Electronic Supplementary Material (ESI) for ChemComm.

Utilization of electron-donating α,β-unsaturated oximes: Regioselective inverse 1,3-dipolar cycloaddition of nitrones

Supporting Information

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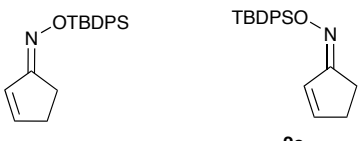
General procedure

^{1}H and ^{13}C NMR spectra were recorded with JEOL JNM-AL300, BRUKER AV-300 or BRUKER AV600 spectrometer at room temperature, with tetramethylsilane as an internal standard ($CDCl_3$ solution). Chemical shifts were recorded in ppm, and coupling constants (J) in Hz. Infrared (IR) spectra were recorded with a Shimadzu FTIR-8200A spectrometer. Mass spectra were recorded on JEOL JMS-700 and JMS-T100LP spectrometers. Melting points were determined by using a Yanaco melting point apparatus MP-S3. Merck silica gel 60 (1.09385) and Merck silica gel 60 F_{254} were used for column chromatography, thin layer chromatography (TLC), and preparative TLC (PTLC) respectively.

Synthesis and Data



2b and **2c**

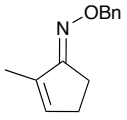
(*E*)-Cyclopent-2-en-1-one *O*-(*tert*-butyldiphenylsilyl) oxime (**2b**) and its (*Z*)-isomer (**2c**)

To a solution of *O*-TBDPS hydroxylamine (2.56 g, 9.43 mmol) in CH_2Cl_2 (128 mL) was added 2-cyclopenten-1-one (**2a**) (0.80 mL, 9.43 mmol). After stirring for 19

h at room temperature, the reaction mixture was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (hexane:$CHCl_3$ = 2:1) to afford (*E*)-TBDPS oxime **2b** (1.28 g, 40%) and (*Z*)-TBDPS oxime **2c** (0.960 g, 30%).

2b: IR (film) 2931, 2856, 1427, 1115, 935, 847, 741, 700 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.85-7.67 (4H, m), 7.49-7.29 (6H, m), 6.60 (1H, dt, *J* = 5.7, 2.7 Hz), 6.27 (1H, dt, *J* = 5.7, 2.2 Hz), 2.92-2.81 (2H, m), 2.63-2.53 (2H, m), 1.10 (9H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.2, 147.0, 135.4, 133.9, 129.5, 129.0, 127.5, 30.6, 27.0, 25.5, 19.4; HRMS (ESI) *m*/*z* calcd for $C_{21}H_{25}NOSi$ $[M+H]^+$ 336.1784, found 336.1768.

2c: IR (KBr) 2964, 2929, 2852, 1425, 1115, 931, 849, 736, 698 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.78-7.67 (4H, m), 7.43-7.30 (6H, m), 6.95 (1H, dt, *J* = 5.7, 1.8 Hz), 6.68 (1H, dt, *J* = 5.7, 2.4 Hz), 2.63-2.51 (4H, m), 1.10 (9H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.2, 148.6, 135.5, 134.0, 129.4, 127.5, 124.6, 30.3, 27.1, 26.4, 19.3; HRMS (ESI) *m*/*z* calcd for $C_{21}H_{25}NOSi$ $[M+K]^+$ 374.1343, found 374.1325.

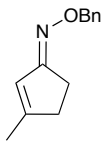


2g

(*E*)-2-Methylcyclopent-2-en-1-one *O*-benzyl oxime (**2g**)

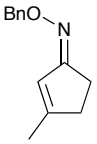
To a mixture of *O*-benzyl hydroxylamine (1.55 g, 9.70 mmol), NaOAc (3.00 g, 18.8 mmol) and Na_2SO_4 (2.30 g, 16.2 mmol) in MeOH (30 mL) was added 2-methyl-2-cyclopenten-1-one (**2a**) (0.800 mL, 8.10 mmol). After stirring for 2 h at room temperature, the reaction mixture was filtered and the filtrate concentrated under reduced pressure. The residue was diluted with water, and the whole was extracted with Et_2O (×3). The combined organic extracts were washed with brine (×1), dried (Na_2SO_4) and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (hexane:AcOEt = 4:1) to afford *E*-benzyl oxime **2g** (1.34 g, 92%) as a yellow oil.

2g: IR (film) 2920, 2852, 1454, 1038, 1018, 926, 696 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.45-7.25 (5H, m), 6.27-6.20 (1H, m), 5.14 (2H, s), 2.72-2.62 (2H, m), 2.48-2.36 (2H, m), 1.83 (3H, q, *J* = 1.8 Hz); ^{13}C NMR (75MHz, $CDCl_3$) δ 167.7, 141.5, 138.4, 137.1, 128.2, 128.0, 127.6, 75.8, 29.0, 26.0, 11.8; HRMS (EI) *m*/*z* calcd for $C_{13}H_{15}NO$ 201.1154, found 201.1145.



2i

and



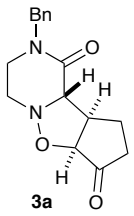
s1

(*E*)-3-Methylcyclopent-2-en-1-one *O*-benzyl oxime (**2i**) and its (*Z*)-isomer (**s1**)

To a mixture of *O*-benzyl hydroxylamine (3.00 g, 18.8 mmol), NaOAc (1.29 g, 15.7 mmol) and Na_2SO_4 (3.20 g, 22.6 mmol) in MeOH (60 mL) was added 3-methyl-2-cyclopenten-1-one (**2i)** (1.50 mL, 15.2 mmol). After stirring for 2 h at room temperature, the reaction mixture was filtered, and the filtrate was condensed under reduced pressure. The residue was diluted with water, and the whole was extracted with Et_2O (×3). The combined organic extracts were washed with brine (×1), dried (Na_2SO_4) and concentrated under reduced pressure. The resulting residue was purified by column chromatography on silica gel (hexane:AcOEt = 10:1) to afford *E*-benzyl oxime **2i** (2.33 g, 76%) and *Z*-benzyl oxime **s1** (0.555 g, 18%) both as a yellow oil.

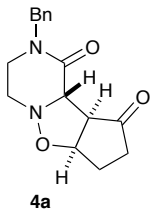
2i: IR (film) 2910, 2868, 1628, 1441, 1041, 1020, 924, 847, 698 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.46-7.20 (5H, m), 5.95-5.87 (1H, m), 5.09 (2H, s), 2.76-2.67 (2H, m), 2.49-2.35 (2H, m), 1.93 (3H, q, J = 1.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.9, 158.8, 138.5, 128.3, 127.8, 127.5, 123.7, 75.5, 34.9, 26.4, 17.9; HRMS (EI) *m*/*z* calcd for $C_{13}H_{15}NO$ 201.1154, found 201.1144.

s1: IR (Film) 2912, 2854, 1622, 1439, 1045, 1026, 852, 698 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.42-7.23 (5H, m), 6.47-6.36 (1H, m), 5.06 (2H, s), 2.70-2.57 (2H, m), 2.50-2.40 (2H, m), 1.95 (3H, q, J = 1.2 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 161.4, 138.4, 128.2, 127.9, 127.5, 120.3, 75.4, 34.0, 27.7, 18.2; HRMS (EI) *m*/*z* calcd for $C_{13}H_{15}NO$ 201.1154, found 201.1151.



3a

and



4a

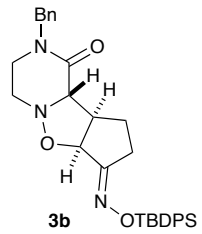
(6a*S*,9a*R*,9b*S*)-2-benzylhexahydrocyclopenta[4,5]isoxazolo[2,3-*a*]pyrazine-1,7(2*H*,6a*H*)-dione (**3a**) and its 1,9-dione isomer (**4a**)

To a solution of 2-cyclopenten-1-one (**2a**) (41.0 µL, 0.490 mmol) in THF (1.0 mL)

was added nitrone **1a** (50.0 mg, 0.245 mmol). After stirring at 60 °C for 24 h, the reaction mixture was concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (hexane:AcOEt = 1:1) to afford 5-acylisoxazolidine **3a** (15.2 mg, 22%) and 4-acylisoxazolidine **4a** (40.3 mg, 58%) both as a yellow oil.

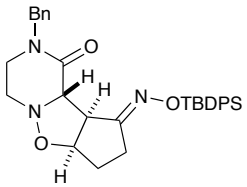
3a: IR (KBr) 2926, 1749, 1645, 1495, 1454, 1356, 1261, 702 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.42-7.17 (5H, m), 4.73 (1H, d, *J* = 14.7 Hz), 4.49 (1H, d, *J* = 14.7 Hz), 4.26 (1H, d, *J* = 8.4 Hz), 3.84 (1H, d, *J* = 3.6 Hz), 3.74-3.54 (2H, m), 3.33 (1H, dt, *J* = 13.2, 4.5 Hz), 3.28-3.16 (1H, m), 3.12 (1H, dt, *J* = 12.3, 4.5 Hz), 2.64-2.14 (4H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 215.0, 167.7, 136.1, 128.8, 128.0, 127.8, 79.6, 70.5, 49.9, 48.0, 47.4, 41.5, 35.8, 24.6; HRMS (EI) *m*/*z* calcd for $C_{16}H_{18}N_2O_3$ 286.1317, found 286.1307.

4a: IR (KBr) 2928, 1744, 1647, 1495, 1454, 1356, 1225, 702 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.17 (5H,m), 4.91 (1H, dt, *J* = 6.6, 3.6 Hz), 4.68 (1H, d, *J* = 14.7 Hz), 4.54 (1H, d, *J* = 14.7 Hz), 4.17 (1H, d, *J* = 1.8 Hz), 3.71-3.58 (1H, m), 3.56 (1H, d, *J* = 6.9 Hz), 3.32 (1H, dt, *J* = 13.8, 4.2 Hz), 3.29-3.16 (1H, m), 3.08 (1H, dt, *J* = 12.0, 4.2 Hz), 2.64-2.45 (1H, m), 2.45-2.30 (1H, m), 2.30-2.13 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 216.3, 166.8, 136.2, 128.7, 128.0, 127.7, 78.6, 68.0, 59.1, 50.3, 47.2, 41.5, 36.7, 27.7; HRMS (EI) *m*/*z* calcd for $C_{16}H_{18}N_2O_3$ 286.1317, found 286.1312.



3b

and



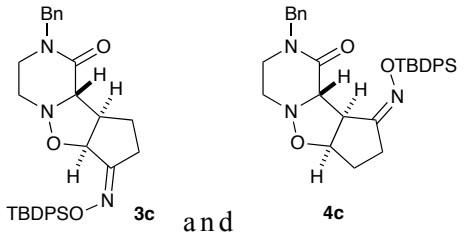
4b

(6a*S*,9a*R*,9b*S*,*E*)-2-benzyl-7-{[(*tert*-butyldiphenylsilyl)oxy]imino}octahydrocyclopenta[4,5]isoxazolo[2,3-*a*]pyrazin-1(2*H*)-one (**3b**) and its 9-imino isomer (**4b**)

To a solution of α,β-unsaturated oxime **2b** (164 mg, 0.490 mmol) in THF (1.0 mL) was added nitrone **1a** (50.0 mg, 0.245 mmol). After stirring at 60 °C for 65 h, the reaction mixture was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 2:1) to afford 5-iminoisoxazolidine **3b** (93.8 mg, 71%) as a white amorphous solid and 4-iminoisoxazolidine **4b** (18.2 mg, 14%) as a yellow oil.

3b: IR (KBr) 2932, 2893, 1653, 1489, 1429, 1115, 939, 889, 700 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.76–7.63 (4H, m), 7.46–7.13 (11H, m), 4.93 (1H, d, *J* = 7.5 Hz), 4.65 (1H, d, *J* = 14.7 Hz), 4.53 (1H, d, *J* = 14.7 Hz), 3.82 (1H, d, *J* = 5.7 Hz), 3.55-3.41 (1H, m), 3.42-3.30 (1H, m), 3.29-3.02 (4H, m), 2.75-2.53 (1H, m), 2.25 (1H, ddt, *J* = 13.8, 9.0, 2.4 Hz), 2.18-2.00 (1H, m), 1.09 (9H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 167.9, 136.1, 135.44, 135.41, 133.6, 133.5, 129.6, 128.8, 128.0, 127.7, 127.5, 81.0, 70.7, 50.9, 49.6, 47.8, 42.6, 27.4, 27.0, 25.1, 19.4 (several signals overlapped); HRMS (EI) *m*/*z* calcd for $C_{32}H_{37}N_3O_3Si$ 539.2604, found 539.2607.

4b: IR (KBr) 2931, 2858, 1653, 1489, 1429, 1115, 935, 702 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.80-7.64 (4H, m), 7.46-7.17 (11H, m), 4.74 (1H, t, *J* = 5.7 Hz), 4.65 (1H, d, *J* = 14.7 Hz), 4.52 (1H, d, *J* = 14.7 Hz), 4.07-3.96 (2H, m), 3.69 (1H, td, *J* = 11.4, 3.9 Hz), 3.39 (1H, ddd, *J* = 14.1, 3.9, 2.7 Hz), 3.28-3.07 (1H, m), 3.07-2.86 (2H, m), 2.88-2.70 (1H, m), 2.22-2.06 (1H, m), 2.06-1.85 (1H, m), 1.11 (9H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.7, 167.2, 136.4, 135.6, 135.5, 133.93, 133.85, 129.5, 129.4, 128.7, 128.1, 127.6, 127.5, 127.4, 80.8, 70.4, 54.7, 50.2, 47.2, 41.3, 30.6, 27.2, 27.0, 19.4 (several signals overlapped); HRMS (EI) *m*/*z* calcd for $C_{32}H_{37}N_3O_3Si$ 539.2604, found 539.2607.



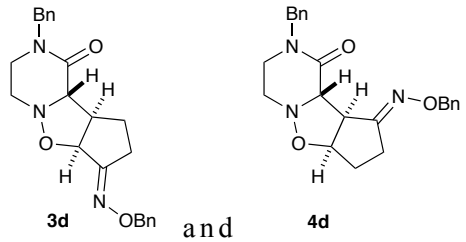
(6a*S*,9a*R*,9b*S*,*Z*)-2-benzyl-7-{[(*tert*-butyldiphenylsilyl)oxy]imino}octahydrocyclopenta[4,5]isoxazolo[2,3-*a*]pyrazin-1(2*H*)-one (**3c**) and its 9-iminoisomer (**4c**)

To a solution of α,β-unsaturated oxime **2c** (164 mg, 0.490 mmol) in THF (1.0 mL) was added nitrone **1a** (50.0 mg, 0.245 mmol). After stirring at 60 °C for 40 h, the reaction mixture was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 4:1) to afford 5-iminoisoxazolidine **3c** (93.8 mg, 71%) as a white amorphous solid and 4-iminoisoxazolidine **4c** (18.2 mg, 14%) as a pale yellow oil.

3c: IR (KBr) 2932, 2858, 1653, 1489, 1429, 1115, 939, 700 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.80-7.65 (4H, m), 7.45-7.18 (11H, m), 5.52 (1H, d, *J* = 7.5 Hz), 4.65 (1H, d, *J* = 15.0 Hz), 4.59 (1H, d, *J* = 15.0 Hz), 3.80 (1H, d, *J* = 6.3 Hz), 3.54-3.35 (2H,

m), 3.35-3.05 (3H, m), 2.65 (1H, ddd, *J* = 16.5, 12.0, 8.1 Hz), 2.49 (1H, ddd, *J* = 16.5, 8.1, 1.8 Hz), 2.22 (1H, br dd, *J* = 13.5, 8.1 Hz), 2.11-1.91 (1H, m), 1.09 (9H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 167.0, 136.1, 135.50, 135.47, 133.6, 129.6, 129.5, 128.8, 128.0, 127.7, 127.52, 127.47, 75.6, 70.0, 50.8, 49.6, 47.9, 42.6, 27.9, 27.1, 27.0, 19.4 (several signals overlapped); HRMS (EI) *m*/*z* calcd for $C_{32}H_{37}N_3O_3Si$ 539.2604, found 539.2603.

4c: IR (KBr) 2932, 2856, 1653, 1427, 1115, 951, 702 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85-7.71 (4H, m), 7.47-7.20 (11H, m), 4.74 (1H, t, *J* = 6.0 Hz), 4.70 (1H, d, *J* = 14.7 Hz), 4.62 (1H, d, *J* = 14.7 Hz), 4.58 (1H, d, *J* = 6.3 Hz), 4.43 (1H, s), 3.76 (1H, td, *J* = 11.7, 4.2 Hz), 3.42 (1H, ddd, *J* = 14.4, 4.2, 1.8 Hz), 3.35-3.18 (1H, m), 2.97 (1H, ddd, *J* = 12.0, 4.8, 1.8 Hz), 2.78-2.58 (1H, m), 2.48 (1H, ddd, *J* = 16.2, 8.4, 1.5 Hz), 2.13 (1H, dd, *J* = 14.4, 8.4 Hz), 1.94-1.76 (1H, m), 1.14 (9H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.4, 166.9, 136.6, 135.6, 133.71, 133.65, 129.5, 128.7, 128.0, 127.5, 80.7, 67.7, 52.2, 50.3, 46.9, 41.2, 31.9, 29.7, 29.5, 27.0, 19.4 (several signals overlapped); HRMS (ESI) *m*/*z* calcd for $C_{32}H_{37}N_3O_3Si$ 539.2604, found 539.2622.



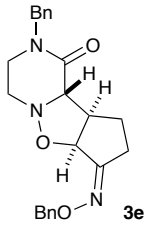
(6a*S*,9a*R*,9b*S*,*E*)-2-benzyl-7-[(benzyloxy)imino]octahydrocyclopenta[4,5]isoxazolo[2,3-*a*]pyrazin-1(2*H*)-one (**3d**) and its 9-iminoisomer (**4d**)

To a solution of α,β-unsaturated oxime **2e** (375 mg, 2.00 mmol) in THF (1.0 mL) was added nitrone **1a** (204 mg, 1.00 mmol). After stirring at 60 °C for 120 h, the reaction mixture was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 4:1) to afford 5-iminoisoxazolidine **3d** (320 mg, 82%) as a white solid and 4-iminoisoxazolidine **4d** (33.7 mg, 9%) as a pale yellow oil.

3d: m.p. 113.5-115.0 °C (AcOEt/hexane, colorless needle); IR (KBr) 2962, 2856, 1660, 1460, 1377, 1007, 978 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.18 (10H, m), 5.17 (1H, d, *J* = 12.3 Hz), 5.12 (1H, d, *J* = 12.3 Hz), 4.92 (1H, d, *J* = 7.5 Hz), 4.63 (1H, d, *J* = 15.6 Hz), 4.57 (1H, d, *J* = 15.6 Hz), 3.79 (1H, d, *J* = 5.1 Hz), 3.57-3.36 (2H, m), 3.28-3.13 (3H, m), 2.79 (1H, ddd, *J* =18.0, 8.7, 3.6 Hz), 2.55 (1H, dt, *J* =

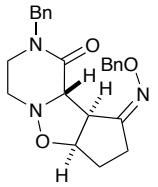
18.0, 8.7 Hz), 2.26-1.97 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 162.9, 137.6, 136.1, 128.8, 128.3, 128.1, 128.0, 127.8, 127.7, 81.1, 76.3, 70.5, 50.6, 49.7, 47.7, 42.2, 27.8, 25.5; HRMS (EI) *m/z* calcd for $C_{23}H_{25}N_3O_3$ 391.1896, found 391.1907.

4d: IR (KBr) 2930, 1647, 1495, 1456, 1028, 700 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.20 (10H, m), 5.15 (2H, s), 4.76 (1H, t, *J* = 6.6 Hz), 4.73 (1H, d, *J* = 14.7 Hz), 4.52 (1H, d, *J* = 14.7 Hz), 4.13 (1H, d, *J* = 2.1 Hz), 3.97 (1H, dt, *J* = 6.6, 2.1 Hz), 3.67-3.53 (1H, m), 3.32 (1H, dt, *J* = 13.5, 4.0 Hz), 3.27-3.14 (1H, m), 3.08 (1H, dt, *J* = 12.3, 4.0 Hz), 2.75 (1H, ddd, *J* = 18.3, 9.6, 3.6 Hz), 2.68-2.51 (1H, m), 2.16-2.00 (1H, m), 2.01-1.83 (1H, m); ^{13}C NMR (75MHz, $CDCl_3$) δ 167.2, 164.8, 137.9, 136.3, 128.7, 128.3, 128.2, 128.1, 127.7, 80.5, 77.2, 76.1, 70.6, 55.1, 50.1, 47.4, 41.6, 30.2, 26.4; HRMS (EI) *m/z* calcd for $C_{23}H_{25}N_3O_3$ 391.1896, found 391.1896.



3e

and



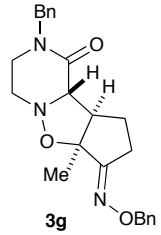
4e

(6a*S*,9a*R*,9b*S*,*Z*)-2-benzyl-7-[(benzyloxy)imino]octahydrocyclopenta[4,5]isoxazolo[2,3-*a*]pyrazin-1(2*H*)-one (**3e**) and its 9-iminoisomer (**4e**)

To a solution of α,β-unsaturated oxime **2f** (91.7 mg, 0.490 mmol) in THF (1.0 mL) was added nitrone **1a** (50.0 mg, 0.245 mmol). After stirring at 60 °C for 20 h, the reaction mixture was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 2:1) to afford 5-iminoisoxazolidine **3e** (63.2 mg, 66%) as a white solid and 4-iminoisoxazolidine **4e** (8.5 mg, 9%) as a pale yellow oil. Nitrone **1a** (6.2 mg, ca. 12%) was recovered with a trace amount of impurity.

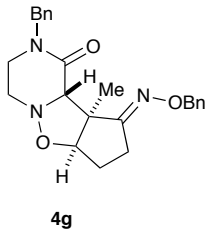
3e: m.p. 103.0-105.0 °C (toluene, colorless needle); IR (KBr) 2927, 2854, 1660, 1452, 1011, 854, 700 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43-7.17 (10H, m), 5.28 (1H, d, *J* = 7.8 Hz), 5.17 (1H, d, *J* = 12.9 Hz), 5.11 (1H, d, *J* = 12.9 Hz), 4.62 (1H, d, *J* = 15.0 Hz), 4.57 (1H, d, *J* = 15.0 Hz), 3.76 (1H, d, *J* = 6.3 Hz), 3.53-3.33 (2H, m), 3.33-3.06 (3H, m), 2.76-2.55 (1H, m), 2.47 (1H, dd, *J* = 16.5, 6.3 Hz), 2.21 (1H, dd, *J* = 13.5, 8.1 Hz), 2.07-1.87 (1H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 161.9, 138.1, 136.1, 128.7, 128.3, 128.0, 127.9, 127.7, 127.6, 76.0, 75.7, 69.8, 51.0, 49.6, 47.9, 42.5, 28.1, 26.8; HRMS (ESI) *m/z* calcd for $C_{23}H_{25}N_3O_3$ $[M+H]^+$ 392.1974, found 392.1960.

4e: IR (film) 2927, 1647, 1489, 1454, 1049, 739 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.48-7.39 (2H, m), 7.39-7.20 (8H, m), 5.19 (1H, d, *J* = 13.5 Hz), 5.15 (1H, d, *J* = 13.5 Hz), 4.74-4.66 (1H, m), 4.68 (1H, d, *J* = 14.4 Hz), 4.57 (1H, d, *J* = 14.4 Hz), 4.35 (1H, br d, *J* = 6.3 Hz), 4.23 (1H, s), 3.72 (1H, td, *J* = 12.0, 4.2 Hz), 3.38 (1H, ddd, *J* = 14.4, 4.2, 2.1 Hz), 3.28-3.13 (1H, m), 2.98 (1H, ddd, *J* = 12.0, 4.8, 2.1 Hz), 2.78-2.58 (1H, m), 2.45 (1H, ddd, *J* = 16.2, 8.4, 1.8 Hz), 2.10 (1H, dd, *J* = 14.1, 8.4 Hz), 1.92-1.74 (1H, m) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.1, 164.6, 138.4, 136.5, 128.7, 128.3, 128.0, 127.63, 127.60, 127.4, 80.9, 75.7, 67.6, 51.9, 50.2, 46.9, 41.2, 31.5, 29.6; HRMS (ESI) *m/z* calcd for $C_{23}H_{25}N_3O_3$ $[M+Na]^+$ 414.1794, found 414.1778.



3g

and



4g

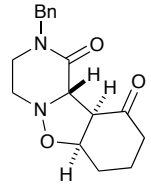
(6a*S*,9a*R*,9b*S*,*E*)-2-benzyl-7-[(benzyloxy)imino]-6a-methyloctahydrocyclopenta[4,5]isoxazolo[2,3-*a*]pyrazin-1(2*H*)-one (**3g**) and its 9-iminoisomer (**4g**)

To a solution of α,β-unsaturated oxime **2g** (99.1 mg, 0.490 mmol) in THF (1.0 mL) was added nitrone **1a** (50.0 mg, 0.245 mmol). After stirring at 110 °C for 30 h, the reaction mixture was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 1:1) followed by PTLC (hexane:AcOEt:Et_2O = 1:2:1) to afford 5-iminoisoxazolidine **3g** (29.5 mg, 30%) and 4-iminoisoxazolidine **4g** (24.8 mg, 25%) both as a yellow oil.

3g: IR (film) 2972, 2929, 1645, 1489, 1454, 1354, 1016 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.45-7.17 (10H, m), 5.14 (2H, s), 4.65 (1H, d, *J* = 14.4 Hz), 4.59 (1H, d, *J* = 14.4 Hz), 3.74 (2H, td, *J* = 11.1, 3.6 Hz), 3.44 (1H, dt, *J* = 13.8, 3.3 Hz), 3.30-3.10 (2H, m), 3.02 (1H, ddd, *J* = 12.3, 4.2, 3.3 Hz), 2.80-2.57 (2H, m), 2.19-1.93 (2H, m), 1.46 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.5, 166.0, 137.8, 136.3, 128.7, 128.23, 128.16, 128.0, 127.7, 87.5, 76.2, 70.5, 56.9, 50.2, 47.5, 41.3, 27.2, 26.8, 22.3 (several signals overlapped); HRMS (ESI) *m/z* calcd for $C_{24}H_{27}N_3O_3$ $[M+K]^+$ 444.1690, found 444.1695.

4g: IR (film) 3033, 2927, 2870, 1496, 1454, 1365, 1018, 733 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.46-7.14 (10H, m), 5.16 (1H, d, *J* = 12.0 Hz), 5.12 (1H, d, *J* = 12.0

Hz), 4.58 (2H, s), 4.33 (1H, br d, J = 6.6 Hz), 3.50 (1H, br s), 3.38-2.90 (4H, m), 2.80-2.40 (2H, m), 2.18-1.85 (2H, m), 1.54 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.8, 163.4, 137.7, 136.2, 128.8, 128.3, 128.2, 127.8, 88.2, 76.3, 66.7, 57.2, 49.9, 49.7, 41.9, 26.8, 24.0, 22.2 (several signals overlapped); HRMS (ESI) m/z calcd for $C_{24}H_{27}N_3O_3$ $[M+Na]^+$ 428.1950, found 428.1933.

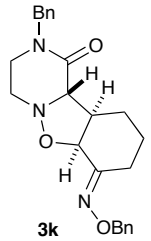


4j

(6a*R*,10a*R*,10b*S*)-2-benzyloctahydro-2*H*-benzo[4,5]isoxazolo[2,3-*a*]pyrazine-1,10-dione (**4j**)

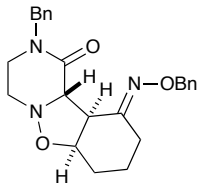
To a solution of 2-cyclohexen-1-one (**2k**) (47.0 μL, 0.490 mmol) in THF (1.0 mL) was added nitrone **1a** (50.0 mg, 0.245 mmol). After stirring at 60 °C for 120 h, the reaction mixture was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 1:3) to afford 4-acylisoxazolidine **4j** (50.2 mg, 68%) as a white solid.

4j: m.p. 128.0-130.0 °C (AcOEt/hexane, colorless plate); IR (KBr) 2924, 2866, 1707, 1637, 1491, 1450, 1356, 1036, 741 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.21 (5H, m), 4.75 (1H, d, J = 1.8 Hz), 4.70 (1H, d, J = 14.4 Hz), 4.65-4.55 (1H, m), 4.55 (1H, d, J = 14.4 Hz), 3.65-3.49 (2H, m), 3.33-3.23 (2H, m), 3.13 (1H, dt, J = 12.3, 4.2 Hz), 2.56 (1H, td, J = 15.9, 4.5 Hz), 2.48-2.29 (1H, m), 2.14-1.78 (4H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.5, 167.7, 136.2, 128.8, 128.2, 127.8, 64.3, 59.3, 50.4, 48.7, 41.5, 40.0, 26.9, 19.4 (several signals overlapped); HRMS (EI) m/z calcd for $C_{17}H_{20}N_2O_3$ 300.1474, found 300.1480.



3k

and



4k

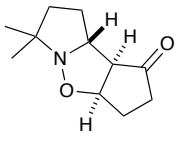
(6a*S*,10a*R*,10b*S*,*E*)-2-benzyl-7-[(benzyloxy)imino]octahydro-2*H*-benzo[4,5]isoxazolo[2,3-*a*]pyrazin-1(6a*H*)-one (**3k**) and its 10-iminoisomer (**4k**)

To a solution of α,β-unsaturated oxime **2l** (98.7 mg, 0.490 mmol) in THF (1.0 mL) was added nitrone **1a** (50.0 mg, 0.245 mmol). After stirring at 60 °C for 244 h,

the reaction mixture was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 6:5) to afford 5-iminoisoxazolidine **3k** (66.6 mg, 67%) and 4-iminoisoxazolidine **4k** (12.4 mg, 12%) both as a pale yellow oil.

3k: IR (Film) 2935, 2866, 1653, 1489, 1454, 1362, 1016, 750 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.41-7.20 (10H, m), 5.16 (1H, d, *J* = 12.1 Hz), 5.11 (1H, d, *J* = 12.1 Hz), 4.69 (1H, d, *J* = 14.7 Hz), 4.61 (1H, d, *J* = 6.9 Hz), 4.52 (1H, d, *J* = 14.7 Hz), 3.94 (1H, d, *J* = 4.5 Hz), 3.50-3.36 (1H, m), 3.34-3.06 (4H, m), 2.84 (1H, dt, *J* = 17.4, 6.6 Hz), 2.31 (1H, dt, *J* = 17.4, 6.6 Hz), 2.06-1.69 (3H, m), 1.68-1.46(1H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 154.8, 137.5, 136.1, 128.8, 128.3, 128.1, 127.9, 127.8, 76.1, 75.8, 69.6, 49.8, 48.5, 47.8, 42.3, 26.4, 22.5, 18.5 (several signals overlapped); HRMS (ESI) *m*/*z* calcd for $C_{24}H_{27}N_3O_3$ $[M+Na]^+$ 428.1950, found 428.1931.

4k: IR (Film) 2929, 2868, 1655, 1489, 1454, 1045, 1016 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.43-7.20 (10H, m), 5.18 (1H, d, *J* = 12.0 Hz), 5.12 (1H, d, *J* = 12.0 Hz), 4.75 (1H, d, *J* = 14.7 Hz), 4.62-4.51 (1H, m), 4.49 (1H, d, *J* = 14.7 Hz), 4.38 (1H, d, *J* = 6.6 Hz), 3.63 (1H, t, *J* = 7.2 Hz), 3.45-3.05 (4H, m), 2.74 (1H, dt, *J* = 17.1, 5.1 Hz), 2.67-2.41 (1H, m), 1.96-1.70 (2H, m), 1.70-1.46 (2H, m); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.0, 155.3, 137.9, 136.3, 128.7, 128.3, 128.2, 128.0, 127.7, 127.6, 76.2, 75.8, 66.7, 50.4, 49.9, 48.6, 42.6, 27.2, 23.3, 16.6; HRMS (ESI) *m*/*z* calcd for $C_{24}H_{27}N_3O_3$ $[M+H]^+$ 406.2131, found 406.2144.



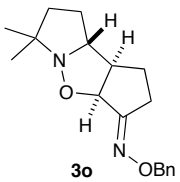
4n

(3a*R*,8a*R*,8b*R*)-6,6-dimethyloctahydro-1*H*-cyclopenta[*d*]pyrrolo[1,2-*b*]isoxazol-1-one (**4n**)

To a solution of 2-cyclopenten-1-one (**2a**) (74.0 μL, 0.884 mmol) in THF (1.8 mL) was added nitrone **1c** (50.0 mg, 0.442 mmol). After stirring at 60 °C for 2 days, the reaction mixture was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 4:1) to afford 4-acylisoxazolidine **4n** (66.3 mg, 77%) as a pale yellow oil.

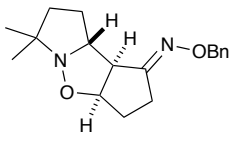
4n: IR (KBr) 2968, 1749, 1558, 1541, 1458, 1155 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 4.80 (1H, br t, *J* = 5.7 Hz), 3.99 (1H, dd, *J* = 9.0, 4.8 Hz), 2.85 (1H, d, *J* = 5.7 Hz),

2.68-2.48 (1H, m), 2.40-2.14 (3H, m), 2.15-1.96 (1H, m), 1.85-1.69 (1H, m), 1.70-1.53 (2H, m), 1.31 (3H, s), 1.03 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 218.1, 79.5, 69.0, 68.7, 63.2, 36.2, 35.8, 31.0, 27.34, 27.31, 23.9; HRMS (EI) *m*/*z* calcd for $C_{11}H_{17}NO_2$ 195.1259, found 195.1251.



3o

and



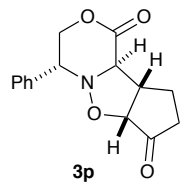
4o

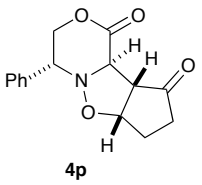
(3a*S*,8a*R*,8b*R*,*E*)-6,6-dimethyloctahydro-3*H*-cyclopenta[*d*]pyrrolo[1,2-*b*]isoxazol-3-one *O*-benzyl oxime (**3o**) and its 9-iminoisomer (**4o**)

To a solution of α,β-unsaturated oxime **2e** (166 mg, 0.880 mmol) in THF (1.8 mL) was added nitrone **1c** (50.0 mg, 0.442 mmol). After stirring at 60 °C for 8 days, the reaction mixture was concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 4:1) to afford 5-iminoisoxazolidine **3o** (44.6 mg, 34%) as a pale yellow oil and 4-iminoisoxazolidine **4o** (29.5 mg, 22%) as a colorless oil.

3o: IR (KBr) 2966, 1456, 1365, 1041, 698 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.23 (5H, m), 5.16 (1H, d, *J* = 12.1 Hz), 5.11 (1H, d, *J* = 12.1 Hz), 4.69 (1H, d, *J* = 5.7 Hz), 3.71 (1H, dd, *J* = 9.3, 4.8 Hz), 2.88 (1H, dd, *J* = 14.4, 7.5 Hz), 2.70 (1H, ddd, *J* = 18.9, 9.6, 6.0 Hz), 2.62-2.45 (1H, m), 2.24-1.70 (4H, m), 1.63-1.46 (2H, m), 1.34 (3H, s), 1.04 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.6, 137.8, 128.2, 127.9, 127.6, 81.3, 75.9, 71.2, 69.1, 54.7, 35.9, 31.5, 28.1, 27.1, 26.6, 24.1; HRMS (EI) *m*/*z* calcd for $C_{18}H_{24}N_2O_2$ 300.1838, found 300.1843.

4o: IR (KBr) 2966, 1456, 1366, 1042, 924, 735, 698 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.24 (5H, m), 5.12 (1H, d, *J* = 12.0 Hz), 5.07 (1H, d, *J* = 12.0 Hz), 4.62 (1H, t, *J* = 5.4 Hz), 4.00 (1H, dd, *J* = 9.0, 4.5 Hz), 3.29 (1H, d, *J* = 5.4 Hz), 2.67 (2H, dd, *J* = 9.6, 5.1 Hz), 2.25-2.05 (2H, m), 1.83-1.54 (4H, m), 1.31 (3H, s), 1.04 (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.4, 137.9, 128.3, 127.9, 127.6, 81.3, 75.7, 71.5, 69.1, 58.8, 36.3, 31.2, 29.8, 27.3, 26.3, 24.1; HRMS (EI) *m*/*z* calcd for $C_{18}H_{24}N_2O_2$ 300.1838, found 300.1847.



3p and 

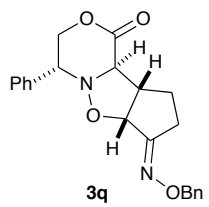
4p

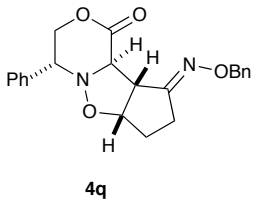
(4*R*,6a*R*,9a*S*,9b*R*)-4-phenylhexahydro-1*H*-cyclopenta[4,5]isoxazolo[3,2-*c*][1,4]oxazine-1,7(6a*H*)-dione (**3p**) and its 9-acylisomer (**4p**)

A mixture of 2-cyclopenten-1-one (**2a**) (44.0 μL, 0.520 mmol) and nitrone **1d** (50.0 mg, 0.260 mmol) was left to stand at room temperature for 5 days. The reaction mixture was purified by column chromatography on silica gel (hexane:AcOEt = 4:1) to afford 5-acylisoxazolidine **3p** (8.5 mg, 12%, unstable) as a white solid and 4-acylisoxazolidine **4p** (43.7 mg, 62%) both as a white amorphous solid.

3p: m.p. 118 °C (decomp.) (AcOEt/hexane, white solid); IR (KBr) 2926, 1751, 1742, 1456, 1400, 1232, 1040, 758 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.42-7.33 (5H, m), 4.48 (1H, dd, *J* =12.0, 3.6 Hz), 4.34-4.27 (2H, m), 4.17 (1H, dd, *J* = 9.6, 3.6 Hz), 3.99 (1H, d, *J* = 6.6 Hz), 3.82-3.74 (1H, m), 2.58 (1H, dt, *J* = 18.0, 10.2 Hz), 2.43-2.34 (1H,m), 2.33-2.25 (2H, m); ^{13}C NMR (150 MHz, $CDCl_3$) δ 212.2, 168.7, 134.6, 129.0, 128.8, 127.4, 79.4, 69.4, 68.1, 61.6, 48.1, 34.6, 23.2; HRMS (ESI) *m*/*z* calcd for $C_{15}H_{15}NO_4$ $[M+Na]^+$ 296.0899, found. 296.0877

4p: IR (KBr) 2941, 1771, 1456, 1394, 1227, 1207, 1153, 1097, 1045, 758, 698 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.45-7.37 (4H, m), 7.38-7.33 (1H, m), 4.97-4.92 (1H, m), 4.52 (1H, d, *J* = 3.0 Hz), 4.34 (1H, dd, *J* =18.0, 9.6 Hz), 4.23 (2H, dd, *J* =18.0, 10.8 Hz), 3.67-3.62 (1H, m), 2.57-2.46 (1H, m), 2.45-2.36 (1H, m), 2.25-2.14 (2H, m); ^{13}C NMR (150 MHz, $CDCl_3$) δ 215.3, 168.6, 135.5, 128.9, 128.8, 127.3, 77.9, 69.2, 66.4, 62.7, 60.0, 35.0, 24.5; HRMS (EI) *m*/*z* calcd for $C_{15}H_{15}NO_4$ 273.1001, found 273.0998.



3q and 

4q

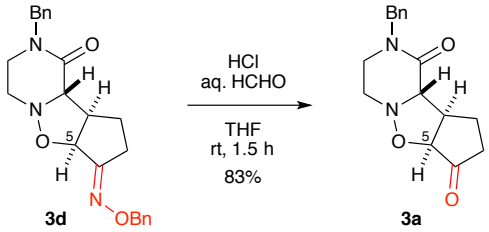
(4*R*,6a*R*,9a*S*,9b*R*,*E*)-7-[(benzyloxy)imino]-4-phenyloctahydro-1*H*-cyclopenta[4,5]isoxazolo[3,2-*c*][1,4]oxazin-1-one (**3q**) and its 9-acylisomer (**4q**)

A mixture of α,β-unsaturated oxime **2e** (97.0 mg, 0.520 mmol) and nitrone **1d** (50.0 mg, 0.260 mmol) was left to stand at room temperature for 6 days. The reaction

mixture was purified by column chromatography on silica gel (hexane:AcOEt = 10:1 to 4:1) and PTLC ($CHCl_3$:AcOEt = 49:1) to afford 5-iminoisoxazolidine **3q** (60.0 mg, 61%) and 4-iminoisoxazolidine **4q** (11.0 mg, 11%) both as a white solid.

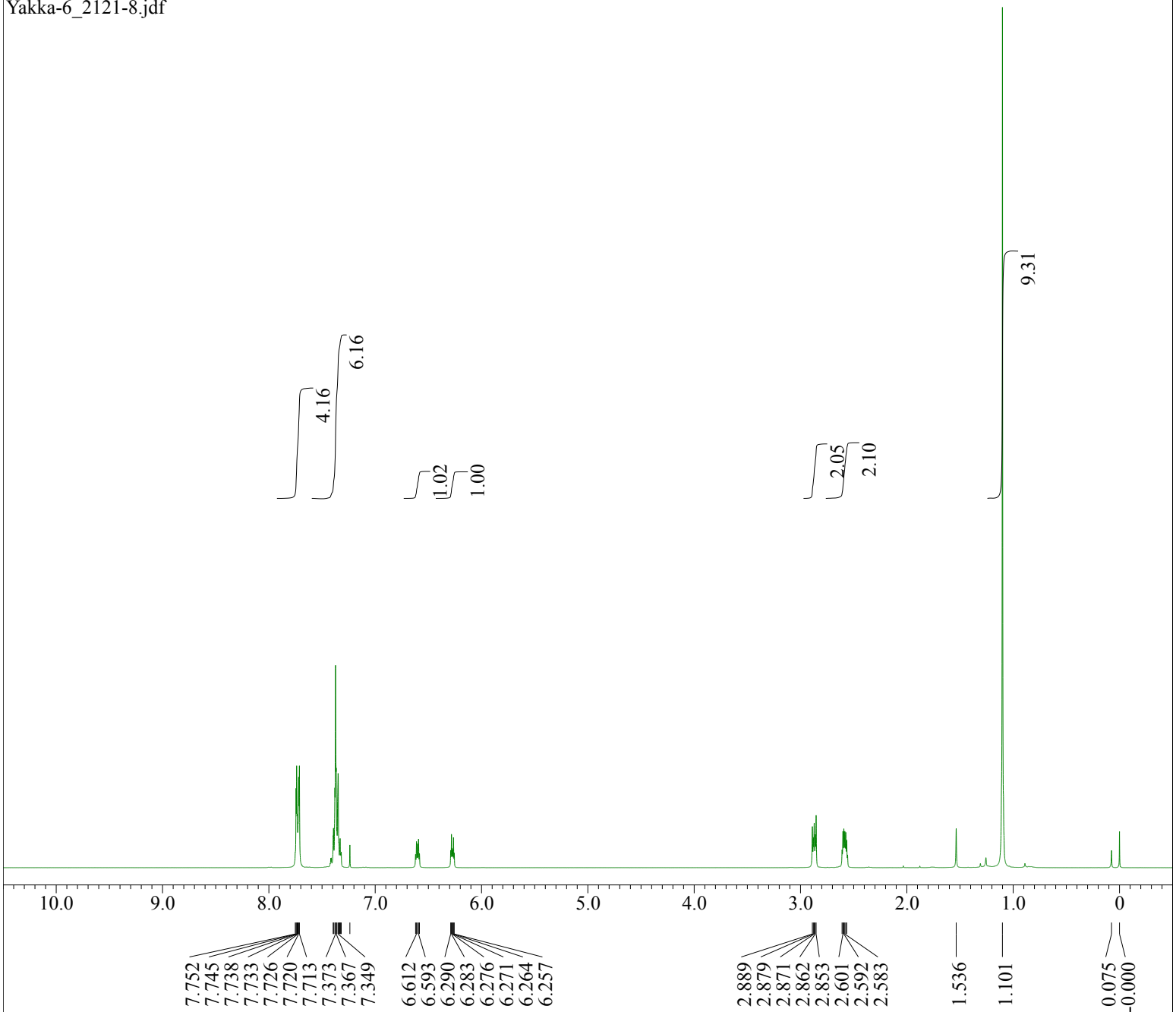
3q: m.p. 175.5-177.0 °C; IR (KBr) 2930, 1744, 1456, 1230, 1047, 1003, 976, 865, 758, 697 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.44-7.24 (10H, m), 5.10 (1H, d, *J* = 12.0 Hz), 5.07 (1H, d, *J* = 12.0 Hz), 4.92 (1H, d, *J* = 7.2 Hz), 4.35 (1H, dd, *J* = 12.0, 3.6 Hz), 4.23 (1H, dd, *J* = 12.0, 10.2 Hz), 4.12 (1H, dd, *J* = 10.2, 3.6 Hz), 3.99 (1H, d, *J* = 7.8 Hz), 3.62-3.54 (1H, m), 2.90 (1H, ddd, *J* = 18.0, 8.4, 1.2 Hz), 2.54-2.41 (1H, m), 2.26-2.17 (1H, m), 2.10-1.99 (1H, m); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.9, 161.0, 137.4, 134.7, 128.9, 128.8, 128.4, 128.1, 127.9, 127.7, 80.9, 76.3 , 70.2, 68.7, 61.3, 51.0, 26.7, 24.3; HRMS (EI) *m/z* calcd for $C_{22}H_{22}N_2O_4$ 378.1580, found 378.1592.

4q: m.p. 119.0-120.0 °C (AcOEt/hexane, colorless prism); IR (KBr) 2904, 1750, 1541, 1508, 1458, 1053, 696 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.45-7.32 (9H, m), 7.33-7.28 (1H, m), 5.17 (1H, d, *J* = 12.0 Hz), 5.14 (1H, d, *J* = 12.0 Hz), 4.78 (1H, t, *J* = 5.1 Hz), 4.46 (1H, d, *J* = 3.6 Hz), 4.36-4.28 (1H, m), 4.25-4.16 (2H, m), 4.06-4.01 (1H, m), 2.74 (1H, ddd, *J* = 18.0, 9.6, 3.0 Hz), 2.64-2.53 (1H, m), 2.07-1.98 (1H, m), 1.95-1.85 (1H, m); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.7, 164.2, 137.8, 135.6, 128.9, 128.7, 128.3, 128.2, 127.8, 127.4, 79.8, 76.2, 69.3, 69.2, 62.5, 55.5, 27.5, 25.6; HRMS (EI) *m/z* calcd for $C_{22}H_{22}N_2O_4$ 378.1580, found 378.1595.



To a solution of 5-iminoisoxazolidine **3d** (30.0 mg, 76.6 μmol) in THF (1.5 mL) were added 35% HCHO aq. (31 μL) and conc. HCl (36%, 24.5 μL). After stirred at room temperature for 1.5 h, the reaction was quenched by adding iced water. The products were extracted with CH_2Cl_2 (×4), washed with water, dried (Na_2SO_4) and concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography (hexane:AcOEt = 1:4) to afford 5-acylisoxazolidine **3a** (18.2 mg, 83%) as a colorless oil.

Yakka-6_2121-8.jdf



X : parts per Million : 1H

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Experiment     = zg30
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Solvent        = CDCl3
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Revision_Time  = 15-NOV-2016 17:58:41
Current_Time   = 15-NOV-2016 17:59:17

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Dim_Units      = [ppm]
Dimensions     = X
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X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.36[K]
X_Points       = 32768
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Filter_Factor  = 3232
Scans          = 16
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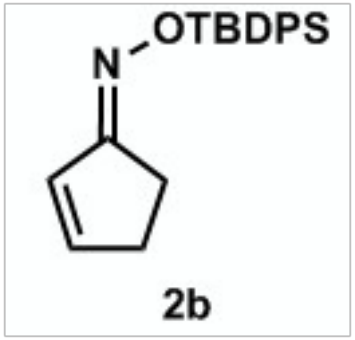

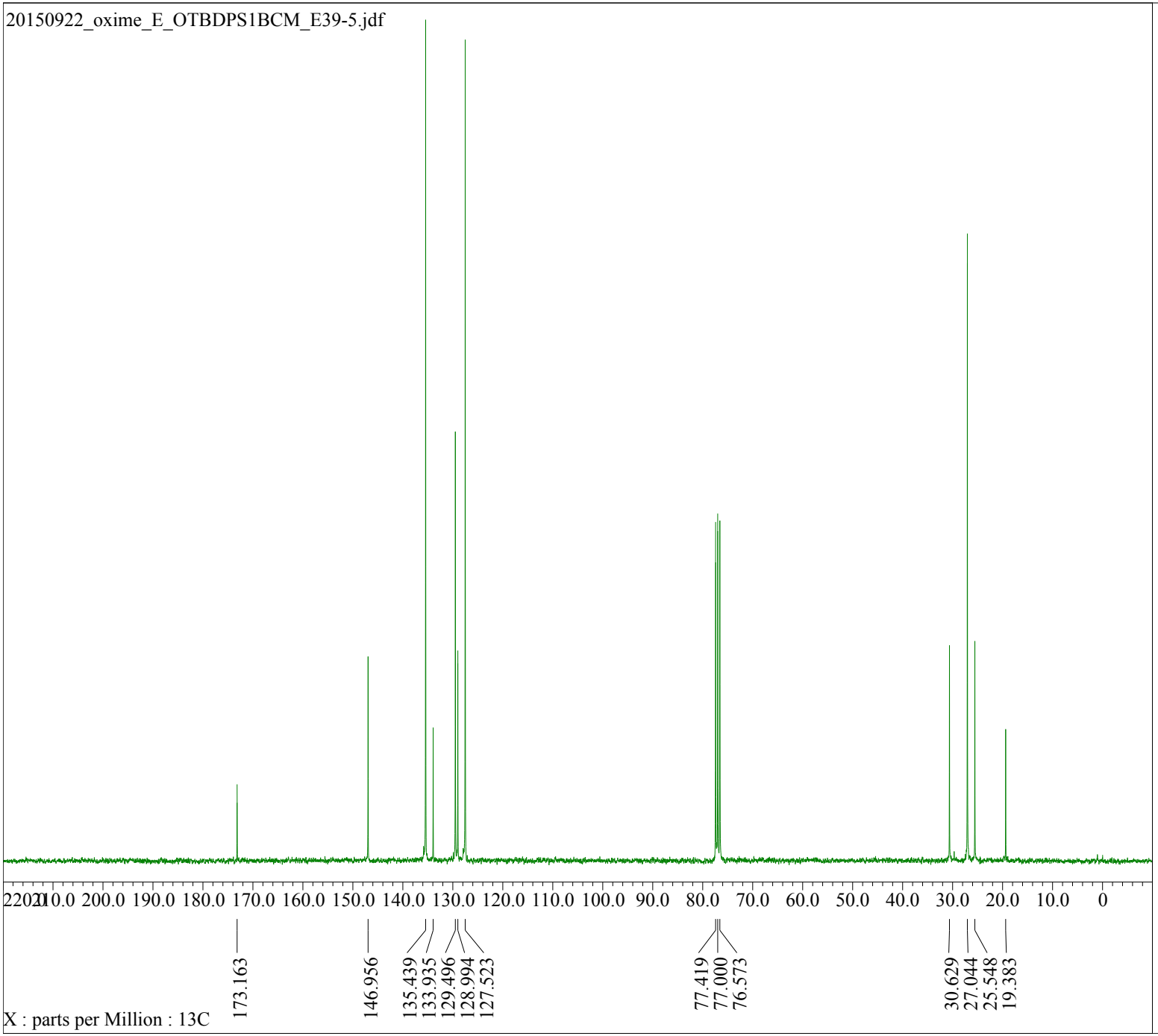


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Filename          = 20150922_oxime_E_OTBDPS1BC
Author            = 橋本普光
Experiment        = BCM
Solvent           = CDCL3
Creation_Time     = 15-NOV-2016 18:19:38
Revision_Time     = 15-NOV-2016 18:22:42
Current_Time      = 15-NOV-2016 18:27:08

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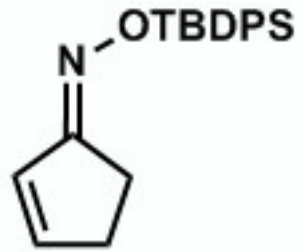
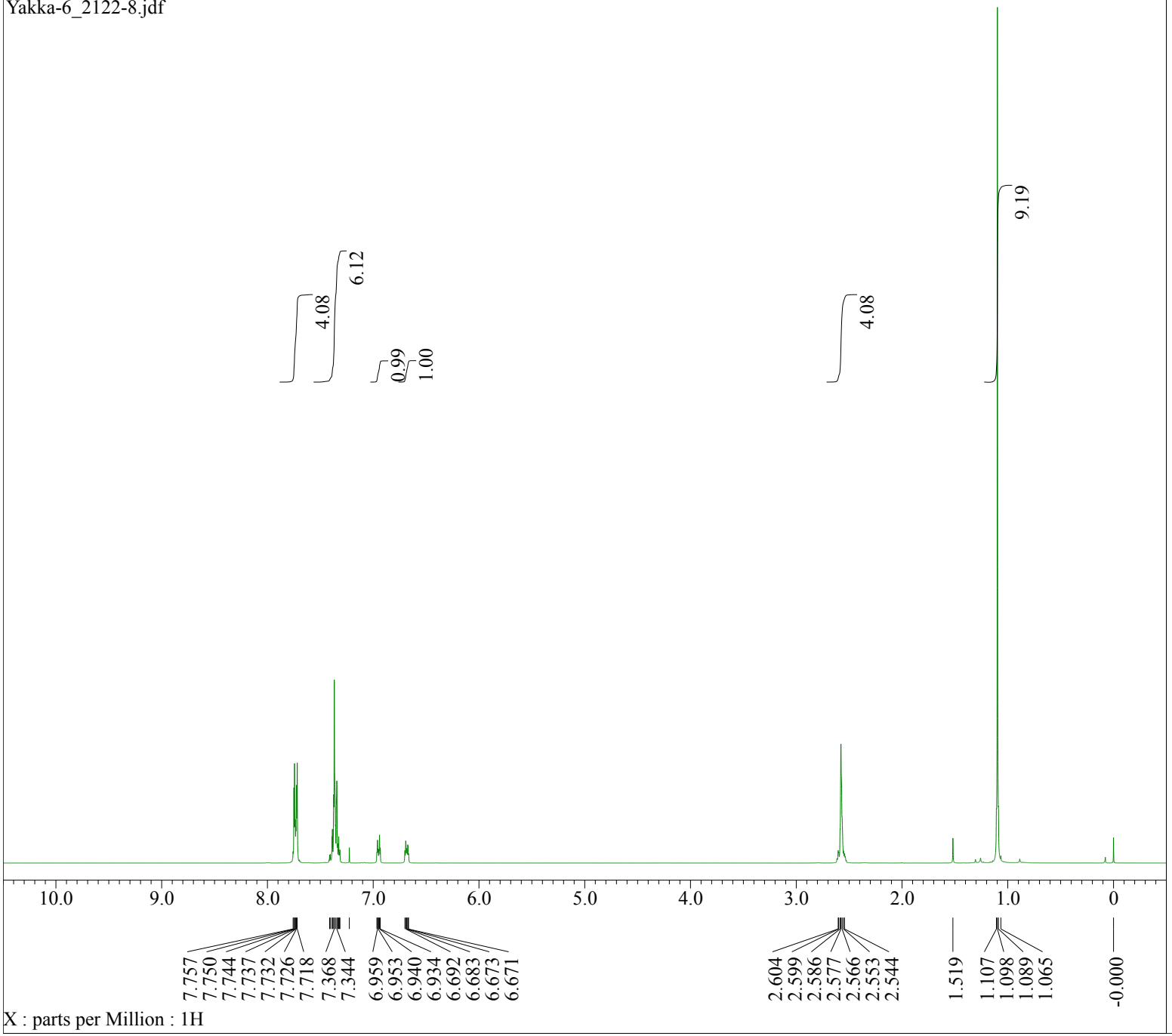


2b

Yakka-6_2122-8.jdf



X : parts per Million : 1H

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Author         = Administrator
Experiment     = zg30
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Solvent        = CDCl3
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Revision_Time  = 15-NOV-2016 18:35:29
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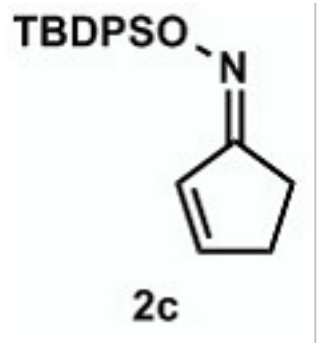
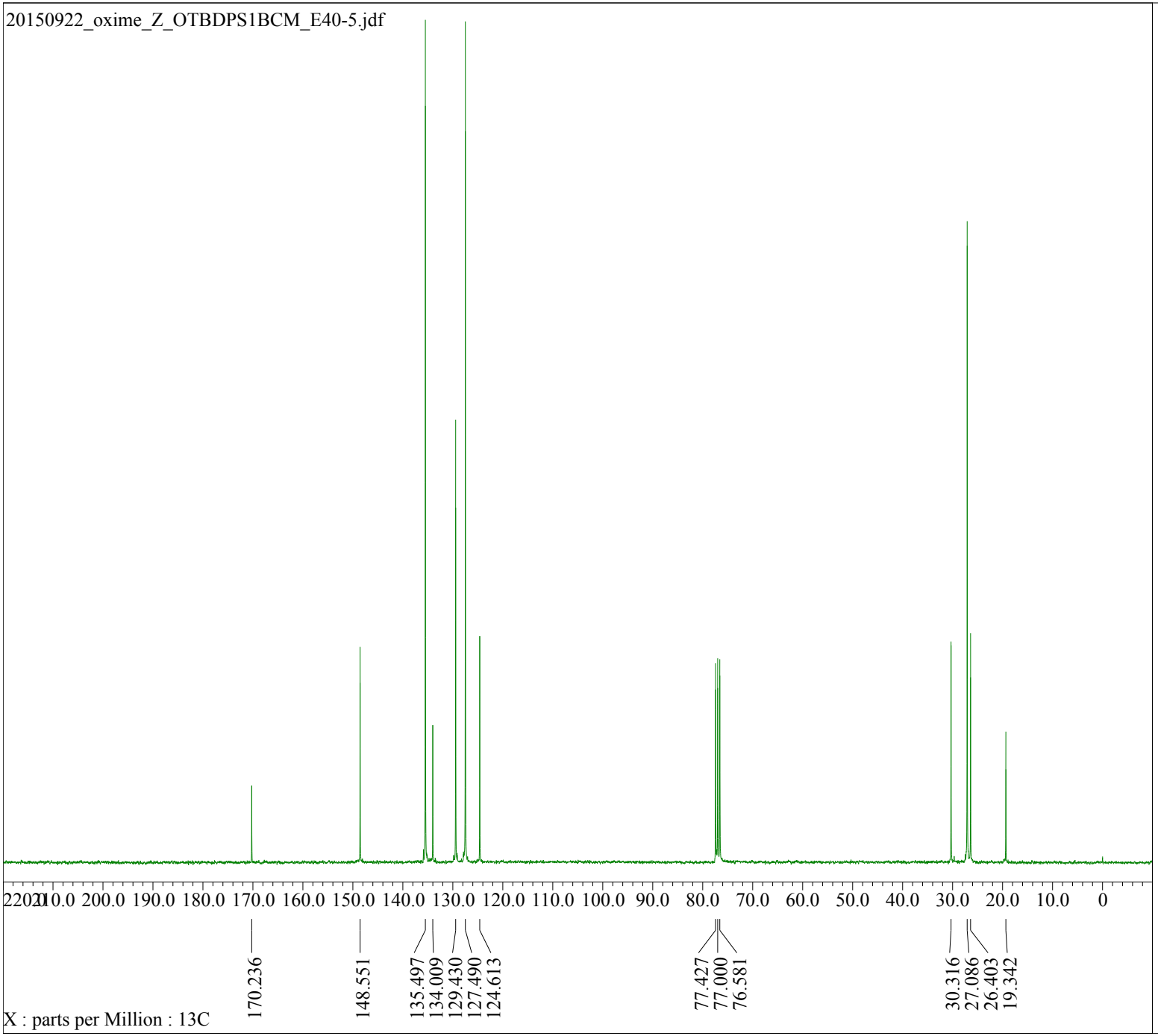


2c

20150922_oxime_Z_OTBDPS1BCM_E40-5.jdf



X : parts per Million : 13C

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Filename          = 20150922_oxime_Z_OTBDPS1BC
Author            = 橋本普光
Experiment        = BCM
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Revision_Time     = 15-NOV-2016 18:30:45
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Data_Format       = 1D COMPLEX
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Dimensions        = X
Spectrometer      = ALICE_NMR

X_Freq            = 75.57583695[MHz]
X_Offset          = 0[Hz]
X_Sweep           = 20.35623437[kHz]

Relaxation_Delay = 1.38999999
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X_Prescans        = 1
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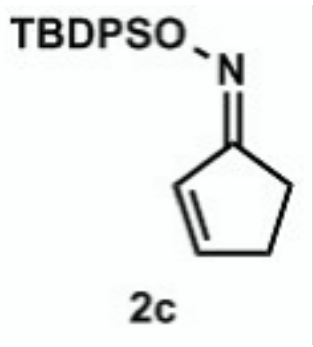
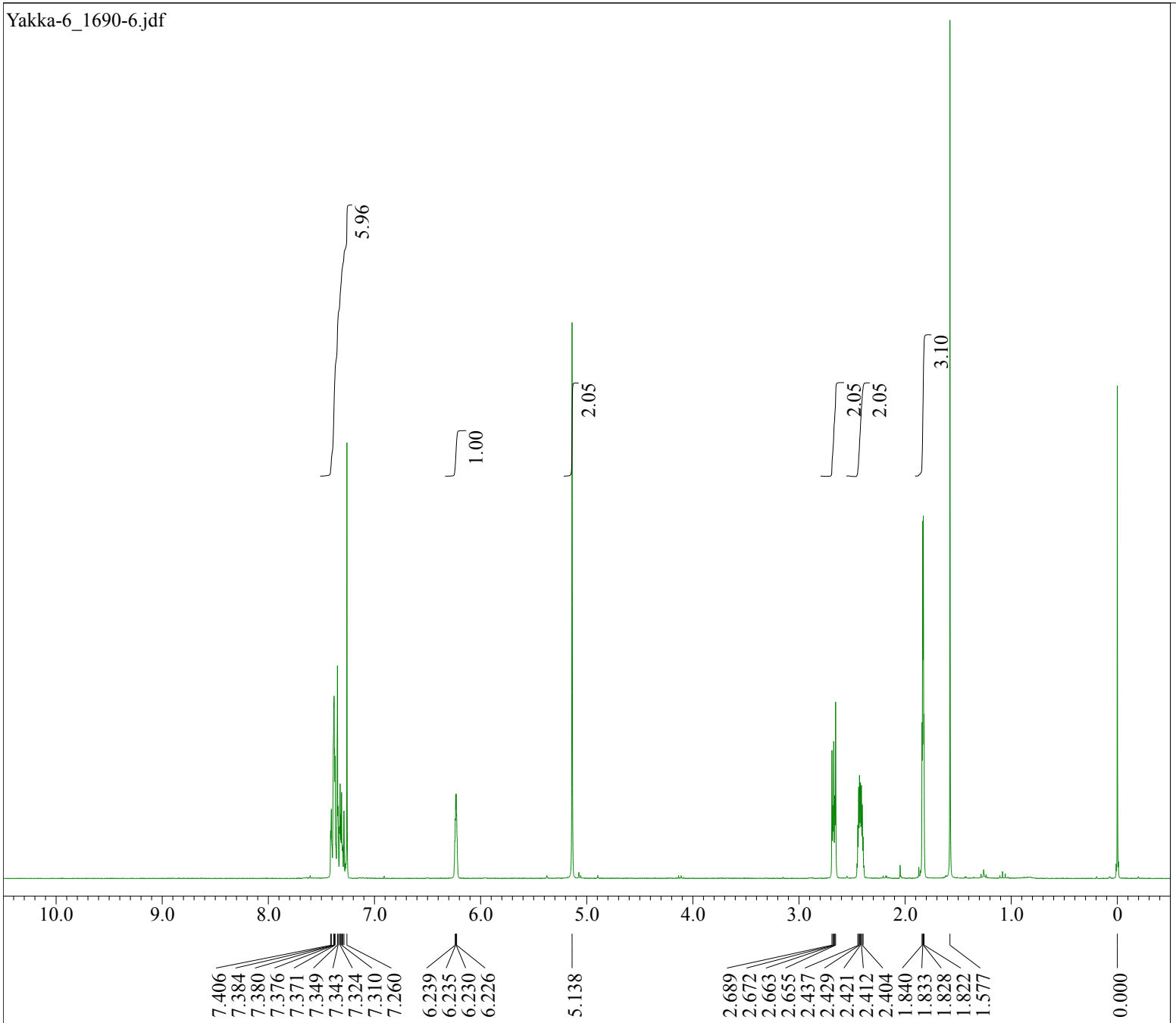


2c

Yakka-6_1690-6.jdf



X : parts per Million : 1H

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Experiment     = zg30
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Revision_Time  = 15-NOV-2016 19:01:46
Current_Time   = 15-NOV-2016 19:02:11

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Spectrometer   = BRUKER_DMX_NMR

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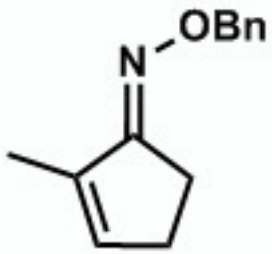
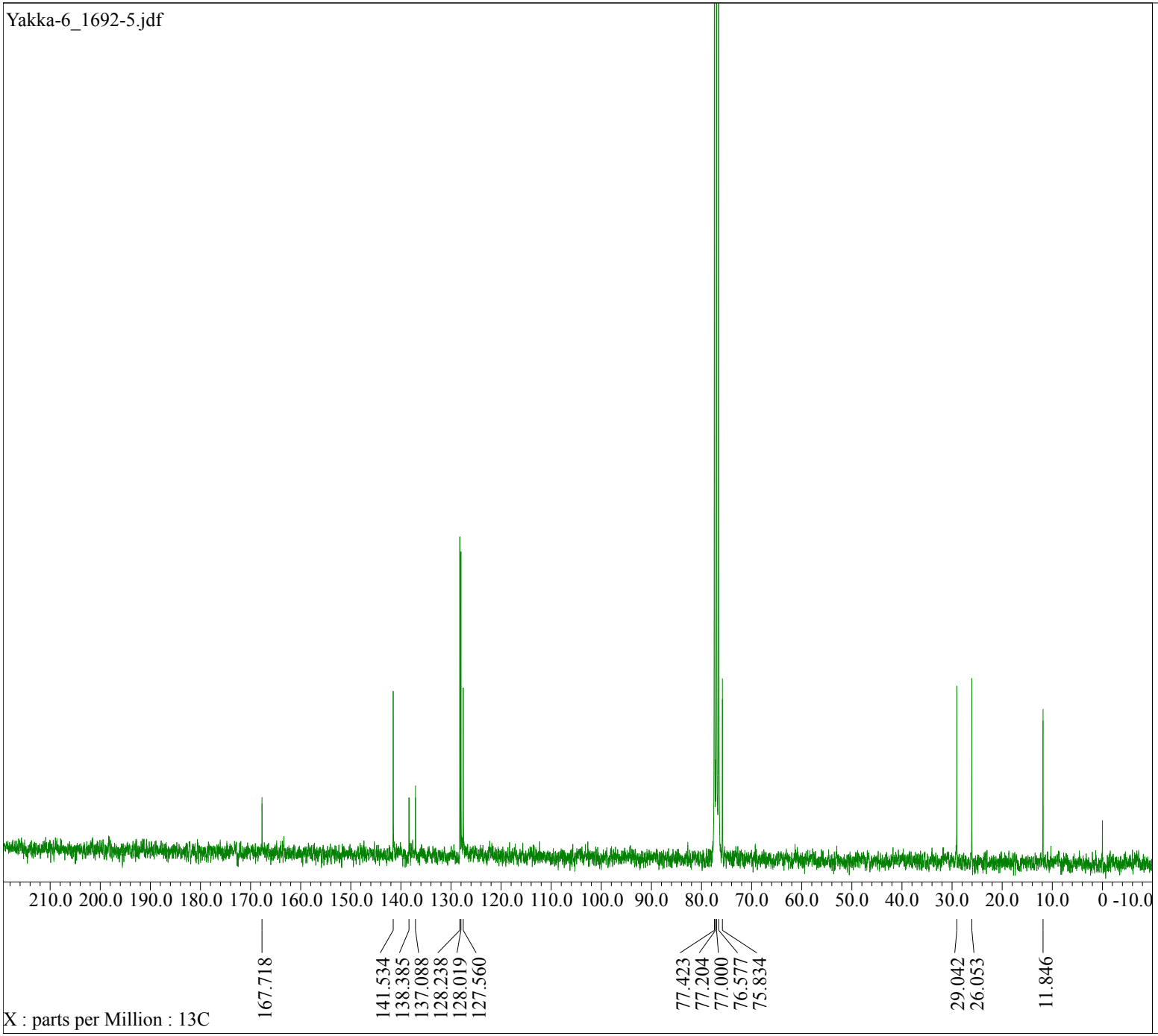


2g

Yakka-6_1692-5.jdf



X : parts per Million : 13C

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Experiment     = zgpg30
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Revision_Time  = 15-NOV-2016 19:05:28
Current_Time   = 15-NOV-2016 19:06:01

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Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

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X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.86[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
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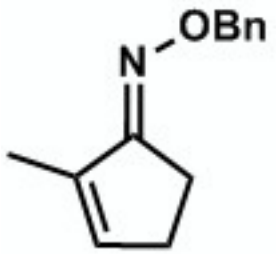
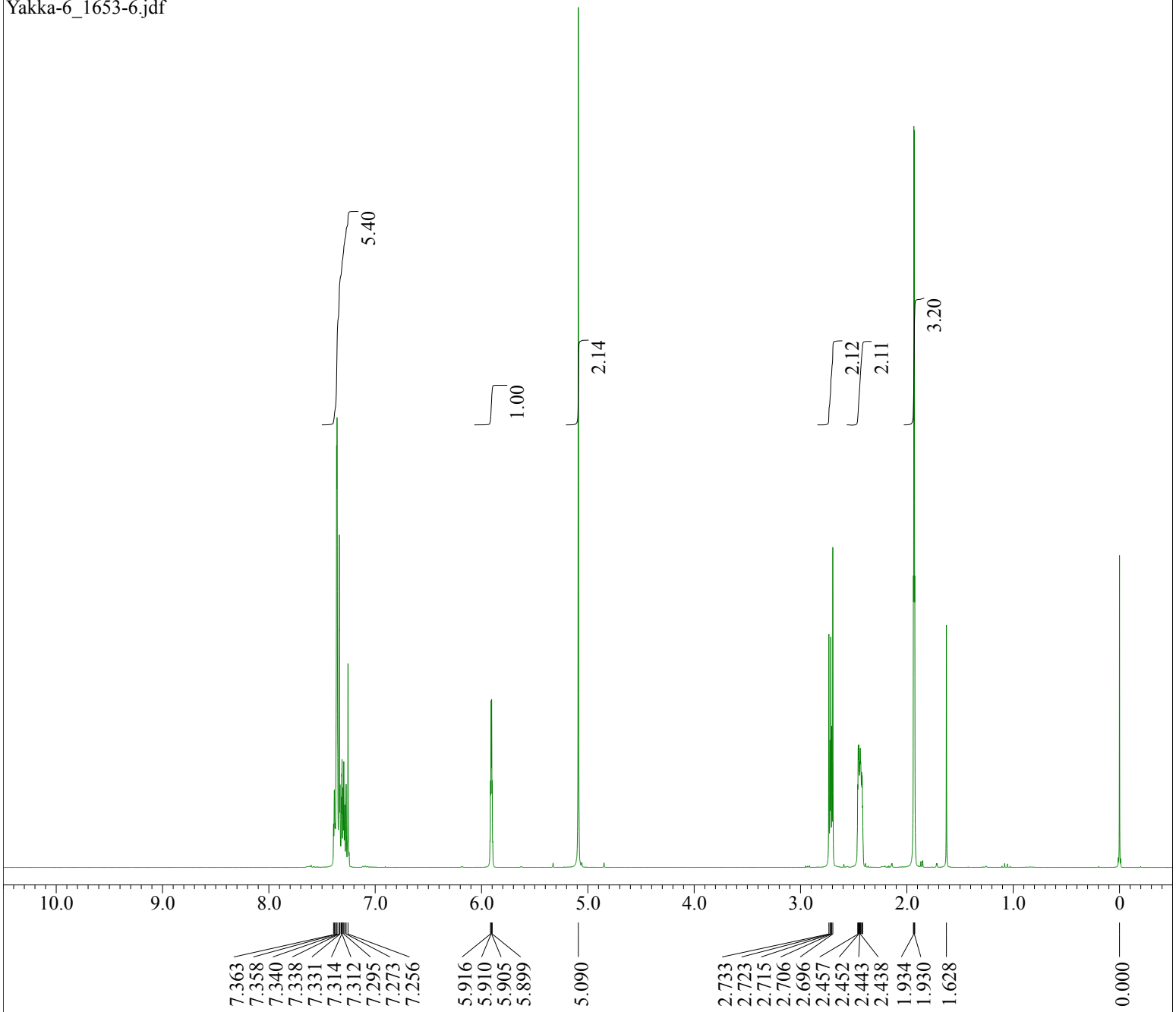


2g

Yakka-6_1653-6.jdf



X : parts per Million : 1H

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Experiment     = zg30
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Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.86[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
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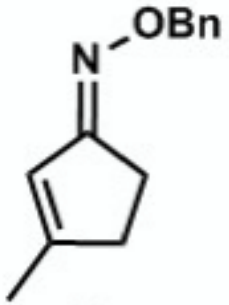
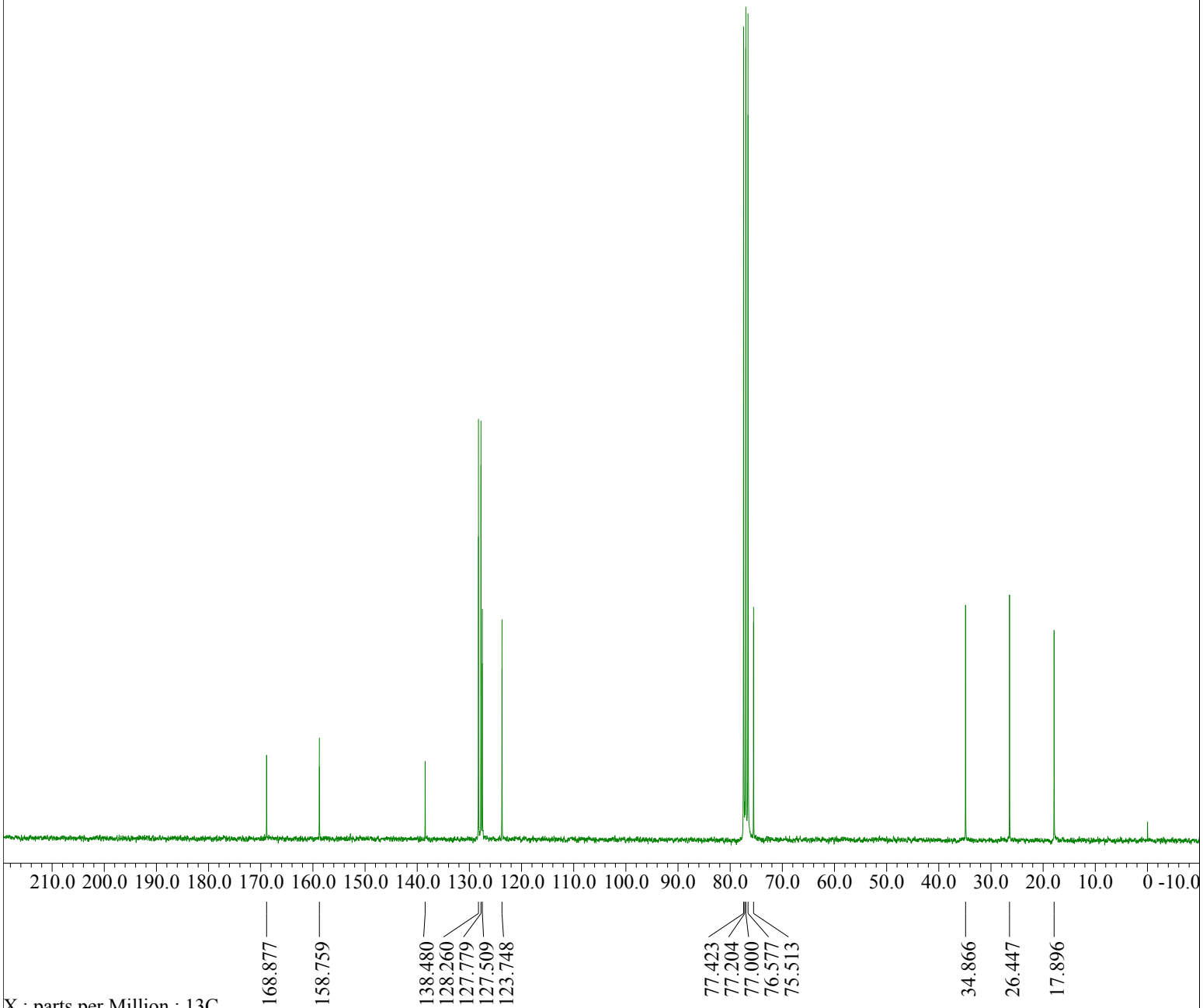


2i

Yakka-6_1655-3.jdf



X : parts per Million : 13C

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Experiment     = zgpg30
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Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

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X_Offset       = 7.54630085[kHz]
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Temp_Get       = 296.76[K]
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Filter_Factor  = 1109
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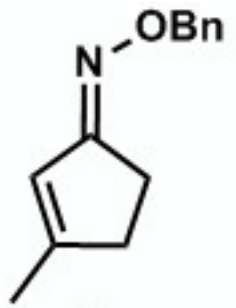
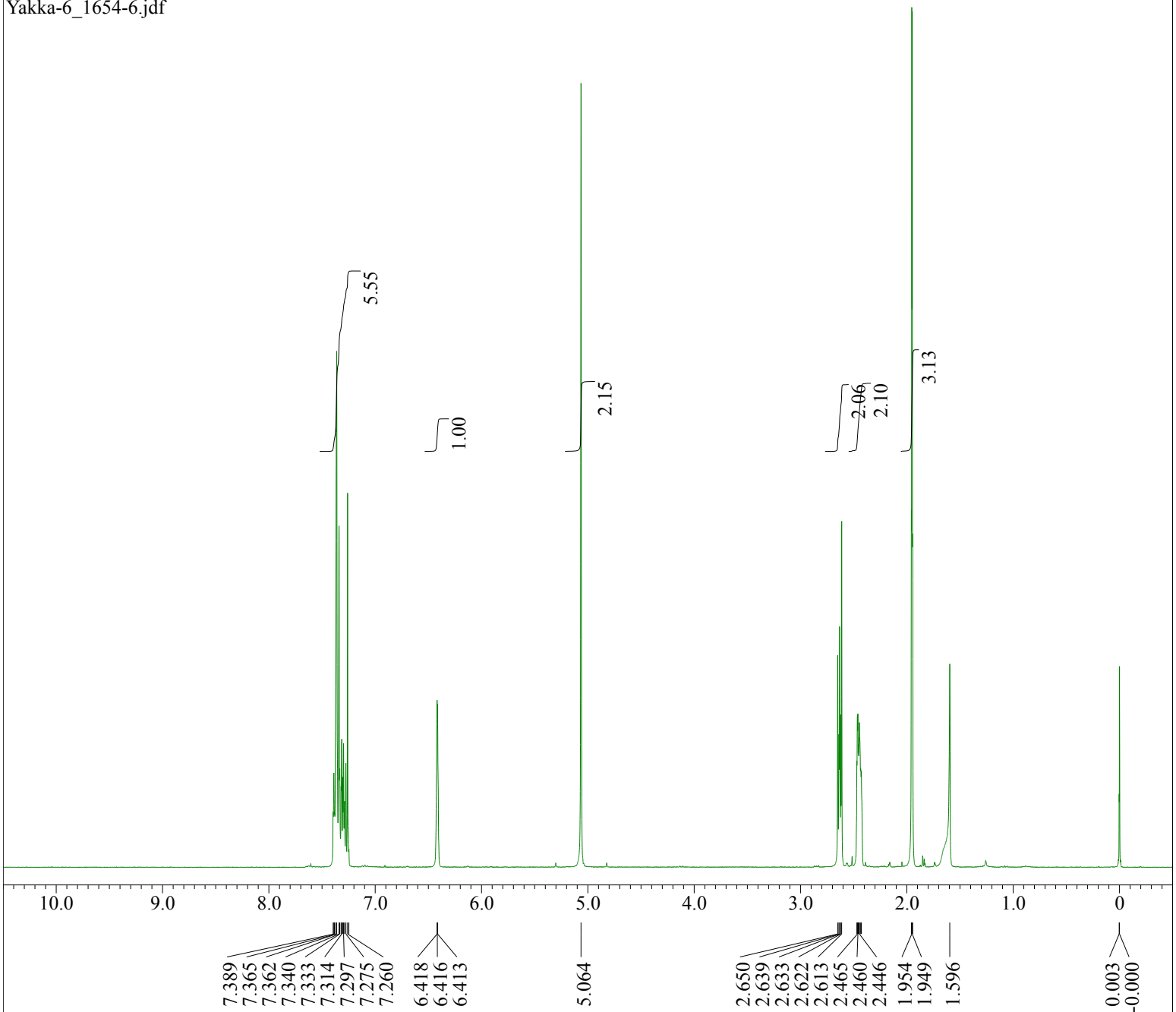


2i

Yakka-6_1654-6.jdf



X : parts per Million : 1H

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Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.86[K]
X_Points       = 32768
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Filter_Factor  = 3232
Scans          = 16
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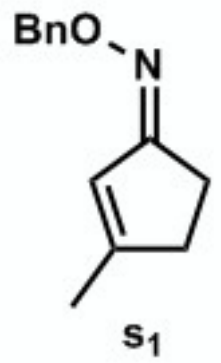
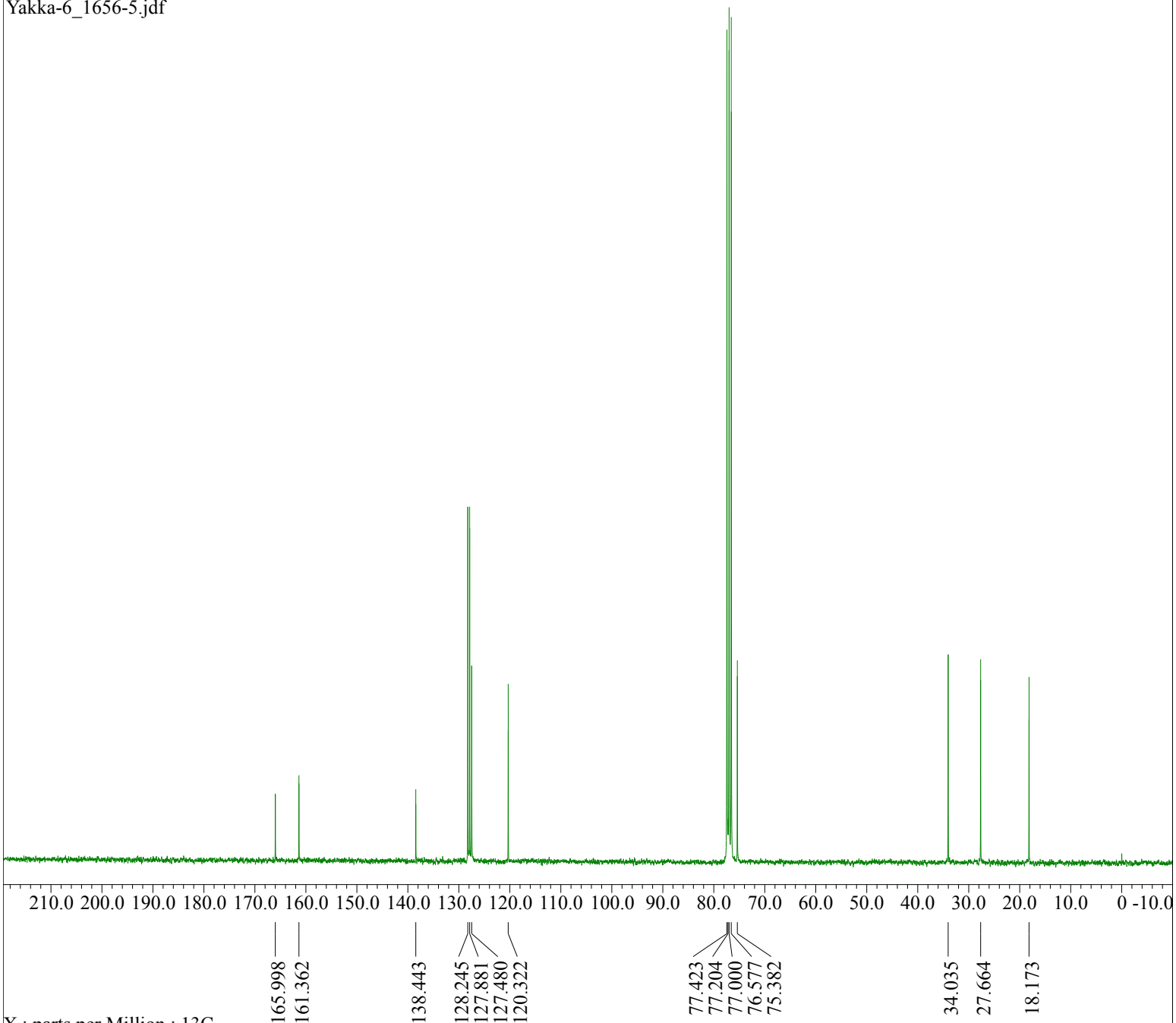


s_1

Yakka-6_1656-5.jdf



X : parts per Million : 13C

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Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
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Revision_Time  = 15-NOV-2016 18:57:03
Current_Time   = 15-NOV-2016 18:57:35

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Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.86[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 4096
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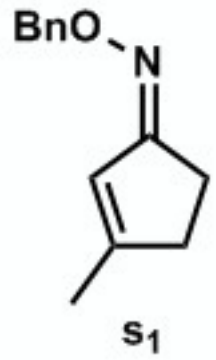
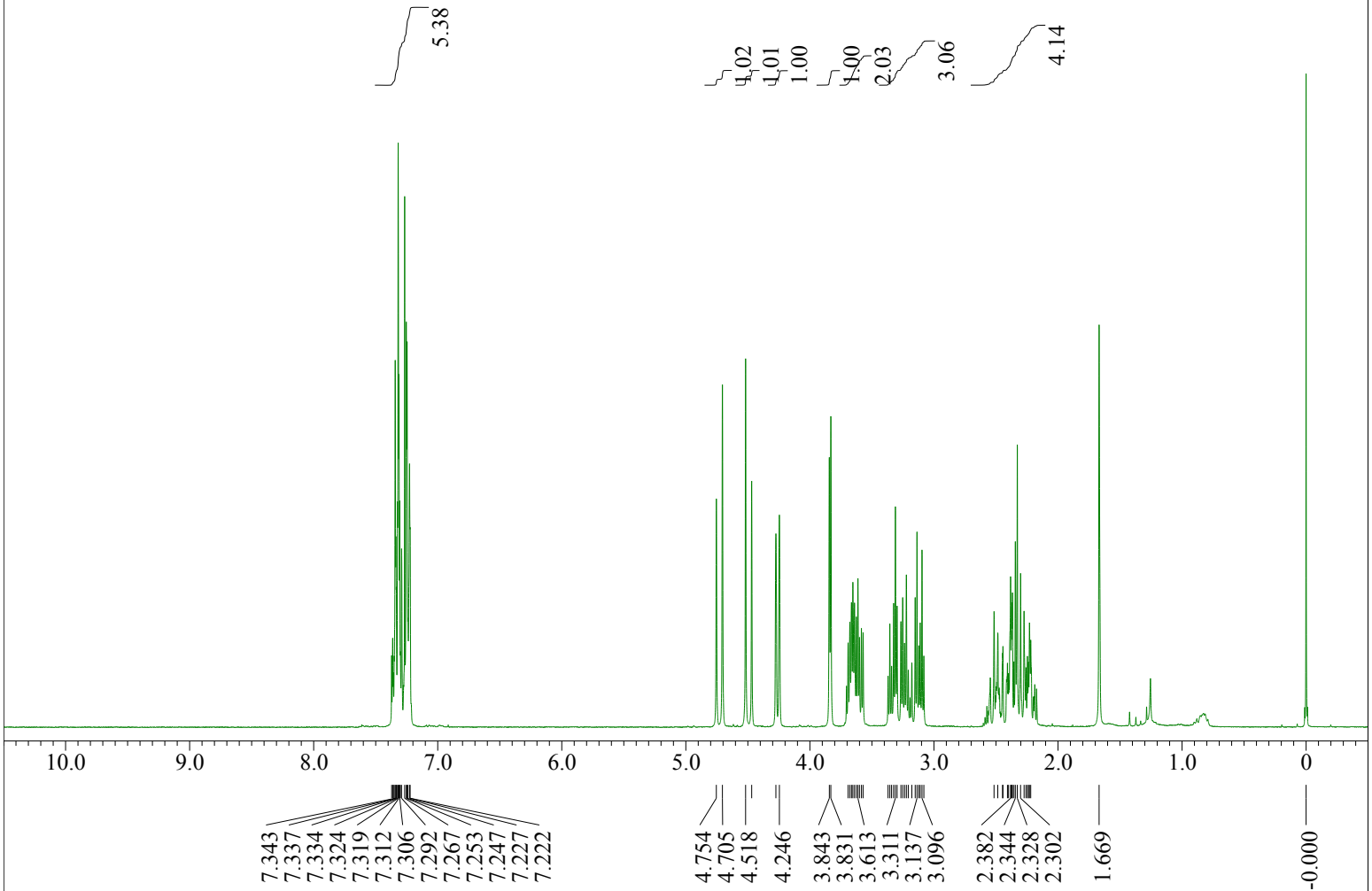


S_1

Yakka-6_2126-9.jdf



X : parts per Million : 1H

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Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
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Revision_Time  = 29-SEP-2016 19:49:00
Current_Time   = 29-SEP-2016 19:50:14

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.36[K]
X_Points       = 32768
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Filter_Factor  = 3232
Scans          = 16
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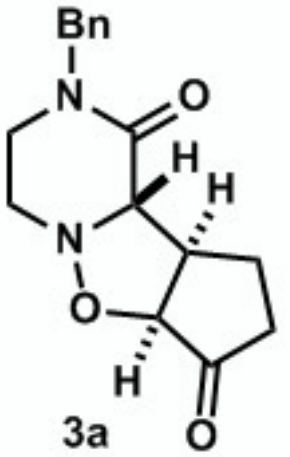
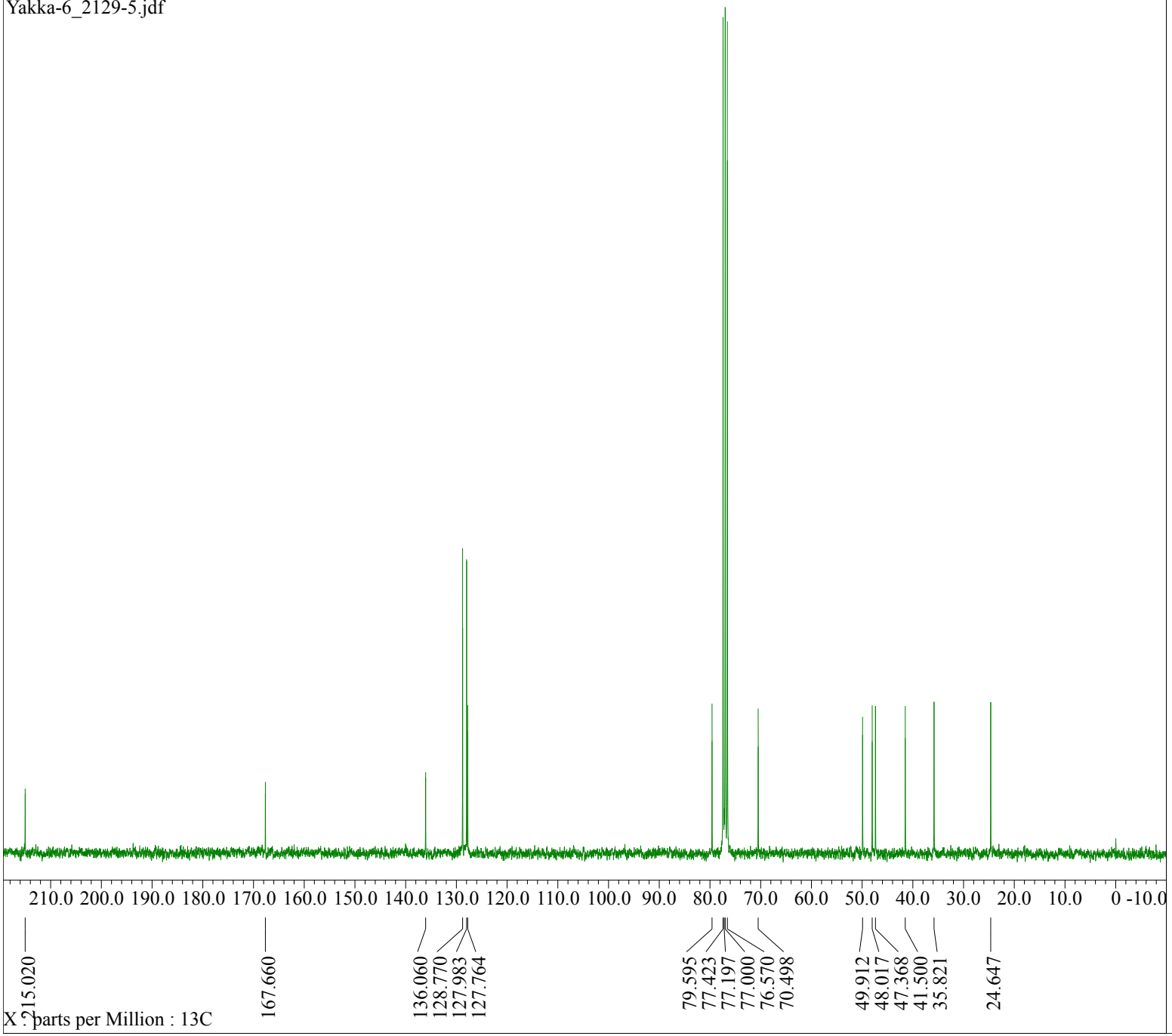


Yakka-6_2129-5.jdf

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Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
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Revision_Time  = 29-SEP-2016 19:56:01
Current_Time   = 29-SEP-2016 19:58:13

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.76[K]
X_Points       = 32768
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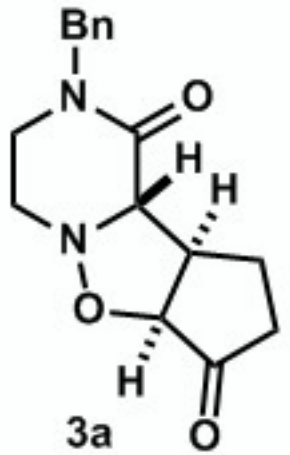
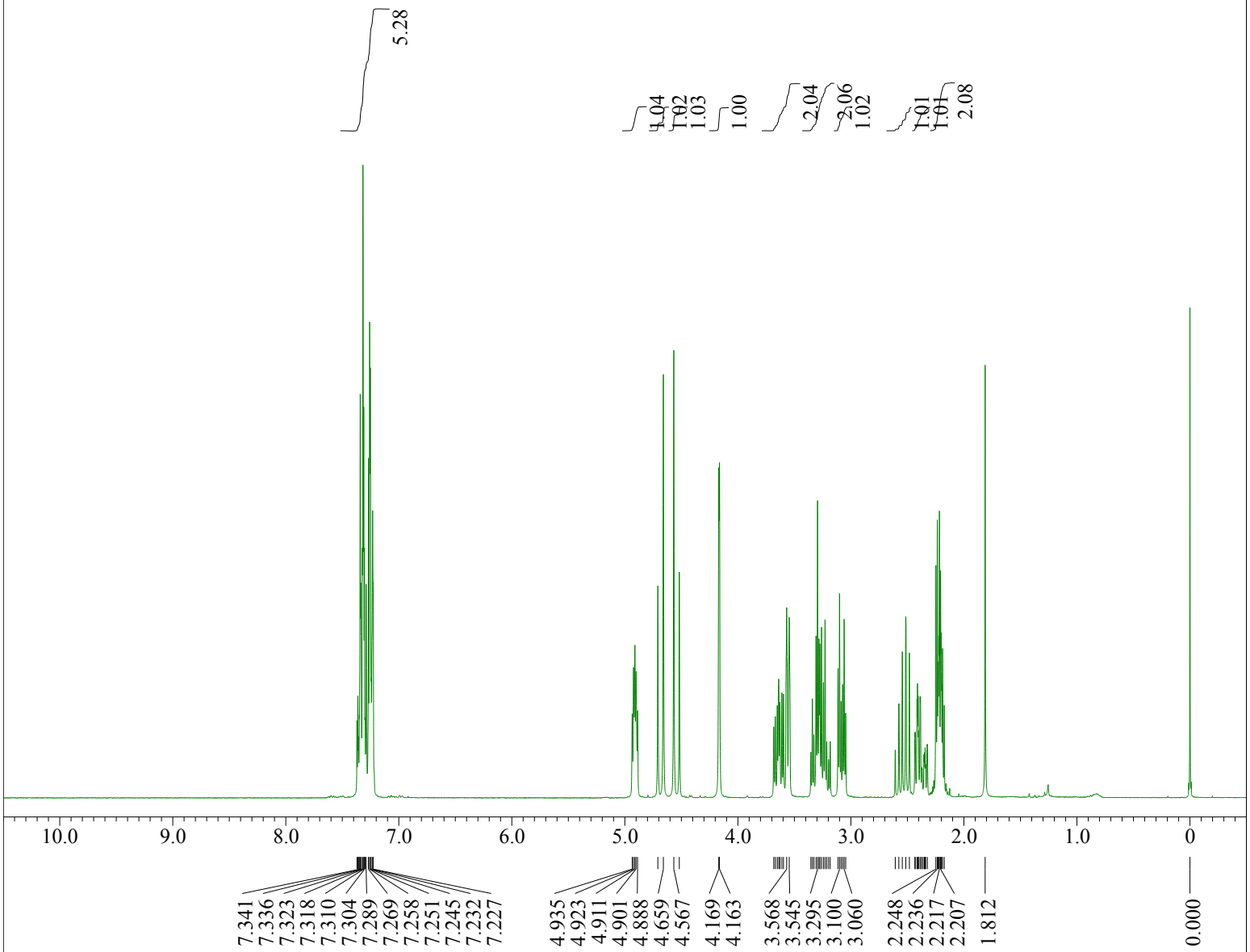


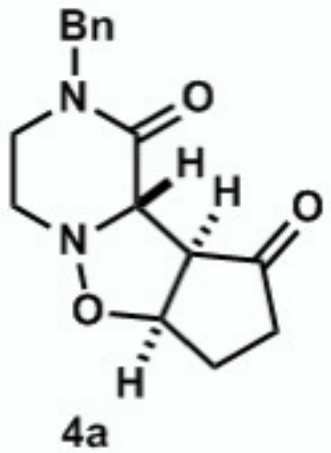
3a

Yakka-6_2133-7.jdf



X : parts per Million : 1H

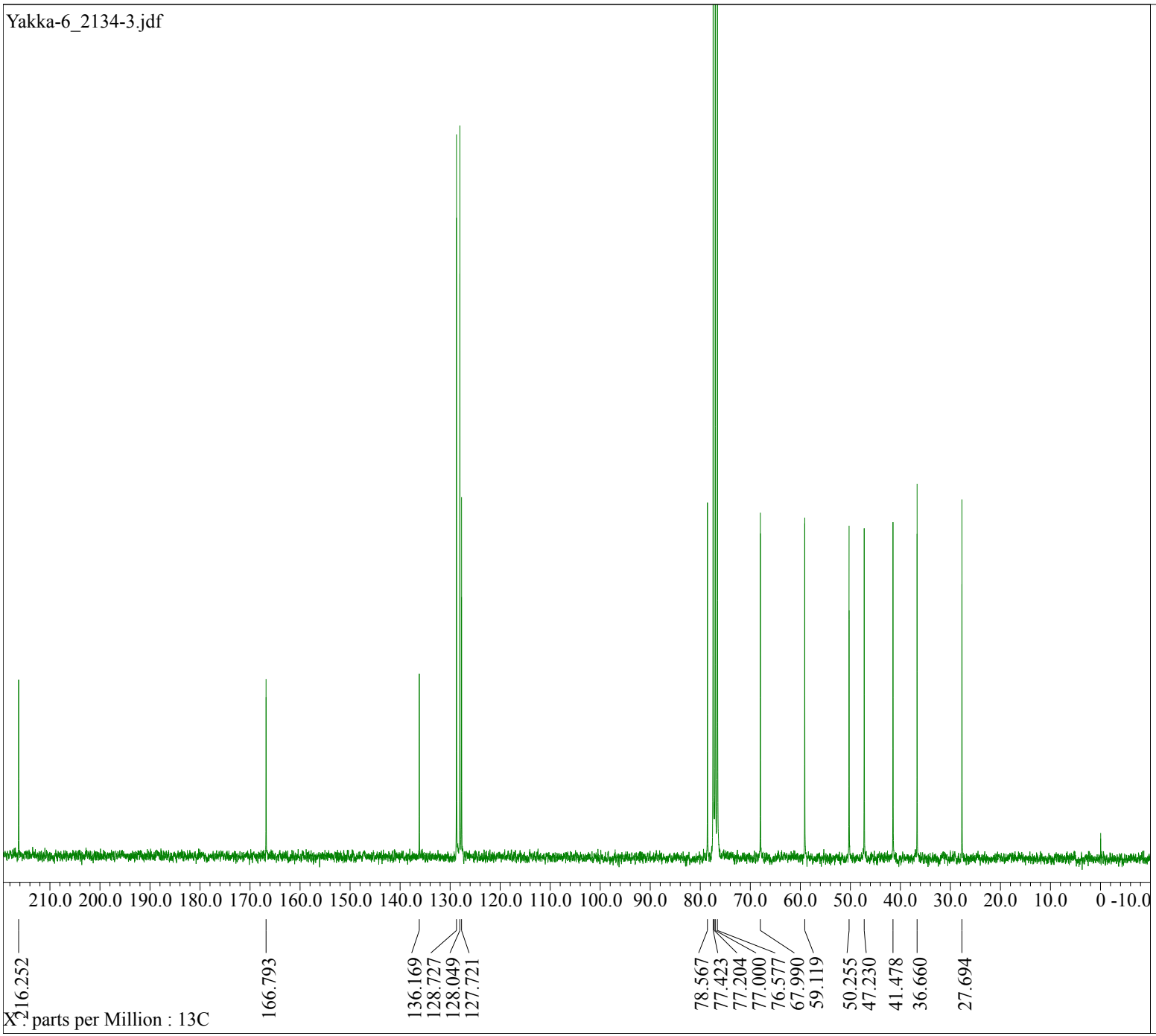
```
Filename       = Yakka-6_2133-7.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 29-SEP-2016 21:02:08
Revision_Time  = 29-SEP-2016 21:08:01
Current_Time   = 29-SEP-2016 21:08:18

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.26[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```

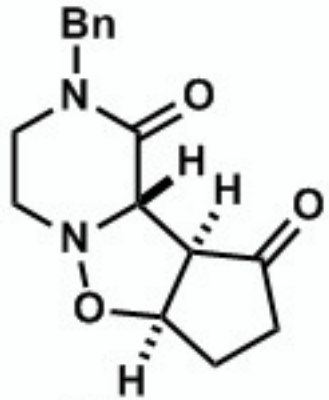


4a

Yakka-6_2134-3.jdf

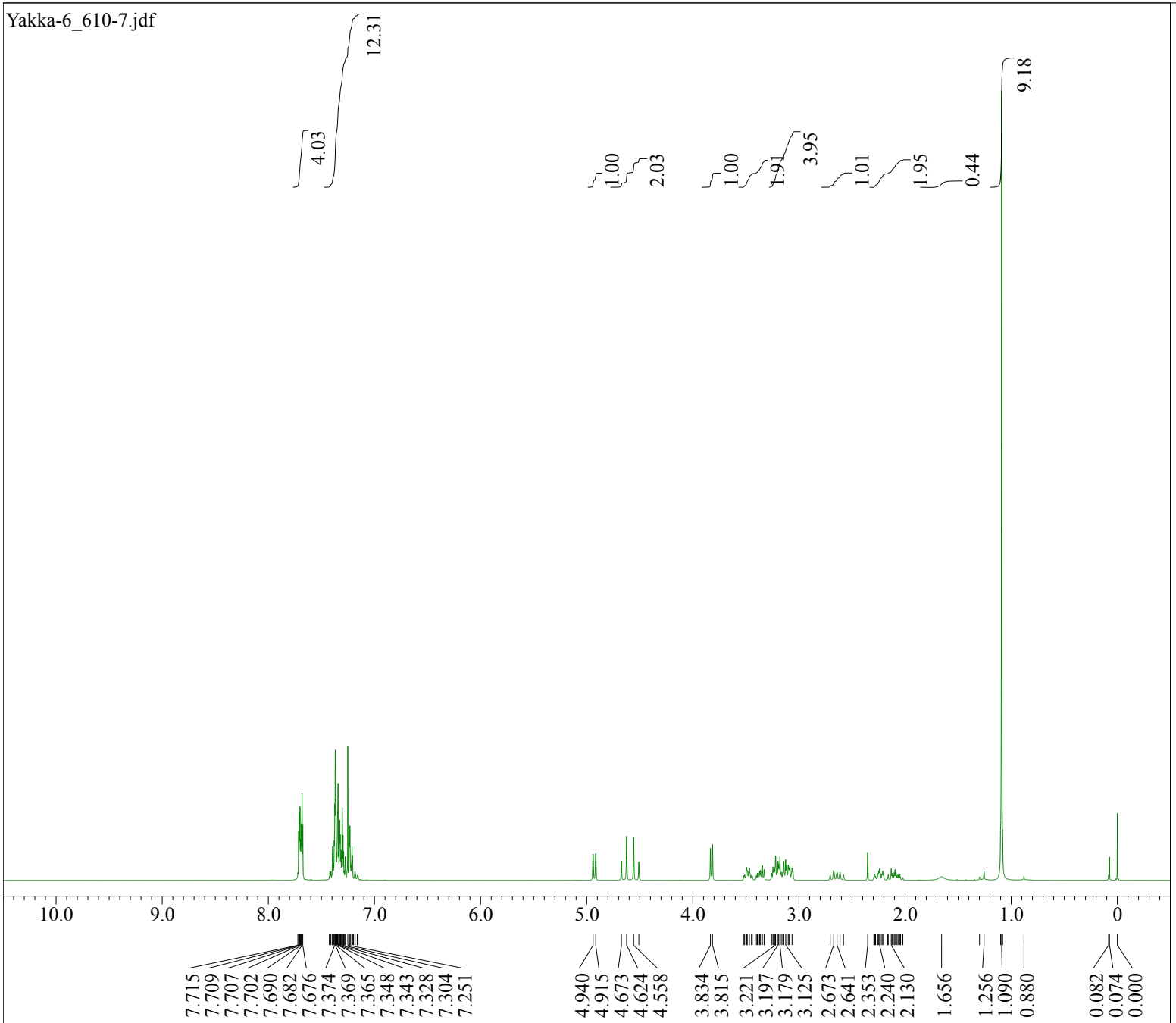
```
Filename       = Yakka-6_2134-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 29-SEP-2016 21:11:07
Revision_Time  = 29-SEP-2016 21:11:50
Current_Time   = 29-SEP-2016 21:12:50

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.76[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 2024
```





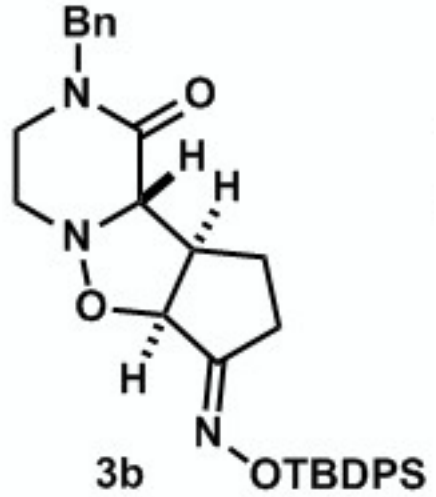
4a

Yakka-6_610-7.jdf



X : parts per Million : 1H

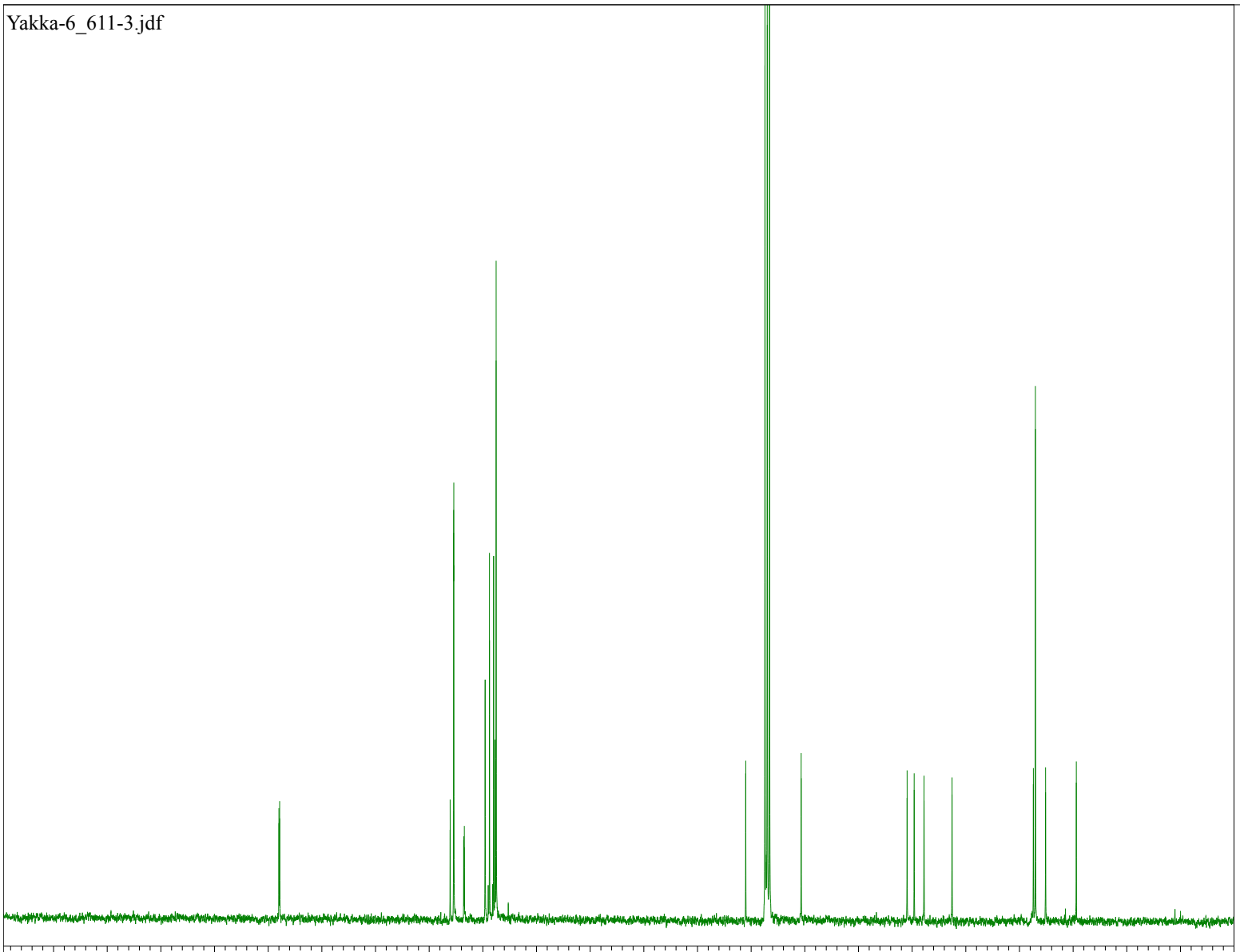
```
Filename       = Yakka-6_610-7.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 29-SEP-2016 21:14:40
Revision_Time  = 29-SEP-2016 21:20:27
Current_Time   = 29-SEP-2016 21:21:23

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.86[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```

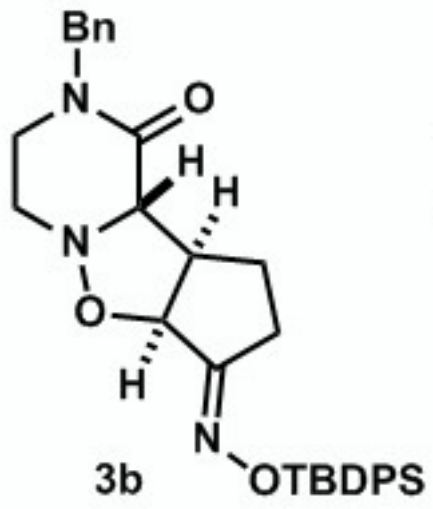


3b

Yakka-6_611-3.jdf

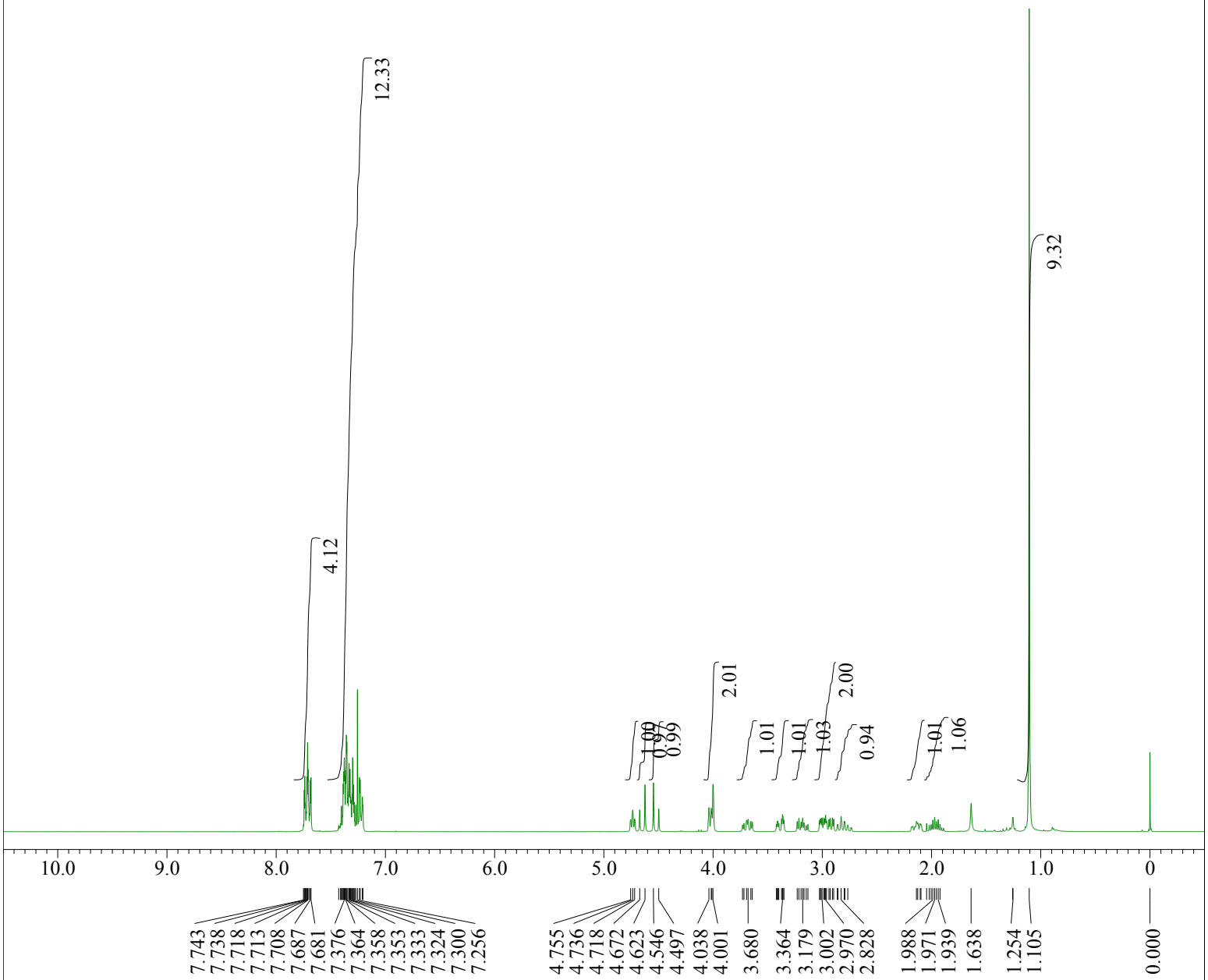
```
Filename       = Yakka-6_611-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 29-SEP-2016 21:22:13
Revision_Time  = 29-SEP-2016 21:23:12
Current_Time   = 29-SEP-2016 21:24:17

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.66[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 2048
```





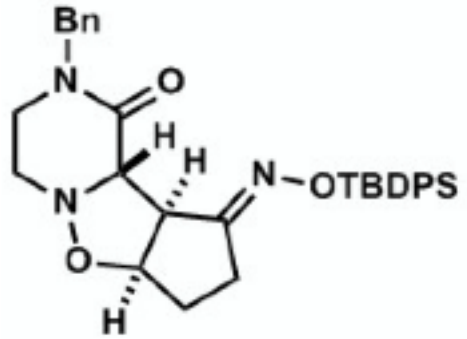
3b

Yakka-6_1606-7.jdf



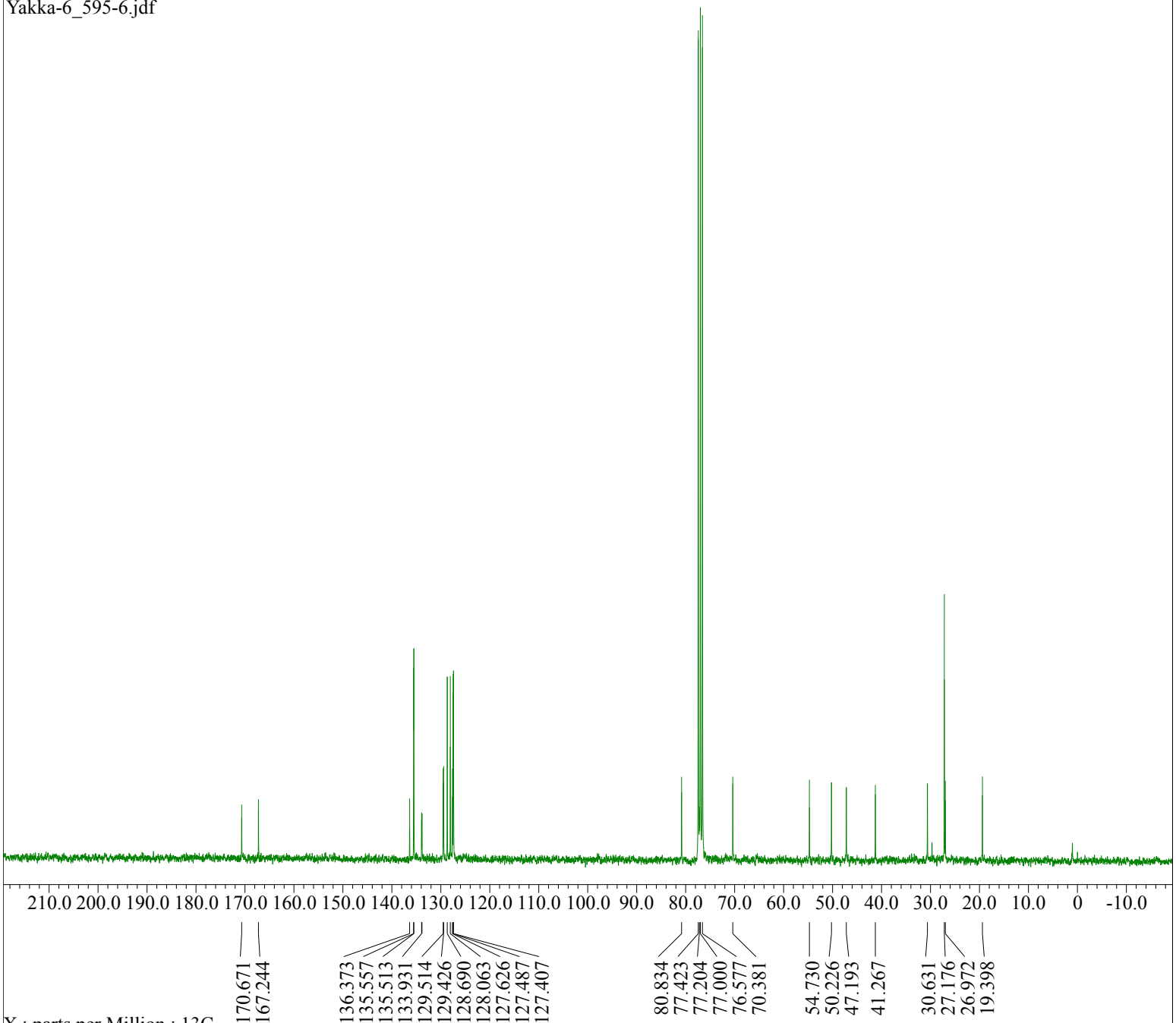
X : parts per Million : 1H

```
Filename       = Yakka-6_1606-7.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 29-SEP-2016 21:27:16
Revision_Time  = 29-SEP-2016 21:30:59
Current_Time   = 29-SEP-2016 21:31:55

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.96[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



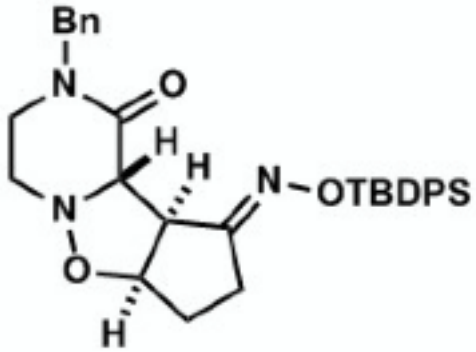
4b

Yakka-6_595-6.jdf



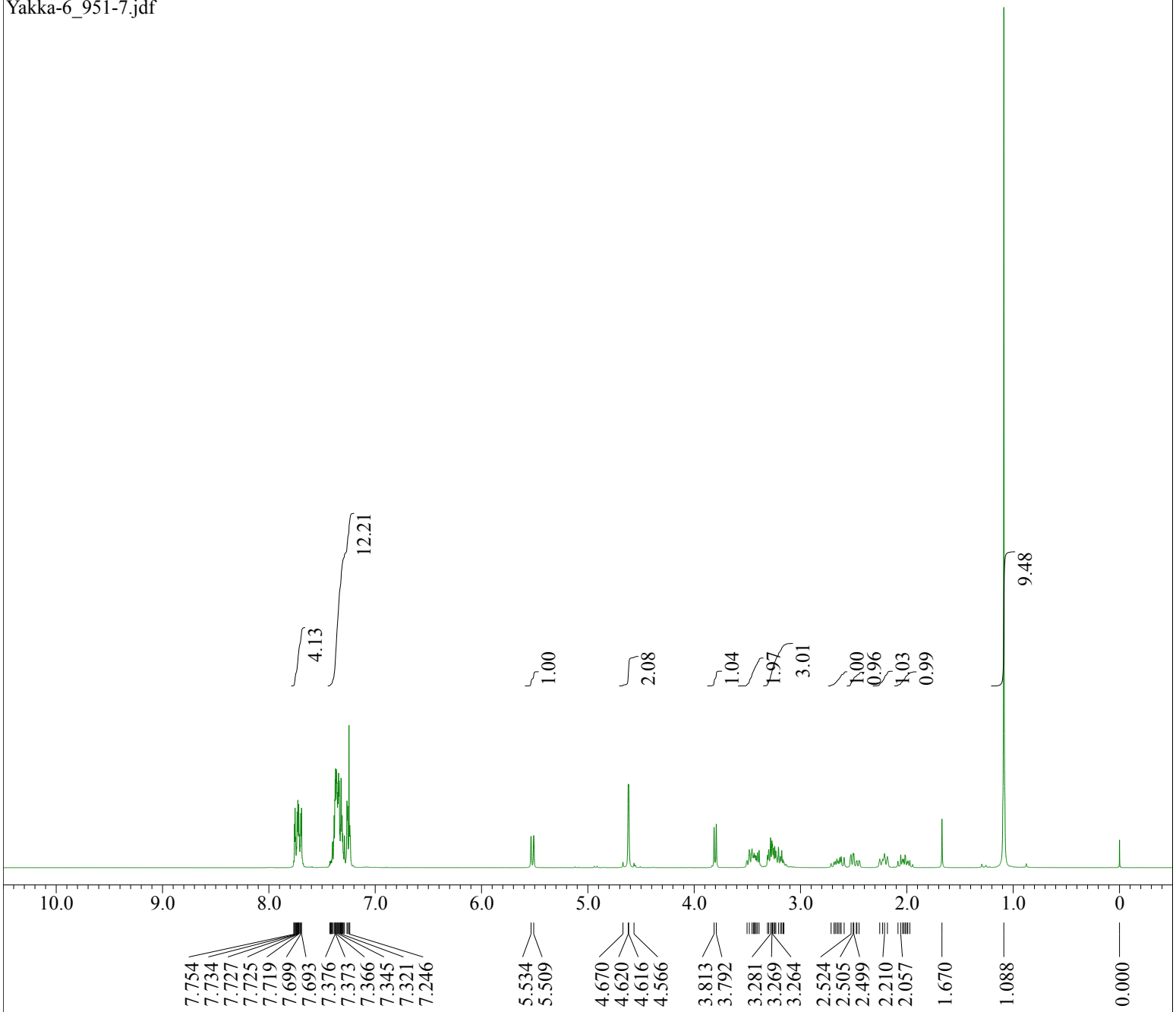
X : parts per Million : 13C

```
Filename       = Yakka-6_595-6.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 29-SEP-2016 21:32:31
Revision_Time  = 29-SEP-2016 21:35:03
Current_Time   = 29-SEP-2016 21:36:01

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.66[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 2048
```



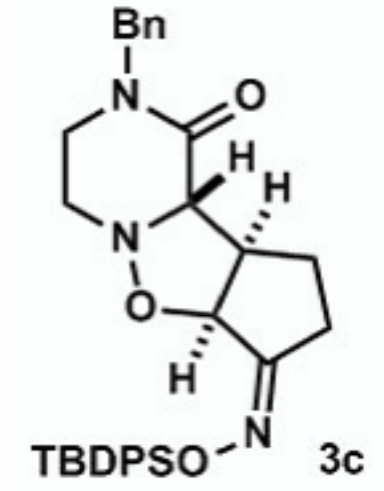
4b

Yakka-6_951-7.jdf

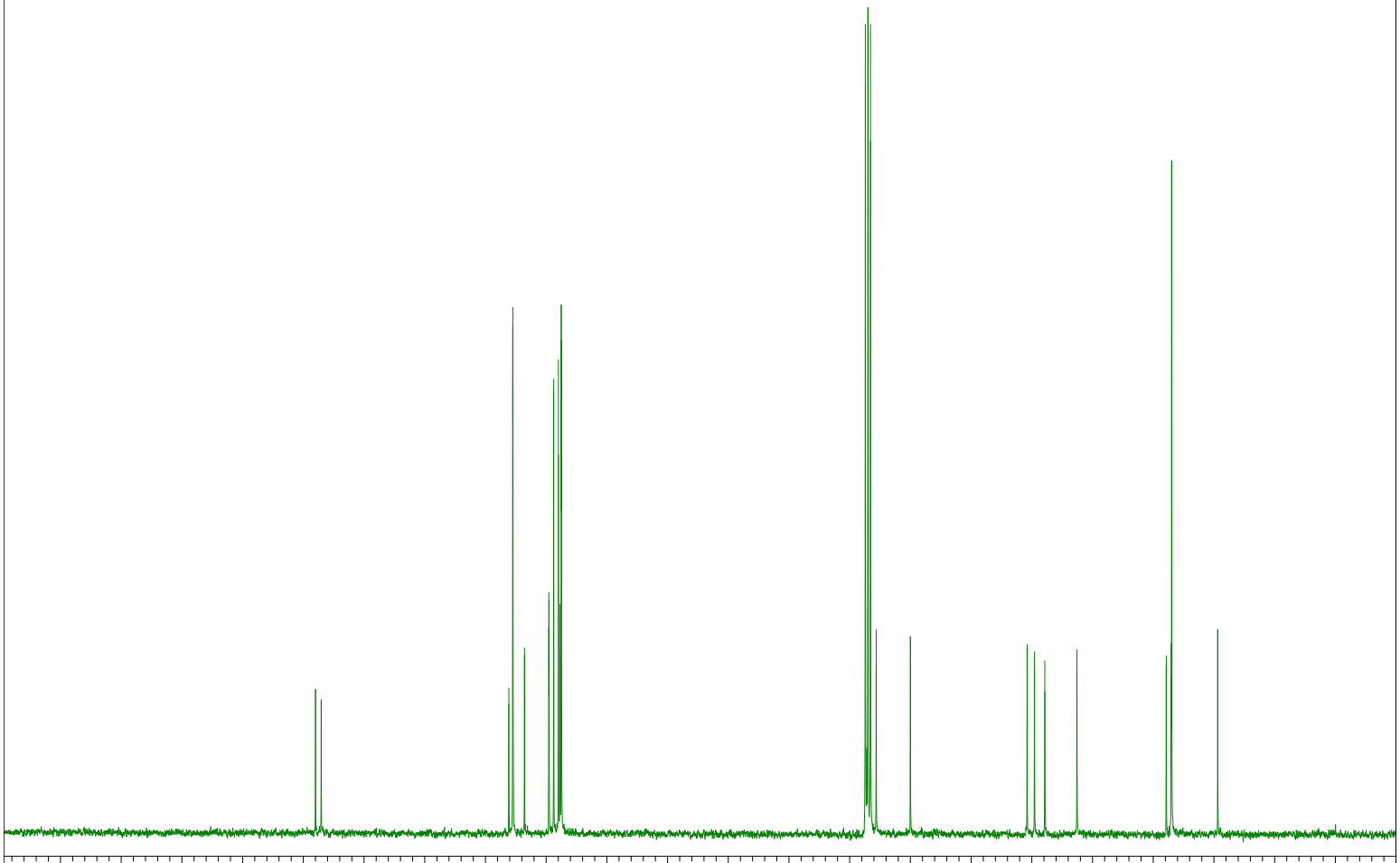


X : parts per Million : 1H

```
Filename       = Yakka-6_951-7.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 29-SEP-2016 21:39:55
Revision_Time  = 29-SEP-2016 21:43:41
Current_Time   = 29-SEP-2016 21:45:54

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.96[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



Yakka-6_952-3.jdf

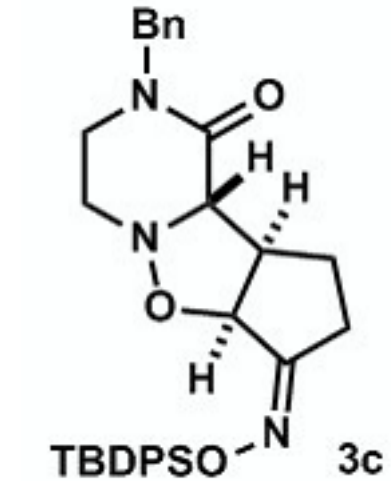


210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0

167.995
167.048
136.147
135.506
135.469
133.567
129.565
129.514
128.756
127.983
127.721
127.516
127.473
77.423
77.204
77.000
76.577
75.637
70.002
50.772
49.562
47.856
42.564
27.854
27.067
26.965
19.376

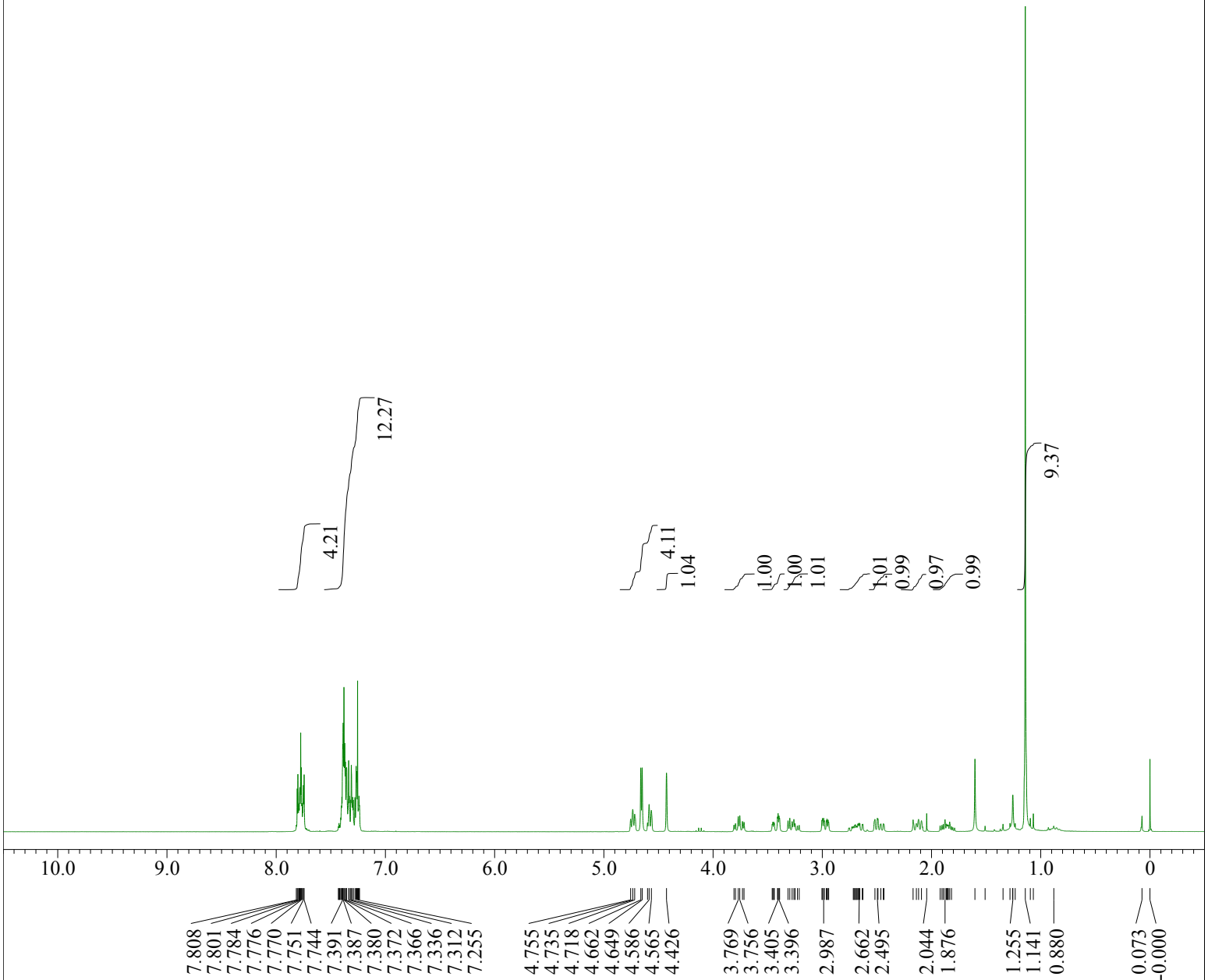
X : parts per Million : 13C

```
Filename       = Yakka-6_952-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 29-SEP-2016 21:46:39
Revision_Time  = 29-SEP-2016 21:47:21
Current_Time   = 29-SEP-2016 21:48:35

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.76[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 2048
```



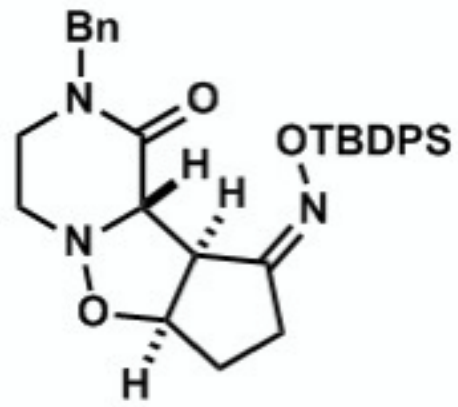
TBDPSO 3c

Yakka-6_955-7.jdf



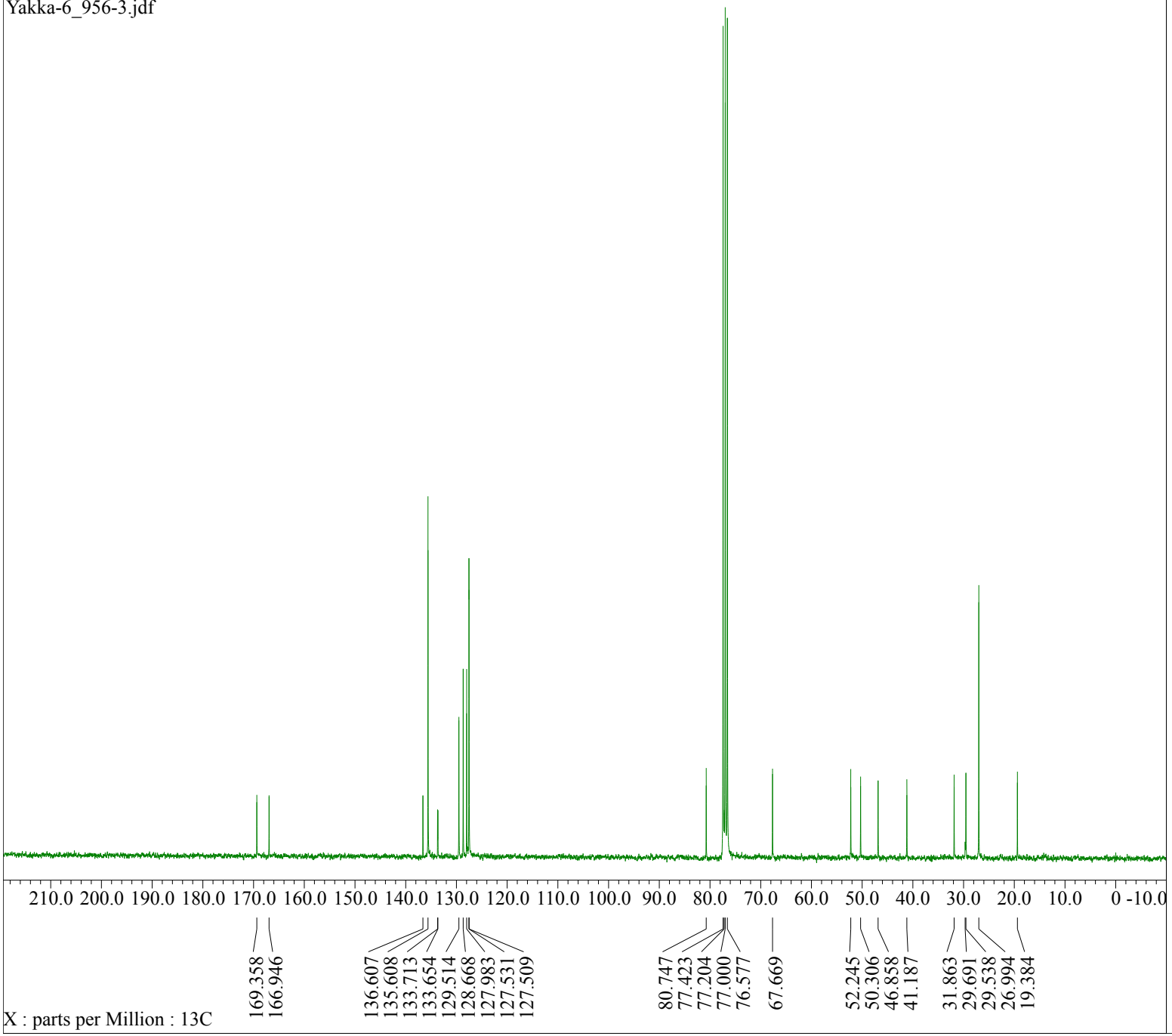
X : parts per Million : 1H

```
Filename       = Yakka-6_955-7.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:41:17
Revision_Time  = 11-NOV-2016 20:45:57
Current_Time   = 11-NOV-2016 20:46:12

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.36[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



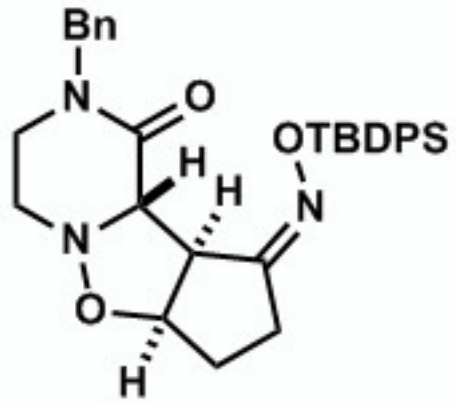
4c

Yakka-6_956-3.jdf



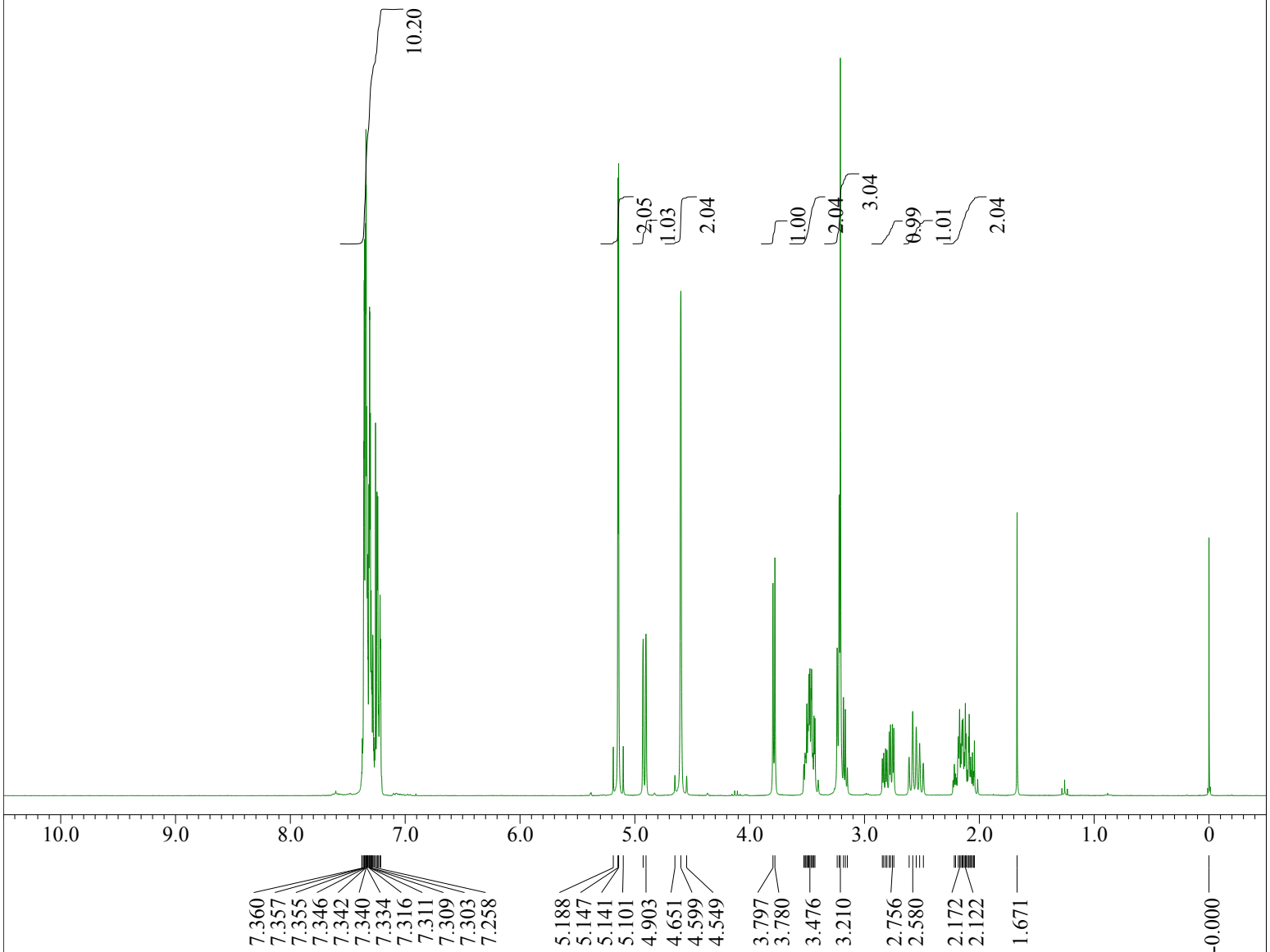
X : parts per Million : 13C

```
Filename       = Yakka-6_956-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:00:26
Revision_Time  = 11-NOV-2016 20:01:25
Current_Time   = 11-NOV-2016 20:02:11

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.76[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 4096
```



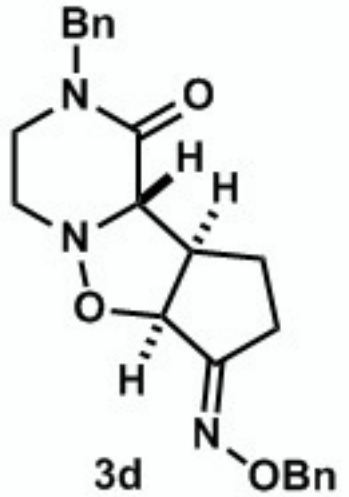
4c

Yakka-6_1603-11.jdf



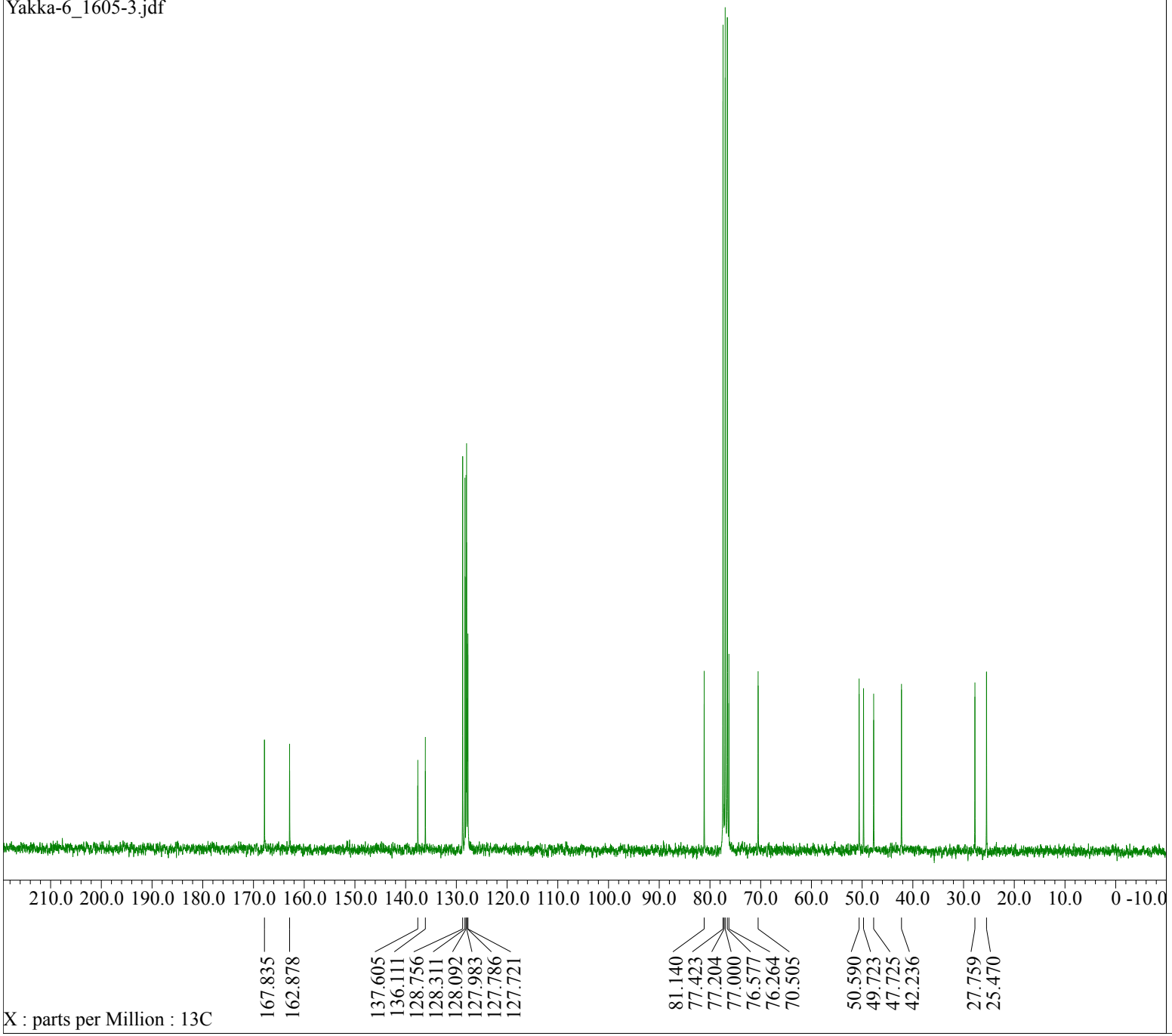
X : parts per Million : 1H

```
Filename       = Yakka-6_1603-11.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:04:27
Revision_Time  = 11-NOV-2016 20:53:22
Current_Time   = 11-NOV-2016 20:53:53

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.86[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



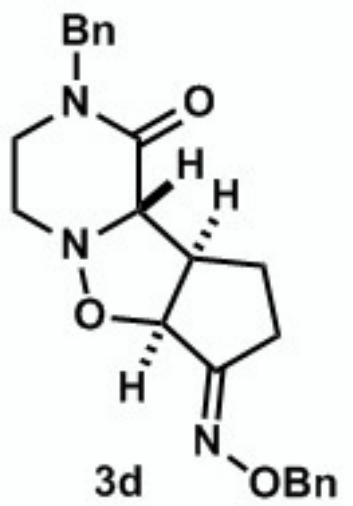
3d

Yakka-6_1605-3.jdf



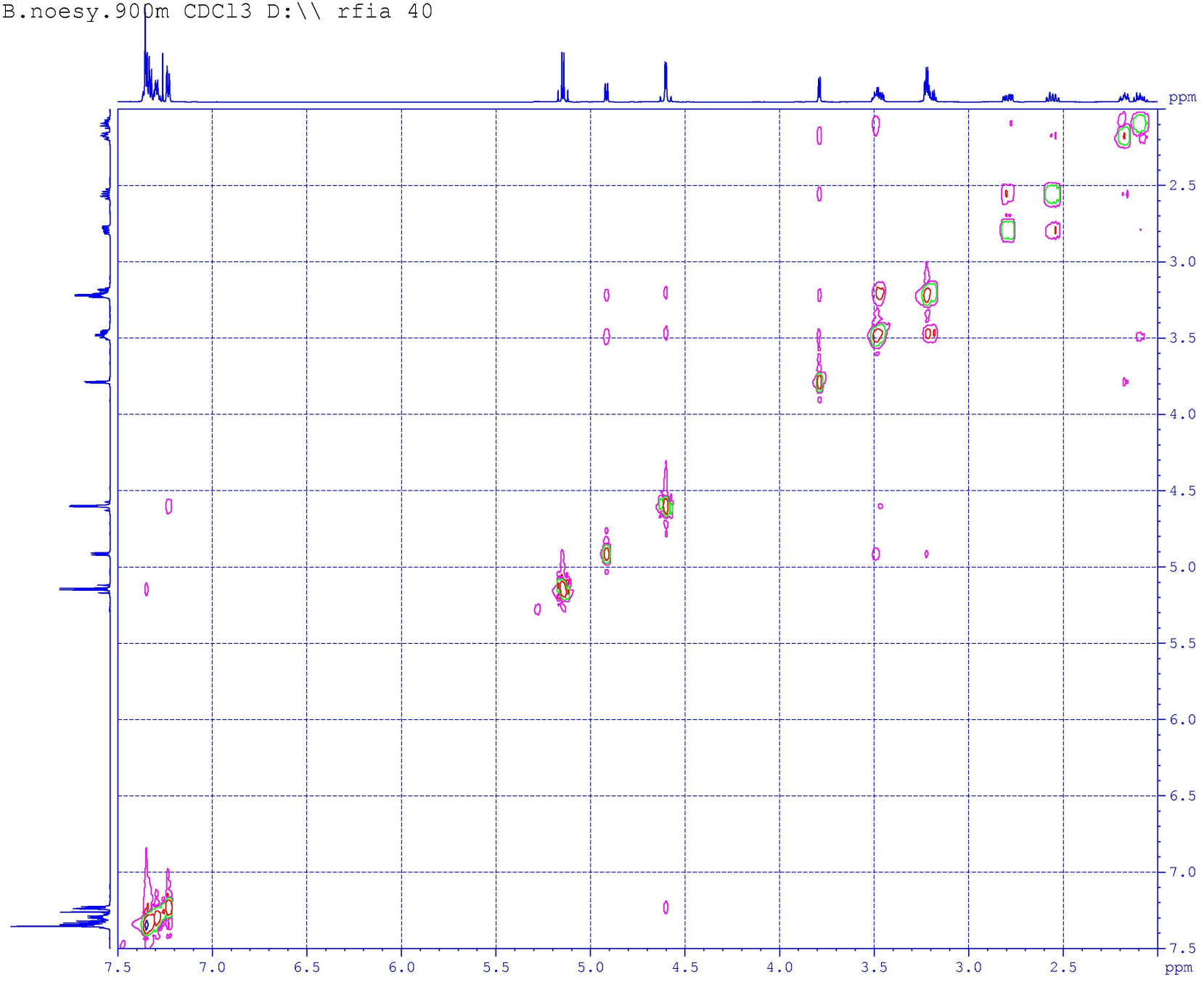
X : parts per Million : 13C

```
Filename       = Yakka-6_1605-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:11:50
Revision_Time  = 11-NOV-2016 20:12:24
Current_Time   = 11-NOV-2016 20:12:53

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.66[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 1024
```

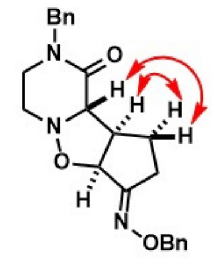


3d

YH_ishiwata_9b_20160916
B.noesy.900m CDCl3 D:\\ rfia 40

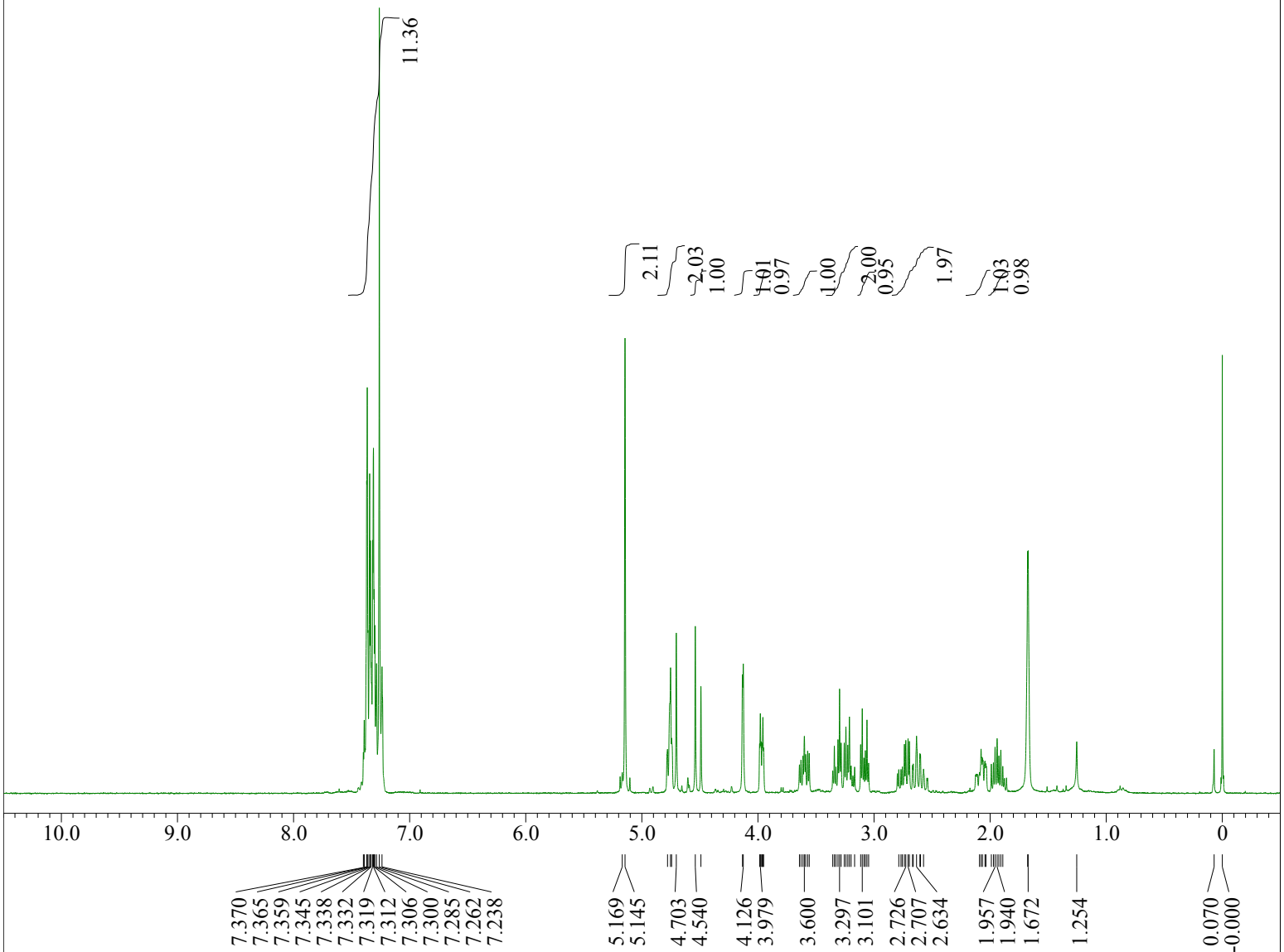



```
Current Data Parameters
NAME            Hashimoto
EXPNO                 502
PROCNO                  1

F2 - Acquisition Parameters
Date_           20160916
Time                10.22
INSTRUM             spect
PROBHD   5 mm CPTCI 1H-
PULPROG      noesygpphpp
TD                  2048
SOLVENT             CDCl3
NS                     8
DS                    16
SWH            5319.149 Hz
FIDRES         2.597241 Hz
AQ             0.1925120 sec
RG                  20.2
DW                94.000 usec
DE                10.00 usec
TE                300.0 K
D0             0.00008458 sec
D1             1.97460401 sec
D8             0.89999998 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00018800 sec

======== CHANNEL f1 ========
SFO1          600.1322868 MHz
NUC1                  1H
P1                  7.40 usec
P2                 14.80 usec
P17              2500.00 usec
PLW1          8.80000019 W
PLW10         0.71284997 W

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPZ1                40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    256
SFO1            600.1323 MHz
FIDRES         20.777925 Hz
SW                 8.863 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                   1024
SF            600.1300097 MHz
WDW                 QSINE
SSB                     2
LB         0 Hz
GB         0
PC                   1.00

F1 - Processing parameters
SI                   1024
MC2            States-TPPI
SF            600.1300097 MHz
WDW                 QSINE
SSB                     2
LB         0 Hz
GB         0
```



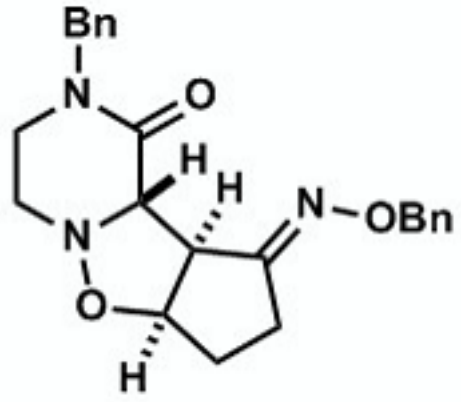
NOESY

Yakka-6_1611-11.jdf



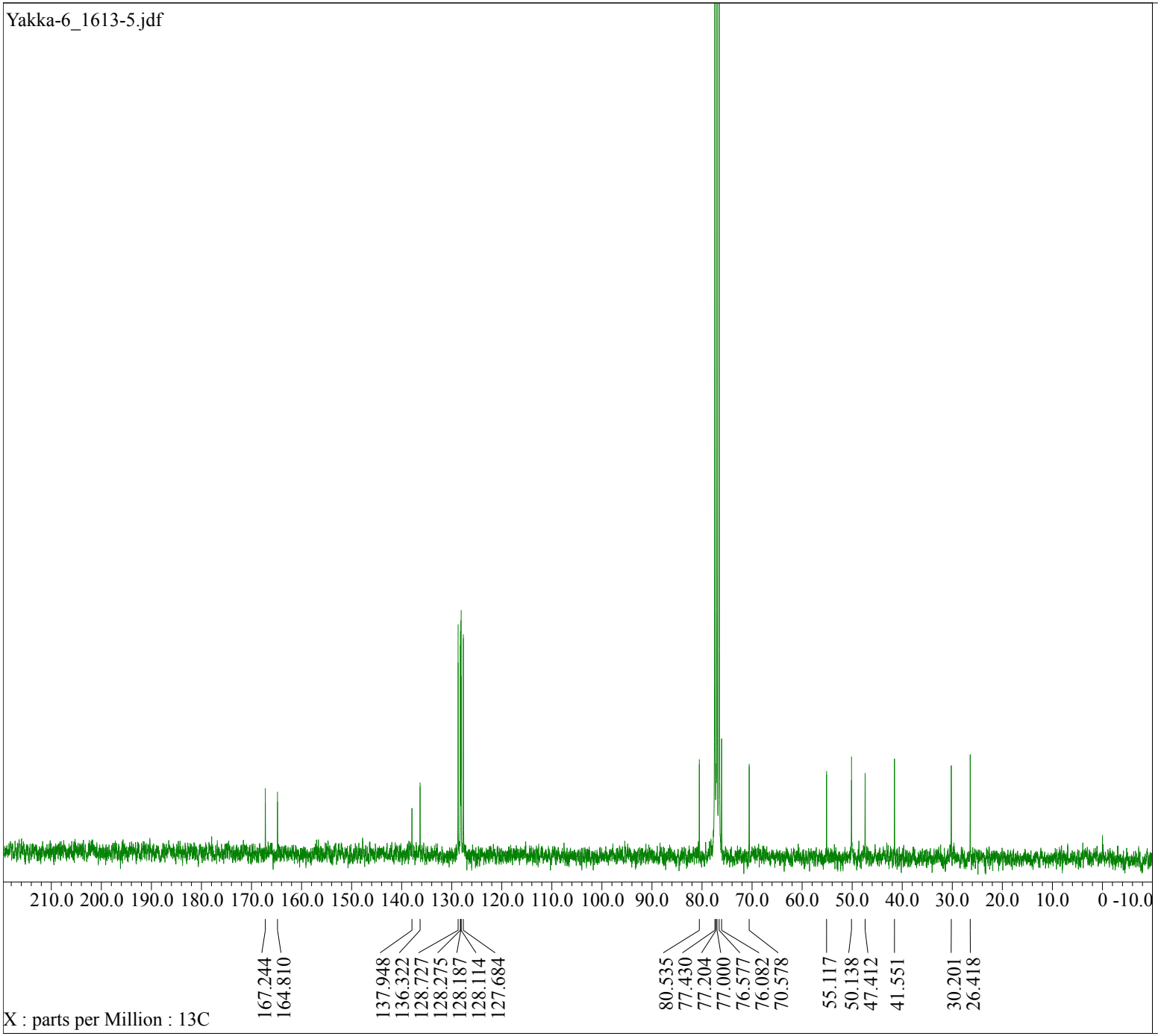
X : parts per Million : 1H

```
Filename       = Yakka-6_1611-11.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:14:49
Revision_Time  = 11-NOV-2016 21:01:23
Current_Time   = 11-NOV-2016 21:01:45

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.76[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



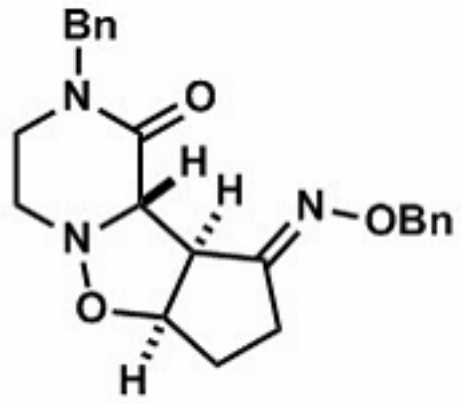
4d

Yakka-6_1613-5.jdf



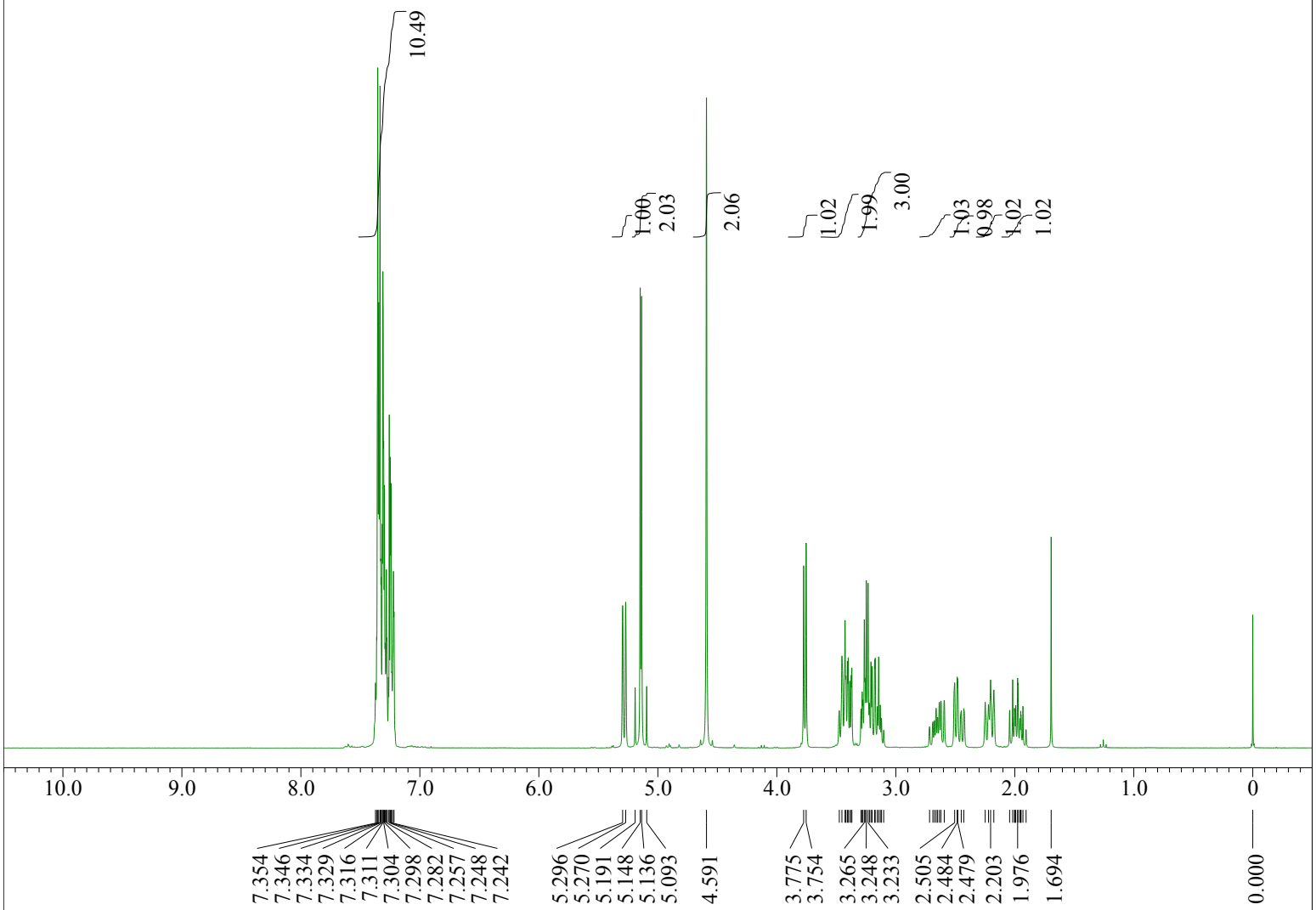
X : parts per Million : 13C

```
Filename       = Yakka-6_1613-5.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:18:21
Revision_Time  = 11-NOV-2016 20:20:19
Current_Time   = 11-NOV-2016 20:21:27

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.46[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 1800
```



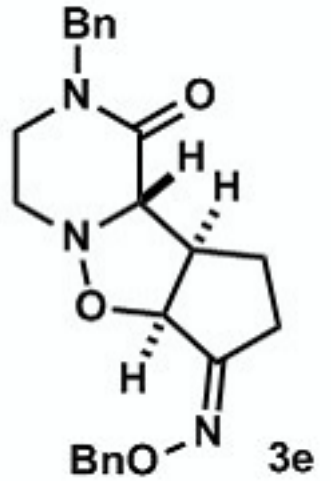
4d

Yakka-6_1602-6.jdf



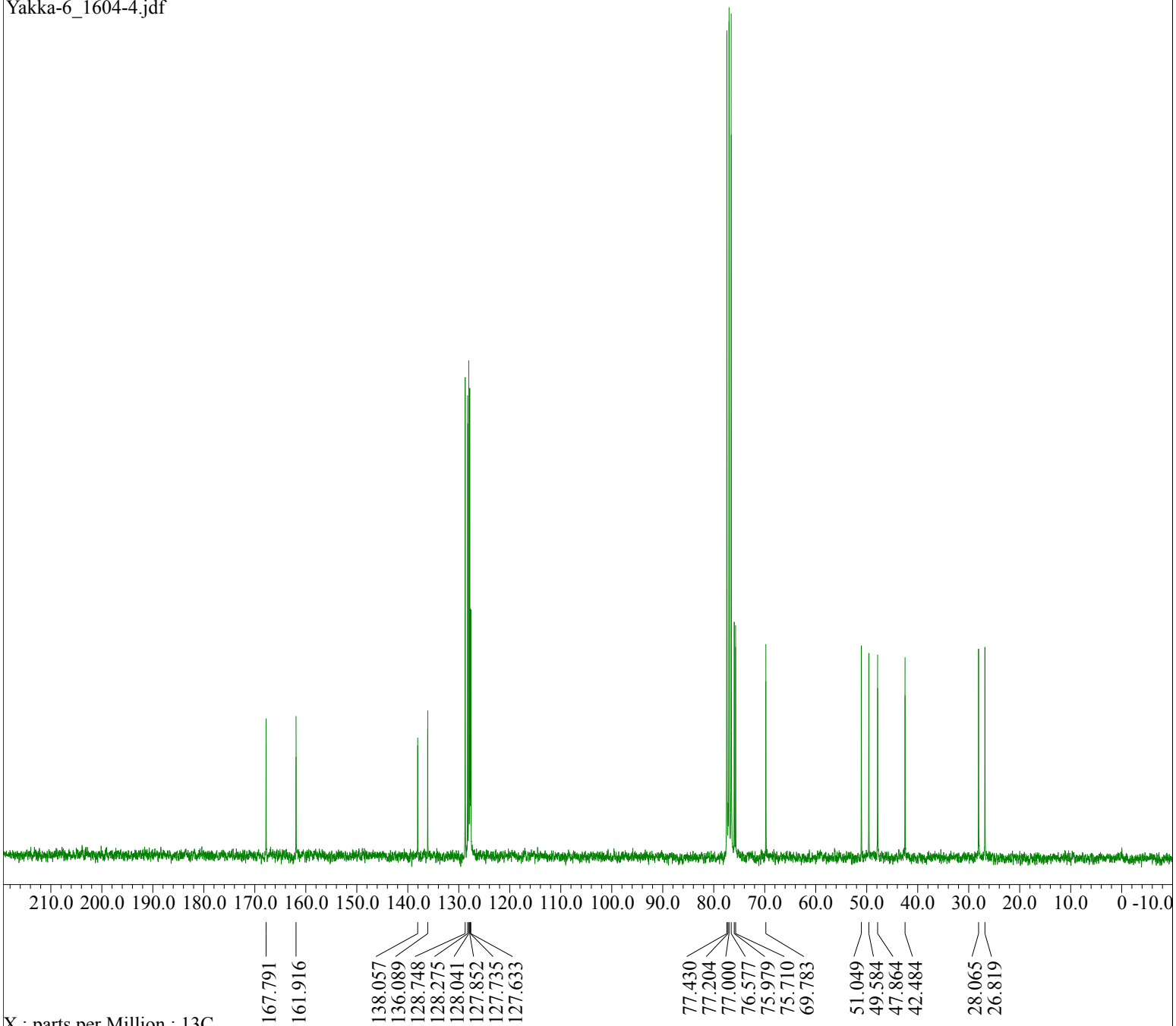
X : parts per Million : 1H

```
Filename       = Yakka-6_1602-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:23:27
Revision_Time  = 11-NOV-2016 21:07:43
Current_Time   = 11-NOV-2016 21:08:00

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.96[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



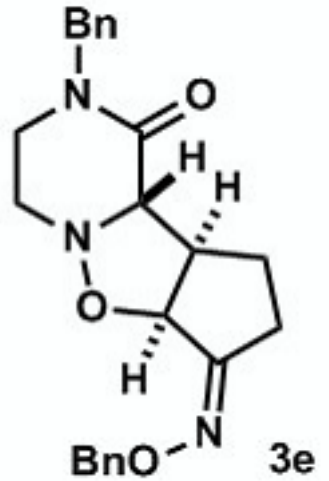
3e

Yakka-6_1604-4.jdf



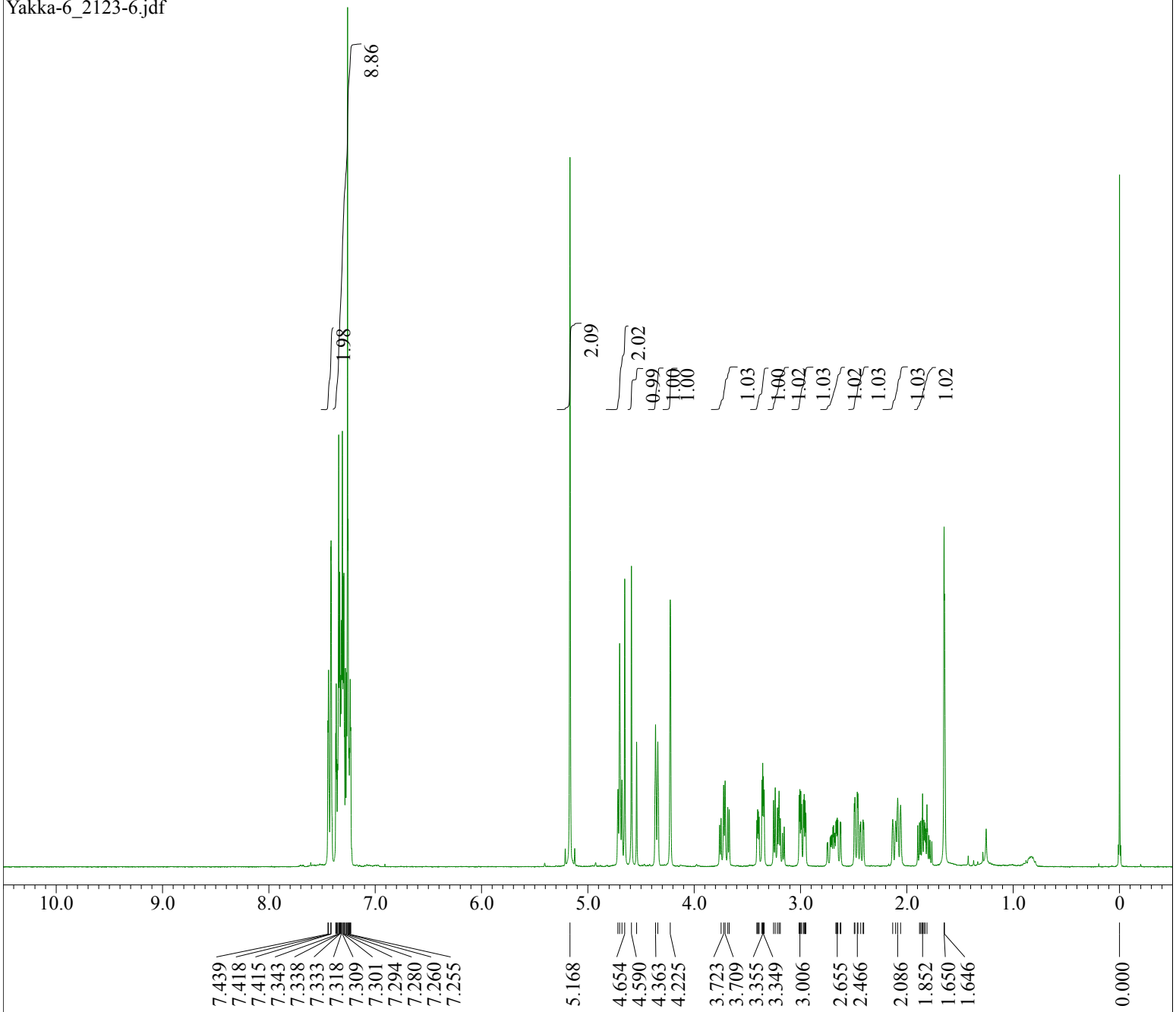
X : parts per Million : 13C

```
Filename       = Yakka-6_1604-4.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:28:00
Revision_Time  = 11-NOV-2016 20:28:35
Current_Time   = 11-NOV-2016 20:28:53

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.66[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 1024
```



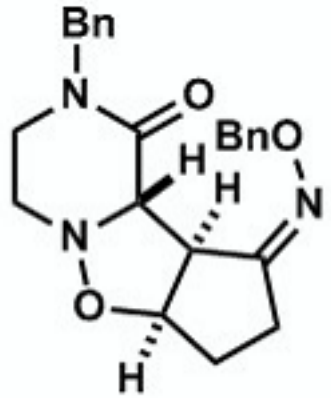
3e

Yakka-6_2123-6.jdf



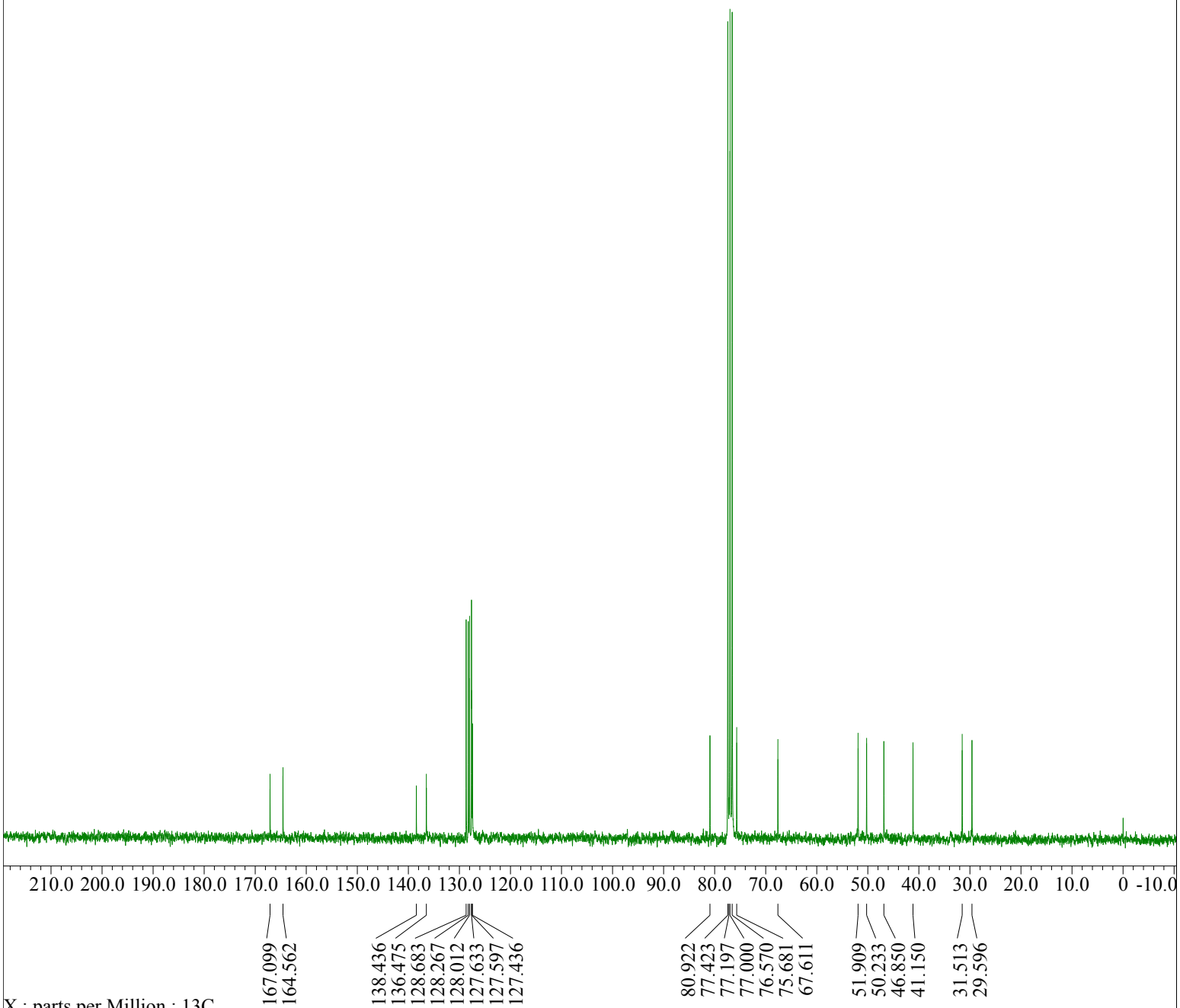
X : parts per Million : 1H

```
Filename       = Yakka-6_2123-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:30:44
Revision_Time  = 11-NOV-2016 21:13:54
Current_Time   = 11-NOV-2016 21:14:26

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.06[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



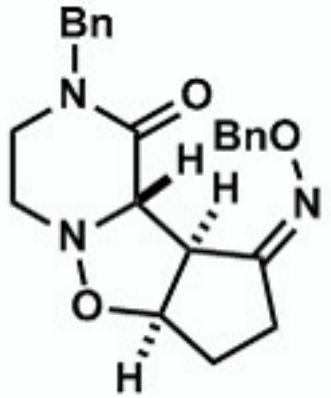
4e

Yakka-6_2130-3.jdf



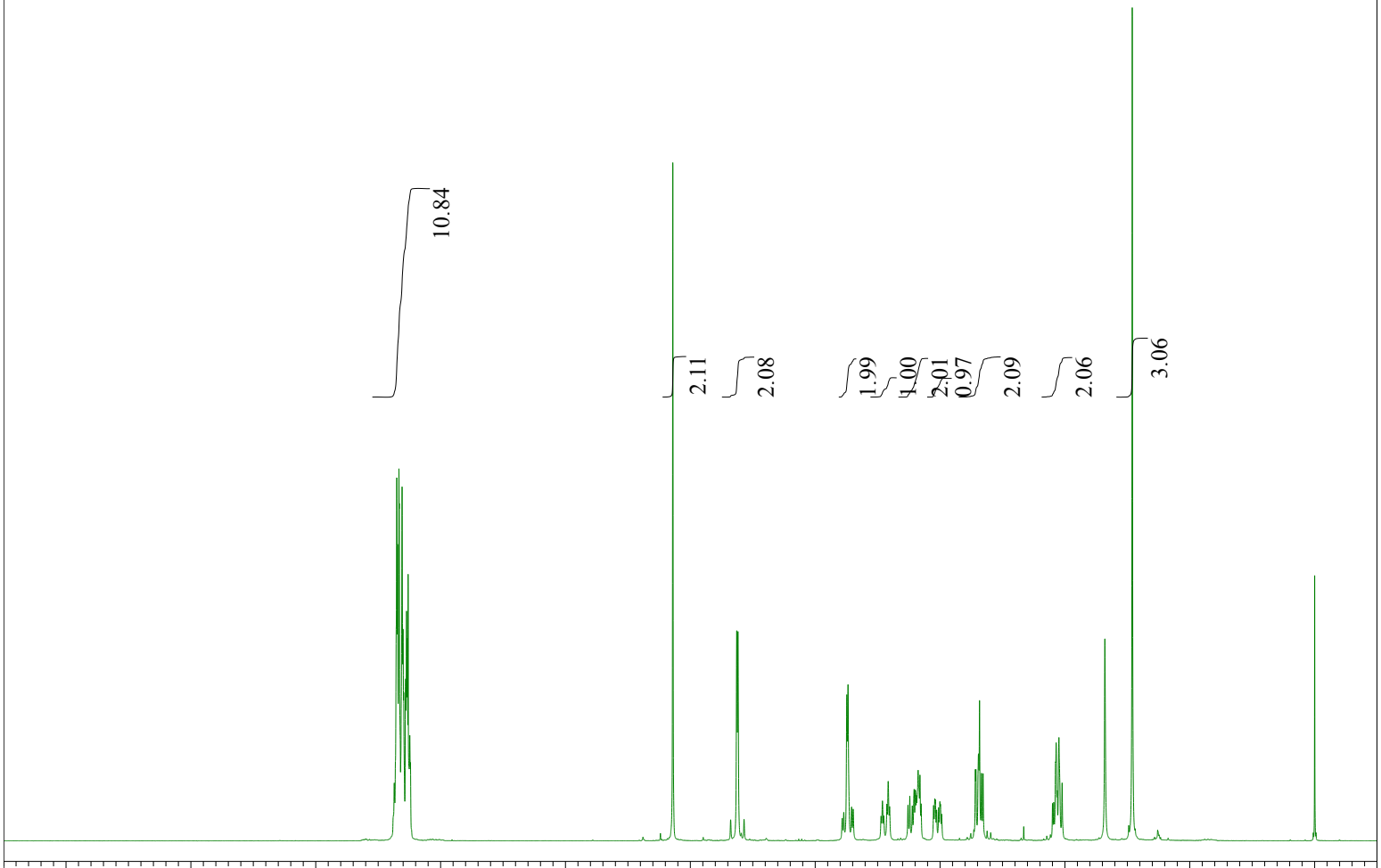
X : parts per Million : 13C

```
Filename       = Yakka-6_2130-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 20:34:07
Revision_Time  = 11-NOV-2016 20:34:58
Current_Time   = 11-NOV-2016 20:35:35

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.76[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 1024
```



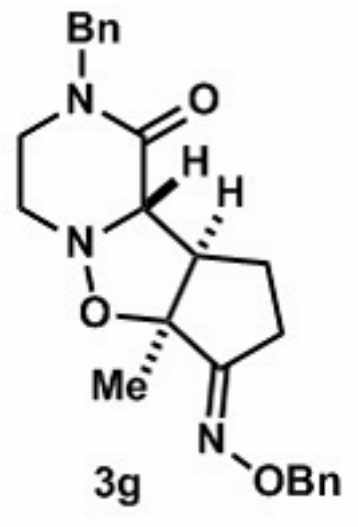
4e

Yakka-6_1732-6.jdf

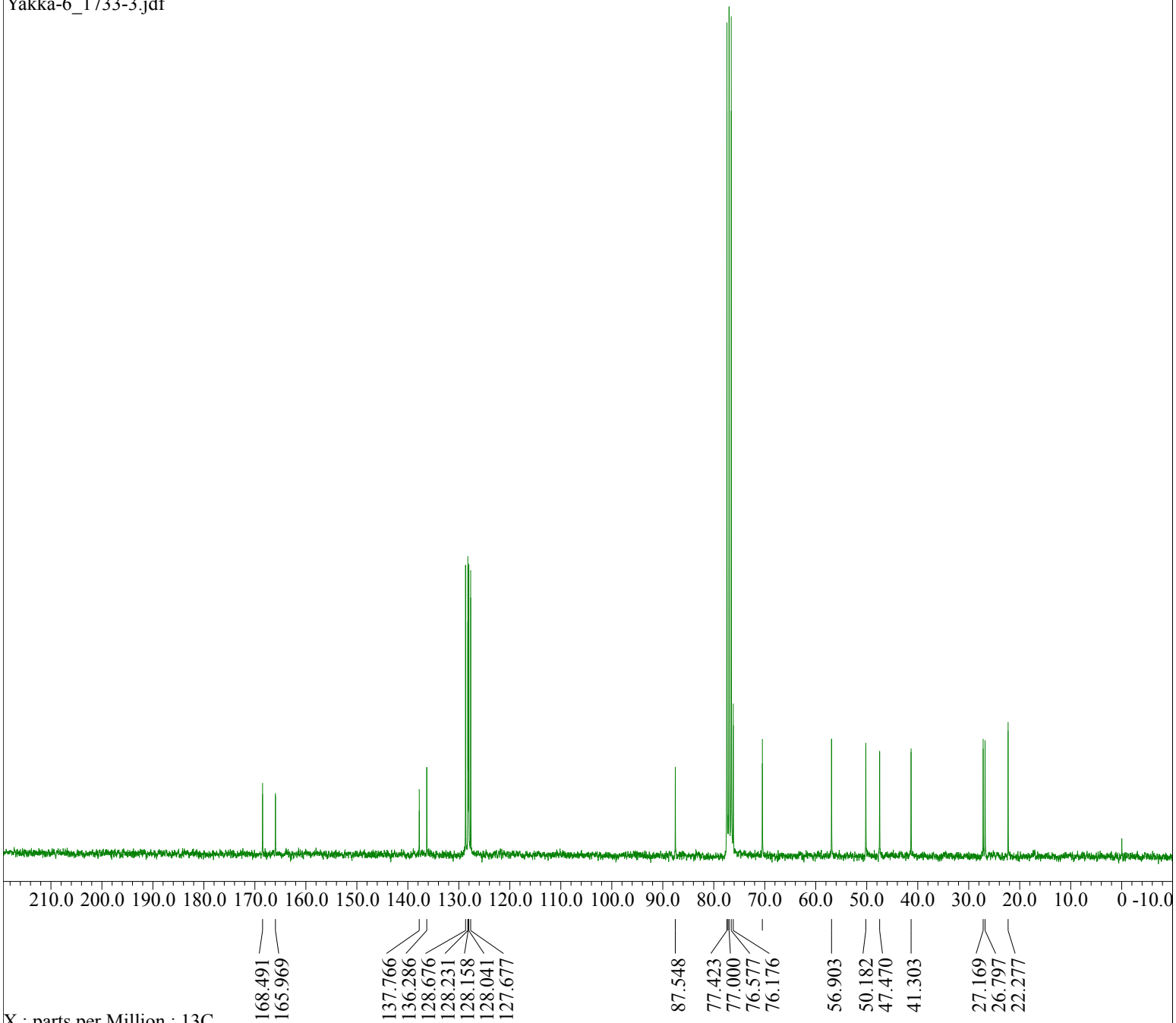


X : parts per Million : 1H

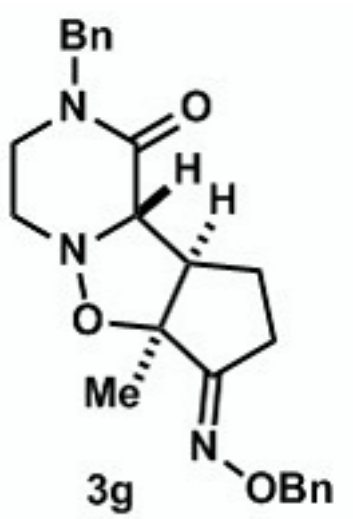
```
Filename       = Yakka-6_1732-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 21:44:35
Revision_Time  = 11-NOV-2016 21:50:18
Current_Time   = 11-NOV-2016 21:50:41

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.06[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



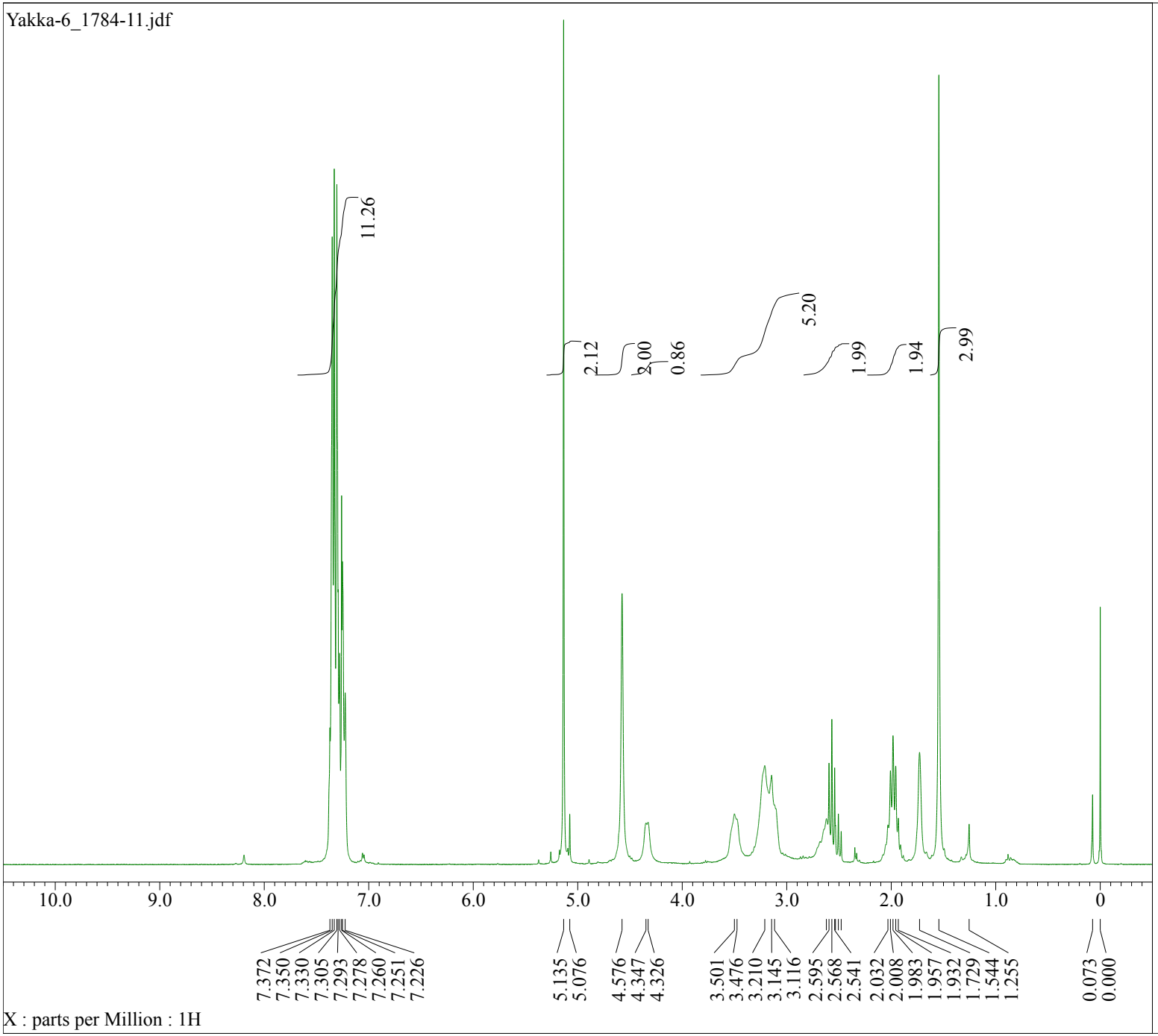
Yakka-6_1733-3.jdf




```
Filename       = Yakka-6_1733-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 21:53:27
Revision_Time  = 11-NOV-2016 21:54:11
Current_Time   = 11-NOV-2016 21:54:45

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.96[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 2048
```

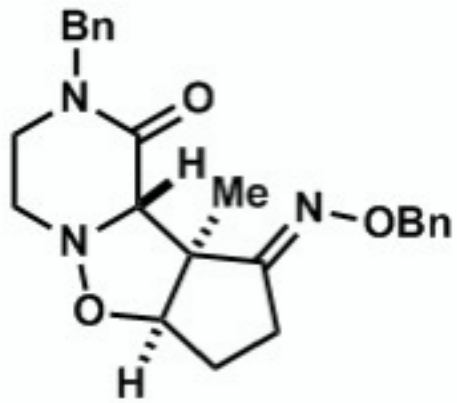


Yakka-6_1784-11.jdf



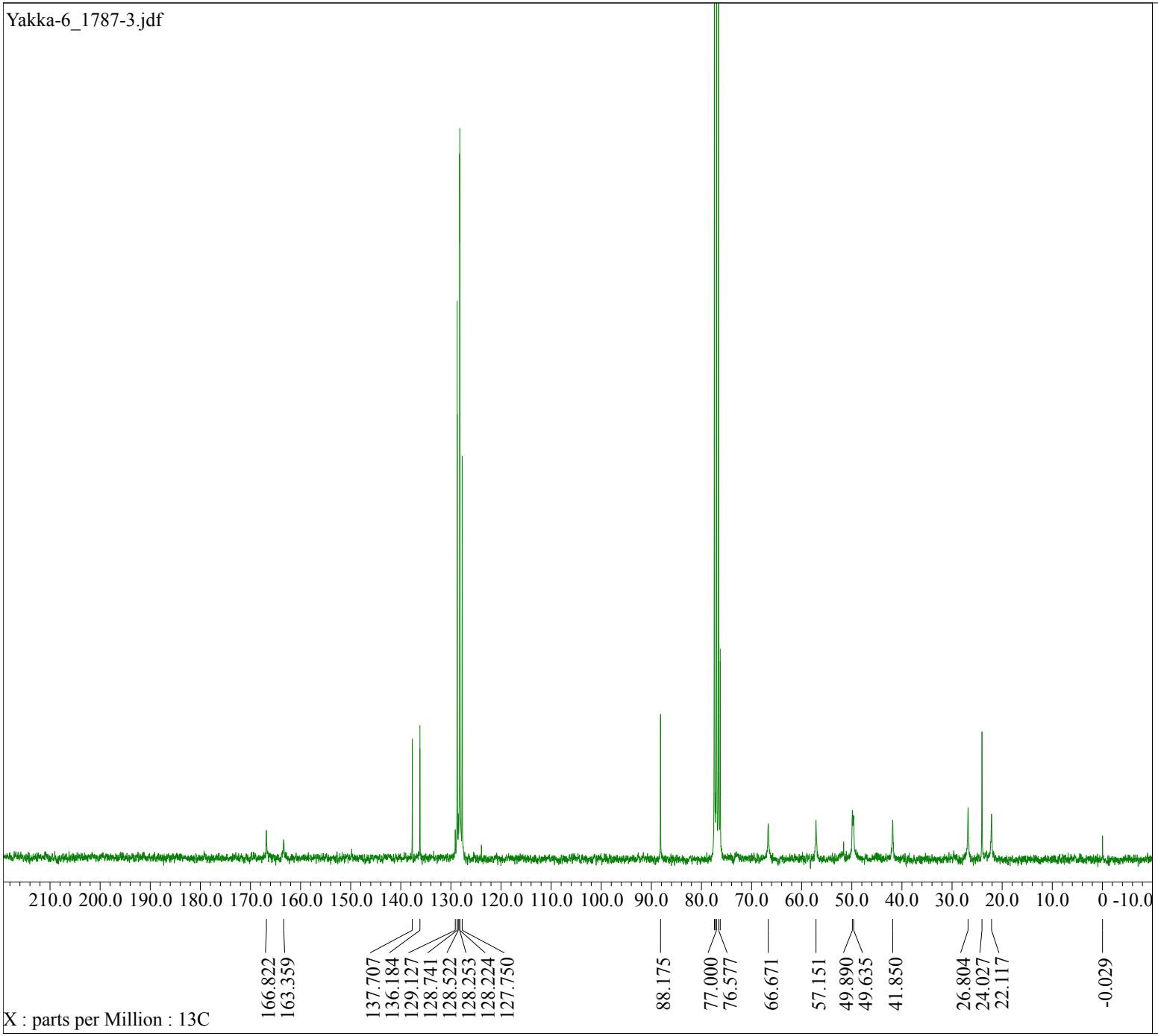
X : parts per Million : 1H

```
Filename       = Yakka-6_1784-11.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 21:56:49
Revision_Time  = 11-NOV-2016 22:10:27
Current_Time   = 11-NOV-2016 22:11:07

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.06[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```

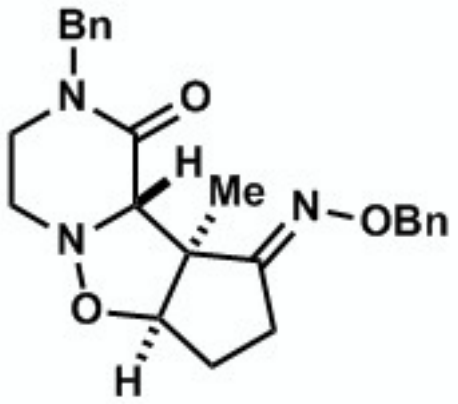


4g

Yakka-6_1787-3.jdf

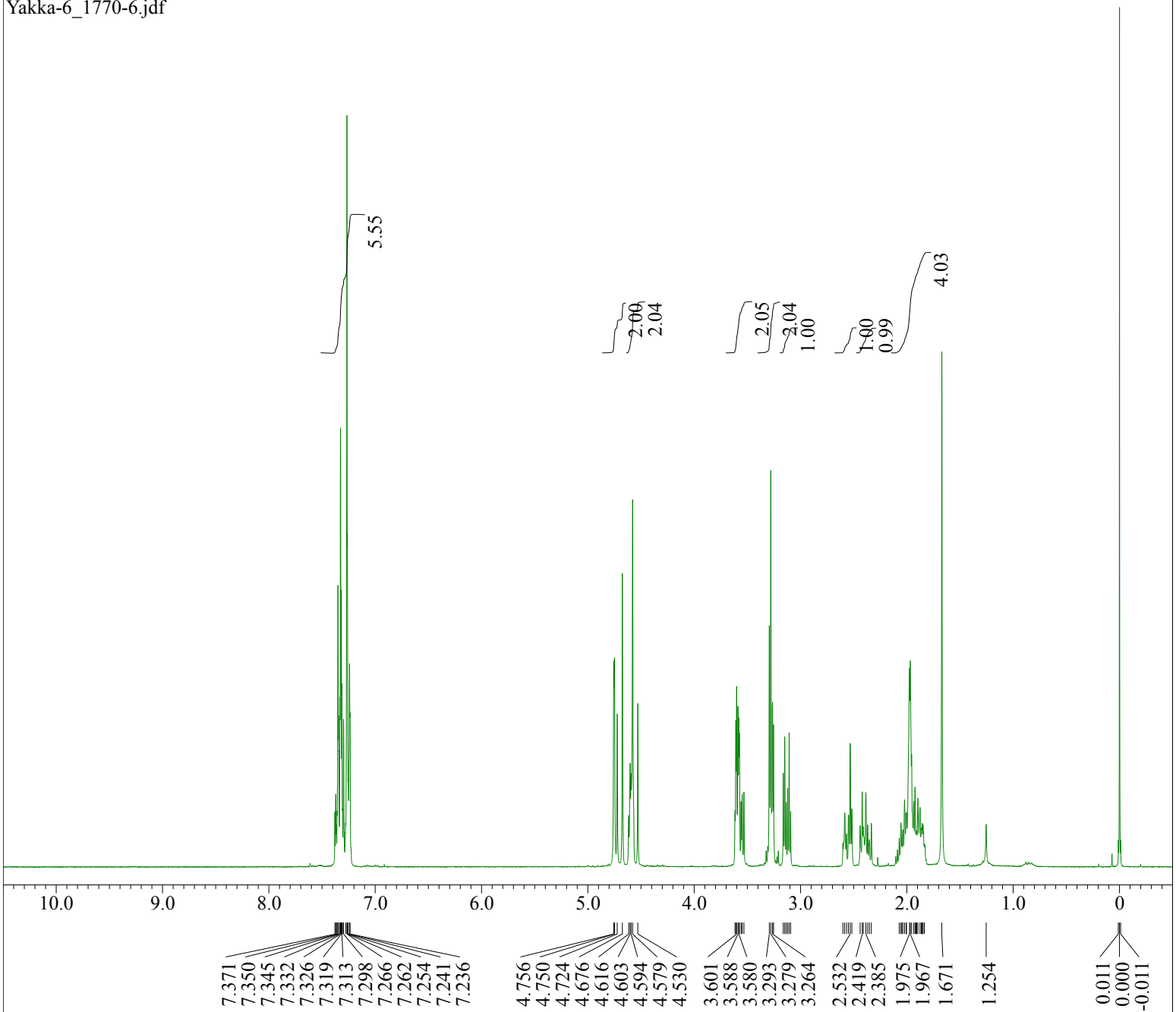



```
Filename       = Yakka-6_1787-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 22:12:05
Revision_Time  = 11-NOV-2016 22:13:35
Current_Time   = 11-NOV-2016 22:14:21

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.76[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 4096
```



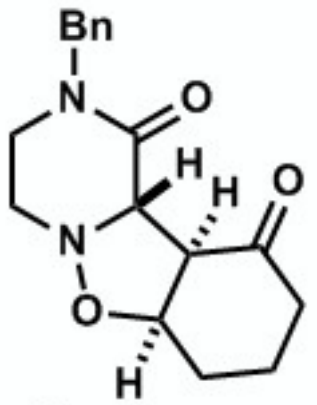
4g

Yakka-6_1770-6.jdf



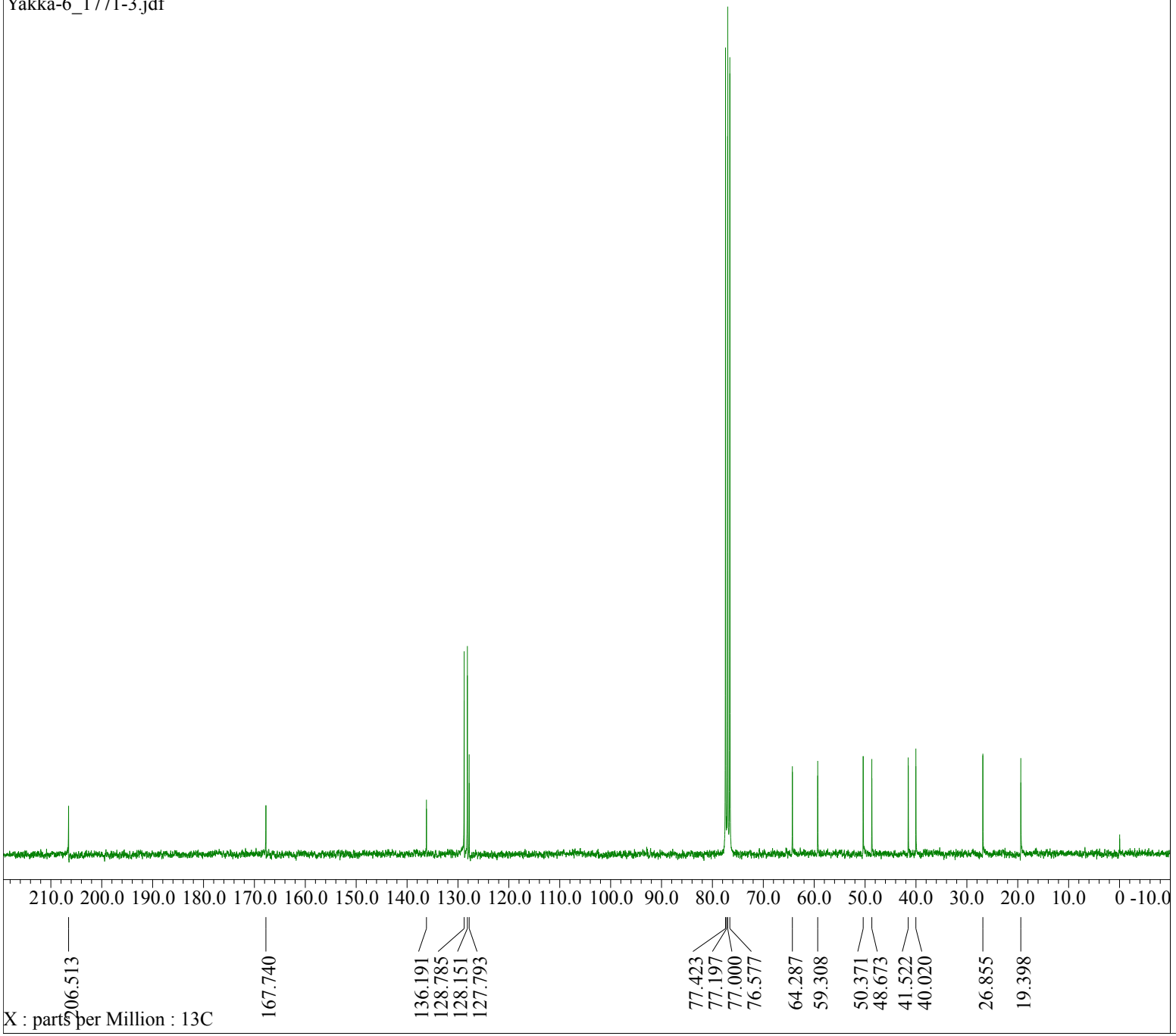
X : parts per Million : 1H

```
Filename       = Yakka-6_1770-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 22:20:03
Revision_Time  = 11-NOV-2016 22:23:42
Current_Time   = 11-NOV-2016 22:24:54

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.86[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



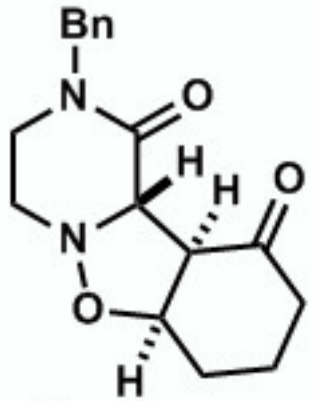
4j

Yakka-6_1771-3.jdf



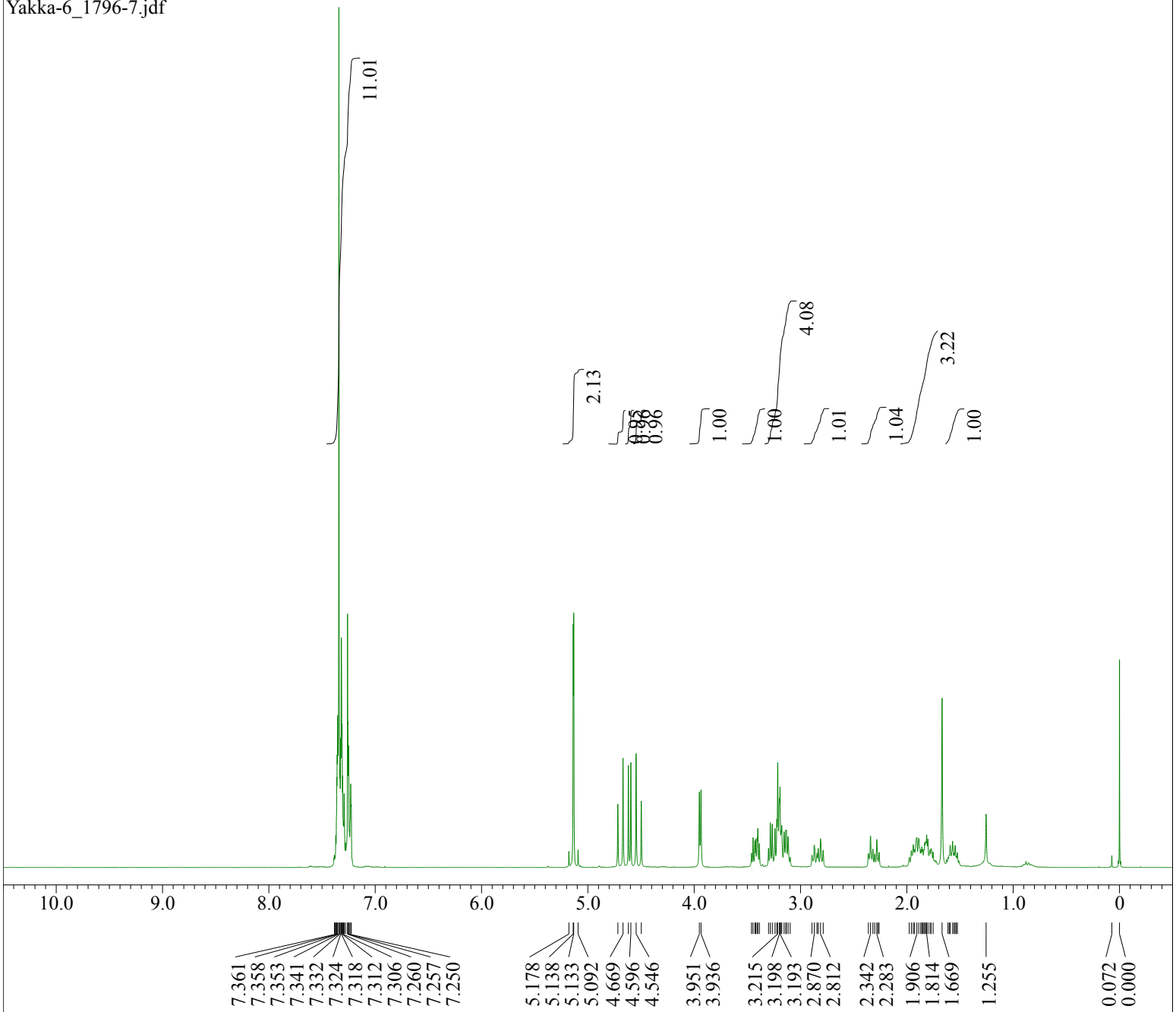
X : parts per Million : 13C

```
Filename       = Yakka-6_1771-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 11-NOV-2016 22:26:25
Revision_Time  = 11-NOV-2016 22:27:12
Current_Time   = 11-NOV-2016 22:27:51

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.56[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 2048
```



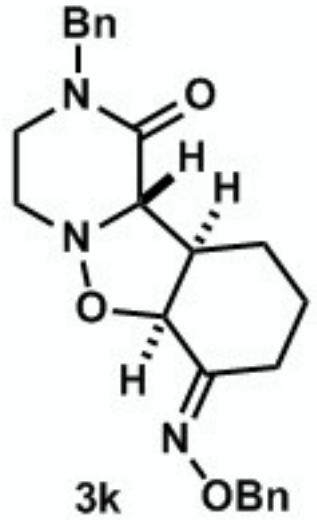
4j

Yakka-6_1796-7.jdf



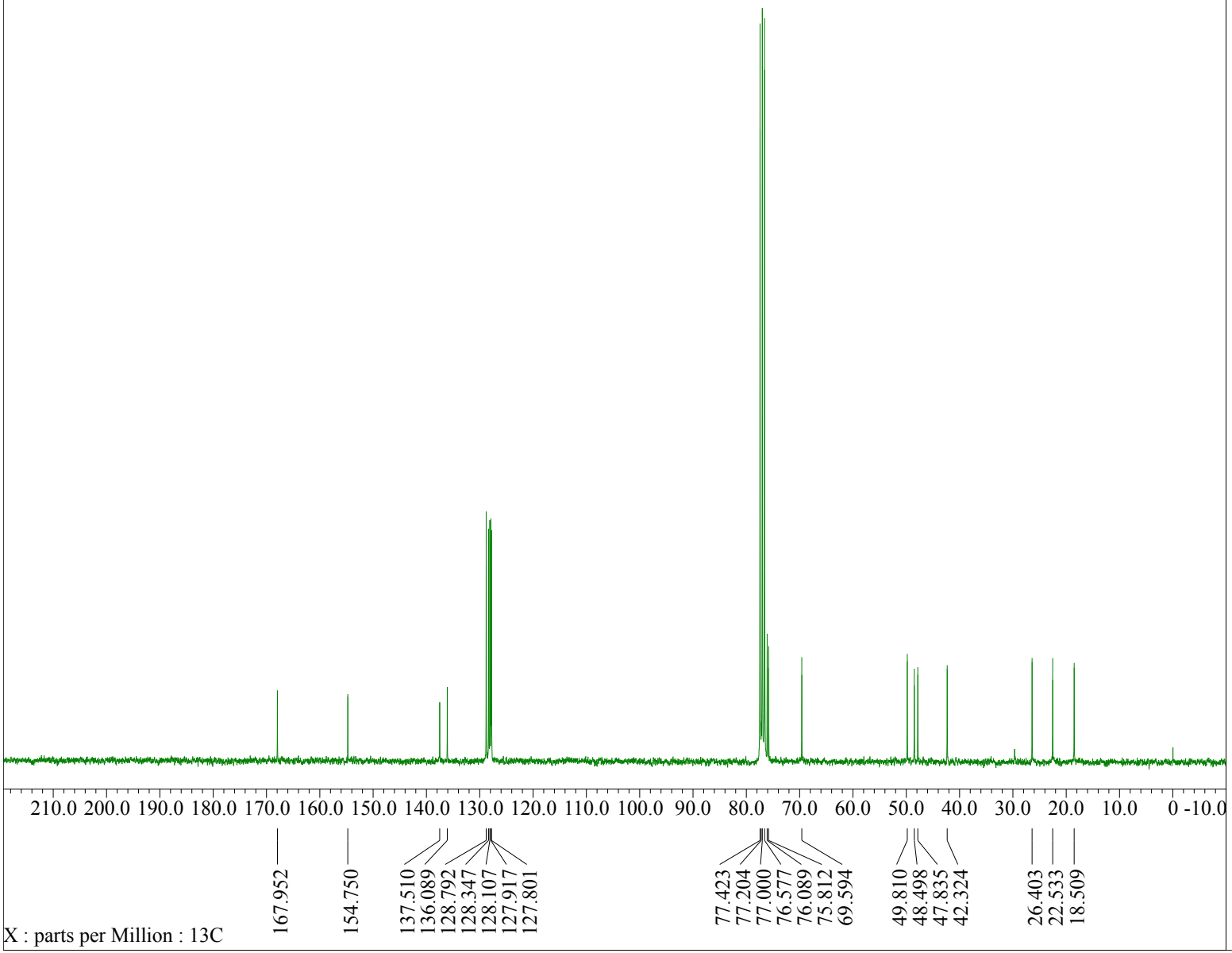
X : parts per Million : 1H

```
Filename       = Yakka-6_1796-7.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 15-NOV-2016 16:28:29
Revision_Time  = 15-NOV-2016 16:33:08
Current_Time   = 15-NOV-2016 16:33:42

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.06[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



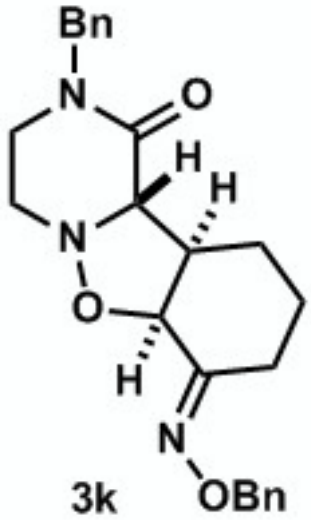
3k

Yakka-6_1797-3.jdf



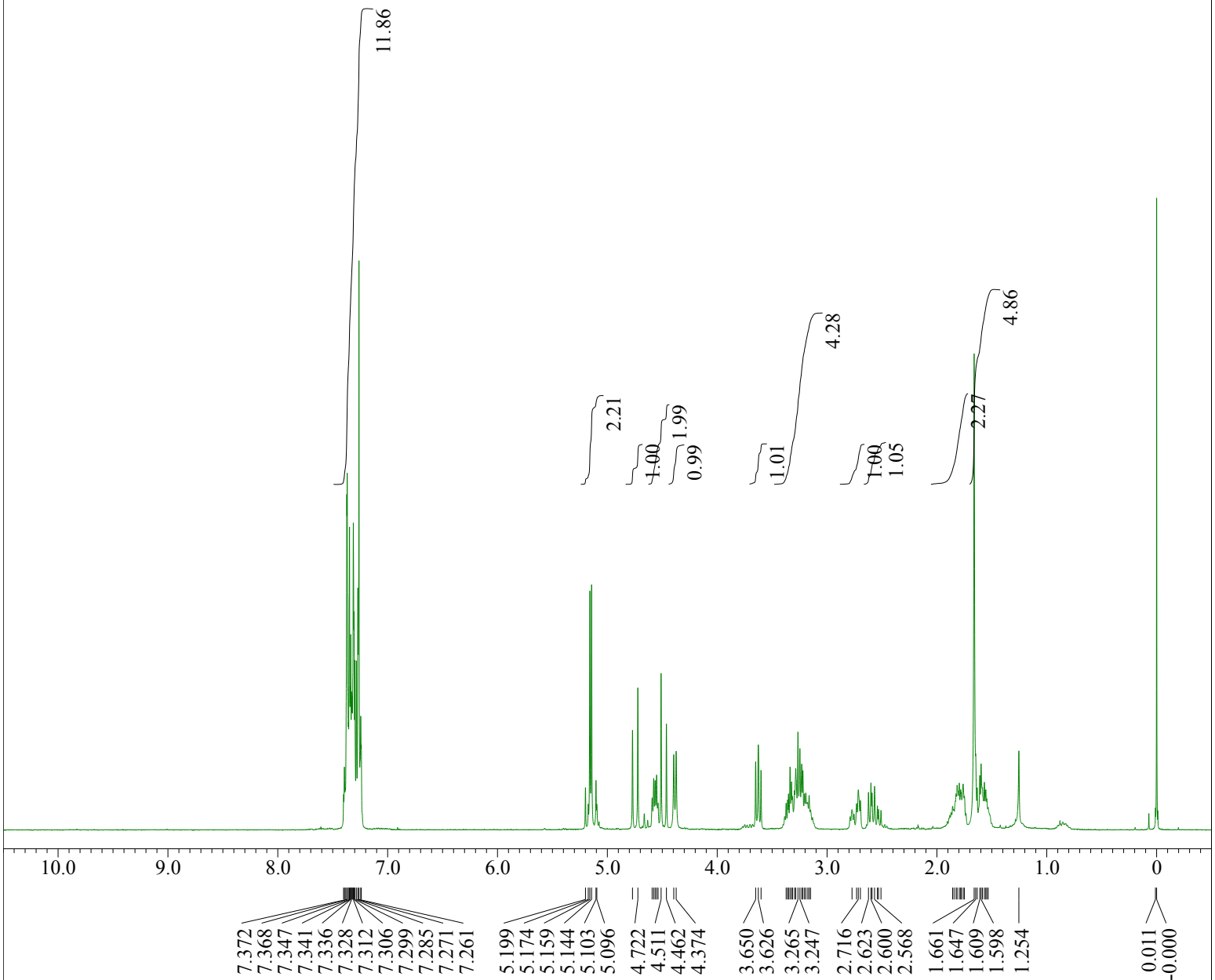
X : parts per Million : 13C

```
Filename       = Yakka-6_1797-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 15-NOV-2016 16:35:17
Revision_Time  = 15-NOV-2016 16:35:59
Current_Time   = 15-NOV-2016 16:36:25

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.76[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 2024
```



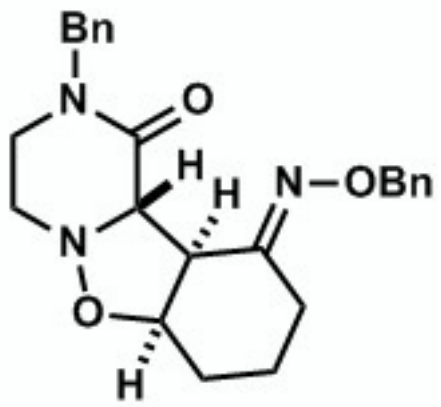
3k

Yakka-6_1789-6.jdf



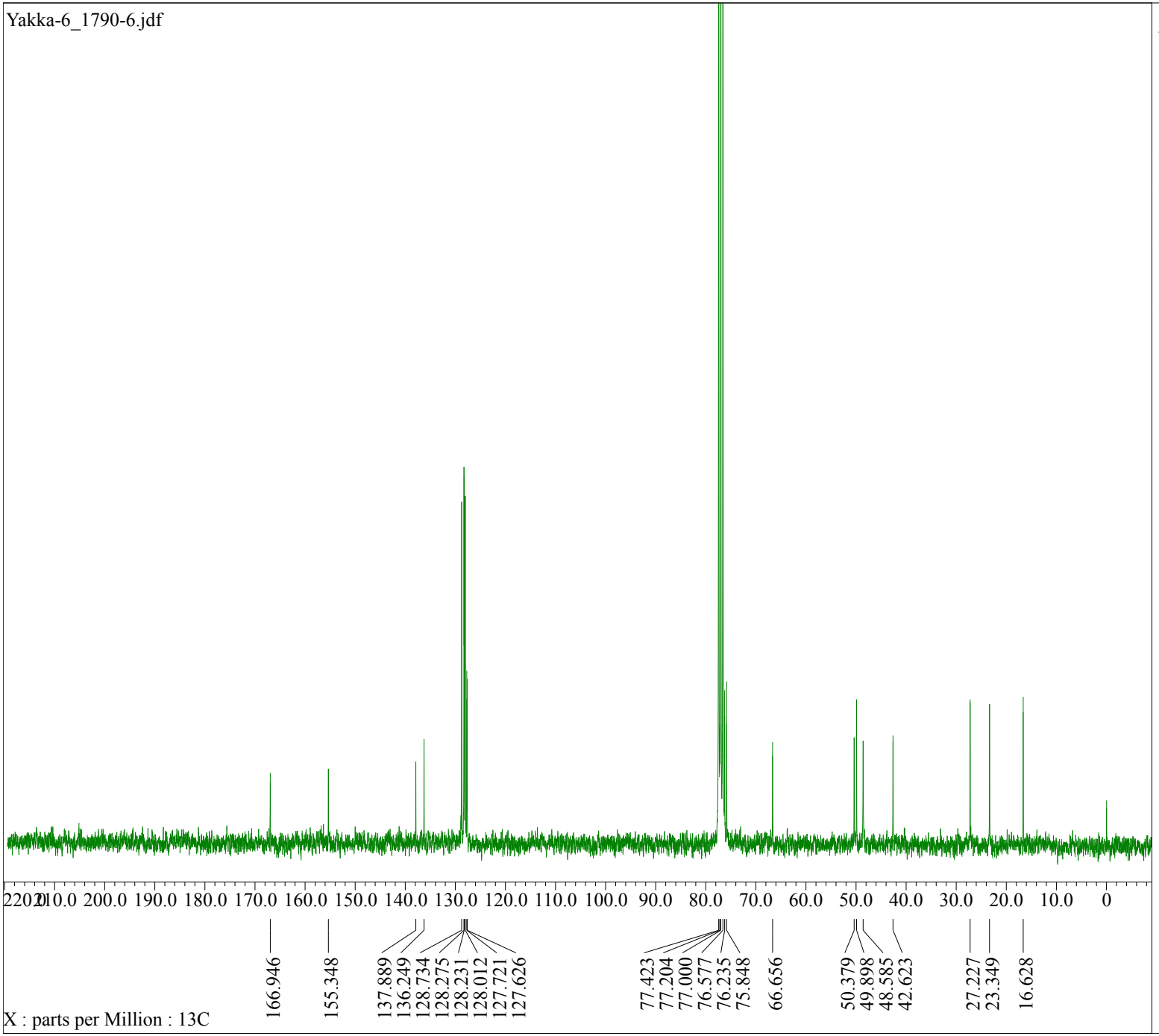
X : parts per Million : 1H

```
Filename       = Yakka-6_1789-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 15-NOV-2016 16:38:54
Revision_Time  = 15-NOV-2016 16:44:13
Current_Time   = 15-NOV-2016 16:44:53

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.26[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



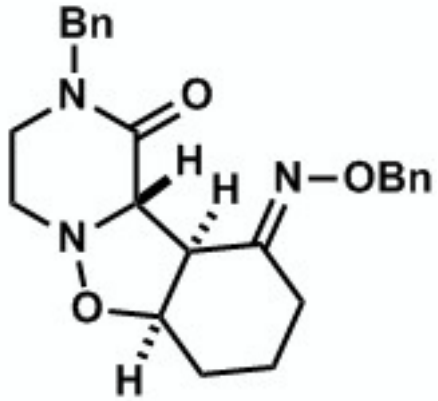
4k

Yakka-6_1790-6.jdf



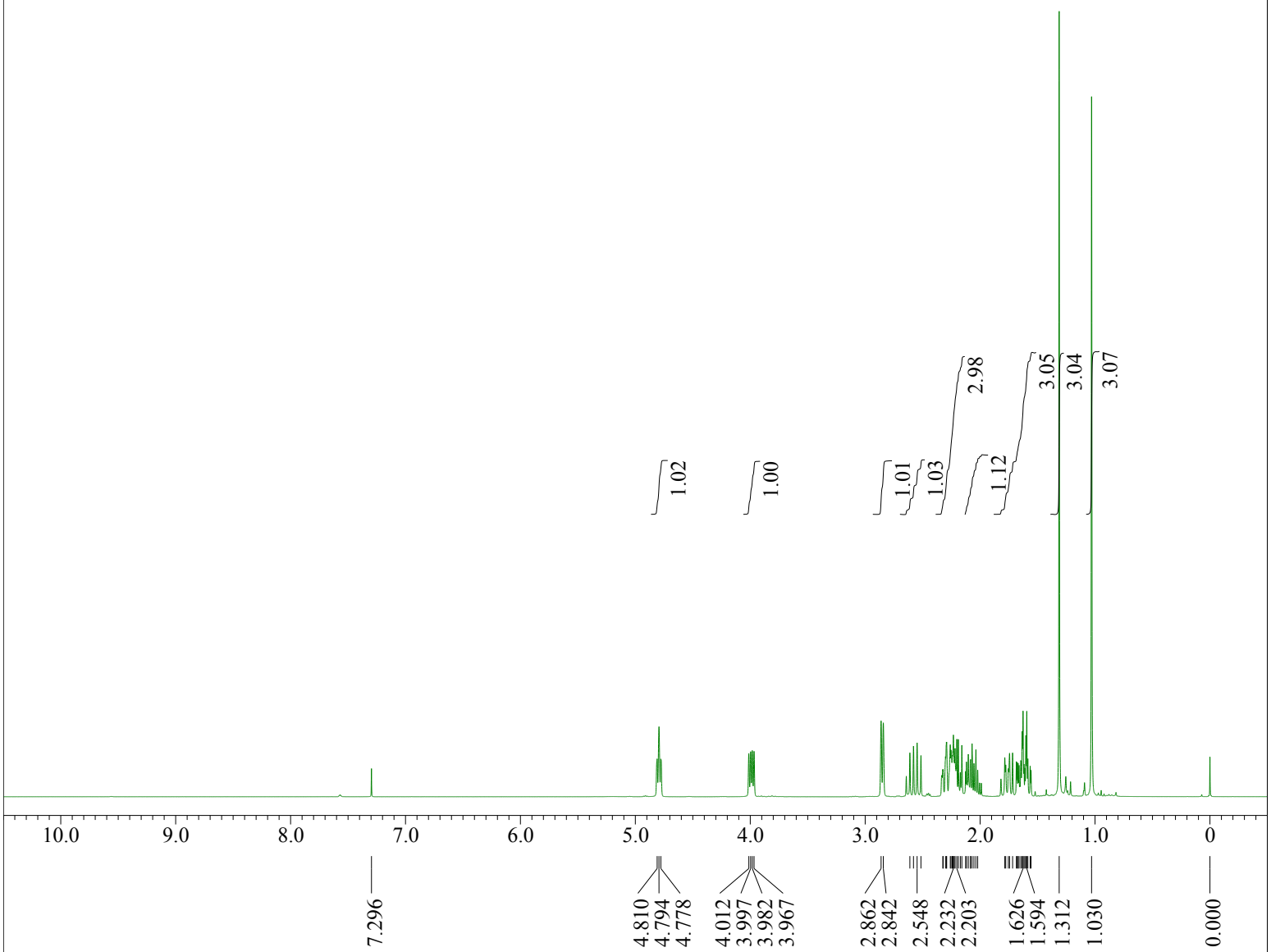
X : parts per Million : 13C

```
Filename       = Yakka-6_1790-6.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 15-NOV-2016 16:45:55
Revision_Time  = 15-NOV-2016 16:49:57
Current_Time   = 15-NOV-2016 16:50:36

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.76[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 2048
```



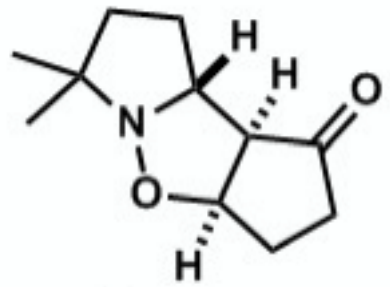
4k

Yakka-5_1450-8.jdf



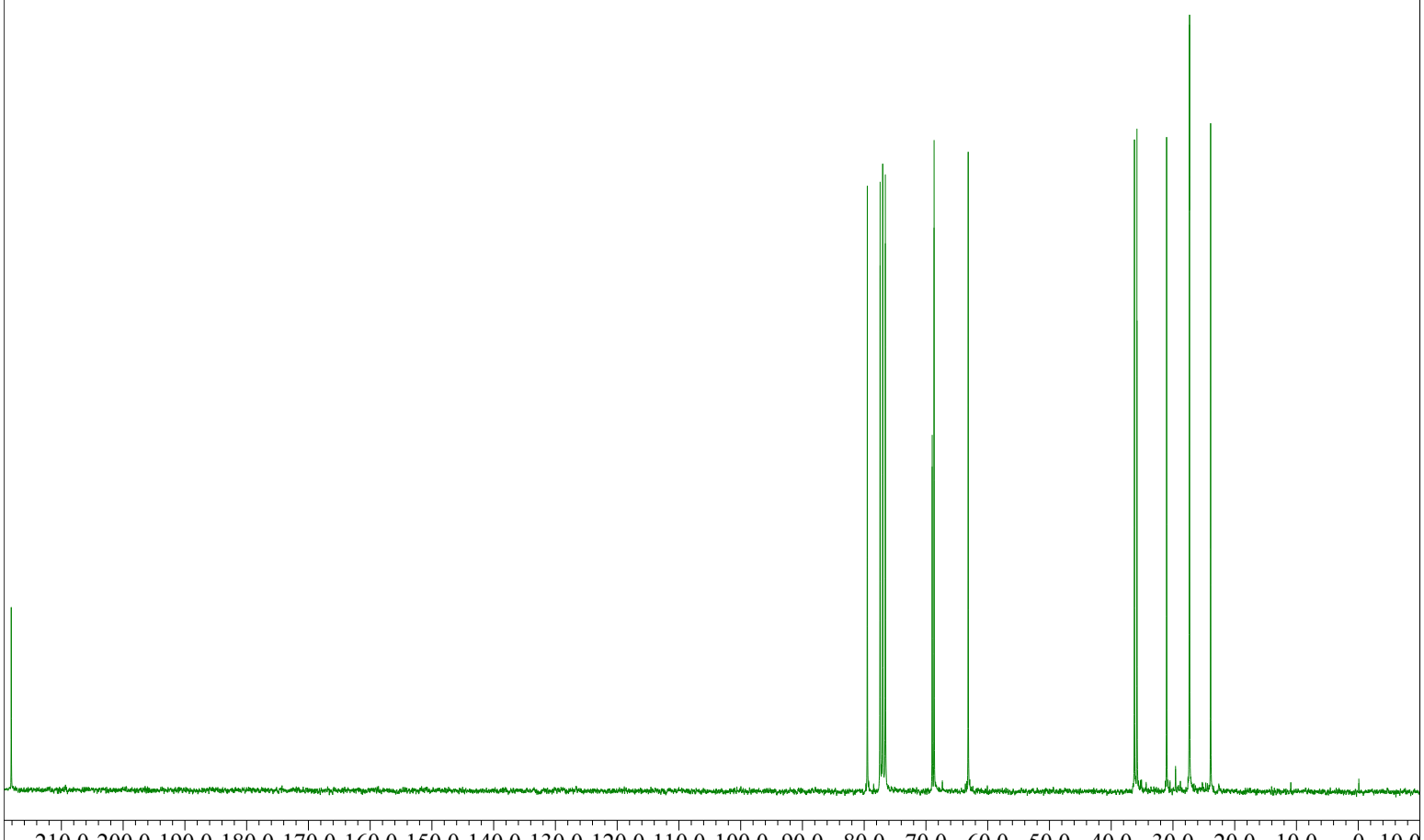
X : parts per Million : 1H

```
Filename       = Yakka-5_1450-8.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 22:13:51
Revision_Time  = 24-NOV-2016 22:17:35
Current_Time   = 24-NOV-2016 22:18:30

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 295.06[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```

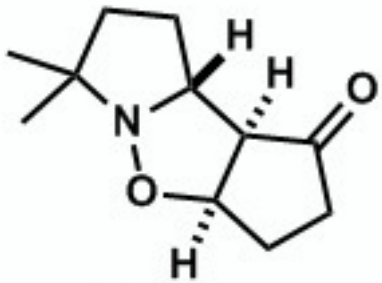


4n

Yakka-6_5280-3.jdf



```
Filename       = Yakka-6_5280-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 18:10:36
Revision_Time  = 24-NOV-2016 18:11:48
Current_Time   = 24-NOV-2016 18:12:25

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.66[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 2048
```



4n

210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0

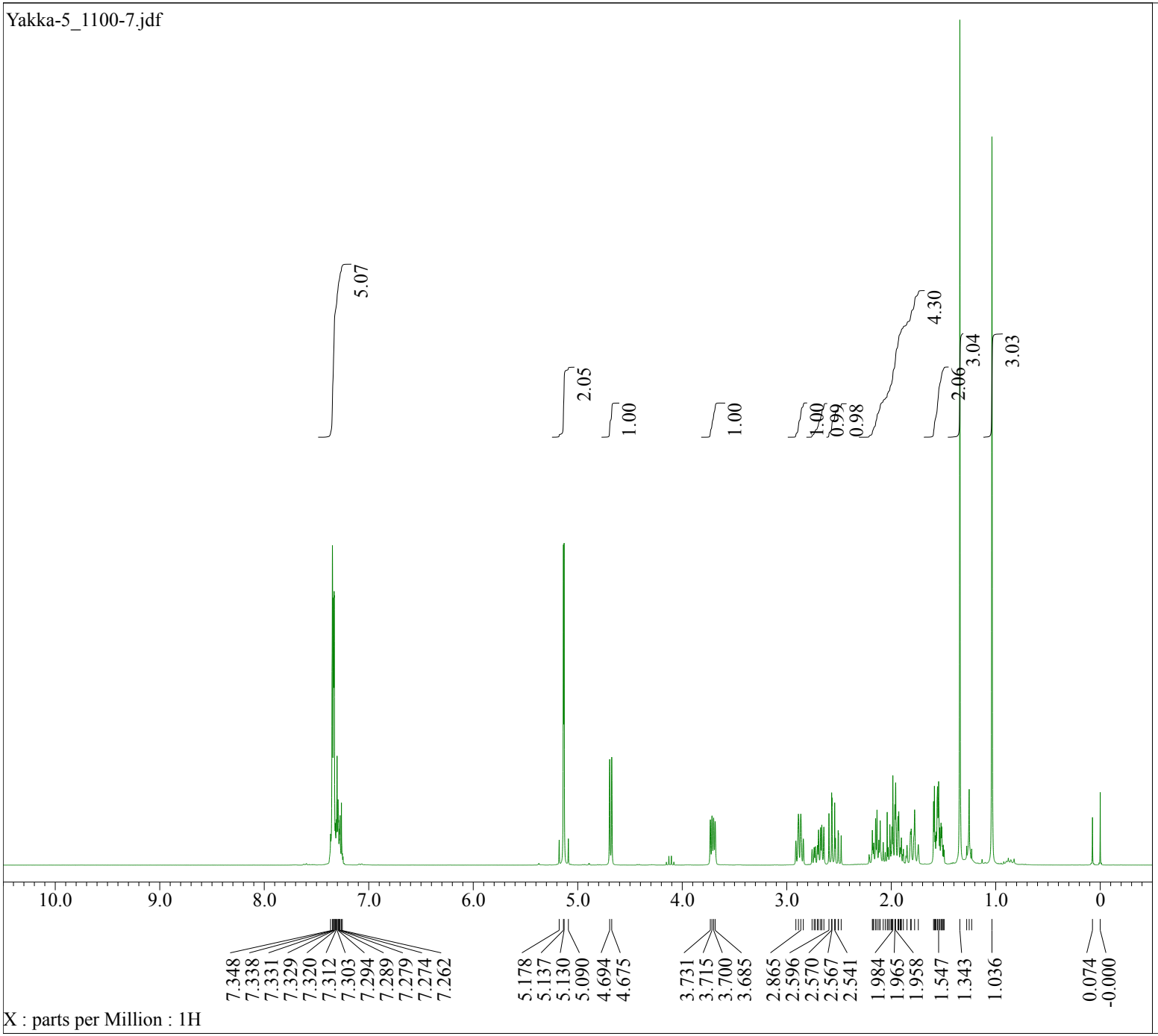
218.118

79.493
77.423
77.000
76.577
68.989
68.683
63.157

36.237
35.829
31.032
27.351
27.315
23.903

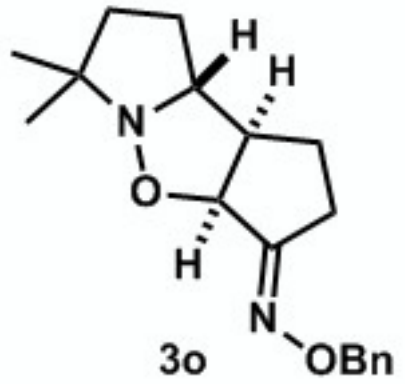
X: parts per Million : 13C

Yakka-5_1100-7.jdf



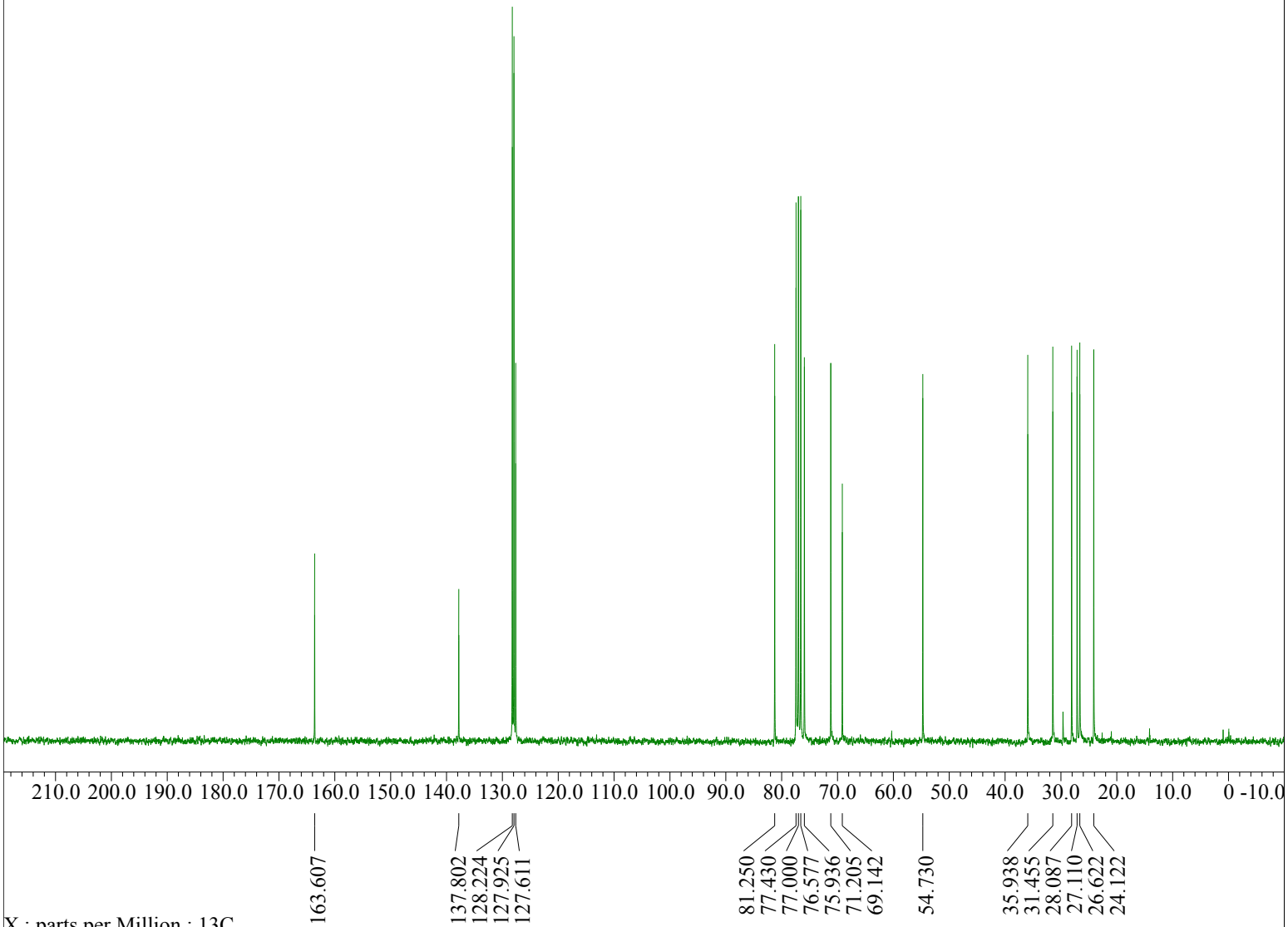
X : parts per Million : 1H

```
Filename       = Yakka-5_1100-7.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 17:45:40
Revision_Time  = 24-NOV-2016 17:49:08
Current_Time   = 24-NOV-2016 17:49:27

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.96[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



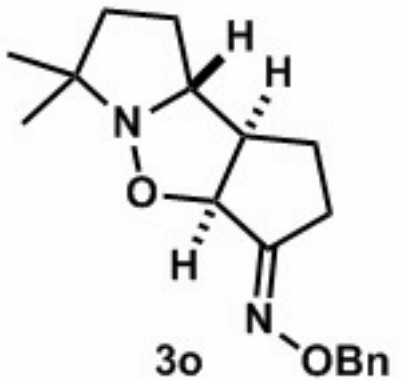
3o

Yakka-5_1101-3.jdf

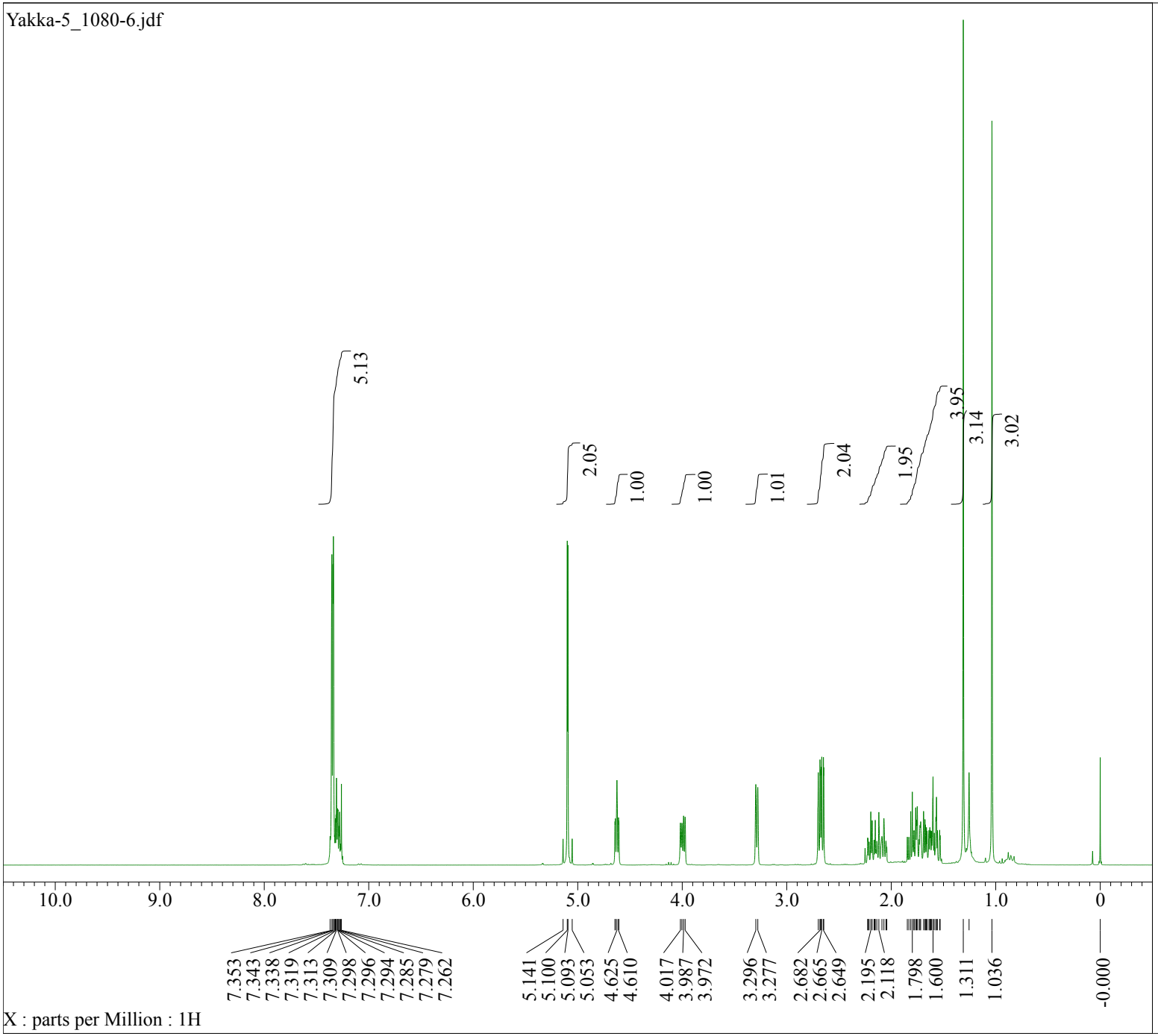


X : parts per Million : 13C

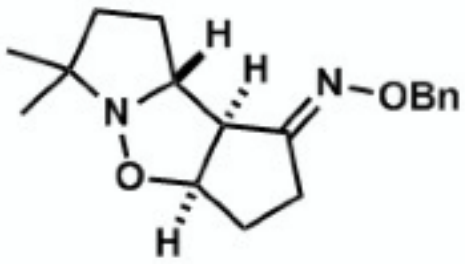
```
Filename       = Yakka-5_1101-3.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 17:53:14
Revision_Time  = 24-NOV-2016 17:53:52
Current_Time   = 24-NOV-2016 17:55:12

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.56[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 1024
```



3o

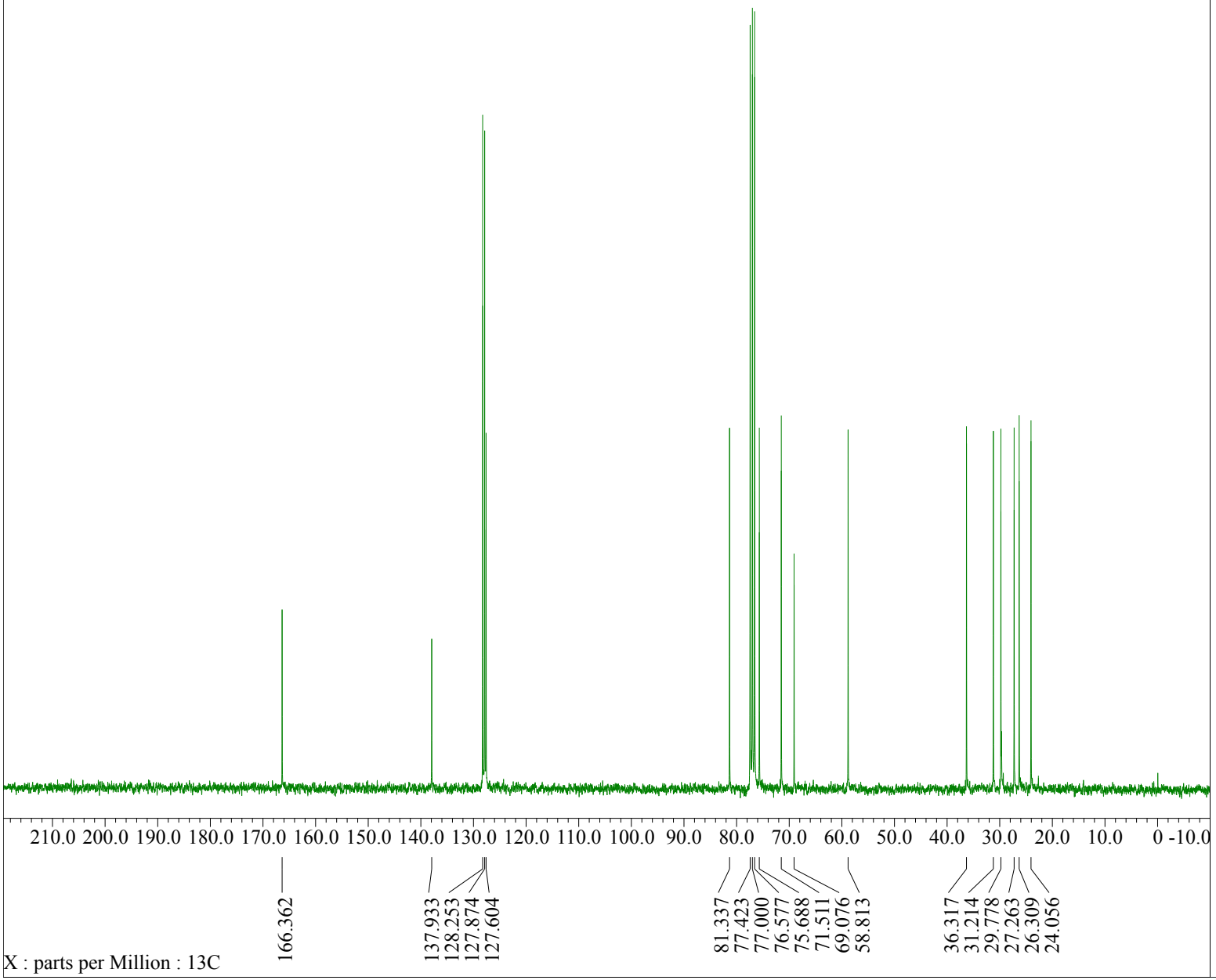



```
Filename       = Yakka-5_1080-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 17:58:00
Revision_Time  = 24-NOV-2016 18:02:25
Current_Time   = 24-NOV-2016 18:02:52

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 300.13185343[MHz]
X_Offset       = 1.85342561[kHz]
X_Sweep        = 6.18811881[kHz]

Temp_Get       = 294.96[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



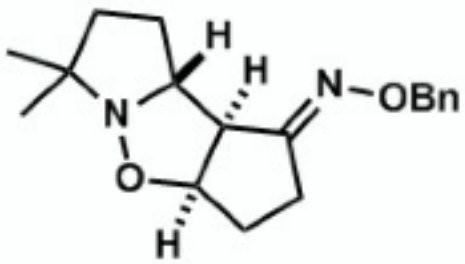
4o

Yakka-5_1082-4.jdf



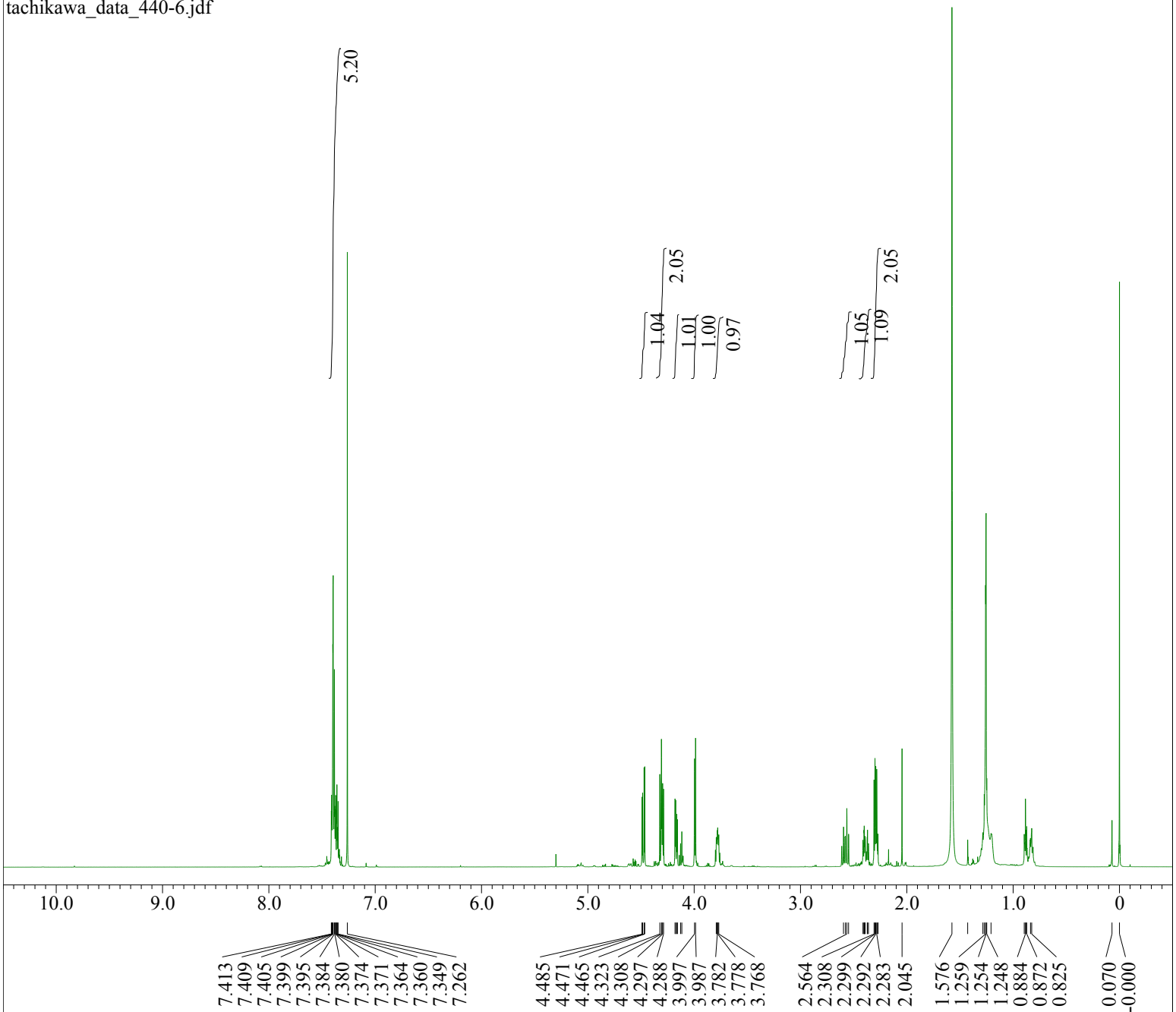
X : parts per Million : 13C

```
Filename       = Yakka-5_1082-4.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 18:04:04
Revision_Time  = 24-NOV-2016 18:06:35
Current_Time   = 24-NOV-2016 18:07:13

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 32768
Dim_Title      = 13C
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 75.4752953[MHz]
X_Offset       = 7.54630085[kHz]
X_Sweep        = 18.02884615[kHz]

Temp_Get       = 296.56[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 1024
```



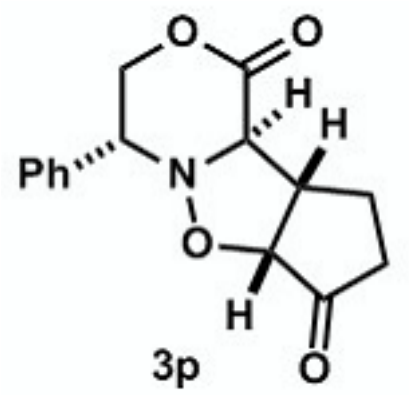
4o

tachikawa_data_440-6.jdf

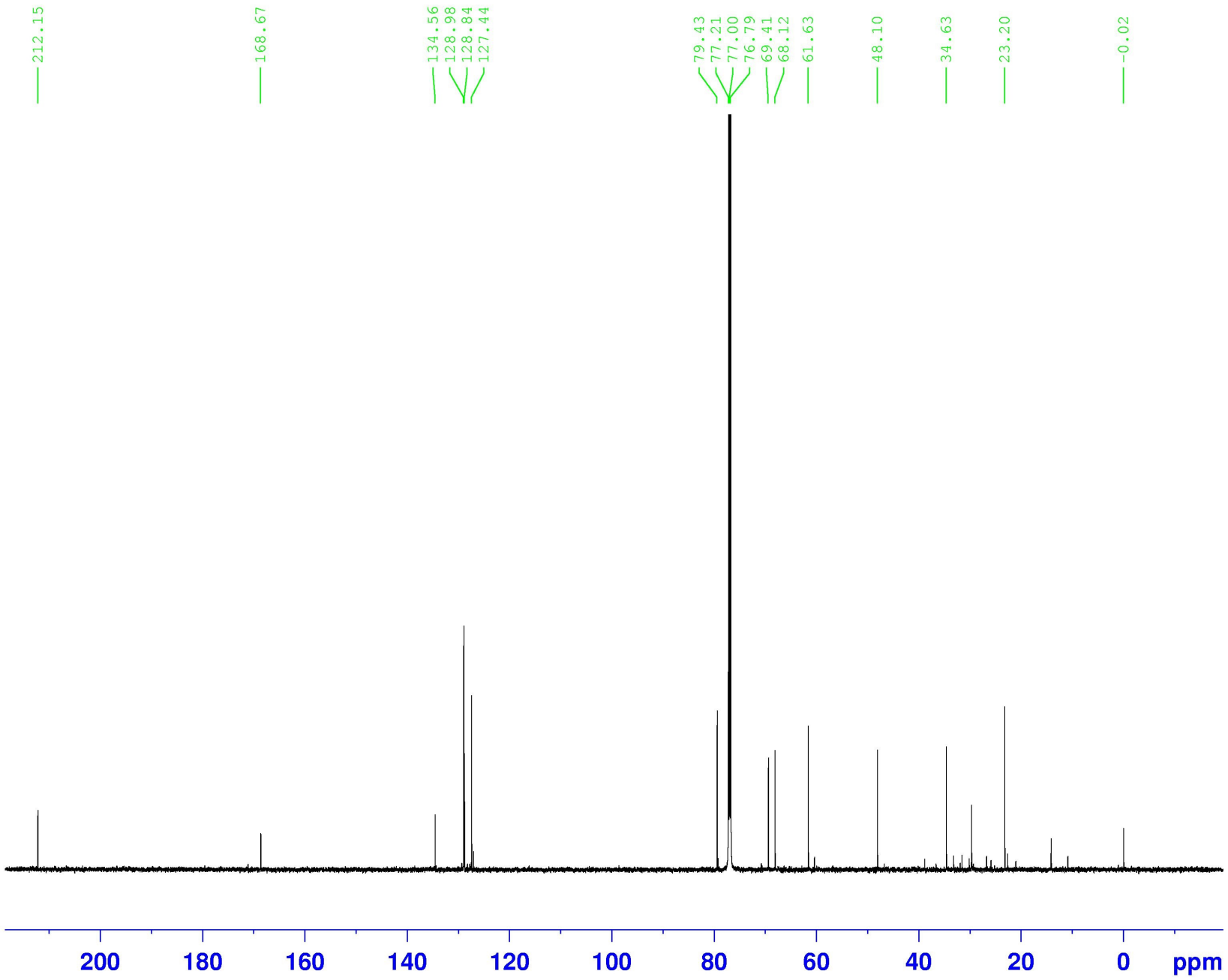


X : parts per Million : 1H

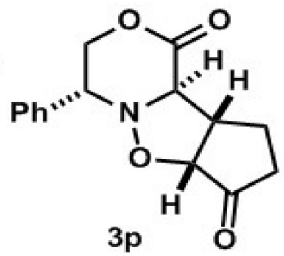
```
Filename       = tachikawa_data_440-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 22:40:10
Revision_Time  = 24-NOV-2016 22:45:46
Current_Time   = 24-NOV-2016 22:47:12

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 16384
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 600.13370605[MHz]
X_Offset       = 3.70604841[kHz]
X_Sweep        = 12.33552632[kHz]

Temp_Get       = 298.0027[K]
X_Points       = 16384
X_Prescans     = 2
Filter_Factor  = 1621
Scans          = 16
```



YH_tachikawa_6_20160909
A.carbon CDCl3 D:\\ rfia 2

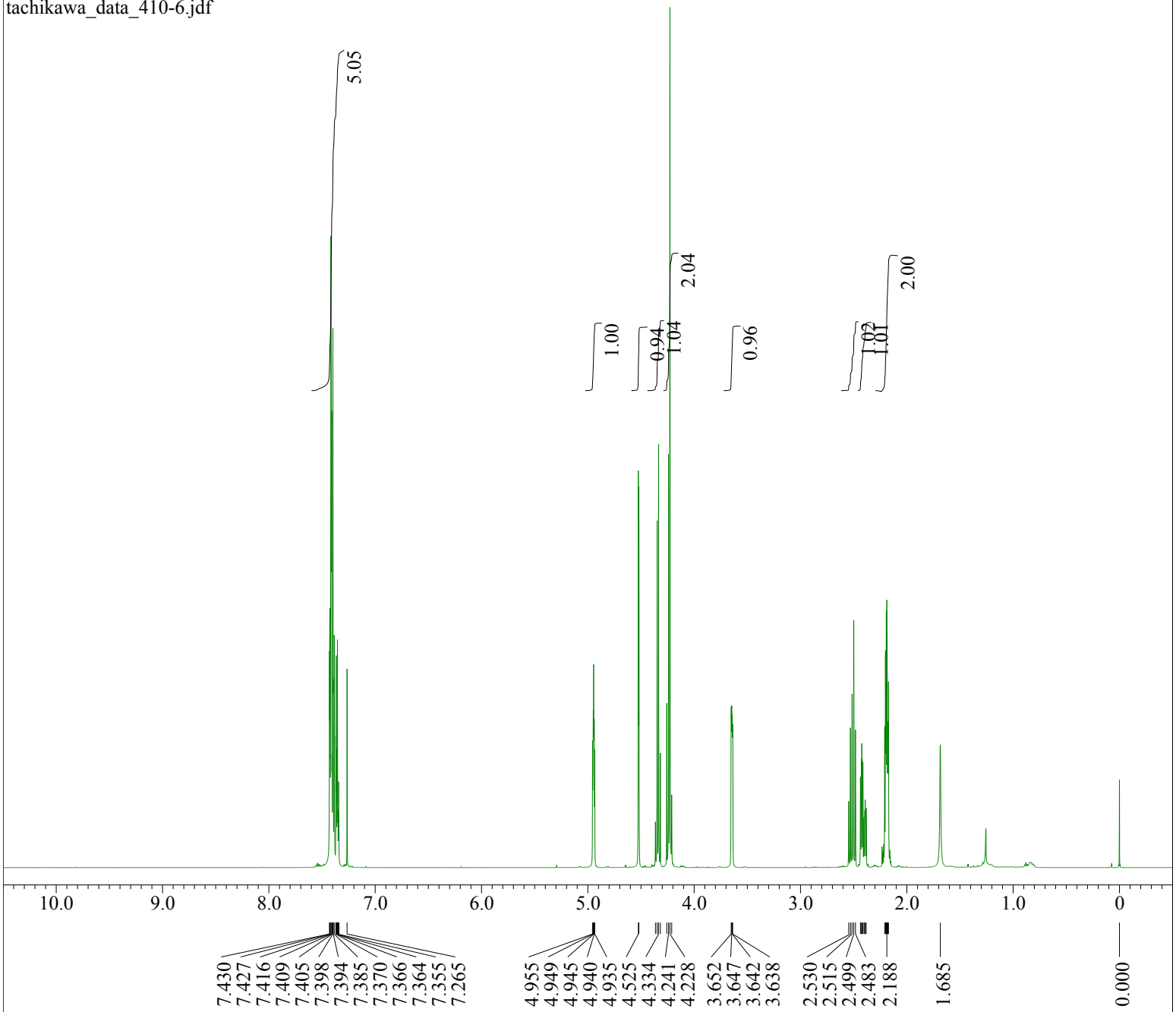



```
Current Data Parameters
NAME                  Hashimoto
EXPNO                       442
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20160909
Time                      20.44
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zgpg30
TD                       65356
SOLVENT                  CDCl3
NS                        4096
DS                           4
SWH                  36057.691 Hz
FIDRES                0.551712 Hz
AQ                    0.9062698 sec
RG                         2050
DW                       13.867 usec
DE                         6.50 usec
TE                        298.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                150.9178988 MHz
NUC1                        13C
P1                        15.00 usec
PLW1               102.00000000 W

======== CHANNEL f2 ========
SFO2                600.1324005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     70.00 usec
PLW2                 8.80000019 W
PLW12                0.09834400 W
PLW13                0.04818900 W

F2 - Processing parameters
SI                         32768
SF                  150.9028103 MHz
WDW                           EM
SSB               0
LB                         1.00 Hz
GB                0
PC                         1.40
```



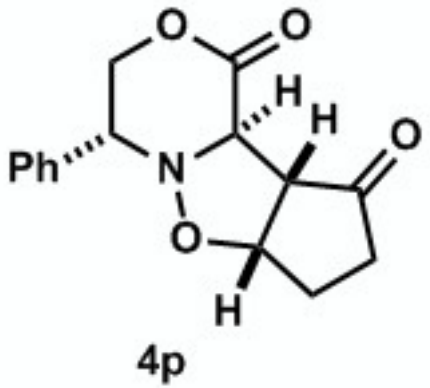
3p

tachikawa_data_410-6.jdf



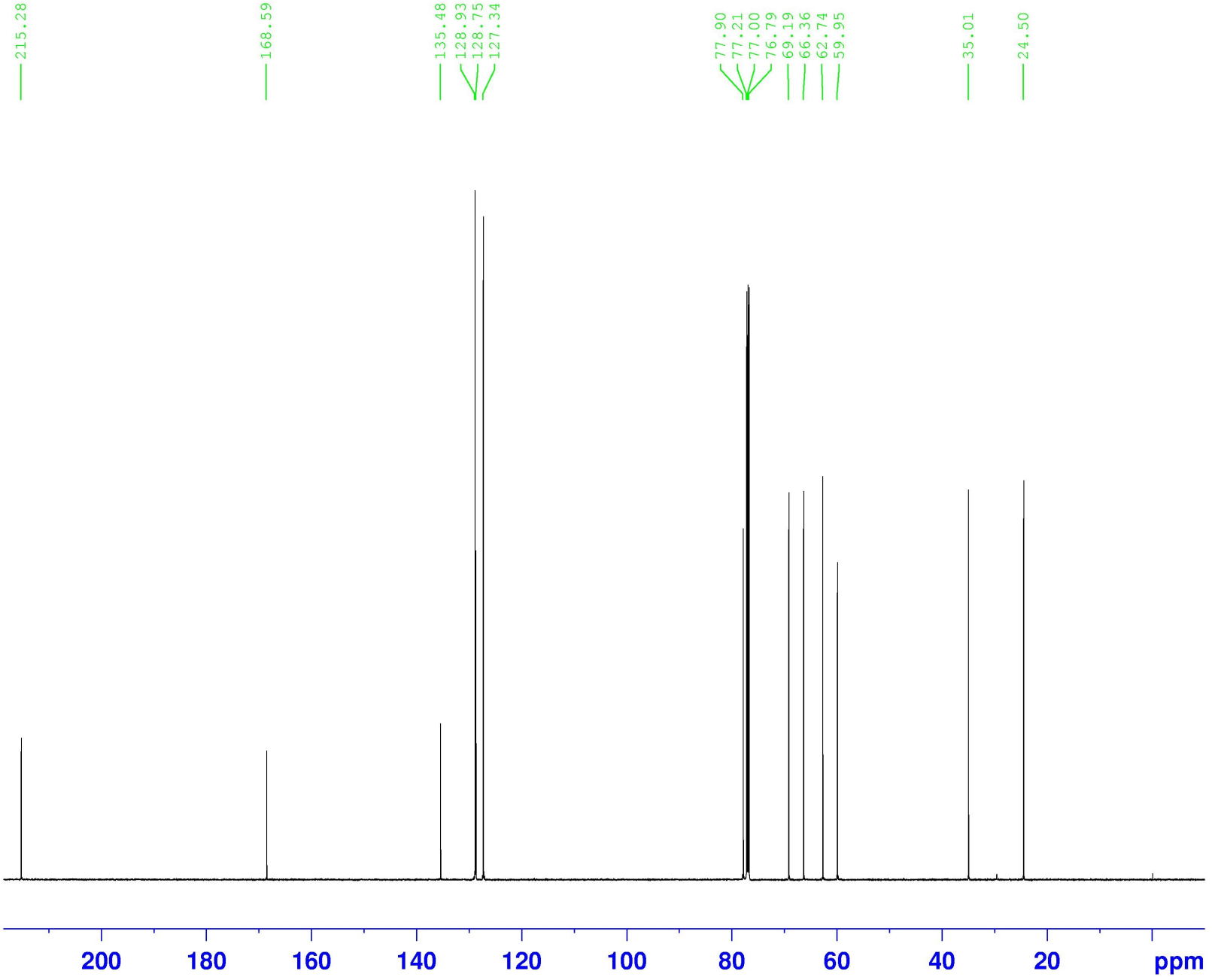
X : parts per Million : 1H

```
Filename       = tachikawa_data_410-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 22:36:01
Revision_Time  = 24-NOV-2016 22:38:22
Current_Time   = 24-NOV-2016 22:38:46

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 16384
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 600.13370605[MHz]
X_Offset       = 3.70604841[kHz]
X_Sweep        = 12.33552632[kHz]

Temp_Get       = 298.0027[K]
X_Points       = 16384
X_Prescans     = 2
Filter_Factor  = 1621
Scans          = 16
```

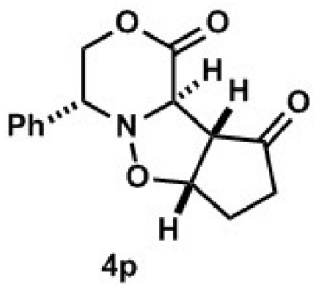


4p

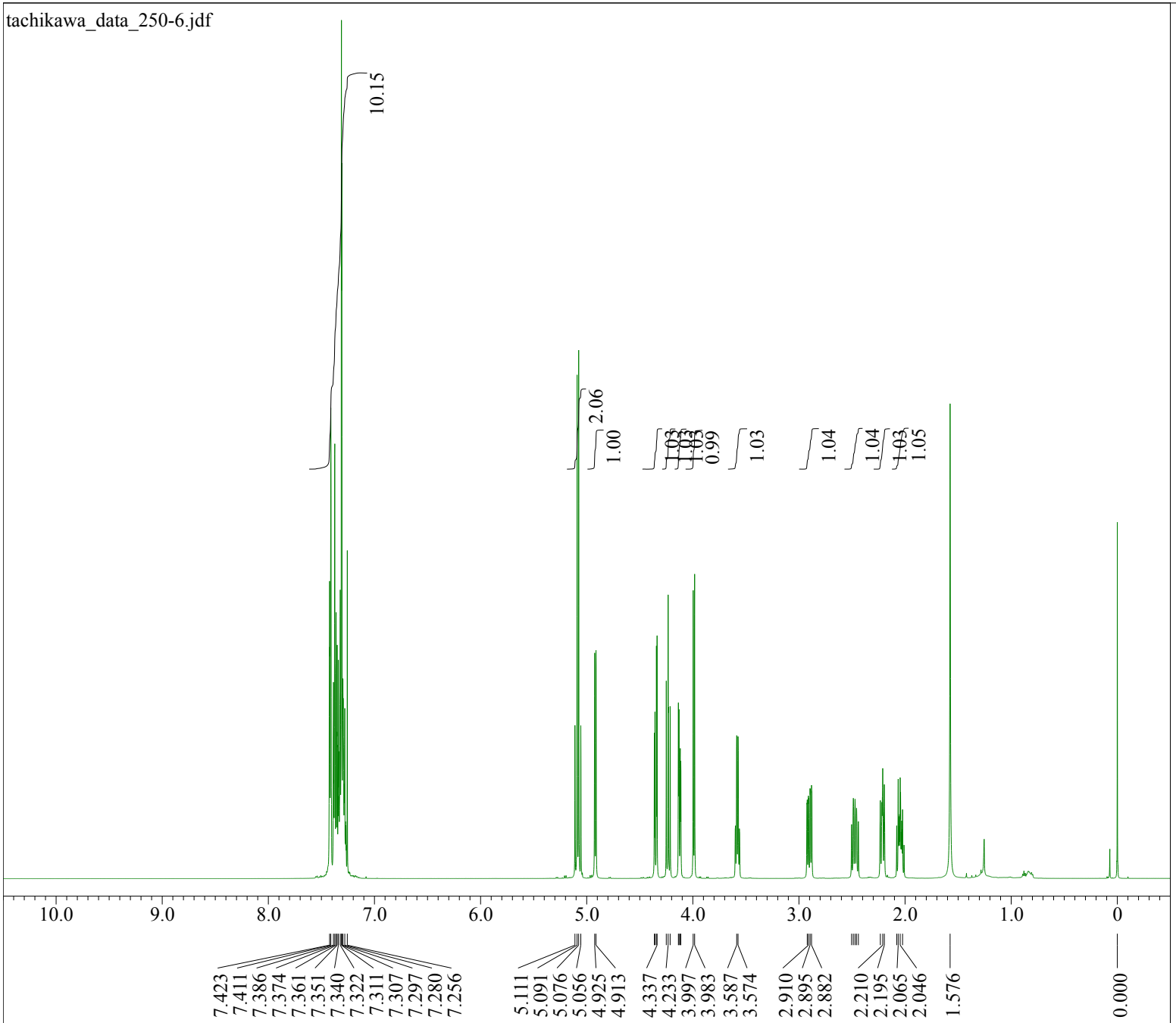
YH_tachikawa_3_data_20160909
A.carbon CDCl3 D:\\ rfia 11



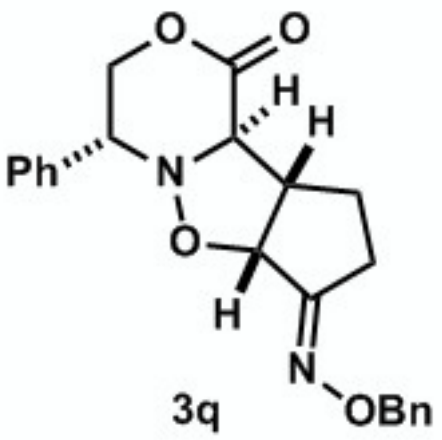

```
Current Data Parameters
NAME                  Hashimoto
EXPNO                       411
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20160909
Time                      13.46
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zgpg30
TD                       65356
SOLVENT                  CDCl3
NS                        1024
DS                           4
SWH                  36057.691 Hz
FIDRES               0.551712 Hz
AQ                   0.9062698 sec
RG                        2050
DW                      13.867 usec
DE                        6.50 usec
TE                       298.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               150.9178988 MHz
NUC1                       13C
P1                       15.00 usec
PLW1              102.00000000 W

======== CHANNEL f2 ========
SFO2               600.1324005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    70.00 usec
PLW2                8.80000019 W
PLW12               0.09834400 W
PLW13               0.04818900 W

F2 - Processing parameters
SI                        32768
SF                 150.9028180 MHz
WDW                          EM
SSB               0
LB                        1.00 Hz
GB                0
PC                        1.40
```



4p

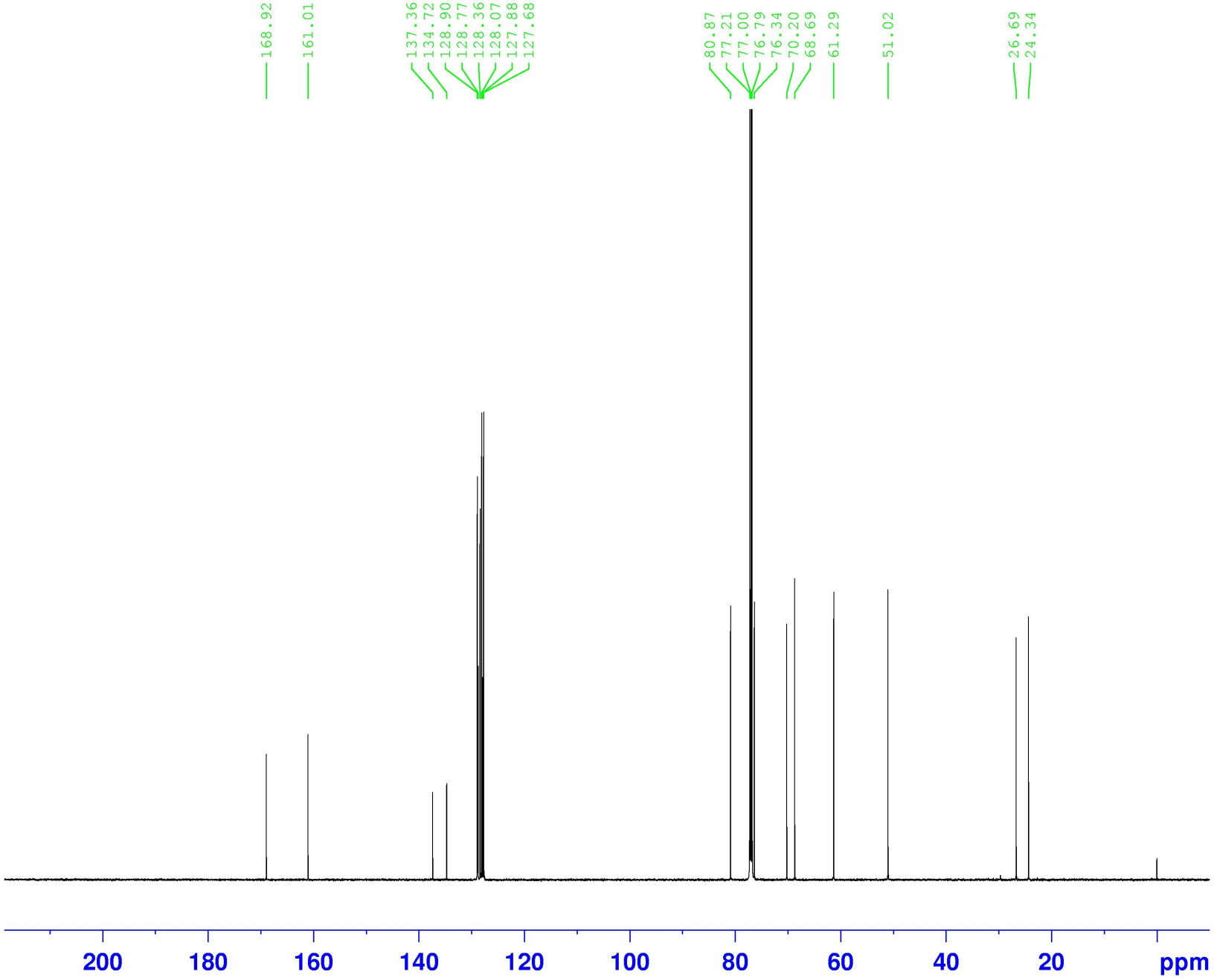



```
Filename       = tachikawa_data_250-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 22:21:57
Revision_Time  = 24-NOV-2016 22:25:09
Current_Time   = 24-NOV-2016 22:25:35

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 16384
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 600.13370605[MHz]
X_Offset       = 3.70604841[kHz]
X_Sweep        = 12.33552632[kHz]

Temp_Get       = 298.0027[K]
X_Points       = 16384
X_Prescans     = 2
Filter_Factor  = 1621
Scans          = 16
```

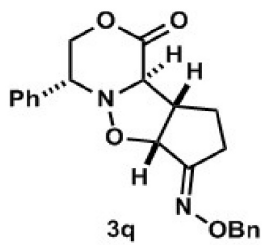


3q

YH_0229_1
A.carbon CDCl3 D:\\ rfia 51

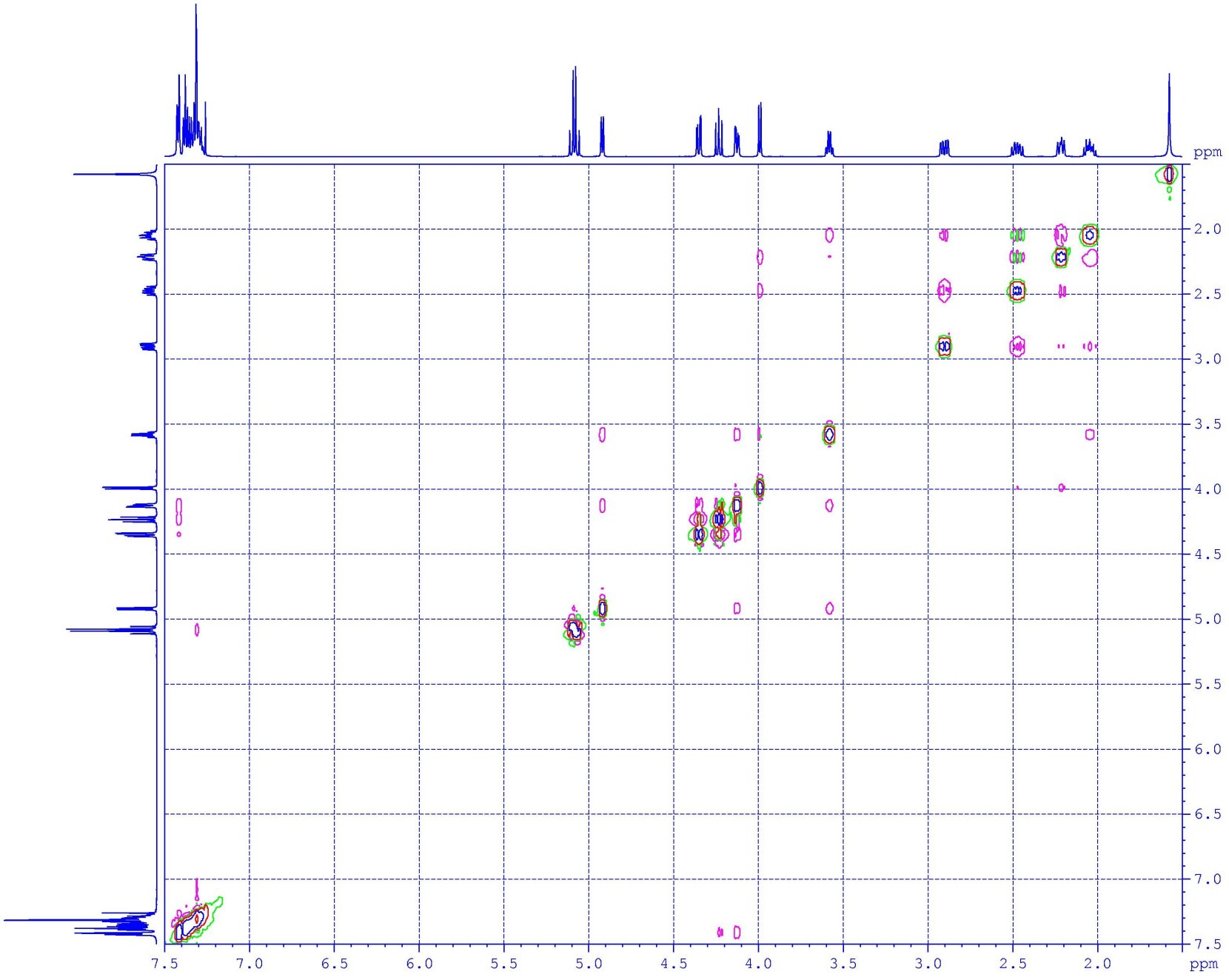



```
Current Data Parameters
NAME            Hashimoto
EXPNO                 253
PROCNO                  1

F2 - Acquisition Parameters
Date_            20160803
Time                18.49
INSTRUM             spect
PROBHD   5 mm CPTCI 1H-
PULPROG           zgpg30
TD                 65356
SOLVENT            CDCl3
NS                  4096
DS                     4
SWH           36057.691 Hz
FIDRES         0.551712 Hz
AQ             0.9062698 sec
RG                  2050
DW               13.867 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        150.9178988 MHz
NUC1                13C
P1                15.00 usec
PLW1       102.00000000 W

======== CHANNEL f2 ========
SFO2        600.1324005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             70.00 usec
PLW2          8.80000019 W
PLW12         0.09834400 W
PLW13         0.04818900 W

F2 - Processing parameters
SI                 32768
SF          150.9028125 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

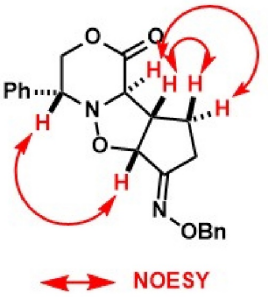


3q

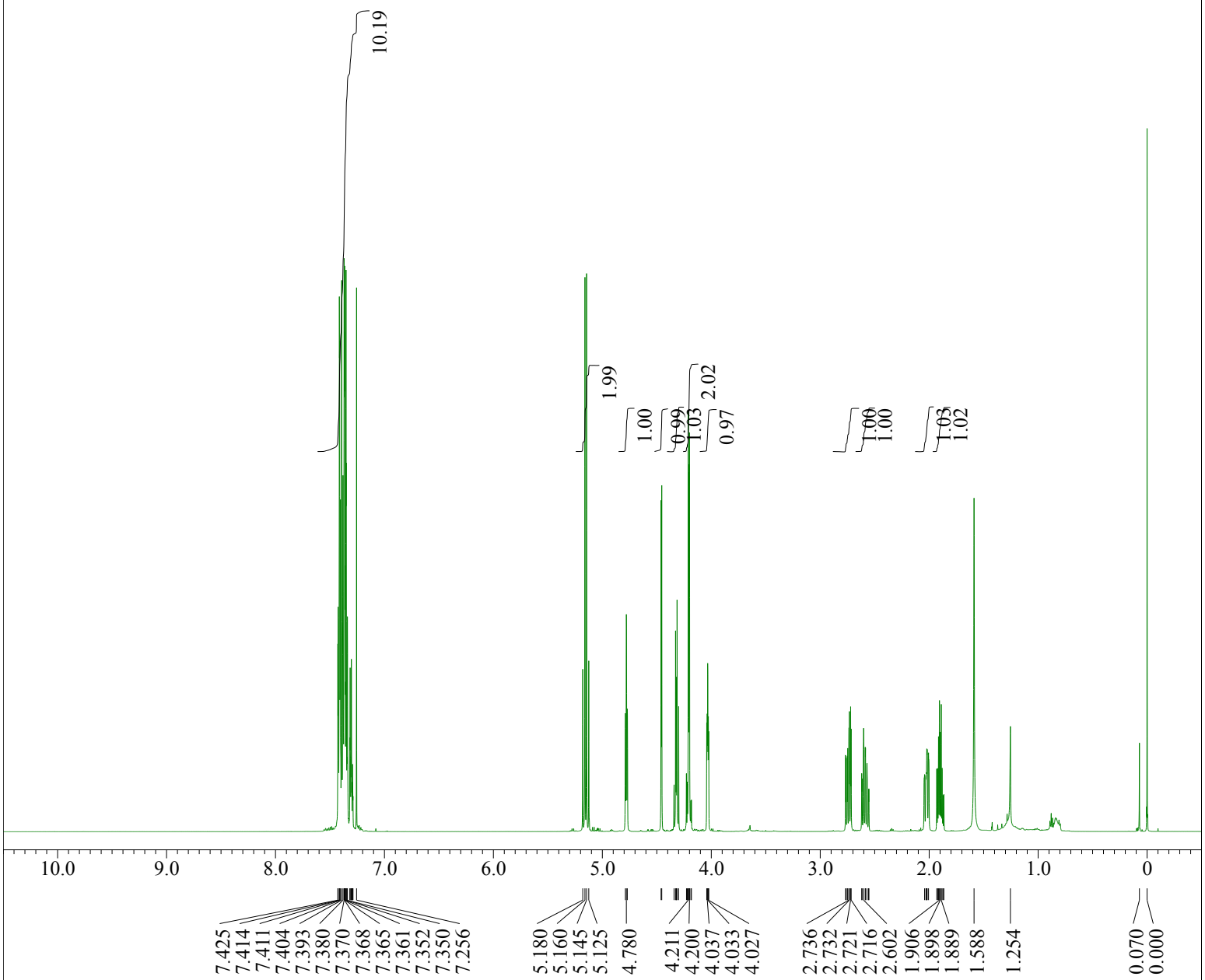
YH_0229_1
B.noesy.600m CDCl3 D:\\ rfia 51




```
Current Data Parameters
NAME            Hashimoto
EXPNO                 252
PROCNO                  1

F2 - Acquisition Parameters
Date_              20160803
Time                 12.13
INSTRUM              spect
PROBHD    5 mm CPTCI 1H-
PULPROG      noesygpphpp
TD                    2048
SOLVENT               CDCl3
NS                      16
DS                      16
SWH               5376.344 Hz
FIDRES            2.625168 Hz
AQ                0.1904640 sec
RG                      16
DW                  93.000 usec
DE                  10.00 usec
TE                  298.0 K
D0                0.00008358 sec
D1                1.97665298 sec
D8                0.60000002 sec
D11               0.03000000 sec
D12               0.00002000 sec
D16               0.00020000 sec
IN0               0.00018600 sec

======== CHANNEL f1 ========
SFO1          600.1323679 MHz
NUC1                    1H
P1                   7.40 usec
P2                  14.80 usec
P17               2500.00 usec
PLW1           8.80000019 W
PLW10          0.71284997 W

====== GRADIENT CHANNEL =====
GPNAM[1]         SINE.100
GPZ1                40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
TD                    256
SFO1             600.1324 MHz
FIDRES          21.001345 Hz
SW                   8.959 ppm
FnMODE        States-TPPI

F2 - Processing parameters
SI                   1024
SF            600.1300122 MHz
WDW                  QSINE
SSB                      2
LB          0 Hz
GB          0
PC                    1.00

F1 - Processing parameters
SI                   1024
MC2           States-TPPI
SF            600.1300122 MHz
WDW                  QSINE
SSB                      2
LB          0 Hz
GB          0
```

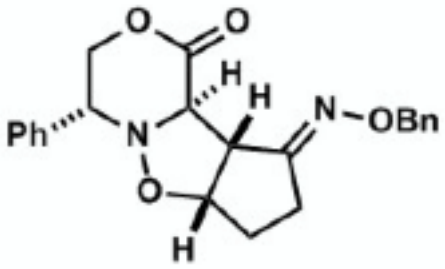


tachikawa_data_260-6.jdf



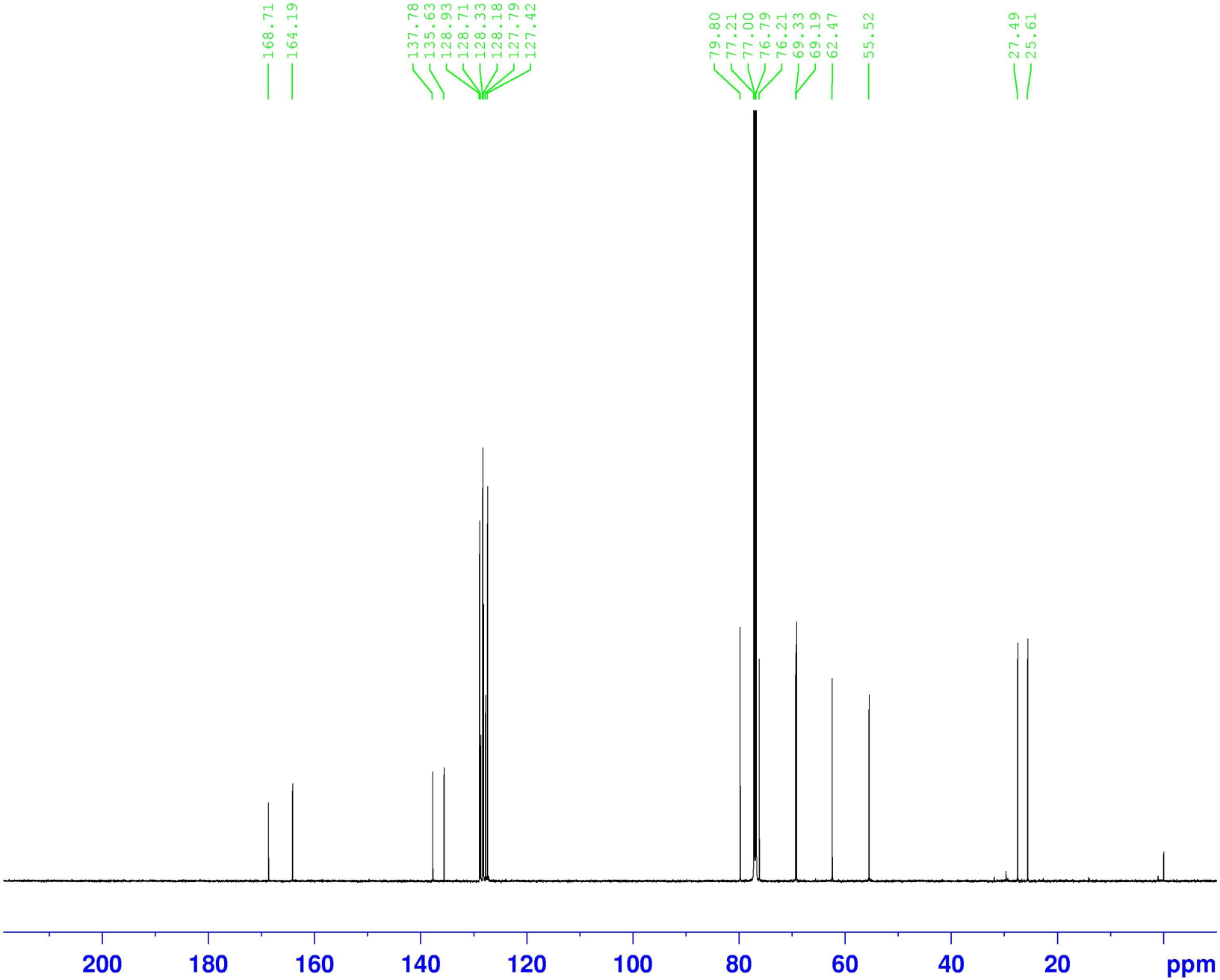
X : parts per Million : 1H

```
Filename       = tachikawa_data_260-6.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 24-NOV-2016 22:30:49
Revision_Time  = 24-NOV-2016 22:33:49
Current_Time   = 24-NOV-2016 22:34:17

Comment        = Parameter file, TOPSPIN  Vers
Data_Format    = 1D COMPLEX
Dim_Size       = 16384
Dim_Title      = 1H
Dim_Units      = [ppm]
Dimensions     = X
Spectrometer   = BRUKER_DMX_NMR

X_Freq         = 600.13370605[MHz]
X_Offset       = 3.70604841[kHz]
X_Sweep        = 12.33552632[kHz]

Temp_Get       = 298.0027[K]
X_Points       = 16384
X_Prescans     = 2
Filter_Factor  = 1621
Scans          = 16
```

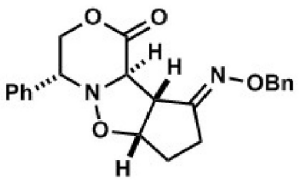


4q

YH_0229_2
A.carbon CDCl3 D:\\ rfia 52




```
Current Data Parameters
NAME                  Hashimoto
EXPNO                       263
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20160804
Time                       4.19
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zgpg30
TD                         65356
SOLVENT                  CDCl3
NS                          4096
DS                             4
SWH                  36057.691 Hz
FIDRES                0.551712 Hz
AQ                    0.9062698 sec
RG                          2050
DW                     13.867 usec
DE                      6.50 usec
TE                      298.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                150.9178988 MHz
NUC1                        13C
P1                       15.00 usec
PLW1              102.00000000 W

======== CHANNEL f2 ========
SFO2                600.1324005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    70.00 usec
PLW2                 8.80000019 W
PLW12                0.09834400 W
PLW13                0.04818900 W

F2 - Processing parameters
SI                         32768
SF                  150.9028125 MHz
WDW                           EM
SSB             0
LB                       1.00 Hz
GB              0
PC                       1.40
```



4q