Electronic Supplementary Information for

Tandem catalysis in domino olefin cross-metathesis/intramolecular oxa-conjugate cyclization: Concise synthesis of 2,6-*cis*-substituted tetrahydropyran derivatives

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General remarks. All reactions sensitive to moisture and/or air were carried out under an atmosphere of argon in dry, freshly distilled solvents under anhydrous conditions using oven-dried glassware unless otherwise noted. Anhydrous dichloromethane (CH_2Cl_2) was purchased from Kanto Chemical Co. Inc. and used directly without further drying. Anhydrous tetrahydrofuran (THF), diethyl ether (Et_2O) and toluene were purchased from Wako Pure Chemical Industries, Ltd. and further purified by a Glass Contour solvent purification system under an atmosphere of argon immediately prior to use. Diisopropylamine, triethylamine, 2,6-lutidine, 1,2-dichloroethane and methanol were distilled from calcium hydride under an atmosphere of argon. All other chemicals were purchased at highest commercial grade and used directly. Microwave irradiation experiments were performed on a Biotage Initiator 2.5 system. Analytical thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F_{254} plates (0.25-mm thickness). Flash column chromatography was carried out using Kanto Chemical silica gel 60N (40–100 mesh, spherical, neutral) or Fuji Silysia silica gel BW-300 (200–400 mesh). Optical rotations were measured on a JASCO P-1020 digital polarimeter. IR spectra were measured on a JASCO FT/IR-4100 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Unity INOVA-500, INOVA-600, or JEOL JNM ECA-600 spectrometer. Chemical shift values of ^{1}H and ^{13}C NMR spectra are reported in ppm (δ) downfield from tetramethylsilane with reference to internal residual solvent [^{1}H NMR, C*H*Cl_3 (7.24), C_6*H*D_5 (7.15); ^{13}C NMR, *C*DCl_3 (77.0), C_6D_6 (128.0), *C*D_3CN (1.28)] unless otherwise noted. Coupling constants (*J*) are reported in Hertz (Hz). The following abbreviations were used to designate the multiplicities: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet; br = broad. Unless otherwise noted, diastereomer ratio (d.r.) was estimated by 500 or 600 MHz ^{1}H NMR analysis. ESI-TOF mass spectra were recorded on a Bruker microTOFfocus spectrometer.

Preparation of starting materials

Compounds **1**[1] and **2**–**4**[2] were prepared as described previously.

Experimental procedure and spectroscopic data for all compounds

General procedure for domino CM/IOCC reaction under "microwave" conditions (GP1)

To a solution of δ-hydroxy olefin in CH_2Cl_2 placed in a Biotage microwave vial were added α,β-unsaturated carbonyl compound (3–10 equiv) and **HG-II** (10 mol%). The vial was flushed with argon and then sealed. The reaction mixture was heated at 100 °C under microwave irradiation for 30 min. The reaction mixture was cooled to room temperature, directly loaded onto a silica gel column and eluted with an appropriate solvent to give the product tetrahydropyran.

General procedure for domino CM/IOCC reaction under "oil bath" conditions (GP2)

To a solution of δ-hydroxy olefin in toluene were added α,β-unsaturated carbonyl compound (3–10 equiv) and **HG-II** (10 mol%), and the resultant solution was heated at 80–110 °C for 11–24 h. The progress of the reaction was monitored by TLC analysis. The reaction mixture was cooled to room temperature and exposed to air with stirring for a while. The reaction mixture was then concentrated under reduced pressure and the residue purified by flash column chromatography on silica gel to give the product tetrahydropyran.

General procedure for domino CM/IOCC reaction by using HG-II and CSA (GP3)

To a solution of δ-hydroxy olefin in CH_2Cl_2 were added α,β-unsaturated carbonyl compound (3–10 equiv), **HG-II** (10 mol%) and CSA (3–10 mol %), and the resultant solution was stirred at 25–35 °C. After completion of the reaction (judged by TLC analysis), the reaction mixture was exposed to air with stirring for a while, directly loaded onto a silica gel column and eluted with an appropriate solvent to give the product tetrahydropyran.

2-{(2*R*,4*S*,2*R*)-6-[2-(*tert*-Butyldiphenylsilyloxy)ethyl]-4-(triisopropylsilyloxy)tetrahydropyran-

2-yl}acetaldehyde (8)

The *title* compound was synthesized according to **GP1** (19% yield, 2,6-*cis*/2,6-*trans* 5:1), **GP2** (73% yield, 2,6-*cis*/2,6-*trans* 2.4:1) or **GP3** (80% yield, 2,6-*cis*/2,6-*trans* >20:1): Colorless oil; $[\alpha]_D^{25}$ +4.1 (*c* 1.00 in $CHCl_3$); IR (film): ν_{max}/cm^{-1} 2942, 2864, 1728, 1463, 1428, 1112, 702; ^1H NMR (600 MHz; $CDCl_3$): δ_H 9.69 (1H, m), 7.65–7.60 (4H, m), 7.42–7.33 (6H, m), 3.87 (1H, m), 3.78–3.67 (3H, m), 3.54 (1H, m), 2.54 (1H, ddd, *J* = 16.0, 7.8, 2.3 Hz), 2.43 (1H, ddd, *J* = 16.0, 4.6, 2.3 Hz), 1.89 (2H, m), 1.76–1.64 (2H, m), 1.29–1.19 (2H, m), 1.05–1.01 (30H, m); ^{13}C NMR (150 MHz; $CDCl_3$): δ_C 201.3, 135.5 (4C), 133.9 (2C), 129.6 (2C), 127.6 (4C), 72.5, 70.8, 68.4, 60.1, 49.5, 41.7, 41.6, 38.8, 26.8 (3C), 19.2, 18.1 (6C), 12.3 (3C); HRMS (ESI): calcd for $C_{34}H_{54}O_4Si_2Na$ $(M + Na)^+$: 605.3453, found 605.3478.

***N*-(2-{(2*R*,4*S*,6*R*)-6-[2-(*tert*-Butyldiphenylsilyloxy)ethyl]-4-(triisopropylsilyloxy)tetrahydropyran-2-yl}acetyl) 2,5-dimethyl-1*H*-pyrrole (9)**

The *title* compound was synthesized according to **GP1** (25% yield, 2,6-*cis*/2,6-*trans* 3:1), **GP2** (64% yield, 2,6-*cis*/2,6-*trans* 10:1) or **GP3** (73%, 2,6-*cis*/2,6-*trans* >20:1): Yellow oil; $[\alpha]_D^{26}$ +11.7 (*c* 1.00 in $CHCl_3$); IR (film): ν_{max}/cm^{-1} 2928, 1716, 1684, 1541, 1457, 1112, 701, 538; ^1H NMR (600 MHz; $CDCl_3$): δ_H 7.63–7.60 (4H, m), 7.39–7.31 (6H, m), 5.78 (2H, s), 3.91–3.85 (2H, m), 3.75–3.67 (2H, m), 3.51 (1H, m), 3.01 (1H, dd, *J* = 16.1, 6.9 Hz), 2.75 (1H, dd, *J* = 16.1, 5.9 Hz), 2.32 (6H, s), 2.00 (1H, m), 1.88 (1H, m), 1.73 (1H, m), 1.65 (1H, m), 1.25–1.17 (2H, m), 1.06–0.99 (30H, m); ^{13}C NMR (150 MHz; $CDCl_3$): δ_C 172.3, 135.5 (4C), 134.0, 133.9, 130.2 (2C), 129.5 (2C), 127.6 (4C), 111.2 (2C), 72.7, 72.3, 68.4, 60.4, 45.3, 41.8, 41.6, 38.9, 30.9, 26.8 (3C), 19.2, 18.1 (6C), 16.4, 12.3 (3C); HRMS (ESI): calcd for $C_{40}H_{61}NO_4Si_2Na$ $(M + Na)^+$: 698.4031, found 698.4061. The spectroscopic data were in accordance with those previously reported.[2]

Methyl (5*R*,7*R*)-9-(*tert*-butyldiphenylsilyloxy)-7-hydroxy-5-(triisopropylsilyloxy)-2-nonenoate (10)

The *title* compound was synthesized according to **GP1** in 98% yield: Colorless oil; $[\alpha]_D^{18}$ −18.0 (*c* 1.00 in $CHCl_3$); IR (film): ν_{max}/cm^{-1} 3521, 2943, 2865, 1726, 1110, 701; ^1H NMR (500 MHz; $CDCl_3$): δ_H 7.66–7.63 (4H, m), 7.43–7.35 (6H, m), 6.94 (1H, ddd, *J* = 15.5, 7.0, 7.0 Hz), 5.85 (1H, d, *J* = 15.5 Hz), 4.26 (1H, m), 4.16 (1H, m), 3.85–3.76 (2H, m), 3.70 (3H, s), 3.52 (1H, br s), 2.55–2.47 (2H, m), 1.72–1.64 (2H, m), 1.61–1.49 (2H, m), 1.14–0.99 (30H, m, 30H); ^{13}C NMR (125 MHz; $CDCl_3$): δ_C 166.7, 145.1, 135.5 (2C), 135.5 (2C), 133.2, 133.1, 129.7 (2C), 127.7 (4C), 123.3, 69.5, 67.1, 62.6, 51.4, 43.3, 40.3, 39.5, 26.8 (3C), 19.0, 18.1 (3C), 18.1 (3C), 12.6 (3C); HRMS (ESI): calcd for $C_{35}H_{56}O_5Si_2Na$ $(M + Na)^+$: 635.3558, found 635.3566.

***rac*-2-{(2*R*,6*S*)-6-[2-(*tert*-Butyldiphenylsilyloxy)ethyl]tetrahydropyran-2-yl}acetaldehyde (11)**

The *title* compound was synthesized according to **GP2** (73% yield, 2,6-*cis*/2,6-*trans* 3:1) or **GP3** (73% yield, 2,6-*cis*/2,6-*trans* >20:1): Colorless oil; IR (film): ν_{max}/cm^{-1} 2932, 2857, 1727, 1428, 1111, 738, 703; ^1H NMR (600 MHz; C_6D_6): δ_H 9.47 (1H, dd, *J* = 3.0, 1.8 Hz), 7.81–7.77 (4H, m), 7.30–7.23 (6H, m), 3.87 (1H, ddd, *J* = 10.2, 8.4, 5.4 Hz), 3.75 (1H, ddd, *J* = 10.2, 6.6, 4.8 Hz), 3.44–3.37 (2H, m), 2.18 (1H, ddd, *J* = 16.2, 7.8, 3.0 Hz), 1.92 (1H, ddd, *J* = 16.2, 4.8, 1.8 Hz), 1.75–1.63 (2H, m), 1.46 (1H, m), 1.26–1.12 (12H, m), 0.98 (1H, dddd, *J* = 12.6, 12.6, 11.4, 4.2 Hz), 0.89 (1H, m); ^{13}C NMR (150 MHz; C_6D_6): δ_C 199.9, 135.96 (2C), 135.94 (2C), 134.31, 134.29, 130.0, 129.9, 128.1 (2C), 128.0 (2C), 74.4, 72.7, 60.6, 50.1, 39.7, 31.5, 31.4, 27.1 (3C), 23.6, 19.4; HRMS (ESI): calcd for $C_{25}H_{34}O_3SiNa$ $(M + Na)^+$: 433.2169, found 433.2186. The spectroscopic data were in accordance with those previously reported.[2]

1-{(2*S*,4*S*,4a*R*,8a*S*)-4-Hydroxyoctahydropyrano[3,2-*b*]pyran-2-yl}acealdehyde (12)

The *title* compound was synthesized according to **GP2** (60% yield, 2,6-*cis*/2,6-*trans* 6:1) or **GP3** (70% yield, 2,6-*cis*/2,6-*trans* >20:1): Colorless crystals; mp 101–102 °C; $[\alpha]_D^{24}$ +21.0 (*c* 1.00 in $CHCl_3$); IR (film): ν_{max}/cm^{-1} 3407, 2926, 2856, 1721, 1093, 1046; ^1H NMR (600 MHz; $CDCl_3$): δ_H 9.76 (1H, dd, *J* = 1.8, 1.8 Hz), 4.00 (1H, m), 3.93 (1H, m), 3.74 (1H, m), 3.37 (1H, m), 3.08 (1H,

ddd, J = 11.0, 9.2, 4.6 Hz), 2.84 (1H, dd, J = 9.2, 9.2 Hz), 2.66 (1H, ddd, J = 16.5, 7.8, 2.3 Hz), 2.50 (1H, ddd, J = 16.5, 4.6, 1.4 Hz), 2.07 (1H, ddd, J = 12.8, 5.0, 2.3 Hz), 2.02 (1H, m), 1.72–1.67 (3H, m), 1.51–1.38 (2H, m); ^{13}C NMR (150 MHz; $CDCl_3$): δ_C 200.3, 83.7, 75.0, 70.6, 70.1, 67.9, 49.0, 38.6, 29.0, 25.4; HRMS (ESI): calcd for $C_{10}H_{16}O_4Na$ $(M + Na)^+$: 223.0946, found 223.0955.

1-{(2*S*,4*R*,4a*R*,8a*S*)-4-Hydroxyoctahydropyrano[3,2-*b*]pyran-2-yl}acetaldehyde (13)

The *title* compound was synthesized according to **GP2** (54% yield, 2,6-*cis*/2,6-*trans* 15:1) or **GP3** (48% yield, 2,6-*cis*/2,6-*trans* 4:1). Data for the 15:1 mixture of diastereomers: Brown oil; $[\alpha]_D^{24}$ −11.2 (*c* 1.00 in $CHCl_3$); IR (film): ν_{max}/cm^{-1} 3435, 2923, 2869, 1723, 1095, 967; ^{1}H NMR (600 MHz; $CDCl_3$; major isomer): δ_H 9.75 (1H, dd, J = 2.8, 1.8 Hz), 4.36 (1H, m), 4.09 (1H, d, J = 3.2 Hz), 3.92 (1H, m), 3.61 (1H, ddd, J = 11.5, 9.6, 4.6 Hz), 3.44 (1H, m), 2.51 (1H, ddd, J = 16.1, 8.7, 3.2 Hz), 2.40 (1H, ddd, J = 16.1, 4.6, 1.9 Hz), 2.33 (1H, s), 2.01 (1H, ddd, J = 7.8, 4.6, 3.2 Hz), 1.92 (1H, ddd, J = 14.2, 3.2, 2.3 Hz), 1.73–1.57 (4H, m), 1.40 (1H, m); ^{13}C NMR (150 MHz; $CDCl_3$; major isomer): δ_C 201.0, 79.6, 70.8, 68.2, 67.3, 65.7, 48.9, 37.4, 29.3, 25.5; HRMS (ESI): calcd for $C_{10}H_{16}O_4Na$ $(M + Na)^+$: 223.0946, found 223.0948.

***rac*-*N*-(2-{(2*R*,6*S*)-6-[2-(*tert*-Butyldiphenylsilyloxy)ethyl]tetrahydropyran-2-yl}acetyl) 2,5-dimethyl-1*H*-pyrrole (14)**

The *title* compound was synthesized according to **GP2** (40% yield, 2,6-*cis*/2,6-*trans* 7:1) or **GP3** (72% yield, 2,6-*cis*/2,6-*trans* >20:1): Yellow oil; IR (film): ν_{max}/cm^{-1} 2930, 1715, 1540, 1364, 1111, 702; ^{1}H NMR (600 MHz; $CDCl_3$): δ_H 7.66–7.61 (4H, m), 7.41–7.31 (6H, m), 5.80–5.76 (2H, m), 3.89 (1H, m), 3.77–3.66 (2H, m), 3.52 (1H, m), 2.96 (1H, dd, J = 16.9, 6.2 Hz), 2.75 (1H, dd, J = 16.9, 6.5 Hz), 2.34–2.31 (6H, m), 1.81 (1H, m), 1.74–1.51 (5H, m), 1.24–1.11 (2H, m), 1.02 (9H, s); ^{13}C NMR (150 MHz; $CDCl_3$): δ_C 172.6, 135.5 (4C), 134.1, 134.0, 130.3 (2C), 129.5 (2C), 127.5 (4C), 111.3, 111.2, 74.9, 74.6, 60.4, 45.8, 39.4, 31.4 (2C), 26.9 (3C), 23.4, 19.2, 16.5 (2C); HRMS (ESI): calcd for $C_{31}H_{41}NO_3SiNa$ $(M + Na)^+$: 526.2748, found 526.2760. The spectroscopic data

were in accordance with those previously reported.[2]

***N*-{2-[(2*S*,4*S*,4a*R*,8a*S*)-4-Hydroxyoctahydropyrano[3,2-*b*]pyran-2-yl]acetyl}**

2,5-dimethyl-1*H*-pyrrole (15)

The *title* compound was synthesized according to **GP2** (71% yield, 2,6-*cis*/2,6-*trans* 9:1) or **GP3** (63% yield, 2,6-*cis*/2,6-*trans* >20:1): Brown oil; $[\alpha]_D^{25}$ −5.8 (*c* 1.00 in $CHCl_3$); IR (film): ν_{max}/cm^{-1} 3735, 1716, 1541, 1507, 1457, 1102, 960; ^1H NMR (600 MHz; $CDCl_3$): δ_H 5.80 (2H, s), 4.12 (1H, m), 3.92 (1H, m), 3.76 (1H, ddd, *J* = 11.0, 8.9, 5.2 Hz), 3.36 (1H, m), 3.14–3.07 (2H, m), 2.85–2.79 (2H, m), 2.36 (6H, s), 2.18 (1H, ddd, *J* = 12.7, 5.2, 2.0 Hz), 2.01 (1H, m), 1.71–1.66 (3H, m), 1.49–1.38 (2H, m); ^{13}C NMR (150 MHz; $CDCl_3$): δ_C 171.9, 130.3 (2C), 111.5 (2C), 83.8, 74.9, 72.3, 70.2, 67.9, 44.7, 38.5, 29.1, 25.4, 16.6 (2C); HRMS (ESI): calcd for $C_{16}H_{23}NO_4Na$ $(M + Na)^+$: 316.1519, found 316.1527. The spectroscopic data were in accordance with those previously reported.[2]

***N*-{2-[(2*S*,4*R*,4a*R*,8a*S*)-4-Hydroxyoctahydropyrano[3,2-*b*]pyran-2-yl]acetyl}**

2,5-dimethyl-1*H*-pyrrole (16)

The *title* compound was synthesized according to **GP2** (52% yield, 2,6-*cis*/2,6-*trans* >20:1) or **GP3** (56% yield, 2,6-*cis*/2,6-*trans* >20:1): Yellow oil; $[\alpha]_D^{25}$ −19.6 (*c* 1.00 in $CHCl_3$); IR (film) ν_{max}/cm^{-1} 3649, 1698, 1653, 1363, 1339, 669; ^1H NMR (600 MHz; $CDCl_3$): δ_H 5.78 (2H, s), 4.44 (1H, m), 4.10 (1H, dd, *J* = 5.8, 3.1 Hz), 3.92 (1H, m), 3.62 (1H, dddd, *J* = 9.6, 5.2, 4.4, 4.1 Hz), 3.44 (1H, ddd, *J* = 11.3, 11.3, 3.4 Hz), 3.03–2.98 (2H, m), 2.76 (1H, dd, *J* = 15.8, 5.2 Hz), 2.36 (6H, s), 2.05–1.97 (2H, m), 1.71–1.58 (3H, m), 1.37 (1H, m); ^{13}C NMR (150 MHz; $CDCl_3$): δ_C 172.1, 130.2 (2C), 111.2 (2C), 79.8, 70.8, 69.0, 68.2, 65.8, 44.7, 37.4, 29.4, 25.5, 16.4 (2C); HRMS (ESI): calcd for $C_{16}H_{23}NO_4Na$ $(M + Na)^+$: 316.1519, found 316.1525. The spectroscopic data were in accordance with those previously reported.[2]

1-{(2*R*,4*S*,6*R*)-6-[2-(*tert*-Butyldiphenylsilyloxy)ethyl]-4-(triisopropylsilyloxy)tetrahydropyran-

2-yl}propan-2-one (18), (6*R*,8*R*)-10-(*tert*-Butyldiphenylsilyloxy)-8-hydroxy-6-(triisopropyloxy)decan-2-one (19), and (6*R*)-10-(*tert*-Butyldiphenylsilyloxy)-6-(triisopropylsilyloxy)-3-decen-2,8-dione (20)

To a solution of α,β-unsaturated ketone **17** (34.9 mg, 0.0586 mmol) in THF (0.2 mL) was added a solution of $RuClH(CO)(PPh_3)_3$ (2.2 mg, 0.0023 mmol) in THF (0.8 mL), and the resultant solution was heated under reflux for 15 h. The reaction mixture was cooled to room temperature and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 3 to 10% EtOAc/hexanes, gradient elution) gave **18**[3] (15.8 mg, 45%, 2,6-*cis*/2,6-*trans* 13:1), **20** (3.4 mg, 10%), and a mixture of **19** and **17** (13.2 mg). The yields of **19** and **17** were estimated to be 16% and 22%, respectively, by 600 MHz ^{1}H NMR analysis of the purified mixture of **19** and **17**. A part of the mixture could be separated by flash column chromatography (silica gel, 3 to 10% EtOAc/hexanes, gradient elution) to obtain an analytically pure sample of **19** (3.3 mg). Data for **18**: Colorless oil; $[\alpha]_D^{16}$ +6.9 (*c* 1.00 in $CHCl_3$); IR (film): ν_{max}/cm^{-1} 2942, 2864, 1718, 1418, 1111, 701; ^{1}H NMR (500 MHz; C_6D_6): δ_H 7.79–7.76 (4H, m), 7.29–7.23 (6H, m), 3.89–3.77 (3H, m), 3.71 (1H, m), 3.50 (1H, m), 2.39 (1H, dd, *J* = 16.0, 7.0 Hz), 2.01 (1H, dd, *J* = 16.0, 5.5 Hz), 2.00–1.90 (2H, m), 1.80 (1H, m), 1.71 (1H, m), 1.70 (3H, s), 1.36 (1H, ddd, *J* = 12.0, 11.5, 11.0 Hz), 1.25 (1H, ddd, *J* = 12.0, 11.5, 11.0 Hz), 1.17 (9H, s), 1.14–1.00 (21H, m); ^{13}C NMR (125 MHz; C_6D_6): δ_C 204.7, 136.0 (2C), 135.9 (2C), 134.28, 134.25, 129.92, 129.91, 128.1 (2C), 128.0 (2C), 72.5, 72.0, 69.0, 60.8, 49.5, 42.4, 42.2, 39.4, 30.5, 27.1 (3C), 19.4, 18.3 (6C), 12.6 (3C); HRMS (ESI): calcd for $C_{35}H_{56}O_4Si_2Na$ $(M + Na)^+$: 619.3609, found 619.3588. The spectroscopic data were in accordance with those previously reported[3]; Data for **19**: Colorless oil; $[\alpha]_D^{27}$ −7.4 (*c* 0.25 in $CHCl_3$); IR (film): ν_{max}/cm^{-1} 2942, 2865, 1716, 1428, 1111, 702; ^{1}H NMR (600 MHz; C_6D_6): δ_H 7.81–7.74 (4H, m), 7.27–7.20 (6H, m), 4.35 (1H, m), 4.20 (1H, ddd, *J* = 10.6, 6.0, 4.6 Hz), 3.94 (1H, m), 3.86 (1H, m), 3.40 (1H, s), 1.97–1.93 (2H, m), 1.78–1.71 (2H, m), 1.67–1.51 (9H, m),

1.18–1.08 (30H, m); ^{13}C NMR (150 MHz; C_6D_6): δ_C 205.9, 136.0 (4C), 134.0, 133.9, 130.0 (2C), 128.1 (4C), 71.2, 66.6, 62.6, 43.2 (2C), 40.6, 36.9, 29.3, 27.0 (3C), 19.7, 19.3, 18.4 (6C), 13.0 (3C); HRMS (ESI): calcd for $C_{35}H_{58}O_4Si_2Na$ $(M + Na)^+$: 621.3771, found 621.3762. Data for **20**: Colorless oil; $[\alpha]_D^{25}$ −7.1 (*c* 0.20 in $CHCl_3$); IR (film): ν_{max}/cm^{-1} 3408, 2928, 2864, 1715, 1678, 1110, 702; ^{1}H NMR (600 MHz; $CDCl_3$): δ_H 7.65–7.60 (4H, m), 7.42–7.34 (6H, m), 6.81 (1H, ddd, *J* = 16.0, 7.8, 7.8 Hz), 6.10 (1H, d, *J* = 16.0 Hz), 4.50 (1H, m), 3.89 (2H, dd, *J* = 6.4, 5.9 Hz), 2.72–2.58 (4H, m), 2.50 (1H, m), 2.39 (1H, m), 2.20 (3H, s), 1.08–0.99 (30H, m); ^{13}C NMR (150 MHz; $CDCl_3$): δ_C 207.6, 198.3, 143.9, 135.5 (4C), 133.8, 133.3, 129.7 (2C), 127.7 (5C), 67.3, 59.3, 50.5, 46.7, 40.4, 26.7 (3C), 19.1, 18.1 (6C), 14.1, 12.4 (3C); HRMS (ESI): calcd for $C_{35}H_{54}O_4Si_2Na$ $(M + Na)^+$: 617.3458, found 617.3491.

(6*R*)-10-(*tert*-Butyldiphenylsilyloxy)-6-(triisopropylsilyloxy)-decan-2,8-dione (21)

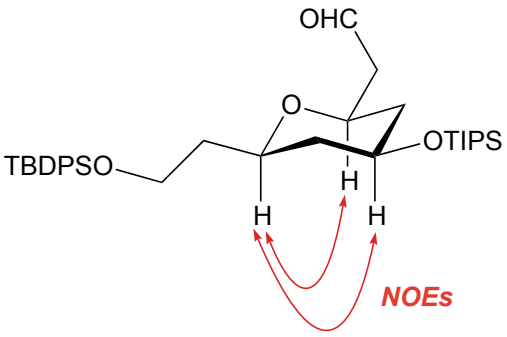
To a solution of α,β-unsaturated ketone **17** (48.2 mg, 0.0809 mmol) in THF (0.2 mL) was added a solution of $RuH_2(CO)(PPh_3)_3$ (3.0 mg, 0.0033 mmol) in THF (0.8 mL), and the resultant solution was heated under reflux for 13 h. The reaction mixture was cooled to room temperature and concentrated under reduced pressure. Purification of the residue by flash column chromatography (silica gel, 3 to 10% EtOAc/hexanes, gradient elution) gave **18** (26.6 mg, 55%, 2,6-*cis*/2,6-*trans* 15:1), a mixture of **19** and **17** (9.6 mg) and an inseparable mixture of **20** and **21** (11.9 mg). The yields of **19** and **17** were estimated to be 17% and 3%, respectively, by 600 MHz ^{1}H NMR analysis of the purified mixture of **19** and **17**. The yields of **20** and **21** were estimated to be 7% and 18%, respectively, by 600 MHz ^{1}H NMR analysis of the purified mixture of **20** and **21**. The structure of **21** was deduced from the ^{1}H NMR (see page S36) and HRMS spectra of the mixture: HRMS (ESI): calcd for $C_{35}H_{56}O_4Si_2Na$ $(M + Na)^+$: 619.3615, found 619.3642.

Stereochemical assignment of the product tetrahydropyrans

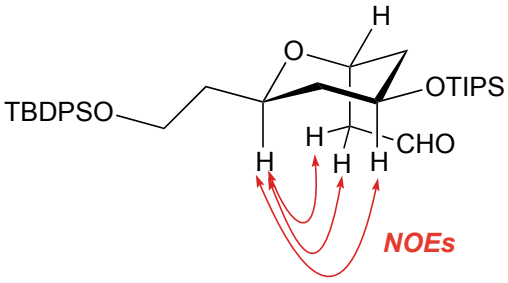
Stereochemical assignment of compounds **9**, **11**, and **14–16** has been reported elsewhere.[2]

Compounds **8**, **12**, and **13**:

Stereochemical assignment of compounds **8**, **12**, and **13** was made by NOE experiments.



8 (major diastereomer)



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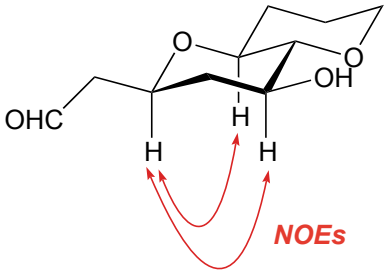
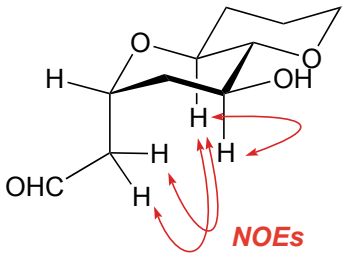
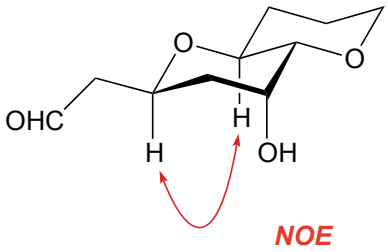


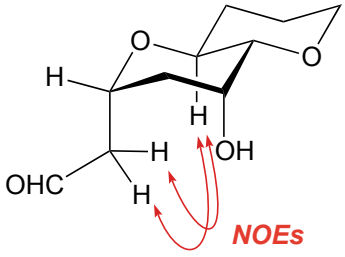
12 (major diastereomer)



12 (minor diastereomer)



13 (major diastereomer)



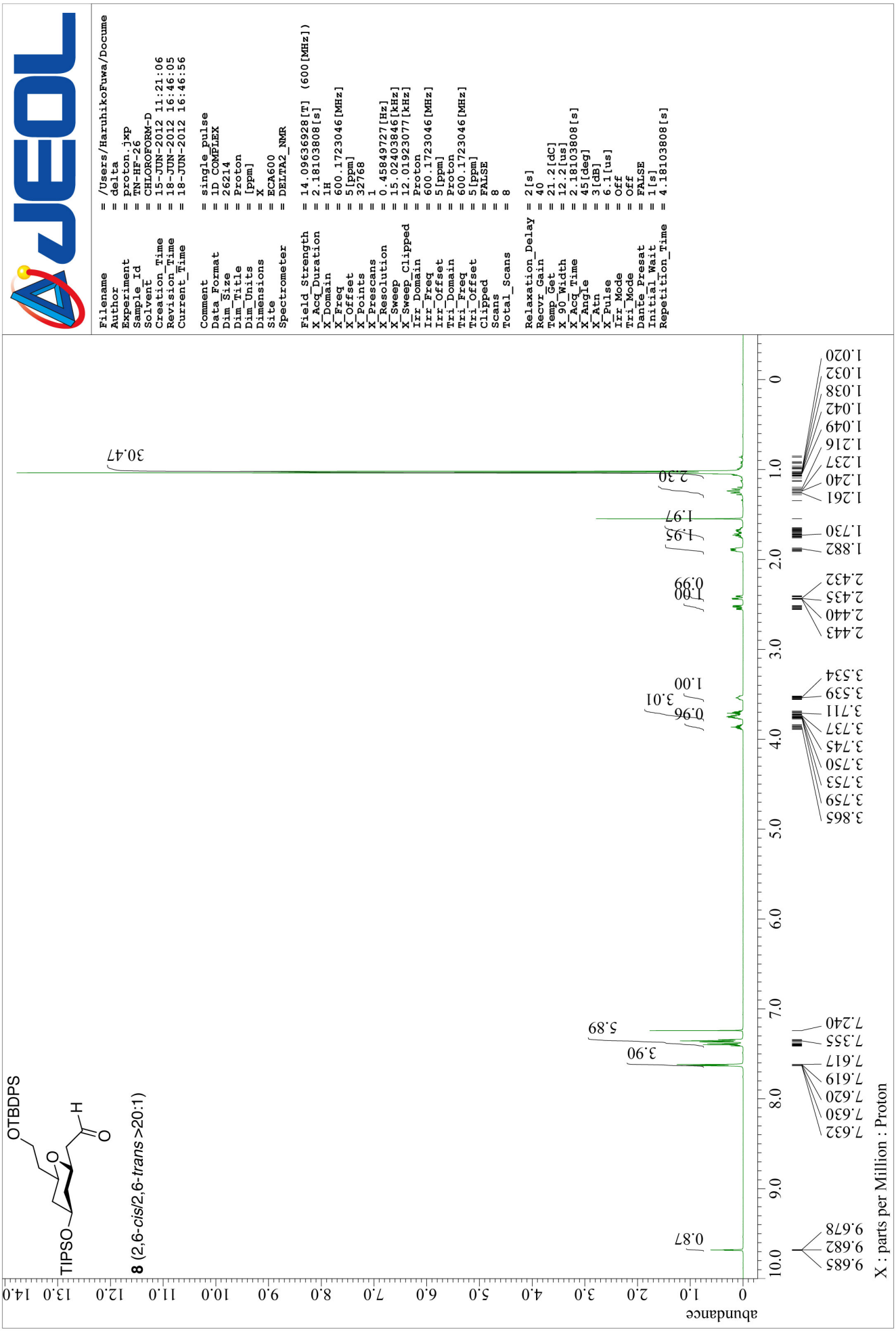
13 (minor diastereomer)

References

1. (*a*) H. Fuwa and M. Sasaki, *Org. Lett.*, 2010, **12**, 584; (*b*) H. Fuwa, T. Suzuki, H. Kubo, T. Yamori and M. Sasaki, *Chem. Eur. J.*, 2011, **17**, 2678.

2. H. Fuwa, N. Ichinokawa, K. Noto and M. Sasaki, *J. Org. Chem.*, 2012, **77**, 2588.

3. H. Fuwa, K. Noto and M. Sasaki, *Org. Lett.*, 2010, **12**, 1636.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X_Prescans = 1
X_Resolution = 0.45849727[Hz]
X_Sweep = 15.02403846[kHz]
X_Sweep_Clipped = 12.01923077[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 600.1723046[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 2[s]
Recvr_Gain = 40
Temp_Get = 21.2[dC]
X_90_Width = 12.2[us]
X_Acq_Time = 2.18103808[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
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Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 4.18103808[s]
OTBDPS
TIPSO
O
H
O
8 (2,6-cis/2,6-trans >20:1)
abundance
X : parts per Million : Proton
30.47
2.30
1.97
1.95
1.00
0.99
3.01
1.00
0.96
5.89
3.90
0.87
9.685
9.682
9.678
7.632
7.630
7.620
7.619
7.617
7.355
7.240
3.865
3.759
3.753
3.750
3.745
3.737
3.711
3.539
3.534
2.443
2.440
2.435
2.432
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1.216
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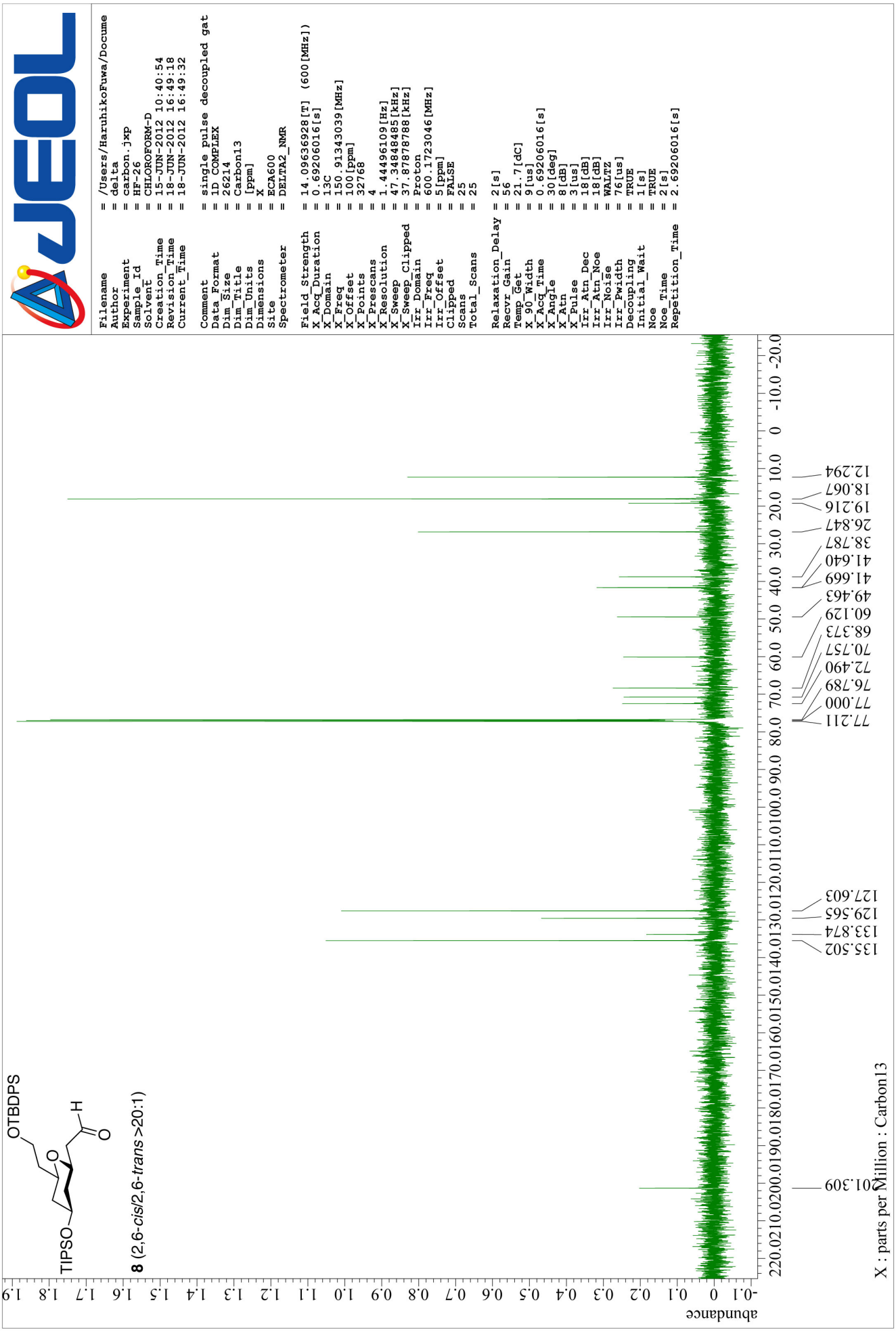
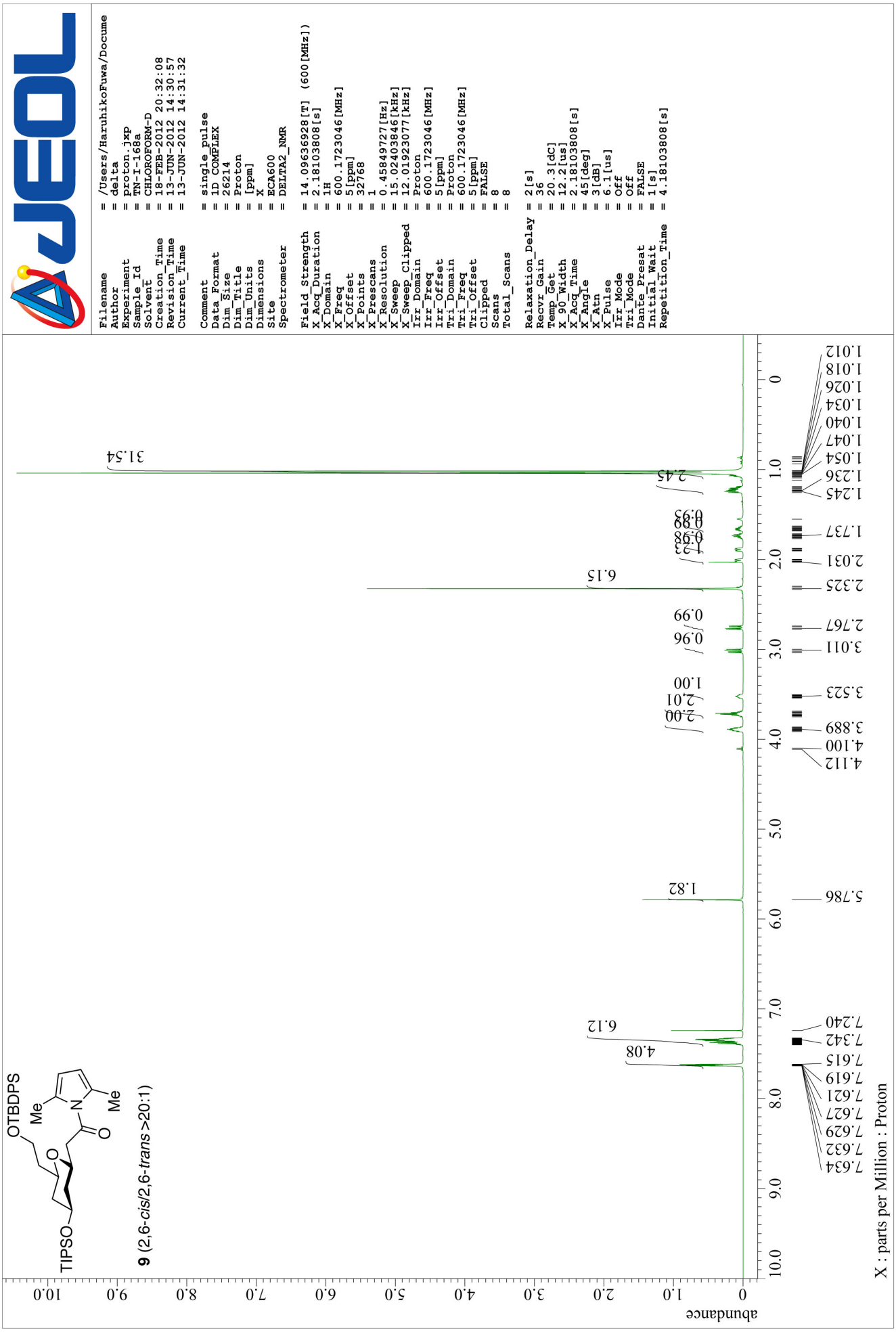
1.042
1.038
1.032
1.020

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Site = ECA600
Spectrometer = DELTA2_NMR
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X_Acq_Duration = 2.18103808[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
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X_Prescans = 1
X_Resolution = 0.45849727[Hz]
X_Sweep = 15.02403846[kHz]
X_Sweep_Clipped = 12.01923077[kHz]
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Tri_Offset = 5[ppm]
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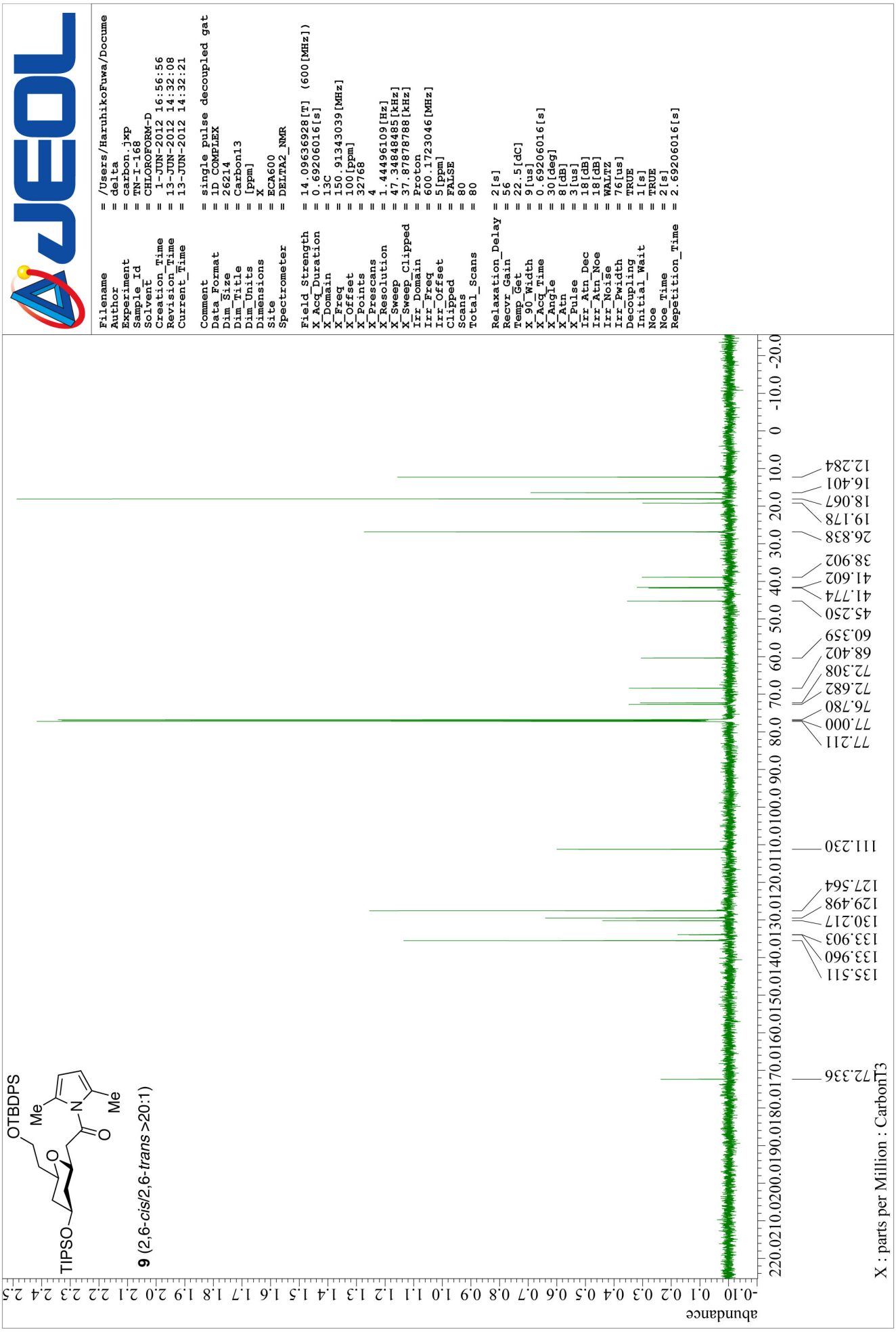
X_90_Width = 12.2[us]
X_Acq_Time = 2.18103808[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.1[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 4.18103808[s]
OTBDPS
Me
N
Me
O
O
TIPSO
9 (2,6-cis/2,6-trans >20:1)
abundance
X : parts per Million : Proton
31.54
6.15
6.12
4.08
1.82
2.45
2.00
2.01
1.00
0.96
0.99
1.23
0.98
0.99
0.95
5.786
4.112
4.100
3.889
3.523
3.011
2.767
2.325
2.031
1.737
1.245
1.236
1.054
1.047
1.040
1.034
1.026
1.018
1.012
7.634
7.632
7.629
7.627
7.621
7.619
7.615
7.342
7.240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X_Acq_Duration = 0.69206016[s]
X_Domain = 13C
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 80
Total_Scans = 80
Relaxation_Delay = 2[s]
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Temp_Get = 22.5[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 8[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 18[dB]
Irr_Atn_Noe = 18[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69206016[s]
OTBDPS
Me
N
Me
O
O
TIPSO
9 (2,6-cis/2,6-trans >20:1)
abundance
X : parts per Million : Carbon13
72.336
135.511
133.960
133.903
130.217
129.498
127.564
111.230
77.211
77.000
76.780
72.682
72.308
68.402
60.359
45.250
41.774
41.602
38.902
26.838
19.178
18.067
16.401
12.284

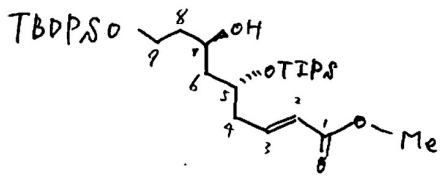
KN-I-071

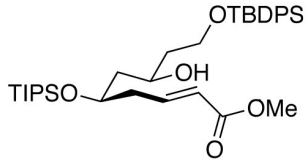
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 20.0 C / 293.1 K
INOVA-500 "varian"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8000.0 Hz
20 repetitions
OBSERVE H1, 499.8593223 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 5 sec





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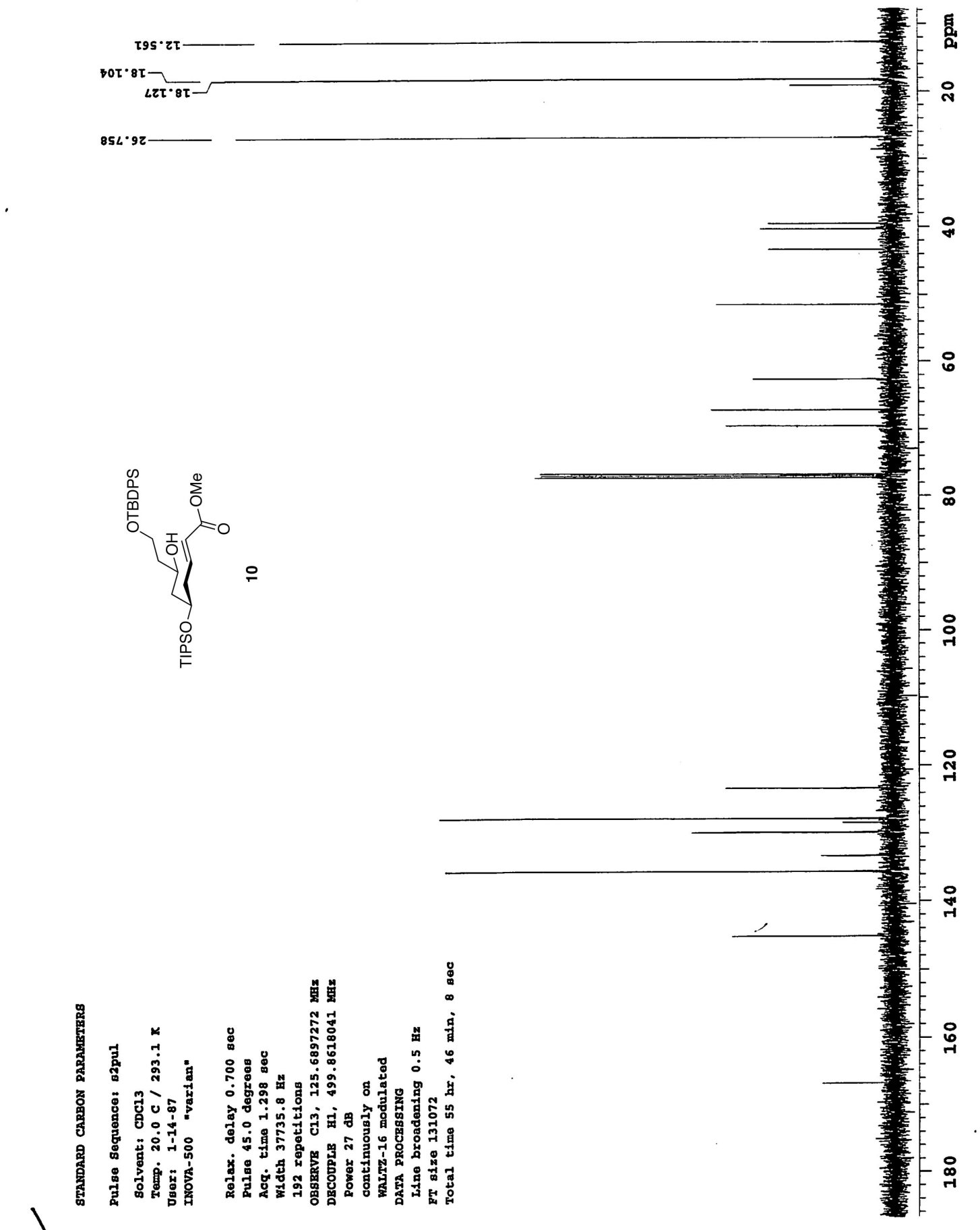

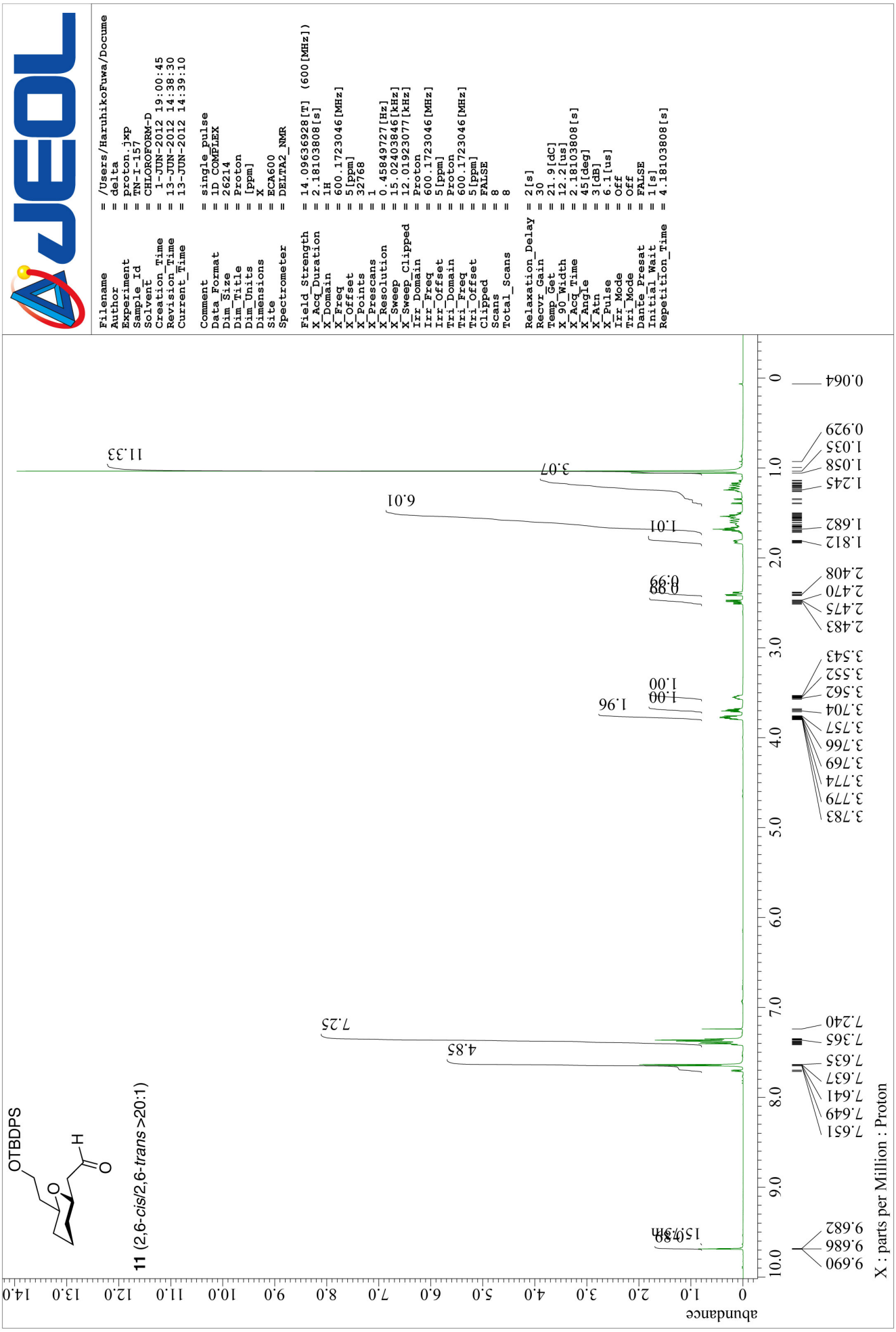

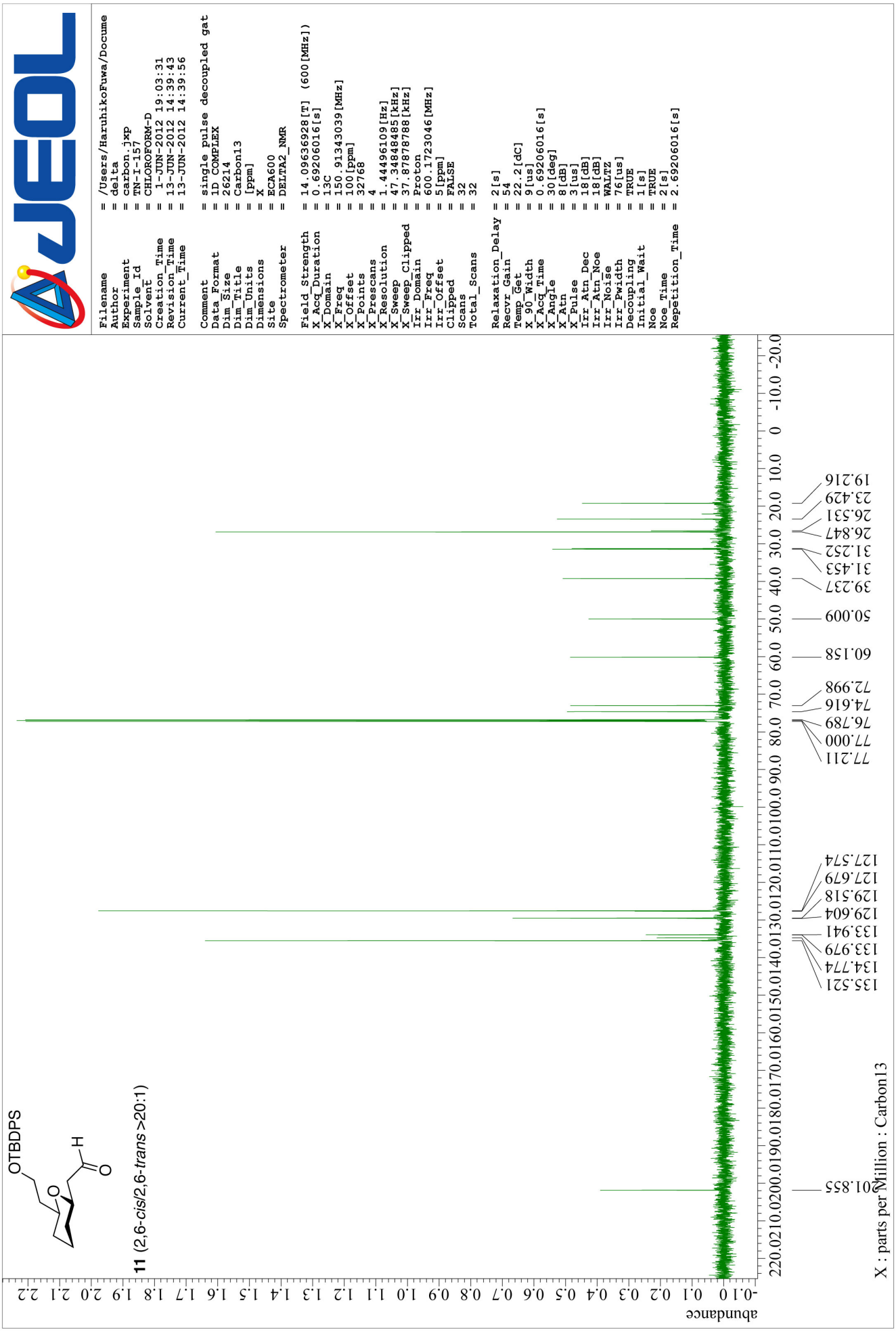
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X_Domain = 13C
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
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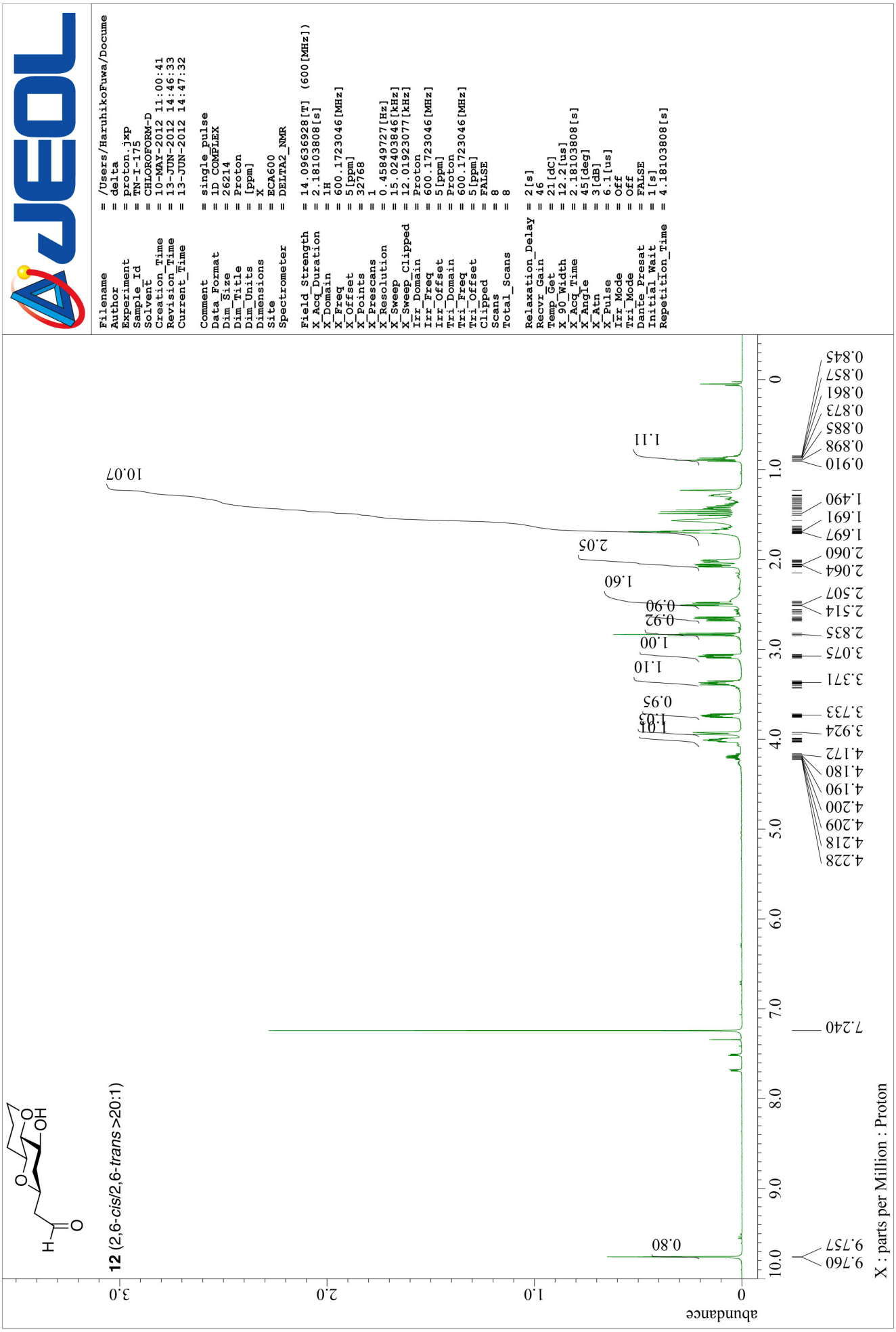
Recvr_Gain = 54
Temp_Get = 22.2[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 8[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 18[dB]
Irr_Atn_Noe = 18[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe_Time = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69206016[s]
OTBDPS
O
H
O
11 (2,6-cis/2,6-trans >20:1)
abundance
X : parts per Million : Carbon13
201.855
135.521
134.774
133.979
133.941
129.604
129.518
127.679
127.574
77.211
77.000
76.789
74.616
72.998
60.158
50.009
39.237
31.453
31.252
26.847
26.531
23.429
19.216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X_Resolution = 0.45849727[Hz]
X_Sweep = 15.02403846[kHz]
X_Sweep_Clipped = 12.01923077[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 600.1723046[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 2[s]
Recvr_Gain = 46
Temp_Get = 21[dC]
X_90_Width = 12.2[us]
X_Acq_Time = 2.18103808[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.1[us]
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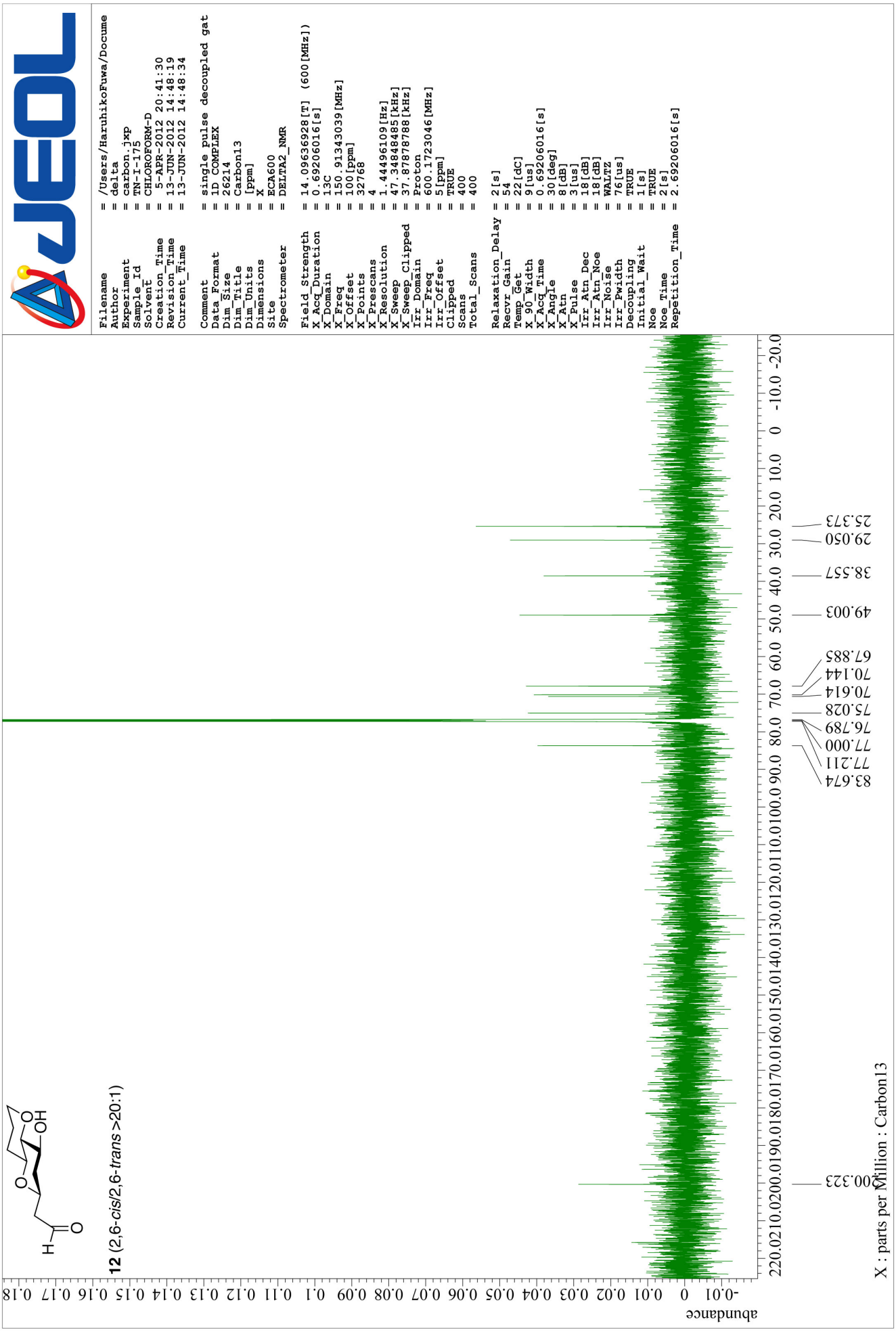
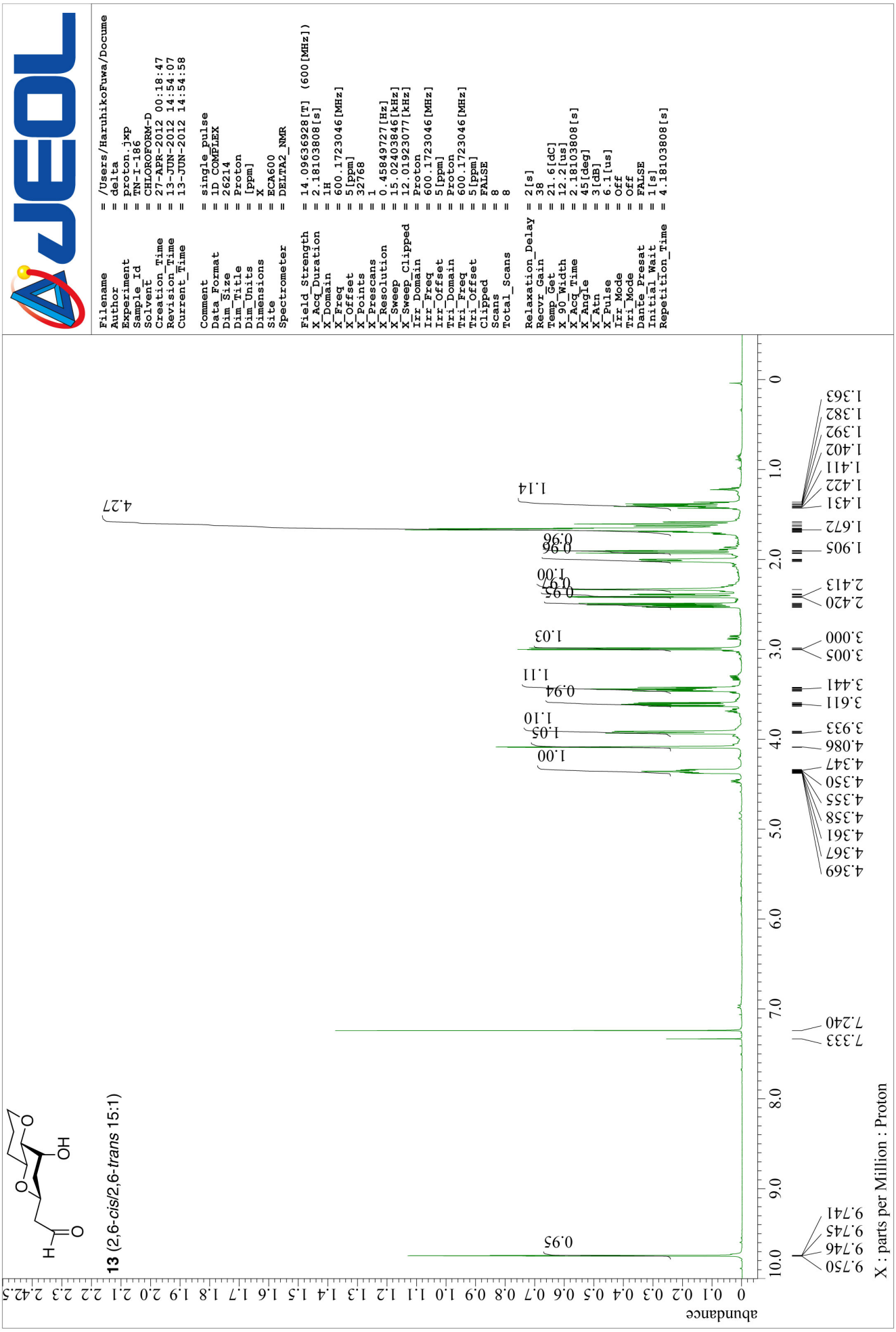
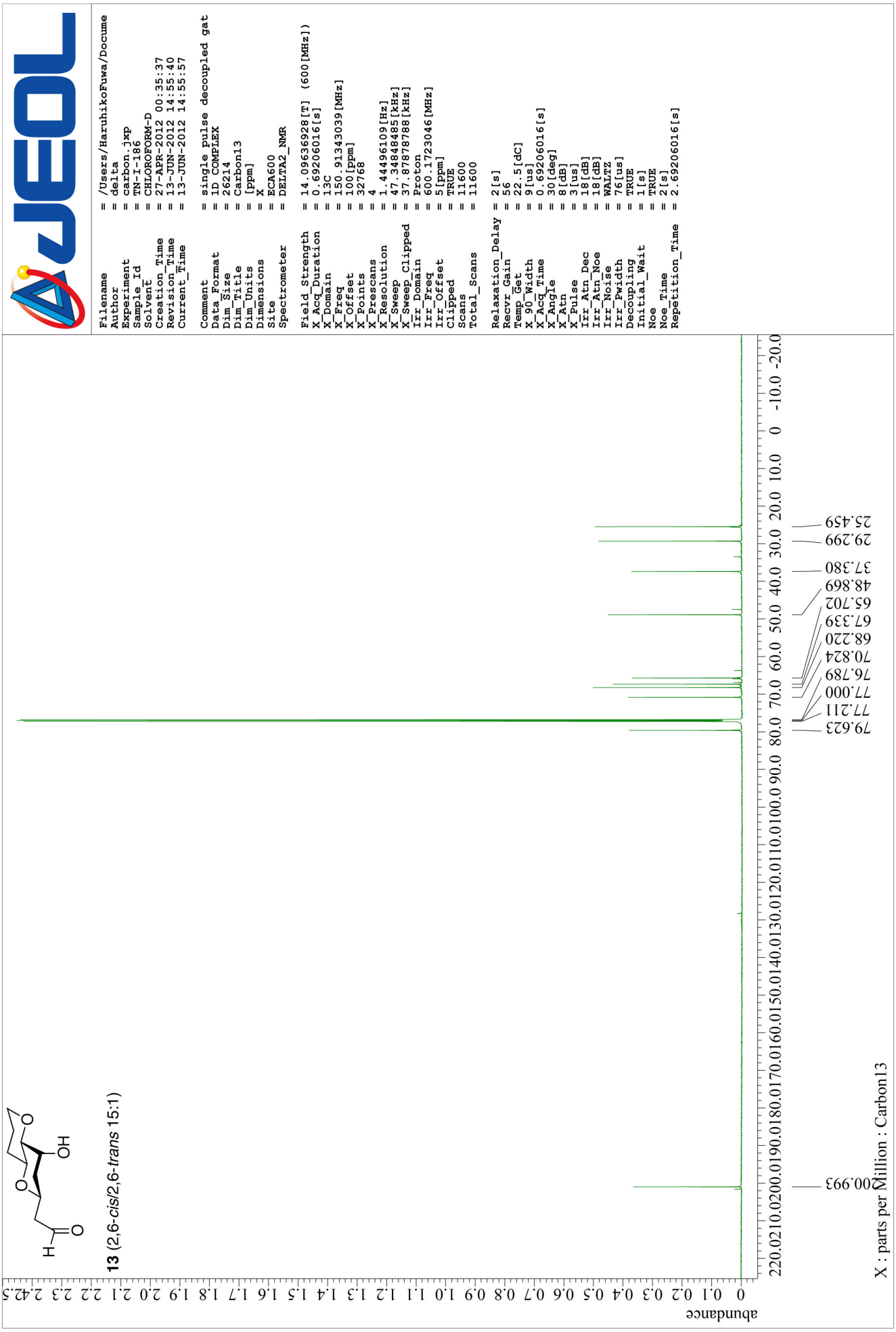
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 4.18103808[s]
10.07
2.05
1.60
1.11
1.00
0.92
0.90
1.10
0.95
1.05
1.03
0.80
7.240
9.760
9.757
4.228
4.218
4.209
4.200
4.190
4.180
4.172
3.924
3.733
3.371
3.075
2.835
2.514
2.507
2.064
2.060
1.697
1.691
1.490
0.910
0.898
0.885
0.873
0.861
0.857
0.845
abundance
X : parts per Million : Proton
12 (2,6-cis/2,6-trans >20:1)



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Dimensions = X
Site = ECA600
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X_Domain = 13C
X_Freq = 150.91343039[MHz]
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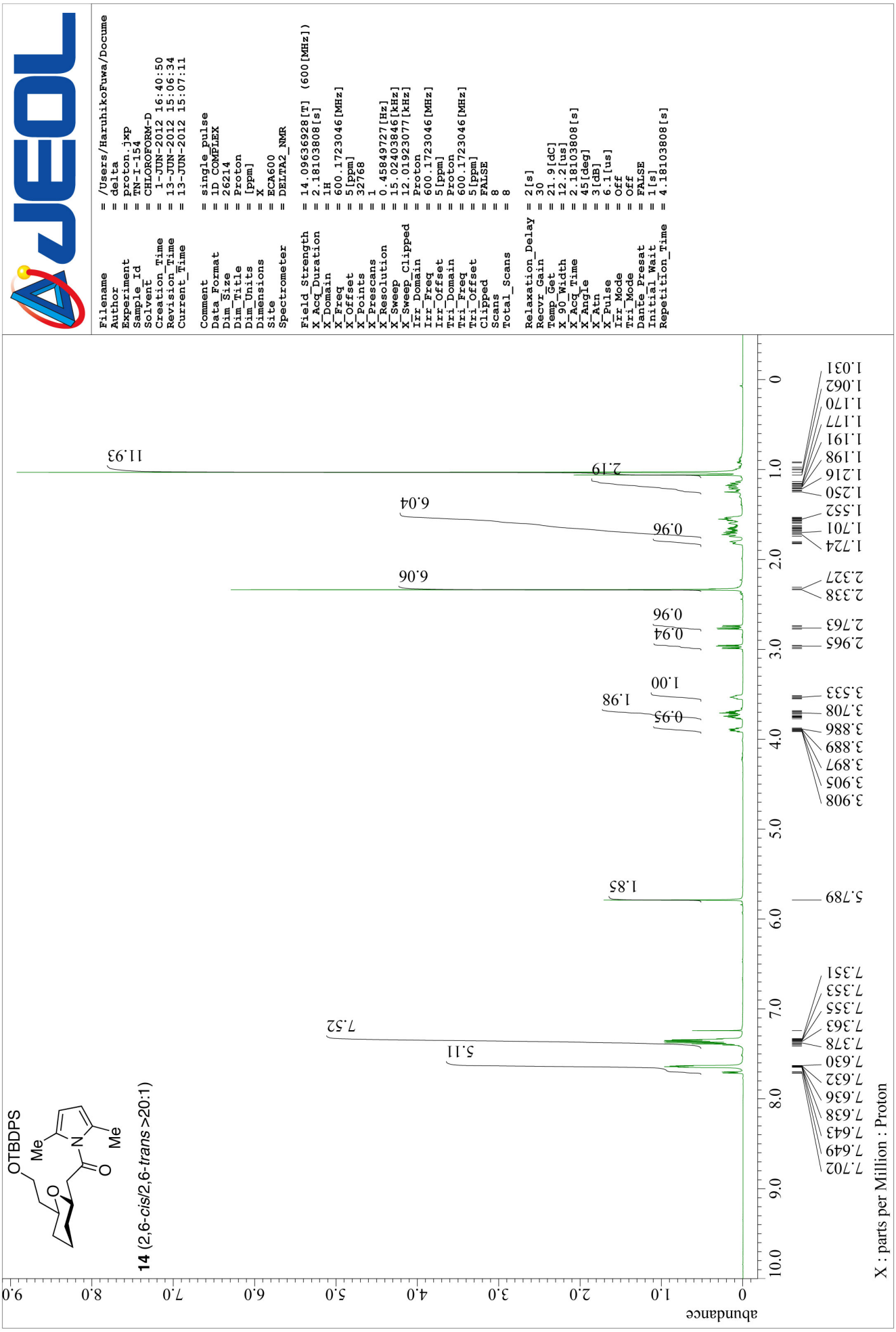
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 11600
Total_Scans = 11600
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 22.5[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 8[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 18[dB]
Irr_Atn_Noe = 18[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe_Time = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69206016[s]
13 (2,6-cis/2,6-trans 15:1)
abundance
X : parts per Million : Carbon13
200.993
79.623
77.211
77.000
76.789
70.824
68.220
67.339
65.702
48.869
37.380
29.299
25.459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Dim_Size = 26214
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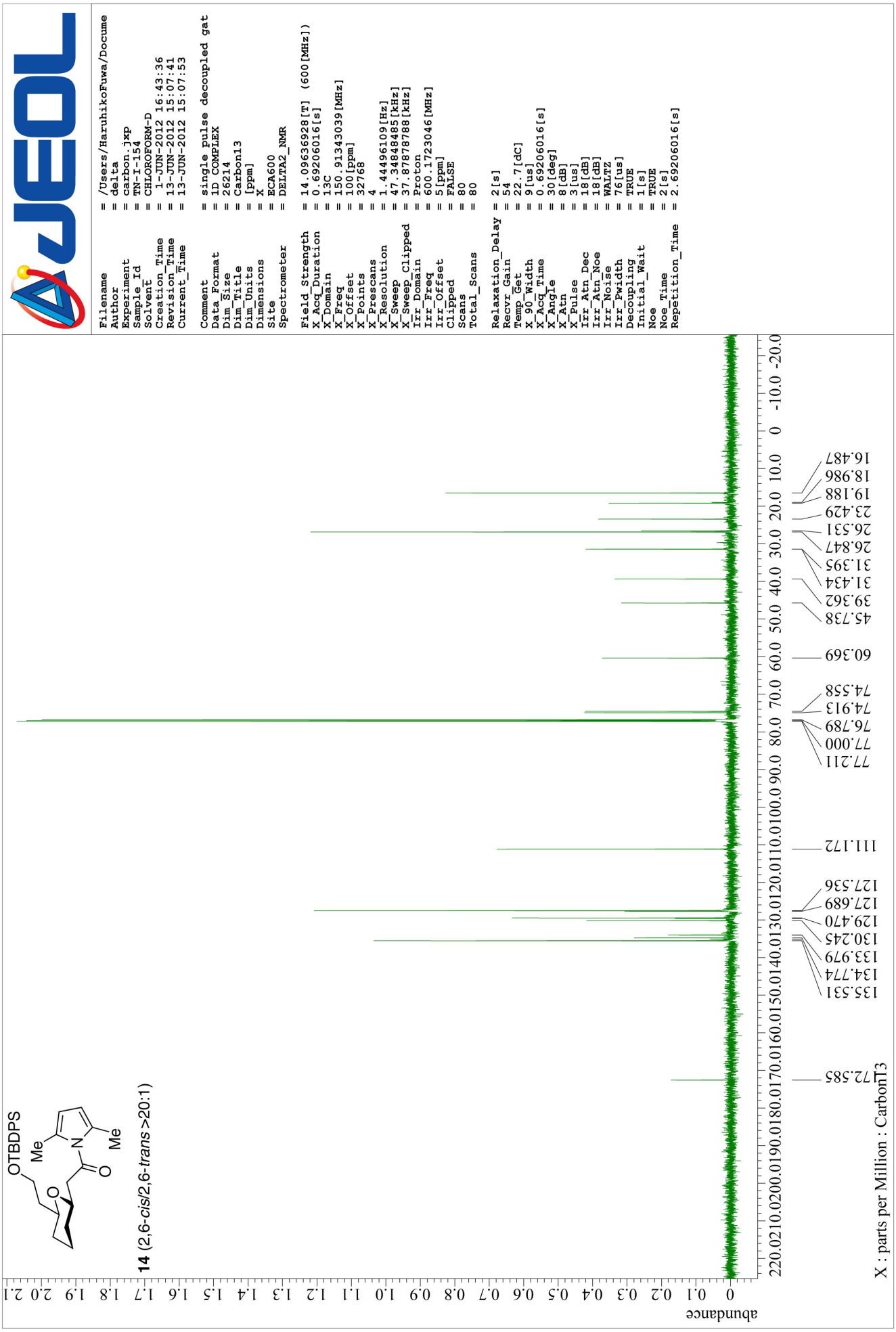
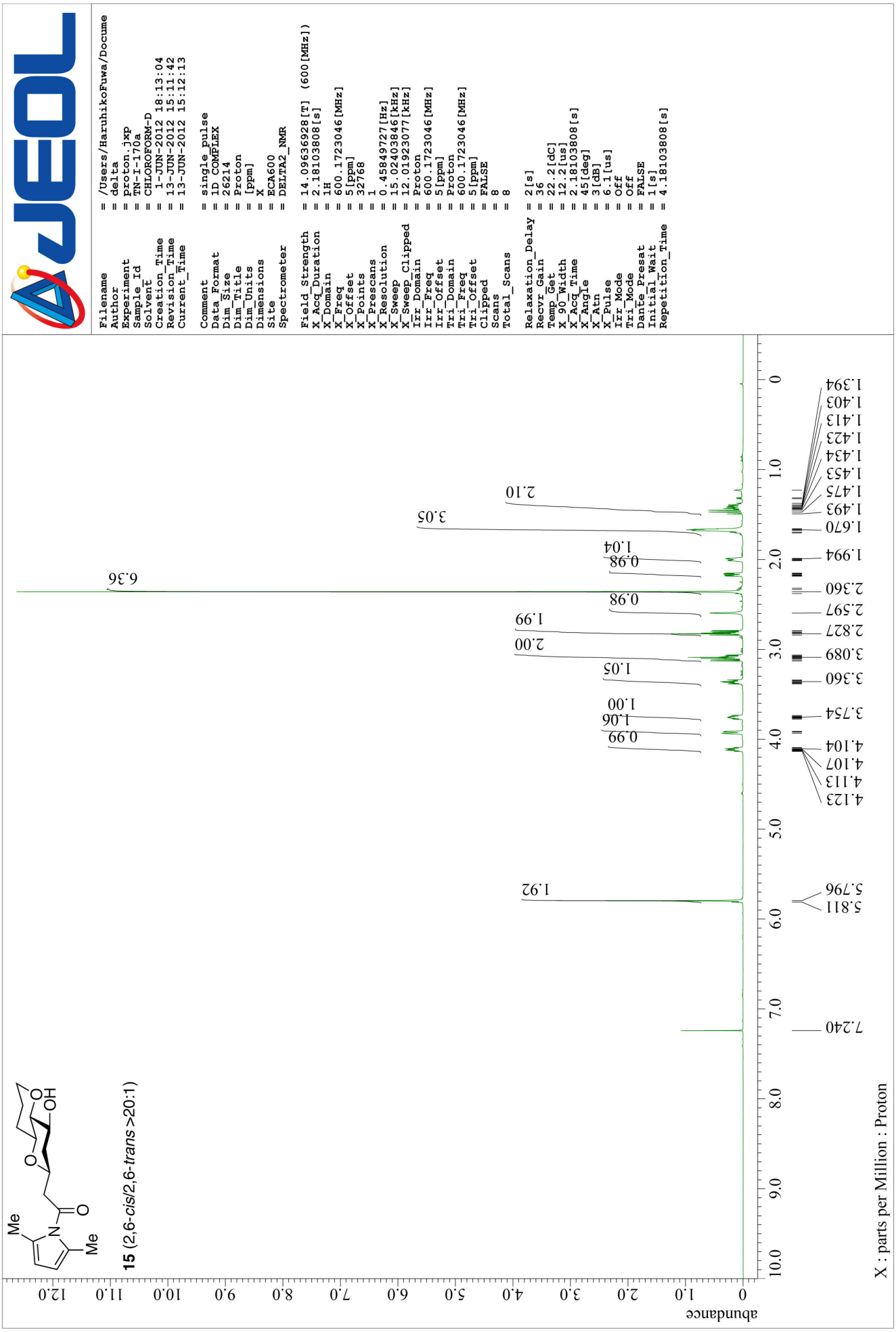
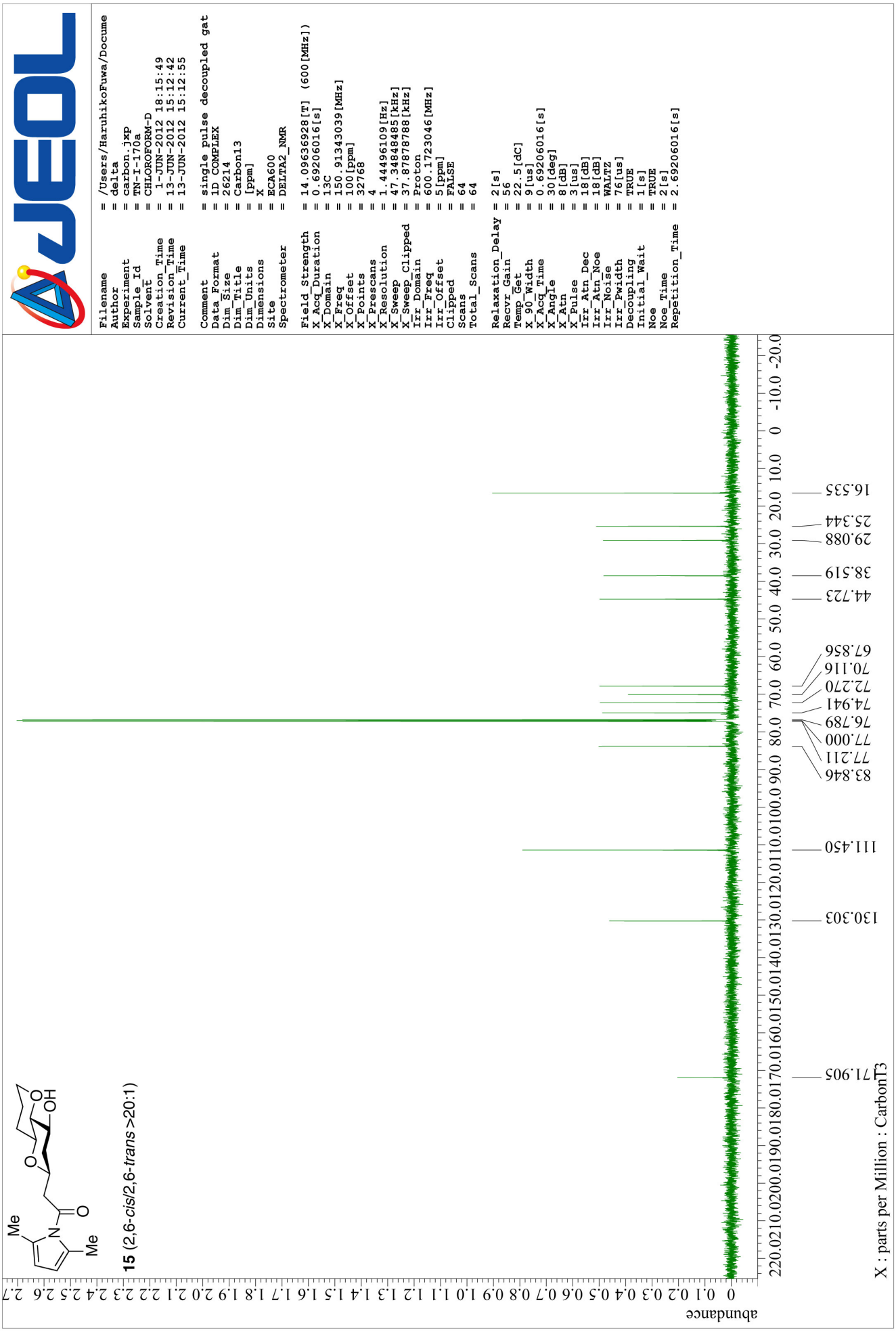
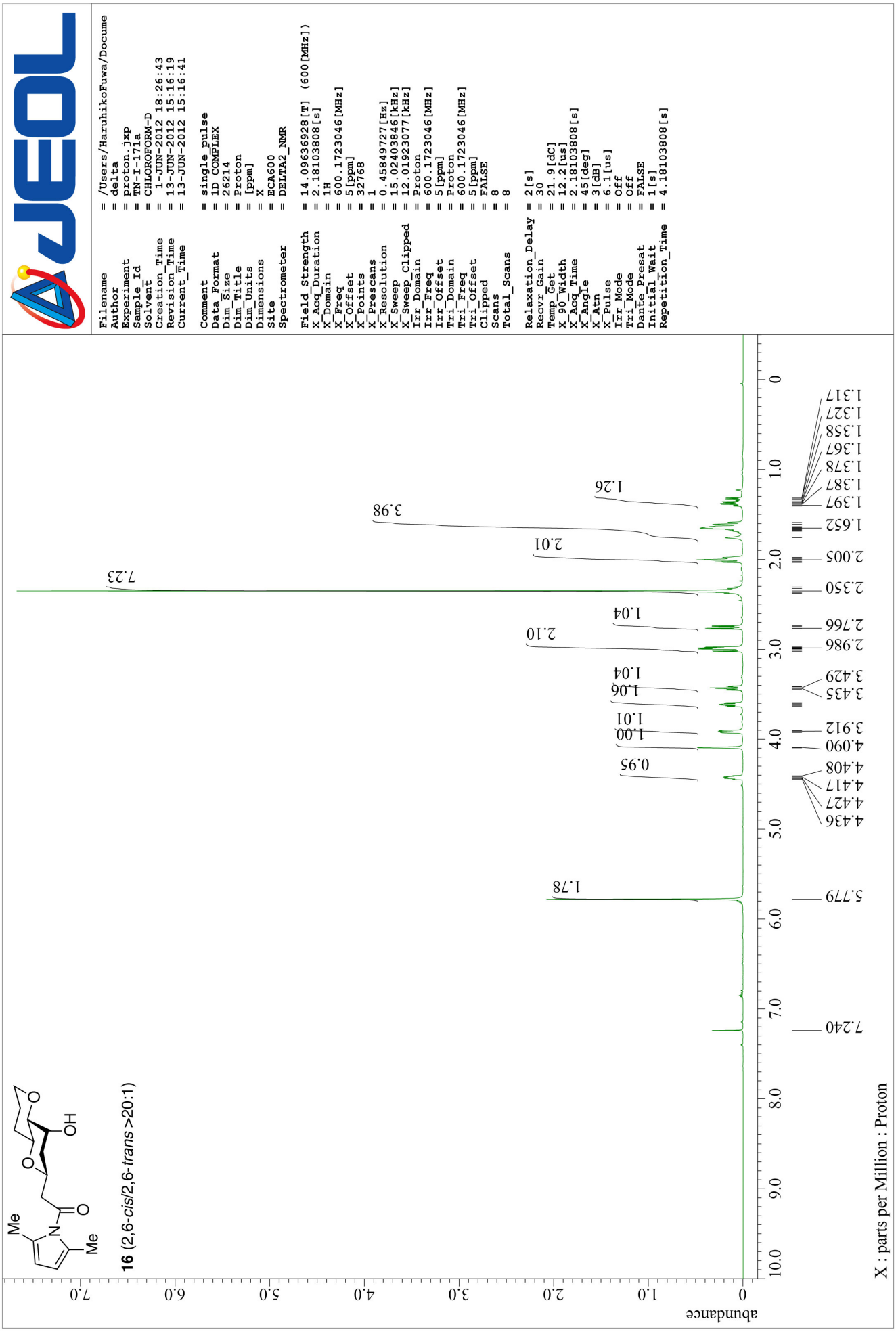
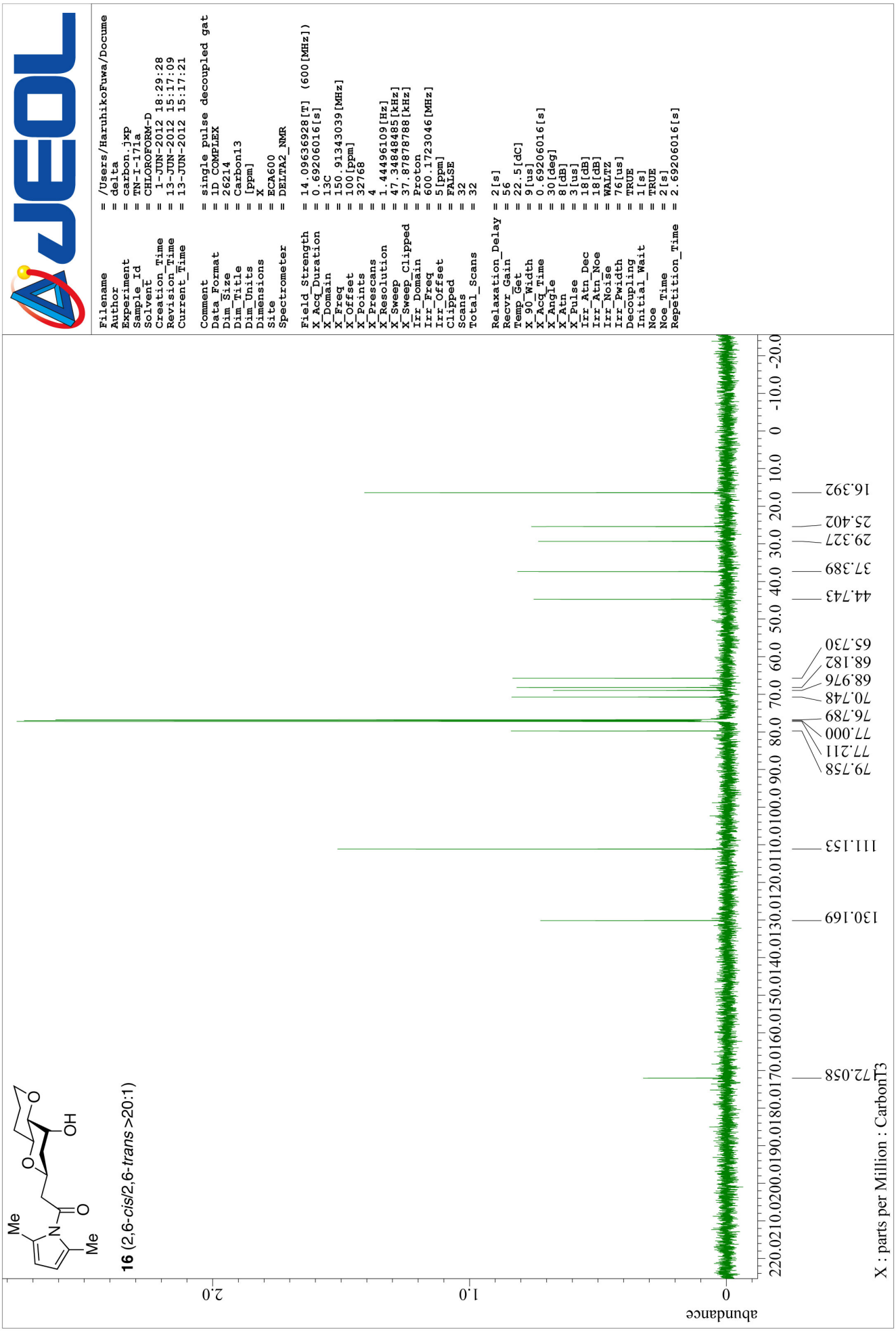
Dim_Units = [ppm]
Dimensions = X
Site = ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 2.18103808[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.45849727[Hz]
X_Sweep = 15.02403846[kHz]
X_Sweep_Clipped = 12.01923077[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 600.1723046[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 2[s]
Recvr_Gain = 30
Temp_Get = 21.9[dC]
X_90_Width = 12.2[us]
X_Acq_Time = 2.18103808[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.1[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 4.18103808[s]
OTBDPS
Me
Me
N
O
O
14 (2,6-cis/2,6-trans >20:1)
11.93
2.19
6.04
0.96
6.06
0.94
0.96
1.00
1.98
0.95
1.85
5.789
5.11
7.52
3.908
3.905
3.897
3.889
3.886
3.708
3.533
2.965
2.763
2.338
2.327
1.724
1.701
1.552
1.250
1.216
1.198
1.191
1.177
1.170
1.062
1.031
7.702
7.649
7.643
7.638
7.636
7.632
7.630
7.378
7.363
7.355
7.353
7.351
abundance
X : parts per Million : Proton

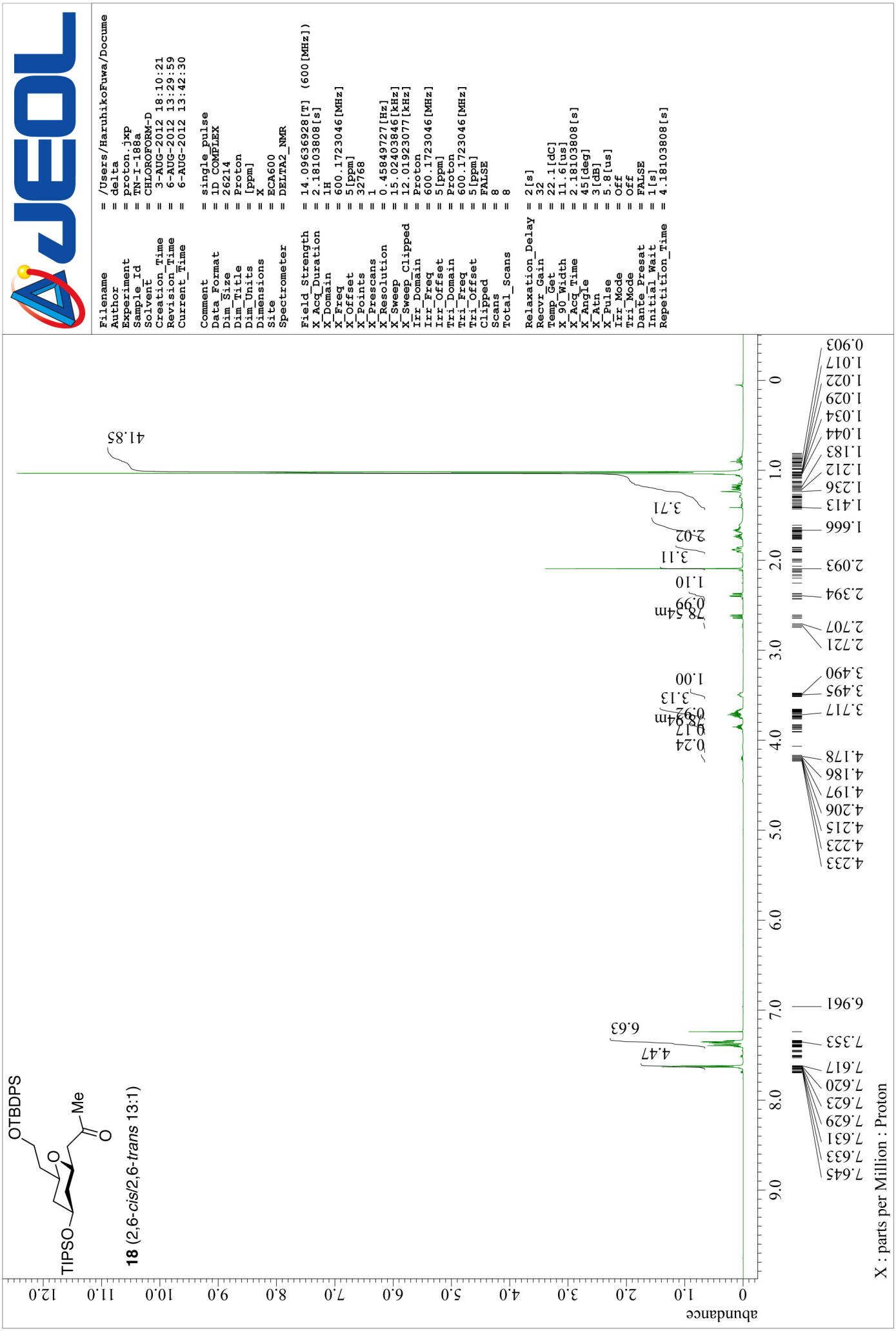

JEOL
Filename = /Users/HaruhikoFuwa/Docume
Author = delta
Experiment = carbon.jxp
Sample_Id = TN-I-154
Solvent = CHLOROFORM-D
Creation_Time = 1-JUN-2012 16:43:36
Revision_Time = 13-JUN-2012 15:07:41
Current_Time = 13-JUN-2012 15:07:53
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 0.69206016[s]
X_Domain = 13C
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 80
Total_Scans = 80
Relaxation_Delay = 2[s]
Recvr_Gain = 54
Temp_Get = 22.7[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 8[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 18[dB]
Irr_Atn_Noe = 18[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe_Time = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69206016[s]
OTBDPS
Me
Me
N
O
O
14 (2,6-cis/2,6-trans >20:1)
abundance
X : parts per Million : Carbon13
172.585
135.531
134.774
133.979
130.245
129.470
127.689
127.536
111.172
77.211
77.000
76.789
74.913
74.558
60.369
45.738
39.362
31.434
31.395
26.847
26.531
23.429
19.188
18.986
16.487

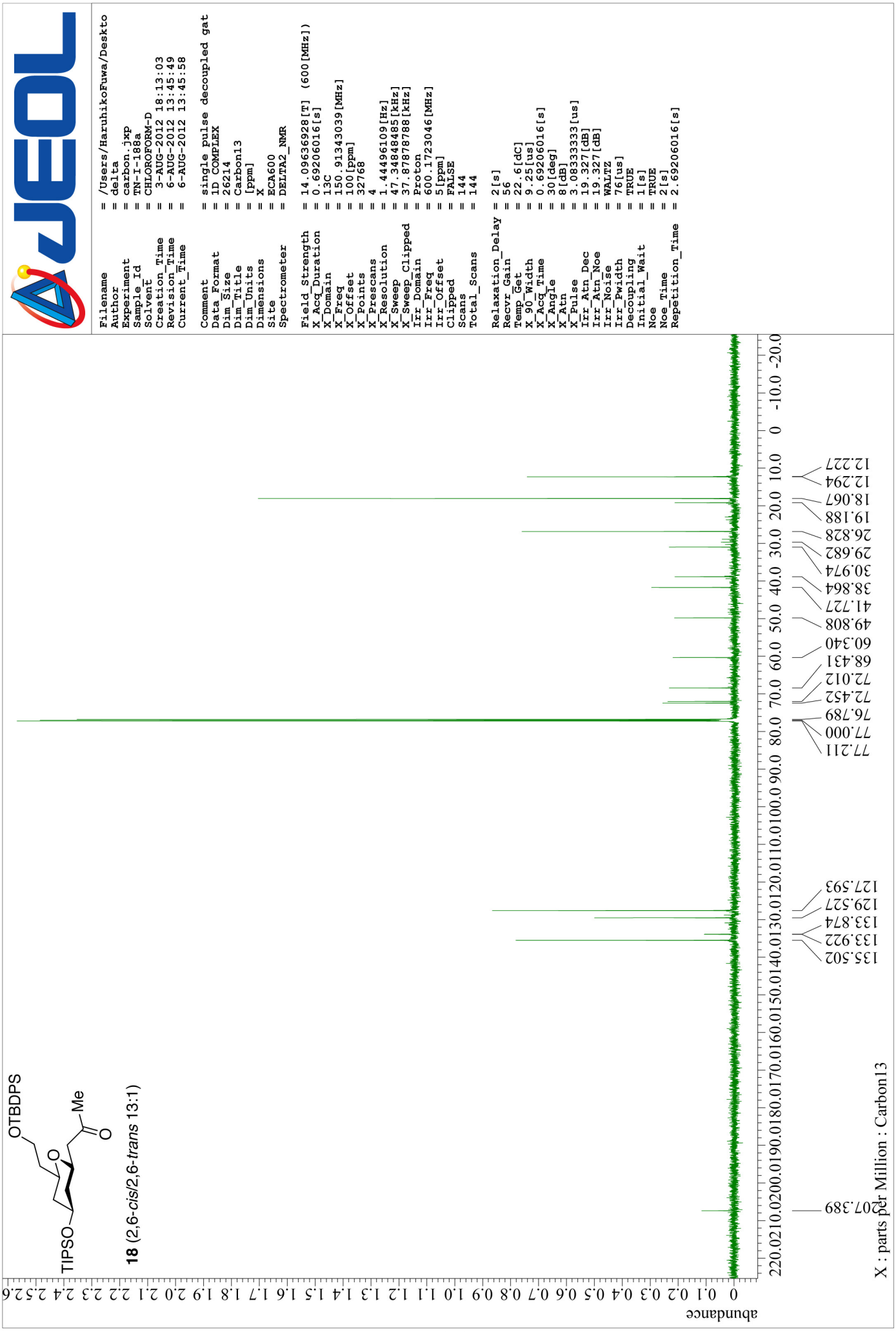


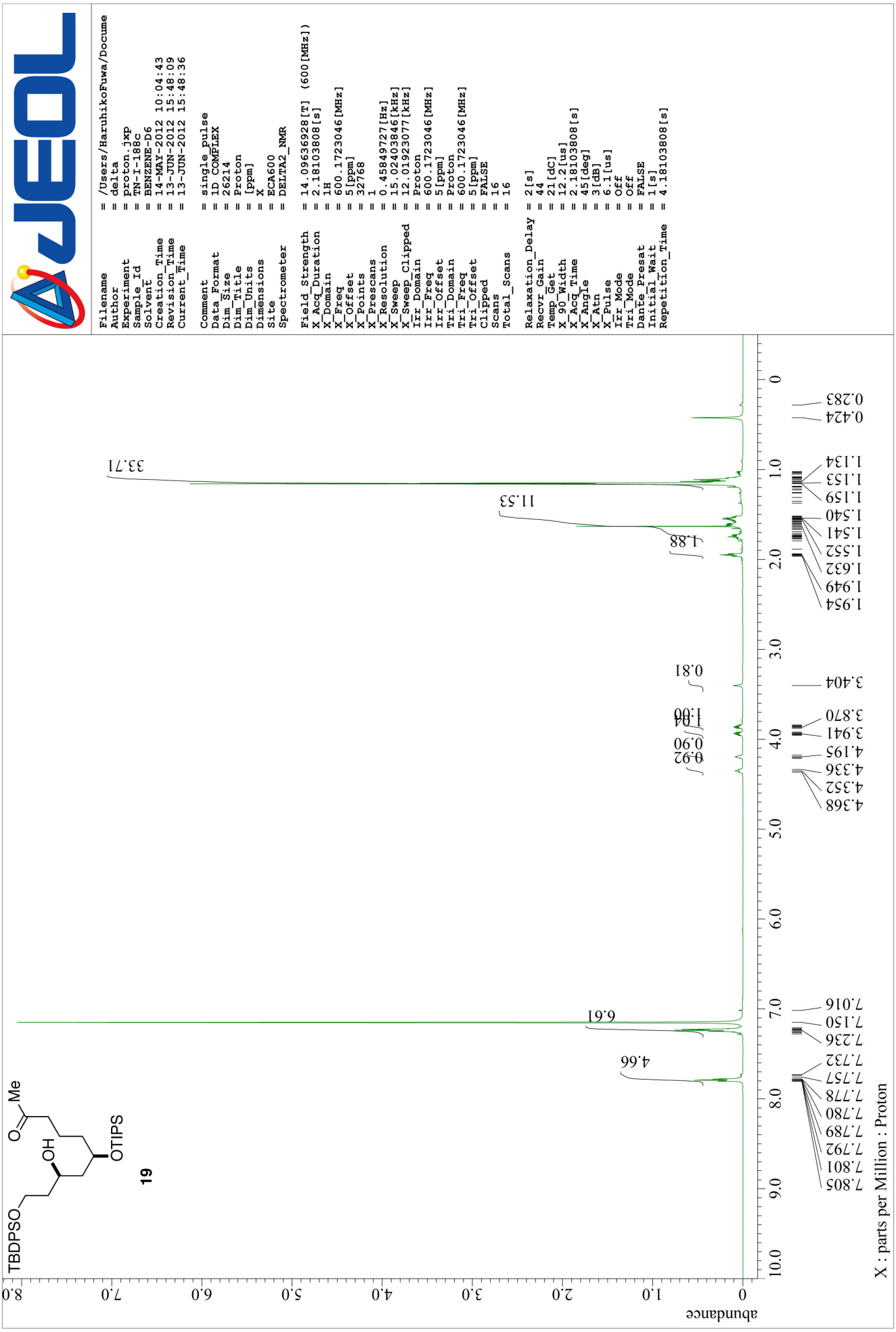


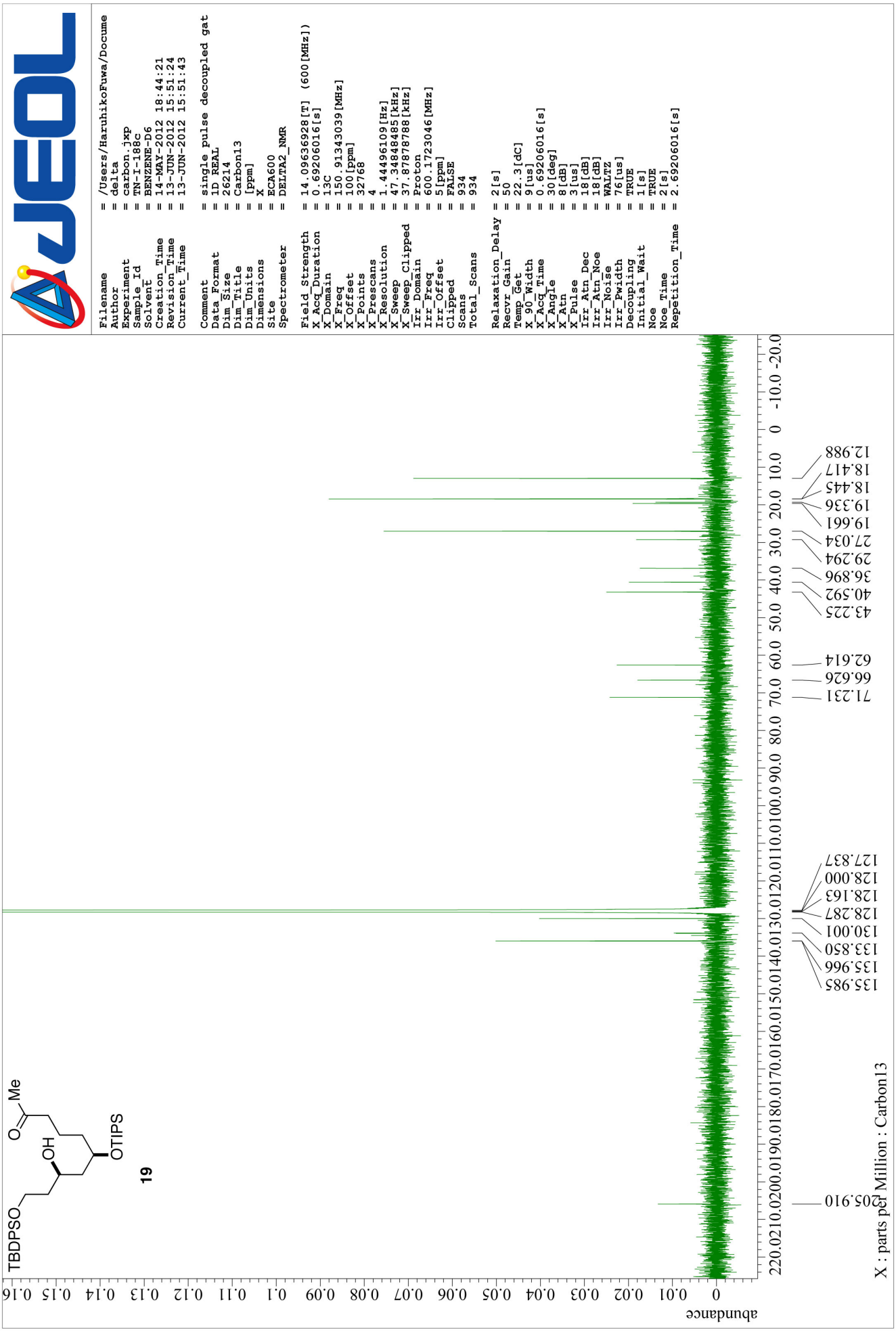


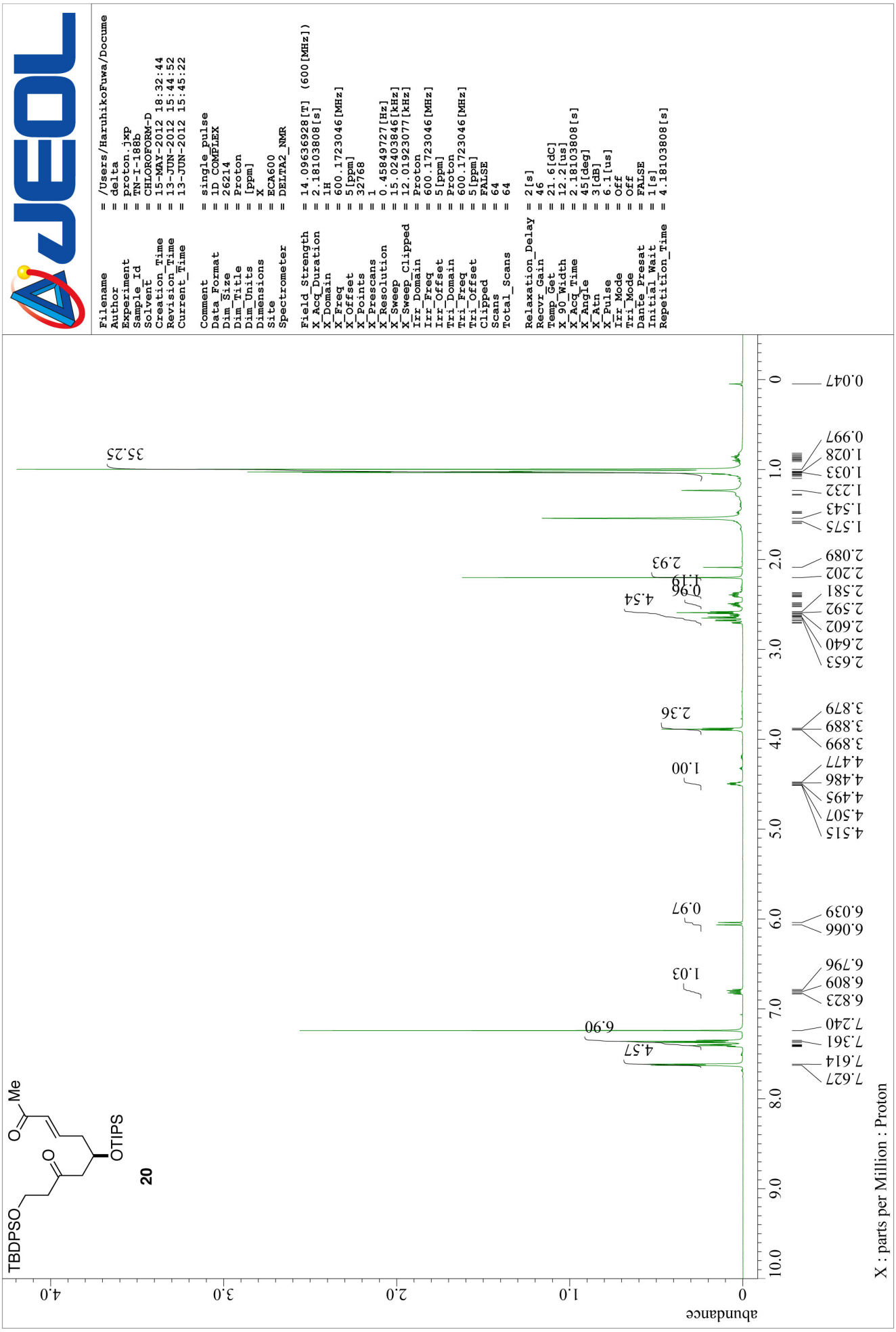


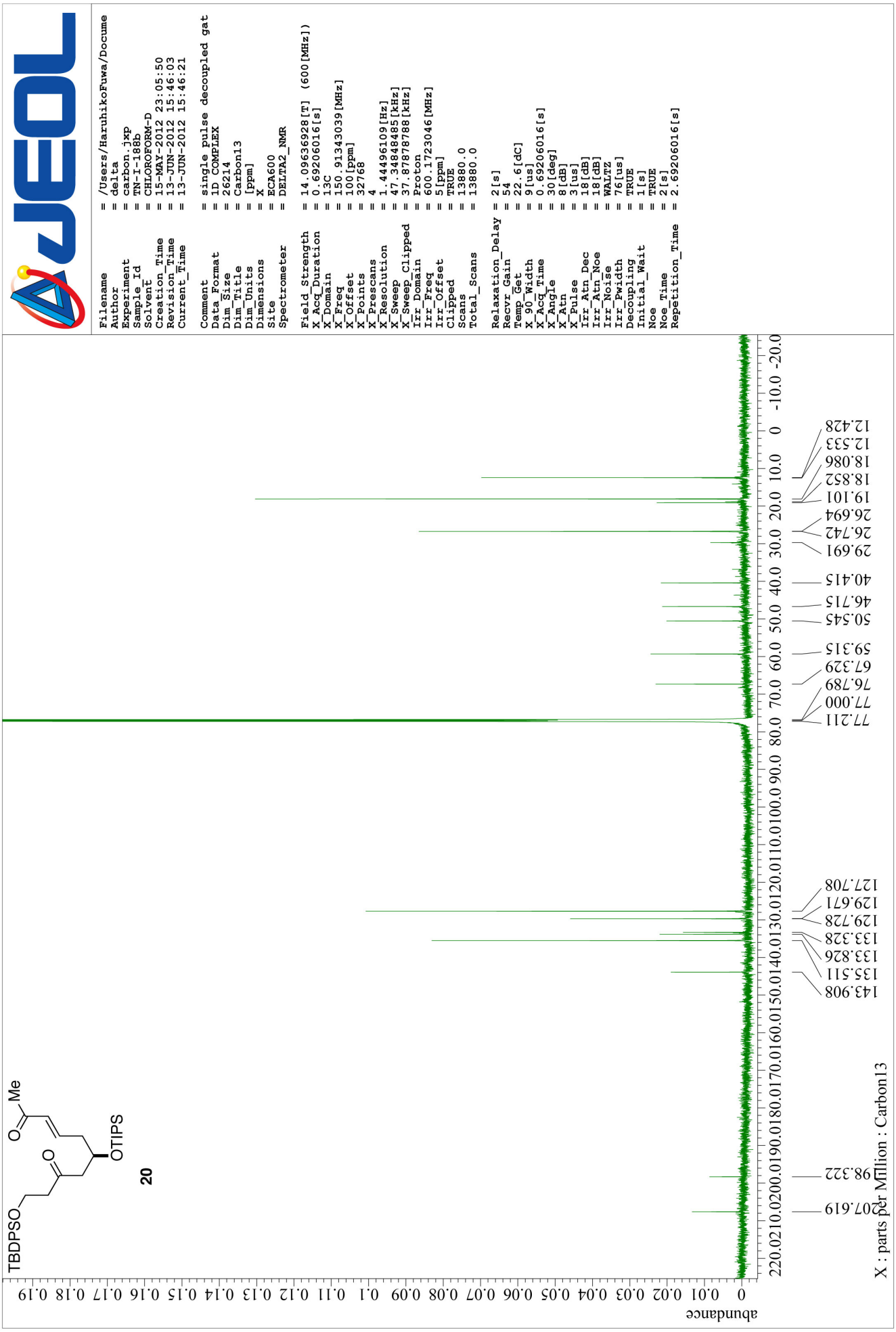

JEOL
Filename = /Users/HaruhikoFuwa/Docume
Author = delta
Experiment = proton.jxp
Sample_Id = TN-I-188a
Solvent = CHLOROFORM-D
Creation_Time = 3-AUG-2012 18:10:21
Revision_Time = 6-AUG-2012 13:29:59
Current_Time = 6-AUG-2012 13:42:30
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 2.18103808[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.45849727[Hz]
X_Sweep = 15.02403846[kHz]
X_Sweep_Clipped = 12.01923077[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 600.1723046[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 2[s]
Recvr_Gain = 32
Temp_Get = 22.1[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 2.18103808[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 5.8[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitionTime = 4.18103808[s]
OTBDPS
TIPSO
O
Me
O
18 (2,6-cis/2,6-trans 13:1)
41.85
3.71
2.02
3.11
1.10
0.99
78.54m
1.00
3.13
0.92
78.94m
0.17
0.24
6.63
4.47
7.645
7.633
7.631
7.629
7.623
7.620
7.617
7.353
6.961
4.233
4.223
4.215
4.206
4.197
4.186
4.178
3.717
3.495
3.490
2.721
2.707
2.394
2.093
1.666
1.413
1.236
1.212
1.183
1.044
1.034
1.029
1.022
1.017
0.903
abundance
X : parts per Million : Proton


JEOL
Filename = /Users/HaruhikoFuwa/Deskto
Author = delta
Experiment = carbon.jxp
Sample_Id = TN-I-188a
Solvent = CHLOROFORM-D
Creation_Time = 3-AUG-2012 18:13:03
Revision_Time = 6-AUG-2012 13:45:49
Current_Time = 6-AUG-2012 13:45:58
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 0.69206016[s]
X_Domain = 13C
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 144
Total_Scans = 144
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 22.6[dC]
X_90_Width = 9.25[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 8[dB]
X_Pulse = 3.08333333[us]
Irr_Atn_Dec = 19.327[dB]
Irr_Atn_Noe = 19.327[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe_Time = TRUE
Repetition_Time = 2[s]
2.69206016[s]
OTBDPS
TIPSO
O
Me
O
18 (2,6-cis/2,6-trans 13:1)
abundance
X : parts per Million : Carbon13
207.389
135.502
133.922
133.874
129.527
127.593
77.211
77.000
76.789
72.452
72.012
68.431
60.340
49.808
41.727
38.864
30.974
29.682
26.828
19.188
18.067
12.294
12.227


JEOL
Filename = /Users/HaruhikoFuwa/Docume
Author = delta
Experiment = proton.jxp
Sample_Id = TN-I-188c
Solvent = BENZENE-D6
Creation_Time = 14-MAY-2012 10:04:43
Revision_Time = 13-JUN-2012 15:48:09
Current_Time = 13-JUN-2012 15:48:36
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 2.18103808[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.45849727[Hz]
X_Sweep = 15.02403846[kHz]
X_Sweep_Clipped = 12.01923077[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 600.1723046[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 2[s]
Recvr_Gain = 44
Temp_Get = 21[dC]
X_90_Width = 12.2[us]
X_Acq_Time = 2.18103808[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.1[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 4.18103808[s]
TBDPSO
OH
OTIPS
O
Me
19
abundance
X : parts per Million : Proton
33.71
11.53
6.61
4.66
1.88
0.81
1.00
0.92
0.90
0.283
0.424
1.134
1.153
1.159
1.540
1.541
1.552
1.632
1.949
1.954
3.404
3.870
3.941
4.195
4.336
4.352
4.368
7.016
7.150
7.236
7.732
7.757
7.778
7.780
7.789
7.792
7.801
7.805




JEOL
Filename = /Users/HaruhikoFuwa/Docume
Author = delta
Experiment = proton.jxp
Sample_Id = TN-I-188b
Solvent = CHLOROFORM-D
Creation_Time = 15-MAY-2012 18:32:44
Revision_Time = 13-JUN-2012 15:44:52
Current_Time = 13-JUN-2012 15:45:22
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 2.18103808[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.45849727[Hz]
X_Sweep = 15.02403846[kHz]
X_Sweep_Clipped = 12.01923077[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 600.1723046[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 64
Total_Scans = 64
Relaxation_Delay = 2[s]
Recvr_Gain = 46
Temp_Get = 21.6[dC]
X_90_Width = 12.2[us]
X_Acq_Time = 2.18103808[s]
X_Angle = 45[deg]
X_Atn = 3[dB]
X_Pulse = 6.1[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 4.18103808[s]
TBDPSO
O
Me
O
OTIPS
20
35.25
6.90
4.57
4.54
2.93
2.36
1.19
1.03
1.00
0.97
0.96
0.047
abundance
X : parts per Million : Proton


JEOL
Filename = /Users/HaruhikoFuwa/Docume
Author = delta
Experiment = carbon.jxp
Sample_Id = TN-I-188b
Solvent = CHLOROFORM-D
Creation_Time = 15-MAY-2012 23:05:50
Revision_Time = 13-JUN-2012 15:46:03
Current_Time = 13-JUN-2012 15:46:21
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 0.69206016[s]
X_Domain = 13C
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 13880.0
Total_Scans = 13880.0
Relaxation_Delay = 2[s]
Recvr_Gain = 54
Temp_Get = 22.6[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 8[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 18[dB]
Irr_Atn_Noe = 18[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69206016[s]
TBDPSO
O
Me
O
OTIPS
20
abundance
X : parts per Million : Carbon13
77.211
77.000
76.789
67.329
59.315
50.545
46.715
40.415
29.691
26.742
26.694
19.101
18.852
18.086
12.533
12.428
143.908
135.511
133.826
133.328
129.728
129.671
127.708
98.322
207.619