Inverse-electron-demand Diels–Alder reaction of α,β-unsaturated hydrazones with 3-methoxycarbonyl α-pyrone

Supporting Information

Yoshimitsu Hashimoto, Ryo Abe, Nobuyoshi Morita and Osamu Tamura*

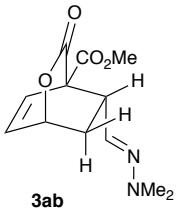
Showa Pharmaceutical University, 3-3165 Higashi-Tamagawagakuen, Machida, Tokyo 194-8543, Japan

Contents

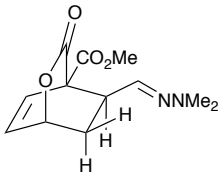
General procedure

^{1}H and ^{13}C NMR spectra were recorded with JEOL JNM-AL300, JEOL JNM-ECZ400S, BRUKER AV300M or BRUKER AV600 spectrometer at room temperature, with tetramethylsilane (δ = 0) as an internal standard ($CDCl_3$ solution). Chemical shifts were expressed 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 F254 were used for column chromatography and thin layer chromatography (TLC), respectively.

Synthesis and Data



3ab



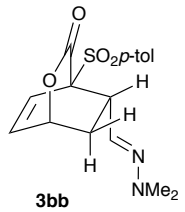
4ab

endo-cycloadduct **3ab** and its *exo*-isomer **4ab**

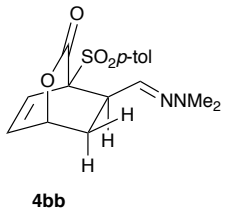
A solution of α-pyrone **1a** (50.0 mg, 0.324 mmol), acrolein hydrazone **2b** (63.7 mg, 0.649 mmol) and $Eu(hfc)_3$ (38.7 mg, 32.4 μmol) in 1,2-dichloroethane (1.6 mL) was stirred at room temperature for 26 hours. After the reaction mixture was concentrated under reduced pressure, the residue was purified by column chromatography on silica gel ($CHCl_3$: acetone = 10 : 1) to afford *endo* adduct **3ab** (65.1 mg, 80%) as a pale yellow solid and *exo* adduct **4ab** (5.3 mg, 6%) as a brown amorphous.

3ab: mp 54.0-54.4 °C; IR (KBr) 2953, 1751, 1277, 1105, 1070 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.78 (1H, brd, *J* = 7.8 Hz), 6.50 (1H, dd, *J* = 7.8, 5.1 Hz), 6.21 (1H, d, *J* = 3.9 Hz), 5.29–5.21 (1H, m), 3.88 (3H, s), 3.48 (1H, dt, *J* = 9.6, 3.9 Hz), 2.68 (6H, s), 2.55 (1H, ddd, *J* = 13.2, 9.6, 3.9 Hz), 1.96 (1H, ddd, *J* = 13.2, 3.9, 1.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3, 168.3, 131.8, 131.1, 130.4, 74.4, 58.3, 52.8, 42.8, 37.6, 31.0; HRMS (FAB) *m*/*z* calcd for $C_{12}H_{17}N_2O_4$ $[M+H]^+$ 253.1188, found 253.1193.

4ab: IR (KBr) 2955, 1746, 1275, 1076 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.75 (1H, dd, *J* = 7.8, 1.8 Hz), 6.60 (1H, dd, *J* = 7.8, 5.1 Hz), 6.58 (1H, d, *J* = 6.6 Hz), 5.29–5.20 (1H, m), 3.85 (3H, s), 2.96 (1H, ddd, *J* = 10.5, 6.6, 4.6 Hz), 2.76 (6H, s), 2.42 (1H, dt, *J* =13.5, 4.6 Hz), 2.06 (1H, ddd, *J* = 13.5, 10.5, 1.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.4, 168.2, 134.6, 132.8, 132.2, 74.1, 57.9, 52.7, 43.0, 39.9, 32.2; HRMS (FAB) *m*/*z* calcd for $C_{12}H_{17}N_2O_4$ $[M+H]^+$ 253.1188, found 253.1200.



3bb



4bb

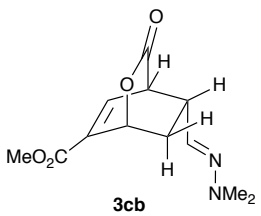
4-*p*-toluenesulfonyl-*endo*-cycloadduct **3bb** and its *exo*-isomer **4bb**

A solution of 3-Ts-α-pyrone **1b** (50.0 mg, 0.200 mmol), acrolein hydrazone **2b** (44.9 mg, 0.400 mmol) and Eu(hfc)$_3$ (23.9 mg, 20.0 μmol) in 1,2-dichloroethane (1.0 mL) was stirred at 50 °C for 10 hours. After the reaction mixture was concentrated under reduced pressure, the residue was purified by column chromatography on silica gel (hexane : AcOEt = 1 : 1 to 1 : 2) to afford a mixture of *endo* adduct **3bb** and *exo* adduct **4bb** (69.2 mg, 99%, **3bb** : **4bb** = 83 : 17) as pale yellow amorphous.

Analytical samples were obtained by separation of the mixture with PTLC (toluene : Et_2O = 1 : 5 (×2)) to afford **3bb** and **4bb** both as a white solid.

3bb: mp 129-130 °C (decomp.); IR (KBr) 2955, 2860, 1759, 1597, 1319, 1153, 1043 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05 (2H, d, *J* = 8.1 Hz), 7.34 (2H, d, *J* = 8.1 Hz), 6.86 (1H, d, *J* = 7.8 Hz), 6.75 (1H, dd, *J* = 7.8, 4.8 Hz), 6.63 (1H, d, *J* = 6.0 Hz), 5.20–5.15 (1H, m), 3.34 (1H, ddd, *J* = 9.6, 6.0, 3.0 Hz), 2.83 (6H, s), 2.52 (1H, ddd, *J* = 13.2, 9.6, 3.6 Hz), 2.44 (3H, s), 2.10 (1H, ddd, *J* = 13.2, 3.0, 1.2 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.6, 145.5, 134.0, 133.4, 132.4, 131.4, 129.1, 127.2, 73.3, 73.2, 42.9, 37.9, 32.5, 21.7; HRMS (ESI) *m*/*z* calcd for $C_{17}H_{20}N_2O_4NaS$ $[M+Na]^+$ 371.1042, found 371.1028.

4bb: mp 137-139 °C (decomp.); IR (KBr) 2955, 2925, 2858, 1763, 1597, 1367, 1155, 1074, 1045 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.06 (2H, d, *J* = 8.4 Hz), 7.34 (2H, d, *J* = 8.4 Hz), 6.63 (1H, dd, *J* = 8.4, 1.8 Hz), 6.58 (1H, dd, *J* = 8.4, 5.4 Hz), 6.51 (1H, d, *J* = 6.3 Hz), 5.20–5.13 (1H, m), 3.36 (1H, ddd, *J* = 10.2, 6.3, 4.8 Hz), 2.77 (6H, s), 2.52 (1H, dt, *J* = 13.2, 4.8 Hz), 2.44 (3H, s), 2.10 (1H, ddd, *J* = 13.2, 10.2, 1.2 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 165.7, 145.5, 134.3, 133.3, 131.34, 131.30, 130.4, 129.4, 73.6, 73.4, 42.6, 39.3, 33.5, 21.7; HRMS (ESI) *m*/*z* calcd for $C_{17}H_{20}N_2O_4NaS$ $[M+Na]^+$ 371.1042, found 371.1040.

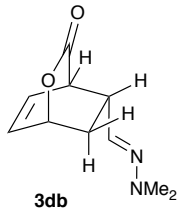


3cb

2-methoxycarbonyl-*exo*-cycloadduct **4cb**

A solution of methyl coumalate **1c** (100 mg, 0.648 mmol), acrolein hydrazone **2b** (127 mg, 1.30 mmol) and Eu(hfc)$_3$ (77.4 mg, 64.8 μmol) in 1,2-dichloroethane (3.2 mL) was stirred at room temperature for 48 hours. After the reaction mixture was concentrated under reduced pressure, the residue was purified by column chromatography on silica gel (hexane : AcOEt = 1 : 1) and ($CHCl_3$: acetone = 10 : 1) to afford *exo* adduct **4cb** (3.1 mg, 2%) as yellow oil.

IR (NaCl) 2953, 2856, 1764, 1717, 1439, 1267, 1013 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41 (1H, dd, *J* = 6.3, 2.1 Hz), 6.33 (1H, d, *J* = 6.3 Hz), 5.71 (1H, ddd, *J* = 3.9, 2.1, 1.8 Hz), 3.82 (3H, s), 3.65 (1H, dd, *J* = 6.3, 2.4 Hz), 2.80–2.71 (1H, m), 2.78 (6H, s), 2.31 (1H, ddd, *J* = 14.1, 5.4, 3.9 Hz), 2.01 (1H, ddd, *J* = 14.1, 10.5, 1.8 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 162.6, 141.1, 136.6, 132.9, 73.7, 52.2, 47.5, 42.8, 36.8, 30.4; HRMS (FAB) *m*/*z* calcd for $C_{12}H_{17}N_2O_4$ $[M+H]^+$ 253.1188, found 253.1182.

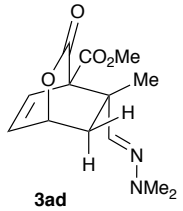


3db

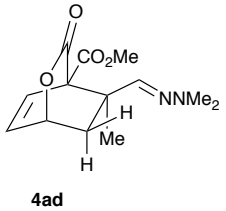
endo-cycloadduct **3db**

A solution of α-pyrone **1d** (50.0 mg, 0.520 mmol), acrolein hydrazone **2b** (102 mg, 1.04 mmol) and Eu(hfc)$_3$ (62.1 mg, 52.0 μmol) in 1,2-dichloroethane (2.6 mL) was stirred 80 °C for 40 hours. After the reaction mixture was concentrated under reduced pressure, the residue was purified by column chromatography on silica gel (hexane : AcOEt = 1 : 1) to afford *endo* adduct **3bd** (9.3 mg, 9%) as a brown oil. Unreacted **1d** was recovered (39.2 mg, 78%).

IR (NaCl) 2954, 2858, 1755, 1747, 1364, 1182, 1015 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.53 (1H, ddd, *J* = 7.8, 7.2, 1.5 Hz), 6.39 (1H, ddd, *J* = 7.8, 6.0, 1.5 Hz), 6.26 (1H, d, *J* = 4.8 Hz), 5.24 (1H, ddd, *J* = 7.2, 3.9, 1.5 Hz), 3.67 (1H, ddd, *J* = 6.0, 2.7, 1.5 Hz), 3.06–2.95 (1H, m), 2.73 (6H, s), 2.46 (1H, ddd, *J* = 13.5, 9.3, 3.9 Hz), 1.86 (1H, ddd, *J* = 13.5, 3.9, 1.5 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.6, 134.7, 132.4, 130.1, 74.3, 45.4, 42.9, 34.8, 30.5; HRMS (FAB) *m*/*z* calcd for $C_{10}H_{15}N_2O_2$ $[M+H]^+$ 195.1134, found 195.1140.



3ad



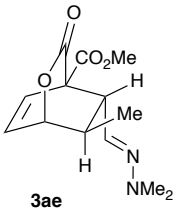
4ad

5-methyl-*endo*-cycloadduct **3ad** and its *exo*-isomer **4ad**

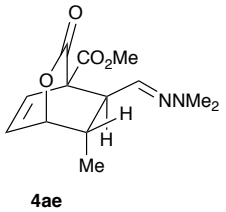
A solution of α-pyrone **1a** (50.0 mg, 0.324 mmol), methacrolein hydrazone **2d** (72.8 mg, 0.649 mmol) and Eu(hfc)$_3$ (38.7 mg, 32.4 μmol) in 1,2-dichloroethane (1.6 mL) was stirred at 0 °C for 4 hour and at room

temperature for 4.5 hours. After the reaction mixture was concentrated under reduced pressure, the residue was purified by column chromatography on silica gel (hexane : AcOEt = 1 : 1) to afford a mixture of *endo* adduct **3ad** and *exo* adduct **4ad** (57.7 mg, 67%, **3ad** : **4ad** = 52 : 48) as yellow oil.

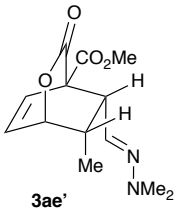
IR (NaCl) 2955, 2856, 1766, 1758, 1744, 1732, 1445, 1067, 1020 cm^{-1} (described without distinction of the two isomers); ^1H NMR (300 MHz, $CDCl_3$, signals from the *exo* isomer **4ad** are marked with an asterisk) δ 6.87* (1H, s), 6.76* (1H, dd, *J* = 7.8, 1.8 Hz), 6.69 (1H, dd, *J* = 7.8, 1.8 Hz), 6.59* (1H, dd, *J* = 7.8, 5.1 Hz), 6.47 (1H, dd, *J* = 7.8, 5.1 Hz), 6.33 (1H, s), 5.22–5.14* (1H, m), 5.21–5.13 (1H, m), 3.84* (3H, s), 3.84 (3H, s), 3.03* (1H, dd, *J* = 13.5, 4.2 Hz), 2.75* (6H, s), 2.67 (6H, s), 2.42 (1H, dd, *J* = 13.5, 1.5 Hz), 2.05 (1H, dd, *J* = 13.5, 4.2), 1.58 (3H, s), 1.46* (1H, dd, *J* = 13.5, 1.5 Hz), 1.19* (3H, s); ^{13}C NMR (75 MHz, $CDCl_3$, signals that could be distinguished from the *exo* isomer **4ad** are marked with an asterisk) δ 169.8, 169.3*, 168.1, 167.7, 140.1*, 136.8, 132.4, 131.7*, 131.4*, 130.1, 74.5, 74.0*, 63.5*, 61.9, 52.5, 52.4, 43.0*, 42.9, 42.7, 42.1, 40.7, 39.5*, 26.7, 23.5*; HRMS (FAB) *m*/*z* calcd for $C_{13}H_{19}N_2O_4$ $[M+H]^+$ 267.1345, found 267.1357.



3ae



4ae



3ae'

6-methyl-*endo*-cycloadduct **3ae**, its *exo*-isomer **4ae** and *cis*-alkene derived *endo*-adduct **3ae'**

A solution of α-pyrone **1a** (50.0 mg, 0.324 mmol), crotonaldehyde hydrazone **2e** (72.7 mg, 0.648 mmol) and $Eu(hfc)_3$ (38.7 mg, 32.4 μmol) in 1,2-dichloroethane (1.6 mL) was stirred at room temperature for 16 hours. After the reaction mixture was concentrated under reduced pressure, the residue was purified by column chromatography on silica gel (hexane : AcOEt = 3 : 2 to 1 : 1) to afford *endo* adduct **3ae** (45.5 mg, 53%) as pale yellow oil and a mixture of **3ae**, *exo* adduct **4ae** and *cis*-alkene derived *endo*-adduct **3ae'** (19.9 mg, 23%, **3ae** : **4ae** : **3ae'** = 71 : 14 : 15) as orange oil.

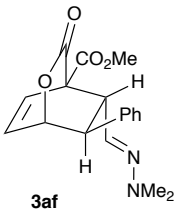
Analytical samples were obtained by separation of the mixture with PTLC (CH_2Cl_2 : AcOEt = 2 : 1) to afford *exo* adduct **4ae** and *cis*-alkene derived *endo*-adduct **3ae'** both as pale yellow oil.

3ae: IR (NaCl) 2968, 2851, 1755, 1724, 1435, 1304, 1279, 1119, 1099 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 6.76 (1H, brd, *J* = 7.8 Hz), 6.53 (1H, dd, *J* = 7.8, 5.4 Hz), 6.23 (1H, d, *J* = 4.2 Hz), 4.90 (1H, dt, *J* = 5.4, 1.2 Hz), 3.85 (3H, s), 2.86 (1H, t, *J* = 4.2 Hz), 2.68 (6H, s), 2.13–2.06 (1H, m), 1.30 (3H, d, *J* = 7.2 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.2, 168.3, 131.4, 131.0, 130.3, 79.6, 58.3, 52.6, 46.0, 42.8, 38.0, 17.8; HRMS (FAB) *m*/*z* calcd for $C_{13}H_{19}N_2O_4$ $[M+H]^+$ 267.1345, found 267.1344.

4ae: IR (NaCl) 2957, 1749, 1734, 1286, 1096, 1034 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 6.84 (1H, dd, *J* =

7.8, 1.8 Hz), 6.53 (1H, dd, *J* = 7.8, 4.8 Hz), 6.49 (1H, d, *J* = 7.2 Hz), 4.98 (1H, ddd, *J* = 4.8, 3.6, 1.8 Hz), 3.82 (3H, s), 2.76 (6H, s), 2.57–2.50 (1H, m), 2.34 (1H, dd, *J* = 7.2, 5.4 Hz), 1.01 (3H, d, *J* = 6.6 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.2, 167.8, 133.9, 133.3, 130.4, 78.3, 58.0, 52.6, 49.3, 43.0, 39.5, 17.8; HRMS (FAB) *m*/*z* calcd for $C_{13}H_{19}N_2O_4$ $[M+H]^+$ 267.1345, found 267.1340.

3ae': IR (NaCl) 2955, 1757, 1734, 1285, 1092, 1076 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.98 (1H, brd, *J* = 7.8 Hz), 6.41 (1H, dd, *J* = 7.8, 4.8 Hz), 6.17 (1H, d, *J* = 5.4 Hz), 4.99 (1H, ddd, *J* = 4.8, 3.6, 1.8 Hz), 3.82 (3H, s), 3.55 (1H, dd, *J* = 10.2, 5.4 Hz), 2.88-2.78 (1H, m), 2.68 (6H, s), 0.91 (3H, d, *J* = 7.8 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.3, 168.3, 133.0, 130.6, 128.3, 78.8, 58.3, 52.6, 42.9, 42.8, 36.4, 15.1; HRMS (FAB) *m*/*z* calcd for $C_{13}H_{19}N_2O_4$ $[M+H]^+$ 267.1345, found 267.1335.

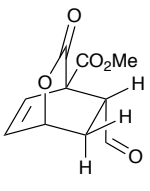


3af

6-phenyl-*endo*-cycloadduct **3af**

A solution of α-pyrone **1a** (265 mg, 1.72 mmol), cinnamaldehyde hydrazone **2f** (30.0 mg, 0.172 mmol) and $Eu(hfc)_3$ (20.5 mg, 17.2 μmol) in 1,2-dichloroethane (0.86 mL) was stirred at 80 °C for 24 hours. After the reaction mixture was concentrated under reduced pressure, the residue was purified by column chromatography on silica gel (hexane : AcOEt = 4 : 1 to 1 : 2) and (hexane : AcOEt = 2 : 1) to afford *endo* adduct **3af** (35.7 mg, 63%) with a trace amount of impurity as a pale yellow solid. Unreacted **1a** (206 mg, 78%) and **2f** (11.0 mg, 37%) were recovered.

mp 96.2-97.3 °C; IR (KBr) 1759, 1285, 1107, 1074 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.25 (5H, m), 6.84 (1H, ddd, *J* = 7.8, 1.8, 1.2 Hz), 6.63 (1H, dd, *J* = 7.8, 5.1 Hz), 6.35 (1H, d, *J* = 3.6 Hz), 5.21 (1H, ddd, *J* = 5.1, 1.8, 1.2 Hz), 3.87 (3H, s), 3.52–3.46 (1H, m), 3.32 (1H, dd, *J* = 5.1, 1.2 Hz), 2.70 (6H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.3, 168.1, 140.1, 130.9, 130.7, 130.5, 128.9, 127.9, 127.5, 79.0, 59.0, 52.7, 48.1, 45.5, 42.8; HRMS (FAB) *m*/*z* calcd for $C_{18}H_{21}N_2O_4$ $[M+H]^+$ 329.1501, found 329.1517.

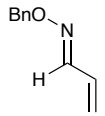


3aa

aldehyde **3aa**

To a solution of hydrazone **3ab** (50.0 mg, 0.198 mmol) in THF (3.9 mL) was added 37% HCHO aq. (81 μL) and conc. HCl (36%, 64 μL). After stirring for 2 hours at room temperature, the reaction mixture was added water. The whole was extracted with CH_2Cl_2 (×4), and combined organic layer was washed with brine (×1). The resulting solution was dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (hexane : AcOEt = 1 : 2) to afford aldehyde **3aa** (41.6 mg, quant) as a white solid.

mp 80.0-80.8 °C; IR (KBr) 2926, 1757, 1738, 1711, 1288, 1099, 1076 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 9.49 (1H, s), 6.84 (1H, d, *J* = 7.8 Hz), 6.52 (1H, dd, *J* = 7.8, 4.8 Hz), 5.36–5.30 (1H, m), 3.93 (3H, s), 3.62 (1H, dd, *J* = 10.2, 3.6 Hz), 2.63 (1H, ddd, *J* = 13.8, 10.2, 3.6 Hz), 2.09 (1H, ddd, *J* = 13.8, 3.6, 1.2 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 196.6, 169.0, 167.5, 131.8, 131.4, 74.0, 55.7, 53.4, 46.7, 27.9; HRMS (FAB) *m*/*z* calcd for $C_{10}H_{11}O_5$ $[M+H]^+$ 211.0606, found 211.0613.

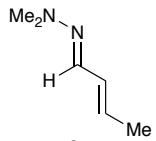


2c

acrolein (*E*)-benzyloxime **2c**

To a solution of *O*-benzylhydroxylamine hydrochloride (5.00 g, 31.3 mmol) and sodium acetate (2.57 g, 31.3 mmol) in MeOH (100 mL) was added acrolein monomer (2.10 mL, 31.5 mmol). After stirring for an hour at room temperature, water was added to the reaction mixture. The whole was extracted with Et_2O (×2), and combined organic layer was washed with water (×1) and brine (×1). The resulting solution was dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (hexane : Et_2O = 15 : 1) to afford oxime **2c** (1.81 g, 36%) as colorless oil.

IR (NaCl) 3065, 2928, 1456, 1366, 1024, 939 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.78 (1H, d, *J* = 10.2 Hz), 7.40–7.26 (5H, m), 6.41 (1H, dt, *J* = 17.4, 10.2 Hz), 5.53 (1H, d, *J* = 10.2 Hz), 5.50 (1H, d, *J* = 17.4 Hz), 5.11 (2H, s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.0, 137.3, 130.7, 128.4, 128.2, 127.9, 123.9, 76.1; HRMS (EI) *m*/*z* calcd for $C_{10}H_{11}NO$ 161.0841, found 161.0845.



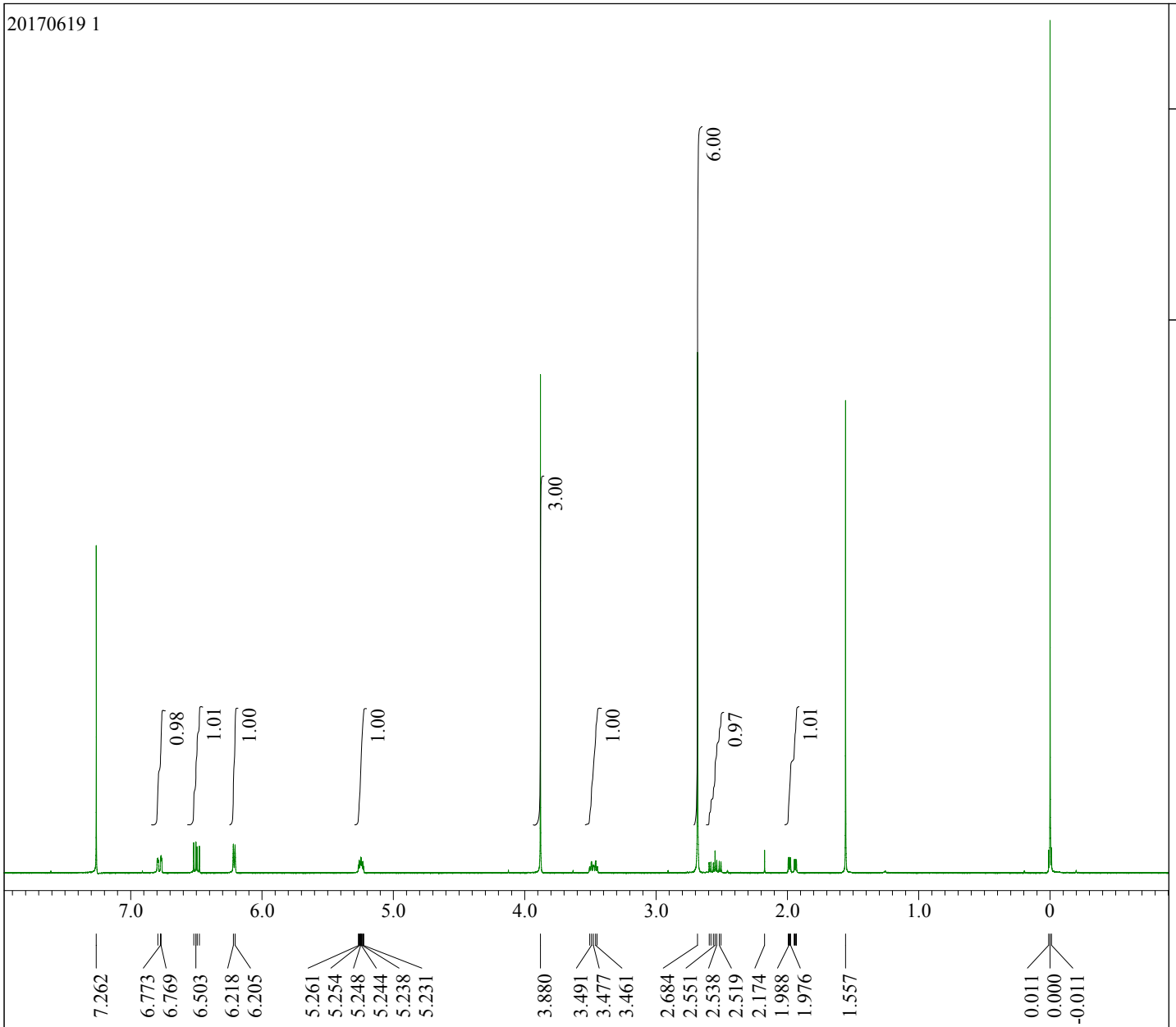
2e

crotonaldehyde dimethylhydrazone **2e**

To a solution of 1,1-dimethylhydrazine (3.30 ml, 40.0 mmol) and acetic acid (2.30 ml, 40.0 mmol) in CH_2Cl_2 (40 mL) was added crotonaldehyde (3.30 ml, 40.0 mmol). After stirring for 45 minutes at 0 °C, the reaction mixture was diluted with CH_2Cl_2. The whole was successively washed with water (×1), sat. $NaHCO_3$ aq. and brine (×1). The resulting solution was dried over $MgSO_4$, and concentrated under reduced pressure. The residue was distilled (2.0 kPa, 55–58 °C) to afford hydrazone **2e** (2.72 g, 61%, *E* : *Z* = 89 : 11) as colorless oil.

IR (NaCl) 2855, 1568, 1470, 1445, 1269, 1134, 1024, 968 cm^{-1}, ^1H NMR (600 MHz, $CDCl_3$, signals from the minor isomer are marked with an asterisk) δ 7.25* (1H, d, *J* = 9.6 Hz), 7.01 (1H, d, *J* = 9.0 Hz), 6.20 (1H, dd, *J* = 15.6, 9.0 Hz), 6.14* (1H, dd, *J* = 10.2, 9.6 Hz), 5.82 (1H, dq, *J* = 15.6, 7.2 Hz), 5.62* (1H, dq, *J* = 10.2, 7.2 Hz), 2.87* (6H, s), 2.81 (6H, s), 1.81 (3H+3H*, d, *J* = 7.2 Hz); ^{13}C NMR (150 MHz, $CDCl_3$, weak signals from the minor isomer are marked with an asterisk) δ 137.0, 131.9*, 130.3, 130.2, 127.7*, 126.3*, 42.9, 42.8*, 18.2, 13.5*; HRMS (ESI) *m*/*z* calcd for $C_6H_{13}N_2$ $[M+H]^+$ 113.1079, found 113.1076.

20170619 1

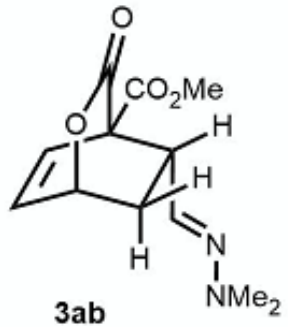


X : ppm : 1H

JEOL Solutions for Innovation

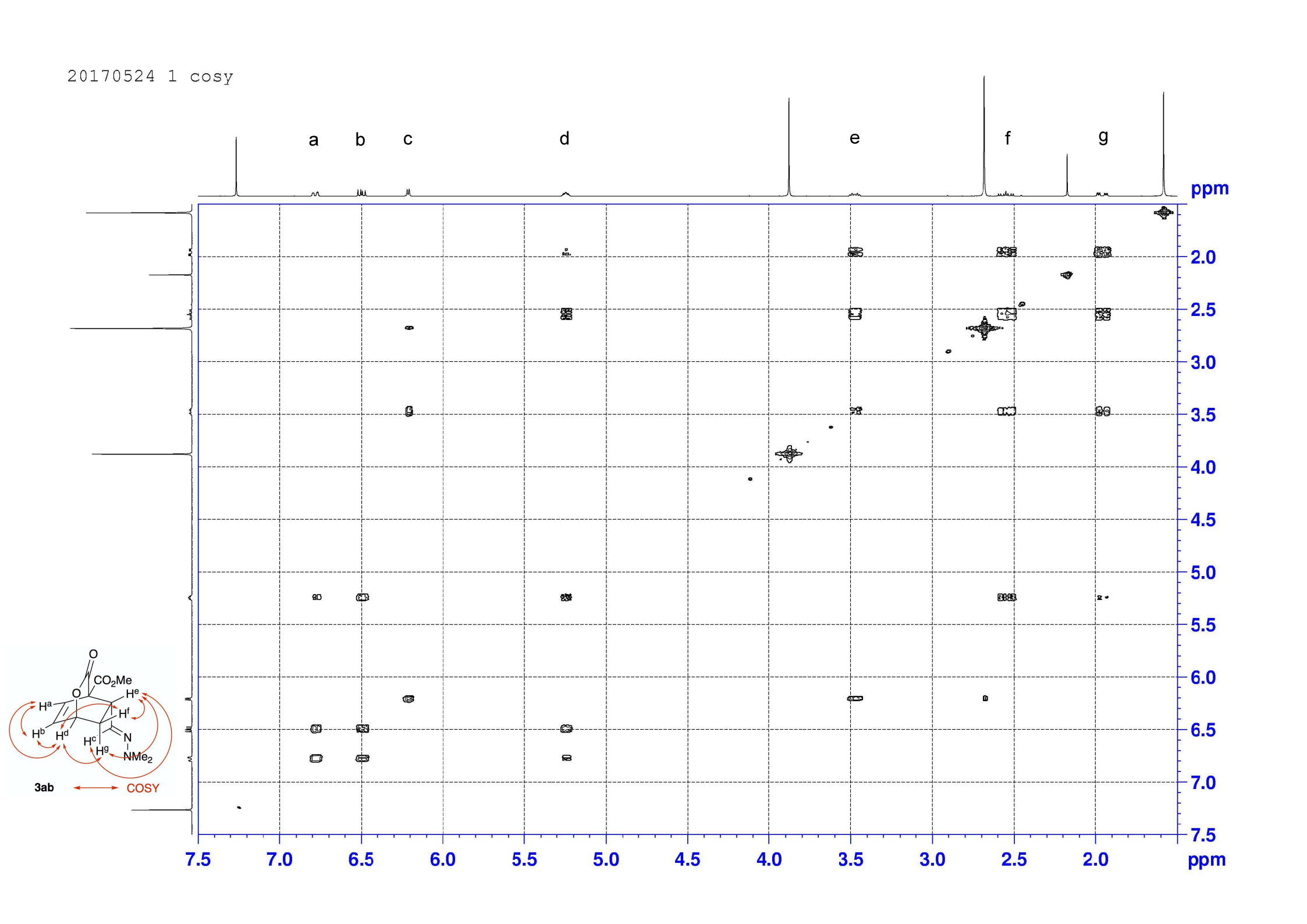
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
```

```
Filename       = Y-Abe_440 p23 9-12-1.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = 20170619 1
Solvent        = CHLOROFORM-D
Creation_Time  = 19-JUN-2017 16:15:28
Revision_Time  = 19-JUN-2017 16:17:57
Current_Time   =  7-SEP-2017 10:31:10

Comment        = 20170619 1
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       = 295.66[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```

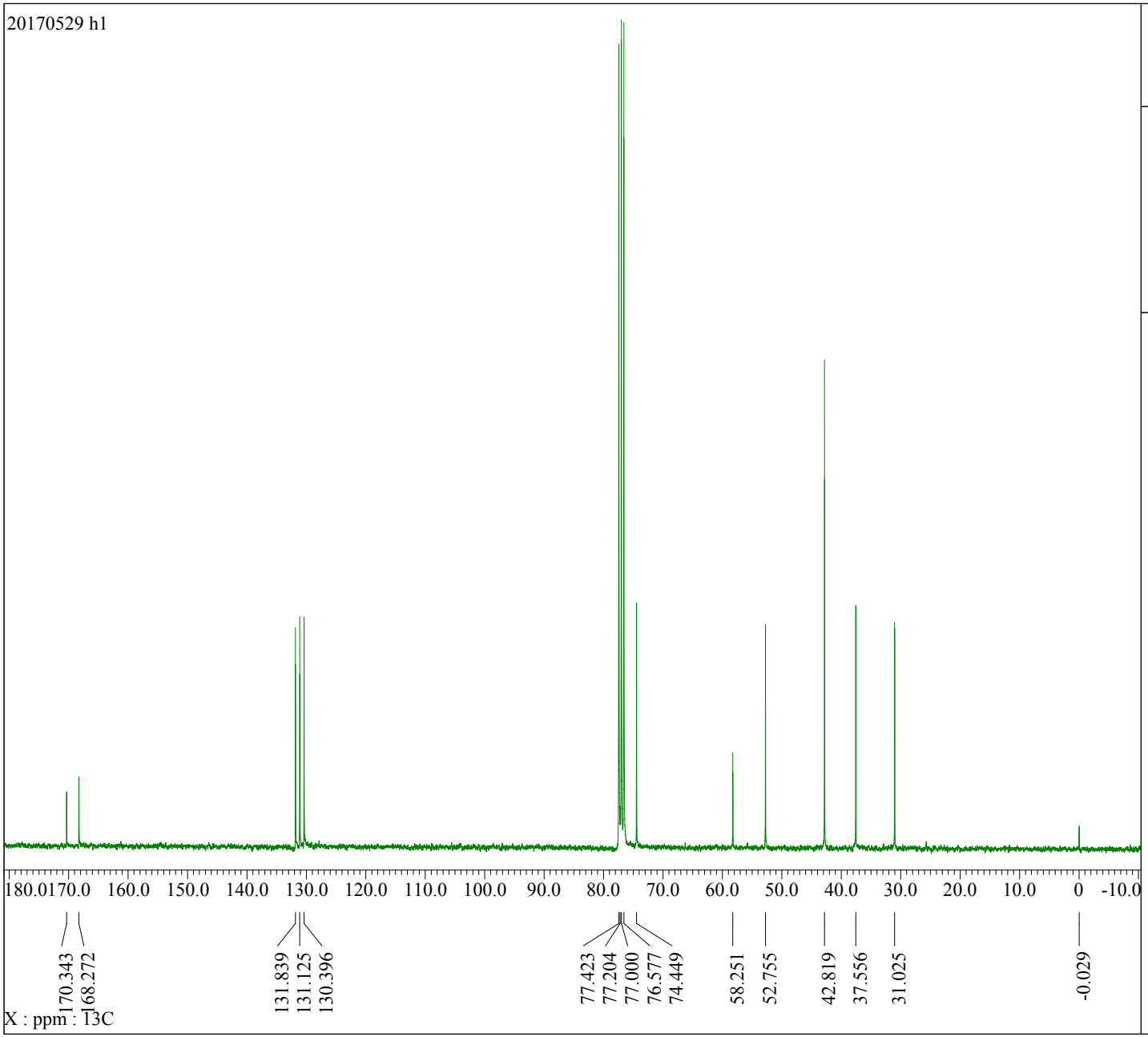


3ab

20170524 1 cosy



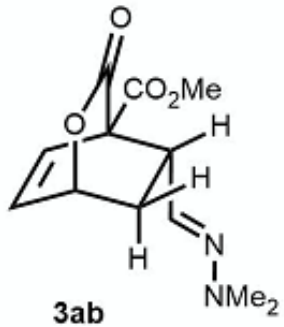
20170529 h1



JEOL Solutions for Innovation

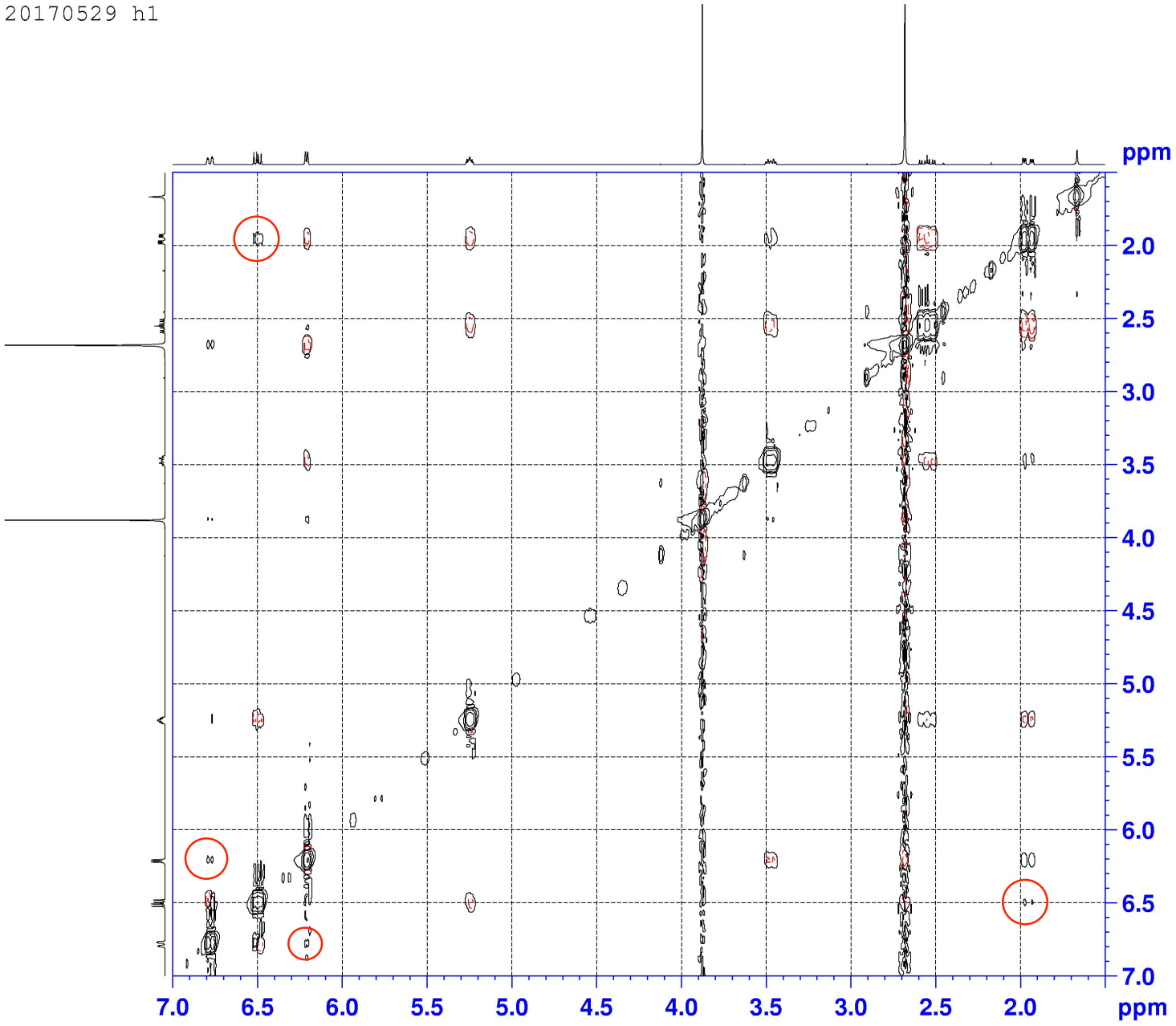
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase

以下に由来:: Y-Abe_222-12.jdf
```

```
Filename       = Y-Abe_222-16.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = 20170529 h1
Solvent        = CHLOROFORM-D
Creation_Time  =  7-SEP-2017 12:32:54
Revision_Time  =  7-SEP-2017 12:40:48
Current_Time   =  7-SEP-2017 12:41:10

Comment        = 20170529 h1
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       = 297.16[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 4096
```

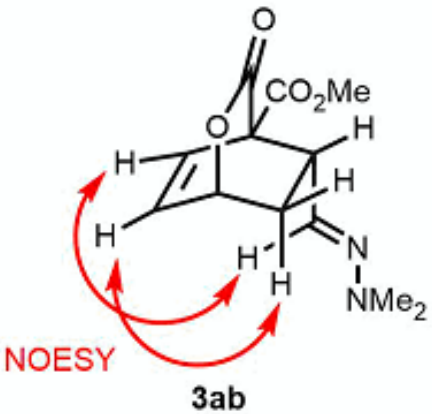


3ab

20170529 h1

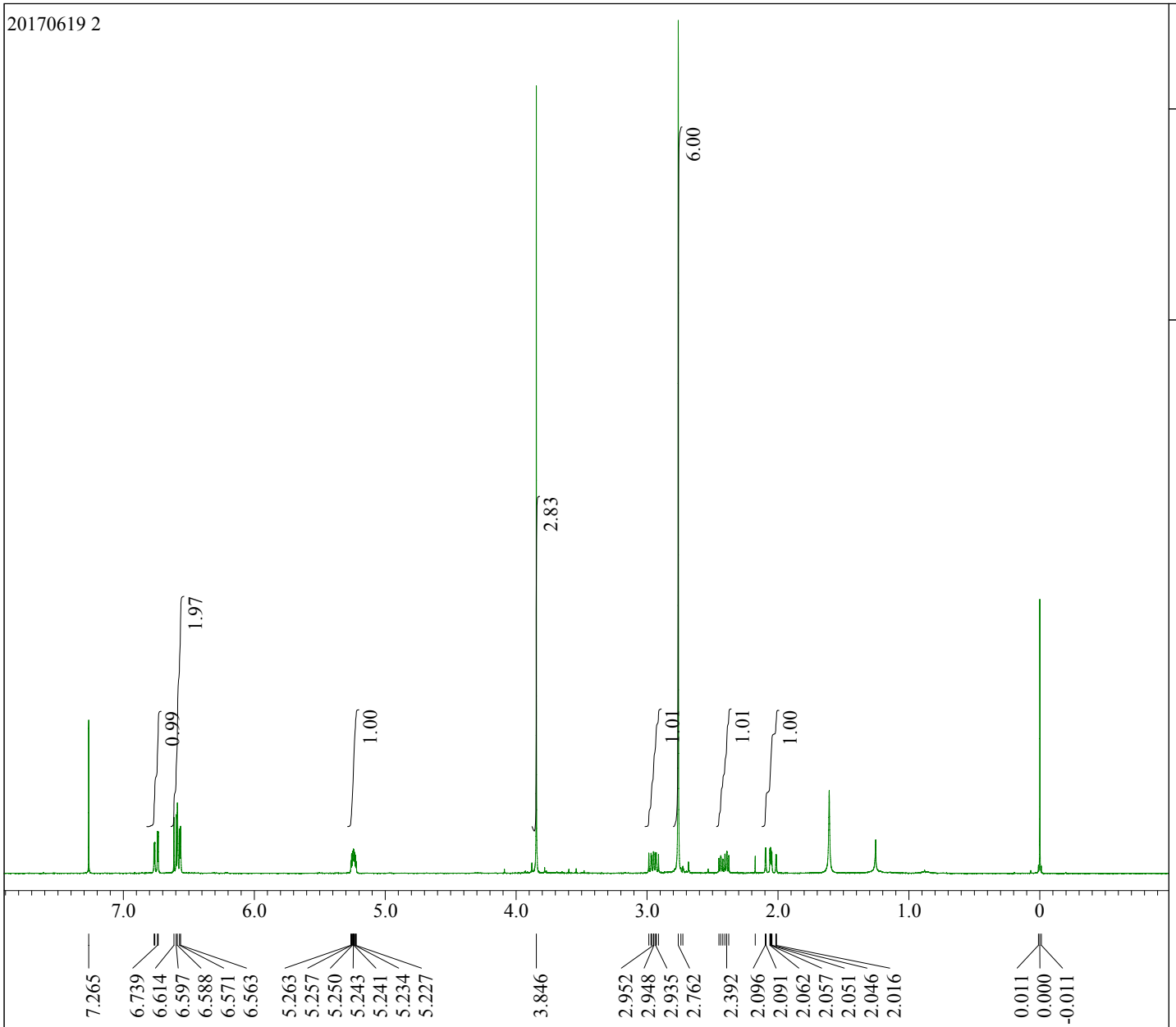



```
Current Data Parameters
NAME                  Y-Abe
EXPNO                   221
PROCNO                    1

F2 - Acquisition Parameters
Date_              20170529
Time                 21.09
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           noesyph
TD                   2048
SOLVENT              CDCl3
NS                     16
DS                      4
SWH               2525.252 Hz
FIDRES               1.233033 Hz
AQ                0.4055040 sec
RG                     32
DW                 198.000 usec
DE                   6.50 usec
TE                  295.5 K
D0              0.00017922 sec
D1              1.92872906 sec
D8              0.89999998 sec
IN0             0.00039600 sec

======== CHANNEL f1 ========
SFO1           300.1311040 MHz
NUC1                    1H
P1                   14.75 usec
PLW1            6.19999981 W

F1 - Acquisition parameters
TD                     256
SFO1              300.1311 MHz
FIDRES             9.864267 Hz
SW                    8.414 ppm
FnMODE         States-TPPI
```



3ab

20170619 2

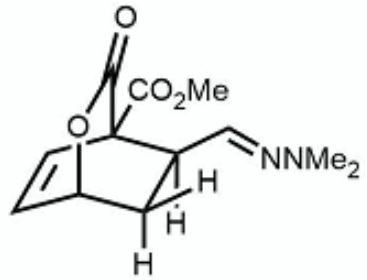


X : ppm : 1H

JEOL Solutions for Innovation

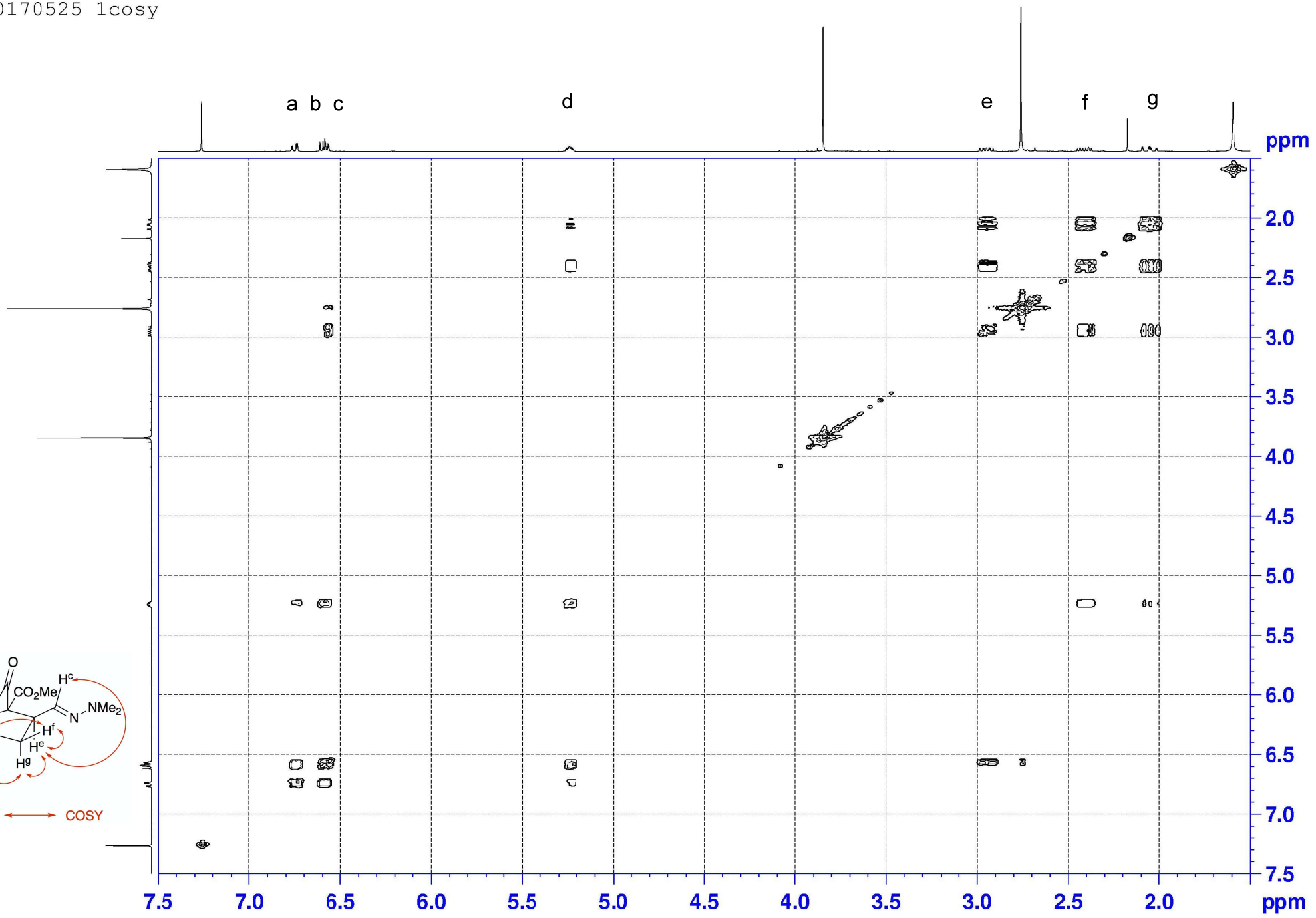
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
```

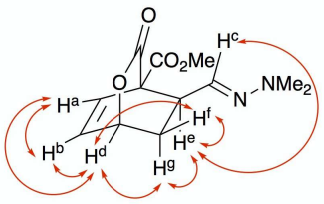
```
Filename       = Y-Abe_450 p23 14-17-2.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = 20170619 2
Solvent        = CHLOROFORM-D
Creation_Time  = 19-JUN-2017 16:20:25
Revision_Time  =  7-SEP-2017 12:28:02
Current_Time   =  7-SEP-2017 12:28:24

Comment        = 20170619 2
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       = 295.56[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



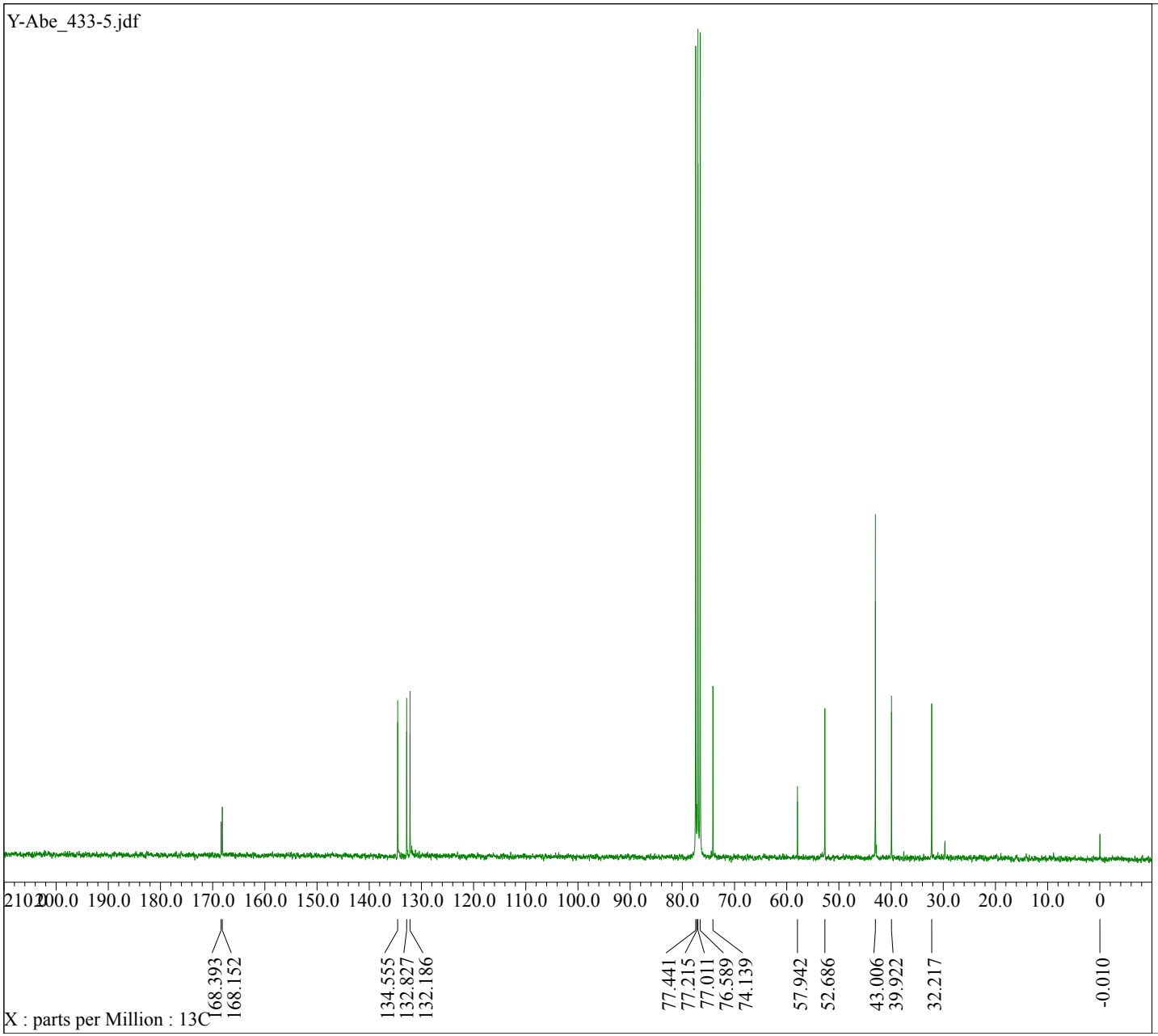
4ab

20170525 1cosy

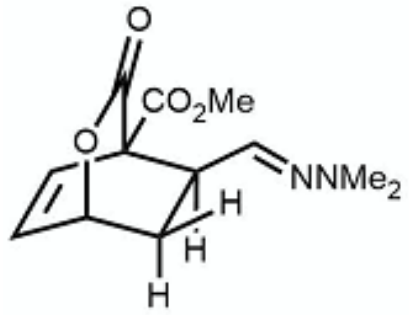




4ab ⟷ COSY

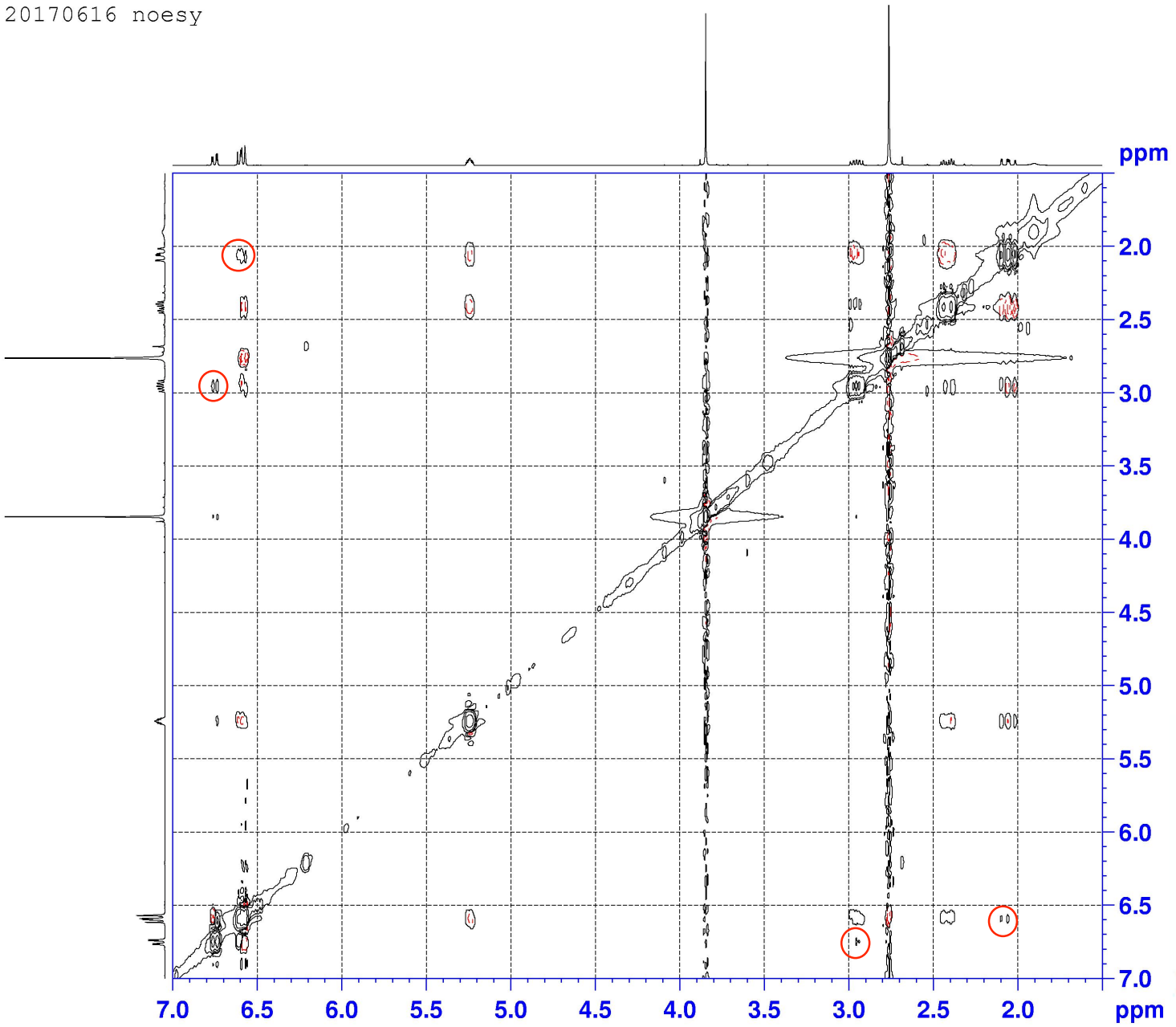



```
Filename       = Y-Abe_433-5.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = Parameter file, TOPSPIN  Vers
Solvent        = CDCl3
Creation_Time  = 21-JUL-2018 14:11:55
Revision_Time  = 21-JUL-2018 14:12:58
Current_Time   = 21-JUL-2018 14:16:03

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       = 297.06[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 4096
```

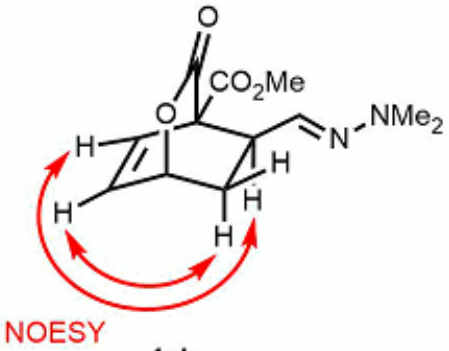


4ab

20170616 noesy

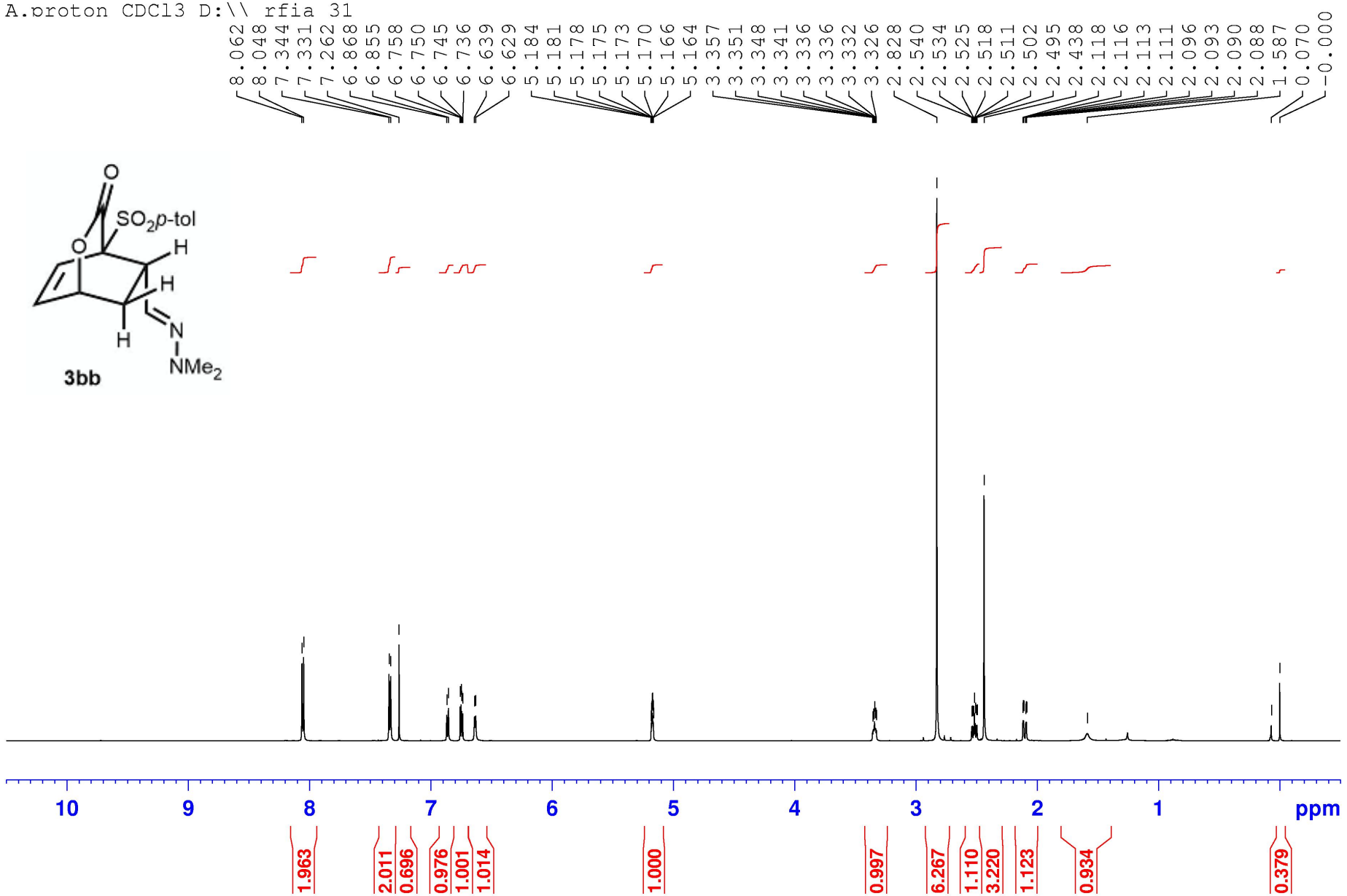



```
Current Data Parameters
NAME                  Y-Abe
EXPNO                   432
PROCNO                    1

F2 - Acquisition Parameters
Date_              20170616
Time                 21.20
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           noesyph
TD                   2048
SOLVENT              CDCl3
NS                     16
DS                      4
SWH               2577.320 Hz
FIDRES            1.258457 Hz
AQ                0.3973120 sec
RG                     32
DW                 194.000 usec
DE                   6.50 usec
TE                  295.5 K
D0              0.00017522 sec
D1              1.93692100 sec
D8              0.89999998 sec
IN0             0.00038800 sec

======== CHANNEL f1 ========
SFO1           300.1310959 MHz
NUC1                    1H
P1                   14.75 usec
PLW1            6.19999981 W

F1 - Acquisition parameters
TD                     256
SFO1             300.1311 MHz
FIDRES          10.067655 Hz
SW                  8.587 ppm
FnMODE       States-TPPI
```

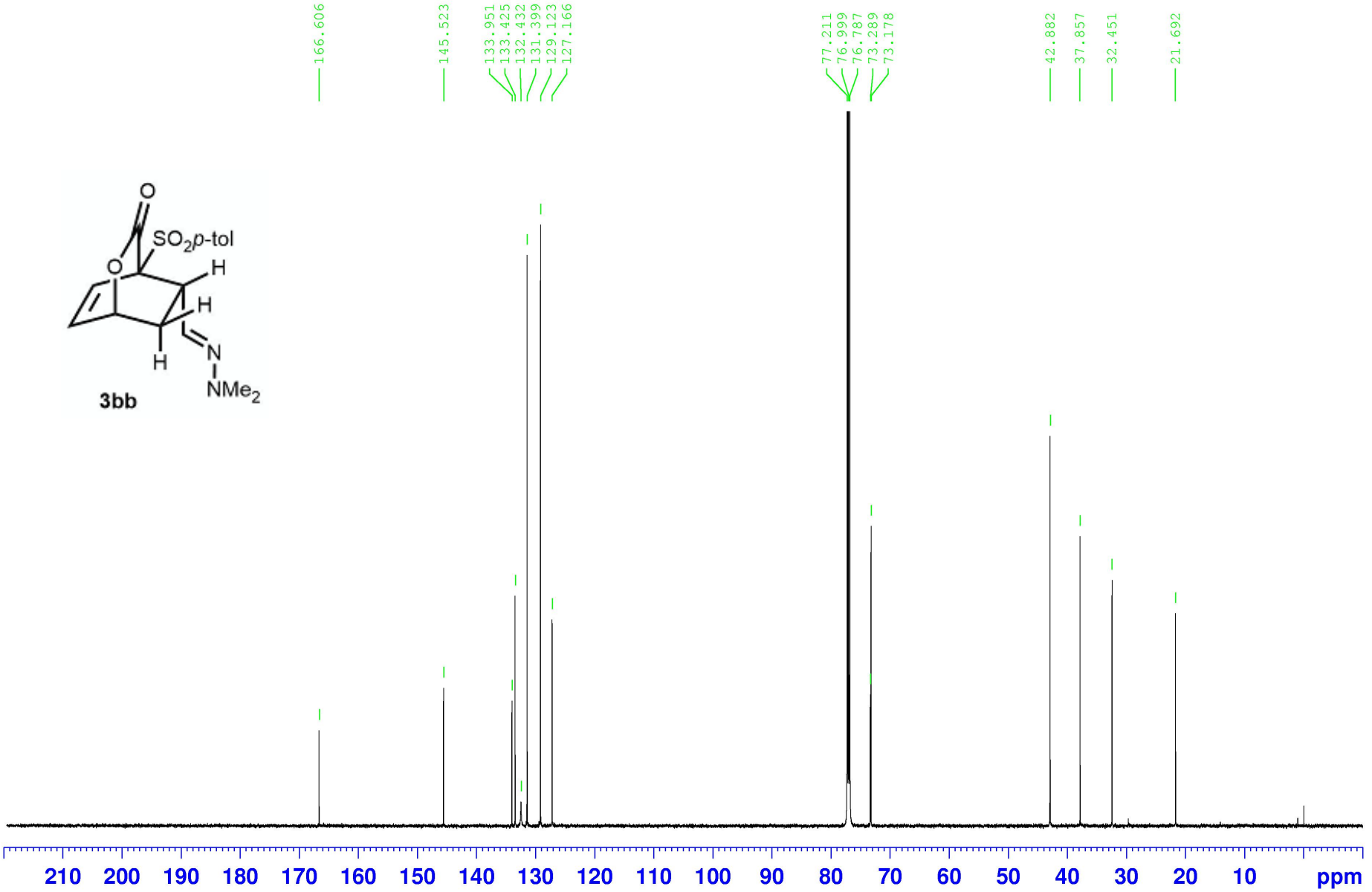


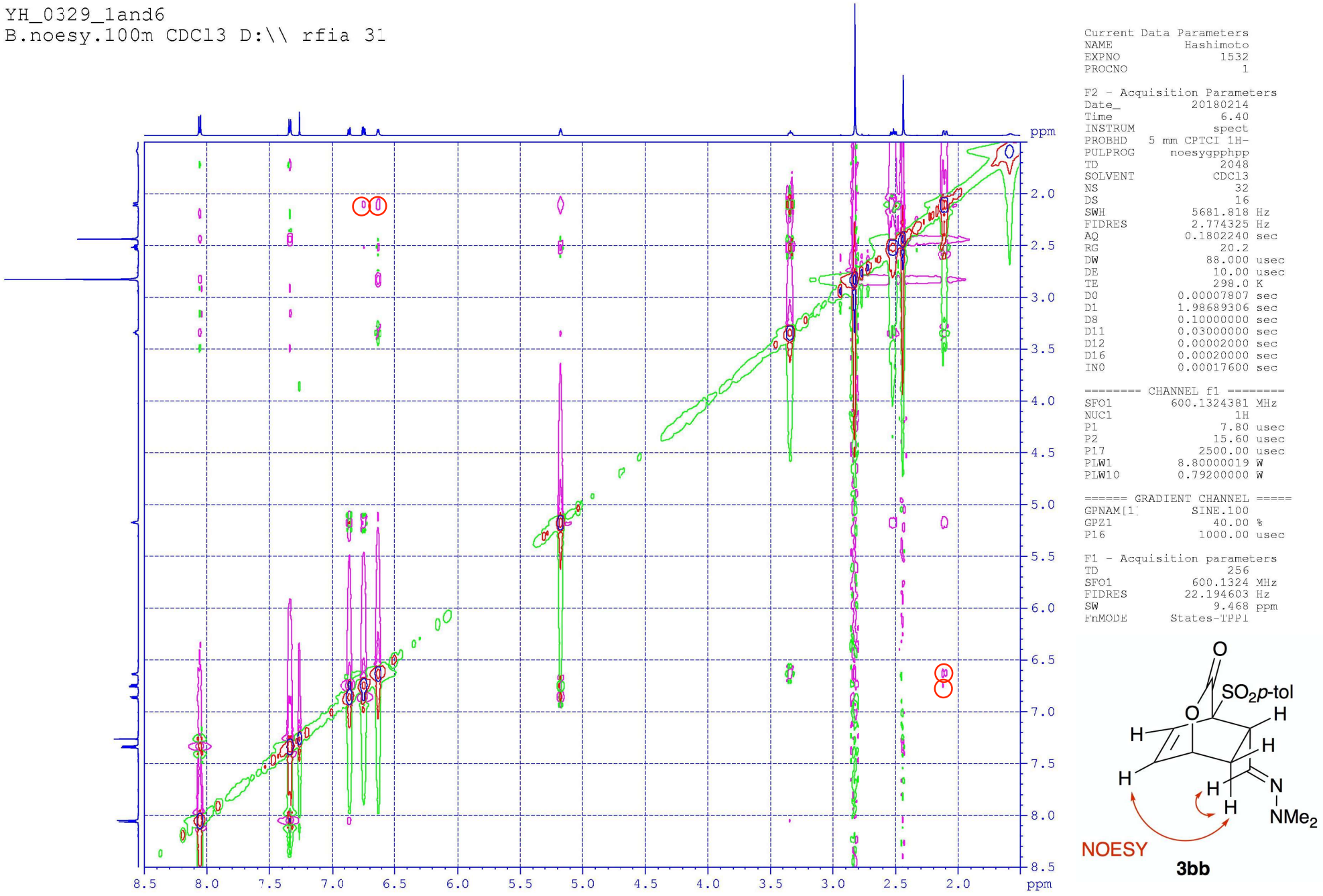
4ab

YH_0329_1and6
A.proton CDCl3 D:\\ rfia 31



YH_0329_1and6
A.carbon CDCl3 D:\\ rfia 31

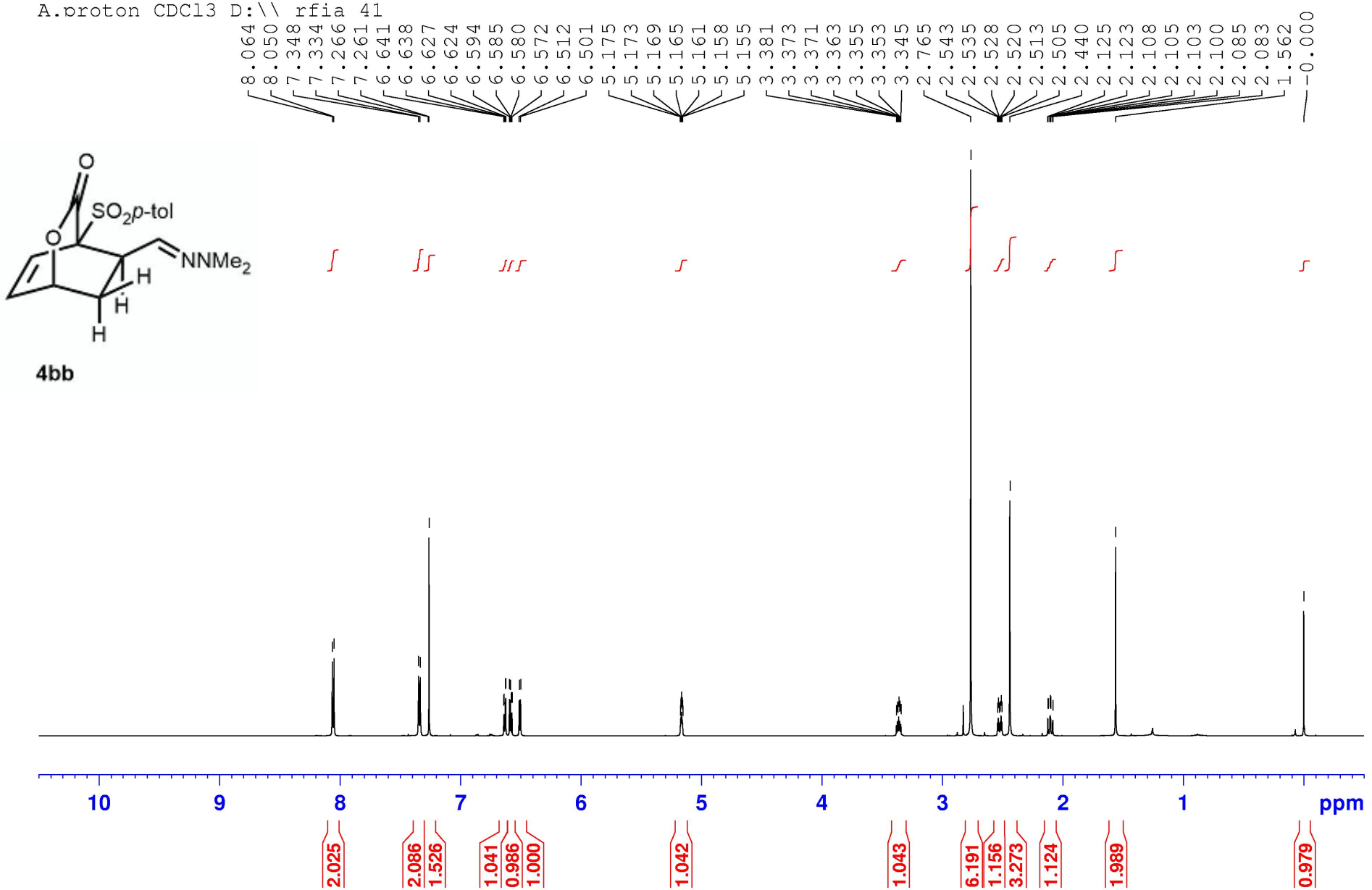




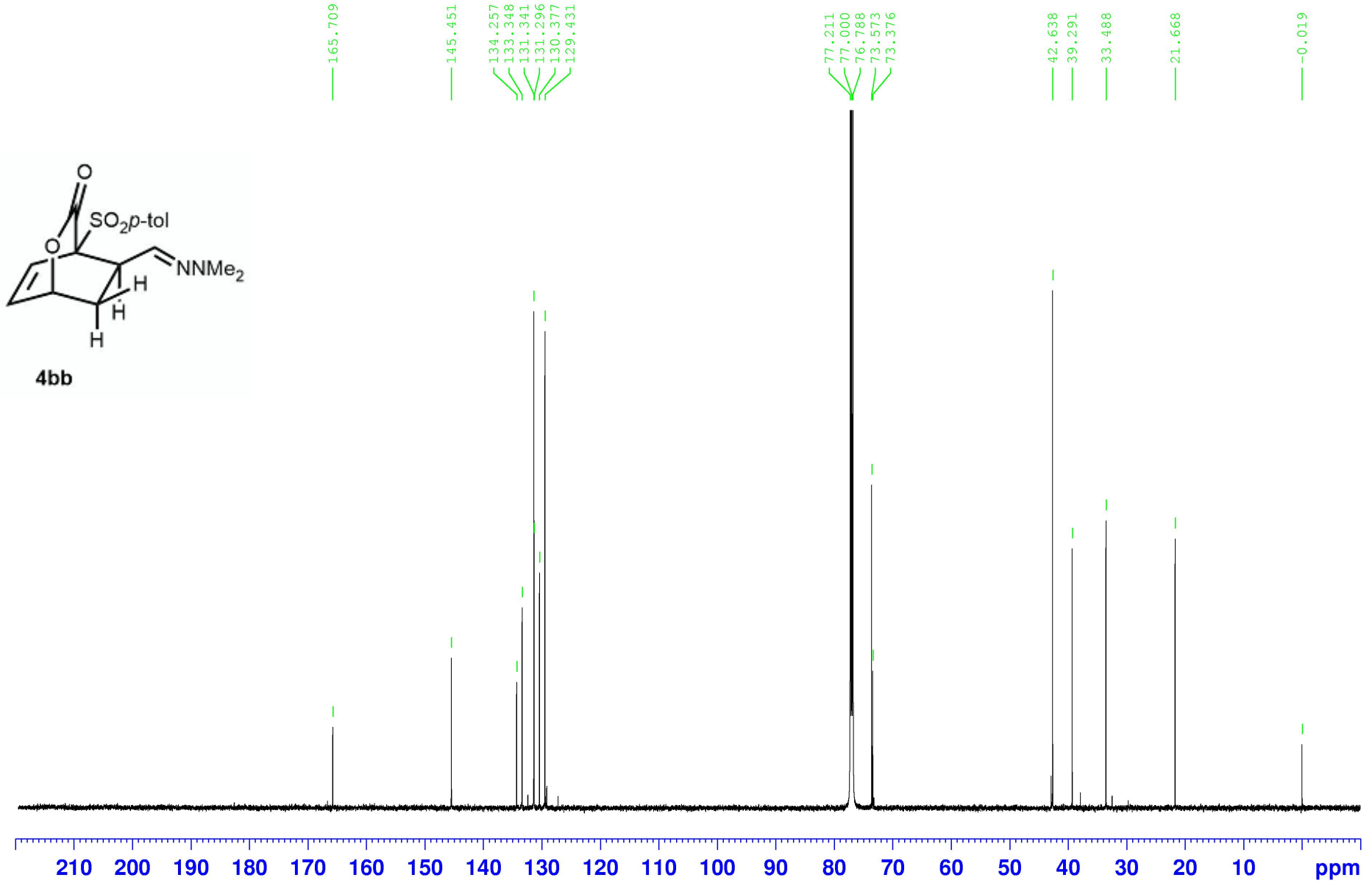

```
Current Data Parameters
NAME                  Hashimoto
EXPNO                      1532
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20180214
Time                       6.40
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG          noesygpphpp
TD                         2048
SOLVENT                  CDCl3
NS                           32
DS                           16
SWH                  5681.818 Hz
FIDRES               2.774325 Hz
AQ                   0.1802240 sec
RG                        20.2
DW                     88.000 usec
DE                     10.00 usec
TE                     298.0 K
D0                   0.00007807 sec
D1                   1.98689306 sec
D8                   0.10000000 sec
D11                  0.03000000 sec
D12                  0.00002000 sec
D16                  0.00020000 sec
IN0                  0.00017600 sec

======== CHANNEL f1 ========
SFO1                600.1324381 MHz
NUC1                        1H
P1                       7.80 usec
P2                      15.60 usec
P17                   2500.00 usec
PLW1                 8.80000019 W
PLW10                0.79200000 W

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

F1 - Acquisition parameters
TD                           256
SFO1                600.1324 MHz
FIDRES               22.194603 Hz
SW                       9.468 ppm
FnMODE           States-TPPI
```


3bb

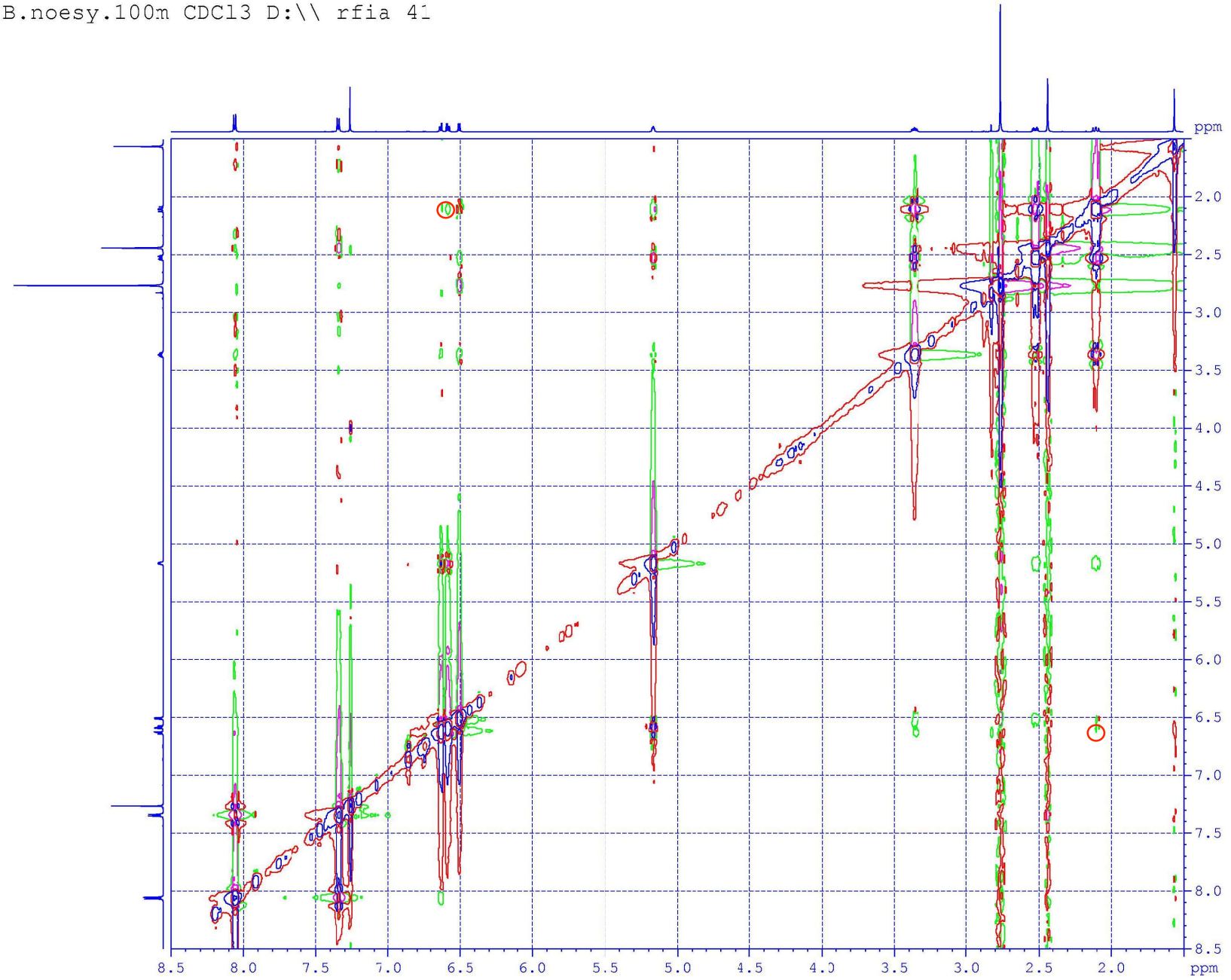
YH_0329_7
A.proton CDCl3 D:\\ rfia 41



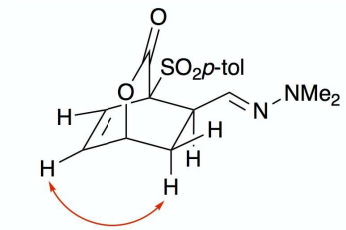
YH_0329_7
A.carbon CDCl3 D:\\ rfia 41



YH_0329_7
B.noesy.100m CDCl3 D:\\ rfia 41

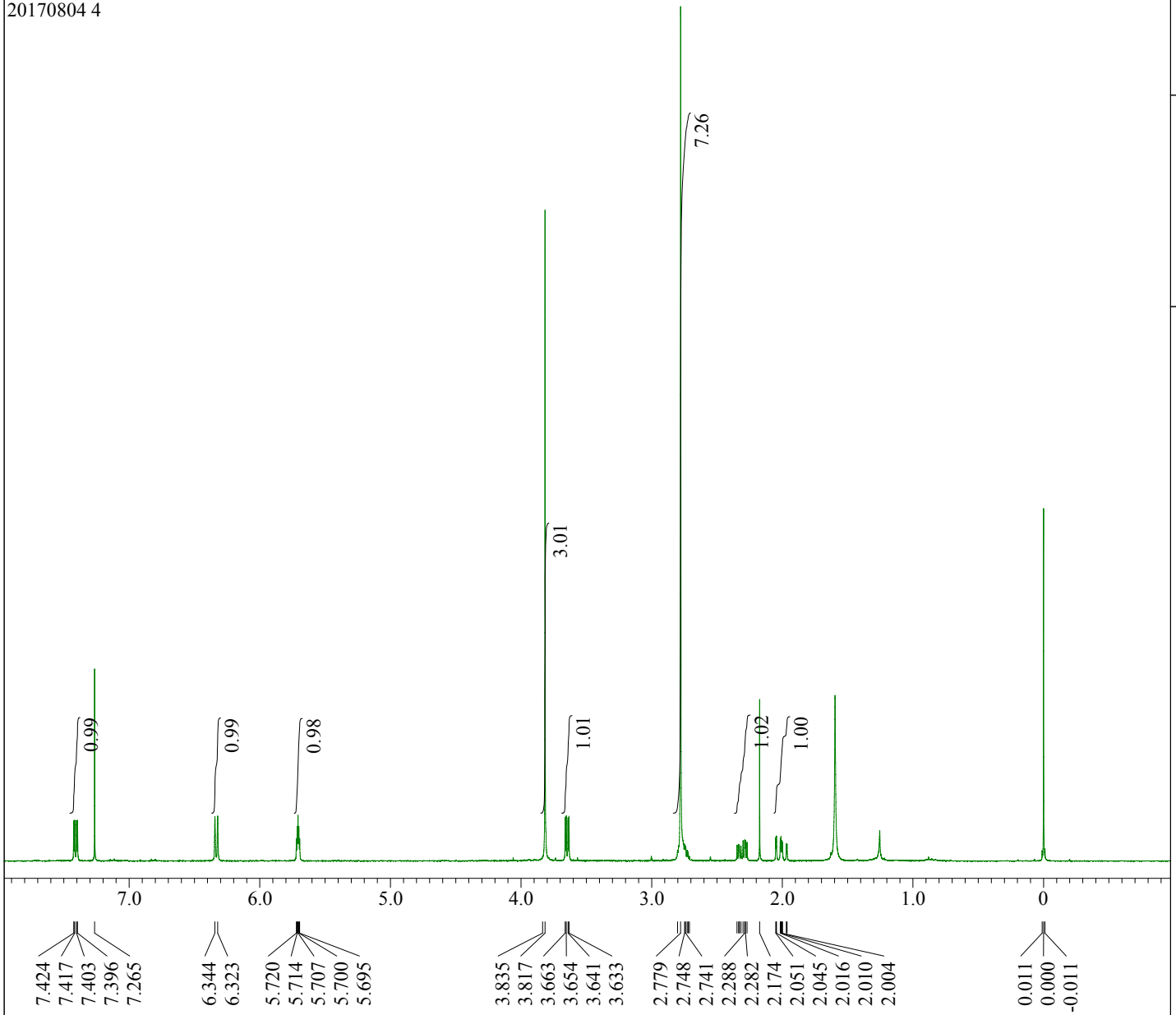



```
Current Data Parameters
NAME            Hashimoto
EXPNO                1522
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180213
Time                15.31
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG      noesygpphpp
TD                   2048
SOLVENT             CDCl3
NS                     32
DS                     16
SWH              5681.818 Hz
FIDRES           2.774325 Hz
AQ               0.1802240 sec
RG                    40.3
DW                 88.000 usec
DE                 10.00 usec
TE                 298.0 K
D0              0.00007807 sec
D1              1.98689306 sec
D8              0.10000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
D16             0.00020000 sec
IN0             0.00017600 sec

======== CHANNEL f1 ========
SFO1          600.1324588 MHz
NUC1                  1H
P1                 7.80 usec
P2                15.60 usec
P17             2500.00 usec
PLW1          8.80000019 W
PLW10         0.79200000 W

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

F1 - Acquisition parameters
TD                   256
SFO1            600.1325 MHz
FIDRES          22.194603 Hz
SW                9.468 ppm
FnMODE        States-TPPI

F2 - Processing parameters
SI                  1024
SF            600.1300099 MHz
WDW                 QSINE
SSB                     2
LB         0 Hz
GB         0
PC                   1.00

F1 - Processing parameters
SI                  1024
MC2           States-TPPI
SF            600.1300099 MHz
WDW                 QSINE
SSB                     2
LB         0 Hz
GB         0
```



4bb

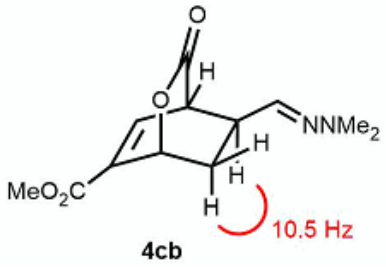
20170804 4



JEOL Solutions for Innovation

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
```

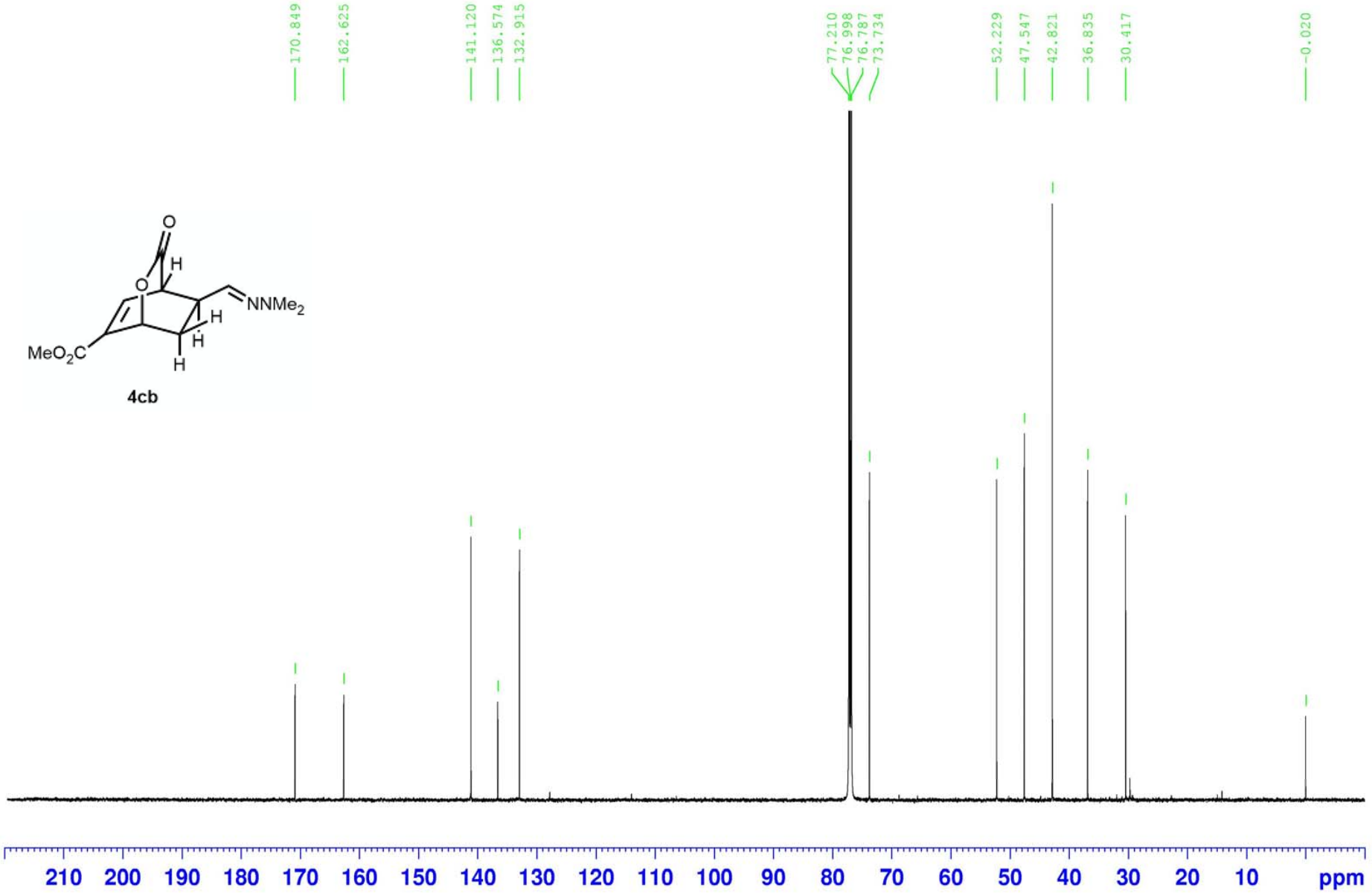
```
Filename       = Y-Abe_1180 honmonohonnmono-2.
Author         = Administrator
Experiment     = zg30
Sample_Id      = 20170804 4
Solvent        = CHLOROFORM-D
Creation_Time  =  5-AUG-2017 11:35:21
Revision_Time  =  7-SEP-2017 14:39:45
Current_Time   =  7-SEP-2017 14:40:26

Comment        = 20170804 4
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       = 296.16[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



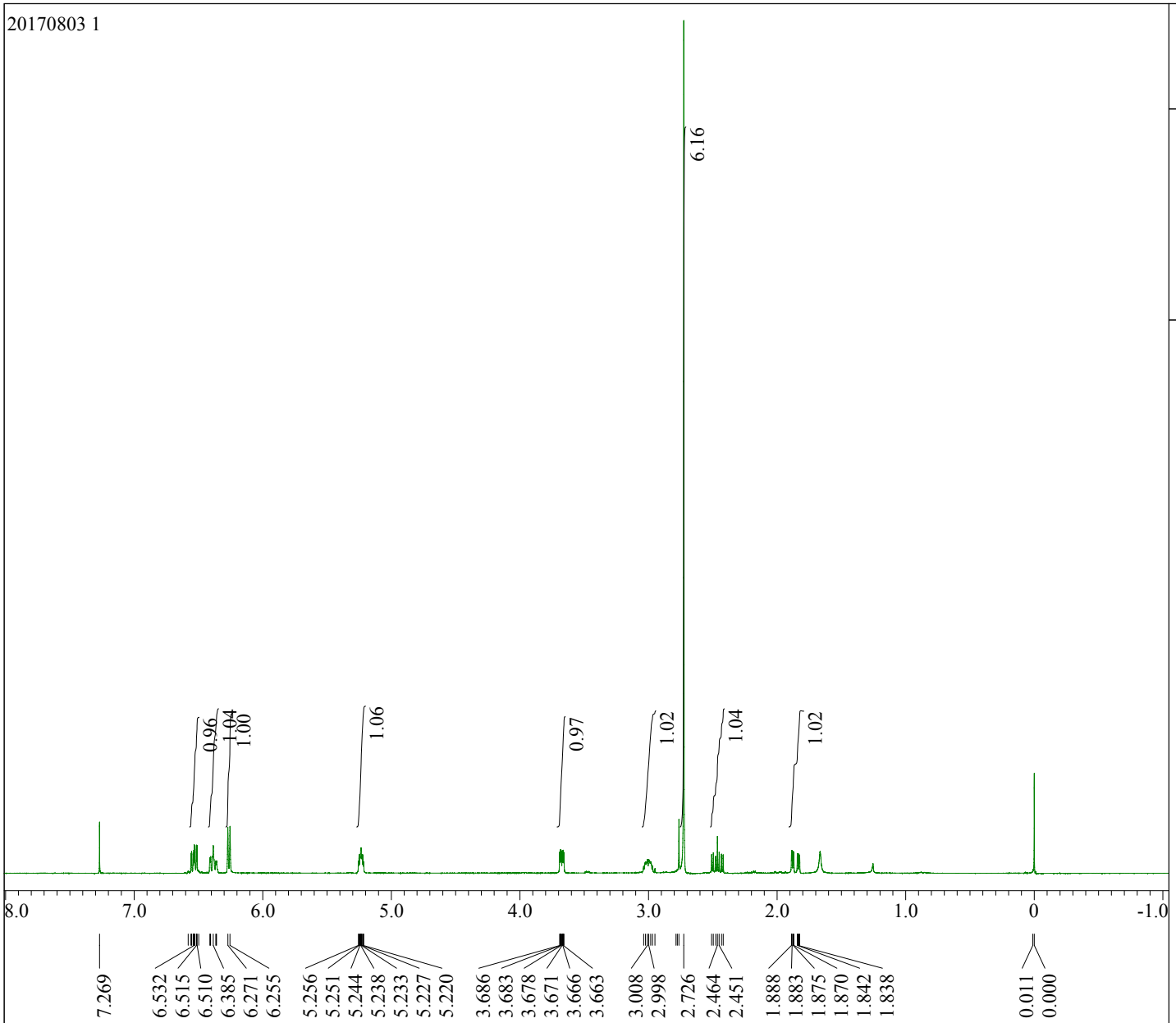
4cb

abe_no3_p43_3_12_15
A.carbon CDCl3 D:\\ rfia 41

4cb



20170803 1

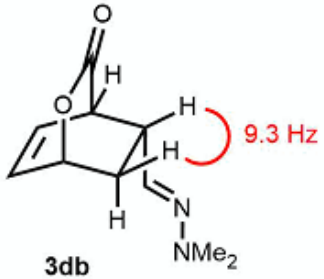


X : ppm : 1H

JEOL Solutions for Innovation

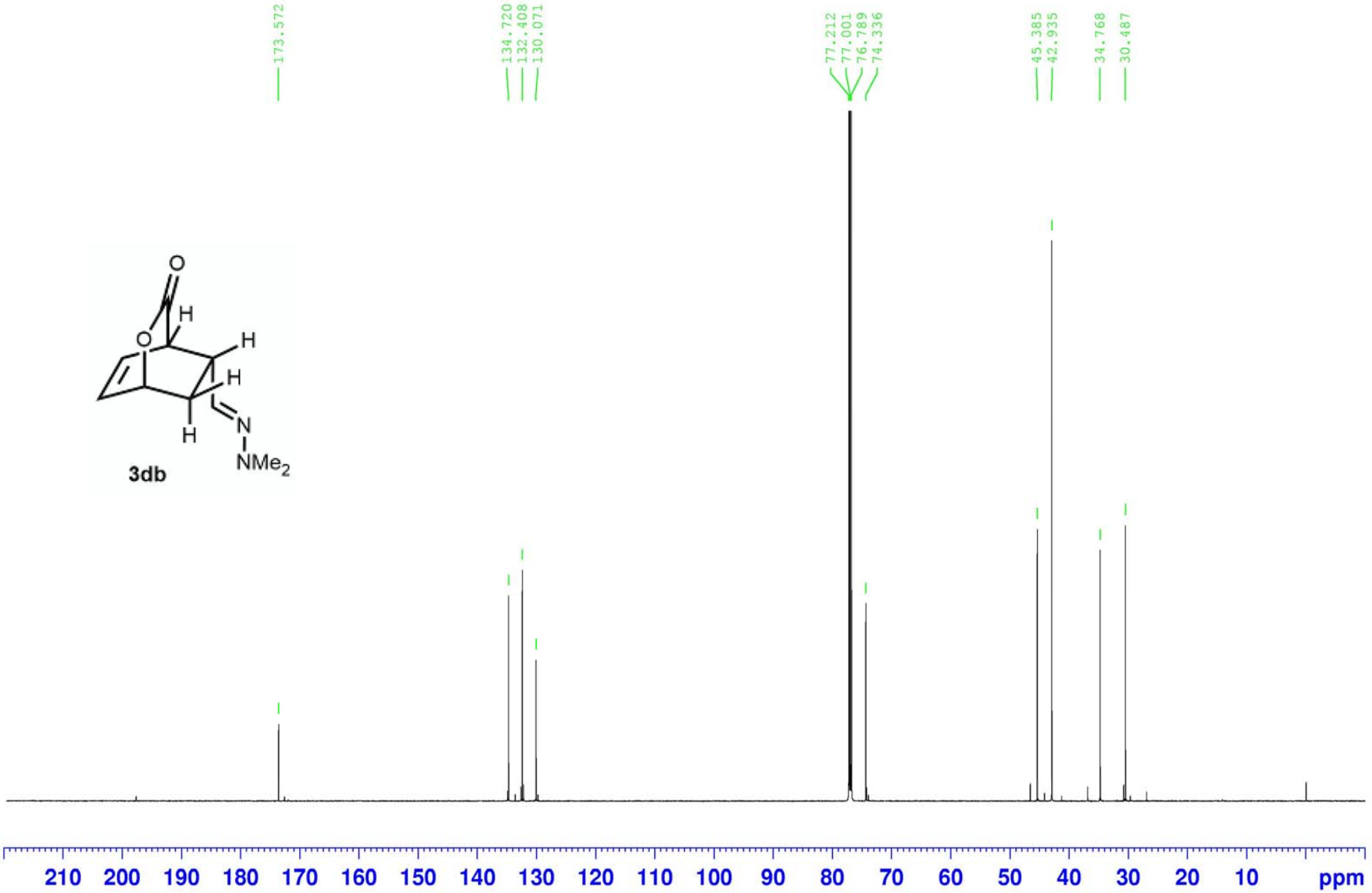
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
```

```
Filename       = Y-Abe_1110-2.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = 20170803 1
Solvent        = CHLOROFORM-D
Creation_Time  =  3-AUG-2017 13:45:51
Revision_Time  =  7-SEP-2017 14:44:06
Current_Time   =  7-SEP-2017 14:44:17

Comment        = 20170803 1
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       = 295.86[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



3db

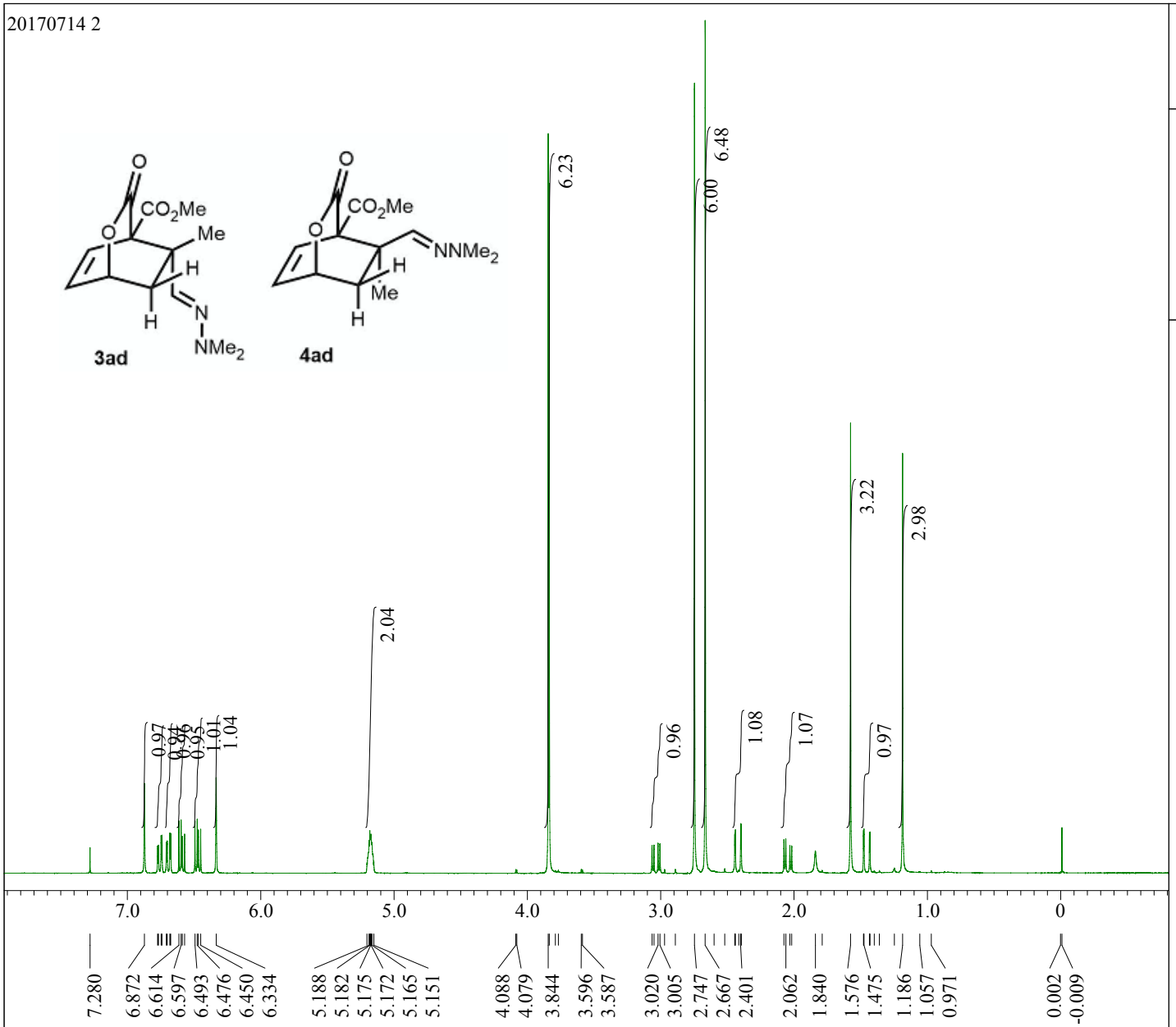
abe_no3_p45_32_44
A.carbon CDCl3 D:\\ rfia 22



20170714 2

3ad

4ad



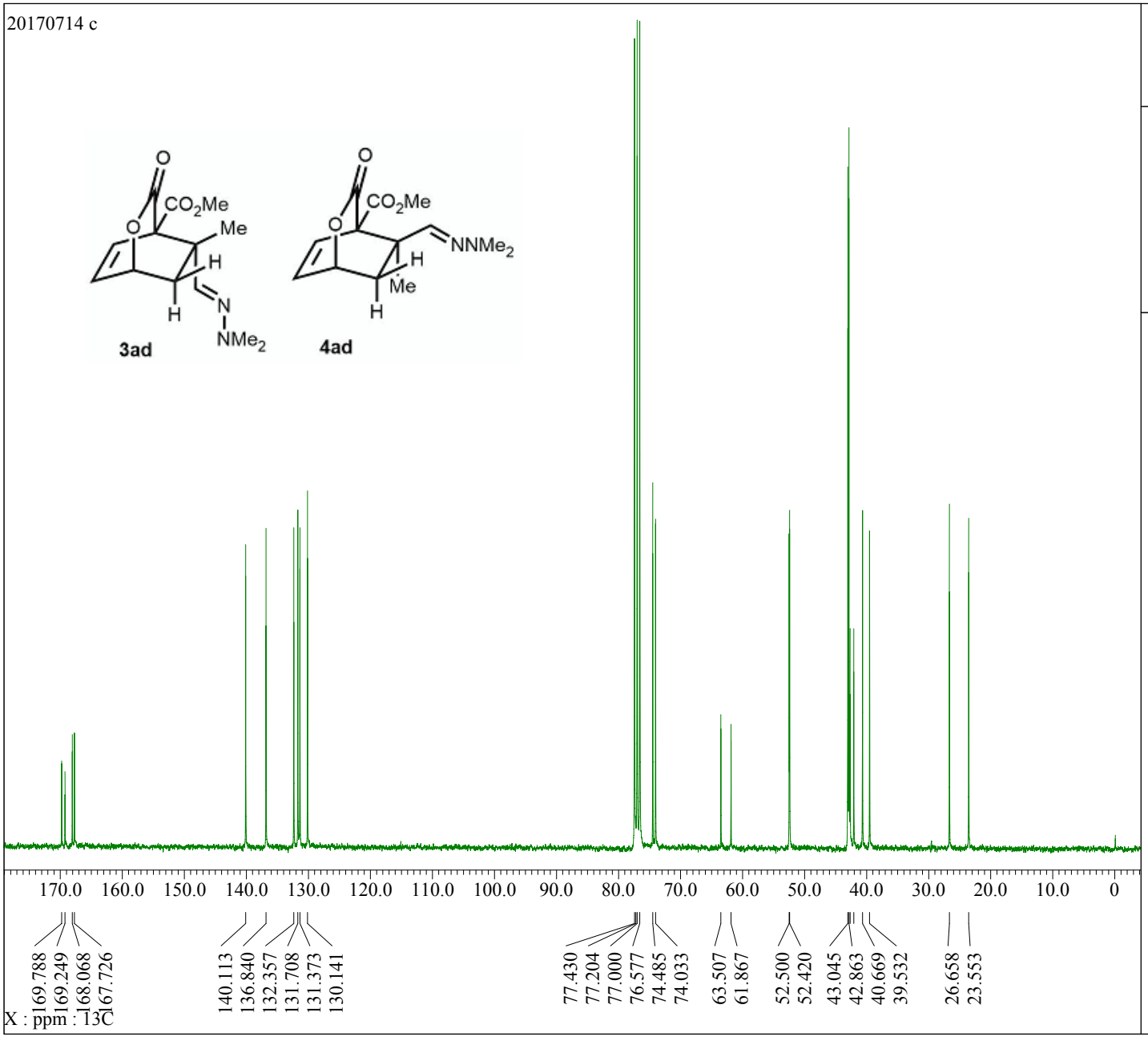
X : ppm : 1H

JEOL Solutions for Innovation

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
phase( -25, -140, 50[%] )
```

```
Filename       = Y-Abe_900 2-1.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = 20170714 2
Solvent        = CHLOROFORM-D
Creation_Time  = 19-JUL-2017 18:06:35
Revision_Time  = 19-JUL-2017 18:10:14
Current_Time   =  7-SEP-2017 14:24:59

Comment        = 20170714 2
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       = 295.56[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```

20170714 c

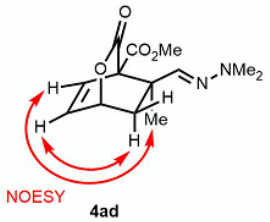


JEOL
Solutions for Innovation

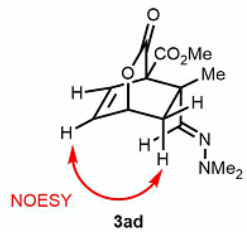
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase

以下に由来:: Y-Abe_882-1.jdf
```

```
Filename       = Y-Abe_882-5.jdf
Author         = Administrator
Experiment     = zgpg30
Sample_Id      = 20170714 c
Solvent        = CHLOROFORM-D
Creation_Time  =  7-SEP-2017 14:26:36
Revision_Time  =  7-SEP-2017 14:26:54
Current_Time   =  7-SEP-2017 14:29:06

Comment        = 20170714 c
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       = 297.26[K]
X_Points       = 32768
X_Prescans     = 4
Filter_Factor  = 1109
Scans          = 4096
```

20170714 noesy



4ad



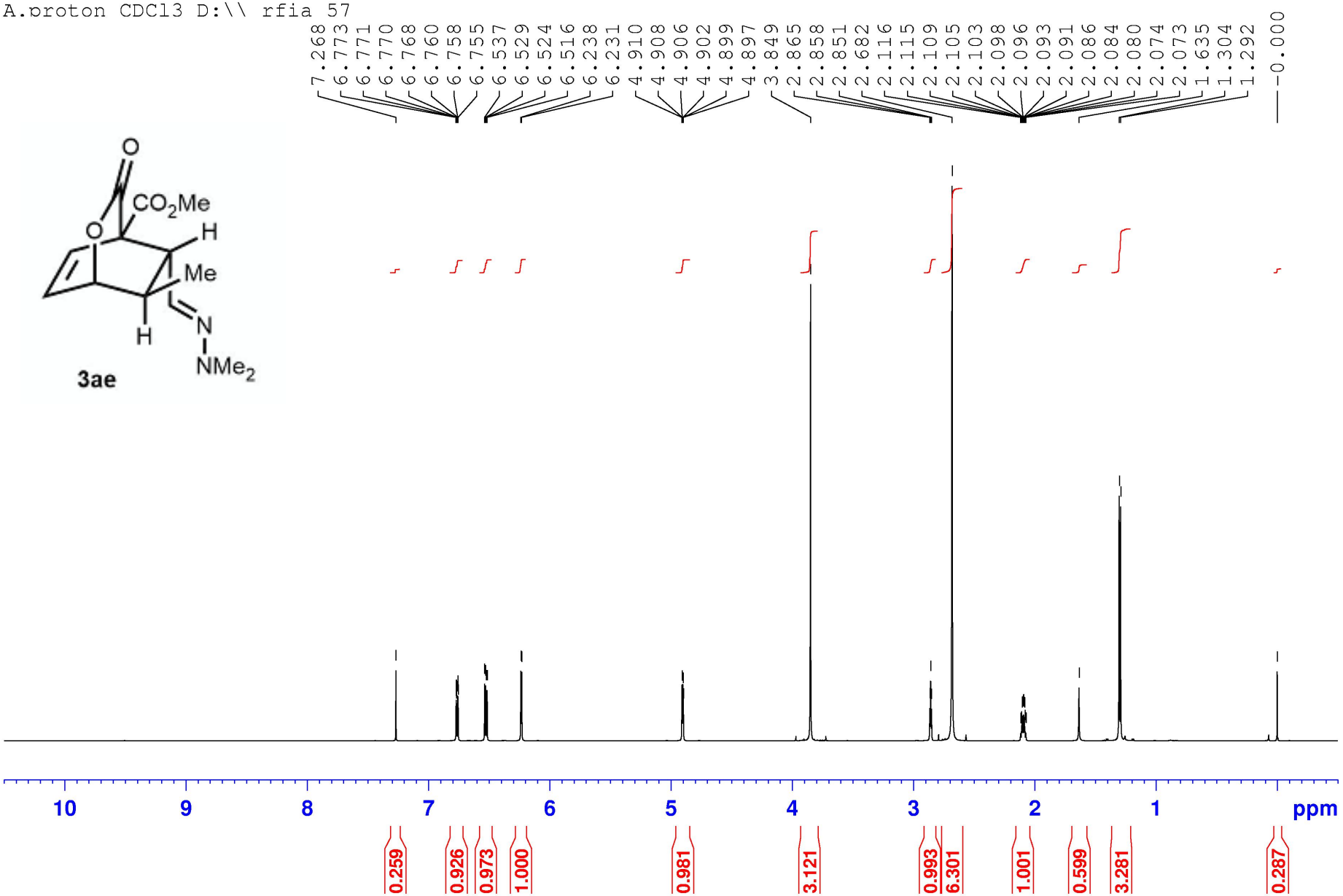
3ad

ppm

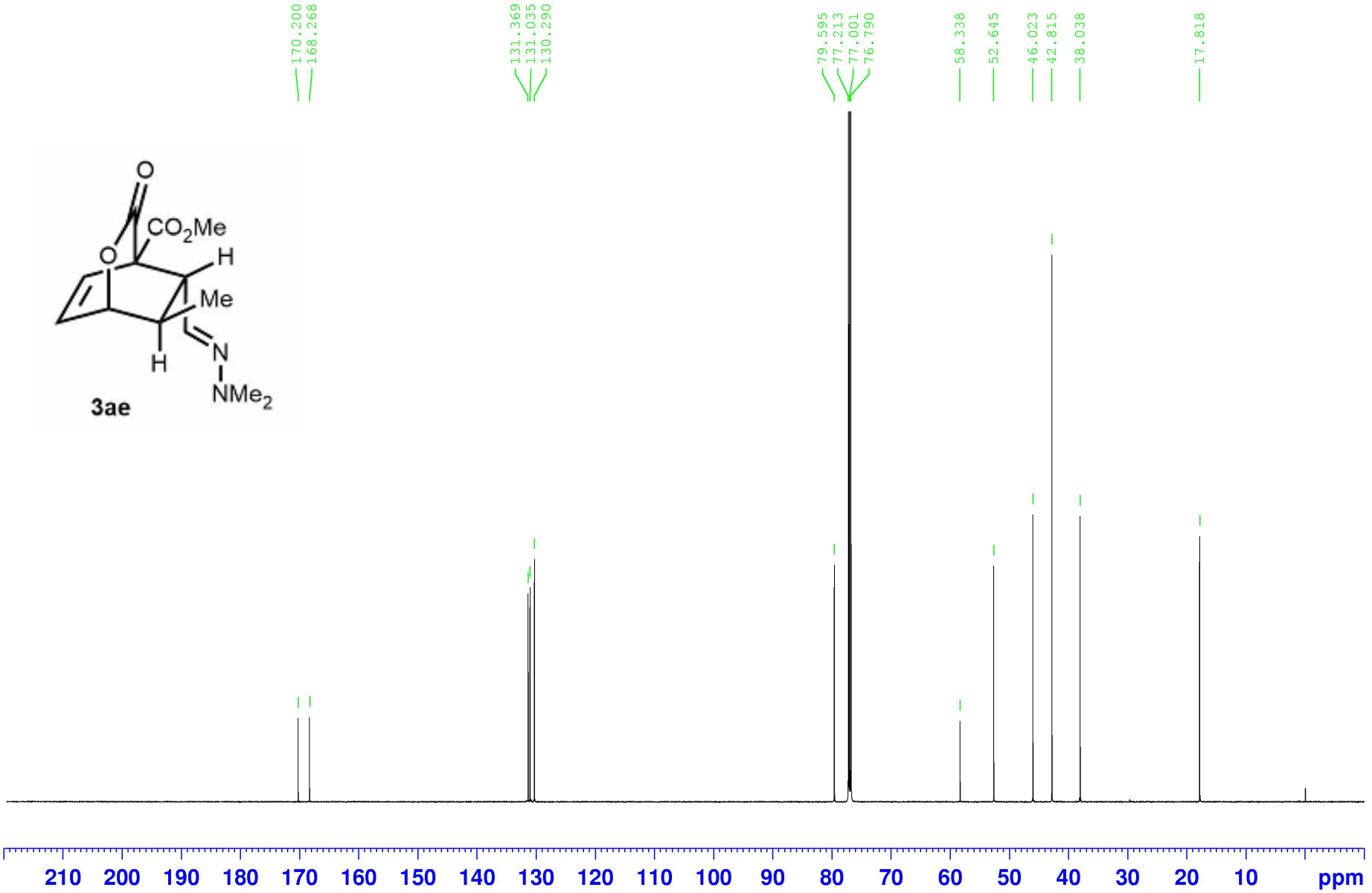
1.0 1.5 2.0 2.5 3.0 3.5 4.0 4.5 5.0 5.5 6.0 6.5 7.0 7.5

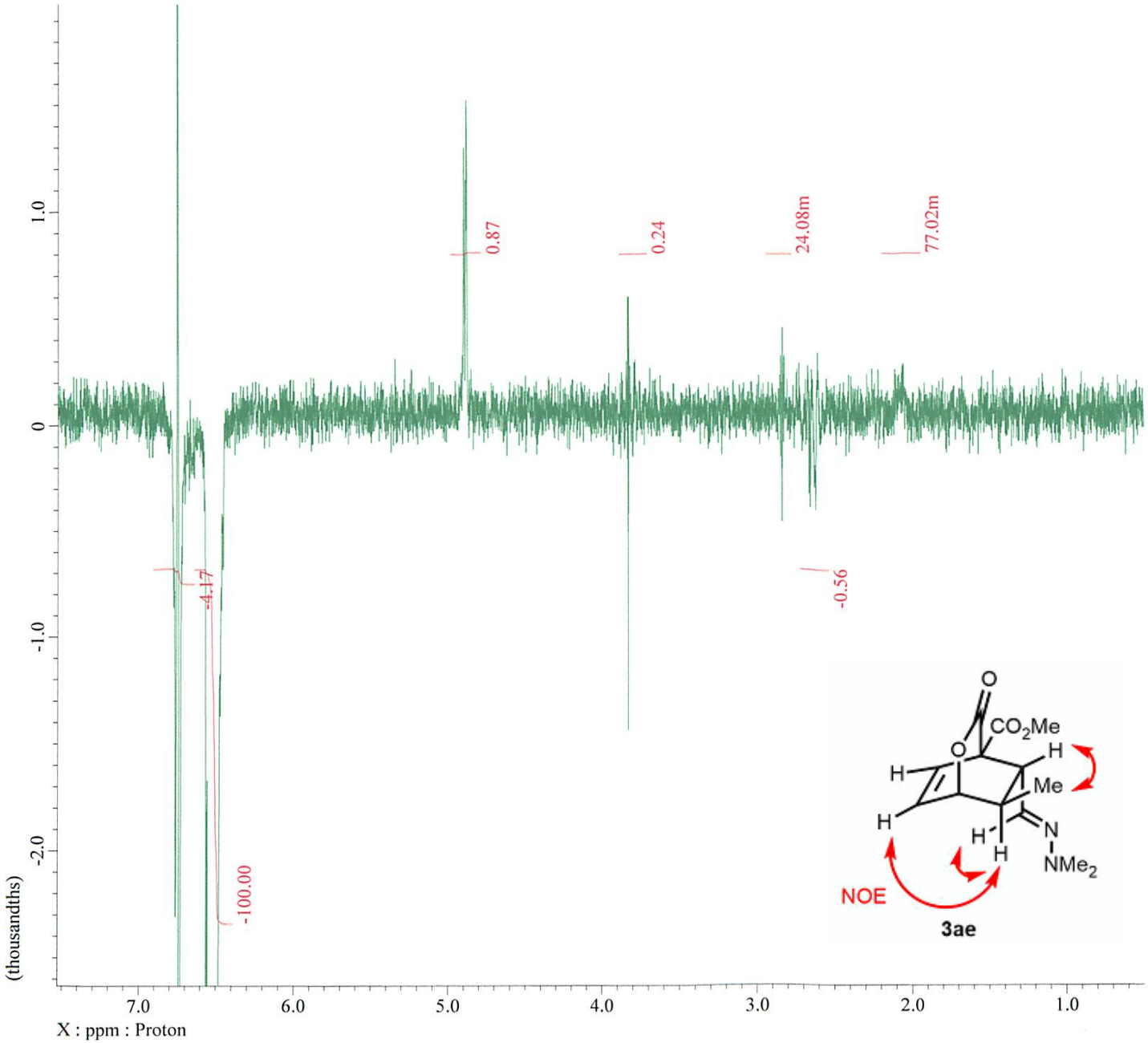
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

YH_0316_2
A.proton CDCl3 D:\\ rfia 57



YH_0316_2
A.carbon CDCl3 D:\\ rfia 57

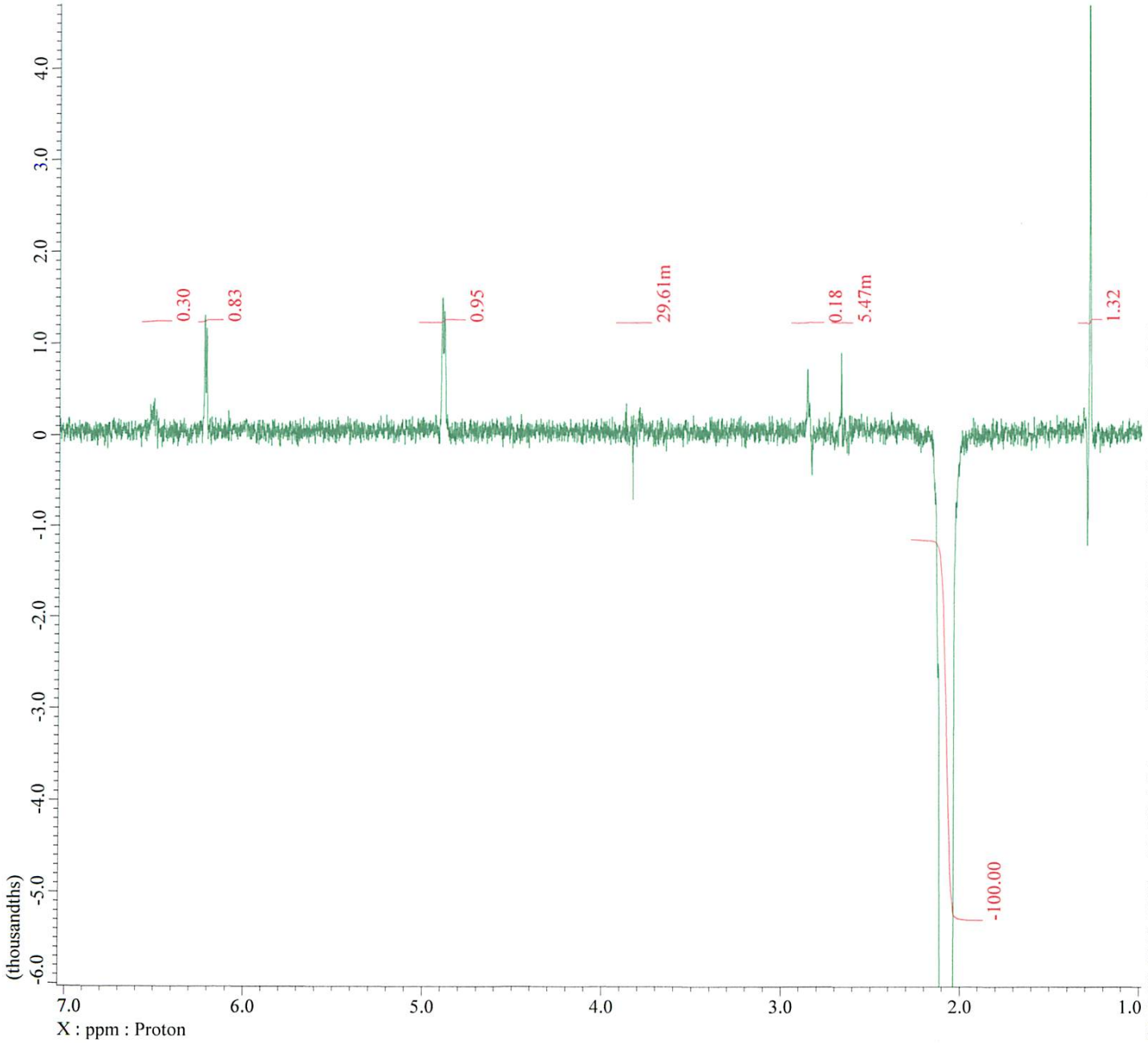




3ae

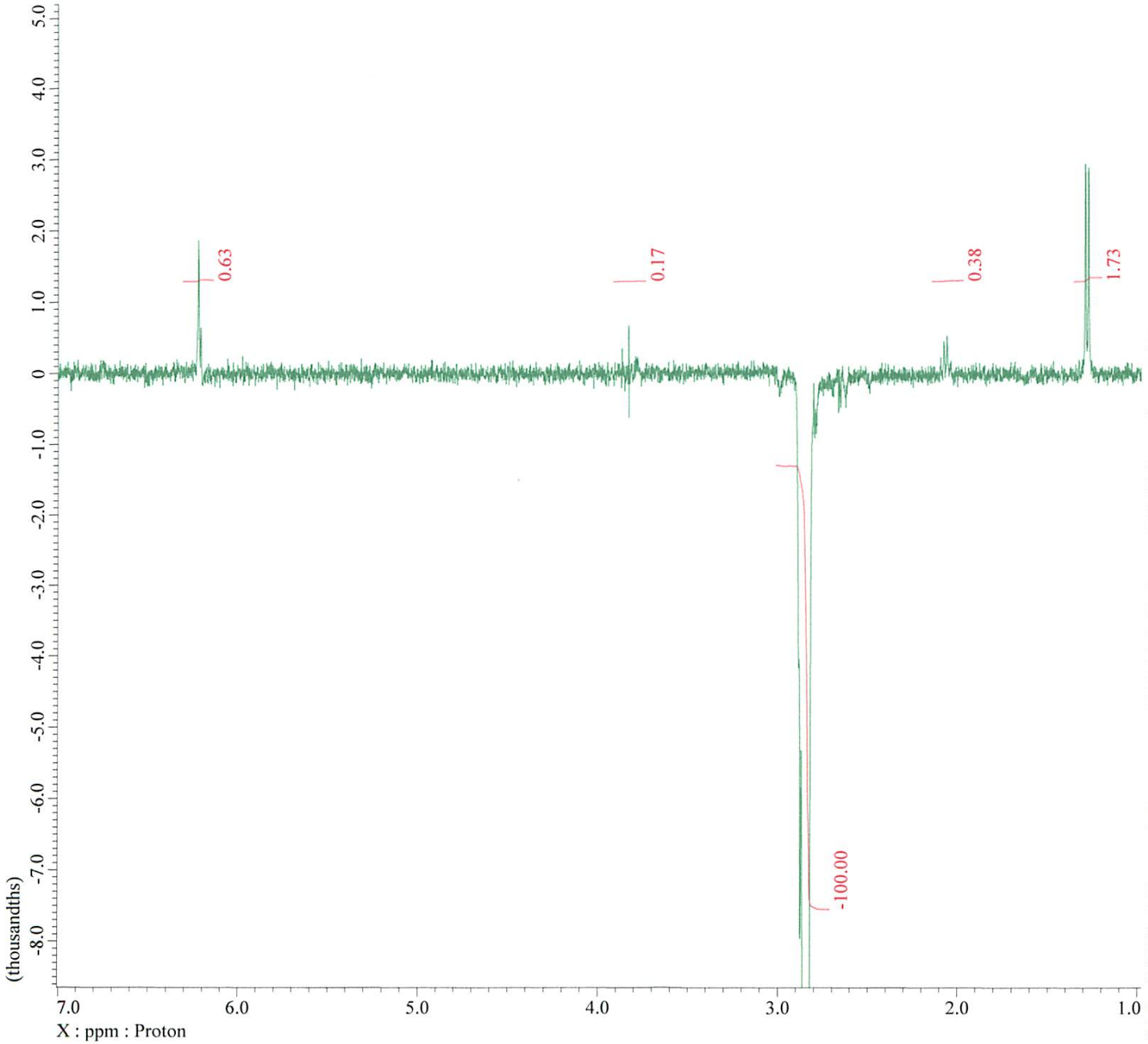
```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm

以下に由来:: YH_0319_1_noe_1d-2-1.jdf
```

Filename	= YH_0319_1_noe_1d-2-7.
Author	= delta
Experiment	= noe_1d.jxp
Sample_Id	= YH_0319_1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-OCT-2017 11:31:23
Revision_Time	= 20-OCT-2017 12:53:10
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
Temp_Get	= 21.8[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 3[dB]
X_Pulse	= 6.1[us]
Irr_Mode	= Off
Obs_Sel_180	= 60[ms]
Obs_Sel_Atn	= 69.125[dB]
Obs_Sel_Offset	= 6.52819633[ppm]



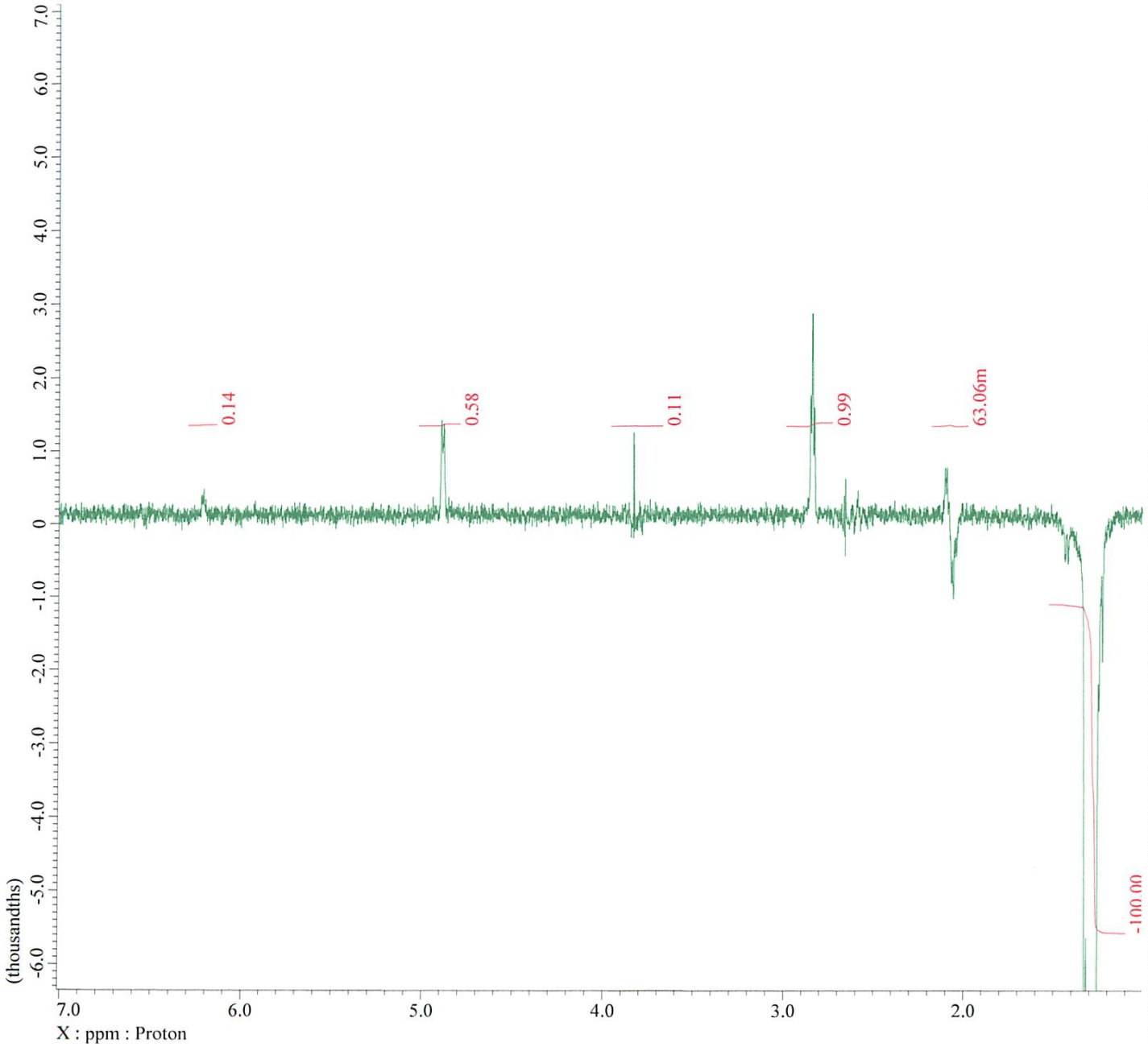

```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm

以下に由来:: YH_0319_1_noe_1d-6-1.jdf
```

Filename	= YH_0319_1_noe_1d-6-6.
Author	= delta
Experiment	= noe_1d.jxp
Sample_Id	= YH_0319_1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-OCT-2017 11:43:35
Revision_Time	= 20-OCT-2017 13:20:15
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
Temp_Get	= 21.8[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 3[dB]
X_Pulse	= 6.1[us]
Irr_Mode	= Off
Obs_Sel_180	= 60[ms]
Obs_Sel_Atn	= 69.125[dB]
Obs_Sel_Offset	= 2.0926187[ppm]




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

以下に由来:: YH_0319_1_noe_1d-5-1.jdf
```

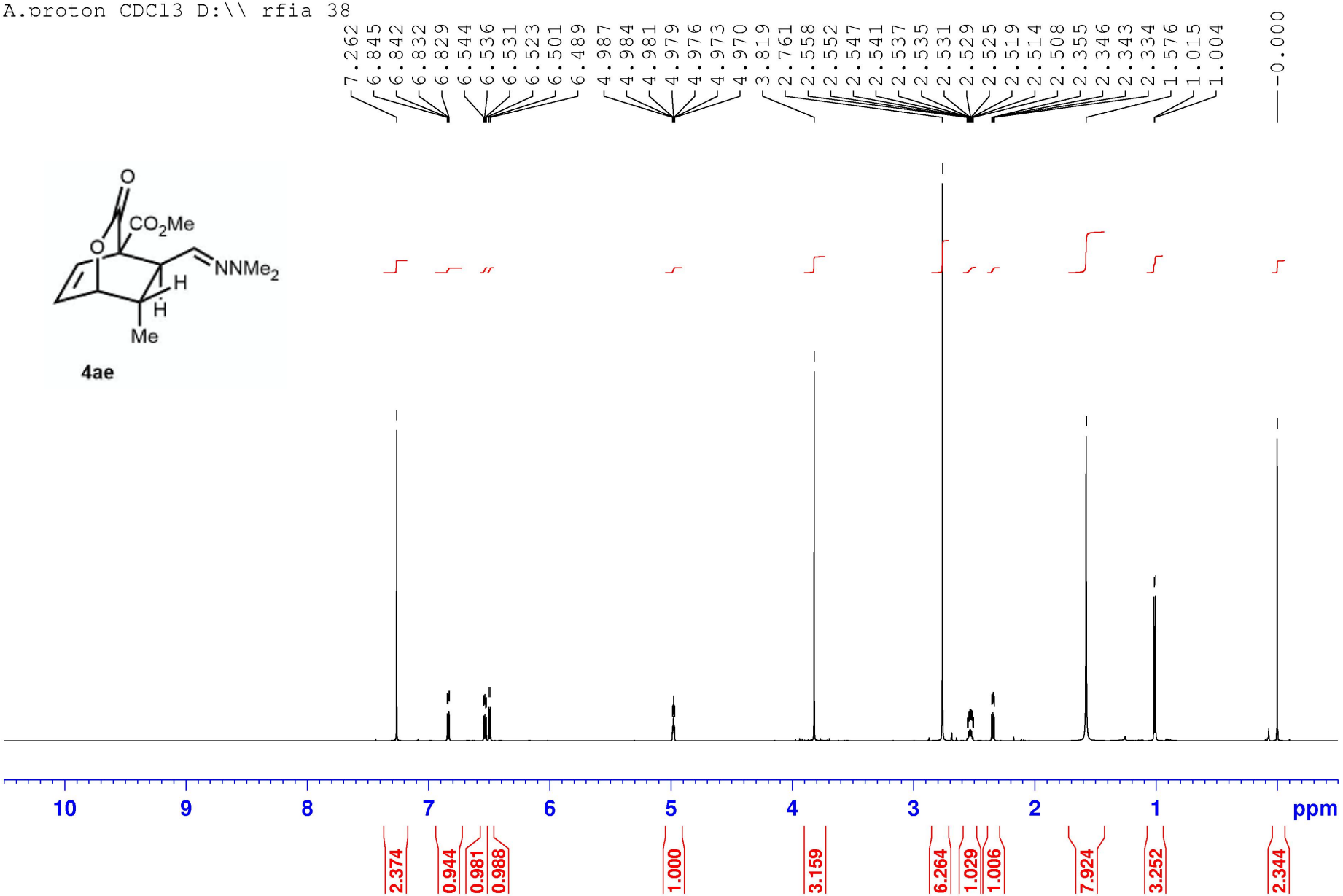
Filename	= YH_0319_1_noe_1d-5-6.
Author	= delta
Experiment	= noe_1d.jxp
Sample_Id	= YH_0319_1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-OCT-2017 11:40:32
Revision_Time	= 20-OCT-2017 13:17:49
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
Temp_Get	= 21.8[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 3[dB]
X_Pulse	= 6.1[us]
Irr_Mode	= Off
Obs_Sel_180	= 60[ms]
Obs_Sel_Atn	= 69.125[dB]
Obs_Sel_Offset	= 2.86090207[ppm]



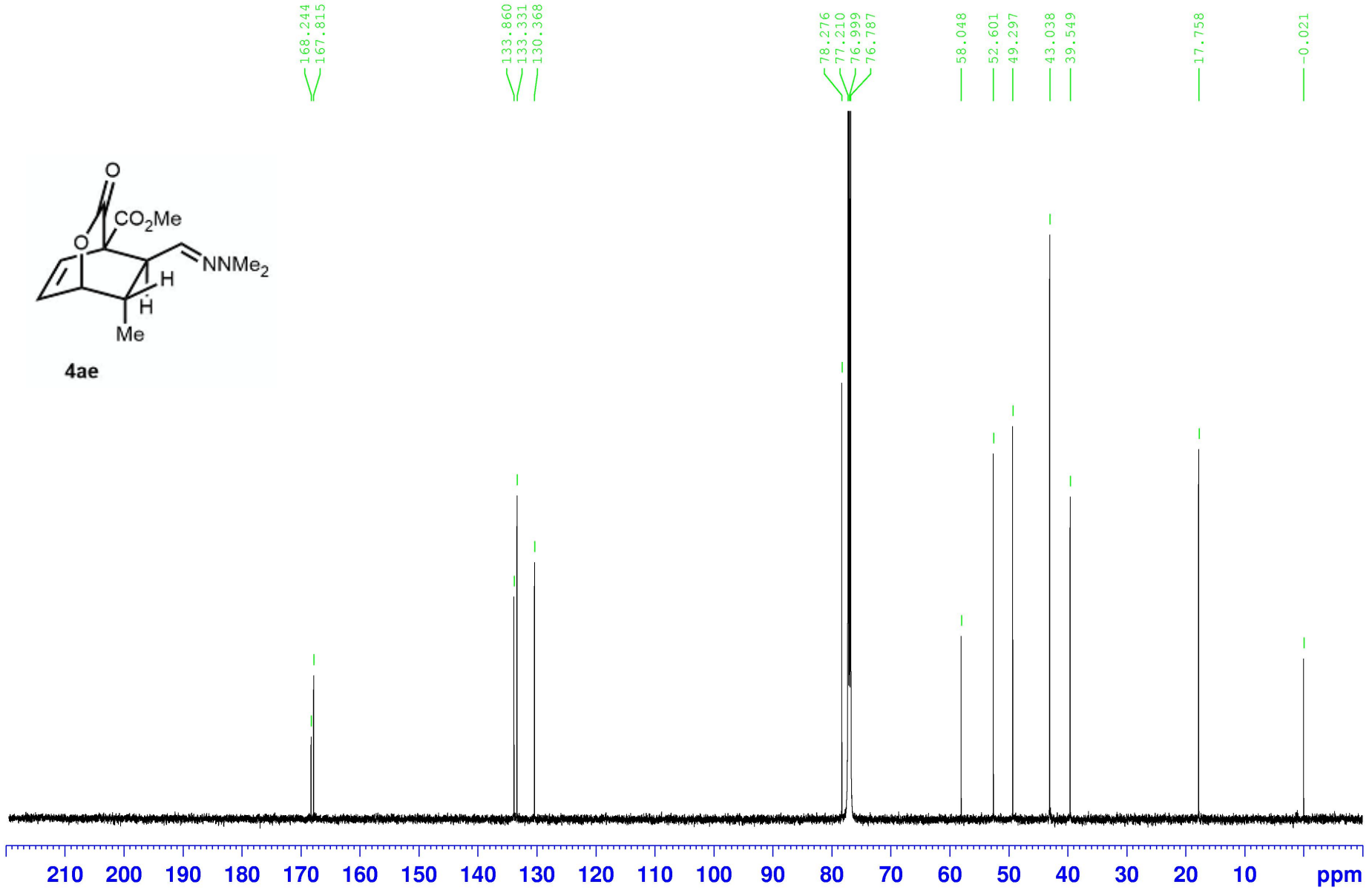

```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm

以下に由来:: YH_0319_1_noe_1d-7-1.jdf
```

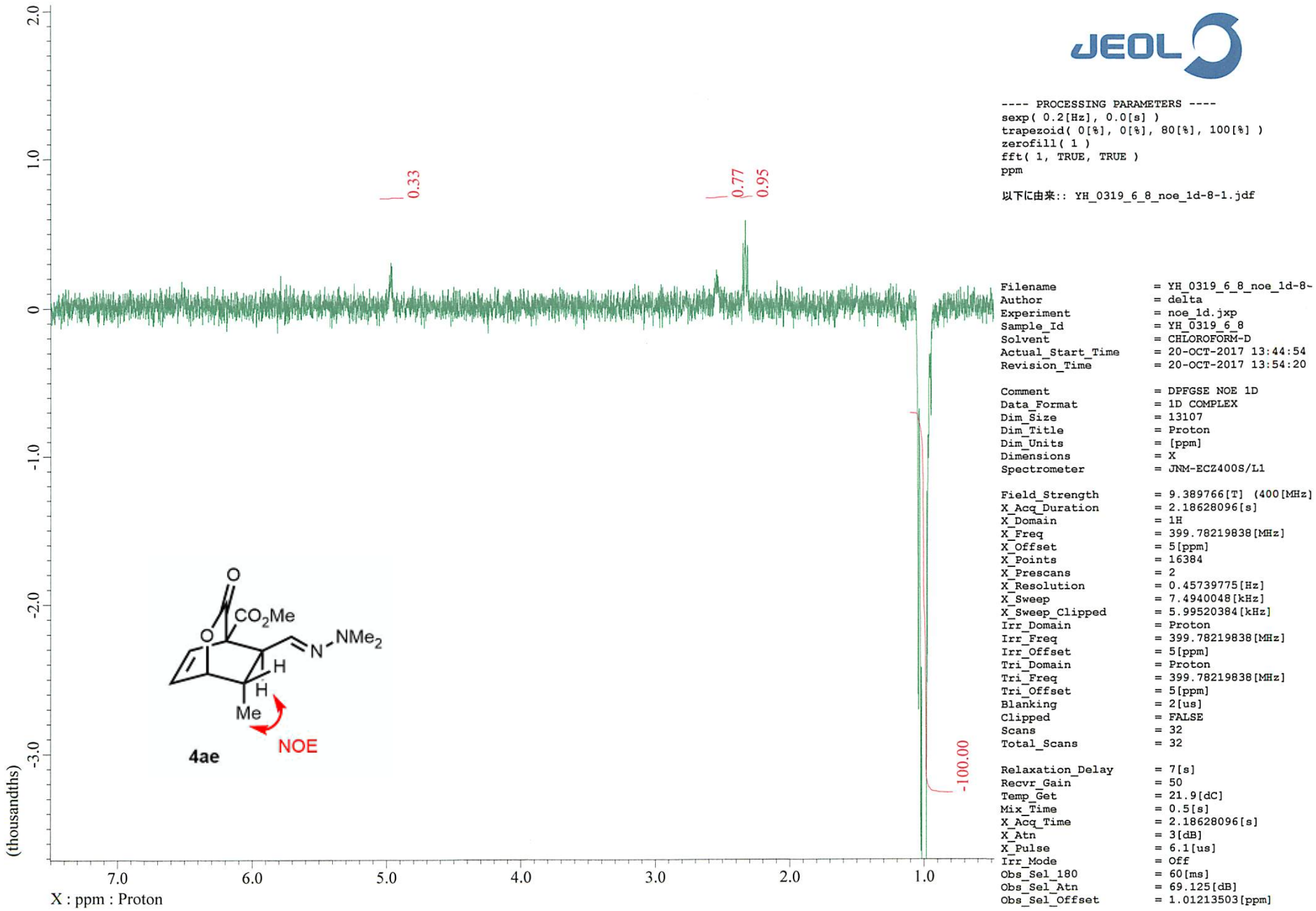
Filename	= YH_0319_1_noe_1d-7-7.
Author	= delta
Experiment	= noe_1d.jxp
Sample_Id	= YH_0319_1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-OCT-2017 11:46:38
Revision_Time	= 20-OCT-2017 13:23:20
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 16
Total_Scans	= 16
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
Temp_Get	= 21.8[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 3[dB]
X_Pulse	= 6.1[us]
Irr_Mode	= Off
Obs_Sel_180	= 60[ms]
Obs_Sel_Atn	= 69.125[dB]
Obs_Sel_Offset	= 1.29994524[ppm]

YH_0319_8
A.proton CDCl3 D:\\ rfia 38



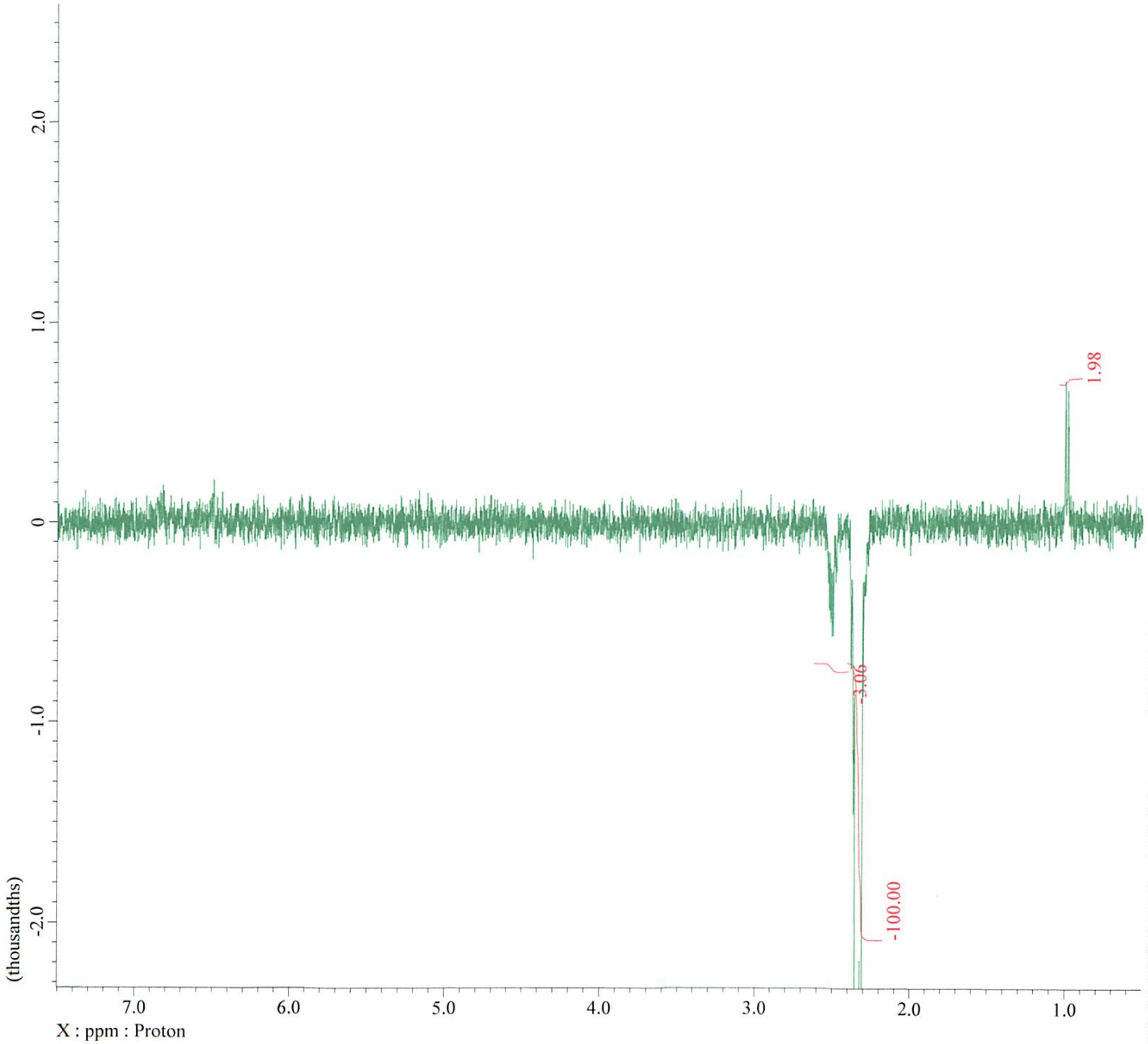
YH_0319_8
A.carbon CDCl3 D:\\ rfia 38






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

以下に由来:: YH_0319_6_8_noe_1d-8-1.jdf
```

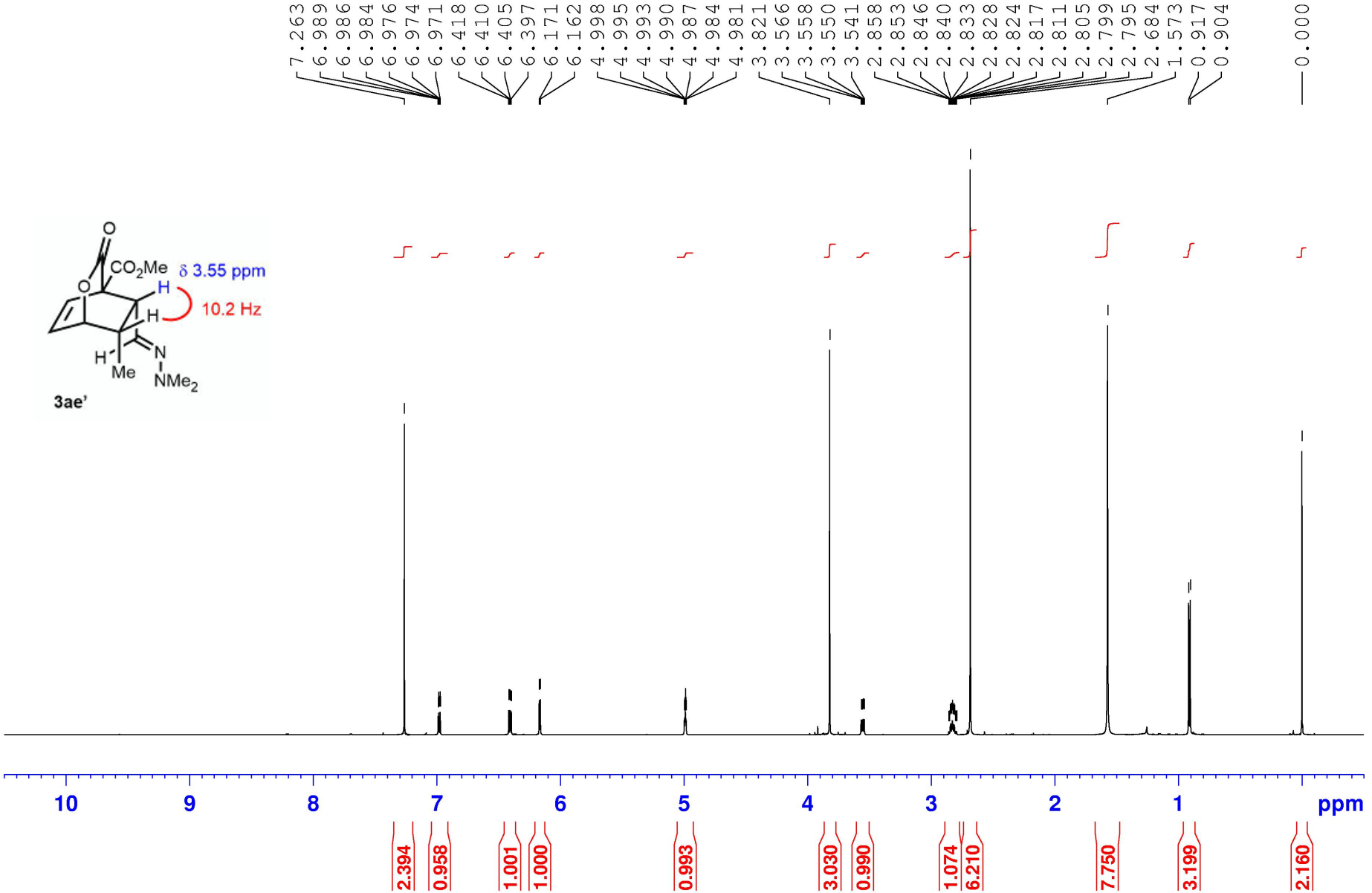
Filename	= YH_0319_6_8_noe_1d-8~
Author	= delta
Experiment	= noe_1d.jxp
Sample_Id	= YH_0319_6_8
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-OCT-2017 13:44:54
Revision_Time	= 20-OCT-2017 13:54:20
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
Temp_Get	= 21.9[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 3[dB]
X_Pulse	= 6.1[us]
Irr_Mode	= Off
Obs_Sel_180	= 60[ms]
Obs_Sel_Atn	= 69.125[dB]
Obs_Sel_Offset	= 1.01213503[ppm]




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

以下に由来:: YH_0319_6_8_noe_1d-7-1.jdf
```

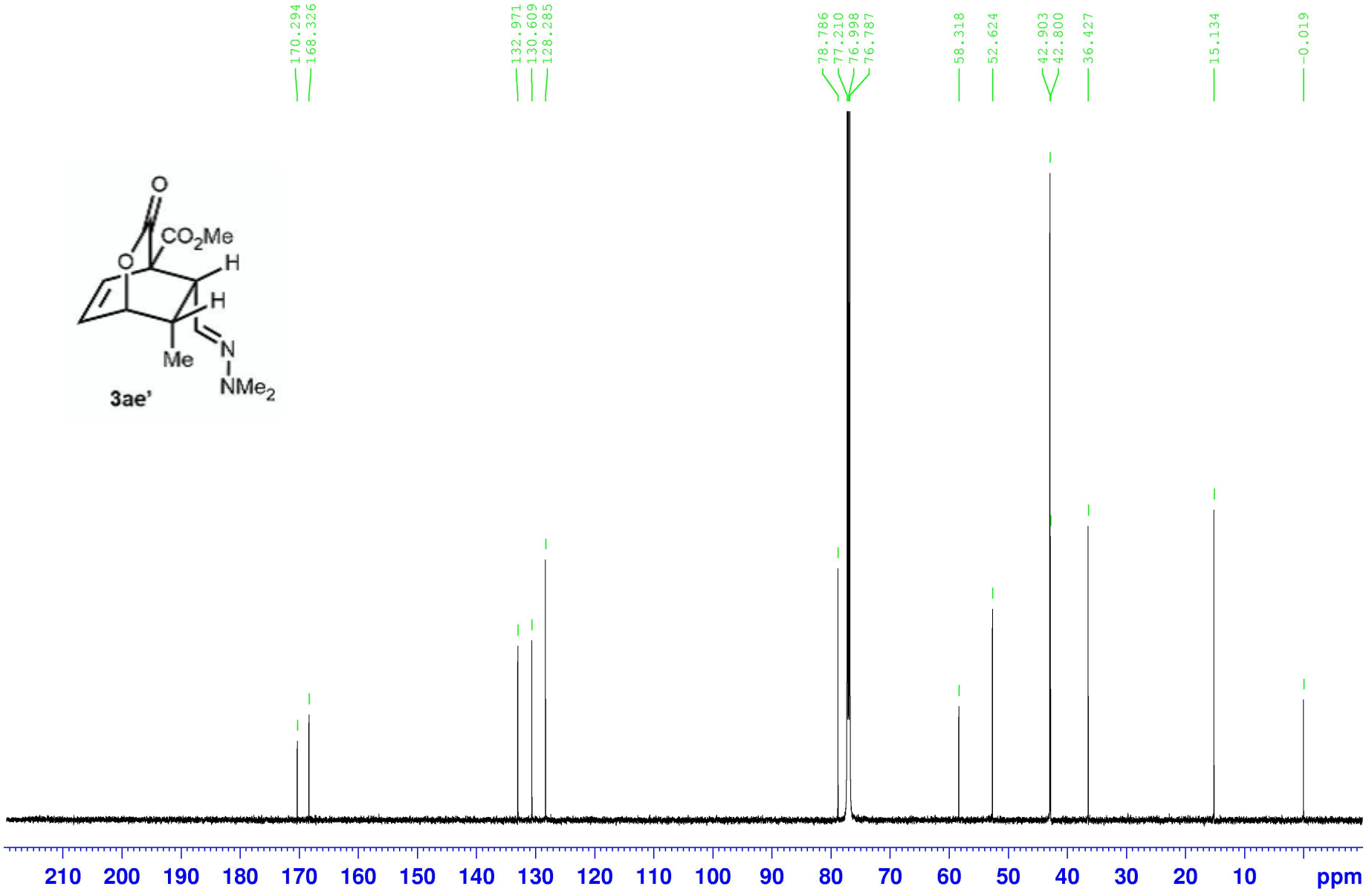
Filename	= YH_0319_6_8_noe_1d-7-
Author	= delta
Experiment	= noe_1d.jxp
Sample_Id	= YH_0319_6_8
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-OCT-2017 13:39:12
Revision_Time	= 20-OCT-2017 13:52:00
Comment	= DPFGSE NOE 1D
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ400S/L1
Field_Strength	= 9.389766[T] (400[MHz]
X_Acq_Duration	= 2.18628096[s]
X_Domain	= 1H
X_Freq	= 399.78219838[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 2
X_Resolution	= 0.45739775[Hz]
X_Sweep	= 7.4940048[kHz]
X_Sweep_Clipped	= 5.99520384[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.78219838[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.78219838[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 32
Total_Scans	= 32
Relaxation_Delay	= 7[s]
Recvr_Gain	= 50
Temp_Get	= 21.9[dC]
Mix_Time	= 0.5[s]
X_Acq_Time	= 2.18628096[s]
X_Atn	= 3[dB]
X_Pulse	= 6.1[us]
Irr_Mode	= Off
Obs_Sel_180	= 60[ms]
Obs_Sel_Atn	= 69.125[dB]
Obs_Sel_Offset	= 2.34604478[ppm]

YH_0319_5
A.proton CDCl3 D:\\ rfia 35

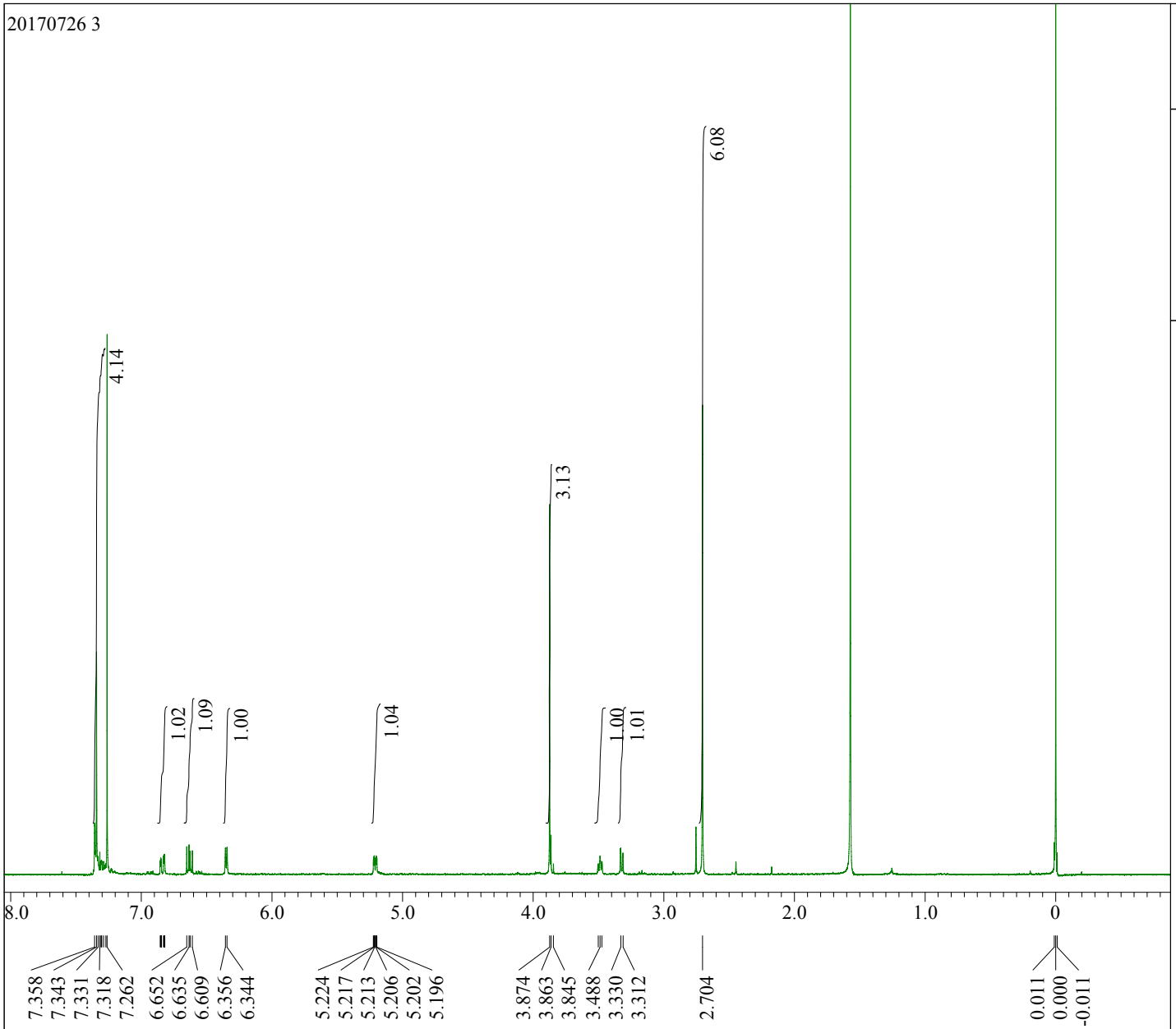


YH_0319_5
A.carbon CDCl3 D:\\ rfia 35

3ae'



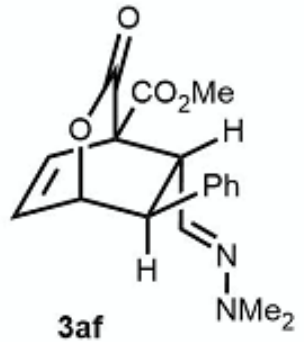
20170726 3



JEOL Solutions for Innovation

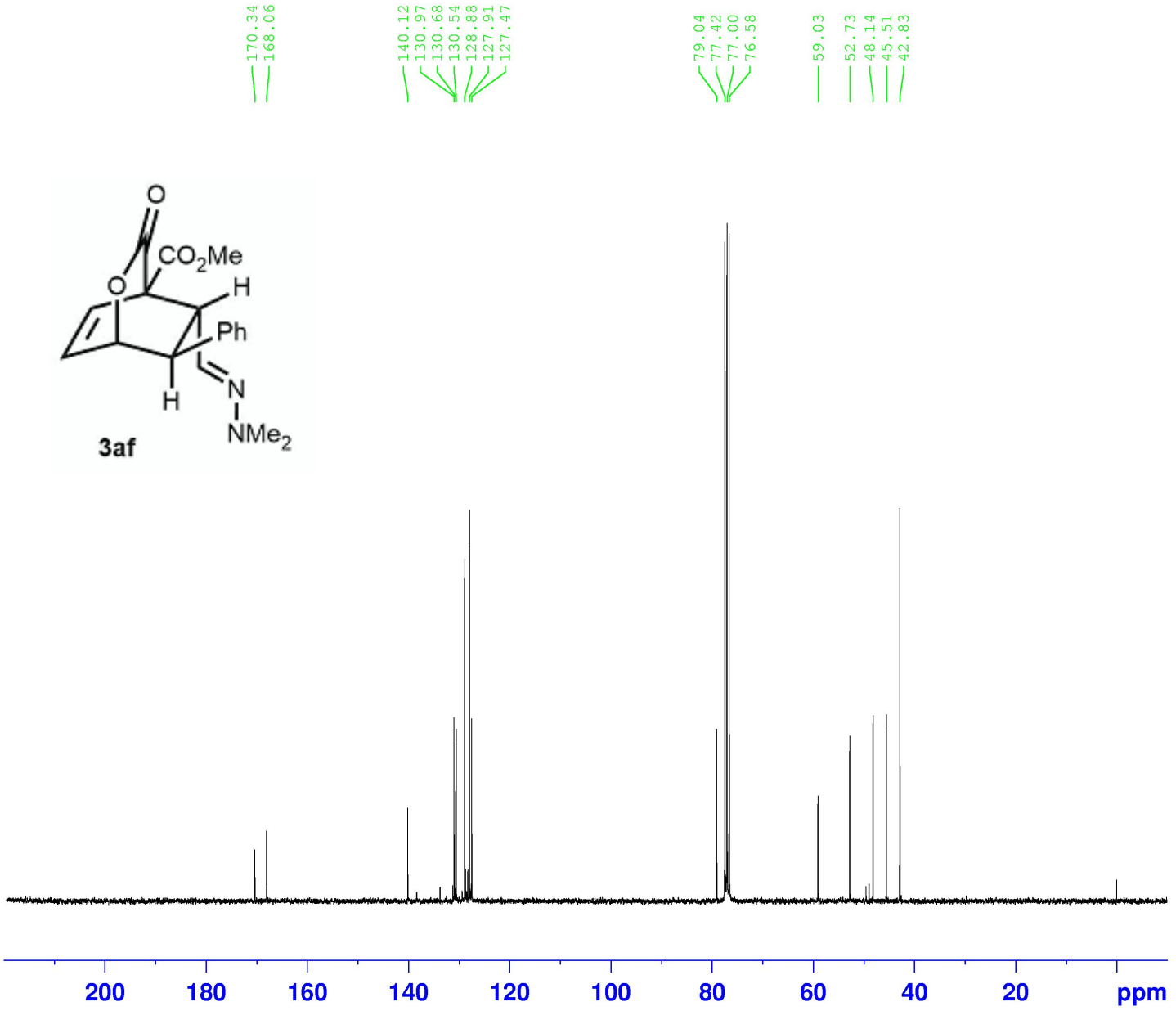
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2, 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
ppm
machinephase
```

```
Filename       = Y-Abe_990-2.jdf
Author         = Administrator
Experiment     = zg30
Sample_Id      = 20170726 3
Solvent        = CHLOROFORM-D
Creation_Time  = 26-JUL-2017 18:52:52
Revision_Time  =  7-SEP-2017 14:33:32
Current_Time   =  7-SEP-2017 14:33:48

Comment        = 20170726 3
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       = 295.46[K]
X_Points       = 32768
X_Prescans     = 2
Filter_Factor  = 3232
Scans          = 16
```



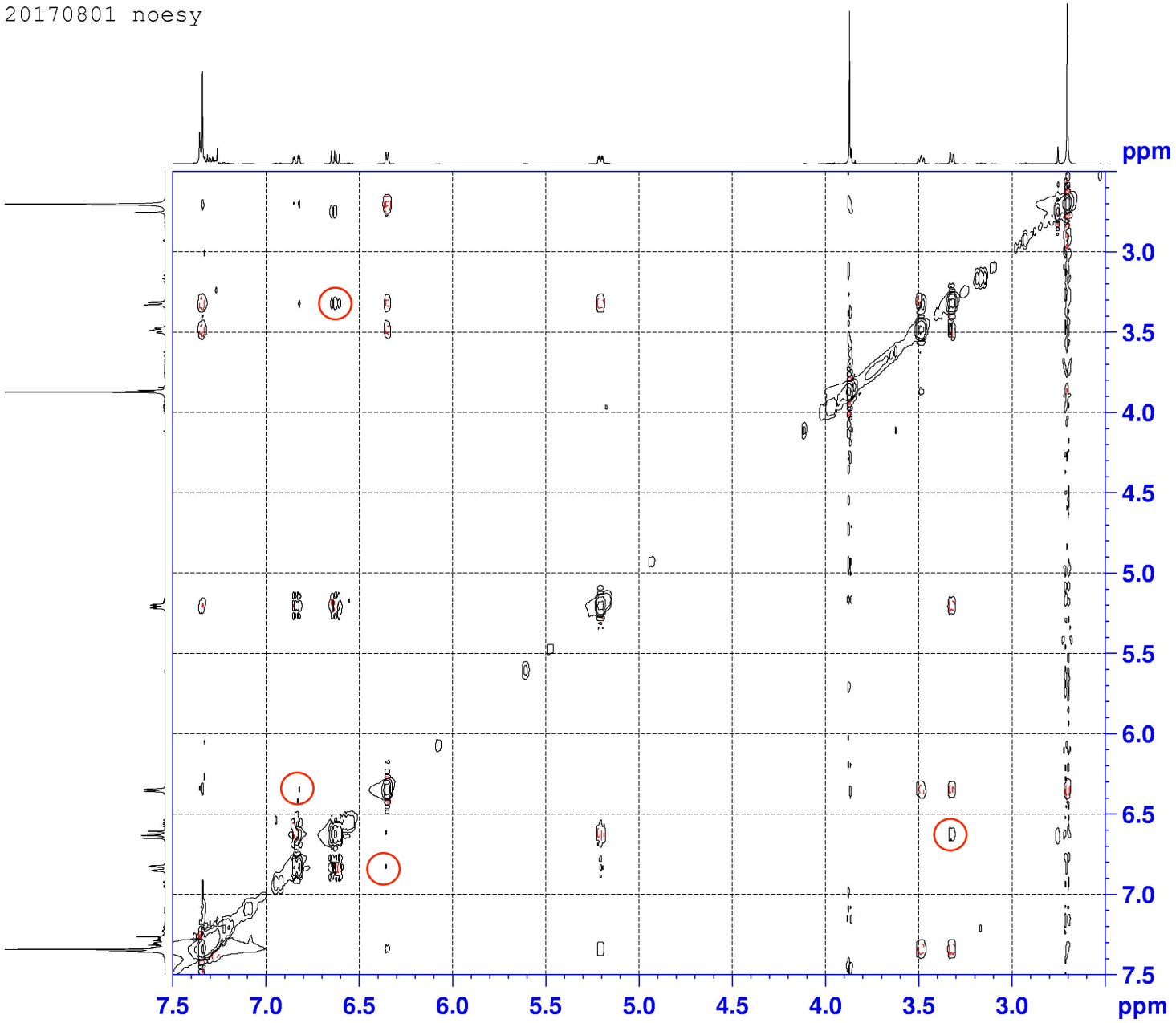
3af

20170801 c

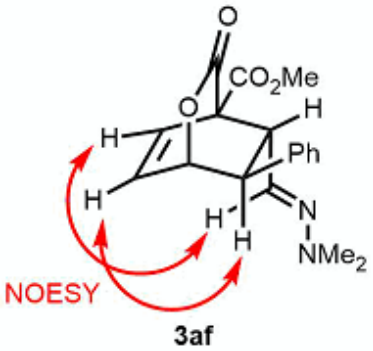



```
Current Data Parameters
NAME                  Y-Abe
EXPNO                  1042
PROCNO                    1

F2 - Acquisition Parameters
Date_              20170802
Time                   5.40
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     4096
DS                        4
SWH             18028.846 Hz
FIDRES           0.275098 Hz
AQ               1.8175317 sec
RG                     2050
DW                27.733 usec
DE                 6.50 usec
TE                 297.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           75.4752953 MHz
NUC1                   13C
P1                 10.00 usec
PLW1          34.00000000 W

======== CHANNEL f2 ========
SFO2          300.1312005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2           6.19999981 W
PLW12          0.21797000 W
PLW13          0.13950001 W

F2 - Processing parameters
SI                    32768
SF             75.4677509 MHz
WDW                      EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

20170801 noesy

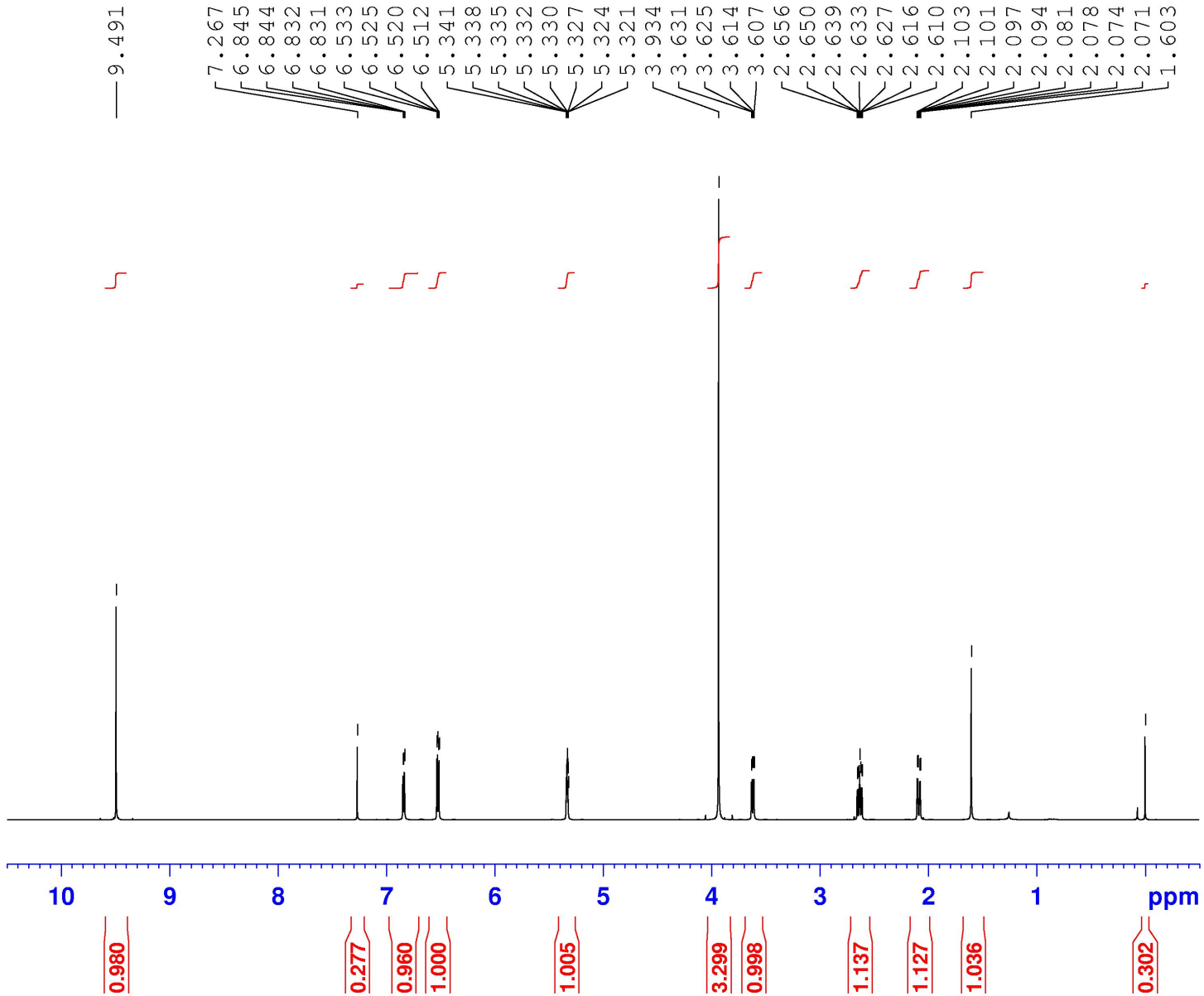



```
Current Data Parameters
NAME                  Y-Abe
EXPNO                  1041
PROCNO                    1

F2 - Acquisition Parameters
Date_              20170801
Time                 21.14
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG            noesyph
TD                    2048
SOLVENT               CDCl3
NS                      16
DS                       4
SWH               2415.459 Hz
FIDRES            1.179423 Hz
AQ                0.4239360 sec
RG                      32
DW                 207.000 usec
DE                   6.50 usec
TE                  295.7 K
D0              0.00018822 sec
D1              1.91029704 sec
D8              0.89999998 sec
IN0             0.00041400 sec

======== CHANNEL f1 ========
SFO1           300.1313051 MHz
NUC1                    1H
P1                   14.75 usec
PLW1           6.19999981 W

F1 - Acquisition parameters
TD                      256
SFO1              300.1313 MHz
FIDRES            9.435387 Hz
SW                   8.048 ppm
FnMODE         States-TPPI
```

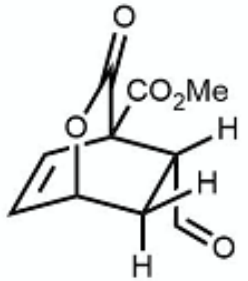


3af

YH_0348_1_2nd
A.proton CDCl3 D:\\ rfia 51

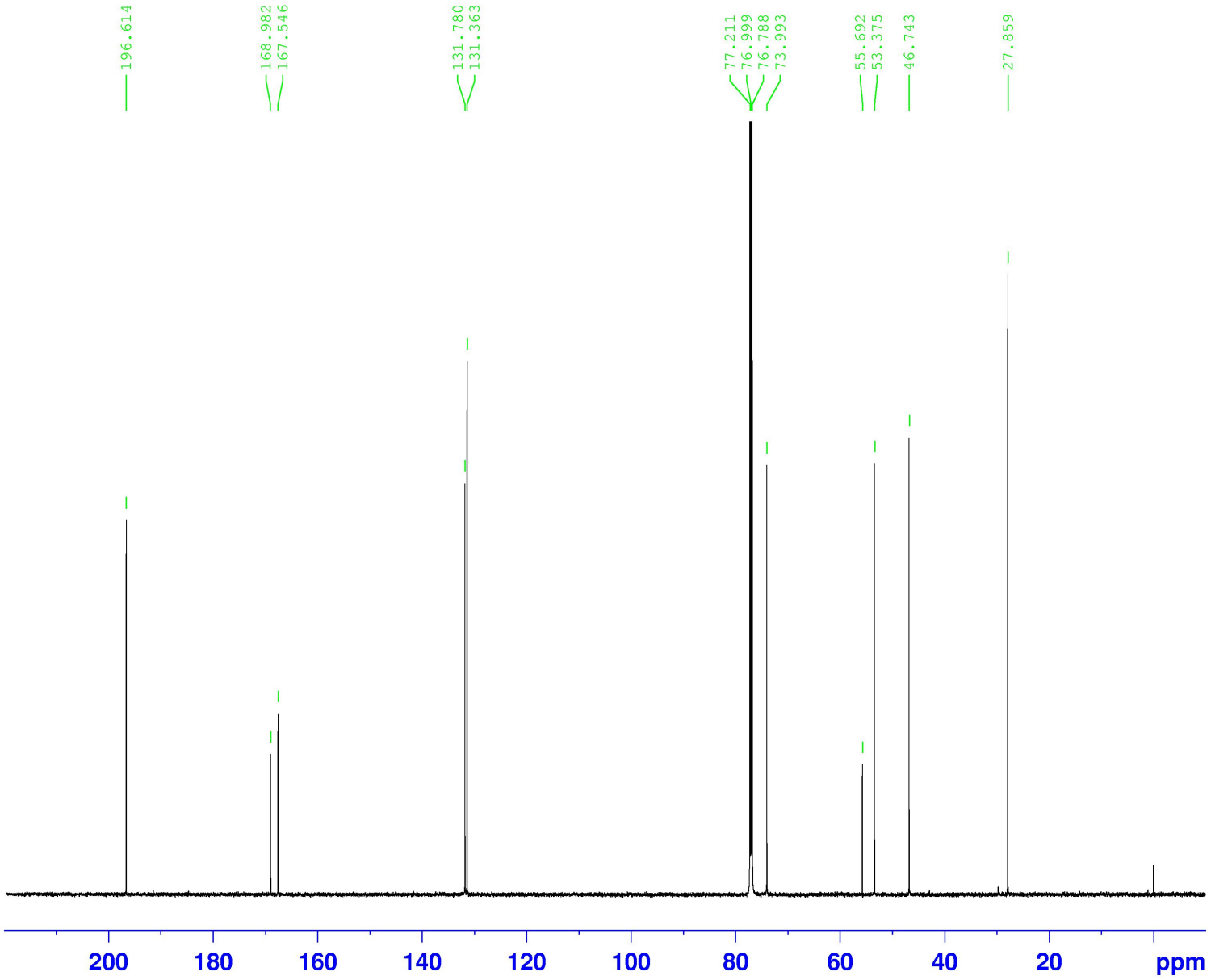



```
Current Data Parameters
NAME                  Hashimoto
EXPNO                      1630
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20180801
Time                      15.21
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                    zg30
TD                        32768
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                  12335.526 Hz
FIDRES                0.376450 Hz
AQ                    1.3281963 sec
RG                         20.2
DW                       40.533 usec
DE                       10.00 usec
TE                       298.0 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                600.1337060 MHz
NUC1                        1H
P1                        7.80 usec
PLW1                 8.80000019 W

F2 - Processing parameters
SI                        32768
SF                  600.1300057 MHz
WDW                          EM
SSB                0
LB                        0.30 Hz
GB                 0
PC                        1.00
```

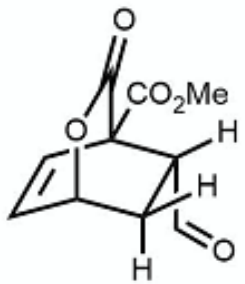


3aa

YH_0348_1_2nd
A.carbon CDCl3 D:\\ rfia 51

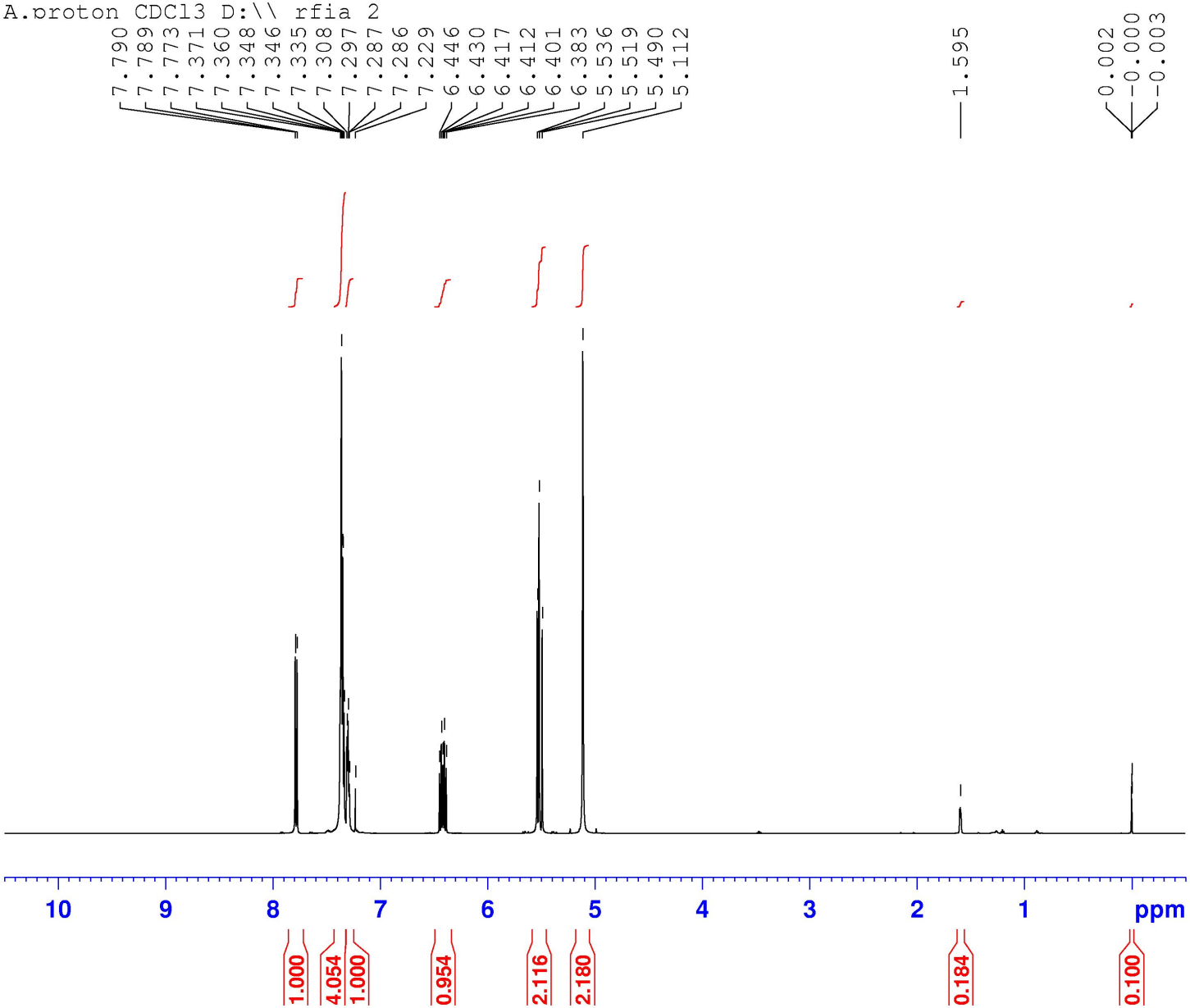



```
Current Data Parameters
NAME            Hashimoto
EXPNO                1632
PROCNO                  1

F2 - Acquisition Parameters
Date_            20180801
Time                17.10
INSTRUM             spect
PROBHD   5 mm CPTCI 1H-
PULPROG            zgpg30
TD                  65356
SOLVENT             CDCl3
NS                   2048
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        107.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              70.00 usec
PLW2          8.80000019 W
PLW12         0.10926000 W
PLW13         0.05353900 W

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

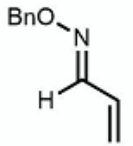


3aa

20170711_ikeda_55_1
A.proton CDCl3 D:\\ rfia 2

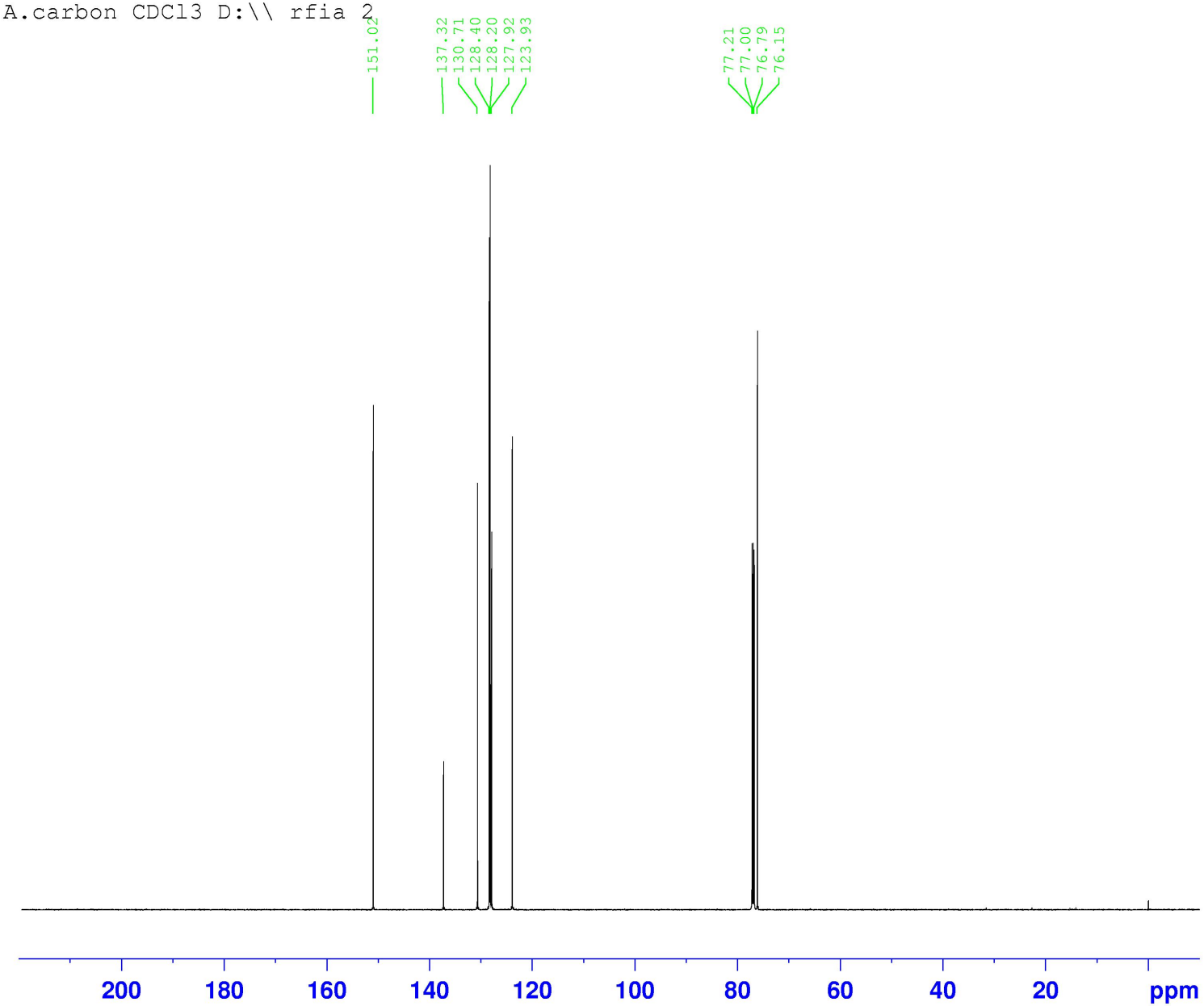



```
Current Data Parameters
NAME            Hashimoto
EXPNO                 790
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170711
Time                23.03
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            12335.526 Hz
FIDRES          0.376450 Hz
AQ              1.3281963 sec
RG                  11.3
DW                40.533 usec
DE                10.00 usec
TE                298.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                  7.40 usec
PLW1          8.80000019 W

F2 - Processing parameters
SI                 32768
SF           600.1300348 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

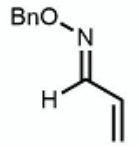


2c

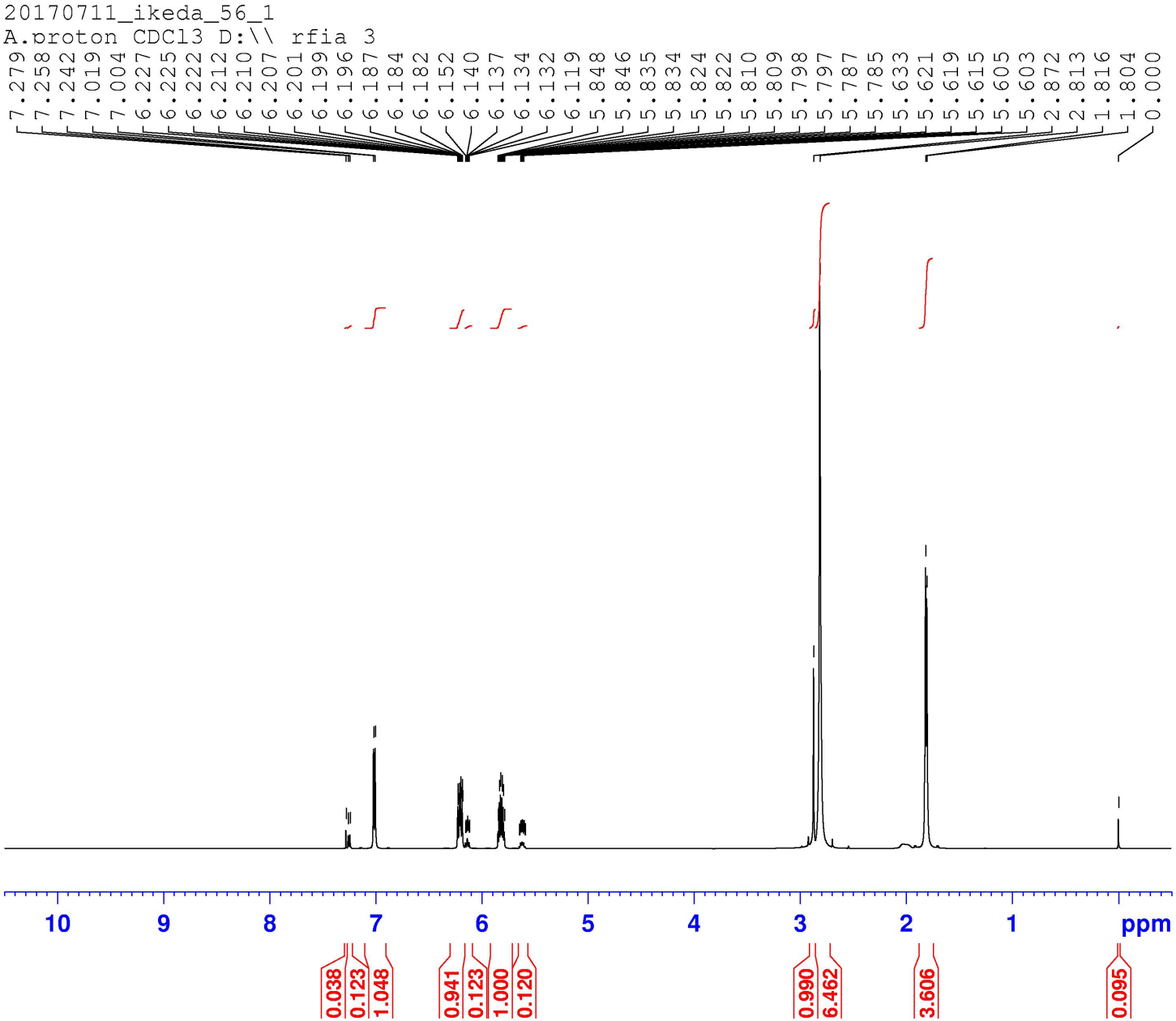
20170711_ikeda_55_1
A.carbon CDCl3 D:\\ rfia 2



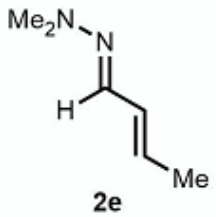

```
Current Data Parameters
NAME                  Hashimoto
EXPNO                       791
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20170712
Time                       0.45
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                 zgpg30
TD                       65356
SOLVENT                  CDCl3
NS                        2048
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                  150.9028202 MHz
WDW                          EM
SSB               0
LB                        1.00 Hz
GB                0
PC                        1.40
```



2c

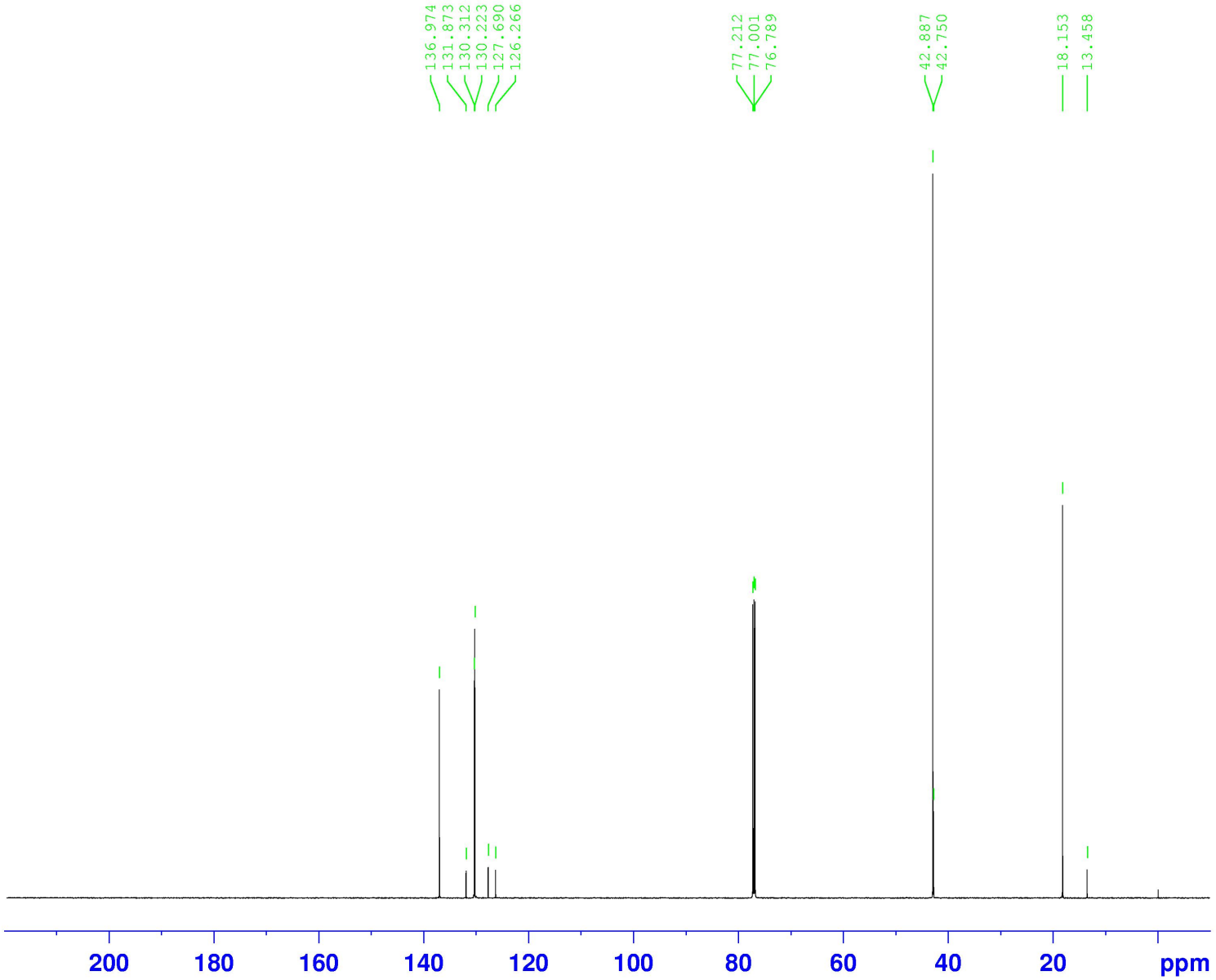



```
Current Data Parameters
NAME                  Hashimoto
EXPNO                       800
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20170712
Time                       0.50
INSTRUM                   spect
PROBHD    5 mm CPTCI 1H-
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  12335.526 Hz
FIDRES                0.376450 Hz
AQ                    1.3281963 sec
RG                          10
DW                     40.533 usec
DE                     10.00 usec
TE                       298.0 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               600.1337060 MHz
NUC1                        1H
P1                       7.40 usec
PLW1                8.80000019 W

F2 - Processing parameters
SI                       32768
SF                 600.1300054 MHz
WDW                         EM
SSB                0
LB                       0.30 Hz
GB                 0
PC                       1.00
```

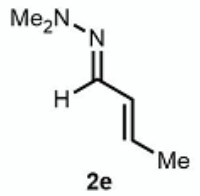


2e

20170711_ikeda_56_1
A.carbon CDCl3 D:\\ rfia 3




```
Current Data Parameters
NAME            Hashimoto
EXPNO                 801
PROCNO                  1

F2 - Acquisition Parameters
Date_            20170712
Time                 2.32
INSTRUM             spect
PROBHD    5 mm CPTCI 1H-
PULPROG           zgpg30
TD                 65356
SOLVENT            CDCl3
NS                  2048
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
```



2e