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Electronic Supplementary Information

Pd-catalyzed enantioselective intramolecular α-arylation of α-substituted cyclic ketones: Facile synthesis of functionalized chiral spirobicycles

Lulu Fan, Shinobu Takizawa,* Yoshiki Takeuchi, Kazuhiro Takenaka and Hiroaki Sasai*

The Institute of Scientific and Industrial Research (ISIR), Osaka University

Mihogaoka, Ibaraki-shi, Osaka 567-0047, Japan.
E-mail: taki@sanken.osaka-u.ac.jp

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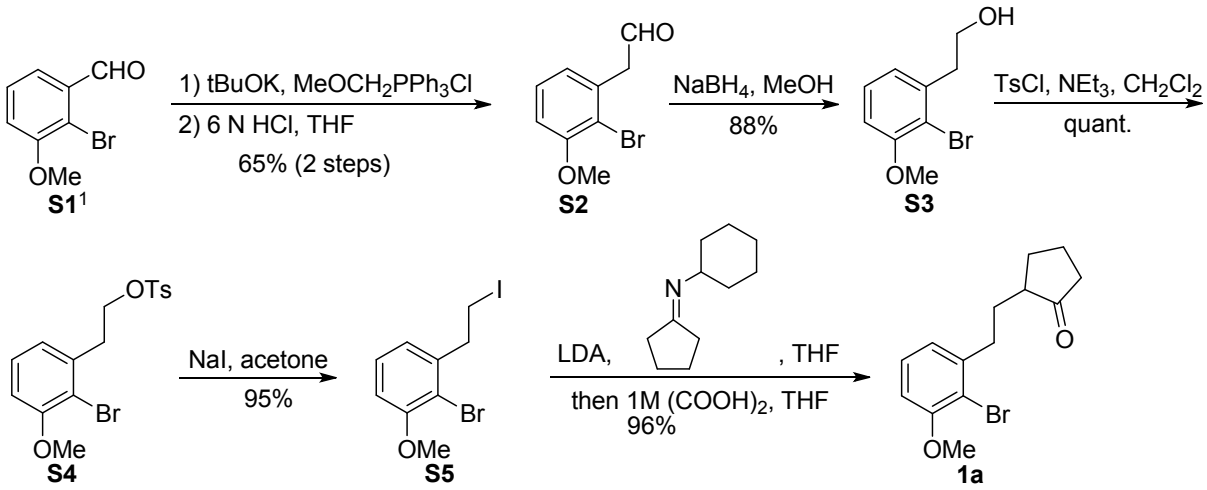
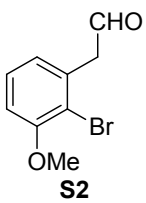
General Information

^{1}H-, ^{13}C- and ^{31}P-NMR spectra were recorded on JEOL JMN ECS400 FT-NMR spectrometer or JMN ECA600 FT-NMR spectrometer (400 MHz for ^{1}H-NMR, 100 MHz for ^{13}C-NMR, and 243 MHz for ^{31}P-NMR) using $CDCl_3$ as solvent with TMS as an internal standard (for ^{1}H- and ^{13}C- NMR) or H_3PO_4 as external standard (for ^{31}P-NMR). ^{1}H-NMR spectra are reported as follows: chemical shift in ppm relative to the chemical shift of $CHCl_3$ at 7.26 ppm, integration, multiplicities (s = singlet, d = doublet, q = quartet, t = triplet, m = multiplet), and coupling constants (Hz). ^{13}C-NMR spectra reported in ppm relative to the central line of triplet for $CDCl_3$ at 77 ppm. FT-MS spectra were obtained with LTQ Orbitrap XL (Thermo Fisher Scientific). ESI-MS spectra were obtained with JMS-T100LC (JEOL). FAB-MS spectra were obtained with JMS-700 (JEOL). Optical rotations were measured with JASCO P-1030 polarimeter. HPLC analyses were performed on a JASCO HPLC system (JASCO PU 980 pump and UV-975 UV/Vis detector) using a mixture of hexane and 2-propanol as eluents. Mp was measured with SHIMADZU DSC-60. Column chromatography on SiO_2 was performed with Kanto Silica Gel 60 (40-100 μm). Commercially available organic and inorganic compounds were used without further purification except for the solvent, which was distilled from sodium/benzophenone or CaH_2.

Preparation of α-substituted cyclic ketones 1

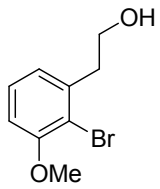
Preparation of 1a from S1[1]





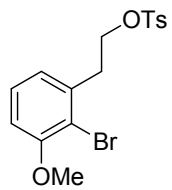
Preparation of S2: To the solution of $MeOCH_2PPh_3Cl$ (17.8 g, 52 mmol) in anhydrous THF (80 mL) was added ^{t}BuOK (5.8 g, 52 mmol) at 0 ˚C under N_2. The suspension was allowed to stir at 0 ˚C for 1 h, and then **S1**[1] (9.3 g, 43 mmol) in anhydrous THF (20 mL) was added dropwise. The mixture was stirred at room temperature for additional 1 h, and then saturated aqueous NH_4Cl solution was added and stirring continued for 5 min. The mixture was extracted three times with EtOAc. The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The residue was dissolved in ether (30 mL) and stirred for 10 min. The insoluble compound was filtered and the mother liquor was

concentrated in vacuo. The crude product was then treated with 6 N HCl (25 mL) in THF (65 mL) for 4 h under reflux condition. The mixture was allowed to cool to room temperature and water (30 ml) was added to the mixture. The organic layer was separated and the aqueous layer was extracted three times with EtOAc. The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was purified by silica column chromatography to afford **S2** in 65% overall yield from **S1**; colorless oil; ^{1}H-NMR ($CDCl_3$): δ 3.89 (d, *J* = 1.8 Hz, 2H), 3.92 (s, 3H), 6.85-6.88 (m, 2H), 7.28 (t, *J* = 7.8 Hz, 1H), 9.75 (t, *J* = 1.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 50.5, 56.2, 110.8, 114.3, 123.4, 128.1, 134.4, 156.3, 198.3; HRMS (ESI): calcd for $C_9H_9BrNaO_2$, m/z 250.9684 ([M+Na]$^+$); found, m/z 250.9677.



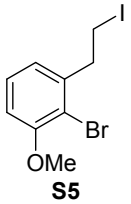
S3

Preparation of S3: To the solution of **S2** (8.2 g, 36 mmol) in dry MeOH (60 mL) was added $NaBH_4$ (1.4 g, 36 mmol) at 0˚C under N_2. The mixture was then stirred at room temperature for 2 h. The solvent was removed under reduced pressure. The residue was treated with 10% HCl and extracted with EtOAc. The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was purified by silica column chromatography to afford product **S3** in 88% yield; colorless oil; ^{1}H-NMR ($CDCl_3$): δ 1.47 (s, 1H), 3.07 (t, *J* = 6.9 Hz, 2H), 3.86-3.91 (m, 5H), 6.80 (dd, *J* = 1.4, 8.2 Hz, 1H), 6.90 (dd, *J* = 1.4, 7.8Hz, 1H), 7.21 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 39.5, 56.3, 62.0, 110.0, 114.1, 123.2, 127.8, 139.5, 156.1; HRMS (ESI): calcd for $C_9H_{11}BrNaO_2$, m/z 252.9840 ([M+Na]$^+$); found, m/z 252.9833.



S4

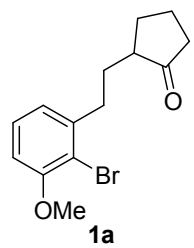
Preparation of S4: To the solution of **S3** (9.0 g, 39 mmol), TsCl (11.1 g, 58 mmol) in dry CH_2Cl_2 (70 mL) was added Et_3N (8.2 mL, 58 mmol) at 0˚C under N_2. The reaction mixture was stirred at room temperature overnight. Water (50 ml) was added. The organic layer was separated and the aqueous layer was extracted three times with CH_2Cl_2. The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was purified by silica column chromatography to afford desired product **S4** quantitatively; colorless oil; ^{1}H-NMR ($CDCl_3$): δ 2.42 (s, 3H), 3.11 (t, *J* = 7.3 Hz, 2H), 3.86 (s, 3H), 4.24 (t, *J* = 6.9 Hz, 2H), 6.77 (d, *J* = 8.2 Hz, 1H), 6.82 (d, *J* = 7.8 Hz, 1H), 7.17 (t, *J* = 7.8 Hz, 1H), 7.26 (d, *J* = 8.7 Hz, 2H), 7.68 (d, *J* = 8.2 Hz, 2H); ^{13}C-NMR ($CDCl_3$): δ 21.6, 36.0, 56.3, 68.7, 110.5, 113.9, 123.5, 127.9, 128.0, 129.7, 132.9, 137.2, 144.6, 156.1; HRMS (ESI): calcd for $C_{16}H_{17}BrNaO_4S$, m/z 406.9929 ([M+Na]$^+$); found, m/z 406.9927.



S5

Preparation of S5: A mixture of **S4** (10.9 g, 282 mmol) and NaI (6.4 g, 424 mmol) in acetone was refluxed for 6 h. The reaction mixture was cooled to room temperature and the solvent was evaporated. The residue was treated with water and extracted with EtOAc. The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. Crystallization (CH_2Cl_2/hex = 5/95) to get main

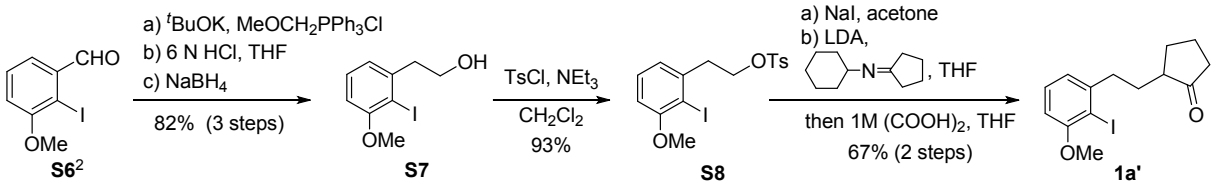
product, the remaining residue was purified by silica column chromatography to afford **S5** in 95% yield; white solid; Mp 65-66 °C; ^{1}H-NMR ($CDCl_3$): δ 3.32-3.39 (m, 4H), 3.89 (s, 3H), 6.83 (d, *J* = 8.2 Hz, 1H), 6.87 (d, *J* = 7.3 Hz, 1H), 7.23 (t, *J* = 8.2 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 3.1, 40.9, 56.4, 110.4, 113.5, 122.7, 128.0, 141.6, 156.2; HRMS (ESI): calcd for $C_9H_{10}BrINaO$, m/z 362.8857 ([M+Na]$^+$); found, m/z 362.8845.



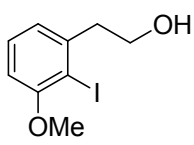
1a

Preparation of 1a: *n*-BuLi (0.54 mL, 2.6 M, 1.40 mmol) was added to the solution of *i*-Pr_2NH (0.25 mL, 1.79 mmol) in THF (5 mL) at 0 ˚C under N_2 and the mixture was stirred for 30 min. The solution of *N*-cyclohexylidenecyclohexylamine (251 mg, 1.4 mmol) in THF (2 mL) was added dropwise to this mixture and the whole was stirred at 0 ˚C for 1 h. The mixture was cooled to -78 ˚C and a THF (2 mL) solution of **S5** (341 mg, 1.00 mmol) was added dropwise. After stirring at -78 ˚C for 1 h, saturated aqueous NH_4Cl solution was added and the whole was extracted with EtOAc. The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. Oxalic acid in H_2O (1M, 1.5 mL, 1.5 mmol) was added to a solution of the residue in THF (6 mL) and the mixture was stirred at room temperature overnight. Water was added and the whole was extracted with EtOAc. The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was purified by silica column chromatography to give product **10a** in 96% yield; pale yellow oil; ^{1}H-NMR ($CDCl_3$): δ 1.54-1.68 (m, 2H), 1.73-1.85 (m, 1H), 1.99-2.18 (m, 4H), 2.27-2.34 (m, 2H), 2.78-2.91 (m, 2H), 3.89 (s, 3H), 6.75 (dd, *J* = 1.4, 8.2 Hz, 1H), 6.87 (dd, *J* = 1.4, 7.8 Hz), 7.19 (t, *J* = 8.2 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 20.6, 29.6, 29.8, 34.1, 38.0, 48.5, 56.2, 109.4, 113.6, 122.2, 127.7, 142.7, 155.9, 220.8; HRMS (ESI): calcd for $C_{14}H_{17}BrNaO_2$, m/z 319.0310 ([M+Na]$^+$); found, m/z 319.0298.

Preparation of 1a' from S6[2]

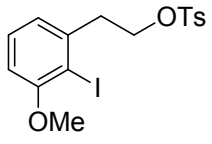


S6[2] **S7** **S8** **1a'**



S7

Preparation of S7: The procedure followed that for preparation of compound **S2** and **S3**. The crude Wittig compound was used for the next reaction without purification. 82% yield overall yield from **S6**[2]; yellow solid; Mp 69-70 °C; ^{1}H-NMR ($CDCl_3$): δ 3.08 (t, *J* = 6.8 Hz, 2H), 3.84 (t, *J* = 6.8 Hz, 2H), 3.87 (s, 3H), 6.89 (d, *J* = 8.4 Hz, 1H), 7.06 (d, *J* = 8.4 Hz, 1H), 7.22 (t, *J* = 8.4, 1 H); ^{13}C-NMR ($CDCl_3$): δ 43.9, 56.4, 62.1, 93.0, 108.9, 122.7, 128.9, 142.9, 158.2; HRMS (ESI): calcd for $C_9H_{11}INaO_2$, m/z 300.9701 ([M+Na]$^+$); found, m/z 300.9695.



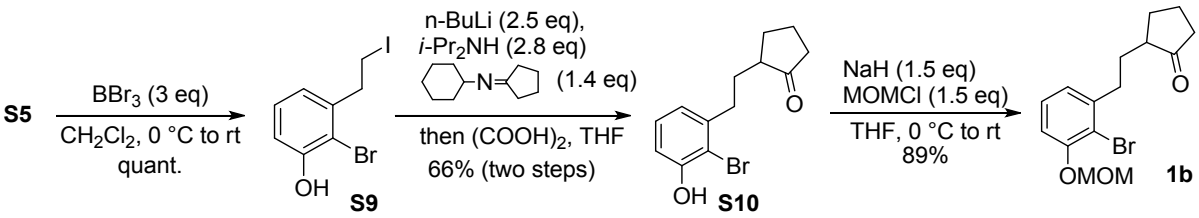
S8

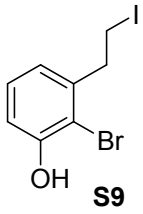
Preparation of S8: The procedure followed that for preparation of compound **S4**. 93% yield; white solid; Mp 86-87 °C; ^{1}H-NMR ($CDCl_3$): δ 2.43 (s, 3H), 3.14 (t, *J* =

7.2 Hz, 2H), 3.86 (s, 3H), 4.21 (t, *J* = 7.2 Hz, 2H), 6.67 (d, *J* = 8.0 Hz, 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 7.20 (t, *J* = 8.0 Hz, 1H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.69 (d, *J* = 8.0, 2H); ^{13}C-NMR ($CDCl_3$): δ 21.6, 40.2, 56.5, 68.9, 92.6, 109.3, 123.1, 127.9, 129.1, 129.7, 132.7, 140.6, 144.6, 158.2; HRMS (ESI): calcd for $C_{16}H_{17}INaO_4S$. m/z 454.9790 ([M+Na]$^+$); found, m/z 454.9780.

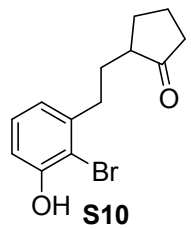
Preparation of 1a': The procedure followed that for preparation of compound **S5** and **1a**. 67% overall yield from **S8**; pale yellow oil; ^{1}H-NMR ($CDCl_3$): δ 1.52-1.72 (m, 2H), 1.74-1.87 (m, 1H), 1.99-2.20 (m, 4H), 2.27-2.37 (m, 2H), 2.79-2.92 (m, 2H), 3.88 (s, 3H), 6.66 (t, *J* = 8.4, 1.4 Hz, 1H), 6.88 (dd, *J* = 8.4, 1.4 Hz, 2H), 7.21 (t, *J* = 8.4 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 221.1, 158.1, 146.4, 129.0, 121.9, 108.5, 92.7, 56.5, 48.6, 39.0, 38.2, 30.2, 29.7, 20.8; HRMS (ESI): calcd for $C_{14}H_{17}INaO_2$. m/z 367.0171 ([M+Na]$^+$); found, m/z 367.0164.

Preparation of 1b from S5

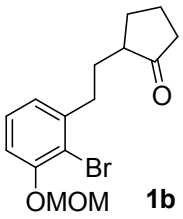




Preparation of S6: To a solution of **S5** (1.08 g, 3.2 mmol) in dry CH_2Cl_2 was added dropwise BBr_3 (9.5 mL, 1M, 9.5 mmol) at 0 ˚C under N_2 and the mixture was stirred at room temperature for 12 h. H_2O was added to the reaction mixture slowly to quench the reaction. Then the reaction mixture was extracted with CH_2Cl_2 and the combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was purified by silica column chromatography to give desired product **S9** quantitatively; white solid; Mp 84-86 ºC; ^{1}H-NMR ($CDCl_3$): δ 3.27-3.37 (m, 4H), 5.63 (s, 1H), 6.82 (dd, *J* = 1.4, 7.3 Hz, 1H), 6.95 (dd, *J* = 1.8, 8.2 Hz, 1H), 7.18 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 2.6, 40.8, 112.3, 114.7, 122.3, 128.6, 140.6, 152.6; HRMS (ESI): calcd for $C_8H_7BrIONa_2$, m/z 370.8520 ([M+2Na-H]$^+$); found, m/z 370.8511.

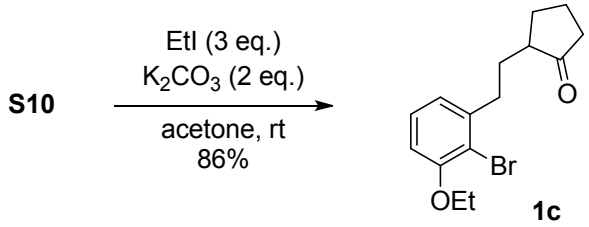


Preparation of S7: The procedure followed that for preparation of **1a**; 66% yield; white solid; Mp 85-86 ºC; ^{1}H-NMR ($CDCl_3$): δ 1.53-1.66 (m, 2H), 1.74-1.86 (m, 1H), 1.99-2.19 (m, 4H), 2.26-2.36 (m, 2H), 2.74-2.86 (m, 2H), 5.71 (s, 1H), 6.81 (dd, *J* = 1.4, 7.8 Hz, 1H), 6.88 (dd, *J* = 1.4, 7.8 Hz, 1H), 7.13 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 20.7, 29.7, 29.8, 34.2, 38.1, 48.5 112.7, 113.6, 121.9, 128.3, 141.8, 152.4, 221.1; HRMS (ESI): calcd for $C_{13}H_{15}BrNaO_2$, m/z 305.0153 ([M+Na]$^+$); found, m/z 305.0154.



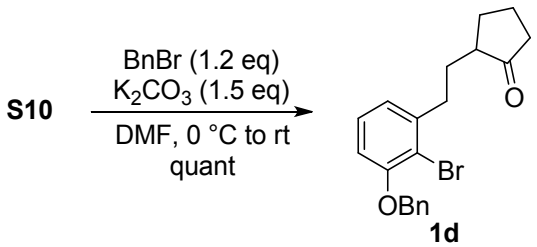
Preparation of 1b: To a solution of **S10** (0.604 g, 2.1 mmol) in anhydrous THF was added NaH (0.128 g, 3.2 mmol) at 0 ˚C. The reaction mixture was allowed to stir at 0 ˚C for 30 min. Chloromethyl methyl ether (0.24 mL, 3.2 mmol) was added dropwise to the mixture at 0 ˚C. The reaction mixture was stirred at room temperature for 4 h. H_2O was added and the reaction mixture was extracted with EtOAc and the combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was purified by silica column chromatography to give desired product **1b** in 89% yield; colorless oil; ^{1}H-NMR ($CDCl_3$): δ 1.53-1.68 (m, 2H), 1.74-1.86 (m, 1H), 1.99-2.19 (m, 4H), 2.24-2.36 (m, 2H), 2.77-2.90 (m, 2H), 3.52 (s, 3H), 5.24 (s, 2H), 6.92 (dd, *J* = 1.4, 7.8 HZ, 1H), 6.99 (dd, *J* = 1.8, 8.2 Hz, 1H), 7.16 (t, *J* = 8.2 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 20.7 29.7, 29.9, 34.3, 38.2, 48.6, 56.4, 95.1, 113.6, 115.0, 123.5, 127.7, 142.9, 153.9, 221.1; HRMS (ESI): calcd for $C_{15}H_{19}BrNaO_3$, m/z 349.0415 ($[M+Na]^+$); found, m/z 349.0408.

Preparation of 1c



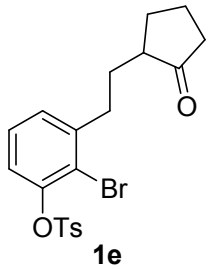
To a solution of **S10** (114 mg, 0.4 mmol), K_2CO_3 (110 mg, 0.8 mmol) in acetone (2 mL) was added ethyl iodide (0.24 mL, 1.2 mmol) at room temperature under N_2. The reaction mixture was stirred for 16 h. The insoluble compound was filtered and the mother liquor was concentrated. The reaction mixture was purified by silica column chromatography to afford **1b** in 86% yield; pale yellow oil; ^{1}H-NMR ($CDCl_3$): δ 1.47 (t, *J* = 7.0 Hz, 3 H), 1.51-1.70 (m, 2 H), 1.71-1.90 (m, 1 H), 1.95-2.21 (m, 4 H), 2.73-2.94 (m, 2H), 4.09 (q, *J* = 7.0 Hz, 2H), 6.73 (dd, *J* = 7.8, 1.4 Hz, 1H), 6.85 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.16 (t, *J* = 7.8 Hz, 1 H); ^{13}C-NMR ($CDCl_3$): δ 14.7, 20.7, 29.7, 29.9, 34.3, 38.2, 48.6, 64.9, 110.8, 114.3, 122.2, 127.7, 142.8, 155.5, 221.1; HRMS (ESI): calcd for $C_{15}H_{19}BrNaO_2$, m/z 333.0466 ($[M+Na]^+$); found, m/z 333.0456.

Preparation of 1d



A round flask was charged with **S10** (239 mg, 0.84 mmol), K_2CO_3 (175 mg, 1.26 mmol) and DMF (5 mL). To the mixture was added BnBr (0.12 mL, 1.01 mmol) dropwise at 0 ˚C. Then the mixture was allowed to stir at room temperature for 2 h. H_2O was added and continued to stir for 10 min. The reaction mixture was

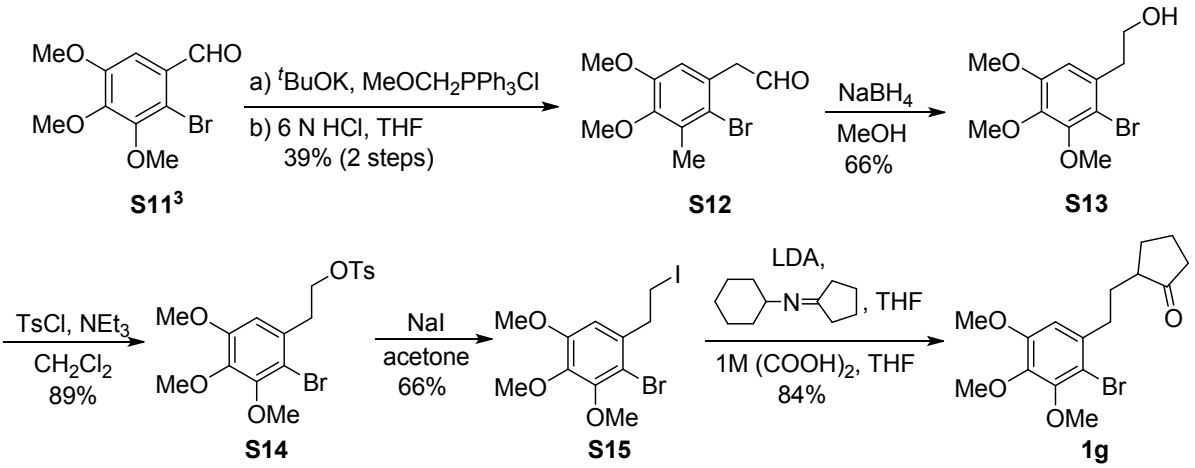
then extracted with EtOAc and the combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was purified by silica column chromatography to give **1d** quantitatively; colorless oil; ^{1}H-NMR ($CDCl_3$): δ 1.55-1.68 (m, 2H), 1.73-1.85 (m, 1H), 1.99-2.19 (m, 4H), 2.27-2.35 (m, 2H), 2.80-2.93 (m, 2H), 5.14 (s, 2H), 6.79 (dd, *J* = 1.4, 7.8 Hz, 1H), 6.89 (dd, *J* = 1.4, 7.8 Hz, 1H), 7.15 (t, *J* = 7.8 Hz, 1H), 7.30-7.34 (m, 1H), 7.39 (t, *J* = 7.3 Hz, 2H), 7.49 (d, *J* = 7.3 Hz, 2H); ^{13}C-NMR ($CDCl_3$): δ 20.7, 29.7, 29.9, 34.2, 38.1, 48.6, 70.8, 111.3, 114.6, 122.6, 126.9, 127.6, 127.8, 128.5, 136.6, 143.0, 155.1, 221.0; HRMS (ESI): calcd for $C_{20}H_{21}BrNaO_2$, m/z 395.0623 ([M+Na]$^+$); found, m/z 395.0617.

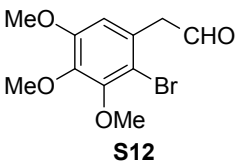


1e

Preparation of 1e: The procedure followed that for preparation of compound **S4**. 82% yield; pale yellow oil. ^{1}H-NMR ($CDCl_3$): δ 1.44-1.62 (m, 3H), 1.72-1.85 (m, 1H), 1.93-2.18 (m, 4H), 2.22-2.36 (m, 2H), 2.46 (s, 3H), 2.72-2.85 (m, 2H), 7.12-7.23 (m, 3H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.78 (d, *J* = 8.5 Hz, 2H); ^{13}C-NMR ($CDCl_3$): δ 20.7, 21.7, 29.7, 29.8, 34.3, 38.9, 48.5, 118.7, 121.4, 127.8, 128.3, 128.7, 129.7, 132.7, 143.8, 145.6, 147.1, 220.8; HRMS (ESI): calcd for $C_{20}H_{21}BrNaO_4S$ m/z 459.0242 ([M+Na]$^+$); found, m/z 459.0229.

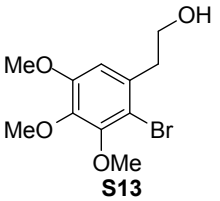
Preparation of 1g from S11[3]





S12

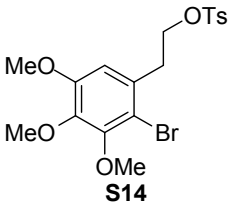
Preparation of S12: The procedure followed that for preparation of compound **S2**; 75% yield; colorless oil; ^{1}H-NMR ($CDCl_3$) δ 3.89-3.78 (m, 11H), 6.56 (s, 1H), 9.71 (t, *J* = 1.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 50.6, 56.0, 60.9, 61.0, 110.1, 111.2, 127.8, 142.4, 151.2, 152.8, 198.3; HRMS (ESI): calcd for $C_{11}H_{13}BrNaO_4$, m/z 310.9895 ([M+Na]$^+$); found, m/z 310.9887.



S13

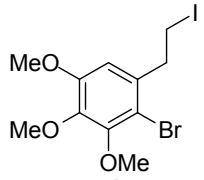
Preparation of S13: The procedure followed that for preparation of compound **S3**; 84% yield; pale yellow oil. ^{1}H-NMR ($CDCl_3$): δ 1.65 (s, 1H), 2.95-3.02 (m, 2H),

3.82-3.90 (m, 11H), 6.64 (s, 1H); ^{13}C-NMR ($CDCl_3$): δ 39.6, 56.1, 60.9, 61.0, 62.0, 109.9, 110.7, 133.3,141.7, 150.9, 152.4; HRMS (ESI): calcd for$C_{11}H_{15}BrNaO_4$, m/z 313.0051 ([M+Na]$^+$); found, m/z 313.0047.



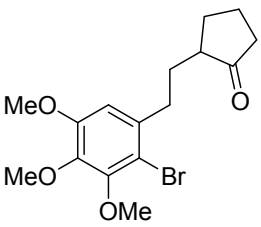
S14

Preparation of S14: The procedure followed that for preparation of compound **S4**; 94% yield; colorless oil. ^{1}H-NMR ($CDCl_3$): δ 2.42 (s, 3H), 3.07 (t, J = 6.8 Hz, 2H), 3.77-3.93 (m, 9H), 4.23 (t, J = 6.9 Hz, 2H), 6.57 (s, 1H), 7.29 (d, J = 8.2 Hz, 2H), 7.70 (d, *J* = 8.2 Hz, 2H); ^{13}C-NMR ($CDCl_3$): δ 21.6, 35.9, 56.1. 60.9, 61.0, 68.8, 110.0, 110.5, 127.7, 129.7, 130.9, 132.8, 142.1, 144.8, 150.9, 152.5; HRMS (ESI): calcd for $C_{18}H_{21}BrNaO_6S$, m/z 467.0140 ([M+Na]$^+$); found, m/z 467.0134.



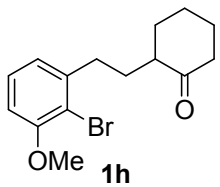
S15

Preparation of S15: The procedure followed that for preparation of compound **S5**; quantitative yield; pale yellow oil. ^{1}H-NMR ($CDCl_3$): δ 3.23-3.30 (m, 2H), 3.31-3.38 (m, 2H), 3.84-3.91 (m, 9H), 6.61 (s, 1H); ^{13}C-NMR ($CDCl_3$): δ 3.4, 41.0, 56.1, 60.99, 61.04, 109.4, 110.3, 135.4. 142.1, 151.1, 152.6; HRMS (ESI): calcd for $C_{11}H_{14}BrINaO_3$, m/z 422.9069 ([M+Na]$^+$); found, m/z 422.9060.



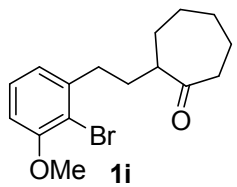
1g

Preparation of 1g-l: The procedure followed that for preparation of compound **1a**; 39% yield; orange oil. ^{1}H-NMR ($CDCl_3$): δ 1.52-1.74 (m, 3H), 1.74-1.88 (m, 1H), 1.97-2.22 (m, 4H), 2.26-2.38 (m, 2H), 2.70-2.85 (m, 2H), 3.81-3.91 (m, 9H), 6.61 (s, 1H); ^{13}C-NMR ($CDCl_3$): δ 20.7, 29.7, 30.2, 34.3, 38.2, 48.6, 56.1, 60.9, 61.1, 108.8, 110.4, 136.8, 141.3, 150.8, 152.5, 221.2; HRMS (ESI): calcd for $C_{16}H_{21}BrNaO_4$, m/z 379.0521 ([M+Na]$^+$); found, m/z 379.0510.



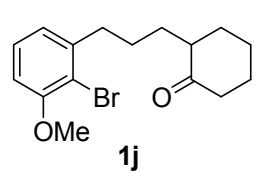
1h

1h: 97% yield; colorless oil; ^{1}H-NMR ($CDCl_3$): δ 1.38-1.54 (m, 2H), 1.59-1.73 (m, 2H), 1.80-1.86 (m, 1H), 2.00-2.18 (m, 3H), 2.25-2.42 (m, 3H), 2.71-2.82 (m, 2H), 3.86 (s, 3H), 6.72 (dd, *J* = 1.1, 7.8 Hz, 1H), 6.84 (dd, *J* = 1.1, 7.8 Hz, 1H), 7.16 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 24.8, 28.0, 29.6, 33.7, 34.0, 42.0, 50.0, 56.2, 109.3, 113.7, 122.3, 127.7, 143.3, 155.9, 213.0; HRMS (ESI): calcd for $C_{15}H_{19}BrNaO_2$, m/z 333.0466 ([M+Na]$^+$); found, m/z 333.0459.



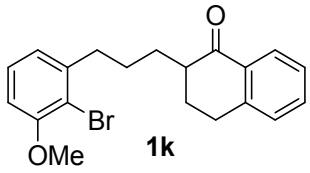
1i

1i: pale yellow oil; ^{1}H-NMR ($CDCl_3$): δ 1.26-1.46 (m, 3H), 1.56-1.69 (m, 2H), 1.83-2.03 (m, 5H), 2.44-2.59 (m, 3H), 2.69-2.80 (m, 2H), 3.88 (s, 3H), 6.74 (dd, *J* = 0.9, 8.2 Hz, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 7.18 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 24.4, 28.5, 29.5, 31.4, 32.3, 34.0, 42.9, 51.6, 56.3, 109.4, 113.7, 122.5, 127.7, 143.2, 156.0, 216.0; HRMS (ESI): calcd for $C_{16}H_{21}BrNaO_2$, m/z 347.0623 ([M+Na]$^+$); found, m/z 347.0614.



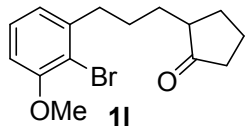
1j

1j: 82% yield; white solid; Mp 53-54 °C; ^{1}H-NMR ($CDCl_3$): δ 1.20-1.39 (m, 2H), 1.52-1.69 (m, 4H), 1.78-1.89 (m, 2H), 1.94-2.02 (m, 1H), 2.06-2.10 (m, 1H), 2.21-2.37 (m, 3H), 2.66-2.78 (m, 2H), 3.84 (s, 3H), 6.70 (dd, *J* = 1.4, 7.8 Hz, 1H), 6.81 (dd, *J* = 1.4, 7.38Hz, 1H), 7.14 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 24.7, 27.2, 27.9, 28.9, 33.7, 36.3, 41.8, 50.4, 56.1, 109.1, 113.5, 122.2, 127.5, 143.3, 155.7, 213.1; HRMS (ESI): calcd for $C_{16}H_{21}BrNaO_2$, m/z 347.0623 ([M+Na]$^+$); found, m/z 347.0616.



1k

1k: 30% yield; pale yellow solid; Mp 79 °C; ^{1}H-NMR ($CDCl_3$): δ 1.53-1.62 (m, 1H), 1.65-1.79 (m, 2H), 1.81-1.92 (m, 1H), 2.02-2.10 (m, 1H), 2.20-2.27 (m, 1H), 2.47-2.54 (m, 1H), 2.75-2.88 (m, 2H), 2.95-2.98 (m, 2H), 3.87 (s, 3H), 6.73 (dd, *J* = 1.4, 8.0 Hz, 1H), 6.87 (dd, *J* = = 1.4, 8.0Hz, 1H), 7.16-7.22 (m, 2H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.44 (td, *J* = 1.4, 7.5 Hz, 1H), 8.02 (dd, *J* = 0.9, 7.5Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 27.1, 28.1, 28.3, 29.0, 36.4, 47.2, 56.1, 109.2, 113.6, 122.3, 126.4, 127.3, 127.6, 128.5, 132.3, 133.0, 143.2, 143.8, 155.8, 200.0; HRMS (ESI): calcd for $C_{20}H_{21}BrNaO_2$, m/z 395.0623 ([M+Na]$^+$); found, m/z 395.0616.

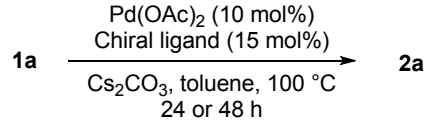


1l

1l: 93% yield; pale yellow oil; ^{1}H-NMR ($CDCl_3$): δ 1.20-1.34 (m, 1H), 1.43-1.53 (m, 1H), 1.59-1.67 (p, *J* = 7.8, 16.0 Hz, 2H), 1.68-1.86 (m, 2H), 1.91-2.10 (m, 3H), 2.16-2.29 (m, 2H), 2.66-2.80 (m, 2H), 3.83 (s, 3H), 6.70 (d, *J* = 7.9 Hz, 1H), 6.80 (dd, *J* = 7.9 Hz, 1H), 7.14 (t, *J* = 7.9 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 20.6, 27.6, 29.3, 29.4, 36.2, 38.0, 48.8, 56.1, 109.2, 113.5, 122.2, 127.5, 143.1, 155.8, 221.1; HRMS (ESI): calcd for $C_{15}H_{19}BrNaO_2$, m/z 333.0466 ([M+Na]$^+$); found, m/z 333.0461.

Screening of reaction conditions for Pd-catalyzed intramolecular α-arylation of 1a

Table S1 Screening of ligands for Pd-catalyzed intramolecular α-arylation reaction[a]



Entry	Ligand	Yield (%)[b]	Ee (%)[c]	Entry	Ligand	Yield (%)[b]	Ee (%)[c]
1[d]	**L1**	75	5 (*S*)	13	**L13**	27	11 (*R*)
2	**L2**	68	19 (*S*)	14	**L14**	59	18 (*R*)
3	**L3**	83	*Rac*	15	**L15**[e]	42	15 (*S*)
4	**L4**	80	*Rac*	16	**L16**[e]	38	16 (*S*)
5	**L5**	61	14 (*S*)	17	**L17**	69	*Rac*
6	**L6**	75	21 (*S*)	18	**L18**	77	46 (*R*)
7	**L7**	86	2 (*S*)	19	**L19**	67	42 (*S*)
8	**L8**	79	4 (*S*)	20	**L20**	12	61 (*S*)
9	**L9**	85	*Rac*	21	**L21**	58	83 (*S*)
10	**L10**	60	15 (*S*)	22	**L22**	5	ND[f]
11	**L11**	70	*rac*	23	**L23**	42	15 (*R*)
12	**L12**	66	11 (*R*)	24	**L24**	81	40 (*S*)

[a]Reaction conditions: **1a** (1.0 eq), $Pd(OAc)_2$ (10 mol %), ligand (15 mol %), Cs_2CO_3 (2.0 eq), toluene (0.2 M) at 100 ˚C. [b]^{1}H-NMR yield using CH_2Br_2 as an internal standard. [c]Ee was determined by HPLC (Chiralpak IA). [d]Ligand(10 mol %). [e]NaO^{t}Bu (3 eq) as base, Ligand (22 mol %). [f]Not determined.

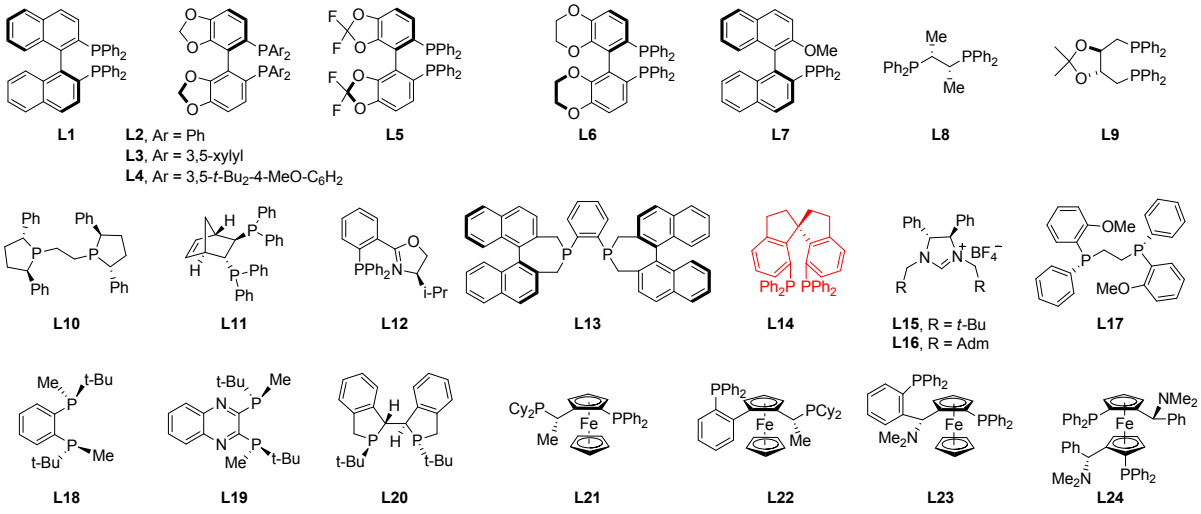
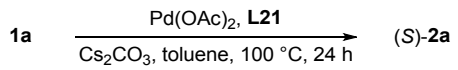


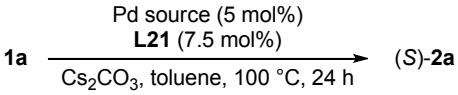
Table S2 Effect of catalyst loading[a]



Entry	$Pd(OAc)_2$ (mol %)	**L21** (mol %)	Yield (%)[b]	Ee (%)[c]
1	10	15	58	83
2	5	7.5	58	85
3	2.5	3.5	48	86

[a]Reaction conditions: **1a** (1.0 eq), $Pd(OAc)_2$/**L21** = 1/1.5, Cs_2CO_3 (2.0 eq), toluene (0.2 M) at 100 ˚C for 24 h. [b]^{1}H-NMR yield using CH_2Br_2 as an internal standard. [c]Ee was determined by HPLC (Chiralpak IA).

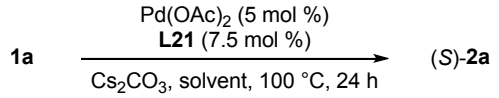
Table S3 Effect of palladium source[a]



Entry	Pd source	Yield (%)[b]	Ee (%)[c]
1	$Pd(OAc)_2$	58	85
2	$Pd(OCOCF_3)_2$	38	79
3	$Pd_2(dba)_3 \cdot CHCl_3$	44	73
4	$[Pd(allyl)Cl]_2$	52	72
5	$PdCl_2(cod)$	45	83
6	$PdCl_2(MeCN)_2$	47	83
7	$Pd(acac)_2$	79	69

[a]Reaction conditions: **1a** (1.0 eq), Pd source (5 mol %), **L21** (7.5 mol %), Cs_2CO_3 (2.0 eq), toluene (0.2 M) at 100 ˚C for 24 h. [b]^{1}H-NMR yield using CH_2Br_2 as an internal standard. [c]Ee was determined by HPLC (Chiralpak IA).

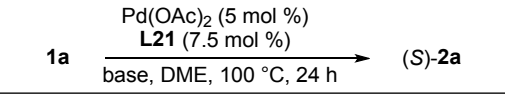
Table S4 Effect of solvent[a]



Entry	Solvent	Yield (%)[b]	Ee (%)[c]
1	Toluene	58	85
2	*t*-BuOH	84	50
3	DMF	80	25
4	MeCN	90	24
5	DME	87	79
6	1,4-dioxane	81	79
7[d]	THF	49	79
8	DMSO	41	18

[a]Reaction conditions: **1a** (1.0 eq), $Pd(OAc)_2$ (5 mol %), **L21** (7.5 mol %), Cs_2CO_3 (2.0 eq), solvent (0.2 M) at 100 ˚C for 24 h. [b]^{1}H-NMR yield using CH_2Br_2 as an internal standard. [c] Ee was determined by HPLC (Chiralpak IA). [d]70 ˚C for 48 h.

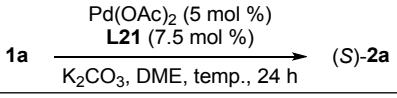
Table S5 Effect of base[a]



Entry	Base	Yield (%)[b]	Ee (%)[c]
1	Cs_2CO_3	87	79
2[d]	Na_2CO_3	3	not determined
3	K_2CO_3	90	80
4	K_3PO_4	89	77
5	NaO^{t}Bu or KO^{t}Bu	complex mixture	—

[a]Reaction conditions: **1a** (1.0 eq), $Pd(OAc)_2$ (5 mol %), **L21** (7.5 mol %), base (2.0 eq), DME (0.2 M) at 100 ˚C for 24 h. [b]^{1}H-NMR yield using CH_2Br_2 as an internal standard. [c]Ee was determined by HPLC (Chiralpak IA). [d]48 h.

Table S6 Effect of temperature[a]

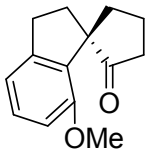


Entry	Temp. (˚C)	Yield (%)[b]	Ee (%)[c]
1	100[d]	90	80
2	90	82	83
3	80	41	83

[a]Reaction conditions: **1a** (1.0 eq), $Pd(OAc)_2$ (5 mol %), **L21** (7.5 mol %), K_2CO_3 (2.0 eq), DME (0.2 M) at temp. mentioned above for 24 h. [b]^{1}H-NMR yield using CH_2Br_2 as an internal standard. [c]Ee was determined by HPLC (Chiralpak IA).

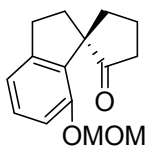
General procedure for the palladium-catalyzed enantioselective intramolecular α-arylation of 1

A test tube was charged with **1** (1 eq), $Pd(OAc)_2$ (5 mol%), (S,R_p)-Josiphos (**L21**, 7.5 mol%) and DME (0.2 M) at room temperature. The mixture was stirred at room temperature for 30 min then at 90 ˚C until the starting material was consumed completely, as indicated by TLC. The reaction mixture was then filtered through a short pad of silica gel and the organic fraction was evaporated. The crude product was purified through preparative thin layer chromatography plate or silica column chromatography to afford the desired product **2**.



2a

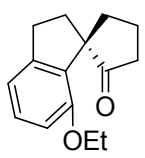
(*S*)-2a: Reaction time: 24 h; 82% yield; yellow oil; $[\alpha]_D^{18}$ = +60.9 (*c* = 0.6, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.80-1.92 (m, 1H), 1.98-2.03 (m, 1H), 2.05-2.32 (m, 4H), 2.35-2.42 (m, 1H), 2.49-2.59 (m, 1H), 2.93-3.05 (m, 2H), 3.75 (s, 3H), 6.66 (d, *J* = 7.8 Hz, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 7.16 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 20.8, 31.5, 35.3, 37.4, 38.3, 55.1, 60.2, 108.6, 116.8, 128.9, 133.9, 146.3, 154.9, 222.8; HRMS (ESI): calcd for $C_{14}H_{16}NaO_2$, m/z 239.1048 ($[M+Na]^+$); found, m/z 239.1040; Enantiomeric excess: 83%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 99/1, flow rate: 0.5 mL/min, 220 nm): major enantiomer: t_R = 21.9 min, minor enantiomer: t_R = 26.0 min.



2b

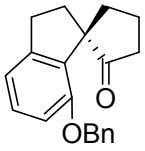
(*S*)-2b: Reaction time: 120 h; 87% yield; colorless oil; $[\alpha]_D^{28}$ = +53.8 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.83-1.94 (m, 1H), 1.99-2.05 (m, 1H), 2.08-2.19 (m, 2H), 2.20-2.30 (m, 2H), 2.37-2.44 (m, 1H), 2.48-2.57 (m, 1H), 3.41 (s, 3H), 5.08 (d, *J* = 6.9 Hz, 1H), 5.14 (d, *J* = 6.4 Hz, 1H), 6.84-6.88 (m, 2H), 7.13 (t, *J* = 7.8 Hz, 1H), ^{13}C-NMR ($CDCl_3$): δ 20.9, 31.5, 35.5, 37.5, 38.4, 56.0, 60.3, 93.6, 111.3, 117.8, 129.0, 134.3, 146.5, 152.2, 222.6; HRMS (ESI): calcd for $C_{15}H1_8NaO_3$, m/z 269.1154 ($[M+Na]^+$); found, m/z 269.1145. Enantiomeric excess: 72%, determined by HPLC (Chiralpak OD-3, *n*-hexane/*i*-PrOH = 99/1, flow rate: 0.5 mL/min, 219

nm): major enantiomer: t_R = 14.0 min, minor enantiomer: t_R = 15.8 min.



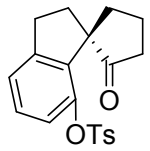
2c

(*S*)-2c: Reaction time: 16 h; 83% yield; pale yellow oil; $[\alpha]_D^{26}$ = +53.7 (*c* = 0.6, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.34 (t, *J* = 7.1 Hz, 3H), 1.77-1.93 (m, 1H), 1.94-2.45 (m, 6H), 2.46-2.63 (m, 1H), 2.88-3.08 (m, 2H), 3.87-4.10 (m, 2H), 6.63 (d, *J* = 8.0 Hz, 1H), 6.81 (d, *J* = 8.0 Hz, 1H), 7.13 (t, *J* = 8.0 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 14.6, 21.0, 31.6, 35.5, 37.5, 38.6, 60.2, 63.3, 109.3, 116.7, 128.9, 134.0, 146.3, 154.3, 222.9; HRMS (ESI): calcd for $C_{15}H_{18}NaO_2$, m/z 253.1204 ($[M+Na]^+$); found, m/z 253.1196; Enantiomeric excess: 74%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 99/1, flow rate: 1 mL/min, 205 nm): major enantiomer: t_R = 8.6 min, minor enantiomer: t_R = 13.1 min.



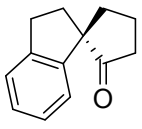
2d

(*S*)-2d: Reaction time: 72 h; quant; colorless oil; $[\alpha]_D^{29}$ = +8.7 (*c* = 1.1, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.73-1.86 (m, 1H), 1.99-2.10 (m, 3H), 2.18-2.32 (m, 4H), 2.95-3.07 (m, 2H), 5.00 (s, 2H), 6.73 (d, *J* = 8.2 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 7.16 (t, *J* = 7.8 Hz, 1H), 7.31-7.42 (m, 5H); ^{13}C-NMR ($CDCl_3$): δ 20.9, 31.6, 35.3, 37.5, 38.4, 60.3, 70.0, 109.6, 117.1, 127.7, 128.0, 128.5, 129.0, 134.1, 136.7, 146.5, 154.0, 222.9; HRMS (ESI): calcd for $C_{20}H_{20}NaO_2$, m/z 315.1361 ($[M+Na]^+$); found, m/z 315.1350. Enantiomeric excess: 83%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 95/5, flow rate: 0.5 mL/min, 220 nm): major enantiomer: t_R = 17.9 min, minor enantiomer: t_R = 36.8 min.



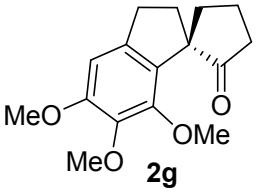
2e

(*S*)-2e: Reaction time: 48 h; 91% yield; colorless oil; $[\alpha]_D^{25}$ = +31.5 (*c* = 0.7, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.76-1.92 (m, 1H), 1.98-2.04 (m, 1H), 2.08-2.24 (m, 3H), 2.33-2.42 (m, 3H), 2.45 (s, 3H), 2.95-3.00 (m, 2H), 6.91 (dd, *J* = 1.1, 8.0 Hz, 1H), 7.05-7.12 (m, 2H), 7.36 (d, *J* = 8.2 Hz, 2H), 7.84 (d, *J* = 8.2 Hz, 2H); ^{13}C-NMR ($CDCl_3$): δ 20.7, 21.7, 31.3, 35.4, 37.7, 38.1, 60.6, 117.4, 122.4, 128.3, 128.9, 129.9, 133.4, 137.0, 145.5, 146.1, 148.1, 220.8; HRMS (ESI): calcd for $C_{20}H_{20}NaO_4S$ m/z 379.0980 ($[M+Na]^+$); found, m/z 379.0972. Enantiomeric excess: 63%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 95/5, flow rate: 1.0 mL/min, 225 nm): major enantiomer: t_R = 13.5 min, minor enantiomer: t_R = 16.8 min.



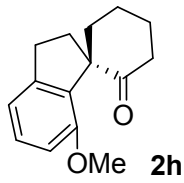
2f

(*S*)-2f[5]: Reaction time: 72 h; 50% yield; colorless oil; $[\alpha]_D^{25}$ = +2.8 (*c* = 0.5, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.95-2.07 (m, 2H), 2.12-2.53 (m, 6H), 2.92-2.99 (m, 1H), 3.04-3.11 (m, 1H), 7.15-7.22 (m, 2H), 7.24-7.26 (m, 1H); ^{13}C-NMR ($CDCl_3$): δ 19.8, 30.9, 37.1, 37.5, 37.7, 61.9, 122.8, 124.7, 126.7, 127.4, 145.5, 221.1; HRMS (ESI): calcd for $C_{13}H_{14}ONa$, m/z 209.0942 ($[M+Na]^+$); found, m/z 209.0934. Enantiomeric excess: 21%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 99/1, flow rate: 1 mL/min, 210 nm): major enantiomer: t_R = 7.3 min, minor enantiomer: t_R = 10.1 min.



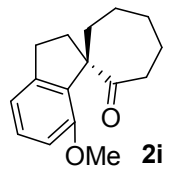
2g

(*S*)-2g: Reaction time: 72 h; 88% yield; brown oil; $[\alpha]_D^{26}$ = +20.6 (*c* = 0.7, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.93-1.75 (m, 1H), 1.94-2.04 (m, 1H), 2.06-2.27 (m, 4H), 2.27-2.59 (m, 2H), 2.83-3.04 (m, 2H), 3.74-3.91 (m, 9H), 6.53 (s, 1H); ^{13}C-NMR ($CDCl_3$): δ 20.7, 31.8, 36.4, 37.7, 38.3, 56.1, 59.9, 60.5, 60.7, 103.2, 131.7, 139.8, 140.5, 148.5, 154.0, 223.4; HRMS (ESI): calcd for $C_{15}H_{18}NaO_2$, m/z 299.1259 ([M+Na]$^+$); found, m/z 299.1251. Enantiomeric excess: 50%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 99/1, flow rate: 0.5 mL/min, 215 nm): major enantiomer: t_R = 29.7 min, minor enantiomer: t_R = 25.8 min.



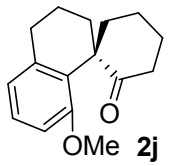
2h

(*S*)-2h: Reaction time: 72 h; 90% yield; white solid; Mp 100-101 ^{o}C; $[\alpha]_D^{25}$ = +21.3 (*c* = 0.9, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.68-1.89 (m, 4H), 2.02-2.15 (m, 2H), 2.24-2.37 (m, 2H), 2.41-2.49 (m, 1H), 2.54-2.60 (m, 1H), 2.88-3.02 (m, 2H), 3.78 (s, 3H), 6.72 (d, *J* = 8.2 Hz, 1H), 6.82 (d, *J* = 7.3 Hz, 1H), 7.19 (t, *J* = 8.2 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 22.3, 25.4, 30.9, 35.1, 36.5, 39.8, 55.3, 61.9, 109.0, 116.9, 129.0, 133.6, 145.2, 156.0, 212.0; HRMS (ESI): calcd for $C_{15}H_{18}NaO_2$, m/z 253.1204 ([M+Na]$^+$); found, m/z 253.1195. Enantiomeric excess: 61%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 99/1, flow rate: 1 mL/min, 219 nm): major enantiomer: t_R = 11.8 min, minor enantiomer: t_R = 16.6 min.



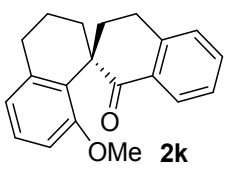
2i

(*S*)-2i: Reaction time: 96 h; 34% yield; yellow oil; $[\alpha]_D^{27}$ = +4.9 (*c* = 0.3, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.41-1.66 (m, 3H), 1.71-1.76 (dd, *J* = 7.8, 14.6 Hz, 1H), 1.86-1.97 (m, 3H), 2.01-2.07 (m, 1H), 2.39-2.45 (m, 1H), 2.55-2.62 (m, 2H), 2.89-2.97 (m, 1H), 3.00-3.07 (m, 1H), 3.21 (td, *J* = 12.4, 2.3 Hz, 1H), 3.77 (s, 3H), 6.64 (d, *J* = 8.2 Hz, 1H), 6.82 (d, *J* = 7.3 Hz, 1H), 7.13 (t, *J* = 7.3 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 25.6, 26.2, 31.1, 31.4, 34.2, 36.8, 44.1, 55.0, 63.9, 108.7, 117.5, 128.7, 136.3, 145.7, 155.3, 218.1. Enantiomeric excess: 54%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 99/1, flow rate: 0.5 mL/min, 220 nm): major enantiomer: t_R = 18.3 min, minor enantiomer: t_R = 21.9 min.



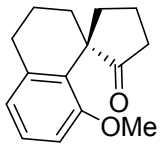
2j

(*S*)-2j: Reaction time: 144 h; 63% yield; pale yellow solid; Mp 110-112 ^{o}C; $[\alpha]_D^{22}$ = +6.0 (*c* = 0.6, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.58-1.68 (m, 1H), 1.70-1.82 (m, 4H), 1.85-2.02 (m, 3H), 2.05-2.18 (m, 2H), 2.35-2.44 (m, 1H), 2.57-2.63 (m, 1H), 2.70-2.84 (m, 2H), 3.74 (s, 3H), 6.71 (t, *J* = 8.2 Hz, 2H), 7.11 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 18.7, 21.6, 24.2, 29.8, 32.7, 35.2, 39.3, 50.6, 55.2, 109.2, 122.0, 127.0, 130.8, 138.5, 155.8, 214.9; HRMS (ESI): calcd for $C_{16}H_{20}NaO_2$, m/z 267.1361 ([M+Na]$^+$); found, m/z 267.1350. Enantiomeric excess: 16%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 99/1, flow rate: 1 mL/min, 220 nm): major enantiomer: t_R = 13.0 min, minor enantiomer: t_R = 20.9 min.



2k

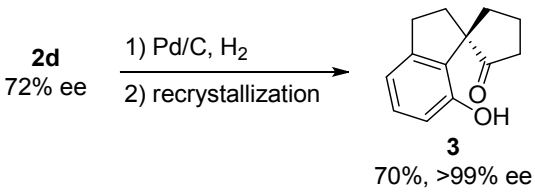
(*S*)-2k: Reaction time: 72 h; 79% yield; pale yellow solid; Mp 98-101 °C; $[\alpha]_D^{22}$ = +10.0 (*c* = 0.9, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.76-1.94 (m, 3H), 2.05-2.11 (m, 1H), 2.20-2.26 (m, 1H), 2.67 (td, *J* = 5.0, 13.7 Hz, 1H), 2.79-2.84 (m, 2H), 2.87-2.95 (m, 1H), 3.12-3.21 (m, 1H), 3.50 (s, 3H), 6.68 (d, *J* = 8.2 Hz, 1H), 6.76 (d, *J* = 6.9 Hz, 1H), 7.13 (t, *J* = 8.2 Hz, 1H), 7.25 (d, *J* = 7.8 Hz, 1H), 7.33 (t, *J* = 7.3 Hz, 1H), 7.47 (td, *J* = 1.4, 7.3 Hz, 1H), 6.12 (dd, *J* = 1.4, 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 18.6, 25.5, 29.7, 29.9, 31.8, 48.1, 55.1, 109.6, 122.2, 126.4, 127.1, 127.7, 128.4, 130.7, 132.5, 132.7, 138.7, 142.2, 156.3, 201.8; HRMS (ESI): calcd for $C_{20}H_{20}NaO_2$, m/z 315.1361 ($[M+Na]^+$); found, m/z 315.1348. Enantiomeric excess: 12%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 99/1, flow rate: 1 mL/min, 239 nm): major enantiomer: t_R = 18.1 min, minor enantiomer: t_R = 26.4 min.



2l

(*S*)-2l: 59% yield; colorless solid; Mp 73-75 °C; $[\alpha]_D^{23}$ = +28.8 (*c* = 0.6, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.51-1.58 (m, 1H), 1.65-1.76 (m, 1H), 1.82-1.99 (m, 3H), 2.02-2.13 (m, 2H), 2.25-2.43 (m, 2H), 2.56-2.66 (m, 1H), 2.73-2.86 (m, 2H), 3.71 (s, 3H), 6.67 (d, *J* = 7.8 Hz, 1H), 6.72 (d, *J* = 7.8 Hz, 1H), 7.10 (t, *J* = 8.2 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 19.4, 19.8, 29.5, 31.1, 36.2, 37.2, 51.4, 55.0, 109.0, 122.1, 127.2, 128.5, 139.2, 156.0, 223.7; HRMS (ESI): calcd for $C_{15}H_{18}NaO_2$, m/z 253.1204 ($[M+Na]^+$); found, m/z 253.1193. Enantiomeric excess: 32%, determined by HPLC (Chiralpak IA, *n*-hexane/*i*-PrOH = 99/1, flow rate: 1 mL/min, 220 nm): major enantiomer: t_R = 21.0 min, minor enantiomer: t_R = 13.0 min.

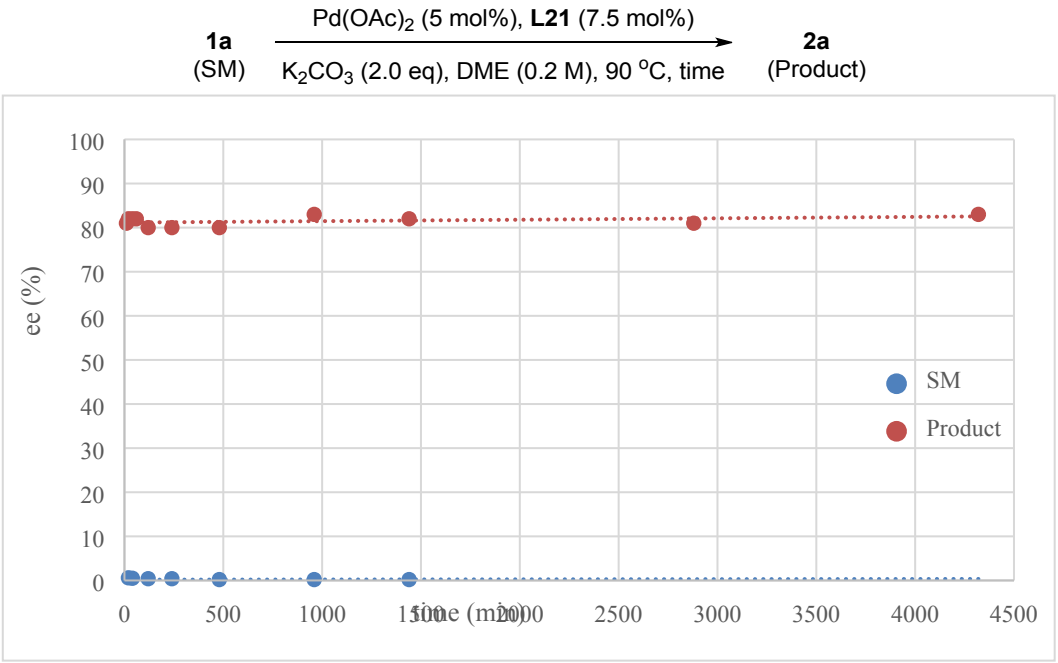
Preparation of (S)-3



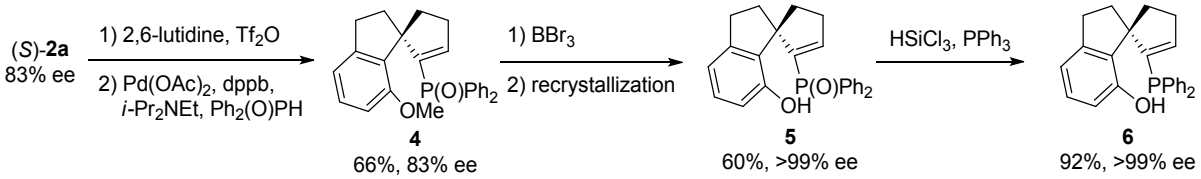
3
70%, >99% ee

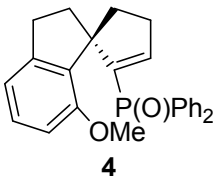
The round flask was charged with (*S*)-**2d** (83% ee, 326 mg, 1.12 mol), palladium on carbon (10%, 119 mg) and MeOH (5.6 mL) under H_2 balloon atmosphere. The reaction mixture was stirred at room temperature for 3.5 h. After filtration and concentration under vacuum, recrystallization in EtOAc/hexane gave optically pure **3**. 70% yield; colorless crystal; Mp 153-155 °C; $[\alpha]_D^{28}$ = +80.6 (*c* = 1.1, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.82-1.95 (m, 1H), 2.00-2.24 (m, 4H), 2.29-2.44 (m, 2H), 2.51-2.61 (m, 1H), 2.92-3.05 (m, 1H), 5.00 (s, 1H), 6.49 (d, *J* = 8.2 Hz, 1H), 6.80 (d, *J* = 7.8 Hz, 1H), 7.03 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 20.9, 31.6, 35.4, 37.5, 38.4, 60.6, 113.7, 116.7, 129.0, 132.3, 146.9, 151.6, 224.6; HRMS (ESI): calcd for $C_{13}H_{14}NaO_2$, m/z 225.0891 ($[M+Na]^+$); found, m/z 225.0883. Enantiomeric excess: >99%, determined by HPLC (Chiralpak OD-3, *n*-hexane/*i*-PrOH = 95/5, flow rate: 0.5 mL/min, 219 nm): major enantiomer: t_R = 10.2 min, minor enantiomer: t_R = 13.1 min.

Conversion dependence on the enantioselectivities between 1a and 2a



Preparation of spiro-type organocatalyst (S)-6

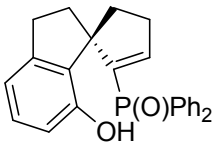




4

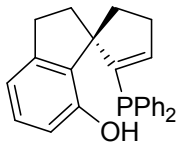
Preparation of (*S*)-4: To the solution of (*S*)-**2a** (448 mg, 2.07 mmol, 83% ee) in CH_2Cl_2 (50 mL) was added 2,6-lutidine (0.30 mL, 2.59 mmol) under N_2. The solution was cooled to 0 ˚C and Tf_2O (0.38 mL, 2.28 mmol) was then added dropwise. The mixture was stirred at room temperature for 2 h before addition of H_2O. The reaction mixture was then extracted with CH_2Cl_2. The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. After the purification by silica column chromatography, the corresponding trifrate was obtained in 71% yield; colorless oil; $[\alpha]_D^{21}$ = -48.7 (*c* = 1.6, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 2.05-2.16 (m, 2H), 2.30-2.52 (m, 3H), 2.58-2.66 (m, 1H), 2.89-3.02 (m, 2H), 3.81 (s, 3H), 5.68 (t, *J* = 2.3 Hz, 1H), 6.73 (d, *J* = 8.2 Hz, 1H), 6.85 (d, *J* = 7.3 Hz, 1H), 7.22 (t, *J* = 7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$): δ 26.8, 30.9, 34.9, 37.6, 55.1, 58.3, 108.6, 112.9, 117.1, 129.4, 131.7, 146.2, 152.5, 156.7; HRMS (ESI): calcd for $C_{15}H_{15}F_3O_4SNa$, m/z 371.0541 ($[M+Na]^+$); found, m/z 371.0536. To the solution of the trifrate (400 mg, 1.15 mmol, 83% ee), $Ph_2P(O)H$ (465 mg, 2.3 mmol), $Pd(OAc)_2$ (12.9 mg, 57.5 μmol) and dppb (24.5 mg, 57.5 μmol) in degassed DMSO (3.5 mL) was added *i*-Pr_2NEt (0.79 mL, 4.6 mmol) under N_2. The reaction mixture was heated at 100 ˚C for 6 h. After cooling to room temperature, the reaction mixture was dituled with EtOAc,

washed with 5% aqueous HCl and saturated $NaHCO_3$. The organic phase was dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The crude product was purified by silica gel column chromatography to give (*S*)-**4** in 93% yield; pale yellow oil; $[\alpha]_D^{17}$ = -2.9 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 2.02-2.10 (m, 2H), 2.43-2.57 (m, 2H), 2.69-2.79 (m, 3H), 3.07-3.15 (m, 1H), 3.64 (s, 3H), 6.16 (dt, *J* = 11.0, 2.7, 1H), 6.32 (d, *J* = 7.8 Hz, 1H), 6.50 (dd, *J* = 0.9, 7.3 Hz, 1H), 6.87 (t, *J* = 7.8 Hz, 1H), 7.13-7.17 (m, 2H), 7.25-7.29 (m, 1H), 7.35-7.47 (m, 5H), 7.64-7.69 (m, 2H); HRMS (ESI): calcd for $C_{26}H_{25}O_2PNa$, m/z 423.1490 ($[M+Na]^+$); found, m/z 423.1474.



5

Preparation of (*S*)-5: To the solution of (*S*)-**4** (410 mg, 1.02 mmol) in CH_2Cl_2 (3.4 mL) was added dropwise BBr_3 (3.1 mL, 3.06 mmol, 1 M in CH_2Cl_2) at 0 ˚C under N_2. The reaction mixture was stirred at room temperature for 16 h. After slow addition of H_2O, the reaction mixture was extracted with CH_2Cl_2. The combined organic layers were washed with water, brine, dried over anhydrous Na_2SO_4, filtered and evaporated under vacuum. The obtained residue was recrystallized from CH_2Cl_2/hexane to give optically pure (*S*)-**5** in 60% yield; colorless solid; Mp 234-236 °C; $[\alpha]_D^{18}$ = +57.1 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR ($CDCl_3$): δ 1.53-1.64 (m, 1H), 1.86-1.91 (m, 1H), 2.06-2.11 (m, 1H), 2.17-2.27 (m, 1H), 2.37-2.43 (m, 1H), 2.51-2.56 (m, 2H), 2.66-2.75 (m, 1H), 6.37 (dt, *J* = 11.4 Hz, 2.7 Hz, 1H), 6.64 (dd, *J* = 0.9, 7.3 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 7.06 (t, *J* = 7.8 Hz, 1H), 7.25-7.30 (m, 2H), 7.39-7.44 (m, 1H), 7.46-7.58 (m, 5H), 7.83-7.88 (m, 2H), 8.46 (s, 1H); ^{13}C-NMR ($CDCl_3$): δ 30.3, 32.0 (d, *J* = 16.3 Hz), 36.1, 39.2 (d, *J* = 7.7 Hz), 62.8 (d, *J* = 11.5 Hz), 117.8, 119.6, 128.1 (d, *J* = 12.0 Hz), 128.4 (d, *J* = 12.0 Hz), 128.7, 130.2 (d, *J* = 105.4 Hz), 130.7 (d, *J* = 105.4 Hz), 131.6 (d, *J* = 9.6 Hz), 131.8 (d, *J* = 2.4 Hz), 131.9 (d, *J* = 9.6 Hz), 132.0 (d, *J* = 2.4 Hz), 136.6, 142.8 (d, *J* = 97.8 Hz), 144.9, 149.9 (d, *J* = 13.4 Hz), 153.6; ^{31}P-NMR ($CDCl_3$): δ 23.0; HRMS (ESI): calcd for $C_{25}H_{23}NaO_2P$, m/z 409.1333 ([M+Na]+); found, m/z 409.1330.



6

Preparation of (*S*)-6: To the solution of optically pure (*S*)-**5** (39 mg, 0.10 mmol) and triphenylphosphine (53 mg, 0.20 mmol) in toluene and THF (1:1) was added dropwise $HSiCl_3$ (0.20 mL, 2.0 mmol) at 0 ˚C under N_2. The reaction mixture was stirred at 100 °C for 5 h. After cooling to room temperature, the mixture was diluted with diethyl ether. To the solution was added 20% NaOH solution. The mixture was transferred to a separating funnel and shaked for 10 min. The organic layer was separated and washed with saturated $NaHCO_3$. The solution was then dried over anhydrous Na_2SO_4 and evaporated under vacuum and the residue was purified by flash chromatography to give optically pure (*S*)-**6** in 92% yield; viscous oil; ^{1}H-NMR ($CDCl_3$): δ 1.81-1.84 (m, 1H), 2.02-2.09 (m, 1H), 2.20-2.31 (m, 2H), 2.63-2.66 (m, 2H), 2.76-2.87 (m, 2H), 5.19 (s, 1H), 5.86-5.87 (m, 1H), 6.62 (d, *J* = 7.6 Hz, 1H), 6.74 (d, *J* = 7.6 Hz, 1H), 7.06 (t, *J* = 7.6 Hz, 1H), 7.22-7.28 (m, 5H), 7.35-7.37 (m, 3H), 7.44-7.49 (m, 2H); ^{13}C-NMR ($CDCl_3$): δ 31.3, 33.1 (d, *J* = 3.8 Hz), 38.5 (d, *J* = 3.8 Hz), 39.8 (d, *J* = 2.9 Hz), 64.3 (d, *J* = 25.9 Hz), 114.1, 116.9, 128.3 (d, *J* = 7.2 Hz), 128.3 (d, *J* = 7.2 Hz), 128.5, 128.8, 128.9 , 133.4, 133.5 (d, *J* = 20.1 Hz), 134.0 (d, *J* = 20.1 Hz), 135.6 (d, *J* = 9.1 Hz), 137.2 (d, *J* = 9.1 Hz) 143.6, 145.3, 148.2 (d, *J* = 20.1 Hz), 152.8; ^{31} P-NMR

($CDCl_3$): δ -29.2; HRMS (ESI): calcd for $C_{25}H_{24}OP$, m/z 371.1565 ($[M+H]^+$); found, m/z 371.1561.

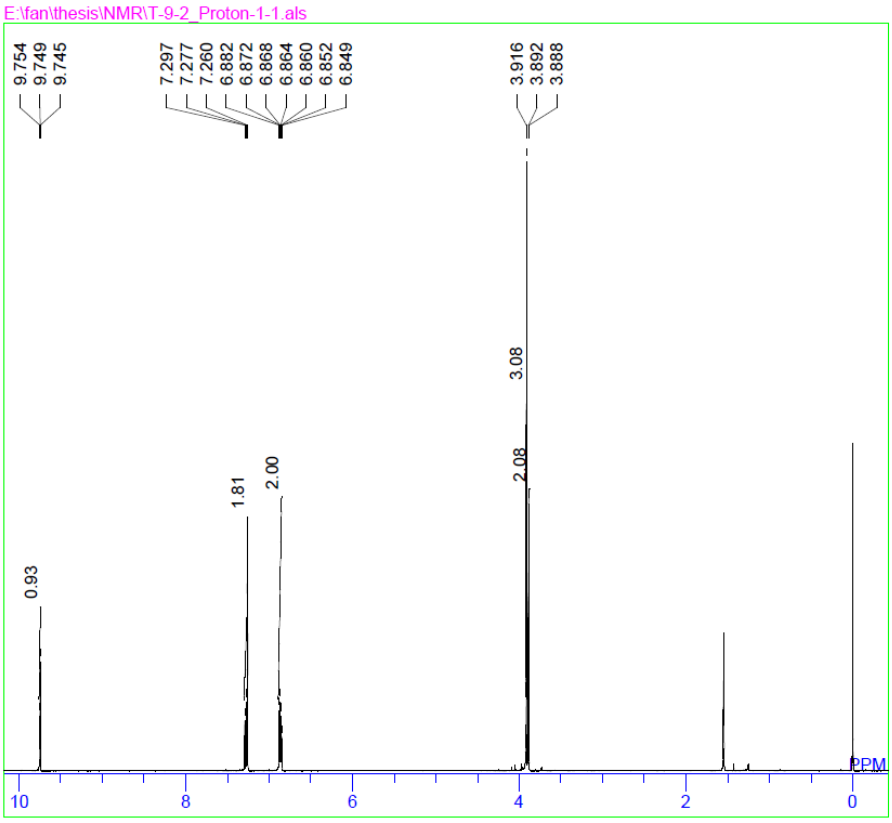
Procedure for enantioselective aza-MBH reaction mediated by (S)-6

Under N_2 atmosphere, a test tube was charged with (*S*)-**6** (2.2 mg, 10 mol%), methyl vinyl ketone (**7**, 15 μL, 0.18 mmol), *m*-chlorophenyl *N*-tosyl imine (**8**, 17.6 mg, 0.06 mmol) and MS 3A in CH_2Cl_2 (0.3 mL). The reaction mixture was stirred at -10 °C for 24 h. After the purification *via* SiO_2 column chromatography, the aza-MBH adduct **9**[5] was obtained in 71% yield. Enantiomeric excess: 54%, determined by HPLC (Chiralpak AD-H, *n*-hexane/*i*-PrOH = 1/4, flow rate: 0.7 mL/min, 227 nm): major enantiomer: t_R =19.7 min, minor enantiomer: t_R = 15.4 min.

References

1. F. R. Fischer and C. Nuckolls, *Angew. Chem., Int. Ed*., 2010, **49**, 7257.
2. K. Mori, Y. Ichikawa, M. Kobayashi, Y. Shibata, M. Yamanaka,and T. Akiyama, *J. Am. Chem. Soc.* 2013, **135**, 3964.
3. G. A. Molander, K. M. George and L. G. Monovich, *J. Org. Chem.* **2003**, *68*, 9533.
4. H. Muratake and M. Natsume, *Tetrahedron Lett*., 1997, **38**, 7581.
5. K. Matsui, S. Takizawa and H. Sasai, *J. Am. Chem. Soc.* 2005, **127**, 3680.

NMR chart



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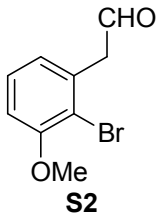
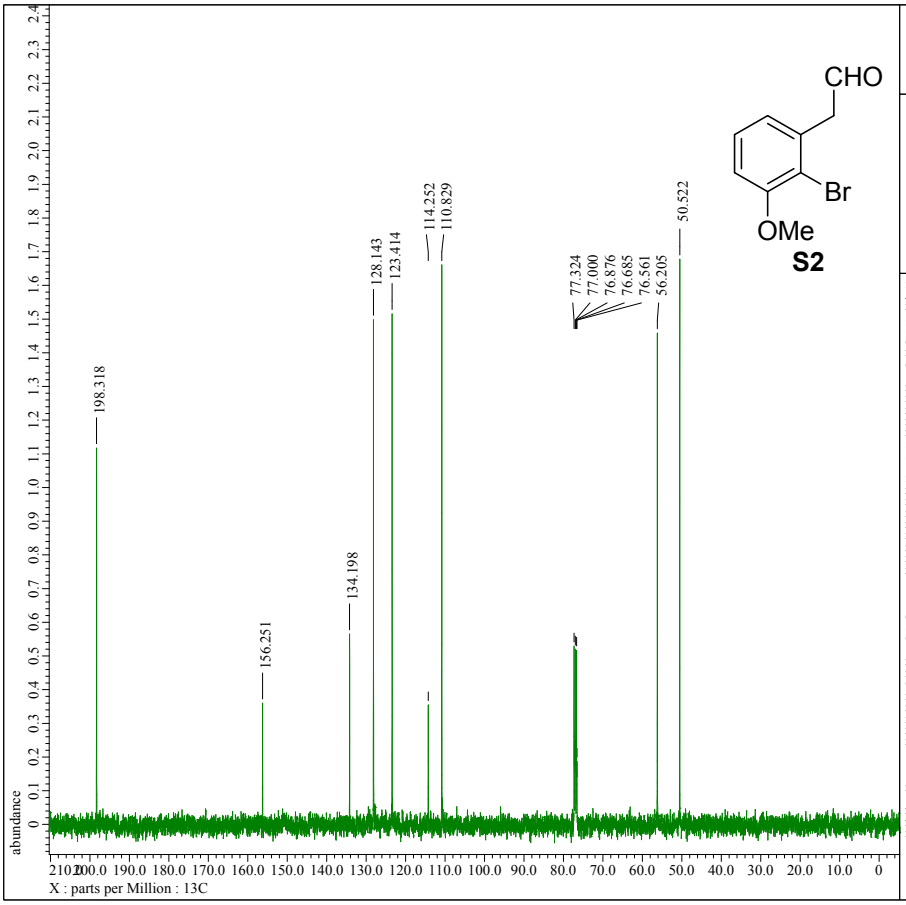


S2



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□□□□□□: 2_step_product_CARBON-2.jdf
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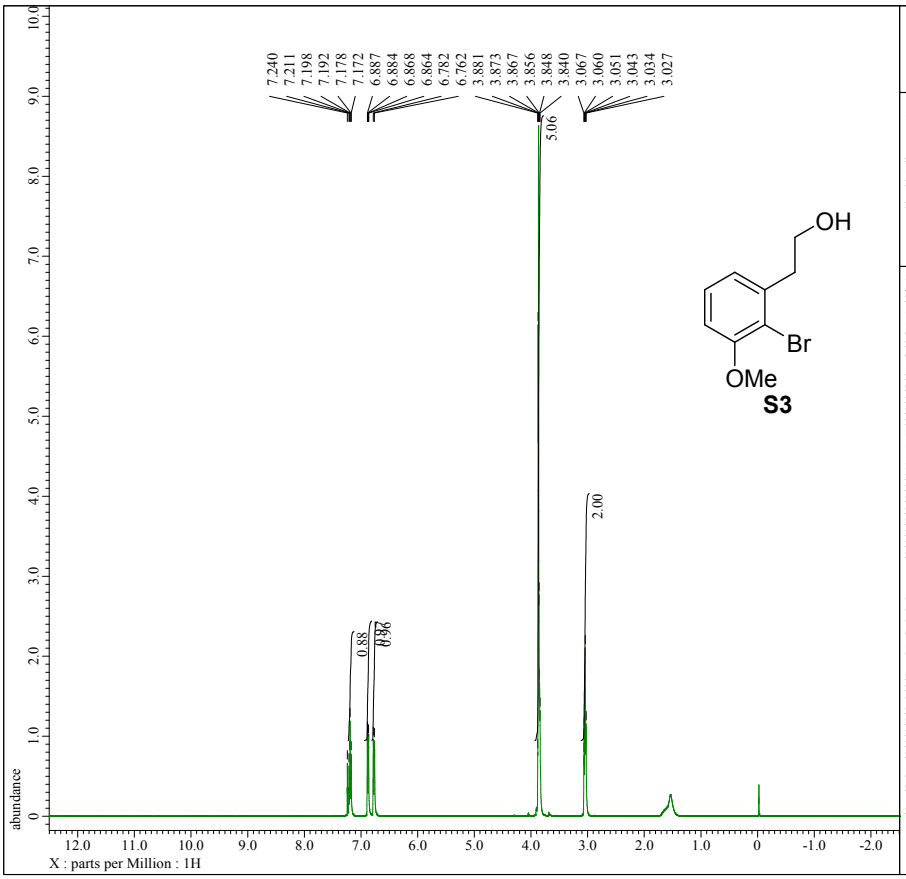

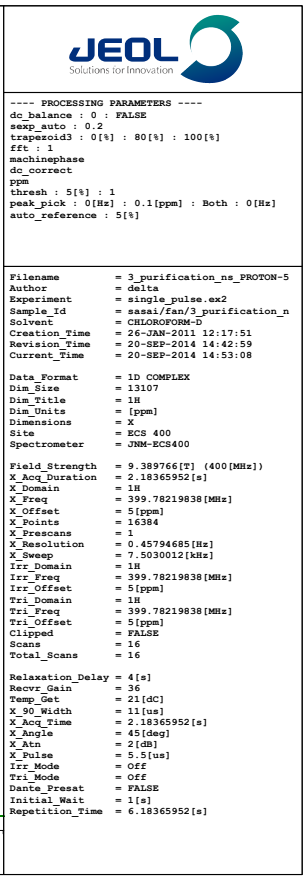


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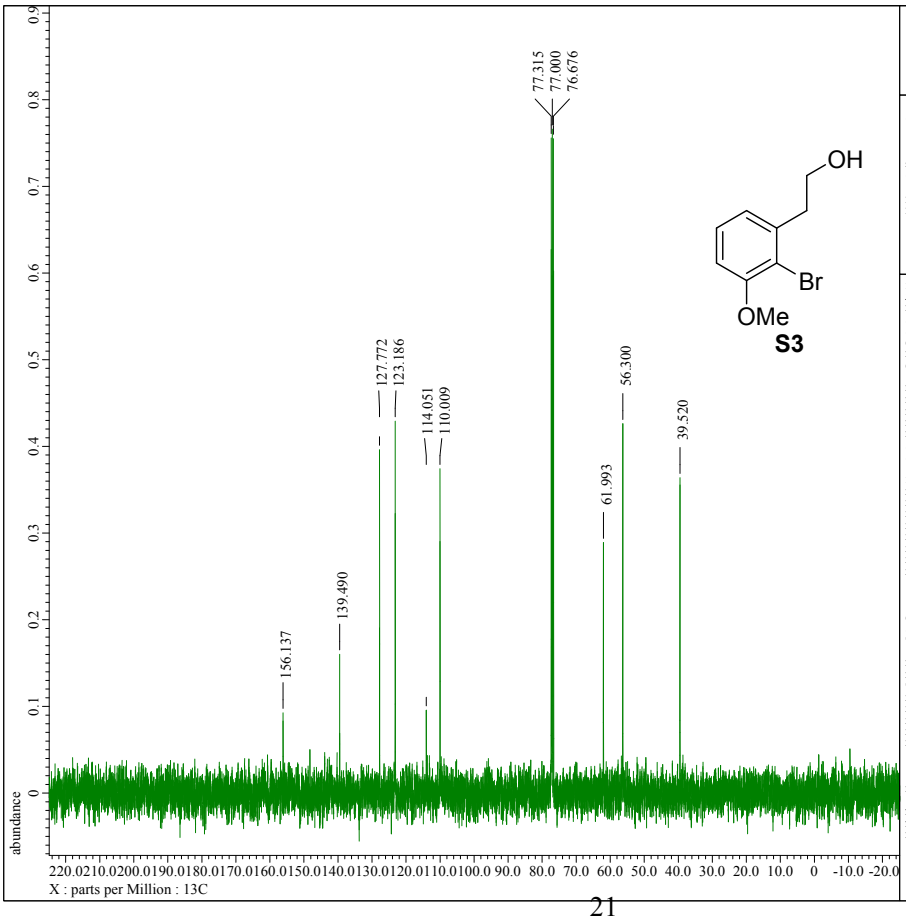

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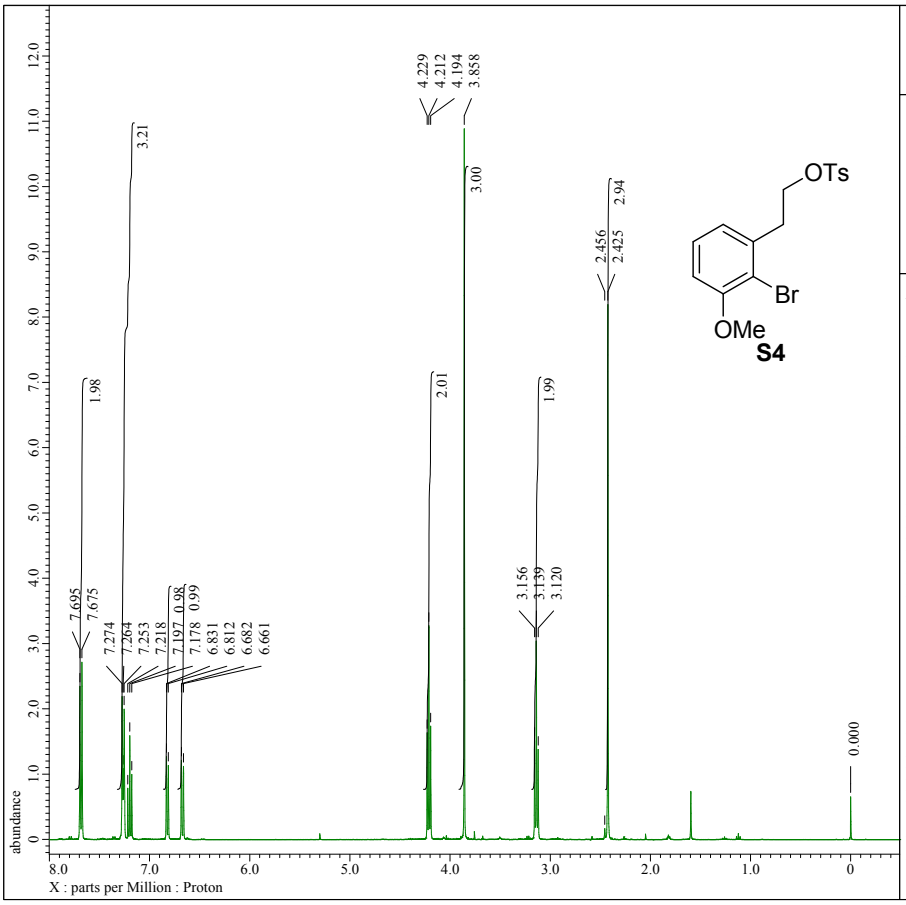


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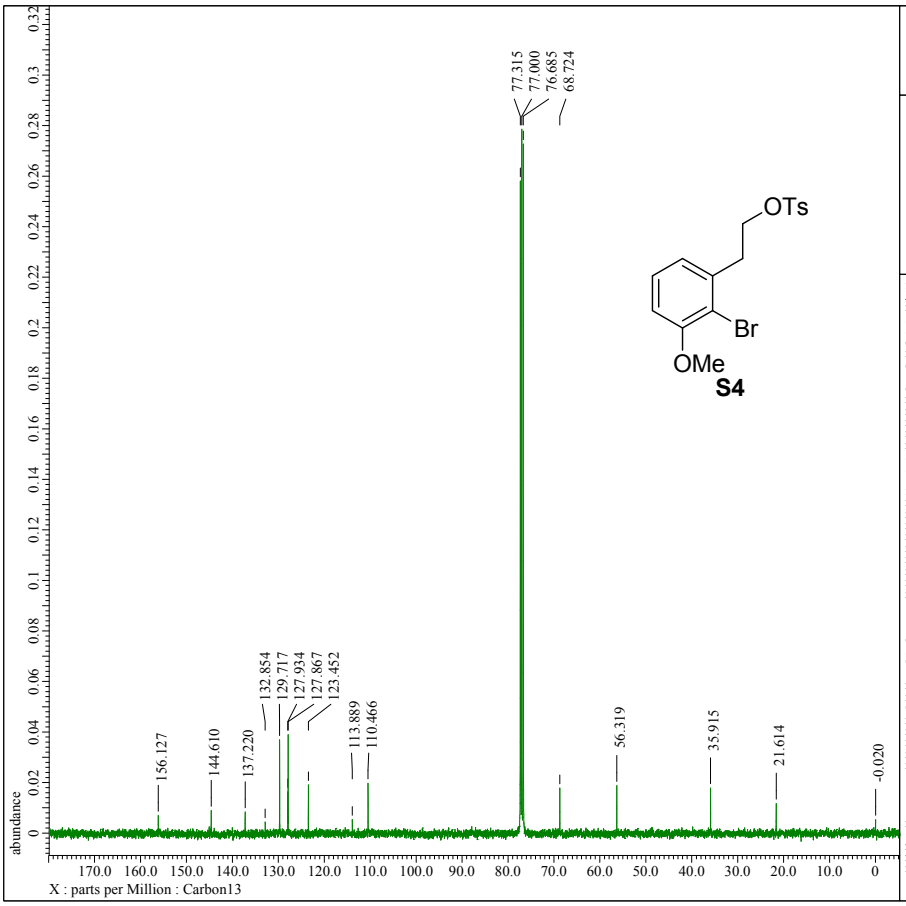


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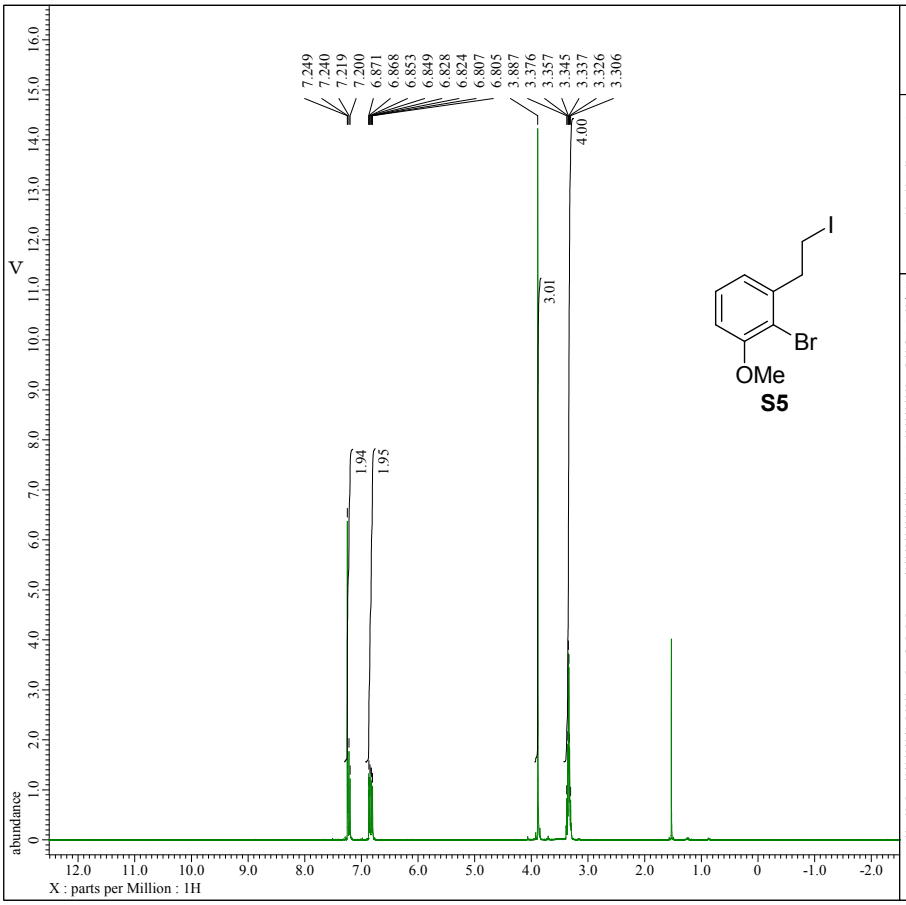
Repetition_Time   = 3.04333312[s]
```



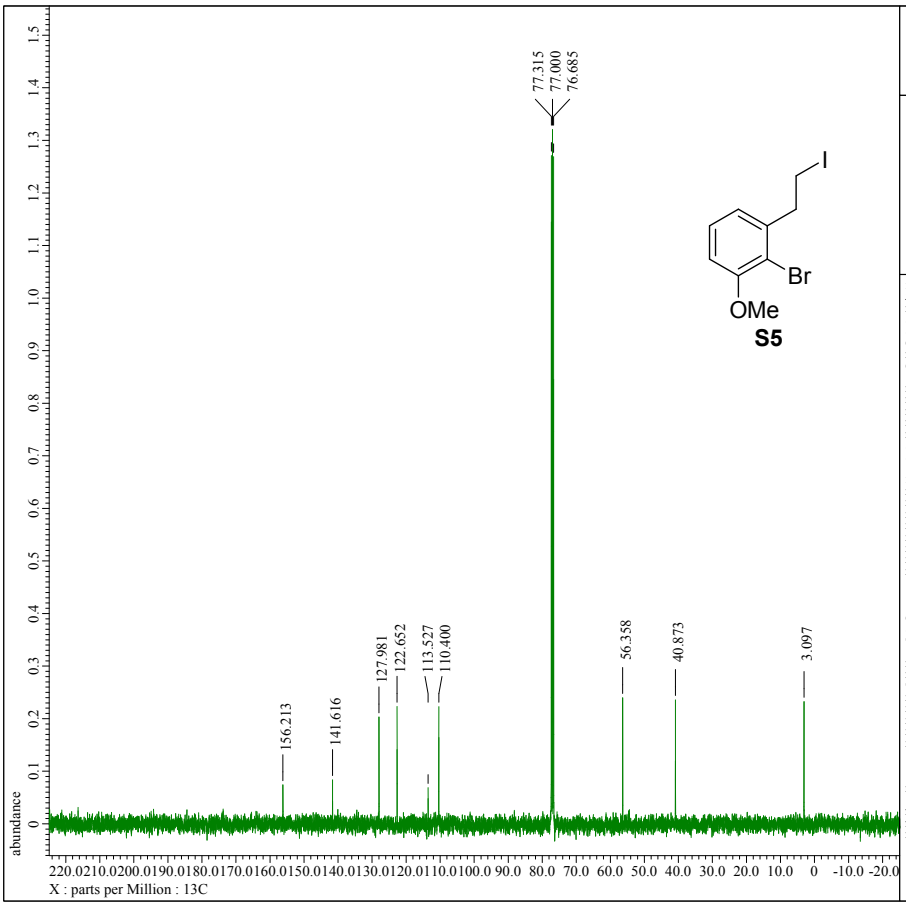

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
```

```
Filename          = iodine-crystal2011.2.8_PRO
Author            = delta
Experiment        = single_pulse.ex2
Sample_Id         = sasai/fan/iodine-crysta120
Solvent           = CHLOROFORM-D
Creation_Time     =  8-FEB-2011 12:10:48
Revision_Time     = 20-SEP-2014 15:32:16
Current_Time      = 20-SEP-2014 15:32:35

Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = 1H
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

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

Relaxation_Delay = 4[s]
Recvr_Gain        = 54
Temp_Get          = 21.8[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.18365952[s]
```



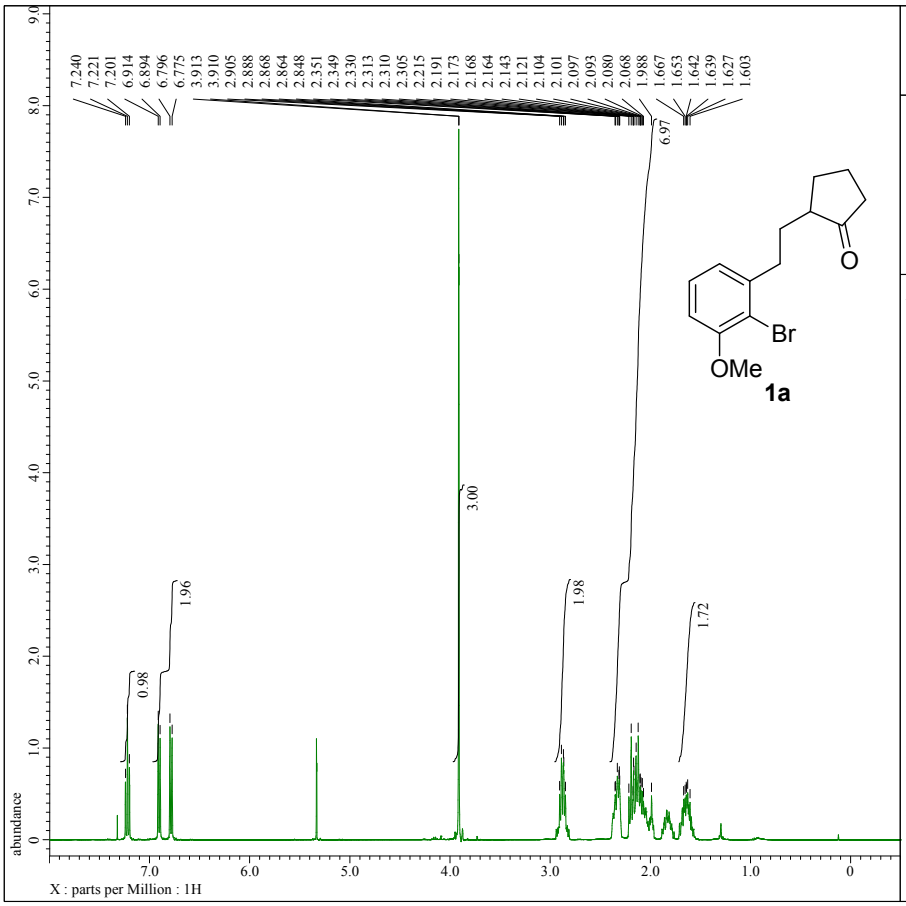

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = iodine-crystal-2_CARBON-5.
Author            = delta
Experiment        = single_pulse_dec
Sample_Id         = sasai/fan/iodine-crystal-2
Solvent           = CHLOROFORM-D
Creation_Time     =  8-FEB-2011 12:27:11
Revision_Time     = 20-SEP-2014 15:34:52
Current_Time      = 20-SEP-2014 15:35:02

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

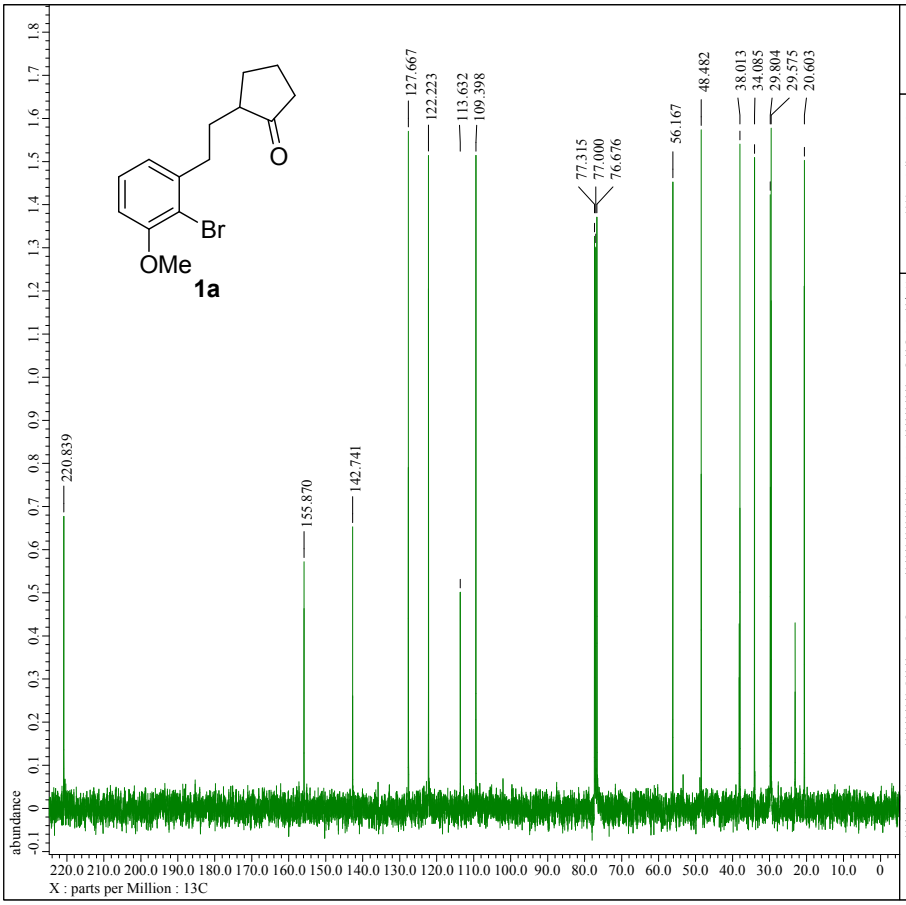
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
Irr_Domain        = 1H
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 74
Total_Scans       = 74

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.2[dC]
X_90_Width        = 9.4[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.13333333[us]
Irr_Atn_Dec       = 21.8[dB]
Irr_Atn_Noe       = 21.8[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



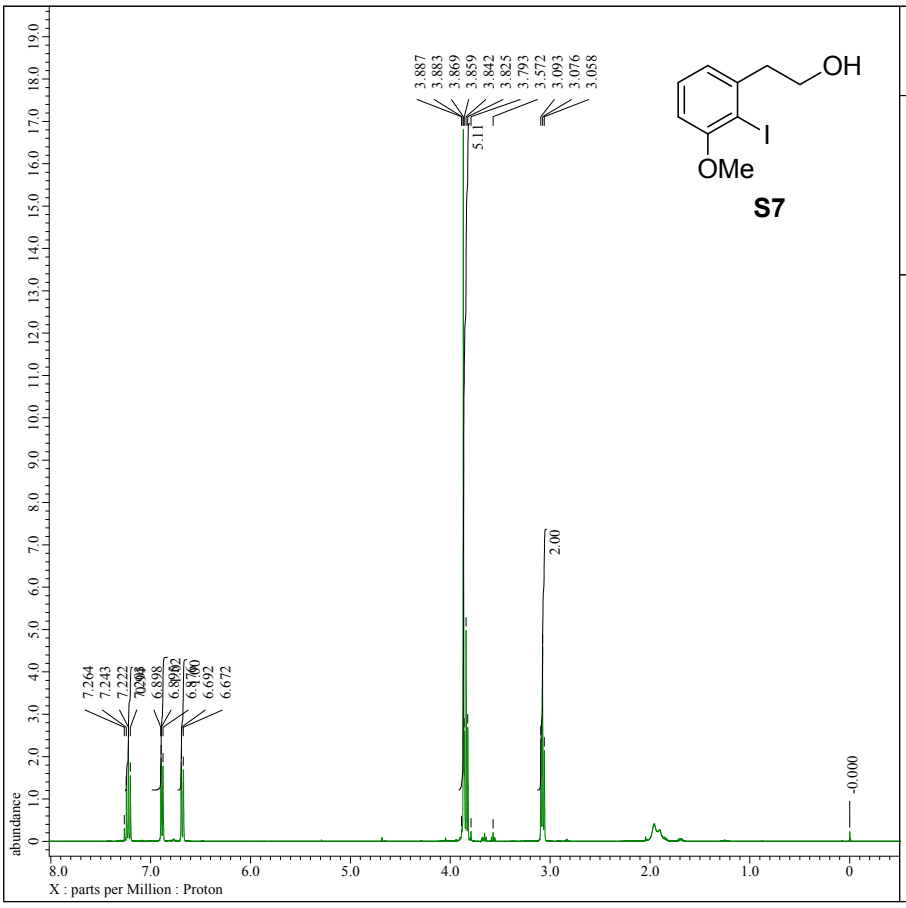

```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp_auto : 0.2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
ppm
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Both : 0[Hz]
auto_reference : 5[%]
```

Filename	= one-2011.2.10_PROTON-5.jdf
Author	= delta
Experiment	= single_pulse.ex2
Sample_Id	= sasai/fan/one-2011.2.10
Solvent	= CHLOROFORM-D
Creation_Time	= 10-FEB-2011 17:00:47
Revision_Time	= 20-SEP-2014 17:33:59
Current_Time	= 20-SEP-2014 17:34:25
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 4[s]
Recvr_Gain	= 24
Temp_Get	= 22.1[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 5.5[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 6.18365952[s]




```
---- PROCESSING PARAMETERS ----
dc_balance : 0 : FALSE
sexp_auto : 2
trapezoid3 : 0[%] : 80[%] : 100[%]
fft : 1
machinephase
dc_correct
thresh : 5[%] : 1
peak_pick : 0[Hz] : 0.1[ppm] : Peaks : 0[Hz]
ppm
```

Filename	= one-2011.2.10_CARBON-5.jdf
Author	= delta
Experiment	= single_pulse_dec
Sample_Id	= sasai/fan/one-2011.2.10
Solvent	= CHLOROFORM-D
Creation_Time	= 10-FEB-2011 17:02:45
Revision_Time	= 20-SEP-2014 17:41:43
Current_Time	= 20-SEP-2014 17:42:21
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
Irr_Domain	= 1H
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 14
Total_Scans	= 14
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 22.1[dC]
X_90_Width	= 9.4[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 3.13333333[us]
Irr_Atn_Dec	= 21.8[dB]
Irr_Atn_Noe	= 21.8[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]



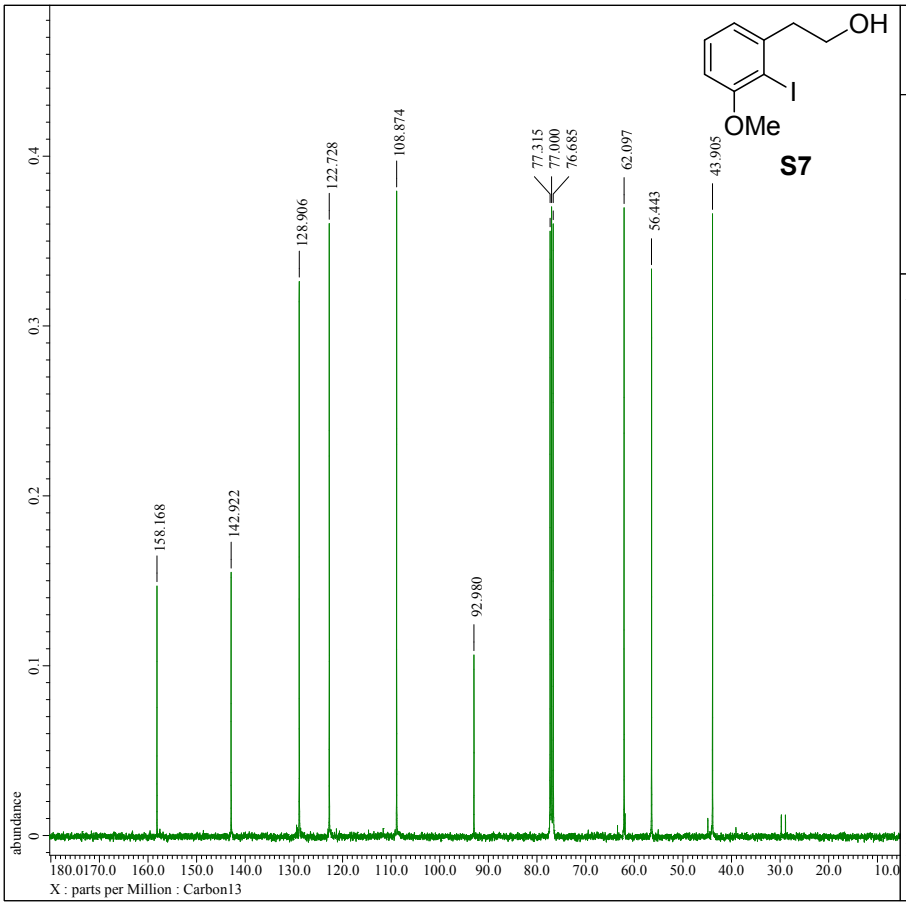

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-168-1_Proton-2-7.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-168-1
Solvent           = CHLOROFORM-D
Creation_Time     = 30-APR-2014 22:47:19
Revision_Time     = 20-SEP-2014 17:17:55
Current_Time      = 20-SEP-2014 17:18:12

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.0024096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 28
Temp_Get          = 18[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

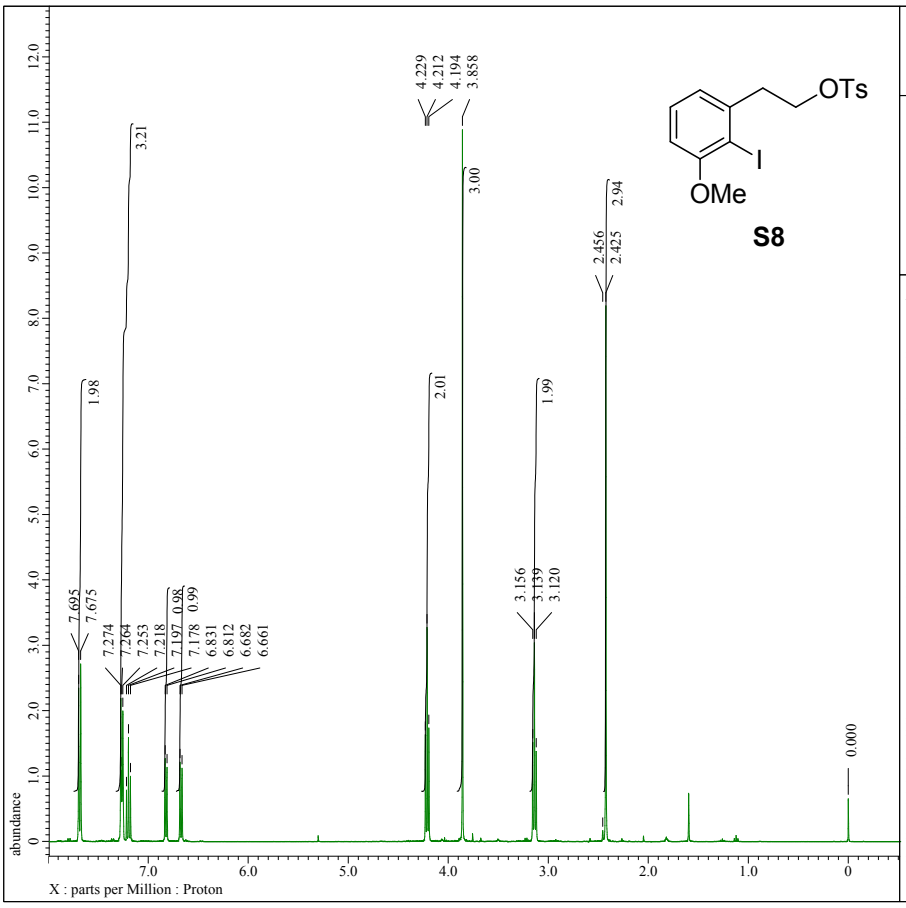
□ □ □□ □ □: T-168-1_Carbon-1-1.jdf
```

```
Filename          = T-168-1_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-168-1
Solvent           = CHLOROFORM-D
Creation_Time     = 30-APR-2014 22:48:31
Revision_Time     = 20-SEP-2014 17:20:03
Current_Time      = 20-SEP-2014 17:20:23

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 18.3[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



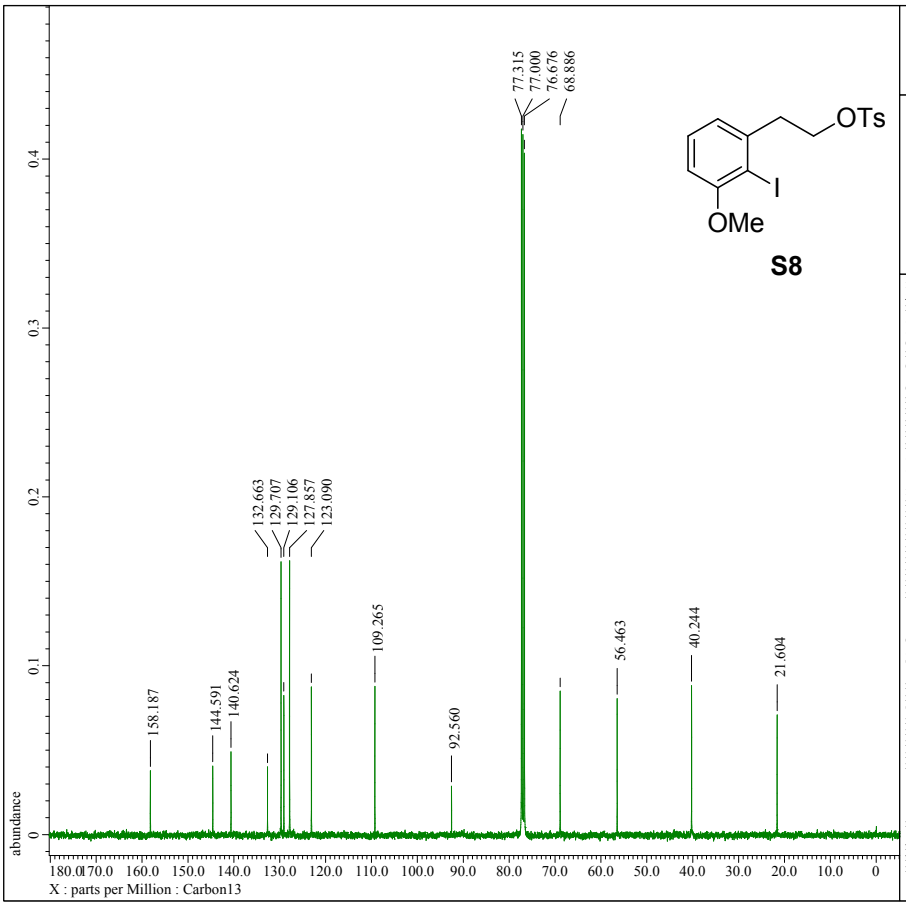

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-171-1_Proton-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-171-1
Solvent           = CHLOROFORM-D
Creation_Time     =  2-MAY-2014 20:04:37
Revision_Time     = 20-SEP-2014 17:24:17
Current_Time      = 20-SEP-2014 17:24:20

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.0024096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 18.4[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



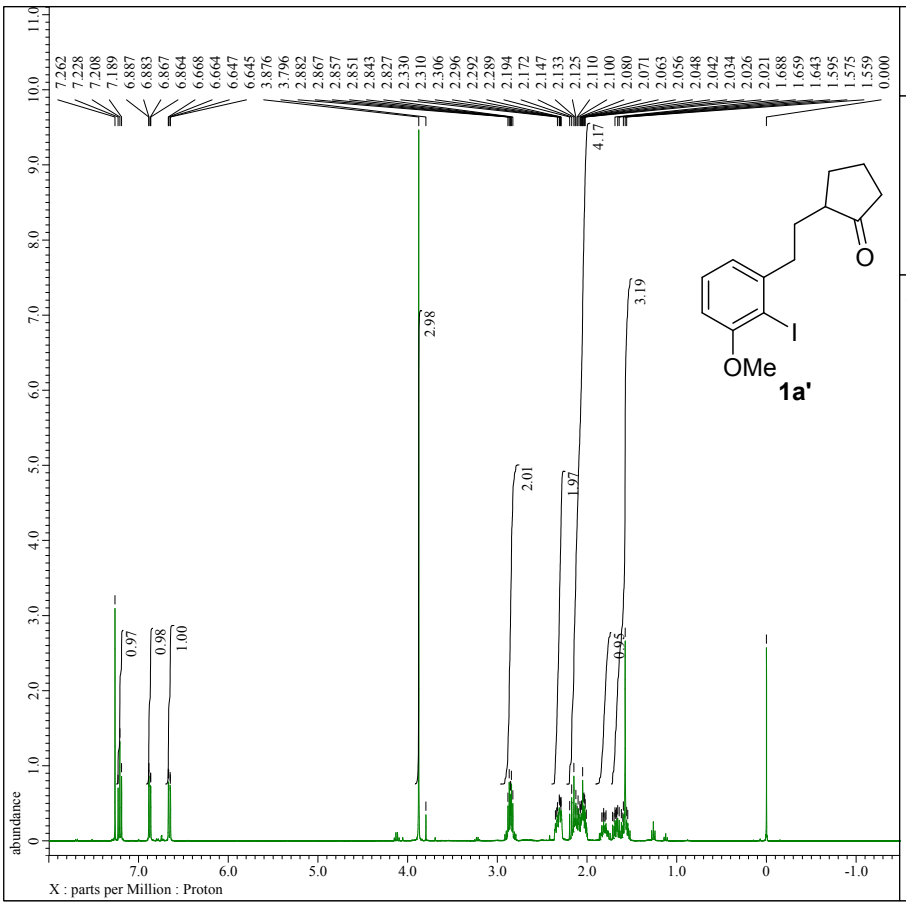

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-171-1_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-171-1
Solvent           = CHLOROFORM-D
Creation_Time     =  2-MAY-2014 20:05:51
Revision_Time     =  2-MAY-2014 20:29:00
Current_Time      = 20-SEP-2014 17:25:32

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 18.8[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



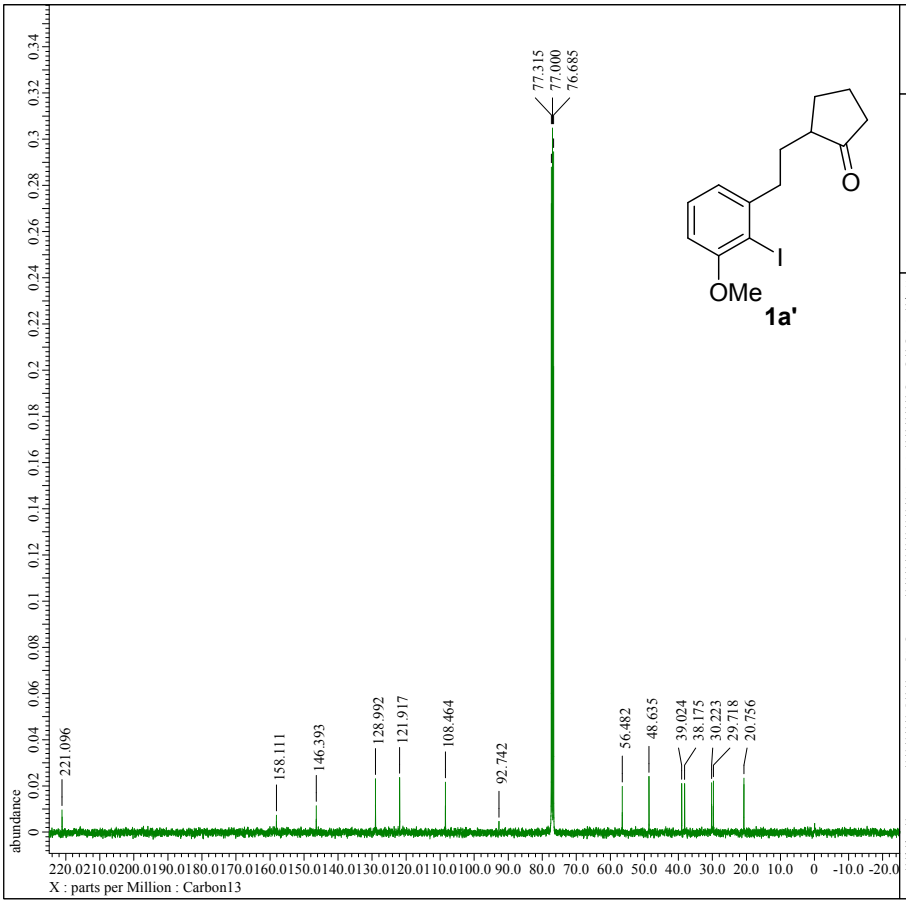

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-176-1_Proton-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-176-1
Solvent           = CHLOROFORM-D
Creation_Time     = 13-MAY-2014 21:26:09
Revision_Time     = 20-SEP-2014 16:52:53
Current_Time      = 20-SEP-2014 16:53:21

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 19.3[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



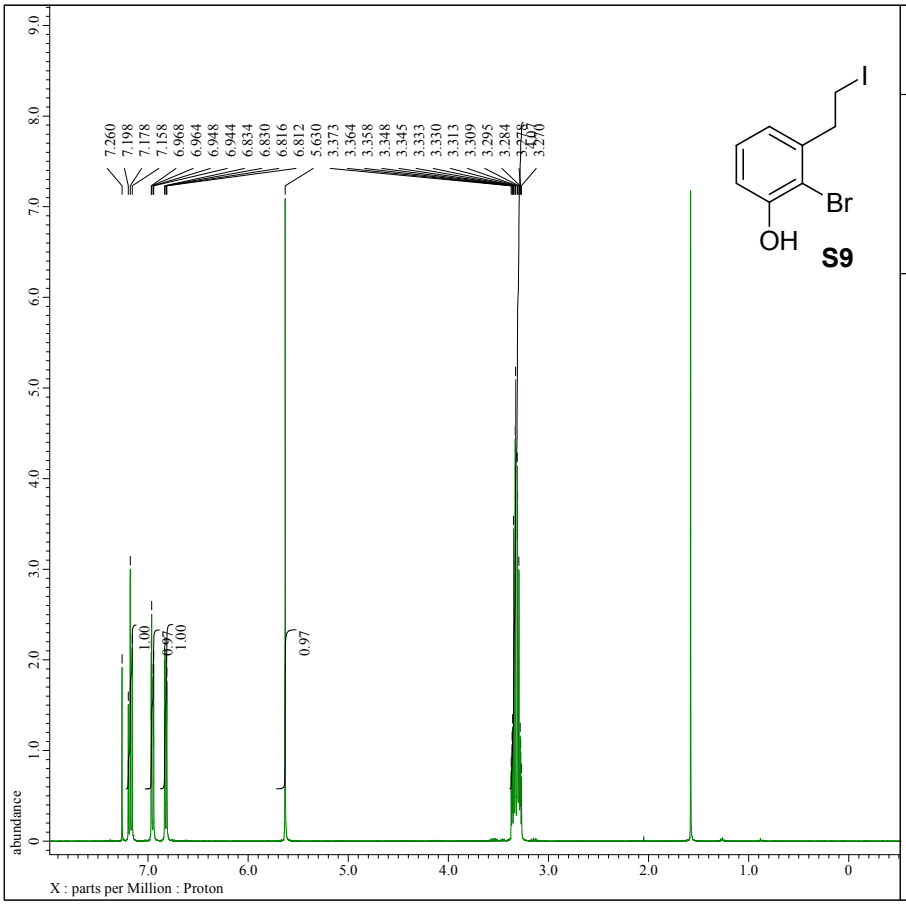

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-176-1_Carbon-1-6.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-176-1
Solvent           = CHLOROFORM-D
Creation_Time     = 13-MAY-2014 21:27:21
Revision_Time     = 20-SEP-2014 16:54:45
Current_Time      = 20-SEP-2014 16:54:52

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 20.4[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21[dB]
Irr_Atn_Noe       = 21[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

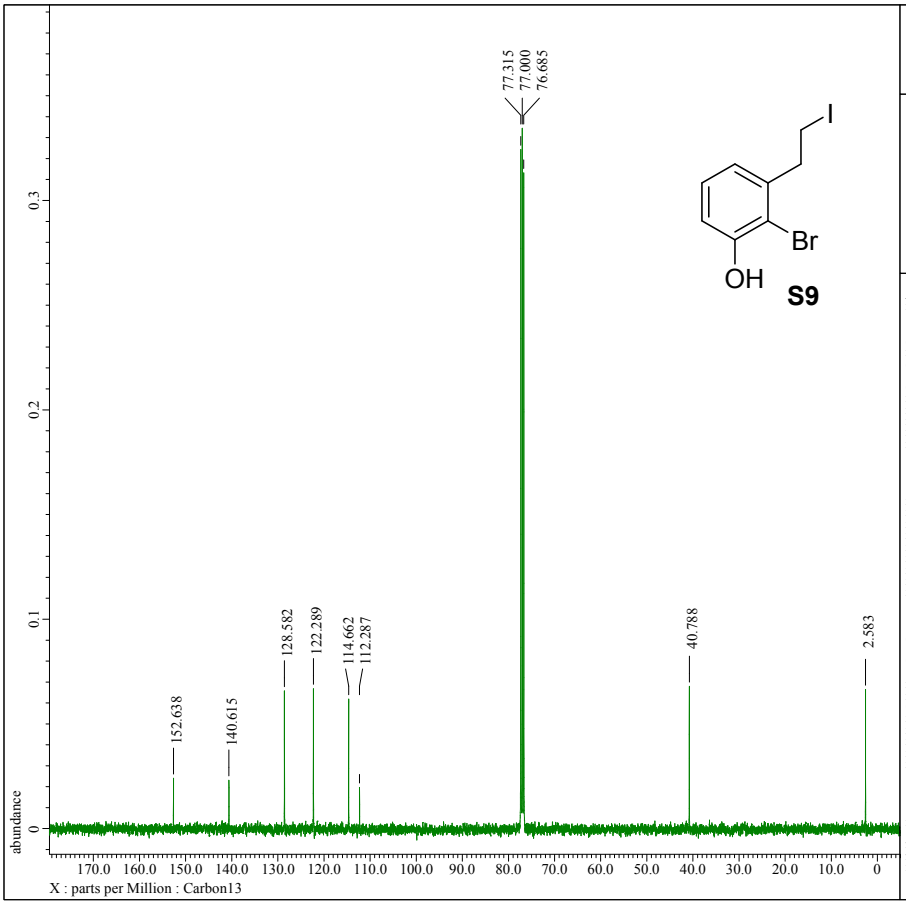
□ □ □ □ □: 205_Proton-1-1.jdf
```

```
Filename          = 205_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 205
Solvent           = CHLOROFORM-D
Creation_Time     = 15-JUN-2012 21:28:59
Revision_Time     = 20-SEP-2014 17:51:27
Current_Time      = 20-SEP-2014 17:52:01

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 21.6[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



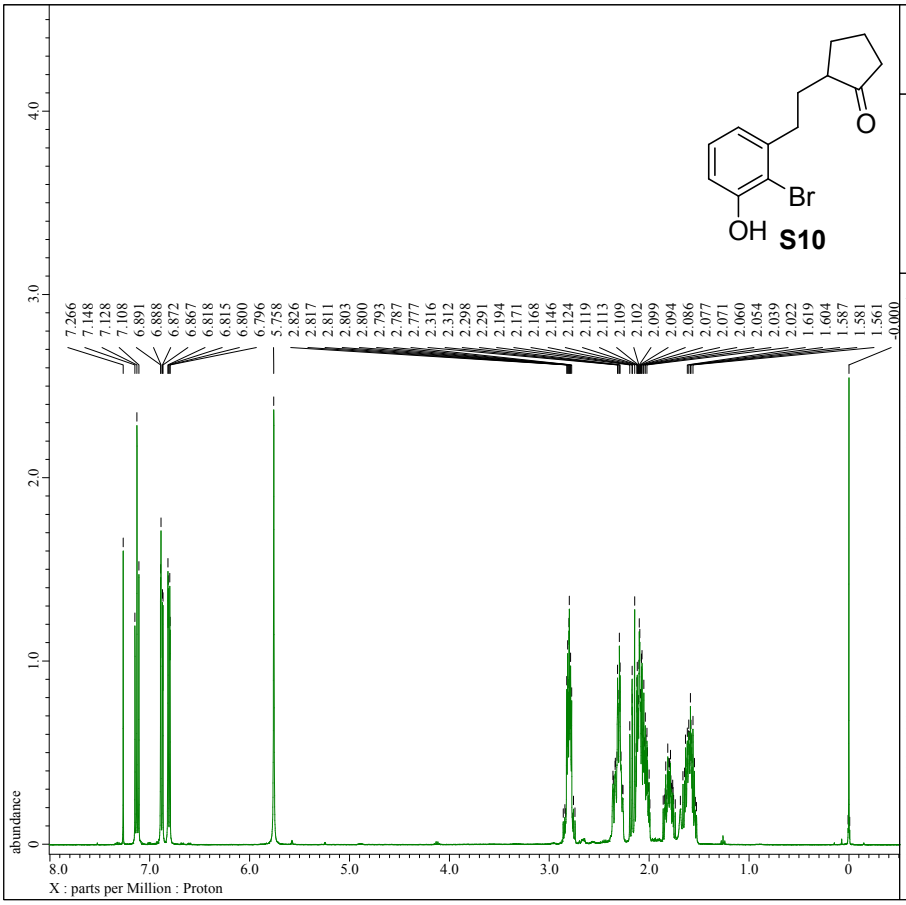

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 205_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 205
Solvent           = CHLOROFORM-D
Creation_Time     = 15-JUN-2012 21:30:31
Revision_Time     = 20-SEP-2014 17:53:27
Current_Time      = 20-SEP-2014 17:53:37

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.9[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



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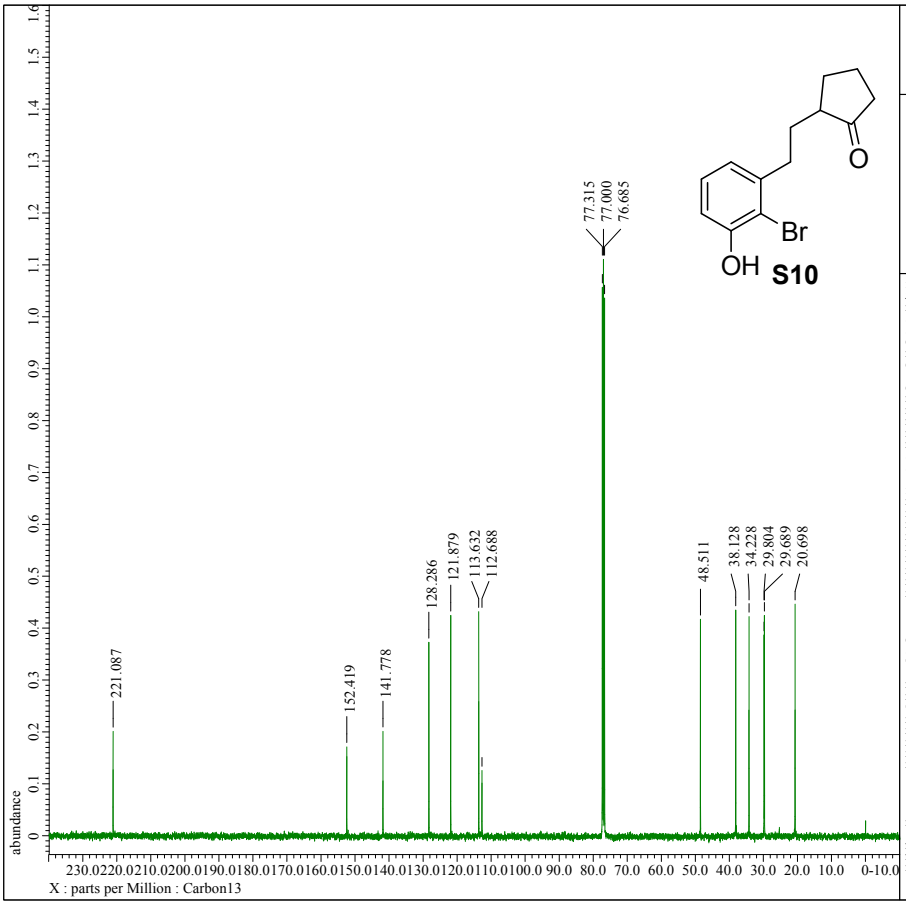
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 211_Proton-2-12.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 211
Solvent           = CHLOROFORM-D
Creation_Time     = 21-SEP-2014 16:42:13
Revision_Time     = 21-SEP-2014 17:14:50
Current_Time      = 21-SEP-2014 17:16:29

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.0024096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 36
Temp_Get          = 20.2[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



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```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

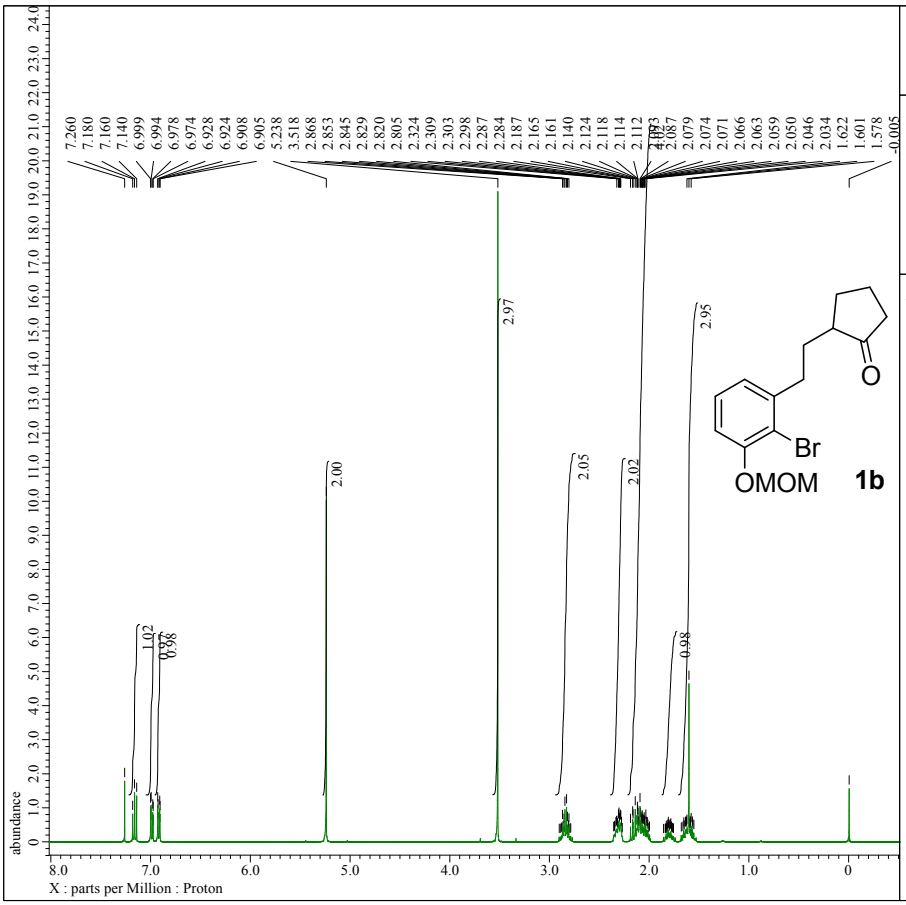
□ □ □ □ □ □: 211_Carbon-2-1.jdf
```

```
Filename          = 211_Carbon-2-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 211
Solvent           = CHLOROFORM-D
Creation_Time     = 21-SEP-2014 16:43:45
Revision_Time     = 21-SEP-2014 17:06:24
Current_Time      = 21-SEP-2014 17:06:37

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 115.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 20.6[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

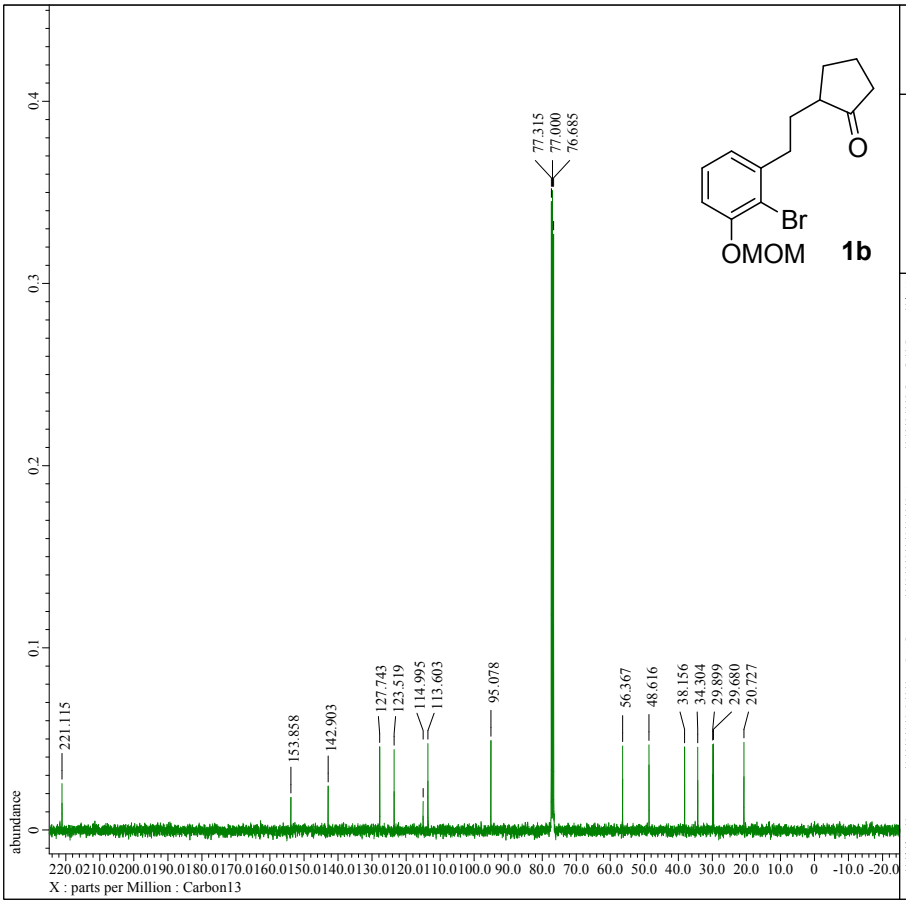
□□□□□□: 283-pure_Proton-1-1.jdf
```

```
Filename          = 283-pure_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 283-pure
Solvent           = CHLOROFORM-D
Creation_Time     = 23-OCT-2012 19:38:44
Revision_Time     = 21-SEP-2014 17:37:18
Current_Time      = 21-SEP-2014 17:39:30

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.0024096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 18.7[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

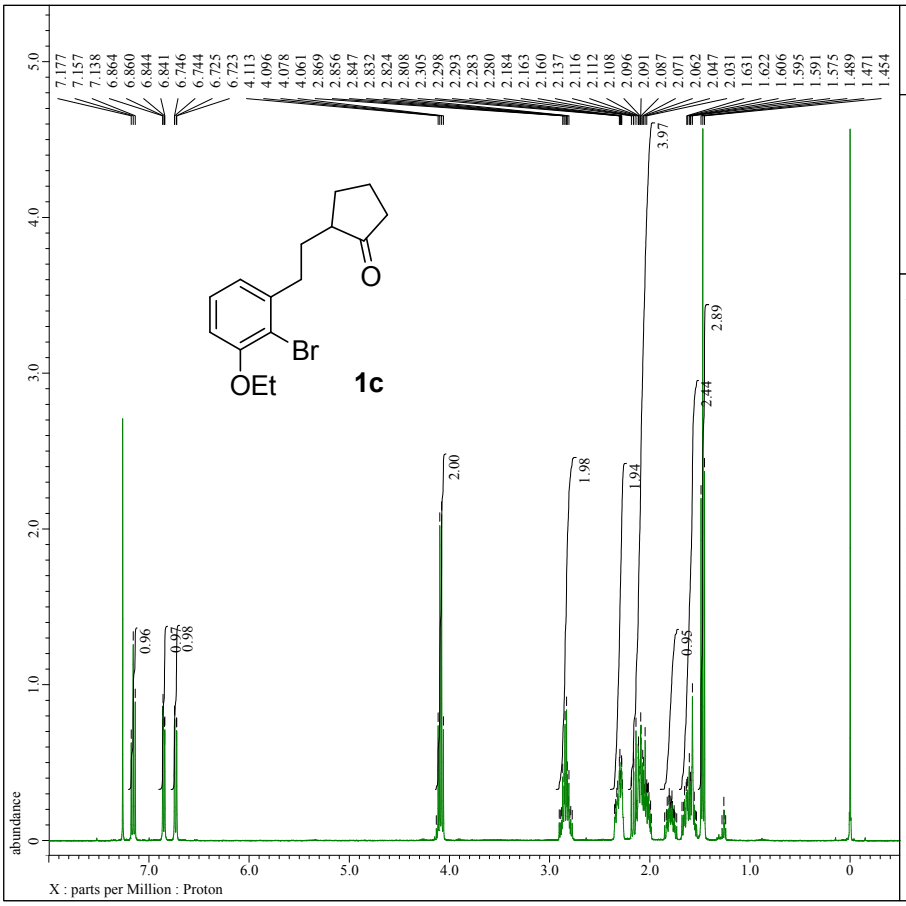
□□□□□□: 283-pure_Carbon-1-1.jdf
```

```
Filename          = 283-pure_Carbon-1-5.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 283-pure
Solvent           = CHLOROFORM-D
Creation_Time     = 23-OCT-2012 19:40:17
Revision_Time     = 21-SEP-2014 17:44:24
Current_Time      = 21-SEP-2014 17:44:43

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 188
Total_Scans       = 188

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.2[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



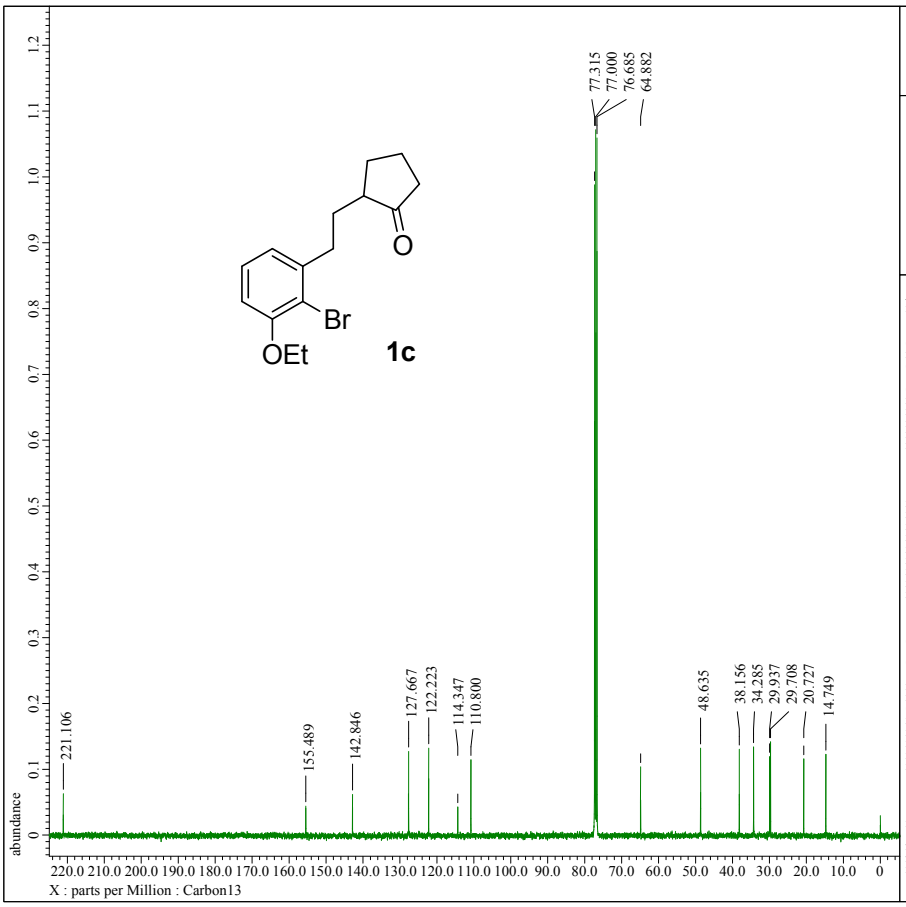

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-211-1_Proton-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-211-1
Solvent           = CHLOROFORM-D
Creation_Time     = 28-JUN-2014 17:56:34
Revision_Time     = 21-SEP-2014 17:55:40
Current_Time      = 21-SEP-2014 17:55:48

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay = 5[s]
Recvr_Gain        = 42
Temp_Get          = 20.6[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



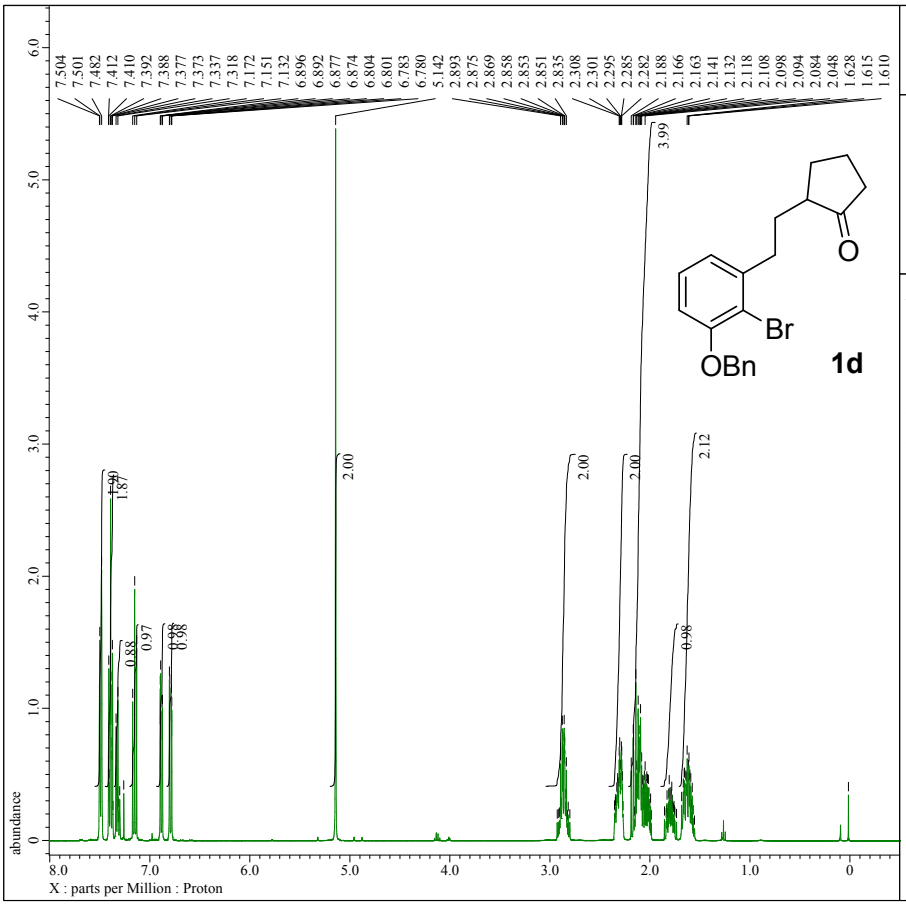

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-211-1_Carbon-1-5.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-211-1
Solvent           = CHLOROFORM-D
Creation_Time     =  1-JUL-2014 10:05:11
Revision_Time     = 21-SEP-2014 17:52:25
Current_Time      = 21-SEP-2014 17:52:38

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

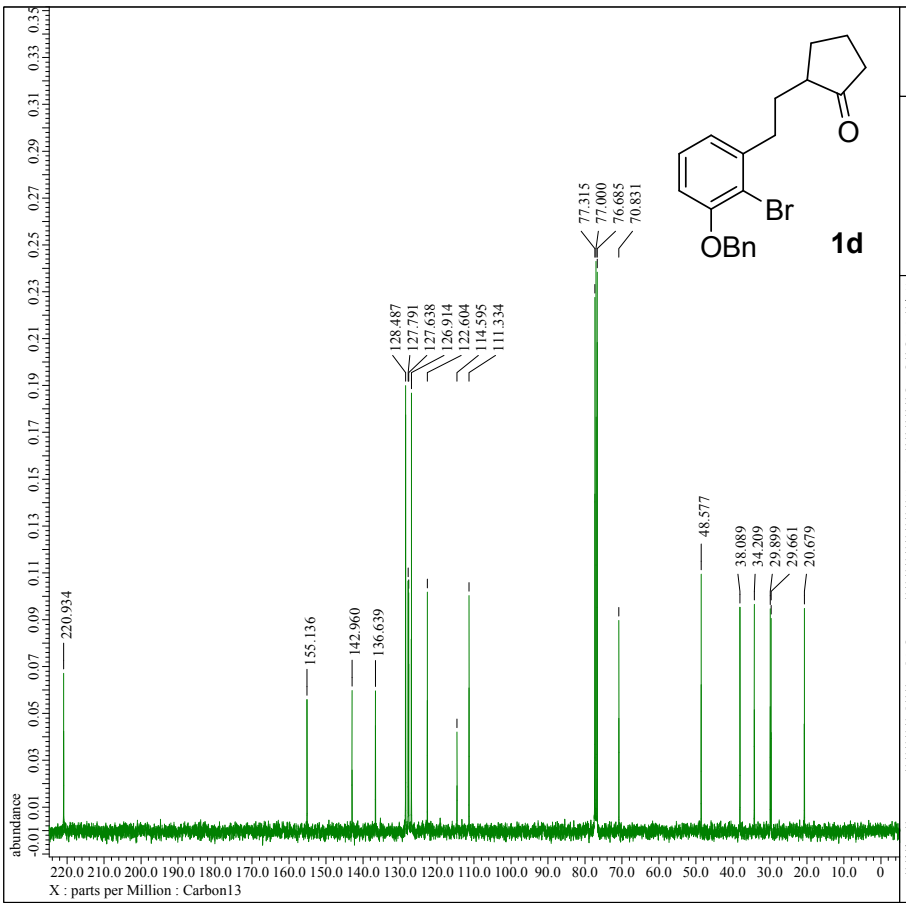
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.2[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
traperoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

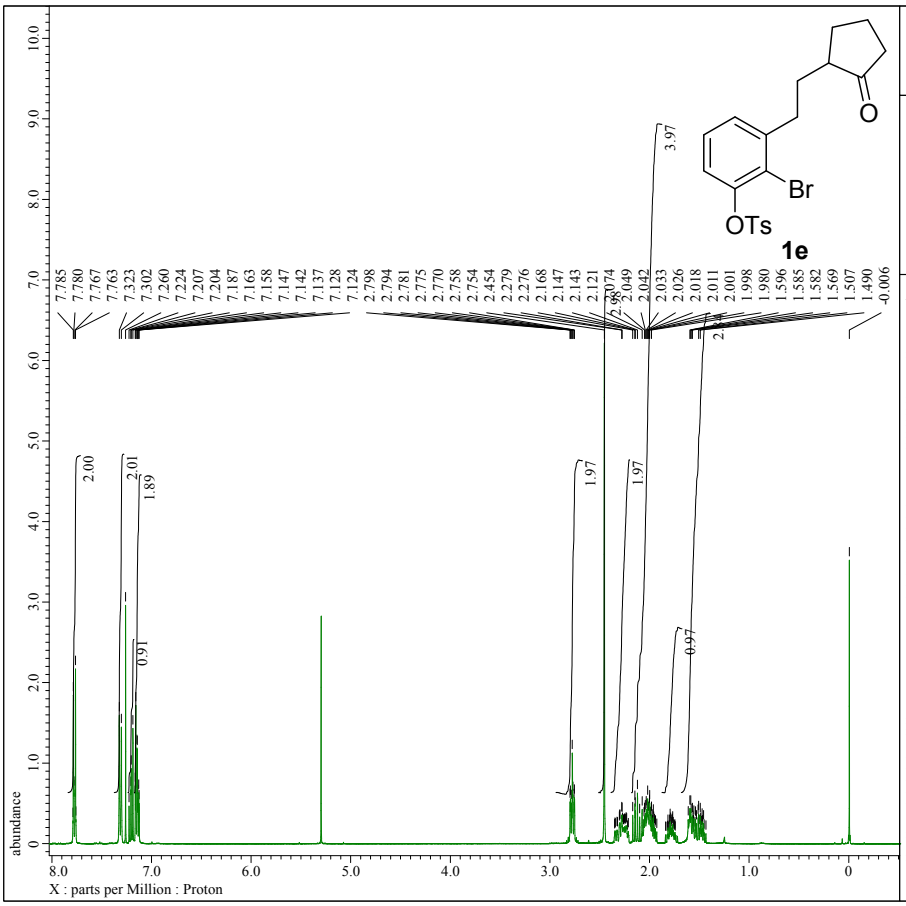
Filename	= 256_Proton-1-3.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= 256
Solvent	= CHLOROFORM-D
Creation_Time	= 10-SEP-2012 21:15:08
Revision_Time	= 21-SEP-2014 18:18:53
Current_Time	= 21-SEP-2014 18:19:24
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 28
Temp_Get	= 24.9[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 1.5[dB]
X_Pulse	= 5.5[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]



---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
traperoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

□□□□□□: 256_Carbon-1-1.jdf

Filename	= 256_Carbon-1-3.jdf
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= 256
Solvent	= CHLOROFORM-D
Creation_Time	= 10-SEP-2012 21:16:51
Revision_Time	= 21-SEP-2014 17:59:27
Current_Time	= 21-SEP-2014 18:00:01
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 101
Total_Scans	= 101
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 25.3[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 5.6[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 21[dB]
Irr_Atn_Noe	= 21[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

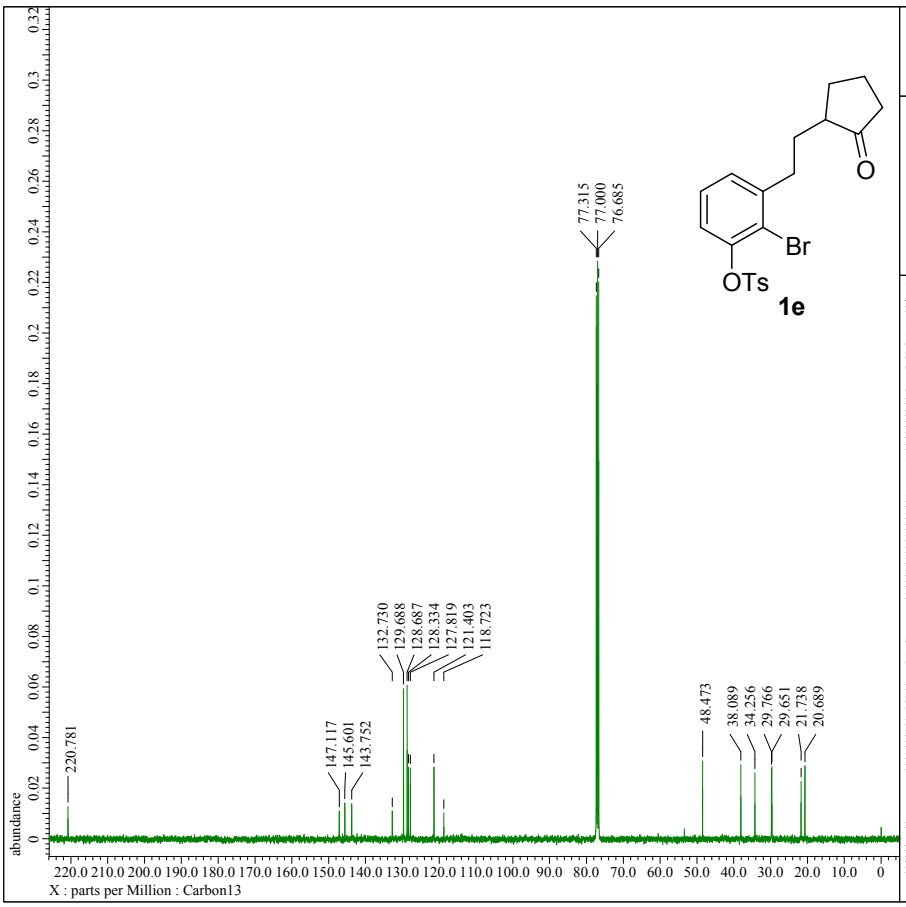
□ □ □ □ □ □: T-231-1_Proton-1-1.jdf
```

```
Filename          = T-231-1_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-231-1
Solvent           = CHLOROFORM-D
Creation_Time     = 18-SEP-2014 11:51:01
Revision_Time     = 21-SEP-2014 18:25:26
Current_Time      = 21-SEP-2014 18:27:58

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay = 5[s]
Recvr_Gain        = 40
Temp_Get          = 19.4[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
DanTe_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

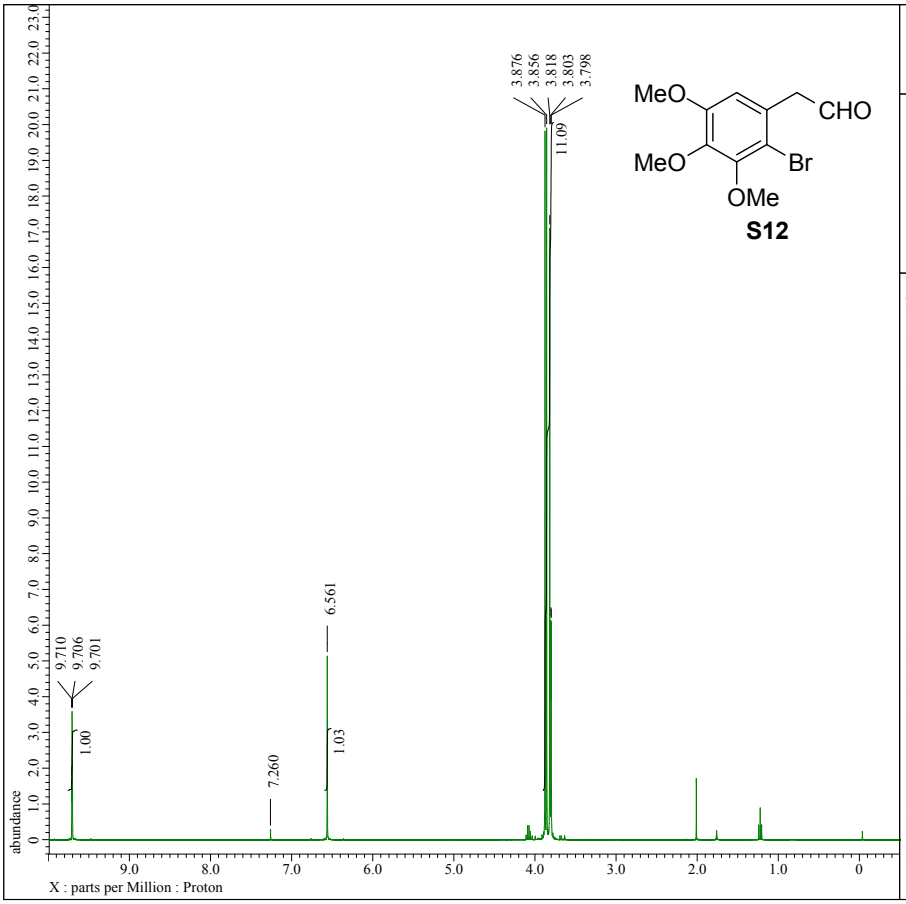
□ □ □ □ □ □: T-231-1_Carbon-1-1.jdf
```

```
Filename          = T-231-1_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-231-1
Solvent           = CHLOROFORM-D
Creation_Time     = 18-SEP-2014 11:52:14
Revision_Time     = 21-SEP-2014 18:29:09
Current_Time      = 21-SEP-2014 18:29:28

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 120[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 512
Total_Scans       = 512

Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 20.5[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21[dB]
Irr_Atn_Noe       = 21[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



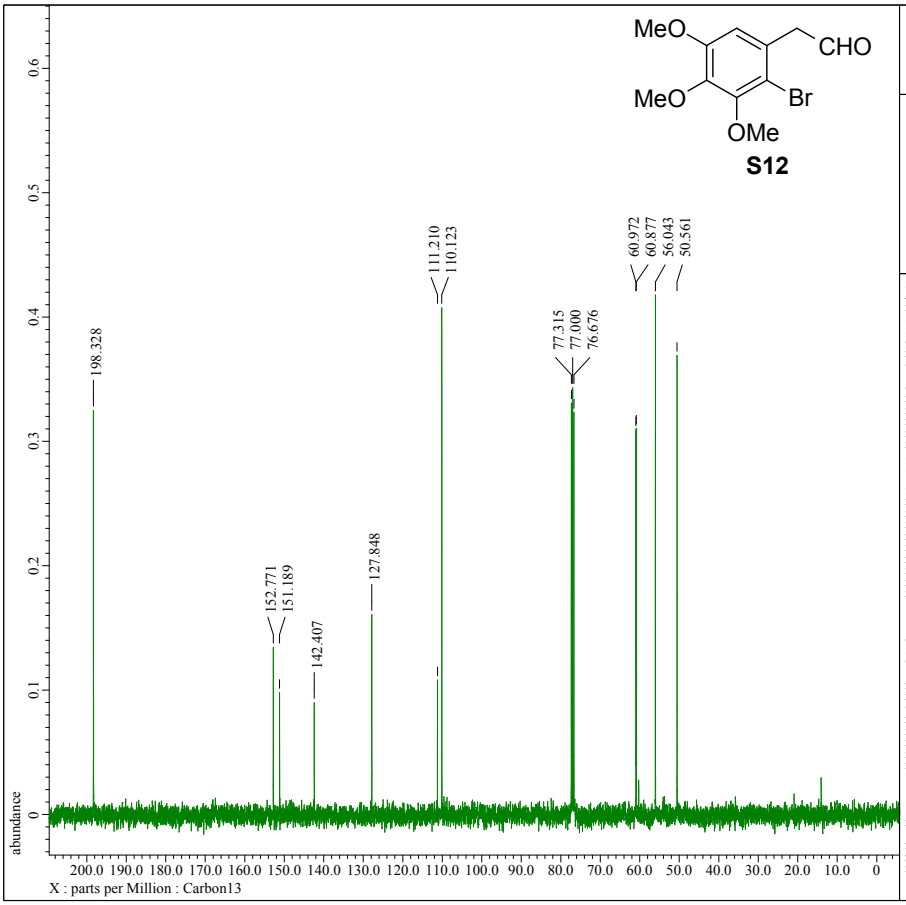

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = III-161-2_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = III-161-2
Solvent           = CHLOROFORM-D
Creation_Time     = 14-APR-2014 21:45:25
Revision_Time     = 23-SEP-2014 20:38:28
Current_Time      = 23-SEP-2014 20:38:45

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 18.9[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



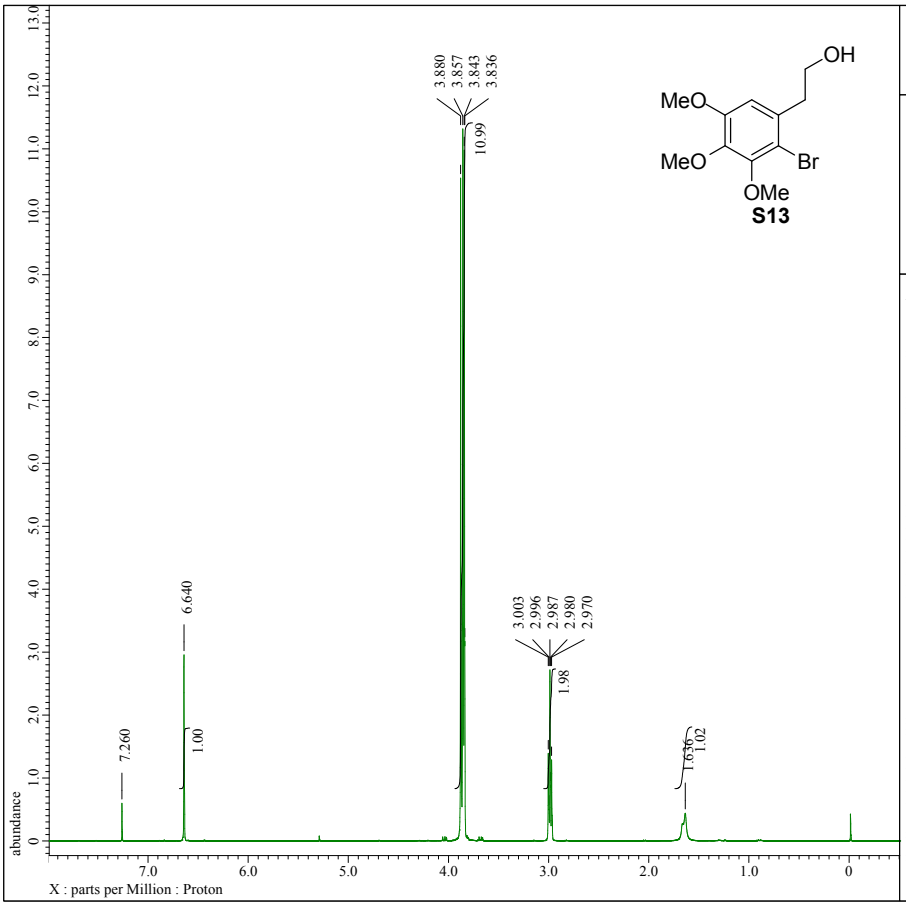

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = III-161-2_Carbon-2-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = III-161-2
Solvent           = CHLOROFORM-D
Creation_Time     = 14-APR-2014 21:53:35
Revision_Time     = 23-SEP-2014 20:52:14
Current_Time      = 23-SEP-2014 20:54:16

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 19
Total_Scans       = 19

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.2[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21[dB]
Irr_Atn_Noe       = 21[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



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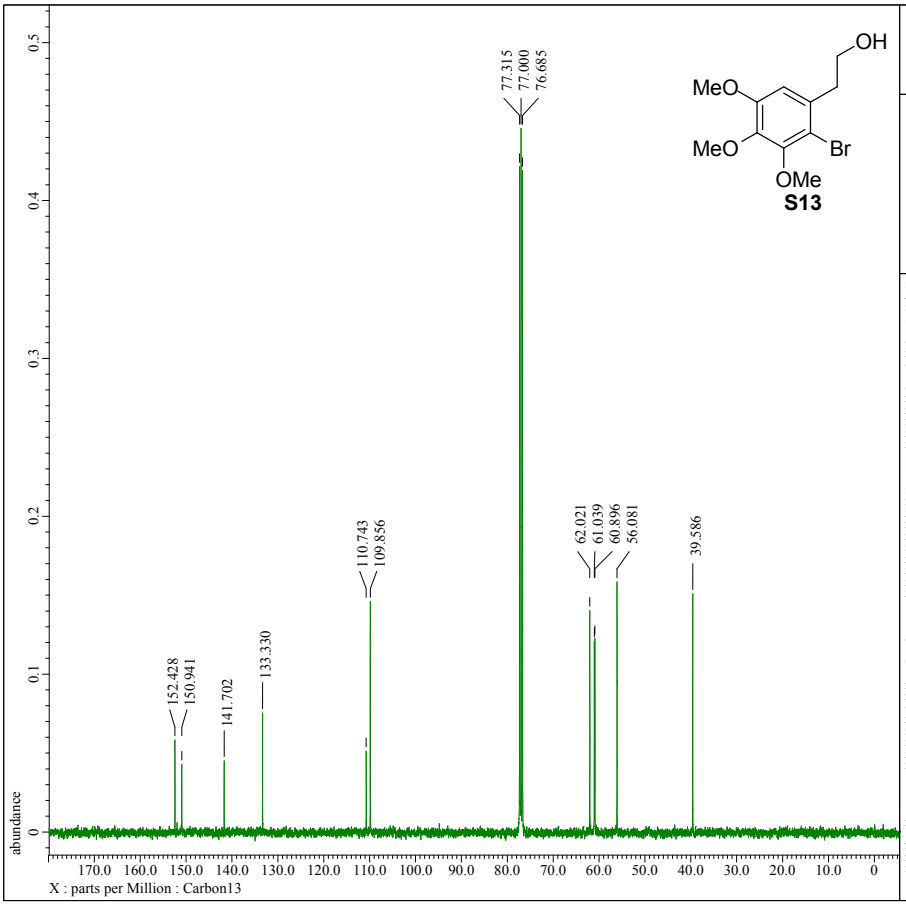
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-169-1_Proton-2-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-169-1
Solvent           = CHLOROFORM-D
Creation_Time     = 30-APR-2014 23:22:23
Revision_Time     = 23-SEP-2014 21:15:46
Current_Time      = 23-SEP-2014 21:16:04

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 32
Temp_Get          = 18[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



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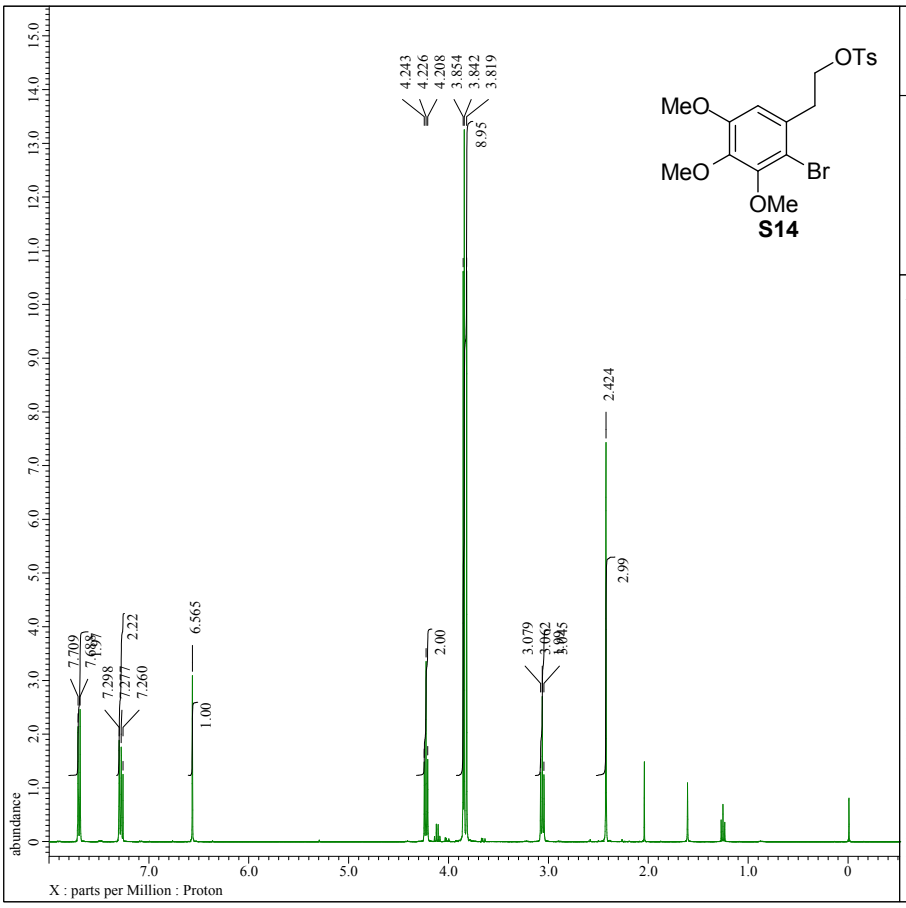
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-169-1_Carbon-2-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-169-1
Solvent           = CHLOROFORM-D
Creation_Time     = 30-APR-2014 23:23:59
Revision_Time     = 30-APR-2014 23:32:46
Current_Time      = 23-SEP-2014 21:23:37

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

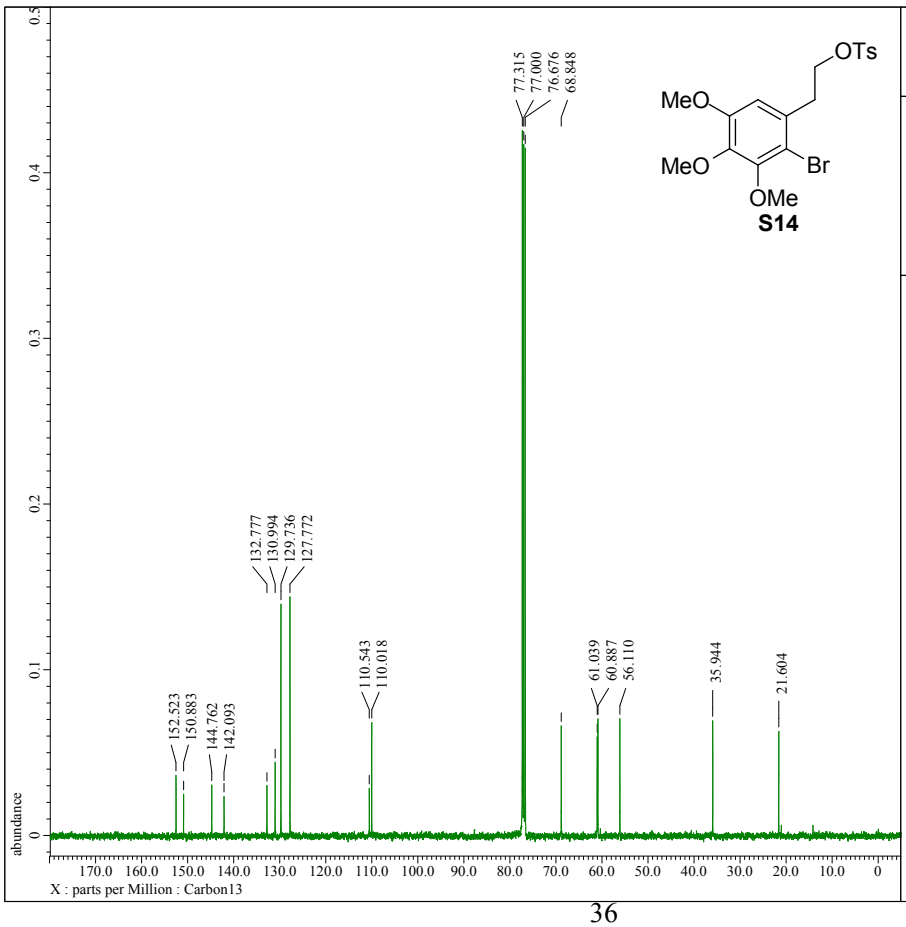
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped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

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 18.3[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



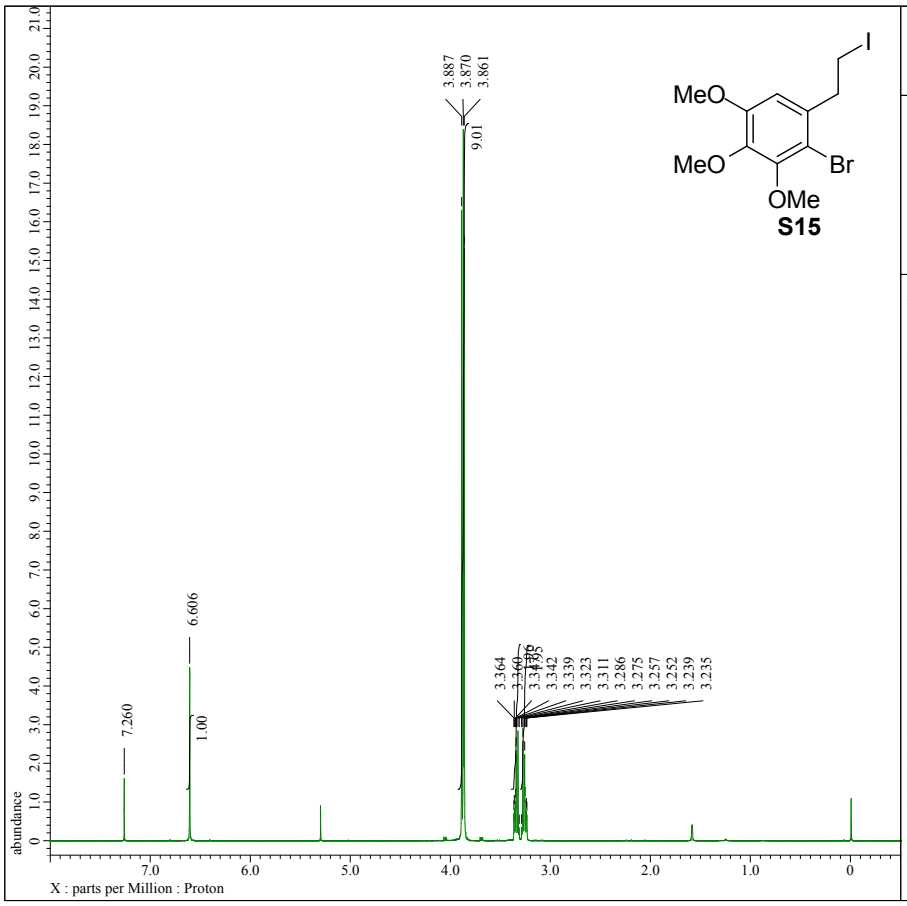
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

Filename	= T-170-1_Proton-1-5.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= T-170-1
Solvent	= CHLOROFORM-D
Creation_Time	= 2-MAY-2014 20:39:26
Revision_Time	= 23-SEP-2014 21:21:36
Current_Time	= 23-SEP-2014 21:21:48
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.0024096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 36
Temp_Get	= 18.5[dC]
X_90_Width	= 12.1[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 6.05[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]



---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

Filename	= T-170-1_Carbon-1-5.jdf
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= T-170-1
Solvent	= CHLOROFORM-D
Creation_Time	= 2-MAY-2014 20:40:40
Revision_Time	= 2-MAY-2014 21:02:22
Current_Time	= 23-SEP-2014 21:37:30
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 512
Total_Scans	= 512
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 18.7[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 21.63[dB]
Irr_Atn_Noe	= 21.63[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]



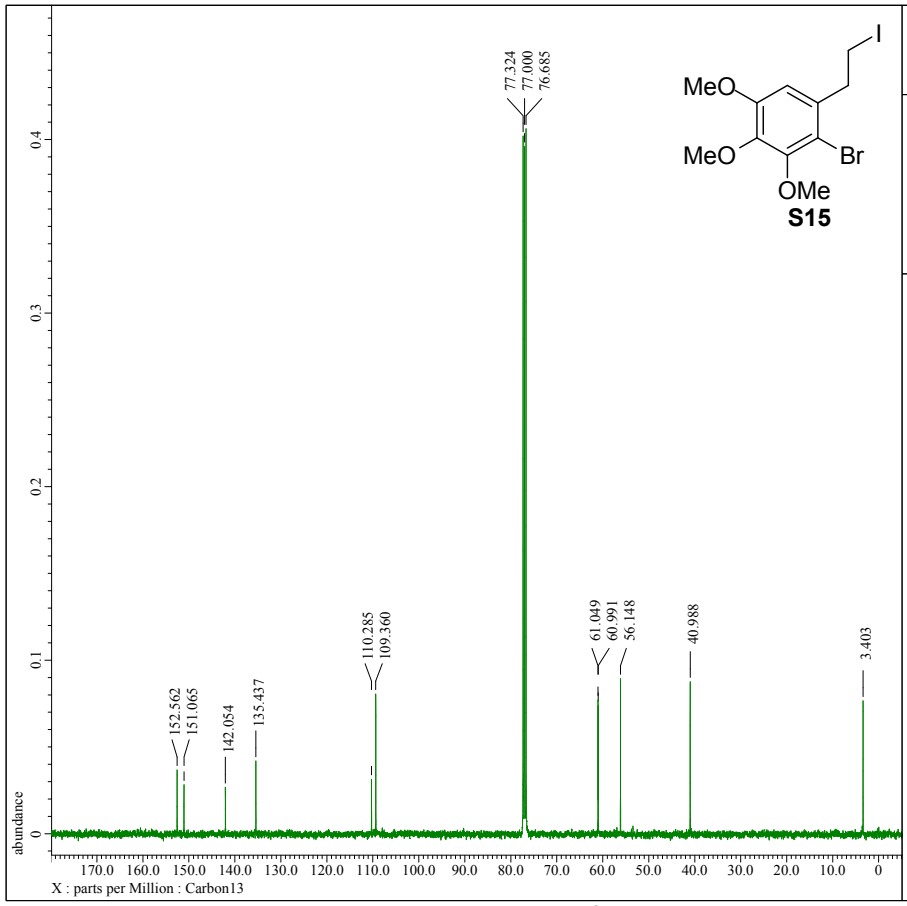

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-173-1_Proton-1-6.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-173-1
Solvent           = CHLOROFORM-D
Creation_Time     =  7-MAY-2014 21:55:17
Revision_Time     = 23-SEP-2014 21:45:12
Current_Time      = 23-SEP-2014 21:45:25

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 38
Temp_Get          = 17.9[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



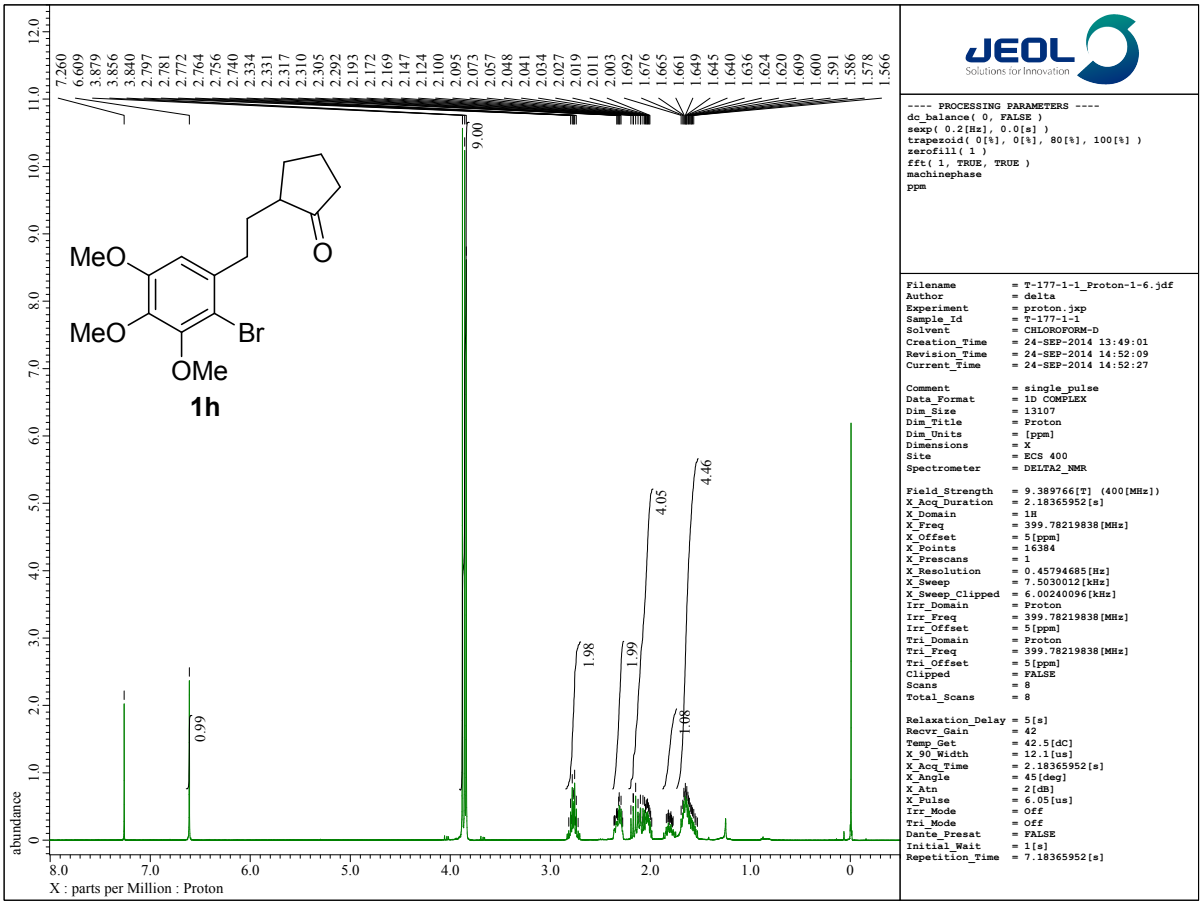

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-173-1_Carbon-1-5.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-173-1
Solvent           = CHLOROFORM-D
Creation_Time     =  7-MAY-2014 21:56:29
Revision_Time     =  7-MAY-2014 22:18:01
Current_Time      = 23-SEP-2014 21:51:21

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 18.1[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



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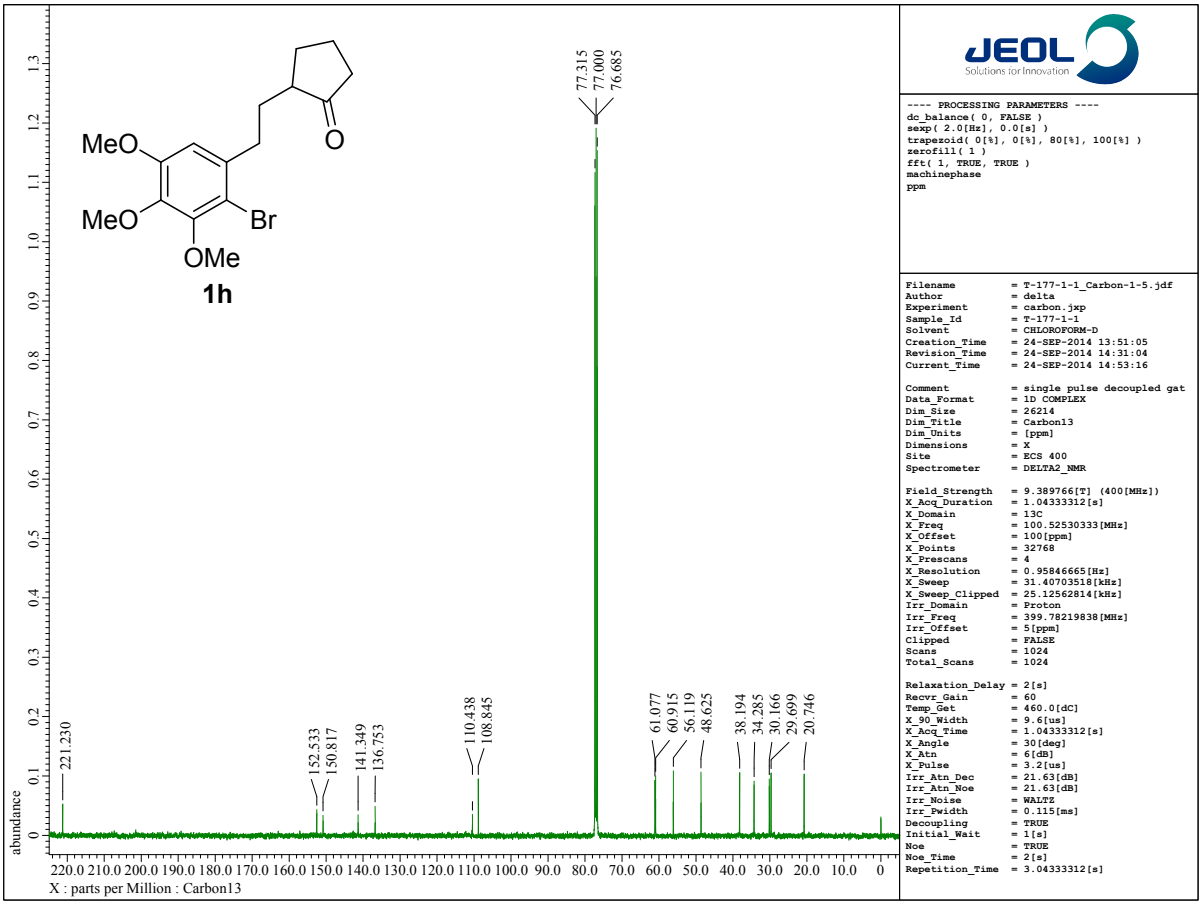
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

Filename	= T-177-1-1_Proton-1-6.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= T-177-1-1
Solvent	= CHLOROFORM-D
Creation_Time	= 24-SEP-2014 13:49:01
Revision_Time	= 24-SEP-2014 14:52:09
Current_Time	= 24-SEP-2014 14:52:27
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 42
Temp_Get	= 42.5[dC]
X_90_Width	= 12.1[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 6.05[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]



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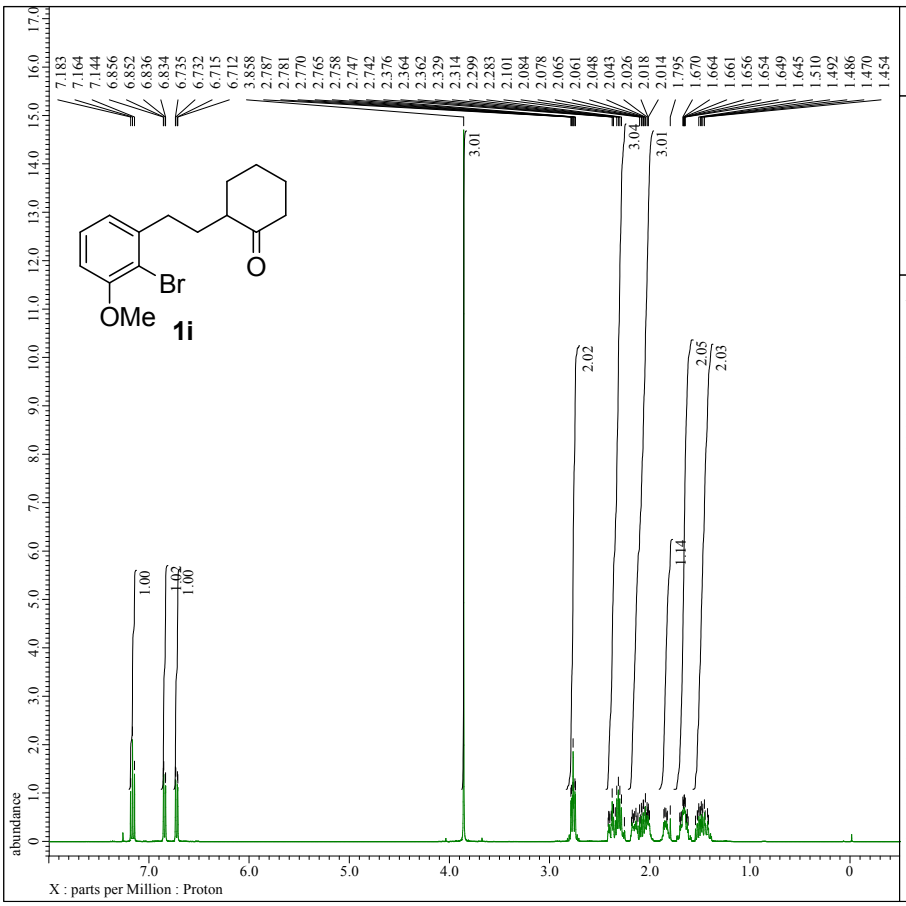
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

Filename	= T-177-1-1_Carbon-1-5.jdf
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= T-177-1-1
Solvent	= CHLOROFORM-D
Creation_Time	= 24-SEP-2014 13:51:05
Revision_Time	= 24-SEP-2014 14:31:04
Current_Time	= 24-SEP-2014 14:53:16
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 1024
Total_Scans	= 1024
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 460.0[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 21.63[dB]
Irr_Atn_Noe	= 21.63[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]



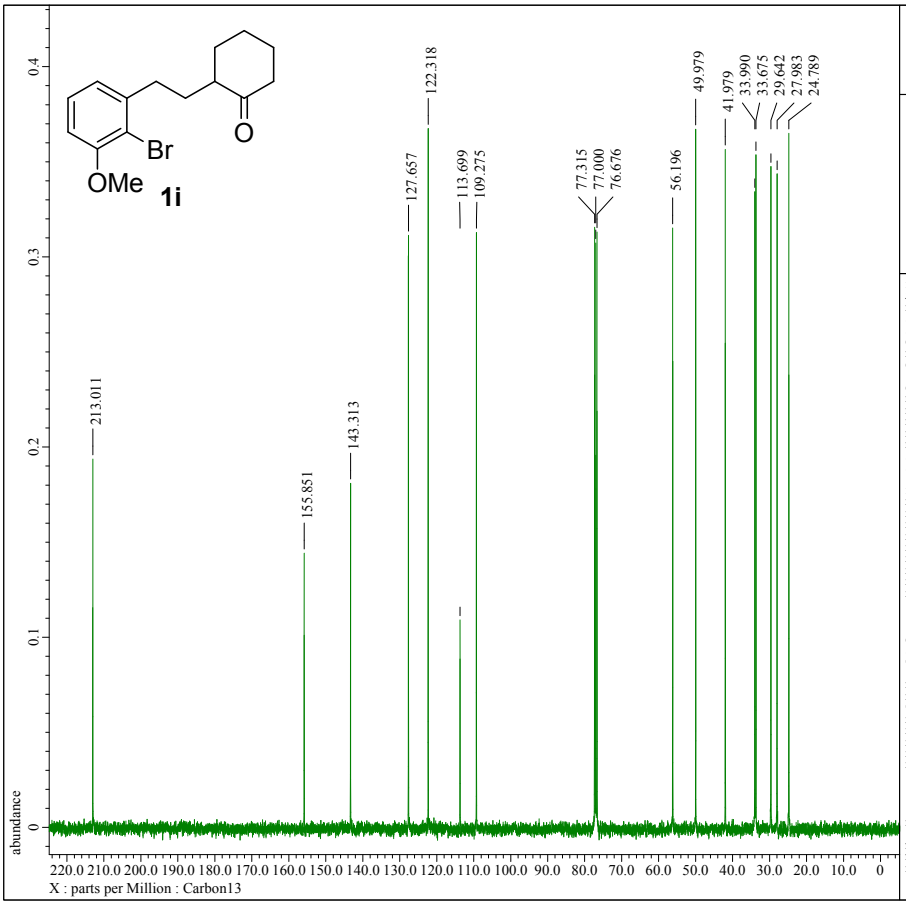

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 394_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 394
Solvent           = CHLOROFORM-D
Creation_Time     = 20-MAR-2013 20:12:44
Revision_Time     = 24-SEP-2014 16:31:02
Current_Time      = 24-SEP-2014 16:33:53

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 22[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



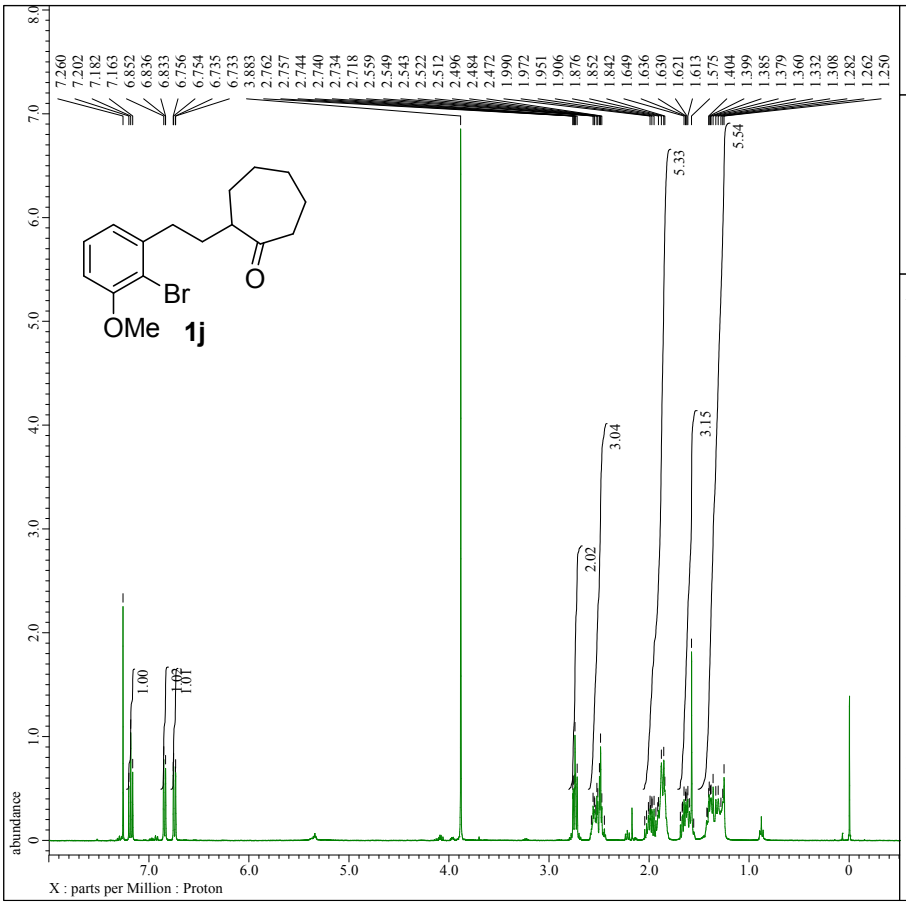

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 394_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 394
Solvent           = CHLOROFORM-D
Creation_Time     = 20-MAR-2013 20:15:34
Revision_Time     = 24-SEP-2014 16:38:40
Current_Time      = 24-SEP-2014 16:39:03

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

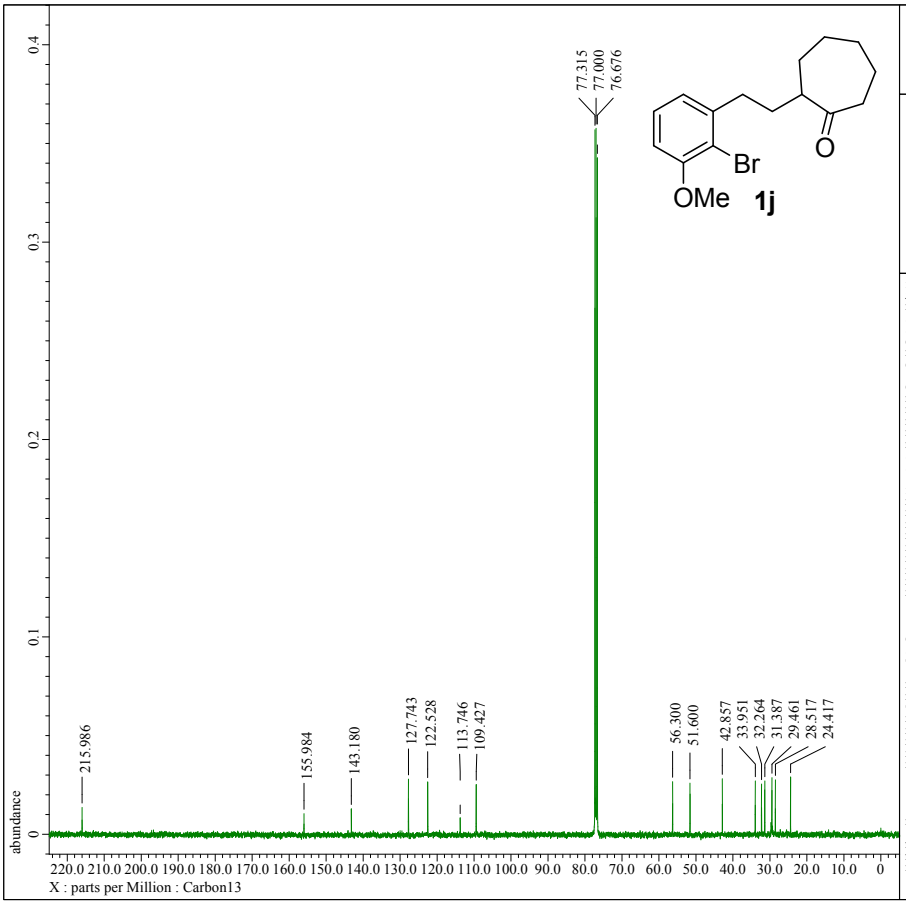
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 141
Total_Scans       = 141

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 22.3[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



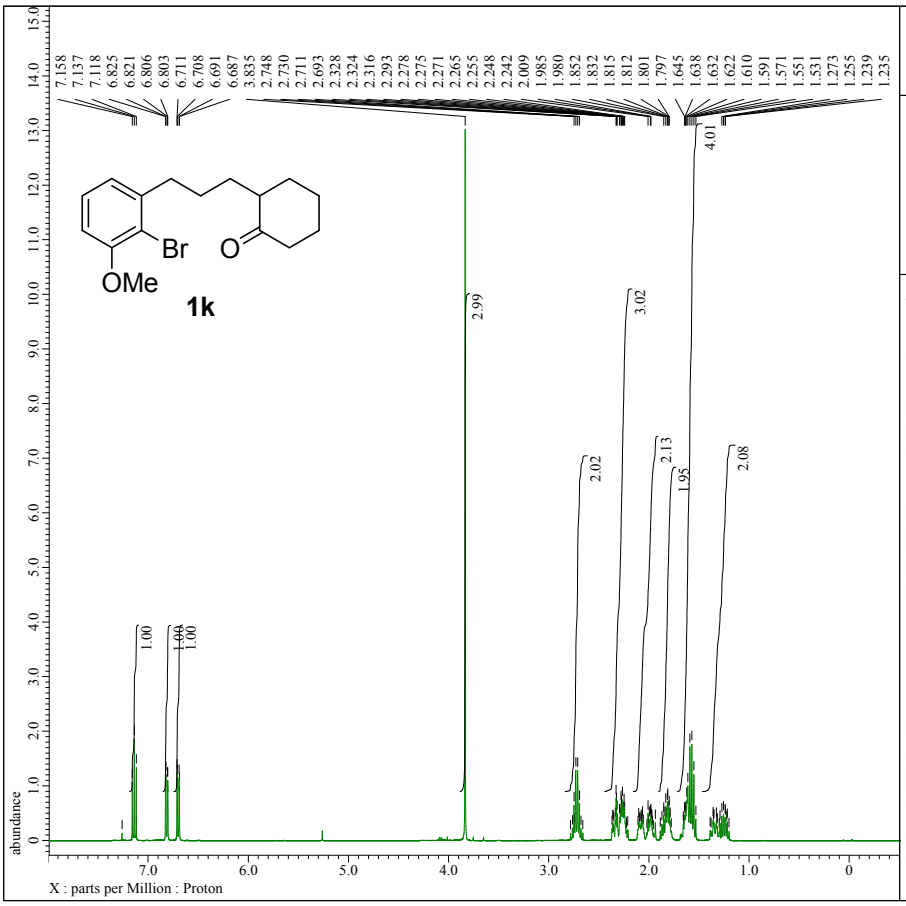
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

Filename	= 630_Proton-1-5.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= 630
Solvent	= CHLOROFORM-D
Creation_Time	= 23-JAN-2014 16:09:52
Revision_Time	= 24-SEP-2014 16:52:14
Current_Time	= 24-SEP-2014 16:52:22
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 5[s]
Recvr_Gain	= 44
Temp_Get	= 21.7[dC]
X_90_Width	= 12.1[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 6.05[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]



---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

Filename	= 630_Carbon-1-3.jdf
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= 630
Solvent	= CHLOROFORM-D
Creation_Time	= 23-JAN-2014 16:14:50
Revision_Time	= 24-SEP-2014 16:57:21
Current_Time	= 24-SEP-2014 16:57:37
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 1024
Total_Scans	= 1024
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 22[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 21.63[dB]
Irr_Atn_Noe	= 21.63[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]



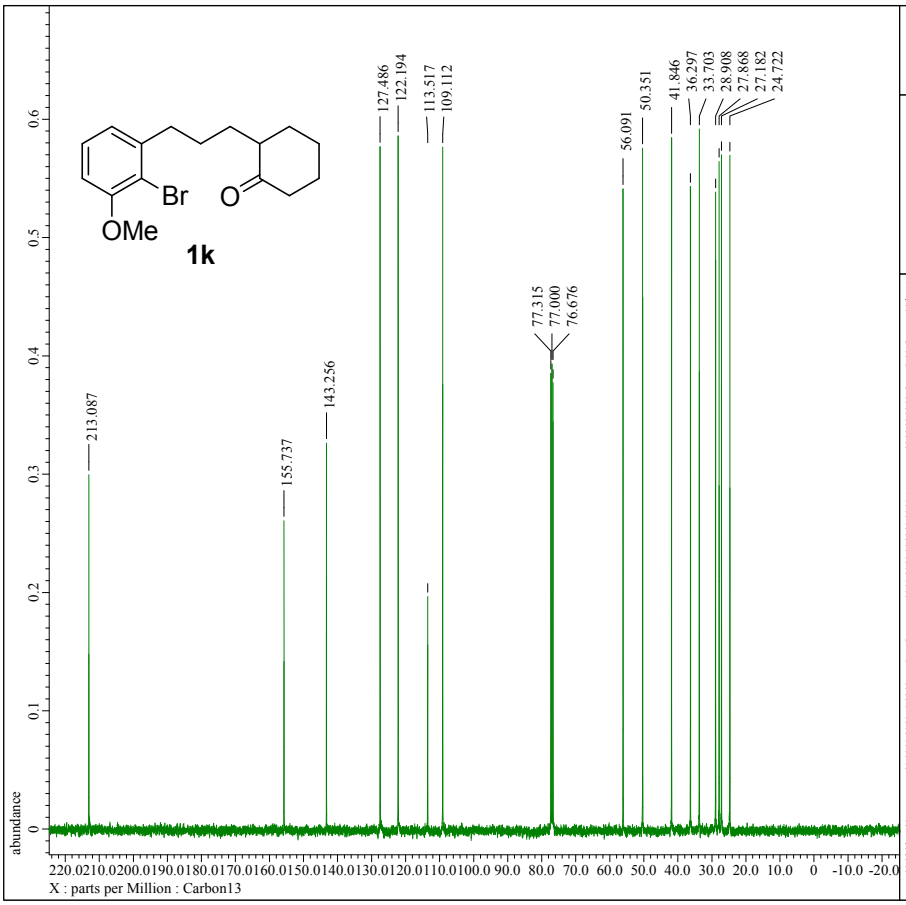

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 562-pure_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 562-pure
Solvent           = CHLOROFORM-D
Creation_Time     = 29-OCT-2013 21:59:23
Revision_Time     = 24-SEP-2014 17:07:59
Current_Time      = 24-SEP-2014 17:08:18

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 20
Temp_Get          = 19.4[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



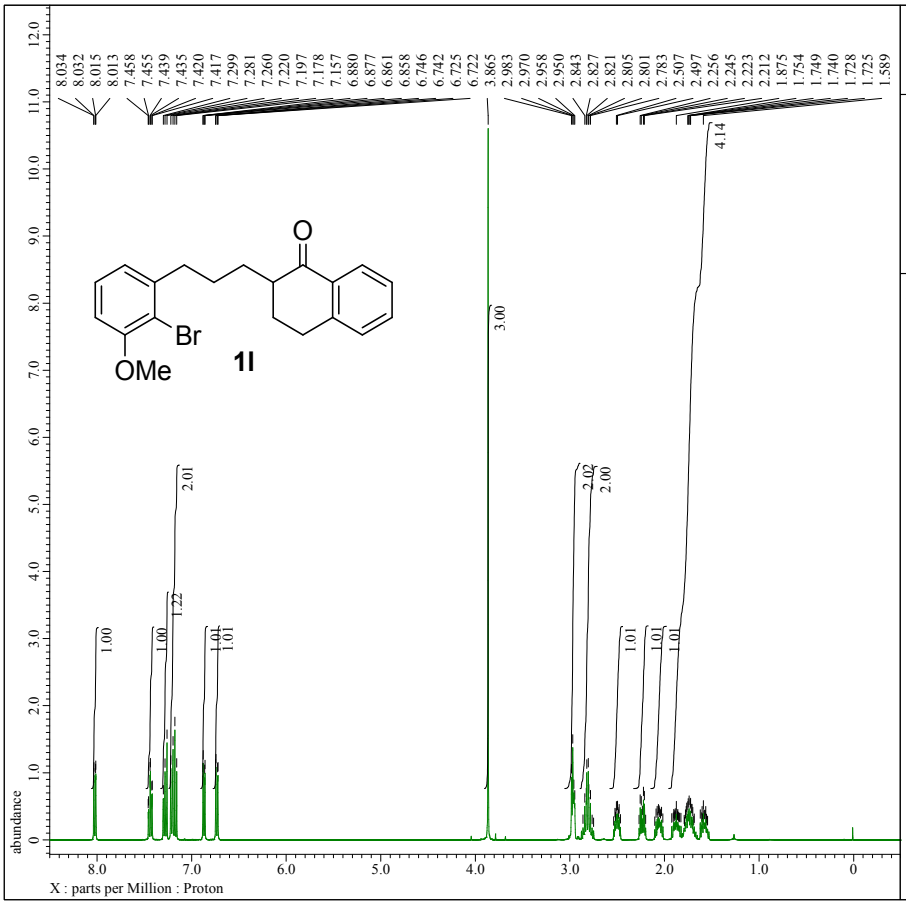

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 562-pure_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 562-pure
Solvent           = CHLOROFORM-D
Creation_Time     = 29-OCT-2013 22:01:51
Revision_Time     = 24-SEP-2014 17:09:04
Current_Time      = 24-SEP-2014 17:09:10

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 107
Total_Scans       = 107

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.7[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



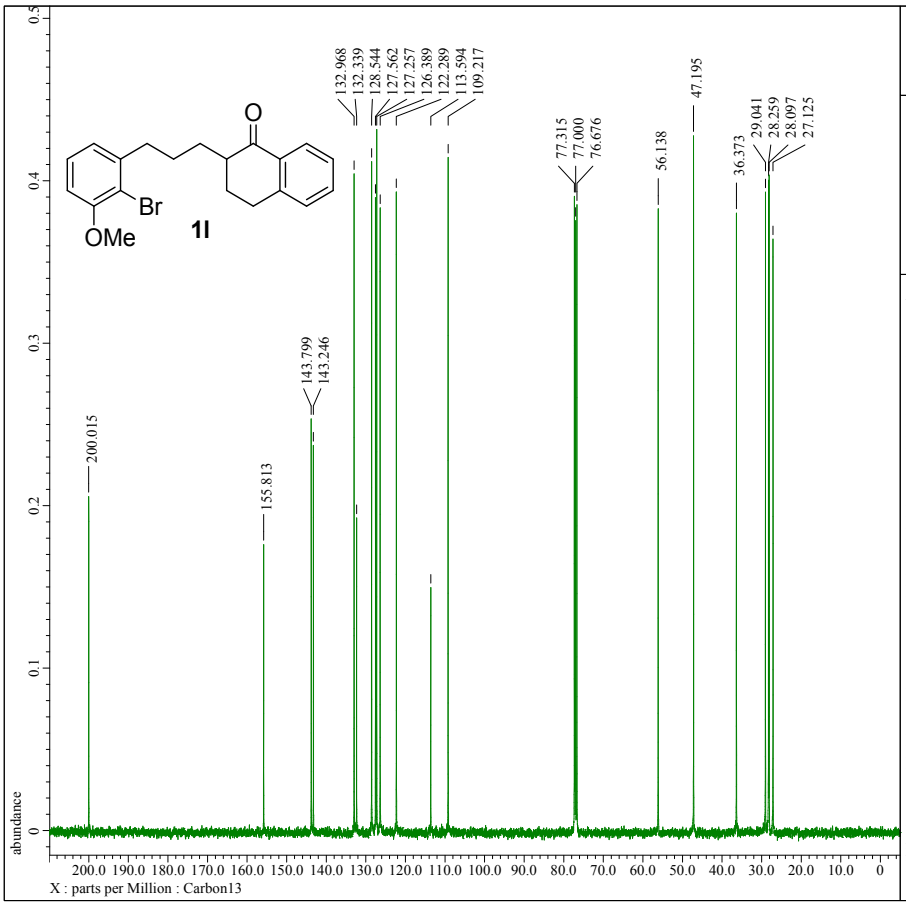

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 572-pure_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 572-pure
Solvent           = CHLOROFORM-D
Creation_Time     =  5-NOV-2013 21:30:24
Revision_Time     = 24-SEP-2014 17:20:57
Current_Time      = 24-SEP-2014 17:21:00

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 20
Temp_Get          = 32.5[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



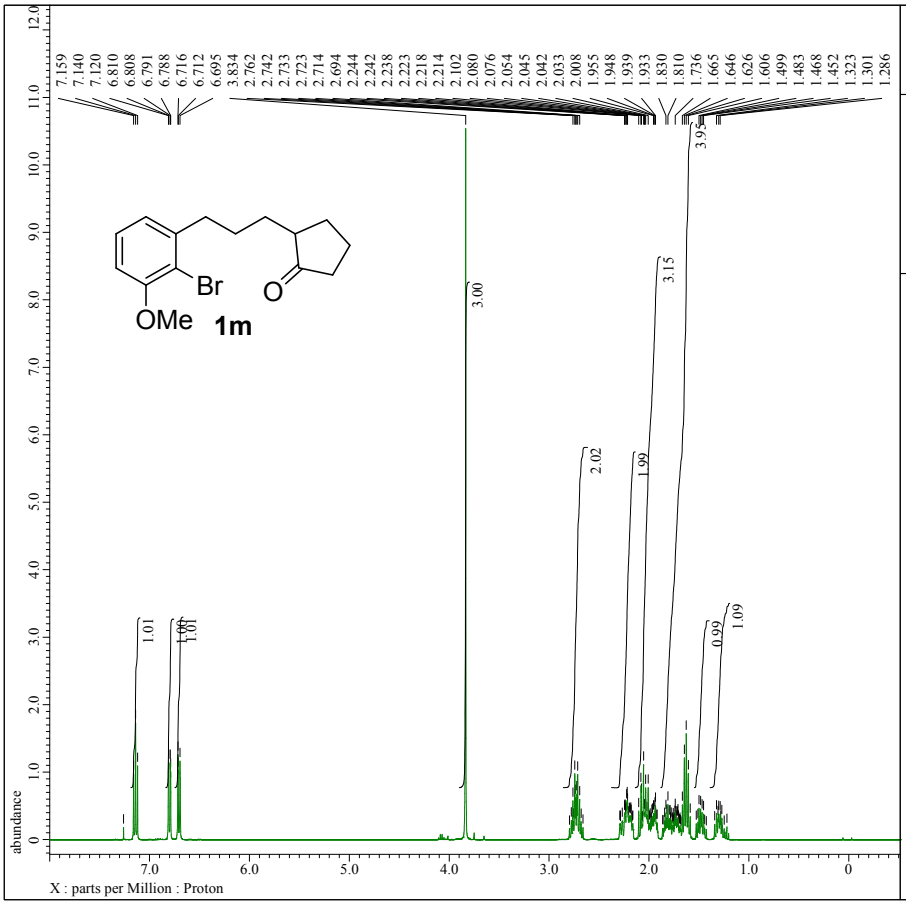

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 572-pure_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 572-pure
Solvent           = CHLOROFORM-D
Creation_Time     =  5-NOV-2013 21:32:59
Revision_Time     = 24-SEP-2014 17:22:32
Current_Time      = 24-SEP-2014 17:23:31

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 244
Total_Scans       = 244

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 33[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



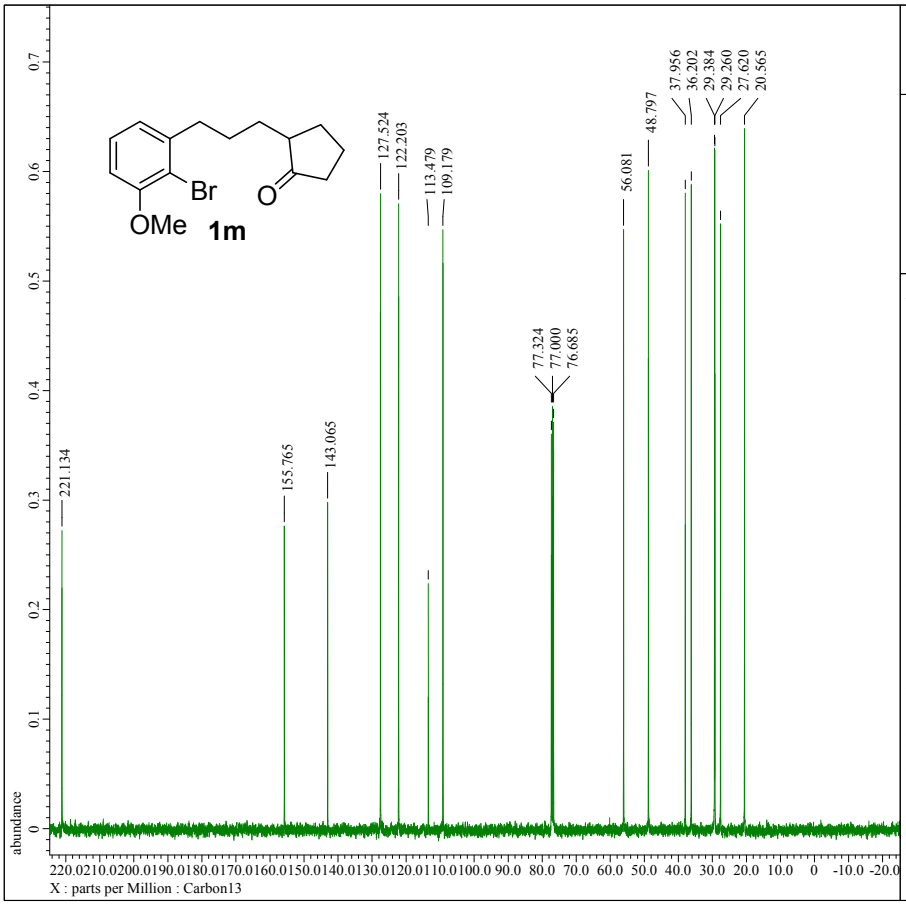

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 561-pure_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 561-pure
Solvent           = CHLOROFORM-D
Creation_Time     = 29-OCT-2013 21:03:31
Revision_Time     = 24-SEP-2014 17:31:30
Current_Time      = 24-SEP-2014 17:32:01

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 20
Temp_Get          = 19.7[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



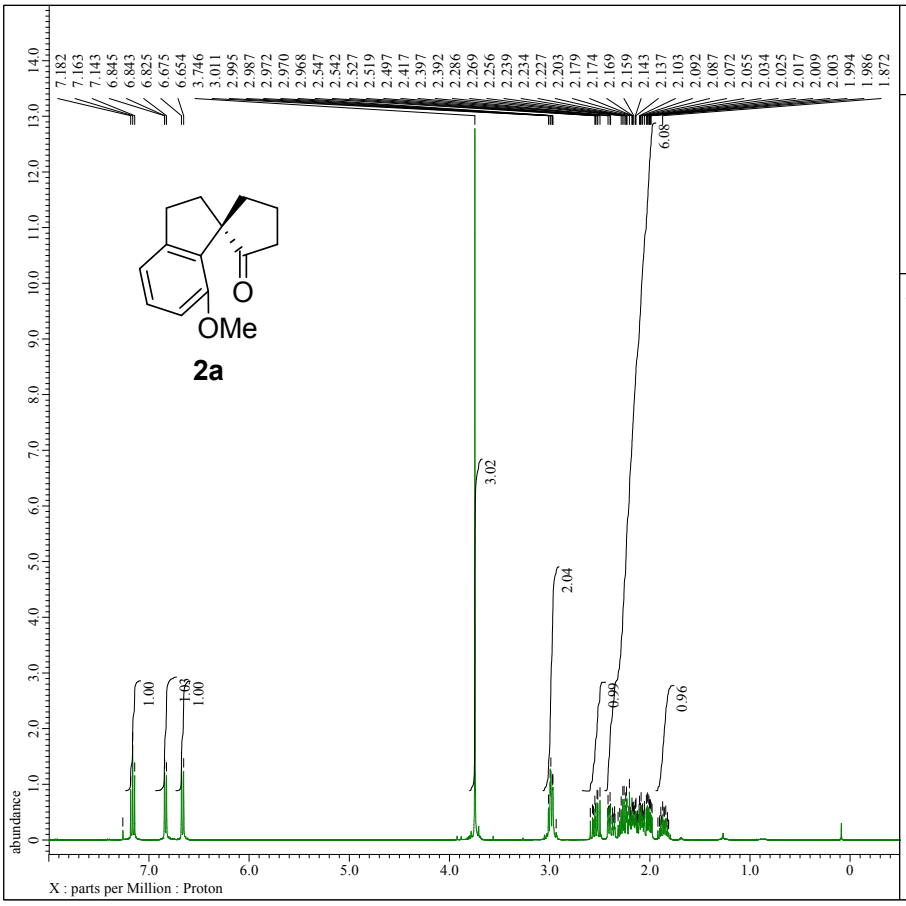

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 561-pure_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 561-pure
Solvent           = CHLOROFORM-D
Creation_Time     = 29-OCT-2013 21:05:56
Revision_Time     = 24-SEP-2014 17:37:41
Current_Time      = 24-SEP-2014 17:38:00

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 63
Total_Scans       = 63

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.9[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



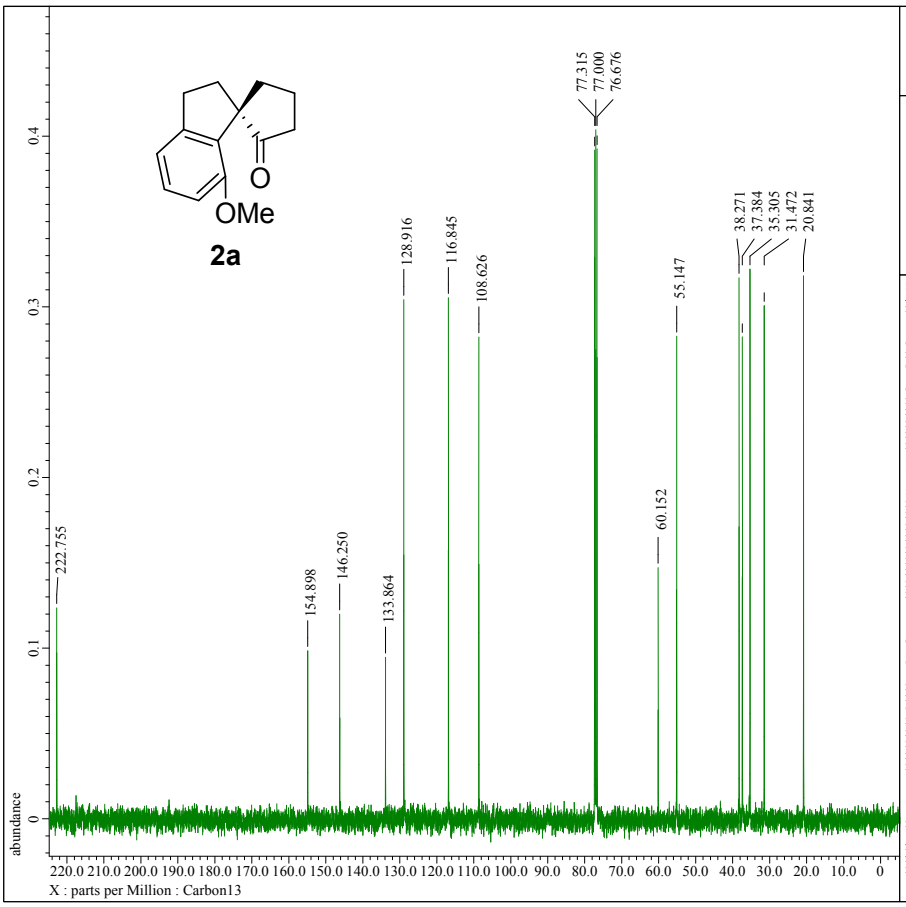

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 135_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 135
Solvent           = CHLOROFORM-D
Creation_Time     = 30-JAN-2012 16:09:56
Revision_Time     = 24-SEP-2014 17:48:04
Current_Time      = 24-SEP-2014 17:48:06

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 20.5[dC]
X_90_Width        = 12[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



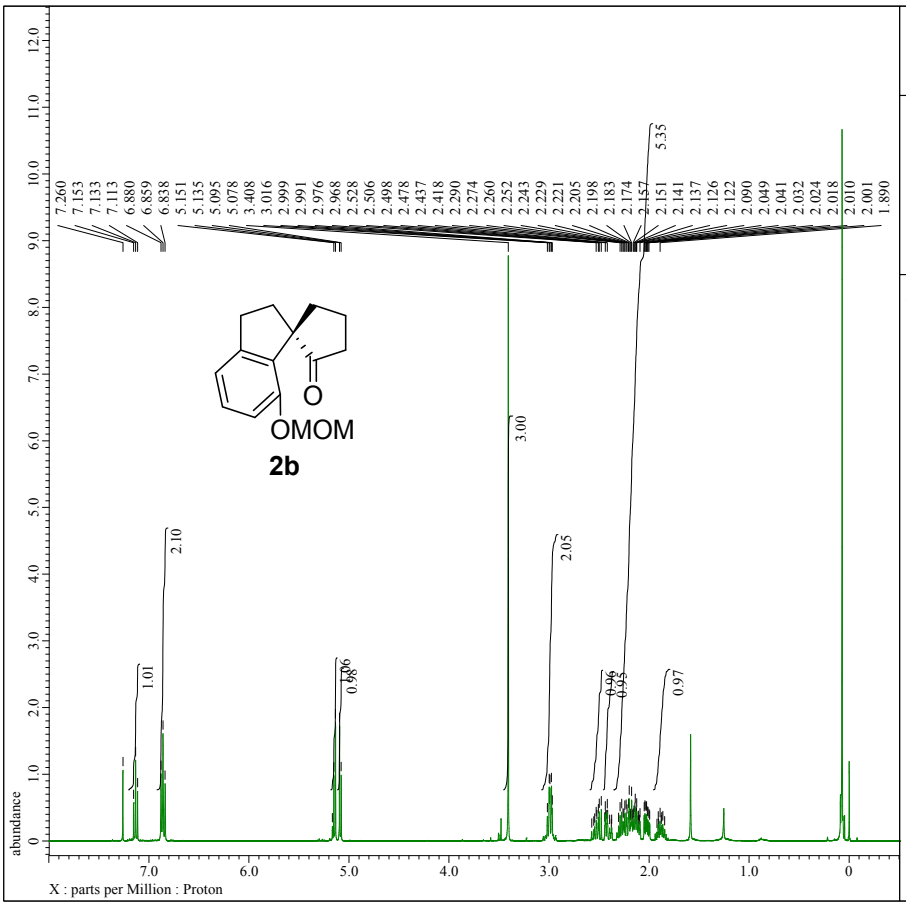

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 135_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 135
Solvent           = CHLOROFORM-D
Creation_Time     = 30-JAN-2012 16:11:33
Revision_Time     = 24-SEP-2014 17:52:49
Current_Time      = 24-SEP-2014 17:52:58

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 38
Total_Scans       = 38

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 20.6[dC]
X_90_Width        = 9.5[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.16666667[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



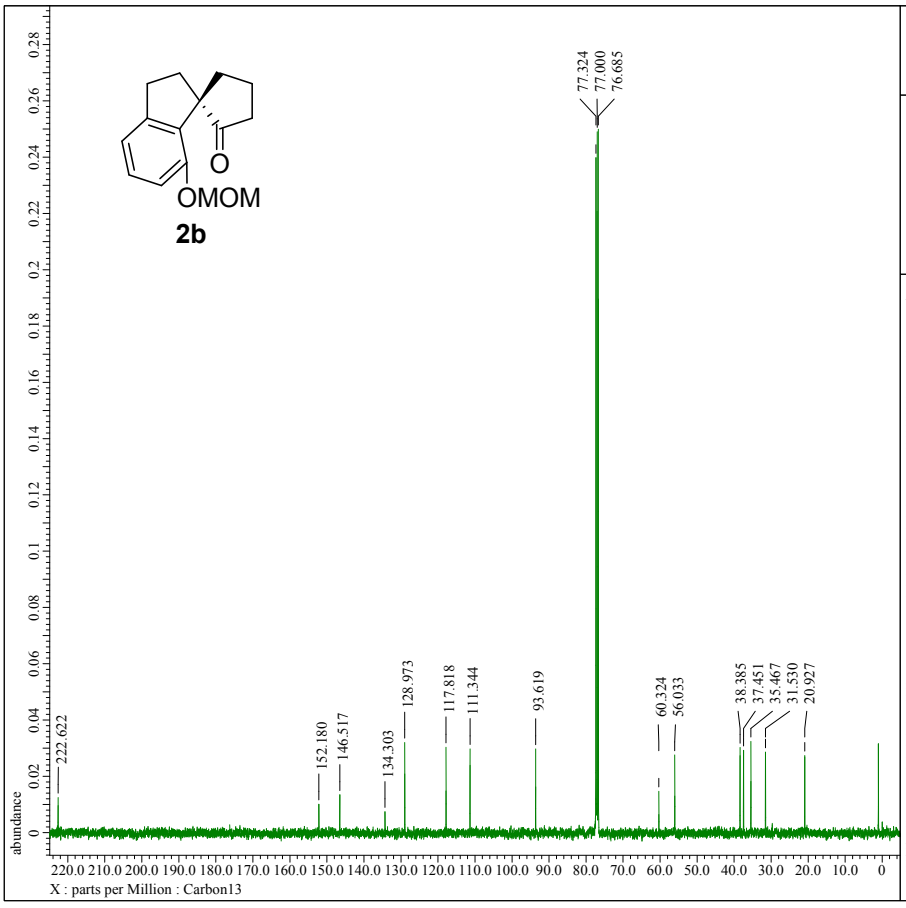

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 417_Proton-2-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 417
Solvent           = CHLOROFORM-D
Creation_Time     =  9-APR-2013 12:25:02
Revision_Time     = 24-SEP-2014 18:01:40
Current_Time      = 24-SEP-2014 18:01:44

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 18.9[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



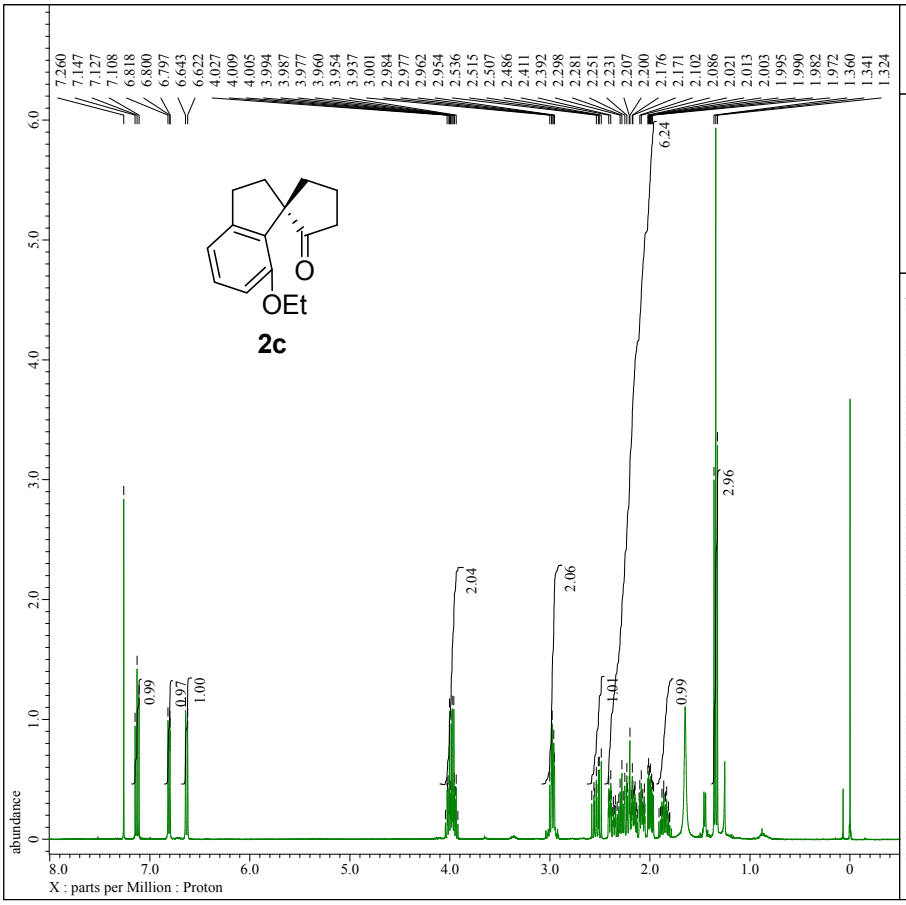

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 417_Carbon-2-5.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 417
Solvent           = CHLOROFORM-D
Creation_Time     =  9-APR-2013 12:57:14
Revision_Time     = 24-SEP-2014 18:04:46
Current_Time      = 24-SEP-2014 18:04:55

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 24099
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 501
Total_Scans       = 501

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.5[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



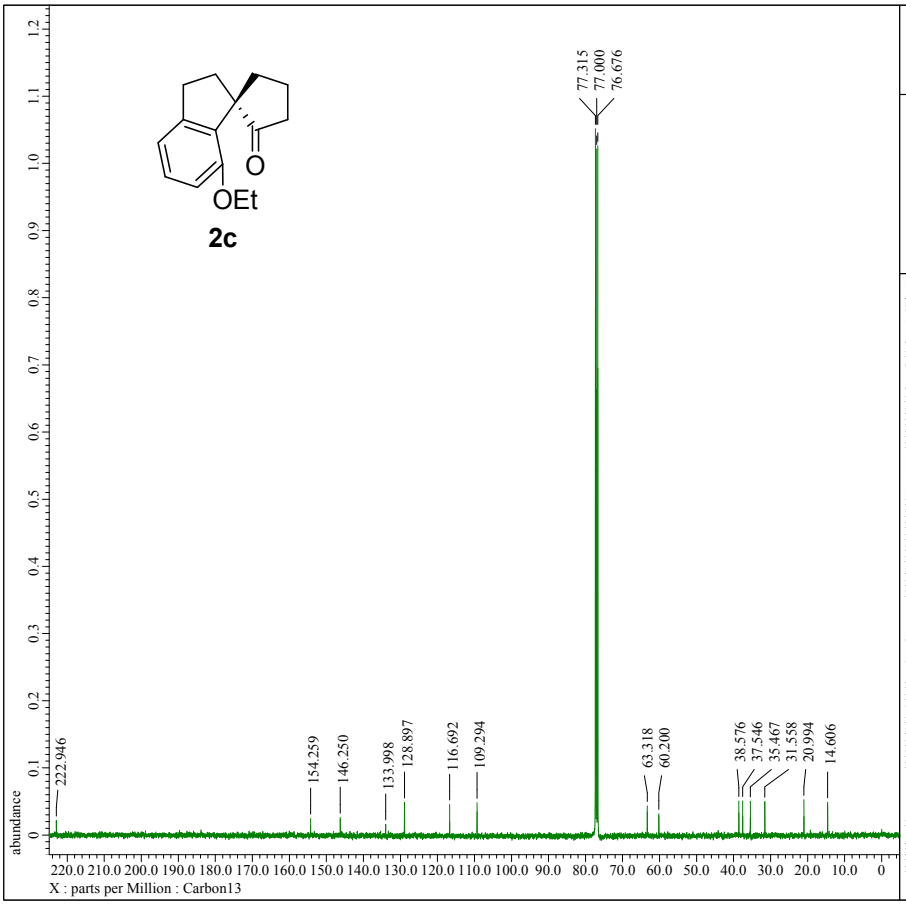

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-214-1-1_Proton-1-7.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-214-1-1
Solvent           = CHLOROFORM-D
Creation_Time     =  3-JUL-2014 20:47:30
Revision_Time     = 24-SEP-2014 18:10:02
Current_Time      = 24-SEP-2014 18:10:19

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay = 5[s]
Recvr_Gain        = 44
Temp_Get          = 21.7[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



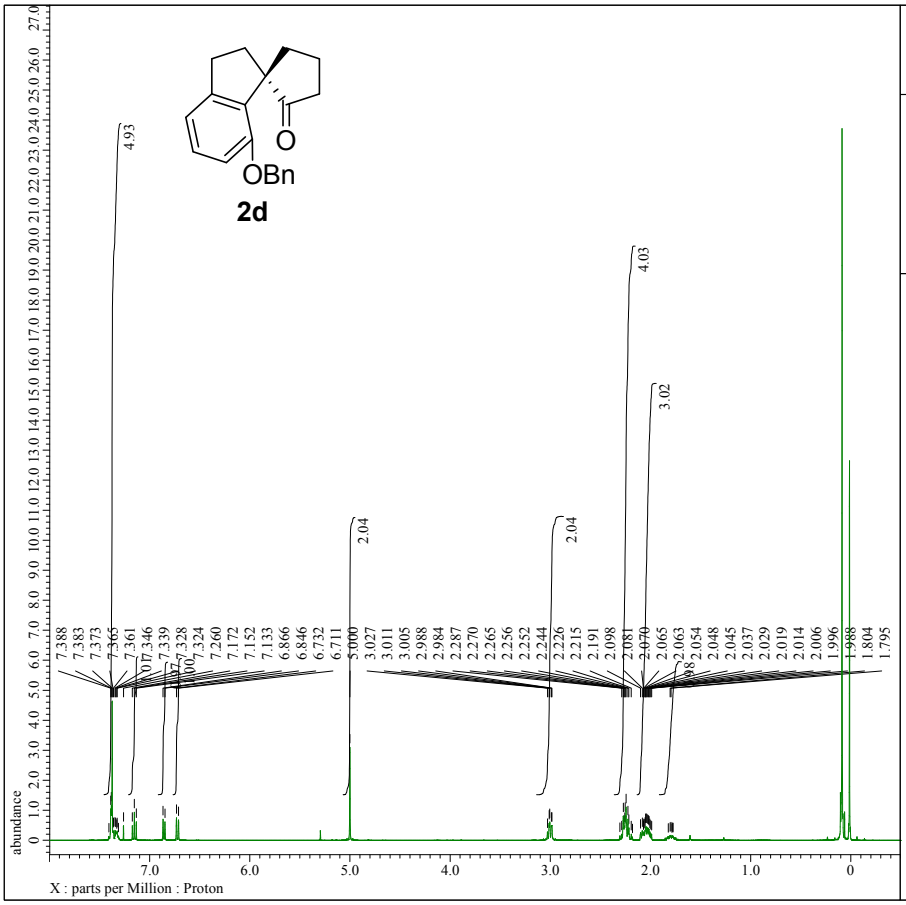

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-214-1-1_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-214-1-1
Solvent           = CHLOROFORM-D
Creation_Time     =  3-JUL-2014 21:19:16
Revision_Time     =  3-JUL-2014 22:12:27
Current_Time      = 24-SEP-2014 18:15:57

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



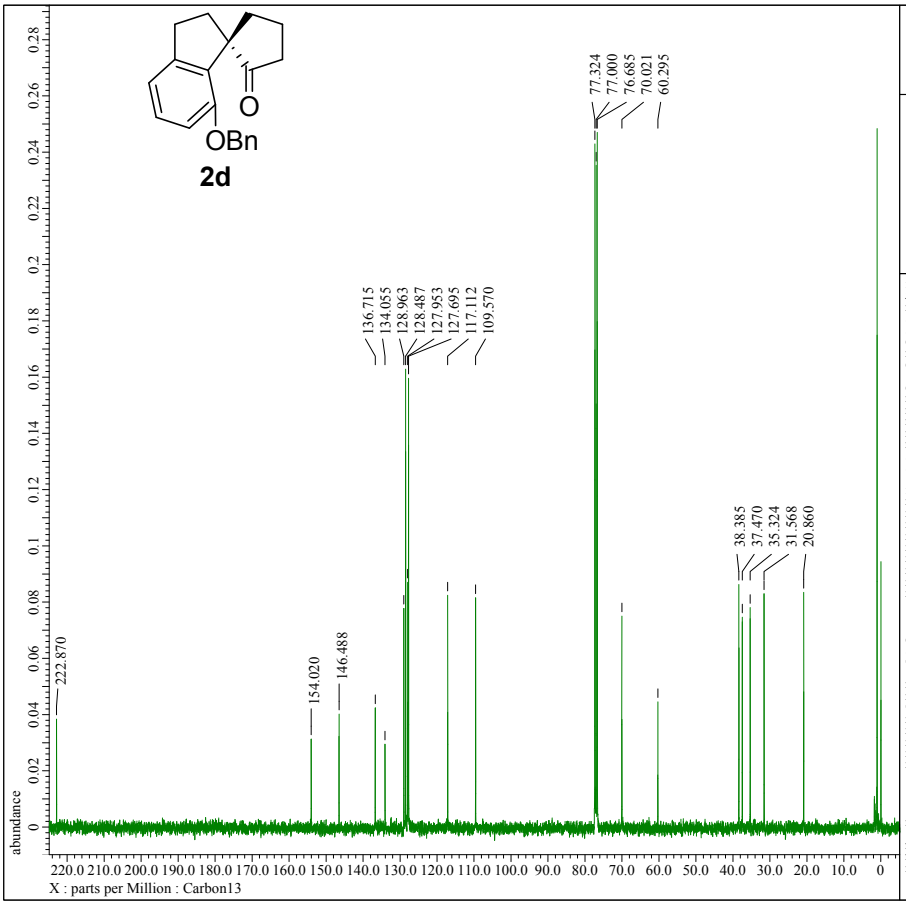

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 420_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 420
Solvent           = CHLOROFORM-D
Creation_Time     =  9-APR-2013 18:39:54
Revision_Time     = 24-SEP-2014 18:22:42
Current_Time      = 24-SEP-2014 18:23:22

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 30
Temp_Get          = 19.7[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



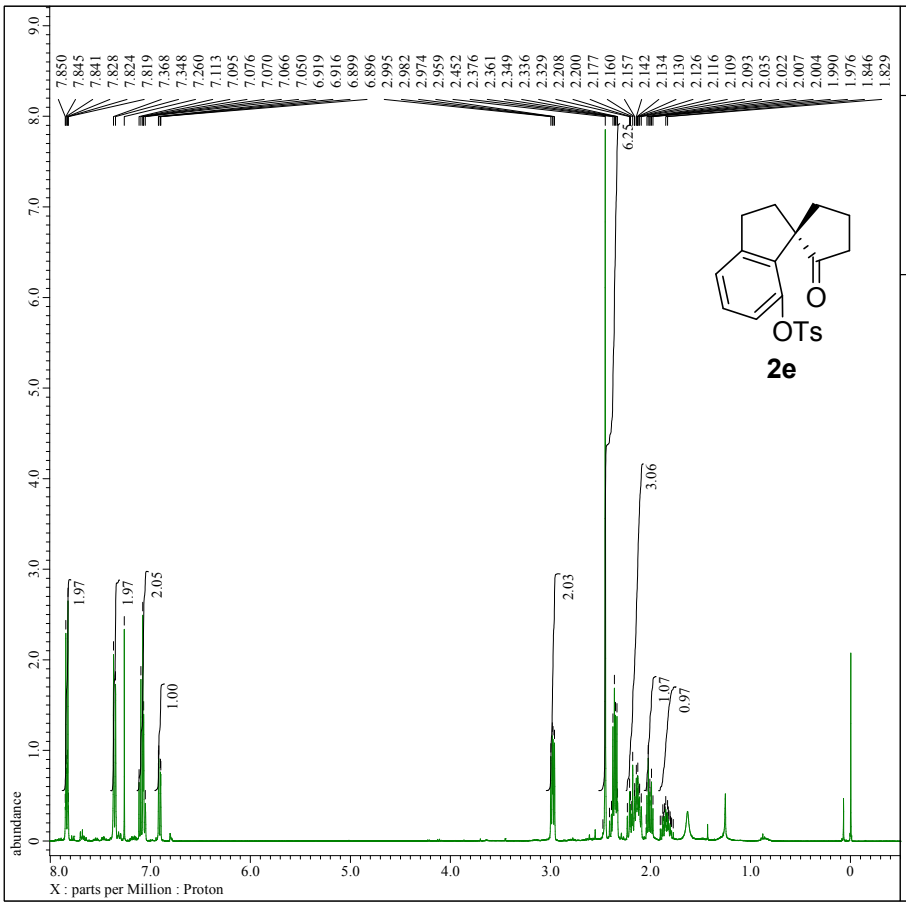

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 420_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 420
Solvent           = CHLOROFORM-D
Creation_Time     =  9-APR-2013 18:43:30
Revision_Time     = 24-SEP-2014 18:27:09
Current_Time      = 24-SEP-2014 18:27:12

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 260
Total_Scans       = 260

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.9[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



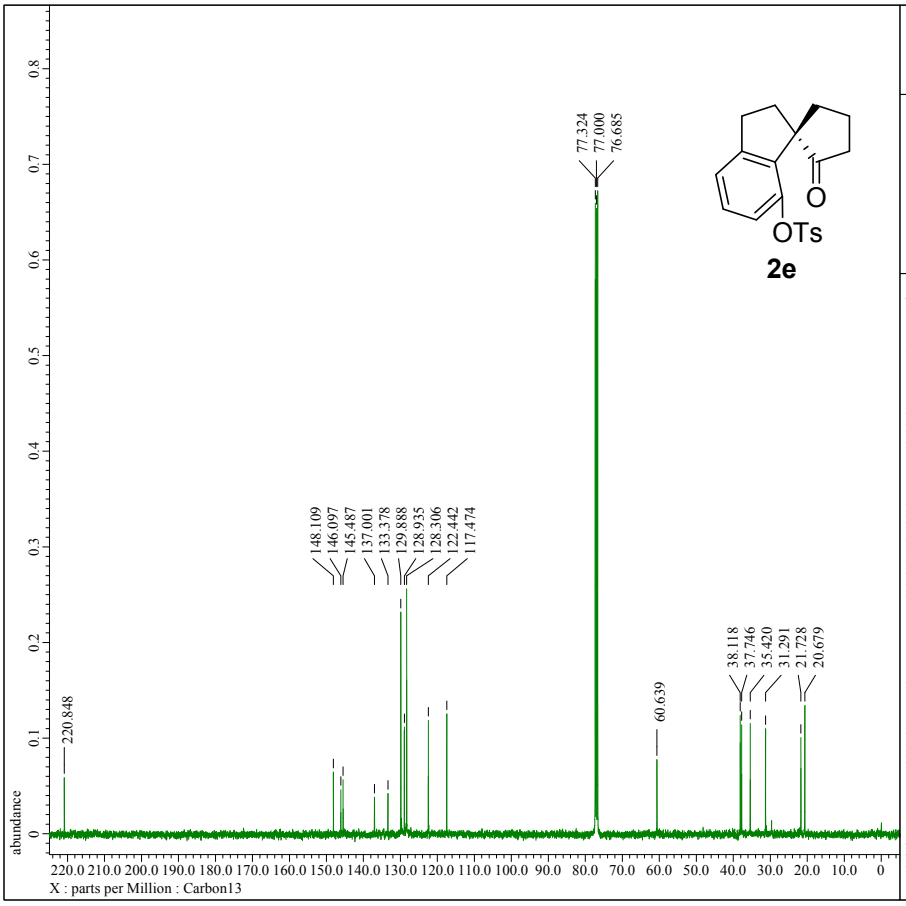

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-235-1_Proton-1-6.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-235-1
Solvent           = CHLOROFORM-D
Creation_Time     =  9-AUG-2014 16:39:37
Revision_Time     = 24-SEP-2014 20:07:58
Current_Time      = 24-SEP-2014 20:08:03

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 21.4[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

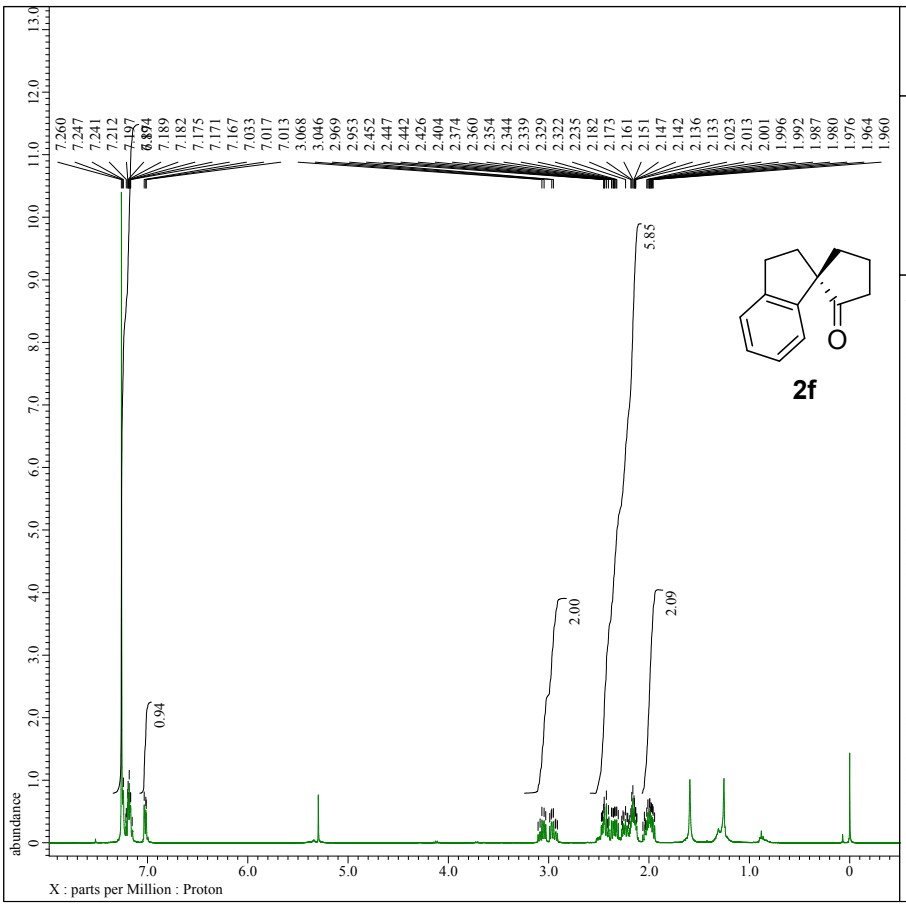
□ □ □ □ □ □: T-235-1_Carbon-1-1.jdf
```

```
Filename          = T-235-1_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-235-1
Solvent           = CHLOROFORM-D
Creation_Time     = 18-SEP-2014 11:14:13
Revision_Time     = 24-SEP-2014 20:04:48
Current_Time      = 24-SEP-2014 20:05:05

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 19.7[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21[dB]
Irr_Atn_Noe       = 21[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



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```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

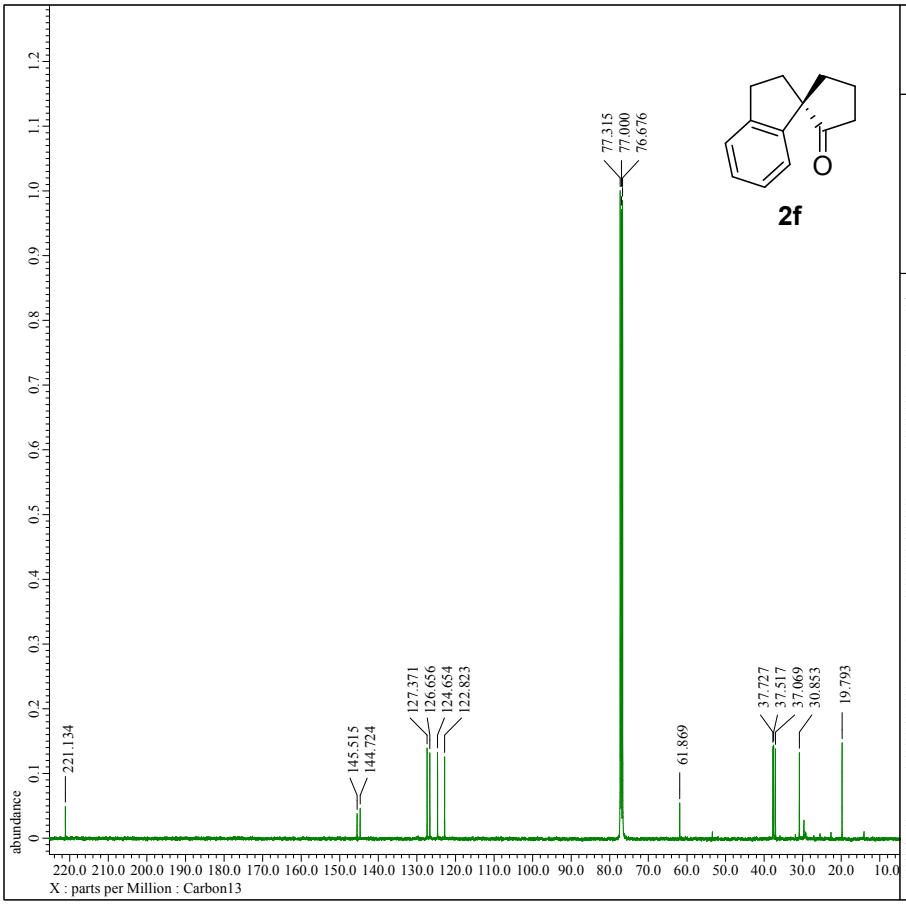
□ □ □ □ □ □: T-120_Proton-2-1.jdf
```

```
Filename          = T-120_Proton-2-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-120
Solvent           = CHLOROFORM-D
Creation_Time     = 26-SEP-2014 23:54:25
Revision_Time     = 27-SEP-2014 10:48:31
Current_Time      = 27-SEP-2014 10:48:37

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 460.0[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



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```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

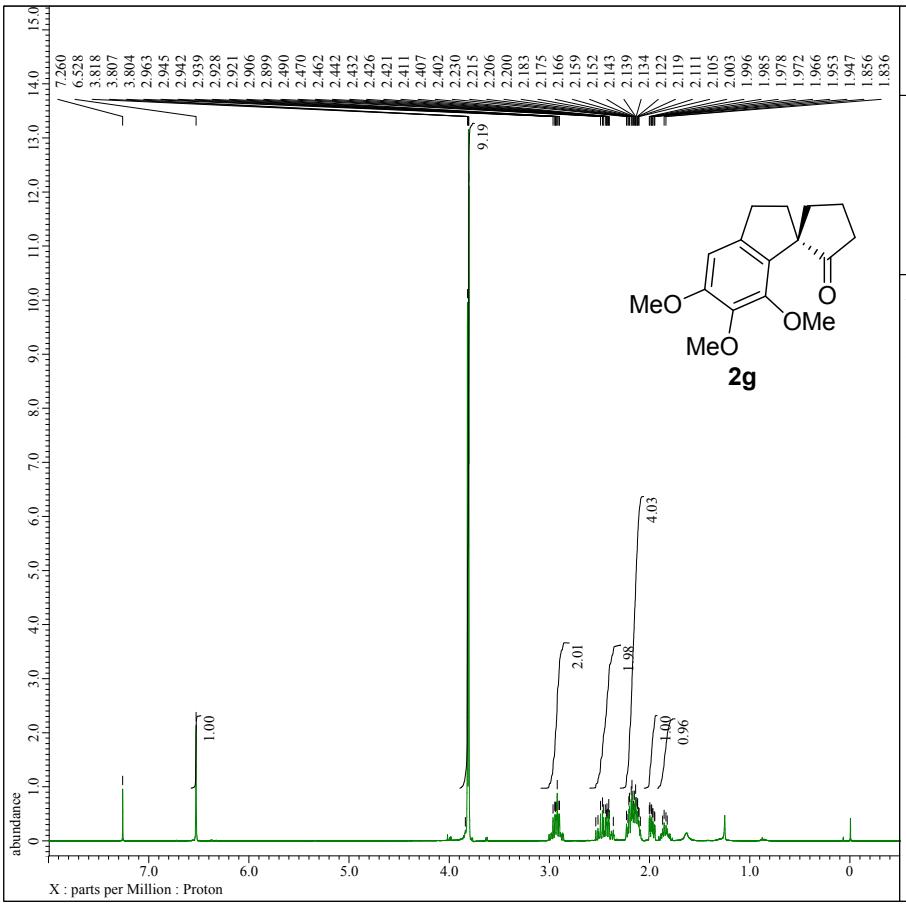
□ □ □ □ □ □: T-120_Carbon-2-1.jdf
```

```
Filename          = T-120_Carbon-2-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-120
Solvent           = CHLOROFORM-D
Creation_Time     = 26-SEP-2014 23:55:57
Revision_Time     = 27-SEP-2014 10:42:02
Current_Time      = 27-SEP-2014 10:42:33

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 110[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 4096
Total_Scans       = 4096

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 460.0[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



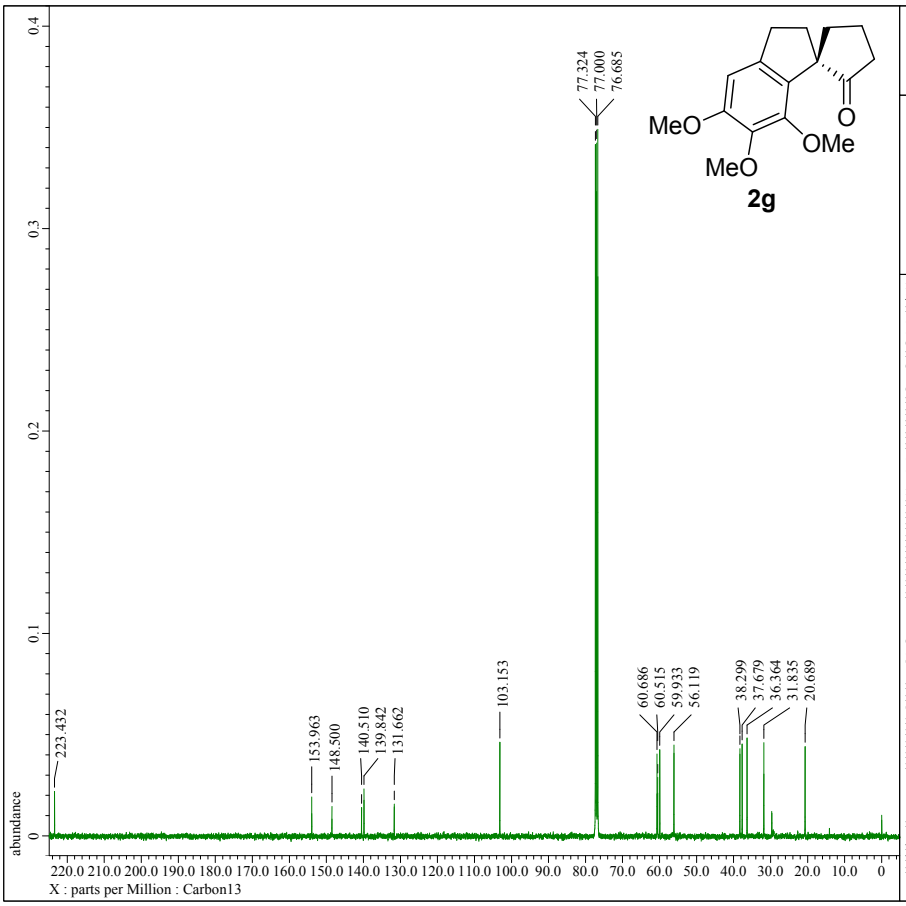

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-196-1_Proton-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-196-1
Solvent           = CHLOROFORM-D
Creation_Time     =  2-JUN-2014 10:55:23
Revision_Time     = 25-SEP-2014 10:40:56
Current_Time      = 25-SEP-2014 10:41:16

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 23.6[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



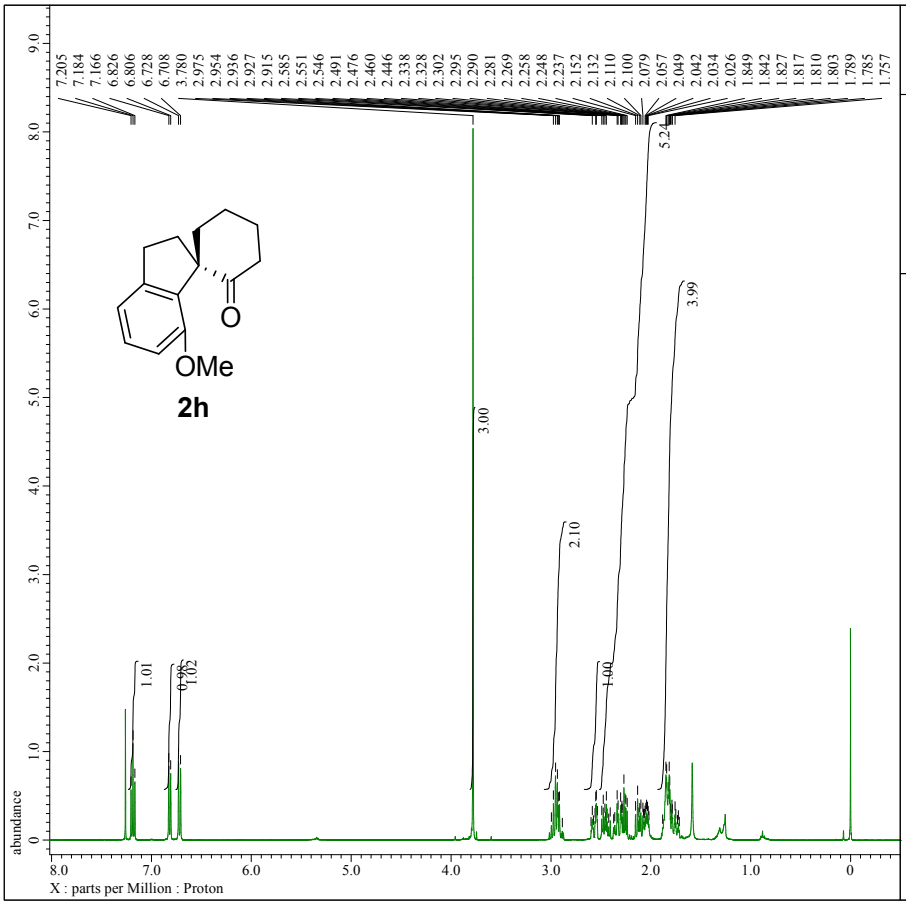

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-192-0-0_Carbon-1-6.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-192-0-0
Solvent           = CHLOROFORM-D
Creation_Time     = 24-SEP-2014 23:22:03
Revision_Time     = 25-SEP-2014 10:24:18
Current_Time      = 25-SEP-2014 10:24:53

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 460.0[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



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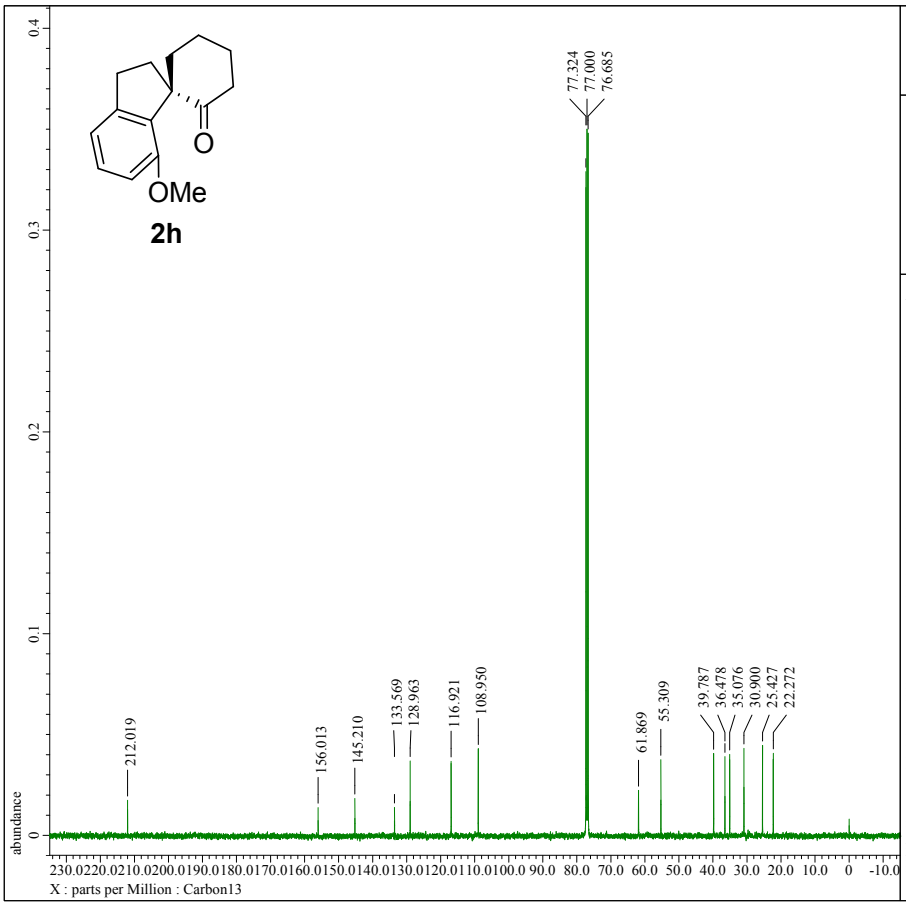
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-419-0_Proton-1-6.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-419-0
Solvent           = CHLOROFORM-D
Creation_Time     = 27-SEP-2014 15:25:33
Revision_Time     = 27-SEP-2014 16:12:37
Current_Time      = 27-SEP-2014 16:12:46

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 40
Temp_Get          = 460.0[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



JEOL Solutions for Innovation

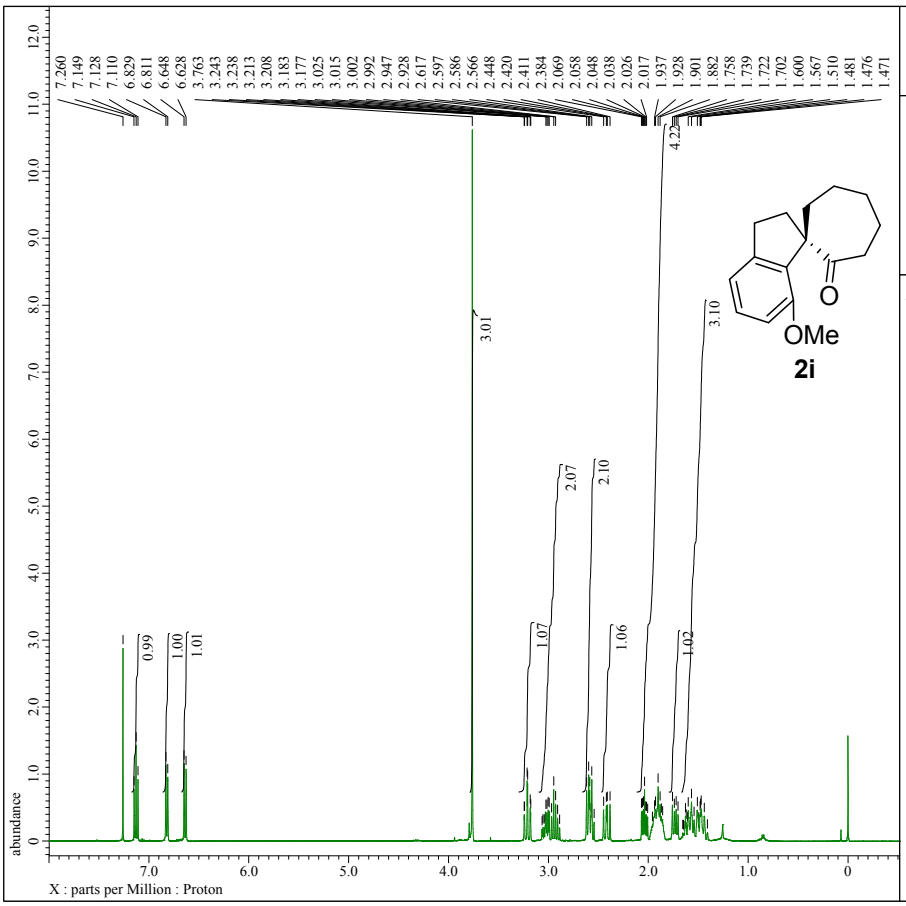
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-419-0_Carbon-1-7.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-419-0
Solvent           = CHLOROFORM-D
Creation_Time     = 27-SEP-2014 15:26:45
Revision_Time     = 27-SEP-2014 16:06:54
Current_Time      = 27-SEP-2014 16:14:43

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 110[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 460.0[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



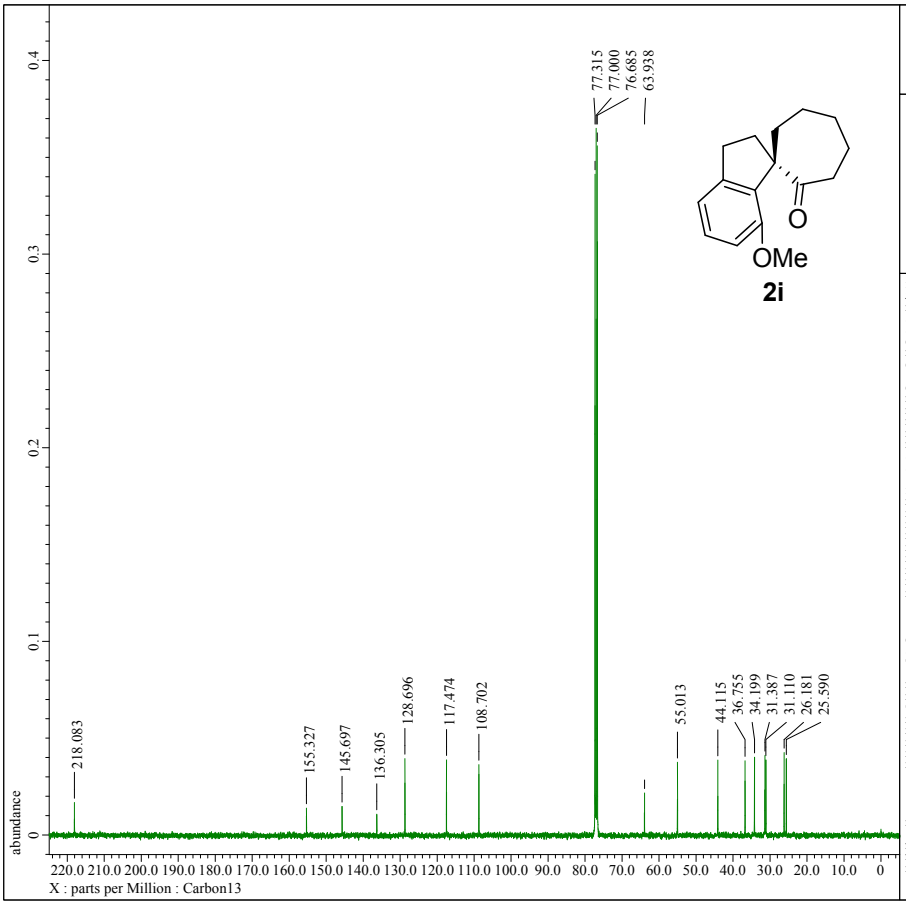

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 632_Proton-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 632
Solvent           = CHLOROFORM-D
Creation_Time     = 23-JAN-2014 22:38:59
Revision_Time     = 24-SEP-2014 23:27:34
Current_Time      = 24-SEP-2014 23:27:46

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 44
Temp_Get          = 21.7[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



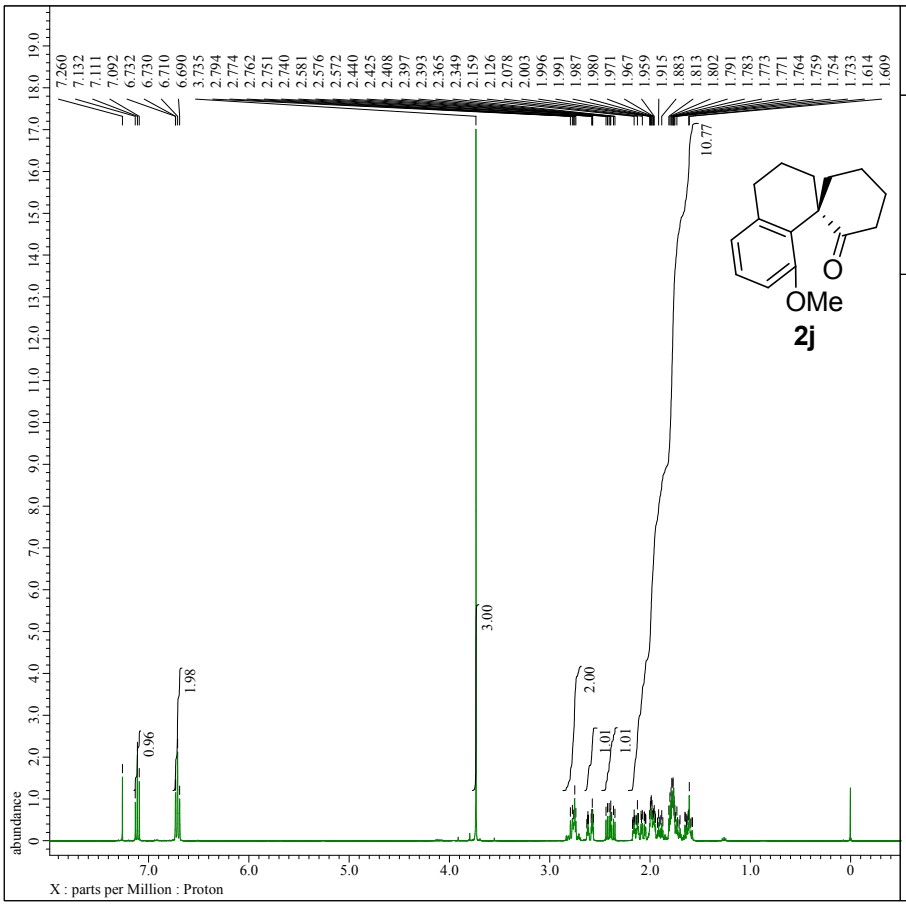

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 632_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 632
Solvent           = CHLOROFORM-D
Creation_Time     = 23-JAN-2014 22:43:16
Revision_Time     = 24-SEP-2014 23:25:21
Current_Time      = 24-SEP-2014 23:25:39

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.8[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



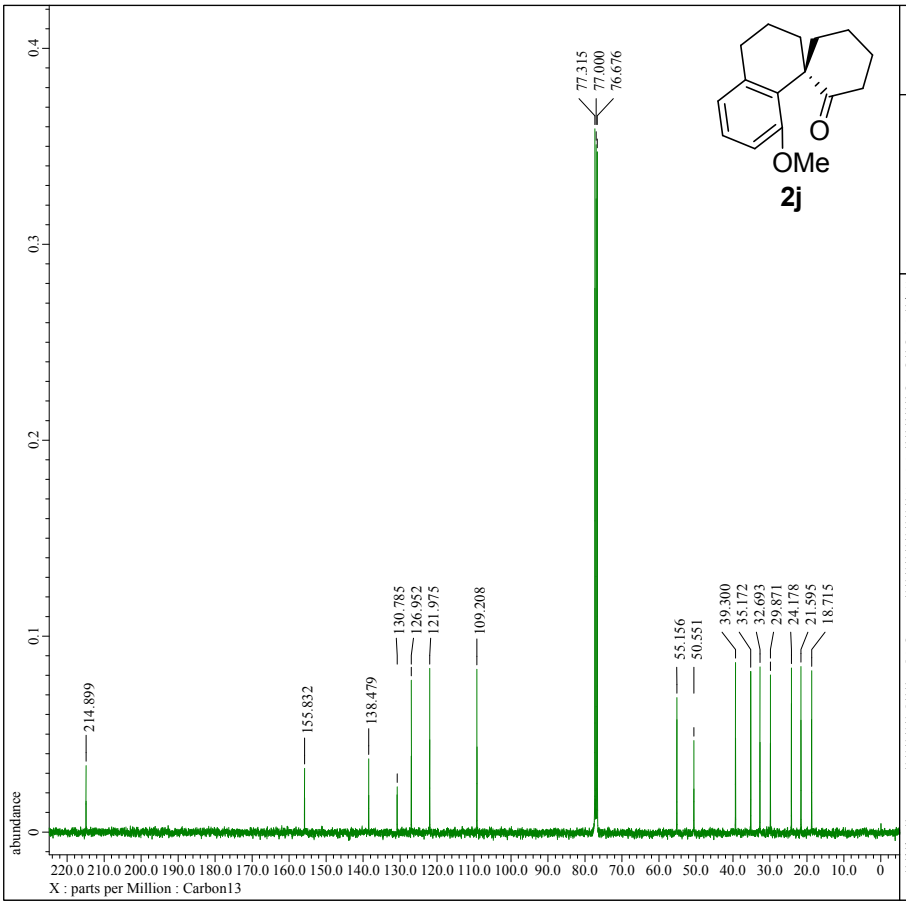

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 566-pure_Proton-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 566-pure
Solvent           = CHLOROFORM-D
Creation_Time     =  6-NOV-2013 20:24:52
Revision_Time     = 24-SEP-2014 23:35:01
Current_Time      = 24-SEP-2014 23:35:13

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 32.7[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



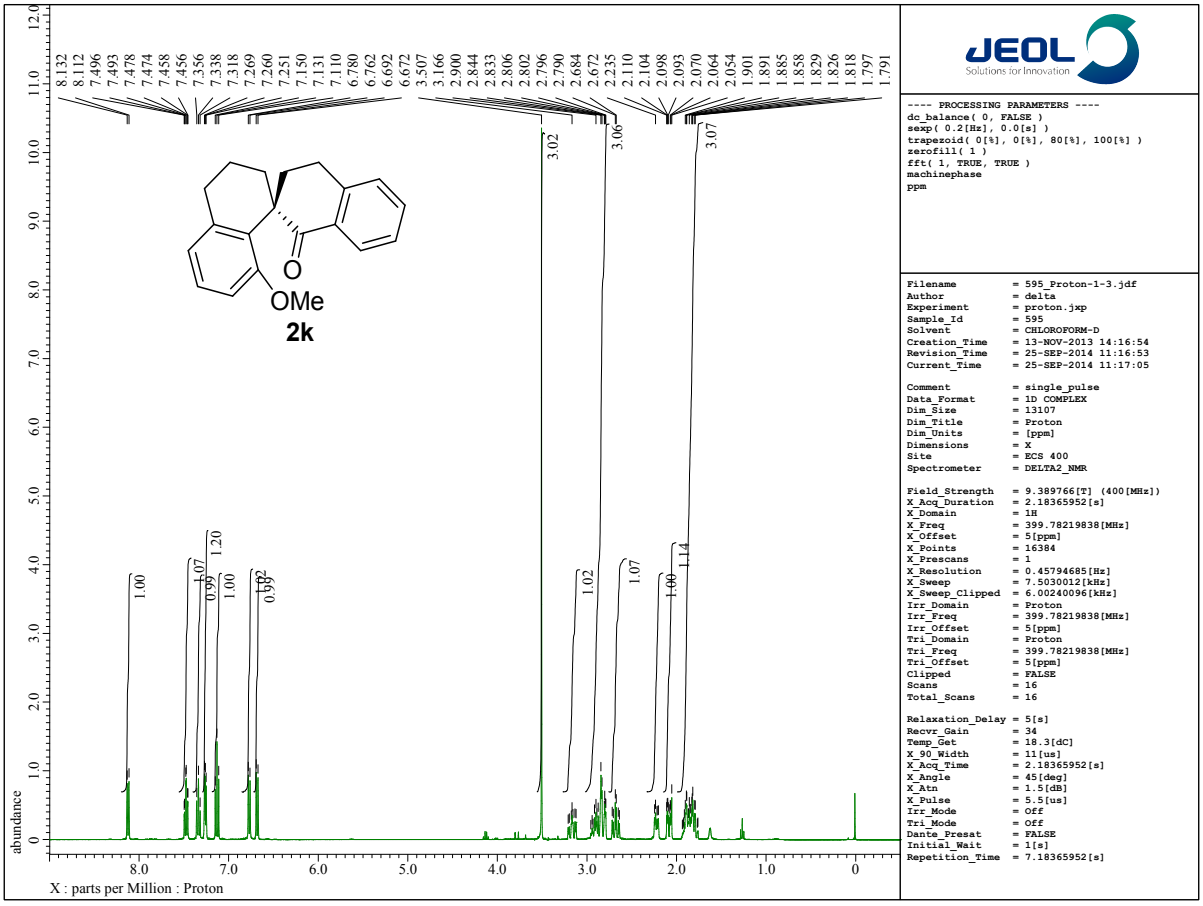

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
traperoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 566-pure_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 566-pure
Solvent           = CHLOROFORM-D
Creation_Time     =  6-NOV-2013 20:27:55
Revision_Time     = 24-SEP-2014 23:36:03
Current_Time      = 24-SEP-2014 23:36:19

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 415
Total_Scans       = 415

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



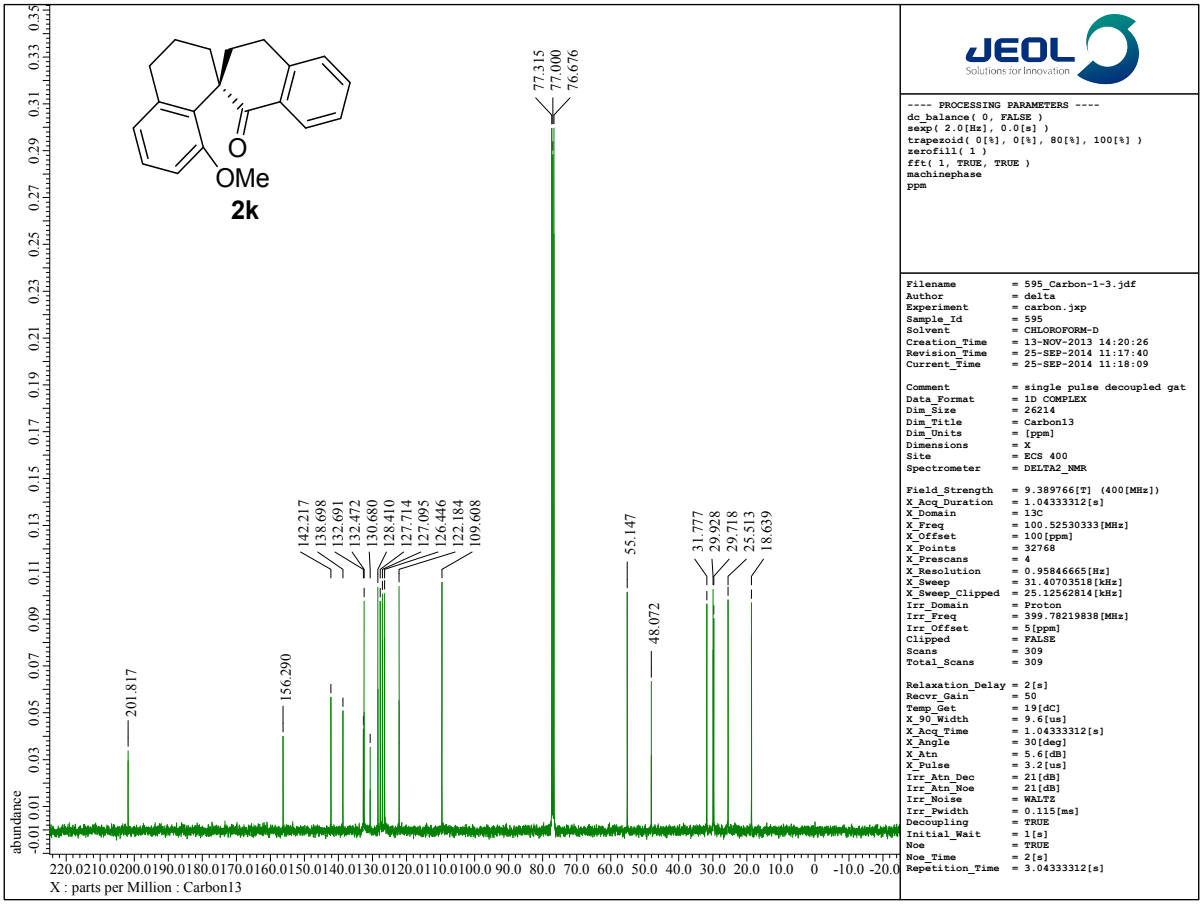

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 595_Proton-1-3.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 595
Solvent           = CHLOROFORM-D
Creation_Time     = 13-NOV-2013 14:16:54
Revision_Time     = 25-SEP-2014 11:16:53
Current_Time      = 25-SEP-2014 11:17:05

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 34
Temp_Get          = 18.3[dC]
X_90_Width        = 11[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 1.5[dB]
X_Pulse           = 5.5[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



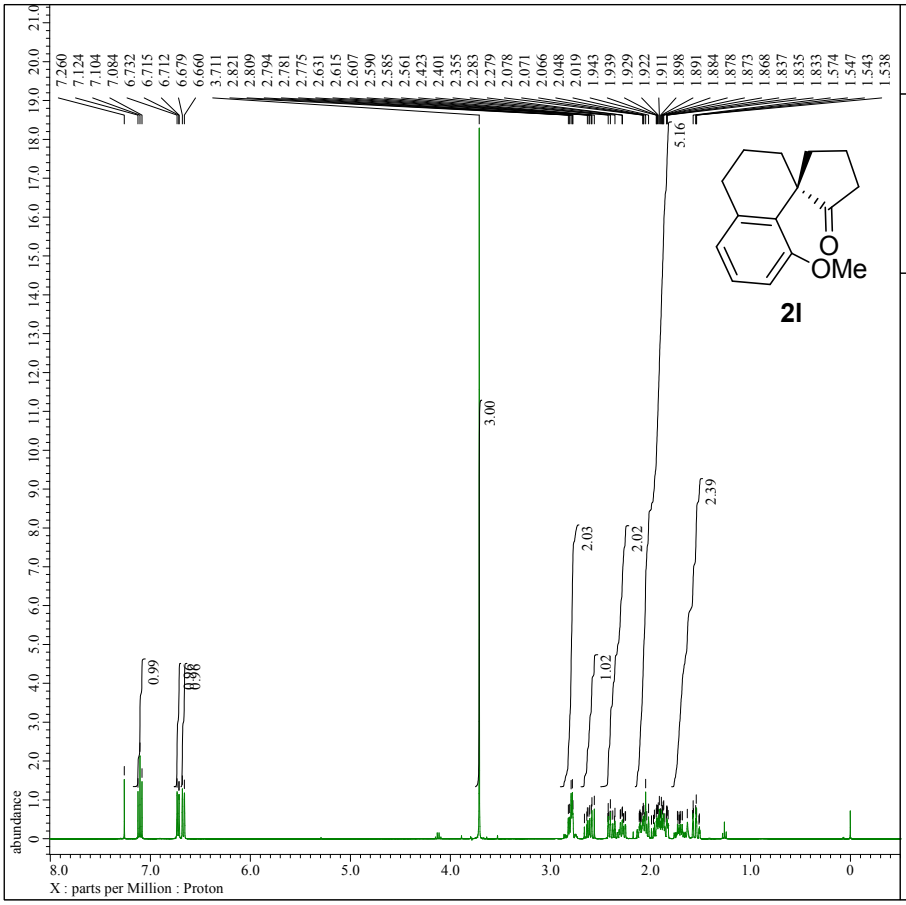

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = 595_Carbon-1-3.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 595
Solvent           = CHLOROFORM-D
Creation_Time     = 13-NOV-2013 14:20:26
Revision_Time     = 25-SEP-2014 11:17:40
Current_Time      = 25-SEP-2014 11:18:09

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

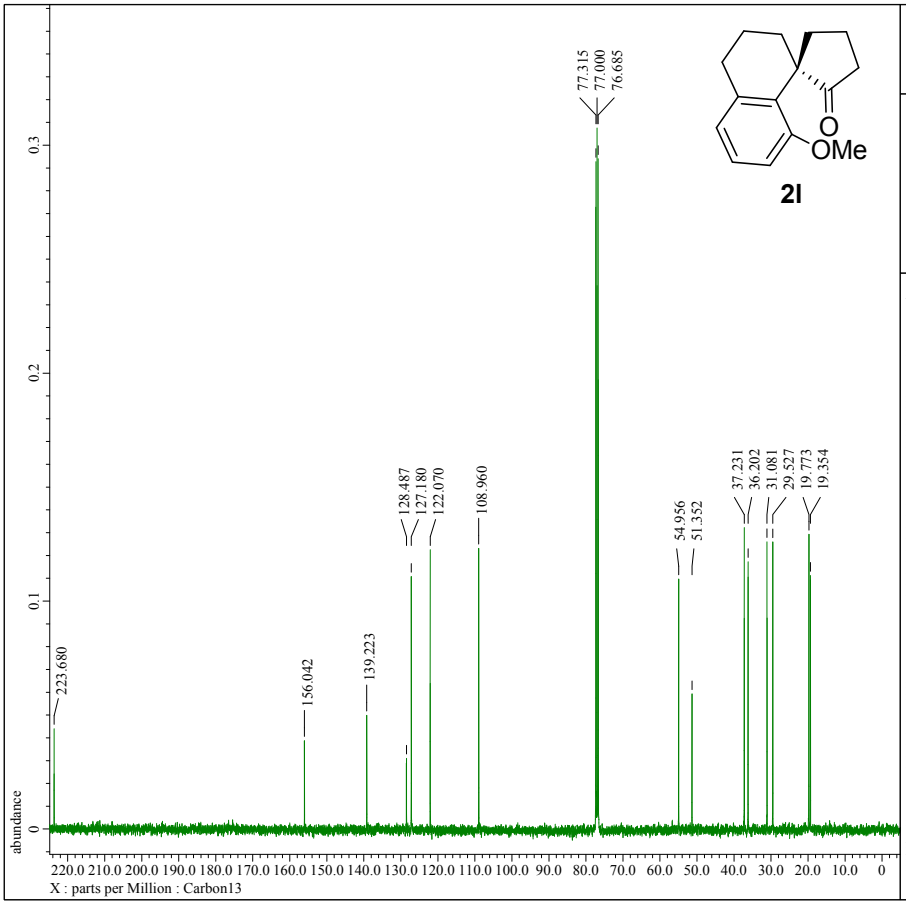
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.04333312[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95846665[Hz]
X_Sweep           = 31.40703518[kHz]
X_Sweep_Clipped   = 25.12562814[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 309
Total_Scans       = 309

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 5.6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21[dB]
Irr_Atn_Noe       = 21[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



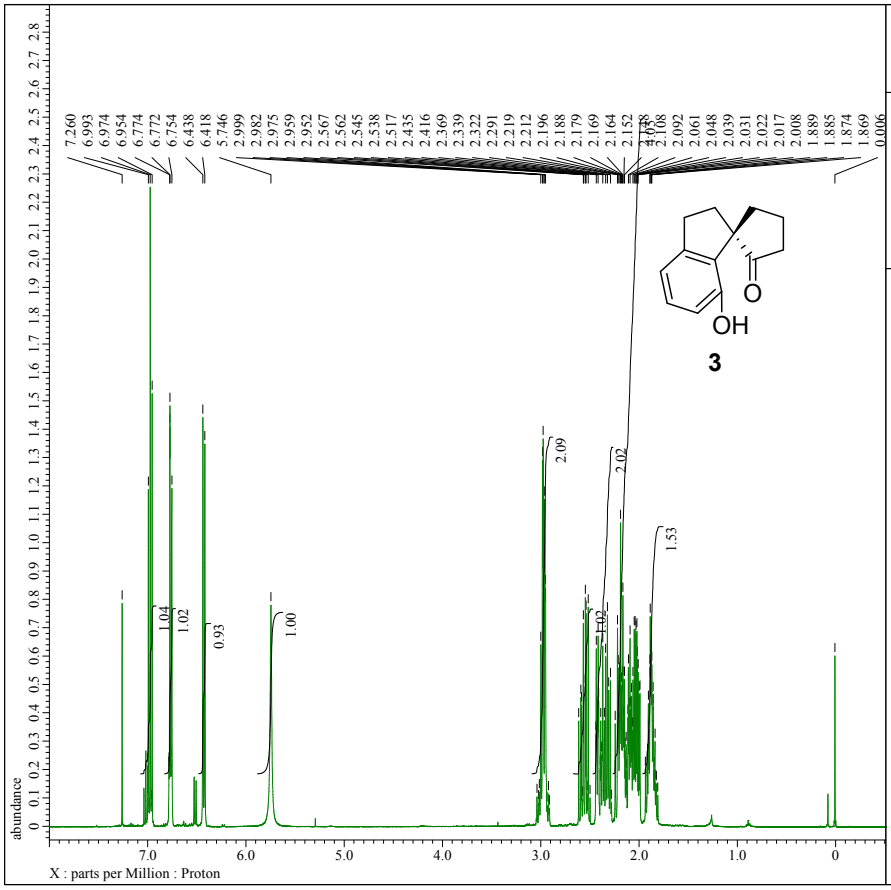
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
traperoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

Filename	= 583-pure_Proton-1-3.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= 583-pure
Solvent	= CHLOROFORM-D
Creation_Time	= 9-NOV-2013 16:35:34
Revision_Time	= 25-SEP-2014 11:26:01
Current_Time	= 25-SEP-2014 11:26:14
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 5[s]
Recvr_Gain	= 34
Temp_Get	= 19.5[dC]
X_90_Width	= 11[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 1.5[dB]
X_Pulse	= 5.5[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]



---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
traperoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

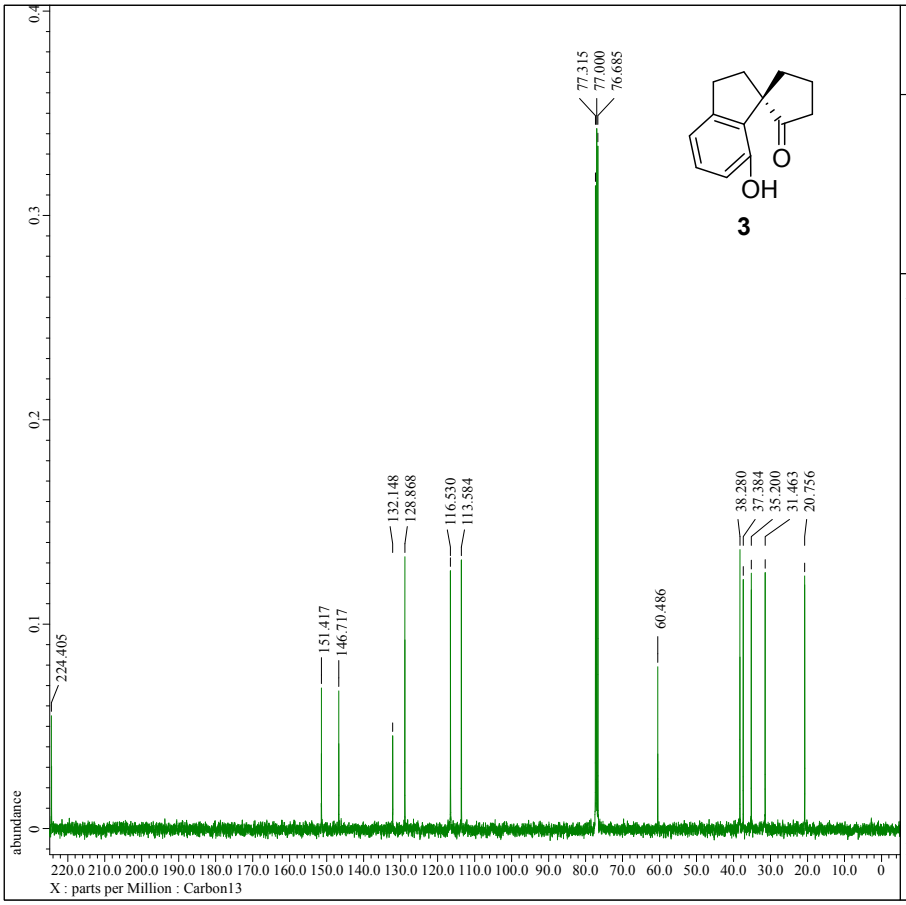
Filename	= 583-pure_Carbon-1-3.jdf
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= 583-pure
Solvent	= CHLOROFORM-D
Creation_Time	= 9-NOV-2013 16:38:32
Revision_Time	= 25-SEP-2014 11:27:03
Current_Time	= 25-SEP-2014 11:27:45
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 305
Total_Scans	= 305
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 19.9[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 5.6[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 21[dB]
Irr_Atn_Noe	= 21[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

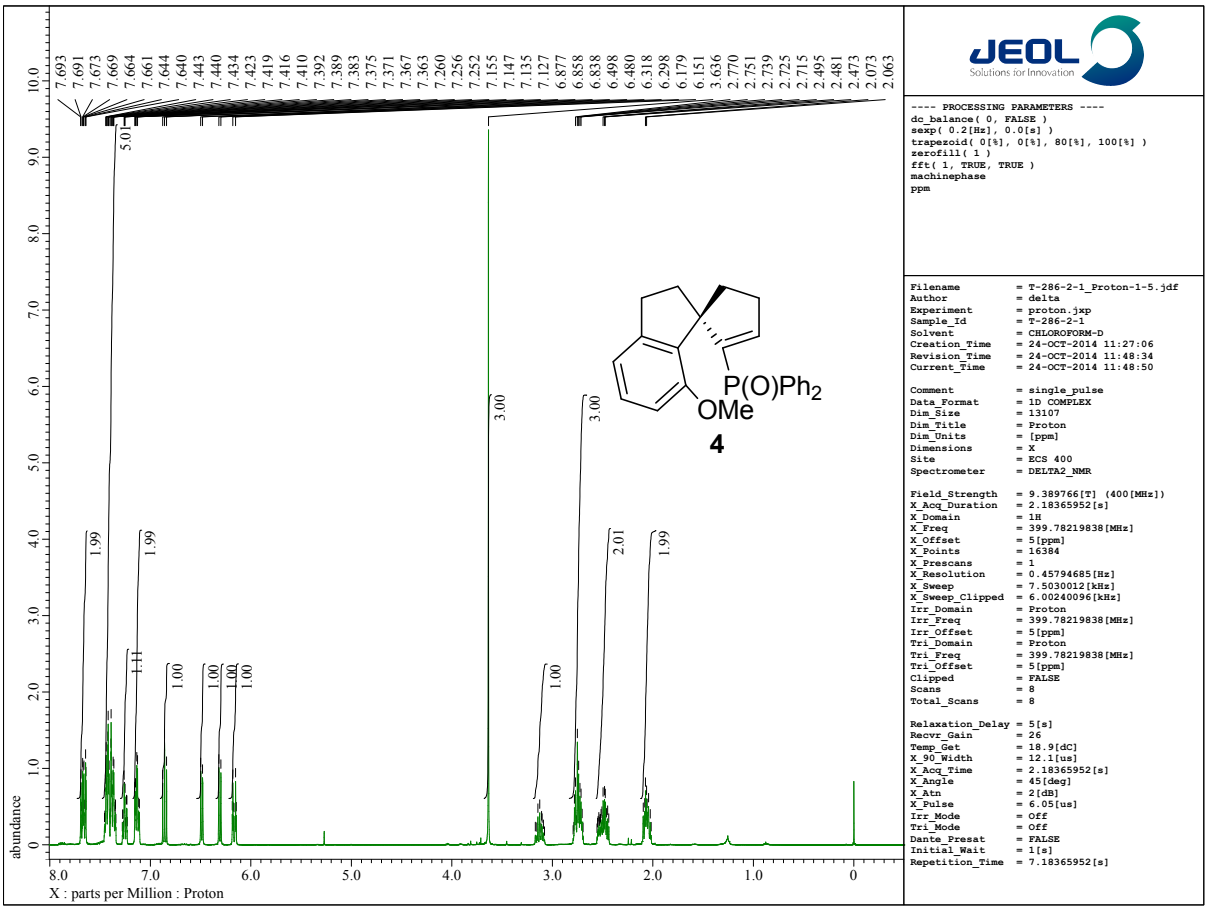
□ □ □□ □ □: 530_Proton-1-1.jdf
```

Filename	= 530_Proton-1-5.jdf
Author	= delta
Experiment	= proton.jxp
Sample_Id	= 530
Solvent	= CHLOROFORM-D
Creation_Time	= 13-SEP-2013 12:55:57
Revision_Time	= 15-NOV-2014 17:35:53
Current_Time	= 15-NOV-2014 17:36:14
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 2.18365952[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45794685[Hz]
X_Sweep	= 7.5030012[kHz]
X_Sweep_Clipped	= 6.00240096[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 5[s]
Recvr_Gain	= 38
Temp_Get	= 26.2[dC]
X_90_Width	= 12.1[us]
X_Acq_Time	= 2.18365952[s]
X_Angle	= 45[deg]
X_Atn	= 2[dB]
X_Pulse	= 6.05[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18365952[s]




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

Filename	= 530_Carbon-1-3.jdf
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= 530
Solvent	= CHLOROFORM-D
Creation_Time	= 13-SEP-2013 12:59:12
Revision_Time	= 15-NOV-2014 17:37:31
Current_Time	= 15-NOV-2014 17:37:44
Comment	= single pulse decoupled gat
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.389766[T] (400[MHz])
X_Acq_Duration	= 1.04333312[s]
X_Domain	= 13C
X_Freq	= 100.52530333[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.95846665[Hz]
X_Sweep	= 31.40703518[kHz]
X_Sweep_Clipped	= 25.12562814[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 174
Total_Scans	= 174
Relaxation_Delay	= 2[s]
Recvr_Gain	= 50
Temp_Get	= 26.1[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 1.04333312[s]
X_Angle	= 30[deg]
X_Atn	= 6[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 21.63[dB]
Irr_Atn_Noe	= 21.63[dB]
Irr_Noise	= WALTZ
Irr_Pwidth	= 0.115[ms]
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.04333312[s]



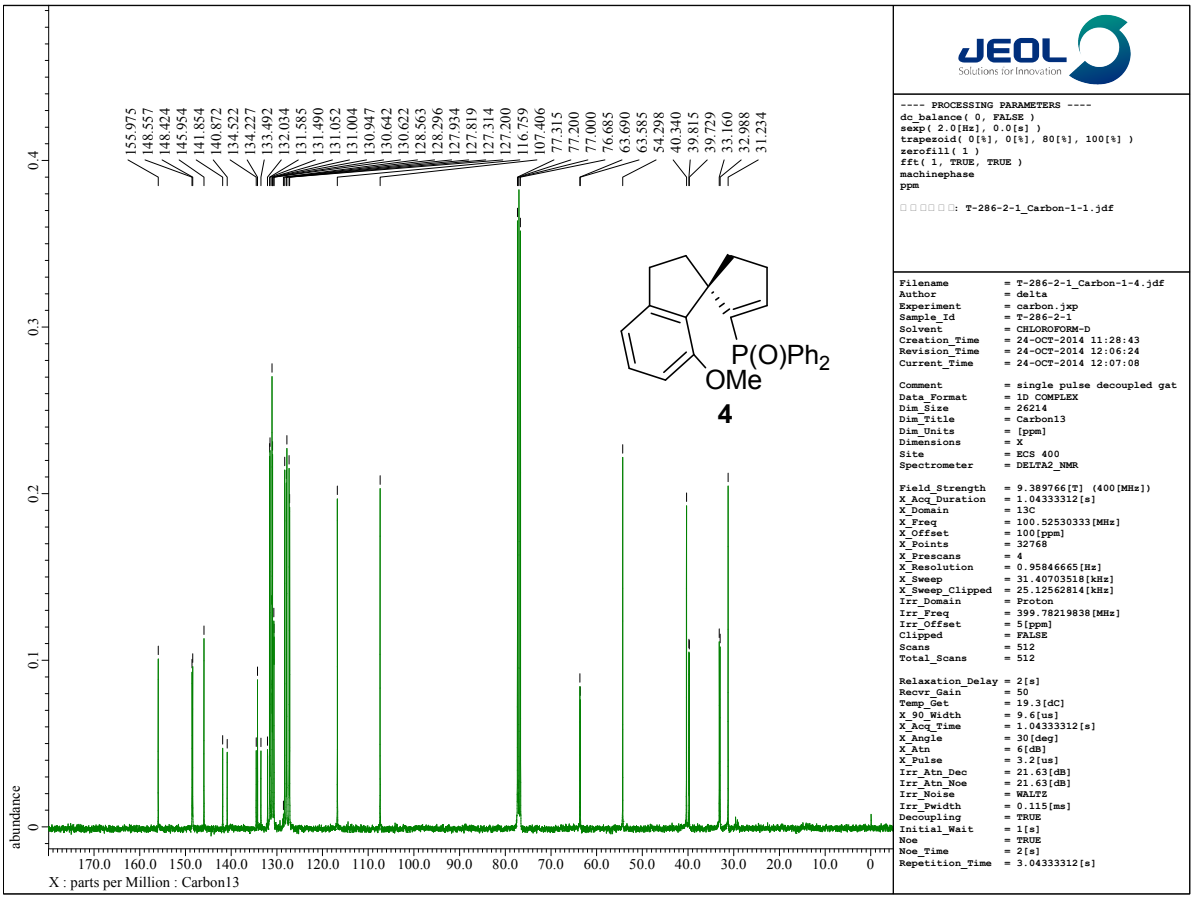

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-286-2-1_Proton-1-5.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-286-2-1
Solvent           = CHLOROFORM-D
Creation_Time     = 24-OCT-2014 11:27:06
Revision_Time     = 24-OCT-2014 11:48:34
Current_Time      = 24-OCT-2014 11:48:50

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 26
Temp_Get          = 18.9[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

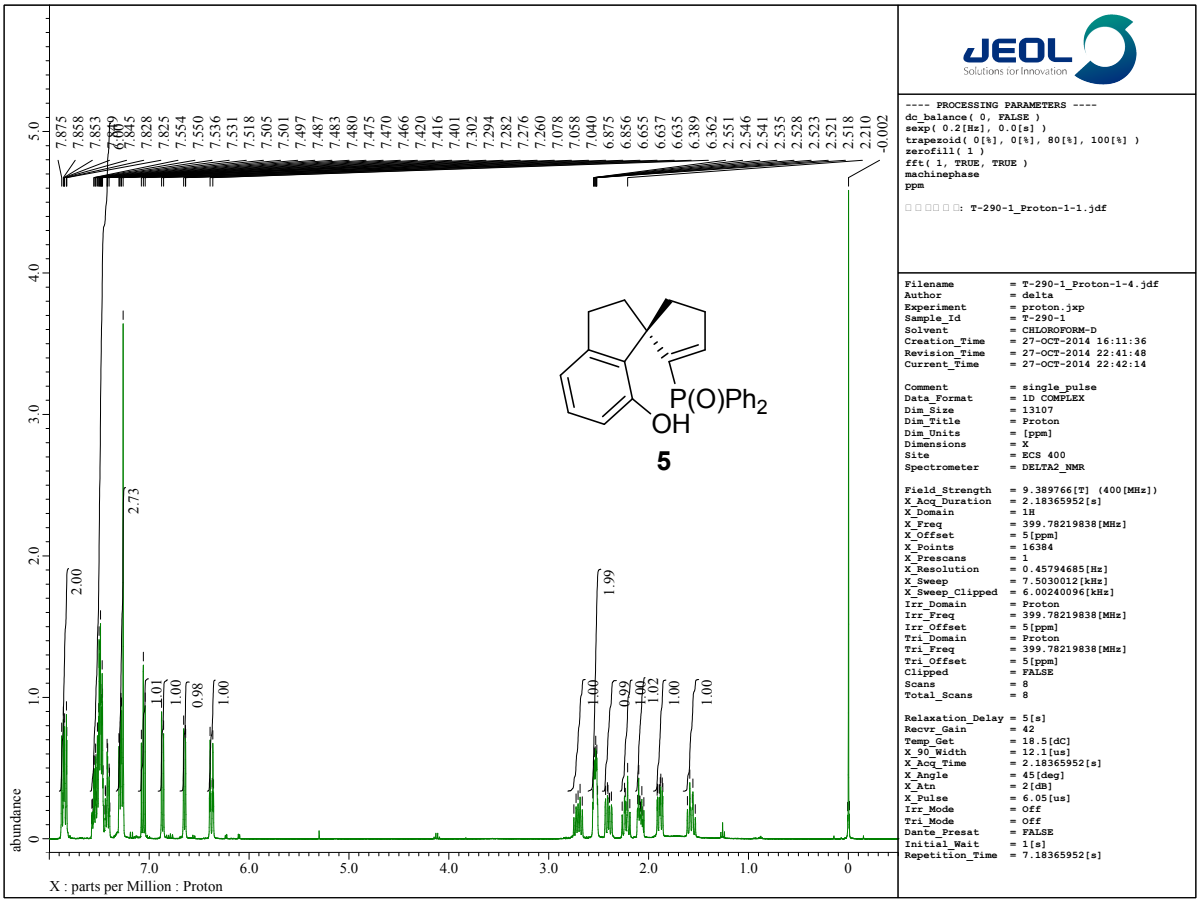
□ □ □□ □ □: T-286-2-1_Carbon-1-1.jdf
```

```
Filename          = T-286-2-1_Carbon-1-4.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-286-2-1
Solvent           = CHLOROFORM-D
Creation_Time     = 24-OCT-2014 11:28:43
Revision_Time     = 24-OCT-2014 12:06:24
Current_Time      = 24-OCT-2014 12:07:08

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 19.3[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```




```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

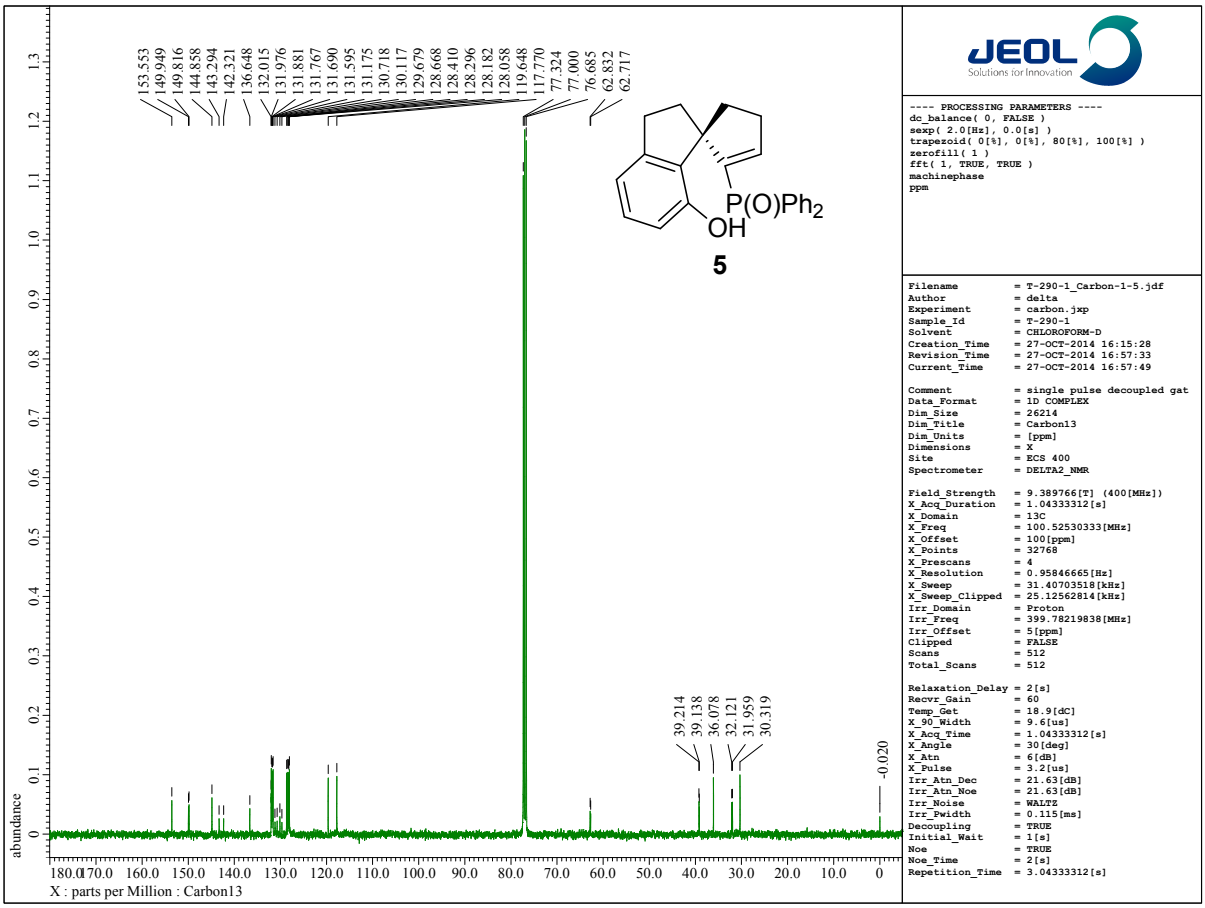
□ □ □□ □ □: T-290-1_Proton-1-1.jdf
```

```
Filename          = T-290-1_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = T-290-1
Solvent           = CHLOROFORM-D
Creation_Time     = 27-OCT-2014 16:11:36
Revision_Time     = 27-OCT-2014 22:41:48
Current_Time      = 27-OCT-2014 22:42:14

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 18.5[dC]
X_90_Width        = 12.1[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 2[dB]
X_Pulse           = 6.05[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



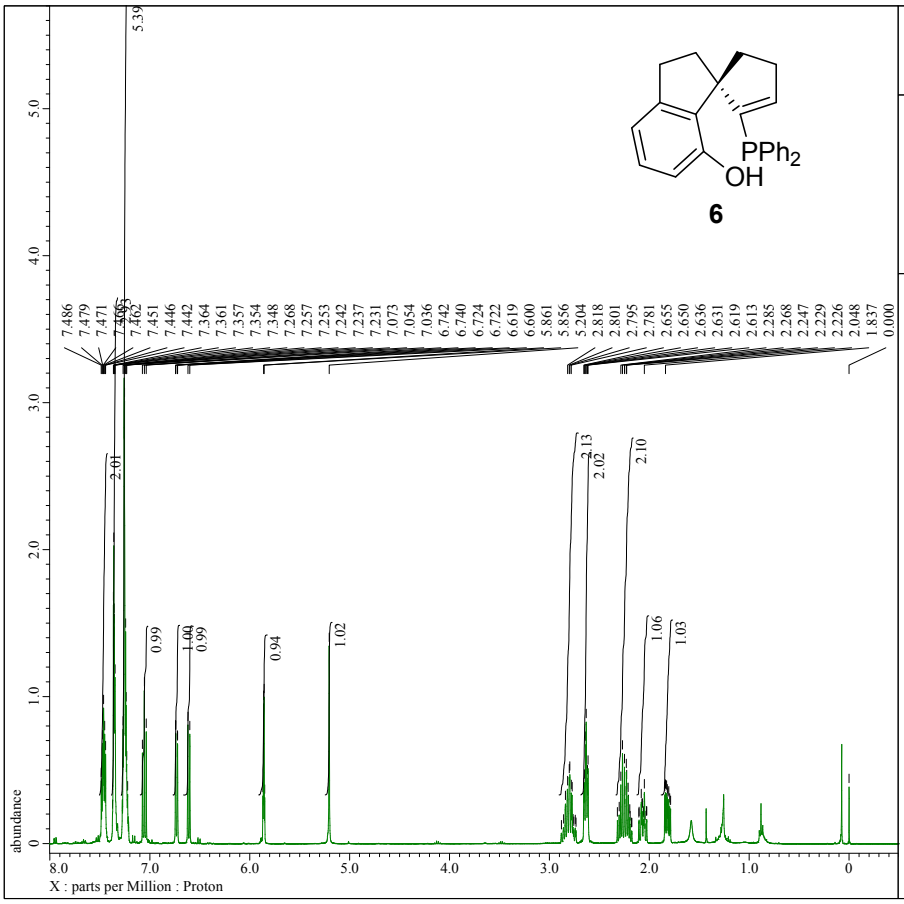

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
```

```
Filename          = T-290-1_Carbon-1-5.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = T-290-1
Solvent           = CHLOROFORM-D
Creation_Time     = 27-OCT-2014 16:15:28
Revision_Time     = 27-OCT-2014 16:57:33
Current_Time      = 27-OCT-2014 16:57:49

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = DELTA2_NMR

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

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 18.9[dC]
X_90_Width        = 9.6[us]
X_Acq_Time        = 1.04333312[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.2[us]
Irr_Atn_Dec       = 21.63[dB]
Irr_Atn_Noe       = 21.63[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.04333312[s]
```



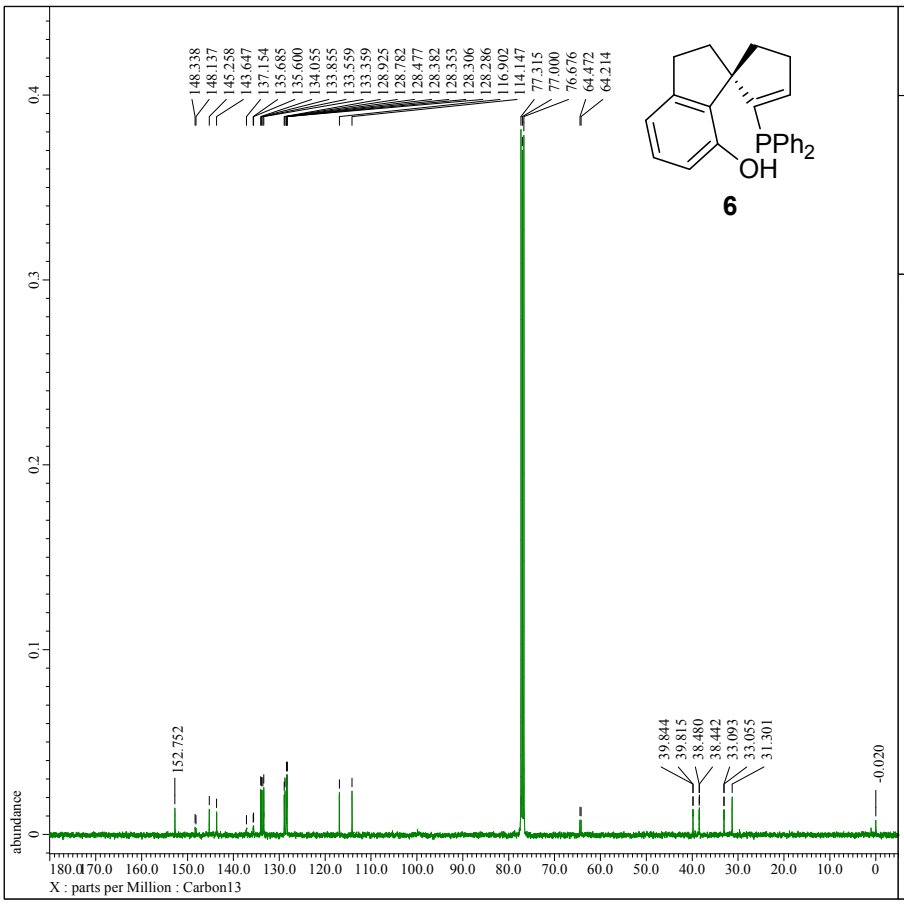

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = T-204-1_Proton-1-5.jdf
Author              = delta
Experiment          = proton.jxp
Sample_Id           = T-204-1
Solvent             = CHLOROFORM-D
Creation_Time       = 21-JUN-2014 18:49:59
Revision_Time       =  4-NOV-2014 10:57:29
Current_Time        =  4-NOV-2014 10:57:42

Comment             = single_pulse
Data_Format         = 1D COMPLEX
Dim_Size            = 13107
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

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

Relaxation_Delay    = 5[s]
Recvr_Gain          = 30
Temp_Get            = 21.9[dC]
X_90_Width          = 12.1[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 2[dB]
X_Pulse             = 6.05[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
```



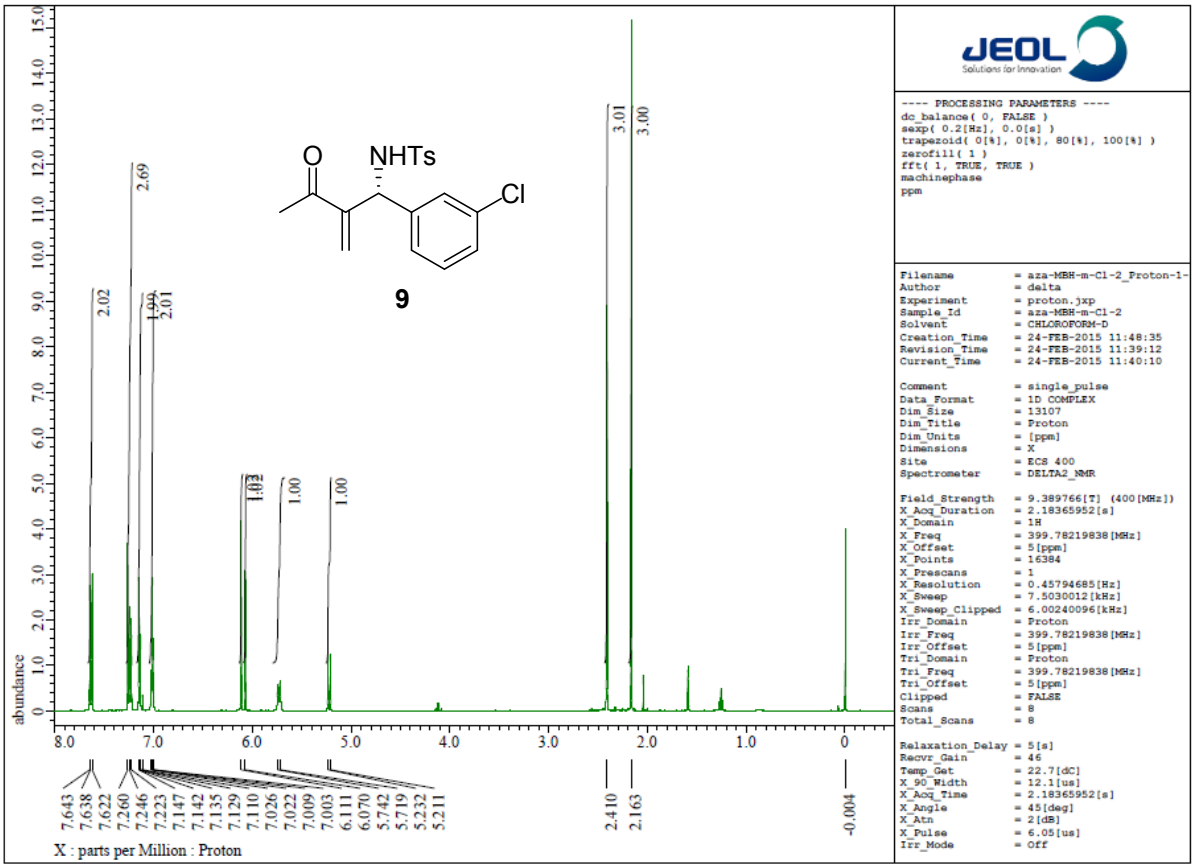

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Filename            = T-204-1_Carbon-1-6.jdf
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = T-204-1
Solvent             = CHLOROFORM-D
Creation_Time       =  4-NOV-2014 14:23:35
Revision_Time       =  4-NOV-2014 15:18:22
Current_Time        =  4-NOV-2014 15:20:45

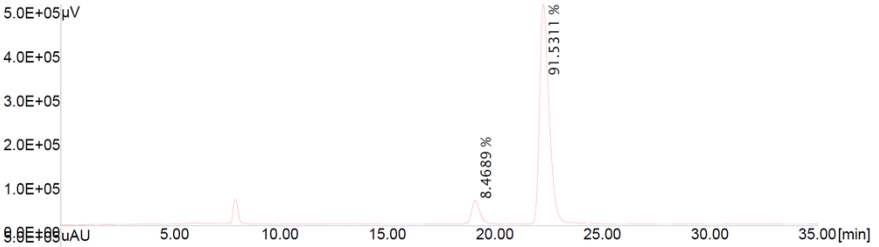
Comment             = single pulse decoupled gat
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = ECS 400
Spectrometer        = DELTA2_NMR

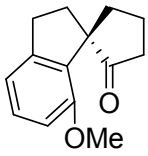
Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 1.04333312[s]
X_Domain            = 13C
X_Freq              = 100.52530333[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95846665[Hz]
X_Sweep             = 31.40703518[kHz]
X_Sweep_Clipped     = 25.12562814[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 1024
Total_Scans         = 1024

Relaxation_Delay    = 2[s]
Recvr_Gain          = 50
Temp_Get            = 19.1[dC]
X_90_Width          = 9.6[us]
X_Acq_Time          = 1.04333312[s]
X_Angle             = 30[deg]
X_Atn               = 6[dB]
X_Pulse             = 3.2[us]
Irr_Atn_Dec         = 21.63[dB]
Irr_Atn_Noe         = 21.63[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.04333312[s]
```

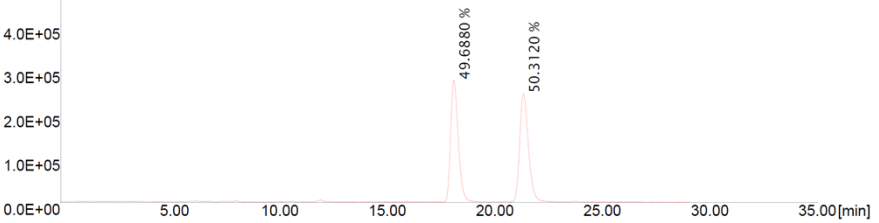

O
NHTs
Cl
9
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
Filename = aza-MBH-m-Cl-2_Proton-1-
Author = delta
Experiment = proton.jxp
Sample_Id = aza-MBH-m-Cl-2
Solvent = CHLOROFORM-D
Creation_Time = 24-FEB-2015 11:48:35
Revision_Time = 24-FEB-2015 11:39:12
Current_Time = 24-FEB-2015 11:40:10
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 22.7[dC]
X_90_Width = 12.1[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 2[dB]
X_Pulse = 6.05[us]
Irr_Mode = Off

HPLC Chart





2a



rac-2a

```
File name : spiro-SL-J001-90degree053.CH3

Info :
Hex/IPA 99/1, sample in Hex/IPA, IA, flow 0.5ml/min,

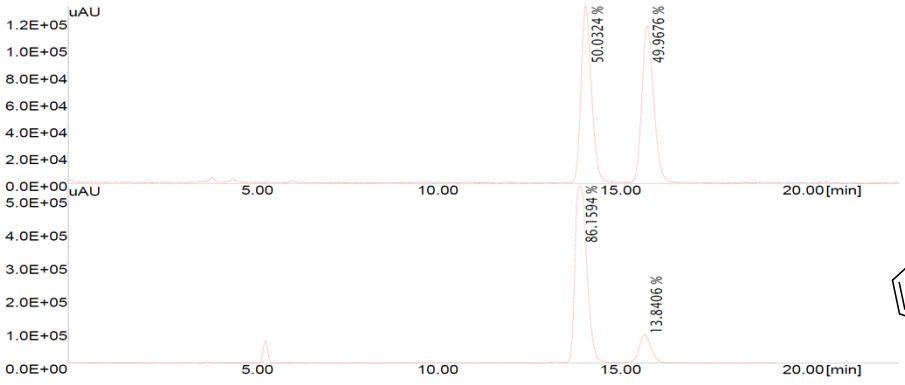
Vial # = 1 Rack # = 1
Injection Date :12-Mar-2013  20:24:10
Curr. Date :  5-Nov-2014  22:46:26
User : LULU
Group : SASAI
Control Method :

 #    Name       RT    Height[オV]   Area[オV.Sec]      %Area

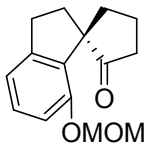
   1             19.283      53739       1415971.250      8.47
   2             22.450     498913      15303756.000     91.53

Total Area of Peak = 16719727.250 [オV.Sec]
File name : SPIRO-RAC181_220.CH1

Info :
IC Hex/IPA99/1
0.5 ml/min  rac
```



rac-2b



2b

```
File name : 20130406-2-RAC370_219.CH1

Info :
H/IPA= 99/1,  0.5 ml/min    OD-3, sample in H/IPA= 99/1

Wavelength = 219 [nm]
Tacc [Sec] = 0.80    Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date : 9-Apr-2013  14:53:46
Curr. Date :  6-Nov-2014  22:37:44
User : LULU
Group : SASAI
Control Method :

 #    Name       RT    Height[uAU]   Area[uAU.Sec]      %Area

   1             14.173      131314       2617201.239     50.03
   2             15.840      116467       2613807.239     49.97

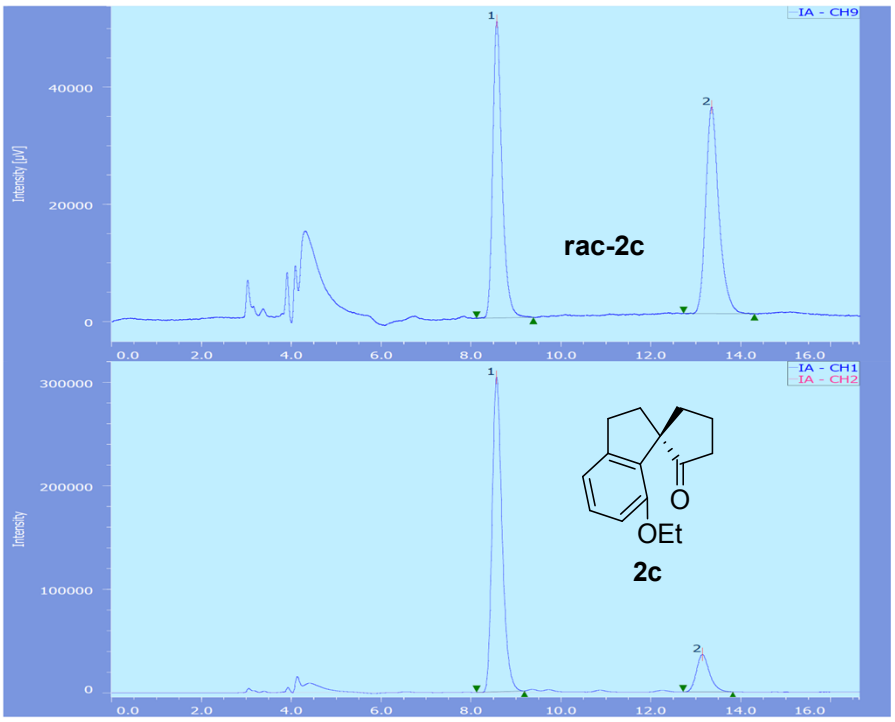
Total Area of Peak = 5231008.478 [uAU.Sec]
File name : 20130406-2-SL-J001-2369_219.CH1

Info :
H/IPA= 99/1,  0.5 ml/min    OD-3, sample in H/IPA= 99/1
Pd0Ac)2 5mol%, 90 degree, DME, ligand 7.5mol%
Wavelength = 219 [nm]
Tacc [Sec] = 0.80    Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date : 9-Apr-2013  13:44:04
Curr. Date :  6-Nov-2014  22:37:44
User : LULU
Group : SASAI
Control Method :

 #    Name       RT    Height[uAU]   Area[uAU.Sec]      %Area

   1             14.027      532218      12006826.179     86.16
   2             15.800       85435       1928764.829     13.84
```



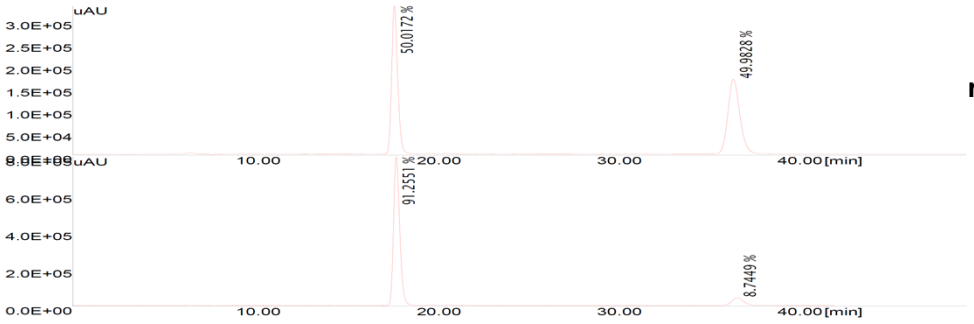
Chromatogram Name IA-CH9
Sample Name
Channel Name 205.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	8.567	726393	50602	50.331	58.977	N/A	9146	10.983	1.324	
2	Unknown	9	13.347	716839	35197	49.669	41.023	N/A	10770	N/A	1.251	

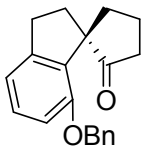
Chromatogram Name IA-CH1
Sample Name
Channel Name UV-975

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	8.558	4669479	303589	86.409	89.338	N/A	7492	10.077	1.305	
2	Unknown	1	13.133	734452	36232	13.591	10.662	N/A	10409	N/A	1.256	

Chromatogram Name IA-CH2
Sample Name
Channel Name CD-1595



rac-2d



2d

```
File name : 5-5-SPIRO KETONE-OBN536_220.CH1

Info :
IPA/Hex= 5/95,   0.5 ml/min     IA, sample in IPA/H= 5/95
rac
Wavelength = 220 [nm]
Tacc [Sec] = 0.80     Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date :19-Jan-2014  22:12:44
Curr. Date :  7-Nov-2014  11:21:42
User : LULU
Group : SASAI
Control Method :

 #     Name        RT     Height[uAU]    Area[uAU.Sec]       %Area

   1                17.813      332194         7738213.946      50.02
   2                36.587      168055         7732898.515      49.98

Total Area of Peak = 15471112.461 [uAU.Sec]
File name : 5-5-SPIRO KETONE-OBN535_220.CH1

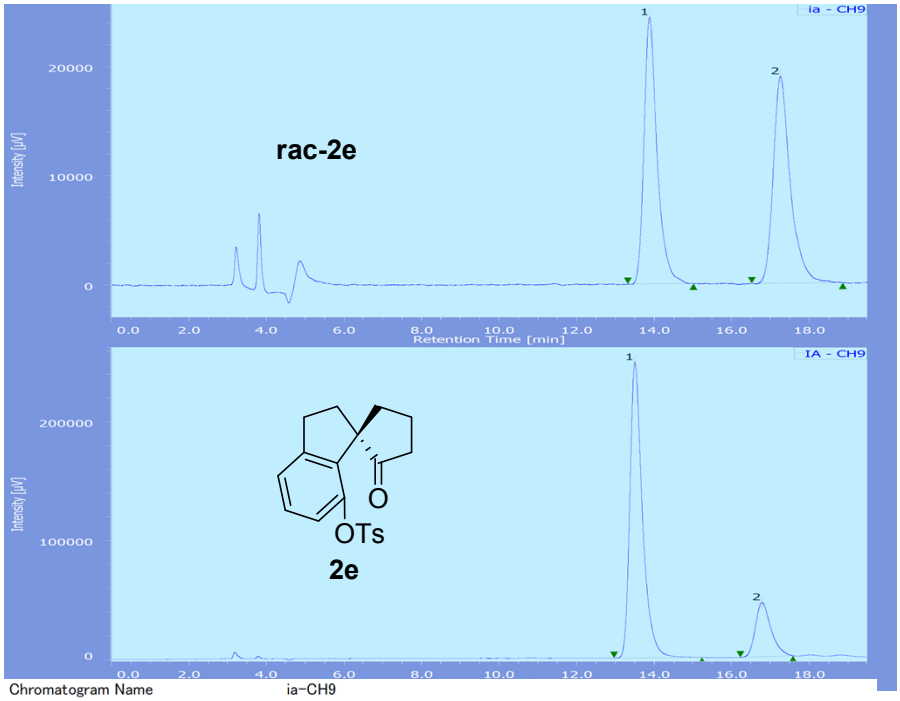
Info :
IPA/Hex= 5/95,   0.5 ml/min     IA, sample in IPA/H= 5/95
SL-J001-2
Wavelength = 220 [nm]
Tacc [Sec] = 0.80     Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date :19-Jan-2014  21:28:54
Curr. Date :  7-Nov-2014  11:21:42
User : LULU
Group : SASAI
Control Method :

 #     Name        RT     Height[uAU]    Area[uAU.Sec]       %Area

   1                17.920      800067        18990750.964      91.26
   2                36.813       41451         1819858.427       8.74

Total Area of Peak = 20810609.391 [uAU.Sec]
```

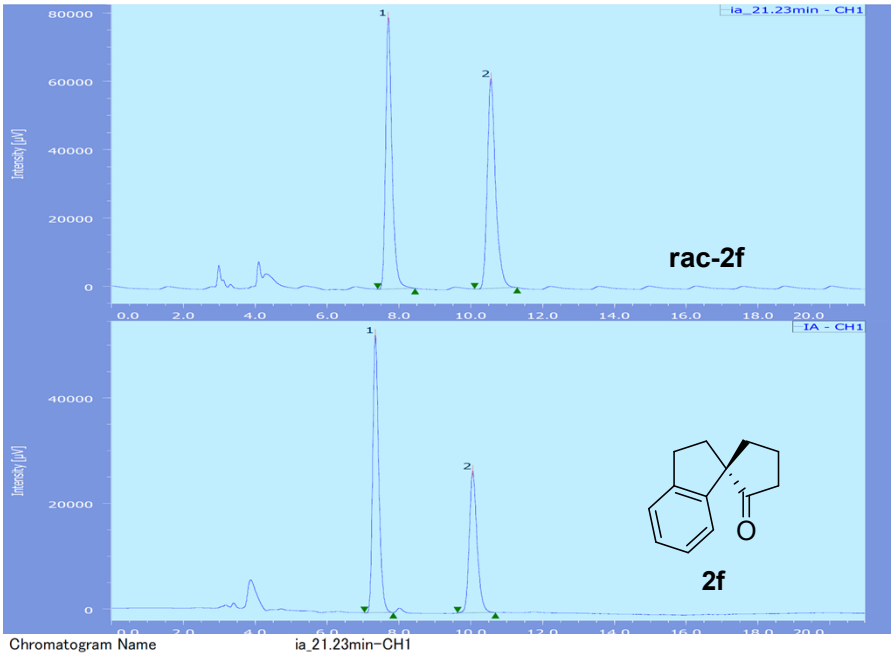


Chromatogram Name ia-CH9
Sample Name
Channel Name 225.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	13.872	573035	24398	49.665	56.309	N/A	9211	5.092	1.392	
2	Unknown	9	17.248	580761	18931	50.335	43.691	N/A	8425	N/A	1.406	

Chromatogram Name IA-CH9
Sample Name
Channel Name 225.0nm

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	9	13.495	5598878	248741	81.421	84.488	N/A	9461	5.179	1.465	
2	Unknown	9	16.773	1277549	45670	18.579	15.512	N/A	8819	N/A	1.332	

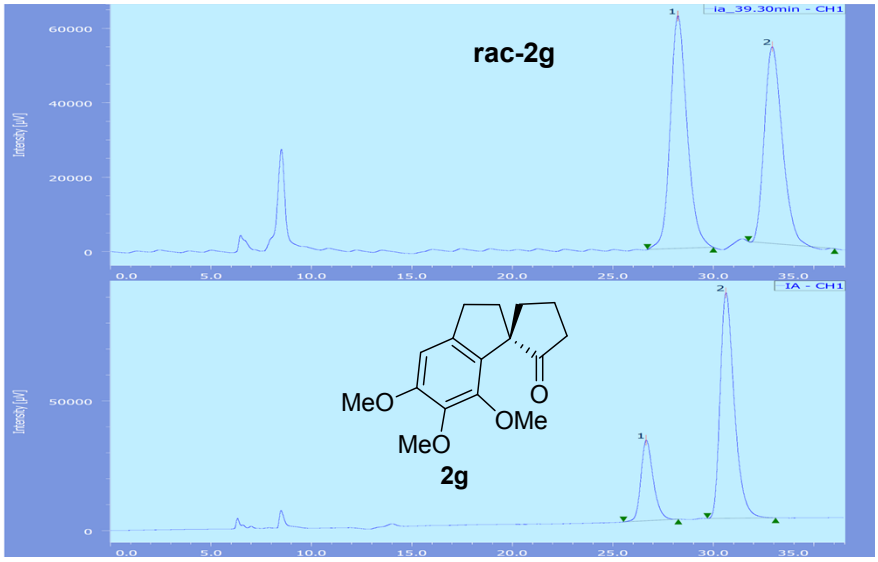


Chromatogram Name ia_21.23min-CH1
Sample Name
Channel Name UV-2075

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	7.700	977943	79344	49.926	56.413	N/A	10167	8.148	1.367	
2	Unknown	1	10.558	980860	61305	50.074	43.587	N/A	11261	N/A	1.306	

Chromatogram Name IA-CH1
Sample Name
Channel Name UV-975

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	7.342	613774	52566	60.319	66.234	N/A	9669	7.978	1.271	
2	Unknown	1	10.050	403768	26798	39.681	33.766	N/A	11067	N/A	1.239	



Channel & Peak Information Table

Chromatogram Name ia_39.30min-CH1

Sample Name

Channel Name UV-975

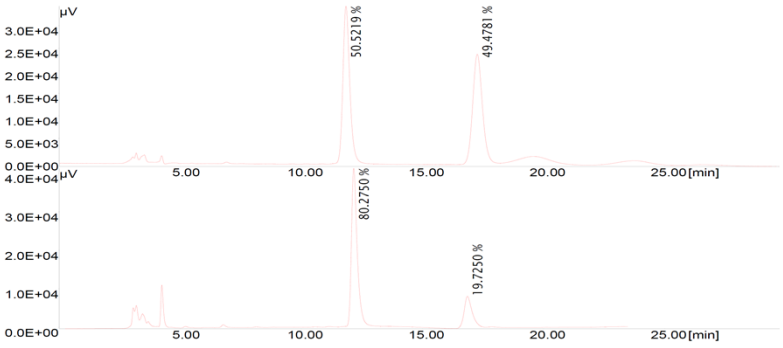
#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	28.208	3653582	62475	52.594	54.181	N/A	5727	3.010	1.190	
2	Unknown	1	32.900	3293219	52833	47.406	45.819	N/A	6479	N/A	1.267	

Chromatogram Name IA-CH1

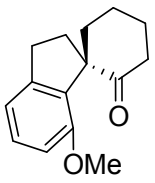
Sample Name

Channel Name UV-975

#	Peak Name	CH	tR [min]	Area [μV·sec]	Height [μV]	Area%	Height%	Quantity	NTP	Resolution	Symmetry Factor	Warning
1	Unknown	1	26.633	1323128	31043	23.709	26.333	N/A	9610	3.396	1.276	
2	Unknown	1	30.600	4257480	86846	76.291	73.667	N/A	9500	N/A	1.407	



rac-2h



2h

File name : spiro-6-OMe-rac078.CH3

Info :
Hex/IPA = 99/ 1, IA, flow 1ml/min, 219nm

Vial # = 1 Rack # = 1
Injection Date :13-Jun-2013 23:08:18
Curr. Date : 8-Nov-2014 13:39:22
User : LULU
Group : SASAI
Control Method :

#	Name	RT	Height[μV]	Area[μV.Sec]	%Area
1		11.842	34945	693415.000	50.52
2		17.258	24348	679088.000	49.48

Total Area of Peak = 1372503.000 [μV.Sec]

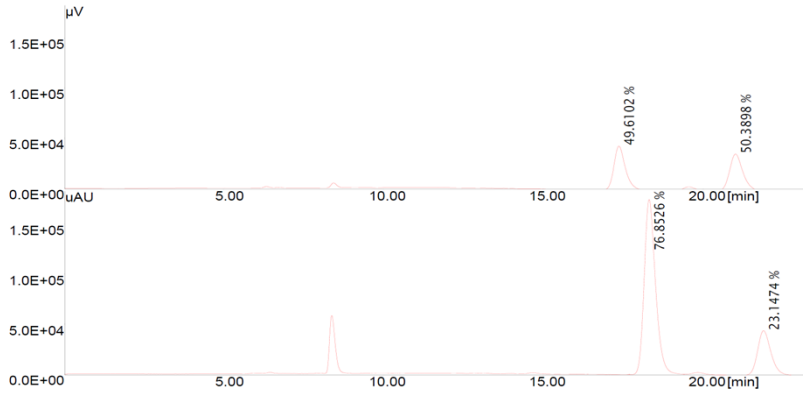
File name : spiro 6-OMe asy098.CH3

Info :
Hex/IPA = 99/ 1, IA, flow 1.0ml/min, 220nm
spiro-6-OMe

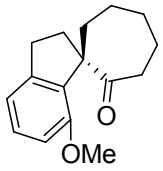
Vial # = 1 Rack # = 1
Injection Date : 8-Nov-2014 13:09:04
Curr. Date : 8-Nov-2014 13:39:22
User : LULU
Group : SASAI
Control Method :

#	Name	RT	Height[μV]	Area[μV.Sec]	%Area
1		12.167	41045	681295.776	80.27
2		16.858	7808	167406.750	19.73

Total Area of Peak = 848702.526 [μV.Sec]



rac-2j



2j

File name : spiro 7-OMe rac102.CH3

Info :
Hex/IPA = 99/ 1, 1A, flow 0.5ml/min, 220nm
spiro-7-OMe 302-1-2

Vial # = 1 Rack # = 1
Injection Date :28-Nov-2014 13:22:14
Curr. Date : 28-Nov-2014 14:03:46
User : LULU
Group : SASAI
Control Method :

#	Name	RT	Height[μV]	Area[μV.Sec]	%Area
1		17.367	43842	1090500.500	49.61
2		21.017	37343	1107638.038	50.39

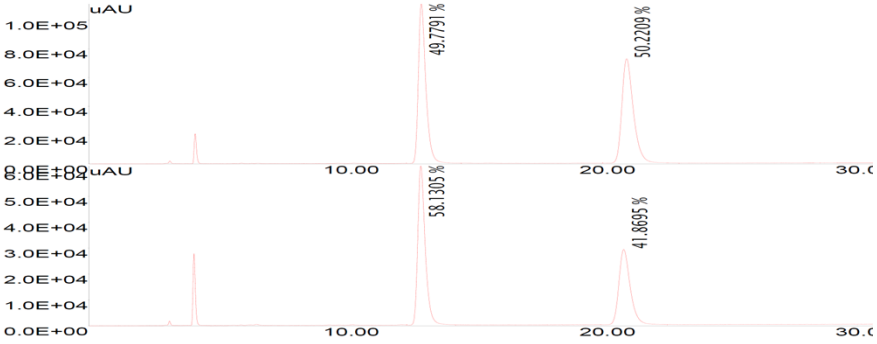
Total Area of Peak = 2198138.538 [μV.Sec]
File name : 5-7-SPIRO KETONE-OMES40_220.CH1

Info :
IPA/Hex= 1/99, 0.5 ml/min 1A, sample in IPA/H= 1/99
SL-J001-2
Wavelength = 220 [nm]
Tacc [Sec] = 0.80 Wacc [nm] = 4.0
Autozero [min] : 0.00

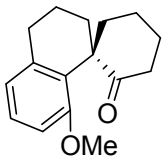
Vial # = 1 Rack # = 0
Injection Date :24-Jan-2014 1:01:06
Curr. Date : 28-Nov-2014 14:03:46
User : LULU
Group : SASAI
Control Method :

#	Name	RT	Height[uAU]	Area[uAU.Sec]	%Area
1		18.320	174754	4293021.875	76.85
2		21.907	44588	1293028.019	23.15

Total Area of Peak = 5586049.894 [uAU.Sec]



rac-2k



2k

File name : 6-6-SPIRO-KETONE-RAC493_220.CH1

Info :
IPA/HEX= 1/99, 1 ml/min 1A, sample in IPA/HEX= 1/99

Wavelength = 220 [nm]
Tacc [Sec] = 0.80 Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date : 7-Nov-2013 16:16:18
Curr. Date : 7-Nov-2014 20:31:56
User : LULU
Group : SASAI
Control Method :

#	Name	RT	Height[uAU]	Area[uAU.Sec]	%Area
1		12.973	110782	2239625.688	49.78
2		20.987	72579	2259501.634	50.22

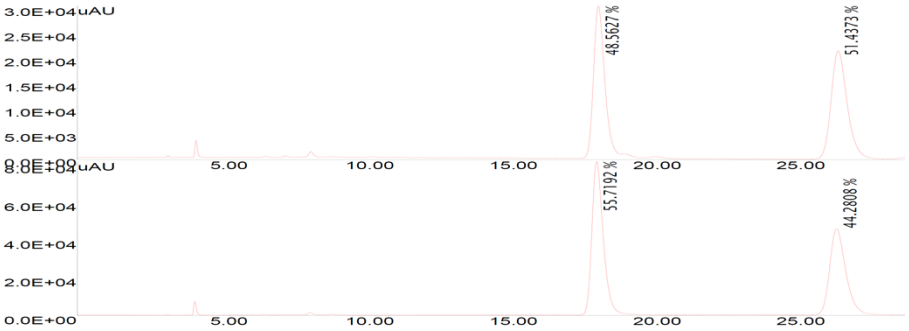
Total Area of Peak = 4499127.322 [uAU.Sec]
File name : 6-6-SPIRO-KETONE494_220.CH1

Info :
IPA/HEX= 1/99, 1 ml/min 1A, sample in IPA/HEX= 1/99
SL-J001-2
Wavelength = 220 [nm]
Tacc [Sec] = 0.80 Wacc [nm] = 4.0
Autozero [min] : 0.00

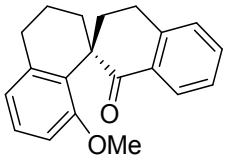
Vial # = 1 Rack # = 0
Injection Date : 7-Nov-2013 17:33:20
Curr. Date : 7-Nov-2014 20:31:56
User : LULU
Group : SASAI
Control Method :

#	Name	RT	Height[uAU]	Area[uAU.Sec]	%Area
1		12.973	61401	1236153.227	58.13
2		20.893	29074	890362.208	41.87

Total Area of Peak = 2126515.435 [uAU.Sec]



rac-2l



2l

```
File name : 6-NAPHALENE-SPIRO-KETONE-RAC495_239.CH1

Info :
IPA/HEX= 1/99,   1 ml/min     IA, sample in IPA/HEX= 1/99

Wavelength = 239 [nm]
Tacc [Sec] = 0.80     Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date :13-Nov-2013  16:07:24
Curr. Date :  7-Nov-2014  20:34:26
User : LULU
Group : SASAI
Control Method :

 #     Name        RT     Height[uAU]    Area[uAU.Sec]       %Area

   1               18.147       29915          791797.045     48.56
   2               26.480       21577          838666.412     51.44

Total Area of Peak = 1630463.457 [uAU.Sec]
File name : 6-NAPHALENE-SPIRO-KETONE496_239.CH1

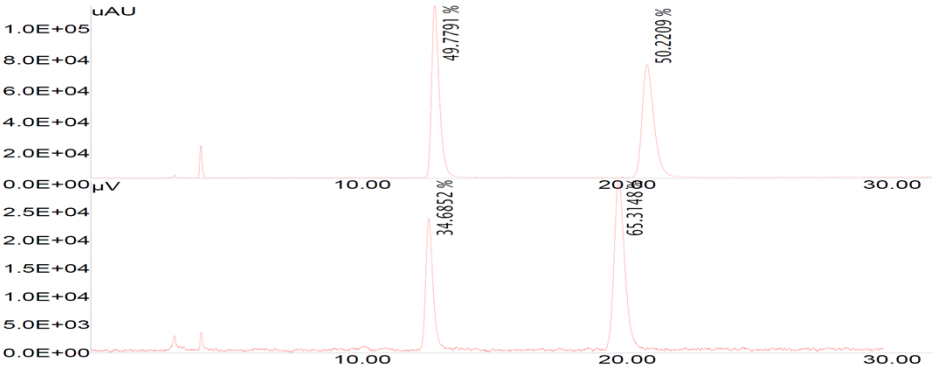
Info :
IPA/HEX= 1/99,   1 ml/min     IA, sample in IPA/HEX= 1/99
SL-J001-2   10mol%
Wavelength = 239 [nm]
Tacc [Sec] = 0.80     Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date :13-Nov-2013  16:42:32
Curr. Date :  7-Nov-2014  20:34:26
User : LULU
Group : SASAI
Control Method :

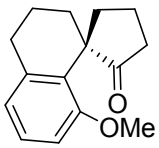
 #     Name        RT     Height[uAU]    Area[uAU.Sec]       %Area

   1               18.093       81159         2235738.833     55.72
   2               26.440       45644         1776771.626     44.28

Total Area of Peak = 4012510.460 [uAU.Sec]
```



rac-2m



2m

```
File name : 6-6-SPIRO-KETONE-RAC493_220.CH1

Info :
IPA/HEX= 1/99,   1 ml/min     IA, sample in IPA/HEX= 1/99

Wavelength = 220 [nm]
Tacc [Sec] = 0.80     Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date : 7-Nov-2013  16:16:18
Curr. Date : 13-Nov-2014  22:15:38
User : LULU
Group : SASAI
Control Method :

 #     Name        RT     Height[uAU]    Area[uAU.Sec]       %Area

   1               12.973      110782         2239625.688     49.78
   2               20.987       72579         2259501.634     50.22

Total Area of Peak = 4499127.322 [uAU.Sec]
File name : 6-5-spiro-ketone087.CH3

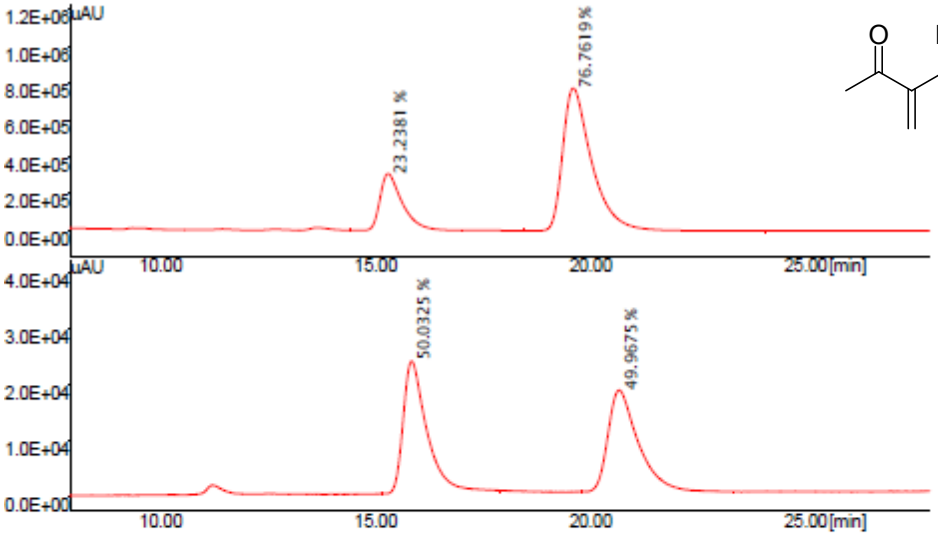
Info :
Hex/IPA = 99/ 1,   IA,  flow 1ml/min,   220nm
SL-M001-1

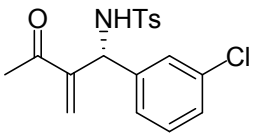
Vial # = 1 Rack # = 1
Injection Date :27-Nov-2013  19:31:48
Curr. Date : 13-Nov-2014  22:15:38
User : LULU
Group : SASAI
Control Method :

 #     Name        RT     Height[μV]     Area[μV.Sec]        %Area

   1               12.750       23448          453672.000     34.69
   2               19.917       30181          854299.561     65.31

Total Area of Peak = 1307971.561 [μV.Sec]
```





9

rac-9

File name : TAKI395_227.CH1

Info :
AD-H Hex/IPA-4/1 Flow-0.7mL/min 9-29-chiral
Wavelength = 227 [nm]
Tacc [Sec] = 0.80 Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date :26-Jan-2015 11:45:26
Curr. Date : 23-Feb-2015 17:21:26
User : TAKI2
Group : SASAI
Control Method :

#	Name	RT	Area[uAU.Sec]	%Area
1		15.400	10994803.821	23.24
2		19.707	36318864.165	76.76

Total Area of Peak = 47313667.986 [uAU.Sec]
File name : TAKI417_229.CH1

Info :
AD-H Hex/IPA-4-1 Flow-0.7mL/min 9-29-rac
Wavelength = 229 [nm]
Tacc [Sec] = 0.80 Wacc [nm] = 4.0
Autozero [min] : 0.00

Vial # = 1 Rack # = 0
Injection Date :23-Feb-2015 16:37:04
Curr. Date : 23-Feb-2015 17:21:26
User : TAKI2
Group : SASAI
Control Method :

#	Name	RT	Area[uAU.Sec]	%Area
1		15.947	905578.852	50.03
2		20.773	904400.813	49.97

Total Area of Peak = 1809979.666 [uAU.Sec]