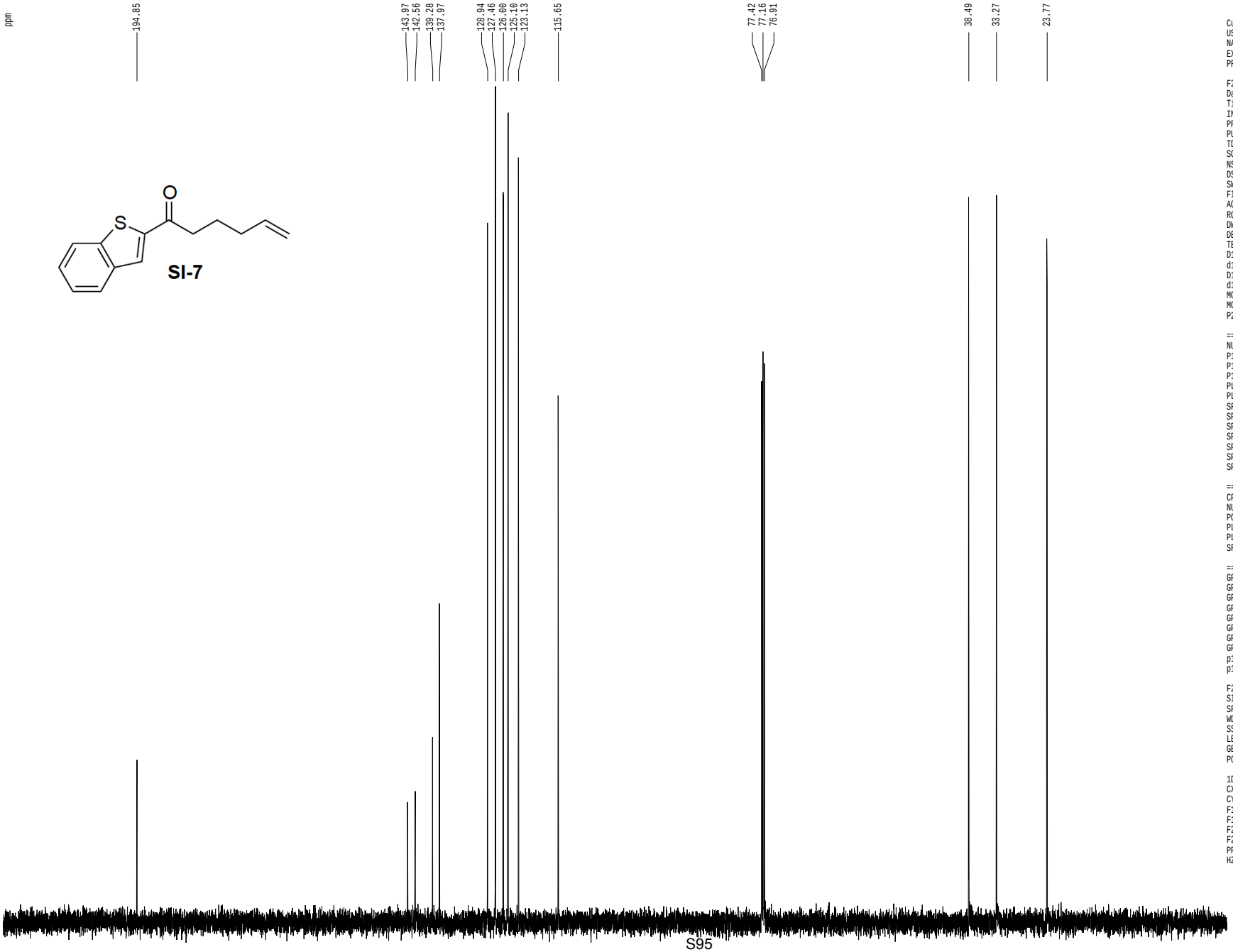
F2 - Acquisition Parameters
Date_ 20131021
Time 17.14
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200325 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-266-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131021
Time             17.17
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                 101
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               31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1              3.20 dB
SP2              3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2     Crp60comp.4
SPOFF1            0.00 Hz
SPOFF2            0.00 Hz

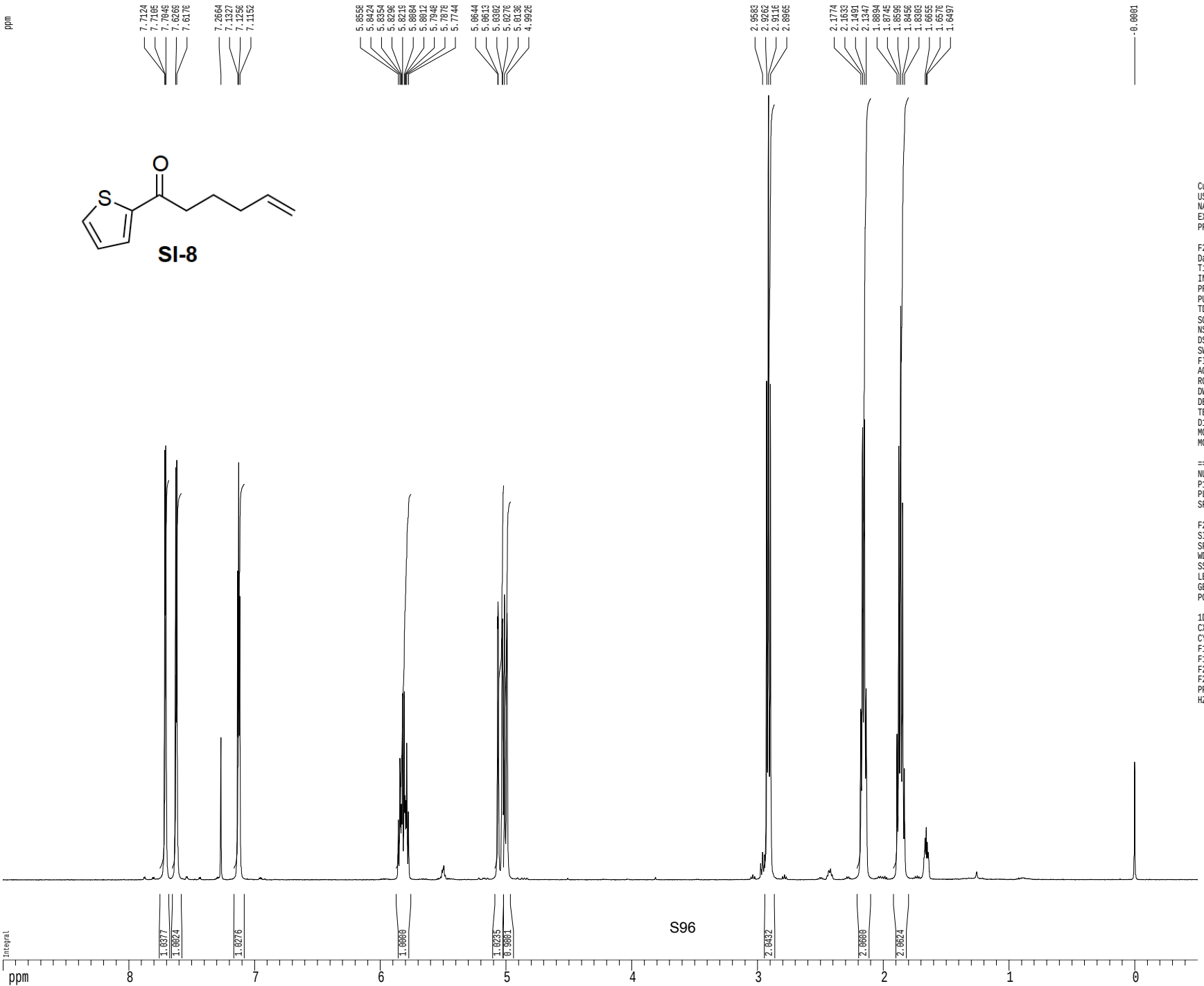
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2              1.60 dB
PL12            24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804108 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM         10.08772 ppm/cm
HZCM        1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-293-1HNMR
EXPNO 1
PROCNO 1

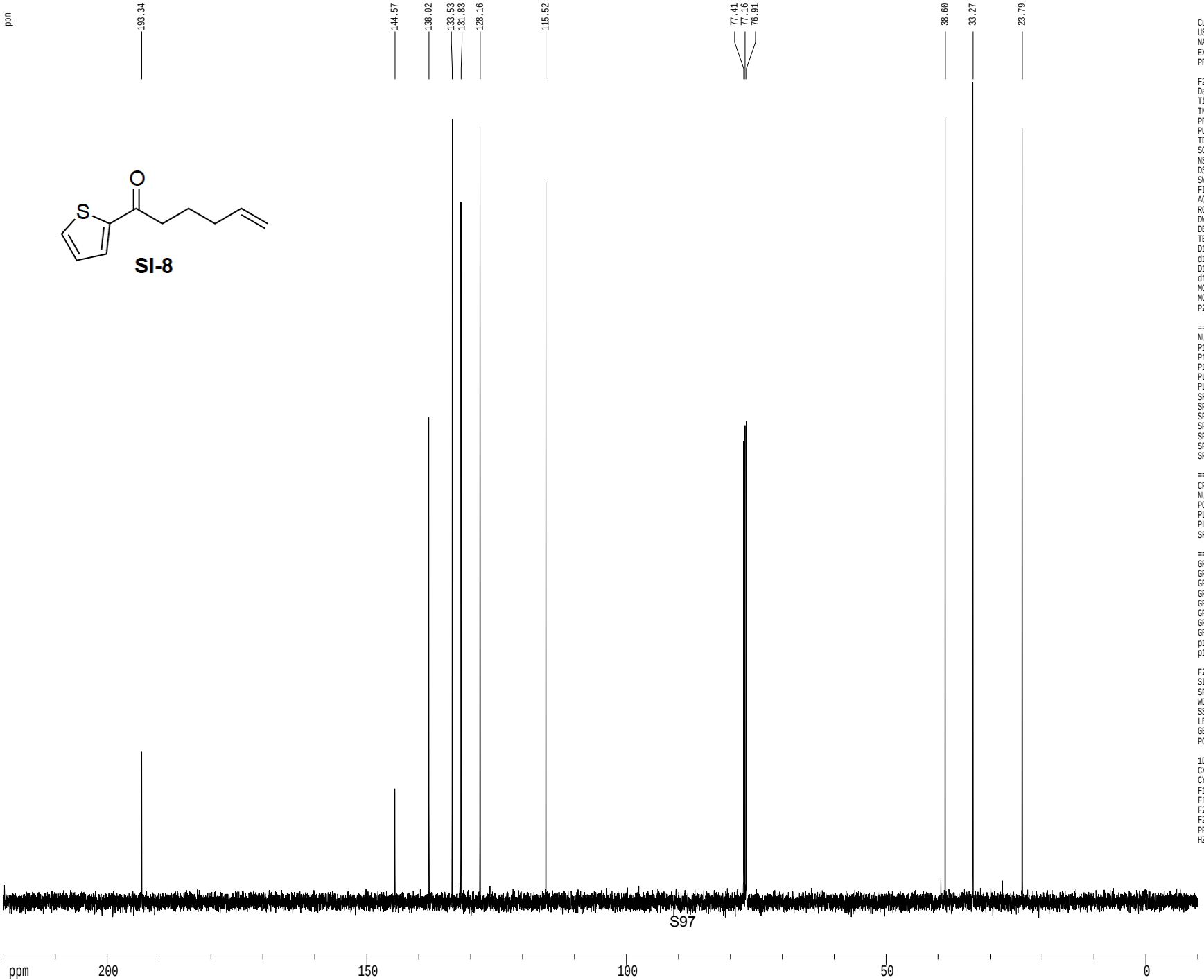
F2 - Acquisition Parameters
Date_ 20131209
Time 15.59
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200278 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-293-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131209
Time             16.01
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                  71
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG             11585.2
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

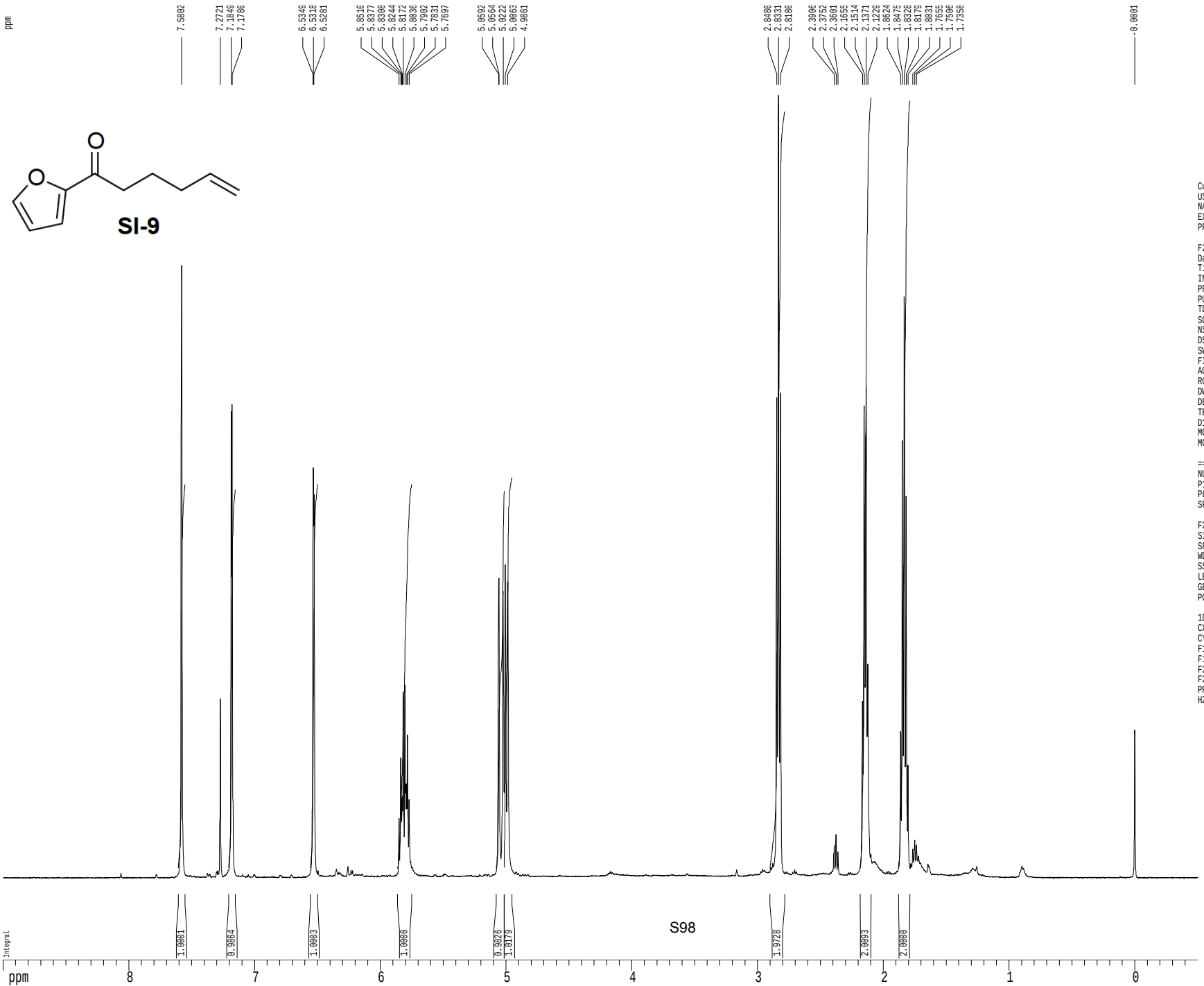
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804113 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             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MOK-I-066-1HNMR
EXPNO 1
PROCNO 1

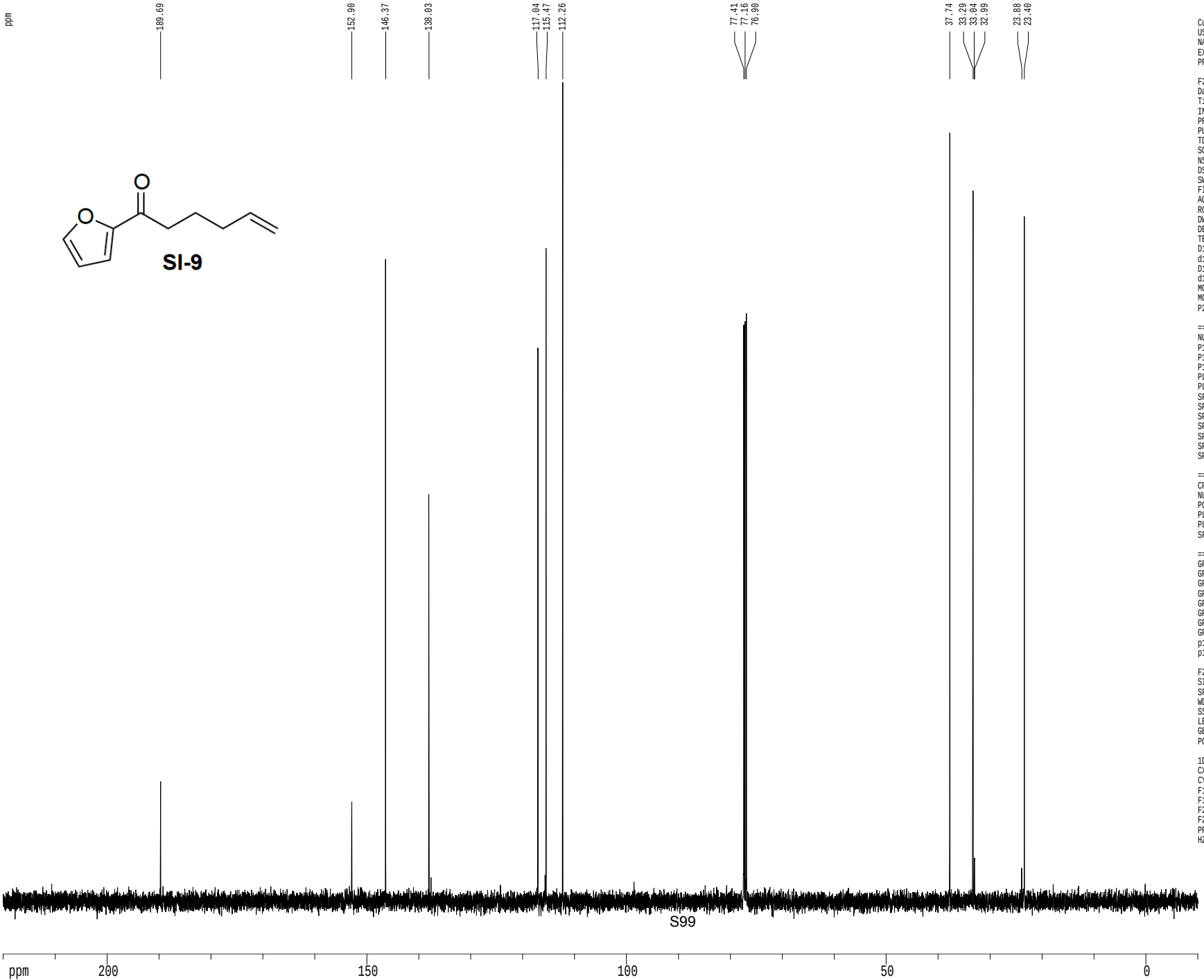
F2 - Acquisition Parameters
Date_ 20131209
Time 18.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200247 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MOK-I-066-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131209
Time              18.36
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   85
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG               4597.6
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

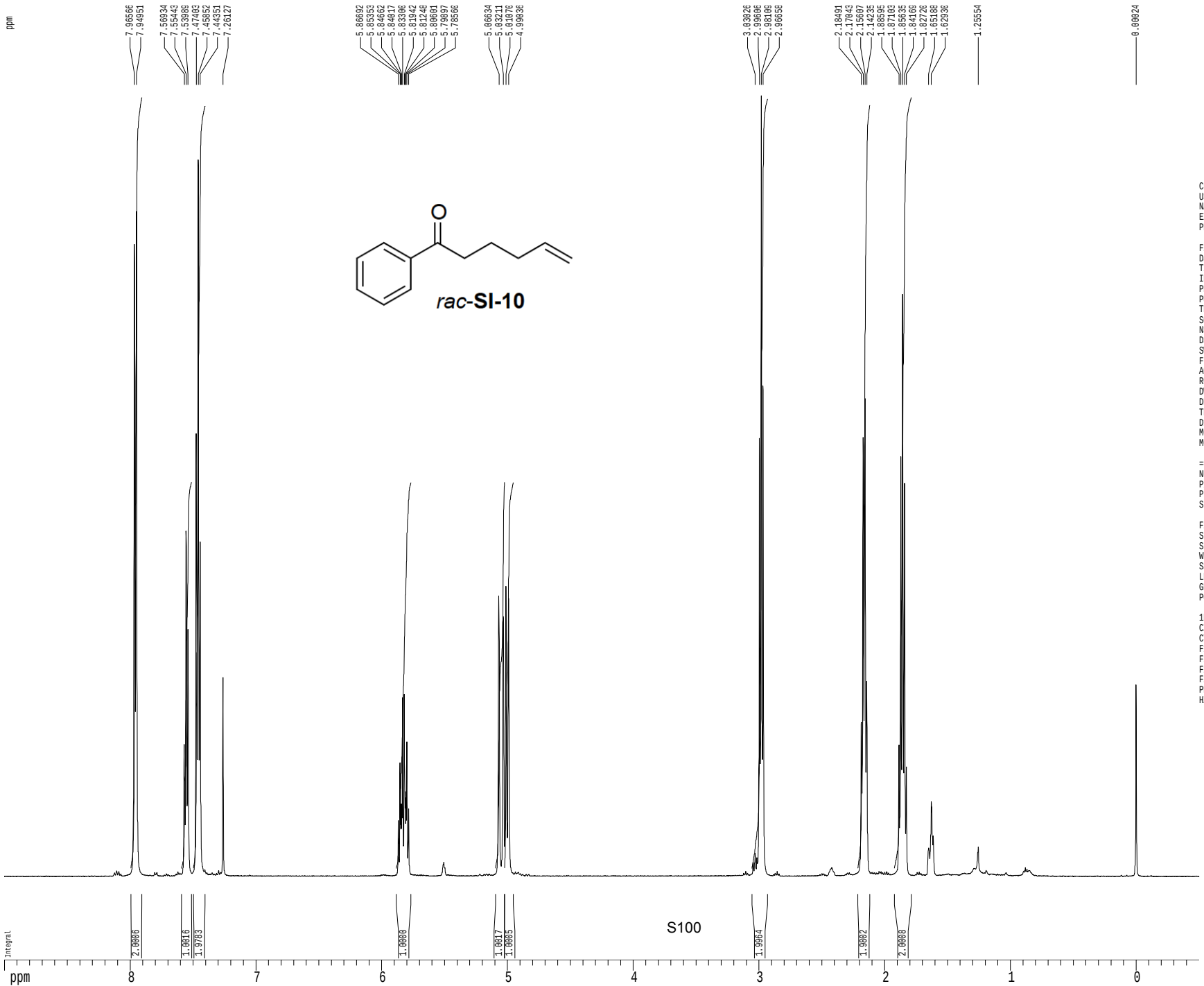
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804103 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             220.000 ppm
F1             27671.69 Hz
F2P             -10.000 ppm
F2             -1257.80 Hz
PPMCM           10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME      MRH-VI-154-1HNMR
EXPNO                  1
PROCNO                 1

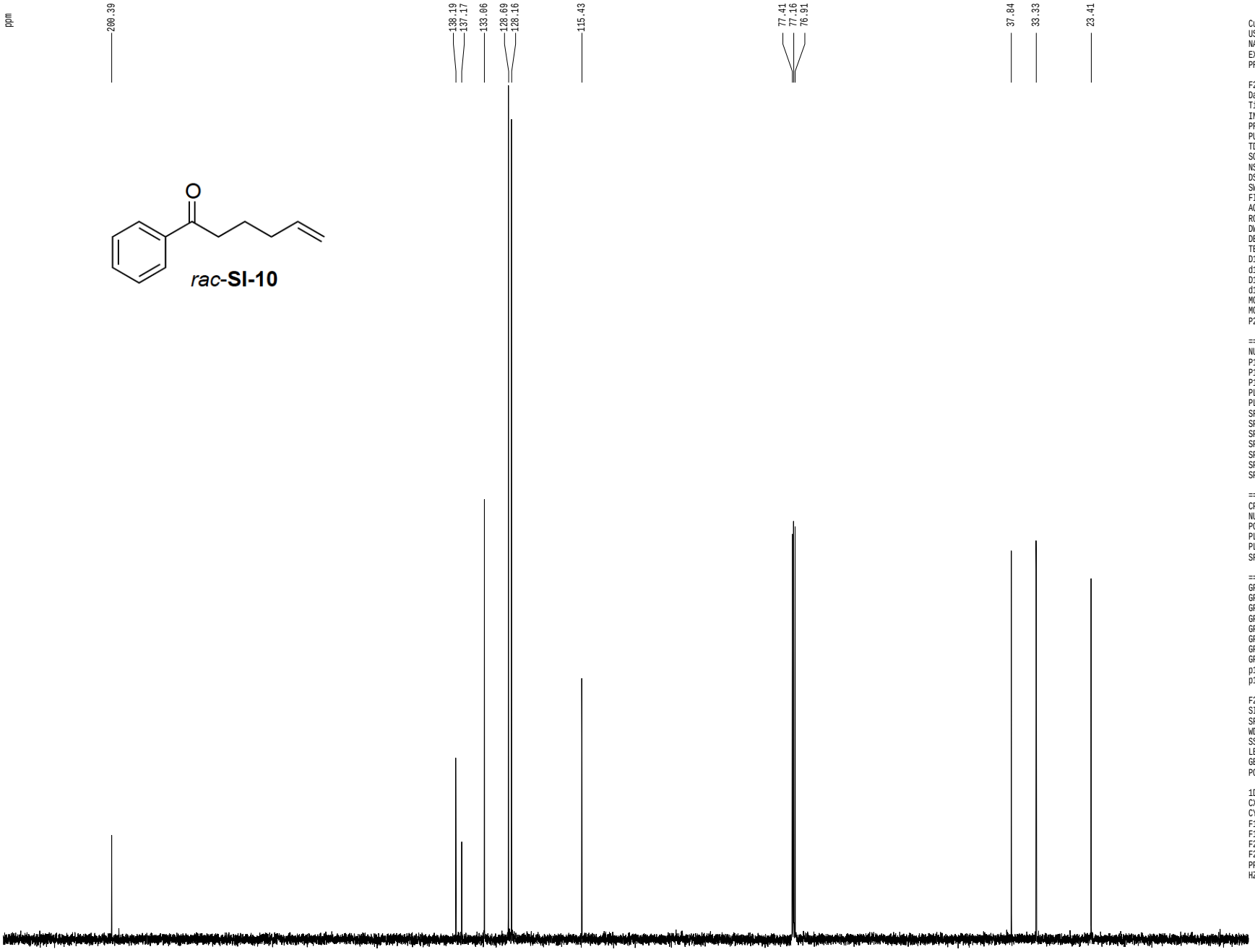
F2 - Acquisition Parameters
Date_           20140511
Time              21.23
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                  6.3
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          500.2200319 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-154-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140511
Time              21.25
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  101
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG               2298.8
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

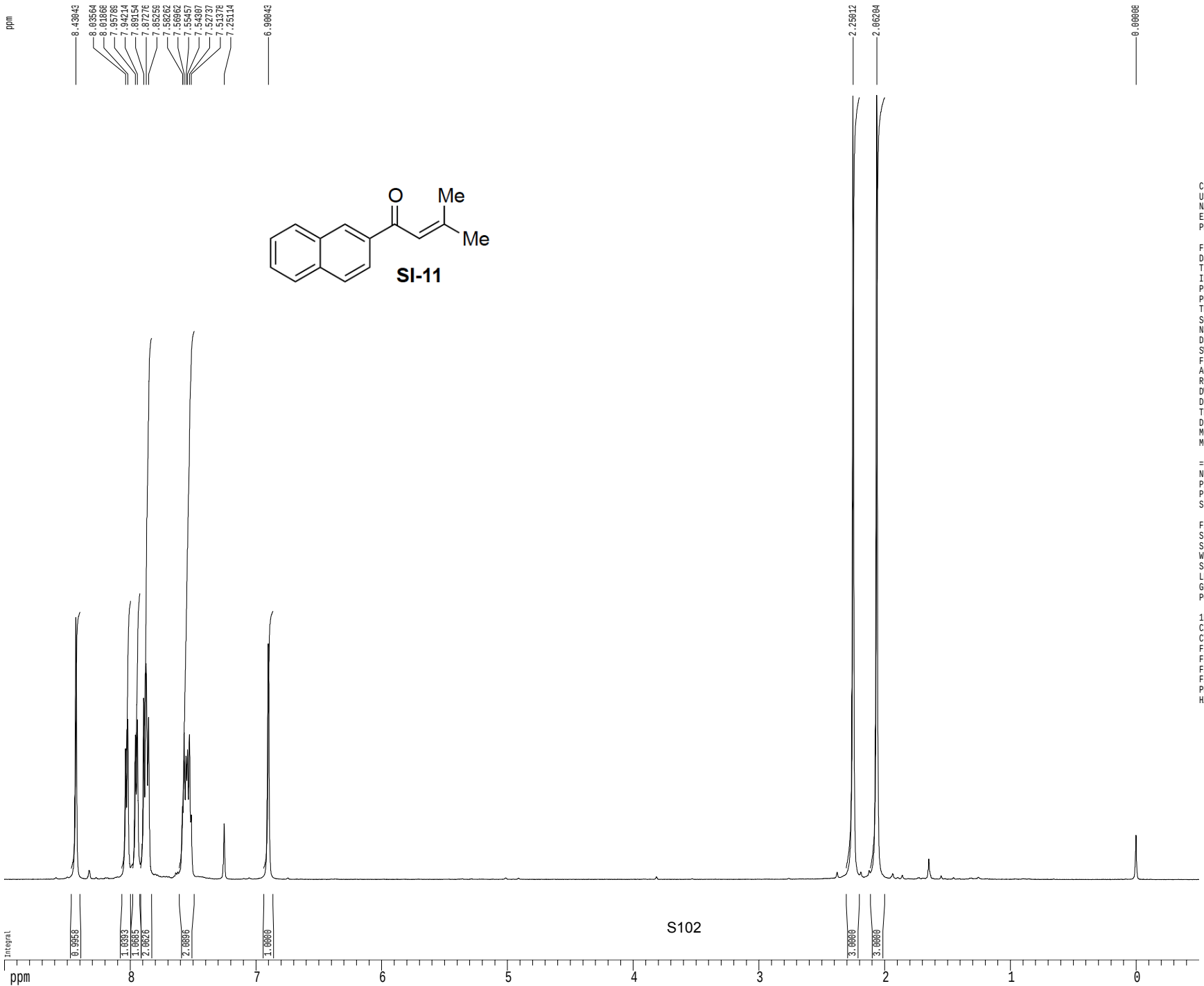
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804103 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             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-24-1HNMR
EXPNO 1
PROCNO 1

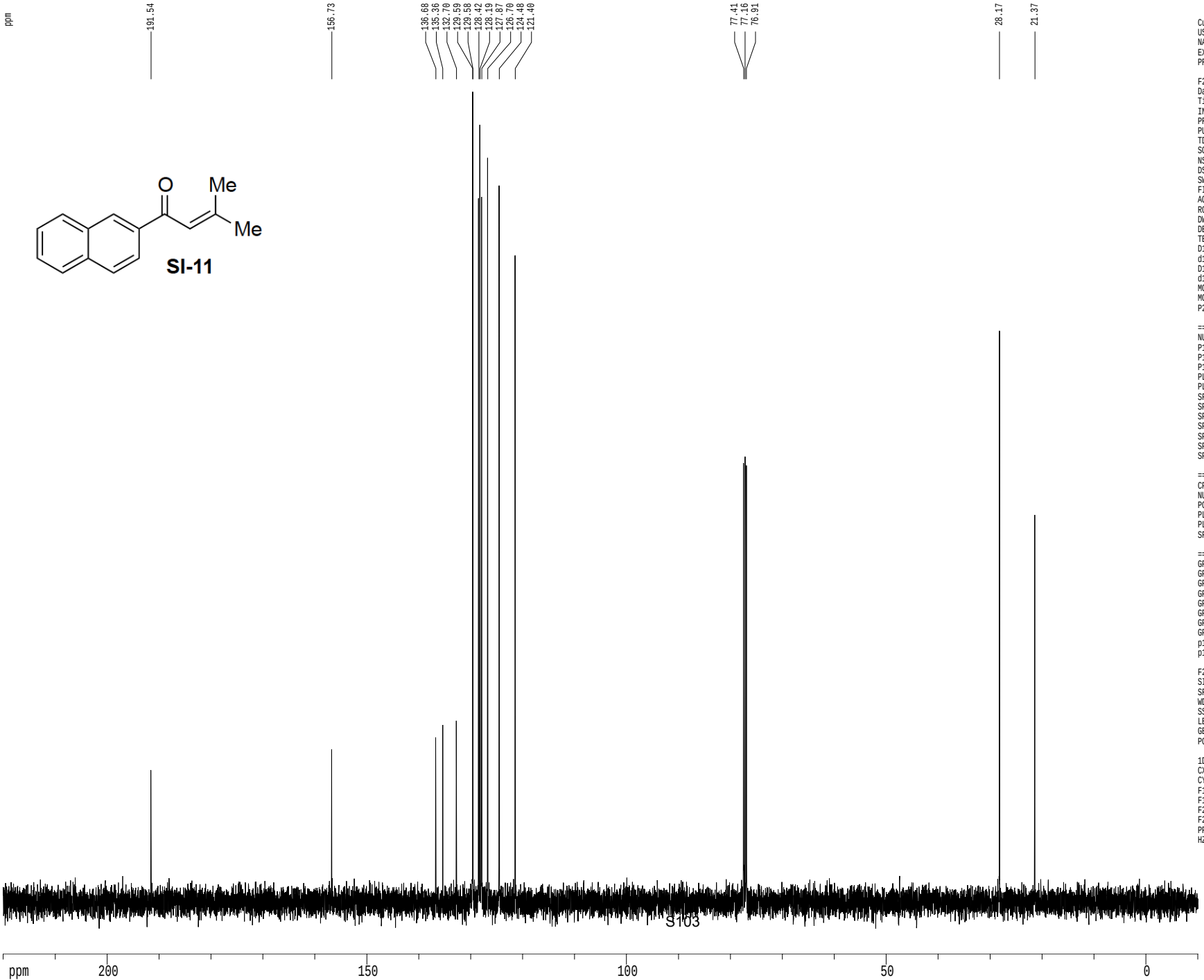
F2 - Acquisition Parameters
Date_ 20140116
Time 17.07
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 6
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200330 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-24-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140116
Time              17.09
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   76
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               31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1               3.20 dB
SP2               3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1            0.00 Hz
SPOFF2            0.00 Hz

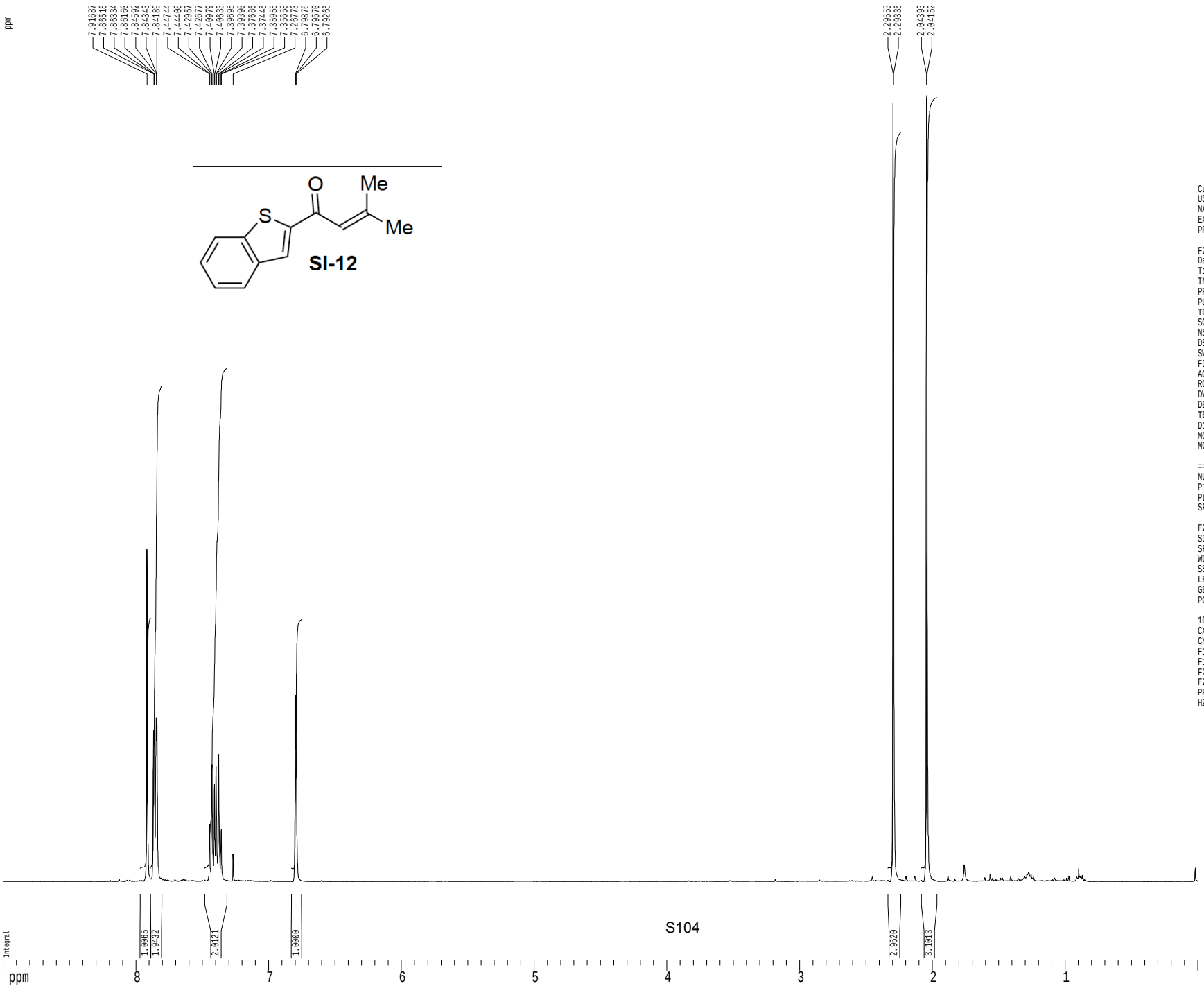
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               1.60 dB
PL12             24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804117 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mkonev
NAME MOKA218_hr
EXPNO 1
PROCNO 1

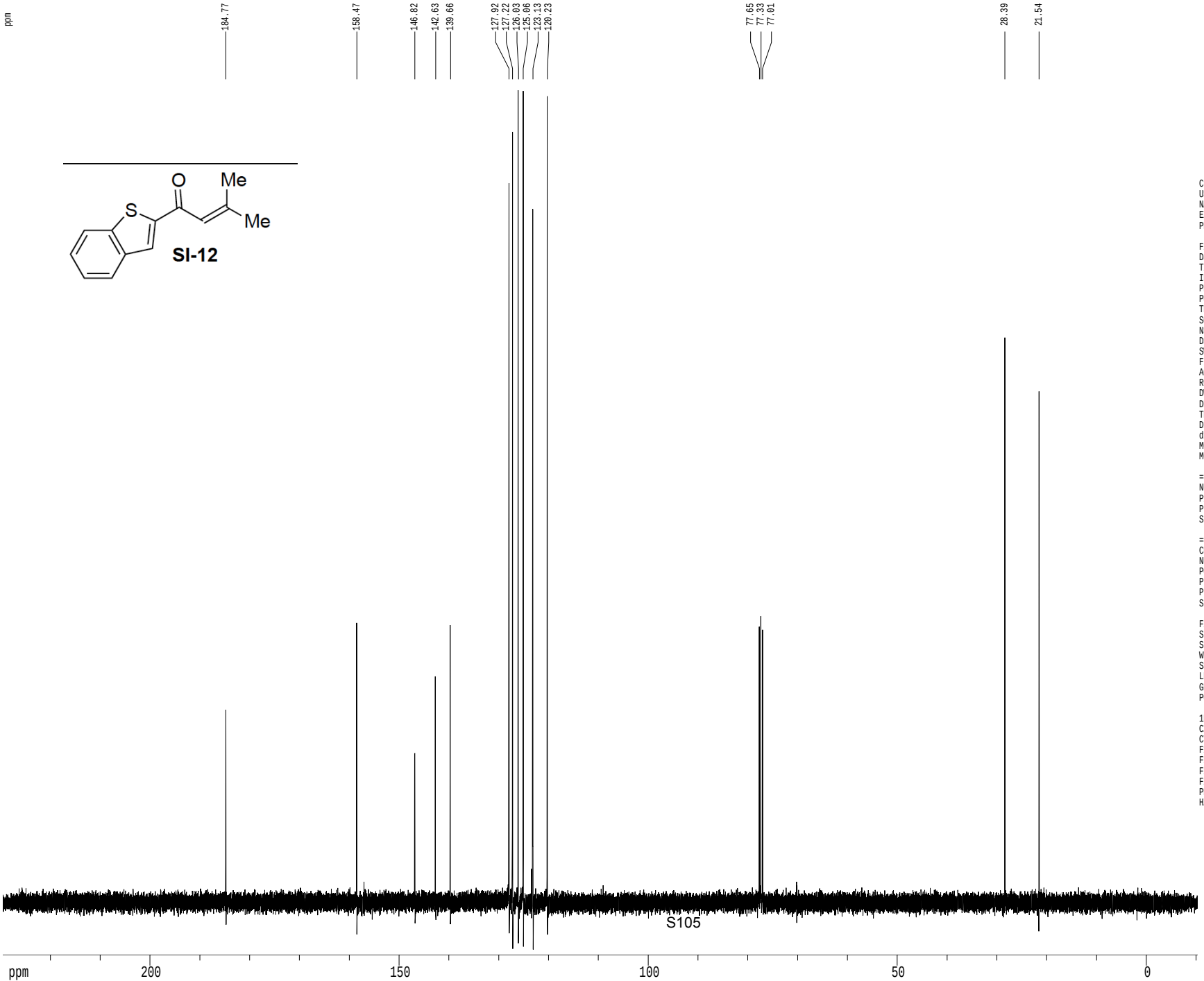
F2 - Acquisition Parameters
Date_ 20140505
Time 6.01
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 71.8
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== 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 no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm

13C spectrum with 1H decoupling




```
Current Data Parameters
USER             mkonev
NAME          MOKA218_cr
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140505
Time                6.04
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  152
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

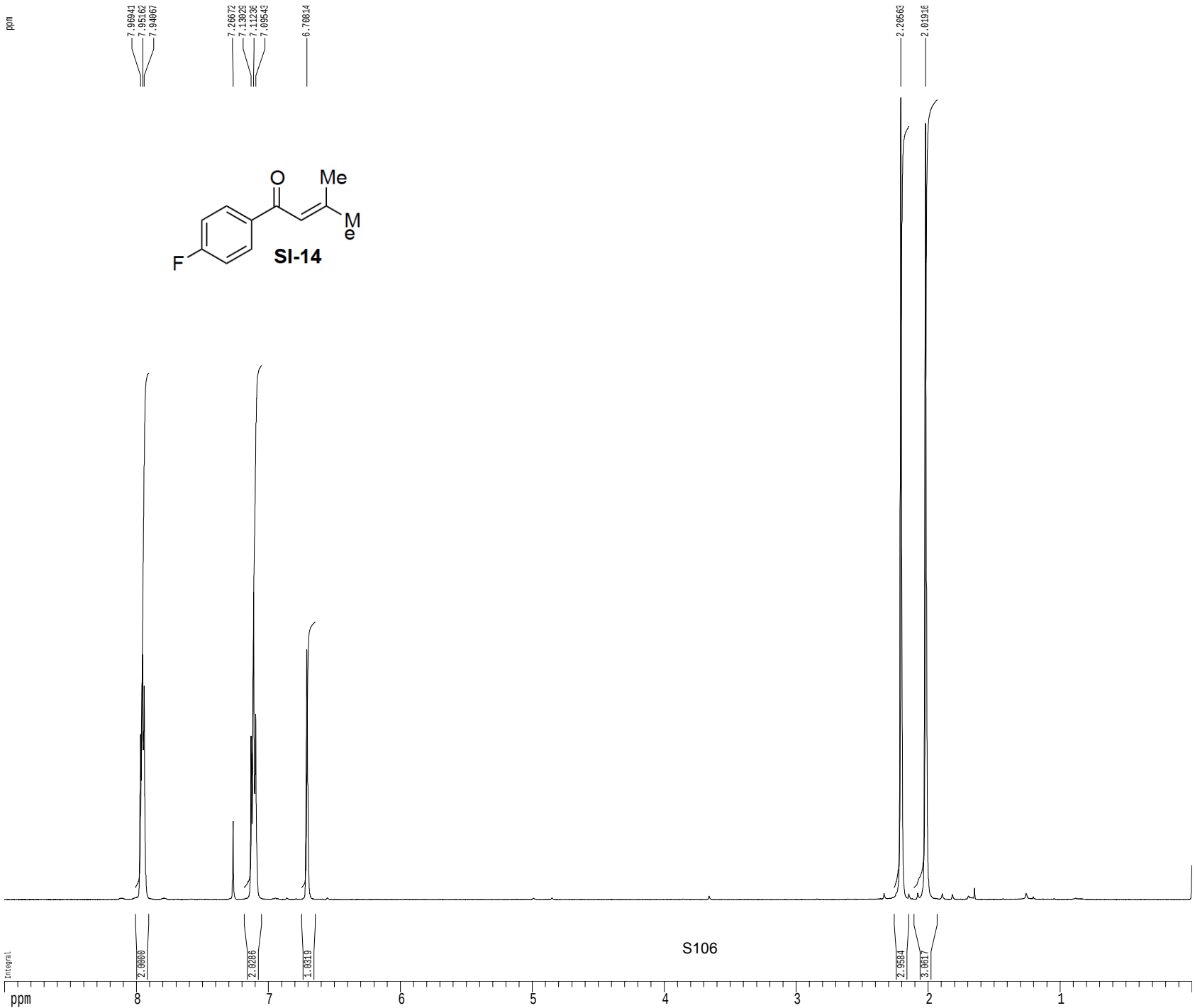
======== CHANNEL f1 =======
NUC1                13C
P1                 7.75 usec
PL1               -3.00 dB
SFO1        100.6237964 MHz

======== 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                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.50 cm
F1P             229.496 ppm
F1             23090.21 Hz
F2P             -10.579 ppm
F2            -1064.37 Hz
PPMCM          10.52959 ppm/cm
HZCM         1059.41138 Hz/cm
```

1H spectrum



Current Data Parameters
USER mkonev
NAME MOKA073h-char
EXPNO 1
PROCNO 1

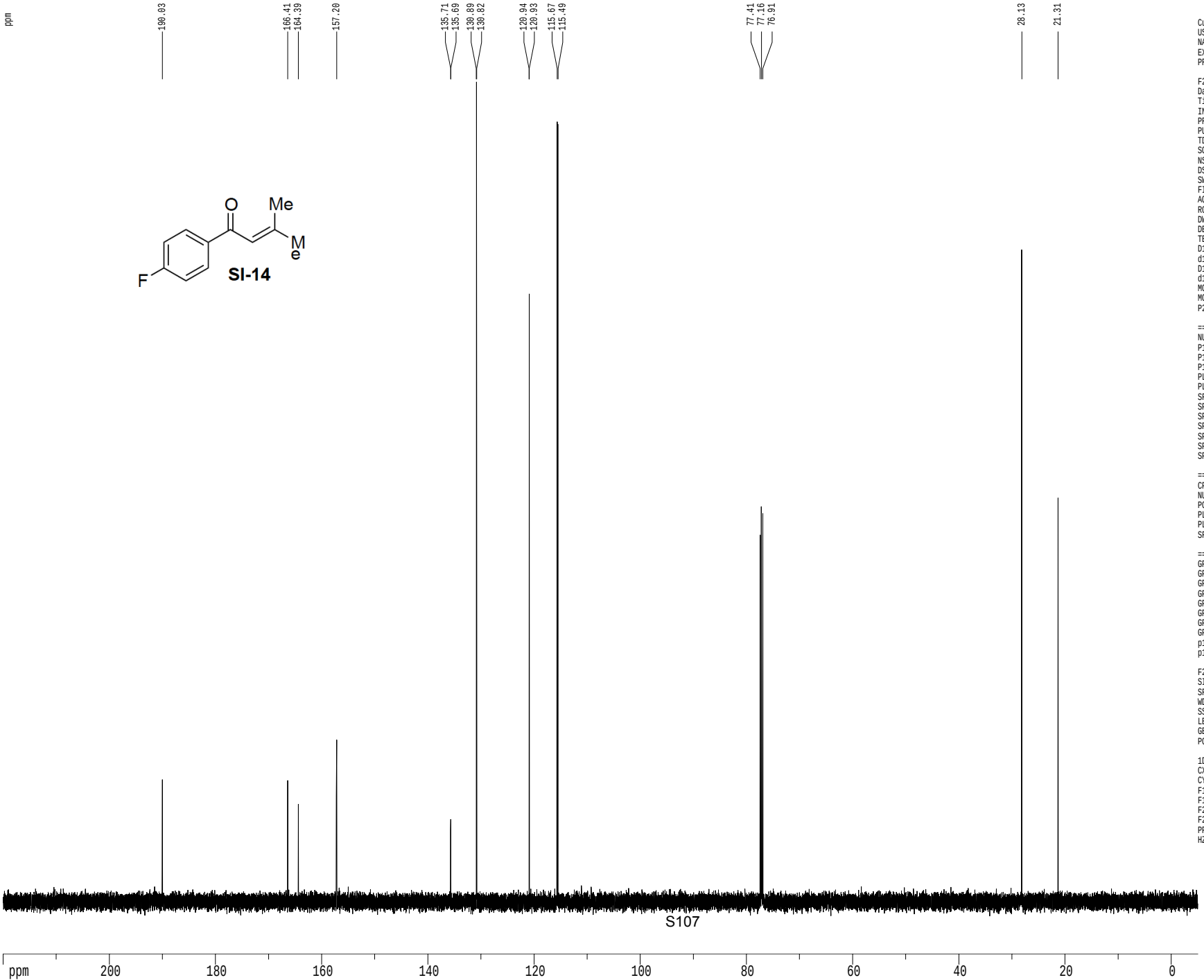
F2 - Acquisition Parameters
Date_ 20131120
Time 1.24
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200276 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.15 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.40632 ppm/cm
HZCM 203.25119 Hz/cm

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




```
Current Data Parameters
USER              mkonev
NAME        MOKA073c-char
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131120
Time                 1.27
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                  65536
SOLVENT             CDCl3
NS                    233
DS                     16
SWH             30303.031 Hz
FIDRES           0.462388 Hz
AQ              1.0813940 sec
RG                11585.2
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                 31.00 usec

======== CHANNEL f1 ========
NUC1                  13C
P1                  15.50 usec
P11                500.00 usec
P12               2000.00 usec
PL0                120.00 dB
PL1                 -1.00 dB
SFO1          125.7942548 MHz
SP1                  3.20 dB
SP2                  3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1               0.00 Hz
SPOFF2               0.00 Hz

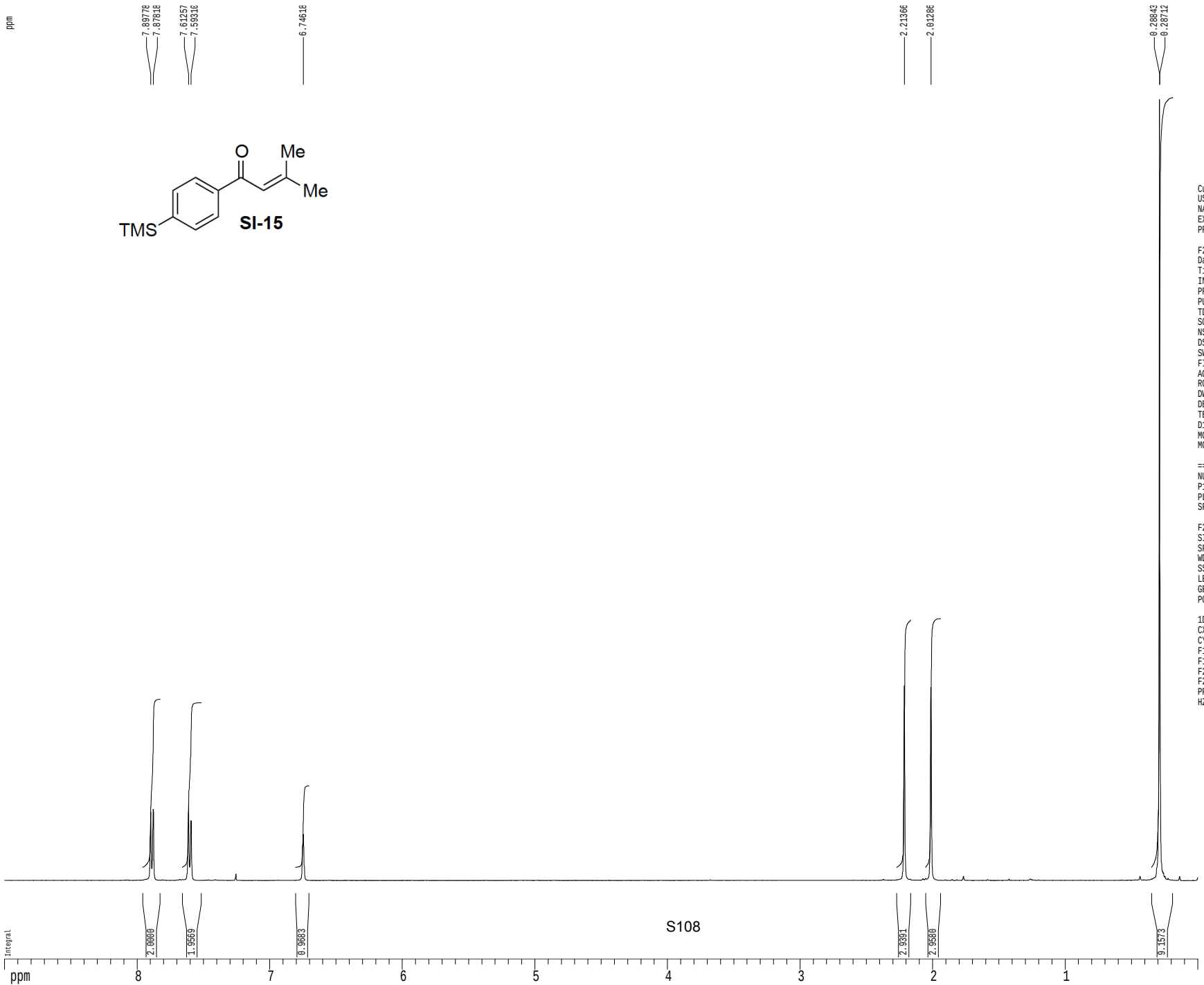
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2          500.2225011 MHz

====== 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            125.7804094 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     2.00

1D NMR plot parameters
CX                    22.80 cm
CY                    15.65 cm
F1P                 220.000 ppm
F1                27671.69 Hz
F2P                  -5.000 ppm
F2                 -628.90 Hz
PPMCM               9.86842 ppm/cm
HZCM             1241.25415 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mkonev
NAME       MOKA072h-char
EXPNO                 1
PROCNO                1

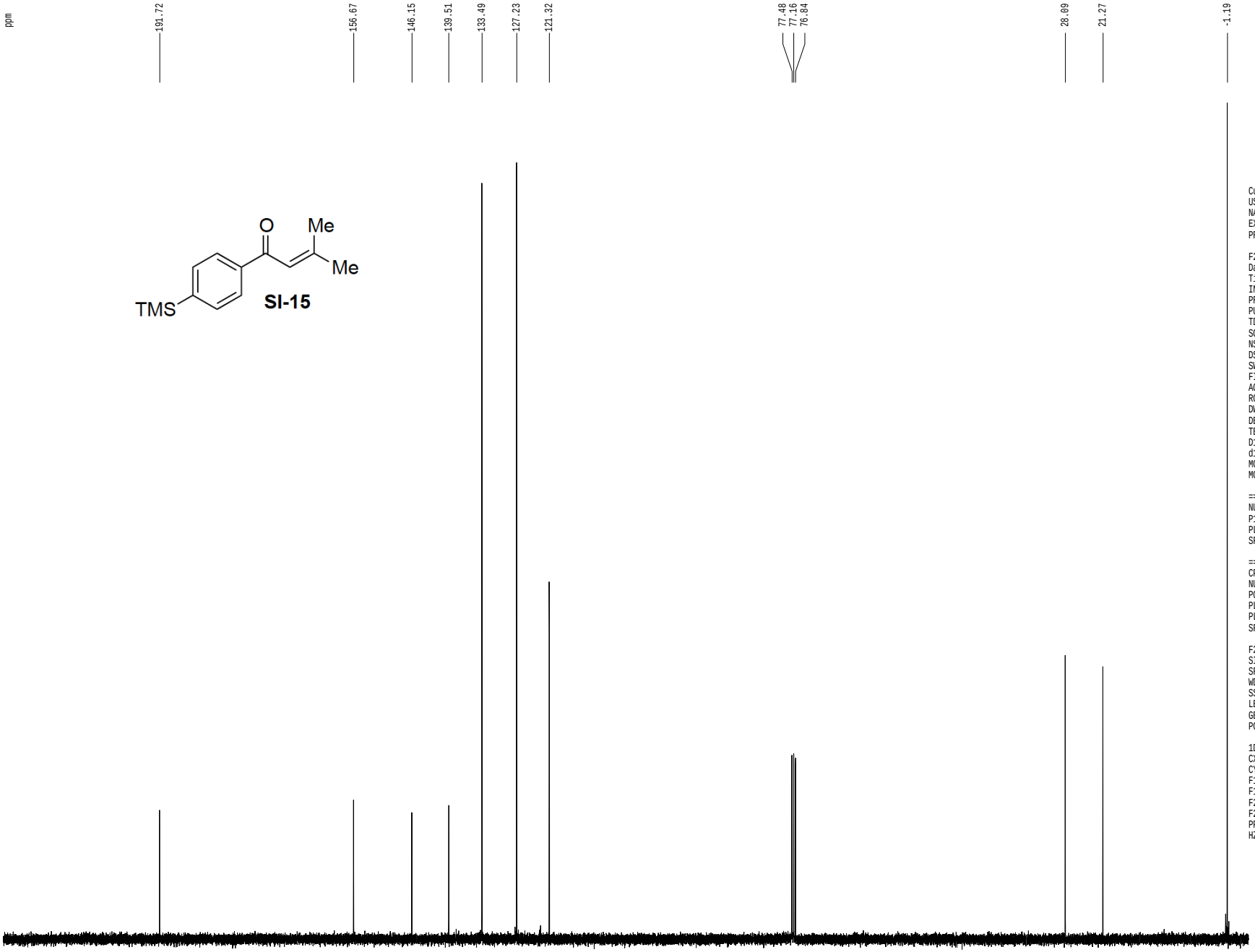
F2 - Acquisition Parameters
Date_           20131117
Time                4.49
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG             zg30
TD                 65536
SOLVENT           CDCl3T
NS                     8
DS                     2
SWH            6410.256 Hz
FIDRES         0.097813 Hz
AQ            5.1118579 sec
RG                  50.8
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          400.1300235 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P                9.000 ppm
F1               3601.17 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM           0.39474 ppm/cm
HZCM          157.94608 Hz/cm
```

13C spectrum with 1H decoupling




```
Current Data Parameters
USER             mkonev
NAME      MOKA072c-char
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20131117
Time               4.55
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  264
DS                    4
SWH           24154.590 Hz
FIDRES         0.368570 Hz
AQ            1.3566452 sec
RG               5792.6
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

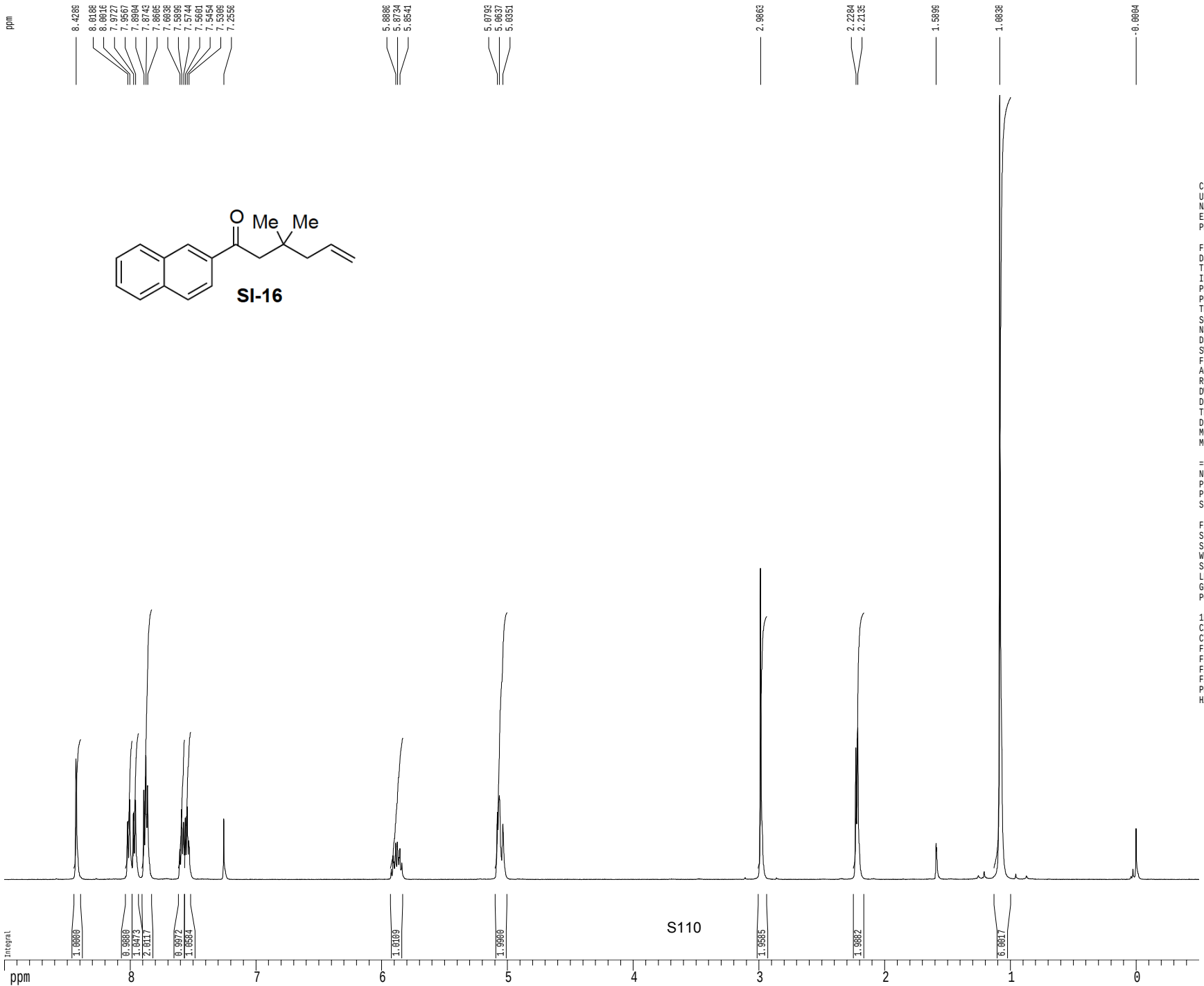
======== CHANNEL f1 =======
NUC1                13C
P1                 7.75 usec
PL1               -3.00 dB
SFO1        100.6237964 MHz

======== 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           100.6127628 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.80 cm
CY                15.50 cm
F1P             220.000 ppm
F1            22134.81 Hz
F2P              -5.000 ppm
F2             -503.07 Hz
PPMCM           9.86842 ppm/cm
HZCM         992.88916 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-27-1HNMR
EXPNO 1
PROCNO 1

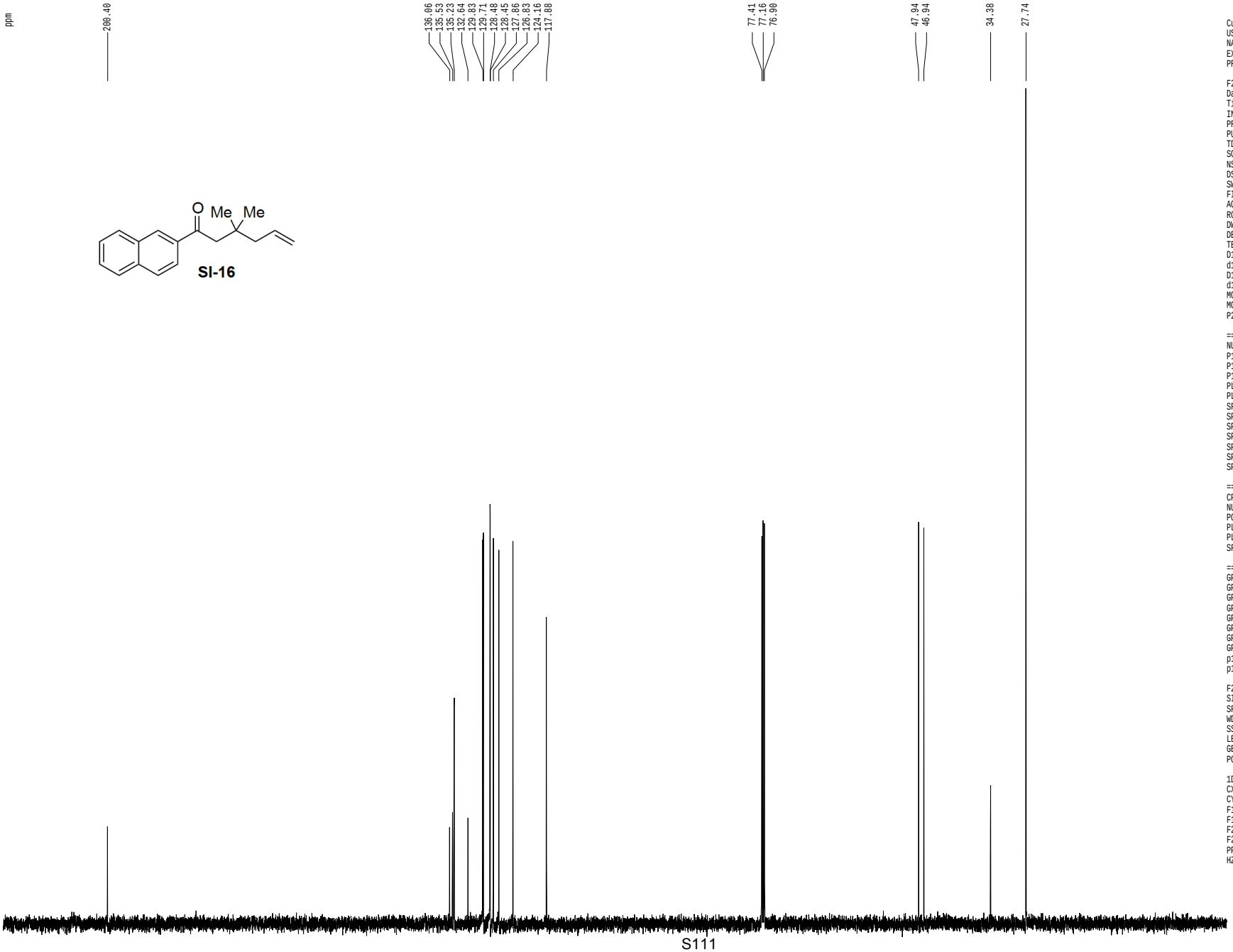
F2 - Acquisition Parameters
Date_ 20131021
Time 17.01
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200338 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-27-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131021
Time              17.04
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                 128
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ           1.0813940 sec
RG                6502
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               31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1               3.20 dB
SP2               3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1            0.00 Hz
SPOFF2            0.00 Hz

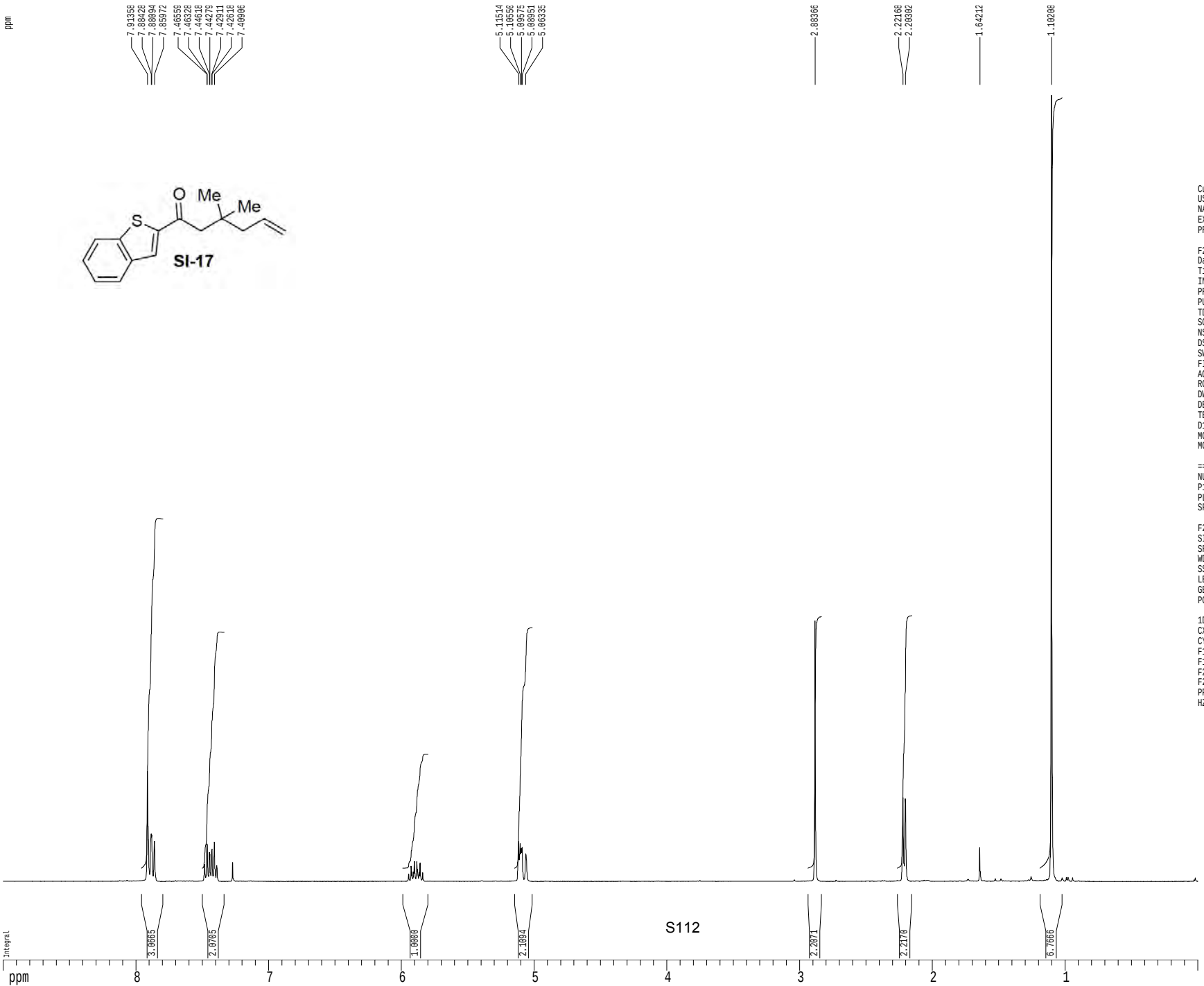
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2           100.00 usec
PL2               1.60 dB
PL12             24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804099 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            220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mkonev
NAME MOKA222_hchar
EXPNO 1
PROCNO 1

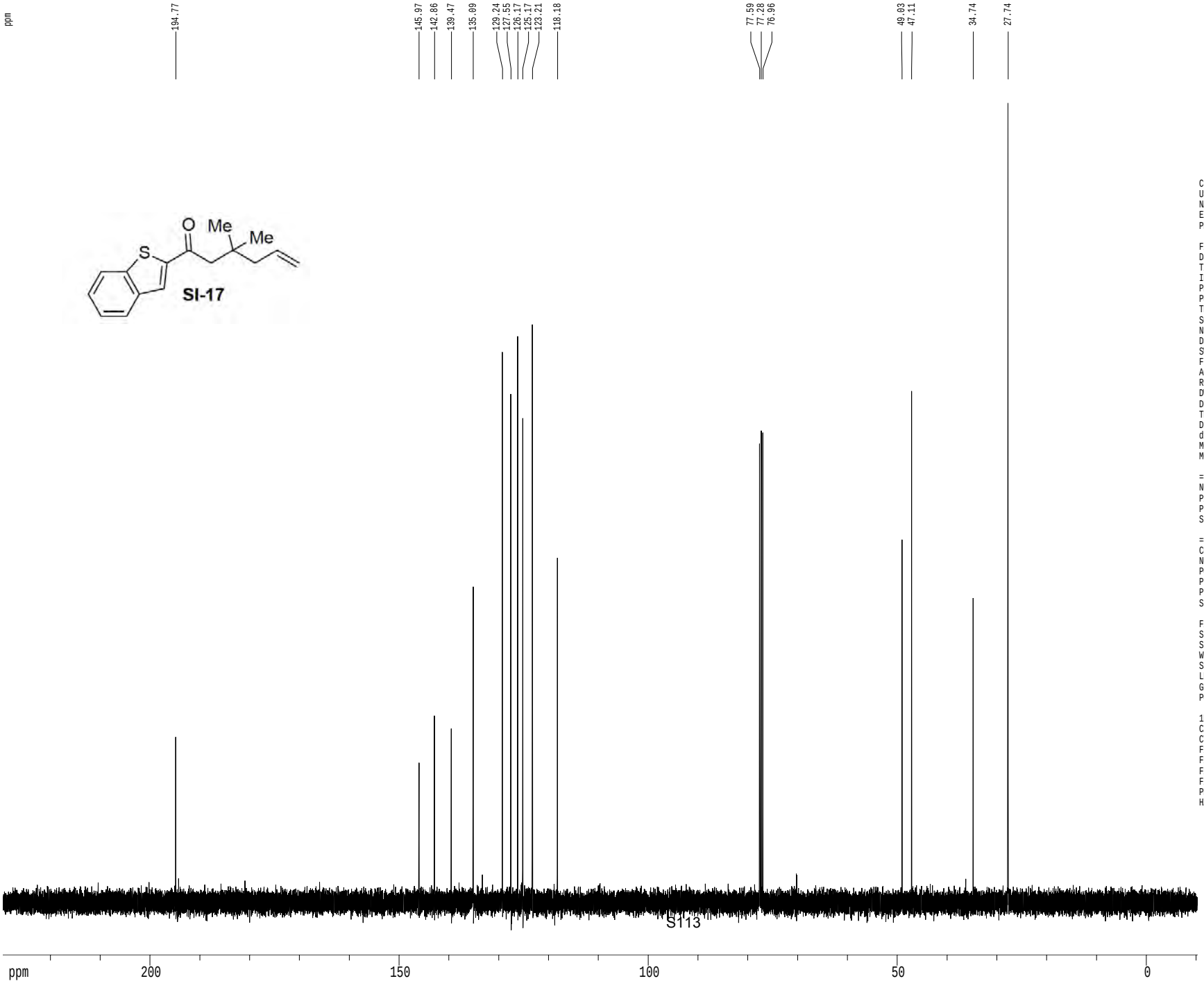
F2 - Acquisition Parameters
Date_ 20140314
Time 9.32
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 101.6
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== 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 no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm

13C spectrum with 1H decoupling




```
Current Data Parameters
USER              mkonev
NAME       MOKA222_cchar
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20140314
Time               9.35
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  222
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

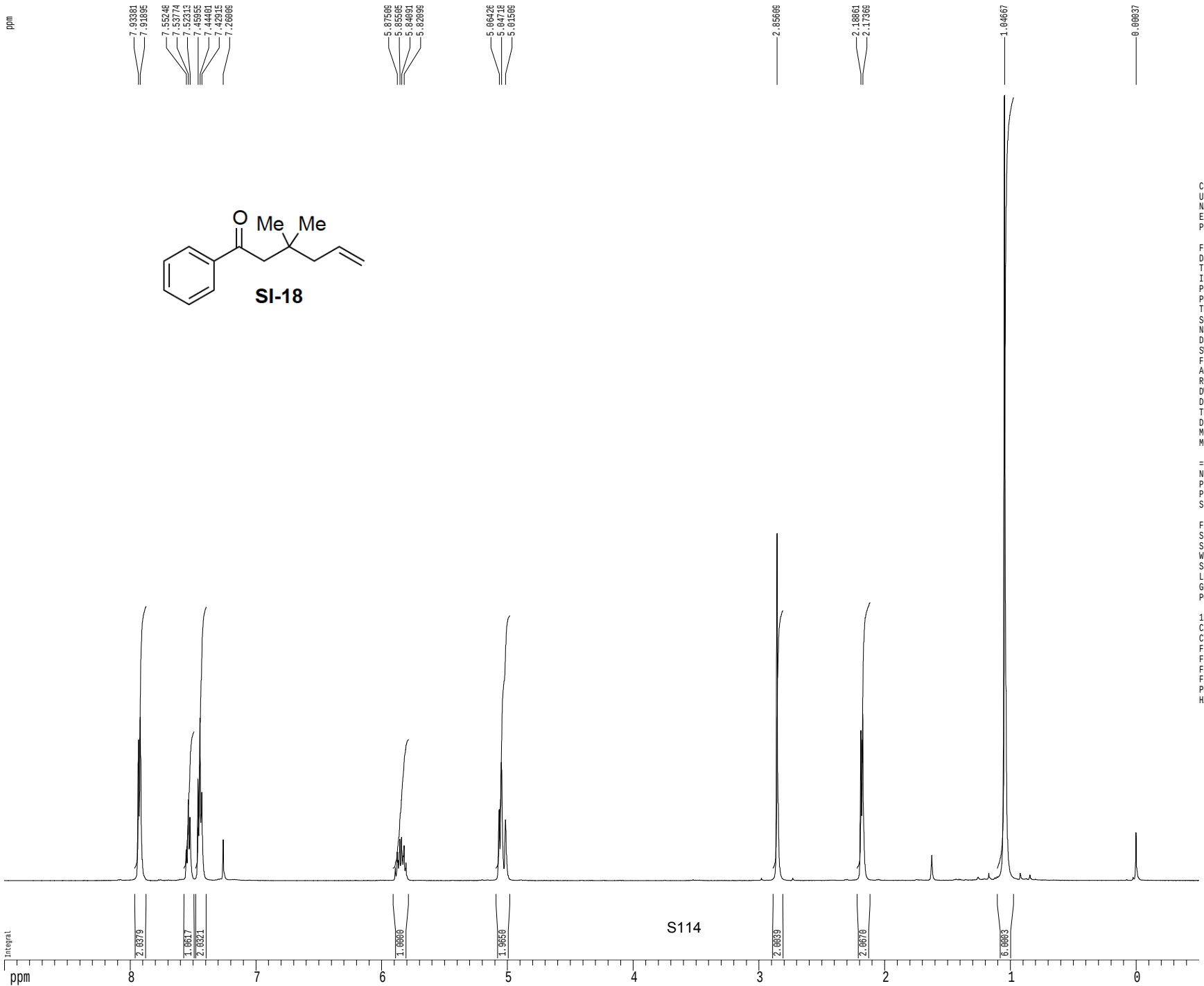
======== CHANNEL f1 =======
NUC1                13C
P1                 7.75 usec
PL1               -3.00 dB
SFO1        100.6237964 MHz

======== 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                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.50 cm
F1P             229.496 ppm
F1             23090.21 Hz
F2P             -10.579 ppm
F2            -1064.37 Hz
PPMCM          10.52959 ppm/cm
HZCM         1059.41138 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-42-1HNMR
EXPNO 1
PROCNO 1

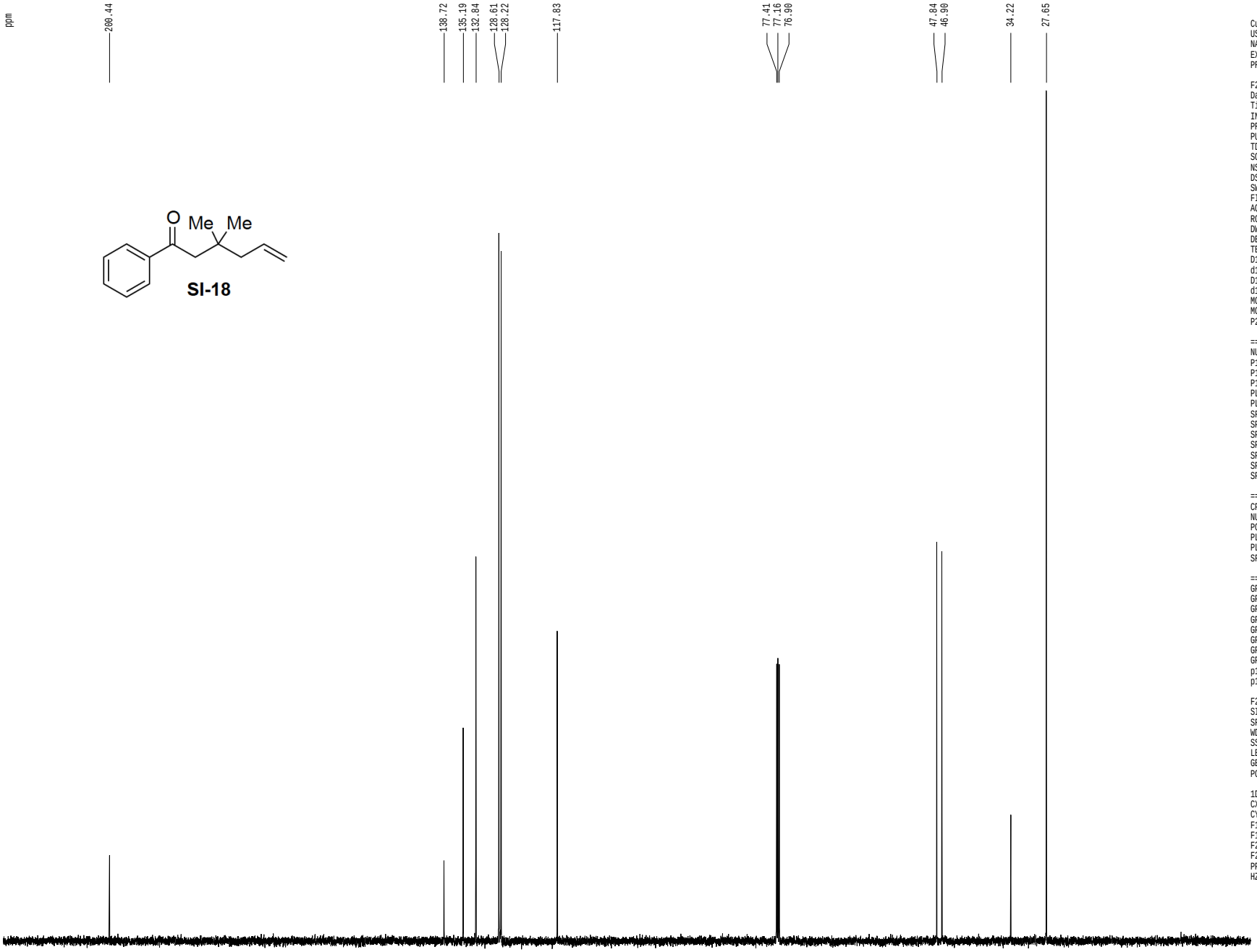
F2 - Acquisition Parameters
Date_ 20131021
Time 17.08
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200311 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-42-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131021
Time              17.10
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  103
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

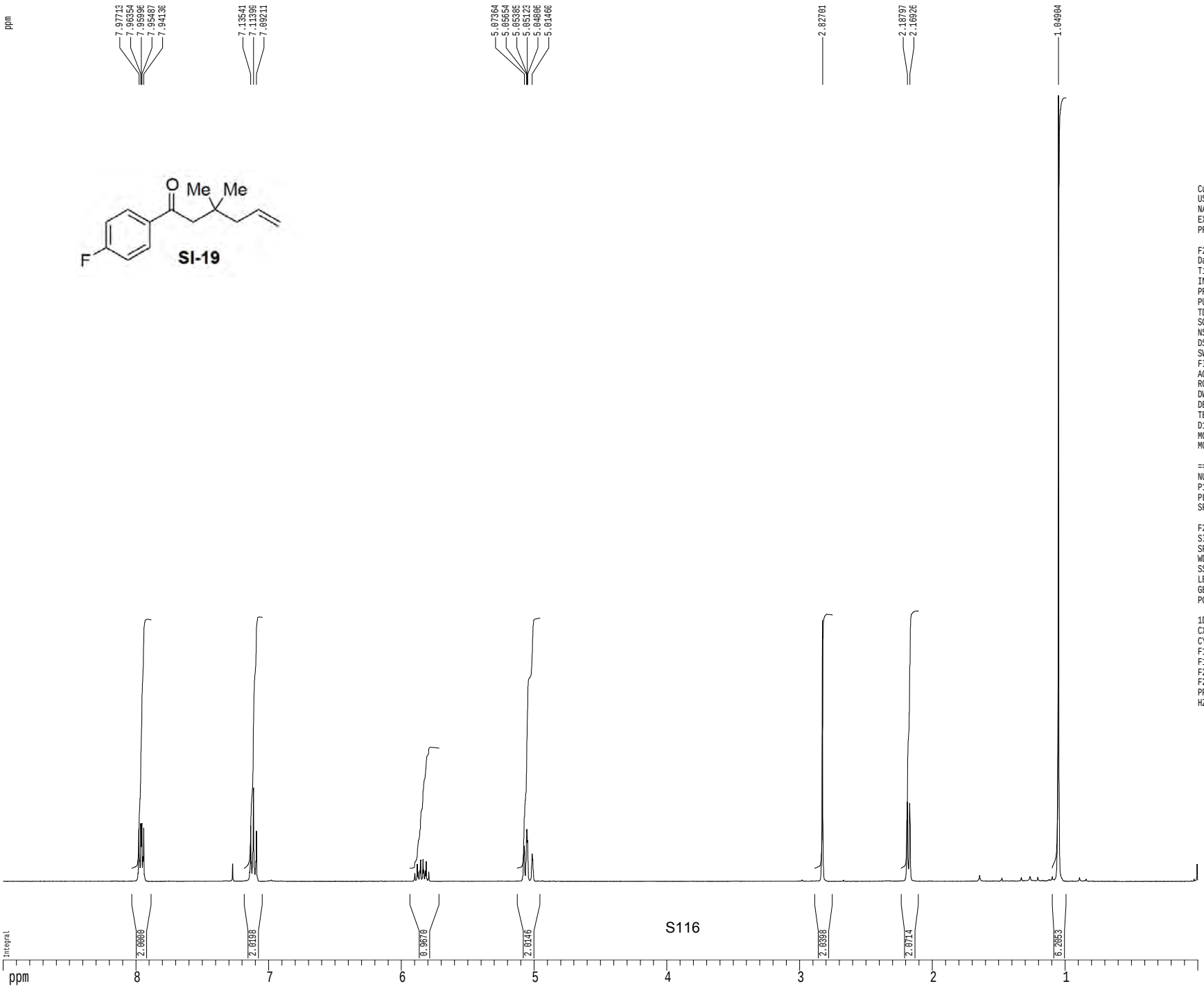
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804099 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             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mkonev
NAME MOKA075h-char
EXPNO 1
PROCNO 1

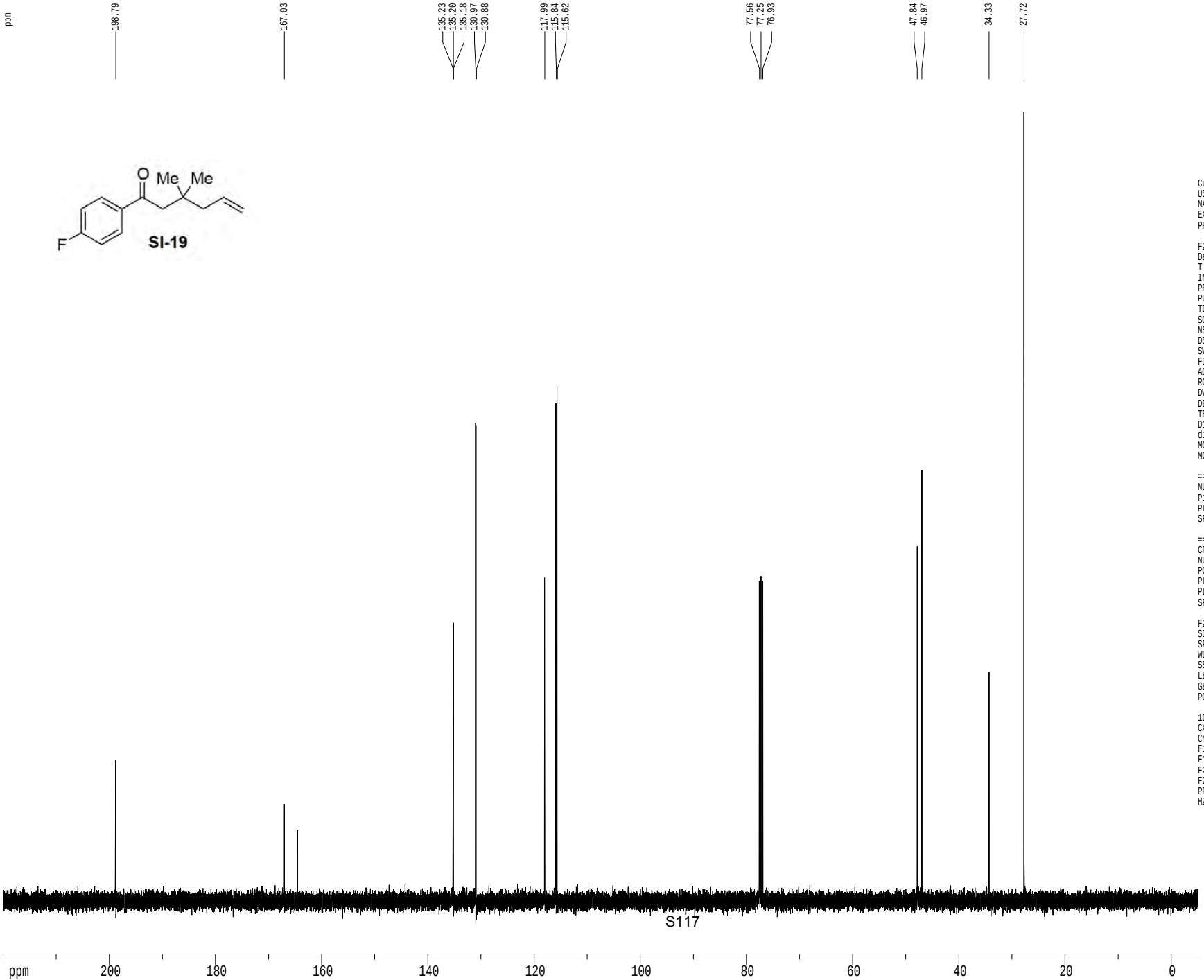
F2 - Acquisition Parameters
Date_ 20131118
Time 3.25
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 90.5
DW 78.000 usec
DE 4.50 usec
TE 298.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 400.1300175 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm

13C spectrum with 1H decoupling




```
Current Data Parameters
USER             mkonev
NAME      MOKA075c-char
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20131118
Time               3.28
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  256
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

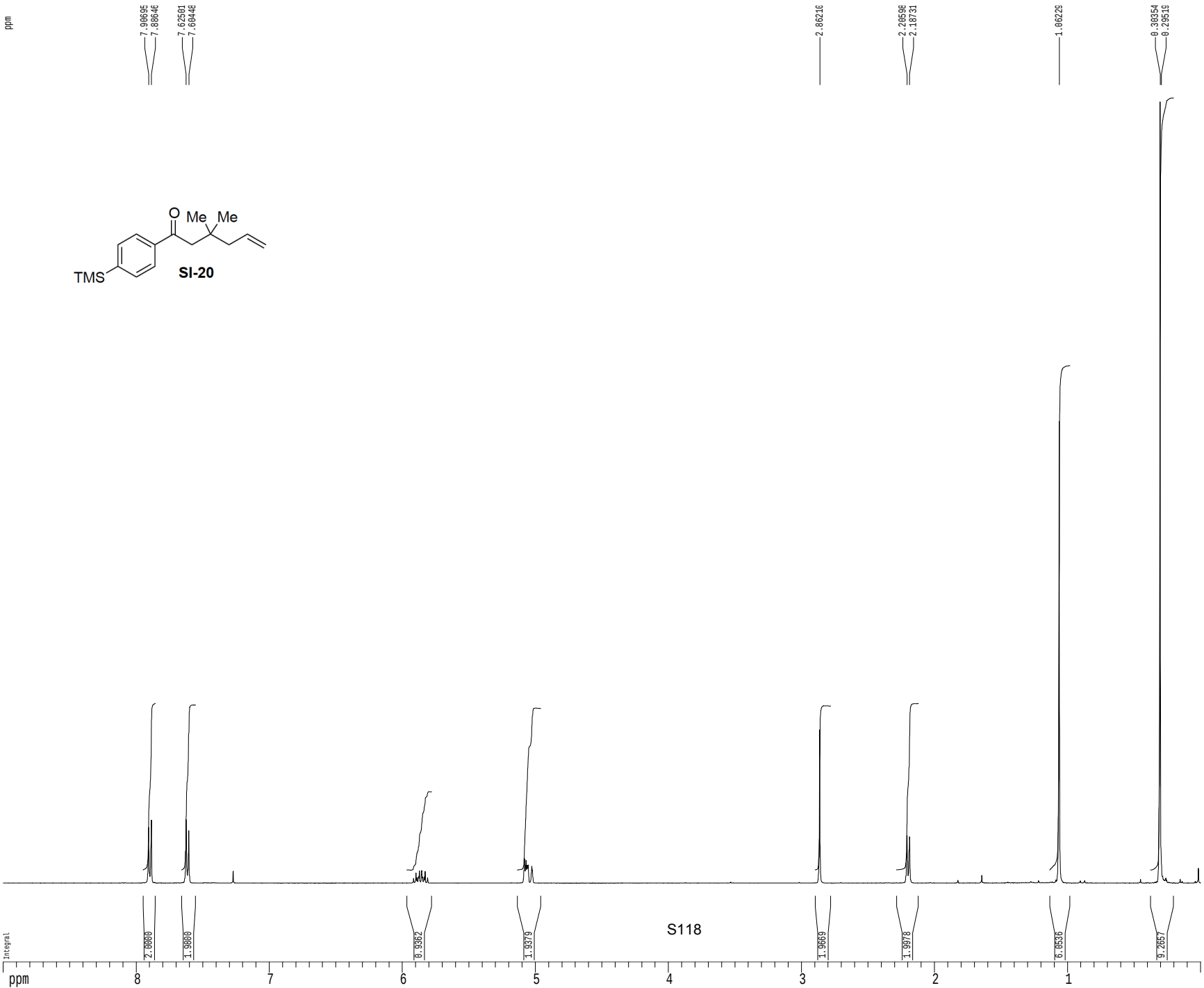
======== CHANNEL f1 =======
NUC1                13C
P1                 7.75 usec
PL1               -3.00 dB
SFO1        100.6237964 MHz

======== 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                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.50 cm
F1P             220.000 ppm
F1             22134.81 Hz
F2P              -5.000 ppm
F2             -503.06 Hz
PPMCM           9.86842 ppm/cm
HZCM          992.88898 Hz/cm
```

1H spectrum



S118

Current
USER
NAME
EXPNO
PROCNO

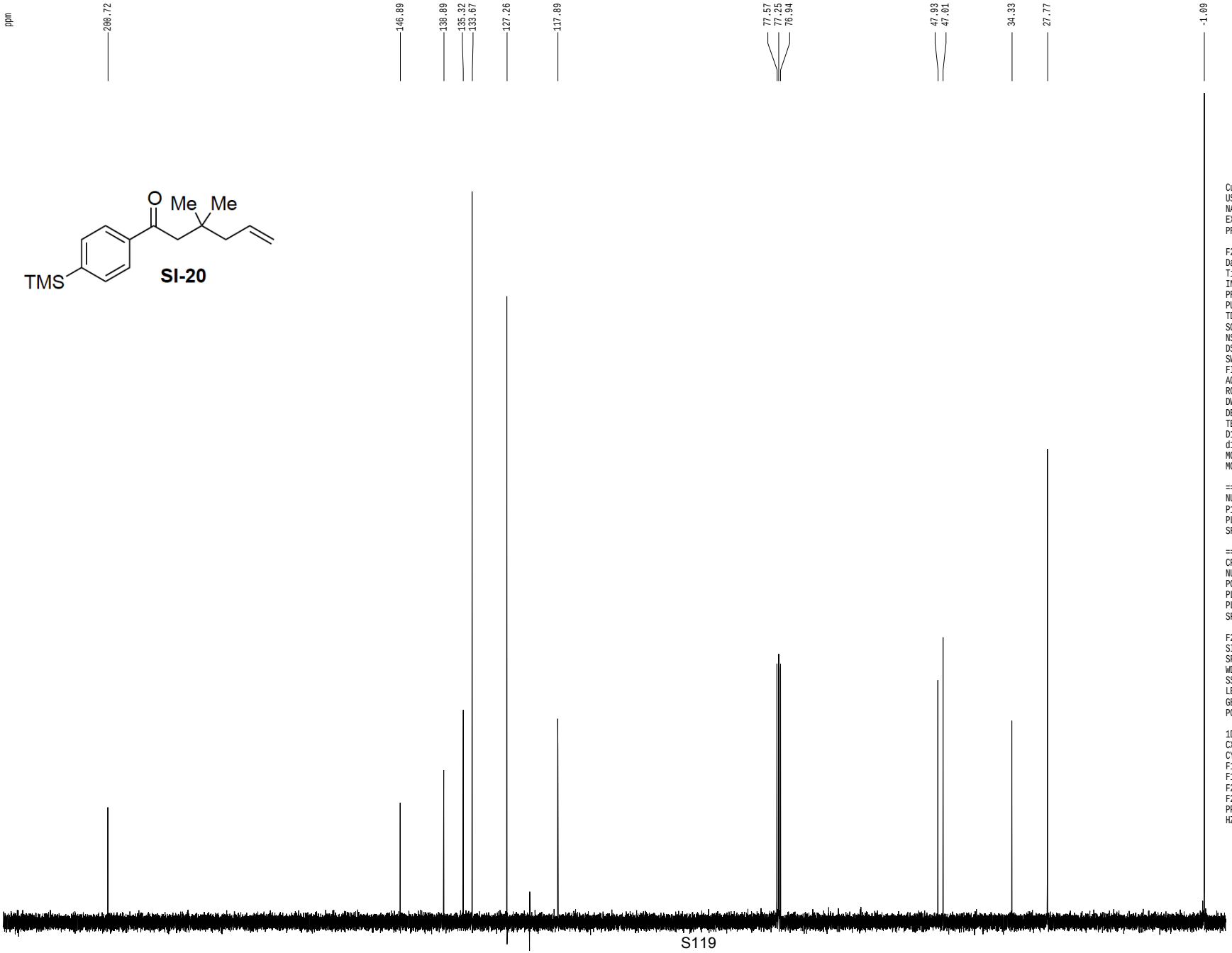
F2 - Acq
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
MCREST
MCWRK

========
NUC1
P1
PL1
SFO1

F2 - Pro
SI
SF
WDW
SSB
LB
GB
PC

1D NMR p
CX
CY
F1P
F1
F2P
F2
PPMCM
HZCM

13C spectrum with 1H decoupling




```
Current Data Parameters
USER             mkonev
NAME      MOKA074c-char
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20131118
Time               0.31
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  216
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

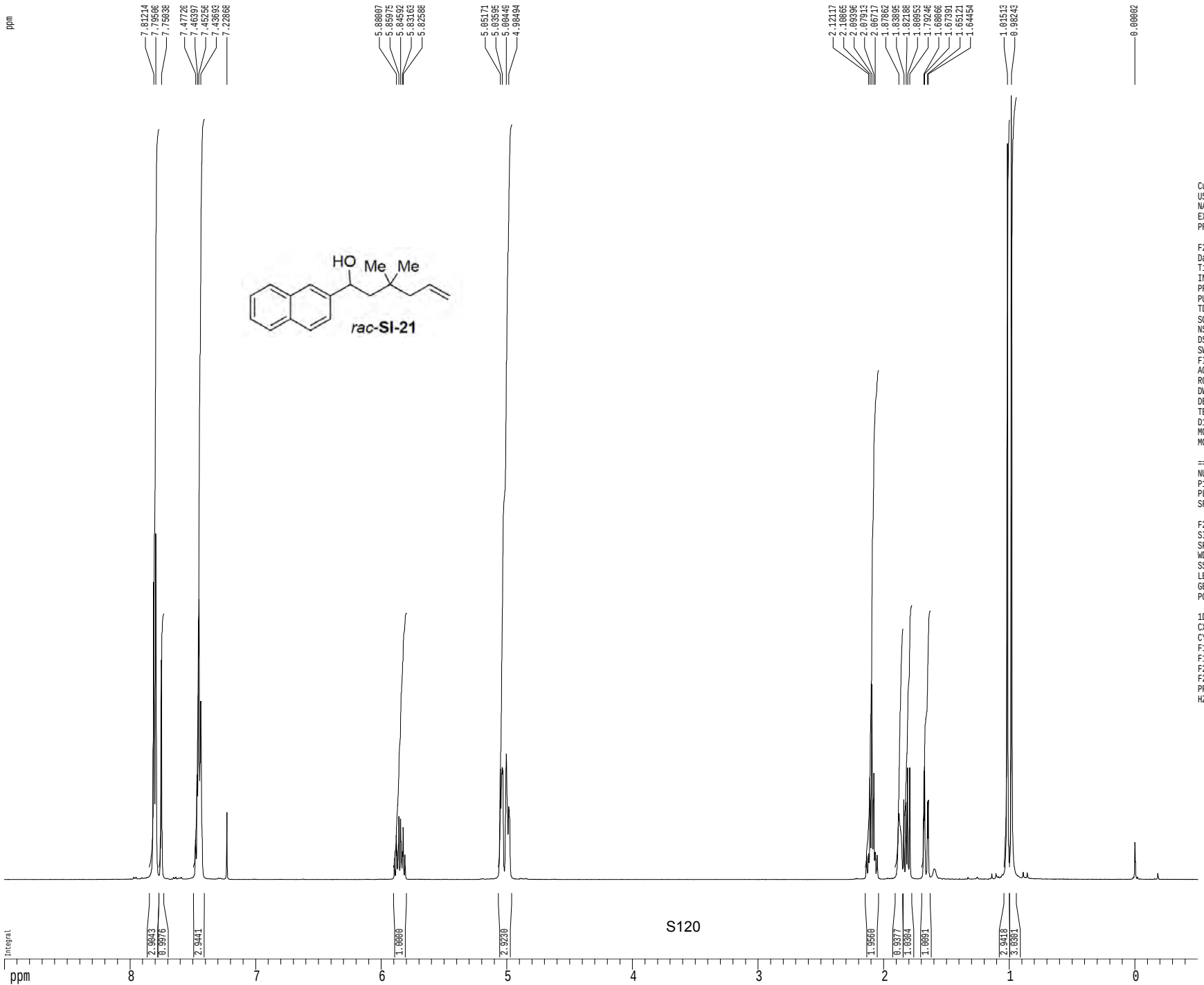
======== CHANNEL f1 ========
NUC1                13C
P1                 7.75 usec
PL1               -3.00 dB
SFO1        100.6237964 MHz

======== 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                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.50 cm
F1P             220.000 ppm
F1             22134.81 Hz
F2P              -5.000 ppm
F2             -503.06 Hz
PPMCM           9.86842 ppm/cm
HZCM          992.88898 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-29-1HNMR
EXPNO 1
PROCNO 1

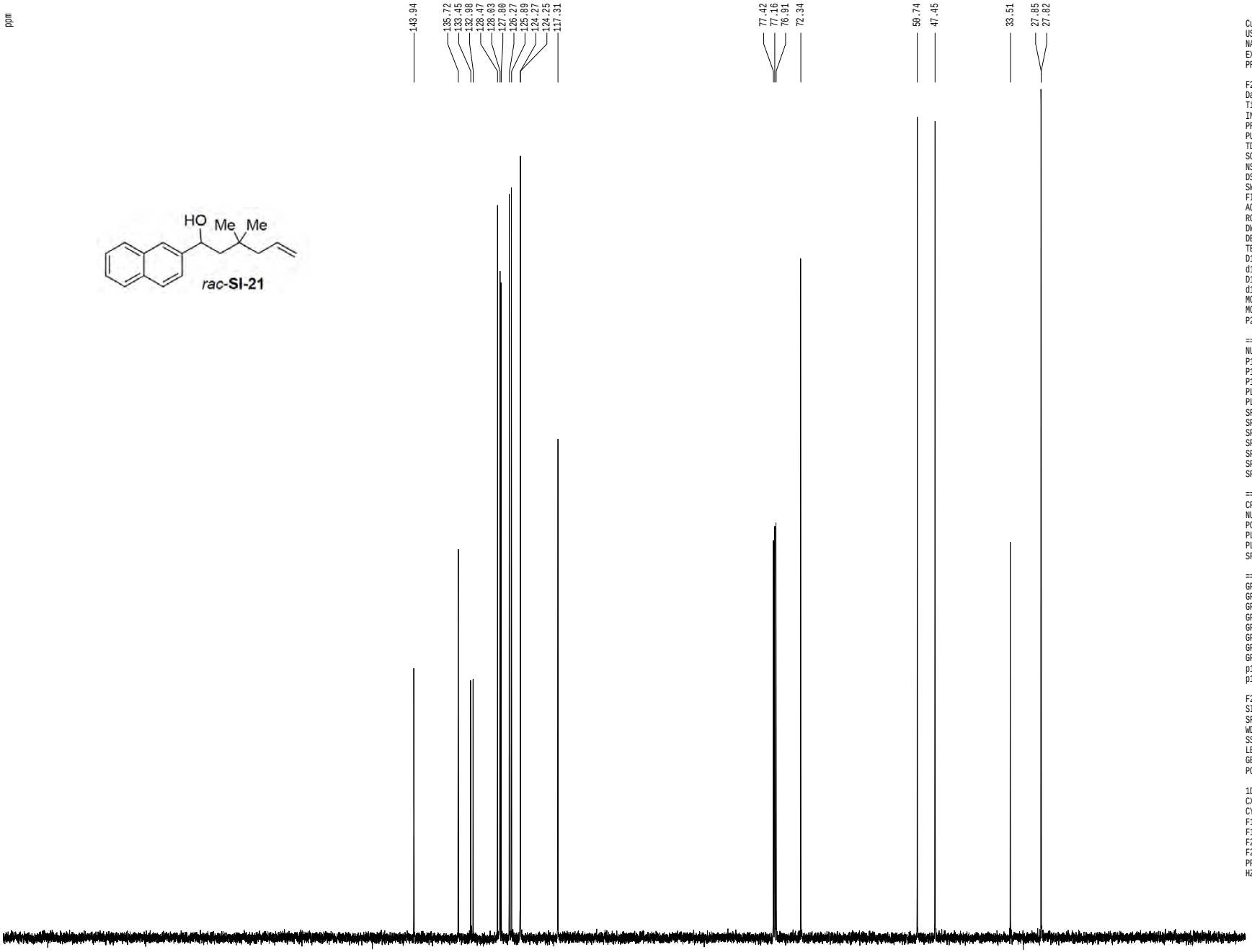
F2 - Acquisition Parameters
Date_ 20130915
Time 17.24
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200465 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-29-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130915
Time              17.27
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  111
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

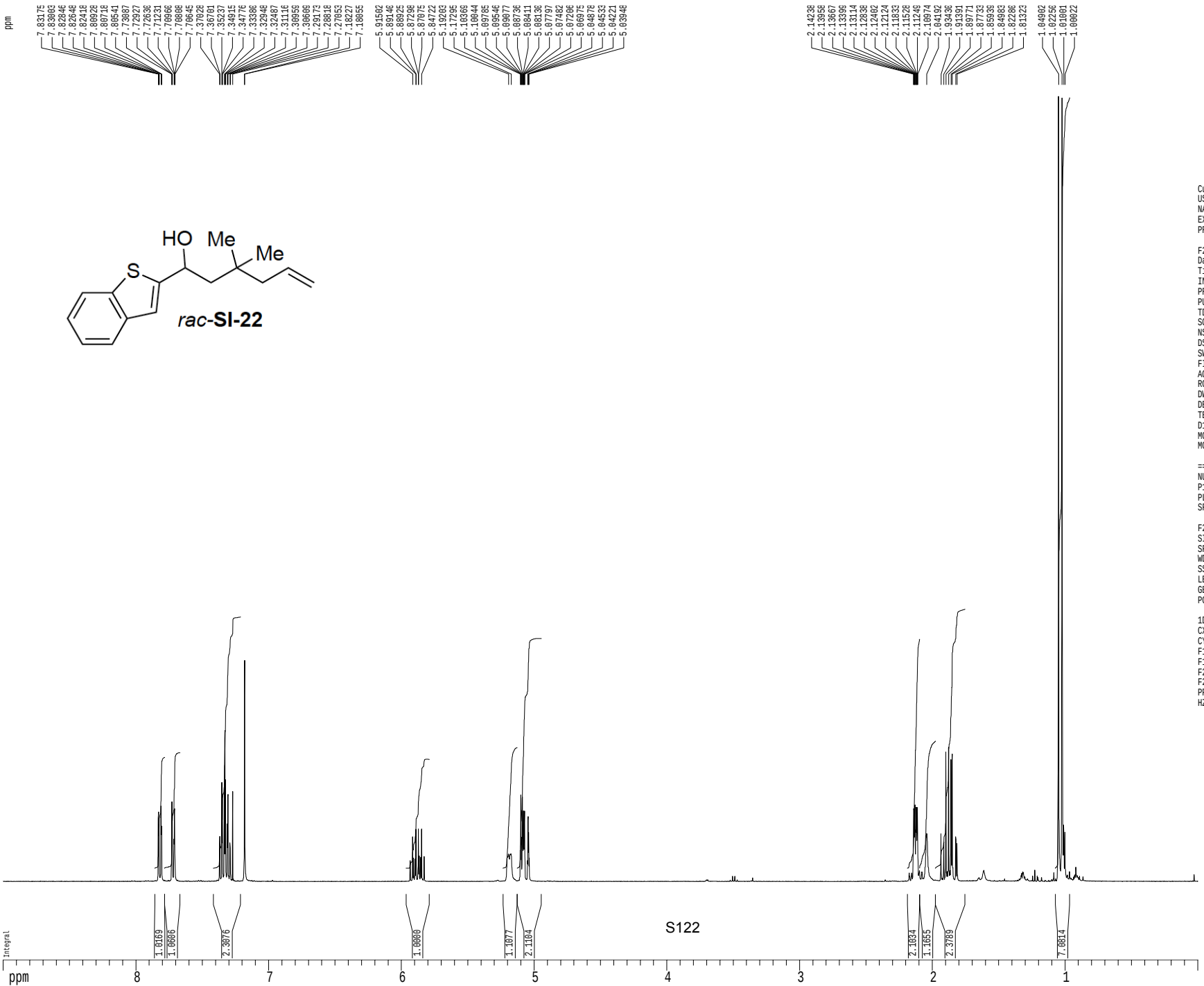
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804136 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             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mkonev
NAME MOKA223_hchar
EXPNO 1
PROCNO 1

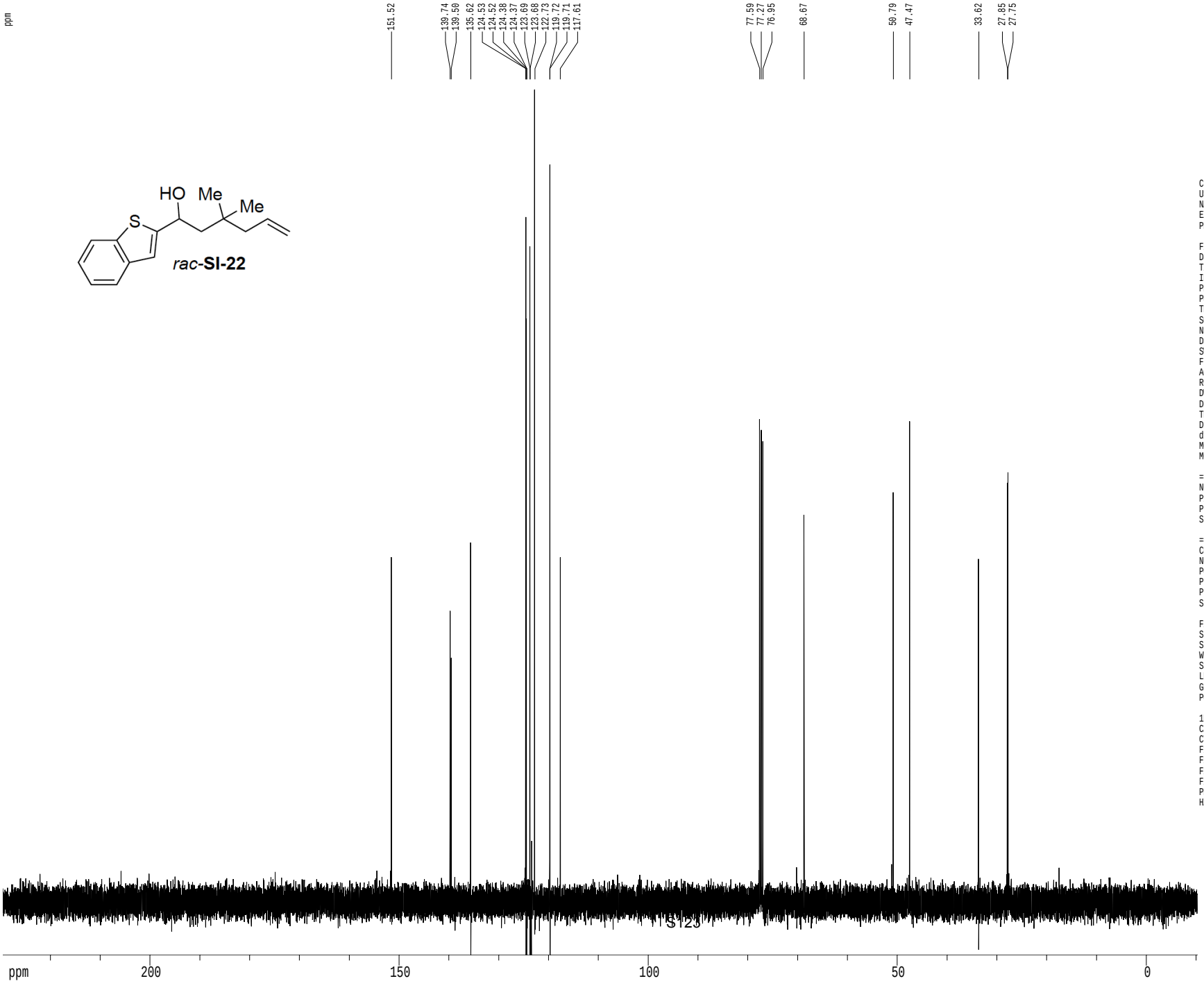
F2 - Acquisition Parameters
Date_ 20140317
Time 8.41
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 114
DW 78.000 usec
DE 4.50 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== 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 no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm

13C spectrum with 1H decoupling




```
Current Data Parameters
USER              mkonev
NAME       MOKA223_cchar
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20140317
Time                8.44
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG           zgdc30
TD                65536
SOLVENT           CDCl3
NS                  224
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

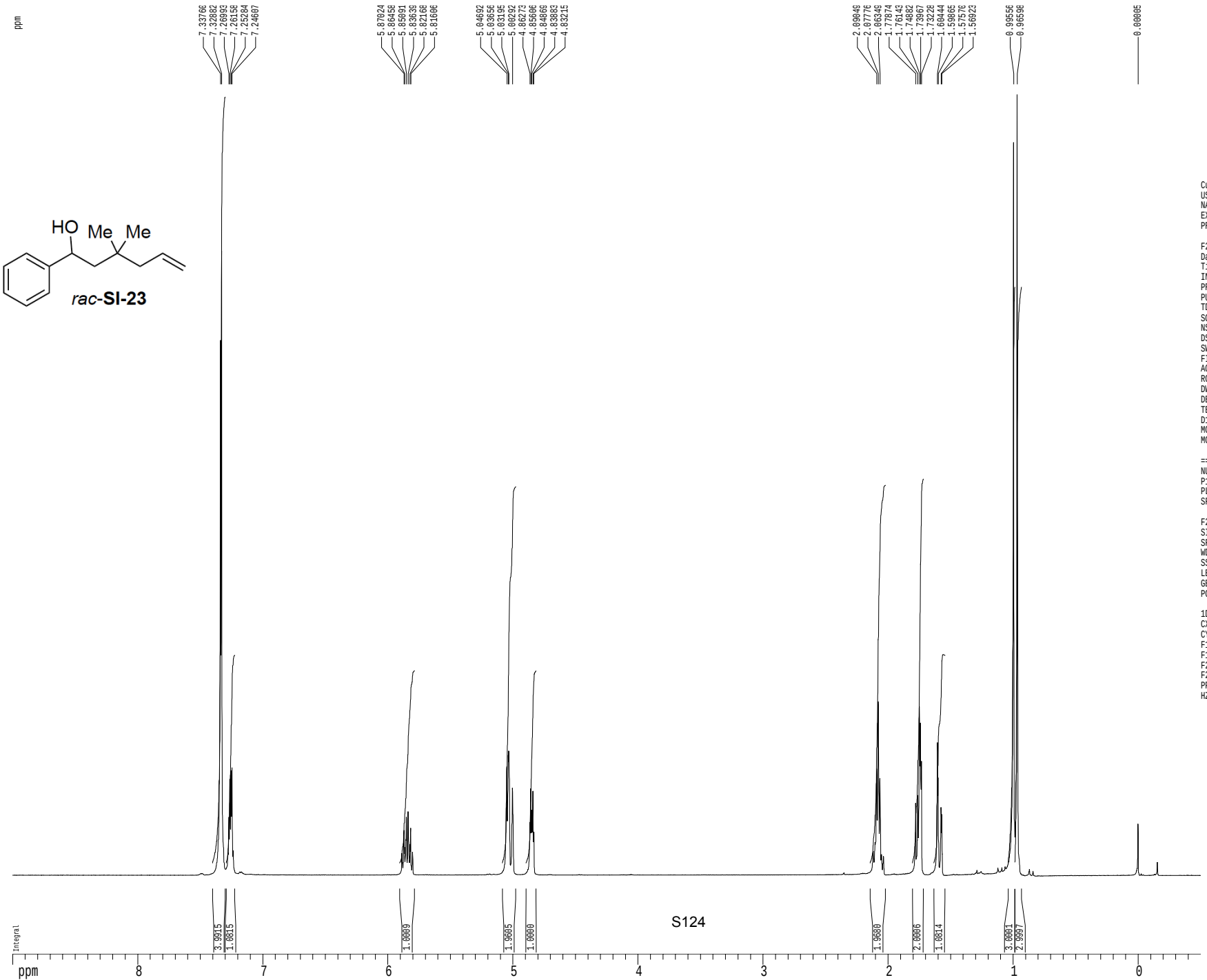
======== CHANNEL f1 ========
NUC1                13C
P1                 7.75 usec
PL1               -3.00 dB
SFO1        100.6237964 MHz

======== 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                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.50 cm
F1P               229.496 ppm
F1              23090.21 Hz
F2P              -10.579 ppm
F2              -1064.37 Hz
PPMCM            10.52959 ppm/cm
HZCM          1059.41138 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-41-1HNMR
EXPNO 1
PROCNO 1

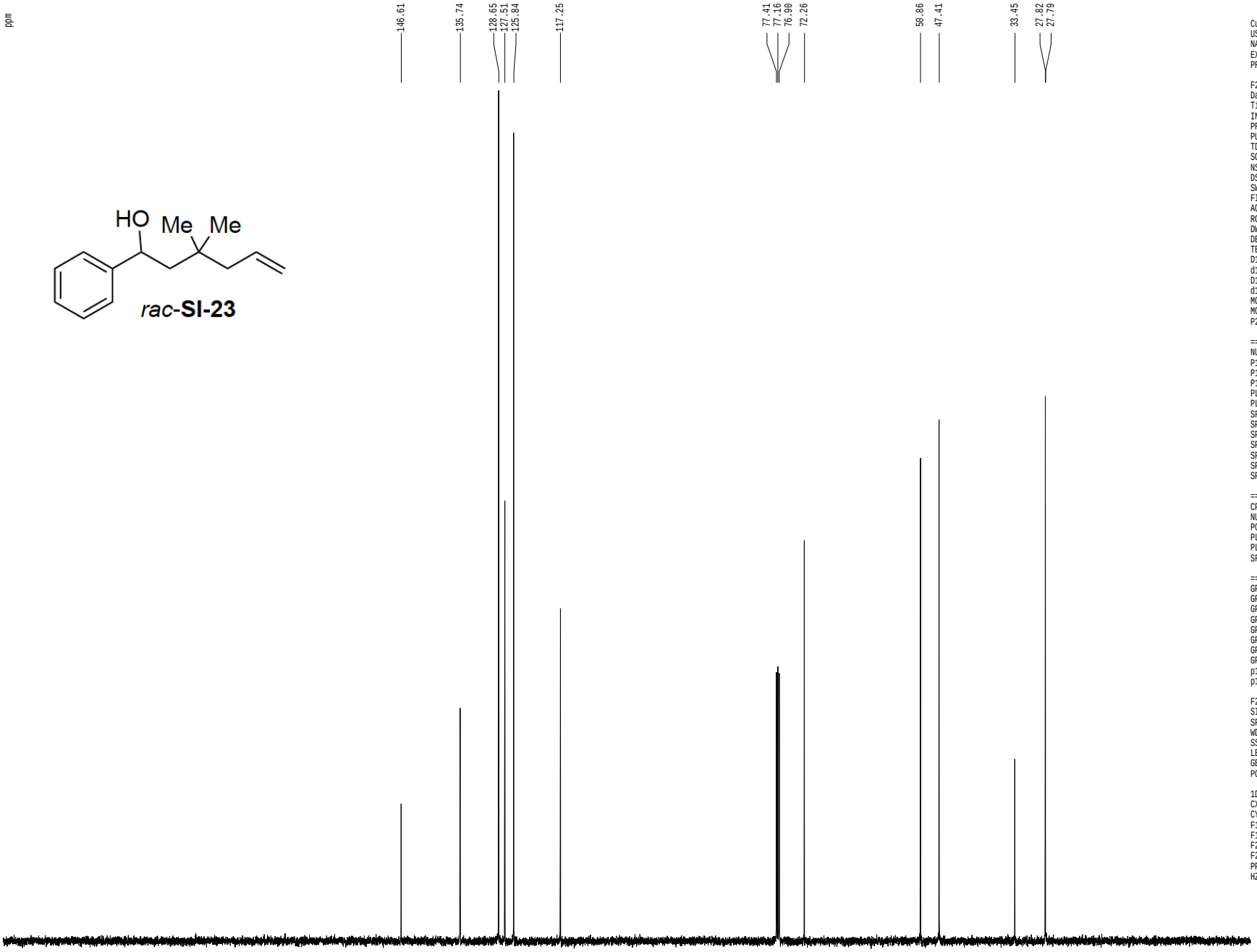
F2 - Acquisition Parameters
Date_ 20130915
Time 17.39
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200373 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-41-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130915
Time              17.41
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  102
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG                 4096
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

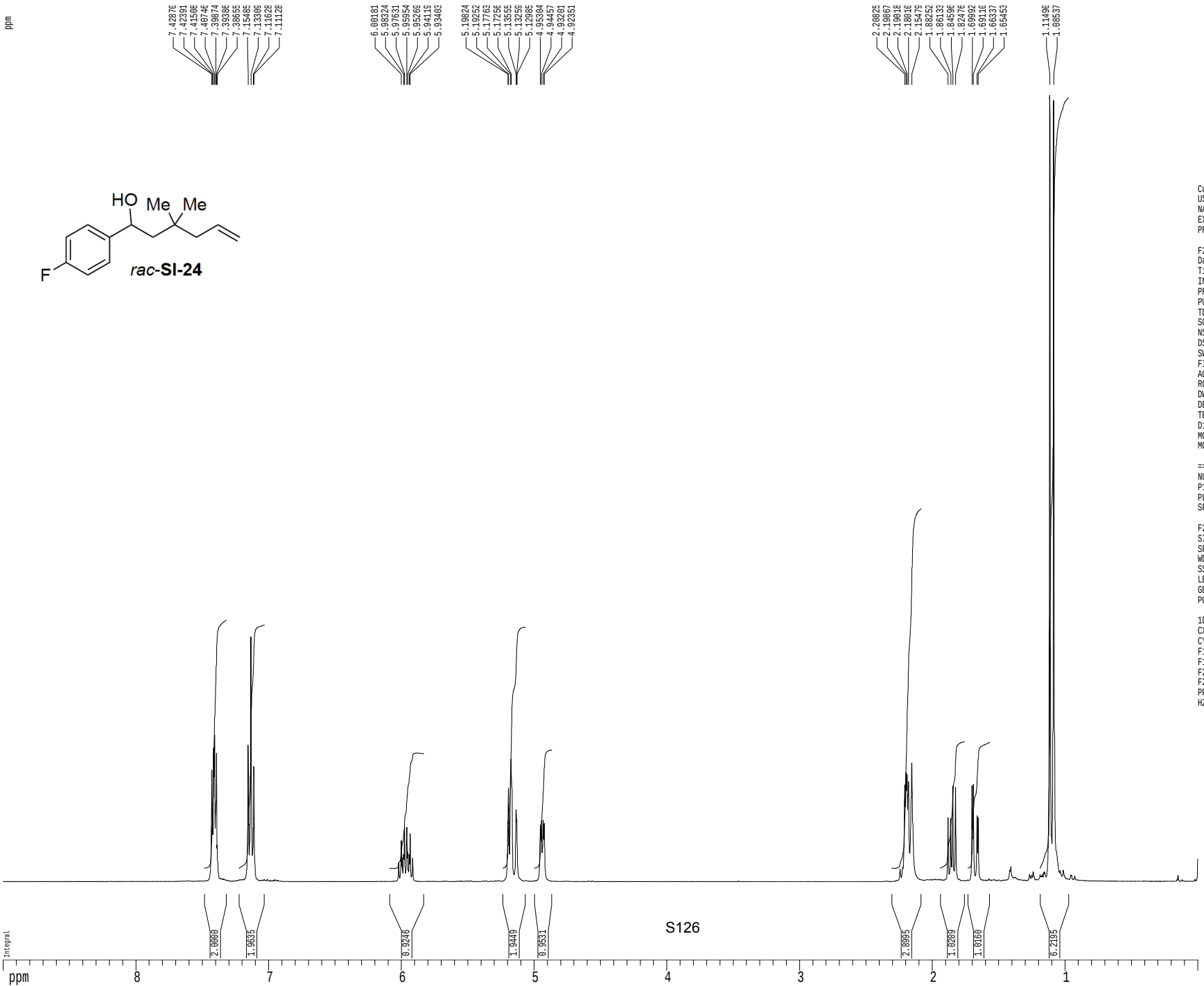
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804113 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             220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mkonev
NAME MOKA078h-char
EXPNO 1
PROCNO 1

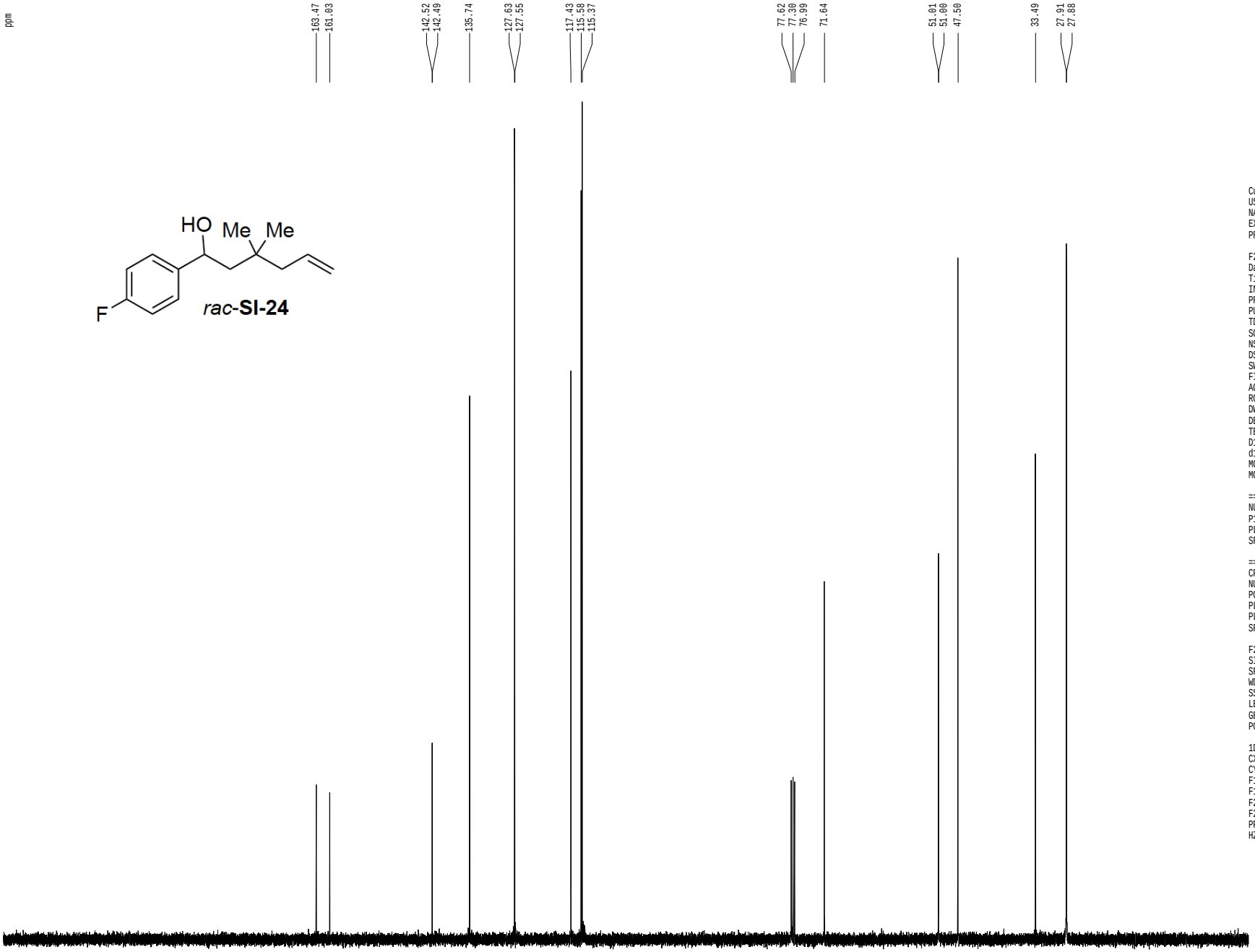
F2 - Acquisition Parameters
Date_ 20131118
Time 1.33
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 65536
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 32
DW 78.000 usec
DE 4.50 usec
TE 298.1 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 400.1299713 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94604 Hz/cm

13C spectrum with 1H decoupling




```
Current Data Parameters
USER            mkonev
NAME      MOKA078c-char
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_          20131118
Time               1.38
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  187
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

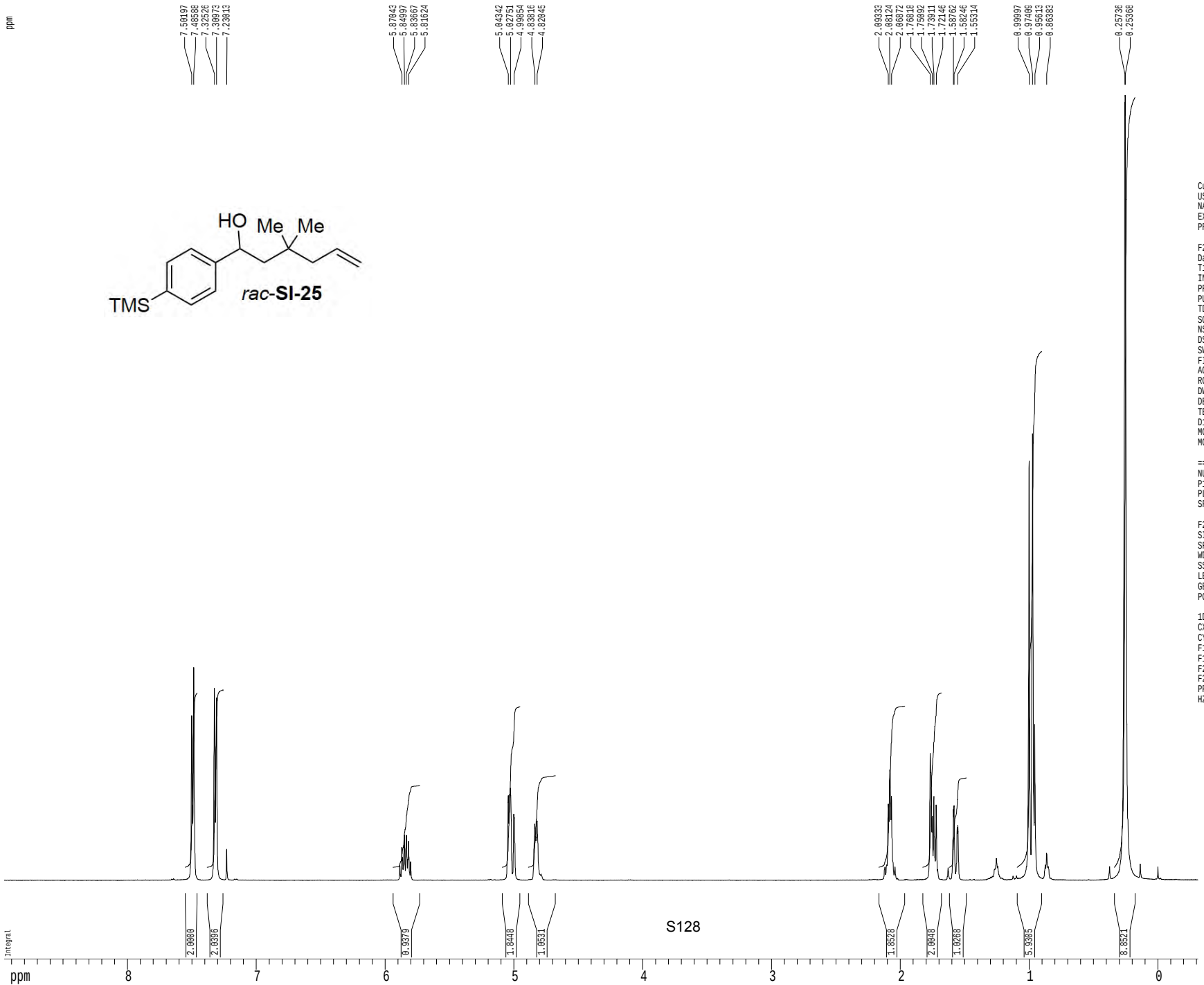
======== CHANNEL f1 =======
NUC1                13C
P1                 7.75 usec
PL1               -3.00 dB
SFO1        100.6237964 MHz

======== 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                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.50 cm
F1P             220.000 ppm
F1             22134.81 Hz
F2P              -5.000 ppm
F2             -503.06 Hz
PPMCM           9.86842 ppm/cm
HZCM          992.88898 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER            mkonev
NAME      MOKA077h-char
EXPNO               1
PROCNO              1

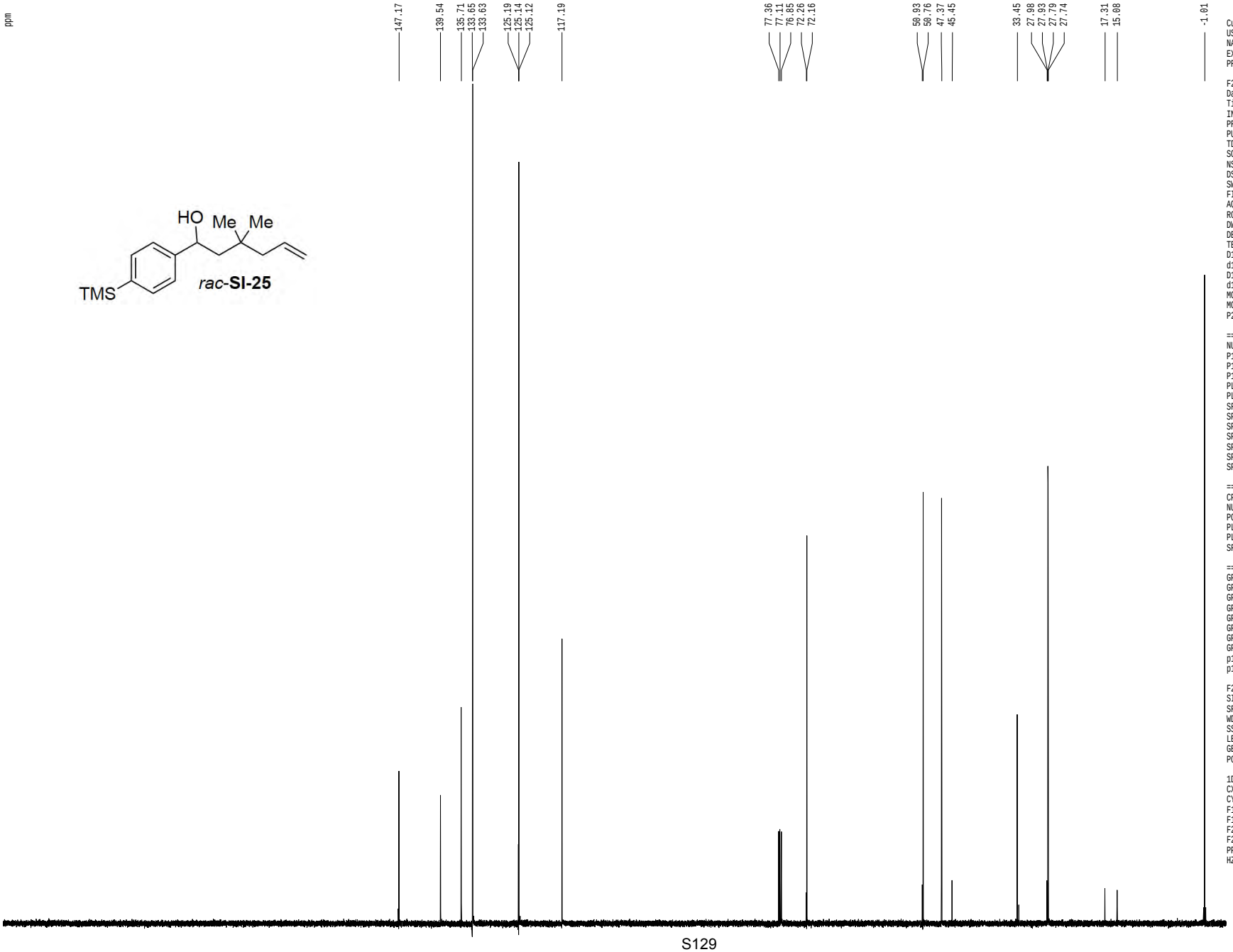
F2 - Acquisition Parameters
Date_          20131120
Time               1.16
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3T
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                  3.2
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          500.2200459 MHz
WDW                  no
SSB                   0
LB                0.00 Hz
GB                    0
PC                4.00

1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P               8.955 ppm
F1              4479.51 Hz
F2P              -0.309 ppm
F2             -154.62 Hz
PPMCM          0.40632 ppm/cm
HZCM         203.25117 Hz/cm
```

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




```
Current Data Parameters
USER            mkonev
NAME      MOKA077c-char
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_          20131120
Time               1.19
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                 129
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG               5160.6
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

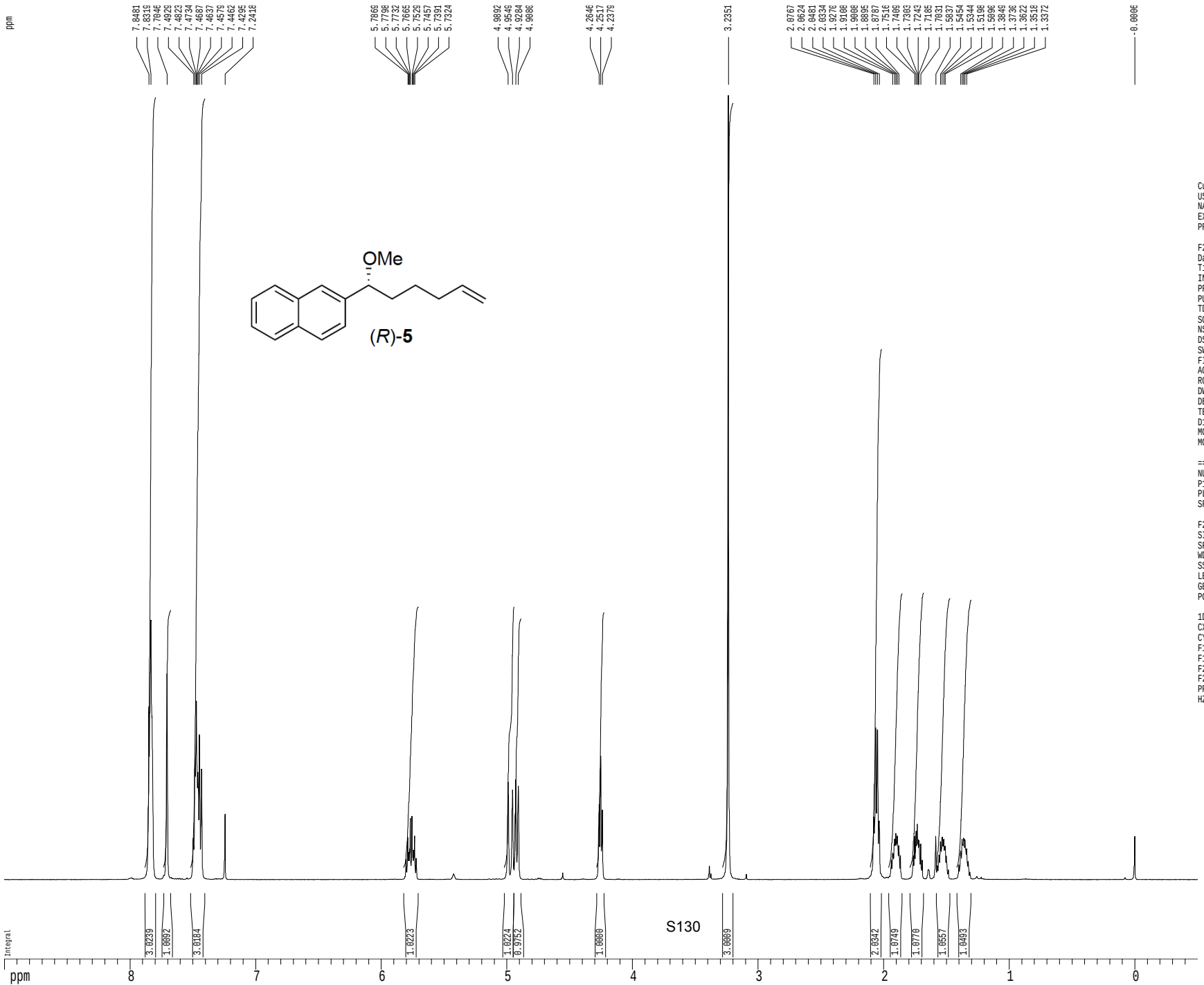
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER            mharri
NAME     MRH-IV-246-1HNMR
EXPNO                1
PROCNO               1

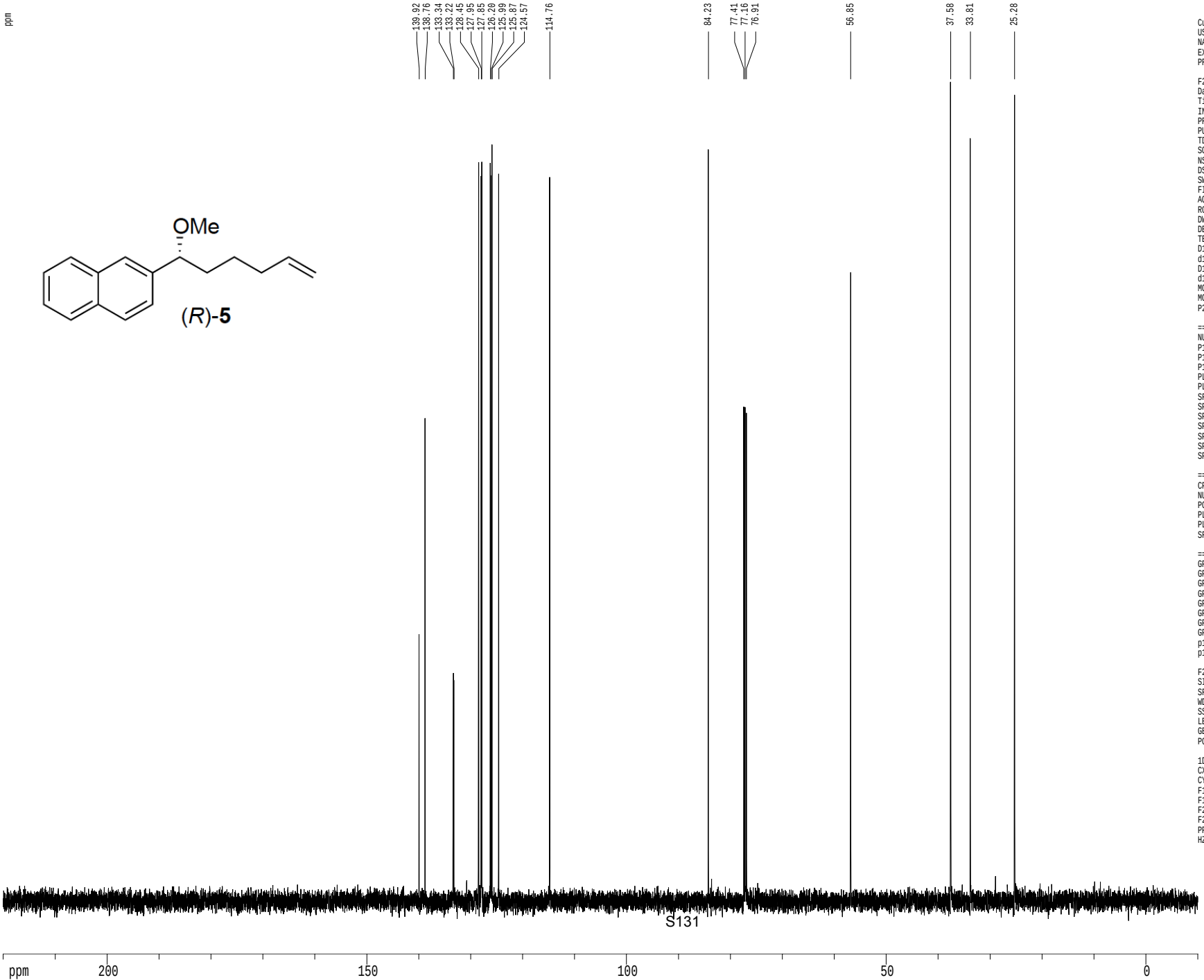
F2 - Acquisition Parameters
Date_          20131209
Time              18.56
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    1
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                  6.3
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          500.2200393 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-IV-246-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131209
Time              18.58
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                   76
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG               5160.6
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

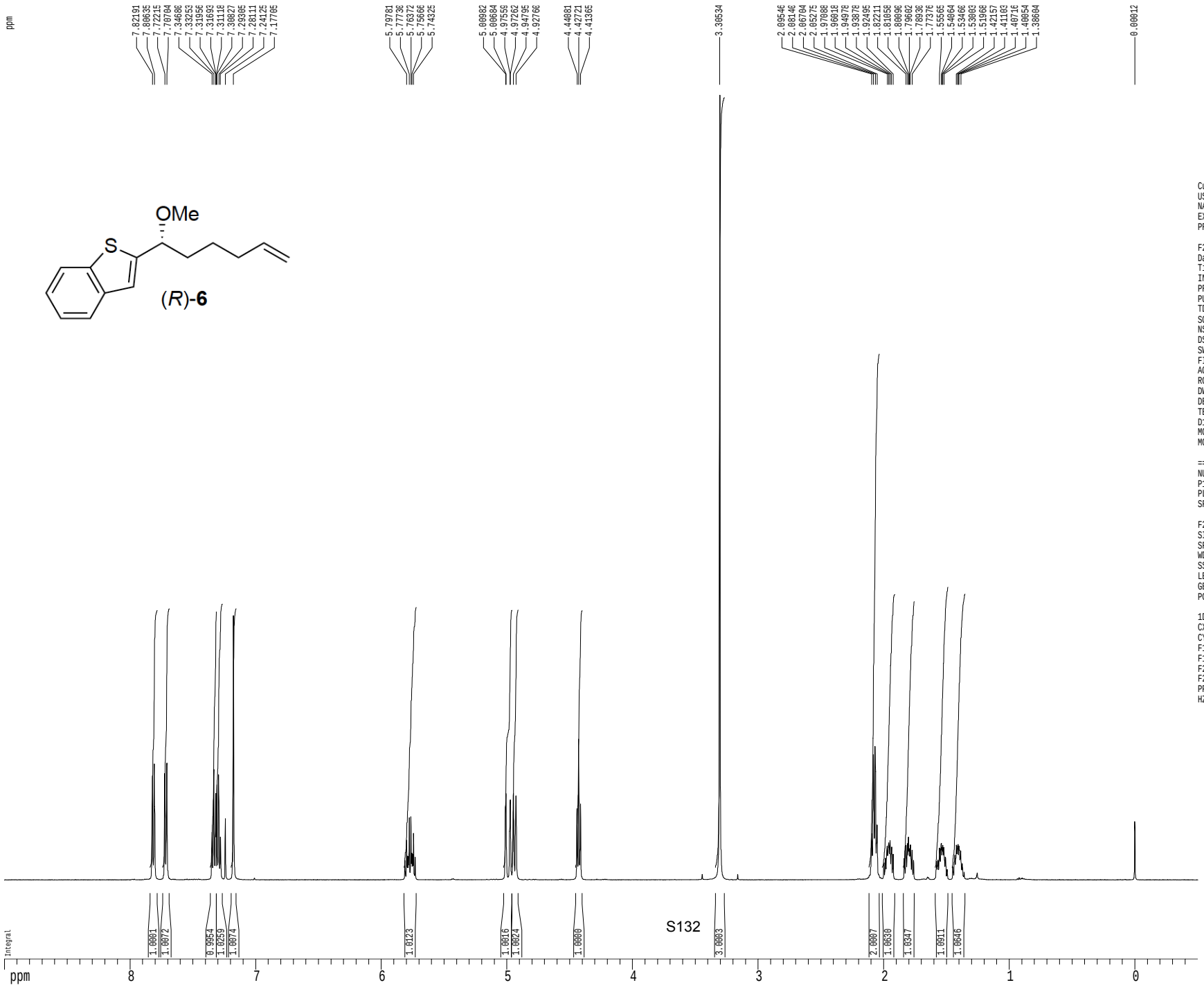
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804108 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             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-248-1HNMR
EXPNO 2
PROCNO 1

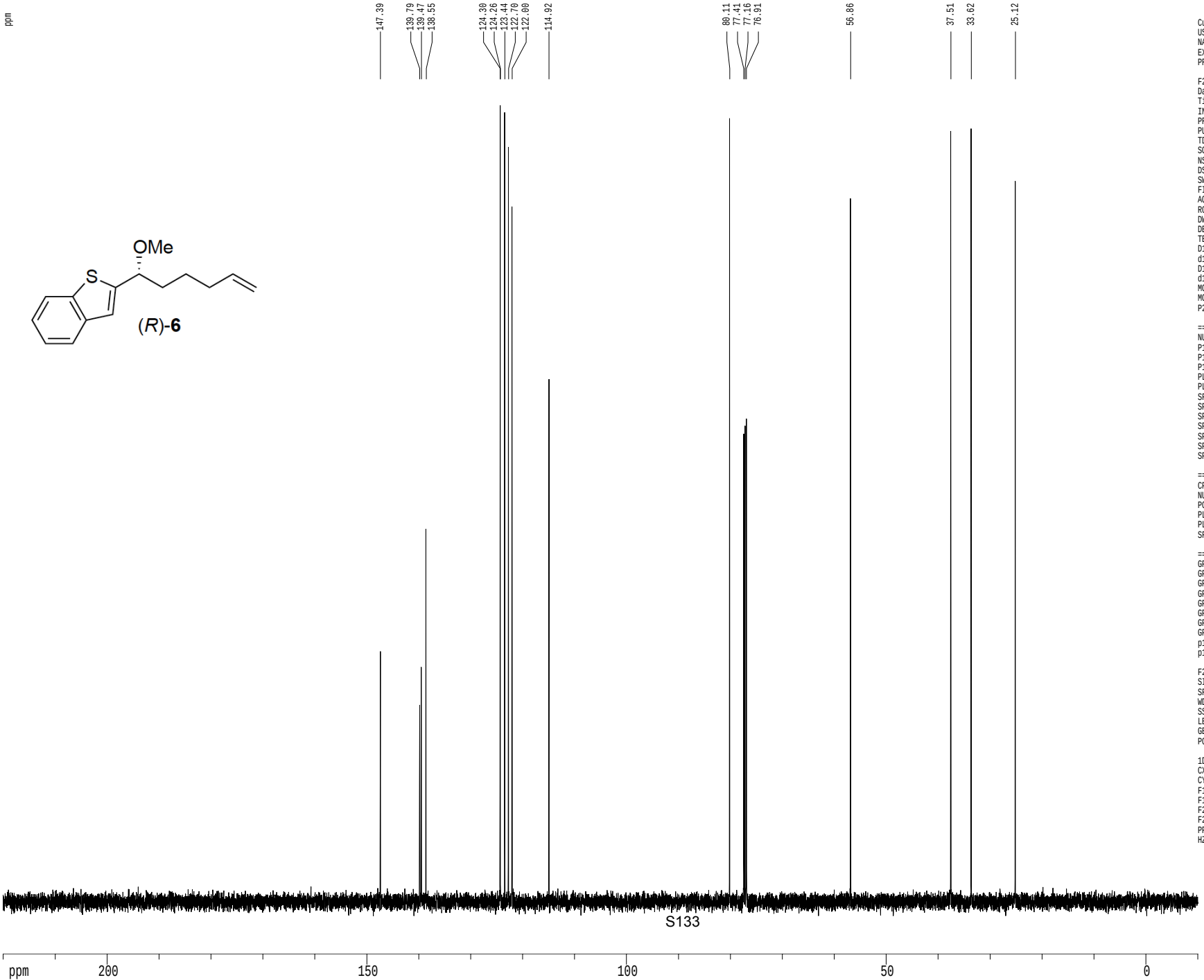
F2 - Acquisition Parameters
Date_ 20130918
Time 17.26
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SF01 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200404 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-248-13CNMR
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20130918
Time              17.28
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                   61
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

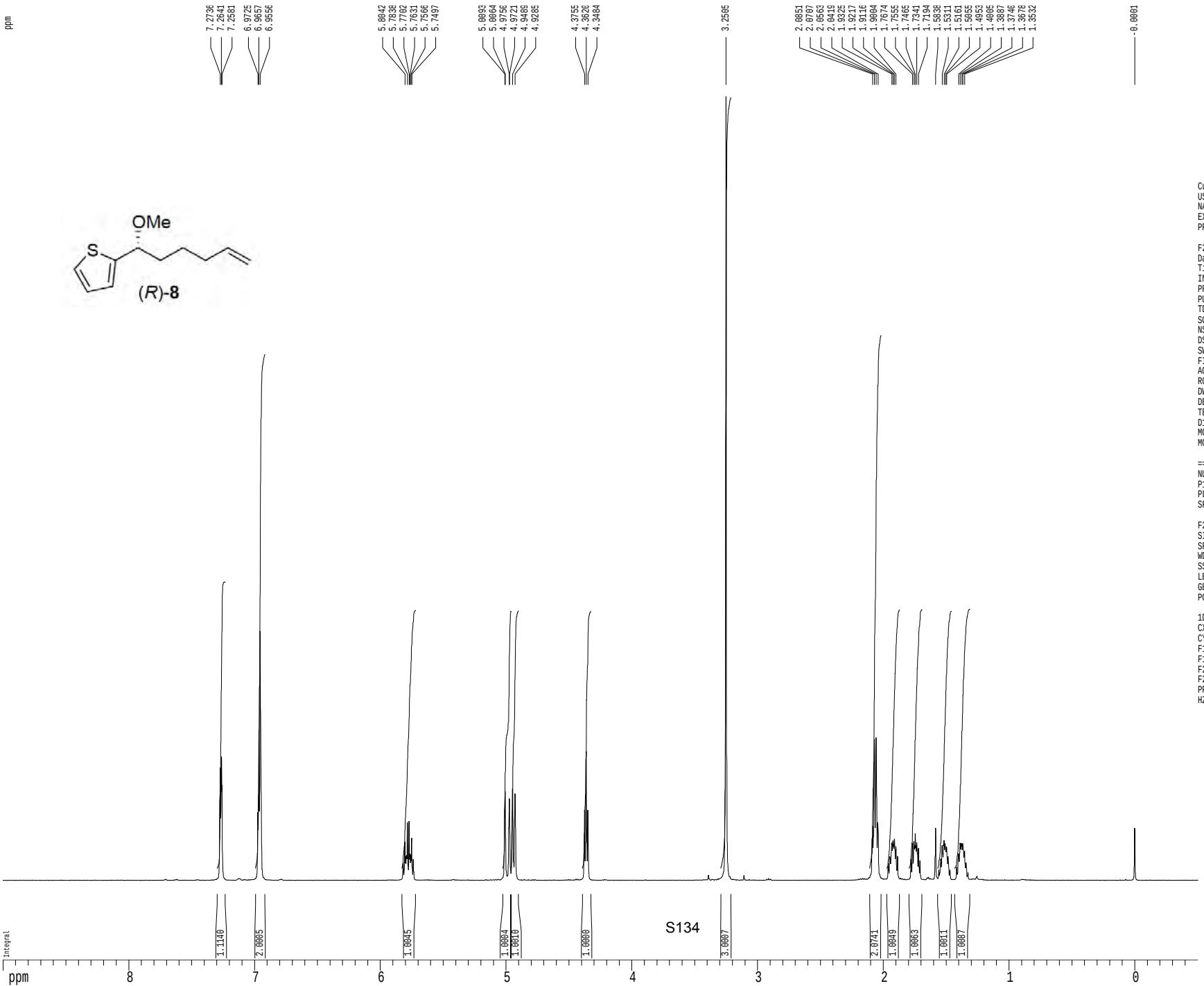
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804117 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            220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-93-1HNMR
EXPNO 1
PROCNO 1

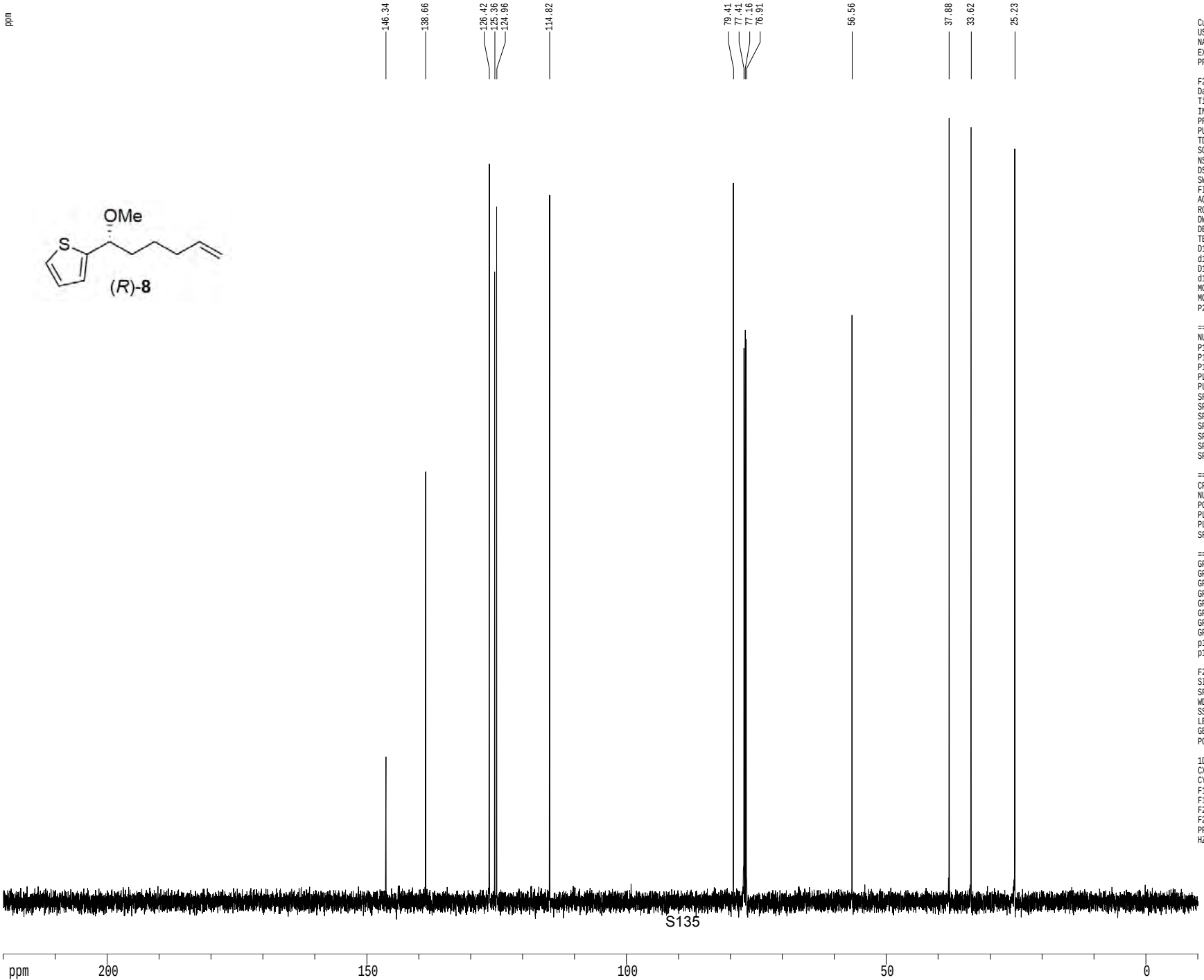
F2 - Acquisition Parameters
Date_ 20131209
Time 16.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200318 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-93-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131209
Time             16.06
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT         CDCl3T
NS                 135
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG             11585.2
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

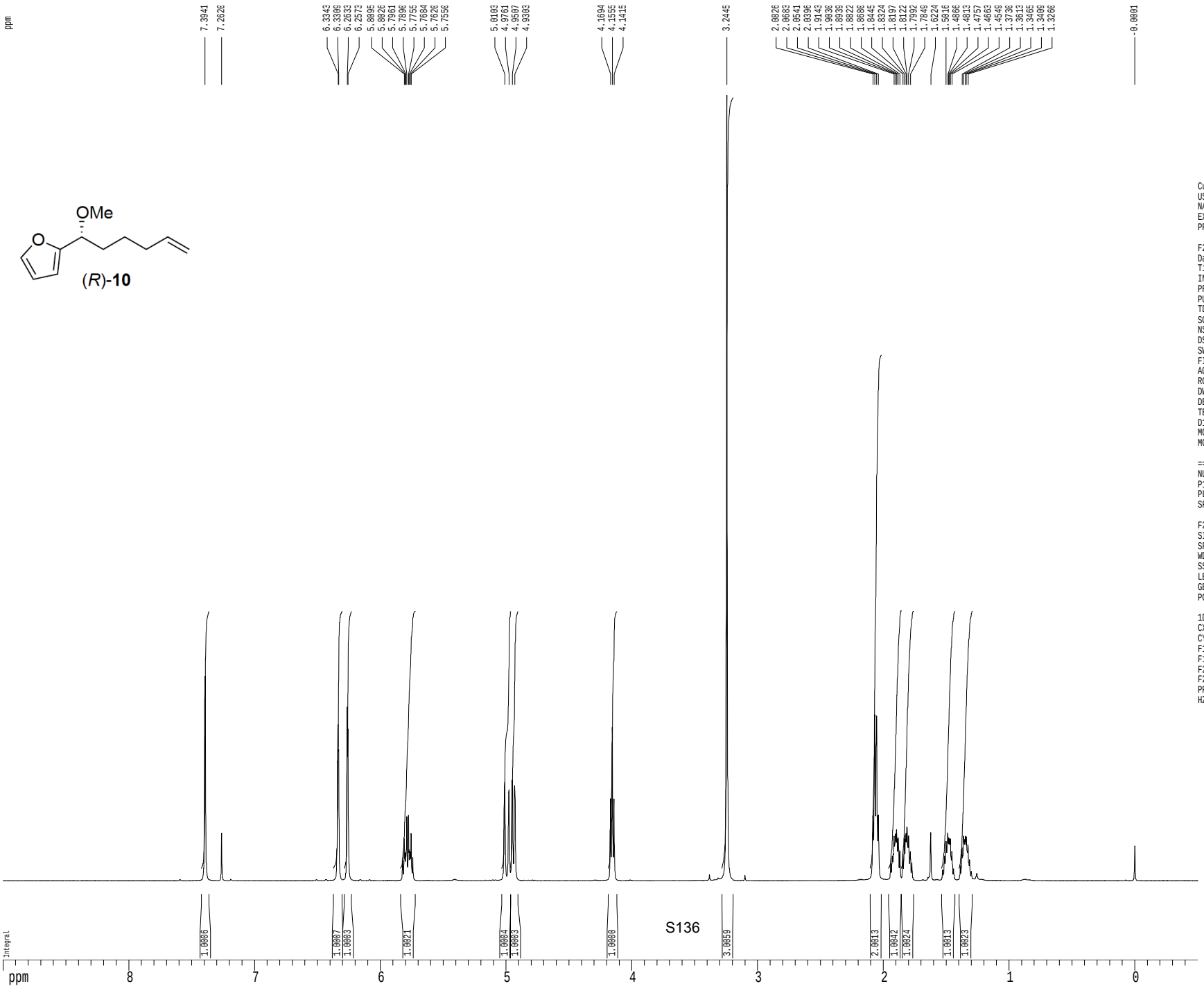
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804090 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             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-296-1HNMR
EXPNO 1
PROCNO 1

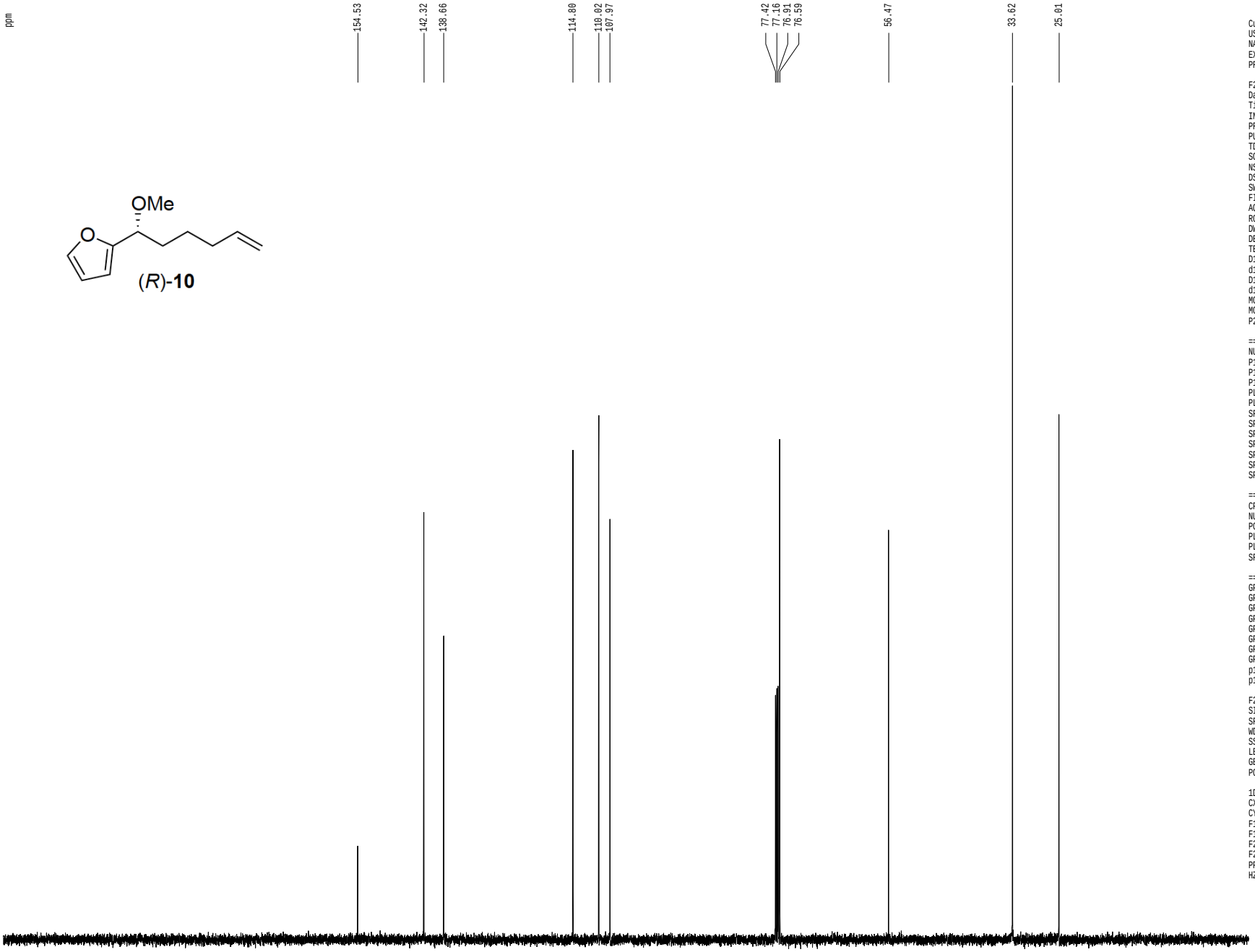
F2 - Acquisition Parameters
Date_ 20131210
Time 18.12
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200298 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-296-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131210
Time              18.15
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                   72
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG                 3251
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

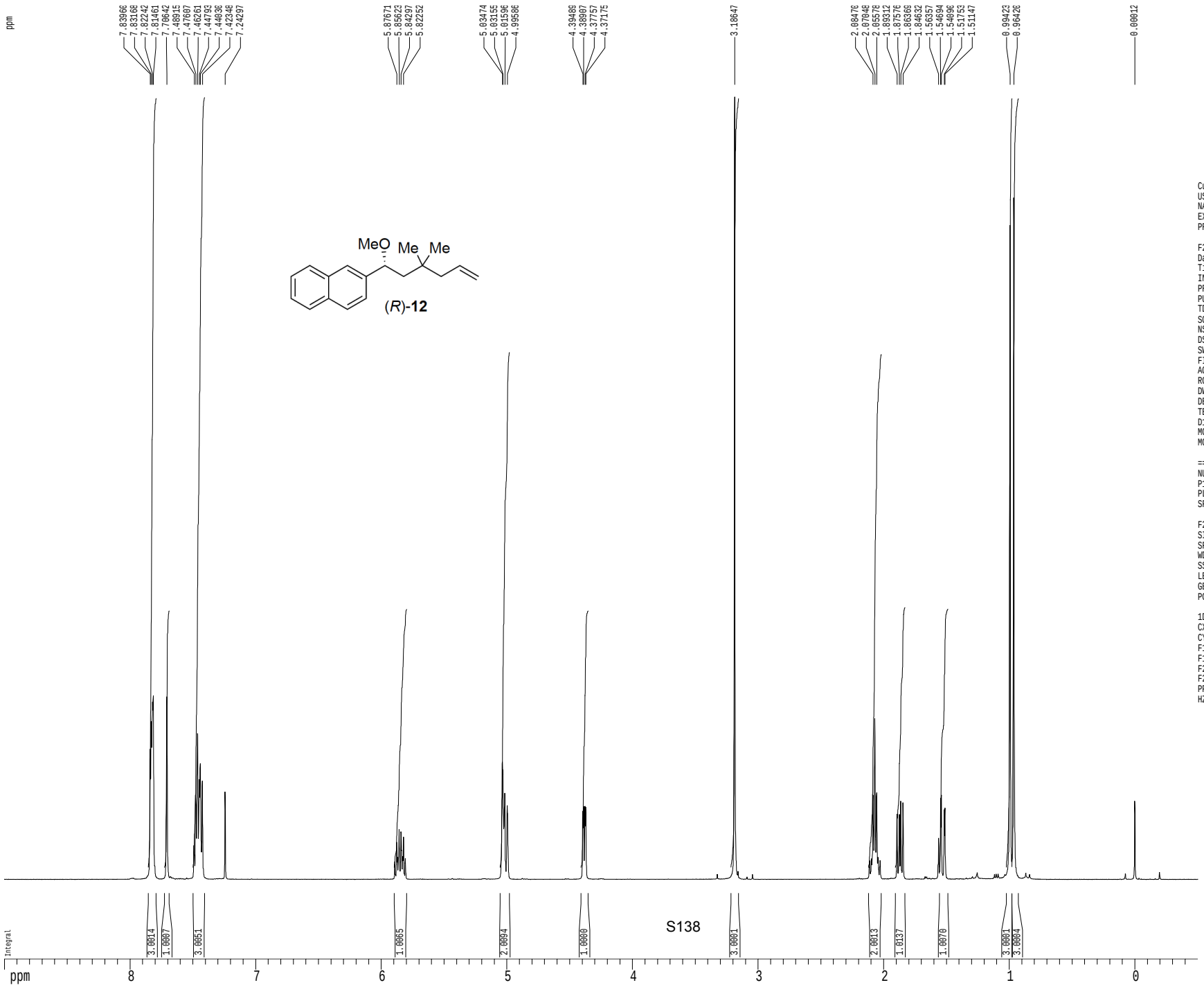
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804090 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-32-1HNMR
EXPNO 1
PROCNO 1

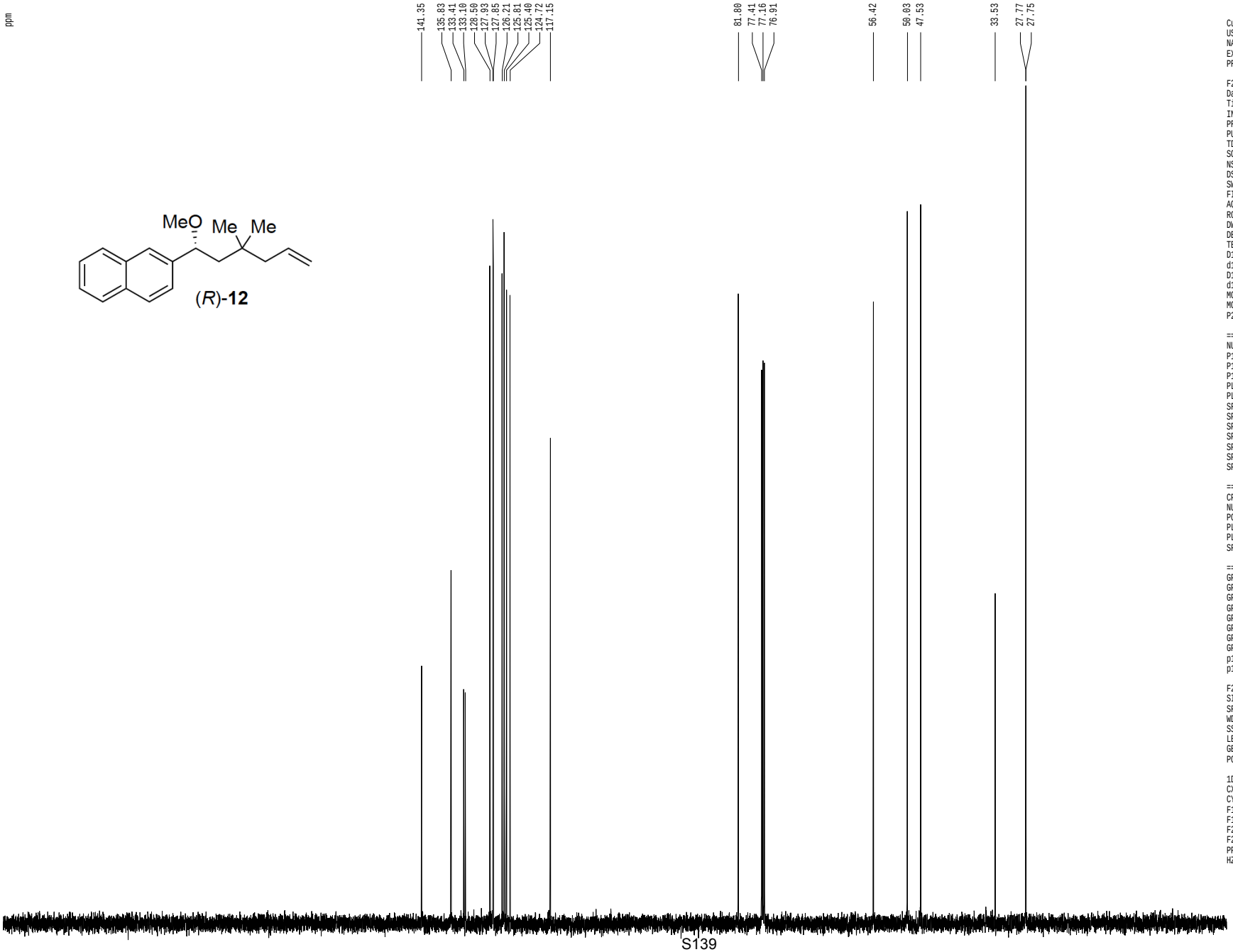
F2 - Acquisition Parameters
Date_ 20130915
Time 17.31
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200396 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-32-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130915
Time              17.33
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  102
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

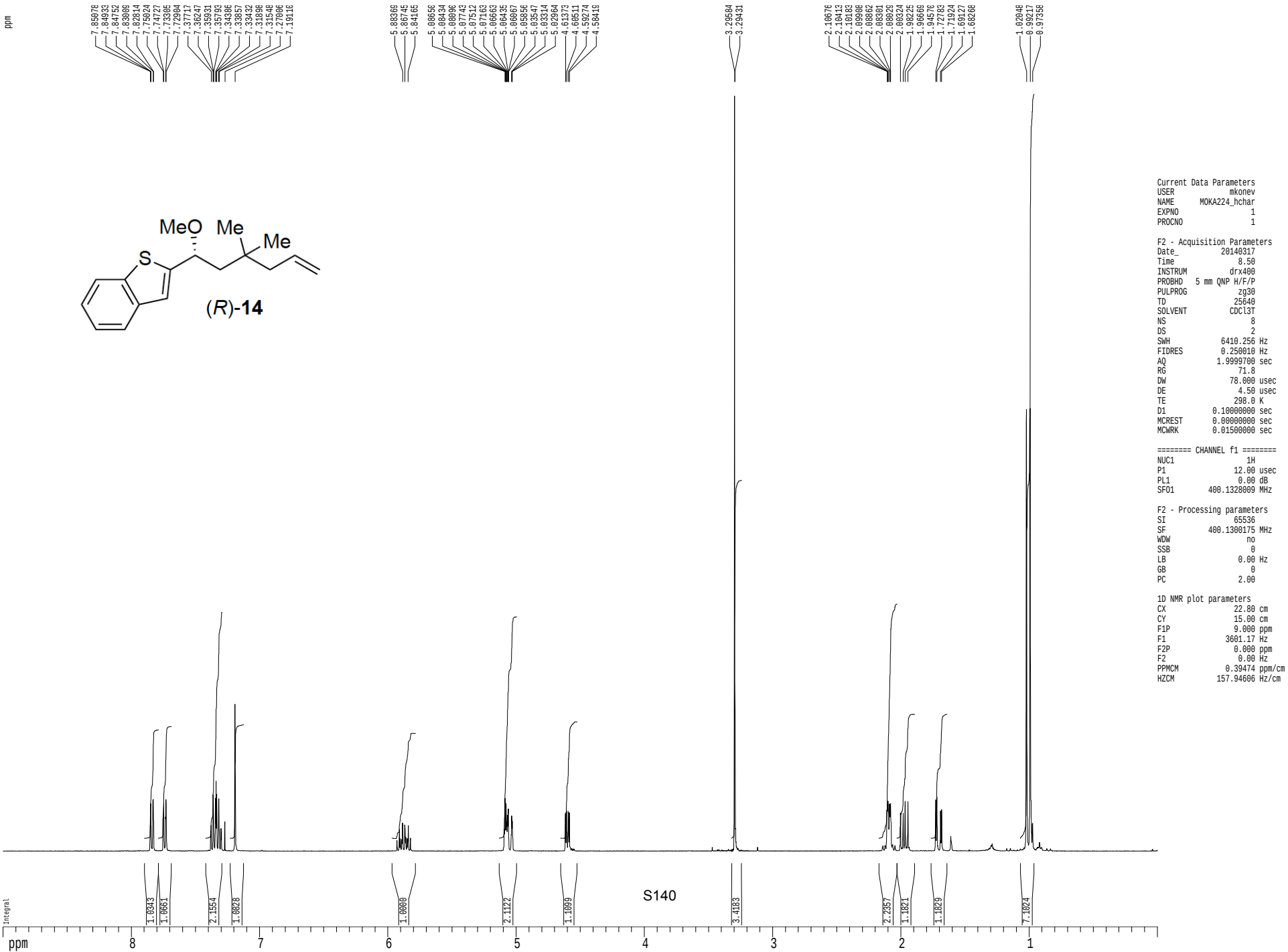
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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

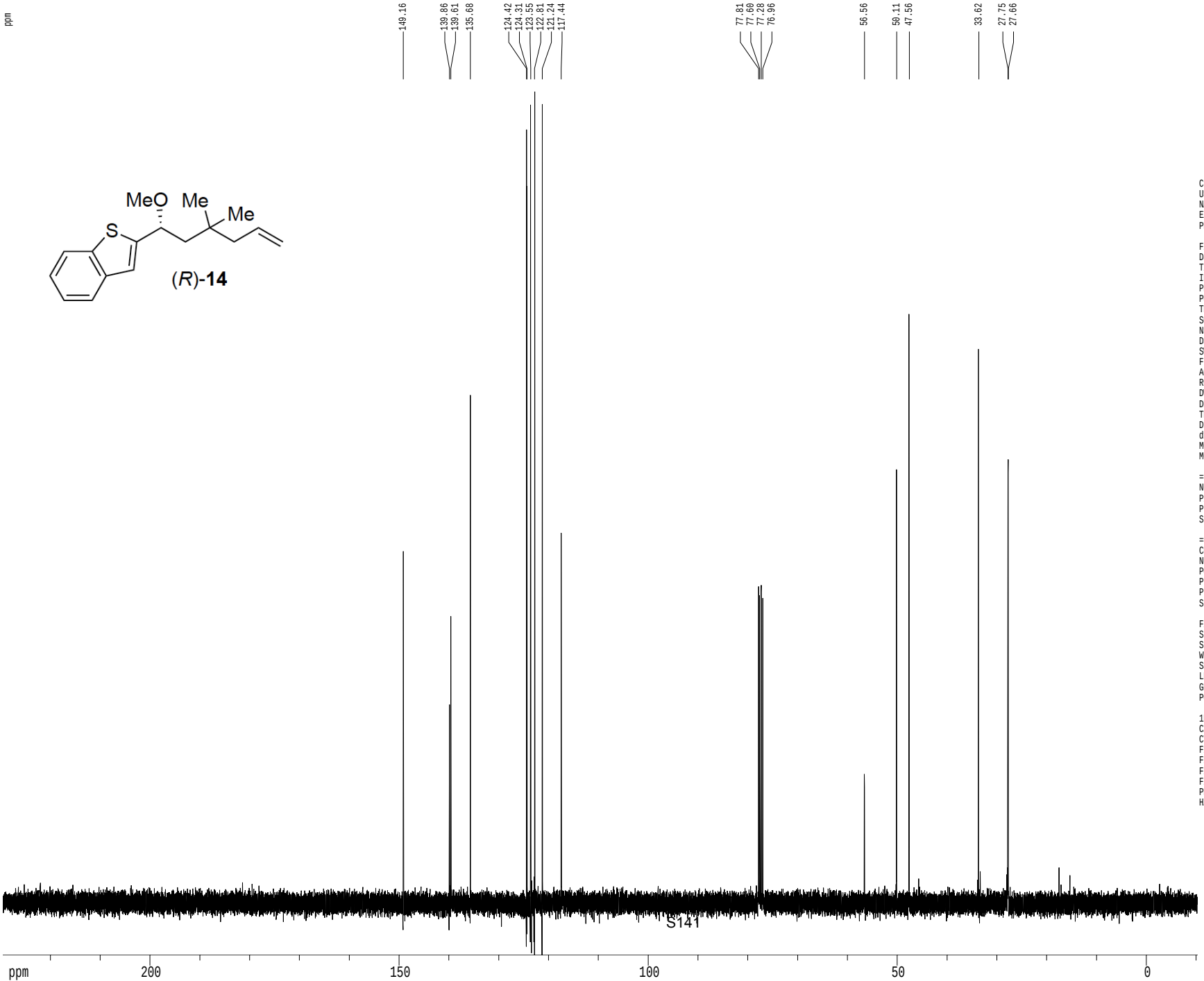
F2 - Processing parameters
SI                 65536
SF          125.7804103 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum


ppm
7.85078
7.84933
7.84752
7.83008
7.82814
7.75924
7.74727
7.73386
7.72904
7.37117
7.36247
7.35931
7.35793
7.34386
7.33857
7.33432
7.31898
7.31548
7.27006
7.19116
5.88368
5.86745
5.84165
5.00656
5.00434
5.00098
5.07743
5.07512
5.07163
5.06663
5.06435
5.06067
5.05856
5.03547
5.03314
5.02904
4.61373
4.60511
4.59274
4.58419
3.29584
3.29431
2.10676
2.10413
2.10183
2.09908
2.08862
2.08301
2.08029
2.08324
1.96668
1.94576
1.72783
1.71924
1.69127
1.68268
1.02048
0.99217
0.97358
MeO
Me
Me
S
(R)-14
Integral
1.0343
1.0661
2.1554
1.0828
1.0000
2.1122
1.1099
3.4183
2.2357
1.1821
1.1829
7.1024
ppm
8
7
6
5
4
3
2
1
Current Data Parameters
USER mkonev
NAME MOKA224_hchar
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140317
Time 8.50
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 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 no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94606 Hz/cm

13C spectrum with 1H decoupling




```
Current Data Parameters
USER             mkonev
NAME      MOKA224_cchar
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20140317
Time               8.54
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  256
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

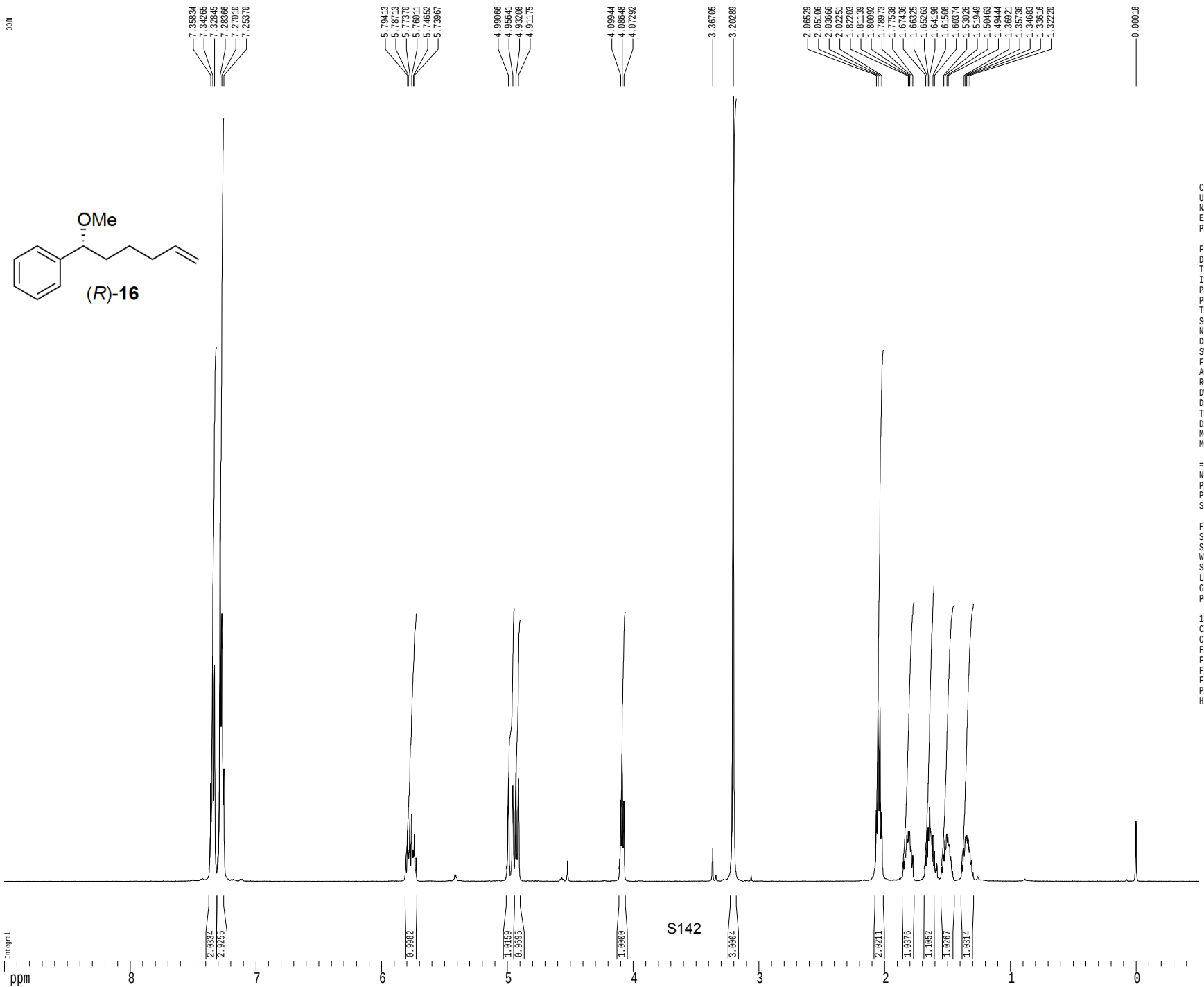
======== CHANNEL f1 =======
NUC1               13C
P1                7.75 usec
PL1              -3.00 dB
SFO1        100.6237964 MHz

======== 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                 no
SSB                  0
LB                0.00 Hz
GB                   0
PC                1.00

1D NMR plot parameters
CX                22.80 cm
CY                15.50 cm
F1P             229.496 ppm
F1            23090.21 Hz
F2P             -10.579 ppm
F2            -1064.37 Hz
PPMCM          10.52959 ppm/cm
HZCM         1059.41138 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-IV-273-1HNMR
EXPNO 1
PROCNO 1

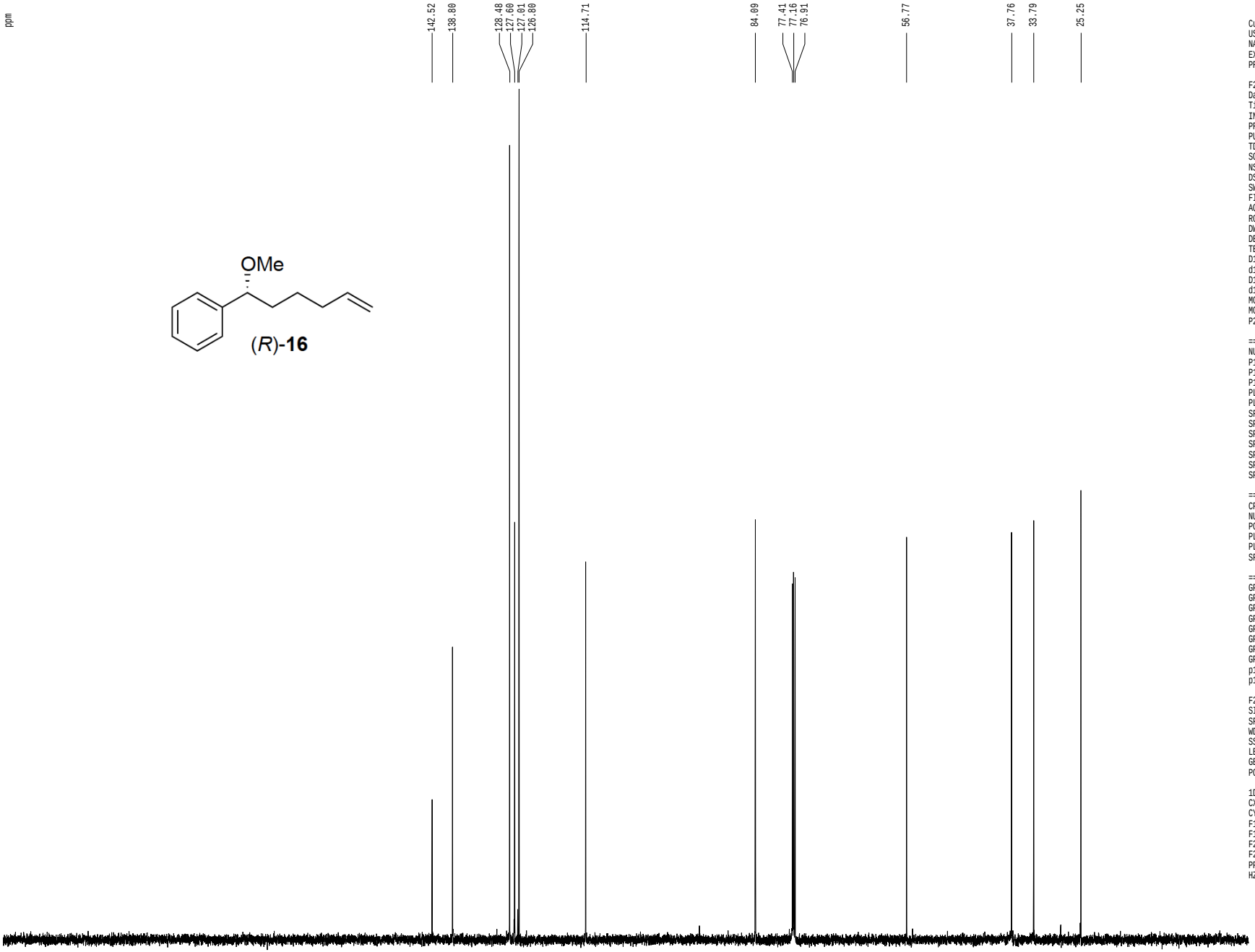
F2 - Acquisition Parameters
Date_ 20140430
Time 15.42
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200357 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-IV-273-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140430
Time              15.46
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   97
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ           1.0813940 sec
RG                 6502
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                31.00 usec

======== CHANNEL f1 =======
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

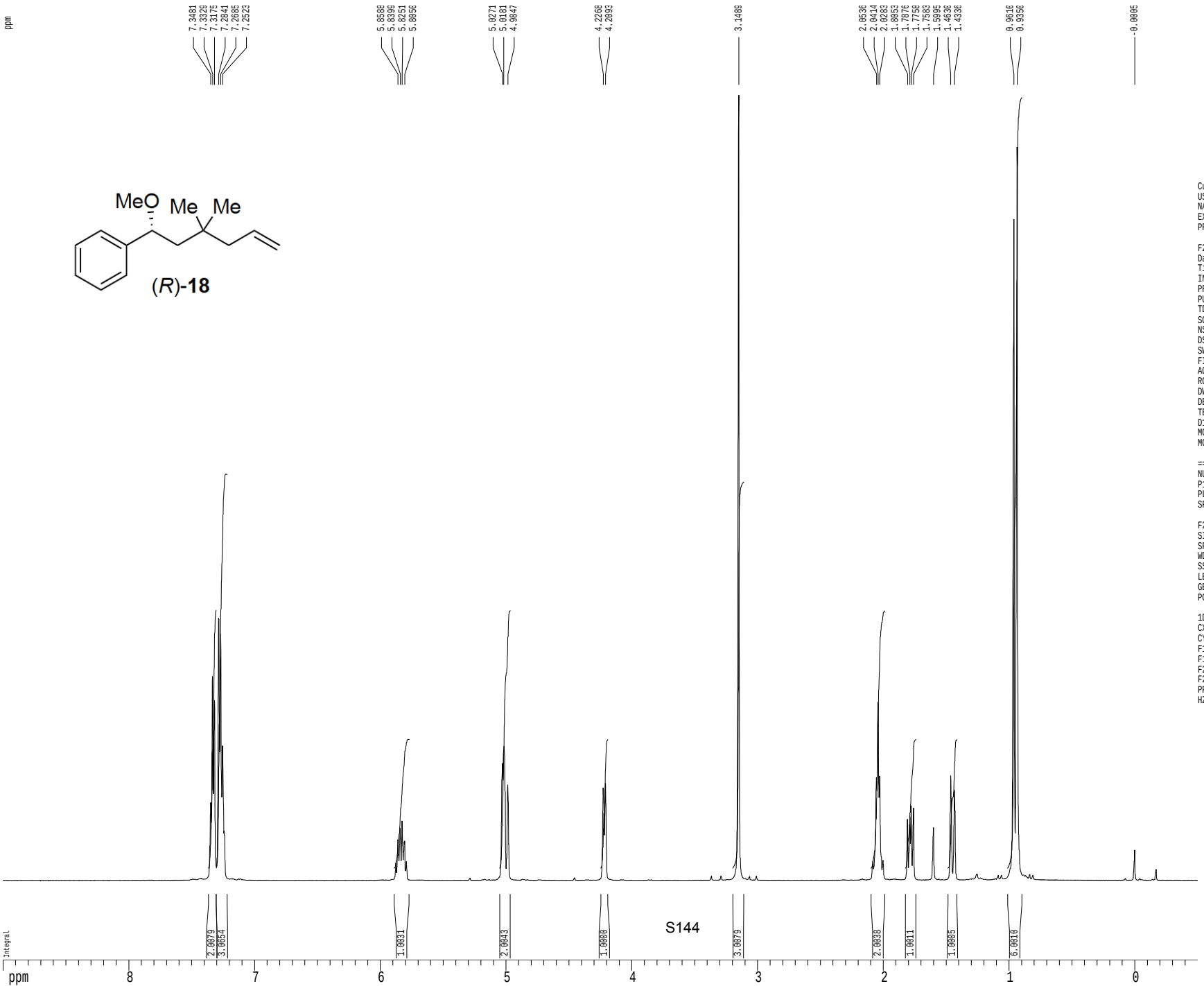
======== CHANNEL f2 =======
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804099 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            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-44-1HNMR
EXPNO 1
PROCNO 1

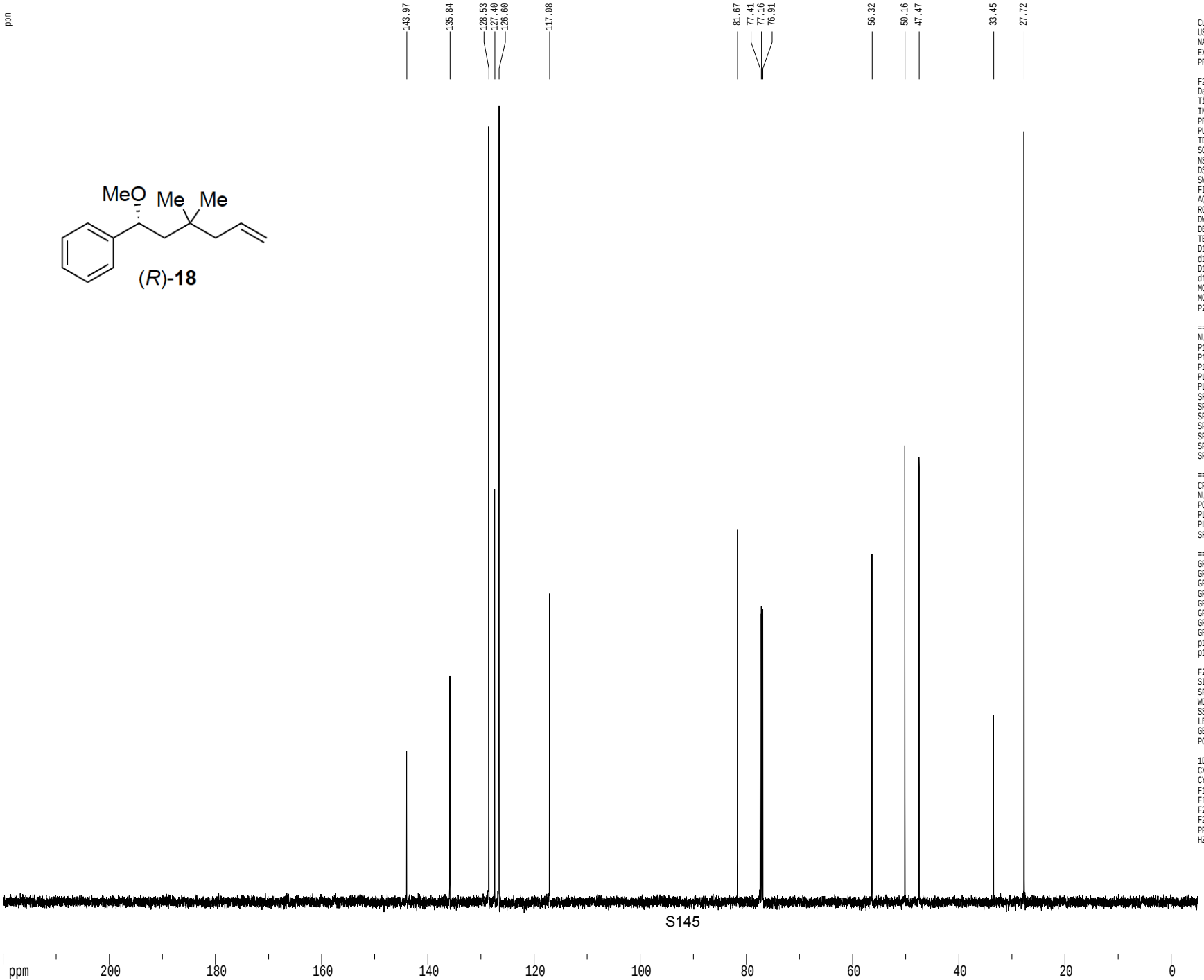
F2 - Acquisition Parameters
Date_ 20140307
Time 18.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200355 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME      MRH-V-44-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140307
Time              18.34
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   76
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

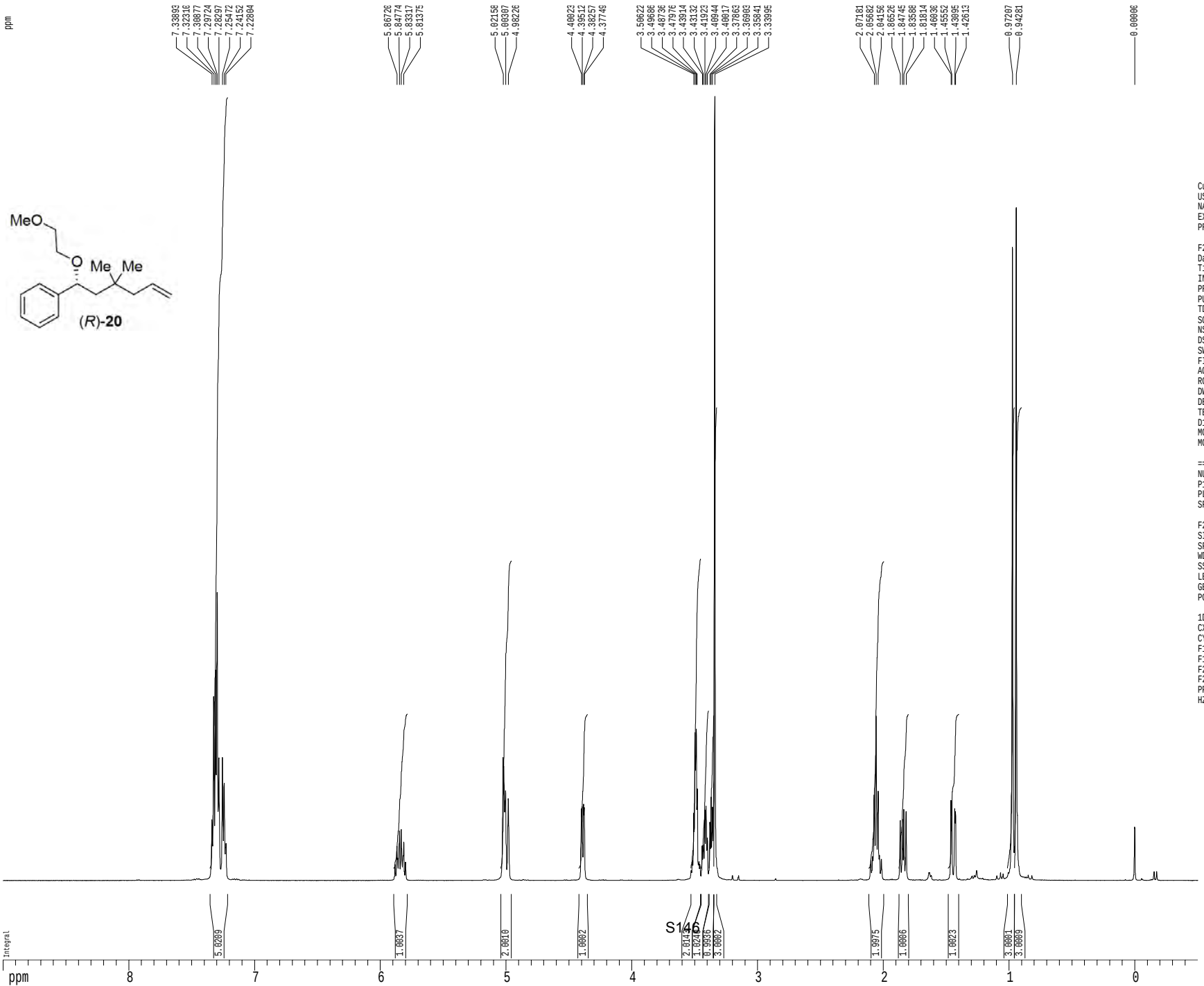
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804094 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             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-185-1HNMR
EXPNO 1
PROCNO 1

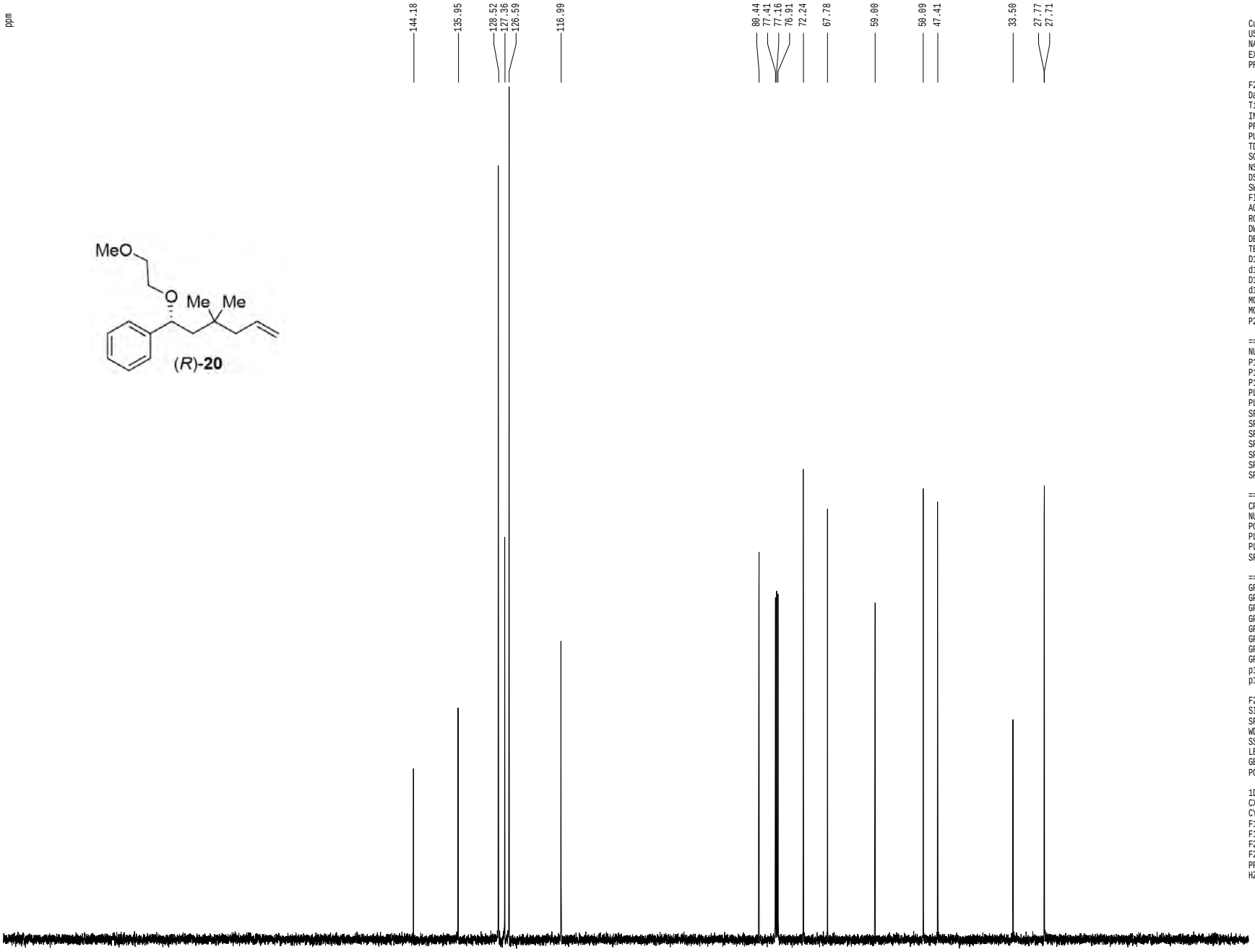
F2 - Acquisition Parameters
Date_ 20130918
Time 17.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200335 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-185-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130918
Time              17.06
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   95
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG              10321.3
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

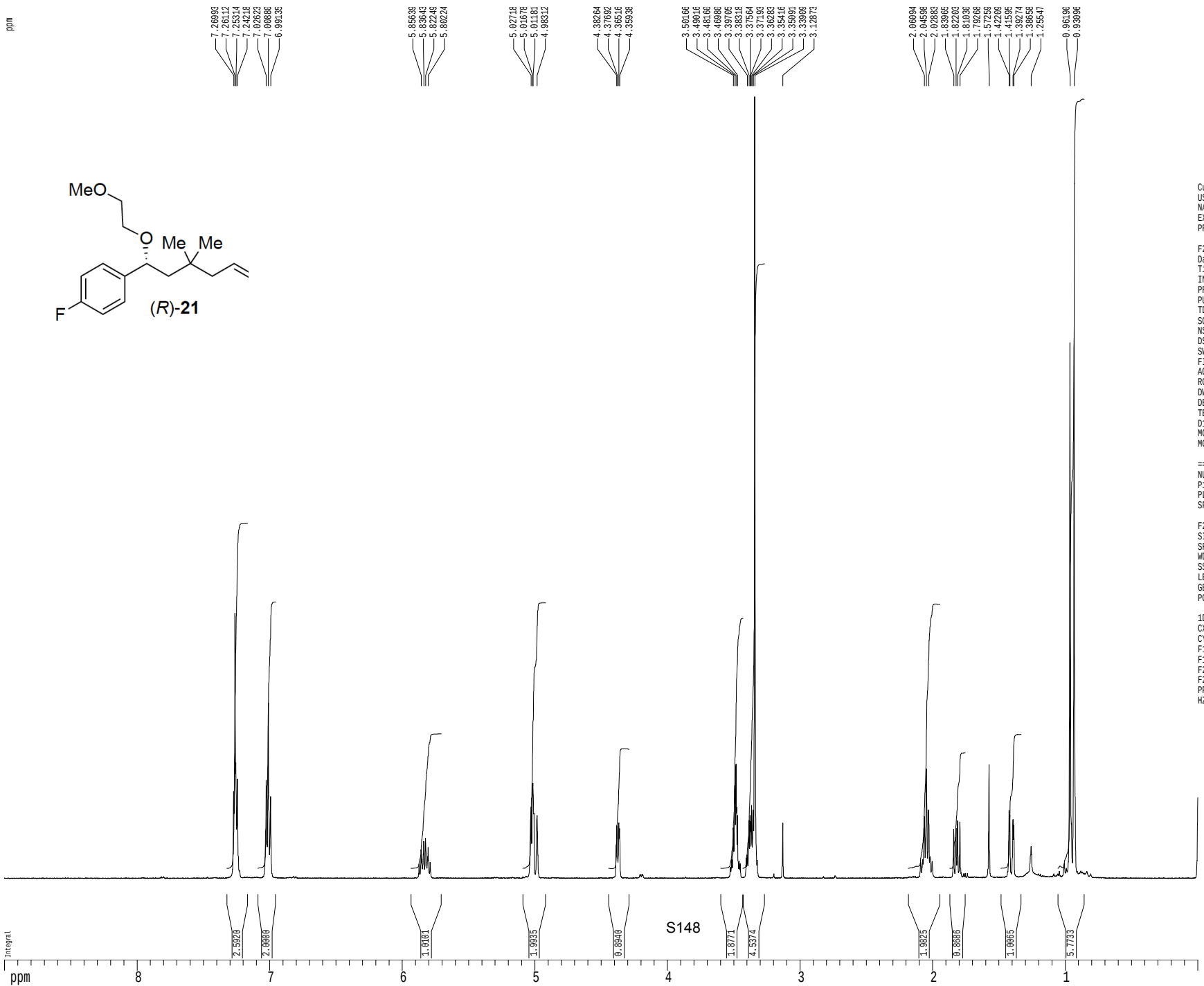
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804099 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            220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mkonev
NAME MOKA081h-char
EXPNO 1
PROCNO 1

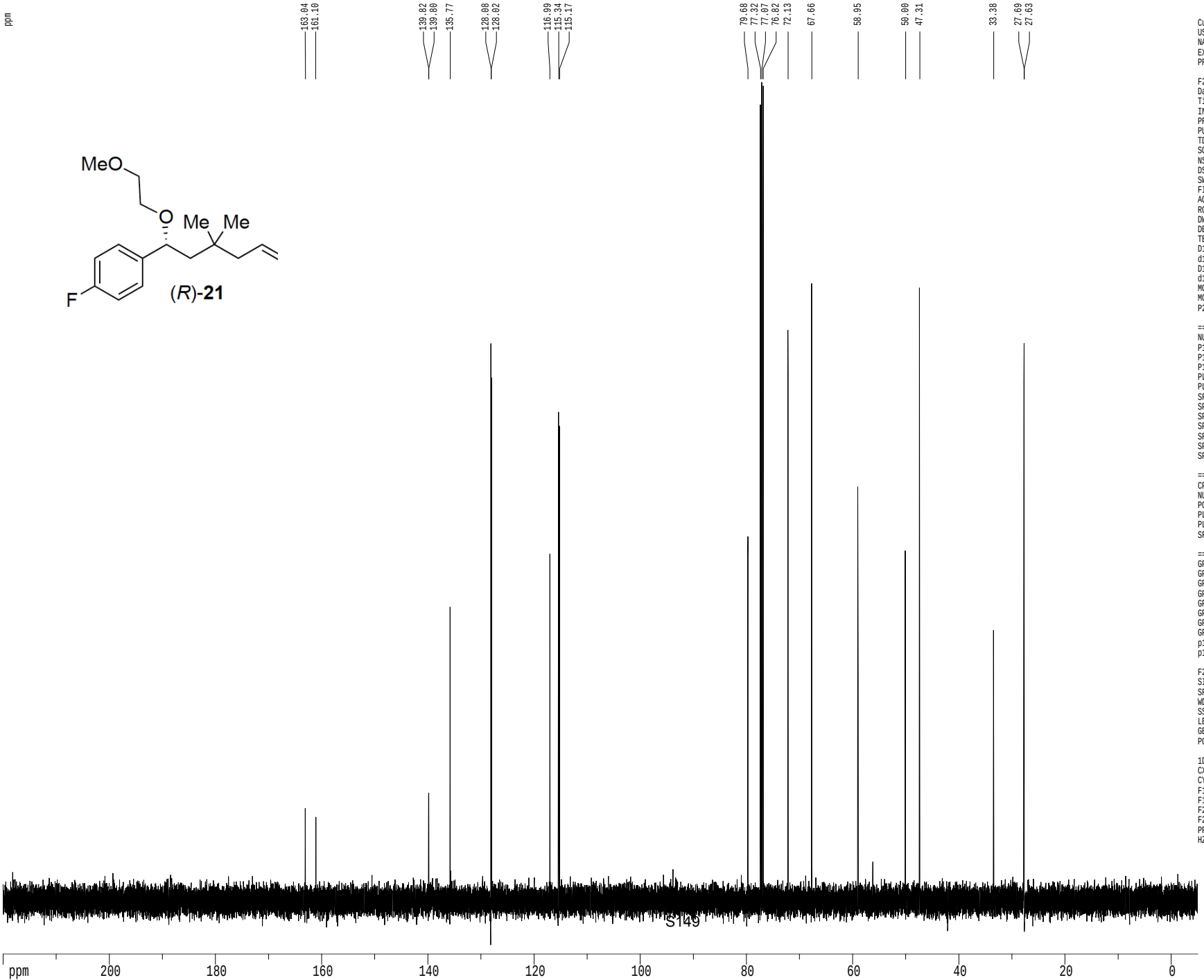
F2 - Acquisition Parameters
Date_ 20131120
Time 1.48
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200303 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm

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




```
Current Data Parameters
USER              mkonev
NAME        MOKA081c-char
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20131120
Time                 1.53
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                  65536
SOLVENT             CDCl3
NS                    352
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                 31.00 usec

======== CHANNEL f1 ========
NUC1                  13C
P1                  15.50 usec
P11                500.00 usec
P12               2000.00 usec
PL0                120.00 dB
PL1                 -1.00 dB
SFO1          125.7942548 MHz
SP1                  3.20 dB
SP2                  3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1               0.00 Hz
SPOFF2               0.00 Hz

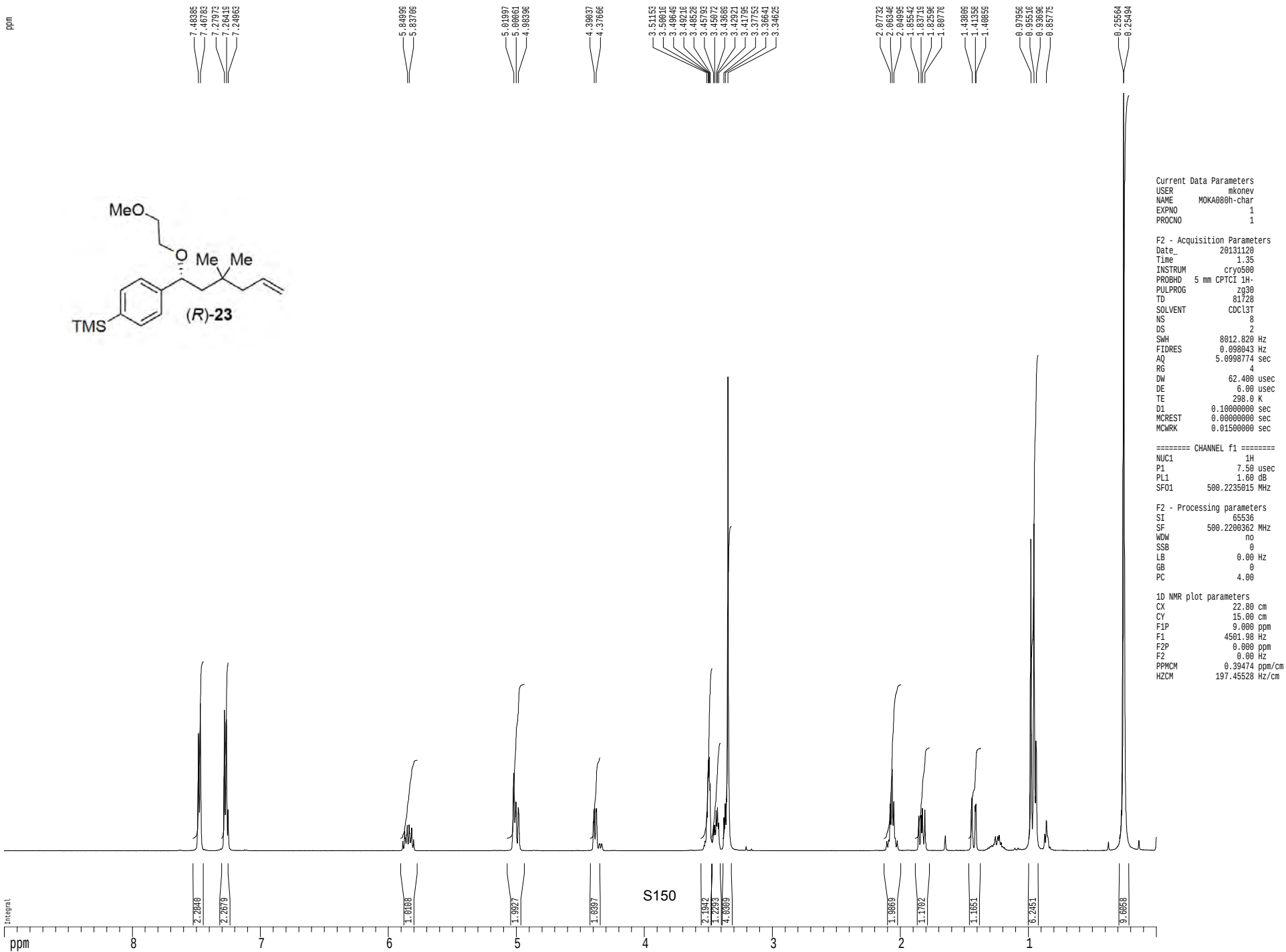
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                  1.60 dB
PL12                24.60 dB
SFO2          500.2225011 MHz

====== 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

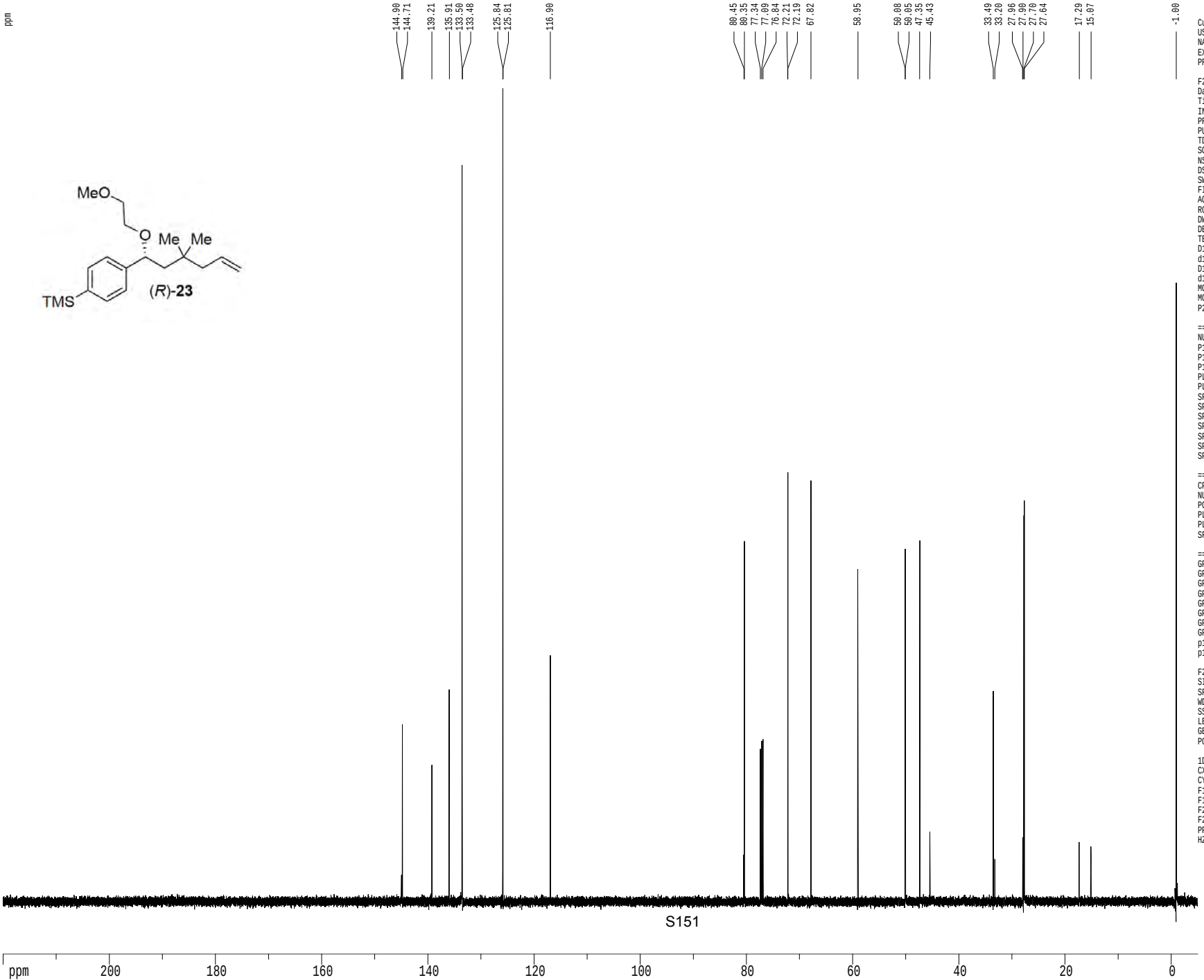
F2 - Processing parameters
SI                    65536
SF            125.7804190 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     2.00

1D NMR plot parameters
CX                    22.80 cm
CY                    15.65 cm
F1P                 220.000 ppm
F1                27671.69 Hz
F2P                  -5.000 ppm
F2                 -628.90 Hz
PPMCM               9.86842 ppm/cm
HZCM             1241.25415 Hz/cm
```

1H spectrum


ppm
7.48385
7.46783
7.27973
7.26419
7.24963
5.84996
5.83769
5.01997
5.00061
4.96396
4.39037
4.37666
3.51153
3.50016
3.49649
3.49216
3.48528
3.45793
3.45072
3.43668
3.42921
3.41795
3.37753
3.36641
3.34625
2.07732
2.06346
2.04995
1.85542
1.83719
1.82596
1.80776
1.43809
1.41358
1.40859
0.97956
0.95516
0.93696
0.85775
0.25564
0.25494
MeO
O
Me
Me
TMS
(R)-23
Current Data Parameters
USER mkonev
NAME MOKA080h-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131120
Time 1.35
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 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200362 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm
Integral
2.2840
2.2679
1.0108
1.9927
1.0397
2.1942
1.2293
4.0389
1.9869
1.1702
1.1651
6.2451
9.6058
ppm
8
7
6
5
4
3
2
1

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




```
Current Data Parameters
USER            mkonev
NAME      MOKA080c-char
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_          20131120
Time               1.38
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                 131
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

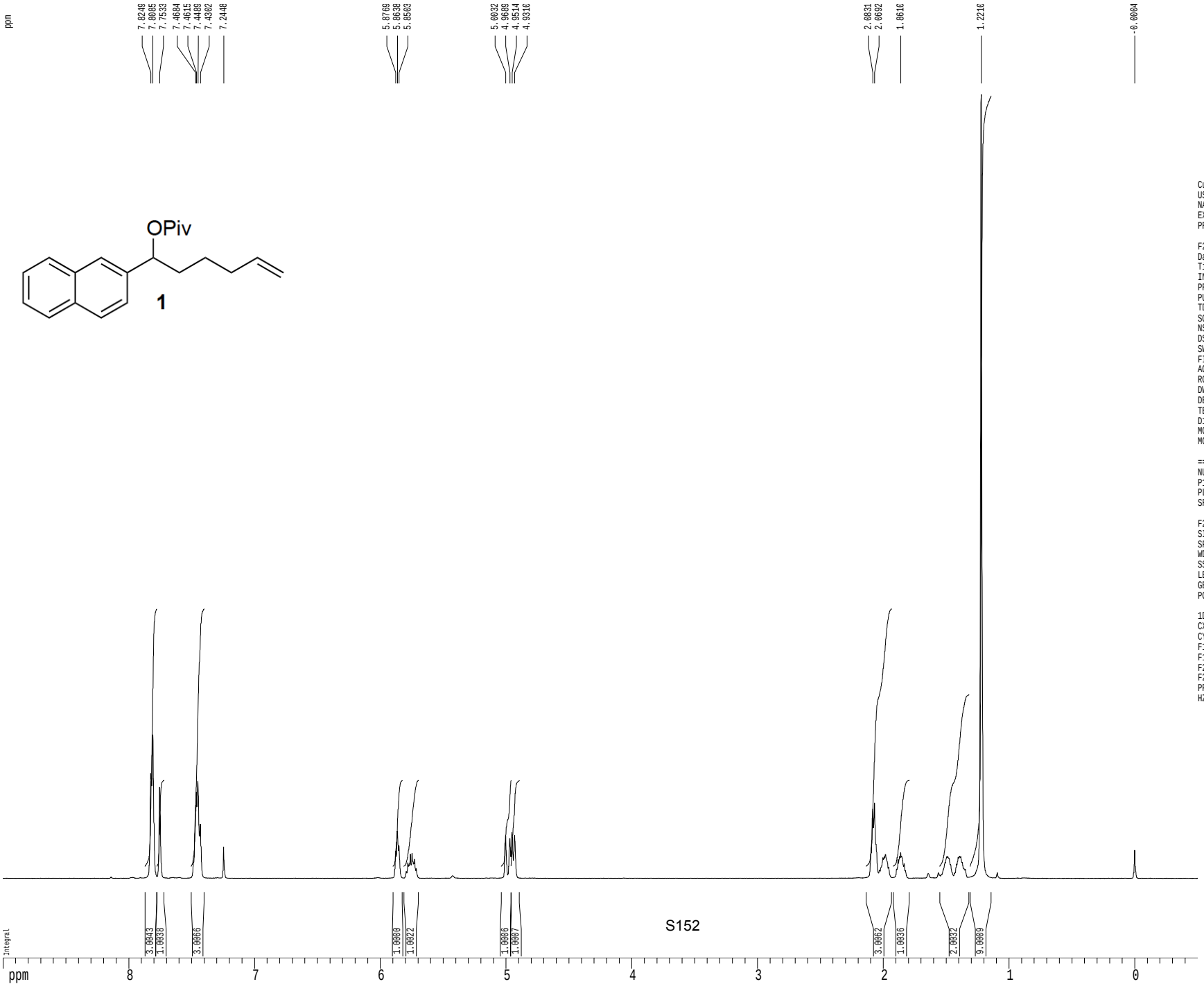
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P            220.000 ppm
F1           27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM          9.86842 ppm/cm
HZCM        1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-IV-223-1HNMR
EXPNO 1
PROCNO 1

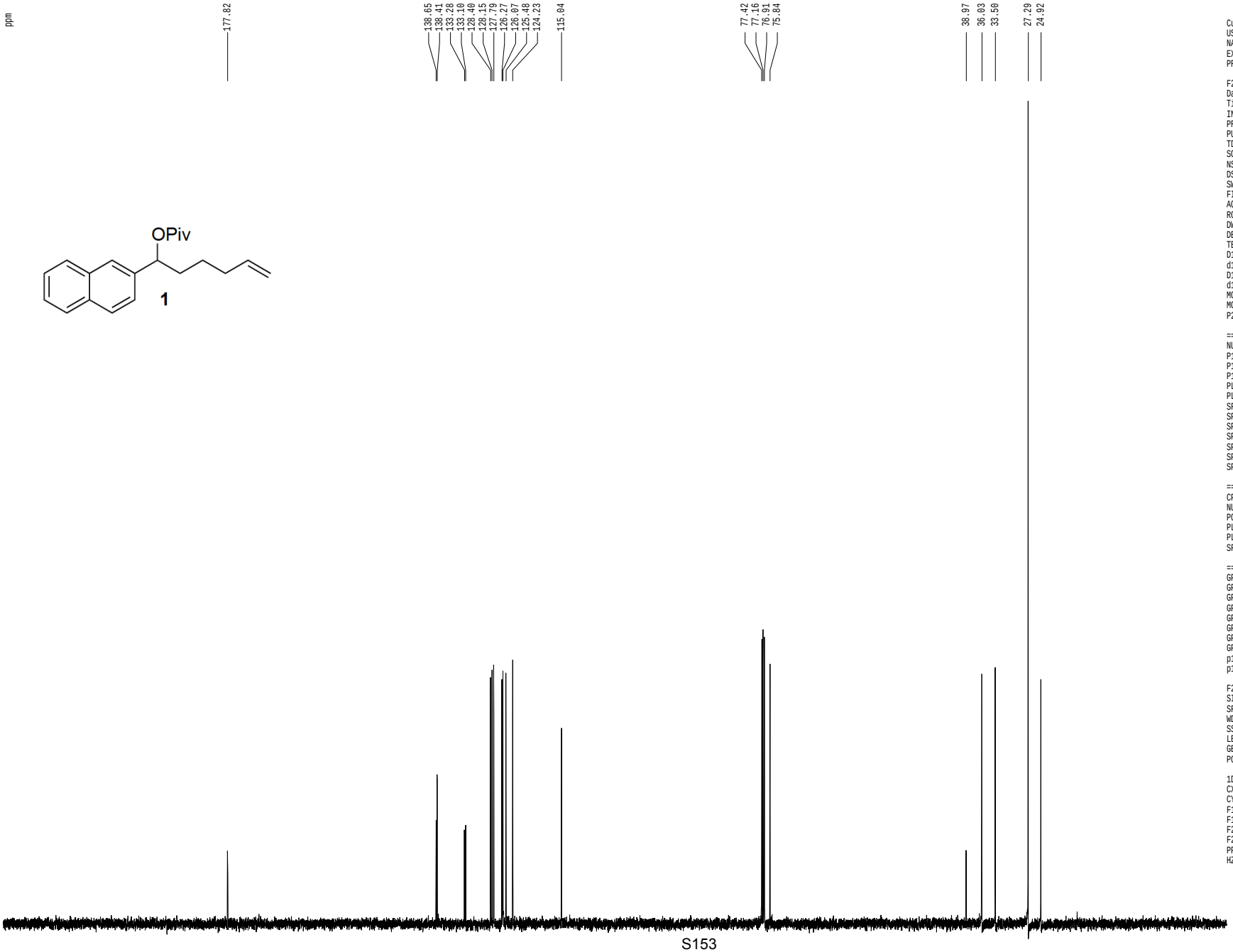
F2 - Acquisition Parameters
Date_ 20140116
Time 17.02
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200373 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-IV-223-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140116
Time              17.04
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   76
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ           1.0813940 sec
RG             14596.5
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

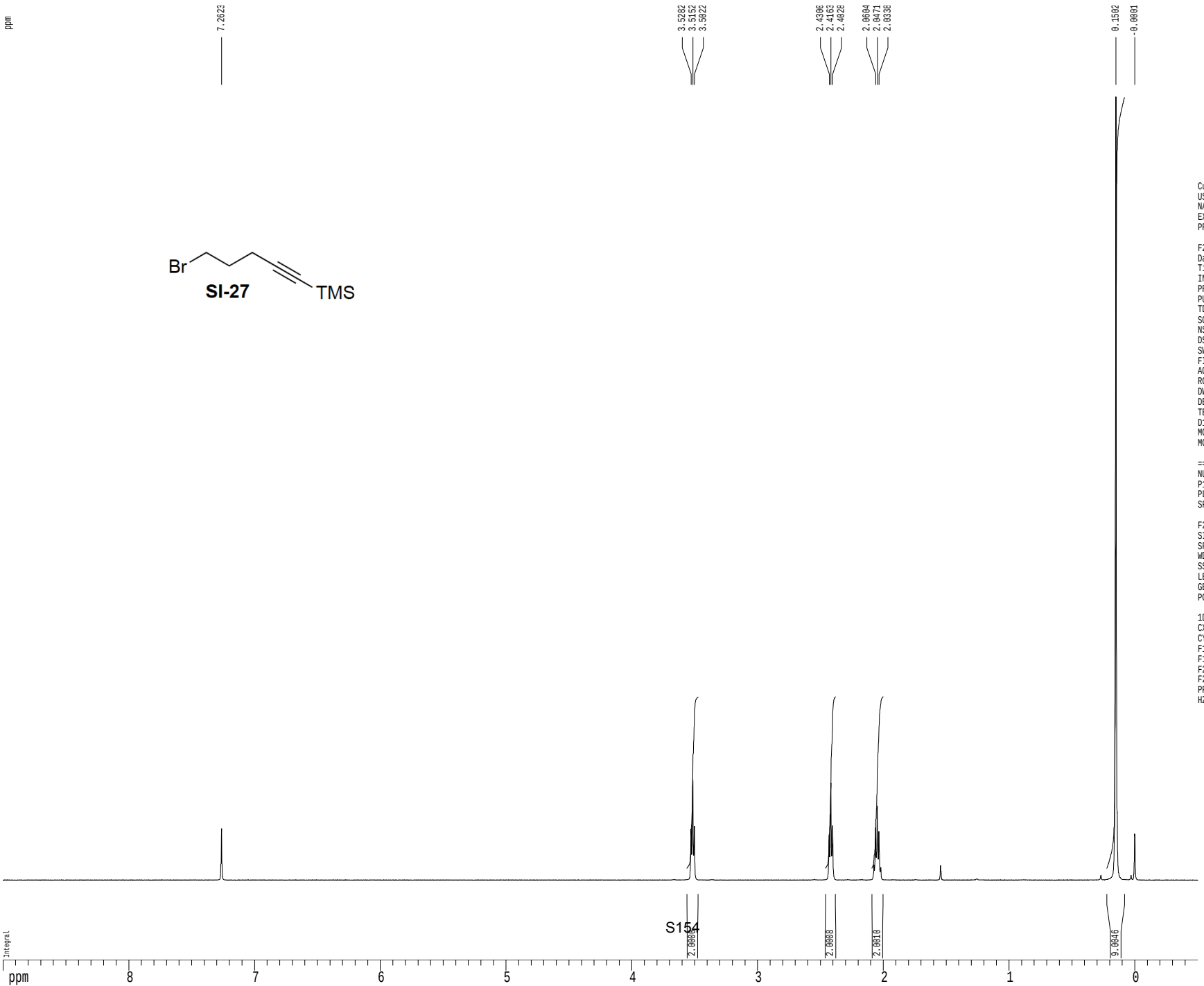
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804099 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER            mharri
NAME      MRH-V-111-1HNMR
EXPNO                 1
PROCNO                1

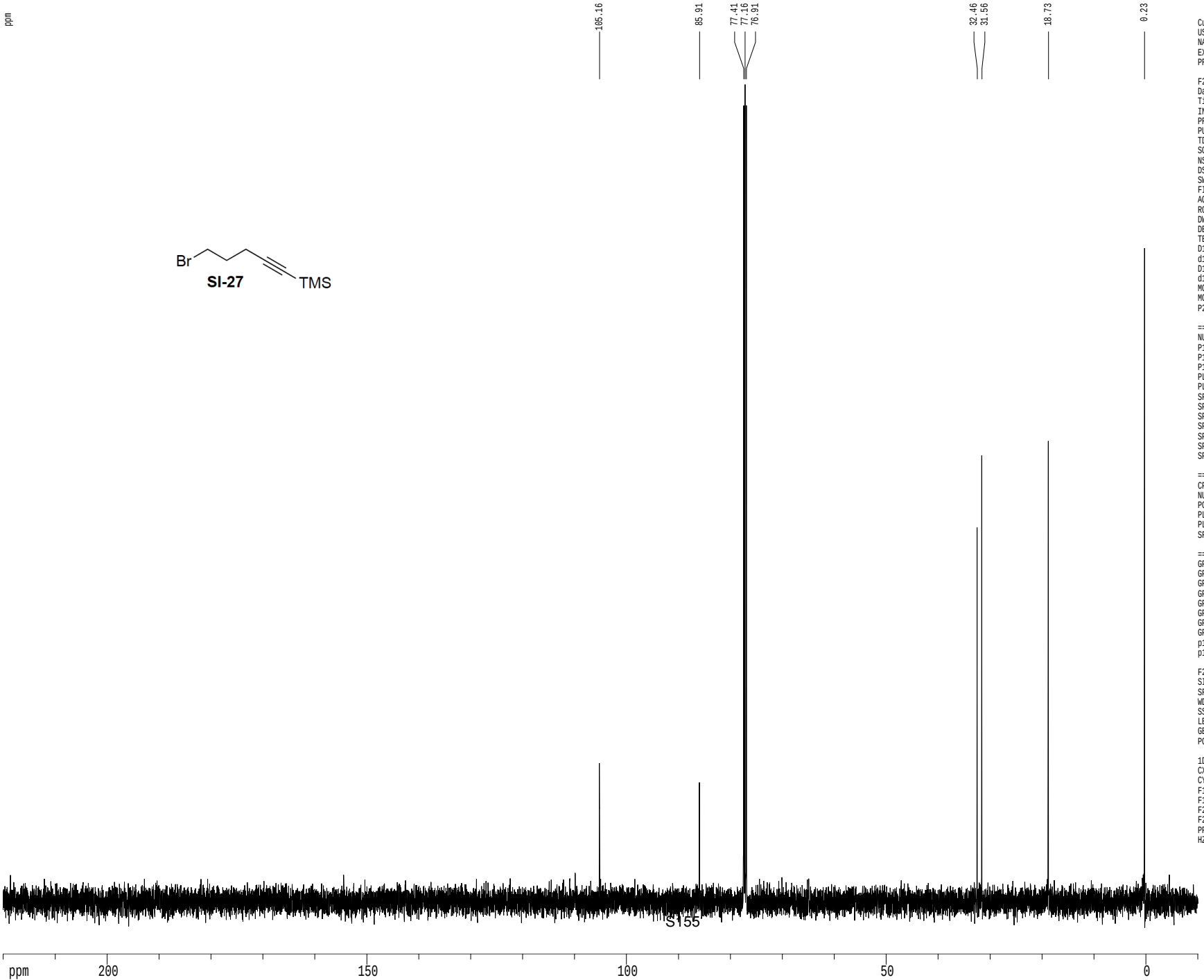
F2 - Acquisition Parameters
Date_          20140116
Time              17.13
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    1
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                    9
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          500.2200281 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               9.000 ppm
F1             4501.98 Hz
F2P             -0.500 ppm
F2             -250.11 Hz
PPMCM          0.41667 ppm/cm
HZCM         208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-111-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140116
Time              17.15
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  126
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG               11585.2
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                 31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 3.20 dB
SP2                 3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1              0.00 Hz
SPOFF2              0.00 Hz

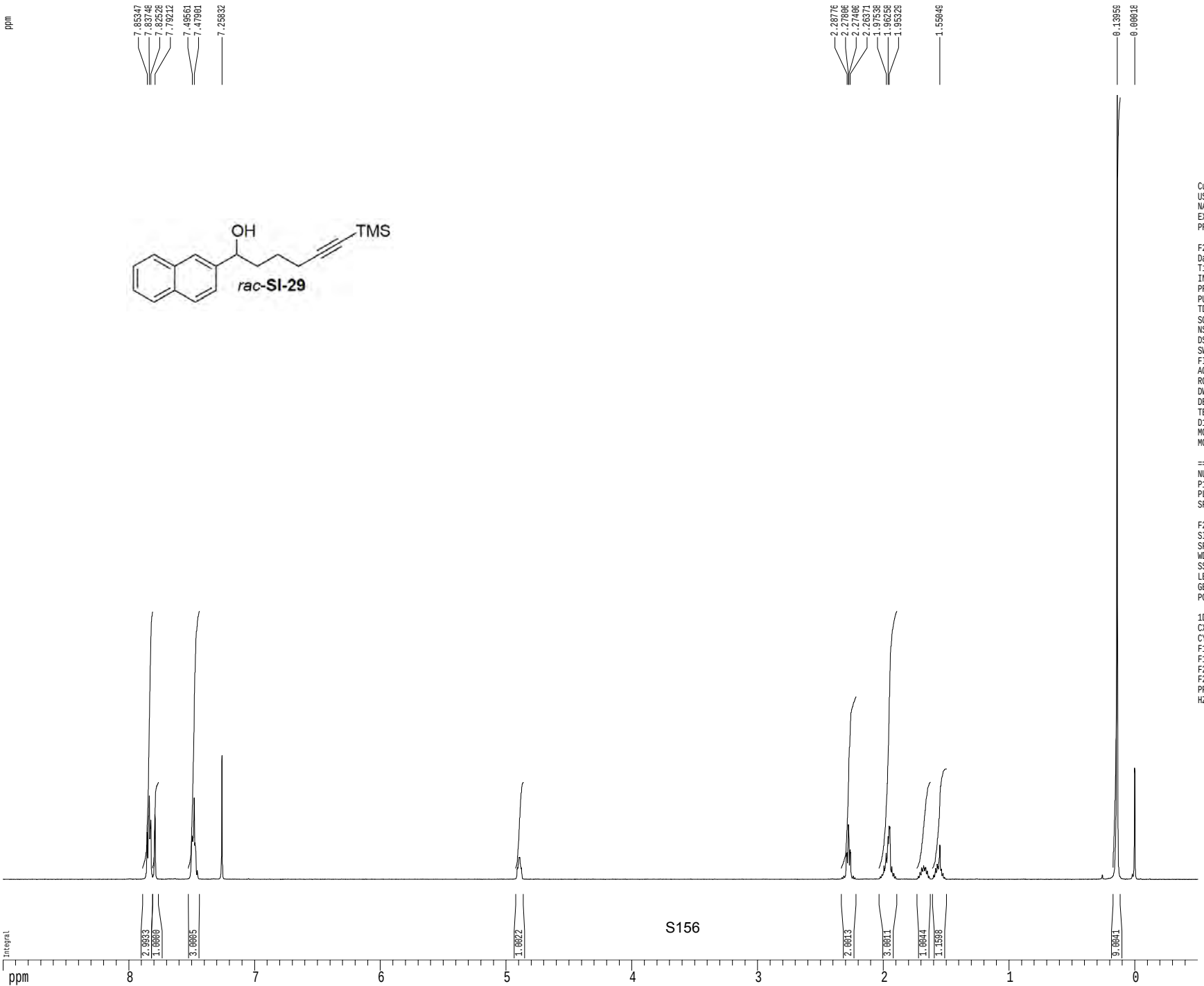
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== 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           125.7804071 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              220.000 ppm
F1             27671.69 Hz
F2P              -10.000 ppm
F2             -1257.80 Hz
PPMCM            10.08772 ppm/cm
HZCM          1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-189-1HNMR
EXPNO 1
PROCNO 1

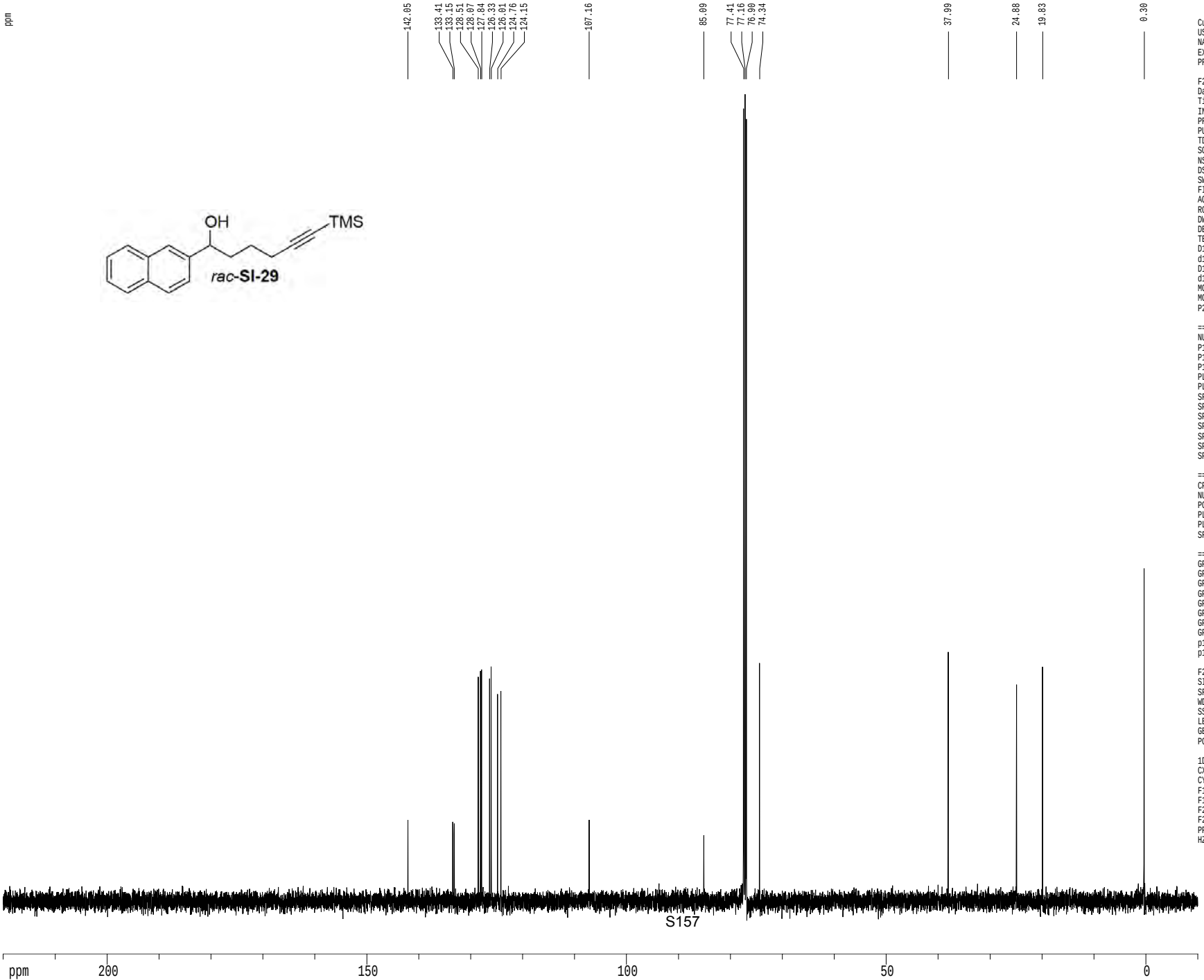
F2 - Acquisition Parameters
Date_ 20130915
Time 17.44
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 11.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200316 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-189-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20130915
Time             17.48
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT         CDCl3T
NS                 210
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

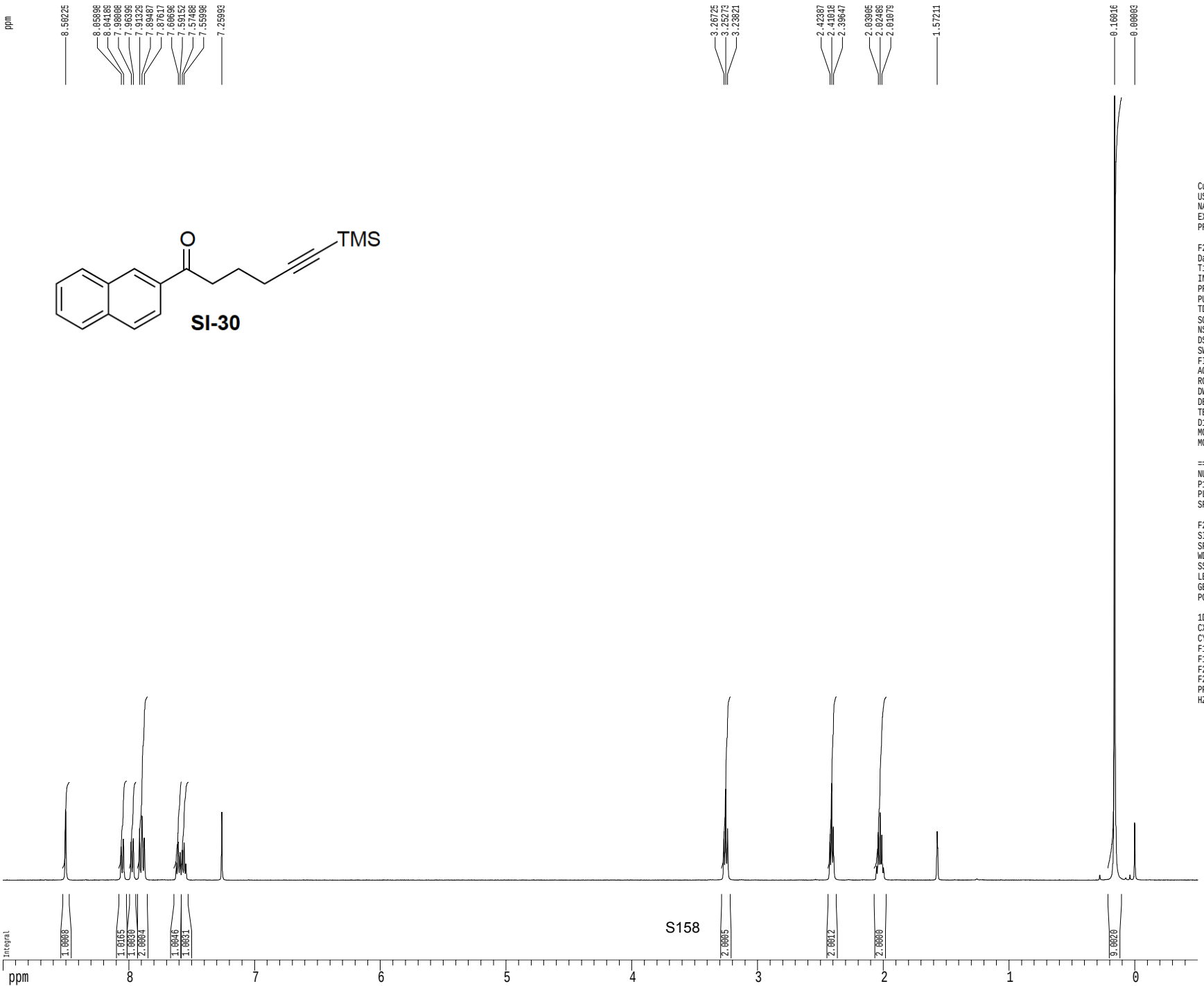
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804076 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            220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER            mharri
NAME      MRH-V-160-1HNMR
EXPNO                1
PROCNO               1

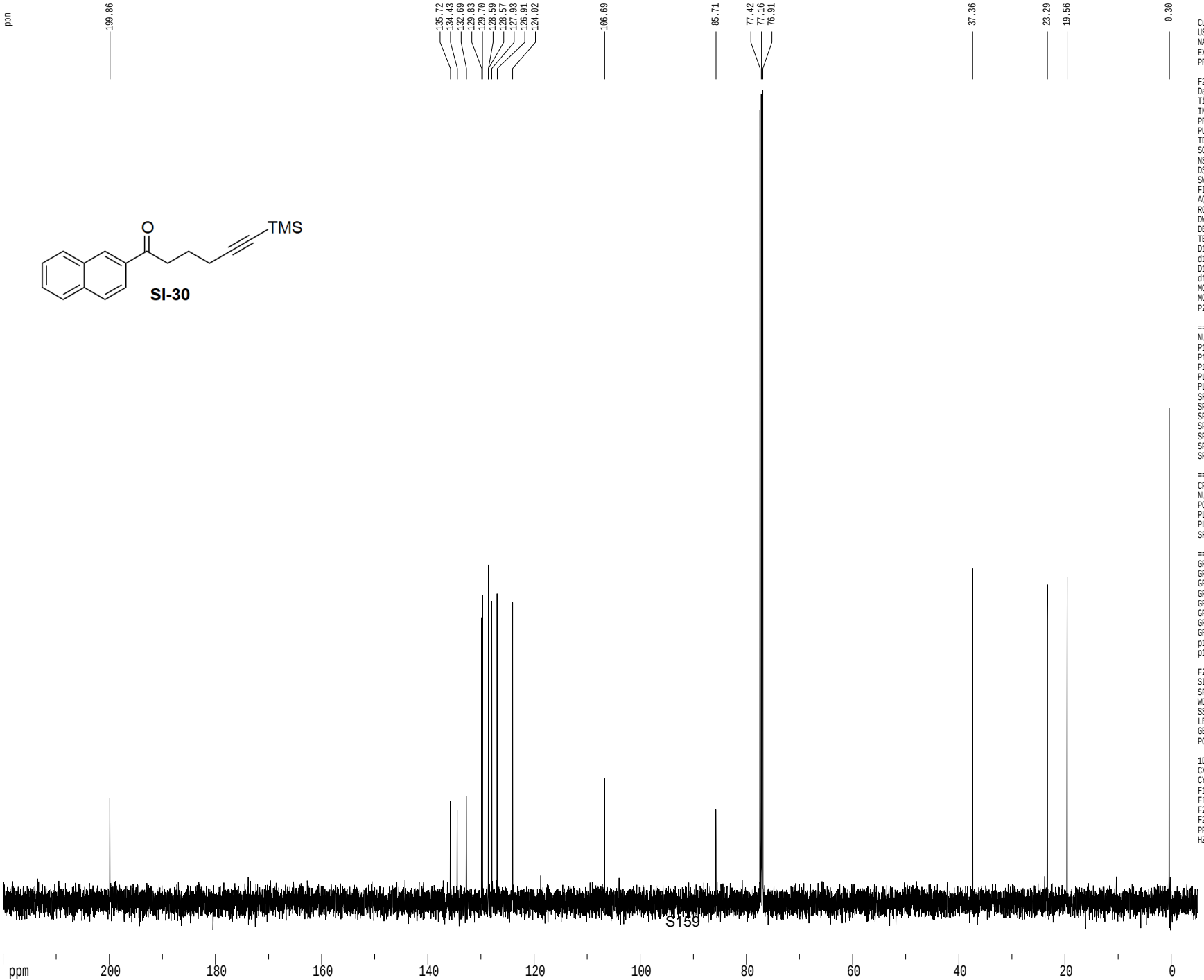
F2 - Acquisition Parameters
Date_          20131209
Time              11.55
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    2
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                  7.1
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          500.2200309 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-160-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131209
Time              11.56
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   71
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG               9195.2
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

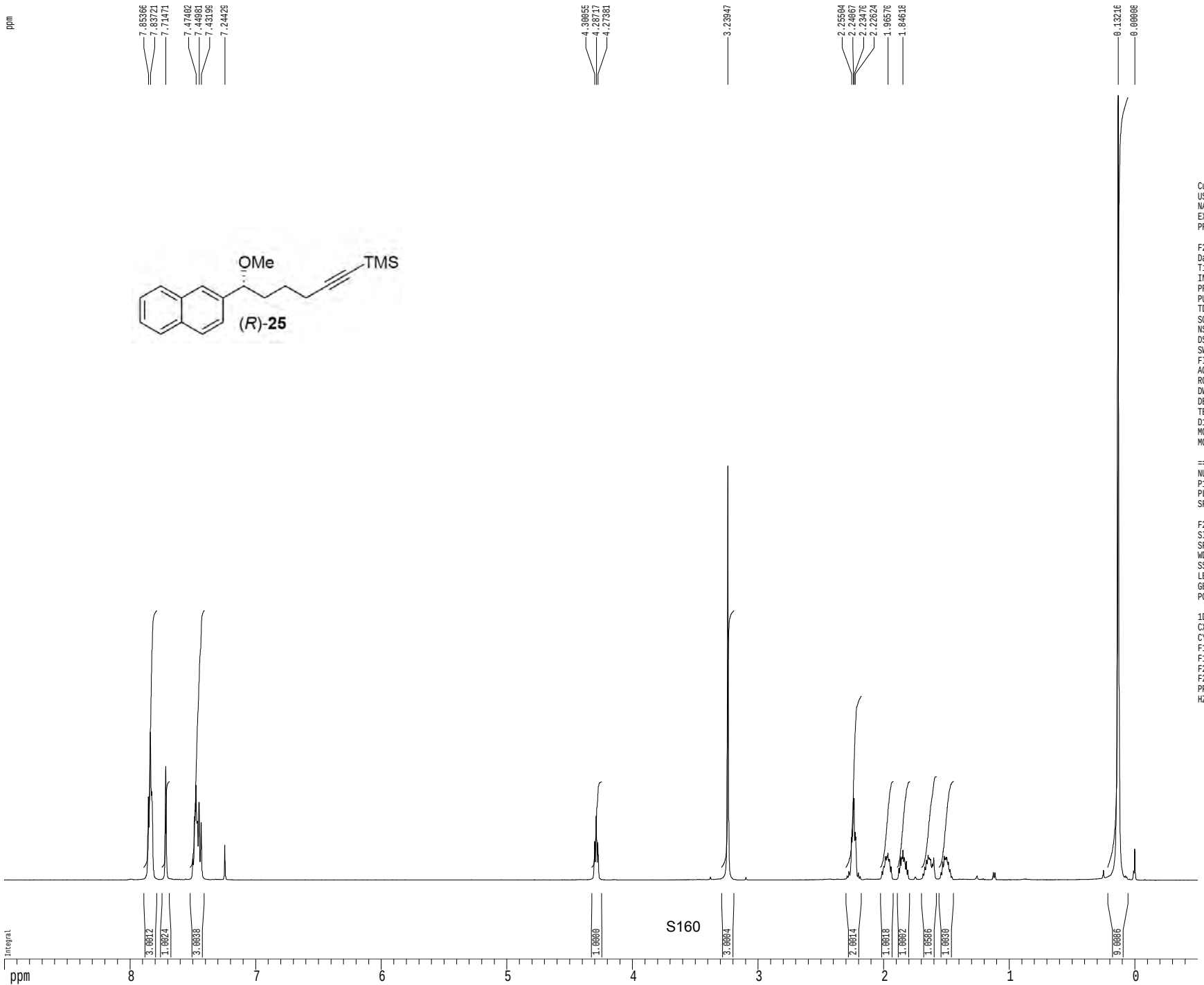
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804080 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            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-192-1HNMR
EXPNO 1
PROCNO 1

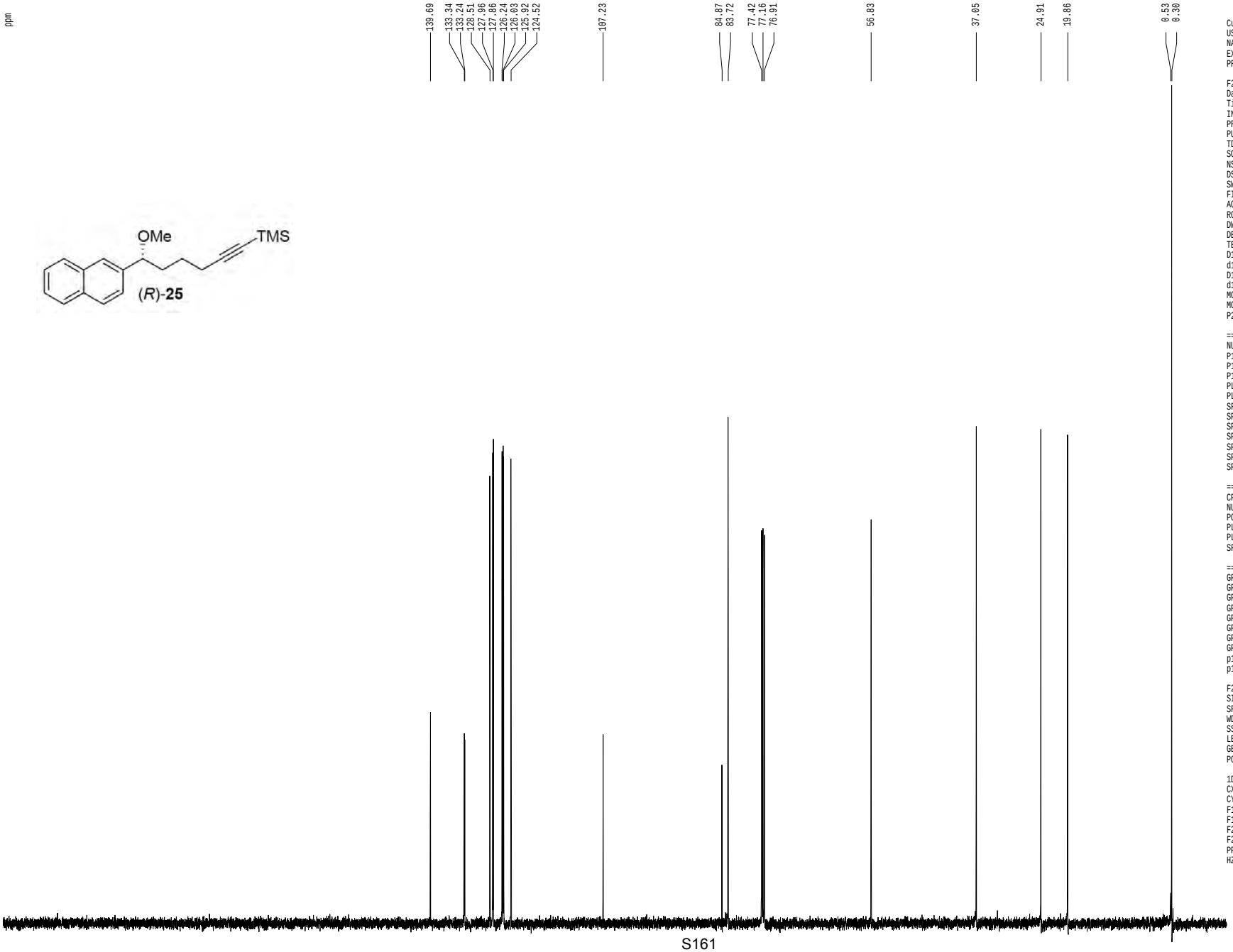
F2 - Acquisition Parameters
Date_ 20131025
Time 17.13
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200390 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-192-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131025
Time              17.16
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  101
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

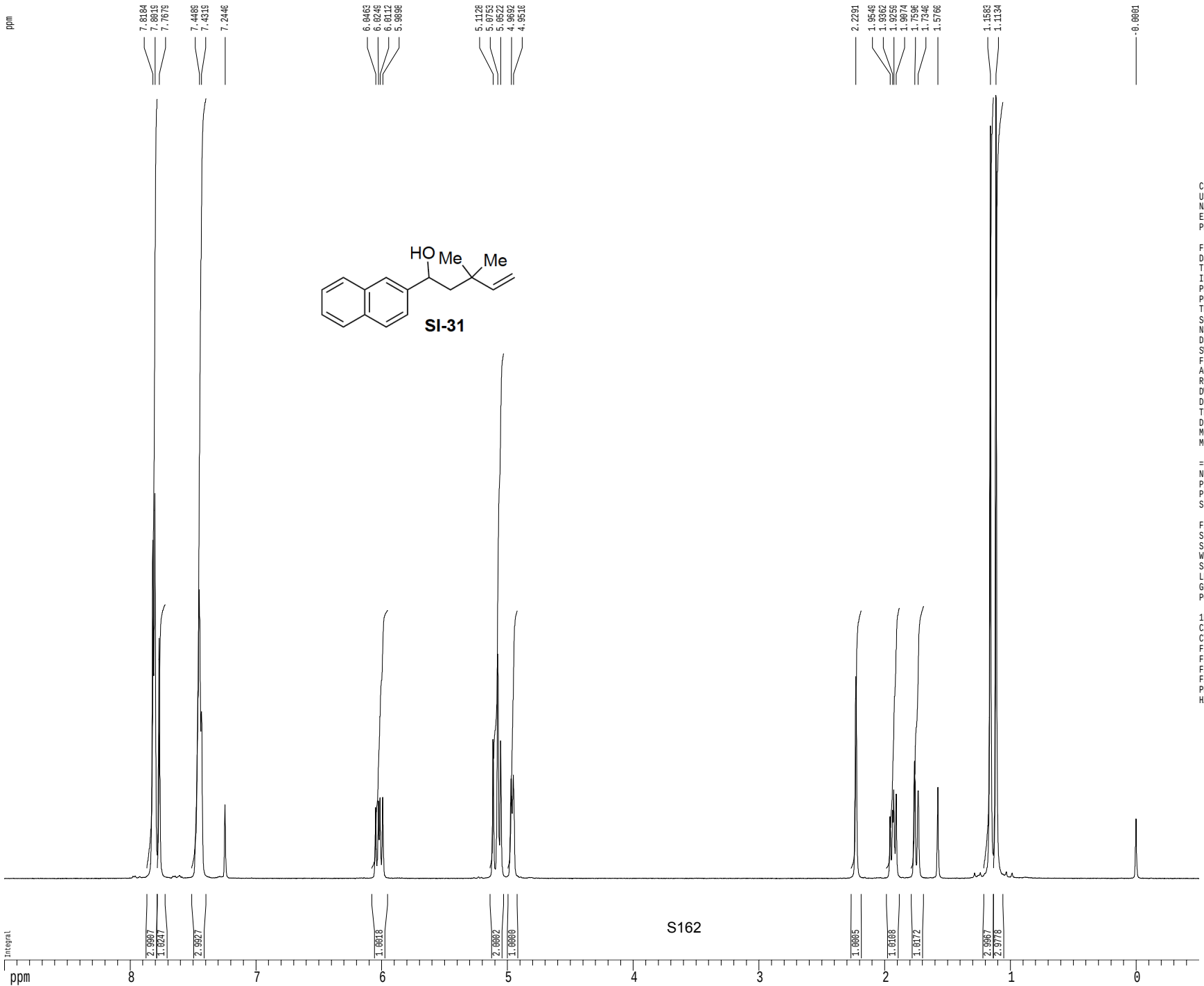
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804108 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             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-86-1HNMR
EXPNO 1
PROCNO 1

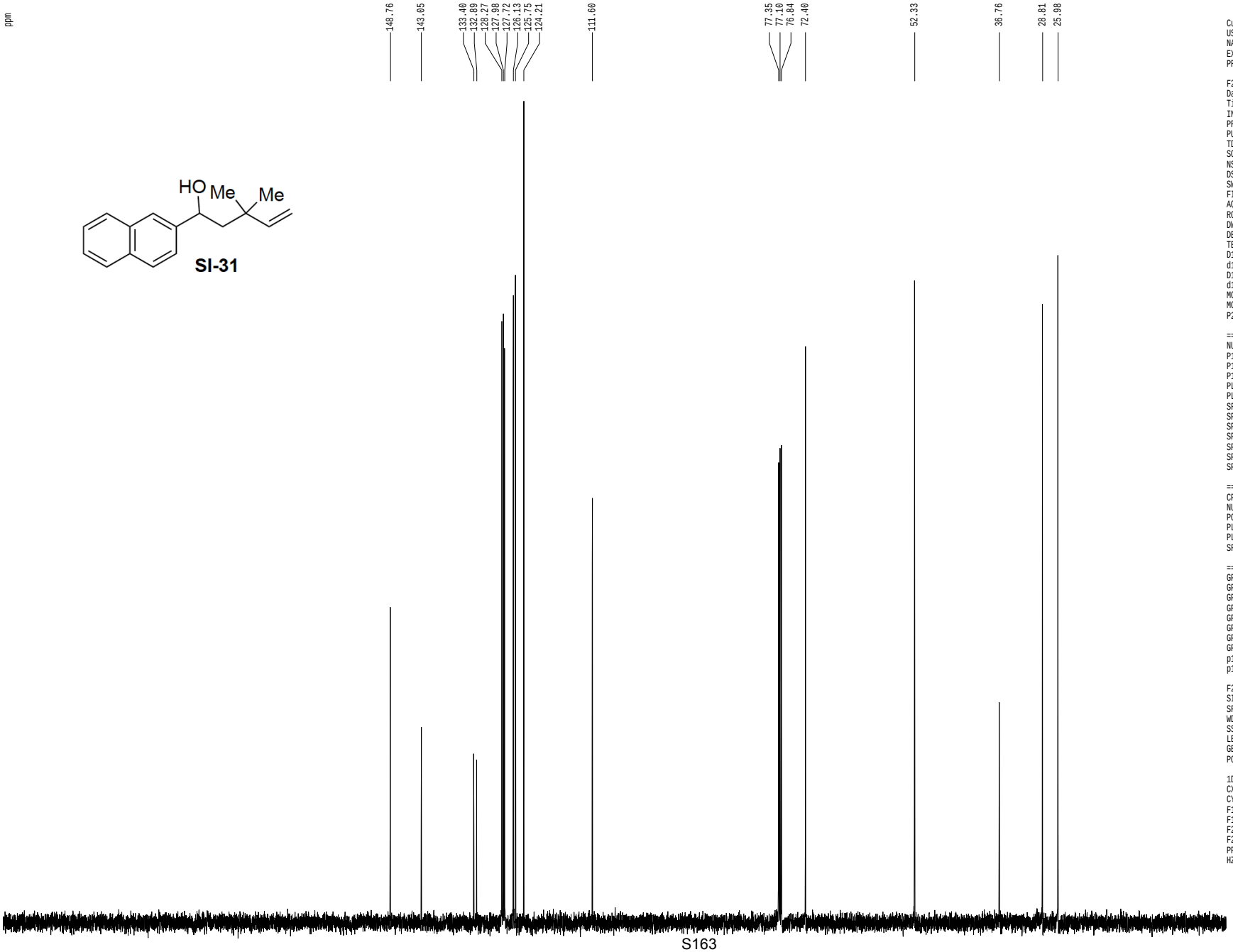
F2 - Acquisition Parameters
Date_ 20140301
Time 6.09
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200384 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-86-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140301
Time               6.12
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  133
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG                1824.6
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                 31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 3.20 dB
SP2                 3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1              0.00 Hz
SPOFF2              0.00 Hz

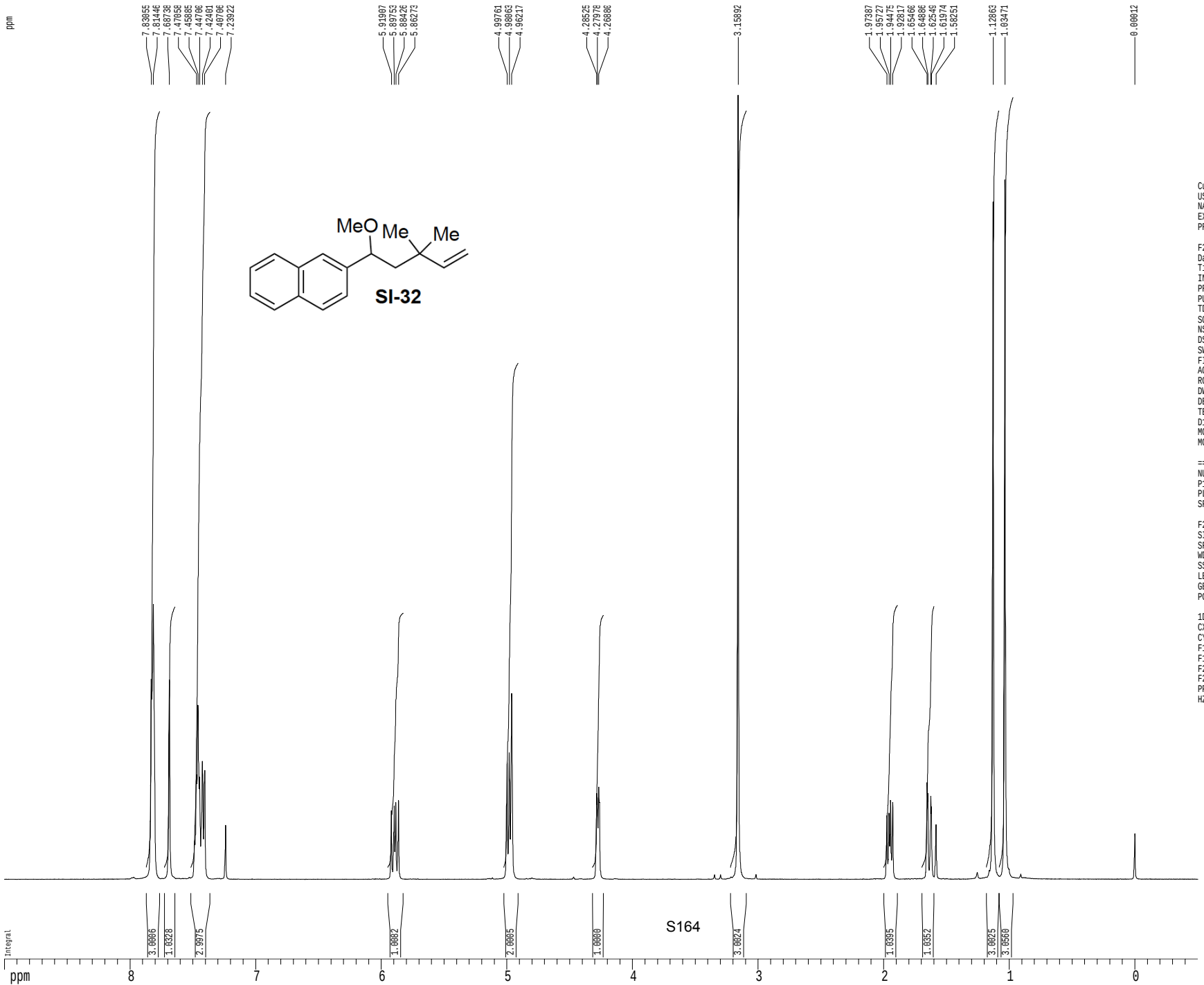
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== 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                   2.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.65 cm
F1P              220.000 ppm
F1             27671.69 Hz
F2P               -5.000 ppm
F2              -628.90 Hz
PPMCM            9.86842 ppm/cm
HZCM          1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-87-1HNMR
EXPNO 1
PROCNO 1

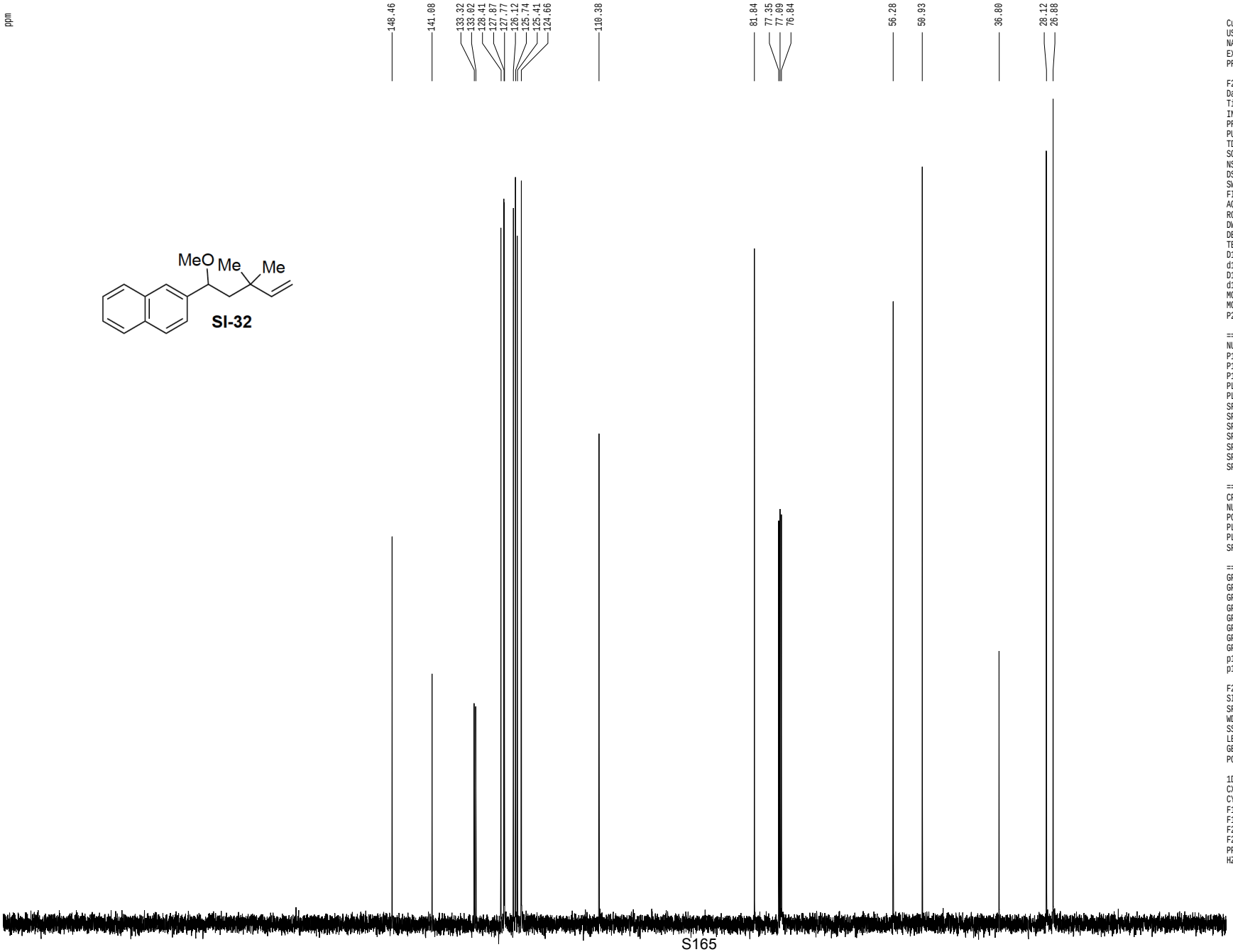
F2 - Acquisition Parameters
Date_ 20140301
Time 6.16
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200403 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME      MRH-VI-87-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140301
Time               6.19
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   95
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG               3649.1
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

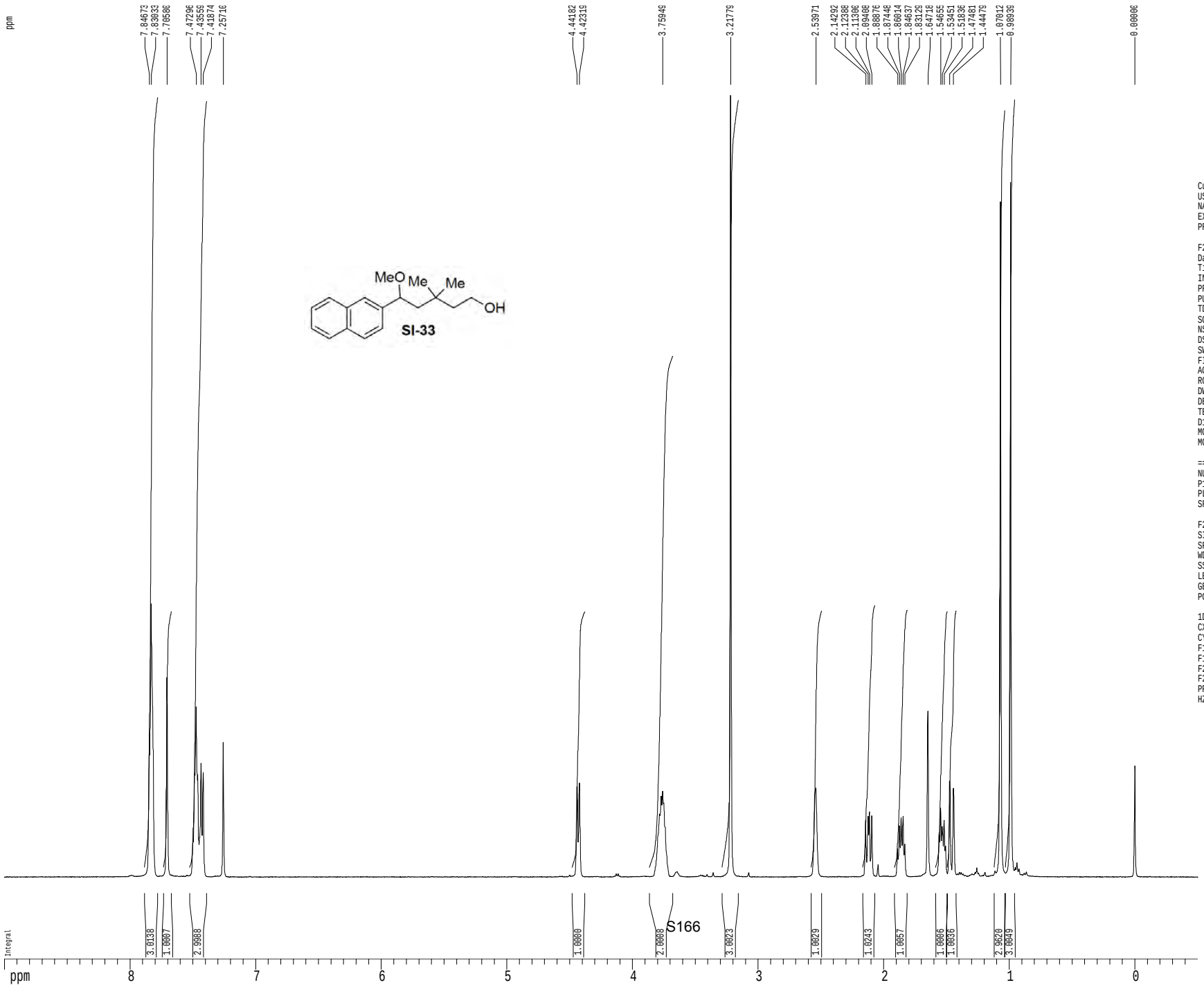
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P             220.000 ppm
F1             27671.69 Hz
F2P              -5.000 ppm
F2              -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME      MRH-VI-88-1HNMR
EXPNO                   1
PROCNO                  1

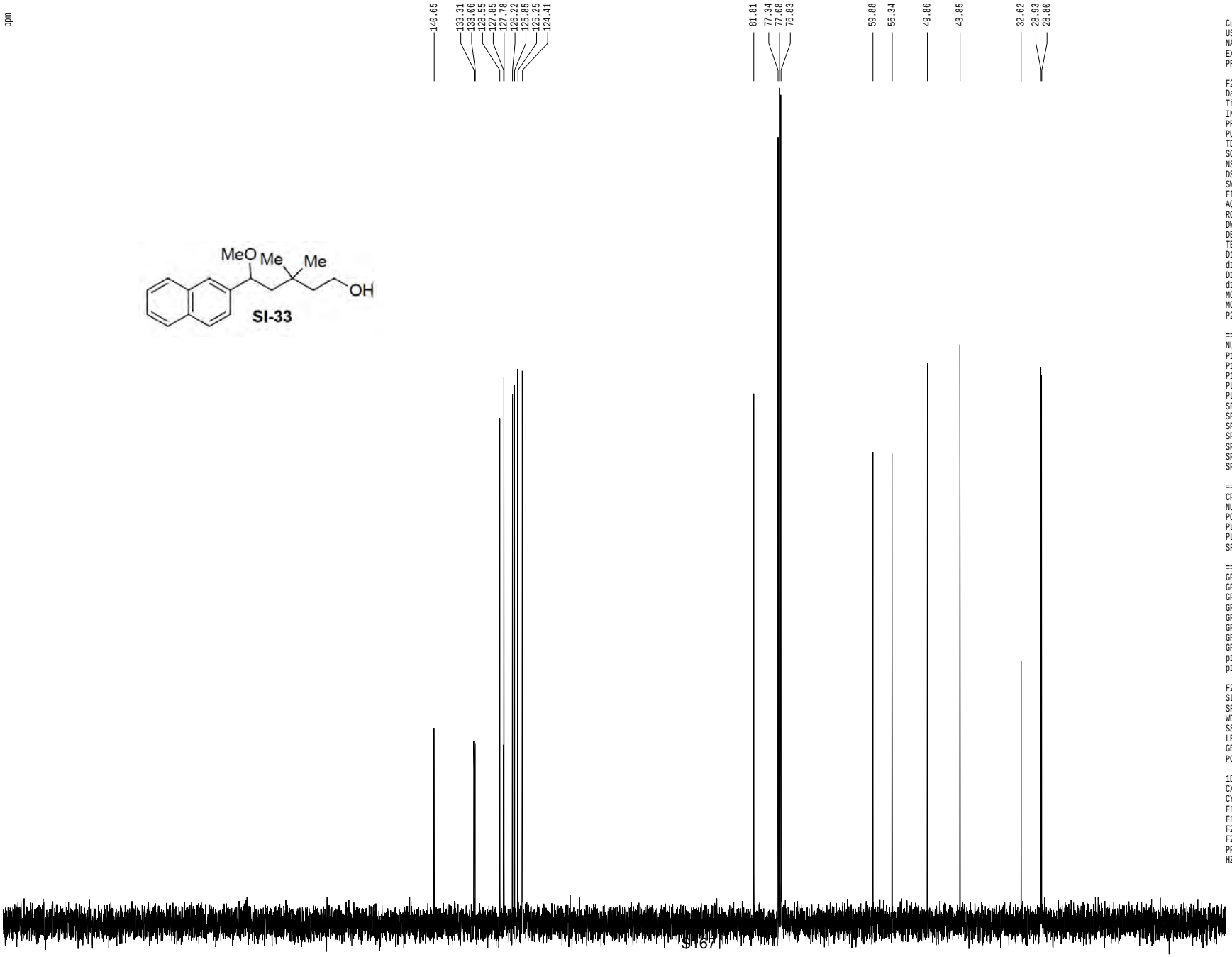
F2 - Acquisition Parameters
Date_           20140301
Time               6.22
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    2
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                    9
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          500.2200315 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              9.000 ppm
F1             4501.98 Hz
F2P             -0.500 ppm
F2            -250.11 Hz
PPMCM          0.41667 ppm/cm
HZCM         208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME      MRH-VI-88-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140301
Time               6.25
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  113
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

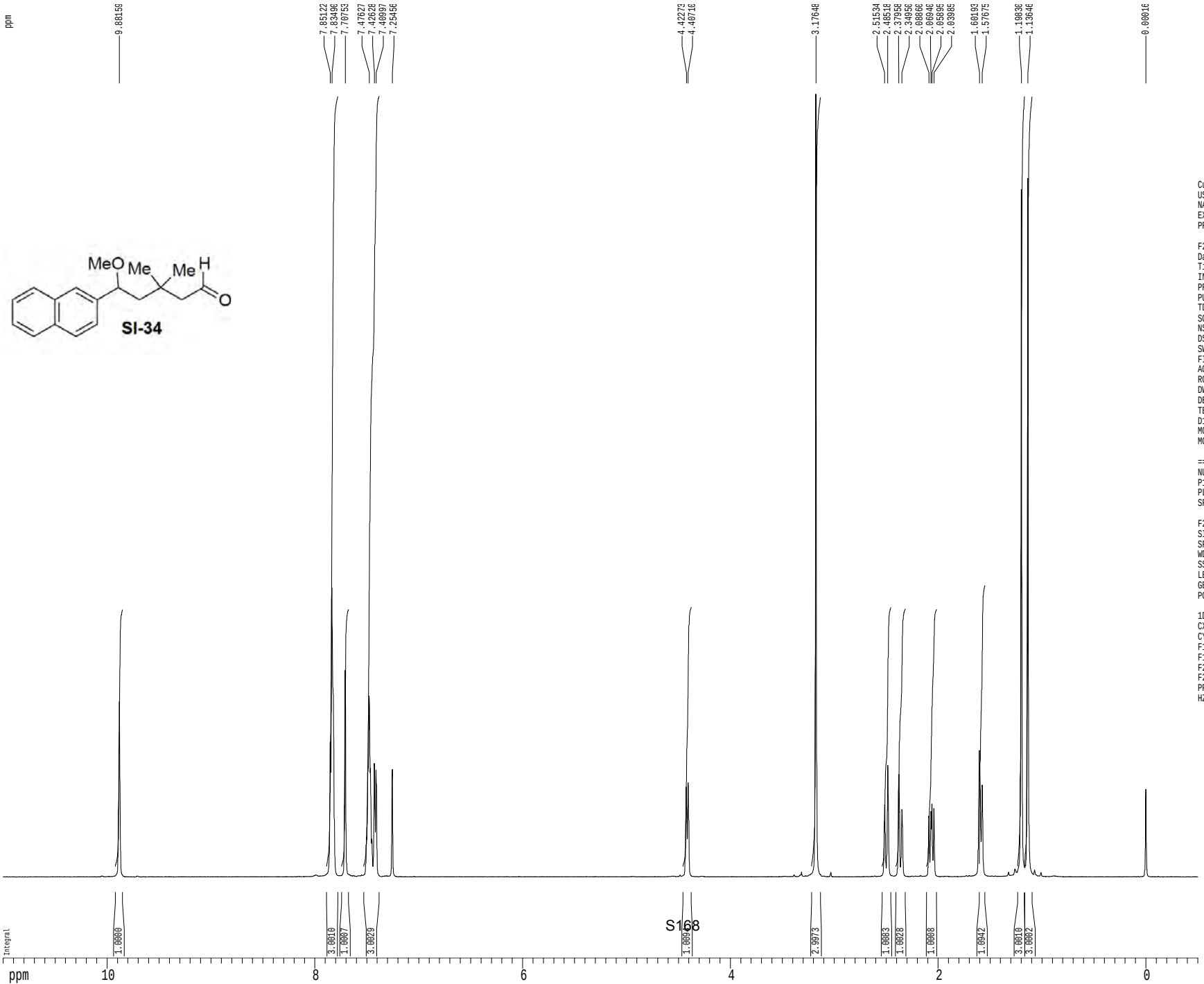
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-89-1HNMR
EXPNO 1
PROCNO 1

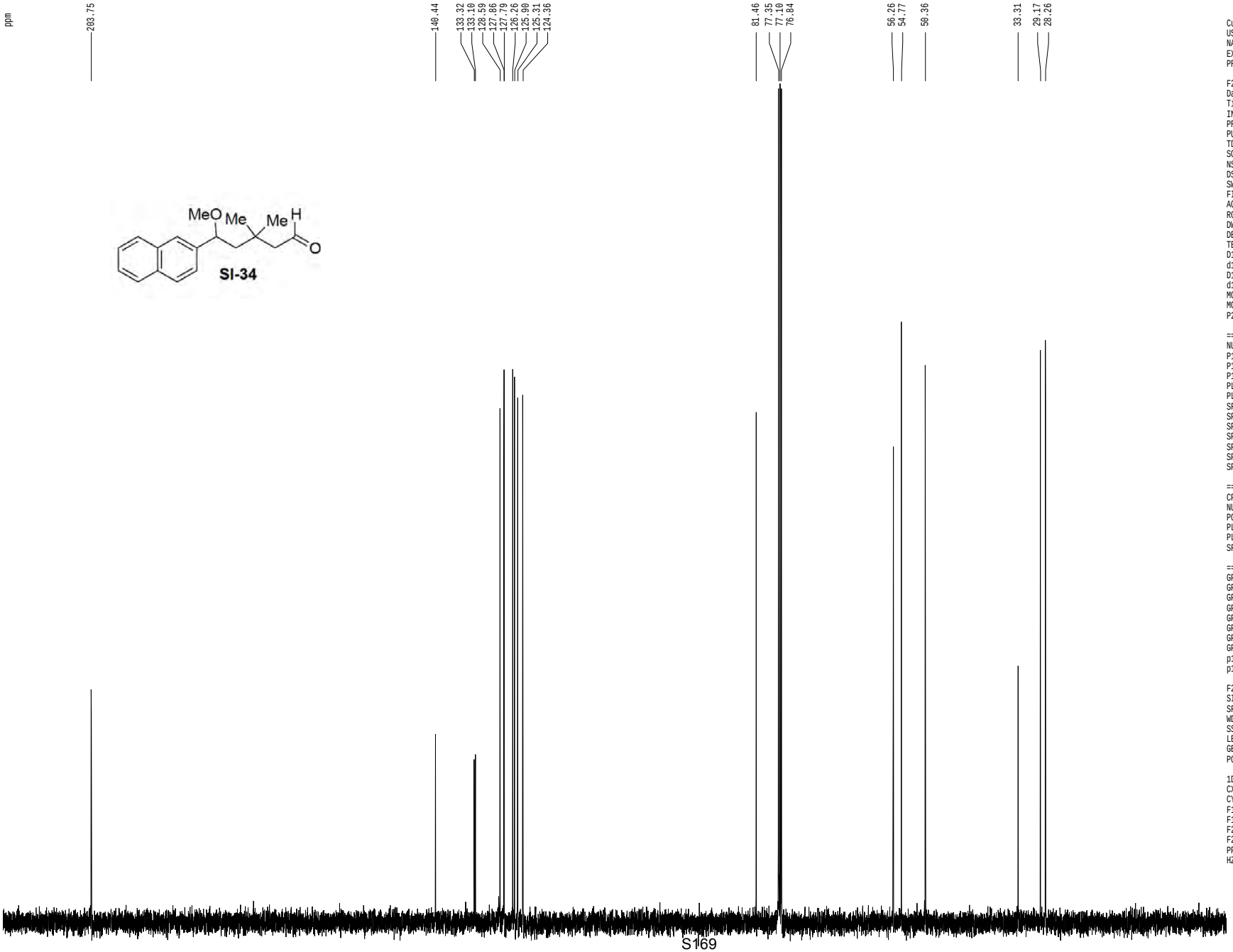
F2 - Acquisition Parameters
Date_ 20140301
Time 6.28
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 10.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200331 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 11.000 ppm
F1 5502.42 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.50439 ppm/cm
HZCM 252.30397 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-89-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140301
Time               6.32
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                  132
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG               4597.6
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

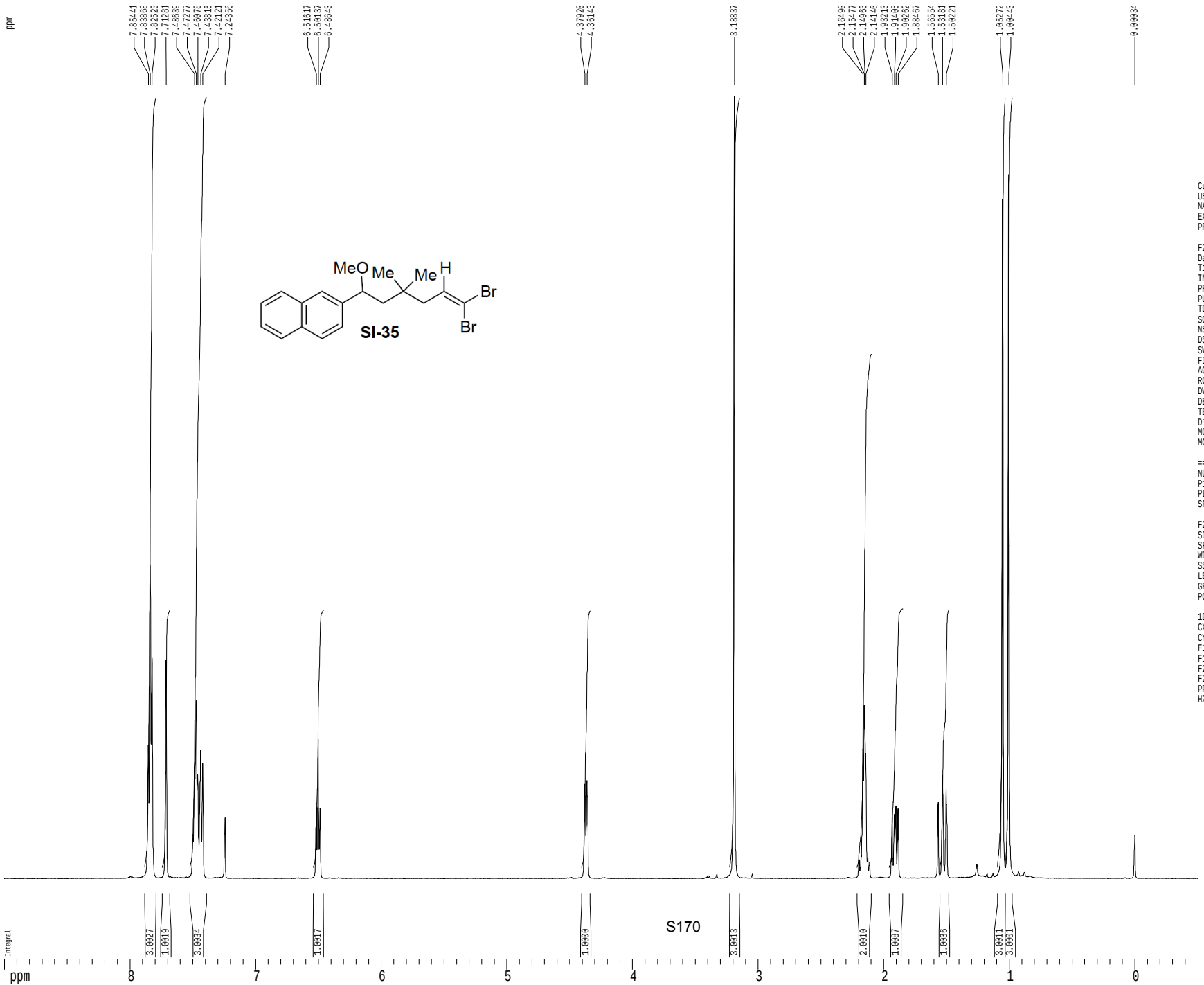
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-94-1HNMR
EXPNO 1
PROCNO 1

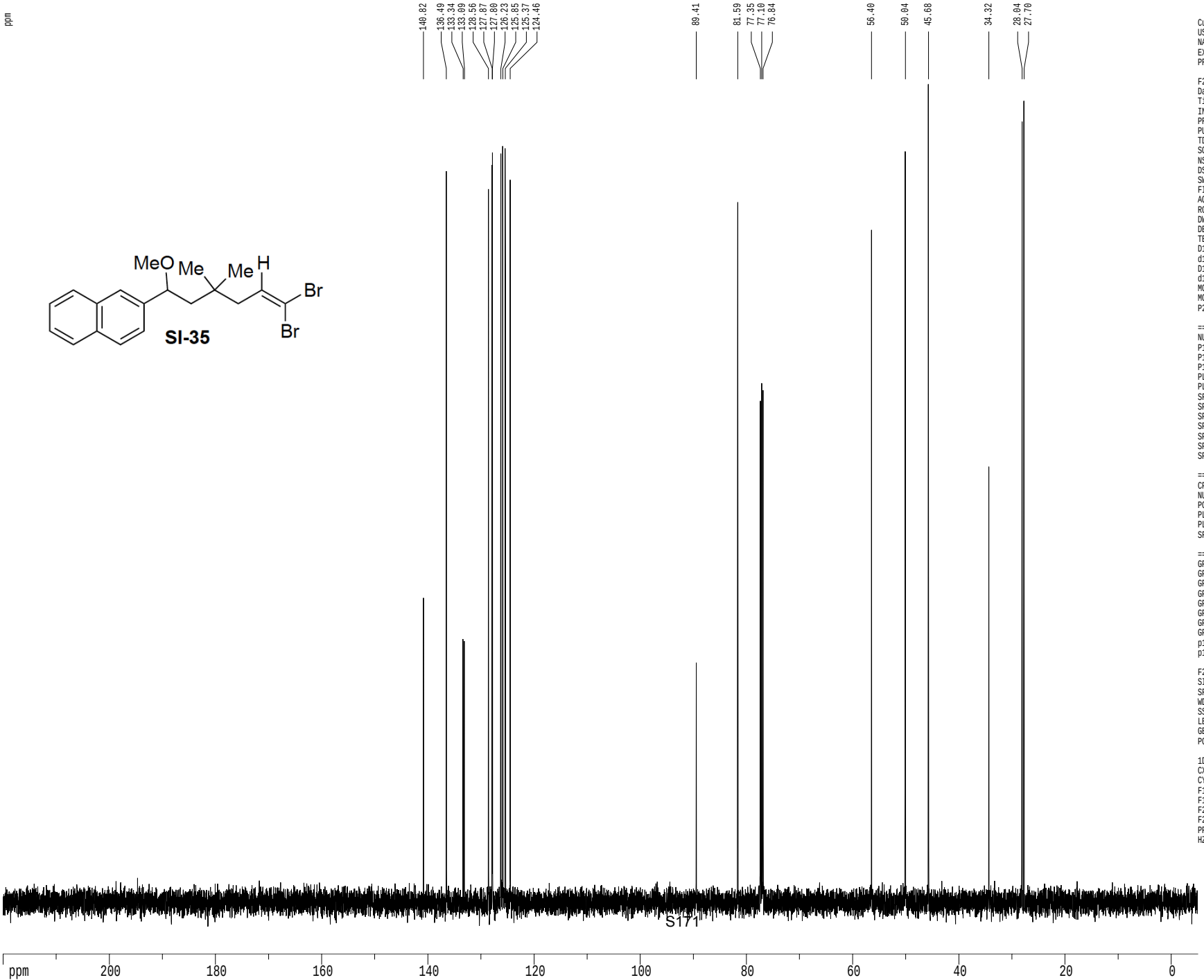
F2 - Acquisition Parameters
Date_ 20140303
Time 14.53
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200377 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-94-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140303
Time              14.55
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   91
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ           1.0813940 sec
RG             14596.5
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

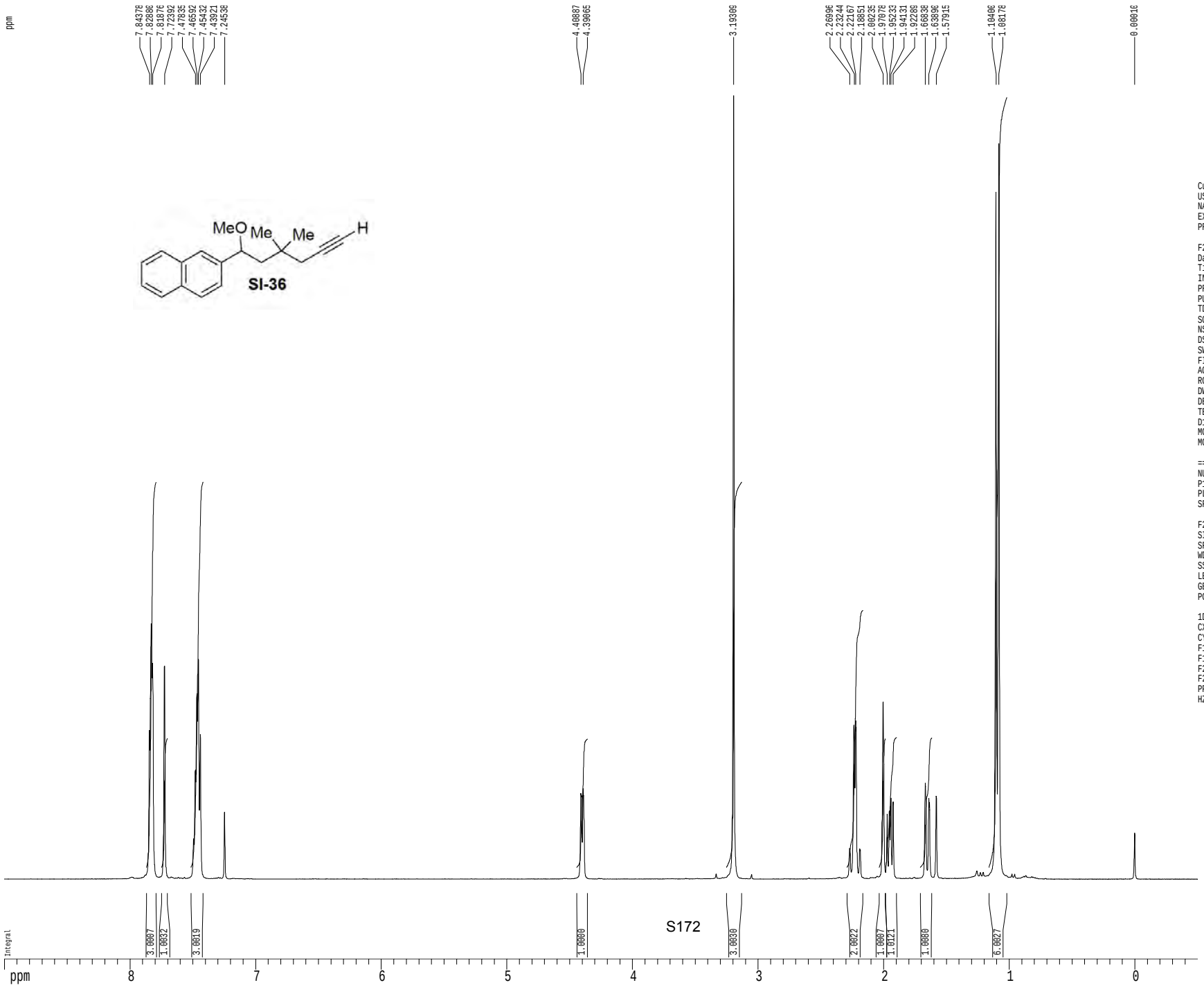
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-95-1HNMR
EXPNO 1
PROCNO 1

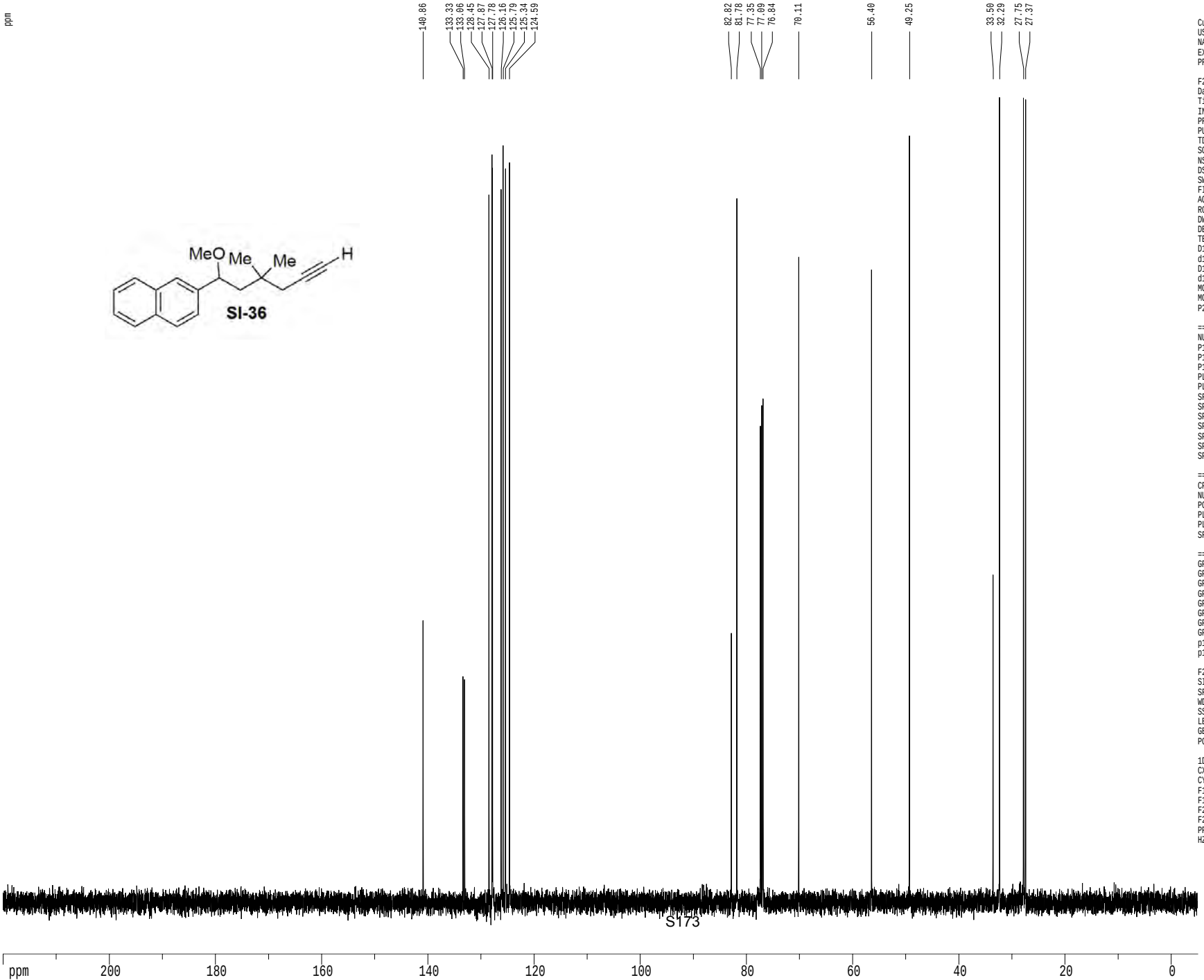
F2 - Acquisition Parameters
Date_ 20140303
Time 14.58
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 9
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200376 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME      MRH-VI-95-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140303
Time              15.01
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   98
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ            1.0813940 sec
RG               3649.1
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

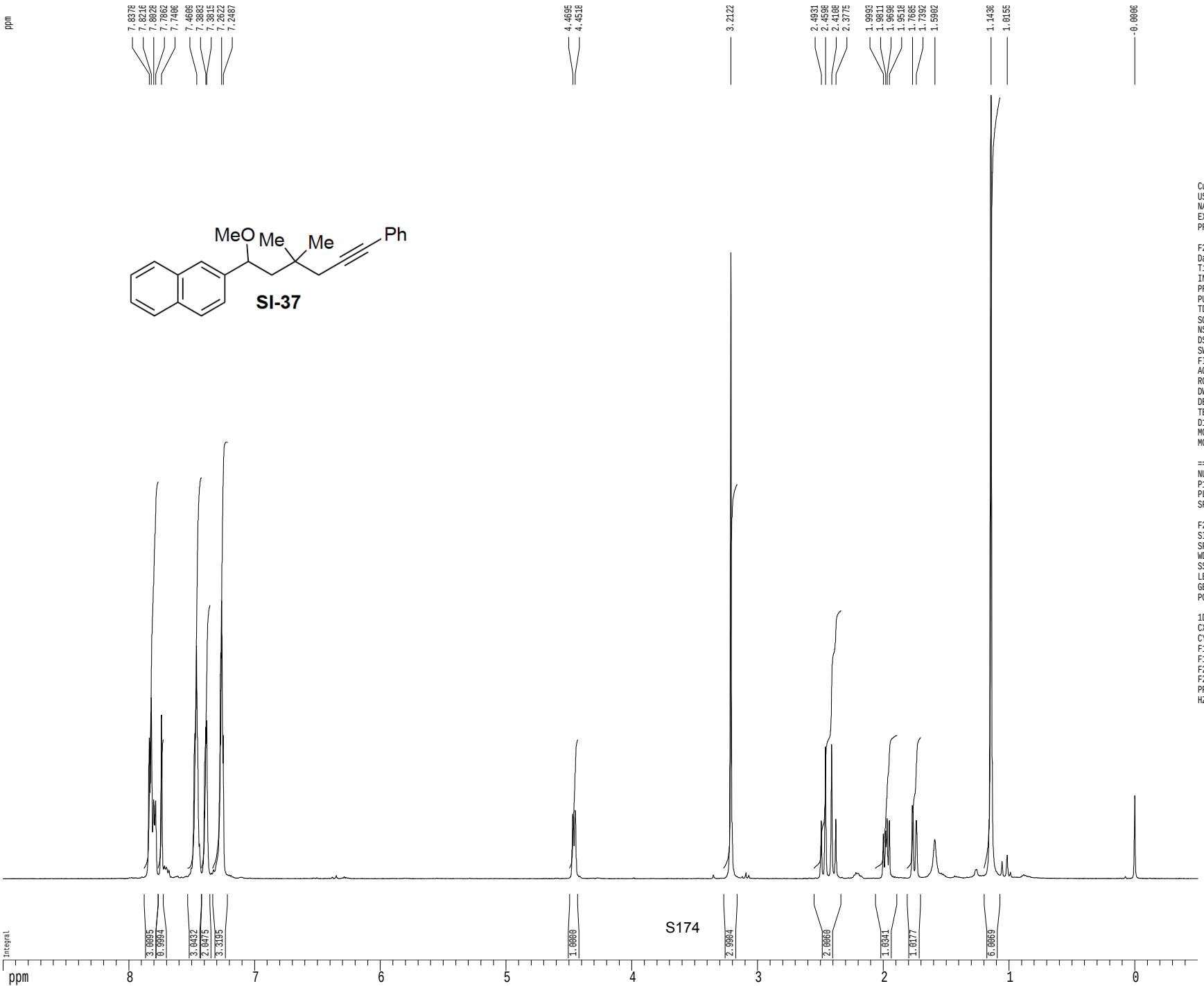
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME      MRH-VI-96-1HNMR
EXPNO                   1
PROCNO                  1

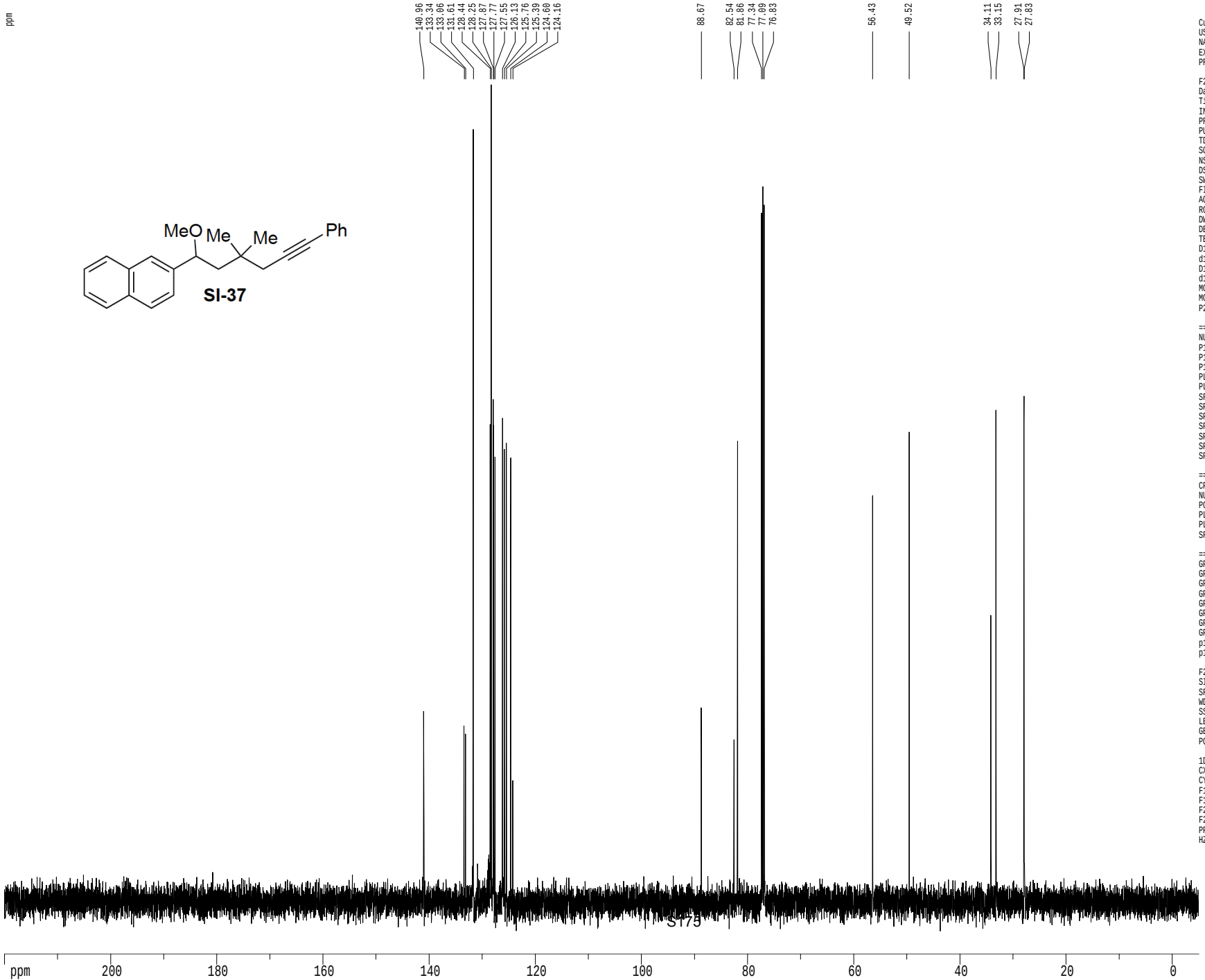
F2 - Acquisition Parameters
Date_           20140303
Time              15.05
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    4
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                    9
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          500.2200359 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               9.000 ppm
F1             4501.98 Hz
F2P             -0.500 ppm
F2             -250.11 Hz
PPMCM          0.41667 ppm/cm
HZCM         208.42502 Hz/cm
```

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-96-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140303
Time              15.07
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                  109
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG                16384
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

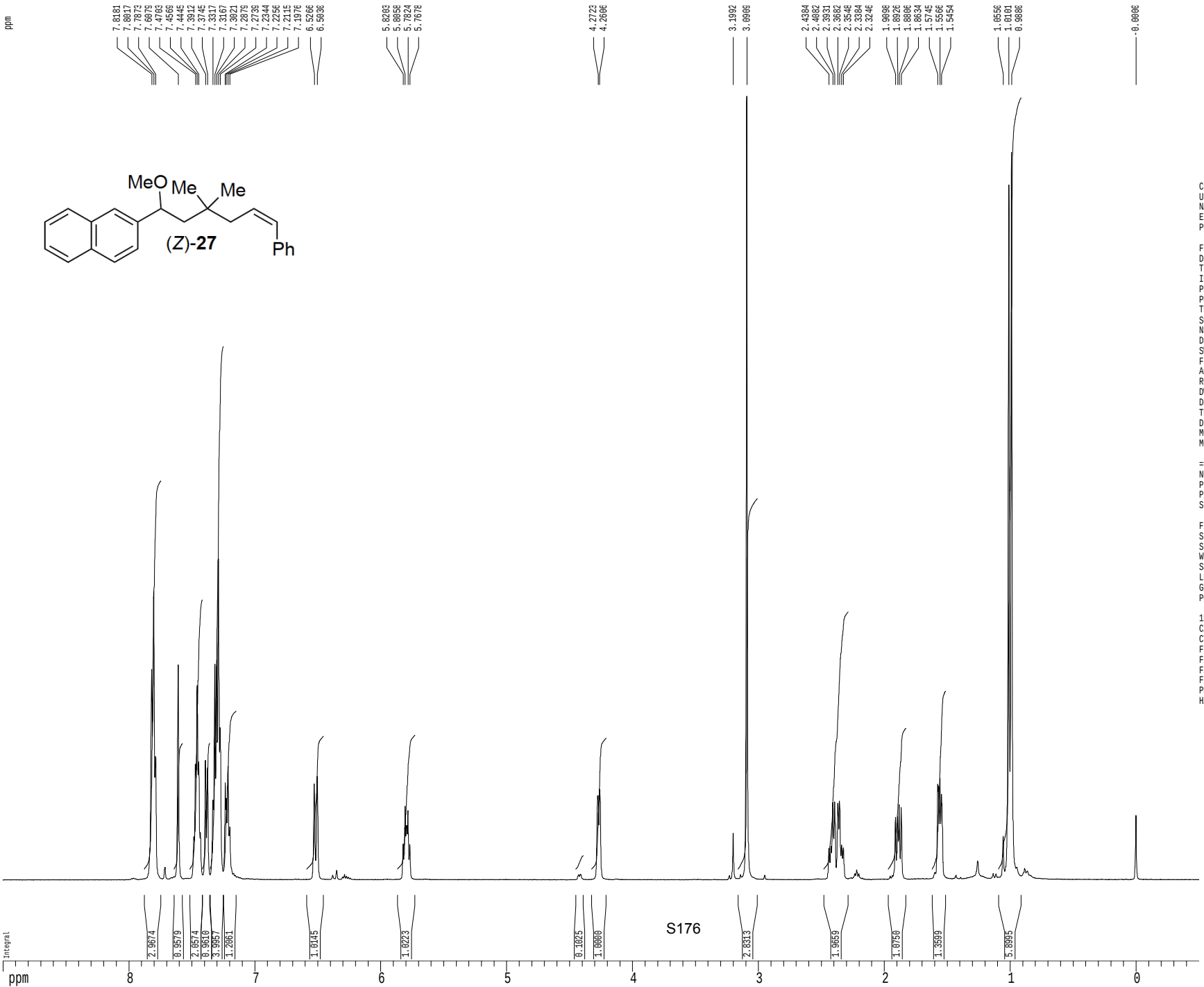
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-105-1HNMR
EXPNO                  1
PROCNO                 1

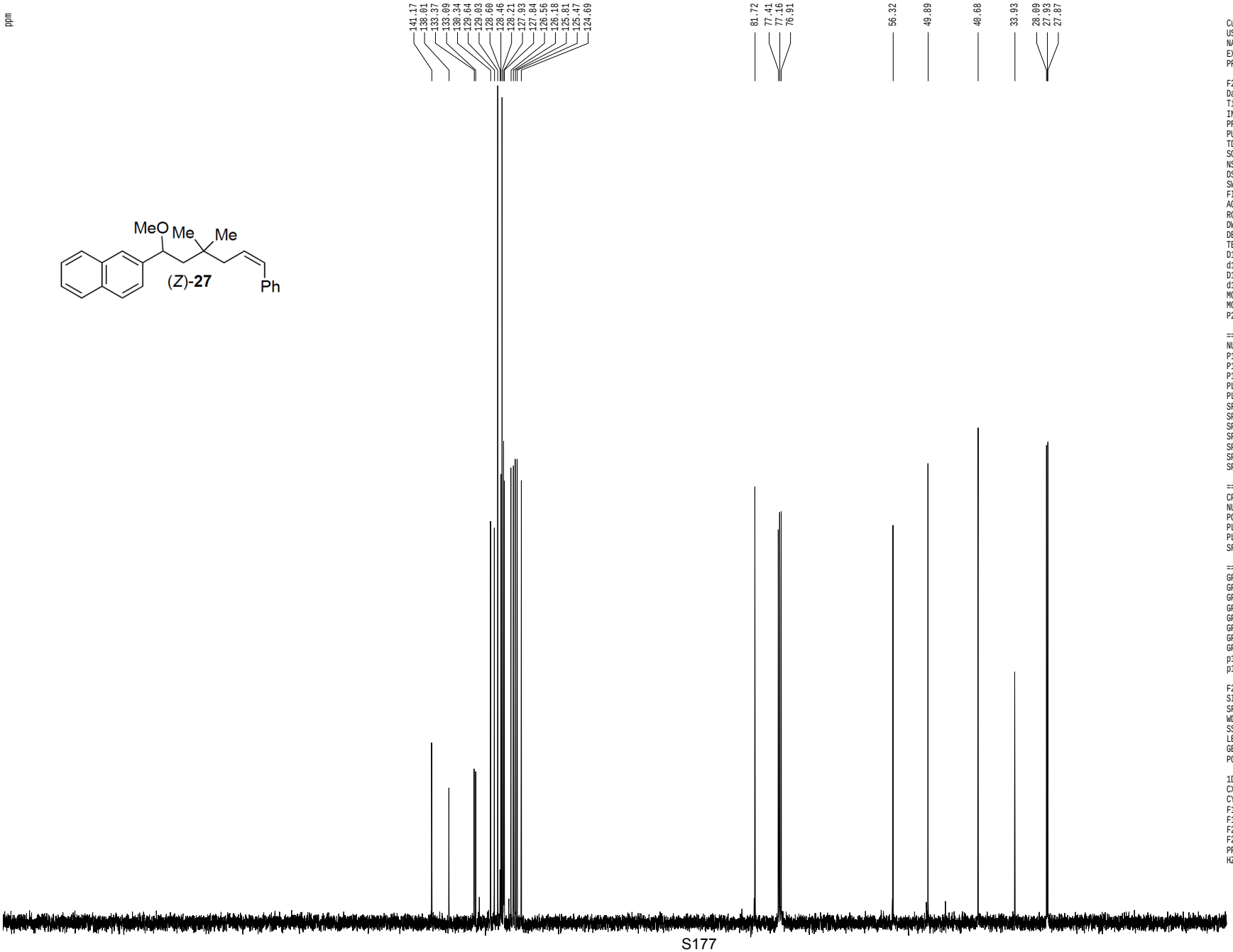
F2 - Acquisition Parameters
Date_           20140303
Time              11.35
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    3
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                  6.3
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          500.2200426 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-105-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140303
Time              11.39
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                  147
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

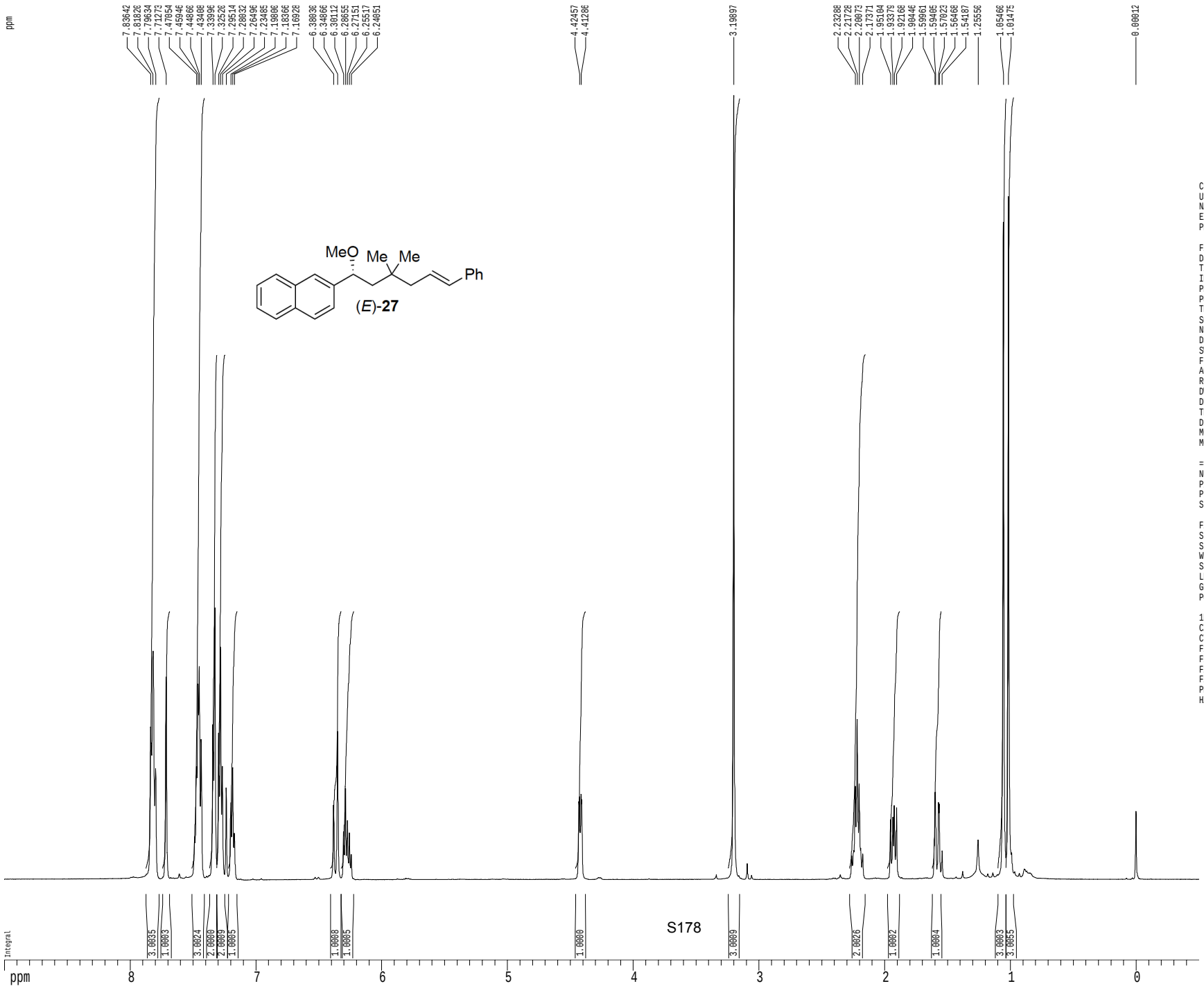
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804113 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             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



S178

```
Current Data Parameters
USER              mharri
NAME      MRH-VI-73-1HNMR
EXPNO                   1
PROCNO                  1

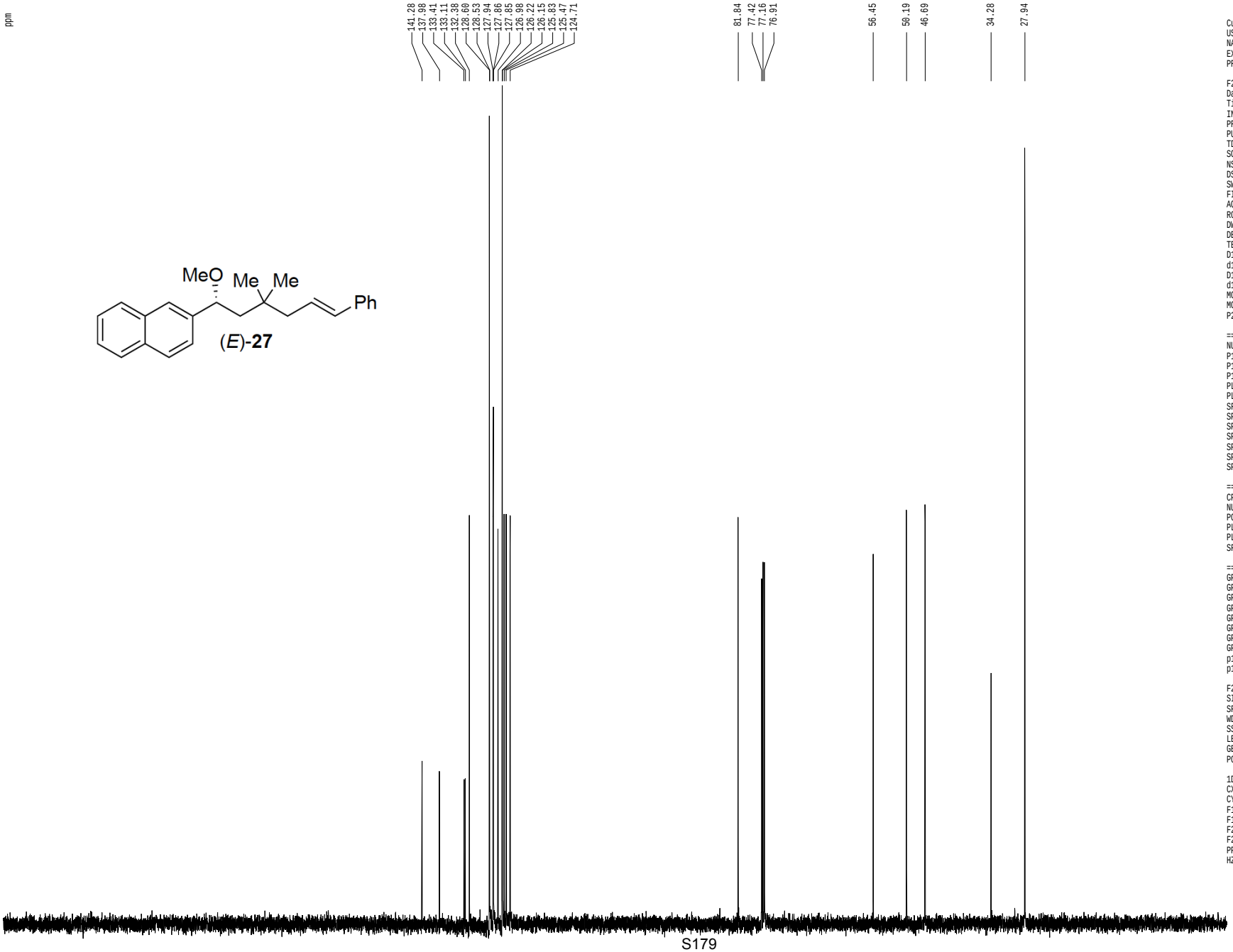
F2 - Acquisition Parameters
Date_           20140116
Time              17.20
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                    8
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          500.2200420 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER            mharri
NAME      MRH-VI-73-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140116
Time              17.23
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                  131
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

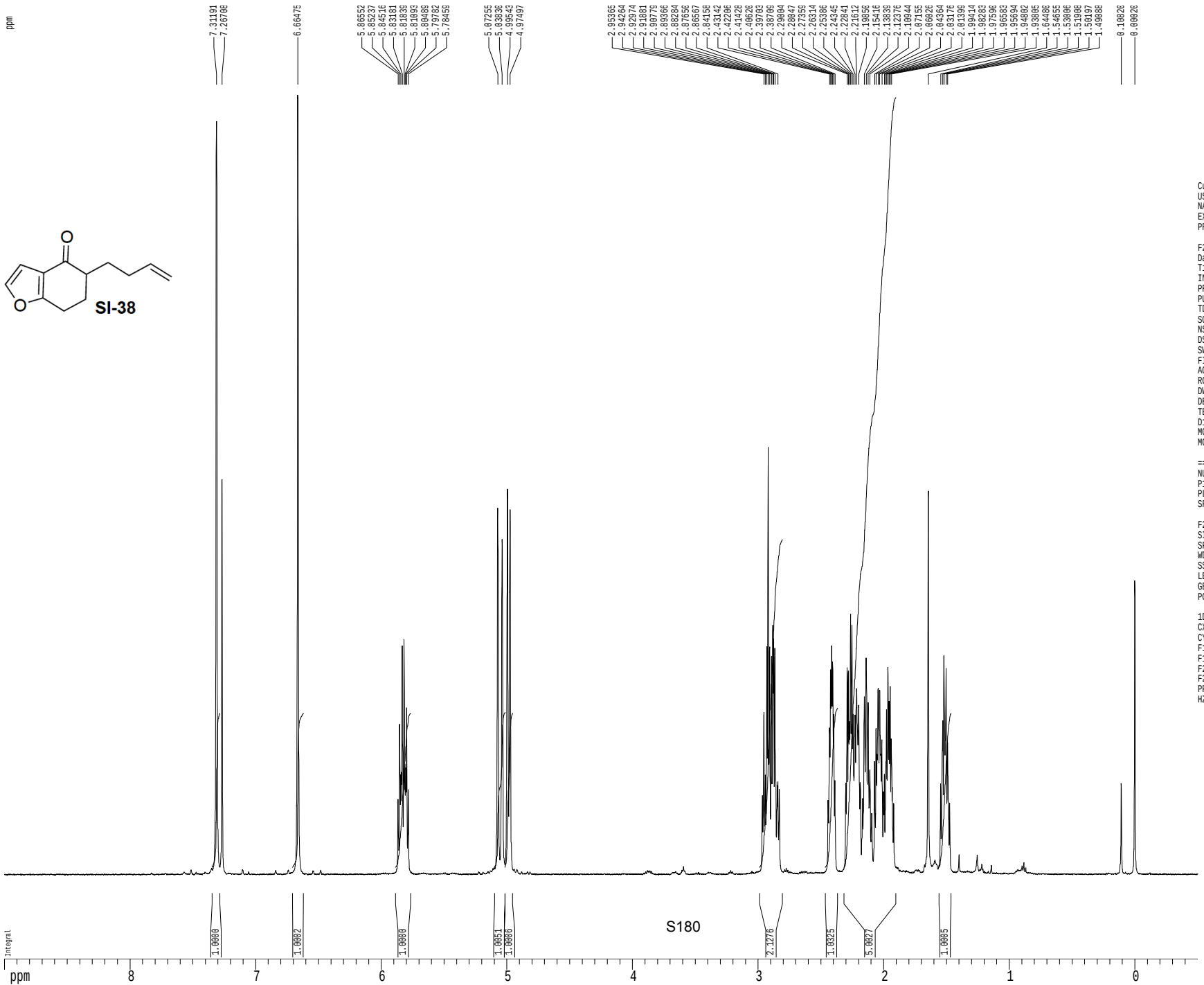
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804108 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            220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-244-1HNMR
EXPNO 1
PROCNO 1

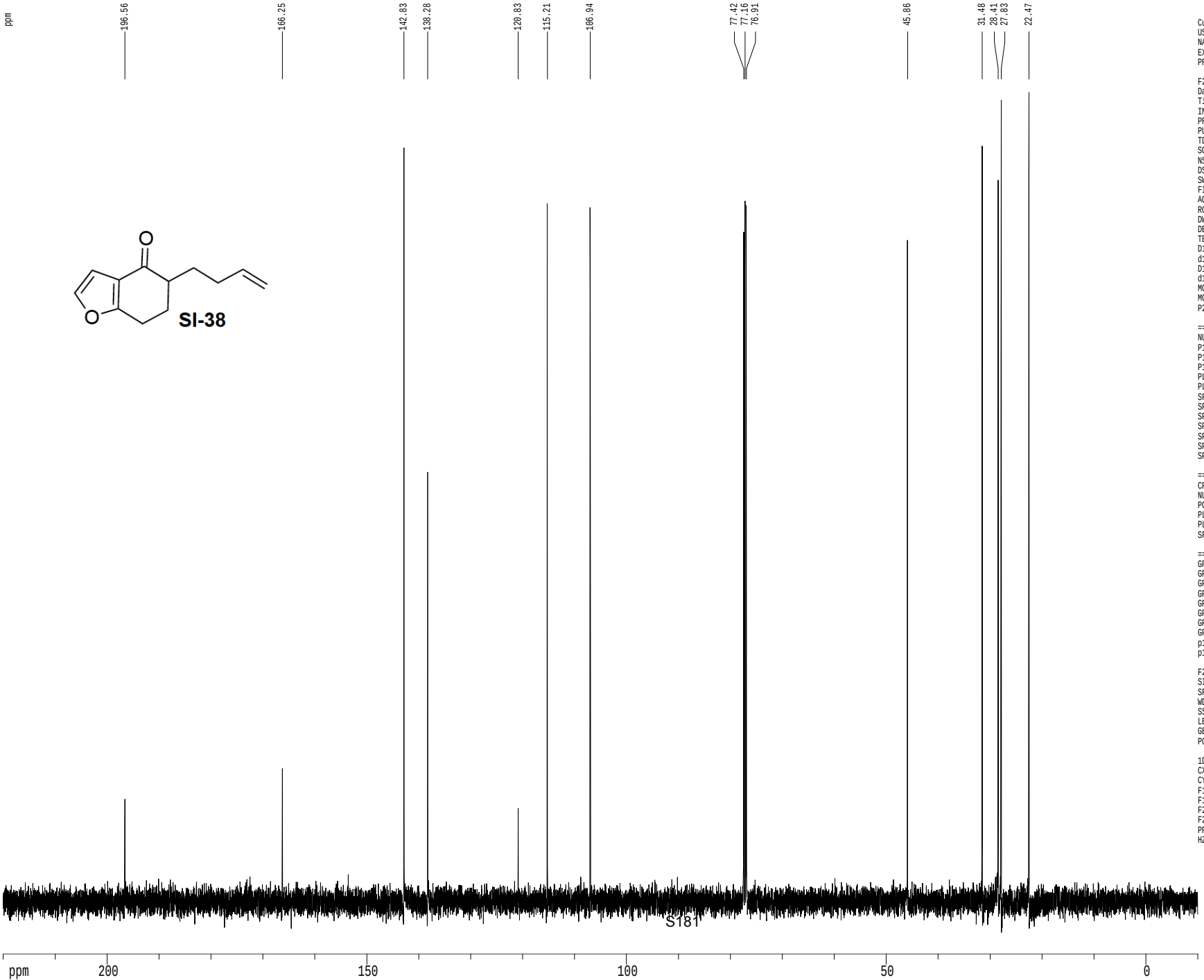
F2 - Acquisition Parameters
Date_ 20131210
Time 15.48
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200272 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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



Current Data Parameters
USER mharri
NAME MRH-V-244-13CNMR
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20131210
Time 15.51
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 108
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 31.00 usec

======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

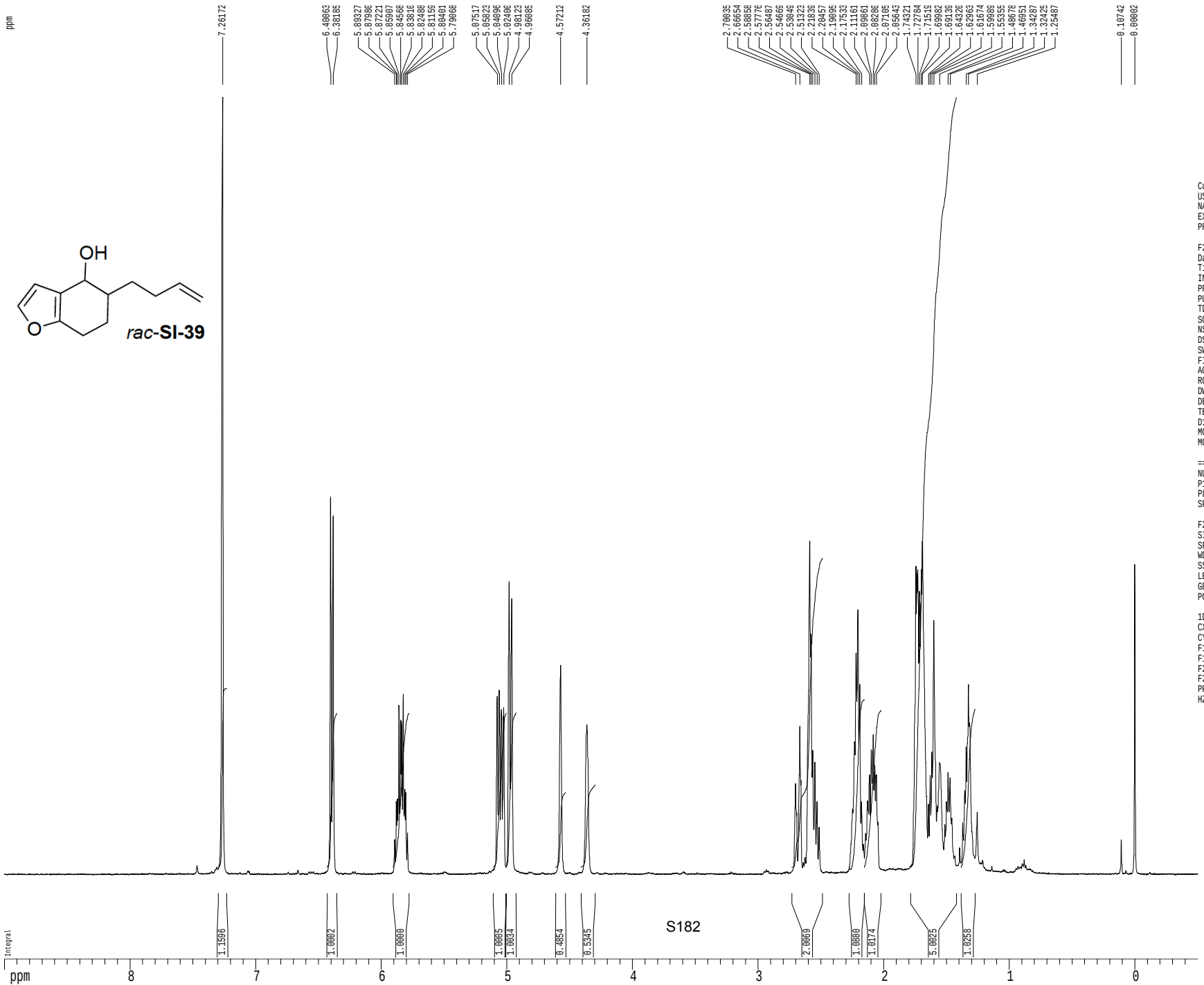
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== 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 125.7804085 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 220.000 ppm
F1 27671.69 Hz
F2P -10.000 ppm
F2 -1257.80 Hz
PPMCM 10.08772 ppm/cm
HZCM 1268.83752 Hz/cm

1H spectrum




```
Current Data Parameters
USER              mharri
NAME      MRH-V-245-1HNMR
EXPNO                  1
PROCNO                 1

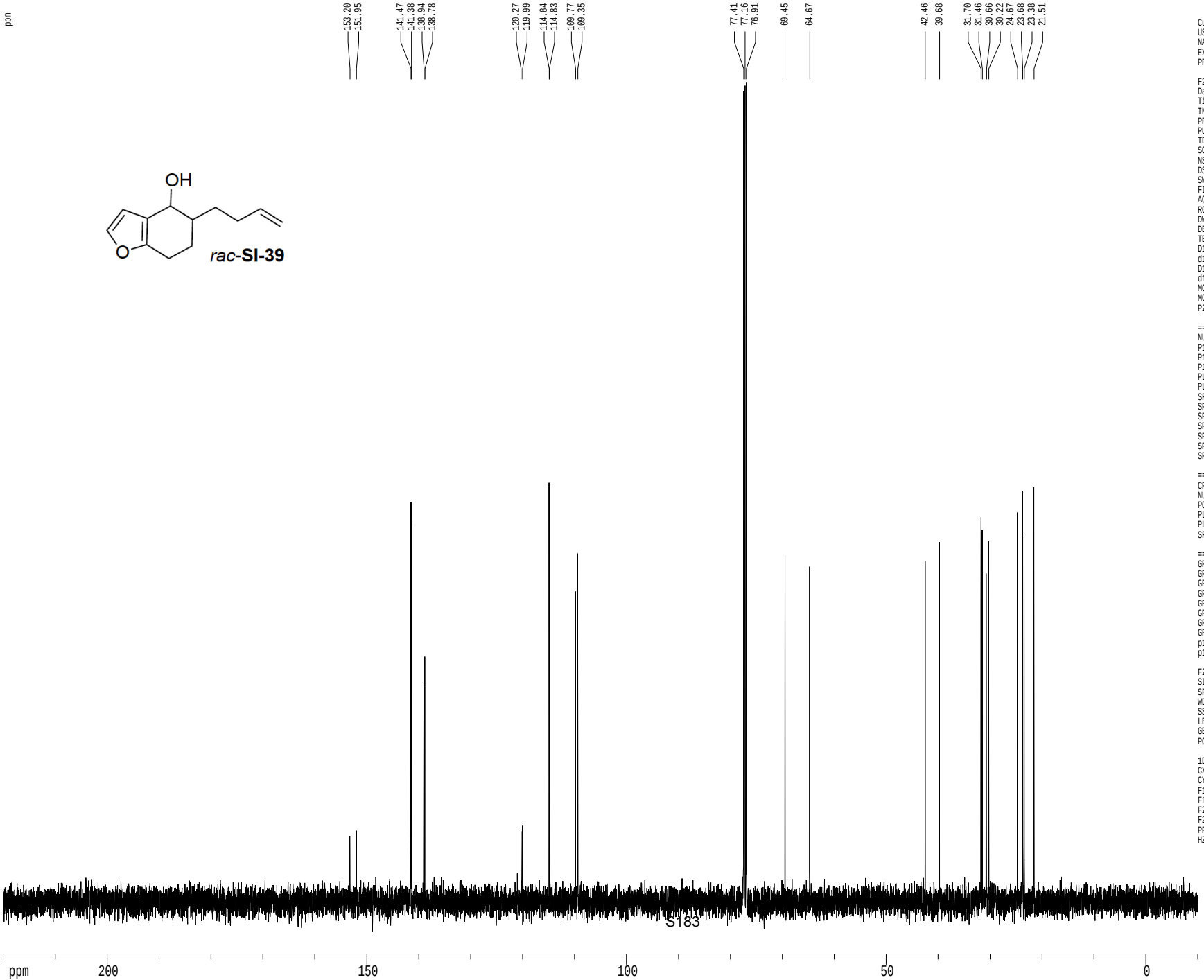
F2 - Acquisition Parameters
Date_           20131210
Time              15.54
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT            CDCl3
NS                     1
DS                     2
SWH             8012.820 Hz
FIDRES          0.098043 Hz
AQ             5.0998774 sec
RG                   7.1
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
SF01        500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF          500.2200300 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                9.000 ppm
F1               4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-245-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131210
Time              15.58
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                  113
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

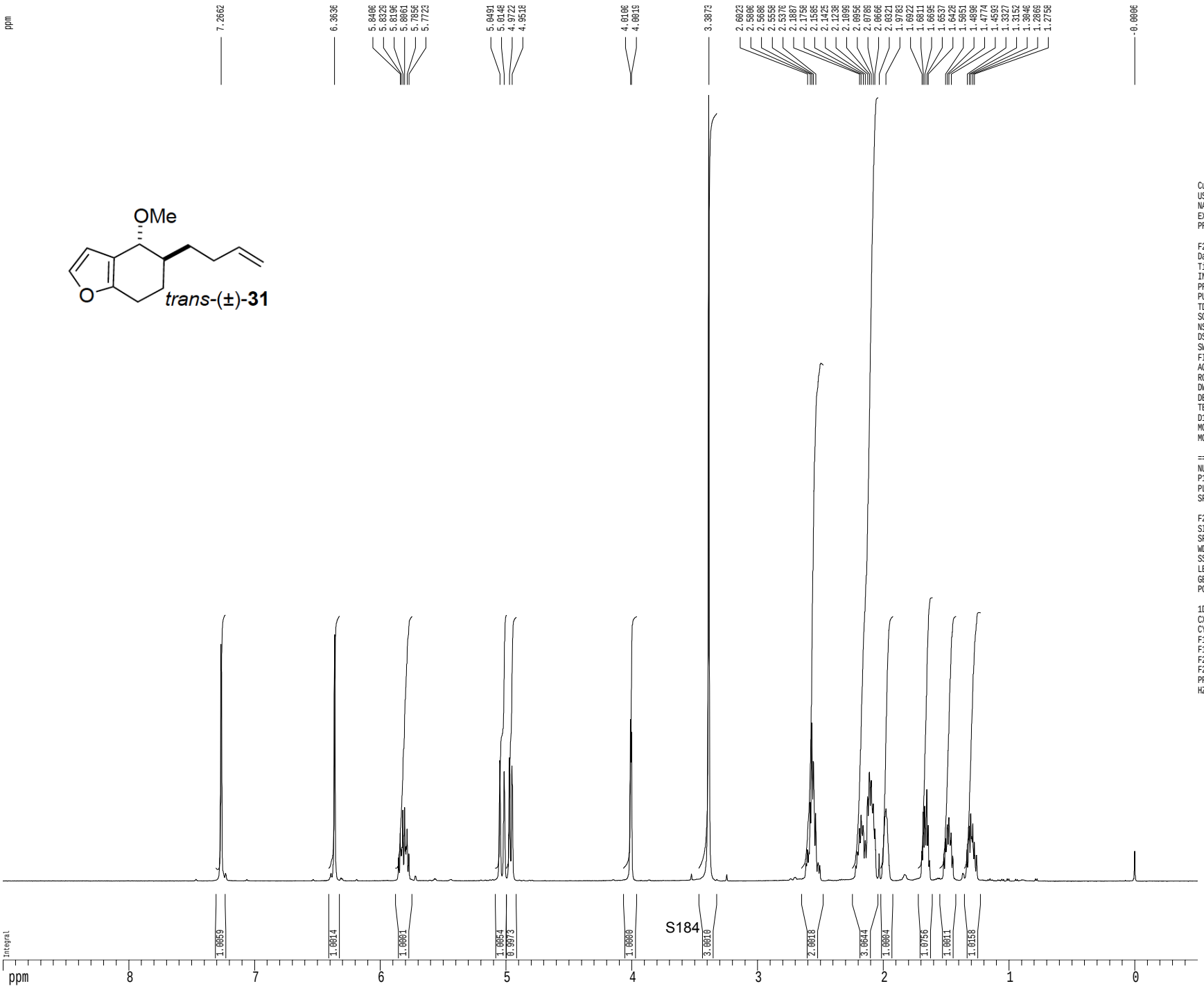
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804085 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-247-B-1HNMR
EXPNO 1
PROCNO 1

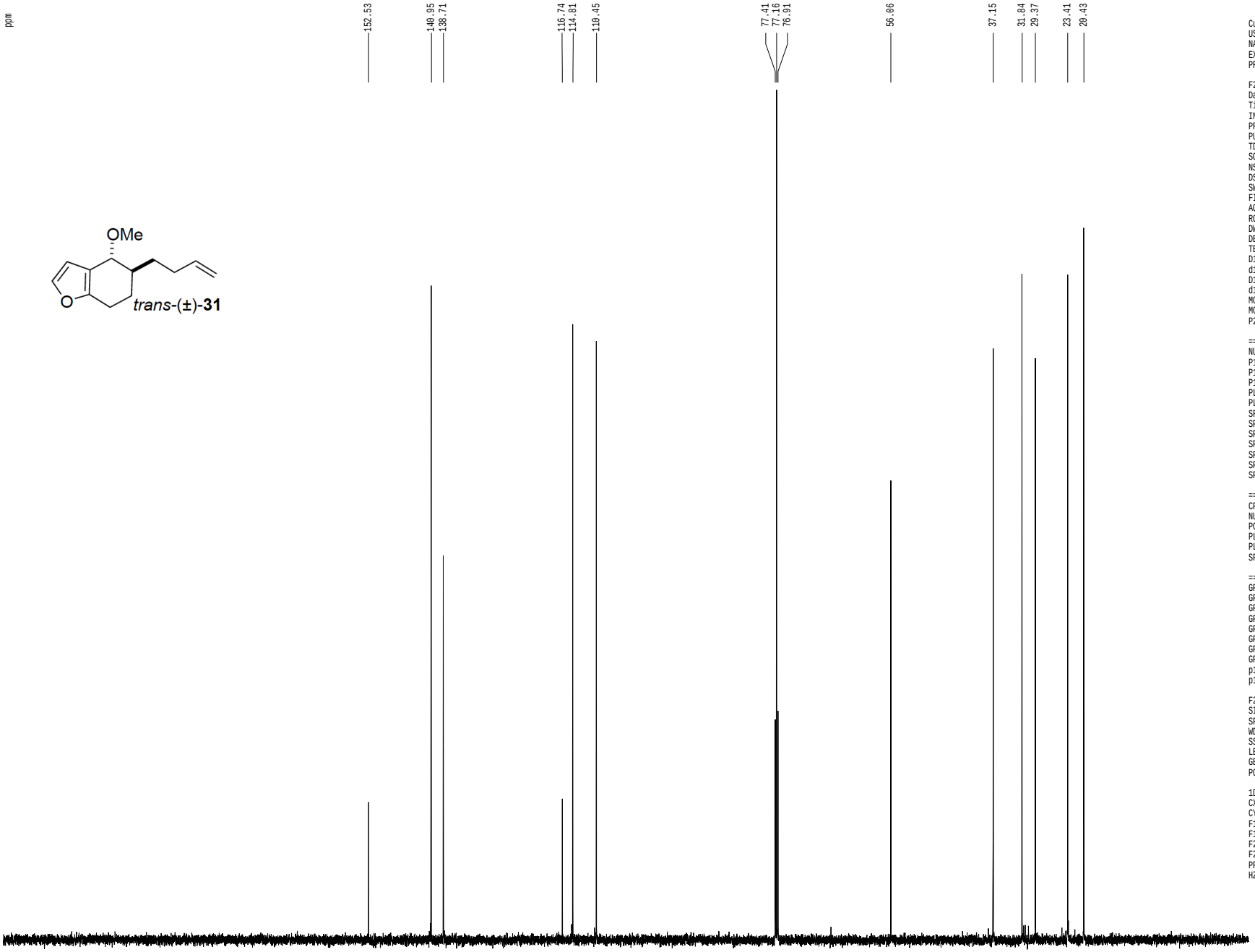
F2 - Acquisition Parameters
Date_ 20131210
Time 15.43
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200310 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-247-B-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131210
Time              15.45
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   73
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG                 6502
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

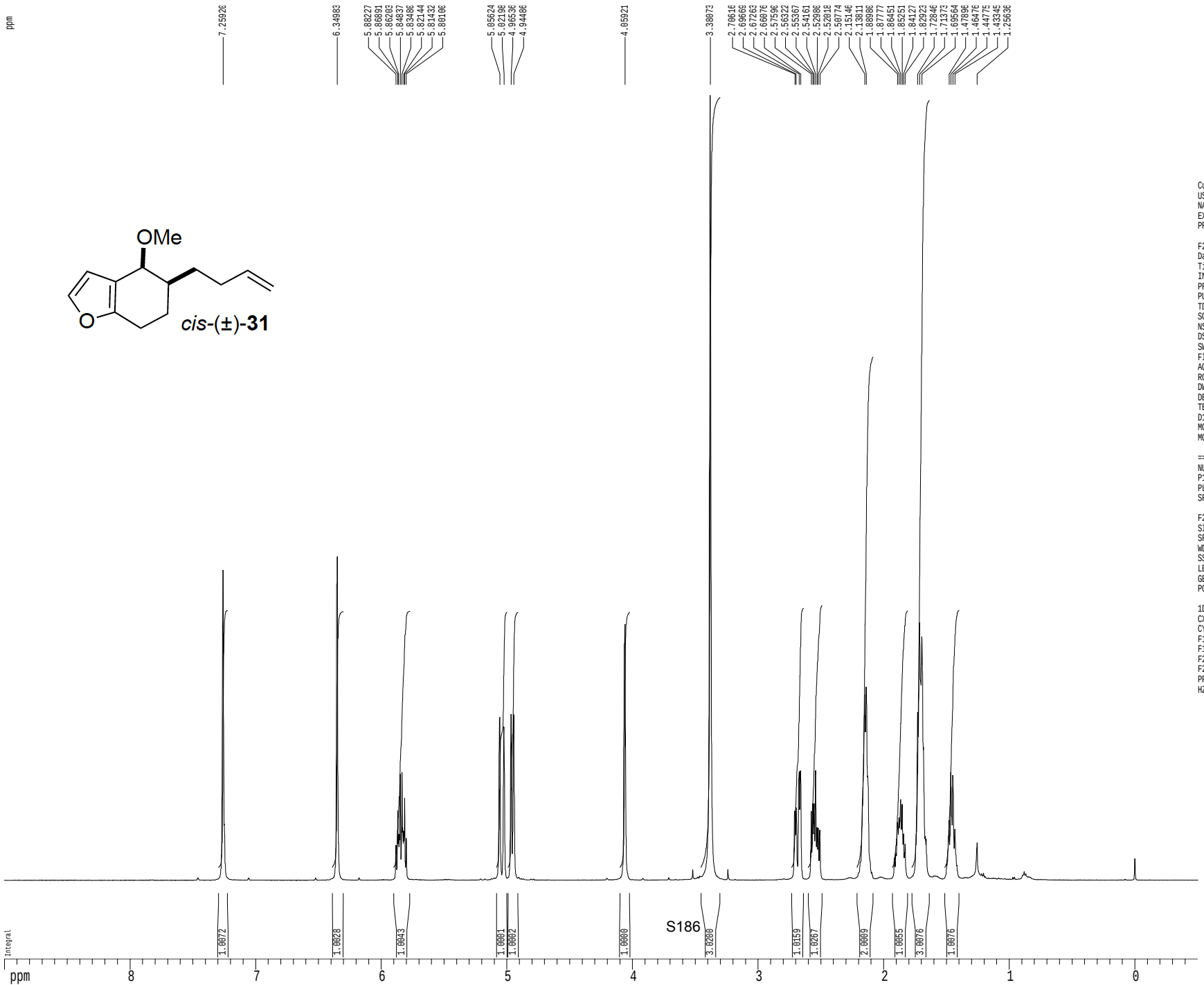
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804113 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-247-A-1HNMR
EXPNO 1
PROCNO 1

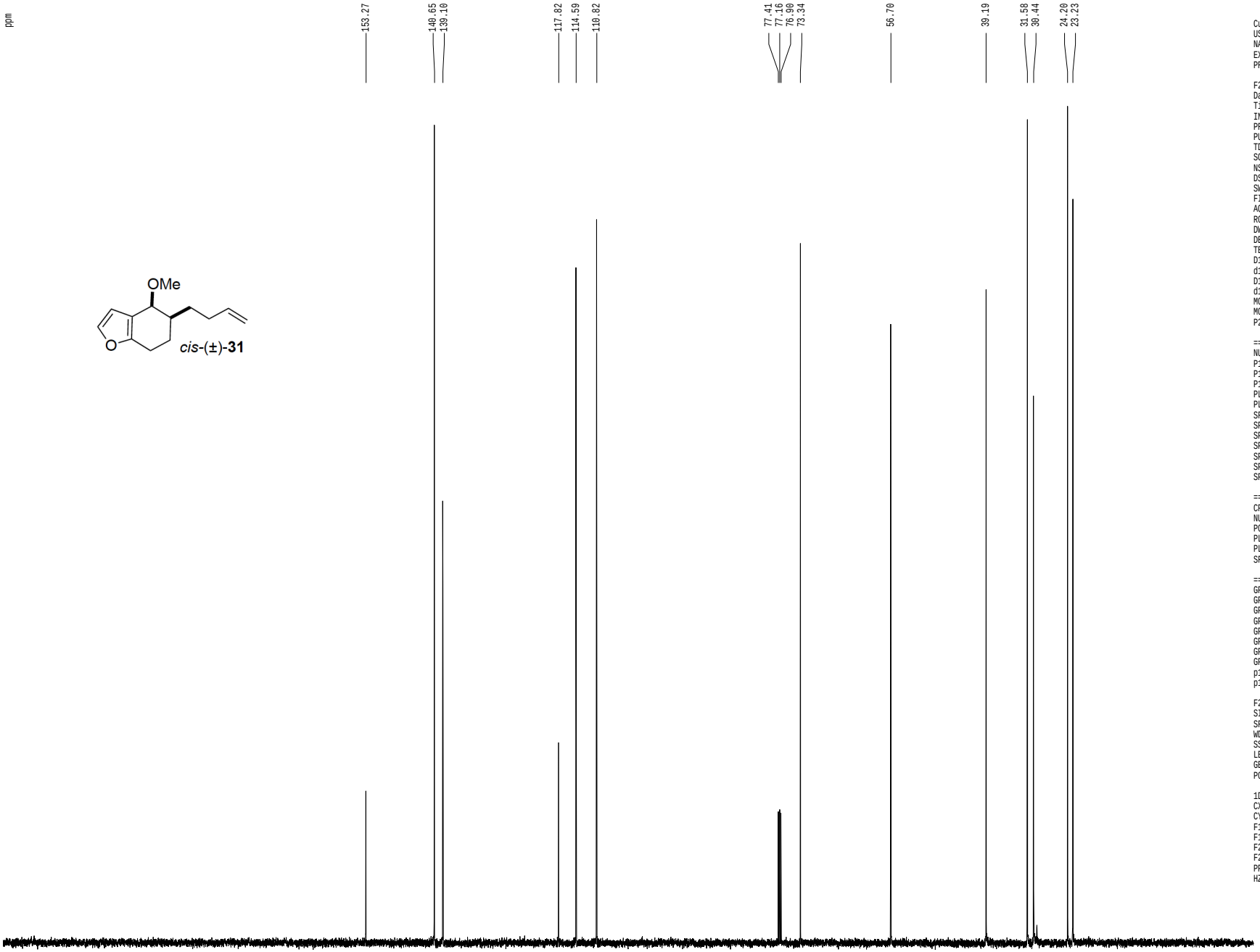
F2 - Acquisition Parameters
Date_ 20131211
Time 15.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 3.2
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200370 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-247-A-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131211
Time              15.34
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   47
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

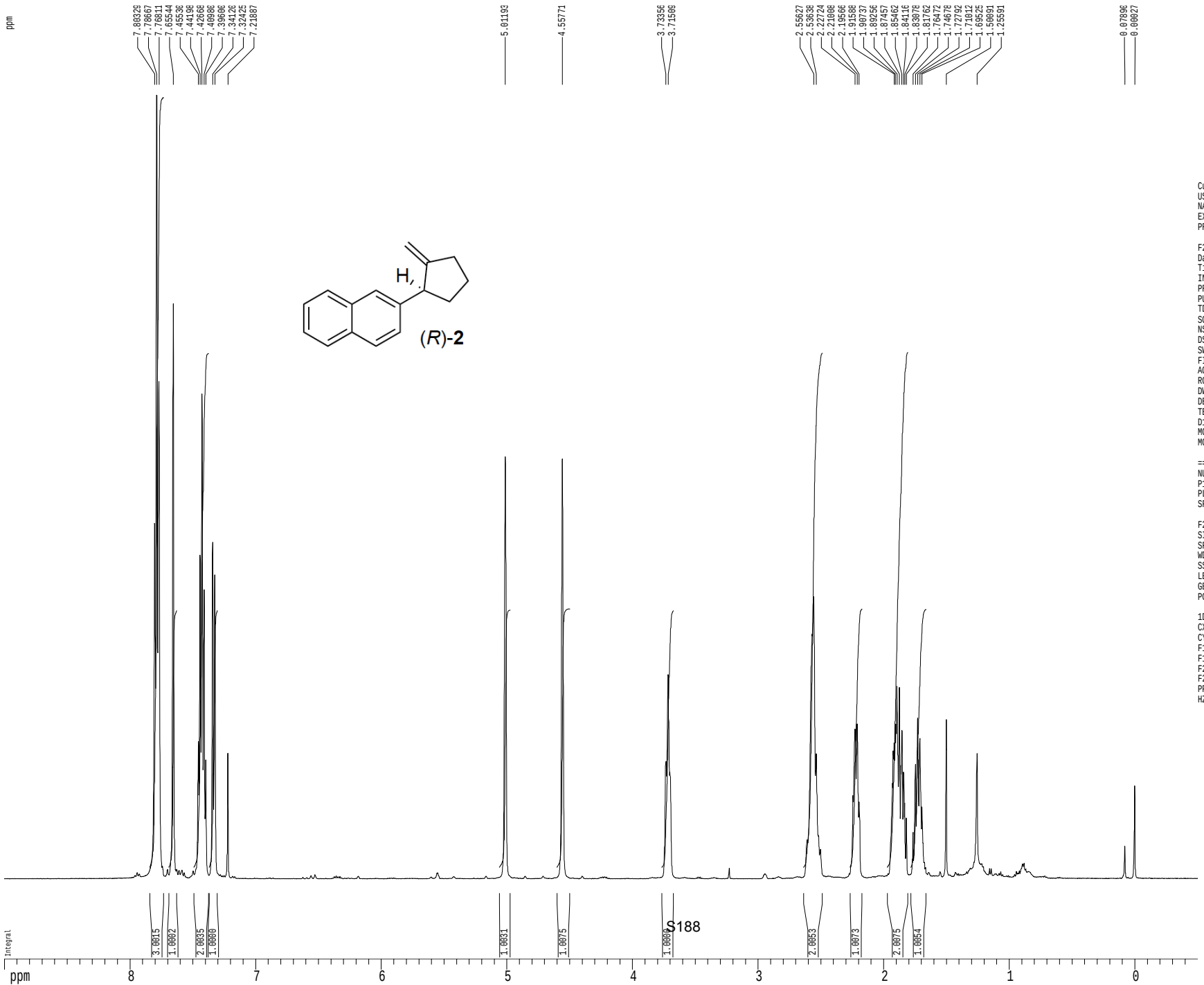
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804136 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             220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-287-1HNMR
EXPNO 1
PROCNO 1

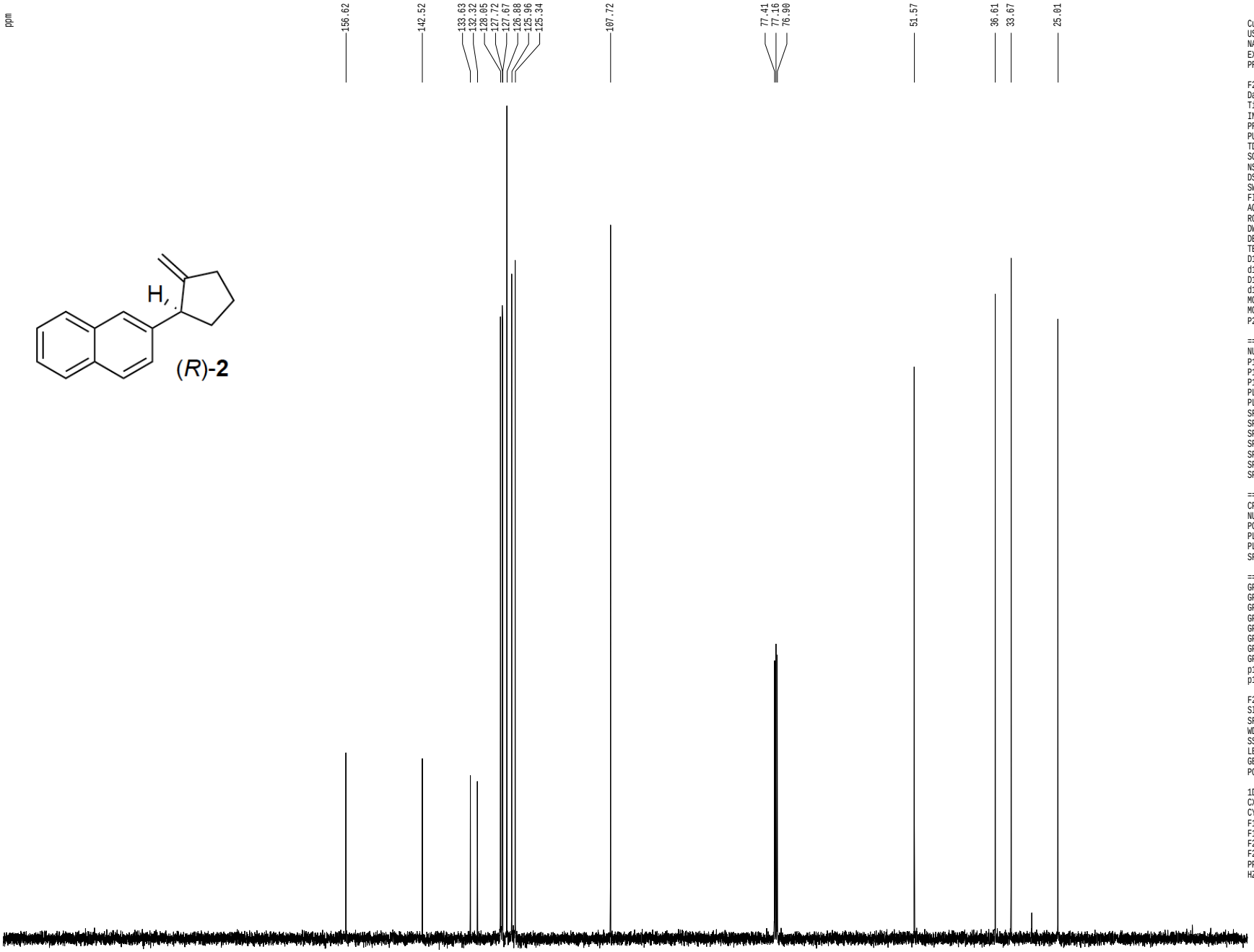
F2 - Acquisition Parameters
Date_ 20131025
Time 17.29
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200514 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42503 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-287-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131025
Time             17.32
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3T
NS                   76
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

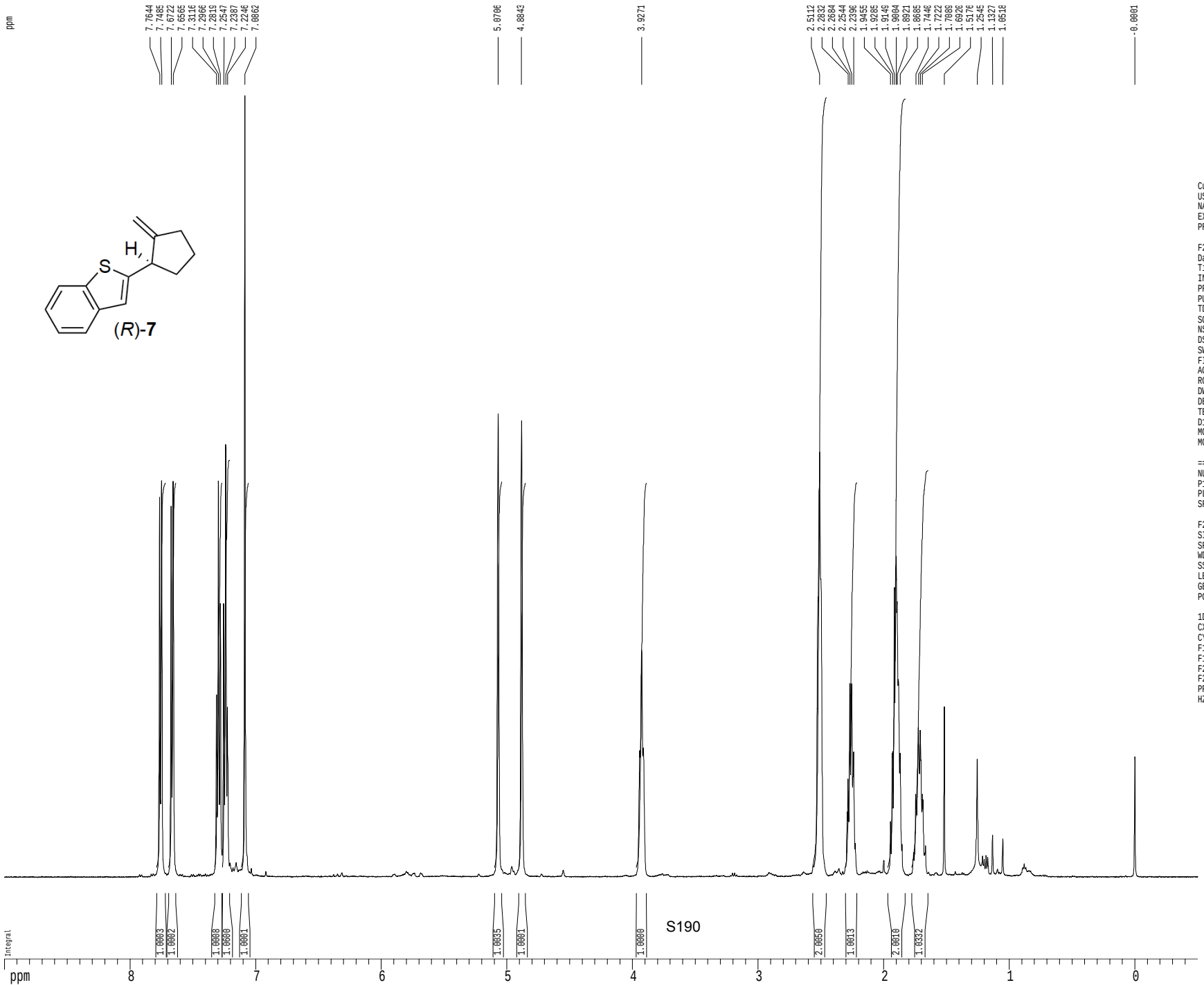
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804136 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             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER            mharri
NAME     MRH-V-282-1HNMR
EXPNO                1
PROCNO               1

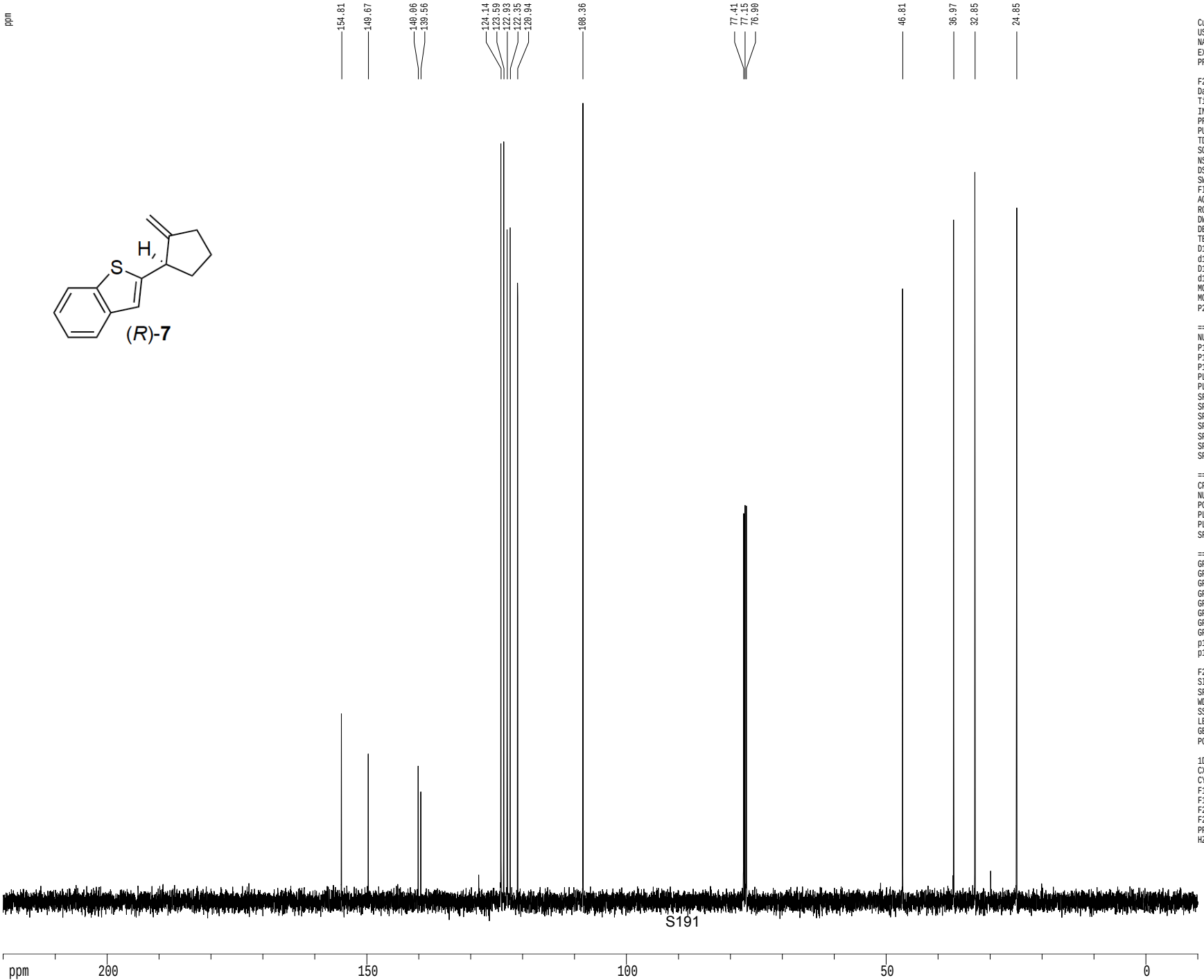
F2 - Acquisition Parameters
Date_          20131210
Time              15.32
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    1
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ           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          500.2200430 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-282-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131210
Time              15.34
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   61
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

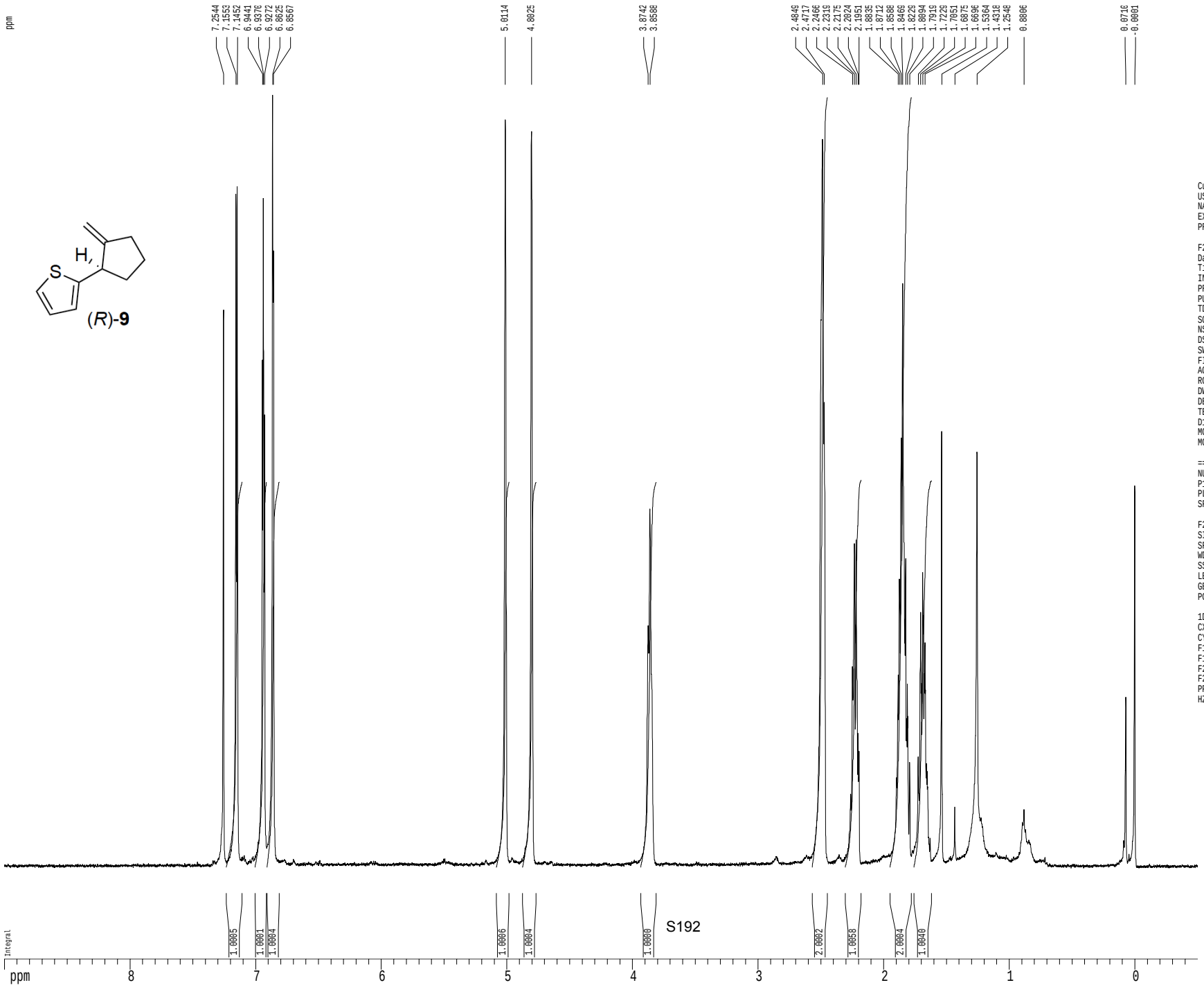
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804127 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM         10.08772 ppm/cm
HZCM        1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-259-1HNMRR
EXPNO 1
PROCNO 1

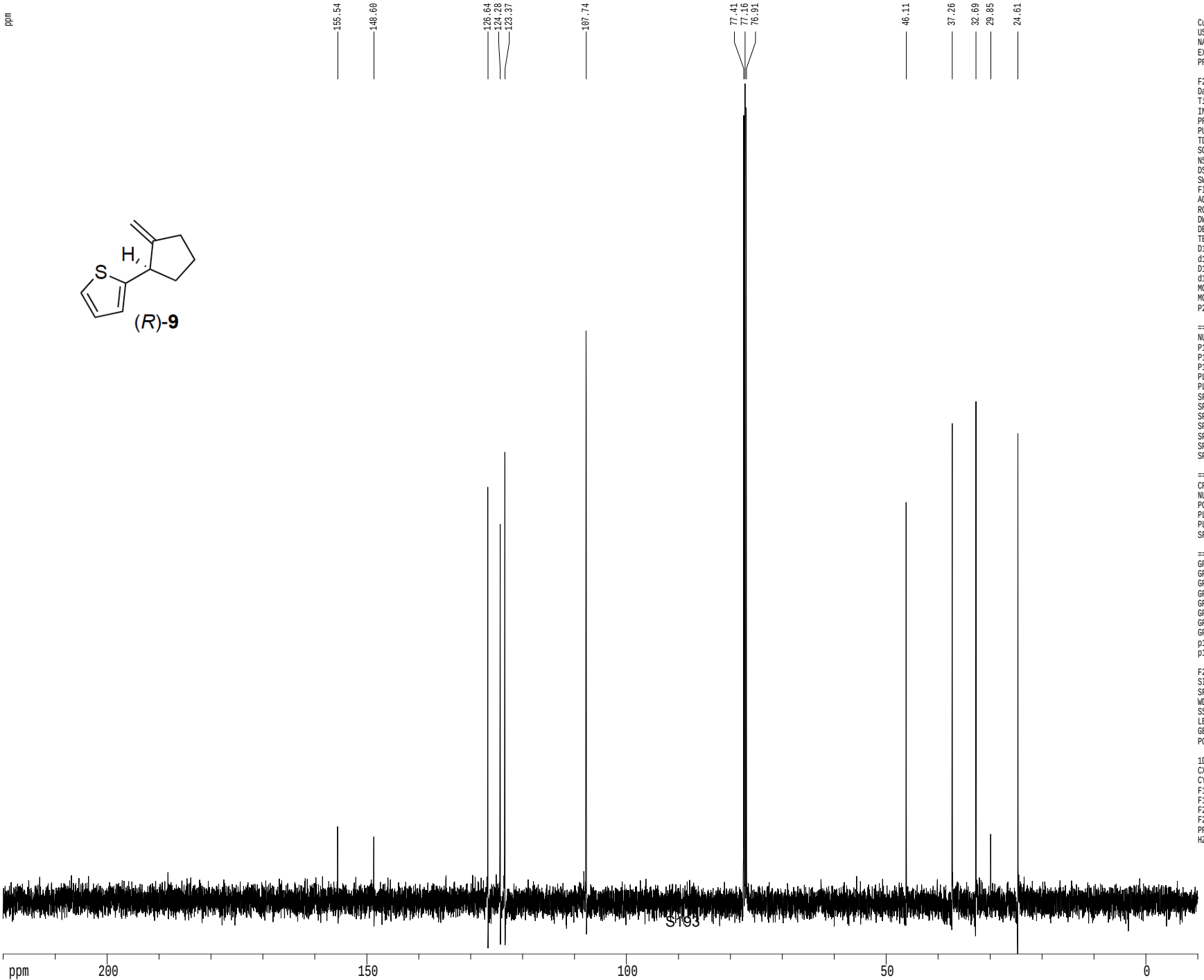
F2 - Acquisition Parameters
Date_ 20131209
Time 11.37
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200332 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-259-13CNMRR
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_          20131209
Time              11.40
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  131
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG               14596.5
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                 31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 3.20 dB
SP2                 3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1              0.00 Hz
SPOFF2              0.00 Hz

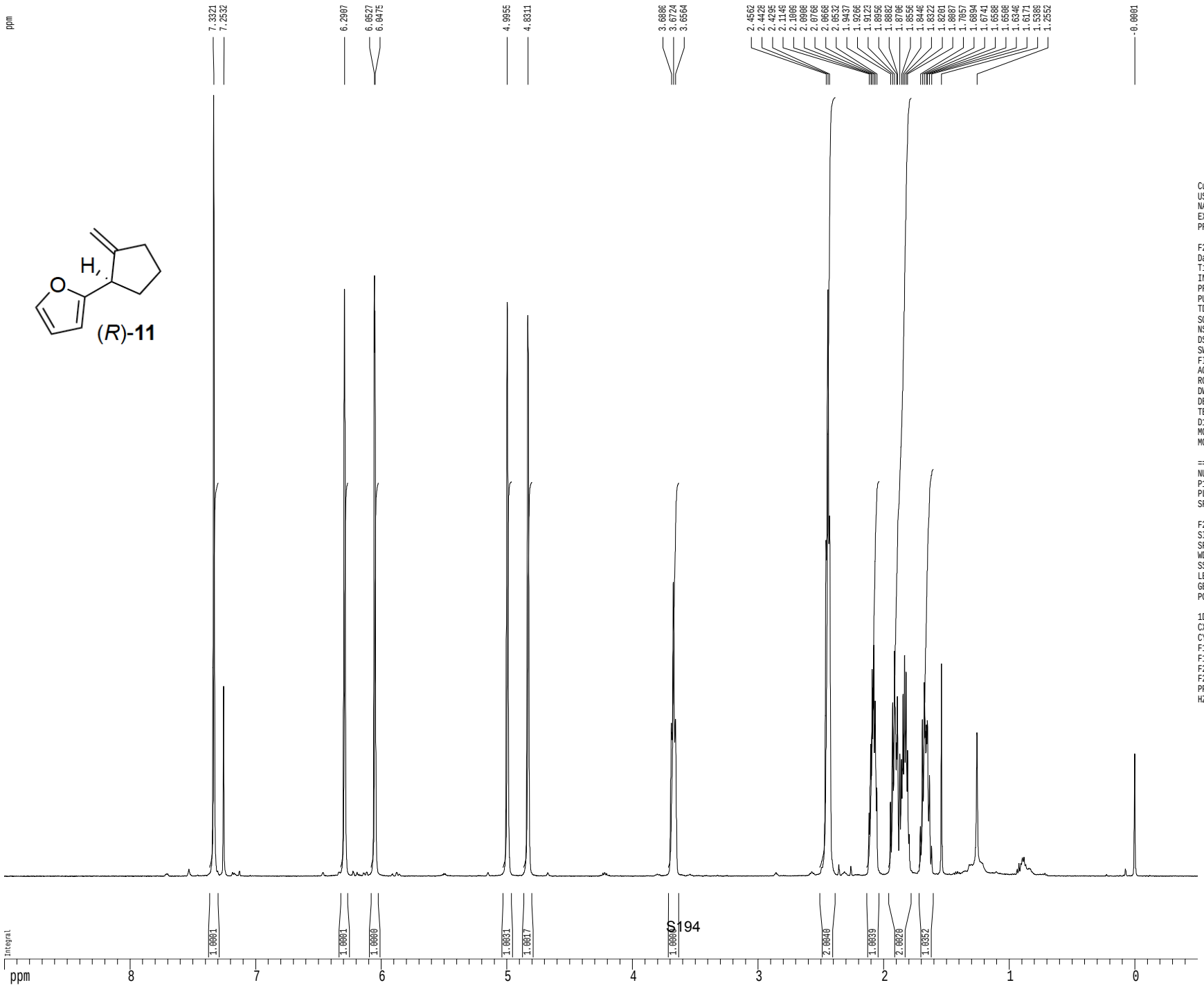
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== 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           125.7804085 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               220.000 ppm
F1              27671.69 Hz
F2P              -10.000 ppm
F2              -1257.80 Hz
PPMCM            10.08772 ppm/cm
HZCM          1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-6-1HNMR
EXPNO 1
PROCNO 1

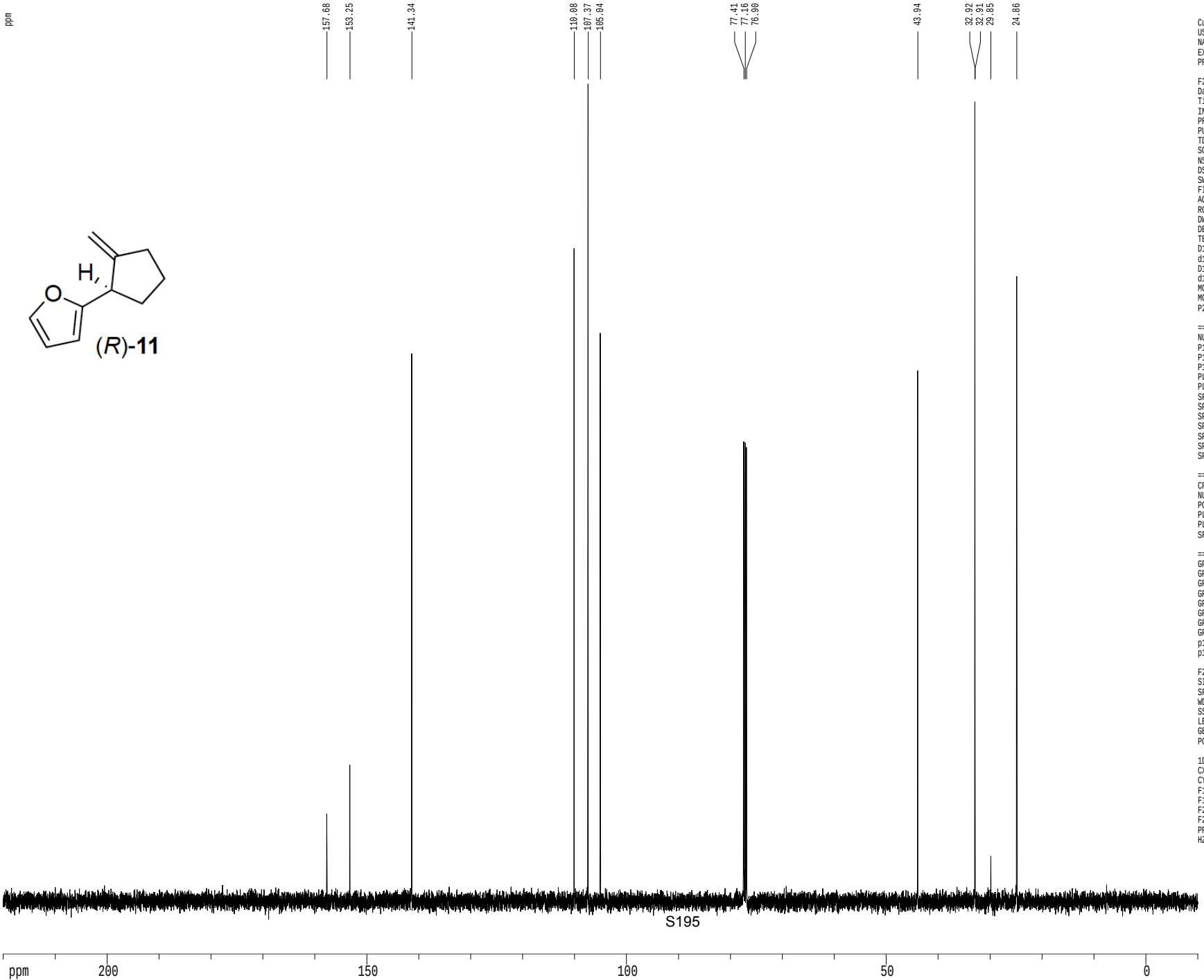
F2 - Acquisition Parameters
Date_ 20131210
Time 18.07
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200341 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-6-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131210
Time             18.09
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                  87
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG                 3251
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

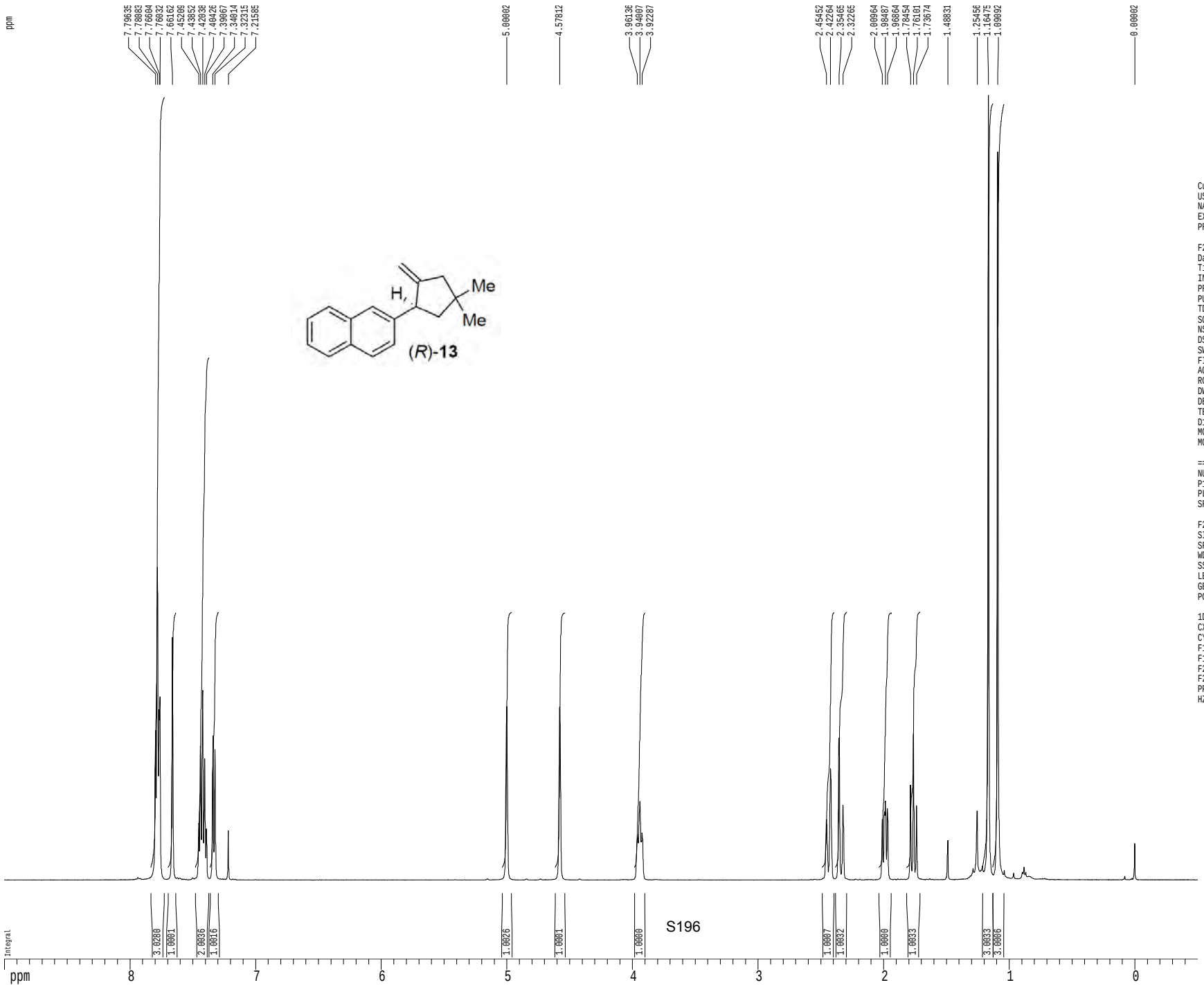
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804090 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-V-277-1HNMR
EXPNO 1
PROCNO 1

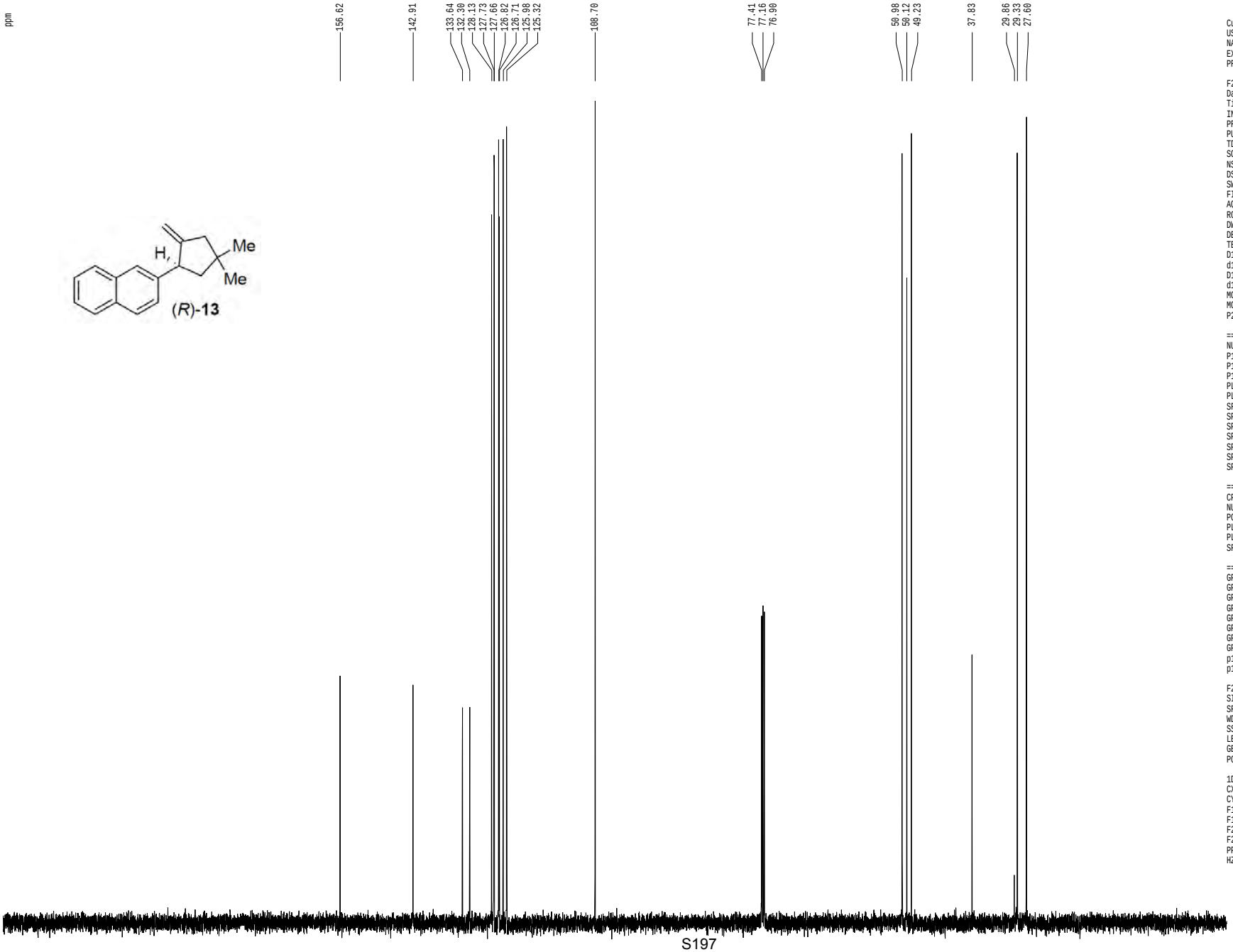
F2 - Acquisition Parameters
Date_ 20131209
Time 11.59
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200524 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42503 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-V-277-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131209
Time              12.01
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                    45
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG                 6502
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

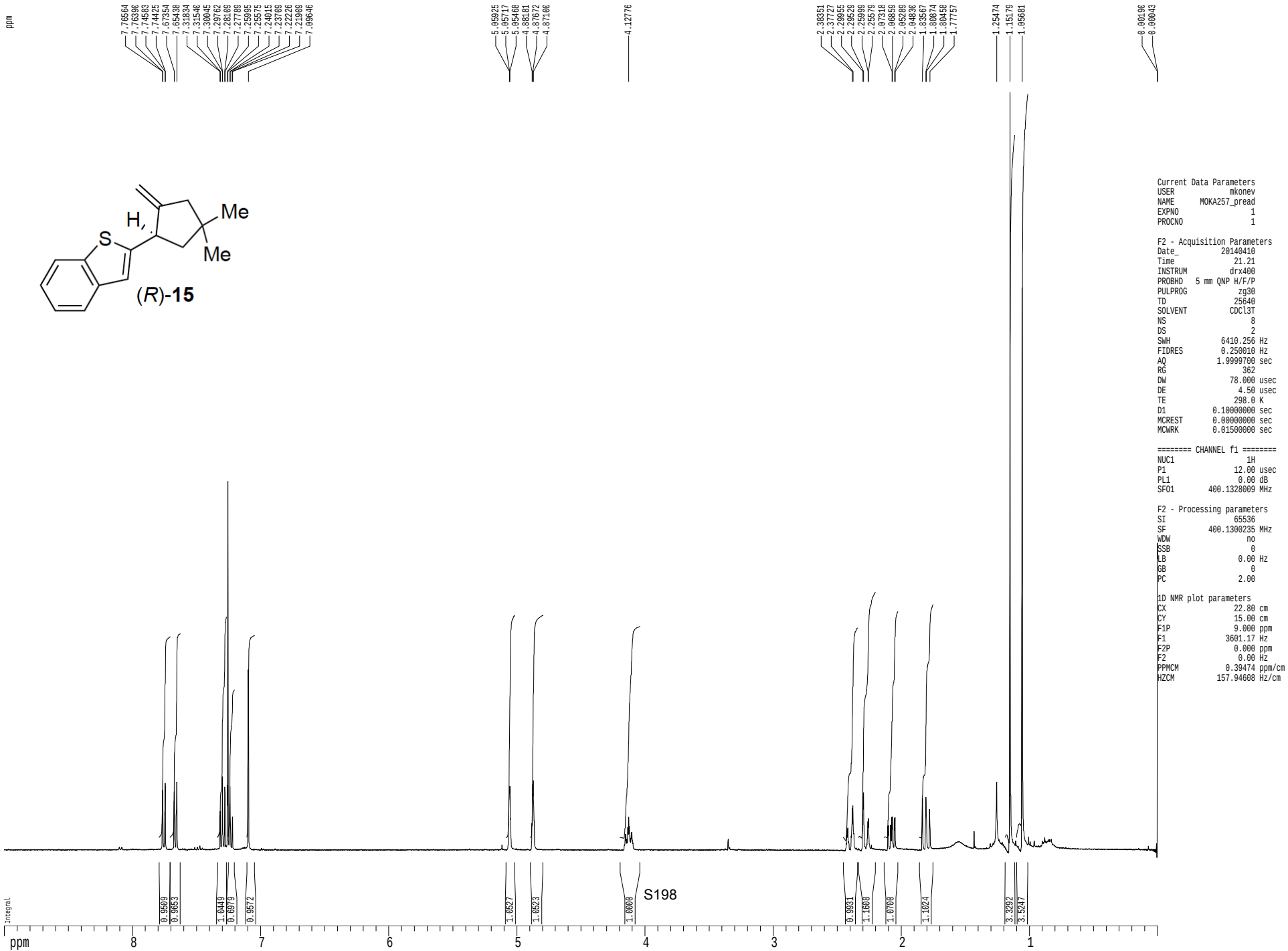
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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

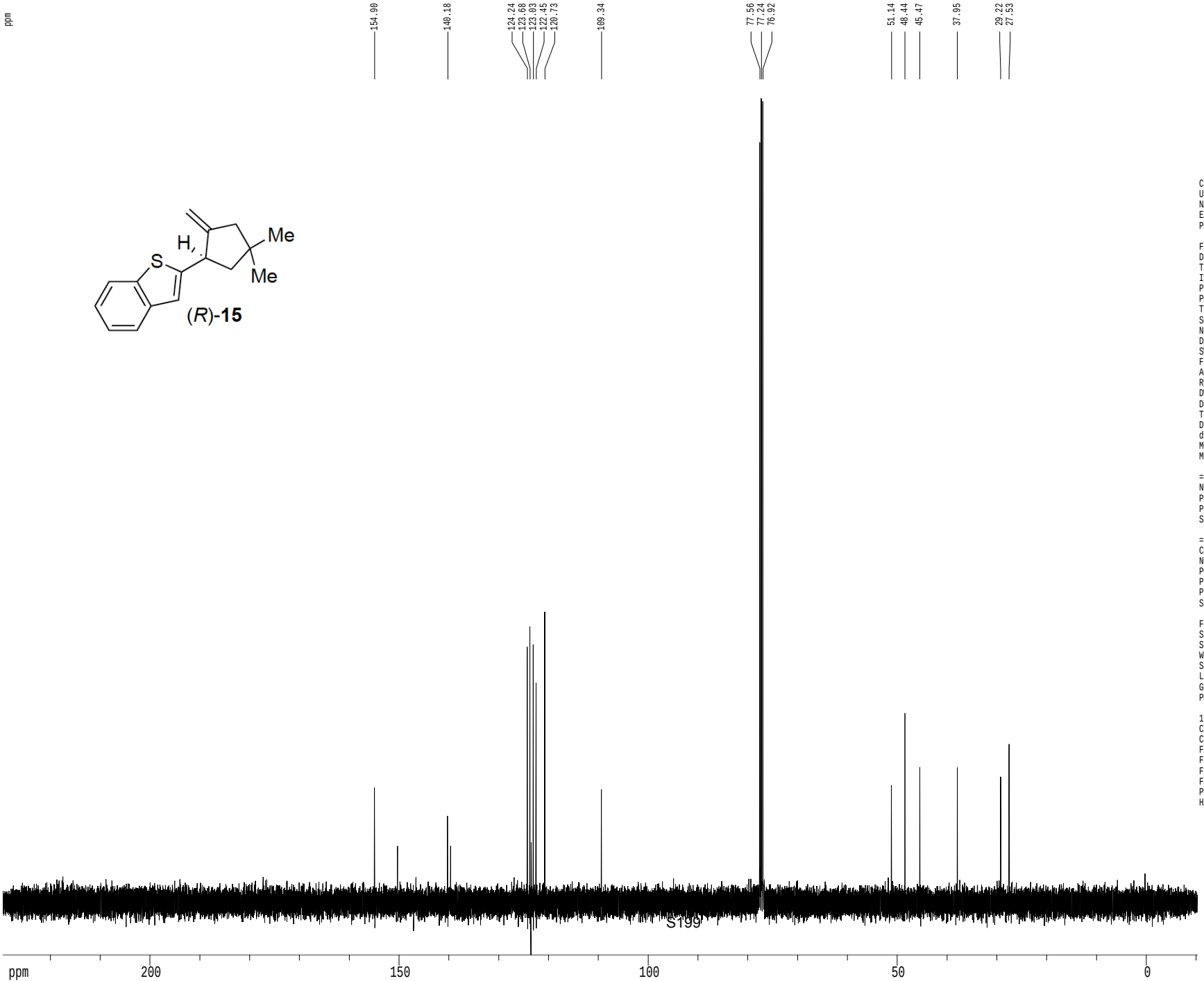
F2 - Processing parameters
SI                 65536
SF          125.7804131 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P             220.000 ppm
F1            27671.69 Hz
F2P             -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum


ppm
7.76564
7.76396
7.74583
7.74425
7.67354
7.65438
7.31834
7.31546
7.30045
7.29762
7.28109
7.27789
7.25995
7.25575
7.24015
7.23709
7.22226
7.21909
7.09646
5.85925
5.85717
5.85468
4.88181
4.87672
4.87106
4.12776
2.38351
2.37727
2.29955
2.29529
2.25999
2.25579
2.07318
2.06859
2.05289
2.04838
1.83567
1.80874
1.80458
1.77757
1.25474
1.15179
1.05661
0.00196
0.00043
H
S
Me
Me
(R)-15
Current Data Parameters
USER mkonev
NAME MOKA257_pread
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140410
Time 21.21
INSTRUM drx400
PROBHD 5 mm QNP H/F/P
PULPROG zg30
TD 25640
SOLVENT CDCl3T
NS 8
DS 2
SWH 6410.256 Hz
FIDRES 0.250010 Hz
AQ 1.9999700 sec
RG 362
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 400.1300235 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3601.17 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 157.94608 Hz/cm
Integral
0.9509
0.9653
1.0449
0.6979
0.9572
1.0527
1.0523
1.0000
0.9931
1.1608
1.0700
1.1824
3.3292
3.5247
ppm
8
7
6
5
4
3
2
1

13C spectrum with 1H decoupling




```
Current Data Parameters
USER              mkonev
NAME      MOKA257_cpread
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140410
Time               21.28
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                  800
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

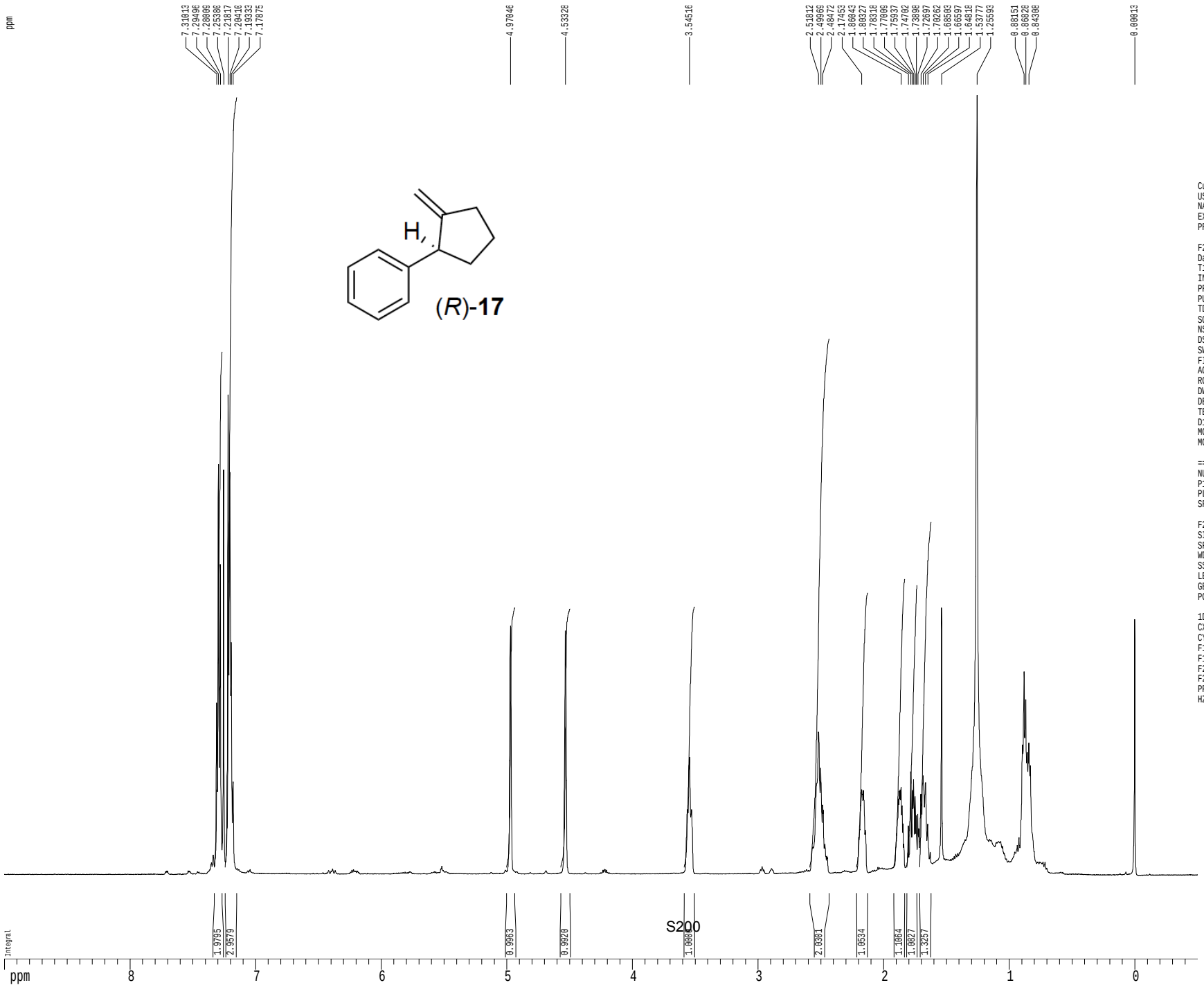
======== CHANNEL f1 ========
NUC1                13C
P1                 7.75 usec
PL1               -3.00 dB
SFO1        100.6237964 MHz

======== 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                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.50 cm
F1P               229.496 ppm
F1               23090.21 Hz
F2P               -10.579 ppm
F2               -1064.37 Hz
PPMCM            10.52959 ppm/cm
HZCM          1059.41138 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-161-1HNMR
EXPNO 1
PROCNO 1

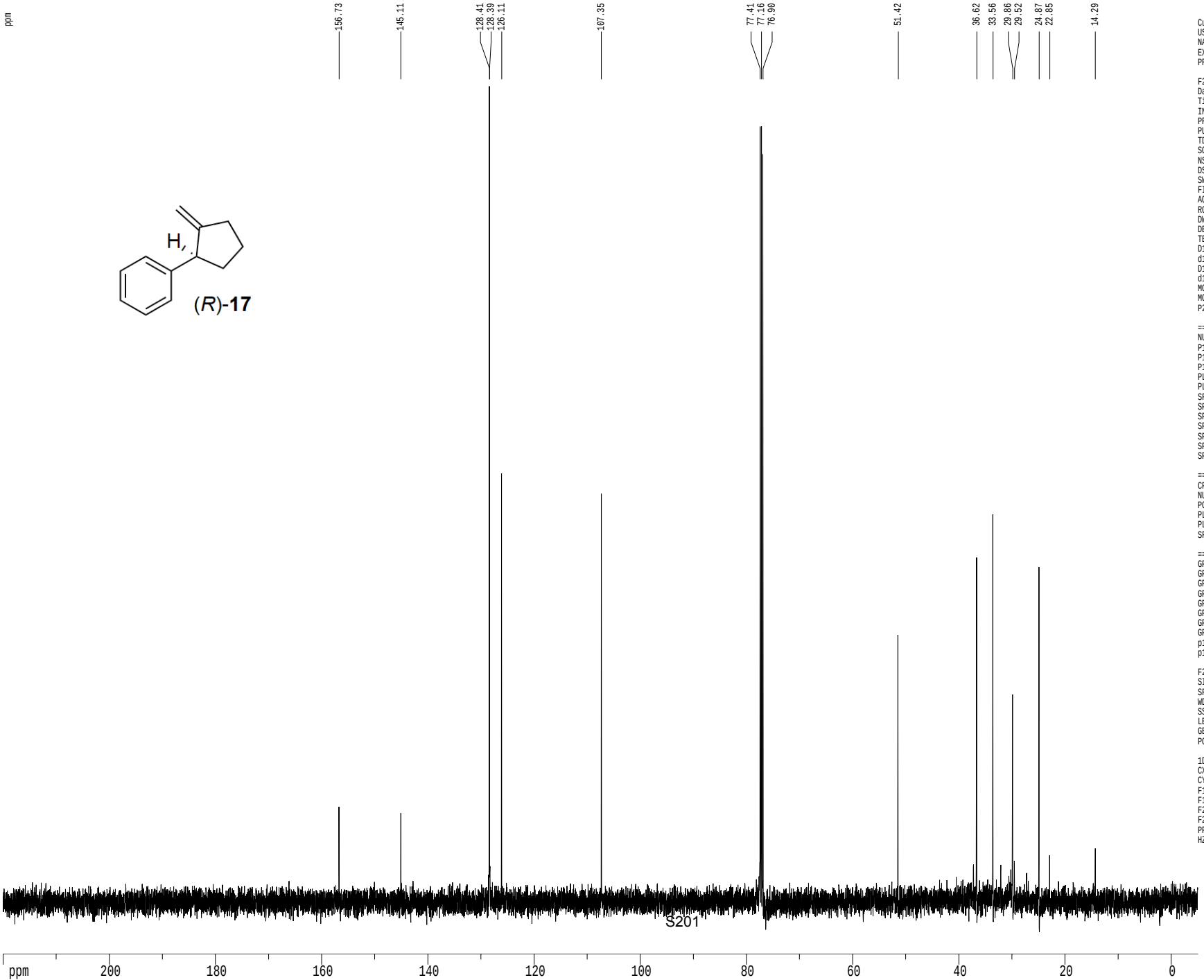
F2 - Acquisition Parameters
Date_ 20140425
Time 16.43
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 7.1
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200355 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-161-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140425
Time              16.48
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                  121
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

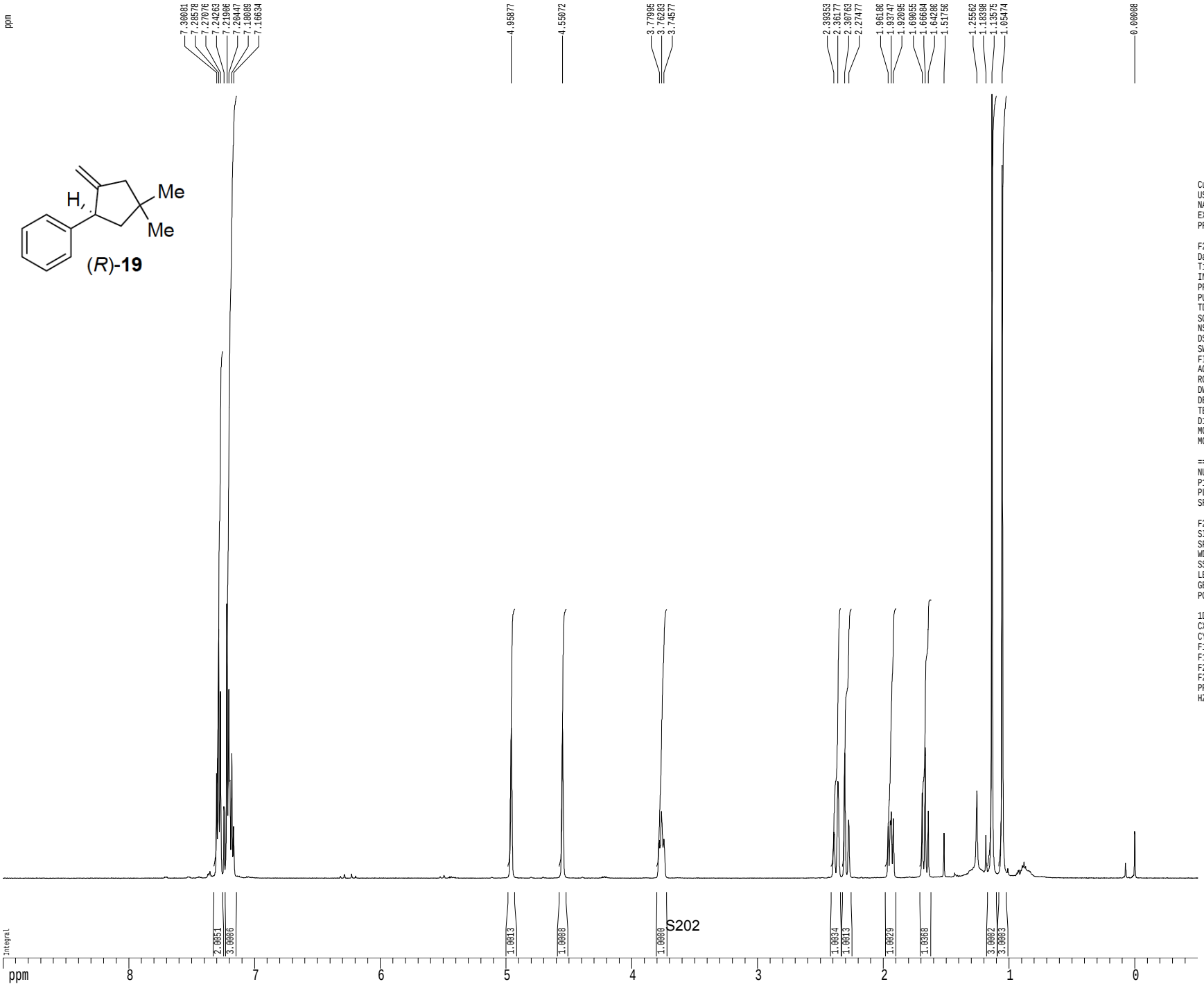
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804090 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             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



S202

```
Current Data Parameters
USER              mharri
NAME     MRH-VI-23-1HNMR
EXPNO                  1
PROCNO                 1

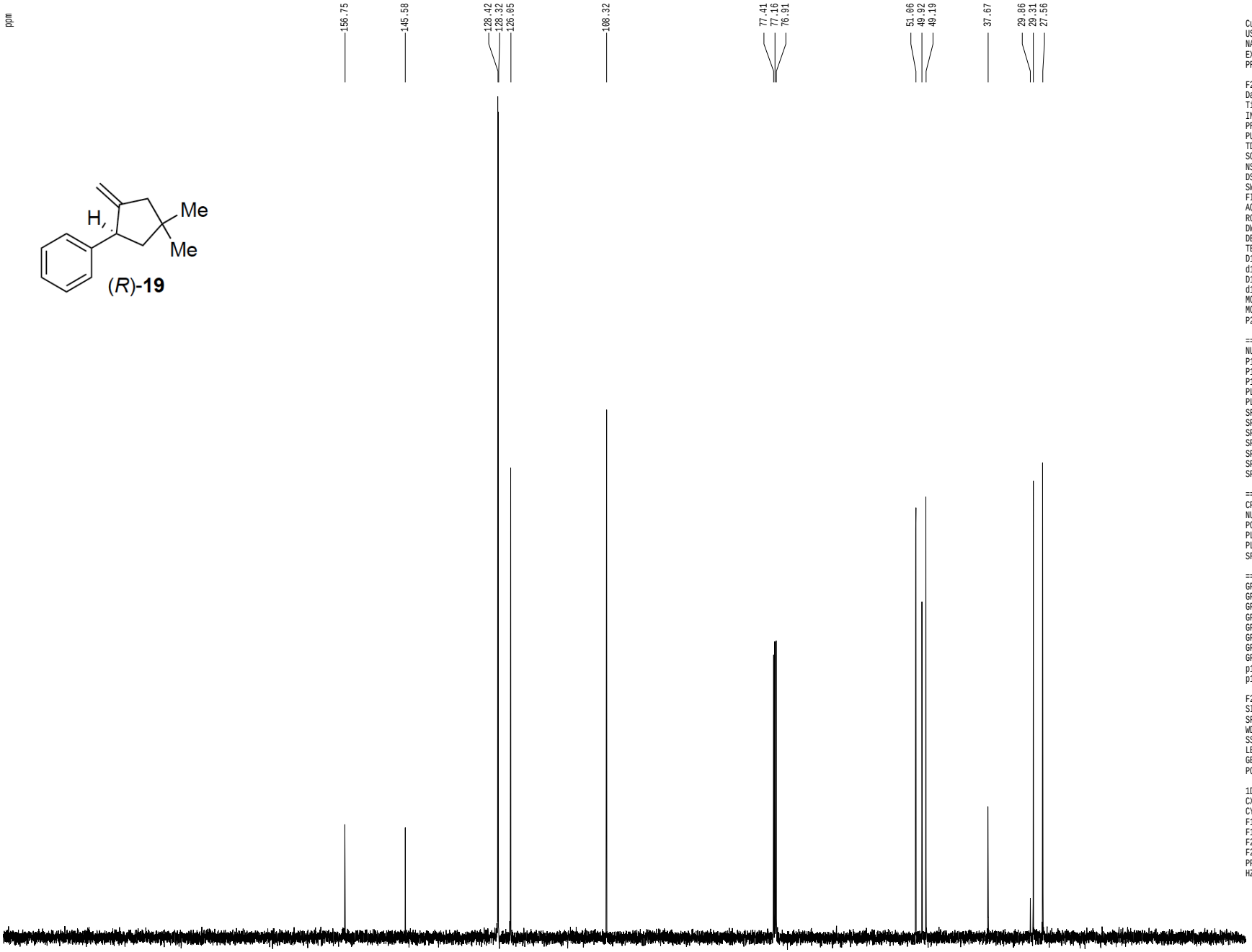
F2 - Acquisition Parameters
Date_           20131209
Time              15.43
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    1
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            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          500.2200396 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               9.000 ppm
F1             4501.98 Hz
F2P             -0.500 ppm
F2             -250.11 Hz
PPMCM          0.41667 ppm/cm
HZCM         208.42502 Hz/cm
```

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-23-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20131209
Time              15.45
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   70
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG                 8192
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

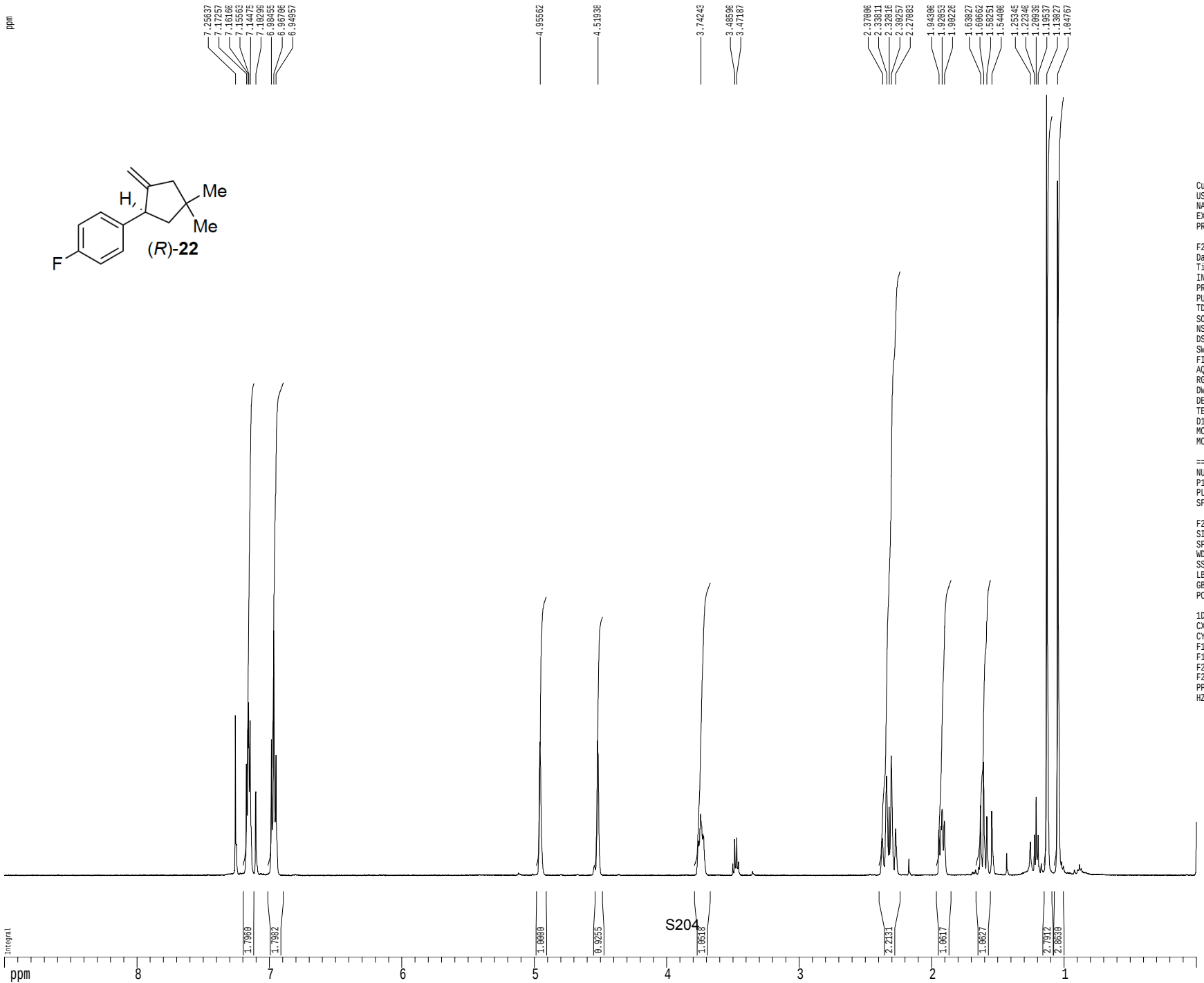
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804094 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             220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mkonev
NAME MOKA163h-char
EXPNO 1
PROCNO 1

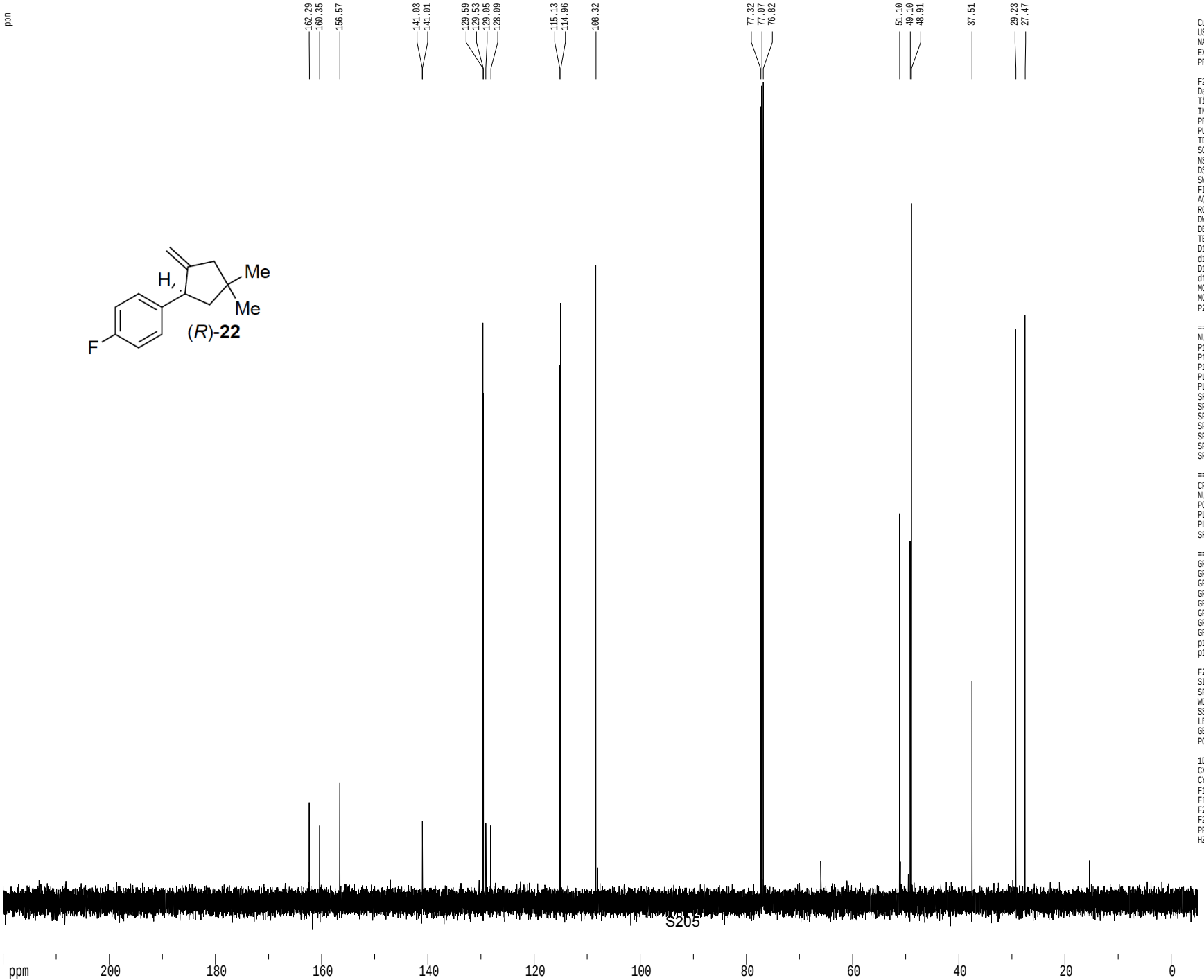
F2 - Acquisition Parameters
Date_ 20131125
Time 1.20
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3T
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200332 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm

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




```
Current Data Parameters
USER            mkonev
NAME      MOKA163c-char
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_          20131125
Time               1.24
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                 259
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ           1.0813940 sec
RG                4096
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               31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1               3.20 dB
SP2               3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2     Crp60comp.4
SPOFF1            0.00 Hz
SPOFF2            0.00 Hz

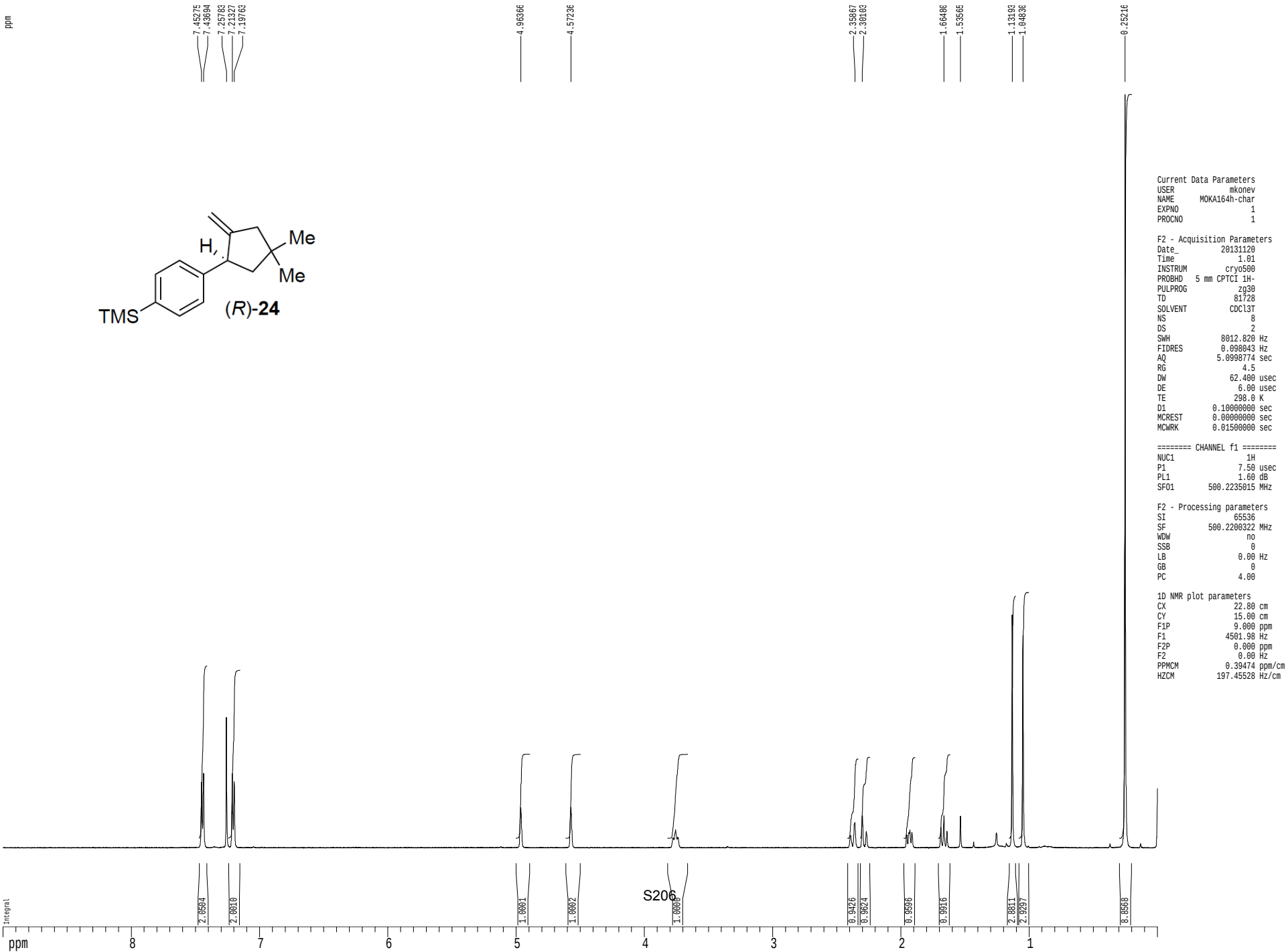
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               1.60 dB
PL12             24.60 dB
SFO2        500.2225011 MHz

====== 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

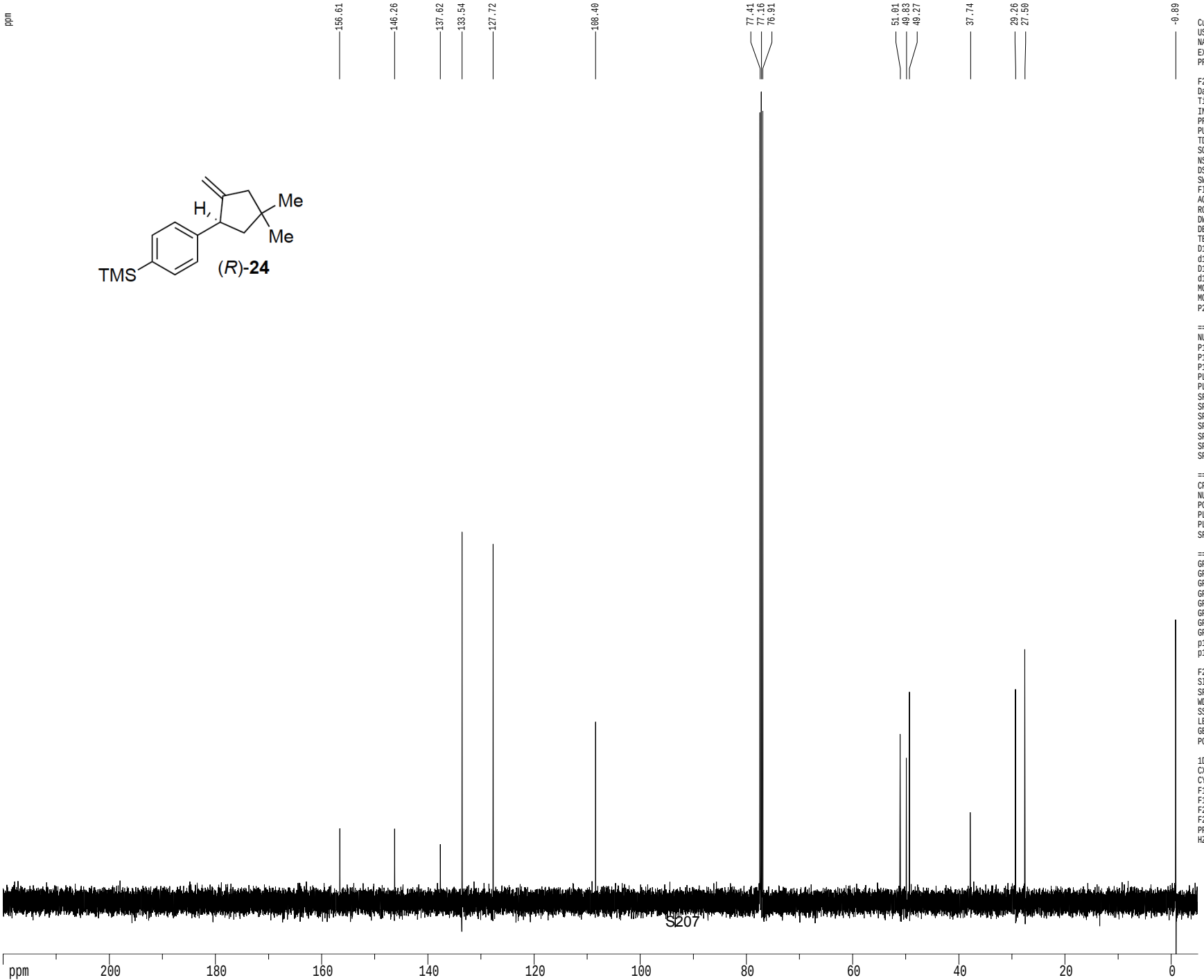
F2 - Processing parameters
SI                65536
SF          125.7804190 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                15.65 cm
F1P            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM          9.86842 ppm/cm
HZCM        1241.25415 Hz/cm
```

1H spectrum


ppm
7.45275
7.43694
7.25783
7.21327
7.19763
4.96366
4.57236
2.35867
2.30103
1.66486
1.53565
1.13193
1.04838
0.25216
Me
Me
H
TMS
(R)-24
Current Data Parameters
USER mkonev
NAME MOKA164h-char
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131120
Time 1.01
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 4.5
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 500.2200322 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.39474 ppm/cm
HZCM 197.45528 Hz/cm
Integral
2.0504
2.0010
1.0001
1.0002
1.0000
0.9426
0.9624
0.9596
0.9916
2.8611
2.9297
8.8568
ppm
8
7
6
5
4
3
2
1

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




```
Current Data Parameters
USER            mkonev
NAME      MOKA164c-char
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_          20131120
Time               1.12
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                 256
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG                6502
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               31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1               3.20 dB
SP2               3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2     Crp60comp.4
SPOFF1            0.00 Hz
SPOFF2            0.00 Hz

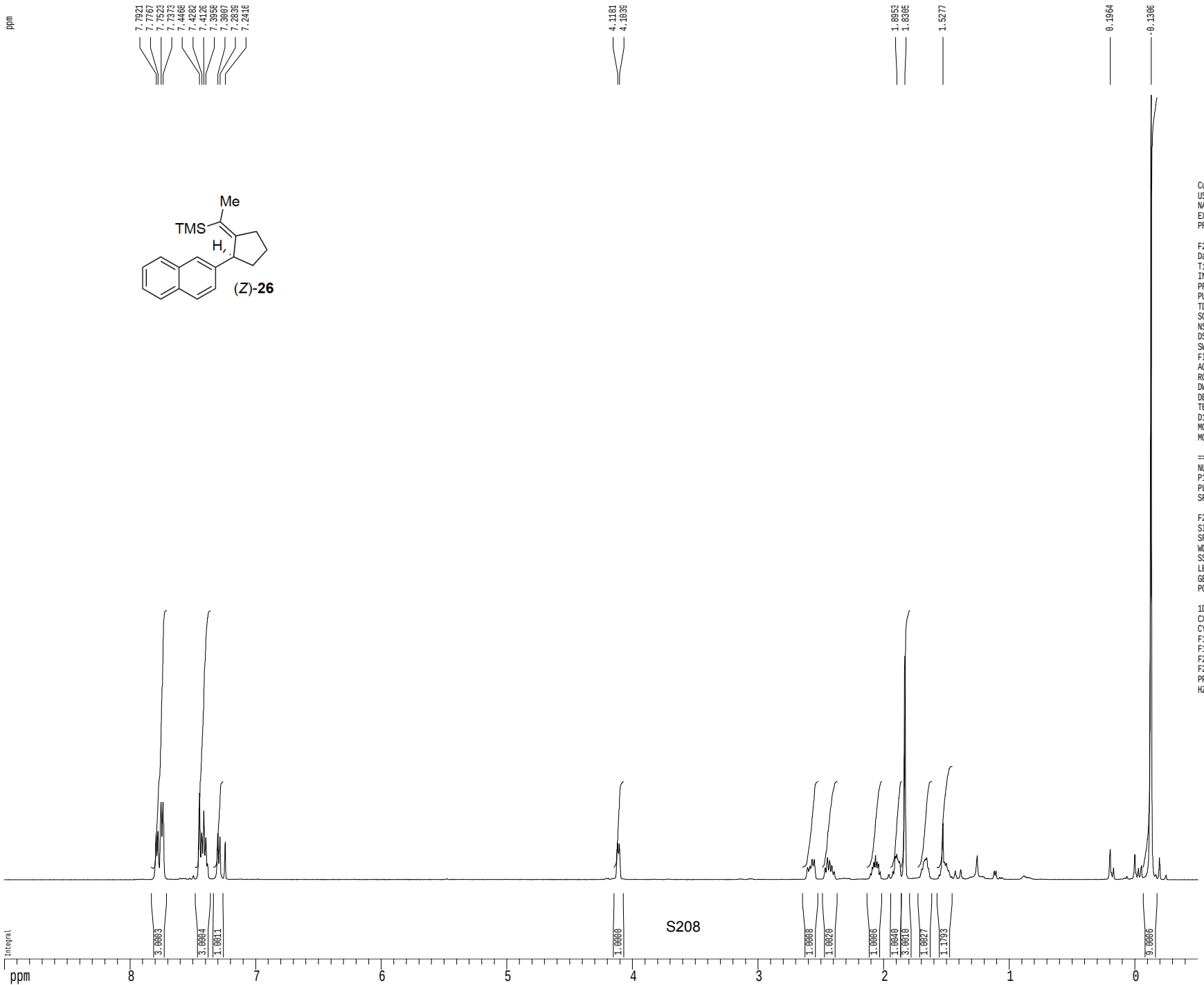
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               1.60 dB
PL12             24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804076 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM          9.86842 ppm/cm
HZCM        1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-84-A-1HNMR
EXPNO 1
PROCNO 1

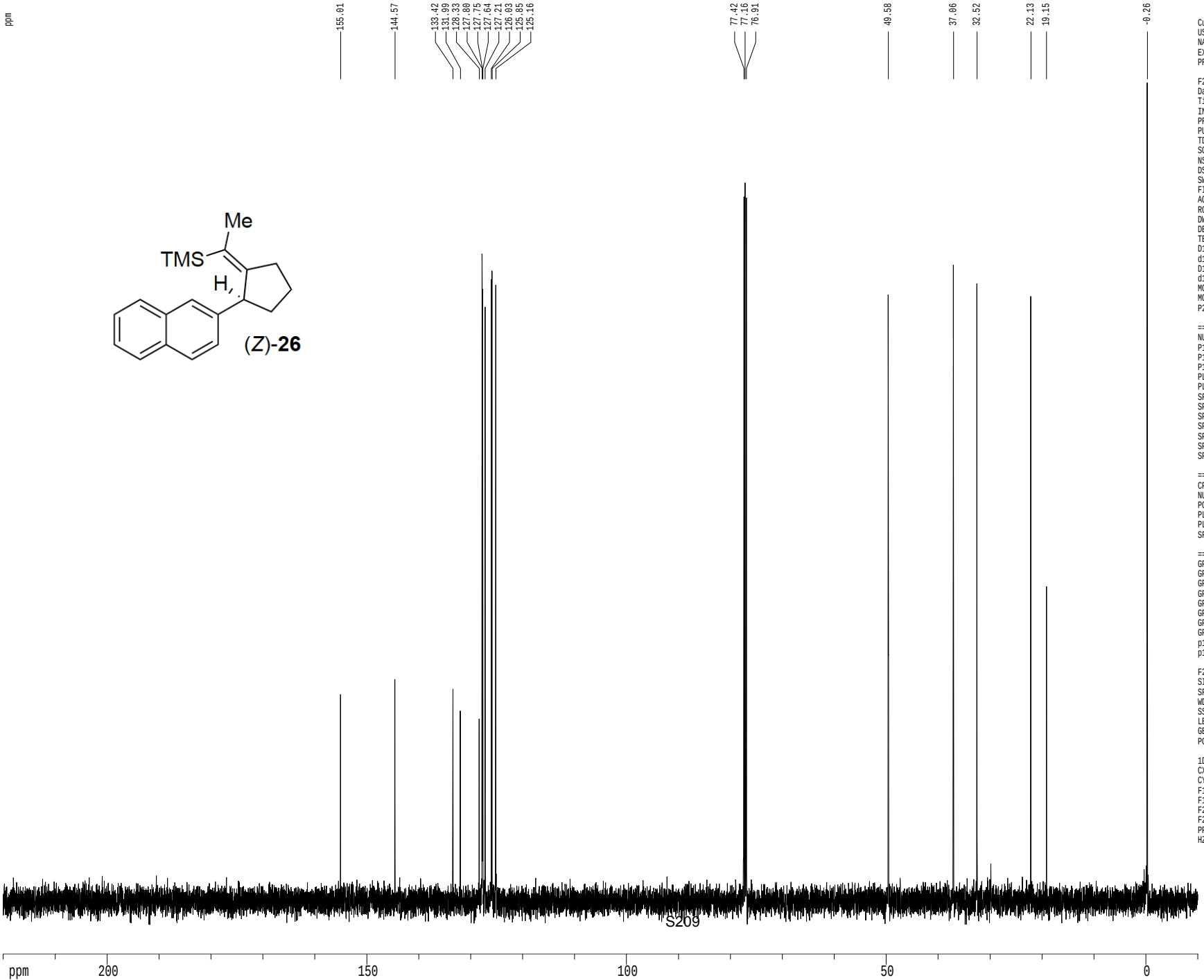
F2 - Acquisition Parameters
Date_ 20140127
Time 20.21
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 9
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200406 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-84-A-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140127
Time             20.25
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                 175
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ           1.0813940 sec
RG               13004
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              31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1               3.20 dB
SP2               3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1            0.00 Hz
SPOFF2            0.00 Hz

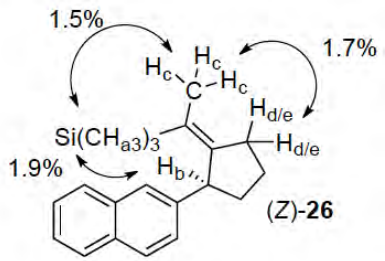
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               1.60 dB
PL12             24.60 dB
SFO2        500.2225011 MHz

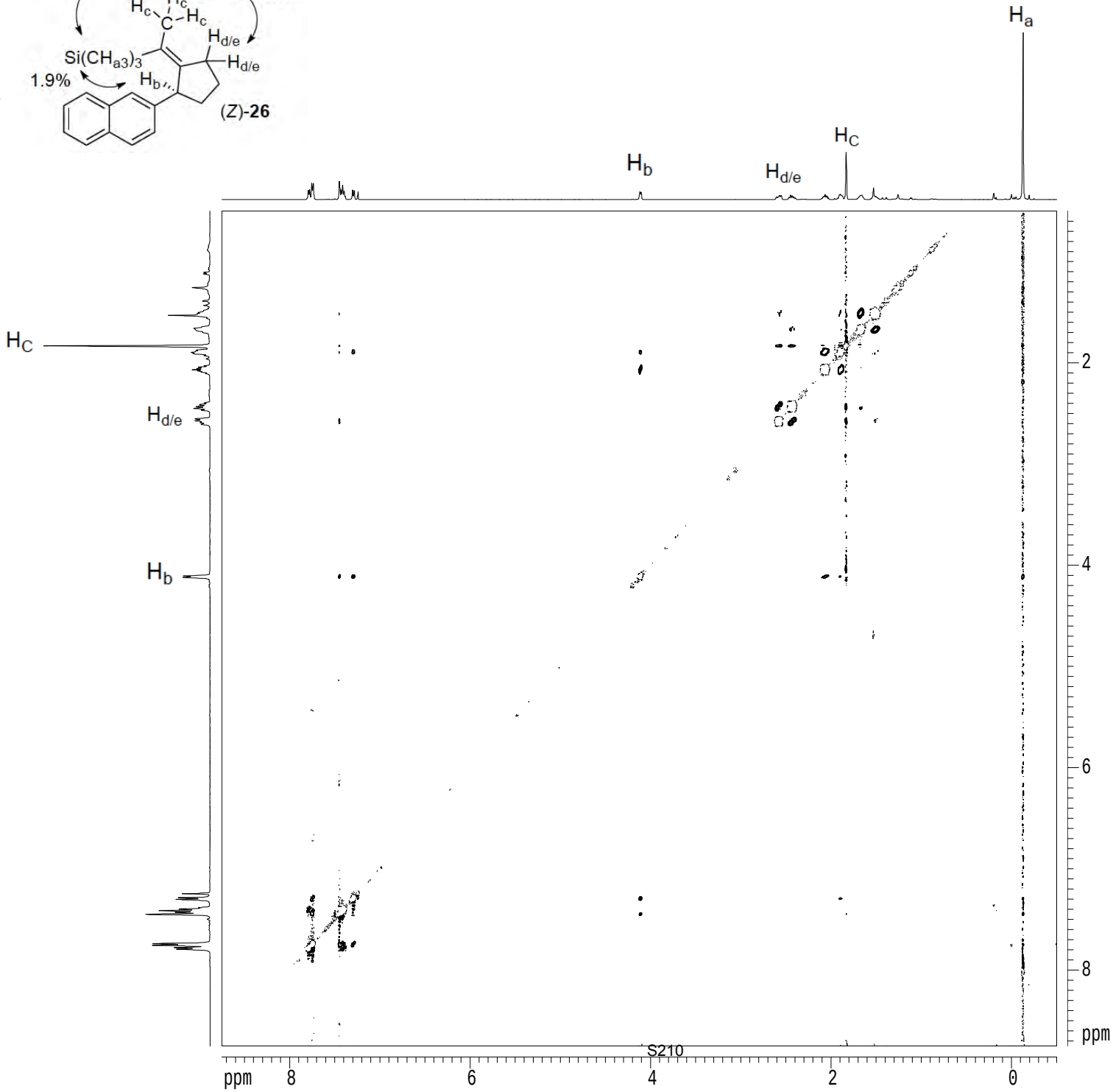
====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPX1              0.00 %
GPX2              0.00 %
GPY1              0.00 %
GPY2              0.00 %
GPZ1             30.00 %
GPZ2             50.00 %
p15             500.00 usec
p16            1000.00 usec

F2 - Processing parameters
SI                65536
SF          125.7804094 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            220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM         10.08772 ppm/cm
HZCM        1268.83752 Hz/cm
```

gnoesy






```
          Current Data Parameters
  USER              mharri
NAME      MRH-VI-84-A-1HNMR
  EXPNO                  2
  PROCNO                 1

       F2 - Acquisition Parameters
  Date_           20140127
  Time              20.30
  INSTRUM         cryo500
  PROBHD   5 mm CPTCI 1H-
  PULPROG       noesygptp
  TD                 2048
  SOLVENT           CDCl3
  NS                     2
  DS                    16
  SWH            4629.629 Hz
  FIDRES         2.260561 Hz
  AQ            0.2212340 sec
  RG                161.3
  DW              108.000 usec
  DE                 6.00 usec
  TE                298.0 K
  D0           0.00000300 sec
  D1           2.00000000 sec
  D8           0.80000001 sec
  D16          0.00020000 sec
  d20          0.39880002 sec
  IN0          0.00010800 sec

      ======== CHANNEL f1 ========
  NUC1                  1H
  P1                  7.50 usec
  P2                 15.00 usec
  PL1                 1.60 dB
  SFO1        500.2221040 MHz

     ====== GRADIENT CHANNEL =====
  GPNAM1          sine.100
  GPNAM2          sine.100
  GPX1                0.00 %
  GPX2                0.00 %
  GPY1                0.00 %
  GPY2                0.00 %
  GPZ1               40.00 %
  GPZ2              -40.00 %
  P16             1000.00 usec

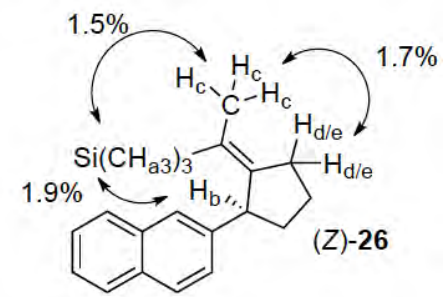
       F1 - Acquisition parameters
  ND0                    2
  TD                   512
  SFO1           500.2221 MHz
  FIDRES         9.042245 Hz
  SW                9.255 ppm
  FnMODE        undefined

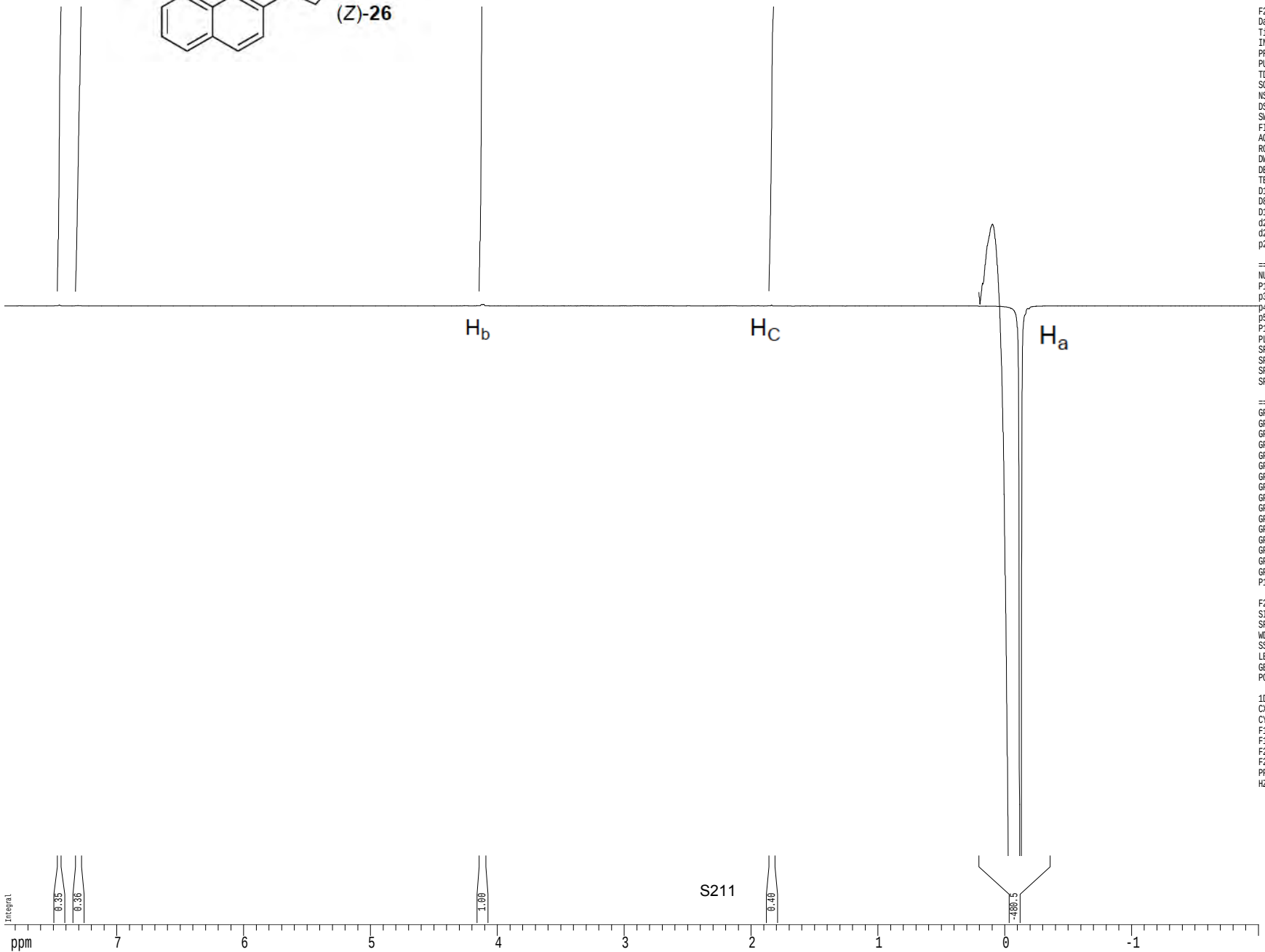
       F2 - Processing parameters
  SI                  1024
  SF           500.2200406 MHz
  WDW                 QSINE
  SSB                     2
  LB                   0.00 Hz
  GB                      0
  PC                   2.00

       F1 - Processing parameters
  SI                  1024
  MC2                 TPPI
  SF           500.2200406 MHz
  WDW                 QSINE
  SSB                     2
  LB                   0.00 Hz
  GB                      0

          2D NMR plot parameters
  CX2                15.00 cm
  CX1                15.00 cm
  F2PLO              8.750 ppm
  F2LO            4376.93 Hz
  F2PHI             -0.500 ppm
  F2HI            -250.11 Hz
  F1PLO              8.750 ppm
  F1LO            4376.93 Hz
  F1PHI              0.500 ppm
  F1HI             250.11 Hz
  F2PPMCM         0.61667 ppm/cm
  F2HZCM        308.46902 Hz/cm
  F1PPMCM         0.55000 ppm/cm
  F1HZCM        275.12103 Hz/cm
```

gnoe






```
Current Data Parameters
USER              mharri
NAME      MRH-VI-84-A-1HNMR
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140128
Time              19.07
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG      gnoe1cc.wu
TD                65536
SOLVENT           CDCl3
NS                  136
DS                    8
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ           4.0894966 sec
RG                 80.6
DW               62.400 usec
DE                6.00 usec
TE               298.0 K
D1           1.00000000 sec
D8           0.50000000 sec
D16          0.00020000 sec
d21          0.33376500 sec
d22          0.16399699 sec
p2                15.00 usec

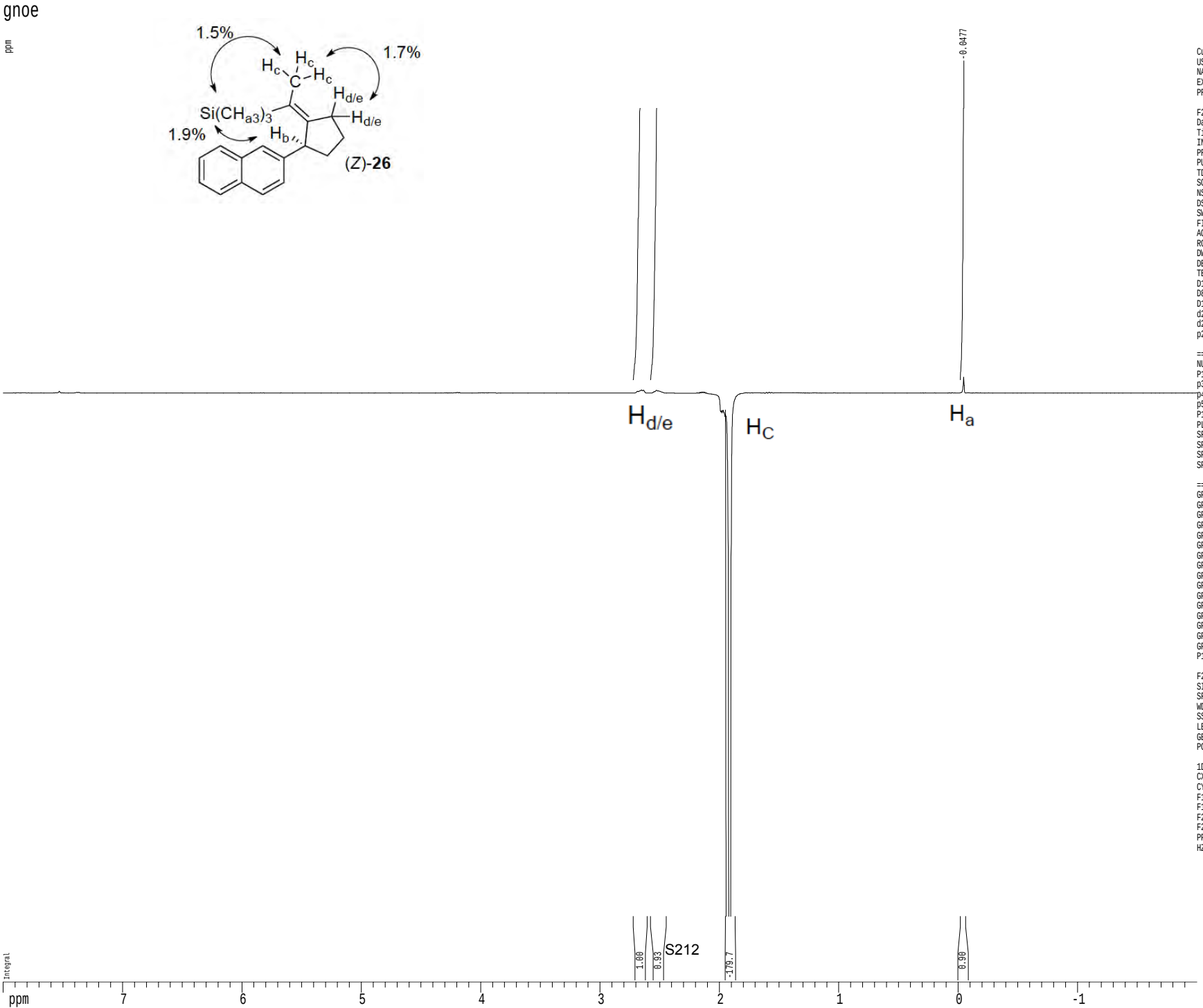
======== CHANNEL f1 ========
NUC1                 1H
P1                 7.50 usec
p3                22.50 usec
p4                30.00 usec
p5                20.00 usec
P12            40000.00 usec
PL1                1.60 dB
SFO1        500.2199763 MHz
SP1               61.60 dB
SPNAM1       gauss1.512
SPOFF1             0.00 Hz

====== GRADIENT CHANNEL =====
GPNAM1          sine.100
GPNAM2          sine.100
GPNAM3          sine.100
GPNAM4          sine.100
GPX1               0.00 %
GPX2               0.00 %
GPX3               0.00 %
GPX4               0.00 %
GPY1               0.00 %
GPY2               0.00 %
GPY3               0.00 %
GPY4               0.00 %
GPZ1               7.00 %
GPZ2               3.00 %
GPZ3               2.30 %
GPZ4              -2.30 %
P16             1000.00 usec

F2 - Processing parameters
SI                65536
SF           500.2200398 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.80 cm
CY                50.00 cm
F1P               7.882 ppm
F1             3942.88 Hz
F2P              -2.000 ppm
F2            -1000.44 Hz
PPMCM          0.43343 ppm/cm
HZCM         216.81247 Hz/cm
```

gnoe




```
Current Data Parameters
USER                mharri
NAME      MRH-VI-84-A-1HNMR
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_            20140128
Time                16.52
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG       gnoe1cc.wu
TD                  65536
SOLVENT            CDCl3T
NS                     256
DS                       8
SWH             10000.000 Hz
FIDRES           0.152588 Hz
AQ              3.2768500 sec
RG                   90.5
DW                 50.000 usec
DE                   6.00 usec
TE                  298.0 K
D1             1.00000000 sec
D8             0.50000000 sec
D16            0.00020000 sec
d21            0.33376500 sec
d22            0.16399699 sec
p2                  15.00 usec

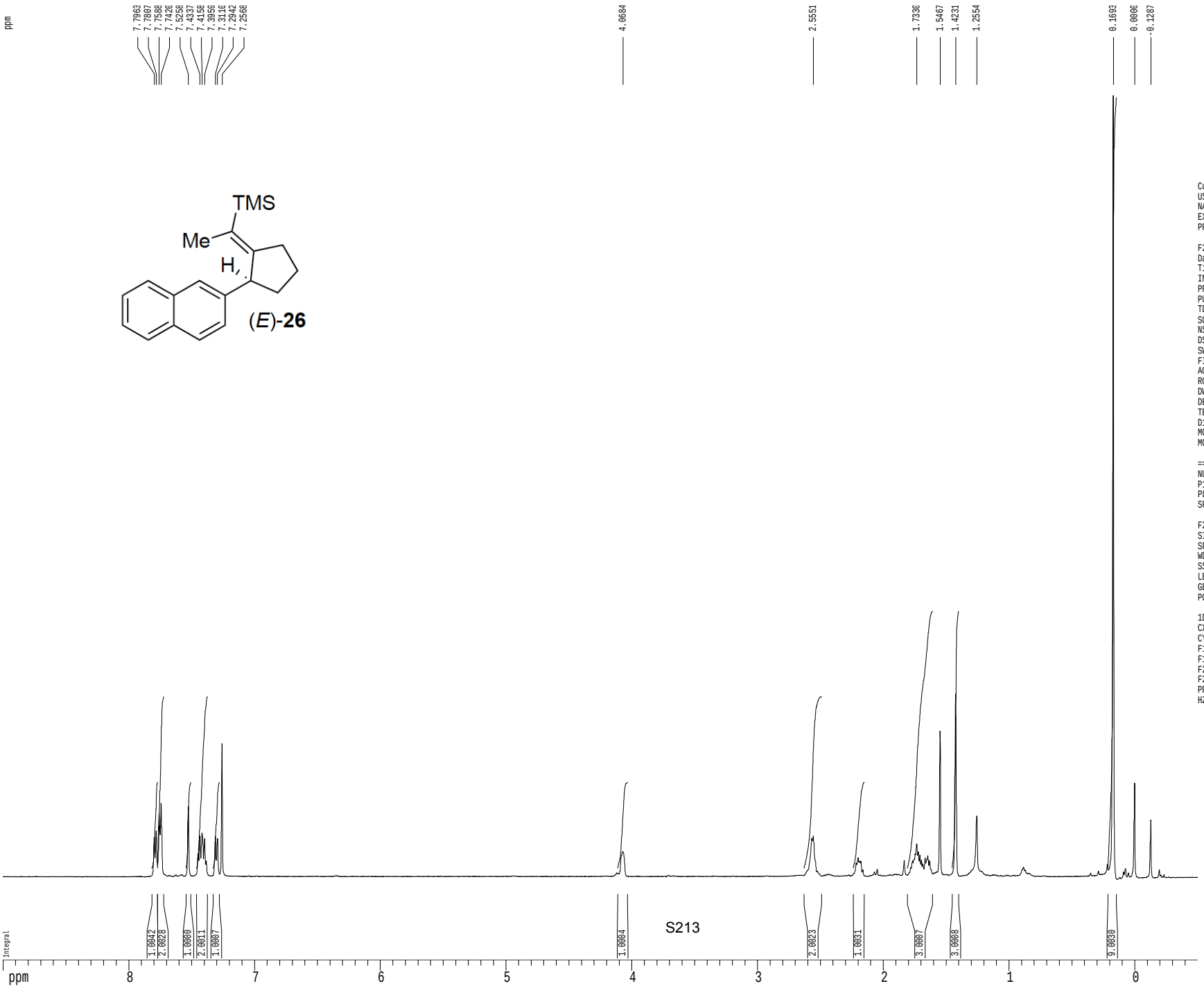
======== CHANNEL f1 ========
NUC1                   1H
P1                   7.50 usec
p3                  22.50 usec
p4                  30.00 usec
p5                  20.00 usec
P12              40000.00 usec
PL1                  1.60 dB
SFO1          500.2209557 MHz
SP1                 61.60 dB
SPNAM1        gauss1.512
SPOFF1               0.00 Hz

====== GRADIENT CHANNEL =====
GPNAM1           sine.100
GPNAM2           sine.100
GPNAM3           sine.100
GPNAM4           sine.100
GPX1                 0.00 %
GPX2                 0.00 %
GPX3                 0.00 %
GPX4                 0.00 %
GPY1                 0.00 %
GPY2                 0.00 %
GPY3                 0.00 %
GPY4                 0.00 %
GPZ1                 7.00 %
GPZ2                 3.00 %
GPZ3                 2.30 %
GPZ4                -2.30 %
P16               1000.00 usec

F2 - Processing parameters
SI                  65536
SF             500.2200000 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.80 cm
CY                  50.00 cm
F1P                 8.000 ppm
F1               4001.76 Hz
F2P                -2.000 ppm
F2              -1000.44 Hz
PPMCM            0.43860 ppm/cm
HZCM           219.39474 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-84-B
EXPNO 1
PROCNO 1

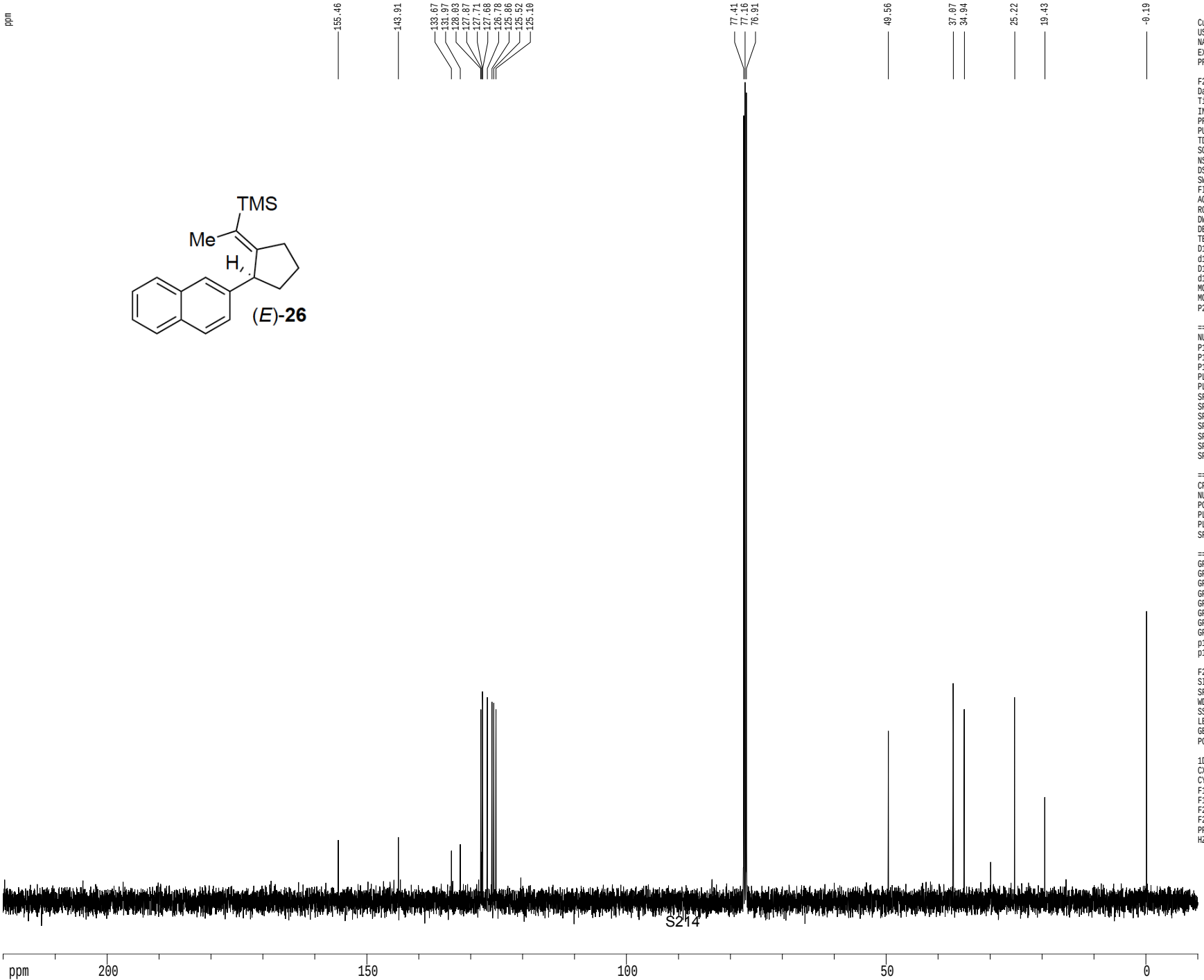
F2 - Acquisition Parameters
Date_ 20140127
Time 20.05
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200331 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-84-B-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140127
Time             20.10
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                 232
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

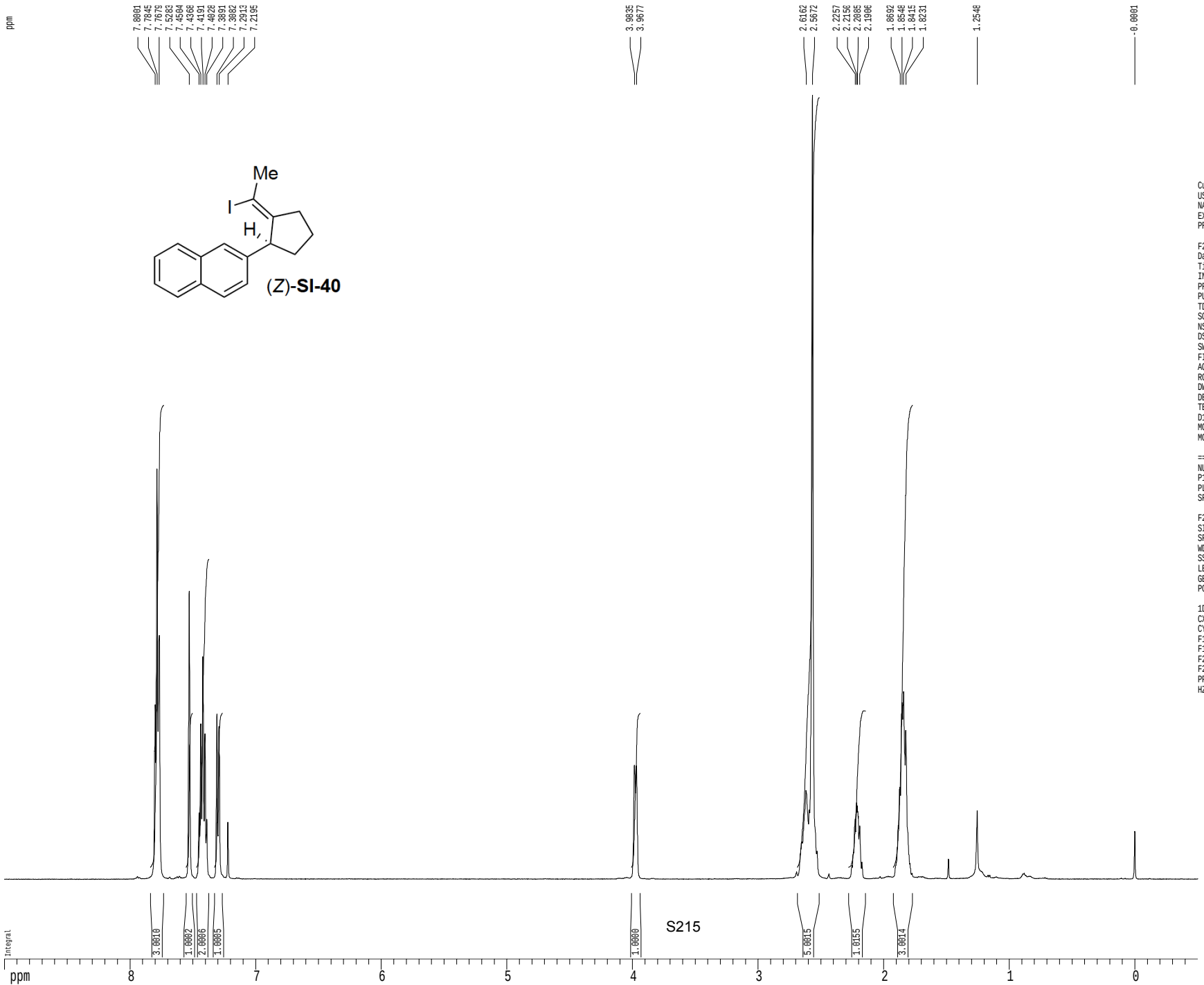
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804080 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-75-A-1HNMR
EXPNO 1
PROCNO 1

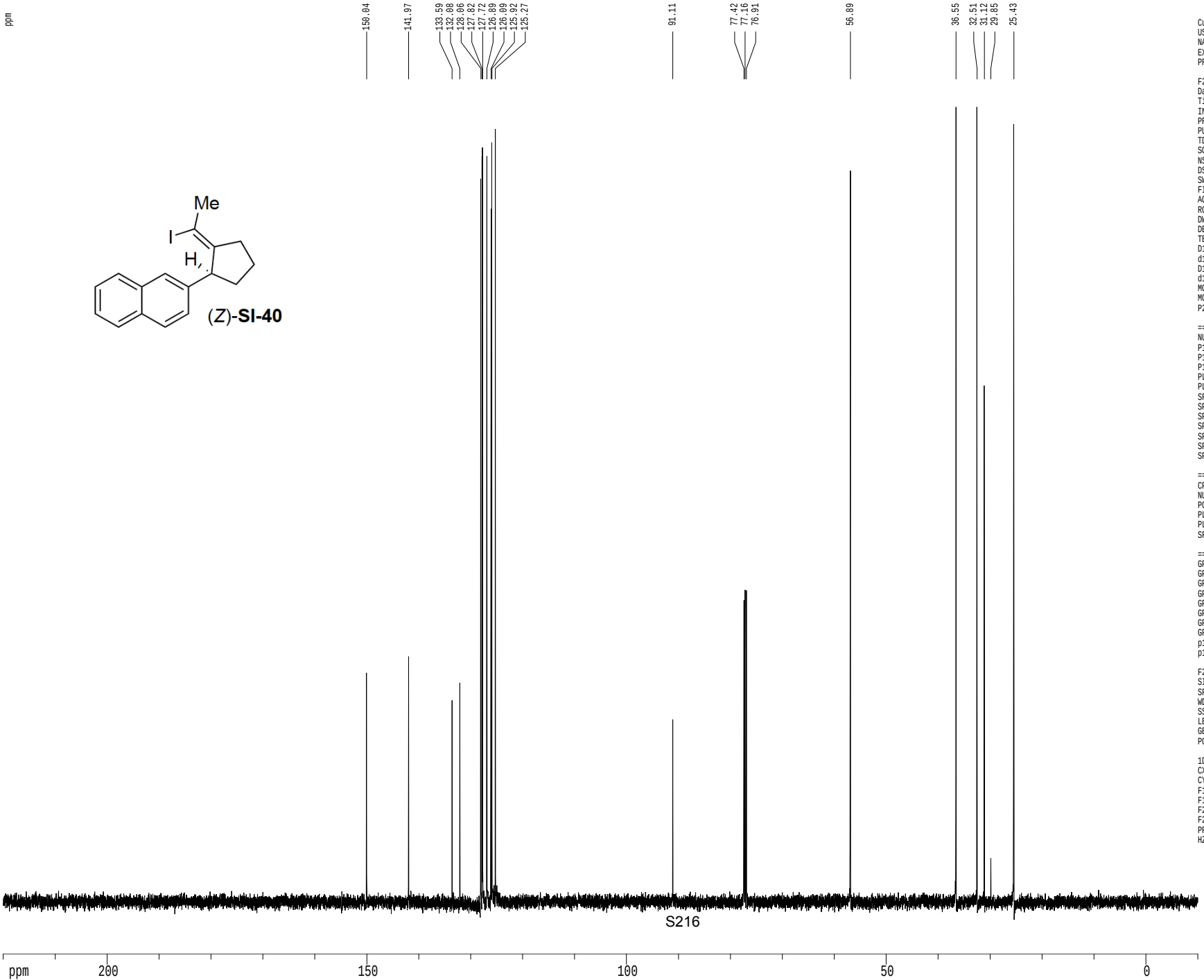
F2 - Acquisition Parameters
Date_ 20140114
Time 10.27
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 3.6
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200494 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42503 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-75-A-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140114
Time              10.30
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   91
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

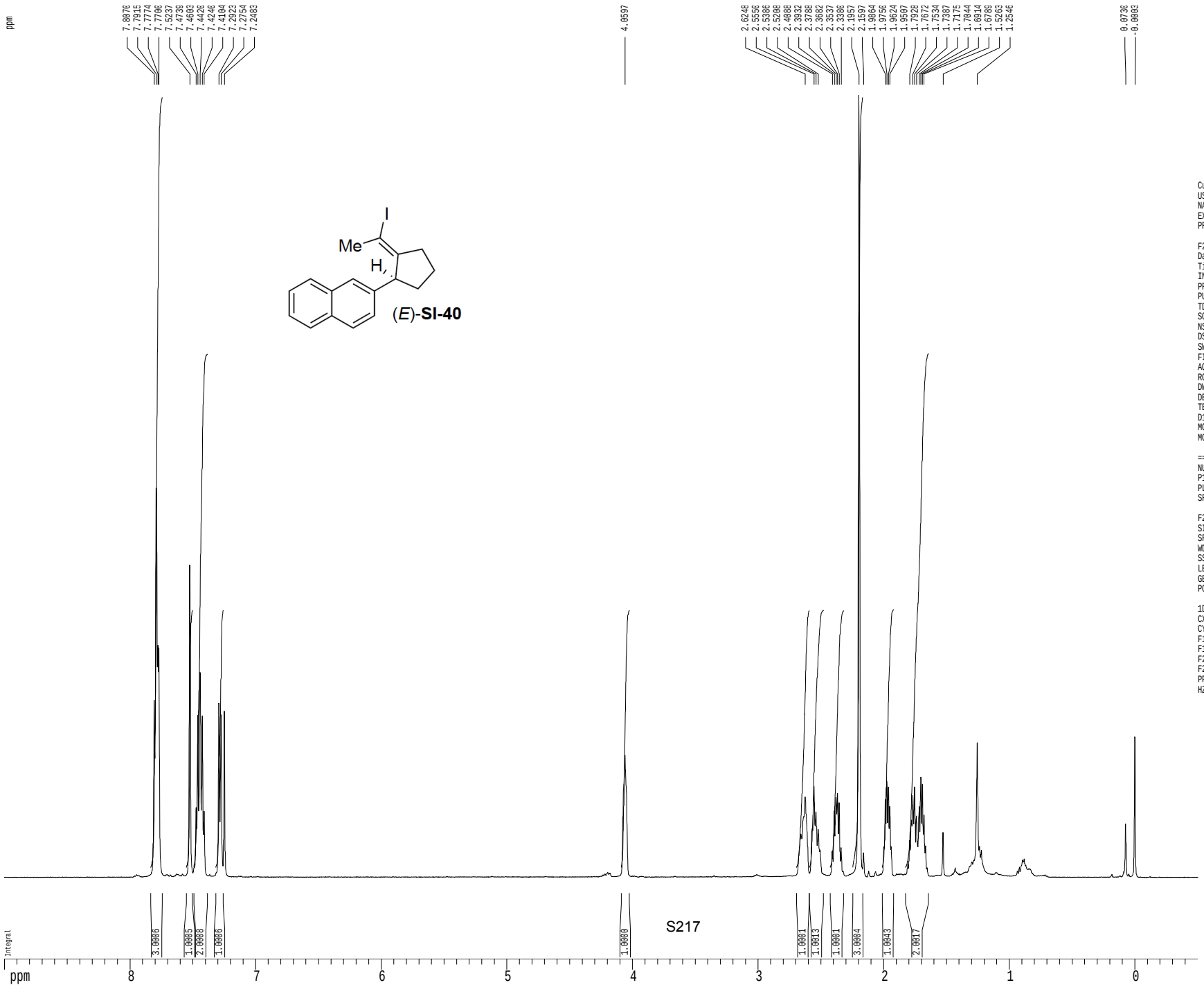
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804150 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83765 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER                mharri
NAME      MRH-VI-75-B-1HNMR
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_            20140114
Time               10.33
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT            CDCl3
NS                     3
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

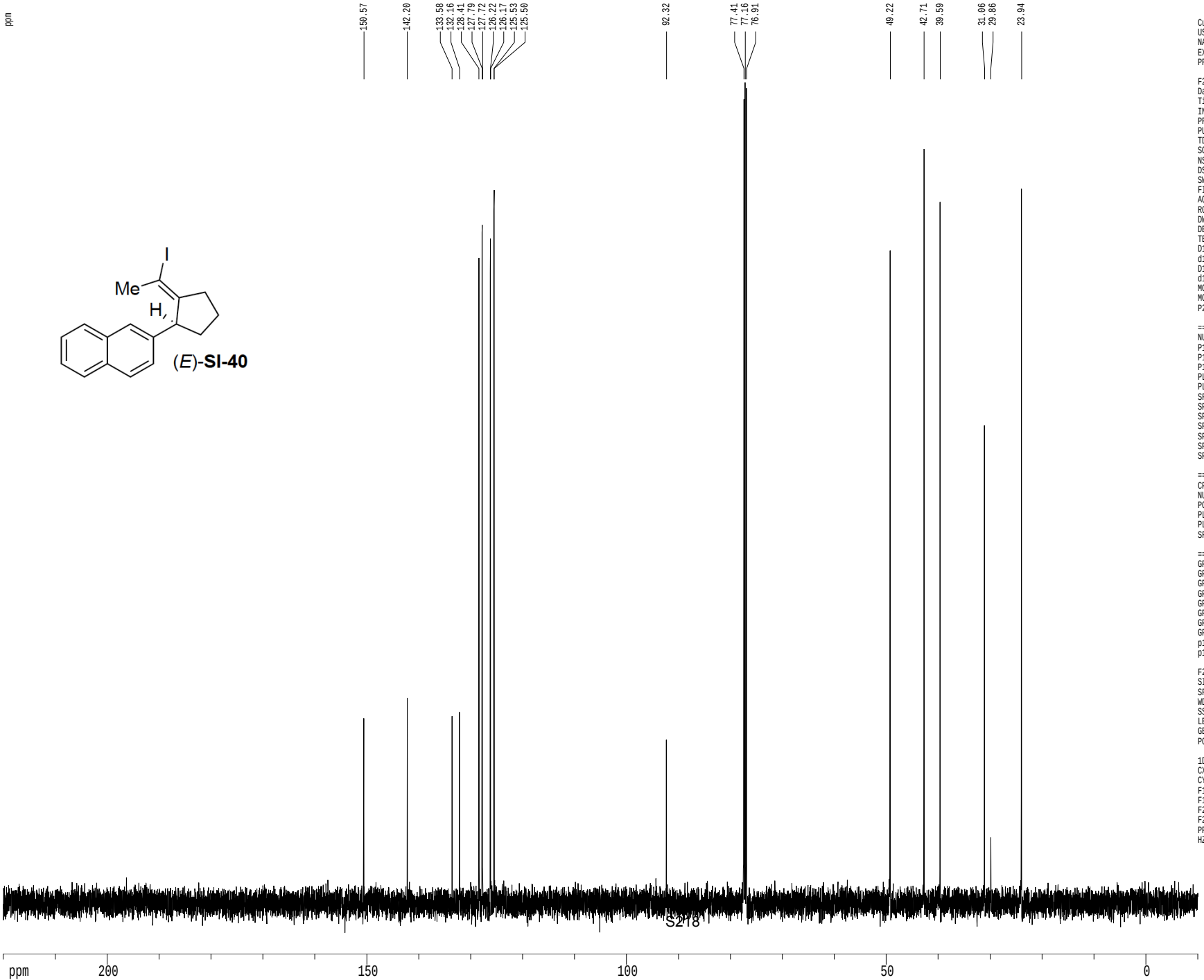
======== CHANNEL f1 ========
NUC1                  1H
P1                  7.50 usec
PL1                 1.60 dB
SFO1        500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF           500.2200352 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                9.000 ppm
F1              4501.98 Hz
F2P               -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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

(*E*)-**SI-40**




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-75-B-13CNMR
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20140114
Time             10.37
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT         CDCl3T
NS                 126
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ           1.0813940 sec
RG                6502
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               31.00 usec

======== CHANNEL f1 ========
NUC1               13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1              3.20 dB
SP2              3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2     Crp60comp.4
SPOFF1           0.00 Hz
SPOFF2           0.00 Hz

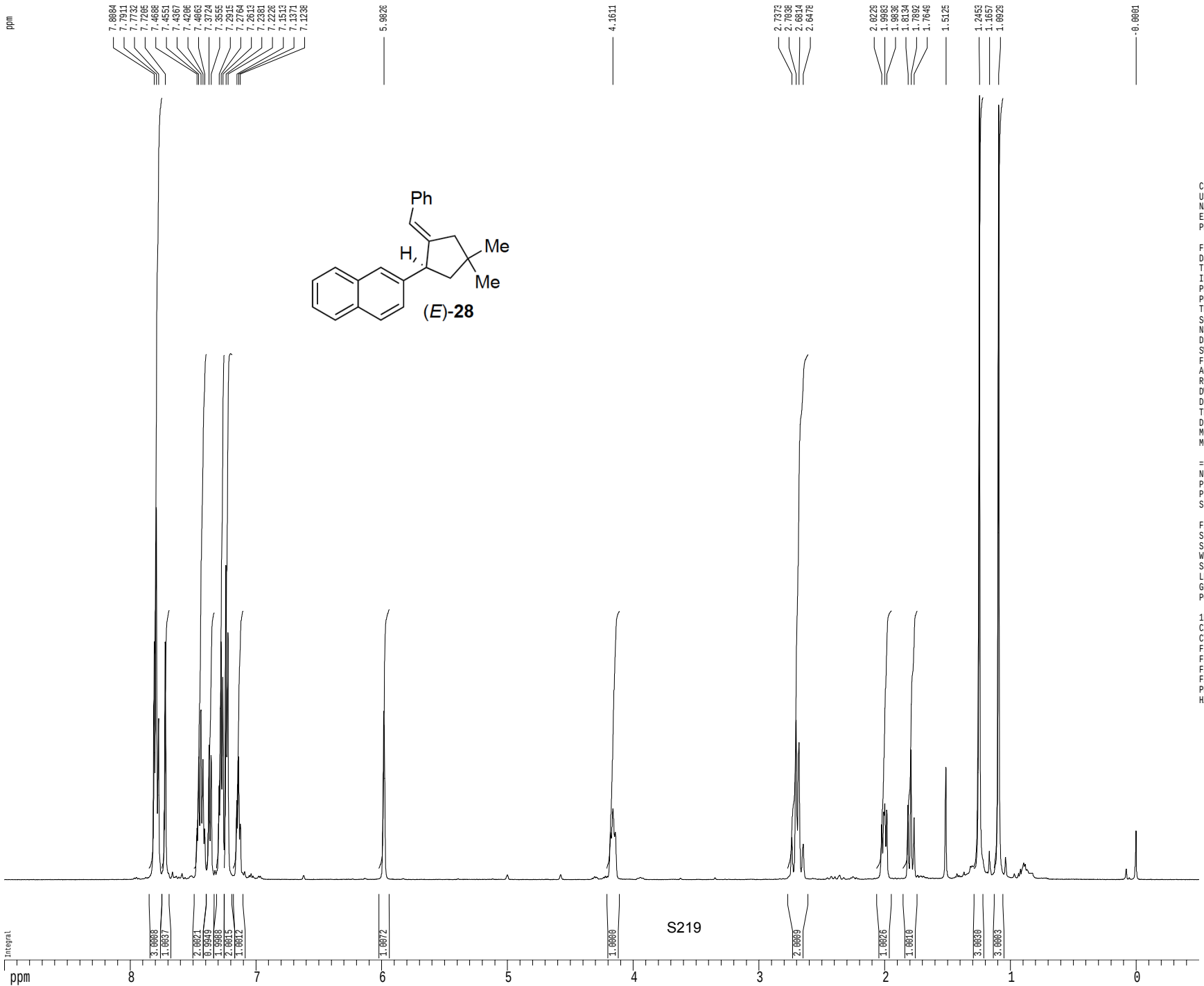
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2              1.60 dB
PL12            24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804094 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            220.000 ppm
F1           27671.69 Hz
F2P            -10.000 ppm
F2           -1257.80 Hz
PPMCM         10.08772 ppm/cm
HZCM        1268.83752 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME      MRH-VI-83-1HNMR
EXPNO                   1
PROCNO                  1

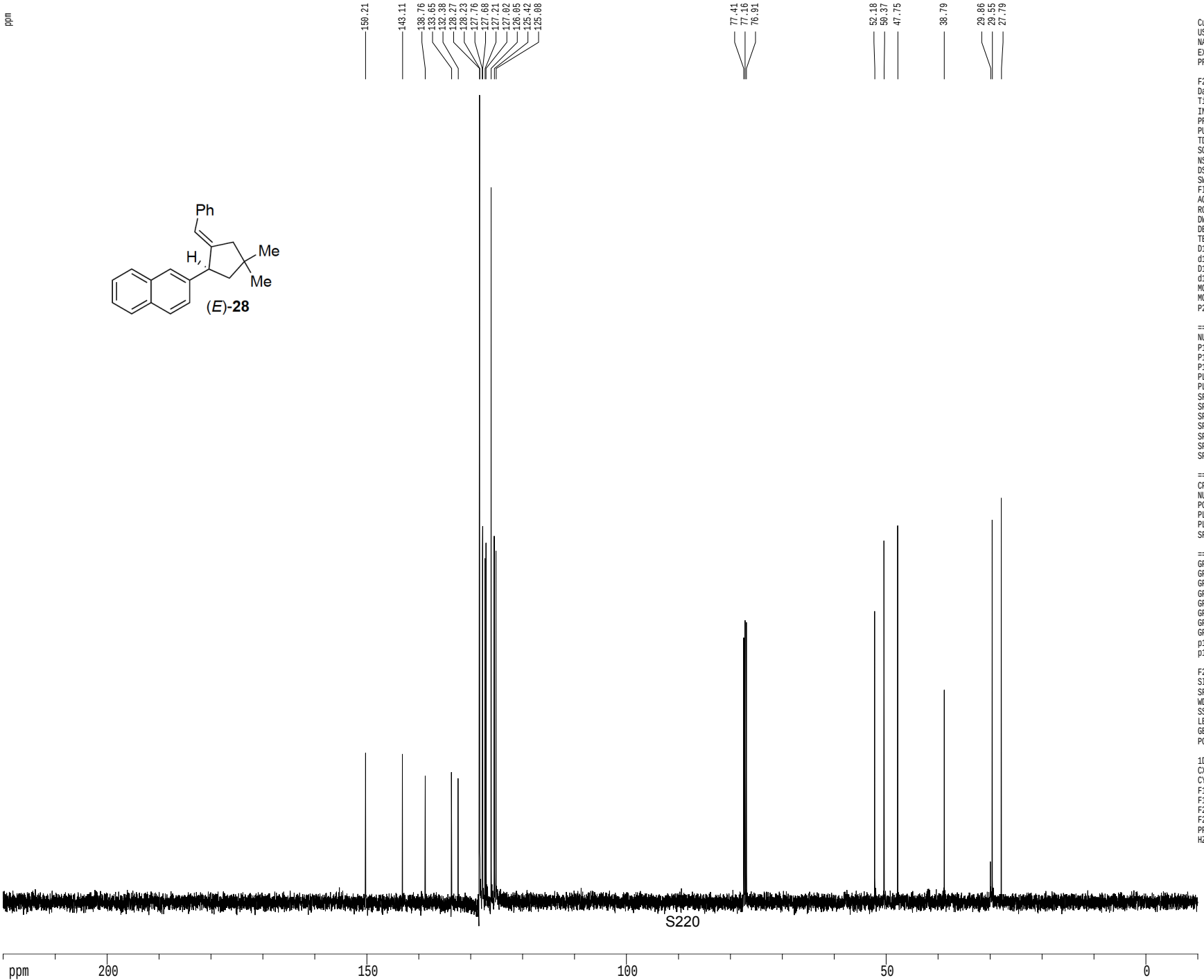
F2 - Acquisition Parameters
Date_           20140127
Time              20.14
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    2
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                    8
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          500.2200505 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42503 Hz/cm
```

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-83-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140127
Time             20.17
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                  91
DS                  16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG              10321.3
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

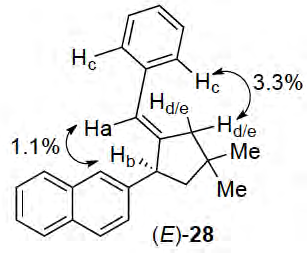
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

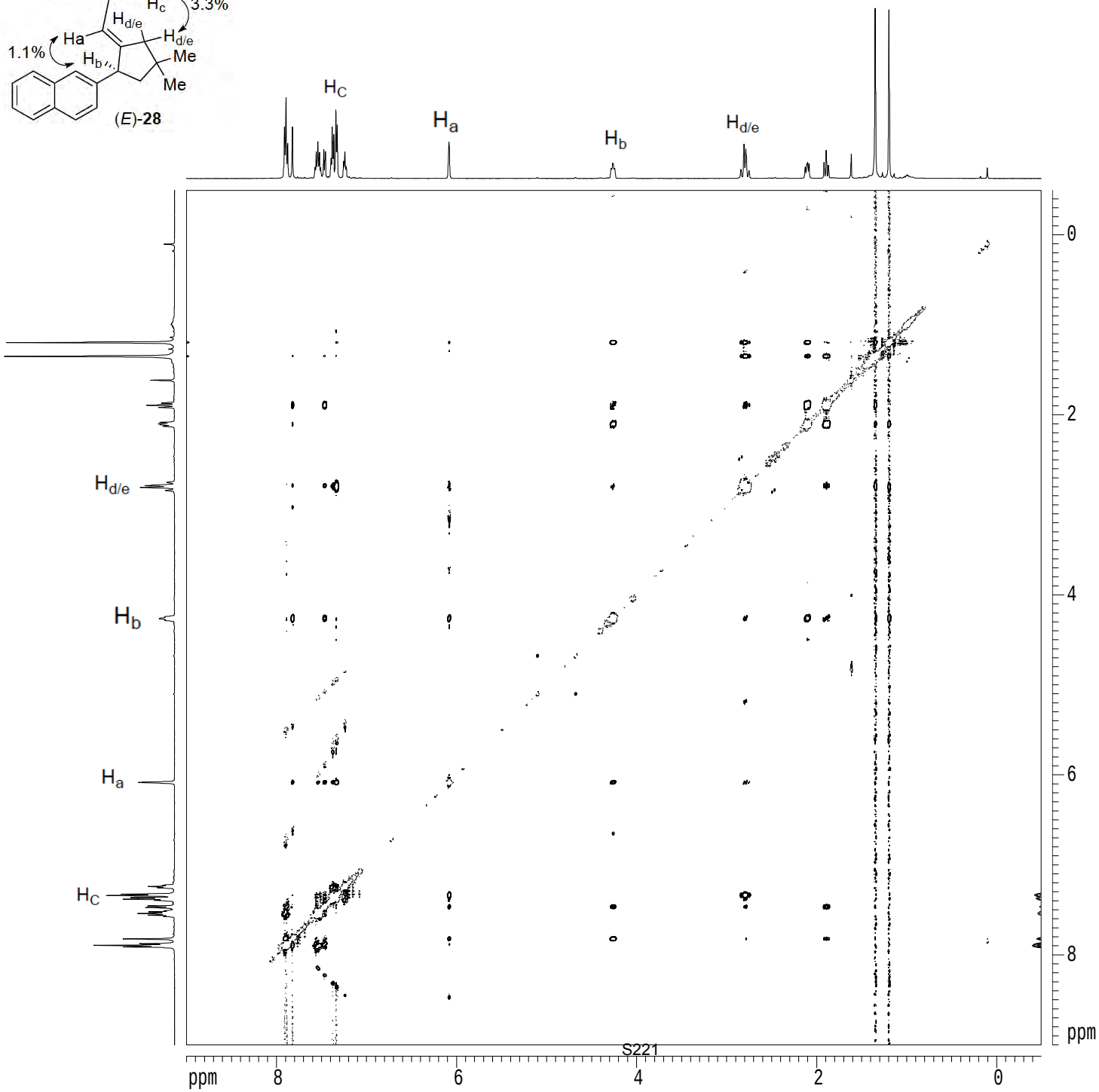
====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPX1               0.00 %
GPX2               0.00 %
GPY1               0.00 %
GPY2               0.00 %
GPZ1              30.00 %
GPZ2              50.00 %
p15             500.00 usec
p16            1000.00 usec

F2 - Processing parameters
SI                 65536
SF          125.7804131 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            220.000 ppm
F1            27671.69 Hz
F2P            -10.000 ppm
F2            -1257.80 Hz
PPMCM          10.08772 ppm/cm
HZCM         1268.83752 Hz/cm
```

gnoesy






```
        Current Data Parameters
USER              mharri
NAME      MRH-VI-83-1HNMR
EXPNO                  2
PROCNO                 1

      F2 - Acquisition Parameters
Date_           20140127
Time              21.27
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG       noesygptp
TD                 2048
SOLVENT           CDCl3
NS                     2
DS                    16
SWH            4770.992 Hz
FIDRES         2.329586 Hz
AQ           0.2146804 sec
RG                80.6
DW             104.800 usec
DE                6.00 usec
TE               298.0 K
D0           0.00000300 sec
D1           2.00000000 sec
D8           0.80000001 sec
D16          0.00020000 sec
d20          0.39880002 sec
IN0          0.00010480 sec

      ======== CHANNEL f1 =======
NUC1                  1H
P1                  7.50 usec
P2                 15.00 usec
PL1                 1.60 dB
SFO1        500.2221384 MHz

     ====== GRADIENT CHANNEL =====
GPNAM1          sine.100
GPNAM2          sine.100
GPX1               0.00 %
GPX2               0.00 %
GPY1               0.00 %
GPY2               0.00 %
GPZ1              40.00 %
GPZ2             -40.00 %
P16             1000.00 usec

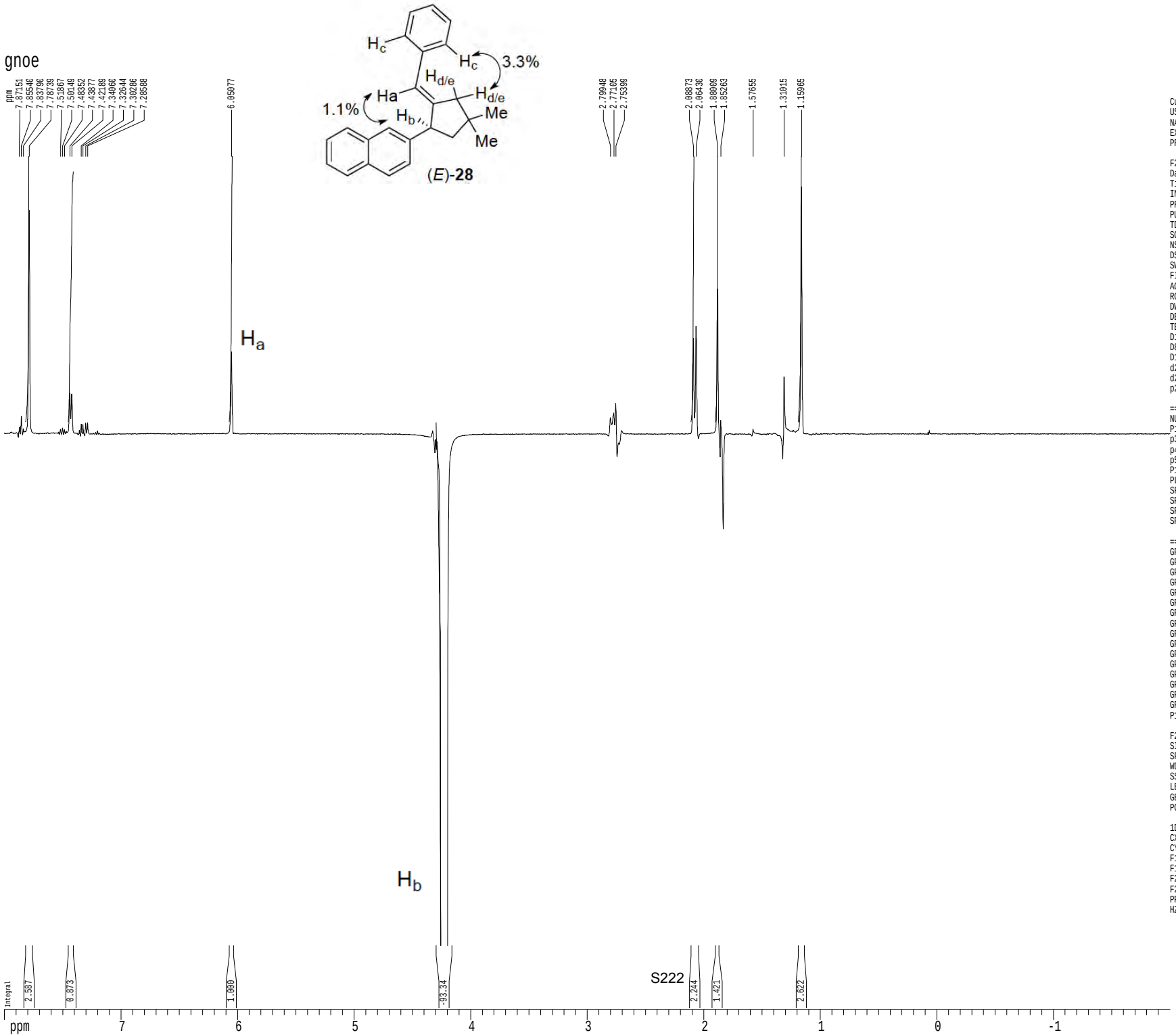
      F1 - Acquisition parameters
ND0                    2
TD                   441
SFO1           500.2221 MHz
FIDRES        10.818577 Hz
SW                9.538 ppm
FnMODE        undefined

      F2 - Processing parameters
SI                  1024
SF          500.2200000 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
PC                  2.00

      F1 - Processing parameters
SI                  1024
MC2                 TPPI
SF          500.2200000 MHz
WDW                 QSINE
SSB                    2
LB                  0.00 Hz
GB                     0

         2D NMR plot parameters
CX2                15.00 cm
CX1                15.00 cm
F2PLO              9.000 ppm
F2LO             4501.98 Hz
F2PHI             -0.494 ppm
F2HI             -247.06 Hz
F1PLO              9.000 ppm
F1LO             4501.98 Hz
F1PHI             -0.494 ppm
F1HI             -247.06 Hz
F2PPMCM          0.63293 ppm/cm
F2HZCM         316.60254 Hz/cm
F1PPMCM          0.63293 ppm/cm
F1HZCM         316.60254 Hz/cm
```

gnoe



Current Data Parameters
USER mharri
NAME MRH-VI-83-1HNMR
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140128
Time 19.25
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG gnoe1cc.wu
TD 65536
SOLVENT CDCl3T
NS 134
DS 8
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 161.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
D8 0.50000000 sec
D16 0.00020000 sec
d21 0.33376500 sec
d22 0.16399699 sec
p2 15.00 usec

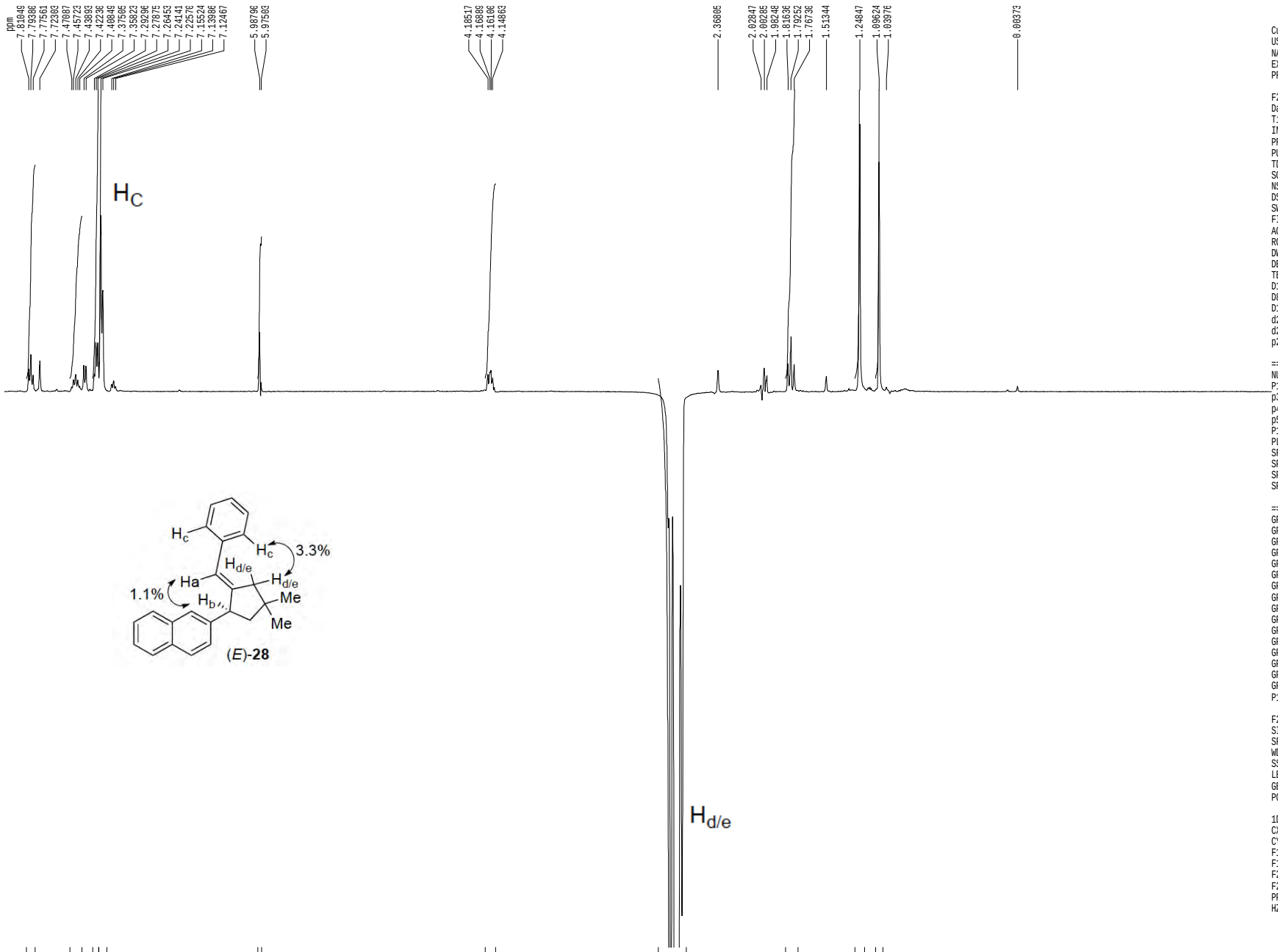
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
p3 22.50 usec
p4 30.00 usec
p5 20.00 usec
P12 40000.00 usec
PL1 1.60 dB
SFO1 500.2221318 MHz
SP1 61.60 dB
SPNAM1 gauss1.512
SPOFF1 0.00 Hz

====== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPNAM3 sine.100
GPNAM4 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPX4 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPY4 0.00 %
GPZ1 7.00 %
GPZ2 3.00 %
GPZ3 2.30 %
GPZ4 -2.30 %
P16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 50.00 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P -2.000 ppm
F2 -1000.44 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39474 Hz/cm

gnoe



Current Data Parameters
USER mharri
NAME MRH-VI-83-1HNMR
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140129
Time 11.16
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG gnoe1cc.wu
TD 65536
SOLVENT CDCl3
NS 256
DS 8
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 161.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
D8 0.50000000 sec
D16 0.00020000 sec
d21 0.33376500 sec
d22 0.16399699 sec
p2 15.00 usec

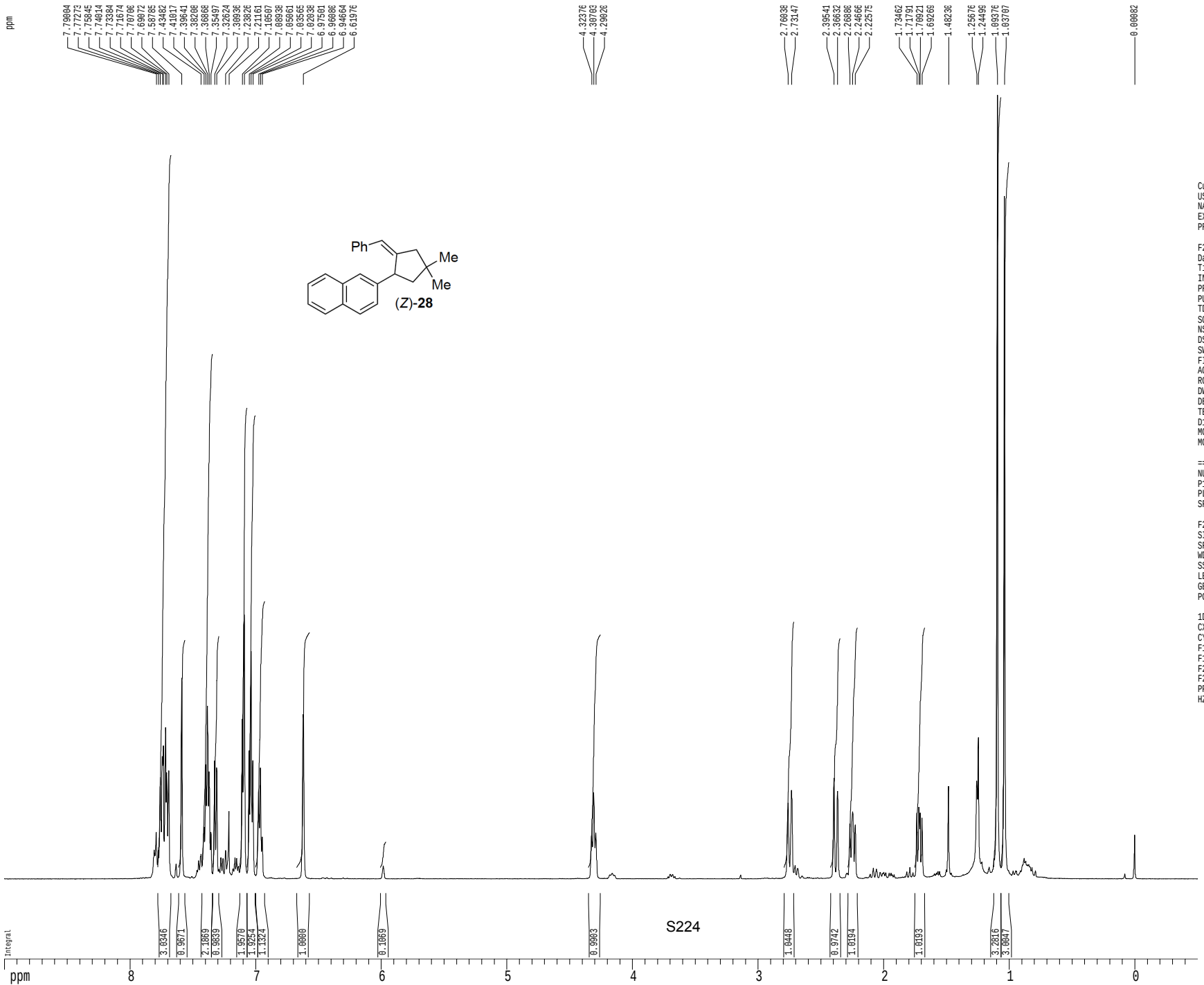
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
p3 22.50 usec
p4 30.00 usec
p5 20.00 usec
P12 40000.00 usec
PL1 1.60 dB
SFO1 500.2214132 MHz
SP1 61.60 dB
SPNAM1 gauss1.512
SPOFF1 0.00 Hz

====== GRADIENT CHANNEL =====
GPNAM1 sine.100
GPNAM2 sine.100
GPNAM3 sine.100
GPNAM4 sine.100
GPX1 0.00 %
GPX2 0.00 %
GPX3 0.00 %
GPX4 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPY3 0.00 %
GPY4 0.00 %
GPZ1 7.00 %
GPZ2 3.00 %
GPZ3 2.30 %
GPZ4 -2.30 %
P16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 500.2200506 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 50.00 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P -2.000 ppm
F2 -1000.44 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.39478 Hz/cm

1H spectrum




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-106-1HNMR
EXPNO                  1
PROCNO                 1

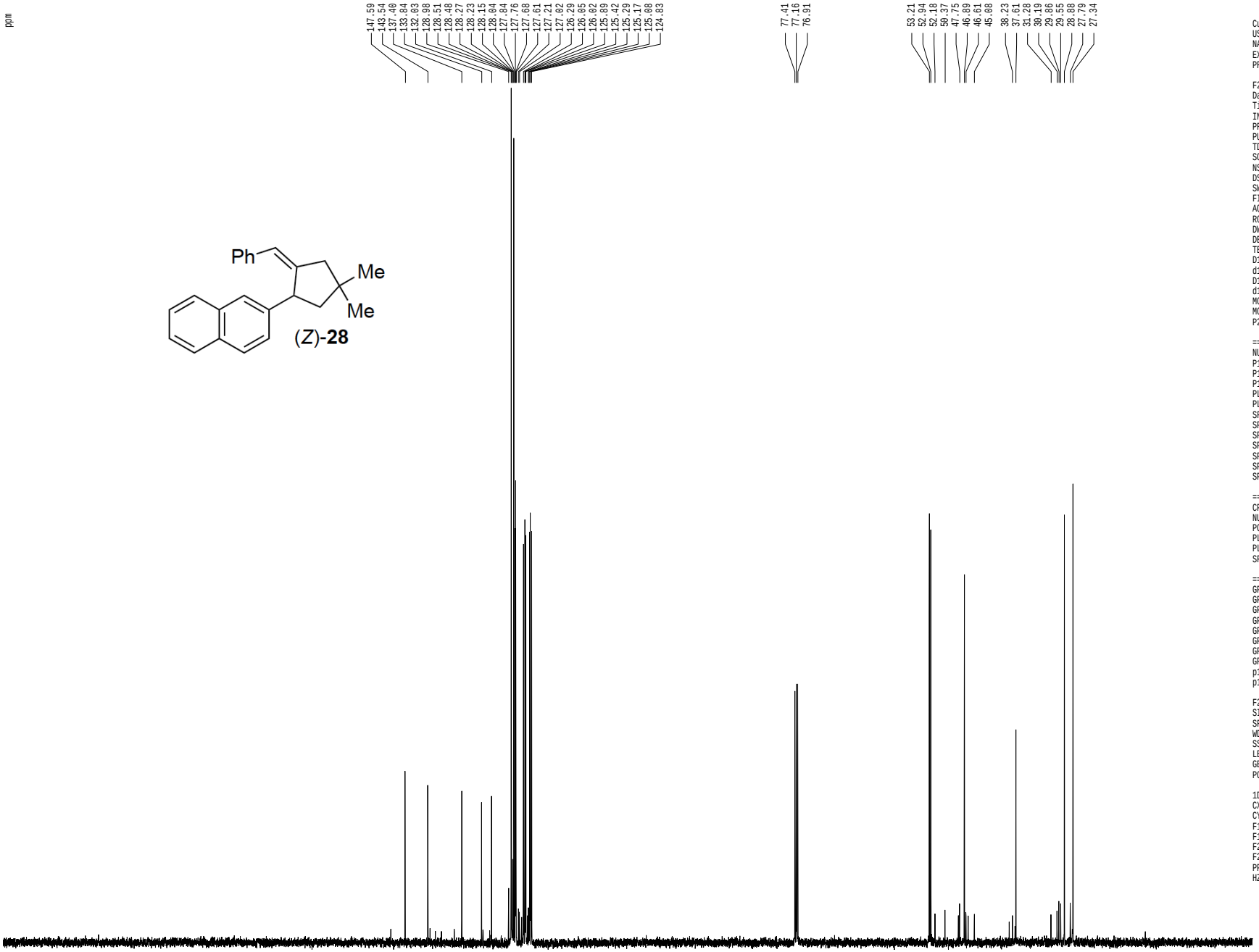
F2 - Acquisition Parameters
Date_          20140305
Time              12.04
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3T
NS                     4
DS                     2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
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          500.2200540 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                9.000 ppm
F1               4501.98 Hz
F2P               -0.500 ppm
F2               -250.11 Hz
PPMCM            0.41667 ppm/cm
HZCM           208.42503 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME      MRH-VI-106-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140305
Time               12.12
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   135
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG                2896.3
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                 31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                 15.50 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 3.20 dB
SP2                 3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1              0.00 Hz
SPOFF2              0.00 Hz

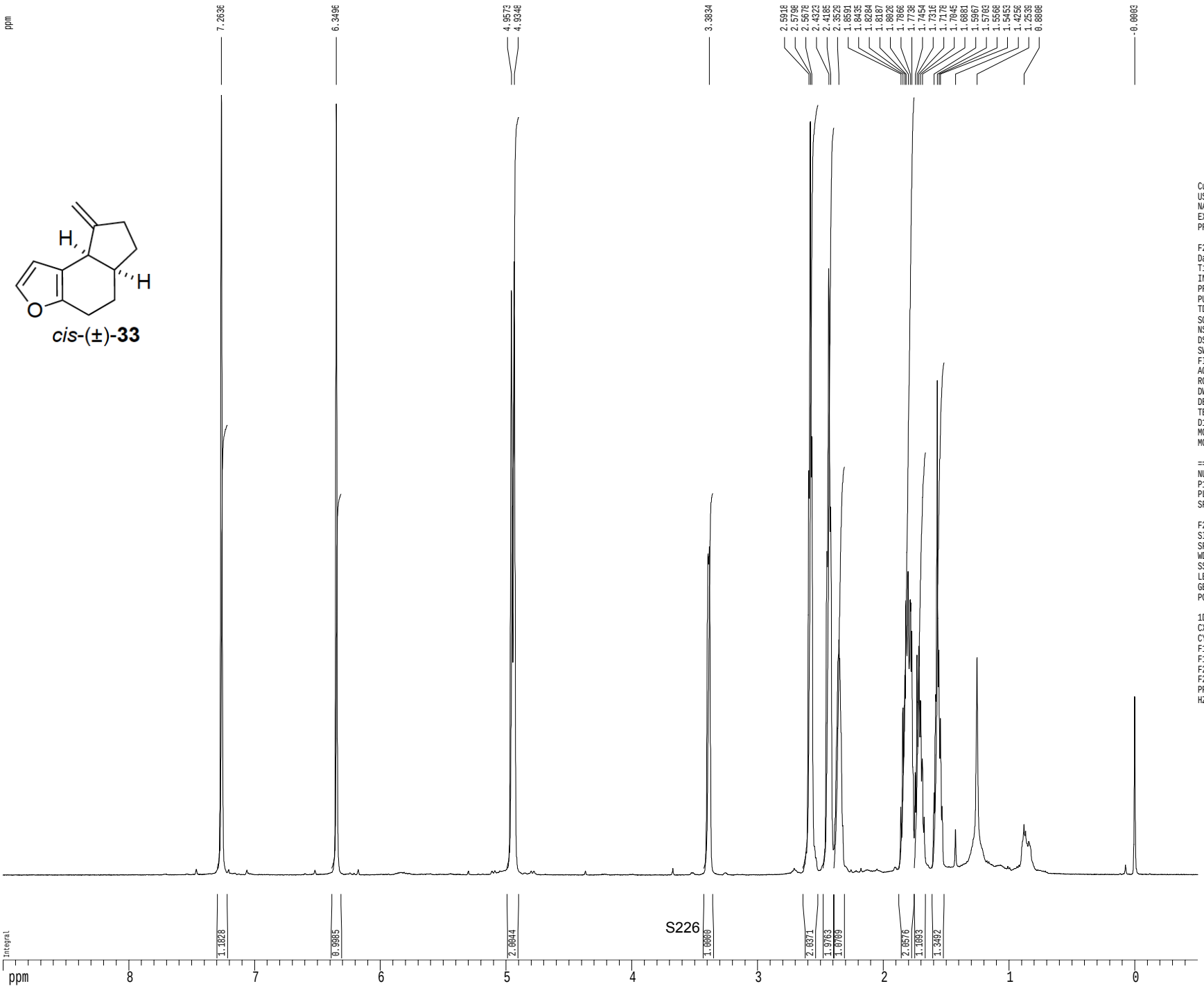
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== 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           125.7804140 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             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



cis-(±)-**33**

```
Current Data Parameters
USER              mharri
NAME     MRH-VI-110-1HNMR
EXPNO                  1
PROCNO                 1

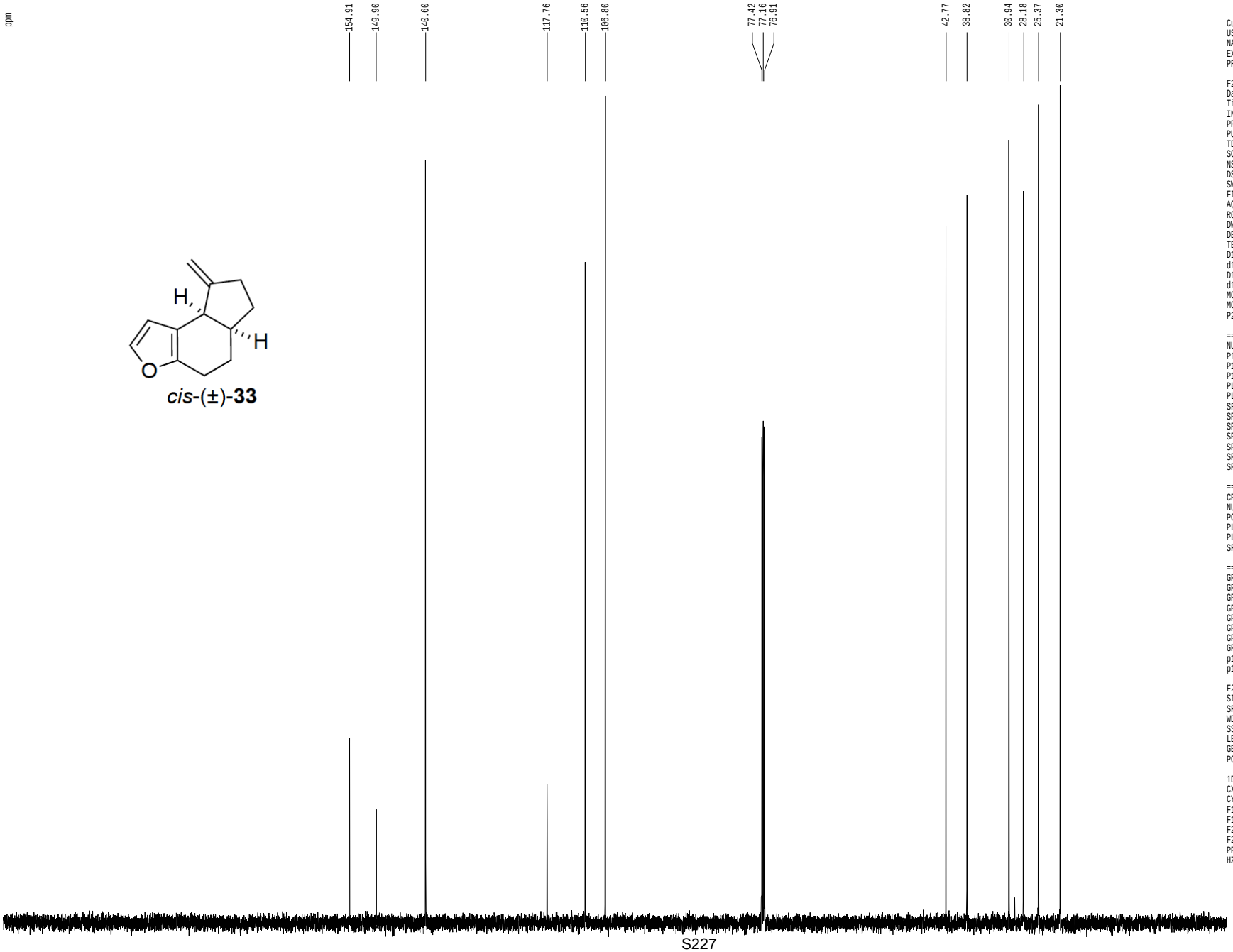
F2 - Acquisition Parameters
Date_           20140328
Time              12.12
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                  5.7
DW               62.400 usec
DE                6.00 usec
TE                290.8 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          500.2200318 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-10-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131022
Time              18.36
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
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                 31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 3.20 dB
SP2                 3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1              0.00 Hz
SPOFF2              0.00 Hz

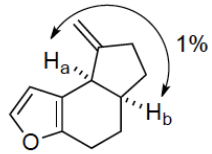
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== 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

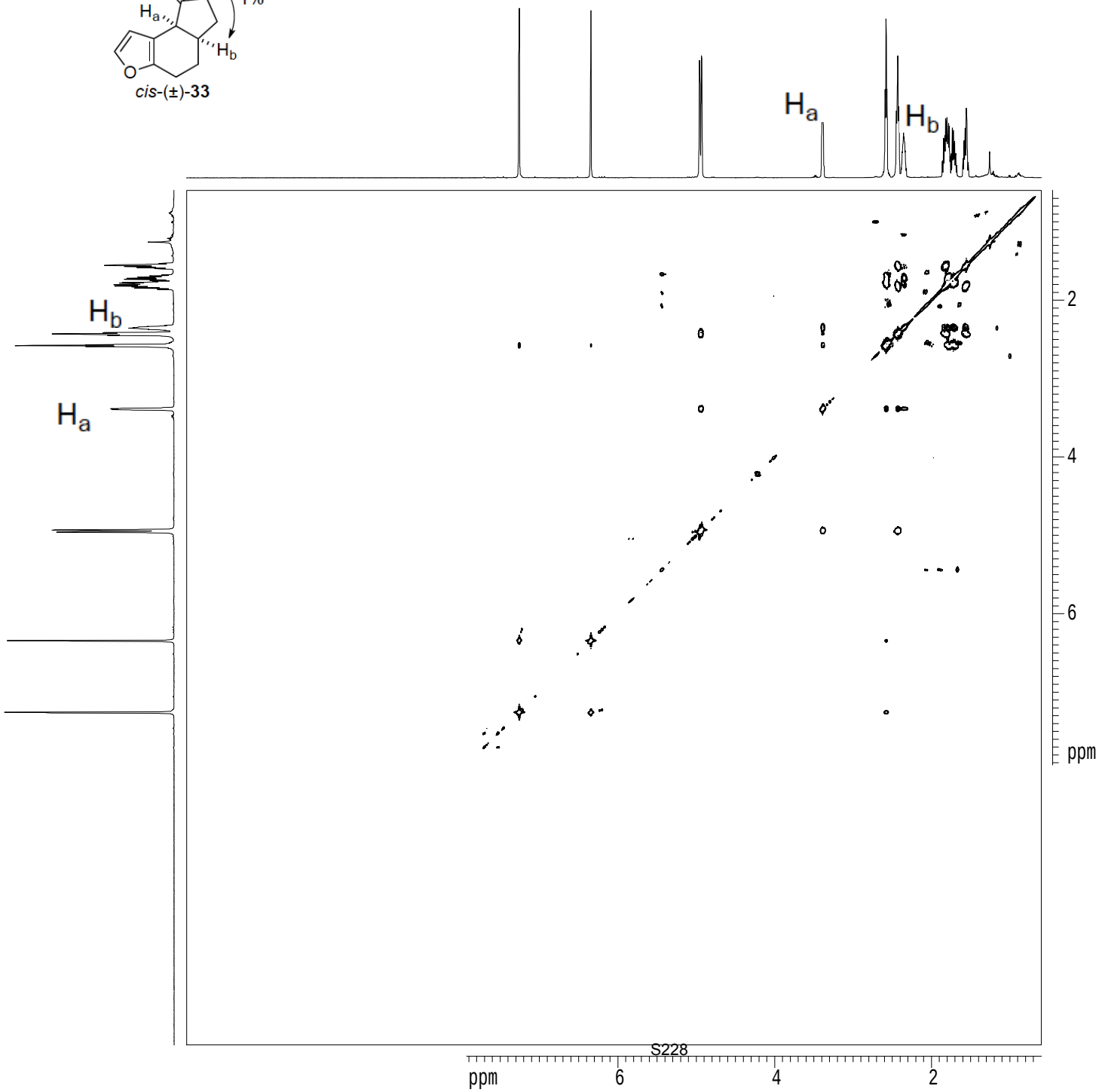
F2 - Processing parameters
SI                  65536
SF           125.7804090 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   2.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.65 cm
F1P              220.000 ppm
F1             27671.69 Hz
F2P              -10.000 ppm
F2             -1257.80 Hz
PPMCM            10.08772 ppm/cm
HZCM          1268.83752 Hz/cm
```

gcosy60



cis-(±)-**33**




```
        Current Data Parameters
 USER             mharri
NAME      MRH-VI-10-1HNMR
 EXPNO                  2
 PROCNO                 1

      F2 - Acquisition Parameters
 Date_           20131213
 Time              18.13
 INSTRUM         cryo500
 PROBHD   5 mm CPTCI 1H-
 PULPROG    cosygp60.prd
 TD                 2048
 SOLVENT          CDCl3T
 NS                    2
 DS                   16
 SWH            4496.403 Hz
 FIDRES         2.195509 Hz
 AQ            0.2277876 sec
 RG                  256
 DW              111.200 usec
 DE                6.00 usec
 TE               298.0 K
 d0           0.00000300 sec
 D1           1.00000000 sec
 d13          0.00000300 sec
 D16          0.00020000 sec
 IN0          0.00022240 sec

     ======== CHANNEL f1 =======
 NUC1                 1H
 P1                 7.50 usec
 PL1                1.60 dB
 SFO1        500.2217508 MHz

     ====== GRADIENT CHANNEL =====
 GPNAM1         sine.100
 GPNAM2         sine.100
 GPX1               0.00 %
 GPX2               0.00 %
 GPY1               0.00 %
 GPY2               0.00 %
 GPZ1              17.00 %
 GPZ2              17.00 %
 P16             1000.00 usec

      F1 - Acquisition parameters
 ND0                  1
 TD                 354
 SFO1          500.2218 MHz
 FIDRES        12.701703 Hz
 SW                8.989 ppm
 FnMODE        undefined

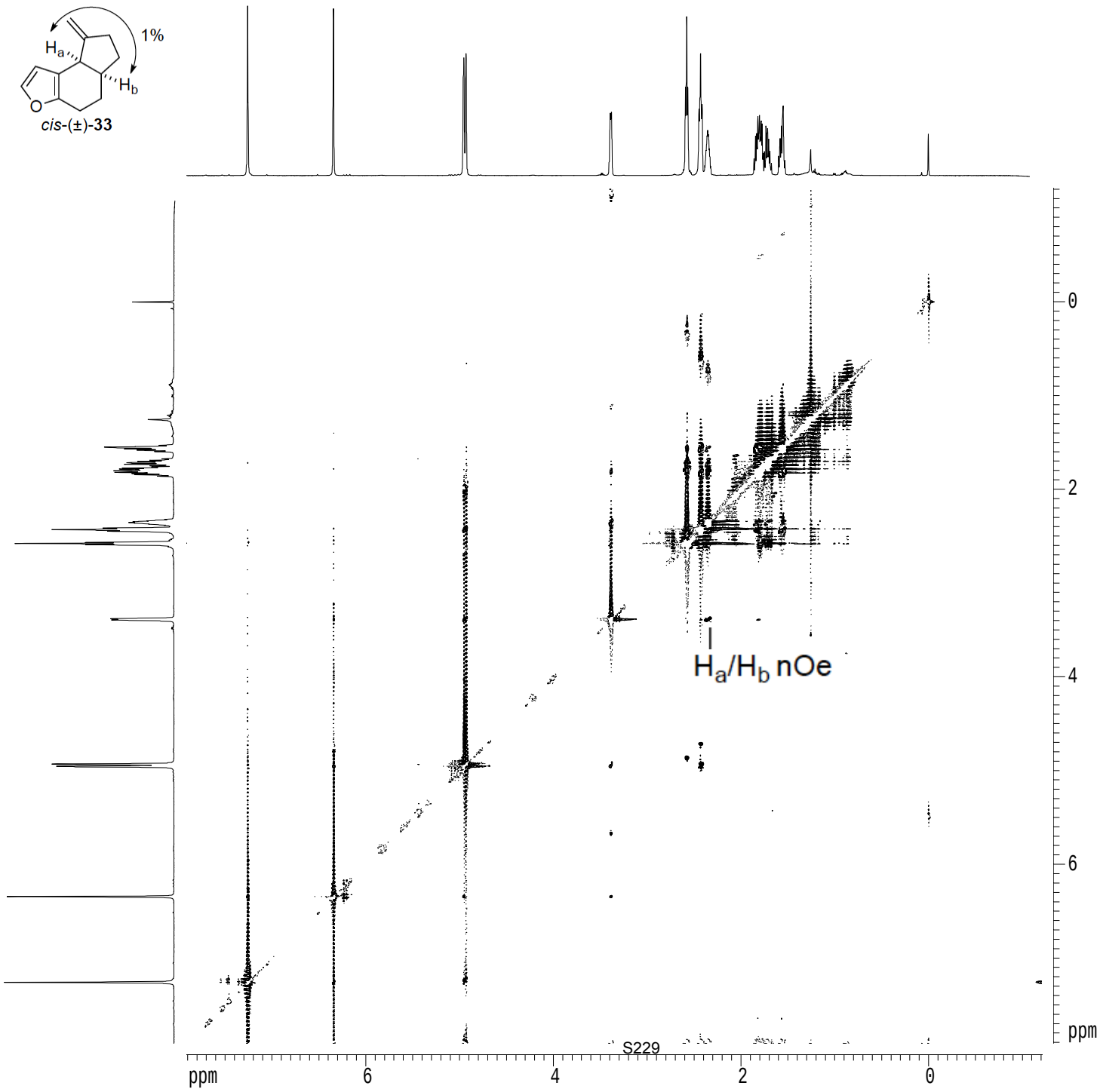
      F2 - Processing parameters
 SI                1024
 SF          500.2200341 MHz
 WDW                SINE
 SSB                   0
 LB                 0.00 Hz
 GB                    0
 PC                 4.00

      F1 - Processing parameters
 SI                1024
 MC2                 QF
 SF          500.2200341 MHz
 WDW                SINE
 SSB                   0
 LB                 0.00 Hz
 GB                    0

        2D NMR plot parameters
 CX2               15.00 cm
 CX1               15.00 cm
 F2PLO            11.500 ppm
 F2LO            5752.53 Hz
 F2PHI             0.600 ppm
 F2HI             300.13 Hz
 F1PLO            11.500 ppm
 F1LO            5752.53 Hz
 F1PHI             0.600 ppm
 F1HI             300.13 Hz
 F2PPMCM         0.72667 ppm/cm
 F2HZCM        363.49323 Hz/cm
 F1PPMCM         0.72667 ppm/cm
 F1HZCM        363.49323 Hz/cm
```

gnoesy

cis-(±)-**33**




```
        Current Data Parameters
USER              mharri
NAME      MRH-VI-10-1HNMR
EXPNO                  3
PROCNO                 1

      F2 - Acquisition Parameters
Date_           20131214
Time                9.40
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG       noesygptp
TD                 2048
SOLVENT          CDCl3T
NS                    2
DS                   16
SWH            4562.044 Hz
FIDRES         2.227561 Hz
AQ           0.2245108 sec
RG                  114
DW              109.600 usec
DE                6.00 usec
TE               298.0 K
D0           0.00000300 sec
D1           2.00000000 sec
D8           0.80000001 sec
D16          0.00020000 sec
d20          0.39880002 sec
IN0          0.00010960 sec

     ======== CHANNEL f1 ========
NUC1                 1H
P1                 7.50 usec
P2                15.00 usec
PL1                1.60 dB
SF01        500.2217091 MHz

     ====== GRADIENT CHANNEL =====
GPNAM1          sine.100
GPNAM2          sine.100
GPX1               0.00 %
GPX2               0.00 %
GPY1               0.00 %
GPY2               0.00 %
GPZ1              40.00 %
GPZ2             -40.00 %
P16             1000.00 usec

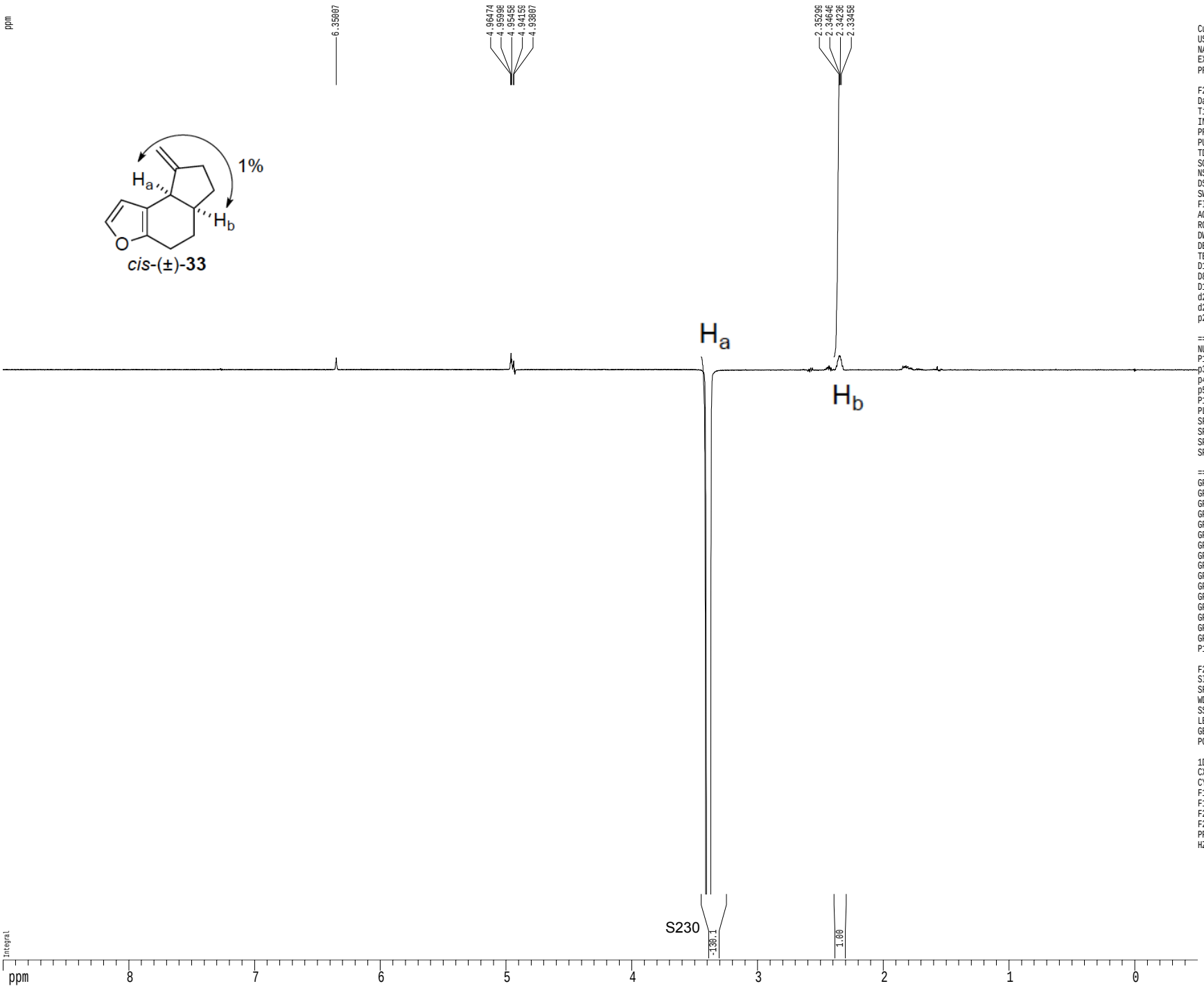
      F1 - Acquisition parameters
ND0                  2
TD                 432
SF01           500.2217 MHz
FIDRES        10.560287 Hz
SW                9.120 ppm
FnMODE        undefined

      F2 - Processing parameters
SI                 1024
SF          500.2200341 MHz
WDW                QSINE
SSB                    2
LB                 0.00 Hz
GB                     0
PC                 4.00

      F1 - Processing parameters
SI                 1024
MC2                TPPI
SF          500.2200341 MHz
WDW                QSINE
SSB                    2
LB                 0.00 Hz
GB                     0

         2D NMR plot parameters
CX2               15.00 cm
CX1               15.00 cm
F2PLO             7.909 ppm
F2LO            3956.09 Hz
F2PHI            -1.211 ppm
F2HI            -605.95 Hz
F1PLO             7.909 ppm
F1LO            3956.09 Hz
F1PHI            -1.211 ppm
F1HI            -605.95 Hz
F2PPMCM         0.60800 ppm/cm
F2HZCM        304.13626 Hz/cm
F1PPMCM         0.60800 ppm/cm
F1HZCM        304.13626 Hz/cm
```

gnoe




```
Current Data Parameters
USER              mharri
NAME      MRH-VI-110-1HNMR
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140328
Time              12.17
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG      gnoe1cc.wu
TD               65536
SOLVENT          CDCl3T
NS                 256
DS                   8
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ           4.0894966 sec
RG                 128
DW              62.400 usec
DE               6.00 usec
TE              290.9 K
D1           1.00000000 sec
D8           0.50000000 sec
D16          0.00020000 sec
d21          0.33376500 sec
d22          0.16399699 sec
p2              15.00 usec

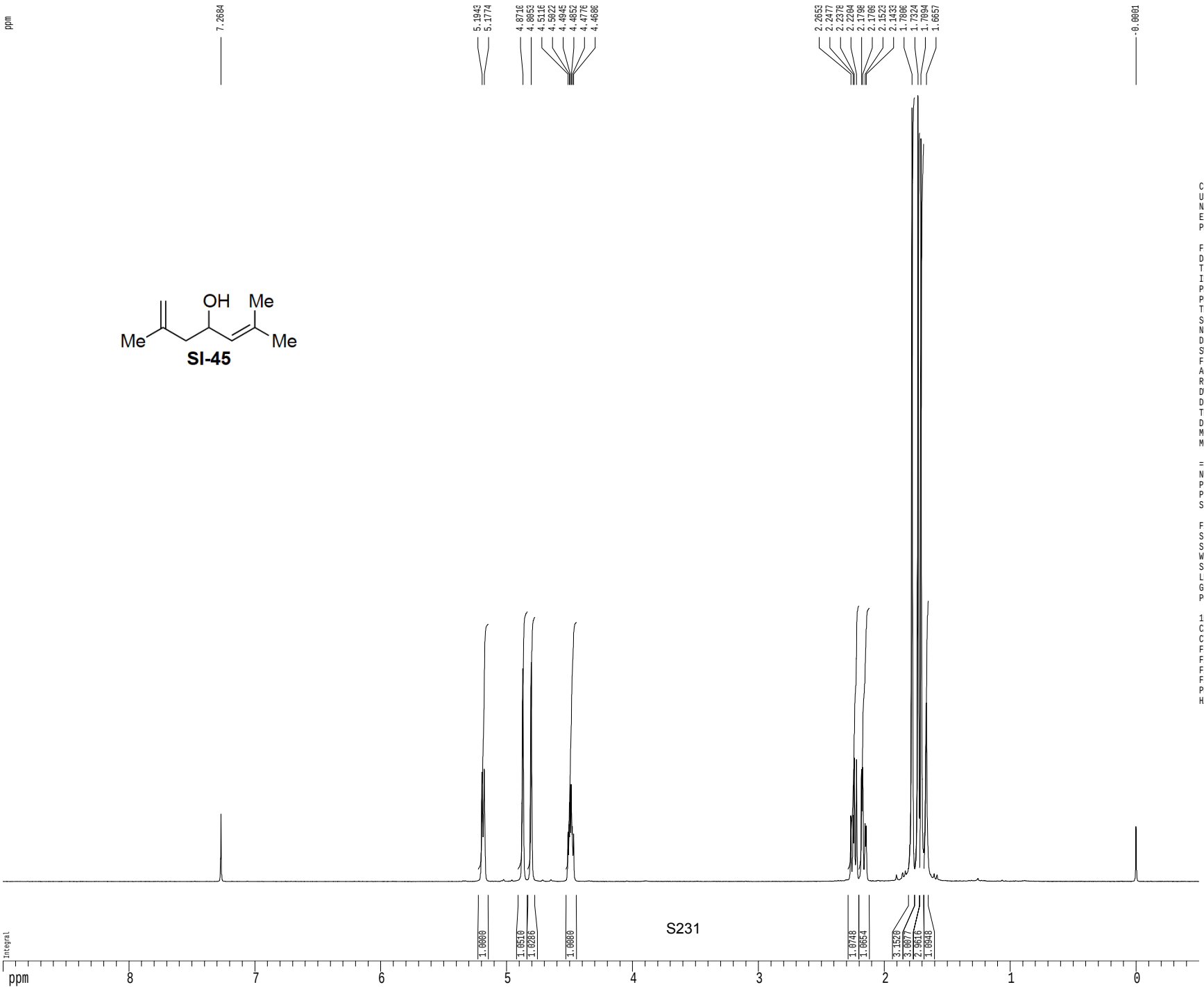
======== CHANNEL f1 ========
NUC1                1H
P1               7.50 usec
p3              22.50 usec
p4              30.00 usec
p5              20.00 usec
P12          40000.00 usec
PL1              1.60 dB
SFO1       500.2217265 MHz
SP1             61.60 dB
SPNAM1      gauss1.512
SPOFF1           0.00 Hz

====== GRADIENT CHANNEL =====
GPNAM1         sine.100
GPNAM2         sine.100
GPNAM3         sine.100
GPNAM4         sine.100
GPX1              0.00 %
GPX2              0.00 %
GPX3              0.00 %
GPX4              0.00 %
GPY1              0.00 %
GPY2              0.00 %
GPY3              0.00 %
GPY4              0.00 %
GPZ1              7.00 %
GPZ2              3.00 %
GPZ3              2.30 %
GPZ4             -2.30 %
P16           1000.00 usec

F2 - Processing parameters
SI                65536
SF         500.2200310 MHz
WDW                  no
SSB                   0
LB                0.00 Hz
GB                    0
PC                1.00

1D NMR plot parameters
CX               22.80 cm
CY               50.00 cm
F1P               9.000 ppm
F1             4501.98 Hz
F2P              -0.500 ppm
F2             -250.11 Hz
PPMCM          0.41667 ppm/cm
HZCM         208.42502 Hz/cm
```

1H spectrum



SI-45

```
Current Data Parameters
USER              mharri
NAME     MRH-VI-21-1HNMR
EXPNO                  1
PROCNO                 1

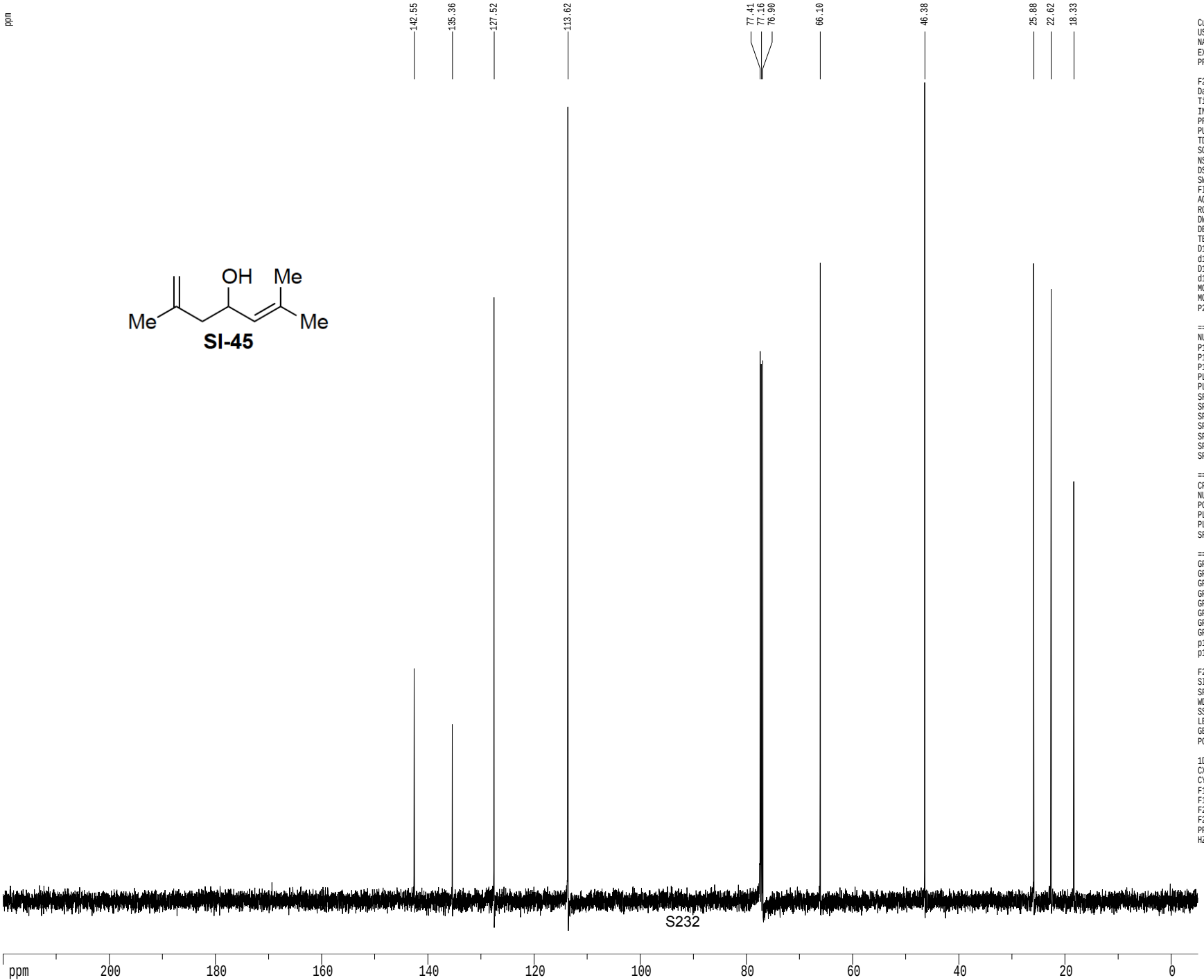
F2 - Acquisition Parameters
Date_           20131209
Time              15.48
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    1
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            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          500.2200266 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-21-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20131209
Time              15.50
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   66
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG                13004
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

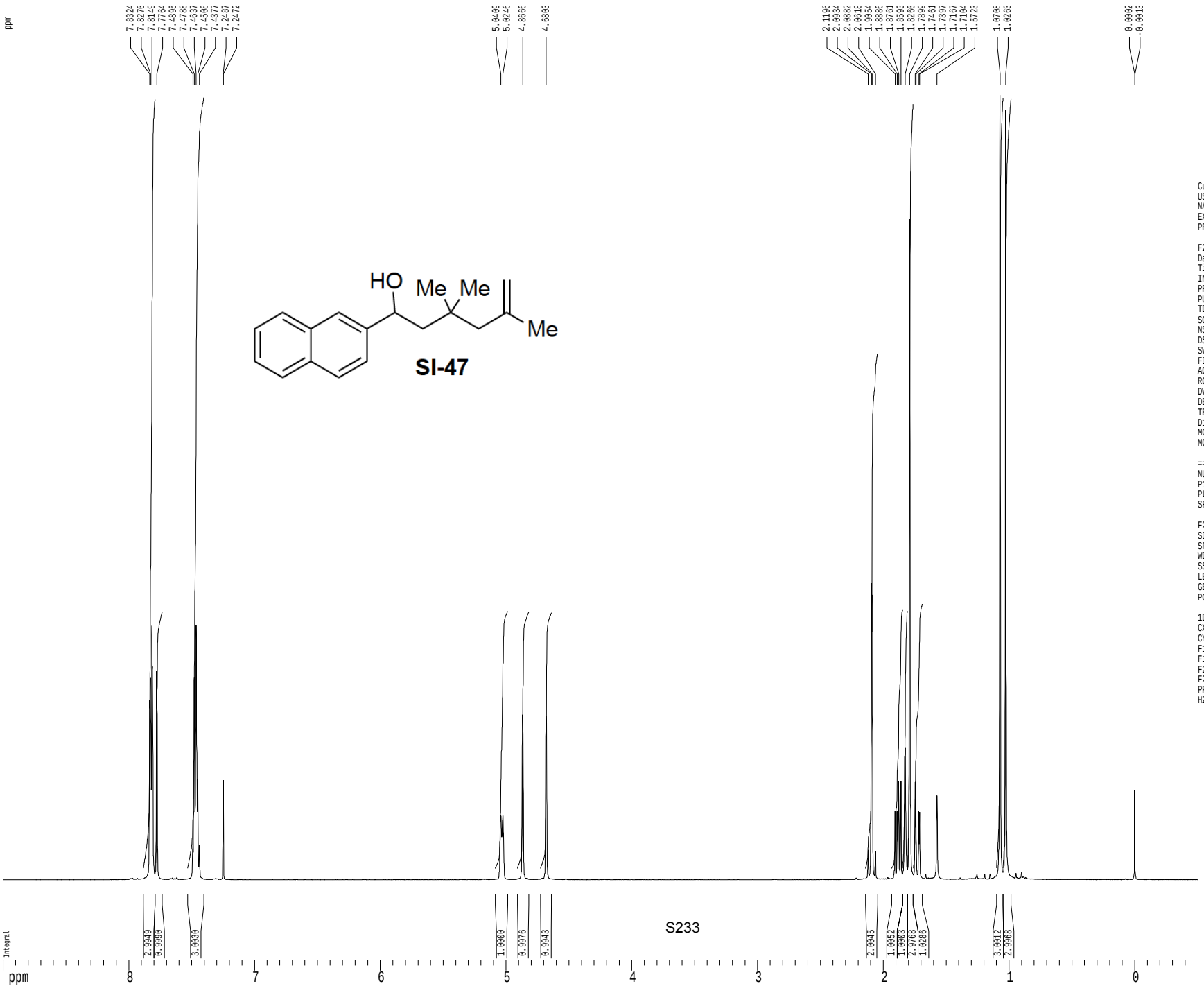
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804099 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            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM          9.86842 ppm/cm
HZCM        1241.25415 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME      MRH-V-235-1HNMR
EXPNO                   1
PROCNO                  1

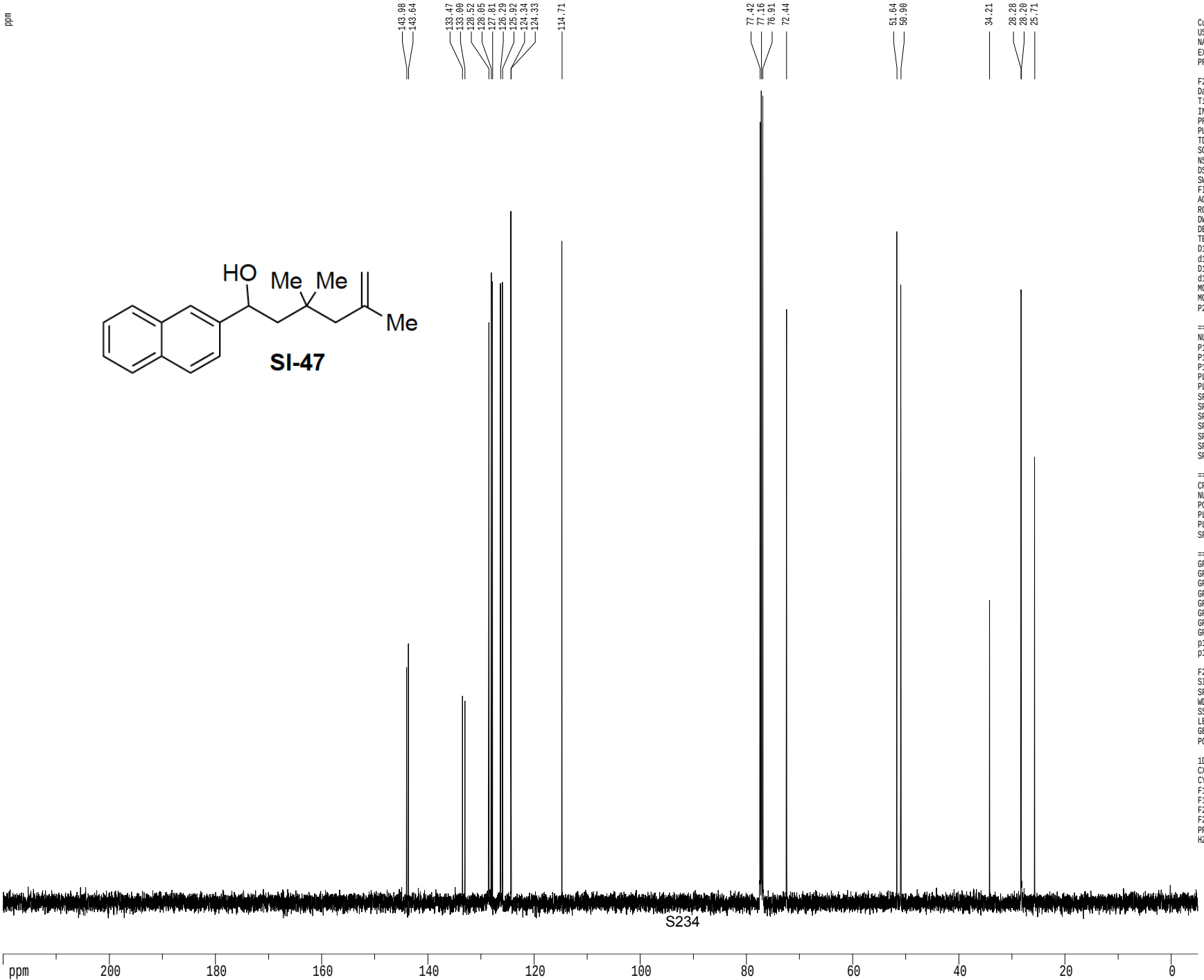
F2 - Acquisition Parameters
Date_           20140508
Time              19.29
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            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          500.2200382 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-235-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140508
Time             19.32
INSTRUM        cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD               65536
SOLVENT          CDCl3
NS                 125
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               31.00 usec

======== CHANNEL f1 =======
NUC1               13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1               3.20 dB
SP2               3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2     Crp60comp.4
SPOFF1            0.00 Hz
SPOFF2            0.00 Hz

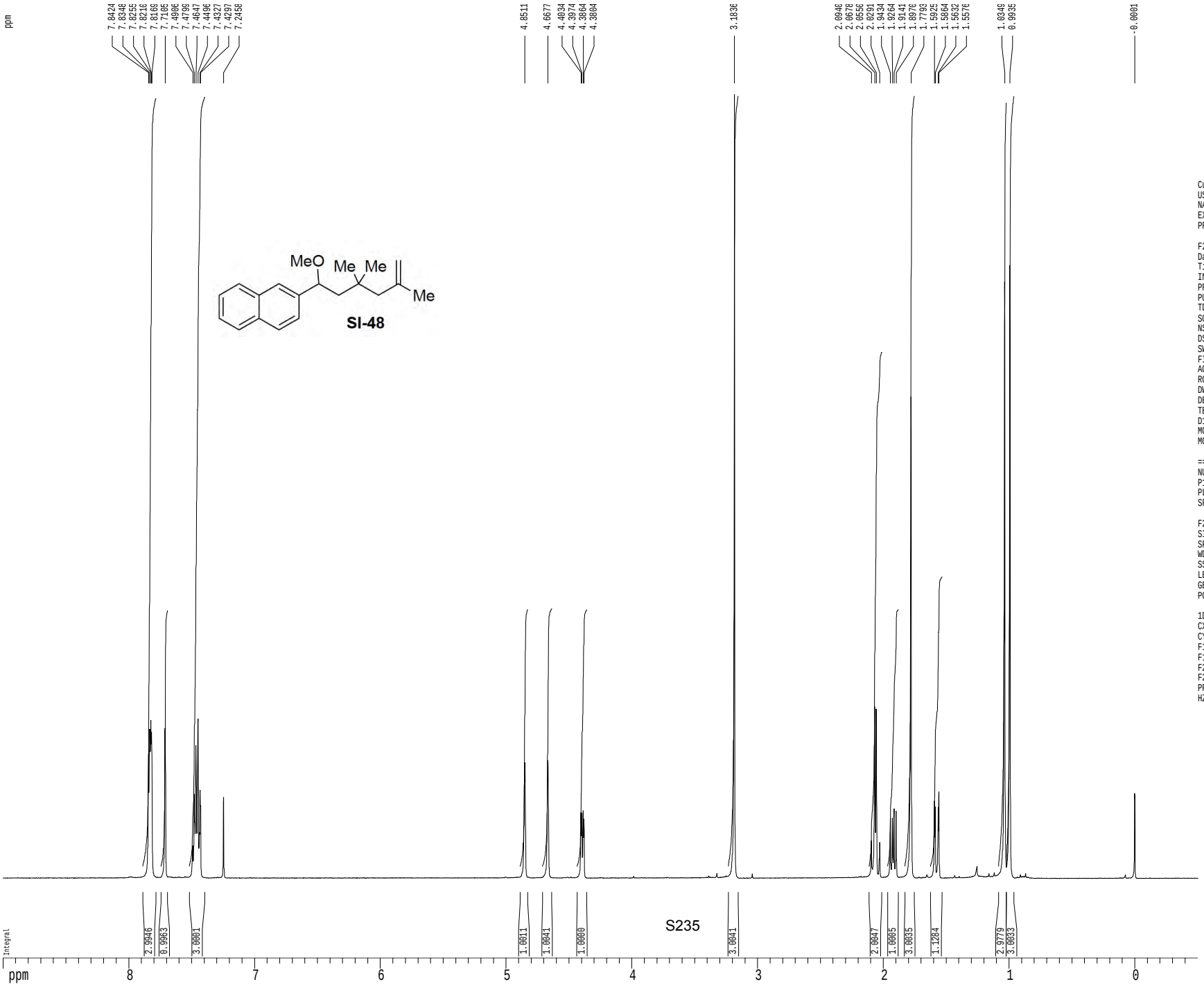
======== CHANNEL f2 =======
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2               1.60 dB
PL12             24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804103 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            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM          9.86842 ppm/cm
HZCM        1241.25415 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER            mharri
NAME     MRH-V-239-1HNMR
EXPNO                 2
PROCNO                1

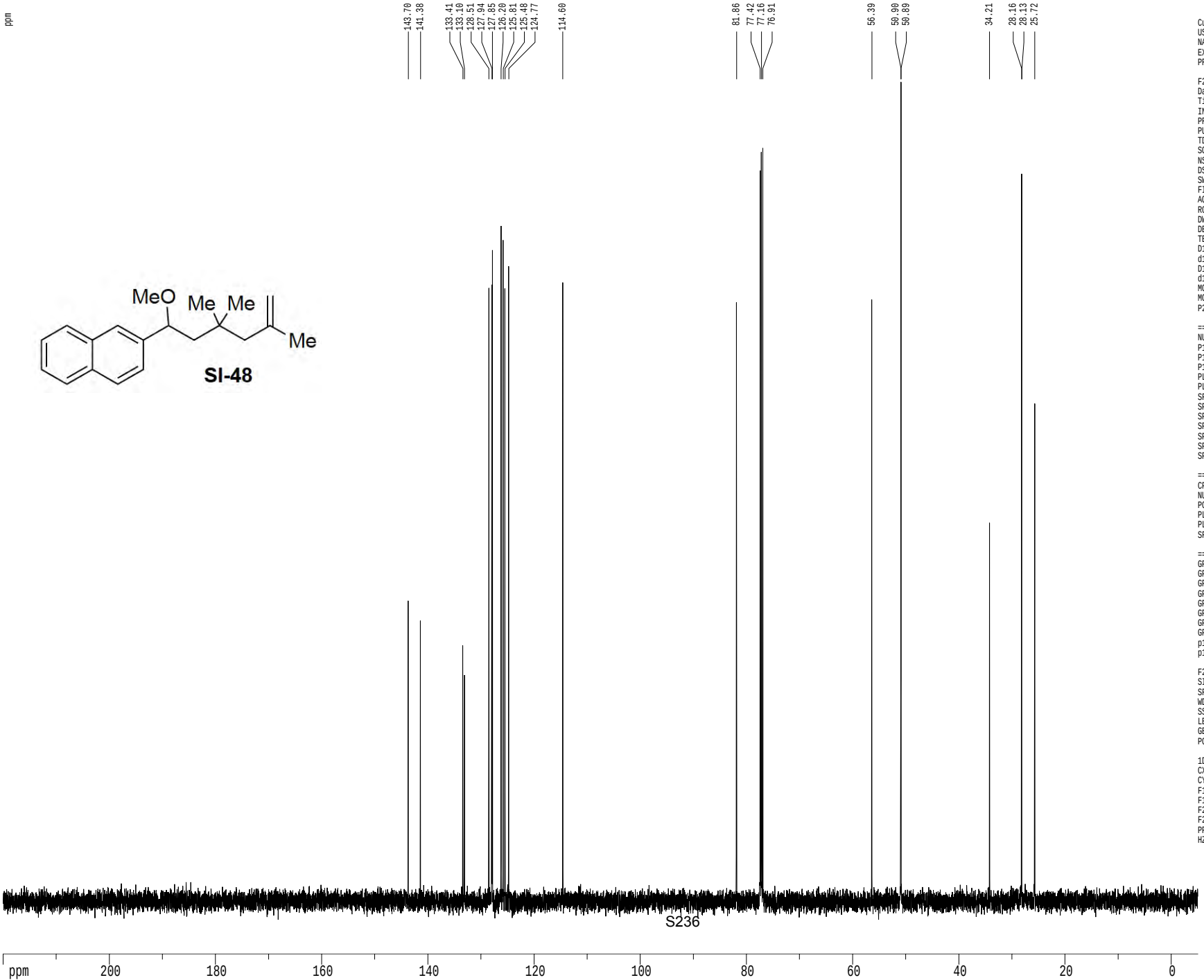
F2 - Acquisition Parameters
Date_          20130918
Time              17.21
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    1
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                  6.3
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          500.2200379 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2              -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER            mharri
NAME     MRH-V-239-13CNMR
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20130918
Time              17.24
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               5160.6
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

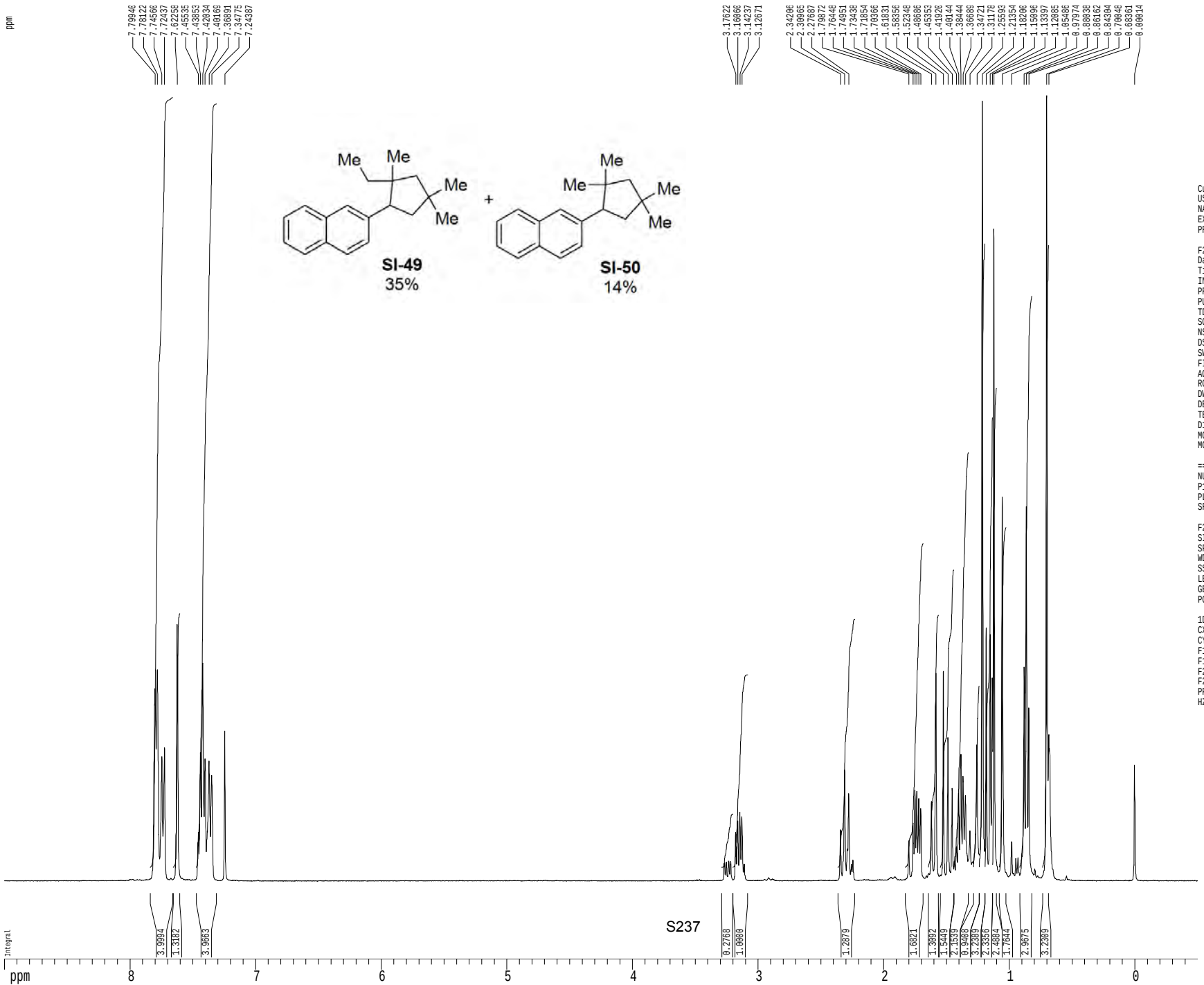
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804094 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             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME         MRH-V-241-A
EXPNO                  1
PROCNO                 1

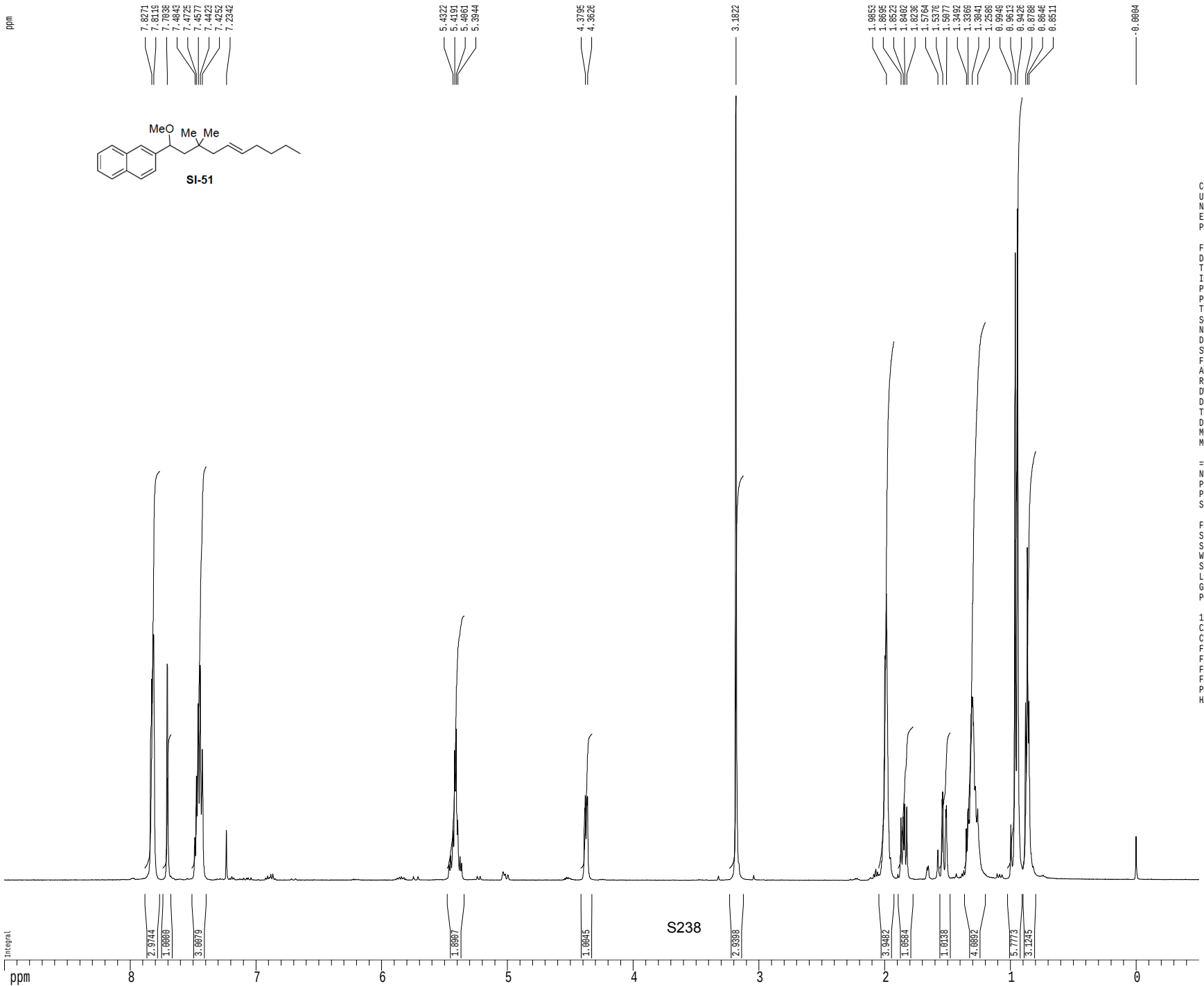
F2 - Acquisition Parameters
Date_            20130904
Time               19.27
INSTRUM           drx400
PROBHD   5 mm QNP H/F/P
PULPROG             zg30
TD                 65536
SOLVENT           CDCl3T
NS                     8
DS                     2
SWH            6410.256 Hz
FIDRES         0.097813 Hz
AQ            5.1118579 sec
RG                161.3
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               -0.60 dB
SFO1        400.1328009 MHz

F2 - Processing parameters
SI                 65536
SF          400.1300276 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                9.000 ppm
F1               3601.17 Hz
F2P               -0.500 ppm
F2               -200.06 Hz
PPMCM            0.41667 ppm/cm
HZCM           166.72086 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-148-1HNMR
EXPNO                  1
PROCNO                 1

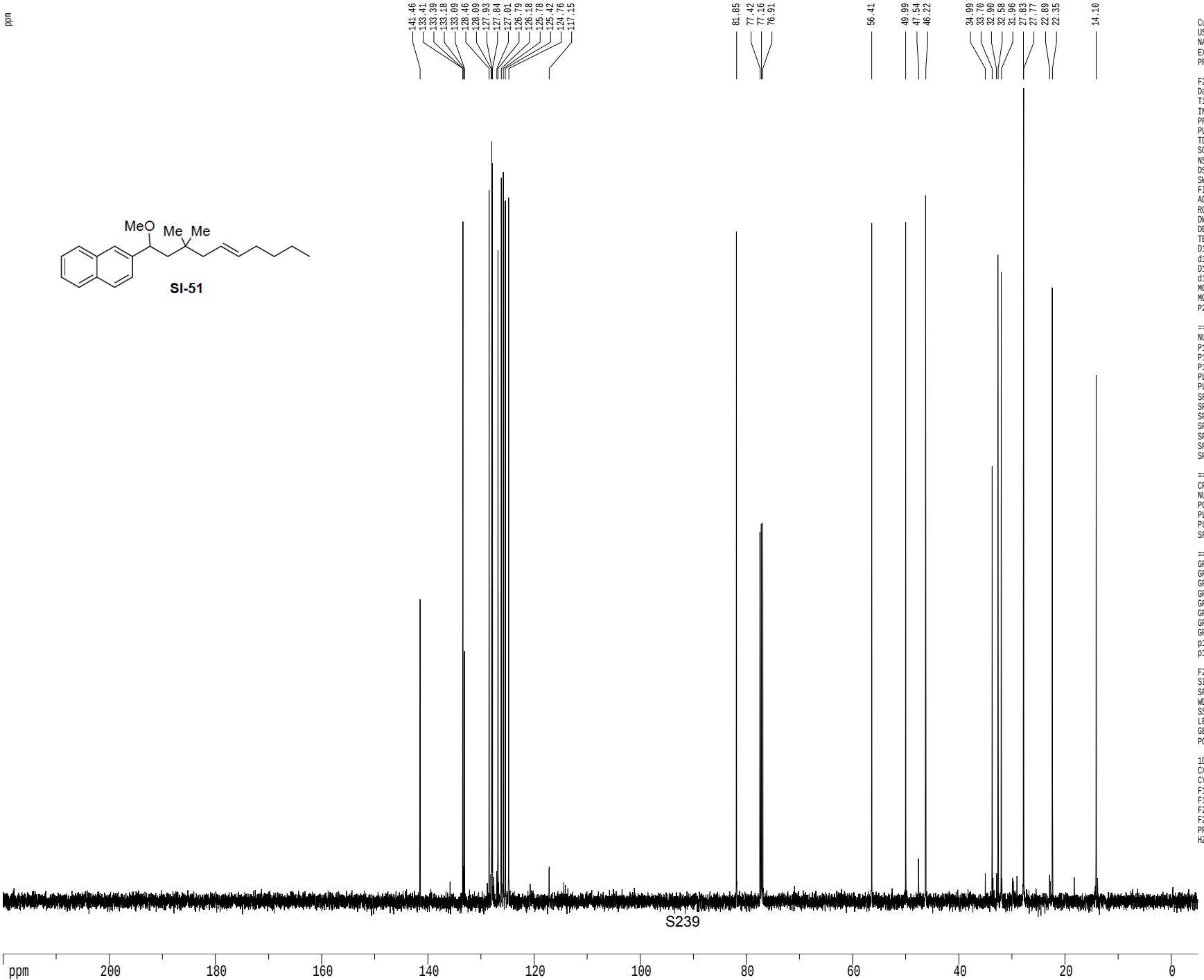
F2 - Acquisition Parameters
Date_           20140506
Time              16.12
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    2
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            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          500.2200453 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               9.000 ppm
F1             4501.98 Hz
F2P             -0.500 ppm
F2             -250.11 Hz
PPMCM          0.41667 ppm/cm
HZCM         208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-148-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140506
Time              16.15
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                    96
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

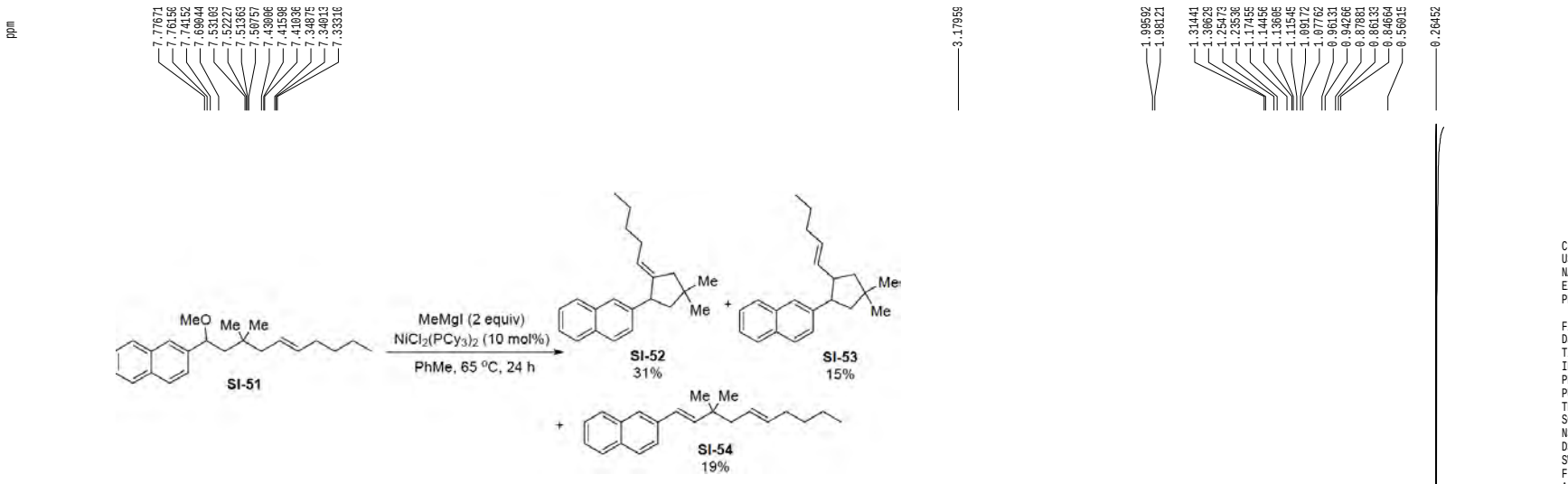
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

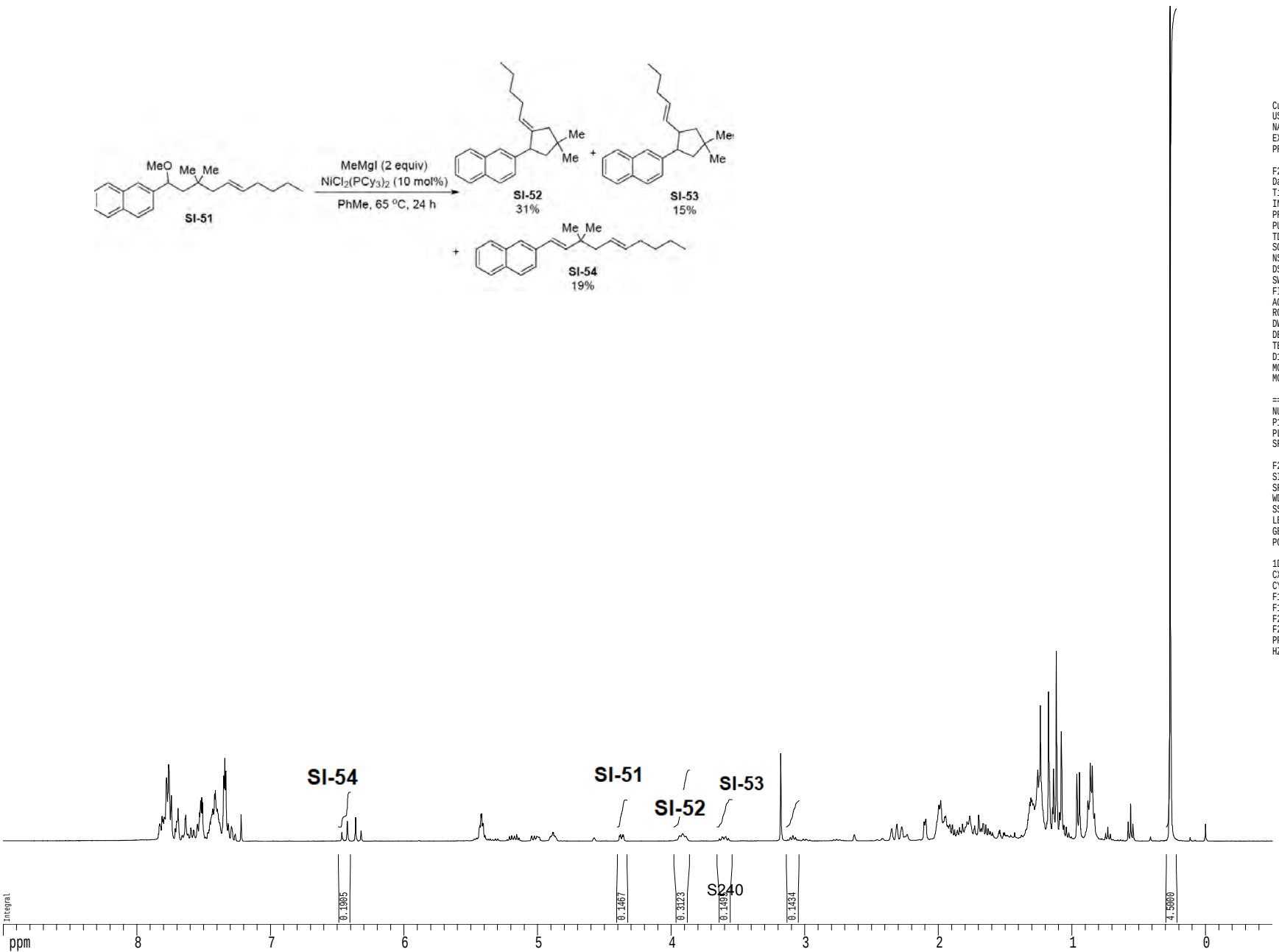
====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPX1               0.00 %
GPX2               0.00 %
GPY1               0.00 %
GPY2               0.00 %
GPZ1              30.00 %
GPZ2              50.00 %
p15             500.00 usec
p16            1000.00 usec

F2 - Processing parameters
SI                 65536
SF          125.7804113 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             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum






```
Current Data Parameters
USER            mharri
NAME      MRH-VI-174-CR
EXPNO                1
PROCNO               1

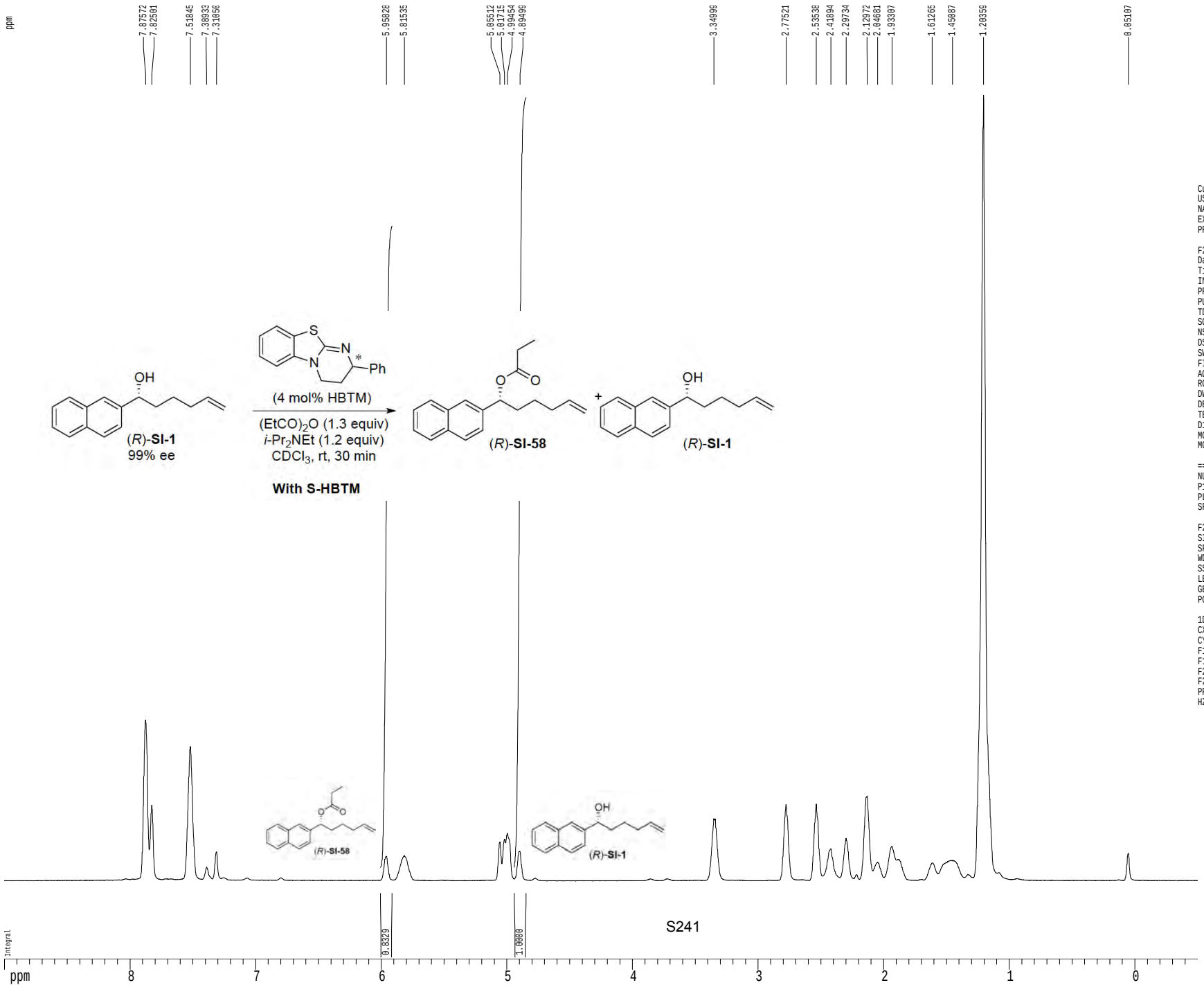
F2 - Acquisition Parameters
Date_          20140508
Time              13.44
INSTRUM          drx400
PROBHD   5 mm QNP H/F/P
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            6410.256 Hz
FIDRES         0.097813 Hz
AQ             5.1118579 sec
RG                   64
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          400.1300371 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               9.000 ppm
F1              3601.17 Hz
F2P              -0.500 ppm
F2              -200.06 Hz
PPMCM           0.41667 ppm/cm
HZCM          166.72086 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME        MRH-VI-175-S
EXPNO                  1
PROCNO                 1

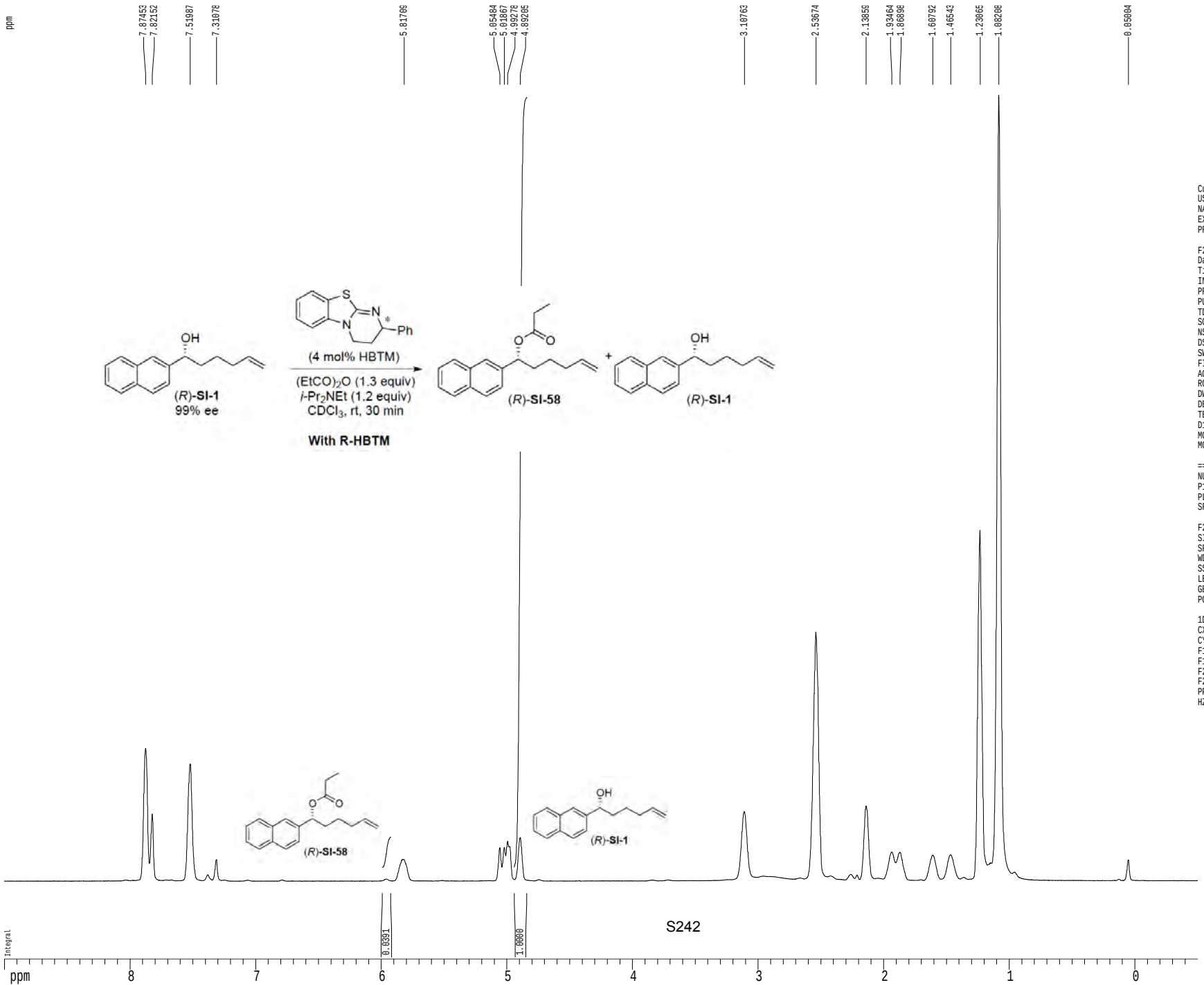
F2 - Acquisition Parameters
Date_           20140513
Time              16.19
INSTRUM           gn500
PROBHD   5 mm broadband
PULPROG            zg30
TD                81728
SOLVENT          CDCl3T
NS                     8
DS                     2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                   114
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               -4.50 dB
SFO1        499.2934950 MHz

F2 - Processing parameters
SI                 65536
SF          499.2900000 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                9.000 ppm
F1               4493.61 Hz
F2P               -0.500 ppm
F2               -249.64 Hz
PPMCM            0.41667 ppm/cm
HZCM          208.03751 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME        MRH-VI-175-R
EXPNO                  1
PROCNO                 1

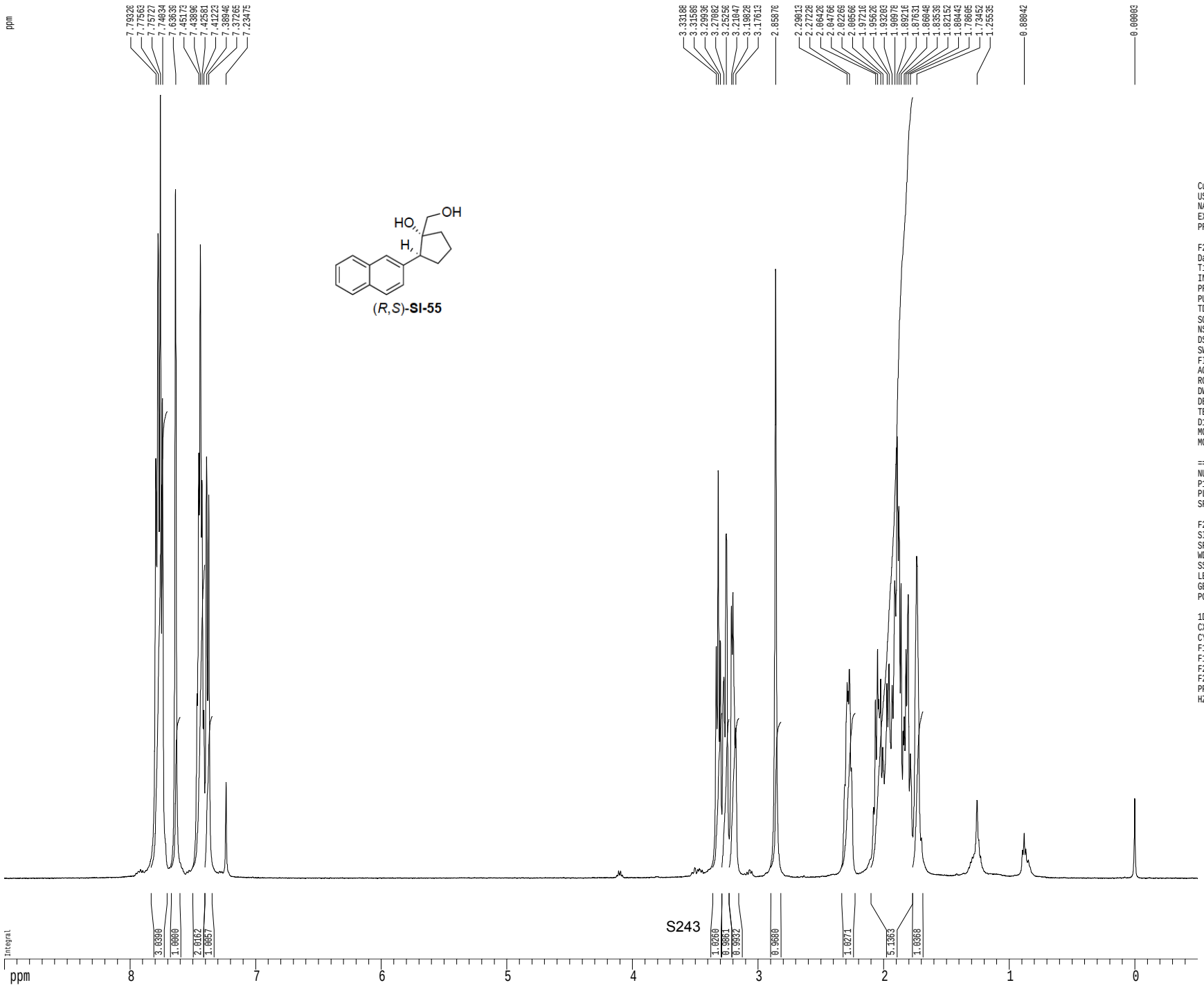
F2 - Acquisition Parameters
Date_           20140513
Time              16.22
INSTRUM           gn500
PROBHD   5 mm broadband
PULPROG            zg30
TD                81728
SOLVENT           CDCl3T
NS                     8
DS                     2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                  71.8
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               -4.50 dB
SFO1        499.2934950 MHz

F2 - Processing parameters
SI                 65536
SF          499.2900000 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                9.000 ppm
F1               4493.61 Hz
F2P               -0.500 ppm
F2               -249.64 Hz
PPMCM            0.41667 ppm/cm
HZCM          208.03751 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-163-1HNMR
EXPNO                  1
PROCNO                 1

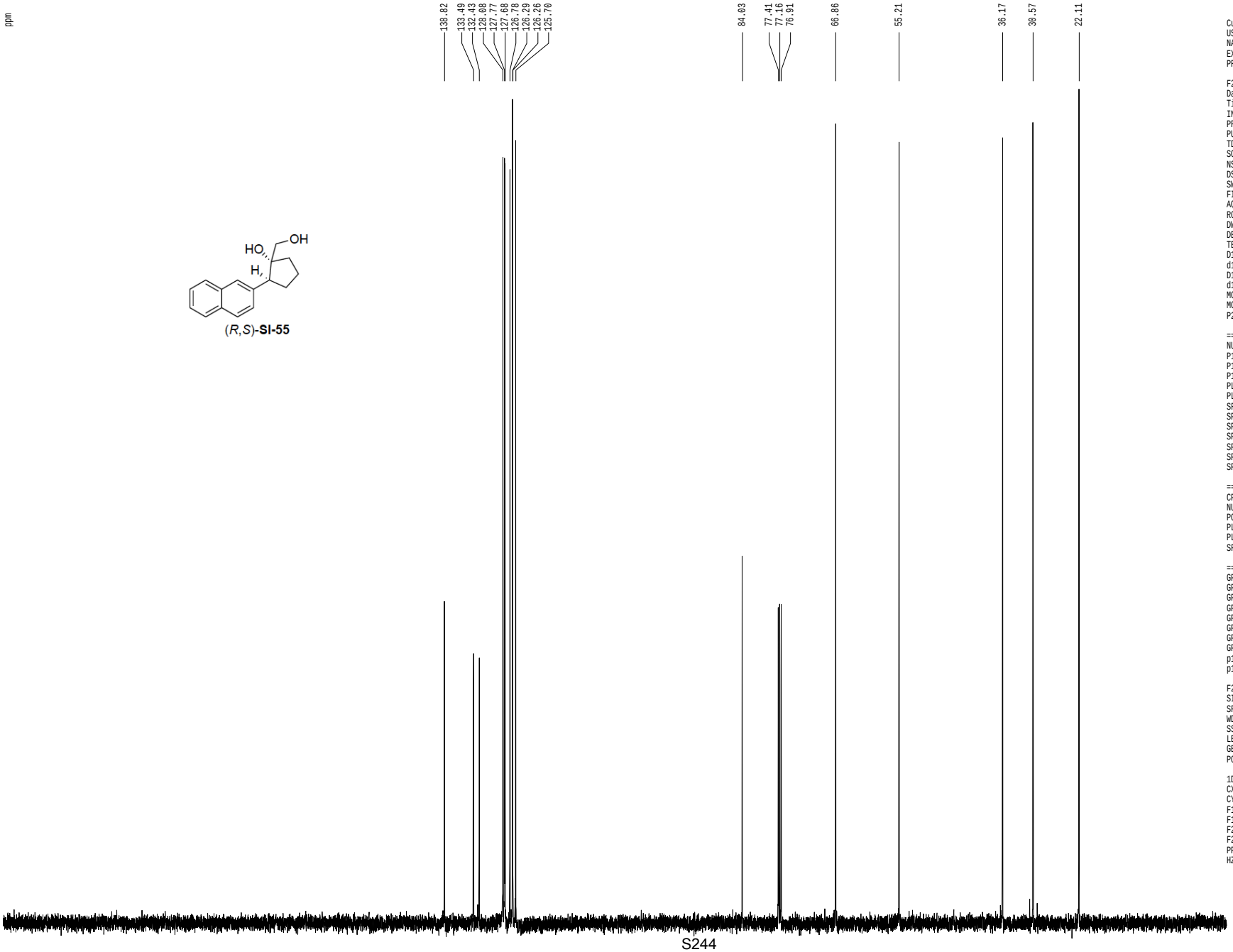
F2 - Acquisition Parameters
Date_           20140430
Time              15.36
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    1
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ            5.0998774 sec
RG                  4.5
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          500.2200453 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               9.000 ppm
F1             4501.98 Hz
F2P             -0.500 ppm
F2            -250.11 Hz
PPMCM          0.41667 ppm/cm
HZCM         208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-163-13CNMR
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140430
Time              15.38
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                   96
DS                   16
SWH           30303.031 Hz
FIDRES         0.462388 Hz
AQ            1.0813940 sec
RG              14596.5
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

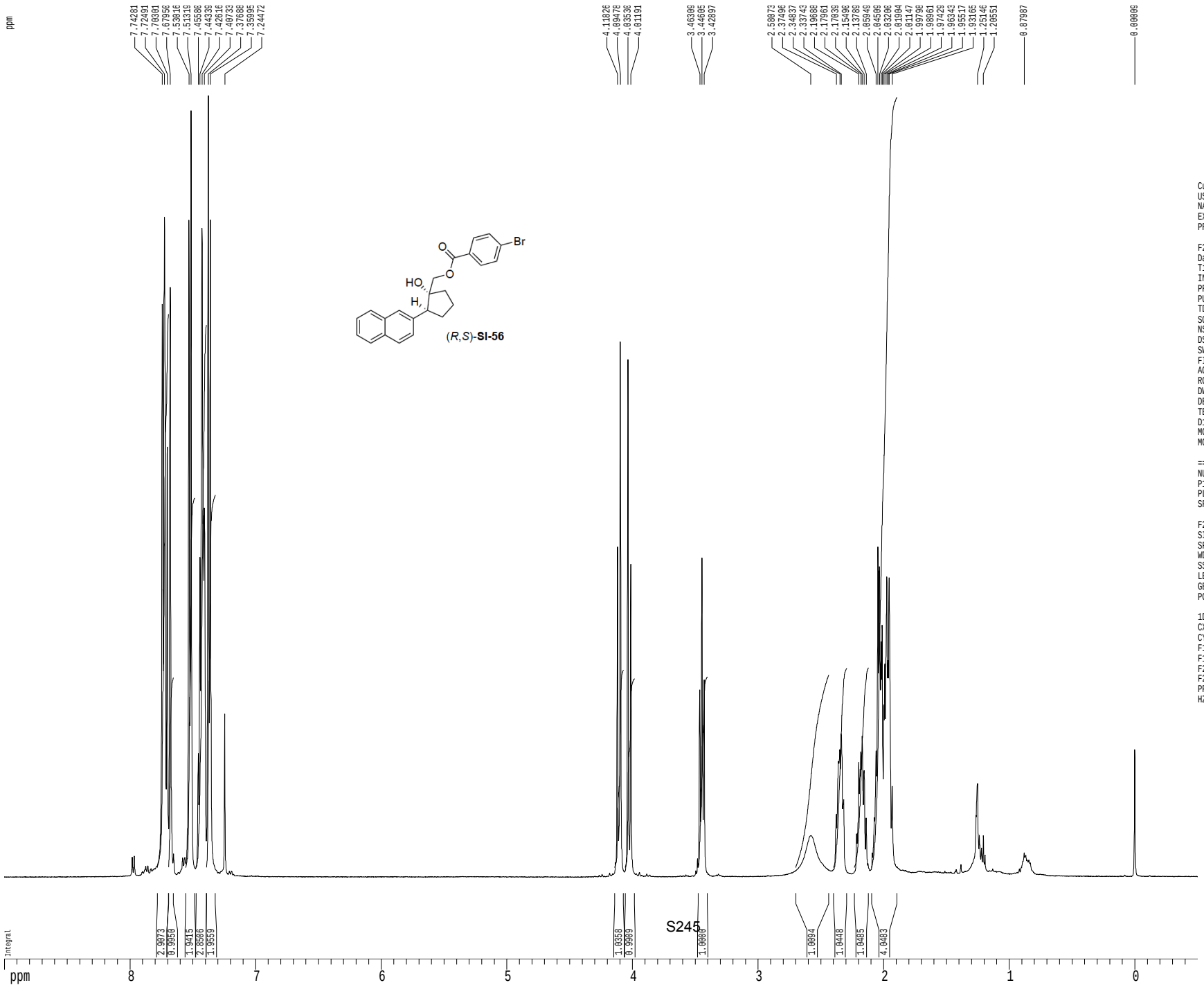
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804168 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             220.000 ppm
F1            27671.69 Hz
F2P              -5.000 ppm
F2             -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-169-1HNMR
EXPNO 1
PROCNO 1

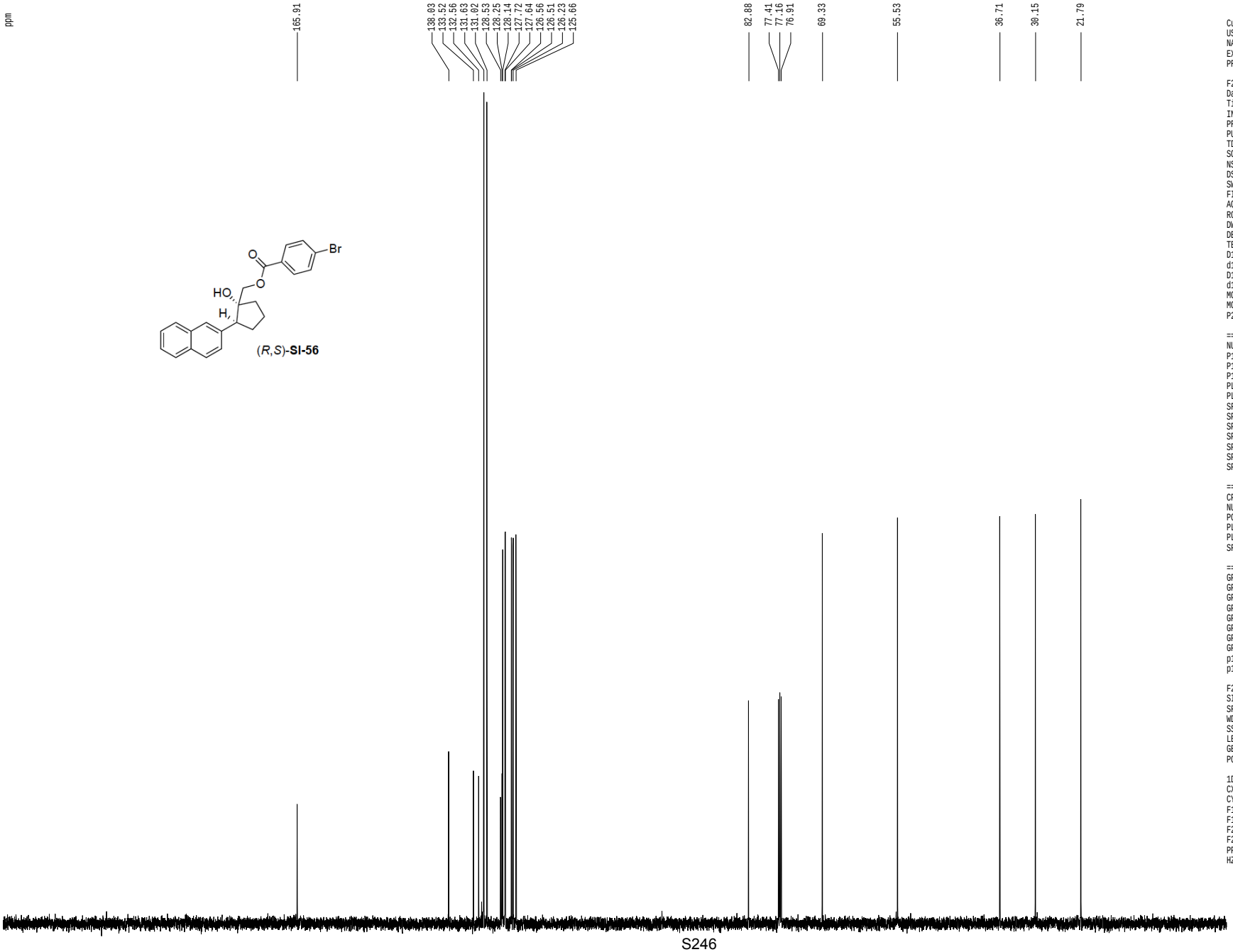
F2 - Acquisition Parameters
Date_ 20140501
Time 11.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 2
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200399 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-169-13CNMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140501
Time              11.07
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                   76
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

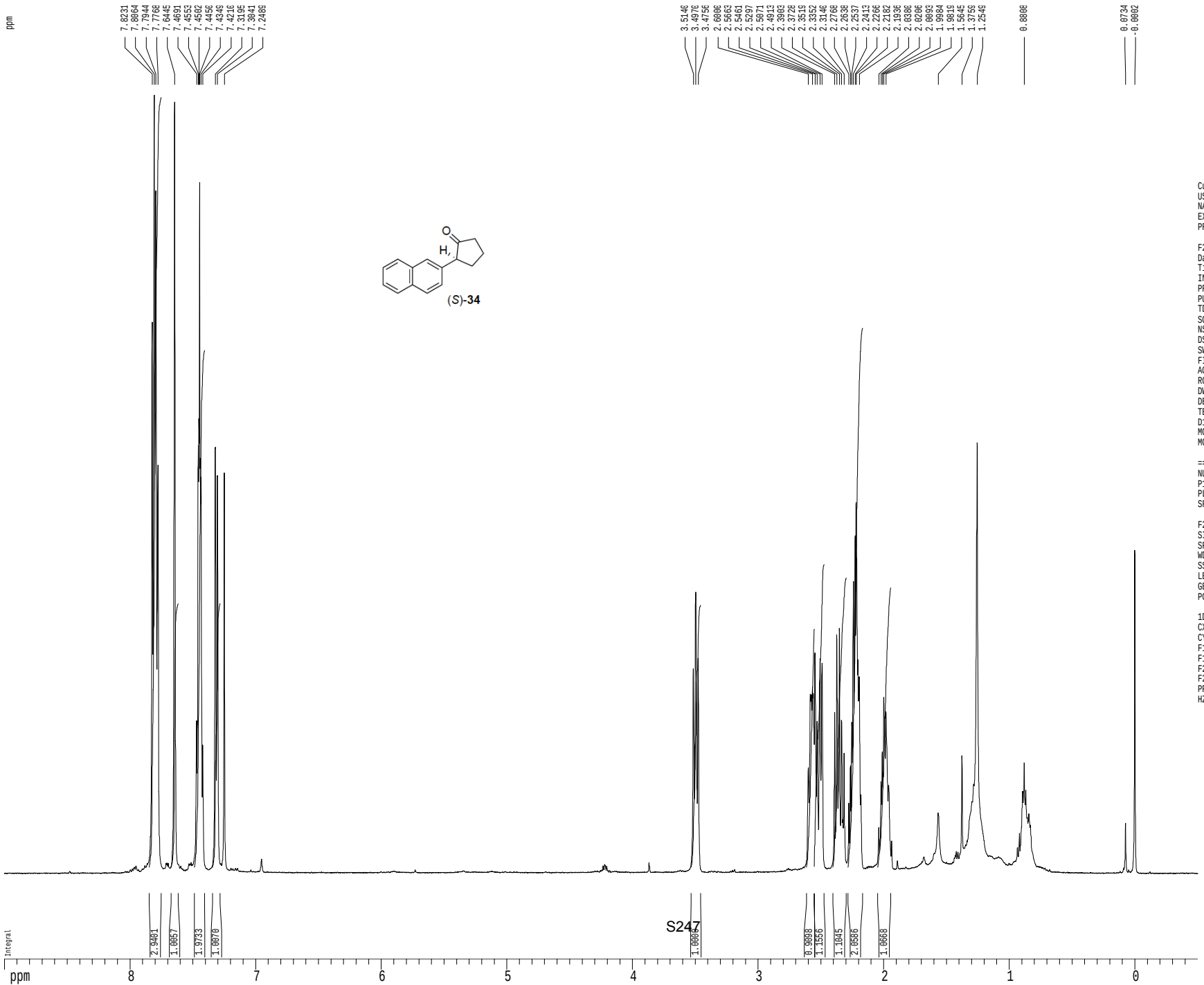
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804145 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            220.000 ppm
F1            27671.69 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



Current Data Parameters
USER mharri
NAME MRH-VI-165-1NMR
EXPNO 1
PROCNO 1

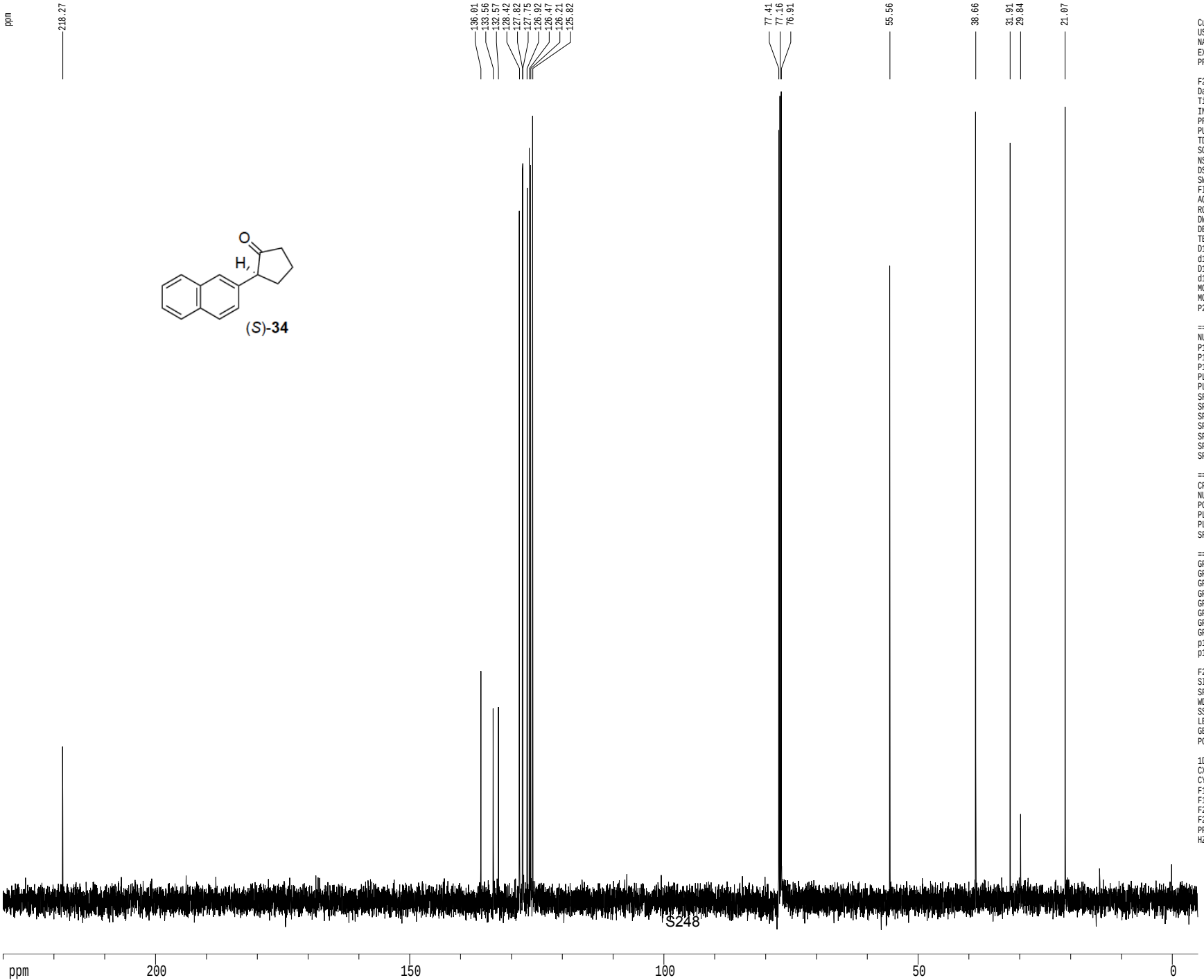
F2 - Acquisition Parameters
Date_ 20140430
Time 15.30
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200380 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 9.000 ppm
F1 4501.98 Hz
F2P -0.500 ppm
F2 -250.11 Hz
PPMCM 0.41667 ppm/cm
HZCM 208.42502 Hz/cm

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




```
Current Data Parameters
USER            mharri
NAME     MRH-VI-165-13CMR
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20140430
Time              15.34
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  107
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                31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

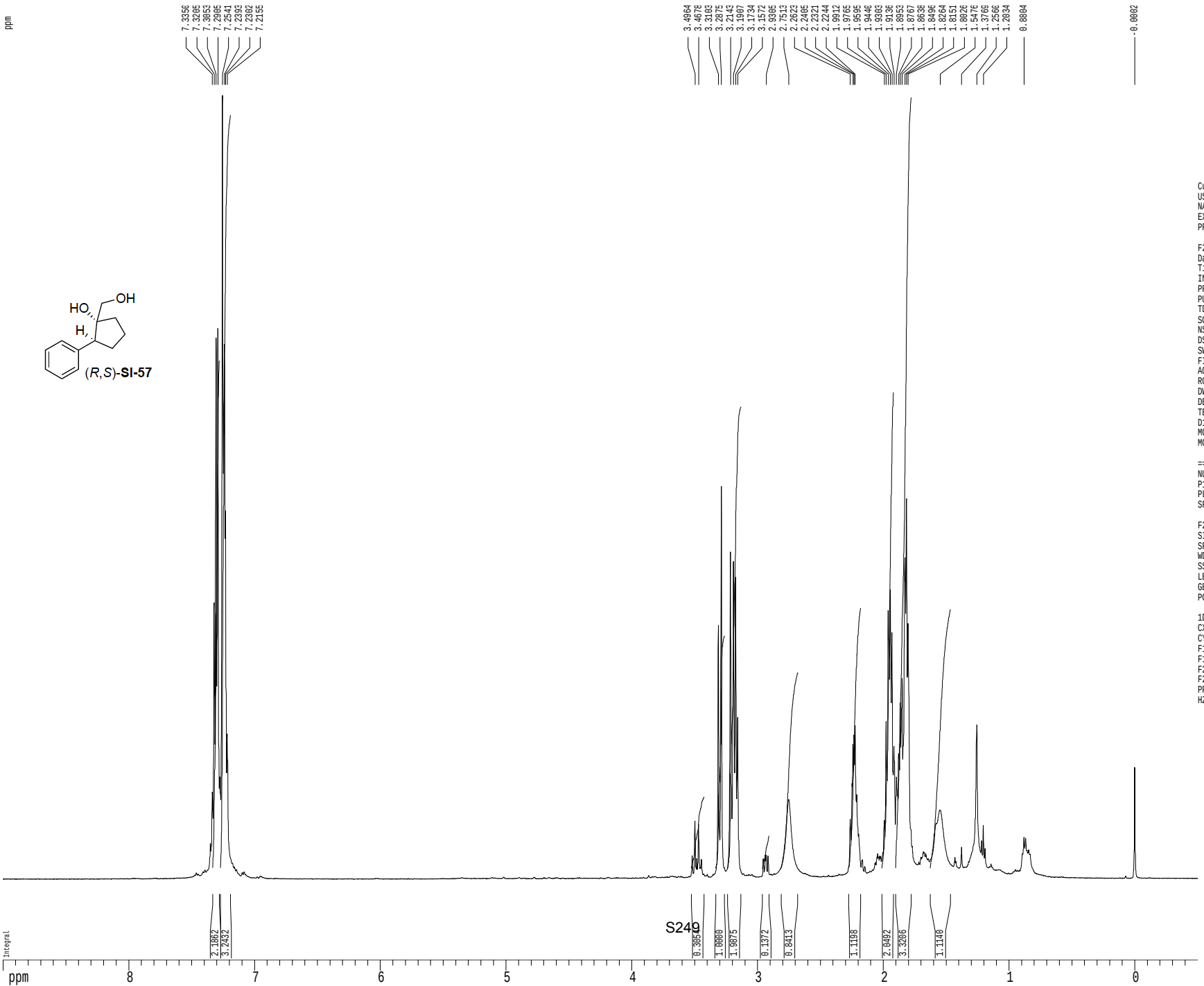
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804113 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            230.000 ppm
F1           28929.49 Hz
F2P             -5.000 ppm
F2            -628.90 Hz
PPMCM          10.30702 ppm/cm
HZCM         1296.42090 Hz/cm
```

1H spectrum




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-168-1HNMR
EXPNO                  2
PROCNO                 1

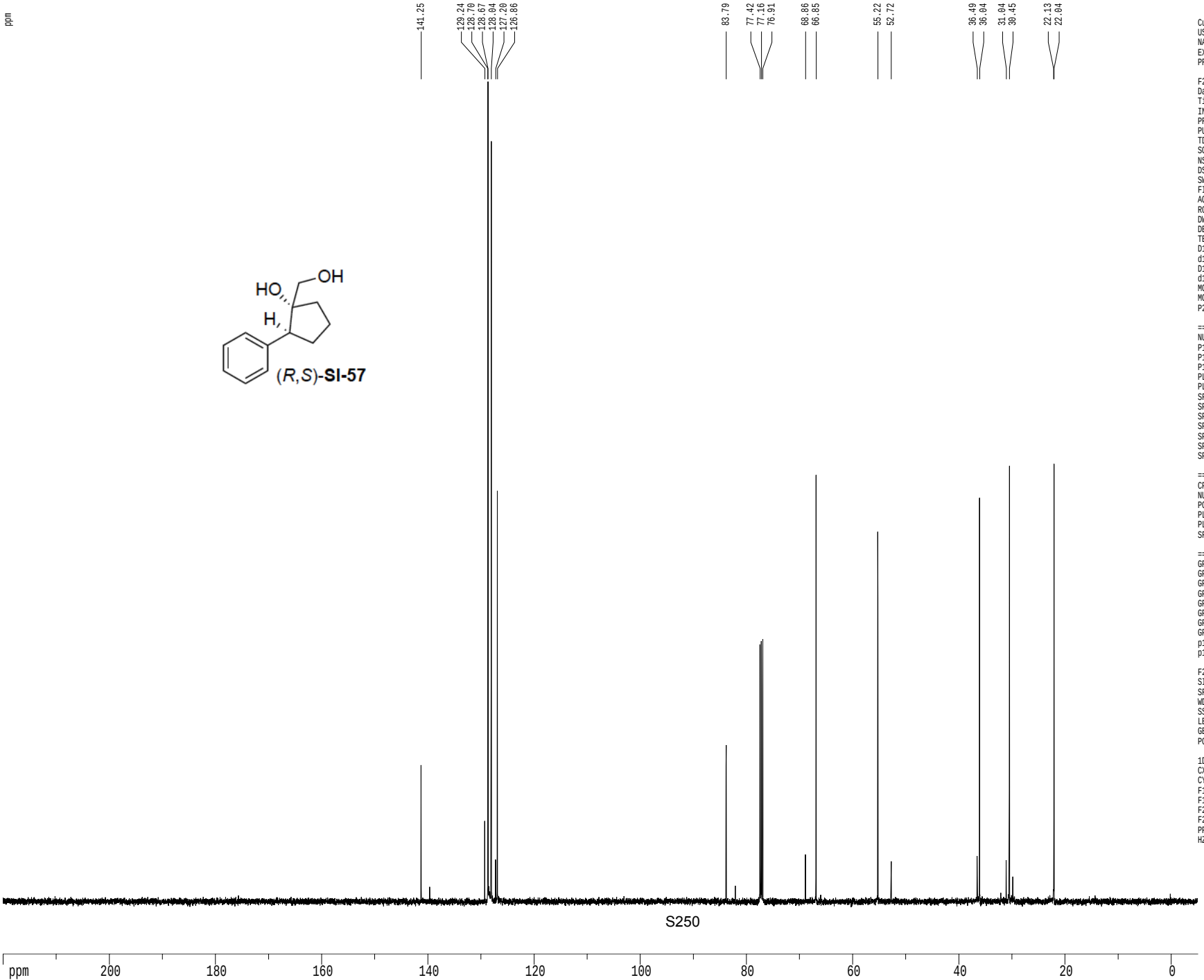
F2 - Acquisition Parameters
Date_           20140501
Time              16.01
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                     2
DS                     2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
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          500.2200347 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               9.000 ppm
F1              4501.98 Hz
F2P             -0.500 ppm
F2             -250.11 Hz
PPMCM          0.41667 ppm/cm
HZCM         208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME      MRH-VI-168-13CNMR
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140501
Time              16.05
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                  171
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG               5160.6
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                31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1                3.20 dB
SP2                3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

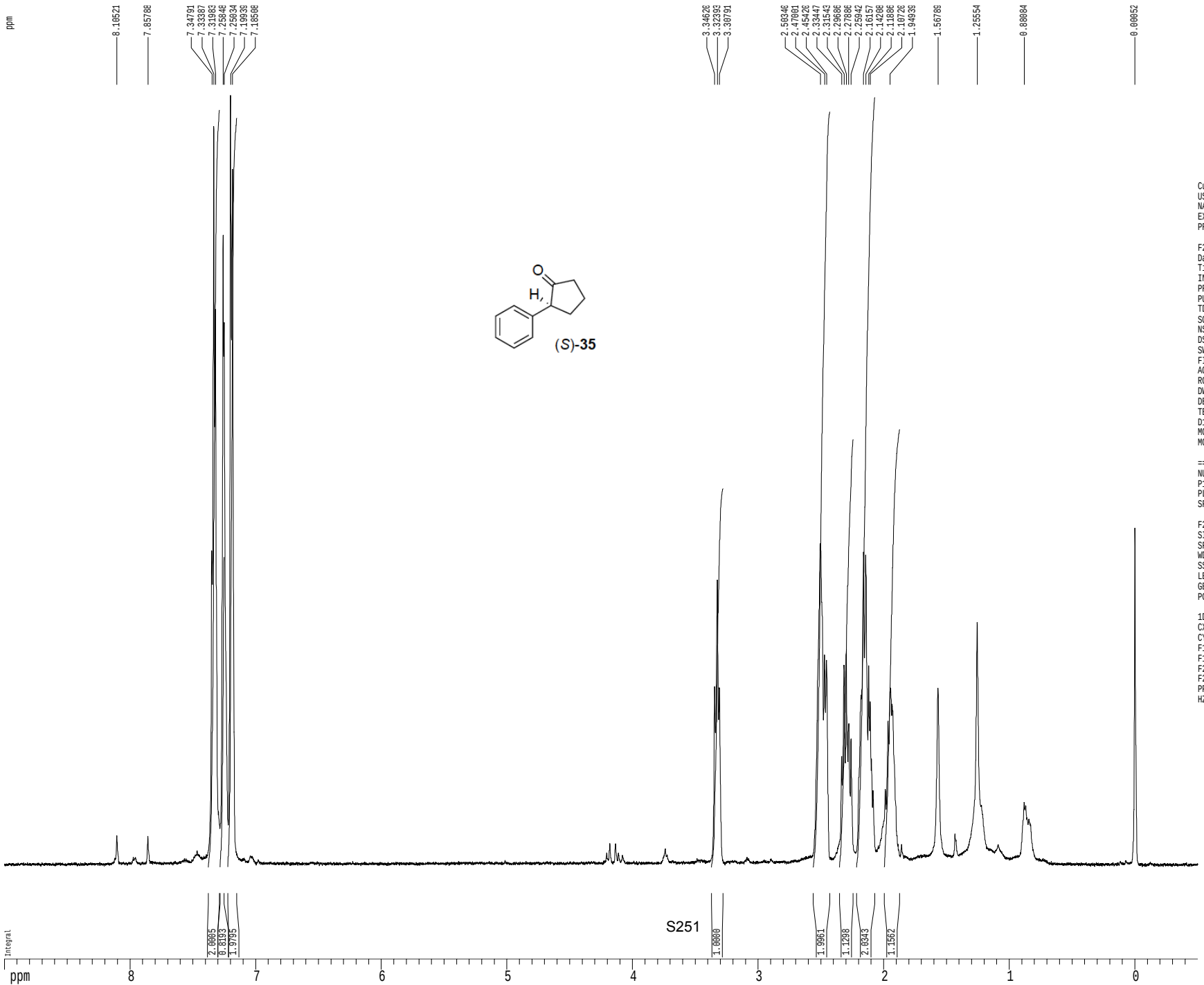
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== 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          125.7804127 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             220.000 ppm
F1             27671.69 Hz
F2P              -5.000 ppm
F2              -628.90 Hz
PPMCM           9.86842 ppm/cm
HZCM         1241.25415 Hz/cm
```

1H spectrum



S251

```
Current Data Parameters
USER              mharri
NAME     MRH-VI-170-1HNMR
EXPNO                  2
PROCNO                 1

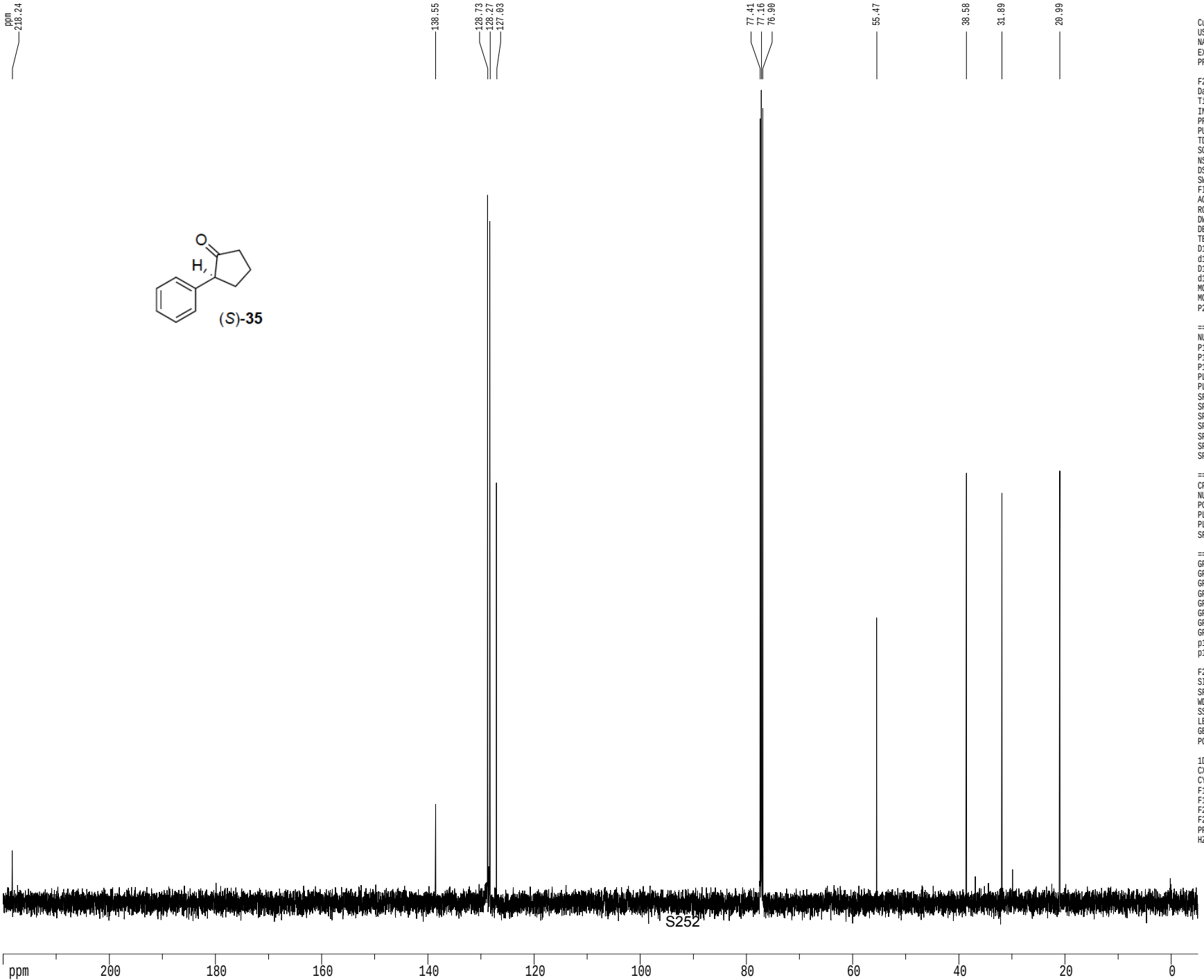
F2 - Acquisition Parameters
Date_           20140502
Time              11.44
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT           CDCl3T
NS                     1
DS                     2
SWH             8012.820 Hz
FIDRES          0.098043 Hz
AQ             5.0998774 sec
RG                   4.5
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          500.2200342 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               9.000 ppm
F1              4501.98 Hz
F2P              -0.500 ppm
F2             -250.11 Hz
PPMCM           0.41667 ppm/cm
HZCM          208.42502 Hz/cm
```

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




```
Current Data Parameters
USER              mharri
NAME     MRH-VI-170-13CNMR
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_          20140502
Time              11.46
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CDCl3T
NS                  206
DS                   16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0813940 sec
RG                5160.6
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                 31.00 usec

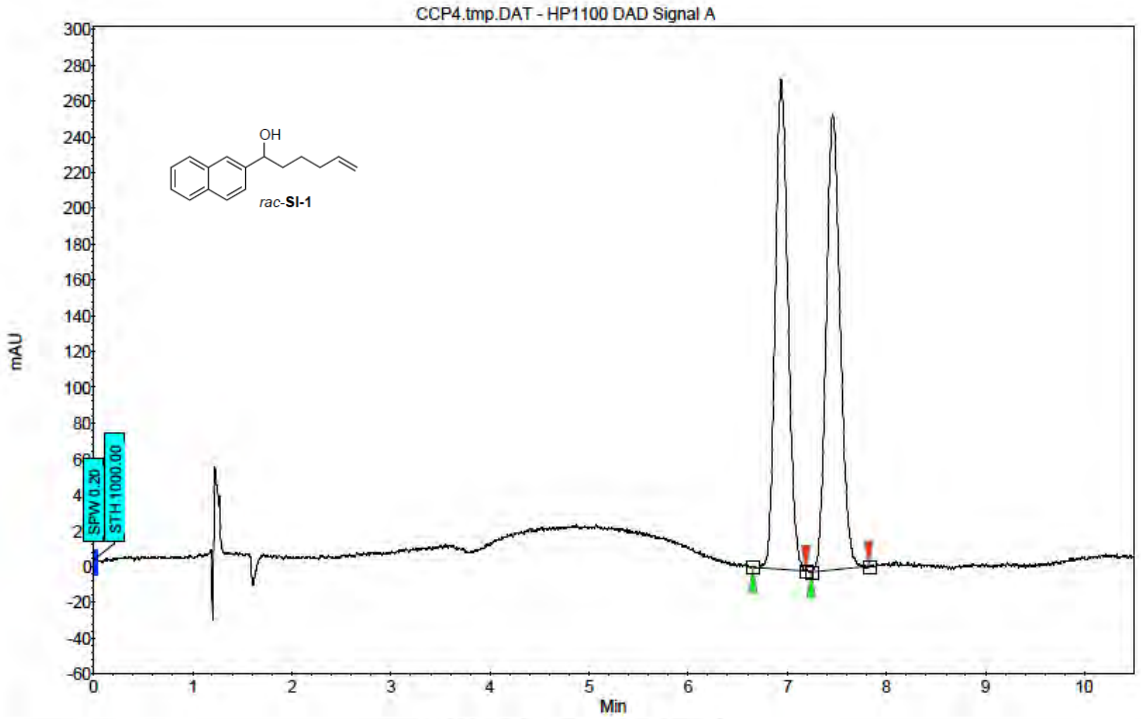
======== CHANNEL f1 ========
NUC1                 13C
P1                 15.50 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 3.20 dB
SP2                 3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1              0.00 Hz
SPOFF2              0.00 Hz

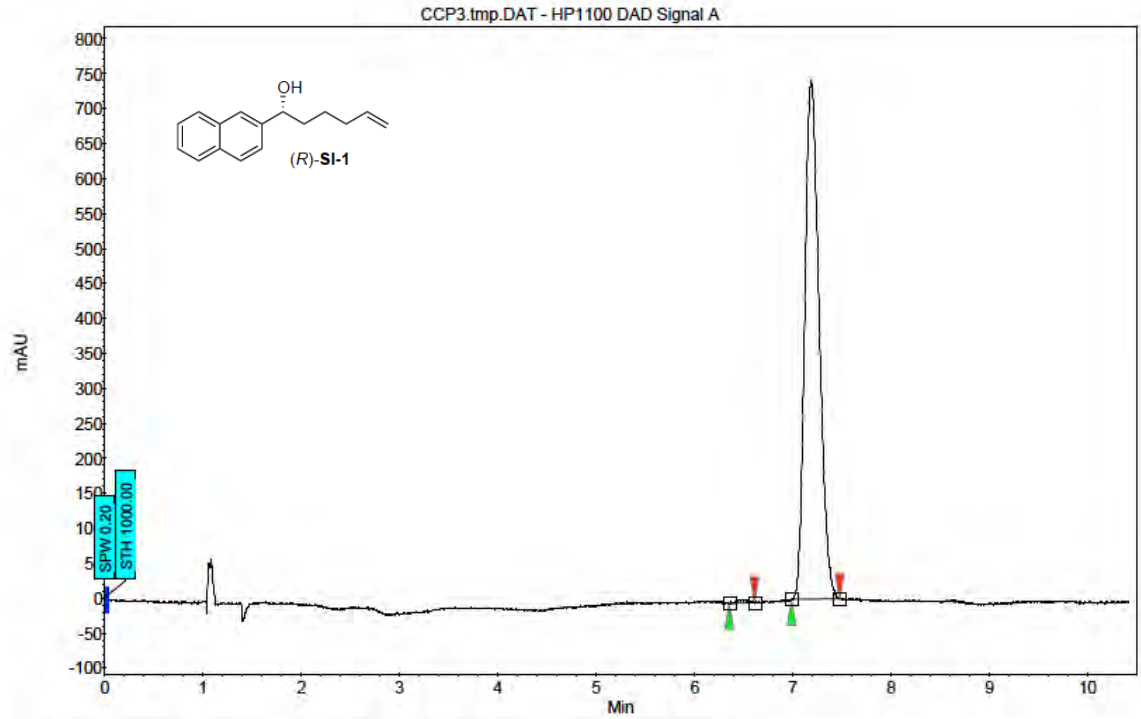
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                 1.60 dB
PL12               24.60 dB
SFO2         500.2225011 MHz

====== 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           125.7804099 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              220.000 ppm
F1             27671.69 Hz
F2P               -5.000 ppm
F2              -628.90 Hz
PPMCM            9.86842 ppm/cm
HZCM          1241.25415 Hz/cm
```





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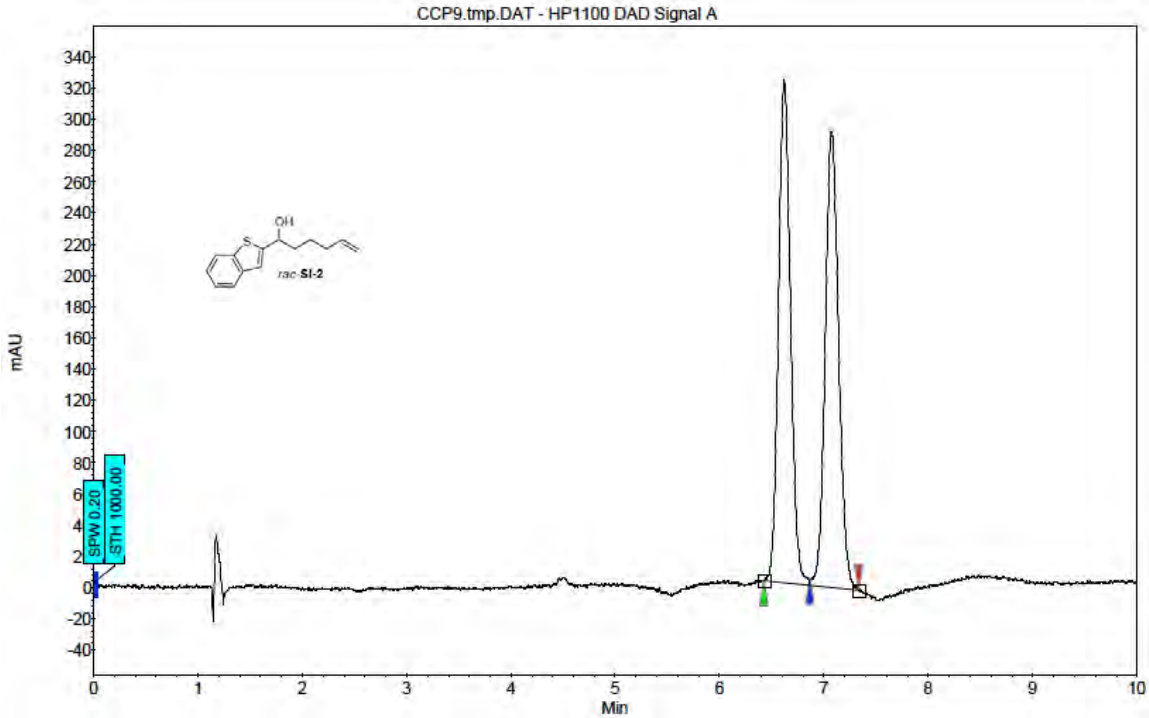
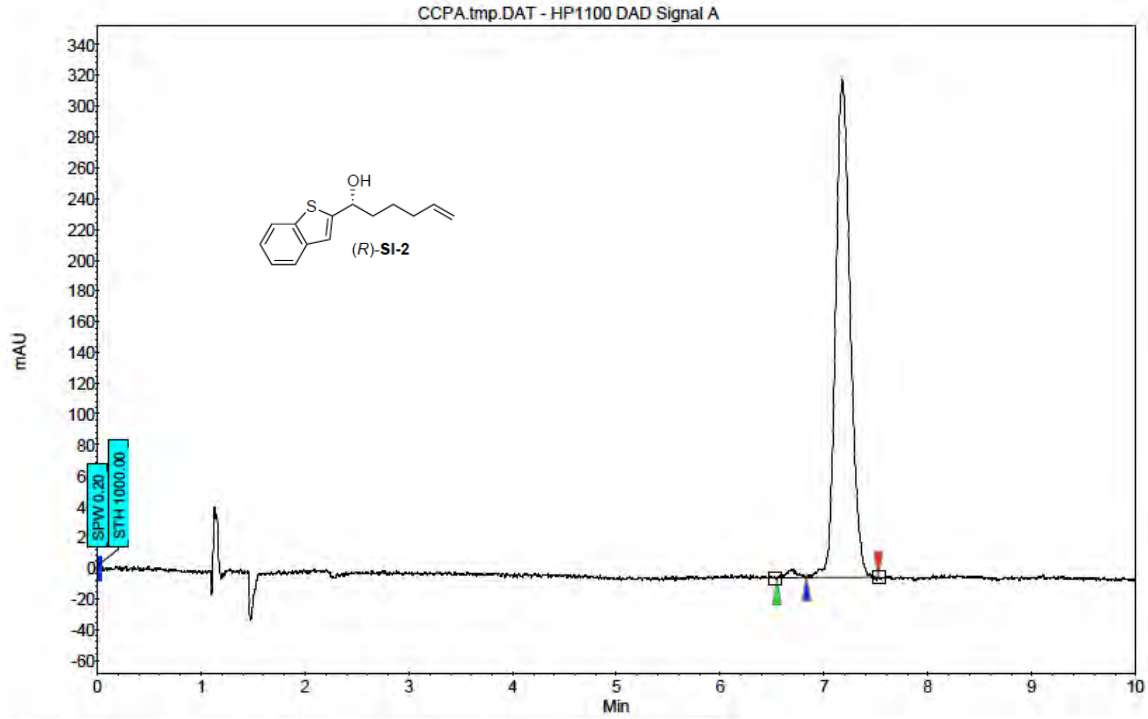
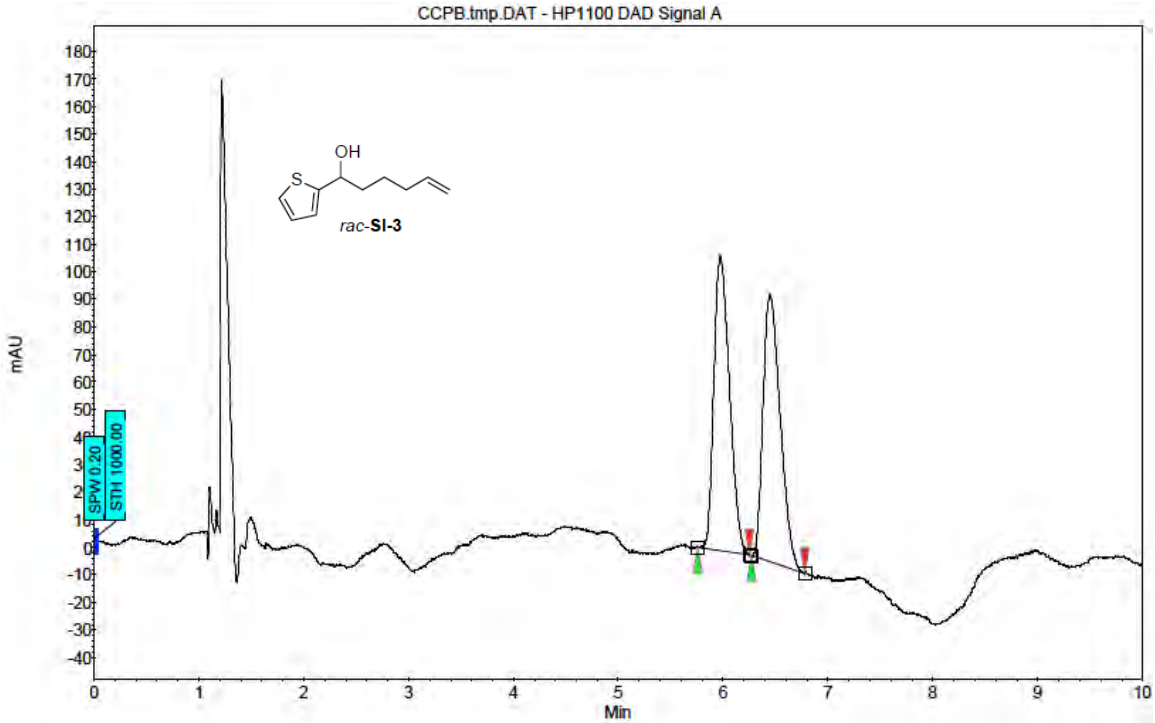
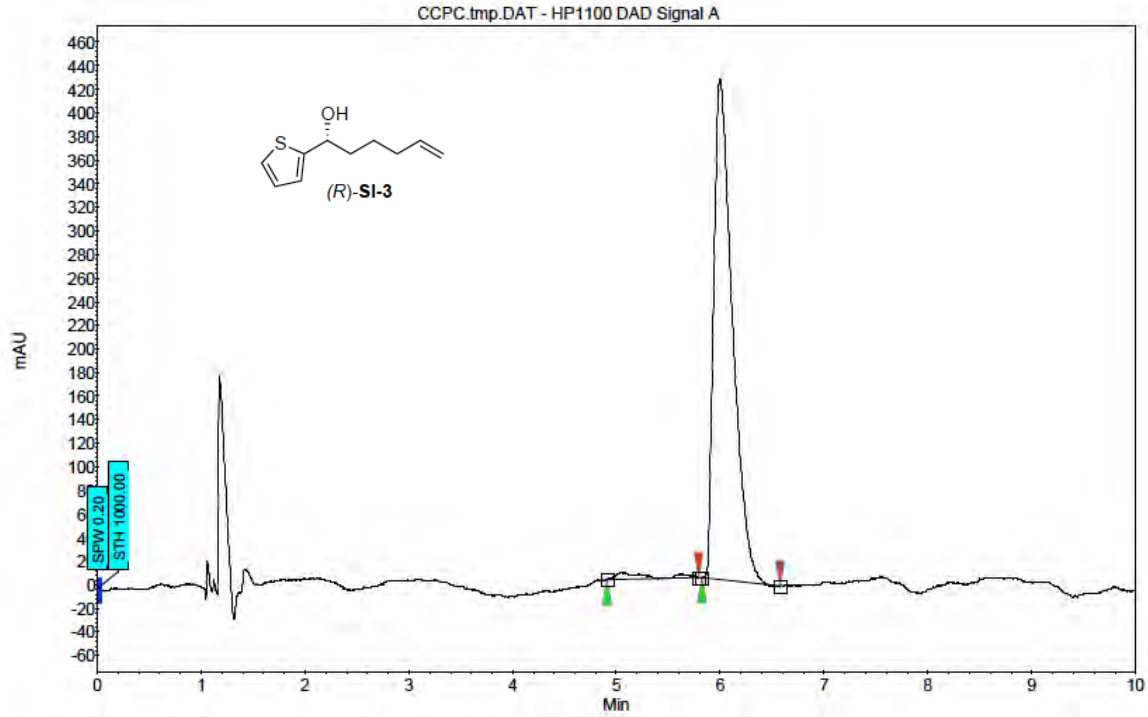
2	UNKNOWN	6.36	6.50	6.61	0.00	0.44	5.2	0.6	0.442
1	UNKNOWN	6.99	7.19	7.48	0.00	99.56	740.8	125.2	99.558
Total						100.00	746.0	125.8	100.000



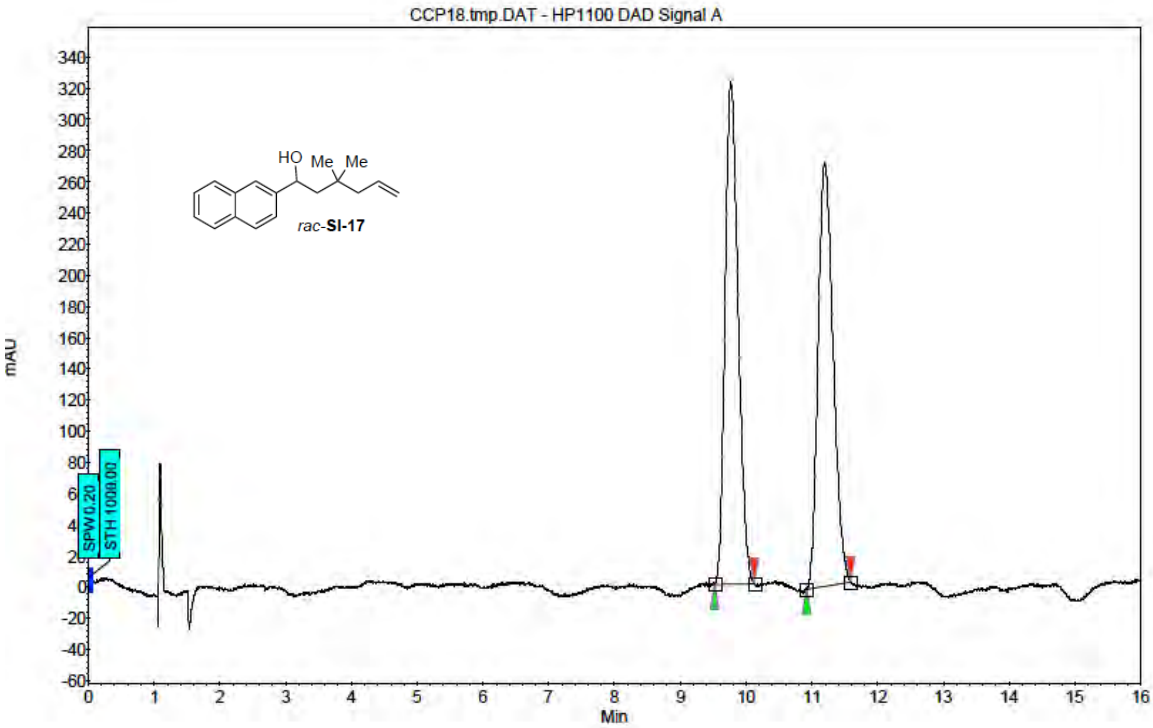


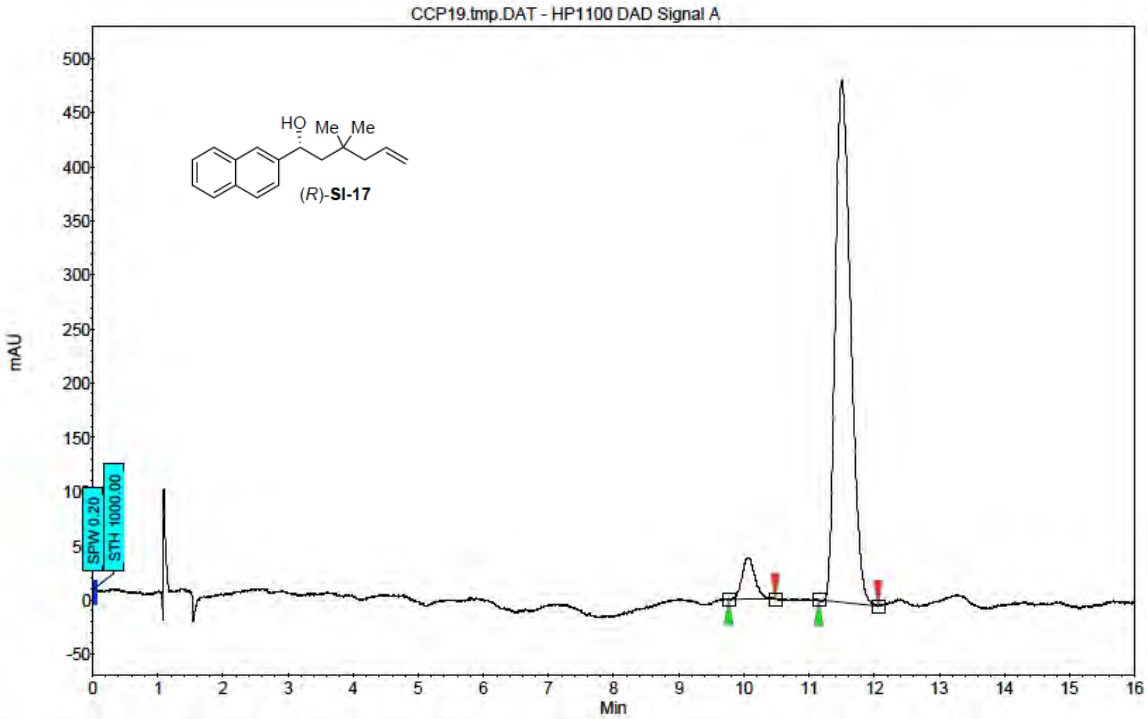
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
2	UNKNOWN	6.55	6.71	6.83	0.00	1.13	5.4	0.6	1.130
1	UNKNOWN	6.83	7.18	7.52	0.00	98.87	323.0	52.3	98.870
Total						100.00	328.4	52.9	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
2	UNKNOWN	4.91	5.05	5.80	0.00	2.20	6.1	2.0	2.200
1	UNKNOWN	5.82	6.00	6.58	0.00	97.80	423.3	88.0	97.800
Total						100.00	429.3	90.0	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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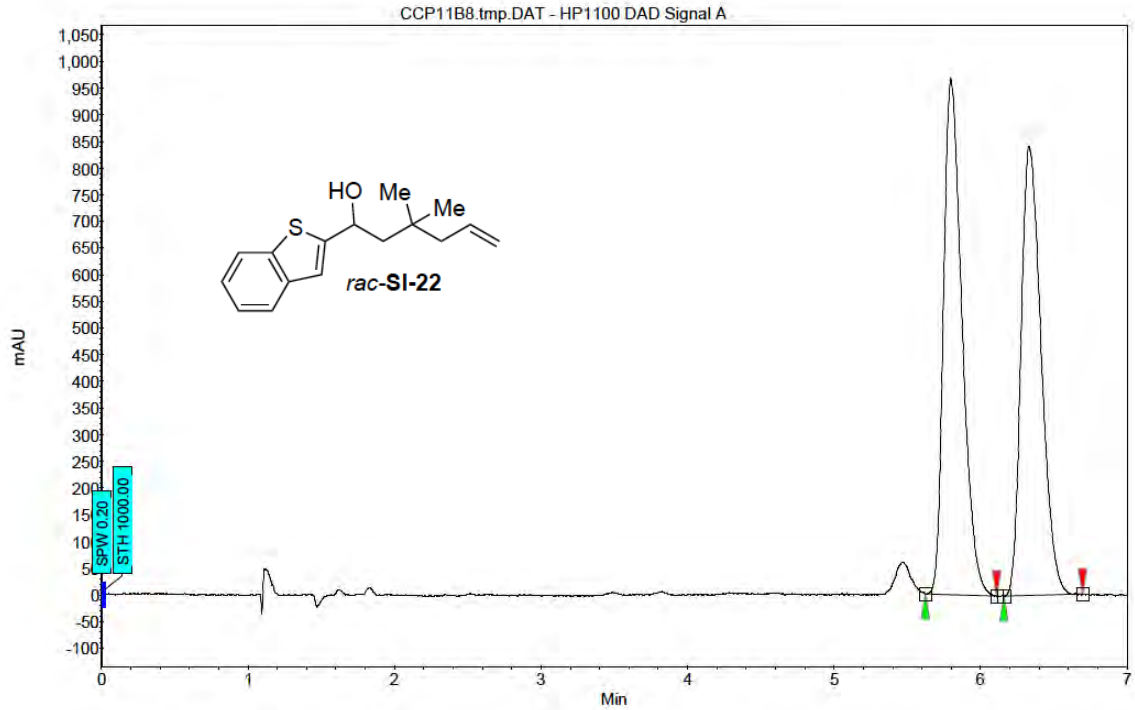
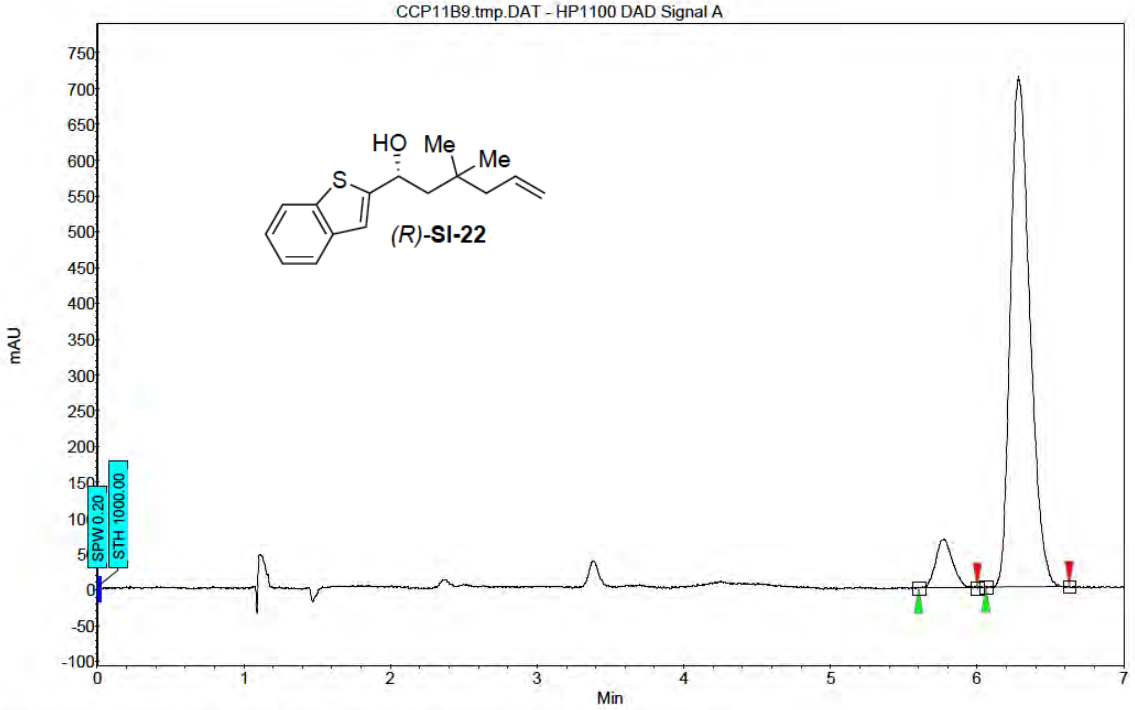
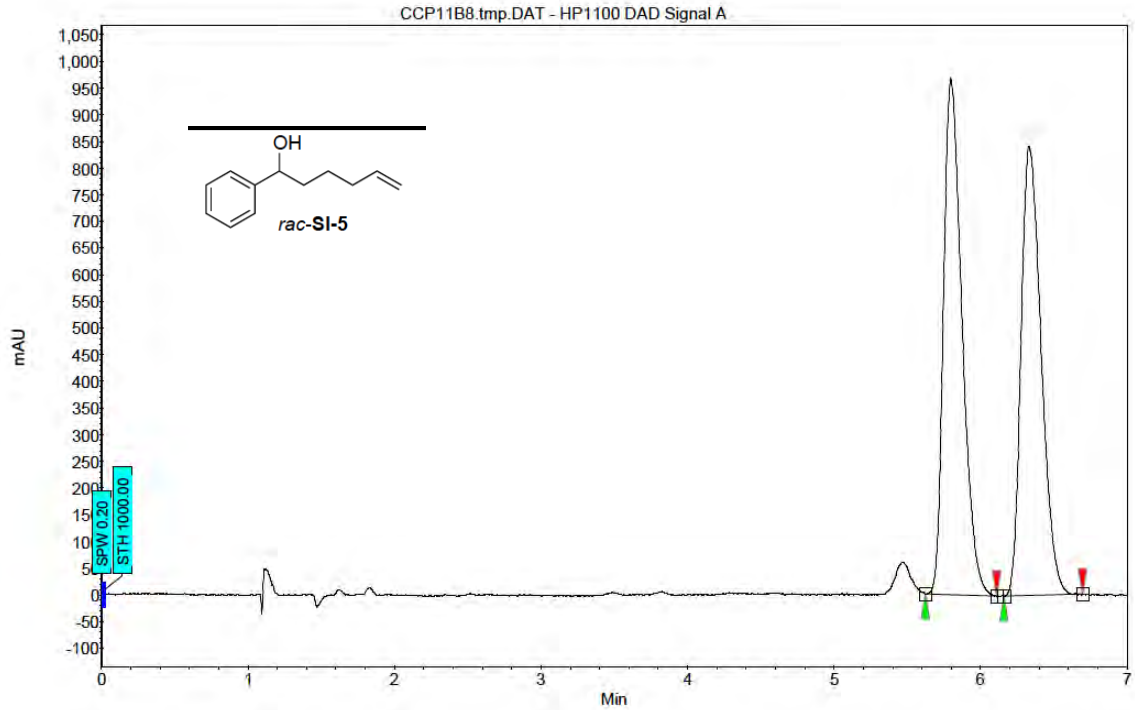
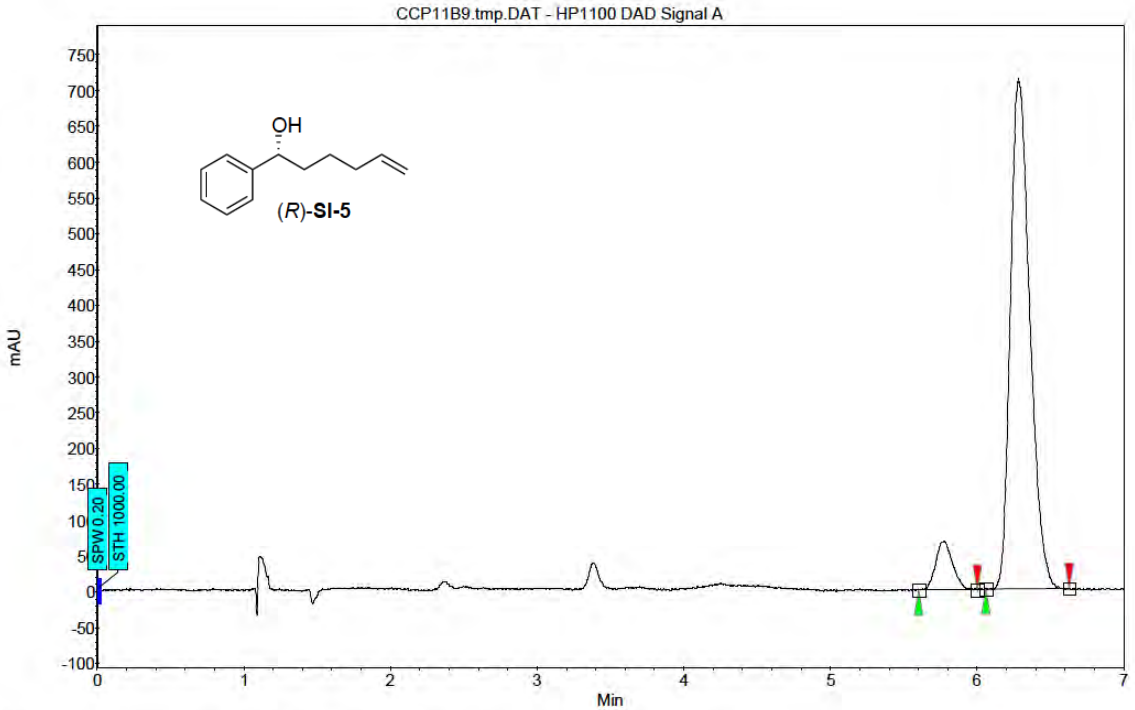
1	UNKNOWN	9.76	10.05	10.48	0.00	6.27	38.5	8.8	6.269
2	UNKNOWN	11.15	11.51	12.06	0.00	93.73	481.5	130.9	93.731
Total						100.00	520.0	139.7	100.000



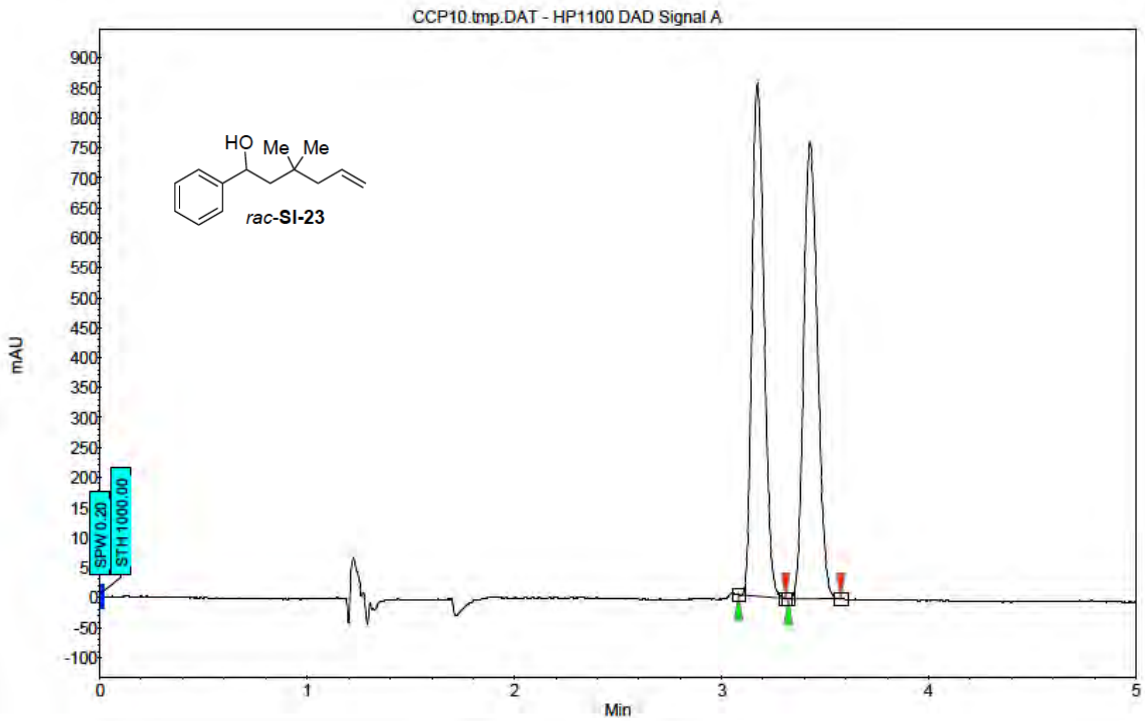


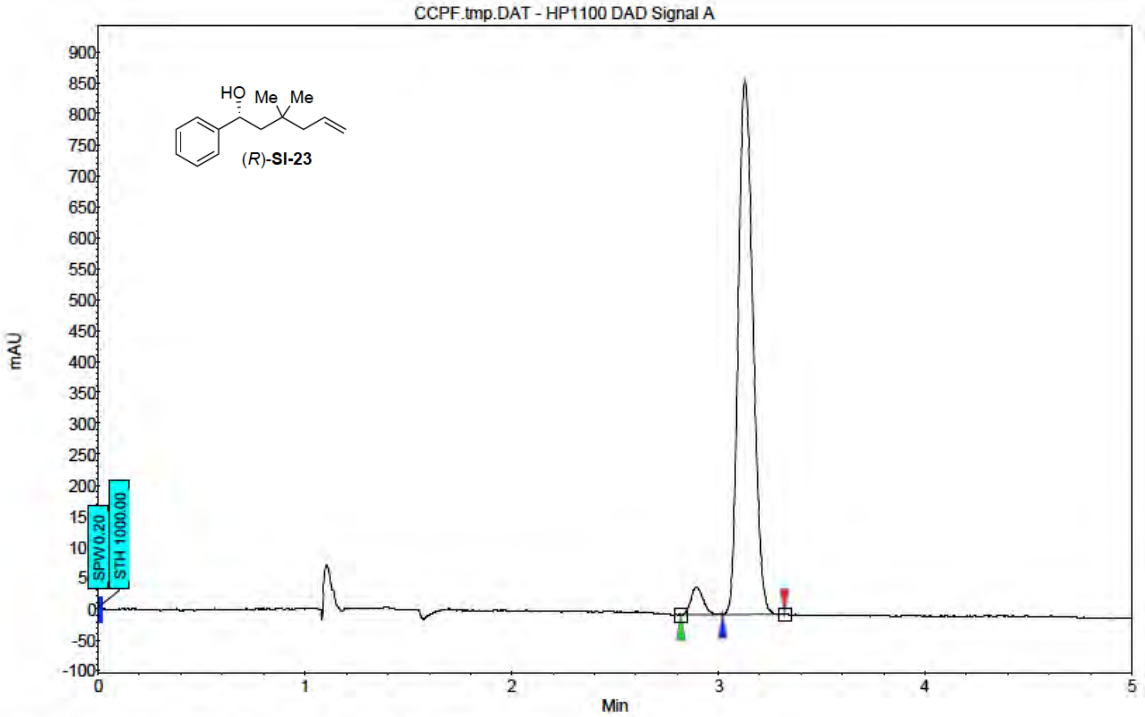
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.60	5.78	6.00	0.00	7.72	67.9	9.1	7.716
2	UNKNOWN	6.06	6.28	6.63	0.00	92.28	712.1	109.2	92.284
Total						100.00	780.0	118.3	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.60	5.78	6.00	0.00	7.72	67.9	9.1	7.716
2	UNKNOWN	6.06	6.28	6.63	0.00	92.28	712.1	109.2	92.284
Total						100.00	780.0	118.3	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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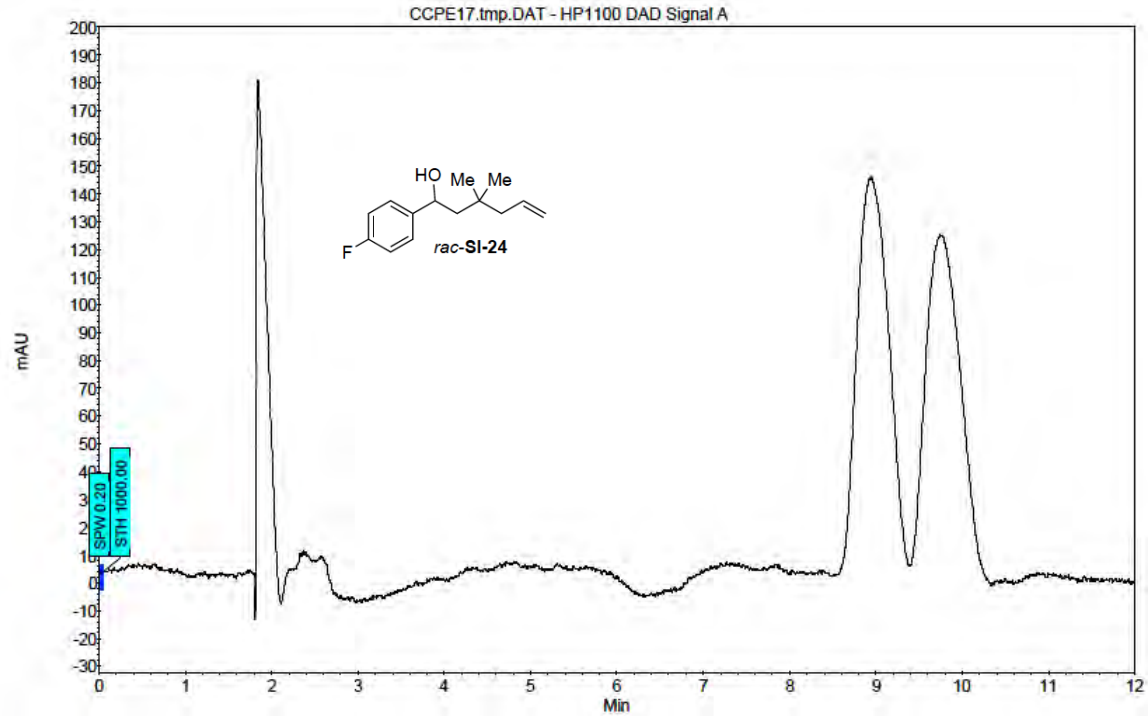
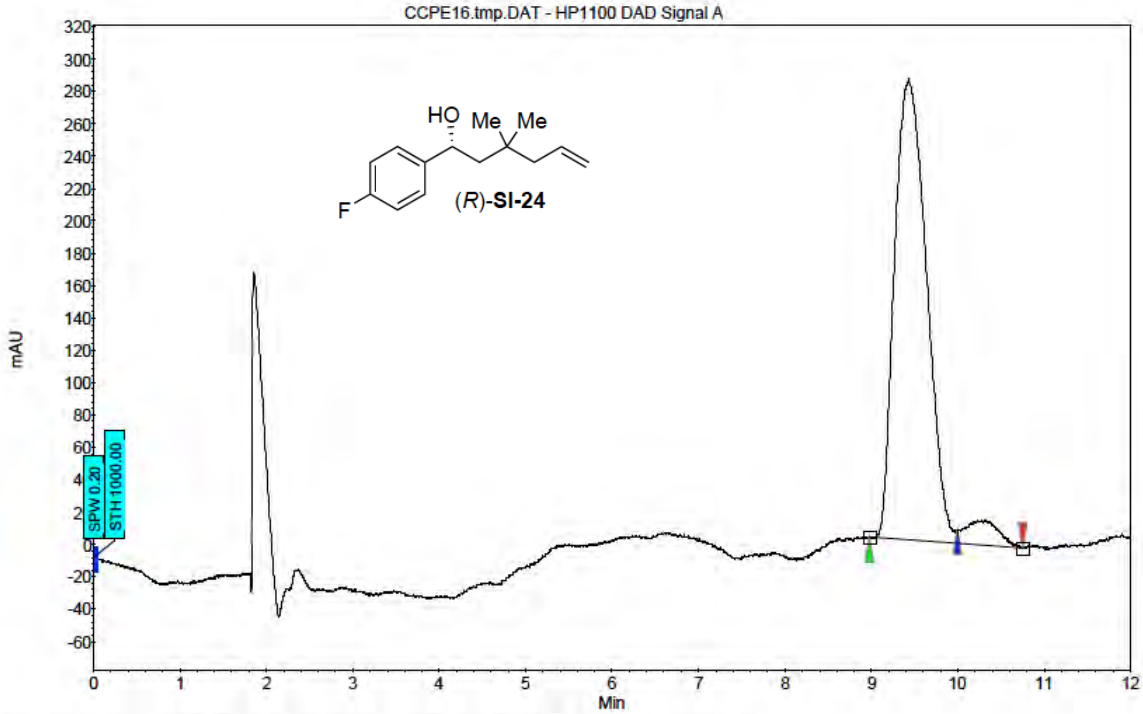
1	UNKNOWN	2.82	2.90	3.02	0.00	4.18	44.7	3.1	4.177
2	UNKNOWN	3.02	3.13	3.32	0.00	95.82	863.7	71.0	95.823
Total						100.00	908.4	74.1	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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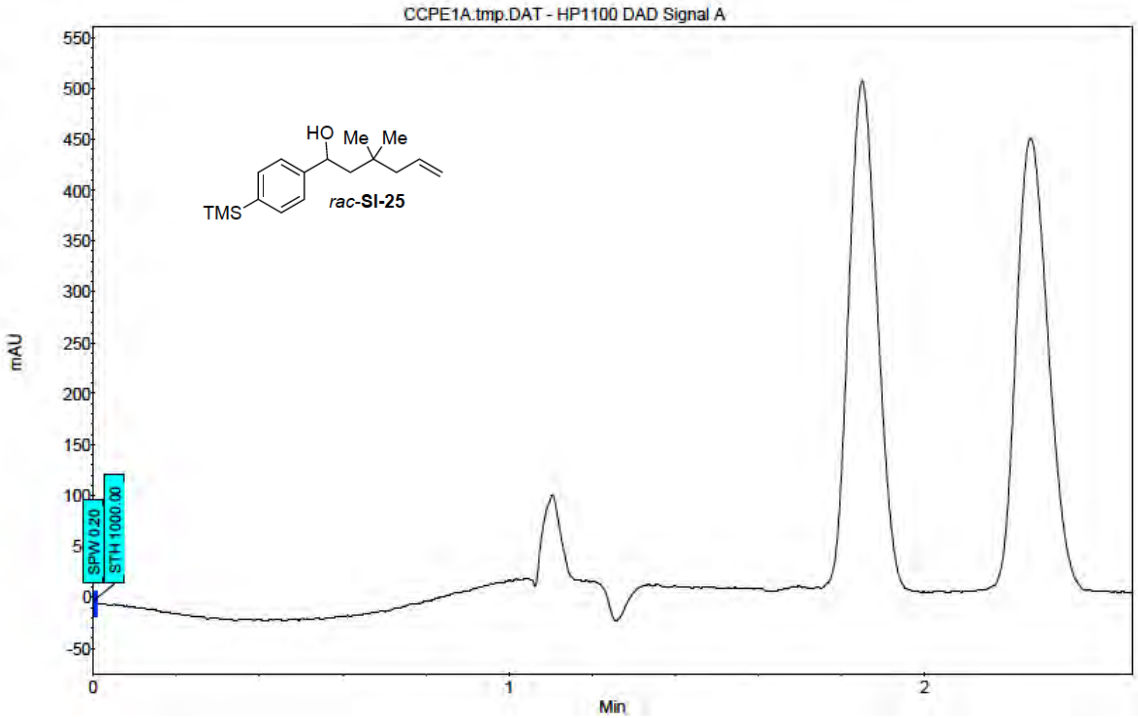
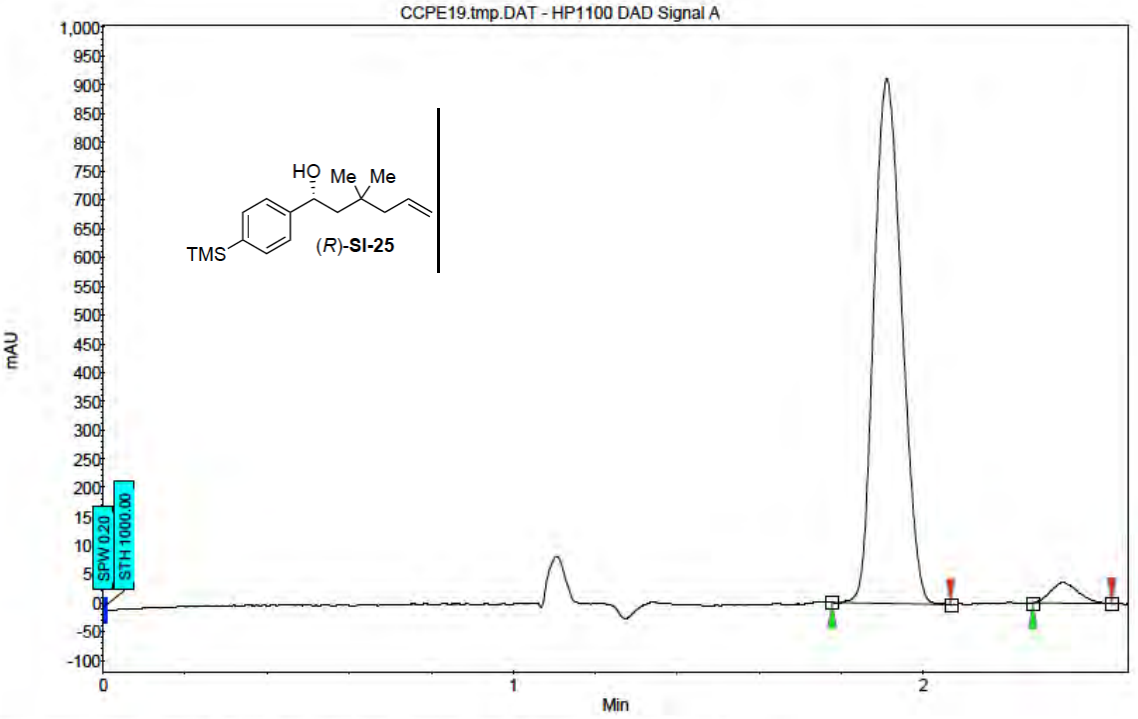
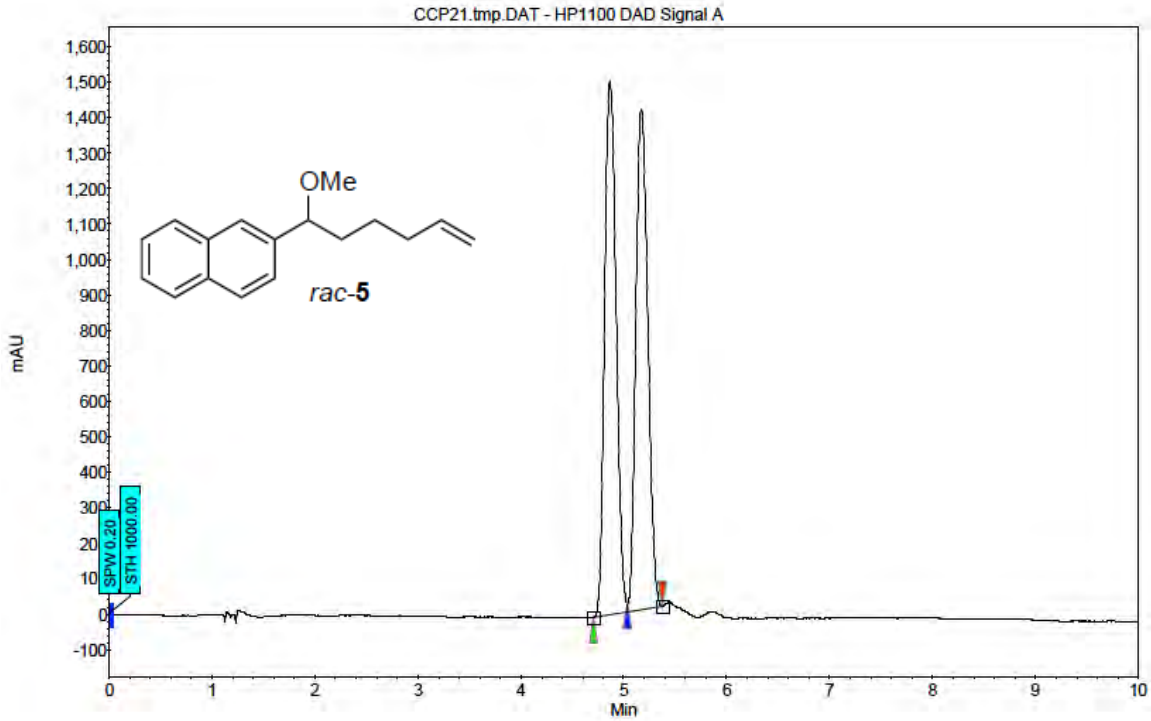
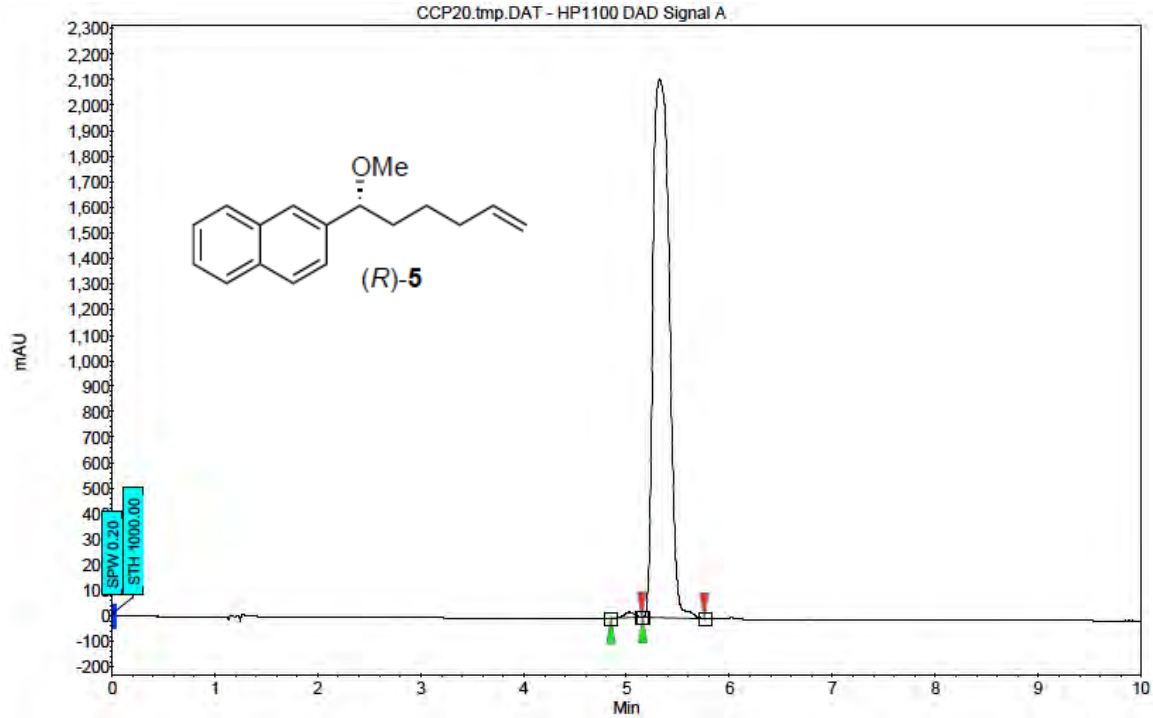
1	UNKNOWN	8.98	9.43	9.99	0.00	94.74	284.7	121.7	94.739
2	UNKNOWN	9.99	10.31	10.75	0.00	5.26	15.5	6.8	5.261
Total						100.00	300.3	128.4	100.000



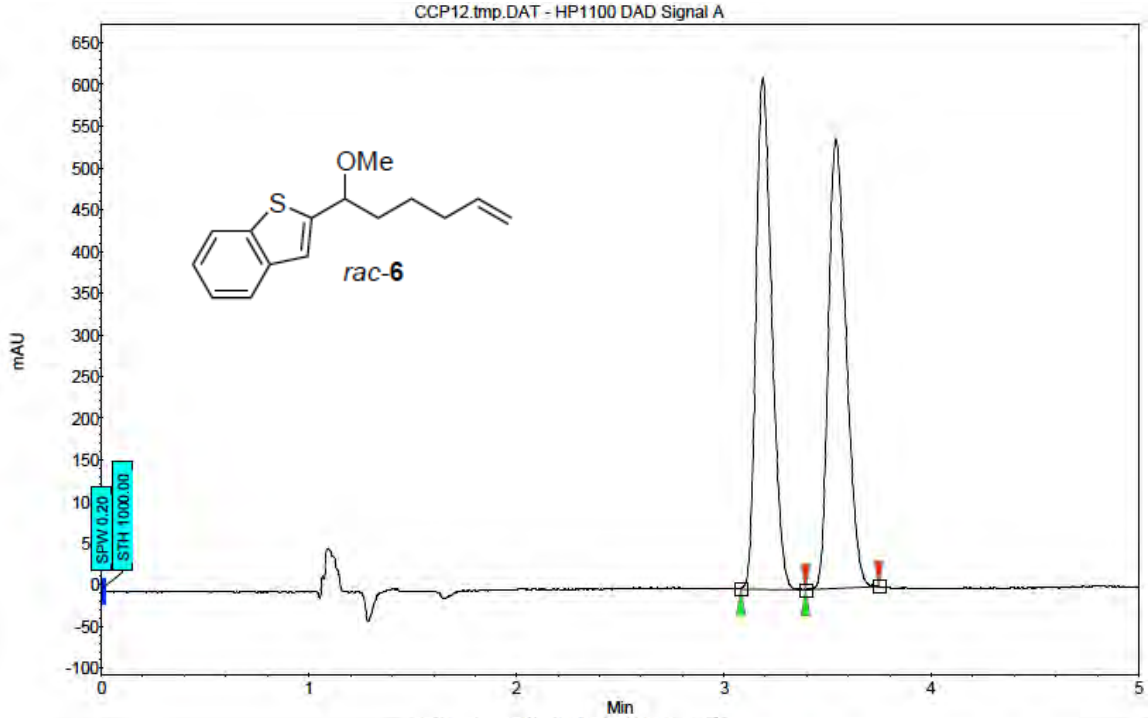


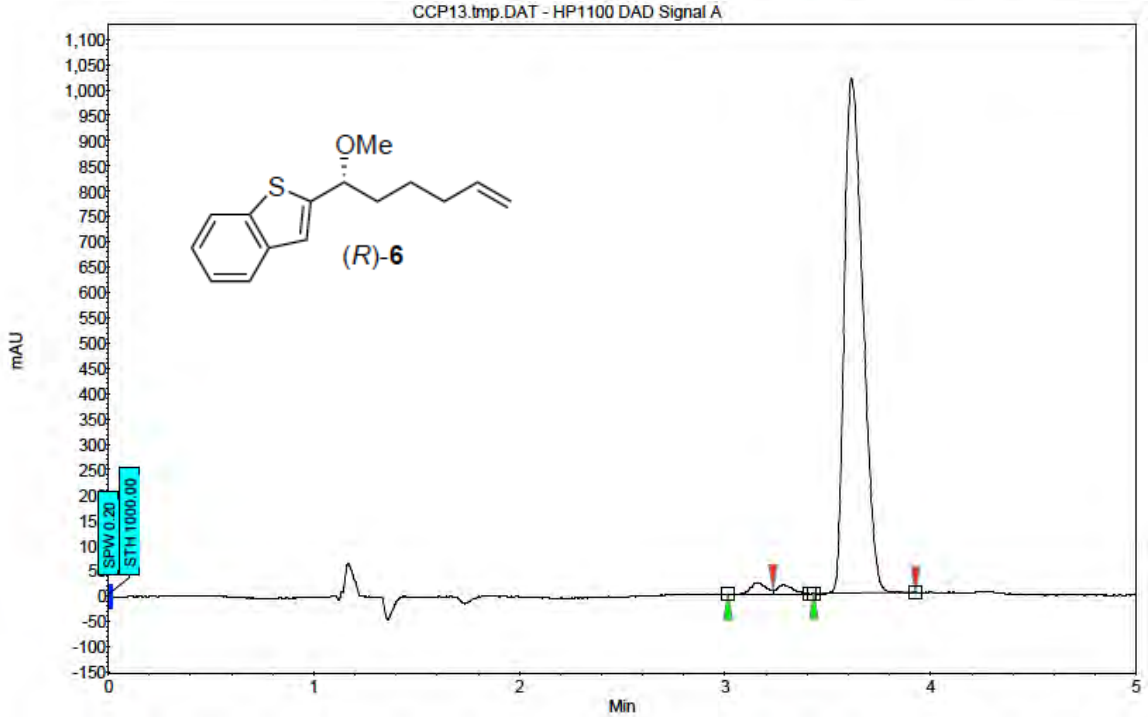
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.78	1.91	2.07	0.00	96.21	909.9	71.4	96.212
2	UNKNOWN	2.27	2.34	2.46	0.00	3.79	35.3	2.8	3.788
Total						100.00	945.1	74.2	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	4.85	5.03	5.15	0.00	0.64	21.6	2.5	0.637
2	UNKNOWN	5.16	5.33	5.76	0.00	99.36	2105.2	384.1	99.363
Total						100.00	2126.8	386.5	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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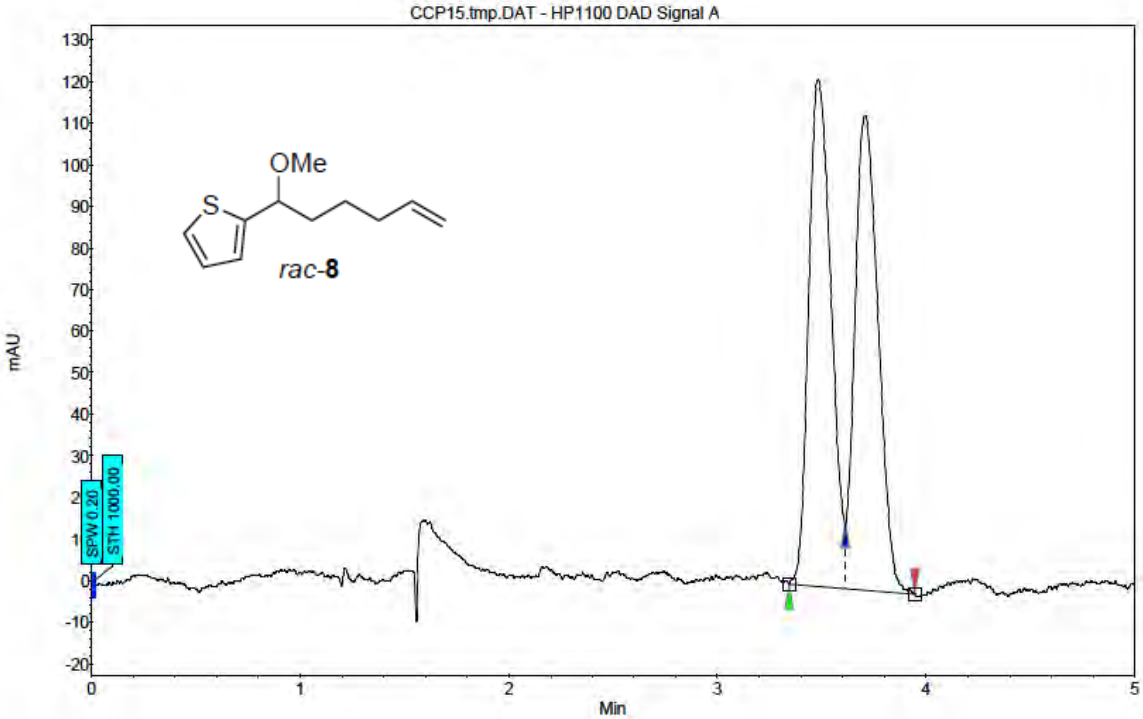
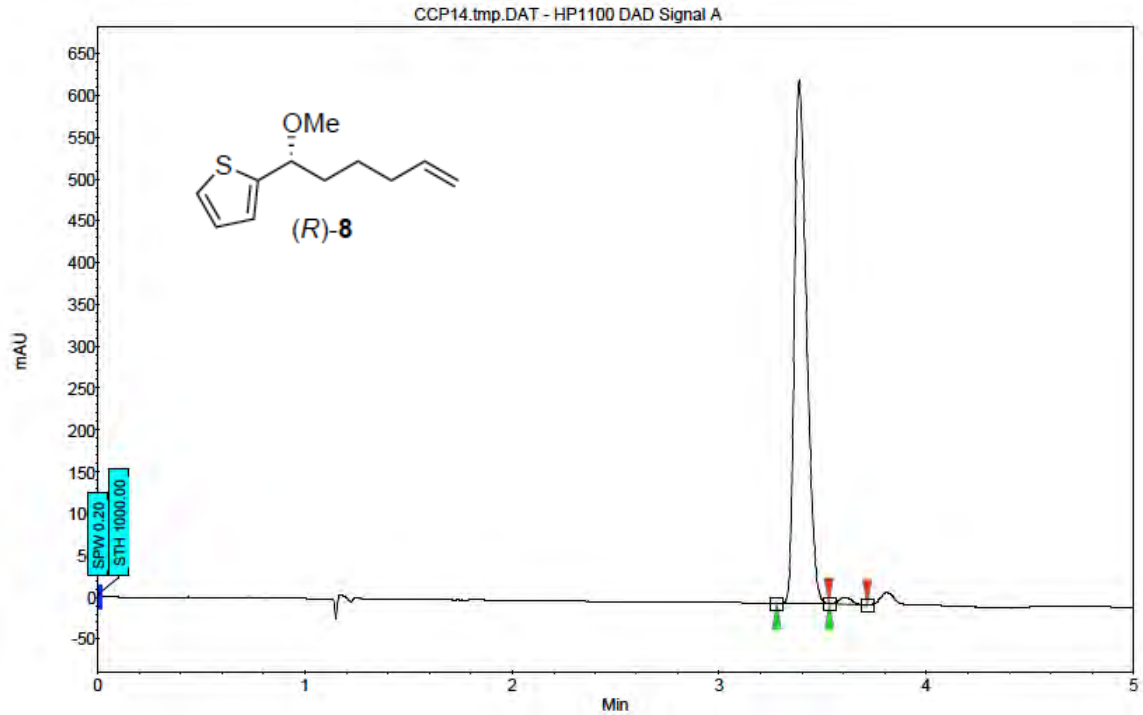
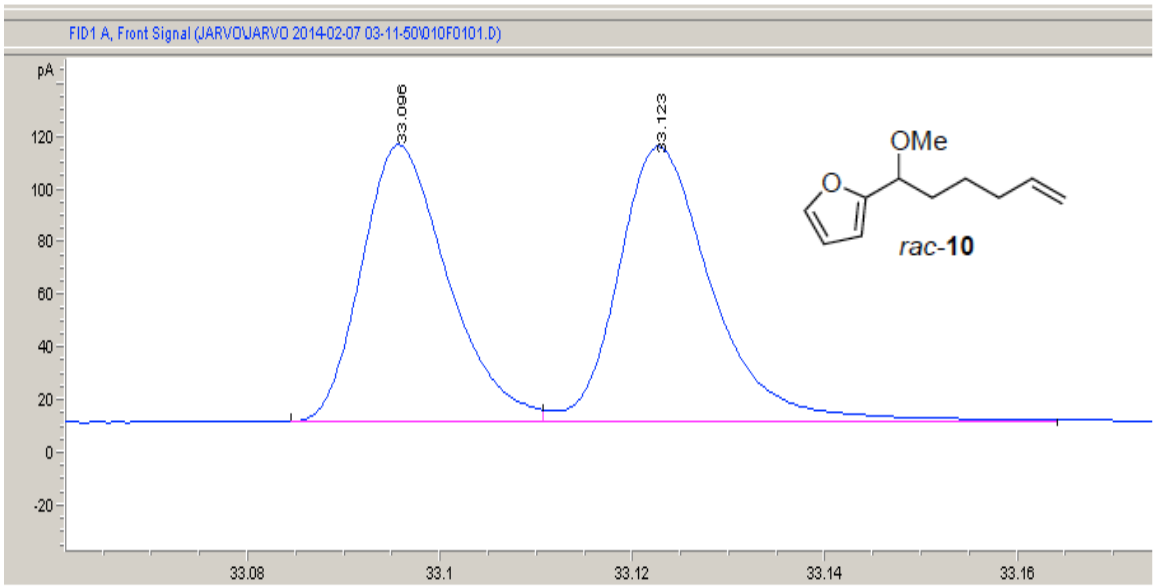
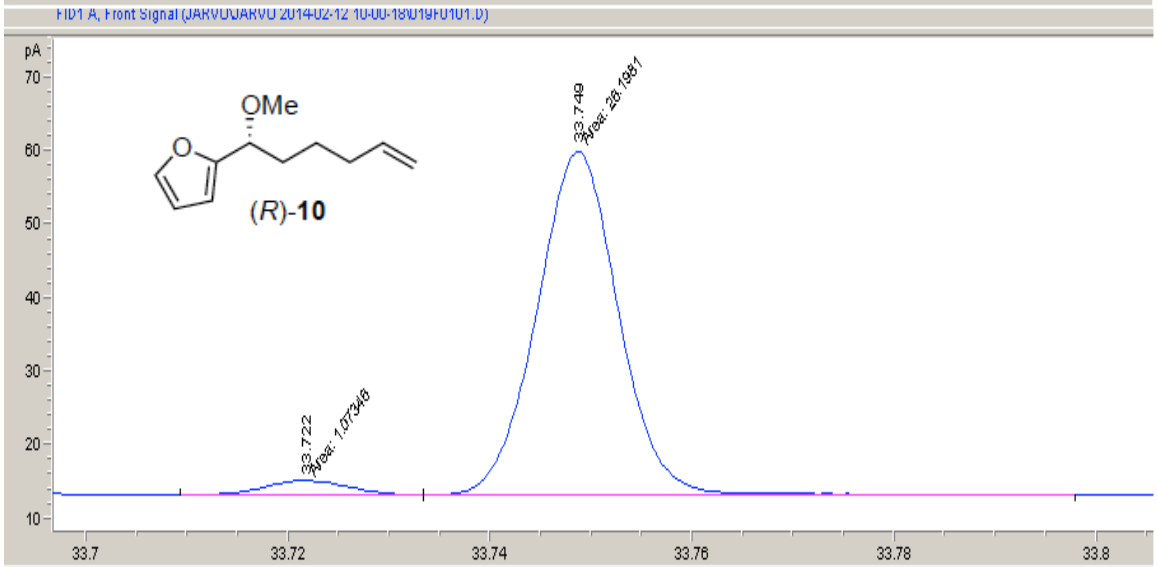
2	UNKNOWN	3.01	3.16	3.23	0.00	1.67	21.4	1.8	1.665
1	UNKNOWN	3.43	3.61	3.93	0.00	98.33	1016.6	106.5	98.335
Total						100.00	1038.0	108.3	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	3.28	3.39	3.53	0.00	98.70	624.7	42.8	98.700
2	UNKNOWN	3.53	3.61	3.71	0.00	1.30	8.3	0.6	1.300
Total						100.00	633.0	43.4	100.000





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File Path	C:\CHEM32\1\DATA\JARVO\JARVO 2014-02-12 10-00-18\
Date	12-Feb-14, 10:03:30
Sample	MDKACH3PROPRAC

#	Time	Area	Height	Width	Area%	Symmetry
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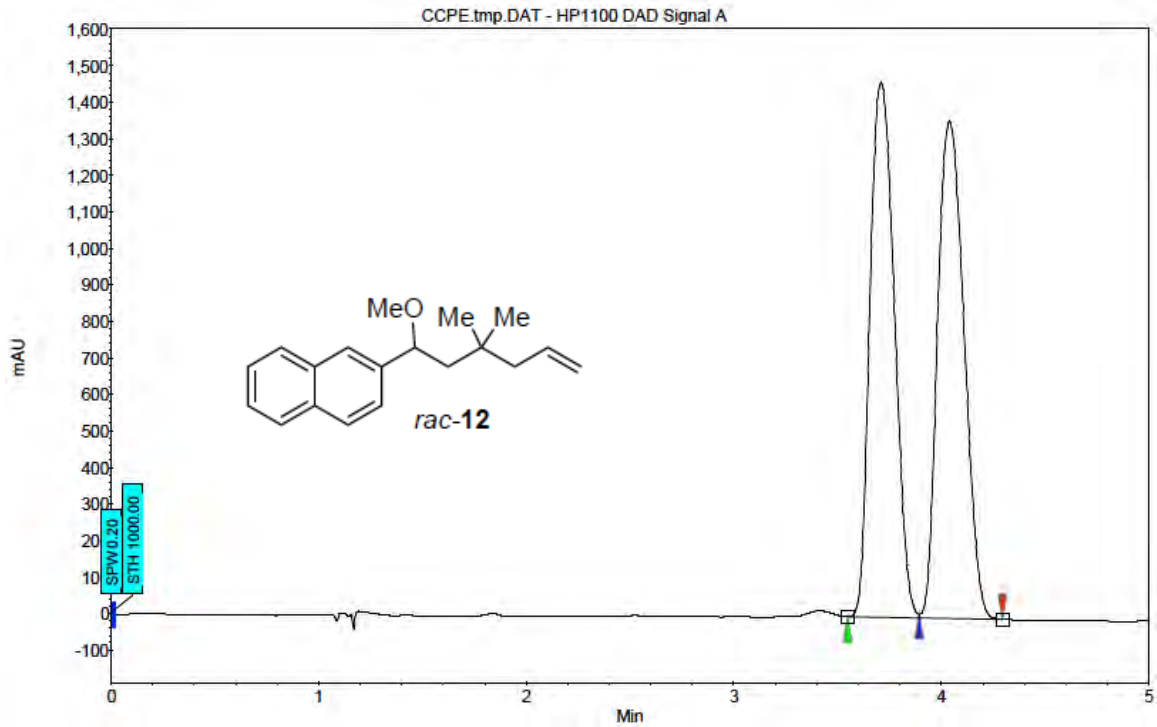
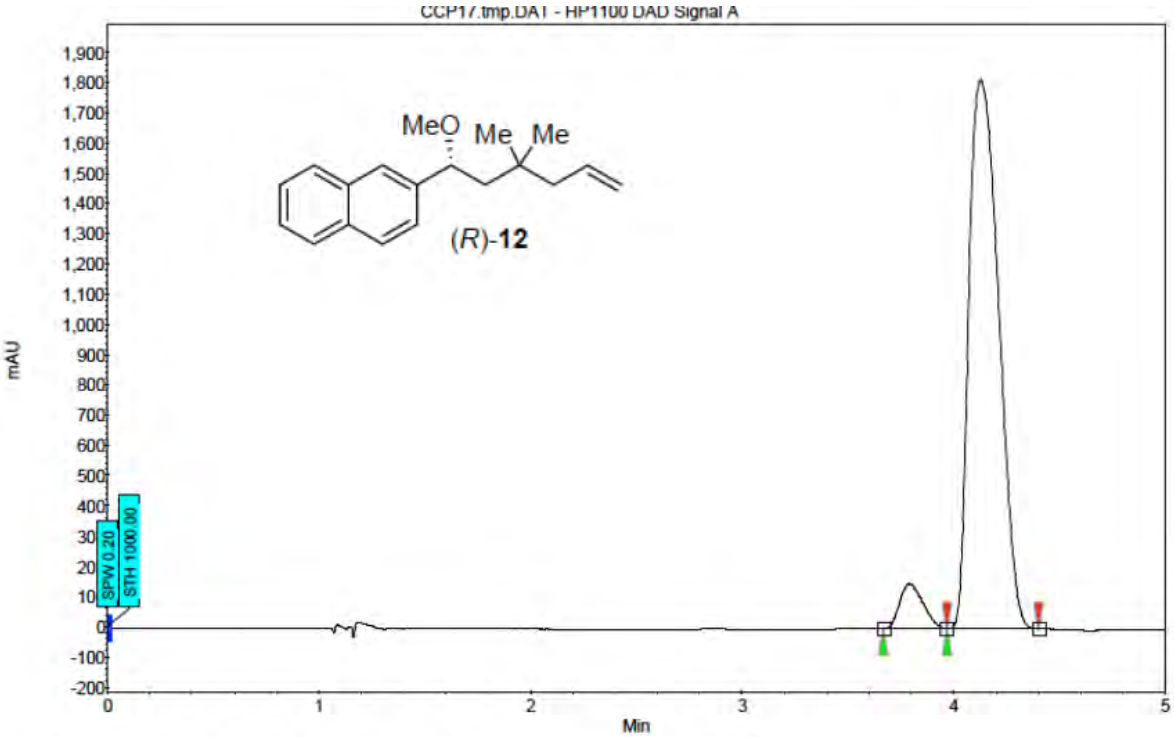
2	33.749	26.2	47.1	9.2779E-3	96.064	1.414





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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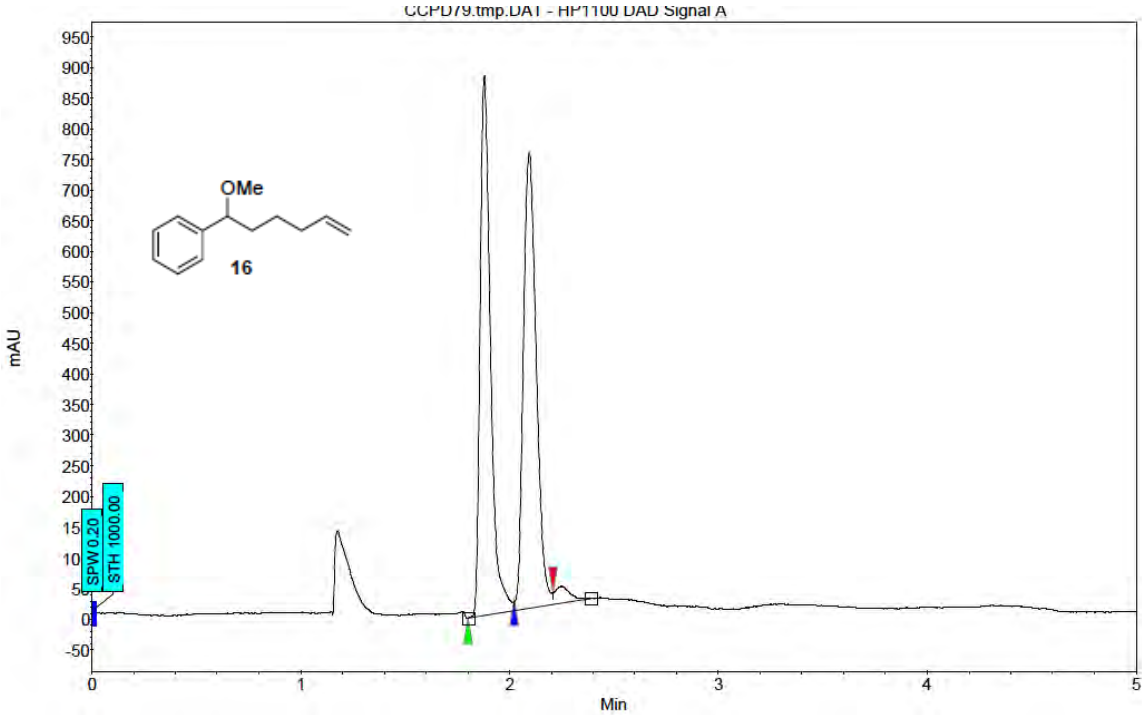
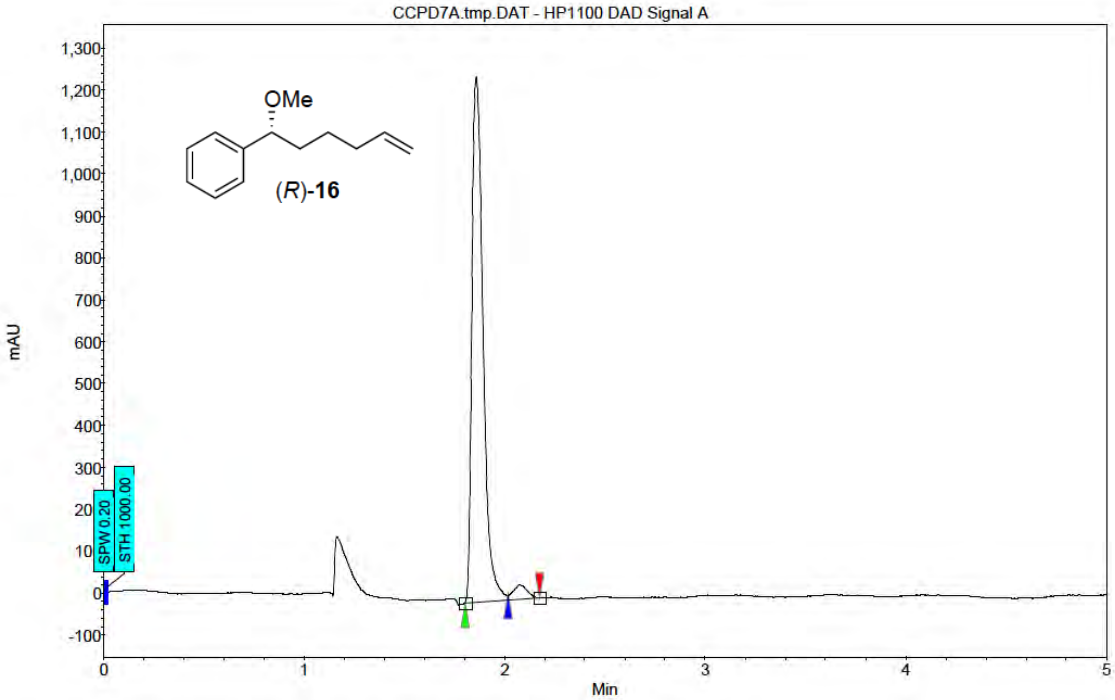
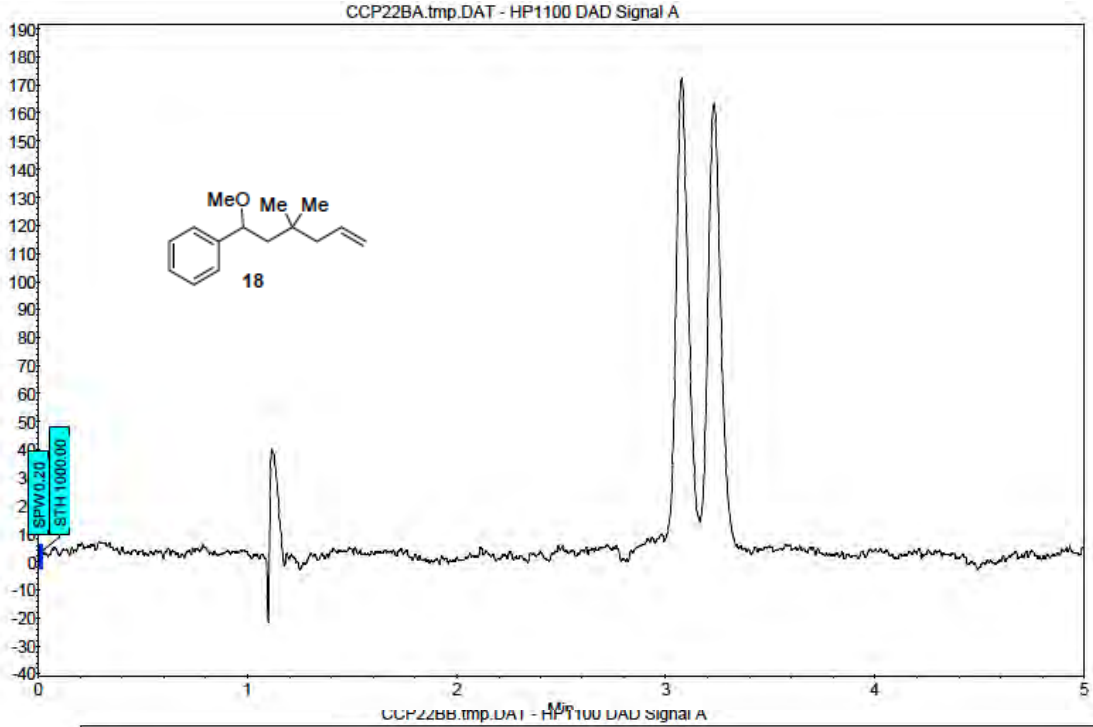
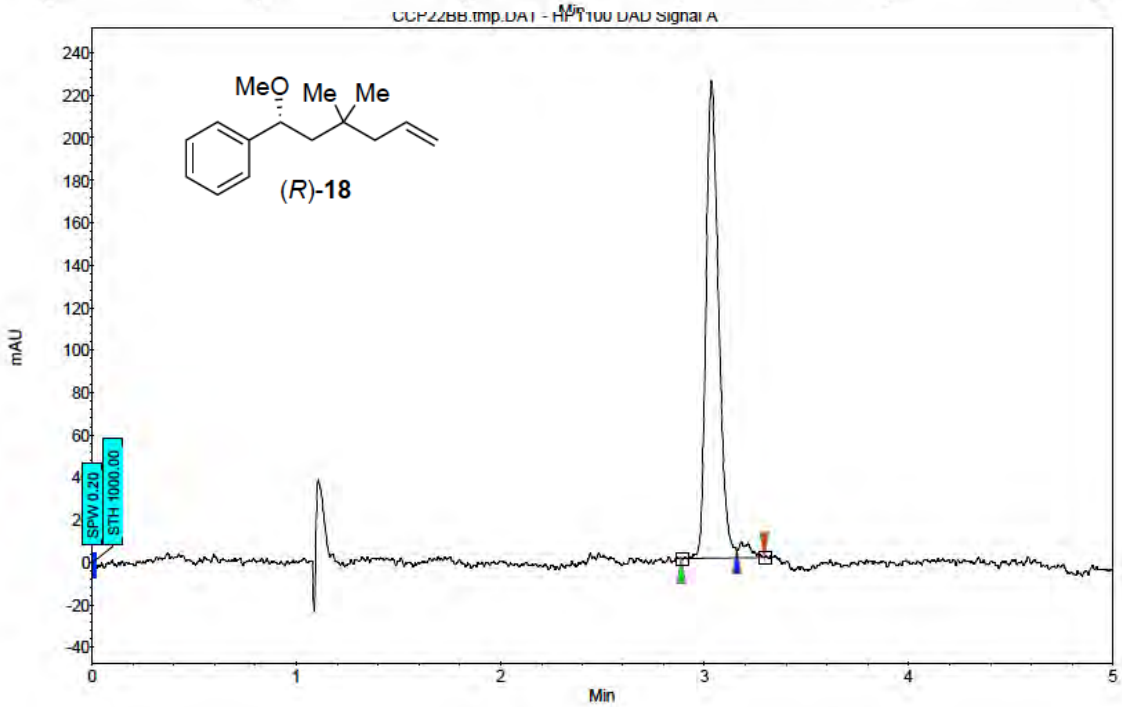
1	UNKNOWN	3.67	3.79	3.97	0.00	6.13	149.5	19.3	6.125
2	UNKNOWN	3.97	4.13	4.40	0.00	93.87	1815.0	295.1	93.875
Total						100.00	1964.4	314.4	100.000



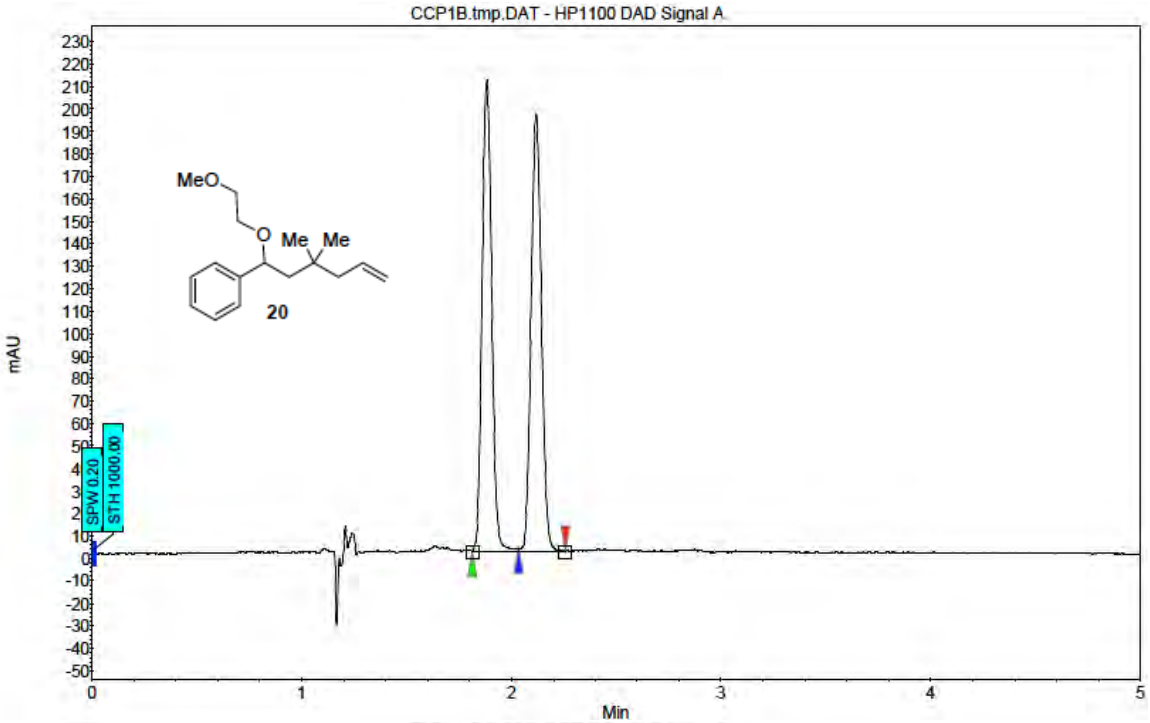


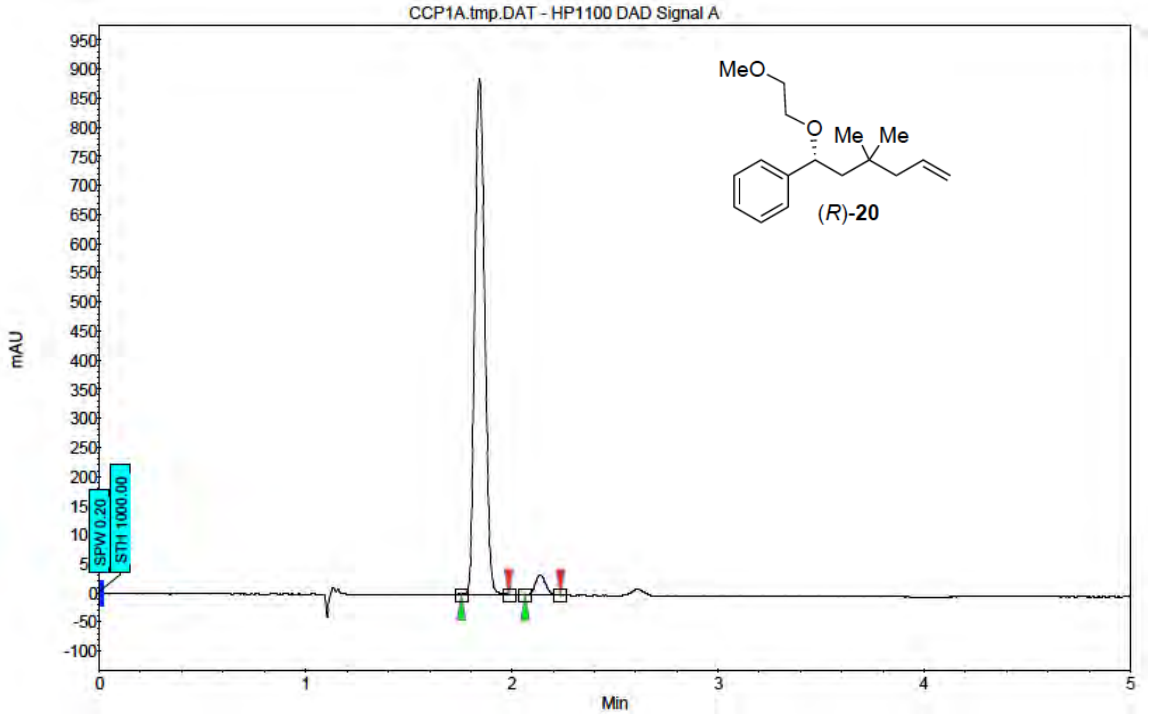
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.80	1.86	2.01	0.00	96.65	1252.1	80.4	96.654
2	UNKNOWN	2.01	2.07	2.17	0.00	3.35	34.3	2.8	3.346
Total						100.00	1286.4	83.2	100.000



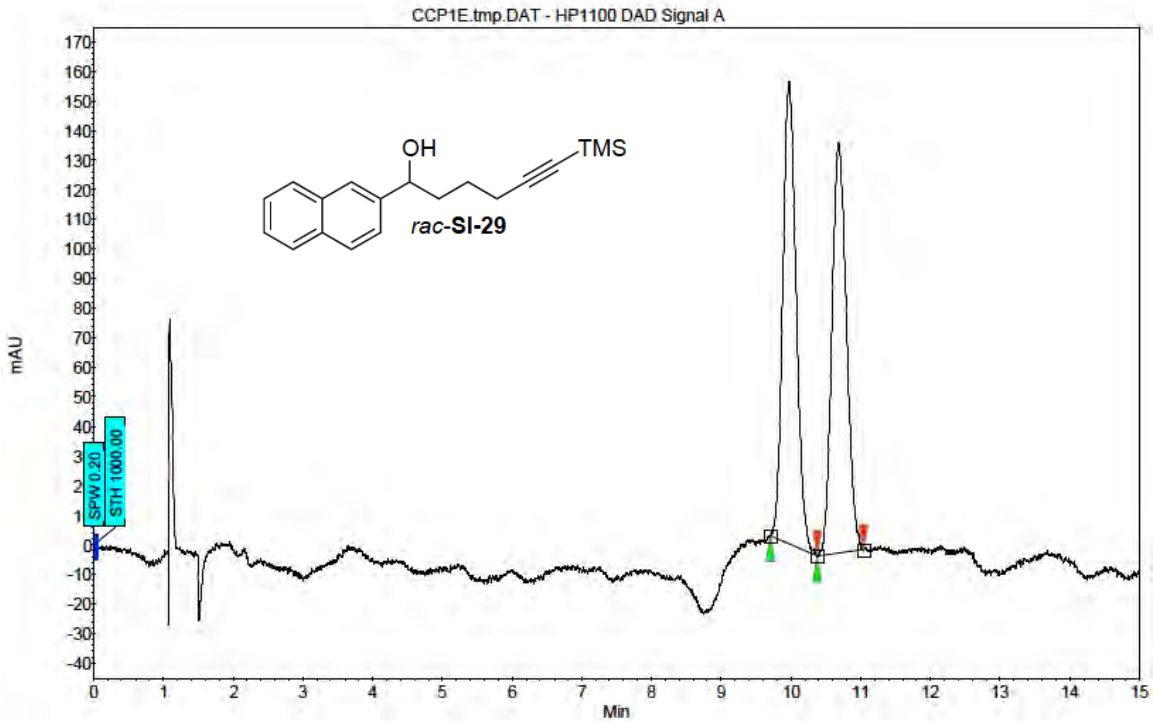


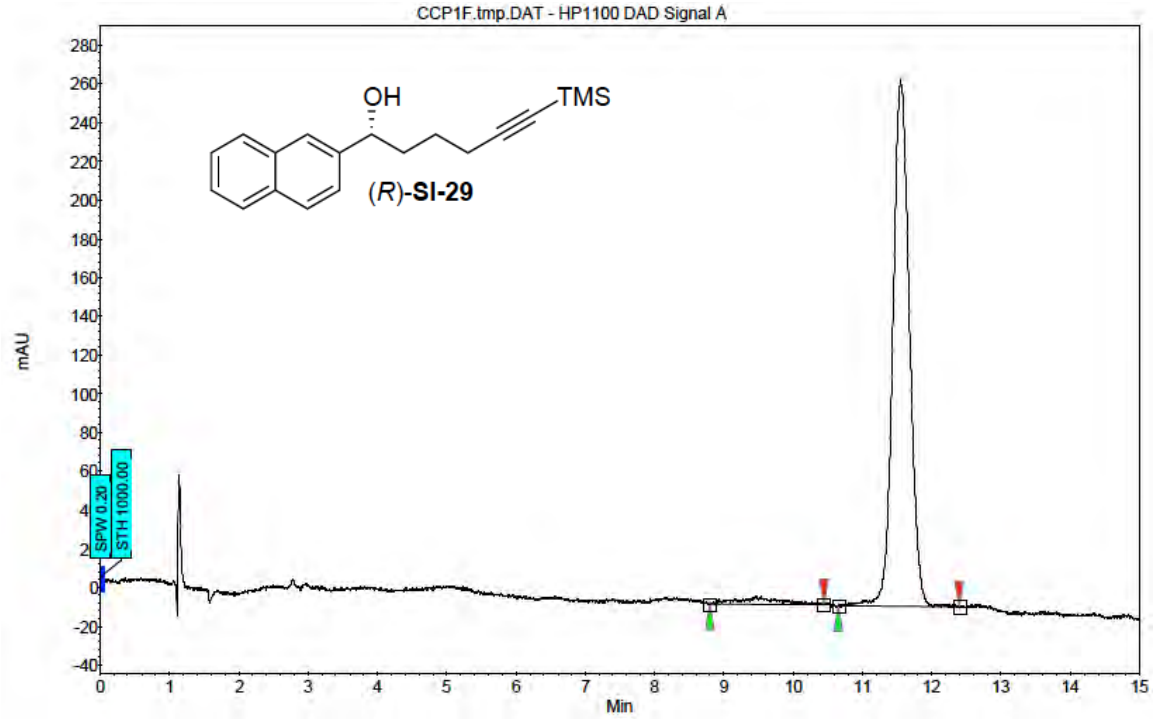
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.89	3.04	3.16	0.00	97.07	224.4	15.9	97.073
2	UNKNOWN	3.16	3.18	3.29	0.00	2.93	7.3	0.5	2.927
Total						100.00	231.8	16.3	100.000



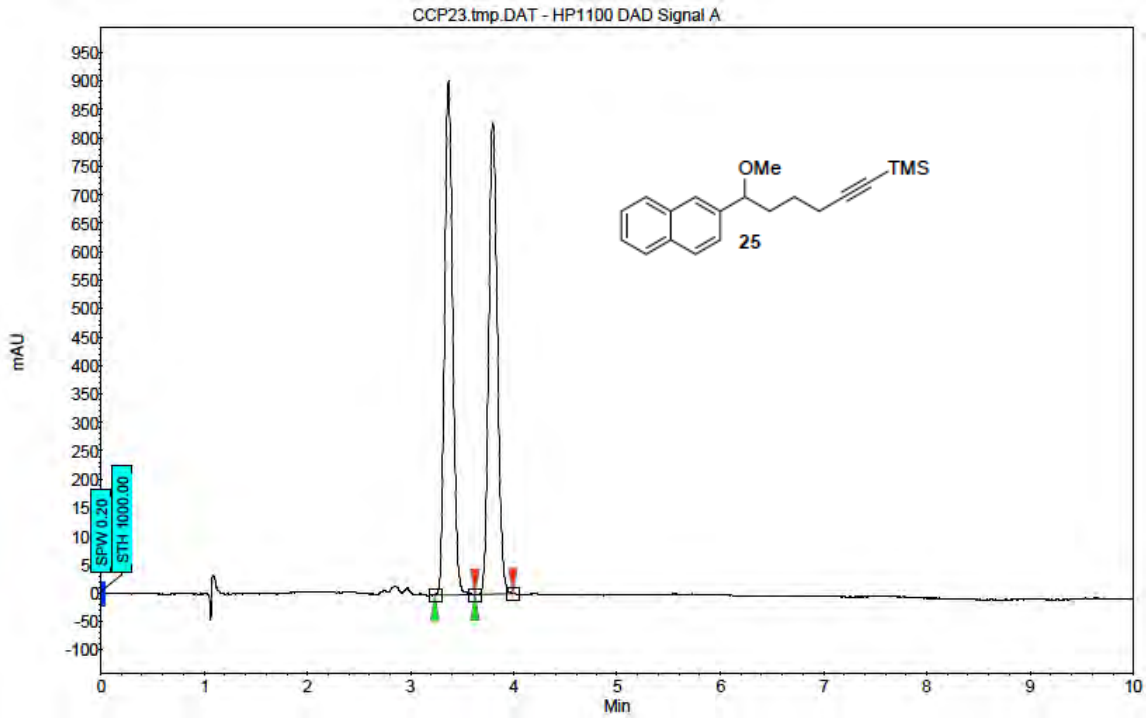


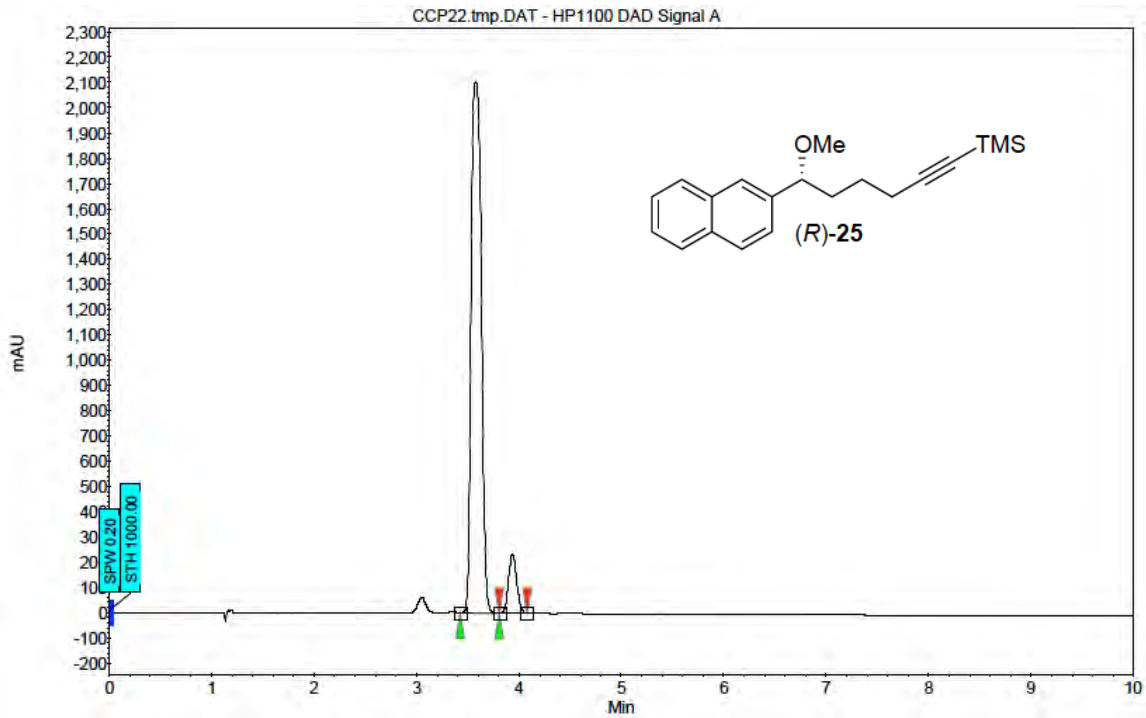
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.76	1.84	1.98	0.00	96.07	884.4	49.2	96.068
2	UNKNOWN	2.06	2.14	2.24	0.00	3.93	34.9	2.0	3.932
Total						100.00	919.3	51.2	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	8.80	9.54	10.44	0.00	3.31	3.7	2.6	3.309
2	UNKNOWN	10.65	11.55	12.39	0.00	96.69	271.7	75.3	96.691
Total						100.00	275.5	77.9	100.000





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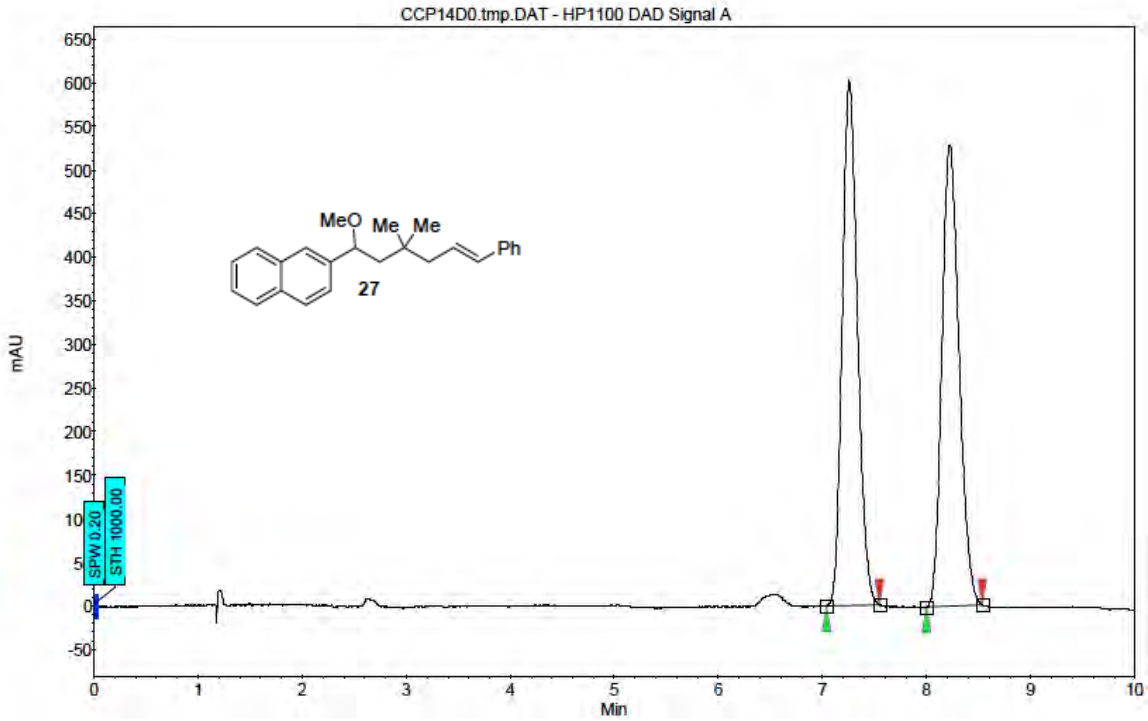
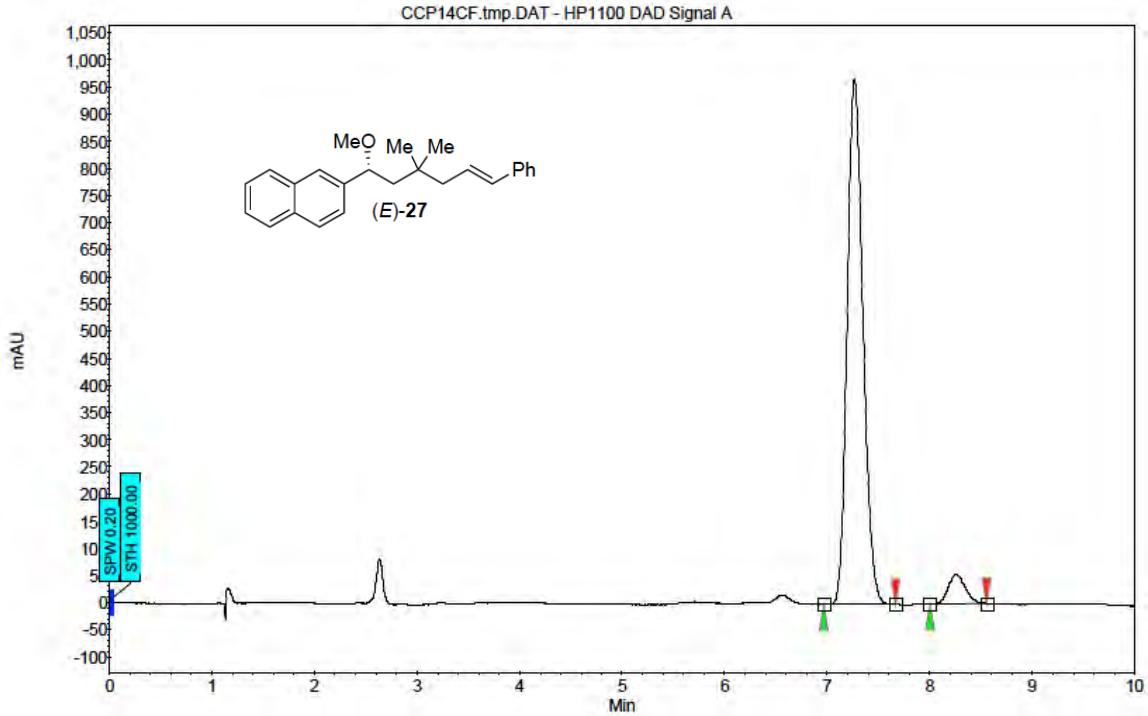
1	UNKNOWN	3.43	3.57	3.81	0.00	91.73	2101.7	240.9	91.735
2	UNKNOWN	3.81	3.94	4.08	0.00	8.27	237.0	21.7	8.265
Total						100.00	2338.7	262.6	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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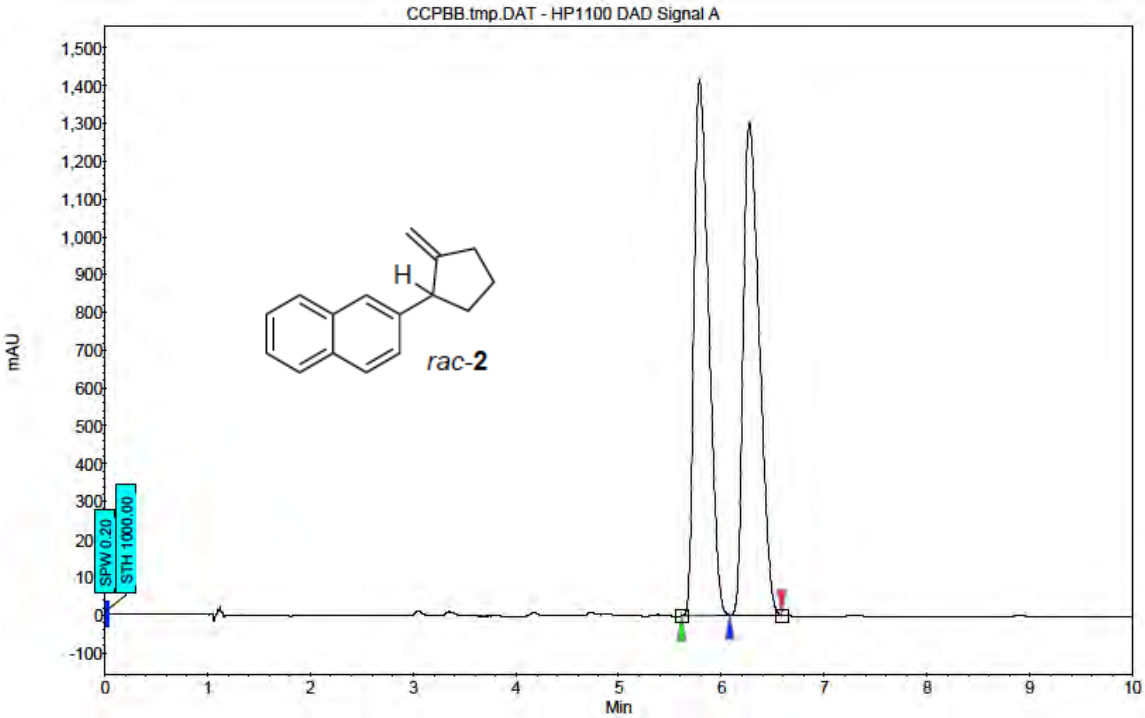
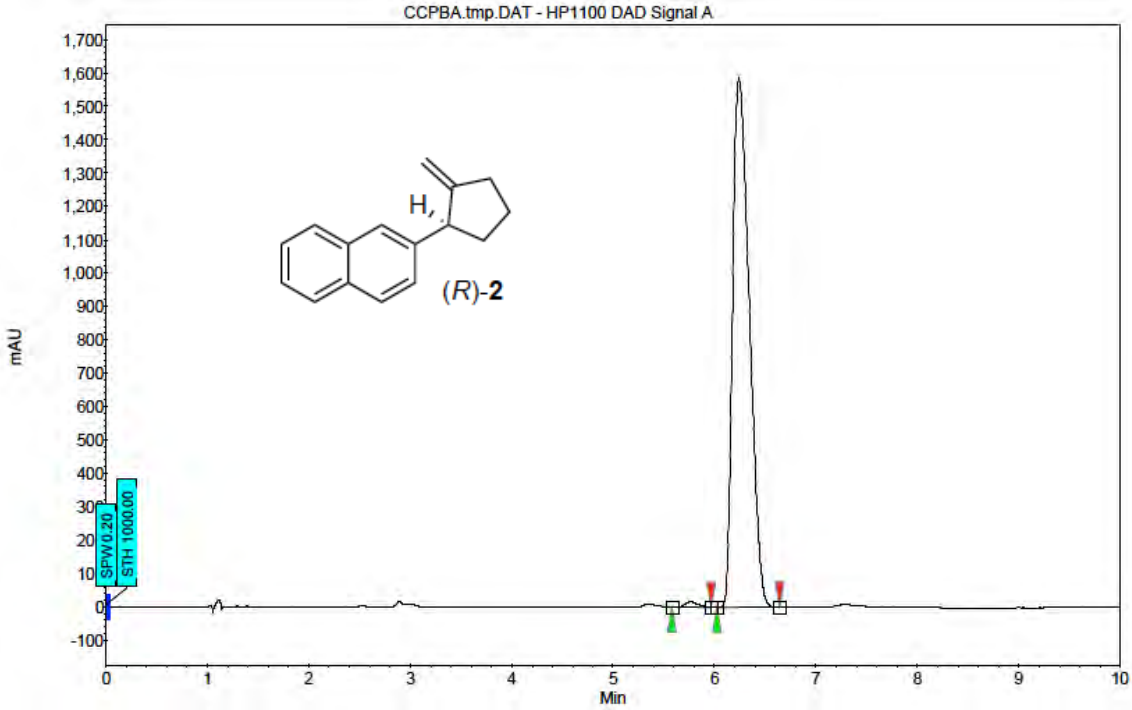
1	UNKNOWN	6.97	7.27	7.67	0.00	94.32	967.1	172.6	94.317
2	UNKNOWN	8.00	8.26	8.55	0.00	5.68	53.6	10.4	5.683
Total						100.00	1020.7	183.0	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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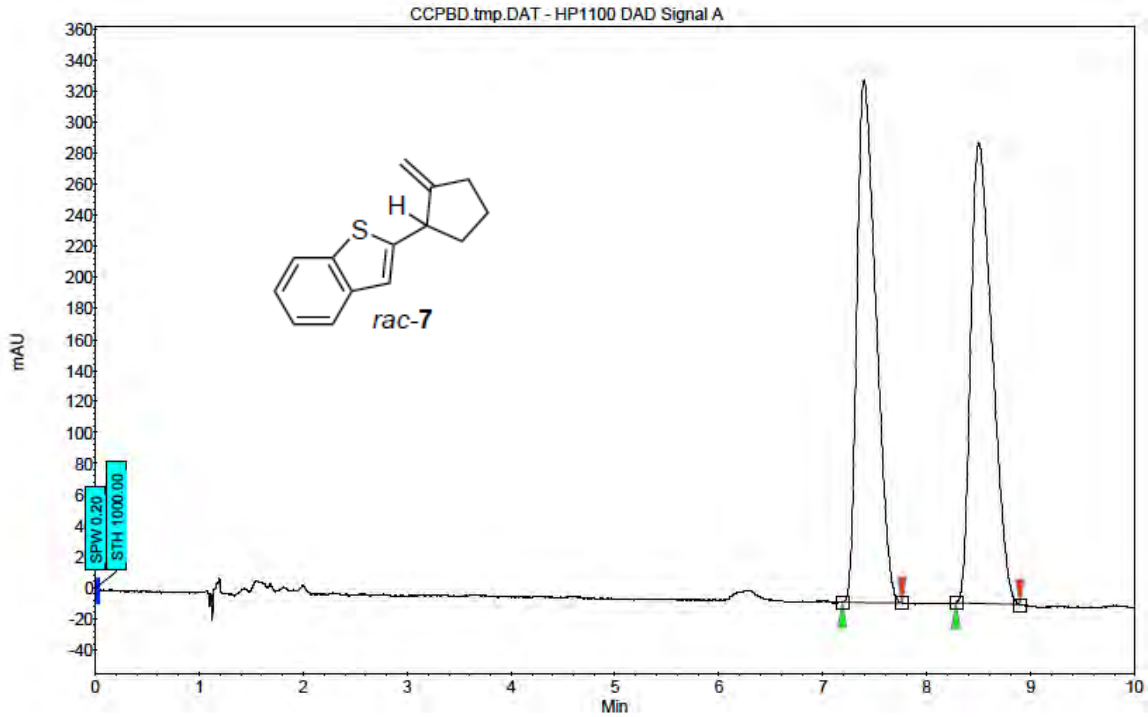
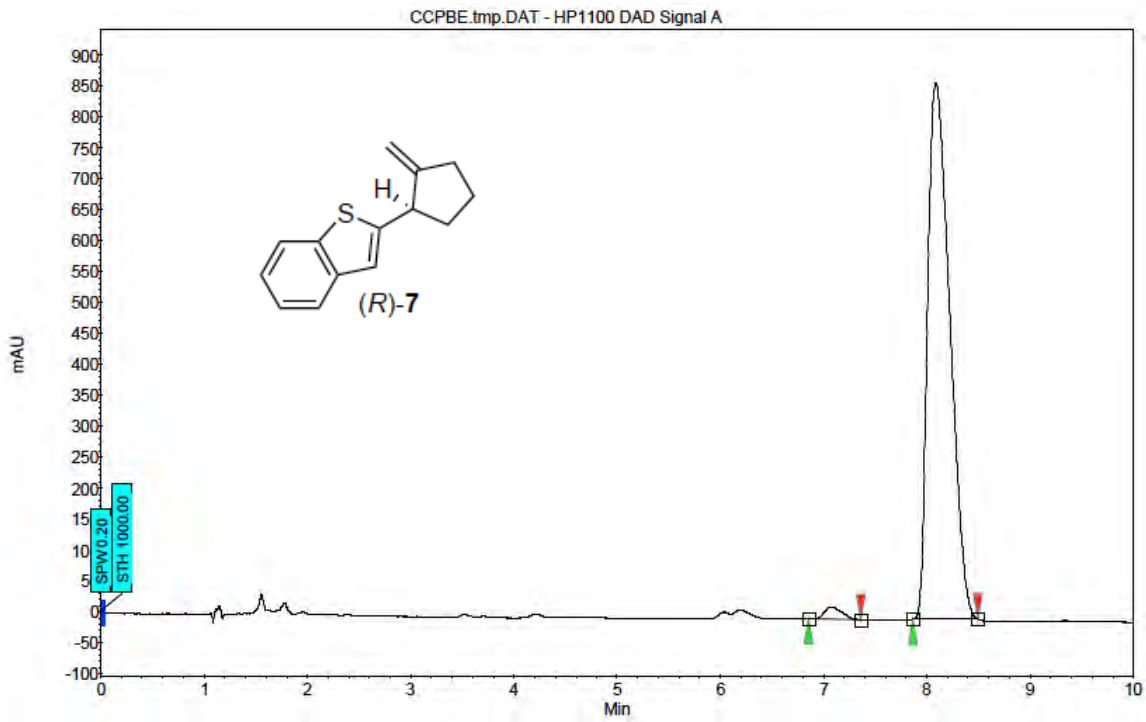
1	UNKNOWN	5.58	5.77	5.97	0.00	0.70	14.1	2.1	0.696
2	UNKNOWN	6.02	6.24	6.64	0.00	99.30	1585.0	301.4	99.304
Total						100.00	1599.1	303.5	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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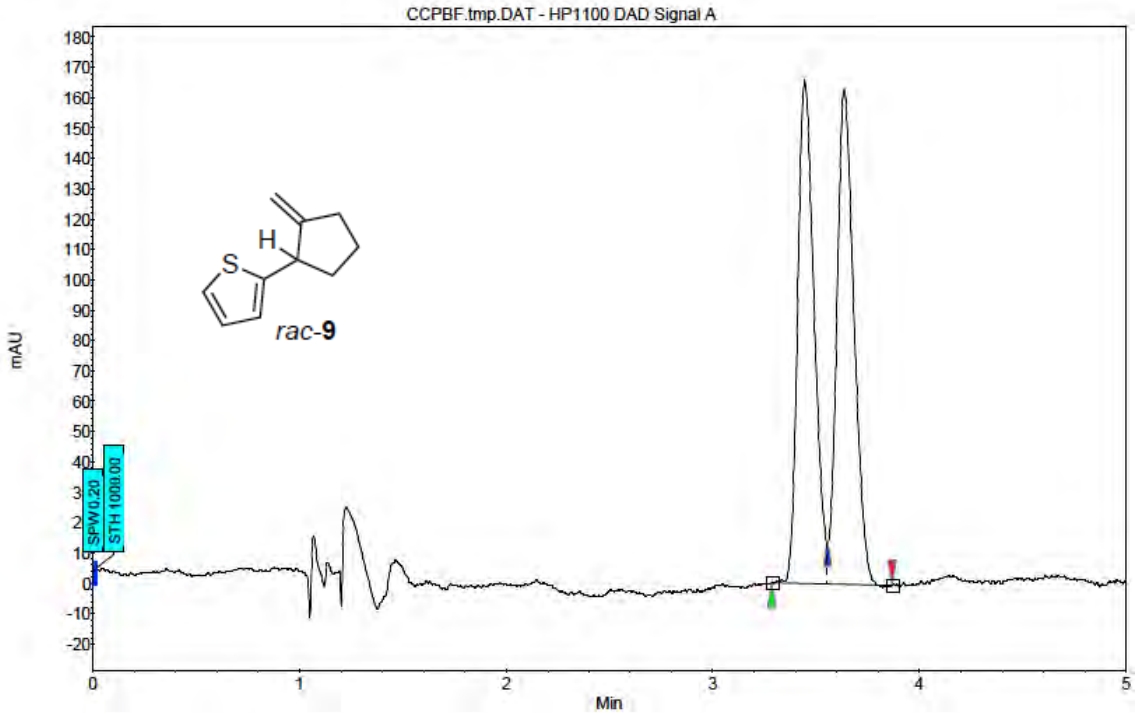
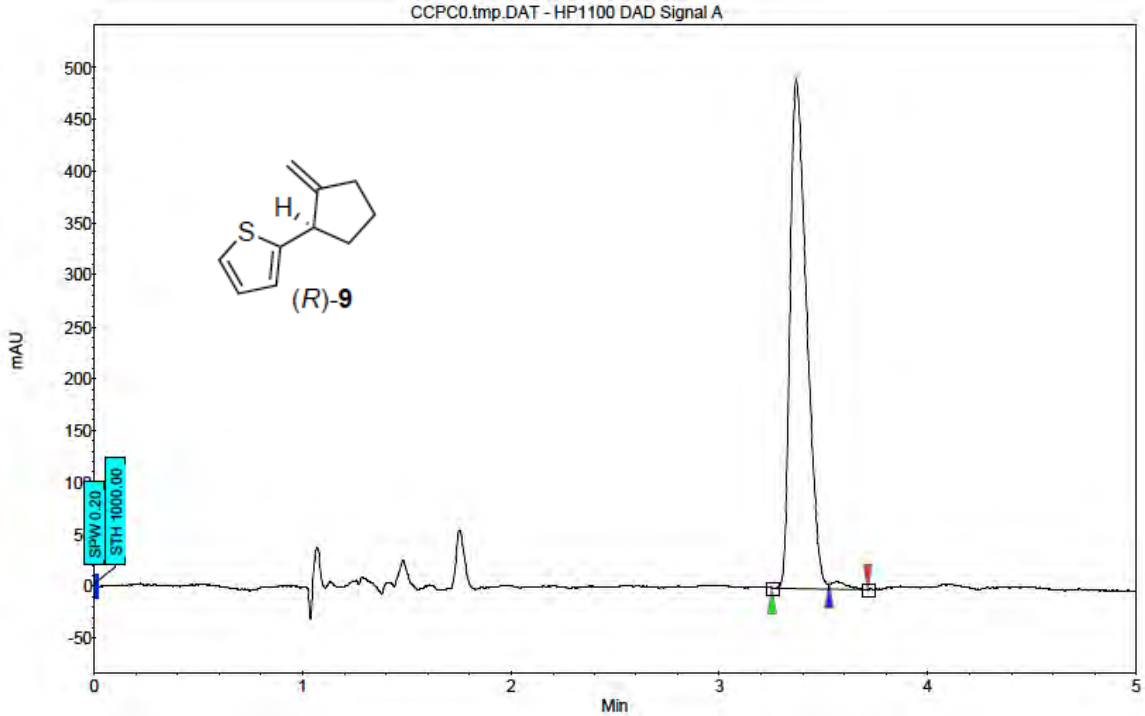
2	UNKNOWN	6.85	7.08	7.36	0.00	1.75	19.5	3.8	1.746
1	UNKNOWN	7.87	8.09	8.49	0.00	98.25	865.4	213.2	98.254
Total						100.00	885.0	217.0	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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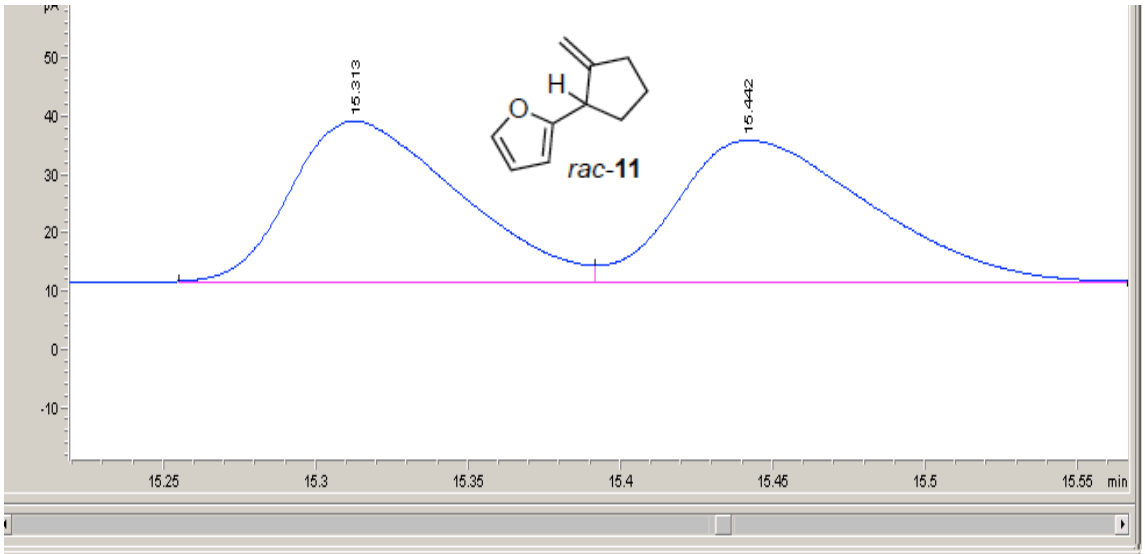
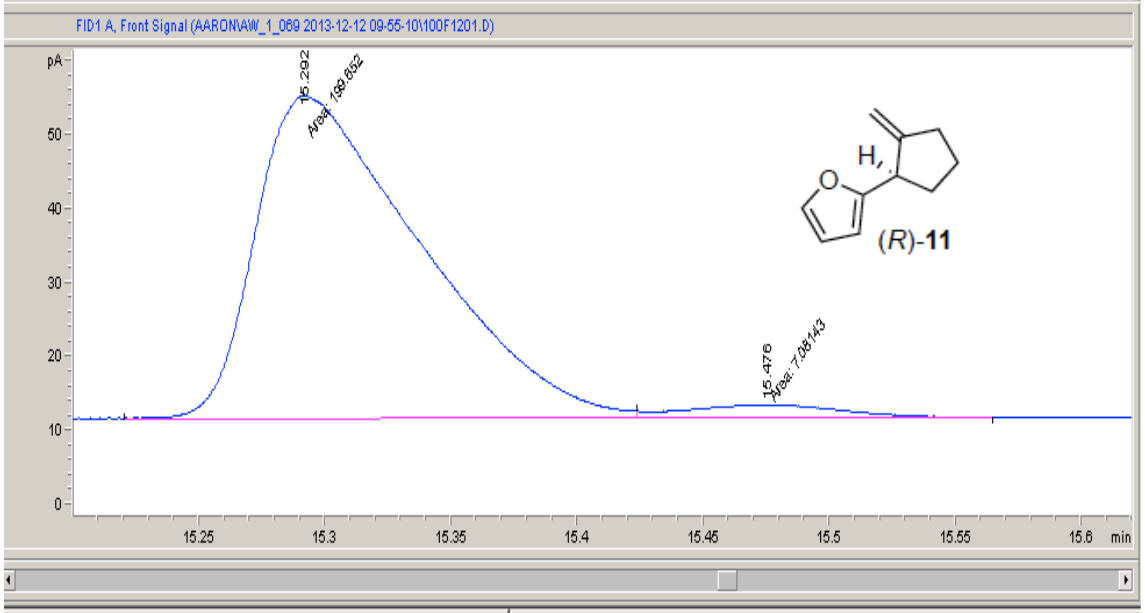
1	UNKNOWN	3.25	3.37	3.53	0.00	98.52	491.0	45.2	98.520
2	UNKNOWN	3.53	3.57	3.71	0.00	1.48	7.7	0.7	1.480
Total						100.00	498.7	45.9	100.000





File Information	
GC-File	100F1201.D
File Path	C:\CHEM32\1\DATA\AARON\AW_1_069 2013-12-12 09-55-10\
Date	12-Dec-13, 12:53:49
Sample	MRH-VI-12

#	Time	Area	Height	Width	Area%	Symmetry
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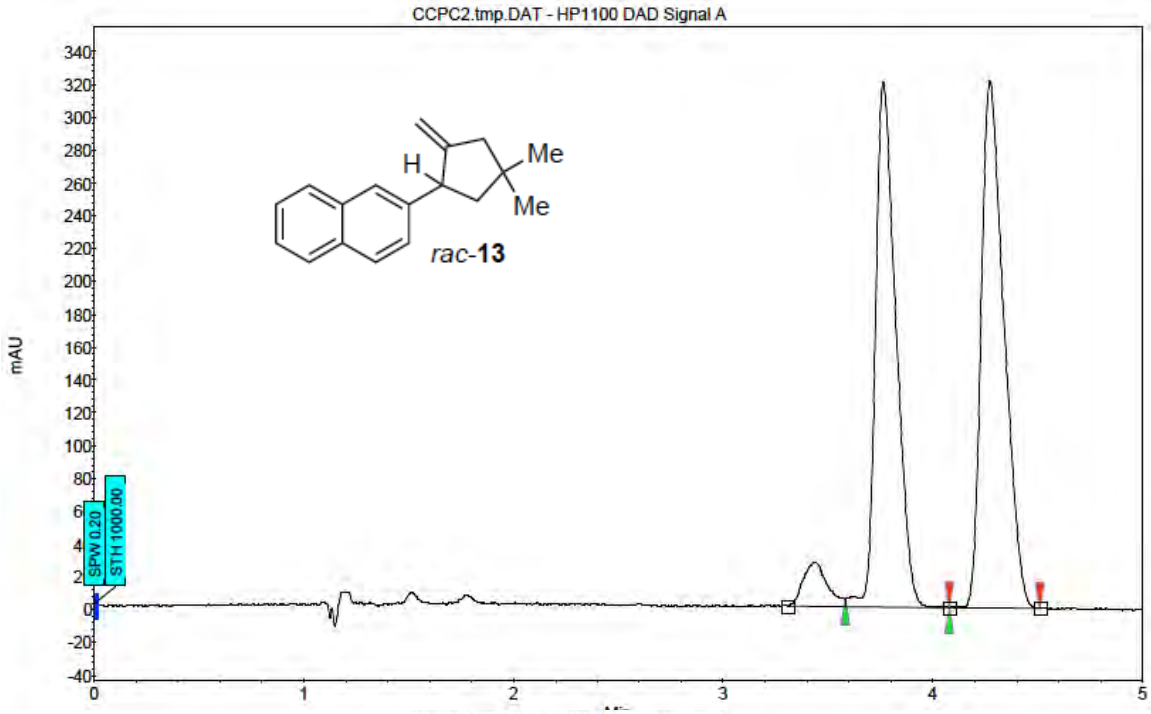
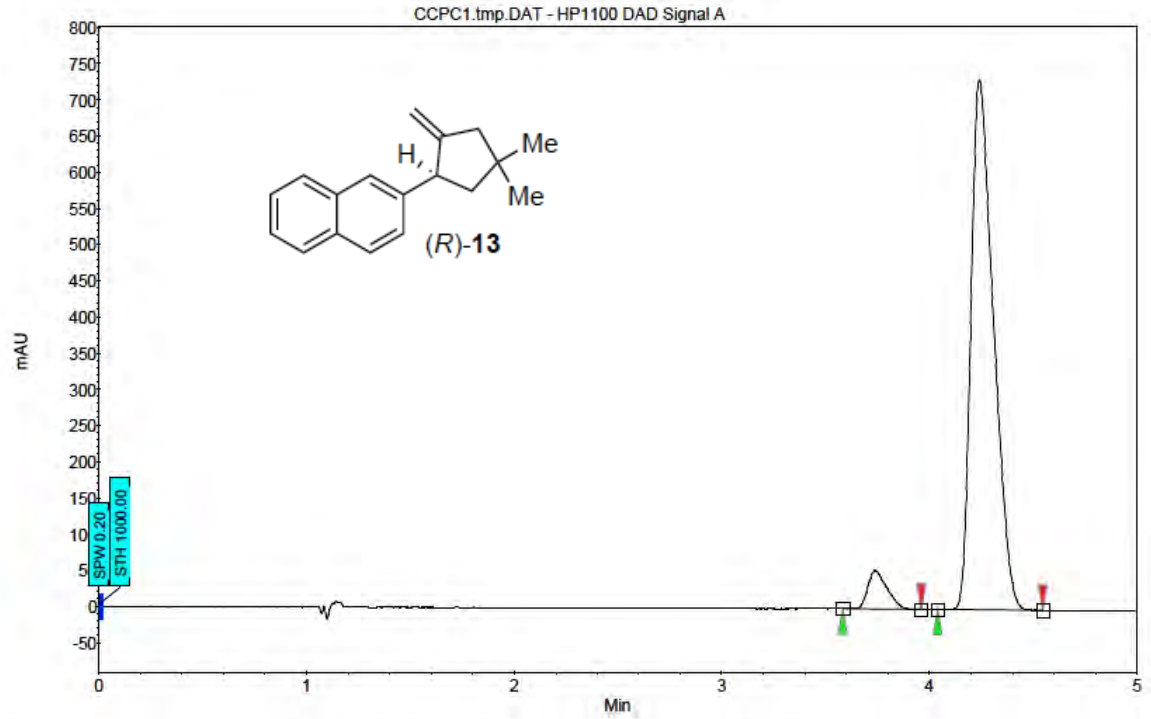
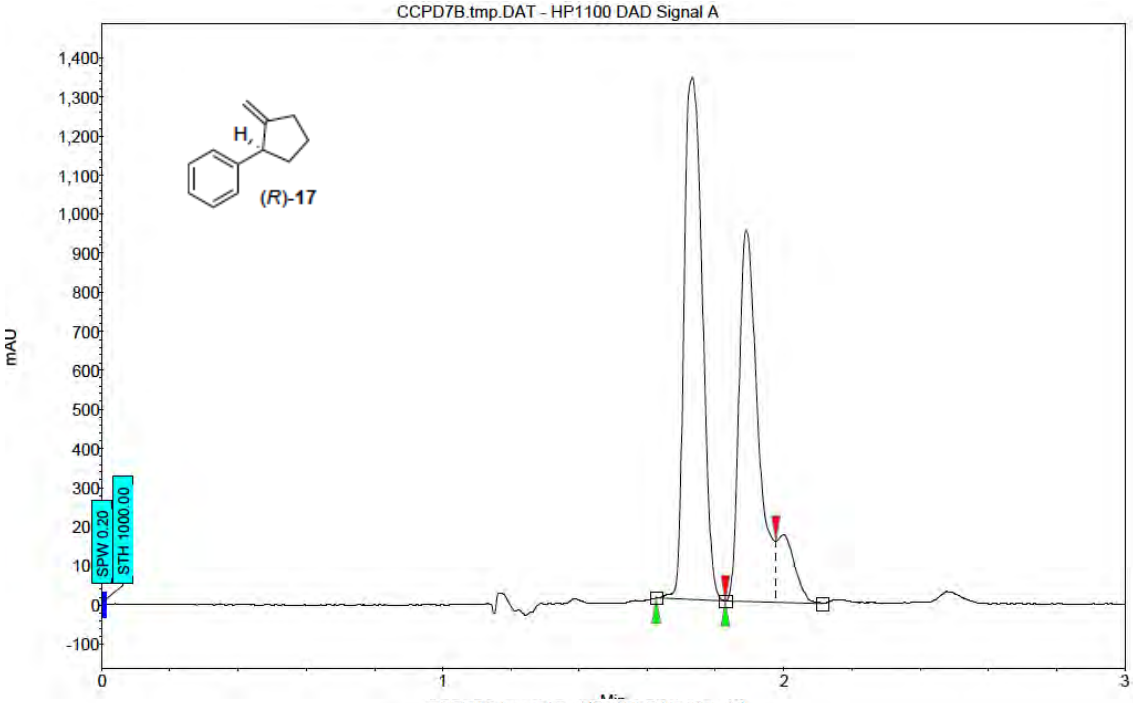
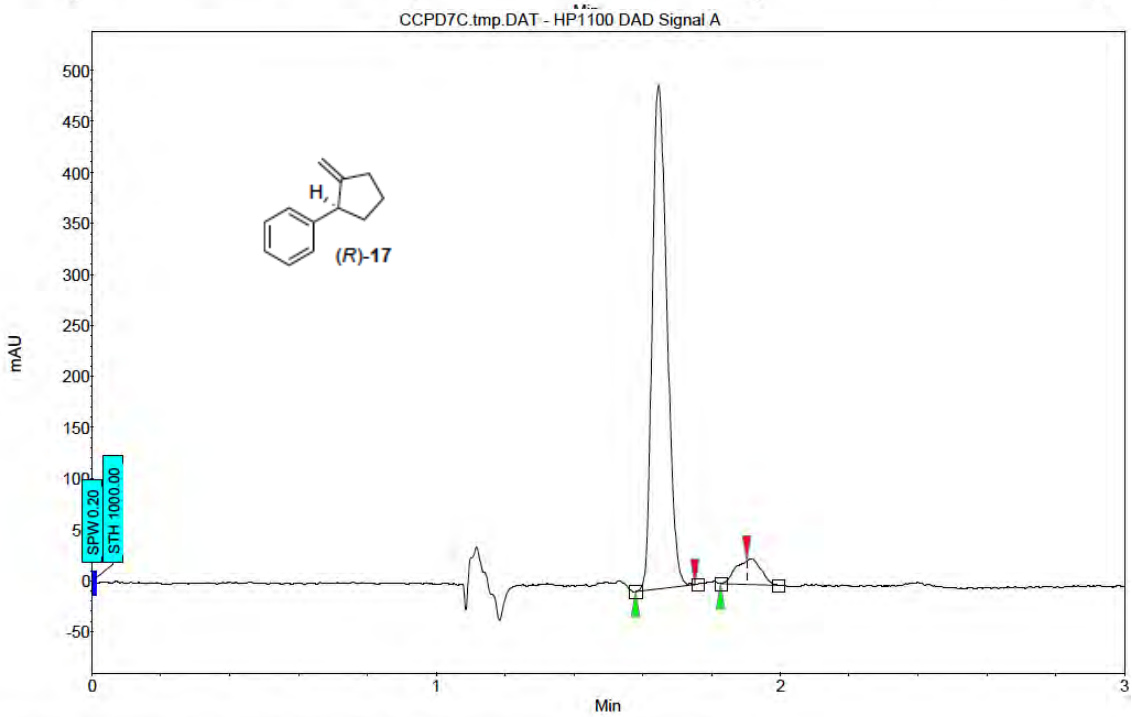




Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	3.58	3.74	3.96	0.00	5.76	52.5	5.6	5.756
2	UNKNOWN	4.04	4.24	4.55	0.00	94.24	731.6	91.8	94.244
Total						100.00	784.1	97.5	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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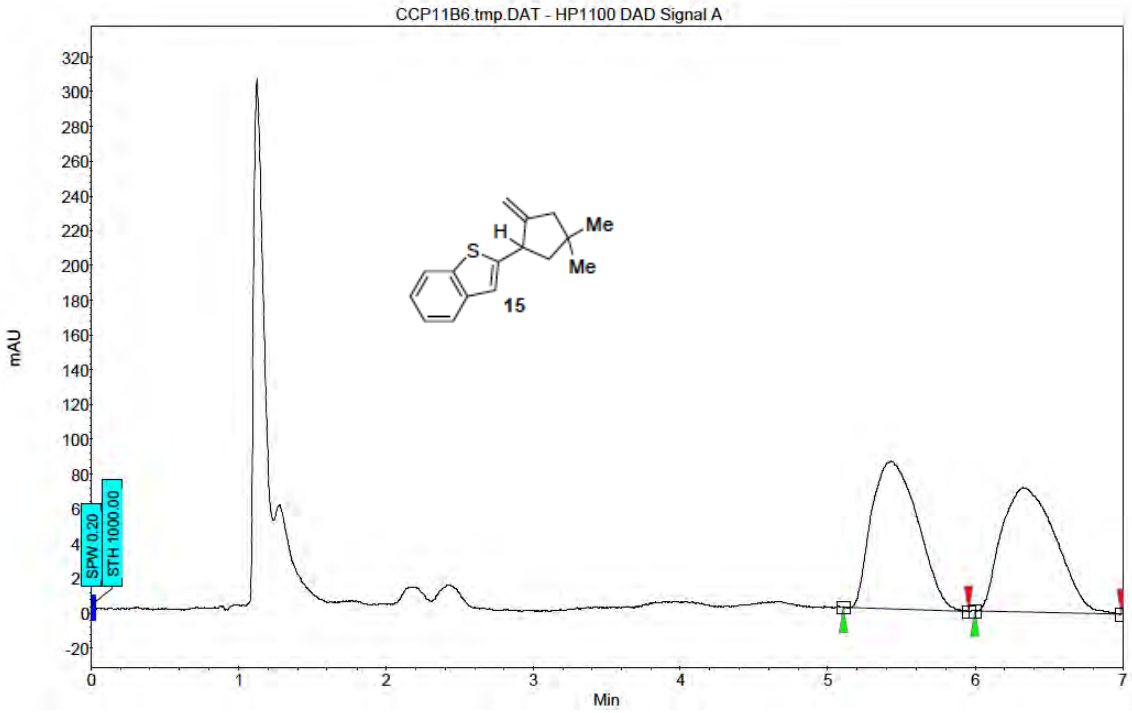
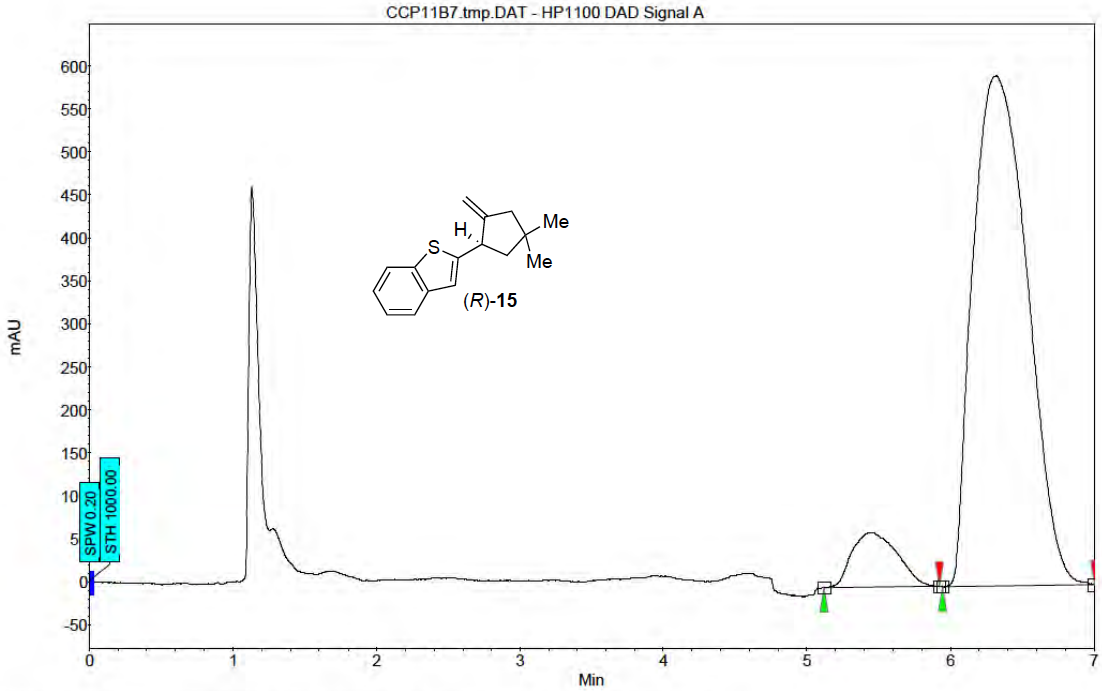
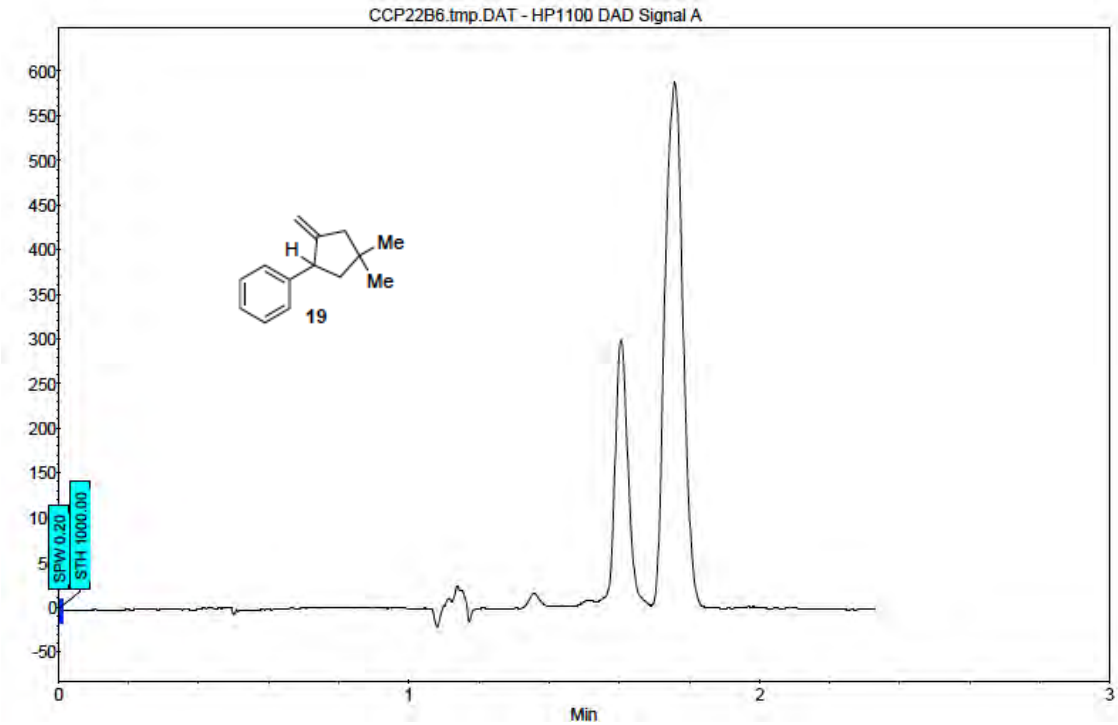
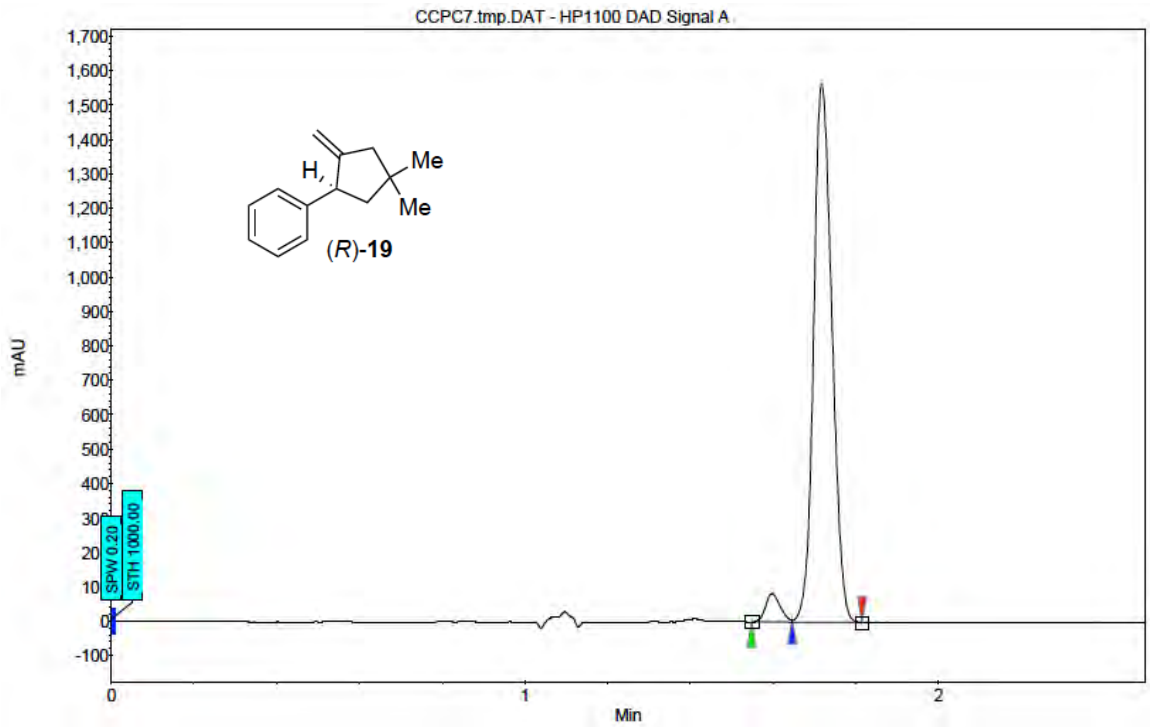
1	UNKNOWN	1.58	1.65	1.75	0.00	96.49	493.5	24.4	96.493
2	UNKNOWN	1.83	1.90	1.90	0.00	3.51	22.7	0.9	3.507
Total						100.00	516.1	25.3	100.000



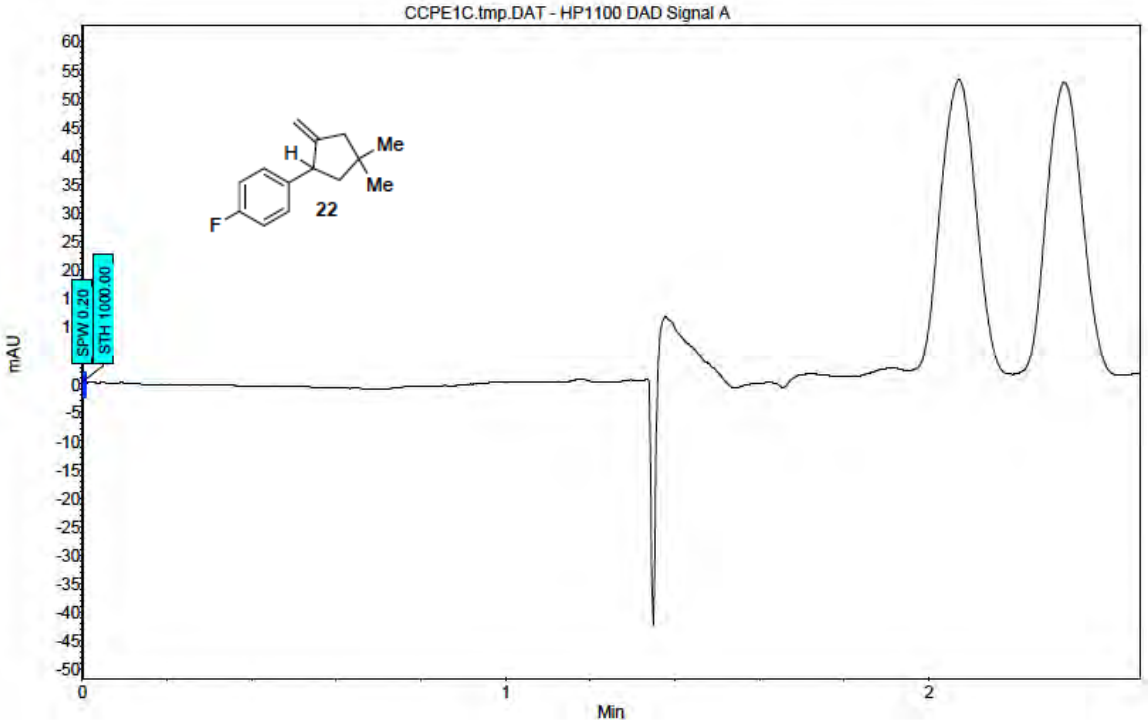


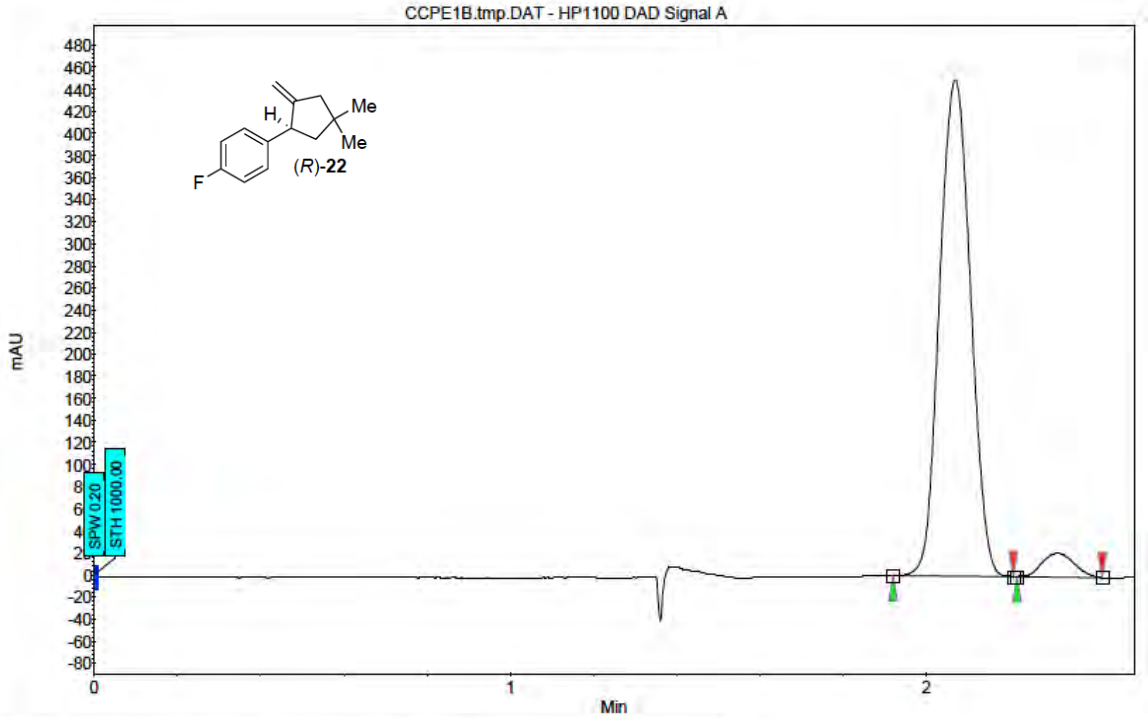
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.12	5.45	5.92	0.00	8.18	63.7	23.2	8.181
2	UNKNOWN	5.94	6.32	7.00	0.00	91.82	593.0	260.4	91.819
Total						100.00	656.7	283.6	100.000



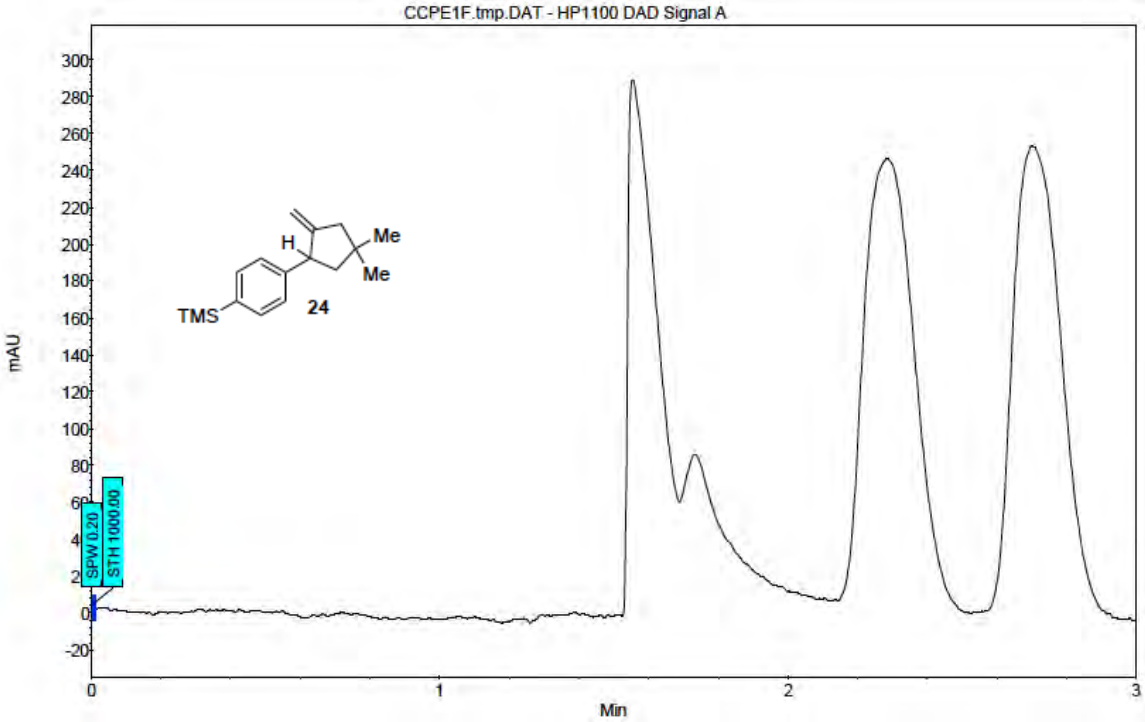


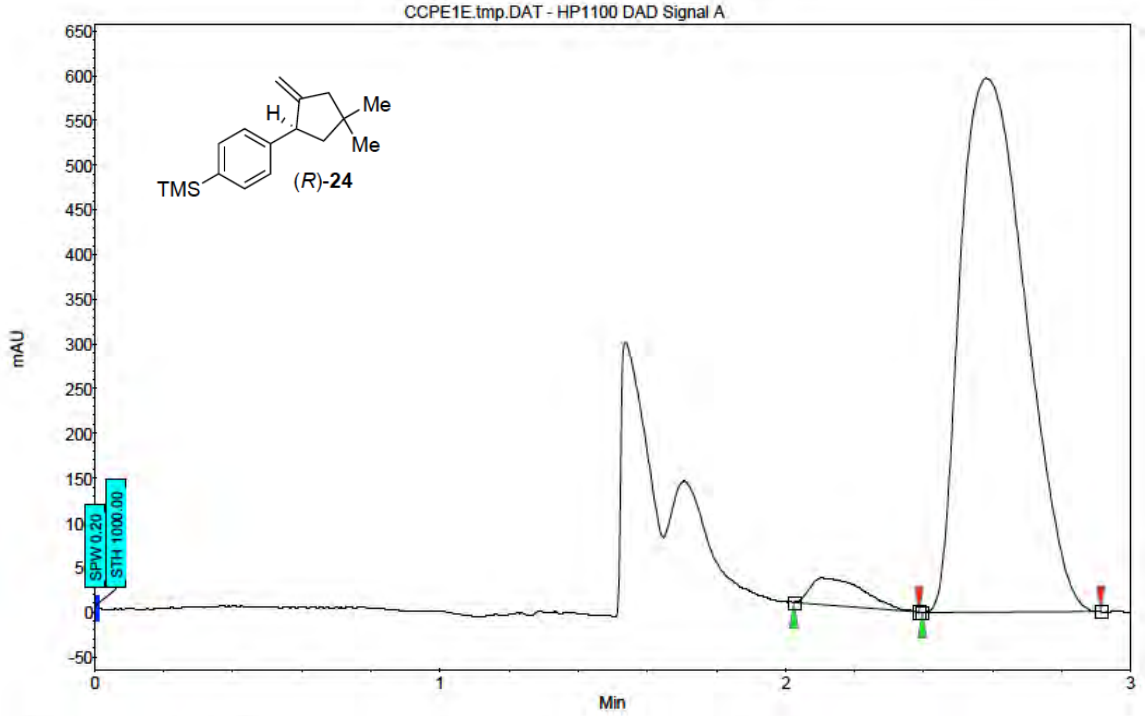
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.55	1.60	1.65	0.00	4.05	82.9	3.4	4.054
2	UNKNOWN	1.65	1.72	1.82	0.00	95.95	1563.6	80.0	95.946
Total						100.00	1646.4	83.4	100.000



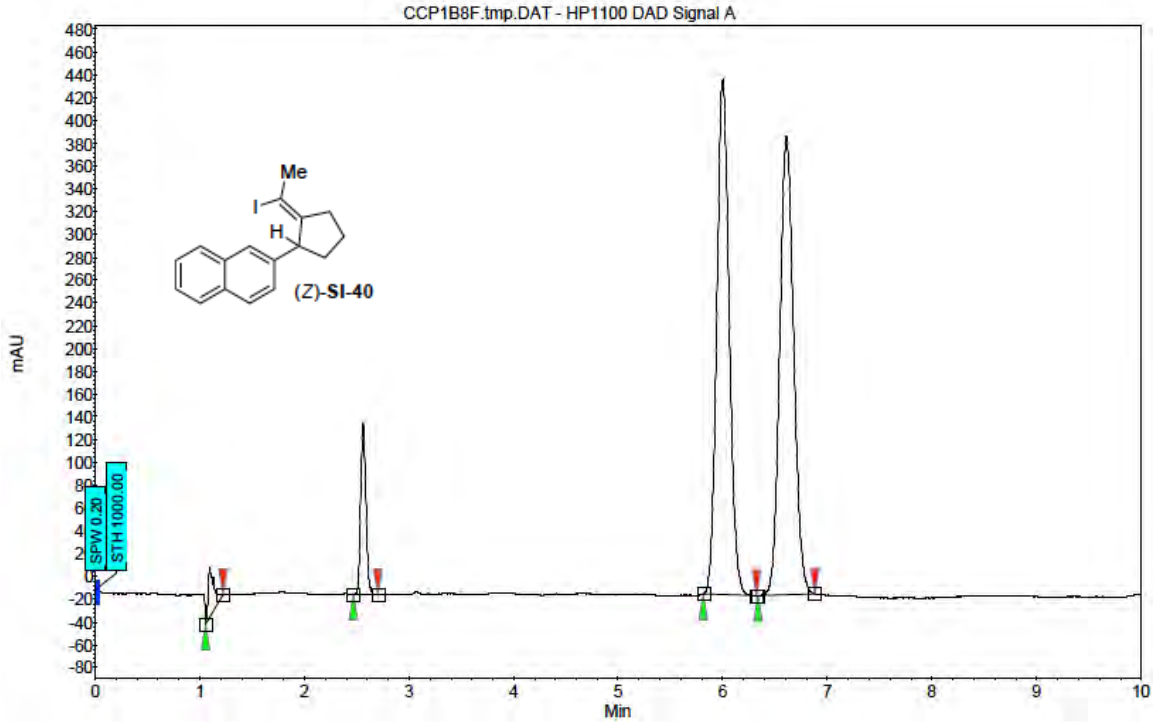


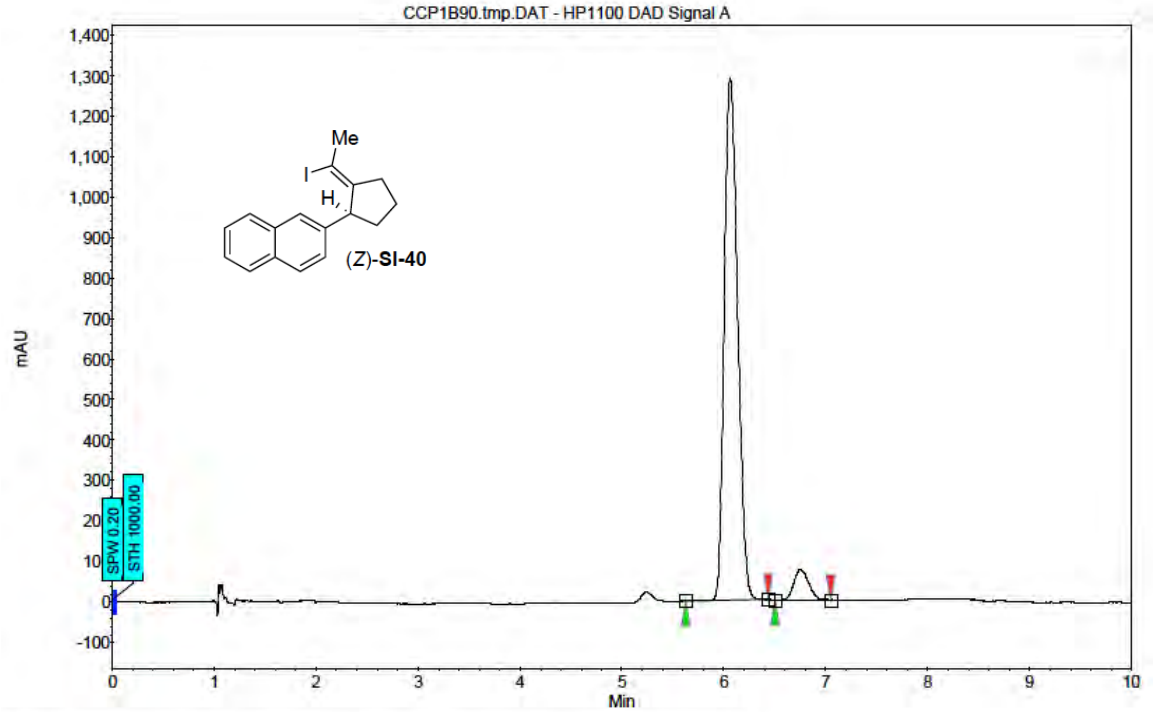
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	1.92	2.07	2.21	0.00	95.21	449.5	38.6	95.213
2	UNKNOWN	2.22	2.31	2.42	0.00	4.79	21.7	1.9	4.787
Total						100.00	471.2	40.6	100.000



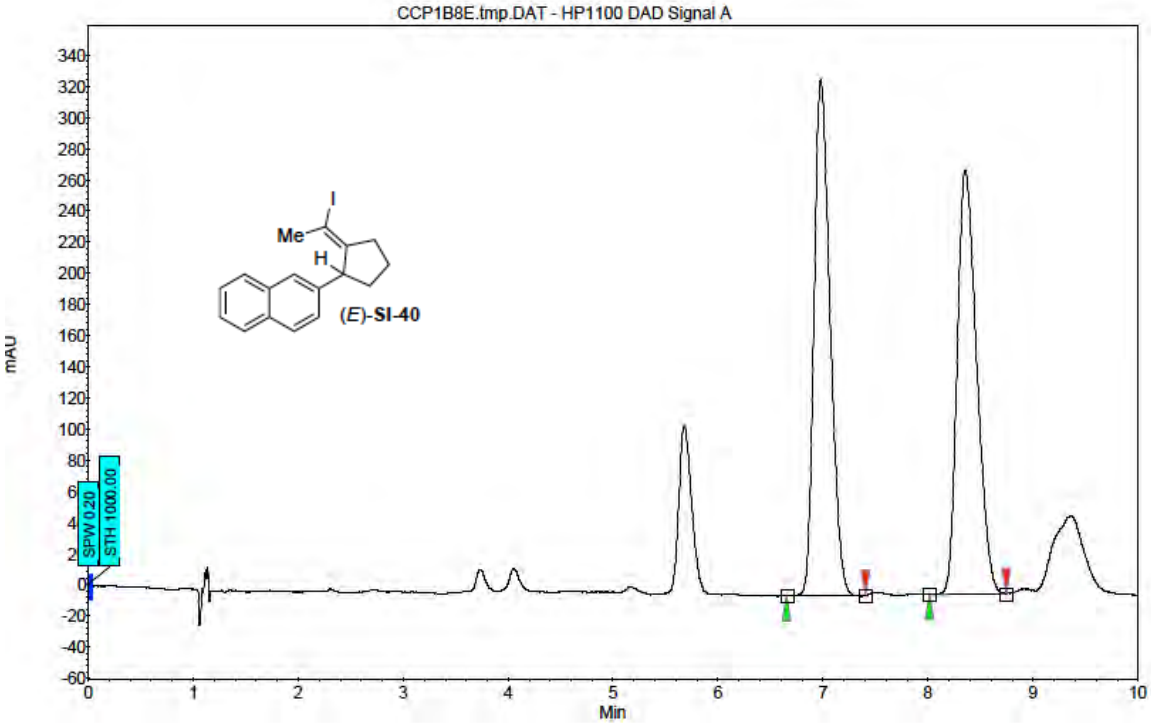


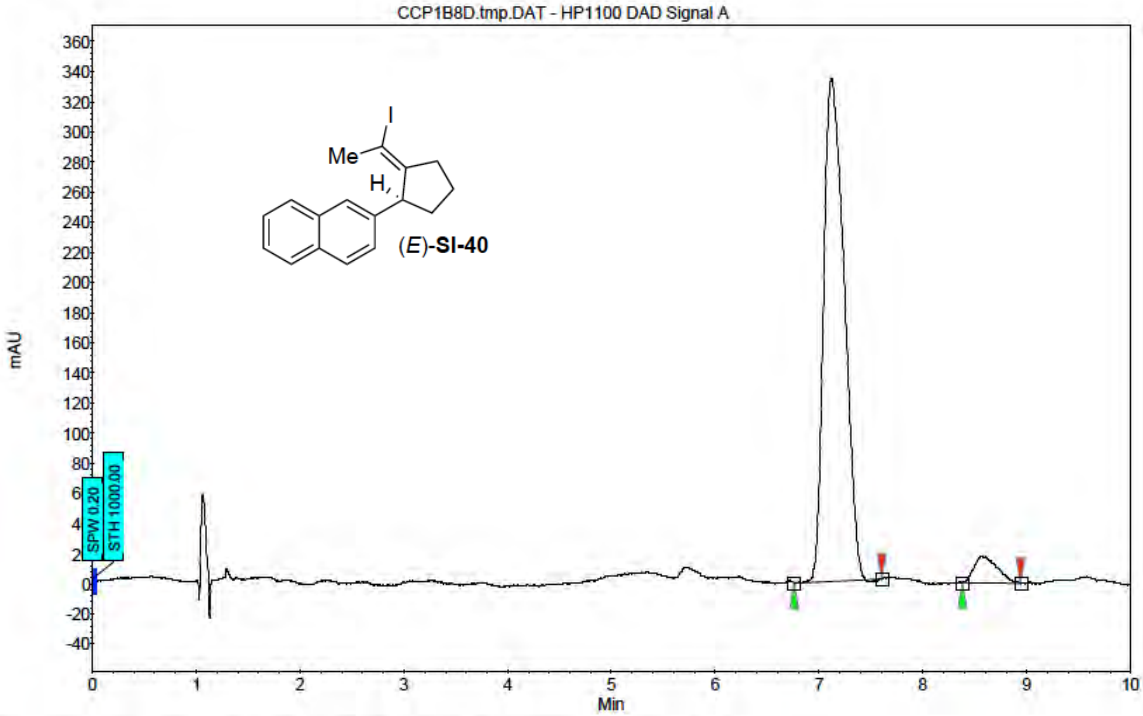
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	2.03	2.11	2.39	0.00	4.02	30.1	5.5	4.015
2	UNKNOWN	2.40	2.58	2.91	0.00	95.98	597.4	130.8	95.985
Total						100.00	627.5	136.2	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.63	6.06	6.44	0.00	94.06	1287.8	195.2	94.062
2	UNKNOWN	6.50	6.75	7.05	0.00	5.94	74.4	12.3	5.938
Total						100.00	1362.2	207.5	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
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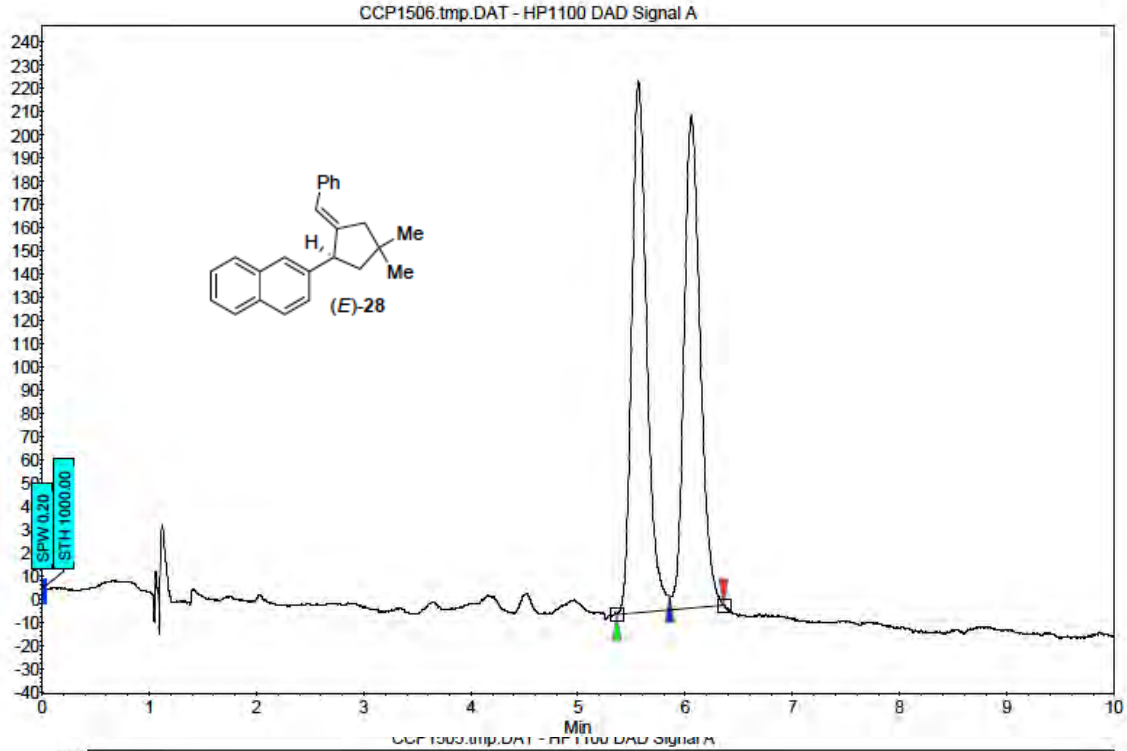
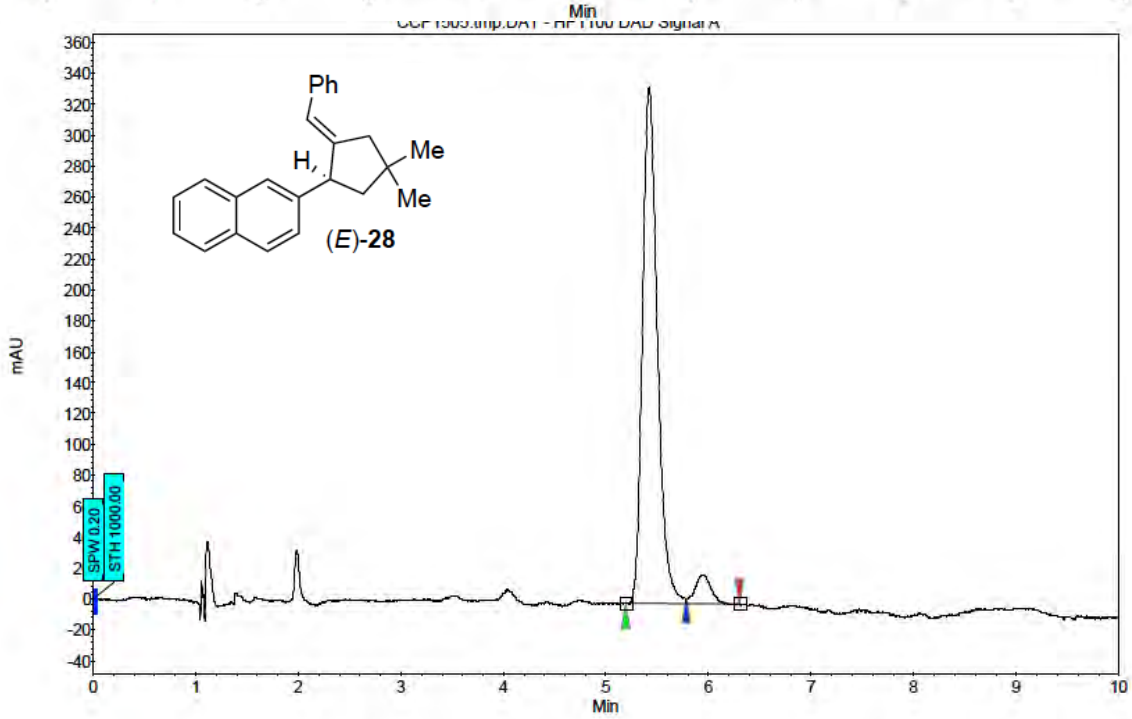
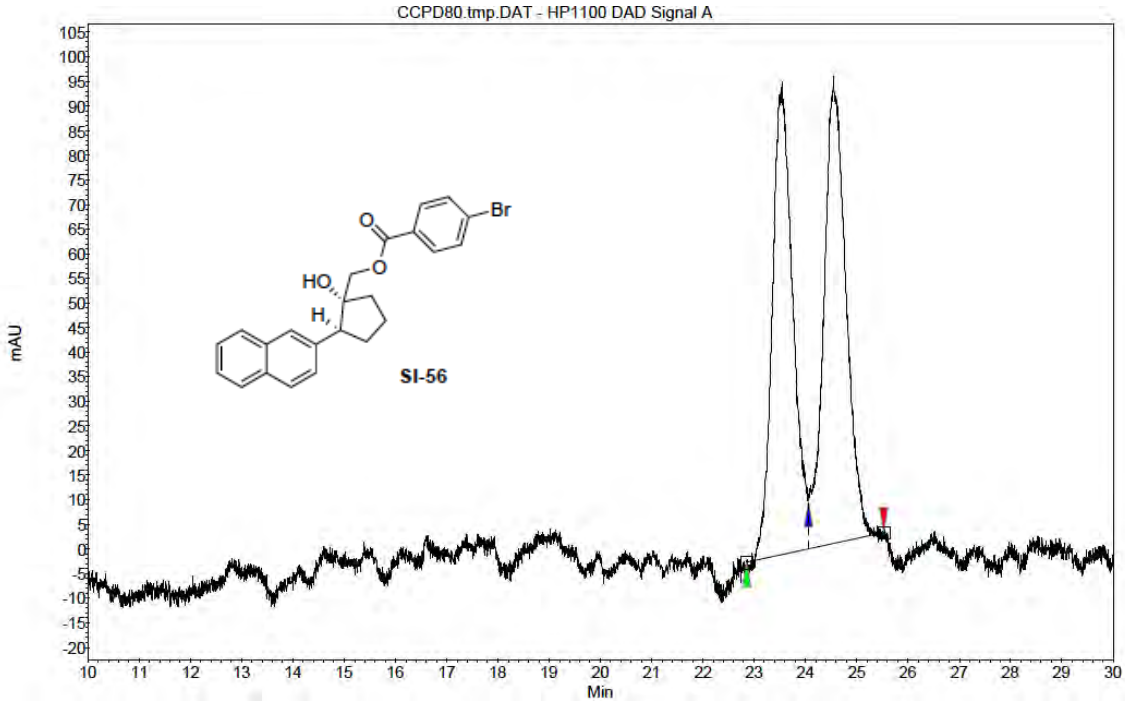
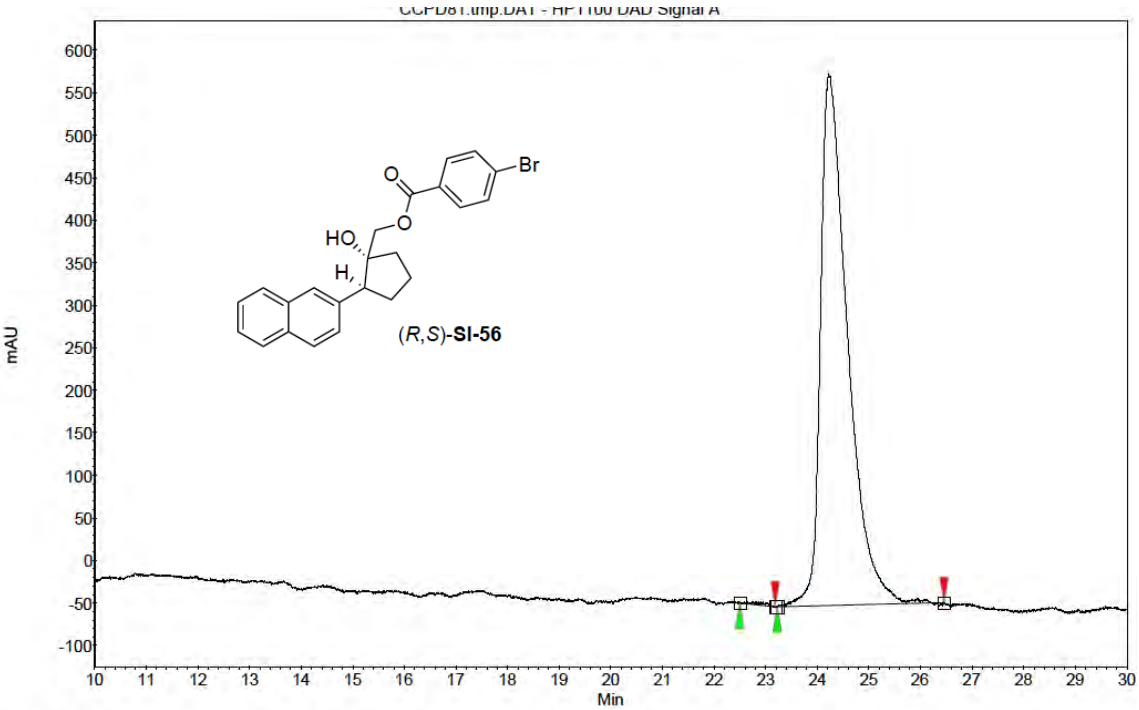
1	UNKNOWN	6.76	7.12	7.60	0.00	94.39	333.3	76.4	94.390
2	UNKNOWN	8.38	8.56	8.94	0.00	5.61	17.5	4.5	5.610
Total						100.00	350.8	81.0	100.000



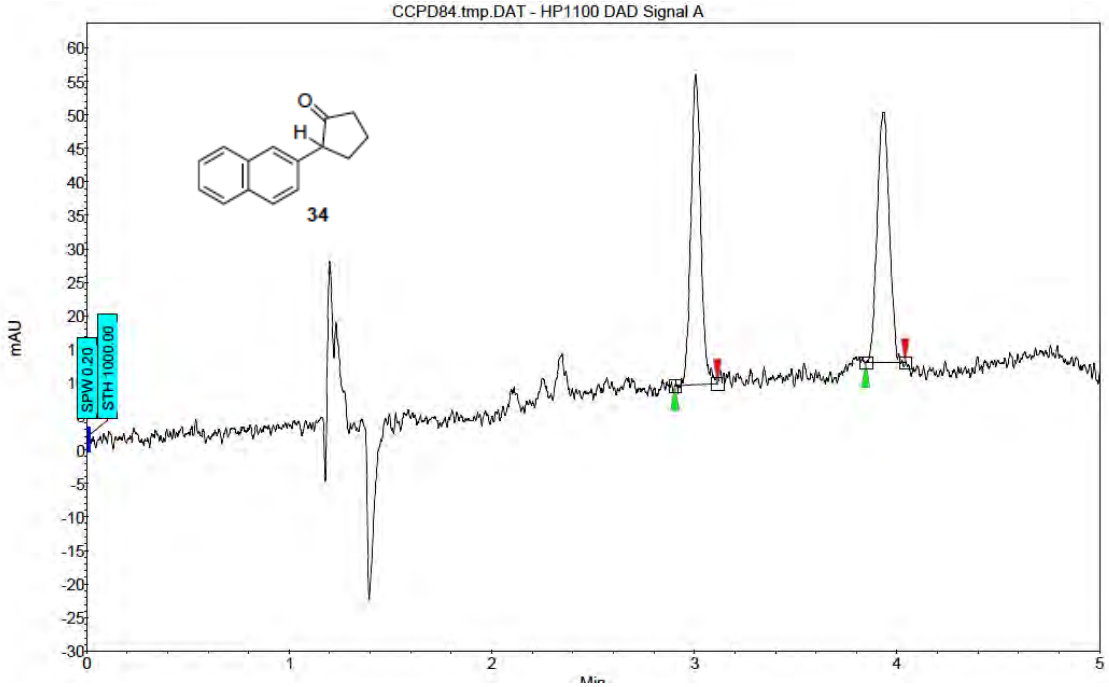


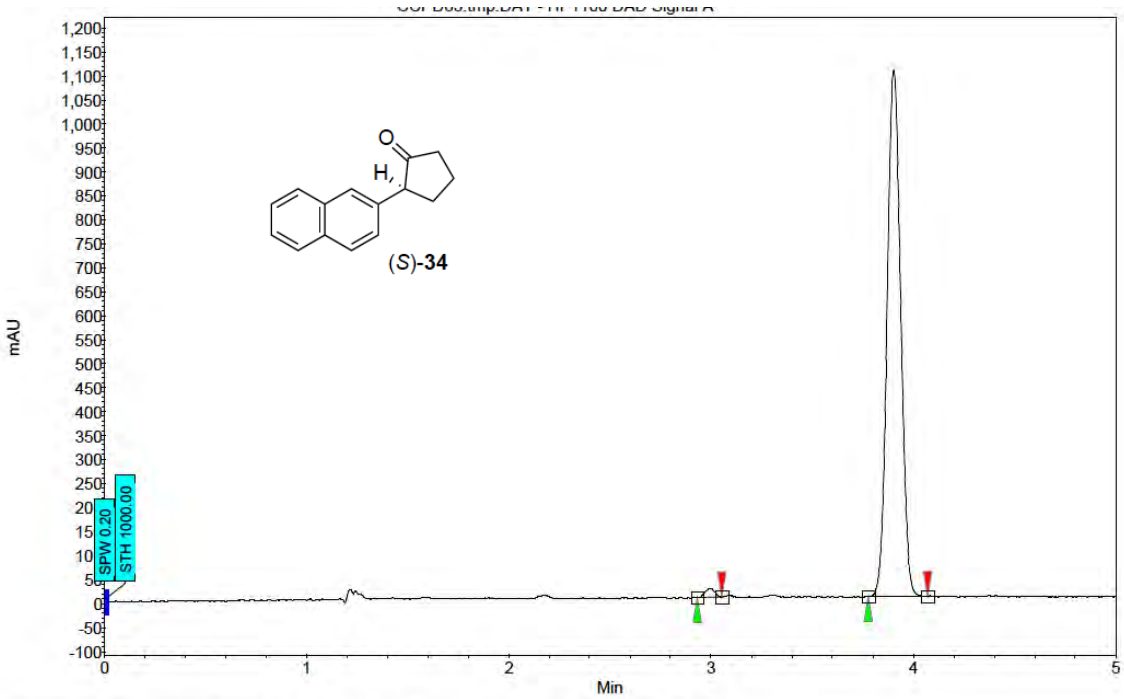
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	5.19	5.42	5.79	0.00	94.50	333.5	56.0	94.498
2	UNKNOWN	5.79	5.96	6.30	0.00	5.50	18.9	3.3	5.502
Total						100.00	352.5	59.3	100.000



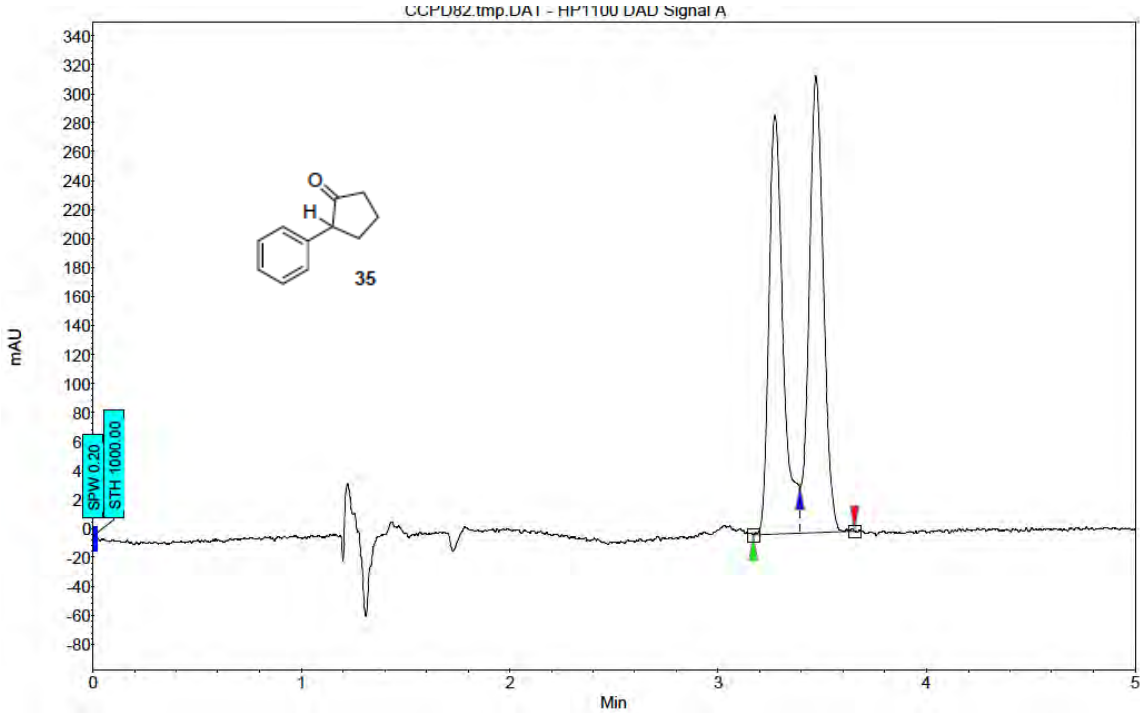


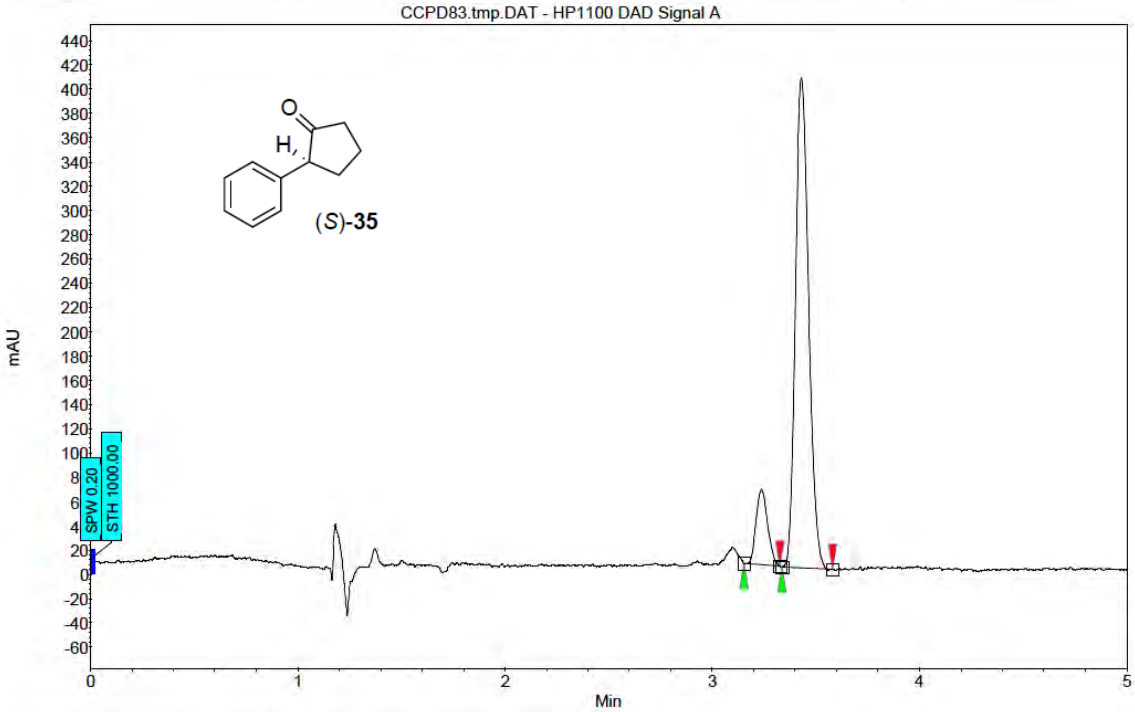
Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
2	UNKNOWN	22.51	22.96	23.18	0.00	0.28	4.0	1.1	0.277
1	UNKNOWN	23.23	24.23	26.46	0.00	99.72	624.2	380.6	99.723
Total						100.00	628.2	381.7	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
2	UNKNOWN	2.93	2.99	3.05	0.00	1.14	18.0	1.0	1.141
1	UNKNOWN	3.78	3.91	4.07	0.00	98.86	1096.7	85.6	98.859
Total						100.00	1114.8	86.5	100.000





Index	Name	Start	Time	End	RT Offset	Quantity	Height	Area	Area
		[Min]	[Min]	[Min]	[Min]	[% Area]	[µV]	[µV.Min]	[%]
1	UNKNOWN	3.15	3.24	3.33	0.00	11.78	61.5	4.0	11.780
2	UNKNOWN	3.34	3.43	3.58	0.00	88.22	403.6	29.7	88.220
Total						100.00	465.2	33.7	100.000