Supporting Information for

A Convergent Assembly of the Spiroacetal Subunit of Didemnaketal B

Haruhiko Fuwa*, Sayaka Noji, and Makoto Sasaki

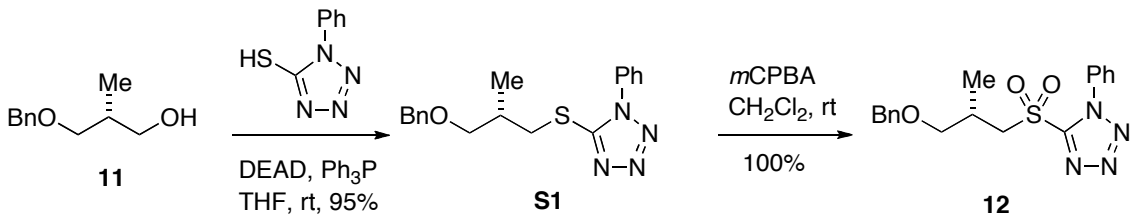
Graduate School of Life Sciences, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan

*Corresponding author. Email: hfuwa@bios.tohoku.ac.jp

Contents

General methods. All reactions sensitive to moisture and/or air were carried out under an atmosphere of argon in dry, freshly distilled solvents under anhydrous conditions using oven-dried glassware unless otherwise noted. Anhydrous dichloromethane (CH_2Cl_2) was purchased from Kanto Chemical Co. Inc. and used directly without further drying. Anhydrous tetrahydrofuran, diethyl ether, and toluene were purchased from Wako Pure Chemical Industries, Ltd. and further purified by a Glass Contour solvent purification system under an atmosphere of argon immediately prior to use. Diisopropylethylamine, triethylamine, 2,6-lutidine, acetonitrile (CH_3CN), benzene, and methanol were distilled from calcium hydride under an atmosphere of argon. Hexamethyphosphoramide (HMPA) and *N,N*,-dimethylpropyleneurea (DMPU) were distilled from calcium hydride under reduced pressure. *N,N*-dimethylformamide (DMF) and dimethyl sulfoxide (DMSO) were distilled from magnesium sulfate under reduced pressure. All other chemicals were purchased at highest commercial grade and used directly. Analytical thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F_{254} plates (0.25-mm thickness). Flash column chromatography was carried out using Kanto chemical silica gel 60N (40-100 mesh, spherical, neutral) or Fuji Silysia silica gel BW-300 (200-400 mesh). Optical rotations were recorded on a JASCO P-1020 digital polarimeter. IR spectra were recorded on a JASCO FT/IR-4100 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a JEOL JNM-ECA-600 spectrometer, and chemical shift values are reported in ppm (δ) downfield from tetramethylsilane with reference to internal solvent [^{1}H NMR, C*H*Cl_3 (7.24), C_6*H*D_5 (7.15); ^{13}C NMR, *C*DCl_3 (77.0), C_6D_6 (128.0)] unless otherwise noted. Coupling constants (*J*) are reported in Hertz (Hz). The following abbreviations were used to designate the multiplicities: s = singlet; d = doublet; t = triplet; m = multiplet; br =

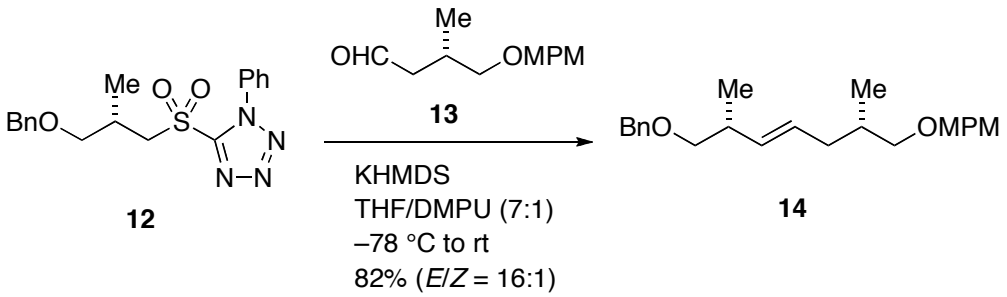
broad. FAB mass spectra were recorded on a JEOL JMS-700 spectrometer and ESI-TOF mass spectra were measured on a Bruker microTOFfocus spectrometer.



Sulfone 12. To a solution of alcohol **11** (0.8965 g, 4.977 mmol) in THF (40 mL) cooled to 0 °C were added 1-phenyl-1*H*-tetrazole-5-thiol (1.33 g, 7.46 mmol), Ph_3P (1.96 g, 7.47 mmol), and DEAD (2.2 M solution in toluene, 3.40 mL, 7.48 mmol), and the resultant solution was stirred at room temperature for 25 min. The reaction was quenched with saturated aqueous $NaHCO_3$ solution at 0 °C, and the resultant mixture was diluted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (8 to 15% EtOAc/hexanes) gave sulfide **S1** (1.6017 g, 95%) as a colorless oil: $[\alpha]_D^{26}$ −8.0 (*c* 1.00, $CHCl_3$); IR (film) 2930, 1595, 1498, 1454, 1385, 1014, 962, 759, 695, 443 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.57—7.49 (m, 5H), 7.33—7.23 (m, 5H), 4.49 (d, *J* = 12.0 Hz, 1H), 4.47 (d, *J* = 12.0 Hz, 1H), 3.54 (dd, *J* = 12.7, 6.2 Hz, 1H), 3.47 (dd, *J* = 9.6, 4.9 Hz, 1H), 3.40 (dd, *J* = 9.6, 5.8 Hz, 1H), 3.38 (dd, *J* = 12.7, 6.8 Hz, 1H), 2.29 (m, 1H), 1.08 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 154.7, 138.2, 133.7, 130.0, 129.7 (2C), 128.3 (2C), 127.6 (3C), 123.8 (2C), 73.5, 73.1, 37.1, 33.6, 16.5; HRMS (ESI) calcd for $C_{18}H_{20}N_4OSNa$ $[(M + Na)^+]$ 363.1250, found 363.1259.

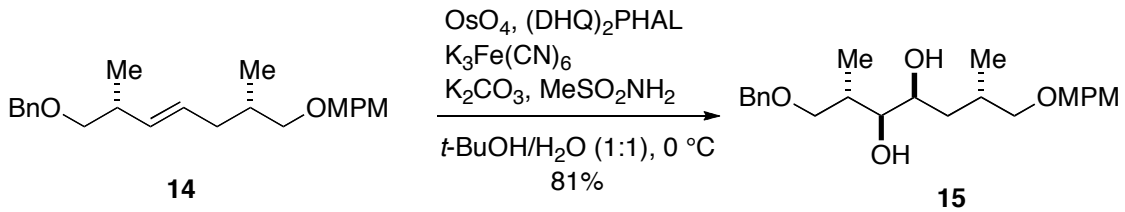
To a solution of sulfide **S1** (0.6626 g, 1.948 mmol) in CH_2Cl_2 (10 mL) cooled

to 0 °C was added *m*-CPBA (77% purity, 1.07 g, 4.77 mmol), and the resultant solution was stirred at room temperature for 22 h. The reaction was quenched with 3.0 M aqueous NaOH solution at 0 °C, and the resultant mixture was stirred at room temperature for 10 min. The resultant mixture was diluted with EtOAc, and the organic layer was washed successively with 3.0 M aqueous NaOH solution, saturated aqueous NH_4Cl solution, and brine. The organic layer was dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (10 to 15% EtOAc/hexanes) gave sulfone **12** (0.7321 g, 100%) as a colorless viscous oil: $[\alpha]_D^{28}$ −14.0 (*c* 1.00, C_6H_6); IR (film) 2861, 1496, 1455, 1336, 1152, 1096, 764, 689, 634, 520 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.63—7.53 (m, 5H), 7.36—7.25 (m, 5H), 4.48 (d, *J* = 11.6 Hz, 1H), 4.43 (d, *J* = 11.6 Hz, 1H), 4.03 (dd, *J* = 14.8, 5.2 Hz, 1H), 3.56 (dd, *J* = 14.8, 7.5 Hz, 1H), 3.52 (dd, *J* = 9.3, 4.8 Hz, 1H), 3.36 (dd, *J* = 9.3, 6.5 Hz, 1H), 2.60 (m, 1H), 1.18 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 154.0, 137.8, 133.0, 131.4, 129.6 (2C), 128.4 (2C), 127.8, 127.6 (2C), 125.2 (2C), 73.1, 73.0, 59.0, 29.3, 17.1; HRMS (ESI) calcd for $C_{18}H_{20}N_4O_3SNa$ [$(M + Na)^+$] 395.1148, found 395.1151.



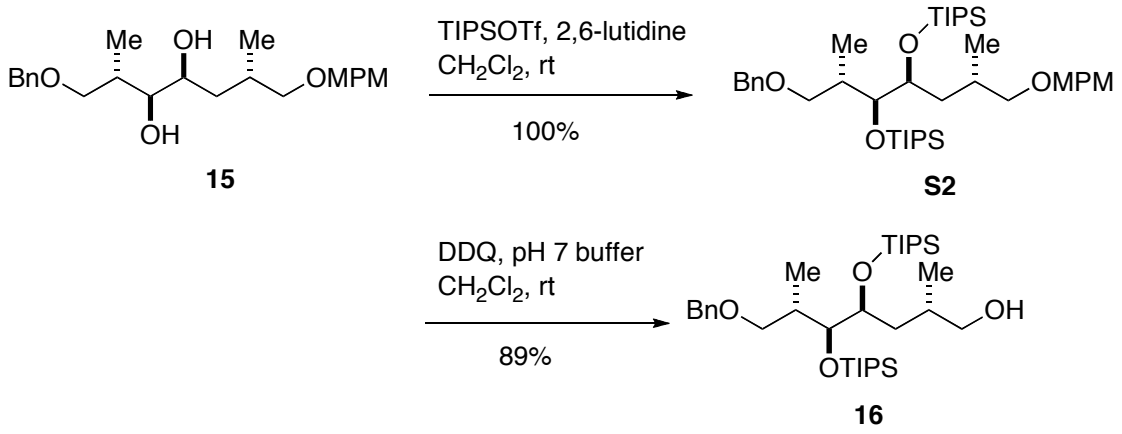
Olefin 14. To a solution of sulfone **12** (2.565 g, 6.893 mmol) in THF/DMPU (4:1, v/v, 40 mL) cooled to −78 °C was added dropwise KHMDS (0.5 M solution in toluene, 12 mL, 6.0 mmol), and the resultant mixture was stirred at −78 °C for 40 min. To this

solution was added dropwise a solution of aldehyde **13** (0.7355 g, 3.311 mmol) in THF (17 mL + 10 mL rinse), and the resultant mixture was allowed to warm to room temperature over a period of 11 h 20 min. The reaction was quenched with saturated aqueous NH_4Cl solution at 0 °C, and the mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (3 to 15% EtOAc/hexanes) gave olefin **14** (0.9981 g, 82%, *E*/*Z* = 16:1 by 600 MHz ^{1}H NMR analysis) as a yellow oil: $[\alpha]_D^{24}$ +2.1 (*c* 1.80, C_6H_6); IR (film) 2852, 1612, 1513, 1454, 1361, 1248, 1093, 1037, 971, 698 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35—7.30 (m, 4H), 7.29—7.22 (m, 3H), 6.89—6.85 (m, 2H), 5.42 (ddd, *J* = 15.4, 6.5, 6.5 Hz, 1H), 5.36 (dd, *J* = 15.4, 6.9 Hz, 1H), 4.51 (d, *J* = 12.0 Hz, 1H), 4.49 (d, *J* = 12.0 Hz, 1H), 4.41 (s, 2H), 3.79 (s, 3H), 3.34 (dd, *J* = 8.9, 6.5 Hz, 1H), 3.29 (dd, *J* = 8.9, 6.2 Hz, 1H), 3.26 (dd, *J* = 9.3, 6.9 Hz, 1H), 3.21 (dd, *J* = 9.3, 6.5 Hz, 1H), 2.46 (m, 1H), 2.14 (ddd, *J* = 12.7, 6.5, 6.5 Hz, 1H), 1.89—1.74 (m, 2H), 1.01 (d, *J* = 6.5 Hz, 3H), 0.89 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.0, 138.7, 134.4, 130.8, 129.1 (2C), 128.3 (2C), 127.9, 127.5 (2C), 127.4, 113.7 (2C), 75.5, 75.0, 72.8, 72.6, 55.2, 36.9, 36.7, 33.6, 17.3, 16.8; HRMS (ESI) calcd for $C_{24}H_{32}O_3Na$ [$(M + Na)^+$] 391.2244, found 391.2255.



1,2-Diol 15. To a solution of olefin **14** (1.437 g, 3.902 mmol, *E*/*Z* = 16:1) in *t*-BuOH/H_2O (1:1, v/v, 34 mL) were added $(DHQ)_2PHAL$ (60.8 mg, 78.1 μmol),

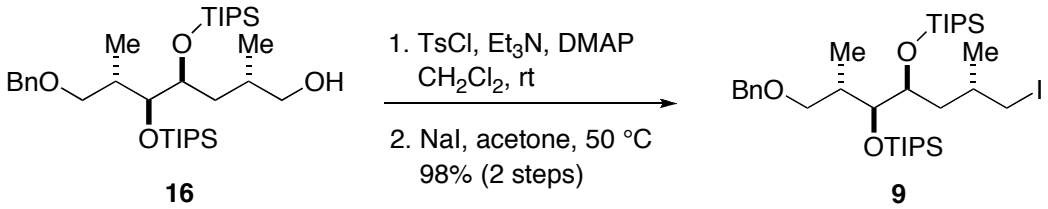
$K_3Fe(CN)_6$ (3.85 g, 11.7 mmol), and K_2CO_3 (1.62 g, 11.7 mmol). The resultant mixture was cooled to 0 °C, treated with OsO_4 (0.039 M solution in *t*-BuOH, 1.0 mL, 39 μmol) and $MeSO_2NH_2$ (371.0 mg, 3.900 mmol), and stirred vigorously at 0 °C for 15 h 20 min. The reaction was quenched with solid Na_2SO_3, and the mixture was stirred at room temperature for 10 min. The resultant mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel (10 to 50% EtOAc/hexanes) to give 1,2-diol **15** (1.442 g) along with unreacted olefin **14** (0.0837 g, 6%, *E*/*Z* = 5:2). The former material was further purified by recrystallization from CH_2Cl_2/hexanes to give 1,2-diol **15** (1.265 g, 81%, dr >20:1 by 600 MHz ^{1}H NMR analysis) as a colorless solid: mp 89.0—89.5 °C; $[\alpha]_D^{23}$ +4.5 (*c* 1.00, $CHCl_3$); IR (film) 3341, 2936, 1514, 1248, 1100, 1032, 816, 698 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.36—7.26 (m, 5H), 7.25—7.20 (m, 2H), 6.89—6.81 (m, 2H), 4.50 (s, 2H), 4.43 (d, *J* = 11.7 Hz, 1H), 4.41 (d, *J* = 11.7 Hz, 1H), 3.78 (s, 3H), 3.67 (ddd, *J* = 10.0, 3.0, 3.0 Hz, 1H), 3.54—3.50 (m, 2H), 3.31—3.24 (m, 3H), 2.04 (m, 1H), 1.98 (m, 1H), 1.69 (ddd, *J* = 14.1, 10.0, 5.9 Hz, 1H), 1.29 (ddd, *J* = 14.1, 7.6, 3.0 Hz, 1H), 0.92 (d, *J* = 7.3 Hz, 3H), 0.91 (d, *J* = 6.8 Hz, 3H) (two protons missing presumably due to H/D exchange); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.1, 137.6, 130.4, 129.2 (2C), 128.5 (2C), 127.8, 127.7 (2C), 113.7 (2C), 78.7, 76.2, 74.1, 73.4, 72.7, 69.9, 55.2, 39.5, 35.8, 30.9, 17.5, 14.6; HRMS (ESI) calcd for $C_{24}H_{34}O_5Na$ [$(M + Na)^+$] 425.2298, found 425.2305.



Alcohol 16. To a solution of 1,2-diol **15** (92.6 mg, 0.230 mmol) in CH_2Cl_2 (2.3 mL) cooled to 0 °C were added 2,6-lutidine (0.11 mL, 0.94 mmol) and TIPSOTf (0.18 mL, 0.67 mmol). The resultant solution was stirred at 0 °C for 2 h and then at room temperature for 1 h. The reaction was quenched with saturated aqueous NH_4Cl solution at 0 °C, and the resultant mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (3 to 5% EtOAc/hexanes) gave silyl ether **S2** (166.6 mg, 100%) as a colorless oil: $[\alpha]_D^{25}$ −28.3 (*c* 1.00, $CHCl_3$); IR (film) 2944, 2866, 1513, 1463, 1248, 1097, 997, 882, 678 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.32—7.26 (m, 4H), 7.26—7.19 (m, 3H), 6.89—6.80 (m, 2H), 4.47 (d, *J* = 12.0 Hz, 1H), 4.43 (d, *J* = 12.0 Hz, 1H), 4.40 (s, 2H), 3.95 (ddd, *J* = 9.3, 3.8, 2.7 Hz, 1H), 3.86 (dd, *J* = 4.1, 3.8 Hz, 1H), 3.78 (s, 3H), 3.64 (dd, *J* = 9.3, 3.1 Hz, 1H), 3.31 (dd, *J* = 9.3, 7.6 Hz, 1H), 3.30 (dd, *J* = 8.9, 3.4 Hz, 1H), 3.14 (dd, *J* = 8.9, 7.6 Hz, 1H), 2.20 (m, 1H), 1.97 (m, 1H), 1.46 (ddd, *J* = 12.4, 9.3, 3.8 Hz, 1H), 1.41 (ddd, *J* = 12.4, 9.6, 2.7 Hz, 1H), 1.14 (d, *J* = 6.8 Hz, 3H), 1.06—0.98 (m, 42H), 0.92 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.0, 138.9, 131.0, 128.9 (2C), 128.2 (2C), 127.6 (2C), 127.3, 113.7 (2C), 78.0, 76.4, 74.3, 73.5, 72.9, 72.4, 55.2, 35.8, 35.0,

29.8, 18.4 (7C), 18.33 (3C), 18.31 (3C), 17.4, 13.1 (6C); HRMS (ESI) calcd for $C_{42}H_{74}O_5Si_2Na$ [(M + Na)$^+$] 737.4967, found 737.4963.

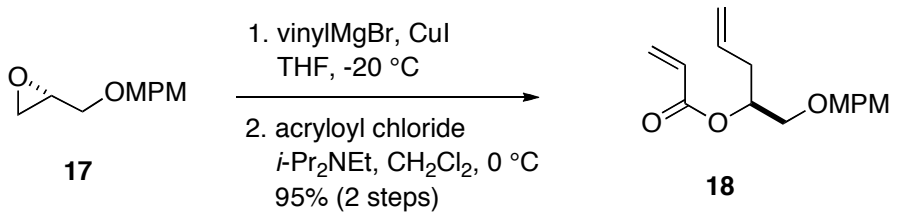
To a solution of silyl ether **S2** (27.9 mg, 39.0 μmol) in CH_2Cl_2/pH 7 buffer (10:1, v/v, 0.77 mL) cooled to 0 °C was added DDQ (9.8 mg, 43 μmol). The resultant mixture was stirred at room temperature for 2 h 40 min, after which point an additional portion of DDQ (0.90 mg, 3.96 μmol) was added to the reaction mixture. After being stirred at room temperature for further 45 min, the reaction was quenched with saturated aqueous $NaHCO_3$ solution at 0 °C. The resultant mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was passed through a pad of silica gel (5 to 8% EtOAc/hexanes) and then purified by preparative HPLC to give alcohol **16** (20.6 mg, 89%) as a colorless oil: $[\alpha]_D^{25}$ −35.7 (*c* 1.00, $CHCl_3$); IR (film) 3375, 2944, 2867, 1463, 1110, 882, 733, 677, 416 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33—7.29 (m, 4H), 7.25 (m, 1H), 4.48 (d, *J* = 12.0 Hz, 1H), 4.46 (d, *J* = 12.0 Hz, 1H), 3.95 (ddd, *J* = 9.2, 3.8, 2.8 Hz, 1H), 3.88 (dd, *J* = 4.1, 3.8 Hz, 1H), 3.65 (dd, *J* = 9.2, 3.1 Hz, 1H), 3.45 (m, 1H), 3.37 (m, 1H), 3.31 (dd, *J* = 8.9, 8.9 Hz, 1H), 2.20 (m, 1H), 1.82 (m, 1H), 1.46 (ddd, *J* = 13.7, 9.2, 3.8 Hz, 1H), 1.41 (ddd, *J* = 13.7, 9.3, 2.8 Hz, 1H), 1.29 (br s, 1H), 1.15 (d, *J* = 6.9 Hz, 3H), 1.09—0.97 (m, 42H), 0.90 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 138.9, 128.2 (2C), 127.6 (2C), 127.4, 77.9, 74.4, 73.3, 72.9, 69.2, 35.4, 35.0, 32.2, 18.33 (6C), 18.30 (6C), 17.4, 16.7, 13.1 (6C); HRMS (ESI) calcd for $C_{34}H_{66}O_4Si_2Na$ [(M + Na)$^+$] 617.4392, found 617.4393.



Iodide 9. To a solution of alcohol **16** (113.7 mg, 0.1913 mmol) in CH_2Cl_2 (2.0 mL) cooled to 0 °C were added Et_3N (53 μL, 0.38 mmol), DMAP (3.0 mg, 20 μmol) and TsCl (55.0 mg, 0.289 mmol), and the resultant solution was stirred at room temperature for 12 h. The reaction was quenched with H_2O at 0 °C, and the resultant mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residual crude tosylate was used in the next reaction without further purification.

To a solution of the above tosylate in acetone (2.5 mL) was added NaI (143 mg, 0.954 mmol), and the resultant mixture was stirred at 50 °C for 15 h. After being cooled to room temperature, the reaction mixture was diluted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (1 to 1.5% EtOAc/hexanes) gave iodide **9** (131.8 mg, 98% for the two steps) as a colorless oil: $[\alpha]_D^{25}$ −33.7 (*c* 1.00, $CHCl_3$); IR (film) 2944, 2866, 1462, 1110, 882, 732, 679 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.35—7.29 (m, 4H), 7.25 (m, 1H), 4.49 (s, 2H), 3.91 (ddd, *J* = 8.9, 3.8, 2.8 Hz, 1H), 3.87 (dd, *J* = 4.1, 3.8 Hz, 1H), 3.66 (dd, *J* = 9.3, 3.1 Hz, 1H), 3.33 (dd, *J* = 8.9, 8.9 Hz, 1H), 3.21 (dd, *J* = 9.3, 4.4 Hz, 1H), 3.12 (dd, *J* = 9.6, 6.5 Hz, 1H), 2.21 (m, 1H), 1.72 (m, 1H), 1.56 (ddd, *J* = 13.4, 8.6, 2.8 Hz, 1H), 1.51 (ddd, *J* = 13.4, 8.9, 3.8 Hz, 1H), 1.16 (d, *J* = 6.9 Hz, 3H), 1.08—0.99 (m, 42H), 0.96 (d, *J* = 6.5 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 138.8, 128.2 (2C), 127.6 (2C), 127.3, 77.9,

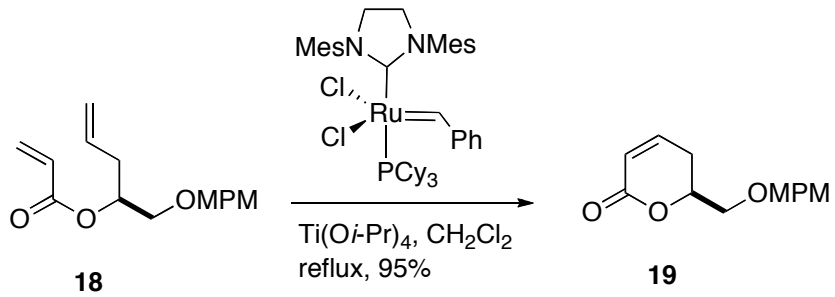
74.5, 73.4, 73.0, 39.3, 35.0, 31.2, 20.4, 19.2, 18.35 (6C), 18.33 (3C), 18.29 (3C), 17.5, 13.1 (6C); HRMS (ESI) calcd for $C_{34}H_{65}IO_3Si_2Na$ [$(M + Na)^+$] 727.3409, found 727.3438.



Diene 18. To a mixture of epoxide **17** (3.28 g, 16.9 mmol) and CuI (322 mg, 1.69 mmol) in THF (140 mL) cooled to −35 °C was added vinylmagnesium bromide (1.0 M solution in THF, 20.3 mL, 20.3 mmol), and the resultant mixture was allowed to warm to −20 °C over a period of 30 min. The reaction was quenched with saturated aqueous NH_4Cl solution. The resultant mixture was allowed to warm to room temperature and then extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (10 to 20% EtOAc/hexanes) gave a homoallylic alcohol, which was contaminated with some impurities but was used in the next reaction without further purification.

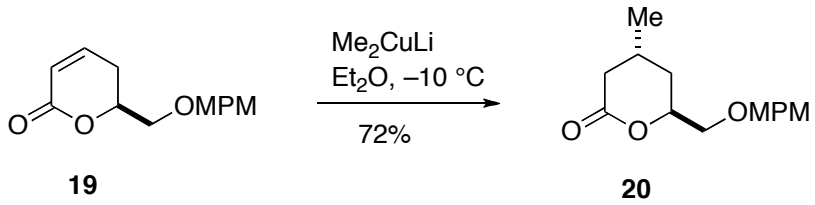
To a solution of the above homoallylic alcohol in CH_2Cl_2 (140 mL) cooled to 0 °C were added *i*-Pr_2NEt (9.00 mL, 49.2 mmol) and acryloyl chloride (1.73 ml, 21.3 mmol), and the resultant solution was stirred at 0 °C for 3 h. The reaction was quenched with saturated aqueous $NaHCO_3$ solution, and the resultant mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (6 to 7.5% EtOAc/hexanes) gave diene **18** (4.41 g, 95%

for the two steps) as a colorless oil: $[\alpha]_D^{24}$ −8.6 (*c* 1.00, C_6H_6); IR (film) 2933, 1723, 1613, 1514, 1406, 1249, 1195, 1037, 809 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.25—7.20 (m, 2H), 6.87—6.83 (m, 2H), 6.39 (dd, *J* = 17.5, 1.4 Hz, 1H), 6.11 (dd, *J* = 17.5, 10.6 Hz, 1H), 5.81 (dd, *J* = 10.6, 1.4 Hz, 1H), 5.72 (dddd, *J* = 17.2, 10.0, 7.2, 7.2 Hz, 1H), 5.14 (m, 1H) 5.07 (dddd, *J* = 17.2, 1.4, 1.4, 1.0 Hz, 1H), 5.04 (dddd, *J* = 10.0, 2.1, 1.4, 1.0 Hz, 1H), 4.49 (d, *J* = 11.7 Hz, 1H), 4.42 (d, *J* = 11.7 Hz, 1H), 3.78 (s, 3H), 3.53 (dd, *J* = 10.6, 5.5 Hz, 1H), 3.51 (dd, *J* = 10.6, 4.8 Hz, 1H), 2.42 (ddddd, *J* = 14.1, 7.2, 7.2, 2.1, 1.4 Hz, 1H), 2.39 (ddddd, *J* = 14.1, 7.2, 7.2, 1.0, 1.0 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.7, 159.2, 133.1, 130.8, 130.1, 129.2 (2C), 128.6, 118.0, 113.8 (2C), 72.8, 72.1, 70.1, 55.3, 35.4; HRMS (ESI) calcd for $C_{16}H_{20}O_4Na$ $[(M + Na)^+]$ 299.1254, found 299.1258.



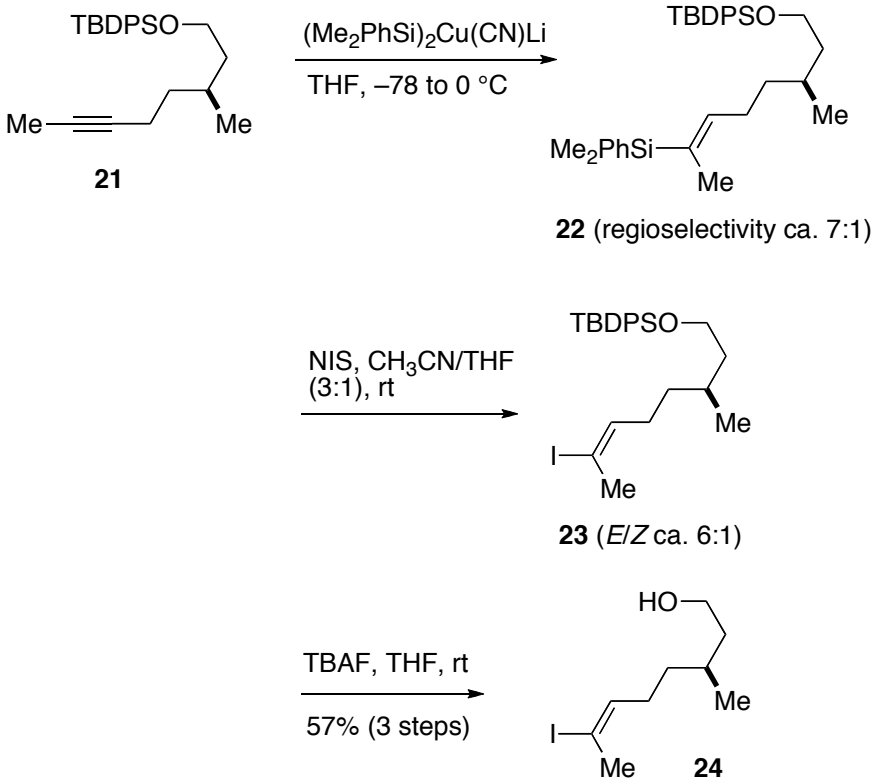
α,β-Unsaturated lactone 19. To a solution of diene **18** (0.510 g, 1.85 mmol) in CH_2Cl_2 (150 mL) was added Ti(O*i*-Pr)$_4$ (0.17 mL, 0.57 mmol), and the resultant solution was stirred at 45 °C for 1 h. To this mixture was added a solution of the Grubbs second-generation catalyst (80 mg, 94 μmol) in CH_2Cl_2 (30 mL), and the resultant solution was stirred at 45 °C for 12 h. After being cooled to room temperature, the reaction mixture was treated with Et_3N and stirred at room temperature for 1.5 h under air. The resultant mixture was passed through a pad of silica gel (EtOAc) and concentrated under reduced pressure. Purification of the residue by flash

chromatography on silica gel (15 to 40% EtOAc/hexanes) gave α,β-unsaturated lactone **19** (0.437 g, 95%) as a pale brown oil: $[\alpha]_D^{24}$ −94.4 (*c* 1.00, $CHCl_3$); IR (film) 2910, 1719, 1611, 1513, 1246, 1085, 1050, 814 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.29—7.20 (m, 2H), 6.91—6.84 (m, 3H), 6.00 (ddd, *J* = 9.6, 2.8, 1.0 Hz, 1H), 4.58 (dddd, *J* = 11.7, 4.8, 4.8, 4.1 Hz, 1H), 4.53 (d, *J* = 11.6 Hz, 1H) 4.50 (d, *J* = 11.6 Hz, 1H), 3.79 (s, 3H), 3.66 (dd, *J* = 10.3, 4.8 Hz, 1H), 3.64 (dd, *J* = 10.3, 4.8 Hz, 1H), 2.53 (dddd, *J* = 18.5, 11.7, 2.8, 2.8 Hz, 1H), 2.37 (dddd, *J* = 18.5, 5.5, 4.1, 1.0 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 163.7, 159.3, 144.9, 129.7, 129.4 (2C), 121.2, 113.8 (2C), 76.6, 73.3, 70.4, 55.3, 26.2; HRMS (ESI) calcd for $C_{14}H_{16}O_4Na$ $[(M + Na)^+]$ 271.0935, found 271.0939.



Lactone 20. To a suspension of CuI (286 mg, 1.50 mmol) in diethyl ether (10 mL) cooled to −10 °C was added MeLi (1.09 M solution in diethyl ether, 2.70 mL, 2.97 mmol), and the resultant mixture was stirred at −10 °C for 1 h. To this mixture was added a solution of α,β-unsaturated lactone **19** (0.241 g, 0.971 mmol) in diethyl ether (5 mL +2 mL rinse). The resultant solution was stirred at −10 °C for 40 min. The reaction was quenched with saturated aqueous NH_4Cl solution, and the resultant mixture was filtered through a pad of Celite, and the filtrate was extracted with diethyl ether. The organic layer was washed with brine. The aqueous layer was cooled to 0 °C and carefully acidified with 1 M aqueous HCl solution (pH = ca. 4). The resultant mixture was extracted repeatedly with EtOAc. The combined organic layer was dried over

Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (30 to 80% EtOAc/hexanes) gave lactone **20** (0.183 g, 72%): $[\alpha]_D^{25}$ +26.6 (*c* 1.00, $CHCl_3$); IR (film) 2955, 1735, 1612, 1513, 1247, 1173, 1080, 1032, 819, 419 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.26—7.20 (m, 2H), 6.89—6.83 (m, 2H), 4.54 (dddd, *J* = 8.6, 5.2, 4.8, 4.8 Hz, 1H), 4.48 (d, *J* = 11.7 Hz, 1H) 4.46 (d, *J* = 11.7 Hz, 1H), 3.78 (s, 3H), 3.57 (dd, *J* = 10.3, 4.8 Hz, 1H), 3.53 (dd, *J* = 10.3, 5.2 Hz, 1H), 2.55 (ddd, *J* = 16.5, 5.5, 0.7 Hz, 1H), 2.18 (m, 1H), 2.11 (dd, *J* = 16.5, 8.9 Hz, 1H), 1.91 (dddd, *J* = 14.0, 8.6, 6.2, 0.7 Hz, 1H), 1.55 (ddd, *J* = 14.0, 6.5, 4.8 Hz, 1H), 1.04 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.6, 159.3, 129.8, 129.3 (2C), 113.8 (2C), 76.1, 73.2, 71.3, 55.2, 37.6, 31.7, 23.7, 21.1; HRMS (ESI) calcd for $C_{15}H_{20}O_4Na$ [$(M + Na)^+$] 287.1254, found 287.1257.



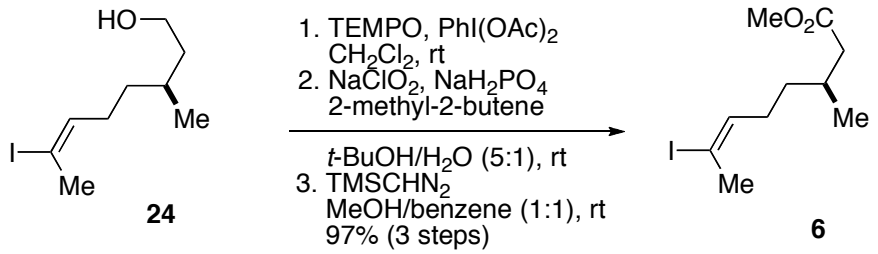
Alcohol 24. To a suspension of CuCN (430 mg, 4.80 mmol) in THF (10 mL) cooled to

0 °C was added $Me_2PhSiLi$ (ca. 0.96 M in THF, 10 mL, 9.6 mmol). After being stirred at 0 °C for 20 min, the reaction mixture was cooled to −78 °C. To this mixture was added a solution of alkyne **21** (0.940 g, 2.49 mmol) in THF (6 mL + 3 mL rinse twice). The resultant mixture was stirred at −78 °C for 2 h and then at 0 °C for 2 h. The reaction was quenched with a 9:1 mixture of saturated aqueous NH_4Cl solution and 28% NH_4OH solution at 0 °C, filtered through a pad of Celite, and the filtrate was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (hexanes) gave vinyl silane **22** (1.361 g), which was contaminated with silane byproducts and used in the next reaction without further purification. ^{1}H NMR spectrum of the crude product indicated that the regioselectivity of the reaction was ca. 7:1. Data for **22**: ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72—7.60 (m, 5H), 7.52—7.26 (m, 10H), 5.77 (ddq, *J* = 6.9, 6.9, 1.7 Hz, 1H), 3.74—3.62 (m, 2H), 2.15—2.00 (m, 2H), 1.64 (d, *J* = 1.7 Hz, 3H), 1.65—1.57 (m, 2H), 1.40—1.31 (m, 2H), 1.16 (m, 1H), 1.03 (s, 9H), 0.83 (d, *J* = 6.5 Hz, 3H), 0.30 (s, 6H); HRMS (FAB) calcd for $C_{33}H_{47}OSi_2$ [$(M + Na)^+$] 515.3165, found 515.3163.

To a solution of the above vinyl silane **22** in CH_3CN/THF (3:1, v/v, 24 mL) was added NIS (1.13 g, 5.04 mmol), and the resultant mixture was stirred at room temperature for 18 h. The reaction was quenched with a 1:1 mixture of saturated aqueous $Na_2S_2O_3$ solution and saturated aqueous $NaHCO_3$ solution at 0 °C, and the resultant mixture was stirred vigorously at room temperature until the layers became colorless. The resultant mixture was diluted with EtOAc, washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (0 to 0.5% diethyl ether/hexanes) gave an

inseparable mixture of vinyl iodide **23** and its isomers (1.207 g), which was used in the next reaction without further purification. ^{1}H NMR spectrum of the crude product indicated that *E/Z* = ca. 6:1 for the major regioisomer. Data for **23**: ^{1}H NMR (600 MHz, C_6D_6) δ 7.84—7.72 (m, 5H), 7.30—7.18 (m, 5H), 6.06 (m, 1H), 3.76—3.58 (m, 2H), 2.08 (s, 3H), 1.75—1.61 (m, 2H), 1.54—1.43 (m, 2H), 1.26 (m, 1H), 1.19 (s, 9H), 1.09 (m, 1H), 0.88 (dddd, *J* = 13.7, 9.2, 7.6, 6.2 Hz, 1H), 0.68 (d, *J* = 6.5 Hz, 3H); HRMS (FAB) calcd for $C_{25}H_{36}IOSi$ [$(M + Na)^+$] 507.1580, found 507.1582.

To a solution of the above vinyl iodide **23** in THF (15 mL) was added TBAF (1.0 M solution in THF, 11.6 mL, 11.6 mmol). The resultant solution was stirred at room temperature for 1 h 50 min and then concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (1 to 3% EtOAc/benzene) followed by preparative HPLC gave alcohol **24** (0.372 g, 57% for the three steps) as a colorless oil: $[\alpha]_D^{26}$ −6.9 (*c* 1.00, C_6H_6); IR (film) 3345, 2923, 1458, 1377, 1373, 1057, 419 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.12 (ddq, *J* = 7.6, 7.6, 1.4 Hz, 1H), 3.71—3.61 (m, 2H), 2.35 (d, *J* = 1.4 Hz, 3H), 2.09—1.95 (m, 2H), 1.63—1.51 (m, 2H), 1.49—1.30 (m, 2H), 1.21 (dddd, *J* = 13.4, 9.3, 7.6, 5.8 Hz, 1H), 0.89 (d, *J* = 6.5 Hz, 3H) (one proton missing presumably due to H/D exchange); ^{13}C NMR (150 MHz, C_6D_6) δ 141.6, 93.6, 60.6, 39.8, 36.2, 29.2, 28.3, 27.3, 19.4; HRMS (ESI) calcd for $C_9H_{18}IO$ [$(M + H)^+$] 269.0397, found 269.0398.

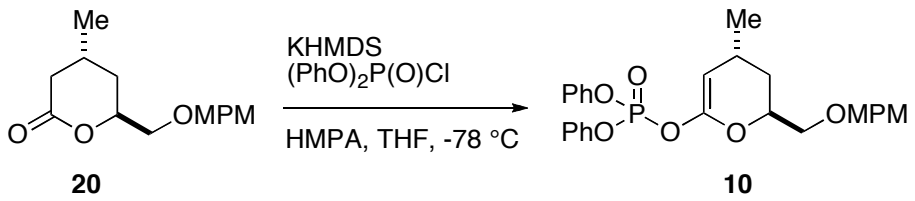


Vinyl iodide 6. To a solution of alcohol **24** (0.238 g, 0.888 mmol) in CH_2Cl_2 (9 mL) were added $PhI(OAc)_2$ (315 mg, 0.978 mmol) and TEMPO (14.0 mg, 89.6 μmol), and the resultant solution was stirred at room temperature for 3.5 h. To the reaction mixture were added additional portions of $PhI(OAc)_2$ (86.0 mg, 0.263 mmol) and TEMPO (14 mg, 89.6 μmol), and the resultant mixture was stirred at room temperature for 2 h. The reaction was quenched with a 1:1 mixture of saturated aqueous $Na_2S_2O_3$ solution and saturated aqueous $NaHCO_3$ solution at 0 °C, and the resultant solution was stirred at room temperature for further 25 min. The resultant mixture was extracted with EtOAc, and the organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residual crude aldehyde was used in the next reaction without further purification.

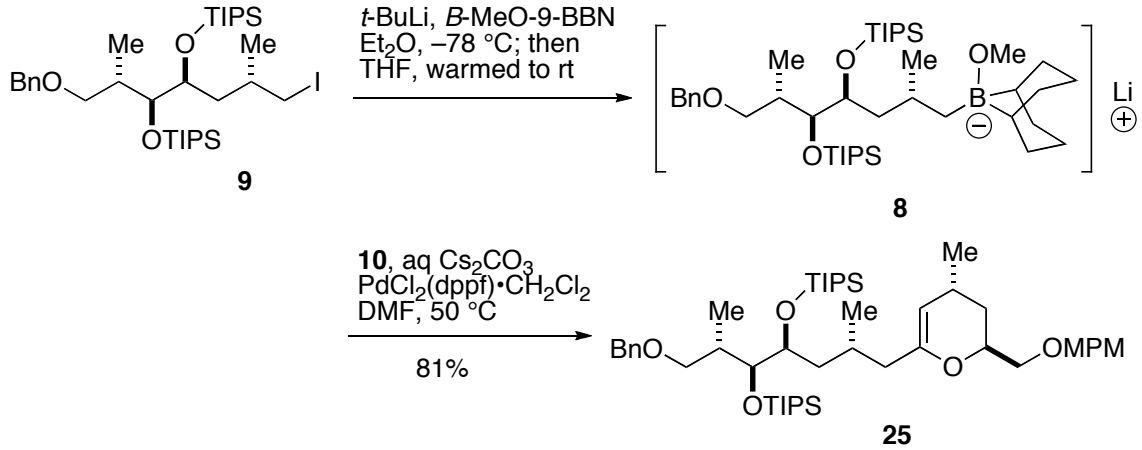
To a solution of the above aldehyde, NaH_2PO_4 (120 mg, 1.00 mmol), and 2-methyl-2-butene (1.90 mL, 17.8 mmol) in *t*-BuOH/H_2O (5:1, v/v, 9 mL) cooled to 0 °C was added $NaClO_2$ (280 mg, 3.12 mmol), and the resultant mixture was stirred at room temperature for 50 min. The reaction mixture was cooled to 0 °C and carefully acidified with 1 M aqueous HCl solution (pH = ca. 4). The resultant mixture was extracted repeatedly with $CHCl_3$, and the organic layer was dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was passed through a pad of silica gel (5 to 10 % EtOAc/hexanes) to give a carboxylic acid (0.244 g, ca. 97%), which was used in the next reaction without further purification.

To a solution of the above carboxylic acid in methanol/benzene (1:1, v/v, 8 mL) cooled to 0 °C was added $TMSCHN_2$ (2.0 M solution in hexanes, 1.29 mL, 2.58 mmol). The reaction mixture was stirred at room temperature for 35 min and then concentrated under reduced pressure. Purification of the residue by flash

chromatography on silica gel (1% EtOAc/hexanes) to give vinyl iodide **6** (0.256 g, 97% for the three steps) as a colorless oil: $[\alpha]_D^{25}$ −5.4 (*c* 1.13, C_6H_6); IR (film) 2952, 1737, 1435, 1202, 1160, 1058 cm^{-1}; ^1H NMR (600 MHz, C_6D_6) δ 6.01 (dd, *J* = 7.6, 7.6 Hz, 1H), 3.33 (s, 3H), 2.06 (s, 3H), 2.00 (dd, *J* = 15.1, 5.8 Hz, 1H), 1.87 (dd, *J* = 15.1, 7.9 Hz, 1H), 1.78 (m, 1H), 1.70—1.55 (m, 2H), 1.05 (dddd, *J* = 13.4, 9.7, 6.2, 6.2 Hz, 1H), 0.86 (dddd, *J* = 13.4, 9.7, 7.9, 6.2 Hz, 1H), 0.73 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (150 MHz, C_6D_6) δ 172.5, 141.1, 93.8, 50.9, 41.2, 35.6, 29.9, 28.2, 27.3, 19.5; HRMS (ESI) calcd for $C_{10}H_{17}IO_2Na$ [$(M + Na)^+$] 319.0165, found 319.0159.

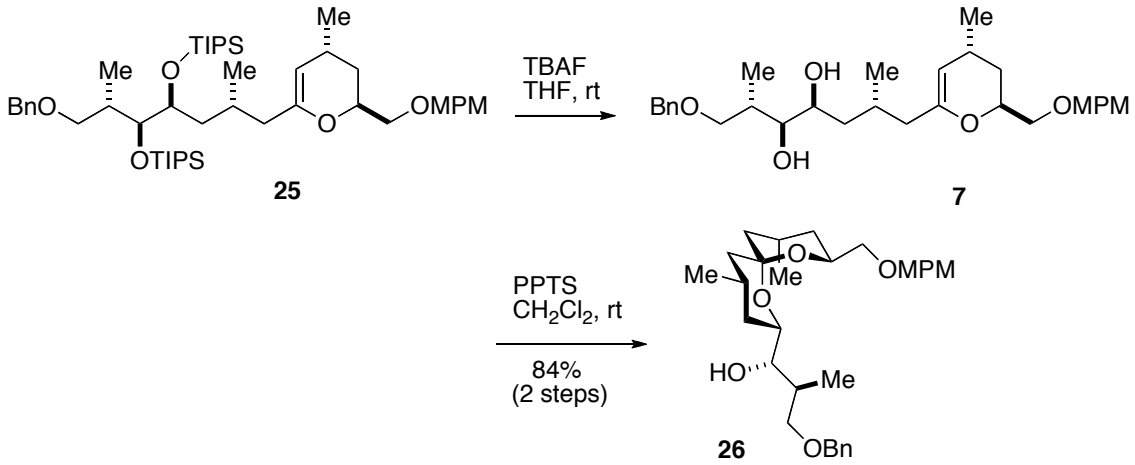


Enol phosphate 10. To a solution of lactone **20** (0.947 g, 3.59 mmol) in THF (35 mL) were added HMPA (1.87 mL, 10.8 mmol) and $(PhO)_2P(O)Cl$ (0.89 mL, 4.3 mmol), and the resultant solution was cooled to −78 °C. To this solution was added KHMDS (0.5 M solution in toluene, 9.30 mL, 4.65 mmol), and the resultant solution was stirred at −78 °C for 40 min. The reaction was quenched with 3% NH_4OH solution. The resultant mixture was diluted with diethyl ether and allowed to warm to room temperature over a period of 40 min. The resultant mixture was extracted with EtOAc, and the organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was passed through a pad of silica gel (eluted with 15% EtOAc/hexanes) to give enol phosphate **10**, which was immediately used in the next reaction.



Endocyclic enol ether 25. To a solution of iodide **9** (1.086 g, 1.542 mmol) in diethyl ether (15 mL) was added *B*-MeO-9-BBN (1.0 M solution in hexanes, 4.0 mL, 4.0 mmol). To this solution cooled to −78 °C was added *t*-BuLi (1.59 M in pentane, 2.90 mL, 4.61 mmol) at once. The resultant mixture was stirred at −78 °C for 5 min and then THF (15 mL) was added. The resultant solution was allowed to warm to room temperature over a period of 3 h 25 min. To this solution was added 3 M aqueous Cs_2CO_3 solution (1.54 mL, 4.62 mmol), and the resultant mixture was stirred at room temperature for 20 min. To this mixture were added a solution of the above crude enol phosphate **10** in DMF (10 mL + 5 mL rinse) and $PdCl_2(dppf)•CH_2Cl_2$ (126 mg, 0.154 mmol). The resultant mixture was stirred at 50 °C for 16 h. The reaction mixture was diluted with H_2O and extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (0.5 to 1% EtOAc/hexanes) gave endocyclic enol ether **25** (1.021 g, 81%) as a colorless oil: $[\alpha]_D^{24}$ −5.5 (*c* 1.00, C_6H_6); IR (film) 2945, 2866, 1513, 1462, 1248, 1095, 882, 678 cm^{-1}; ^1H NMR (600 MHz, C_6D_6) δ 7.37—7.32 (m, 2H), 7.26—7.22 (m, 2H), 7.21—7.16 (m, 2H), 7.09 (m, 1H), 6.83—6.78 (m, 2H), 4.50 (d, *J* = 4.1 Hz, 1H), 4.49 (d, *J* = 12.0 Hz, 1H) 4.46 (d, *J* = 12.0 Hz, 1H),

4.42 (s, 2H), 4.21 (m, 1H), 4.17 (m, 1H), 4.15 (dd, J = 5.2, 3.8 Hz, 1H), 3.79 (dd, J = 8.9, 3.4 Hz, 1H), 3.66 (dd, J = 9.6, 5.2 Hz, 1H), 3.61 (dd, J = 7.9, 7.9 Hz, 1H), 3.51 (dd, J = 9.6, 6.2 Hz, 1H), 3.31 (s, 3H), 2.46 (m, 1H), 2.25 (m, 1H), 2.20—2.11 (m, 2H), 2.05 (m, 1H), 1.88 (ddd, J = 13.0, 10.0, 2.7 Hz, 1H), 1.79 (ddd, J = 13.4, 8.9, 5.8 Hz, 1H), 1.66 (br dd, J = 11.3, 11.3 Hz, 1H), 1.51 (ddd, J = 13.4, 2.8, 2.8 Hz, 1H), 1.47 (d, J = 6.8 Hz, 3H), 1.24—1.13 (m, 42H), 1.11 (d, J = 6.9 Hz, 3H), 0.97 (d, J = 6.9 Hz, 3H); ^{13}C NMR (150 MHz, C_6D_6) δ 159.7, 152.1, 139.5, 131.0, 129.5 (2C), 128.4 (2C), 128.3 (2C), 127.5, 114.1 (2C), 103.4, 77.9, 75.1, 73.7, 73.31, 73.29, 72.2, 71.2, 54.7, 43.9, 39.2, 36.6, 32.1, 27.6, 24.5, 23.1, 20.0, 18.74 (3C), 18.71 (3C), 18.68 (3C), 18.6 (3C), 17.3, 13.6 (6C); HRMS (ESI) calcd for $C_{49}H_{84}O_6Si_2Na$ [(M + Na)$^+$] 847.5699, found 847.5676.

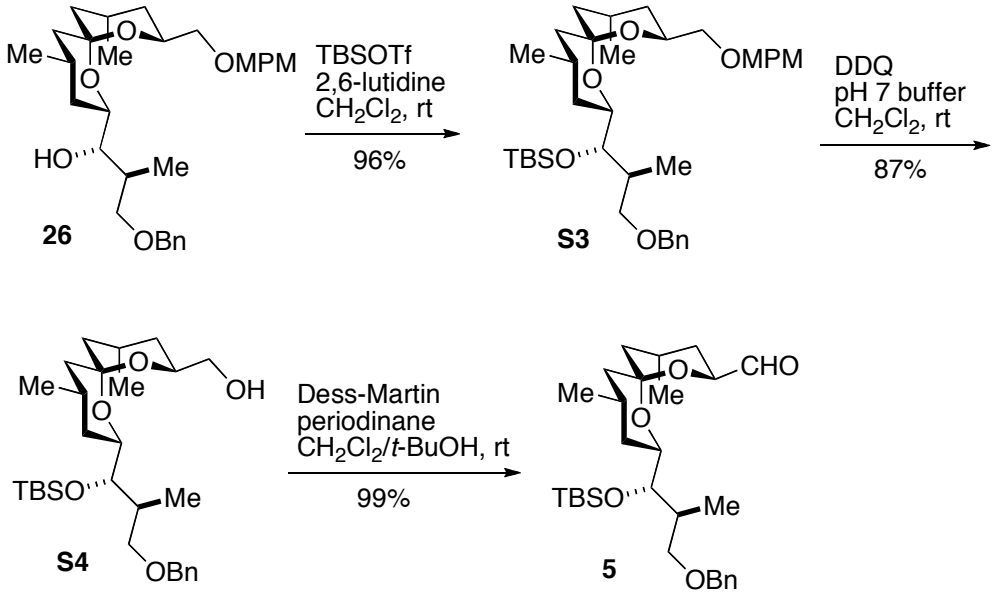


Spiroacetal 26. To a solution of endocyclic enol ether **25** (1.275 g, 1.546 mmol) in THF (8 mL) was added TBAF (1.0 M solution in THF, 15.2 mL, 15.2 mmol). The resultant solution was stirred at room temperature for 3 h 10 min and then concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (20 to 30% EtOAc/benzene) gave dihydroxy enol ether **7** (0.8194 g), which was used in the next reaction without further purification: $[\alpha]_D^{25}$ +43.5 (c 1.00, C_6H_6); IR (film) 3461,

2953, 2867, 1513, 1455, 1248, 1092, 420 cm^{-1}; ^1H NMR (600 MHz, C_6D_6) δ 7.28—7.22 (m, 2H), 7.21—7.11 (m, 4H), 7.06 (m, 1H), 6.84—6.79 (m, 2H), 4.50 (d, *J* = 4.1 Hz, 1H), 4.41 (d, *J* = 11.7 Hz, 1H) 4.38 (d, *J* = 11.7 Hz, 1H), 4.19 (s, 2H), 4.11 (m, 1H), 3.80 (br ddd, *J* = 9.6, 2.4, 2.4 Hz, 1H), 3.55 (dd, *J* = 10.0, 6.2 Hz, 1H), 3.41—3.35 (m, 3H), 3.34—3.27 (m, 2H), 3.30 (s, 3H), 2.80 (br s, 1H), 2.32 (m, 1H), 2.18—2.06 (m, 3H), 2.01 (m, 1H), 1.90 (ddd, *J* = 14.4, 10.0, 4.8 Hz, 1H), 1.66 (ddd, *J* = 13.4, 9.6, 6.2 Hz, 1H), 1.32 (m, 1H), 1.24 (ddd, *J* = 13.7, 8.9, 3.4 Hz, 1H), 1.03 (d, *J* = 6.5 Hz, 3H), 0.91 (d, *J* = 6.9 Hz, 3H), 0.90 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (150 MHz, C_6D_6) δ 159.7, 152.2, 138.4, 130.9, 129.6 (2C), 128.6 (2C), 128.5, 128.3 (2C), 114.1 (2C), 103.5, 78.6, 73.9, 73.4, 73.1, 72.4, 71.4, 70.1, 54.7, 42.5, 41.4, 36.5, 31.8, 28.0, 24.7, 23.2, 19.8, 15.0; HRMS (ESI) calcd for $C_{31}H_{44}O_6Na$ [$(M + Na)^+$] 535.3041, found 535.3042.

To a solution of the above dihydroxy enol ether **7** in CH_2Cl_2 (20 mL) cooled to 0 °C was added PPTS (79.0 mg, 0.314 mmol), and the resultant mixture was stirred at room temperature for 2 days. The reaction was quenched with saturated aqueous $NaHCO_3$ solution at 0 °C. The resultant mixture was diluted with EtOAc and washed with brine. The organic layer was dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (10 to 13% EtOAc/hexanes) gave spiroacetal **26** (0.6651 g, 84% for the two steps) as a colorless oil: $[\alpha]_D^{24}$ +25.1 (*c* 1.31, $CHCl_3$); IR (film) 3494, 2930, 1613, 1513, 1455, 1247, 1090, 982, 698 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.33—7.28 (m, 4H), 7.27—7.20 (m, 3H), 6.86—6.82 (m, 2H), 4.48 (d, *J* = 11.6 Hz, 1H), 4.46 (s, 2H) 4.45 (d, *J* = 11.6 Hz, 1H), 3.96 (m, 1H), 3.87 (ddd, *J* = 11.7, 3.4, 3.0 Hz, 1H), 3.77 (s, 3H), 3.60 (dd, *J* = 9.3, 5.8 Hz, 1H), 3.47 (dd, *J* = 9.3, 5.8 Hz, 1H), 3.41 (dd, *J* = 10.3, 6.2 Hz, 1H), 3.36 (dd, *J* = 10.3, 4.1 Hz, 1H), 3.26 (ddd, *J* = 7.5, 6.2, 3.4 Hz, 1H), 2.75 (d, *J* = 6.2 Hz,

1H), 2.11—2.00 (m, 2H), 1.97 (m, 1H), 1.65 (ddd, J = 13.0, 3.8, 1.4 Hz, 1H), 1.61 (dd, J = 14.1, 5.8 Hz, 1H), 1.53 (ddd, J = 13.0, 11.7, 5.8 Hz, 1H), 1.51—1.45 (m, 2H), 1.29 (br ddd, J = 13.0, 2.7, 2.4 Hz, 1H), 1.16 (m, 1H), 1.14 (d, J = 7.2 Hz, 3H), 0.99 (m, 1H), 0.98 (d, J = 6.8 Hz, 3H), 0.86 (d, J = 6.5 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.0, 138.5, 130.7, 129.0 (2C), 128.3 (2C), 127.5 (2C), 127.4, 113.6 (2C), 98.2, 77.2, 73.5, 73.3, 73.1, 72.9, 69.6, 64.7, 55.2, 44.4, 40.5, 35.74, 35.68, 33.0, 24.8, 24.6, 22.1, 21.1, 14.8; HRMS (ESI) calcd for $C_{31}H_{44}O_6Na$ [(M + Na)$^+$] 535.3041, found 535.3045.



Aldehyde 5. To a solution of spiroacetal **26** (645.3 mg, 1.260 mmol) in CH_2Cl_2 (12 mL) cooled to 0 °C were added 2,6-lutidine (0.30 mL, 2.5 mmol) and TBSOTf (0.50 mL, 1.9 mmol), and the resultant solution was stirred at room temperature for 3 h 50 min. The reaction was quenched with saturated aqueous NH_4Cl solution at 0 °C, and the resultant mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (3% EtOAc/hexanes) gave silyl ether **S3** (0.7592 g, 96%) as a colorless oil: $[\alpha]_D^{25}$ +0.6 (c 1.96, $CHCl_3$); IR (film) 2951, 2855, 1513,

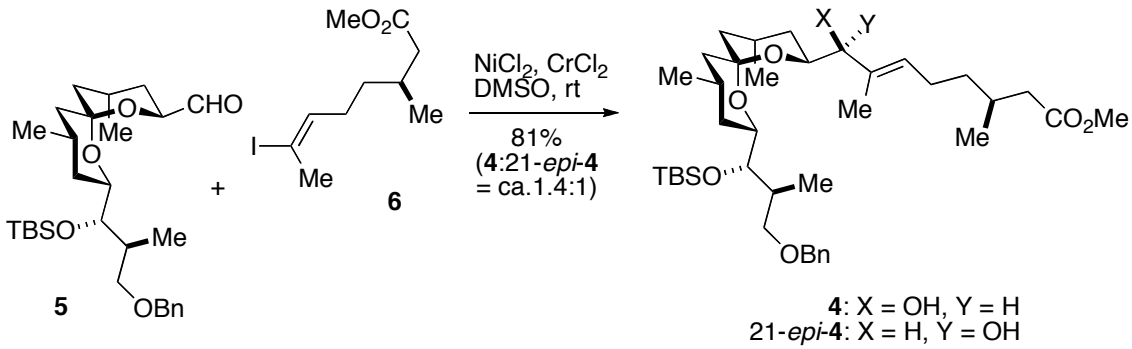
1455, 1249, 1086, 1038, 984, 836, 775 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.33—7.28 (m, 4H), 7.26—7.20 (m, 3H), 6.86—6.82 (m, 2H), 4.50—4.42 (m, 4H), 3.94 (m, 1H), 3.78 (s, 3H), 3.73 (ddd, *J* = 12.0, 3.8, 2.1 Hz, 1H), 3.59 (dd, *J* = 8.9, 3.4 Hz, 1H), 3.49 (dd, *J* = 6.5, 3.8 Hz, 1H), 3.40 (dd, *J* = 10.0, 5.8 Hz, 1H), 3.35 (dd, *J* = 10.0, 5.2 Hz, 1H), 3.26 (dd, *J* = 8.9, 8.2 Hz, 1H), 2.04 (dddd, *J* = 14.8, 13.7, 6.9, 3.4 Hz, 1H), 1.98—1.88 (m, 2H), 1.64 (ddd, *J* = 13.1, 3.8, 1.4 Hz, 1H), 1.61—1.50 (m, 3H), 1.41 (dd, *J* = 13.7, 3.4 Hz, 1H), 1.32 (br ddd, *J* = 13.1, 2.8, 2.8 Hz, 1H), 1.13 (d, *J* = 7.2 Hz, 3H), 1.03 (d, *J* = 6.9 Hz, 3H), 0.99—0.89 (m, 2H), 0.85 (d, *J* = 6.5 Hz, 3H), 0.83 (s, 9H), 0.00 (s, 3H), −0.01 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 159.0, 139.0, 130.7, 129.2 (2C), 128.2 (2C), 127.5 (2C), 127.3, 113.6 (2C), 97.8, 76.1, 73.4, 73.3, 72.92, 72.88, 72.5, 64.4, 55.2, 44.7, 40.8, 35.9, 33.4, 33.2, 25.9 (3C), 25.2, 24.7, 22.3, 21.0, 18.1, 15.9, −4.3, −4.7; HRMS (ESI) calcd for $C_{37}H_{58}O_6SiNa$ [$(M + Na)^+$] 649.3895, found 649.3894.

To a solution of silyl ether **S3** (23.6 mg, 37.8 μmol) in CH_2Cl_2/pH 7 buffer (10:1, v/v, 0.77 mL) cooled to 0 °C was added DDQ (9.5 mg, 42 μmol), and the resultant solution was stirred at room temperature for 2.5 h. The reaction was quenched with saturated aqueous $NaHCO_3$ solution at 0 °C, and the resultant mixture was stirred at room temperature for 10 min. The resultant mixture was extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (5:1:25, $CHCl_3$/EtOAc/hexanes) gave alcohol **S4** (16.6 mg, 87%) as a colorless oil: $[\alpha]_D^{24}$ +7.5 (*c* 1.00, $CHCl_3$); IR (film) 3480, 2927, 1455, 1254, 1076, 981, 882, 836, 775 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.35—7.29 (m, 4H), 7.25 (m, 1H), 4.49 (d, *J* = 12.0 Hz, 1H), 4.45 (d, *J* = 12.0 Hz, 1H), 3.86 (dddd, *J* = 11.3, 6.9, 4.1, 3.4 Hz, 1H), 3.69

(ddd, J = 11.7, 3.8, 2.0 Hz, 1H), 3.59 (dd, J = 8.9, 3.5 Hz, 1H), 3.57 (m, 1H), 3.49 (dd, J = 6.5, 3.8 Hz, 1H), 3.47 (dd, J = 11.3, 6.9 Hz, 1H), 3.26 (dd, J = 8.9, 8.0 Hz, 1H), 2.04 (m, 1H), 1.98—1.83 (m, 2H), 1.63 (ddd, J = 13.1, 3.8, 1.7 Hz, 1H), 1.60—1.50 (m, 3H), 1.43 (ddd, J = 13.7, 4.1, 1.0 Hz, 1H), 1.21 (br ddd, J = 13.1, 3.4, 3.4 Hz, 1H), 1.14 (d, J = 7.2 Hz, 3H), 1.03 (d, J = 6.8 Hz, 3H), 1.01—0.89 (m, 2H), 0.86 (d, J = 6.5 Hz, 3H), 0.84 (s, 9H), 0.03 (s, 3H), 0.00 (s, 3H) (one proton missing presumably due to H/D exchange); ^{13}C NMR (150 MHz, $CDCl_3$) δ 138.9, 128.2 (2C), 127.5 (2C), 127.3, 97.7, 76.1, 73.2, 72.9, 72.7, 66.4, 65.4, 44.6, 40.9, 35.9, 33.1, 32.2, 25.9 (3C), 25.3, 24.6, 22.3, 21.0, 18.1, 15.9, −4.3, −4.6; HRMS (ESI) calcd for $C_{29}H_{50}O_5SiNa$ [$(M + Na)^+$] 529.3320, found 529.3336.

To a solution of alcohol **S4** (21.4 mg, 42.3 μmol) in CH_2Cl_2/*t*-BuOH (10:1, v/v, 0.88 mL) cooled to 0 °C was added Dess—Martin periodinane (90 mg, 0.21 mmol), and the resultant solution was stirred at room temperature for 1 h. The reaction was quenched with a 1:1 mixture of saturated aqueous $Na_2S_2O_3$ solution and saturated aqueous $NaHCO_3$ solution at 0 °C, and the resultant solution was stirred at room temperature for 15 min. The resultant mixture was extracted with diethyl ether, and the organic layer was washed with brine, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (7% EtOAc/hexanes) gave aldehyde **5** (21.2 mg, 99%) as a colorless oil: $[\alpha]_D^{26}$ −10.8 (c 0.50, $CHCl_3$); IR (film) 2952, 2927, 2855, 1739, 1456, 1076, 978, 836, 774 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 9.65 (s, 1H), 7.33—7.29 (m, 4H), 7.25 (m, 1H), 4.49 (d, J = 12.0 Hz, 1H), 4.43 (d, J = 12.0 Hz, 1H), 4.16 (dd, J = 11.7, 3.8 Hz, 1H), 3.69 (ddd, J = 11.7, 3.4, 2.0 Hz, 1H), 3.56 (dd, J = 8.9, 3.4 Hz, 1H), 3.48 (dd, J = 6.8, 3.4 Hz, 1H), 3.26 (dd, J = 8.9, 7.9 Hz, 1H), 2.06—1.93 (m, 3H), 1.72 (ddd, J = 13.4, 3.8,

1.7 Hz, 1H), 1.65 (ddd, J =13.4, 11.7, 5.5 Hz, 1H), 1.64—1.59 (m, 2H), 1.55 (m, 1H), 1.44 (dd, J = 14.1, 4.1 Hz, 1H), 1.16 (d, J = 7.3 Hz, 3H), 1.05—0.92 (m, 2H), 1.01 (d, J = 6.9 Hz, 3H), 0.88 (d, J = 6.5 Hz, 3H), 0.83 (s, 9H), −0.02 (s, 3H), −0.03 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 202.8, 138.9, 128.2 (2C), 127.5 (2C), 127.3, 98.3, 75.9, 73.3, 73.1, 72.9, 70.9, 44.2, 40.6, 36.0, 32.9, 30.9, 25.9 (3C), 25.2, 24.4, 22.2, 20.5, 18.1, 15.7, −4.3, −4.6; HRMS (ESI) calcd for $C_{29}H_{48}O_5SiNa$ [(M + Na)$^+$] 527.3163, found 527.3173.

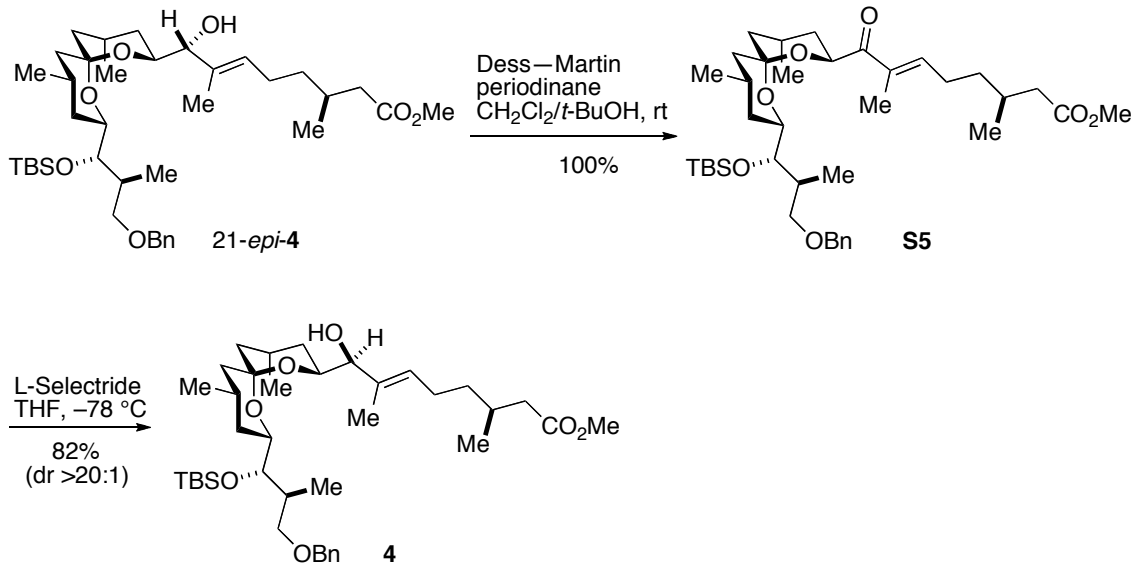


C9—C28 Subunit 4 and 21-*epi*-4. To a mixture of $NiCl_2/CrCl_2$ (1%, w/w, 38.1 mg, weighted in a glove box) was added a mixture of aldehyde **5** (15.6 mg, 30.9 μmol) and vinyl iodide **6** (20.5 mg, 69.3 μmol) in degassed DMF (0.5 mL + 0.4 mL rinse twice). The resultant mixture was stirred at room temperature for 13 h. The reaction was quenched with D/L-serine (1.0 M solution in saturated aqueous $NaHCO_3$ solution) at 0 °C. The resultant solution was stirred at room temperature for 15 min. The resultant mixture was extracted with diethyl ether, and the organic layer was washed with brine, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (3 to 10% EtOAc/hexanes) gave a 1.4:1 mixture of C9—C28 subunit **4** and **21-*epi*-4** (16.9 mg, 81%) as a colorless oil. Separation of the two diasteromeric alcohols by preparative TLC (13% EtOAc/hexanes,

developed thrice) gave C9—C28 subunit **4** (8.5 mg, 41%) and **21-*epi*-4** (5.0 mg, 24%). Data for **4**: $[\alpha]_D^{27}$ +25.1 (*c* 0.37, $CHCl_3$); IR (film) 3574, 2952, 2931, 1739, 1455, 1254, 1078, 1025, 837, 776 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.33—7.29 (m, 4H), 7.25 (m, 1H), 5.40 (ddq, *J* = 7.2, 7.2, 1.4 Hz, 1H), 4.48 (d, *J* = 12.0 Hz, 1H), 4.45 (d, *J* = 12.0 Hz, 1H), 3.75 (m, 1H), 3.73 (dd, *J* = 10.7, 3.1 Hz, 1H), 3.69 (m, 1H), 3.64 (s, 3H), 3.59 (dd, *J* = 8.9, 3.8 Hz, 1H), 3.51 (dd, *J* = 6.5, 3.8 Hz, 1H), 3.27 (dd, *J* = 8.9, 8.2 Hz, 1H), 2.70 (br s, 1H), 2.29 (dd, *J* = 14.8, 5.8 Hz, 1H), 2.11 (dd, *J* = 14.8, 7.9 Hz, 1H), 2.09—1.80 (m, 6H), 1.64 (m, 1H), 1.59 (d, *J* = 1.4 Hz, 3H), 1.61—1.50 (m, 3H), 1.42 (dd, *J* = 14.1, 3.8 Hz, 1H), 1.39—1.20 (m, 3H), 1.11 (m, 1H), 1.10 (d, *J* = 7.2 Hz, 3H), 1.04 (d, *J* = 6.9 Hz, 3H), 0.98 (m, 1H), 0.92 (d, *J* = 6.8 Hz, 3H), 0.87 (d, *J* = 6.8 Hz, 3H), 0.84 (s, 9H), 0.07 (s, 3H), 0.01 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.6, 138.9, 133.2, 130.1, 128.2 (2C), 127.5 (2C), 127.3, 97.8, 81.9, 76.1, 73.2, 72.9, 72.8, 66.9, 51.4, 44.7, 41.6, 40.9, 36.2, 35.8, 33.2, 32.7, 30.1, 25.9 (3C), 25.3, 25.0, 24.5, 22.3, 20.9, 19.5, 18.2, 16.0, 11.5, −4.1, −4.5; HRMS (ESI) calcd for $C_{39}H_{66}O_7SiNa$ $[(M + Na)^+]$ 697.4470, found 697.4482.

Data for **21-*epi*-4**: $[\alpha]_D^{28}$ +15.3 (*c* 1.54, $CHCl_3$); IR (film) 3481, 2952, 2928, 2856, 1740, 1455, 1254, 1076, 982, 837, 775 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.33—7.30 (m, 4H), 7.25 (m, 1H), 5.46 (dd, *J* = 7.2, 7.2 Hz, 1H), 4.48 (d, *J* = 12.0 Hz, 1H), 4.44 (d, *J* = 12.0 Hz, 1H), 4.08 (br s, 1H), 3.86 (ddd, *J* = 11.0, 3.8, 3.8 Hz, 1H), 3.70 (ddd, *J* = 12.1, 4.1, 2.1 Hz, 1H), 3.64 (s, 3H), 3.58 (dd, *J* = 8.9, 3.8 Hz, 1H), 3.49 (dd, *J* = 6.2, 4.1 Hz, 1H), 3.27 (dd, *J* = 8.9, 7.8 Hz, 1H), 2.37 (br s, 1H), 2.30 (dd, *J* = 14.8, 5.9 Hz, 1H), 2.11 (dd, *J* = 14.8, 8.2 Hz, 1H), 2.08—1.80 (m, 6H), 1.68—1.49 (m, 4H), 1.55 (s, 3H), 1.43—1.33 (m, 2H), 1.30—1.20 (m, 2H), 1.07 (d, *J* = 7.2 Hz, 3H), 1.02 (d, *J* = 6.8 Hz, 3H), 0.99 (m, 1H), 0.95 (m, 1H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.85 (d, *J* = 6.9 Hz, 3H), 0.84

(s, 9H), 0.04 (s, 3H), 0.00 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.6, 138.9, 132.5, 128.2 (2C), 127.5 (2C), 127.3, 125.7, 98.3, 77.2, 76.3, 73.1, 72.9, 72.7, 66.4, 51.3, 44.7, 41.6, 41.1, 36.4, 35.9, 33.4, 30.1, 29.3, 26.0 (3C), 25.1, 24.9, 24.6, 22.3, 20.9, 19.6, 18.2, 15.9, 13.6, −3.9, −4.5; HRMS (ESI) calcd for $C_{39}H_{66}O_7SiNa$ [(M + Na)$^+$] 697.4470, found 697.4476.



Conversion of 21-*epi*-4 to 4. To a solution of a 1.4:1 mixture of C9—C28 subunit **4** and **21-*epi*-4** (24.5 mg, 36.3 μmol) in CH_2Cl_2/*t*-BuOH (10:1, v/v, 0.99 mL) cooled to 0 °C was added Dess—Martin periodinane (76 mg, 0.18 mmol), and the resultant solution was stirred at room temperature for 1 h. The reaction was quenched with a 1:1 mixture of saturated aqueous $Na_2S_2O_3$ solution and saturated aqueous $NaHCO_3$ solution at 0 °C, and the resultant solution was stirred at room temperature for 15 min. The resultant mixture was extracted with diethyl ether, and the organic layer was washed with brine, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (7% EtOAc/hexanes) gave enone **S5** (24.7 mg, 100%) as a colorless oil: $[\alpha]_D^{26}$ +15.7 (*c* 1.15, $CHCl_3$); IR

(film) 2952, 2926, 2857, 1739, 1683, 1455, 1255, 1074, 1025, 836 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.35—7.27 (m, 4H), 7.25 (m, 1H), 6.57 (ddq, *J* = 7.2, 7.2, 1.0 Hz, 1H), 4.97 (dd, *J* = 11.0, 3.8 Hz, 1H), 4.47 (d, *J* = 12.0 Hz, 1H), 4.43 (d, *J* = 12.0 Hz, 1H), 3.64 (s, 3H), 3.61 (ddd, *J* = 11.7, 3.8, 2.4 Hz, 1H), 3.56 (dd, *J* = 8.9, 3.4 Hz, 1H), 3.47 (dd, *J* = 6.5, 3.8 Hz, 1H), 3.27 (dd, *J* = 8.9, 7.9 Hz, 1H), 2.29 (dd, *J* = 14.8, 5.9 Hz, 1H), 2.21 (m, 1H), 2.14 (dd, *J* = 14.8, 8.3 Hz, 1H), 2.03 (ddd, *J* = 14.5, 6.9, 3.4 Hz, 1H), 2.00—1.91 (m, 4H), 1.81 (ddd, *J* = 13.1, 3.8, 1.4 Hz, 1H), 1.77 (s, 3H), 1.71 (ddd, *J* = 13.1, 11.0, 5.5 Hz, 1H), 1.64 (dd, *J* = 13.7, 5.9 Hz, 1H), 1.57—1.49 (m, 2H), 1.48—1.39 (m, 2H), 1.31 (dddd, *J* = 13.4, 10.0, 7.6, 5.9 Hz, 1H), 1.18 (d, *J* = 6.9 Hz, 3H), 1.02 (d, *J* = 6.9 Hz, 3H), 1.00—0.88 (m, 2H), 0.96 (d, *J* = 6.8 Hz, 3H), 0.84 (d, *J* = 6.5 Hz, 3H), 0.82 (s, 9H), −0.03 (s, 3H), −0.06 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 199.9, 173.2, 142.2, 138.9, 135.4, 128.2 (2C), 127.5 (2C), 127.3, 98.4, 76.2, 73.1, 73.0, 72.9, 67.4, 51.4, 44.3, 41.3, 40.7, 36.0, 35.42, 33.36, 33.3, 30.2, 26.5, 25.9 (3C), 25.00, 24.95, 22.2, 20.7, 19.6, 18.1, 15.8, 11.7, −4.1, −4.6; HRMS (ESI) calcd for $C_{39}H_{64}O_7SiNa$ [$(M + Na)^+$] 695.4314, found 695.4331.

To a solution of enone **S5** (7.0 mg, 10 μmol) in THF (0.5 mL) cooled to −78 °C was added dropwise L-selectride (1.0 M solution in THF, 0.04 mL, 0.04 mmol), and the resultant solution was stirred at −78 °C for 1 h. The reaction was quenched with saturated aqueous $NaHCO_3$ solution, and the resultant mixture was allowed to warm to 0 °C and treated with 30% aqueous H_2O_2 solution (0.2 mL). After being stirred at room temperature for 1 h, the resultant mixture was extracted with EtOAc. The organic layer was washed successively with H_2O, saturated aqueous $Na_2S_2O_3$ solution, and brine. The organic layer was dried over Na_2SO_4, filtered, and concentrated under reduced pressure. Purification of the residue by flash chromatography on silica gel (5 to 10%

EtOAc/hexanes) gave C9—C28 subunit **4** (5.8 mg, 82%, dr > 20:1 by 600 MHz ^{1}H NMR analysis) as a colorless oil.

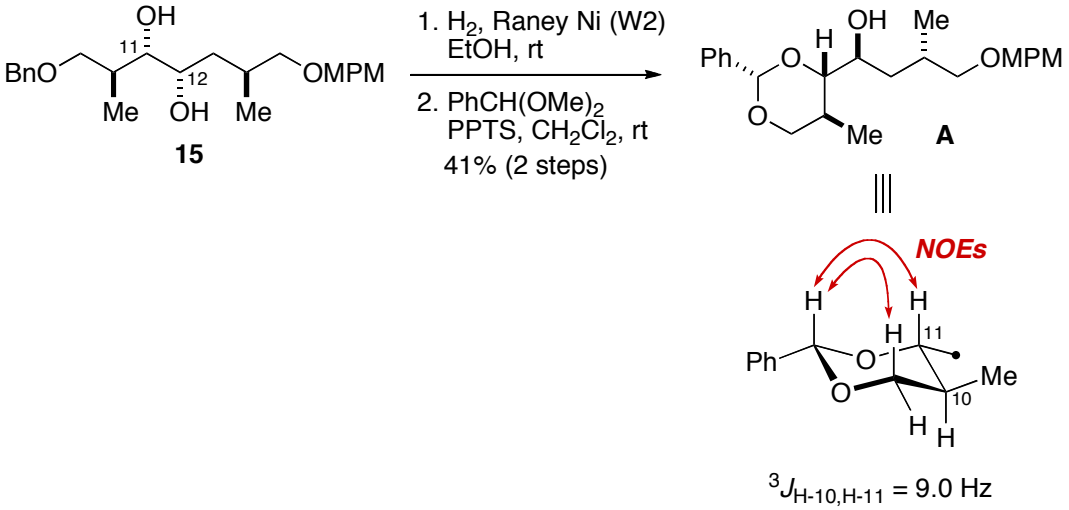


Figure S1. Stereochemical confirmation of compound **15** by conformational analysis on compound **A**.

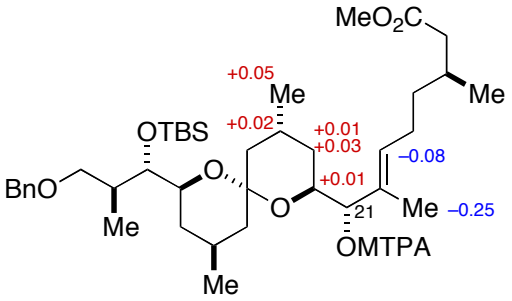
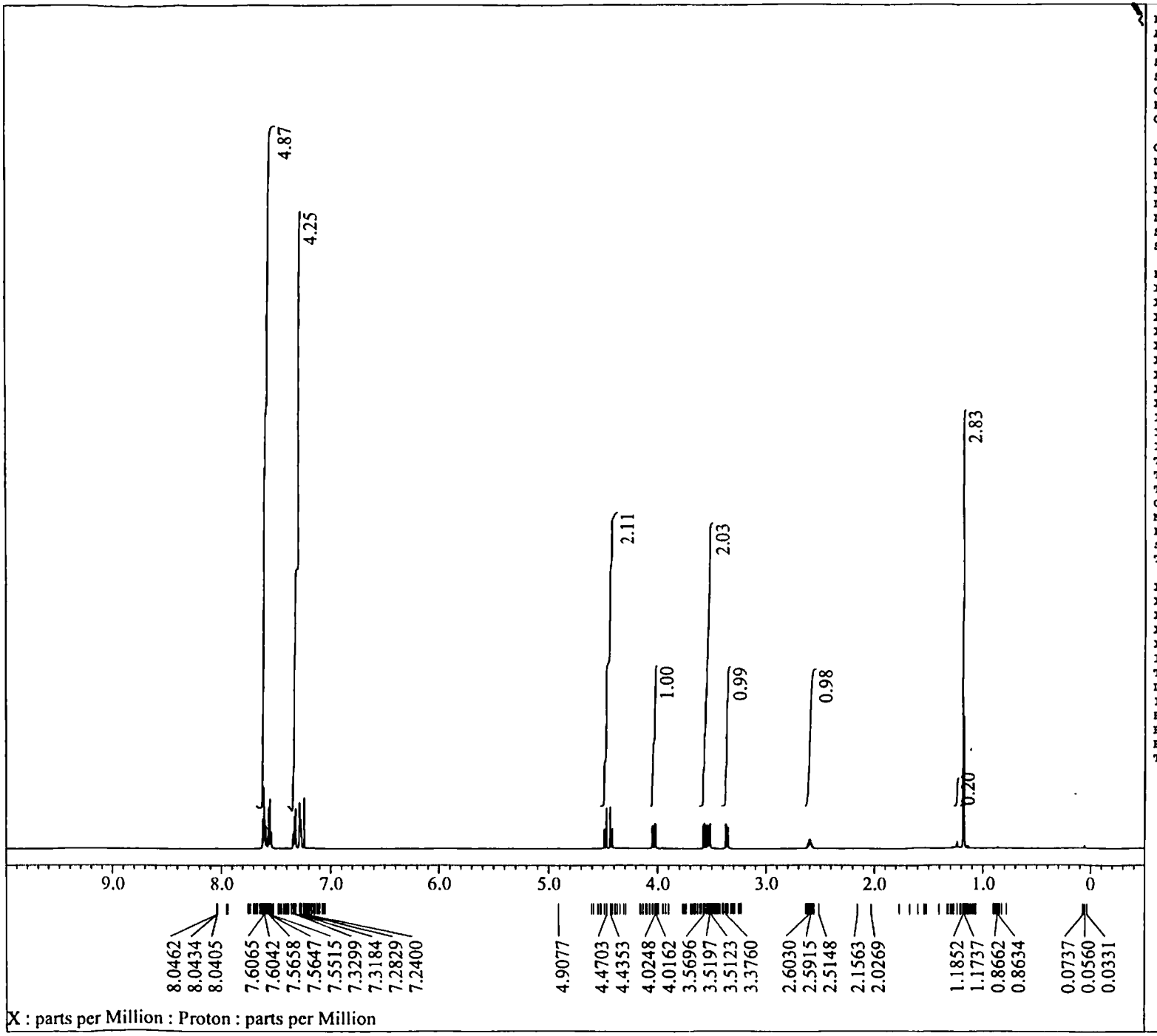
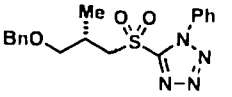


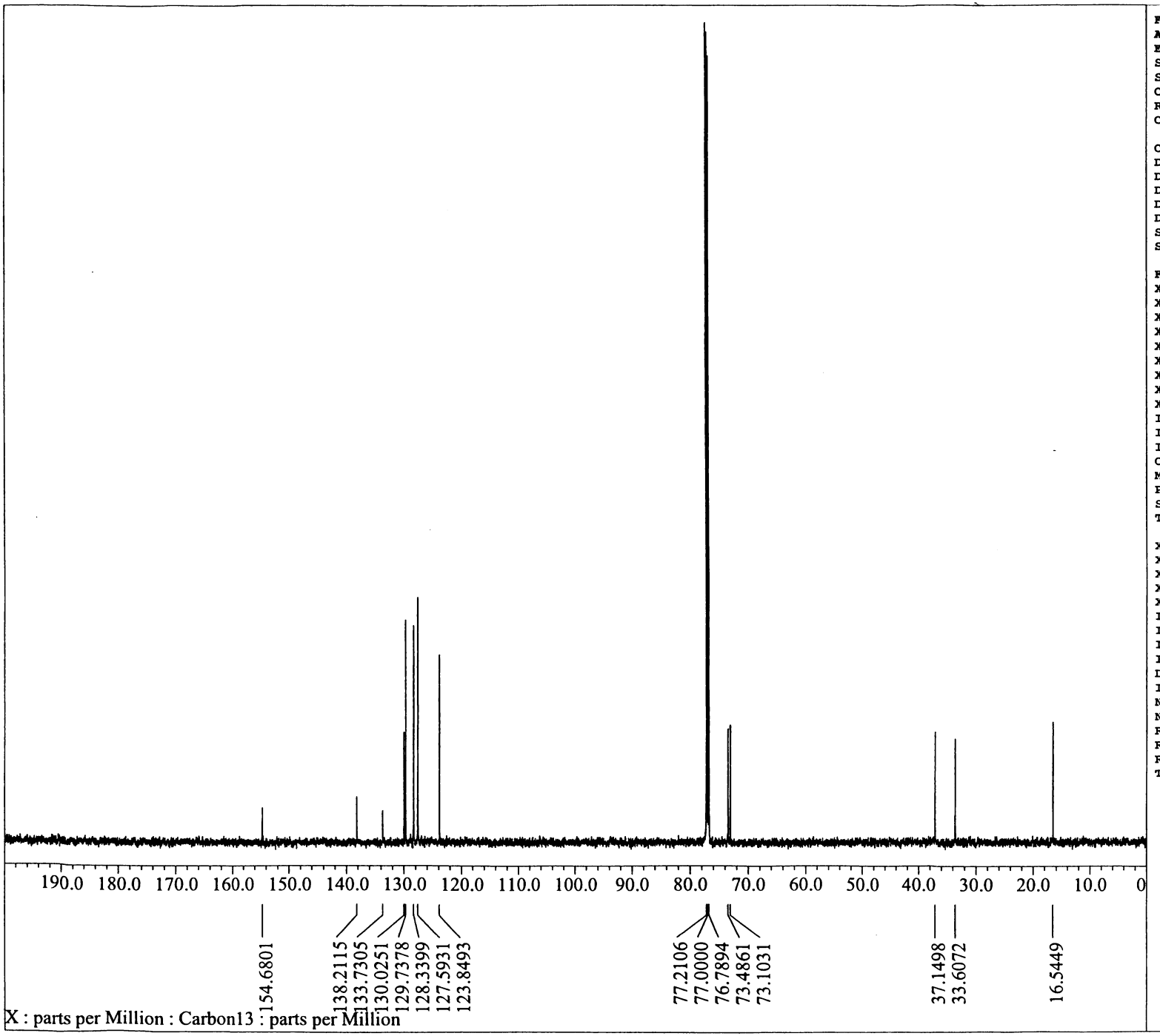
Figure S2. Assignment of the absolute configuration of the C21 stereogenic center of compound **4** by modified Mosher method.



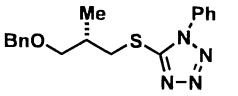
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-II-115
Solvent	= CHLOROFORM-D
Creation_Time	= 16-SEP-2010 13:59:42
Revision_Time	= 16-SEP-2010 14:01:52
Current_Time	= 16-SEP-2010 14:02:32
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 42
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.2[dC]



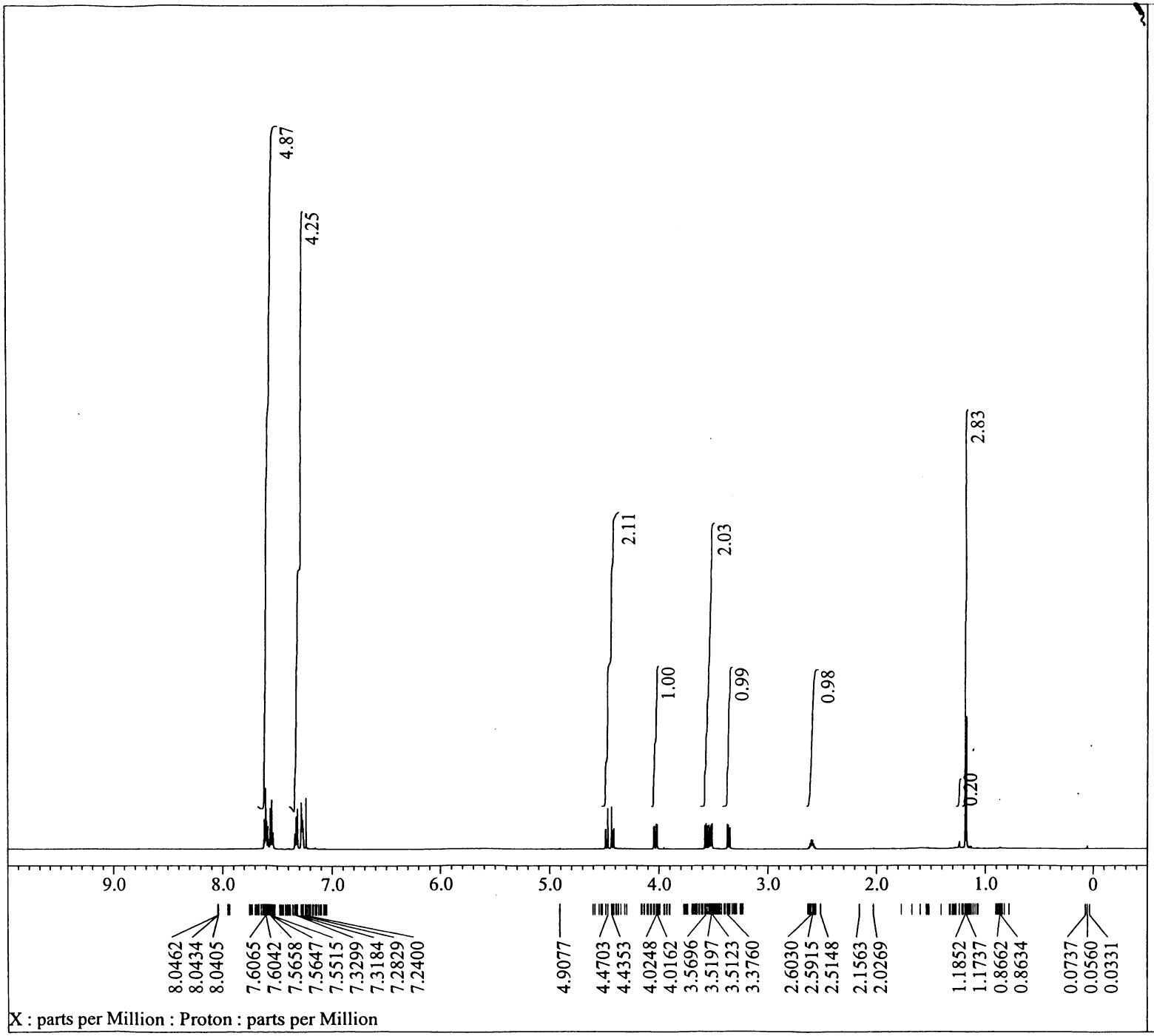
12 (600 MHz, $CDCl_3$)



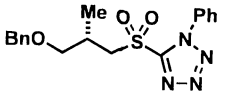

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-II-112
Solvent           = CHLOROFORM-D
Creation_Time     = 11-SEP-2010 15:24:55
Revision_Time     = 11-SEP-2010 15:37:31
Current_Time      = 11-SEP-2010 15:38:08

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 256
Total_Scans       = 256

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.3[dC]
```



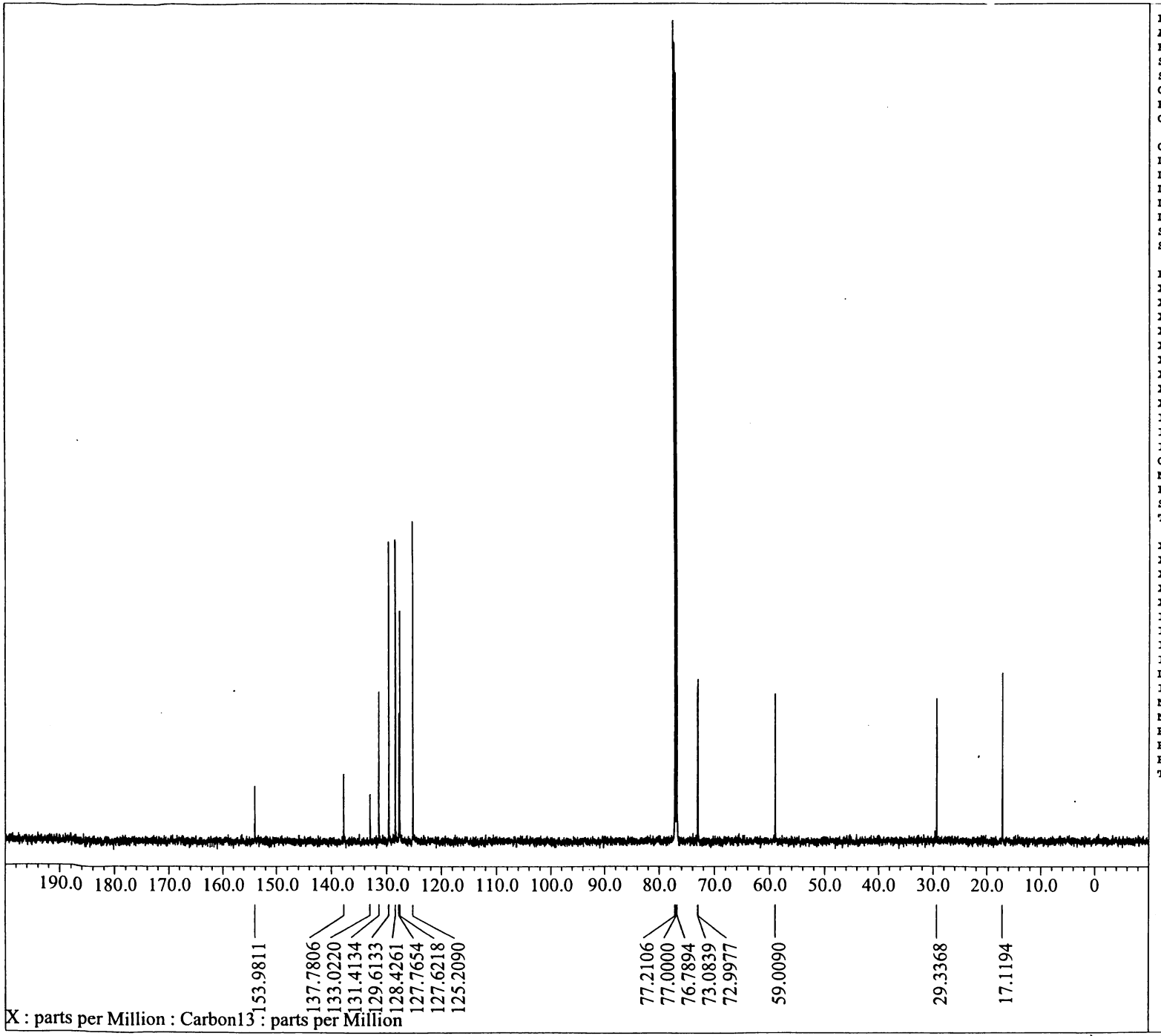
S1 (150 MHz, $CDCl_3$)



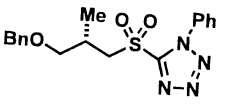
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-II-115
Solvent	= CHLOROFORM-D
Creation_Time	= 16-SEP-2010 13:59:42
Revision_Time	= 16-SEP-2010 14:01:52
Current_Time	= 16-SEP-2010 14:02:32
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 42
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.2[dC]



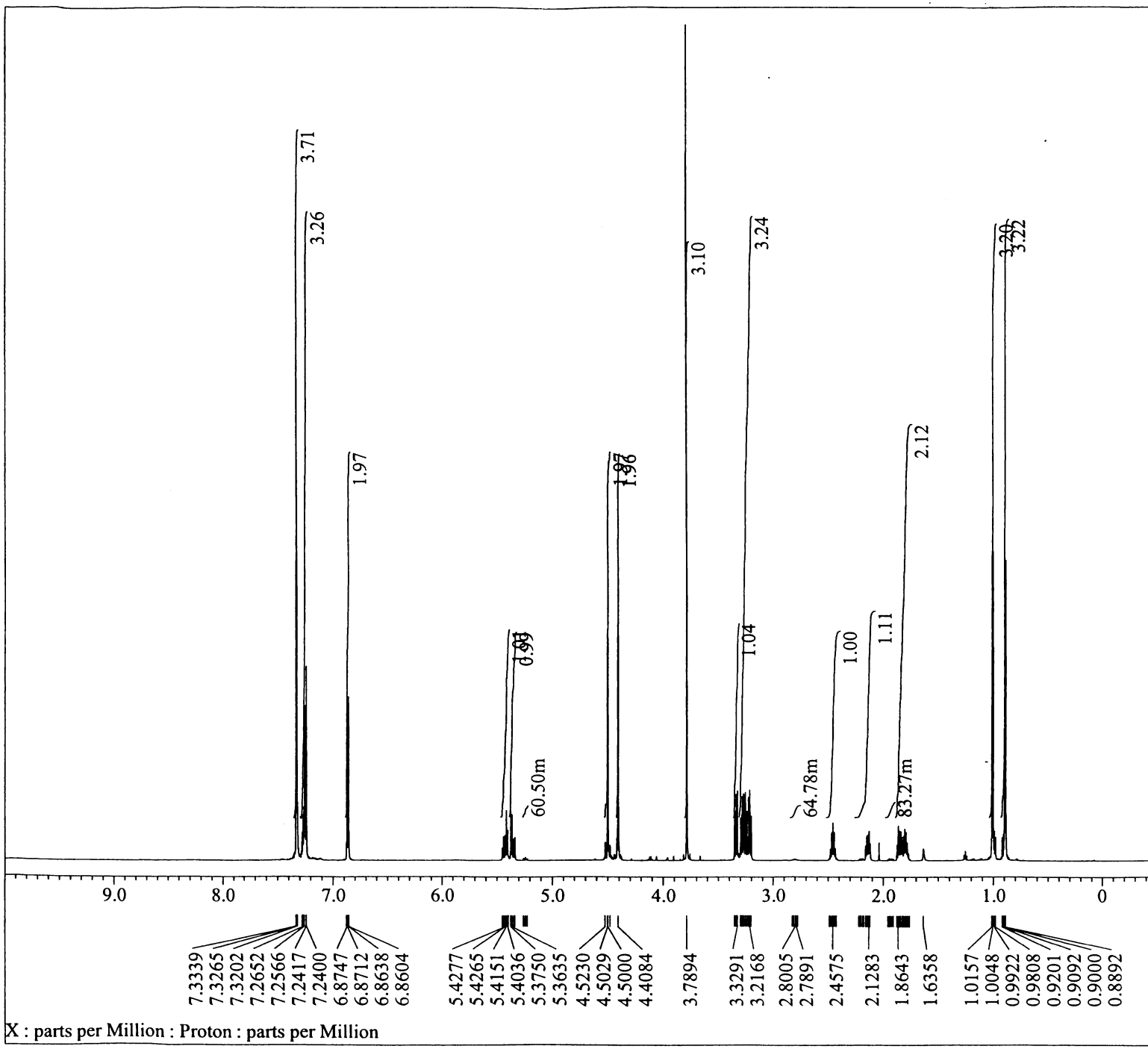
12 (600 MHz, $CDCl_3$)



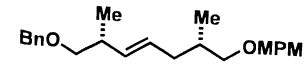

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-II-115
Solvent           = CHLOROFORM-D
Creation_Time     = 16-SEP-2010 14:00:54
Revision_Time     = 16-SEP-2010 14:11:51
Current_Time      = 16-SEP-2010 14:12:52

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 219
Total_Scans       = 219

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 50
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.1[dC]
```



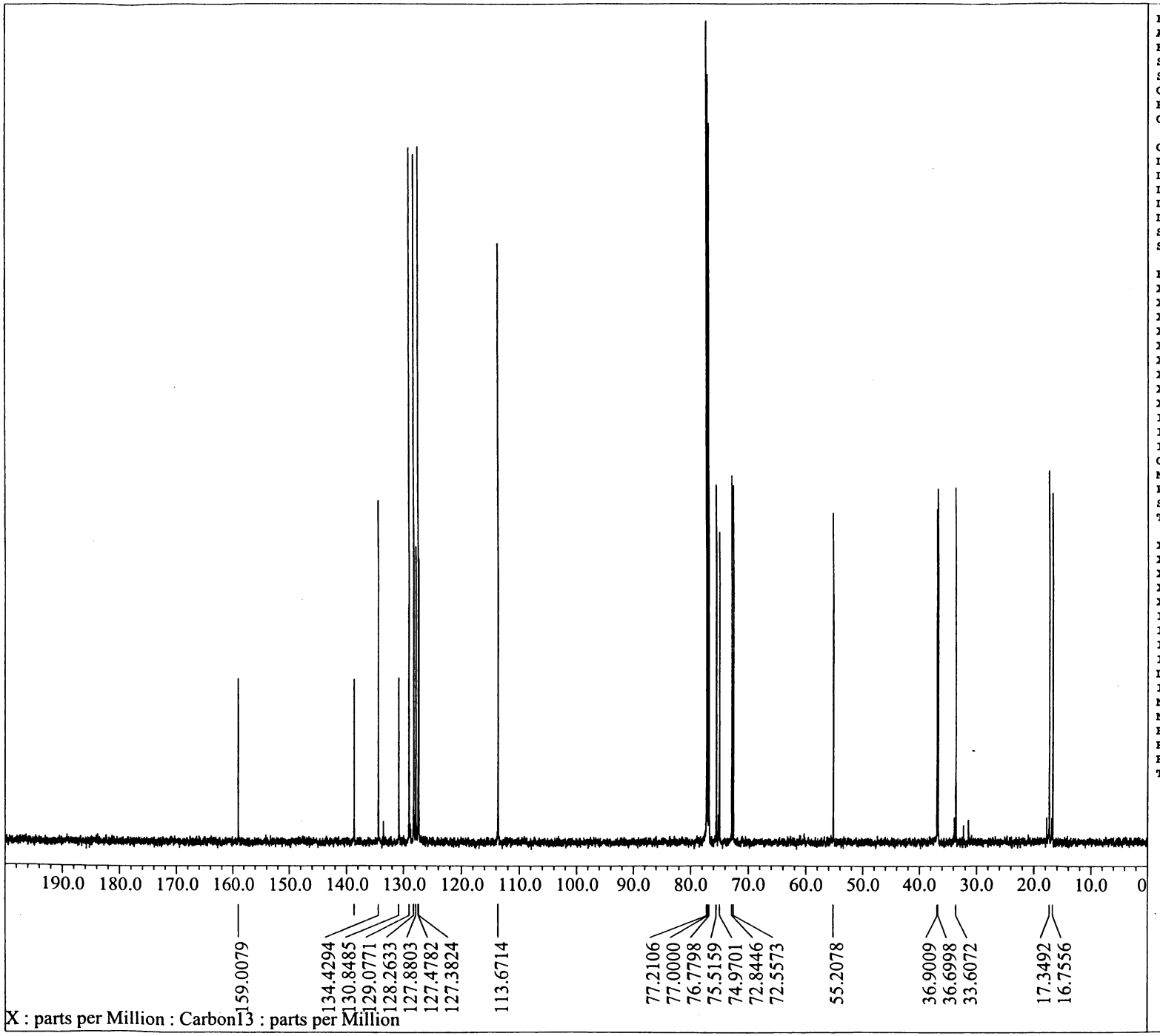
12 (150 MHz, $CDCl_3$)



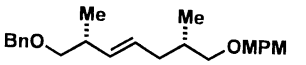

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SN-II-167-1
Solvent           = CHLOROFORM-D
Creation_Time     = 15-SEP-2010 13:18:44
Revision_Time     = 15-SEP-2010 13:26:52
Current_Time      = 15-SEP-2010 13:27:20

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 5[us]
Scans             = 8
Total_Scans       = 8

X_90_Width        = 12.4[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Recvr_Gain        = 30
Relaxation_Delay  = 1[s]
Repetition_Time   = 3.9097984[s]
Temp_Get          = 22.4[dC]
```



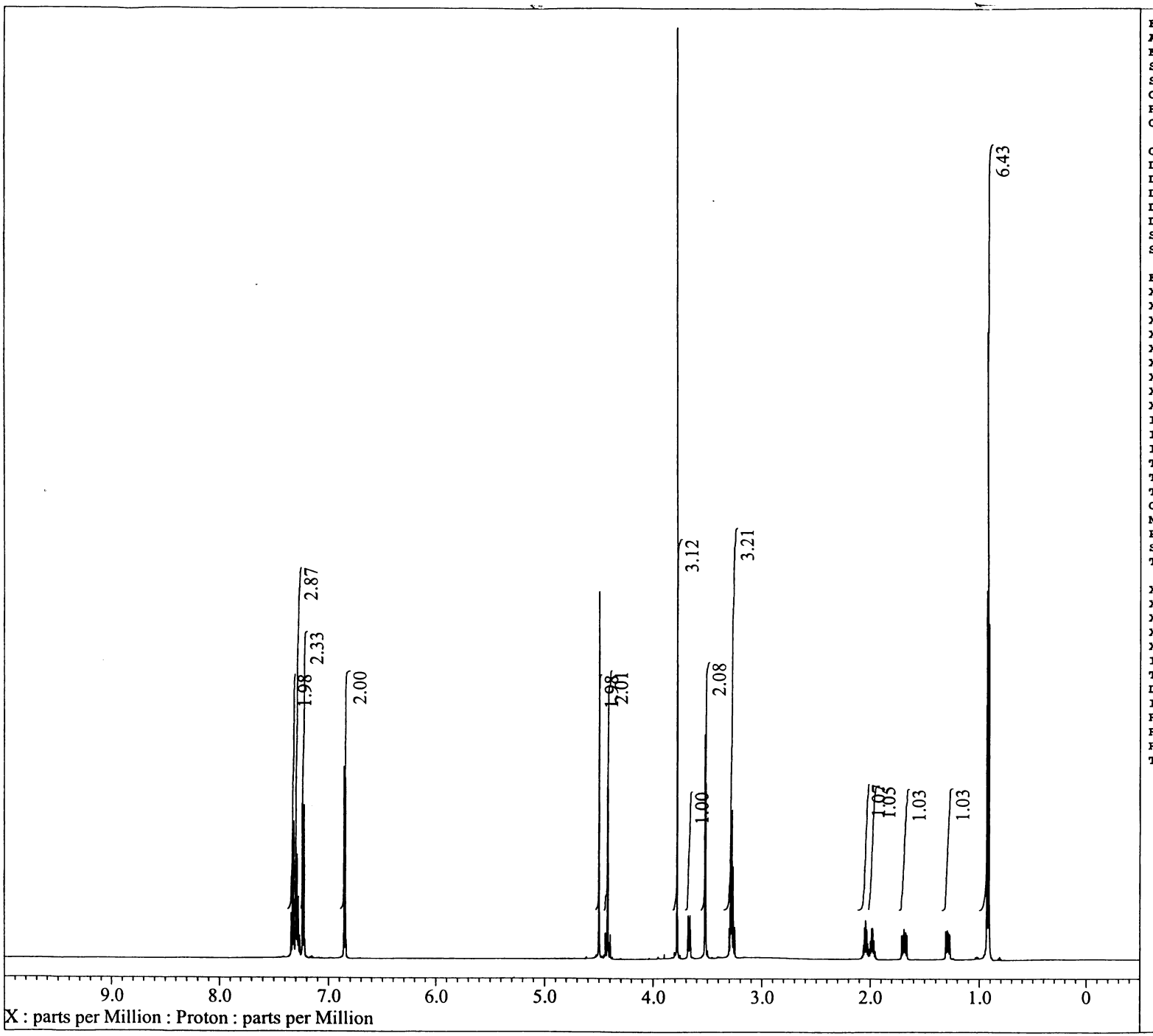
14 (600 MHz, $CDCl_3$)



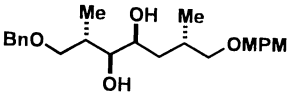

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-II-167-1
Solvent           = CHLOROFORM-D
Creation_Time     = 15-SEP-2010 13:21:02
Revision_Time     = 15-SEP-2010 13:34:56
Current_Time      = 15-SEP-2010 13:35:11

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 256
Total_Scans       = 256

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 58
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.1[dC]
```



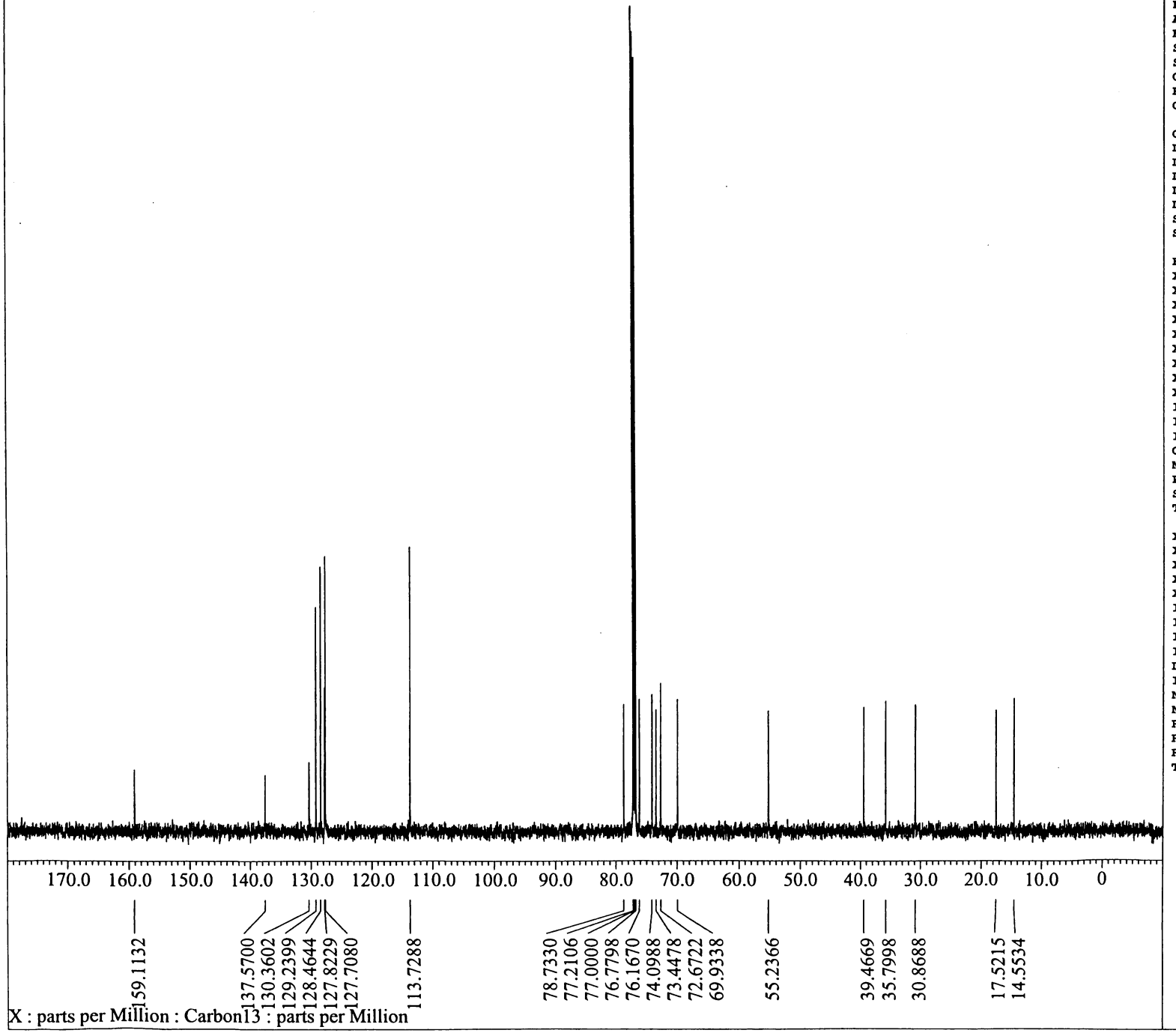
14 (150 MHz, $CDCl_3$)



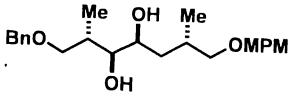

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SN-II-168-2-1
Solvent           = CHLOROFORM-D
Creation_Time     = 15-SEP-2010 15:38:48
Revision_Time     = 15-SEP-2010 15:41:22
Current_Time      = 15-SEP-2010 15:41:38

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 5[us]
Scans             = 8
Total_Scans       = 8

X_90_Width        = 12.4[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Recvr_Gain        = 40
Relaxation_Delay  = 1[s]
Repetition_Time   = 3.9097984[s]
Temp_Get          = 22.3[dC]
```



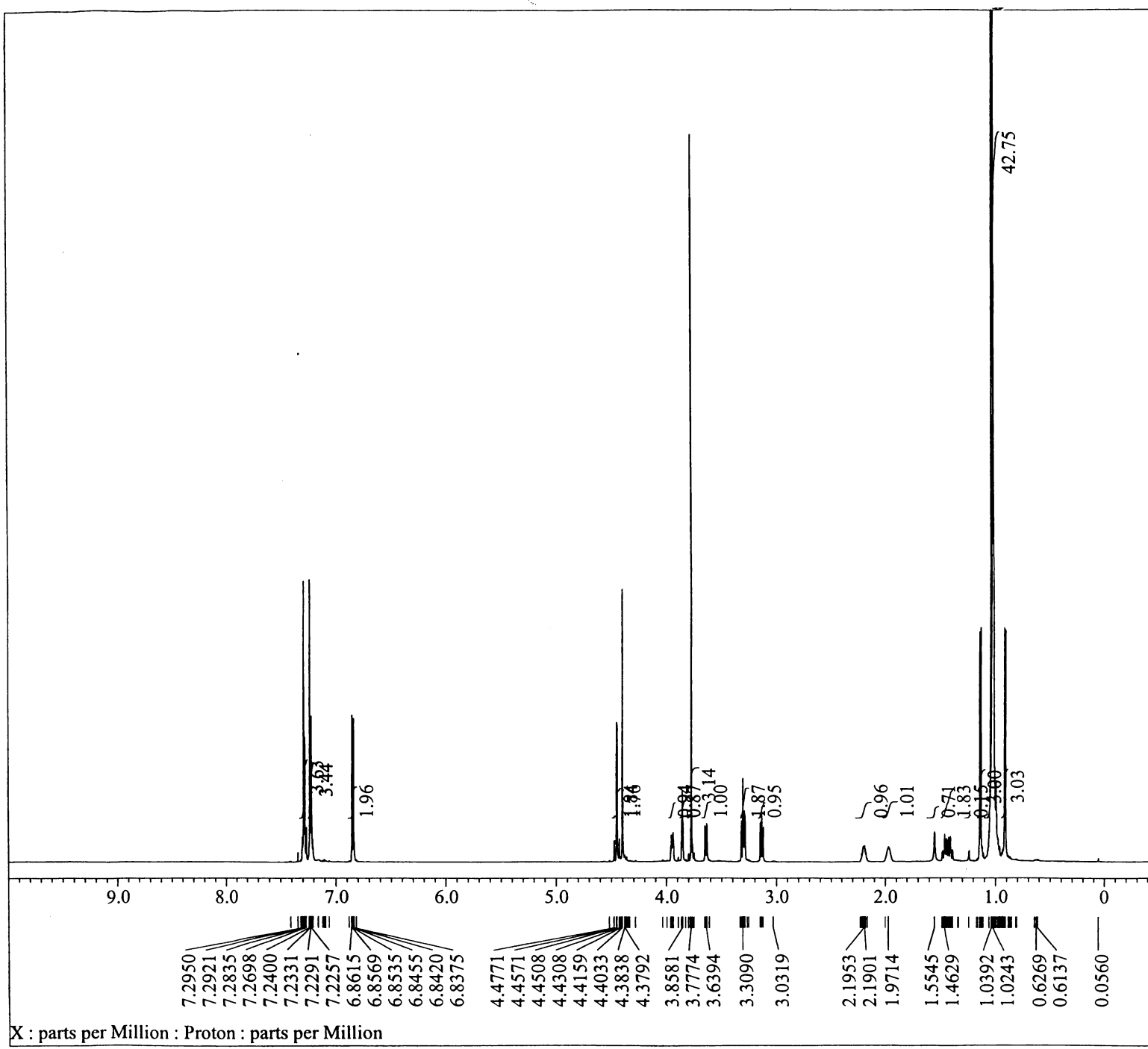
15 (600 MHz, $CDCl_3$)



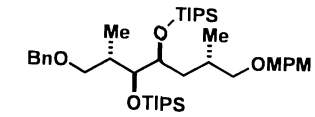

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-II-168-2-1
Solvent           = CHLOROFORM-D
Creation_Time     = 15-SEP-2010 15:41:04
Revision_Time     = 15-SEP-2010 15:45:50
Current_Time      = 15-SEP-2010 15:46:08

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 80
Total_Scans       = 80

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.1[dC]
```



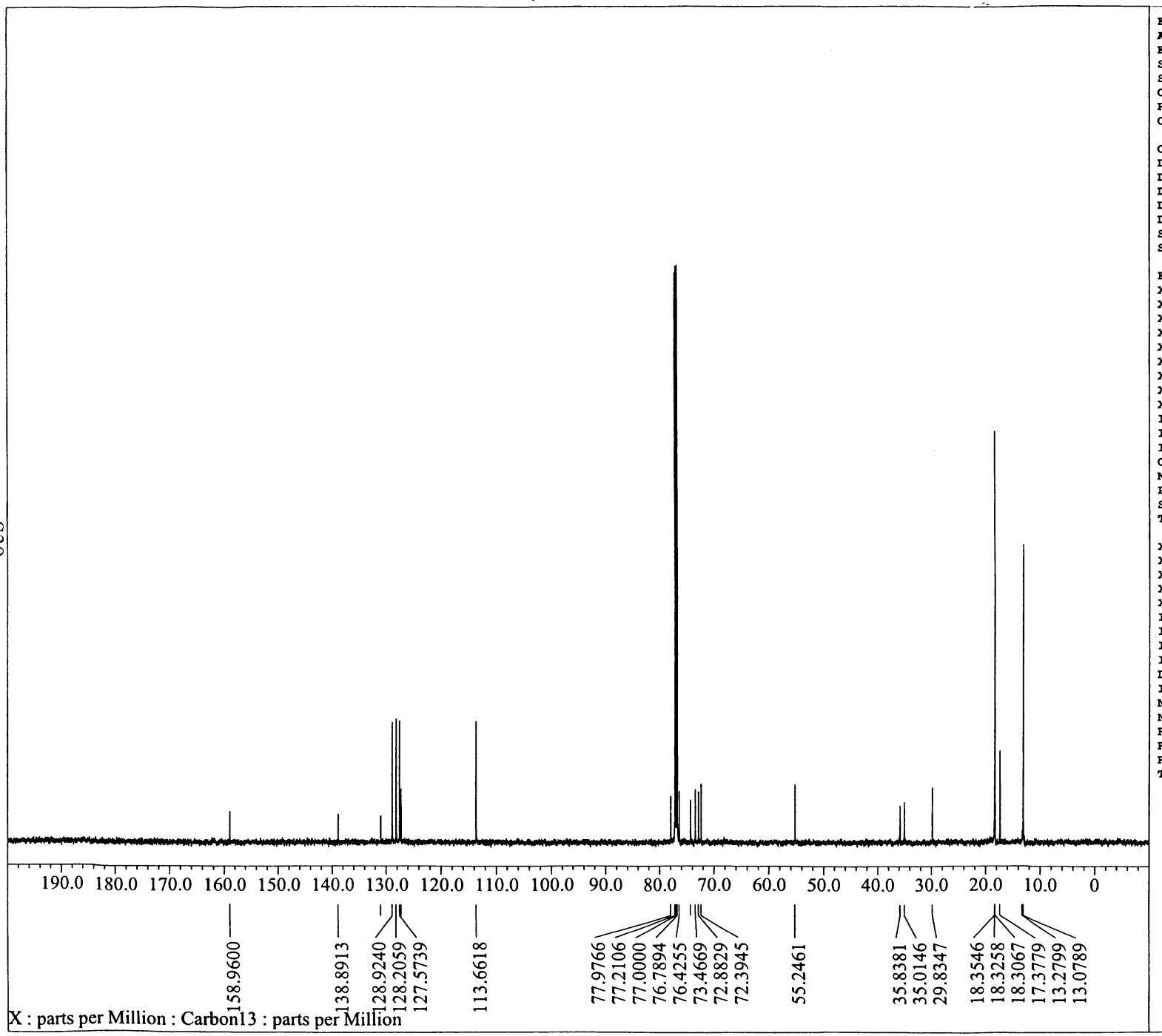
15 (150 MHz, $CDCl_3$)



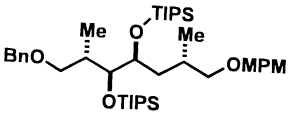

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SN-III-45
Solvent           = CHLOROFORM-D
Creation_Time     = 14-SEP-2010 15:36:45
Revision_Time     = 14-SEP-2010 15:40:00
Current_Time      = 14-SEP-2010 15:40:19

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 5[us]
Scans             = 8
Total_Scans       = 8

X_90_Width        = 12.4[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Recvr_Gain        = 36
Relaxation_Delay  = 1[s]
Repetition_Time   = 3.9097984[s]
Temp_Get          = 22.3[dC]
```



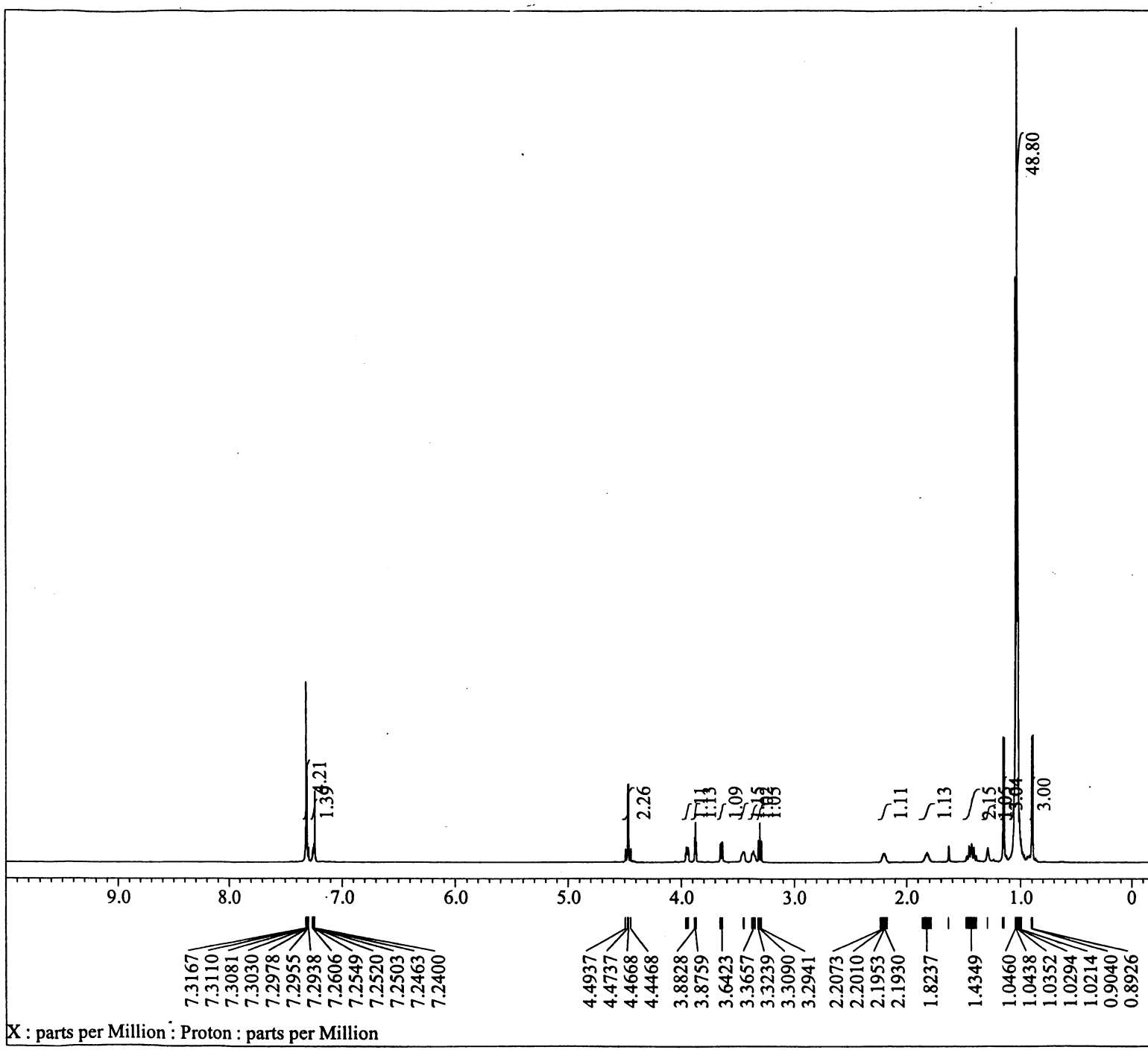
S2 (600 MHz, $CDCl_3$)



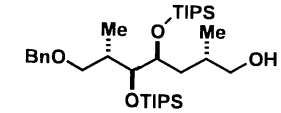

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-45
Solvent           = CHLOROFORM-D
Creation_Time     = 14-SEP-2010 15:37:59
Revision_Time     = 14-SEP-2010 15:49:40
Current_Time      = 14-SEP-2010 15:50:15

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 238
Total_Scans       = 238

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 50
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23[dC]
```



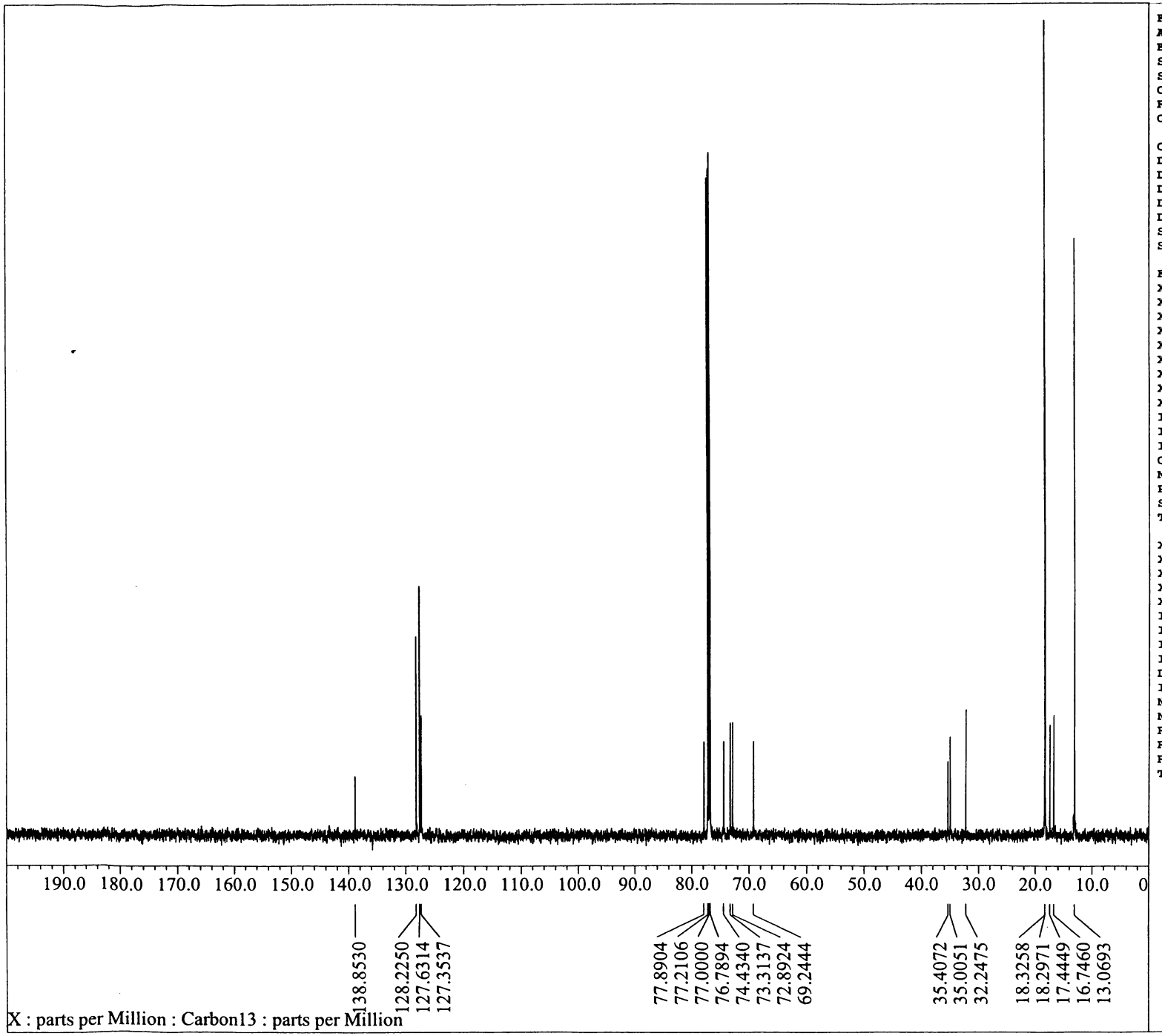
S2 (150 MHz, $CDCl_3$)



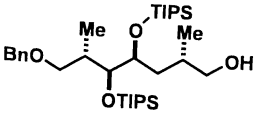
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-46
Solvent	= CHLOROFORM-D
Creation_Time	= 26-MAY-2010 17:46:46
Revision_Time	= 26-MAY-2010 17:55:15
Current_Time	= 26-MAY-2010 17:55:49
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 30
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 21.6[dC]



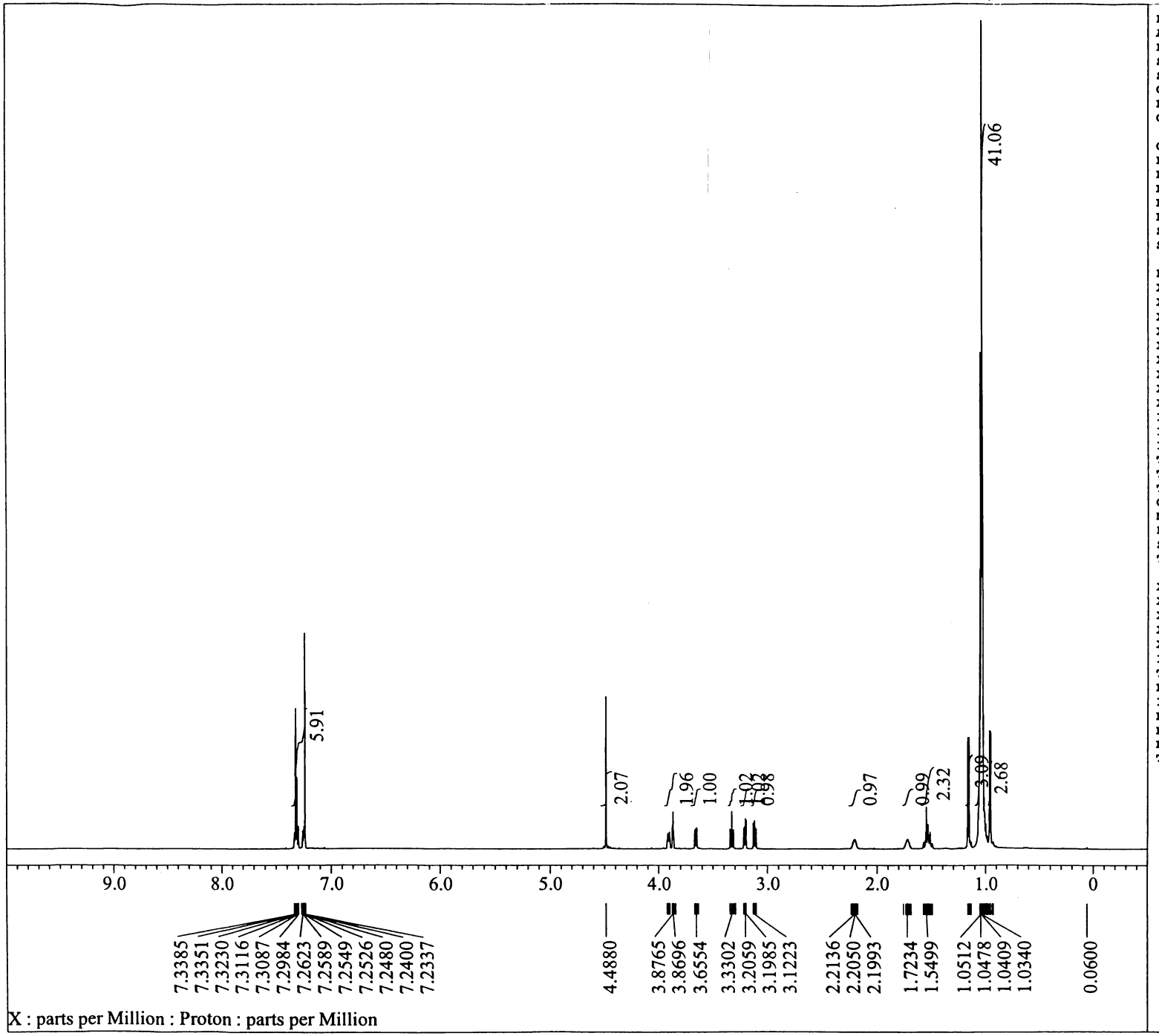
16 (600 MHz, $CDCl_3$)



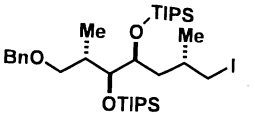

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-46
Solvent           = CHLOROFORM-D
Creation_Time     = 14-SEP-2010 16:36:31
Revision_Time     = 14-SEP-2010 16:44:46
Current_Time      = 14-SEP-2010 16:45:04

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 85
Total_Scans       = 85

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.1[dC]
```



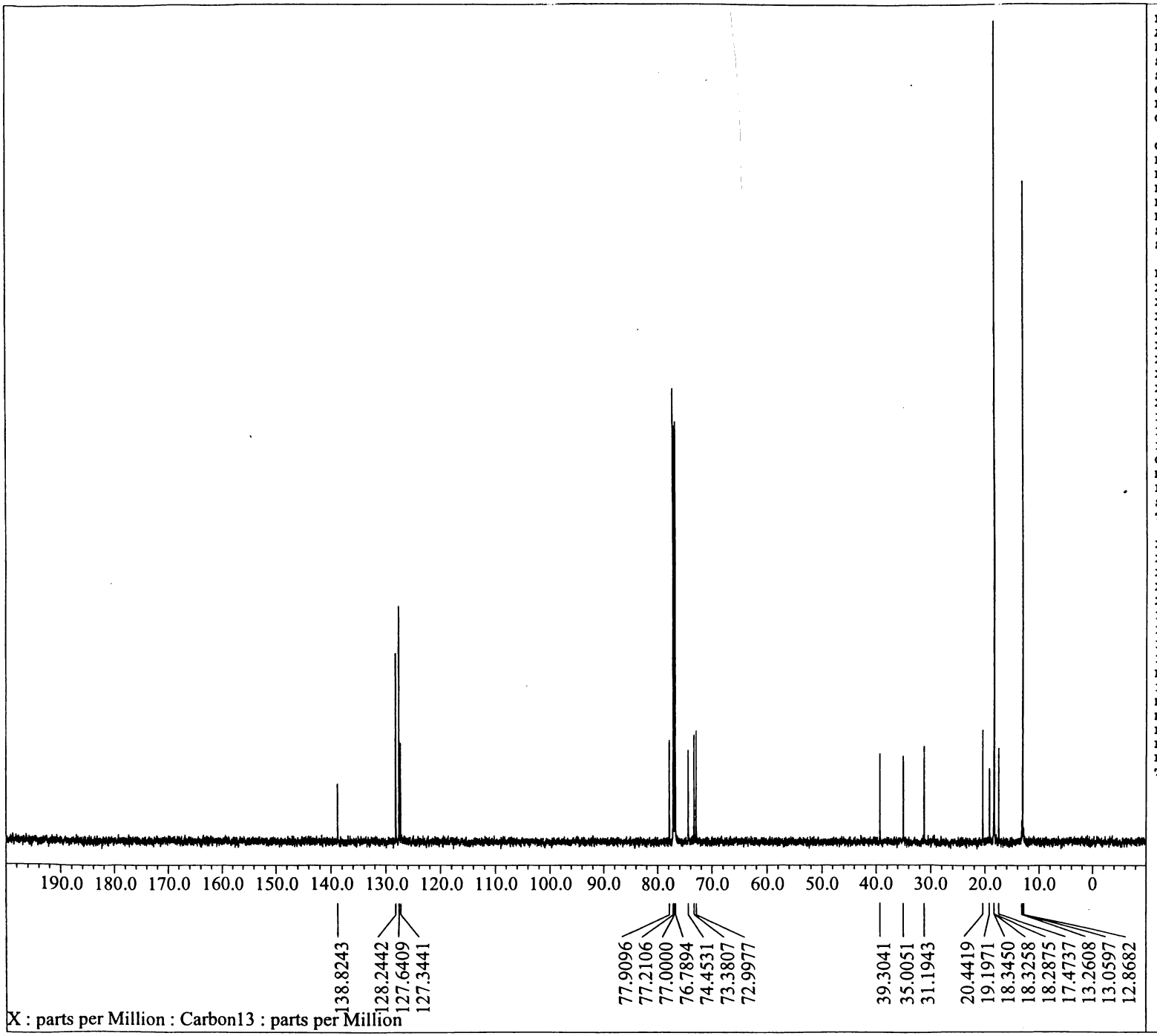
16 (150 MHz, $CDCl_3$)



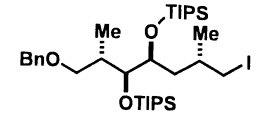
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-63
Solvent	= CHLOROFORM-D
Creation_Time	= 12-JUN-2010 22:08:15
Revision_Time	= 12-JUN-2010 22:14:46
Current_Time	= 12-JUN-2010 22:15:15
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 28
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.4[dC]



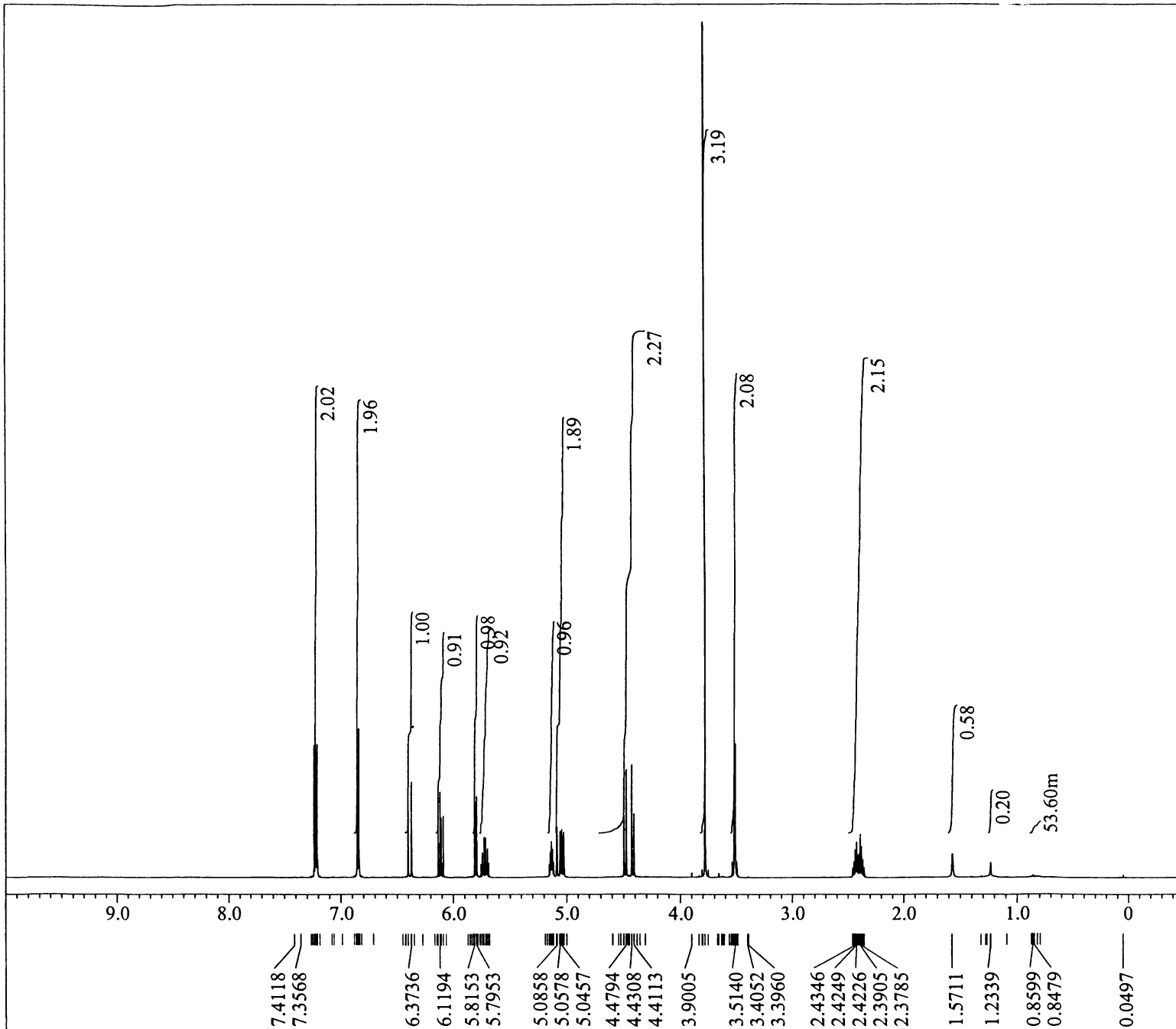
9 (600 MHz, $CDCl_3$)



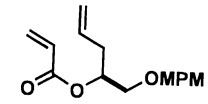
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= SN-III-63 2
Solvent	= CHLOROFORM-D
Creation_Time	= 12-JUN-2010 22:14:07
Revision_Time	= 12-JUN-2010 22:18:50
Current_Time	= 12-JUN-2010 22:21:24
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 0.69206016[s]
X_Domain	= 13C
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 20[us]
Scans	= 64
Total_Scans	= 64
X_90_Width	= 8.4[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 6.4[dB]
X_Pulse	= 2.8[us]
Irr_Atn_Dec	= 18[dB]
Irr_Atn_Noe	= 18[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 76[us]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Recvr_Gain	= 56
Relaxation_Delay	= 2[s]
Repetition_Time	= 2.69206016[s]
Temp_Get	= 23[dC]



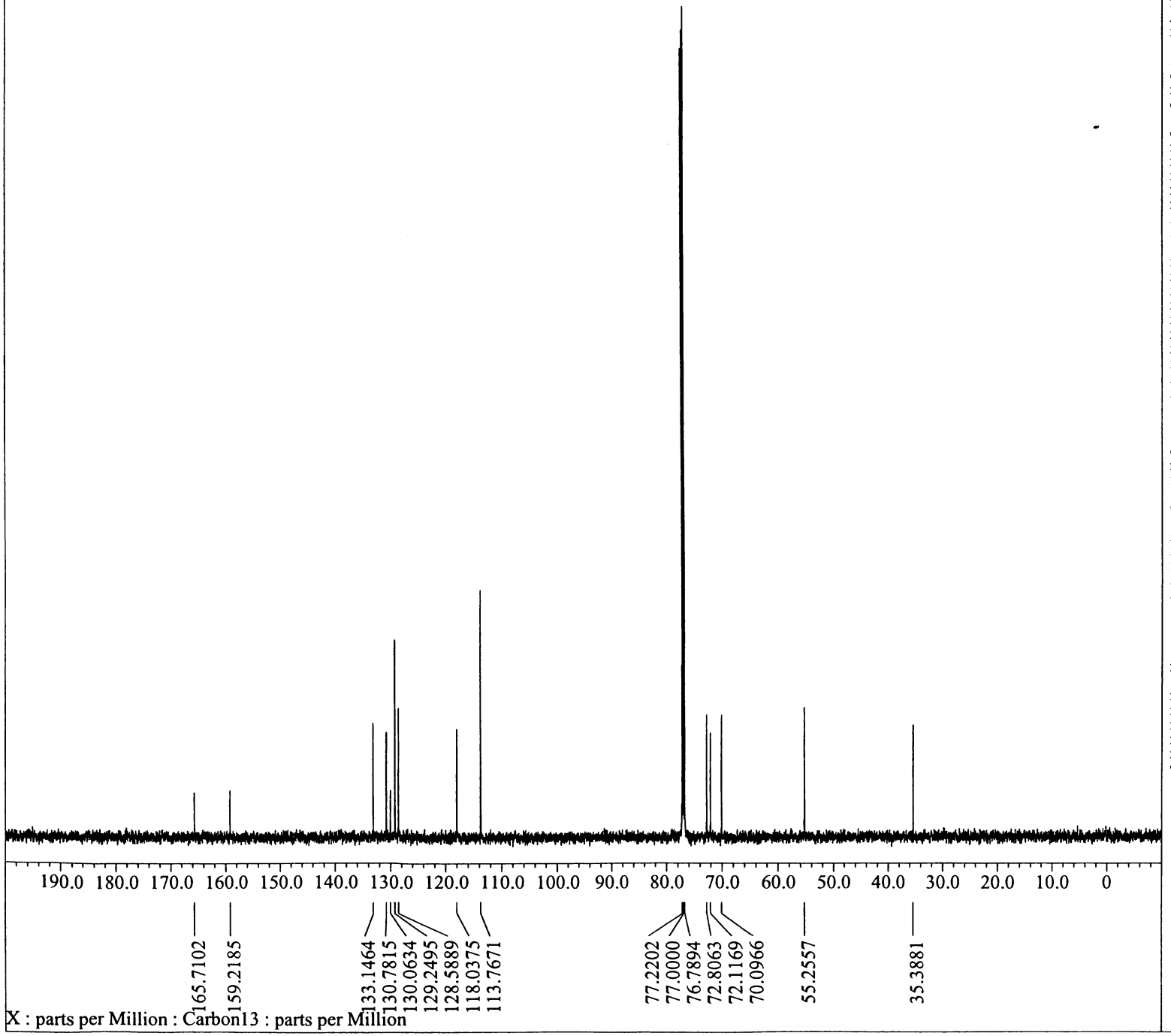
9 (150 MHz, $CDCl_3$)



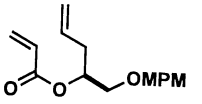
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-198
Solvent	= CHLOROFORM-D
Creation_Time	= 15-SEP-2010 18:45:17
Revision_Time	= 15-SEP-2010 18:48:56
Current_Time	= 15-SEP-2010 18:49:14
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 44
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.5[dC]



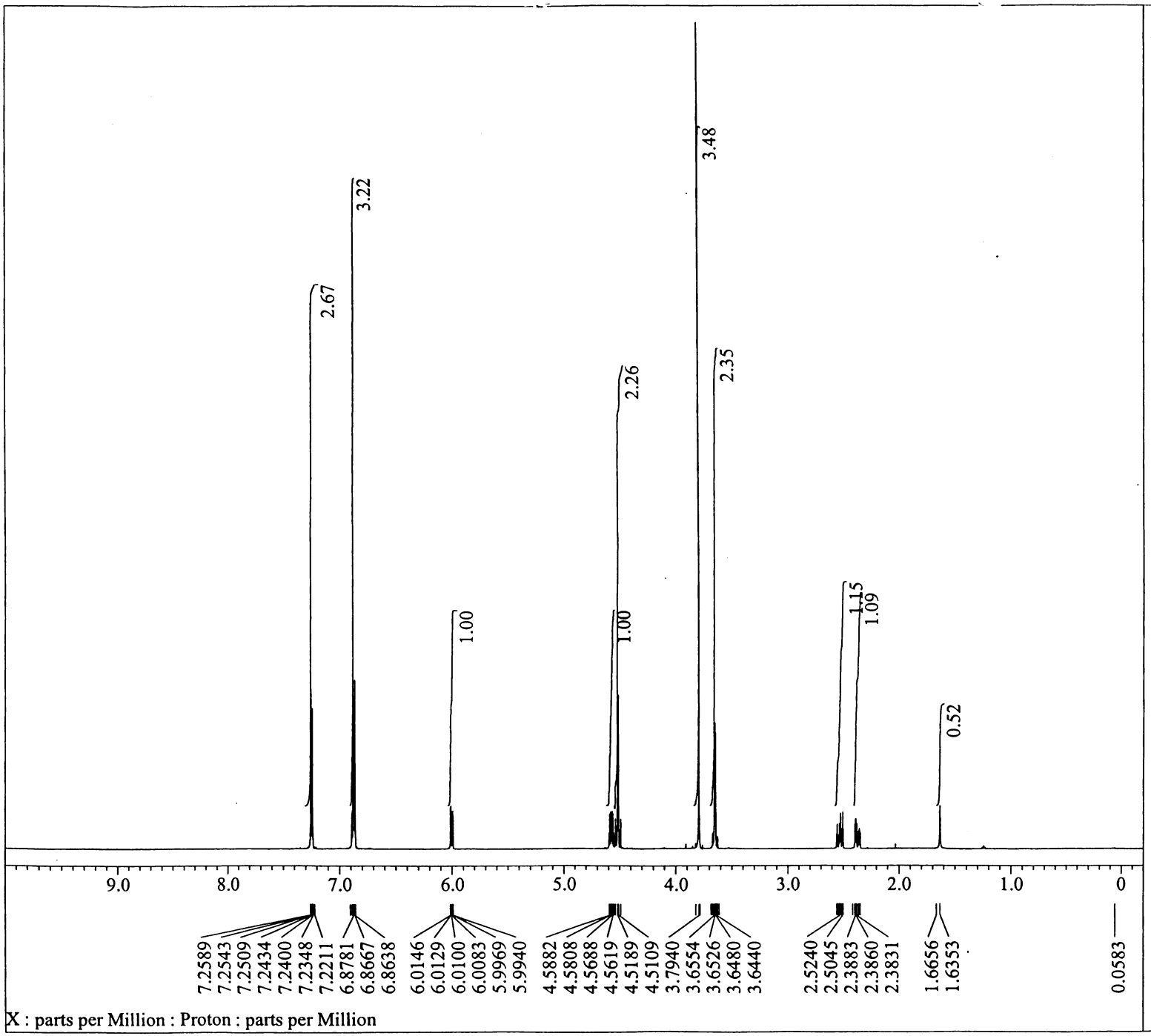
18 (600 MHz, $CDCl_3$)



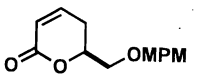

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-198
Solvent           = CHLOROFORM-D
Creation_Time     = 15-SEP-2010 18:46:29
Revision_Time     = 15-SEP-2010 18:53:40
Current_Time      = 15-SEP-2010 18:54:07

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 136
Total_Scans       = 136

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 50
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23[dC]
```



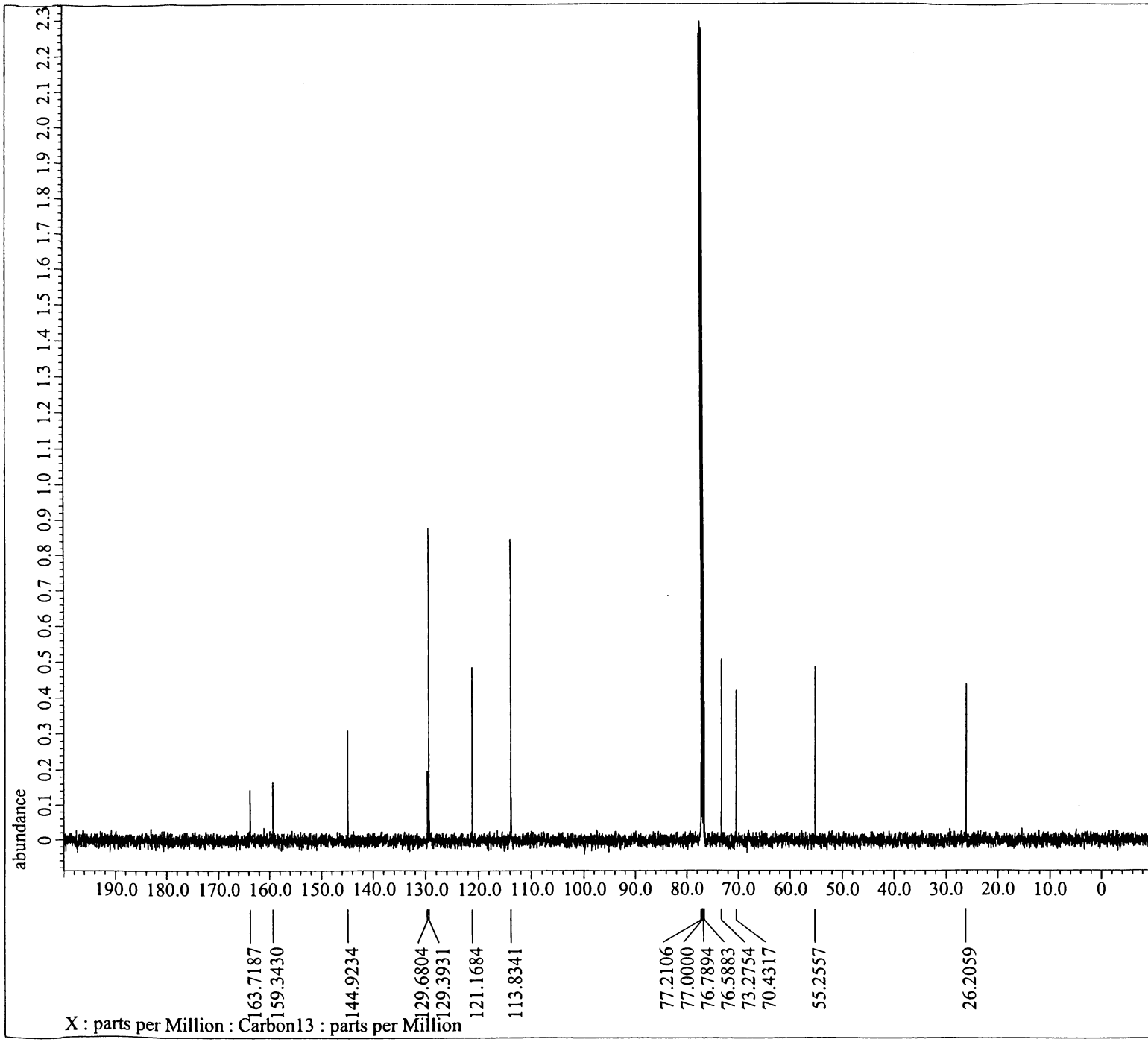
18 (150 MHz, $CDCl_3$)



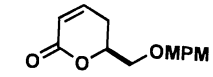

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SN-III-48
Solvent           = CHLOROFORM-D
Creation_Time     =  4-JUN-2010 20:54:25
Revision_Time     =  4-JUN-2010 20:57:55
Current_Time      =  4-JUN-2010 20:58:26

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 5[us]
Scans             = 8
Total_Scans       = 8

X_90_Width        = 12.4[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Recvr_Gain        = 42
Relaxation_Delay  = 1[s]
Repetition_Time   = 3.9097984[s]
Temp_Get          = 21.9[dC]
```



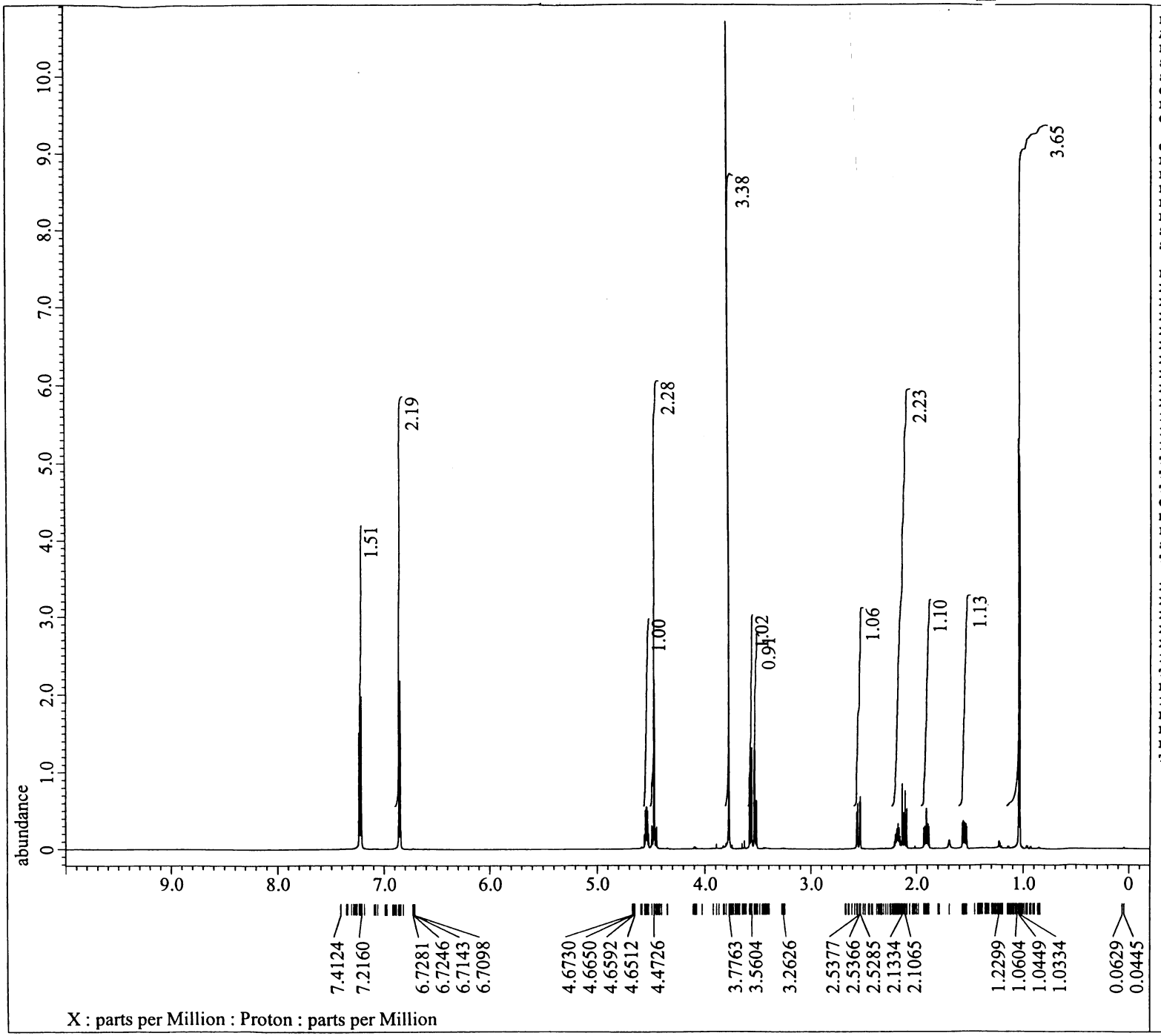
19 (600 MHz, $CDCl_3$)



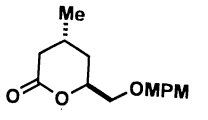

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-48
Solvent           = CHLOROFORM-D
Creation_Time     = 17-SEP-2010 11:46:40
Revision_Time     = 17-SEP-2010 11:51:17
Current_Time      = 17-SEP-2010 11:52:24

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 77
Total_Scans       = 77

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.1[dC]
```



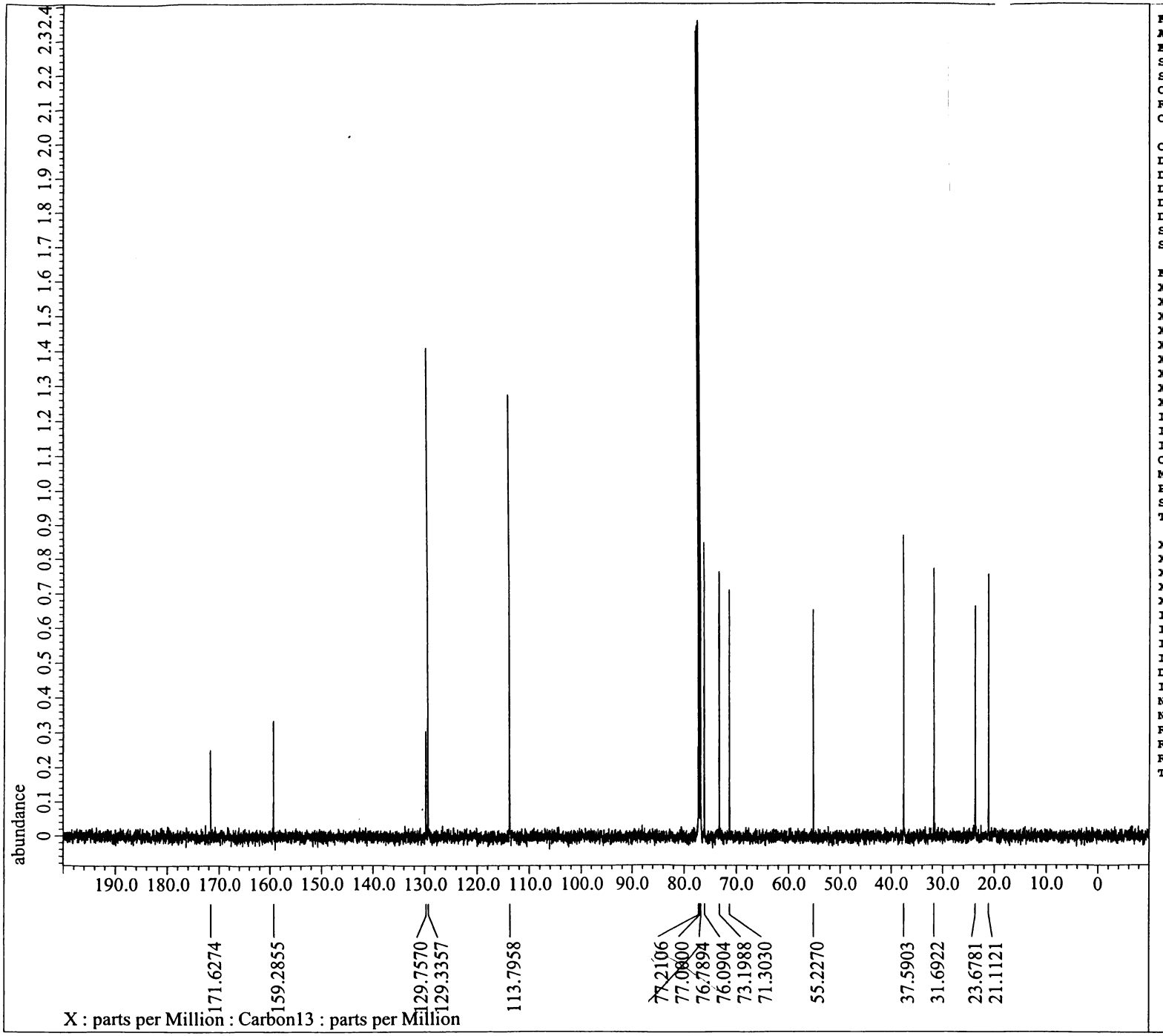
19 (150 MHz, $CDCl_3$)



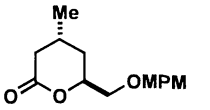
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-27
Solvent	= CHLOROFORM-D
Creation_Time	= 3-JUN-2010 20:20:53
Revision_Time	= 3-JUN-2010 20:22:16
Current_Time	= 3-JUN-2010 20:22:33
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 36
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22[dC]



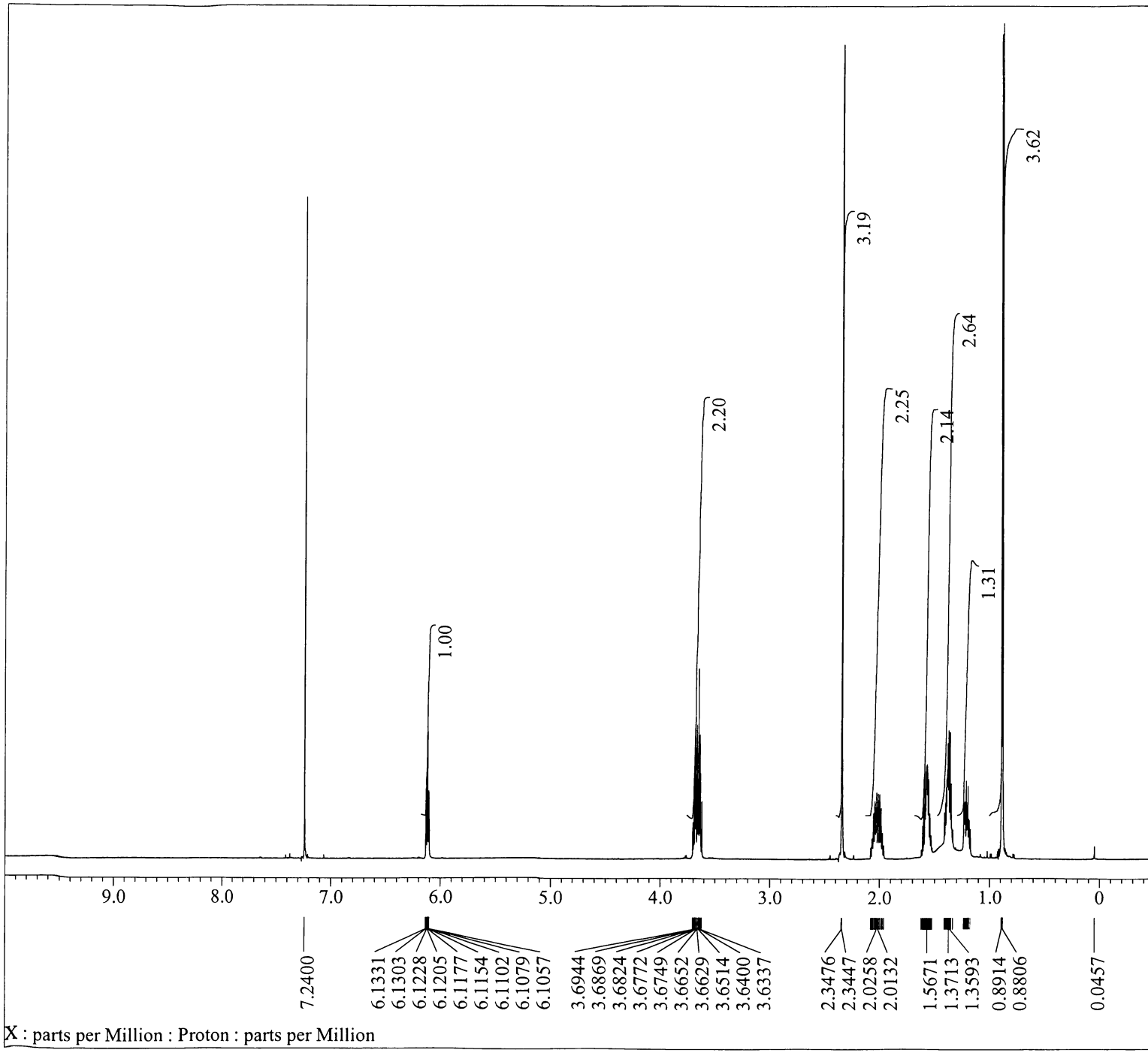
20 (600 MHz, $CDCl_3$)



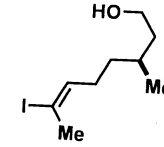

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-27
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JUN-2010 20:38:01
Revision_Time     = 12-JUN-2010 20:41:52
Current_Time      = 12-JUN-2010 20:42:45

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 80
Total_Scans       = 80

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 22.8[dC]
```



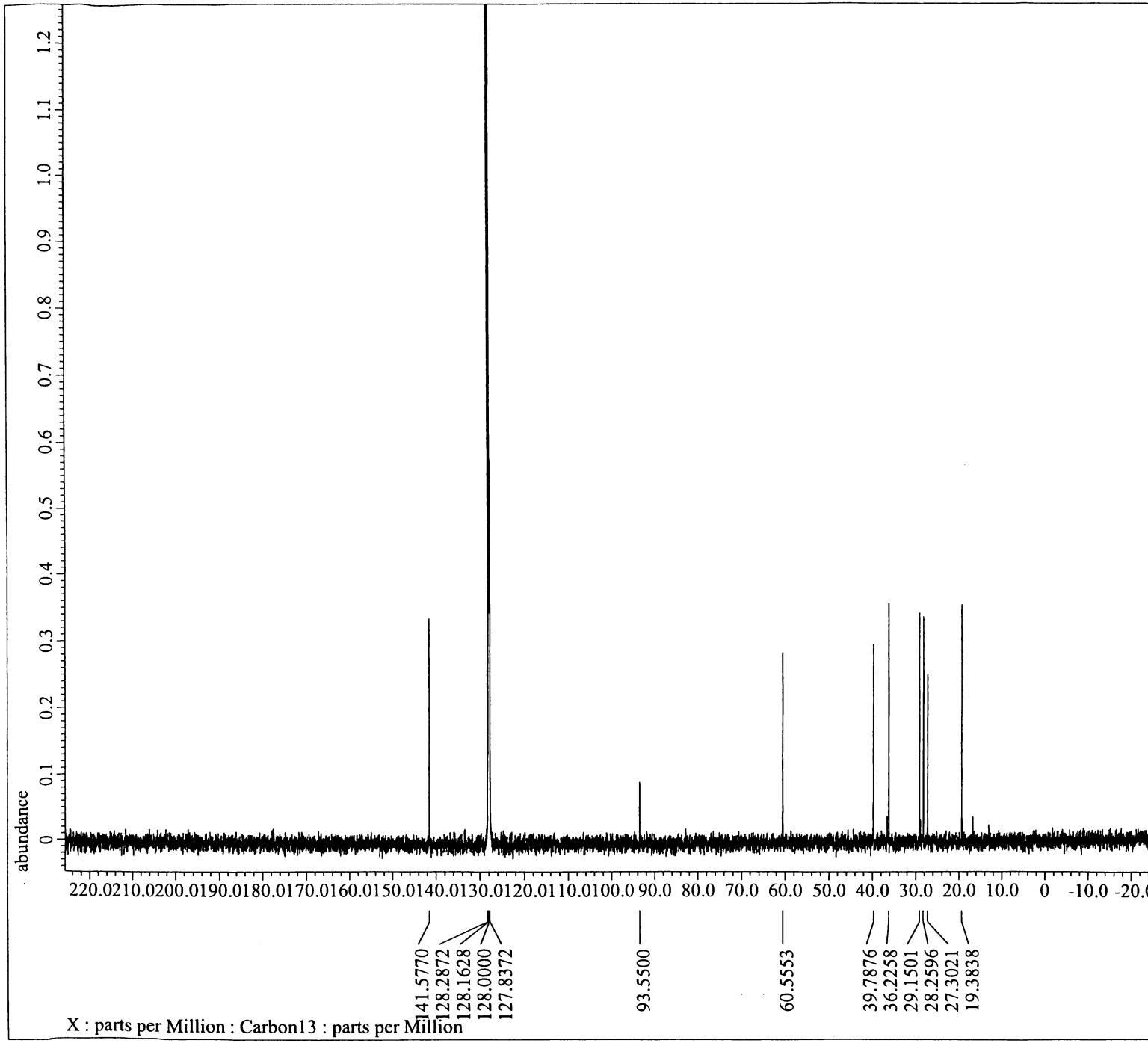
20 (150 MHz, $CDCl_3$)



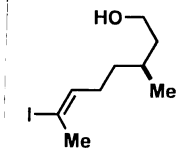

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SN-III-97-3
Solvent           = CHLOROFORM-D
Creation_Time     =  7-OCT-2010 10:41:30
Revision_Time     =  7-OCT-2010 10:45:29
Current_Time      =  7-OCT-2010 10:49:09

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 5[us]
Scans             = 8
Total_Scans       = 8

X_90_Width        = 12.4[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Recvr_Gain        = 48
Relaxation_Delay  = 1[s]
Repetition_Time   = 3.9097984[s]
Temp_Get          = 22.2[dC]
```



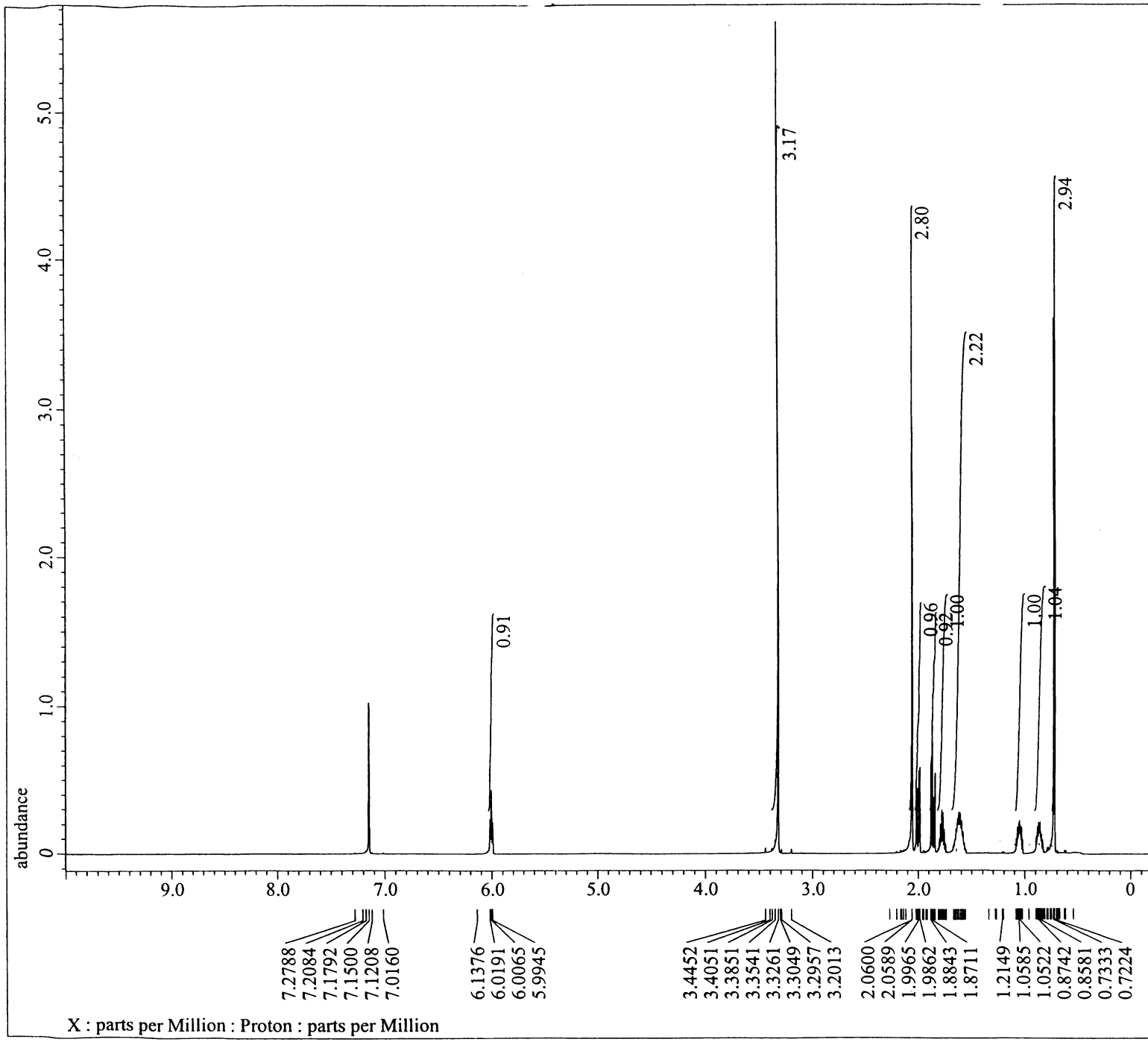
24 (600 MHz, $CDCl_3$)



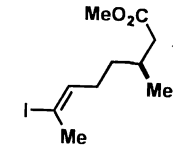

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-97
Solvent           = BENZENE-D6
Creation_Time     = 23-JUL-2010 12:11:46
Revision_Time     = 23-JUL-2010 12:14:23
Current_Time      = 23-JUL-2010 12:14:36

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 46
Total_Scans       = 46

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 50
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 22.9[dC]
```



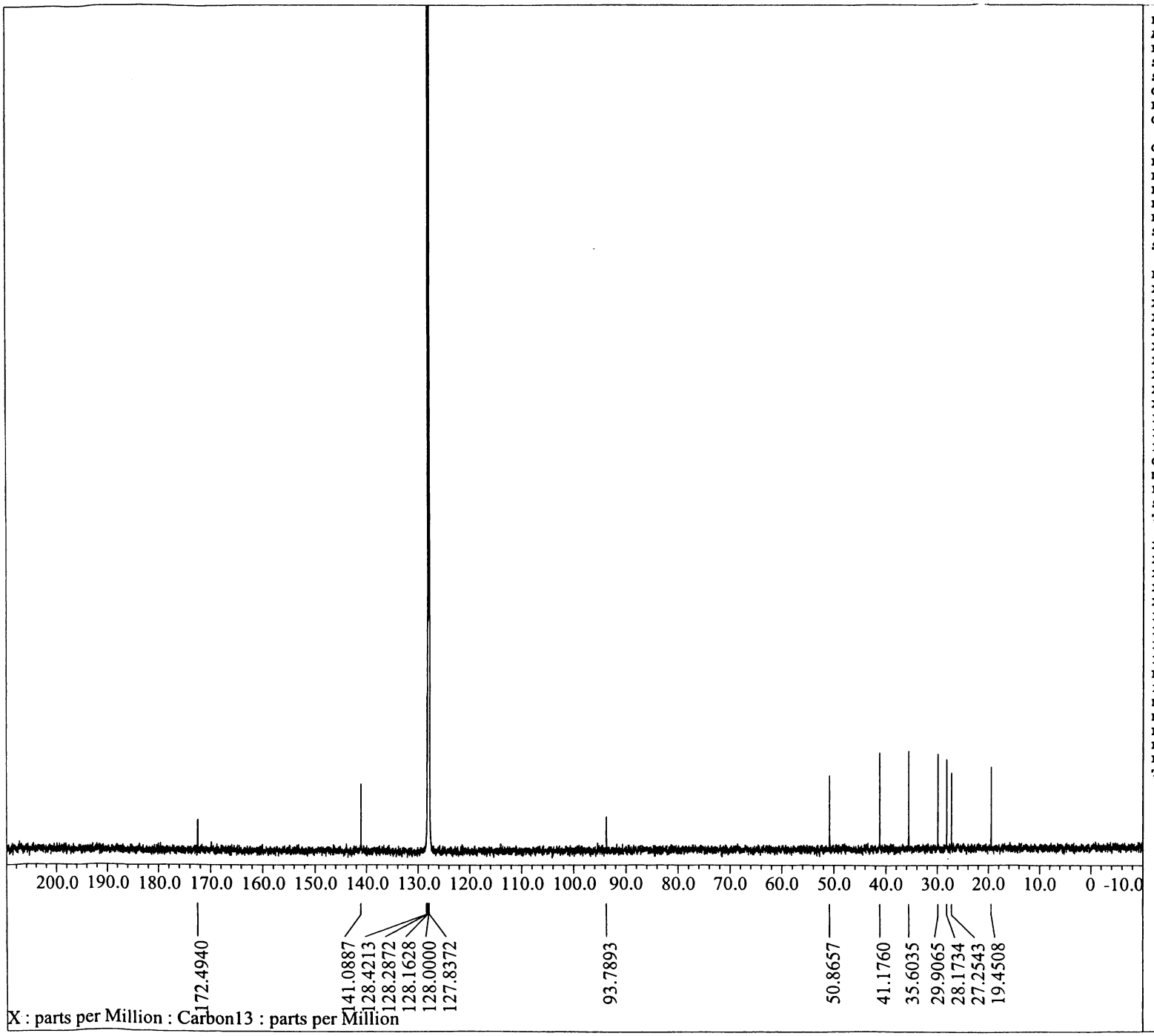
24 (150 MHz, C_6D_6)



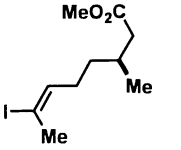
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-93
Solvent	= BENZENE-D6
Creation_Time	= 12-JUL-2010 20:27:08
Revision_Time	= 12-JUL-2010 20:27:59
Current_Time	= 12-JUL-2010 20:28:16
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 36
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.3[dC]



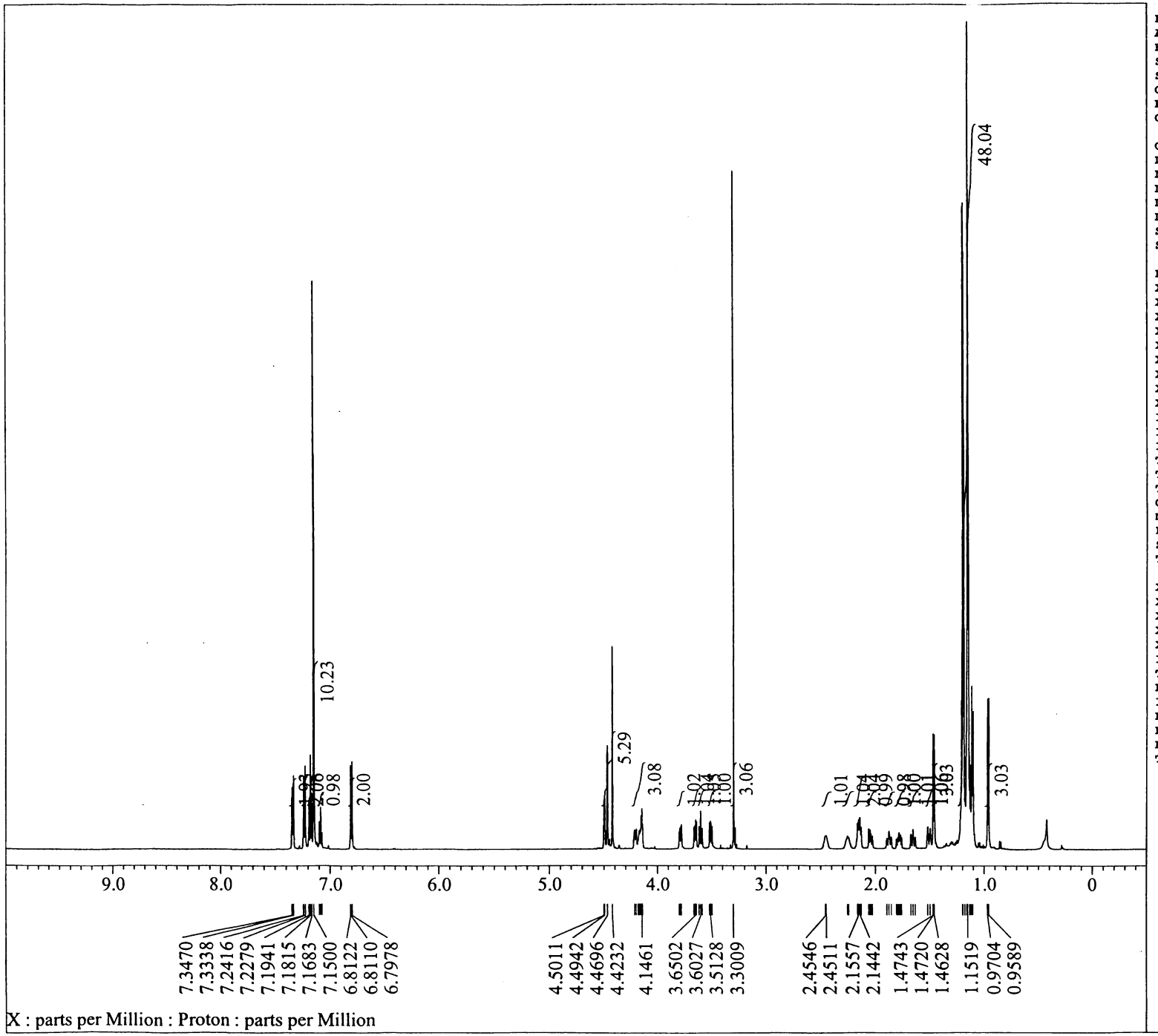
6 (600 MHz, C_6D_6)



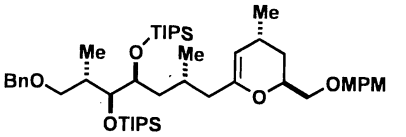

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-93
Solvent           = BENZENE-D6
Creation_Time     = 13-SEP-2010 13:27:46
Revision_Time     = 13-SEP-2010 13:37:34
Current_Time      = 13-SEP-2010 13:37:59

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 178
Total_Scans       = 178

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 50
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23[dC]
```



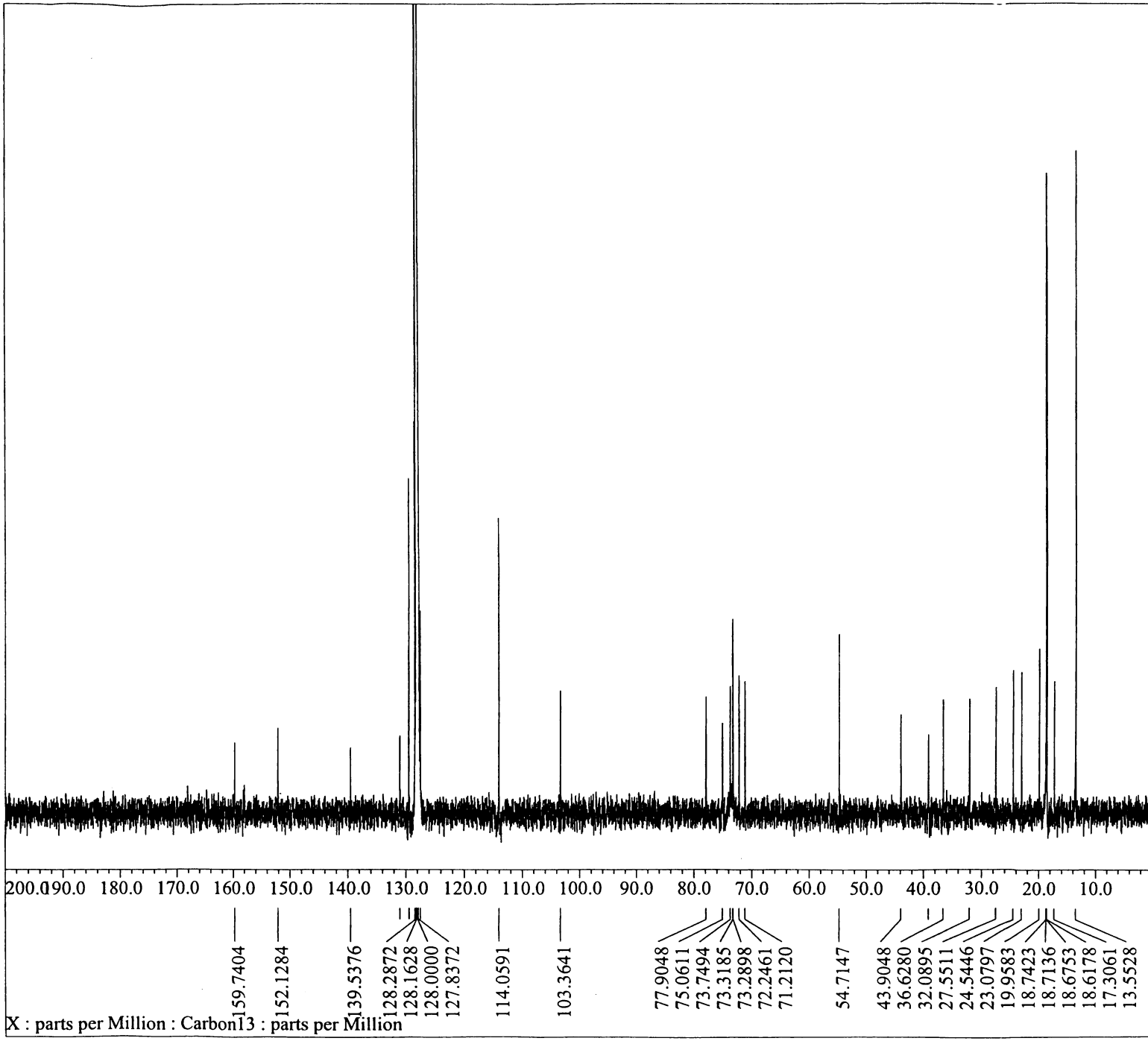
6 (150 MHz, C_6D_6)



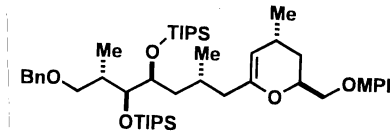
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-149
Solvent	= BENZENE-D6
Creation_Time	= 21-SEP-2010 14:37:41
Revision_Time	= 21-SEP-2010 14:45:40
Current_Time	= 21-SEP-2010 14:45:56
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 40
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.3[dC]



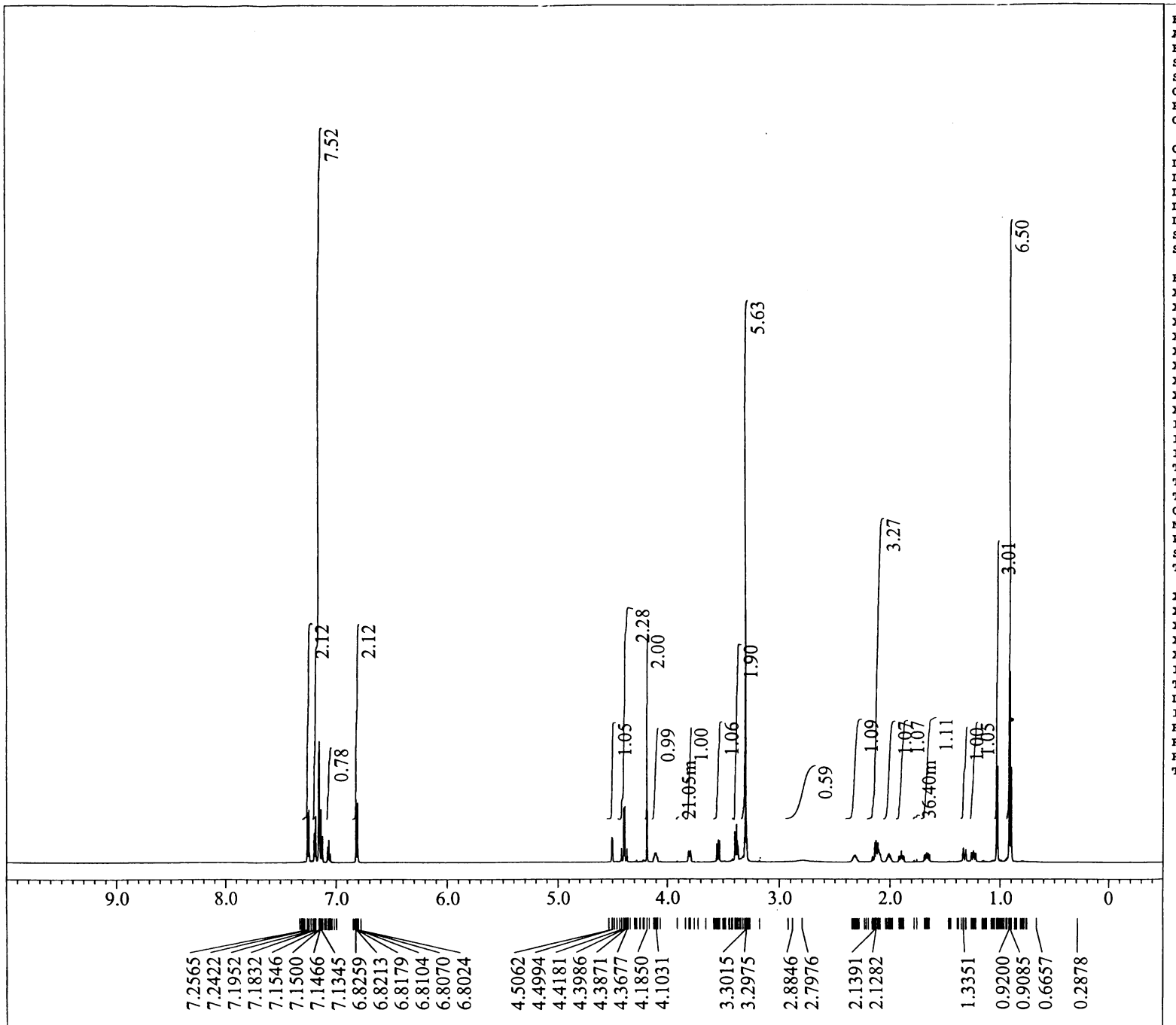
25 (600 MHz, C_6D_6)



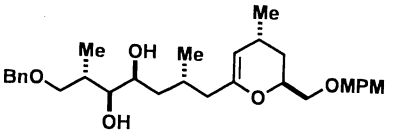

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-149
Solvent           = BENZENE-D6
Creation_Time     = 21-SEP-2010 14:40:05
Revision_Time     = 21-SEP-2010 14:59:08
Current_Time      = 21-SEP-2010 14:59:30

Comment           = single pulse decoupled gat
Data_Format       = 1D REAL
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 372
Total_Scans       = 372

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 52
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.2[dC]
```



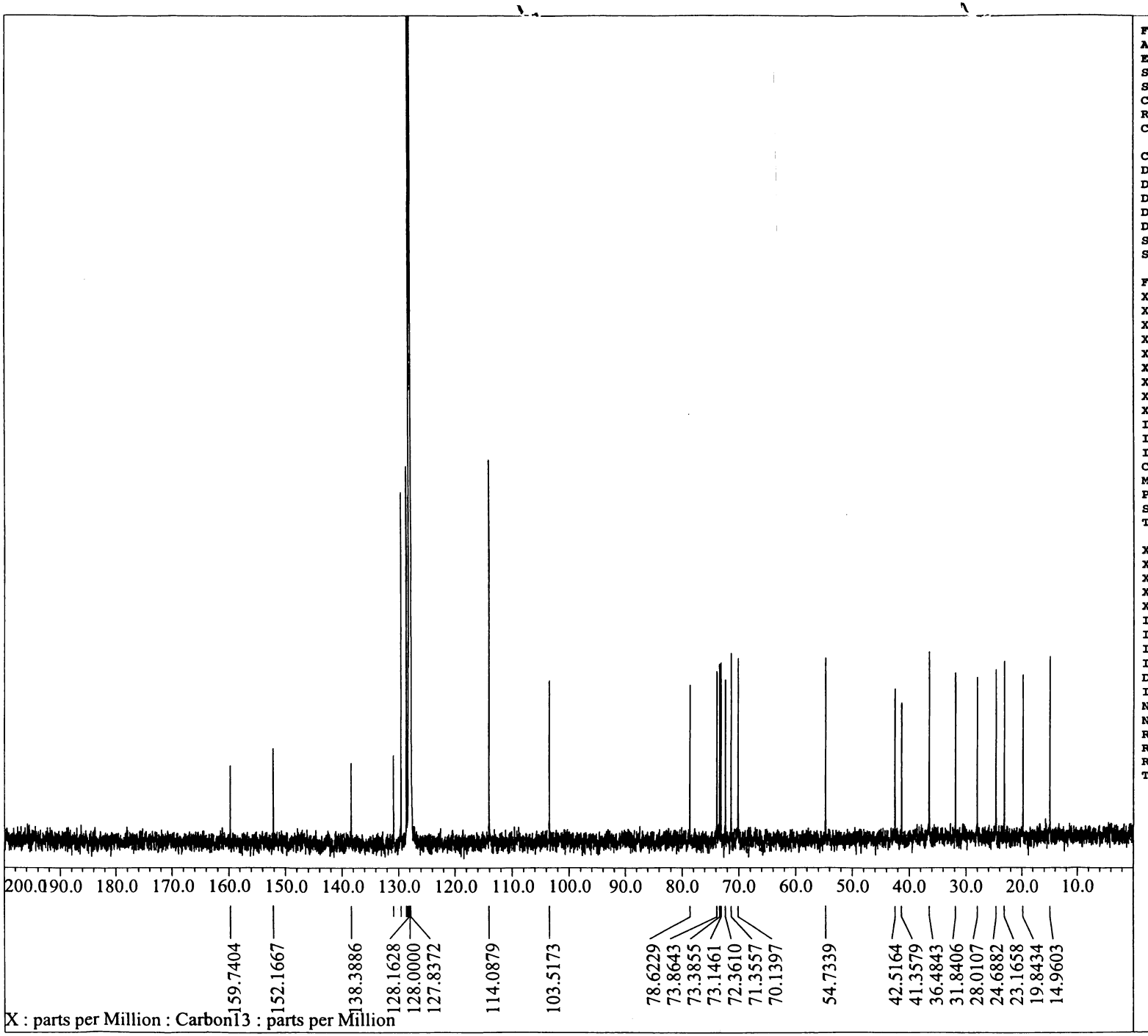
25 (150 MHz, C_6D_6)



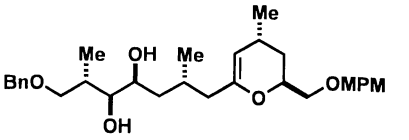

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SN-III-73-1
Solvent           = BENZENE-D6
Creation_Time     = 19-JUN-2010 20:09:38
Revision_Time     = 15-SEP-2010 18:43:55
Current_Time      = 15-SEP-2010 18:44:49

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 5[us]
Scans             = 4
Total_Scans       = 4

X_90_Width        = 12.4[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Recvr_Gain        = 36
Relaxation_Delay  = 1[s]
Repetition_Time   = 3.9097984[s]
Temp_Get          = 22.2[dC]
```



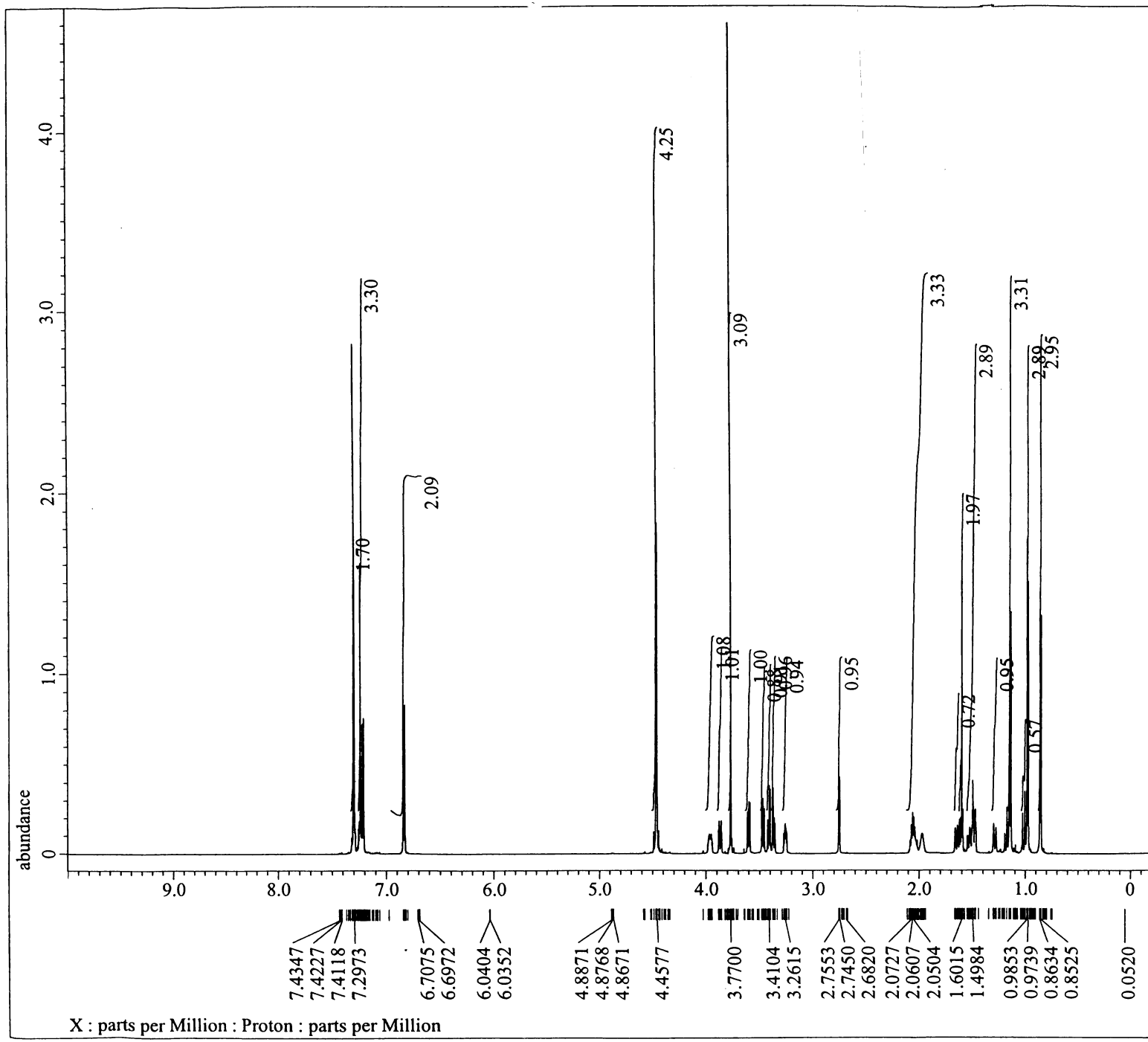
7 (600 MHz, C_6D_6)



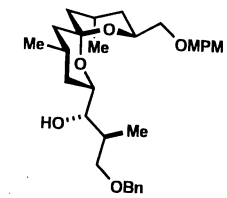
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= SN-III-73-1
Solvent	= BENZENE-D6
Creation_Time	= 19-JUN-2010 20:11:34
Revision_Time	= 19-JUN-2010 20:18:33
Current_Time	= 19-JUN-2010 20:21:45
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 0.69206016[s]
X_Domain	= 13C
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 20[us]
Scans	= 128
Total_Scans	= 128
X_90_Width	= 8.4[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 6.4[dB]
X_Pulse	= 2.8[us]
Irr_Atn_Dec	= 18[dB]
Irr_Atn_Noe	= 18[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 76[us]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Recvr_Gain	= 50
Relaxation_Delay	= 2[s]
Repetition_Time	= 2.69206016[s]
Temp_Get	= 23[dC]



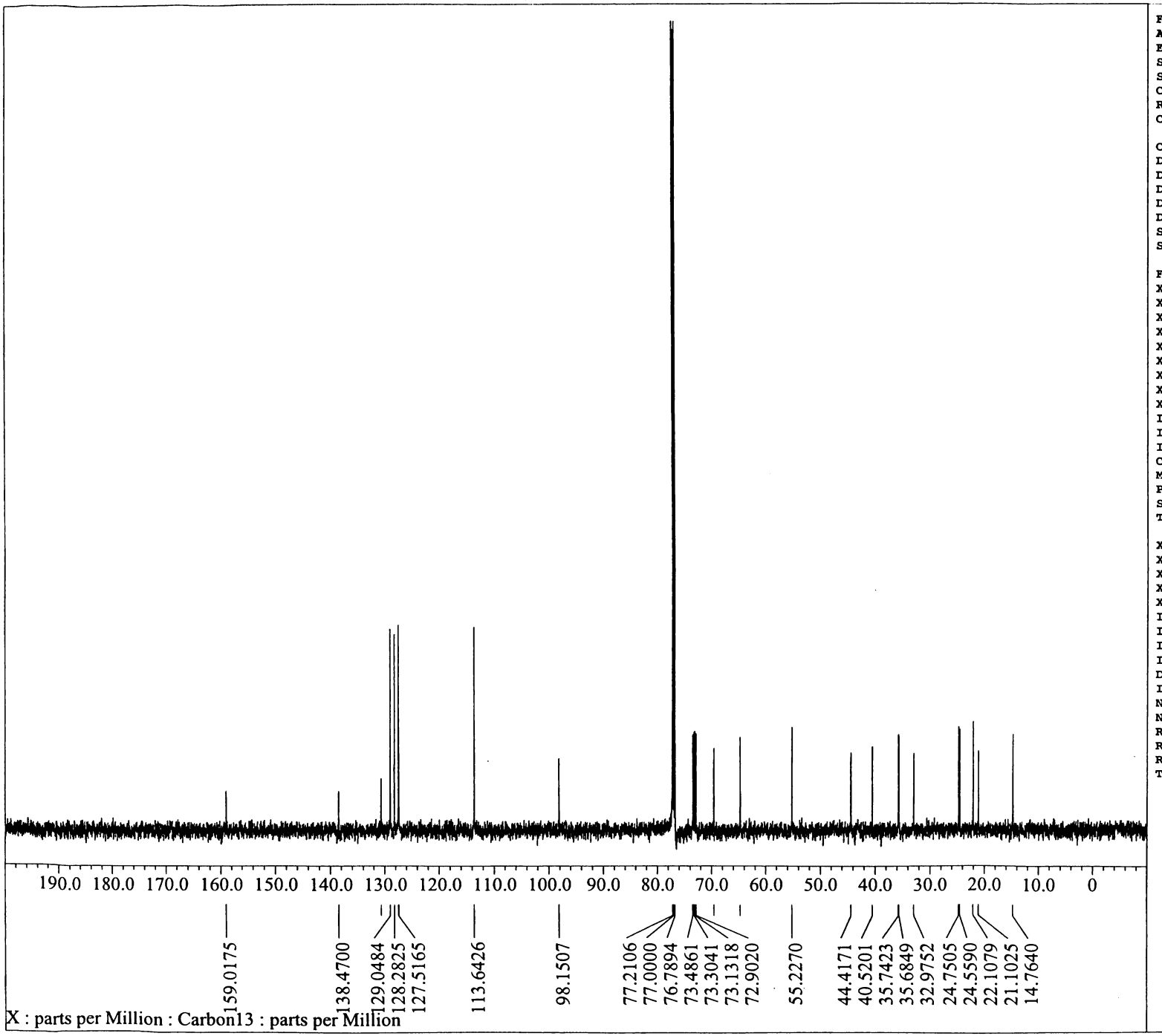
7 (150 MHz, C_6D_6)



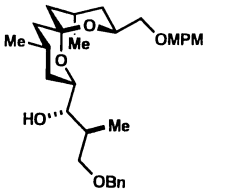
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-74
Solvent	= CHLOROFORM-D
Creation_Time	= 21-JUN-2010 12:40:12
Revision_Time	= 21-JUN-2010 12:42:34
Current_Time	= 21-JUN-2010 12:42:55
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 40
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.4[dC]



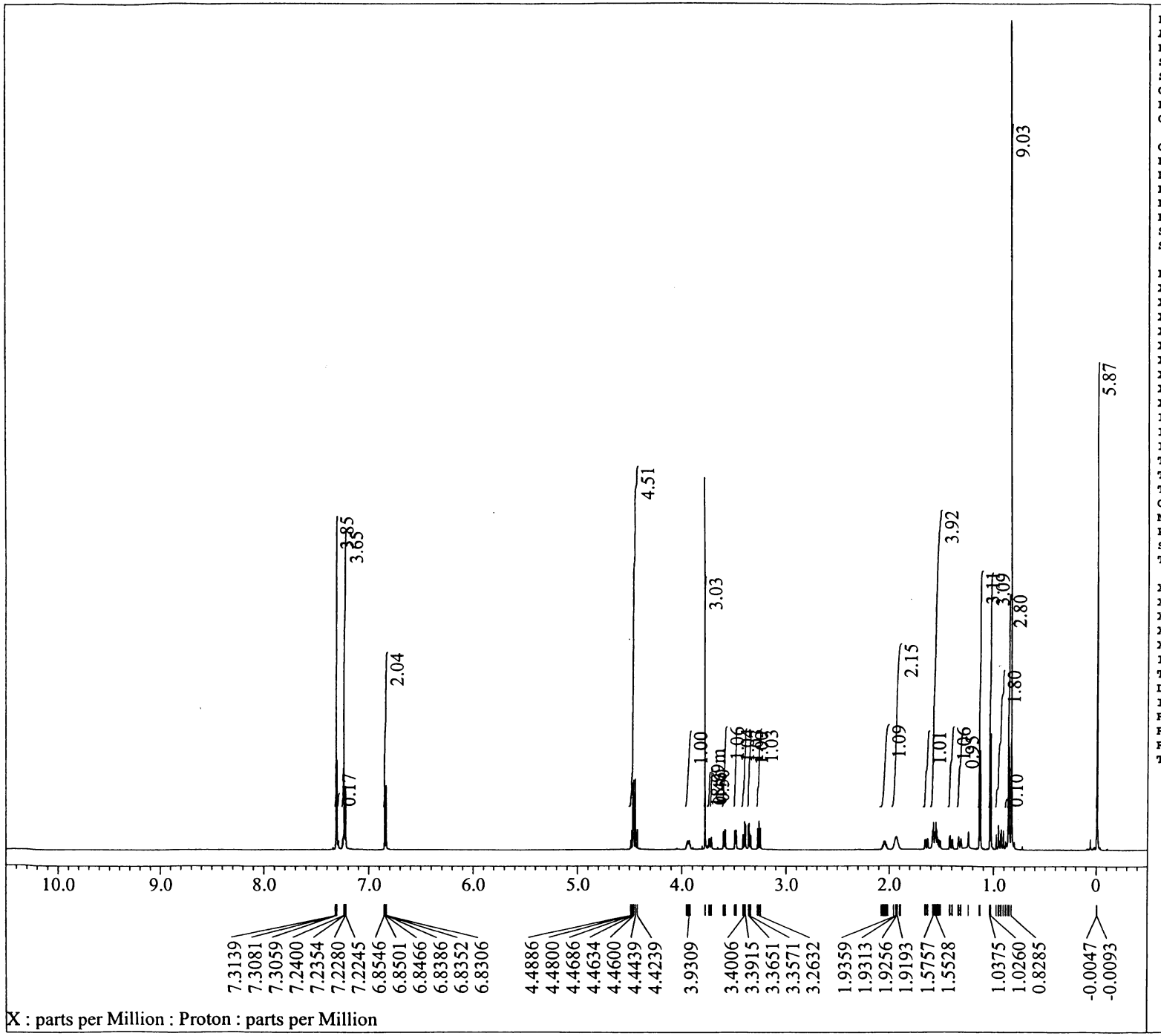
26 (600 MHz, $CDCl_3$)



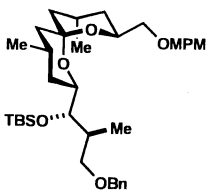
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= SN-III-74
Solvent	= CHLOROFORM-D
Creation_Time	= 17-SEP-2010 11:59:45
Revision_Time	= 17-SEP-2010 12:03:22
Current_Time	= 17-SEP-2010 12:03:46
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 0.69206016[s]
X_Domain	= 13C
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 20[us]
Scans	= 58
Total_Scans	= 58
X_90_Width	= 8.4[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 6.4[dB]
X_Pulse	= 2.8[us]
Irr_Atn_Dec	= 18[dB]
Irr_Atn_Noe	= 18[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 76[us]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Recvr_Gain	= 56
Relaxation_Delay	= 2[s]
Repetition_Time	= 2.69206016[s]
Temp_Get	= 23.1[dC]



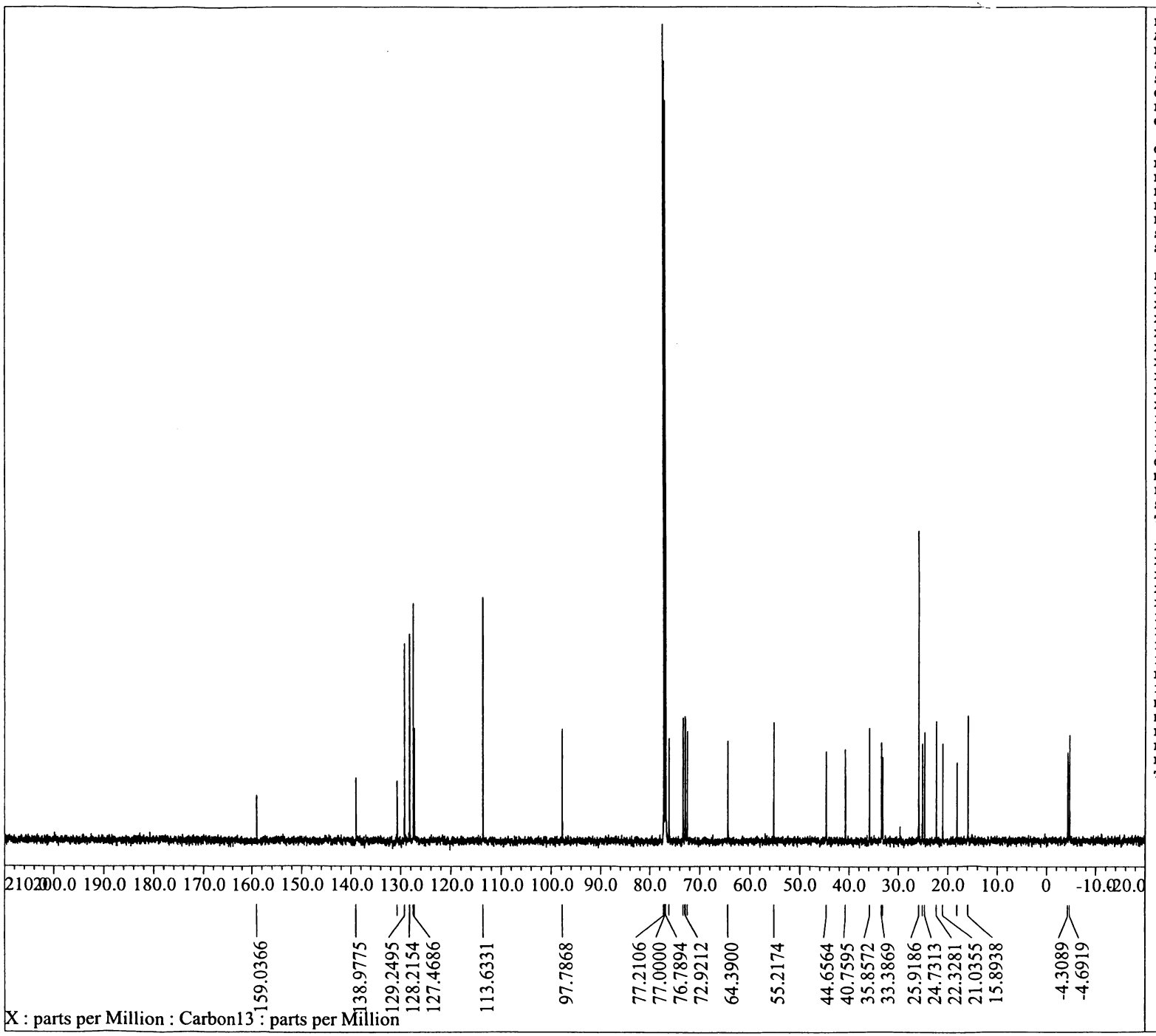
26 (150 MHz, $CDCl_3$)



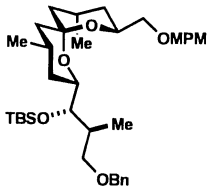
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-39-1
Solvent	= CHLOROFORM-D
Creation_Time	= 13-SEP-2010 19:43:16
Revision_Time	= 13-SEP-2010 19:52:24
Current_Time	= 13-SEP-2010 19:53:01
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 36
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.2[dC]



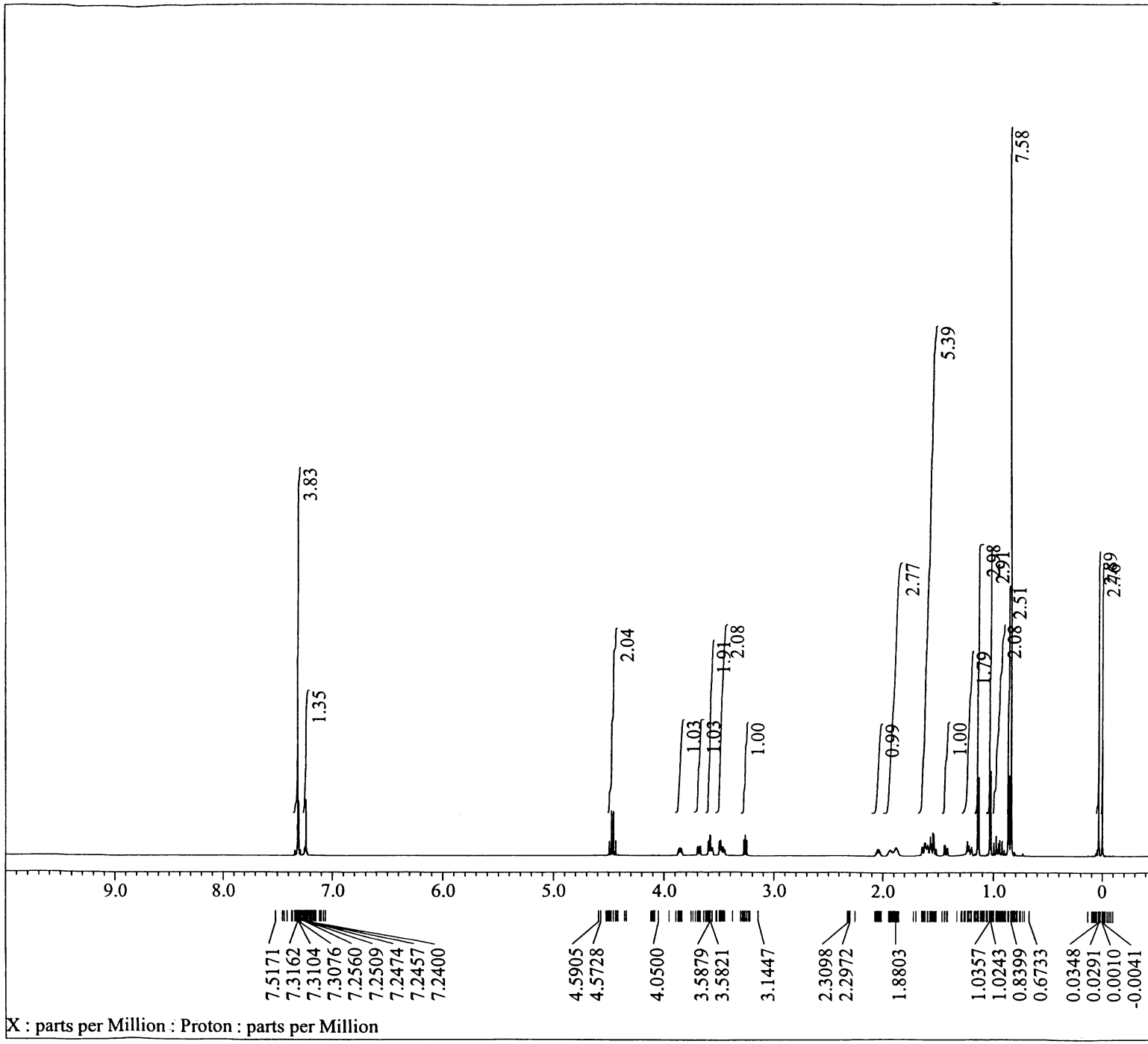
S3 (600 MHz, $CDCl_3$)



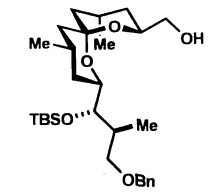

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-39-1
Solvent           = CHLOROFORM-D
Creation_Time     = 13-SEP-2010 19:46:07
Revision_Time     = 13-SEP-2010 20:00:09
Current_Time      = 13-SEP-2010 20:00:56

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 261
Total_Scans       = 261

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.2[dC]
```



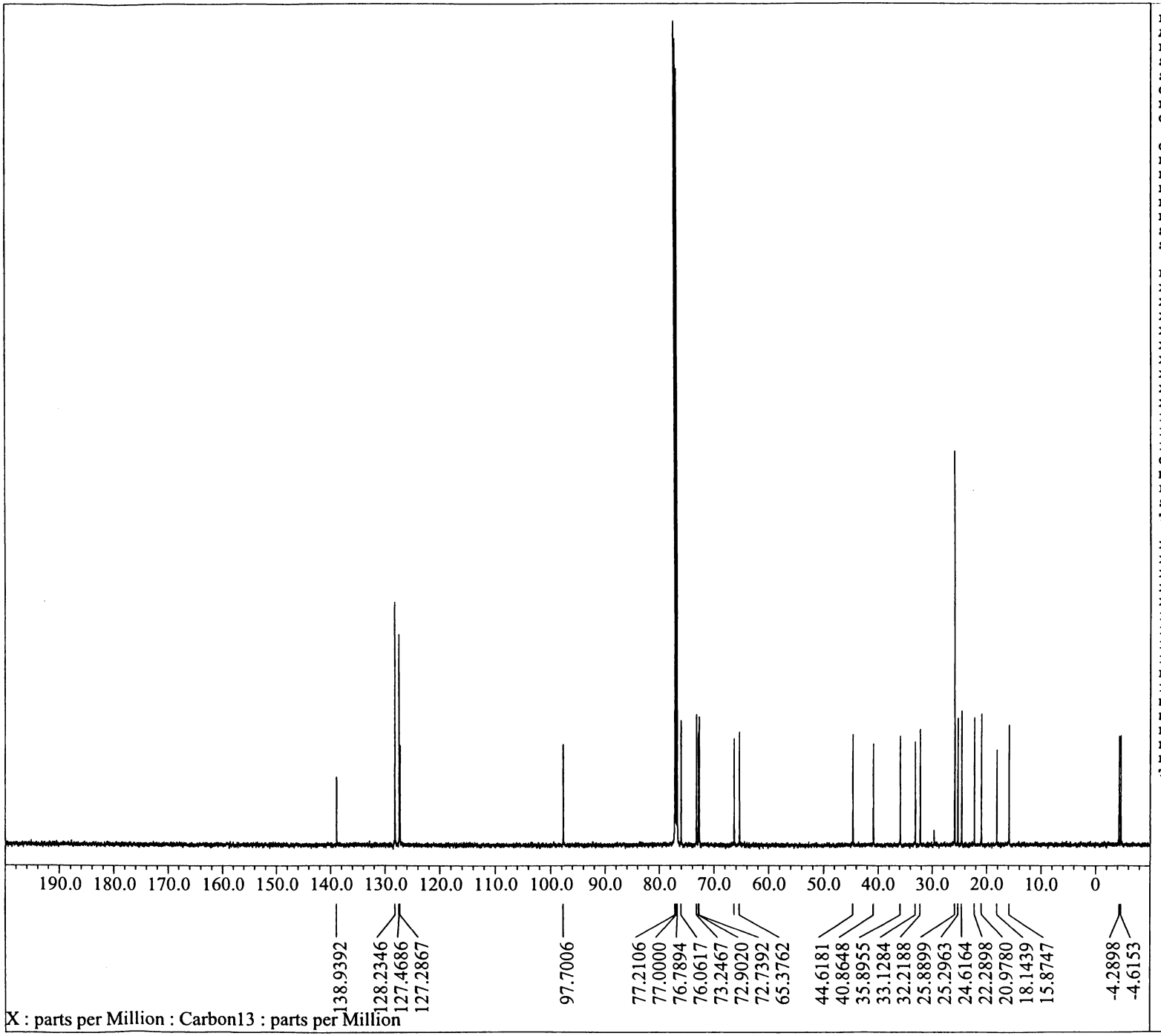
S3 (150 MHz, $CDCl_3$)



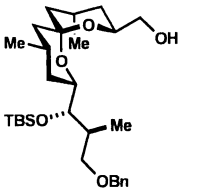

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SN-III-40
Solvent           = CHLOROFORM-D
Creation_Time     = 21-JUL-2010 19:11:27
Revision_Time     = 16-SEP-2010 14:07:25
Current_Time      = 16-SEP-2010 14:08:12

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 5[us]
Scans             = 8
Total_Scans       = 8

X_90_Width        = 12.4[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Recvr_Gain        = 36
Relaxation_Delay  = 1[s]
Repetition_Time   = 3.9097984[s]
Temp_Get          = 22.4[dC]
```



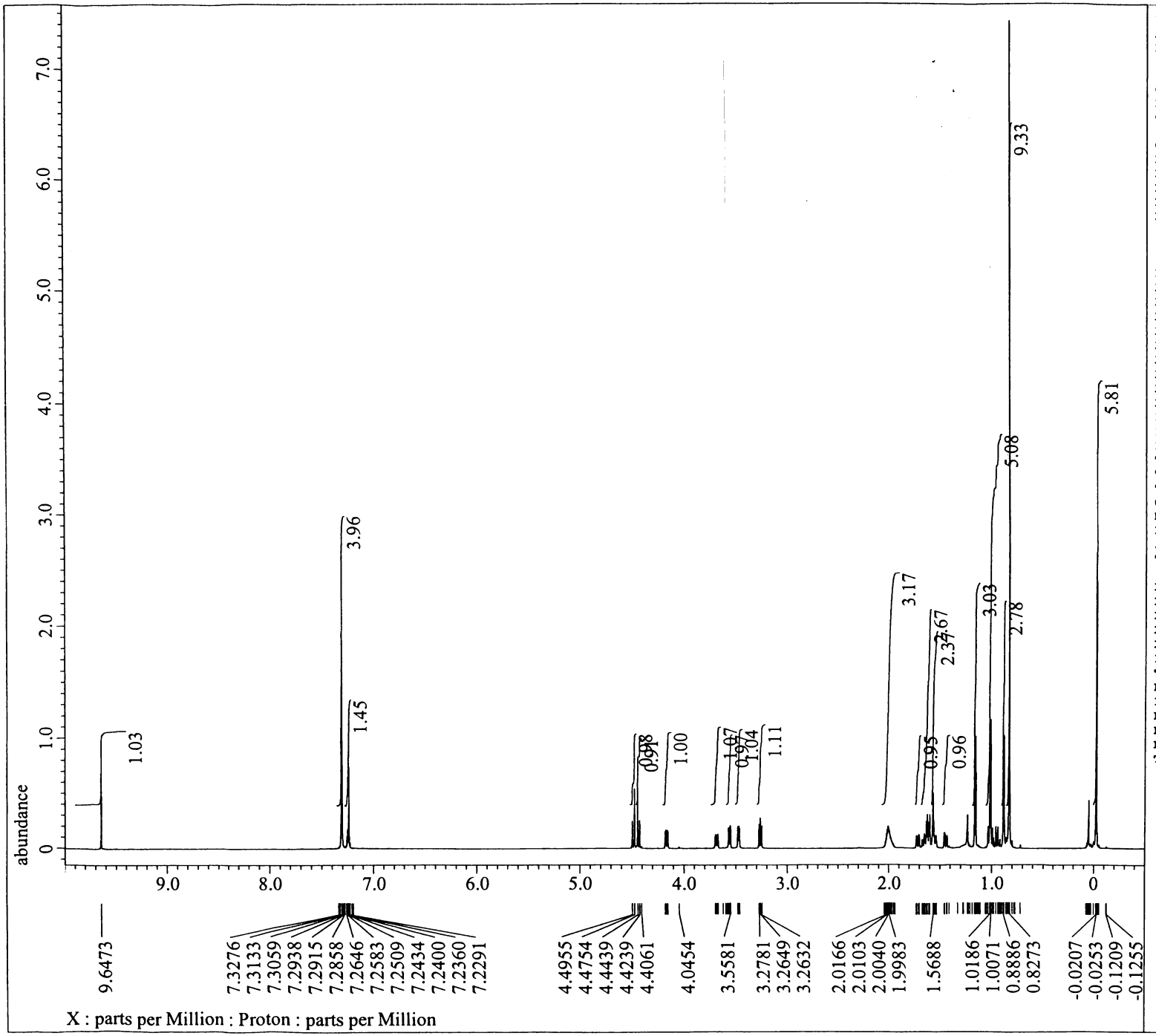
S4 (600 MHz, $CDCl_3$)



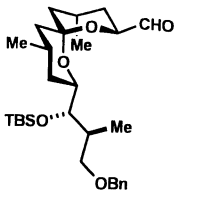

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-40
Solvent           = CHLOROFORM-D
Creation_Time     = 20-MAY-2010 17:40:09
Revision_Time     = 17-SEP-2010 11:36:00
Current_Time      = 17-SEP-2010 11:36:34

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 720
Total_Scans       = 720

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 22.3[dC]
```



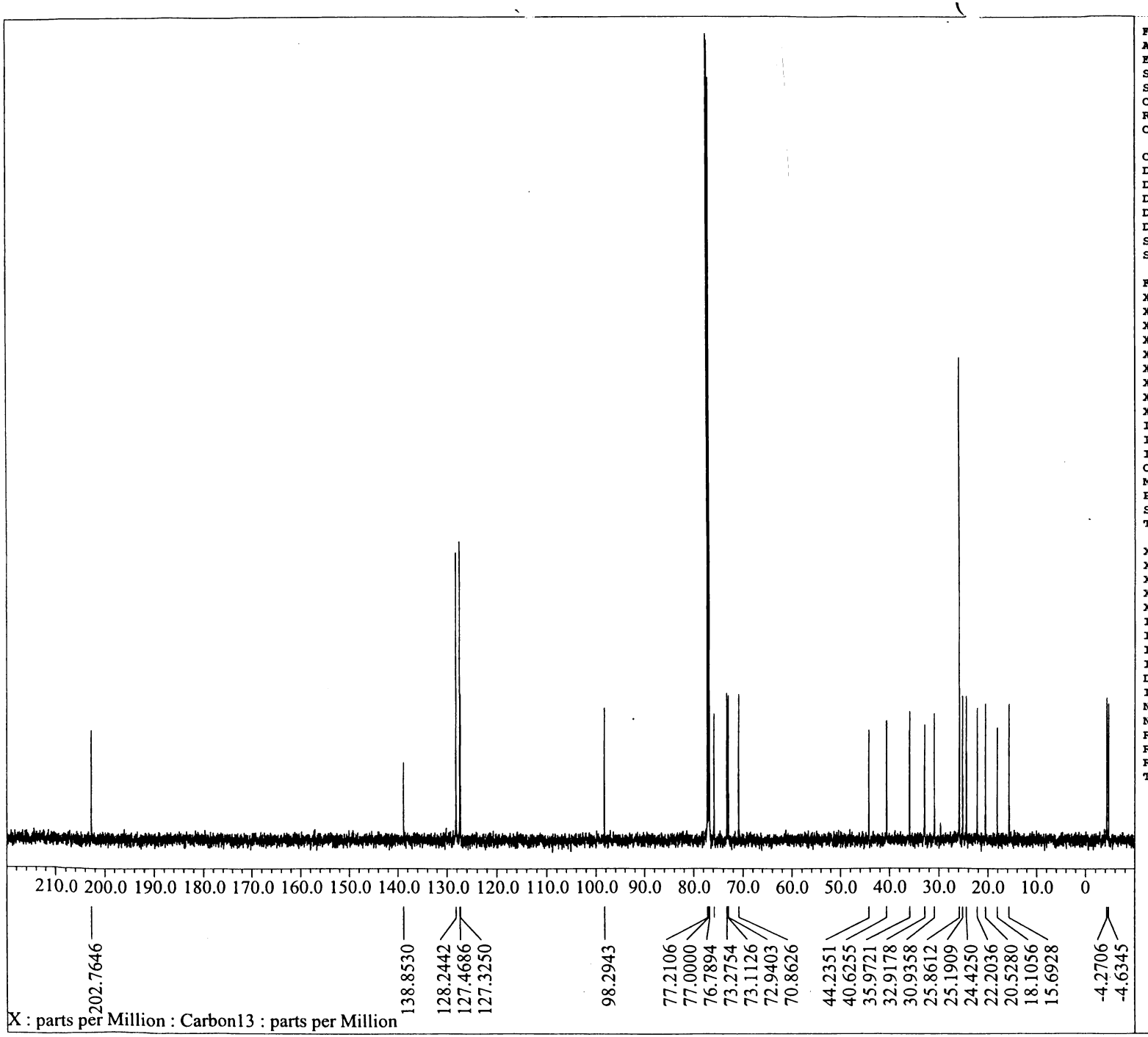
S4 (150 MHz, $CDCl_3$)



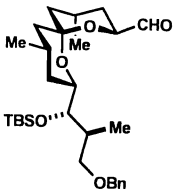
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-80-1
Solvent	= CHLOROFORM-D
Creation_Time	= 16-JUL-2010 19:05:26
Revision_Time	= 16-JUL-2010 19:07:10
Current_Time	= 16-JUL-2010 19:07:15
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 38
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.6[dC]



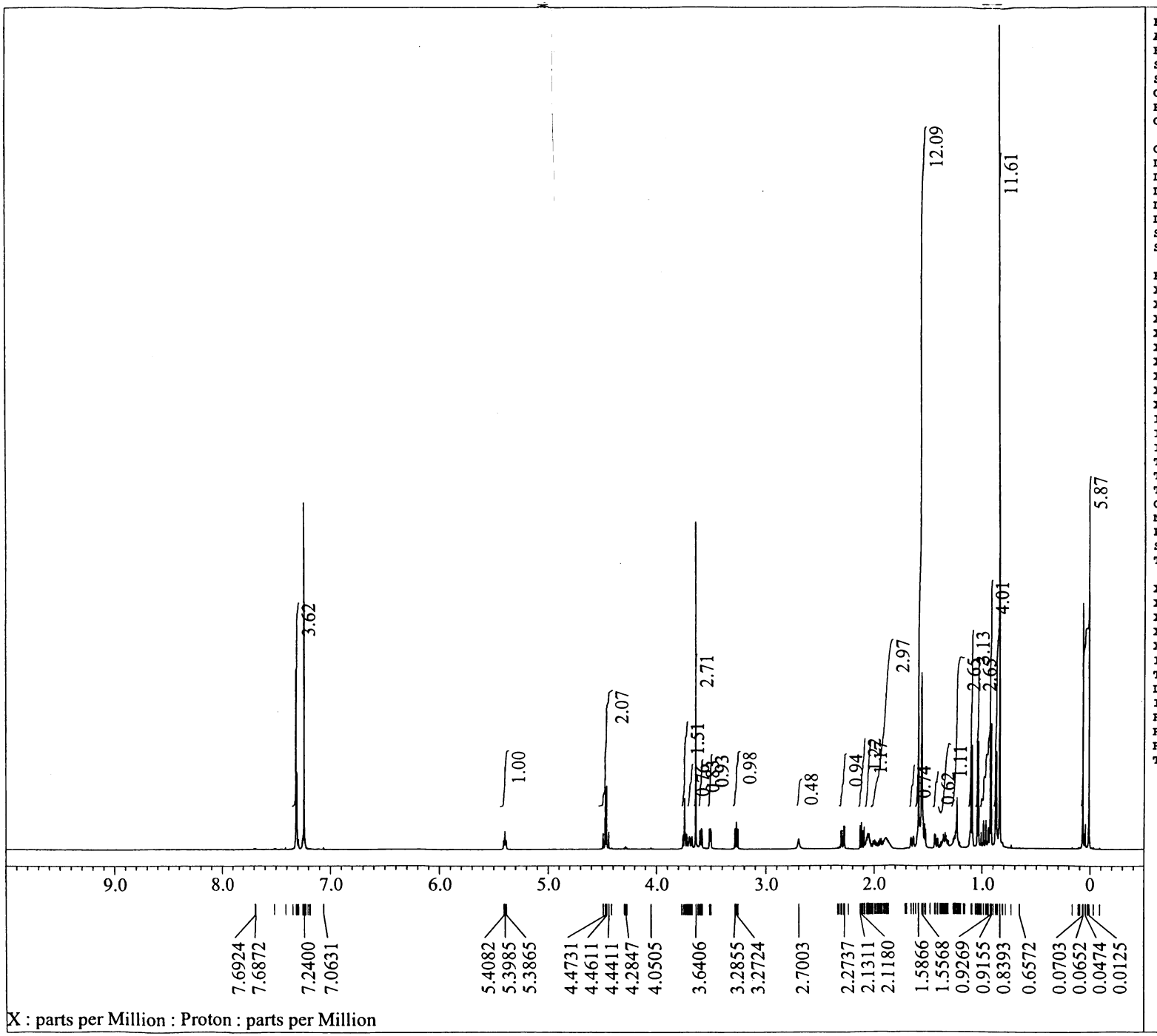
5 (600 MHz, $CDCl_3$)



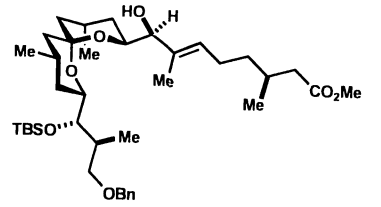

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-113
Solvent           = CHLOROFORM-D
Creation_Time     =  4-AUG-2010 17:38:25
Revision_Time     =  4-AUG-2010 17:42:28
Current_Time      =  4-AUG-2010 17:42:49

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 97
Total_Scans       = 97

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.5[dC]
```



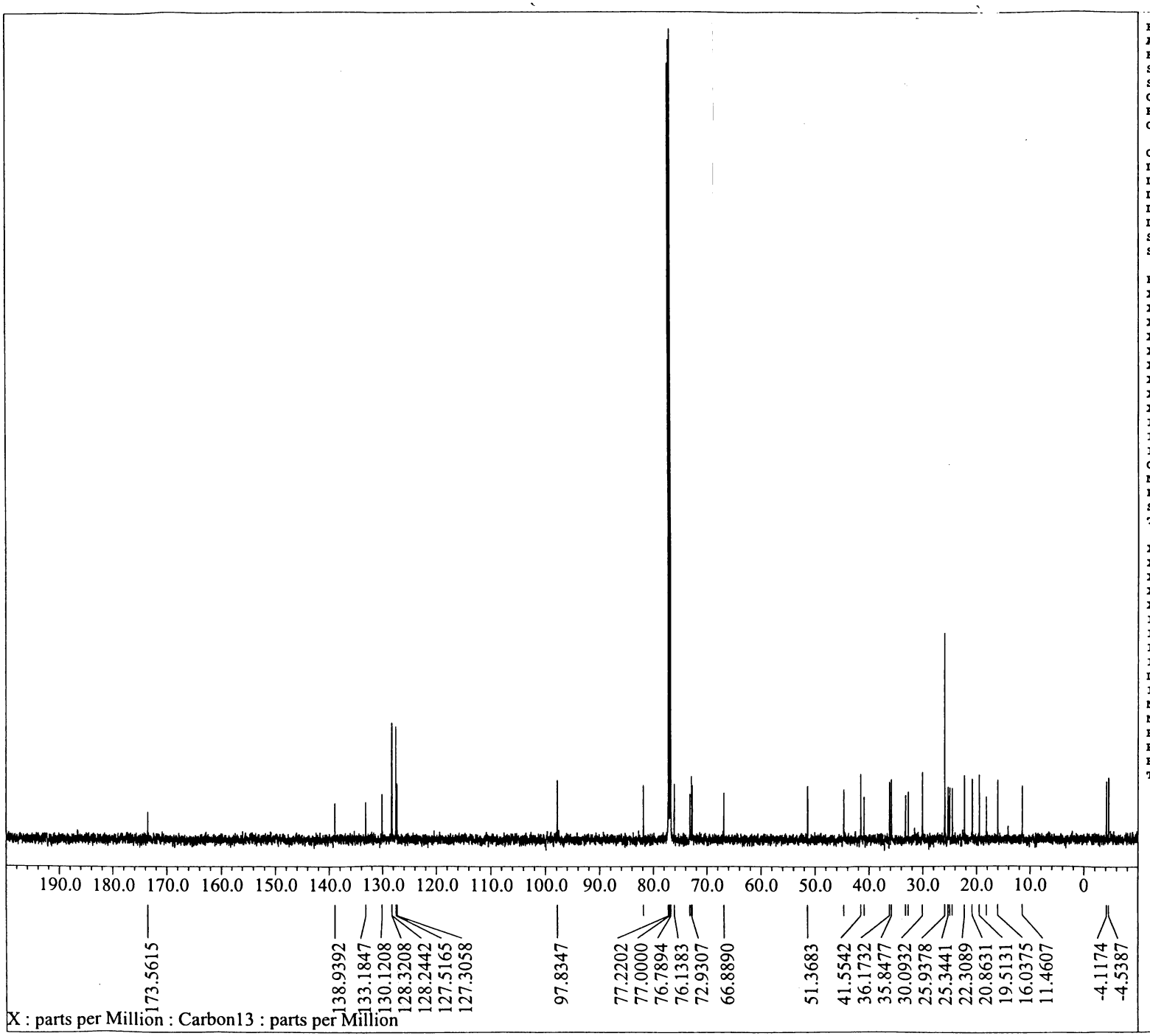
5 (150 MHz, $CDCl_3$)



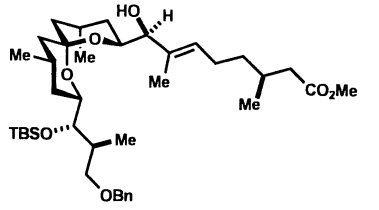
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-82-5
Solvent	= CHLOROFORM-D
Creation_Time	= 24-JUL-2010 14:53:37
Revision_Time	= 24-JUL-2010 14:57:23
Current_Time	= 24-JUL-2010 14:57:49
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 48
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.6[dC]



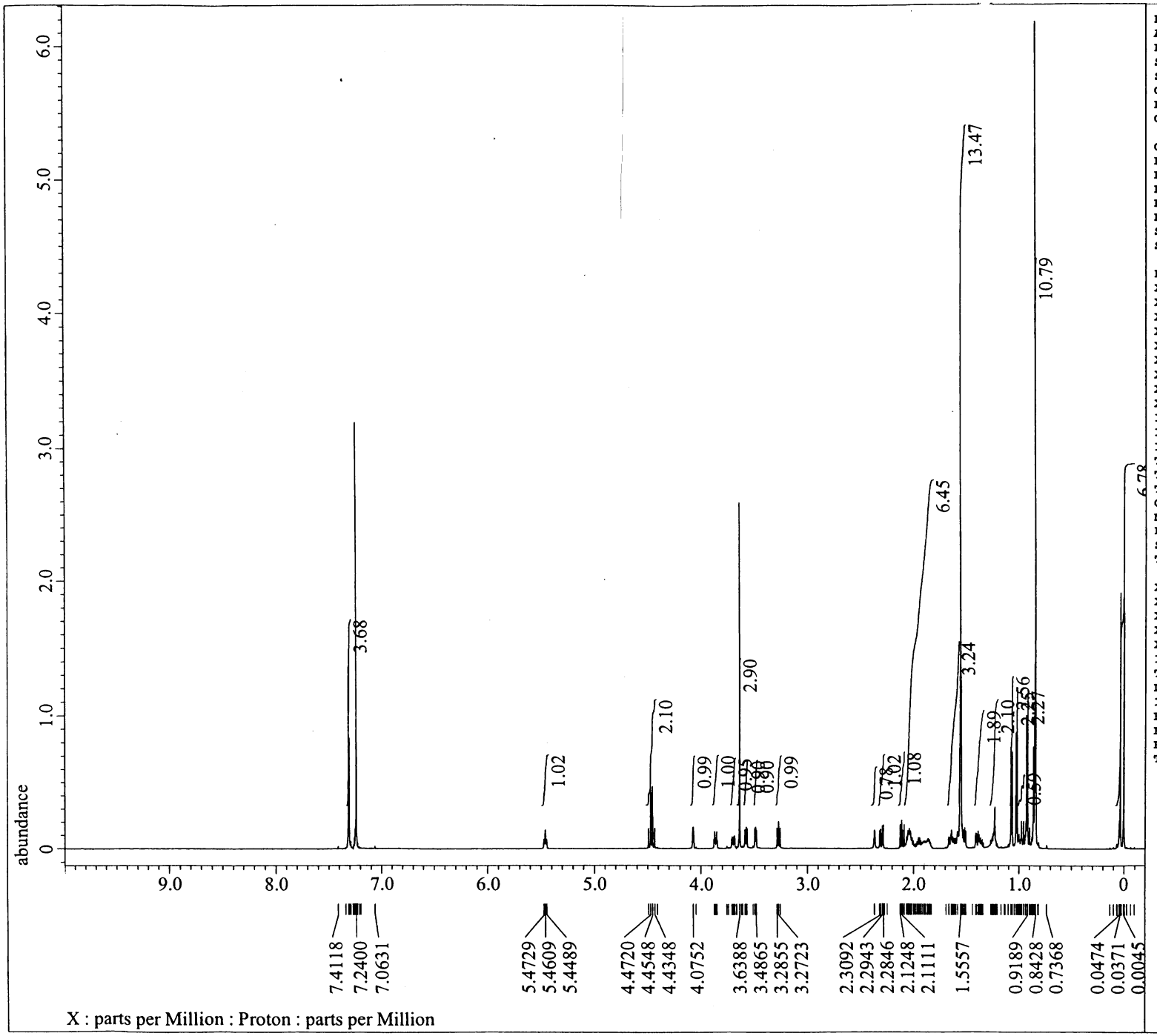
4 (600 MHz, $CDCl_3$)



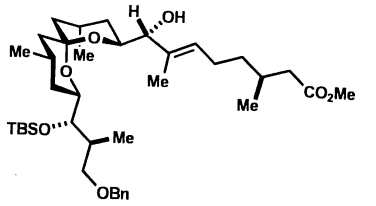

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-121-1-3
Solvent           = CHLOROFORM-D
Creation_Time     = 18-AUG-2010 21:12:14
Revision_Time     = 18-AUG-2010 21:18:25
Current_Time      = 18-AUG-2010 21:18:42

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 162
Total_Scans       = 162

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 54
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.4[dC]
```



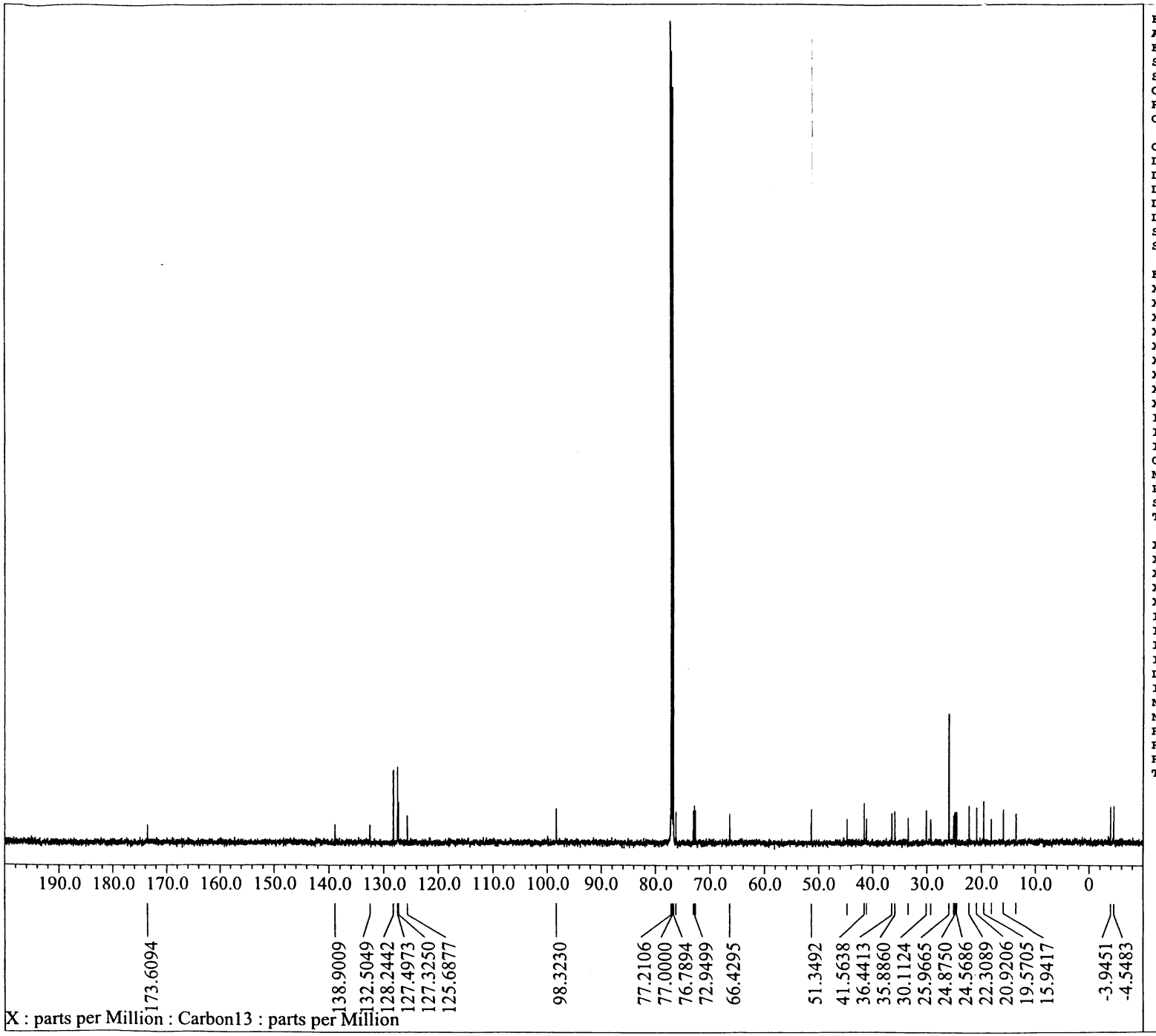
4 (150 MHz, $CDCl_3$)



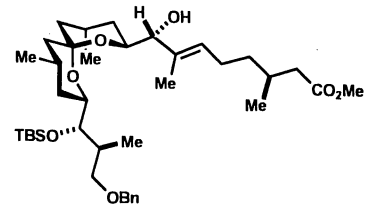

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = SN-III-82-8
Solvent           = CHLOROFORM-D
Creation_Time     = 24-JUL-2010 17:42:03
Revision_Time     = 24-JUL-2010 17:44:00
Current_Time      = 24-JUL-2010 17:44:18

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 2.9097984[s]
X_Domain          = 1H
X_Freq            = 600.1723046[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.34366642[Hz]
X_Sweep           = 11.26126126[kHz]
X_Sweep_Clipped   = 9.00900901[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 600.1723046[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 5[us]
Scans             = 8
Total_Scans       = 8

X_90_Width        = 12.4[us]
X_Acq_Time        = 2.9097984[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.2[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Recvr_Gain        = 50
Relaxation_Delay  = 1[s]
Repetition_Time   = 3.9097984[s]
Temp_Get          = 22.7[dC]
```



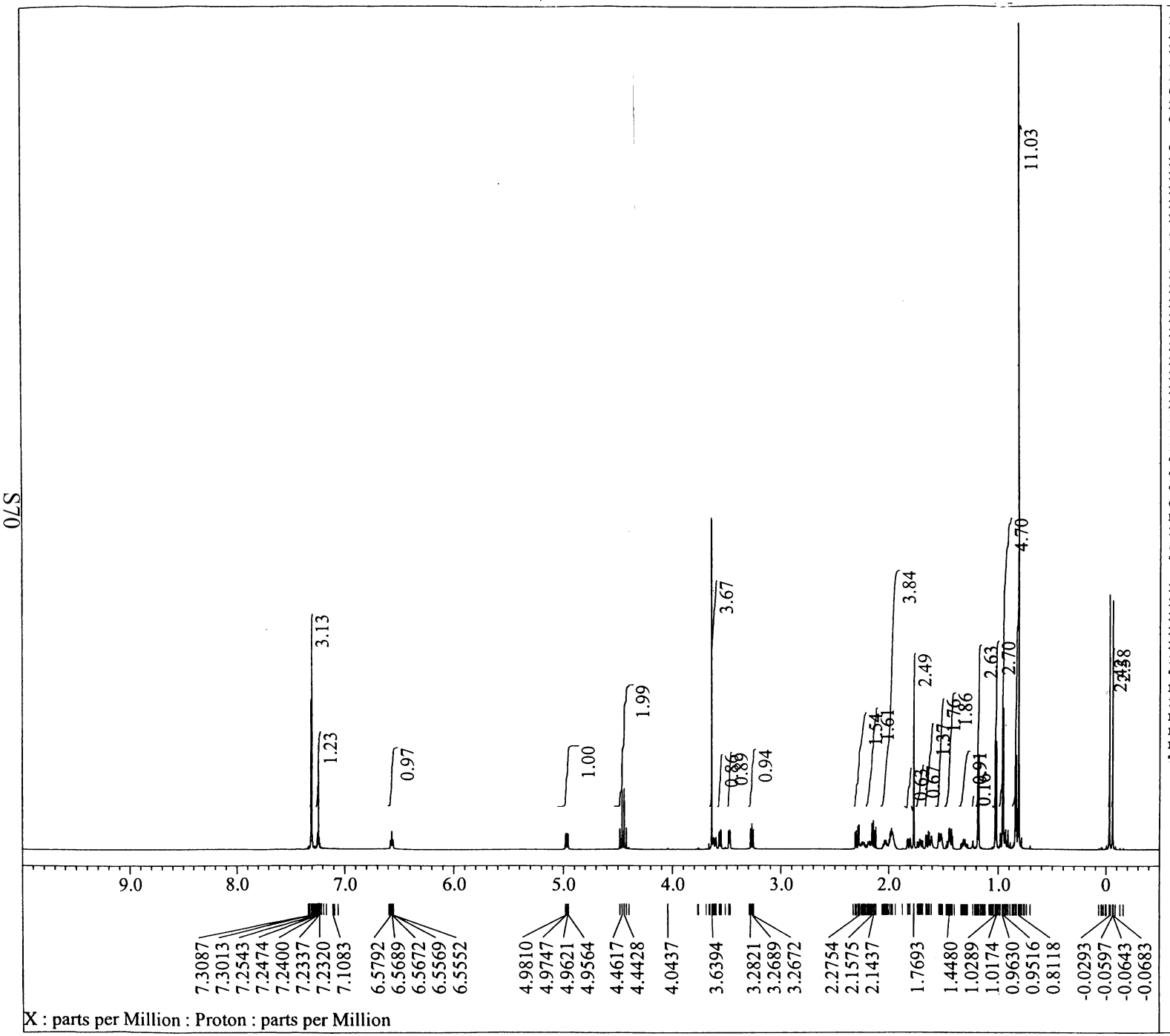
21-*epi*-4 (600 MHz, $CDCl_3$)



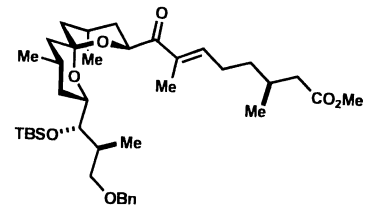

```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-121-1-6
Solvent           = CHLOROFORM-D
Creation_Time     = 17-AUG-2010 19:17:48
Revision_Time     = 17-AUG-2010 19:36:56
Current_Time      = 17-AUG-2010 19:37:12

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 452.0
Total_Scans       = 452.0

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.6[dC]
```



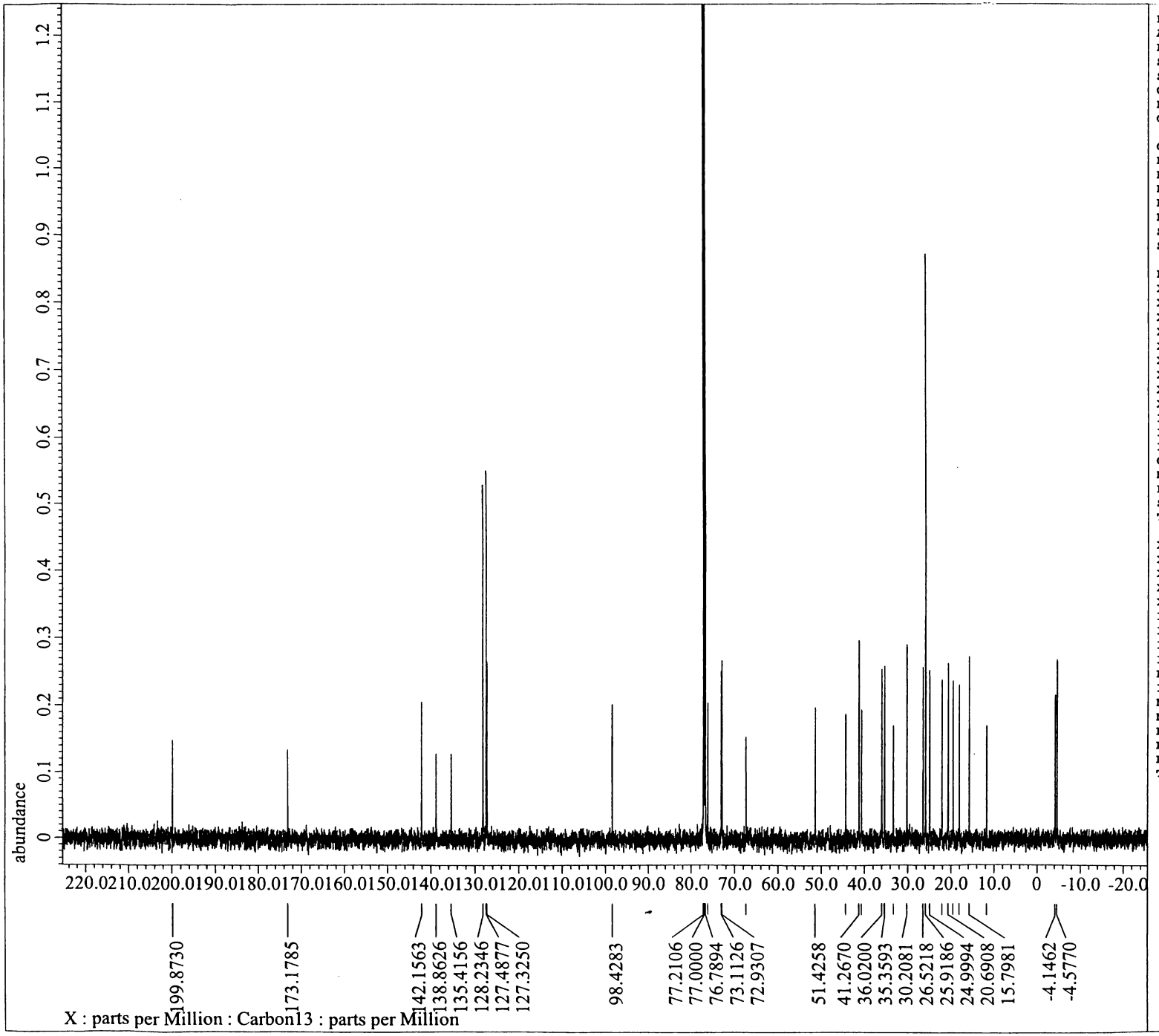
21-*epi*-4 (150 MHz, $CDCl_3$)



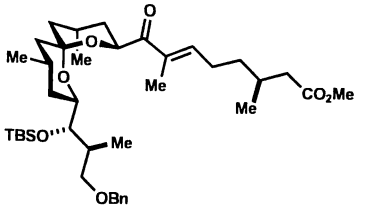
Filename	= C:\Users\delta\Documents\J
Author	= delta
Experiment	= proton.jxp
Sample_Id	= SN-III-135
Solvent	= CHLOROFORM-D
Creation_Time	= 25-AUG-2010 16:07:24
Revision_Time	= 25-AUG-2010 16:12:16
Current_Time	= 25-AUG-2010 16:12:38
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA600
Spectrometer	= DELTA2_NMR
Field_Strength	= 14.09636928[T] (600[MHz])
X_Acq_Duration	= 2.9097984[s]
X_Domain	= 1H
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.34366642[Hz]
X_Sweep	= 11.26126126[kHz]
X_Sweep_Clipped	= 9.00900901[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Mod_Return	= 1
Probe_Recovery	= 5[us]
Scans	= 8
Total_Scans	= 8
X_90_Width	= 12.4[us]
X_Acq_Time	= 2.9097984[s]
X_Angle	= 45[deg]
X_Atn	= 3[dB]
X_Pulse	= 6.2[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Recvr_Gain	= 36
Relaxation_Delay	= 1[s]
Repetition_Time	= 3.9097984[s]
Temp_Get	= 22.5[dC]



S5 (600 MHz, $CDCl_3$)




```
Filename          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = SN-III-136
Solvent           = CHLOROFORM-D
Creation_Time     =  2-SEP-2010 20:11:53
Revision_Time     =  2-SEP-2010 20:20:24
Current_Time      =  2-SEP-2010 20:20:40

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 14.09636928[T] (600[MHz])
X_Acq_Duration    = 0.69206016[s]
X_Domain          = 13C
X_Freq            = 150.91343039[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.44496109[Hz]
X_Sweep           = 47.34848485[kHz]
X_Sweep_Clipped   = 37.87878788[kHz]
Irr_Domain        = Proton
Irr_Freq          = 600.1723046[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Mod_Return        = 1
Probe_Recovery    = 20[us]
Scans             = 148
Total_Scans       = 148

X_90_Width        = 8.4[us]
X_Acq_Time        = 0.69206016[s]
X_Angle           = 30[deg]
X_Atn             = 6.4[dB]
X_Pulse           = 2.8[us]
Irr_Atn_Dec       = 18[dB]
Irr_Atn_Noe       = 18[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Recvr_Gain        = 56
Relaxation_Delay  = 2[s]
Repetition_Time   = 2.69206016[s]
Temp_Get          = 23.6[dC]
```



S5 (150 MHz, $CDCl_3$)