Supporting Information (Experimental Section)

Synthetic Study on Pactamycin: Stereoselective Synthesis of the Cyclopentane Core Framework

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General Remarks

Nuclear magnetic resonance [^{1}H NMR (500 MHz), ^{13}C NMR (125 MHz)] or [^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz)] spectra were determined on JEOL ECA-500SL or JEOL AF-400 instrument. Chemical shifts for ^{1}H NMR were reported in parts per million downfields from tetramethylsilane (δ) as the internal standard and coupling constants were 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 centerline of a triplet at 77.0 ppm for deuteriochloroform.

High-resolution mass spectra (HRMS) were obtained on a BRUKER DALTONICS micrOTOF (ESI).

Infrared (IR) spectra were recorded on a SHIMADZU IRPrestige-21.

Optical rotations were measured on a JASCO P-1030 Polarimeter at RT using the sodium D line.

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 made on 7 x 20 cm plates prepared with a 0.25 mm layer of Merck silica gel 60 F_{254}. Compounds were eluted from the adsorbent with 10% methanol in chloroform. Column chromatography separations were performed on Fuji Sylsia silica gel PSQ 60B or silica gel PSQ 100B.

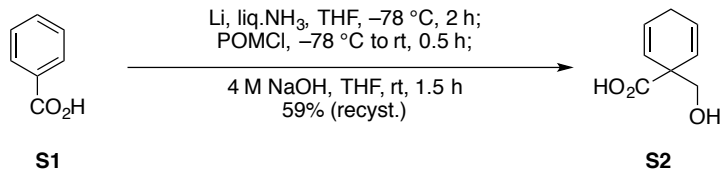
Reagents and solvents were commercial grades and were used as supplied with the following exceptions.

1) Dichloromethane, tetrahydrofuran and toluene: dried over molecular sieves 4A.

2) Methanol and acetonitrile: dried over molecular sieves 3A.

All reactions sensitive to oxygen and/or moisture were conducted under an argon atmosphere.

Hydroxycarboxylic acid **S2**



S1 **S2**

To a stirred solution of benzoic acid (**S1**, 20.7 g, 170 mmol) in a 2:1 mixture of liquid NH_3 and THF (total 300 mL) was added Li (4.3 g, 510 mmol) at –78 °C. After 2 hours, pivaloyloxymethyl chloride (POMCl, 50 mL, 540 mmol) was added at the same temperature. The resulting mixture was stirred at –78 °C for 0.5 hours. Then the reaction mixture was stirred at room temperature to remove excess NH_3. The residue was dissolved with THF (60 mL) and 4 M NaOH (200 mL). The resulting mixture was stirred at room temperature for 1.5 hours and extracted with CH_2Cl_2. The aqueous layer was acidified with conc. HCl at 0 °C and extracted with a 2:1 mixture of EtOAc/THF. The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by recrystallization ($CHCl_3$/*n*-hexane = 2/1) to afford **S2** (15.4 g, 59 %) as a colorless solid.

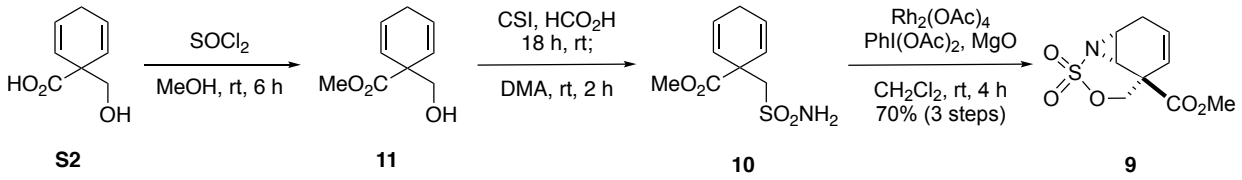
IR (film, cm^{-1}): 3330, 2884, 1695, 1290, 1243, 1225, 1084, 1024.

^1H NMR (500 MHz, $CDCl_3$, δ): 6.04 (dt, J = 10.2, 3.4 Hz, 2H), 5.81 (dt, J = 10.2, 2.0 Hz, 2H), 3.73 (s, 2H), 2.80-2.68 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 178.8, 128.2, 123.6, 68.5, 50.4, 26.3.

HRMS (ESI): Calcd for $C_8H_{10}O_3Na$ 177.0522 [$(M+Na)^+$], found 177.0526.

Aziridine **9**



S2 **11** **10** **9**

To a stirred solution of the **S2** (15.4 g, 100 mmol) in MeOH (500 mL) was added $SOCl_2$ (23 mL, 300 mmol) at 0 °C. The resulting mixture was stirred at room temperature for 6 hours. Then the reaction mixture was concentrated under reduced pressure. The crude material including **11** was applied to the following reaction without further purification.

To a stirred formic acid (11 mL, 250 mmol) was added CSI (22 mL, 250 mmol) at 0 °C. After 18 hours at room temperature, DMA (33 mL) and a solution of the crude material including **11** in DMA (66 mL) were added at the 0 °C. The resulting mixture was stirred at room temperature for 2 hours. Then the reaction mixture was quenched with H_2O at 0 °C and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude material including **10** was applied to the following reaction without further purification.

To a stirred solution of the crude material including **10** in CH_2Cl_2 (700 mL) were added MgO (7.5 g, 190 mmol), $PhI(OAc)_2$ (29 g, 89 mmol) and $Rh_2(OAc)_4$ (360 mg, 0.81 mmol) at 0 °C. The resulting mixture was stirred at room temperature for 4 hours. Then the reaction mixture was filtered through a pad of Celite and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 20/1 to 2/1) to afford **9** (17.1 g, 70%, 3 steps) as a yellow solid.

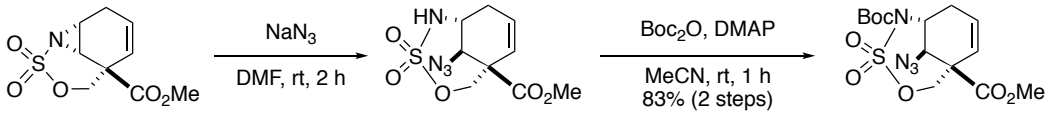
IR (film, cm^{-1}): 3038, 2959, 1740, 1375, 1260, 1184, 1018.

^1H NMR (500 MHz, $CDCl_3$, δ): 6.07 (ddd, J = 9.9, 5.1, 2.3 Hz, 1H), 5.80-5.78 (m, 1 H), 4.95 (d, J = 11.3 Hz, 1H), 4.26 (d, J = 11.3 Hz, 1H), 3.92 (d, J = 5.7 Hz, 1H), 3.86 (s, 3H), 3.13 (t, J = 6.8 Hz, 1H), 2.90 (dt, J = 20.4, 2.8 Hz, 1H), 2.69-2.62 (m, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 170.1, 128.1, 122.6, 75.0, 53.3, 43.0, 39.0, 38.4, 18.9.

HRMS (ESI) Calcd for $C_9H_{11}NO_5SNa$ 268.0250 [$(M+Na)^+$], found 268.0254.

Azide **13**



To a stirred solution of **9** (16.9 g, 68.9 mmol) in DMF (200 mL) was added NaN_3 (13.4 g, 207 mmol) at 0 °C. The resulting mixture was stirred at room temperature for 2 hours. Then the reaction mixture was quenched with 2 M HCl at 0 °C and extracted with Et_2O. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude material including **12a** was applied to the following reaction without further purification.

To a stirred solution of the crude material including **12a** in MeCN (230 mL) were added Boc_2O (15.0 g, 68.9 mmol) and DMAP (8.40 g, 68.9 mmol) at room temperature. The resulting mixture was stirred at the same temperature for 1 hour. Then the reaction mixture was concentrated under reduced pressure. After concentration, saturated aqueous NH_4Cl was added at 0 °C and extracted with Et_2O. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 10/1 to 4/1) to afford **13** (22.0 g, 83%, 2 steps) as a colorless amorphous.

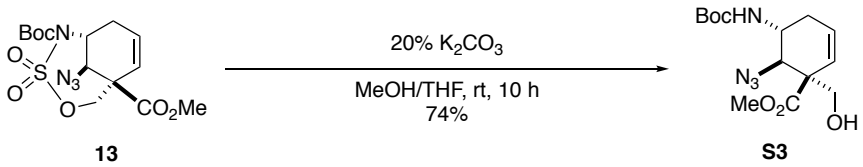
IR (film, cm^{-1}): 2984, 2110, 1740, 1728, 1387, 1300, 1186, 1070.

^1H NMR (400 MHz, $CDCl_3$, δ): 6.12-6.09 (m, 1H), 6.06-6.02 (m, 1H), 4.95 (dt, J = 10.0, 1.6 Hz, 1H), 4.85 (d, J = 12.0 Hz, 1H), 4.29 (t, J = 1.6 Hz, 1H), 4.16 (d, J = 12.0 Hz, 1H), 3.86 (s, 3H), 2.80 (dddd, J = 20.5, 10.2, 3.9, 1.4 Hz, J = 1H), 2.56 (brd, J = 20.5 Hz, 1H), 1.47 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 169.1, 150.8, 129.8, 120.1, 86.3, 73.6, 64.4, 53.1, 52.4, 50.2, 27.8, 26.8.

HRMS (ESI): Calcd for $C_{14}H_{20}N_4O_7SNa$ 411.0945 [$(M+Na)^+$], found 411.0943.

Alcohol **S3**



13 **S3**

To a stirred solution of **13** (393 mg, 1.16 mmol) in a 1:1 mixture of THF and MeOH (total 8 mL) was added 20% aqueous K_2CO_3 (4 mL) at 0 °C. The resulting mixture was stirred at room temperature for 10 hours. Then the reaction mixture was acidified with 1 M HCl at 0 °C and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 4/1 to 1/2) to afford **S3** (281 mg, 74%) as a colorless amorphous.

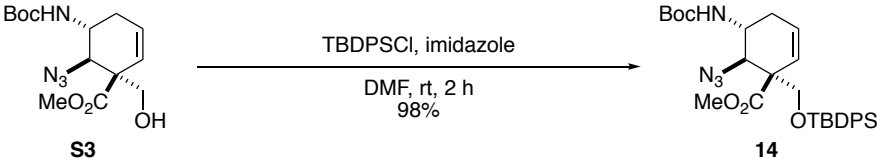
IR (film, cm^{-1}): 3370, 2980, 2108, 1721, 1701, 1525, 1437, 1368, 1306, 1244, 1171, 1020.

^1H NMR (500 MHz, $CDCl_3$, δ): 5.89 (ddd, J = 9.9, 5.1, 2.3 Hz, 1H), 5.56-5.53 (m, 1H), 4.65 (brs, 1H), 4.33-4.28 (m, 1H), 3.97 (dd, J = 11.3, 5.1 Hz, 1H), 3.76 (s, 3H), 3.73 (dd, J = 11.3, 9.1 Hz, 2H), 2.57 (dt, J = 18.1, 5.1 Hz, 1H), 2.33 (brs, 2H), 1.47 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 171.9, 155.2, 129.2, 124.6, 79.7, 65.6, 63.2, 55.2, 52.3, 48.2, 31.4, 28.2.

HRMS (ESI): Calcd for $C_{14}H_{22}N_4O_5Na$ 349.1482 [$(M+Na)^+$], found 349.1486.

TBDPS ether **14**

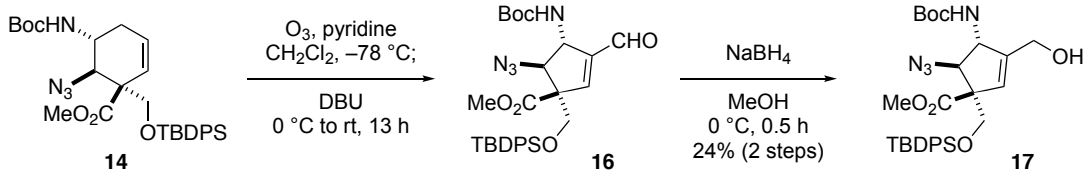


S3 **14**

To a stirred solution of **S3** (4.18 g, 12.8 mmol) in DMF (13 mL) were added imidazole (2.7 g, 39 mmol) and *t*-butyldiphenylchlorosilane (TBDPSCl, 5.0 mL, 19 mmol) at 0 °C. The resulting mixture was stirred at room temperature for 2 hours. Then the reaction mixture was quenched with saturated aqueous NH_4Cl at 0 °C and extracted with Et_2O. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 20/1 to 1/1) to afford **14** (7.00 g, 98%) as a colorless amorphous.

IR (film, cm^{-1}): 3374, 3265, 2955, 2887, 2104, 1732, 1706, 1503, 1366, 1244, 1167, 1113.
^1H NMR (500 MHz, $CDCl_3$, δ): 7.65-7.26 (m, 4H), 7.45-7.37 (m, 6H), 5.85 (ddd, J = 9.6, 5.1, 2.3 Hz, 1H), 5.40 (brd, J = 9.6 Hz, 1H), 4.59 (brs, 1H), 4.48-4.41 (m, 1H), 4.05 (brs, 1H), 4.01 (d, J = 10.2 Hz, 1H), 3.92 (d, J = 10.2 Hz, 1H), 3.66 (s, 3H), 2.62 (dt, J = 17.6, 6.2 Hz, 1H), 2.16 (brs, 1H), 1.46 (s, 9H), 1.04 (s, 9H).
^{13}C NMR (125 MHz, $CDCl_3$, δ): 171.0, 155.1, 135.6, 135.5, 133.0, 132.9, 129.8, 128.1, 127.7, 126.7, 79.8, 65.0, 62.5, 55.7, 52.1, 48.2, 31.5, 28.3, 26.8, 19.3.
HRMS (ESI): Calcd for $C_{30}H_{40}N_4O_5SiNa$ 587.2660 [$(M+Na)^+$], found 587. 2661.

Allyl alcohol **17**



To a stirred solution of **14** (6.00 g, 11 mmol) and pyridine (2.6 mL) in CH_2Cl_2 (53 mL) was stirred under O_3 bubbling at –78 °C for 1 hour. Then the reaction mixture was bubbled argon gas to purge of unreacted ozone and added 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU, 3.2 mL) at 0 °C. The resulting mixture was stirred at room temperature for 13 hours. Then the reaction mixture was quenched with saturated aqueous NH_4Cl at 0 °C and extracted with Et_2O. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude material including **16** was applied to the following reaction without further purification.

To a stirred solution of the crude material including **16** in MeOH (23 mL) was added $NaBH_4$ (145 mg, 3.70 mmol) at 0 °C. The resulting mixture was stirred at the same temperature for 0.5 hours. Then the reaction mixture was quenched with saturated aqueous NH_4Cl at the same temperature and extracted with EtOAc. The organic layer was washed with H_2O and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 5/1 to 1/1) to afford **17** (1.50 g, 24%, 2 steps) as a colorless amorphous.

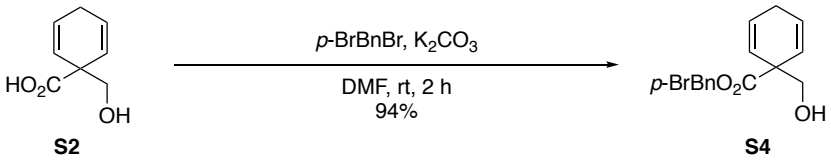
IR (film, cm^{-1}): 3408, 3368, 2932, 2859, 2108, 1719, 1508, 1368, 1252, 1165, 1115.

^1H NMR (500 MHz, $CDCl_3$, δ): 7.63-7.61 (m, 4H), 7.47-7.38 (m, 6H), 5.57 (brs, 1H), 4.87 (brs, 1H), 4.70 (brs, 1H), 4.20-4.11 (m, 2H), 4.02 (d, *J* = 10.2 Hz, 1H), 3.96-3.95 (m, 1H), 3.88 (d, *J* = 10.2 Hz, 1H), 3.69 (s, 3H), 2.50 (brs, 1H), 1.46 (s, 9H), 1.06 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 170.9, 155.9, 145.6, 135.6, 132.9, 132.7, 130.0, 129.9, 127.8, 127.5, 80.6, 69.9, 65.3, 63.1, 61.1, 59.2, 52.2, 28.3, 26.9, 19.3.

HRMS (ESI): Calcd for $C_{30}H_{40}N_4O_6SiNa$ 603.2609 [$(M+Na)^+$], found 603.2610.

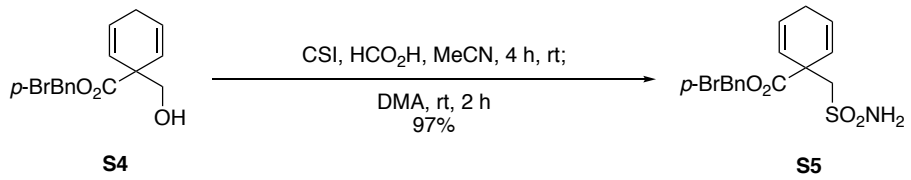
p-Bromobenzyl ester **S4**



To a stirred solution of **S2** (2.50 g, 16.2 mmol) in DMF (54 mL) were added K_2CO_3 (2.46 g, 17.8 mmol) and *p*-bromobenzyl bromide (*p*-BrBnBr, 4.96 mL, 19.5 mmol) at 0 °C. The resulting mixture was stirred at room temperature for 2 hours. Then the reaction mixture was quenched with saturated aqueous NH_4Cl at 0 °C and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 5/1 to 2/1) to afford **S4** (4.98 g, 95%) as a colorless oil.

IR (film, cm^{-1}): 3458, 3034, 2947, 2878, 1724, 1489, 1452, 1408, 1371, 1271, 1229, 1200, 1070.
^1H NMR (400 MHz, $CDCl_3$, δ): 7.49 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 8.3 Hz, 2H), 6.02 (dt, *J* = 10.2, 3.4 Hz, 2H), 5.80 (dt, *J* = 10.2, 2.0 Hz, 2H), 5.11 (s, 2H), 3.71 (brs, 2H), 2.72 (m, 2H).
^{13}C NMR (125 MHz, $CDCl_3$, δ): 173.2, 134.8, 131.7, 129.6, 129.4, 128.4, 127.8, 124.0, 122.1, 68.6, 65.9, 50.7, 26.3.
HRMS (ESI): Calcd for $C_{15}H_{15}O_3BrNa$ 354.0102 [$(M+Na)^+$], found 345.0097.

Sulfamate **S5**



S4 **S5**

To a stirred formic acid (0.70 mL, 18 mmol) was added CSI (1.6 mL, 18 mmol) and MeCN (2.3 mL) at 0 °C. After 4 hours at room temperature, DMA (3 mL) and a solution of **S4** in DMA (6 mL) was added at room temperature. The resulting mixture was stirred at the same temperature for 2 hours. Then the reaction mixture was quenched with H_2O at 0 °C and extracted with EtOAc. The organic layer was washed with H_2O and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 5/1 to 2/1) to afford **S5** (3.54 g, 97%) as a colorless oil.

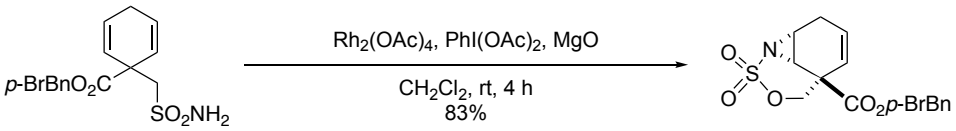
IR (film, cm^{-1}): 3370, 3291, 1724, 1375, 1221, 1186.

^1H NMR (500 MHz, $CDCl_3$, δ): 7.50 (d, *J* = 8.5 Hz, 2H), 7.21 (d, *J* = 8.5 Hz, 2H), 6.0 (dt, *J* = 10.2, 3.4 Hz, 2H), 5.77 (dt, *J* = 10.2, 2.3 Hz, 2H), 5.12 (s, 2H), 4.64 (brs, 2H), 4.26 (s, 2H), 2.72 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 171.7, 134.5, 131.7, 129.6, 128.7, 122.4, 122.3, 74.6, 66.4, 48.2, 26.2.

HRMS (ESI): Calcd for $C_{15}H_{16}O_5NSBrNa$ 423.9830 $[(M+Na)^+]$, found 423.9825.

Aziridine **S6**

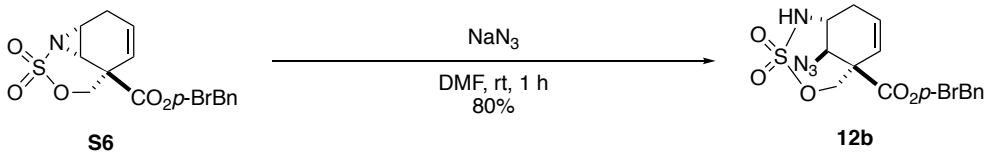


S5 **S6**

To a stirred solution of **S5** (3.00 g, 7.46 mmol) in CH_2Cl_2 (750 mL) were added MgO (680 mg, 16.9 mmol), $PhI(OAc)_2$ (2.71 g, 8.41 mmol) and $Rh_2(OAc)_4$ (211 mg, 0.477 mmol) at 0 °C. The resulting mixture was stirred at room temperature for 4 hours. Then the reaction mixture was filtered through a pad of Celite and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 10/1 to 2/1) to afford **S6** (2.49 g, 83%) as a colorless oil.

IR (film, cm^{-1}): 3043, 2963, 1740, 1489, 1375, 1253, 1188, 1070, 1015.
^1H NMR (500 MHz, $CDCl_3$, δ): 7.53 (d, *J* = 8.5 Hz, 2H), 7.23 (d, *J* = 8.5 Hz, 2H), 6.06 (ddd, *J* = 9.6, 5.1, 2.3 Hz, 1H), 5.75 (brd *J* = 8.5 Hz, 1 H), 5.22 (d, *J* = 12.5 Hz, 1H), 5.12 (d, *J* = 12.5 Hz, 1H), 4.93 (d, *J* = 11.3 Hz, 1H), 4.25 (d, *J* = 11.3 Hz, 1H), 3.88 (brd, *J* = 5.7 Hz, 1H), 3.12 (t, *J* = 6.8 Hz, 1H), 2.88 (brd, *J* = 20.4 Hz, 1H), 2.69-2.62 (m, 1H).
^{13}C NMR (125 MHz, $CDCl_3$, δ): 169.6, 133.4, 132.1, 130.2, 128.6, 123.2, 122.5, 75.0, 67.5, 42.9, 39.1, 38.4, 19.1.
HRMS (ESI): Calcd for $C_{15}H_{14}O_5NSBrNa$ 421.9666 $[(M+Na)^+]$, found 421.9668.

Azide **12b**



S6 **12b**

To a stirred solution of **S6** (1.77 g, 4.42 mmol) in DMF (20 mL) was added NaN_3 (780 mg, 12.0 mmol) at 0 °C. The resulting mixture was stirred at room temperature for 1 hours. Then the reaction mixture was quenched with saturated aqueous NH_4Cl at 0 °C and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 10/1 to 2/1) to afford **12b** (1.42 g, 80%) as a colorless oil. The colorless amorphous **12b** was recrystallized from EtOAc for X-ray crystallographic analysis.

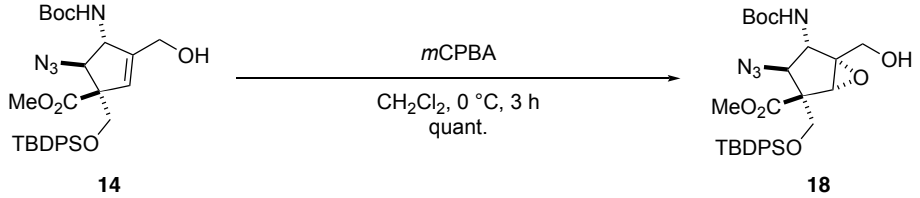
IR (film, cm^{-1}): 3292, 2959, 2930, 2122, 1735, 1458, 1425, 1373, 1253, 1179.

^1H NMR (400 MHz, $CDCl_3$, δ): 7.52 (d, *J* = 8.5 Hz, 2H), 7. 26 (d, *J* = 8.5 Hz, 2H), 5.93 (dd, *J* = 10.2, 5.1 Hz, 1H), 5.93 (brd, *J* = 10.2 Hz, 1H), 5.20 (s, 2H), 4.97 (d, *J* = 4.0, 1.6 Hz, 1H), 4.59 (d, *J* = 12.0 Hz, 1H), 4.32 (brs, 1H), 4.06 (d, *J* = 12.0 Hz, 1H), 3.93 (brs, 1H), 2.71 (brd, *J* = 16.4 Hz, 1H), 1.96 (dd, *J* = 18.7, 5.1 Hz, 1H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 168.5, 133.6, 132.0, 130.4, 126.3, 123.2, 72.2, 67.2, 61.1, 53.8, 49.0, 27.6.

HRMS (ESI): Calcd for $C_{15}H_{15}N_4O_5SNa$ 464.9840 [$(M+Na)^+$], found 464.9839.

Epoxyalcohol **18**

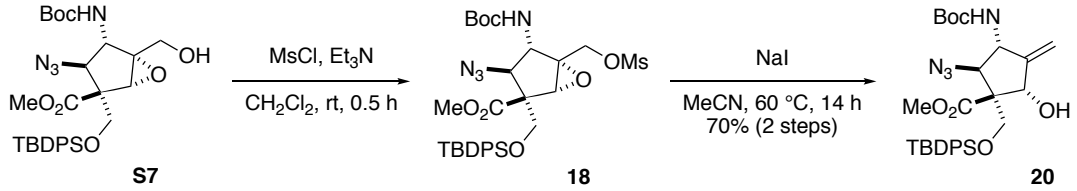


14 **18**

To a stirred solution of **14** (794 mg, 1.37 mmol) in CH_2Cl_2 (4.0 mL) was added *m*CPBA (546 mg, 2.06 mmol) at room temperature. The resulting mixture was stirred at the same temperature for 3 hours. Then the reaction mixture was quenched with saturated aqueous $Na_2S_2O_3$ and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 7/1 to 2/1) to afford **18** (823 mg, quant.) as a colorless amorphous.

IR (film, cm^{-1}): 3358, 3072, 3049, 2954, 2927, 2856, 2108, 1724, 1699, 1521, 1429, 1367, 1244, 1165, 1112.
^1H NMR (500 MHz, $CDCl_3$, δ): 7.73-7.67 (m, 4H), 7.46-7.38 (m, 6H), 4.78 (brd, *J* = 8.5 Hz, 1H), 4.44 (brt, *J* = 8.5 Hz, 1H), 4.24 (d, *J* = 9.6 Hz, 1H), 3.99 (d, *J* = 13.0, 1H), 3.84 (s, 1H), 3.79 (s, 3H), 3.76 (d, *J* = 9.6 Hz, 1H), 3.11 (d, *J* = 7.9, 1H), 1.46 (s, 9H), 1.04 (s, 9H).
^{13}C NMR (125 MHz, $CDCl_3$, δ): δ 169.1, 168.7, 156.1, 135.5, 135.4, 134.4, 133.2, 132.7, 132.5, 131.4, 130.0, 129.7, 129.6, 128.1, 127.69, 127.67, 80.9, 66.4, 65.4, 63.3, 60.9, 59.0, 57.5, 56.3, 52.4, 28.1, 26.5, 19.1.
HRMS (ESI): Calcd for $C_{30}H_{40}N_4O_7SiNa$ 619.2558 [$(M+Na)^+$], found 619.2559.

Allyl alcohol **20**



To a stirred solution of **S7** (745 mg, 1.25 mmol) in CH_2Cl_2 (4.0 mL) were added Et_3N (0.69 mL, 5.0 mmol) and MsCl (0.19 mL, 2.5 mmol) at 0 ºC. The resulting mixture was stirred at room temperature for 0.5 hours. Then the reaction mixture was quenched with saturated aqueous NH_4Cl and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The crude material including **18** was applied to the following reaction without further purification.

To a stirred solution of the crude material including **18** in MeCN (5.0 mL) was added NaI (3.7 g, 25 mmol) at room temperature. The resulting mixture was stirred at 60 ºC for 14 hours Then the reaction mixture was quenched with saturated aqueous $Na_2S_2O_3$ and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 10/1 to 4/1) to afford **20** (508 mg, 70%, 2 steps) as a yellow amorphous.

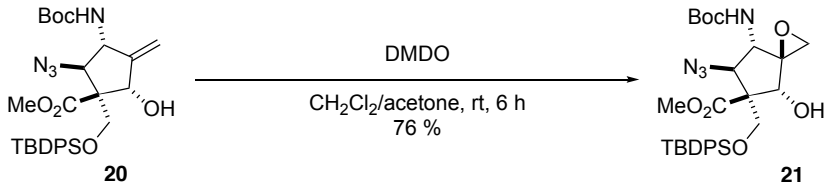
IR (film, cm^{-1}): 3508, 3369, 3072, 3051, 2954, 2931, 2891, 2858, 2106, 1724, 1506, 1429, 1367, 1251, 1165, 1112.

^1H NMR (500 MHz, $CDCl_3$, δ): 7.66-7.64 (m, 4H), 7.48-7.39 (m, 6H), 5.46 (brs, 1H), 5.20 (brs, 1H), 4.61 (brs, 1H), 4.56 (t, *J* = 7.9 Hz, 1H), 4.16 (d, *J* = 10.2 Hz, 1H), 3.95 (d, *J* = 10.2 Hz, 1H), 3.86 (d, *J* = 7.9 Hz, 1H), 3.71 (s, 3H), 3.43 (d, *J* = 7.9 Hz, 1H), 1.47 (s, 9H), 1.04 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 171.5, 155.0, 149.6, 135.5, 132.1, 132.0, 130.0, 127.9 127.8, 109.6, 80.4, 74.6, 67.1, 63.3, 58.7, 56.7, 52.1, 28.2, 26.7, 19.1.

HRMS (ESI): Calcd for $C_{30}H_{40}N_4O_6SiNa$ 603.2609 [$(M+Na)^+$], found 603.2607.

Epoxide **21**



To a stirred solution of **20** (146 mg, 0.251 mmol) in CH_2Cl_2 (0.84 mL) was added dimethyldioxirane (DMDO, 8.4 mL ca. 0.1 M in acetone) at room temperature. The resulting mixture was stirred at the same temperature for 6 hours. Then the reaction mixture was concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 9/1 to 5/2) to afford **21** (114 mg, 76%) as a colorless amorphous.

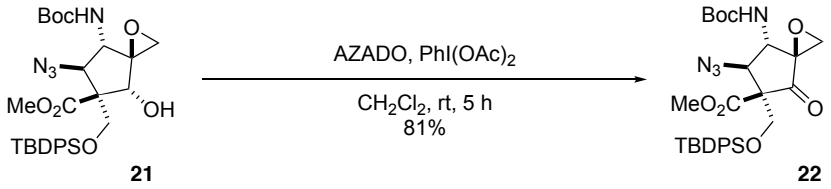
IR (film, cm^{-1}):3374, 2952, 2107, 1724, 1522, 1428, 1368, 1251, 1164, 1114.

^1H NMR (500 MHz, $CDCl_3$, δ): 7.72-7.70 (m, 4H), 7.46-7.40 (m, 6H), 4.99 (d, *J* = 6.2 Hz, 1H), 4.55 (d, *J* = 9.1 Hz, 1H), 4.25 (m, 2H), 4.22-4.11 (m, 2H), 3.95 (t, *J* = 7.9 Hz, 1H), 3.73 (s, 3H), 3.08 (d, *J* = 4.5 Hz, 1H), 2.77 (d, *J* = 4.5 Hz, 1H), 1.44 (s, 9H), 1.06 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 171.0, 155.2, 135.6, 132.4, 130.0, 127.8, 81.1, 76.5, 66.3, 66.3, 64.5, 63.2, 59.5, 58.1, 52.3, 48.2, 28.2, 26.7, 19.2.

HRMS (ESI): Calculated for $C_{30}H_{40}N_4O_7SiNa$ 619.2558 $[(M+Na)]^+$, found 619.2547.

Ketone **22**



To a stirred solution of **21** (81.0 mg, 0.136 mmol) in CH_2Cl_2 (0.5 mL) were added $PhI(OAc)_2$ (52.5 mg, 0.163 mmol) and AZADO (2.1 mg, 0.014 mmol) at room temperature. The resulting mixture was stirred at the same temperature for 5 hours. Then, the reaction mixture was poured into saturated aqueous $Na_2S_2O_3$ and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 9/1 to 10/3) to afford **22** (65.5 mg, 81%.) as a colorless amorphous.

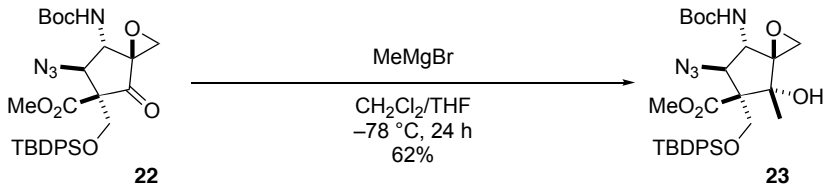
IR (film, cm^{-1}): 3383, 2952, 2872, 2107, 1771, 1702, 1429, 1367, 1252, 1215, 1163, 1114, 1072.

^1H NMR (500 MHz, $CDCl_3$, δ): 7.64-7.60 (m, 4H), 7.45-7.37 (m, 6H), 5.09 (d, J = 9.1 Hz, 1H), 4.76 (brs, 1H), 4.65 (brd, J = 6.8 Hz, 1H), 4.34 (d, J = 10.8 Hz, 1H), 3.96 (d, J = 10.8 Hz, 1H), 3.71 (s, 3H), 3.06 (d, J = 6.8 Hz, 2H), 1.48 (s, 9H), 1.02 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 203.5, 165.8, 135.6, 135.5, 130.14, 130.08, 128.0, 127.9, 81.1, 65.1, 61.1, 61.3, 60.3, 53.6, 53.0, 52.2, 28.2, 26.7, 19.2.

HRMS (ESI): Calculated for $C_{30}H_{38}O_7N_4SiNa$ 617.2402 $[(M+Na)]^+$, found 617.2402.

Alcohol **23**



To a stirred solution of **22** (65.2 mg, 0.101 mmol) in CH_2Cl_2 (0.5 mL) was added MeMgBr (0.895 mL, 0.98 M in THF, 0.808 mmol) at –78 ºC. The resulting mixture was stirred at 24 hours. Then the reaction mixture was poured into saturated aqueous NH_4Cl and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography (SiO_2, *n*-hexane/EtOAc = 20/3 to 3/2) to afford **23** (38.2 mg, 78%, 2 steps) as a colorless amorphous.

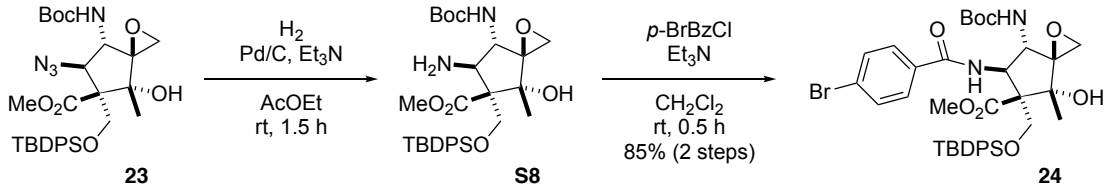
IR (film, cm^{-1}): 3375, 2933, 2858, 2104, 1731, 1684, 1508, 1369, 1278, 1249, 1165, 1114.

^1H NMR (500 MHz, $CDCl_3$, δ): 7.79 (d, *J* = 7.4 Hz, 2H), 7.34 (d, *J* = 7.4 Hz, 2H), 7.45-7.40 (m, 6H), 5.40 (s, 1H), 5.30 (d, *J* = 7.9 Hz, 1H), 4.83 (d, *J* = 8.5 Hz, 1H), 4.20-4.11 (m, 3H), 3.66 (s, 3H), 2.85 (d, *J* = 4.5 Hz, 1H), 2.76 (d, *J* = 4.5 Hz, 1H), 1.46 (s, 9H), 1.07 (s, 9H), 0.94 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 170.6, 155.7, 135.9, 135.7, 132.6, 132.5, 129.8, 127.8, 127.7, 81.3, 80.7, 68.2, 65.1, 63.0, 61.9, 60.3, 51.9, 47.4, 28.2, 26.8, 19.1, 17.9.

HRMS (ESI): Calculated for $C_{31}H_{42}O_7SiNa$ 633.2715 $[(M+Na)]^+$, found 633.2720.

Amide **24**



To a stirred solution of **23** (17.7 mg, 0.029 mmol) in EtOAc (0.5 mL) were added Pd/C (12 mg) at room temperature. The resulting mixture was stirred at the same temperature for 1.5 hours. Then the reaction mixture was filtered through a pad of Celite and concentrated under reduced pressure. The crude material including **S8** was applied to the following reaction without further purification.

The crude material including **S8** in CH_2Cl_2 (0.5 mL) were added *p*-bromobenzoyl chloride (*p*-BrBzCl, 7.6 mg, 0.035 mmol) and Et_3N (10 μL, 0.073 mmol). The resulting mixture was stirred at 0.5 hours. Then the reaction mixture was poured into saturated aqueous NH_4Cl and extracted with EtOAc. The residue was purified by prep. TLC (SiO_2, *n*-hexane/EtOAc = 2/1) to afford **22** (18.9 mg, 85%, 2 steps) as a colorless solid. The colorless solid **24** was recrystallized from EtOAc for X-ray crystallographic analysis.

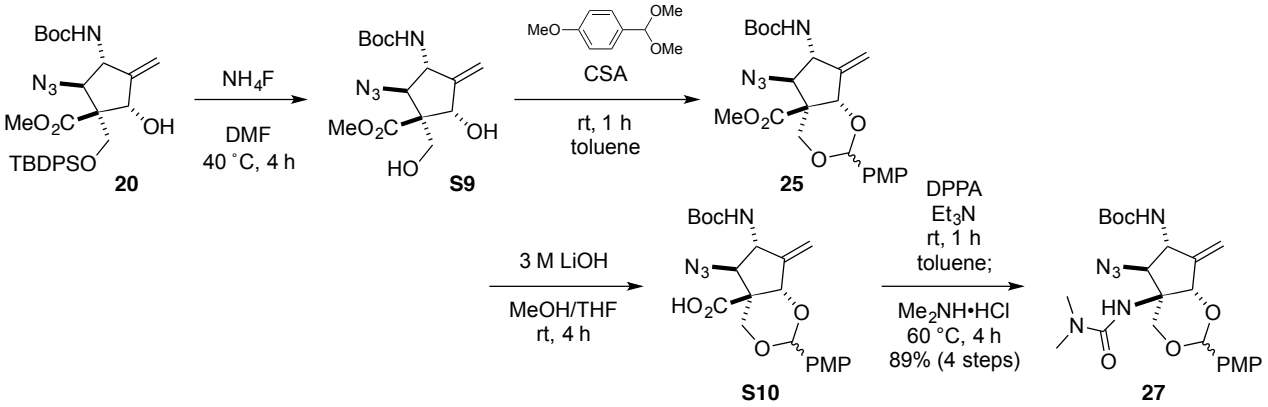
IR (film, cm^{-1}):3440, 2933, 2858, 1719, 1681, 1523, 1481, 1473, 1368, 1246, 1163, 1114.

^1H NMR (500 MHz, $CDCl_3$, δ): 7.61 (d, *J* = 6.8 Hz, 4H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.43-7.36 (m, 3H), 7.29 (d, *J* = 7.4 Hz, 2H) 7.12 (d, *J* = 9.1 Hz, 1H), 5.74 (s, 1H), 5.56 (t, *J* = 9.6 Hz, 1H), 5.22 (d, *J* = 7.9 Hz, 1H), 4.27 (d, *J* = 10.8 Hz, 1H), 4.14 (d, *J* = 10.8 Hz, 1H), 4.10 (t, *J* = 8.5 Hz, 1H),3.66 (s, 3H), 2.84 (s, 2H), 1.43 (s, 9H), 0.99 (s, 3H), 0.90 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): δ172.8, 166.1, 156.2, 135.7, 135.6, 133.3, 132.5, 132.2, 131.6, 129.8, 128.7, 17.7, 127.6, 126.0, 80.9, 80.7, 67.2, 65.1, 63.5, 60.6, 56.0, 52.1, 46.8, 28.2, 26.7, 18.9, 18.4.

HRMS (ESI): Calculated for $C_8H_{10}O_3Na$ 789.2177 $[(M+Na)]^+$, found 789.2174.

Urea **27**



To a stirred solution of **20** (497 mg, 0.856 mmol) in DMF (2.0 mL) were added NH_4F (158 mg, 4.28 mmol) at room temperature. The resulting mixture was stirred at 40 ºC for 4 hours. Then the reaction mixture was quenched with saturated aqueous NH_4Cl and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude material including **S9** was applied to the following reaction without further purification.

To a stirred solution of the crude material including **S9** in toluene (4.0 mL) were added *p*-anisaldehyde dimethylacetal (0.29 mL, 1.7 mmol) and 10-camphor sulfonic acid (CSA, 20 mg, 0.086 mmol) at room temperature. The resulting mixture was stirred at the same temperature for 1 hour. Then the reaction mixture was quenched with Et_3N (60 μL) and concentrated under reduced pressure. The crude material including **25** was applied to the following reaction without further purification.

To a stirred solution of the crude material including **25** in a 1:1 mixture of MeOH and THF (total 4.0 mL) were added 3 M LiOH (1.4 mL, 4.3 mmol) at 0 ºC. The resulting mixture was stirred at room temperature for 4 hours. Then the reaction mixture was quenched with saturated aqueous NH_4Cl and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude material including **S10** was applied to the following reaction without further purification.

To a stirred solution of the crude material including **S10** in toluene (2.8 mL) were added Et_3N (0.6 mL, 4.3 mmol) and diphenylphosphoryl azide (DPPA, 0.29 mL, 1.3 mmol) at room temperature. After 1 hours, the reaction mixture was added $Me_2NH{\cdot}HCl$ (210 mg, 2.6 mmol) at the same temperature. The resulting mixture was stirred at 60 °C for 4 hours. Then the reaction mixture was quenched with saturated aqueous NH_4Cl at 0 °C and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified with column chromatography (SiO_2, *n*-hexane/EtOAc = 2/1 to 1/1) to afford **27** (372 mg, 89%, 4 steps) as a white solid.

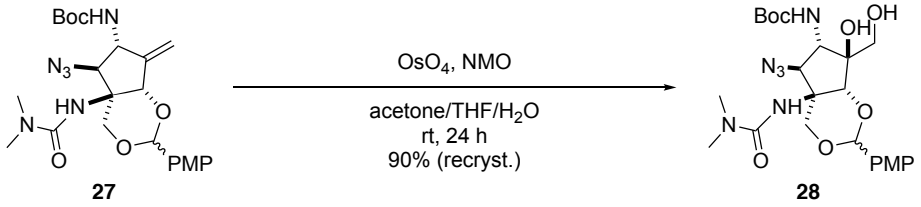
IR (film, cm^{-1}): 2976, 2883, 2380, 2351, 2310, 2110, 1699, 1645, 1633, 1614, 1587, 1516, 1487, 1454, 1435, 1390, 1363, 1247, 1168, 1126, 1089, 1010.

^1H NMR (500 MHz, $CDCl_3$, δ): 7.39 (d, J = 8.5 Hz, 2H), 6.89 (d, J = 8.5 Hz, 2H), 5.73 (s, 1H), 5.54 (m, 2H), 5.43 (brs, 1H), 4.91 (brd, J = 9.1 Hz, 1H), 4.83 (brt, J = 9.1 Hz, 1H), 4.69 (s, 1H), 4.64 (d, J = 11.3 Hz, 1H), 4.50 (d, J = 8.5 Hz, 1H), 3.88 (d, J = 11.3 Hz, 1H), 3.81 (s, 1H), 3.80 (s, 3H), 2.95 (brs, 1H), 2.88 (s, 6H), 1.49 (s, 9H),

^{13}C NMR (125 MHz, $CDCl_3$, δ): 160.1, 156.8, 155.7, 146.2, 130.1, 127.4, 127.3, 118.1, 113.7, 99.6, 80.4, 78.6, 68.3, 65.9, 57.4, 56.6, 55.3, 38.3, 36.2, 28.4.

HRMS (ESI): Calcd for $C_{23}H_{32}N_6O_6Na$ 511.2276 $[(M+Na)]^+$, found 511.2274.

Acetal **28**



To a stirred solution of **27** (1.16 g, 2.37 mmol) in a 3:3:1 mixture of acetone, THF and H_2O (total 42 mL) were added *N*-methylmorpholine *N*-oxide (NMO, 833 mg, 7.11 mmol) and OsO_4 (6.0 mL, 2.0% aqueous solution, 0.47 mmol) at room temperature. The resulting mixture was stirred at the same temperature for 24 hours Then the reaction mixture was quenched with saturated aqueous $Na_2S_2O_3$ and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by recrystallization (MeOH/*n*-hexane = 3/1) to afford **28** (1.11 g, 90 %) as a colorless solid. The colorless amorphous **28** was recrystallized from EtOAc for X-ray crystallographic analysis.

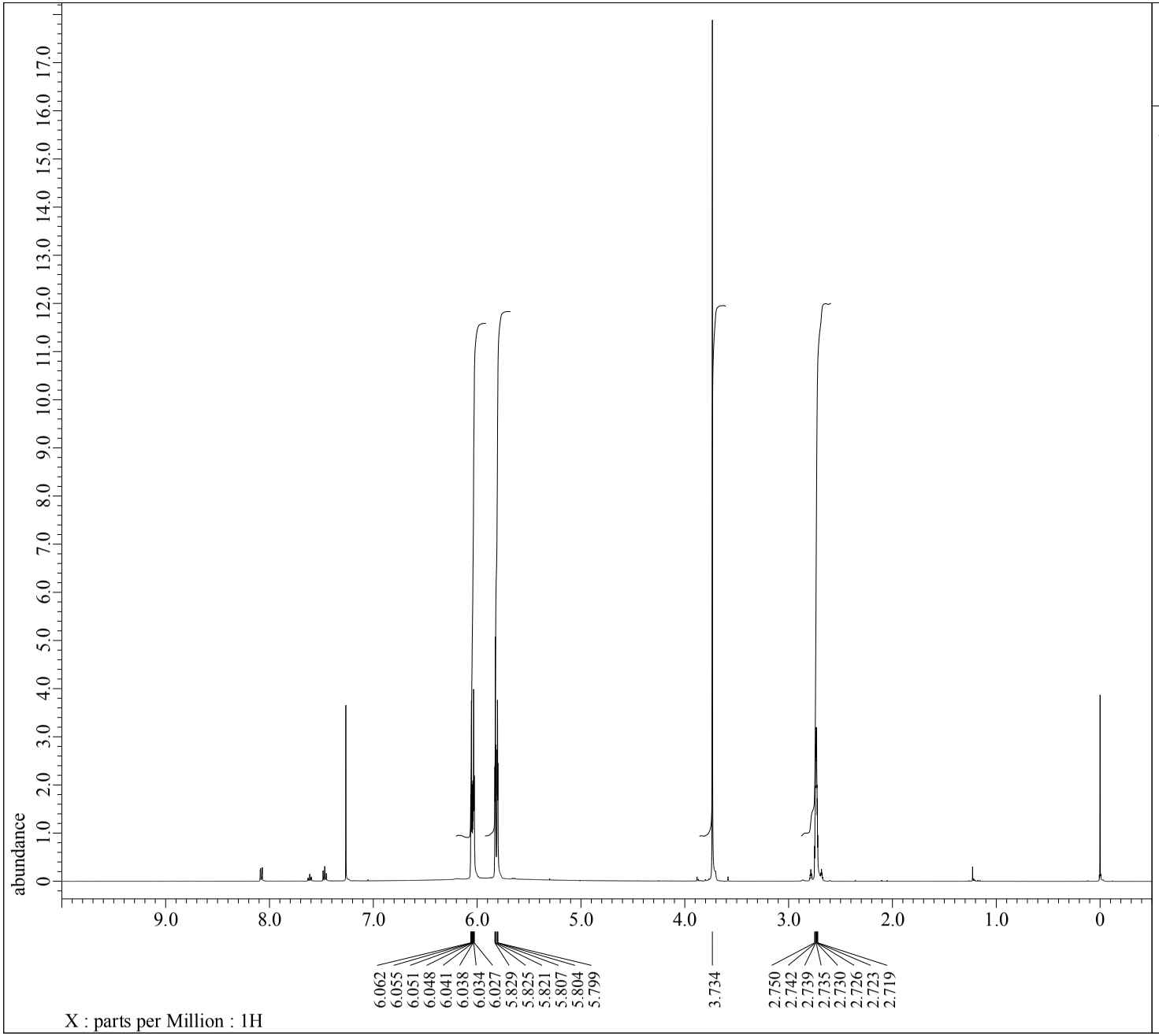
IR (film, cm^{-1}): 3375, 2976, 2935, 2839, 2108, 1691, 1643, 1616, 1517, 1502, 1467, 1367, 1251, 1168, 1031.

^1H NMR (500 MHz, $CDCl_3$, δ): 7.31 (d, *J* = 8.5 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 5.60 (s, 1H), 5.51 (brs, 1H), 5.17 (s, 1H), 5.04 (s, 1H), 4.48 (d, *J* = 11.3 Hz, 1H), 4.41 (d, *J* = 3.4 Hz, 2H), 4.20 (d, *J* = 11.3 Hz, 1H), 3.80 (s, 3H), 3.67 (t, *J* = 11.3 Hz, 1H), 3.01 (dd, *J* = 9.6 Hz, 3.4 Hz, 3H), 2.89 (s, 6H), 1.46 (s, 9H).

^{13}C NMR (125 MHz, $CDCl_3$, δ): 160.2, 157.2, 156.2, 129.6, 127.0, 113.8, 99.5, 84.4, 80.6, 80.0, 68.8, 67.3, 65.0, 63.6, 56.8, 55.3, 36.3, 28.3.

HRMS (ESI): Calcd for $C_{23}H_{34}N_6O_8Na$ 545.2330 [$(M+Na)^+$], found 545.2327.

NMR Spectra Data



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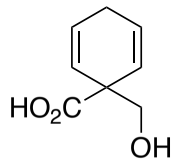
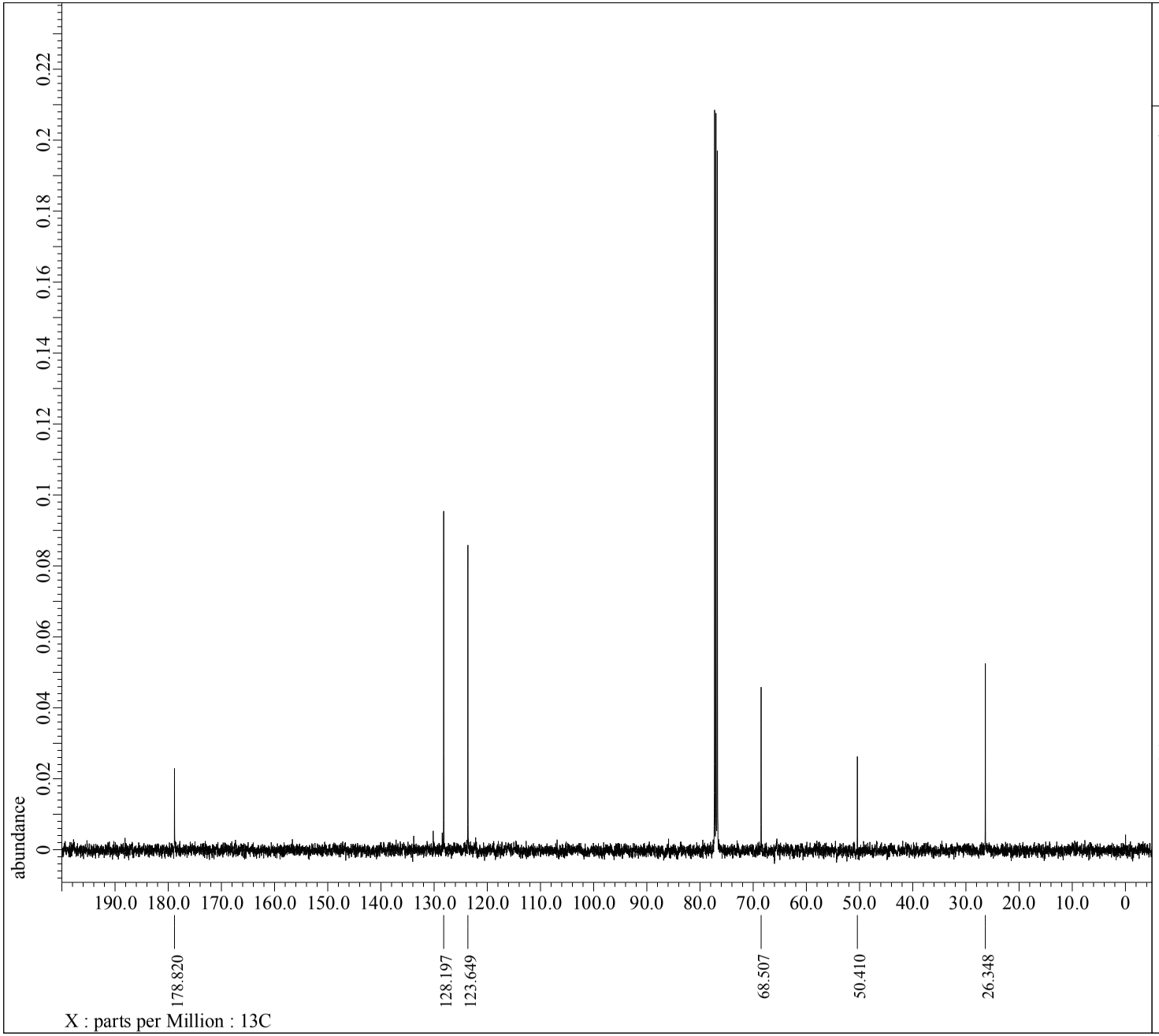


S2



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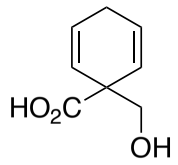
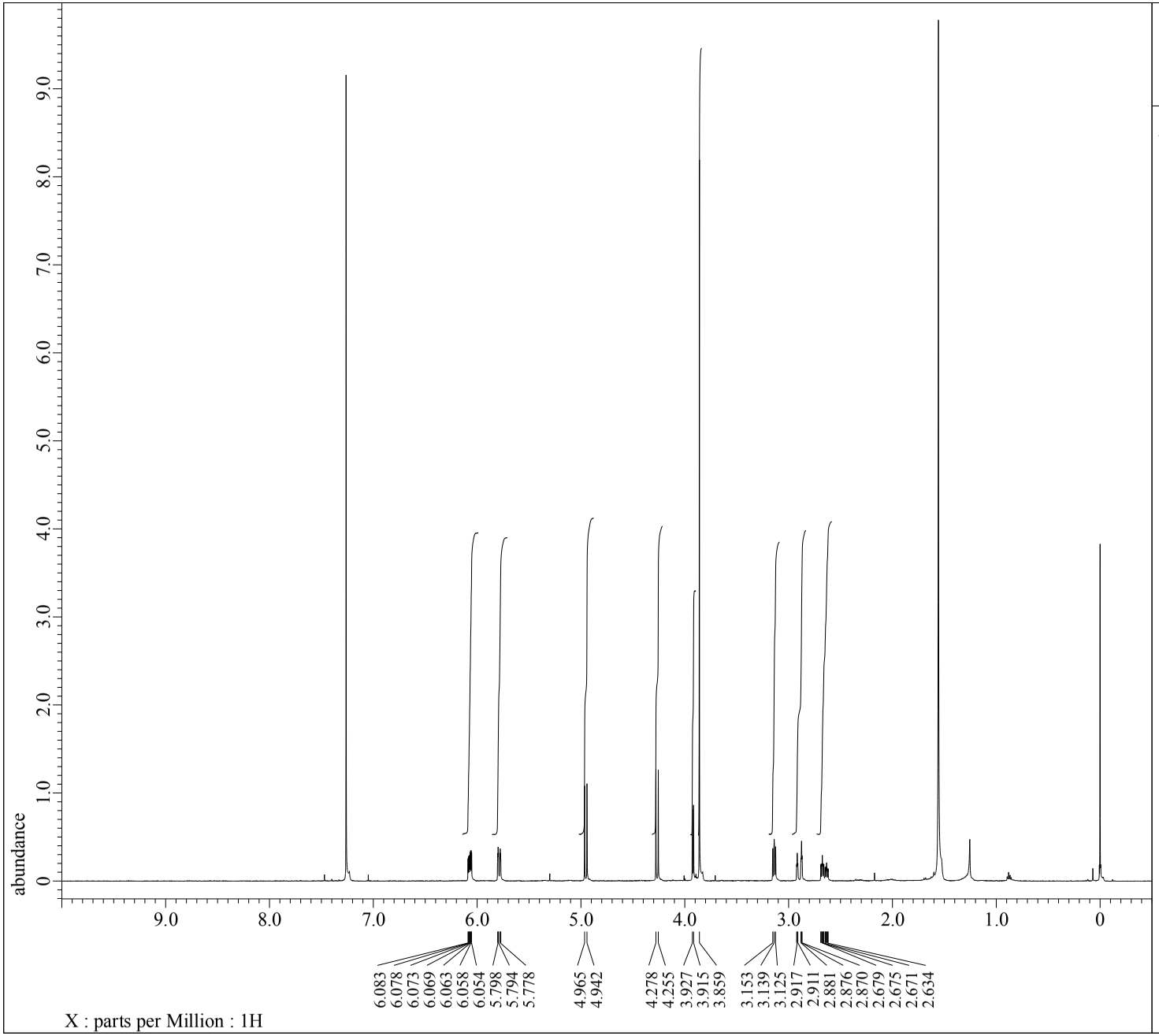


S2



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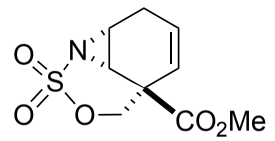
Filename	= SY-120829-1-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= SY-120829-1
Solvent	= CHLOROFORM-D
Creation_Time	= 29-AUG-2012 17:30:07
Revision_Time	= 20-FEB-2017 23:08:26
Current_Time	= 20-FEB-2017 23:09:45
Comment	= pure
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 1.76422912[s]
X_Domain	= 1H
X_Freq	= 495.13191398[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.5668198[Hz]
X_Sweep	= 9.28677563[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 495.13191398[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 54
Temp_Get	= 24.6[dC]
X_90_Width	= 8.7[us]
X_Acq_Time	= 1.76422912[s]
X_Angle	= 45[deg]
X_Atn	= 4[dB]
X_Pulse	= 4.35[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.76422912[s]



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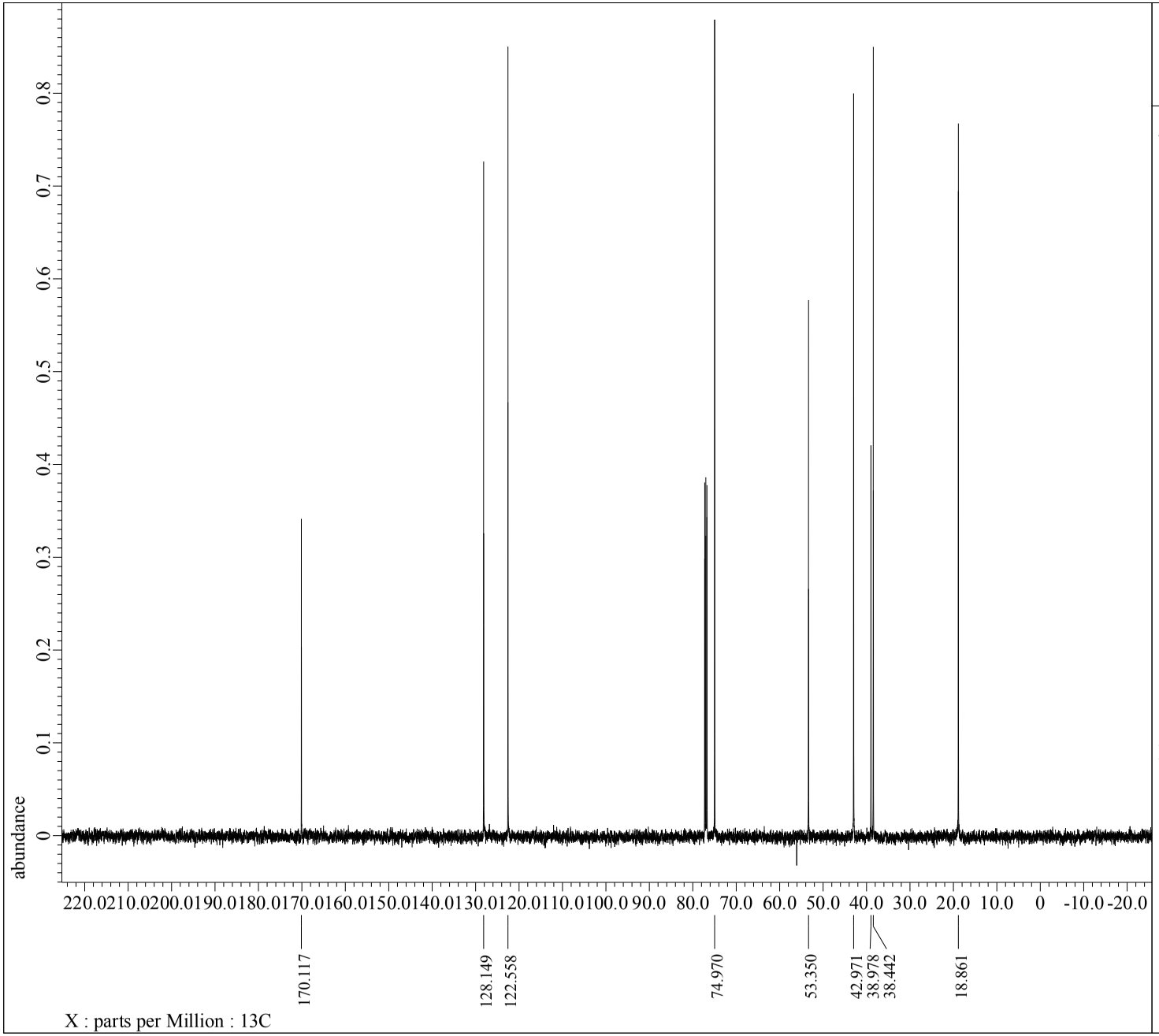


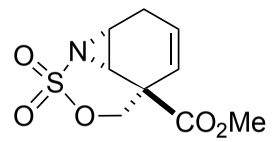
JEOL
Solutions for Innovation

```
Filename          = SY-131213-2-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = SY-131213-2
Solvent           = CHLOROFORM-D
Creation_Time     = 13-DEC-2013 14:40:06
Revision_Time     = 20-FEB-2017 23:12:23
Current_Time      = 20-FEB-2017 23:12:43

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T]  (500[MHz])
X_Acq_Duration    = 0.8388608[s]
X_Domain          = 13C
X_Freq            = 124.5010059[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.1920929[Hz]
X_Sweep           = 39.0625[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay  = 2[s]
Recvr_Gain        = 52
Temp_Get          = 460.0[dC]
X_90_Width        = 15.6[us]
X_Acq_Time        = 0.8388608[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 5.2[us]
Irr_Atn_Dec       = 25.434[dB]
Irr_Atn_Noe       = 25.434[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.8388608[s]
```



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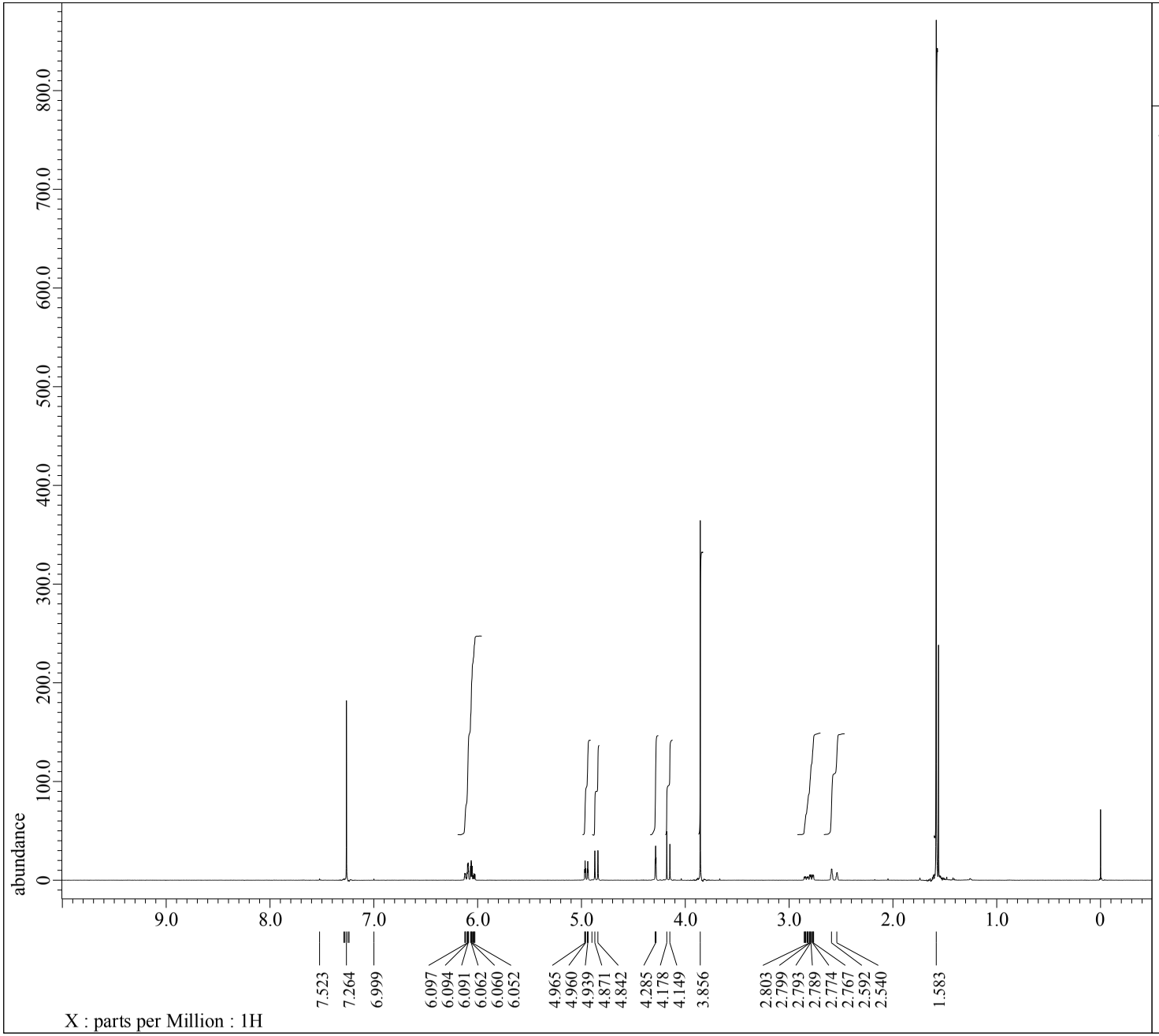


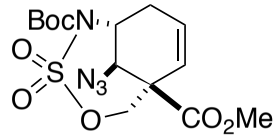
JEOL
Solutions for Innovation

```
Filename          = SY-121220-11NON_E8_FT-2.jd
Author            = MI
Experiment        = NON
Solvent           = CDCL3
Creation_Time     = 15-FEB-2017 00:17:36
Revision_Time     = 20-FEB-2017 23:29:33
Current_Time      = 20-FEB-2017 23:31:30

Comment           = pure
Data_Format       = 1D COMPLEX
Dim_Size          = 16384
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = ALICE_NMR

X_Domain          = 1H
X_Freq            = 399.7844939[MHz]
X_Offset          = 0[Hz]
X_Points          = 16384
X_Prescans        = 1
X_Sweep           = 7.99200781[kHz]
Scans             = 8

Relaxation_Delay = 4.94999981
Recvr_Gain        = 18
Temp_Get          = 21.29999924[dC]
```



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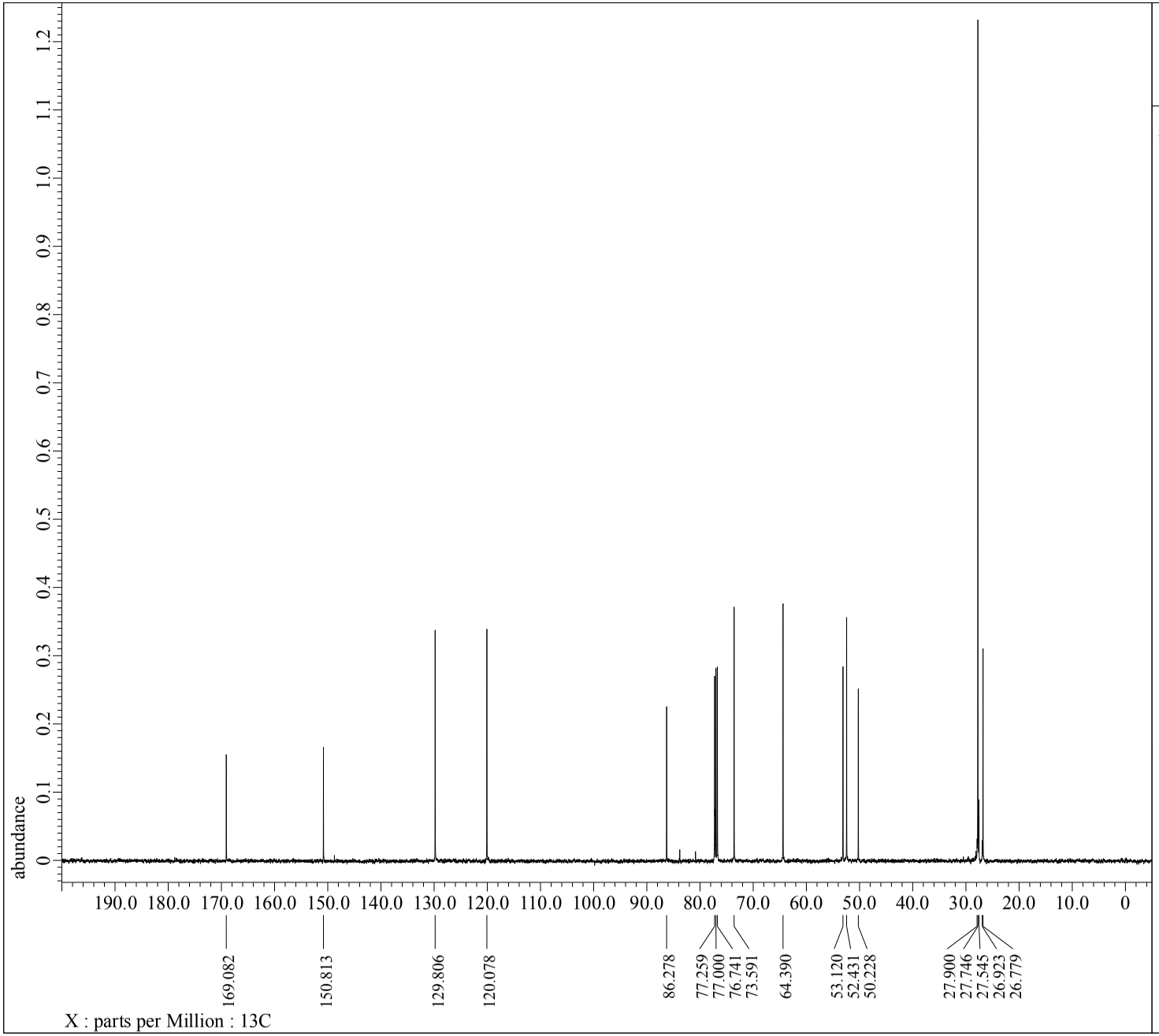


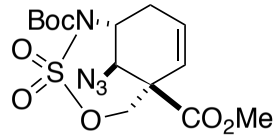
JEOL
Solutions for Innovation

```
Filename          = SY-131106-1-4.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = SY
Solvent           = CHLOROFORM-D
Creation_Time     =  6-NOV-2013 23:23:55
Revision_Time     = 20-FEB-2017 23:33:33
Current_Time      = 20-FEB-2017 23:34:08

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 0.8388608[s]
X_Domain          = 13C
X_Freq            = 124.5010059[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.1920929[Hz]
X_Sweep           = 39.0625[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 800
Total_Scans       = 800

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 25[dC]
X_90_Width        = 15.6[us]
X_Acq_Time        = 0.8388608[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 5.2[us]
Irr_Atn_Dec       = 25.434[dB]
Irr_Atn_Noe       = 25.434[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.8388608[s]
```



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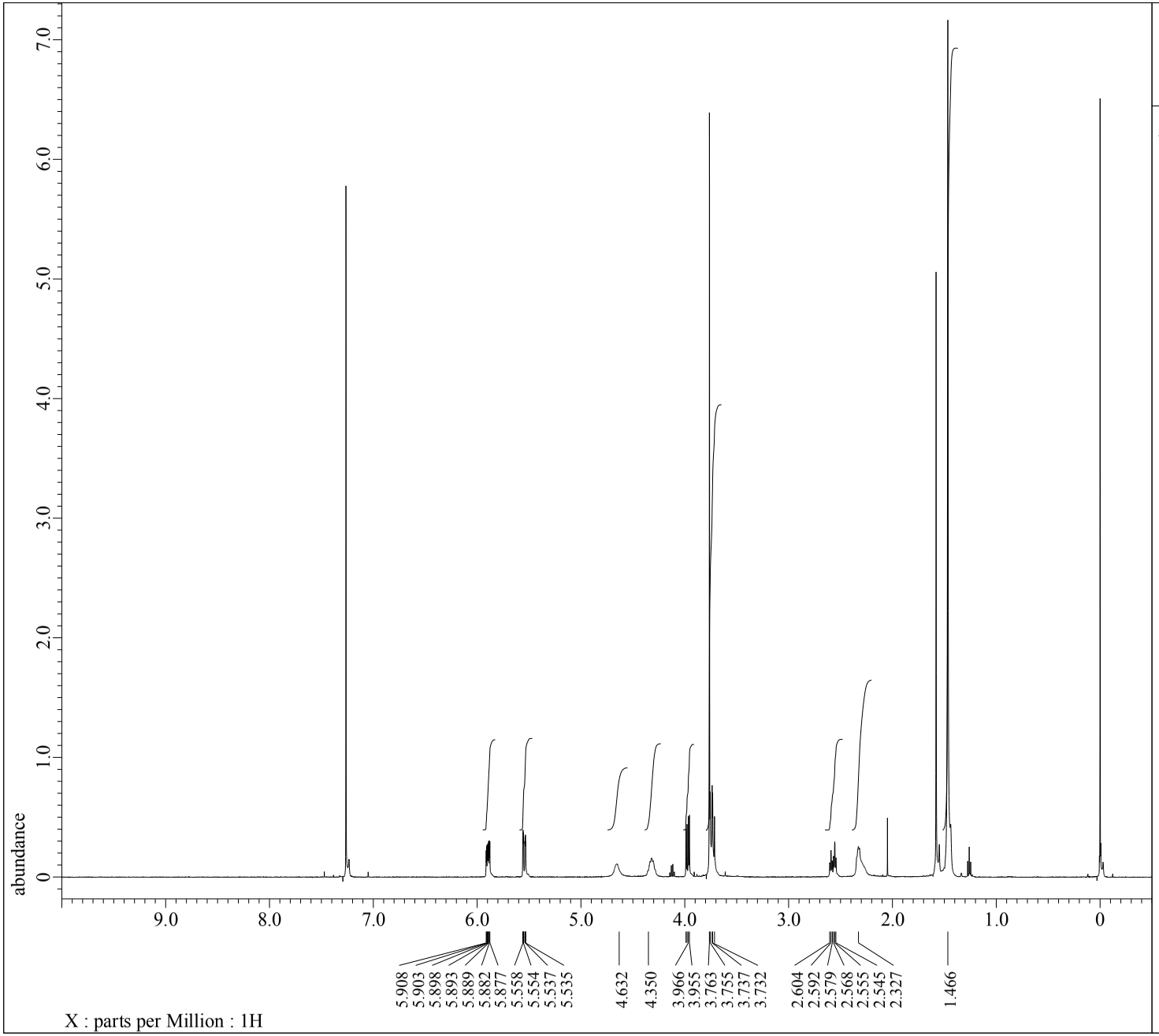
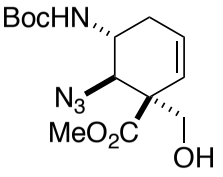
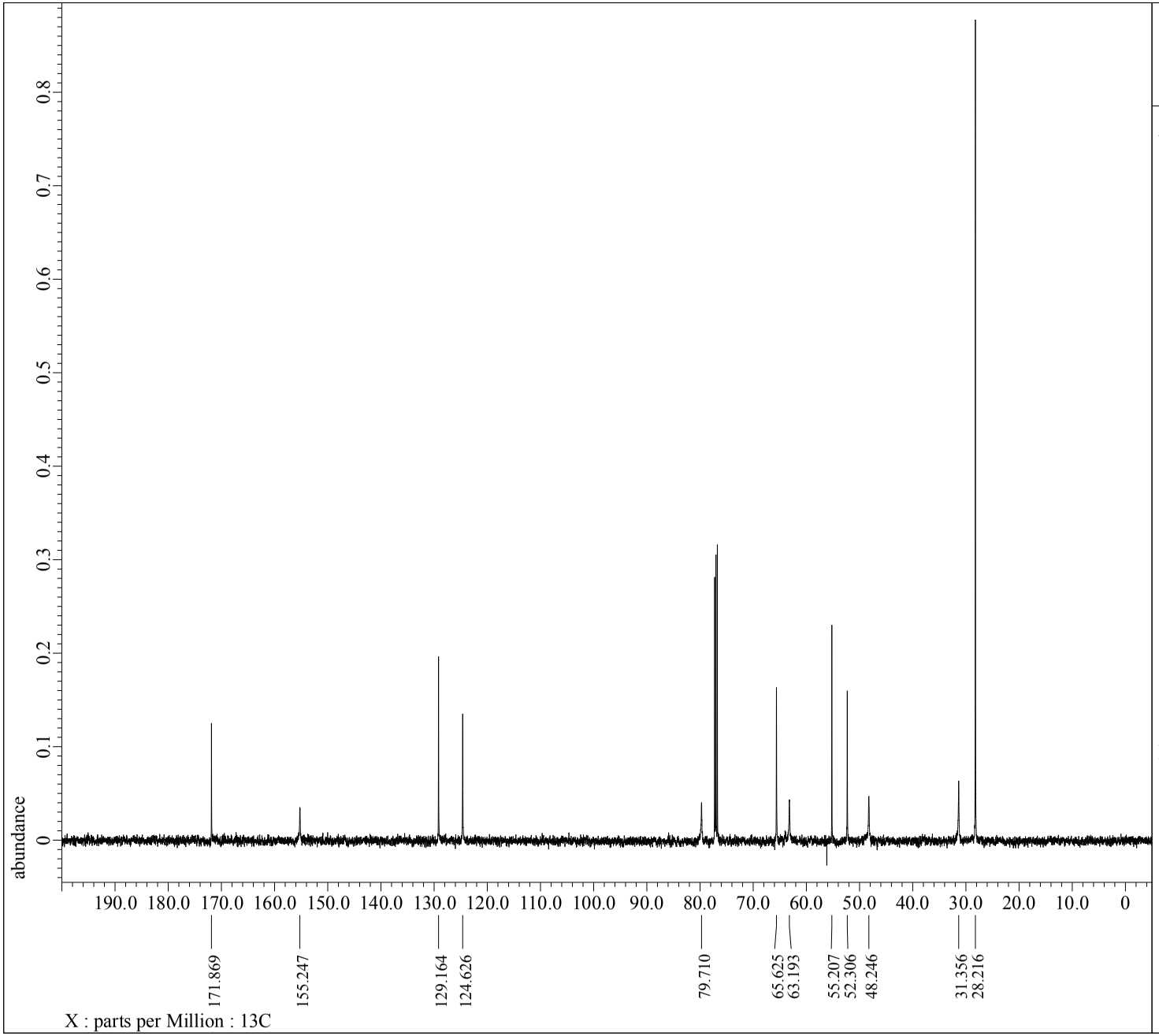


JEOL
Solutions for Innovation

```
Filename          = SY-121127-2-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = SY-121127-2
Solvent           = CHLOROFORM-D
Creation_Time     = 27-NOV-2012 16:06:22
Revision_Time     = 21-FEB-2017 11:15:32
Current_Time      = 21-FEB-2017 11:17:37

Comment           = pure
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 56
Temp_Get          = 20.5[dC]
X_90_Width        = 8.7[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.35[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```

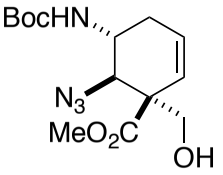


S3

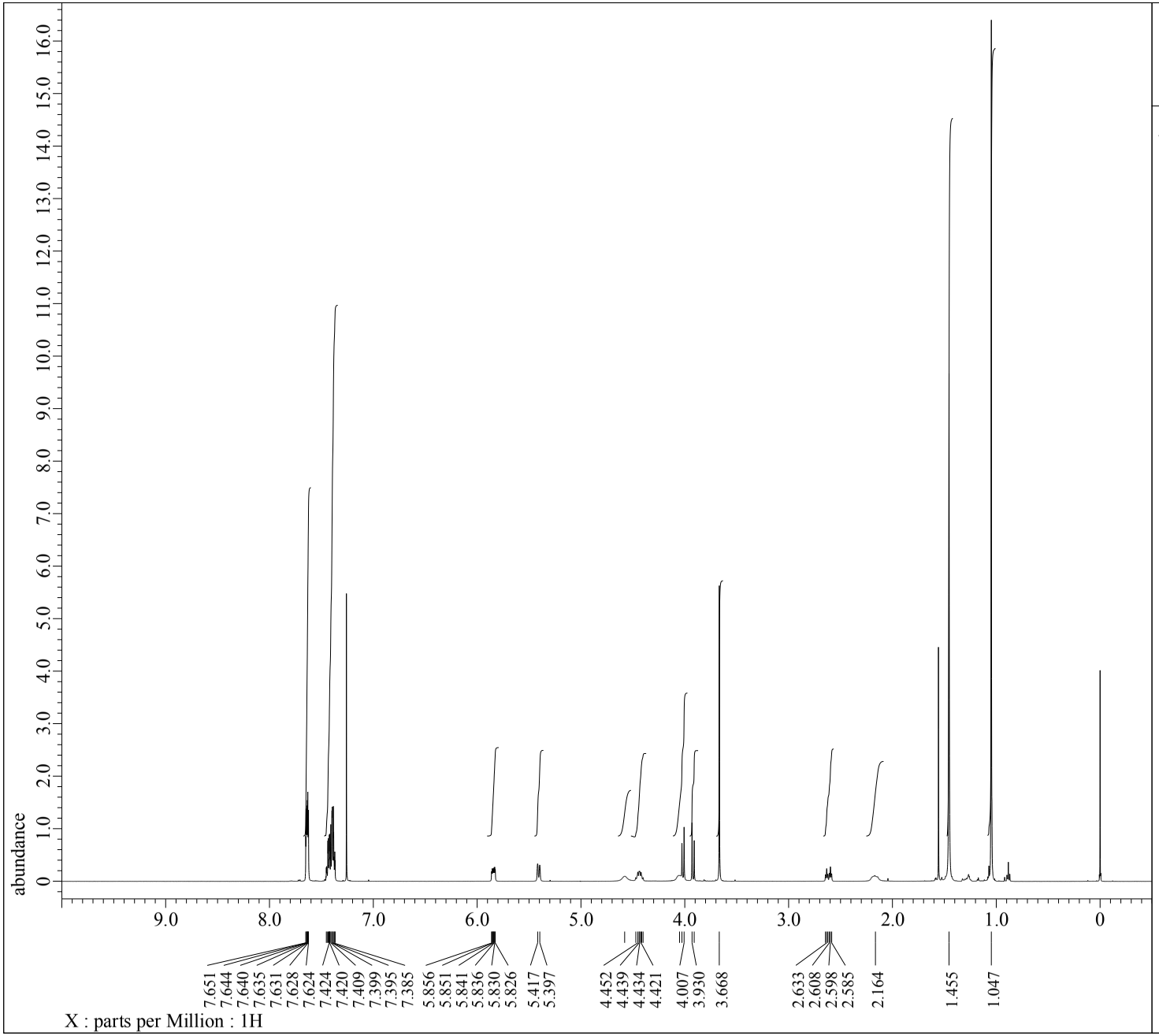


JEOL
Solutions for Innovation

```
Filename          = SY-131213-3-6.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = SY-131213-3
Solvent           = CHLOROFORM-D
Creation_Time     = 13-DEC-2013 14:55:58
Revision_Time     = 21-FEB-2017 11:19:36
Current_Time      = 21-FEB-2017 11:20:08

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 0.8388608[s]
X_Domain          = 13C
X_Freq            = 124.5010059[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.1920929[Hz]
X_Sweep           = 39.0625[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 200
Total_Scans       = 200

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 460.0[dC]
X_90_Width        = 15.6[us]
X_Acq_Time        = 0.8388608[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 5.2[us]
Irr_Atn_Dec       = 25.434[dB]
Irr_Atn_Noe       = 25.434[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.8388608[s]
```

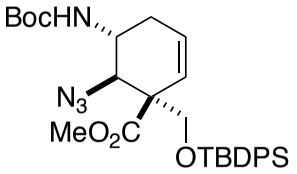


S3



JEOL
Solutions for Innovation

```
Filename          = SY-131206-1-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = SY-131206-1
Solvent           = CHLOROFORM-D
Creation_Time     =  6-DEC-2013 16:50:22
Revision_Time     = 21-FEB-2017 11:32:55
Current_Time      = 21-FEB-2017 11:34:45

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 46
Temp_Get          = 460.0[dC]
X_90_Width        = 7.36[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.68[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```



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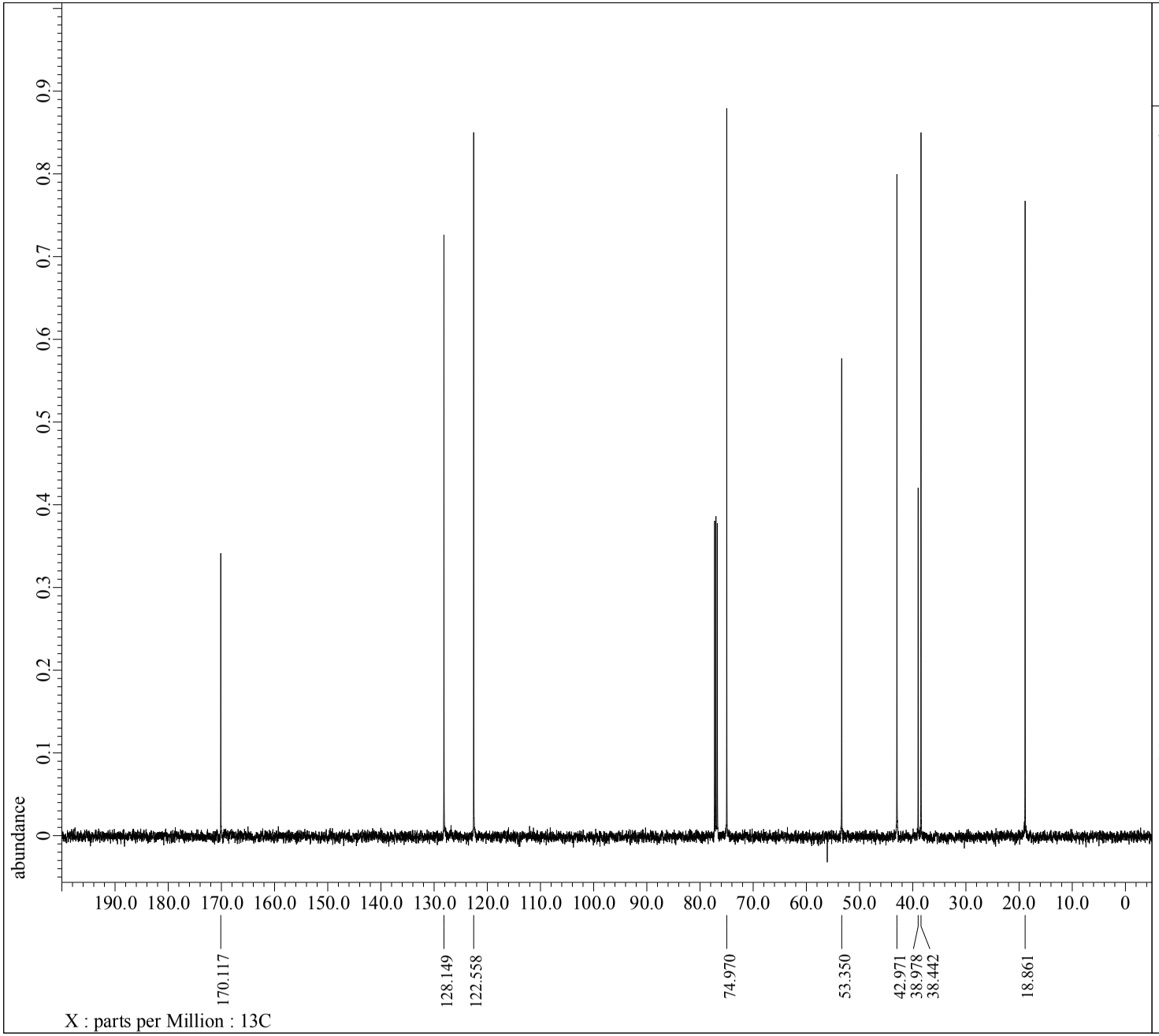
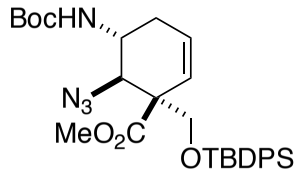


JEOL
Solutions for Innovation

Filename	= SY-131213-2-5.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= SY-131213-2
Solvent	= CHLOROFORM-D
Creation_Time	= 13-DEC-2013 14:40:06
Revision_Time	= 21-FEB-2017 11:36:02
Current_Time	= 21-FEB-2017 11:36:37
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 200
Total_Scans	= 200
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 460.0[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.434[dB]
Irr_Atn_Noe	= 25.434[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]



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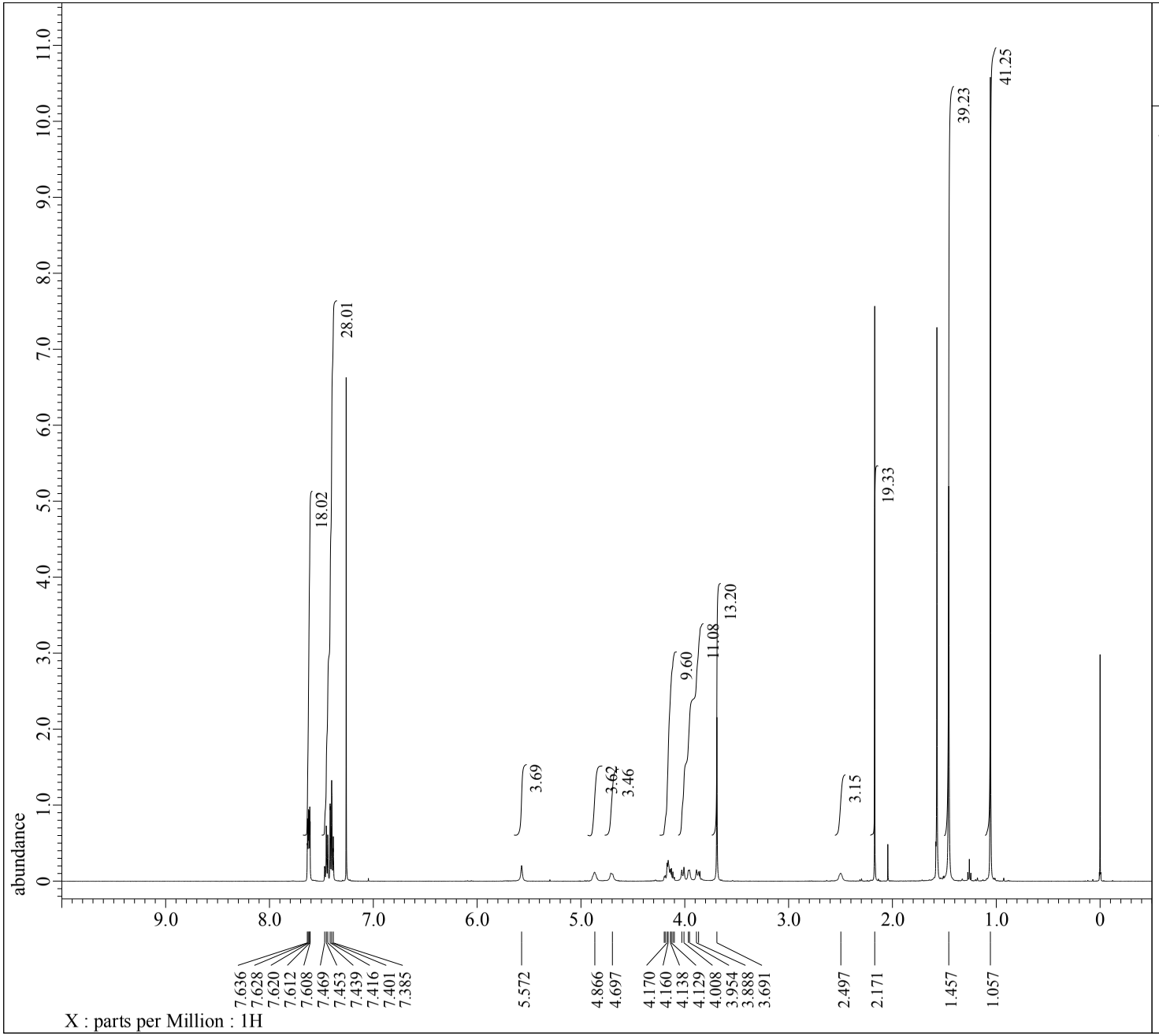


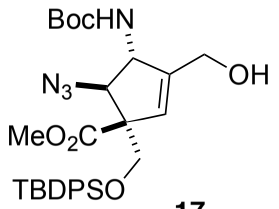
JEOL
Solutions for Innovation

```
Filename          = SY-131107-8-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = SY-131107-8
Solvent           = CHLOROFORM-D
Creation_Time     =  7-NOV-2013 21:17:09
Revision_Time     = 21-FEB-2017 11:45:00
Current_Time      = 21-FEB-2017 11:45:38

Comment           = red
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 52
Temp_Get          = 25.1[dC]
X_90_Width        = 7.36[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 3.68[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```



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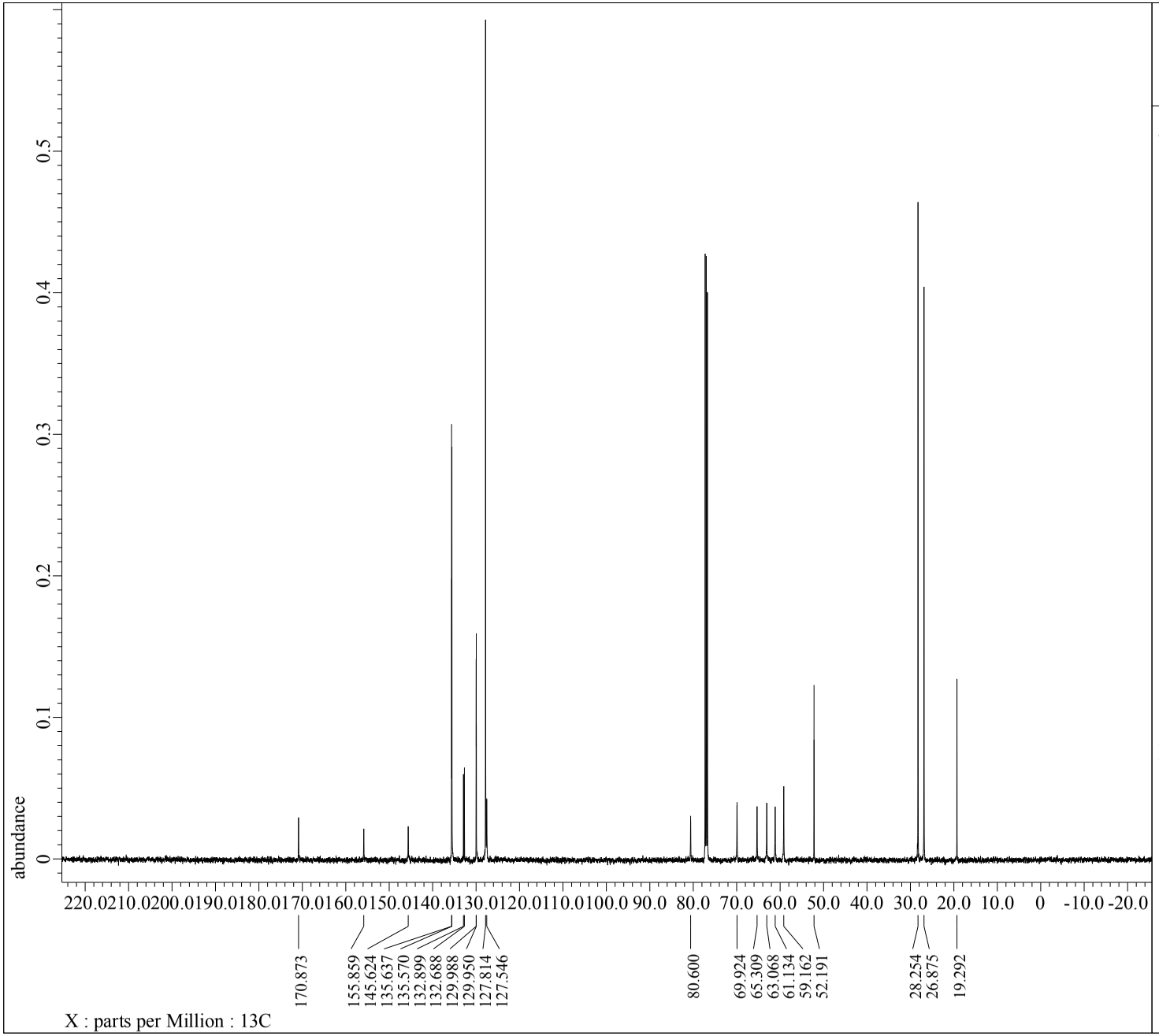
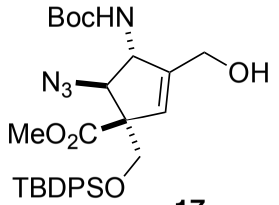
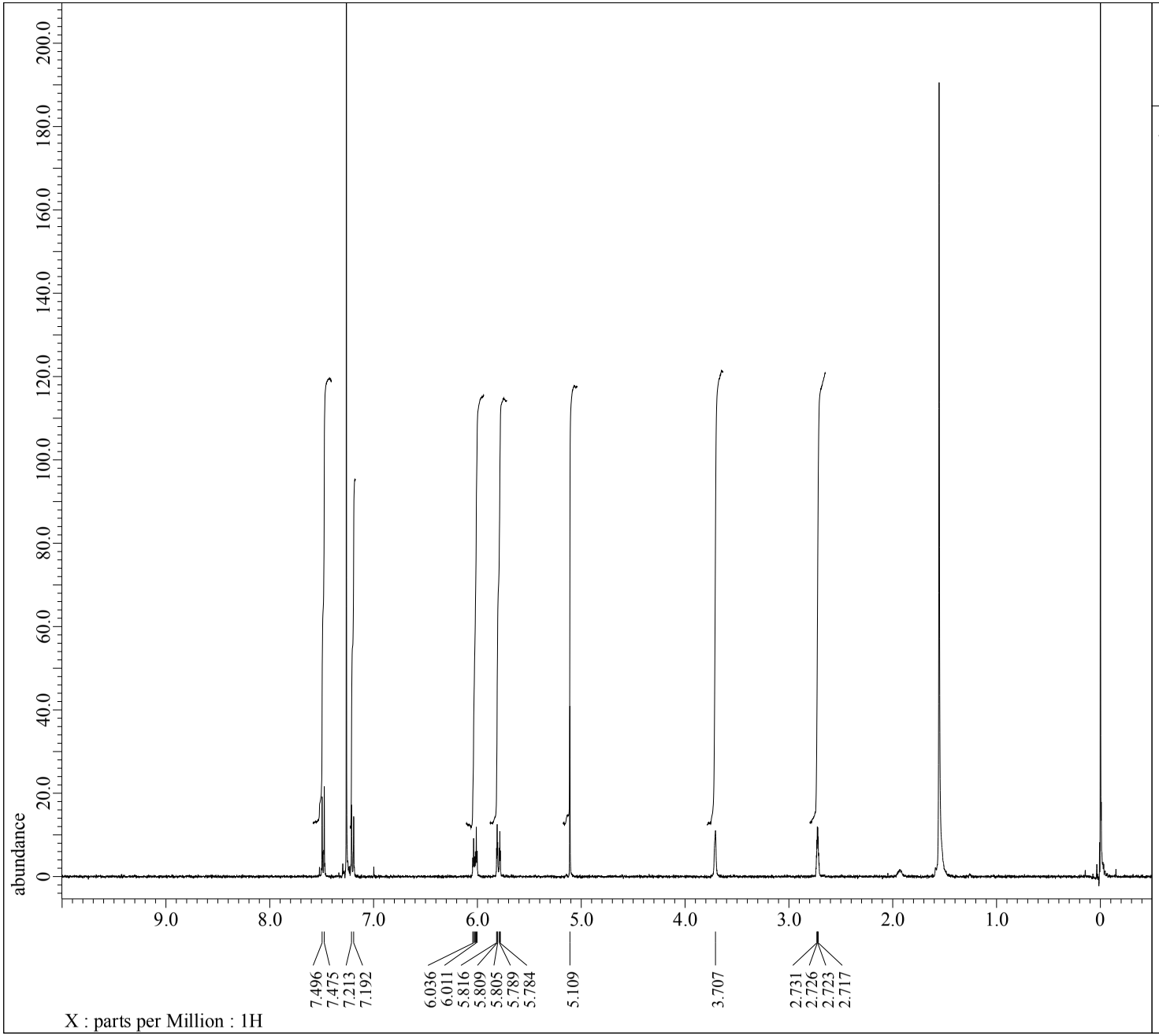


JEOL
Solutions for Innovation

Filename	= SY-131121-7-4.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= SY
Solvent	= CHLOROFORM-D
Creation_Time	= 22-NOV-2013 01:44:26
Revision_Time	= 21-FEB-2017 11:47:09
Current_Time	= 21-FEB-2017 11:48:55
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 2400
Total_Scans	= 2400
Relaxation_Delay	= 2[s]
Recvr_Gain	= 52
Temp_Get	= 460.0[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.434[dB]
Irr_Atn_Noe	= 25.434[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]

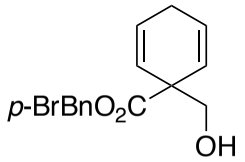


17

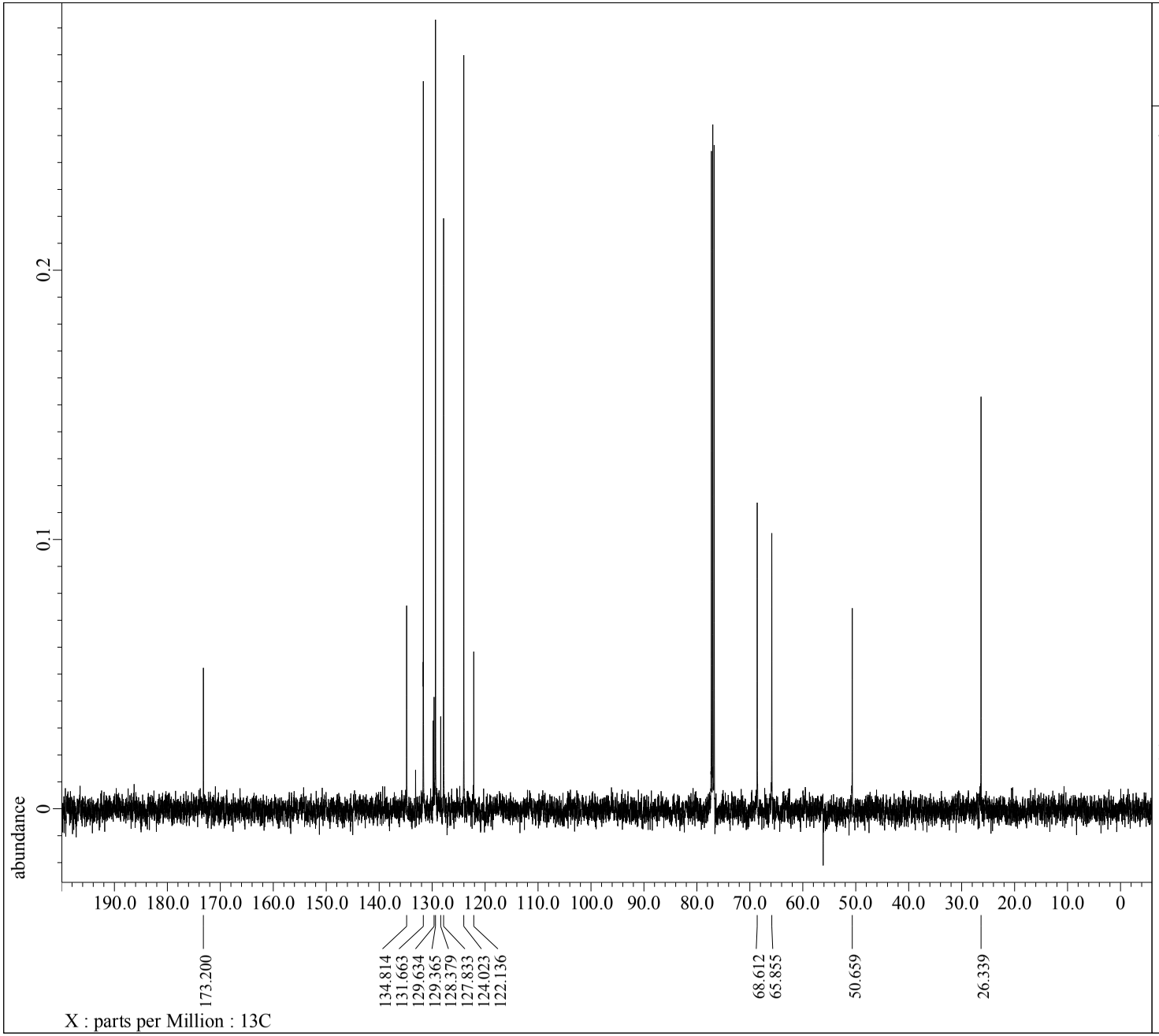


JEOL
Solutions for Innovation

```
Filename          = SY-150430-21NON_E6_FT-2.jd
Author            = MI
Experiment        = NON
Solvent           = CDCL3
Creation_Time     = 15-FEB-2017 00:14:08
Revision_Time     = 21-FEB-2017 12:10:41
Current_Time      = 21-FEB-2017 12:11:14

Comment           = 800-pure
Data_Format       = 1D COMPLEX
Dim_Size          = 16384
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = ALICE_NMR

X_Domain          = 1H
X_Freq            = 399.7844939[MHz]
X_Offset          = 0[Hz]
X_Points          = 16384
X_Prescans        = 1
X_Sweep           = 7.99200781[kHz]
Scans             = 8

Relaxation_Delay = 4.94999981
Recvr_Gain        = 23
Temp_Get          = 21.79999924[dC]
```

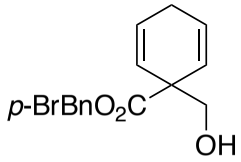


S4

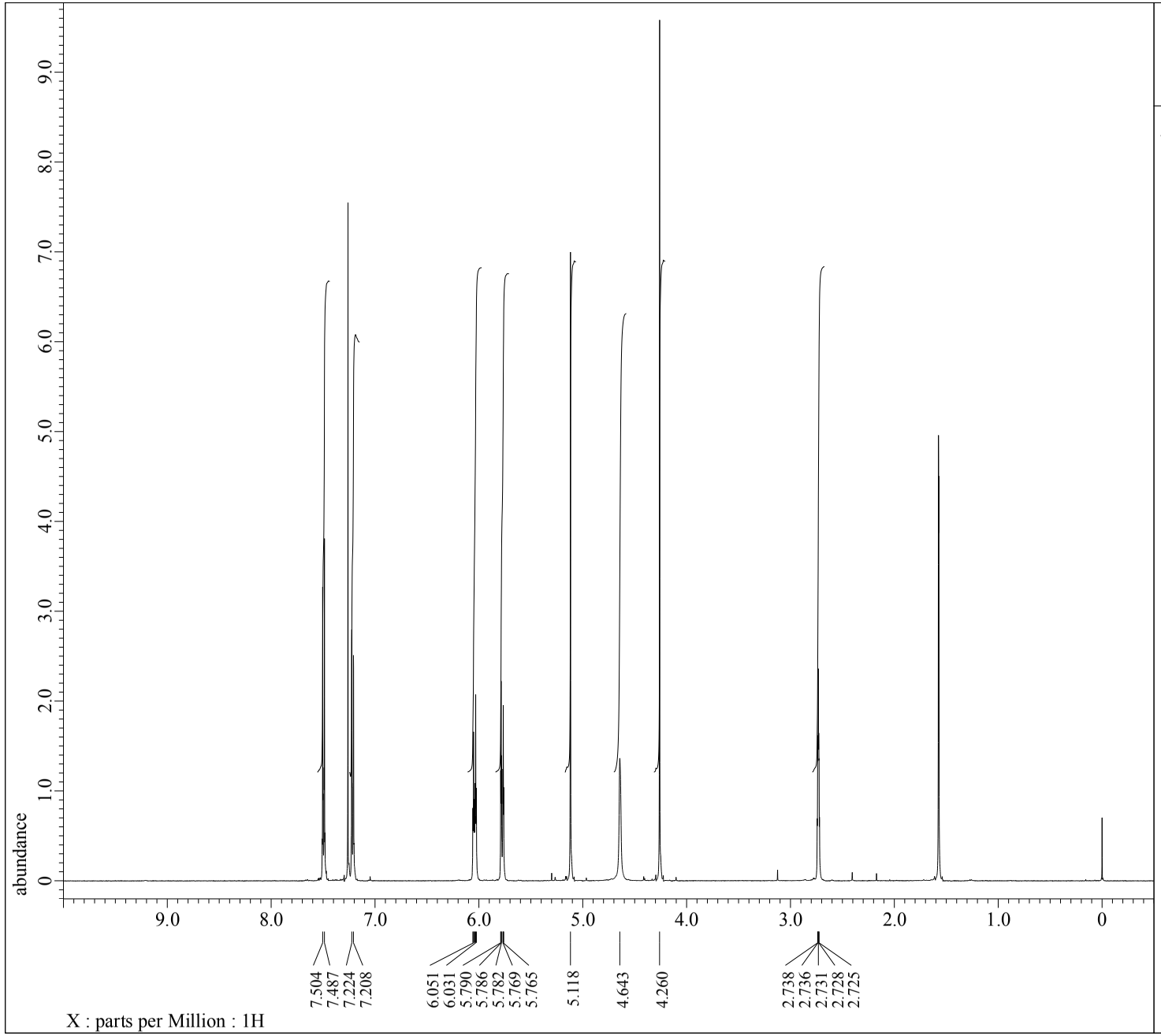


JEOL
Solutions for Innovation

Filename	= SY-150914-7-4.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= SY-150914-7
Solvent	= CHLOROFORM-D
Creation_Time	= 14-SEP-2015 16:22:14
Revision_Time	= 21-FEB-2017 12:13:00
Current_Time	= 21-FEB-2017 12:13:31
Comment	= p-Br-1
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 200
Total_Scans	= 200
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 25.4[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.042[dB]
Irr_Atn_Noe	= 25.042[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]

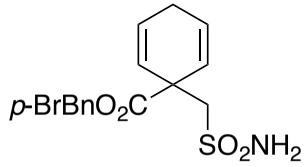


S4

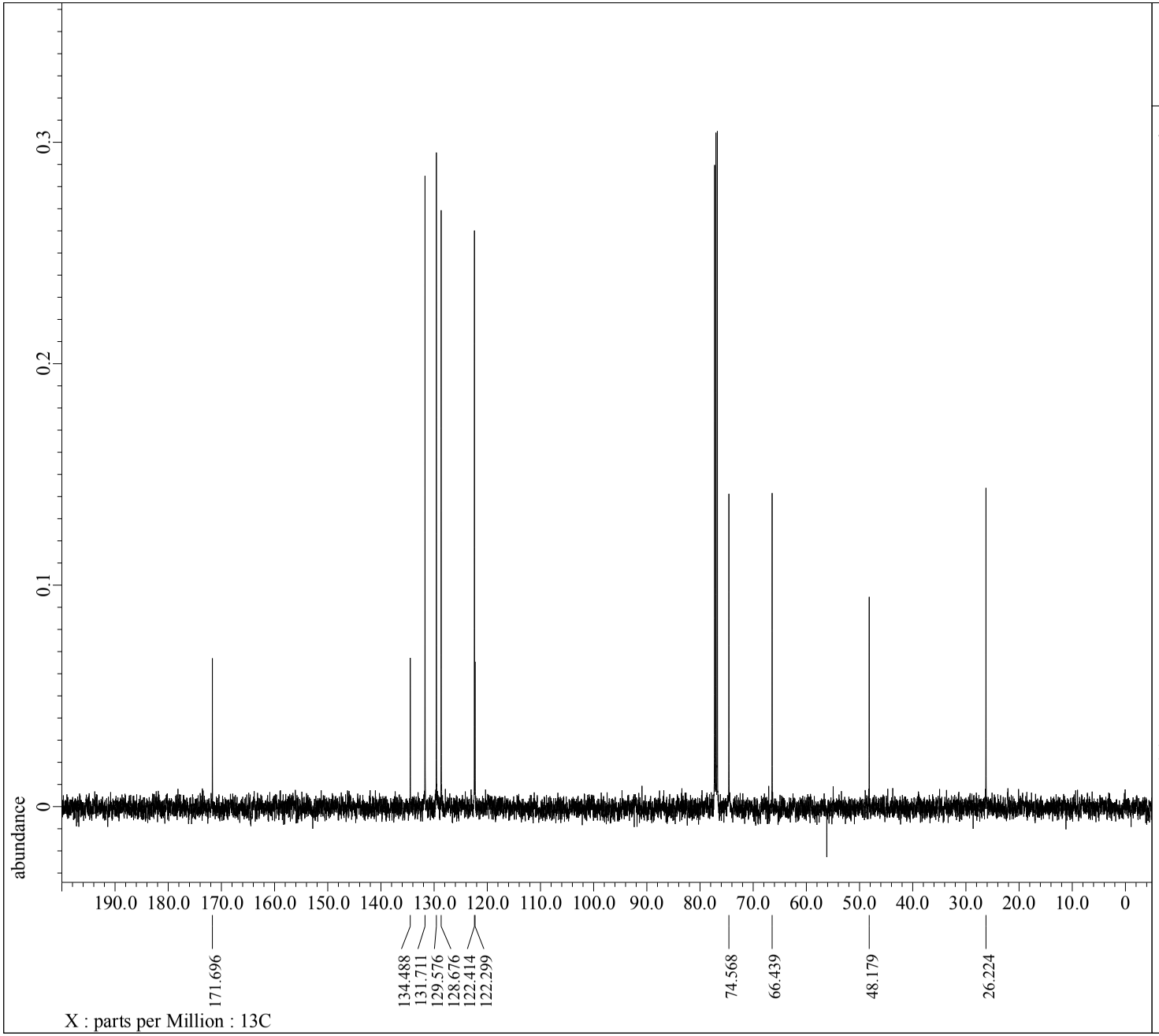


JEOL
Solutions for Innovation

```
Filename          = SY-151222-2-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = SY-151222-2
Solvent           = CHLOROFORM-D
Creation_Time     = 22-DEC-2015 21:36:28
Revision_Time     = 21-FEB-2017 17:44:25
Current_Time      = 21-FEB-2017 17:47:12

Comment           = p-BrBn-SO2NH2
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 52
Temp_Get          = 24[dC]
X_90_Width        = 8.16[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```

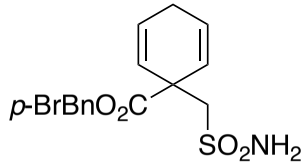


S5

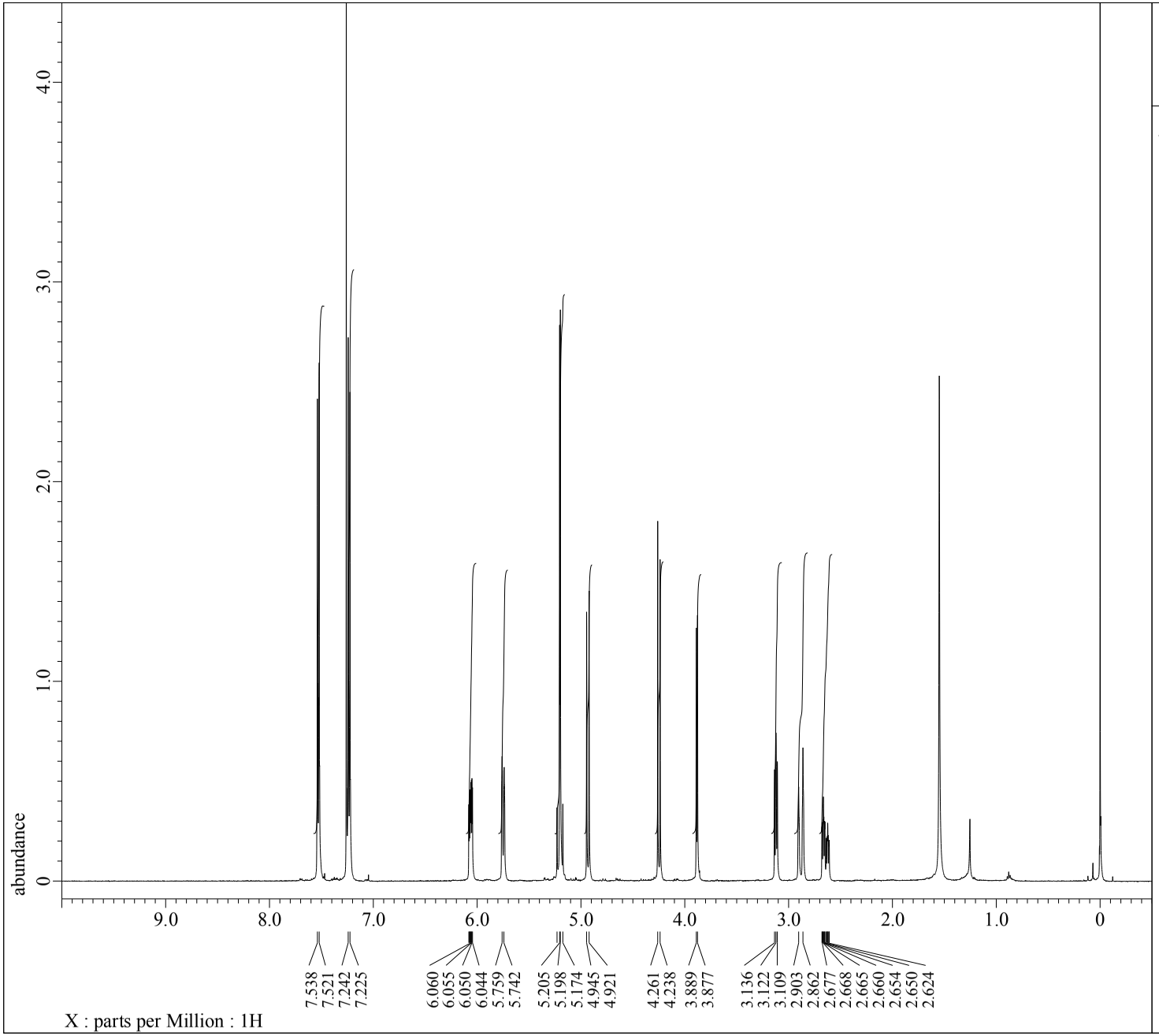


JEOL
Solutions for Innovation

Filename	= SY-151222-3-5.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= SY-151222-3
Solvent	= CHLOROFORM-D
Creation_Time	= 22-DEC-2015 21:55:56
Revision_Time	= 21-FEB-2017 17:49:10
Current_Time	= 21-FEB-2017 17:49:41
Comment	= p-BrBn-SO2NH2
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 221
Total_Scans	= 221
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 24.2[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.042[dB]
Irr_Atn_Noe	= 25.042[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]

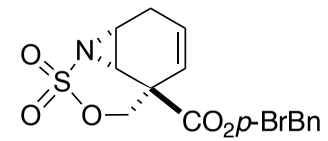


S5

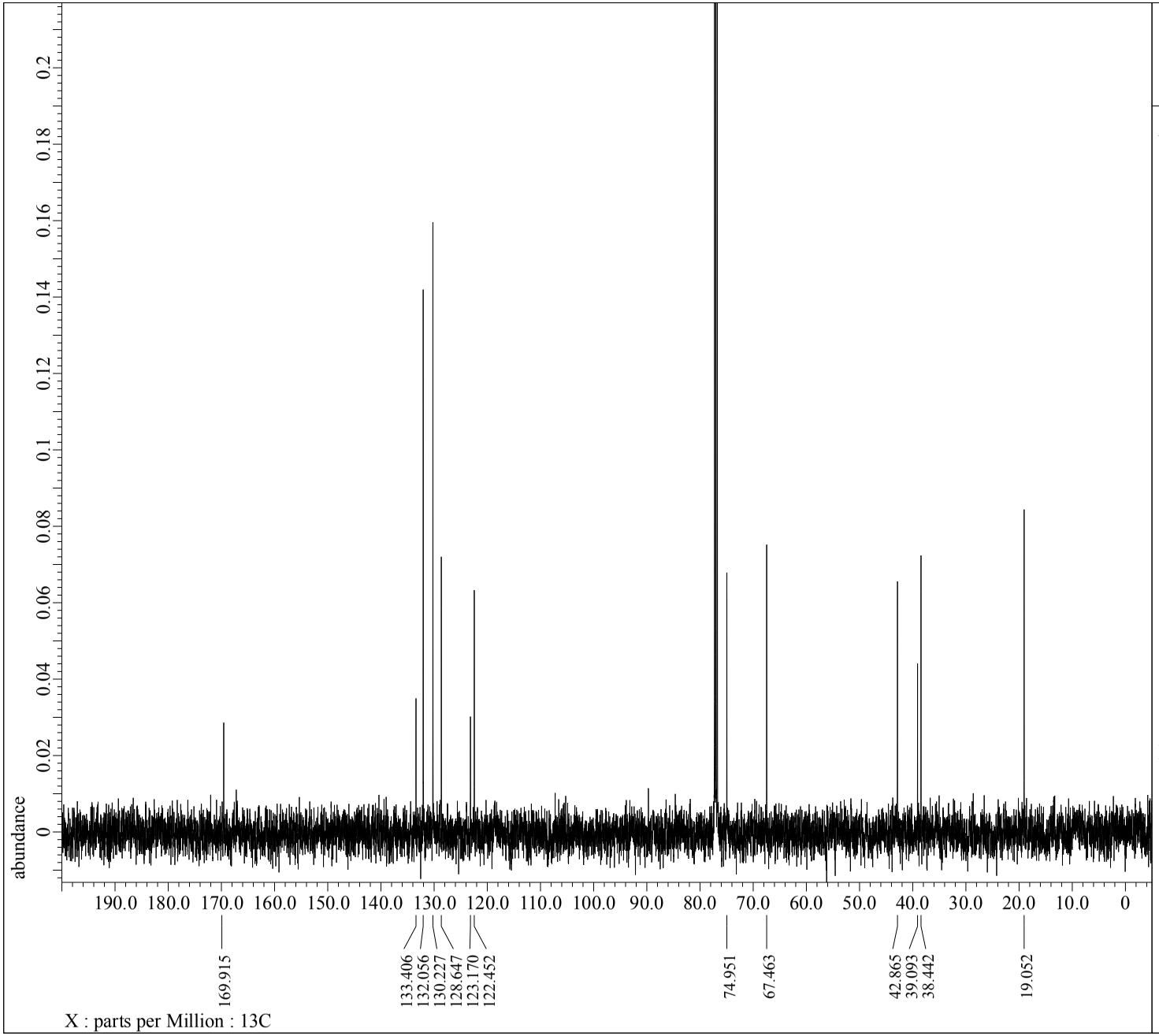


JEOL
Solutions for Innovation

```
Filename          = SY-150511-3-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = SY-150511-3
Solvent           = CHLOROFORM-D
Creation_Time     = 11-MAY-2015 16:34:18
Revision_Time     = 21-FEB-2017 23:23:09
Current_Time      = 21-FEB-2017 23:24:55

Comment           = 805-ptlc
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 50
Temp_Get          = 25.4[dC]
X_90_Width        = 8.16[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```

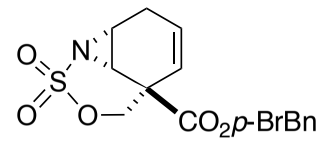


S6

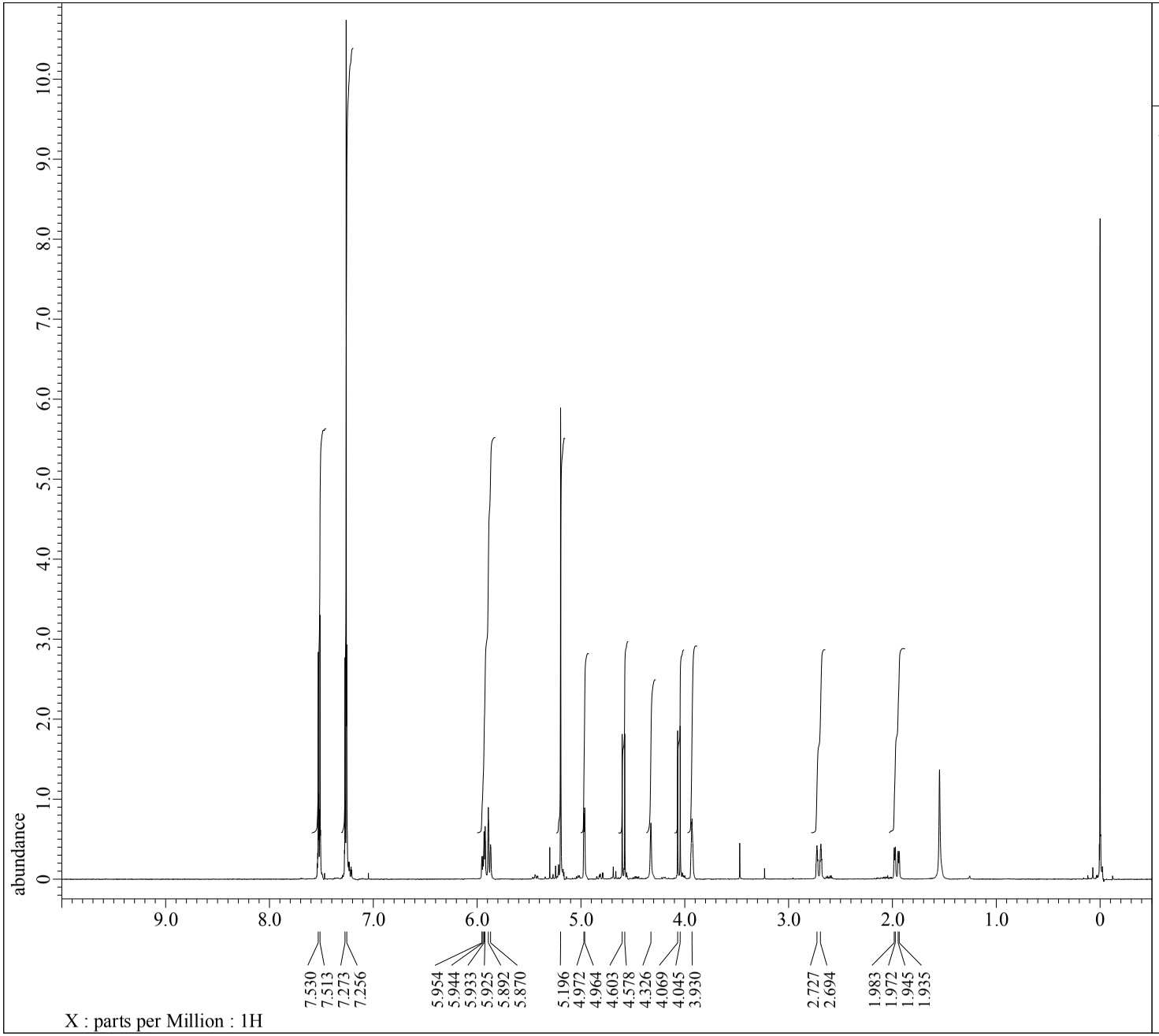


JEOL
Solutions for Innovation

Filename	= SY-151223-3-7.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= SY-151223-3
Solvent	= CHLOROFORM-D
Creation_Time	= 23-DEC-2015 10:42:45
Revision_Time	= 21-FEB-2017 23:34:29
Current_Time	= 21-FEB-2017 23:36:28
Comment	= p-BrBn-aziridine
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 94
Total_Scans	= 94
Relaxation_Delay	= 2[s]
Recvr_Gain	= 48
Temp_Get	= 24.1[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.042[dB]
Irr_Atn_Noe	= 25.042[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]

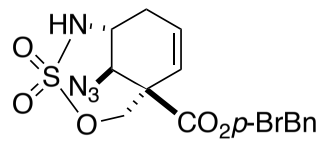


S6

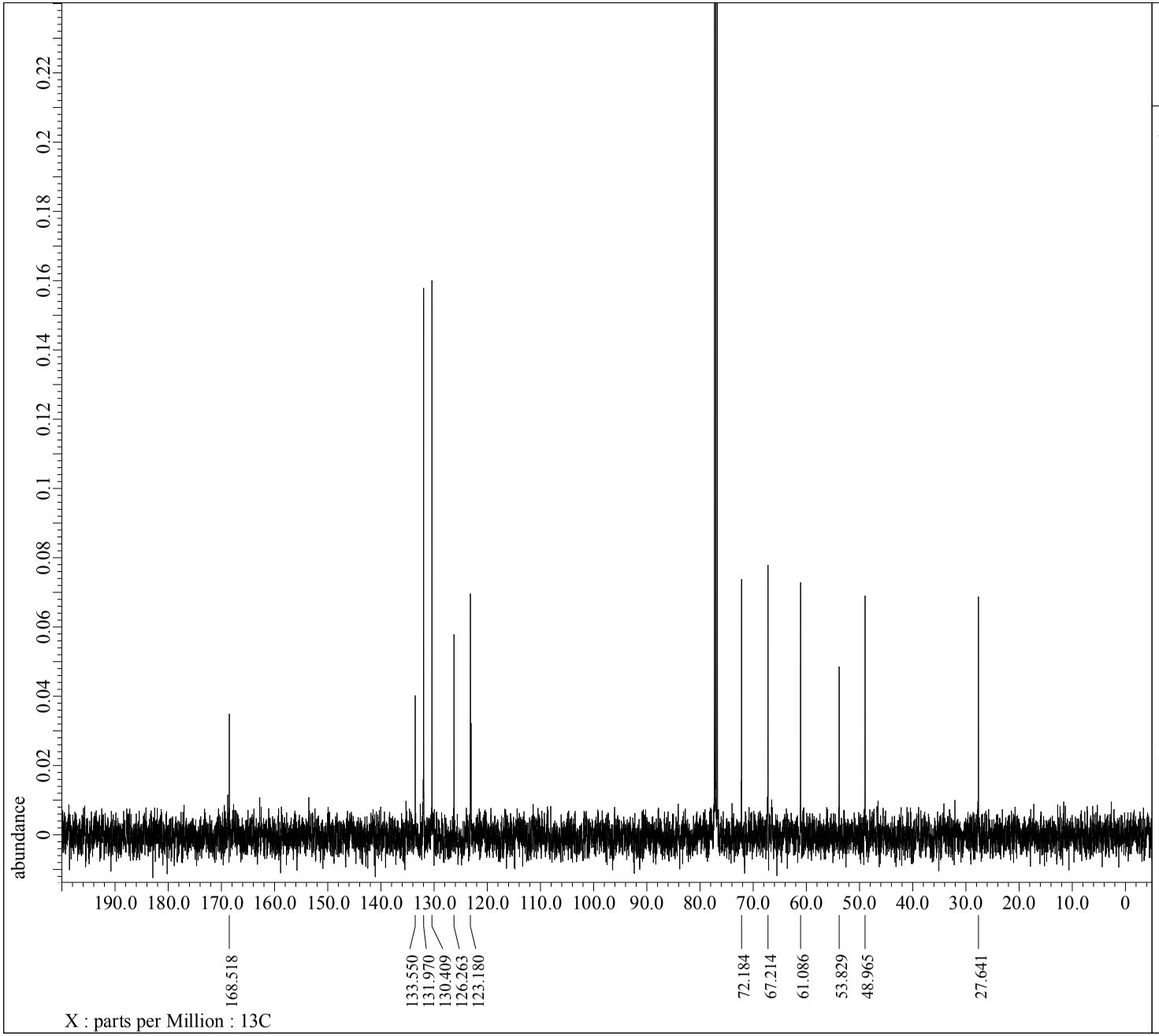


JEOL
Solutions for Innovation

```
Filename          = SY-151223-1-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = SY-151223-1
Solvent           = CHLOROFORM-D
Creation_Time     = 23-DEC-2015 10:05:52
Revision_Time     = 22-FEB-2017 10:22:47
Current_Time      = 22-FEB-2017 10:23:03

Comment           = p-BrBn-N3-roeki
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 54
Temp_Get          = 23.8[dC]
X_90_Width        = 8.16[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```

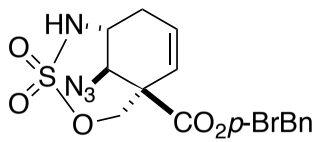


12b

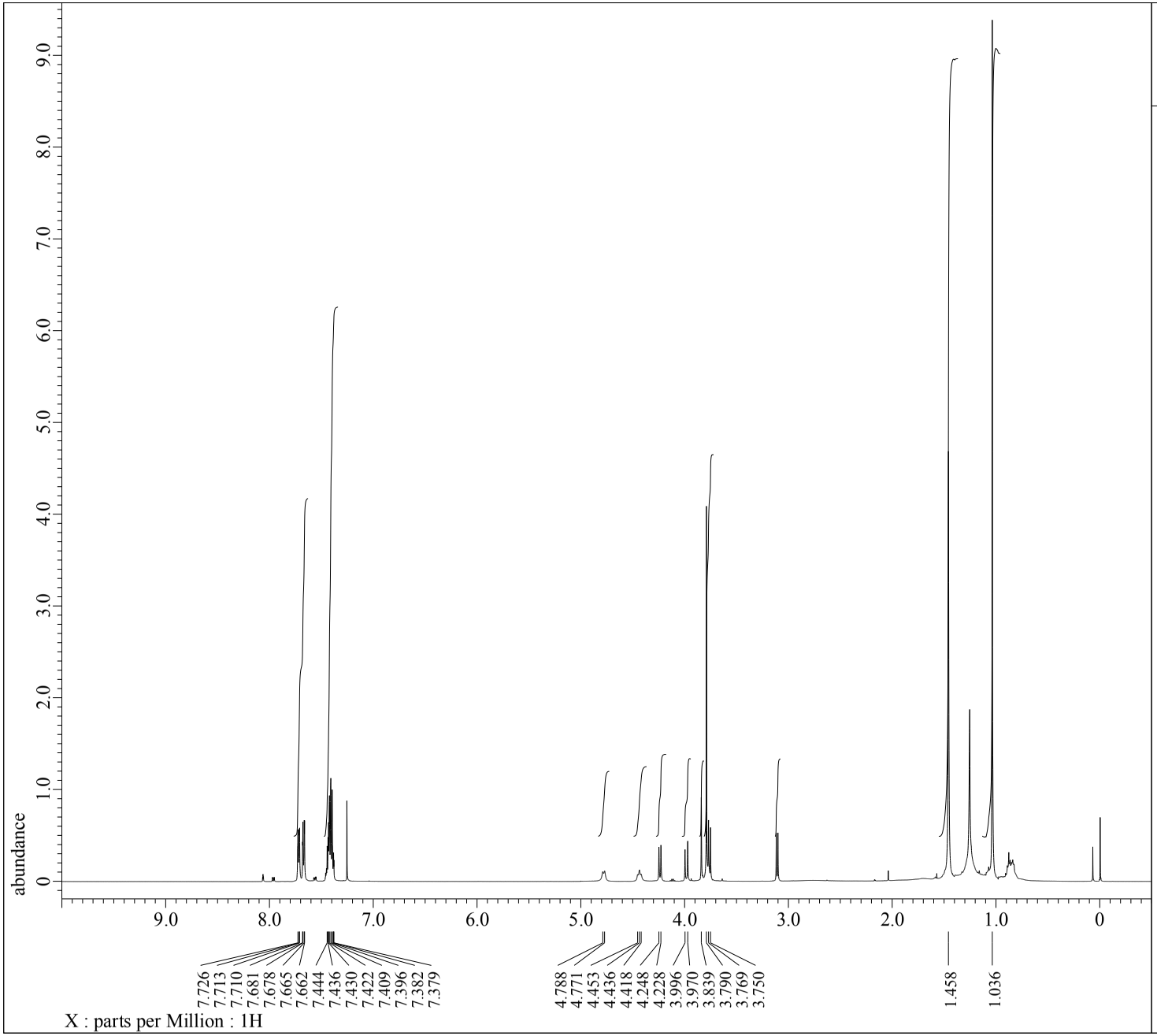


JEOL
Solutions for Innovation

Filename	= SY-151223-2-6.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= SY-151223-2
Solvent	= CHLOROFORM-D
Creation_Time	= 23-DEC-2015 10:27:59
Revision_Time	= 22-FEB-2017 10:25:59
Current_Time	= 22-FEB-2017 10:26:36
Comment	= p-BrBn-N3-roeki
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 145
Total_Scans	= 145
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 24.1[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.042[dB]
Irr_Atn_Noe	= 25.042[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]

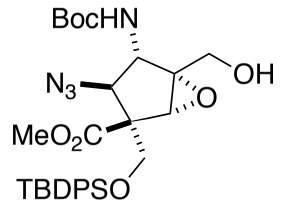


12b

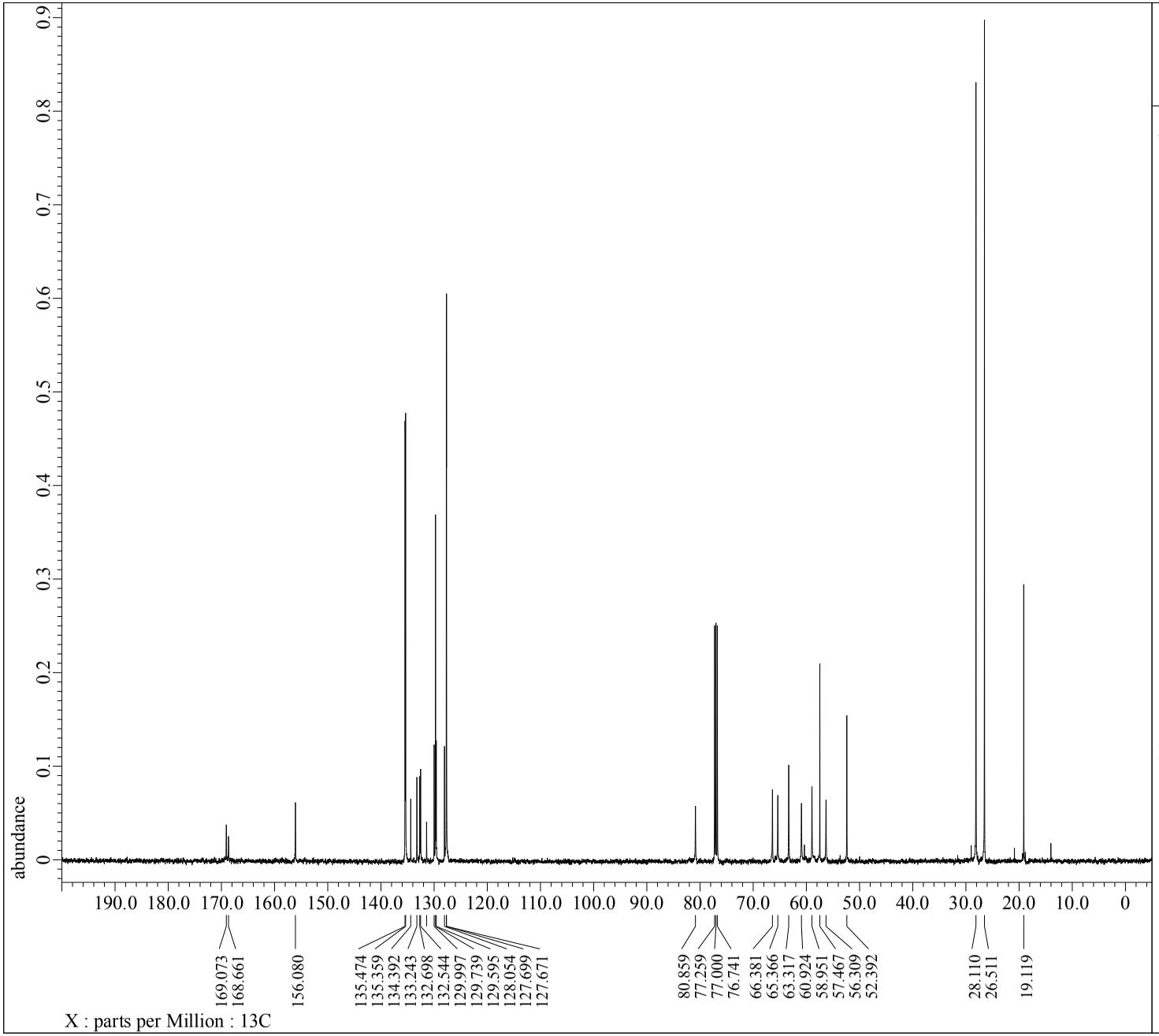


JEOL
Solutions for Innovation

```
Filename          = AG-151211-3-4.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#545987
Solvent           = CHLOROFORM-D
Creation_Time     = 11-DEC-2015 14:46:17
Revision_Time     = 22-FEB-2017 11:30:13
Current_Time      = 22-FEB-2017 11:31:57

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 34
Temp_Get          = 26.5[dC]
X_90_Width        = 8.16[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```

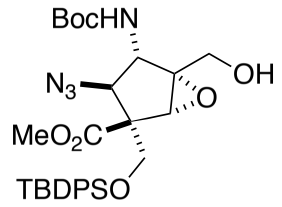


S7

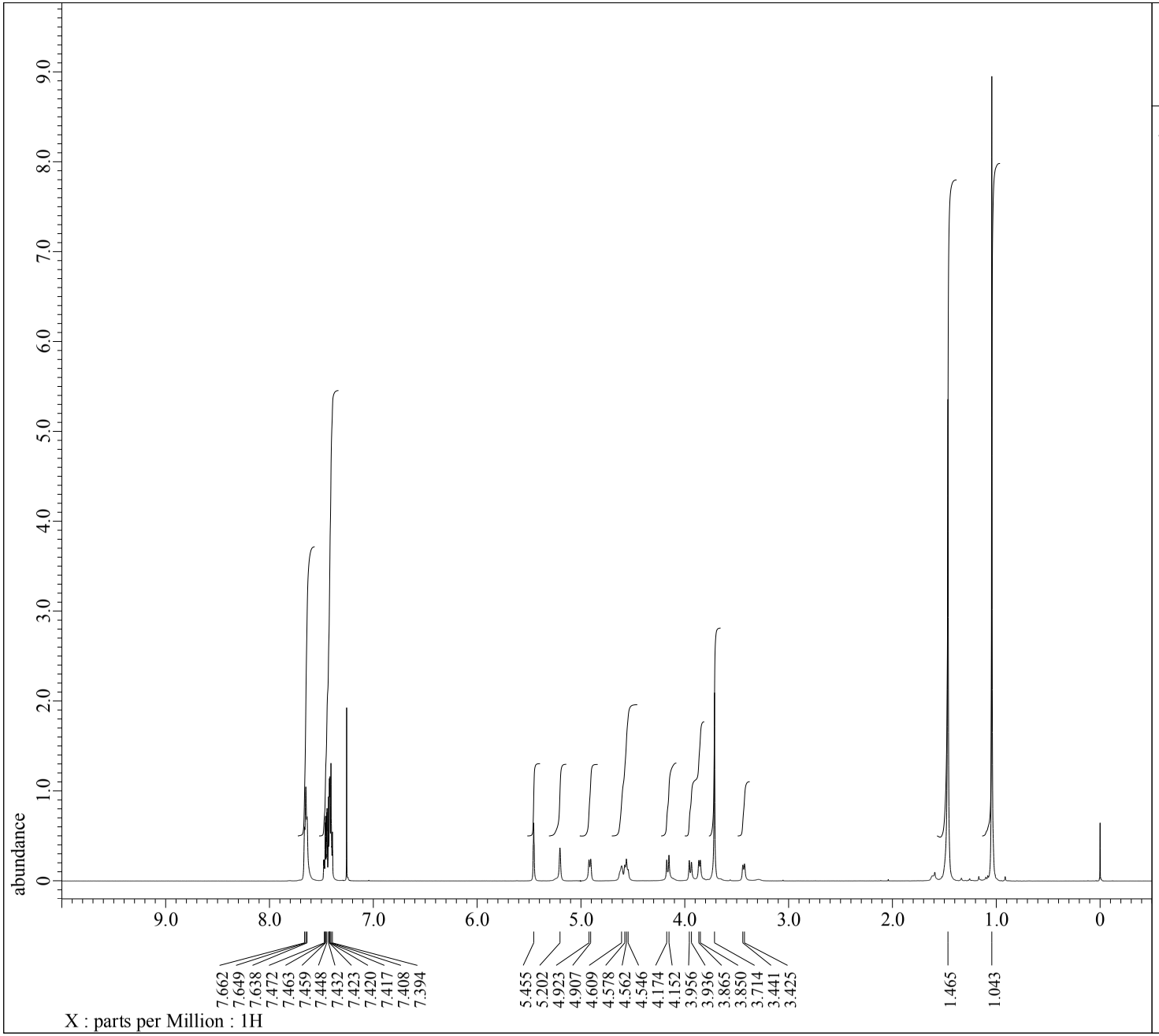


JEOL
Solutions for Innovation

Filename	= AG-151211-1-4.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#326402
Solvent	= CHLOROFORM-D
Creation_Time	= 11-DEC-2015 09:23:21
Revision_Time	= 22-FEB-2017 12:50:13
Current_Time	= 22-FEB-2017 12:52:59
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 917
Total_Scans	= 917
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 25[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.042[dB]
Irr_Atn_Noe	= 25.042[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]

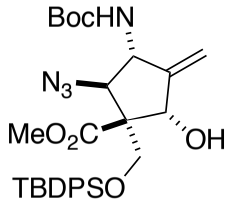


S7

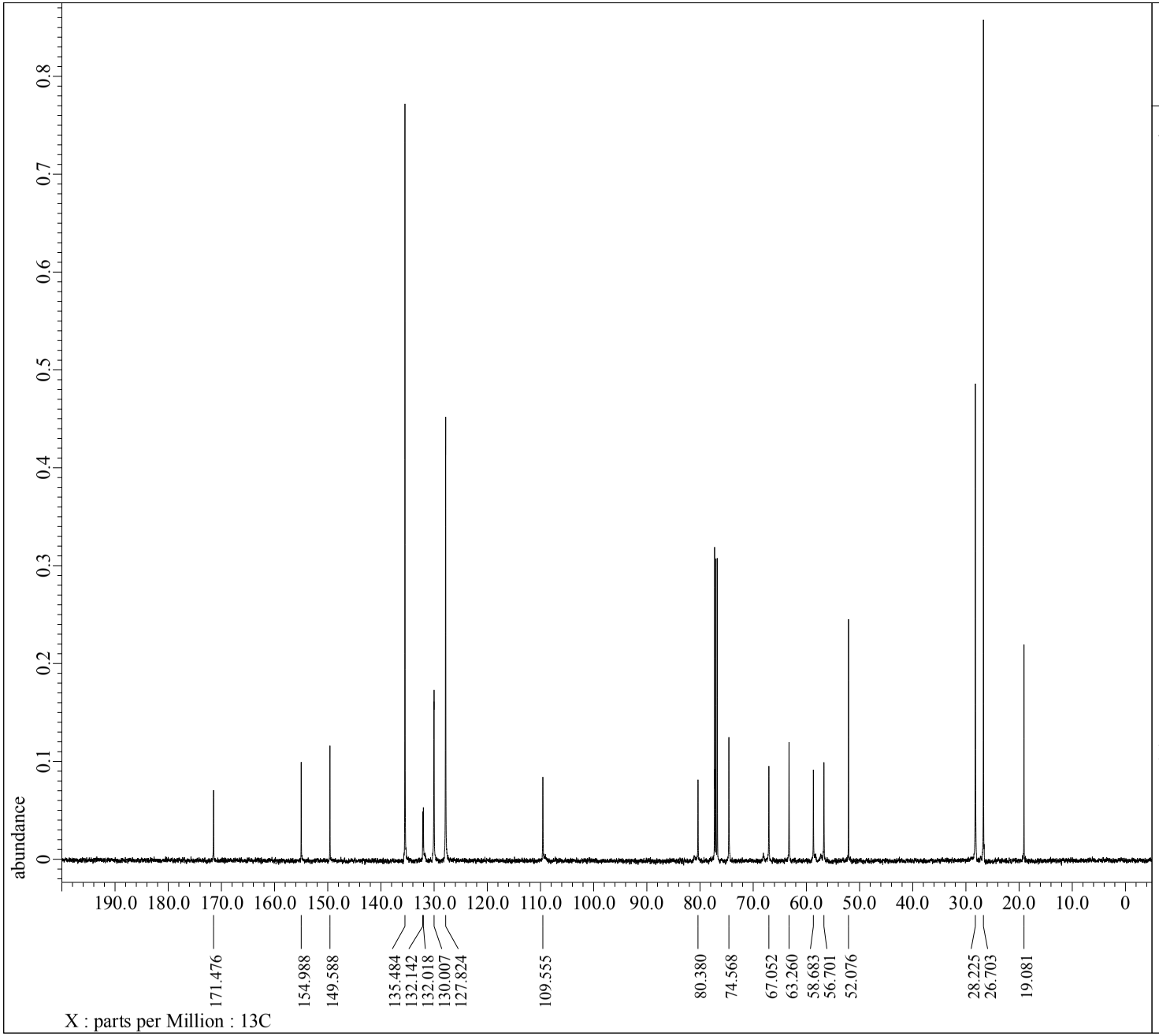


JEOL
Solutions for Innovation

Filename	= AG-160112-1-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#569052
Solvent	= CHLOROFORM-D
Creation_Time	= 12-JAN-2016 15:25:30
Revision_Time	= 22-FEB-2017 15:22:16
Current_Time	= 22-FEB-2017 15:22:37
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 1.76422912[s]
X_Domain	= 1H
X_Freq	= 495.13191398[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.5668198[Hz]
X_Sweep	= 9.28677563[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 495.13191398[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 32
Temp_Get	= 25.5[dC]
X_90_Width	= 8.16[us]
X_Acq_Time	= 1.76422912[s]
X_Angle	= 45[deg]
X_Atn	= 4[dB]
X_Pulse	= 4.08[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.76422912[s]

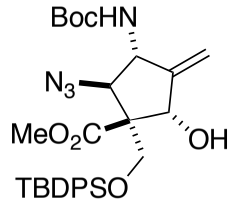


20

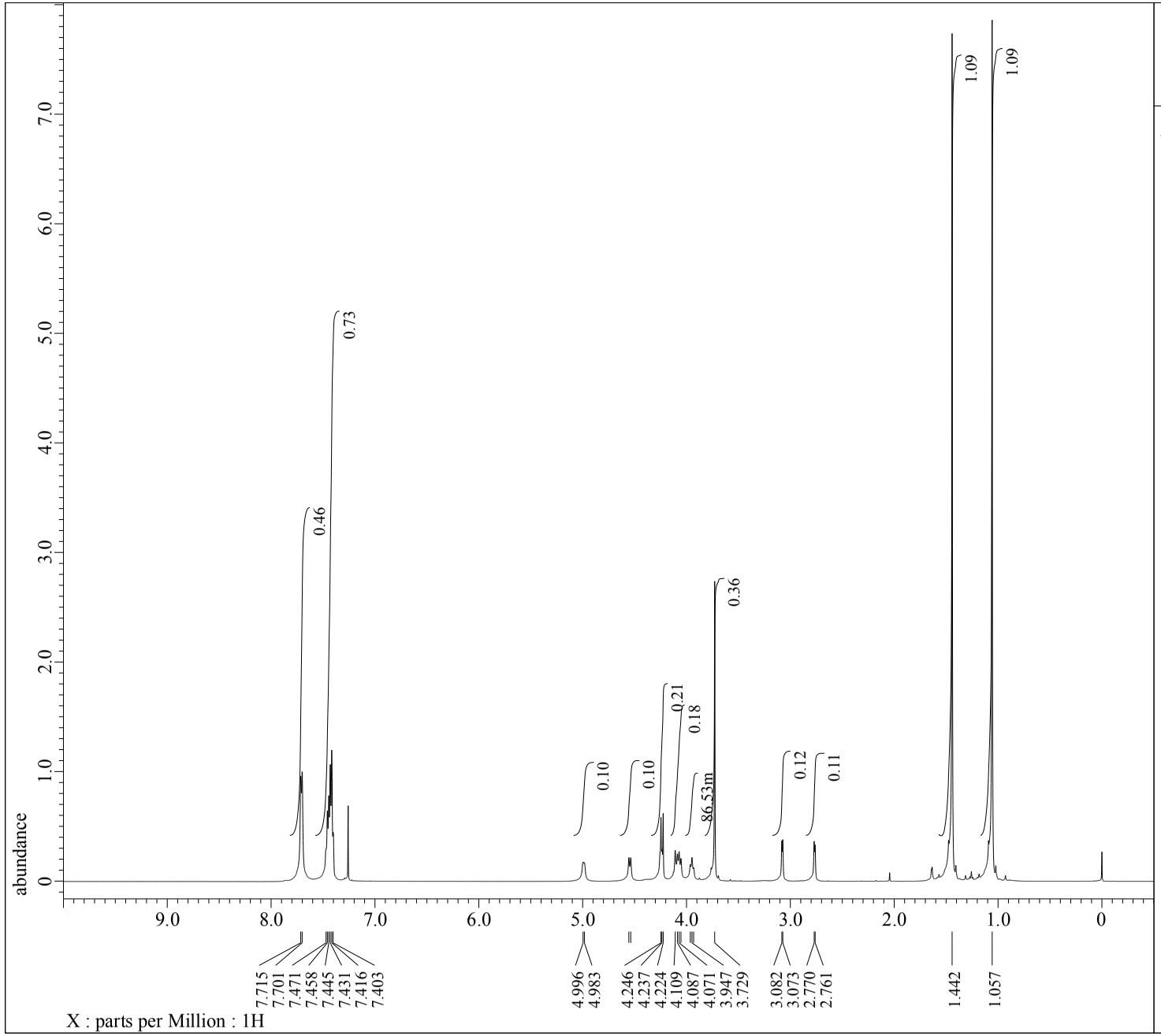


JEOL
Solutions for Innovation

Filename	= AG-160112-2-6.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#573865
Solvent	= CHLOROFORM-D
Creation_Time	= 12-JAN-2016 16:20:02
Revision_Time	= 22-FEB-2017 15:30:39
Current_Time	= 22-FEB-2017 15:31:21
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 1000
Total_Scans	= 1000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 25.8[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.042[dB]
Irr_Atn_Noe	= 25.042[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]

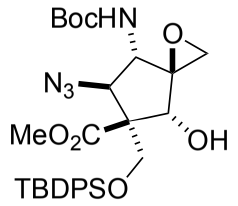


20



JEOL
Solutions for Innovation

```
Filename          = 170222-1 alcohol-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#327734
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2017 08:31:52
Revision_Time     =  2-MAR-2017 11:39:12
Current_Time      =  2-MAR-2017 11:39:50

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 34
Temp_Get          = 20.6[dC]
X_90_Width        = 8.16[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```



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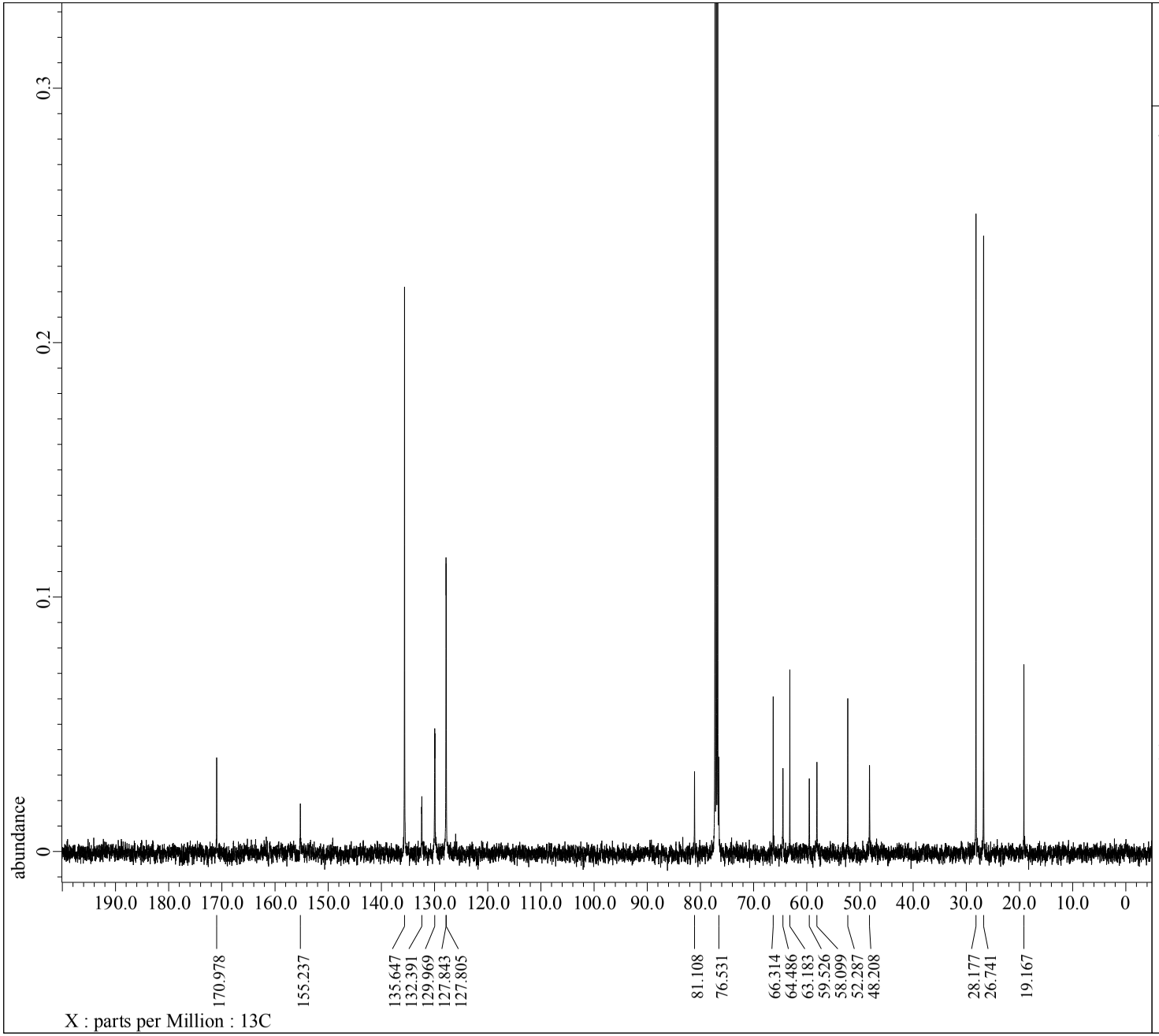
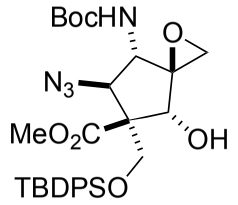
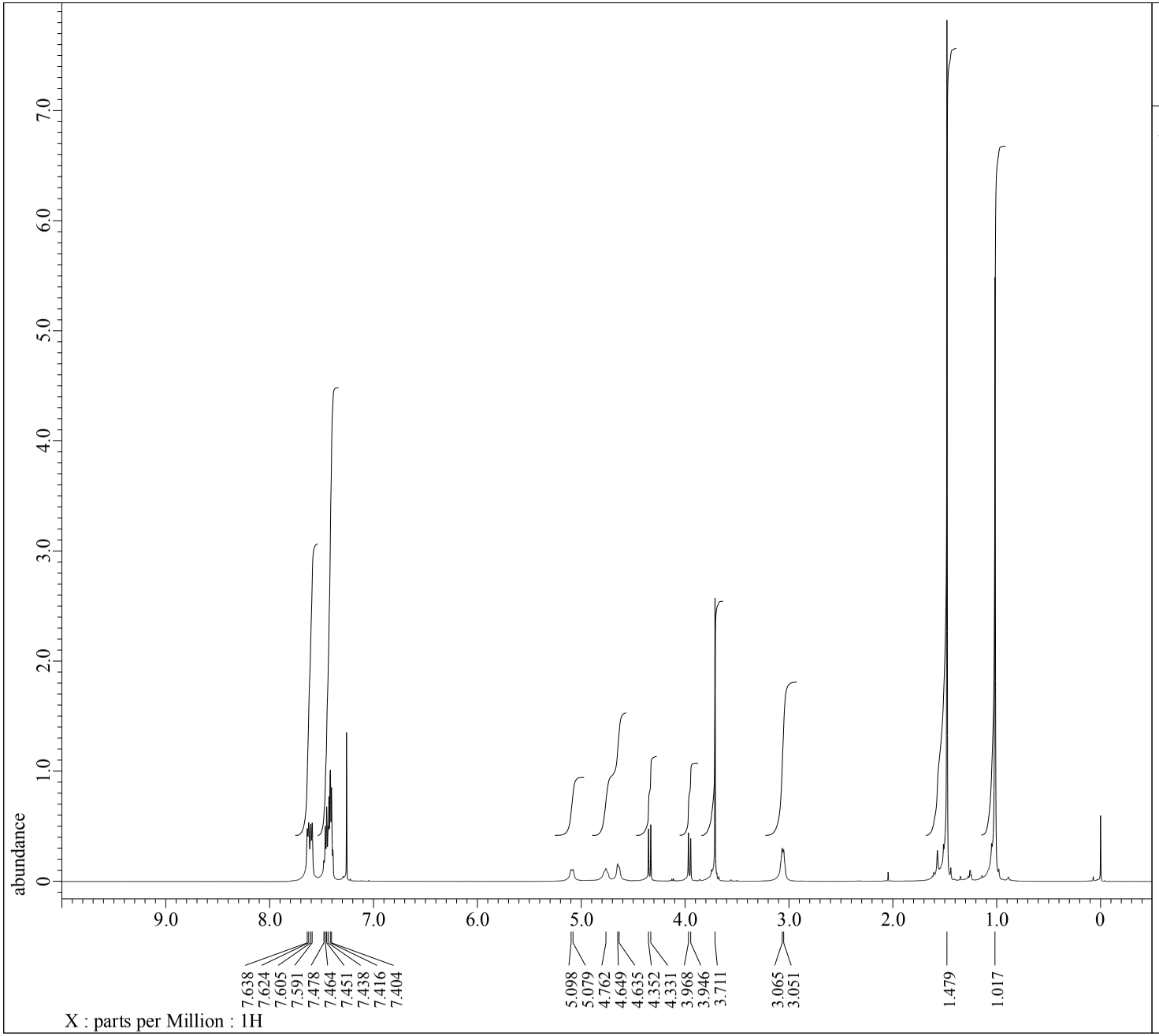


JEOL
Solutions for Innovation

Filename	= 170222-2 alcohol C-5.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#333734
Solvent	= CHLOROFORM-D
Creation_Time	= 22-FEB-2017 09:38:40
Revision_Time	= 2-MAR-2017 11:41:19
Current_Time	= 2-MAR-2017 11:41:53
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 1200
Total_Scans	= 1200
Relaxation_Delay	= 2[s]
Recvr_Gain	= 54
Temp_Get	= 21[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 23.2[dB]
Irr_Atn_Noe	= 23.2[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]

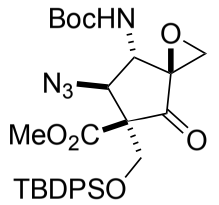


21

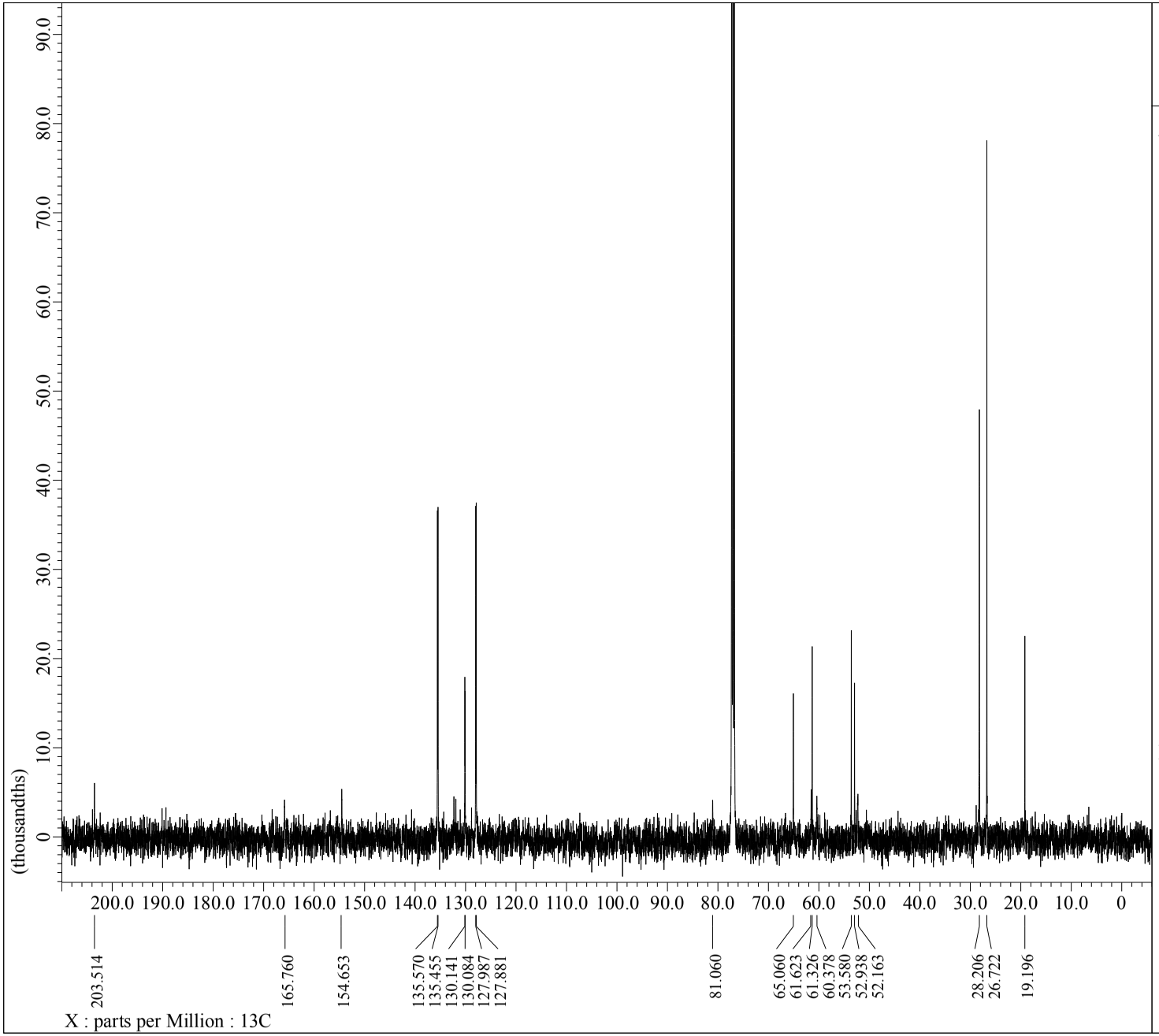


JEOL
Solutions for Innovation

```
Filename          = 170221-1 ketone-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#792400
Solvent           = CHLOROFORM-D
Creation_Time     = 21-FEB-2017 21:27:12
Revision_Time     =  2-MAR-2017 12:08:26
Current_Time      =  2-MAR-2017 12:09:13

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 16
Total_Scans       = 16

Relaxation_Delay = 5[s]
Recvr_Gain        = 40
Temp_Get          = 21.1[dC]
X_90_Width        = 8.16[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```

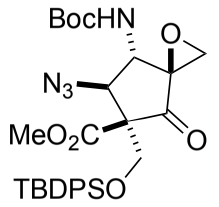


22

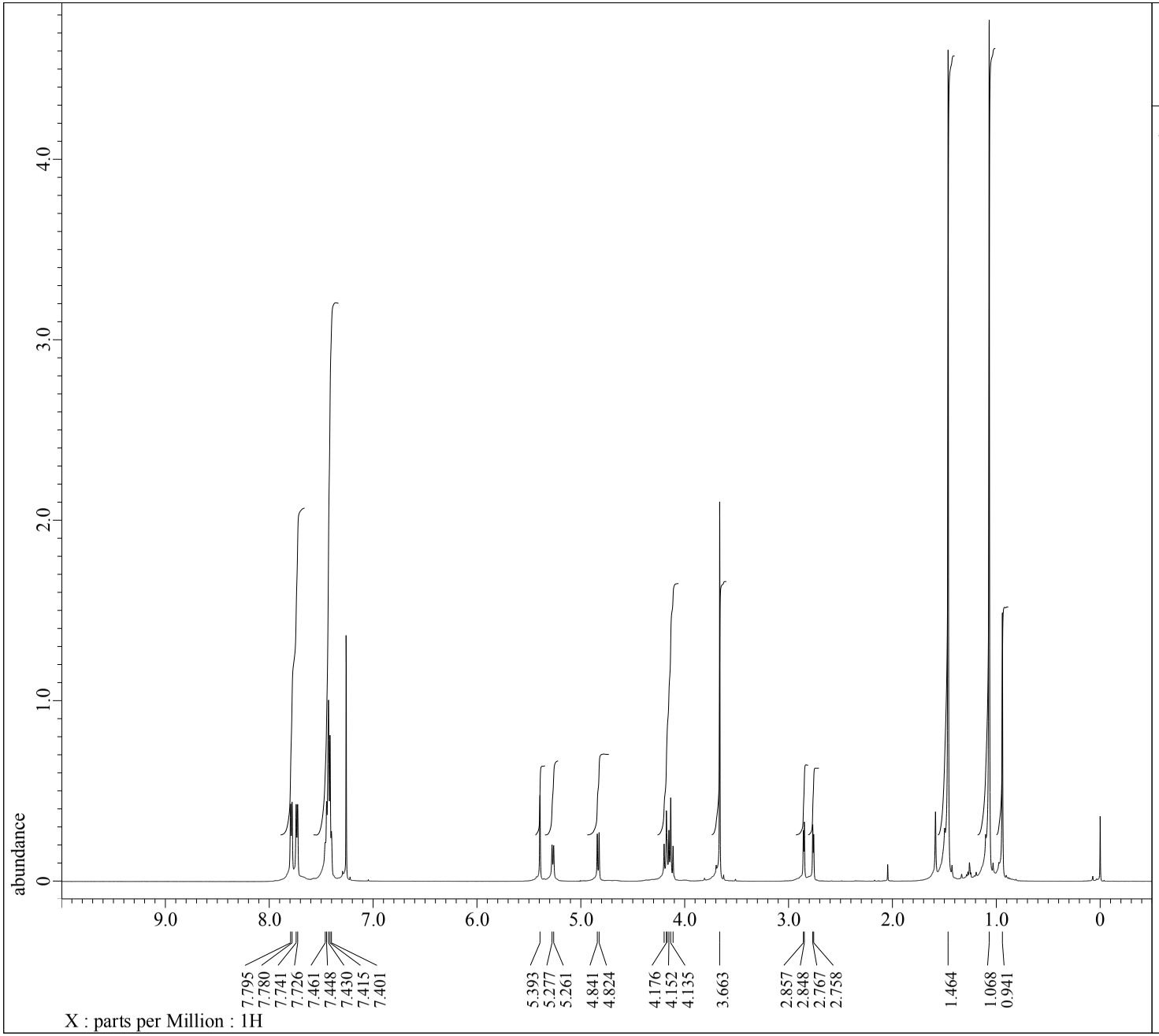


JEOL
Solutions for Innovation

```
Filename          = 170221-2 ketone C-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#800445
Solvent           = CHLOROFORM-D
Creation_Time     = 21-FEB-2017 22:55:20
Revision_Time     =  2-MAR-2017 12:20:39
Current_Time      =  2-MAR-2017 12:25:21

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 0.8388608[s]
X_Domain          = 13C
X_Freq            = 124.5010059[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.1920929[Hz]
X_Sweep           = 39.0625[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1600
Total_Scans       = 1600

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.3[dC]
X_90_Width        = 15.6[us]
X_Acq_Time        = 0.8388608[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 5.2[us]
Irr_Atn_Dec       = 23.2[dB]
Irr_Atn_Noe       = 23.2[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.8388608[s]
```

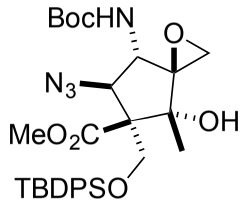


22

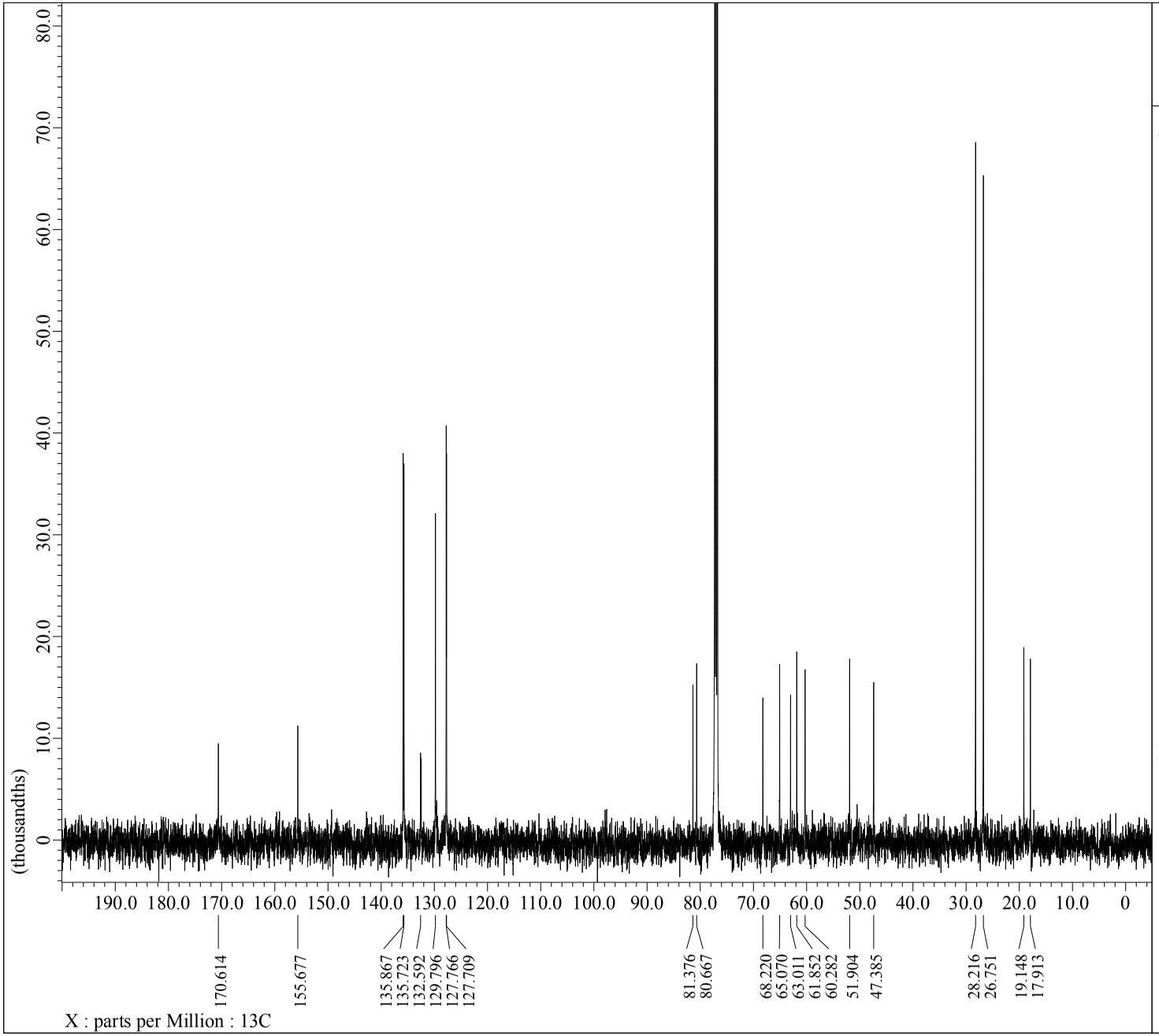


JEOL
Solutions for Innovation

```
Filename          = 170222-5 methyl-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#612463
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2017 16:27:17
Revision_Time     =  4-MAR-2017 15:09:21
Current_Time      =  4-MAR-2017 15:10:01

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 16
Total_Scans       = 16

Relaxation_Delay = 5[s]
Recvr_Gain        = 38
Temp_Get          = 21.7[dC]
X_90_Width        = 8.16[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```

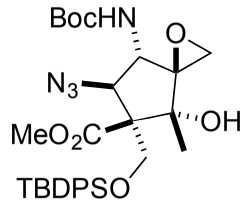


23

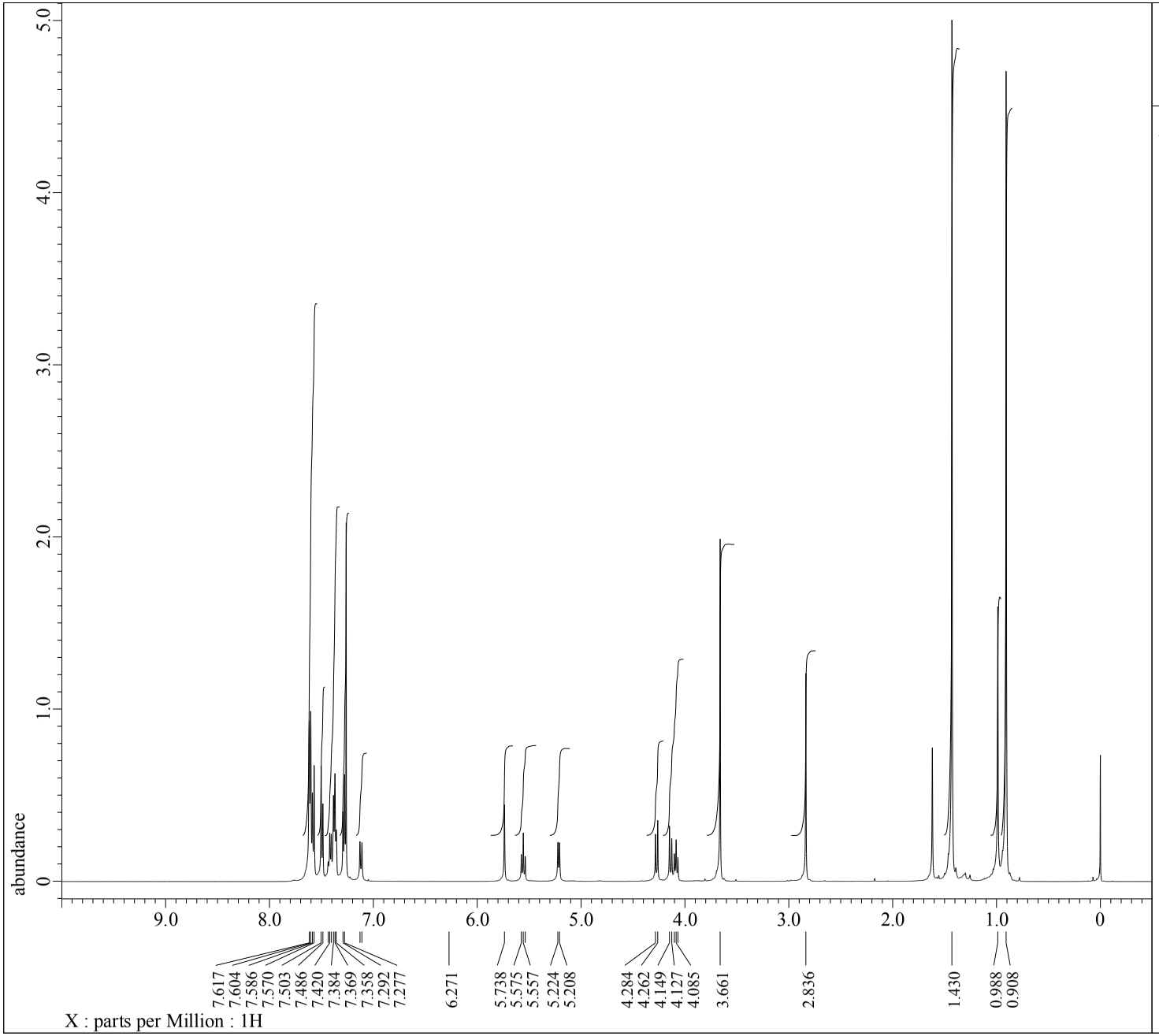


JEOL
Solutions for Innovation

```
Filename          = 170222-6 methyl C-5.jdf
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = S#640775
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2017 18:29:53
Revision_Time     =  4-MAR-2017 15:16:53
Current_Time      =  4-MAR-2017 15:17:45

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 0.8388608[s]
X_Domain          = 13C
X_Freq            = 124.5010059[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.1920929[Hz]
X_Sweep           = 39.0625[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1600
Total_Scans       = 1600

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.8[dC]
X_90_Width        = 15.6[us]
X_Acq_Time        = 0.8388608[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 5.2[us]
Irr_Atn_Dec       = 23.2[dB]
Irr_Atn_Noe       = 23.2[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.8388608[s]
```

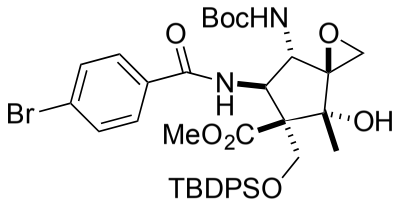


23



JEOL
Solutions for Innovation

```
Filename          = 170222-7 clystal-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#856570
Solvent           = CHLOROFORM-D
Creation_Time     = 22-FEB-2017 23:16:55
Revision_Time     =  7-MAR-2017 15:46:37
Current_Time      =  7-MAR-2017 15:47:06

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 32
Total_Scans       = 32

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 21.3[dC]
X_90_Width        = 8.16[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```



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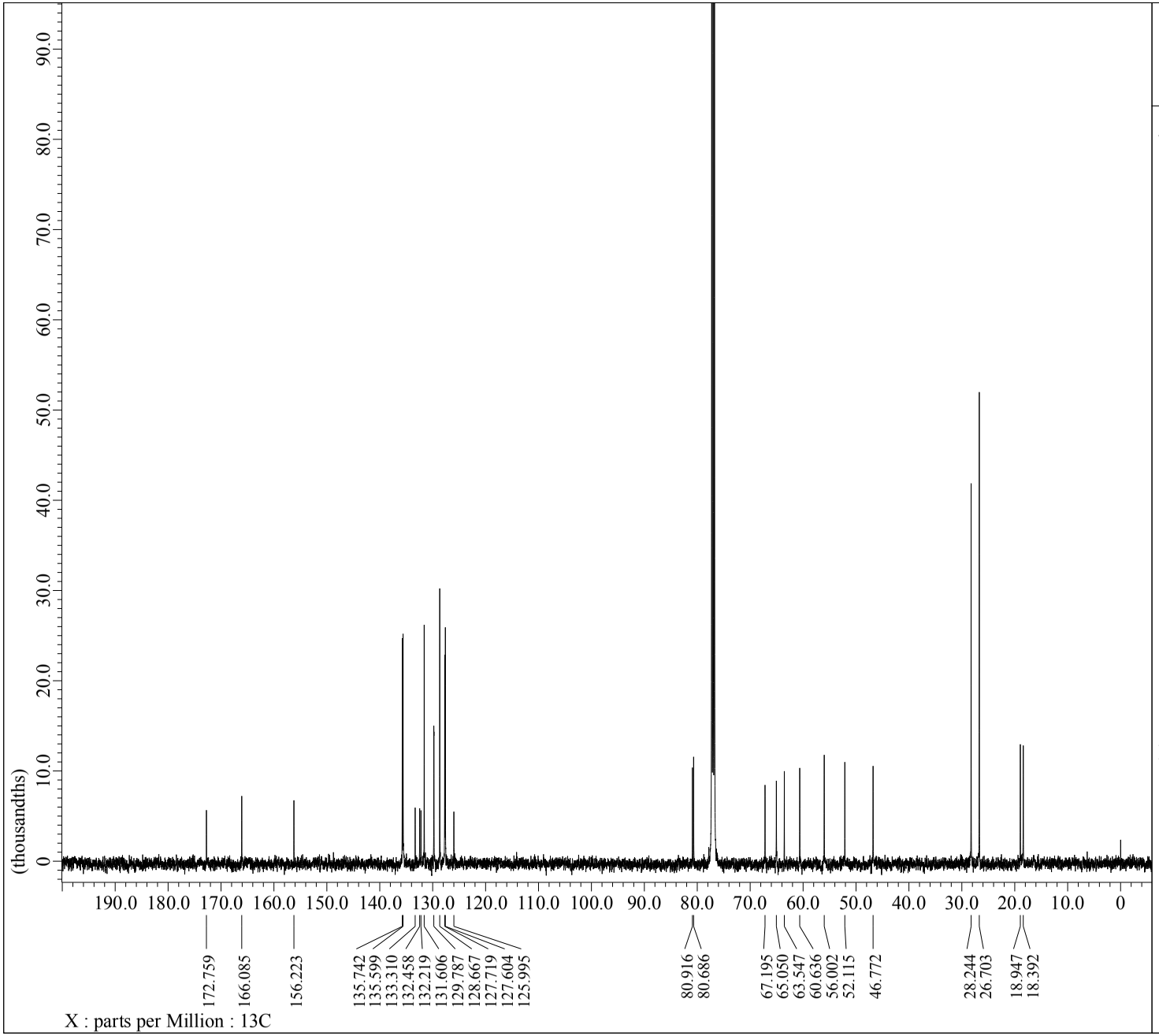
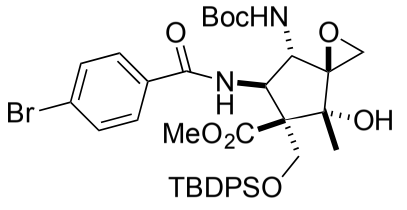
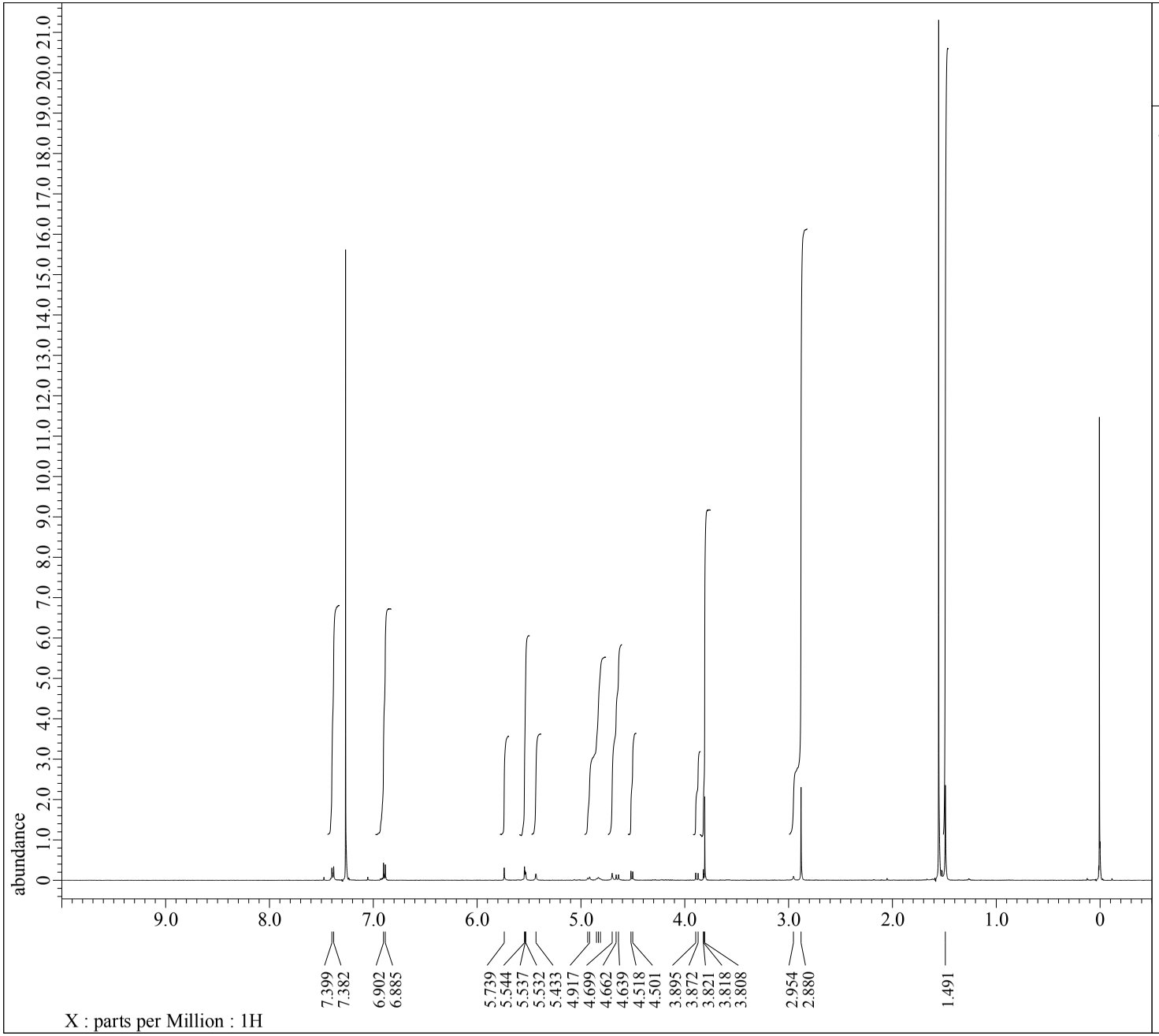


JEOL
Solutions for Innovation

Filename	= 170222-8 clystal C-5.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#863482
Solvent	= CHLOROFORM-D
Creation_Time	= 23-FEB-2017 08:53:42
Revision_Time	= 16-MAR-2017 17:44:03
Current_Time	= 16-MAR-2017 17:44:38
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 12000
Total_Scans	= 12000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 21.3[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 23.2[dB]
Irr_Atn_Noe	= 23.2[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]

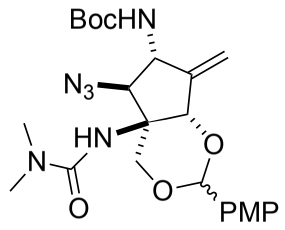


24



JEOL
Solutions for Innovation

Filename	= AG-141122-1-4.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= S#583354
Solvent	= CHLOROFORM-D
Creation_Time	= 22-NOV-2014 16:01:55
Revision_Time	= 22-FEB-2017 17:08:31
Current_Time	= 22-FEB-2017 17:08:57
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 1.76422912[s]
X_Domain	= 1H
X_Freq	= 495.13191398[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.5668198[Hz]
X_Sweep	= 9.28677563[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 495.13191398[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 56
Temp_Get	= 24.5[dC]
X_90_Width	= 8.16[us]
X_Acq_Time	= 1.76422912[s]
X_Angle	= 45[deg]
X_Atn	= 4[dB]
X_Pulse	= 4.08[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.76422912[s]



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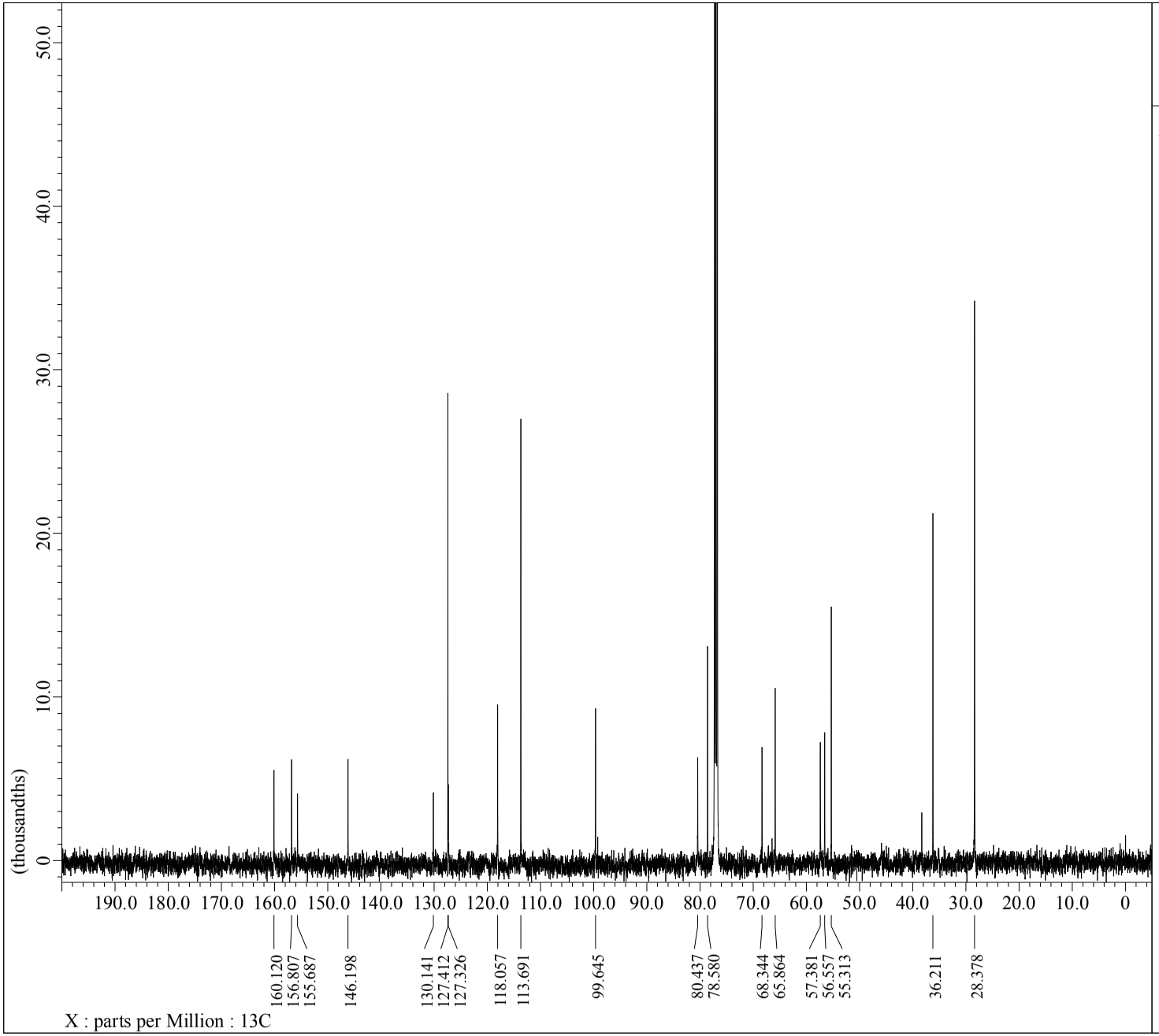
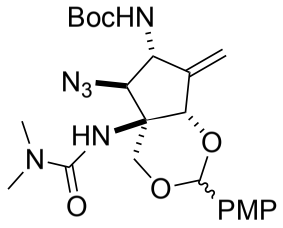


JEOL
Solutions for Innovation

Filename	= AG-151221-5-6.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= ./N/Goto/AG-151222-1
Solvent	= CHLOROFORM-D
Creation_Time	= 22-DEC-2015 07:21:20
Revision_Time	= 22-FEB-2017 17:11:00
Current_Time	= 22-FEB-2017 17:11:39
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8000
Total_Scans	= 8000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 48
Temp_Get	= 25.1[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.042[dB]
Irr_Atn_Noe	= 25.042[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]



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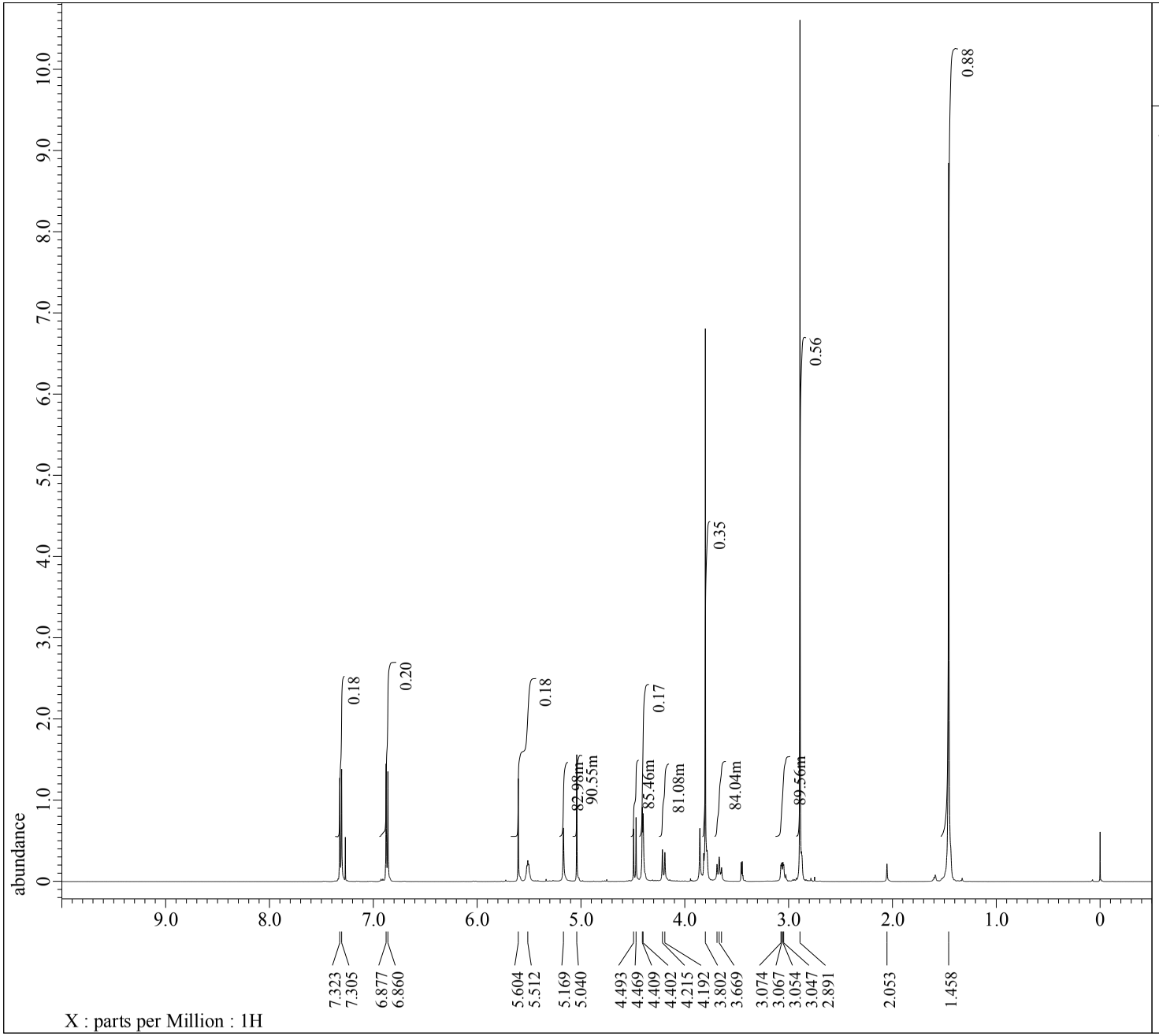
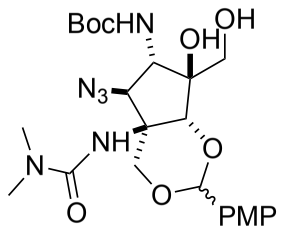



```
Filename          = AG-151221-1-5.jdf
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = S#740384
Solvent           = CHLOROFORM-D
Creation_Time     = 21-DEC-2015 20:10:52
Revision_Time     = 22-FEB-2017 23:54:55
Current_Time      = 22-FEB-2017 23:55:32

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECA 500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.62926421[T] (500[MHz])
X_Acq_Duration    = 1.76422912[s]
X_Domain          = 1H
X_Freq            = 495.13191398[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.5668198[Hz]
X_Sweep           = 9.28677563[kHz]
Irr_Domain        = 1H
Irr_Freq          = 495.13191398[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = 1H
Tri_Freq          = 495.13191398[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 25[dC]
X_90_Width        = 8.16[us]
X_Acq_Time        = 1.76422912[s]
X_Angle           = 45[deg]
X_Atn             = 4[dB]
X_Pulse           = 4.08[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.76422912[s]
```



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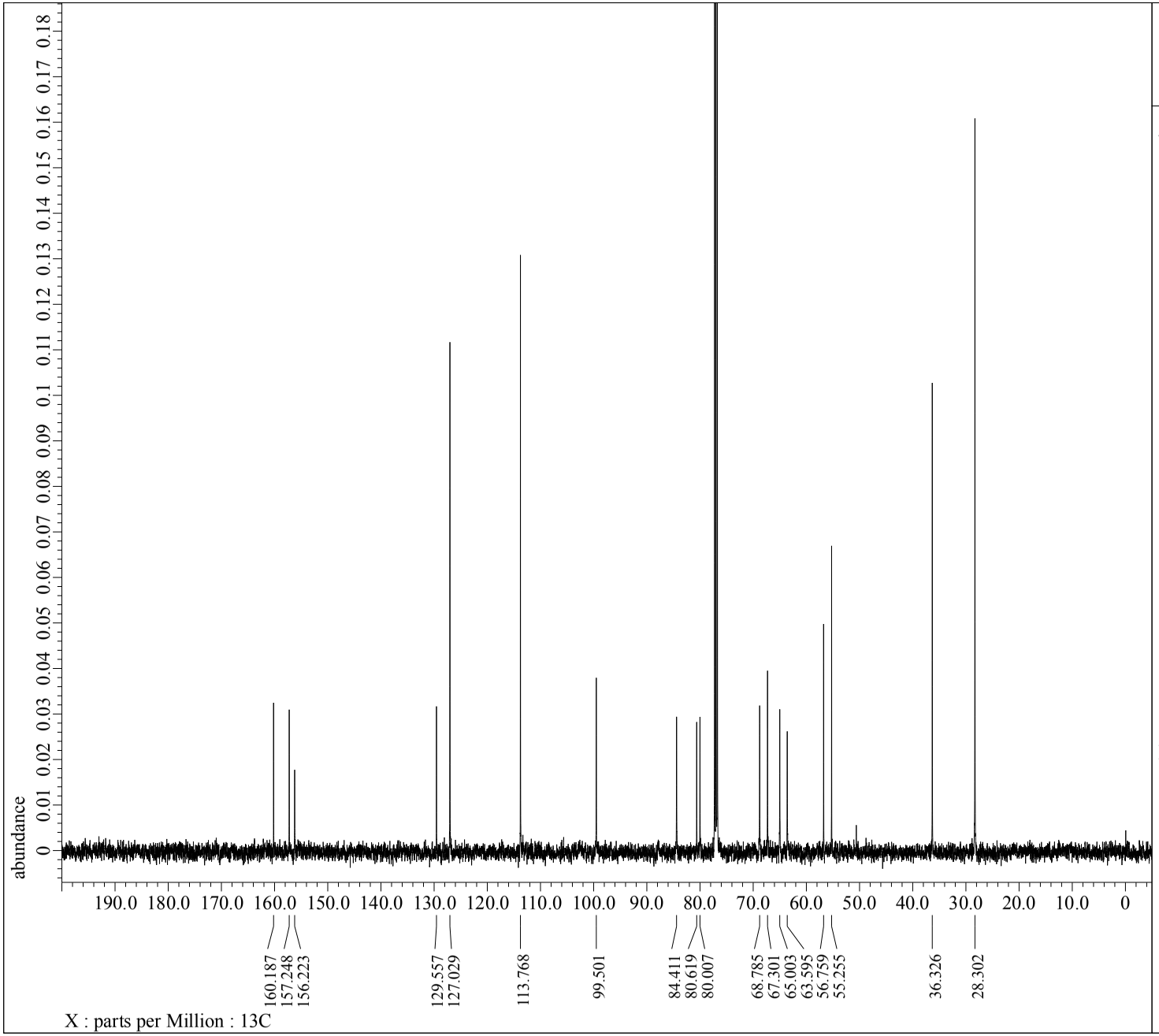
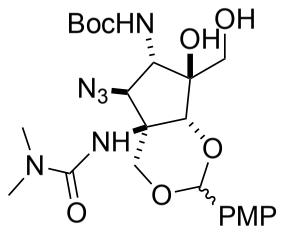


JEOL
Solutions for Innovation

Filename	= AG-151221-2-5.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= S#742515
Solvent	= CHLOROFORM-D
Creation_Time	= 21-DEC-2015 21:01:08
Revision_Time	= 22-FEB-2017 23:57:11
Current_Time	= 22-FEB-2017 23:58:00
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECA 500
Spectrometer	= DELTA2_NMR
Field_Strength	= 11.62926421[T] (500[MHz])
X_Acq_Duration	= 0.8388608[s]
X_Domain	= 13C
X_Freq	= 124.5010059[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.1920929[Hz]
X_Sweep	= 39.0625[kHz]
Irr_Domain	= 1H
Irr_Freq	= 495.13191398[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 1000
Total_Scans	= 1000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 48
Temp_Get	= 25.2[dC]
X_90_Width	= 15.6[us]
X_Acq_Time	= 0.8388608[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 5.2[us]
Irr_Atn_Dec	= 25.042[dB]
Irr_Atn_Noe	= 25.042[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 2.8388608[s]



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