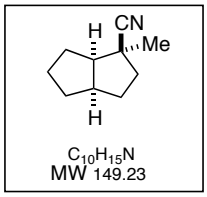
Table of Contents

Chemical Materials and Methods.

Unless stated otherwise, reactions were conducted in oven-dried glassware under an atmosphere of nitrogen or argon using anhydrous solvents (either freshly distilled or passed through activated alumina columns). HMPA and TMSCl were purified by distillation over CaH_2. TBSCl and methyl vinyl ketone were purified by distillation from neat solutions. All other commercially obtained reagents were used as received. Reaction temperatures were controlled using an IKAmag temperature modulator. Thin-layer chromatography (TLC) was conducted with E. Merck silica gel 60 F254 pre-coated plates, (0.25 mm) and visualized by exposure to UV light (254 nm) or stained with anisaldehyde, ceric ammonium molybdate, potassium permanganate and iodine. Flash column chromatography was performed using normal phase silica gel (60 Å, 230-240 mesh, Merck KGA) with a Biotage Isolera One chromatography system. ^{1}H NMR spectra were recorded on Bruker spectrometers (at 500 or 600 MHz) and are reported relative to deuterated solvent signals. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. ^{13}C NMR spectra were recorded on Bruker Spectrometers (at 125 or 150 MHz). Data for ^{13}C NMR spectra are reported in terms of chemical shift. IR spectra were recorded on a Varian 640-IR spectrometer and are reported in terms of frequency of absorption (cm^{-1}). Blue LEDs (30 cm, 1 watt) were purchased from http://www.creativelightings.com and powered by 8 AA batteries. Optical rotations were measured with a Jasco P-1010 polarimeter. High resolution mass spectra were obtained from the UC Irvine Mass Spectrometry Facility with a Micromass LCT spectrometer. See *JOC Standard Abbreviations and Acronyms* for abbreviations (available at http://pubs.acs.org/userimages/ContentEditor/1218717864819/joceah_abbreviations.pdf).

Synthetic Procedures



***rac*-1-methyl-*cis*-octahydropentalene-1-*endo*-carbonitrile (S2):** The nitrile was prepared according to the procedure of van Leusen.[1] A solution of *cis*-bicyclo[3.3.0]-octa-2-one[2] (3.50 g, 27.7 mmol) in dimethoxyethane (140 mL) and EtOH (4.0 mL) was cooled to 0 °C. To the stirred solution was added toluenesulfonylmethyl isocyanide (5.94 g, 30.5 mmol) and potassium *tert*-butoxide (6.82 g, 60.9 mmol). The resulting mixture was stirred and allowed to warm to RT. After 18 h, 150 mL of hexanes was added and the resulting mixture was filtered through Celite and the filtrate was concentrated. The residue was purified by column chromatography (100 g of SiO_2, 0–5% ethyl acetate/ hexanes) to provide *cis*-octahydropentalene-1-carbonitrile[3] (**S1**) as a pale yellow oil (1.9 g, 51%) and ~0.9:1 mixture of unassigned diastereomers: ^{1}H NMR ($CDCl_3$, 500 MHz, for both isomers) δ 2.53 (m), 2.47 (m), 2.26 (m), 1.80 (m), 1.40 (m), 1.2 (m); ^{13}C NMR ($CDCl_3$, 125 MHz, for both isomers) δ 123.2, 121.8, 49.3, 45.0, 43.0, 42.9, 35.6, 34.6, 33.8, 33.5, 33.0, 32.8, 32.4, 31.9, 31.4, 30.3, 27.2, 25.6; IR (thin film) 2865, 2235, 1451, 1319 cm^{-1}; HRMS (GC/TOF) calculated for $C_9H_{13}N$ (M+Na) 158.0946, observed 158.0947. The mixture of nitriles (1.20 g, 8.95 mmol) was dissolved in THF (25 mL) and cooled to −78 °C. A solution of 0.5 M LDA (24 mL, 12 mmol) in THF was added slowly over 5 min. After 15 min, neat MeI (0.72 mL, 12 mmol) was added dropwise to the yellow solution. After 30 min, saturated aqueous NH_4Cl (100 mL) and Et_2O (100 mL) was added to the solution. The layers were separated and the aqueous layer was extracted with Et_2O (2 x 100 ml). The combined organic layers were dried (Na_2SO_4) and concentrated to yield **S2** (1.20 g, ~95%) as a clear oil and a ~5:1 mixture of diastereomers. The indicated stereochemistry is of the major isomer and was determined by NOE as shown on page 13: ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.61 (m, 2H, minor isomer), 2.55 (m, 1H, major isomer), 2.23

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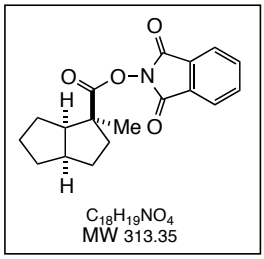
[1] O. H. Oldenziel, D. van Leusen, A. M. van Leusen, *J. Org. Chem.* **1977**, *42*, 3114.

[2] Prepared in two steps. 2-endo-hydroxy-*cis*-bicyclo[3.3.0]-octane was prepared according to the procedure of Whitesell (J. K. Whitesell, P. D. White *Synthesis*, **1975**, 602) from cyclooctene oxide. Oxidation of the secondary alcohol to form *cis*-bicyclo[3.3.0]-octa-2-one is known using

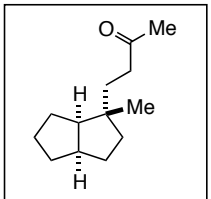
[2] Prepared in two steps. 2-endo-hydroxy-*cis*-bicyclo[3.3.0]-octane was prepared according to the procedure of Whitesell (J. K. Whitesell, P. D. White *Synthesis*, **1975**, 602) from cyclooctene oxide. Oxidation of the secondary alcohol to form *cis*-bicyclo[3.3.0]-octa-2-one is known using CrO_3 (J. K. Whitesell, M. A. Minton, S. W. Felman, *J. Org. Chem.* **1983**, *48*, 2193.). The Swern protocol was employed in these studies with similar results.

[3] This compound was prepared previously through hydrogenation of 2,3,3a,4,5,6-hexahydropentalene-1-carbonitrile (see, A. C. Cope, M. Brown, *J. Am. Chem. Soc.* **1958**, *80*, 2859).

(m, 1H, major isomer), 2.06 (m), 1.78 (m), 1.69 (m), 1.50 (m), 1.42 (s, 3H, minor isomer), 1.41 (s, 3H, major isomer), 1.34 (m); ^{13}C NMR ($CDCl_3$, 125 MHz, for major isomer) δ 124.8, 53.9, 43.1, 38.9 (2C), 34.1, 31.4, 31.3, 26.8, 25.4; IR (thin film) 2951, 2231, 1453, 1380 cm^{-1}; HRMS (GC/TOF) calculated for $C_{10}H_{15}N$ (M+Na) 172.1102, observed 172.111.

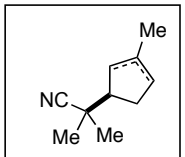


***rac*-1,3-dioxoisoindolin-2-yl 1-methyl-*cis*-octahydropentalene-1-*endo*-carboxylate (15):** A solution of KOH (3.0 g) in ethylene glycol (30 mL) was prepared by degassing with argon for 5 minutes and heating to 100 °C. The solution was added to a vial containing **S2** (1.30 g, 5.99 mmol) and a stir bar. The vial was sealed and heated to 160 °C for 18 h. The viscous solution was allowed to cool and added to a mixture of 1N HCl (300 mL) and CH_2Cl_2 (150 mL) and the layers were separated. The aqueous layer was extracted with CH_2Cl_2 (3 x 150 mL). The combined organic layers were dried (Na_2SO_4) and concentrated to provide the crude acid (**S3)** that was used without further purification: ^{1}H NMR ($CDCl_3$, 500 MHz, for major isomer) δ 2.53 (m, 1H), 2.24 (m, 1H), 2.04 (m, 1H), 1.78 (m, 2H), 1.44 (m, 1H), 1.33 (m, 1H), 125 (m, 2H) 1.22 (s, 3H), 1.11 (m, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz, for major isomer) δ 183.4, 53.9, 41.8, 35.3, 32.6, 31.3, 30.6, 27.5, 24.7; IR (thin film) 2951,1696, 1468, 1288 cm^{-1}; HRMS (ESI/TOF) calculated for $C_{10}H_{15}O_2$ (M-H) 167.1072, observed 167.1065. The acid **S3** from above was dissolved in THF (60 mL) and *N*-hydroxy-phthalimide (1.45 g, 8.90 mmol), *N*,*N*'-dicyclohexylcarbodiimide (1.83 g, 8.90 mmol), and 4-dimethylaminopyridine (70 mg, 0.29 mmol) were added sequentially. After 18 h, saturated aqueous NH_4Cl (100 mL) and ethyl ether (100 mL) were added to the resulting solution. The layers were separated and the aqueous layer was extracted with Et_2O (2 x 100 ml). The combined organic layers were dried (Na_2SO_4) and concentrated. The residue was purified by column chromatography (SiO_2, 1–10% ethyl acetate/hexanes) to yield **15** (1.35 g, 72%) as a white solid and a 7.8:1 mixture of diastereomers: m.p. 83-85 °C; R_f 0.28 (10% ethyl acetate/hexanes), ^{1}H NMR ($CDCl_3$, 500 MHz, for major isomer) δ 7.86 (m, 2H), 7.77 (m, 2H), 2.64 (m, 1H), 2.44 (m, 1H), 2.16 (m, 1H), 2.08 (m, 1H), 1.94 (m, 2H), 1.66 (m, 2H), 1.47 (s, 3H), 1.38 (m, 2H), 1.24 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz, for major isomer) δ 173.2, 162.3, 134.8, 129.2, 124.0, 54.0, 42.0, 35.4, 35.0, 33.0, 31.6, 30.3, 27.6, 24.7; IR (thin film) 2950, 1783, 1745, 1467, 1369 cm^{-1}; HRMS (ESI/TOF) calculated for $C_{18}H_{19}NO_4$ (M+Na) 336.1212, observed 336.1211.



$C_{13}H_{22}O$
MW 194.31

***rac*-4-(1-*endo*-methyl-*cis*-octahydropentalen-1-yl)butan-2-one (18):** To a solution of **18** (63 mg, 0.20 mmol) in 2:1 THF:H_2O (1.3 mL) in a 1 dram vial was added 1,4-dihydrobenzyl-nicotinamide (85 mg, 0.30 mmol),[4] methyl vinyl ketone (25 μL, 0.30 mmol), and $Ru(bpy)_3Cl_2$ (1.5 mg, 0.0020 mmol). The red solution was irradiated with blue LEDs (placed in the center of a 30 cm loop of blue LEDs) for 90 min. Hexanes was added to the mixture and the mixture was loaded onto a silica column. Purification by column chromatography (SiO_2, 1–10% ethyl acetate/hexanes) yielded **18** (27 mg, 70%) as a clear oil and as a single diastereomer:[5] R_f 0.39 (10% ethyl acetate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.45 (m, 1H), 2.34 (m, 2H), 2.12 (s, 3H), 1.92 (apt. q, J = 8.6 Hz, 1H), 1.82 (m, 2H), 1.58 (m, 1H), 1.47 (m, 3H), 1.30 (m, 3H), 1.18 (m, 2H), 1.09 (m, 1H), 0.84 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 210.1, 53.6, 43.6, 42.8, 39.9, 37.8, 35.3, 35.1, 31.5, 30.1, 29.4, 28.1, 21.4; IR (thin film) 2944, 2863, 1718, 1764, 1356 cm^{-1}; HRMS (GC/TOF) calculated for $C_{13}H_{22}O$ (M+NH_4) 212.2014, observed 212.2009.



2:1 $\Delta^{1,2}$:$\Delta^{1,5}$
$C_{10}H_{15}N$
MW 149.23

(*R*)-2-methyl-2-(3-methylcyclopent-2-enyl)propanenitrile and (*R*)-2-methyl-2-(3-methylcyclopent-3-enyl)propanenitrile (20): The original procedure reported by Kreiser has been slightly modified.[6] To a 2-L 1-necked round bottom with a large stir bar was added sequentially EtOH (1.2 L), (+)-fenchone (100 g, 0.65 mol), NH_2OH-HCl (78 g, 1.1 mol), and NaOAc (106 gr, 1.30 mol). The flask was equipped with a reflux condenser and the stirred mixture was heated at reflux for 36 h. The reaction was cooled to RT and concentrated on a rotary evaporator to approximately 400 mL. H_2O (1 L) was added to the reaction mixture and the oxime was isolated as a white solid by filtration. The white solid was added to a 2L round bottom with a large stir bar and 4 M H_2SO_4 (1 L) was added. The flask was equipped with a reflux condenser and the stirred mixture was heated at reflux for 8 h and cooled to RT. Pentane (300 mL) was added to the reaction mixture and the organic layer was separated, dried over Na_2SO_4, and concentrated. Distillation (110 °C, 10 tor) afforded the nitrile **20** as 2.0:1 mixture of olefin isomers (81.0 g, 83%). The spectral data obtained matched that obtained by Kreiser. Higher field

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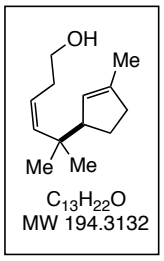
[4] Prepared according to the procedure of Zhu from nicotinamide (X. Q. Zhu, J. Y. Zhang, J. P. Cheng, *J. Org. Chem.* **2006**, *71*, 7007).

[5] See the supporting information of the preceding communication in this issue for stereochemical assignment.

[6] W. Kreiser, P. Below *Liebigs Ann. Chem.* ***1985***, 203.

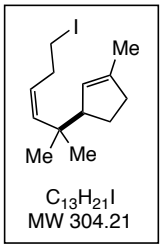
NMR data is provided for reference: ^{1}H NMR ($CDCl_3$, 600 MHz) δ 5.26 (s, 1H, major isomer), 5.23 (s, 1H, minor isomer), 2.73 (m, 1H, major isomer), 2.40 (m, 1H, minor isomer), 2.24 (m), 2.08 (m), 1.74 (m), 1.66 (s, 3H, minor isomer), 1.29 (s, 3H, minor isomer), 1.28 (s, 3H, minor isomer), 1.26 (s, 3H, major isomer), 1.24 (s, 3H, major isomer); ^{13}C NMR ($CDCl_3$, 155 MHz, major and minor) δ 144.8, 139.3, 125.1, 124.4, 123.2, 123.0, 55.0, 47.8, 38.7, 36.6, 36.55, 36.5, 35.5, 28.6, 25.6, 25.4, 24.6, 24.55, 16.8, 16.7.



(*R*,*Z*)-5-methyl-5-(3-methylcyclopent-2-enyl)hex-3-en-1-ol (21): A 3L flask was charged with the nitrile **20** (23.5 g, 158 mmol) and CH_2Cl_2 (1 L). The flask was equipped with an addition funnel and cooled to −78 °C via a dry ice bath and DIBAL (126 mL of ~25% solution in toluene, 189 mmol) was added slowly. The dry ice/ acetone bath was removed and the solution was allowed to warm to RT. After 2 h, an ice bath was added and 2N HCl (600 mL) was slowly added. The reaction was allowed to warm to RT and stirring was maintained for 18 h. The two layers were separated and the organic layer was dried over Na_2SO_4 and concentrated to afford the crude aldehyde, which was used without further purification. 3-Hydroxypropyltriphenylphosphonium bromide[7] (126 g, 316 mmol) was added to a 5-L 3-necked flask, THF (1.6 L) was added, and the flask was equipped with an addition funnel, an overhead mechanical stirrer, and an internal temperature probe. The mixture was cooled to 0 °C and *n*BuLi (250 mL of a 2.5 M in hexanes solution, 630 mmol) was added slowly while maintaining the temperature below 10 °C as a dark solution was formed. After 15 min, TMSCl (39.0 mL, 316 mmol) in THF (100 mL) was added via addition funnel. After 20 min, the reaction was cooled to −78 °C and the aldehyde from above was added in THF (50 mL) over 1 h while the temperature was maintained below −60 °C. After the addition, the cooling bath was removed and the reaction was allowed to warm to 0 °C. After 10 min, 1.0 L of 2 M H_2SO_4 was added and vigorous stirring was maintained. After 18 h at RT, the layers were separated and the aqueous layer was washed with Et_2O (2 × 200 mL). The combined organic layers were concentrated and passed through a silica plug (~300 g SiO_2, 20% ethyl acetate/hexanes). The material was further purified to separate the olefin isomers by column chromatography (600 g of 6% $AgNO_3$ embedded SiO_2, 10% acetone/hexanes) to obtain the more polar desired alcohol **21** as a light yellow oil (16.8 g,

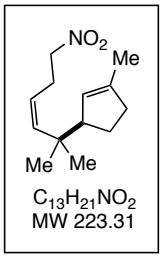
[7] Accessed by refluxing 3-bromo-1-propanol and triphenylphosphine in toluene and filtering the resulting solid. See, for example, R. Wang, S. C. Lu, Y. M. Zhang, Z. J. Shi, W. Zhang, *Org. Biomol. Chem.* **2011**, *9*, 5802.

55% overall (82% if based on the 66% of desired olefin isomer in starting material)): R_f 0.35 (20% ethyl acetate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 5.42 (d, J = 12.3 Hz, 1H), 5.21 (m, 2H), 3.62 (t, J = 6.5 Hz, 2H), 2.46 (q, J = 7.1 Hz, 2H), 2.30 (m, 2H), 2.11 (m, 3H), 1.70 (bs, 1H), 1.64 (s, 3H), 1.06 (s, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 140.4, 139.8, 125.1, 123.8, 63.0, 50.1, 39.00, 38.95, 34.8, 32.4, 26.9, 26.8, 16.9; IR (thin film) 3343, 2949, 1378, 1049 cm^{-1}; HRMS (GC/TOF) calculated for $C_{13}H_{22}O$ (M+NH_4) 212.2014, observed 212.2018; $[\alpha]_D^{24}$ +62.2°, $[\alpha]_{577}^{24}$ +62.8°, $[\alpha]_{546}^{24}$+71.64, $[\alpha]_{435}^{24}$ +122.4°, $[\alpha]_{405}^{24}$+144.6°, (c = 1.0, $CHCl_3$).



$C_{13}H_{21}I$
MW 304.21

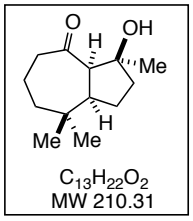
(*R*,*Z*)-1-methyl-3-(2-methyl-6-nitrohex-3-en-2-yl)cyclopent-1-ene (S4): The alcohol **21** (10.2 g, 52.6 mmol) was dissolved in benzene (260 mL) and cooled in an ice bath. To the solution was added sequentially imidazole (3.93 g, 57.8 mmol), PPh_3 (14.4 g, 55.2 mmol), and I_2 (13.9 g, 55.2 mmol) and the ice bath was removed and the mixture was stirred for 18 h at RT. $NaHSO_3$ (1 g) and saturated aqueous NH_4Cl (500 mL) was added to the mixture. The layers were separated and the aqueous layer was extracted with Et_2O (200 ml). The combined organic layers were dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography (SiO_2, 1% ethyl acetate/hexanes) to afford the desired iodide **S4** as a clear oil (15.1 g, 94%): R_f 0.75 (5% ethyl acetate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 5.38 (d, J = 12.2 Hz, 1H), 5.23 (bs, 1H), 5.11 (dt, J = 12.1, 7.3 Hz, 1H), 3.12 (t, J = 7.5 Hz, 2H), 2.76 (dq, J = 1.6, 7.3 Hz, 2H), 2.62 (m, 1H), 2.16 (m, 2H), 1.90 (m, 1H), 1.66 (s, 3H), 1.63 (m, 1H), 1.11 (s, 3H), 1.10 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 141.9, 140.4, 127.4, 126.0, 57.9, 39.9, 36.9, 32.8, 26.6, 26.3, 16.9, 5.9; IR (thin film) 2970, 1554, 1389, 1375, 1248 cm^{-1}; HRMS (GC/TOF) calculated for $C_{13}H_{21}I$ (M+NH_4) 322.1032, observed 322.1038; $[\alpha]_D^{24}$ +41.3°, $[\alpha]_{577}^{24}$ +43.6°, $[\alpha]_{546}^{24}$ +49.6, $[\alpha]_{435}^{24}$ +84.9°, $[\alpha]_{405}^{24}$ +99.6°, (c = 1.0, CH_2Cl_2).



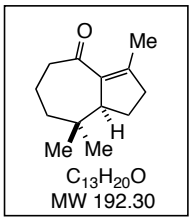
$C_{13}H_{21}NO_2$
MW 223.31

(*R*,*Z*)-1-methyl-3-(2-methyl-6-nitrohex-3-en-2-yl)cyclopent-1-ene (22): An aluminum foil covered flask was charged with iodide **S4** (20.3 g, 66.7 mmol) and a large stir bar and the flask was cooled in a ice bath. $AgNO_2$ (15.3 g, 100 mol) was added to the flask in two portions and, after 5 min, the ice bath was removed and the heterogeneous mixture was allowed to stir for 18 h. 1% Ethyl acetate/ hexanes (100 mL) was added and the mixture was applied to an equilibrated SiO_2 column. Column chromatography (2–5% ethyl acetate/hexanes) afforded compound **22** as a light yellow oil (9.07 g, 67%): R_f 0.33 (5% ethyl acetate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 5.44

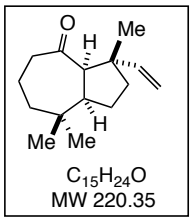
(d, J = 12.2 Hz, 1H), 5.21 (bs, 1H), 5.09 (dt, J = 12.1, 7.3 Hz, 1H), 4.36 (t, J = 7.2 Hz, 2H), 2.89 (q, J = 7.2 Hz, 2H), 2.62 (m, 1H), 2.16 (m, 2H), 1.88 (m, 1H), 1.70 (s, 3H), 1.54 (m, 1H), 1.06 (s, 3H), 1.04 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 142.6, 142.3, 125.8, 121.5, 75.7, 57.9, 40.0, 36.9, 26.9, 26.5, 26.4, 26.3, 16.9; IR (thin film) 2962, 1554, 1435, 1377 cm^{-1}; HRMS (GC/TOF) calculated for $C_{13}H_{21}NO_2$ (M+NH_4) 241.1916, observed 241.1921; $[\alpha]_D^{24}$ +48.8°, $[\alpha]_{577}^{24}$ +49.8°, $[\alpha]_{546}^{24}$ +57.1°, $[\alpha]_{435}^{24}$ +98.7°, $[\alpha]_{405}^{24}$+115.6°, (c = 1.0, CH_2Cl_2).



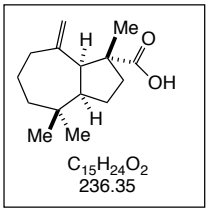
(3*S*,3a*R*,8a*R*)-3-hydroxy-3,8,8-trimethyloctahydroazulen-4(5*H*)-one (23) To a solution of **22** (6.00 g, 26.9 mmol) in toluene (260 mL) was added triethylamine (1.9 mL, 14 mmol) and phenyl isocyanate (8.8 mL, 81 mmol) and the solution was heated to 90 °C for 18 h during which time a brown heterogeneous mixture was formed. Water (5 mL) was added to the mixture and it was allowed to cool to RT and partially concentrated (~50 mL). The mixture was passed through a silica plug with 40% ethyl acetate/hexanes and concentrated to afford the intermediate isoxazoline product contaminated with byproducts derived from phenyl isocyanate. This residue was dissolved in MeOH (200 mL) and H_2O (15 mL) and 10% Pd/C (~300 mg), boric acid (4.90 g, 80.7 mmol), and Raney-Ni (~300 mg) were added. The solution was equipped with a hydrogen balloon, and the flask was evacuated and backfilled with H_2 (3×), and mixture was allowed to stir for 36 h until **23** was observed as the major product by LRMS. The mixture was filtered (Celite) and the solution was concentrated under reduced pressure to approximately 100 mL. Et_2O (300 mL) and 1N HCl (300 mL) was added and the layers were separated. The aqueous layer was extracted with Et_2O (2 x 300 mL). The combined organic layers were dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography (SiO_2, 30% ethyl acetate/hexanes) to afford **23** as a clear oil (3.80 g, 67%): R_f: 0.29 (20% ethyl acteate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 4.53 (bs, 1H), 2.62 (d, J = 10.8 Hz, 1H), 2.43 (m, 1H), 2.33 (m, 1H), 2.07 (dt, J = 4.8, 10.7 Hz, 1H), 1.95 (m, 1H), 1.74 (m, 1H), 1.62 (m, 3H), 1.50 (m, 1H), 1.30 (s, 3H), 1.32 (m, 2H), 1.00 (s, 6H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 218.6, 80.9, 58.4, 50.3, 44.7, 44.1, 40.1, 35.0, 31.3, 26.7, 25.4, 19.1, 18.2; IR (thin film) 3480, 2962, 1683, 1223 cm^{-1}; HRMS (ESI/TOF) calculated for $C_{13}H_{22}O_2$ (M+Na) 233.1517, observed 233.1519; $[\alpha]_D^{24}$ −67.9°, $[\alpha]_{577}^{24}$ −72.1°, $[\alpha]_{546}^{24}$ −84.1°, $[\alpha]_{435}^{24}$ −174.7°, $[\alpha]_{405}^{24}$ −220.1°, (c = 1.0, CH_2Cl_2).



(*S*)-3,8,8-trimethyl-1,2,6,7,8,8a-hexahydroazulen-4(5*H*)-one (24): A solution of **23** (2.80 g, 13.3 mmol) was dissolved in toluene (130 mL) and *p*-toluenesulfonic acid (0.232 g, 1.33 mmol) was added and the solution was heated to 100 °C. After 6 h, the dark solution was cooled and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, 3-4% ethyl acetate/hexanes) to afford enone **24** as a yellow oil (2.32 g, 91%): R_f: 0.44 (20% ethyl acetate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.87 (m, 1H), 2.30 (m, 4H), 1.97 (s, 3H) 1.85 (m, 1H), 1.70 (m, 1H), 1.57 (m, 4H), 0.87 (s, 3H), 0.63 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 203.5, 155.2, 138.6, 55.6, 46.3, 45.5, 38.6, 36.2, 30.5, 25.0, 21.3, 19.2, 16.9; IR (thin film) 2959, 1667, 1614, 1325 cm^{-1}; HRMS (ESI/TOF) calculated for $C_{13}H_{20}O$ (M+Na) 215.1412, observed 215.1412; $[\alpha]_D^{24}$ +80.9°, $[\alpha]_{577}^{24}$ +84.7,° $[\alpha]_{546}^{24}$ +95.6°, $[\alpha]_{435}^{24}$ +150.6° (*c* = 1.0, CH_2Cl_2).



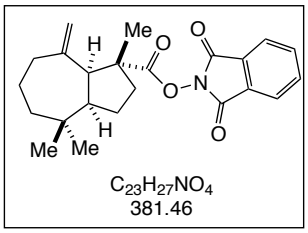
(3*S*,3a*R*,8a*R*)-3,8,8-trimethyl-3-vinyloctahydroazulen-4(5*H*)-one (25): CuCN (229 mg, 2.58 mmol) was added to a 2-necked round bottom flask with an internal temperature probe and cooled to 0 °C and THF (1 mL) and HMPA (2 mL) was added. The mixture was stirred and maintained below 10 °C while a solution of 0.7 M vinyl magnesium bromide (7.1 mL, 5.0 mmol) in THF was added slowly. The dark red solution was stirred for 10 min and then **24** (200 mg, 1.0 mmol) in THF (1 mL) was added slowly. The reaction was maintained at 0° C for 7 h with stirring as the reaction became dark and heterogeneous and then 1 N HCl (50 mL) and Et_2O (50 mL) was added. The layers were separated and the aqueous layer was extracted with Et_2O (2 x 50 ml). The combined organic layers were dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography (SiO_2, 0–5% ethyl acetate/hexanes) to afford **25** as a clear oil and 4.8:1 mixture of diastereomers (178 mg, 77%): R_f: 0.7 (10% Ethyl acetate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz, for major isomer) δ 5.80 (dd, *J* = 10.6, 17.6 Hz, 1H), 4.97 (d, *J* = 17.6 Hz, 1H), 4.92 (d, *J* = 10.6 Hz, 1H), 2.49 (d, *J* = 7.6 Hz, 1H), 2.35 (m, 2H), 2.13 (apt q, *J* = 9.8 Hz, 1H); 1.92 (m, 1H), 1.86 (m, 2H), 1.68 (m, 3H), 1.44 (m, 2H), 1.32 (m, 1H), 0.95 (s, 3H), 0.90 (s, 3H), 0.91 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz, for major isomer) δ 215.4, 147.8, 110.4, 65.5, 49.6, 49.5, 45.0, 37.3, 36.1, 36.0, 32.7, 26.5, 25.0, 24.3, 22.7; IR (thin film) 2955, 1690, 1453, 1365 cm^{-1}; HRMS (ESI/TOF) calculated for $C_{15}H_{24}O$ (M+Na) 243.1725, observed 243.1723; $[\alpha]_D^{24}$ +61.5° $[\alpha]_{577}^{24}$ +63.8°, $[\alpha]_{546}^{24}$ +72.5°, $[\alpha]_{435}^{24}$ 147.8°, $[\alpha]_{405}^{24}$ +189.9° (*c* = 1.0, CH_2Cl_2).



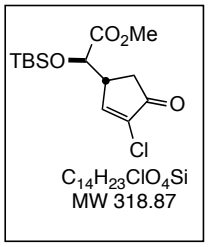
1*R*,3a*R*,8a*S*)-1,4,4-trimethyl-8-methylenedecahydroazulene-1-carboxylic acid (26):[8] A solution of 1 M (trimethylsilyl)methyllithium in pentane (3.3 mL, 3.3 mmol) was added to pentane (3.3 mL) at – 78 °C. To the cooled white mixture, **25** (150 mg, 0.67 mmol) in toluene (3 mL) was added slowly by syringe pump over 30 min. After 30 min, saturated aqueous $NaHCO_3$ (20 mL) and Et_2O (20 mL) was added. The organic layers were separated and the aqueous layer was extracted with Et_2O (2 x 50 ml). The combined organic layers were dried with Na_2SO_4 and concentrated to yield the crude β-silyl alcohol, which was used without further purification. The residue was dissolved in CH_2Cl_2 (6.7 mL) and cooled to –78 °C. Ozone was bubbled through the solution until a light blue color persisted (~3 min) and the starting material was consumed by TLC. Oxygen was bubbled through the solution until the blue color disappeared and an argon balloon was placed on the reaction. Triphenylphosphine (260 mg, 1.0 mmol) and HF-pyridine (200 μL) was added and the solution was placed in an ice bath. After 1 h at 0 °C, $NaHCO_3$ (50 mL) was slowly added to the solution and the mixture was allowed to warm to RT. After gas evolution ceased, CH_2Cl_2 (20 mL) was added. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 30 ml). The combined organic layers were dried with Na_2SO_4 and concentrated to yield the crude aldehyde product, which was used without further purification. The residue was dissolved in acetone (4.5 mL) and H_2O (150 μL) and 1 M 2-methyl-2-butene in THF (2 mL, 2 mmol), NaH_2PO_4 (119 mg, 1.0 mmol), $NaClO_2$ (180 mg, 2.0 mmol) was added. After 30 min, saturated aqueous NH_4Cl (30 mL) and CH_2Cl_2 (20 mL) were added. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 x 30 ml). The combined organic layers were dried with Na_2SO_4 and concentrated. The residue was purified by column chromatography (SiO_2, 0–15% ethyl acetate/hexanes) to afford the desired acid **26** as a waxy solid (117 mg, 74%): Rf: 0.38 (20% ethyl acetate/hexanes); ^{1}H NMR (C_6D_6, 500 MHz) δ 4.83 (d, J = 2.0 Hz, 1H), 4.75 (d, J = 2.0 Hz, 1H), 3.37 (d, J = 7.9 Hz, 1H), 2.47 (ddd, J = 5.4, 8.8, 13.8

[8] Carboxylic acid **26** could also be prepared through hydrolysis of the corresponding tertiary nitrile ((1*R*,3a*R*,8a*S*)-1,4,4-trimethyl-8-methylenedecahydroazulene-1-carbonitrile) which was prepared in the preceding communication in this issue. A solution 30% KOH in ethylene glycol (2 mL) was prepared by degassing with argon for 5 minutes and heating to 100°C. The solution was added to a vial containing the nitrile (100 mg) and a stir bar. The vial was sealed and heated to 160 °C for 18 h. The viscous solution was allowed to cool and added to a mixture of 1N HCl (100 mL) and CH_2Cl_2 (100 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 50 ml). The combined organic layers were dried with Na_2SO_4 and concentrated. The material could be used without further purification and the spectral data matched the material described here. This hydrolysis reaction also provides the stereochemical assignment of **25** and **26**, as the assignment of the tertiary nitrile starting material was secured by X-ray crystallography of a synthetic precursor.

Hz, 1H), 1.81 (m, 1H), 1.52 (m, 5H), 1.38 (m, 2H), 1.18 (s, 3H), 1.08 (m, 1H), 0.97 (s, 3H), 0.79 (s, 3H); ^{13}C NMR (C_6D_{6e}, 125 MHz) δ 186.1, 151.7, 116.0, 55.8, 54.7, 53.1, 37.9, 36.7, 36.3, 35.7, 33.9, 29.1, 26.1, 25.8, 23.2; IR (thin film) 2955, 1690, 1453, 1365 cm^{-1}; HRMS (ESI/TOF) calculated for $C_{15}H_{24}O_2$ (M–H) 235.1698, observed 235.1697; $[\alpha]_D^{24}$ +61.5° $[\alpha]_{577}^{24}$ +63.8°, $[\alpha]_{546}^{24}$ +72.5°, $[\alpha]_{435}^{24}$ 147.8°, $[\alpha]_{405}^{24}$ +189.9° (*c* = 1.0, CH_2Cl_2).



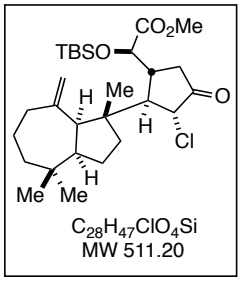
(1*R*,3a*R*,8a*S*)-1,3-dioxoisoindolin-2-yl 1,4,4-trimethyl-8-methylenedecahydroazulene-1-carboxylate (27): To a solution of **26** (0.10 g, 0.43 mmol) in THF (2 mL) was added *N*-hydroxyphthalimide (120 mg, 0.72 mmol), *N*,*N*'-dicyclohexylcarbodiimide (130 mg, 0.64 mmol) and 4-dimethylaminopyridine (2.6 mg, 0.021 mmol). The mixture was stirred for 18 h, filtered, and concentrated under reduced pressure. The residue was quickly purified by column chromatography (SiO_2, 0–5% ethyl acetate/hexanes) to afford **27** as a white solid (135 mg, 84%): m.p. 96–97 °C; R_f: 0.26 (10% ethyl acetate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz, for major isomer) δ 7.86 (dd, *J* = 3.0, 5.4 Hz, 2H), 7.76 (dd, *J* = 3.0, 5.4 Hz, 2H), 4.95 (s, 1H), 4.81 (s, 1H), 3.34 (d, *J* = 7.9 Hz, 1H), 2.47 (m, 1H), 2.35 (dd, *J* = 4.8, 12.2 Hz, 1H), 2.22 (apt q, *J* = 10.4 Hz, 1H), 1.79 (m, 6H), 1.38 (m, 1H), 1.31 (s, 3 H), 1.26 (m, 1H), 1.02 (s, 3H), 0.92 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 175.1, 162.4, 150.5, 134.8, 129.2, 124.0, 116.8, 55.8, 53.7, 52.8, 37.7, 36.8, 36.2, 35.8, 33.9, 28.7, 26.0, 25.4, 22.4; IR (thin film) 2925, 1779, 1744, 1364, 1034 cm^{-1}; HRMS (ESI/TOF) calculated for $C_{23}H_{27}NO_4$ (M+Na) 404.1838, observed 404.1836; $[\alpha]_D^{24}$ –19.4° $[\alpha]_{577}^{24}$ –21.1°, $[\alpha]_{546}^{24}$ –25.3°, $[\alpha]_{435}^{24}$ –43.1°, $[\alpha]_{405}^{24}$ –47.3° (*c* = 1.0, CH_2Cl_2).



(*R*)-methyl 2-(*tert*-butyldimethylsilyloxy)-2-((*R*)-3-chloro-4-oxocyclopent-2-enyl)acetate (28): A solution of (*R*)-methyl 2-(*tert*-butyldimethylsilyloxy)-2-((*R*)-4-oxocyclopent-2-enyl)ethanoate (500 mg, 1.76 mmol)[9] in CH_2Cl_2 (18 mL) was cooled to 0 °C and Et_4NCl_3 (620 mg, 2.7 mmol)[10] was added. The yellow solution was stirred for 20 min and concentrated. The residue was purified by column chromatography (SiO_2, 10–20% ethyl acetate/hexanes) to afford the desired enone **28** as a clear oil (465 mg, 83%): R_f: 0.26 (10% Ethyl

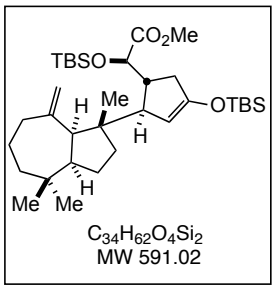
[9] M. J. Schnermann, L. E. Overman, *J. Am. Chem. Soc.* **2011**, *133*, 16425.
[10] Prepared according to: T. Schlama, K. Gabriel, V. Gouverneur, C. Mioskowski, *Angew. Chem., Int. Ed.* **1997**, *36*, 2342; *Angew. Chem.* **1997**, *109*, 2440.

acetate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz, for major isomer) δ 7.44 (d, 1H, J = 2.8 Hz), 4.19 (d, 1H, J = 5.4 Hz), 3.76 (s, 3H), 3.33 (m, 1H), 2.60 (dd, 1H, J = 6.7, 18.6 Hz), 2.38 (dd, 1H, J = 2.1, 18.6 Hz), 0.86 (s, 9H), 0.05 (s, 3H), 0.03 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 199.5, 172.1, 156.0, 137.4, 73.4, 52.4, 42.7, 36.5, 25.7, 18.3, –4.8, –5.3; IR (thin film) 2954, 1731, 1257, 1130 cm^{-1}; HRMS (ESI/TOF) calculated for $C_{14}H_{23}ClO_4Si$ (M+Na) 341.0952, observed 341.0953; $[\alpha]_D^{24}$ +72.8°, $[\alpha]_{577}^{24}$ +75.0°, $[\alpha]_{546}^{24}$+80.2°, $[\alpha]_{435}^{24}$ +142.8°, $[\alpha]_{405}^{24}$+163.8°, (c = 0.6, $CHCl_3$).



(R)-methyl 2-(tert-butyldimethylsilyloxy)-2-((1R,2R,3R)-3-chloro-4-oxo-2-((1R,3aR,8aS)-1,4,4-trimethyl-8-methylenedecahydroazulen-1-yl)cyclopentyl)acetate (30): To a 1 dram vial containing **27** (50 mg, 0.13 mmol), **28** (61 mg, 0.20 mmol), diethyl 1,4-dihydro-2,6-dimethyl-3,5-pyridinedicarboxylate (49 mg, 0.20 mmol) was added degassed methylene chloride (730 μL, Ar sparged), iPr$_2$EtN (44 μL, 0.29 mmol) and a 0.01 M solution of Ru(bpy)$_3$BF$_4$ in CH_2Cl_2 (130 μL, 0.0013 mmol).[11] The resulting red mixture was stirred surrounded by a blue LED strip as above for 2.5 h until **27** was consumed (TLC monitoring). Hexanes (1 mL) was added to the mixture and then the mixture was loaded on a SiO_2 column (10 g, equilibrated with hexanes). The column was quickly eluted with 5% ethyl acetate/hexanes to afford **30** as a white solid (41 mg, 61%): Rf: 0.32 (10% ethyl acetate/hexanes); ^{1}H NMR ($CDCl_3$, 500 MHz, for major isomer) δ 4.92 (s, 1H), 4.84 (s, 1H), 4.19 (d, J = 6.0 Hz, 1H), 3.93 (d, J = 3.5 Hz, 1H), 3.34 (s, 3H), 2.77 (d, J = 3.1 Hz, 1H), 2.69 (d, J = 8.9 Hz, 1H), 2.60 (m, 1H), 2.16 (m, 3H), 1.62 (m, 3H), 1.58 (m, 2H), 1.45 (m, 1H), 1.20 (m, 2H), 1.10 (s, 3H), 1.01 (s, 9H), 0.91 (s, 3H), 0.82 (m, 1H), 0.55 (s, 3H), 0.14 (s, 3H), 0.12 (s, 3H);^{13}C NMR ($CDCl_3$, 125 MHz) δ 207.9, 172.8, 154.7, 115.1, 76.9, 60.2, 56.1, 55.6, 54.0, 51.6, 50.0, 40.5, 40.0, 38.3, 38.2, 37.5, 36.6, 34.7, 29.3, 26.5, 26.25, 26.15, 26.1, 20.5, 18.8, –4.8, –4.9; IR (thin film) 2852, 1755, 1258, 1148 cm^{-1}; HRMS (ESI/TOF) calculated for $C_{28}H_{47}ClO_4Si$ (M+Na) 533.2830, observed 533.2821; $[\alpha]_D^{24}$ +41.6° $[\alpha]_{577}^{24}$ +42.8°, $[\alpha]_{546}^{24}$ +48.1°, $[\alpha]_{435}^{24}$ +86.5°, $[\alpha]_{405}^{24}$ +97.6° (c = 1.0, CH_2Cl_2). X-ray quality crystals (mp 116-117 °C) of **30** were obtained by dissolving in ethyl acetate and exposing to heptane vapor.

[11] The 0.01 M solution of Ru(bpy)$_3$(BF$_4$)$_2$ was prepared by adding Ru(bpy)$_3$Cl$_2$ (7.4 mg, 0.010 mmol) to 1 mL CH_2Cl_2 and 1 mL saturated aqueous NaBF$_4$ and agitating the resulting mixture for 2 min. The organic layer was passed through a short plug of Na_2SO_4 and used without further purification.

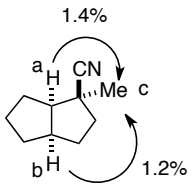


(*R*)-methyl 2-(*tert*-butyldimethylsilyloxy)-2-((1*R*,2*S*)-4-(*tert*-butyldimethylsilyloxy)-2-((1*S*,3a*R*,8a*S*)-1,4,4-trimethyl-8-methylenedecahydroazulen-1-yl)cyclopent-3-enyl)acetate (8): A 0.2 M solution of $Me_2CuCNLi_2$ in Et_2O (~10 mL) was prepared by addition of 1.5 M MeLi in Et_2O (2.7 mL, 4.0 mmol) to a stirred mixture of CuCN (180 mg, 2.0 mmol) in Et_2O (7.5 mL) at 0 °C to form a clear homogenous solution. To a flask at −20 °C containing TBSCl (60 mg, 0.40 mmol) was added THF (0.20 mL), HMPA (0.20 mL), and 0.2 M $Me_2CuCNLi_2$ in Et_2O (0.80 mL, 0.16 mmol) to form a clear homogenous solution. A solution of **30** (41 mg, 0.080 mmol) in THF (0.20 mL) was added slowly to generate a persistent yellow solution. After 1 h, 10:1 saturated NH_4Cl/NH_4OH (5 mL) was added to the solution and Et_2O (5 mL) and the layers were separated. The aqueous layer was washed with additional Et_2O (2 × 5 mL). The organic phases were combined, dried over Na_2SO_4, filtered, and concentrated. The residue was purified by column chromatography (SiO_2, 1–2% ethyl acetate/hexanes) to afford **8** (36 mg, 76%) that matched the material prepared previously by ^1H and ^{13}C NMR.[9]

Stereochemical Assignments

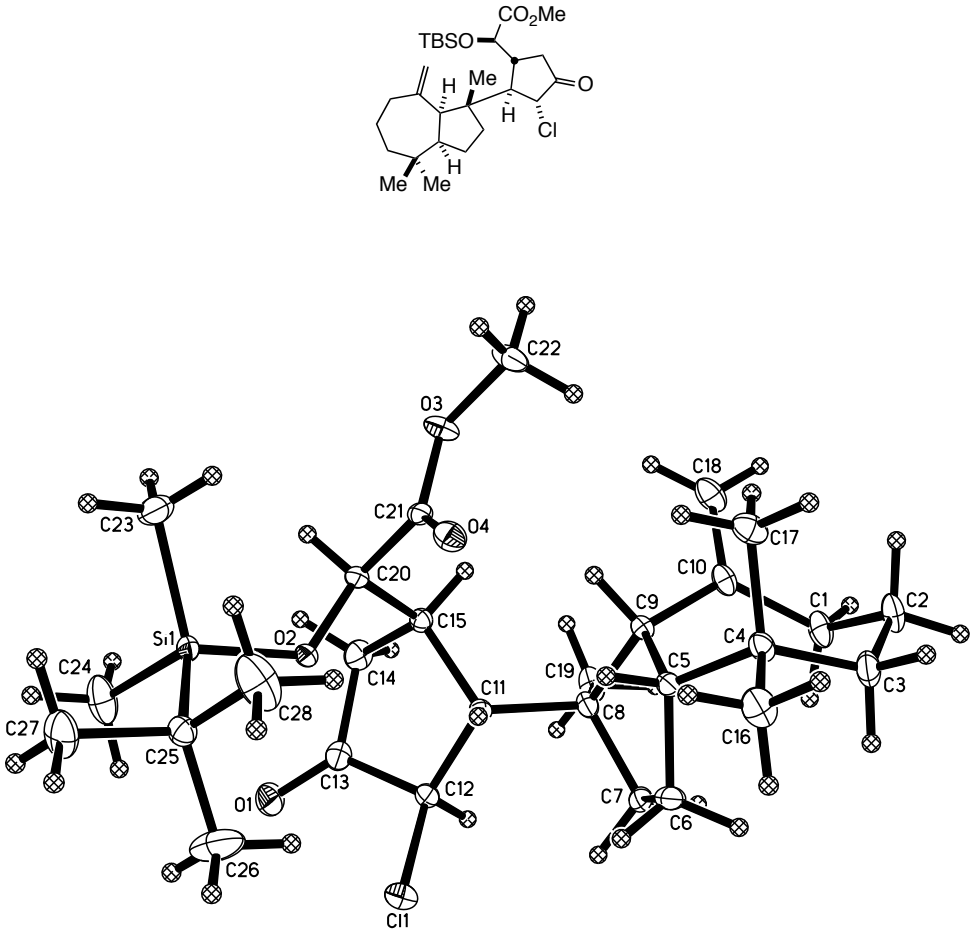
rac-1-methyl-*cis*-octahydropentalene-1-*endo*-carbonitrile (S2):

1D - NOE

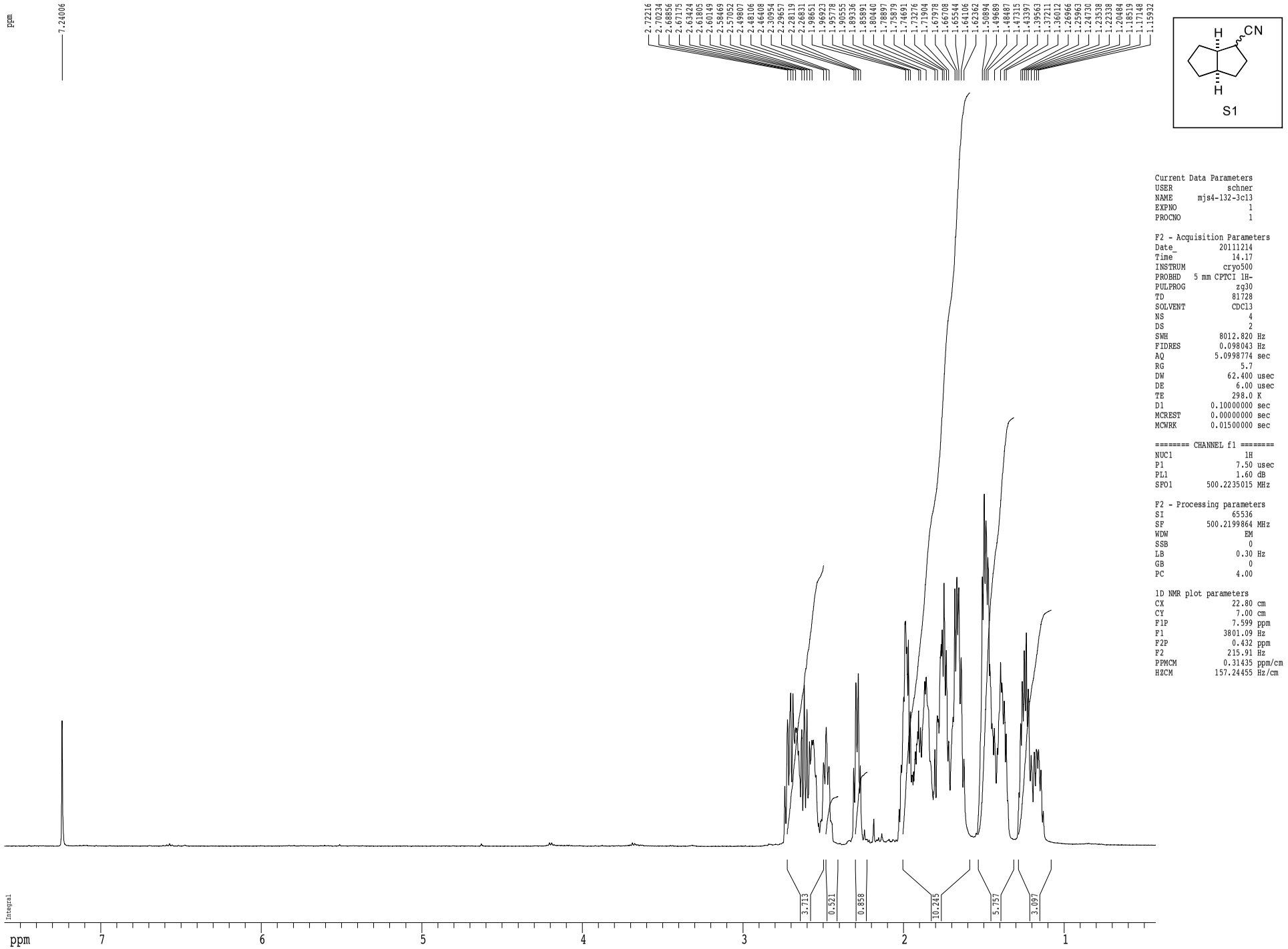


methine b 2.55 (m, 1H), 43.0
methine a 2.23 (m, 1H), 54.0
methyl c 1.41 (s, 3H), 25.3
(assigned by chemical shift
COSY, HMQC)

X-ray Structure of 30 (CCDC 885482):



1H spectrum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10.245
5.757
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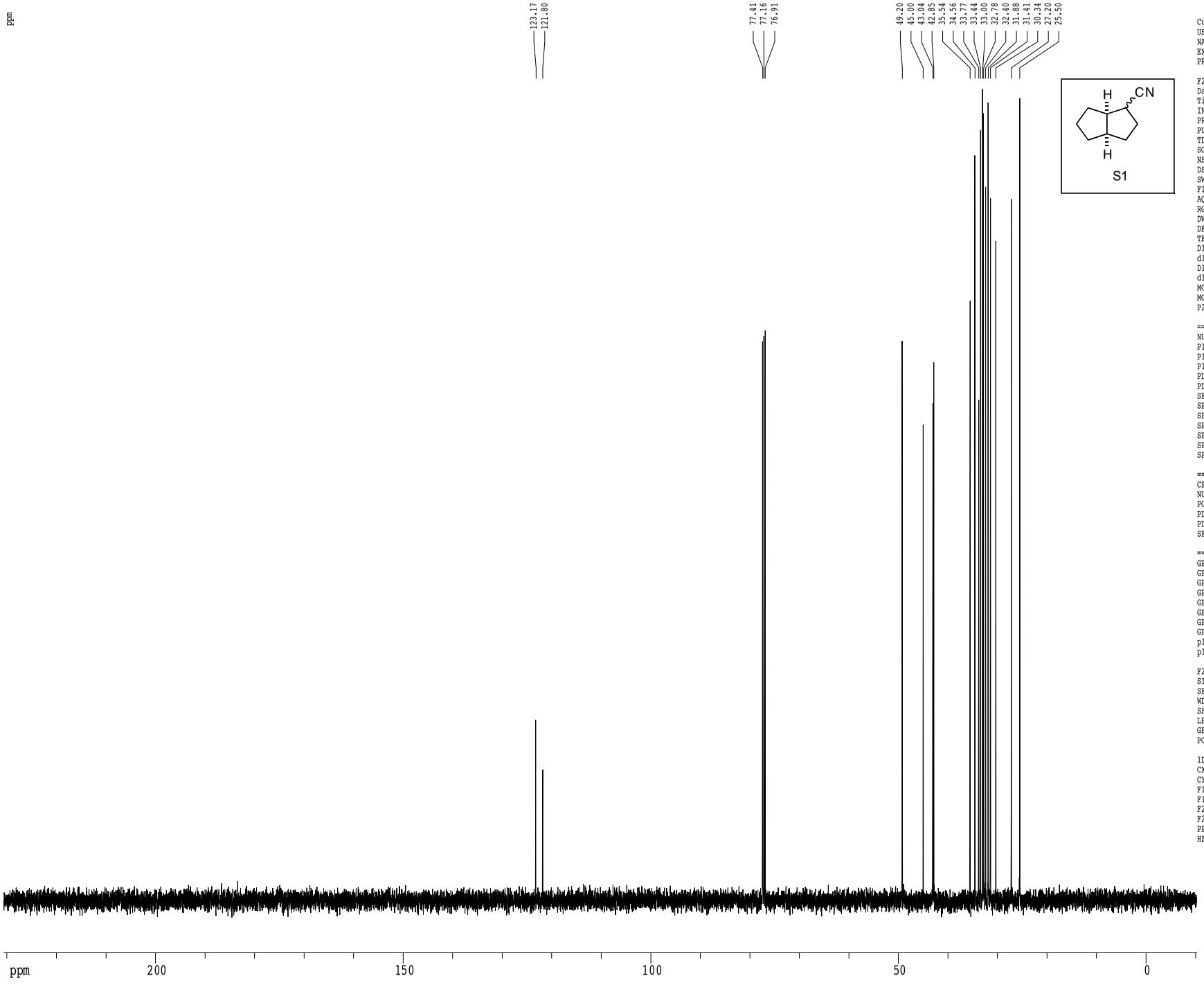
ppm
7
6
5
4
3
2
1

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



Current Data Parameters

USER	schner
NAME	mjs4-132-3c13
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20111214
Time	14.20
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	5792.6
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

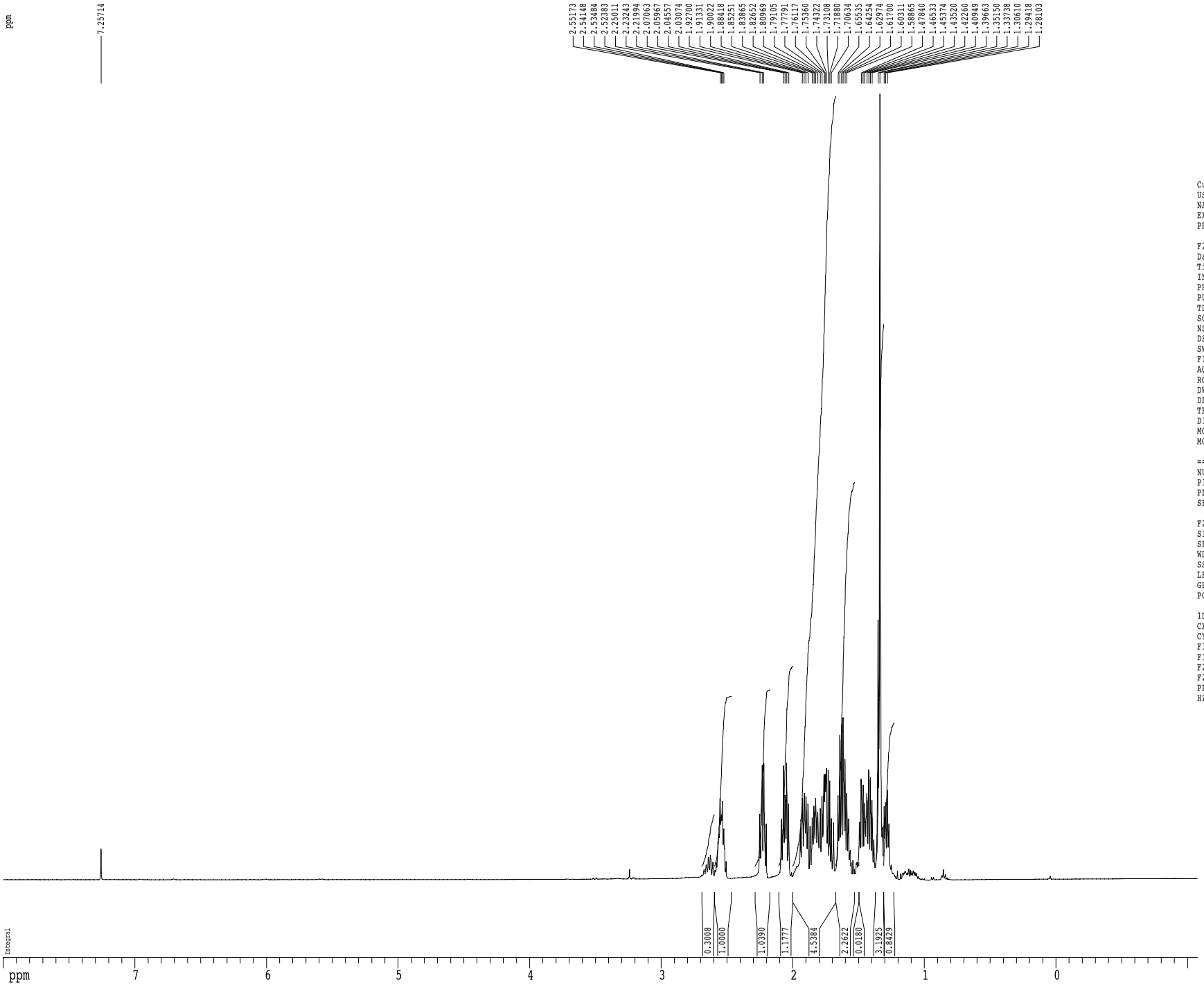
F2 - Processing parameters

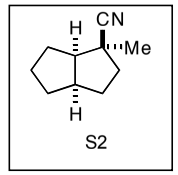
SI	65536
SF	125.7804002 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	15.65 cm
F1P	230.637 ppm
F1	29009.68 Hz
F2P	-10.287 ppm
F2	-1293.96 Hz
PPMCM	10.56688 ppm/cm
HZCM	1329.10693 Hz/cm

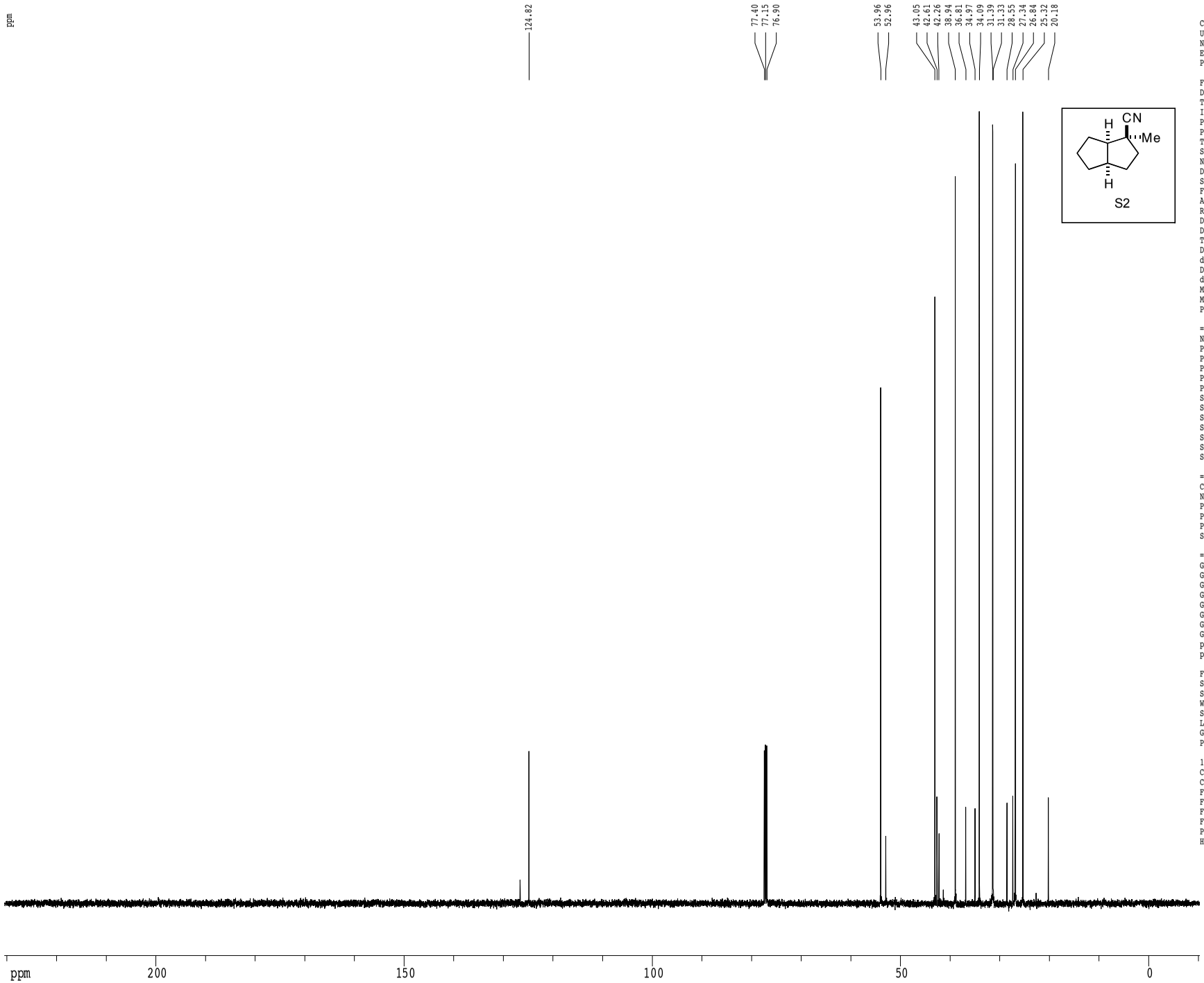
1H spectrum






```
Current Data Parameters
USER                schner
NAME          mjs4-121-3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111011
Time               13.45
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                    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           500.2200324 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                 8.000 ppm
F1                4001.76 Hz
F2P                -1.074 ppm
F2               -537.30 Hz
PPMCM             0.39799 ppm/cm
HZCM            199.08173 Hz/cm
```

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



Current Data Parameters
USER schner
NAME mjs4-121-3
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111011
Time 13.48
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 74
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

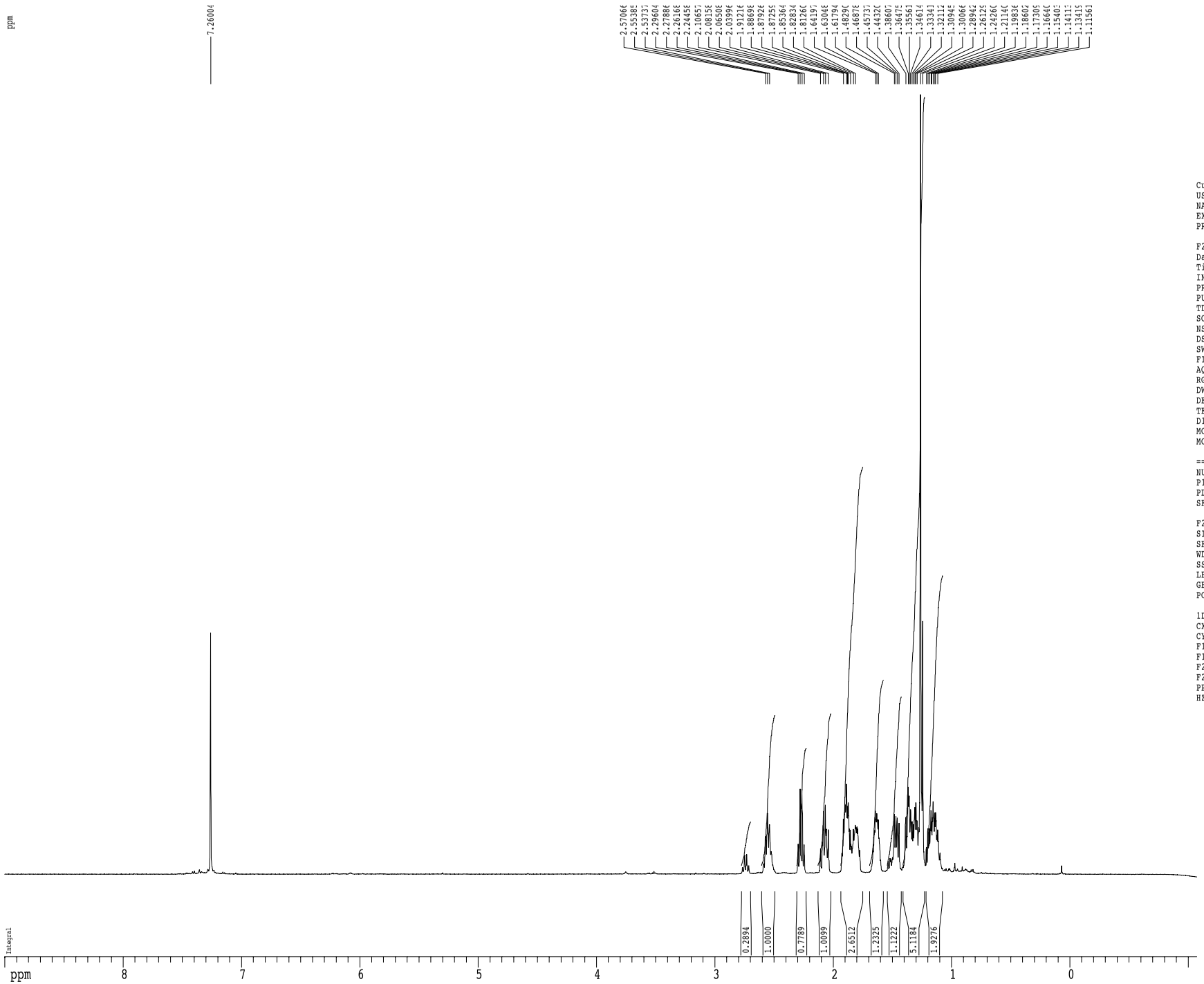
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

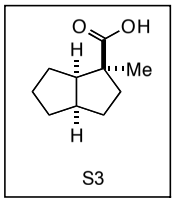
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== 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 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 230.637 ppm
F1 29009.68 Hz
F2P -10.287 ppm
F2 -1293.96 Hz
PPMCM 10.56688 ppm/cm
HZCM 1329.10706 Hz/cm





S3

Current Data Parameters
USER schner
NAME ms4-127a-3c13
EXPNO 1
PROCNO 1

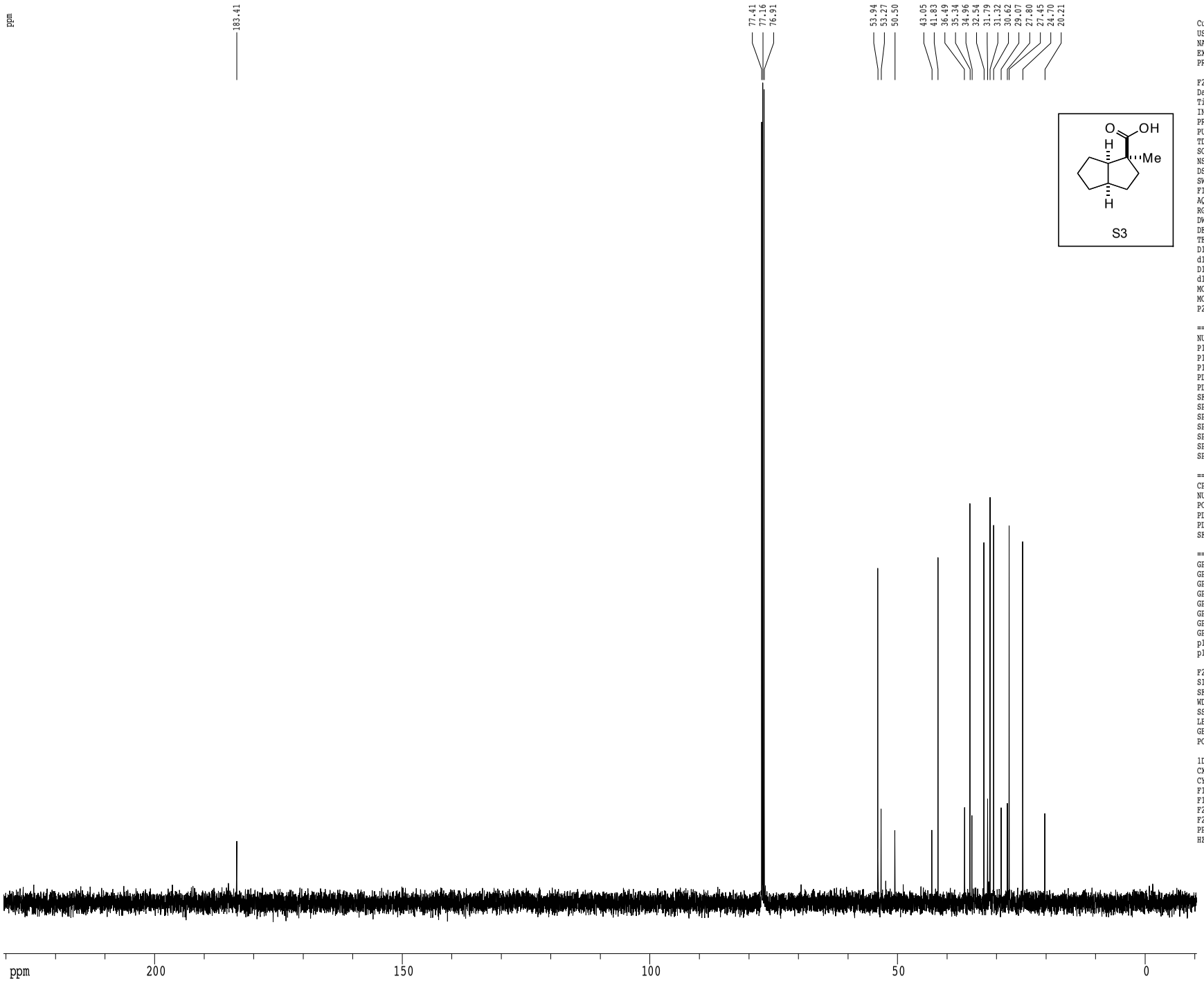
F2 - Acquisition Parameters
Date_ 20111014
Time 10.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200312 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 -1.072 ppm
F2 -536.08 Hz
PPMCM 0.44174 ppm/cm
HZCM 220.96764 Hz/cm

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



Current Data Parameters

USER	schner
NAME	ms4-127a-3c13
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20111014
Time	10.08
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	SpinEchopg30gp.prd
TD	65536
SOLVENT	CDCl3
NS	188
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

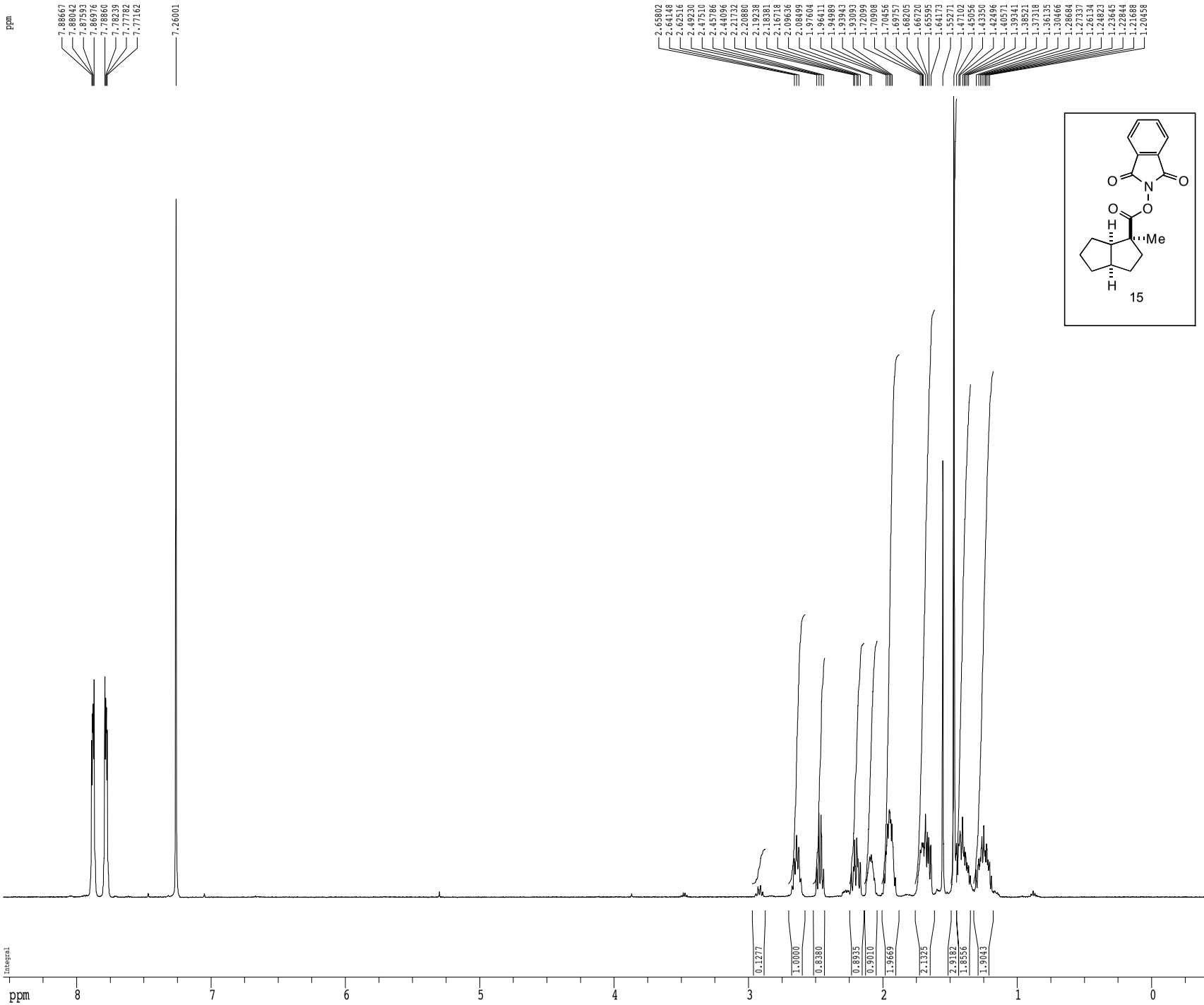
F2 - Processing parameters

SI	65536
SF	125.7804076 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

1D NMR plot parameters

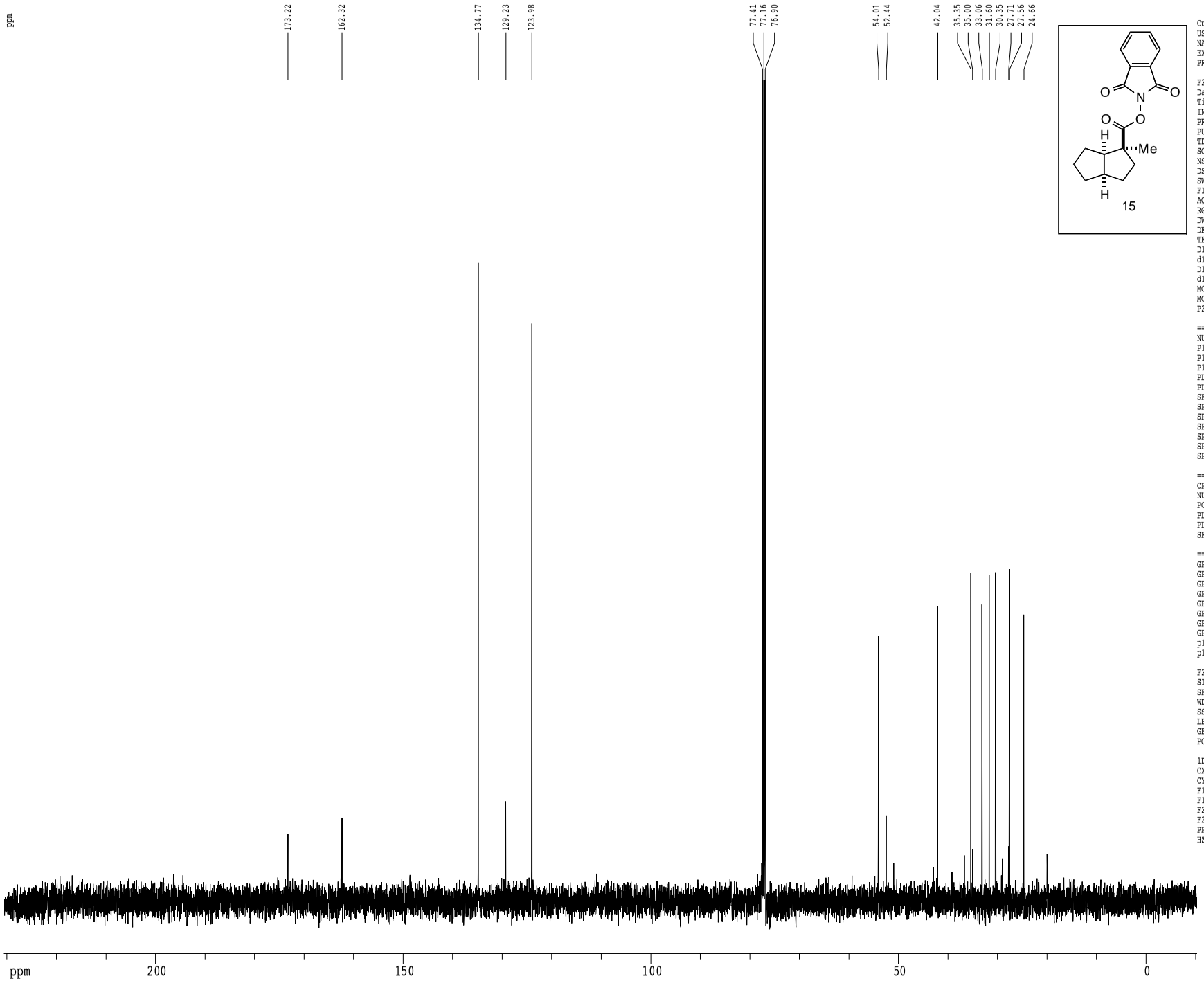
CX	22.80 cm
CY	15.65 cm
F1P	230.460 ppm
F1	28987.37 Hz
F2P	-10.460 ppm
F2	-1315.66 Hz
PPMCM	10.56667 ppm/cm
HZCM	1329.08032 Hz/cm

1H spectrum




```
Current Data Parameters
USER                schner
NAME       mjs4-155b-3c13
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111219
Time                 9.37
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  81728
SOLVENT             CDCl3
NS                     10
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            500.2200315 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                  8.545 ppm
F1                 4274.47 Hz
F2P                 -0.512 ppm
F2                -256.15 Hz
PPMCM             0.39725 ppm/cm
HZCM            198.71114 Hz/cm
```

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



Current Data Parameters

USER	schner
NAME	mjs4-155b-3c13
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20111219
Time	9.38
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	SpinEchopg30gp.prd
TD	65536
SOLVENT	CDCl3
NS	318
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

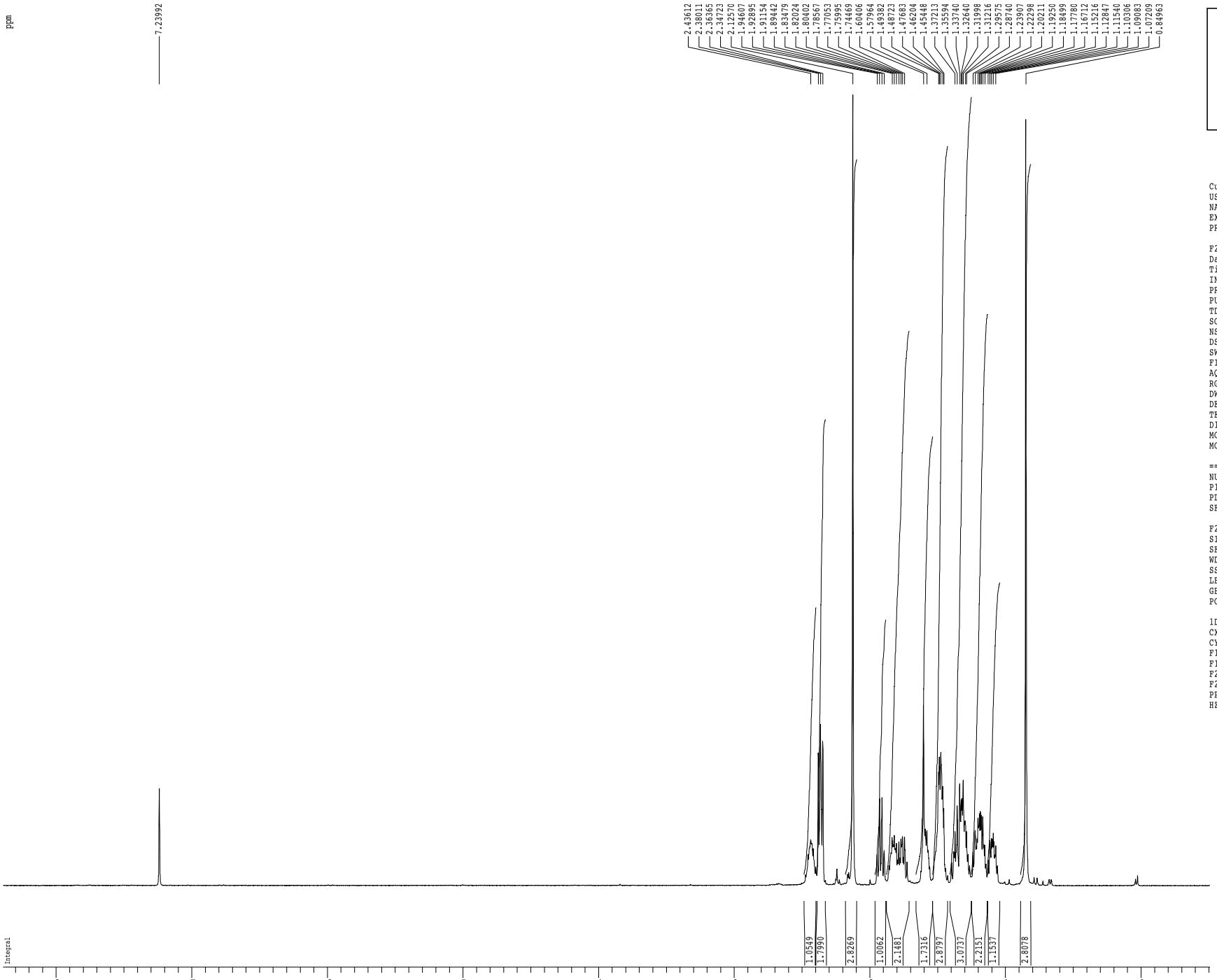
F2 - Processing parameters

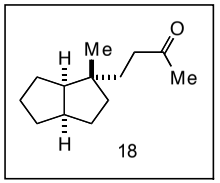
SI	65536
SF	125.7804080 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	30.00 cm
F1P	230.637 ppm
F1	29009.68 Hz
F2P	-10.287 ppm
F2	-1293.96 Hz
PPMCM	10.56688 ppm/cm
HZCM	1329.10693 Hz/cm

1H spectrum





Current Data Parameters
USER schner
NAME mjs4-246-3c13
EXPNO 1
PROCNO 1

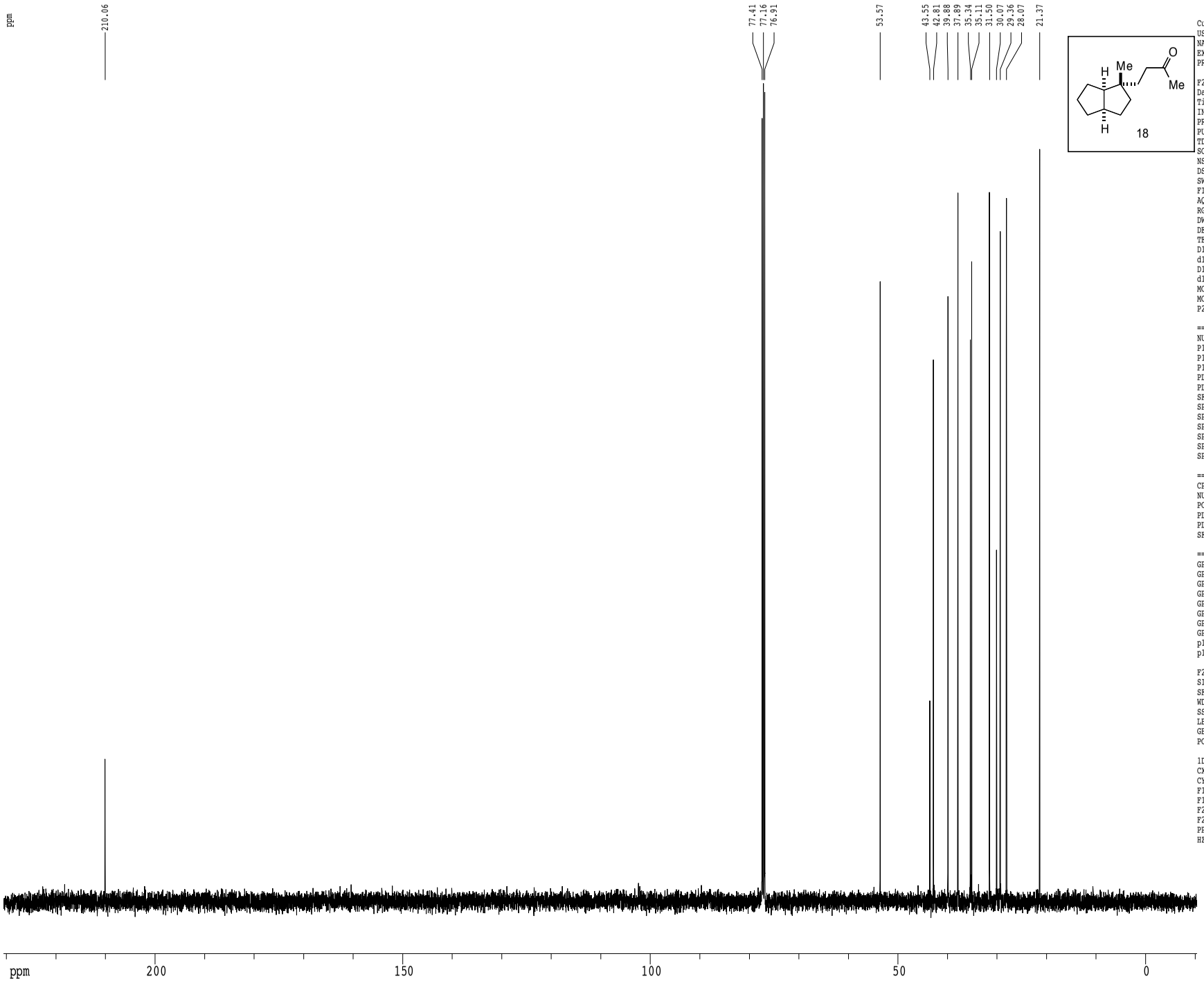
F2 - Acquisition Parameters
Date_ 20120511
Time 16.39
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 7
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200427 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 8.389 ppm
F1 4196.36 Hz
F2P -0.505 ppm
F2 -252.55 Hz
PPMCM 0.39008 ppm/cm
HZCM 195.12761 Hz/cm

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



Current Data Parameters

USER	schner
NAME	mjs4-246-3c13
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20120511
Time	16.45
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	SpinEchopg30gp.prd
TD	65536
SOLVENT	CDCl3
NS	225
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 =====

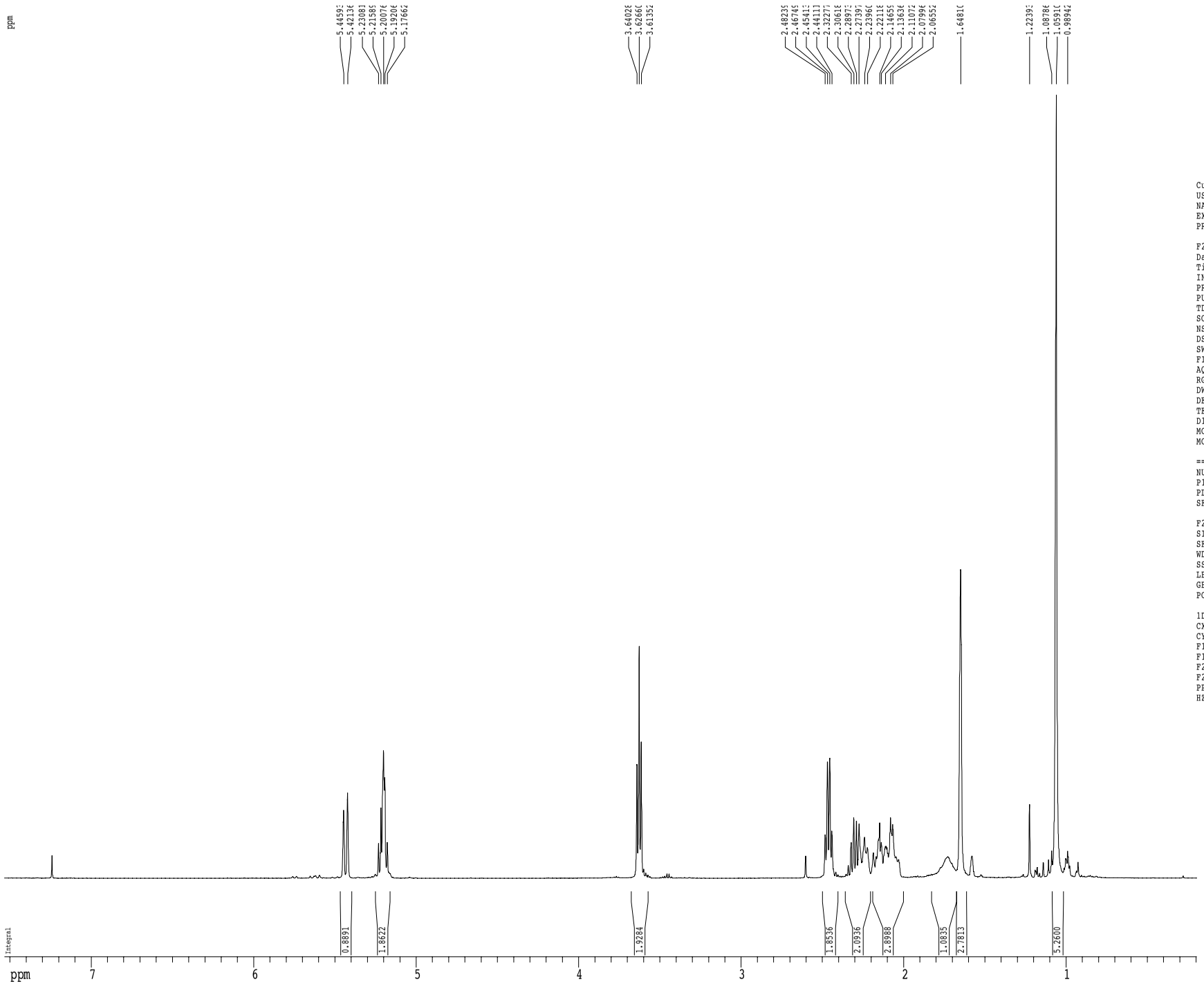
GPNAM1	SINE.100
GPNAM2	SINE.100
GPX1	0.00 %
GPX2	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPZ1	30.00 %
GPZ2	50.00 %
p15	500.00 usec
p16	1000.00 usec

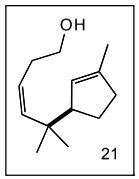
F2 - Processing parameters

SI	65536
SF	125.7804090 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	230.540 ppm
F1	28997.42 Hz
F2P	-10.380 ppm
F2	-1305.61 Hz
PPMCM	10.56667 ppm/cm
HZCM	1329.08032 Hz/cm





Current Data Parameters
USER schner
NAME ms003-24-3-1c13
EXPNO 1
PROCNO 1

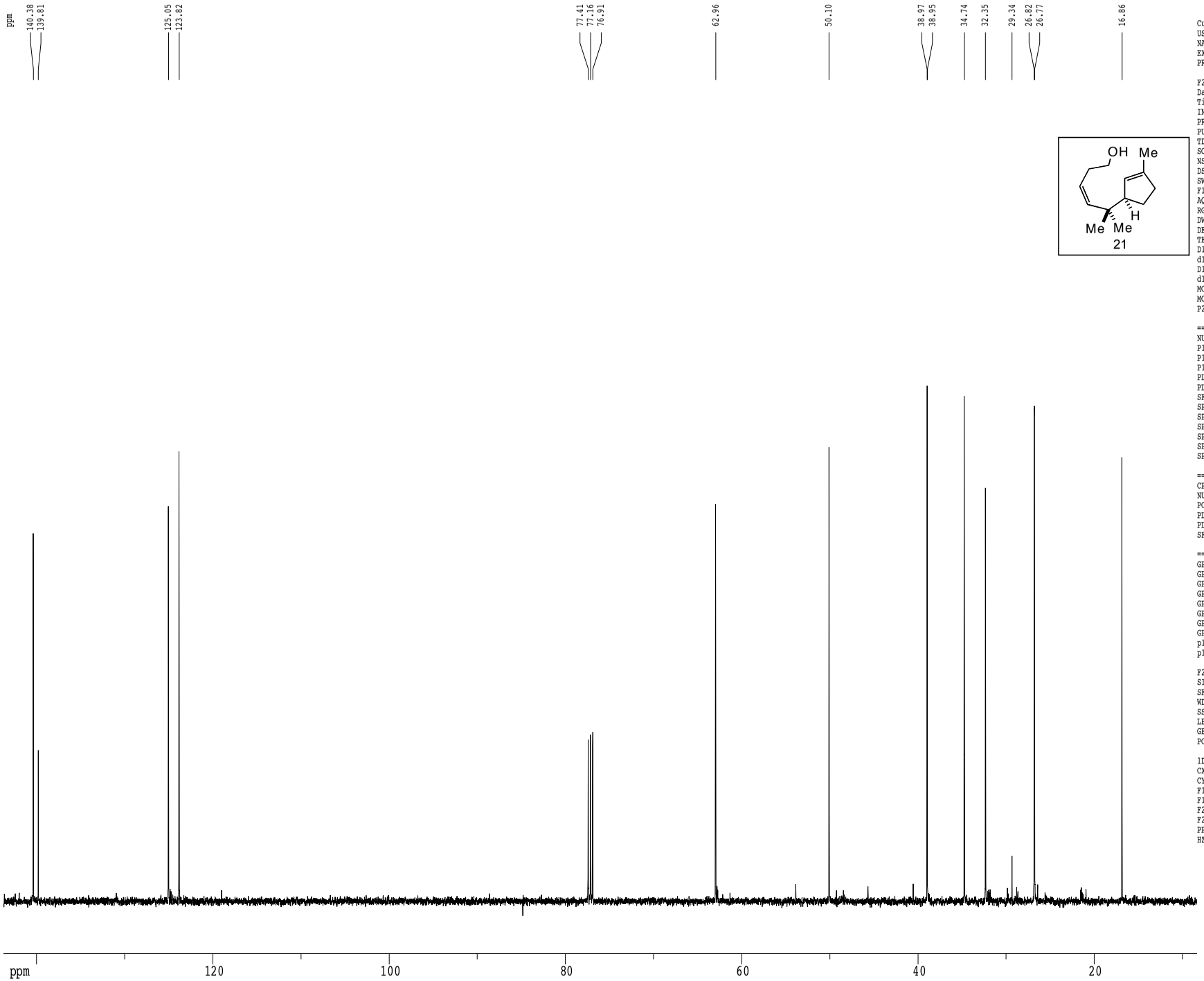
F2 - Acquisition Parameters
Date_ 20100831
Time 11.42
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 7
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2199863 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 7.533 ppm
F1 3767.96 Hz
F2P 0.197 ppm
F2 98.54 Hz
PPMCM 0.32174 ppm/cm
HZCM 160.93959 Hz/cm

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



Current Data Parameters
USER schner
NAME ms003-24-3-1c13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100831
Time 11.44
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 45
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 2580.3
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

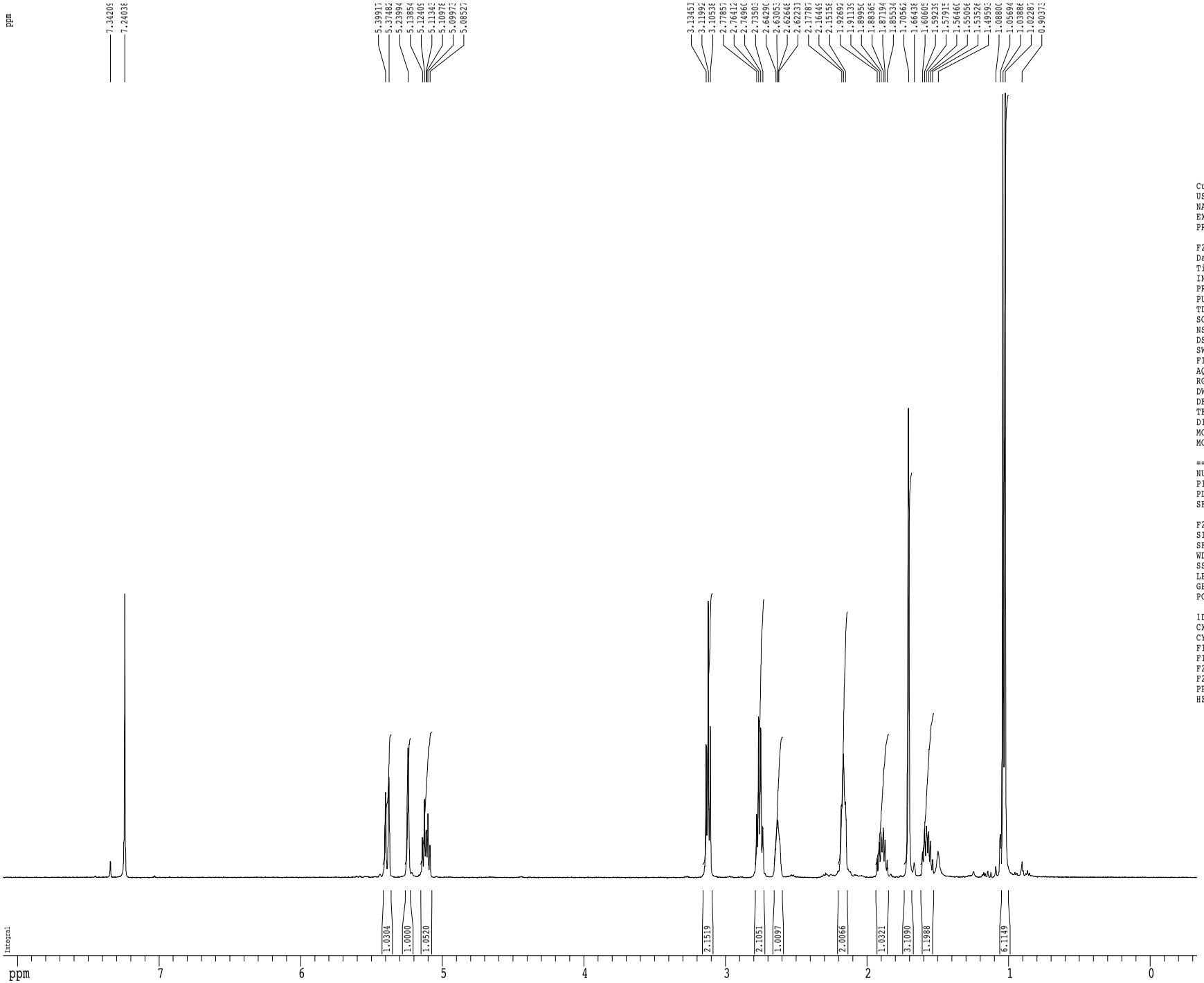
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

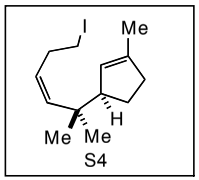
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== 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 125.7803974 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 10.00 cm
F1P 143.731 ppm
F1 18078.48 Hz
F2P 8.354 ppm
F2 1050.73 Hz
PPMCM 5.93758 ppm/cm
HZCM 746.83142 Hz/cm





Current Data Parameters
USER schner
NAME mjs4-183-3
EXPNO 1
PROCNO 1

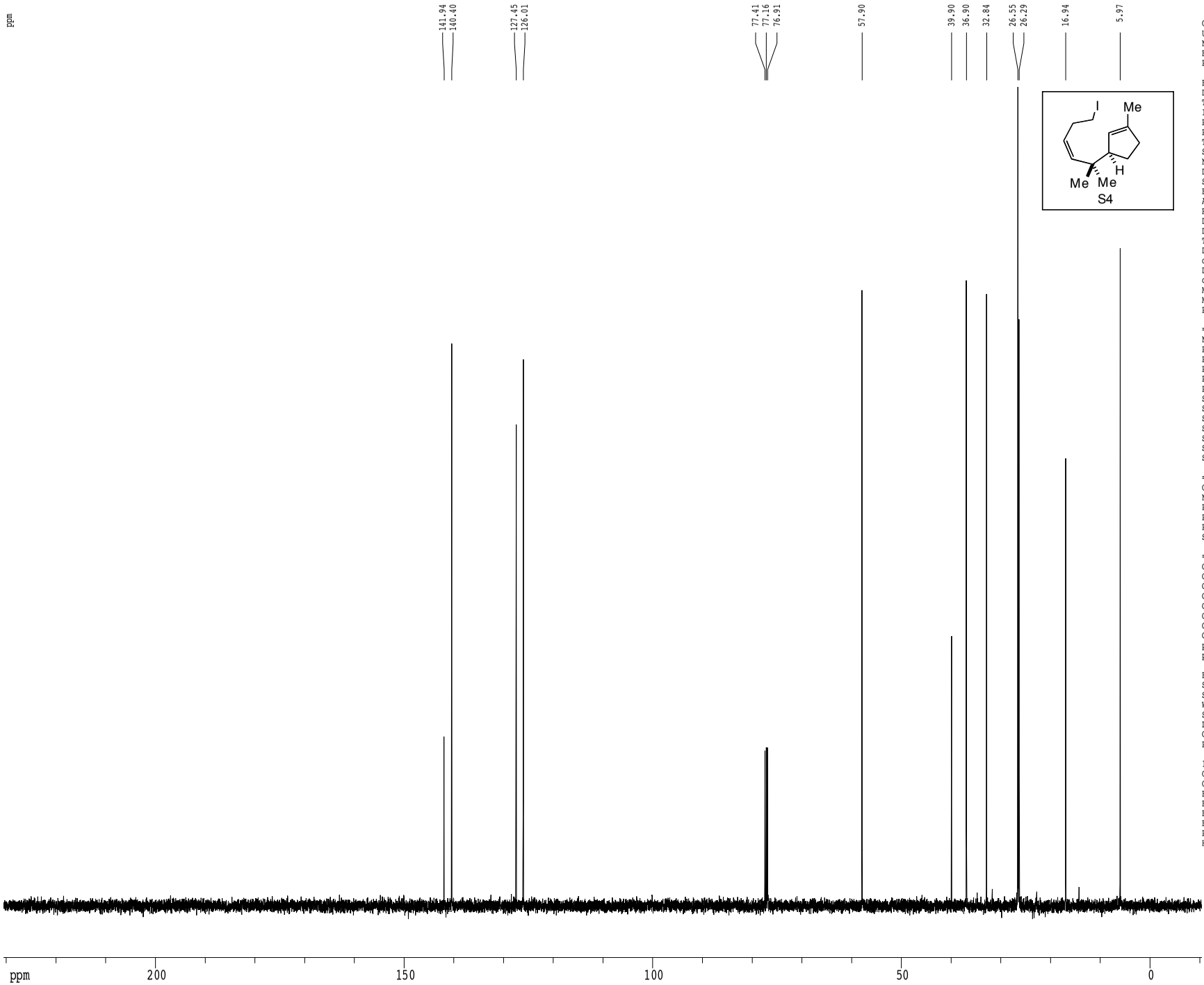
F2 - Acquisition Parameters
Date_ 20120221
Time 11.27
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 10
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 1024
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =======
NUC1 1H
P1 12.20 usec
PL1 -5.00 dB
SFO1 499.5134966 MHz

F2 - Processing parameters
SI 65536
SF 499.5100372 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 8.099 ppm
F1 4045.35 Hz
F2P -0.329 ppm
F2 -164.37 Hz
PPMCM 0.36964 ppm/cm
HZCM 184.63689 Hz/cm

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



Current Data Parameters
USER schner
NAME ms02-268-3c13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100413
Time 13.02
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 11
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

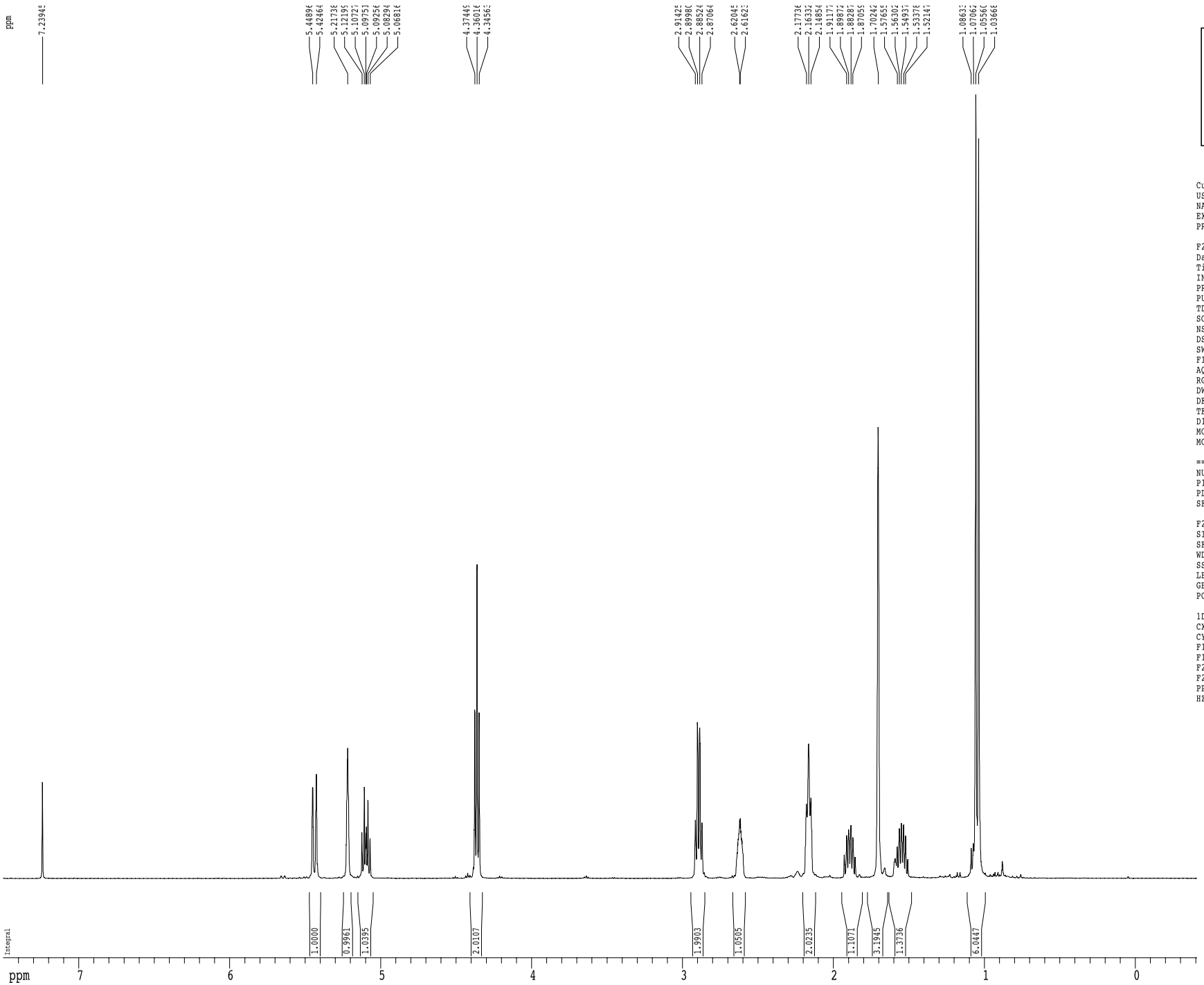
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

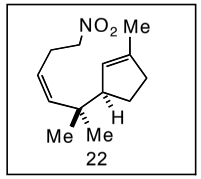
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== 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 125.7804136 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 230.503 ppm
F1 28992.79 Hz
F2P -10.417 ppm
F2 -1310.24 Hz
PPMCM 10.56667 ppm/cm
HZCM 1329.08032 Hz/cm





Current Data Parameters
USER schner
NAME ms02-258-3c13
EXPNO 1
PROCNO 1

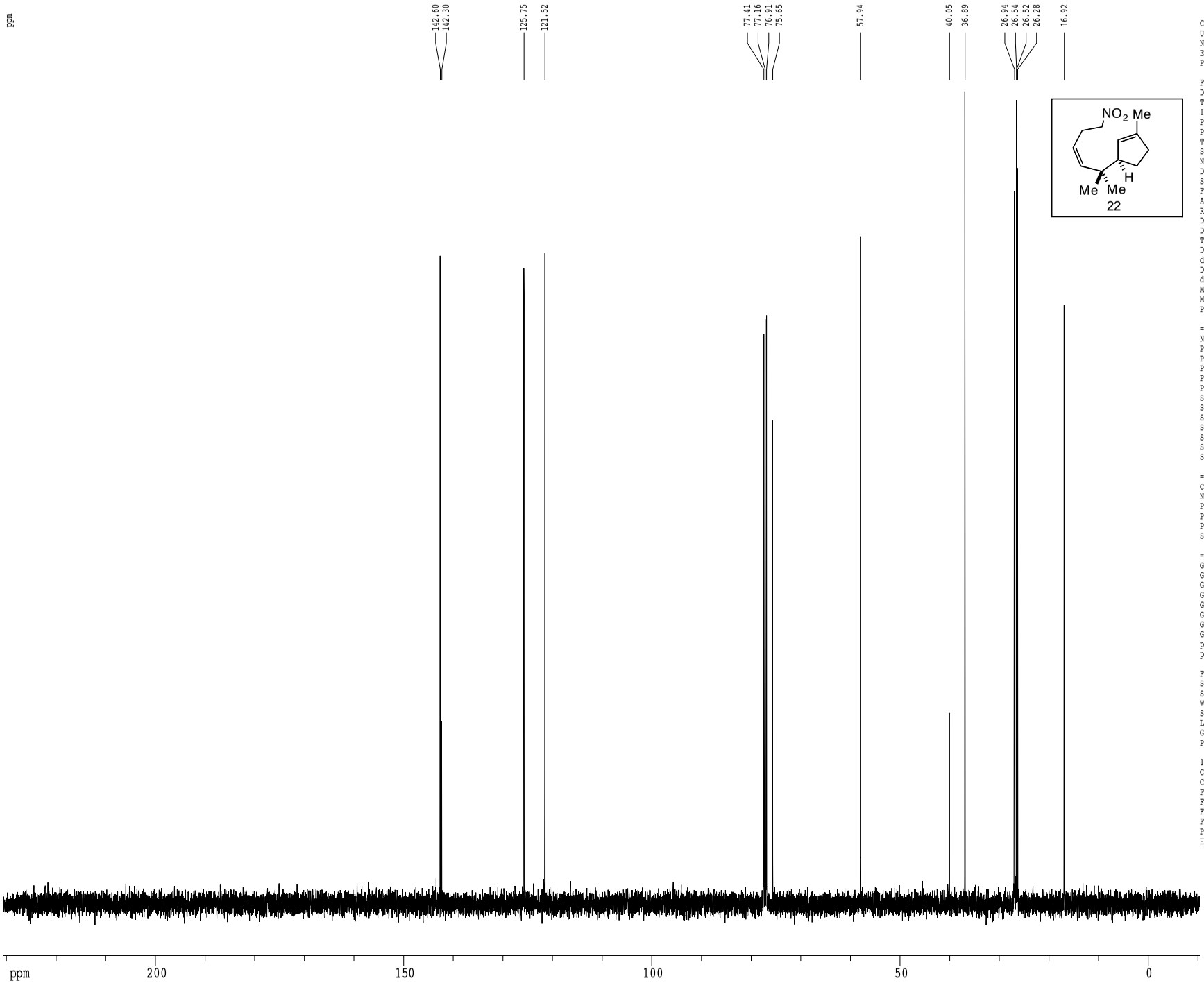
F2 - Acquisition Parameters
Date_ 20100403
Time 9.33
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 7
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 3.6
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200416 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 7.499 ppm
F1 3750.97 Hz
F2P -0.407 ppm
F2 -203.62 Hz
PPMCM 0.34674 ppm/cm
HZCM 173.44678 Hz/cm

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



Current Data Parameters
USER schner
NAME ms02-258-3c13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100403
Time 9.36
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 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

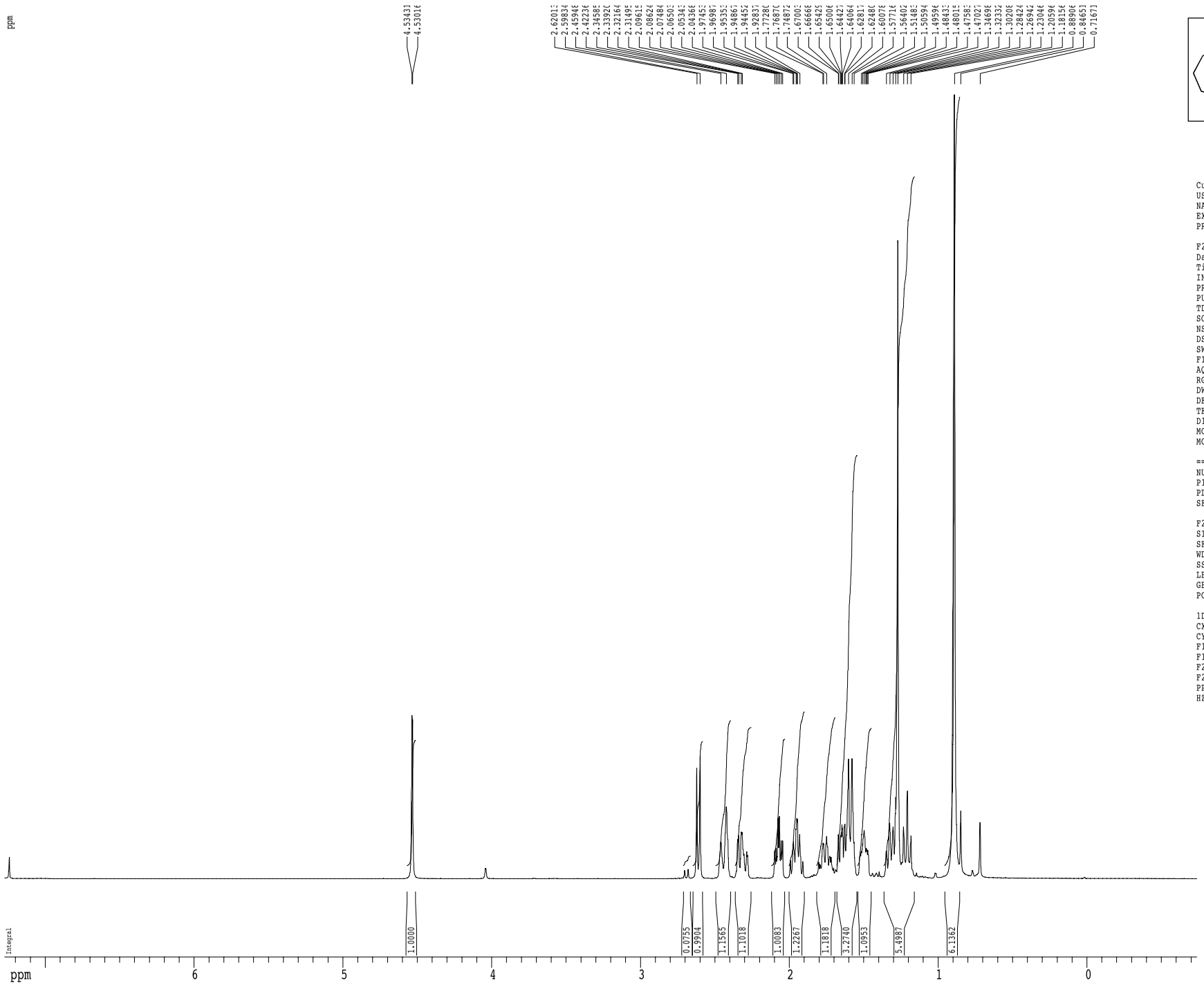
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

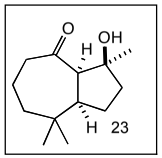
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== 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 125.7804085 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 230.544 ppm
F1 28997.88 Hz
F2P -10.376 ppm
F2 -1305.15 Hz
PPMCM 10.56667 ppm/cm
HZCM 1329.08032 Hz/cm





Current Data Parameters
USER schner
NAME ms003-121-3c13
EXPNO 1
PROCNO 1

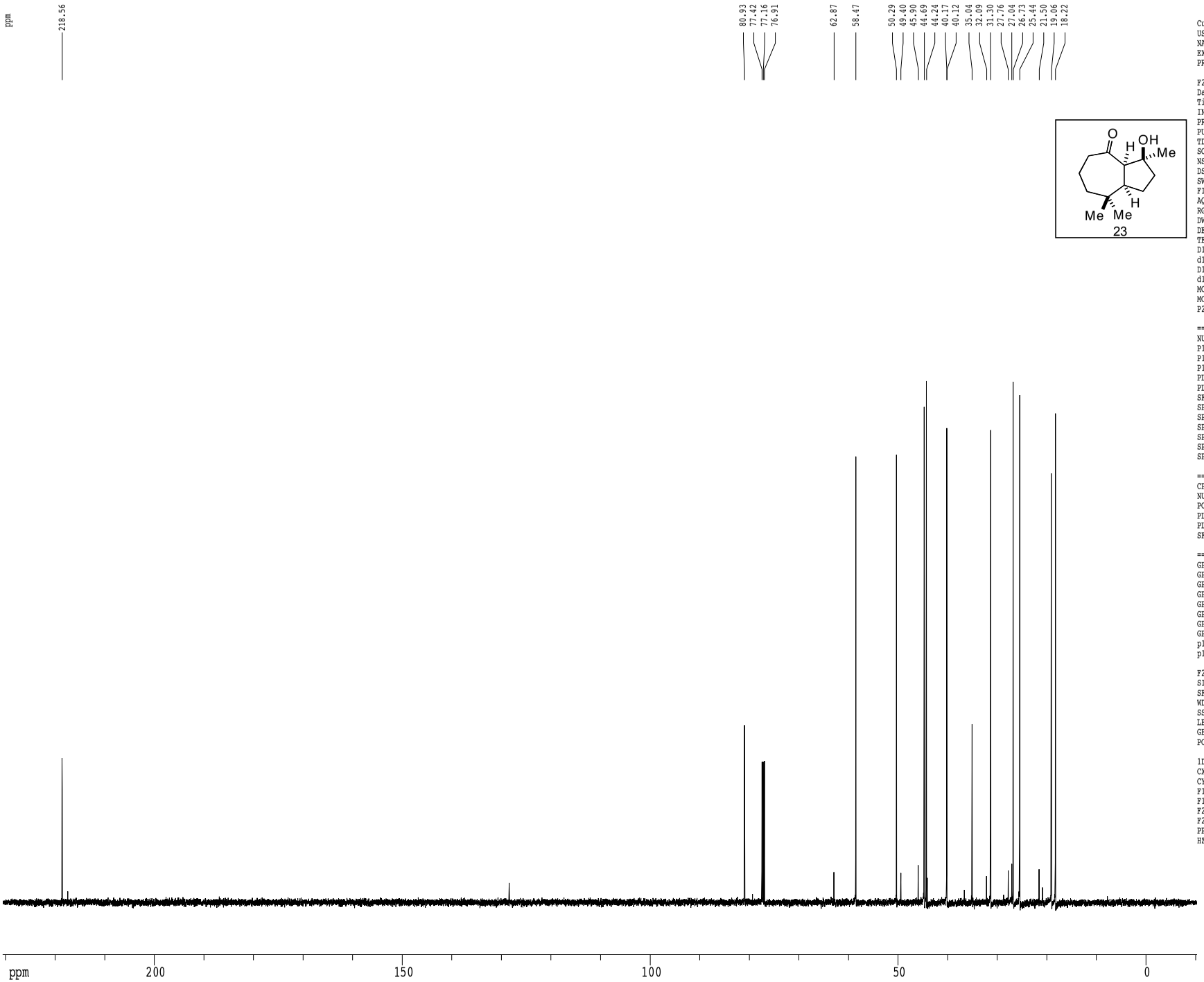
F2 - Acquisition Parameters
Date_ 20101004
Time 12.23
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 5
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200420 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 7.272 ppm
F1 3637.44 Hz
F2P -0.738 ppm
F2 -368.97 Hz
PPMCM 0.35128 ppm/cm
HZCM 175.71974 Hz/cm

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



Current Data Parameters
USER schner
NAME ms003-121-3c13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101004
Time 12.25
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 50
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

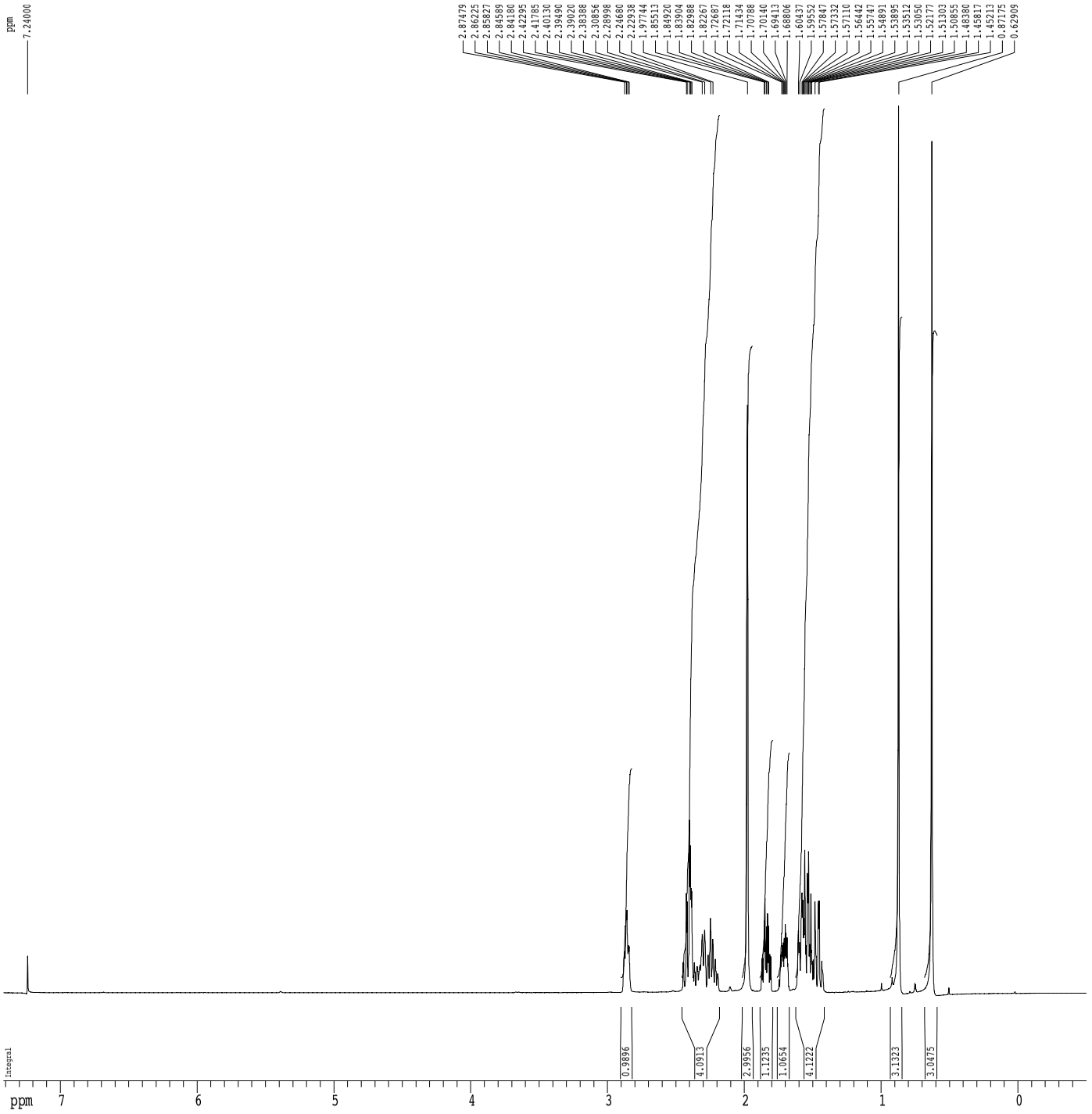
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

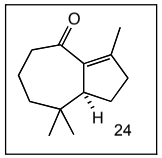
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7804145 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 10.00 cm
F1P 230.637 ppm
F1 29009.68 Hz
F2P -10.287 ppm
F2 -1293.96 Hz
PPMCM 10.56688 ppm/cm
HZCM 1329.10693 Hz/cm

1H spectrum





Current Data Parameters

USER	schner
NAME	ms003-13-3-c13
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20100614
Time	17.00
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	10
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

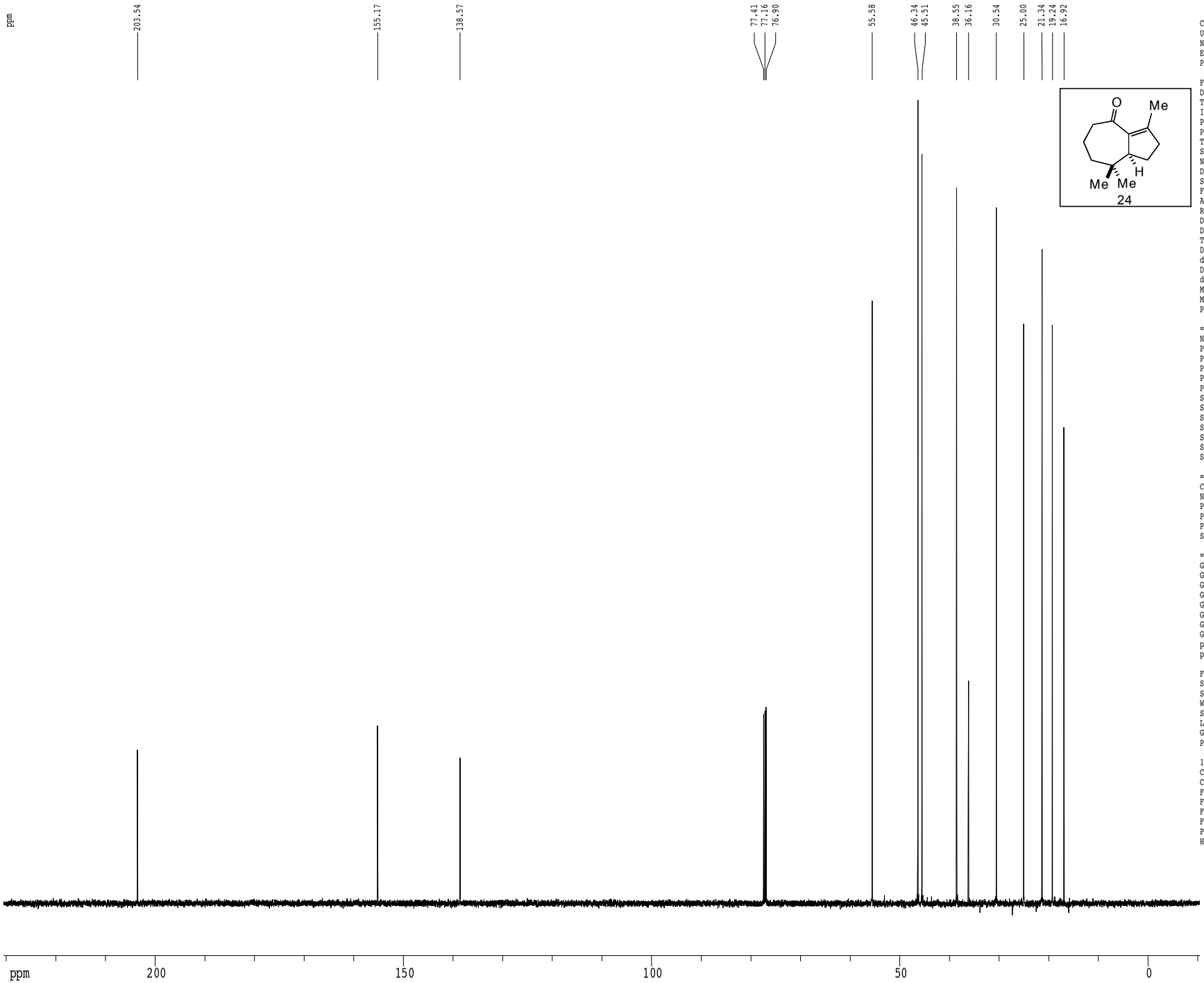
F2 - Processing parameters

SI	65536
SF	500.2200420 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	18.32 cm
CY	15.00 cm
F1P	7.414 ppm
F1	3708.49 Hz
F2P	-0.499 ppm
F2	-249.75 Hz
PPMCM	0.43194 ppm/cm
HZCM	216.06465 Hz/cm

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



Current Data Parameters
USER schner
NAME ms003-13-3-c13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100614
Time 17.03
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

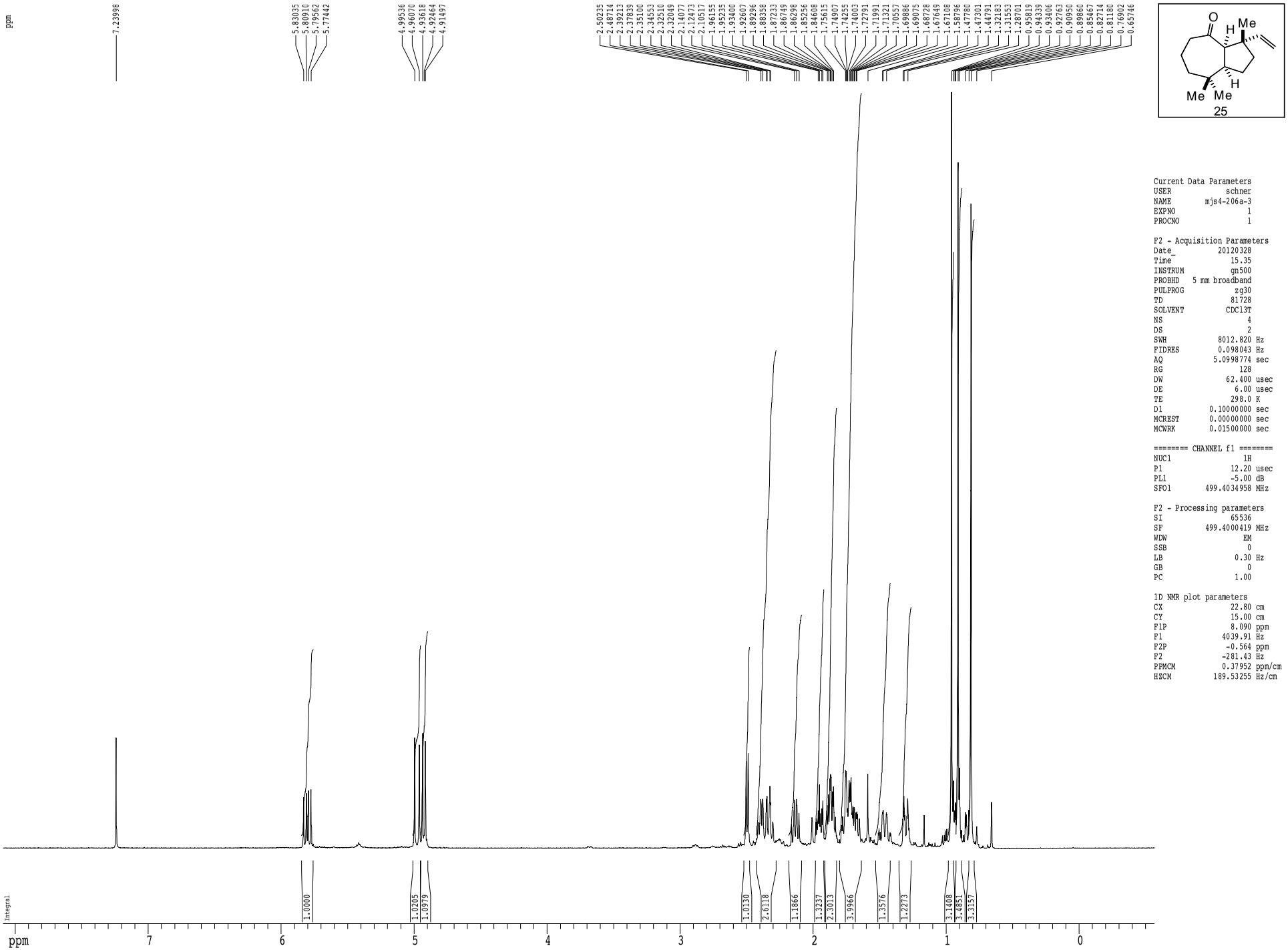
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== 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 125.7804131 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 230.507 ppm
F1 28993.25 Hz
F2P -10.413 ppm
F2 -1309.78 Hz
PPMCM 10.56667 ppm/cm
HZCM 1329.08032 Hz/cm

1H spectrum



Current Data Parameters

USER	schner
NAME	mjs4-206a-3
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20120328
Time	15.35
INSTRUM	gn500
PROBHD	5 mm broadband
PULPROG	zg30
TD	81728
SOLVENT	CDCl3T
NS	4
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	128
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	12.20 usec
PL1	-5.00 dB
SFO1	499.4034958 MHz

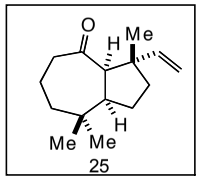
F2 - Processing parameters

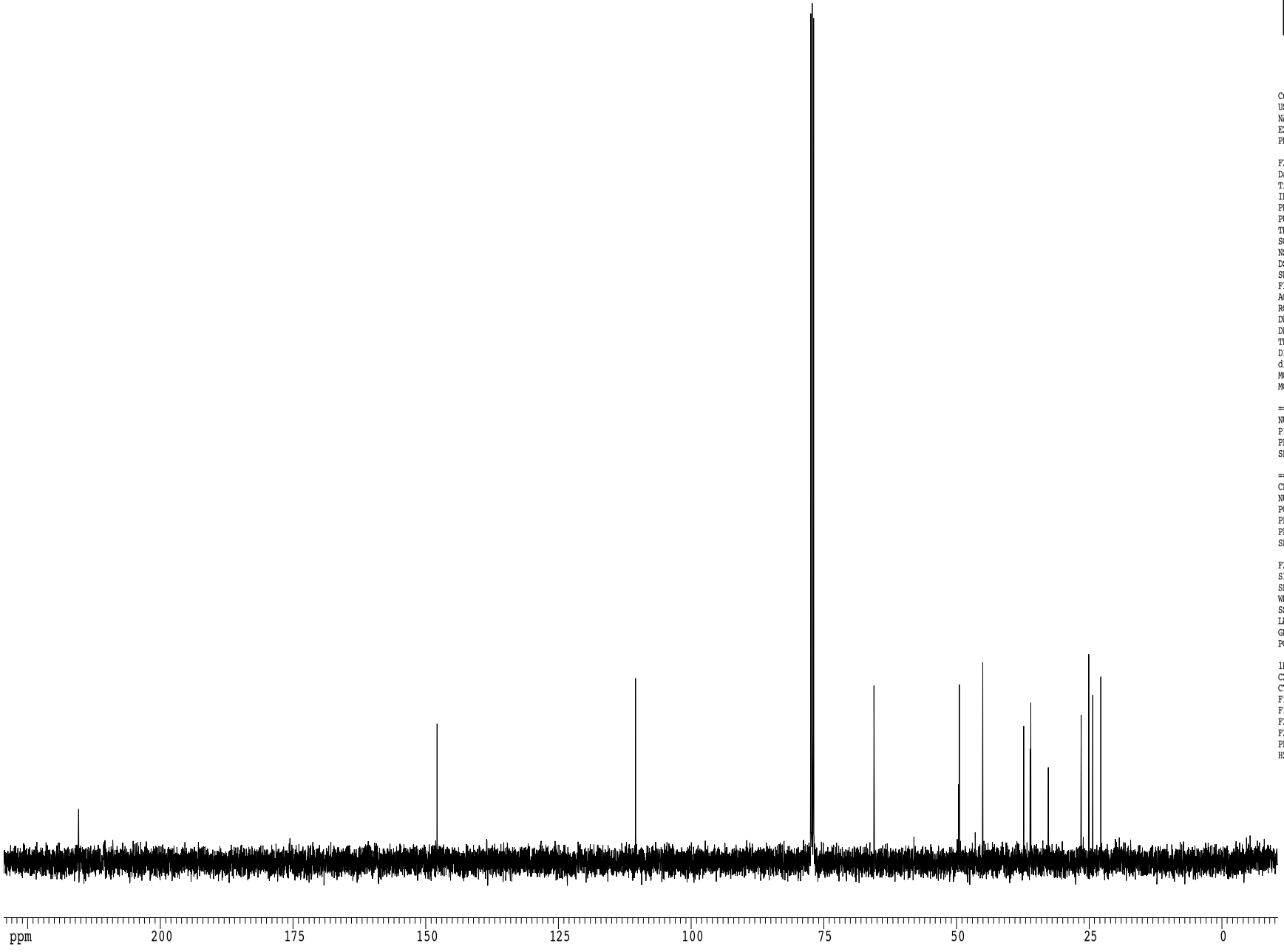
SI	65536
SF	499.4000419 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	8.090 ppm
F1	4039.91 Hz
F2P	-0.564 ppm
F2	-281.43 Hz
PPMCM	0.37952 ppm/cm
HZCM	189.53255 Hz/cm

13C spectrum with 1H decoupling





Current Data Parameters

USER	schner
NAME	mjs4-206a-3
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20120328
Time	15.36
INSTRUM	gn500
PROBHD	5 mm broadband
PULPROG	zgdc30
TD	65536
SOLVENT	CDCl3
NS	68
DS	4
SWH	30303.031 Hz
FIDRES	0.462388 Hz
AQ	1.0813940 sec
RG	46341
DW	16.500 usec
DE	4.50 usec
TE	298.0 K
D1	0.25000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	7.70 usec
PL1	0.00 dB
SFO1	125.5880432 MHz

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

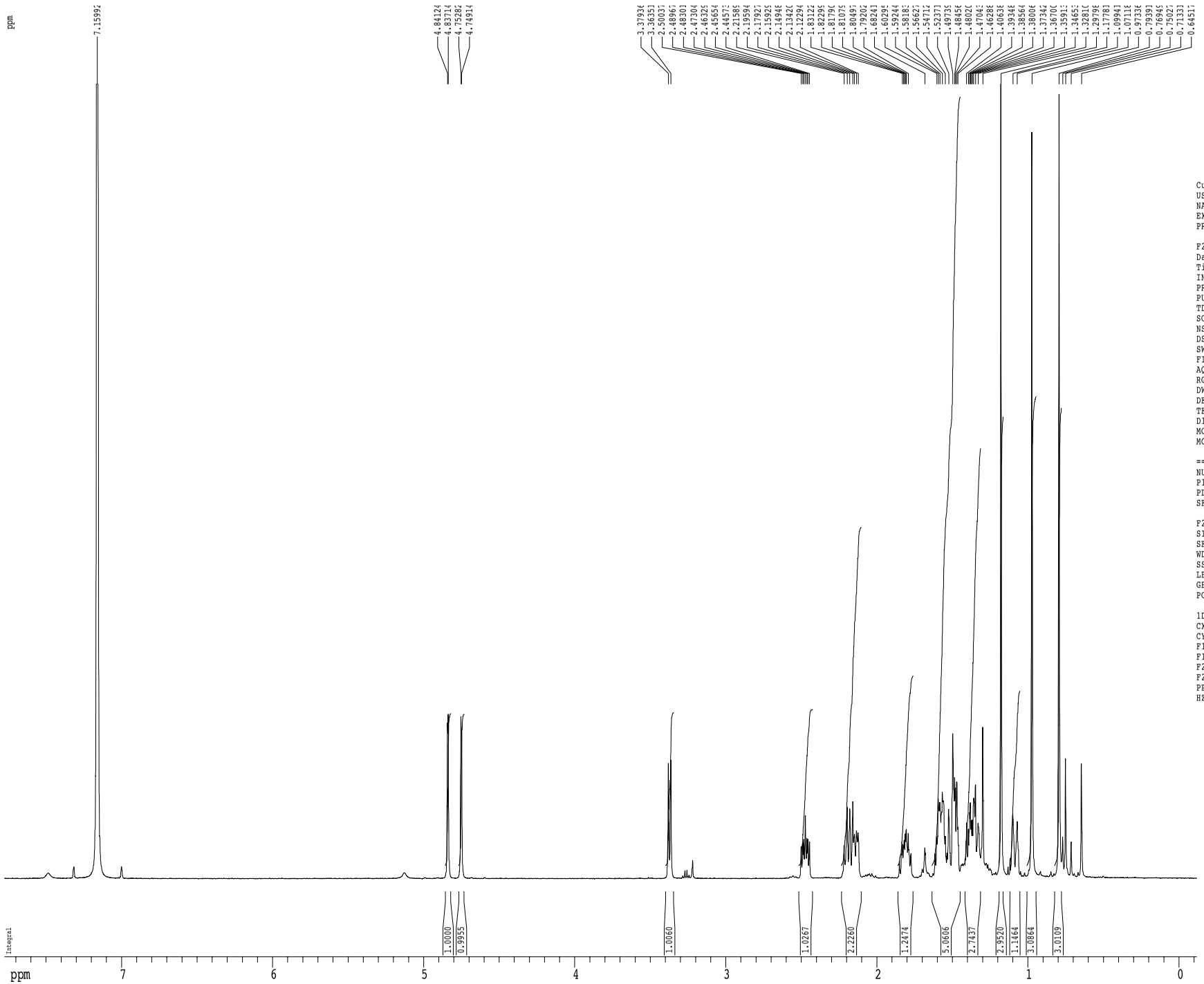
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-3.00 dB
PL12	13.20 dB
SFO2	499.4024970 MHz

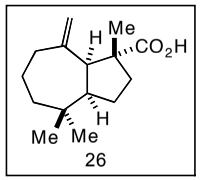
F2 - Processing parameters

SI	65536
SF	125.5742178 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	229.520 ppm
F1	28821.76 Hz
F2P	-10.507 ppm
F2	-1319.36 Hz
PPMCM	10.52747 ppm/cm
HZCM	1321.97864 Hz/cm





26

Current Data Parameters
USER schner
NAME mjs4-198i-2c13
EXPNO 1
PROCNO 1

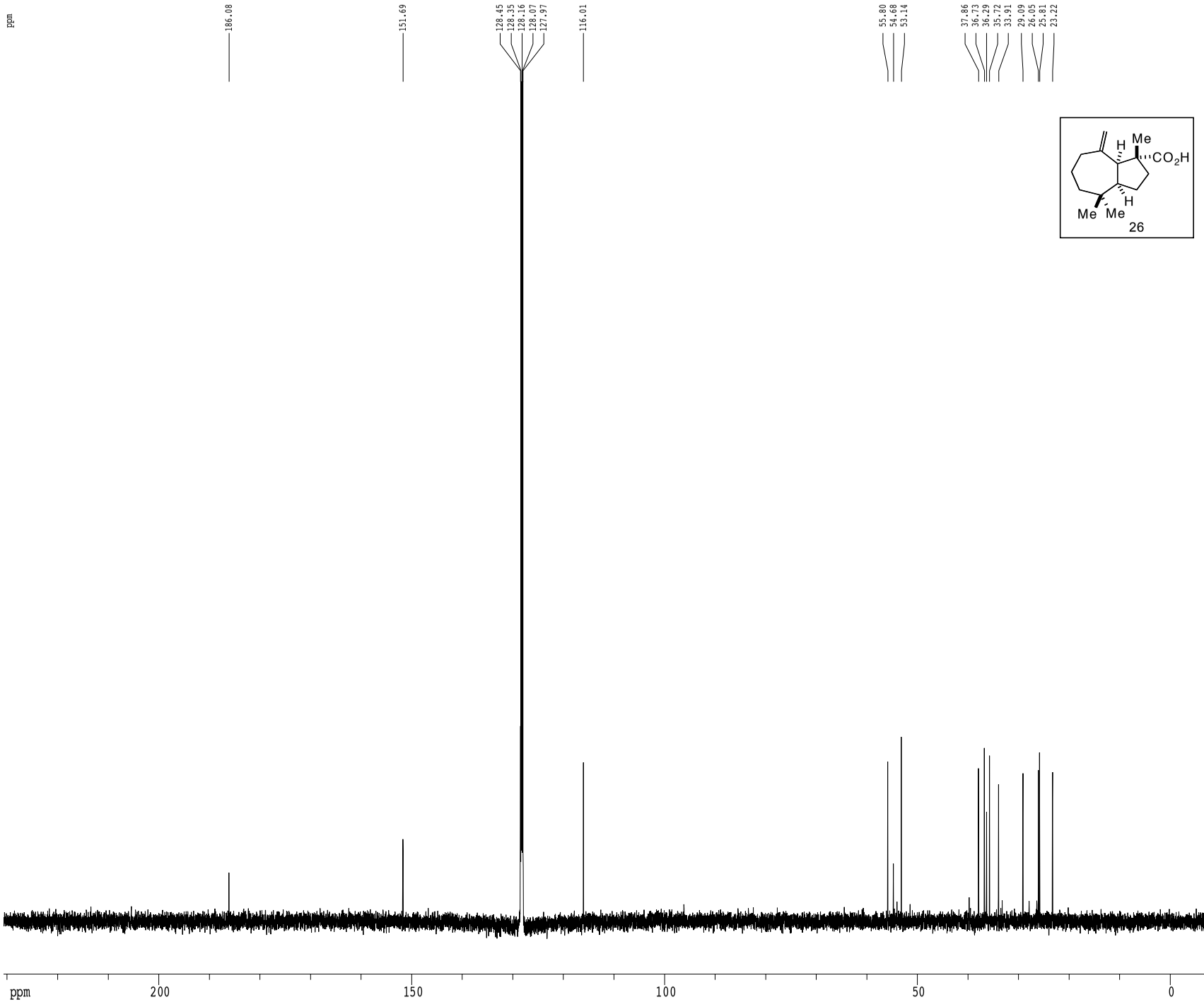
F2 - Acquisition Parameters
Date_ 20120314
Time 15.16
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT c6d6
NS 4
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200556 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 35.00 cm
F1P 7.774 ppm
F1 3888.48 Hz
F2P -0.116 ppm
F2 -58.14 Hz
PPMCM 0.34604 ppm/cm
HZCM 173.09708 Hz/cm

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



Current Data Parameters
USER schner
NAME mjs4-258iii-3-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120517
Time 13.04
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 53
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

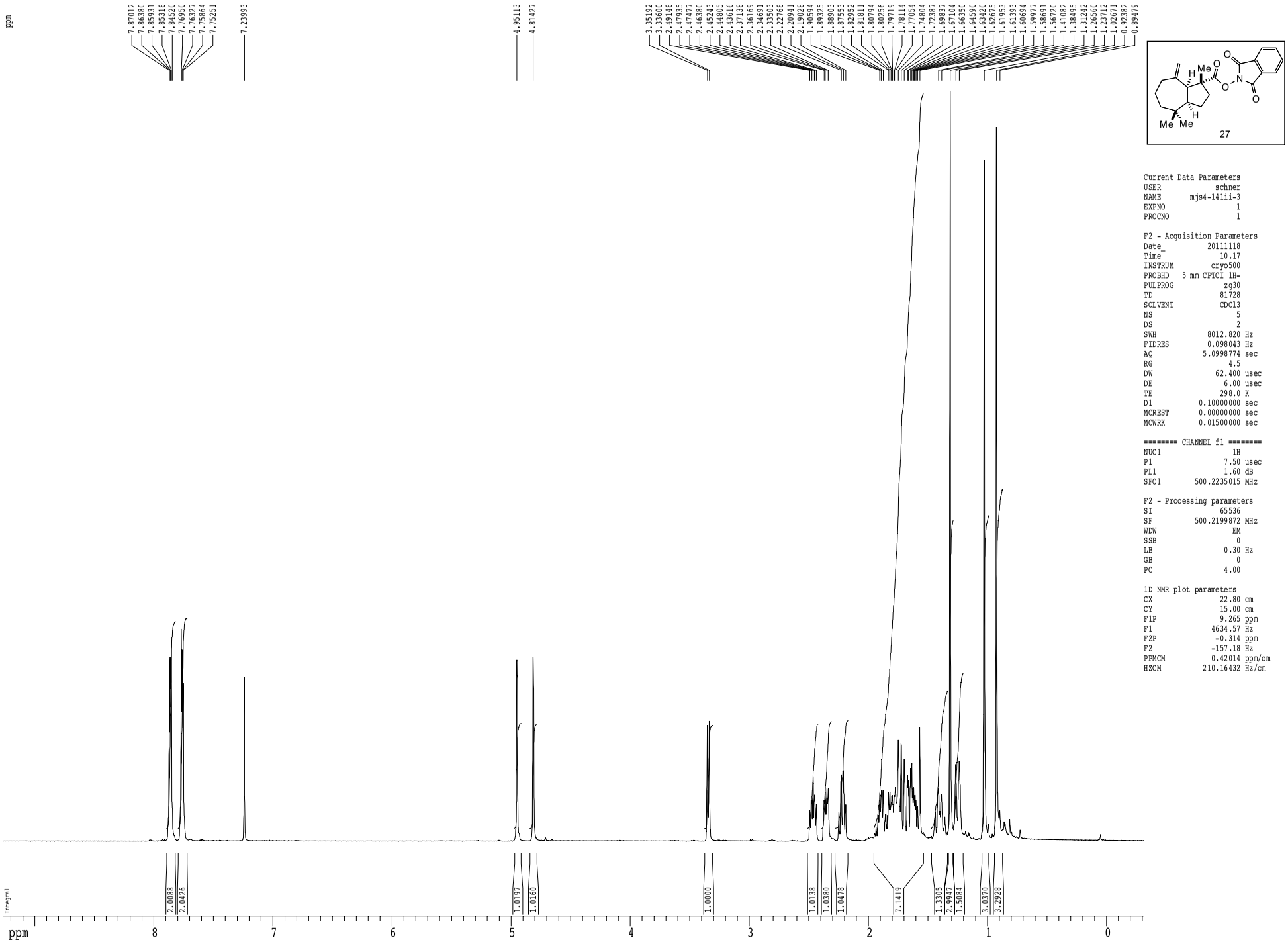
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======== 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 125.7803760 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 65.00 cm
F1P 230.637 ppm
F1 29009.67 Hz
F2P -10.287 ppm
F2 -1293.96 Hz
PPMCM 10.56688 ppm/cm
HZCM 1329.10657 Hz/cm



27

Current Data Parameters
USER schner
NAME mjs4-141ii-3
EXPNO 1
PROCNO 1

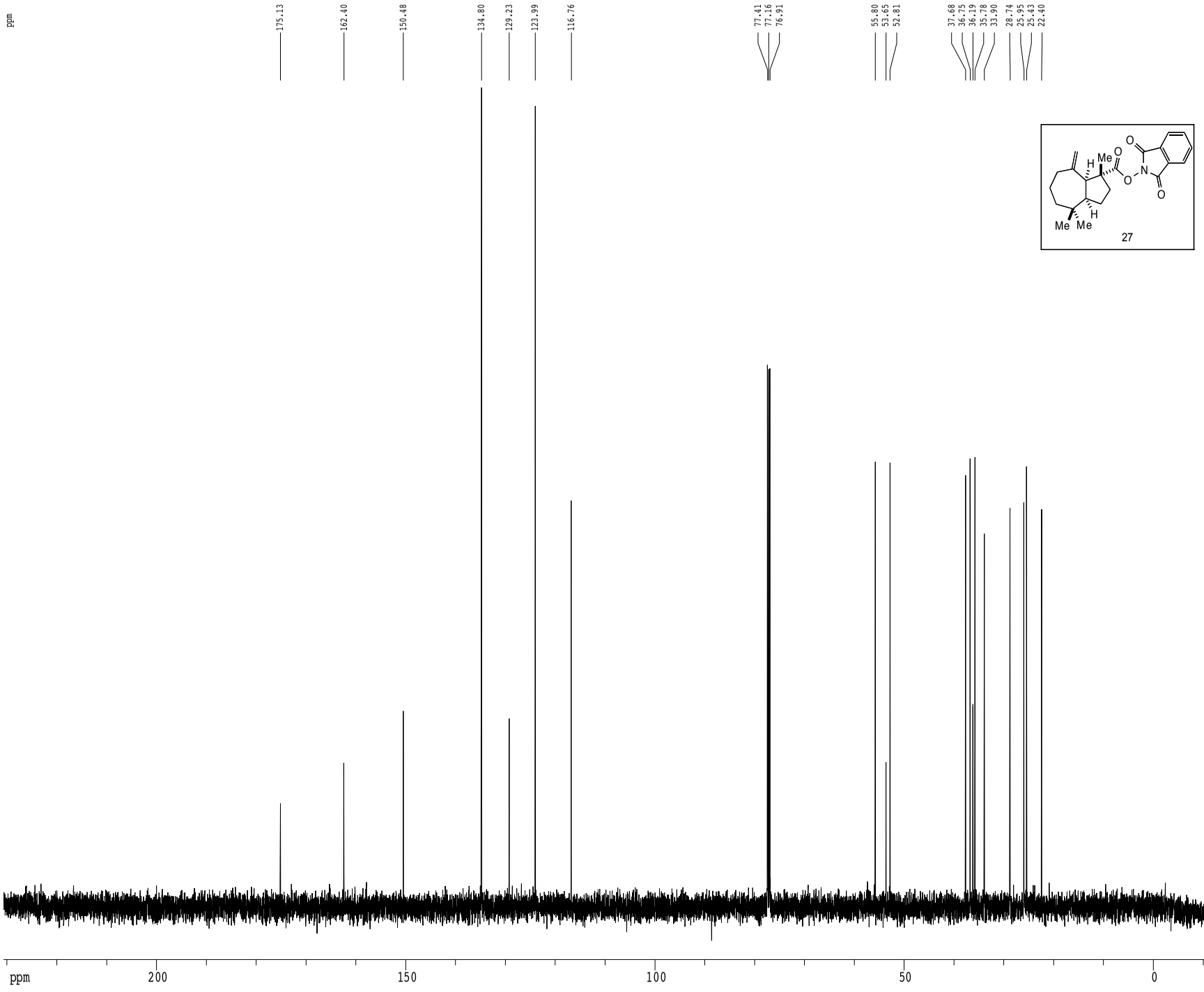
F2 - Acquisition Parameters
Date_ 20111118
Time 10.17
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 5
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2199872 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 9.265 ppm
F1 4634.57 Hz
F2P -0.314 ppm
F2 -157.18 Hz
PPMCM 0.42014 ppm/cm
HZCM 210.16432 Hz/cm

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



27

Current Data Parameters
USER schner
NAME mjs4-141ii-3
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111118
Time 10.18
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 50
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

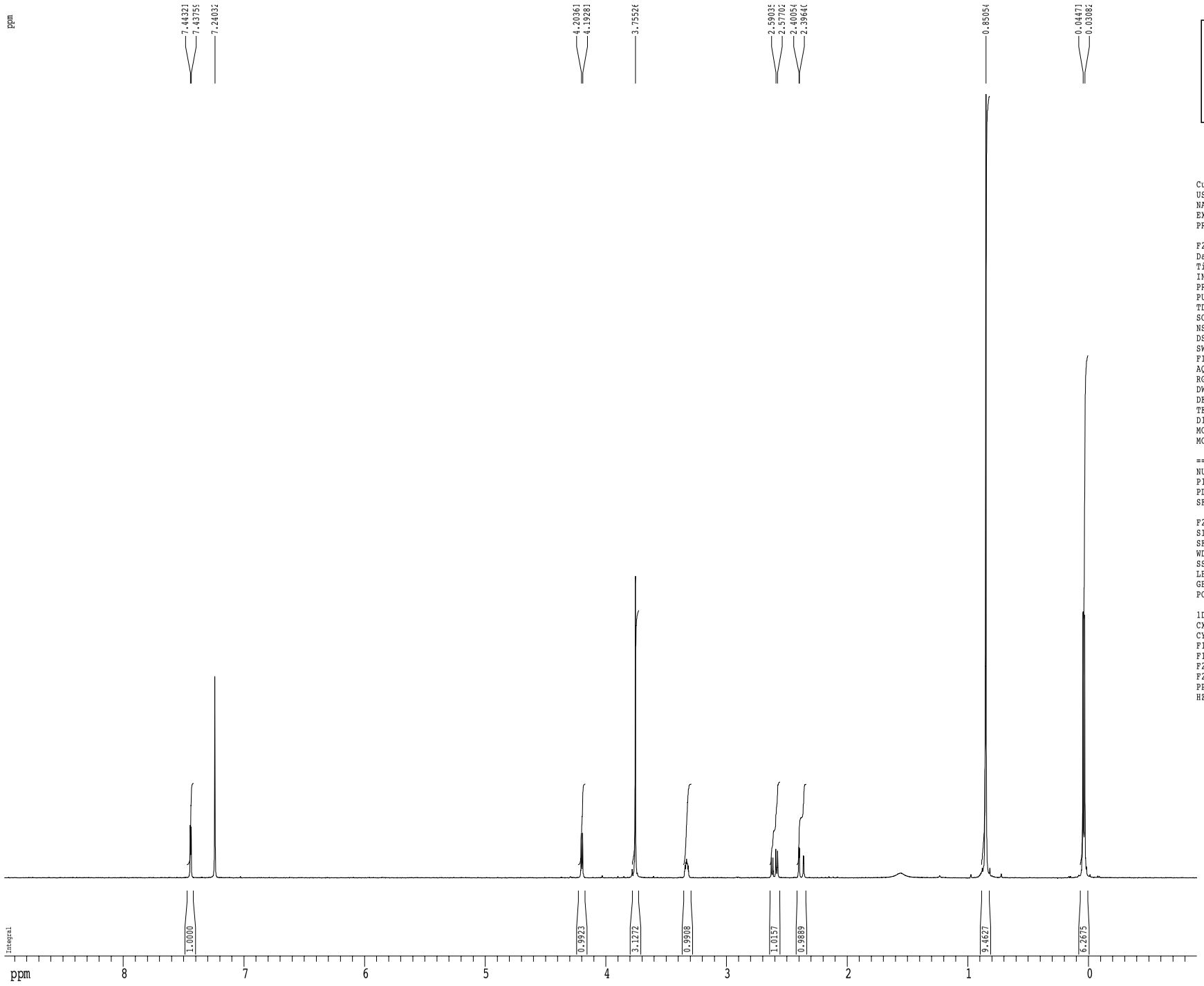
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

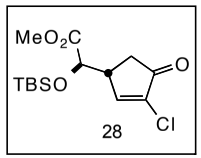
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== 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 125.7803955 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 230.637 ppm
F1 29009.67 Hz
F2P -10.287 ppm
F2 -1293.96 Hz
PPMCM 10.56688 ppm/cm
HZCM 1329.10681 Hz/cm





Current Data Parameters
USER schner
NAME mjs4-217-3c13
EXPNO 1
PROCNO 1

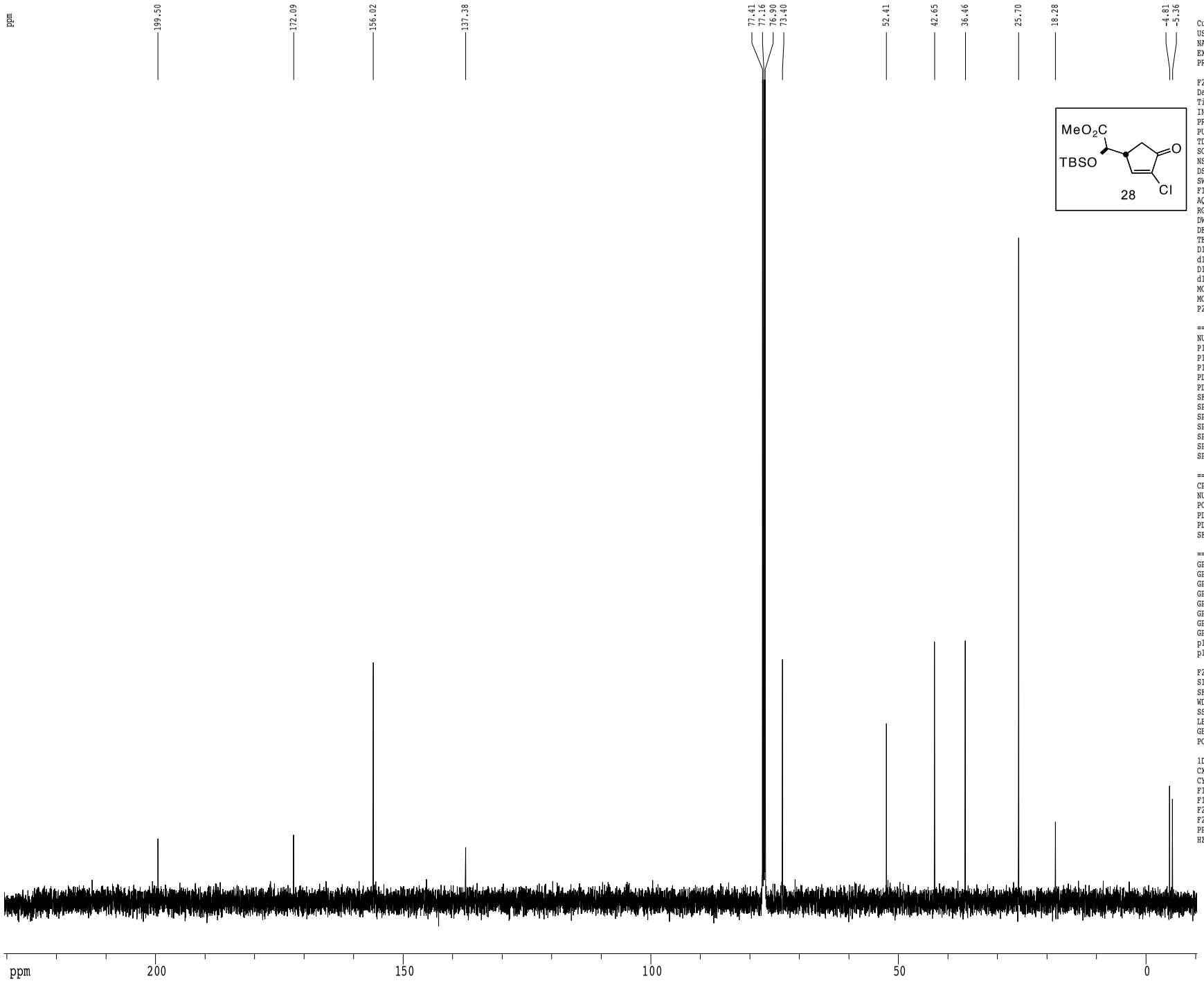
F2 - Acquisition Parameters
Date_ 20120523
Time 8.10
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 7
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200417 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 8.983 ppm
F1 4493.43 Hz
F2P -0.896 ppm
F2 -448.14 Hz
PPMCM 0.43328 ppm/cm
HZCM 216.73543 Hz/cm

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



Current Data Parameters
USER schner
NAME mjs4-217-3c13
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120523
Time 8.13
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 418
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

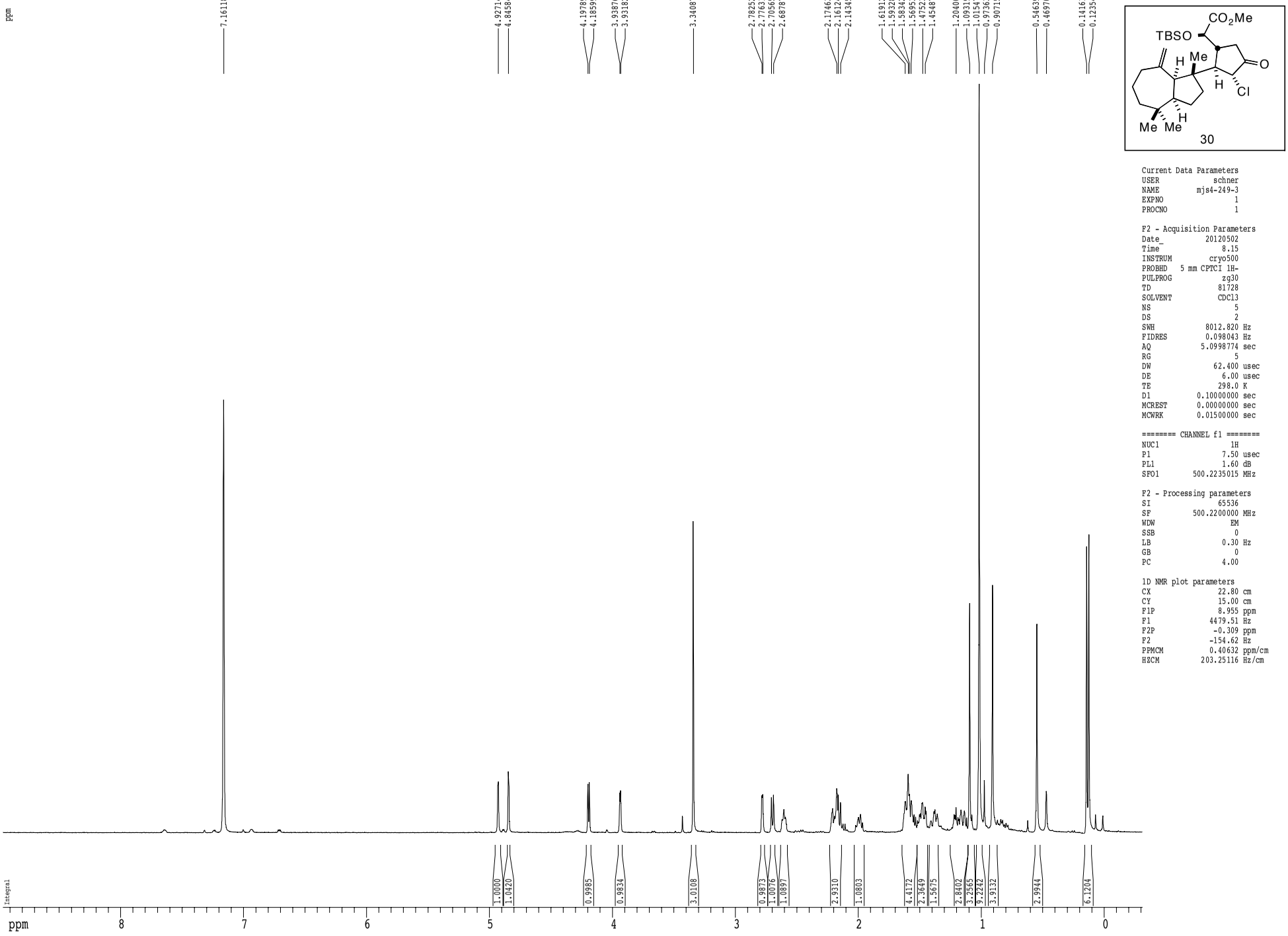
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.20 dB
SP2 3.20 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

======== 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 125.7804080 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 30.00 cm
F1P 230.637 ppm
F1 29009.68 Hz
F2P -10.287 ppm
F2 -1293.96 Hz
PPMCM 10.56688 ppm/cm
HZCM 1329.10693 Hz/cm



30

Current Data Parameters
USER schner
NAME mjs4-249-3
EXPNO 1
PROCNO 1

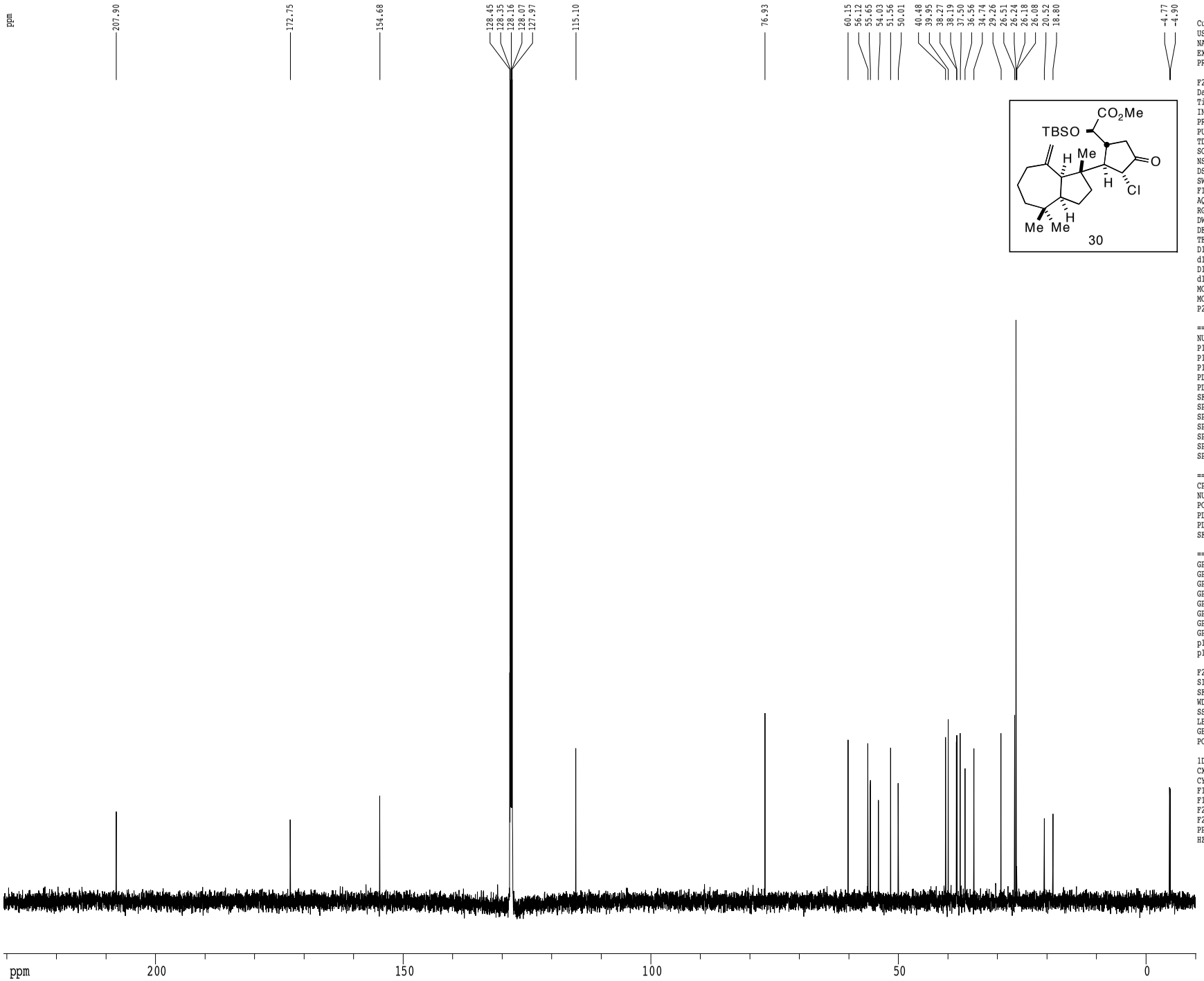
F2 - Acquisition Parameters
Date_ 20120502
Time 8.15
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 5
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

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

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.955 ppm
F1 4479.51 Hz
F2P -0.309 ppm
F2 -154.62 Hz
PPMCM 0.40632 ppm/cm
HZCM 203.25116 Hz/cm

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



Current Data Parameters

USER	schner
NAME	mjs4-249-3
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20120502
Time	8.17
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	SpinEchopg30gp.prd
TD	65536
SOLVENT	CDCl3
NS	69
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	125.7803622 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	80.00 cm
F1P	230.637 ppm
F1	29009.67 Hz
F2P	-10.287 ppm
F2	-1293.96 Hz
PPMCM	10.56688 ppm/cm
HZCM	1329.10645 Hz/cm