Supporting Information

Site-Selective Aliphatic C–H Chlorination Using *N*-Chloroamides Enables a Synthesis of Chlorolissoclimide

Ryan K. Quinn,[b] Zef A. Könst,[a] Sharon E. Michalak,[a] Yvonne Schmidt,[a] Anne R. Szklarski,[a] Sangkil Nam,[c] David A. Horne,[c] Christopher D. Vanderwal,*[a] Erik J. Alexanian*[b]

[a]Department of Chemistry, University of California, Irvine, 1102 Natural Sciences II, Irvine, California 92697-2025, United States. [b]Department of Chemistry, The University of North Carolina, Chapel Hill, NC, 27599. [c]Department of Molecular Medicine, Beckman Research Institute, City of Hope Comprehensive Cancer Center, 1500 East Duarte Road, Duarte, CA 91010.

cvd@uci.edu

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General Methods

All reactions were performed in oven-dried (120 °C) or flame-dried glassware under an atmosphere of dry argon unless otherwise noted. Reaction solvents including dichloromethane (CH_2Cl_2, Fisher, HPLC Grade), hexanes (Fisher, HPLC Grade), diethyl ether (Et_2O, Fisher, BHT stabilized, HPLC Grade), benzene (C_6H_6, Fisher, HPLC Grade), tetrahydrofuran (THF, Fisher, HPLC Grade), and toluene ($PhCH_3$, Fisher, HPLC Grade) were dried by percolation through a column packed with neutral alumina and a column packed with Q5 reactant, a supported copper catalyst for scavenging oxygen, under a positive pressure of argon.

Solvents for workup and chromatography were: acetone (Fisher, ACS grade), hexanes (Fisher or EMD, ACS Grade), EtOAc (EtOAc, Fisher, ACS Grade), dichloromethane (CH_2Cl_2, Fisher, ACS Grade), and methanol (MeOH, Fisher, ACS Grade). Column chromatography was performed using EMD Millipore 60 Å (0.040–0.063 mm) mesh silica gel (SiO_2). Analytical thin-layer chromatography was performed on Merck silica gel 60 F254 TLC plates. Visualization was accomplished with UV (254 or 210 nm), and *p*-anisaldehyde, or ceric ammonium molybdate and heat as developing agents.

Chloroform-*d* ($CDCl_3$, D 99.8%, DLM-7) and methanol-*d*$_4$ (CD_3OD, D 99.8%, DLM-24) and dichloromethane-*d*$_2$ (CD_2Cl_2, D 99.9%, DLM-23) were purchased from Cambridge Isotope Laboratories. Citric acid (ACS grade, anhydrous, Fisher), K_2CO_3 (anhydrous, 99%, Alfa Aesar), $NaHCO_3$ (ACS grade, Fisher), NaOH (ACS grade, Macron or Fisher), $Na_2S_2O_3$ (ACS grade, Fisher), *p*-anisaldehyde (99%, Acros Organics), trimethylaluminum ($Al(CH_3)_3$, Sure Pack™reagent grade, Sigma Aldrich), benzoyl peroxide

(BPO, reagent grade, Sigma Aldrich), *tert*-butylhydroperoxide (TBHP, 5.5 M in decanes, Sigma Aldrich), di-*iso*-butylaluminum hydride (*i*Bu_2AlH, Sure Pack™reagent grade, Sigma Aldrich), ammonia (2.0 M in methanol, Acros Organics), selenium (IV) oxide (SeO_2, 99.8%, Acros Organics), dimthyl sulfoxide (DMSO, extra dry with molecular sieves, water <50 ppm, Acros Organics) were used without further purification.

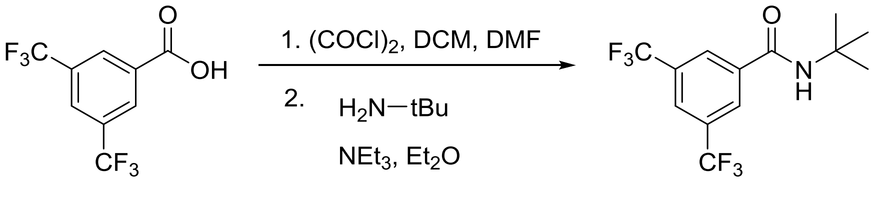
The following reagents were distilled from the indicated drying agents under argon prior to use: triethylamine (Et_3N, EMD, CaH_2), pyridine (Alfa Aesar, CaH_2), 2,4,6-collidine (Alfa Aesar, CaH_2), and *N*,*N*-diisopropylethylamine (*i*-Pr_2NEt, Alfa Aesar, CaH_2). Oxalyl chloride ($(COCl)_2$, Sigma Aldrich) trimethylsilyl triflate (TMSOTf, Alfa Aesar), dibutylboron triflate (*n*Bu_2BOTf, Acros Organics), and thionyl chloride ($SOCl_2$, Sigma Aldrich), were fractionally distilled prior to use.

Infrared (IR) spectra were obtained using a Jasco 260 Plus Fourier transform infrared spectrometer. GC Spectra were obtained using a Shimadzu GC-2010 gas chromatograph with a Shimadzu AOC-20s Autosampler, and Shimadzu SHRXI-5MS GC column. The results of the kinetic isotope study were analyzed using an Agilent Gas Chromatograph- Mass Spectrometer with a 6850 series GC system and a 5973 Network Mass Selective Detector. Proton and carbon magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded at 298K on a Bruker GN500 (500 MHz, ^{1}H; 125 MHz, ^{13}C), Bruker CRYO500 (500 MHz, ^{1}H; 125 MHz, ^{13}C), Bruker AVANCE600 (600 MHz, ^{1}H), Bruker model DRX 400, DRX 500, or a Bruker AVANCE III 600 CryoProbe (^{1}H NMR at 400, 500 or 600 MHz and ^{13}C NMR at 100, 126 or 151 MHz) spectrometer with solvent resonance as the internal standard (^{1}H NMR: $CHCl_3$ at 7.28* ppm, C_6D_6 at 7.16 ppm, CDH_2SOCD_3 at 2.49 ppm, $CDHCl_2$ at 5.32 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm). ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, ddd = doublet of doublet of doublets, td = triplet of doublets, tdd = triplet of doublet of doublets, qd = quartet of doublets, m = multiplet, br. s. = broad singlet), coupling constants (Hz), and integration. Mass spectra for the methods development were obtained using a Thermo LTqFT mass spectrometer with electrospray introduction and external calibration. Mass spectrometry data for the synthesis were obtained from the University of California, Irvine Mass Spectrometry Facility. High-resolution mass spectra (HRMS) were recorded on a Waters LCT Premier spectrometer using ESI-TOF (electrospray ionization-time of flight) and data are reported in the form of (*m/z*). Melting points (mp) were recorded on a Laboratory Devices Mel-Temp II melting point apparatus and are uncorrected. Thin layer chromatography (TLC) was performed on SiliaPlate 250µm thick silica gel plates provided by Silicycle. Visualization was accomplished with short wave UV light (254 nm), aqueous basic potassium permanganate solution, or ethanolic acidic *p*-anisaldehyde solution followed by heating. Flash chromatography was performed using SiliaFlash P60 silica gel (40-63 µm) purchased from Silicycle.

*$CHCl_3$ at 7.26 ppm for compounds S8-S16 and 39-46.

Compound Preparation

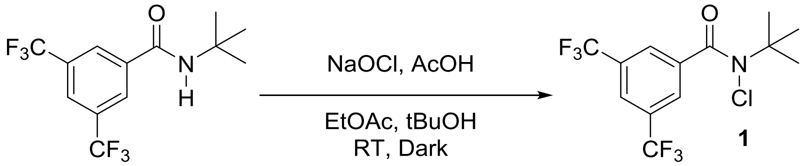
Amide Synthesis



***N*-(tert-butyl)-3,5-bis(trifluoromethyl)benzamide** was synthesized by reacting tert-butylamine with the corresponding acid chloride in THF. To a 0 ºC solution of 10.0g carboxylic acid (38.75 mmol) in DCM (150 mL) and DMF (100 uL) was added 6.5 mL oxalyl chloride (71.50 mmol) dropwise under an argon atmosphere. The solution was stirred at 0 ºC for 15 min. then warmed to room temperature for 1.5 hours.

The resultant solution was evaporated almost to dryness under reduced pressure to remove the DCM. The reaction mixture was dissolved in dry THF and cooled to 0 °C. Then 10.1 mL of tertbutylamine (71.5 mmol) was added dropwise. The solution was allowed to warm to room temperature and stirred overnight. The reaction was diluted with Et_2O, washed with a 2.5M NaOH solution, 3x 1M HCl solution, 1x Brine, dried with magnesium sulfate and concentrated under reduced pressure. The product was purified via single solvent recrystallization from benzene to give the product (11.1 g, 35.5 mmol, 92% yield) as a white crystalline solid. Physical and spectral data were in accordance with literature data.[1]

N-Chlorination with Sodium Hypochlorite



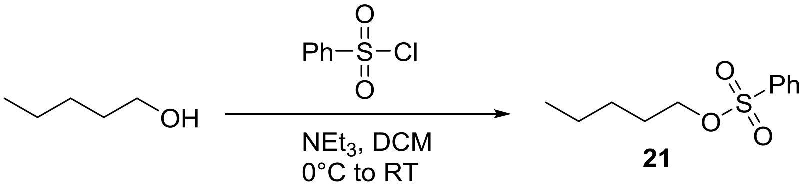
1

General Procedure: To a 250 mL foil wrapped flask under N_2, amide (3.50 g, 11.2 mmol) was added and dissolved in a mixture of ethyl acetate (20 mL) and *tert*-butanol (0.85 mL, 11.2 mmol). 50 mL of a sodium hypochlorite solution was then added (~1.5M in H_2O Sigma-Aldrich), followed by acetic acid (6.4 mL, 112 mmol). The reaction was stirred at room temperature for 3-4 hours. When the reaction was complete as judged by TLC analysis (3 hours usually sufficient) the reaction was diluted with Et_2O, washed three times with saturated sodium bicarbonate solution, once with water, and once with brine. The organic layer was dried with magnesium sulfate and concentrated under reduced pressure. The crude material usually contains traces of chlorinated ethyl acetate. The crude product was purified via column chromatography (1-5% EtOAc/Hexane) to give the choloroamide product (3.8g, 10.9 mmol, 98% yield) as a clear oil. *General storage: N-Chloro reagents were stored in foil-wrapped vials in the freezer when not in use. The reagents can be weighed out on the bench top without risk of decomposition.*

Analytical data for **Chloroamide** : **^{1}H NMR** ^{1}H NMR (600MHz ,CHLOROFORM-d) δ = 8.09 (s, 2 H), 7.97 (s, 1 H), 1.62 (s, 9 H) ppm; **^{13}C NMR** (CHLOROFORM-*d*, 125 MHz) 172.2, 138.3, 131.9, 131.7, 131.5, 131.3, 128.6, 125.6, 124.4, 124.3, 124.2, 124.1, 123.8, 122.0, 120.2, 64.7, 27.9 ppm; **IR** (thin film, cm^{-1}) 2985.3, 2939.9, 1459.9, 1385.6, 1369.2, 1182.2, 1139.7, 906.4, 702.9, 681.7; **HRMS** (ESI) Calcd. for $[C_{13}H_{12}NClF_6O_3+Na]^+$ = 370.04, Found = 370.04.

Substrate Synthesis

Note: Hydrocarbon substrates were obtained commercially and used without further purification unless otherwise noted.



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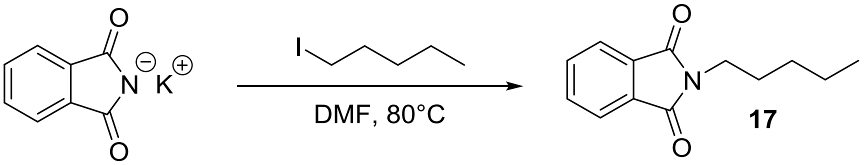
Benzenesulfonyl Pentanol (21). To a 0° solution of pentanol (2.46 mL, 22.7 mmol) and trimethylamine (4.16 mL, 29.49 mmol) in DCM (25 mL) benzenesulfonyl chloride was added dropwise. The reaction was allowed to warm to room temperature and stir overnight. The reaction was diluted with Et$_2$O (100 mL) and the organic layer was washed twice with 100 mL of 1N HCL solution, and once with brine. The organic layer was then dried with magnesium sulfate and concentrated under reduced pressure. The crude material was purified using column chromatography (5-10% EtOAc/Hexanes) to isolate benzenesulfonyl pentanol (4.77 g, 20.9 mmol, 92% yield) as a clear liquid.

Analytical data for **Benzensulfonyl Pentanol:** **^{1}H NMR** (600MHz ,CHLOROFORM-d) δ = 7.92 (d, *J* = 8.3 Hz, 2 H), 7.67 (s, 1 H), 7.61 - 7.53 (m, 2 H), 4.05 (t, *J* = 6.6 Hz, 2 H), 1.69 - 1.59 (m, 2 H), 1.33 - 1.20 (m, 4 H), 0.85 (t, *J* = 7.2 Hz, 3 H); **^{13}C NMR** (125MHz ,CHLOROFORM-d) δ = 136.2, 133.7, 129.2, 127.8, 70.9, 28.5, 27.4, 22.0, 13.8 ppm; **IR** (thin film, cm^{-1}) 2959.2, 2934.1, 2870.5, 1449.2, 1360.3,1186.9, 1097.3, 958.4, 914.1, 826.4, 755.9, 590.1; **HRMS** (ESI) Calcd. for $[C_{11}H_{16}SO_3+Na]^+$ = 251.07, Found = 251.07.



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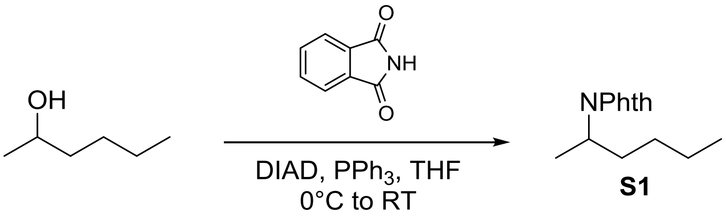
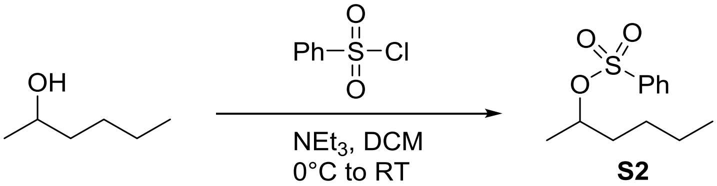
***N*-Pentyl Phthalimide (17):** was synthesized via an alkylation reaction of iodopentane with phthalimide potassium salt: To a room temperature solution of iodopentane (2.5 mL, 19.14 mmol) in DMF (200 mL), the phthalimide salt (7.09 g, 38.3 mmol) was added. The reaction was headed to 80 °C overnight. The reaction was cooled to room temperature and diluted with Et$_2$O and water. The organic layer was separated and washed 8 times with 100 mL of water to remove the residual phthalimide salt. The organic layer was then dried with magnesium sulfate and concentrated under reduced pressure. The crude material was purified using column chromatography (25% EtOAc/Hexanes) to isolate N-Phth pentane (3.45 g, 15.5 mmol, 82% yield) as a clear, yellowish liquid.

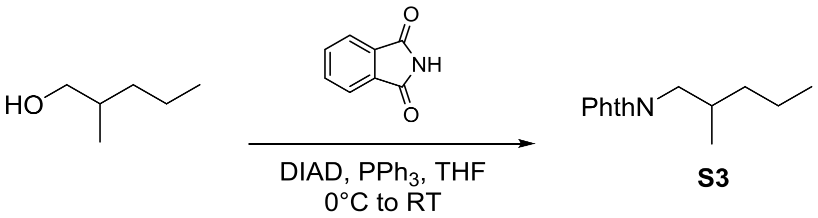
Analytical data for ***N*-Pentyl Phthalimide:** ^{1}**H NMR** (600MHz ,CHLOROFORM-d) δ = 7.88 - 7.75 (m, 1 H), 7.73 - 7.59 (m, 2 H), 3.74 - 3.56 (m, 2 H), 1.75 - 1.57 (m, 2 H), 1.31 (br. s., 4 H), 0.94 - 0.79 (m, 3 H); ^{13}**C NMR** (125MHz ,CHLOROFORM-d) δ = 168.4, 133.8, 132.1, 123.1, 37.9, 28.9, 28.2, 13.9 ppm; **IR** (thin film, cm^{-1}) 3468.4, 3061.4, 2934.2, 1773.2, 1712.5, 1614.13, 1465.6, 1397.2, 1367.3, 1186.0, 1058.7, 980.6880.3, 793.5, 719.3, 620.0, 530.3; **HRMS** (ESI) Calcd. for $[C_{13}H_{15}NO_2+H]^+$ = 218.11, Found = 218.04.



2-Phthalimidyl Hexane (S1). Phthalimide substrate **S1** was prepared via Mitsunobu reaction from the corresponding alcohol. To a 250 mL flame-dried round bottom flask 2-hexanol (1.2 mL, 9.8 mmol) was added and dissolved in THF. Phthalimide (2.16 g, 14.7 mmol) and triphenylphosphine (3.85 g, 14.7 mmol) were then added and the reaction was cooled to 0°C. Diisopropyl azodicarboxylate (2.97 mL, 14.7 mmol) was then added dropwise over 5 min. The reaction was allowed to warm to room temperature and stir overnight. When the reaction was complete by TLC the reaction was concentrated under reduced pressure and purified directly via column chromatography (20% EtOAc/Hexane) to give the product (1.7 g, 7.2 mmol, 73% yield) as clear oil. Physical and spectral data were in accordance with literature data.[2]

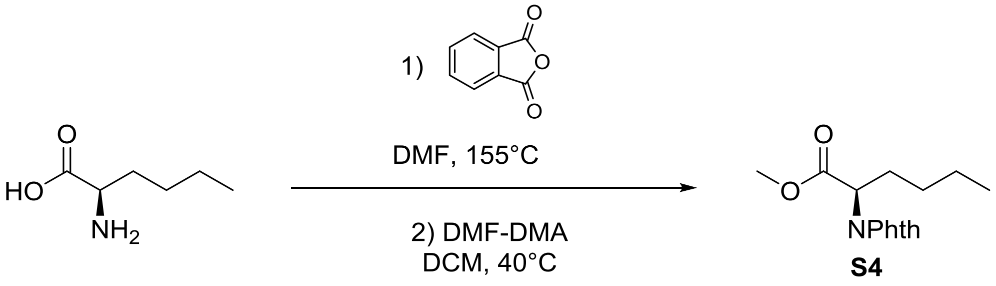


S2. Sulfonate substrate **S2** was prepared via a condensation reaction from the corresponding alcohol in an identical manner as described above for **benzenesulfonyl pentanol.** The crude material was purified using column chromatography (5-10% EtOAc/Hexanes) to isolate **S2** (1.5 g, 6.1 mmol, 94% yield) as a clear yellowish liquid. Physical and spectral data were in accordance with literature data.[3]



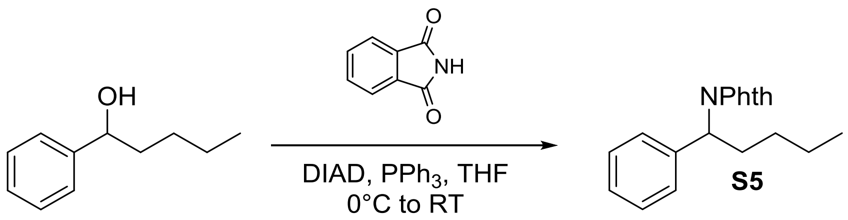
S3. Phthalimide substrate **S3** was prepared via Mitsunobu reaction from the corresponding alcohol in an identical manner as described above for substrate **S3**. The crude reaction mixture was purified directly via column chromatography (20% EtOAc/Hexane) to give the product (1.6 g, 6.9 mmol, 70% yield) as clear oil.

Analytical data for **S3:** ^{1}**H NMR** (600MHz ,CHLOROFORM-d) δ = 7.92 - 7.82 (m, 2 H), 7.77 - 7.70 (m, 2 H), 3.64 - 3.45 (m, 2 H), 2.07 - 1.95 (m, 1 H), 1.51 - 1.40 (m, 1 H), 1.40 - 1.26 (m, 2 H), 1.23 - 1.09 (m, 1 H), 0.95 - 0.86 (m, 6 H); ^{13}**C NMR** (125MHz ,CHLOROFORM-d) δ = 168.7, 133.8, 132.1, 123.2, 44.4, 36.6, 32.4, 19.9, 17.4, 14.3 ppm; **IR** (thin film, cm^{-1}) 2959.2, 2930.3, 1773.3, 1711.5, 1465.6, 1435.7, 1398.1, 1186.97, 1061.6, 723.2; **HRMS** (ESI) Calcd. for $[C_{14}H_{17}NO_2+Na]^+$ = 254.12, Found = 254.12.



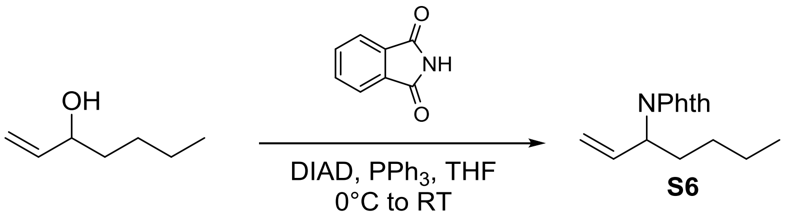
S4 was synthesized via the following two step protocol. To a flame-dried 25 mL flask 1.0 g (7.6 mmol) of DL-norleucene was added, followed by phthalic anhydride (1.7 g, 11.4 mmol). The two solids were then dissolved in DMF (15 mL) and heated to 155° C for 12 hours. The reaction was cooled to room temperature and poured into 50 mL of 1N HCl, and cooled to 0° C for 20 minutes (if precipitation does not occur extract with Et_2O to isolate). The phthalimide product precipitates as a colorless solid which was collected (2. g 7.6 mmol, *quant*) via vacuum filtration and dried under vacuum. The phthalimide was used in the next step without further purification. Phthalimide protected norleucene (2.0 g, 7.6 mmol) was added to a flask fitted with a reflux condenser and dissolved in DCM (25 mL). Dimethylformamide-dimethylacetal (3.1 mL, 22.9 mmol) was then added dropwise and the reaction was refluxed for 4h. When TLC analysis revealed the reaction was complete, the reaction was cooled to room temperature, diluted with Et_2O, washed twice with saturated sodium bicarbonate solution, four times with saturated ammonium chloride solution, and once with brine. The organic layer was dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified via column chromatography (30-40% EtOAc/Hexanes gradient) to give **S4**

(1.98 g, 7.2 mmol, 96% yield) as a yellowish oil. Physical and spectral data were in accordance with literature data.[4]



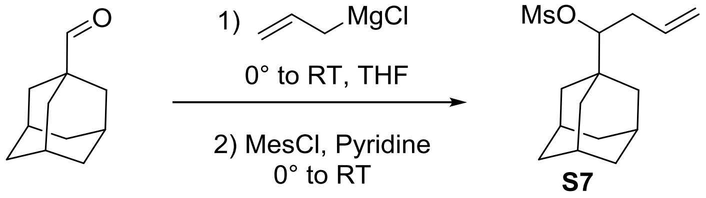
S5. Phthalimide substrate **S5** was prepared via Mitsunobu reaction from the corresponding alcohol in an identical manner as described above for substrate **S1**. The crude reaction mixture was purified directly via column chromatography (25% EtOAc/Hexane) to give the product (2.1 g, 7.1 mmol, 74% yield) as clear oil.

Analytical data for **S5:** ^{1}**H NMR** (600MHz ,CHLOROFORM-d) δ = 7.84 - 7.80 (m, 2 H), 7.72 - 7.68 (m, 2 H), 7.58 (d, *J* = 7.7 Hz, 2 H), 7.35 (t, *J* = 7.7 Hz, 2 H), 7.28 (t, *J* = 1.0 Hz, 1 H), 5.47 - 5.21 (m, 1 H), 2.72 - 2.46 (m, 1 H), 2.36 - 2.25 (m, 1 H), 1.47 - 1.29 (m, 4 H), 0.91 (t, *J* = 7.2 Hz, 3 H); ^{13}**C NMR** (125MHz ,CHLOROFORM-d) δ = 168.4, 139.9, 133.9, 131.9, 128.5, 128.2, 127.8, 123.2, 55.1, 30.7, 29.2, 22.3, 14.0 ppm; **IR** (thin film, cm^{-1}) 3062.4, 3031,5, 2958.3, 2929.3, 2867.6, 1773.2, 1711.5, 1465.6, 1387.5, 1068.9, 880.3, 721.2; **HRMS** (ESI) Calcd. for $[C_{19}H_{19}NO_2+Na]^+$ = 316.13, Found = 316.13.



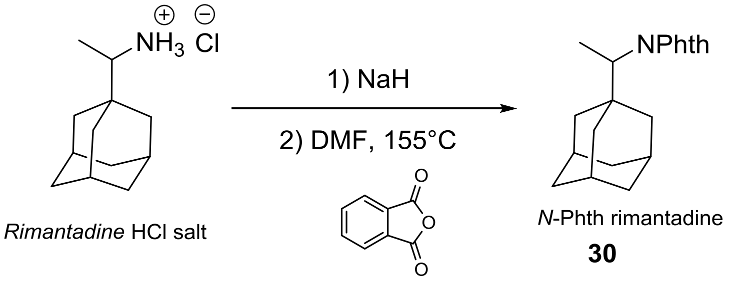
S6. Phthalimide substrate **S6** was prepared via Mitsunobu reaction from the corresponding alcohol in an identical manner as described above for substrate **S1**. The crude reaction mixture was purified directly via column chromatography (20% EtOAc/Hexane) to give the product (1.4 g, 5.7 mmol, 67% yield) as clear oil.

Analytical data for **S6:** ^{1}**H NMR** (600MHz ,CHLOROFORM-d) d = 7.87 - 7.78 (m, 2 H), 7.75 - 7.63 (m, 2 H), 5.71 (dddd, *J* = 5.9, 8.4, 10.1, 17.1 Hz, 1 H), 5.03 (d, *J* = 18.3 Hz, 0 H), 4.94 (d, *J* = 10.3 Hz, 1 H), 4.29 (tt, *J* = 5.2, 10.2 Hz, 1 H), 2.81 (td, *J* = 9.2, 14.2 Hz, 1 H), 2.55 - 2.46 (m, 1 H), 1.75 (tdd, *J* = 5.4, 10.3, 13.8 Hz, 1 H), 1.39 - 1.17 (m, 4 H), 0.86 (t, *J* = 7.3 Hz, 3 H); ^{13}**C NMR** (125MHz ,CHLOROFORM-d) δ = 168.1, 135.9, 133.9, 131.9 123.2, 117.3, 54.2, 31.7, 28.6, 22.2, 13.9 ppm; **IR** (thin film, cm^{-1}) 2961..2, 2931.2, 2874.4, 1465.6, 1450.2, 1360.5, 1186.1, 1097.3, 966.2, 820.6, 756.9, 689.4, 590.1; **HRMS** (ESI) Calcd. for $[C_{15}H_{17}NO_2+Na]^+$ = 266.12, Found = 266.10.



S7 was synthesized via the following two step protocol starting from 1-adamantanecarboxaldehyde (accessed via a protocol reported by Leadbeater[5]). To a 0° solution of aldehyde (3.92 g, 16.2 mmol) in THF (80 mL) allylmagnesiumchloride solution (21 mL, 1.0M in THF) was added dropwise. The reaction was allowed to warm to room temperature and stir overnight. The reaction was quenched with saturated ammonium chloride solution and extracted three times with ether. The combined organic layers were washed with brine, dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified via column chromatography (10% EtOAc/Hexane) to give the product (2.9 g, 14.1 mmol, 87% yield) as colorless solid. The adamantanol (500 mg, 2.4 mmol) was then dissolved in pyridine (8 mL) and cooled to 0° C. Mesyl chloride was then added dropwise and the reaction was allowed to warm to warm to room temperature and stirred for 6 hours. When the reaction was complete judging by TLC analysis the crude reaction mixture was diluted with Et_2O, and washed three times with 1N HCl and once with brine. The organic layer was dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified via column chromatography (10% EtOAc/Hexane) to give the product (655 mg, 2.3 mmol, 96% yield).

Analytical data for **S7: ^{1}H NMR** (600MHz ,CHLOROFORM-d) δ = 5.95 - 5.85 (m, 1 H), 5.23 - 5.12 (m, 2 H), 4.40 (dd, *J* = 2.9, 9.2 Hz, 1 H), 3.03 (s, 3 H), 2.54 (tdd, *J* = 1.4, 5.3, 15.0 Hz, 1 H), 2.44 - 2.35 (m, 1 H), 2.04 (br. s., 3 H), 1.77 - 1.70 (m, 3 H), 1.70 - 1.63 (m, 6 H), 1.59 (dd, *J* = 1.5, 12.1 Hz, 3 H); **^{13}C NMR** (125MHz ,CHLOROFORM-d) δ = 135.4, 118.2, 91.6, 39.3, 38.2, 36.8, 36.7, 33.9, 28.1 ppm; **IR** (thin film, cm^{-1})2906.2, 2851.2, 1642.1, 1451.2, 1333.5, 1171.5, 905.4, 791.4, 732.8, 539.0; **HRMS** (ESI) Calcd. for $[C_{15}H_{24}SO_3+Na]^+$ = 307.13, Found = 307.14.

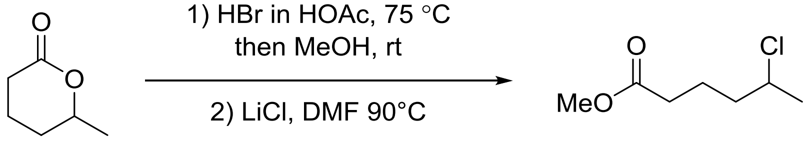


***N*-Phth Rimantadine (30).** The hydrochloride salt (1.44 g, 6.7 mmol) was dissolved in DMF (15 mL) and cooled to 0 °C. Neat sodium hydride (161.3 mg, 6.7 mmol) was then added with gas evolution and the reaction was stirred at 0 °C for 10 min. The reaction was then allowed to warm to room temperature over 30 min. Phthalic Anhydride (1.48 g, 10.4 mmol) was then added and the reaction was heated to reflux overnight. The reaction was then diluted with Et$_2$O and 1N HCl. The organic layer was separated and washed twice more with 1N HCl, and once with brine. The organic layer was then dried with magnesium sulfate and concentrated under reduced pressure. The crude material was purified using column chromatography (10% EtOAc/Hexanes) to isolate *N*-Phth Rimantadine (1.53 g, 4.95 mmol, 74% yield) as a white solid.

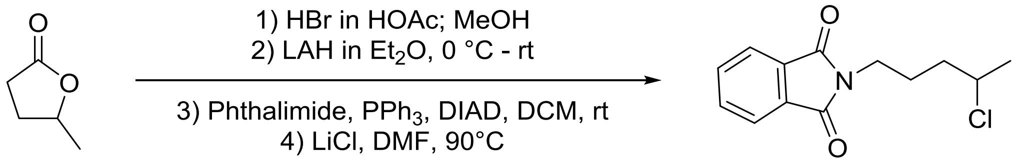
Analytical data for ***N*-Phth Rimantadine (30): ^{1}H NMR** (600MHz ,CHLOROFORM-d) δ = 7.93 - 7.79 (m, 2 H), 7.78 - 7.65 (m, 2 H), 4.05 (q, *J* = 7.3 Hz, 1 H), 1.99 (br. s., 3 H), 1.73 - 1.57 (m, 12 H), 1.51 (d, *J* = 7.3 Hz, 3 H); **^{13}C NMR** (125 MHz ,CHLOROFORM-d) δ = 169.5, 169.4, 133.9, 133.7, 132.3, 131.6, 123.2, 122.9, 56.8, 39.5, 37.8, 36.8, 28.4, 11.8 ppm, **IR** (thin film, cm^{-1}) 2095.3, 2849.3, 2255.3, 1774.2, 1451.2, 1373, 1170.6, 1095.3, 912.2, 726.1, 531.3; **HRMS** (ESI) Calcd. for $[C_{20}H_{23}NO_2+H]^+$ = 332.16, Found = 332.16.

Synthesis of Chloride Standards

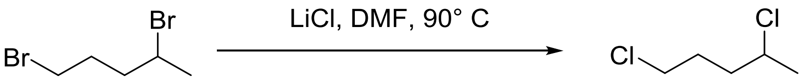
Note: Chloride standards were obtained commercially and used without further purification unless otherwise noted. Commercially obtained chloride include: chlorocyclohexane, 1-chloroadamantane, 2-chloroadamantane, 1-chlorohexane, 2-chlorohexane, 3-chlorohexane.



Methyl 5-chlorohexanoate was synthesized via the following two-step procedure: The first step of the procedure is adapted from the method reported by Wolf et. al.[6] The lactone (2.0 g, 17.5 mmol) was added to a flask containing a solution of 33% HBr in AcOH (5 mL) and fitted with a reflux condenser. The reaction was heated to 75 °C for 4 hours then cooled to room temperature, at which point methanol (8.0 mL) was added and the mixture was stirred at room temperature overnight. The reaction was then partially concentrated under reduced pressure, taken up in EtOAc, washed three times with a saturated aqueous solution of sodium bicarbonate, brine, and the organic layer was dried with magnesium sulfate and concentrated under reduced pressure. The crude product was purified using column chromatography (5% EtOAc/Hexane) to isolate methyl 5-bromohexanoate 72% yield (2.05 g) as a clear liquid. Methyl 5-bromohexanoate (2.0 mL, 9.6 mmol) was then dissolved in DMF (15 mL) and lithium chloride was added in one portion (1.22 g, 28.9 mmol). The reaction was heated to 90° C and stirred overnight. When the reaction was complete as judged my CG-MS analysis, the reaction was diluted with Et_2O, washed three times with 1N HCl, and once with brine. The organic layer was dried over magnesium sulfate and concentrated under reduced pressure. The product required no purification. Physical and spectral data were in accordance with literature data.[7]

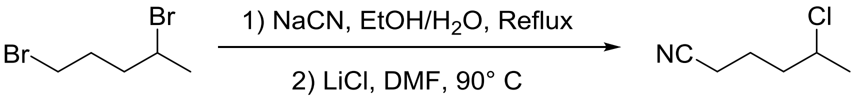


***N*-4-chloropentyl phthalimide** was synthesized via a four-step protocol. *N*-4-bromopentyl phthalimide was previously synthesized in our laboratory, for synthetic details regarding its synthesis see our previous publication. *N*-4-bromopentyl phthalimide (500 mg, 1.6 mmol) was then dissolved in DMF (5 mL) and lithium chloride was added in one portion (214 mg, 5.1 mmol). The reaction was heated to 90° C and stirred overnight. When the reaction was complete as judged my CG-MS analysis, the reaction was diluted with Et_2O, washed three times with 1N HCl, and once with brine. The organic layer was dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified using column chromatography (15% EtOAc/Hexane) to isolate *N*-4-chloropentyl phthalimide (354 mg, 1.4 mmol, 88% yield) as a clear oil. Physical and spectral data were in accordance with literature data.[8]



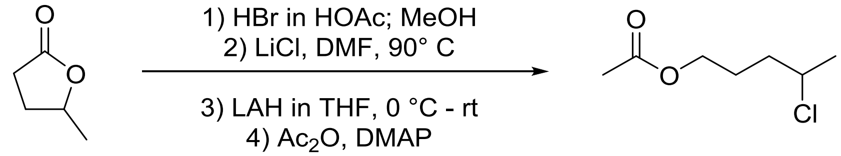
1,4-Dichloropentane was prepared via substitution of the corresponding dibromide. 1,4-dibromopentane (1.0 g, 4.3 mmol) was dissolved in DMF (15 mL) and lithium chloride was added in one portion (1.1 g, 26.2 mmol). The reaction was heated to 90° C and stirred overnight. When the reaction was complete as judged my CG-MS analysis, the reaction was diluted with Et_2O, washed three times with 1N HCl, and once with brine. The organic layer was dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified using column chromatography (2% EtOAc/Hexane) to isolate 1,4-Dichloropentane (467 mg, 3.3 mmol, 77% yield) as a yellowish liquid.

Analytical data for **1,4-Dichloropentane: ^{1}H NMR** ^{1}H NMR (600MHz ,CHLOROFORM-d) δ = 4.12 - 4.01 (m, 1 H), 3.65 - 3.55 (m, 2 H), 2.11 - 2.00 (m, 1 H), 2.00 - 1.87 (m, 2 H), 1.87 - 1.77 (m, 1 H), 1.60 - 1.52 (m, 3 H); **^{13}C NMR** (125 MHz ,CHLOROFORM-d) δ = 57.9, 44.5, 37.4, 29.6, 25.5 ppm; **IR** (thin film, cm^{-1})2956.0, 2929.3, 1725.1, 1445.4, 1379.8, 1287.3, 773.3, 729.9, 652.8, 611.3; **LRMS** Calcd. for $[C_5H_{10}Cl_2]^+$ = 140.02, Found = 140.09



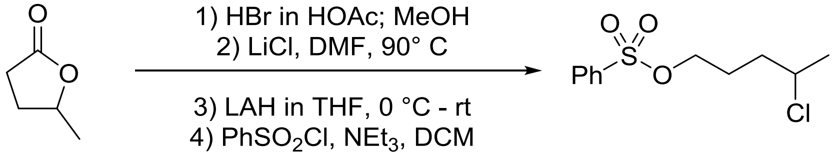
2-Chloro-5-Cyanopentane was prepared via a two-step protocol. 2-Bromo-5-Cyanopentane was previously synthesized in our laboratory, for synthetic details regarding its synthesis see our previous publication.[9] 2-Bromo-5-Cyanopentane (1.8 mL, 10.2 mmol) was then dissolved in DMF (15 mL) and lithium chloride was added in one portion (1.3 g, 30.6 mmol). The reaction was heated to 90° C and stirred overnight. When the reaction was complete as judged my CG-MS analysis, the reaction was diluted with Et_2O, washed three times with 1N HCl, and once with brine. The organic layer was dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified using column chromatography (10% EtOAc/Hexane) to isolate 2-Chloro-5-Cyanopentane (1.1 g, 8.6 mmol, 84% yield) as a yellowish liquid.

Analytical data for **2-Chloro-5-Cyanopentane: ^{1}H NMR** (600MHz ,CHLOROFORM-d) δ = 4.11 - 3.99 (m, 1 H), 2.47 - 2.37 (m, 2 H), 2.00 - 1.87 (m, 2 H), 1.86 - 1.75 (m, 2 H), 1.58 - 1.49 (m, 3 H); **^{13}C NMR** (125 MHz ,CHLOROFORM-d) δ = 119.3, 57.4, 38.8, 25.4, 22.6, 16.8 ppm; **IR** (thin film, cm^{-1}) 2972.2, 2932.3, 2874.4, 2246.6, 1454.1, 1429.9, 1379.8, 1255.4, 1123.3, 612.3; **HRMS** (ESI) Calcd. for $[C_6H_{10}ClN+Na]^+$ = 154.04, Found = 154.04**.**



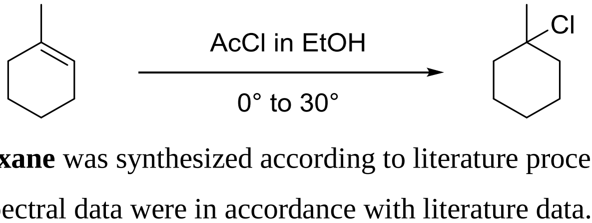
4-Chloropentyl Acetate was synthesized via the following four-step protocol. The first step of the procedure is adapted from the method reported by Wolf et. al.[6] The lactone (2.0 g, 17.5 mmol) was added to a flask containing a solution of 33% HBr in AcOH (5 mL) and fitted with a reflux condenser. The reaction was heated to 75 °C for 4 hours then cooled to room temperature, at which point methanol (8.0 mL) was added and the mixture was stirred at room temperature overnight. The reaction was then partially concentrated under reduced pressure, taken up in EtOAc, washed three times with a saturated aqueous solution of sodium bicarbonate, brine, and the organic layer was dried with magnesium sulfate and concentrated under reduced pressure. The crude product (3.6 g, 17.5, *quant.*) was used in the next step without purification. The crude bromoester (2.0 g, 14.2 mmol) was dissolved in DMF (15 mL) and lithium chloride (1.8 g, 42.5 mmol) was added in one portion. The reaction was heated to 90° C and stirred overnight. When the reaction was complete as judged my CG-MS analysis, the reaction was diluted with Et_2O, washed three times with 1N HCl, and once with brine. The organic layer was dried over magnesium sulfate and concentrated under reduced pressure. The crude chloroester (1.9 g, 13.1 mmol, 92% yield) was used in the next step without purification. The chloroester (2.0 g, 13.8 mmol) was added dropwise to a 0° solution of lithium aluminum hydride (675 mg, 20.7 mmol) in THF (30 mL). The reaction was stirred for 30 min at 0° then allowed to warm to room temperature and stirred for an additional 2 hours. The reaction was cooled back to 0° and quenched slowly by the sequential addition of water (2 mL), NaOH solution (4 mL, 2.5M), and water (10 mL). The reaction was filtered over celite, and concentrated under reduced pressure. The crude reaction mixture was diluted with Et_2O, washed with saturated sodium bicarbonate, and brine, dried with magnesium sulfate and concentrated under reduced pressure. The crude product was purified using column chromatography (15% EtOAc/Hexane) to isolate the chloro-alcohol (1.31g, 10.72 mmol, 78% yield) as a clear liquid. Lastly, the chloro-alcohol (500 mg, 4.1 mmol) was acetylated in in neat acetic anhydride (1 mL) using catalytic DMAP (24 mg, 0.2 mmol). The reaction was complete after stirring overnight at room temperature. The reaction was diluted with Et_2O, washed twice with saturated sodium bicarbonate, once with water, dried over magnesium sulfate and concentrated under reduced pressure. The product (660 mg, 4.0 mmol, 98% yield) did not require purification.

Analytical data for **4-Chloropentyl Acetate: ^{1}H NMR** (600MHz ,CHLOROFORM-d) δ = 4.15 - 4.01 (m, 3 H), 2.06 (s, 3 H), 1.94 - 1.70 (m, 4 H), 1.54 (d, *J* = 6.6 Hz, 3 H); **^{13}C NMR** (125 MHz, CHLOROFORM-d) δ = 171.1, 63.8, 58.2, 36.7, 25.9, 25.4, 20.9 ppm; **IR** (thin film, cm^{-1}) 2967.9, 1740.4, 1446.3, 1366.3, 1240.9, 1038.5, 608.4; **HRMS** (ESI) Calcd. for $[C_7H_{13}ClO_2+Na]^+$ = 187.04, Found = 187.04**.**

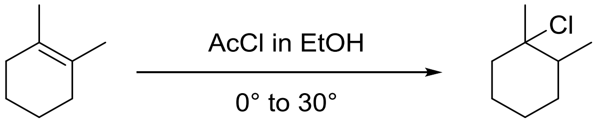


4-Chlorpentyl Benzenesulfonate was synthesized from the same chloroalcohol used to synthesize 4-Chloropentyl Acetate above. The chloroalcohol (500 mg, 4.1 mmol) was dissolved in DCM (8 mL) and triethyl amine (0.74 mL, 5.3 mmol) was added. The reaction was cooled to 0° and benzene sulfonyl chloride (0.57 mL, 4.5 mmol) was added dropwise. The reaction was allowed to warm to room temperature and was stirred overnight. When the reaction was complete as judged via TLC analysis the reaction was diluted with Et_2O, washed twice with 1N HCl and once with brine. The organic layer was dried over magnesium sulfate and concentrated under reduced pressure. The crude product was purified using column chromatography (10% EtOAc/Hexane) to isolate 4-Chlorpentyl Benzenesulfonate (667 mg, 2.5 mmol, 62% yield).

Analytical data for **4-Chlorpentyl Benzenesulfonate: ^{1}H NMR** ^{1}H NMR (600MHz ,CHLOROFORM-d) δ = 8.00 - 7.86 (m, 2 H), 7.74 - 7.64 (m, 8 H), 7.63 - 7.49 (m, 2 H), 4.16 - 4.04 (m, 2 H), 4.02 - 3.91 (m, 1 H), 1.97 - 1.86 (m, 1 H), 1.86 - 1.74 (m, 2 H), 1.74 - 1.63 (m, 1 H), 1.49 (d, *J* = 6.6 Hz, 3 H); **^{13}C NMR** (125 MHz ,CHLOROFORM-d) δ = 135.9, 133.9, 129.3, 127.9, 70.1, 57.8, 35.9, 26.1, 25.4 ppm; **IR** (thin film, cm^{-1}) 3067.2, 2972.7, 1447.3, 139.6, 1186.0, 1097.3, 969.3, 917.5, 826.7, 751.3, 689.4, 589.1; **HRMS** (ESI) Calcd. for $[C_{11}H_{15}ClSO_3+Na]^+$ = 385.03, Found = 385.03**.**



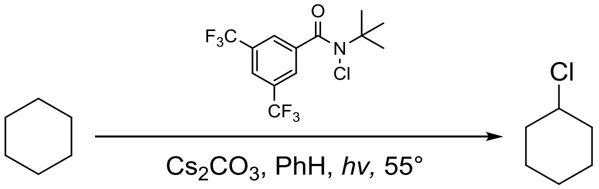
1-Chloro-1-Methylcyclohexane was synthesized according to literature procedures and used without purification. Physical and spectral data were in accordance with literature data.[10]



1-Chloro-1,2-Dimethylcyclohexane was synthesized according to literature procedures referenced above and used without purification. Physical and spectral data were in accordance with literature data. Both NMR and GC analysis show both the *trans* and *cis* products in a 87:13 ratio respectively. The selectivity is consistent with literature precedent.[11]

C-H Chlorination Procedures with N-Chloro Amides

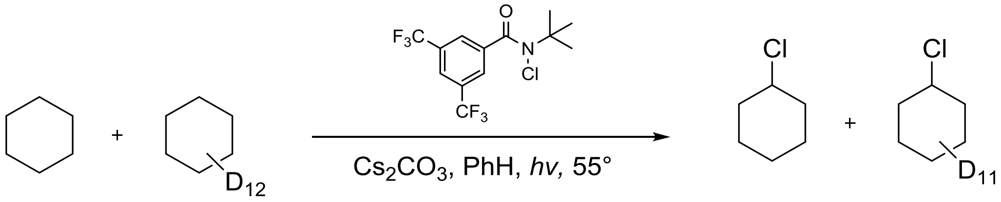
Chlorination of Cycloalkanes (Table 1)



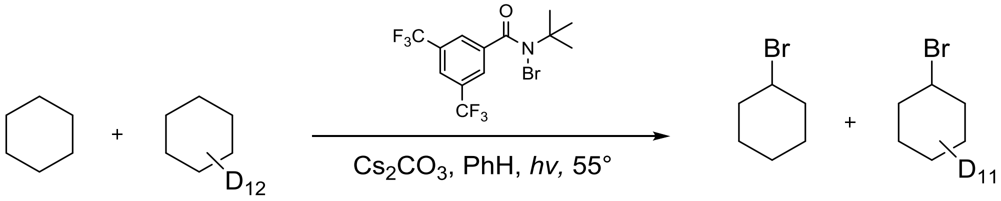
A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (50.0 mg, 0.143 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (280 µL). Cyclohexane (15.5 µL, 0.143 mmol), and cesium carbonate (46.2 mg, 0.143 mmol) were then added. *Note: cycloalkane was added as a stock solution in benzene to improve the reproducibility of the results.* The reaction was then sealed with teflon tape and taken out of the glovebox, and irradiated with two 23W compact fluorescent light bulbs for 90 minutes at 55° C. A white, semi-soluble precipitate forms as the reaction reaches completion. Upon competition, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. 71.6% GC yield of cyclohexylchloride. A small amount of dichloride products (2% GC yield) are also formed in the reaction.

Reactions in Table 1 were performed in an identical fashion to the procedure described above.

KIE Study

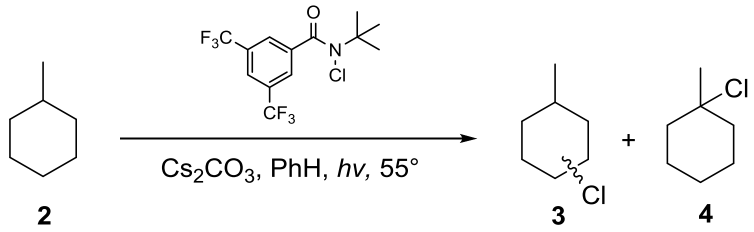


A flame-dried, 1 dram vial was charged with a stir bar and fitted with a PTFE lined screw cap. Chloroamide (20 mg, 0.058 mmol) was added to the vial in the absence of ambient light, and the reaction was taken into a glovebox, and dissolved in 200 uL of dry, freeze-pump-thawed benzene. Cyclohexane (27.6 uL, 0.255 mmol) and Cyclohexane-d_{12} (27.5 uL, 0.255 mmol), and cesium carbonate (18.9 mg, 0.058 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 45 minutes at 55° C. The reaction was then diluted with DCM and analyzed using an Agilent Gas Chromatograph- Mass Spectrometer with a 6850 series GC system and a 5973 Network Mass Selective Detector to determine the ratio of non-deuterated to deuterated product (Ratio = 4.9 = $K_{H/D}$).



A flame-dried, 1 dram vial was charged with a stir bar and fitted with a PTFE lined screw cap. Bromoamide (20 mg, 0.058 mmol) was added to the vial in the absence of ambient light, and the reaction was taken into a glovebox, and dissolved in 200 uL of dry, freeze-pump-thawed benzene. Cyclohexane (27.6 uL, 0.255 mmol) and Cyclohexane-d_{12} (27.5 uL, 0.255 mmol), and cesium carbonate (18.9 mg, 0.058 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 45 minutes at 55° C. The reaction was then diluted with DCM and analyzed using an Agilent Gas Chromatograph- Mass Spectrometer with a 6850 series GC system and a 5973 Network Mass Selective Detector to determine the ratio of non-deuterated to deuterated product (Ratio = 4.9 = $K_{H/D}$).

Chlorination of Methylcyclohexane (Table 2)



Methylcyclohexane: A flame-dried, 1 dram vial was charged with a stir bar and. chloroamide (50.0 mg, 0.143 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (140 μL). Methylcyclohexane (18.2 μL, 0.143 mmol), and cesium carbonate (46.2 mg, 0.143 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 2 hours at 55° C. Upon competition, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. The approximate yield was also verified with NMR analysis using 2,5 dimethylfuran as an internal standard. 74% GC yield of combined bromide products. *The secondary chloride products were assigned via analogy to our C-H bromination chemistry.*

Figure S1.

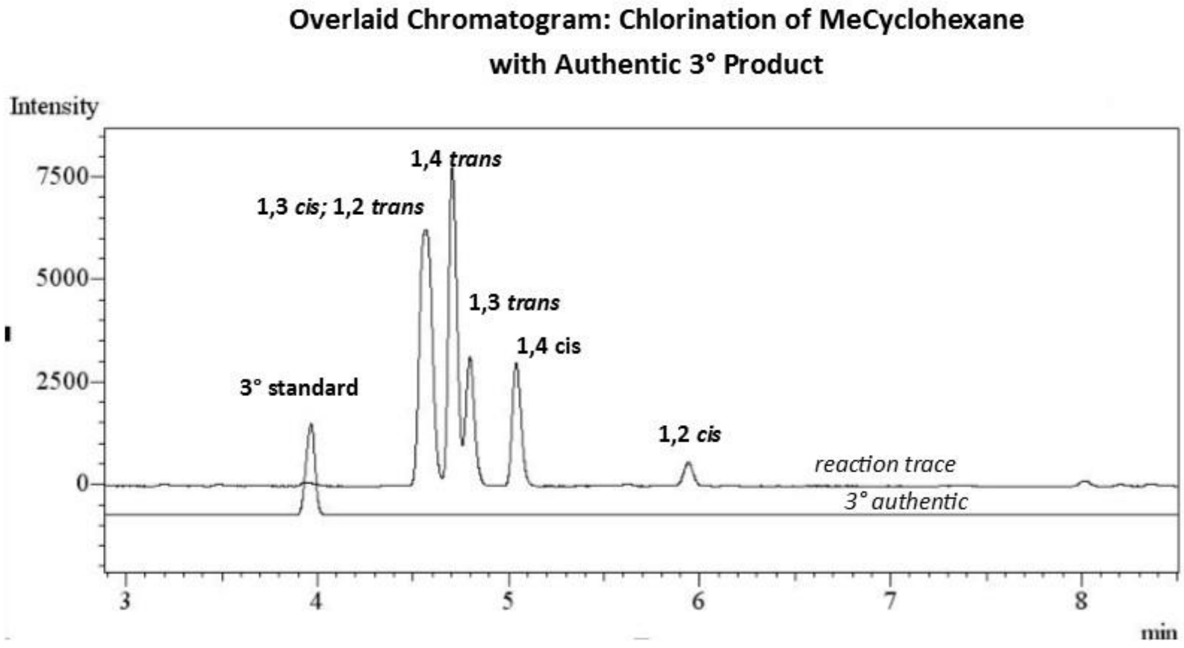
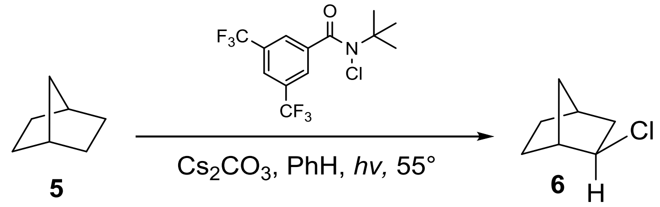


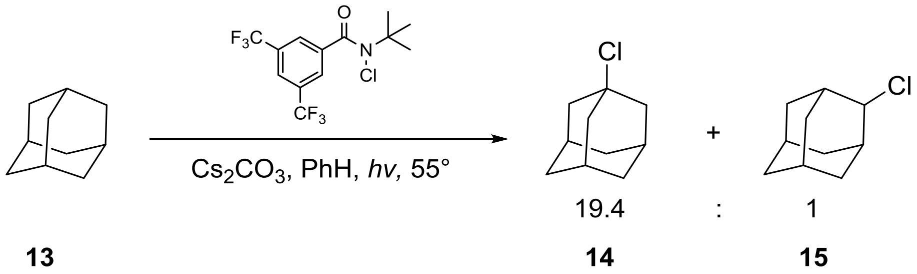
Table S1.

Chlorination of Methylcyclohexane: Selectivity		
Product	Retention Time	Percent Area
3° chloride	4.00	1.51
1,3-*cis;* 1,2-*trans*	4.58	36.19
1,4-*trans*	4.75	31.58
1,3-*trans*	4.81	13.65
1,4-*cis*	5.05	14.65
1,2-*cis*	5.95	Trace

Bromination of Complex, Cyclic Alkanes (Table 2)



Norbornane: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (100.0 mg, 0.29 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (0.29 mL). Norbornane (27.9 mg, 0.29 mmol), and cesium carbonate (94.2 mg, 0.29 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. The reaction gives only the 2-exo product. 53.3% GC yield of 2-*exo*-chloronorbornane. The crude reaction mixture was purified using column chromatography (5% EtOAc/Hexane) to isolate 2-*exo*-chloronorbornane (18.2 mg, 0.14 mmol, 54% yield) as an off-white solid. Physical and spectral data were in accordance with literature data.[12]



Adamantane: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (50.0 mg, 0.143 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (140 μL).

Adamantane (19.5 mg, 0.143 mmol), and cesium carbonate (46.7 mg, 0.143 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 2 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. The reaction gives both 1-and 2-Chloroadamante in a 19 to 1 ratio by GC respectively. 69.3% GC yield of combined chloroadamantanes.

Figure S2.

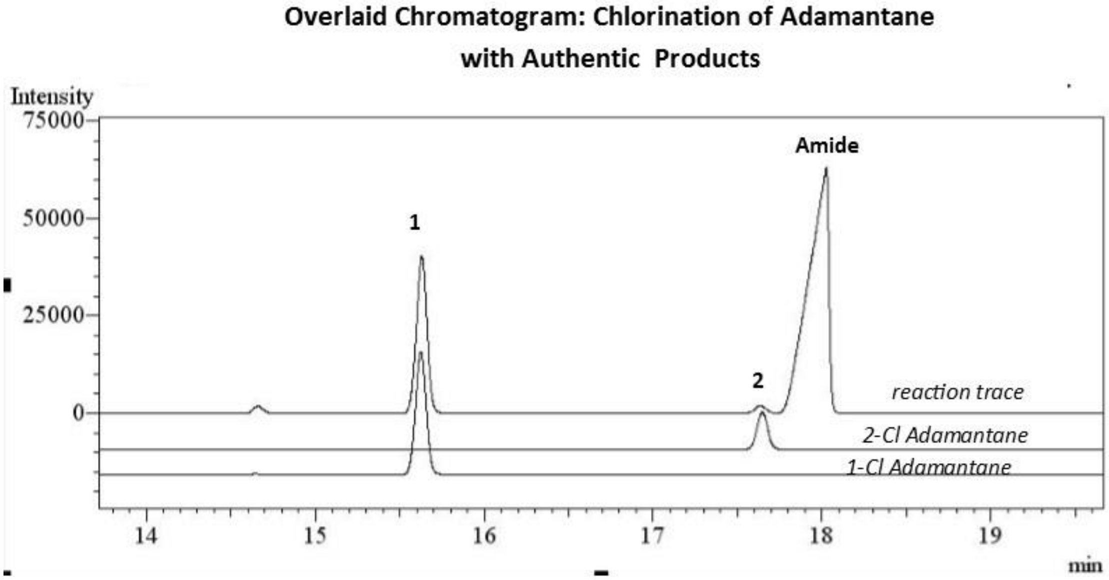
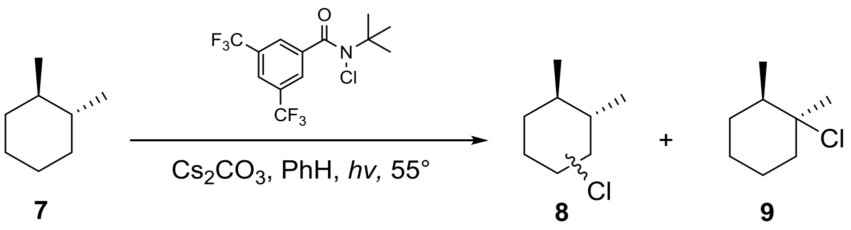


Table S2.

Chlorination of Adamantane: Selectivity		
Product	Retention Time	Percent Area
1-Chloroadamantane	15.63	94.91
2-Chloroadamantane	17.65	5.09

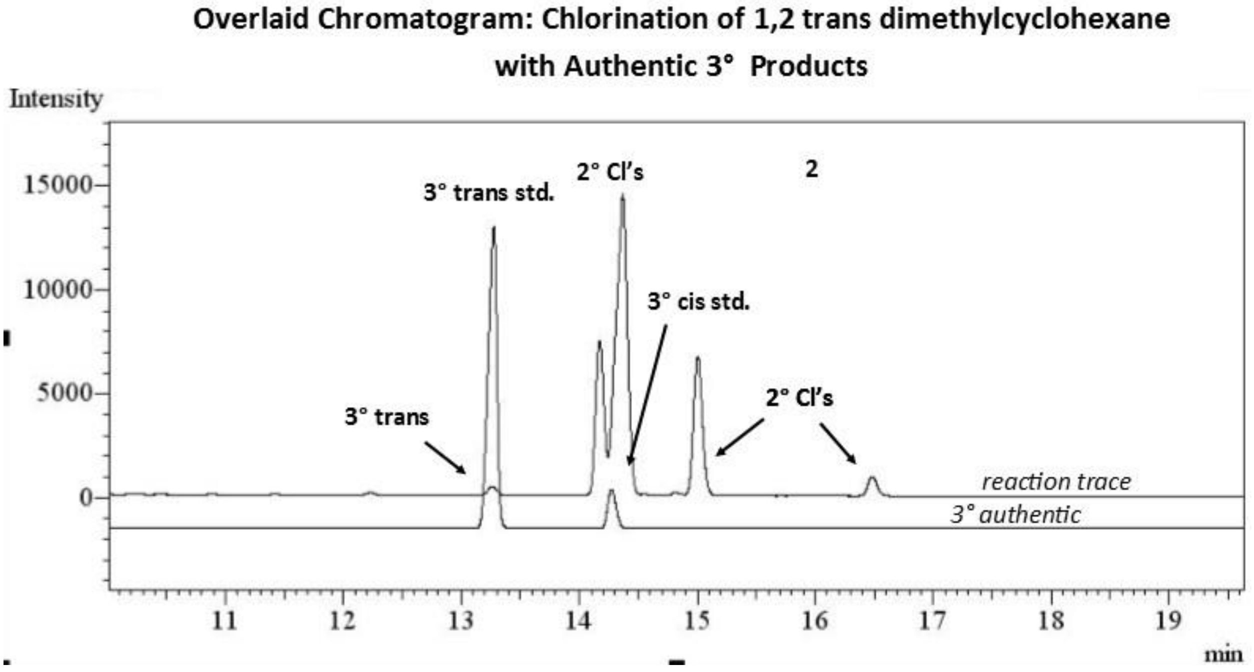


1,2-*trans*-dimethylcyclohexane: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide **1** (50.0 mg, 0.143 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (140 μL). 1,2-*trans*-dimethylcyclohexane (30.5 μL, 0.143 mmol), and cesium carbonate (46.7 mg, 0.058

mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 2 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. Upon completion, the reaction mixture was concentrated under reduced pressure and dissolved in pentanes. The resulting suspension was run through a plug of silica and concentrated a second time. The reaction was analyzed by NMR using 2,5-dimethylfuran as an internal standard to determine the yield of secondary chloride products. GC analysis was used to determine the relative amount and yield, of the tertiary chloride products. 79.0% NMR yield of combined chloride products.

By analogy to our bromination chemistry, none of the cis tertiary product is observed in the reaction trace. Bromination of the trans isomer of starting material gives only 0.9% functionalization at a tertiary position to give only the trans isomer. Additionally, formation of the cis product from the trans isomer of starting material involves a thermodynamically unfavorable trapping step.

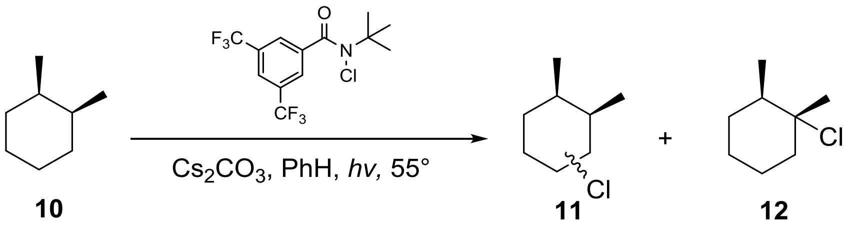
Figure S3.



Overlaid Chromatogram: Chlorination of 1,2 trans dimethylcyclohexane with Authentic 3° Products

Table S3.

Chlorination of 1,2 *trans*-dimethylcyclohexane: Selectivity		
Product	Retention Time	Percent Area
3° trans	13.25	0.91
	14.17	20.32
	14.36	56.71
	15.07	19.41
	16.48	2.63



1,2-*cis*-dimethylcyclohexane: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (50.0 mg, 0.143 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (140 μL). 1,2-*cis*-dimethylcyclohexane (30.5 μL, 0.143 mmol), and cesium carbonate (46.6 mg, 0.143 mmol) were then added.. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 2 hours at 55°. Upon completion, the reaction mixture was concentrated under reduced pressure and dissolved in pentanes. The resulting suspension was run through a plug of silica and concentrated a second time. The reaction was analyzed by NMR using 2,5-dimethylfuran as an internal standard to determine the yield of secondary bromide products. GC analysis was used to determine the relative amount and yield, of the tertiary bromide products. 72.9% NMR yield of combined chloride products.

Figure S4.

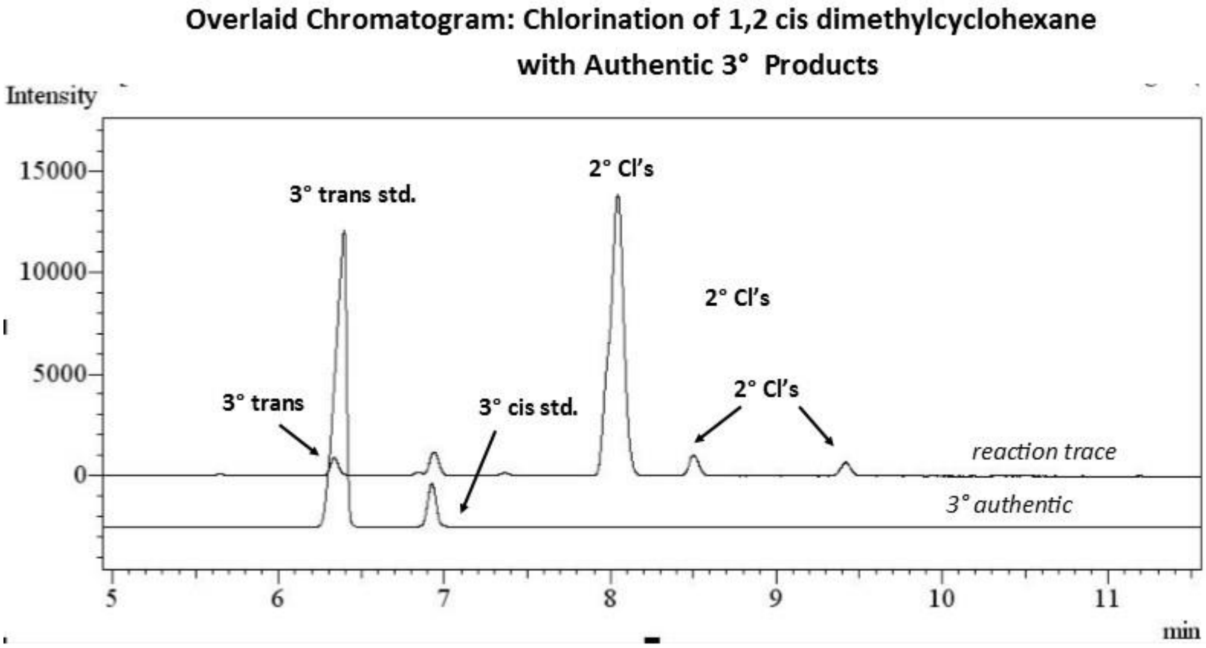
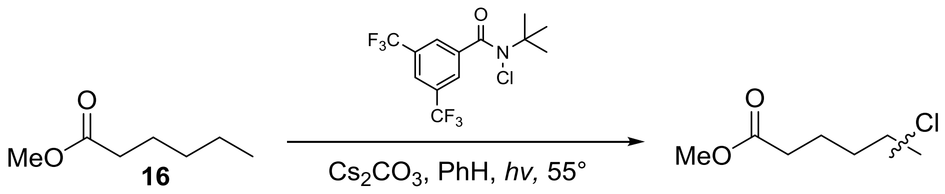


Table S4.

Chlorination of 1,2 *cis*-dimethylcyclohexane: Selectivity		
Product	Retention Time	Percent Area
3° trans	6.34	3.32
3° cis	6.94	4.81
	8.04	85.56
	8.52	4.30
	9.42	2.98

Chlorination of Electron Withdrawing Alkanes (Table 3)



Methyl hexanoate: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (100.0 mg, 0.286 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (560 µL). Methyl hexanoate (21.2 µL, 0.143 mmol), and cesium carbonate (93.2 mg, 0.286 mmol) were then added. *Note: alkane was added as a stock solution in benzene to improve the reproducibility of the results.* The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. 83.2% GC yield of combined chloride products. *Only the major isomer was synthesized independently. The secondary chloride products were assigned via analogy to our C-H bromination chemistry*.

Figure S5.

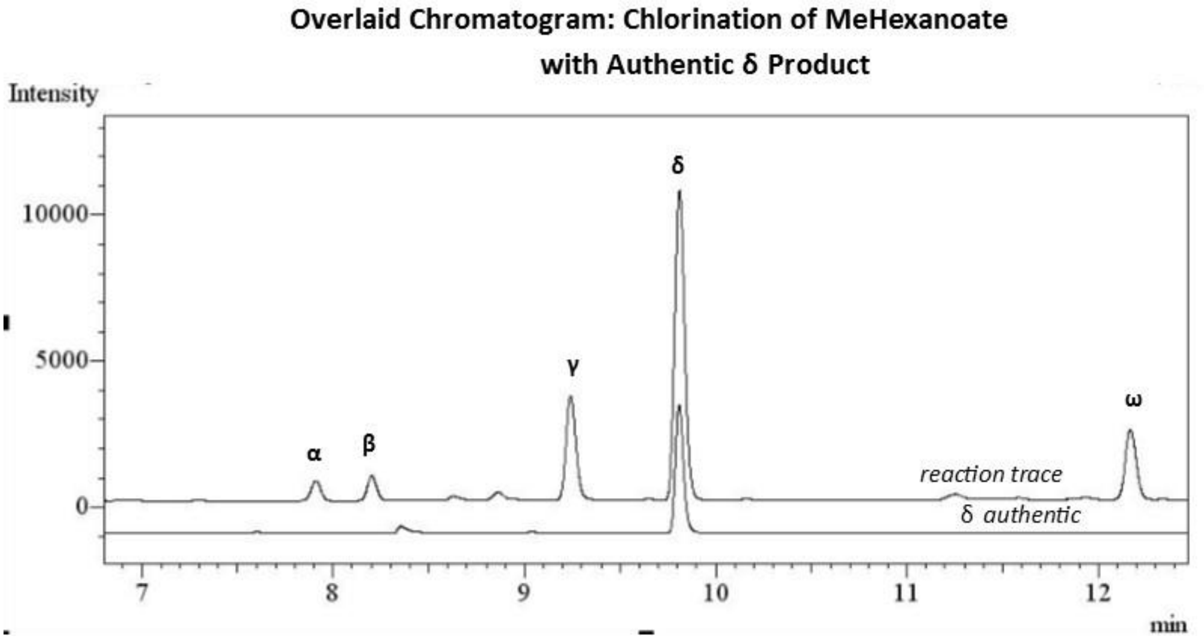
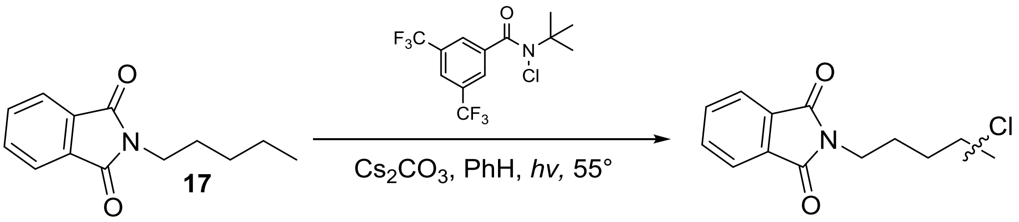


Table S5.

Chlorination of Methylhexanoate: Selectivity		
Product	Retention Time	Percent Area
α	7.91	3.64
β	8.20	4.62
γ	9.24	19.67
δ	9.81	57.63
ω	12.16	14.41

The response factors for all isomers of chloro-Methylhexanoate were calculated to be nearly identical; therefore, the relative percent area of the product peaks reflects the relative amounts of the different isomers formed in the reaction.



N-Penthylphthalimide: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (100.0 mg, 0.286 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (280 μL). *N*-Pentylphthalimide (31.0 mg, 0.143 mmol), and cesium carbonate (93.4 mg, 0.143 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. 79.7% GC yield of combined chloride products.

Only the major isomer was synthesized independently. The secondary chloride products were assigned via analogy to our C-H bromination chemistry.

Figure S6.

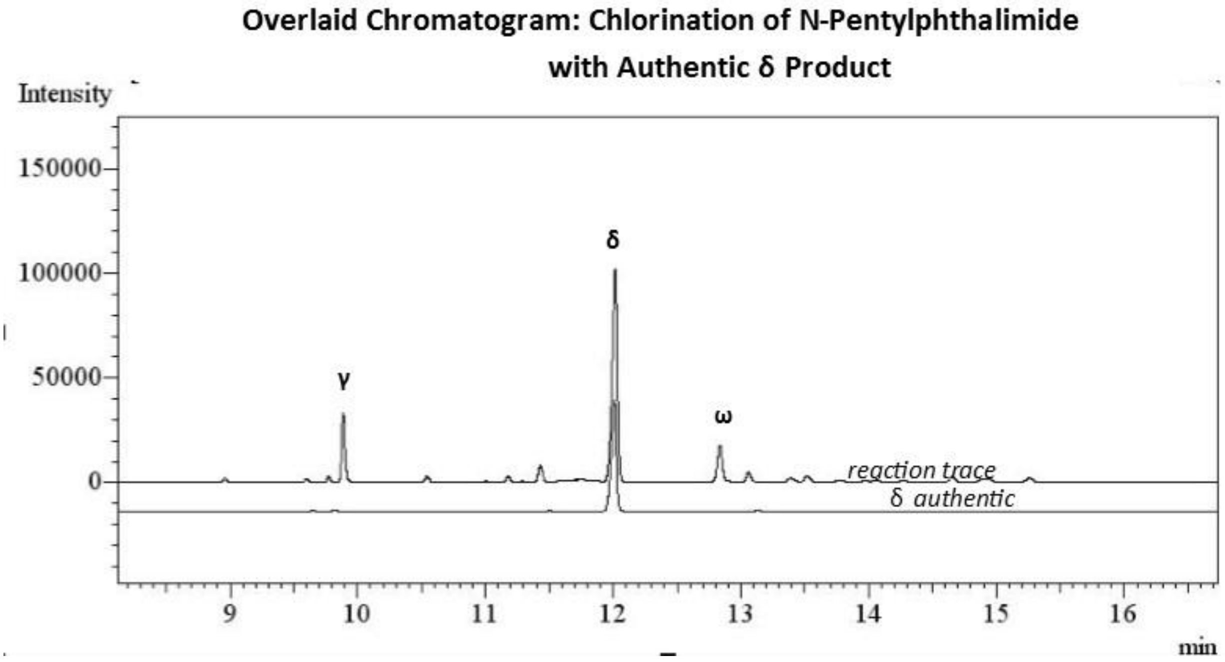
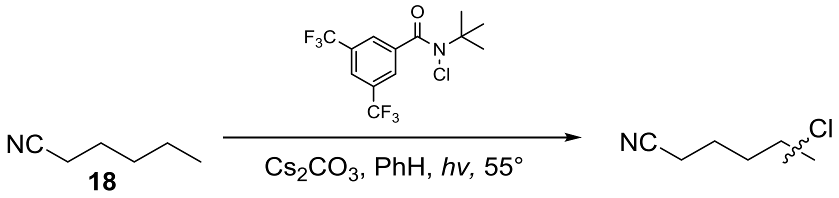


Table S6.

Chlorination of N-Pentylphthalimide: Selectivity		
Product	Retention Time	Percent Area
α	----	----
β	----	----
γ	11.43	4.77
δ	12.02	81.35
ω	12.84	13.87



Hexanenitrile: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (100.0 mg, 0.286 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (280 μL). Hexanenitrile (17.2 μL, 0.143 mmol), and cesium carbonate (93.8 mg, 0.286 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. 78.6% GC yield of combined chloride products.

Only the major isomer was synthesized independently. The secondary chloride products were assigned via analogy to our C-H bromination chemistry.

Figure S7.

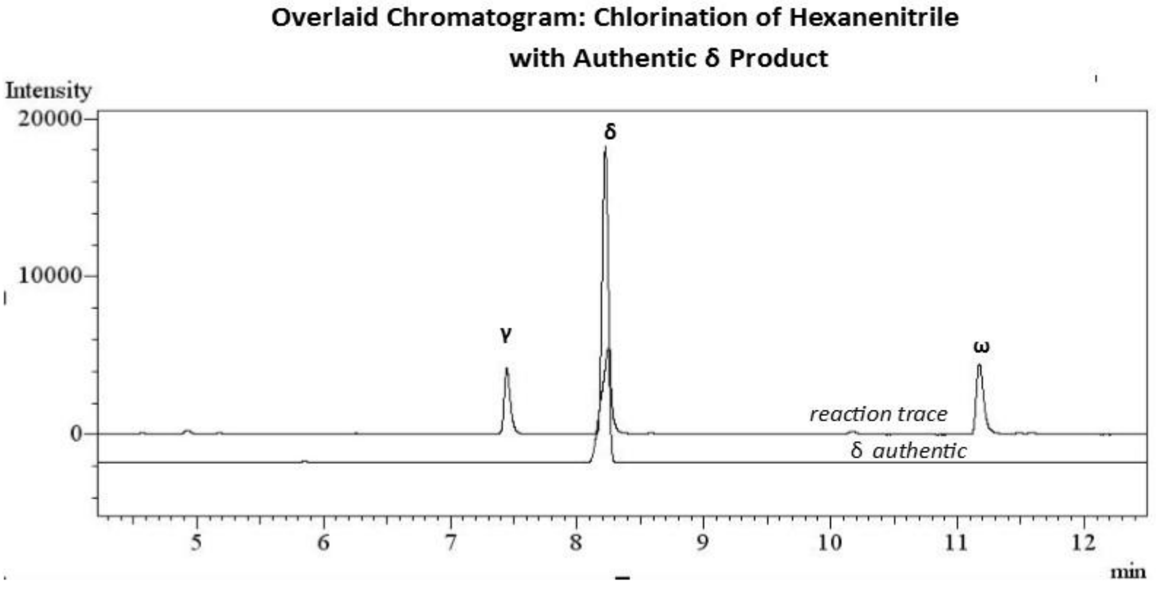
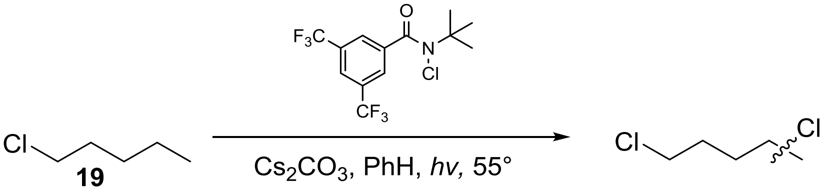

Overlaid Chromatogram: Chlorination of Hexanenitrile with Authentic δ Product

Table S7.

Chlorination of Hexane Nitrile: Selectivity		
Product	Retention Time	Percent Area
α	---	---
β	---	---
γ	11.17	15.32
δ	12.81	73.93
ω	14.66	10.74



1-Chlorohexane: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (100.0 mg, 0.286 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (280 µL). 1-Chlorohexane (17.3 µL, 0.143 mmol), and cesium carbonate (93.0 mg, 0.286 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. 74.0% GC yield of combined chloride products.

Only the major isomer was synthesized independently. The secondary chloride products were assigned via analogy to our C-H bromination chemistry.

Figure S8.

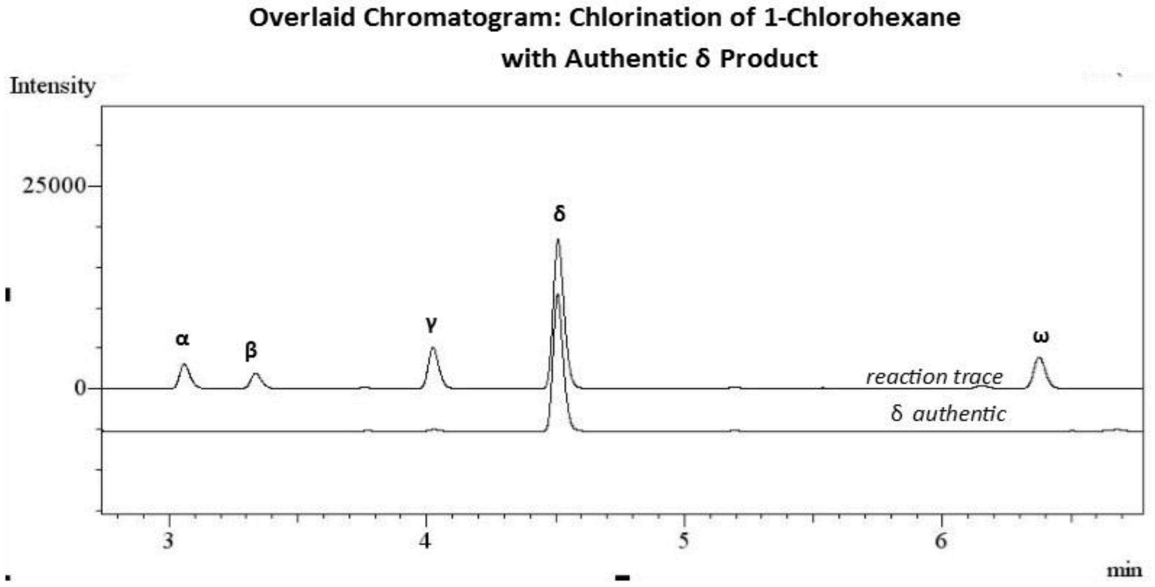
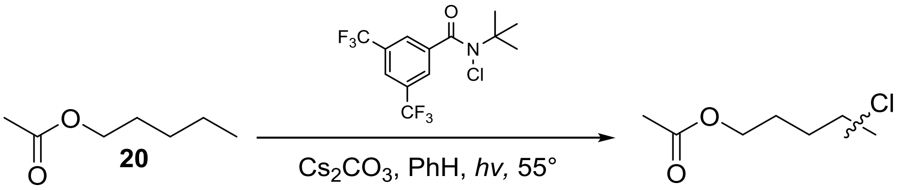


Table S8.

Chlorination of 1-Chlorohexane: Selectivity		
Product	Retention Time	Percent Area
α	3.06	9.03
β	3.37	5.54
γ	4.02	15.29
δ	4.51	57.23
ω	6.37	12.91



Amyl Acetate: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (100.0 mg, 0.286 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (280 μL). Amyl acetate (21.3 μL, 0.143 mmol), and cesium carbonate (93.1 mg, 0.286 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. 89.4% GC yield of combined chloride products.

Only the major isomer was synthesized independently. The secondary chloride products were assigned via analogy to our C-H bromination chemistry.

Figure S9.

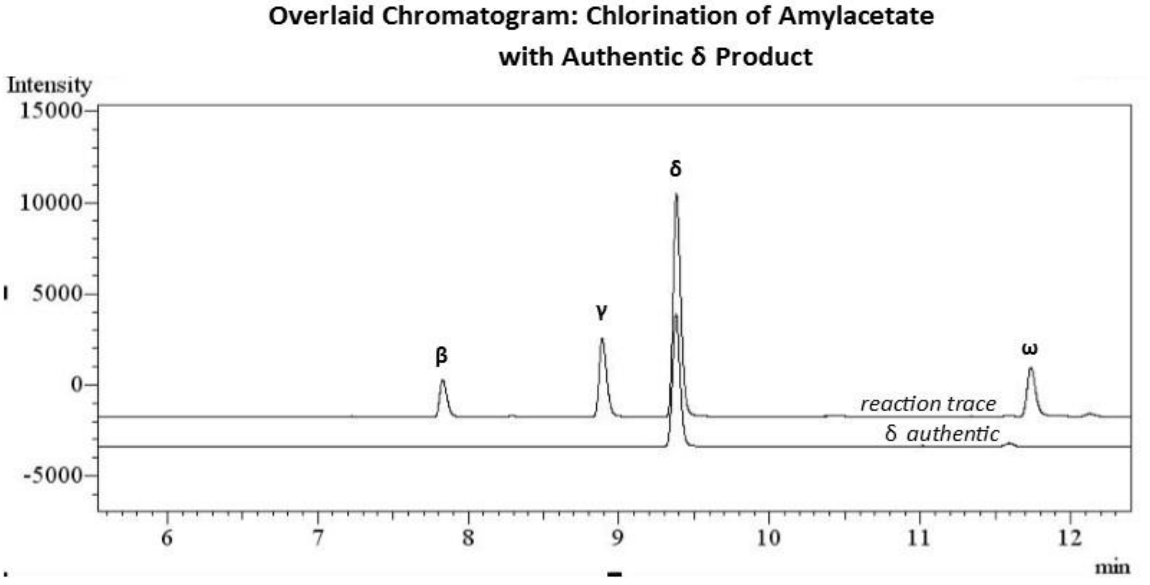
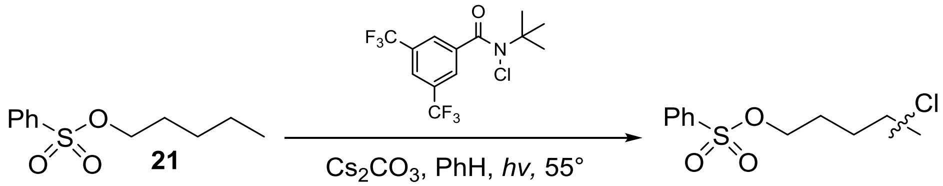


Table S9.

Chlorination of Amyl Acetate: Selectivity		
Product	Retention Time	Percent Area
α	---	---
β	7.83	8.9
γ	8.88	19.69
δ	9.83	57.85
ω	11.73	13.47



Pentyl Benzenesulfonate: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (100.0 mg, 0.286 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (280 μL). Pentyl benzenesulfonate (32.6 mg, 0.143 mmol), and cesium carbonate (92.5 mg, 0.286 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. 86.5% GC yield of combined chloride products.

Only the major isomer was synthesized independently. The secondary chloride products were assigned via analogy to our C-H bromination chemistry.

Figure S10.

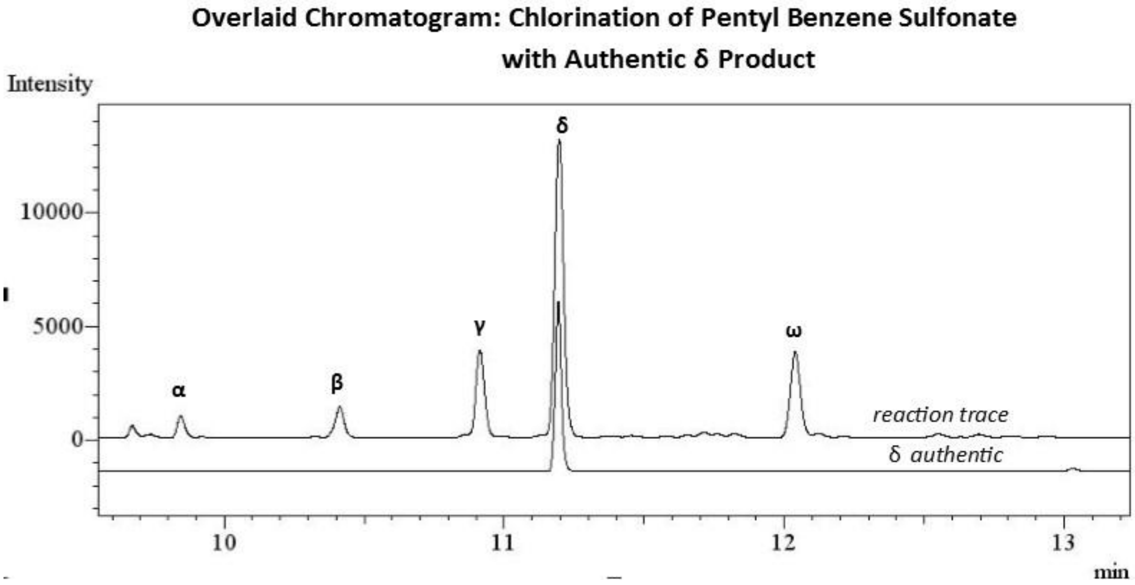
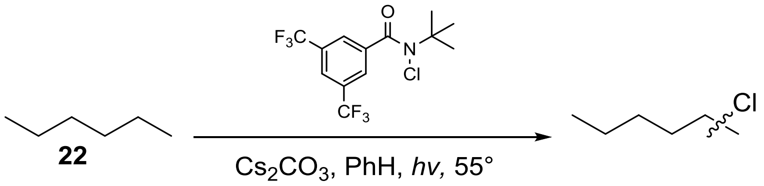


Table S10.

Chlorination of Pentyl Benzene Sulfonate: Selectivity		
Product	Retention Time	Percent Area
α	9.84	3.48
β	10.41	6.05
γ	10.91	16.13
δ	11.19	55.76
ω	12.04	18.57



n-Hexane: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (40.0 mg, 0.116 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (120 μL). n-Hexane (15.2 μL, 0.116 mmol), and cesium carbonate (37.8 mg, 0.116 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. 70.4% GC yield of combined chloride products.

Only the major isomer was synthesized independently. The secondary chloride products were assigned via analogy to our C-H bromination chemistry.

Figure S11.

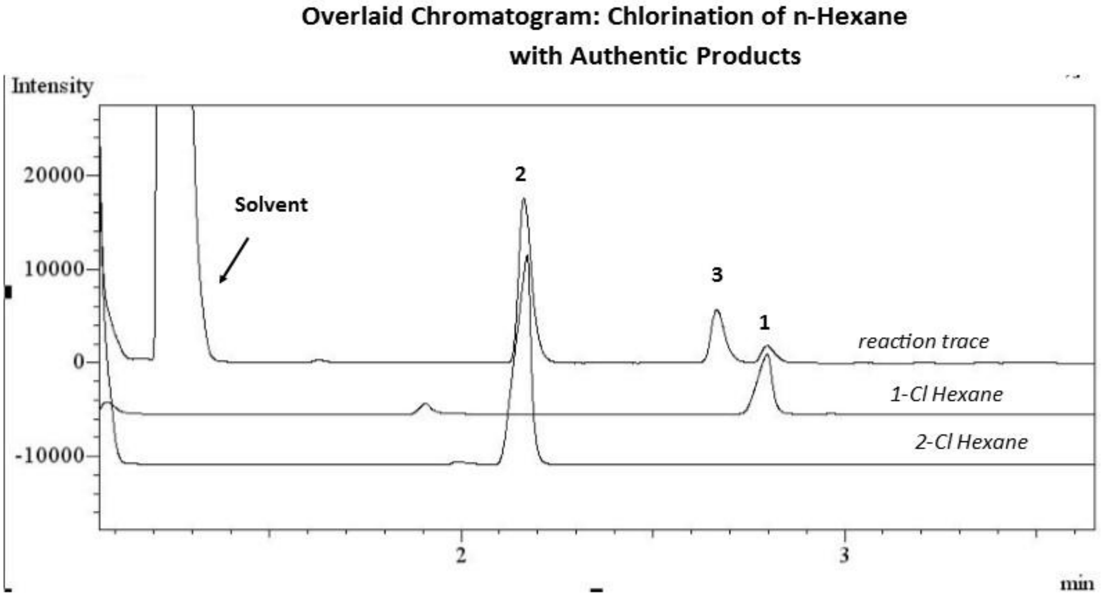
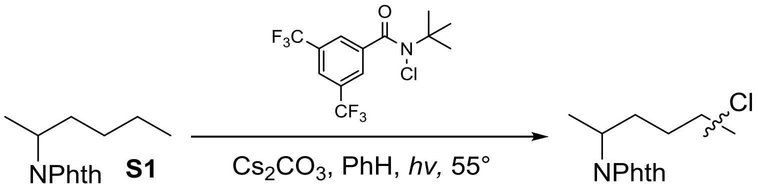


Table S11.

Chlorination of n-Hexane: Selectivity		
Product	Retention Time	Percent Area
2-Chlorohexane	2.16	69.51
3-Chlorohexane	2.66	23.38
1-Chlorohexane	2.79	7.10

Chlorination of in the Presence of More Reactive C-H Bonds and Substrate Unsaturation (Table 4)



S1: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (150.0 mg, 0.44 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (440 μL). The phthalimide substrate (50.2 mg, 0.22 mmol), and cesium carbonate (144.8 mg, 0.44 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. The crude reaction mixture was purified using column chromatography (5% EtOAc/Hexane) to isolate a mixture of major chloride products for characterization. 1D selective TOCSY NMR experiments were used to determine the identity of the major products. Reaction yield (88%) was calculated from crude NMR spectra using 2,5 dimethylfuran as an internal standard.

Analytical Data for mixture of chloride products formed from **S1:** **^{1}H NMR** ^{1}H NMR (600MHz ,CHLOROFORM-d) δ = 7.91 - 7.80 (m, 2 H), 7.79 - 7.66 (m, 2 H), 4.44 - 4.28 (m, 1 H), 4.13 - 3.94 (m, 0.8 H), 3.51 (t, J = 6.6 Hz, 0.4 H), 2.33 (dtd, J = 4.4, 9.9, 14.0 Hz, 0.5 H), 2.25 - 2.07 (m, 1 H), 2.07 - 1.95 (m, 0.5 H), 1.89 (tdd, J = 5.4, 10.7, 13.8 Hz, 0.5 H), 1.84 - 1.69 (m, 1.5 H), 1.69 - 1.55 (m, 1.5 H), 1.55 - 1.43 (m, 6 H); **^{13}C NMR** (125MHz ,CHLOROFORM-d) δ = 168.5, 134.0, 133.9, 131.9, 131.8, 123.2, 123.1, 58.3, 57.9, 47.3, 47.2, 46.7, 44.8, 37.6, 37.2, 32.9, 32.1, 31.1, 30.7, 25.4, 25.3, 24.1, 18.8, 18.7 ppm; **IR** (thin film, cm^{-1}) 2954.1, 2928.2, 1771.2, 1713.2, 1455.4, 1435.8, 1400.0, 1189.4, 1031.1, 723.2; **HRMS** (ESI) Calcd. for $[C_{14}H_{16}ClNO_2+Na]^+$ = 288.08, Found = 288.08

Figure S12.

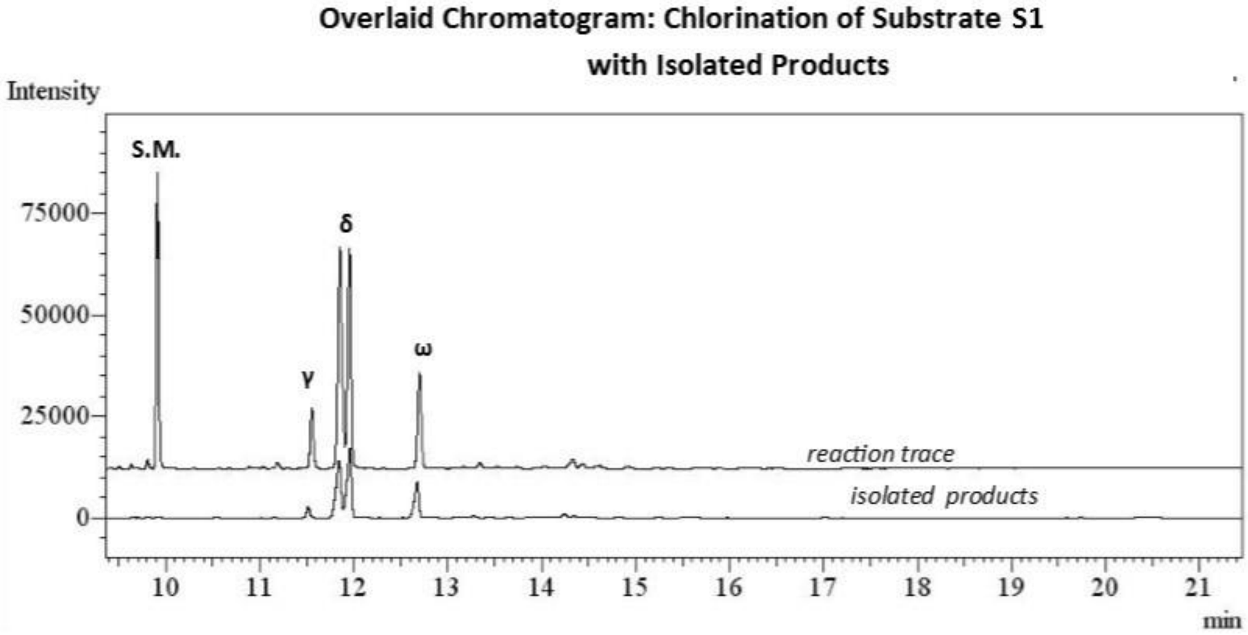
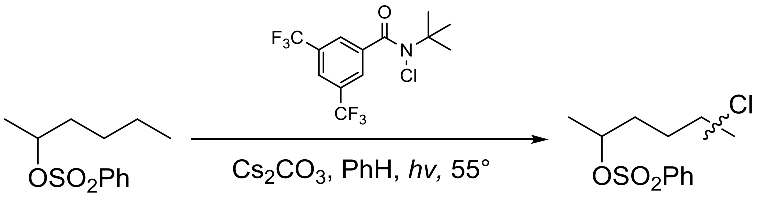


Table S12.

Chlorination of Phthalimide Substrate **S1**: Selectivity		
Product	Retention Time	Percent Area
α	---	---
β	---	---
γ	11.51	8.72
δ	11.81	42.75
δ	11.91	32.74
ω	12.04	18.57



S2

Chlorination of S2: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (150.0 mg, 0.44 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (440 µL). The sulfonate substrate (52.9 mg, 0.22 mmol), and cesium carbonate (144.8 mg, 0.44 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard.

The crude reaction mixture was purified using column chromatography (5% EtOAc/Hexane) to isolate a mixture of major chloride products for characterization. 1D selective TOCSY NMR experiments were used to determine the identity of the major products. Reaction yield (76%) was calculated from crude NMR spectra using 2,5 dimethylfuran as an internal standard.

Analytical Data for mixture of chloride products formed from **S2:** **^{1}H NMR** (600MHz ,CHLOROFORM-d) δ = 7.96 - 7.91 (m, 2 H), 7.70 - 7.64 (m, 1 H), 7.60 - 7.53 (m, 2 H), 4.76 - 4.62 (m, 1 H), 3.98 - 3.85 (m, 0.8 H), 3.45 (t, *J* = 6.6 Hz, 0.2 H), 1.90 - 1.50 (m, 0.8 H), 1.47 - 1.43 (m, 3 H), 1.42 - 1.31 (m, 2 H), 1.30 - 1.25 (m, 3 H) **^{13}C NMR** (125MHz ,CHLOROFORM-d) δ = 137.3, 133.7, 133.6, 129.4, 129.3, 129.2, 129.1, 127.9, 127.8, 127.7, 127.6, 80.5, 79.77, 37.7, 35.8, 35.1, 33.8, 33.3, 31.9, 25.4, 25.3, 21.0, 20.9 ppm; **IR** (thin film, cm^{-1}) 3010.3, 2926.0, 1777.3, 1710.3, 1448.9, 1452.0, 1400.8, 1387.4, 1190.2, 1033.0, 750.2; **HRMS** (ESI) Calcd. for $[C_{12}H_{17}ClSO_3+Na]^+$ = 288.05, Found = 299.05

Figure S13.

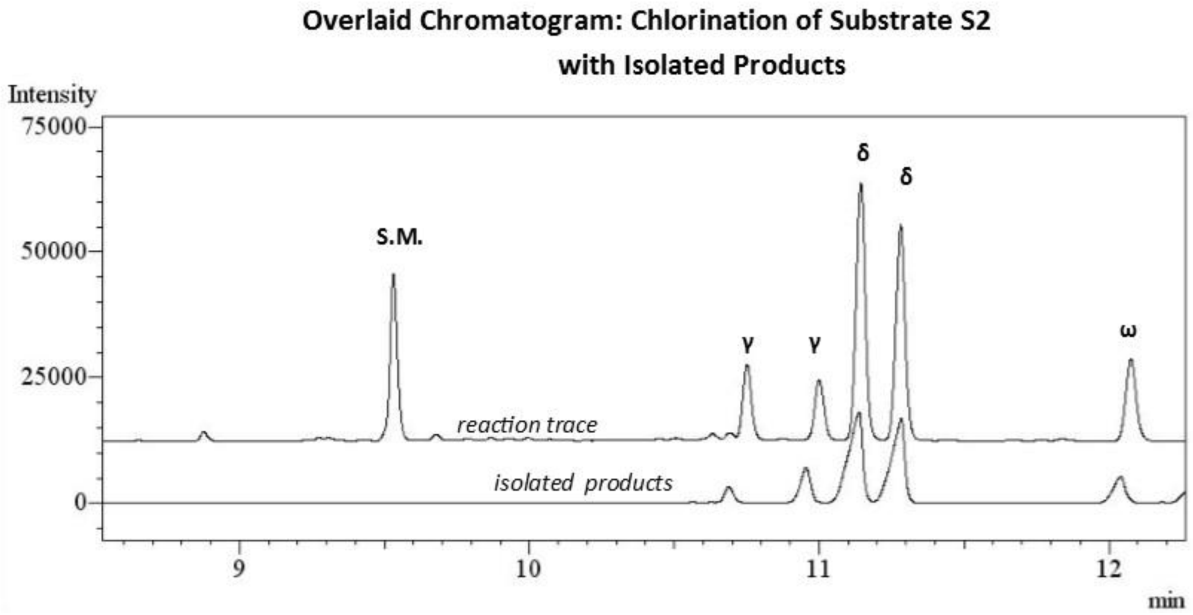
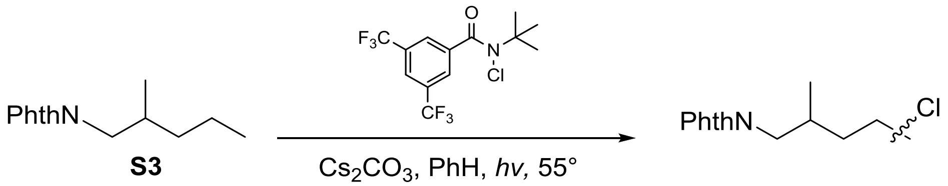


Table S13.

Chlorination of Sulfonate Substrate **S2**: Selectivity		
Product	Retention Time	Percent Area
α	---	---
β	---	---
γ	10.75	9.41
γ	11.00	8.90
δ	11.14	37.05
δ	11.28	31.23
ω	12.08	13.36



Chlorination of S3: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (150.0 mg, 0.44 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (440 μL). The phthalimide substrate (50.3 mg, 0.22 mmol), and cesium carbonate (144.8 mg, 0.44 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. The crude reaction mixture was purified using column chromatography (5% EtOAc/Hexane) to isolate a mixture of major chloride products for characterization. 1D selective TOCSY NMR experiments were used to determine the identity of the major products. Reaction yield (69%) was calculated from crude NMR spectra using 2,5 dimethylfuran as an internal standard.

Analytical Data for mixture of chloride products formed from **S3:** **^{1}H NMR** (600MHz ,CHLOROFORM-d) δ = 7.90 - 7.81 (m, 2 H), 7.78 - 7.69 (m, 13 H), 4.33 - 4.19 (m, 0.3 H), 4.11 (ddd, *J* = 3.5, 6.6, 10.5 Hz, 0.3 H), 3.70 - 3.63 (m, 0.3 H), 3.63 - 3.48 (m, 2 H), 2.33 (dtd, *J* = 3.5, 6.9, 13.6 Hz, 0.5 H), 2.28 - 2.20 (m, 0.3 H), 2.08 - 1.96 (m, 0.3 H), 1.96 - 1.85 (m, 3 H), 1.85 - 1.74 (m, 0.7 H), 1.74 - 1.66 (m, 1 H), 1.58 - 1.44 (m, 3 H), 1.01 - 0.91 (m, 3 H); **^{13}C NMR** (125MHz ,CHLOROFORM-d) δ = 168.8, 168.7, 168.6, 134.1, 134.0, 133.9, 133.8, 132.0, 131.9, 131.8, 123.3, 123.2, 56.2, 56.1, 45.2, 45.1, 44.7, 43.9, 43.8, 43.0, 32.2, 31.5, 30.8, 30.7, 30.6, 29.9, 26.1, 25.2, 18.1, 17.4, 16.7 ppm; **IR** (thin film, cm^{-1}) 3013.0, 2926.0, 1780.7, 1703.1, 1458.2, 1452.9, 1382.8, 1374.2, 1210.0, 1058.1, 701.2; **HRMS** (ESI) Calcd. for $[C_{14}H_{16}ClNO_2+Na]^+$ = 288.07, Found = 288.08

Figure S14.

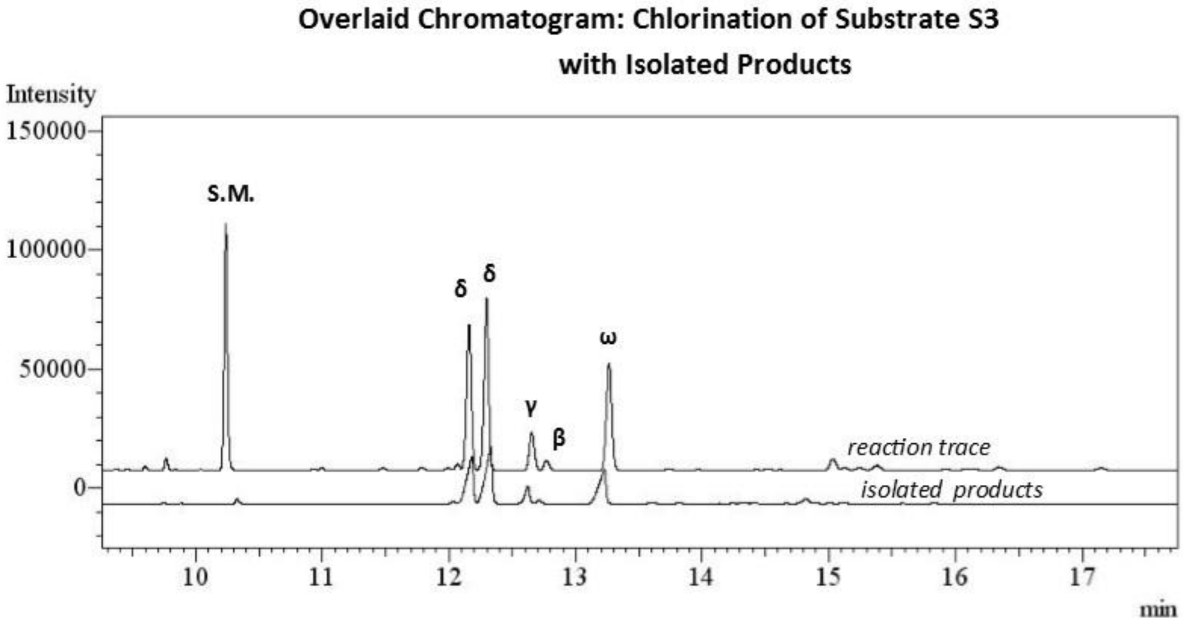
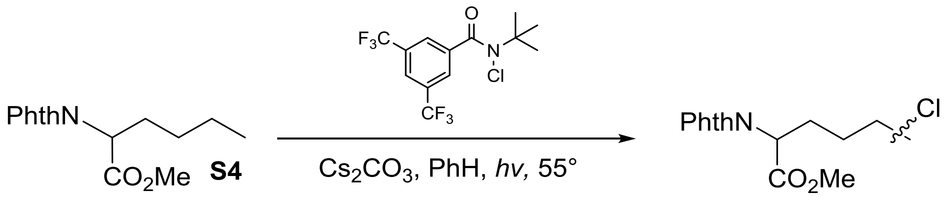


Table S14.

Chlorination of Phthalimide Substrate **S3**: Selectivity		
Product	Retention Time	Percent Area
α	---	---
β	12.77	2.05
γ	12.65	8.17
δ	12.96	29.96
δ	11.28	36.36
ω	13.27	25.49



Chlorination of S4: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (150.0 mg, 0.44 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (440 µL). The phthalimide substrate (58.2 mg, 0.22 mmol), and cesium carbonate (144.8 mg, 0.44 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. The crude reaction mixture was purified using column chromatography (10-20% EtOAc/Hexane) to isolate a mixture of major chloride products for characterization. 1D selective TOCSY NMR experiments were used to determine the identity of the major products. Reaction yield (66%) was calculated from crude NMR spectra using 2,5 dimethylfuran as an internal standard.

Analytical Data for mixture of chloride products formed from **S4:** **^{1}H** (600MHz ,CHLOROFORM-d) δ = 7.96 - 7.86 (m, 2 H), 7.83 - 7.72 (m, 2 H), 4.92 - 4.81 (m, 1 H), 4.13 - 3.97 (m, 0.8 H), 3.79 - 3.72 (m, 3 H), 3.52 (t, *J* = 6.6 Hz, 0.4 H), 2.59 - 2.49 (m, 0.5 H), 2.47 - 2.38 (m, 0.8 H), 2.38 - 2.23 (m, 1 H), 1.88 - 1.76 (m, 4 H), 1.74 - 1.65 (m, 1 H), 1.54 - 1.45 (m, 3 H); **^{13}C NMR** (125MHz ,CHLOROFORM-d) δ = 169.5, 169.4, 167.7, 167.6, 167.5, 134.4, 134.3, 131.7, 123.7, 123.6, 123.5, 57.8, 57.4, 52.9, 52.8, 51.9, 51.4, 44.5, 37.0, 36.7, 36.6, 31.7, 28.0, 26.4, 26.1, 25.4, 25.3, 23.6 ppm; **IR** (thin film, cm^{-1}) 3102.6, 2987.4, 2929.2, 1779.3, 1751.0, 1710.5, 1526.6, 1446.9, 1342.3, 1309.3, 1275.3, 882.9, 758.4, 719.4; **HRMS** (ESI) Calcd. for $[C_{25}H_{16}ClNO_4+Na]^+$ = 332.07, Found = 332.06

Figure S15.

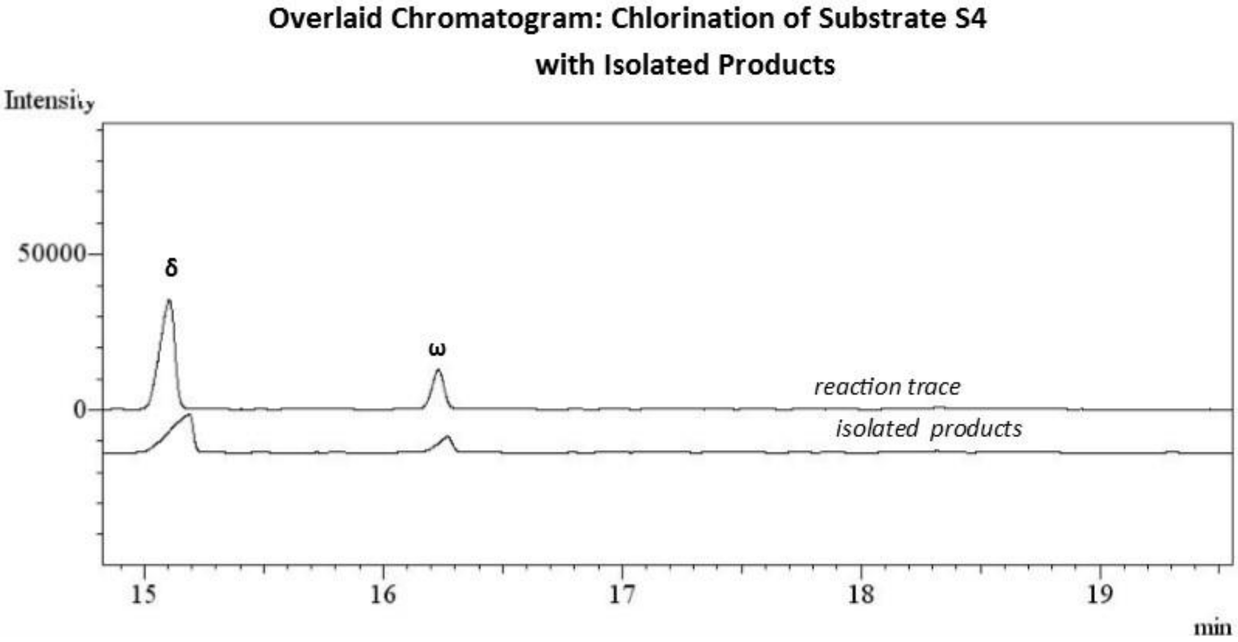
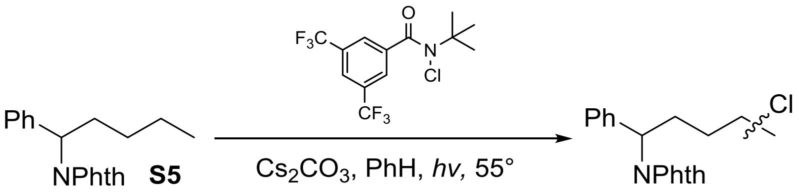


Table S15.

Chlorination of Phthalimide Substrate **S4**: Selectivity		
Product	Retention Time	Percent Area
α	---	---
β	---	---
γ	---	---
δ	15.14	77.54
ω	16.23	22.45



Chlorination of S5: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (150.0 mg, 0.44 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (440 μL). The phthalimide substrate (64.1 mg, 0.22 mmol), and cesium carbonate (144.8 mg, 0.44 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. The crude reaction mixture was purified using column chromatography (5% EtOAc/Hexane) to isolate a mixture of major chloride products for characterization. 1D selective TOCSY NMR experiments were used to determine the identity of the major products. Reaction yield (81%) was calculated from crude NMR spectra using 2,5 dimethylfuran as an internal standard.

Analytical Data for mixture of chloride products formed from **S5:** ^{1}**H NMR** (600MHz ,CHLOROFORM-d) δ = 7.85 - 7.80 (m, 2 H), 7.74 - 7.68 (m, 2 H), 7.60 - 7.54 (m, 2 H), 7.39 - 7.33 (m, 2 H), 7.32 - 7.25 (m, 1 H), 5.40 - 5.29 (m, 1 H), 4.19 - 4.02 (m, 0.8 H), 3.53 (t, *J* = 6.6 Hz, 0.4 H), 2.88 - 2.74 (m, 0.3 H), 2.74 - 2.55 (m, 1 H), 2.55 - 2.41 (m, 0.4 H), 1.88 (qd, *J* = 7.0, 13.9 Hz, 0.3 H), 1.83 - 1.70 (m, 0.5 H), 1.56 - 1.49 (m, 3 H); ^{13}**C NMR** (125MHz ,CHLOROFORM-d) δ = 168.4, 168.3, 168.2, 139.5, 139.3, 129.2, 134.1, 124.0, 133.9, 131.8, 131.7, 128.8, 128.7, 128.6, 128.2, 128.1, 128.0, 127.9, 127.8, 123.4, 123.3, 123.2, 58.2, 57.9, 54.9, 54.8, 45.4, 44.7, 37.7, 37.8, 37.6, 34.7, 32.1, 31.6, 30.3, 28.4, 28.1, 25.4, 25.3, 24.4, 22.7, 14.2 ppm; **IR** (thin film, cm^{-1}) 3022.4, 2959.2, 2929.3, 2859.4, 1769.3, 1710.9, 1463.7, 1397.1, 1072.3, 884.1, 757.5, 721.2; **HRMS** (ESI) Calcd. for $[C_{19}H_{18}ClNO_2+Na]^+$ = 350.09, Found = 350.10

Figure S16.

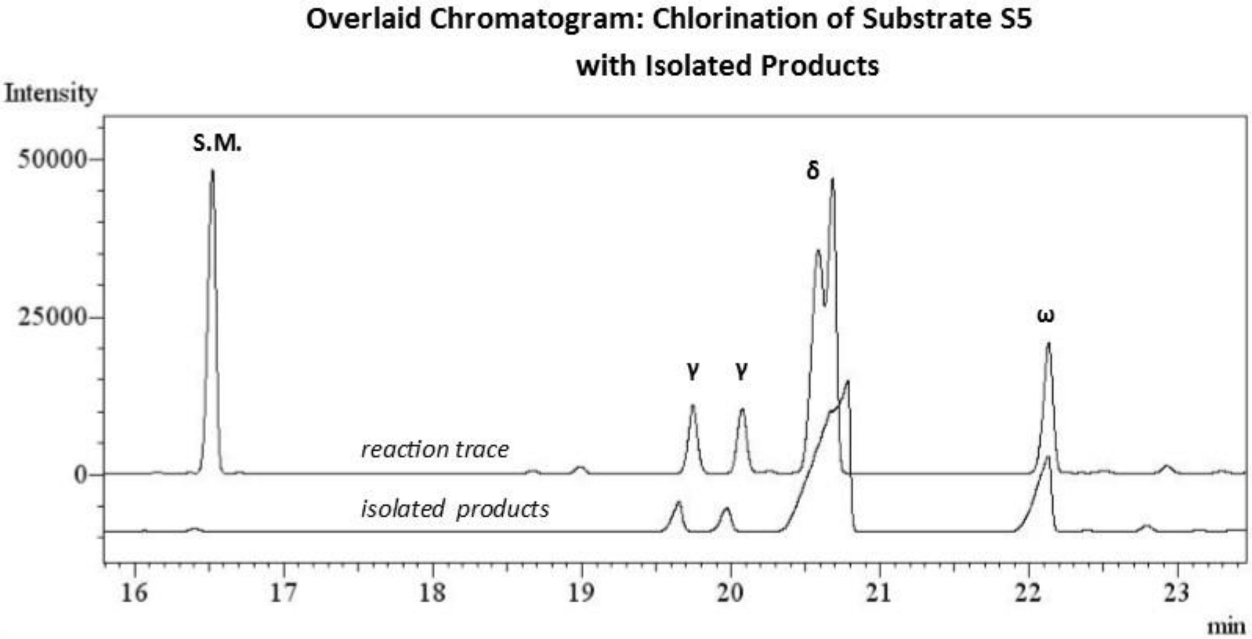
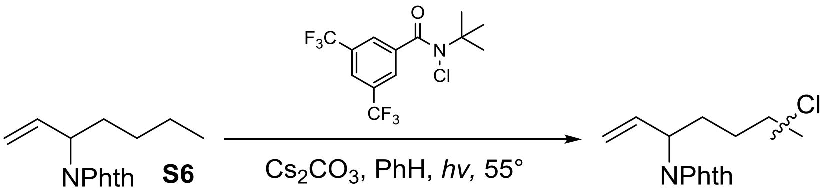


Table S16.

Chlorination of Phthalimide Substrate **S5**: Selectivity		
Product	Retention Time	Percent Area
α	---	---
β	---	---
γ	19.74	8.06
γ	20.07	7.68
δ	20.58	34.94
δ	20.68	32.90
ω	22.13	16.41



Chlorination of S6: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (150.0 mg, 0.44 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (440 μL). The phthalimide substrate (52.8 mg, 0.22 mmol), and cesium carbonate (144.8 mg, 0.44 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard.

The crude reaction mixture was purified using column chromatography (5% EtOAc/Hexane) to isolate a mixture of major chloride products for characterization. 1D selective TOCSY NMR experiments were used to determine the identity of the major products. Reaction yield (78%) was calculated from crude NMR spectra using 2,5 dimethylfuran as an internal standard.

Analytical Data for mixture of chloride products formed from **S6:** **^{1}H NMR** (600MHz ,CHLOROFORM-d) δ = 7.87 - 7.83 (m, 2 H), 7.77 - 7.71 (m, 2 H), 6.33 - 6.19 (m, 1 H), 5.33 - 5.17 (m, 2 H), 4.81 - 4.68 (m, 1 H), 4.13 - 3.96 (m, 1 H), 3.52 (t, *J* = 6.6 Hz, 0.5 H), 2.39 - 2.26 (m, 0.7 H), 2.26 - 2.03 (m, 3 H), 2.03 - 1.88 (m, 0.5 H), 1.88 - 1.70 (m, 3 H), 1.70 - 1.60 (m, 2 H), 1.55 - 1.43 (m, 3 H); **^{13}C NMR** (125MHz ,CHLOROFORM-d) δ = 168.1, 135.4, 135.3, 134.1, 134.0, 131.8, 123.3, 123.2, 118.0, 112.9, 58.0, 57.9, 53.9, 53.8, 53.5, 44.7, 37.2, 37.0, 32.0, 31.2, 29.3, 29.2, 25.4, 25.3, 23.8 ppm; **IR** (thin film, cm^{-1}) 2938.4, 2864.1, 1463.1, 1448.2, 1363.5, 1127.1, 1007.2, 980.4, 962.2, 824.4, 751.8, 684.9, 591.0; **HRMS** (ESI) Calcd. for $[C_{15}H_{16}ClNO_2+Na]^+$ = 300.07, Found = 300.08

Figure S17.

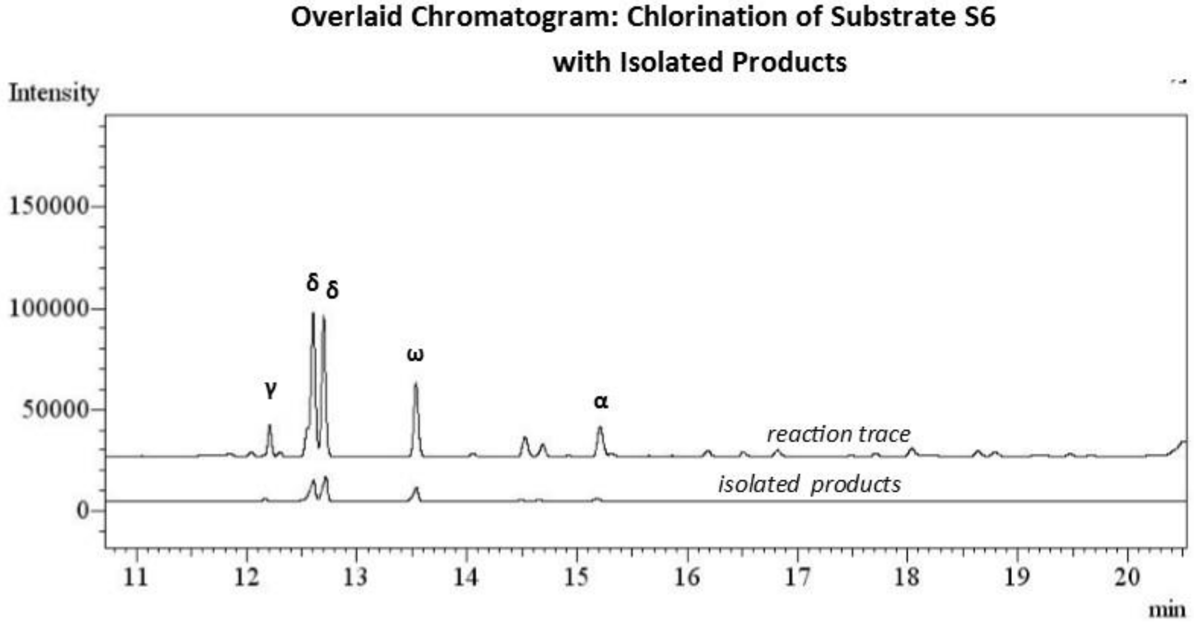
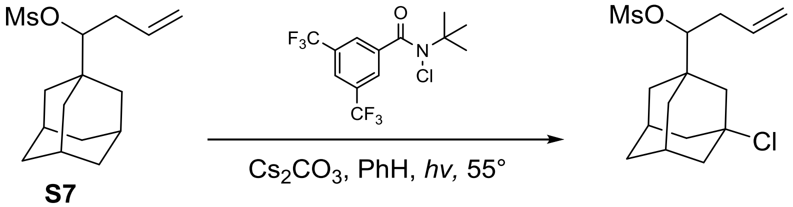


Table S17.

Chlorination of Phthalimide Substrate **S6**: Selectivity		
Product	Retention Time	Percent Area
α	15.14	8.99
β	---	---
γ	12.13	36.32
δ	12.53	28.77
δ	12.63	19.28
ω	13.46	8.99

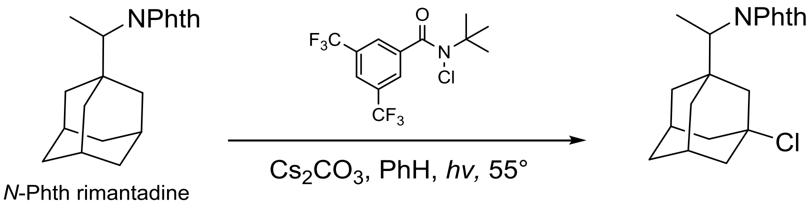
Chlorination of Complex Substrates



S7

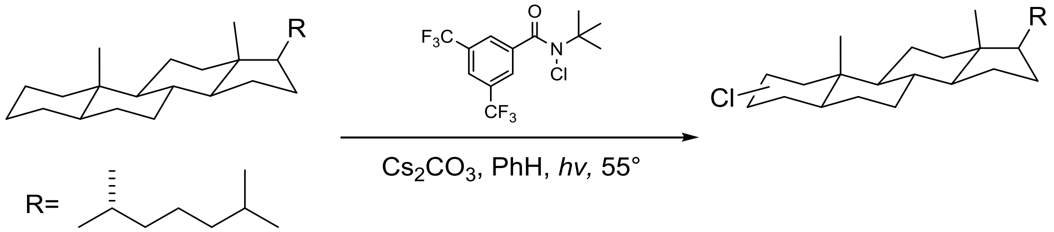
Chlorination of S7: A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (160.0 mg, 0.46 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (950 µL). The adamantane substrate (130.4 mg, 0.46 mmol), and cesium carbonate (150.s mg, 0.46 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. The crude reaction mixture was purified using column chromatography (5% EtOAc/Hexane) to isolate a single isomer of product (95 mg, 0.29 mmol, 65% yield) as a white solid.

Analytical Data for single chloride product formed from **S7:** **^{1}H NMR** (600MHz ,CHLOROFORM-d) δ = 5.96 - 5.81 (m, 1 H), 5.26 - 5.09 (m, 2 H), 4.47 (dd, *J* = 2.9, 9.4 Hz, 1 H), 3.09 - 2.98 (m, 3 H), 2.61 - 2.49 (m, 1 H), 2.41 (td, *J* = 8.9, 15.1 Hz, 1 H), 2.34 - 2.20 (m, 2 H), 2.17 - 2.10 (m, 2 H), 2.10 - 1.94 (m, 4 H), 1.76 - 1.49 (m, 7 H); **^{13}C NMR** (125MHz ,CHLOROFORM-d) δ = 134.7, 118.7, 89.4, 67.8, 47.6, 46.9, 46.8, 41.1, 39.3, 36.9, 36.5, 34.7, 34.1, 31.0, 30.9 ppm; **IR** (thin film, cm^{-1}) 2933.2, 2858.1, 2254.4, 1774.2, 1710.5, 1612.2, 1451.2, 1355.7, 1170.6, 1108.9, 1036.5, 912.2, 879.4, 835.0, 724.1, 531.3; **HRMS** (ESI) Calcd. for $[C_{15}H_{23}ClSO_3+Na]^+$ = 341.10, Found = 341.09



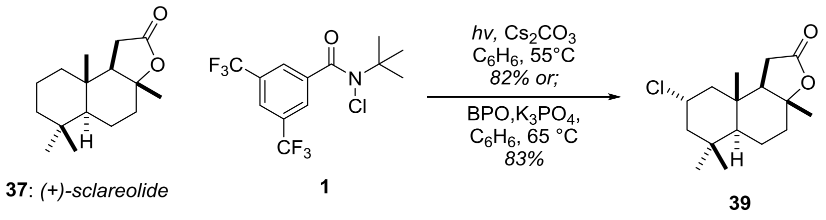
Rimantadine (30): A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (300.0 mg, 0.88 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (950 μL). The phthalimide substrate (272.3 mg, 0.22 mmol), and cesium carbonate (286.1 mg, 0.88 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. The crude reaction mixture was purified using column chromatography (5% EtOAc/Hexane) to isolate a single isomer of product (199 mg, 0.58 mmol, 66% yield) as a white solid. NMR of the crude reaction mixture showed a small amount of dichloride formed (10:1 mono:di).

Analytical Data for **31:** **^{1}H NMR**^{1}H NMR (600MHz ,CHLOROFORM-d) δ = 7.89 - 7.79 (m, 2 H), 7.77 - 7.62 (m, 2 H), 4.11 (q, *J* = 7.5 Hz, 1 H), 2.26 - 2.16 (m, 2 H), 2.11 - 1.90 (m, 6 H), 1.70 - 1.53 (m, 6 H), 1.51 (d, *J* = 7.3 Hz, 3 H); **^{13}C NMR** (125MHz ,CHLOROFORM-d) δ = 169.2, 169.1, 134.1, 133.9, 132.1, 131.4, 123.4, 123.1, 68.6, 55.6, 49.4, 46.9, 46.8, 42.1, 37.8, 37.6, 34.8, 31.4, 31.3, 11.96 ppm; **IR** (thin film, cm^{-1}) 2935.1, 2888.9, 1709.5, 1642.1, 1333.5, 1172.5, 905.4, 835.9, 791.6, 736.7, 539.0; **HRMS** (ESI) Calcd. for $[C_{20}H_{22}BrNO_2+H]^+$ = 366.12, Found = 366.12



Chlorination of Cholestane (32): A flame-dried, 1 dram vial was charged with a stir bar and chloroamide (80.0 mg, 0.23 mmol) in the dark (overhead lights in the laboratory turned off), fitted with a PTFE lined screw cap and the reaction was taken into a glovebox, and dissolved in of dry, freeze-pump-thawed benzene (800 μL). The alkane substrate (88.3 mg, 0.23 mmol), and cesium carbonate (72.3 mg, 0.23 mmol) were then added. The reaction was then sealed with teflon tape and taken out of the glovebox and irradiated with two 23W compact fluorescent light bulbs for 4 hours at 55°. Upon completion, the reaction mixture was diluted with DCM (3 mL) and directly analyzed by gas chromatography using dodecane as an internal standard. NMR of the crude reaction mixture was compared to existing literature data to determine the yield (75%) and selectivity of the reaction. The analytical data was consistent with the literature.[13]

Synthesis of Chlorolissoclimide: Experimental Procedures



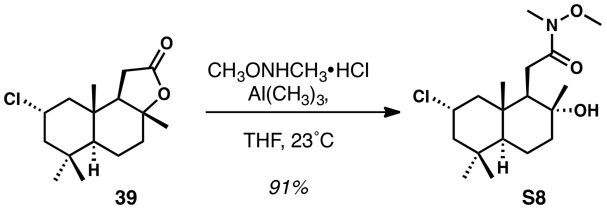
2-Chlorosclareolide 39.

(+)-Sclareolide **37** (600 mg, 2.39 mmol, 1.0 equiv), Cs_2CO_3 (780 mg, 2.39 mmol, 1.0 equiv), and chloroamide **1** (1.74 g, 5.0 mmol, 2.1 equiv) were dissolved in degassed benzene in the glovebox (5 mL). The mixture was stirred and irradiated with 2 - 23W CFL bulbs (1650 lumens each) at 55 ˚C for 36 h (when ^{1}H NMR revealed ~95% conversion). The mixture was filtered and concentrated under reduced pressure. Direct chromatographic purification (SiO_2, 5-10% EtOAc in hexanes) afforded a clear amorphous solid (535 mg, 3.9 mmol, 78% yield) Another 22mg were present in the mixed fractions to give a total of 557mg, 81.6% yield. (The reaction can be performed without the use of freeze pump thawed PhH or a glove box by degassing the reaction mixture with Ar for 15 minutes prior to heating to achieve 73% yield.)

Alternative Procedure Using Benzoyl Peroxide as the Radical Initiator: (+)-Sclareolide **37** (1.00 g, 3.99 mmol, 1.0 equiv), benzoyl peroxide (165 mg, 0.68 mmol, 0.17 equiv), K_3PO_4 (144 mg, 0.68 mmol, 0.17 equiv), and chloroamide **1** (3.5 g, 10.0 mmol, 2.5 equiv) were dissolved in benzene (13 mL) and the mixture was degassed for 20 min with an Ar balloon bearing a 18 gauge needle. A reflux condenser was attached to the flask and the mixture was heated at reflux for 24 h. A 30 μL aliquot revealed a ~50% conversion (^{1}H NMR). The reaction mixture was cooled to ambient temperature and more benzoyl peroxide (100 mg, 0.41 mmol, 0.10 equiv) and K_3PO_4 (87 mg, 0.41 mmol, 0.10 equiv) were added. The solution was degassed in the same manner and heated for 16 h after which aliquot NMR revealed ~75% conversion. This process was repeated again and after another 15 h no sclareolide remained. The mixture was concentrated under a stream of air. Direct chromatographic purification (SiO_2, 10% EtOAc in hexanes) afforded a clear amorphous solid (941 mg, 3.31 mmol, 83% yield). Our data match those previously reported by Groves.[13]

^{1}H NMR (500 MHz, CDCl$_3$) δ 4.22 (tt, *J* = 12.2, 4.2 Hz, 1H), 2.43 (dd, *J* =16.2, 14.8 Hz, 1H), 2.29 (dd, *J* = 16.2, 6.5 Hz, 1H), 2.13 (dt, *J* = 12.0, 3.3 Hz, 1H), 1.98–2.07 (m, 2H), 1.91 (dq, *J* = 14.4, 3.3 Hz, 1H), 1.70 (td, *J* = 12.4, 4.3 Hz, 1H), 1.54 (t, *J* = 12.7 Hz, 1H), 1.32–1.42 (m, 2H), 1.33 (s, 3H), 1.13 (dd, *J* = 12.7, 2.7 Hz, 1H), 0.972 (s, 3H), 0.968 (s, 3H), 0.90 (s, 3H); **^{13}C NMR** (125 MHz, CDCl$_3$) δ 176.0, 85.8, 58.6, 55.7, 53.8, 52.4, 49.7, 38.4, 38.2, 35.8, 32.9, 28.6, 21.6, 21.3, 20.1, 15.8; **IR** (film) 2951, 2871, 1773, 1387, 1197 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{16}H_{25}O_2Cl$ [M]$^+$: 284.1543; found 284.1548; **$[\alpha]^{25}_D$** +48.2° (*c* = 1.00, CHCl$_3$).

(Note: The following two-step sequence to make **40** is based on the procedures for the non-chlorinated variant reported by Boukouvalas[14] with slight modifications.)

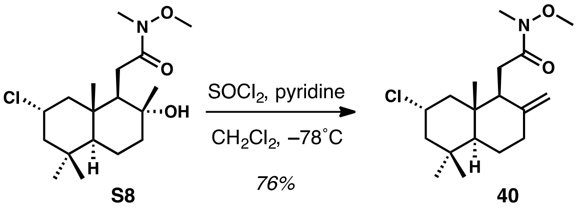


Amide S8.

Al(CH$_3$)$_3$ (2.0 M in toluene, 3.50 mL, 6.99 mmol, 2.1 equiv) was added to a suspension of N,O-dimethylhydroxylamine hydrochloride (647 mg, 6.66 mmol, 2.0 equiv) in CH$_2$Cl$_2$ (12 mL) at 0˚C. The white suspension became a clear solution. The ice bath was removed and after 2 h a solution of **39** (950 mg, 3.33 mmol, 1.0 equiv in 12 mL CH$_2$Cl$_2$) was added dropwise over ~5 min at ambient temperature. After stirring for 2 hours at 23 ˚C, the flask was cooled in an ice bath and 5 mL of 10% aq. H$_2$SO$_4$ was added.

The ice bath was removed and the crude mixture was diluted with CH$_2$Cl$_2$ (40 mL), H$_2$O (10 mL), and 1M HCl (30 mL). The organic phase was separated and the aqueous phase was extracted with CH$_2$Cl$_2$ (2 x 40 mL). The combined organic layers were dried over MgSO$_4$, filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography (SiO$_2$, 70%→80% EtOAc in hexanes) to afford amide **S8** as a thick clear oil (1.05 g, 3.04 mmol, 91% yield).

^{1}H NMR (600 MHz, CDCl$_3$) δ 4.17 (tt, *J* = 12.3, 4.0 Hz, 1H), 3.74 (s, 1H), 3.20 (s, 1H), 2.58 (d, *J* = 16.1 Hz, 1H), 2.48 (dd, *J* = 16.4, 6.7 Hz, 1H), 2.08 (ap d, *J* = 10.9, 1H), 2.03 (dd, *J* = 6.5, 3.6 Hz, 1H), 1.92–1.98 (m, 2H), 1.69 (dq, *J* = 13.8, 2.4 Hz, 1H), 1.50 (t, *J* = 12.7 Hz, 1H), 1.47 (dd, *J* = 13.3, 3.9 Hz, 1H), 1.32 (t, *J* = 12.2 Hz, 1H), 1.26 (qd, *J* = 13.2, 3.2 Hz, 1H) 1.16 (s, 1H), 1.09 (dd, *J* = 12.3, 2.0 Hz, 1H), 0.95 (s, 3H), 0.88 (s, 3H), 0.85 (s, 3H); **^{13}C NMR** (125 MHz, CDCl$_3$) δ 175.5, 72.7, 61.3, 56.3, 55.8, 55.1, 54.9, 51.9, 49.6, 44.1, 41.0, 35.9, 33.1, 27.0, 23.4, 21.8, 20.1, 16.5; **IR** (film) ν 3500, 2966, 2927, 1655, 1454, 1340, 1158 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{18}H_{32}O_3ClNa$ [M + Na]$^+$: 368.1968; found 368.1963; **$[\alpha]^{25}_D$** +32.9˚ (*c* = 1.00, $CHCl_3$).

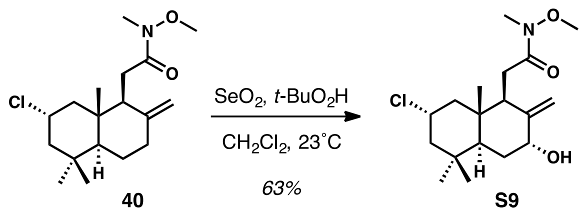


Alkene 40.

Alcohol **S8** (510 mg, 1.46 mmol, 1.0 equiv) was dissolved in CH_2Cl_2 (10 mL). Pyridine (236 μL, 2.92 mmol, 2.0 equiv) was added, and the solution was cooled to –78 ˚C. Pyridine (969 μL, 12.0 mmol, 8.25 equiv) was added to a conical flask containing $SOCl_2$ (530 μL, 7.30 mmol, 5 equiv) and CH_2Cl_2 (15 mL) and this clear mixture was transferred via Teflon® cannula[15] dropwise over 15 min to the flask containing alcohol **S8**.

The reaction was complete after 45 min at –78 ˚C and sat. aq. $NaHCO_3$ (20 mL) was added. The product mixture was warmed to ambient temperature, the phases were separated and the aqueous phase was extracted (2 x 30 mL CH_2Cl_2). The combined organic extracts were dried ($MgSO_4$), filtered, and concentrated under reduced pressure. The pale yellow crude oil was purified by flash column chromatography (SiO_2, 11% EtOAc in hexanes) to afford alkene **40** as a thick oil (364 mg, 1.11 mmol, 76% yield).

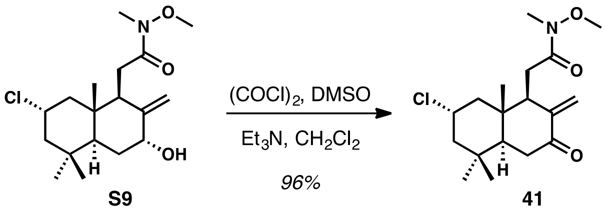
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.78 (s, 1H), 4.48 (s, 1H), 4.17 (tt, *J* = 12.3, 3.9 Hz, 1H), 3.73 (s, 3H), 3.16 (s, 3H), 2.72 (dd, *J* = 15.0, 10.7 Hz, 1H), 2.58 (d, *J* = 10.1 Hz, 1H), 2.37–2.42 (m, 2H), 2.09–2.18 (m, 2H), 1.98 (ddd, *J* = 12.8, 3.7, 2.2 Hz, 1H), 1.70–1.76 (m, 1H), 1.55 (t, *J* = 12.6 Hz, 1H), 1.50 (t, *J* = 12.2 Hz, 1H), 0.97 (s, 3H), 0.85 (s, 3H), 0.79 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 174.1, 148.3, 107.0, 61.4, 55.8, 53.9, 52.0, 51.4, 49.3, 41.2, 37.1, 36.1, 33.3, 32.5, 24.1, 23.4, 22.2, 15.3; IR (film) ν 2939, 2852, 1776, 1667, 1461, 1385, 1004 cm^{-1}; **HRMS** (ESI) *m/z* calc'd for $C_{18}H_{30}O_2ClNNa$ [M + Na]$^+$ 350.1863, found 350.1866; **$[\alpha]^{25}_D$** +7.38˚ (*c* = 1.00, $CHCl_3$).



Allylic Alcohol S9.

A solution of *t*-butyl hydroperoxide (133 μL, 5.5 M in decane, 730 μmol, 4.4 equiv) was added to an ice cold stirring suspension of selenium dioxide (5.5 mg, 49.8 μmol, 0.3 equiv) in CH_2Cl_2 (2.5 mL). After 30 min, a solution of amide **40** (54.4 mg, 166 μmol, 1.0 equiv) was added dropwise and the solution was allowed to warm to room temperature. TLC analysis indicated complete consumption of starting material after 8 h. The excess peroxide was quenched with sat. aq. Na_2SO_3. The biphasic solution was diluted with water (5 mL) and CH_2Cl_2 (5 mL) and the aqueous phase was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic phases were dried ($MgSO_4$), and concentrated *in vacuo*. The crude oil was purified by column chromatography (SiO_2, 80% EtOAc in hexanes) to give alcohol **S9** as a white amorphous solid (36.4 mg, 105 μmol, 63% yield).

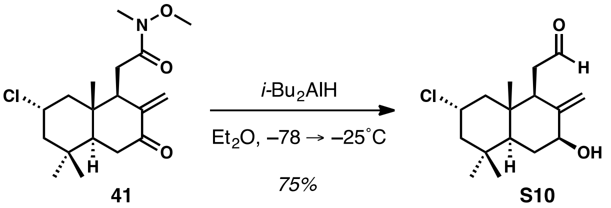
^{1}H NMR (500 MHz, $CDCl_3$) δ 5.01 (s, 1H), 4.61 (s, 1H), 4.37 (s, 1H), 4.17 (tt, *J* = 12.3, 3.9 Hz, 1H), 3.74 (s, 3H), 3.16 (s, 3H), 3.06 (d, *J* = 10.8 Hz, 1H), 2.68 (dd, *J* = 15.8, 10.9 Hz, 1H), 2.42 (dd, *J* = 16.1, 3.3 Hz, 1H), 2.13 (ap d, *J* = 12.1 Hz, 1H) 2.00 (ddd, *J* = 12.8, 3.8, 2.1 Hz, 1H), 1.91 (dt, *J* = 13.9, 2.7 Hz, 1H), 1.84 (dd, *J* = 12.9, 2.9 Hz, 1H), 1.60 (t, *J* = 12.6 Hz, 1H), 1.56 (t, *J* = 12.2 Hz, 1H), 1.48 (td, *J* = 13.4, 3.1 Hz, 1H), 0.98 (s, 3H), 0.87 (s, 3H), 0.76 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 177.5, 149.6, 110.2, 73.1, 61.4, 55.7, 52.0, 49.0, 46.2, 46.0, 41.3, 35.7, 32.4, 33.0, 29.8, 26.7, 22.1, 14.4; **IR** (film) ν 3405, 2960, 2939, 2897, 1645, 1460, 1436, 1389, 766 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{18}H_{30}O_3ClNNa$ $[M + Na]^+$: 366.1812, found 366.1818; **$[\alpha]^{25}_D$** –25.7° (*c* = 0.60, $CHCl_3$).



Enone 41.

A solution of oxalyl chloride (16.7 μL, 192 μmol, 1.2 equiv) in CH_2Cl_2 (800 μL) was cooled to –78 ˚C and DMSO (27 μL, 384 μmol, 2.4 equiv) was added. After 10 min alcohol **S9** (55 mg in 800 μL CH_2Cl_2, 160 μmol, 1.0 equiv) was added dropwise over 10 min. The solution was stirred for 20 min and Et_3N (111.5 μL, 800 μmol, 5 equiv) was added. The suspension was allowed to warm to 0 ˚C. After 40 min at 0 ˚C no starting material was present by TLC analysis and the mixture was diluted with hexanes (5 mL) and sat. aq. NH_4Cl (5 mL). The biphasic solution was further diluted with 1:1 hexanes in EtOAc (15 mL) and water. The organic phase was washed sequentially with brine (10 mL) and water (10 mL), dried ($MgSO_4$), filtered, and concentrated under reduced pressure. Flash column chromatography (SiO_2, 35% EtOAc in hexanes) gave **41** as a thick oil (53 mg, 154 μmol, 96% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.00 (d, *J* = 2.5 Hz, 1H), 5.11 (d, *J* = 2.4 Hz, 1H), 4.18 (tt, *J* = 12.4, 3.8 Hz, 1H), 3.74 (s, 3H), 3.19 (s, 3H), 2.97–3.03 (m, 1H), 2.58–2.72 (m, 3H), 2.28 (dd, *J* = 17.9, 14.1 Hz, 1H), 2.23 (ap d *J* = 12.5 Hz, 1H), 2.05 (ddd, *J* = 12.9, 3.7, 2.2 Hz, 1Hz), 1.73 (dd, *J* = 14.0, 4.6 Hz, 1H), 1.60 (t, *J* = 12.7 Hz, 1H), 1.56 (t, *J* = 12.3 Hz, 1H), 0.96 (s, 3H), 0.94 (s, 3H), 0.91 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 200.6, 146.8, 121.1, 72.1, 61.5, 54.6, 51.6, 49.8, 49.6, 48.9, 39.5, 37.5, 36.0, 32.7, 32.3, 28.9, 21.4, 15.1; **IR** (film) ν 2963, 1693, 1662, 1608, 1461, 1415, 1388, 1264 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{18}H_{28}O_3ClNNa$ $[M + Na]^+$: 364.1655, found 364.1658; **$[\alpha]^{22}_D$** –36.3˚ (*c* = 0.51, $CHCl_3$).

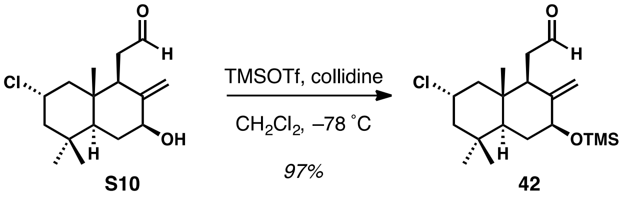


Aldehyde S10.

Enone **41** (72 mg, 205 μmol, 1.0 equiv) was dissolved in Et_2O (4 mL, 0.05 M) and the solution was cooled to –78˚C, forming a suspension. A solution of *i*Bu_2AlH in toluene (1.0 M, 615 μL, 615 μmol, 3.0 equiv) was added slowly along the side of the flask and the reaction mixture became homogenous. After 40 min at –78 ˚C, the flask was warmed to –25 ˚C over 1 h then stirred at that temperature. After 1.5 h acetone (50 μL) was added followed by 1M HCl (1 mL). The biphasic solution was warmed to ambient temperature and 1M HCl (3 mL) and water (3 mL) were added. The aqueous phase was extracted (3 x 6 mL 1:1 EtOAc in hexanes) and the combined organic extracts were dried ($MgSO_4$), filtered and concentrated. The crude oil was purified by chromatography (SiO_2, 22%→27% EtOAc in hexanes) to afford **S10** as a thick oil (44 mg, 154 μmol, 75% yield).

^{1}H NMR (500 MHz $CDCl_3$) δ 9.66 (d, *J* = 3.0 Hz, 1H) 5.23 (s, 1H), 4.62 (s, 1H), 4.16 (tt, *J* = 12.3, 4.0 Hz, 1H), 4.10 (br s, 1H), 2.63 (ddd, *J* = 17.0, 11.1, 3.0 Hz, 1H), 2.48 (dd, *J* = 17.0, 3.5 Hz, 1H), 2.41 (ap d, *J* = 11.0 Hz, 1H), 2.07–2.14 (m, 3H), 2.02 (ddd, *J* = 13.0, 3.9, 2.1 Hz, 1H), 1.86 (d, *J* = 4.1 Hz, 1H), 1.55 (t, *J* = 12.7 Hz, 1H), 1.41 (t, *J* = 12.2 Hz, 1H), 1.23–1.32 (m, 2H), 1.00 (s, 3H), 0.88 (s, 3H), 0.75 (s, 3H); **^{13}C**

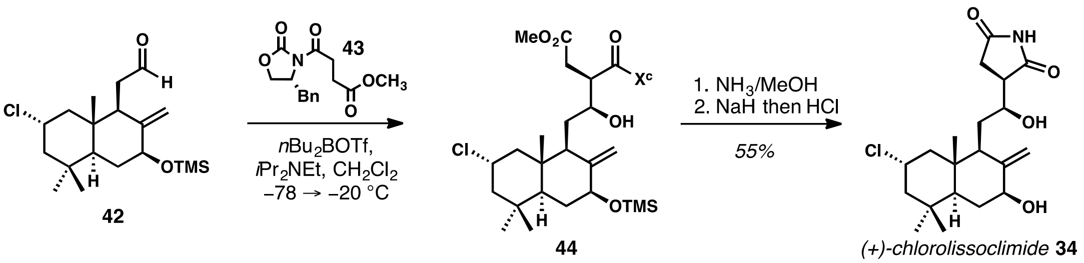
NMR(125 MHz, $CDCl_3$) δ 201.6, 149.3, 105.9, 72.97, 55.0, 51.84, 51.76, 49.4, 48.5, 40.7, 39.3, 35.9, 33.2, 32.7, 22.1, 15.1; **IR** (film) ν 3400, 2960, 2853, 2725, 1720, 1647, 1459, 1391 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{16}H_{25}O_2ClNH_4$ $[M + NH_4]^+$: 302.1887, found 302.1882. **$[\alpha]^{22}_D$** –18.1° (*c* = 1.0, $CHCl_3$).



TMS ether 42.

Aldehyde **S10** (12.5 mg, 43 μmol, 1.0 equiv) was dissolved in CH_2Cl_2 (450 μL) and cooled to –78 ˚C. To the reaction mixture were sequentially added 2,4,6-collidine (57 μL, 430 μmol, 10 equiv) and TMSOTf (47 μL, 258 μmol, 6.0 equiv). After 1 h at –78 ˚C, triethylamine (50 μL) and methanol (100 μL) were added and the flask was warmed to ambient temperature. The solution was diluted with 10% EtOAc in hexanes (5 mL) and washed consecutively with water, citric acid (10% aq.), and brine (4 mL each). The organic extract was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography to give aldehyde **42** a thin clear film (14.5 mg, 40.6 μmol, 97% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.64 (d, *J* = 2.8 Hz, 1H), 5.25 (s, 1H), 4.58 (s, 1H), 4.15 (tt, *J* = 12.2, 3.8 Hz, 1H), 4.02 (ap dd, *J* = 10.1, 5.2 Hz, 1H), 2.62 (ddd, *J* = 16.8, 10.9, 3.1 Hz, 1H), 2.46 (dd, *J* = 16.8, 3.5 Hz, 1H), 2.39 (ap d, *J* = 10.6 Hz, 1H), 2.08 (ap d, *J* = 12.1 Hz, 1H), 2.03 (ddd, *J* = 12.9, 3.9, 2.0 Hz, 1H), 1.93 (ddd, *J* = 11.9, 4.2, 1.7 Hz, 1H), 1.54 (t, *J* = 12.7 Hz, 1H), 1.39 (t, *J* = 11.9 Hz, 1H), 1.32 (ap t, *J* = 11.1 Hz, 1H), 1.29 (t, *J* = 15.1 Hz, 1H), 0.98 (s, 3H), 0.87 (s, 3H), 0.75 (s, 3H), 0.13 (s, 9H); **^{13}C NMR**(125 MHz, $CDCl_3$) δ 201.8, 148.5, 107.0, 73.5, 55.1, 51.9, 51.8, 49.5, 48.6, 40.7, 39.4, 35.8, 33.6, 33.1, 22.1, 15.1, –0.1; **IR** (film) ν 2956, 2851, 2718, 1726, 1650, 1460, 1393, 1251 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{19}H_{33}O_2ClSiNH_4$ $[M + NH_4]^+$: 374.2282, found 374.2285; **$[\alpha]_D^{22}$** –8.60° (*c* = 1.10, $CHCl_3$).



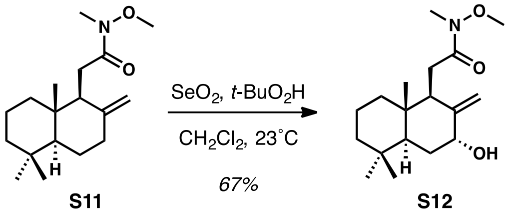
Chlorolissoclimide 34.

Imide **43**[16] (14.0 mg, 48 µmol, 1.3 equiv) was dried by azeotropic distillation from of benzene (20 µL) in a 1 dram vial and dissolved in CH_2Cl_2 (250 µL, 0.1 M). The solution was cooled to –78 ˚C and $n$$Bu_2BOTf$ (1.0 M in CH_2Cl_2, 52 µL, 52 µmol, 1.4 equiv)[17] was added along the side of the vial. The clear solution was stirred at –78 ˚C for 30 min. At that temperature, $i$$Pr_2NEt$ (1.0 M in CH_2Cl_2, 67 µL, 67 µmol, 1.8 equiv) was added and the mixture was stirred for 20 min. To ensure complete enolization, the vial was warmed to 0 ˚C for 30 sec and cooled back to –78 ˚C. The solution often becomes pale yellow upon warming but remained clear. Finally, a solution of aldehyde **42** (13.2 µg in 100 µL CH_2Cl_2, 37 µmol, 1.0 equiv) was added at –78 ˚C and the reaction vial was warmed over 1.5 hours to –25 ˚C and a temperature between –30 and –25 ˚C was maintained for 20 h. The reaction mixture was quenched with methanol (50 µL) warmed to ambient temperature, and water (3 mL) and CH_2Cl_2 (3 mL) were added. The phases were separated and the aqueous phase was extracted with CH_2Cl_2 (2 x 3 mL). The combined organic extracts were dried ($MgSO_4$), filtered, and concentrated *in vacuo* to yield crude product **44** a yellow oil.

The crude aldol product was transferred to a 1 dram vial, NH_3 (2.0 M in methanol, 800 µL) was added, and the reaction was stirred at 23 ˚C for 24 h. The solution was concentrated *in vacuo* to afford a mixture of chlorolissoclimide (**34**), and the primary amide of methyl ester **44**. To complete succinimide formation, the crude material was dissolved in dry THF (400 µL) and NaH (55% suspension in mineral oil, 3.5 mg, 80 µmol) was added at ambient temperature. The solution evolved bubbles for ~10 min. Water (200 µL) was added followed by 1M HCl (100 µL). The solution was quickly added to a mixture of pH 7 buffer (2 mL), brine (1 mL), and water (1 mL) and extracted with CH_2Cl_2 (4 x 4 mL CH_2Cl_2). The combined organic extracts were dried ($MgSO_4$) and concentrated under reduced pressure. Flash chromatrography (SiO_2, 60%→70% EtOAc in hexanes) afforded a modestly (~85%) pure product. To further purify the product, this mixture was taken up in CH_2Cl_2 (250 µL) and hexanes was added until a white precipitate formed (~300 µL). The suspension was centrifuged for 1 min and the solvent was decanted with a pipette, leaving pure chlorolissoclimide (**34**) as a white film (7.8 mg, 20.3 µmol, 55% yield).

The data obtained for our synthetic sample of **34** matched those reported by Biard. For ^1H NMR and ^{13}C NMR data comparison to natural **34** in CD_2Cl_2 see Table S18, below. [18]

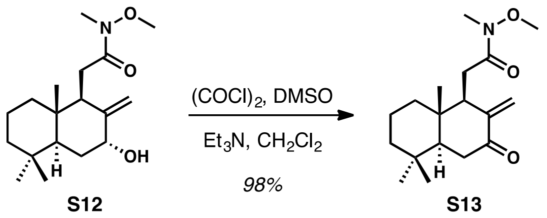
^{1}H NMR (500 MHz $CDCl_3$) δ 8.88 (br s, 1H), 5.30 (s, 1H), 4.90 (s, 1H), 4.29 (br s, 1H), 4.17 (tt, *J* = 12.2, 3.9 Hz, 1H), 4.00 (s, 1H), 3.03 (d, *J* = 3.2 Hz, 1H), 2.88–2.95 (m, 1H), 2.85 (dd, *J* = 18.0, 5.0 Hz, 1H), 2.68 (dd, *J* = 18.0, 9.2 Hz, 1H), 2.61 (br s, 1H), 2.20 (, *J* = 10.5 Hz, 1H), 2.09 (dd, *J* = 10.3, 4.8 Hz, 1H), 2.02 (d, *J* = 10.9 Hz, 1H), 1.82 (ddd, *J* = 13.2, 11.6, 5.3 Hz, 1H), 1.69 (s, 1H), 1.55–1.63 (m, 1H), 1.52 (t, *J* = 12.7 Hz, 1H), 1.23–1.28 (m, 2H), 1.20 (t, *J* = 14.2 Hz, 1H), 0.99 (s, 3H), 0.87 (s, 3H), 0.74 (s, 3H); **^{13}C NMR**(125 MHz, $CDCl_3$) δ 178.4, 176.0, 149.6, 105.5, 73.3, 68.9, 55.0, 52.2, 51.9, 51.7, 49.4, 46.7, 41.6, 35.9, 33.2, 33.1, 29.3, 29.0, 22.0, 15.0; **IR** (film) ν 2972, 2951, 1773, 1709, 1647, 1538, 1461, 1368 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{20}H_{30}O_4ClNH_4$ $[M + Na]^+$: 406.1761, found 406.1756. **$[\alpha]^{22}_D$** +50° (*c* = 0.11, CH_3OH), [lit.: $[\alpha]_D^{25}$ +119.3° (c = 1.59, CH_3OH)].



Alcohol S12.

Weinreb amide **S11**[3] (115 mg, 392 μmol) was subjected to the reaction conditions described above for **S9** to provide alcohol **S12** (81.4 mg, 263 μmol, 67% yield) as a thick oil after column chromatography (SiO_2, 80% EtOAc in hexanes).

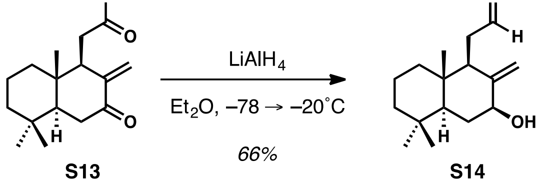
^{1}H NMR (600 MHz, $CDCl_3$) δ 4.96 (s, 1H), 4.56 (s, 1H), 4.36 (t, *J* = 2.3 Hz, 1H), 3.72 (s, 3H), 3.15 (s, 3H), 2.98 (d, *J* = 10.7 Hz, 1H), 2.64 (t, *J* = 5.3 Hz, 1H), 2.45 (dd, *J* = 16.3, 3.4 Hz, 1H), 1.89 (dt, *J* = 13.9, 2.8 Hz, 1H), 1.77 (dd, *J* = 13.2, 2.8 Hz, 1H), 1.59–1.47 (m, 4H), 1.43 (d, *J* = 11.4 Hz, 1H) 1.27–1.18 (m, 2H), 0.90 (s, 3H), 0.82 (s, 3H), 0.71 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 174.4, 150.7, 109.1, 73.5, 61.3, 47.2, 46.2, 41.9, 39.0, 38.6, 33.2, 33.0, 32.4, 30.4, 27.8, 21.6, 19.3, 13.8; **IR** (film) ν 3412, 2928, 1644, 1459, 1441, 1387 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{18}H_{31}NO_3Na$ $[M + Na]^+$: 332.2202; found 332.2202; **$[\alpha]^{25}_D$** –29.9° (*c* = 0.46, $CHCl_3$).



Enone S13.

Alcohol **S12** (140 mg, 450 μmol) was subjected to the reaction conditions described for **41** to provide alcohol **S13** (137 mg, 445 μmol, 98% yield) as an oil after flash column chromatography (SiO_2, 40% EtOAc in hexanes).

^{1}H NMR (600 MHz, $CDCl_3$) δ 5.94 (s, 1H), 5.05 (s, 1H), 3.71 (s, 3H), 3.15 (s, 3H), 2.92 (s, 1H), 1.71–1.66 (m, 2H), 1.60, (dt, *J* = 13.6, 3.2 Hz, 1H), 1.57 (m, 1H), 1.48 (d, *J* = 13.4 Hz, 1H), 1.27–1.19 (m, 3H), 0.89 (s, 6H), 0.85 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 202.0, 173.6, 147.9, 119.7, 61.4, 60.3, 50.7, 50.1, 41.5, 38.5, 38.1, 37.1, 33.4, 32.5, 29.3, 28.7, 20.9, 18.8, 14.4; **IR** (film) ν 2927, 1693, 1664, 1607, 1460.1, 1414, 1387 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{18}H_{29}NO_3Na$ $[M + Na]^+$: 330.2045; found 330.2038; **$[\alpha]^{23}_D$** –41.8° (*c* = 0.45, $CHCl_3$).

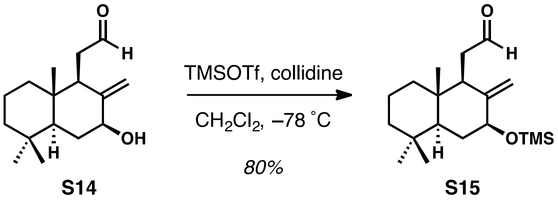


Aldehyde S14.

Enone **S13** (20 mg, 65 μmol, 1 equiv) was azeotroped in a 1 dram vial from benzene (30 μL) then taken up in Et_2O (650 μL, 0.1 M). The clear solution was cooled to –78 °C to give a cloudy white suspension. A solution of $LiAlH_4$ (1M in Et_2O, 130 μmol, 130 μL, 2 equiv) was added dropwise down the side of the vial. After 3 h at –78 °C, no starting material remained by TLC analysis and 50 μL of EtOAc was added. The reaction mixture was warmed to –10 °C and water (10 μL) and 1M aq. NaOH (10 μL) were added sequentially followed by warming to room temperature. The reaction mixture was poured into ice water (5 mL) and extracted with (3 x 4 mL Et_2O). The combined organic phases were dried over $MgSO_4$, filtered, and concentrated. The crude substance was purified by flash column chromatography (SiO_2, 25% EtOAc in hexanes) to aldehyde give **S14** as a clear oil (10.8 mg, 43 μmol, 66% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 5.18 (s, 1H), 4.58 (s, 1H), 4.09 (m, 1H), 2.56 (ddd, *J* = 16.8, 10.6, 3.1 Hz, 1H), 2.48 (dd, *J* = 16.8, 4.0 Hz, 1H), 2.14–2.11 (m, 1H), 1.77 (d, *J* = 5.3 Hz, 1H), 1.76–1.59 (m, 1H), 1.54–

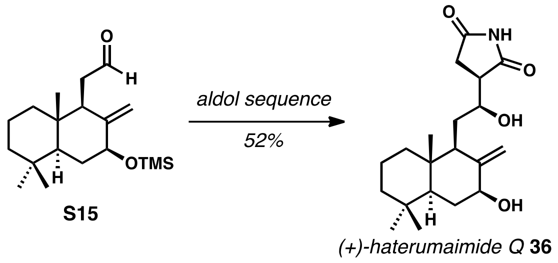
1.50 (m, 1H), 1.45 (d, *J* = 13.0 Hz, 1H), 1.34–1.26 (m, 2H), 1.22 (td, *J* = 13.1, 4.0 Hz, 1H), 1.06 (td, *J* = 12.6, 3.9 Hz, 1H), 0.93 (s, 3H), 0.83 (s, 3H), 0.70 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 202.8, 150.5, 104.8, 73.4, 52.8, 49.0, 41.8, 39.6, 39.1, 38.6, 33.45, 33.42, 21.6, 19.1, 14.5; **IR** (film) ν 3422, 2924, 2846, 2721, 1722, 1649, 1460, 1388 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{16}H_{26}O_2Na$ [M + Na]$^+$: 273.1830; found 273.1827; **[α]$^{22}_{D}$** –15.3° (*c* = 0.2, $CHCl_3$)



TMS ether S15.

Aldehyde **S14** (9.0 mg, 36 μmol) was subjected to the reaction conditions described for **42** and afforded TMS ether **S15** as a thin film (9.2 mg, 28 μmol, 80% yield) after flash column chromatrography (SiO_2, 5% EtOAc in hexanes).

^{1}H NMR (600 MHz, $CDCl_3$) δ 9.63 (s, 1H), 5.21 (s, 1H), 4.54 (s, 1H), 4.02 (m, 1H), 2.53 (ddd, *J* = 19.9, 10.5, 3.12 Hz, 1H), 2.45 (dd, *J* = 16.7, 4.0 Hz, 1H), 2.28 (d, *J* = 10.5, 1H) 1.92 (app. d, *J* = 10, 1H) 1.59–1.56 (m, 1H), 1.55–1.48 (m, 2H), 1.44 (d, *J* = 13.2 Hz, 1H), 1.36 (q, *J* = 12.6 Hz, 1H), 1.25–1.16 (m, 2H), 1.04 (t, *J* = 12.9 Hz, 1H), 0.89 (s, 3H), 0.82 (s, 3H), 0.69 (s, 3H), 0.13 (s, 9H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 203.0, 149.7, 105.9, 73.9, 52.9, 49.2, 41.8, 39.6, 39.2, 38.6, 34.2, 33.4, 29.5, 21.6, 19.2, 14.5, –0.10; **IR** (film) ν 2951, 2845, 2712, 1727, 1649, 1459, 1389, 1250 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{19}H_{34}O_2SiNa$ [M + Na]$^+$: 345.2226; found 345.2229; **[α]$_{D}^{25}$** -10.4° (*c* = 0.38, $CHCl_3$).

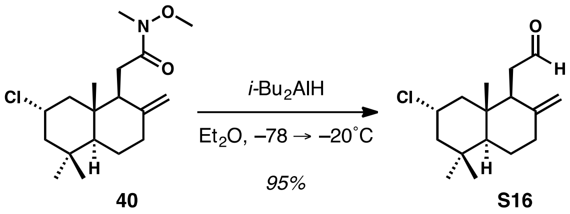


Haterumaimide Q 36.

Aldehyde **S15** (9.3 mg, 30 μmol) was subjected to the reaction conditions described for **34** and afforded haterumaimide Q (**36**) as a thin film (5.5 mg, 18 μmol, 52% yield) after flash column chromatography (SiO_2, 60% EtOAc in hexanes). The data obtained for our synthetic sample of **36** matched those reported by Ueda.[19]

For ^{1}H NMR and ^{13}C NMR data comparison to natural **3** in $(CD_3)_3SO$ see Table S19, below.

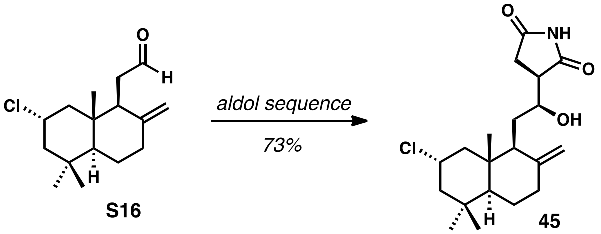
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.78, (s, 1H), 5.30 (s, 1H), 4.86 (s, 1H), 4.36 (t, *J* = 7.0 Hz, 1H), 3.99 (dd, *J* = 11.1, 5.4 Hz, 1H), 2.92–2.89 (m, 1H, H_{13}), 2.87 (dd, *J* = 16.5, 5.5 Hz, 1H), 2.67 (dd, *J* = 16.5, 7.6 Hz, 1H), 2.15–2.09 (m, 1H), 1.93 (s, 1H), 1.84–1.76 (m, 1H), 1.59–1.52 (m, 2H), 1.50 (d, *J* = 11.3 Hz, 1H), 1.45 (d, *J* = 13.1 Hz, 1H), 1.17 (td, *J* = 13.1, 4.1 Hz, 1H), 1.14 (td, *J* = 13.1, 2.4 Hz, 1H), 0.95–0.85 (m, 1H), 0.89 (s, 3H), 0.81 (s, 3H), 0.68 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 178.6, 176.2, 150.8, 104.3, 73.8, 69.1, 53.3, 52.0, 46.8, 41.9, 39.5, 39.1, 33.8, 33.6, 33.5, 29.4, 29.0, 21.6, 19.3, 14.5; IR (film) cm^{-1} ; **HRMS** (ES+) *m/z* calc'd for $C_{20}H_{31}O_4NNa$ $[M + Na]^+$: 372.2151, found 373.2145; **$[\alpha]_D^{25}$** +31.1° (*c* = 0.040, $CHCl_3$) [lit.: $[\alpha]_D^{25}$ +36.0° (c = 0.19, $CHCl_3$)]



Aldehyde S16.

Alkene **40** (22 mg, 67.2 μmol, 1.0 equiv) was dissolved in Et_2O (1.3 mL, 0.05 M) and the solution was cooled to –78˚C. A solution of *i*Bu_2AlH in toluene (1.0 M, 134 μL, 134 μmol, 2.0 equiv) was added slowly along the side of the flask. After 2 h at –78 ˚C, the flask was warmed to –20 ˚C and quenched with 50 μL of acetone followed by water (200 μL). The biphasic solution was warmed to ambient temperature and diluted with more 1M HCl (1 mL) and water (2 mL). The aqueous phase was extracted with Et_2O (3 x 5 mL) and the combined organic extracts were dried ($MgSO_4$), filtered and concentrated under reduced pressure. The crude oil was purified by chromatography (SiO_2, 5% EtOAc in hexanes) and afforded **S16** a clear oil (17.2 mg, 64 μmol, 95% yield).

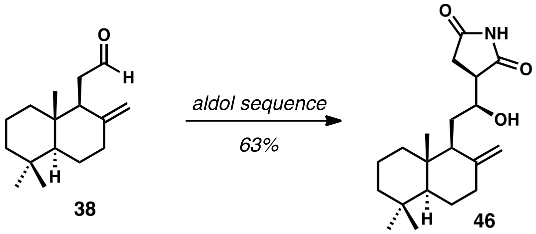
^{1}H NMR (600 MHz, $CDCl_3$) δ 9.65 (d, *J* = 3.2 Hz, 1H), 4.87 (s, 1H), 4.42 (s, 1H), 4.16 (tt, *J* = 12.6, 3.9 Hz, 1H), 2.54 (ddd, *J* = 17.2, 11.8, 3.3 Hz, 1H), 2.41–2.47 (m, 3H), 2.07–2.13 (m, 2H), 2.00 (ddd, *J* = 12.8, 3.8, 2.2 Hz, 1H), 1.74–1.79 (m, 1H), 1.56 (t, *J* = 12.6 Hz, 1H), 1.45 (t, *J* = 12.2 Hz, 1H), 1.32 (qd, *J* = 12.7, 4.1 Hz, 1H), 1.26 (dd, *J* = 12.6, 2.3 Hz, 1H), 1.24 (s, 3H), 0.88 (s, 3H), 0.76 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 202.4, 147.1, 109.2, 55.4, 54.1, 52.0, 50.5, 49.3, 41.0, 39.6, 37.1, 36.1, 33.3, 23.3, 22.2, 15.1; IR (film) ν 2960, 2853, 2714, 1726, 1345, 1460, 1390, 1339 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{16}H_{25}OClNH_4$ $[M + NH_4]^+$: 286.1938, found 286.1945; $[\alpha]_D^{25}$ –1.0° (*c* = 0.52, $CHCl_3$).



7-Deoxychlorolissoclimide 45.

Aldehyde **S16** (13 mg, 48.4 μmol) was subjected to the reaction conditions described for **34** to yield **45** (13.1 mg, 35.0 μmol, 73% yield) after column chromatography (SiO_2, 55% EtOAc in hexanes) as a clear oil.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (s, 1H), 4.97 (s, 1H), 4.36 (s, 1H), 4.16 (tt, *J* = 12.2, 3.9 Hz, 1H), 2.89–2.94 (m, 1H), 2.89 (dd, *J* = 17.4, 5.3 Hz, 1H), 2.70 (dd, *J* = 17.3, 8.6 Hz, 1H), 2.45 (ap d, *J* = 13.0 Hz, 1H), 2.23 (d, *J* = 11.4 Hz, 1H), 1.96–2.03 (m, 2H), 1.92 (d, *J* = 3.6 Hz, 1H), 1.72–1.80 (m, 2H), 1.68 (d, *J* = 10.8 Hz, 1H), 1.59 (dd, *J* = 14.0, 7.9 Hz, 1H), 1.52 (t, *J* = 12.8 Hz, 1H), 1.30 (qd, *J* = 15.1, 4.3 Hz, 1H), 1.28 (t, *J* = 11.8 Hz, 1H), 1.13 (dd, *J* = 12.5, 2.5 Hz, 1H), 0.96 (s, 3H), 0.86 (s, 3H), 0.75 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 178.6, 176.2, 147.7, 108.6, 77.2, 69.3, 55.4, 54.6, 53.6, 52.0, 49.6, 46.8, 42.0, 37.8, 36.1, 33.3, 29.4, 29.2, 23.7, 22.1, 15.0; **IR** (film) ν 3500, 2958, 2936, 2853, 1776, 1712, 1463, 1388, 1186 cm^{-1}; **HRMS** (ES+) *m/z* calc'd for $C_{20}H_{30}ClNO_3Na$ $[M + Na]^+$: 390.1812, found 390.1820; $[\alpha]_D^{25}$ +64.3 (*c* = 0.19, $CHCl_3$).



7-Deoxyhaterumaimide Q 46.

Known aldehyde **38**^{3} (11.7 mg, 50.0 μmol) was subject to the reaction conditions described for **34** and chromatographically purified (SiO_2, 50% EtOAc in hexanes) to yield **46** (11.0 mg, 33 μmol, 63% yield) as a clear oil. The characterization data were consistent with previously reported data.[20]

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 (s, 1H), 4.91 (s, 1H), 4.67 (s, 1H), 4.35 (ap t, *J* = 6.9 Hz, 1H), 2.93–2.98 (m, 1H), 2.87 (dd, *J* = 17.7, 5.3 Hz, 1H), 2.67 (dd, *J* = 17.7, 9.0 Hz, 1H), 2.42 (ddd, *J* = 12.9, 4.1, 2.5

Hz, 1H), 2.03–1.95 (m, 2H), 1.79–1.66 (m, 3H), 1.65–1.55 (m, 2H), 1.52 (tt, *J* = 13.0, 3.6 Hz, 1H), 1.41 (ap d, *J* = 13.3 Hz, 1H), 1.33 (qd, *J* = 12.9, 4.4 Hz, 1H), 1.17 (td, *J* = 13.4, 3.8 Hz, 1H), 1.08 (dd, *J* = 12.6, 2.7 Hz, 1H), 0.94 (td, *J* = 12.6, 4.2 Hz, 1H), 0.89–0.84 (m, 1H), 0.88 (s, 3H), 0.80 (s, 3H), 0.69 (s, 3H). **^{13}C NMR** (125 MHz, $CDCl_3$) δ 178.9, 176.5, 149.0, 107.4, 69.5, 55.7, 53.8, 46.8, 42.0, 39.8, 39.2, 38.2, 33.6, 33.5, 29.4, 29.1, 24.3, 21.6, 19.3, 14.4; **IR** (film) ν 3441, 3224, 3079, 2933, 2845, 1774, 1713, 1459, 1364, 1188 cm^{-1}; **HRMS** [ES+] *m/z* calc'd for $C_{20}H_{31}O_3NNa$ [M + Na]$^+$: 356.2202, found 356.2203. **$[\alpha]_D^{22}$** +51.1° (*c* = 0.35, $CHCl_3$) [lit.: $[\alpha]_D^{20}$ +40.6° (c = 0.71, $CHCl_3$)].

Comparison Tables for chlorolissoclimide (1) and haterumaimide Q (2).

Table 1: Synthetic and natural chlorolissoclimide (**1**) in CD_2Cl_2.

Atom	δ C nat. (ppm)	δ C syn. (ppm)	Δ	δ H nat. (ppm)	Multiplicity, *J* (Hz)	δ H syn. (ppm)	Multiplicity, *J* (Hz)	Δ
1	50.1	50.1	-	2.35 1.30	tdd (12.5, 4.0, 2.1) t (12.5)	2.23 1.28	ap d (12.1) t (12.0)	0.12[21] -0.02
2	56.4	56.5	0.1	4.20	tt (12.5, 4.0)	4.20	tt (12.2, 3.9)	-
3	52.8	52.8	-	2.01 1.53	ddd (12.5, 4.0, 2.1) t (12.5)	2.01 1.53	ddd (12.8, 3.9, 2.0) t (12.6)	-
4	36.8	36.7	-0.1	-	-	-	-	-
5	52.9	52.9	-	1.20	s	1.20	s	-
6	34.0	34.0	-	2.10 1.22	s s	2.09 1.22	m s	0.01 -
7	74.1	74.1	-	4.00	d (9.1)	4.00	br s	-
8	150.9	150.9	-	-	-	-	-	-
9	52.3	52.3	-	1.65	dd (11.3, 7.8)	1.63	m (overlapping)	-0.02
10	42.4	42.4	-	-	-	-	-	-
11	29.9	30.0	0.1	1.80 1.60	ddd (14.6, 11.3, 5.6) dt (14.6, 7.8)	1.82 1.60	ddd (15.2, 11.2, 5.6) m (overlapping)	0.02 -
12	69.7	69.7	-	4.32	dddd (7.8, 5.6, 4.0, 2.2)	4.32	m	-
13	47.5	47.5	-	2.90	ddd (9.1, 5.2, 2.2)	2.89	ddd (9.0, 5.1, 2.2)	0.01
14	30.1	30.2	0.1	2.85 2.65	dd (17.7, 5.2) dd (17.7, 9.1)	2.83 2.66	dd (17.8, 5.1) dd (17.8, 9.2)	0.02 0.01
15	180.0	180.3	0.3	-	-	-	-	-
16	177.5	177.8	0.3	-	-	-	-	-
17	105.8	105.9	0.1	5.31 4.92	d (1.6) d (1.6)	5.32 4.91	s s	0.01 -0.01
18	33.8	33.8	-	1.00	s	0.98	s	-0.02
19	22.6	22.7	0.1	0.85	s	0.87	s	0.02
20	15.5	15.6	0.1	0.75	s	0.74	s	-0.01
NH				8.31	br s	8.35	br s	0.04
7-OH				N.O.	-	2.14	d (4.3)	-
12-OH				2.37	br s	2.42	d (2.8)	0.05

Table 2: Synthetic and natural haterumaimide Q (**3**) in (CD$_3$)$_3$SO.

Atom	δ C nat. (ppm)	δ C syn. (ppm)	Δ	δ H nat. (ppm)	Multiplicity, *J* (Hz)	δ H syn. (ppm)	Multiplicity, *J* (Hz)	Δ
1	38.0	38.0	-	1.60 0.91	ddd (13.5, 5.5, 3.0) dd (13.5, 4.0)	1.60 0.91	m td (10.7, 3.7)	- -0.02
2	18.9	18.9	-	1.50 1.42	m m	1.50	m m	-
3	41.5	41.5	-	1.36 1.11	ddd (13.5, 5.5, 3.0) m	1.36 1.53	d (13.0) m	-
4	33.1	33.1	-	-	-	-	-	-
5	52.2	52.2	-	1.12	dd (12.0, 3.5)	1.20	m	-
6	33.5	33.5	-	1.87 1.13	ddd (12.0, 4.4, 3.5) m	1.87 1.22	m m	0.01 -
7	72.0	71.9	-0.1	3.75	ddd (11.0, 5.5, 4.5)	3.76	dd (9.9, 4.8)	0.01
8	151.1	151.1	-	-	-	-	-	-
9	49.8	49.8	-	1.44	dd (10.0, 5.5)	1.63	dd (12.1)	-0.02
10	38.7	38.6	0.1	-	-	-	-	-
11	29.5	29.5	-	1.58 1.40	m m	1.58	m m	-
12	68.8	66.8	2.0	3.99	dddd (9.0, 6.5, 5.0, 2.0)	3.99	m	-
13	45.3	45.3	-	2.80	ddd (8.5, 5.0, 2.0)	2.80	ap t (6.3 Hz)	-
14	28.9	28.9	-	2.52 2.46	dd (17.5, 5.0) dd (17.5, 8.5)	2.45 – 2.61	m	-
15	181.1	181.1	-	-	-	-		-
16	178.8	178.8	-	-	-	-		-
17	103.6	103.6	-	5.18 4.76	s s	5.19 4.76	s s	0.01 -
18	33.3	33.3	-	0.85	s	0.85	s	-
19	21.5	21.5	-	0.75	s	0.75	s	-
20	14.2	14.2	-	0.57	s	0.58	s	0.01
NH				10.99	br s	10.98	br s	-0.01
7-OH				4.91	d (4.5)	4.89	s	-0.02
12-OH				4.92	d (5.0)	4.90	s	-0.02

Cell Viability Assays.

MTS assays were performed for cell viability as described by the supplier (Promega; Madison, WI). Briefly, 5000 cells/well for solid tumor cell lines were seeded in 96-well plates, incubated overnight at 37ºC in 5% (v/v) CO_2 and exposed to compounds in a dose-dependent manner for 48 h. For assays using blood tumor cells, 10000 cells/well were seeded in 96-well plates, followed by treating cells with compounds in a dose-dependent manner for 48h. Dimethyl sulfoxide (DMSO) was used as the vehicle control. Viable cells were determined by tetrazolium conversion to its formazan dye. Absorbance was monitored at 490 nm using an automated ELISA plate reader. IC_{50} values were determined using CalcuSyn software.

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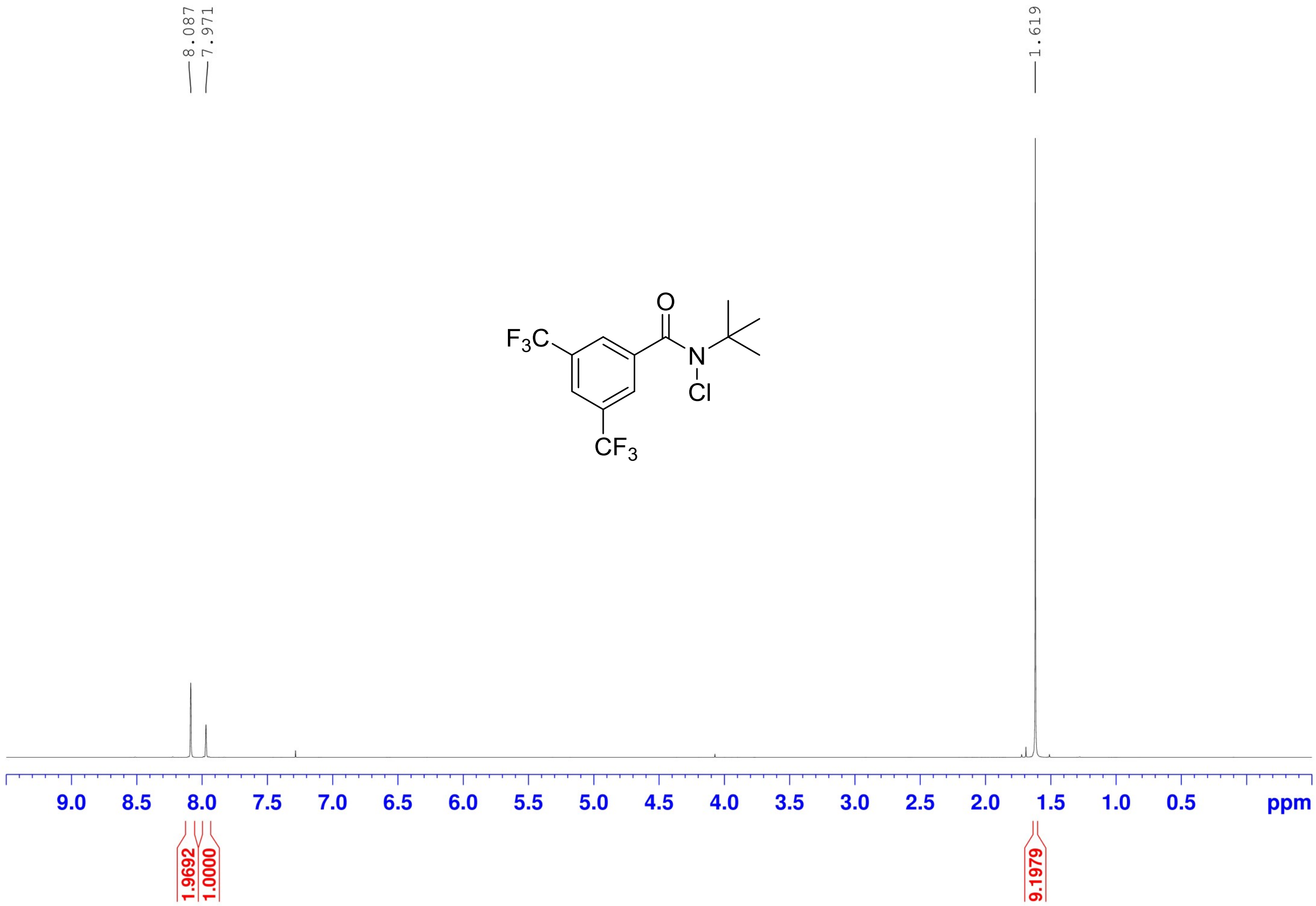
(21) Only peak listings were available for comparison, therefore, we cannot rule out a typographical error in the original report for this resonance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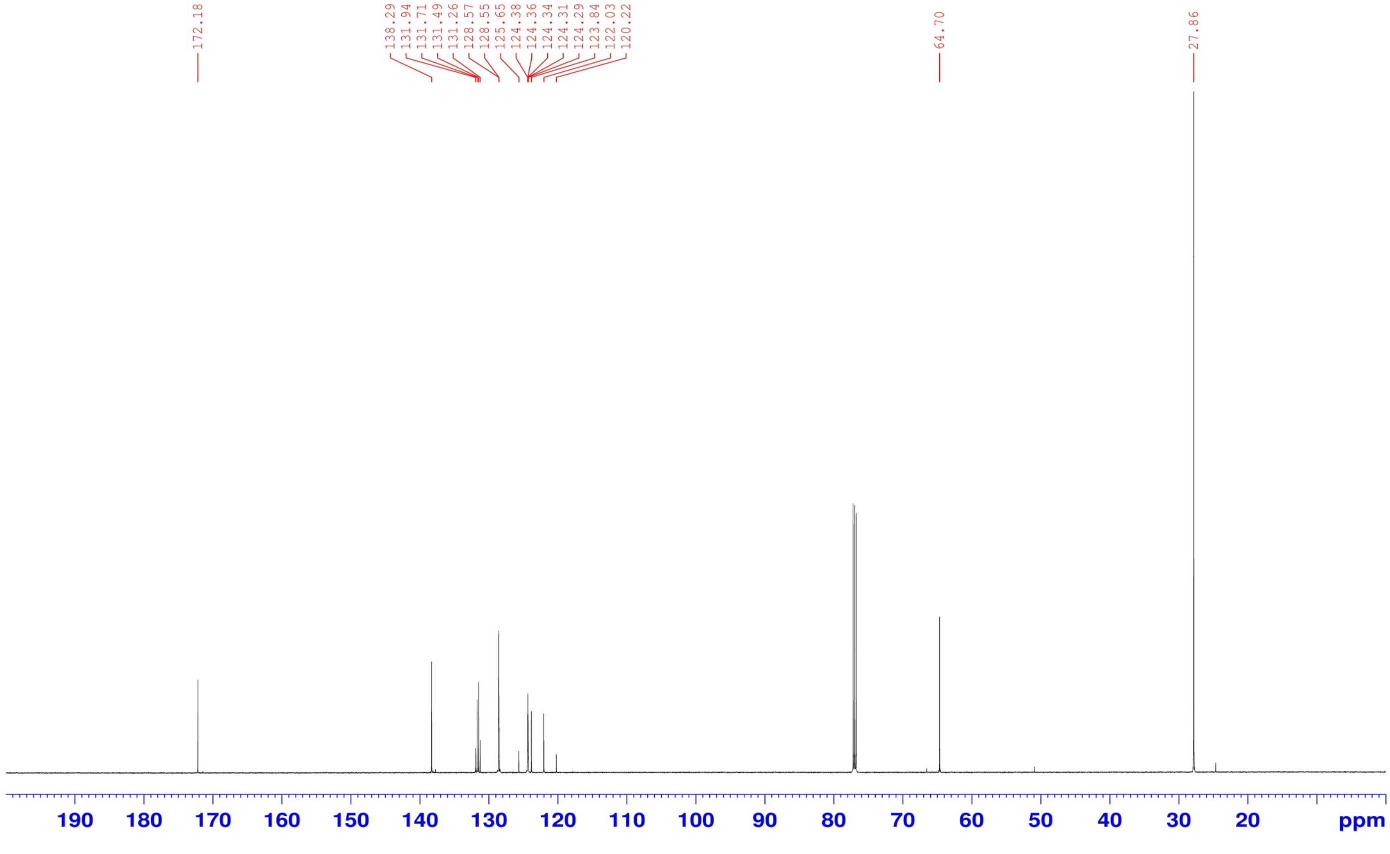
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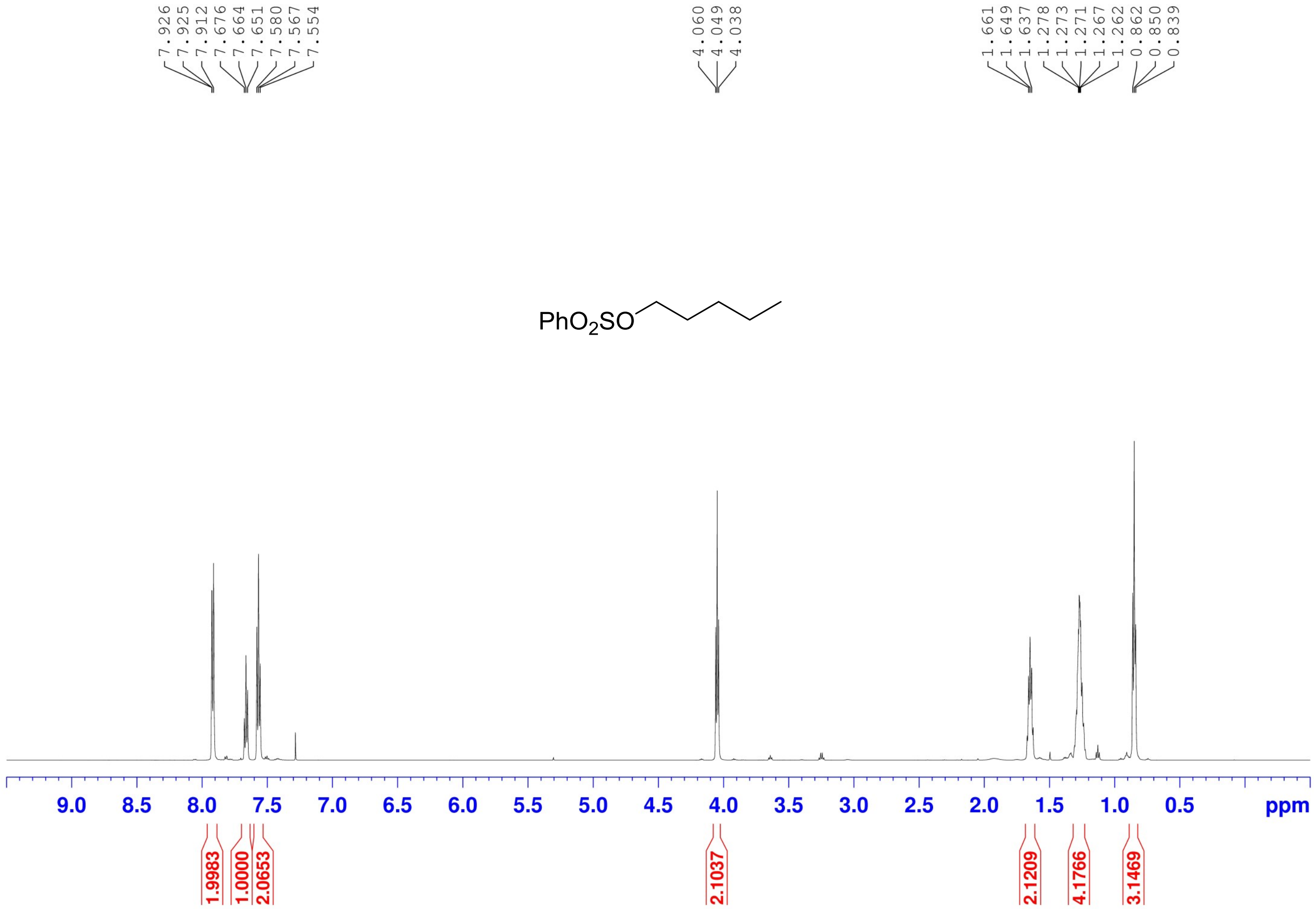
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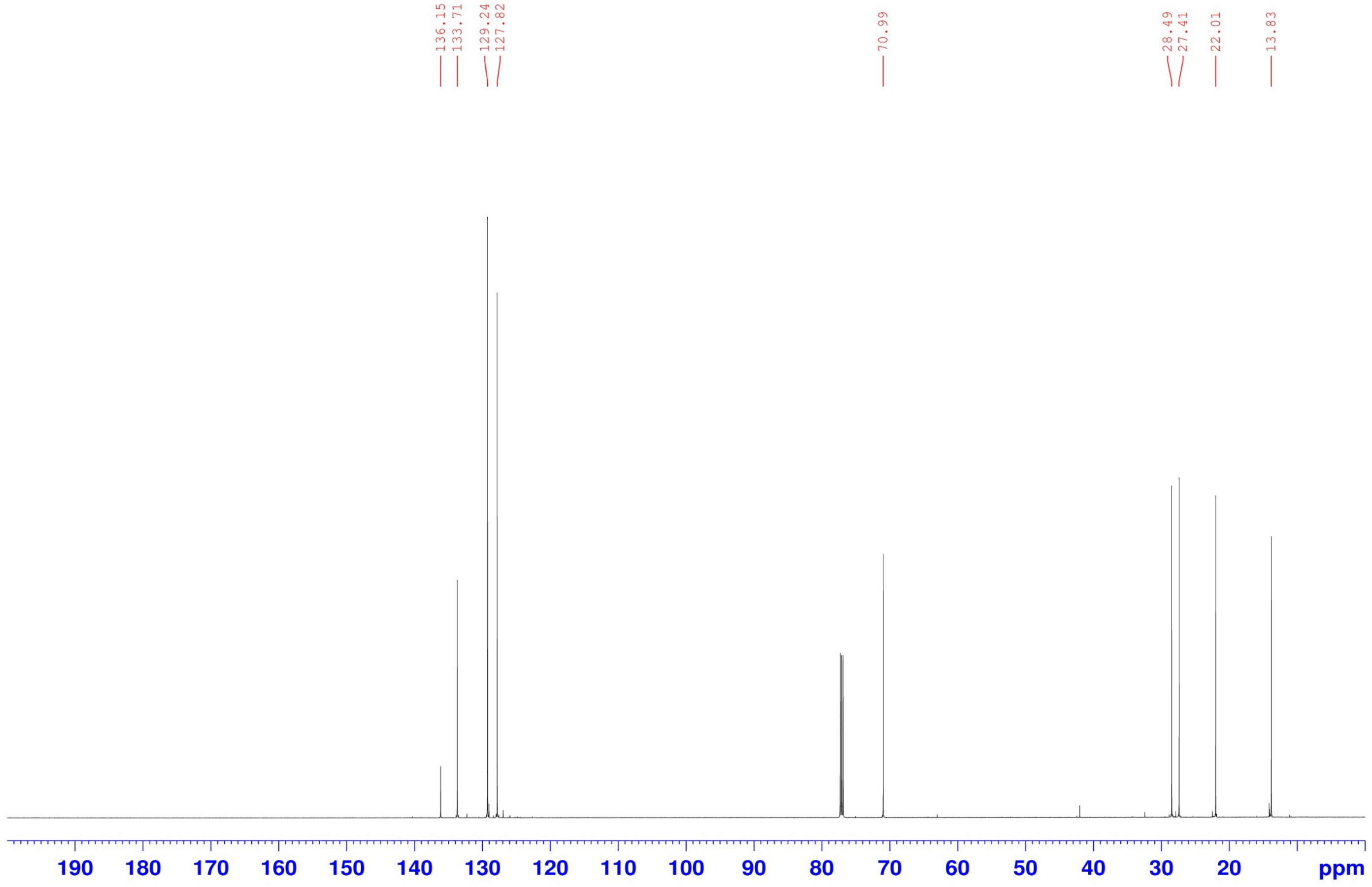
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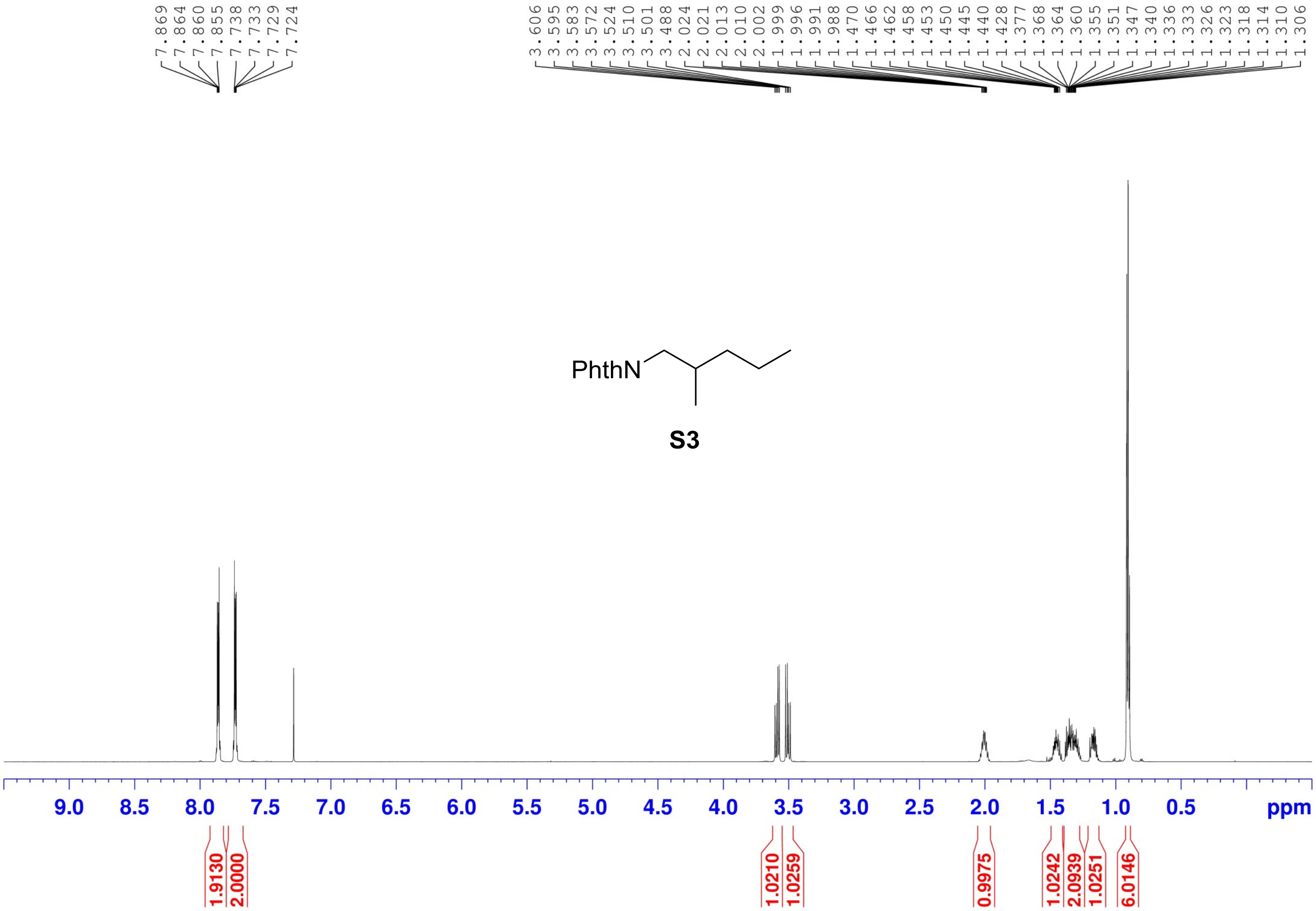
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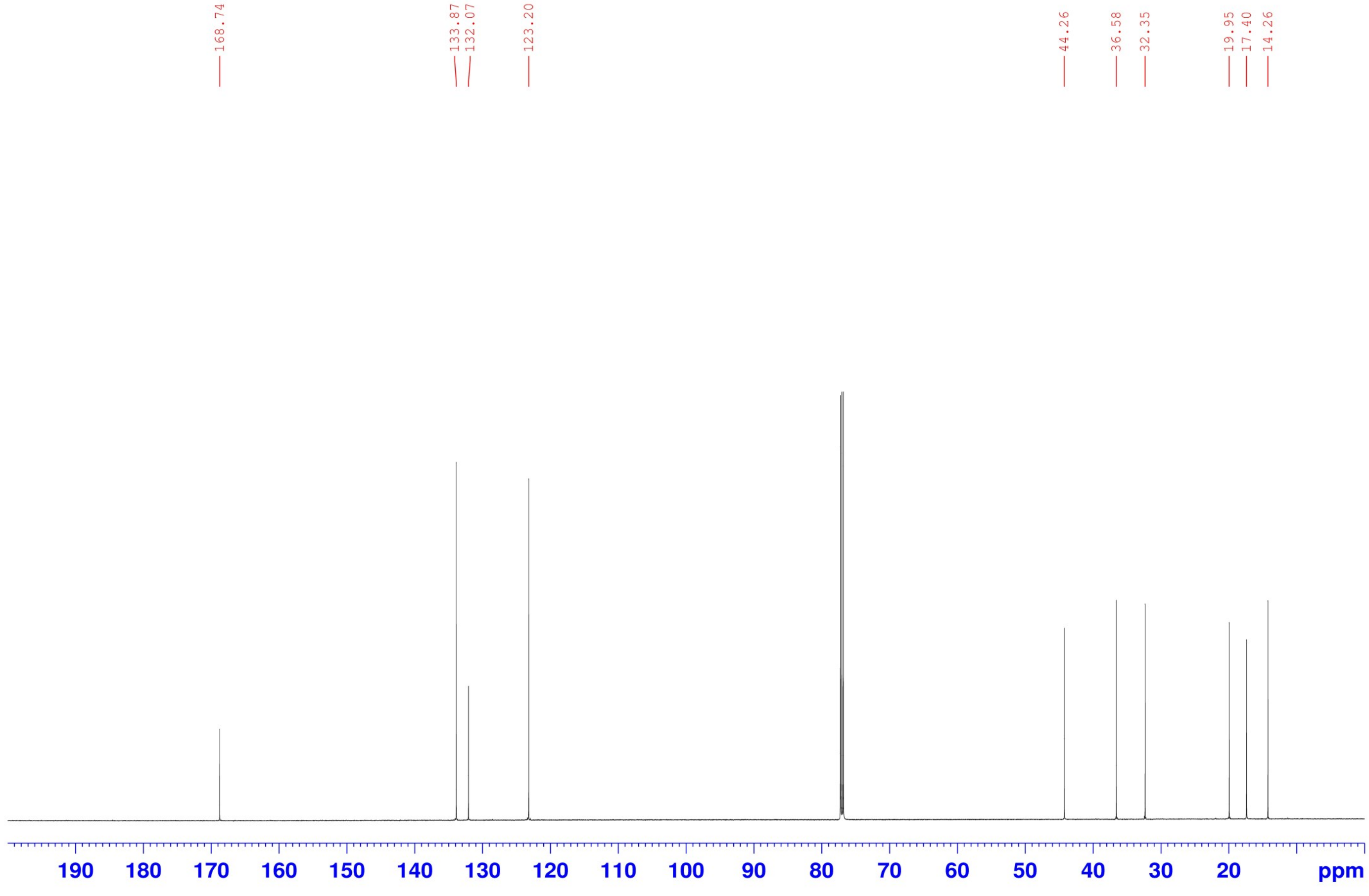
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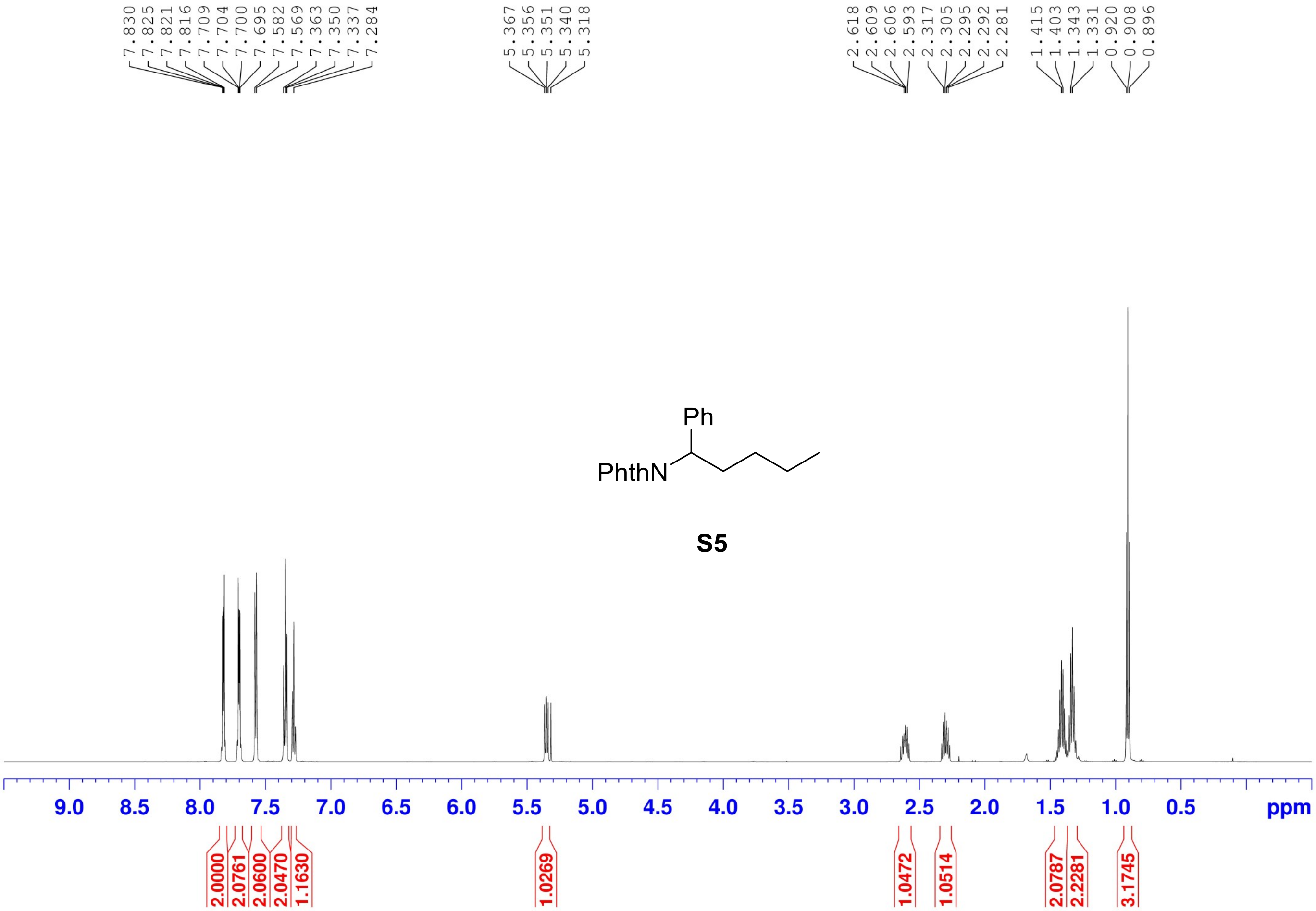
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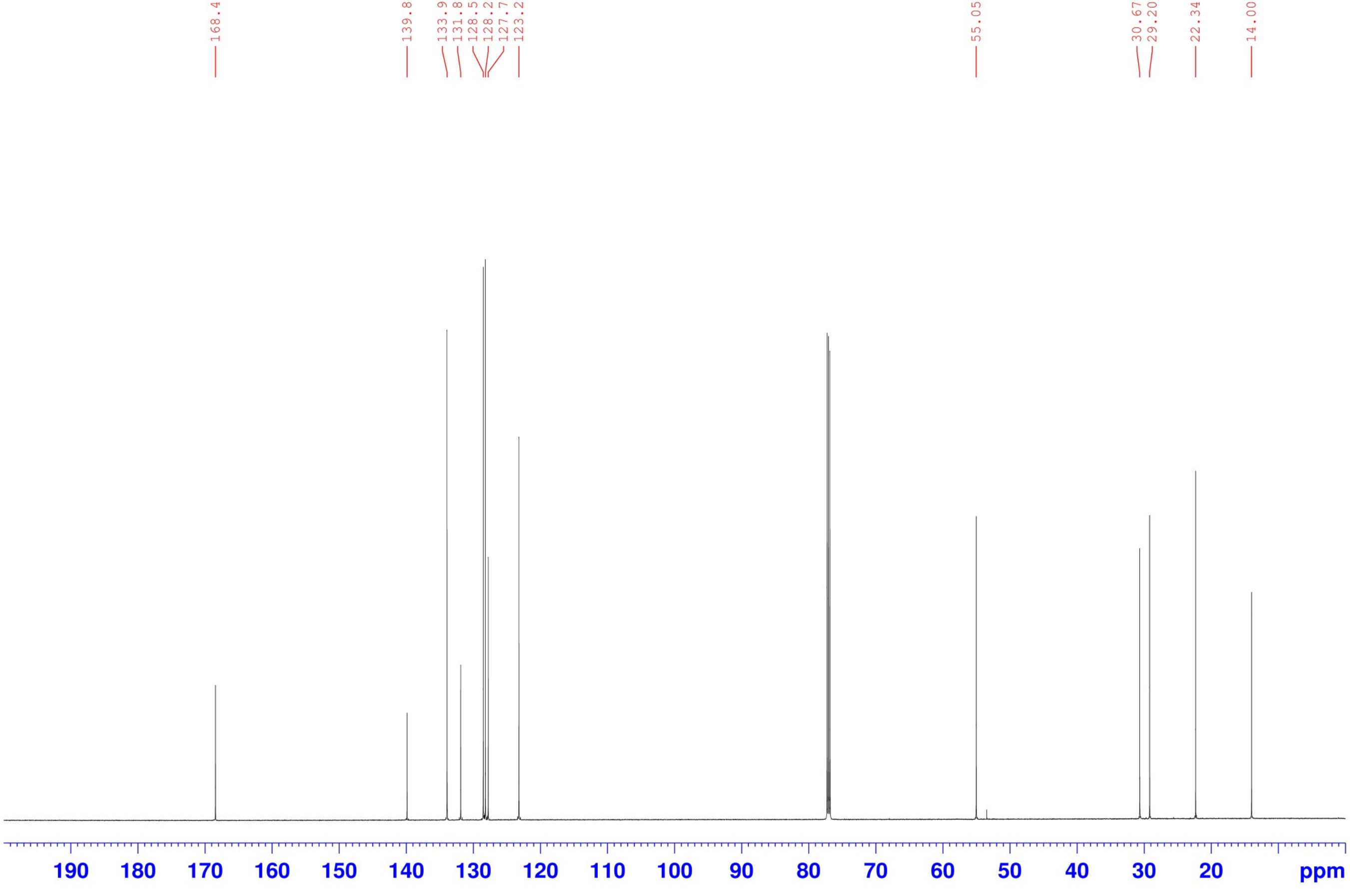
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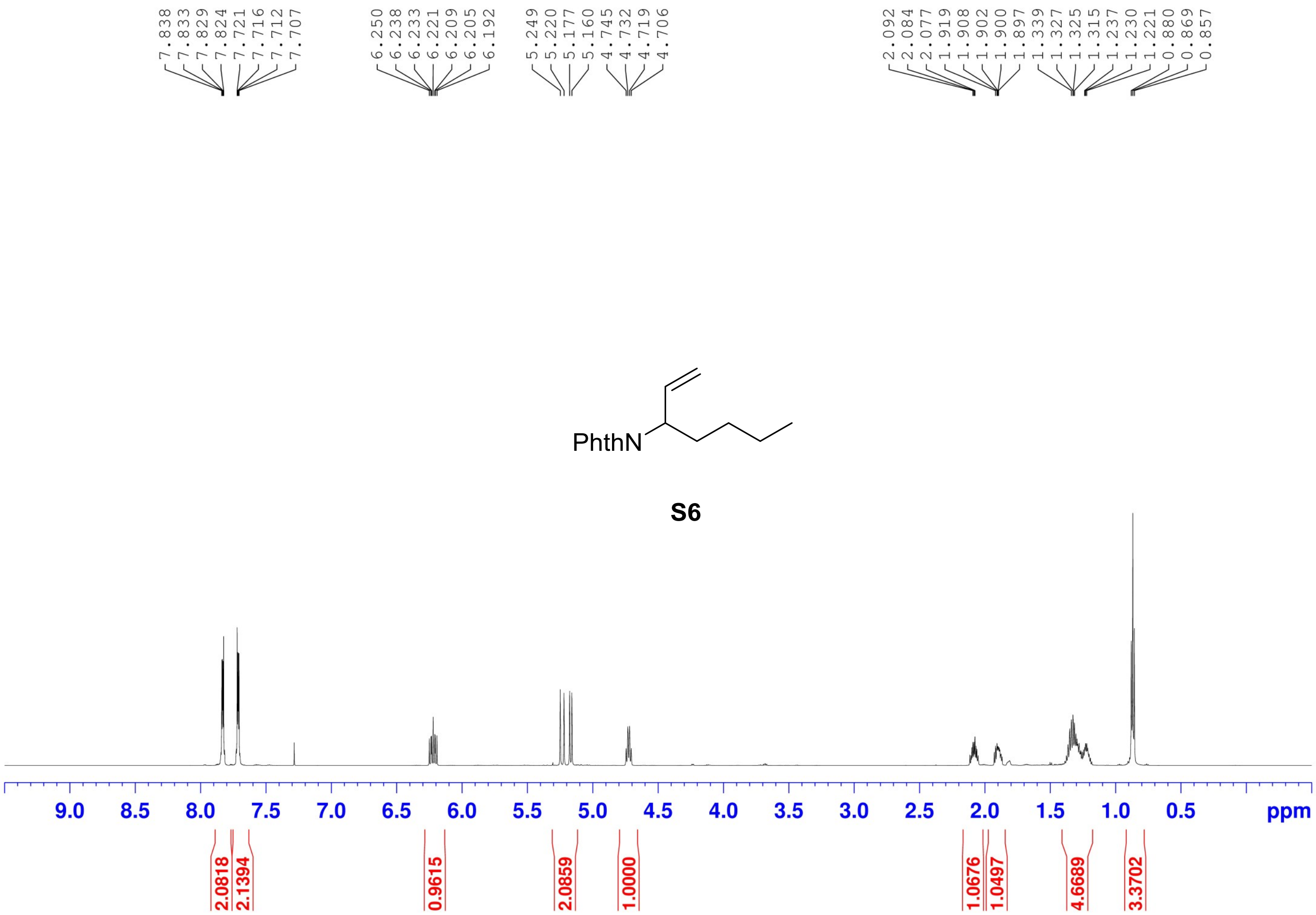

S6

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7.829
7.824
7.721
7.716
7.712
7.707
6.250
6.238
6.233
6.221
6.209
6.205
6.192
5.249
5.220
5.177
5.160
4.745
4.732
4.719
4.706
2.092
2.084
2.077
1.919
1.908
1.902
1.900
1.897
1.339
1.327
1.325
1.315
1.237
1.230
1.221
0.880
0.869
0.857
PhthN
9.0
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8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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0.9615
2.0859
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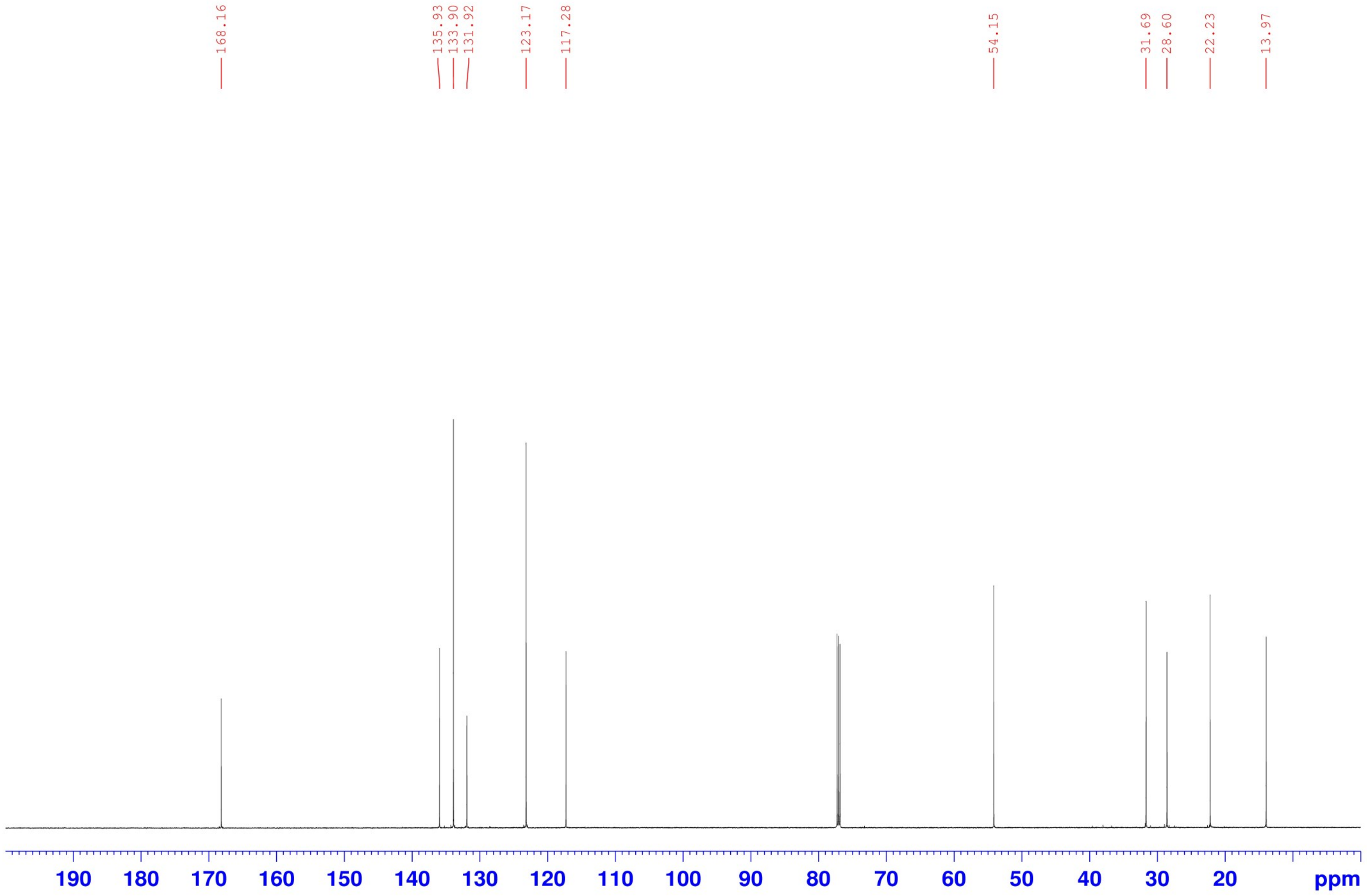
3.3702



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117.28
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22.23
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160
150
140
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80
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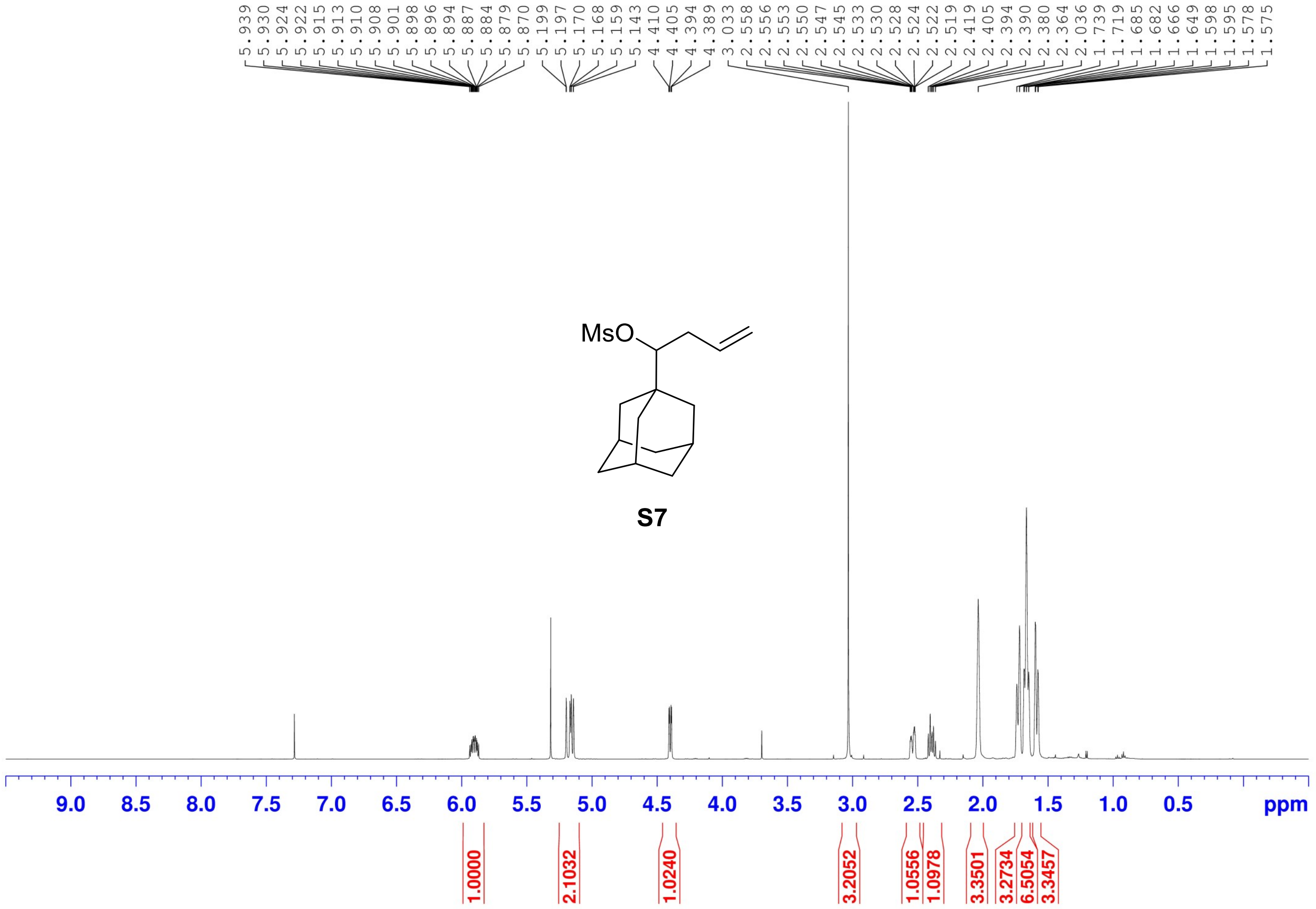
30
20
ppm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5.901
5.898
5.896
5.894
5.887
5.884
5.879
5.870
5.199
5.197
5.170
5.168
5.159
5.143
4.410
4.405
4.394
4.389
3.033
2.558
2.556
2.553
2.550
2.547
2.545
2.533
2.530
2.528
2.524
2.522
2.519
2.419
2.405
2.394
2.390
2.380
2.364
2.036
1.739
1.719
1.685
1.682
1.666
1.649
1.598
1.595
1.578
1.575
MsO
S7
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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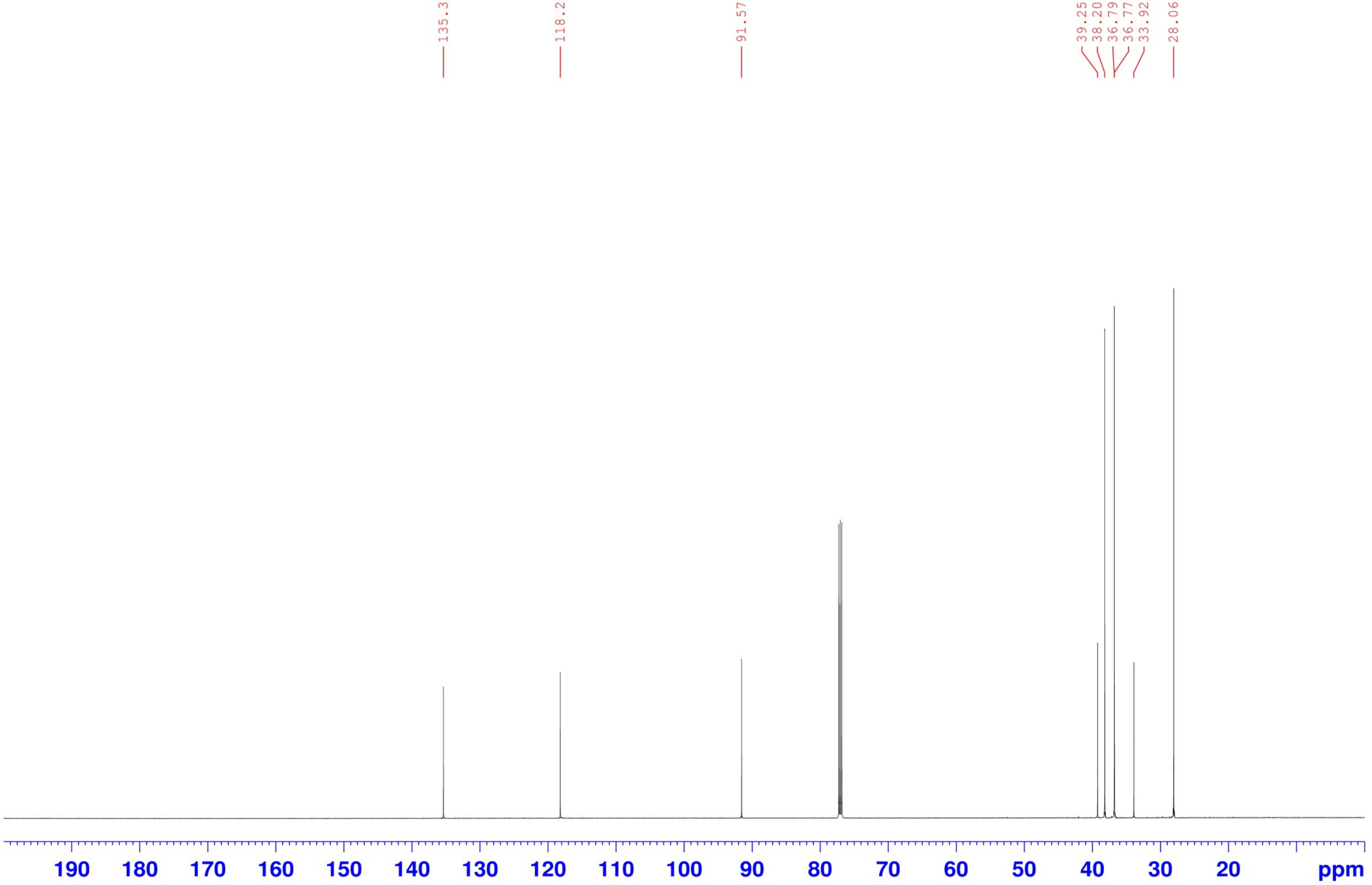
1.0240
3.2052
1.0556
1.0978
3.3501
3.2734
6.5054
3.3457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118.20
91.57
39.25
38.20
36.79
36.77
33.92
28.06
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160
150
140
130
120
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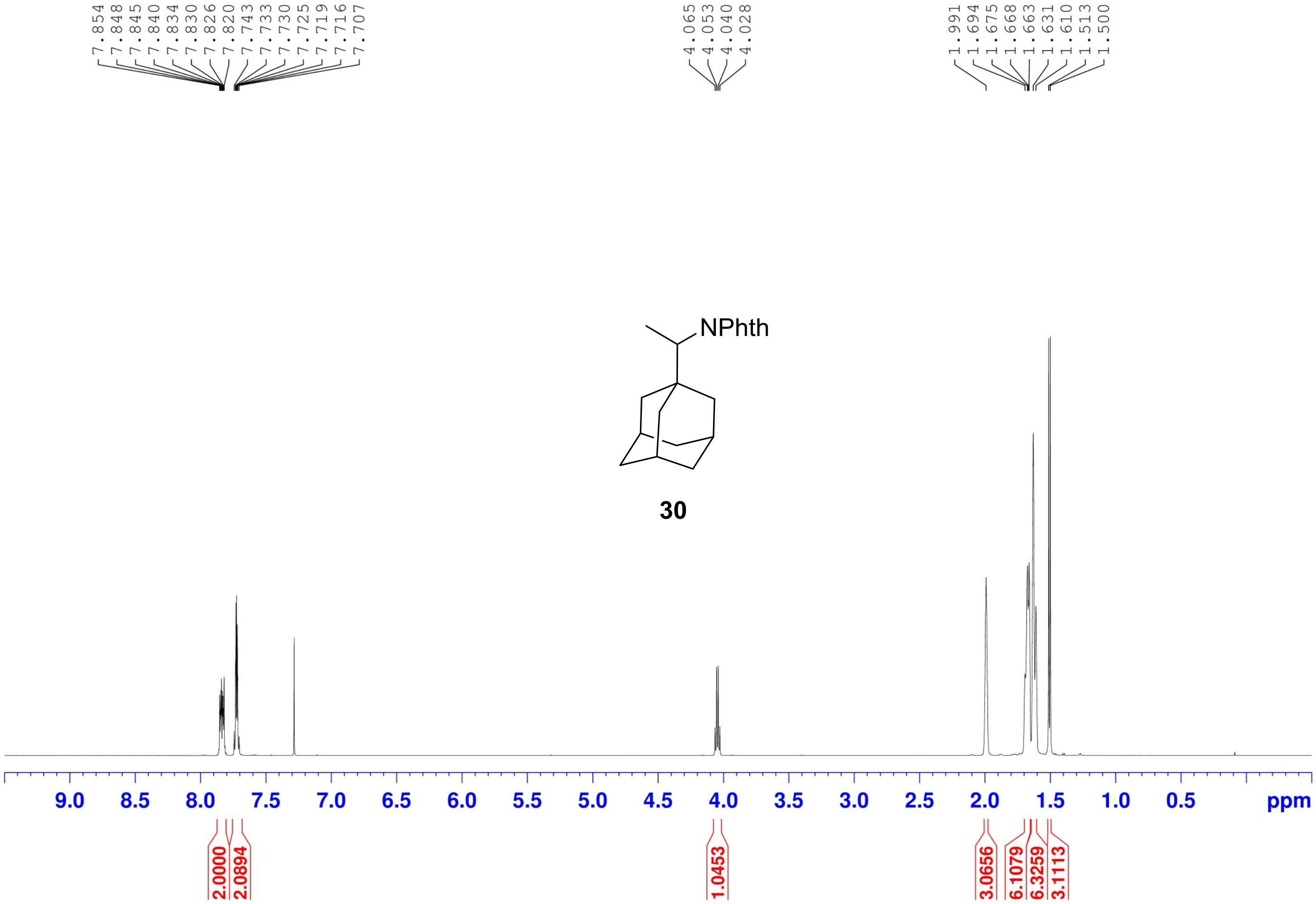
100
90
80
70
60
50
40
30
20
ppm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7.845
7.840
7.834
7.830
7.826
7.820
7.743
7.733
7.730
7.725
7.719
7.716
7.707
4.065
4.053
4.040
4.028
1.991
1.694
1.675
1.668
1.663
1.631
1.610
1.513
1.500
NPhth
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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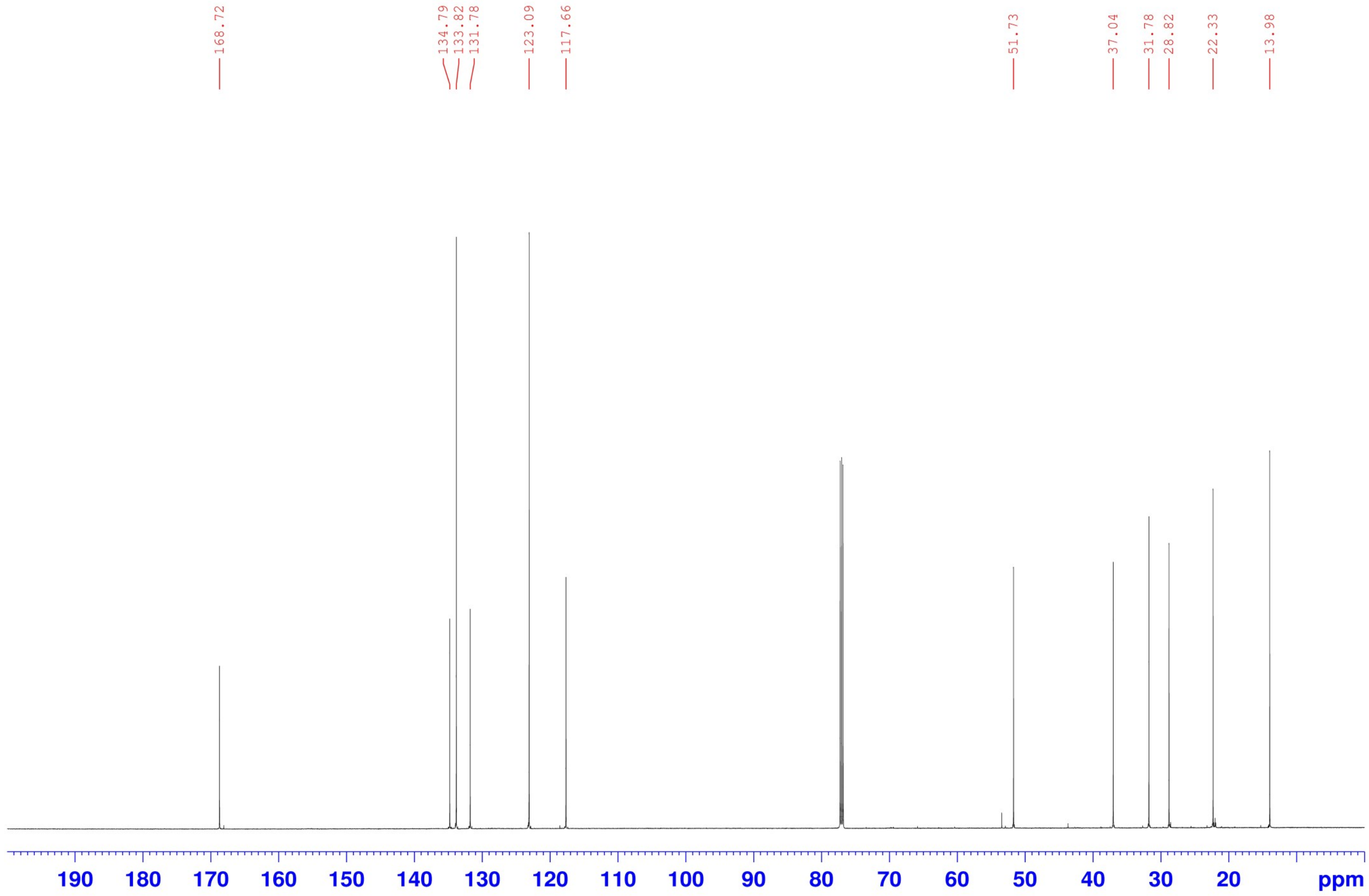
3.0656
6.1079
6.3259
3.1113

30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51.73
37.04
31.78
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22.33
13.98
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180
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160
150
140
130
120
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100
90
80
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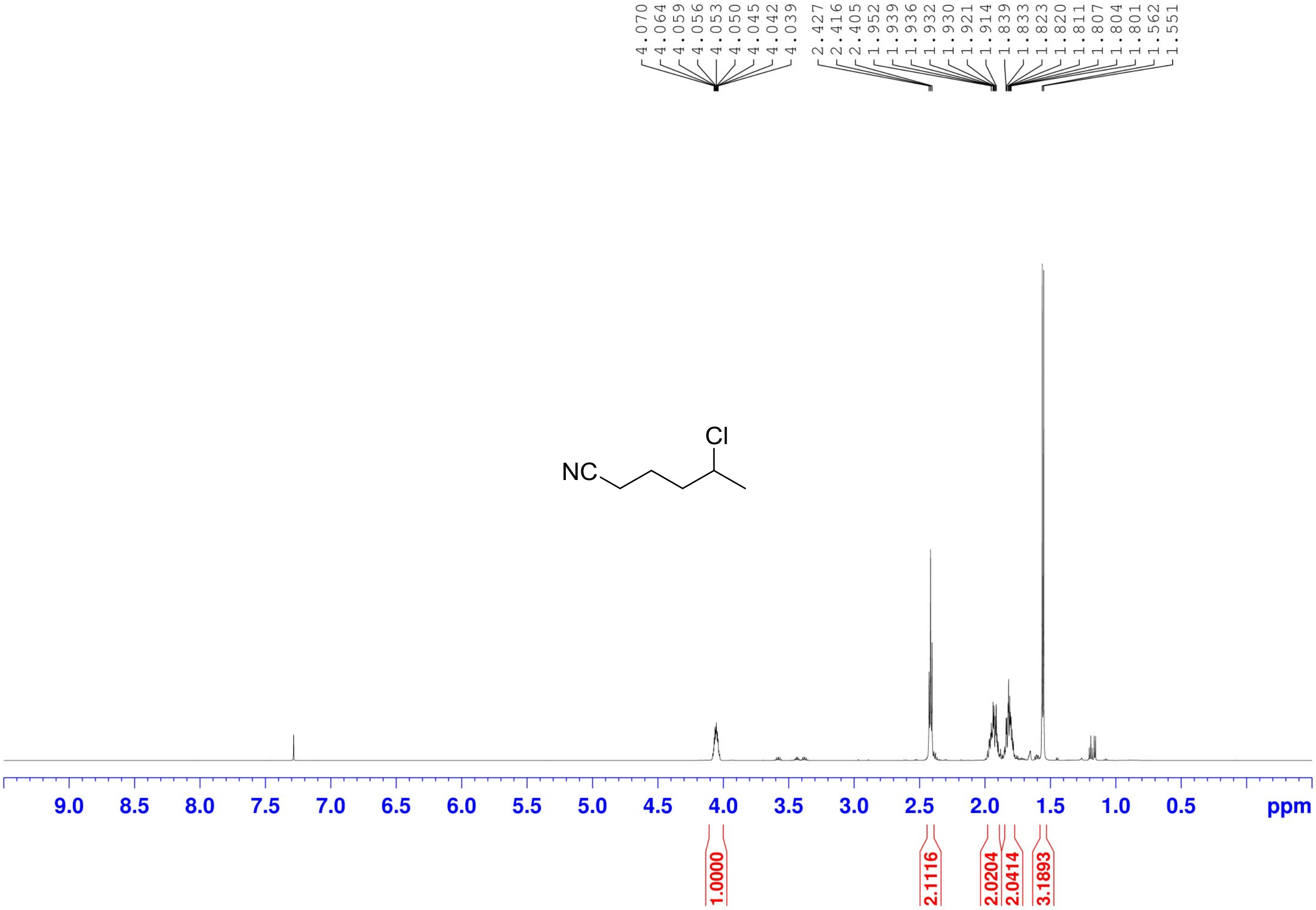
40
30
20
ppm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4.059
4.056
4.053
4.050
4.045
4.042
4.039
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2.416
2.405
1.952
1.939
1.936
1.932
1.930
1.921
1.914
1.839
1.833
1.823
1.820
1.811
1.807
1.804
1.801
1.562
1.551
Cl
NC
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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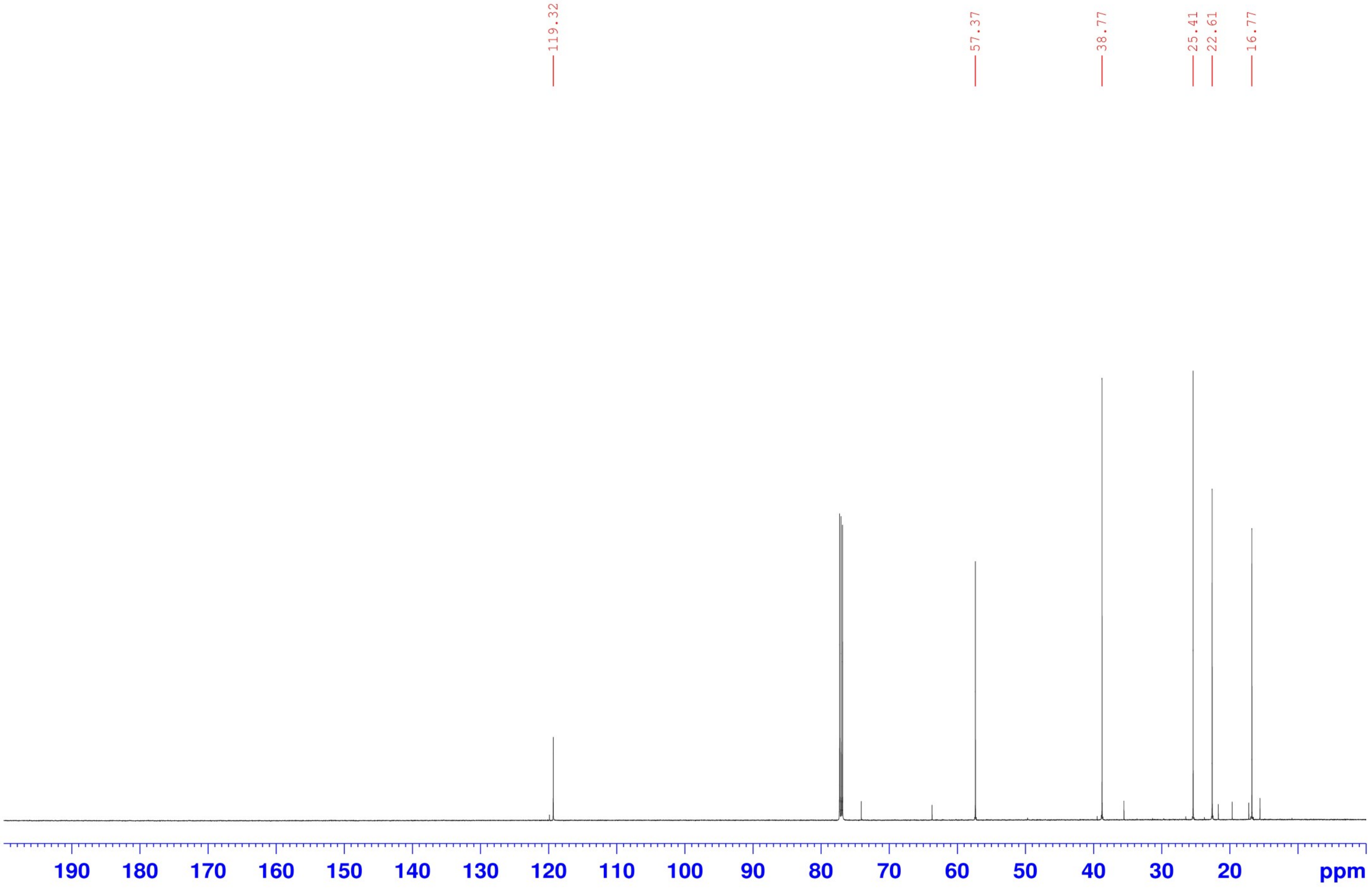
2.1116
2.0204
2.0414
3.1893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57.37
38.77
25.41
22.61
16.77
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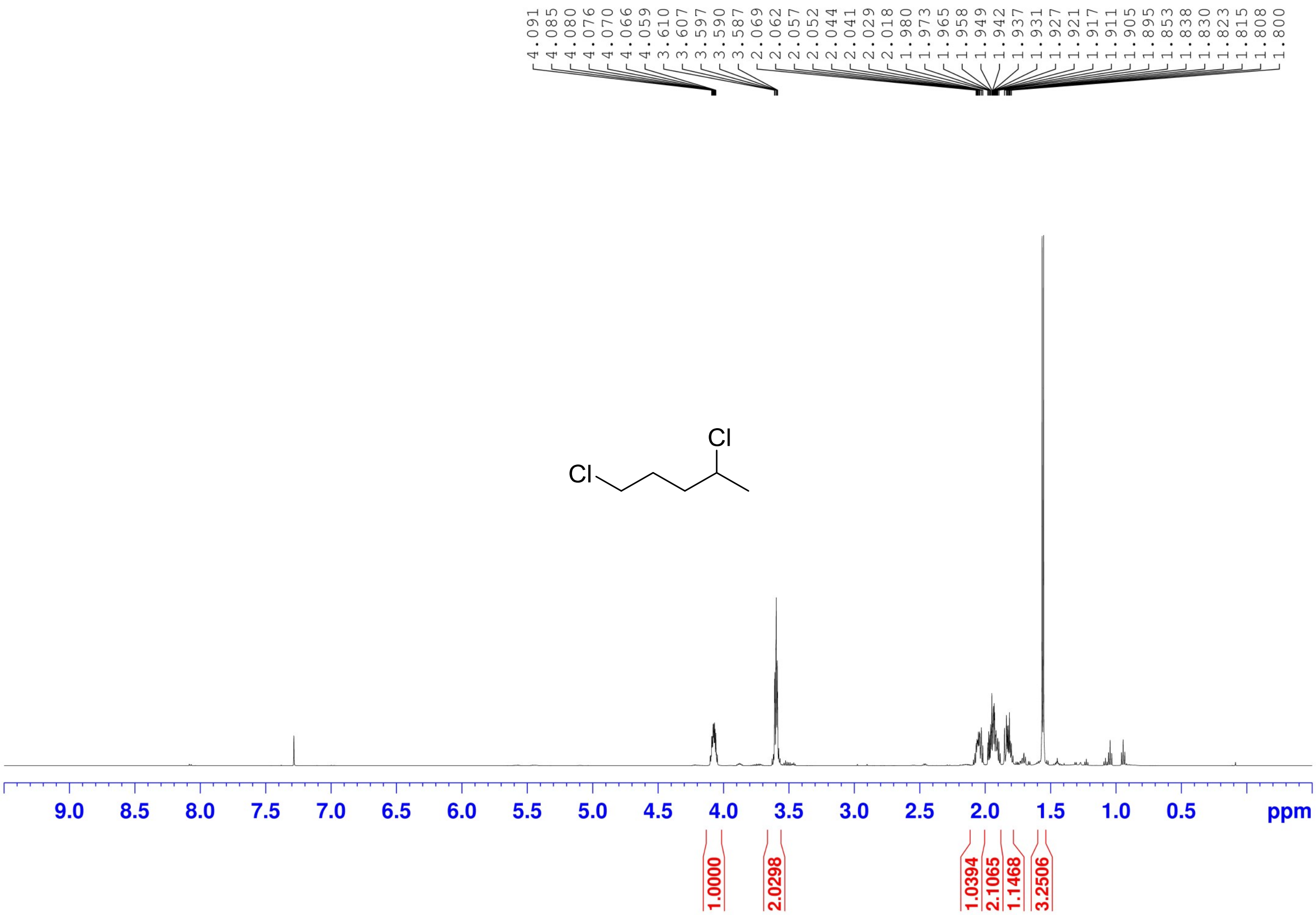
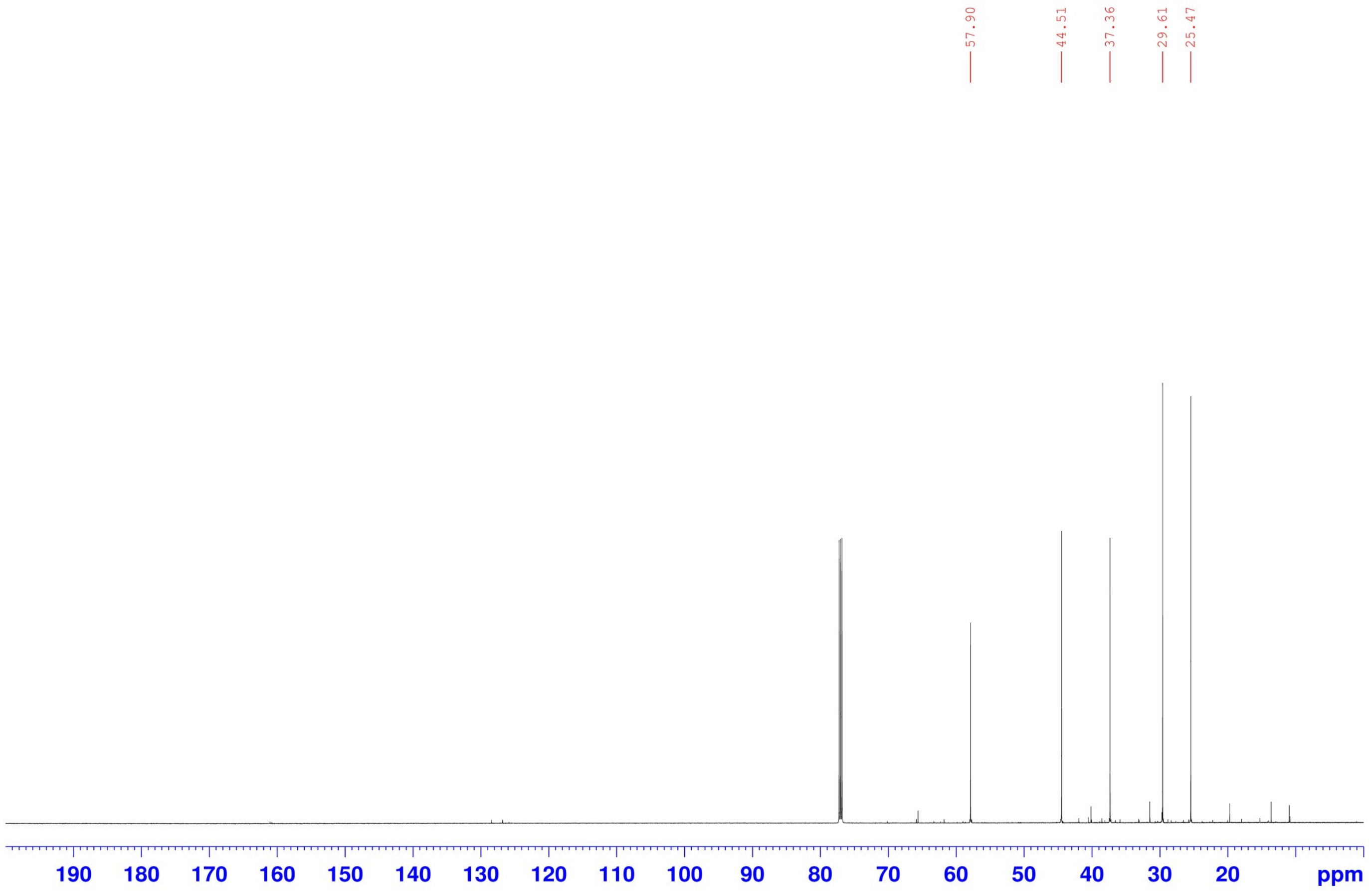
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


4.091
4.085
4.080
4.076
4.070
4.066
4.059
3.610
3.607
3.597
3.590
3.587
2.069
2.062
2.057
2.052
2.044
2.041
2.029
2.018
1.980
1.973
1.965
1.958
1.949
1.942
1.937
1.931
1.927
1.921
1.917
1.911
1.905
1.895
1.853
1.838
1.830
1.823
1.815
1.808
1.800
Cl
Cl
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.0000
2.0298
1.0394
2.1065
1.1468
3.2506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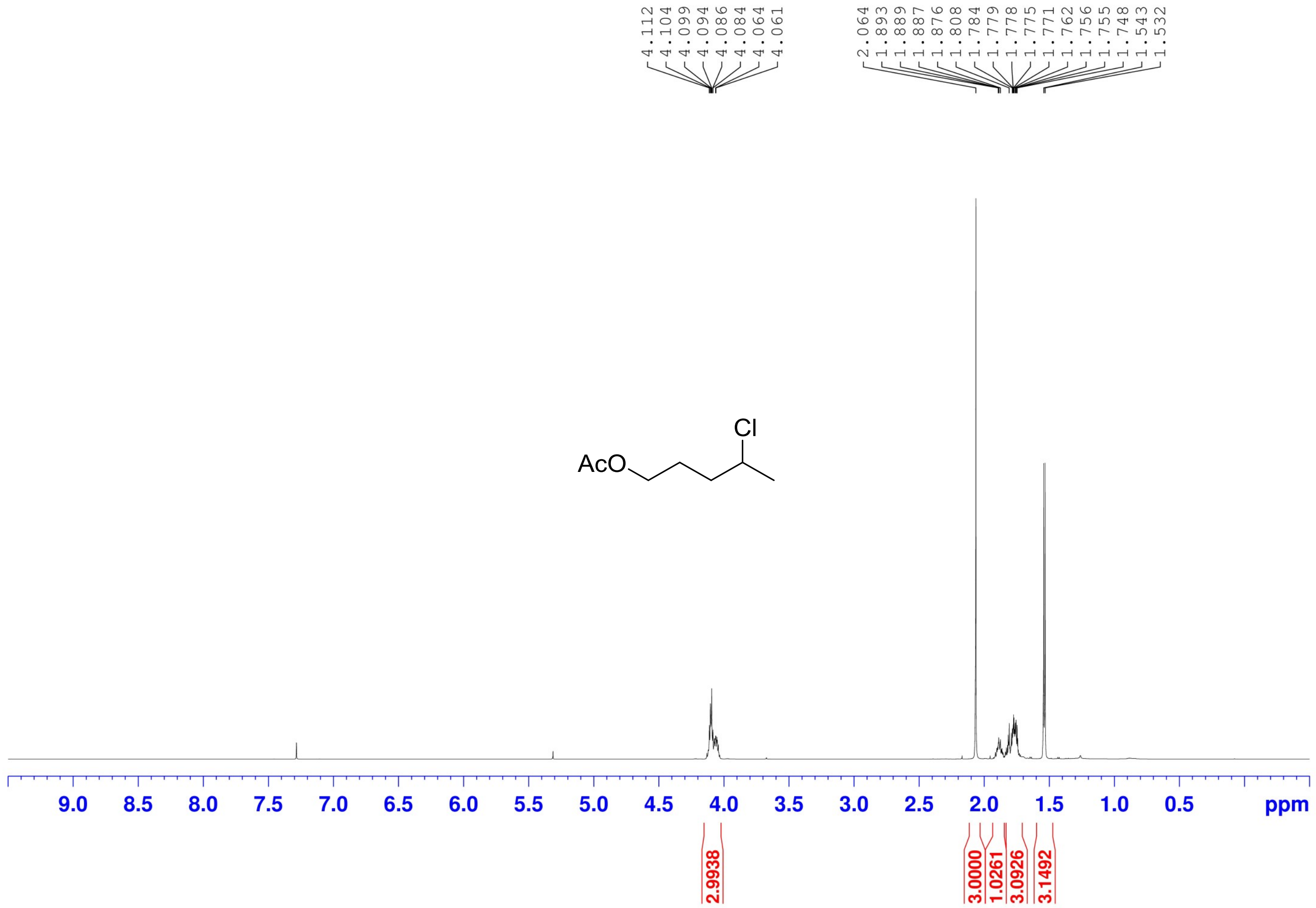
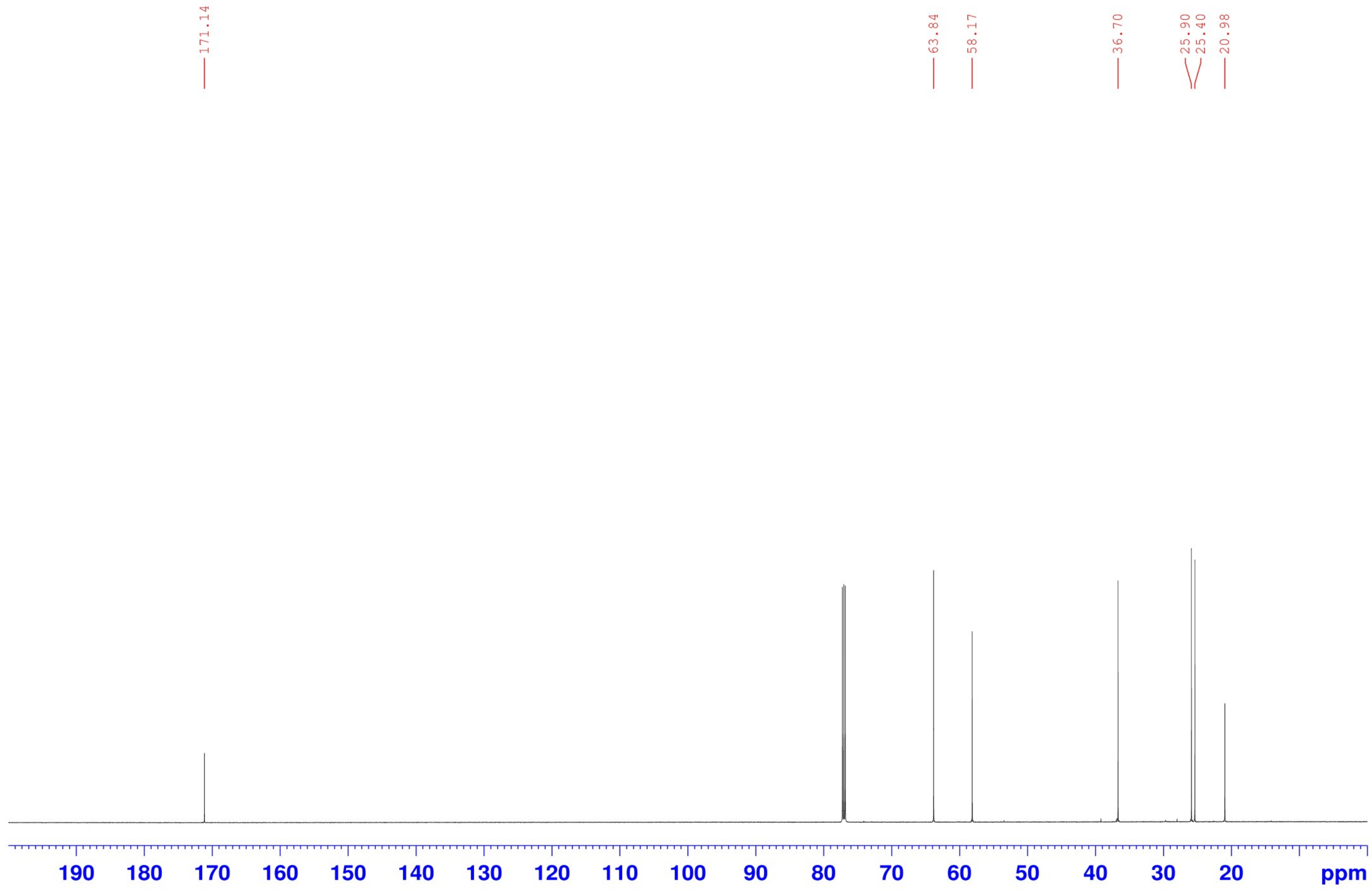
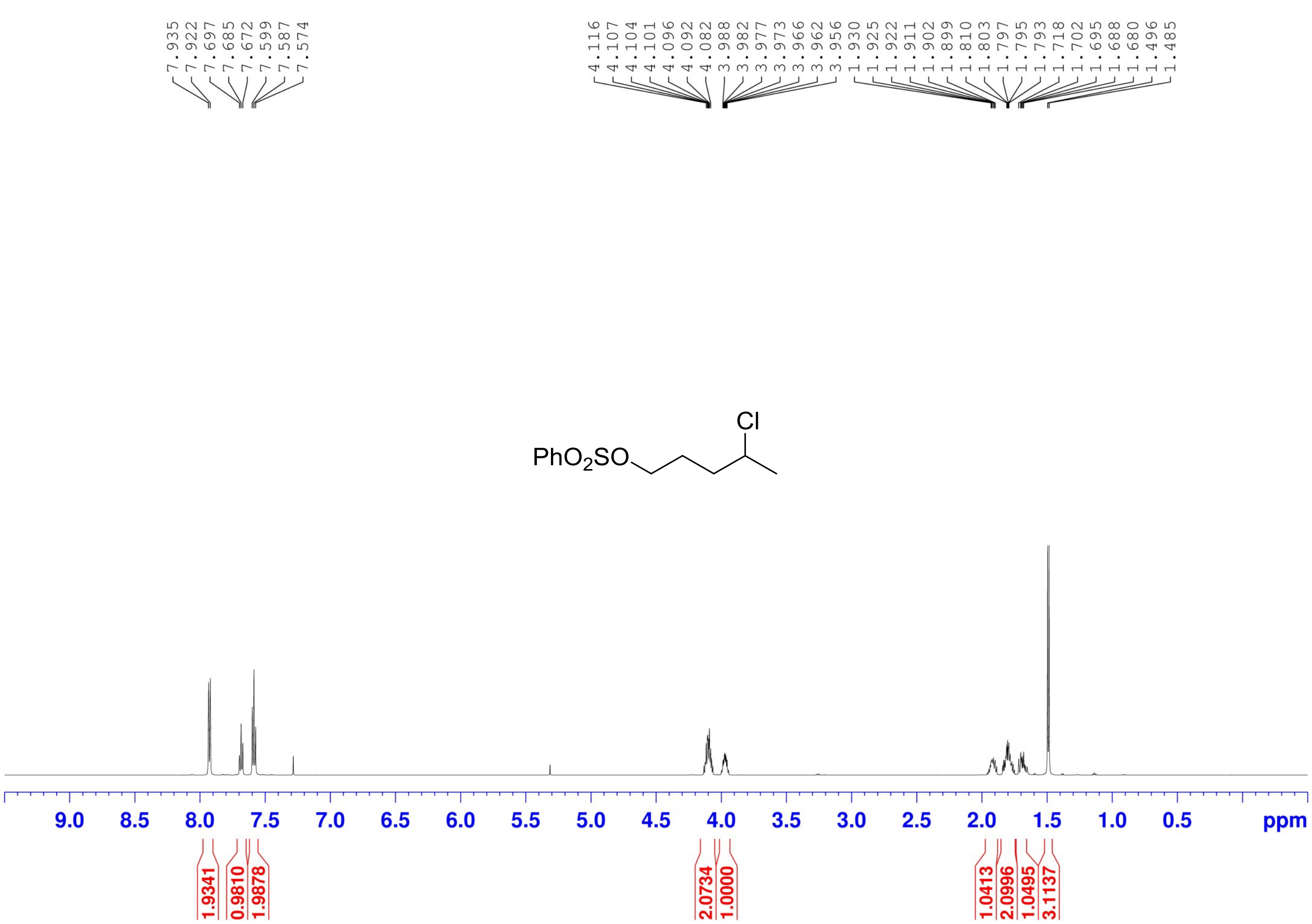
44.51
37.36
29.61
25.47
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


4.112
4.104
4.099
4.094
4.086
4.084
4.064
4.061
2.064
1.893
1.889
1.887
1.876
1.808
1.784
1.779
1.778
1.775
1.771
1.762
1.756
1.755
1.748
1.543
1.532
Cl
AcO
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.9938
3.0000
1.0261
3.0926
3.1492


171.14
63.84
58.17
36.70
25.90
25.40
20.98
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.922
7.697
7.685
7.672
7.599
7.587
7.574
4.116
4.107
4.104
4.101
4.096
4.092
4.082
3.988
3.982
3.977
3.973
3.966
3.962
3.956
1.930
1.925
1.922
1.911
1.902
1.899
1.810
1.803
1.797
1.795
1.793
1.718
1.702
1.695
1.688
1.680
1.496
1.485
Cl
PhO2SO
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
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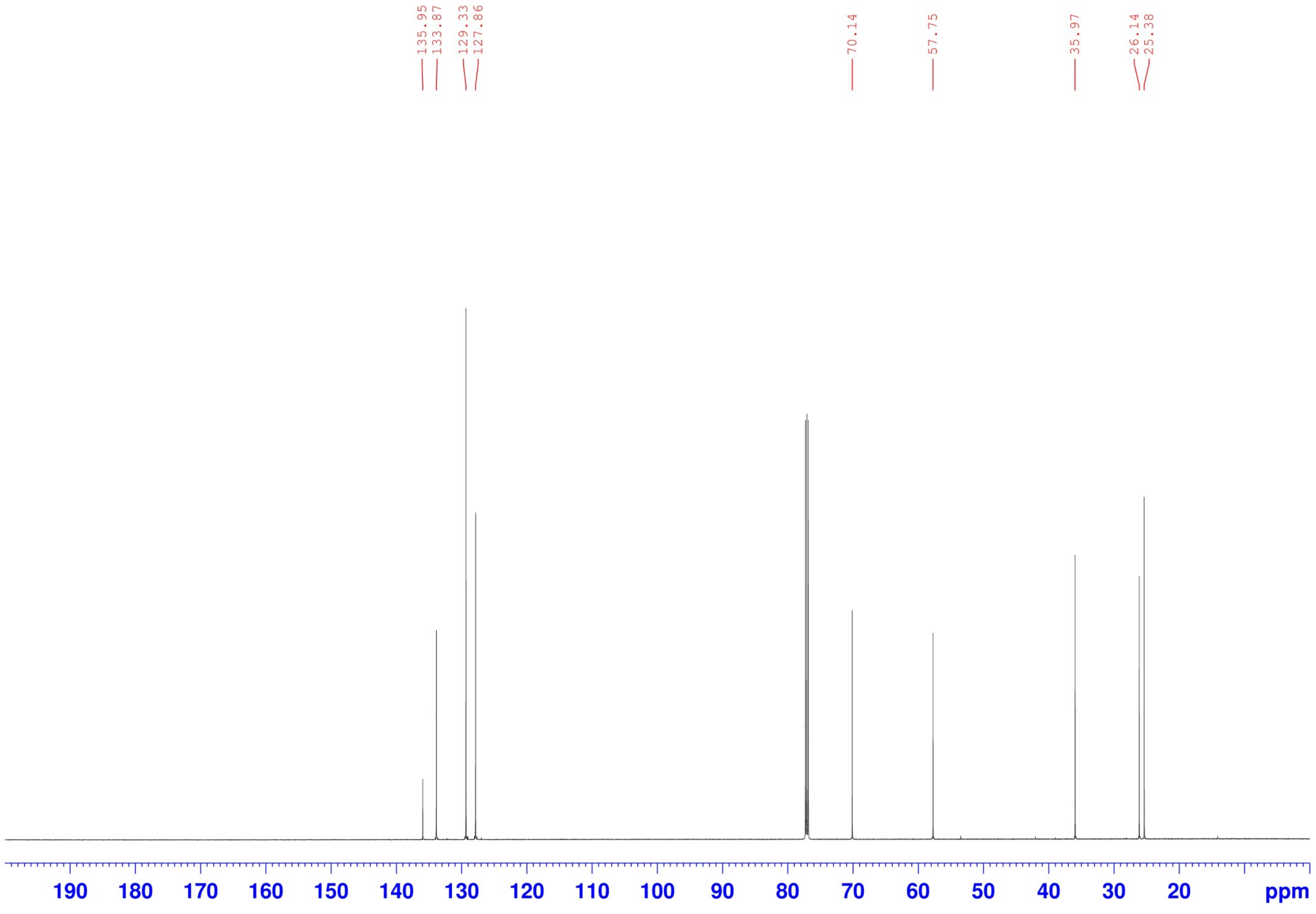
0.9810
1.9878
2.0734
1.0000
1.0413
2.0996
1.0495
3.1137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133.87
129.33
127.86
70.14
57.75
35.97
26.14
25.38
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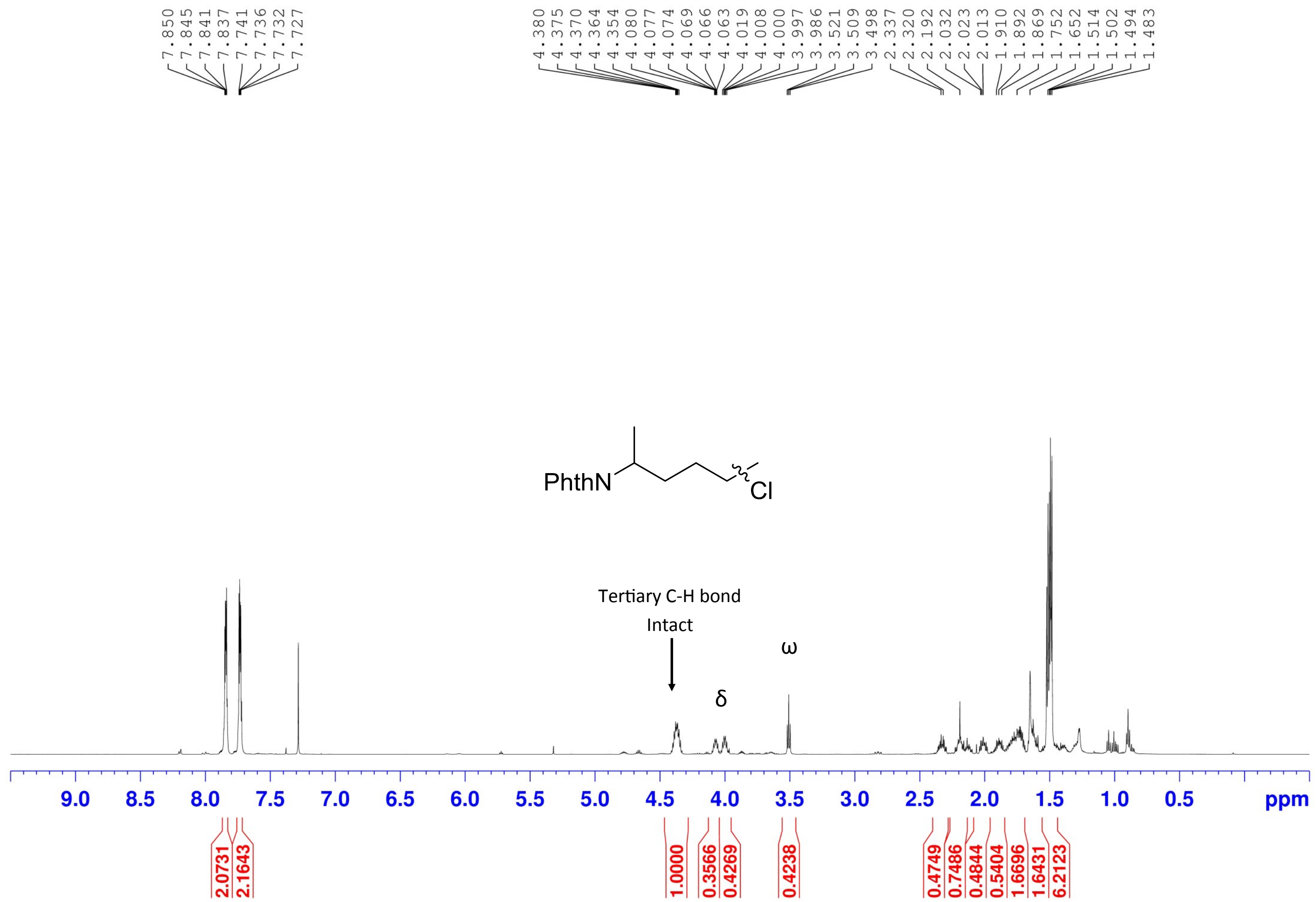
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


7.850
7.845
7.841
7.837
7.741
7.736
7.732
7.727
4.380
4.375
4.370
4.364
4.354
4.080
4.077
4.074
4.069
4.066
4.063
4.019
4.008
4.000
3.997
3.986
3.521
3.509
3.498
2.337
2.320
2.192
2.032
2.023
2.013
1.910
1.892
1.869
1.752
1.652
1.514
1.502
1.494
1.483
PhthN
Cl
Tertiary C-H bond
Intact
δ
ω
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.0731
2.1643
1.0000
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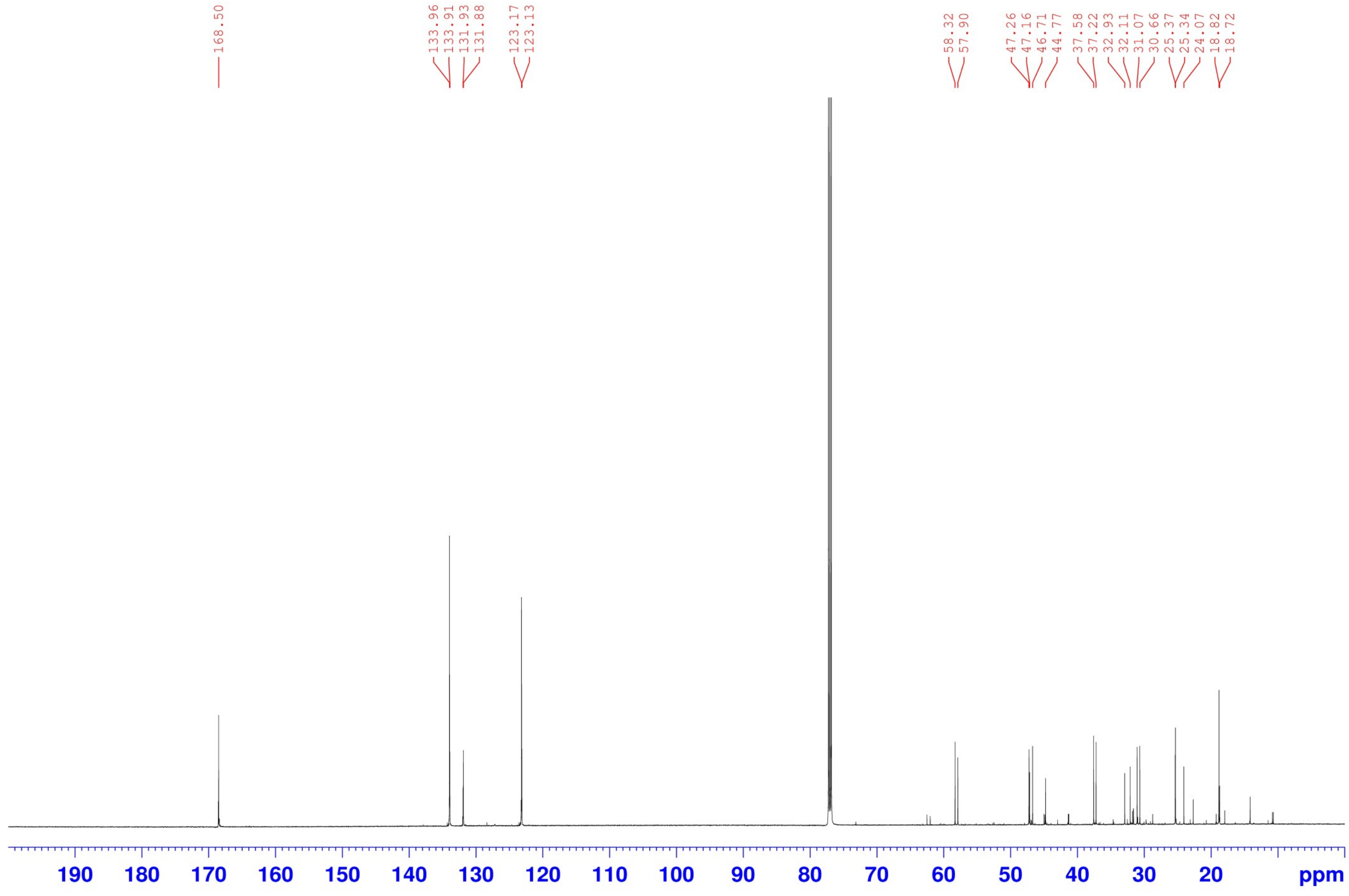
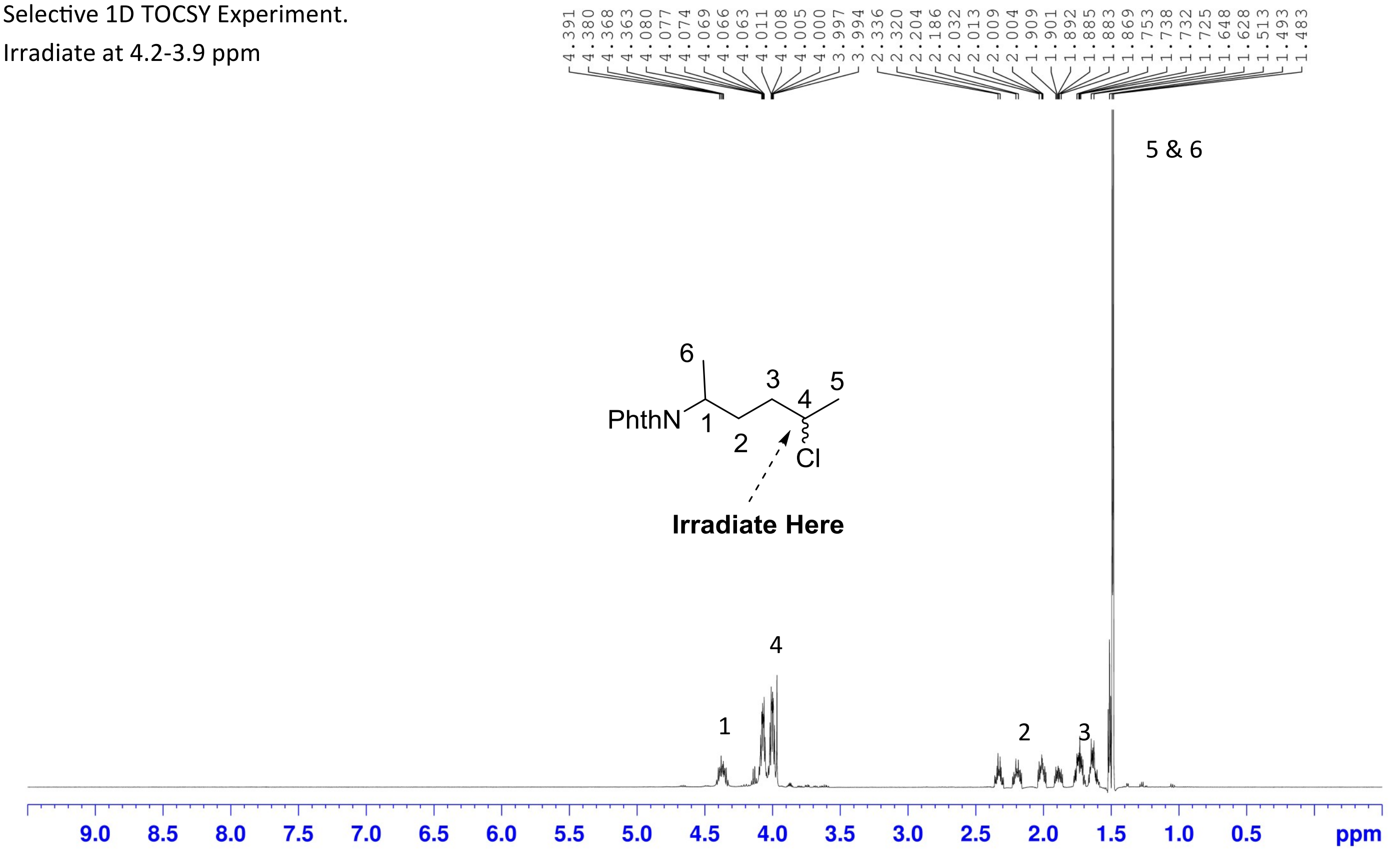
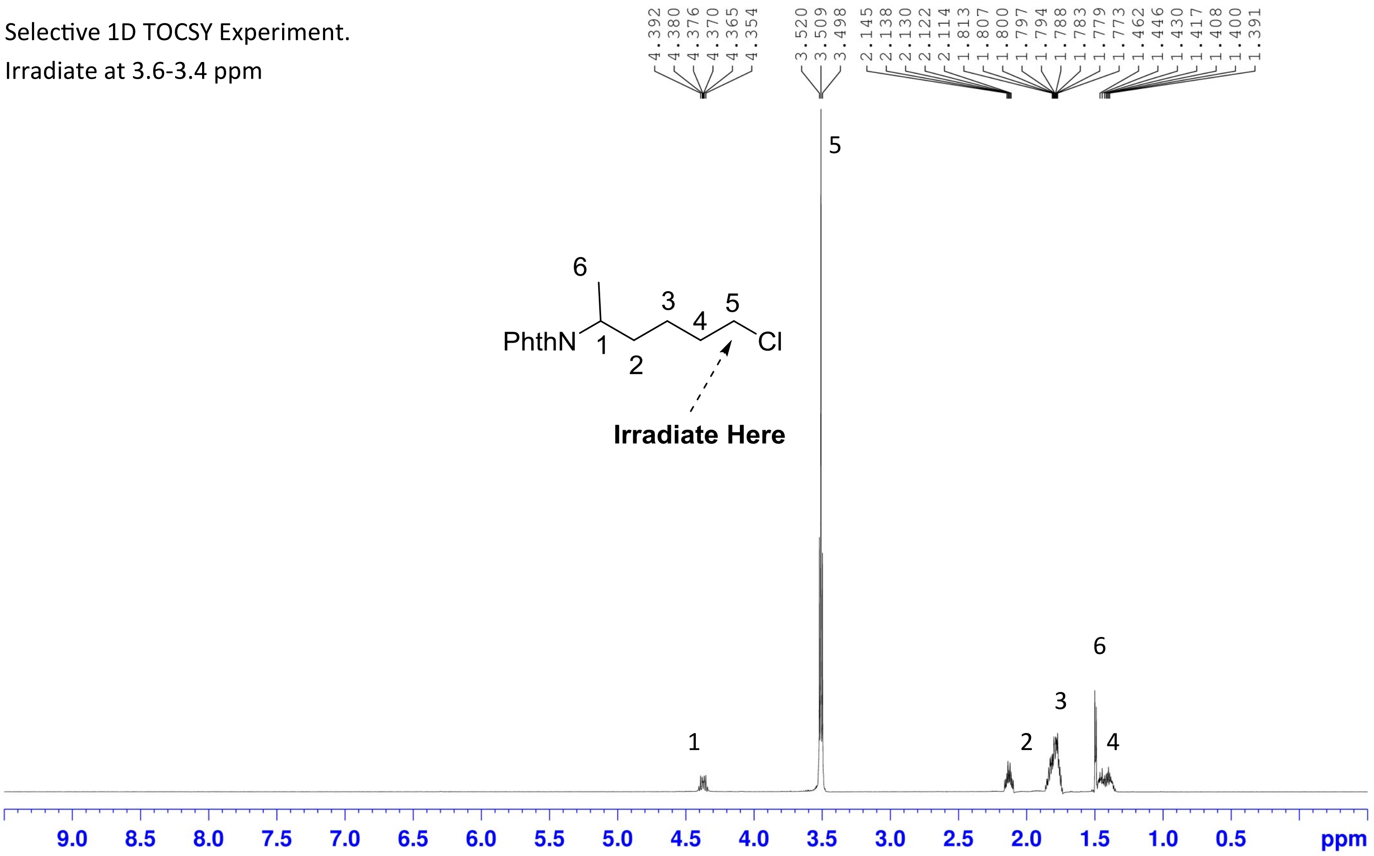
0.4269
0.4238
0.4749
0.7486
0.4844
0.5404
1.6696
1.6431
6.2123

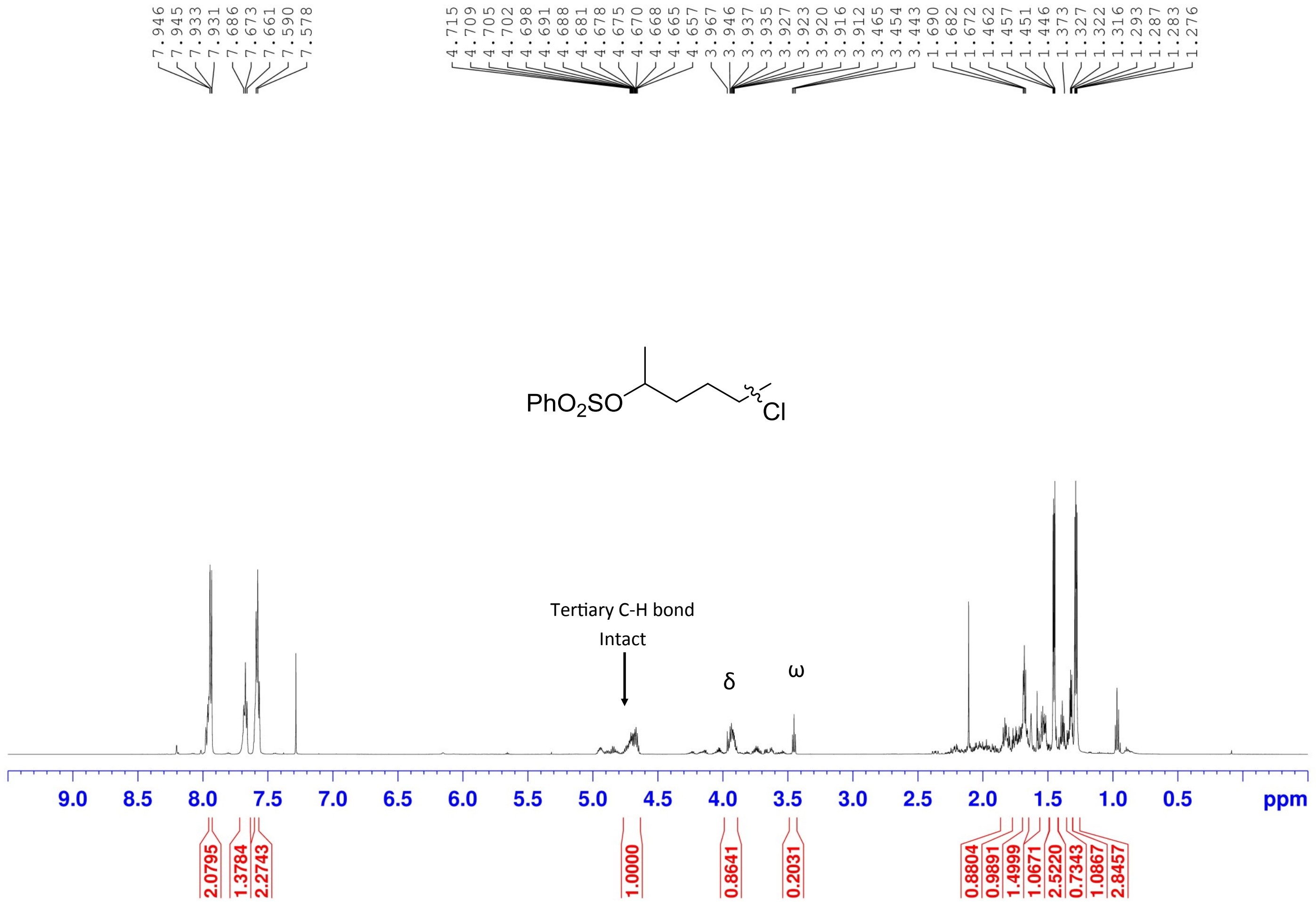

168.50
133.96
133.91
131.93
131.88
123.17
123.13
58.32
57.90
47.26
47.16
46.71
44.77
37.58
37.22
32.93
32.11
31.07
30.66
25.37
25.34
24.07
18.82
18.72
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Selective 1D TOCSY Experiment.
Irradiate at 4.2-3.9 ppm



Selective 1D TOCSY Experiment.
Irradiate at 3.6-3.4 ppm

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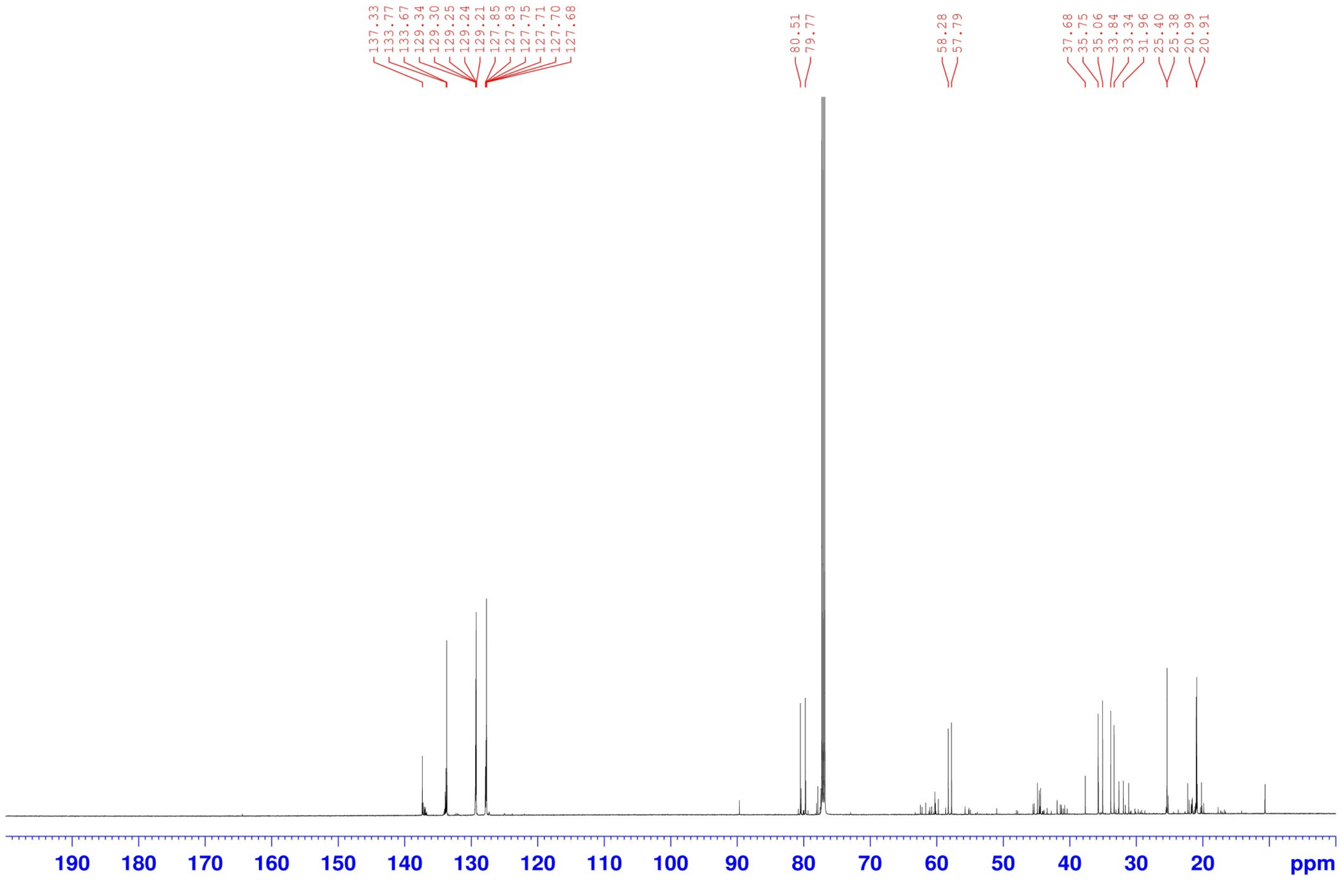
7.933
7.931
7.686
7.673
7.661
7.590
7.578
4.715
4.709
4.705
4.702
4.698
4.691
4.688
4.681
4.678
4.675
4.670
4.668
4.665
4.657
3.967
3.946
3.937
3.935
3.927
3.923
3.920
3.916
3.912
3.465
3.454
3.443
1.690
1.682
1.672
1.462
1.457
1.451
1.446
1.373
1.327
1.322
1.316
1.293
1.287
1.283
1.276
PhO2SO
Cl
Tertiary C-H bond
Intact
δ
ω
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.0795
1.3784
2.2743
1.0000
0.8641
0.2031
0.8804
0.9891
1.4999
1.0671
2.5220
0.7343
1.0867
2.8457


137.33
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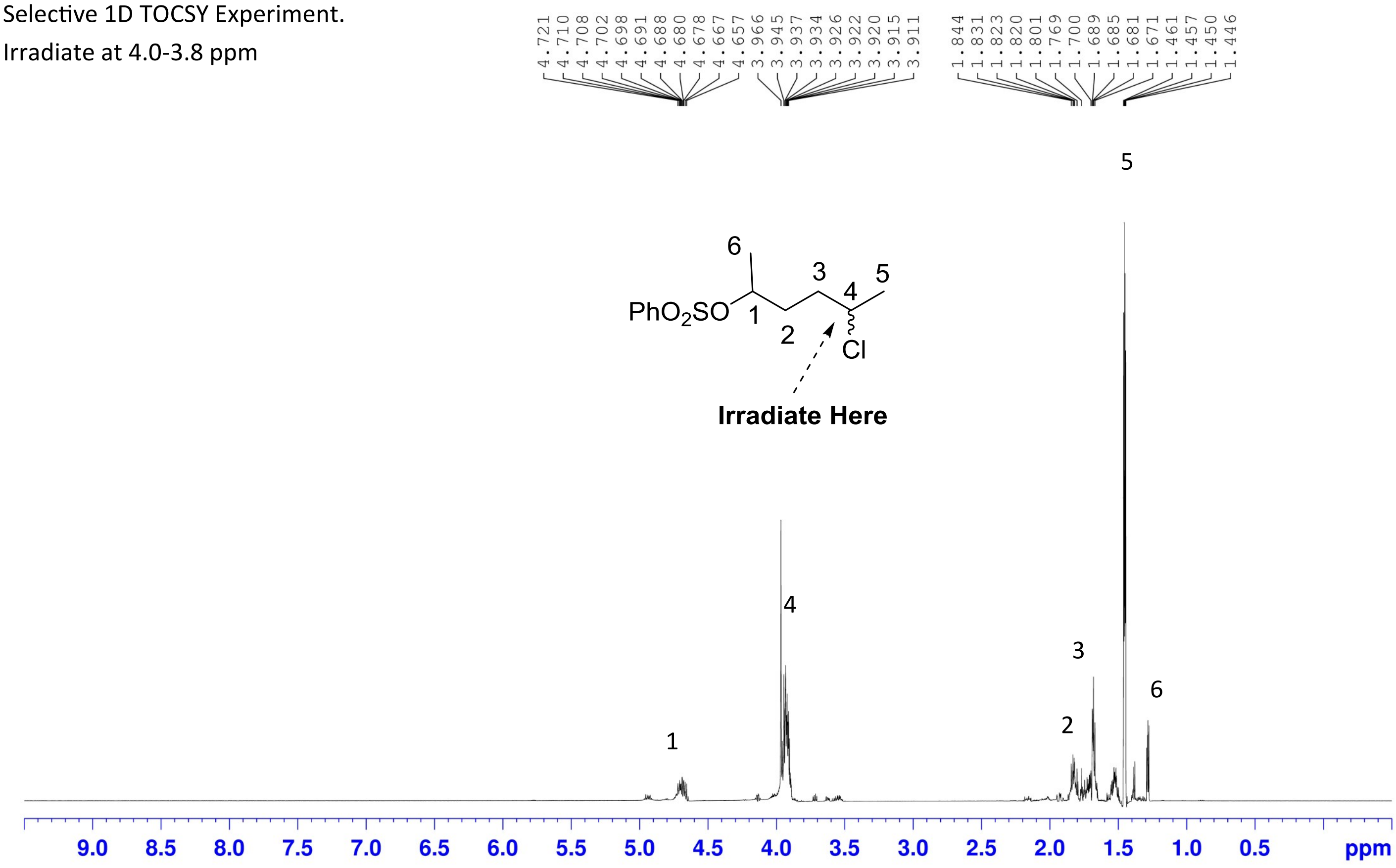
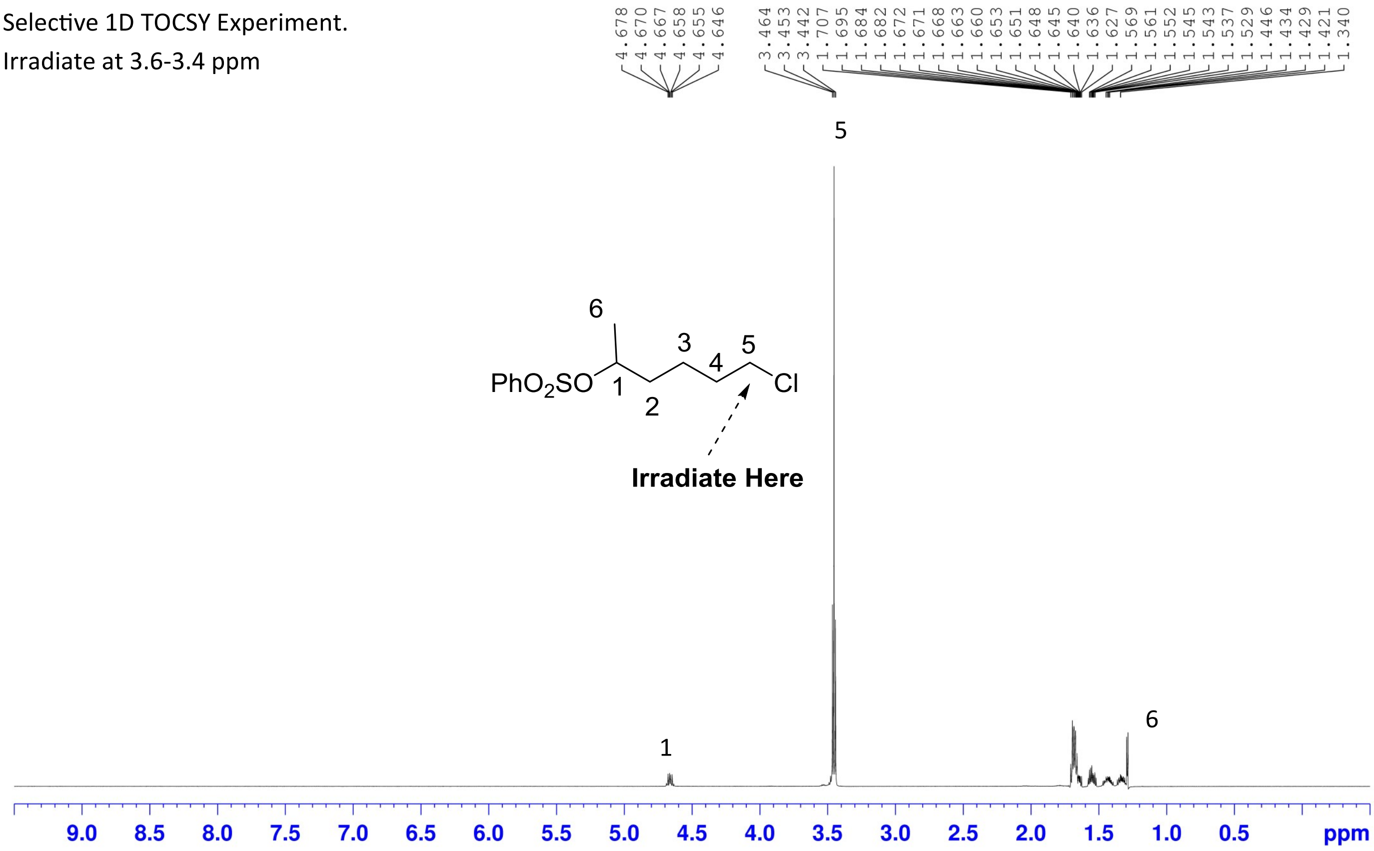
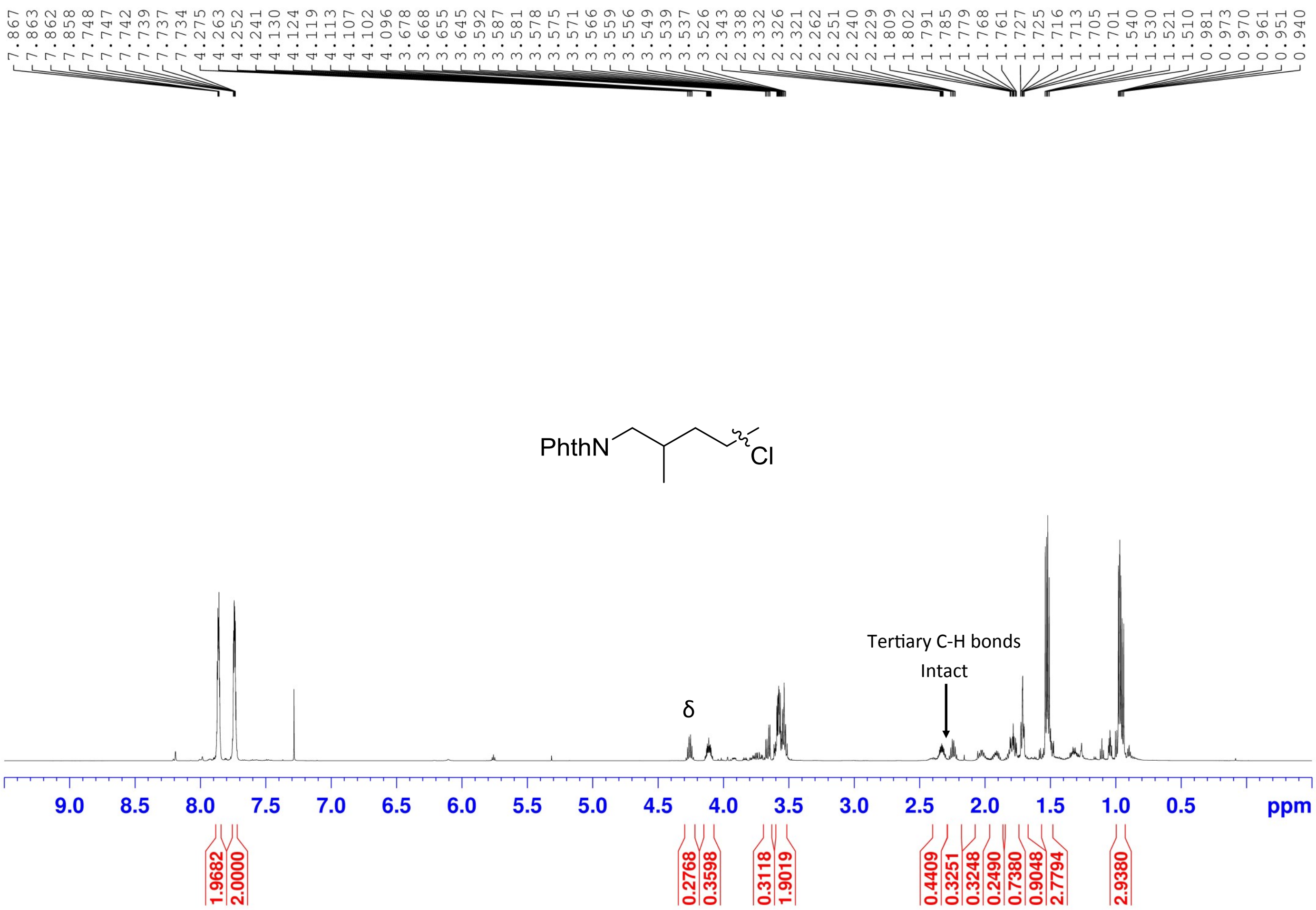
133.67
129.34
129.30
129.25
129.24
129.21
127.85
127.83
127.75
127.71
127.70
127.68
80.51
79.77
58.28
57.79
37.68
35.75
35.06
33.84
33.34
31.96
25.40
25.38
20.99
20.91
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

Selective 1D TOCSY Experiment.
Irradiate at 4.0-3.8 ppm



Selective 1D TOCSY Experiment.
Irradiate at 3.6-3.4 ppm

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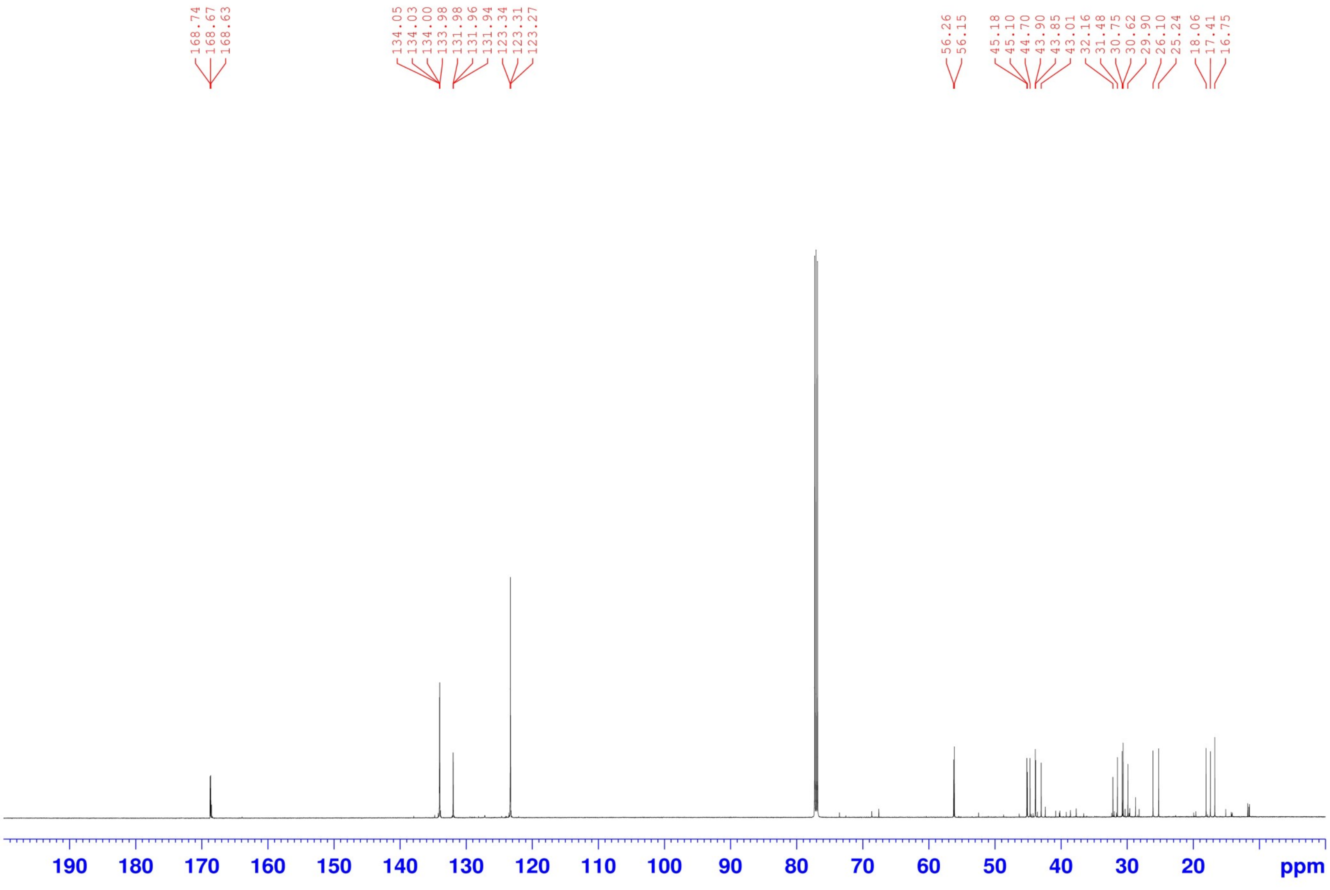
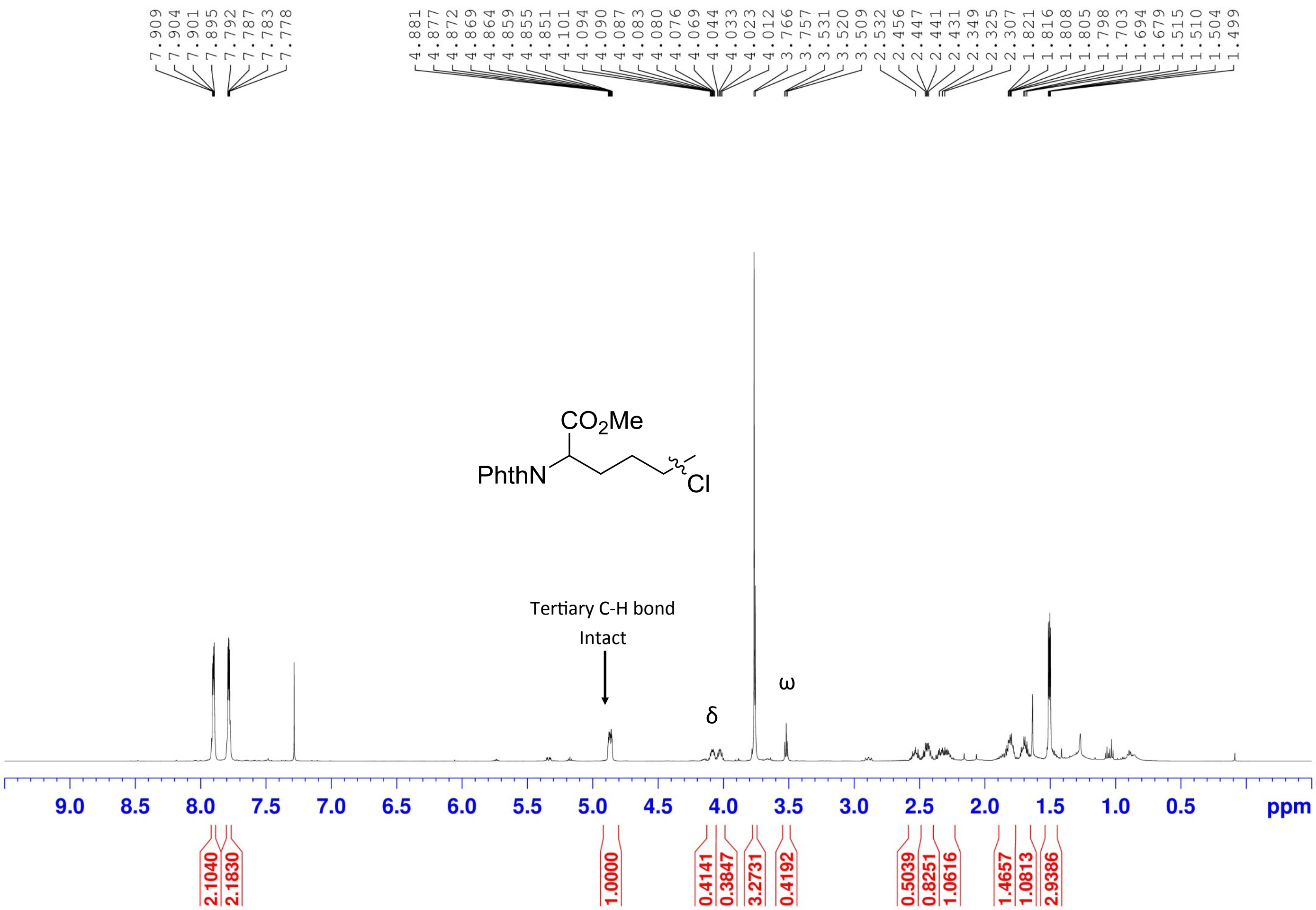
7.747
7.742
7.739
7.737
7.734
4.275
4.263
4.252
4.241
4.130
4.124
4.119
4.113
4.107
4.102
4.096
3.678
3.668
3.655
3.645
3.592
3.587
3.581
3.578
3.575
3.571
3.566
3.559
3.556
3.549
3.539
3.537
3.526
2.343
2.338
2.332
2.326
2.321
2.262
2.251
2.240
2.229
1.809
1.802
1.791
1.785
1.779
1.768
1.761
1.727
1.725
1.716
1.713
1.705
1.701
1.540
1.530
1.521
1.510
0.981
0.973
0.970
0.961
0.951
0.940
PhthN
Cl
Tertiary C-H bonds
Intact
δ
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.9682
2.0000
0.2768
0.3598
0.3118
1.9019
0.4409
0.3251
0.3248
0.2490
0.7380
0.9048
2.7794
2.9380


168.74
168.67
168.63
134.05
134.03
134.00
133.98
131.98
131.96
131.94
123.34
123.31
123.27
56.26
56.15
45.18
45.10
44.70
43.90
43.85
43.01
32.16
31.48
30.75
30.62
29.90
26.10
25.24
18.06
17.41
16.75
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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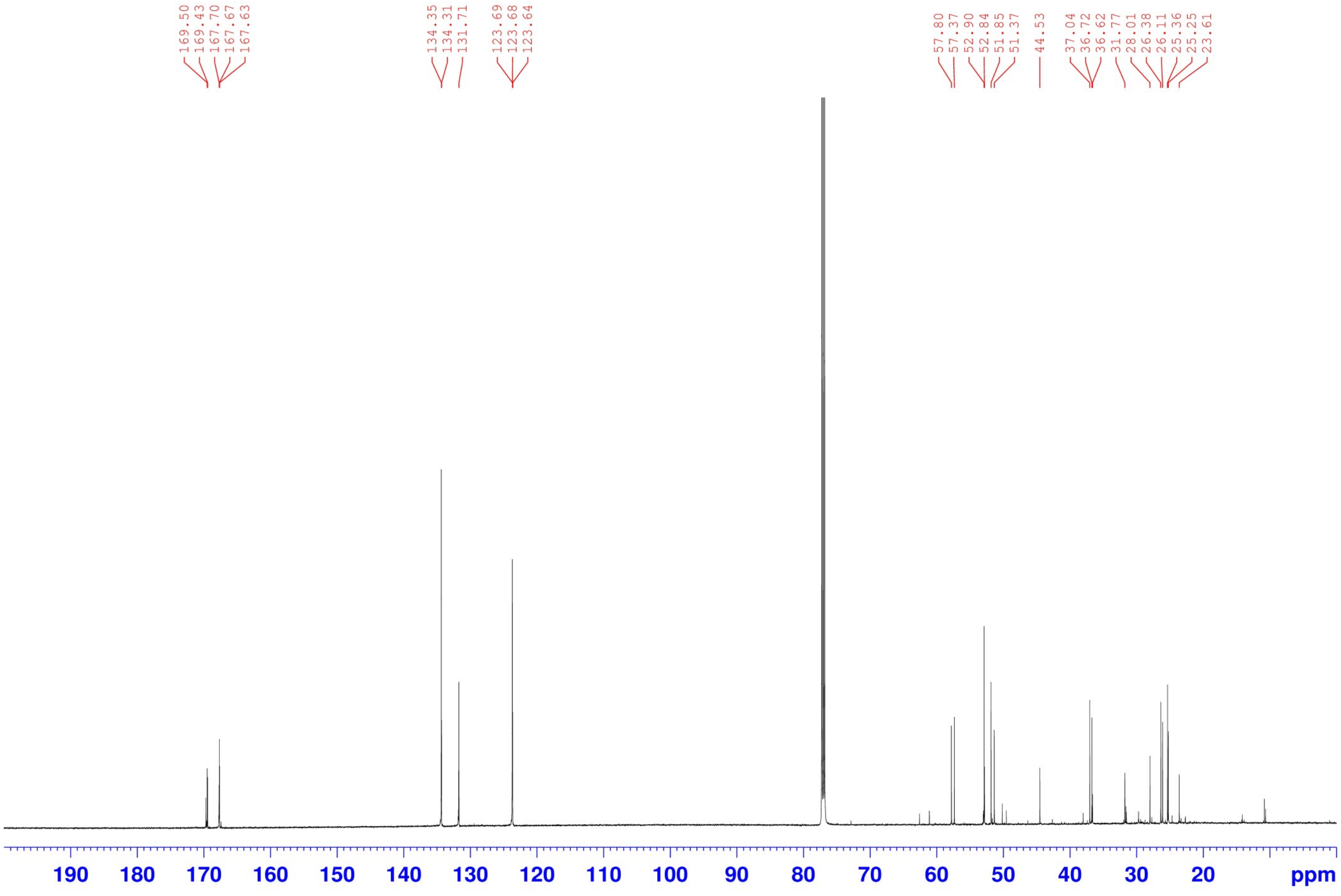
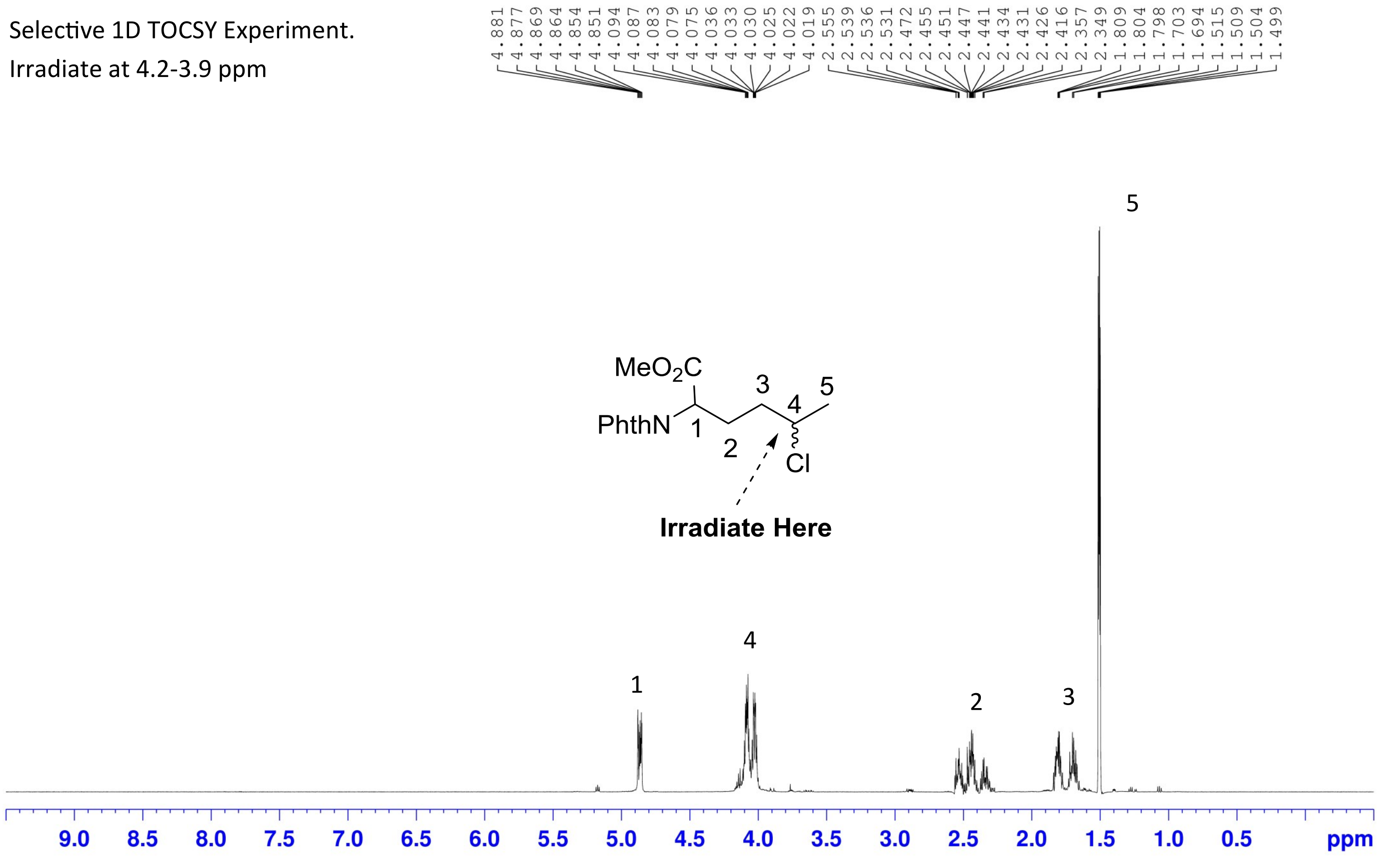
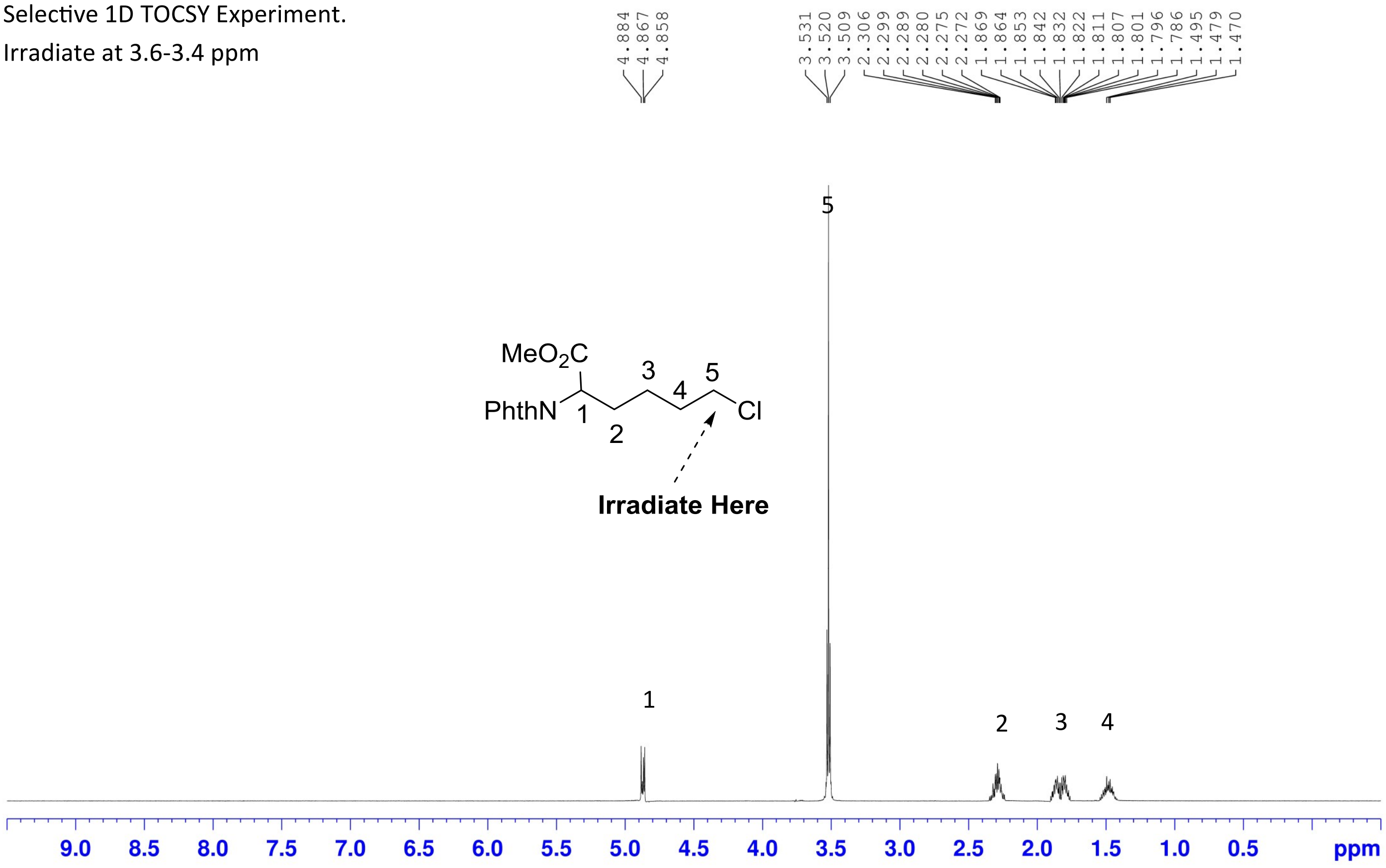
7.904
7.901
7.895
7.792
7.787
7.783
7.778
4.881
4.877
4.872
4.869
4.864
4.859
4.855
4.851
4.101
4.094
4.090
4.087
4.083
4.080
4.076
4.069
4.044
4.033
4.023
4.012
3.766
3.757
3.531
3.520
3.509
2.532
2.456
2.447
2.441
2.431
2.349
2.325
2.307
1.821
1.816
1.808
1.805
1.798
1.703
1.694
1.679
1.515
1.510
1.504
1.499
CO_2Me
PhthN
Cl
Tertiary C-H bond
Intact
δ
ω
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.1040
2.1830
1.0000
0.4141
0.3847
3.2731
0.4192
0.5039
0.8251
1.0616
1.4657
1.0813
2.9386

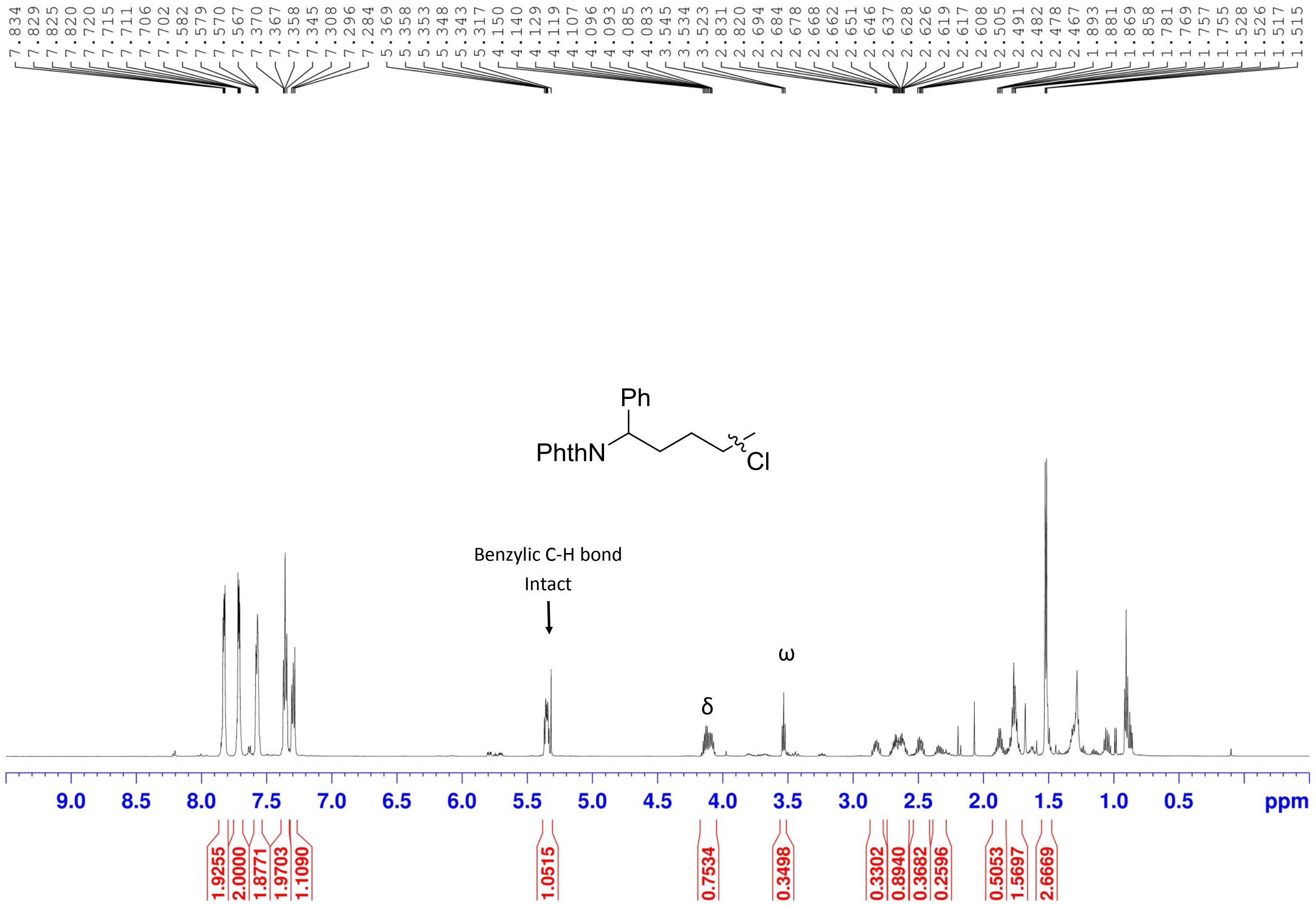

169.50
169.43
167.70
167.67
167.63
134.35
134.31
131.71
123.69
123.68
123.64
57.80
57.37
52.90
52.84
51.85
51.37
44.53
37.04
36.72
36.62
31.77
28.01
26.38
26.11
25.36
25.25
23.61
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

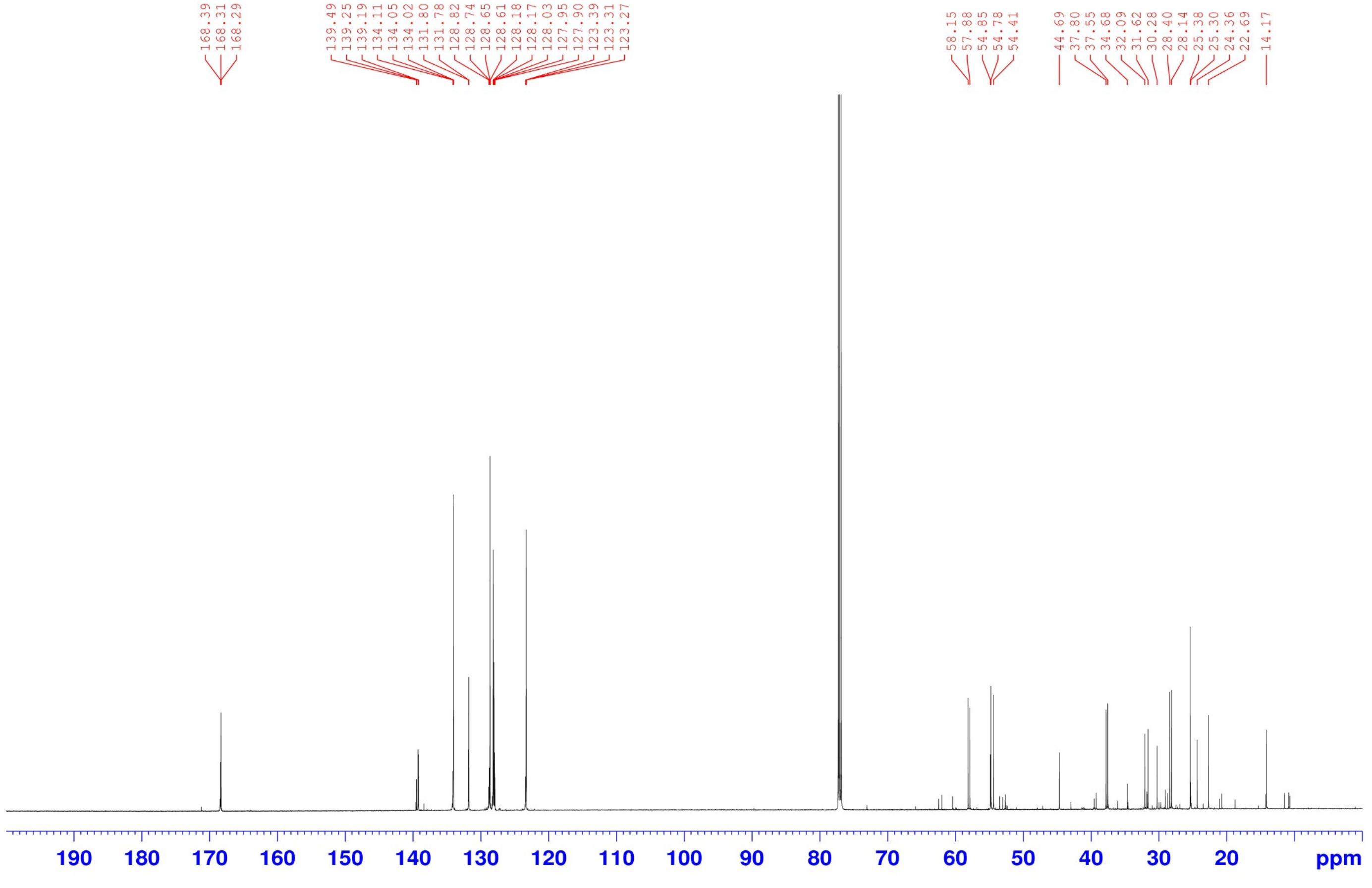
Selective 1D TOCSY Experiment.
Irradiate at 4.2-3.9 ppm



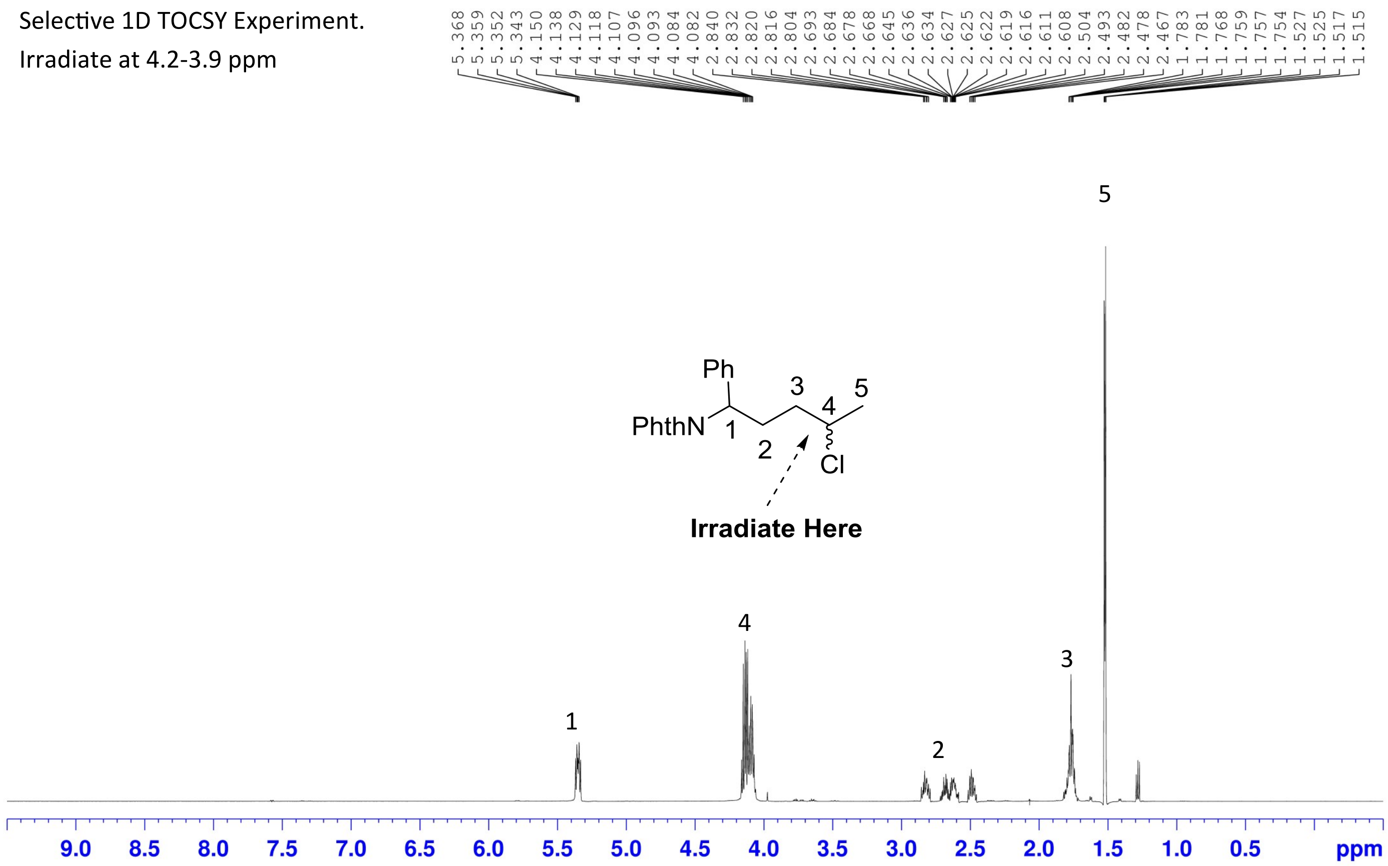
Selective 1D TOCSY Experiment.
Irradiate at 3.6-3.4 ppm



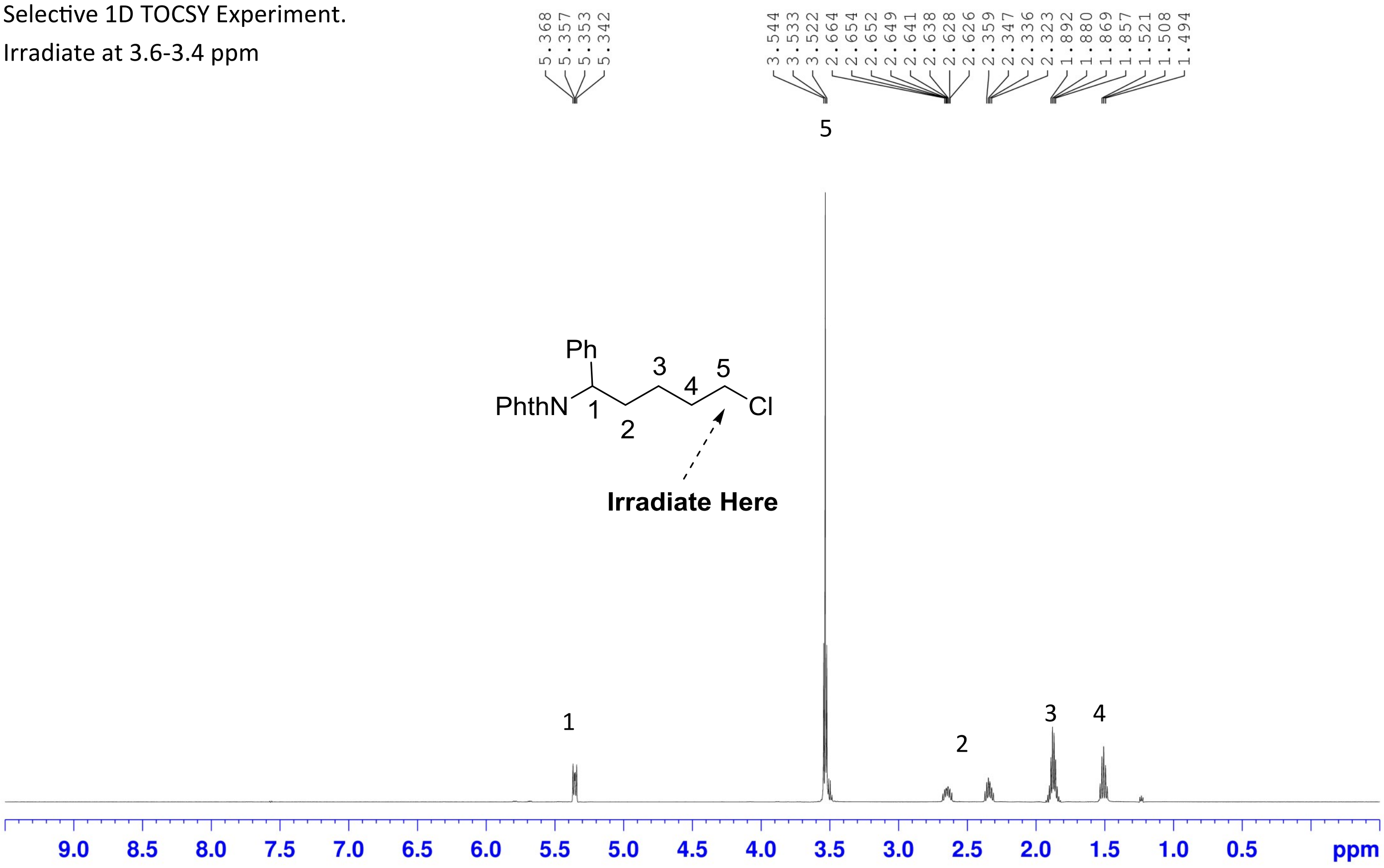

7.834
7.829
7.825
7.820
7.720
7.715
7.711
7.706
7.702
7.582
7.579
7.570
7.567
7.370
7.367
7.358
7.345
7.308
7.296
7.284
5.369
5.358
5.353
5.348
5.343
5.317
4.150
4.140
4.129
4.119
4.107
4.096
4.093
4.085
4.083
3.545
3.534
3.523
2.831
2.820
2.694
2.684
2.678
2.668
2.662
2.651
2.646
2.637
2.628
2.626
2.619
2.617
2.608
2.505
2.491
2.482
2.478
2.467
1.893
1.881
1.869
1.858
1.781
1.769
1.757
1.755
1.528
1.526
1.517
1.515
Ph
PhthN
Cl
Benzylic C-H bond
Intact
δ
ω
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.9255
2.0000
1.8771
1.9703
1.1090
1.0515
0.7534
0.3498
0.3302
0.8940
0.3682
0.2596
0.5053
1.5697
2.6669


168.39
168.31
168.29
139.49
139.25
139.19
134.11
134.05
134.02
131.80
131.78
128.82
128.74
128.65
128.61
128.18
128.17
128.03
127.95
127.90
123.39
123.31
123.27
58.15
57.88
54.85
54.78
54.41
44.69
37.80
37.55
34.68
32.09
31.62
30.28
28.40
28.14
25.38
25.30
24.36
22.69
14.17
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

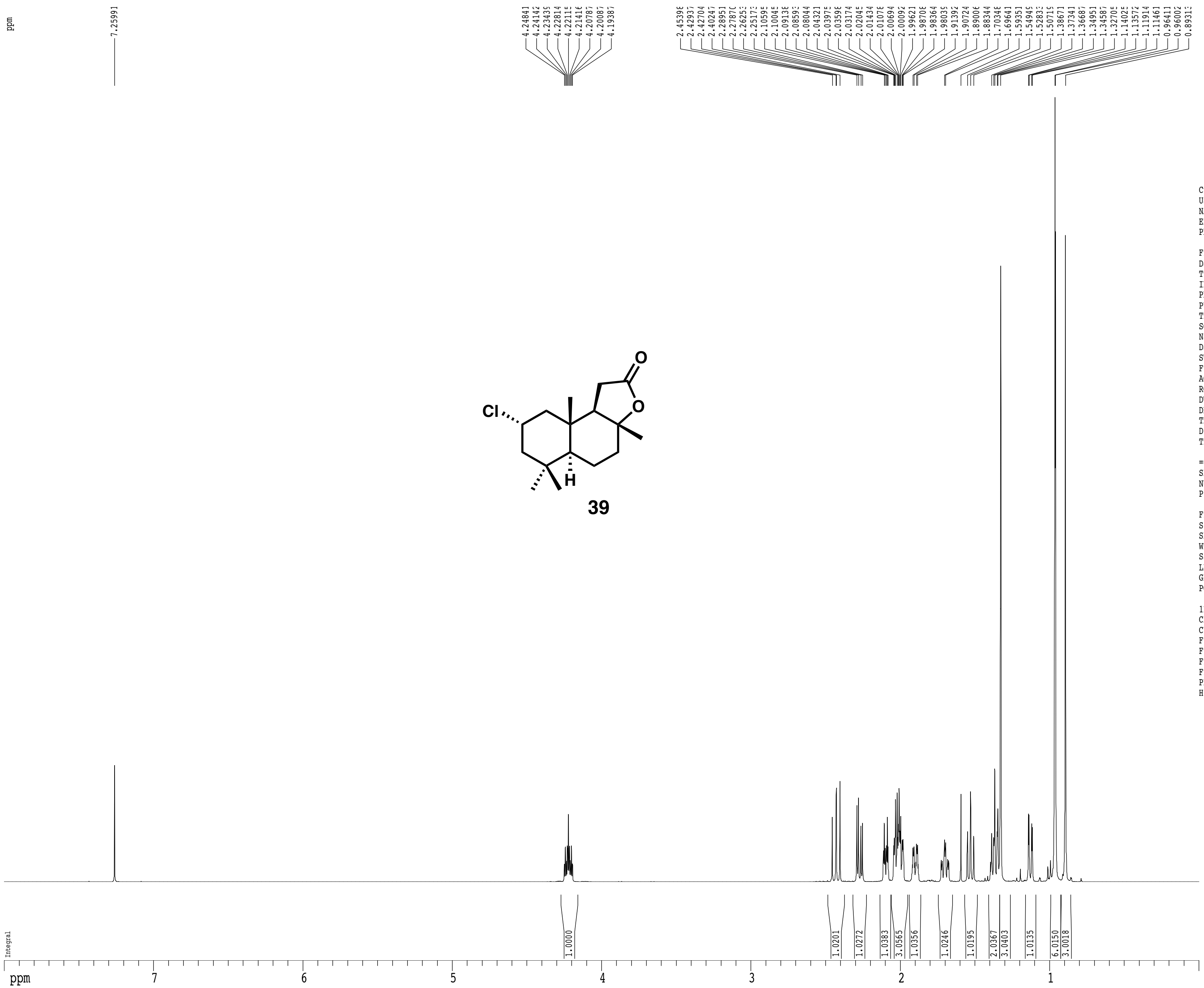
Selective 1D TOCSY Experiment.
Irradiate at 4.2-3.9 ppm



Selective 1D TOCSY Experiment.
Irradiate at 3.6-3.4 ppm



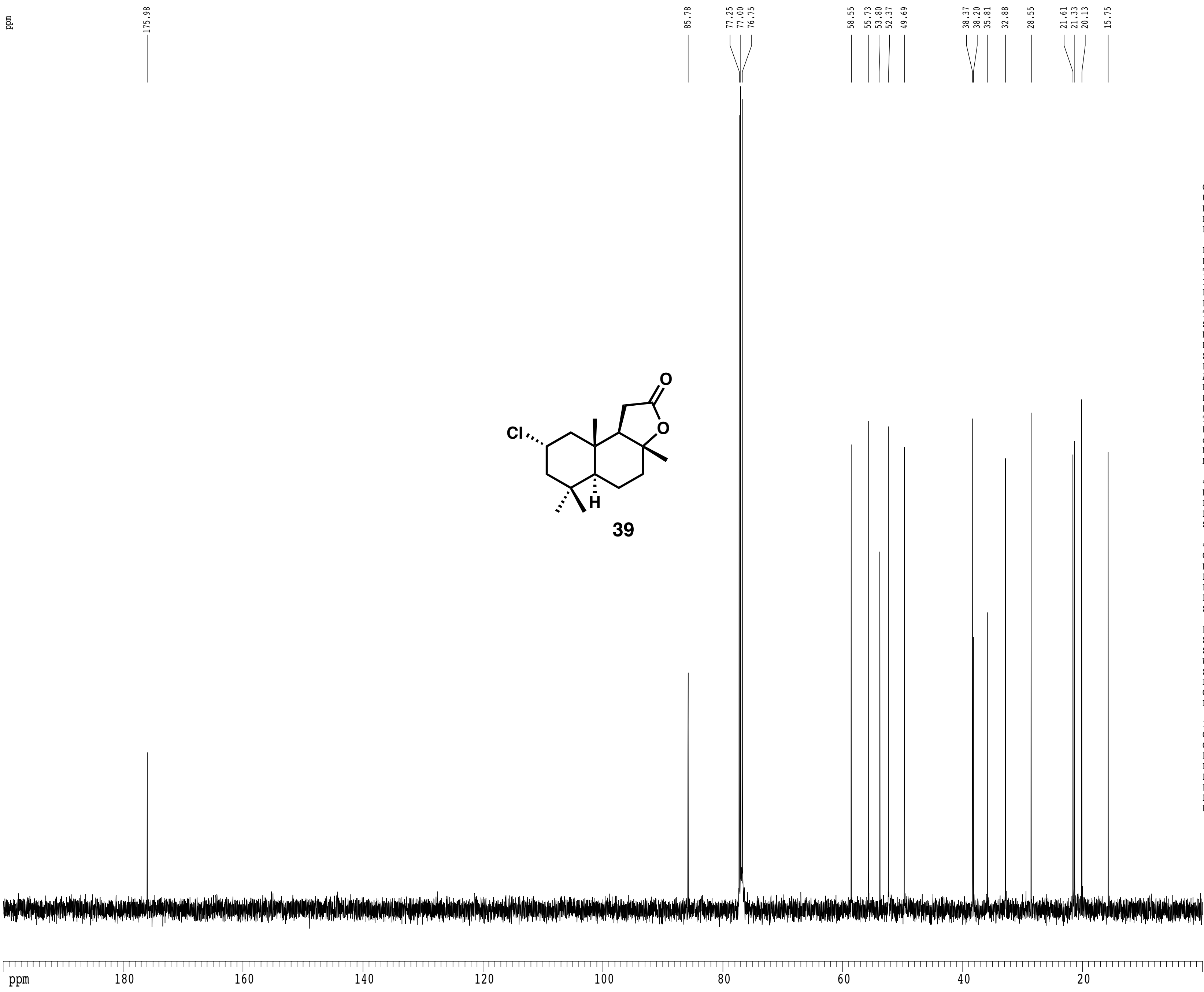
1H spectrum



39

```
Current Data Parameters
USER              zkonst
NAME               3-157
EXPNO                211
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151006
Time               11.42
INSTRUM            av600
PROBHD   5 mm TBI 1H/13
PULPROG             zg30
TD                 57690
SOLVENT            CDCl3
NS                    16
DS                     0
SWH            9615.385 Hz
FIDRES          0.166673 Hz
AQ             2.9999299 sec
RG                   181
DW                52.000 usec
DE                 14.54 usec
TE                 298.1 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        600.1342009 MHz
NUC1                  1H
P1                  8.00 usec

F2 - Processing parameters
SI                 65536
SF          600.1300357 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                8.000 ppm
F1               4801.04 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.35088 ppm/cm
HZCM           210.57196 Hz/cm
```

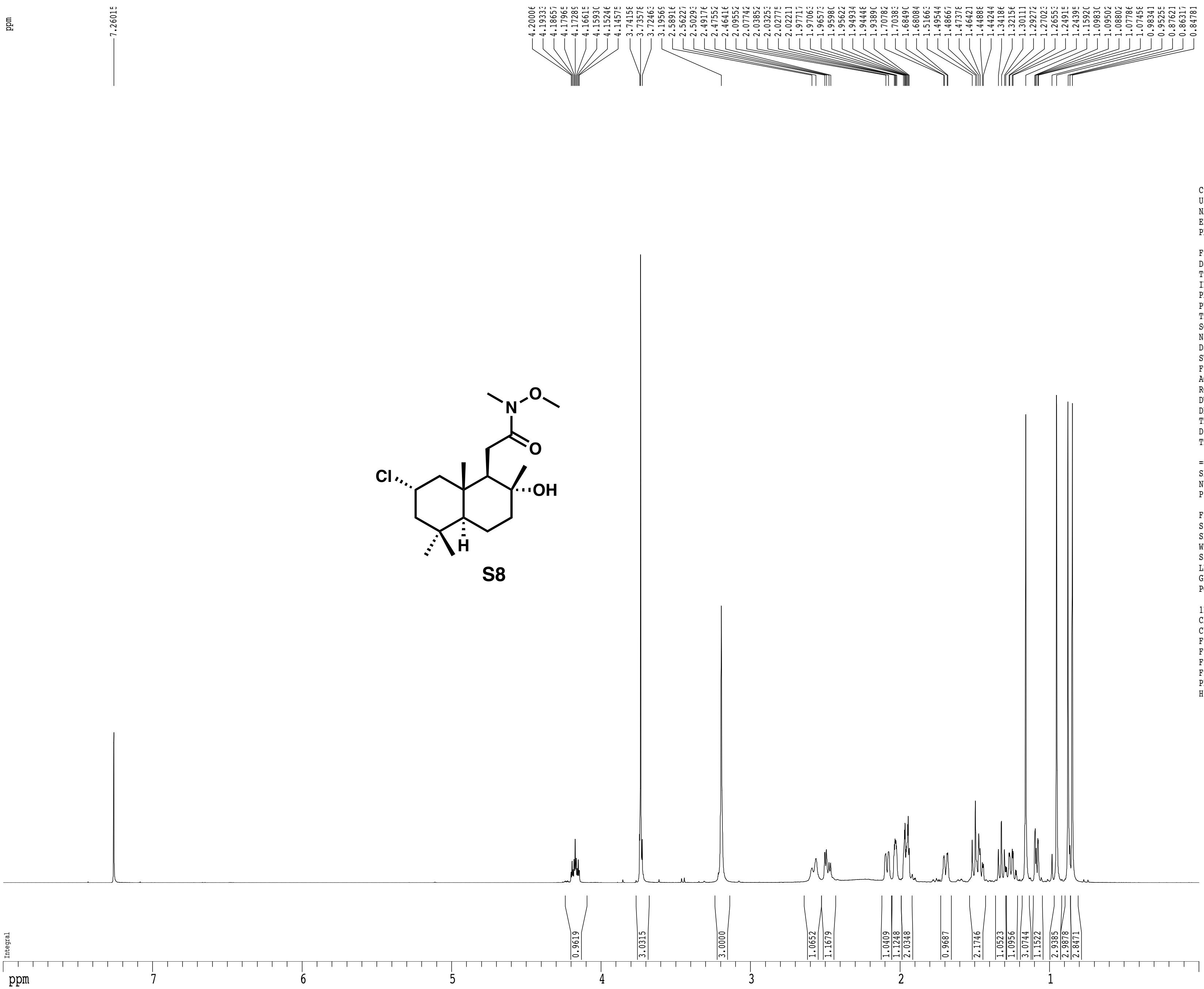
13C spectrum with 1H decoupling



39

```
Current Data Parameters
USER              zkonst
NAME               3-157
EXPNO                212
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151006
Time               18.30
INSTRUM            gn500
PROBHD   5 mm broadband
PULPROG          zgdc30
TD                 65536
SOLVENT            CDCl3
NS                   440
DS                     4
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0814105 sec
RG               23170.5
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                  9.00 usec
PL1                -0.60 dB
SFO1         125.5327181 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               12.80 dB
SFO2         499.1824959 MHz

F2 - Processing parameters
SI                 65536
SF           125.5189198 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              200.000 ppm
F1              25103.79 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            8.77193 ppm/cm
HZCM          1101.04321 Hz/cm
```

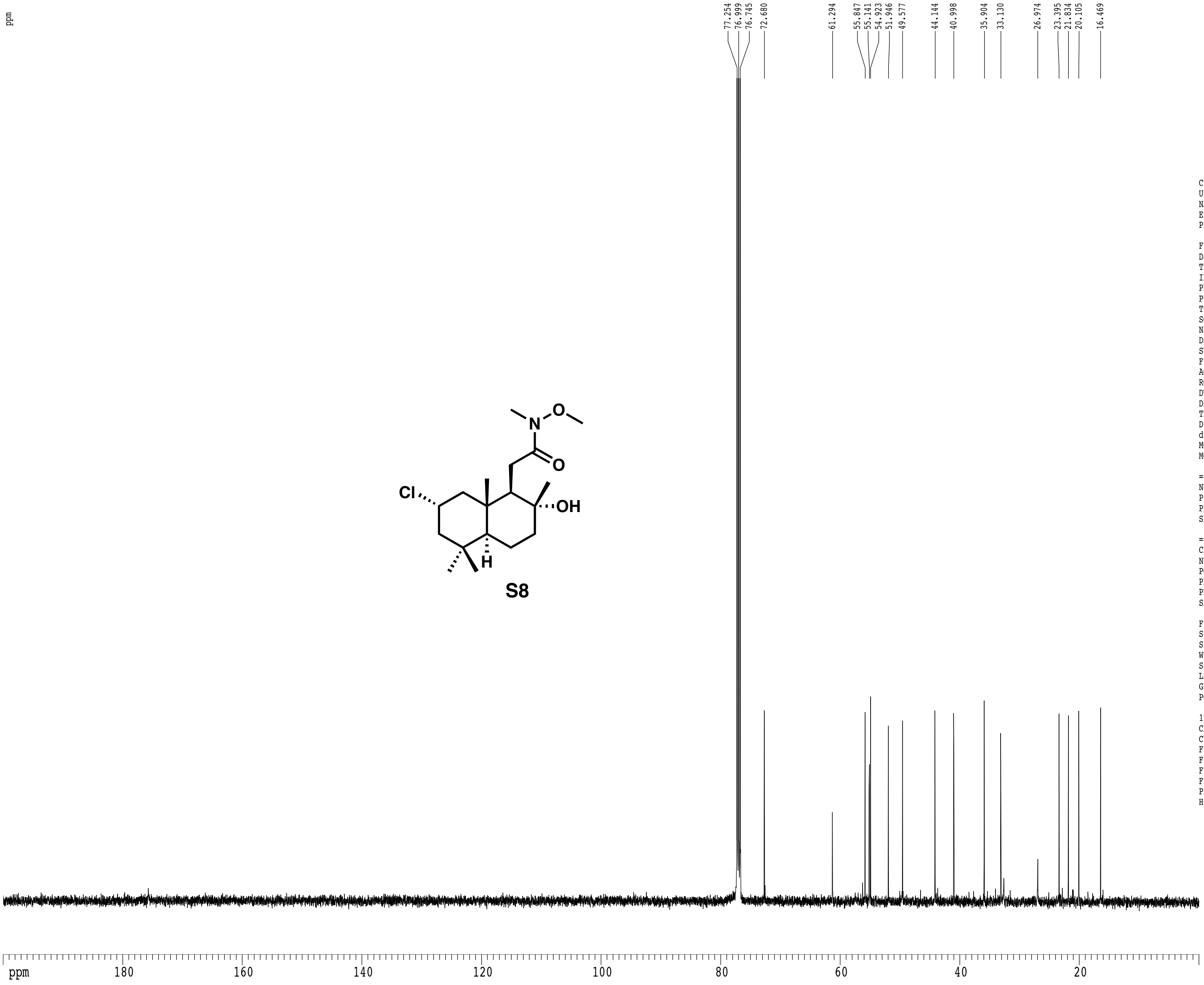
1H spectrum



S8

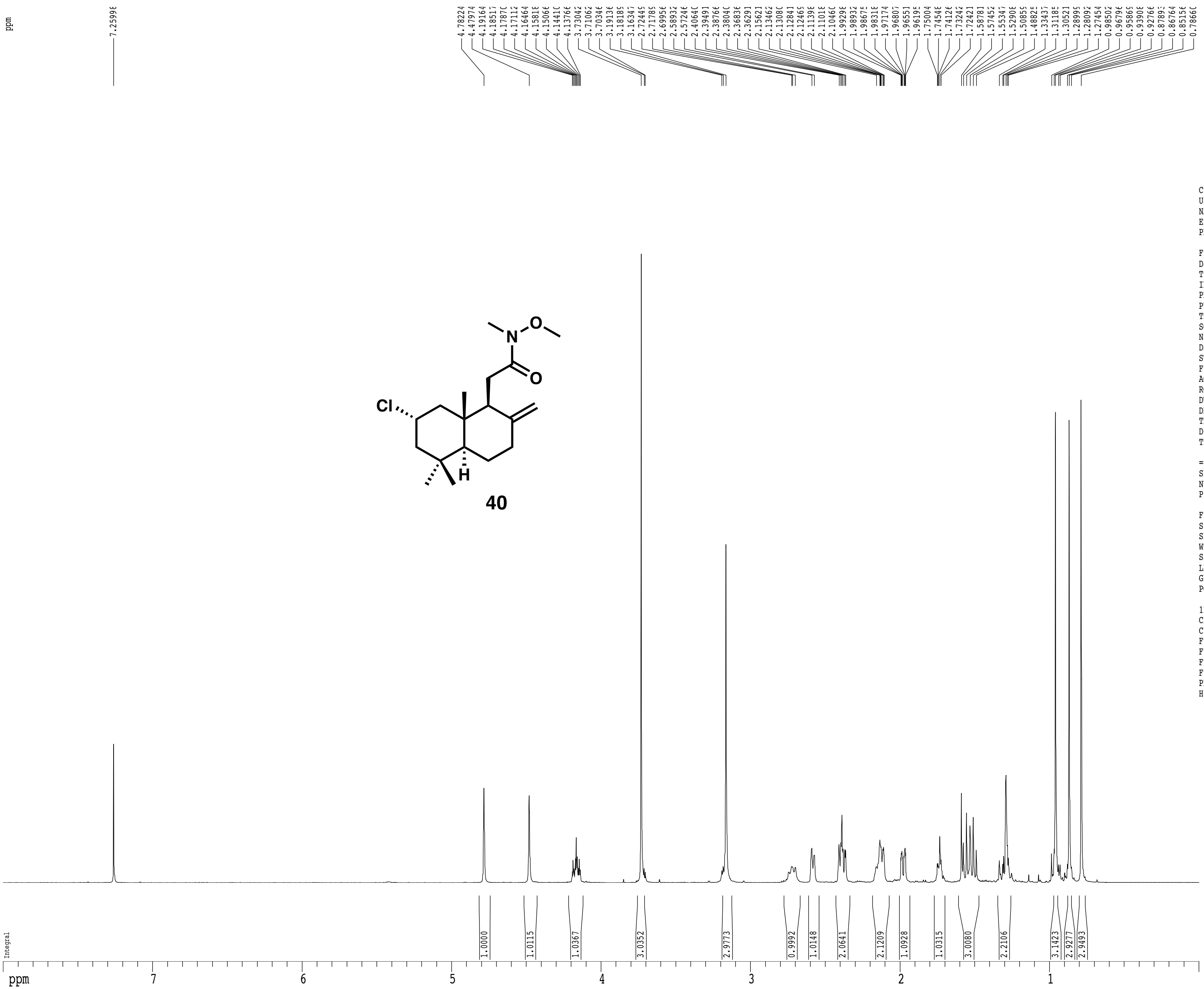
```
Current Data Parameters
USER              zkonst
NAME                2-43
EXPNO                 15
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140226
Time               20.57
INSTRUM            av600
PROBHD   5 mm TBI 1H/13
PULPROG             zg30
TD                 57690
SOLVENT            CDCl3
NS                    16
DS                     1
SWH            9615.385 Hz
FIDRES         0.166673 Hz
AQ            2.9999299 sec
RG                   161
DW                52.000 usec
DE                 14.54 usec
TE                 298.0 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        600.1342009 MHz
NUC1                  1H
P1                  8.00 usec

F2 - Processing parameters
SI                 65536
SF           600.1300351 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 12.00 cm
F1P                8.000 ppm
F1               4801.04 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.35088 ppm/cm
HZCM           210.57195 Hz/cm
```

13C spectrum with 1H decoupling



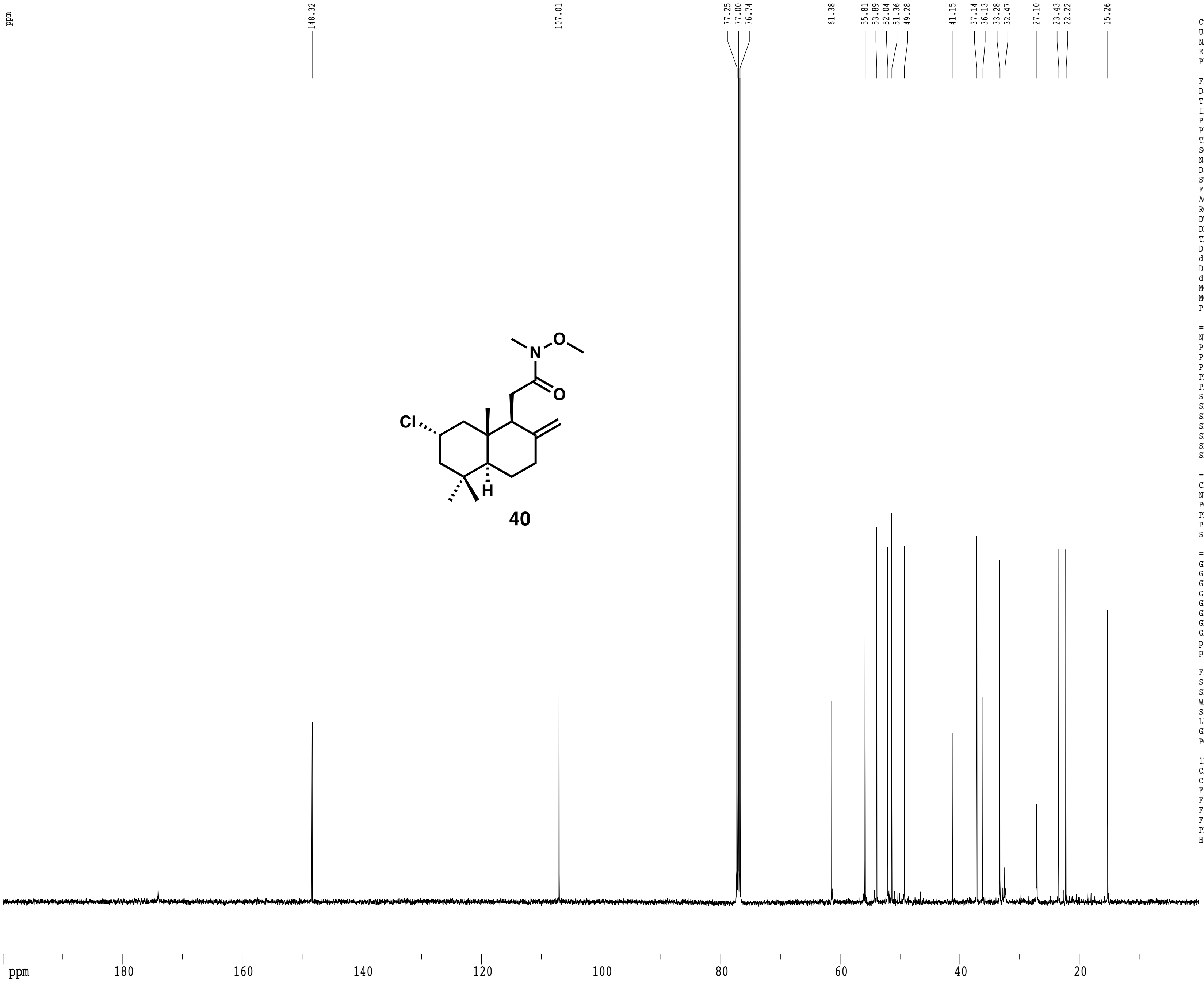
```
Current Data Parameters
USER              zkonst
NAME                2-43
EXPNO                 17
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140226
Time               21.32
INSTRUM            gn500
PROBHD   5 mm broadband
PULPROG          zgdc30
TD                181814
SOLVENT            CDCl3
NS                  2000
DS                     4
SWH            30303.031 Hz
FIDRES          0.166671 Hz
AQ             2.9999809 sec
RG                 16384
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         125.5603801 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.20 dB
SFO2         499.2924964 MHz

F2 - Processing parameters
SI                 65536
SF           125.5465779 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 34.00 cm
F1P              200.000 ppm
F1              25109.32 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            8.77193 ppm/cm
HZCM          1101.28577 Hz/cm
```

1H spectrum



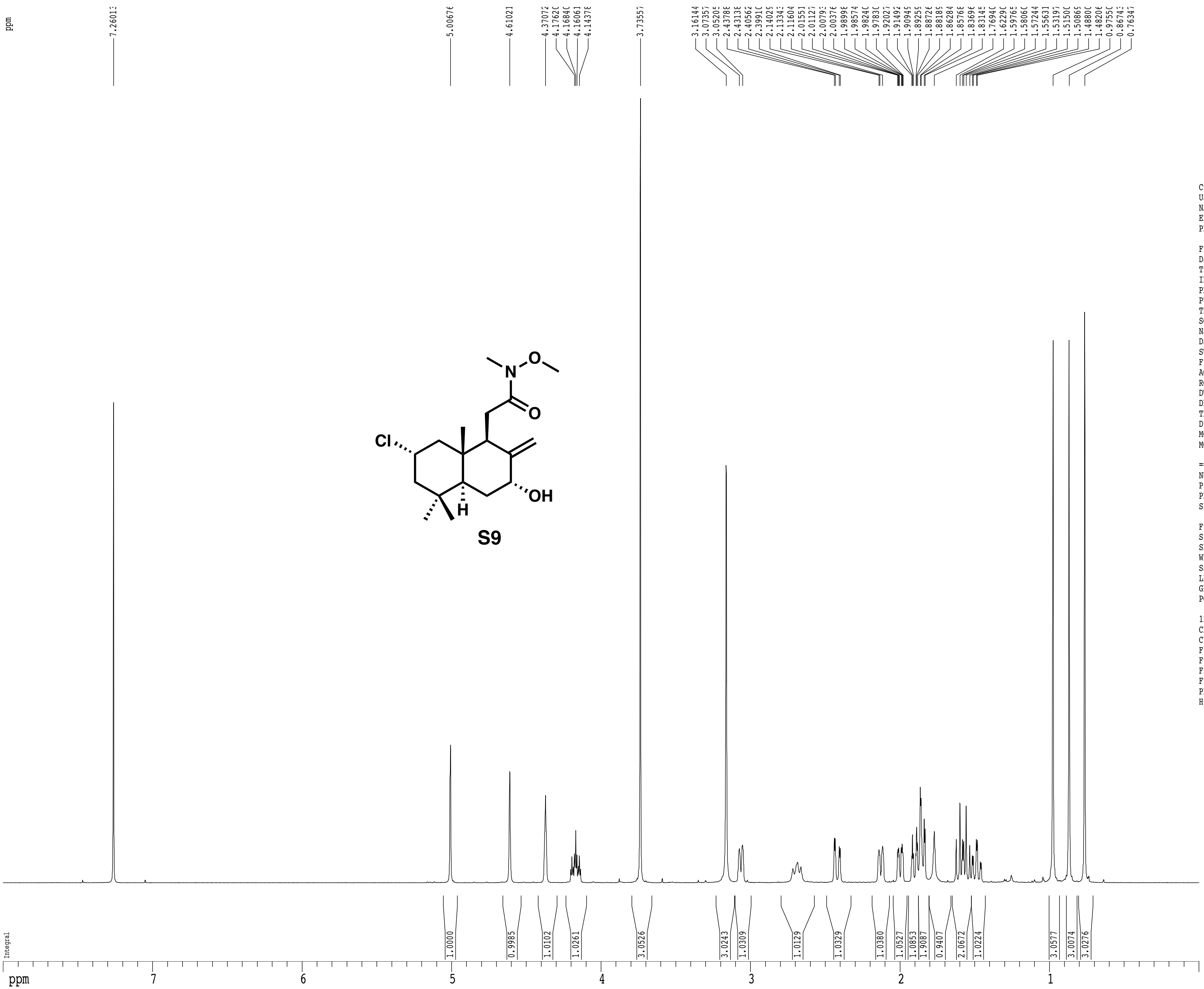
```
Current Data Parameters
USER              zkonst
NAME                2-46
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140228
Time               18.37
INSTRUM            av600
PROBHD   5 mm TBI 1H/13
PULPROG             zg30
TD                 57690
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             9615.385 Hz
FIDRES          0.166673 Hz
AQ             2.9999299 sec
RG                   161
DW                52.000 usec
DE                 14.54 usec
TE                 298.1 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        600.1342009 MHz
NUC1                  1H
P1                  8.00 usec

F2 - Processing parameters
SI                 65536
SF           600.1300340 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 12.00 cm
F1P                8.000 ppm
F1               4801.04 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.35088 ppm/cm
HZCM           210.57195 Hz/cm
```

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



```
Current Data Parameters
USER                zkonst
NAME                  2-46
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140228
Time                 21.05
INSTRUM            cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                   65536
SOLVENT              CDCl3
NS                    4000
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              1.00000000 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             125.7804286 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                   22.80 cm
CY                   18.00 cm
F1P                200.000 ppm
F1                25156.09 Hz
F2P                  0.000 ppm
F2                    0.00 Hz
PPMCM              8.77193 ppm/cm
HZCM            1103.33716 Hz/cm
```

1H spectrum



S9

Current Data Parameters
USER zkonst
NAME 3-123
EXPNO 9
PROCNO 1

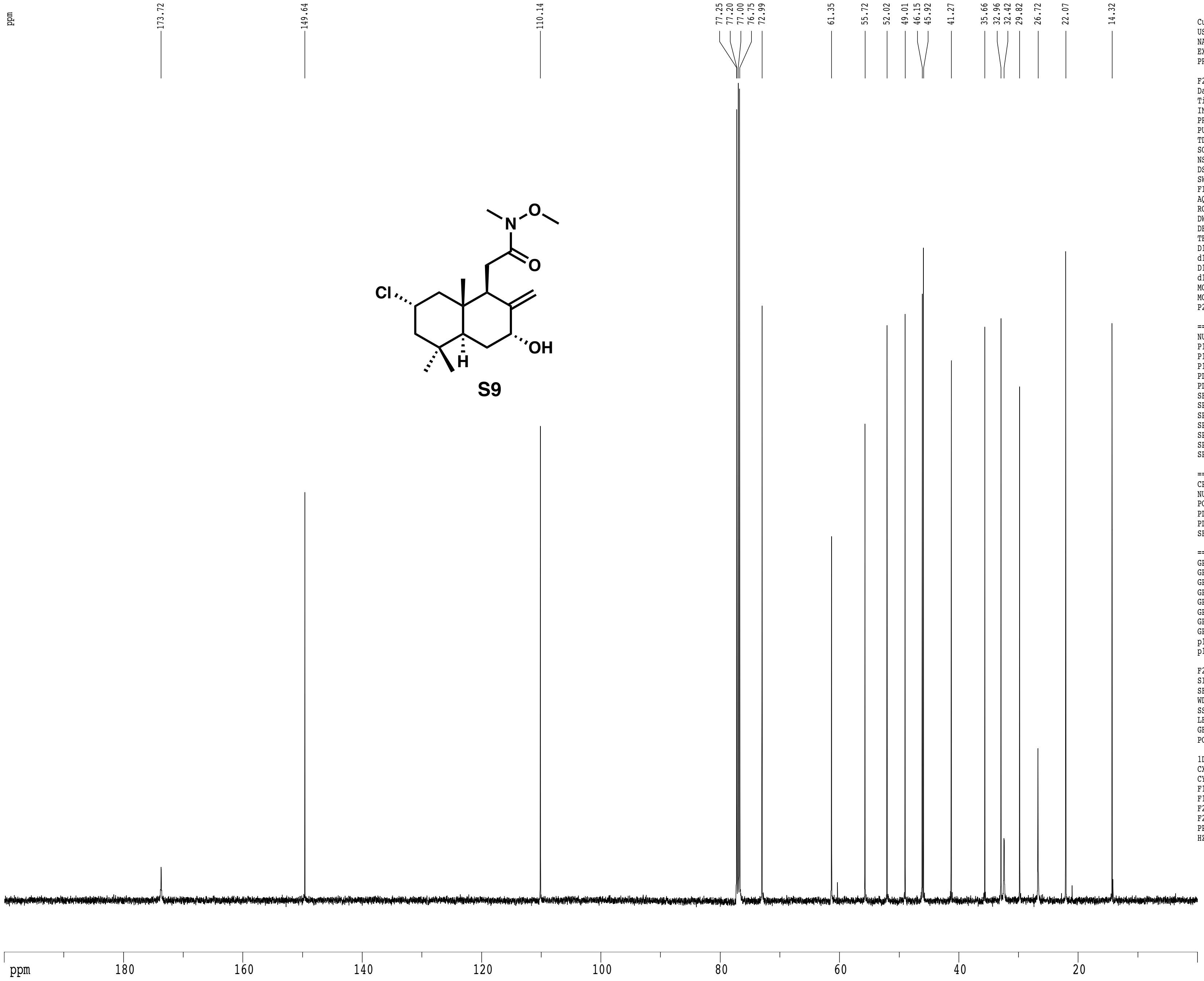
F2 - Acquisition Parameters
Date_ 20150818
Time 14.35
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 48074
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.166677 Hz
AQ 2.9999299 sec
RG 9
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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

F2 - Processing parameters
SI 65536
SF 500.2200312 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 8.000 ppm
F1 4001.76 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.51581 Hz/cm

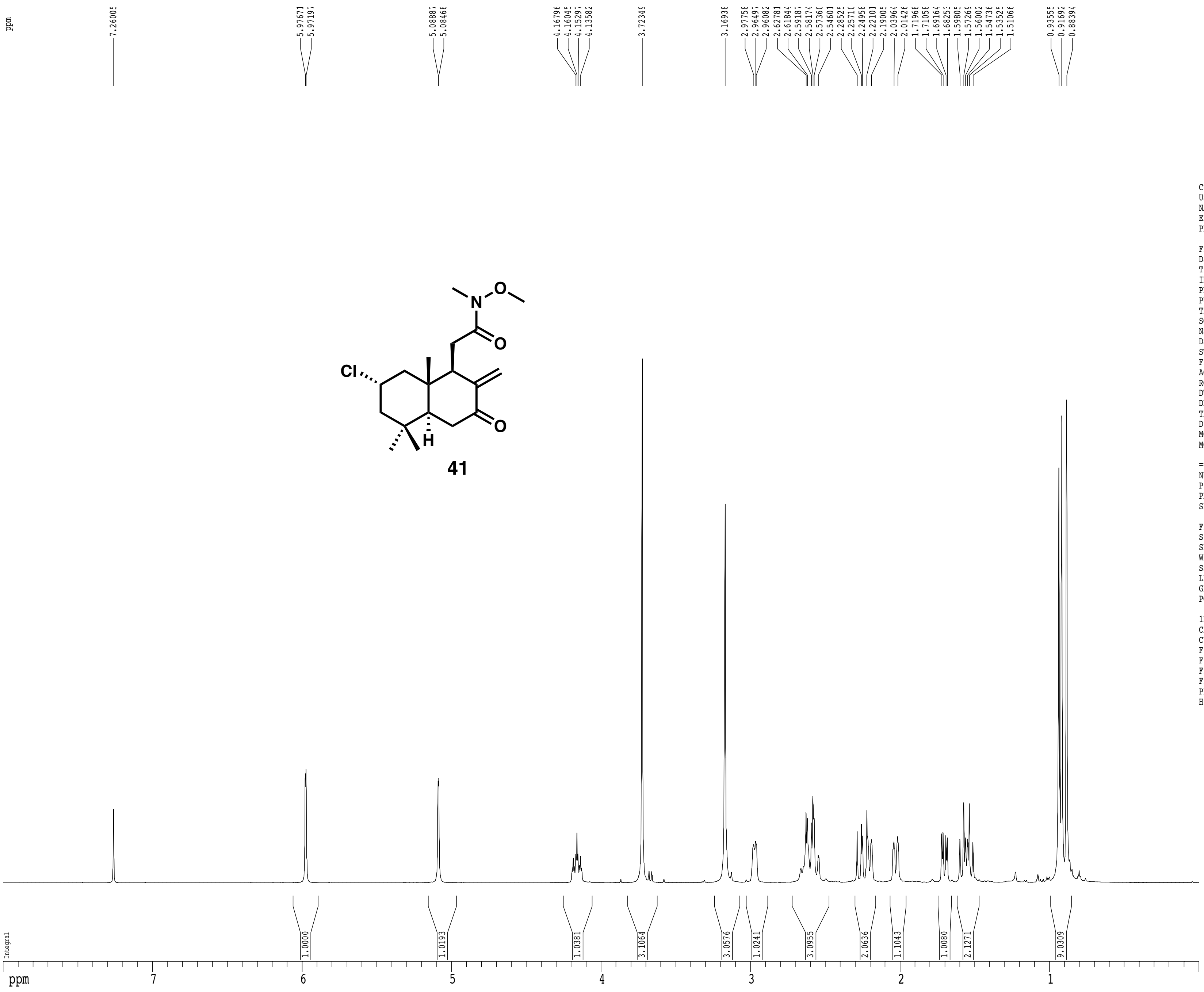
Z-restored spin-echo 13C spectrum with 1H decoupling



S9

```
Current Data Parameters
USER              zkonst
NAME               3-123
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150905
Time               11.49
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   480
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0814105 sec
RG                  4096
DW                16.500 usec
DE                  6.00 usec
TE                 298.0 K
D1            1.50000000 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           125.7804314 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              200.000 ppm
F1              25156.09 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            8.77193 ppm/cm
HZCM          1103.33716 Hz/cm
```

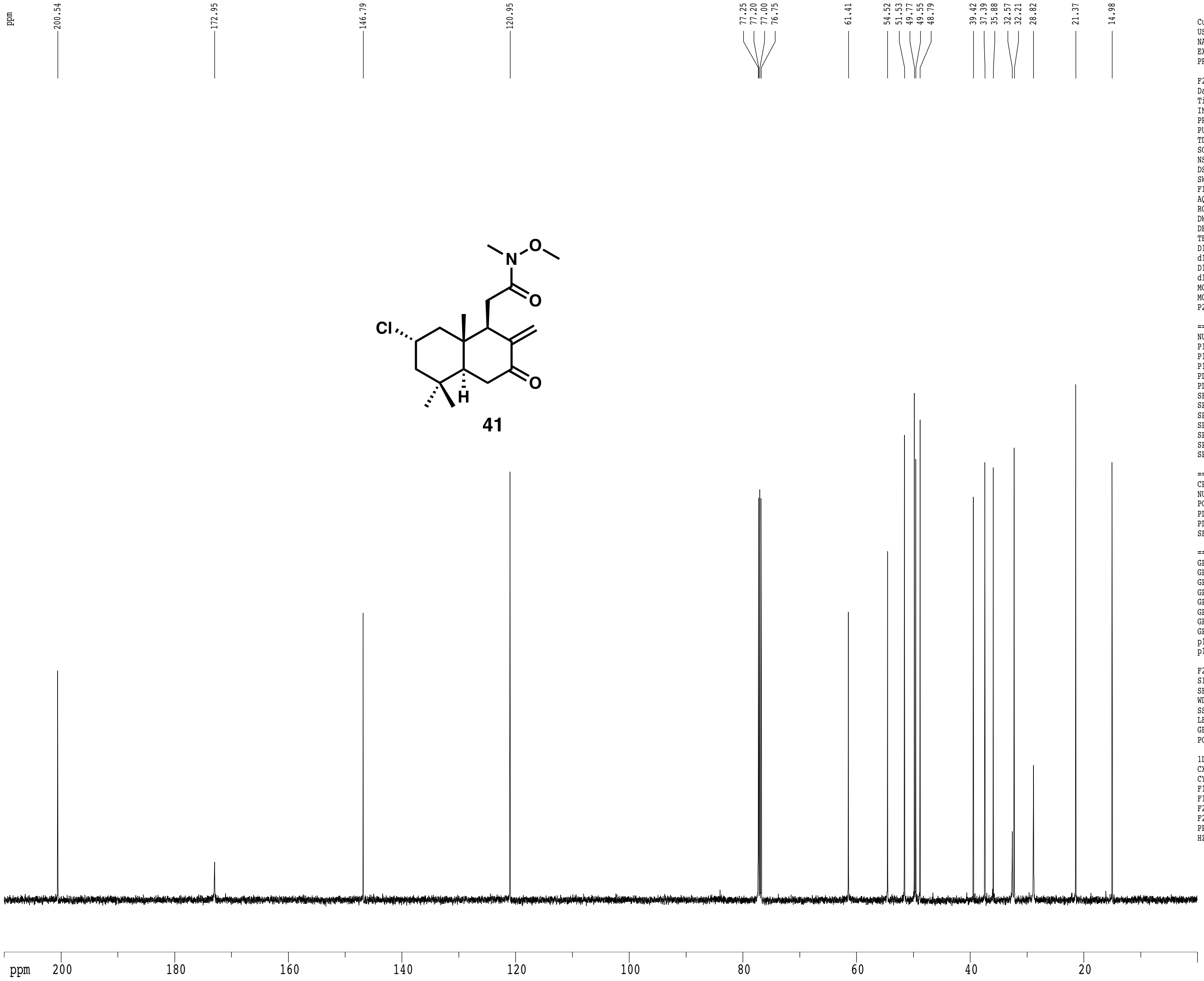
1H spectrum



41

```
Current Data Parameters
USER              zkonst
NAME               3-147
EXPNO                  9
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150910
Time               14.09
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 32048
SOLVENT            CDCl3
NS                     8
DS                     0
SWH          8012.820 Hz
FIDRES       0.250026 Hz
AQ          1.9998451 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        500.2200312 MHz
WDW                   EM
SSB                    0
LB               0.30 Hz
GB                     0
PC                  4.00

1D NMR plot parameters
CX               22.80 cm
CY               10.00 cm
F1P              8.000 ppm
F1             4001.76 Hz
F2P              0.000 ppm
F2                0.00 Hz
PPMCM          0.35088 ppm/cm
HZCM         175.51581 Hz/cm
```

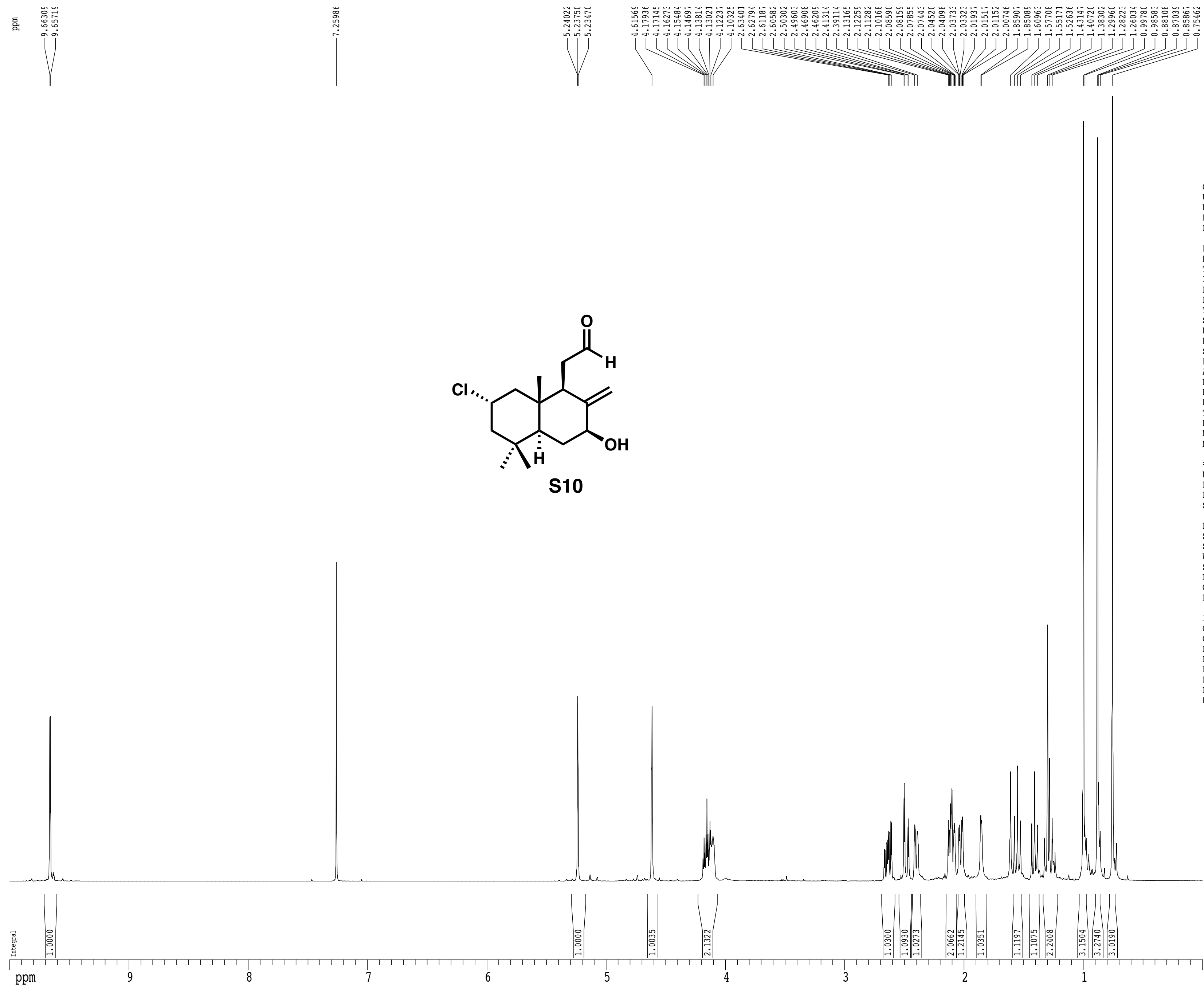
Z-restored spin-echo 13C spectrum with 1H decoupling



41

```
Current Data Parameters
USER              zkonst
NAME               3-147
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150910
Time               14.15
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   176
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0814105 sec
RG                9195.2
DW                16.500 usec
DE                  6.00 usec
TE                 298.0 K
D1            1.50000000 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           125.7804337 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              210.000 ppm
F1              26413.89 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            9.21053 ppm/cm
HZCM          1158.50391 Hz/cm
```

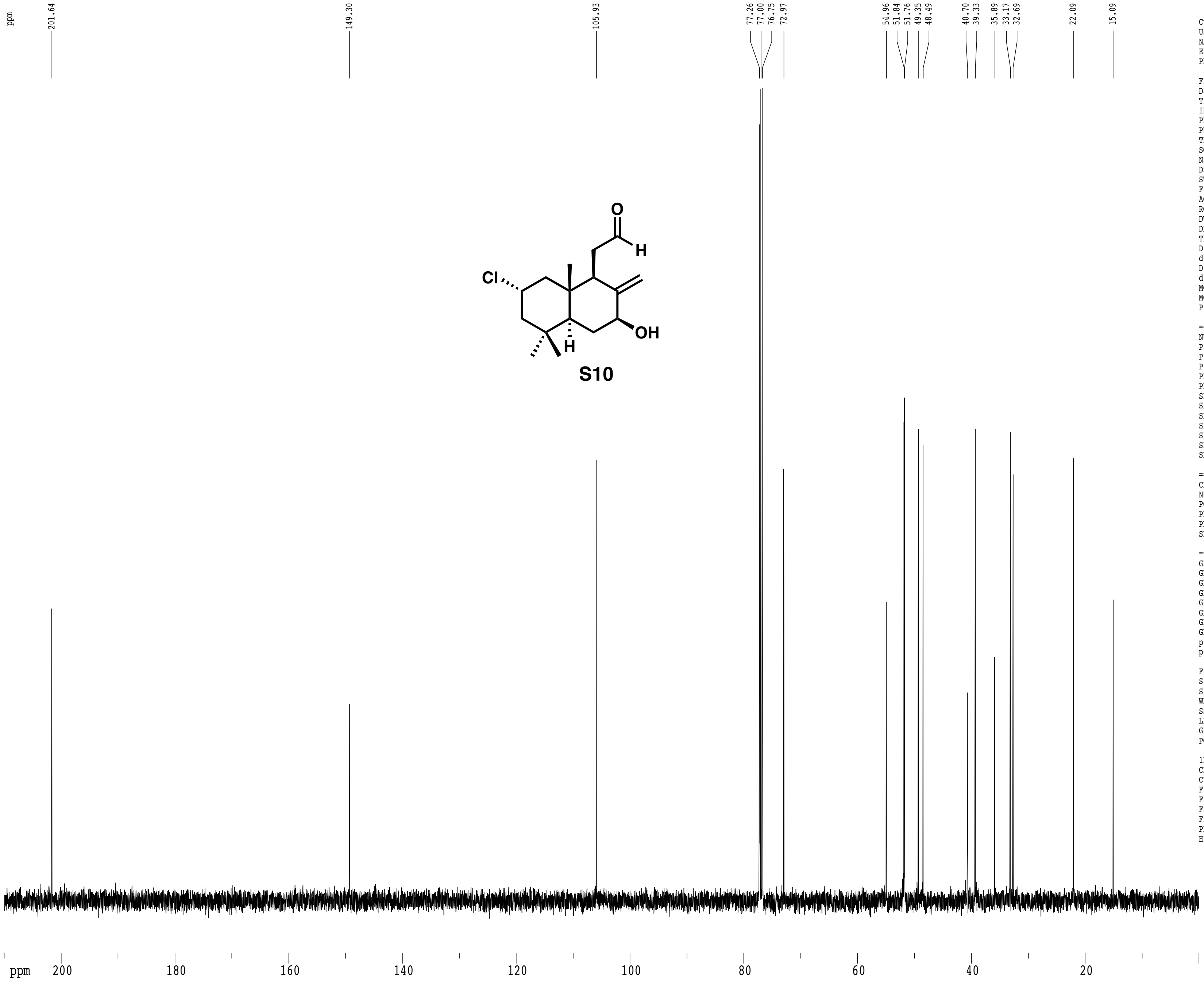
1H spectrum



S10

```
Current Data Parameters
USER              zkonst
NAME               3-137
EXPNO                 17
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150819
Time               13.44
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 48074
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8012.820 Hz
FIDRES          0.166677 Hz
AQ             2.9999299 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           500.2200312 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               10.000 ppm
F1               5002.20 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.43860 ppm/cm
HZCM           219.39476 Hz/cm
```

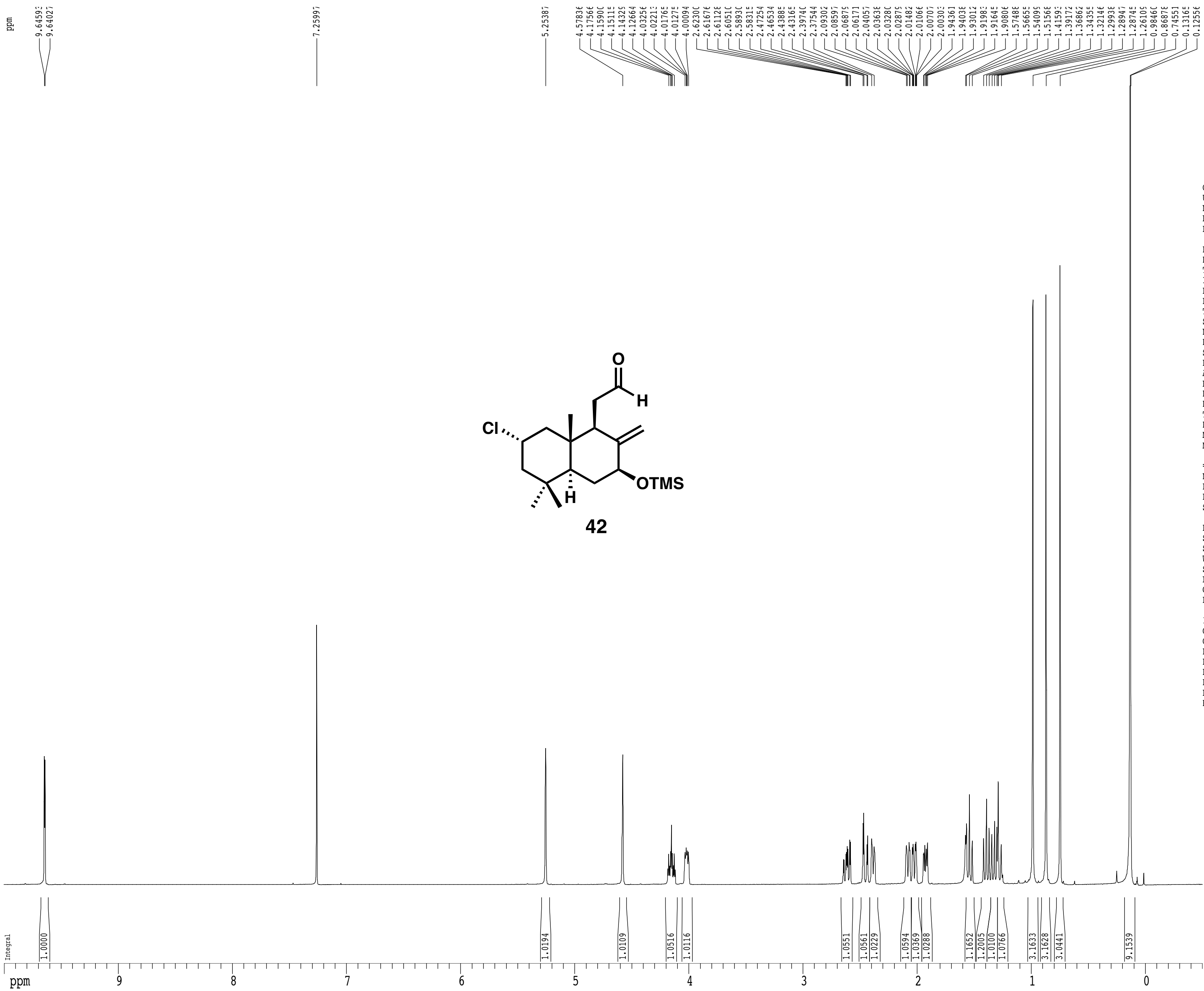
Z-restored spin-echo 13C spectrum with 1H decoupling



S10

```
Current Data Parameters
USER              zkonst
NAME               3-137
EXPNO                 19
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150819
Time               13.50
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   224
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0814105 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                 33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 16.55 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 2.70 dB
SP2                 2.70 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               24.50 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           125.7804291 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.65 cm
F1P              210.000 ppm
F1              26413.89 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            9.21053 ppm/cm
HZCM          1158.50391 Hz/cm
```

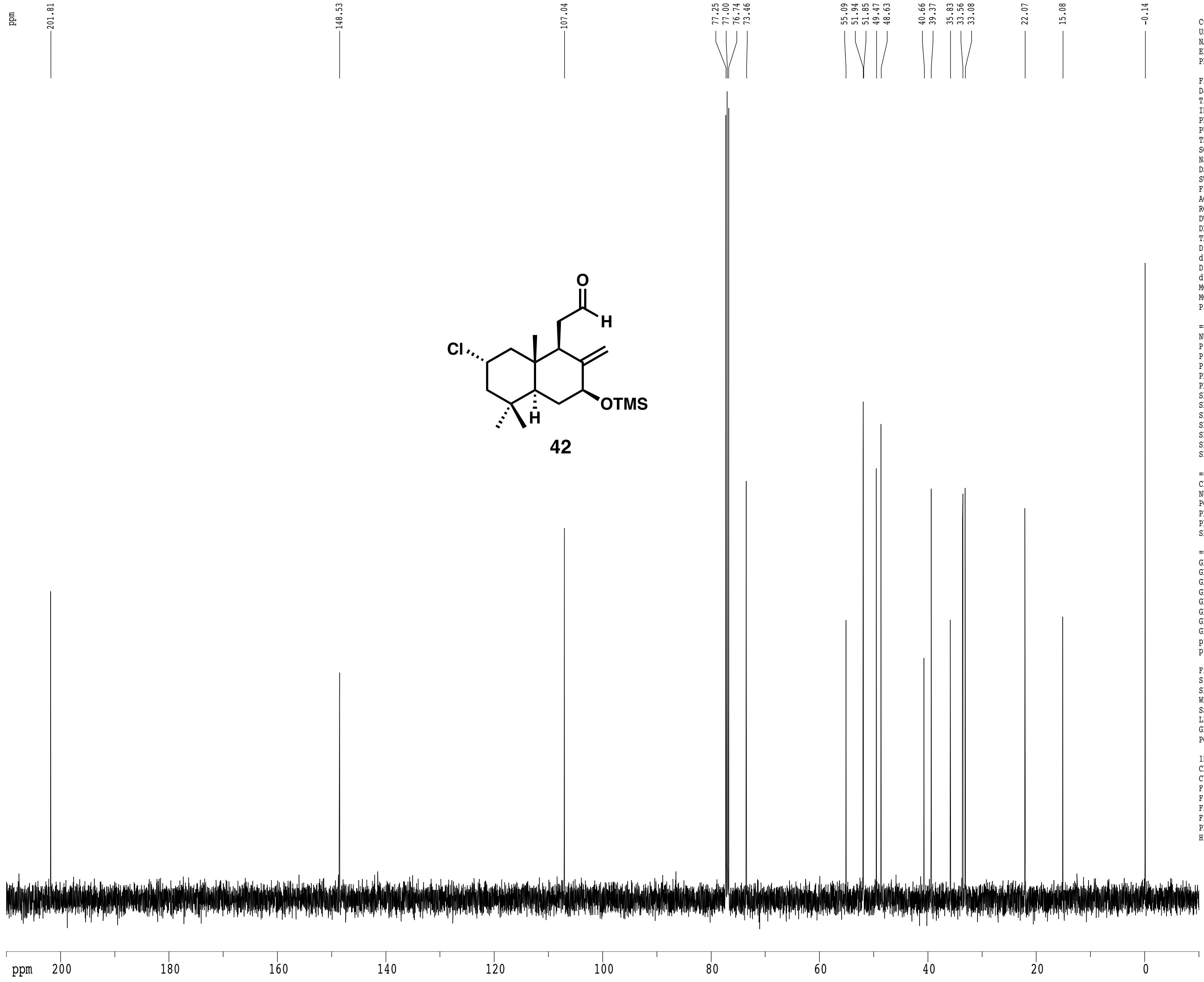
1H spectrum



42

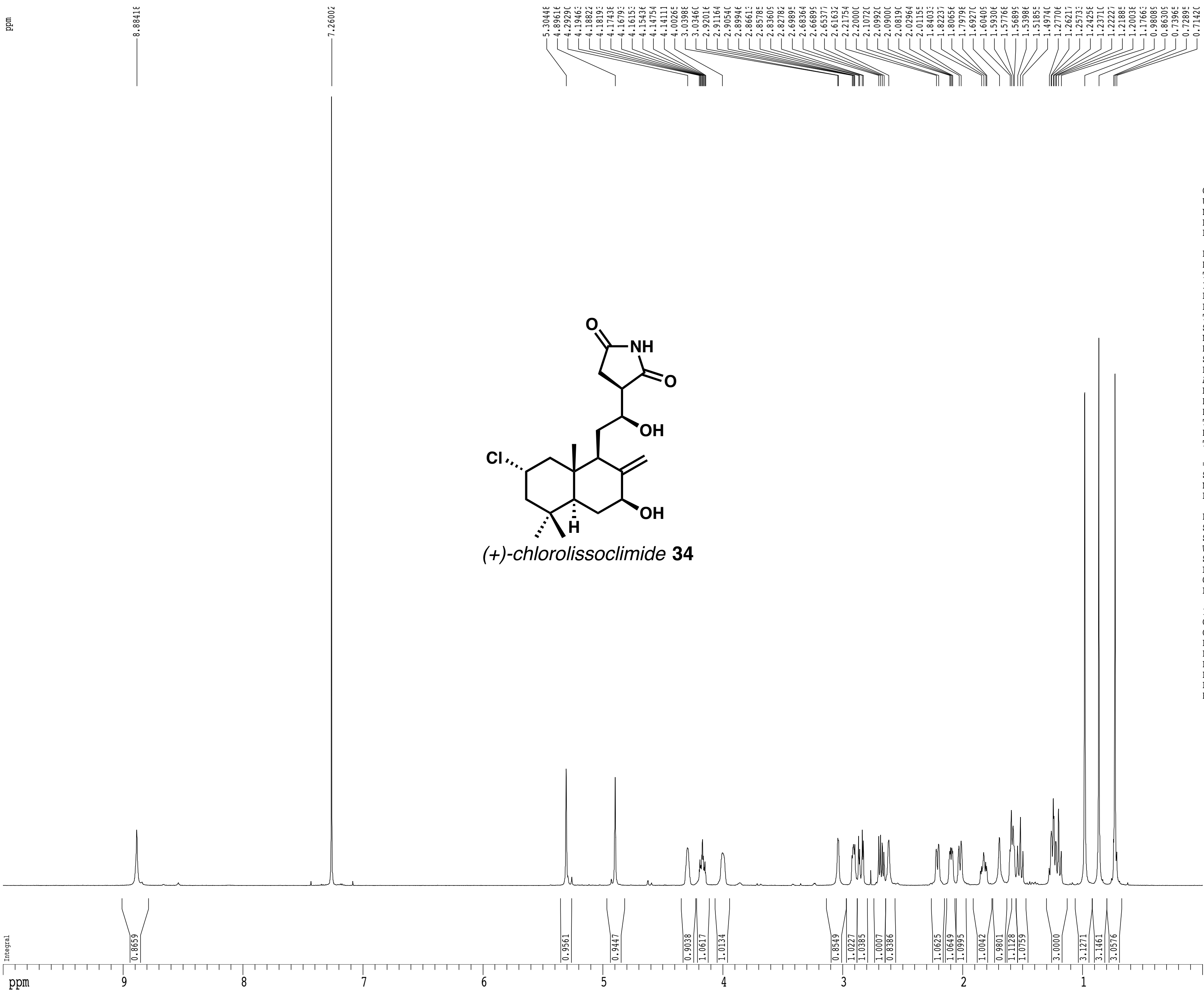
```
Current Data Parameters
USER               zkonst
NAME                3-134
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150818
Time                12.17
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG              zg30
TD                  48074
SOLVENT             CDCl3
NS                     16
DS                      0
SWH             8012.820 Hz
FIDRES          0.166677 Hz
AQ             2.9999299 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            500.2200313 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   4.00

1D NMR plot parameters
CX                  22.80 cm
CY                  40.00 cm
F1P                10.000 ppm
F1                5002.20 Hz
F2P                -0.500 ppm
F2                -250.11 Hz
PPMCM             0.46053 ppm/cm
HZCM            230.36450 Hz/cm
```

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



```
Current Data Parameters
USER              zkonst
NAME               3-134
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150818
Time               12.21
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                    80
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0814105 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                 33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 16.55 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 2.70 dB
SP2                 2.70 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               24.50 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           125.7804286 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

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

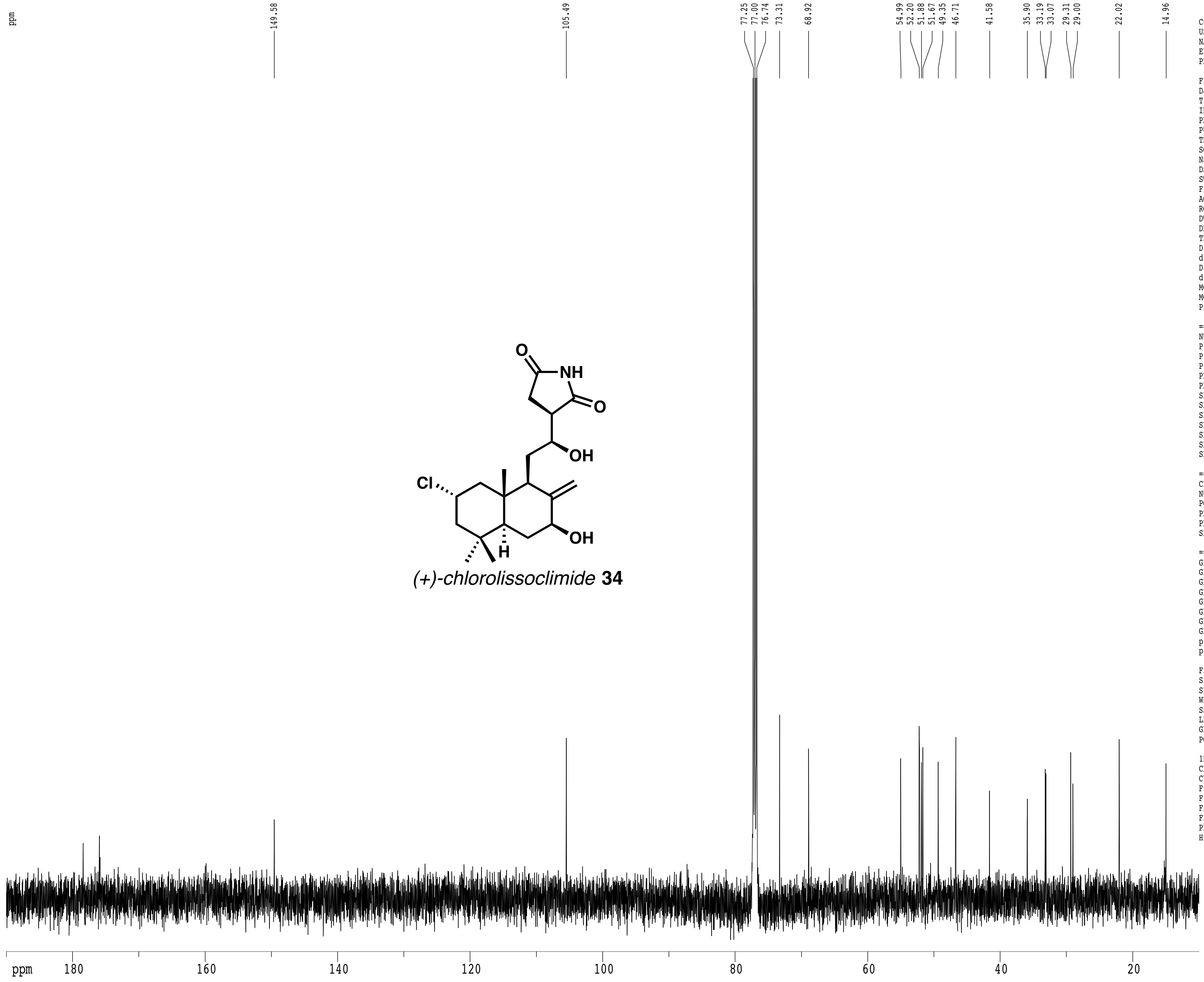
1H spectrum



(+)-chlorolissoclimide **34**

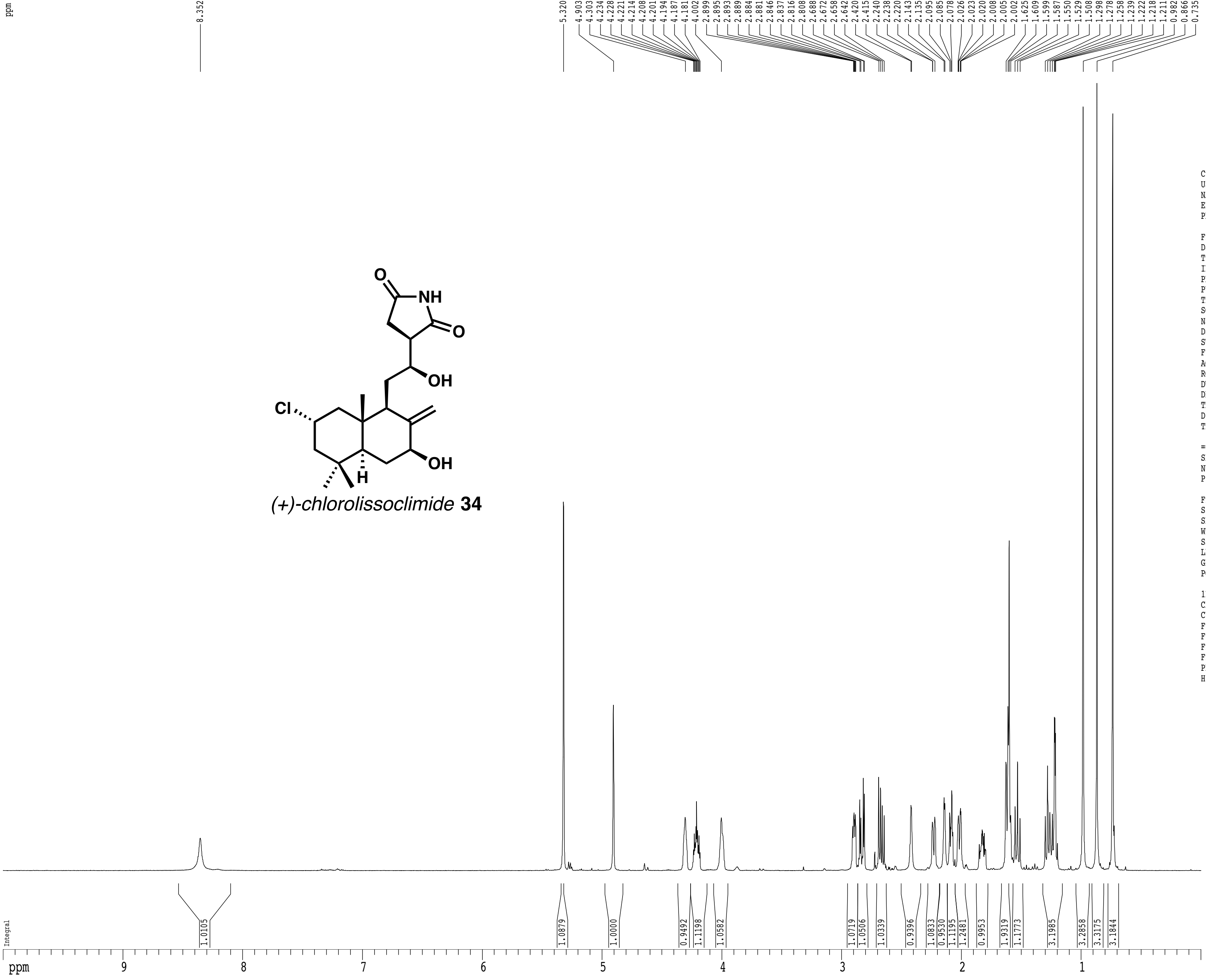
```
Current Data Parameters
USER             zkonst
NAME              3-141
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20150903
Time               9.06
INSTRUM           av600
PROBHD   5 mm TBI 1H/13
PULPROG            zg30
TD                57690
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            9615.385 Hz
FIDRES         0.166673 Hz
AQ            2.9999299 sec
RG                  256
DW               52.000 usec
DE                14.54 usec
TE                298.0 K
D1           0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        600.1342009 MHz
NUC1                 1H
P1                 8.00 usec

F2 - Processing parameters
SI                65536
SF          600.1300357 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P              10.000 ppm
F1              6001.30 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM           0.43860 ppm/cm
HZCM          263.21494 Hz/cm
```

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


(+)-chlorolissoclimide **34**

```
Current Data Parameters
USER              zkonst
NAME               2-235
EXPNO                309
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141216
Time               19.27
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                  2590
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0814105 sec
RG                7298.2
DW                16.500 usec
DE                  6.00 usec
TE                 298.0 K
D1            1.00000000 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           125.7804291 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                130.00 cm
F1P              190.000 ppm
F1              23898.28 Hz
F2P               10.000 ppm
F2               1257.81 Hz
PPMCM            7.89474 ppm/cm
HZCM           993.00342 Hz/cm
```

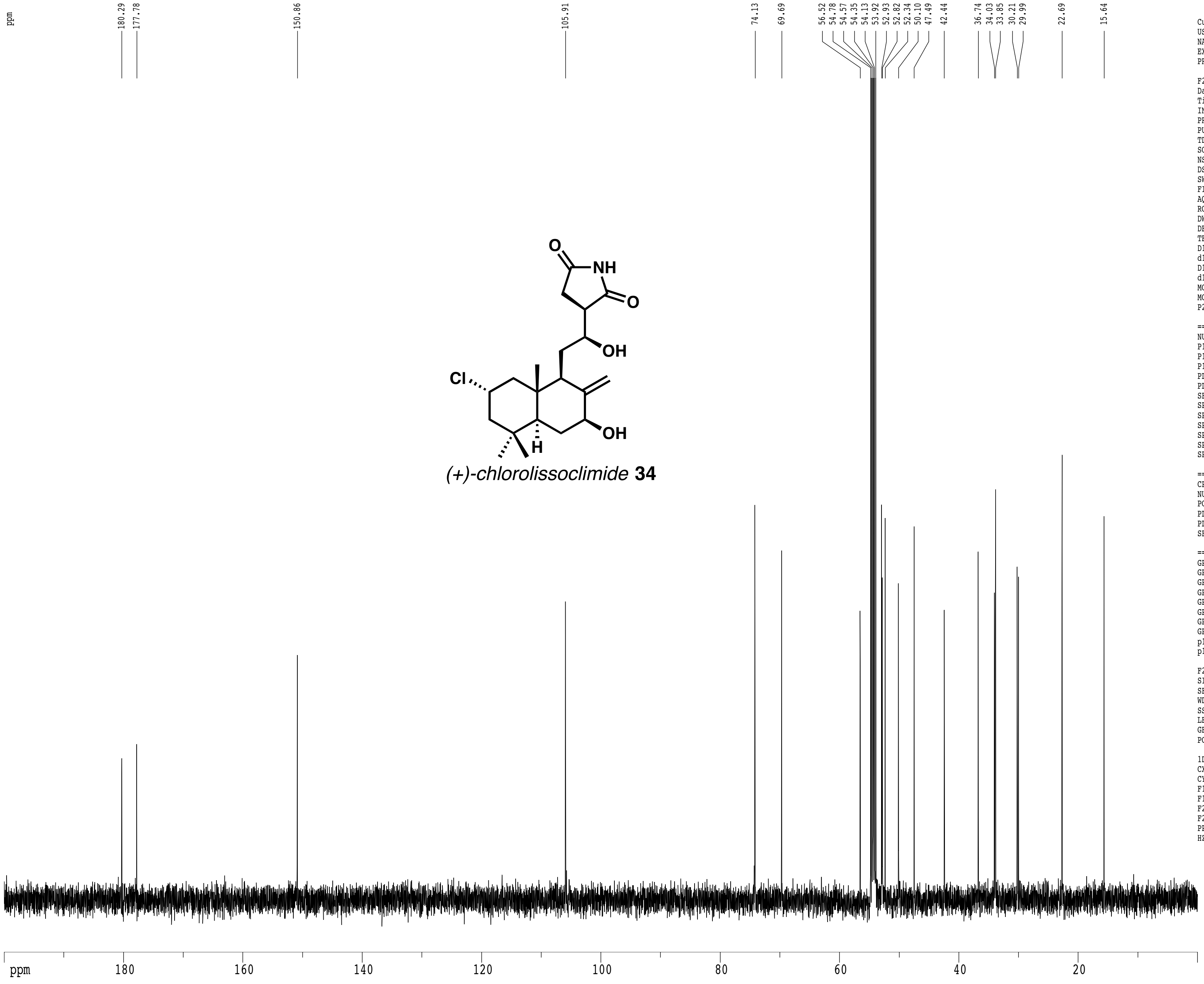
1H spectrum



(+)-chlorolissoclimide **34**

```
Current Data Parameters
USER              zkonst
NAME               3-141
EXPNO                 12
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150903
Time               12.36
INSTRUM            av600
PROBHD   5 mm TBI 1H/13
PULPROG             zg30
TD                 57690
SOLVENT            CD2Cl2
NS                    16
DS                     2
SWH             9615.385 Hz
FIDRES          0.166673 Hz
AQ             2.9999299 sec
RG                   256
DW                52.000 usec
DE                 14.54 usec
TE                 298.0 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1342009 MHz
NUC1                  1H
P1                  8.00 usec

F2 - Processing parameters
SI                 65536
SF           600.1300256 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P               10.000 ppm
F1               6001.30 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.43860 ppm/cm
HZCM           263.21494 Hz/cm
```

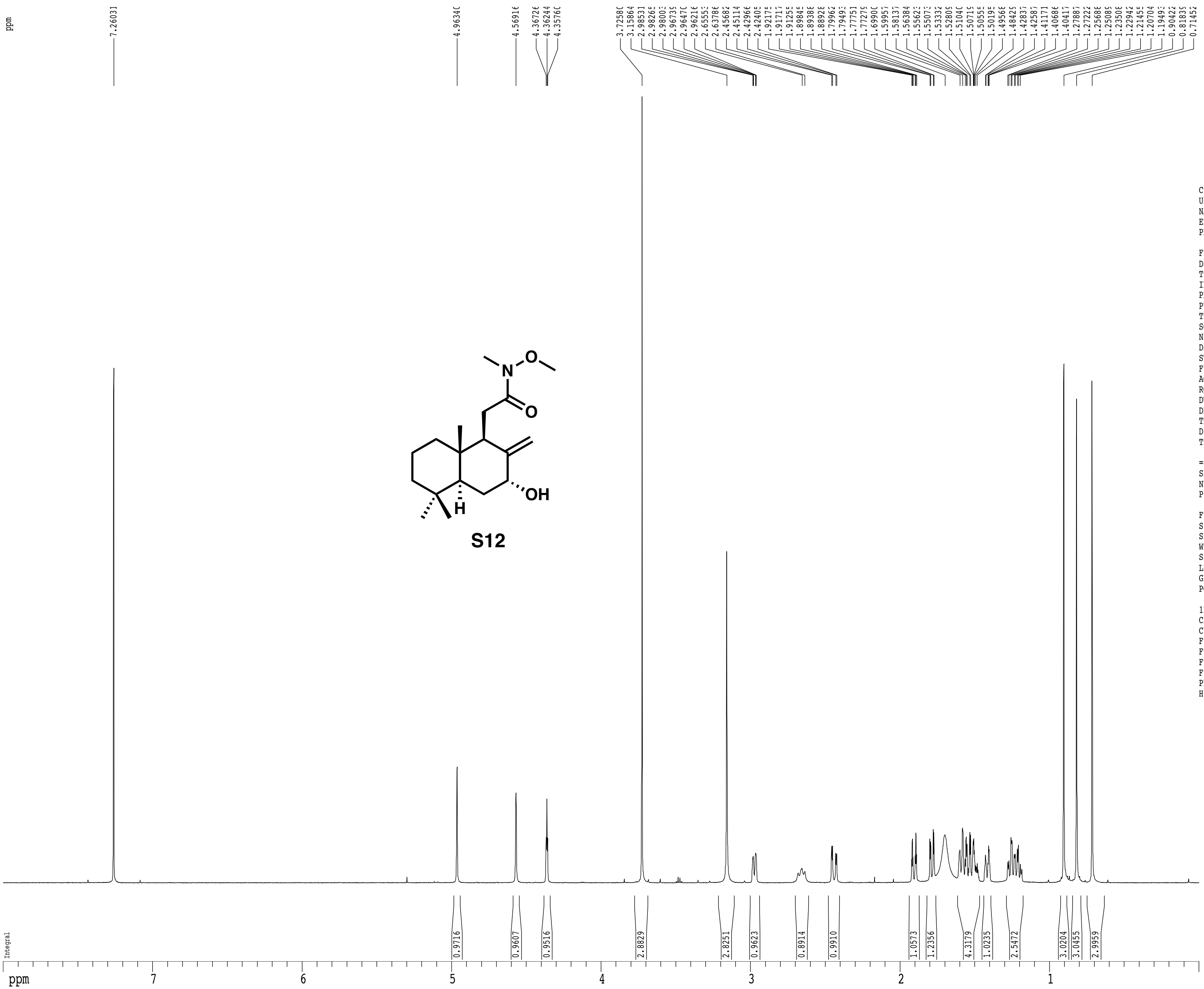
Z-restored spin-echo 13C spectrum with 1H decoupling



(+)-chlorolissoclimide **34**

```
Current Data Parameters
USER              zkonst
NAME               3-141
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_          20150903
Time              12.56
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT          CD2Cl2
NS                  616
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           1.00000000 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          125.7803067 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                60.00 cm
F1P             200.000 ppm
F1             25156.06 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM           8.77193 ppm/cm
HZCM         1103.33606 Hz/cm
```

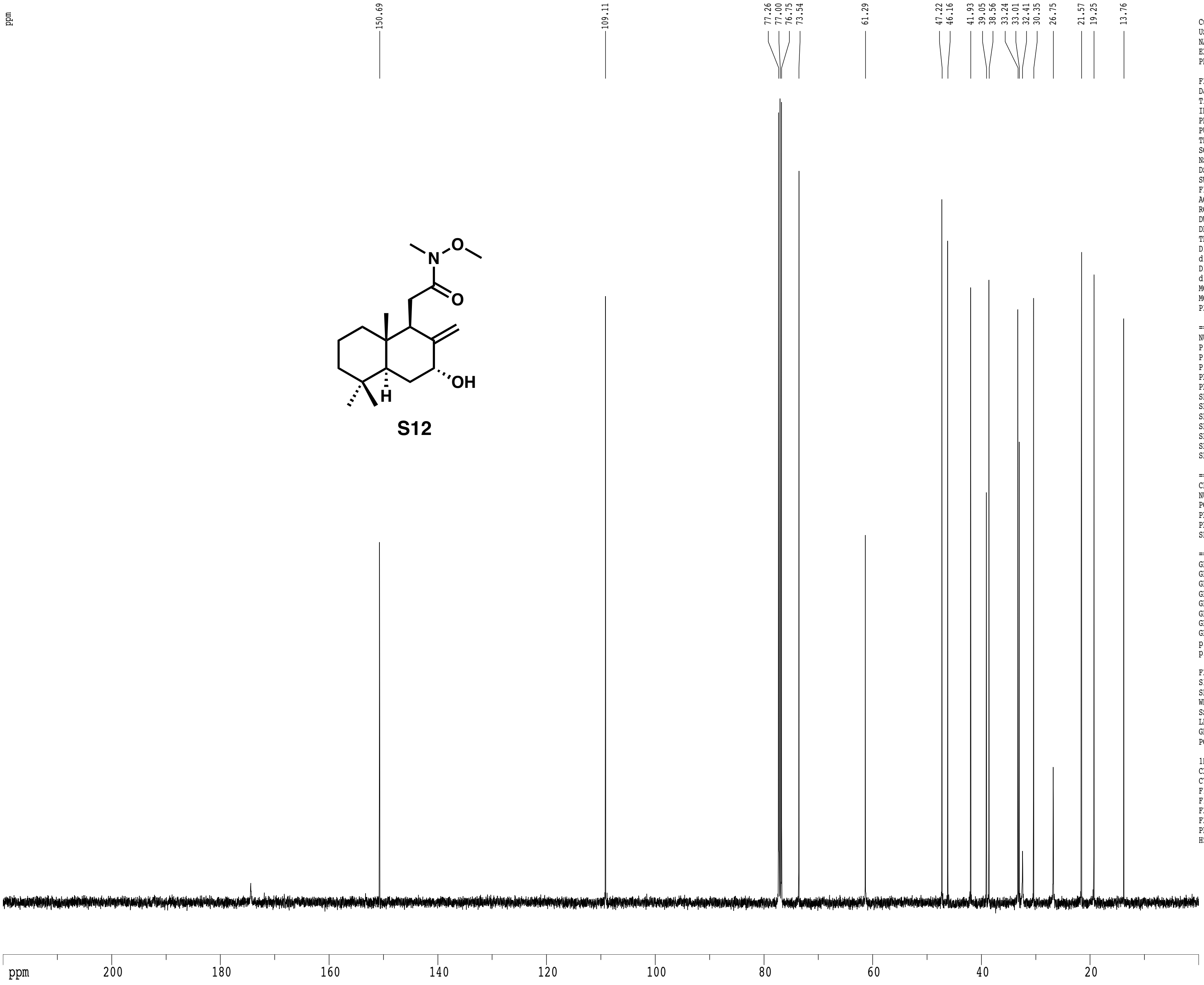
1H spectrum



S12

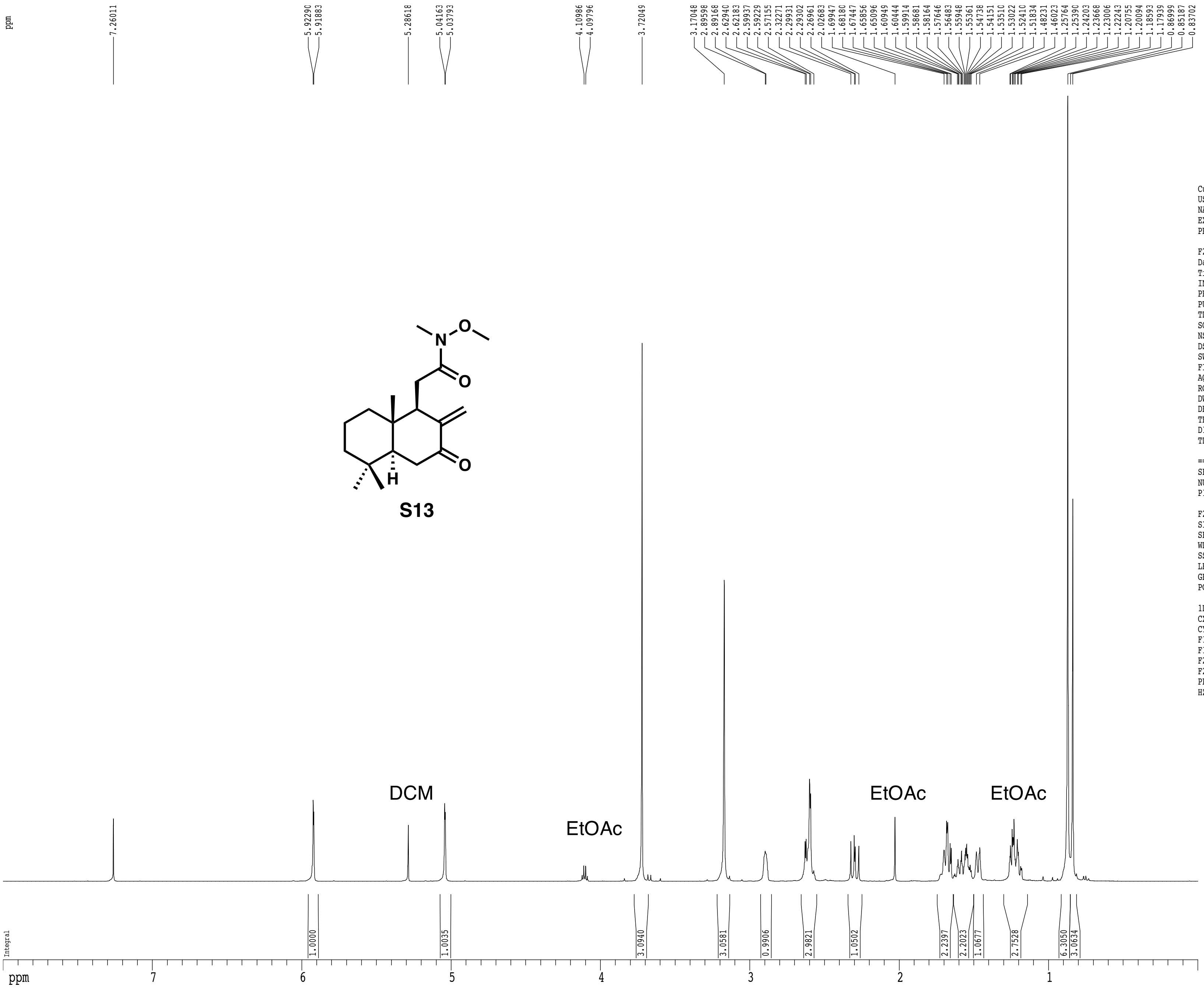
```
Current Data Parameters
USER              michal
NAME           SEM-1-272
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150817
Time               18.26
INSTRUM            av600
PROBHD   5 mm BBO BB-1H
PULPROG             zg30
TD                 38460
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             9615.385 Hz
FIDRES          0.250010 Hz
AQ             1.9999700 sec
RG                   575
DW                52.000 usec
DE                 14.33 usec
TE                 298.2 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        600.1342009 MHz
NUC1                  1H
P1                  9.00 usec

F2 - Processing parameters
SI                 65536
SF          600.1300343 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                8.000 ppm
F1               4801.04 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.35088 ppm/cm
HZCM           210.57195 Hz/cm
```

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



```
Current Data Parameters
USER               michal
NAME     allylic alcohol-ZK
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150903
Time                17.17
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                  65536
SOLVENT             CDCl3
NS                    396
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             1.50000000 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            125.7804305 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               27671.70 Hz
F2P                 0.000 ppm
F2                   0.00 Hz
PPMCM             9.64912 ppm/cm
HZCM           1213.67090 Hz/cm
```

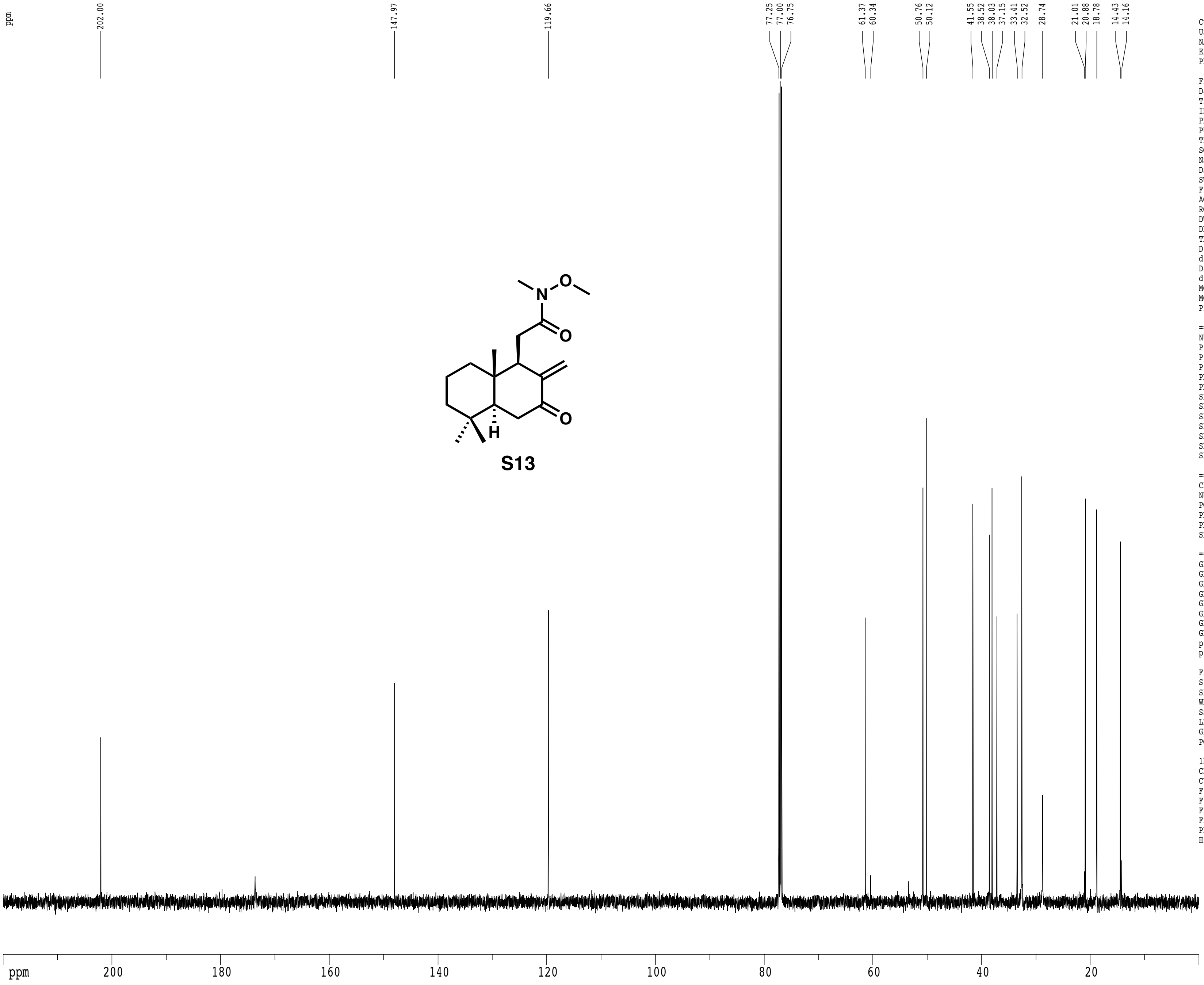
1H spectrum



S13

```
Current Data Parameters
USER              michal
NAME          SEM-1-298
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150904
Time               20.06
INSTRUM            av600
PROBHD   5 mm TBI 1H/13
PULPROG             zg30
TD                 98074
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             9615.385 Hz
FIDRES          0.098042 Hz
AQ             5.0998979 sec
RG                   144
DW                52.000 usec
DE                 14.54 usec
TE                 298.0 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1342009 MHz
NUC1                  1H
P1                  8.00 usec

F2 - Processing parameters
SI                 65536
SF           600.1300356 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                8.000 ppm
F1               4801.04 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.35088 ppm/cm
HZCM           210.57196 Hz/cm
```

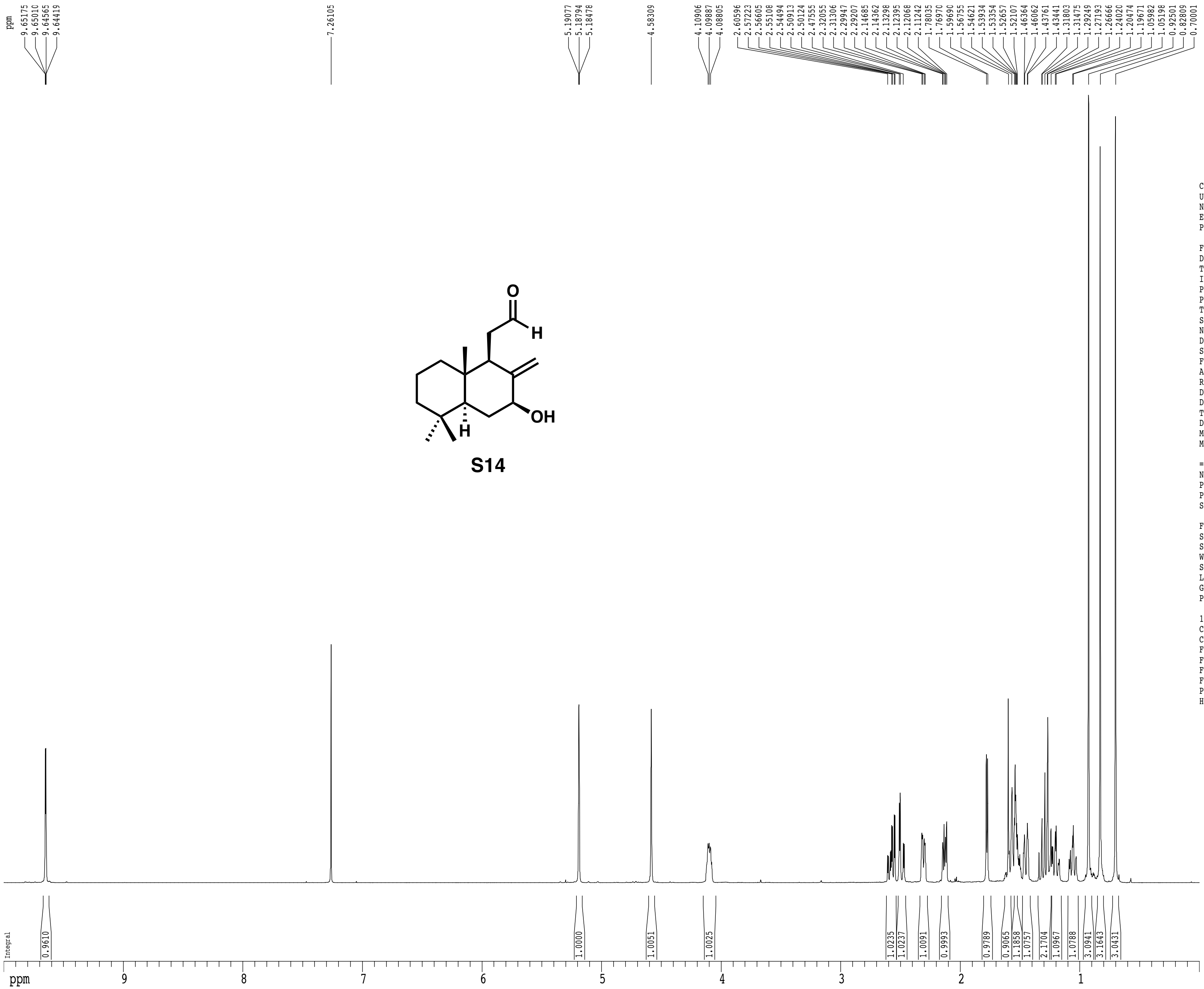
Z-restored spin-echo 13C spectrum with 1H decoupling



S13

```
Current Data Parameters
USER              michal
NAME           SEM-1-298
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150904
Time               19.45
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   137
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0814105 sec
RG                  2048
DW                16.500 usec
DE                  6.00 usec
TE                 298.0 K
D1            1.50000000 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           125.7804309 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              27671.70 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            9.64912 ppm/cm
HZCM          1213.67090 Hz/cm
```

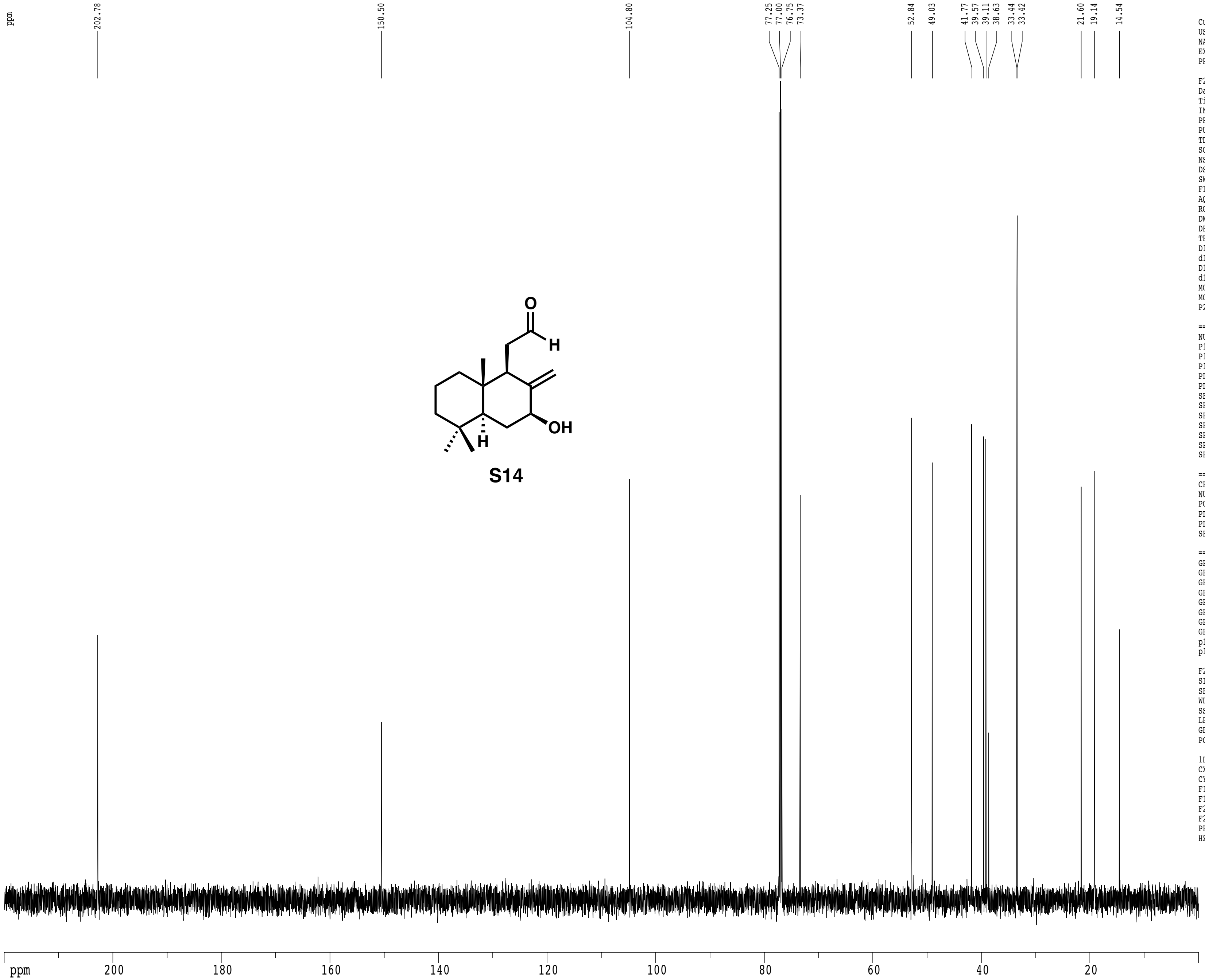
1H spectrum



S14

```
Current Data Parameters
USER              michal
NAME          SEM-1-284
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150819
Time               18.00
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 32048
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.250026 Hz
AQ             1.9998451 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           500.2200307 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  4.00

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

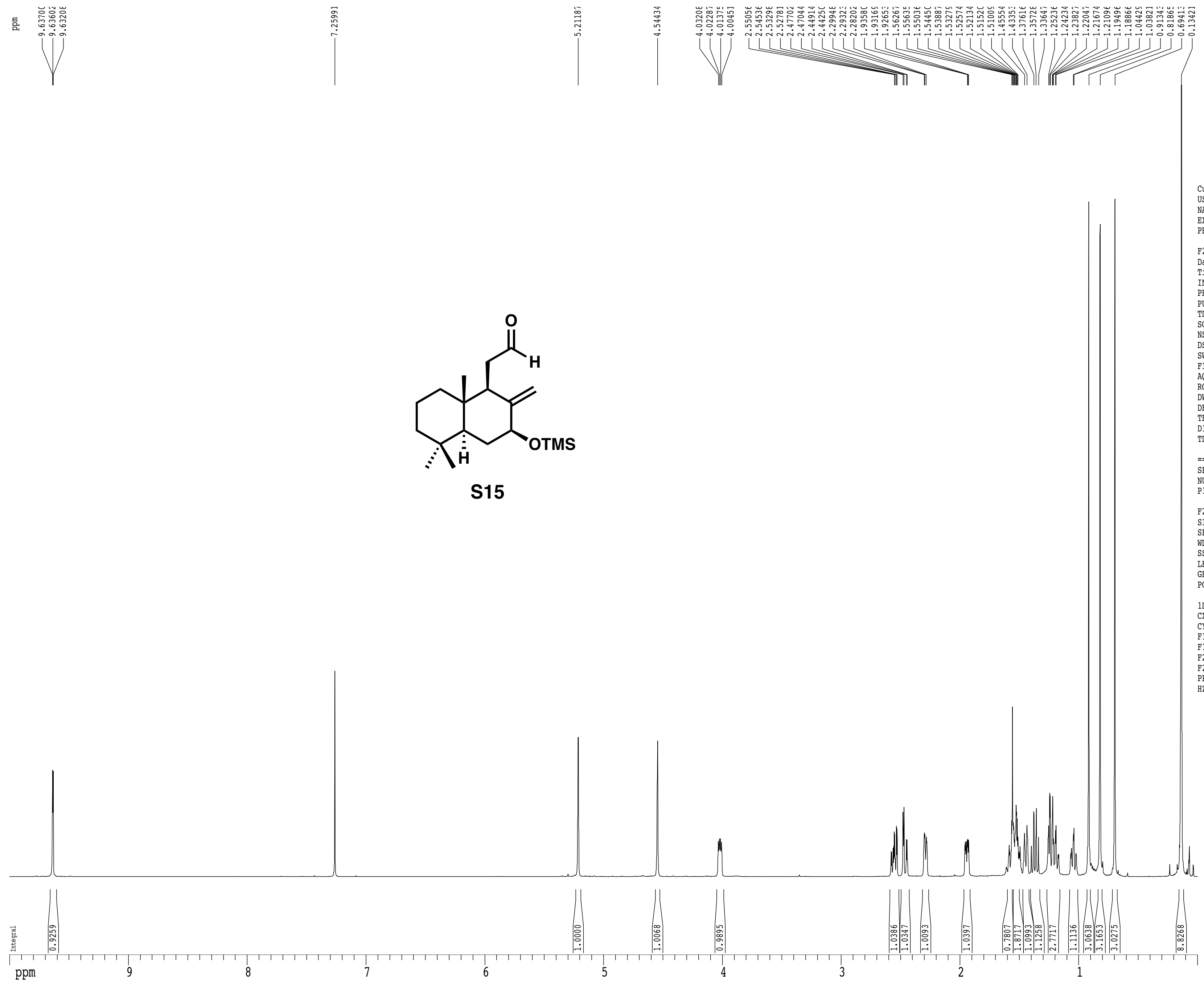
Z-restored spin-echo 13C spectrum with 1H decoupling



S14

```
Current Data Parameters
USER              michal
NAME         SEM-1-284-c
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150819
Time               18.02
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   106
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                 33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 16.55 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 2.70 dB
SP2                 2.70 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               24.50 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           125.7804286 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                0.000 ppm
F2                  0.00 Hz
PPMCM            9.64912 ppm/cm
HZCM          1213.67078 Hz/cm
```

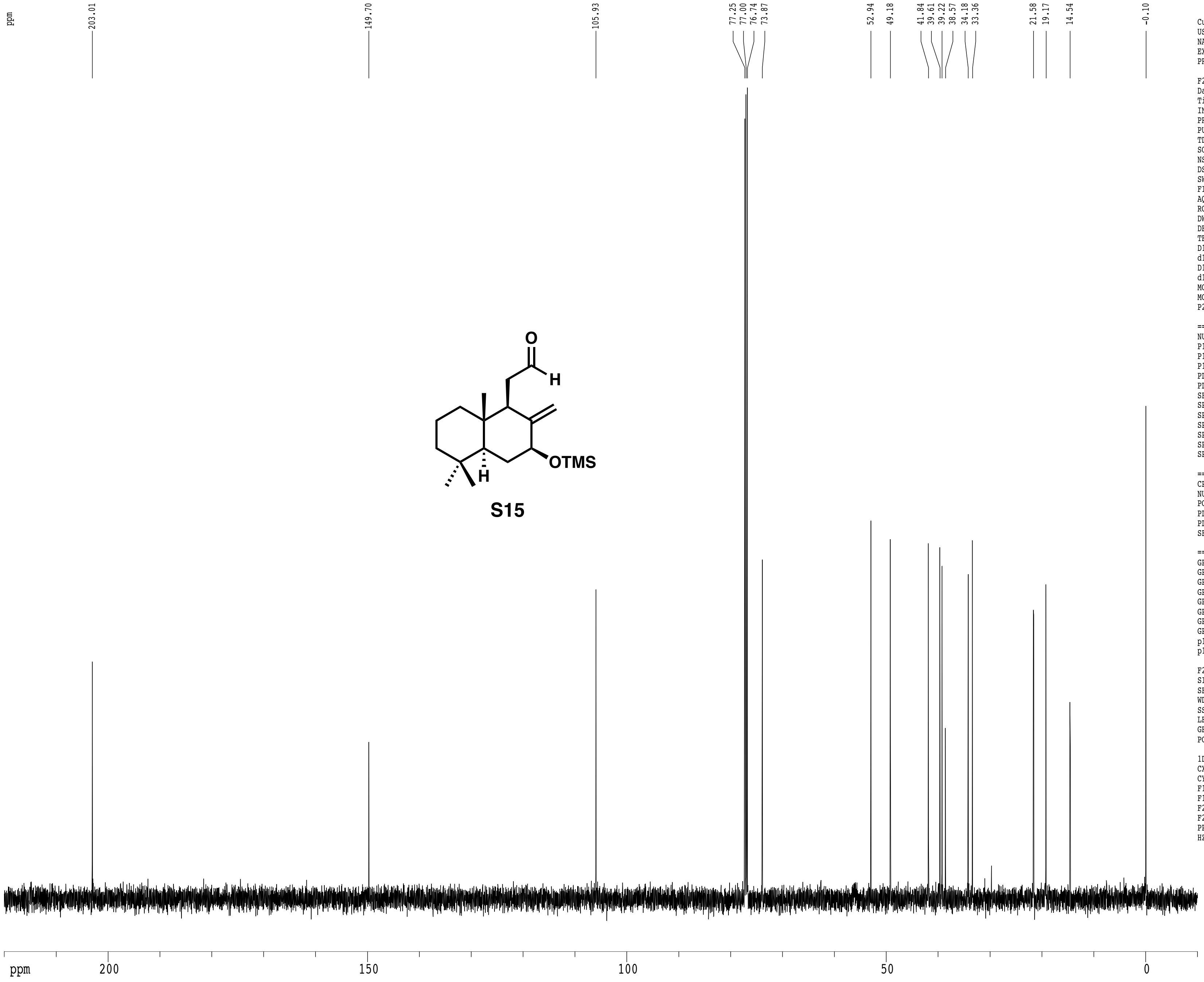
1H spectrum



S15

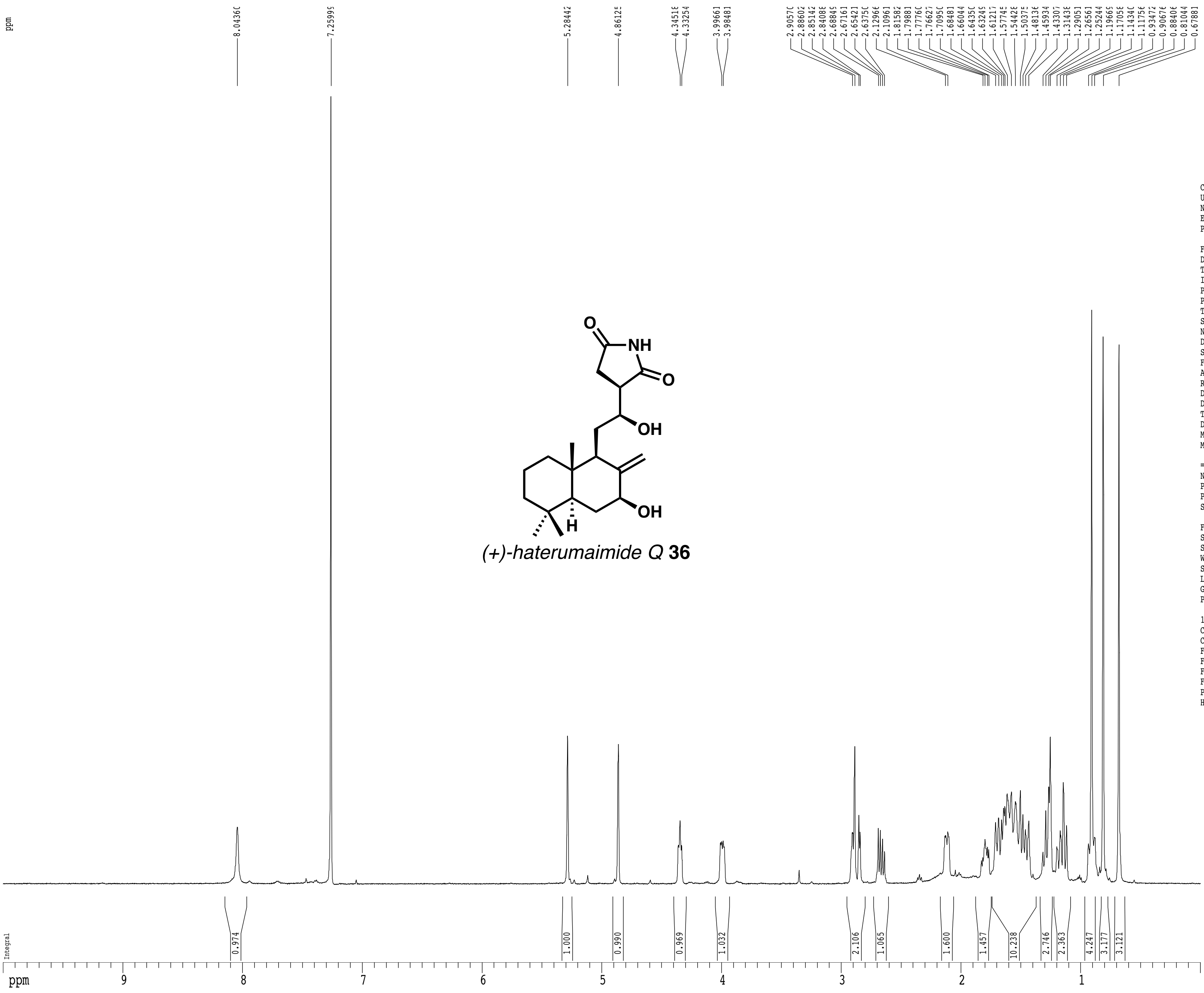
```
Current Data Parameters
USER             michal
NAME          SEM-1-286
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20150820
Time              19.36
INSTRUM           av600
PROBHD   5 mm TBI 1H/13
PULPROG            zg30
TD                38460
SOLVENT           CDCl3
NS                   24
DS                    2
SWH            9615.385 Hz
FIDRES         0.250010 Hz
AQ            1.9999700 sec
RG                  228
DW               52.000 usec
DE                14.54 usec
TE                298.0 K
D1           0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        600.1342009 MHz
NUC1                 1H
P1                 8.00 usec

F2 - Processing parameters
SI                65536
SF          600.1300359 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.80 cm
CY                50.00 cm
F1P              10.000 ppm
F1              6001.30 Hz
F2P               0.000 ppm
F2                 0.00 Hz
PPMCM           0.43860 ppm/cm
HZCM          263.21494 Hz/cm
```

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



```
Current Data Parameters
USER              michal
NAME          SEM-1-286
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150820
Time               19.26
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   151
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                 33.10 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                 16.55 usec
P11               500.00 usec
P12              2000.00 usec
PL0               120.00 dB
PL1                -1.00 dB
SFO1         125.7942548 MHz
SP1                 2.70 dB
SP2                 2.70 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               24.50 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           125.7804282 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
```

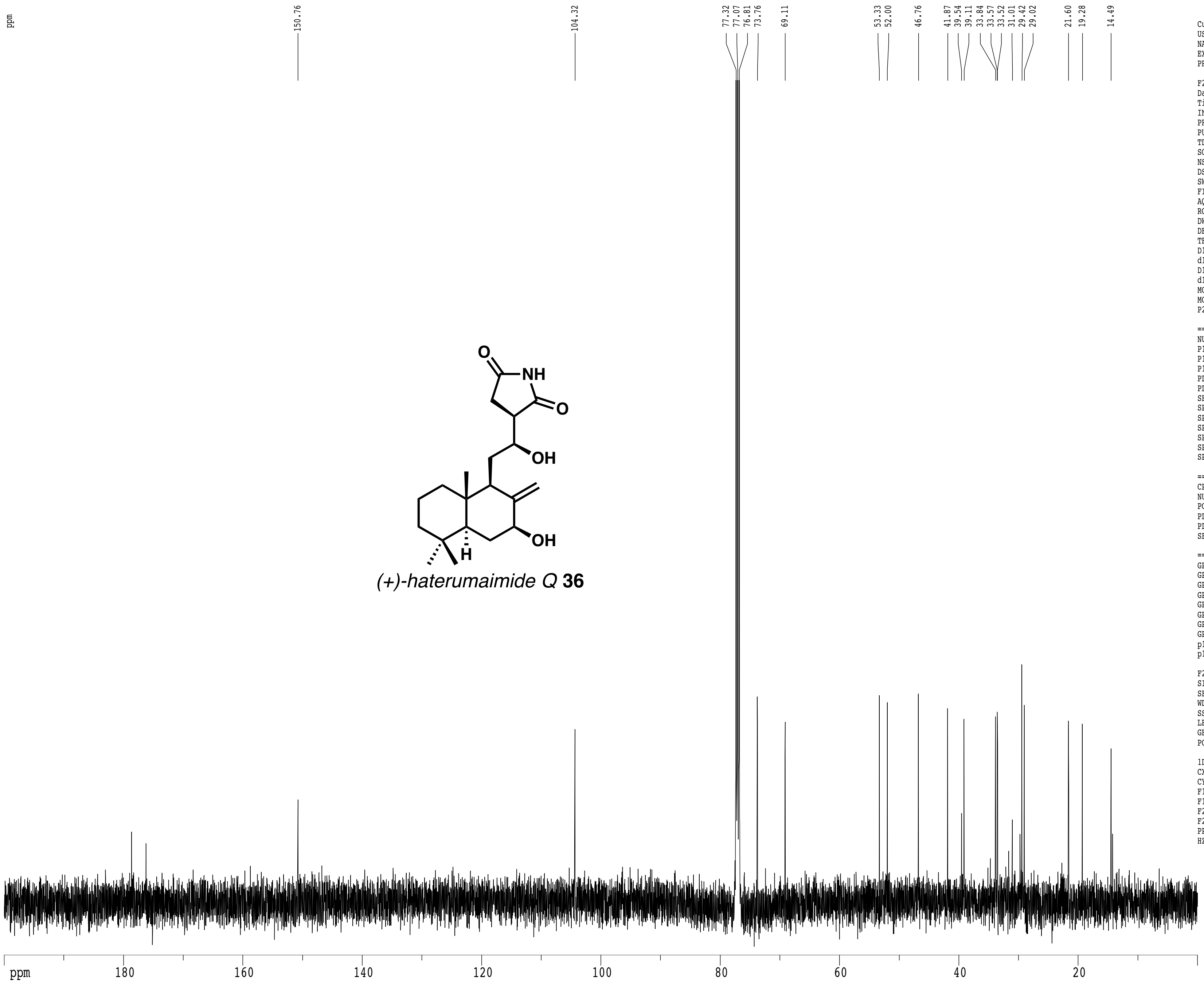
1H spectrum



(+)-haterumaimide Q **36**

```
Current Data Parameters
USER              zkonst
NAME           ZK-1-264
EXPNO                 8
PROCNO                1

F2 - Acquisition Parameters
Date_          20140127
Time              15.24
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG            zg30
TD                32048
SOLVENT           CDCl3
NS                   32
DS                    2
SWH          8012.820 Hz
FIDRES       0.250026 Hz
AQ          1.9998451 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         500.2200313 MHz
WDW                  EM
SSB                   0
LB               0.30 Hz
GB                    0
PC                 4.00

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

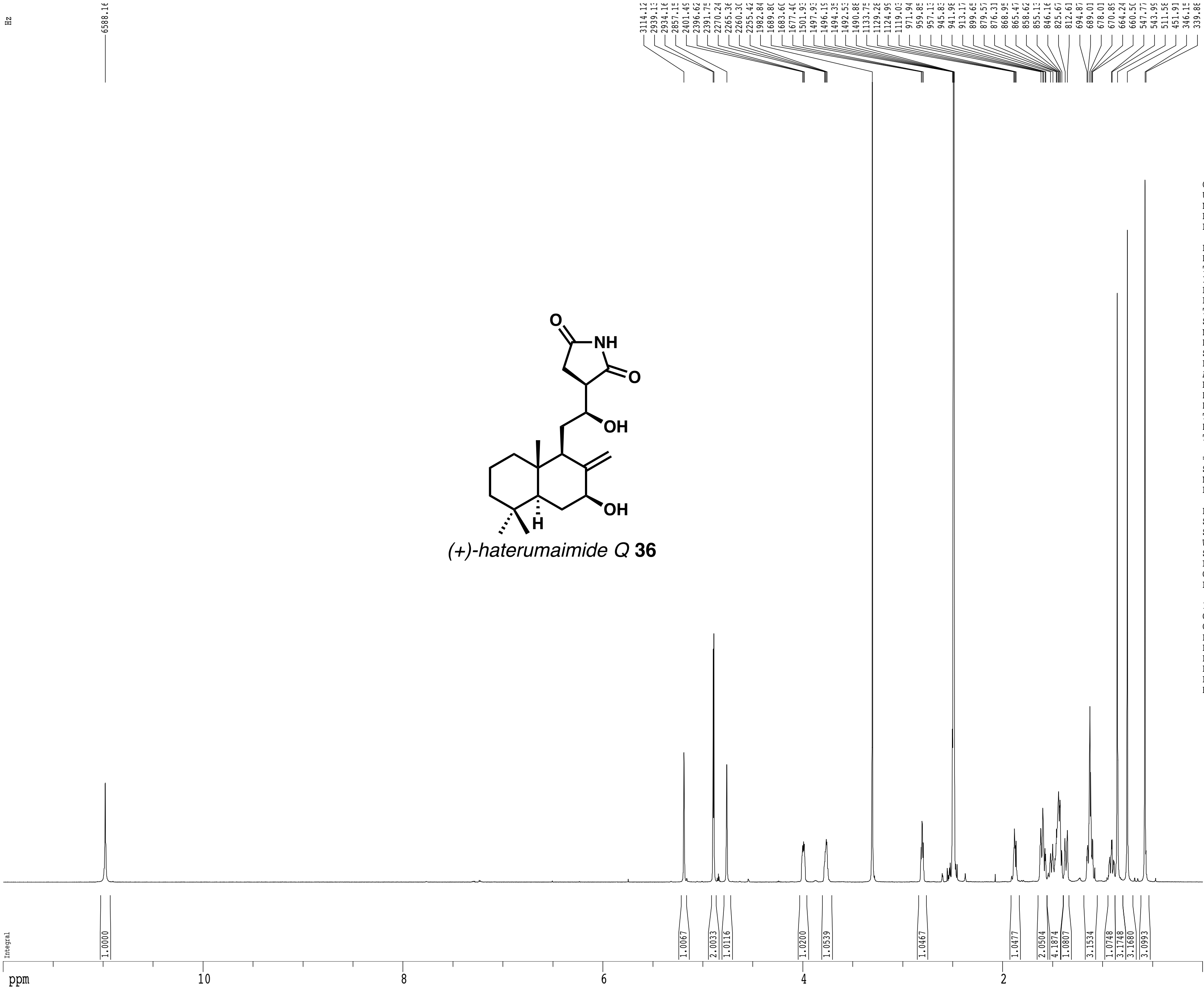
Z-restored spin-echo 13C spectrum with 1H decoupling



(+)-haterumaimide Q **36**

```
Current Data Parameters
USER                zkonst
NAME              ZK-1-229
EXPNO                  100
PROCNO                   1

F2 - Acquisition Parameters
Date_             20131121
Time                  1.26
INSTRUM            cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                   65536
SOLVENT              CDCl3
NS                    5726
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             125.7804190 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                   22.80 cm
CY                  150.00 cm
F1P                200.000 ppm
F1                25156.08 Hz
F2P                  0.000 ppm
F2                    0.00 Hz
PPMCM              8.77193 ppm/cm
HZCM            1103.33704 Hz/cm
```

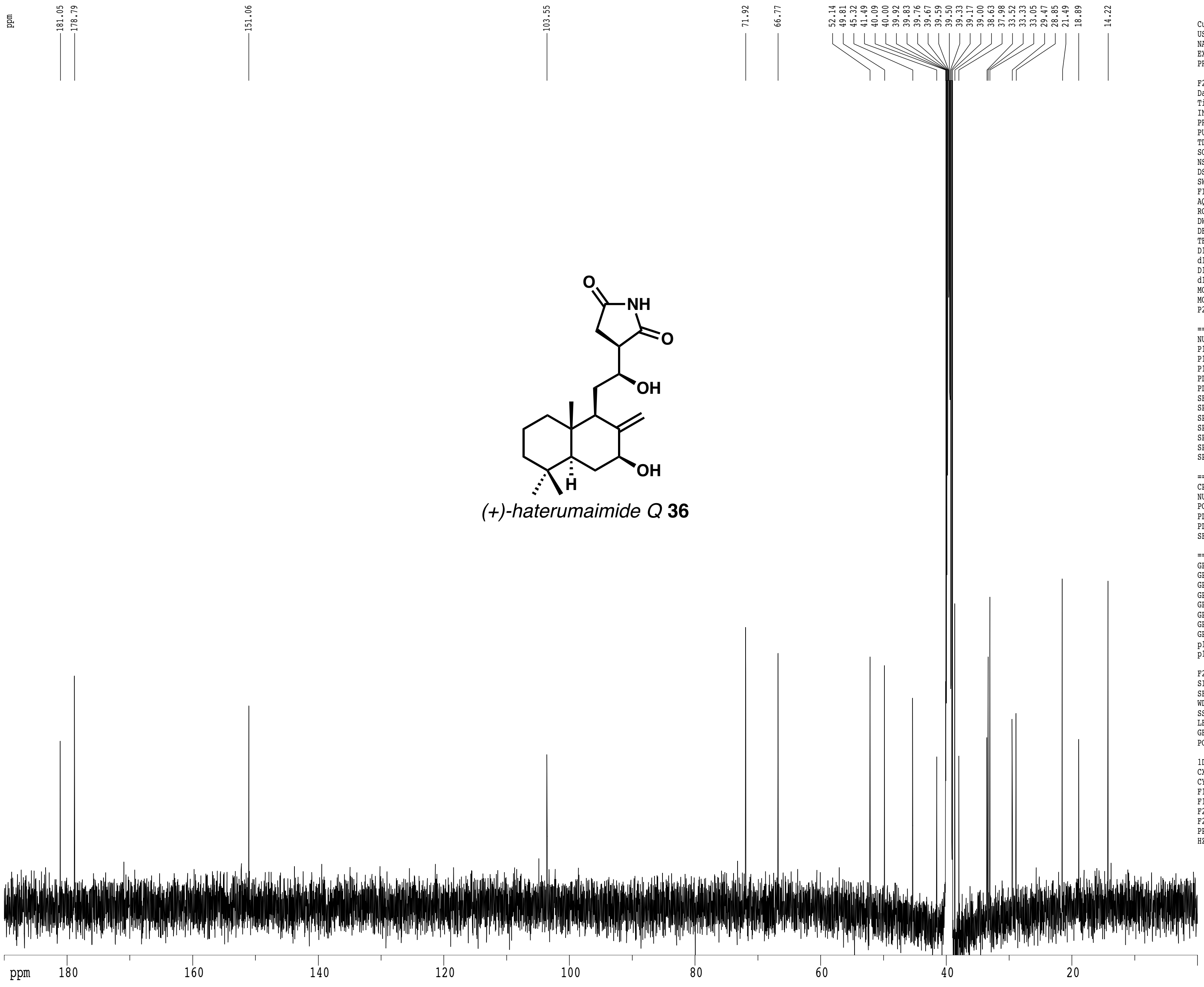
1H spectrum



(+)-haterumaimide Q **36**

```
Current Data Parameters
USER              zkonst
NAME               3-143
EXPNO                 16
PROCNO                 1

F2 - Acquisition Parameters
Date_          20150905
Time              11.34
INSTRUM           av600
PROBHD   5 mm TBI 1H/13
PULPROG            zg30
TD                76922
SOLVENT            DMSO
NS                   64
DS                    2
SWH            9615.385 Hz
FIDRES         0.125002 Hz
AQ            3.9999940 sec
RG                  362
DW               52.000 usec
DE                14.54 usec
TE                298.0 K
D1           0.10000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        600.1342009 MHz
NUC1                 1H
P1                 8.00 usec

F2 - Processing parameters
SI                65536
SF          600.1300199 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                22.80 cm
CY                40.00 cm
F1P              12.000 ppm
F1              7201.56 Hz
F2P              -0.000 ppm
F2                -0.00 Hz
PPMCM           0.52632 ppm/cm
HZCM          315.85791 Hz/cm
```

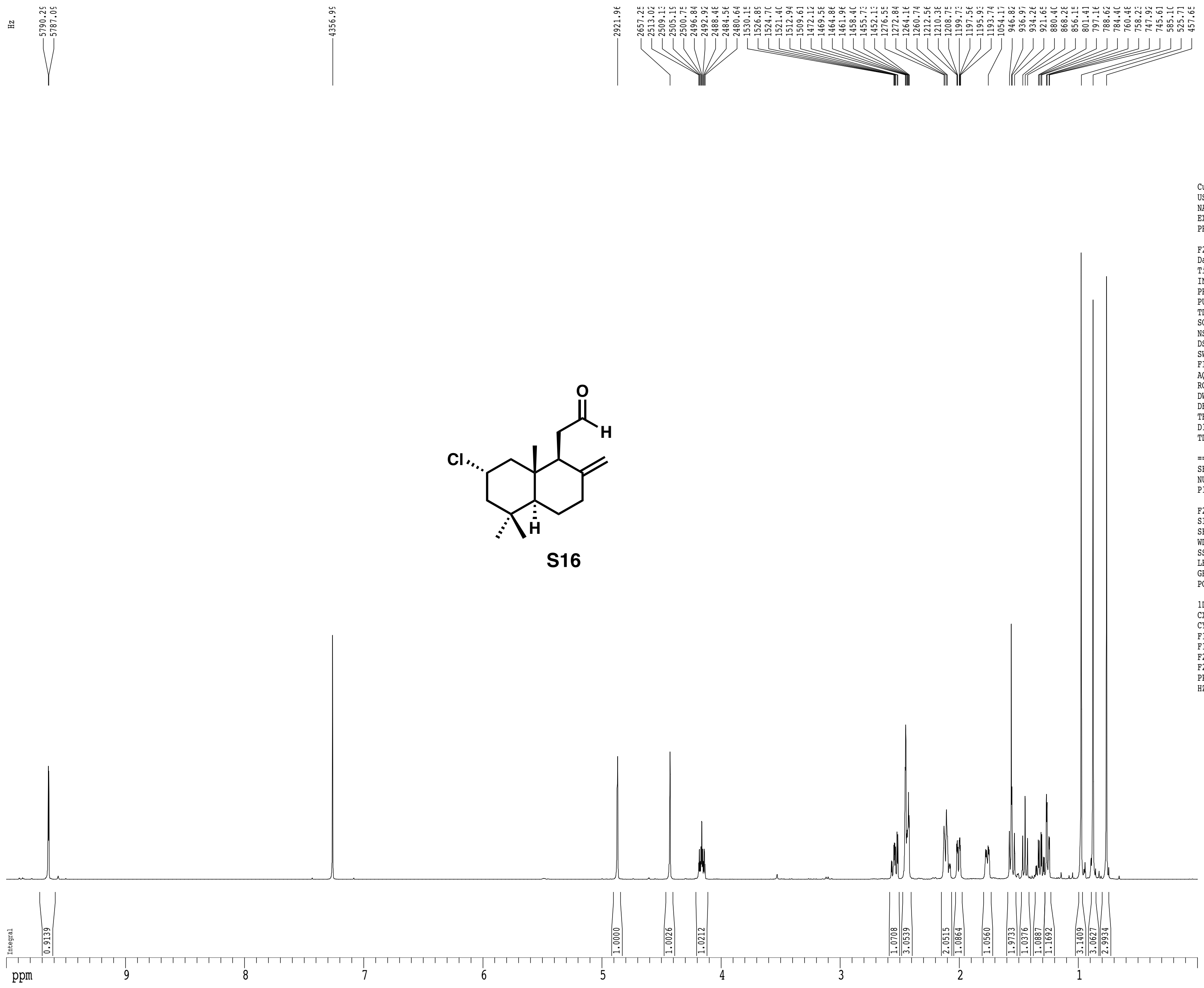
Z-restored spin-echo 13C spectrum with 1H decoupling



(+)-haterumaimide Q **36**

```
Current Data Parameters
USER                zkonst
NAME                 3-143
EXPNO                   13
PROCNO                   1

F2 - Acquisition Parameters
Date_             20150903
Time                 16.26
INSTRUM            cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                   65536
SOLVENT               DMSO
NS                     680
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              1.00000000 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             125.7804817 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    2.00

1D NMR plot parameters
CX                   22.80 cm
CY                  300.00 cm
F1P                190.000 ppm
F1                23898.29 Hz
F2P                  0.000 ppm
F2                    0.00 Hz
PPMCM              8.33333 ppm/cm
HZCM            1048.17065 Hz/cm
```

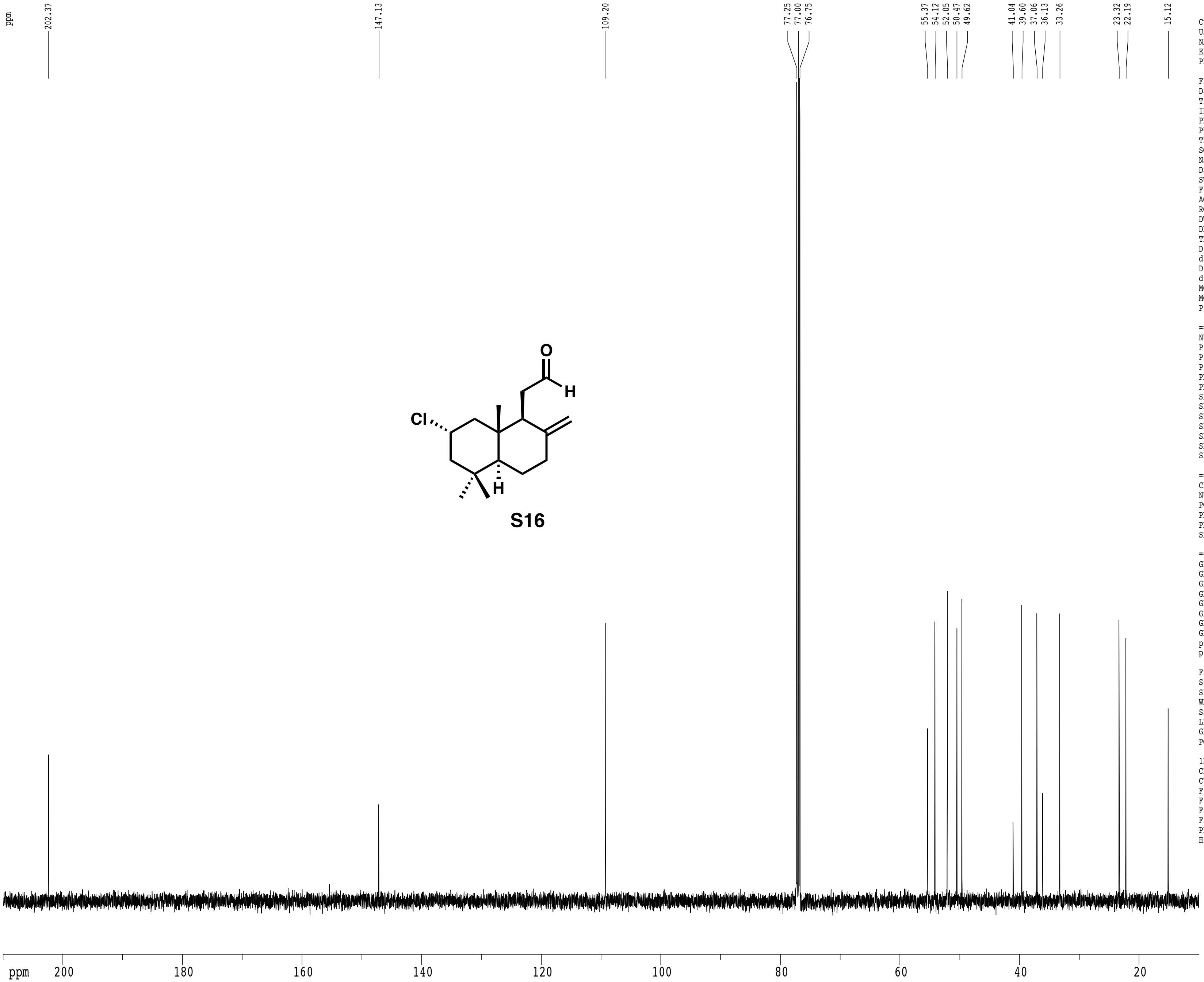
1H spectrum



S16

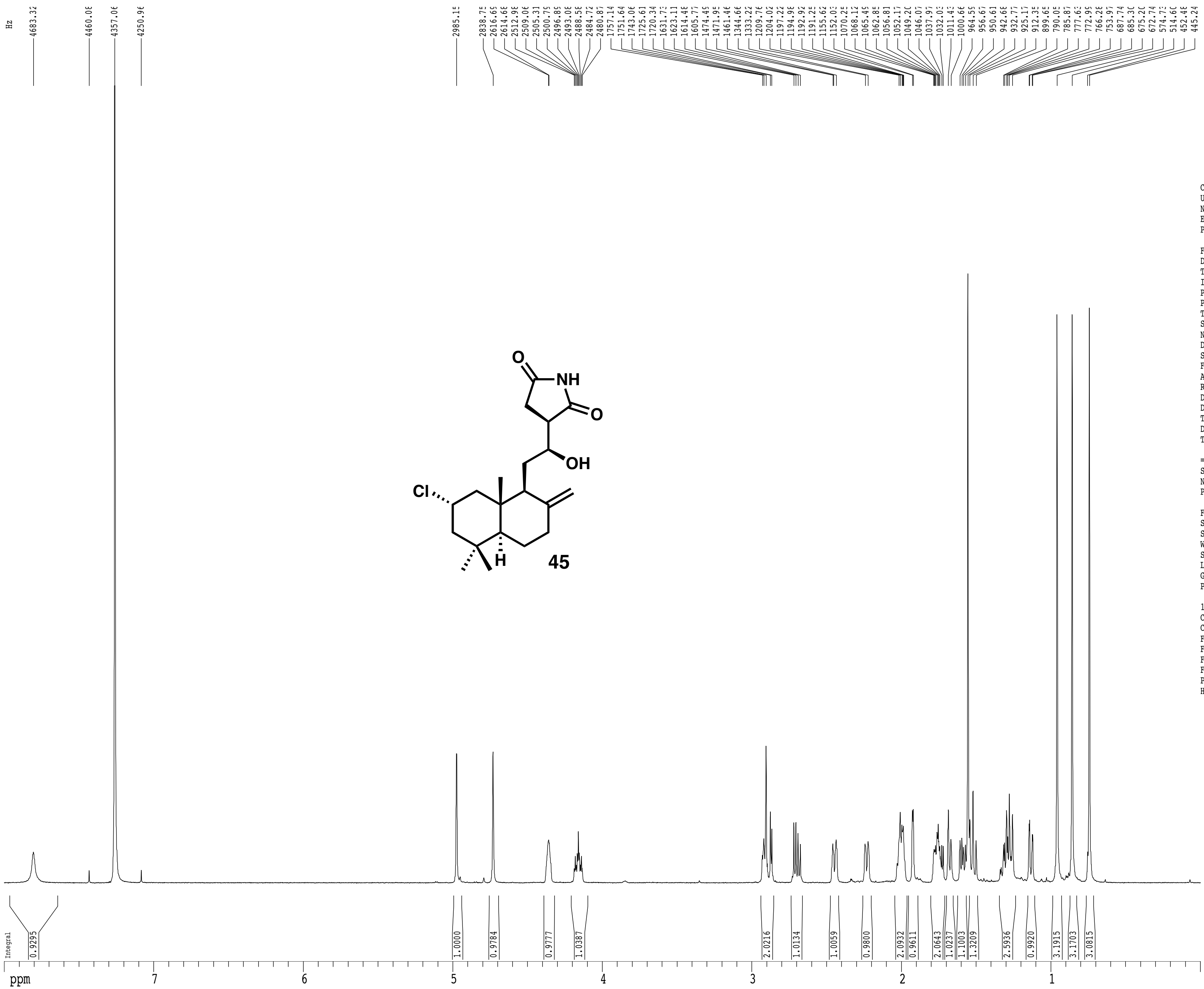
```
Current Data Parameters
USER              zkonst
NAME                2-49
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140304
Time               19.07
INSTRUM            av600
PROBHD   5 mm TBI 1H/13
PULPROG             zg30
TD                 57690
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             9615.385 Hz
FIDRES          0.166673 Hz
AQ             2.9999299 sec
RG                   256
DW                52.000 usec
DE                 14.54 usec
TE                 294.2 K
D1            0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        600.1342009 MHz
NUC1                  1H
P1                  8.00 usec

F2 - Processing parameters
SI                 65536
SF           600.1300365 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 12.00 cm
F1P               10.000 ppm
F1               6001.30 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.43860 ppm/cm
HZCM           263.21494 Hz/cm
```

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



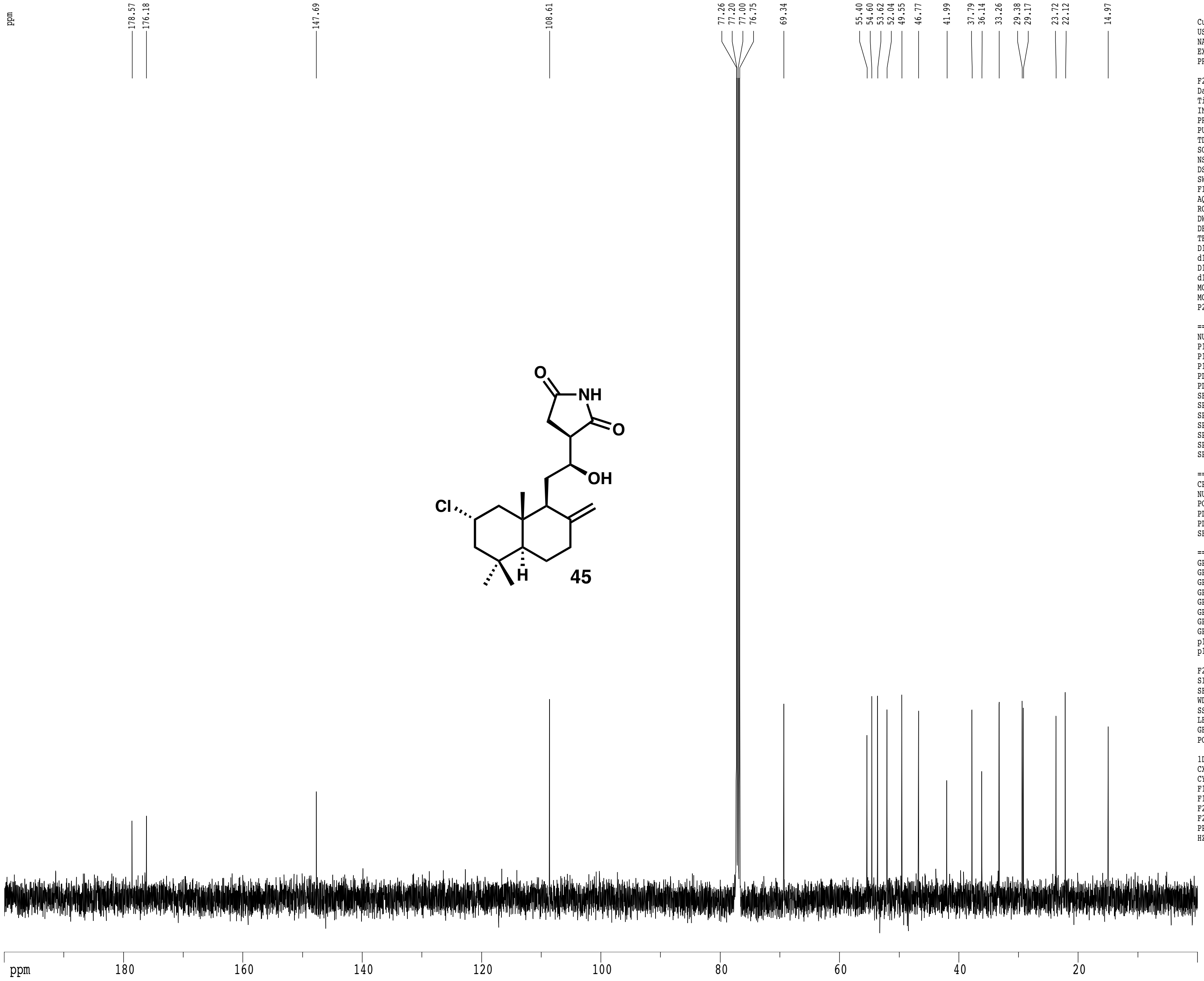
```
Current Data Parameters
USER              zkonst
NAME                2-49
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140304
Time               19.17
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                   600
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           125.7804277 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 16.00 cm
F1P              210.000 ppm
F1              26413.89 Hz
F2P               10.000 ppm
F2               1257.81 Hz
PPMCM            8.77193 ppm/cm
HZCM          1103.33716 Hz/cm
```

1H spectrum



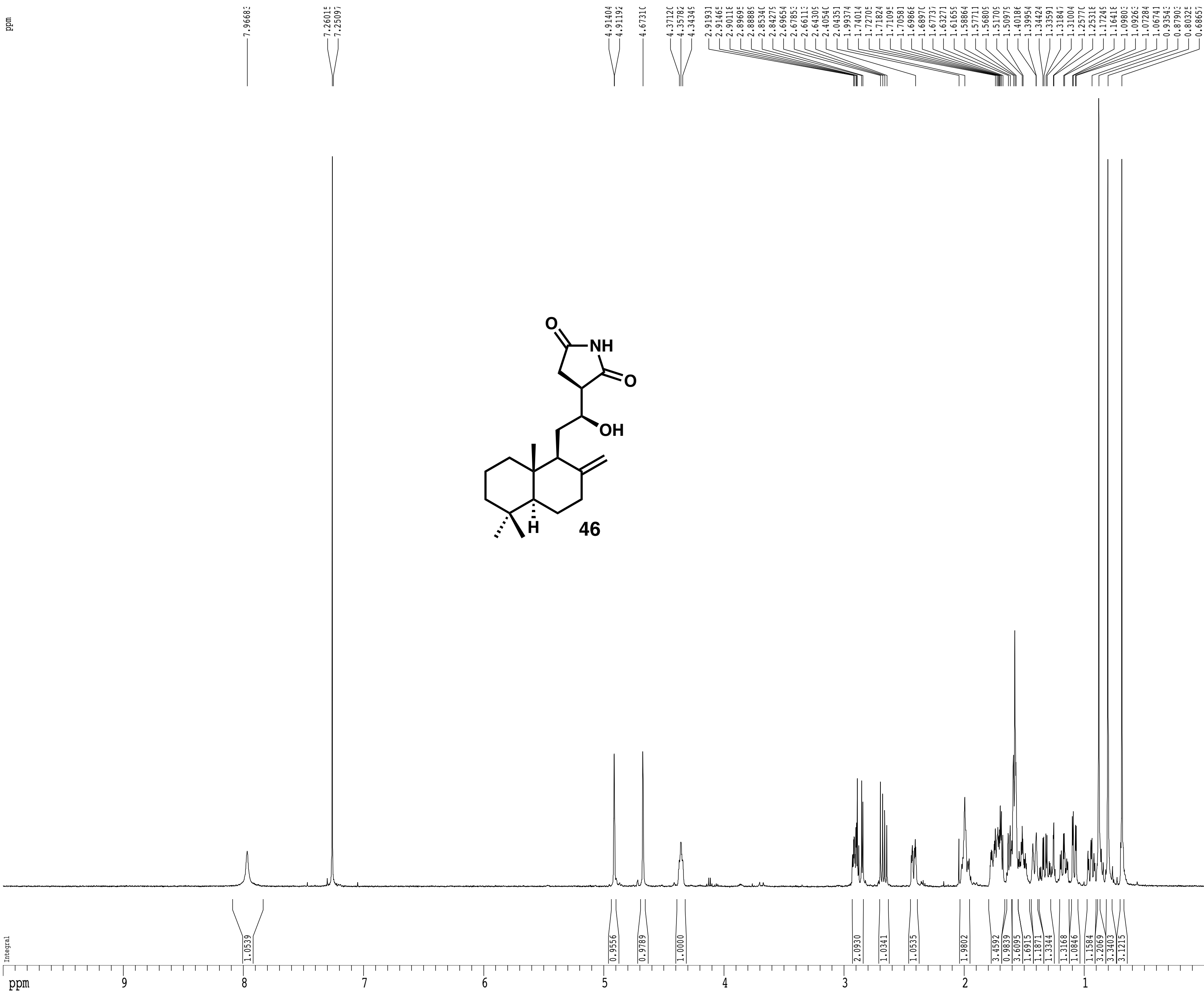
```
Current Data Parameters
USER              zkonst
NAME                2-63
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140318
Time               17.56
INSTRUM            av600
PROBHD   5 mm TBI 1H/13
PULPROG             zg30
TD                 57690
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            9615.385 Hz
FIDRES         0.166673 Hz
AQ            2.9999299 sec
RG                   512
DW               52.000 usec
DE                14.54 usec
TE                298.0 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        600.1342009 MHz
NUC1                  1H
P1                  8.00 usec

F2 - Processing parameters
SI                 65536
SF          600.1300337 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 45.00 cm
F1P                8.000 ppm
F1               4801.04 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.35088 ppm/cm
HZCM           210.57195 Hz/cm
```

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



```
Current Data Parameters
USER              zkonst
NAME                2-63
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140318
Time               18.09
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                    16
SWH            30303.031 Hz
FIDRES          0.462388 Hz
AQ             1.0814105 sec
RG                7298.2
DW                16.500 usec
DE                  6.00 usec
TE                 298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
D16           0.00020000 sec
d17           0.00019600 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
P2                 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           125.7804268 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  2.00

1D NMR plot parameters
CX                 22.80 cm
CY                 60.00 cm
F1P              200.000 ppm
F1              25156.09 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            8.77193 ppm/cm
HZCM          1103.33716 Hz/cm
```

1H spectrum



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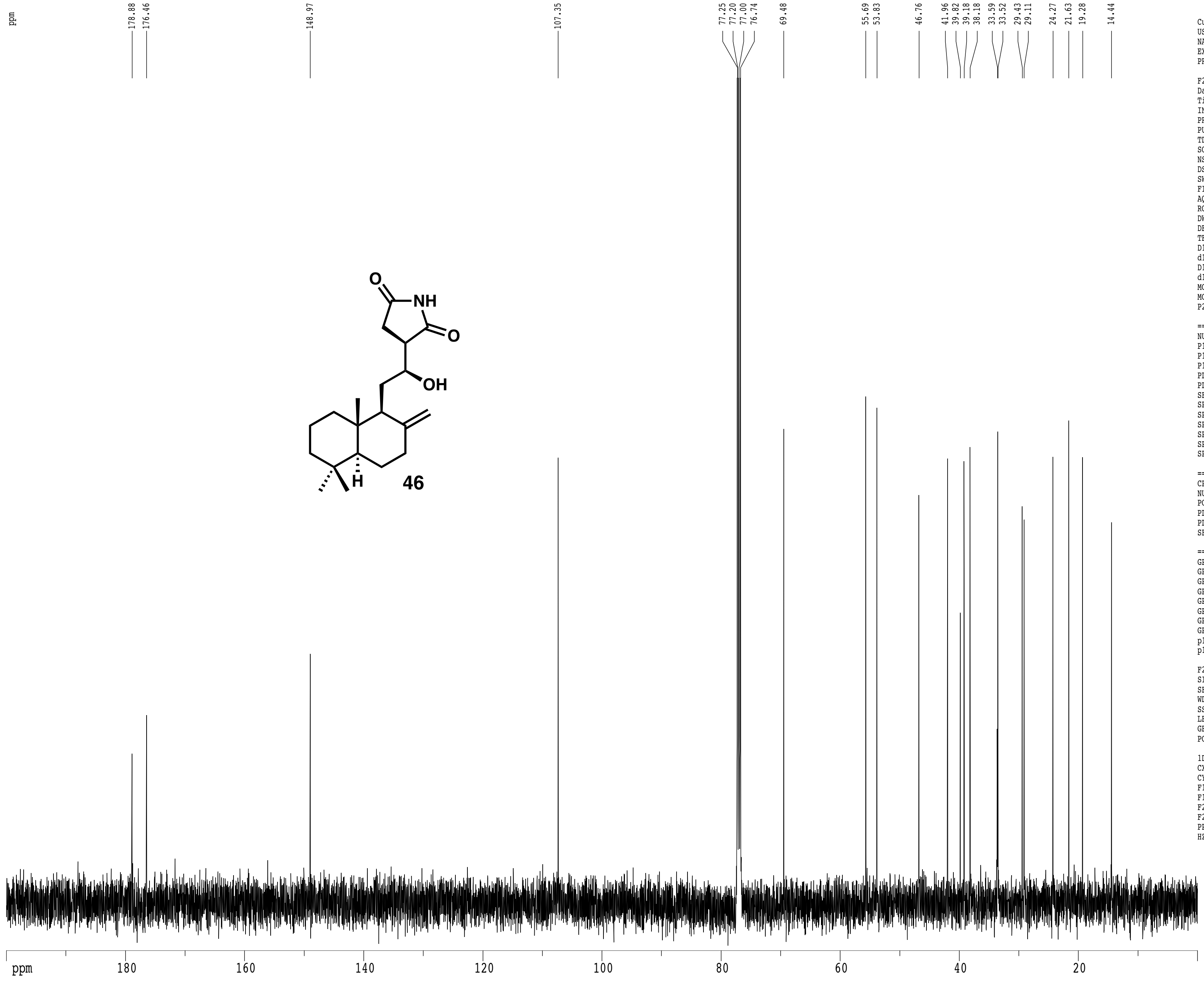
```
Current Data Parameters
USER              zkonst
NAME            ZK-1-216
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20131120
Time               16.27
INSTRUM            gn500
PROBHD   5 mm broadband
PULPROG             zg30
TD                 32048
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES          0.250026 Hz
AQ             1.9998451 sec
RG                1149.4
DW                62.400 usec
DE                  6.00 usec
TE                 298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.20 usec
PL1                -5.00 dB
SFO1         499.2934950 MHz

F2 - Processing parameters
SI                 65536
SF           499.2900292 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P               10.000 ppm
F1               4992.90 Hz
F2P                0.000 ppm
F2                  0.00 Hz
PPMCM            0.43860 ppm/cm
HZCM           218.98686 Hz/cm
```

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



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```
Current Data Parameters
USER                zkonst
NAME                 3-145
EXPNO                   12
PROCNO                   1

F2 - Acquisition Parameters
Date_            20150904
Time                18.07
INSTRUM           cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                  65536
SOLVENT             CDCl3
NS                    760
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             1.00000000 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            125.7804277 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   2.00

1D NMR plot parameters
CX                  22.80 cm
CY                 100.00 cm
F1P               200.000 ppm
F1               25156.09 Hz
F2P                 0.000 ppm
F2                   0.00 Hz
PPMCM             8.77193 ppm/cm
HZCM           1103.33716 Hz/cm
```