

Supporting Information (Experimental Section)

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

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General Remarks

Nuclear magnetic resonance [^1H NMR (500 MHz), ^{13}C NMR (125 MHz)] or [^1H NMR (400 MHz), ^{13}C NMR (100 MHz)] spectra were determined on JEOL ECA-500SL or JEOL AF-400 instrument. Chemical shifts for ^1H 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.

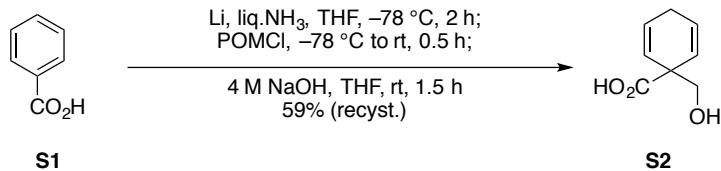
Analytical thin layer chromatography (TLC) was performed on Merck precoated analytical plates, 0.25 mm thick, silica gel 60 F₂₅₄. Preparative TLC separations were made on 7 x 20 cm plates prepared with a 0.25 mm layer of Merck silica gel 60 F₂₅₄. Compounds were eluted from the adsorbent with 10% methanol 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**



To a stirred solution of benzoic acid (**S1**, 20.7 g, 170 mmol) in a 2:1 mixture of liquid NH₃ 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 NH₃. 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 CH₂Cl₂. 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 Na₂SO₄ and concentrated under reduced pressure. The residue was purified by recrystallization (CHCl₃/n-hexane = 2/1) to afford **S2** (15.4 g, 59 %) as a colorless solid.

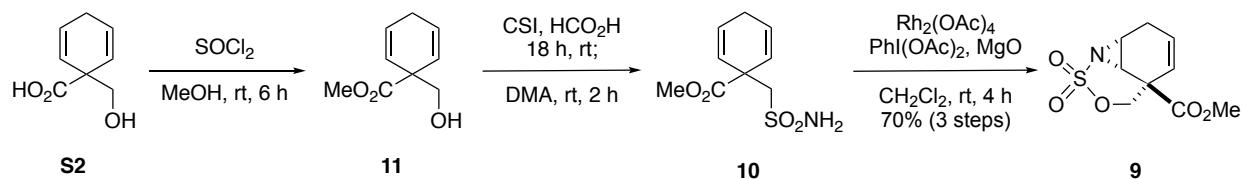
IR (film, cm⁻¹): 3330, 2884, 1695, 1290, 1243, 1225, 1084, 1024.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, δ): 178.8, 128.2, 123.6, 68.5, 50.4, 26.3.

HRMS (ESI): Calcd for C₈H₁₀O₃Na 177.0522 [(M+Na)⁺], found 177.0526.

Aziridine 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), $\text{PhI}(\text{OAc})_2$ (29 g, 89 mmol) and $\text{Rh}_2(\text{OAc})_4$ (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.

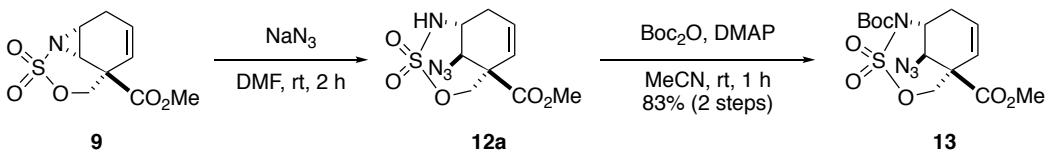
IR (film, cm^{-1}): 3038, 2959, 1740, 1375, 1260, 1184, 1018.

^1H NMR (500 MHz, CDCl_3 , δ): 6.07 (ddd, $J = 9.9, 5.1, 2.3$ Hz, 1H), 5.80–5.78 (m, 1H), 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 $\text{C}_9\text{H}_{11}\text{NO}_5\text{SNa}$ 268.0250 $[(\text{M}+\text{Na})^+]$, 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.

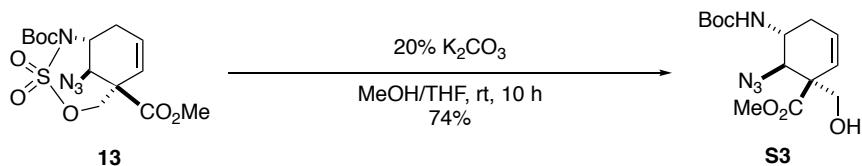
IR (film, cm^{-1}): 2984, 2110, 1740, 1728, 1387, 1300, 1186, 1070.

^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 (dd, 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 $\text{C}_{14}\text{H}_{20}\text{N}_4\text{O}_7\text{SNa}$ 411.0945 [$(\text{M}+\text{Na})^+$], found 411.0943.

Alcohol **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.

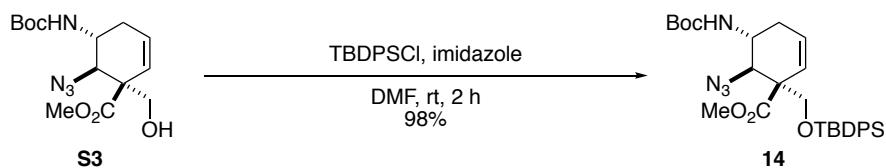
IR (film, cm^{-1}): 3370, 2980, 2108, 1721, 1701, 1525, 1437, 1368, 1306, 1244, 1171, 1020.

^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 $\text{C}_{14}\text{H}_{22}\text{N}_4\text{O}_5\text{Na}$ 349.1482 [(M+Na) $^+$], found 349.1486.

TBDPS ether **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 NH₄Cl at 0 °C and extracted with Et₂O. The organic layer was washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography (SiO₂, *n*-hexane/EtOAc = 20/1 to 1/1) to afford **14** (7.00 g, 98%) as a colorless amorphous.

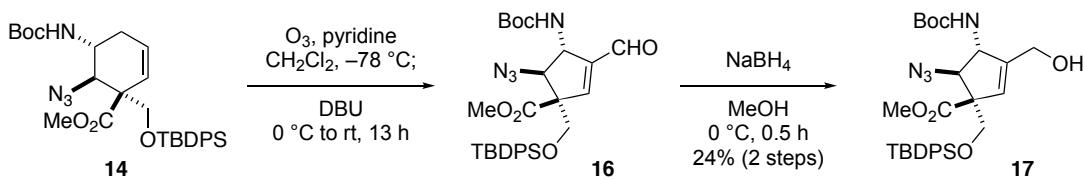
IR (film, cm⁻¹): 3374, 3265, 2955, 2887, 2104, 1732, 1706, 1503, 1366, 1244, 1167, 1113.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, 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 C₃₀H₄₀N₄O₅SiNa 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 CH₂Cl₂ (53 mL) was stirred under O₃ 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 NH₄Cl at 0 °C and extracted with Et₂O. The organic layer was washed with brine, dried over anhydrous Na₂SO₄ 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 NaBH₄ (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 NH₄Cl at the same temperature and extracted with EtOAc. The organic layer was washed with H₂O and brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography (SiO₂, *n*-hexane/EtOAc = 5/1 to 1/1) to afford **17** (1.50 g, 24%, 2 steps) as a colorless amorphous.

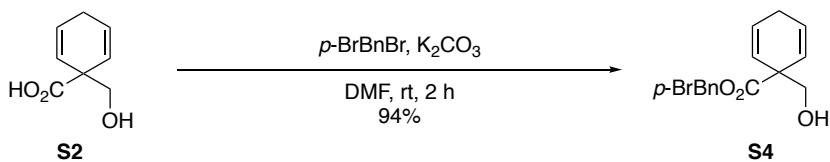
IR (film, cm^{-1}): 3408, 3368, 2932, 2859, 2108, 1719, 1508, 1368, 1252, 1165, 1115.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, 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.

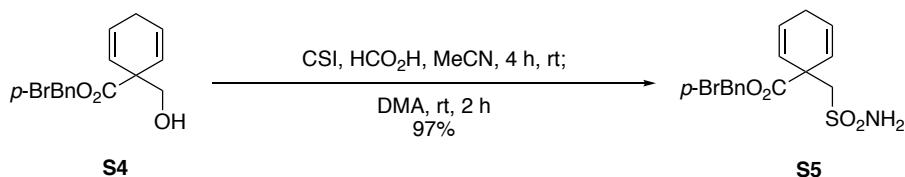
IR (film, cm^{-1}): 3458, 3034, 2947, 2878, 1724, 1489, 1452, 1408, 1371, 1271, 1229, 1200, 1070.

^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 $\text{C}_{15}\text{H}_{15}\text{O}_3\text{BrNa}$ 354.0102 [(M+Na) $^+$], found 345.0097.

Sulfamate **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.

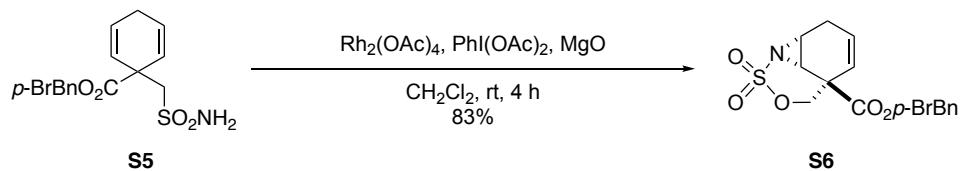
IR (film, cm^{-1}): 3370, 3291, 1724, 1375, 1221, 1186.

^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 $\text{C}_{15}\text{H}_{16}\text{O}_5\text{NSBrNa}$ 423.9830 [$(\text{M}+\text{Na})^+$], found 423.9825.

Aziridine **S6**

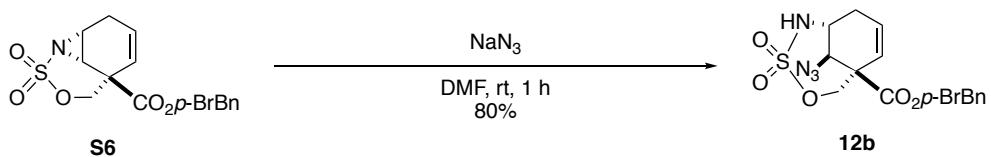


To a stirred solution of **S5** (3.00 g, 7.46 mmol) in CH_2Cl_2 (750 mL) were added MgO (680 mg, 16.9 mmol), $\text{PhI}(\text{OAc})_2$ (2.71 g, 8.41 mmol) and $\text{Rh}_2(\text{OAc})_4$ (211 mg, 0.477 mmol) at 0 °C. The resulting mixture was stirred at room temperature for 4 hours. Then the reaction 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, 1H), 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 $\text{C}_{15}\text{H}_{14}\text{O}_5\text{NSBrNa}$ 421.9666 [$(\text{M}+\text{Na})^+$], found 421.9668.

Azide **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.

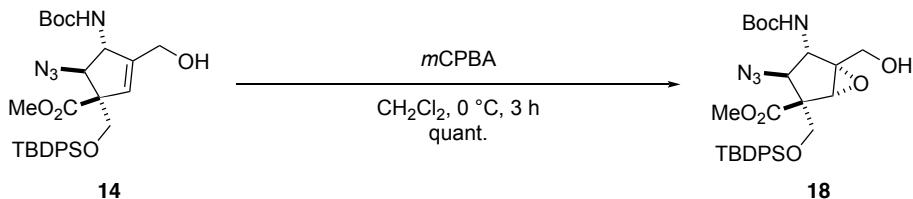
IR (film, cm^{-1}): 3292, 2959, 2930, 2122, 1735, 1458, 1425, 1373, 1253, 1179.

^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 $\text{C}_{15}\text{H}_{15}\text{N}_4\text{O}_5\text{SNa}$ 464.9840 $[(\text{M}+\text{Na})^+]$, found 464.9839.

Epoxyalcohol **18**



To a stirred solution of **14** (794 mg, 1.37 mmol) in CH₂Cl₂ (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 Na₂S₂O₃ and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography (SiO₂, *n*-hexane/EtOAc = 7/1 to 2/1) to afford **18** (823 mg, quant.) as a colorless amorphous.

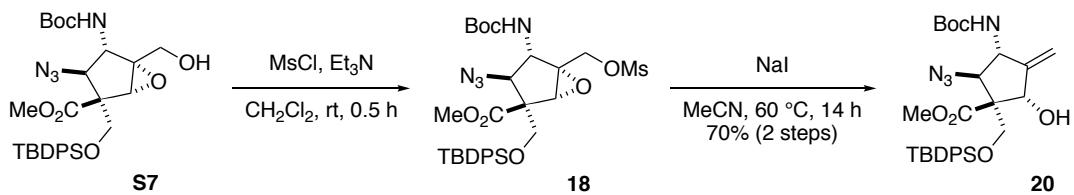
IR (film, cm⁻¹): 3358, 3072, 3049, 2954, 2927, 2856, 2108, 1724, 1699, 1521, 1429, 1367, 1244, 1165, 1112.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, 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 C₃₀H₄₀N₄O₇SiNa 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 °C. 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 °C for 14 hours. Then the reaction mixture was quenched with saturated aqueous $\text{Na}_2\text{S}_2\text{O}_3$ 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.

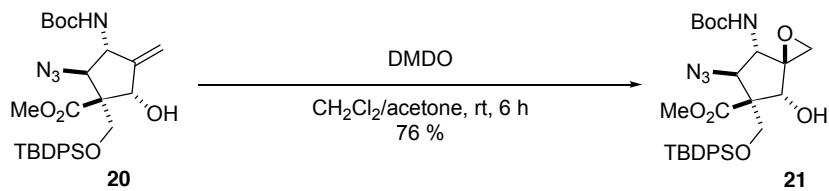
IR (film, cm^{-1}): 3508, 3369, 3072, 3051, 2954, 2931, 2891, 2858, 2106, 1724, 1506, 1429, 1367, 1251, 1165, 1112.

^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 $\text{C}_{30}\text{H}_{40}\text{N}_4\text{O}_6\text{SiNa}$ 603.2609 $[(\text{M}+\text{Na})^+]$, found 603.2607.

Epoxide **21**



To a stirred solution of **20** (146 mg, 0.251 mmol) in CH₂Cl₂ (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 (SiO₂, *n*-hexane/EtOAc = 9/1 to 5/2) to afford **21** (114 mg, 76%) as a colorless amorphous.

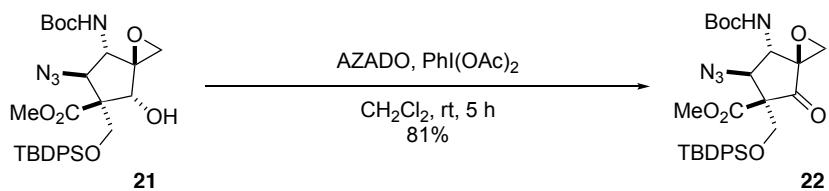
IR (film, cm⁻¹): 3374, 2952, 2107, 1724, 1522, 1428, 1368, 1251, 1164, 1114.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, 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 C₃₀H₄₀N₄O₇SiNa 619.2558 [(M+Na)]⁺, found 619.2547.

Ketone 22



To a stirred solution of **21** (81.0 mg, 0.136 mmol) in CH₂Cl₂ (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 Na₂S₂O₃ and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography (SiO₂, *n*-hexane/EtOAc = 9/1 to 10/3) to afford **22** (65.5 mg, 81%) as a colorless amorphous.

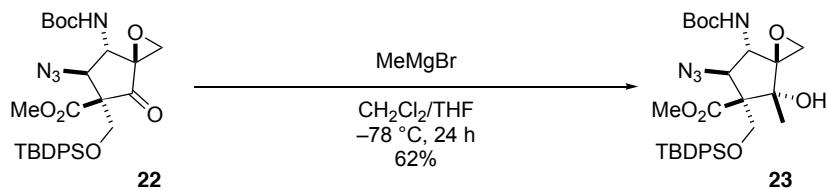
IR (film, cm⁻¹): 3383, 2952, 2872, 2107, 1771, 1702, 1429, 1367, 1252, 1215, 1163, 1114, 1072.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, 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 CH₂Cl₂ (0.5 mL) was added MeMgBr (0.895 mL, 0.98 M in THF, 0.808 mmol) at -78 °C. The resulting mixture was stirred at 24 hours. Then the reaction mixture was poured into saturated aqueous NH₄Cl and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography (SiO₂, *n*-hexane/EtOAc = 20/3 to 3/2) to afford **23** (38.2 mg, 78%, 2 steps) as a colorless amorphous.

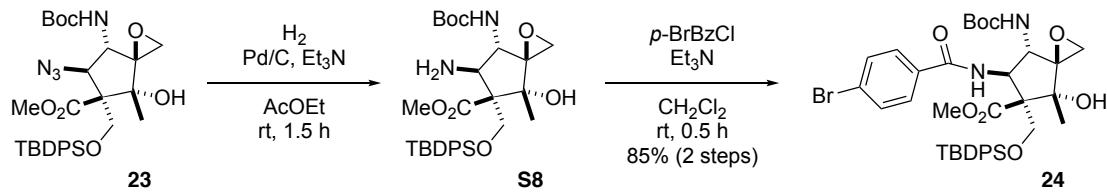
IR (film, cm⁻¹): 3375, 2933, 2858, 2104, 1731, 1684, 1508, 1369, 1278, 1249, 1165, 1114.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, 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 C₃₁H₄₂O₇SiNa 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 CH₂Cl₂ (0.5 mL) were added *p*-bromobenzoyl chloride (*p*-BrBzCl, 7.6 mg, 0.035 mmol) and Et₃N (10 μ L, 0.073 mmol). The resulting mixture was stirred at 0.5 hours. Then the reaction mixture was poured into saturated aqueous NH₄Cl and extracted with EtOAc. The residue was purified by prep. TLC (SiO₂, *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.

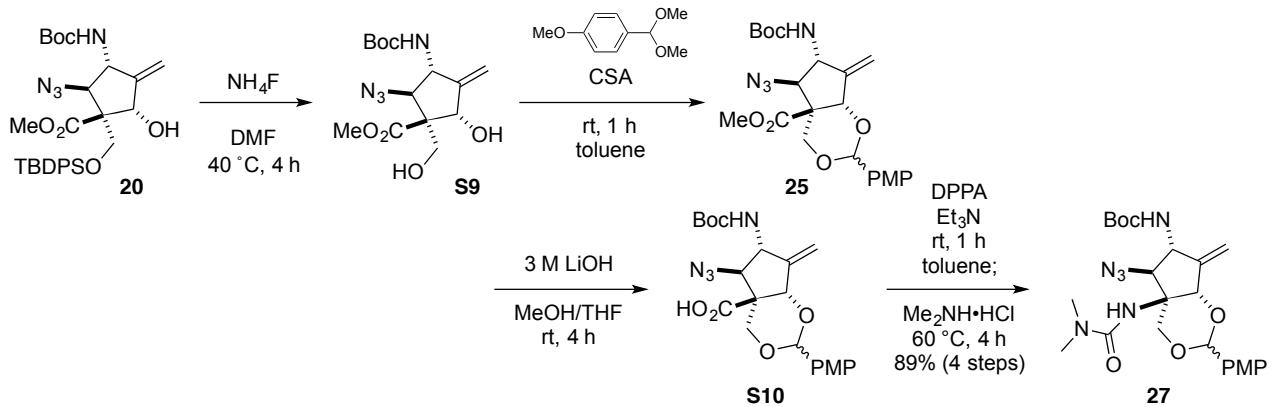
IR (film, cm⁻¹): 3440, 2933, 2858, 1719, 1681, 1523, 1481, 1473, 1368, 1246, 1163, 1114.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, δ): 172.8, 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 C₈H₁₀O₃Na 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 NH₄F (158 mg, 4.28 mmol) at room temperature. The resulting mixture was stirred at 40 °C for 4 hours. Then the reaction mixture was quenched with saturated aqueous NH₄Cl and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na₂SO₄ 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 Et₃N (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 °C. The resulting mixture was stirred at room temperature for 4 hours. Then the reaction mixture was quenched with saturated aqueous NH₄Cl and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na₂SO₄ 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 Et₃N (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 Me₂NH·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 NH₄Cl at 0 °C and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified with column chromatography (SiO₂, *n*-hexane/EtOAc = 2/1 to 1/1) to afford **27** (372 mg, 89%, 4 steps) as a white solid.

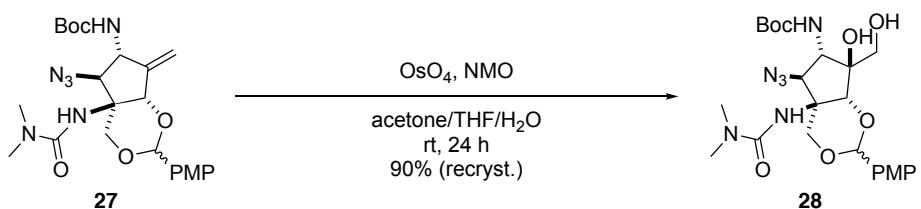
IR (film, cm⁻¹): 2976, 2883, 2380, 2351, 2310, 2110, 1699, 1645, 1633, 1614, 1587, 1516, 1487, 1454, 1435, 1390, 1363, 1247, 1168, 1126, 1089, 1010.

¹H NMR (500 MHz, CDCl₃, 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),

¹³C NMR (125 MHz, CDCl₃, 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 C₂₃H₃₂N₆O₆Na 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 H₂O (total 42 mL) were added *N*-methylmorpholine *N*-oxide (NMO, 833 mg, 7.11 mmol) and OsO₄ (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 Na₂S₂O₃ and extracted with EtOAc. The organic layer was washed with brine, dried over anhydrous Na₂SO₄ 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.

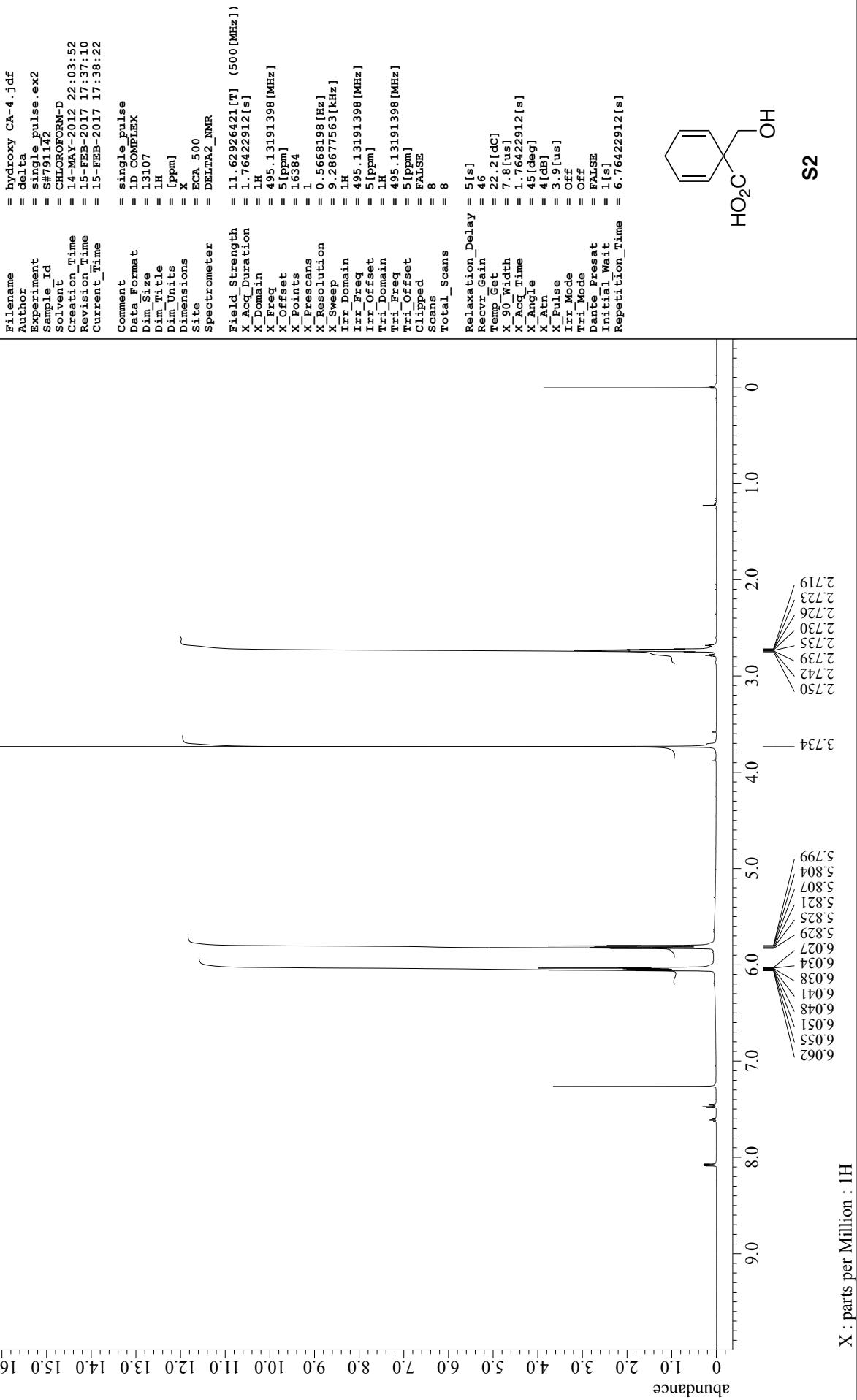
IR (film, cm^{-1}): 3375, 2976, 2935, 2839, 2108, 1691, 1643, 1616, 1517, 1502, 1467, 1367, 1251, 1168, 1031.

¹H NMR (500 MHz, CDCl₃, 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).

¹³C NMR (125 MHz, CDCl₃, 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 C₂₃H₃₄N₆O₈Na 545.2330 [(M+Na)⁺], found 545.2327.

NMR Spectra Data



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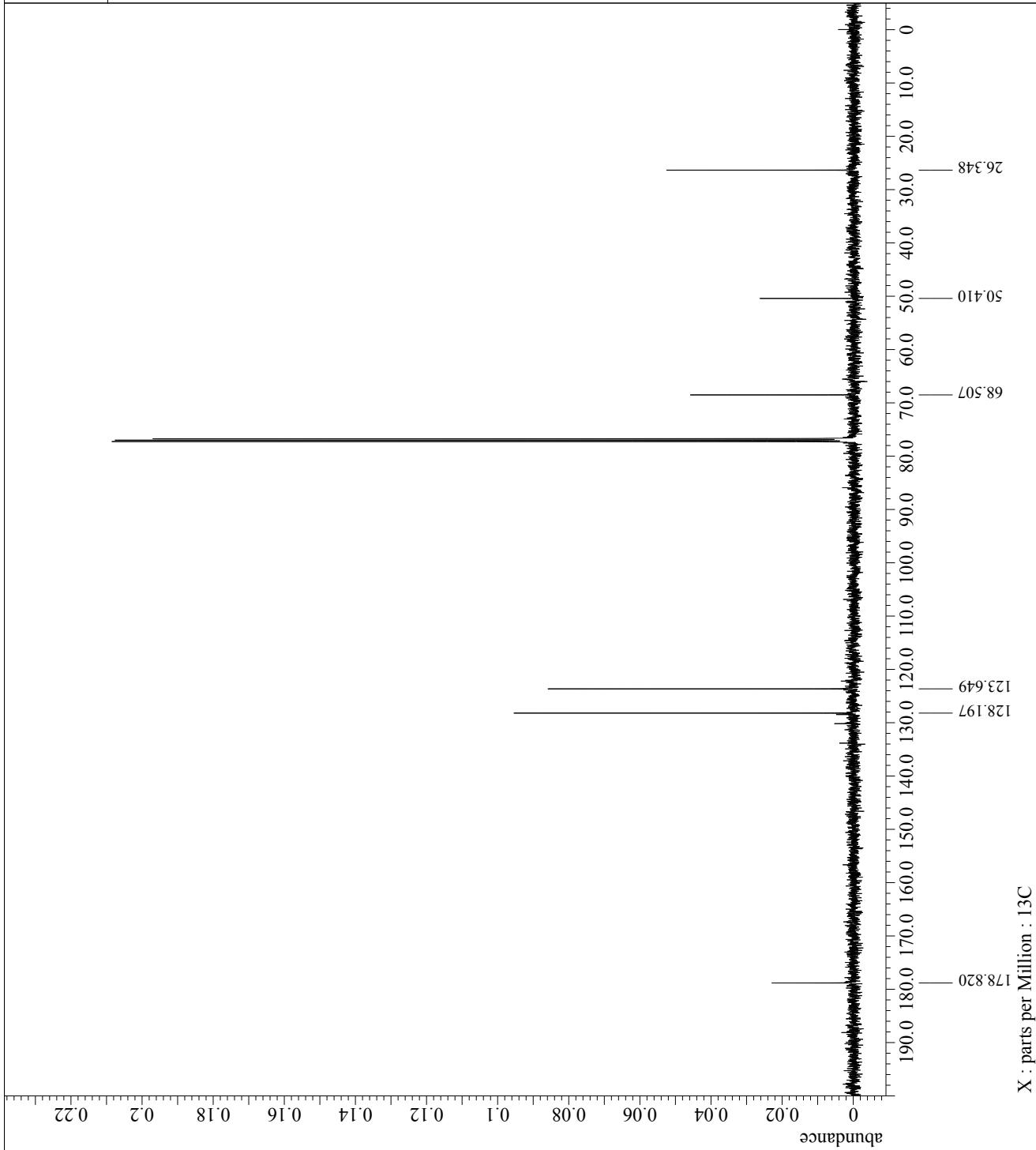
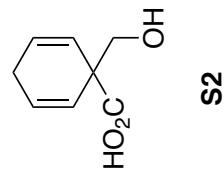
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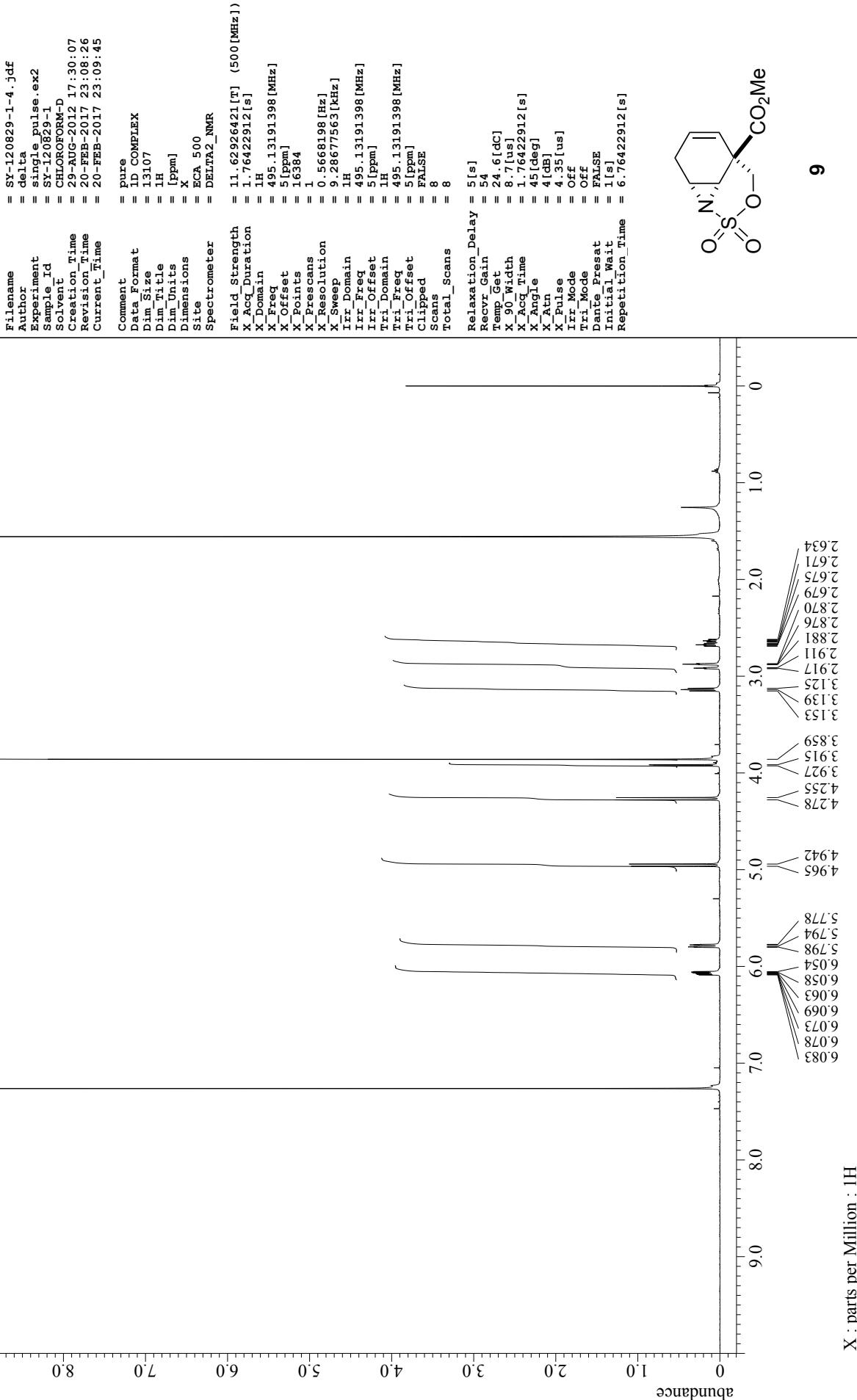
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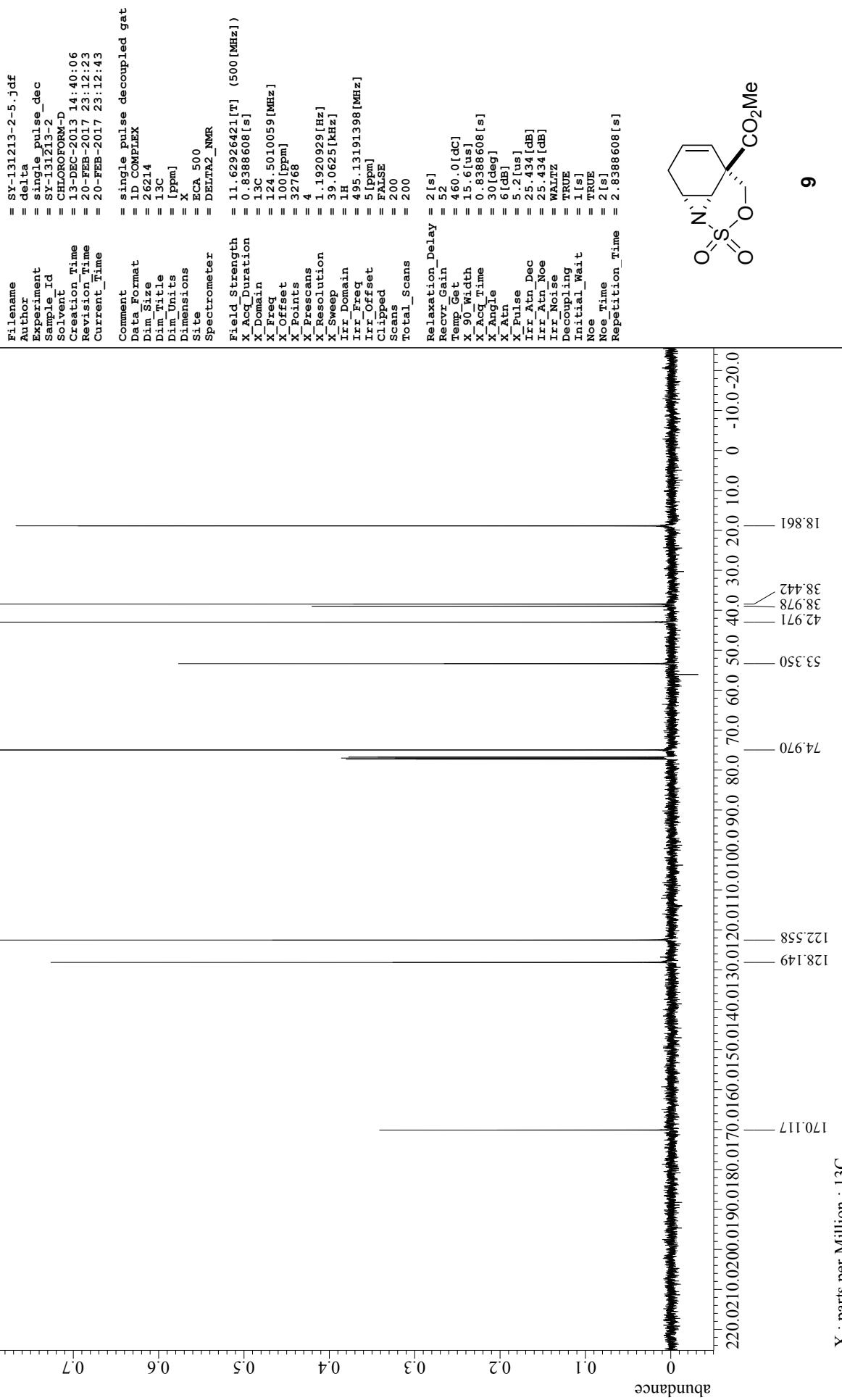
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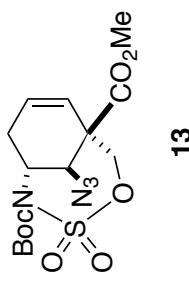
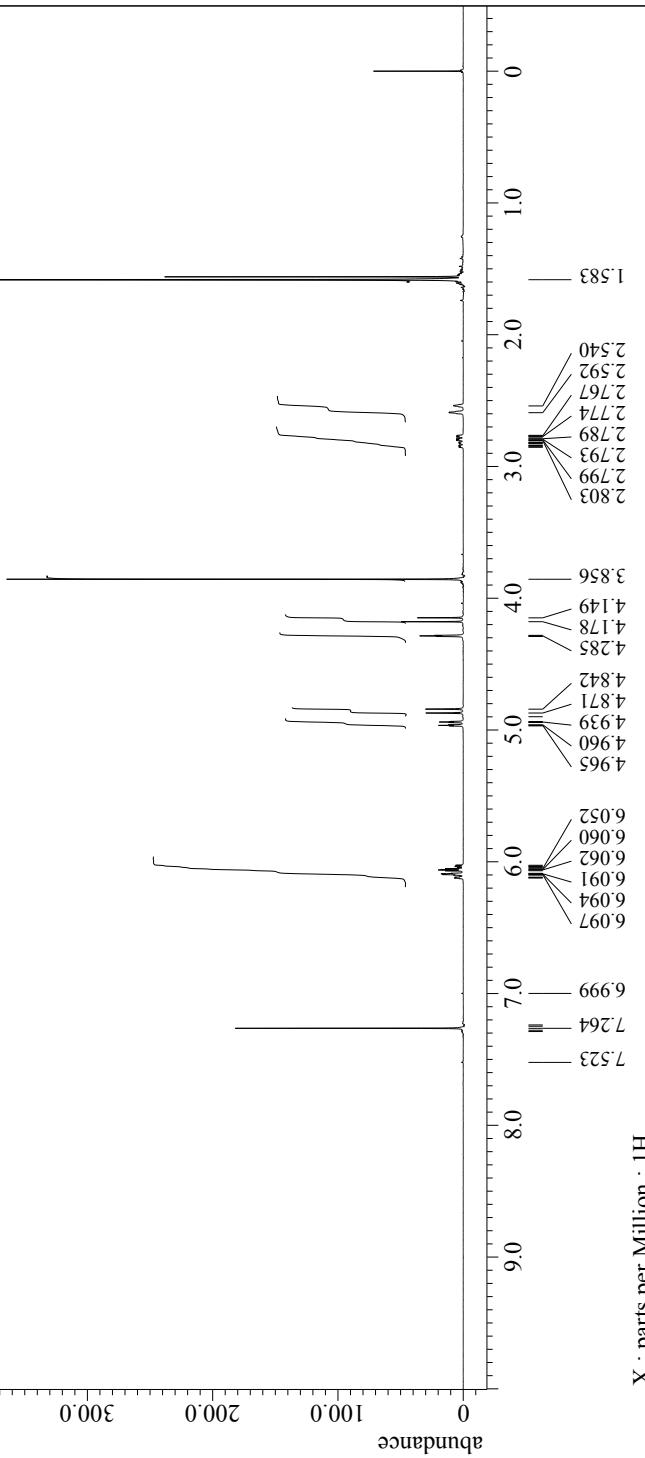
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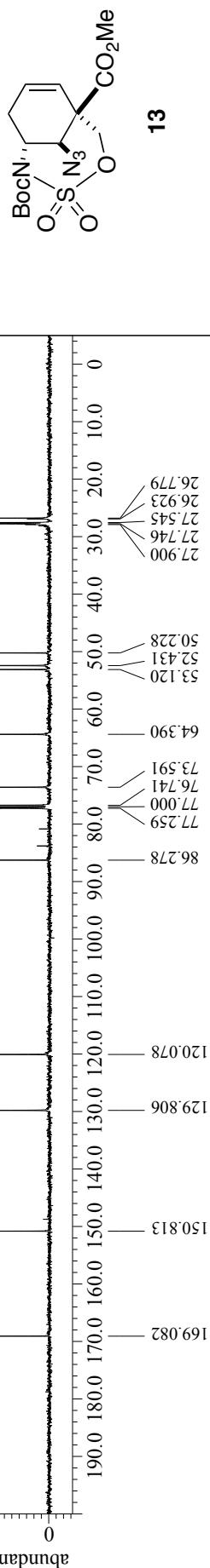
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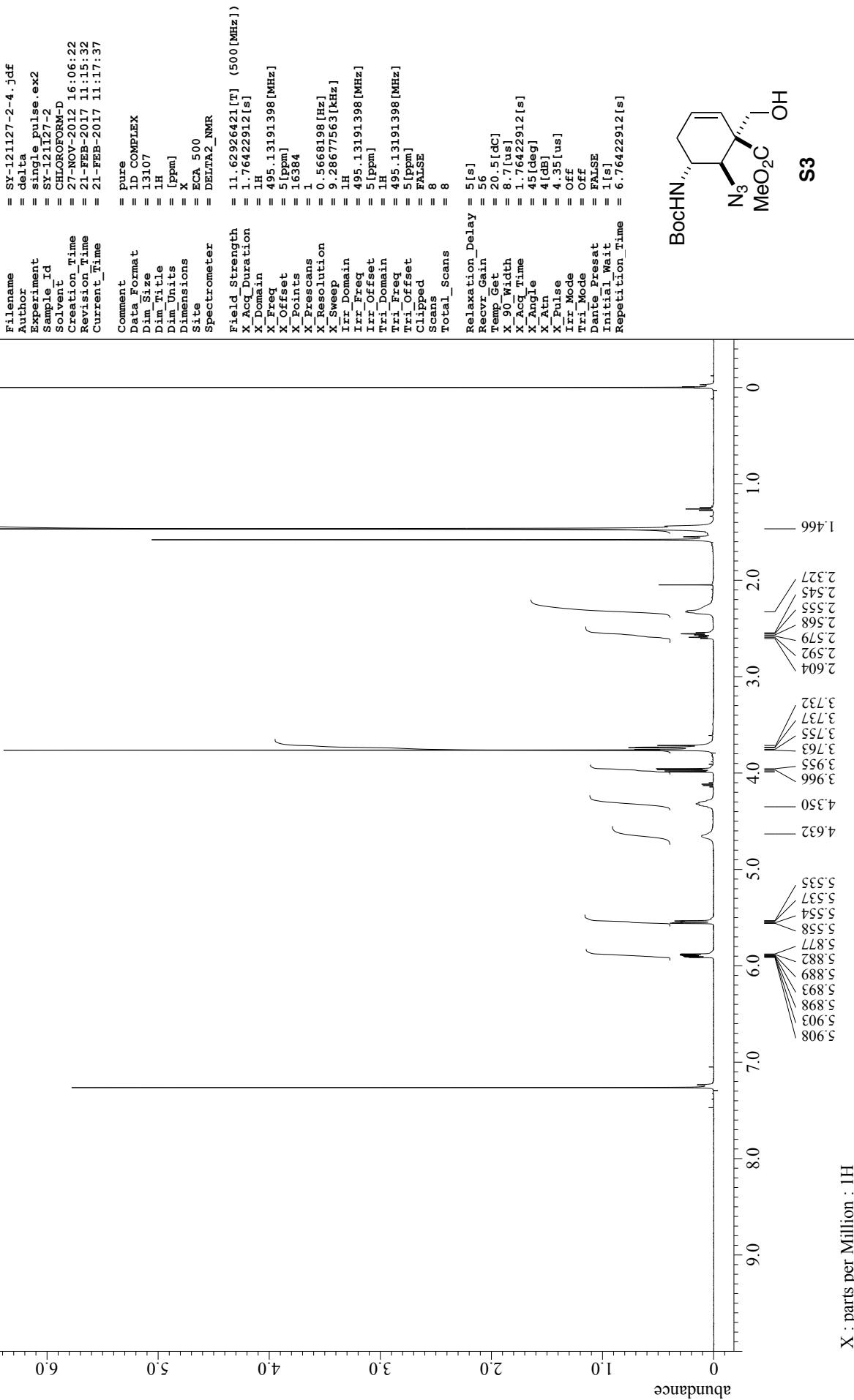
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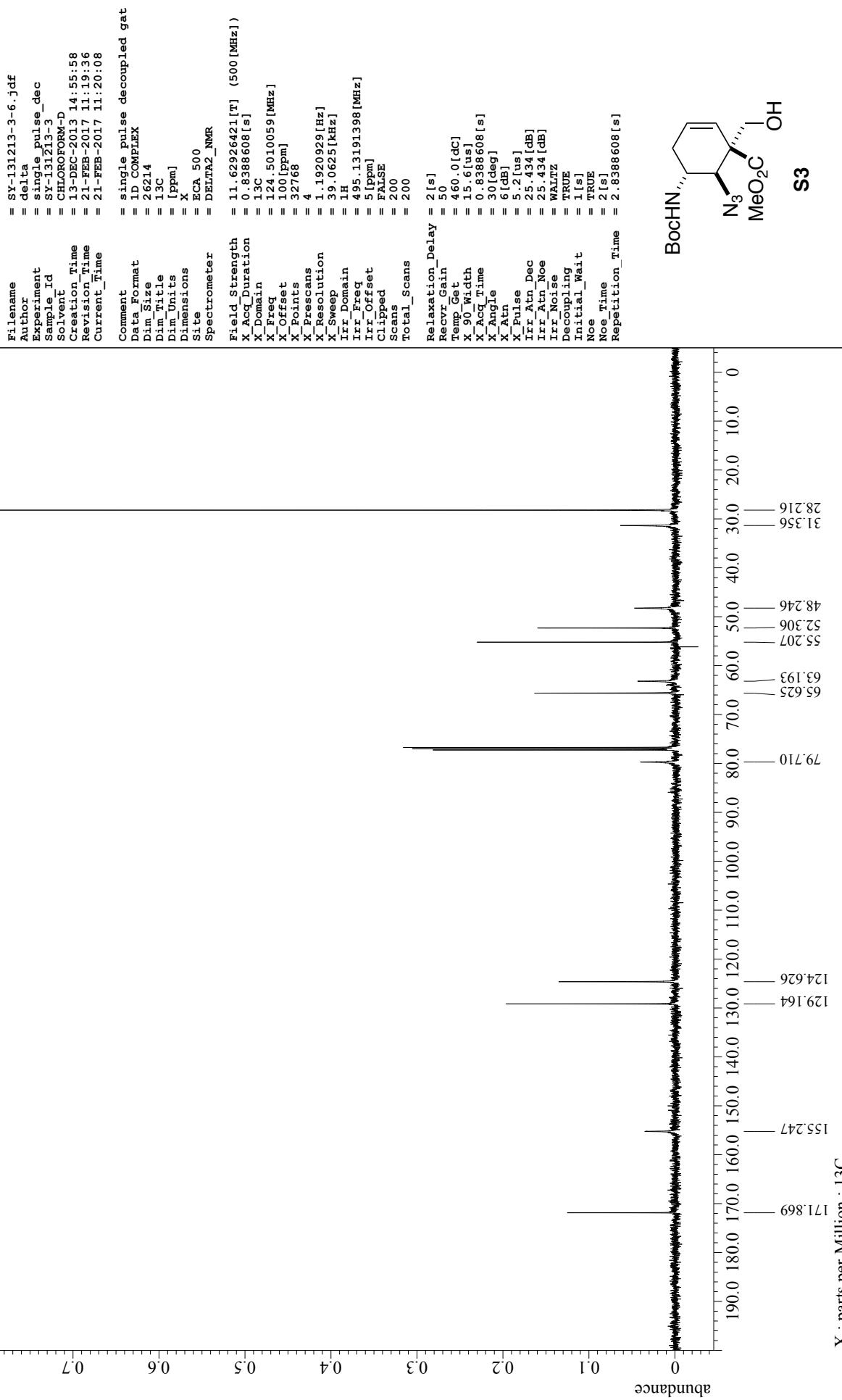
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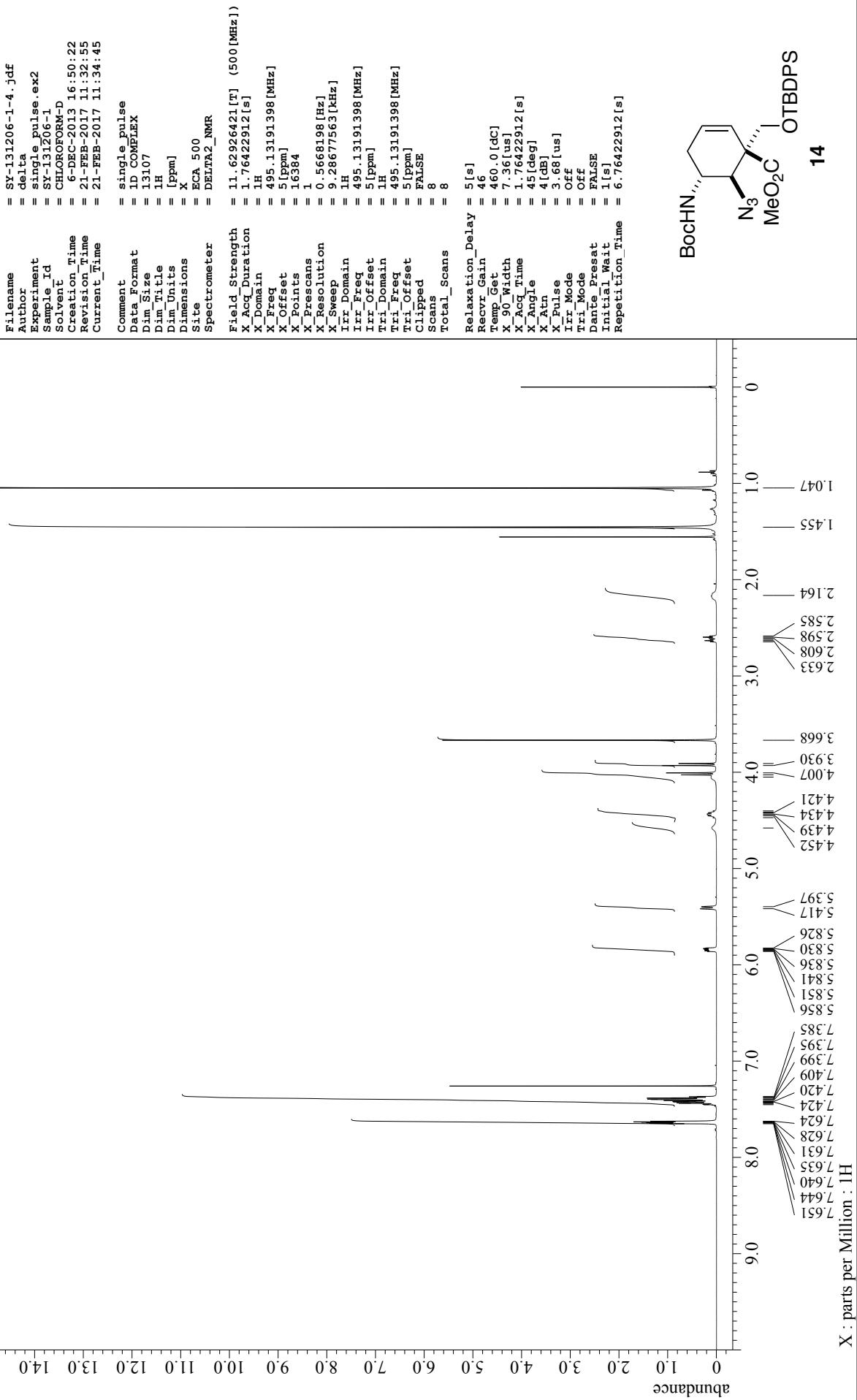
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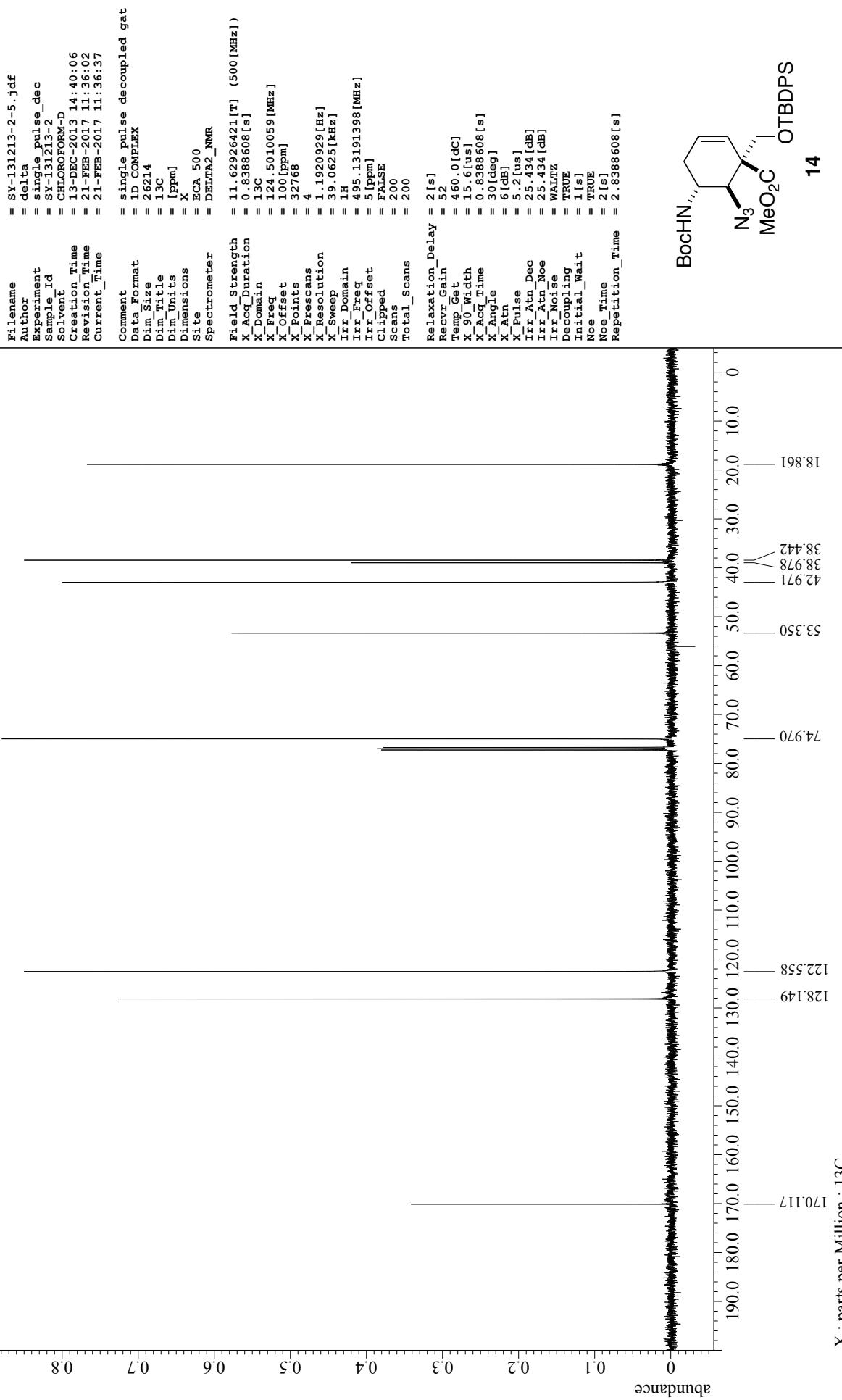
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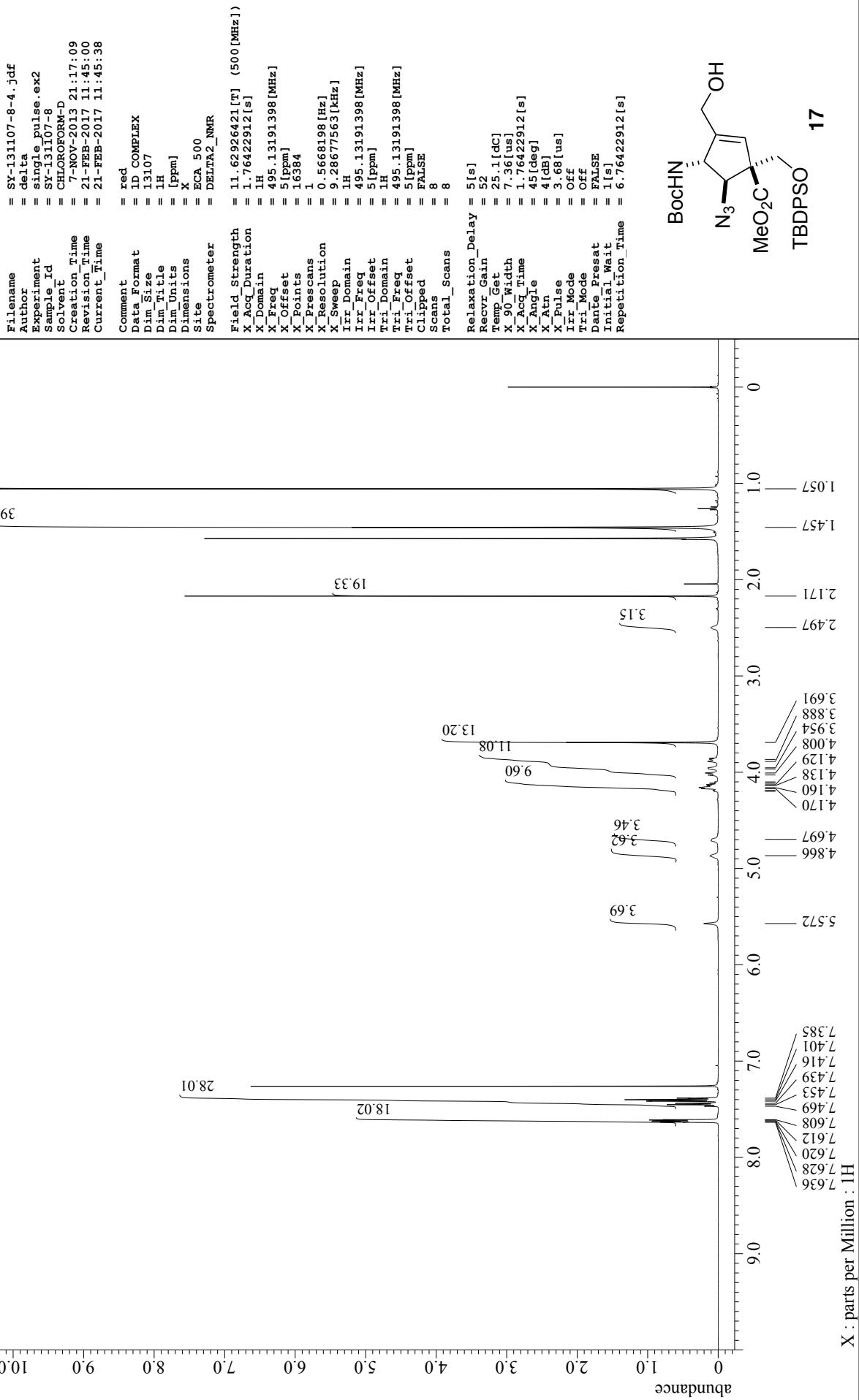


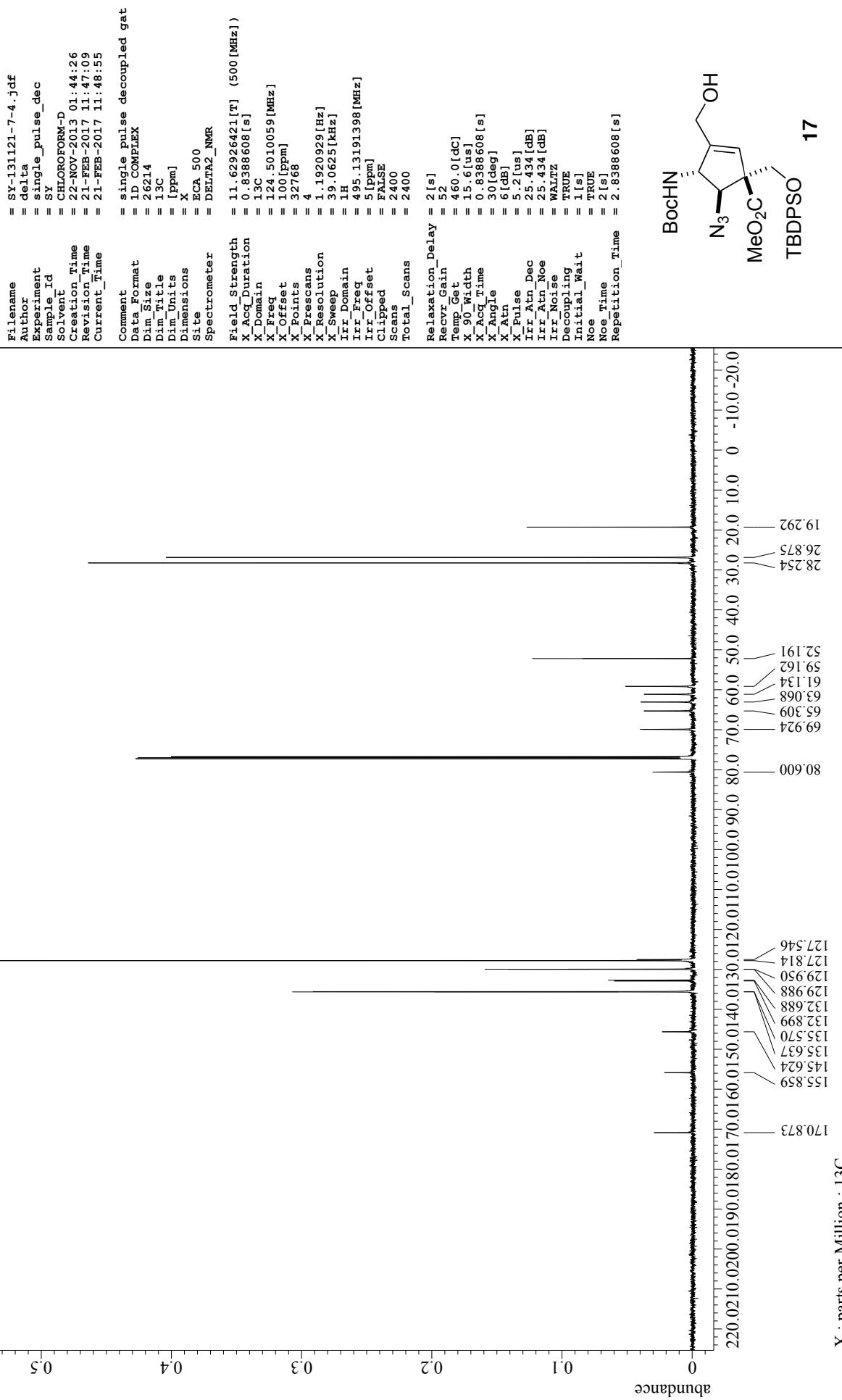










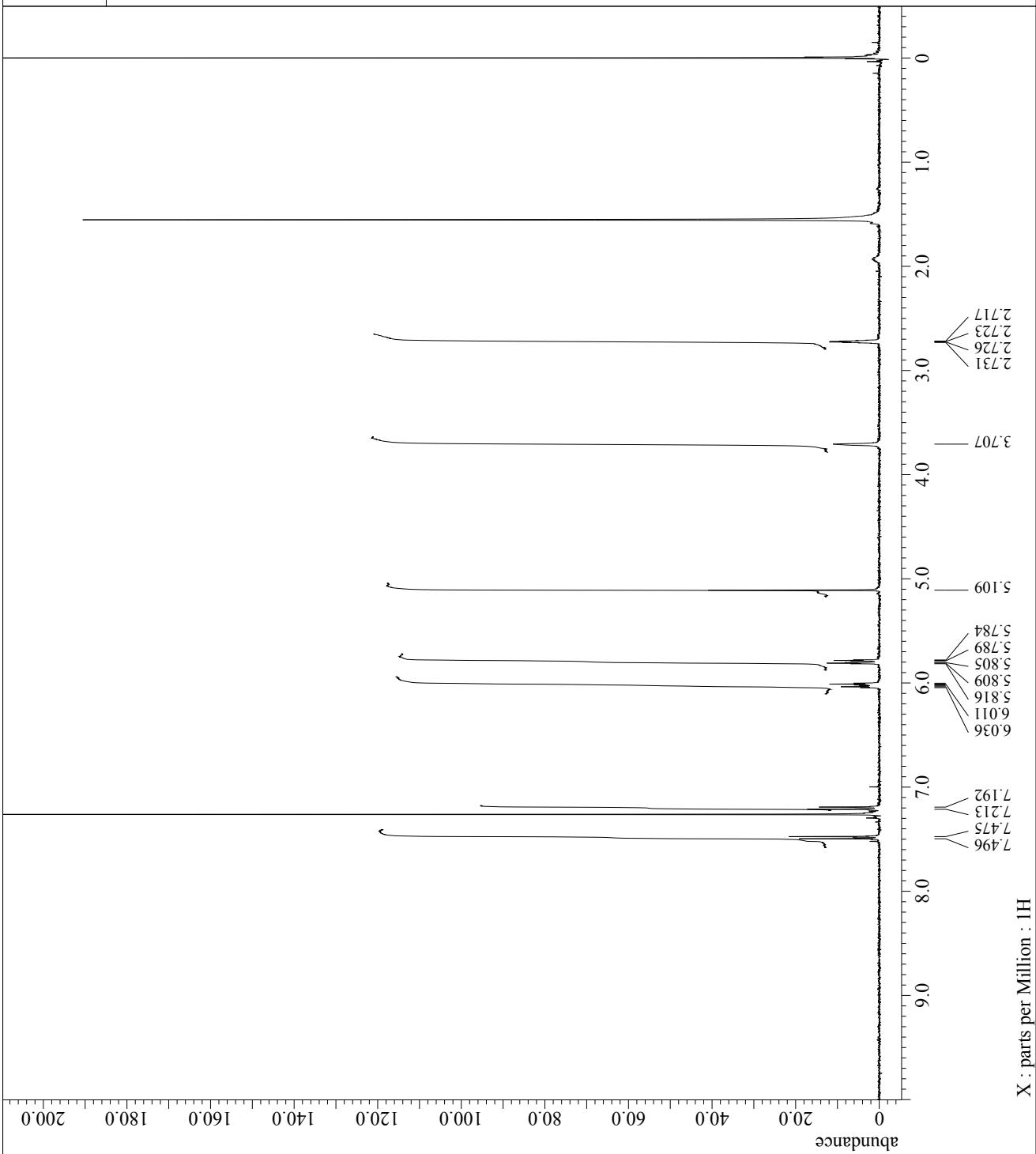


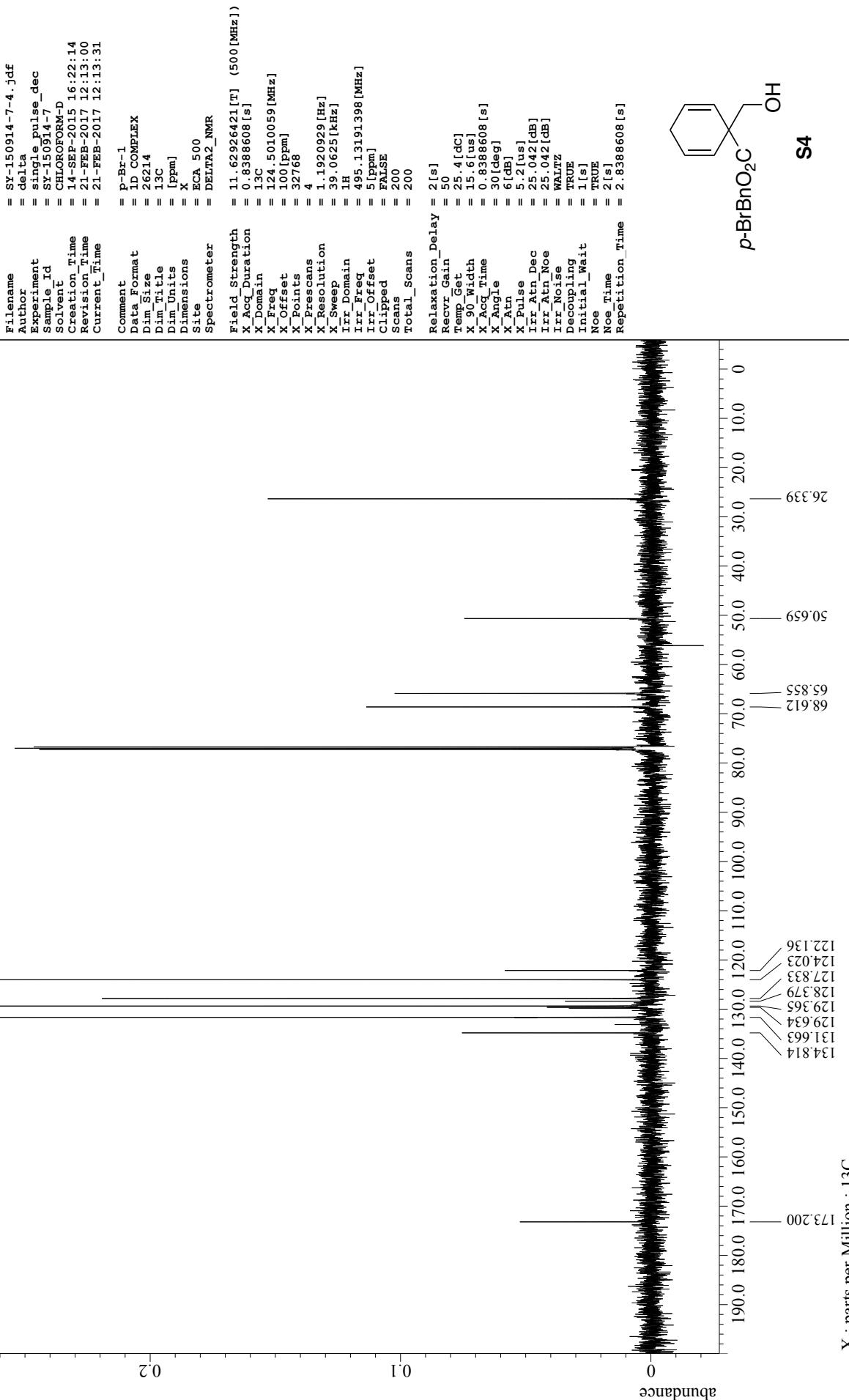
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Dim_Units  = [ppm]
Dimensions = X
Spectrometer = ALICE_NMR
X_Domain   = 1H
X_Freq     = 399.7844939 [MHz]
X_Offset   = 0 [Hz]
X_Points   = 16384
X_Prescans = 1
X_Sweep   = 7.99200781 [kHz]
X_Scans    = 8

Relaxation_Delay = 4.94999981
Recvr_Gain      = 23
Temp_Get        = 21.79999924 [°C]

```





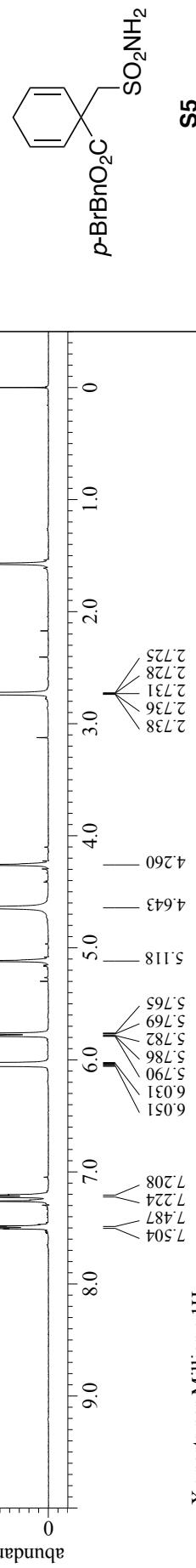
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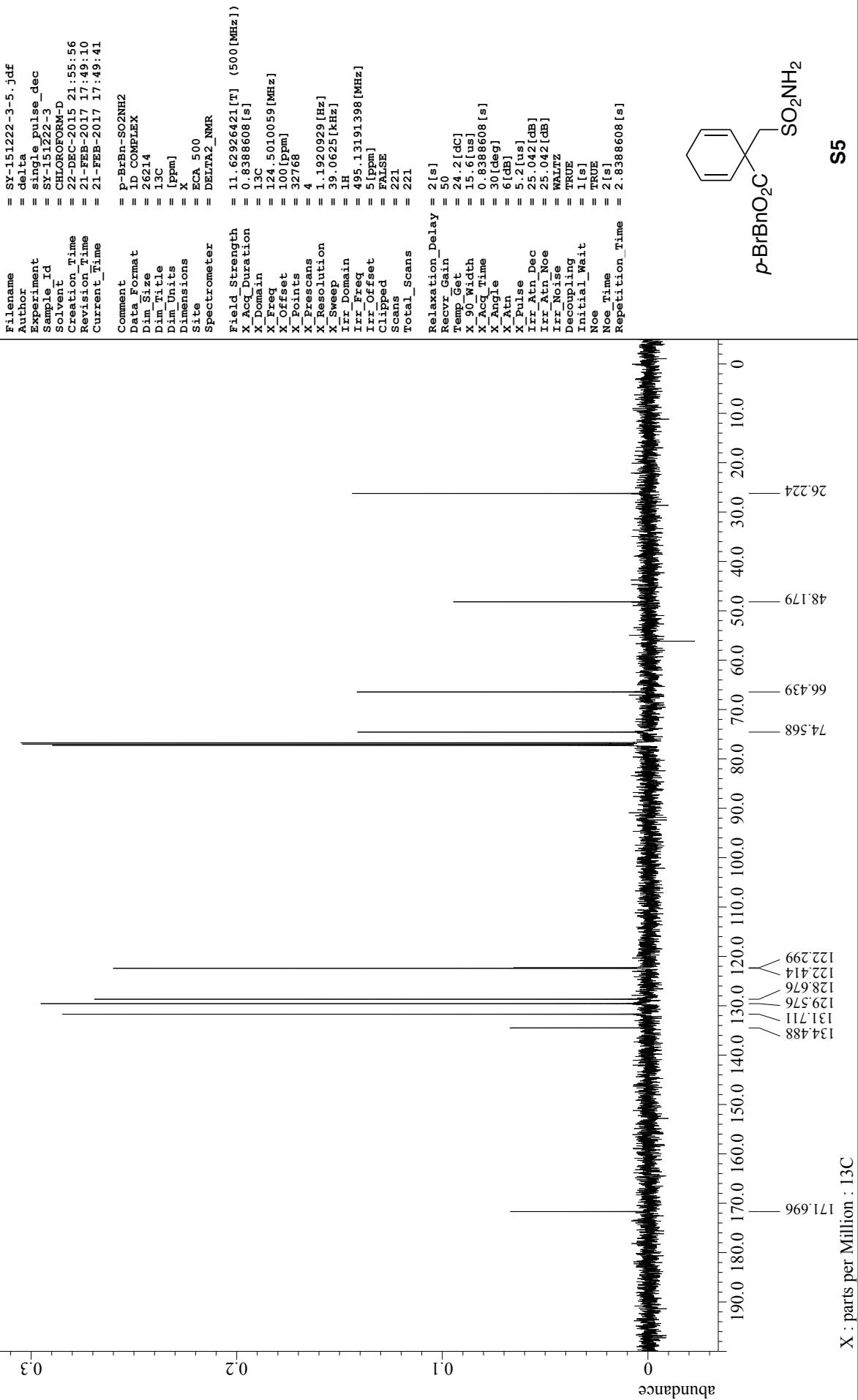
Filename = SY-151222-2-5.jdf
Author = delta
Experiment = single pulse ex2
Sample_Id = SY-151222-2
Solvent = CHLOROFORM-D
Revision_Time = 22-FEB-2015 21:36:28
Current_Time = 21-FEB-2017 17:44:25
Comment = p-BrBn-SO2NH2
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

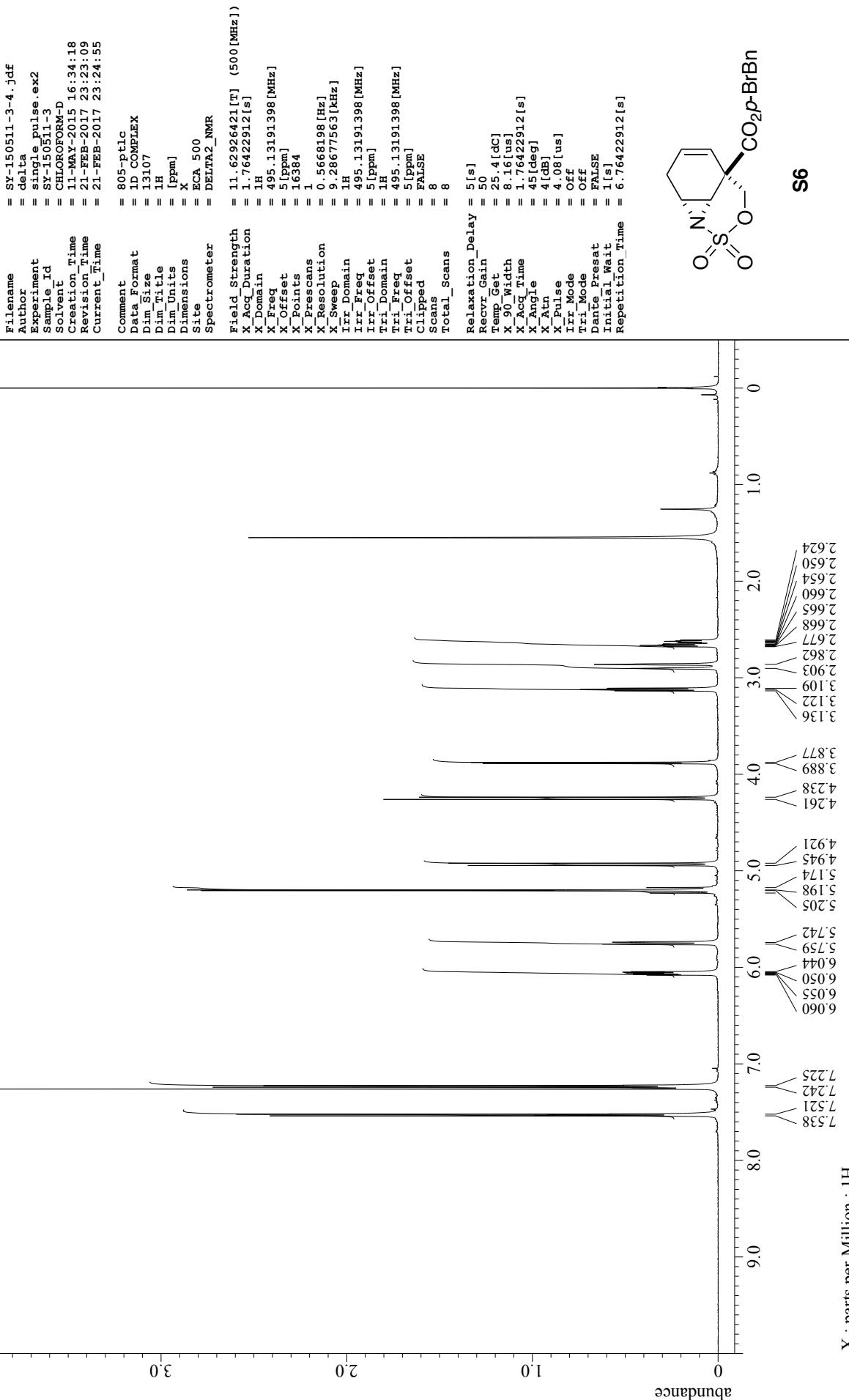
Field_Strength = 11.6226421 [T] (500 [MHz])
X_Acq_Duration = 1.76422912 [s]
X_Domain = 1H
X_Freq = 495.13191398 [MHz]
X_Offset = 5 [ppm]
X_Points = 16384
X_Presans = 0.5668198 [Hz]
X_Resolution = 9.28677563 [kHz]
Irr_Domain = 1H
Irr_Frq = 495.13191398 [MHz]
Irr_Offset = 5 [ppm]
Tri_Domain = 1H
Tri_Freq = 495.13191398 [MHz]
Tri_Offset = 5 [ppm]
Clipped = FALSE
Scans = 8
total_Scans = 8

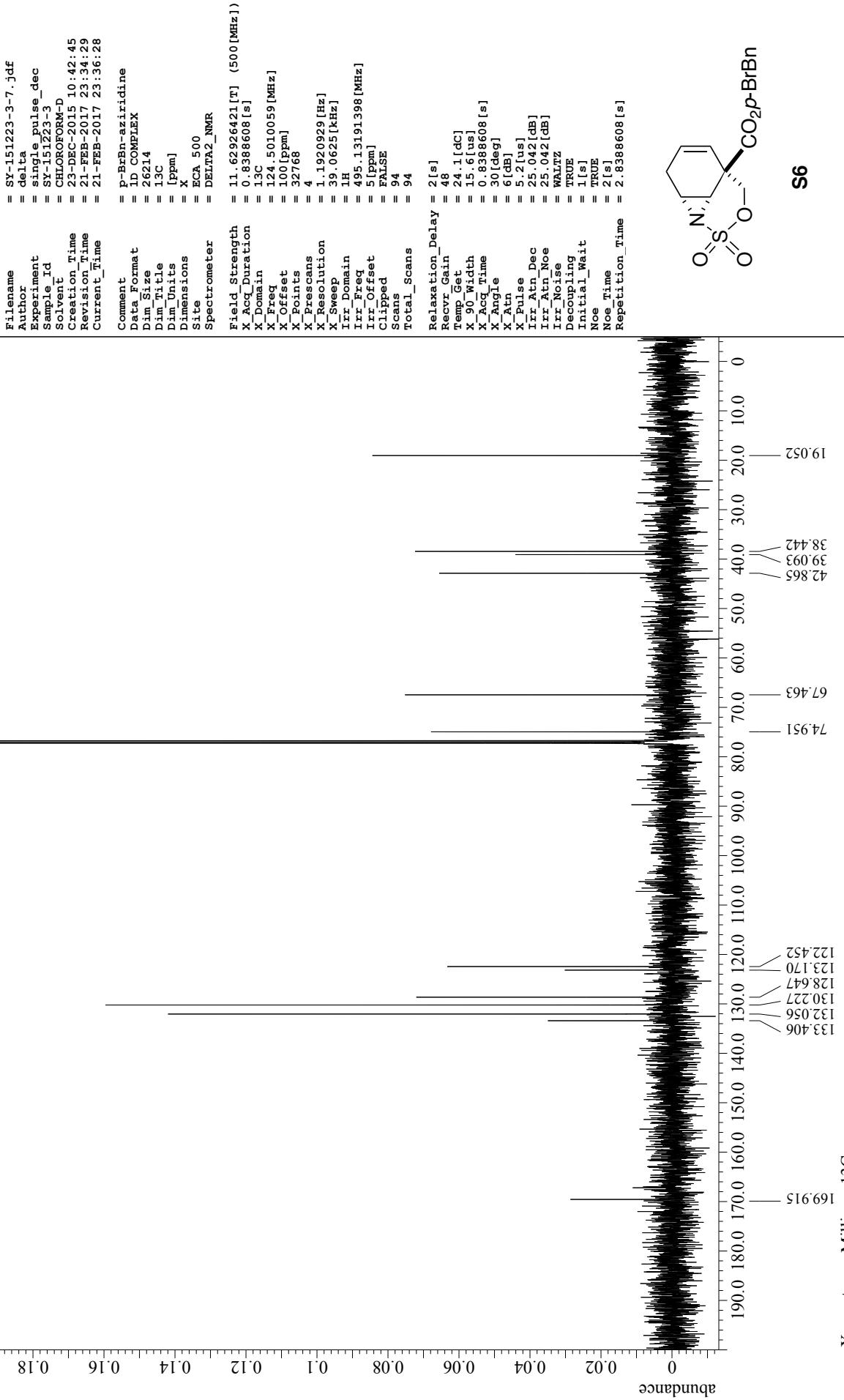
Relaxation_Delay = 5 [s]
Recls_Gain = 52
Temp_Get = 24 [dC]
X_90_Width = 8.16 [us]
X_Acq_Time = 1.76422912 [s]
X_Angle = 45 [deg]
X_Atn = 4 [dB]
X_Pulse = 4.08 [us]
Irr_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1 [s]
Repetition_Time = 6.76422912 [s]

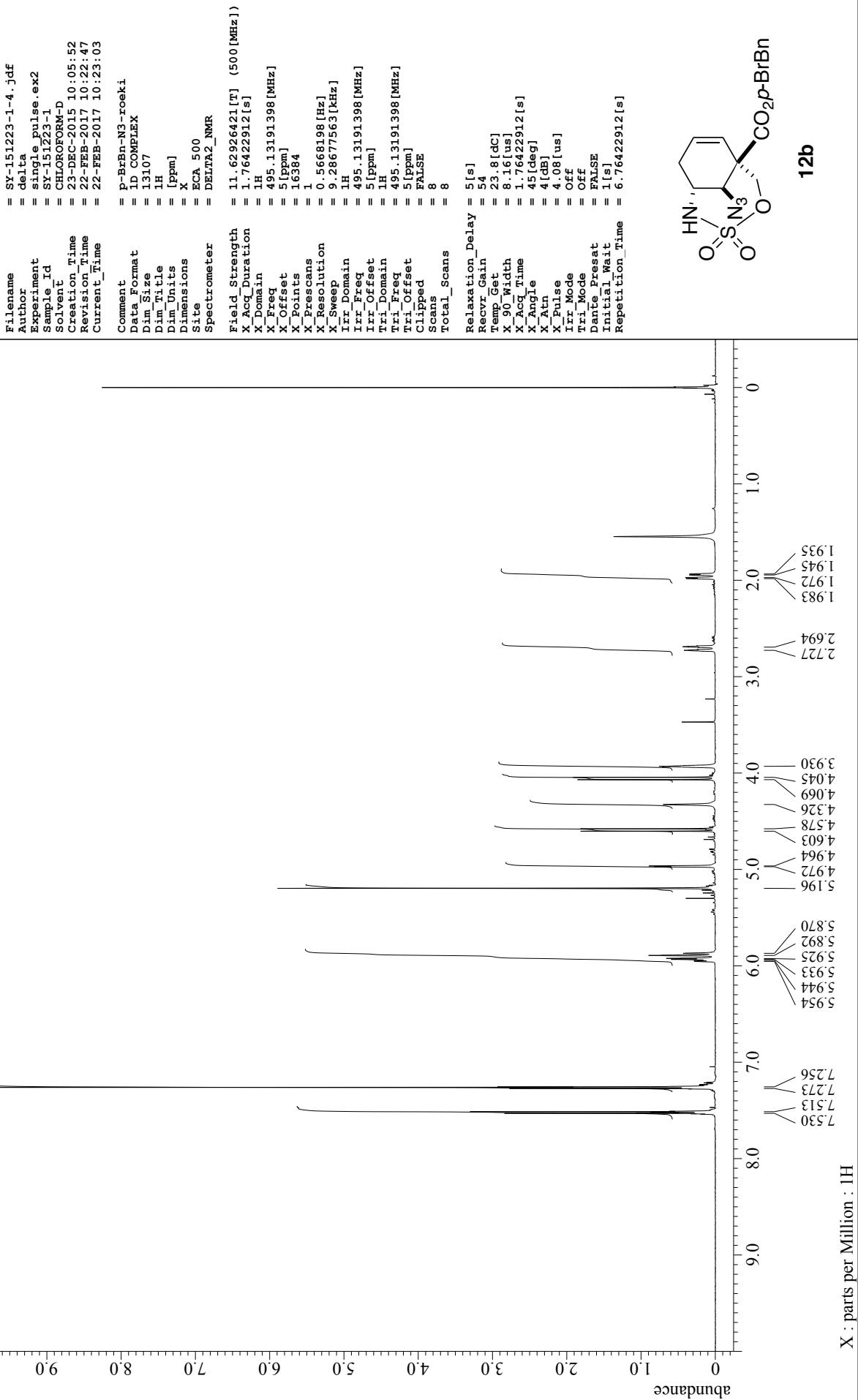
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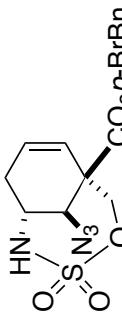
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Filename = SY-151223-2-6.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = SY-151223-2-
Solvent = CHLOROFORM-D
Creation_Time = 23-DEC-2015 10:27:59
Revision_Time = 22-FEB-2017 10:25:59
Current_Time = 22-FEB-2017 10:26:36
Comment = P-BrBn-N3-roski
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECA 500
Spectrometer = DELTA2_NMR

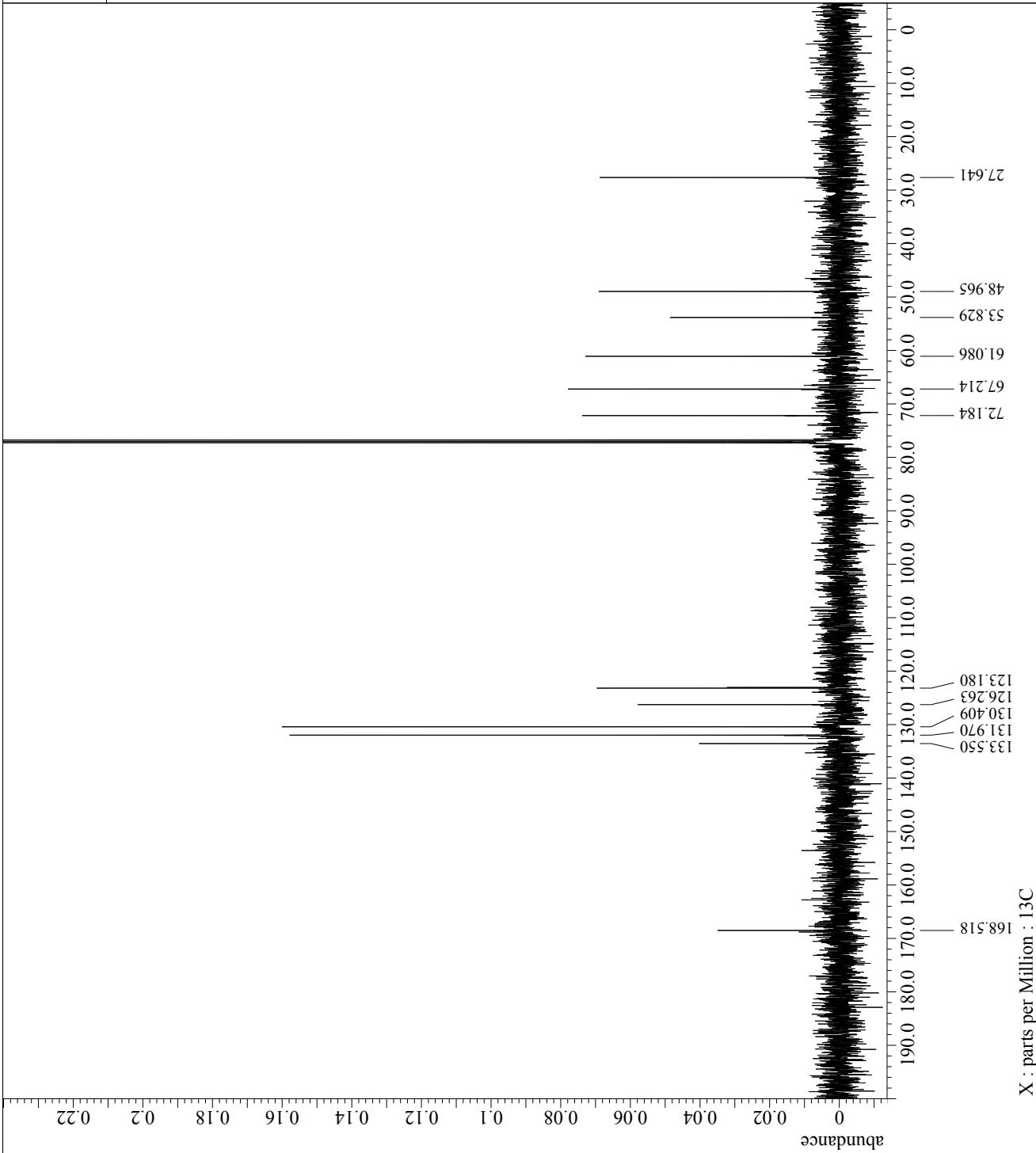
Field_Strength = 11.62226421 [T] (500 [MHz])
X_Acq_Duration = 0.8388608 [s]
X_Domain = 13C
X_Freq = 124.5010059 [MHz]
X_Offset = 100 [ppm]
X_Points = 32768
X_Presans = 4
X_Resolution = 1.1920929 [Hz]
X_Sweep = 39.0625 [kHz]
Irr_Domain = 1H
Irr_Freq = 495.13191398 [MHz]
Irr_Offset = 5 [ppm]
Clipped = FALSE
Scans = 145
Total_Scans = 145

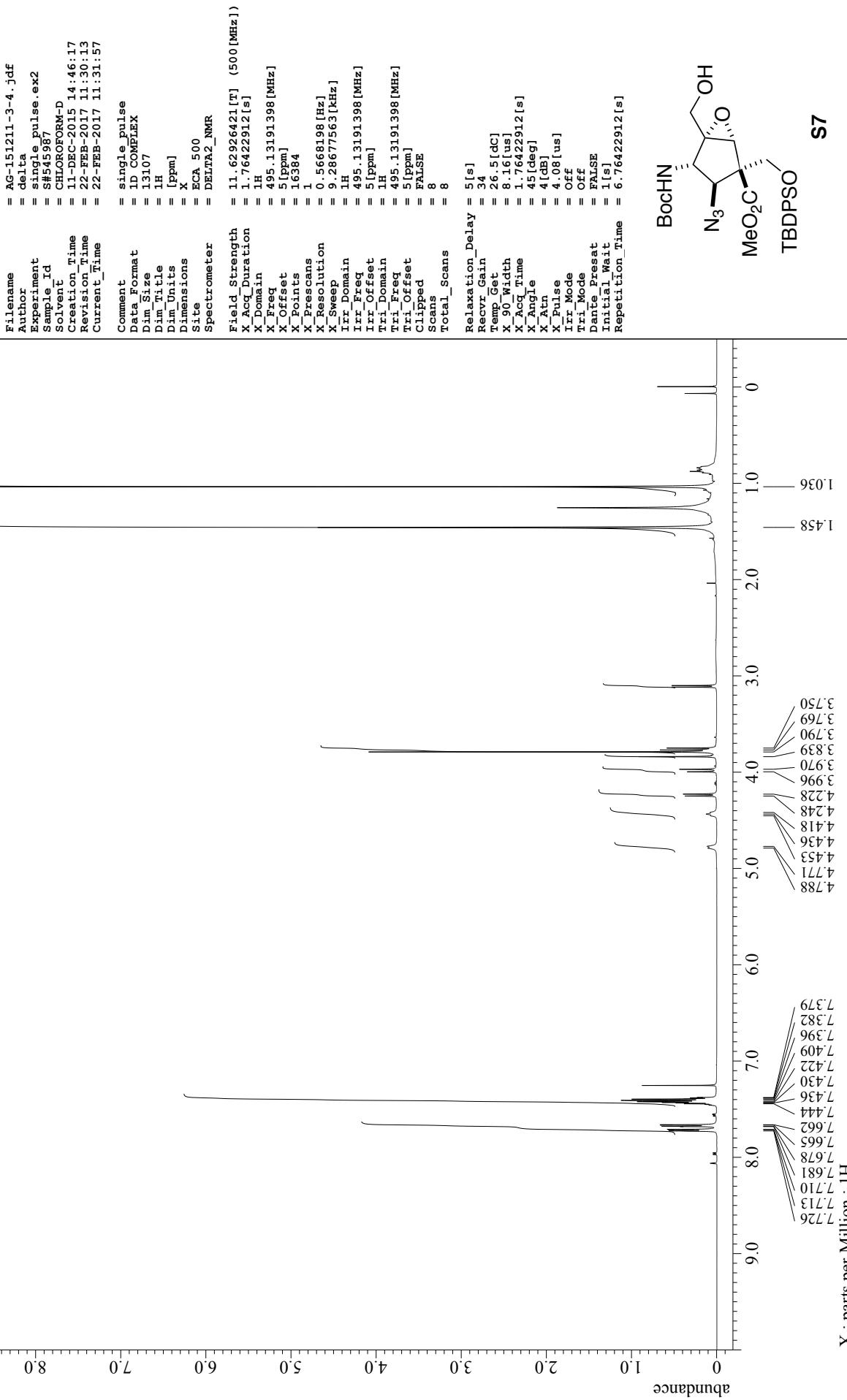
Relaxation_Delay = 2 [s]
Recv_Gain = 50
Temp_Gate = 24.1 [°C]
X_90_Width = 15.6 [us]
X_Acq_Time = 0.8388608 [s]
X_Angle = 30 [deg]
X_Atn = 6 [dB]
X_Pulse = 5.2 [us]
Irr_Atn_Dec = 25.042 [dB]
Irr_Atn_Noe = 25.042 [dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1 [s]
Noe = TRUE
Noe_Time = 2 [s]
Repetition_Time = 2.8388608 [s]

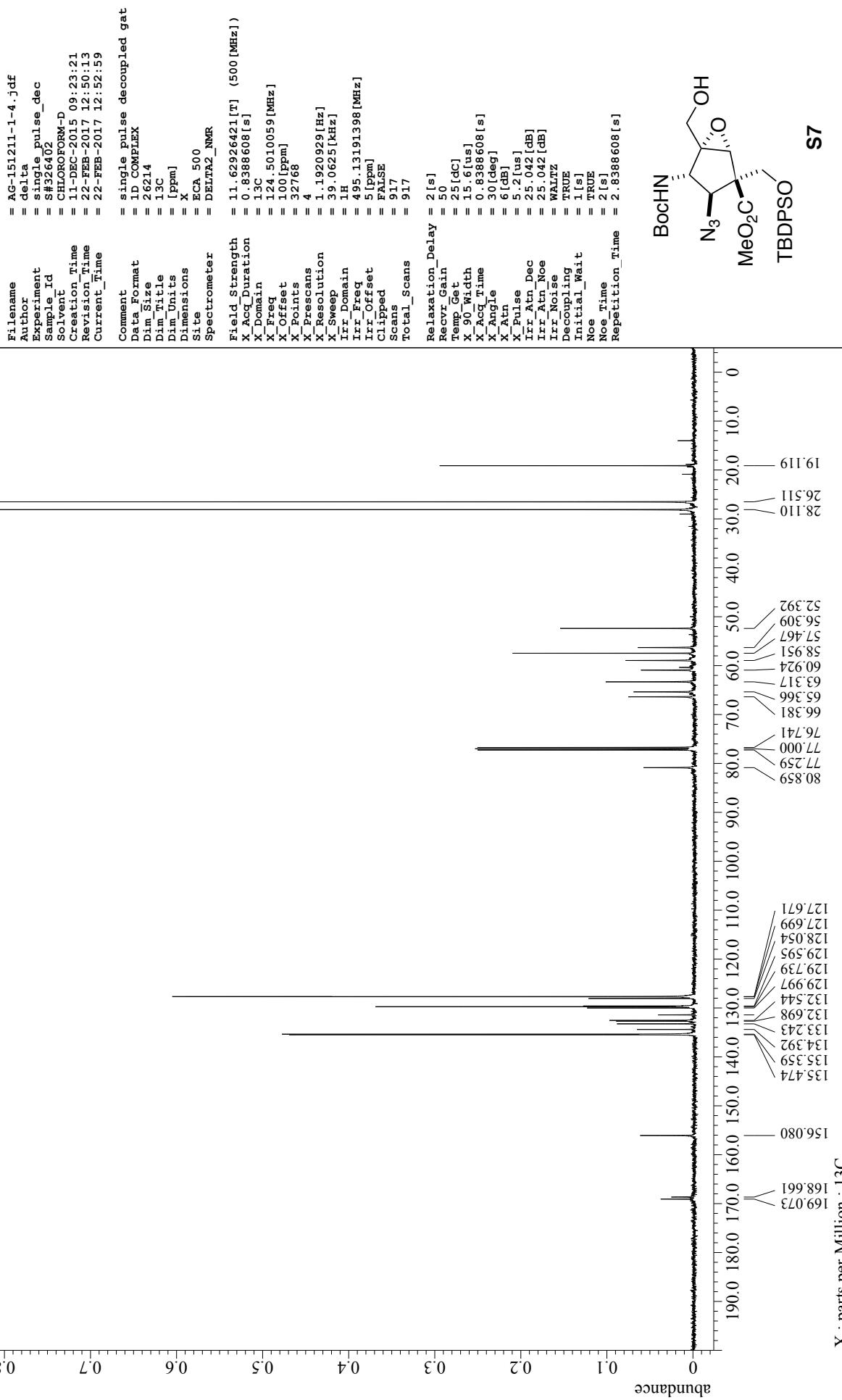
```



12b









Solutions for Innovation

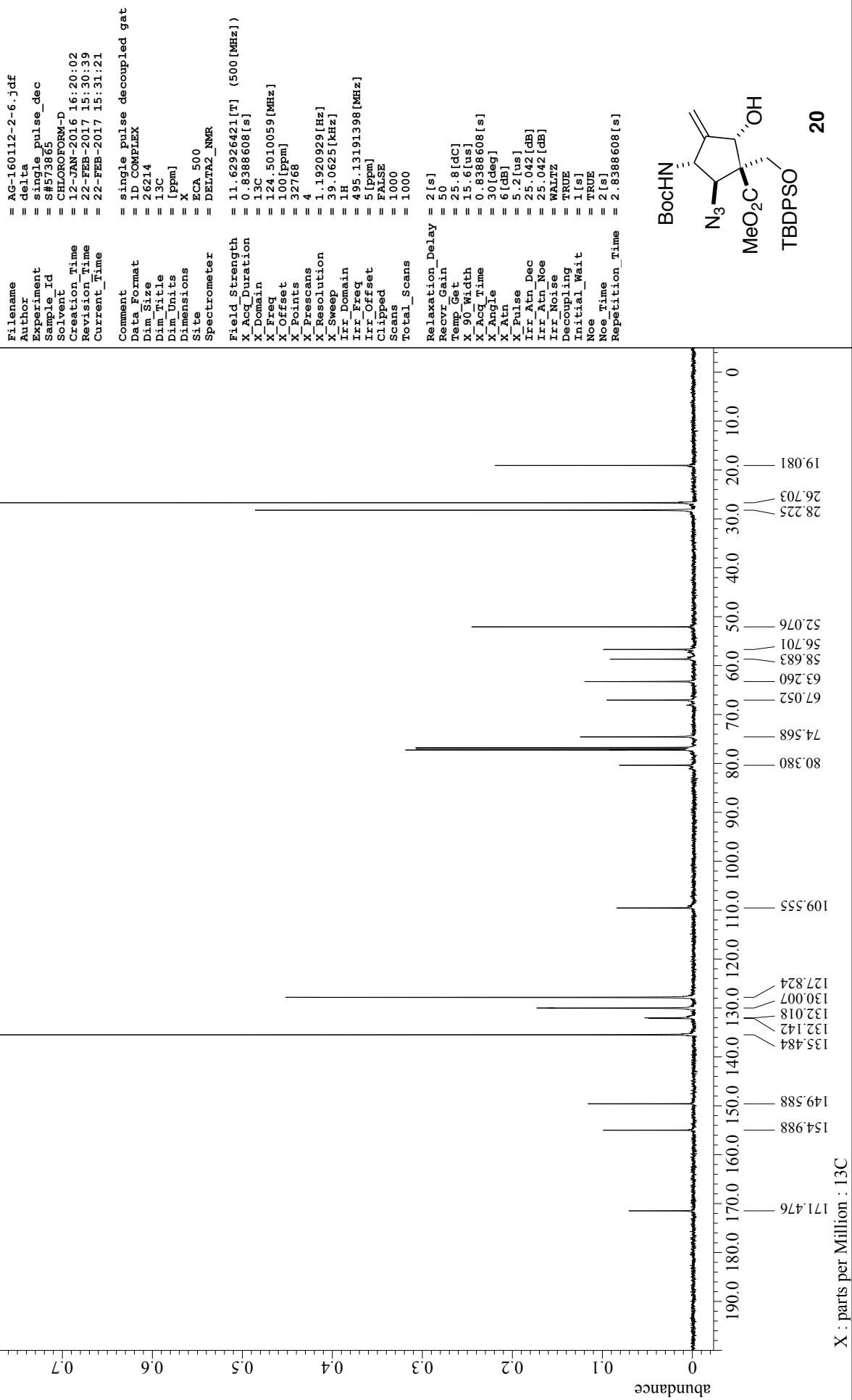
Filename = AG-160112-1-4.jdf
Author = delta.ca
Experiment = single_pulse.ex2
Sample_Id = S#539052
Solvent = CHLOROFORM-D
Creation_Time = 12-FEB-2016 15:25:30
Revision_Time = 22-FEB-2017 15:22:16
Current_Time = 22-FEB-2017 15:22:37

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

Field_Strength = 11.62926421[T] (500 [MHz])
X_Acc_Duration = 1.76422212[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 = 9.28677163 [kHz]
Xr_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]
Recv_Gain = 32
Temp_Get = 25.5 [dC]
X_90_Width = 8.16 [us]
X_Acc_Time = 1.76422212[s]
X_Angle = 45 [deg]
X_Atn = 4 [dB]
X_Pulse = 4.08 [us]
Xr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1 [s]
Repetition_Time = 6.76422212 [s]

Chemical Structure Labels (ppm):
1.662, 7.649, 7.638, 7.632, 7.448, 7.459, 7.463, 7.638, 7.442, 7.432, 7.417, 7.408, 7.394, 3.441, 3.714, 3.850, 3.865, 3.936, 3.956, 4.115, 4.152, 4.546, 4.578, 4.609, 4.923, 4.907, 5.202, 5.455, 7.342, 7.417, 7.420, 7.443, 7.448, 7.459, 7.463, 7.638, 7.649, 7.652, 7.663, 7.666, 7.672, 7.675, 7.678, 7.681, 7.684, 7.687, 7.690, 7.693, 7.696, 7.699, 7.702, 7.705, 7.708, 7.711, 7.714, 7.717, 7.720, 7.723, 7.726, 7.729, 7.732, 7.735, 7.738, 7.741, 7.744, 7.747, 7.750, 7.753, 7.756, 7.759, 7.762, 7.765, 7.768, 7.771, 7.774, 7.777, 7.780, 7.783, 7.786, 7.789, 7.792, 7.795, 7.798, 7.801, 7.804, 7.807, 7.810, 7.813, 7.816, 7.819, 7.822, 7.825, 7.828, 7.831, 7.834, 7.837, 7.840, 7.843, 7.846, 7.849, 7.852, 7.855, 7.858, 7.861, 7.864, 7.867, 7.870, 7.873, 7.876, 7.879, 7.882, 7.885, 7.888, 7.891, 7.894, 7.897, 7.900, 7.903, 7.906, 7.909, 7.912, 7.915, 7.918, 7.921, 7.924, 7.927, 7.930, 7.933, 7.936, 7.939, 7.942, 7.945, 7.948, 7.951, 7.954, 7.957, 7.960, 7.963, 7.966, 7.969, 7.972, 7.975, 7.978, 7.981, 7.984, 7.987, 7.990, 7.993, 7.996, 7.999, 8.002, 8.005, 8.008, 8.011, 8.014, 8.017, 8.020, 8.023, 8.026, 8.029, 8.032, 8.035, 8.038, 8.041, 8.044, 8.047, 8.050, 8.053, 8.056, 8.059, 8.062, 8.065, 8.068, 8.071, 8.074, 8.077, 8.080, 8.083, 8.086, 8.089, 8.092, 8.095, 8.098, 8.101, 8.104, 8.107, 8.110, 8.113, 8.116, 8.119, 8.122, 8.125, 8.128, 8.131, 8.134, 8.137, 8.140, 8.143, 8.146, 8.149, 8.152, 8.155, 8.158, 8.161, 8.164, 8.167, 8.170, 8.173, 8.176, 8.179, 8.182, 8.185, 8.188, 8.191, 8.194, 8.197, 8.200, 8.203, 8.206, 8.209, 8.212, 8.215, 8.218, 8.221, 8.224, 8.227, 8.230, 8.233, 8.236, 8.239, 8.242, 8.245, 8.248, 8.251, 8.254, 8.257, 8.260, 8.263, 8.266, 8.269, 8.272, 8.275, 8.278, 8.281, 8.284, 8.287, 8.290, 8.293, 8.296, 8.299, 8.302, 8.305, 8.308, 8.311, 8.314, 8.317, 8.320, 8.323, 8.326, 8.329, 8.332, 8.335, 8.338, 8.341, 8.344, 8.347, 8.350, 8.353, 8.356, 8.359, 8.362, 8.365, 8.368, 8.371, 8.374, 8.377, 8.380, 8.383, 8.386, 8.389, 8.392, 8.395, 8.398, 8.401, 8.404, 8.407, 8.410, 8.413, 8.416, 8.419, 8.422, 8.425, 8.428, 8.431, 8.434, 8.437, 8.440, 8.443, 8.446, 8.449, 8.452, 8.455, 8.458, 8.461, 8.464, 8.467, 8.470, 8.473, 8.476, 8.479, 8.482, 8.485, 8.488, 8.491, 8.494, 8.497, 8.500, 8.503, 8.506, 8.509, 8.512, 8.515, 8.518, 8.521, 8.524, 8.527, 8.530, 8.533, 8.536, 8.539, 8.542, 8.545, 8.548, 8.551, 8.554, 8.557, 8.560, 8.563, 8.566, 8.569, 8.572, 8.575, 8.578, 8.581, 8.584, 8.587, 8.590, 8.593, 8.596, 8.599, 8.602, 8.605, 8.608, 8.611, 8.614, 8.617, 8.620, 8.623, 8.626, 8.629, 8.632, 8.635, 8.638, 8.641, 8.644, 8.647, 8.650, 8.653, 8.656, 8.659, 8.662, 8.665, 8.668, 8.671, 8.674, 8.677, 8.680, 8.683, 8.686, 8.689, 8.692, 8.695, 8.698, 8.701, 8.704, 8.707, 8.710, 8.713, 8.716, 8.719, 8.722, 8.725, 8.728, 8.731, 8.734, 8.737, 8.740, 8.743, 8.746, 8.749, 8.752, 8.755, 8.758, 8.761, 8.764, 8.767, 8.770, 8.773, 8.776, 8.779, 8.782, 8.785, 8.788, 8.791, 8.794, 8.797, 8.800, 8.803, 8.806, 8.809, 8.812, 8.815, 8.818, 8.821, 8.824, 8.827, 8.830, 8.833, 8.836, 8.839, 8.842, 8.845, 8.848, 8.851, 8.854, 8.857, 8.860, 8.863, 8.866, 8.869, 8.872, 8.875, 8.878, 8.881, 8.884, 8.887, 8.890, 8.893, 8.896, 8.899, 8.902, 8.905, 8.908, 8.911, 8.914, 8.917, 8.920, 8.923, 8.926, 8.929, 8.932, 8.935, 8.938, 8.941, 8.944, 8.947, 8.950, 8.953, 8.956, 8.959, 8.962, 8.965, 8.968, 8.971, 8.974, 8.977, 8.980, 8.983, 8.986, 8.989, 8.992, 8.995, 8.998, 9.001, 9.004, 9.007, 9.010, 9.013, 9.016, 9.019, 9.022, 9.025, 9.028, 9.031, 9.034, 9.037, 9.040, 9.043, 9.046, 9.049, 9.052, 9.055, 9.058, 9.061, 9.064, 9.067, 9.070, 9.073, 9.076, 9.079, 9.082, 9.085, 9.088, 9.091, 9.094, 9.097, 9.100, 9.103, 9.106, 9.109, 9.112, 9.115, 9.118, 9.121, 9.124, 9.127, 9.130, 9.133, 9.136, 9.139, 9.142, 9.145, 9.148, 9.151, 9.154, 9.157, 9.160, 9.163, 9.166, 9.169, 9.172, 9.175, 9.178, 9.181, 9.184, 9.187, 9.190, 9.193, 9.196, 9.199, 9.202, 9.205, 9.208, 9.211, 9.214, 9.217, 9.220, 9.223, 9.226, 9.229, 9.232, 9.235, 9.238, 9.241, 9.244, 9.247, 9.250, 9.253, 9.256, 9.259, 9.262, 9.265, 9.268, 9.271, 9.274, 9.277, 9.280, 9.283, 9.286, 9.289, 9.292, 9.295, 9.298, 9.301, 9.304, 9.307, 9.310, 9.313, 9.316, 9.319, 9.322, 9.325, 9.328, 9.331, 9.334, 9.337, 9.340, 9.343, 9.346, 9.349, 9.352, 9.355, 9.358, 9.361, 9.364, 9.367, 9.370, 9.373, 9.376, 9.379, 9.382, 9.385, 9.388, 9.391, 9.394, 9.397, 9.400, 9.403, 9.406, 9.409, 9.412, 9.415, 9.418, 9.421, 9.424, 9.427, 9.430, 9.433, 9.436, 9.439, 9.442, 9.445, 9.448, 9.451, 9.454, 9.457, 9.460, 9.463, 9.466, 9.469, 9.472, 9.475, 9.478, 9.481, 9.484, 9.487, 9.490, 9.493, 9.496, 9.499, 9.502, 9.505, 9.508, 9.511, 9.514, 9.517, 9.520, 9.523, 9.526, 9.529, 9.532, 9.535, 9.538, 9.541, 9.544, 9.547, 9.550, 9.553, 9.556, 9.559, 9.562, 9.565, 9.568, 9.571, 9.574, 9.577, 9.580, 9.583, 9.586, 9.589, 9.592, 9.595, 9.598, 9.601, 9.604, 9.607, 9.610, 9.613, 9.616, 9.619, 9.622, 9.625, 9.628, 9.631, 9.634, 9.637, 9.640, 9.643, 9.646, 9.649, 9.652, 9.655, 9.658, 9.661, 9.664, 9.667, 9.670, 9.673, 9.676, 9.679, 9.682, 9.685, 9.688, 9.691, 9.694, 9.697, 9.700, 9.703, 9.706, 9.709, 9.712, 9.715, 9.718, 9.721, 9.724, 9.727, 9.730, 9.733, 9.736, 9.739, 9.742, 9.745, 9.748, 9.751, 9.754, 9.757, 9.760, 9.763, 9.766, 9.769, 9.772, 9.775, 9.778, 9.781, 9.784, 9.787, 9.790, 9.793, 9.796, 9.799, 9.802, 9.805, 9.808, 9.811, 9.814, 9.817, 9.820, 9.823, 9.826, 9.829, 9.832, 9.835, 9.838, 9.841, 9.844, 9.847, 9.850, 9.853, 9.856, 9.859, 9.862, 9.865, 9.868, 9.871, 9.874, 9.877, 9.880, 9.883, 9.886, 9.889, 9.892, 9.895, 9.898, 9.901, 9.904, 9.907, 9.910, 9.913, 9.916, 9.919, 9.922, 9.925, 9.928, 9.931, 9.934, 9.937, 9.940, 9.943, 9.946, 9.949, 9.952, 9.955, 9.958, 9.961, 9.964, 9.967, 9.970, 9.973, 9.976, 9.979, 9.982, 9.985, 9.988, 9.991, 9.994, 9.997, 9.999, 10.000, 10.001, 10.002, 10.003, 10.004, 10.005, 10.006, 10.007, 10.008, 10.009, 10.010, 10.011, 10.012, 10.013, 10.014, 10.015, 10.016, 10.017, 10.018, 10.019, 10.020, 10.021, 10.022, 10.023, 10.024, 10.025, 10.026, 10.027, 10.028, 10.029, 10.030, 10.031, 10.032, 10.033, 10.034, 10.035, 10.036, 10.037, 10.038, 10.039, 10.040, 10.041, 10.042, 10.043, 10.044, 10.045, 10.046, 10.047, 10.048, 10.049, 10.050, 10.051, 10.052, 10.053, 10.054, 10.055, 10.056, 10.057, 10.058, 10.059, 10.060, 10.061, 10.062, 10.063, 10.064, 10.065, 10.066, 10.067, 10.068, 10.069, 10.070, 10.071, 10.072, 10.073, 10.074, 10.075, 10.076, 10.077, 10.078, 10.079, 10.080, 10.081, 10.082, 10.083, 10.084, 10.085, 10.086, 10.087, 10.088, 10.089, 10.090, 10.091, 10.092, 10.093, 10.094, 10.095, 10.096, 10.097, 10.098, 10.099, 10.100, 10.101, 10.102, 10.103, 10.104, 10.105, 10.106, 10.107, 10.108, 10.109, 10.110, 10.111, 10.112, 10.113, 10.114, 10.115, 10.116, 10.117, 10.118, 10.119, 10.120, 10.121, 10.122, 10.123, 10.124, 10.125, 10.126, 10.127, 10.128, 10.129, 10.130, 10.131, 10.132, 10.133, 10.134, 10.135, 10.136, 10.137, 10.138, 10.139, 10.140, 10.141, 10.142, 10.143, 10.144, 10.145, 10.146, 10.147, 10.148, 10.149, 10.150, 10.151, 10.152, 10.153, 10.154, 10.155, 10.156, 10.157, 10.158, 10.159, 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10.660, 10.661, 10.662, 10.663, 10.664, 10.665, 10.666, 10.667, 10.668, 10.669, 10.670, 10.671, 10.672, 10.673, 10.674, 10.675, 10.676, 10.677, 10.678, 10.679, 10.680, 10.681, 10.682, 10.683, 10.684, 10.685, 10.686, 10.687, 10.688, 10.689, 10.690, 10.691, 10.692, 10.693, 10.694, 10.695, 10.696, 10.697, 10.698, 10.699, 10.700, 10.701, 10.702, 10.703, 10.704, 10.705, 10.706, 10.707, 10.708, 10.709, 10.710, 10.711, 10.712, 10.713, 10.714, 10.715, 10.716, 10.717, 10.718, 10.719, 10.720, 10.721, 10.722, 10.723, 10.724, 10.725, 10.726, 10.727, 10.728, 10.729, 10.730, 10.731, 10.732, 10.733, 10.734, 10.735, 10.736, 10.737, 10.7



```

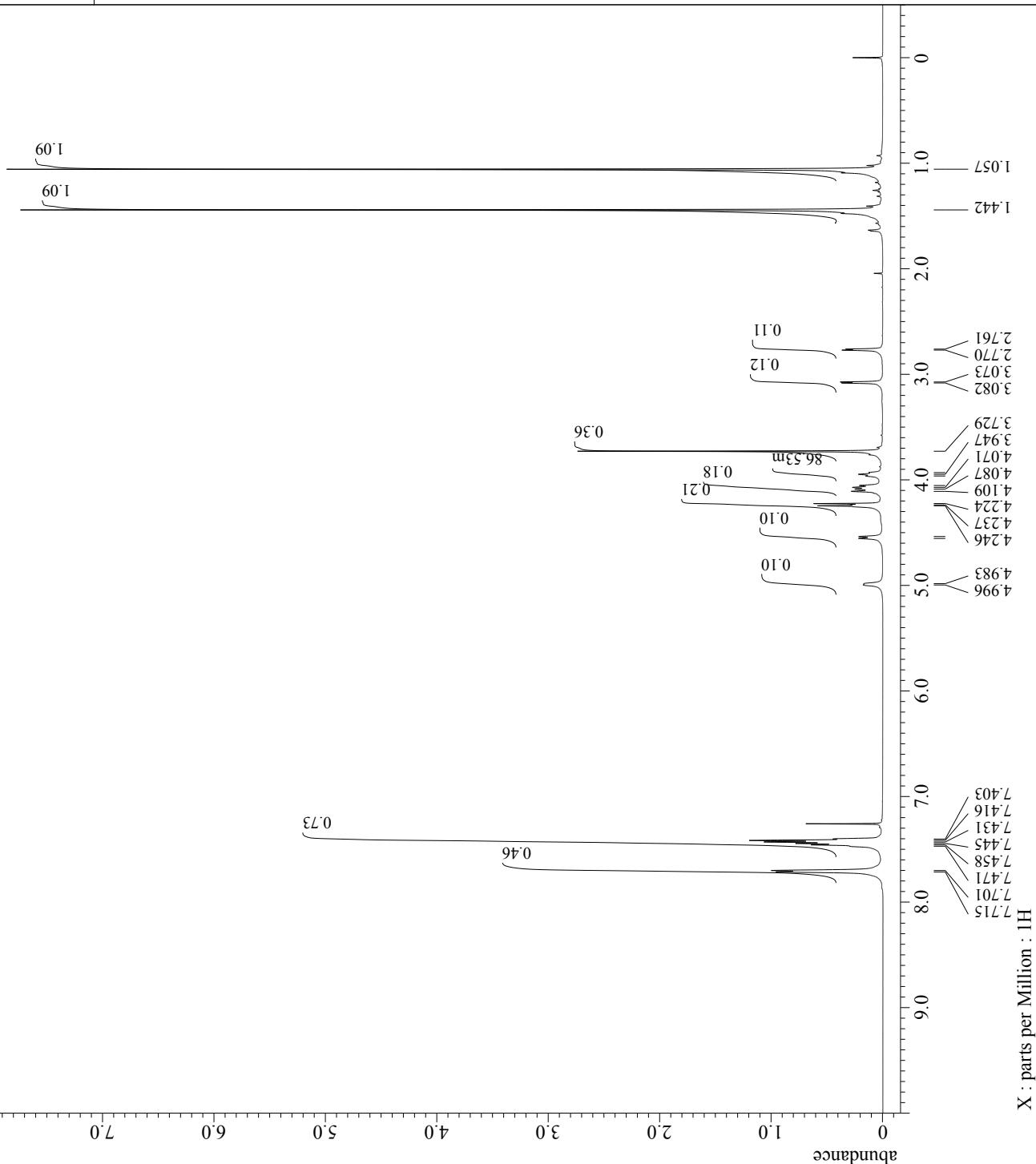
Filename = 1070222-1.alcohol1-5.jdf
Author = delta
Experiment = single_pulse_ex2
Sample_Id = SH#227734
Solvent_C = CHLOROFORM-D
Creation_Time = 22-FEB-2017 08:31:52
Revision_Time = 2-MAR-2017 11:39:12
Current_Time = 2-MAR-2017 11:39:50

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 = 11.62296421 [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_Resolutions = 0.5668198 [Hz]
X_Sweep = 9.2867756 [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]
Recovery_Gain = 34
temp_Get = 20.6 [°C]
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_Preset = FALSE
Initial_Wait = 1 [s]
Repetition_Time = 6.76422912 [s]

```



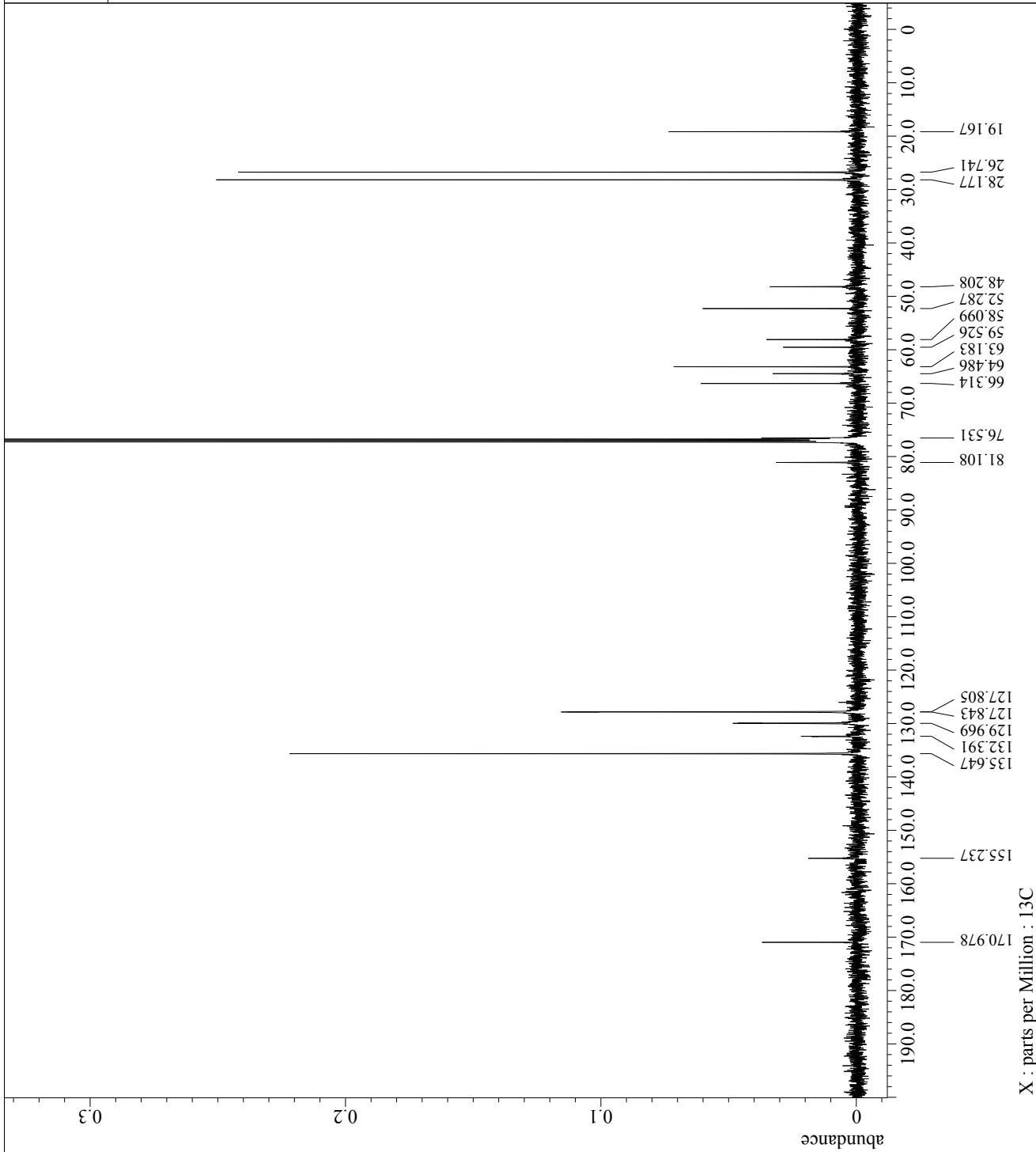
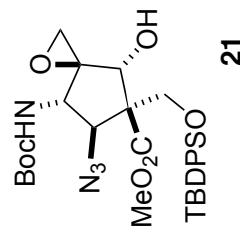
```

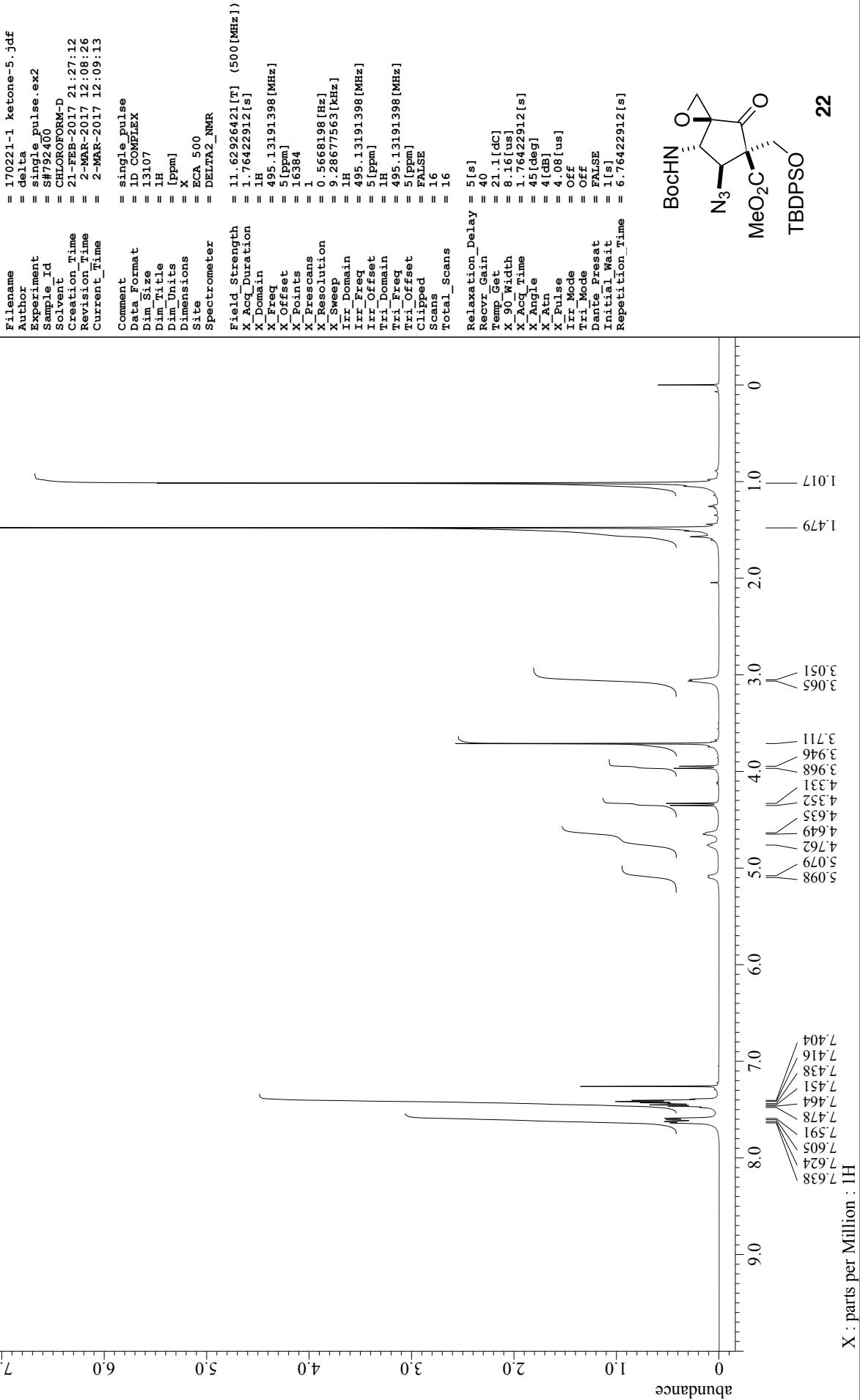
Filename = 170222-2.alcohol.C-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = S1333734
Solvent = CHLOROFORM-D
Revision_Time = 22-FEB-2017 09:38:40
Current_Time = 2-MAR-2017 11:41:19
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [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]
ClipSpeed = TRUE
Scans = 1200
Total_Scans = 1200

Relaxation_Delay = 2 [s]
Recv_Gain = 54
temp_get = 21 [degC]
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_Noise = WALTZ
Irr_Noise = TRUE
Decoupling = TRUE
Initial_Wait = 1 [s]
Noe = TRUE
Noe_Time = 2 [s]
Repetition_Time = 2.8388608 [s]

```





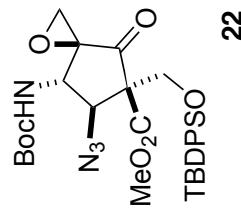
```

File name = 170221-2_ketone_C-5-.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = S1800445
Solvent = CHLOROFORM-D
Revision Time = 21-FEB-2017 22:55:20
Current_Time = 2-MAR-2017 12:20:39
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

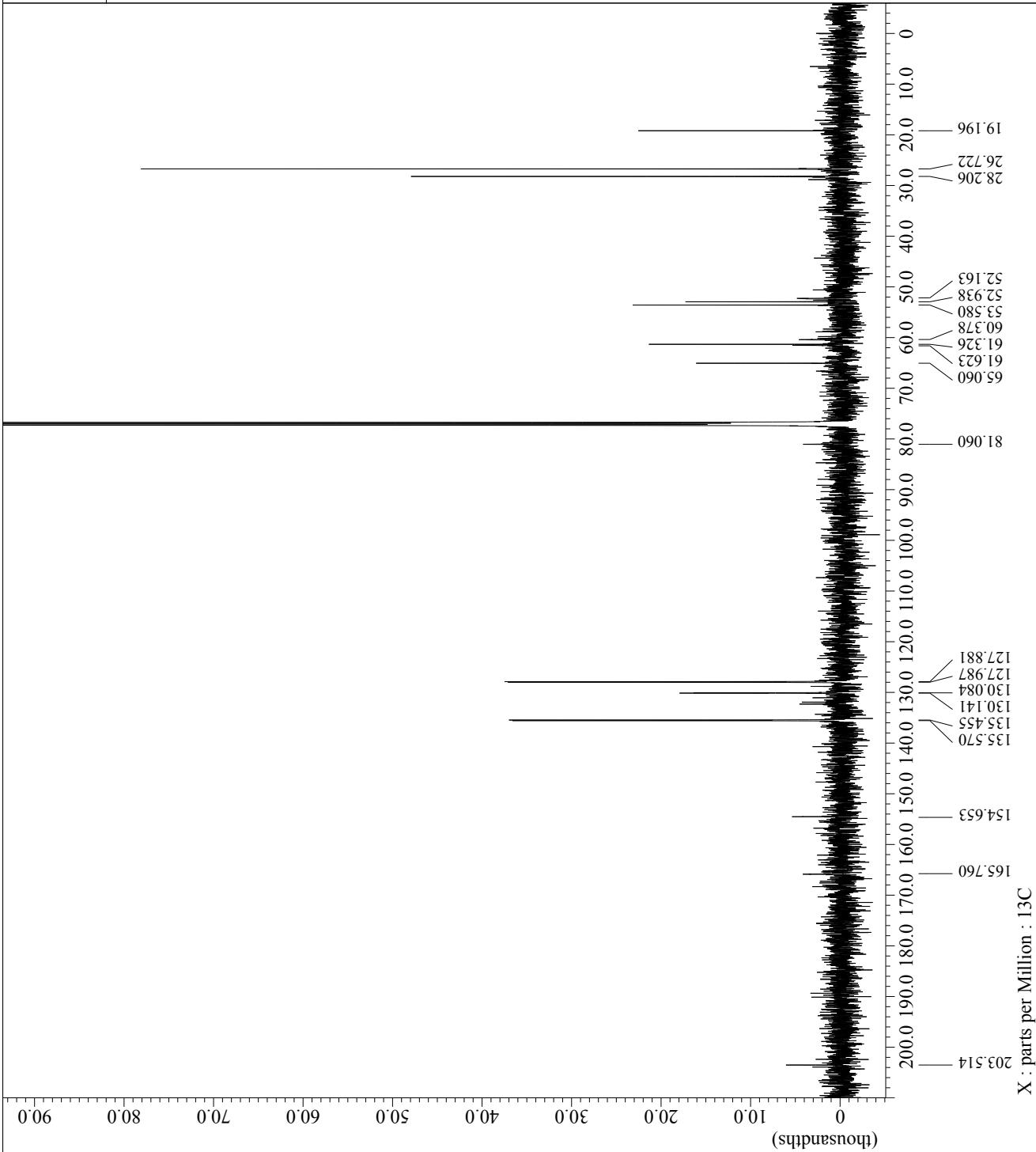
Field_Strength = 11.62926421 [T] (500 [MHz])
X_Acq_Duration = 0.8388608 [s]
X_Domain = 13C
X_Freq = 124.5010059 [MHz]
X_Offset = 100 [ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.1920929 [Hz]
X_Sweep = 39.0625 [kHz]
Irr_Domain = 1H
Irr_Freq = 495.13191398 [MHz]
Irr_Offset = 5 [ppm]
ClipSpeed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

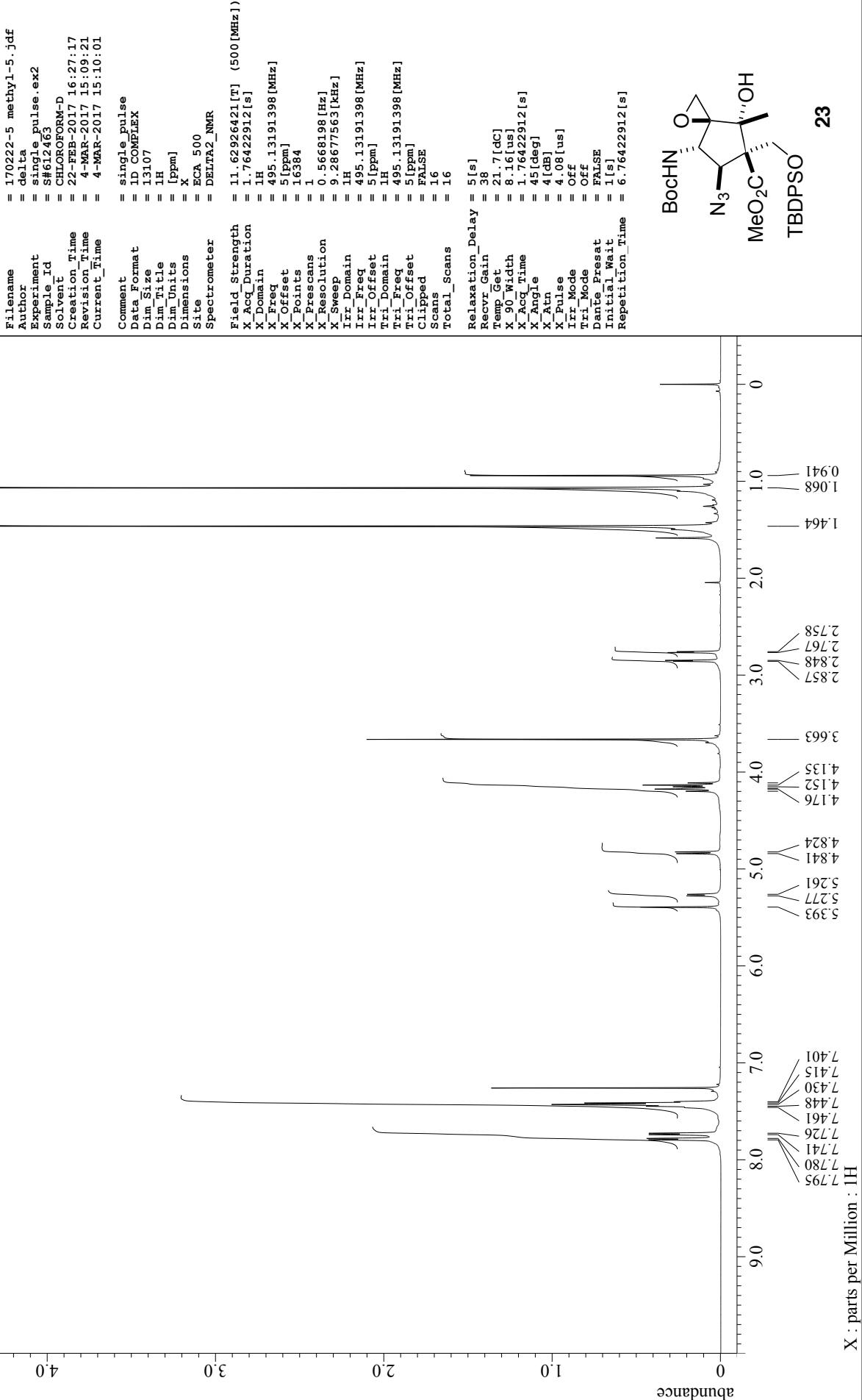
Relaxation_Delay = 2 [s]
Recv_Gain = 50
Temp_Get = 21.3 [dC]
X_90_Width = 15.6 [us]
X_Acq_Time = 0.8388608 [s]
X_Angle = 30 [deg]
X_Atn = 6 [dB]
X_Pulse = 5.2 [us]
Irr_Atn_Dec = 23.2 [dB]
Irr_Atn_Noe = 23.2 [dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1 [s]
Noe = TRUE
Noe_Time = 2 [s]
Repetition_Time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

```



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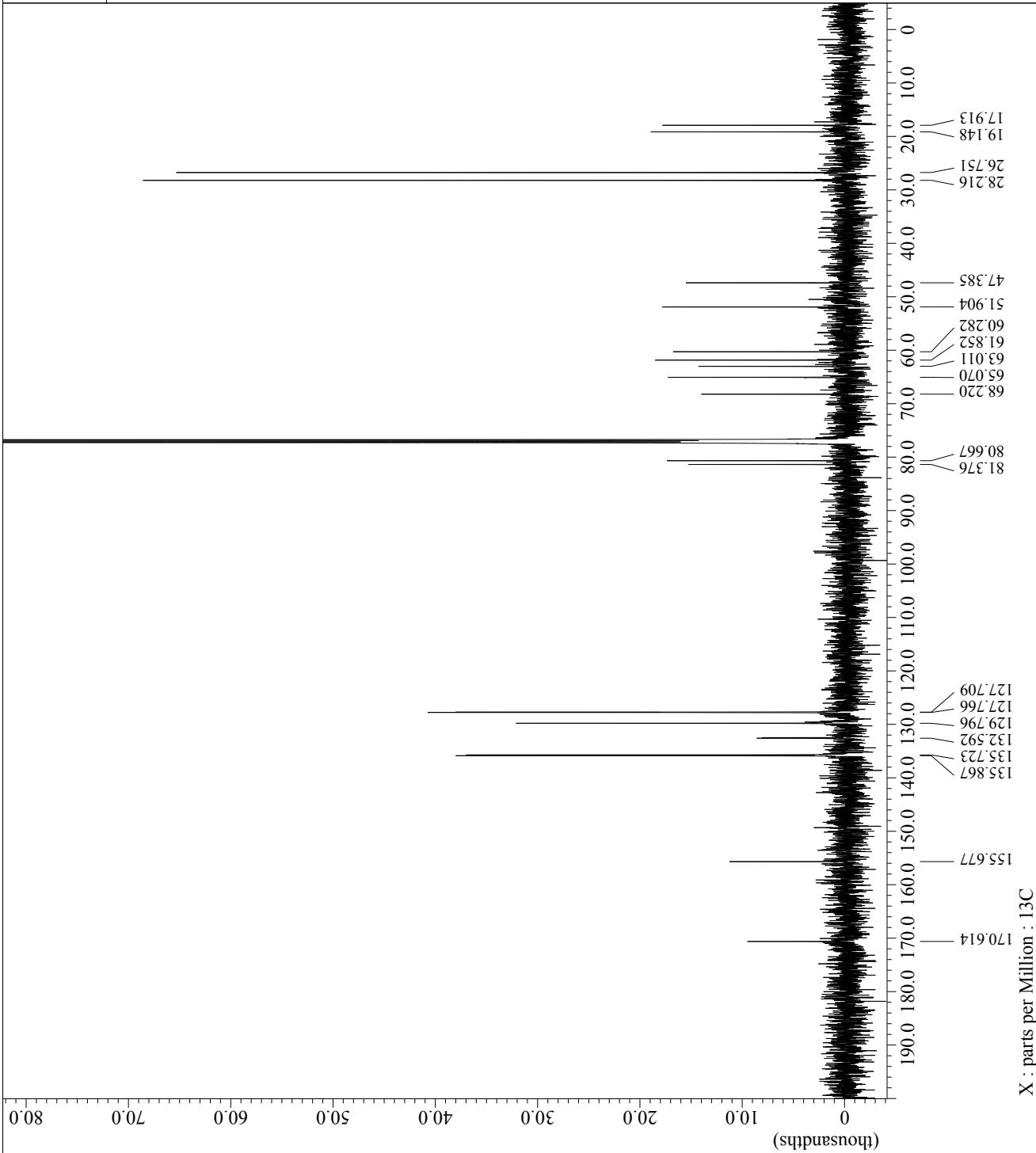
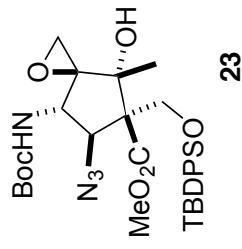
```

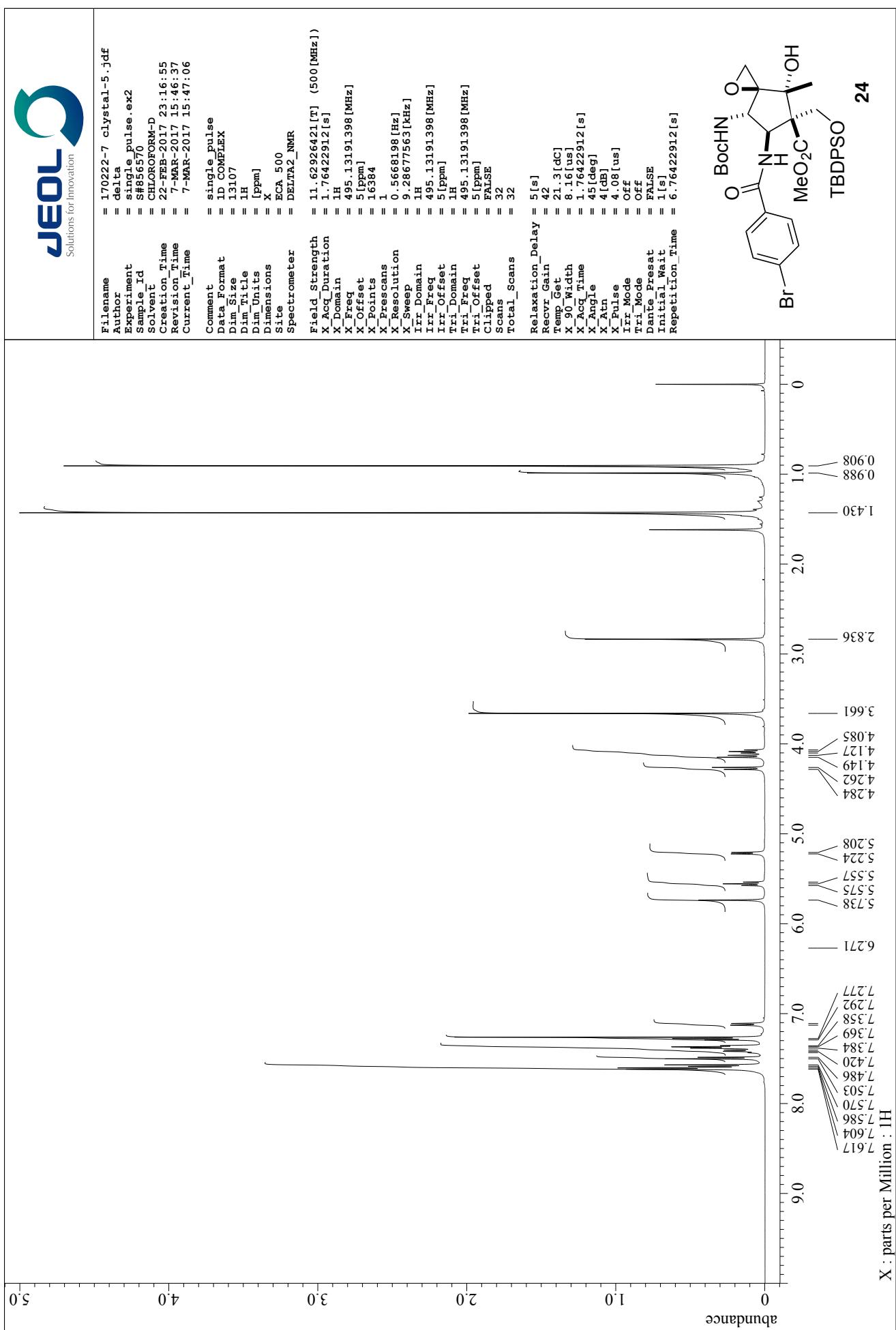
File_name = 170222-6_methyl_C-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = S1640775
Solvent = CHLOROFORM-D
Revision_Time = 22-FEB-2017 18:29:53
Current_Time = 4-MAR-2017 15:16:53
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [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]
ClipSpeed = FALSE
Scans = 1600
Total_Scans = 1600

Relaxation_Delay = 2 [s]
Recv_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]

```





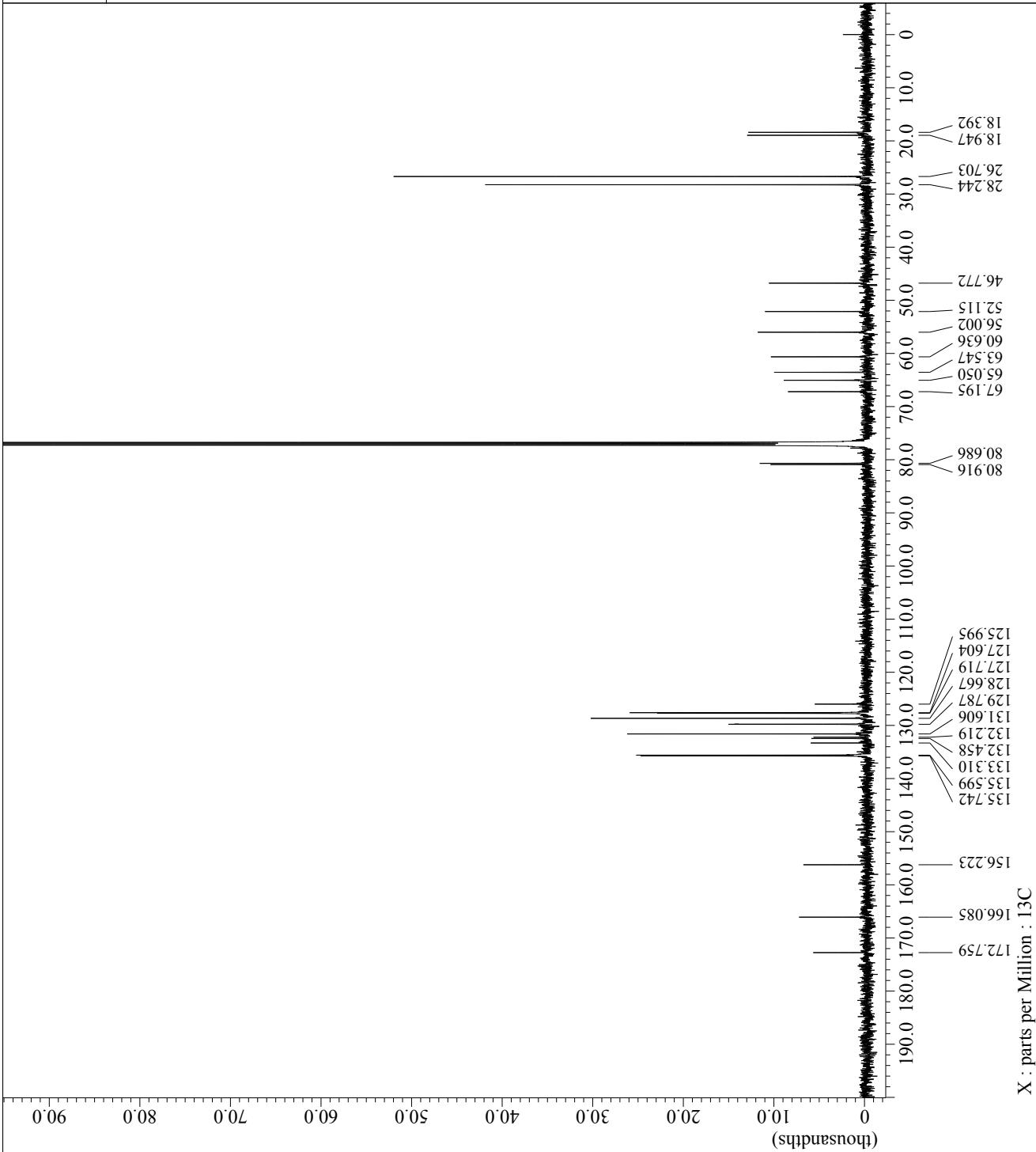
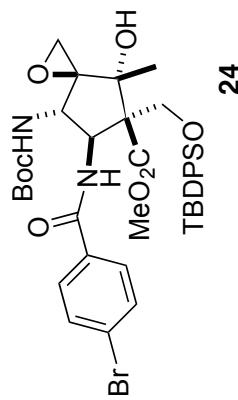
```

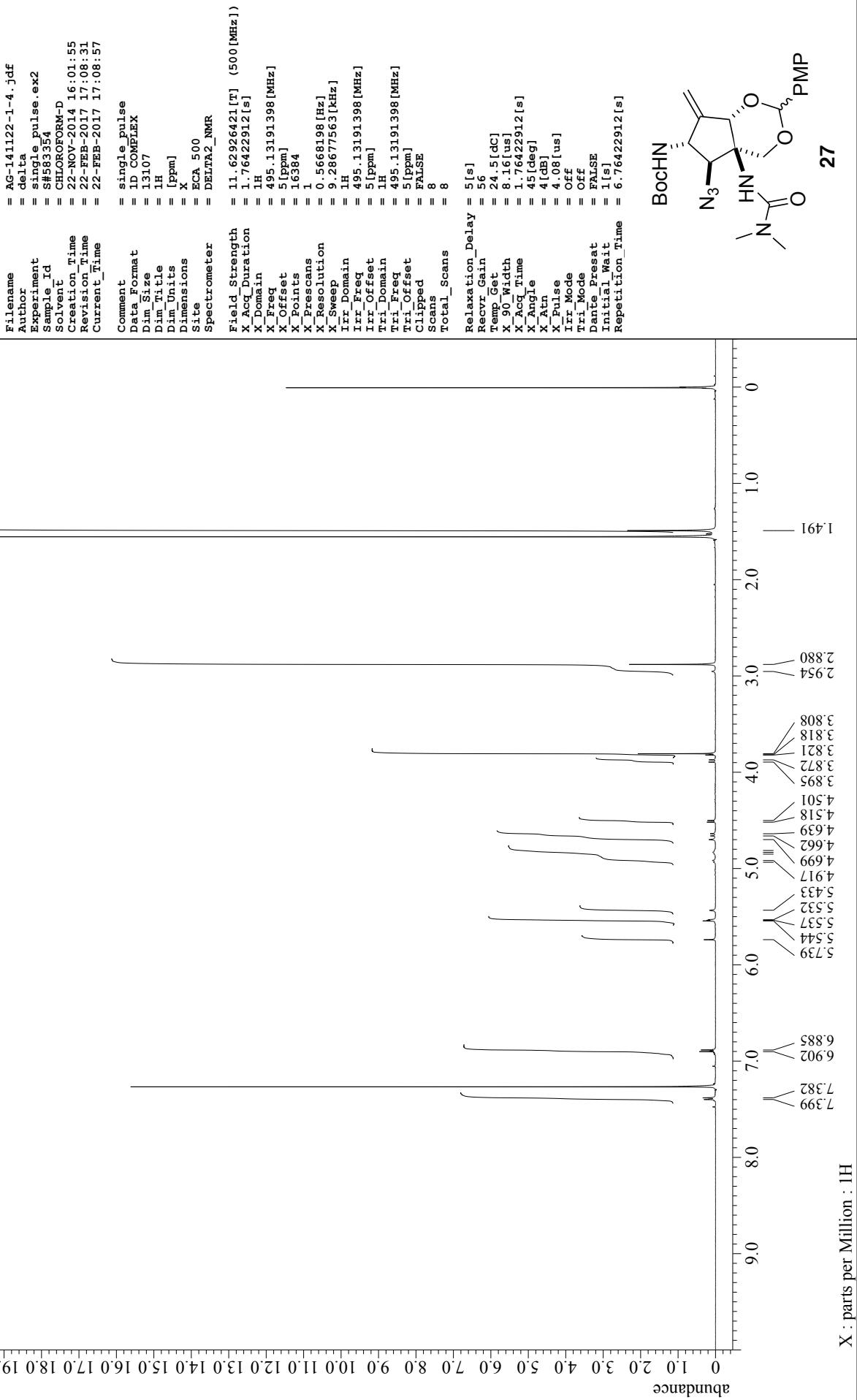
Filename = 170222-8_crystal_C-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = S1863482
Solvent = CHLOROFORM-D
Revision_Time = 23-FEB-2017 08:53:42
Current_Time = 16-MAR-2017 17:44:03
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = 13C
Dim_Units = [ppm]
Dimensions = X
Site = ECA_500
Spectrometer = DELTA2_NMR

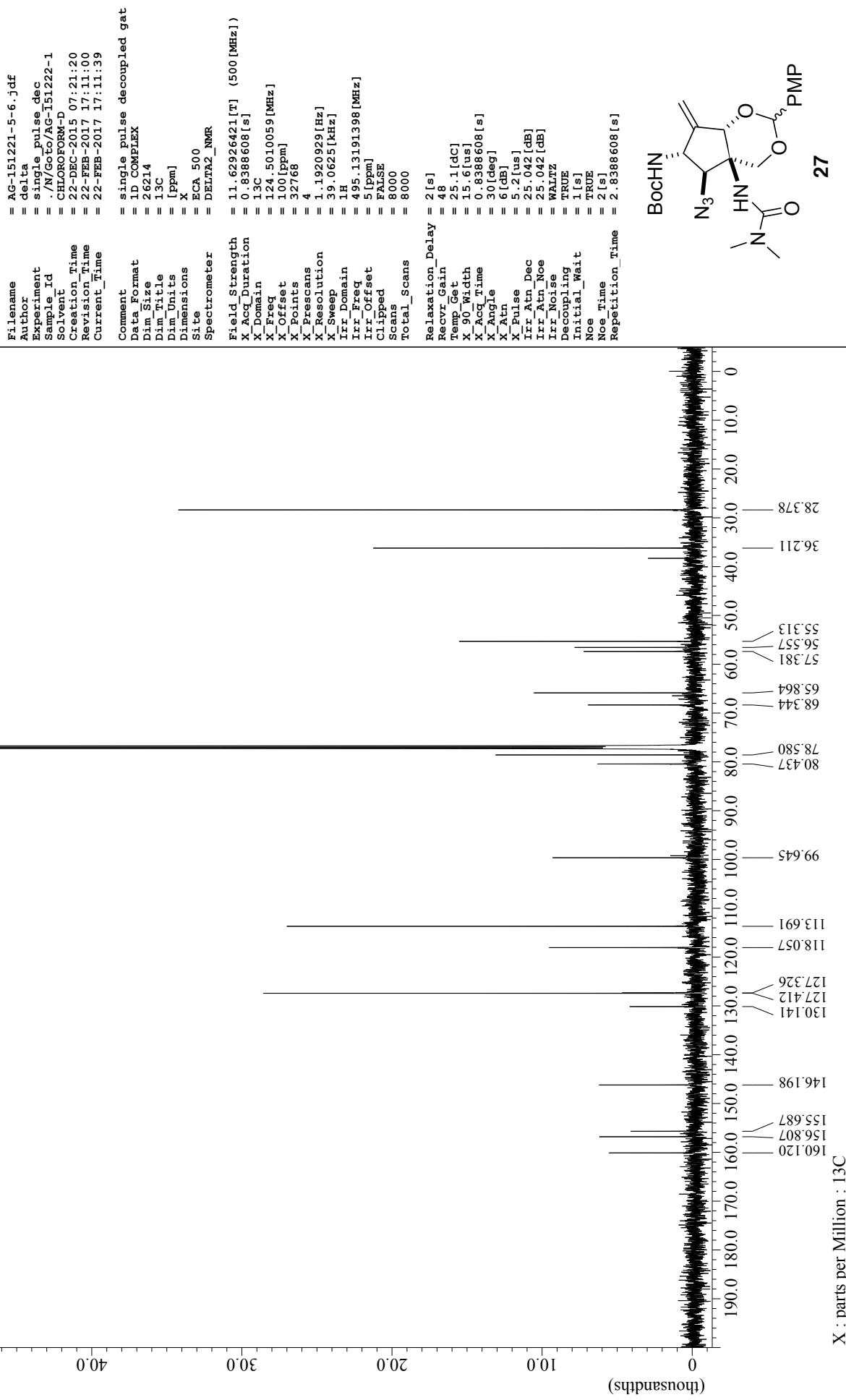
Field_Strength = 11.62926421 [T] (500 [MHz])
X_Acq_Duration = 0.8388608 [s]
X_Domain = 13C
X_Freq = 124.5010059 [MHz]
X_Offset = 100 [ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.1920929 [Hz]
X_Sweep = 39.0625 [kHz]
Irr_Domain = 1H
Irr_Freq = 495.13191398 [MHz]
Irr_Offset = 5 [ppm]
ClipSpeed = FALSE
Scans = 12000
Total_Scans = 12000

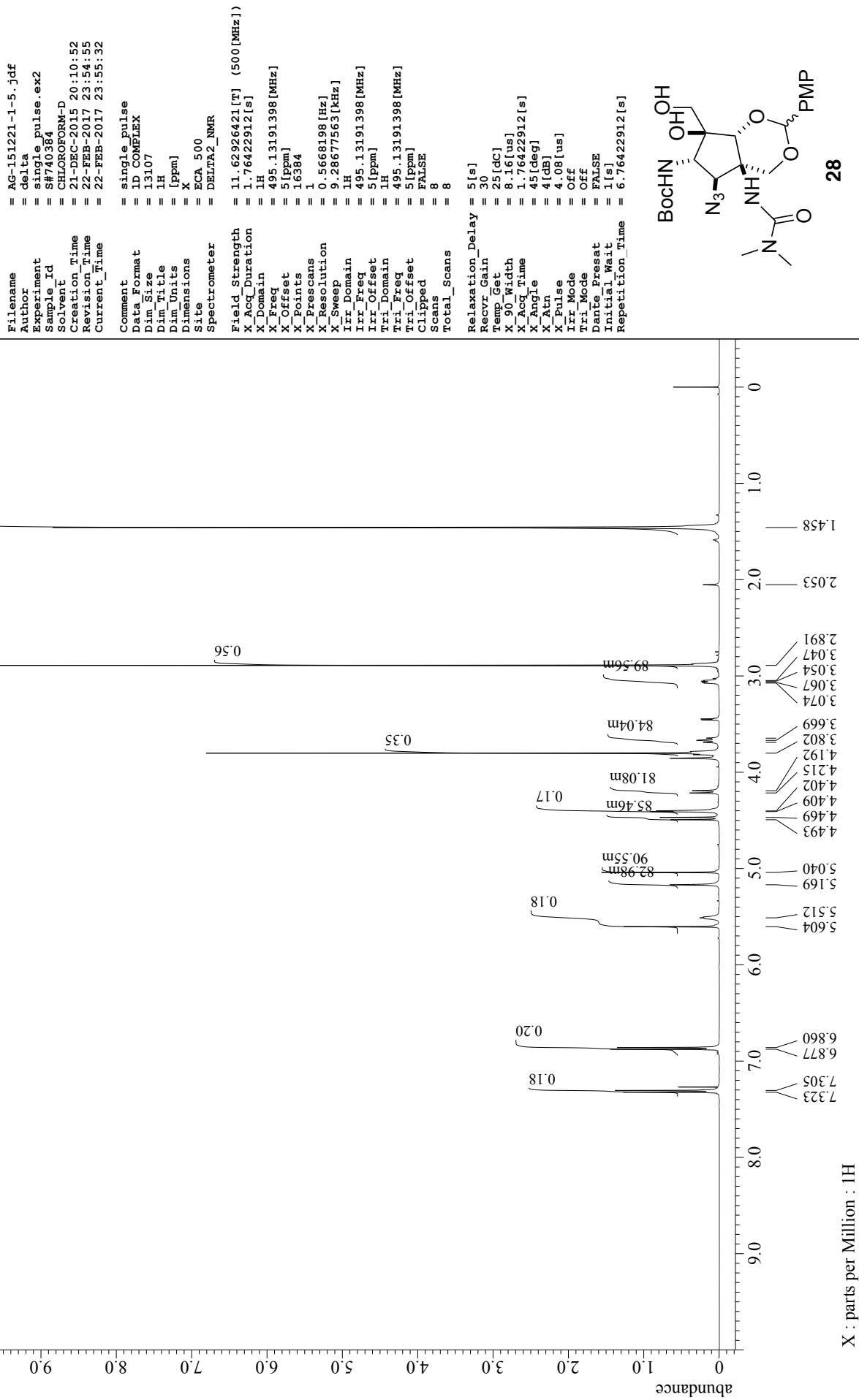
Relaxation_Delay = 2 [s]
Recv_Gain = 50
Temp_Get = 21.3 [dC]
X_90_Width = 15.6 [us]
X_Acq_Time = 0.8388608 [s]
X_Angle = 30 [deg]
X_Atn = 6 [dB]
X_Pulse = 5.2 [us]
Irr_Atn_Dec = 23.2 [dB]
Irr_Atn_Noe = 23.2 [dB]
Irr_Noise = WALTZ
Decoupling = TRUE
Initial_Wait = 1 [s]
Noe = TRUE
Noe_Time = 2 [s]
Repetition_Time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

```









```

File_name = AG-151221-2-5.jdf
Author = delta
Experiment = single_pulse_dec
Sample_Id = S174515
Solvent = CHLOROFORM-D
Revision_Time = 21-FEB-2015 21:01:08
Current_Time = 22-FEB-2017 23:57:11
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]
ClipSpeed = FALSE
Scans = 1000
Total_Scans = 1000

Relaxation_Delay = 2 [s]
Recv_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]

```

