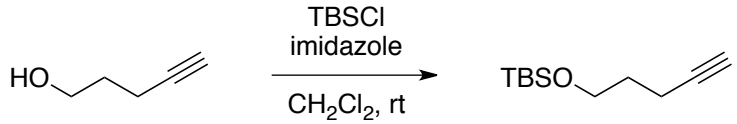
General Remarks: Nuclear magnetic resonance (^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz)) spectra were determined on a JEOL-ECS400 instrument. Chemical shifts for ^{1}H NMR are reported in parts per million downfields from tetramethylsilane (δ) as the internal standard and coupling constants are in hertz (Hz). The following abbreviations are used for spin multiplicity: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Chemical shifts for ^{13}C NMR were reported in ppm relative to the center line of a triplet at 77.16 ppm for deuteriochloroform. Infrared (IR) spectra were recorded on a JASCO FT/IR-410 Fourier Transform Infrared Spectrophotometer and were reported in wavenumbers (cm^{-1}). High resolution mass spectra (HRMS) were obtained on a JEOL JMS-T100LP AccuTOF LC-plus either in positive electrospray ionization (ESI) method or in positive direct analysis in real time (DART) ionization method, using sodium trifluoroacetate (TFANa) or polyethylene glycol (PEG) as the internal standard. Melting points (mp) were determined on a Yanaco Micro Melting Point Apparatus. Analytical thin layer chromatography (TLC) was performed on Merck precoated analytical plates, 0.25 mm thick, silica gel 60 F_{254}. Preparative TLC separations were performed on Merck analytical plates (0.25 or 0.50 mm thick) precoated with silica gel 60 F_{254} unless otherwise noted. Flash chromatography separations were performed on KANTO CHEMICAL Silica Gel 60 (spherical, 40-100 mesh) unless otherwise noted. Reagents were commercial grades and were used without any purification. Dehydrated tetrahydrofuran, diethyl ether, dichloromethane, and toluene were purchased from Kanto Chemicals Co., Inc., and were purified using a Glass Contour Solvent System. Dehydrated *N*,*N*-dimethylformamide was purchased from Kanto Chemicals Co., Inc. and stored over activated MS4A.* 3-Bromopropyl *tert*-butyldimethylsilyl ether,[1] (iodomethyl)tributylstannane,[2] and hydroxycarbonimidic dibromide (**40**)[3] were prepared according to the reported procedure. Cerium chloride was dried according to the reported procedure.[4] All reactions sensitive to oxygen or moisture were conducted under an argon atmosphere.

Silyl ether S1



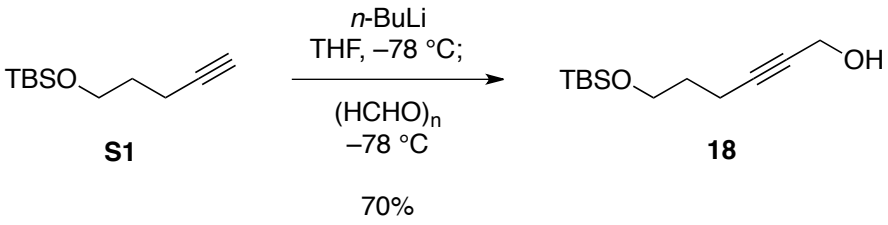
17 99% **S1**

To a solution of 4-pentyn-1-ol (**17**, 24.8 g, 295 mmol) and imidazole (21.1 g, 309 mmol) in dichloromethane (295 mL) was added *tert*-butyldimethylsilyl chloride (44.9 g, 298 mmol) at 0 °C,

* Molecular sieves were "activated" in the following manner: A round-bottom flask containing molecular sieves was heated in a regular microwave for 1.5-2.0 minute and the flask was immediately evacuated. When cooled to room temperature, the flask was backfilled with argon. The above procedure was repeated three times.

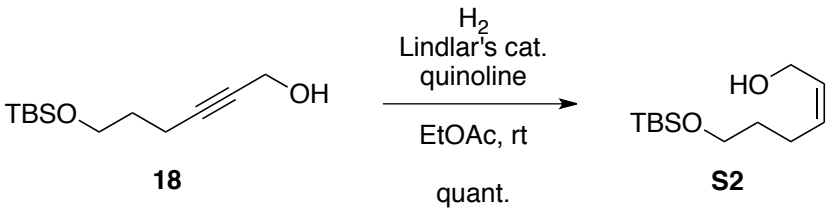
and the resulting mixture was stirred for 1 h. After completion of the reaction, the reaction mixture was partitioned between dichloromethane and water. The aqueous phase was extracted twice with dichloromethane. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (2% ethyl acetate/hexane) to afford **S1** (58.2 g, 293 mmol, 99.3 %) as a colorless oil. IR (film) 3313, 2954 2932, 2859, 1469, 1254, 1107, 981, 836, 777; ^{1}H NMR ($CDCl_3$) δ 3.70 (t, *J* = 6.5 Hz, 2H), 2.27 (td, *J* = 6.6, 2,7 Hz, 2H), 1.92 (t, *J* = 2.7 Hz, 1H), 1.72 (tt, *J* = 6.6, 6.5 Hz, 2H), 0.89 (s, 9H), 0.06 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 84.3 (C), 68.2 (CH), 61.4 (CH_2), 31.5 (CH_2), 25.9 (CH_3), 18.3 (C), 14.8 (CH_2), –5.4 (CH_3); HRMS (DART) 199.1518 (calcd for $C_{11}H_{22}OSi$ 199.1512).

Propargyl alcohol 18



To a solution of silyl ether **S1** (58.2 g, 293 mmol) in THF (293 mL) was added *n*-butyllithium (2.65 M solution in *n*-hexane, 133 mL, 352 mmol) at 0 °C slowly over 25 minutes. The mixture was stirred for 30 min, paraformaldehyde (9.70 g, 322 mmol) was added. The mixture was stirred for 6 h. After completion of the reaction, the reaction mixture was quenched with saturated aqueous ammonium chloride at 0 °C. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was distilled (1 mmHg, 105-115 °C) to afford **18** (47.2 g, 207 mmol, 70.6%) as a colorless oil. IR (film) 3346, 2861, 1728, 1470, 1390, 1252, 1112, 1071, 1012, 970, 848, 782; ^{1}H NMR ($CDCl_3$) δ 4.25 (dt, *J* = 6.0, 2.3 Hz, 2H), 3.68 (t, *J* = 6.9 Hz, 2H), 2.30 (tt, *J* = 6.9, 2.3 Hz, 2H), 1.71 (tt, *J* = 6.9, 6.9 Hz, 2H), 1.52 (td, *J* = 6.0, 2.3 Hz, 1H), 0.89 (s, 9H), 0.05 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 86.2 (C), 78.4 (C), 61.6 (CH_2), 51.4 (CH_2), 31.6 (CH_2), 25.9 (CH_3), 18.3 (C), 15.2 (CH_2), –5.3 (CH_3); HRMS (ESI+) 251.1443 (calcd for $C_{12}H_{24}NaO_2Si$ 251.1443).

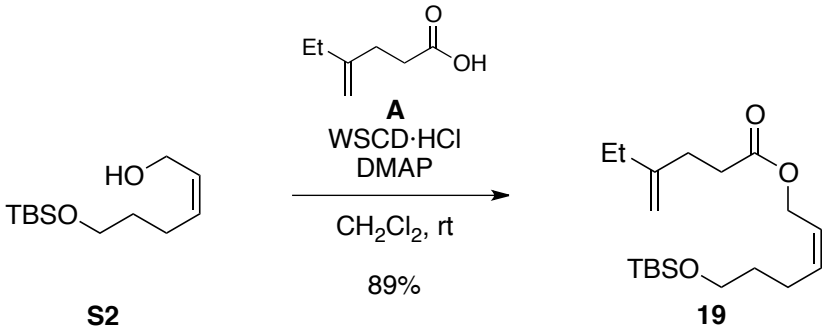
(*Z*)-Allyl alcohol S2



18 **S2**

To a solution of propargyl alcohol **18** (33.8 g, 147 mmol) in ethyl acetate (300 ml) were added quinoline (18.5 ml, 154 mmol) and Lindlar's catalyst (1.56 g, 14.7 mmol) at room temperature and then hydrogen gas was purged. After stirring for 3 h, the reaction mixture was filtered through a pad of celite. The filtrate was extracted five times with hydrochloric acid (1.0 M) aqueous. The combined organic phases were extracted with saturated aqueous sodium bicarbonate, washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (10% ethyl acetate/hexane) to afford **S2** (34.1 g, 100%) as a yellow oil. IR (film) 3347, 3015, 2932, 2859, 1469, 1389, 1254, 1103, 1042, 1009, 838, 777; ^{1}H NMR ($CDCl_3$) δ 5.70 (dt, *J* = 11.0, 6.2 Hz, 1H), 5.55 (dt, *J* = 11.0, 7.1 Hz, 1H), 4.16 (d, *J* = 6.2 Hz, 2H), 3.63 (t, *J* = 6.0 Hz, 2H), 2.19 (td, *J* = 7.3, 7.1 Hz, 2H), 1.76 (br s, 1H), 1.58 (m, 2H), 0.90 (s, 9H), 0.06 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 132.5 (CH), 129.2 (CH), 61.8 (CH_2), 58.2 (CH_2), 32.1 (CH_2), 25.9 (CH_3), 23.3 (CH_2), 18.3 (C), –5.3 (CH_3); HRMS (ESI+) 253.1595 (calcd for $C_{12}H_{26}NaO_2Si$ 253.1600).

Ester 19

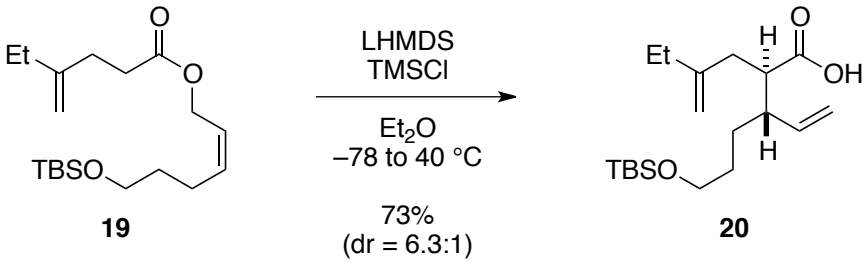


S2 **19**

To a solution of **S2** (26.0 g, 113 mmol) and **A** (15.2 g, 119 mmol) in dichloromethane (225 mL) was added WSCD·HCl (23.8 g, 124 mmol) and DMAP (689 mg, 5.64 mmol) at 0 °C, and the resulting mixture was stirred at room temperature for 13 h. After completion of the reaction, the reaction mixture was quenched with water. The solution was partitioned between dichloromethane and water. The aqueous phase was extracted twice with dichloromethane. The combined organic extracts were dried over sodium sulfate, filtered and concentrated in reduced pressure. The residue was purified by flash column chromatography (3% ethyl acetate/hexane) to afford **19** (34.5

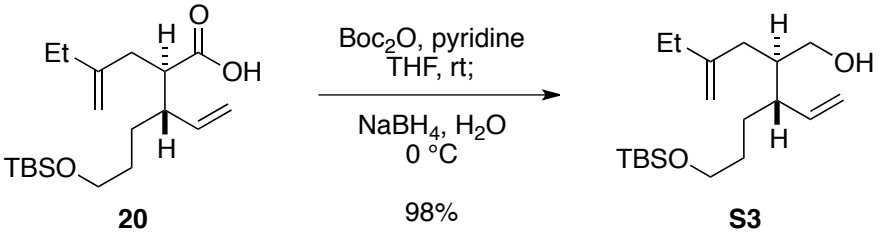
g, 101 mmol, 89.3%) as a colorless oil. IR (film) 2932, 2859, 1740, 1648, 1466, 1363, 1254, 1156, 777, 966, 891, 838; ^{1}H NMR ($CDCl_3$) δ 5.63 (dt, *J* = 11.0, 7.2 Hz, 1H), 5.55 (dt, *J* = 11.0, 6.6 Hz, 1H), 4.75 (s, 1H), 4.70 (s, 1H), 4.63 (d, *J* = 6.6 Hz, 2H) 3.61 (t, *J* = 6.0 Hz, 2H), 2.45 (t, *J* = 8.2 Hz, 2H), 2.35 (t, *J* = 8.2 Hz, 2H), 2.17 (dt, *J* = 7.6, 7.2 Hz, 2H), 2.03 (q, *J* = 7.6 Hz, 2H), 1.58 (m, 2H), 1.03 (t, *J* = 7.6 Hz, 3H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 173.3 (C), 149.7 (C), 134.7 (CH), 123.8 (CH), 108.1 (CH_2), 62.3 (CH_2), 60.3 (CH_2), 32.8 (CH_2), 32.5 (CH_2), 31.0 (CH_2), 28.9 (CH_2), 25.9 (CH_3), 23.9 (CH_2), 18.3 (C), 12.3 (CH_3), –5.3 (CH_3); HRMS (ESI+) 363.2323 (calcd for $C_{19}H_{36}NaO_3Si$ 363.2331).

Carboxylic acid 20



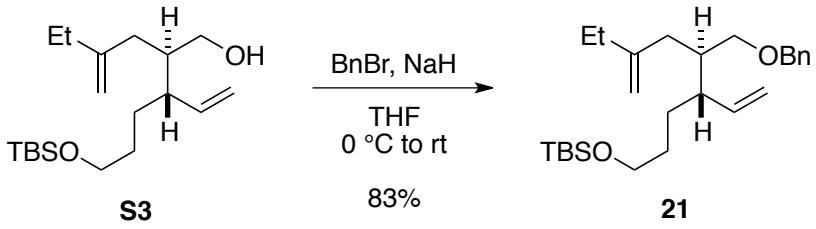
To a solution of **19** (31.5 g, 92.5 mmol) in diethyl ether (308 mL) was added trimethylsilyl chloride (35.1 mL, 278 mmol) at –78 °C slowly over 10 min. The mixture was stirred for 30 min, LHMDS (1.3 M solution in THF, 213 mL, 278 mmol) was added slowly over 35 min. After the mixture was stirred at –78 °C for 1 h, the cooling bath was removed and the mixture was stirred for an additional 1 h. Then the mixture was heated in an oil bath at 50 °C for 18 h. After completion of the reaction, the reaction mixture was quenched with saturated aqueous ammonium chloride. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (3 to 50% ethyl acetate/hexane) to afford **20** (23.3 g, 68.4 mmol, 73.9%, dr = 6.3:1) as a yellow oil. IR (film) 3442, 2085, 1642, 1468, 1290, 1250, 1110, 1000, 778; ^{1}H NMR ($CDCl_3$) δ 5.64 (ddd, *J* = 16.8, 10.0, 9.6 Hz 1H), 5.07 (dd, *J* = 10.0, 1.6 Hz, 1H), 5.02 (dd, *J* = 16.8, 1.6 Hz, 1H), 4.77 (s, 1H), 4.76 (s, 1H), 3.59 (t, *J* = 6.4 Hz, 2H), 2.61 (dt, *J* = 9.6, 6.0 Hz, 1H), 2.33 (dd, *J* = 10.1, 9.6 Hz, 1H), 2.28-2.17 (m, 2H), 2.09-1.94 (m, 2H), 1.67-1.51 (m, 2H), 1.48-1.41 (m, 1H), 1.37-1.29 (m, 1H), 1.01 (t, *J* = 8.0 Hz, 3H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 180.2 (C), 148.3 (C), 138.7 (CH), 117.0 (CH_2), 109.8 (CH_2), 62.9 (CH_2), 48.3 (CH), 46.2 (CH), 36.4 (CH_2), 30.4 (CH_2), 28.6 (CH_2), 28.4 (CH_2), 25.9 (CH_3), 18.3 (C), 12.2 (CH_3), –5.3 (CH_3); HRMS (ESI–) 339.2360 (calcd for $C_{19}H_{35}O_3Si$ 339.2356).

Primary alcohol S3



To a solution of **20** (12.4 g, 36.5 mmol) and pyridine (3.50 mL, 43.8 mmol) in THF (73.0 mL) was added di-*t*-butyl dicarbonate (8.70 g, 40.1 mmol) at room temperature, and the resulting mixture was stirred for 11 h. After completion of the reaction, the reaction mixture was added to a solution of sodium tetrahydroborate (7.60 g, 183 mmol) in water (73.0 mL) at 0 °C. After stirring for 2.5 h, the reaction mixture was quenched with saturated aqueous ammonium chloride. The resulting solution was extracted twice with ethyl acetate. The combined organic phases were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (3 to 10% ethyl acetate/hexane) to afford **S3** (11.8 g, 36.1 mmol, 98.9%) as a colorless oil. IR (film) 3436, 2931, 1640, 1317, 1251, 1098, 914, 889, 831, 779; ^{1}H NMR ($CDCl_3$) δ 5.63 (ddd, *J* = 17.2, 10.1, 9.1 Hz 1H), 5.07 (dd, *J* = 10.1, 1.8 Hz, 1H), 5.03 (dd, *J* = 17.2, 1.8 Hz, 1H), 4.80 (s, 1H), 4.78 (s, 1H), 3.66-3.53 (m, 4H), 2.17-1.98 (m, 5H), 1.72 (m, 1H), 1.55 (m, 2H), 1.46 (m, 1H), 1.35 (m, 1H), 1.33 (m, 1H), 1.03 (t, *J* = 7.8, 3H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 150.4 (C), 140.7 (CH), 116.0 (CH_2), 109.8 (CH_2), 63.6 (CH_2), 63.0 (CH_2), 45.2 (CH), 42.6 (CH), 36.7 (CH_2), 30.7 (CH_2), 28.3 (CH_2), 27.9 (CH_2), 26.0 (CH_3), 18.3 (C), 12.3 (CH_3), –5.3 (CH_3), –5.3 (CH_3); HRMS (ESI+) 349.2547 (calcd for $C_{19}H_{38}NaO_2Si$ 349.2539).

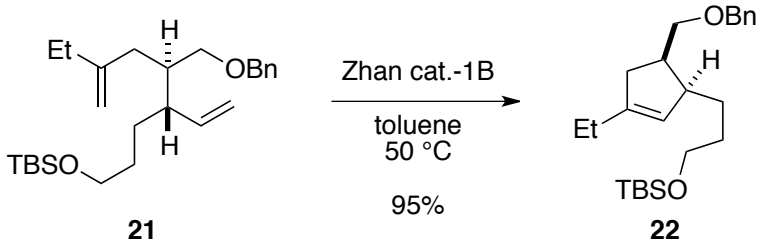
Benzyl ether 21



To a solution of **S3** (8.50 g, 26.0 mmol) in THF (87.0 mL) was added sodium hydride (60% in oil, 2.70 g, 67.5 mmol). The mixture was stirred for 30 min, benzyl bromide (3.70 mL, 31.2 mmol) was added at 0 °C slowly over 15 min. After stirring for 30 h, the reaction mixture was quenched with aqueous solution of ammonia. After stirring for 21 h, the resulting solution was extracted twice with ethyl acetate. The combined organic phases were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (1% diethyl ether/hexane) to afford **21** (9.03 g, 21.6 mmol, 83.0%) as a

colorless oil. IR (film) 3070, 3031, 2929, 2857, 1642, 1457, 1362, 1253, 1095, 1003, 913, 835, 776; ^{1}H NMR ($CDCl_3$) δ 7.32 (m, 5H), 5.58 (ddd, *J* = 17.0, 9.9, 8.0 Hz 1H), 5.02 (dd, *J* = 9.9, 2.0 Hz, 1H), 4.98 (dd, *J* = 17.0, 2.0 Hz, 1H), 4.77 (s, 1H), 4.70 (s, 1H), 4.45 (s, 2H), 3.58 (t, *J* = 6.0 Hz, 2H), 3.44 (dd, *J* = 9.2, 5.5 Hz, 1H), 3.31 (dd, *J* = 9.2, 6.0 Hz, 1H), 2.21-1.96 (m, 5H), 1.86 (m, 1H), 1.57-1.29 (m, 4H), 1.02 (t, *J* = 7.6 Hz, 3H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 149.8 (C), 140.3 (CH), 138.8 (C), 128.2 (CH), 127.4 (CH), 127.3 (CH), 115.9 (CH_2), 109.6 (CH_2), 73.0 (CH_2), 70.8 (CH_2), 63.2 (CH_2), 44.6 (CH), 40.4 (CH), 36.3 (CH_2), 31.1 (CH_2), 28.3 (CH_2), 27.9 (CH_2), 26.0 (CH_3), 18.4 (C), 12.3 (CH_3), –5.2 (CH_3); HRMS (ESI+) 439.3012 (calcd for $C_{26}H_{44}NaO_2Si$ 439.3008).

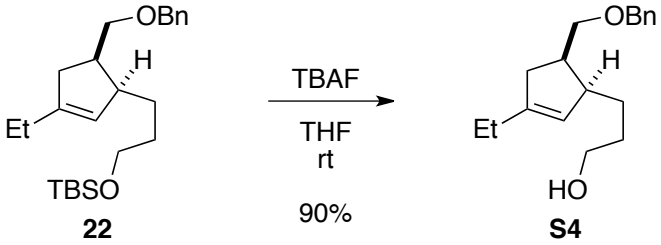
Cyclopentene 22



21 **22**

To a solution of **21** (9.00 g, 21.6 mmol) in toluene (72.0 mL) was added Zhan cat.-1B (**B**, 793 mg, 1.08 mmol), and the resulting mixture was stirred at 50 °C for 7 h. After completion of the reaction, the reaction mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography (3 to 5% diethyl ether/hexane) to afford **22** (8.01 g, 20.6 mmol, 95.3%) as a colorless oil. IR (film) 3063, 3032, 2927, 2854, 1455, 1362, 1253, 1093, 1028, 834, 777; ^{1}H NMR ($CDCl_3$) δ 7.33 (m, 5H), 5.37 (br d, *J* = 1.6 Hz 1H), 4.52 (d, *J* = 12.0 Hz, 1H), 4.49 (d, *J* = 12.0 Hz, 1H), 3.58 (t, *J* = 6.4 Hz, 2H), 3.54 (dd, *J* = 9.0, 6.8 Hz, 1H), 3.40 (dd, *J* = 9.0, 7.6 Hz, 1H) 2.63 (m, 2H), 2.26 (dd, *J* = 15.6, 7.6 Hz, 1H), 2.09 (m, 1H), 2.05 (q, *J* = 7.6 Hz, 2H), 1.60-1.06 (m , 4H), 1.02 (t, *J* = 7.6 Hz, 3H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 145.3 (C), 138.7 (C), 128.3 (CH), 127.7 (CH), 127.4 (CH), 126.1 (CH), 73.1 (CH_2), 71.1 (CH_2), 63.6 (CH_2), 45.7 (CH), 41.4 (CH), 37.8 (CH_2), 31.5 (CH_2), 26.1 (CH_2), 26.0 (CH_3), 24.3 (CH_2), 18.4 (C), 12.3 (CH_3), –5.2 (CH_3); HRMS (ESI+) 411.2679 (calcd for $C_{24}H_{40}NaO_2Si$ 411.2695).

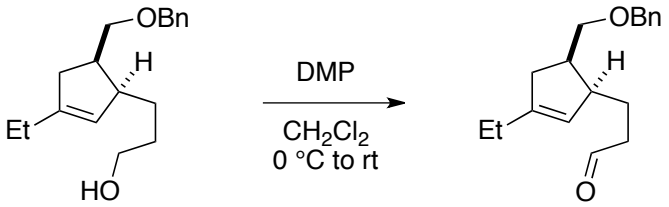
Primary alcohol S4



22 **S4**

To a stirred solution of **22** (8.01 g, 20.6 mmol) in THF (41.2 mL) was added tetra-*n*-butylammonium fluoride (1.0 M solution in THF, 30.9 mL, 30.9 mmol) at room temperature. After stirring for 3 h at room temperature, the reaction was quenched with water. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (5 to 50% ethyl acetate/hexane) to afford **S4** (5.10 g, 18.6 mmol, 90.2%) as a colorless oil. IR (film) 3327, 3045, 2838, 1705, 1648, 1453, 1364, 1202, 1125, 1028, 875, 824; ^{1}H NMR ($CDCl_3$) δ 7.37-7.27 (m, 5H), 5.37 (br d, *J* = 1.2 Hz, 1H), 4.51 (s, 2H), 3.61 (dt, *J* = 6.0, 5.5 Hz 2H), 3.55 (dd, *J* = 8.8, 6.8 Hz, 1H), 3.42 (dd, *J* = 8.8, 6.8 Hz, 1H), 2.64 (m, 2H), 2.27 (dd, *J* = 15.2, 7.6 Hz, 1H), 2.07 (m, 1H), 2.04 (q, *J* = 7.0 Hz, 2H), 1.65-1.56 (m, 2H), 1.53-1.43 (m, 2H), 1.18 (m, 1H), 1.02 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 145.6 (C), 138.6 (C), 128.3 (CH), 127.7 (CH), 127.5 (CH), 125.9 (CH), 73.1 (CH_2), 71.1 (CH_2), 63.3 (CH_2), 45.6 (CH), 41.4 (CH), 37.7 (CH_2), 31.4 (CH_2), 26.1 (CH_2), 24.3 (CH_2), 12.2 (CH_3); HRMS (ESI+) 297.1824 (calcd for $C_{18}H_{26}NaO_2$ 297.1831).

Aldehyde 23

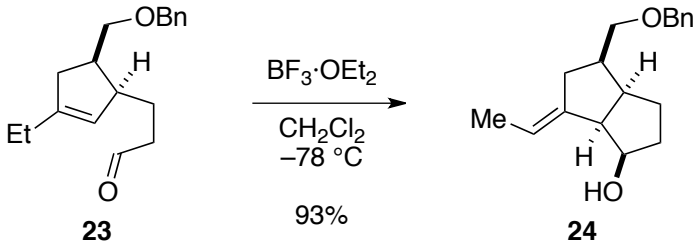


S4 **23**

To a stirred solution of **S4** (2.50 g, 9.11 mmol) in dichloromethane (30.0 mL) was added Dess-Martin periodinane (5.80 g, 13.7 mmol) at 0 °C. After stirring for 30 min, the cooling bath was removed and the mixture was stirred for an additional 1 h. The reaction was quenched with saturated aqueous sodium thiosulfate and sodium hydrogen carbonate. The solution was partitioned between dichloromethane and water. The aqueous phase was extracted twice with dichloromethane. The combined organic extracts were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (10% ethyl acetate/hexane) to afford **23** (2.24 g, 8.22 mmol, 90.2%) as a colorless oil. IR (film) 3033, 2874, 2841, 2717, 1722, 1496, 1453, 1365, 1205, 1076, 1027, 912; ^{1}H NMR ($CDCl_3$) δ 9.73 (br s, 1H), 7.37-7.27 (m, 5H), 5.32 (br s, 1H), 4.51 (s, 2H), 3.55 (dd, *J* = 8.0, 8.0 Hz, 1H), 3.45 (dd, *J* = 8.0, 6.8 Hz, 1H), 2.71-2.60 (m, 2H), 2.48-2.39 (m, 2H), 2.26 (dd, *J* = 15.6, 7.6 Hz, 1H), 2.07 (m, 1H), 2.03 (q, *J* = 7.2 Hz, 2H), 1.81 (m, 1H), 1.43 (m, 1H), 1.01 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$) δ 202.9 (CH), 146.6 (C), 138.4 (C), 128.3 (CH), 127.7 (CH), 127.6 (CH), 125.0 (CH), 73.1 (CH_2), 70.8 (CH_2), 45.2 (CH), 42.6 (CH_2), 41.5 (CH), 37.5 (CH_2), 24.3 (CH_2), 22.4 (CH_2), 12.2 (CH_3);

HRMS (ESI+) 295.1674 (calcd for $C_{18}H_{24}NaO_2$ 295.1674).

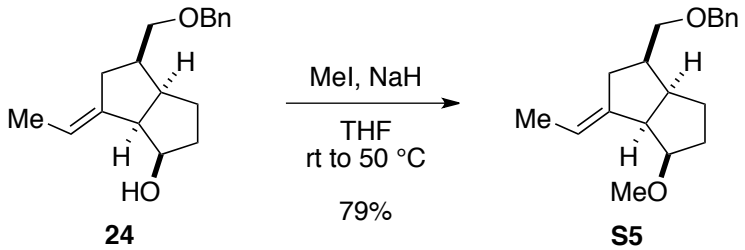
Secondary alcohol 24



23 **24**

To a stirred suspension containing of **23** (2.24 g, 8.22 mmol) in dichloromethane (30.0 mL) was added $BF_3{\cdot}OEt_2$ (4.60 mL, 36.6 mmol) at –78 °C. After stirring for 45 min at the same temperature, the reaction was quenched with methanol at –78 °C. The solution was partitioned between dichloromethane and water. The aqueous phase was extracted twice with dichloromethane. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue filtered by flash column chromatography (10% ethyl acetate/hexane) to afford **24** (2.09 g, 7.67 mmol, 93.3%) as a colorless oil. IR (film) 3531, 3031, 2841, 1452, 1366, 1237, 1125, 1071, 1011, 910, 785; ^{1}H NMR ($CDCl_3$) δ 7.37-7.27 (m, 5H), 5.31 (m, 1H), 4.52 (s, 2H), 4.14 (m, 1H), 3.53 (dd, *J* = 8.9, 8.2 Hz, 1H), 3.49 (dd, *J* = 8.9, 7.4 Hz, 1H), 3.08 (dd, *J* = 8.3, 7.3 Hz, 1H), 2.62 (dddd, *J* = 8.3, 8.2, 8.2, 8.2 Hz, 1H), 2.55 (dd, *J* = 15.1, 6.4 Hz, 1H), 2.29 (dddd, *J* = 8.2, 8.2, 7.4, 6.4, 1H), 1.90 (d, *J* = 2.8 Hz, 1H), 1.76 (m, 1H), 1.68 (m, 3H), 1.65 (m, 1H), 1.56 (m, 1H), 1.51 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 142.1 (C), 138.5 (C), 128.4 (CH), 127.6 (CH), 127.5 (CH), 119.6 (CH), 73.4 (CH), 73.2 (CH_2), 71.8 (CH_2), 54.6 (CH), 44.8 (CH), 41.2 (CH), 36.0 (CH_2), 32.8 (CH_2), 23.6 (CH_2), 15.1 (CH_3); HRMS (ESI+) 295.1681 (calcd for $C_{18}H_{24}NaO_2$ 295.1674).

Methyl ether S5



24 **S5**

To a solution of **24** (22.6 mg, 0.0829 mmol) in THF (0.25 mL) were added sodium hydride (60% in oil, 5.0 mg, 0.124 mmol) and methyl iodide (excess amount) at room temperature. After stirring for 3 h at 50 °C, the reaction mixture was quenched with aqueous solution of ammonia. The resulting solution was extracted twice with ethyl acetate. The combined organic phases were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by preparative TLC separation (20% ethyl acetate/hexane) to afford **S5**

(18.9 mg, 0.0659 mmol, 79.5%) as a colorless oil. IR (film) 2921, 1453, 1365, 1201, 1094, 1004; ^{1}H NMR ($CDCl_3$) δ 7.34-7.27 (m, 5H), 5.31 (m, 1H), 4.54 (d, *J* = 12.4 Hz, 1H), 4.50 (d, *J* = 12.4 Hz, 1H), 3.73 (ddd, *J* = 6.9, 6.4, 6.0 Hz, 1H), 3.51 (d, *J* = 6.9 Hz, 2H), 3.35 (s, 3H), 3.04 (m, 1H), 2.57 (dddd, *J* = 7.8, 7.4, 7.4, 7.3 Hz, 1H), 2.50 (dd, *J* = 14.6, 7.1 Hz, 1H), 2.29 (ddddd, *J* = 12.4, 7.4, 7.1, 6.9, 6.9 Hz, 1H), 1.76 (m, 1H), 1.64 (m, 2H), 1.61 (d, *J* = 9.6 Hz, 3H) 1.54-1.43 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 140.8 (C), 138.6 (C), 128.3 (CH), 127.6 (CH), 127.5 (CH), 118.3 (CH), 85.1 (CH), 73.1 (CH_2), 71.7 (CH_2), 57.8 (CH_3), 51.1 (CH), 42.8 (CH), 41.8 (CH), 32.9 (CH_2), 31.4 (CH_2), 21.8 (CH_2), 15.2 (CH_3); HRMS (ESI+) 309.1822 (calcd for $C_{19}H_{26}NaO_2$ 309.1831).

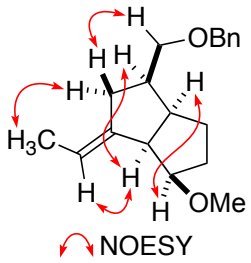
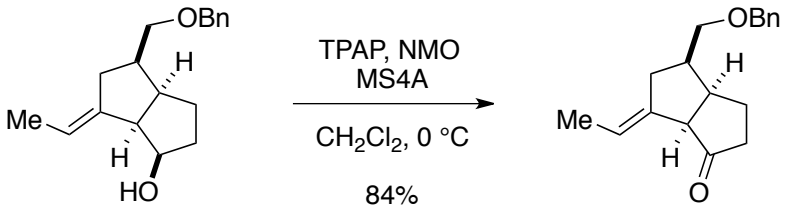


Fig. S1 NOESY correlations of **S5.**

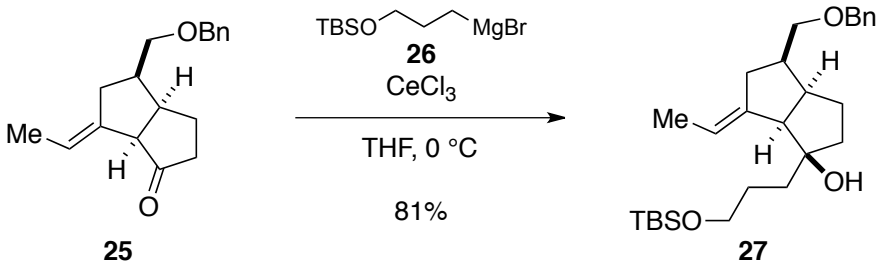
Ketone 25



24 **25**

To a stirred solution of **24** (1.98 g, 7.27 mmol), NMO (2.56 g, 21.8 mmol), and MS4A (3.64 g, 0.5 g/1 mmol of **24**) in dichloromethane (24.2 mL) was added TPAP (256 mg, 0.727 mmol) at 0 °C. After stirring for 1.5 h, the mixture was filtered by flash column chromatography (20% ethyl acetate/hexane) to afford **25** (1.67 g, 6.11 mmol, 84.0%) as a colorless oil. IR (film) 3453, 3032, 2842, 1722, 1496, 1453, 1408, 1368, 1205, 1125, 1079, 1027, 912, 803; ^{1}H NMR ($CDCl_3$) δ 7.39-7.29 (m, 5H), 5.56 (m, 1H), 4.56 (br d, *J* = 11.9 Hz, 1H), 4.54 (br d, *J* = 11.9 Hz, 1H), 3.54 (m, 2H), 3.05 (br s, 1H), 2.89 (m, 1H), 2.60-2.48 (m, 2H), 2.30 (dd, *J* = 18.3, 8.2 Hz, 1H), 2.20 (ddd, *J* = 18.3, 12.4, 8.7, Hz, 1H), 1.95 (m, 1H), 1.87 (m, 1H), 1.58 (dd, *J* = 7.3, 0.9 Hz, 3H), 1.49 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 217.7 (C), 138.4 (C), 136.8 (C), 128.4 (CH), 127.7 (CH), 120.4 (CH), 73.3 (CH_2), 70.8 (CH_2), 57.3 (CH), 44.3 (CH), 41.1 (CH), 38.9 (CH_2), 30.2 (CH_2), 21.1 (CH_2), 15.1 (CH_3); Two CH of the benzyl group are overlapping; HRMS (ESI+) 293.1521 (calcd for $C_{18}H_{22}NaO_2$ 293.1518).

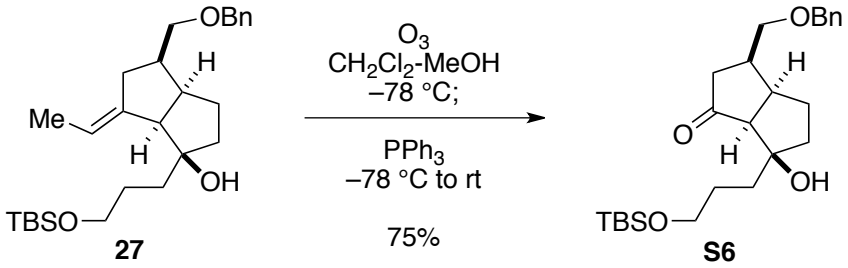
Tertiary alcohol 27



25 **27**

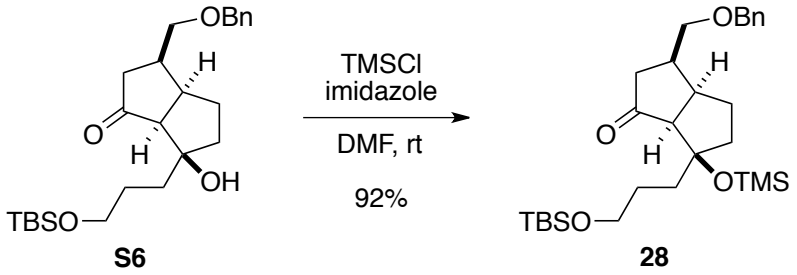
To a stirred suspension of magnesium (375 mg, 15.4 mmol) in tetrhydrofuran (20.0 mL) was added 1,2-dibromoethane (53.0 mL, 0.617 mmol) and a solution of 3-bromopropyl *tert*-butyldimethylsilyl ether (3.91 g, 15.4 mmol) in THF (10.0 mL) at room temperature. After magnesium was vanished, the mixture was added cerium chloride (0.5 M suspension in THF, 30.9 mL, 15.4 mmol) at 0 °C. After stirring for 15 min, to the resulting solution was added a solution of **25** (1.67 g, 6.17 mmol) in THF (12.3 mL) slowly over 10 min. After stirring for 45 min, the reaction was quenched with saturated aqueous ammonium chloride. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (10% ethyl acetate/hexane) to afford **27** (2.24 g, 5.04 mmol, 81.6%) as a colorless oil. IR (film) 3541, 3031, 2847, 1496, 1455, 1363, 1250, 1122, 1078, 1026, 937, 851, 781; ^{1}H NMR ($CDCl_3$) δ 7.37-7.27 (m, 5H), 5.30 (m, 1H), 4.52 (s, 2H), 3.63 (m, 2H), 3.53 (dd, *J* = 8.8, 7.6 Hz, 1H), 3.48 (dd, *J* = 8.8, 7.2 Hz, 1H), 2.78 (d, *J* = 9.0 Hz, 1H), 2.66 (ddd, *J* = 16.5, 9.0, 8.8, 1H), 2.52 (dd, *J* = 14.6, 6.4, 1H), 2.23 (m, 1H), 1.87 (m, 1H), 1.75 (m, 3H), 1.68 (d, *J* = 6.4, 3H), 1.61 (m, 3H), 1.54 (m, 2H), 1.38 (m, 1H), 0.90 (s, 9H), 0.05 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 142.7 (C), 138.5 (C), 128.3 (CH), 127.6 (CH), 127.5 (CH), 118.8 (CH), 81.2 (C), 73.1 (CH_2), 71.8 (CH_2), 63.6 (CH_2), 59.0 (CH), 45.6 (CH), 40.8 (CH), 39.5 (CH_2), 39.1 (CH_2), 32.1 (CH_2), 28.1 (CH_2), 25.9 (CH_3), 24.6 (CH_2), 18.3 (C), 15.1 (CH_3), –5.3 (CH_3); HRMS (ESI+) 467.2948 (calcd for $C_{27}H_{44}NaO_3Si$ 467.2957).

Hydroxy ketone S6



Ozone was carefully bubbled into a solution of **27** (635 mg, 1.43 mmol) in dichloromethane (5.00 mL) and methanol (5.00 mL) at –78 °C. After completion of the reaction, argon was bubbled into the reaction mixture to remove remaining ozone. After addition of triphenylphosphine (446 mg, 1.71 mmol), the reaction mixture was allowed to warm up to room temperature. The solvent was removed under reduced pressure. The residue was purified by flash column chromatography (20% ethyl acetate/hexane) to afford **S6** (466.5 mg, 1.08 mmol, 75.5%) as a colorless oil. IR (film) 3497, 2953, 2856, 1728, 1457, 1364, 1254, 1096, 1009, 980, 837, 778; ^{1}H NMR ($CDCl_3$) δ 7.38-7.28 (m, 5H), 4.53 (s, 2H), 3.64 (t, *J* = 6.0 Hz, 2H), 3.56 (d, *J* = 7.4 Hz, 2H), 2.90 (dt, *J* = 17.2, 9.2 Hz, 1H), 2.81 (s, 1H), 2.57 (dddt, *J* = 17.2, 12.8, 7.4, 6.0 Hz, 1H), 2.52 (d, *J* = 9.2 Hz, 1H), 2.27 (dd, *J* = 17.6, 7.4, 1H), 2.17 (dd, *J* = 17.6, 12.8, 1H), 1.84 (m, 1H), 1.79-1.67 (m, 6H), 1.49 (m, 1H), 0.89 (s, 9H), 0.05 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 219.3 (C), 138.5 (C), 128.4 (CH), 127.7 (CH), 127.6 (CH), 82.5 (C), 73.3 (CH_2), 71.5 (CH_2), 63.4 (CH_2), 62.4 (CH), 44.1 (CH), 41.3 (CH_2), 41.2 (CH_2), 39.7 (CH_2), 35.7 (CH), 27.7 (CH_2), 25.9 (CH_3), 25.0 (CH_2), 18.3 (C), –5.4 (CH_3); HRMS (ESI+) 455.2601 (calcd for $C_{25}H_{40}NaO_4Si$ 455.2594).

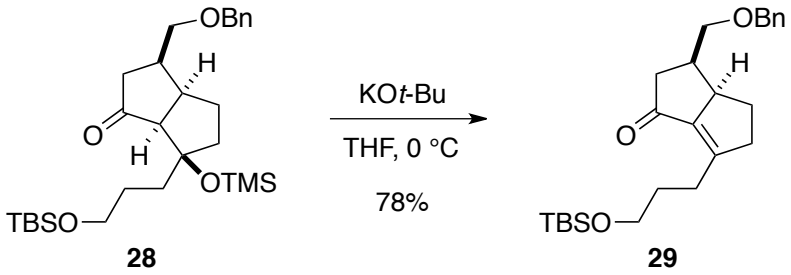
Silyl ether 28



To a solution of **S6** (1.90 g, 4.39 mmol) and imidazole (598 mg, 8.78 mmol) in *N,N*-dimethylformamide (15.0 mL) was added trimethylsilyl chloride (830 mL, 6.72 mmol) at room temperature, and the resulting mixture was stirred for 1.3 h. After completion of the reaction, the solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column

chromatography (10% ethyl acetate/hexane) to afford **28** (2.06 g, 4.08 mmol, 92.9%) as a colorless oil. IR (film) 2954, 2857, 1742, 1496, 1453, 1363, 1253, 1096, 1047, 839, 776; ^{1}H NMR ($CDCl_3$) δ 7.36-7.28 (m, 5H), 4.53 (s, 2H), 3.64 (m, 2H), 3.56 (m, 2H), 2.83 (m, 1H), 2.51 (d, *J* = 9.6 Hz, 1H), 2.46 (m, 1H), 2.11 (d, *J* = 10.5, 2H), 1.91-1.70 (m, 5H), 1.67-0.91 (m, 3H), 0.89 (s, 9H), 0.07 (s, 9H), 0.04 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 214.8 (C), 138.3 (C), 128.3 (CH), 127.6 (CH), 127.5 (CH), 87.2 (C), 73.1 (CH_2), 71.5 (CH_2), 63.2 (CH_2), 61.0 (CH), 43.1 (CH), 41.8 (CH_2), 41.4 (CH_2), 37.5 (CH_2), 35.4 (CH), 28.3 (CH_2), 25.9 (CH_3), 25.1 (CH_2), 18.3 (C), 2.2 (CH_3), –5.3 (CH_3); HRMS (ESI+) 527.2981 (calcd for $C_{28}H_{48}NaO_4Si_2$ 527.2989).

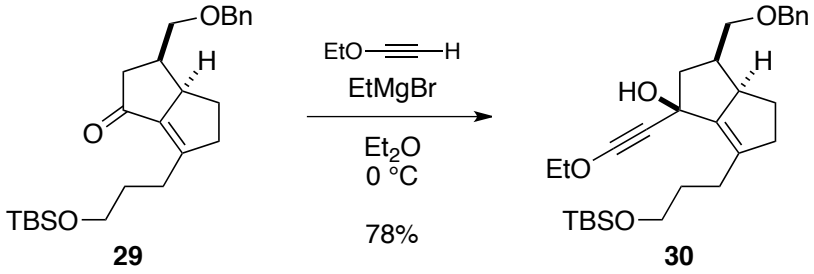
Unsaturated ketone 29



28 **29**

To a solution of potassium *t*-butoxide (1.0 M solution in THF, 20.0 mL, 20 mmol) was added a solution of **28** (1.00 g, 1.98 mmol) in THF (20.0 mL) at 0 °C slowly over 1 h, and the mixture was stirred for 15 min at the same temperature. After the ice bath was removed, the mixture was stirred for an additional 15 min at room temperature. After completion of the reaction, the solution was cooled to 0 °C and quenched with saturated aqueous ammonium chloride. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (20% ethyl acetate/hexane) to afford **29** (645 mg, 1.56 mmol, 78.7%) as a yellow oil. IR (film) 2930, 2856, 1706, 1652, 1457, 1363, 1254, 1100, 837, 776; ^{1}H NMR ($CDCl_3$) δ 7.35-7.25 (m, 5H), 4.45 (s, 2H), 3.59 (t, *J* = 6.9 Hz, 2H), 3.52 (m, 1H), 3.51 (dd, *J* = 9.2, 6.9, 1H), 3.27 (dd, *J* = 9.2, 6.9, 1H), 2.79 (m, 1H), 2.70 (dd, *J* = 17.9, 9.2, 1H), 2.57 (m, 1H), 2.54 (m, 1H), 2.50 (m, 1H), 2.49 (d, *J* = 17.9, 1H), 2.48 (m, 1H), 2.01 (dt, *J* = 12.2, 6.8, 1H), 1.82 (dt, *J* = 12.2, 9.2, 1H), 1.68 (tt, *J* = 14.7, 6.9, 2H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 202.0 (C), 154.3 (C), 138.9 (C), 138.2 (C), 128.4 (CH), 127.6 (CH), 127.6 (CH), 73.2 (CH_2), 70.4 (CH_2), 62.9 (CH_2), 51.0 (CH), 48.5 (CH_2), 41.2 (CH_2), 35.9 (CH), 31.0 (CH_2), 27.4 (CH_2), 25.9 (CH_3), 25.6 (CH_2), 18.3 (C), –5.3 (CH_3); HRMS (ESI+) 437.2476 (calcd for $C_{25}H_{38}NaO_3Si$ 437.2488).

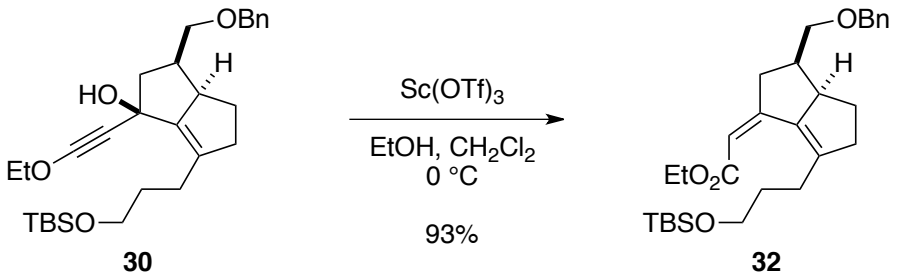
Tertiary alcohol 30



29 **30**

To a solution of ethoxyacetylene (40% solution in hexanes, 1.20 mL, 7.25 mmol) in diethyl ether (6.00 mL) was added ethylmagnesium bromide (3.0 M solution in diethyl ether, 1.45 mL, 4.3 mmol) at 0 °C. After the mixture was stirred for 15 min, a solution of **29** (600 mg, 1.45 mmol) in THF (4.00 mL) was added. After the mixture was stirred at 0 °C for another 45 min, the reaction was quenched with saturated aqueous ammonium chloride. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (10% ethyl acetate/hexane) to afford **30** (552 mg, 1.14 mmol, 78.6%) as a colorless oil. IR (film) 3438, 2835, 1473, 1390, 1200, 1122, 1074, 923, 859, 789; ^{1}H NMR ($CDCl_3$) δ 7.34-7.23 (m, 5H), 4.46 (s, 2H), 4.06 (q, *J* = 7.2 Hz, 2H), 3.85 (s, 1H), 3.66 (ddd, *J* = 10.6, 6.0, 5.7 Hz, 1H), 3.57 (m, 1H), 3.56 (dd, *J* = 9.4, 6.4 Hz, 1H), 3.50 (dd, *J* = 9.4, 4.6 Hz, 1H), 3.27 (m, 1H), 2.62-2.35 (m, 2H), 2.52 (dd, *J* = 14.2, 7.8 Hz, 1H), 2.38 (dd, *J* = 15.1, 8.7 Hz, 1H), 2.27-2.15 (m, 3H), 1.84 (ddd, *J* = 11.9, 6.8, 6.6 Hz, 1H), 1.68-1.57 (m, 3H), 1.34 (t, *J* = 7.2 Hz, 3H), 0.91 (s, 9H), 0.08 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 150.0 (C), 138.1 (C), 133.0 (C), 128.3 (CH), 127.8 (CH), 127.6 (CH), 92.1 (C), 74.3 (CH_2), 73.3 (CH_2), 71.4 (CH_2), 67.1 (C), 62.3 (CH_2), 53.3 (CH), 53.0 (CH_2), 41.5 (C), 39.3 (CH_2), 36.7 (CH), 30.2 (CH_2), 26.3 (CH_2), 26.0 (CH_3), 23.8 (CH_2), 18.4 (C), 14.4 (CH_3), –5.1 (CH_3), –5.2 (CH_3); HRMS (ESI+) 507.2901 (calcd for $C_{29}H_{44}NaO_4Si$ 507.2907).

Unsaturated ester 32

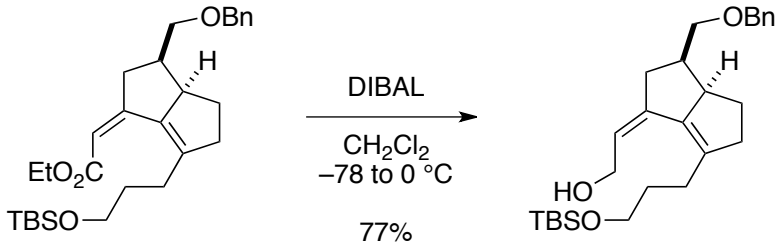


30 **32**

To a solution of **30** (532 mg, 1.10 mmol) in dichloromethane (3.60 mL) and ethanol (3.60 mL) was added scandium trifluoromethanesulfonate (27.1 mg, 0.0550 mmol) at 0 °C, and the resulting

mixture was stirred for 10 min. After completion of the reaction, the mixture was quenched with unsaturated aqueous sodium hydrogen carbonate. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by flash column chromatography (10% ethyl acetate/hexane) to afford **32** (501 mg, 1.03 mmol, 93.6%) as a colorless oil. IR (film) 2952, 2930, 2856, 1717, 1633, 1459, 1375, 1254, 1178, 1099, 1044; ^{1}H NMR ($CDCl_3$) δ 7.36-7.28 (m, 5H), 5.67 (s, 1H), 4.46 (s, 2H), 4.14 (q, *J* = 7.1 Hz, 2H), 3.56-3.52 (m, 2H), 3.46 (m, 1H), 3.44 (dd, *J* = 9.2, 7.4 Hz, 1H), 3.11 (dd, *J* = 9.2, 8.2 Hz, 1H), 2.78 (ddd, *J* = 17.8, 7.8, 2.3 Hz, 1H), 2.76 (m, 1H), 2.59 (ddd, *J* = 17.8, 2.3, 2.3 Hz, 1H), 2.51 (m, 1H), 2.31 (m, 1H), 2.08 (m, 2H), 1.92 (m, 1H), 1.78 (m, 1H), 1.68 (m, 1H), 1.53 (m, 1H), 1.26 (t, *J* = 7.1 Hz, 3H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 166.3 (C), 151.4 (C), 146.1 (C), 138.3 (C), 138.2 (C), 128.4 (CH), 127.7 (CH), 127.6 (CH), 111.4 (CH), 73.2 (CH_2), 71.0 (CH_2), 63.4 (CH_2), 59.7 (CH_2), 53.9 (CH), 41.1(CH_2), 40.3 (CH_2), 35.1 (CH), 30.2 (CH_2), 28.1 (CH_2), 26.0 (CH_3), 24.4 (CH_2), 18.3 (C), 14.4 (CH_3), –5.3 (CH_3), –5.3 (CH_3); HRMS (ESI+) 507.2922 (calcd for $C_{29}H_{44}NaO_4Si$ 507.2907).

Allyl alcohol 33

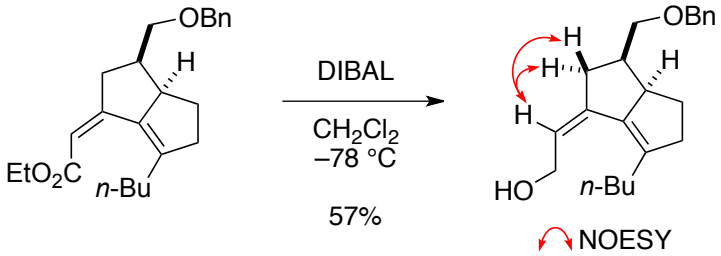


32 **33**

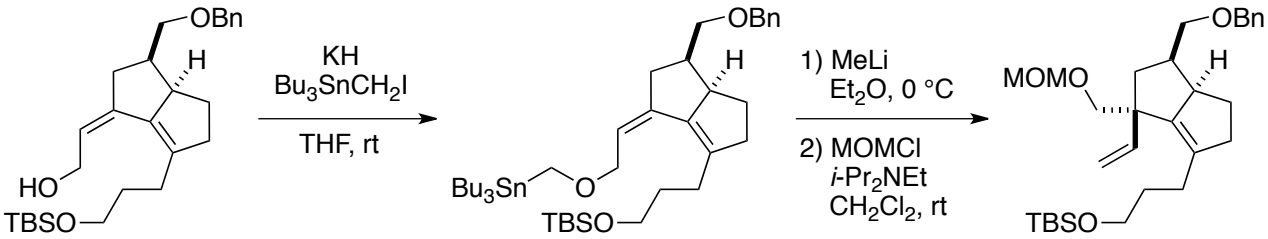
To a solution of DIBAL (1.0 M solution in toluene, 370 mL, 0.37 mmol) was added a solution of **32** (59.2 mg, 0.122 mmol) in dichloromethane (2.00 mL) at –78 °C slowly over 30 min, and the mixture was stirred for 30 min. After the cooling bath was removed, the mixture was stirred for an additional 30 min at 0 °C. After completion of the reaction, the solution was quenched with saturated aqueous potassium sodium tartrate. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by preparative TLC separation (50% ethyl acetate/hexane) to afford **33** (41.9 mg, 0.0946 mmol, 77.5%) as a colorless oil. IR (film) 2928, 1651, 1543, 1458, 1256, 1099, 996, 935, 836; ^{1}H NMR ($CDCl_3$) δ 7.36-7.27 (m, 5H), 5.39 (t, *J* = 7.3 Hz, 1H), 4.46 (s, 2H), 4.18 (br d, *J* = 7.3 Hz, 2H), 3.60 (m, 2H), 3.42 (dd, *J* = 8.7, 8.2 Hz, 1H), 3.26 (m, 1H), 3.16 (dd, *J* = 8.7, 7.3 Hz, 1H), 2.71 (dd, *J* = 15.8, 7.8 Hz, 1H), 2.69 (m, 1H), 2.36 (d, *J* = 15.8 Hz, 1H), 2.34 (m. 1H), 2.31 (m, 1H), 2.11 (m, 2H),

1.85 (m, 1H), 1.77 (m, 1H), 1.70 (m, 1H), 1.55 (m, 2H), 0.90 (s, 9H), 0.06 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 139.5 (C), 138.5 (C), 137.8 (C), 136.3 (C), 128.3 (CH), 127.7 (CH), 127.5 (CH), 120.5 (CH), 73.1 (CH_2), 71.6 (CH_2), 63.2 (CH_2), 61.9 (CH_2), 54.1 (CH), 40.5 (CH_2), 39.8 (CH_2), 35.3 (CH), 31.3 (CH_2), 27.1 (CH_2), 26.0 (CH_3), 25.5 (CH_2), 18.5 (C), –5.3 (CH_3), –5.3 (CH_3); HRMS (ESI+) 465.2794 (calcd for $C_{27}H_{42}NaO_3Si$ 465.2801).

The geometry of the trisubstituted olefin moiety was determined by a NOESY experiment of a related compound.



MOM ether 37



To a suspension of potassium hydride (30% in oil, 20.5 mg, 0.153 mmol) in THF (300 mL) were added a solution of **33** (64.6 mg, 0.153 mmol) in THF (300 mL) and a solution of Bu_3SnCH_2I (65.9 mg, 0.153 mmol) in THF (300 mL) at 0 °C, and the resulting mixture was stirred for 4 h at room temperature. After completion of the reaction, the solution was quenched at 0 °C carefully with water and partitioned between ethyl acetate and water. The organic extract was dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was filtered with silica gel (0 to 100 % ethyl acetate/hexane) and concentrated under reduced pressure to afford **34** as a colorless oil (120 mg). This oil was used for the next step without further purification. To a solution of methyllithium (1.17 M solution in diethyl ether, 280 mL, 0.322 mmol) was added a solution of **34** in diethyl ether (600 mL) at 0 °C slowly over 5 min. After the mixture was stirred for 1 h at the same temperature. The reaction mixture was quenched with saturated aqueous ammonium chloride. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure to afford alcohol **36** as a

colorless oil (100 mg), which was used for the next step without further purification. To a solution of **36** in dichloromethane (1.00 mL) was added *N, N*-diisopropylethylamine (69.0 mL, 0.400 mmol) and chloromethyl methyl ether (24.0 mL, 0.300 mmol), and the resulting mixture was stirred for 2 h at room temperature. After completion of the reaction, the solution was quenched with saturated aqueous ammonium chloride, and partitioned between ethyl acetate and water. The organic extract was dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by preparative TLC separation (25% ethyl acetate/hexane) to afford **37** (49.5 mg, 0.0988 mmol, 64.5% for 3 steps) as a colorless oil. IR (film) 2929, 2856, 1459, 1364, 1255, 1149, 1103, 1046, 919, 836, 774; ^{1}H NMR ($CDCl_3$) δ 7.36-7.27 (m, 5H), 5.95 (dd, *J* = 17.4, 10.8 Hz, 1H), 5.08 (br d, *J* = 17.4 Hz, 1H), 5.00 (br d, *J* = 10.8 Hz, 1H), 4.61 (s, 2H), 4.48 (d, *J* = 11.5 Hz, 1H), 4.43 (d, *J* = 11.5 Hz, 1H), 3.56 (t, *J* = 7.3 Hz, 2H), 3.55 (d, *J* = 8.0 Hz, 1H), 3.45 (d, *J* = 8.0 Hz, 1H), 3.43 (dd, *J* = 8.9, 6.4 Hz, 1H), 3.34 (s, 3H), 3.24 (m, 1H), 3.22 (dd, *J* = 8.9, 7.4 Hz, 1H), 2.55 (m, 1H), 2.35 (dd, *J* = 15.1, 8.7 Hz, 1H), 2.24 (m, 1H), 2.20 (dd, *J* = 12.6, 7.3 Hz, 1H), 2.13 (m, 2H), 2.04 (d, *J* = 12.6 Hz, 1H), 1.82 (ddd, *J* = 11.9, 6.9, 6.9 Hz, 1H), 1.59-1.49 (m, 3H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR ($CDCl_3$) δ 145.7 (C), 142.7 (CH), 138.6 (C), 134.1 (C), 128.3 (CH), 127.7 (CH), 127.4 (CH), 112.1 (CH_2), 96.6 (CH_2), 73.1 (CH_2), 73.1 (CH_2), 71.4 (CH_2), 63.4 (CH_2), 55.2 (CH_3), 55.0 (CH), 46.8 (C), 43.1 (CH_2), 39.9 (CH_2), 37.3 (CH), 31.6 (CH_2), 26.0 (CH_3), 25.5 (CH_2), 25.3 (CH_2), 18.3 (C), –5.3 (CH_3); HRMS (ESI+) 523.3194 (calcd for $C_{30}H_{48}NaO_4Si$ 523.3220).

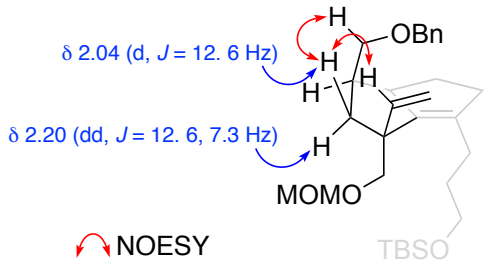
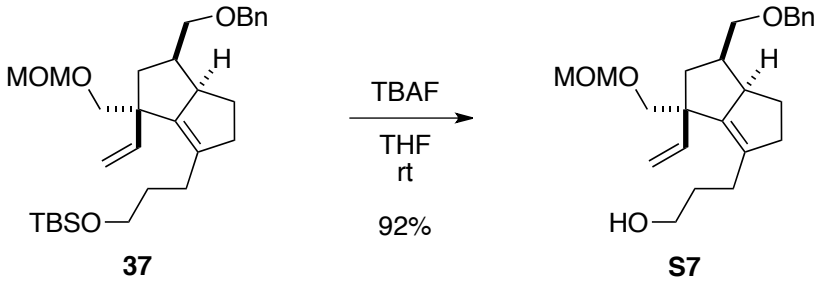


Fig. S2 NOESY correlations and selected coupling constants of **37**.

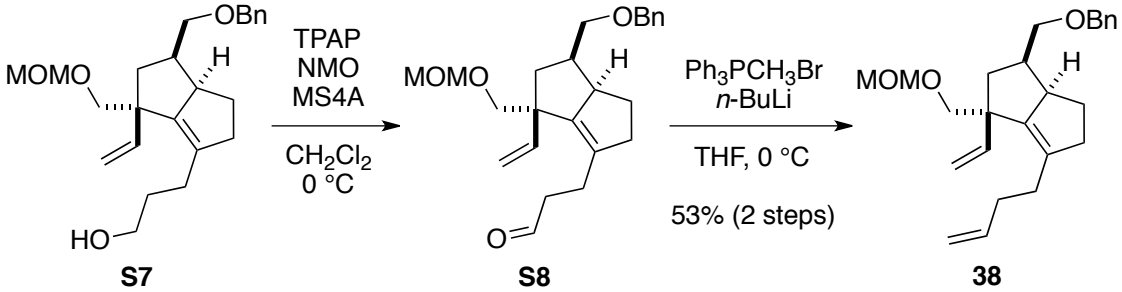
Primary alcohol S7



To a stirred solution of **37** (49.5 mg, 0.0988 mmol) in tetrahydrofuran (300 mL) was added

tetra-*n*-butylammonium fluoride (1.0 M solution in THF, 150 mL, 0.150 mmol) at room temperature. After stirring for 2 h at room temperature, the reaction was quenched with water. The solution was partitioned between ethyl acetate and water. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were washed with brine, dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by preparative TLC separation (50% ethyl acetate/hexane) to afford **S7** (35.5 mg, 0.0918 mmol, 92.9%) as a colorless oil. IR (film) 3436, 2935, 2861, 1634, 1452, 1368, 1211, 1149, 1106, 1044, 916; ^{1}H NMR ($CDCl_3$) δ 7.34-7.27 (m, 5H), 5.95 (dd, *J* = 17.4, 10.6 Hz, 1H), 5.10 (dd, *J* = 17.4, 0.9 Hz, 1H), 5.05 (dd, *J* = 10.6, 0.9 Hz, 1H), 4.63 (d, *J* = 6.9 Hz, 1H), 4.61 (d, *J* = 6.9 Hz, 1H), 4.48 (d, *J* = 11.9 Hz, 1H), 4.44 (d, *J* = 11.9 Hz, 1H), 3.55 (s, 2H), 3.54 (m, 2H), 3.45 (dd, *J* = 8.7, 7.6 Hz, 1H), 3.35 (s, 3H), 3.28 (m, 1H), 3.24 (dd, *J* = 9.2, 7.6 Hz, 1H), 2.58 (m, 1H), 2.43 (ddd, *J* = 14.2, 7.8, 7.3 Hz, 1H), 2.32-2.24 (m, 2H), 2.10 (m, 1H), 2.07 (dd, *J* = 13.8, 7.4 Hz, 1H), 1.90 (dd, *J* = 13.8, 3.7 Hz, 1H), 1.86 (m, 1H), 1.68-1.52 (m, 3H); ^{13}C NMR ($CDCl_3$) δ 146.4 (C), 142.5 (CH), 138.5 (C), 133.9 (C), 128.3 (CH), 127.7 (CH), 127.5 (CH), 112.4 (CH_2), 96.6 (CH_2), 73.1 (CH_2), 72.4 (CH_2), 71.5 (CH_2), 61.7 (CH_2), 55.4 (CH_3), 54.2 (CH), 47.1 (C), 43.5 (CH_2), 39.6 (CH_2), 36.7 (CH), 30.4 (CH_2), 25.7 (CH_2), 24.9 (CH_2); HRMS (ESI+) 409.2355 (calcd for $C_{24}H_{34}NaO_4$ 409.2355).

Triene 38

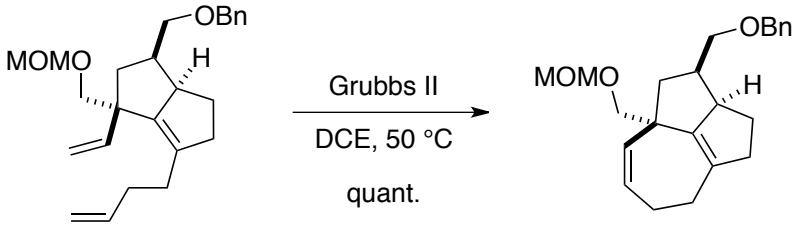


S7 **S8** **38**

To a suspension of alcohol **S7** (10 mg, 0.028 mmol) and MS4A (14 mg, 0.5 g/1 mmol of **S7**) in dichloromethane (0.25 mL) were added TPAP (1.0 mg, 0.0028 mmol) and NMO (10 mg, 0.085 mmol) at 0 °C. After completion of the reaction, the resulting solution was filtered over silica gel and concentrated under reduced pressure to afford **S8** as an oil, which was used for the next step without further purification. To a suspension of methyltriphenylphosphonium bromide (39 mg, 0.11 mmol) in THF (0.25 mL) was added *n*-butyllithium (2.4 M solution in *n*-hexane, 9.0 mL, 0.11 mmol) at 0 °C. After 15 minutes, to the resulting solution was added a solution of aldehyde **S8** (8.4 mg, 0022 mmol) in THF (0.25 mL) at 0 °C. After completion of the reaction, the solution was partitioned between ethyl acetate and aqueous solution of ammonium chloride. The aqueous phase was extracted twice with ethyl acetate. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by

preparative TLC separation (20% ethyl acetate/hexane) to afford **38** (5.9 mg, 0.015 mmol, 53% for 2 steps) as a colorless oil. IR (film) 2928, 2844, 1638, 1452, 1367, 1211, 1149, 1106, 1043, 913; ^{1}H NMR ($CDCl_3$) δ 7.34-7.26 (m, 5H), 5.94 (dd, *J* = 17.2, 10.8 Hz, 1H), 5.78 (ddt, *J* = 17.2, 10.7, 6.4 Hz, 1H), 5.08 (dd, *J* = 17.2, 1.4 Hz, 1H), 5.01 (dd, *J* = 10.8, 1.4 Hz, 1H), 4.99 (dd, *J* = 17.2, 1.1 Hz, 1H), 4.92 (dd, *J* = 10.7, 1.1 Hz, 1H), 4.61 (s, 2H), 4.48 (d, *J* = 11.9 Hz, 1H), 4.43 (d, *J* = 11.9 Hz, 1H), 3.55 (d, *J* = 9.2 Hz, 1H), 3.47 (d, *J* = 9.2 Hz, 1H), 3.45 (dd, *J* = 9.2, 9.0 Hz, 1H), 3.34 (s, 3H), 3.26 (m, 1H), 3.23 (dd, *J* = 9.0, 7.3 Hz, 1H), 2.55 (m, 1H), 2.35 (dd, *J* = 14.6, 8.7 Hz, 1H), 2.29-2.19 (m, 4H), 2.12-2.08 (m, 2H), 2.03 (d, *J* = 13.3 Hz, 1H), 1.82 (ddd, *J* = 11.9, 6.9, 6.2 Hz, 1H), 1.53 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 146.1 (C), 142.7 (CH), 138.8 (CH), 138.5 (C), 133.8 (CH), 128.3 (CH), 127.7 (CH), 127.5 (CH), 114.3 (CH_2), 112.1 (CH_2), 96.7 (CH_2), 73.2 (CH_2), 73.1 (CH_2), 71.3 (CH_2), 55.2 (CH), 55.0 (CH), 46.8 (C), 43.3 (CH_2), 39.8 (CH_2), 37.3 (CH_3), 32.5 (CH_2), 28.5 (CH_2), 25.5 (CH_2); HRMS (ESI+) 405.2414 (calcd for $C_{25}H_{34}NaO_3$ 405.2405).

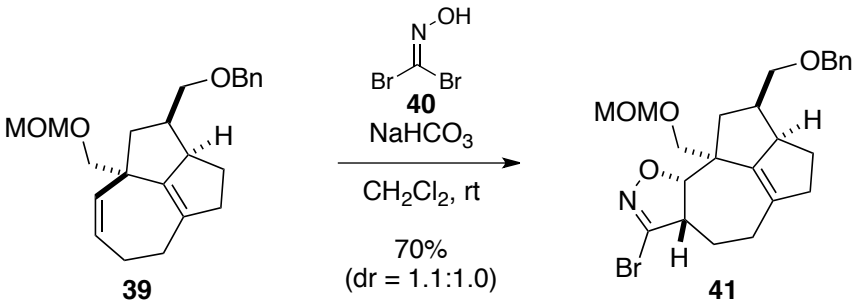
Tricyclic compound 39



38 **39**

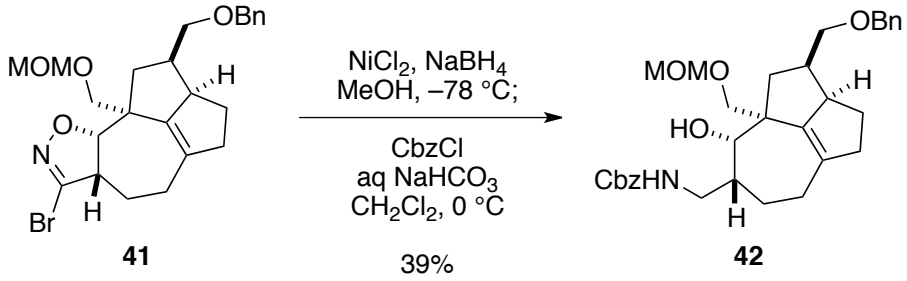
To a solution of **38** (5.4 mg, 0.014 mmol) in 1,2-dichloroethane (0.25 mL) was added Grubbs 2nd catalyst (0.4 mg, 0.0017 mmol), and the resulting mixture was stirred at 50 °C for 25 min. After completion of the reaction, the reaction mixture was concentrated under reduced pressure. The residue was purified by preparative TLC separation (25% ethyl acetate/hexane) to afford **39** (4.8 mg, 100%) as a colorless oil. IR (film) 2927, 2853, 1452, 1364, 1149, 1107, 1043; ^{1}H NMR ($CDCl_3$) δ 7.36-7.27 (m, 5H), 5.77 (ddd, *J* = 11.0, 8.3, 5.5 Hz, 1H), 5.62 (dd, *J* = 11.0, 2.3 Hz, 1H), 4.61 (s, 2H), 4.47 (s, 2H), 3.61 (d, *J* = 9.2 Hz, 1H), 3.47 (d, *J* = 9.2 Hz, 1H), 3.41 (dd, *J* = 8.7, 8.7 Hz, 1H), 3.35 (m, 1H), 3.34 (s, 3H), 3.27 (dd, *J* = 8.7, 6.4 Hz, 1H), 2.62-2.51 (m, 2H), 2.42 (m, 1H), 2.24 (m, 1H), 2.23 (m, 1H), 2.11 (m, 2H), 2.04 (m, 1H), 1.91 (ddd, *J* = 12.6, 6.9, 6.4 Hz, 1H), 1.62 (dddd, *J* = 12.6, 9.6, 9.2, 9.2 Hz, 1H), 1.44 (dd, *J* = 13.1, 7.8, 1H); ^{13}C NMR ($CDCl_3$) δ 144.0 (C), 138.6 (C), 136.9 (CH), 136.4 (C), 129.3 (CH), 128.3 (CH), 127.7 (CH), 127.5 (CH), 96.5 (CH_2), 73.1 (CH_2), 72.3 (CH_2), 72.1 (CH_2), 55.1 (CH_3), 52.1 (CH), 46.9 (C), 43.9 (CH_2), 40.3 (CH_2), 35.7 (CH), 28.3 (CH_2), 26.2 (CH_2), 25.3 (CH_2); HRMS (ESI+) 377.2081 (calcd for $C_{23}H_{30}NaO_3$ 377.2093).

Isoxazoline 41



To a suspension of tricyclic compound **39** (7.0 mg, 0.0197 mmol) and sodium hydrogen carbonate (10.0 mg) in dichloromethane (0.30 mL) were added oxime **40** (12.0 mg, 0.0592 mmol) and the resulting mixture was stirred at room temperature for 48 h. The reaction was quenched with saturated aqueous ammonium chloride. The solution was partitioned between dichloromethane and water. The aqueous phase was extracted twice with dichloromethane. The combined organic extracts were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by preparative TLC separation (20% ethyl acetate/hexane) to afford **41** (3.4 mg, 0.00714 mmol, 36%) and its diastereomer (3.2 mg, 0.00671 mmol, 34%) as a colorless oil respectively. Less polar isomer (**41**): IR (film) 2919, 2853, 1719, 1453, 1404, 1280, 1110, 1042, 914; ^{1}H NMR ($CDCl_3$) δ 7.36-7.28 (m, 5H), 4.60 (d, *J* = 11.6, 1H), 4.55 (s, 2H), 4.48 (d, *J* = 11.9, 1H), 4.41 (d, *J* = 11.9, 1H), 3.58 (d, *J* = 9.8, 1H), 3.48 (d, *J* = 9.8, 1H), 3.42-3.33 (m, 3H), 3.31 (s, 3H), 3.26 (dd, *J* = 9.2, 5.5 Hz, 1H), 2.60 (m, 1H), 2.34 (m, 1H), 2.26 (m, 3H), 2.22 (m, 1H), 2.01 (m, 1H), 1.95 (m, 2H), 1.88 (dddd, *J* = 13.1, 8.5, 8.5, 1.6 Hz, 1H), 1.65 (ddd, *J* = 18.5, 13.1, 9.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 143.8 (C), 143.6 (C), 138.4 (C), 135.0 (C), 128.4 (CH), 127.8 (CH), 127.6 (CH), 96.6 (CH_2), 88.8 (CH), 73.1 (CH_2), 71.6 (CH_2), 70.4 (CH_2), 55.3 (CH_3), 54.4 (CH), 53.8 (CH), 46.9 (C), 44.4 (CH_2), 41.2 (CH_2), 36.9 (CH), 28.1 (CH_2), 24.8 (CH_2), 24.2 (CH_2); HRMS (ESI+) 498.1248 (calcd for $C_{24}H_{30}BrNNaO_4$ 498.1256). More polar isomer: IR (film) 2919, 1454, 1404, 1150, 1105, 1041, 917; ^{1}H NMR ($CDCl_3$) δ 7.36-7.28 (m, 5H), 4.80 (d, *J* = 9.8 Hz, 1H), 4.61 (s, 2H), 4.51 (d, *J* = 11.6, 1H), 4.46 (d, *J* = 11.6, 1H), 3.67 (d, *J* = 9.8, 1H), 3.53-3.38 (m, 3H), 3.36 (s, 3H), 3.34-3.30 (m, 2H), 2.50 (m, 1H), 2.31 (m, 2H), 2.20 (m, 2H), 2.07 (m, 2H), 1.98 (m, 1H), 1.86 (m, 2H), 1.76 (ddd, *J* = 17.8, 12.8, 8.7, 1H); ^{13}C NMR ($CDCl_3$) δ 143.2 (C), 141.0 (C), 138.7 (C), 137.1 (C), 128.3 (CH), 127.8 (CH), 127.4 (CH), 96.6 (CH_2), 86.8 (CH), 73.2 (CH_2), 72.4 (CH_2), 71.9 (CH_2), 55.5 (CH_3), 53.6 (CH), 53.2 (CH), 46.4 (C), 40.8 (CH_2), 40.5 (CH_2), 36.0 (CH), 25.5 (CH_2), 25.0 (CH_2), 24.8 (CH_2); HRMS (ESI+) 498.1274 (calcd for $C_{24}H_{30}BrNNaO_4$ 498.1256).

Aminoalcohol 42



To a solution of isoxazoline **41** (less polar isomer, 5.2 mg, 0.0109 mmol) in methanol (0.3 mL) were added nickel chloride (8.2 mg, 0.0633 mmol) and sodium tetrahydroborate (11.2 mg, 0.266 mmol) at –78 °C. After completion of the reaction, the reaction mixture were added dichloromethane (0.2 mL), aqueous sodium hydrogen carbonate (0.2 mL), and benzyl chloroformate (25 mL, 0.177 mmol) at 0 °C. After stirring for 1.5 h, the reaction mixture was quenched with saturated aqueous ammonium chloride. The resulting solution was extracted twice with ethyl acetate. The combined organic phases were dried over sodium sulfate, filtered and concentrated under reduced pressure. The residue was purified by preparative TLC separation (50% ethyl acetate/hexane) to afford **42** (2.3 mg, 0.0043 mmol, 39%) as a colorless oil. IR (film) 3349, 2923, 2858, 1720, 1518, 1448, 1250, 1108, 1038; ^{1}H NMR ($CDCl_3$) δ 7.35-7.27 (m, 10H), 5.36 (t, *J* = 5.7 Hz, 1H), 5.08 (s, 2H), 4.63 (d, *J* = 6.6 Hz, 1H), 4.60 (d, *J* = 6.6 Hz, 1H), 4.46 (s, 2H), 3.83 (dd, *J* = 5.5, 4.8 Hz, 1H), 3.75 (d, *J* = 9.8 Hz, 1H), 3.54 (d, *J* = 9.8 Hz, 1H), 3.48 (d, *J* = 5.5 Hz, 1H), 3.42-3.33 (m, 2H), 3.37 (s, 3H), 3.30-3.23 (m, 3H), 2.53 (m, 1H), 2.37 (m, 1H), 2.34 (d, *J* = 11.9 Hz, 1H), 2.24-2.17 (m, 2H), 2.10-1.97 (m, 2H), 1.90 (ddd, *J* = 12.4, 7.8, 5.2 Hz, 1H), 1.79 (m, 1H), 1.66-1.59 (m, 2H), 1.40 (dd, *J* = 11.9, 6.9 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 156.6 (C), 143.4 (C), 138.4 (C), 136.8 (C), 136.3 (C), 128.5 (CH), 128.4 (CH), 128.1 (CH), 128.0 (CH), 127.8 (CH), 127.6 (CH), 96.8 (CH_2), 78.4 (CH), 73.2 (CH_2), 72.0 (CH_2), 70.9 (CH_2), 66.5 (CH_2), 55.8 (CH_3), 52.0 (CH), 49.2 (C), 44.3 (CH_2), 43.0 (CH_2), 41.9 (CH), 40.8 (CH_2), 35.0 (CH), 27.2 (CH_2), 27.0 (CH_2), 25.8 (CH_2); HRMS (ESI+) 558.2843 (calcd for $C_{32}H_{41}NNaO_6$ 558.2831).

References

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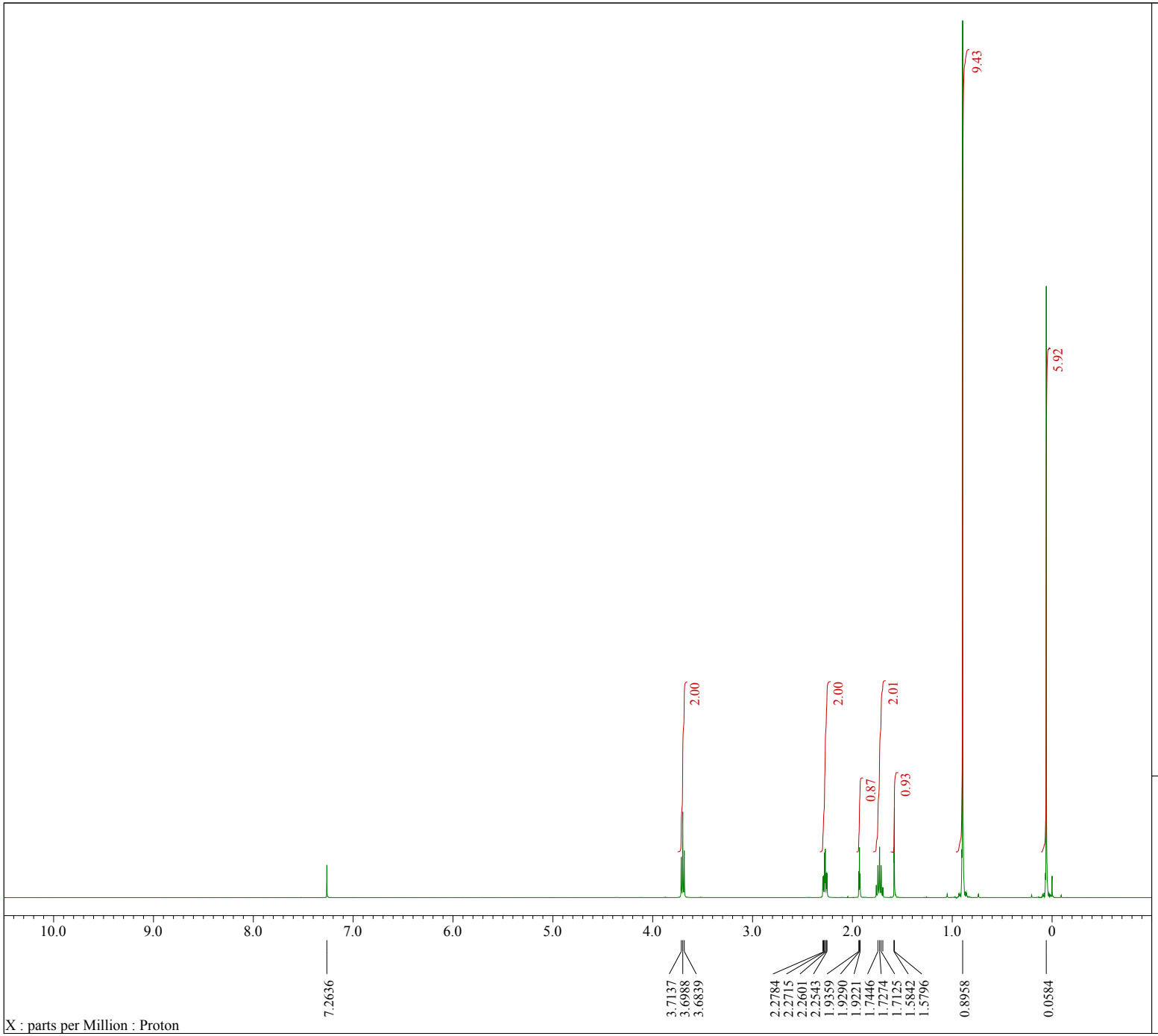


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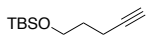
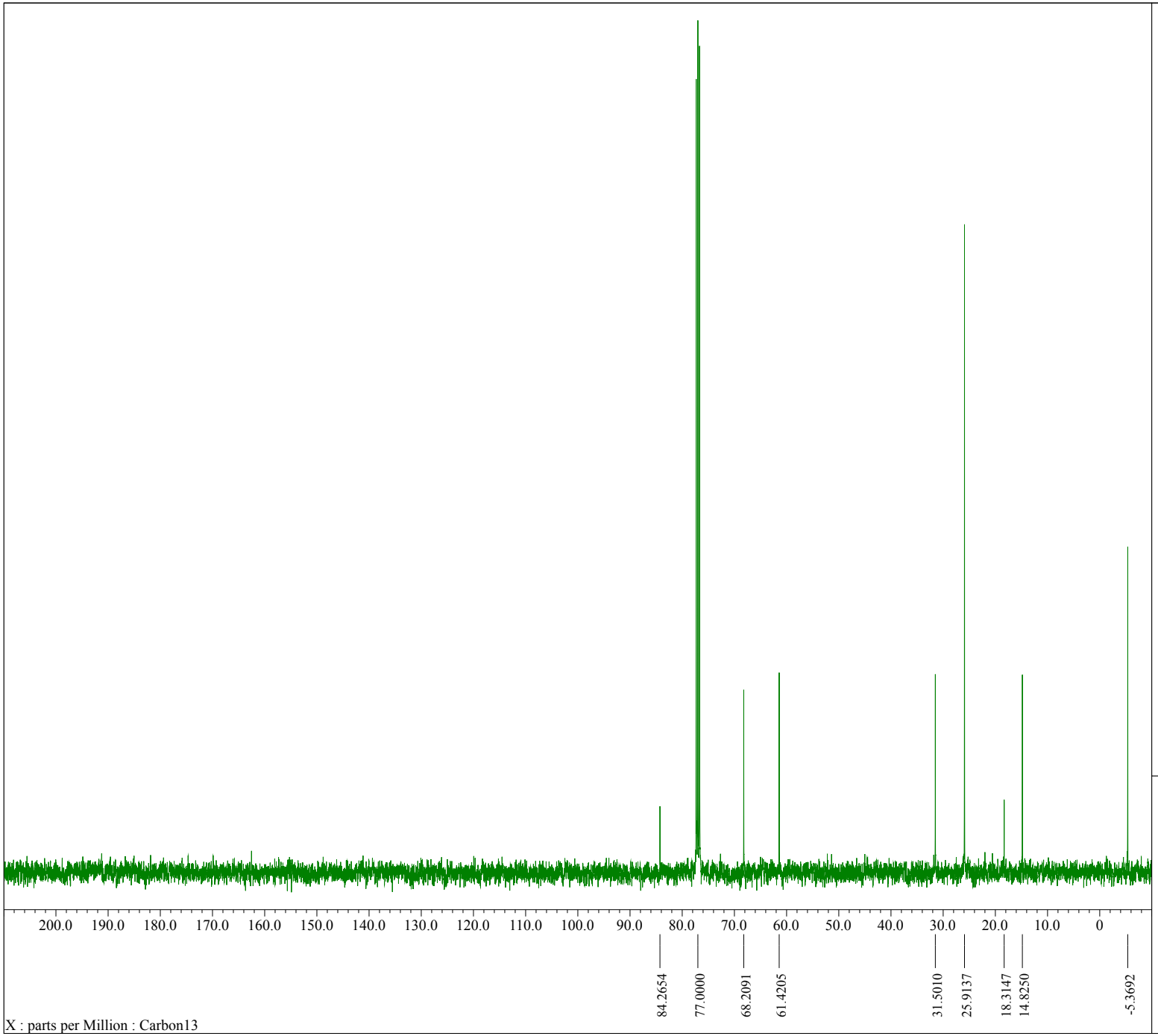


S1
^{1}H-NMR
(400 MHz, $CDCl_3$)

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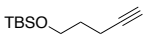
Filename          = yk05352data_bcm-2-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk05352data
Solvent           = CHLOROFORM-D
Creation_Time     =  4-SEP-2016 19:01:14
Revision_Time     = 20-FEB-2018 16:15:41
Current_Time      = 20-FEB-2018 16:29:20

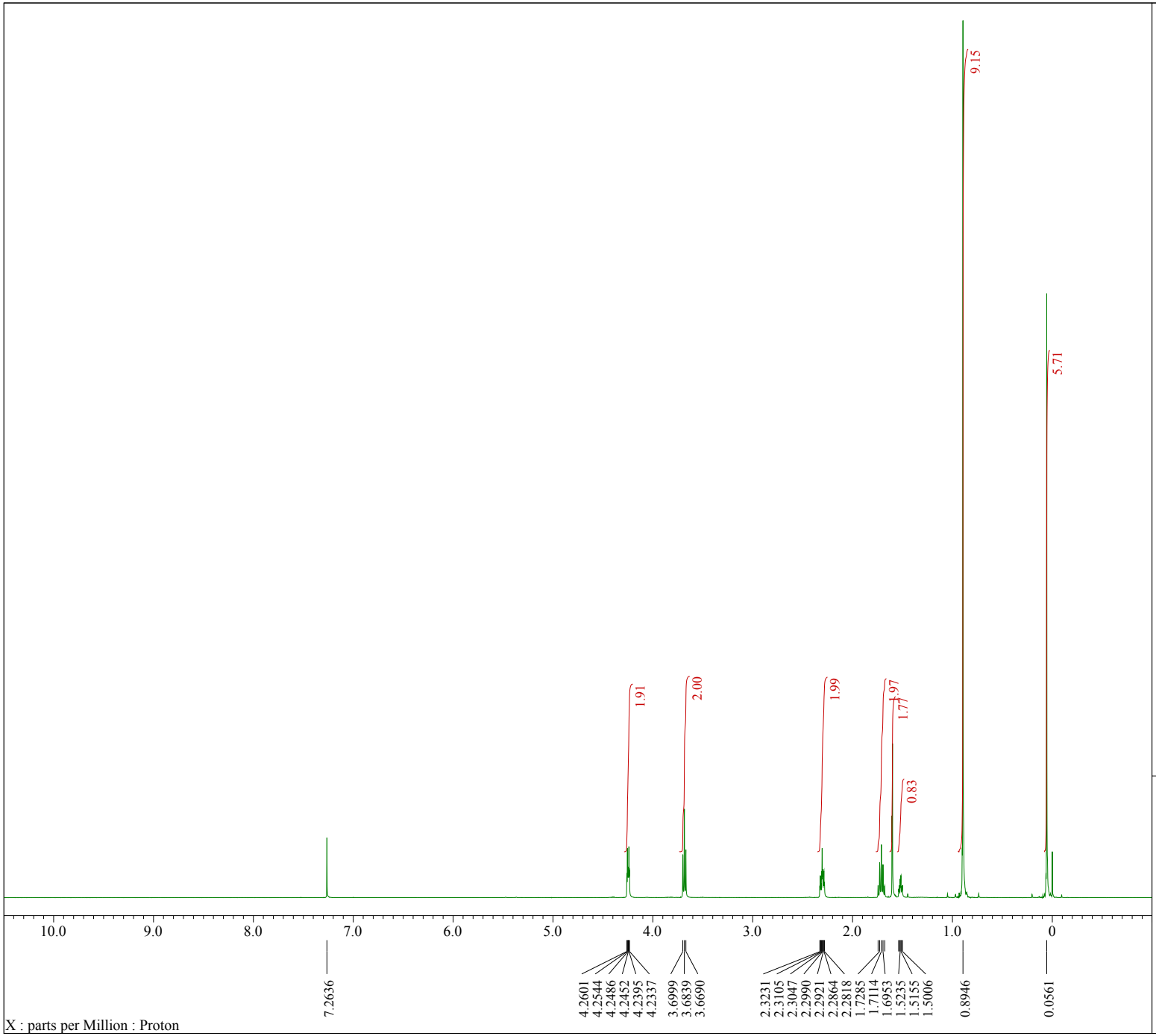
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 24[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



S1
^{13}C-NMR
(100 MHz, $CDCl_3$)

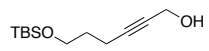



```
Filename          = yk05376data_non-data-2-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk05376data
Solvent           = CHLOROFORM-D
Creation_Time     =  4-SEP-2016 19:10:00
Revision_Time     = 20-FEB-2018 19:32:25
Current_Time      = 20-FEB-2018 19:33:35

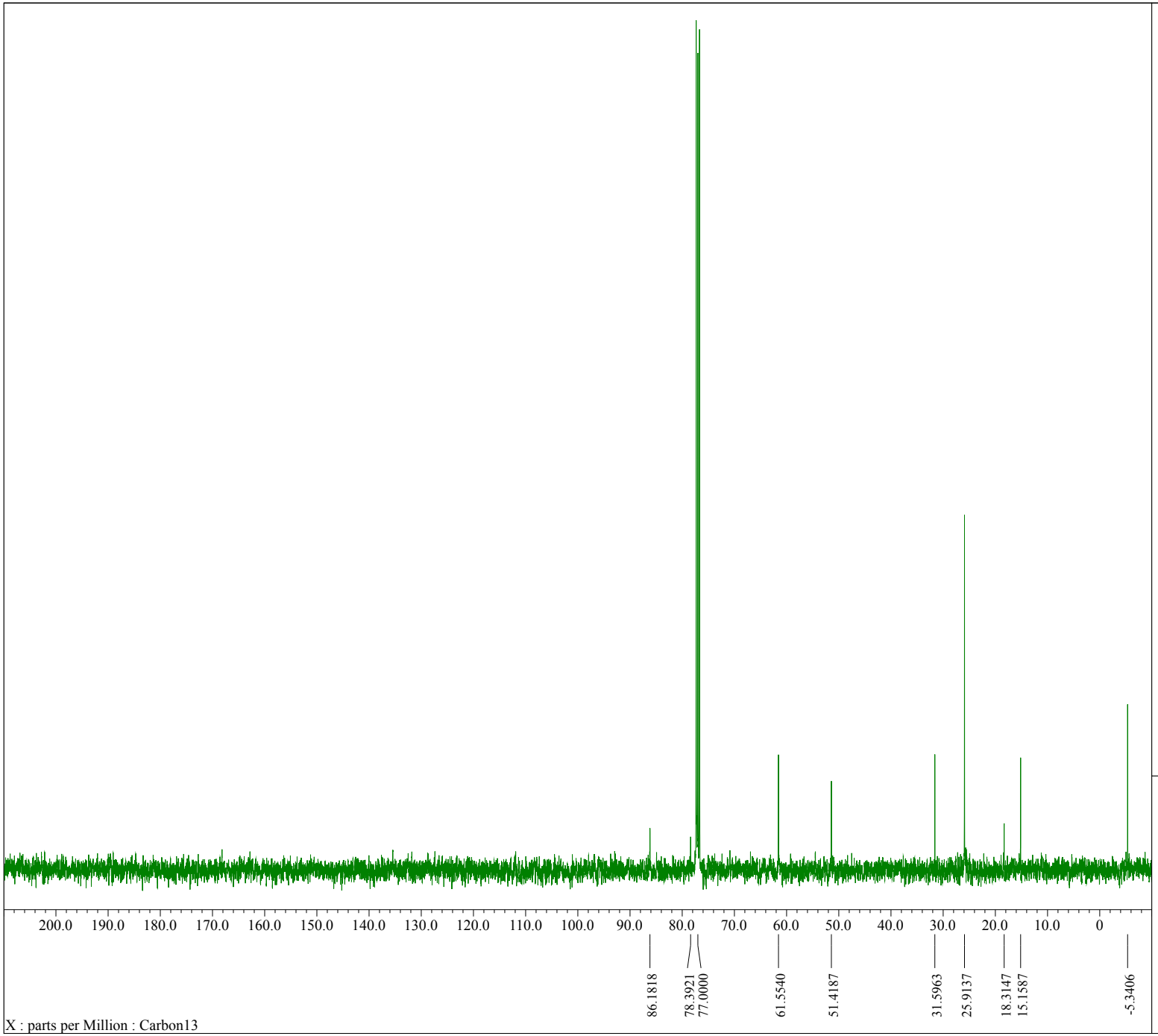
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 23.7[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



18
^{1}H-NMR
(400 MHz, $CDCl_3$)

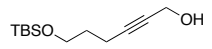



```
Filename          = yk05376data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk05376data
Solvent           = CHLOROFORM-D
Creation_Time     =  4-SEP-2016 19:32:13
Revision_Time     = 20-FEB-2018 16:32:03
Current_Time      = 20-FEB-2018 16:32:34

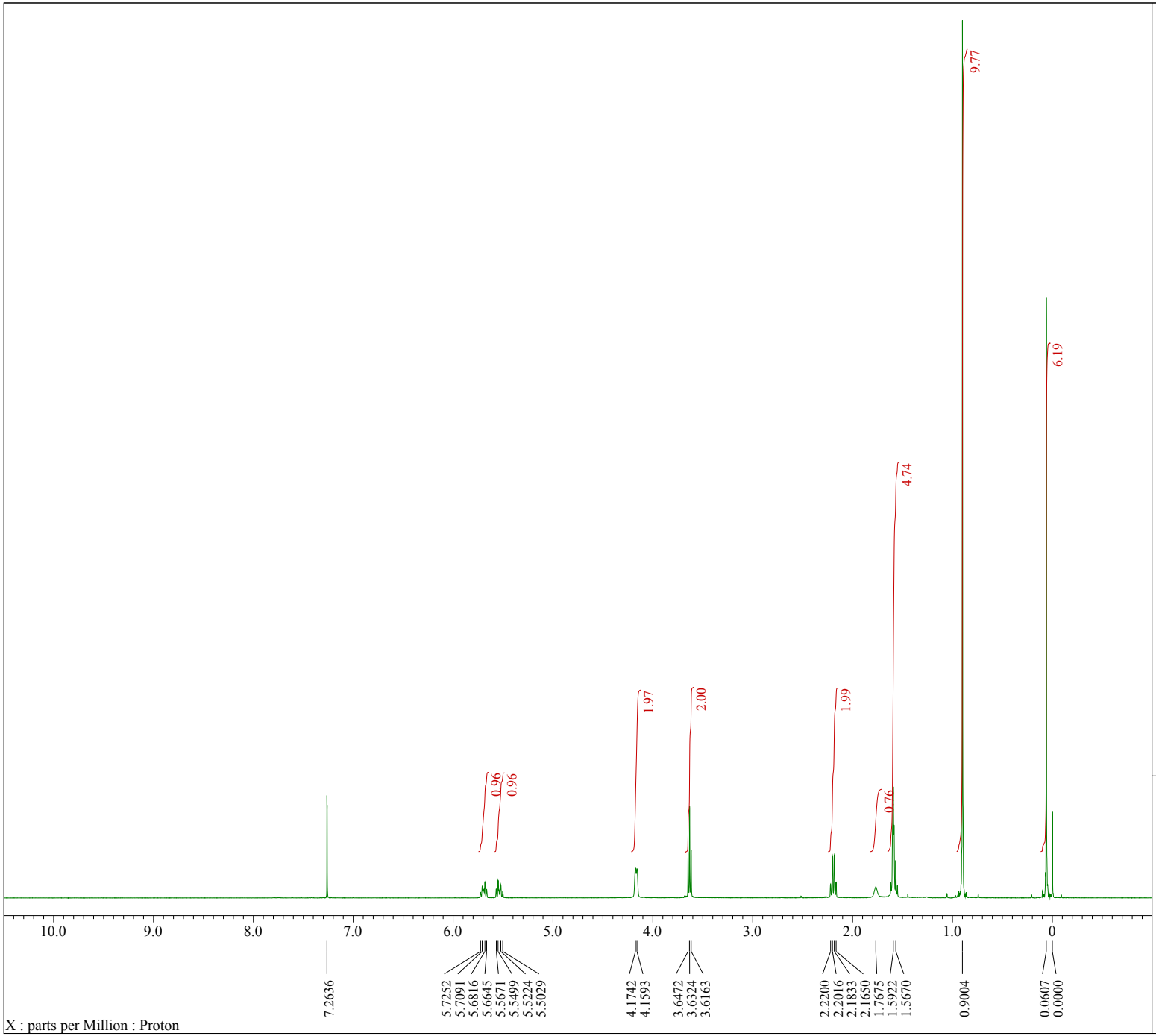
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.9[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



18
^{13}C-NMR
(100 MHz, $CDCl_3$)

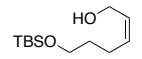



```
Filename          = yk05378_non-data-3-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk05378
Solvent           = CHLOROFORM-D
Creation_Time     = 19-SEP-2016 17:28:08
Revision_Time     = 20-FEB-2018 19:35:53
Current_Time      = 20-FEB-2018 19:36:34

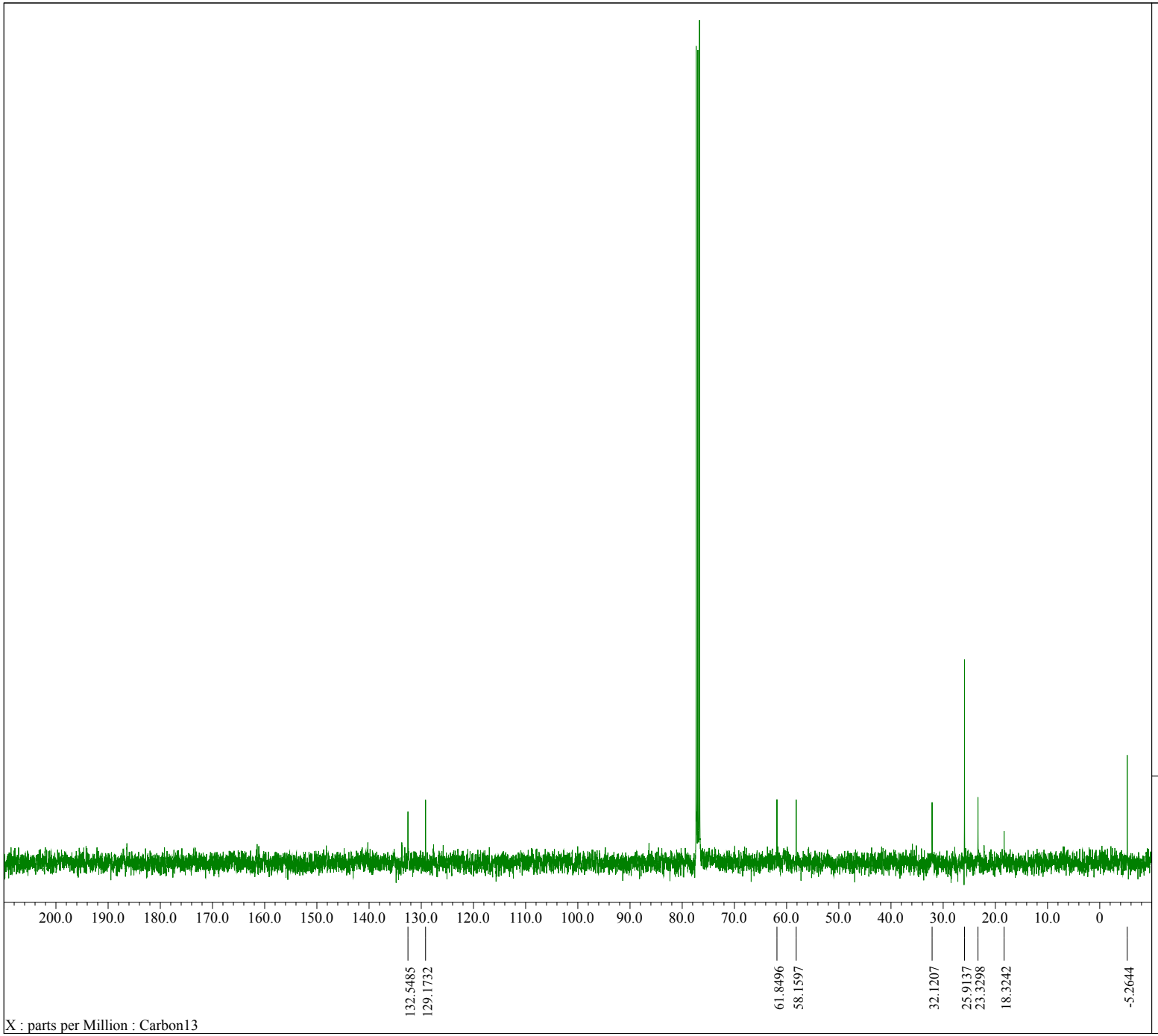
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 23.7[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



S2
^{1}H-NMR
(400 MHz, $CDCl_3$)

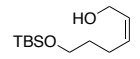



```
Filename          = yk05378_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk05378
Solvent           = CHLOROFORM-D
Creation_Time     = 19-SEP-2016 17:29:51
Revision_Time     = 20-FEB-2018 16:34:39
Current_Time      = 20-FEB-2018 16:35:08

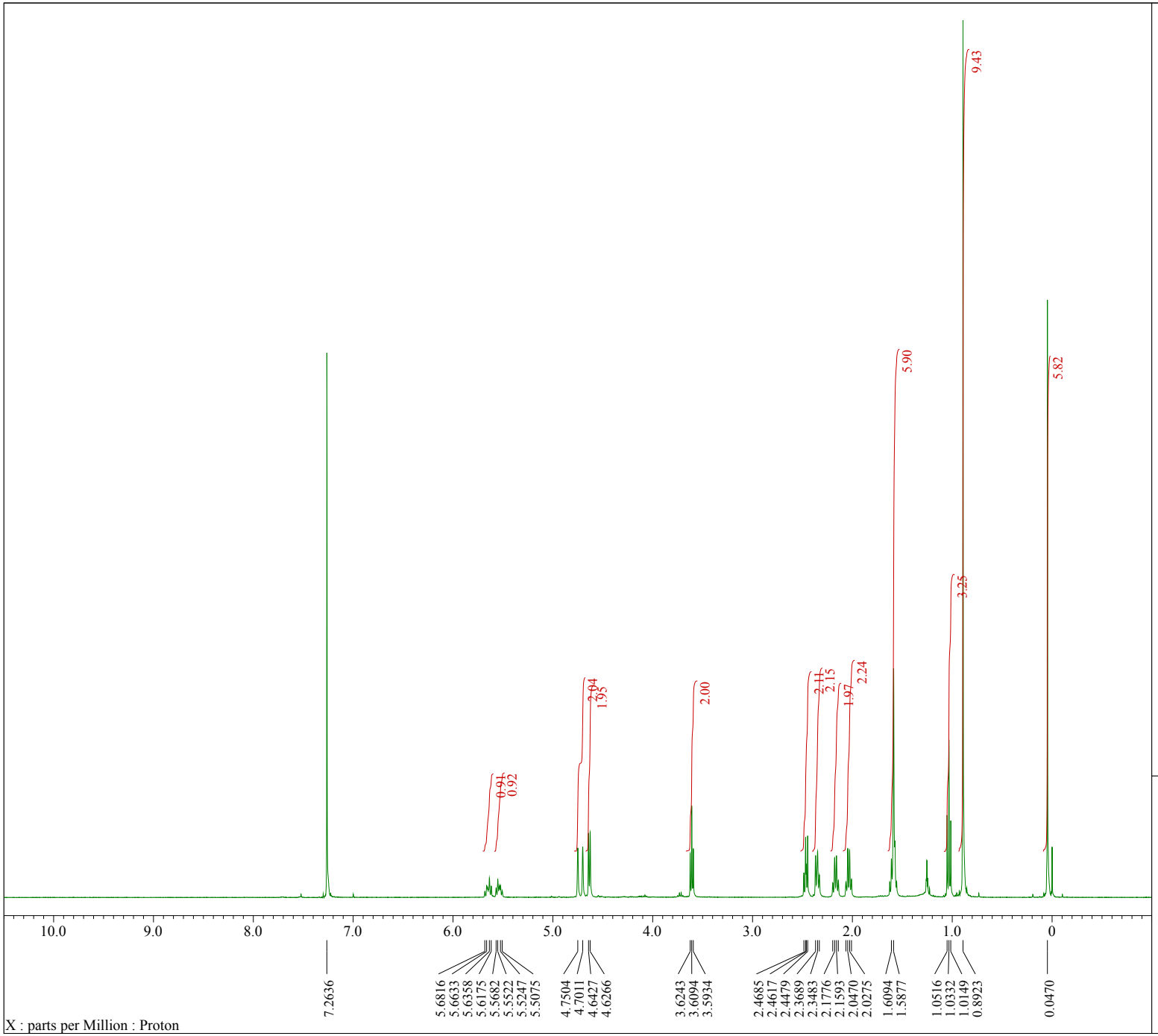
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.6[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



S2
^{13}C-NMR
(100 MHz, $CDCl_3$)

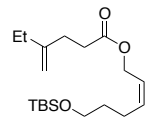



```
Filename          = yk05380data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk05380data
Solvent           = CHLOROFORM-D
Creation_Time     =  5-OCT-2016 22:50:55
Revision_Time     = 20-FEB-2018 19:39:32
Current_Time      = 20-FEB-2018 19:40:04

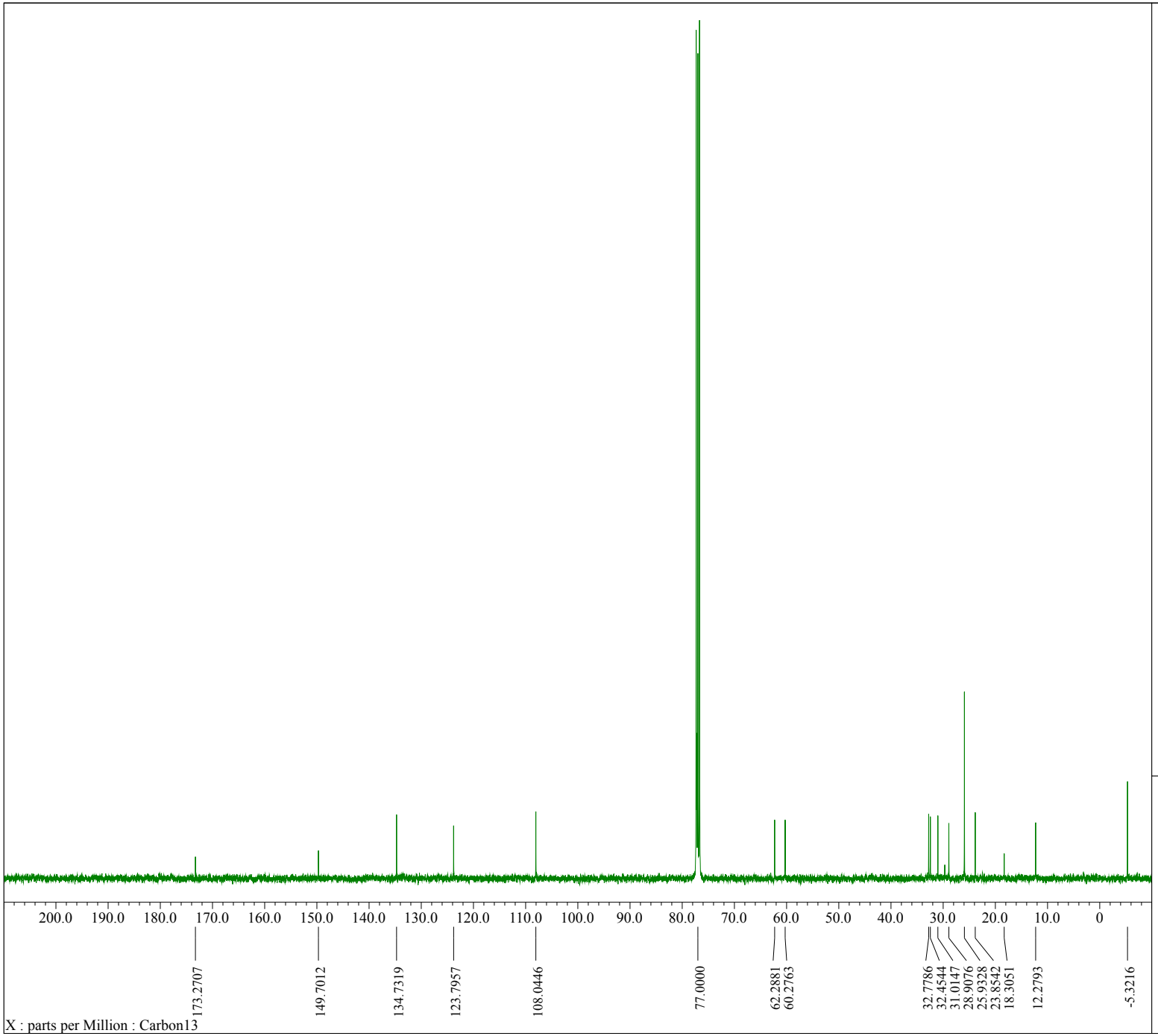
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 23.4[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



19
^{1}H-NMR
(400 MHz, $CDCl_3$)

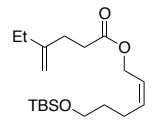



```
Filename          = yk05380data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk05380data
Solvent           = CHLOROFORM-D
Creation_Time     =  6-OCT-2016 01:03:14
Revision_Time     = 20-FEB-2018 16:37:04
Current_Time      = 20-FEB-2018 16:37:40

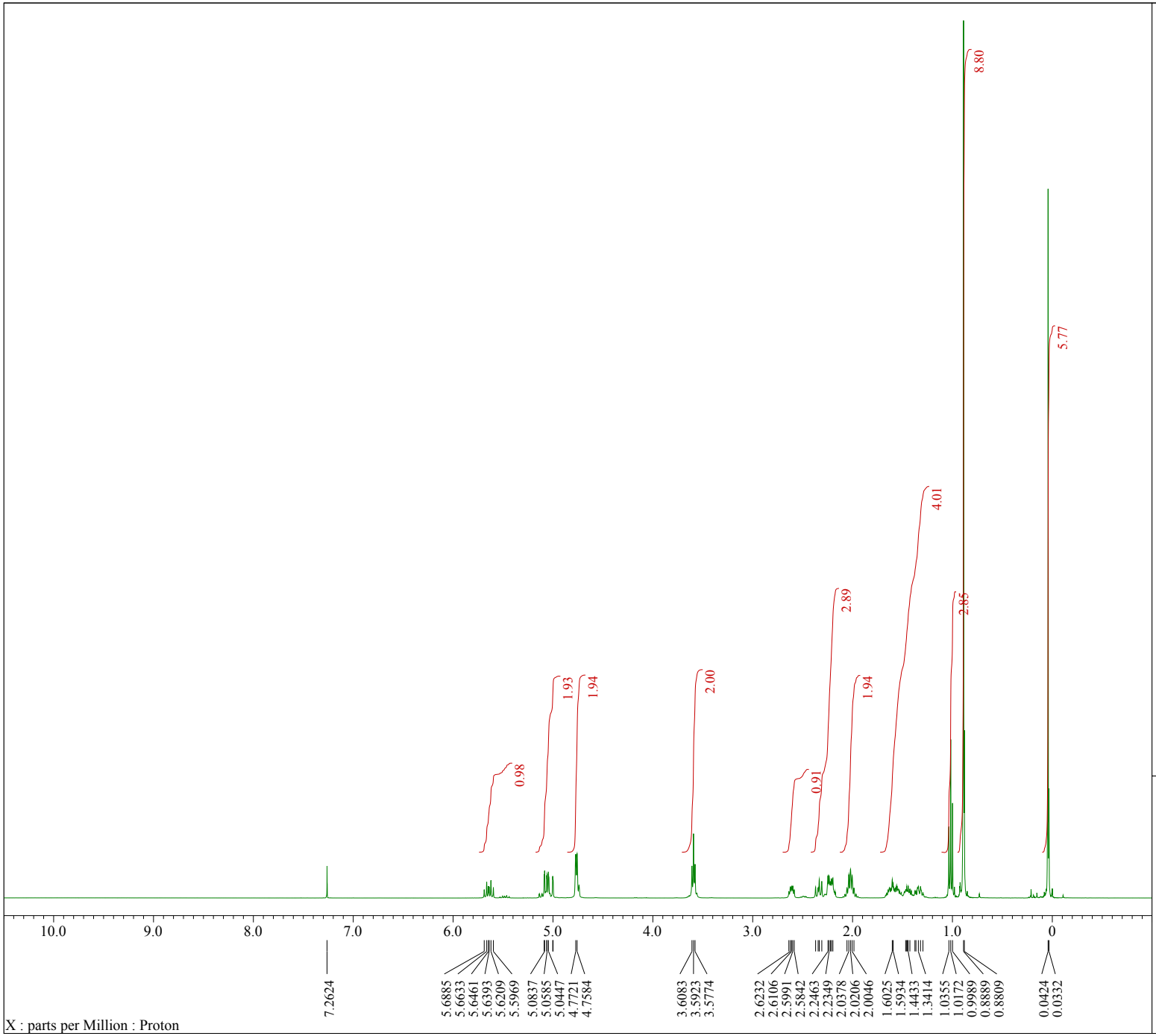
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 2000
Total_Scans       = 2000

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.6[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



19
^{13}C-NMR
(100 MHz, $CDCl_3$)

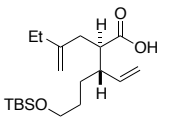



```
Filename          = yk05381data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk05381data
Solvent           = CHLOROFORM-D
Creation_Time     =  8-NOV-2016 09:46:02
Revision_Time     = 20-FEB-2018 19:45:38
Current_Time      = 20-FEB-2018 19:46:29

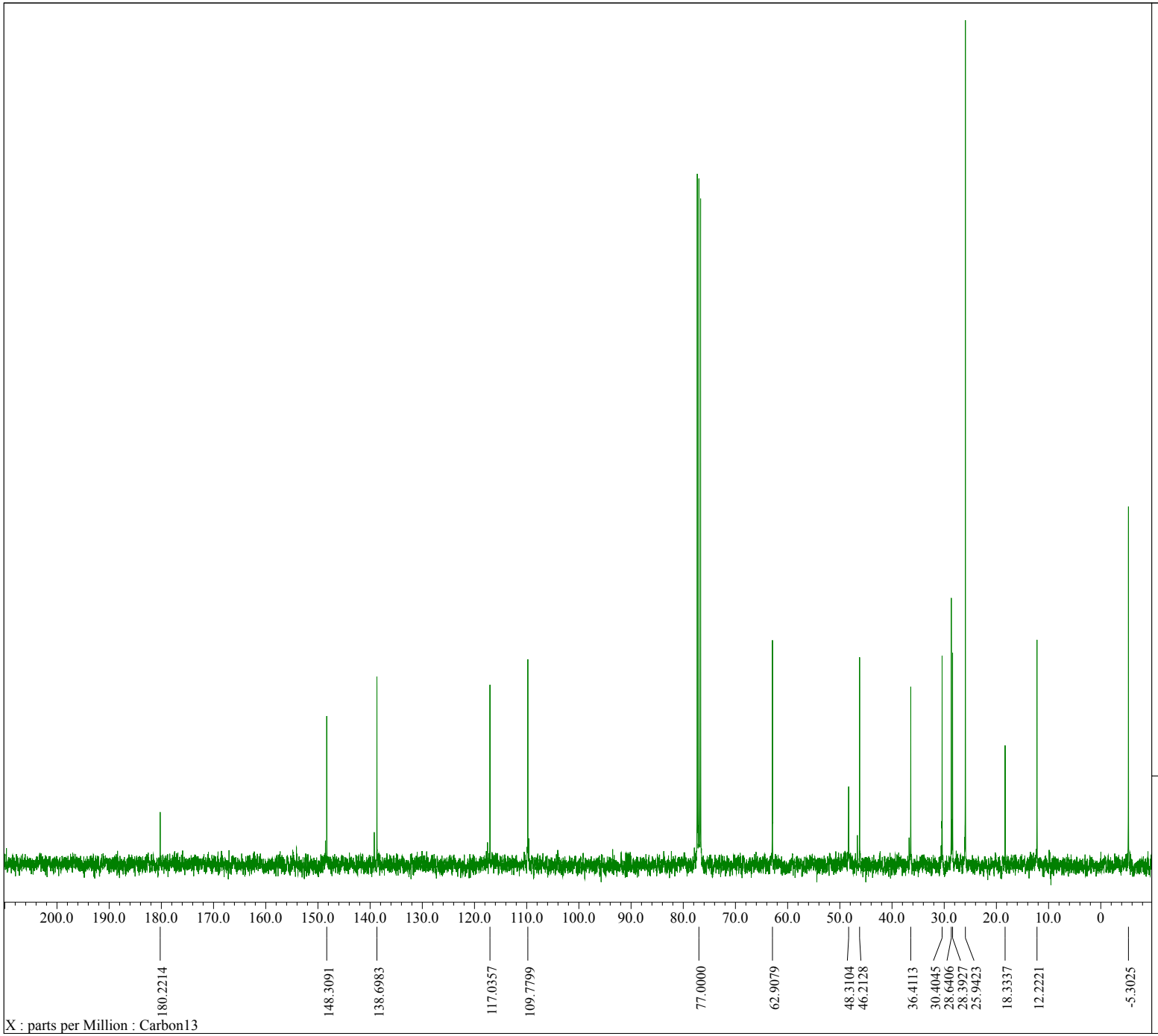
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 23.4[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



20
^{1}H-NMR
(400 MHz, $CDCl_3$)

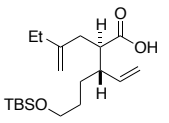



```
Filename          = yk05381data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk05381data
Solvent           = CHLOROFORM-D
Creation_Time     =  8-NOV-2016 09:48:33
Revision_Time     = 20-FEB-2018 16:39:32
Current_Time      = 20-FEB-2018 16:40:03

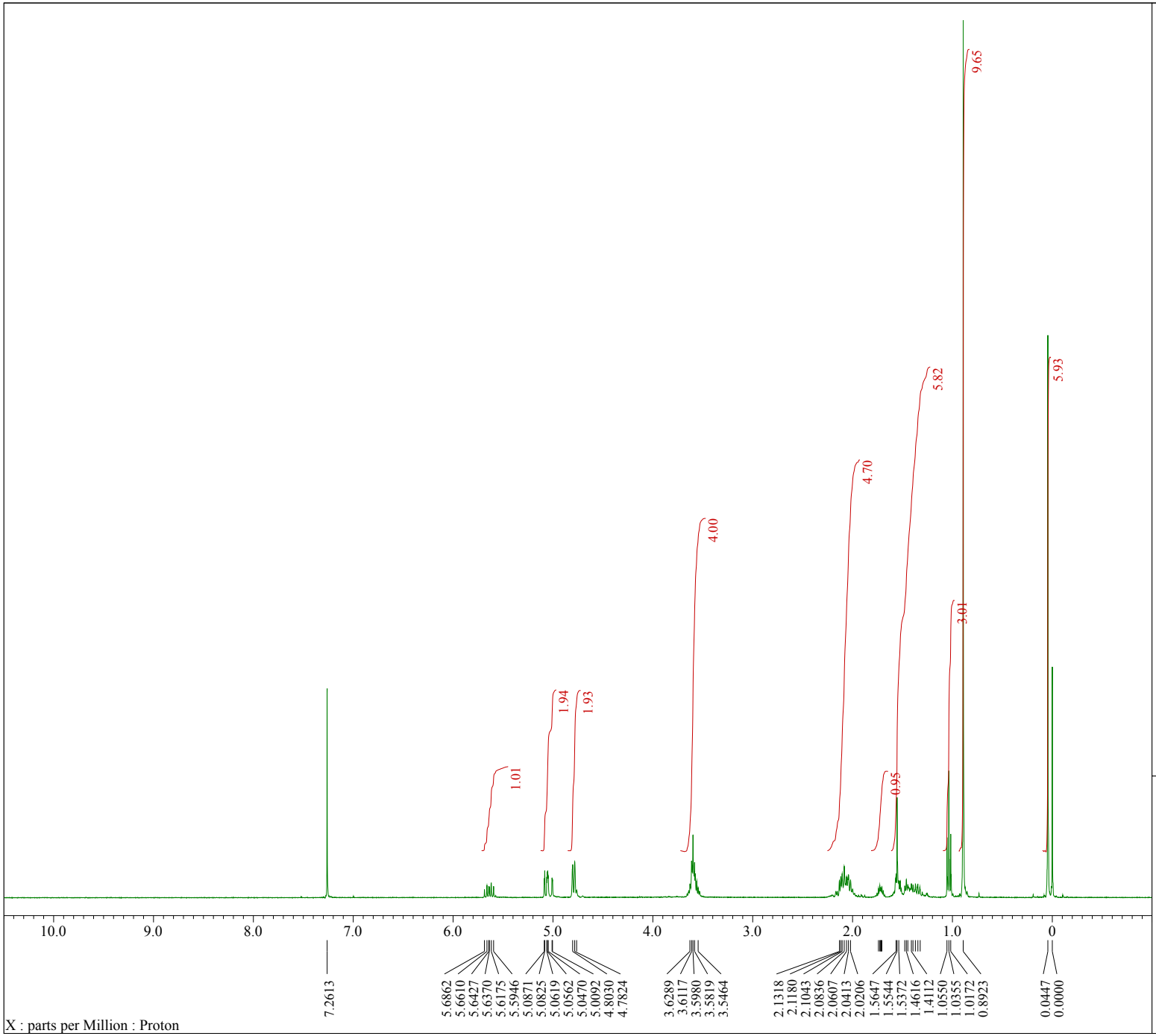
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.6[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



20
^{13}C-NMR
(100 MHz, $CDCl_3$)

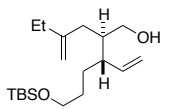



```
Filename          = yk05385data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk05385data
Solvent           = CHLOROFORM-D
Creation_Time     = 11-DEC-2016 16:20:56
Revision_Time     = 20-FEB-2018 19:49:26
Current_Time      = 20-FEB-2018 19:49:58

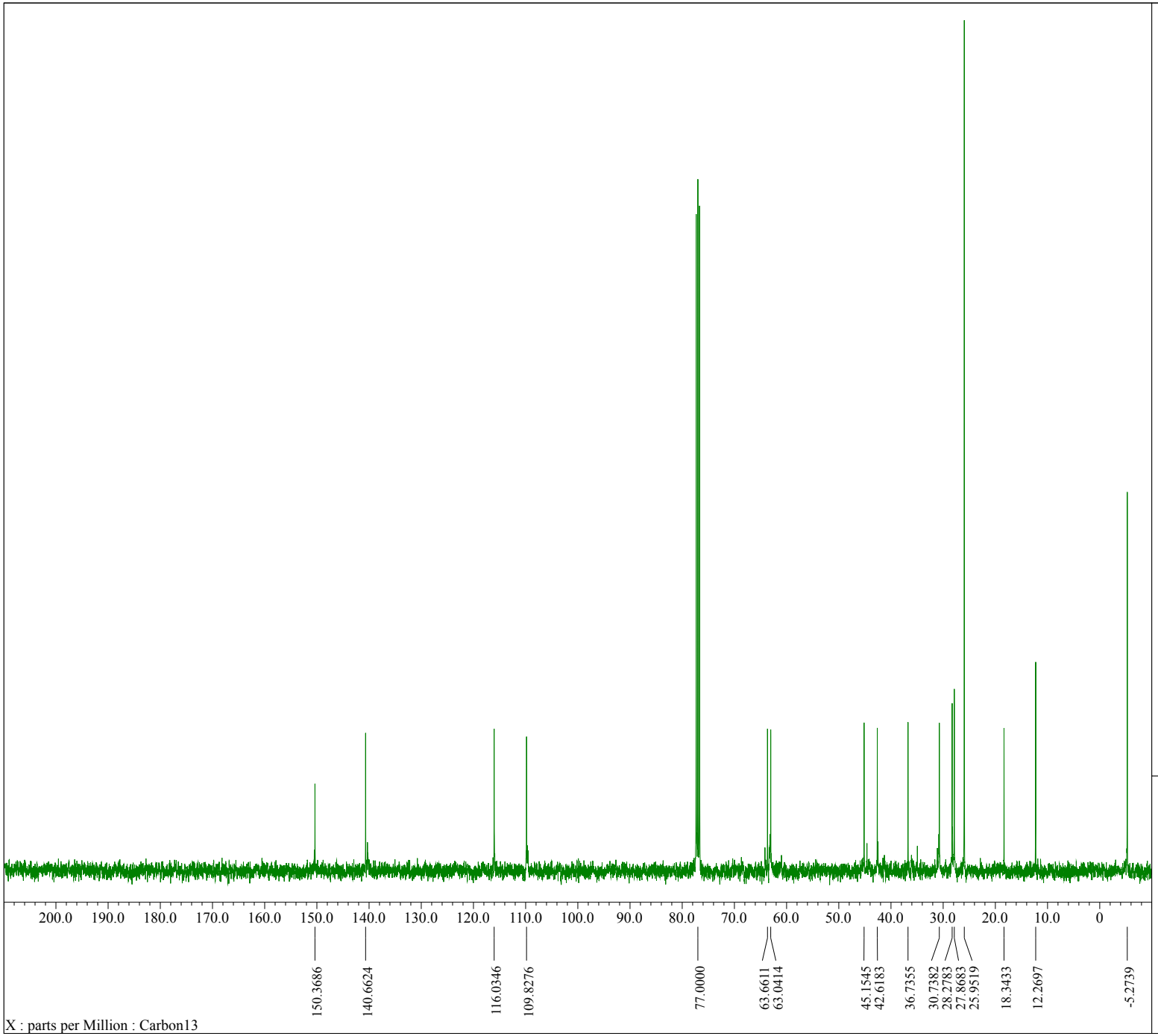
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 22.4[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



S3
^{1}H-NMR
(400 MHz, $CDCl_3$)

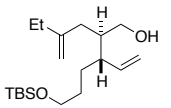



```
Filename          = yk05385data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk05385data
Solvent           = CHLOROFORM-D
Creation_Time     =  8-NOV-2016 13:38:55
Revision_Time     = 20-FEB-2018 16:41:48
Current_Time      = 20-FEB-2018 16:42:23

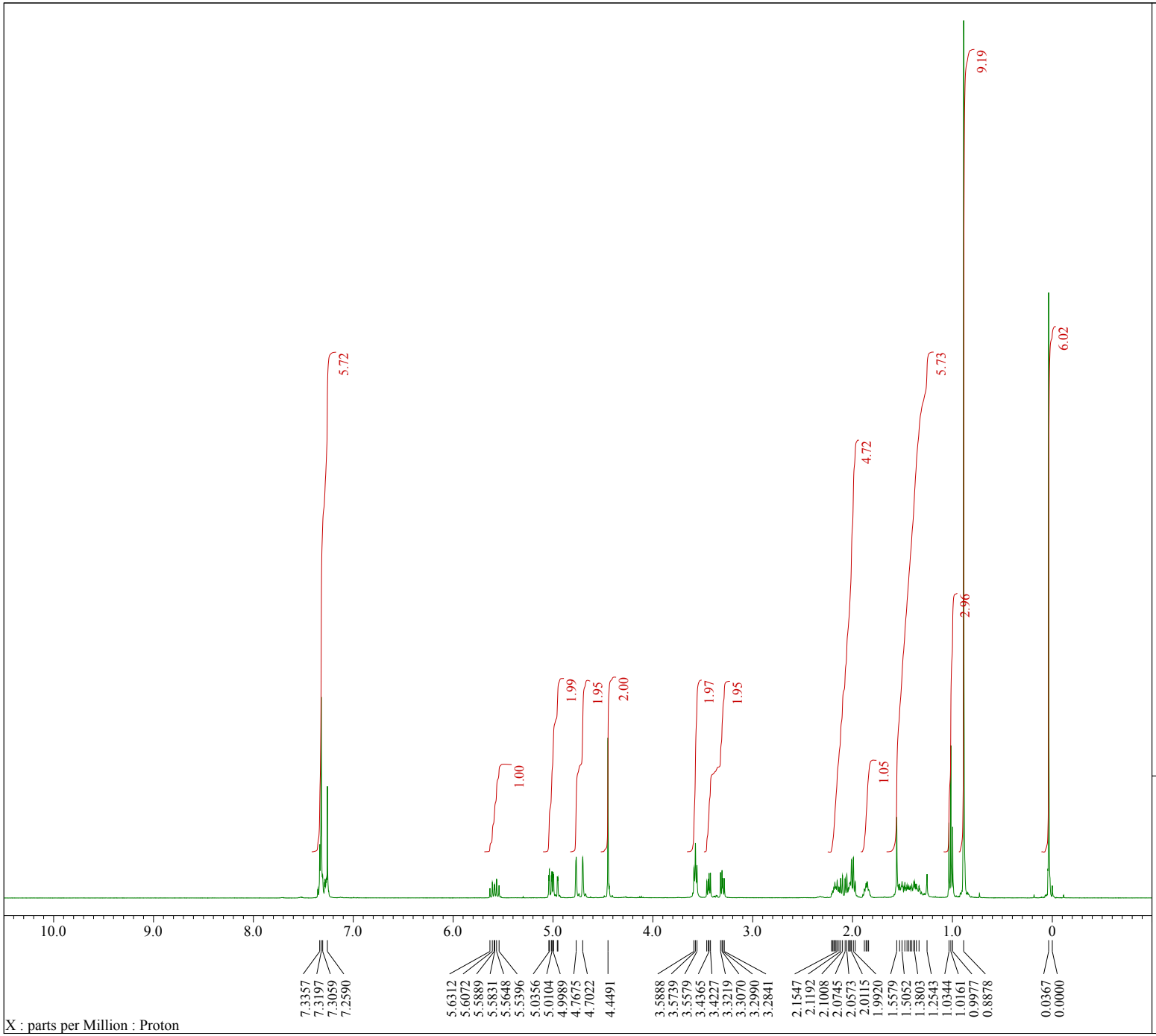
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.3[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



S3
^{13}C-NMR
(100 MHz, $CDCl_3$)

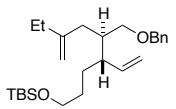



```
Filename          = yk06042_non-data-1-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06042
Solvent           = CHLOROFORM-D
Creation_Time     = 11-JAN-2017 21:18:09
Revision_Time     = 20-FEB-2018 19:52:52
Current_Time      = 20-FEB-2018 19:53:21

Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 16
Total_Scans       = 16

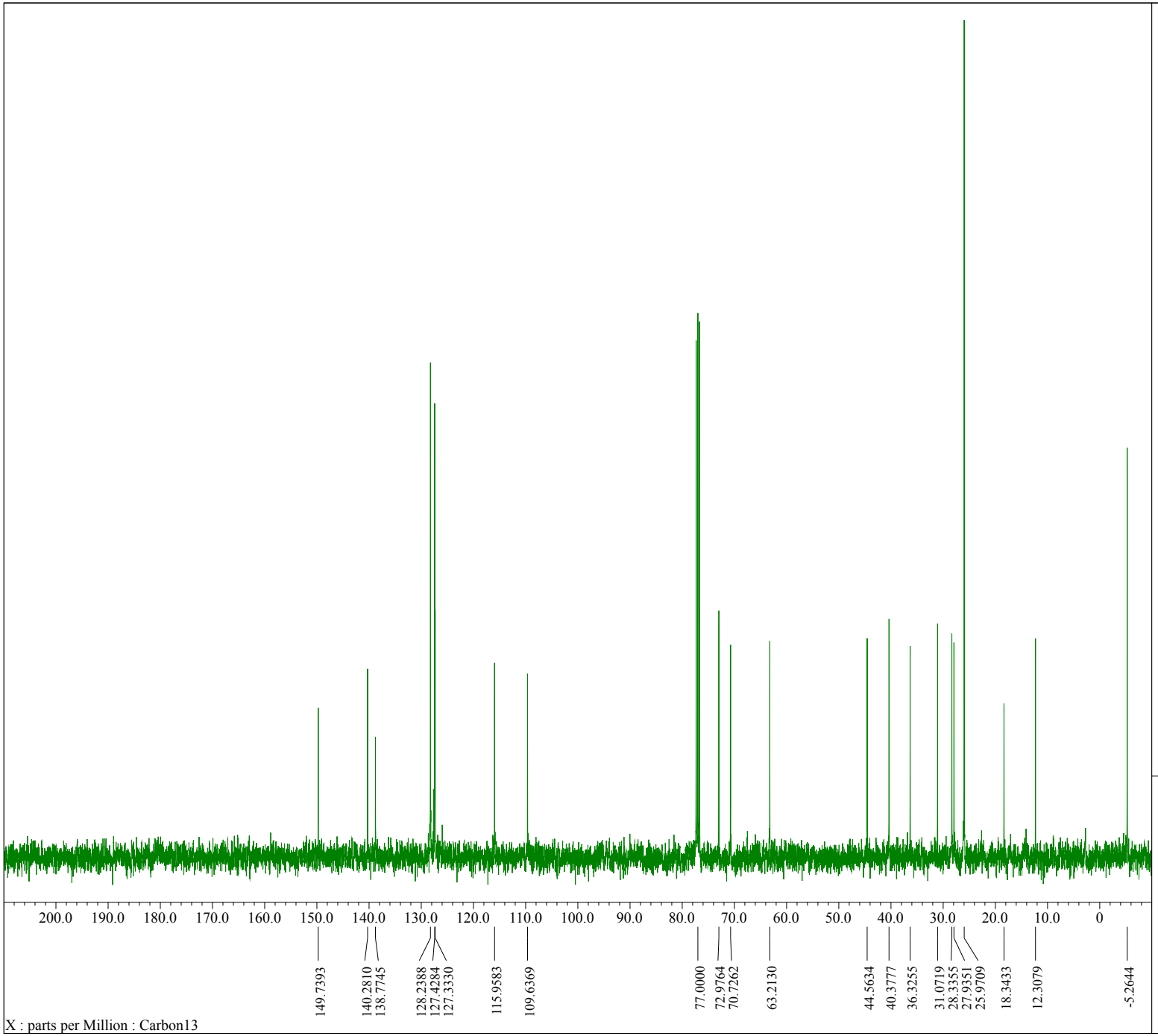
Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 22.1[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



21

^{1}H-NMR

(400 MHz, $CDCl_3$)

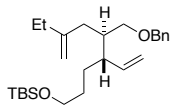



```
Filename          = yk06042data_bcm1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = 7
Solvent           = CHLOROFORM-D
Creation_Time     = 15-JAN-2017 01:04:06
Revision_Time     = 20-FEB-2018 16:44:27
Current_Time      = 20-FEB-2018 16:44:57

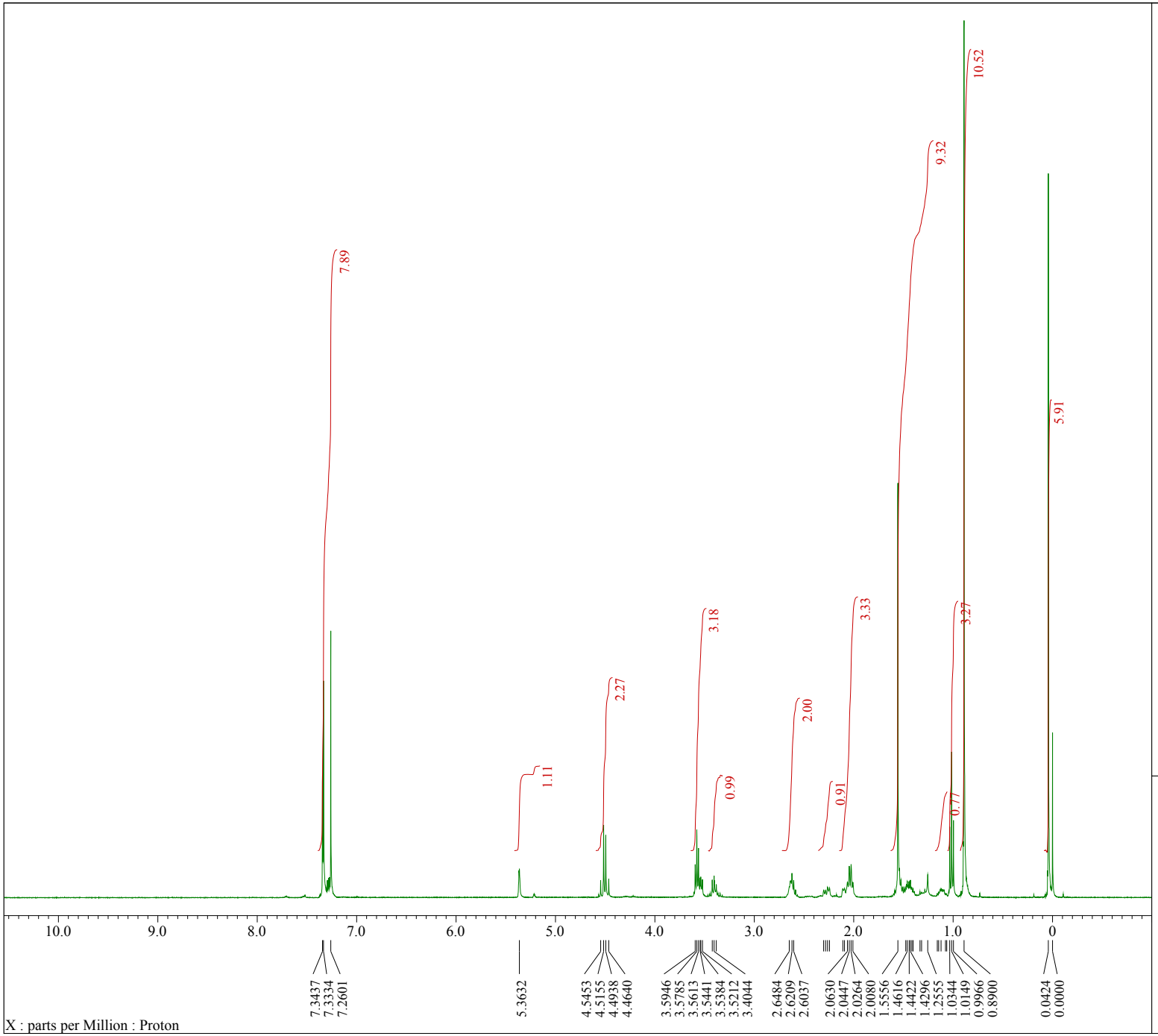
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 50
Total_Scans       = 50

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 21.4[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



21
^{13}C-NMR
(100 MHz, $CDCl_3$)

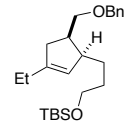



```
Filename          = yk06047data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk05400data
Solvent           = CHLOROFORM-D
Creation_Time     = 31-OCT-2016 19:24:34
Revision_Time     = 20-FEB-2018 19:56:44
Current_Time      = 20-FEB-2018 19:57:51

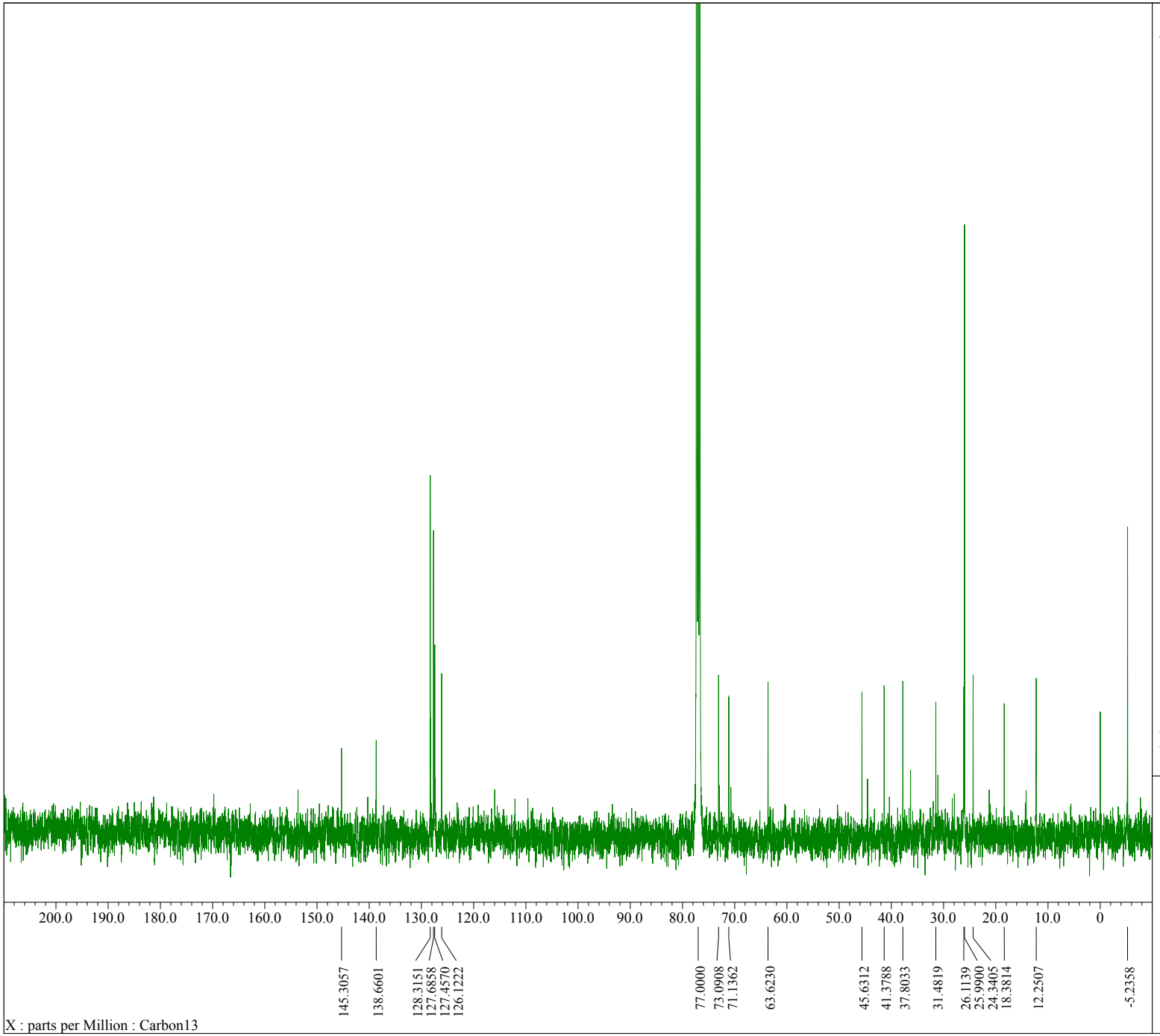
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 23.5[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



22
^{1}H-NMR
(400 MHz, $CDCl_3$)

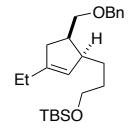



```
Filename          = YK06047data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = 8
Solvent           = CHLOROFORM-D
Creation_Time     = 15-JAN-2017 04:37:02
Revision_Time     = 20-FEB-2018 16:47:34
Current_Time      = 20-FEB-2018 16:48:23

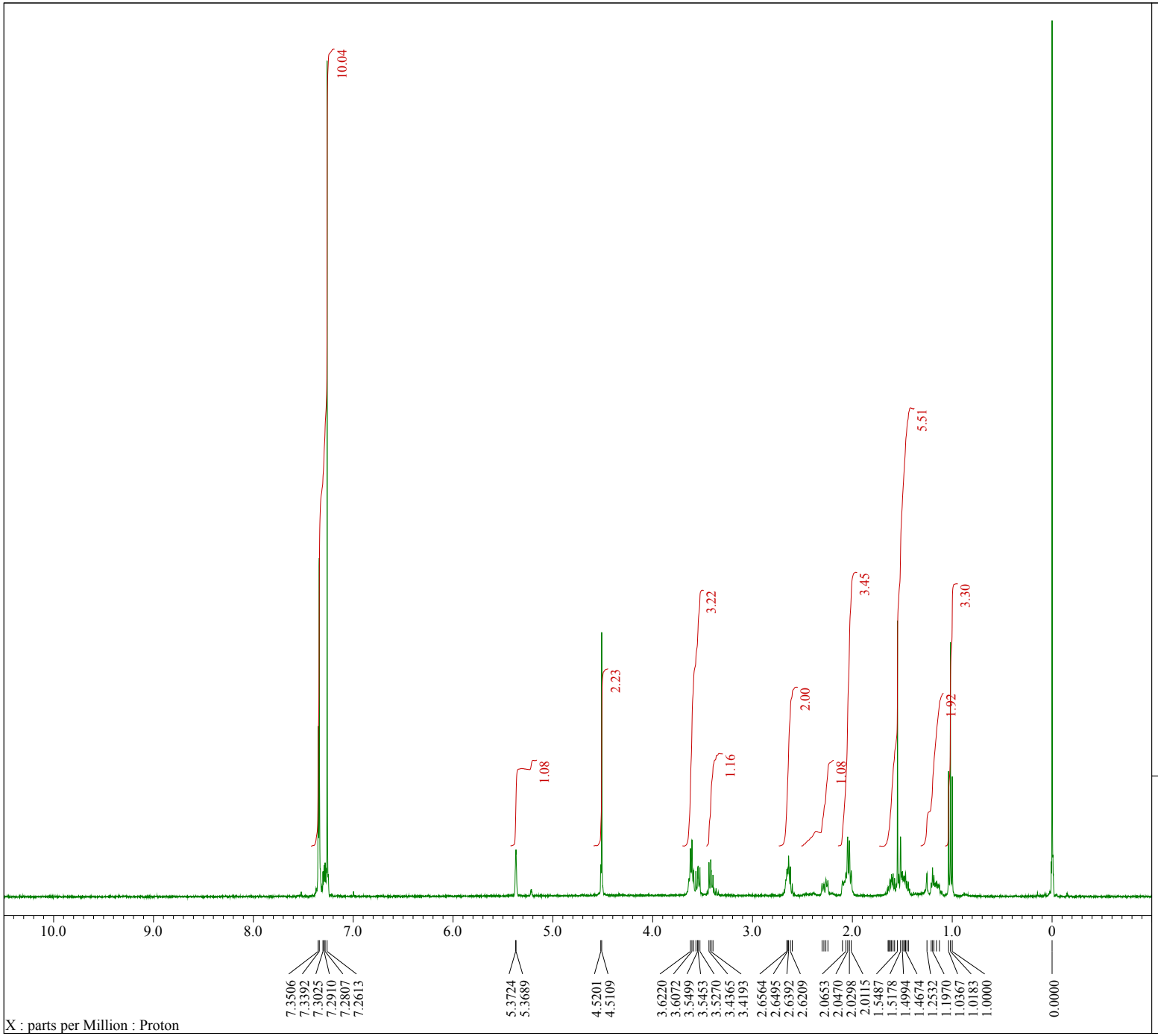
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 5321
Total_Scans       = 5321

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 21.1[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



22
^{13}C-NMR
(100 MHz, $CDCl_3$)

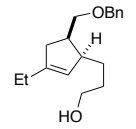



```
Filename          = yk06052data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06052data
Solvent           = CHLOROFORM-D
Creation_Time     = 11-DEC-2016 16:14:22
Revision_Time     = 20-FEB-2018 20:01:24
Current_Time      = 20-FEB-2018 20:01:50

Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

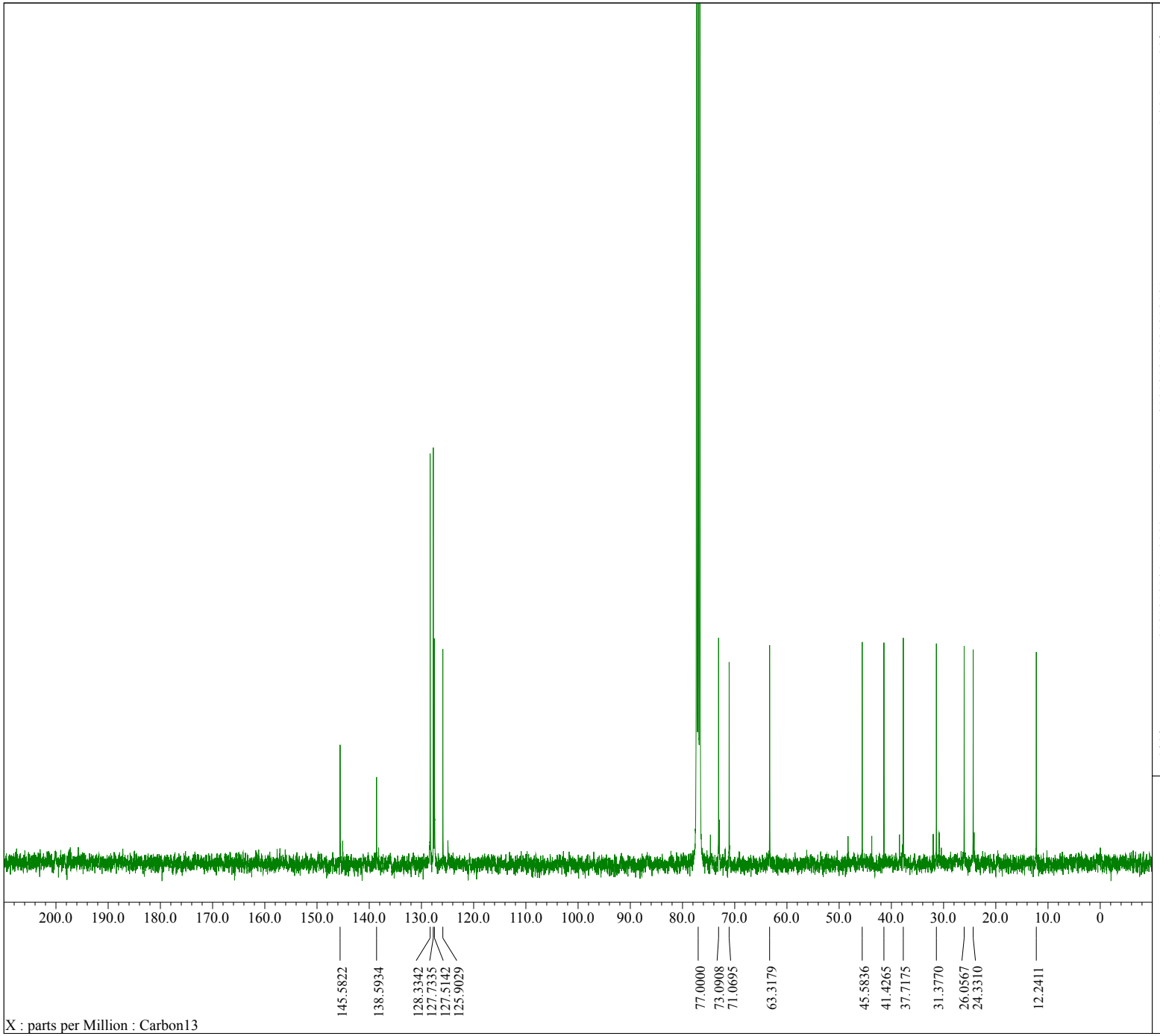
Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 22.4[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



S4

^{1}H-NMR

(400 MHz, $CDCl_3$)

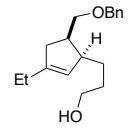



```
Filename          = yk06052data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06052data
Solvent           = CHLOROFORM-D
Creation_Time     =  9-NOV-2016 01:03:53
Revision_Time     = 20-FEB-2018 16:50:20
Current_Time      = 20-FEB-2018 16:51:21

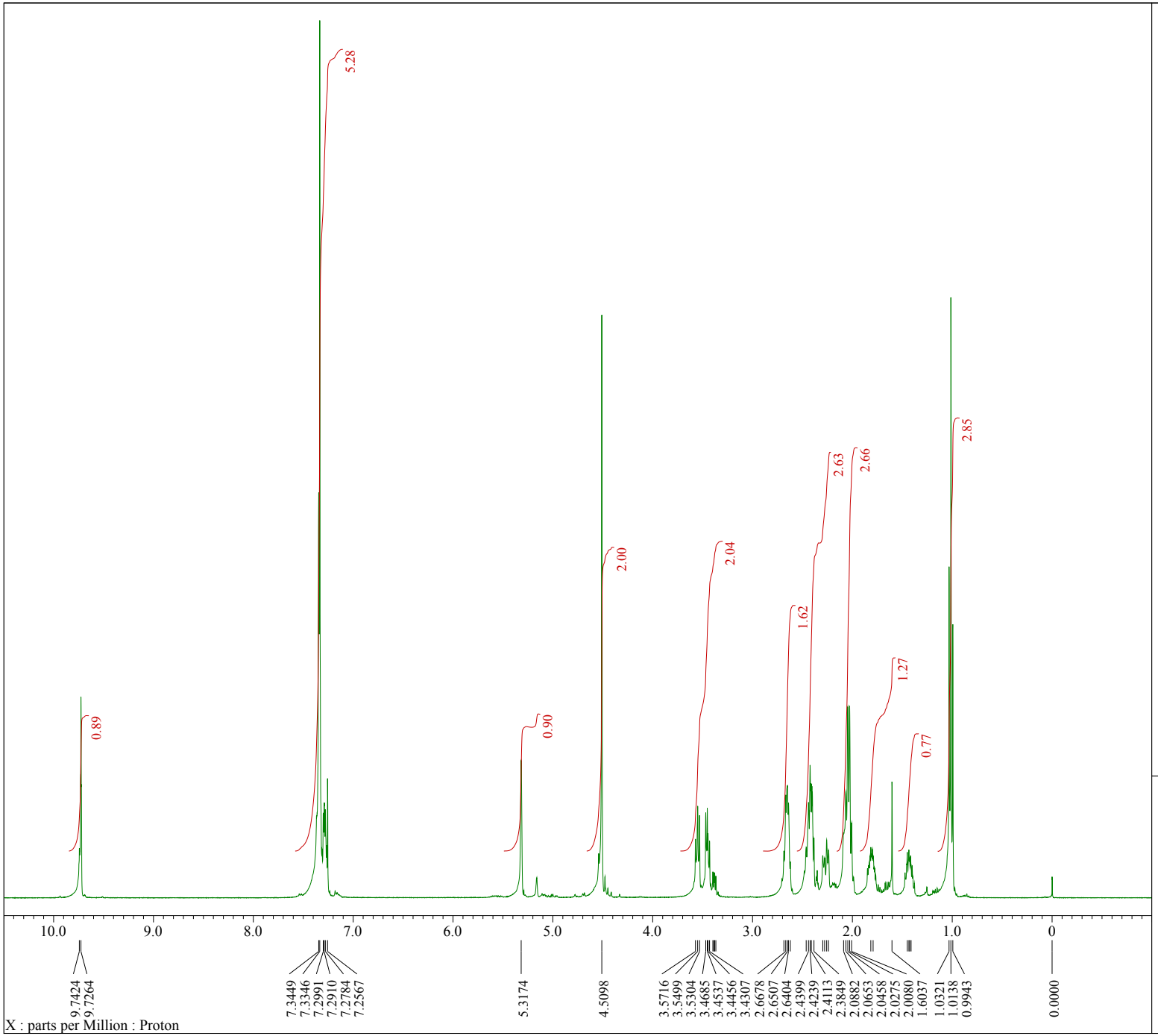
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 5000
Total_Scans       = 5000

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.3[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



S4
^{13}C-NMR
(100 MHz, $CDCl_3$)

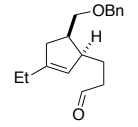



```
Filename          = yk06058data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06058data
Solvent           = CHLOROFORM-D
Creation_Time     =  9-NOV-2016 15:27:50
Revision_Time     = 20-FEB-2018 20:09:39
Current_Time      = 20-FEB-2018 20:10:14

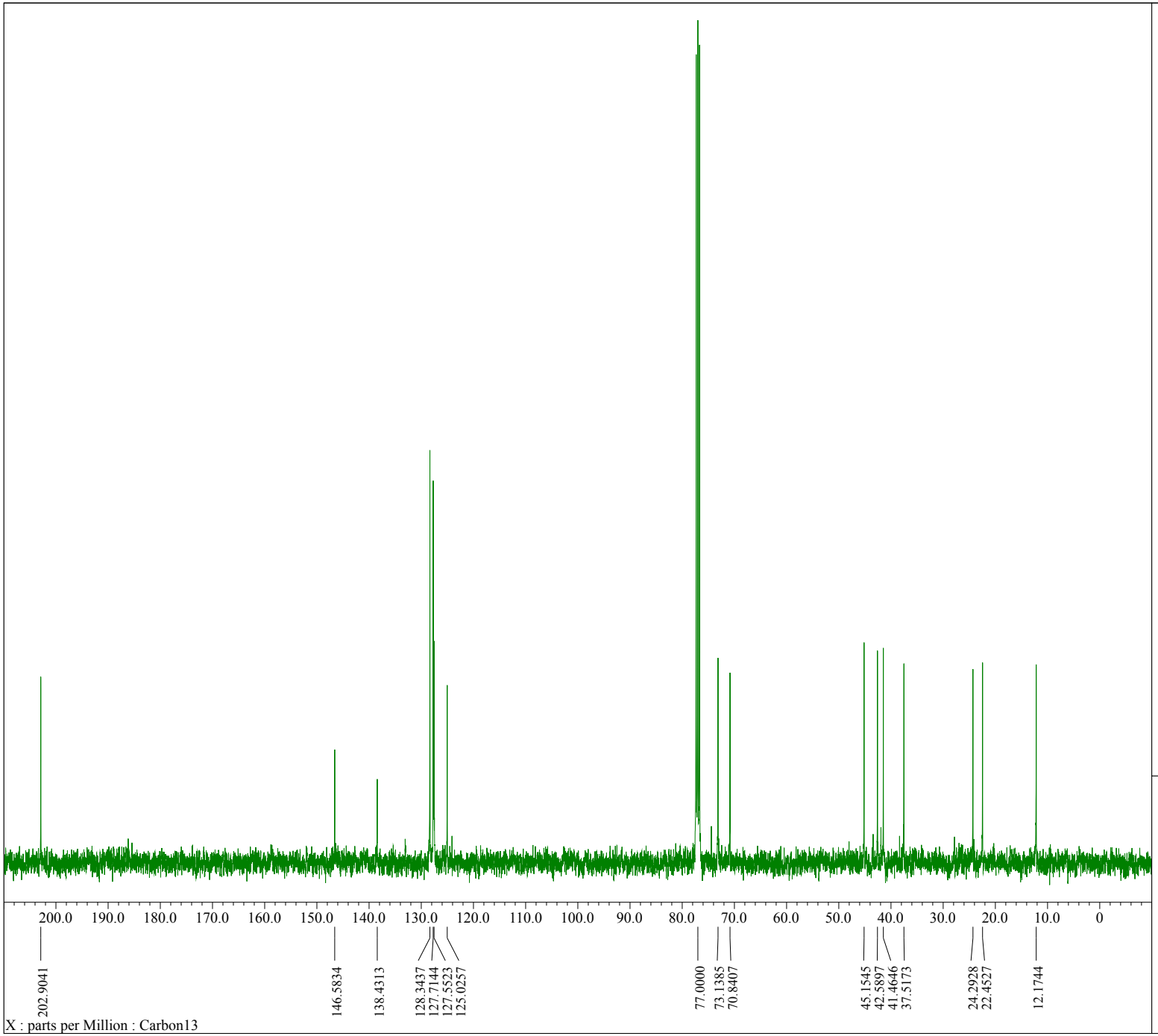
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 23.3[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



23
^{1}H-NMR
(400 MHz, $CDCl_3$)

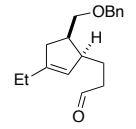



```
Filename          = yk06058data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06058data
Solvent           = CHLOROFORM-D
Creation_Time     =  9-NOV-2016 15:31:19
Revision_Time     = 20-FEB-2018 16:52:45
Current_Time      = 20-FEB-2018 16:53:14

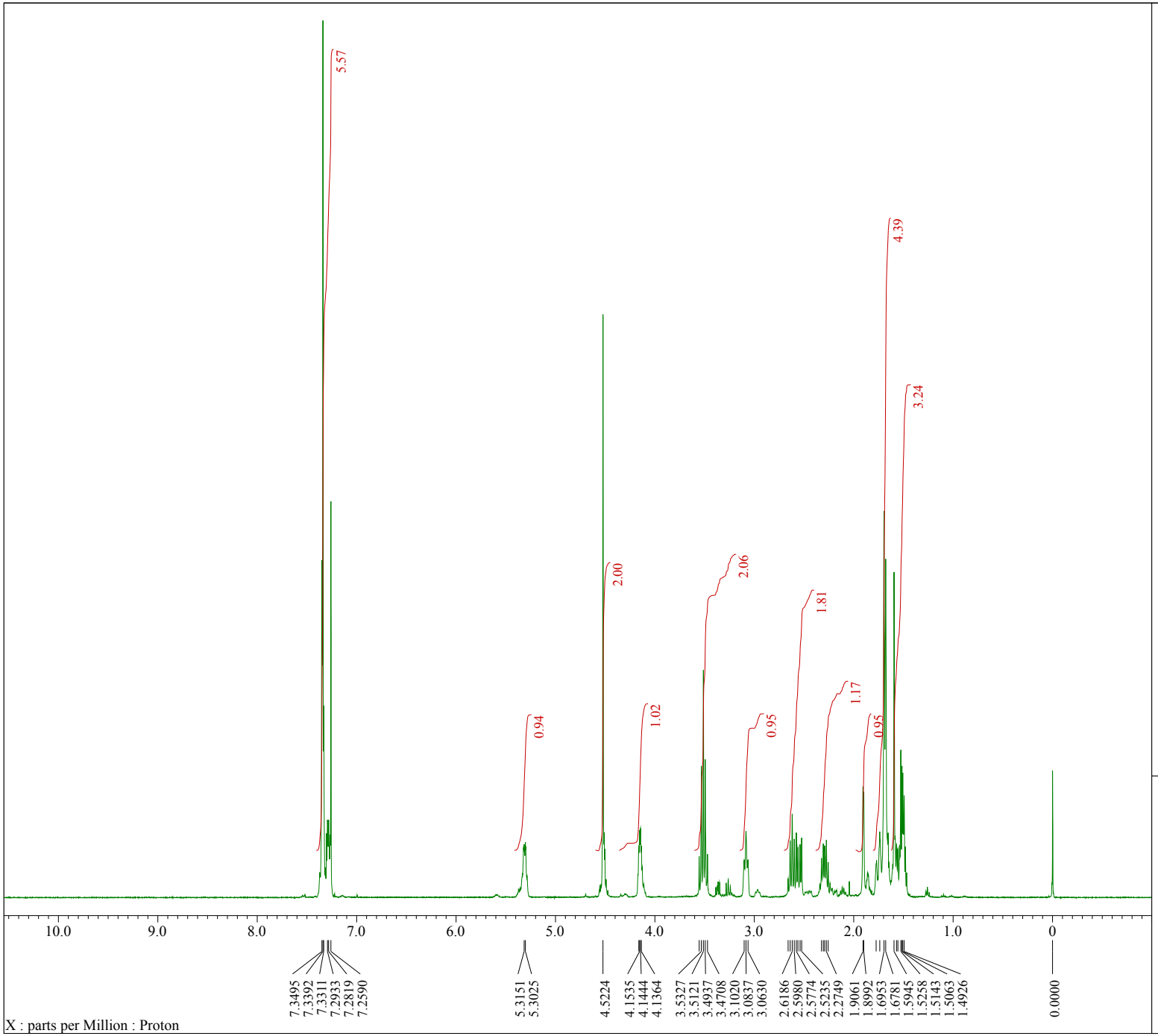
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.4[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



23
^{13}C-NMR
(100 MHz, $CDCl_3$)

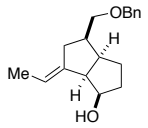



```
Filename          = yk06065_non-data-1-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06065
Solvent           = CHLOROFORM-D
Creation_Time     =  2-NOV-2016 23:15:55
Revision_Time     = 20-FEB-2018 20:13:18
Current_Time      = 20-FEB-2018 20:13:59

Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

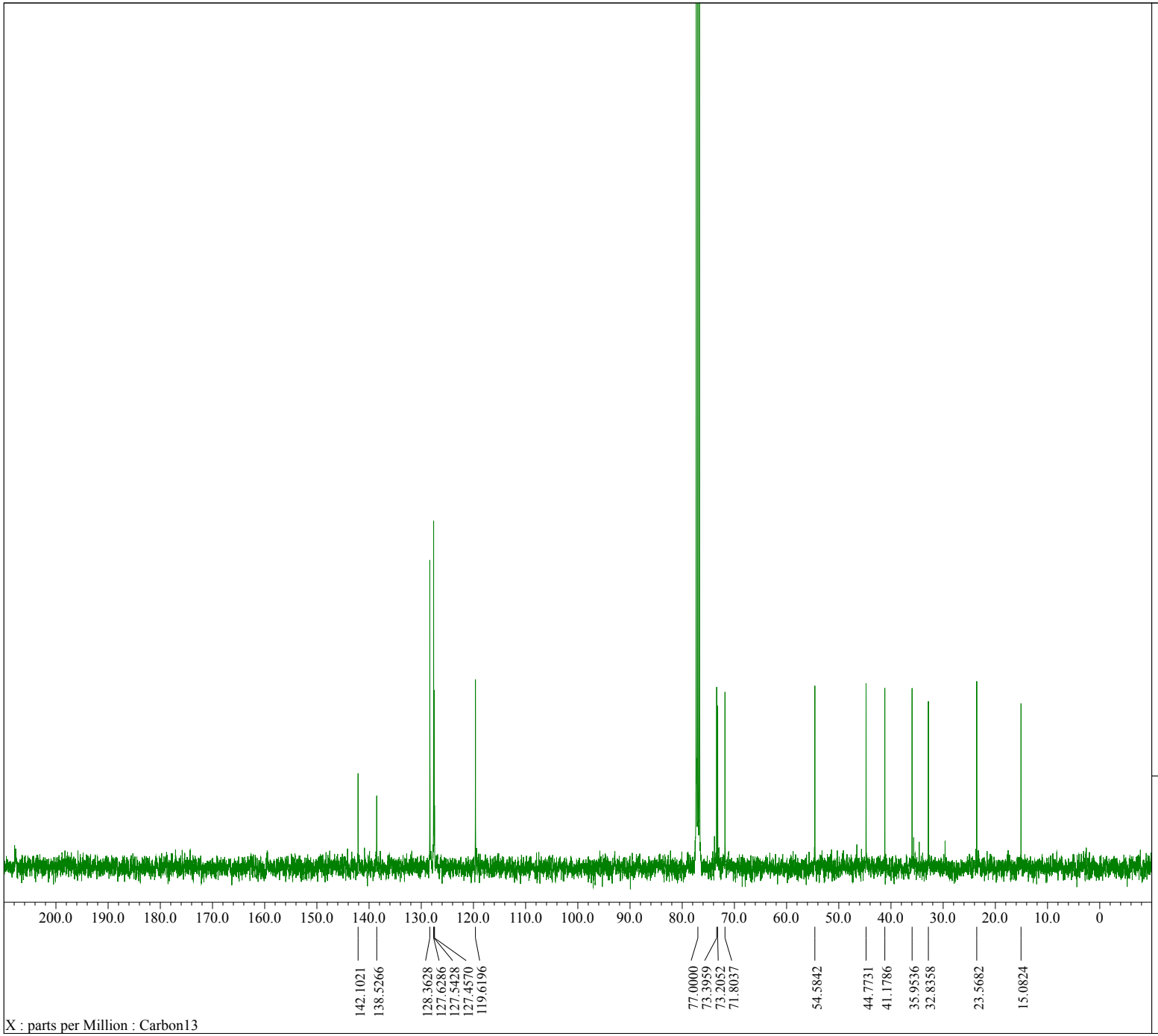
Relaxation_Delay = 5[s]
Recvr_Gain        = 40
Temp_Get          = 23[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



24

^{1}H-NMR

(400 MHz, $CDCl_3$)

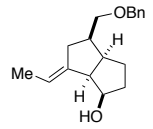



```
Filename          = yk06065_bcm-2-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06065
Solvent           = CHLOROFORM-D
Creation_Time     =  2-NOV-2016 23:35:43
Revision_Time     = 20-FEB-2018 16:54:43
Current_Time      = 20-FEB-2018 16:55:18

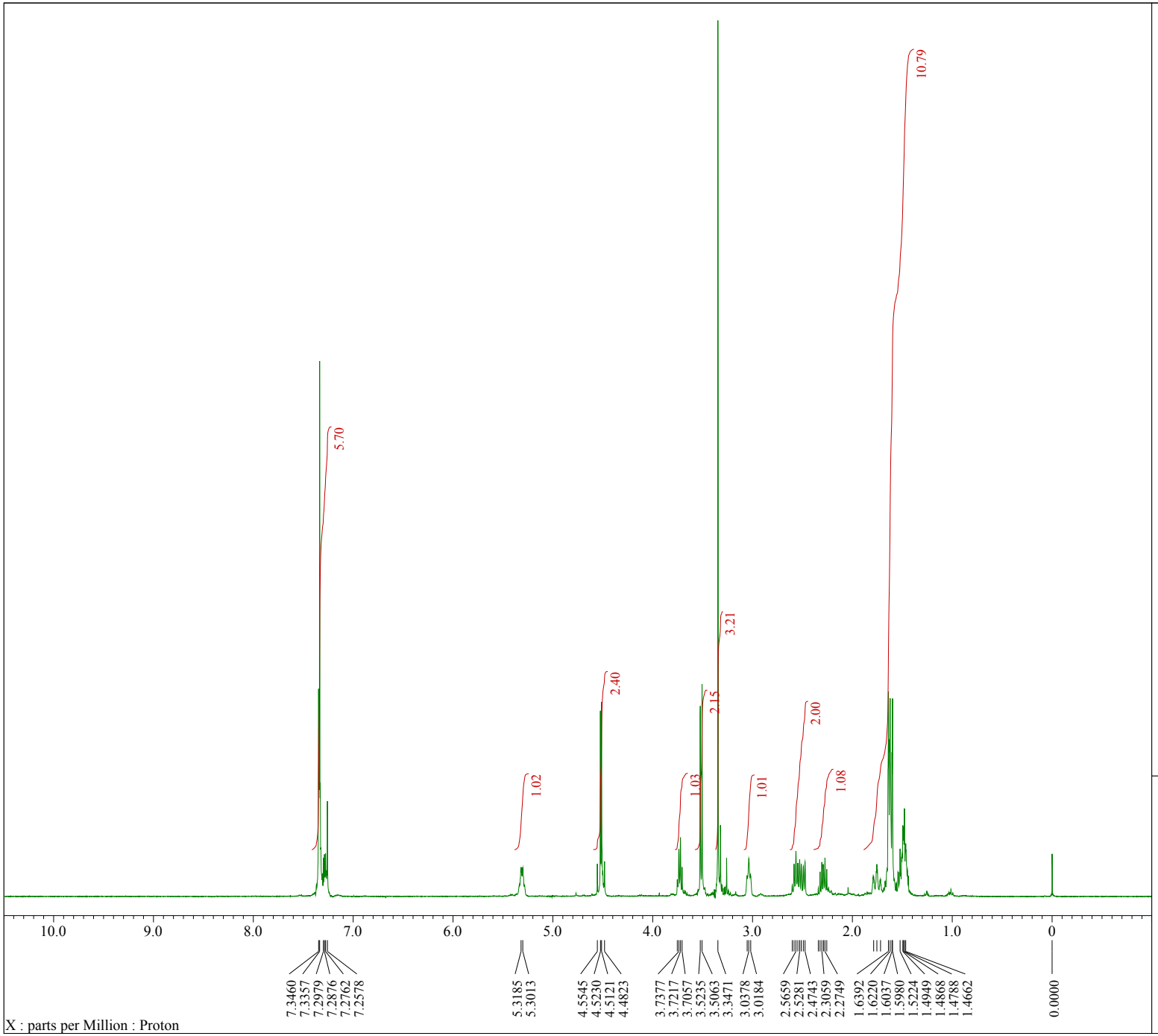
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.4[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



24
^{13}C-NMR
(100 MHz, $CDCl_3$)

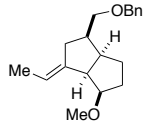



```
Filename          = yk05351_non-data-1-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk05351
Solvent           = CHLOROFORM-D
Creation_Time     =  5-AUG-2016 20:03:37
Revision_Time     = 20-FEB-2018 20:17:02
Current_Time      = 20-FEB-2018 20:17:35

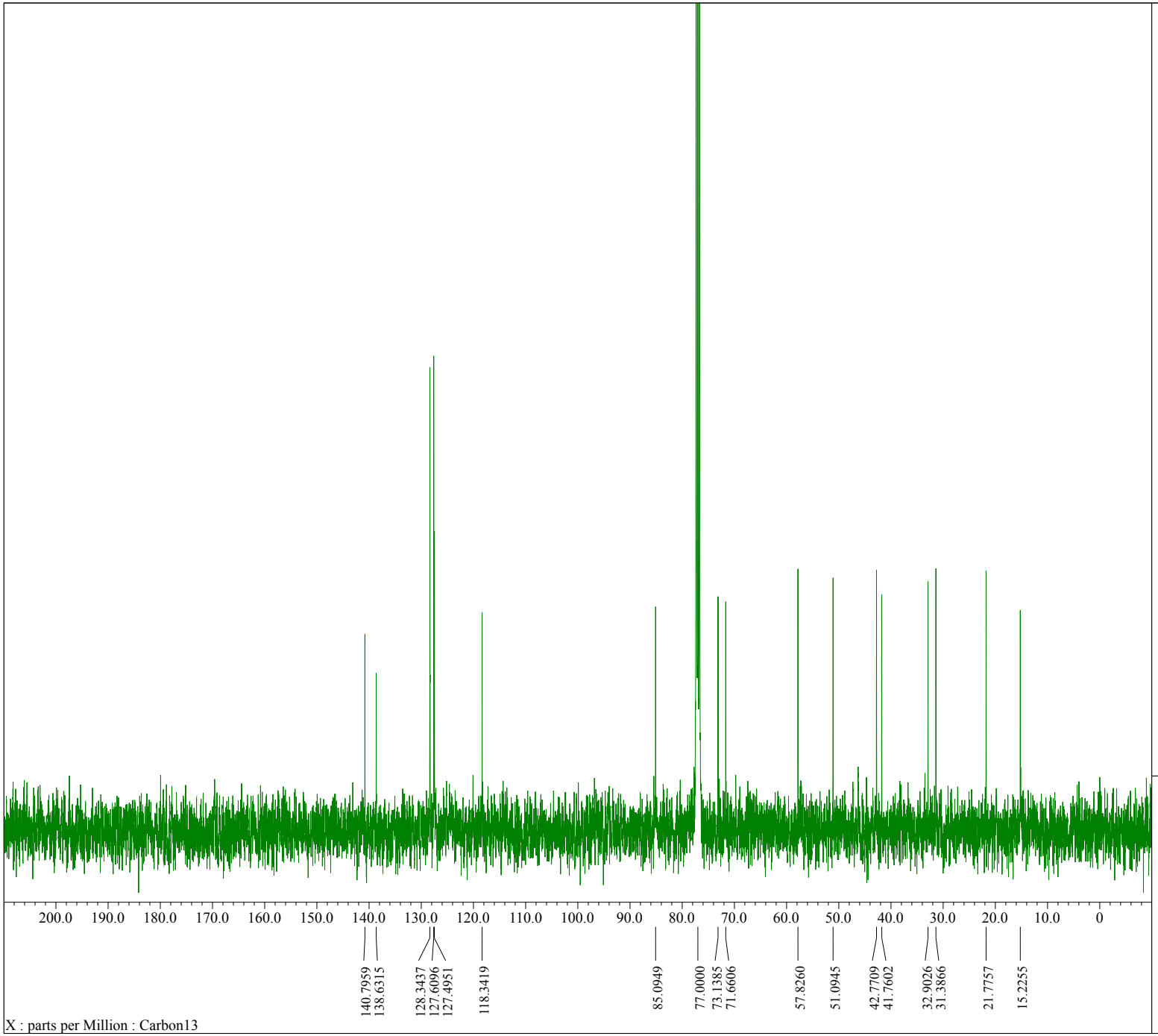
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1
Total_Scans       = 1

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 23.8[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 90[deg]
X_Atn             = 1[dB]
X_Pulse           = 10.025[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



S5
^{1}H-NMR
(400 MHz, $CDCl_3$)

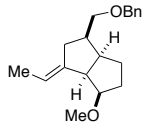



```
Filename          = yk05351_bcm-2-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06076
Solvent           = CHLOROFORM-D
Creation_Time     = 20-JAN-2017 22:41:10
Revision_Time     = 20-FEB-2018 16:57:43
Current_Time      = 20-FEB-2018 16:58:55

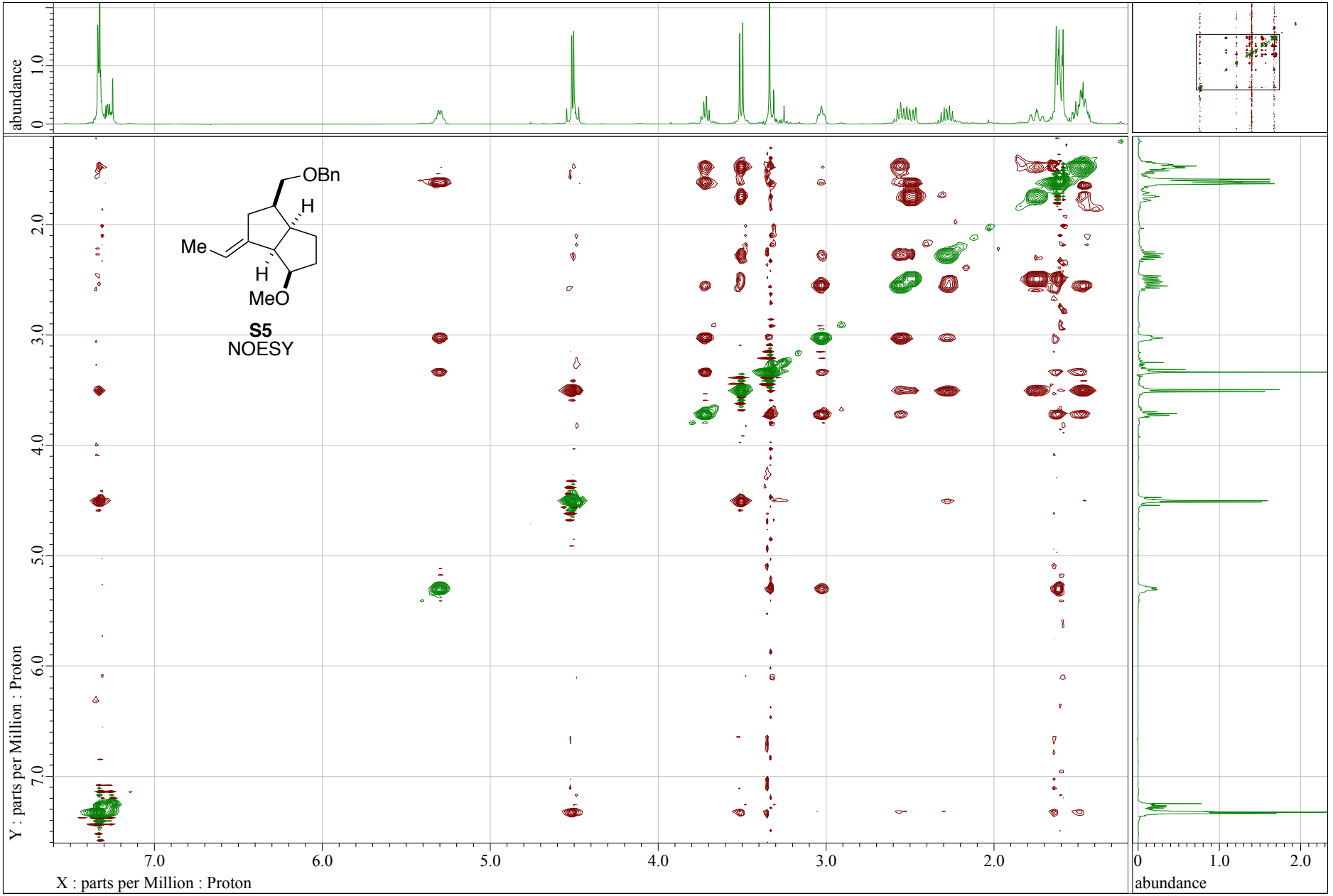
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

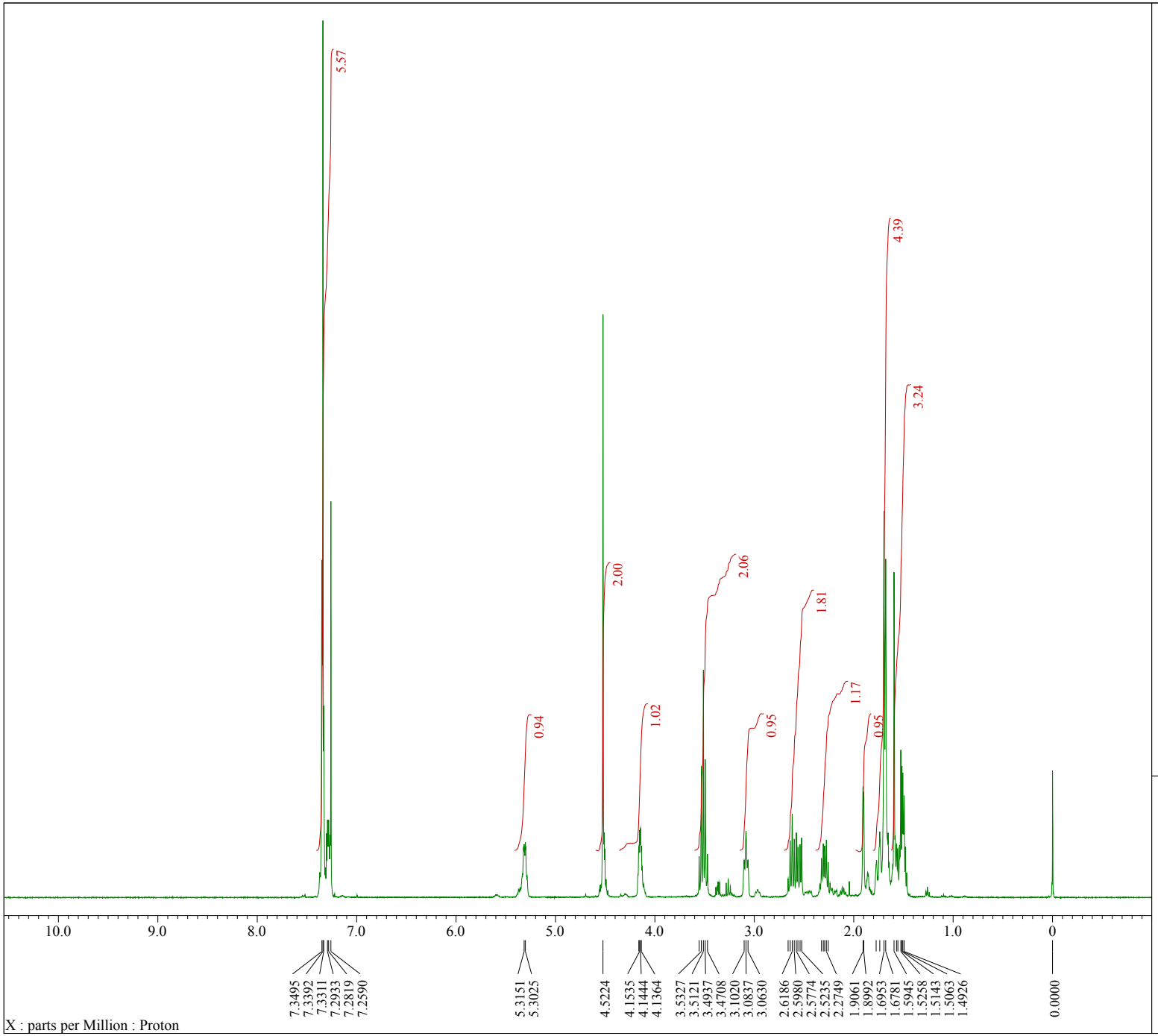
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 2000
Total_Scans       = 2000

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 22.1[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



S5
^{13}C-NMR
(100 MHz, $CDCl_3$)


abundance
1.0
0
OBn
H
Me
H
MeO
S5
NOESY
2.0
3.0
4.0
5.0
6.0
7.0
Y : parts per Million : Proton
7.0
6.0
5.0
4.0
3.0
2.0
X : parts per Million : Proton
0
1.0
2.0
abundance

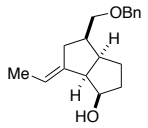



```
Filename          = yk06065_non-data-1-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06065
Solvent           = CHLOROFORM-D
Creation_Time     =  2-NOV-2016 23:15:55
Revision_Time     = 20-FEB-2018 20:13:18
Current_Time      = 20-FEB-2018 20:13:59

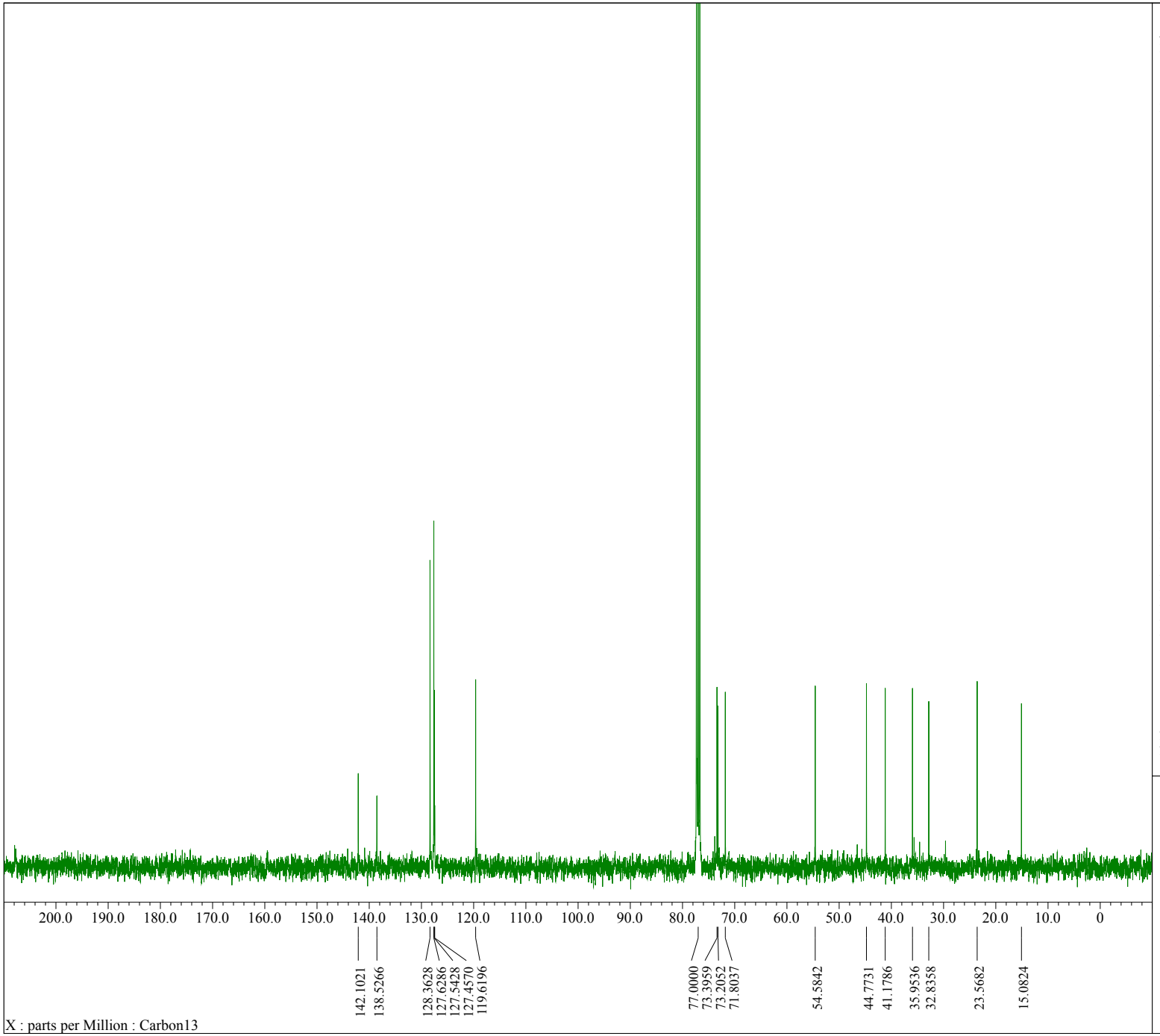
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 40
Temp_Get          = 23[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



24
^{1}H-NMR
(400 MHz, $CDCl_3$)

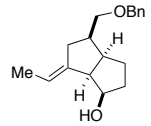



```
Filename          = yk06065_bcm-2-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06065
Solvent           = CHLOROFORM-D
Creation_Time     =  2-NOV-2016 23:35:43
Revision_Time     = 20-FEB-2018 16:54:43
Current_Time      = 20-FEB-2018 16:55:18

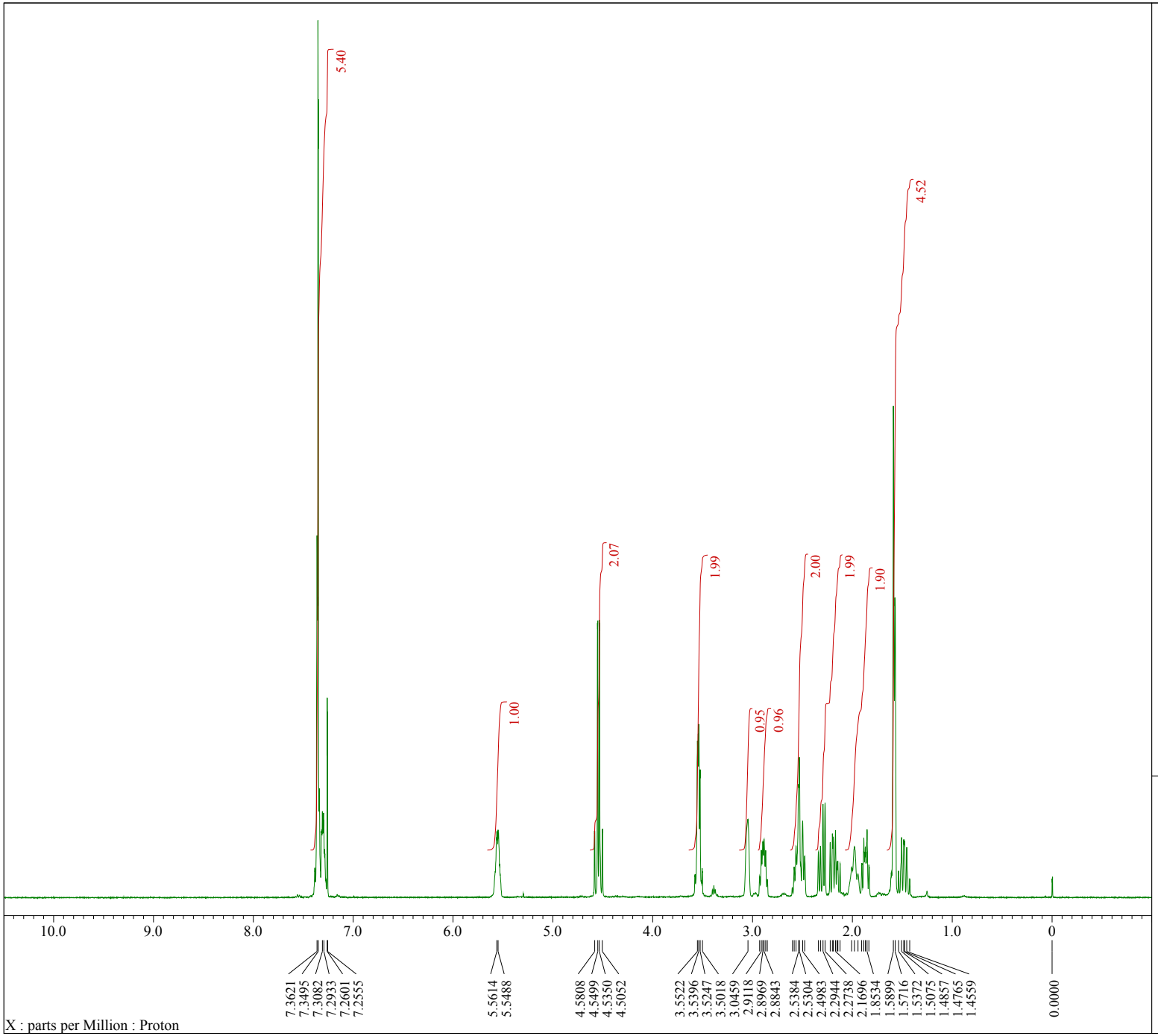
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.4[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



24
^{13}C-NMR
(100 MHz, $CDCl_3$)

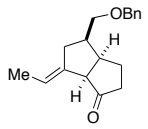



```
Filename          = yk060767_non-data-1-3.j
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk060767
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JAN-2017 22:10:44
Revision_Time     = 20-FEB-2018 20:20:44
Current_Time      = 20-FEB-2018 20:21:13

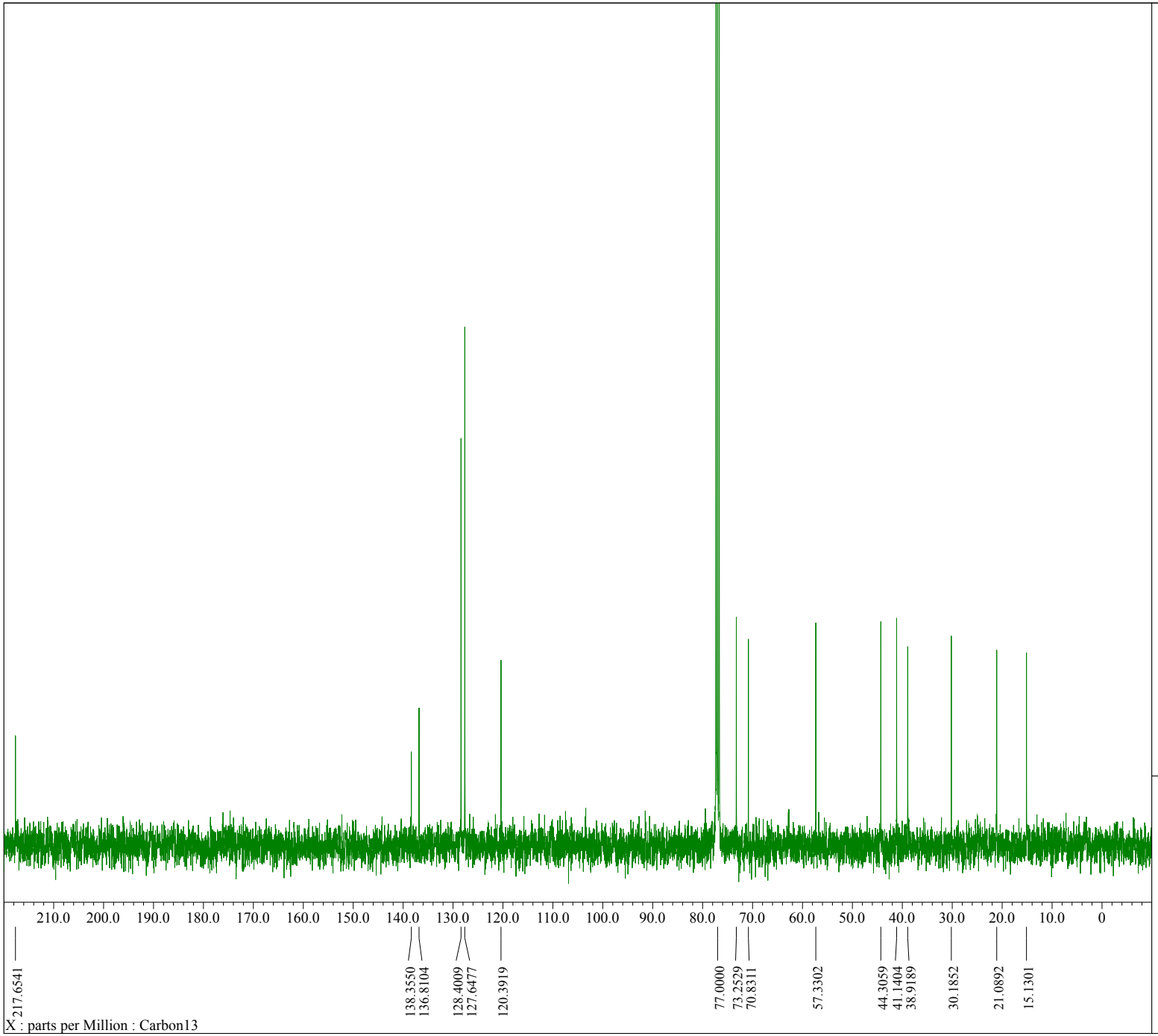
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 21.9[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



25
^{1}H-NMR
(400 MHz, $CDCl_3$)

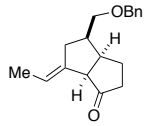



```
Filename          = yk060767_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk060767
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JAN-2017 22:13:17
Revision_Time     = 20-FEB-2018 17:00:43
Current_Time      = 20-FEB-2018 17:01:25

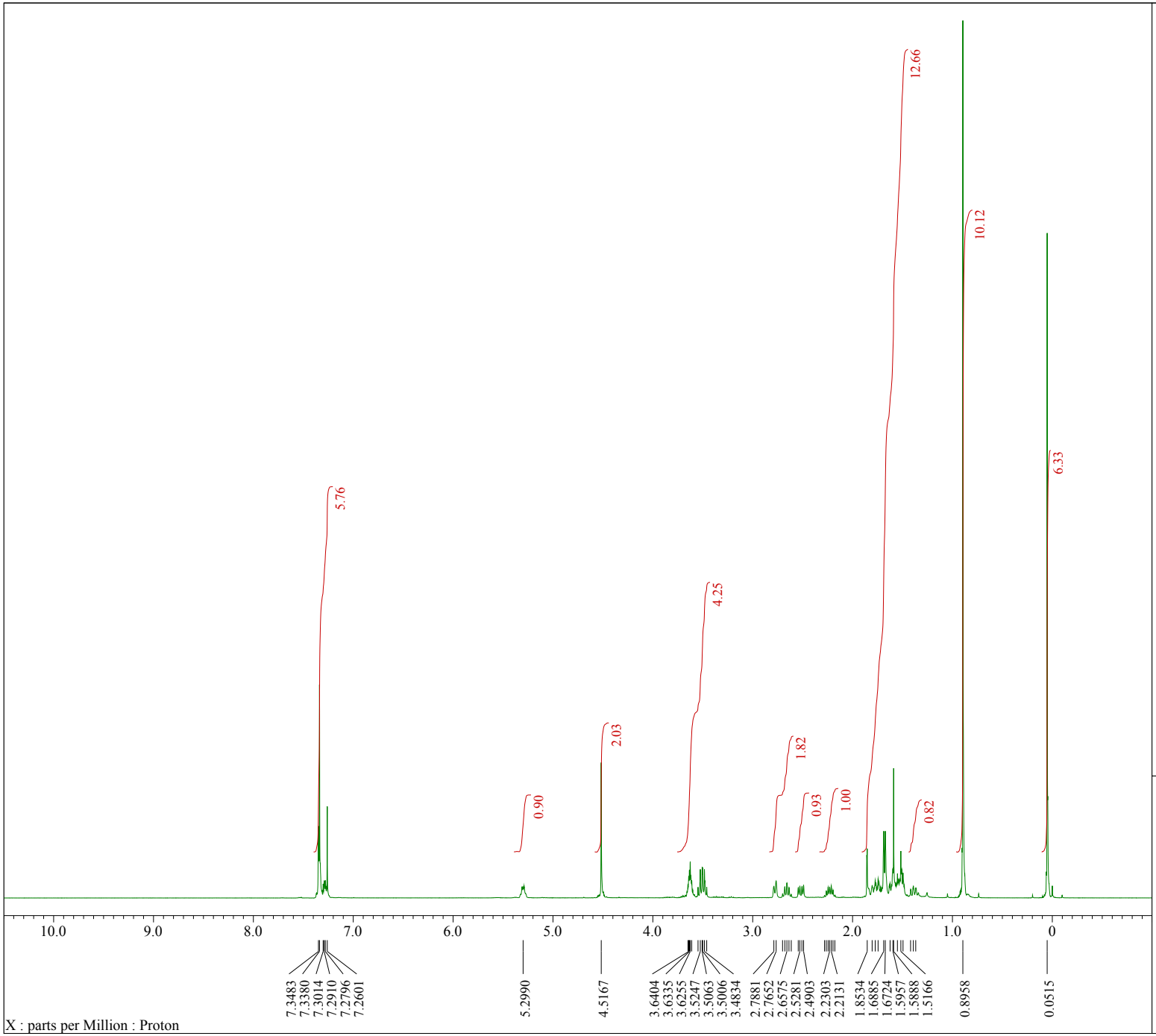
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 22[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



25
^{13}C-NMR
(100 MHz, $CDCl_3$)

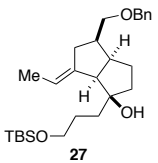



```
Filename          = yk06068data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06068data
Solvent           = CHLOROFORM-D
Creation_Time     = 11-JAN-2017 21:29:03
Revision_Time     = 20-FEB-2018 20:24:27
Current_Time      = 20-FEB-2018 20:24:58

Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

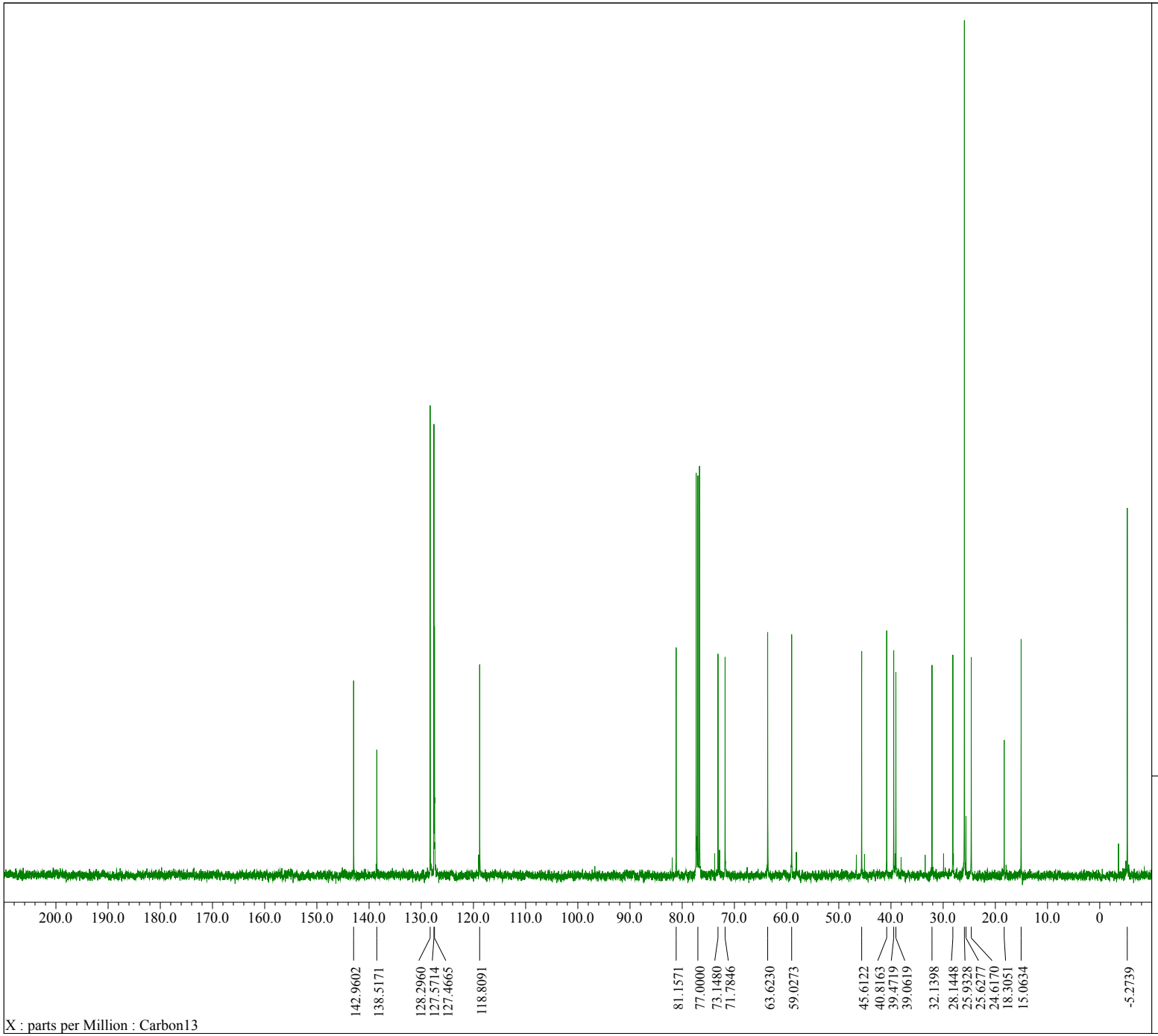
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 16
Total_Scans       = 16

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 22.1[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



27

^{1}H-NMR
(400 MHz, $CDCl_3$)

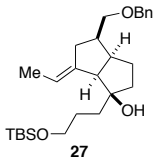



```
Filename          = yk06068data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06068data
Solvent           = CHLOROFORM-D
Creation_Time     =  9-NOV-2016 11:17:20
Revision_Time     = 20-FEB-2018 17:03:17
Current_Time      = 20-FEB-2018 17:03:50

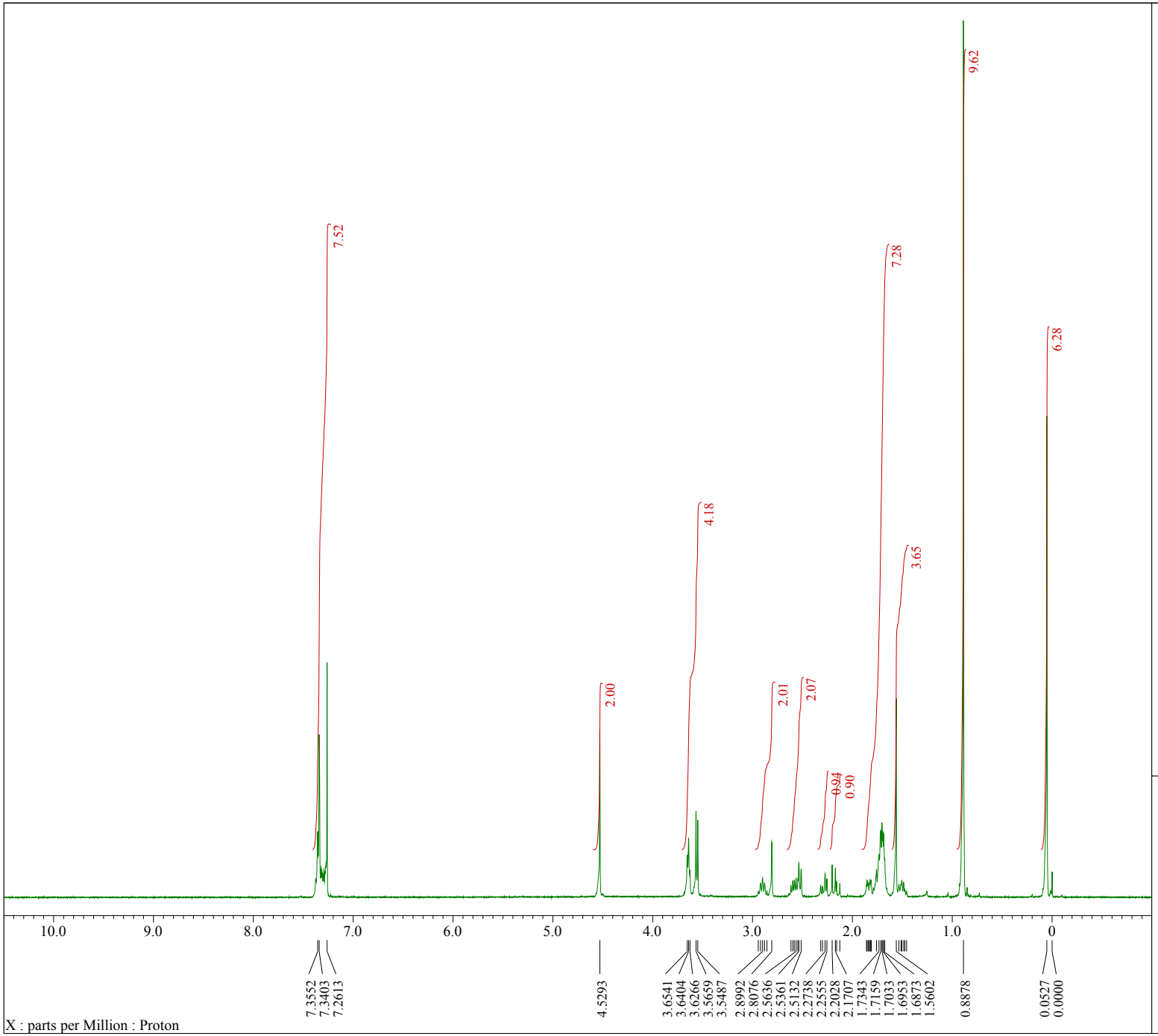
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.4[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



27
^{13}C-NMR
(100 MHz, $CDCl_3$)

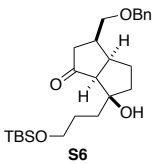



```
Filename          = yk06069_non-data-2-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06069
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JAN-2017 23:56:28
Revision_Time     = 20-FEB-2018 20:28:16
Current_Time      = 20-FEB-2018 20:29:19

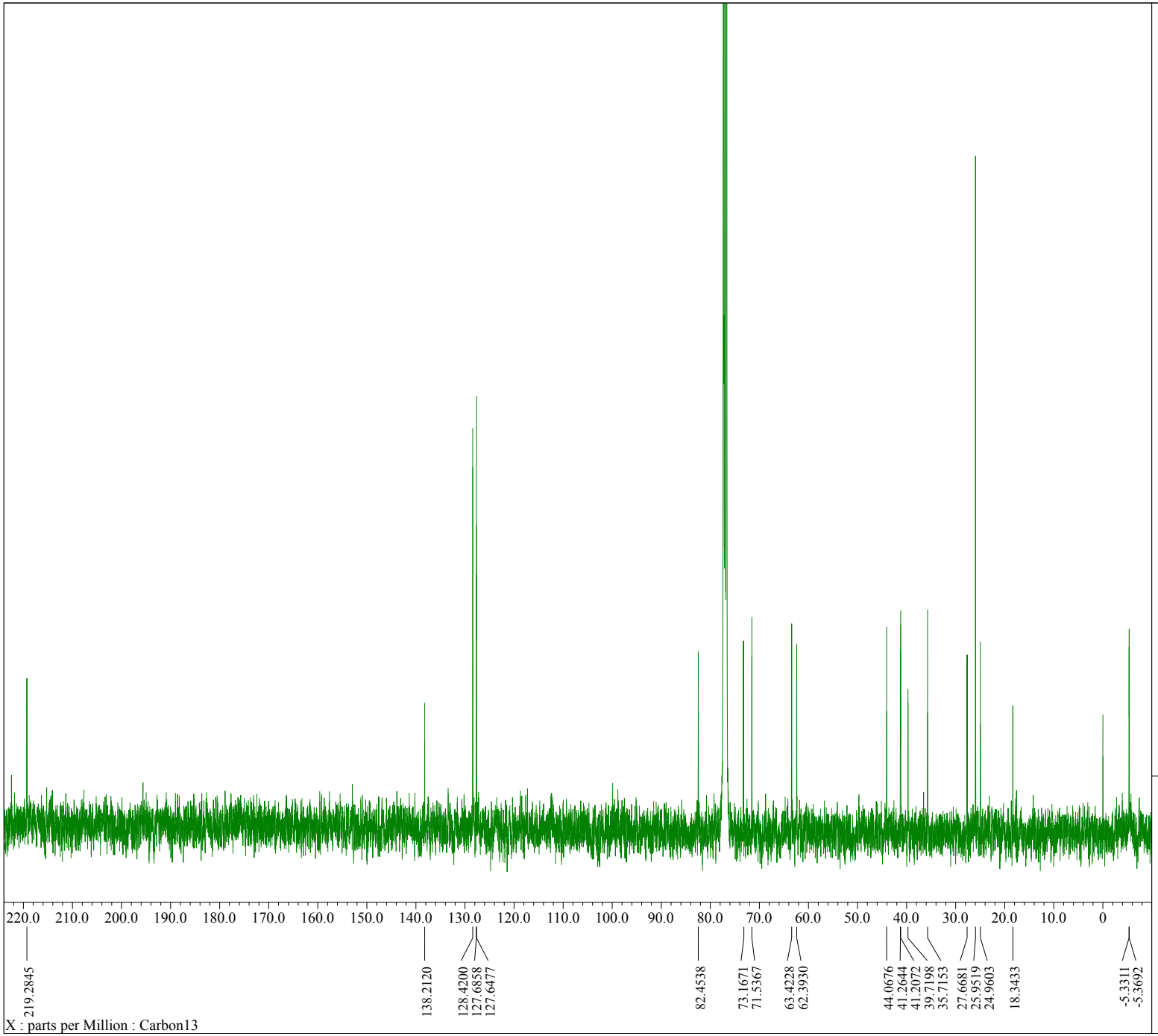
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 21.9[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



S6
^{1}H-NMR
(400 MHz, $CDCl_3$)

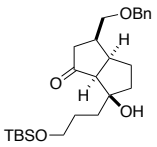



```
Filename          = yk06069_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06069
Solvent           = CHLOROFORM-D
Creation_Time     = 14-JAN-2017 19:55:12
Revision_Time     = 20-FEB-2018 17:06:03
Current_Time      = 20-FEB-2018 17:07:00

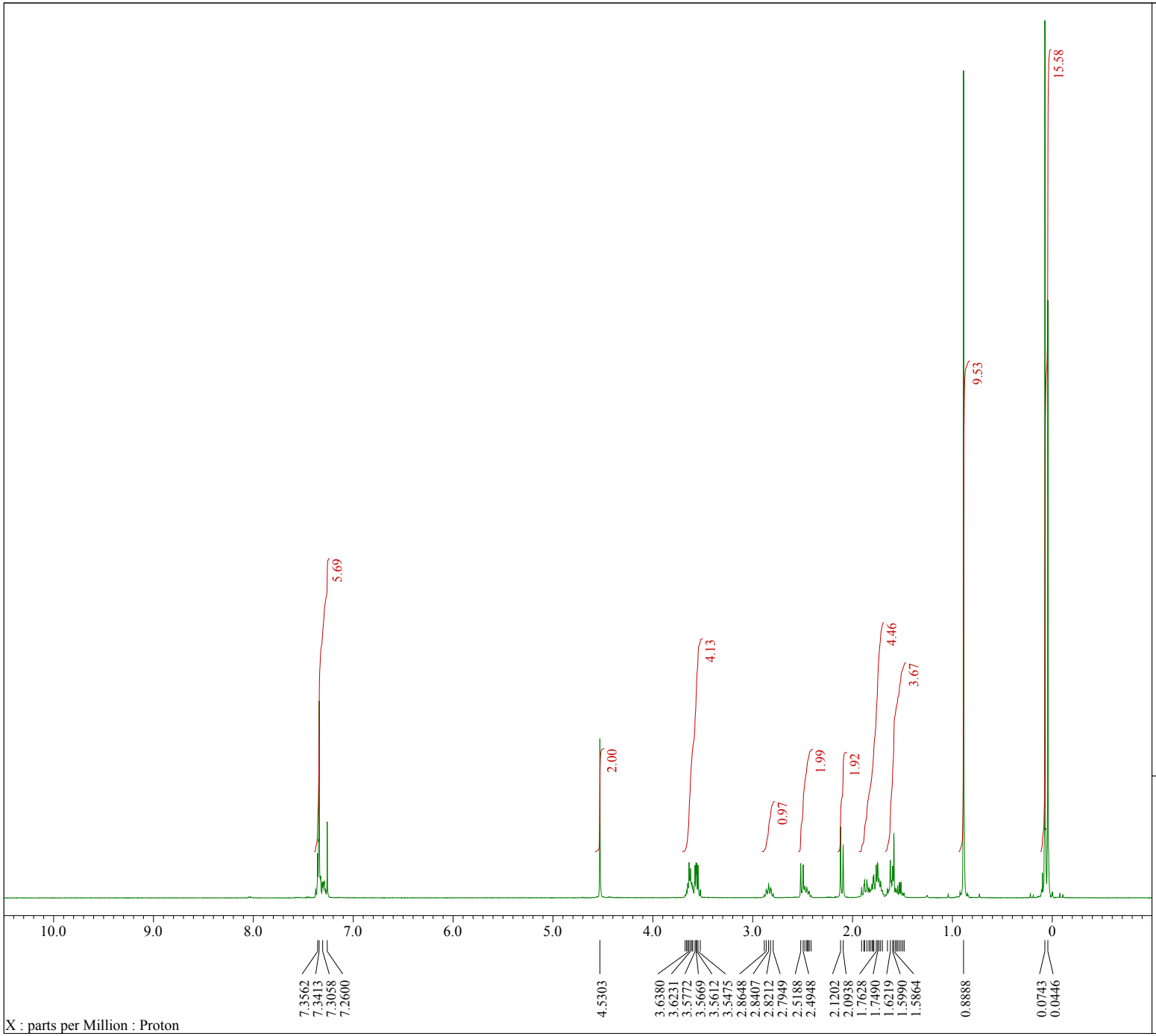
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 6000
Total_Scans       = 6000

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 21.4[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



S6
^{13}C-NMR
(100 MHz, $CDCl_3$)

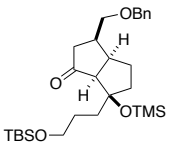



```
Filename          = yk06070_non-data-1-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06070
Solvent           = CHLOROFORM-D
Creation_Time     = 16-JAN-2017 22:56:03
Revision_Time     = 20-FEB-2018 20:32:09
Current_Time      = 20-FEB-2018 20:32:36

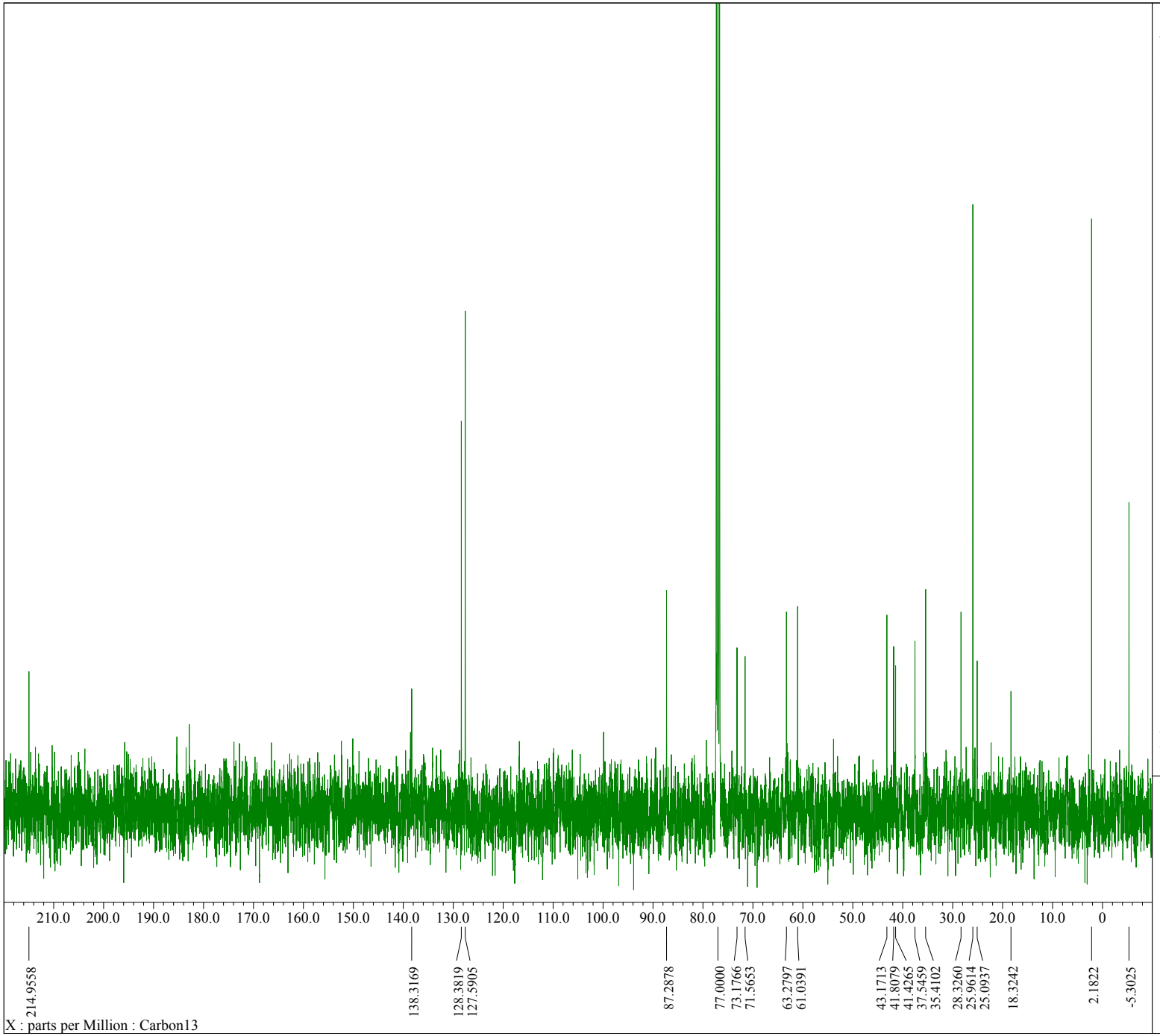
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 21.4[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



28
^{1}H-NMR
(400 MHz, $CDCl_3$)

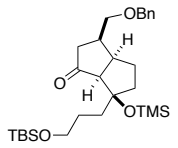



```
Filename          = yk06070_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06070
Solvent           = CHLOROFORM-D
Creation_Time     = 16-JAN-2017 23:04:36
Revision_Time     = 20-FEB-2018 17:09:10
Current_Time      = 20-FEB-2018 17:09:51

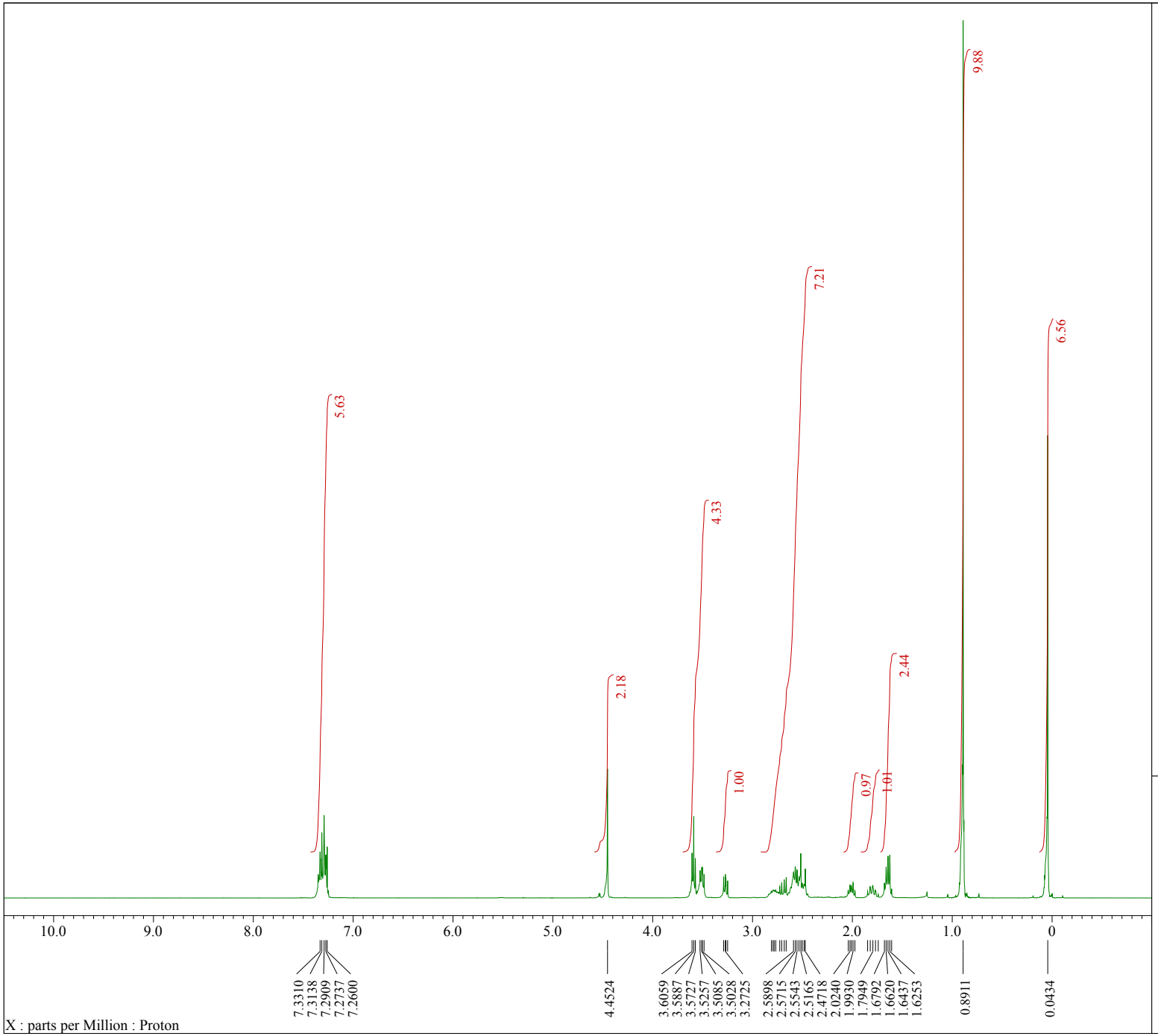
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay  = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 21.5[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



28
^{13}C-NMR
(100 MHz, $CDCl_3$)

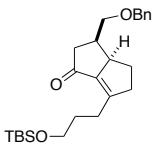



```
Filename          = yk06071_non-data-1-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06077
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JAN-2017 22:06:54
Revision_Time     = 20-FEB-2018 20:36:04
Current_Time      = 20-FEB-2018 20:36:44

Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

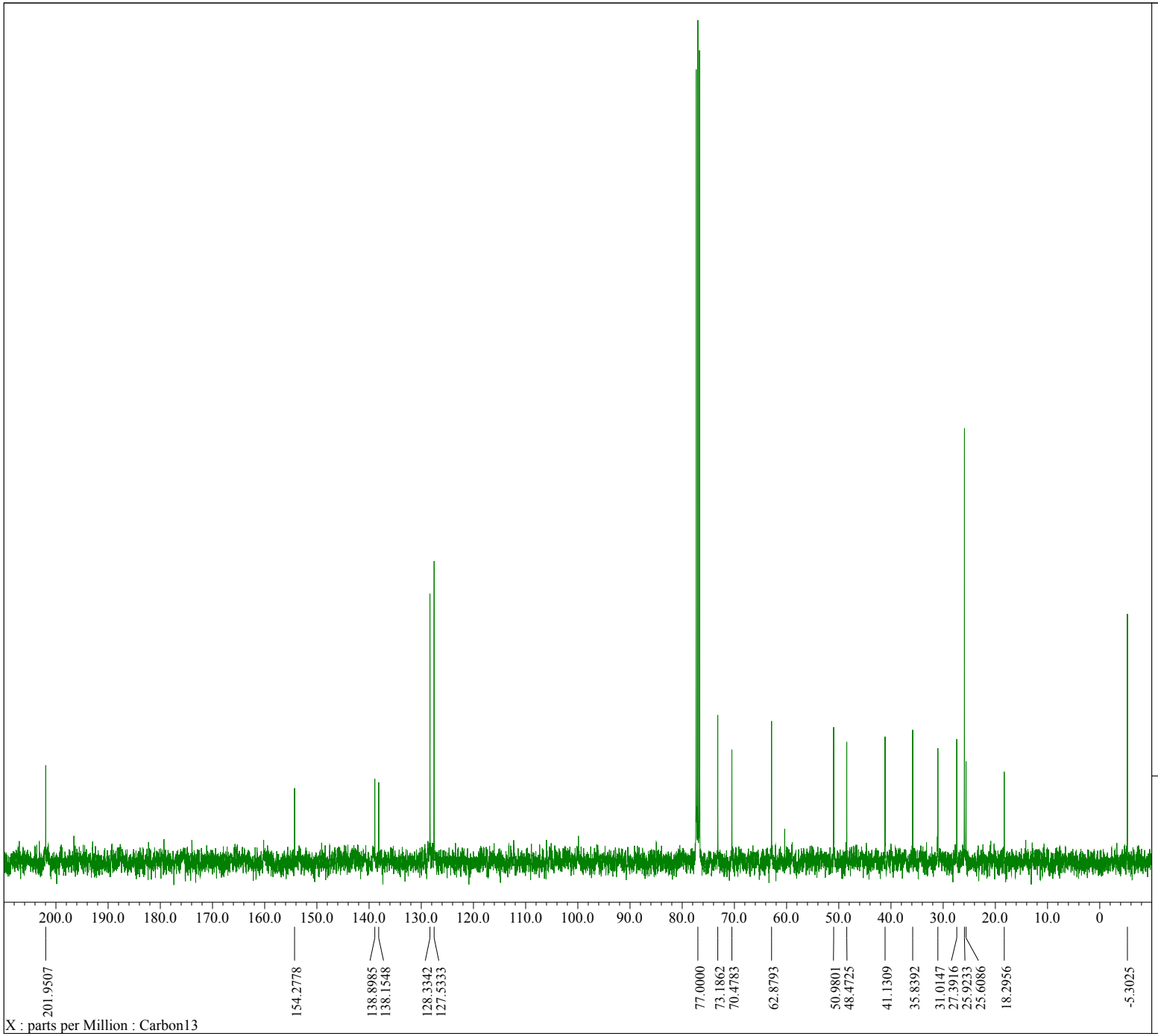
Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 21.9[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



29

^{1}H-NMR

(400 MHz, $CDCl_3$)

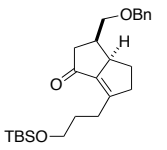



```
Filename          = yk06071data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06071data
Solvent           = CHLOROFORM-D
Creation_Time     = 13-JAN-2017 20:52:10
Revision_Time     = 20-FEB-2018 17:11:34
Current_Time      = 20-FEB-2018 17:12:04

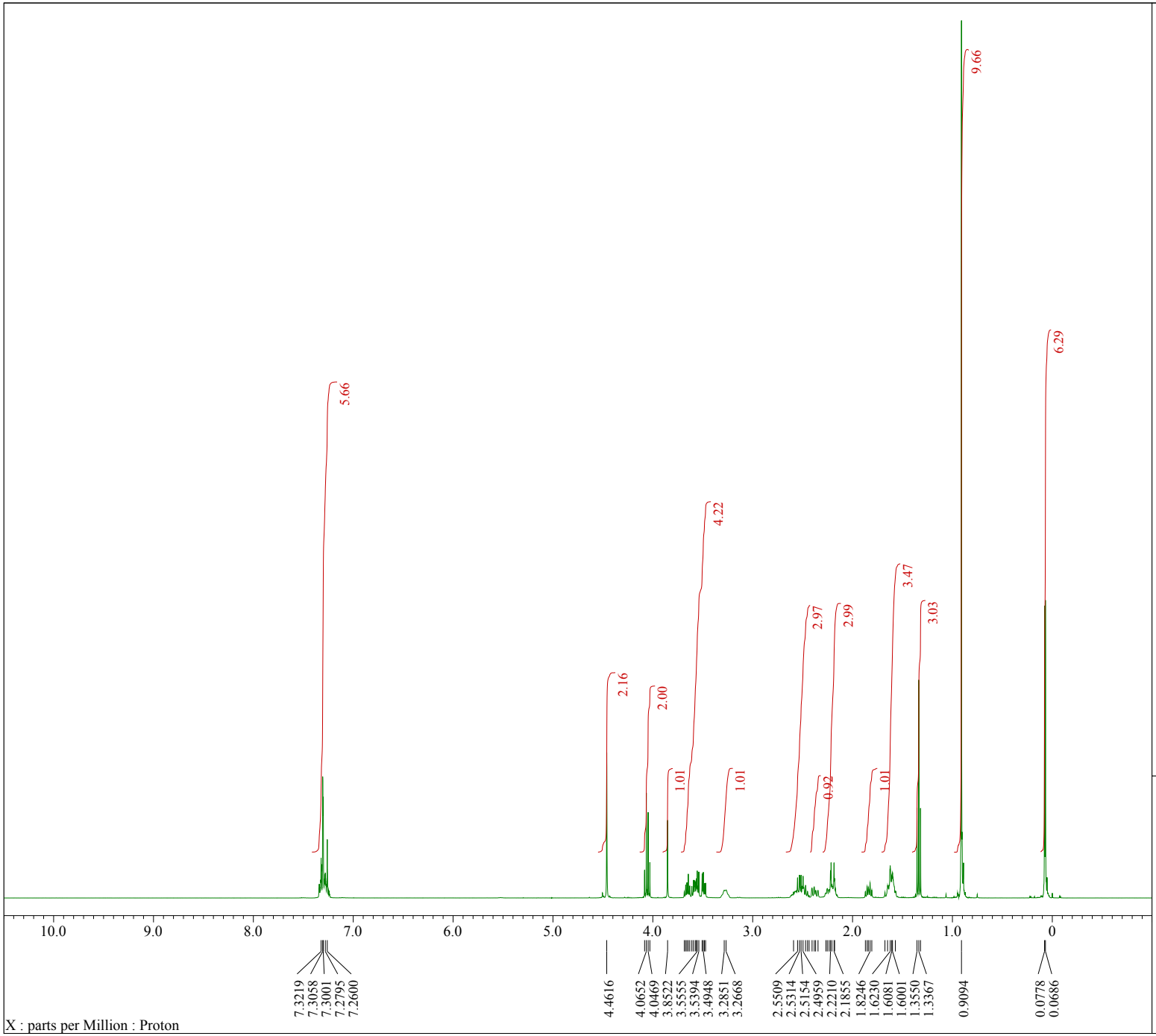
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 22.1[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



29
^{13}C-NMR
(100 MHz, $CDCl_3$)

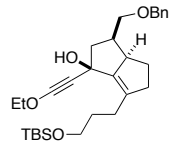



```
Filename          = yk06072_non-data-2-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06079
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JAN-2017 23:51:55
Revision_Time     = 20-FEB-2018 20:40:01
Current_Time      = 20-FEB-2018 20:40:50

Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

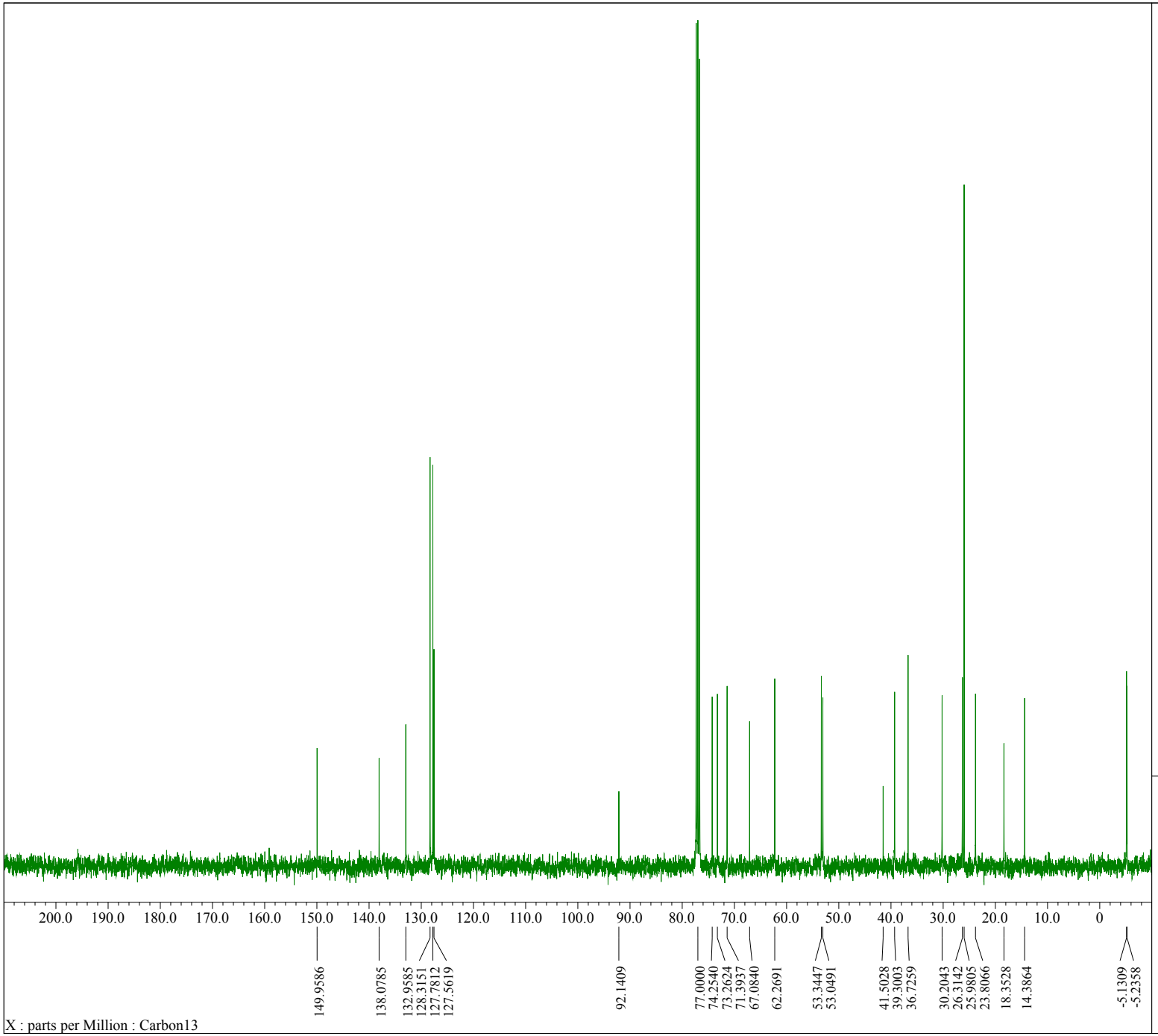
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 21.9[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



30

^{1}H-NMR
(400 MHz, $CDCl_3$)

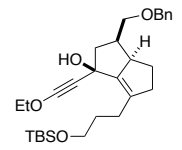



```
Filename          = yk06072data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06072data
Solvent           = CHLOROFORM-D
Creation_Time     = 13-NOV-2016 17:26:34
Revision_Time     = 20-FEB-2018 17:14:03
Current_Time      = 20-FEB-2018 17:14:48

Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

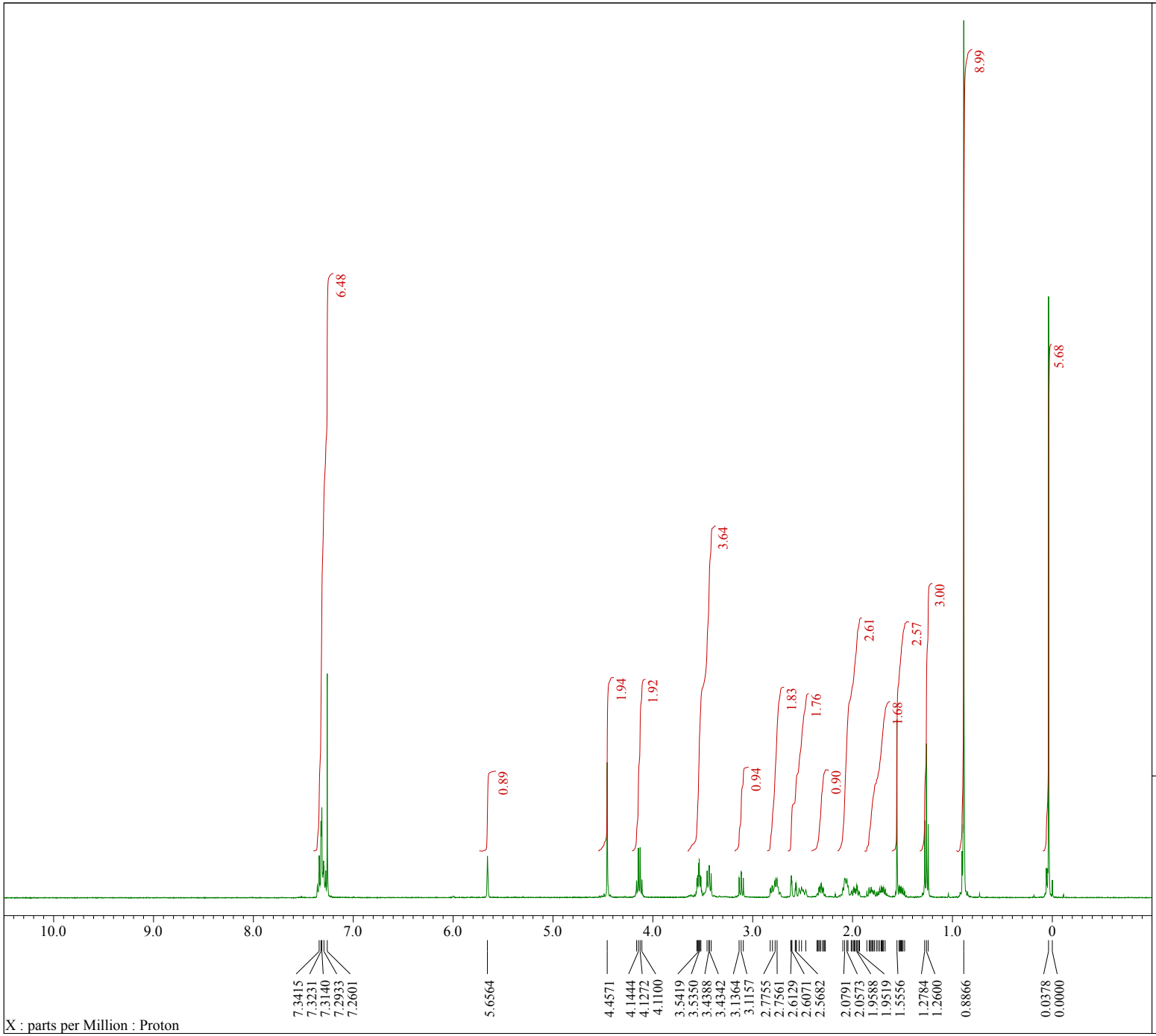
Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.3[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



30

^{13}C-NMR

(100 MHz, $CDCl_3$)

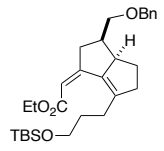



```
Filename          = yk06073data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06080data
Solvent           = CHLOROFORM-D
Creation_Time     = 13-JAN-2017 17:54:50
Revision_Time     = 20-FEB-2018 20:44:45
Current_Time      = 20-FEB-2018 20:45:11

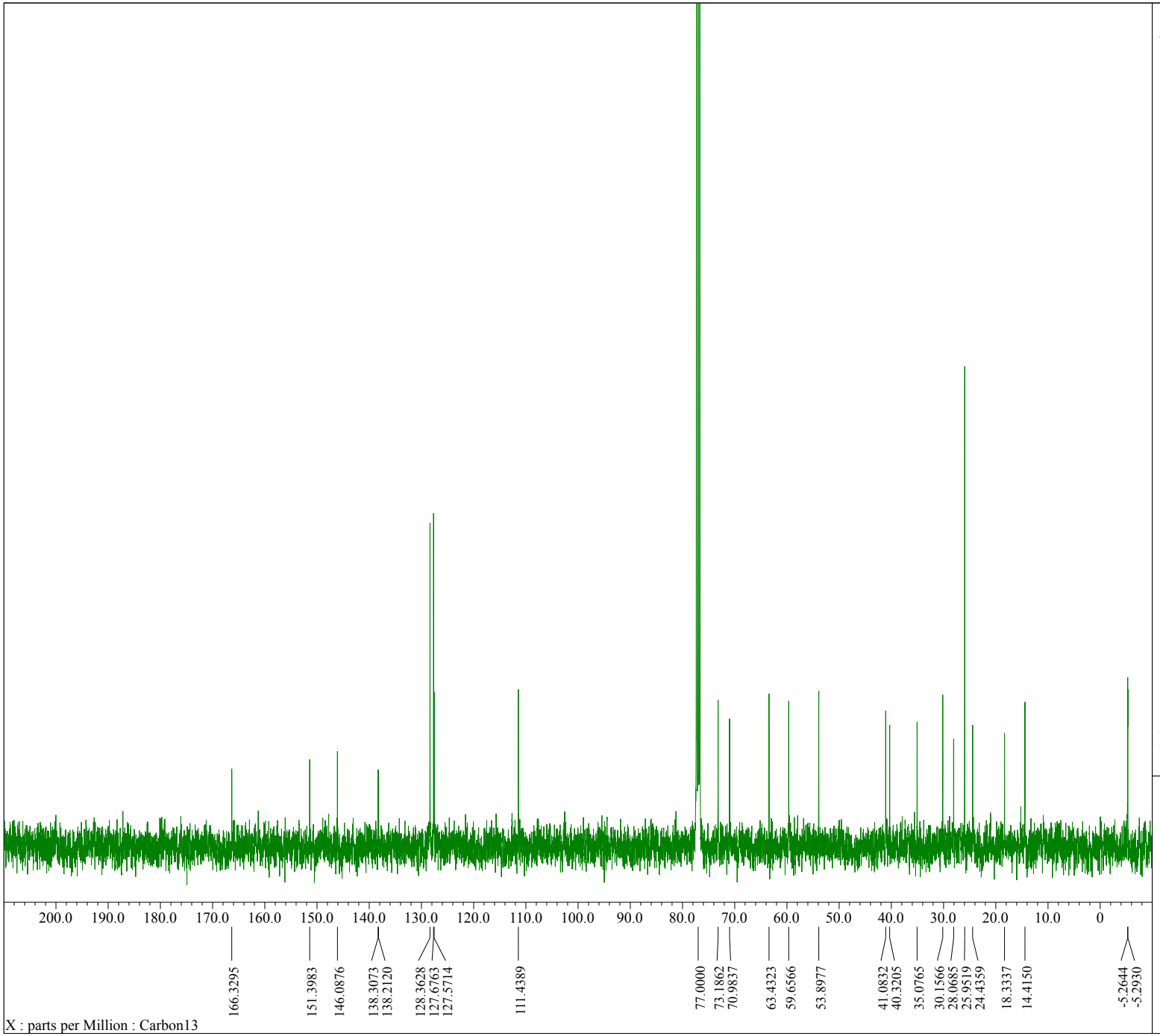
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 22.2[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



32
^{1}H-NMR
(400 MHz, $CDCl_3$)

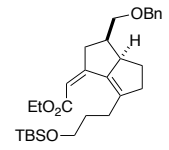



```
Filename          = yk06073_bcm-2-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06073
Solvent           = CHLOROFORM-D
Creation_Time     = 13-JAN-2017 22:32:00
Revision_Time     = 20-FEB-2018 17:16:57
Current_Time      = 20-FEB-2018 17:17:31

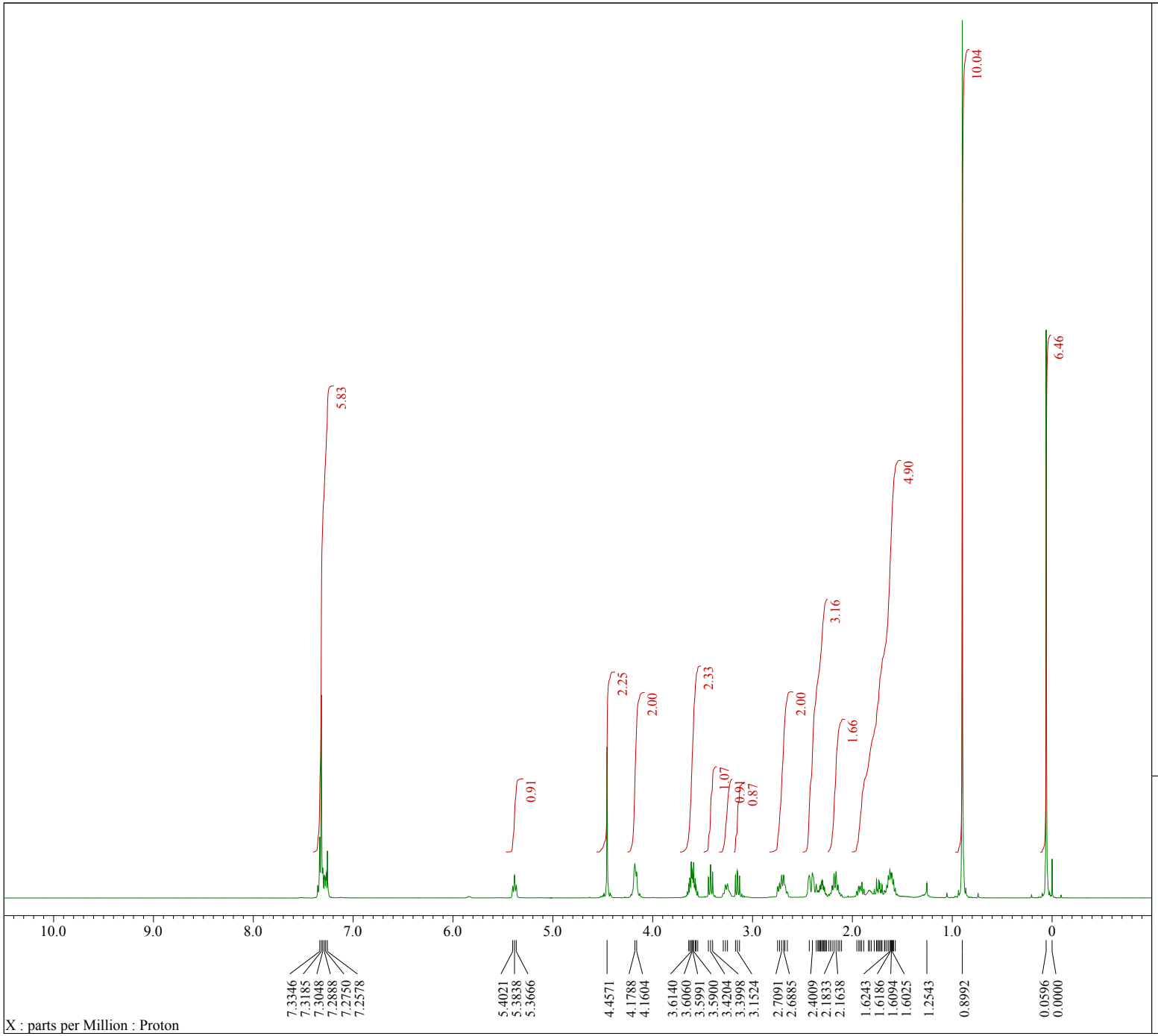
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 22.1[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



32
^{13}C-NMR
(100 MHz, $CDCl_3$)

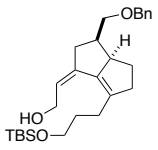



```
Filename          = yk06078pt_non-data-1-3.
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06078pt
Solvent           = CHLOROFORM-D
Creation_Time     = 13-NOV-2016 18:14:30
Revision_Time     = 20-FEB-2018 20:48:59
Current_Time      = 20-FEB-2018 20:50:20

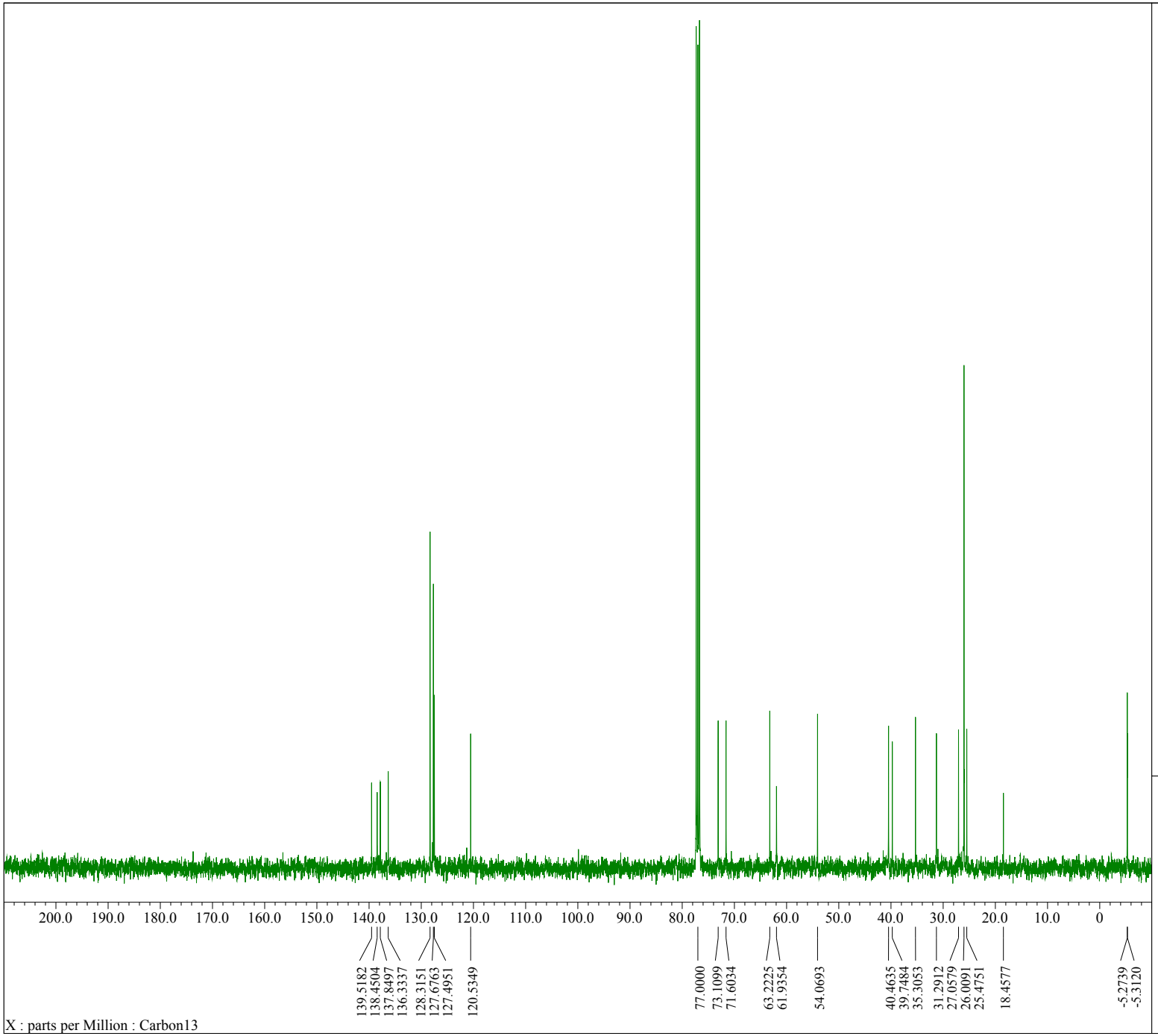
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 23.2[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



33
^{1}H-NMR
(400 MHz, $CDCl_3$)

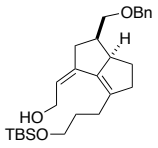



```
Filename          = yk06078pt_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06078pt
Solvent           = CHLOROFORM-D
Creation_Time     = 13-NOV-2016 18:22:13
Revision_Time     = 20-FEB-2018 17:19:07
Current_Time      = 20-FEB-2018 17:19:38

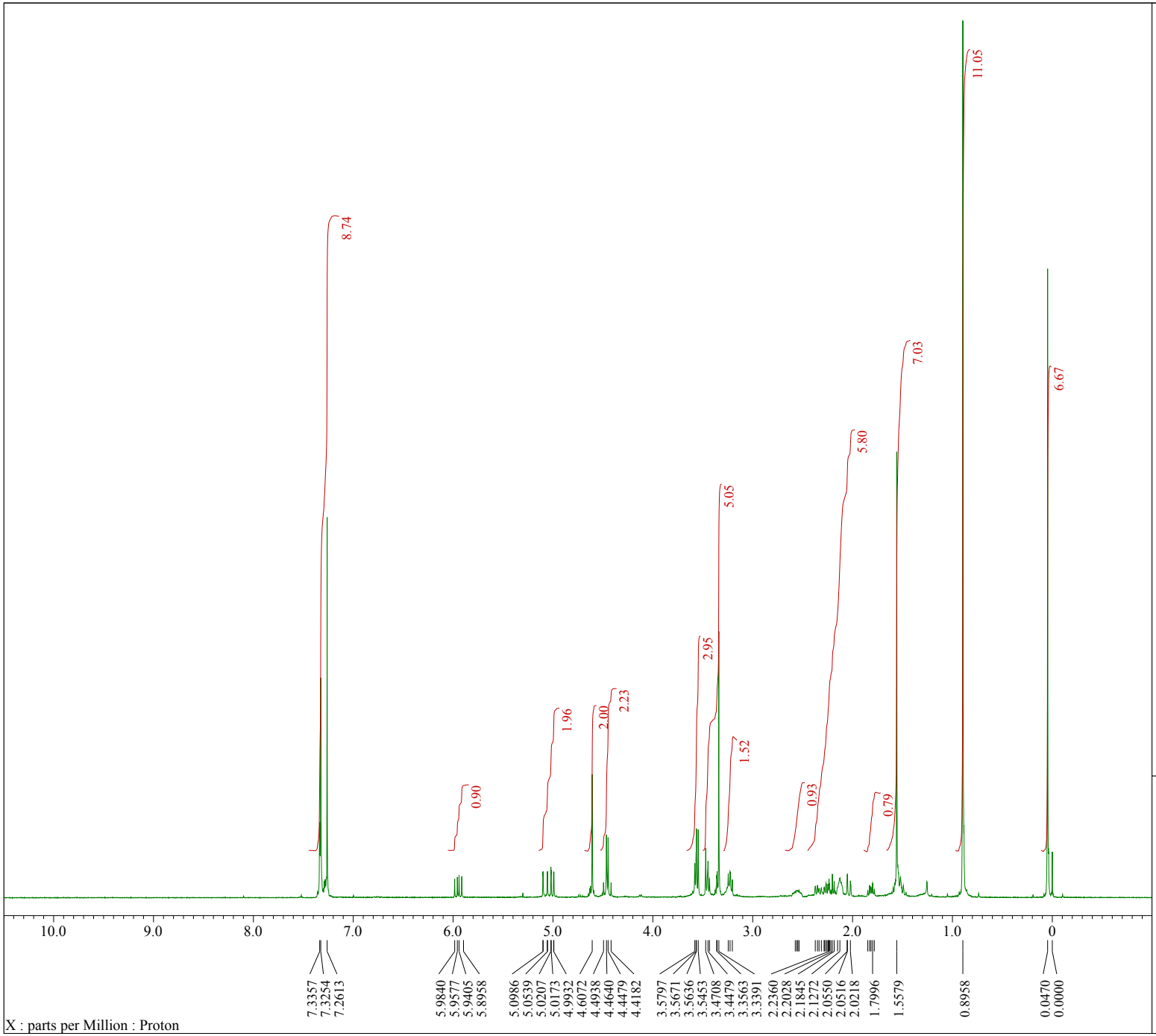
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 128
Total_Scans       = 128

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 23.3[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



33
^{13}C-NMR
(100 MHz, $CDCl_3$)

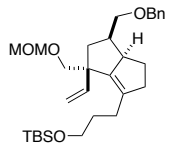



```
Filename          = yk06103data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06103data
Solvent           = CHLOROFORM-D
Creation_Time     =  9-JAN-2017 13:00:47
Revision_Time     = 20-FEB-2018 20:54:20
Current_Time      = 20-FEB-2018 20:54:58

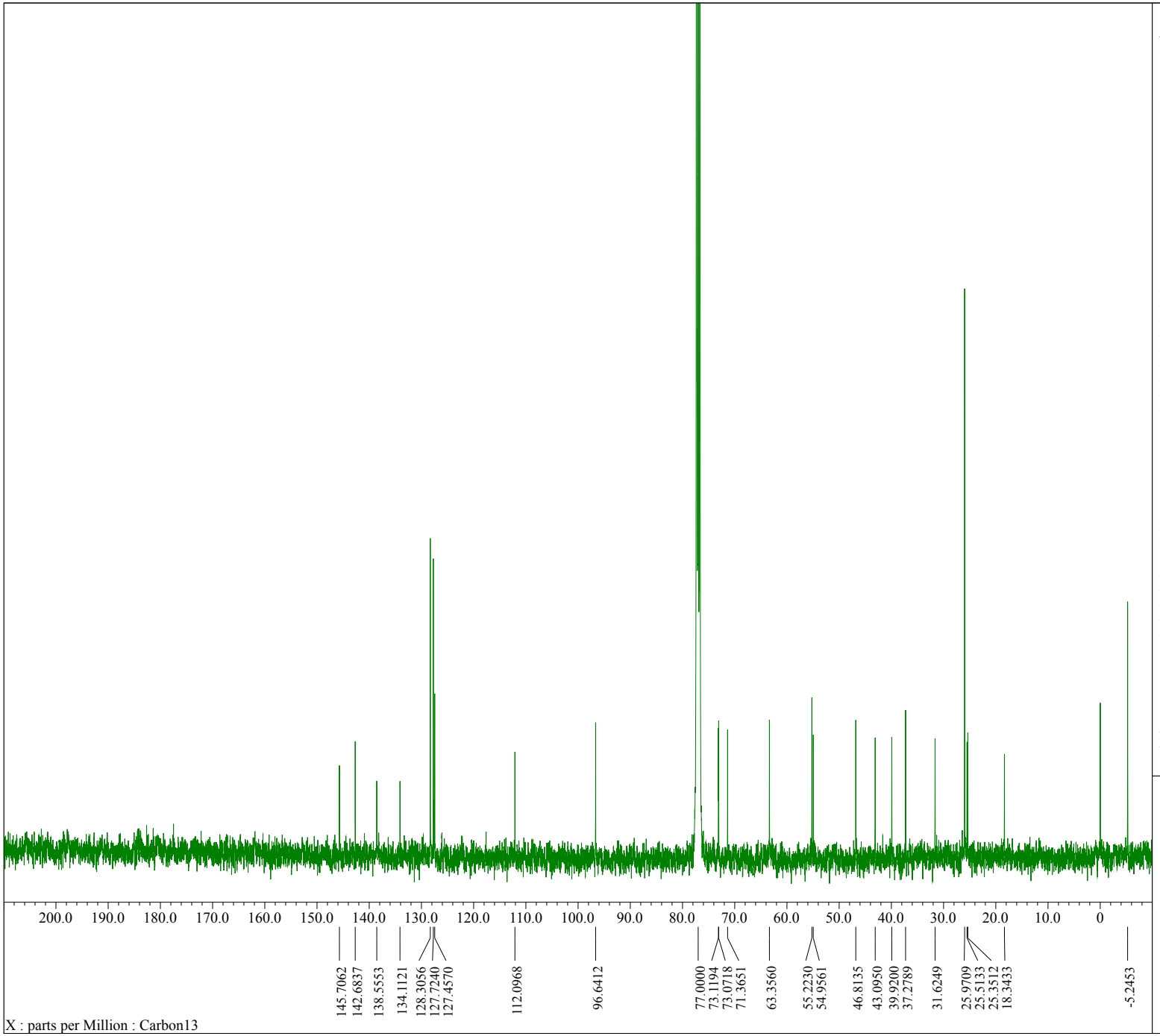
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 16
Total_Scans       = 16

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 21.8[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



37
^{1}H-NMR
(400 MHz, $CDCl_3$)

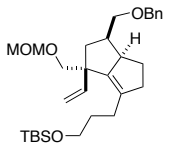



```
Filename          = yk06103_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = 103
Solvent           = CHLOROFORM-D
Creation_Time     = 15-JAN-2017 11:36:02
Revision_Time     = 20-FEB-2018 17:21:40
Current_Time      = 20-FEB-2018 17:22:28

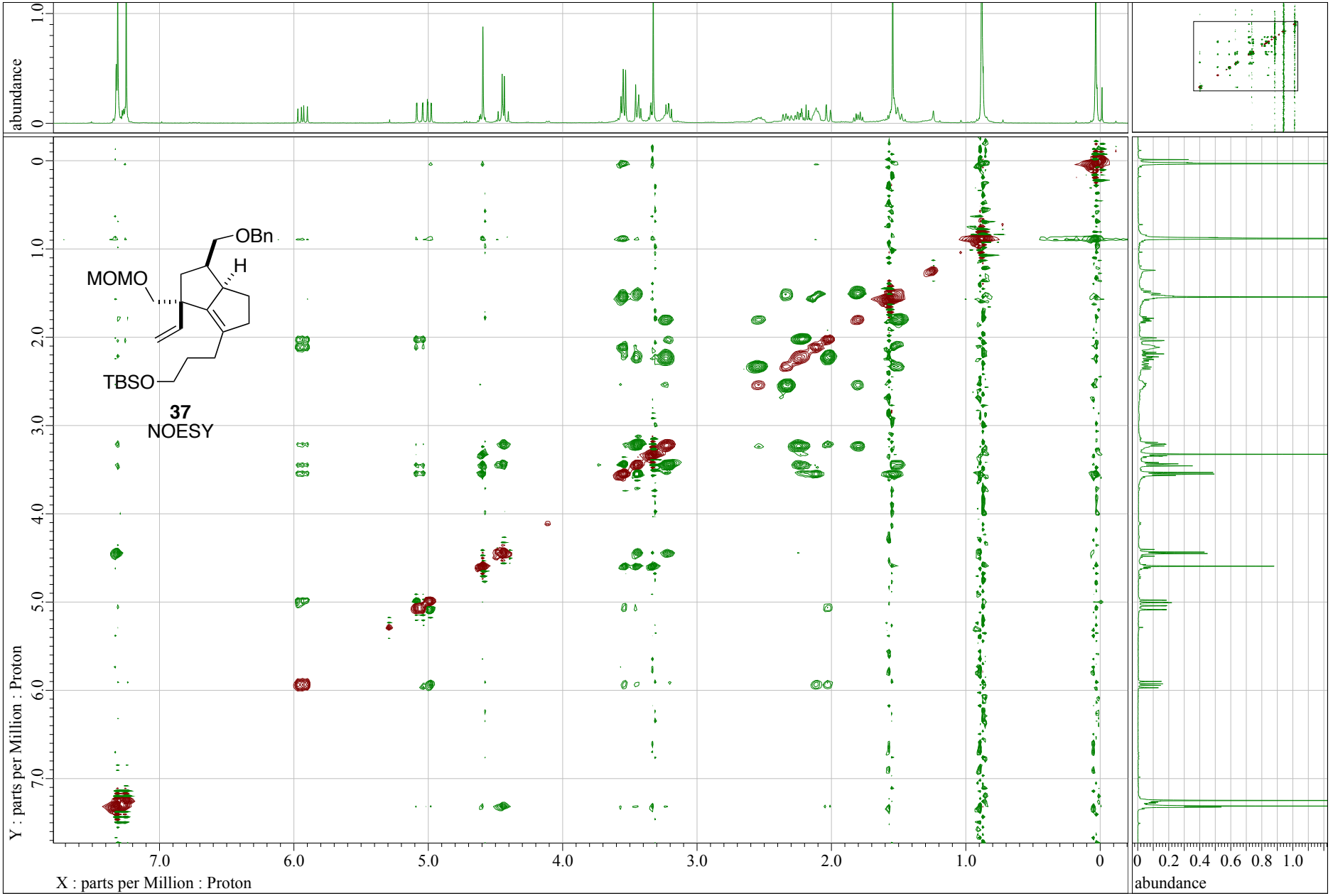
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

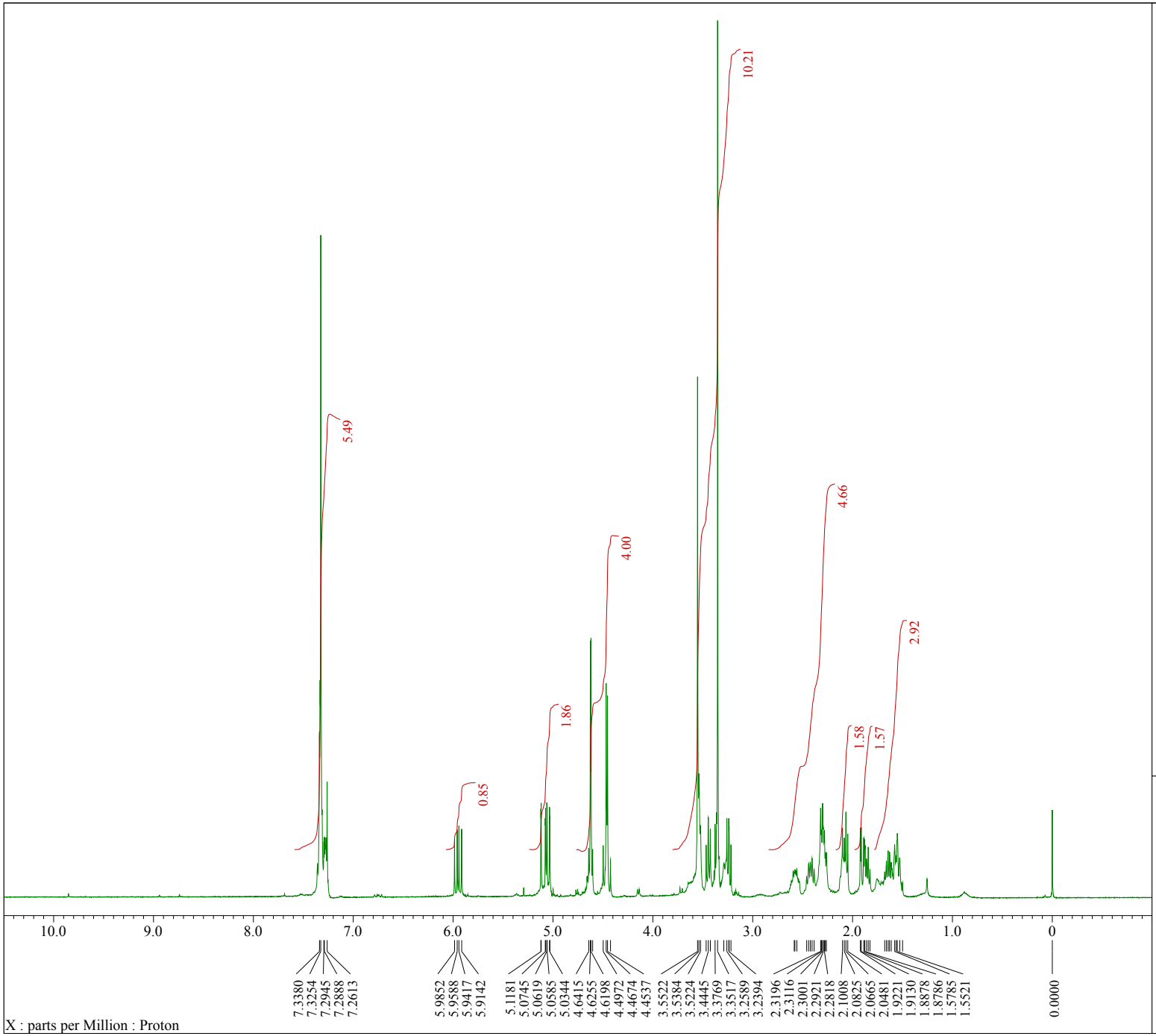
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 20000
Total_Scans       = 20000

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 21[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



37
^{13}C-NMR
(100 MHz, $CDCl_3$)


abundance
OBn
H
MOMO
TBSO
37
NOESY
Y : parts per Million : Proton
X : parts per Million : Proton
abundance

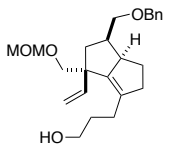



```
Filename          = yk06104_non-data-1-3.jd
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = 104
Solvent           = CHLOROFORM-D
Creation_Time     = 15-JAN-2017 09:19:14
Revision_Time     = 20-FEB-2018 20:59:23
Current_Time      = 20-FEB-2018 21:00:24

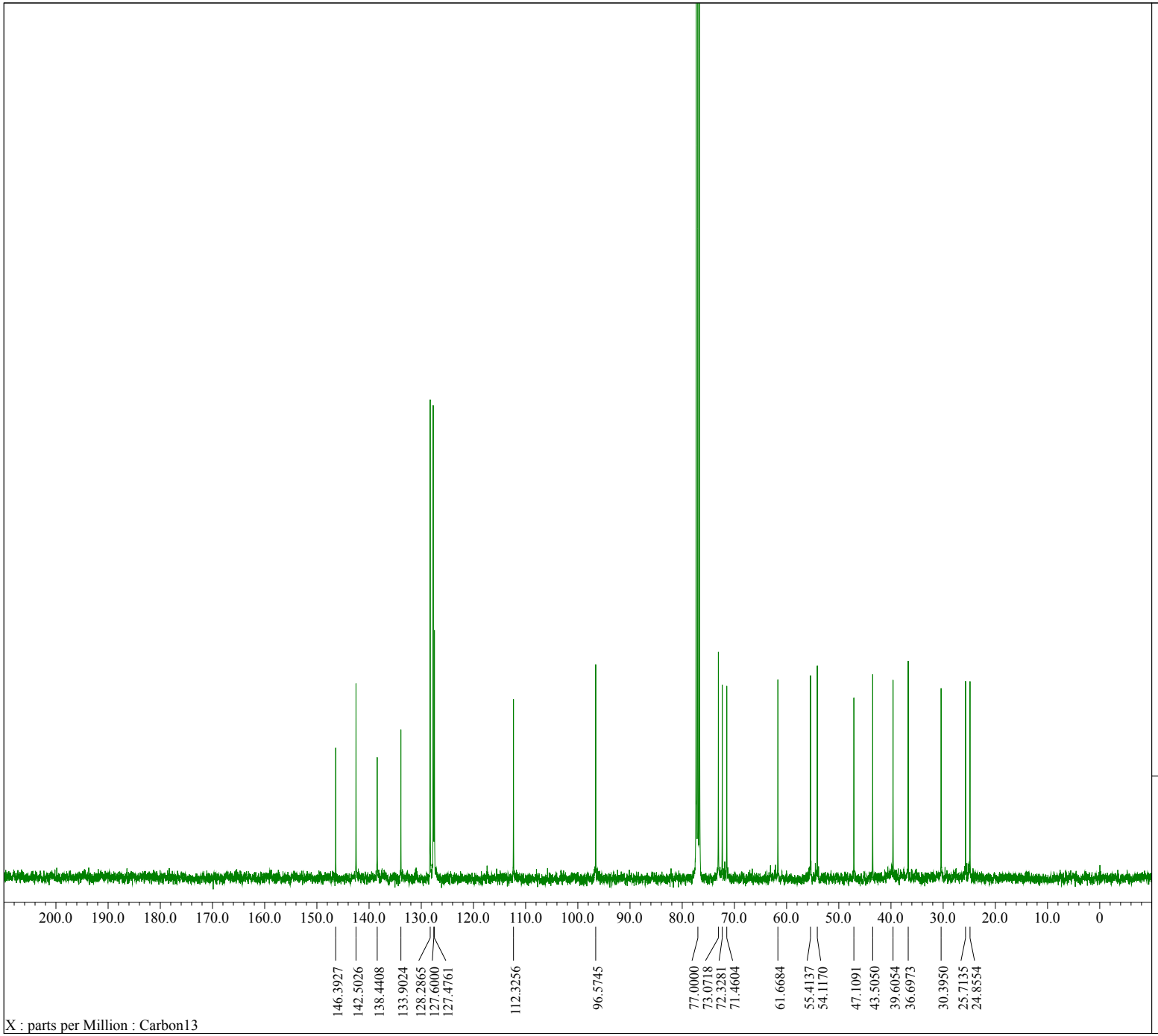
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 2
Total_Scans       = 2

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 20.9[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



S7
^{1}H-NMR
(400 MHz, $CDCl_3$)

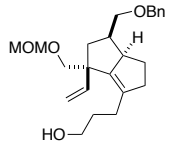



```
Filename          = yk06104_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = 104
Solvent           = CHLOROFORM-D
Creation_Time     = 15-JAN-2017 09:25:34
Revision_Time     = 20-FEB-2018 17:25:14
Current_Time      = 20-FEB-2018 17:25:57

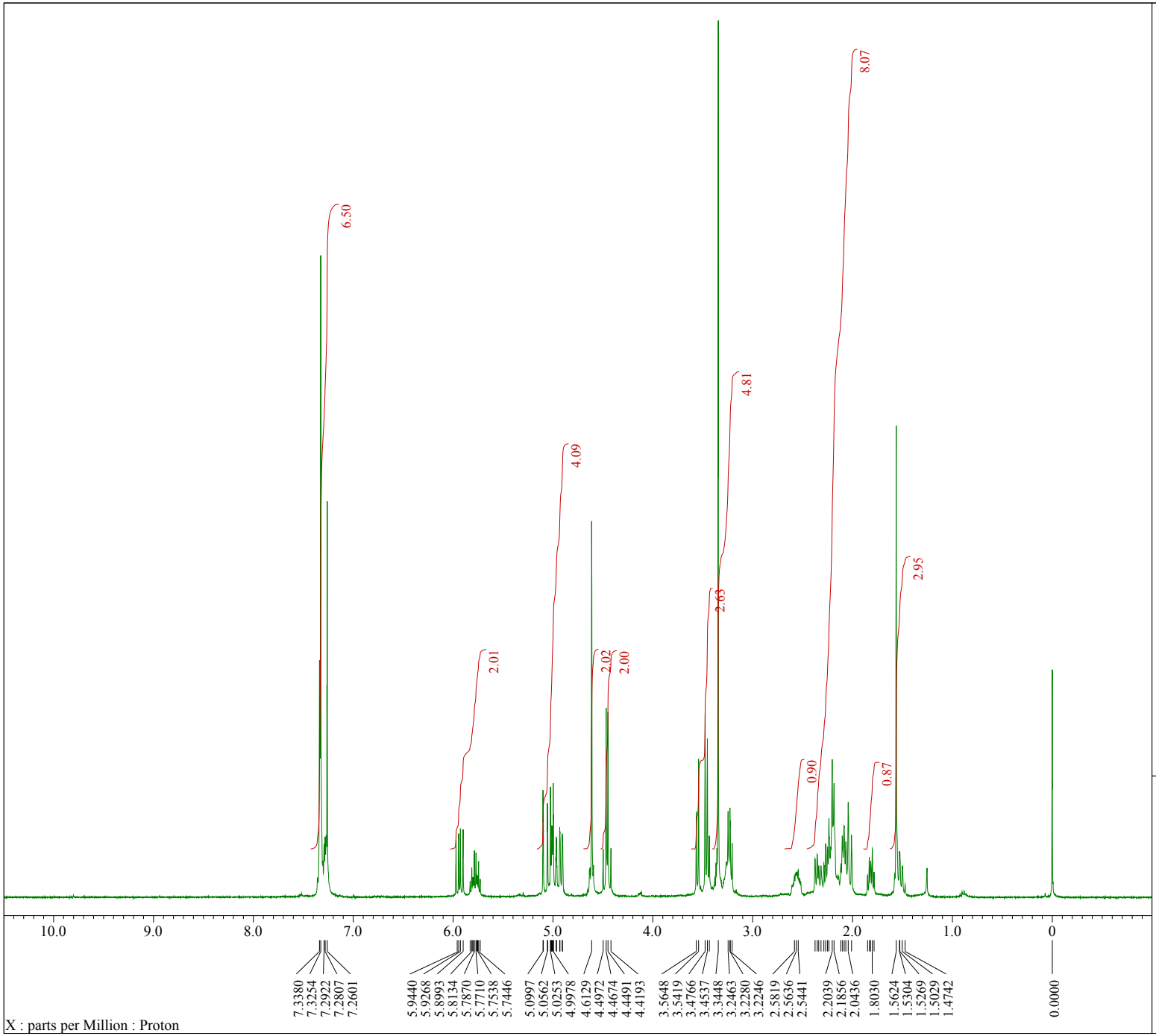
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 3000
Total_Scans       = 3000

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 21[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



S7
^{13}C-NMR
(100 MHz, $CDCl_3$)

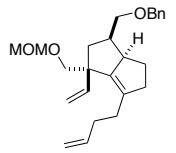



```
Filename          = yk06121data_non-data-2-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06121data
Solvent           = CHLOROFORM-D
Creation_Time     = 22-DEC-2016 15:45:26
Revision_Time     = 20-FEB-2018 21:04:12
Current_Time      = 20-FEB-2018 21:04:50

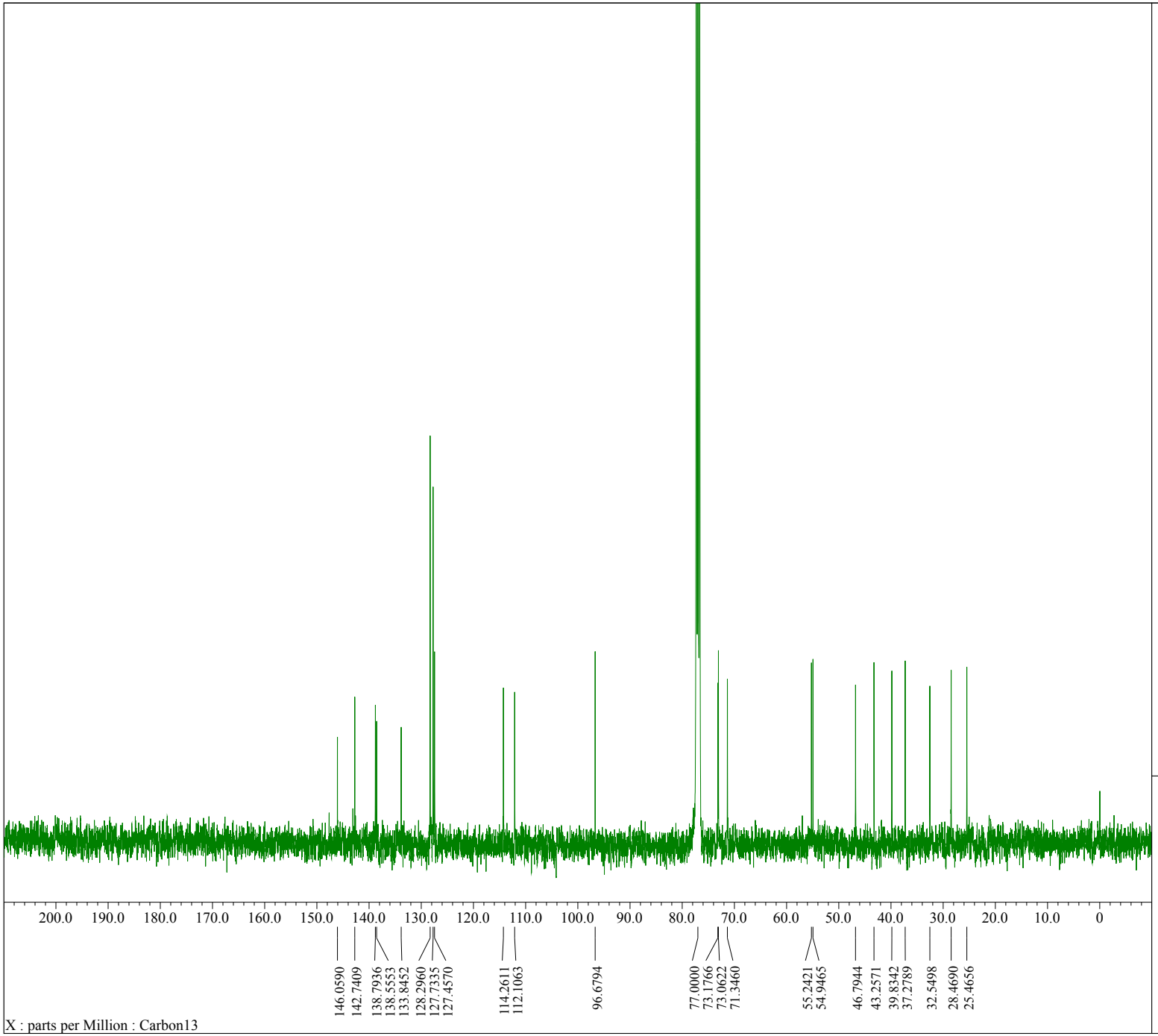
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 23.1[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



38
^{1}H-NMR
(400 MHz, $CDCl_3$)

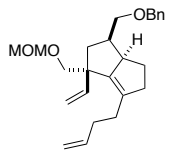



```
Filename          = yk06121data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06121data
Solvent           = CHLOROFORM-D
Creation_Time     = 22-DEC-2016 01:03:56
Revision_Time     = 20-FEB-2018 17:27:28
Current_Time      = 20-FEB-2018 17:28:08

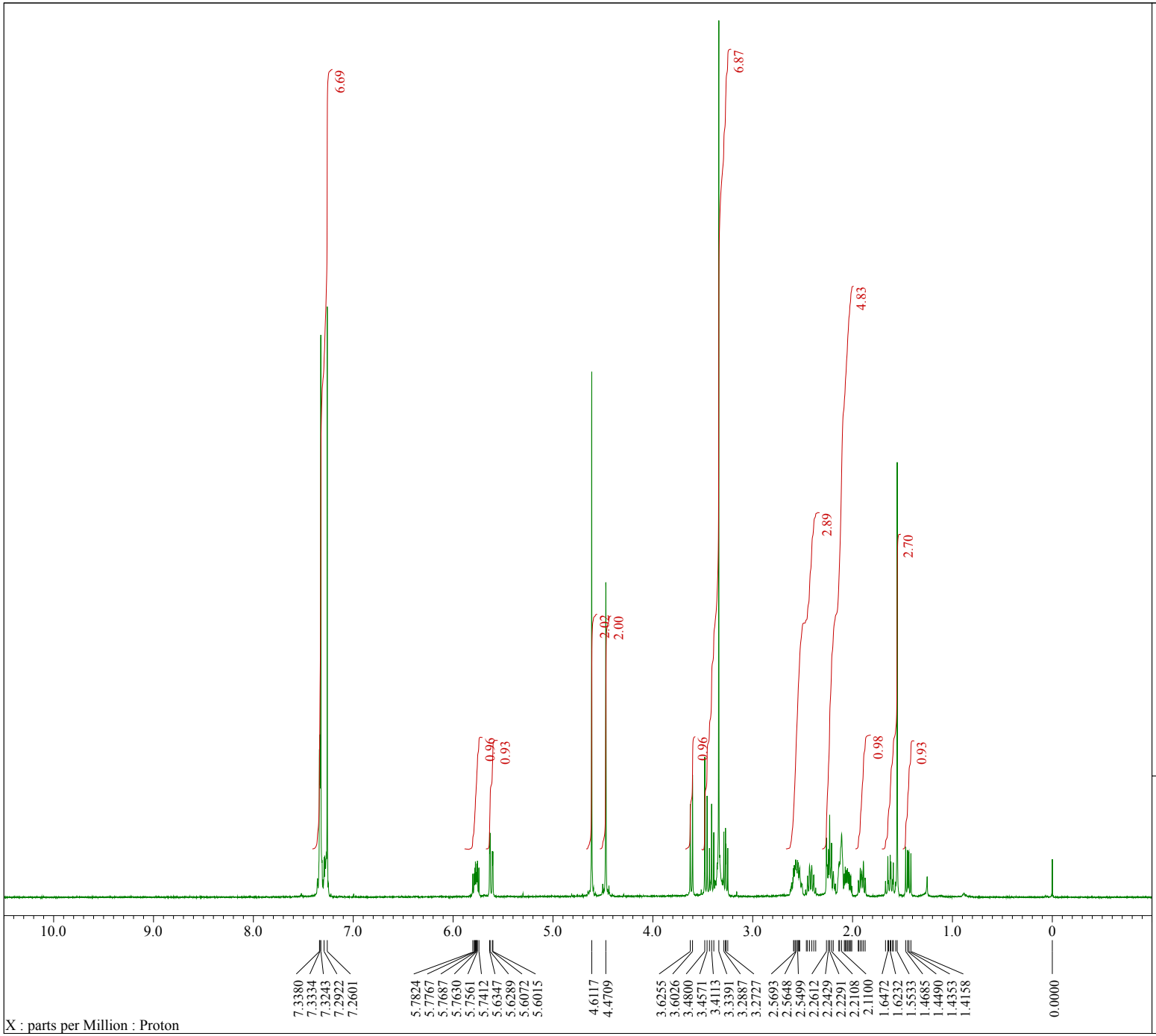
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 6000
Total_Scans       = 6000

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 22.8[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



38
^{13}C-NMR
(100 MHz, $CDCl_3$)

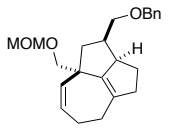



```
Filename          = yk06122data_non-data-1-
Author            = delta
Experiment        = single_pulse.jxp
Sample_Id         = yk06122data
Solvent           = CHLOROFORM-D
Creation_Time     = 13-JAN-2017 11:28:55
Revision_Time     = 20-FEB-2018 21:08:19
Current_Time      = 20-FEB-2018 21:10:51

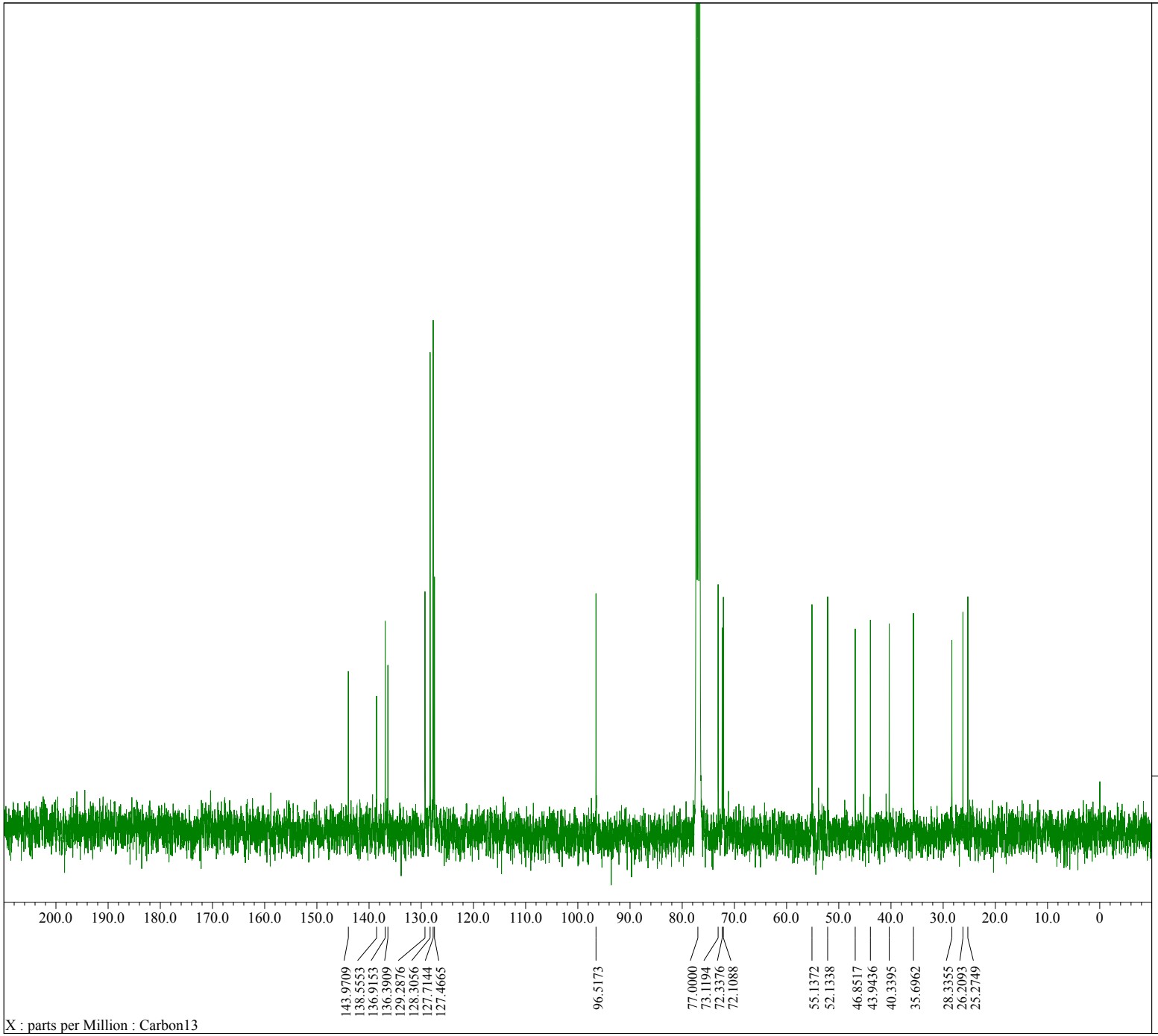
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 22[dC]
X_90_Width        = 10.025[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1[dB]
X_Pulse           = 5.0125[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 7.18365952[s]
```



39
^{1}H-NMR
(400 MHz, $CDCl_3$)

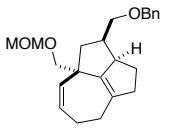



```
Filename          = yk06122data_bcm-1-3.jdf
Author            = delta
Experiment        = single_pulse_dec.jxp
Sample_Id         = yk06122data
Solvent           = CHLOROFORM-D
Creation_Time     = 23-DEC-2016 01:02:27
Revision_Time     = 20-FEB-2018 17:29:40
Current_Time      = 20-FEB-2018 17:30:21

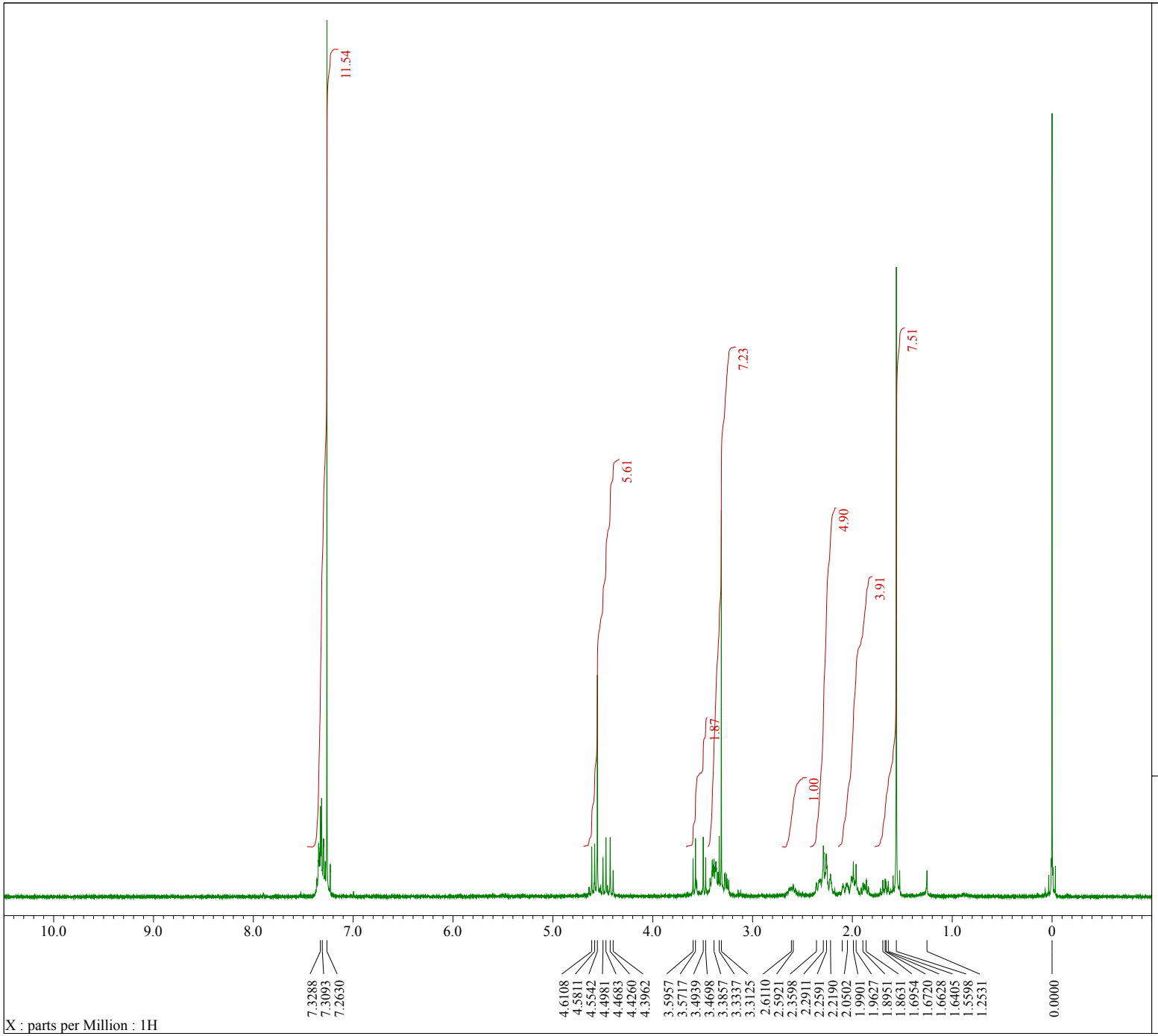
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 6000
Total_Scans       = 6000

Relaxation_Delay = 1.5[s]
Recvr_Gain        = 50
Temp_Get          = 22.9[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 4[dB]
X_Pulse           = 2.9[us]
Irr_Atn_Dec       = 22.569[dB]
Irr_Atn_Noe       = 22.569[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe Time          = 1.5[s]
```



39
^{13}C-NMR
(100 MHz, $CDCl_3$)

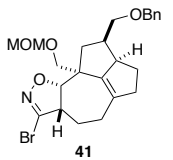



```
Filename          = yk06136-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = 1H-single_pulse
Solvent           = CHLOROFORM-D
Creation_Time     = 17-FEB-2017 22:16:16
Revision_Time     = 22-FEB-2018 11:07:52
Current_Time      = 22-FEB-2018 11:08:38

Comment           = 1H-single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 4.41450496[s]
X_Domain          = 1H
X_Freq            = 395.88430144[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22652597[Hz]
X_Sweep           = 7.42280285[kHz]
Irr_Domain        = 1H
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 395.88430144[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

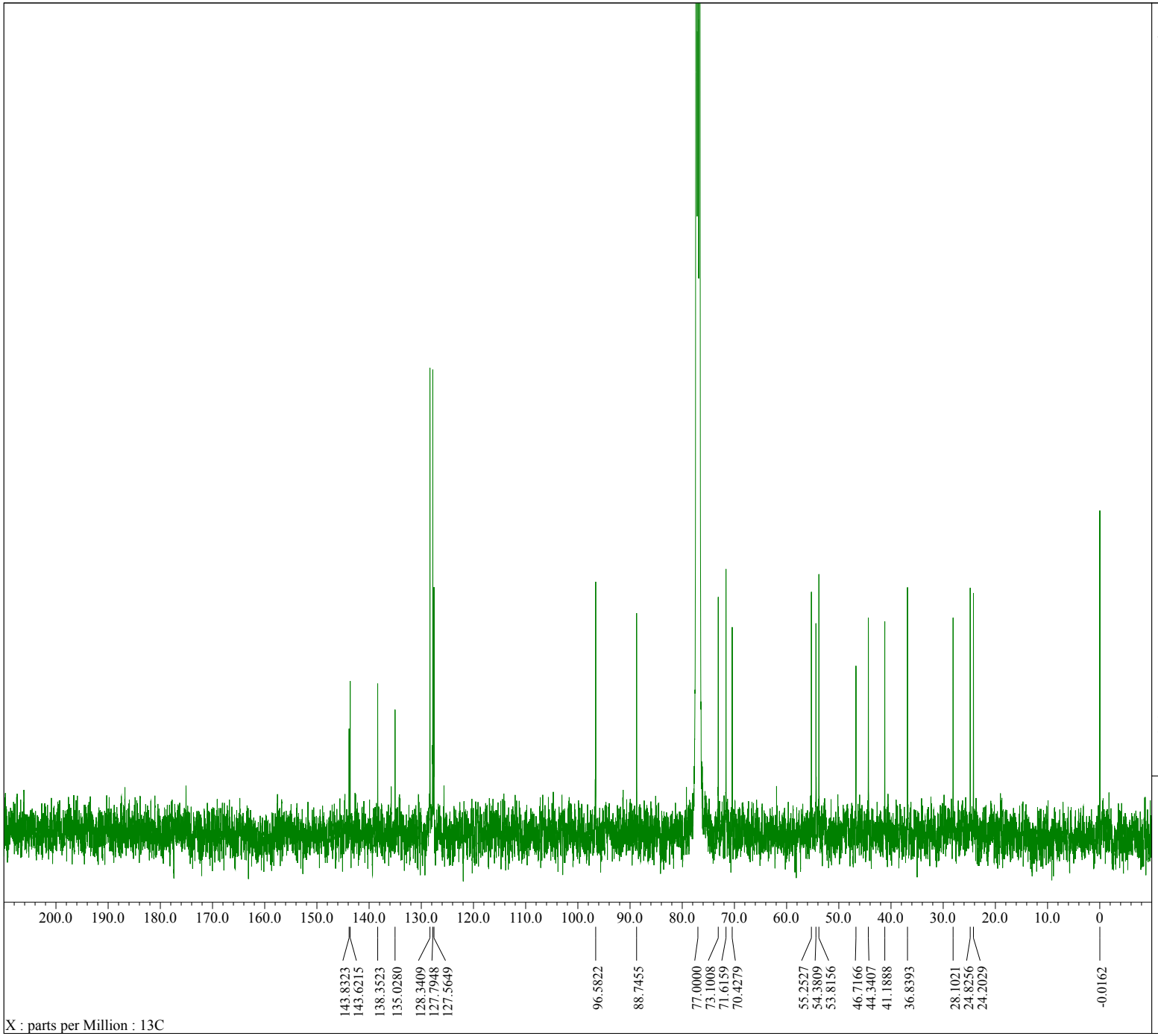
Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 19.5[dC]
X_90_Width        = 10.09[us]
X_Acq_Time        = 4.41450496[s]
X_Angle           = 45[deg]
X_Atn             = 1.1[dB]
X_Pulse           = 5.045[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 9.41450496[s]
```



41

^{1}H-NMR

(400 MHz, $CDCl_3$)

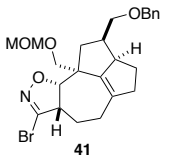



```
Filename          = yk0613613CC-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = 13C-single_pulse_dec
Solvent           = CHLOROFORM-D
Creation_Time     = 18-FEB-2017 07:58:33
Revision_Time     = 22-FEB-2018 10:55:39
Current_Time      = 22-FEB-2018 10:56:38

Comment           = single pulse decoupled
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

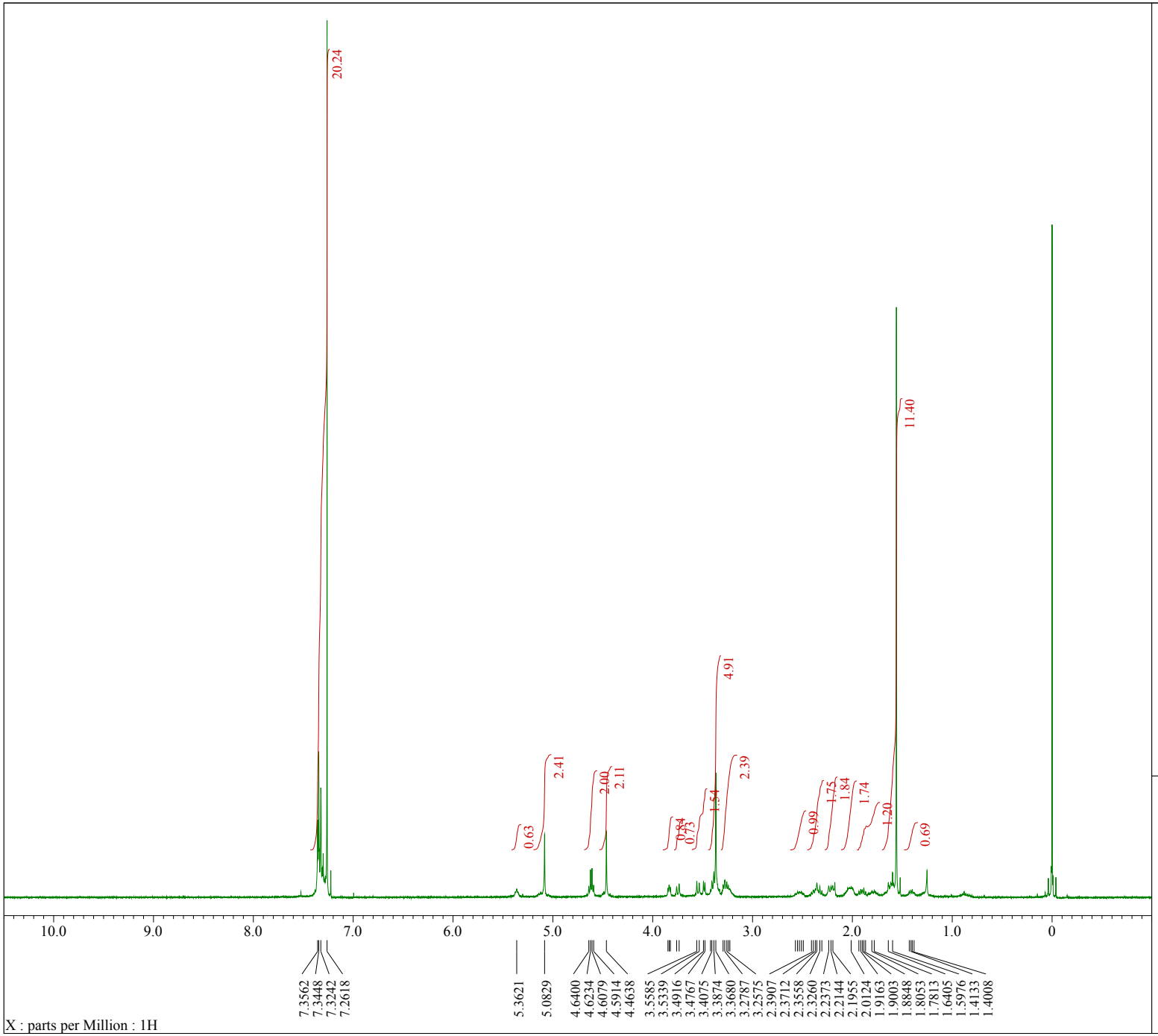
Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 1.048576[s]
X_Domain          = 13C
X_Freq            = 99.54517646[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95367432[Hz]
X_Sweep           = 31.25[kHz]
Irr_Domain        = 1H
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 11409
Total_Scans       = 11409

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.8[dC]
X_90_Width        = 8.82[us]
X_Acq_Time        = 1.048576[s]
X_Angle           = 30[deg]
X_Atn             = 4.2[dB]
X_Pulse           = 2.94[us]
Irr_Atn_Dec       = 22.23613[dB]
Irr_Atn_Noe       = 22.23613[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition Time   = 3.048576[s]
```



41

^{13}C-NMR
(100 MHz, $CDCl_3$)

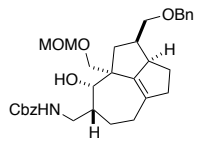



```
Filename          = yk06148-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = 1H-single_pulse
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2017 22:16:55
Revision_Time     = 22-FEB-2018 11:15:05
Current_Time      = 22-FEB-2018 11:15:41

Comment           = 1H-single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 4.41450496[s]
X_Domain          = 1H
X_Freq            = 395.88430144[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.22652597[Hz]
X_Sweep           = 7.42280285[kHz]
Irr_Domain        = 1H
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 395.88430144[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 32
Total_Scans       = 32

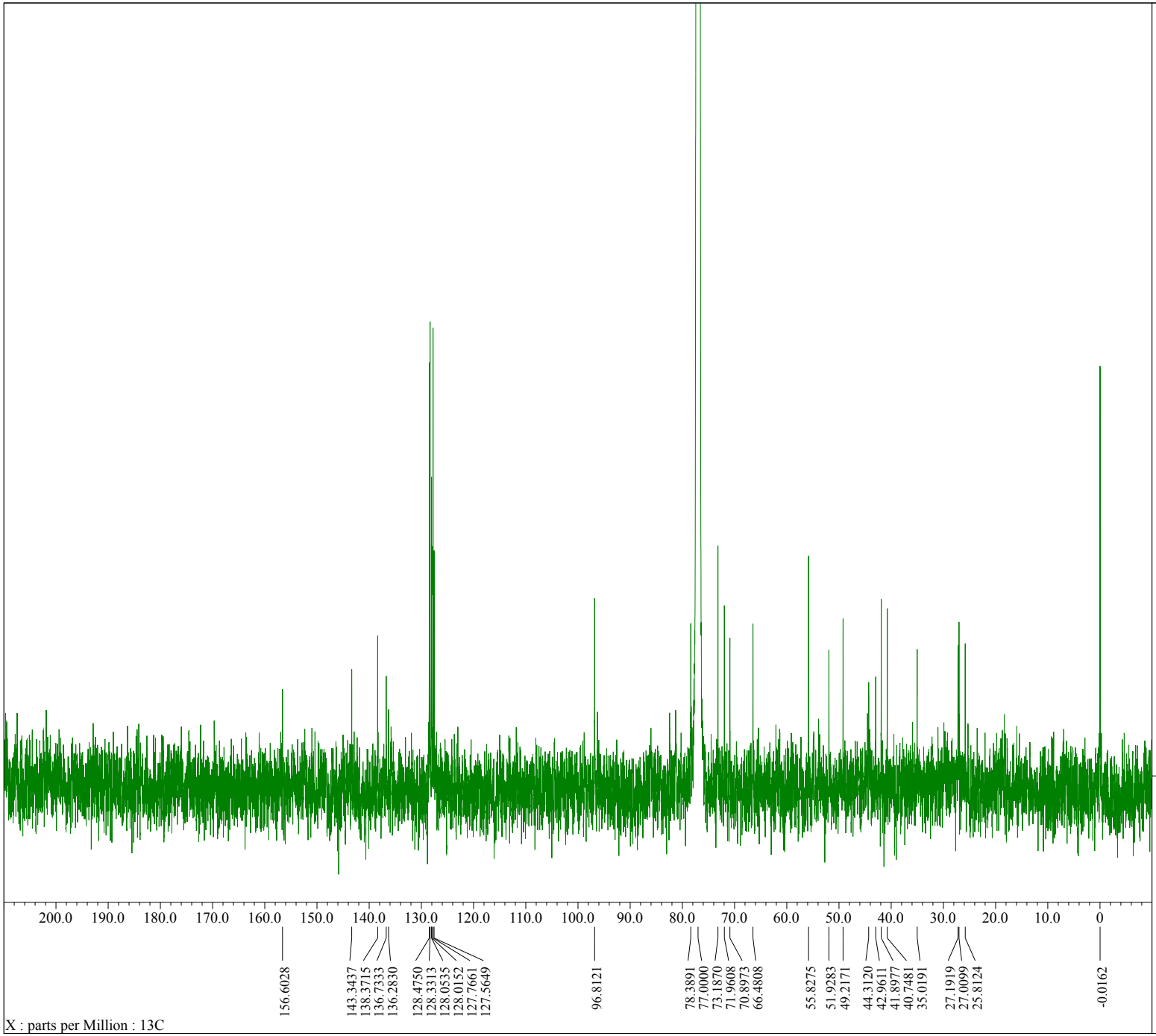
Relaxation_Delay  = 5[s]
Recvr_Gain        = 56
Temp_Get          = 19.6[dC]
X_90_Width        = 10.09[us]
X_Acq_Time        = 4.41450496[s]
X_Angle           = 45[deg]
X_Atn             = 1.1[dB]
X_Pulse           = 5.045[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition Time   = 9.41450496[s]
```



42

^{1}H-NMR

(400 MHz, $CDCl_3$)

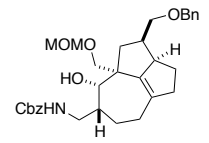



```
Filename          = yk06148c13-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = 13C-single_pulse_dec
Solvent           = CHLOROFORM-D
Creation_Time     = 23-FEB-2017 08:16:46
Revision_Time     = 22-FEB-2018 11:01:17
Current_Time      = 22-FEB-2018 11:02:08

Comment           = single pulse decoupled
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 1.048576[s]
X_Domain          = 13C
X_Freq            = 99.54517646[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95367432[Hz]
X_Sweep           = 31.25[kHz]
Irr_Domain        = 1H
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 11753
Total_Scans       = 11753

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.8[dC]
X_90_Width        = 8.82[us]
X_Acq_Time        = 1.048576[s]
X_Angle           = 30[deg]
X_Atn             = 4.2[dB]
X_Pulse           = 2.94[us]
Irr_Atn_Dec       = 22.23613[dB]
Irr_Atn_Noe       = 22.23613[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition Time   = 3.048576[s]
```



42
^{13}C-NMR
(100 MHz, $CDCl_3$)