Electronic Supplementary Information

Structure-activity relationship studies on vitamin D-based selective SREBP/SCAP inhibitor KK-052

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General

^{1}H and ^{13}C NMR spectra were recorded on JEOL AL-400 NMR (400 MHz) and ECP-600 NMR (600 MHz) spectrometers (Tokyo, Japan). ^{1}H NMR spectra were referenced with $(CH_3)_4Si$ (δ 0.00 ppm) or $CHCl_3$ (δ 7.26 ppm) as an internal standard. ^{13}C NMR spectra were referenced with deuterated solvent (δ 77.0 ppm for $CDCl_3$). IR spectra were recorded on a JASCO FT-IR-800 Fourier transform infrared spectrophotometer (Tokyo, Japan). High-resolution mass spectra were obtained on a SHIMADZU LCMS-IT-TOF mass spectrometer (Kyoto, Japan) with an electrospray ionization (ESI) method. Optical rotations were measured on a JASCO DIP-370 digital polarimeter (Tokyo, Japan). Column chromatography was performed on silica gel 60N (40-50 μm, Kanto Chemical Co., Inc., Tokyo, Japan). Preparative thin-layer chromatography was performed on silica gel 60 F_{254} (0.5 mm, Merck, Tokyo, Japan). All experiments were performed under anhydrous conditions in an argon atmosphere unless otherwise stated.

Synthesis of compounds 7a,b-14a,b

(6*R*)-3,3-Difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(4-methylphenyl)-2*H*-tetrazol-2-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (7a) and (6*R*)-3,3-difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(4-methylphenyl)-1*H*-tetrazol-1-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (7b)

To a solution of 5-(4-methylphenyl)-1*H*-tetrazole (29.5 mg, 0.184 mmol), Ph_3P (52.7 mg, 0.201 mmol), and 24,24-difluoro-CD-ring (**6**) (42.4 mg, 0.092 mmol) in CH_2Cl_2 (3 mL) was added diisopropyl azodicarboxylate (88 μL, 1.9 M in

toluene, 0.166 mmol) at 0°C, and the mixture was stirred at 0°C for 5 min and then at room temperature for 40 min. The mixture was evaporated in vacuo, and the residue was roughly purified by flash column chromatography on silica gel (hexane : EtOAc = 5 : 1 – 3 : 1) to obtain crude products (less polar and more polar products).

p-Toluenesulfonic acid monohydrate (109.6 mg, 0.576 mmol) was added to a solution of the above less polar crude product in MeOH (5 mL) and CH_2Cl_2 (2 mL). The mixture was stirred at room temperature for 1 h under air. *p*-Toluenesulfonic acid monohydrate (109.6 mg, 0.576 mmol) was added to the mixture and stirred at the same temperature for a further 30 min. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with CH_2Cl_2 three times, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 3 : 1) to obtain **7a** (13.5 mg, 30%) as a colorless oil.

7a: $[\alpha]_D^{27}$ +35.9 (c 1.04, $CHCl_3$); IR (neat) 3442, 1464, 1380, 1176, 1041, 1017, 830, 754 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.54 (s, 3H, 18-CH_3), 0.94 (d, *J* = 6.6 Hz, 3H, 21-CH_3), 1.25-2.04 (m, 23H), 2.41 (s, 3H, *p*-CH_3), 2.83-2.85 (m, 1H), 5.21-5.35 (m, 3H), 7.28 (d, *J* = 7.8 Hz, 2H), 8.02 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 21.5, 22.0, 23.3, 23.6, 26.8, 27.4 (t, *J* = 24.4 Hz, C23), 27.4, 29.1, 35.6, 40.1, 45.8, 50.1, 55.6, 56.1, 73.3 (t, *J* = 26.6 Hz, C25), 111.8, 124.8, 125.5 (t, *J* = 246.3 Hz, C24), 126.7, 129.5, 140.3, 147.7, 165.1 (tetrazole-C); HRMS (ESI^+) calcd for $C_{28}H_{40}N_4OF_2Na$ $[M+Na]^+$ 509.3062, found 509.3075.

p-Toluenesulfonic acid monohydrate (203.6 mg, 1.07 mmol) was added to

a solution of the above more polar crude product in MeOH (5 mL) and CH_2Cl_2 (2 mL). The mixture was stirred at room temperature for 70 min under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with CH_2Cl_2 three times, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 1) to obtain **7b** (12.7 mg, 28%) as a colorless oil.

7b: $[\alpha]_D^{27}$ +41.3 (c 0.98, $CHCl_3$); IR (neat) 3418, 1479, 1380, 1176, 1013, 826, 759 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.48 (s, 3H, 18-CH_3), 0.94 (d, *J* = 7.2 Hz, 3H, 21-CH_3), 1.24-2.01 (m, 23H), 2.45 (s, 3H, *p*-CH_3), 2.56-2.58 (m, 1H), 5.04-5.15 (m, 3H), 7.34 (d, *J* = 7.2 Hz, 2H), 7.59 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 21.5, 22.1, 23.1, 23.6, 26.7, 27.3, 27.4 (t, *J* = 24.5 Hz, C23), 28.9, 35.6, 40.0, 45.6, 45.6, 55.5, 56.1, 73.3 (t, *J* = 27.3 Hz, C25), 113.1, 121.2, 125.4 (t, *J* = 245.6 Hz, C24), 128.7, 129.8, 141.6, 146.2, 154.1 (tetrazole-C); HRMS (ESI^+) calcd for $C_{28}H_{40}N_4OF_2Na$ $[M+Na]^+$ 509.3062, found 509.3079.

(6*R*)-3,3-Difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(4-trifluoromethylphenyl)-2*H*-tetrazol-2-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (8a) and (6*R*)-3,3-difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(4-trifluoromethylphenyl)-1*H*-tetrazol-1-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (8b)

To a solution of 5-(4-trifluoromethylphenyl)-1*H*-tetrazole (61.5 mg, 0.287 mmol), Ph_3P (72.2 mg, 0.275 mmol), and 24,24-difluoro-CD-ring (**6**) (60.1 mg, 0.131 mmol) in CH_2Cl_2 (8 mL) was added diisopropyl azodicarboxylate (124 μL, 1.9 M

in toluene, 0.235 mmol) at 0°C, and the mixture was stirred at 0°C for 70 min. The mixture was evaporated in vacuo, and the residue was roughly purified by flash column chromatography on silica gel (hexane : EtOAc = 4 : 1) to obtain crude products (less polar and more polar products).

p-Toluenesulfonic acid monohydrate (386.8 mg, 2.03 mmol) was added to a solution of the above less polar crude product in MeOH (5 mL) and CH_2Cl_2 (2 mL). The mixture was stirred at room temperature for 35 min under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with CH_2Cl_2 three times, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 3 : 1) to obtain **8a** (41.4 mg, 58%) as a white powder.

8a: mp. 53.0-54.0 °C; $[\alpha]_D^{27}$ +35.2 (c 3.17, $CHCl_3$); IR (neat) 3431, 1471, 1324, 1173, 1133, 1066, 858 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.55 (s, 3H, 18-CH_3), 0.95 (d, *J* = 6.0 Hz, 3H, 21-CH_3), 1.26-2.05 (m, 23H), 2.83-2.86 (m, 1H), 5.27-5.35 (m, 3H), 7.74 (d, *J* = 8.1 Hz, 2H), 8.26 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.4, 23.56, 26.8, 27.4, 27.4 (t, *J* = 24.4 Hz, C23), 29.1, 35.6, 40.1, 45.9, 50.3, 55.6, 56.1, 73.3 (t, *J* = 27.2 Hz, C25), 111.5, 123.9 (q, *J* = 270.0 Hz, -CF_3), 125.5 (t, *J* = 246.9 Hz, C24), 125.8, 127.0, 131.0, 131.9 (q, *J* = 31.7 Hz, *p*-C-CF_3), 148.3, 163.8 (tetrazole-C); HRMS (ESI^-) calcd for $C_{28}H_{37}N_4OF_5Cl$ $[M+Cl]^-$ 575.2582, found 575.2577.

p-Toluenesulfonic acid monohydrate (411.7 mg, 2.05 mmol) was added to a solution of the above more polar crude product in MeOH (5 mL) and CH_2Cl_2 (6 mL). The mixture was stirred at room temperature for 60 min under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room

temperature, the mixture was extracted with CH_2Cl_2 three times, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 1) to obtain **8b** (15.1 mg, 21%) as a white powder.

8b: mp. 174.5-175.0 °C; $[\alpha]_D^{27}$ +40.8 (c 1.16, $CHCl_3$); IR (neat) 3522, 1459, 1328, 1173, 1129, 1073, 858 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.45 (s, 3H, 18-CH_3), 0.93 (d, *J* = 6.4 Hz, 3H, 21-CH_3), 1.22-2.10 (m, 23H), 2.53-2.57 (m, 1H), 5.04-5.20 (m, 3H), 7.82 (d, *J* = 8.7 Hz, 2H), 7.85 (d, *J* = 8.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.4, 23.55, 26.7, 27.3, 27.3 (t, *J* = 24.8 Hz, C23), 28.9, 35.6, 39.9, 45.6, 45.9, 55.5, 56.1, 73.3 (t, *J* = 27.2 Hz, C25), 112.5, 123.5 (q, *J* = 271.7 Hz, -CF_3), 125.4 (t, *J* = 246.0 Hz, C24), 126.1, 127.8, 129.3, 133.1 (q, *J* = 32.7 Hz, *p*-C-CF_3), 146.9, 153.0 (tetrazole-C); HRMS (ESI^-) calcd for $C_{28}H_{37}N_4OF_5Cl$ $[M+Cl]^-$ 575.2582, found 575.2590.

(6*R*)-3,3-Difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(4-chlorophenyl)-2*H*-tetrazol-2-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (9a) and (6*R*)-3,3-difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(4-chlorophenyl)-1*H*-tetrazol-1-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (9b)

To a solution of 5-(4-chlorophenyl)-1*H*-tetrazole (44.7 mg, 0.248 mmol), Ph_3P (67.6 mg, 0.258 mmol), and 24,24-difluoro-CD-ring (**6**) (49.8 mg, 0.109 mmol) in CH_2Cl_2 (8 mL) was added diisopropyl azodicarboxylate (103 μL, 1.9 M in toluene, 0.196 mmol) at 0°C, and the mixture was stirred at the same temperature for 110 min. The mixture was evaporated in vacuo, and the residue was roughly purified by flash column chromatography on silica gel (hexane :

EtOAc = 5 : 1) to obtain crude products (less polar and more polar products).

p-Toluenesulfonic acid monohydrate (435.3 mg, 2.29 mmol) was added to a solution of the above less polar crude product in MeOH (5 mL) and CH_2Cl_2 (5 mL). The mixture was stirred at room temperature for 1 h under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with CH_2Cl_2 three times, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 3 : 1) to obtain **9a** (31.4 mg, 57%) as a colorless oil.

9a: $[\alpha]_D^{27}$ +41.4 (c 2.42, $CHCl_3$); IR (neat) 3414, 1456, 1326, 1175, 1093, 1017, 841, 759 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.54 (s, 3H, 18-CH_3), 0.94 (d, *J* = 6.4 Hz, 3H, 21-CH_3), 1.23-2.05 (m, 23H), 2.81-2.85 (m, 1H), 5.20-5.35 (m, 3H), 7.45 (dt, 2.3, 8.2 Hz, 2H), 8.07 (dt, 2.3, 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.3, 23.5, 26.7, 27.4 (t, *J* = 24.8 Hz, C23), 29.0, 35.6, 40.1, 45.8, 50.2, 55.6, 56.1, 73.3 (t, *J* = 27.2 Hz, C25), 111.6, 125.5 (t, *J* = 246.0 Hz, C24), 126.1, 128.0, 129.1, 136.1, 148.0, 164.1 (tetrazole-C); HRMS (ESI^+) calcd for $C_{27}H_{37}N_4OF_2ClNa$ $[M+Na]^+$ 529.2516, found 529.2531.

p-Toluenesulfonic acid monohydrate (389.5 mg, 2.05 mmol) was added to a solution of the above more polar crude product in MeOH (5 mL) and CH_2Cl_2 (5 mL). The mixture was stirred at room temperature for 85 min under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with CH_2Cl_2 three times, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 1) to obtain **9b** (15.3 mg, 28%) as a colorless oil.

9b: $[\alpha]_D^{27}$ +43.0 (c 1.18, $CHCl_3$); IR (neat) 3423, 1471, 1380, 1174, 1093, 1013, 838, 739 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.47 (s, 3H, 18-CH_3), 0.93 (d, *J* = 6.4 Hz, 3H, 21-CH_3), 1.24-2.05 (m, 23H), 2.54-2.58 (m, 1H), 5.03-5.18 (m, 3H), 7.53 (dt, *J* = 2.1, 8.2 Hz, 2H), 7.65 (dt, *J* = 2.1, 8.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.1, 23.5, 26.7, 27.3, 27.3 (t, *J* = 24.3 Hz, C23), 28.9, 35.6, 39.9, 45.6, 45.8, 55.5, 56.1, 73.3 (t, *J* = 27.2 Hz, C25), 112.8, 122.6, 125.4 (t, *J* = 246.0 Hz, C24), 129.5, 130.1, 137.6, 146.7, 153.2 (tetrazole-C); HRMS (ESI^+) calcd for $C_{27}H_{37}N_4OF_2ClNa$ $[M+Na]^+$ 529.2516, found 529.2510.

(6*R*)-3,3-Difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(3-methylphenyl)-2*H*-tetrazol-2-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (10a) and (6*R*)-3,3-difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(3-methylphenyl)-1*H*-tetrazol-1-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (10b)

To a solution of 5-(3-methylphenyl)-1*H*-tetrazole (28.6 mg, 0.179 mmol), Ph_3P (47.8 mg, 0.182 mmol), and 24,24-difluoro-CD-ring (**6**) (51.4 mg, 0.112 mmol) in CH_2Cl_2 (8 mL) was added diisopropyl azodicarboxylate (83 μL, 1.9 M in toluene, 0.157 mmol) at 0°C, and the mixture was stirred at 0°C for 5 min and then at room temperature for 20 min. To the mixture were added diisopropyl azodicarboxylate (83 μL, 1.9 M in toluene, 0.157 mmol) and Ph_3P (83.7 mg, 0.319 mmol) and stirred at room temperature for 40 min. The mixture was evaporated in vacuo, and the residue was roughly purified on a preparative silica gel TLC plate (hexane : EtOAc = 3 : 1) to obtain crude products (less polar and more polar products).

p-Toluenesulfonic acid monohydrate (584.6 mg, 3.07 mmol) was added to

a solution of the above less polar crude product in MeOH (10 mL) and CH_2Cl_2 (5 mL). The mixture was stirred at room temperature for 90 min under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with CH_2Cl_2 three times, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 2 : 1) to obtain **10a** (38.1 mg, 70%) as a colorless oil.

10a: $[\alpha]_D^{27}$ +23.1 (c 2.93, $CHCl_3$); IR (neat) 3423, 1471, 1380, 1180, 1017, 858, 754 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.55 (s, 3H, 18-CH_3), 0.95 (d, *J* = 6.0 Hz, 3H, 21-CH_3), 1.26-2.06 (m, 23H), 2.43 (s, 3H), 2.83-2.86 (m, 1H), 5.22-5.35 (m, 3H), 7.27 (d, *J* = 7.8 Hz, 1H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.93 (d, *J* = 7.8 Hz, 1H), 7.97 (s, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 21.4, 22.0, 23.3, 23.6, 26.8, 27.4 (t, *J* = 24.4 Hz, C23), 27.4, 29.0, 35.6, 40.1, 45.8, 50.2, 55.6, 56.1, 73.4 (t, *J* = 27.3 Hz, C25), 111.8, 123.9, 125.5 (t, *J* = 246.3 Hz, C24), 127.4, 127.5, 128.8, 130.9, 138.6, 147.8, 165.1 (tetrazole-C); HRMS (ESI^+) calcd for $C_{28}H_{40}N_4OF_2Na$ $[M+Na]^+$ 509.3062, found 509.3068.

p-Toluenesulfonic acid monohydrate (376.1 mg, 1.98 mmol) was added to a solution of the above more polar crude product in MeOH (10 mL) and CH_2Cl_2 (5 mL). The mixture was stirred at room temperature for 60 min under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with CH_2Cl_2 three times, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 2) to obtain **10b** (11.0 mg, 20%) as a colorless oil.

10b: $[\alpha]_D^{27}$ +44.6 (c 0.85, $CHCl_3$); IR (neat) 3411, 1475, 1380, 1180, 1125, 1021,

918, 854, 739 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.46 (s, 3H, 18-CH_3), 0.93 (d, *J* = 6.0 Hz, 3H, 21-CH_3), 1.24-1.99 (m, 23H), 2.43 (s, 3H), 2.53-2.56 (m, 1H), 5.04-5.16 (m, 3H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H), 7.45 (d, *J* = 7.8 Hz, 1H), 7.51 (s, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 21.4, 22.0, 23.1, 23.6, 26.7, 27.3, 27.3 (t, *J* = 24.5 Hz, C23), 28.9, 35.6, 40.0, 45.5, 45.6, 55.5, 56.1, 73.3 (t, *J* = 27.2 Hz, C25), 113.0, 124.1, 125.4 (t, *J* = 246.3 Hz, C24), 128.9, 129.5, 139.2, 146.2, 154.2 (tetrazole-C); HRMS (ESI^+) calcd for $C_{28}H_{40}N_4OF_2Na$ $[M+Na]^+$ 509.3062, found 509.3039.

(6*R*)-3,3-Difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(3,5-dichlorophenyl)-2*H*-tetrazol-2-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (11a) and (6*R*)-3,3-difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(3,5-dichlorophenyl)-1*H*-tetrazol-1-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (11b)

To a solution of 5-(3,5-dichlorophenyl)-1*H*-tetrazole (40.8 mg, 0.190 mmol), Ph_3P (47.6 mg, 0.181 mmol), and 24,24-difluoro-CD-ring (**6**) (40.8 mg, 0.089 mmol) in CH_2Cl_2 (4 mL) was added diisopropyl azodicarboxylate (138 μL, 1.9 M in toluene, 0.262 mmol) at 0°C, and the mixture was stirred at 0°C for 35 min and then at room temperature for 25 min. The mixture was evaporated in vacuo, and the residue was roughly purified on a preparative silica gel TLC plate (hexane : EtOAc = 3 : 1) to obtain crude products (less polar and more polar products).

p-Toluenesulfonic acid monohydrate (200.3 mg, 1.05 mmol) was added to a solution of the above less polar crude product in MeOH (10 mL) and CH_2Cl_2 (5 mL). The mixture was stirred at room temperature for 2 h under air. After the

reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with EtOAc three times, washed with brine, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 1) and followed by re-purification on a preparative silica gel TLC plate (hexane : EtOAc = 2 : 1) to obtain **11a** (27.6 mg, 57%) as a colorless oil.

11a: $[\alpha]_D^{27}$ +33.8 (c 2.12, $CHCl_3$); IR (neat) 3439, 1571, 1515, 1444, 1399, 1173, 1017, 862, 735 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.55 (s, 3H, 18-CH_3), 0.95 (d, *J* = 6.0 Hz, 3H, 21-CH_3), 1.24-2.05 (m, 23H), 2.81-2.84 (m, 1H), 5.25-5.34 (m, 3H), 7.45 (t, *J* = 2.4 Hz, 1H), 8.07 (d, *J* = 2.4 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.3, 23.6, 26.8, 27.4 (t, *J* = 24.4 Hz, C23), 27.4, 29.0, 35.6, 40.1, 45.9, 50.4, 55.6, 56.1, 73.4 (t, *J* = 27.3 Hz, C25), 111.4, 125.1, 125.5 (t, *J* = 246.3 Hz, C24), 130.0, 130.4, 135.6, 148.3, 162.9 (tetrazole-C); HRMS (ESI^-) calcd for $C_{28}H_{37}N_4O_3F_2Cl_2$ $[M+HCOO]^-$ 585.2216, found 585.2215.

p-Toluenesulfonic acid monohydrate (580.1 mg, 3.05 mmol) was added to a solution of the above more polar crude product in MeOH (20 mL). The mixture was stirred at room temperature for 1 h under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with EtOAc three times, washed with brine, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 1) and followed by re-purification on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 1) to obtain **11b** (15.0 mg, 31%) as a colorless oil.

11b: $[\alpha]_D^{27}$ +30.1 (c 1.15, $CHCl_3$) ; IR (neat) 3435, 1567, 1527, 1451, 1380, 1176, 1013, 905, 866, 727 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.55 (s, 3H, 18-

CH_3), 0.95 (d, *J* = 6.0 Hz, 3H, 21-CH_3), 1.24-2.05 (m, 23H), 2.81-2.84 (m, 1H), 5.25-5.34 (m, 3H), 7.45 (t, *J* = 2.4 Hz, 1H), 8.07 (d, *J* = 2.4 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.1, 23.7, 26.7, 27.3, 27.3 (t, *J* = 24.4 Hz, C23), 28.9, 35.6, 39.9, 45.6, 46.1, 55.5, 56.1, 73.4 (t, *J* = 27.3 Hz, C25), 112.4, 125.4 (t, *J* = 245.7 Hz, C24), 127.0, 127.2, 131.2, 136.1, 147.1, 152.0 (tetrazole-C); HRMS (ESI$^-$) calcd for $C_{28}H_{37}N_4O_3F_2Cl_2$ $[M+HCOO]^-$ 585.2216, found 585.2221.

(6*R*)-3,3-Difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(3-fluorophenyl)-2*H*-tetrazol-2-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (12a) and (6*R*)-3,3-difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(3-fluorophenyl)-1*H*-tetrazol-1-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (12b)

To a solution of 5-(3-fluorophenyl)-1*H*-tetrazole (36.4 mg, 0.222 mmol), Ph_3P (58.9 mg, 0.225 mmol), and 24,24-difluoro-CD-ring (**6**) (52.9 mg, 0.115 mmol) in CH_2Cl_2 (8 mL) was added diisopropyl azodicarboxylate (103 μL, 1.9 M in toluene, 0.196 mmol) at 0°C, and the mixture was stirred at 0°C for 20 min. The mixture was evaporated in vacuo, and the residue was roughly purified by flash column chromatography on silica gel (hexane : EtOAc = 5 : 1 – 2 : 1) to obtain crude products (less polar and more polar products).

p-Toluenesulfonic acid monohydrate (123.3 mg, 0.65 mmol) was added to a solution of the above less polar crude product in MeOH (10 mL). The mixture was stirred at room temperature for 1 h under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with EtOAc three times, washed with brine and dried over

Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 2 : 1) to obtain **12a** (34.8 mg, 62%) as a colorless oil.

12a: $[\alpha]_D^{27}$ +38.9 (c 2.68, $CHCl_3$); IR (neat) 3439, 1471, 1380, 1225, 1176, 1021, 763 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.54 (s, 3H, 18-CH_3), 0.94 (d, *J* = 6.0 Hz, 3H, 21-CH_3), 1.24-2.04 (m, 23H), 2.83-2.85 (m, 1H), 5.28-5.37 (m, 3H), 7.22 (dd, *J* = 8.4, 10.2 Hz, 1H), 7.27 (t, *J* = 7.5 Hz, 3H), 7.24-7.46 (m, 1H), 8.12 (td, *J* = 1.8, 7.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.3, 23.5, 26.8, 27.3 (t, *J* = 24.5 Hz, C23), 27.3, 29.0, 35.6, 40.1, 45.8, 50.2, 55.6, 56.1, 73.3 (t, *J* = 27.3 Hz, C25), 111.5, 113.8 (d, *J* = 24.5 Hz), 117.0 (d, *J* = 21.5 Hz), 122.4 (d, *J* = 2.9 Hz), 125.5 (t, *J* = 246.3 Hz, C24), 129.6 (d, *J* = 8.7 Hz), 130.5 (d, *J* = 8.6 Hz), 148.1, 163.0 (d, *J* = 244.2 Hz, *m*-C-F), 164.0 (d, *J* = 3.0 Hz, tetrazole-C); HRMS (ESI^+) calcd for $C_{27}H_{37}N_4OF_3Na$ $[M+Na]^+$ 513.2812, found 513.2817.

p-Toluenesulfonic acid monohydrate (211.9 mg, 1.11 mmol) was added to a solution of the above more polar crude product in MeOH (10 mL). The mixture was stirred at room temperature for 40 min under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with EtOAc three times, washed with brine and dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 1) to obtain **12b** (10.2 mg, 18%) as a colorless oil.

12b: $[\alpha]_D^{27}$ +66.9 (c 0.79, $CHCl_3$); IR (neat) 3411, 1475, 1384, 1204, 1176, 1017, 739 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.54 (s, 3H, 18-CH_3), 0.94 (d, *J* = 6.0 Hz, 3H, 21-CH_3), 1.24-2.04 (m, 23H), 2.83-2.85 (m, 1H), 5.28-5.37 (m, 3H), 7.22

(dd, *J* = 8.4, 10.2 Hz, 1H), 7.27 (t, *J* = 7.5 Hz, 3H), 7.24-7.46 (m, 1H), 8.12 (td, *J* = 1.8, 7.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.0, 23.6, 26.7, 27.3, 27.3 (t, *J* = 24.5 Hz, C23), 27.3, 28.9, 35.6, 39.9, 45.6, 45.9, 55.5, 56.1, 73.3 (t, *J* = 26.6 Hz, C25), 112.7, 116.1 (d, *J* = 23.0 Hz), 118.3 (d, *J* = 21.6 Hz), 124.6 (d, *J* = 4.4 Hz), 125.4 (t, *J* = 245.6 Hz, C24), 126.1 (d, *J* = 8.6 Hz), 131.0 (d, *J* = 8.6 Hz), 146.7, 153.0 (tetrazole-C), 162.7 (d, *J* = 247.1 Hz, *m*-C-F); HRMS (ESI$^+$) calcd for $C_{27}H_{37}N_4OF_3Na$ $[M+Na]^+$ 513.2812, found 513.2821.

(6*R*)-3,3-Difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(2-chlorophenyl)-2*H*-tetrazol-2-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (13a) and (6*R*)-3,3-difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(2-chlorophenyl)-1*H*-tetrazol-1-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (13b)

To a solution of 5-(2-chlorophenyl)-1*H*-tetrazole (39.9 mg, 0.221 mmol), Ph_3P (58.2 mg, 0.222 mmol), and 24,24-difluoro-CD-ring (**6**) (51.7 mg, 0.113 mmol) in CH_2Cl_2 (8 mL) was added diisopropyl azodicarboxylate (103 μL, 1.9 M in toluene, 0.196 mmol) at 0°C, and the mixture was stirred at 0°C for 35 min. The mixture was evaporated in vacuo, and the residue was roughly purified by flash column chromatography on silica gel (hexane : EtOAc = 5 : 1) to obtain crude products (less polar and more polar products).

p-Toluenesulfonic acid monohydrate (573.2 mg, 3.01 mmol) was added to a solution of the above less polar crude product in MeOH (20 mL). The mixture was stirred at room temperature for 5 h under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the

mixture was extracted with EtOAc three times, washed with brine and dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 3 : 1) to obtain **13a** (22.7 mg, 40%) as a colorless oil.

13a: $[\alpha]_D^{27}$ +37.7 (c 1.75, $CHCl_3$); IR (neat) 3435, 1446, 1380, 1176, 1125, 1073, 1038, 754 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.54 (s, 3H, 18-CH_3), 0.94 (d, *J* = 6.4 Hz, 3H, 21-CH_3), 1.24-2.05 (m, 23H), 2.83-2.87 (m, 1H), 5.29-5.39 (m, 3H), 7.35-7.42 (m, 2H), 7.52-7.54 (m, 1H), 7.91-7.96 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.4, 23.6, 26.8, 27.4 (t, *J* = 24.8 Hz, C23), 27.4, 29.0, 35.6, 40.1, 45.8, 50.3, 55.6, 56.1, 73.3 (t, *J* = 26.7 Hz, C25), 111.6, 125.5 (t, *J* = 246.0 Hz, C24), 126.8, 126.8, 130.8, 130.9, 131.3, 133.1, 148.1, 163.2 (tetrazole-C); HRMS (ESI^+) calcd for $C_{27}H_{37}N_4OF_2ClNa$ $[M+Na]^+$ 529.2516, found 529.2519.

p-Toluenesulfonic acid monohydrate (580.7 mg, 3.05 mmol) was added to a solution of the above more polar crude product in MeOH (20 mL). The mixture was stirred at room temperature for 90 min under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with EtOAc three times, washed with brine and dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 1) to obtain **13b** (20.7 mg, 36%) as a colorless oil.

13b: $[\alpha]_D^{27}$ +29.4 (c 1.59, $CHCl_3$); IR (neat) 3407, 1459, 1380, 1176, 1125, 1073, 1020, 767 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.40 (s, 3H, 18-CH_3), 0.91 (d, *J* = 6.9 Hz, 3H, 21-CH_3), 1.18-2.02 (m, 23H), 2.36-2.40 (m, 1H), 4.91-5.06 (m, 3H), 7.40-7.45 (m, 2H), 7.51-7.58 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.8, 18.6,

21.9, 23.1, 23.5, 26.7, 27.2, 27.3 (t, *J* = 24.8 Hz, C23), 28.5, 35.6, 40.0, 45.4, 55.4, 56.0, 73.3 (t, *J* = 26.7 Hz, C25), 112.0, 124.4, 125.4 (t, *J* = 246.0 Hz, C24), 127.2, 130.1, 131.9, 132.5, 133.9, 147.1, 152.2 (tetrazole-C); HRMS (ESI^+) calcd for $C_{27}H_{37}N_4OF_2ClNa$ $[M+Na]^+$ 529.2516, found 529.2531.

(6*R*)-3,3-Difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(2-fluorophenyl)-2*H*-tetrazol-2-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (14a) and (6*R*)-3,3-difluoro-6-[(1*R*,3a*S*,7a*R*,*E*)-4-{2-[5-(2-fluorophenyl)-1*H*-tetrazol-1-yl]ethylidene}-7a-methyloctahydro-1*H*-inden-1-yl]-2-methylheptan-2-ol (14b)

To a solution of 5-(2-fluorophenyl)-1*H*-tetrazole (36.5 mg, 0.222 mmol), Ph_3P (59.2 mg, 0.226 mmol), and 24,24-difluoro-CD-ring (**6**) (51.4 mg, 0.112 mmol) in CH_2Cl_2 (8 mL) was added diisopropyl azodicarboxylate (103 μL, 1.9 M in toluene, 0.196 mmol) at 0°C, and the mixture was stirred at 0°C for 30 min. The mixture was evaporated in vacuo, and the residue was roughly purified by flash column chromatography on silica gel (hexane : EtOAc = 5 : 1 – 2 : 1) to obtain crude products (less polar and more polar products).

p-Toluenesulfonic acid monohydrate (585.7 mg, 3.08 mmol) was added to a solution of the above less polar crude product in MeOH (20 mL). The mixture was stirred at room temperature for 105 min under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with EtOAc three times, washed with brine, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 2 : 1) to obtain **14a** (30.8 mg, 56%) as a colorless oil.

14a: $[\alpha]_D^{27}$ +36.7 (c 2.37, $CHCl_3$); IR (neat) 3435, 1479, 1376, 1228, 1180, 1037, 754 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 0.54 (s, 3H, 18-CH_3), 0.94 (d, *J* = 6.0 Hz, 3H, 21-CH_3), 1.24-2.04 (m, 23H), 2.83-2.85 (m, 1H), 5.28-5.37 (m, 3H), 7.22 (dd, *J* = 8.4, 10.2 Hz, 1H), 7.27 (t, *J* = 7.5 Hz, 3H), 7.24-7.46 (m, 1H), 8.12 (td, *J* = 1.8, 7.8 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.8, 18.6, 22.0, 23.3, 23.6, 26.8, 27.3 (t, *J* = 24.5 Hz, C23), 27.4, 29.1, 35.6, 40.1, 45.8, 50.3, 55.6, 56.1, 73.3 (t, *J* = 27.3 Hz, C25), 111.6, 115.8 (d, *J* = 11.4 Hz), 116.6 (d, *J* = 20.1 Hz), 124.4 (d, *J* = 4.2 Hz), 125.5 (t, *J* = 246.3 Hz, C24), 129.9, 131.1 (d, *J* = 8.7 Hz), 148.0, 160.1 (d, *J* = 254.3 Hz, *o*-C-F), 161.2 (d, *J* = 4.4 Hz, tetrazole-C); HRMS (ESI^+) calcd for $C_{27}H_{37}N_4OF_3$ $[M+Na]^+$ 513.2812, found 513.2797.

p-Toluenesulfonic acid monohydrate (619.1 mg, 3.25 mmol) was added to a solution of the above more polar crude product in MeOH (20 mL). The mixture was stirred at room temperature for 1 h under air. After the reaction was quenched with H_2O and saturated aqueous $NaHCO_3$ at room temperature, the mixture was extracted with EtOAc three times, washed with brine, dried over Na_2SO_4, filtered, and concentrated. The residue was purified on a preparative silica gel TLC plate (hexane : EtOAc = 1 : 1) to obtain **14b** (21.6 mg, 39%) as a colorless oil.

14b: $[\alpha]_D^{27}$ +38.0 (c 1.66, $CHCl_3$); IR (neat) 3423, 1479, 1384, 1217, 1173, 1021, 774, 739 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.36 (s, 3H, 18-CH_3), 0.90 (d, *J* = 6.4 Hz, 3H, 21-CH_3), 1.20-2.06 (m, 23H), 2.47-2.52 (m, 1H), 4.98-5.12 (m, 3H), 7.24-7.29 (m, 1H), 7.33 (td, *J* = 1.8, 7.4 Hz, 1H), 7.56-7.62 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.7, 18.6, 21.9, 23.1, 23.5, 26.7, 27.3, 27.3 (t, *J* = 24.3 Hz, C23), 28.6, 35.6, 40.0, 45.5, 45.6, 45.7, 55.4, 56.0, 73.3 (t, *J* = 27.2 Hz, C25), 112.1, 112.9 (d, *J* = 14.3 Hz), 116.3 (d, *J* = 21.0 Hz), 125.0 (d, *J* =

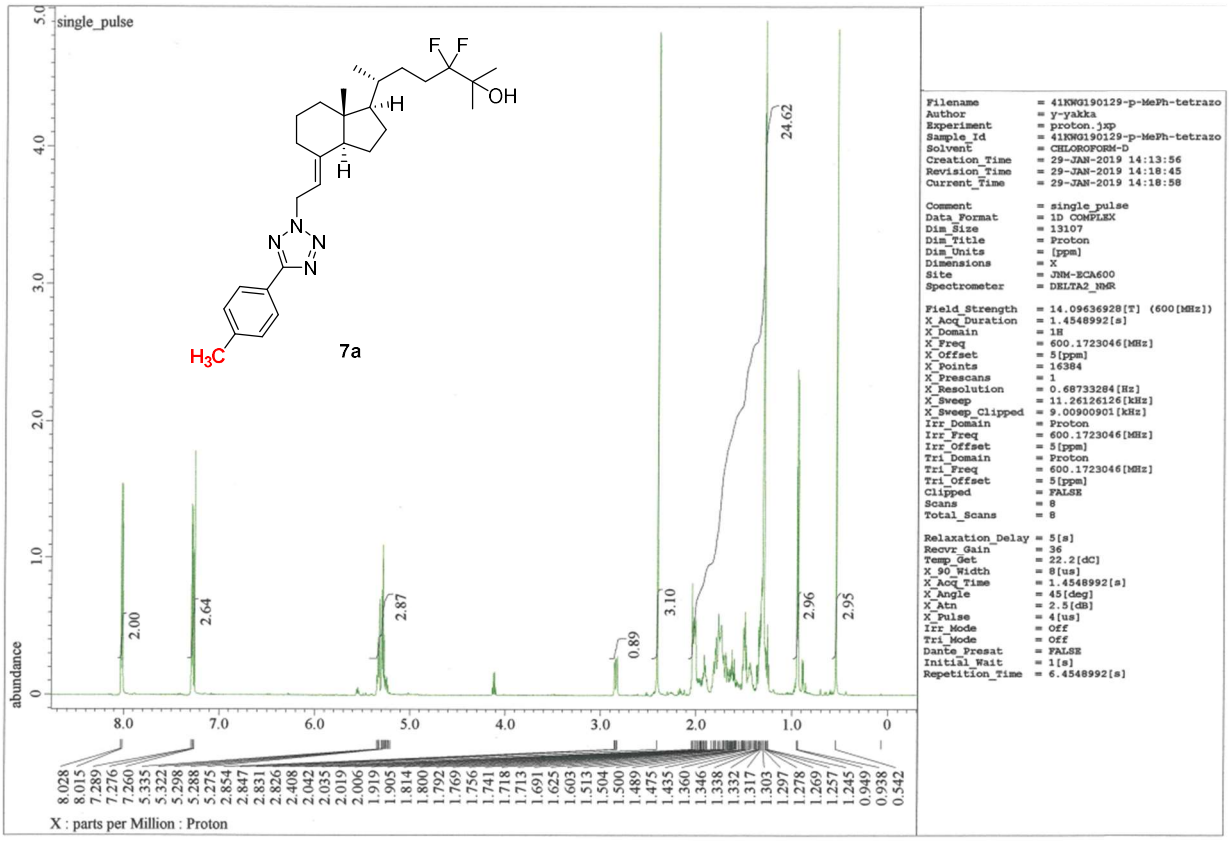
2.9 Hz), 125.4 (t, *J* = 246.0 Hz, C24), 131.8, 133.5 (d, *J* = 7.6 Hz), 147.0, 150.1 (tetrazole-C), 159.6 (d, *J* = 248.8 Hz, *o*-C-F); HRMS (ESI^+) calcd for $C_{27}H_{37}N_4OF_3Na$ $[M+Na]^+$ 513.2812, found 513.2825.

Cell Culture

CHO-K1 cells were maintained in medium A (1:1 mixture of Ham's F-12 medium and DMEM, supplemented with 100 units/mL penicillin, 100 μg/mL streptomycin sulfate, and 5% [v/v] fetal bovine serum) at 37°C in a humidified 5% CO_2 incubator.

Luciferase Reporter Assay

CHO-K1 cells were seeded in 96-well plates at 8×10^3 cells per well in medium A and incubated for 24 h. For the SREBP reporter assay, cells were co -transfected with an SRE-1-driven luciferase reporter plasmid (pSRE-Luc) and an actin promoter-driven β-galactosidase expression plasmid (pAc -β-gal) at a 20:1 ratio, using FuGENE HD Transfection Reagent (Promega) according to the manufacturer's protocol. For the VDR reporter assay, Cignal Vitamin D Receptor Reporter (QIAGEN) was used for transfection instead of pSRE-Luc. After 20 h, the medium was changed to medium B (1:1 mixture of Ham's F-12 medium and DMEM, supplemented with 100 units/mL penicillin, 100 μg/mL streptomycin sulfate, 5% [v/v] lipid-depleted serum, 50 μM compactin (Tokyo Chemical Industry), and 50 μM lithium mevalonate (Sigma-Aldrich) containing the specific test compounds. After 24 h incubation, the cells in each well were lysed with 100 μL of 1x Reporter Lysis Buffer (Promega), and 50 μL aliquots were used to measure luciferase and β-gal activities. Luciferase activity was measured using the Steady-Glo Luciferase Assay System (Promega), and β-gal activity was measured using the β-Galactosidase Enzyme Assay System (Promega). Luciferase activity was normalized to β-gal activity.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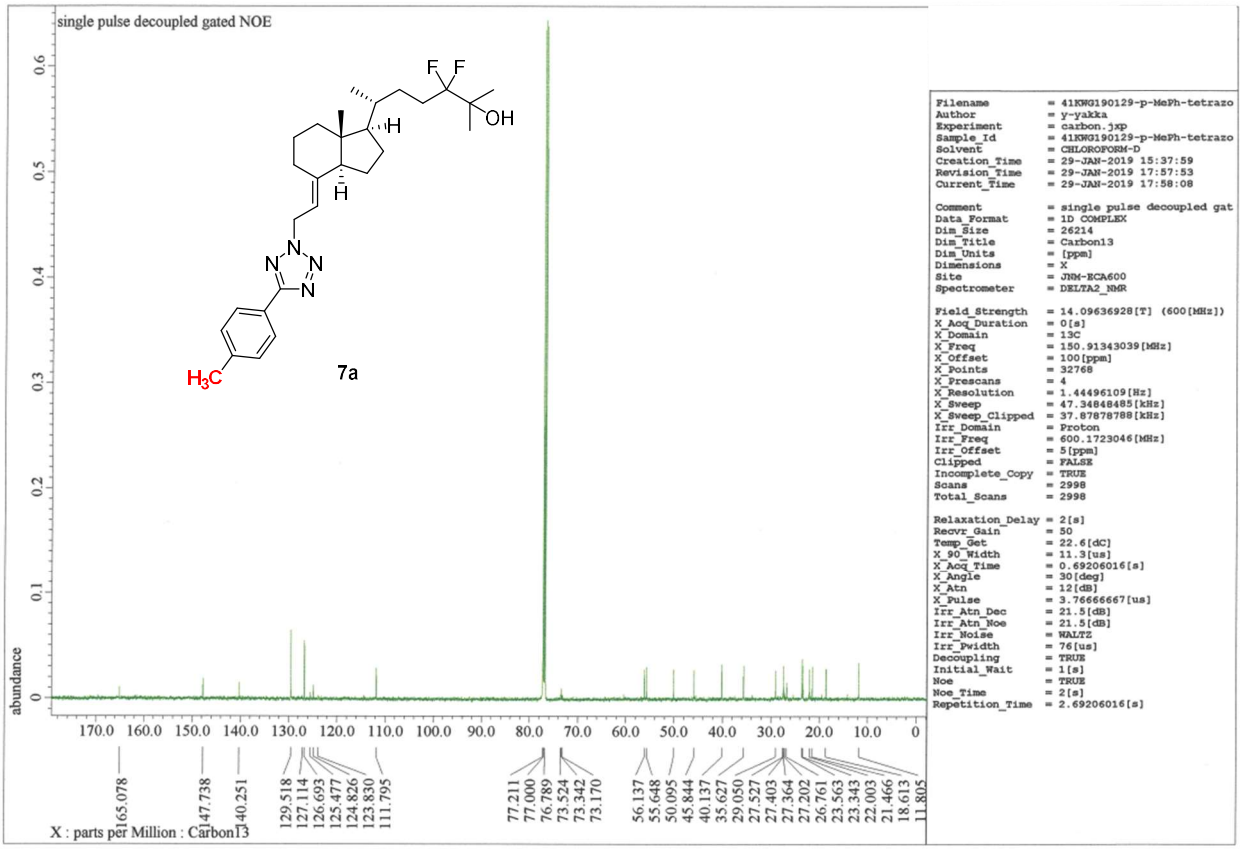
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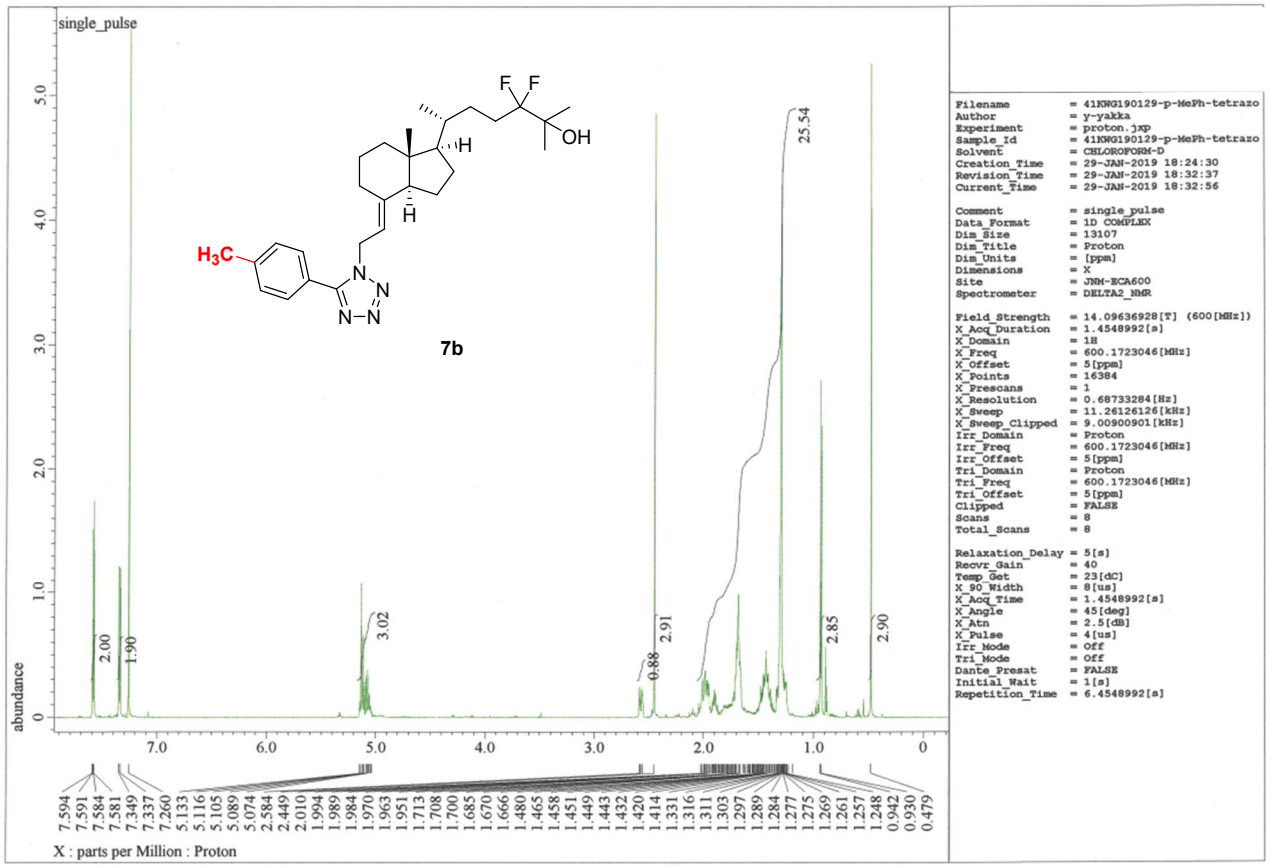
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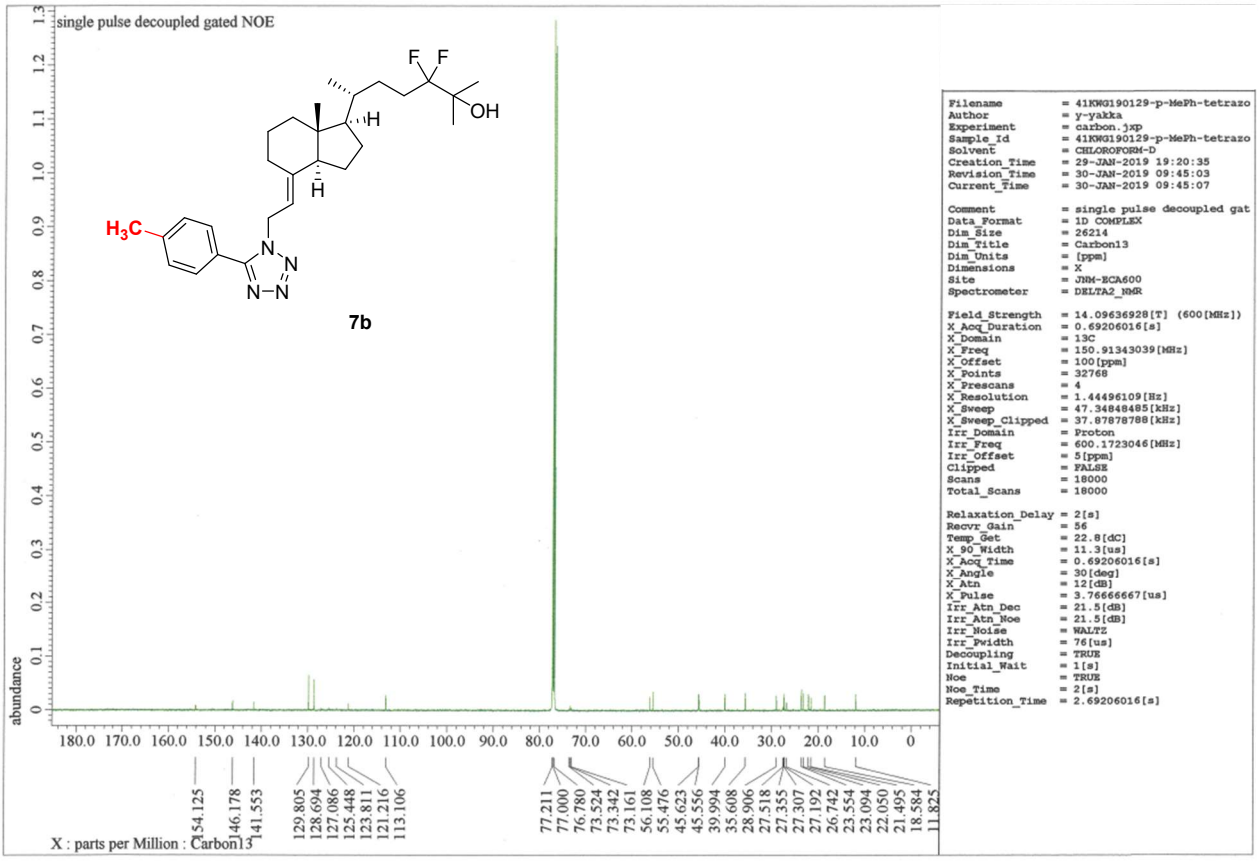
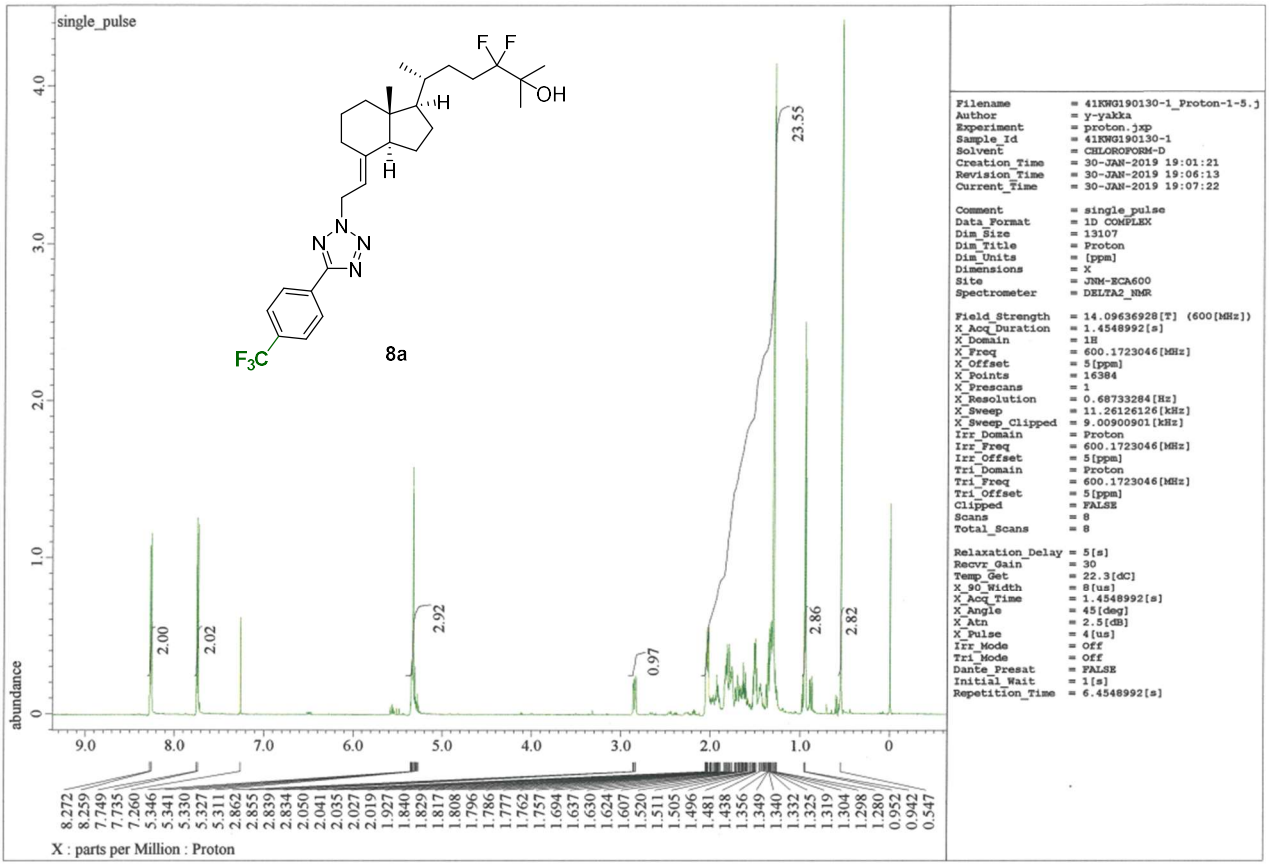
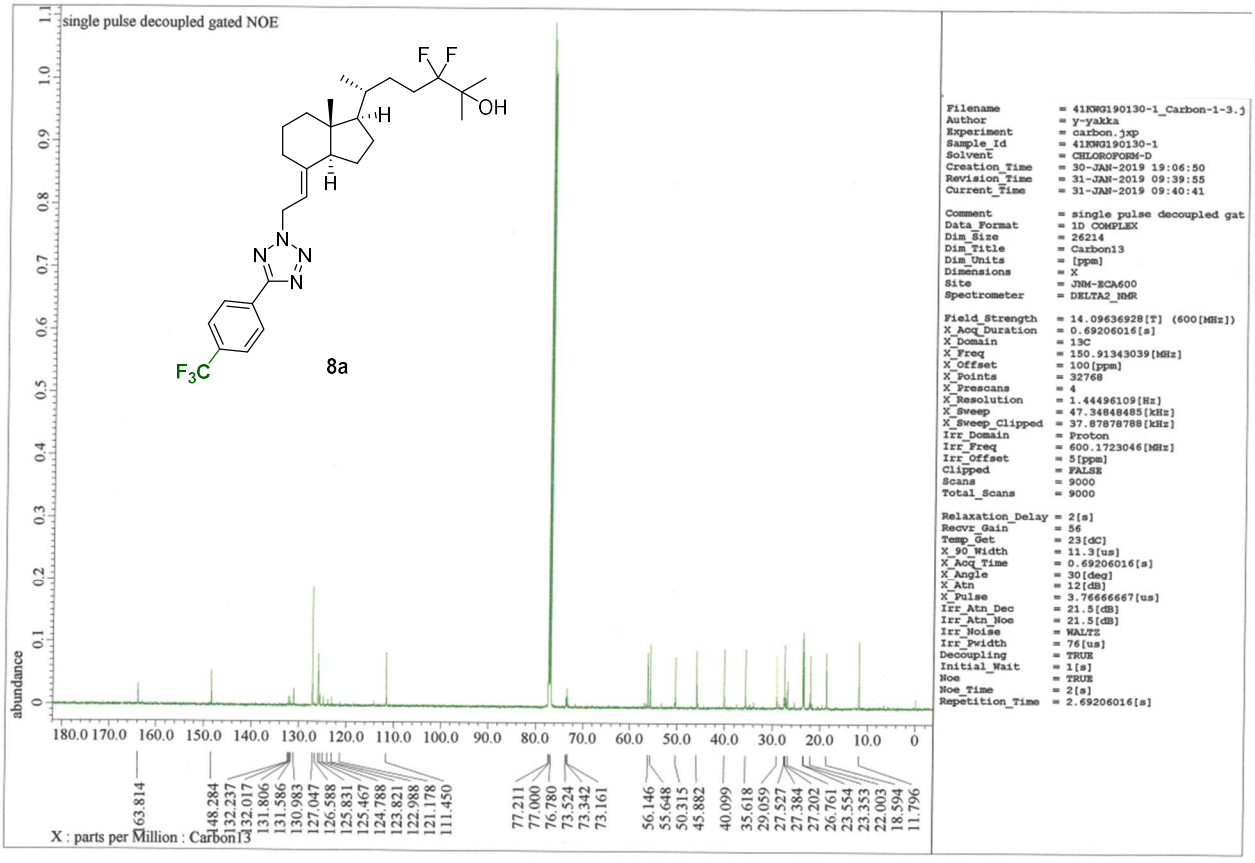
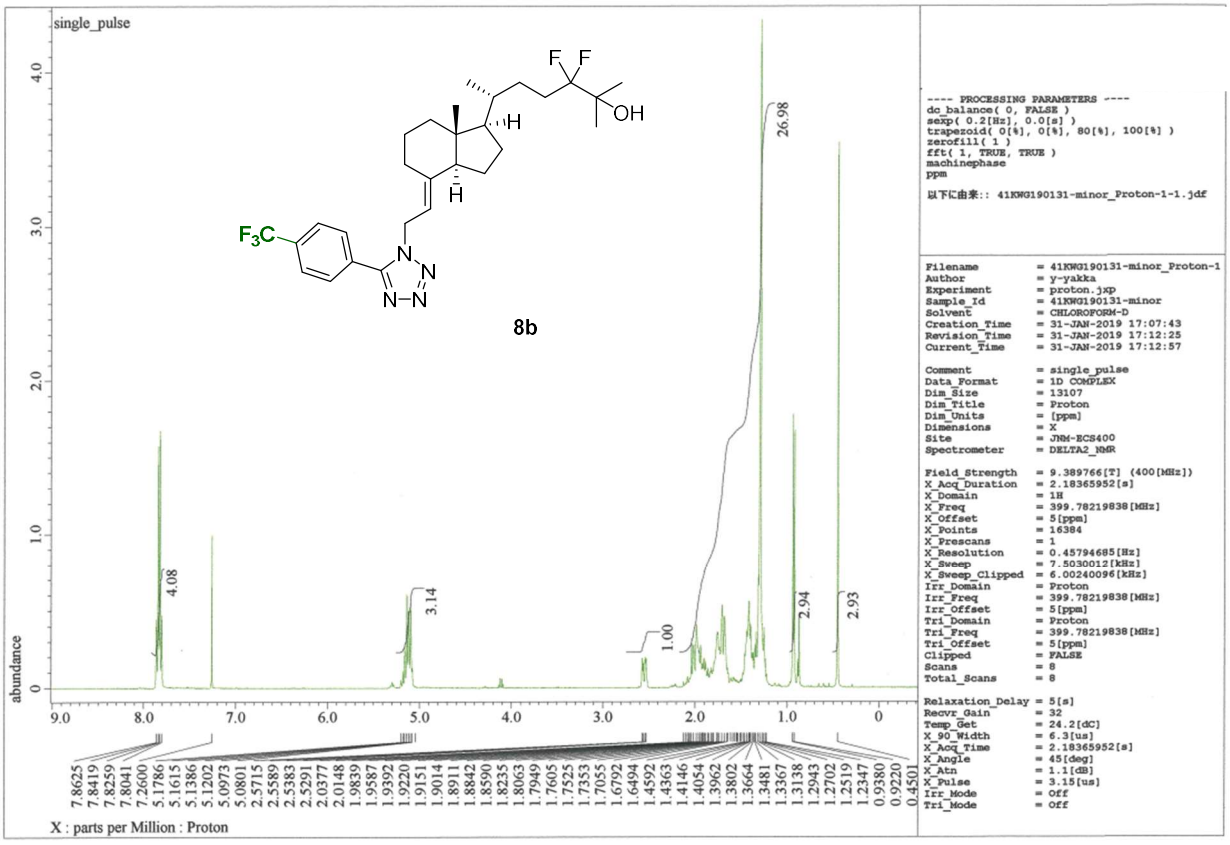
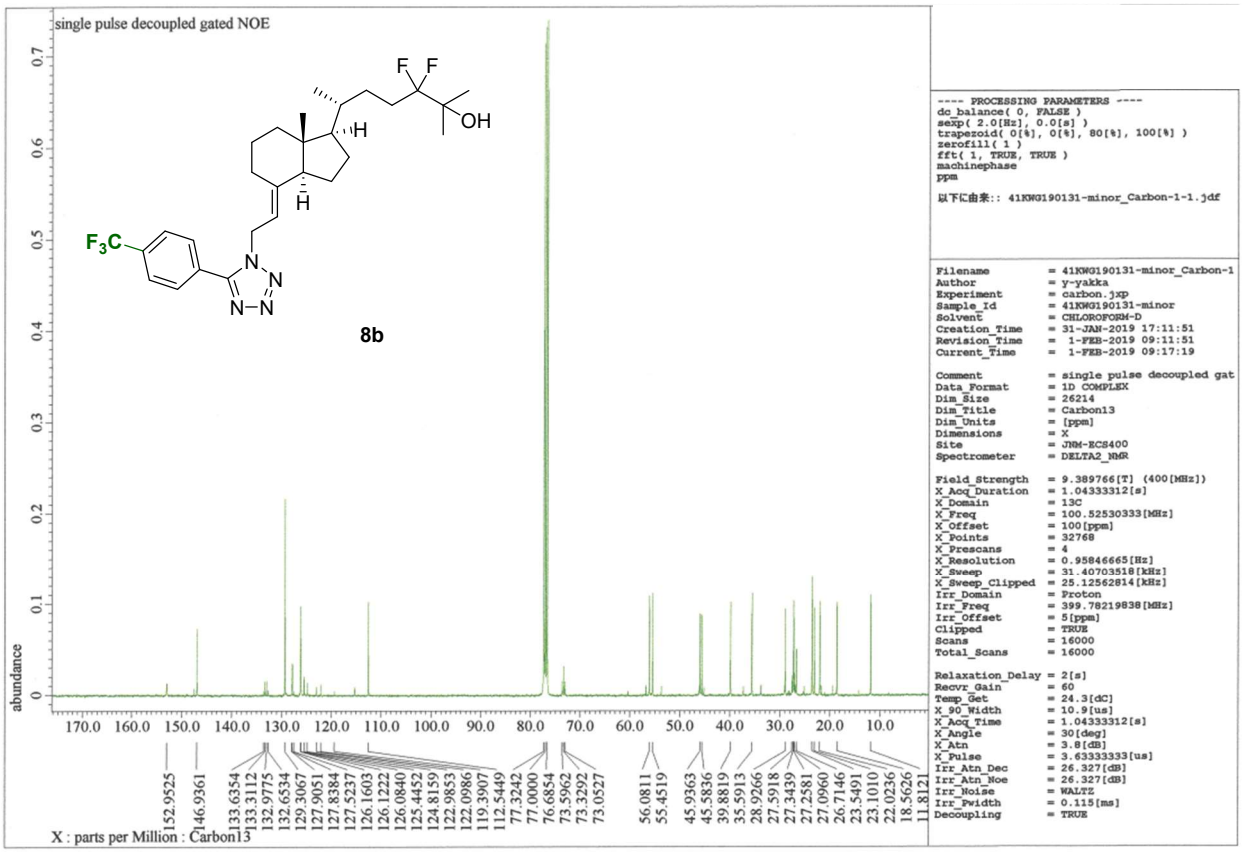
0
7.594 7.591 7.584 7.581 7.349 7.337 7.260 5.133 5.116 5.105 5.089 5.074 2.584 2.449 2.010 1.994 1.989 1.984 1.970 1.963 1.951 1.713 1.708 1.700 1.685 1.670 1.666 1.480 1.465 1.458 1.451 1.449 1.443 1.432 1.420 1.414 1.331 1.316 1.311 1.303 1.297 1.289 1.284 1.277 1.275 1.269 1.261 1.257 1.248 0.942 0.930 0.479
X : parts per Million : Proton
Filename = 41KWG190129-p-MePh-tetrazo
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190129-p-MePh-tetrazo
Solvent = CHLOROFORM-D
Creation_Time = 29-JAN-2019 18:24:30
Revision_Time = 29-JAN-2019 18:32:37
Current_Time = 29-JAN-2019 18:32:56
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 1.4548992[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.68733284[Hz]
X_Sweep = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[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 = 5[s]
Recvr_Gain = 40
Temp_Get = 23[dC]
X_90_Width = 8[us]
X_Acq_Time = 1.4548992[s]
X_Angle = 45[deg]
X_Atn = 2.5[dB]
X_Pulse = 4[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.4548992[s]

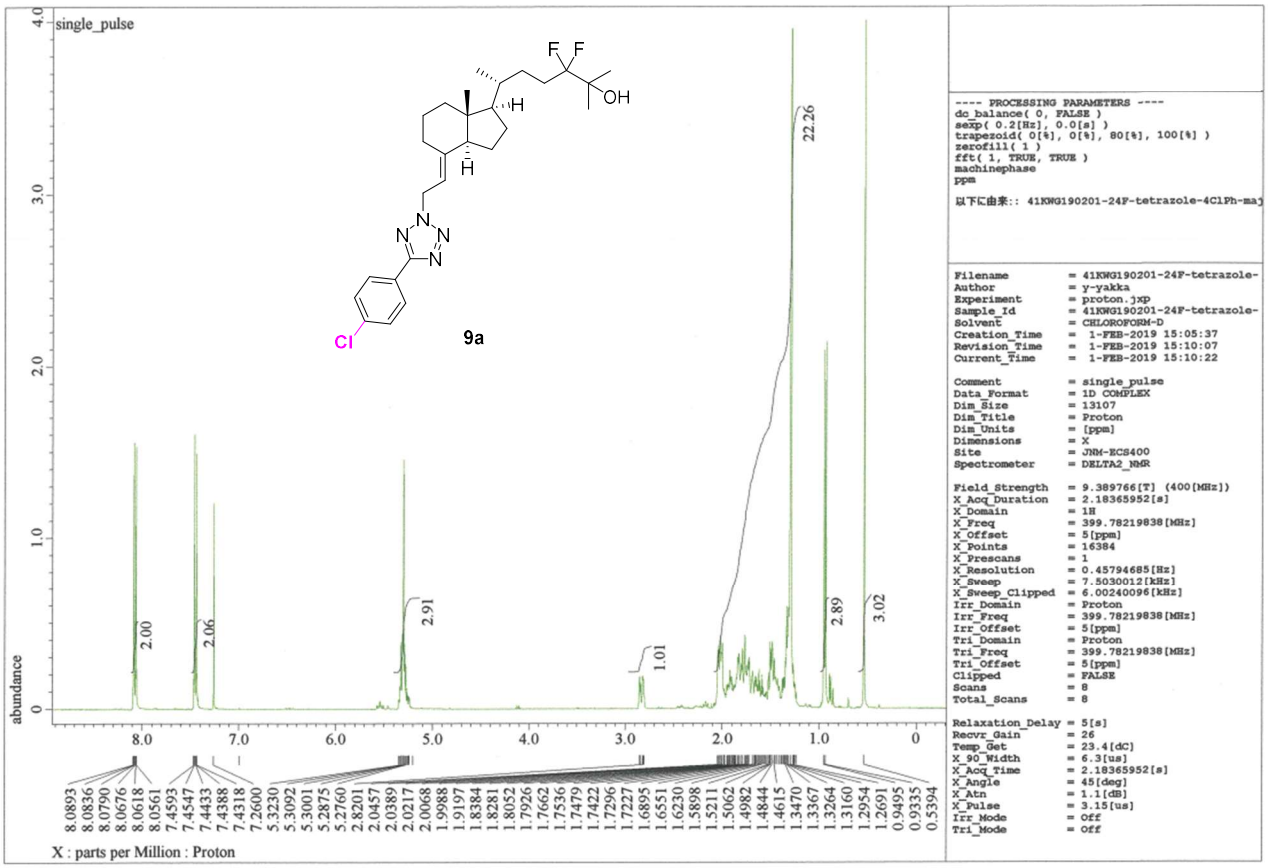

single pulse decoupled gated NOE
F F
OH
H
H
H3C
N
N
N–N
7b
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
154.125 146.178 141.553 129.805 128.694 127.086 125.448 123.811 121.216 113.106 77.211 77.000 76.780 73.524 73.342 73.161 56.108 55.476 45.623 45.556 39.994 35.608 28.906 27.518 27.355 27.307 27.192 26.742 23.554 23.094 22.050 21.495 18.584 11.825
X : parts per Million : Carbon13
Filename = 41KWG190129-p-MePh-tetrazo
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190129-p-MePh-tetrazo
Solvent = CHLOROFORM-D
Creation_Time = 29-JAN-2019 19:20:35
Revision_Time = 30-JAN-2019 09:45:03
Current_Time = 30-JAN-2019 09:45:07
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-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 = 18000
Total_Scans = 18000
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 22.8[dC]
X_90_Width = 11.3[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.76666667[us]
Irr_Atn_Dec = 21.5[dB]
Irr_Atn_Noe = 21.5[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]

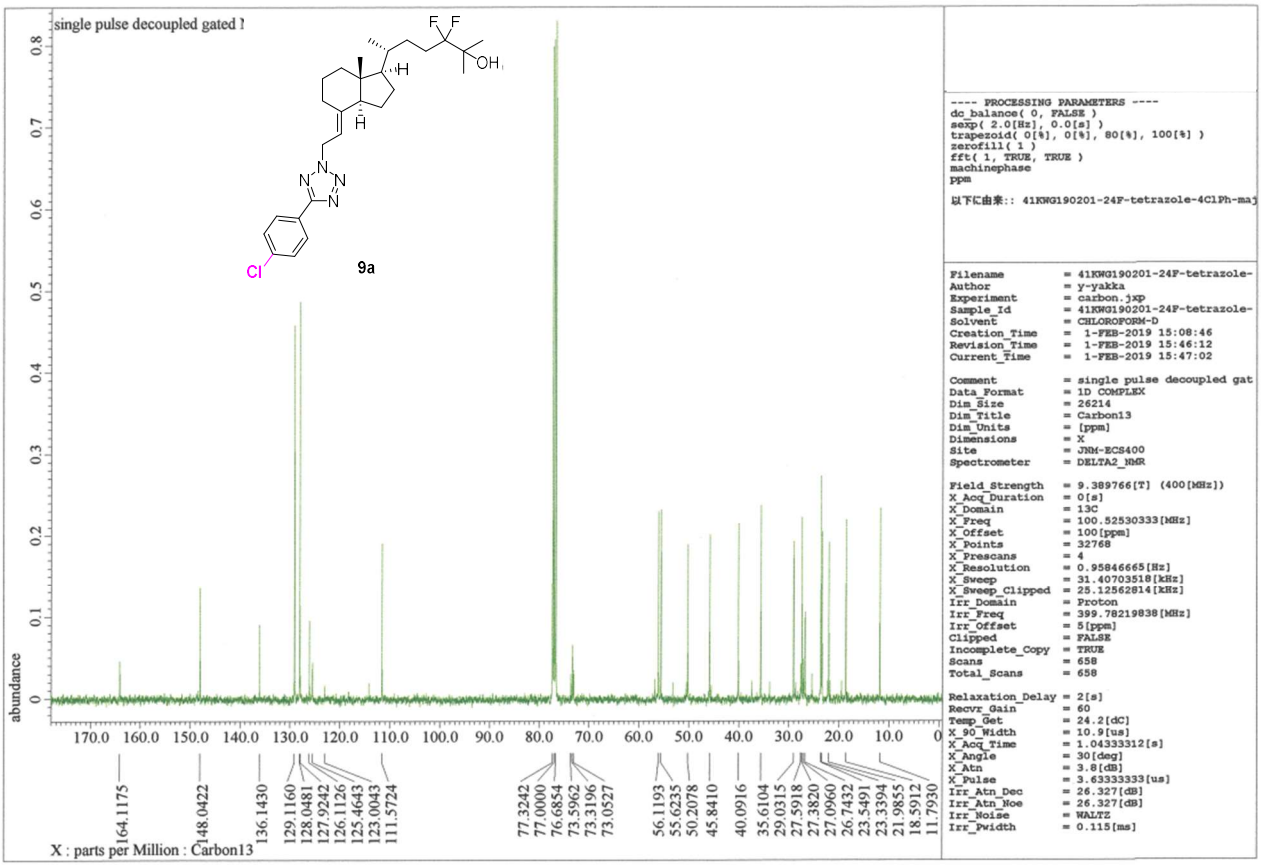

single_pulse
8a
abundance
X : parts per Million : Proton
Filename = 41KWG190130-1_Proton-1-5.j
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190130-1
Solvent = CHLOROFORM-D
Creation_Time = 30-JAN-2019 19:01:21
Revision_Time = 30-JAN-2019 19:06:13
Current_Time = 30-JAN-2019 19:07:22
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 1.4548992[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.68733284[Hz]
X_Sweep = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[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 = 5[s]
Recvr_Gain = 30
Temp_Get = 22.3[dC]
X_90_Width = 8[us]
X_Acq_Time = 1.4548992[s]
X_Angle = 45[deg]
X_Atn = 2.5[dB]
X_Pulse = 4[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.4548992[s]

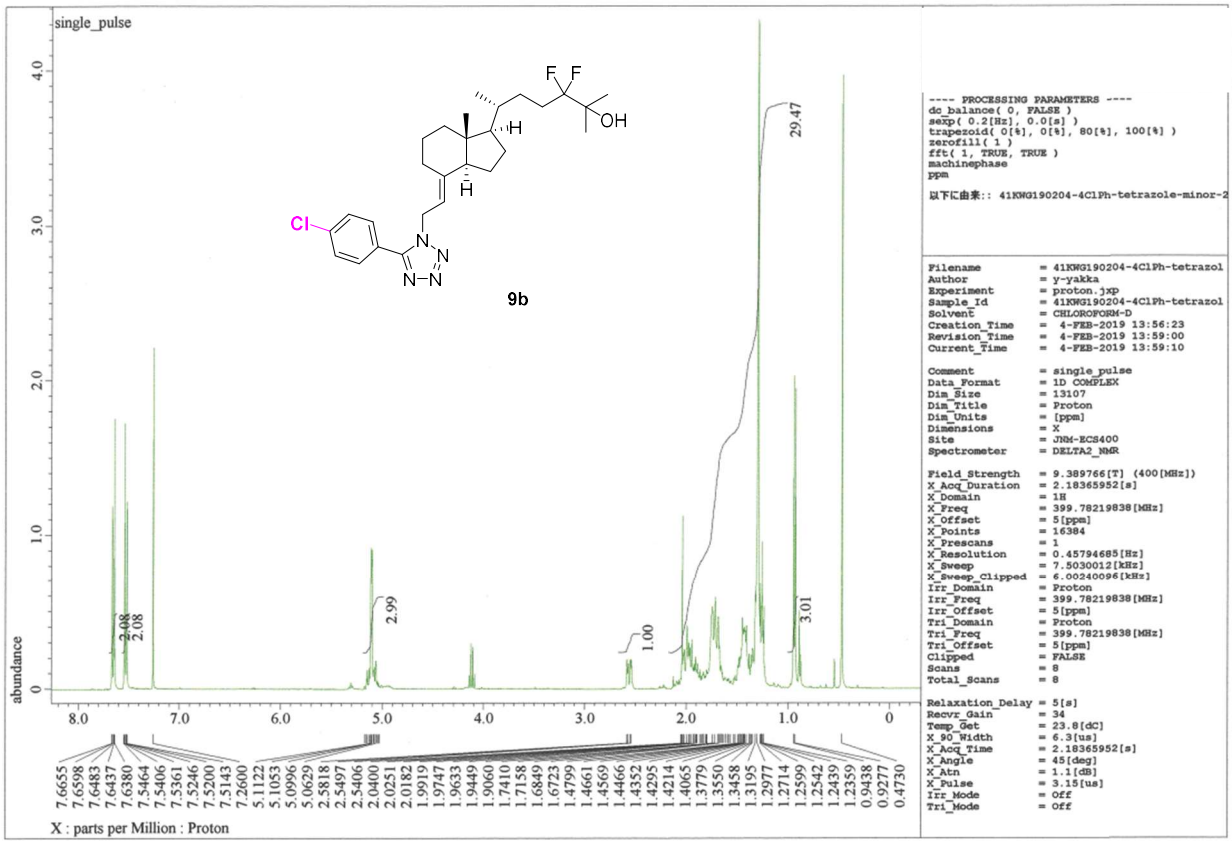

single pulse decoupled gated NOE
8a
abundance
X : parts per Million : Carbon13
Filename = 41KWG190130-1_Carbon-1-3.j
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190130-1
Solvent = CHLOROFORM-D
Creation_Time = 30-JAN-2019 19:06:50
Revision_Time = 31-JAN-2019 09:39:55
Current_Time = 31-JAN-2019 09:40:41
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-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 = 9000
Total_Scans = 9000
Relaxation_Delay = 2[s]
Recvr_Gain = 56
Temp_Get = 23[dC]
X_90_Width = 11.3[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.76666667[us]
Irr_Atn_Dec = 21.5[dB]
Irr_Atn_Noe = 21.5[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]

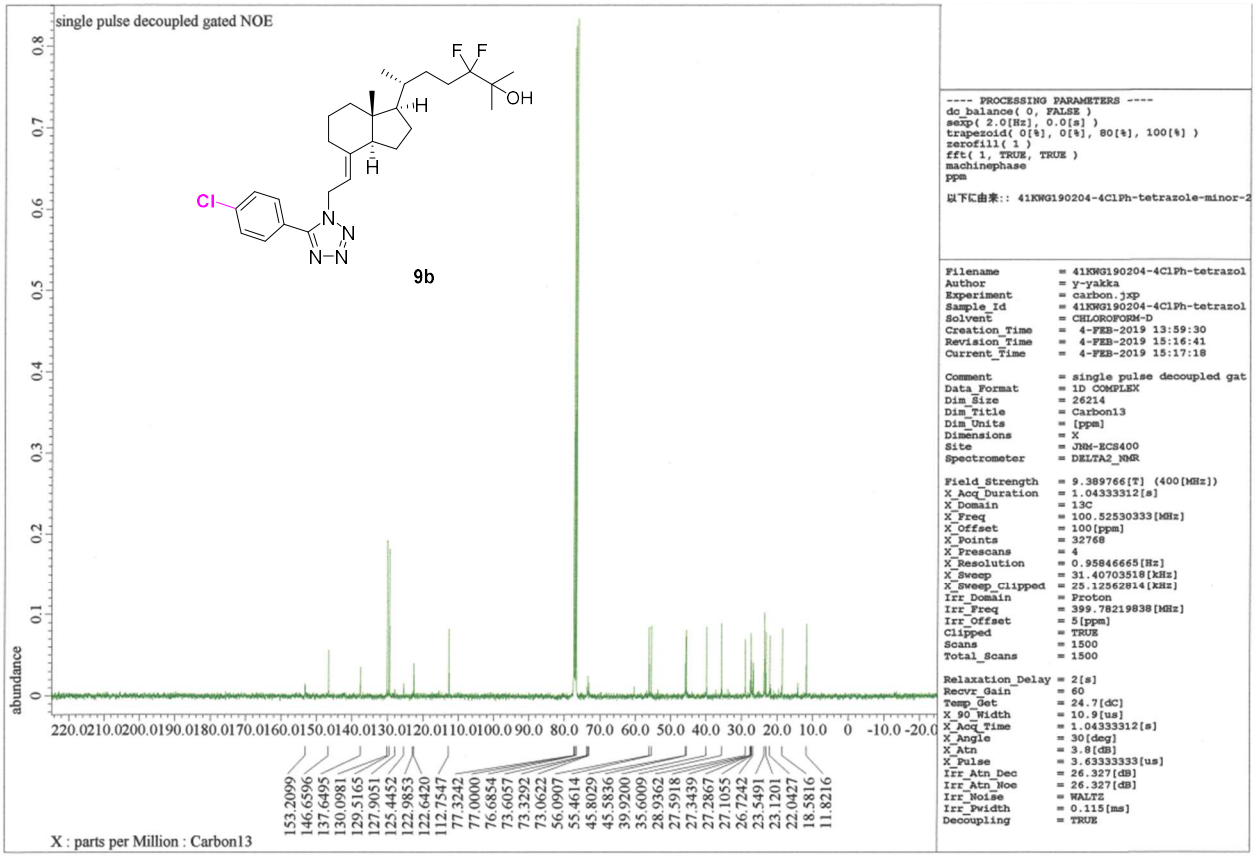

single_pulse
F F
OH
H
H
F3C
N
N
N
N
8b
abundance
X : parts per Million : Proton
4.08
3.14
1.00
26.98
2.94
2.93
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: 41KWG190131-minor_Proton-1-1.jdf
Filename = 41KWG190131-minor_Proton-1
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190131-minor
Solvent = CHLOROFORM-D
Creation_Time = 31-JAN-2019 17:07:43
Revision_Time = 31-JAN-2019 17:12:25
Current_Time = 31-JAN-2019 17:12:57
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 32
Temp_Get = 24.2[dC]
X_90_Width = 6.3[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.1[dB]
X_Pulse = 3.15[us]
Irr_Mode = Off
Tri_Mode = Off

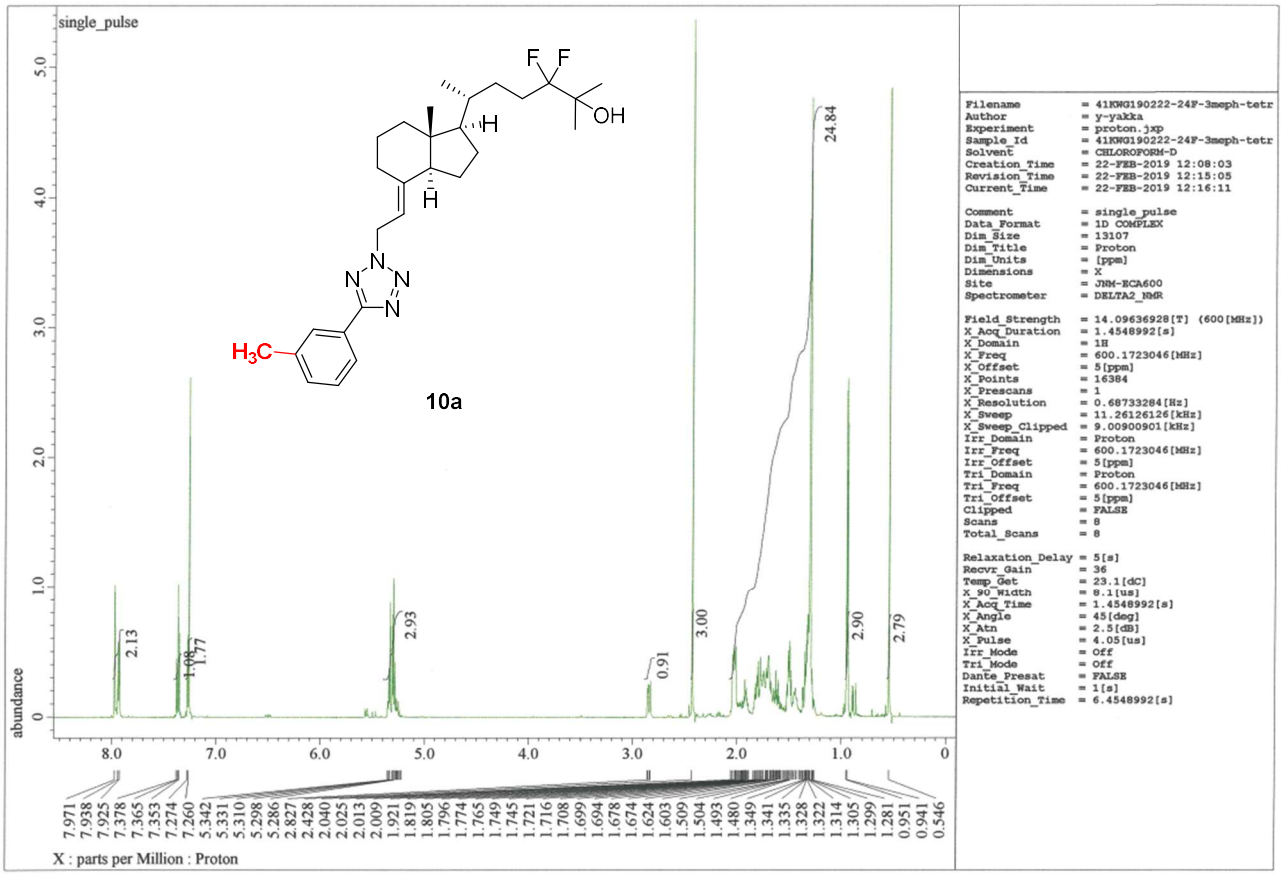

single pulse decoupled gated NOE
F F
OH
H
H
F3C
N
N
N
N
8b
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: 41KWG190131-minor_Carbon-1-1.jdf
Filename = 41KWG190131-minor_Carbon-1
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190131-minor
Solvent = CHLOROFORM-D
Creation_Time = 31-JAN-2019 17:11:51
Revision_Time = 1-FEB-2019 09:11:51
Current_Time = 1-FEB-2019 09:17:19
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 16000
Total_Scans = 16000
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 24.3[dC]
X_90_Width = 10.9[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.63333333[us]
Irr_Atn_Dec = 26.327[dB]
Irr_Atn_Noe = 26.327[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE

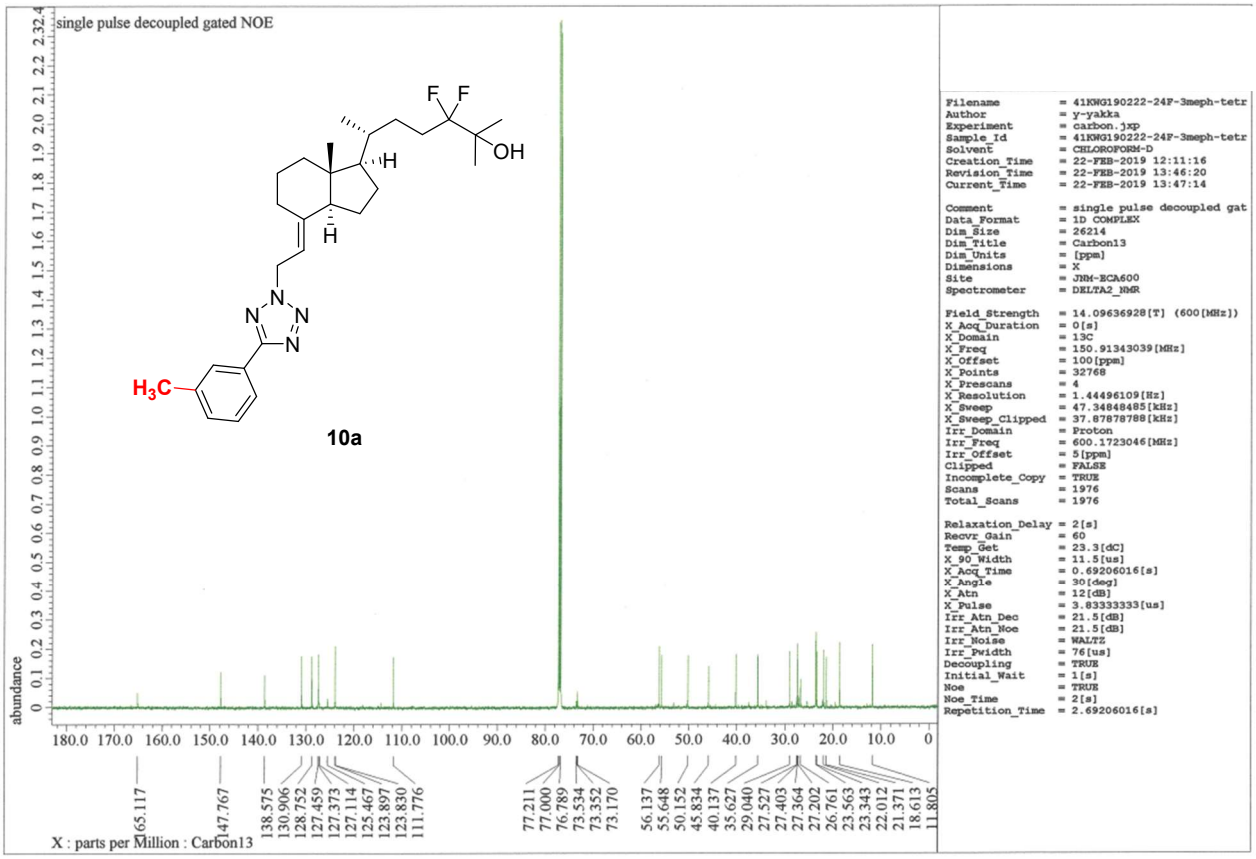

single_pulse
9a
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: 41KWG190201-24F-tetrazole-4ClPh-maj
Filename = 41KWG190201-24F-tetrazole-
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190201-24F-tetrazole-
Solvent = CHLOROFORM-D
Creation_Time = 1-FEB-2019 15:05:37
Revision_Time = 1-FEB-2019 15:10:07
Current_Time = 1-FEB-2019 15:10:22
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 26
Temp_Get = 23.4[dC]
X_90_Width = 6.3[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.1[dB]
X_Pulse = 3.15[us]
Irr_Mode = Off
Tri_Mode = Off

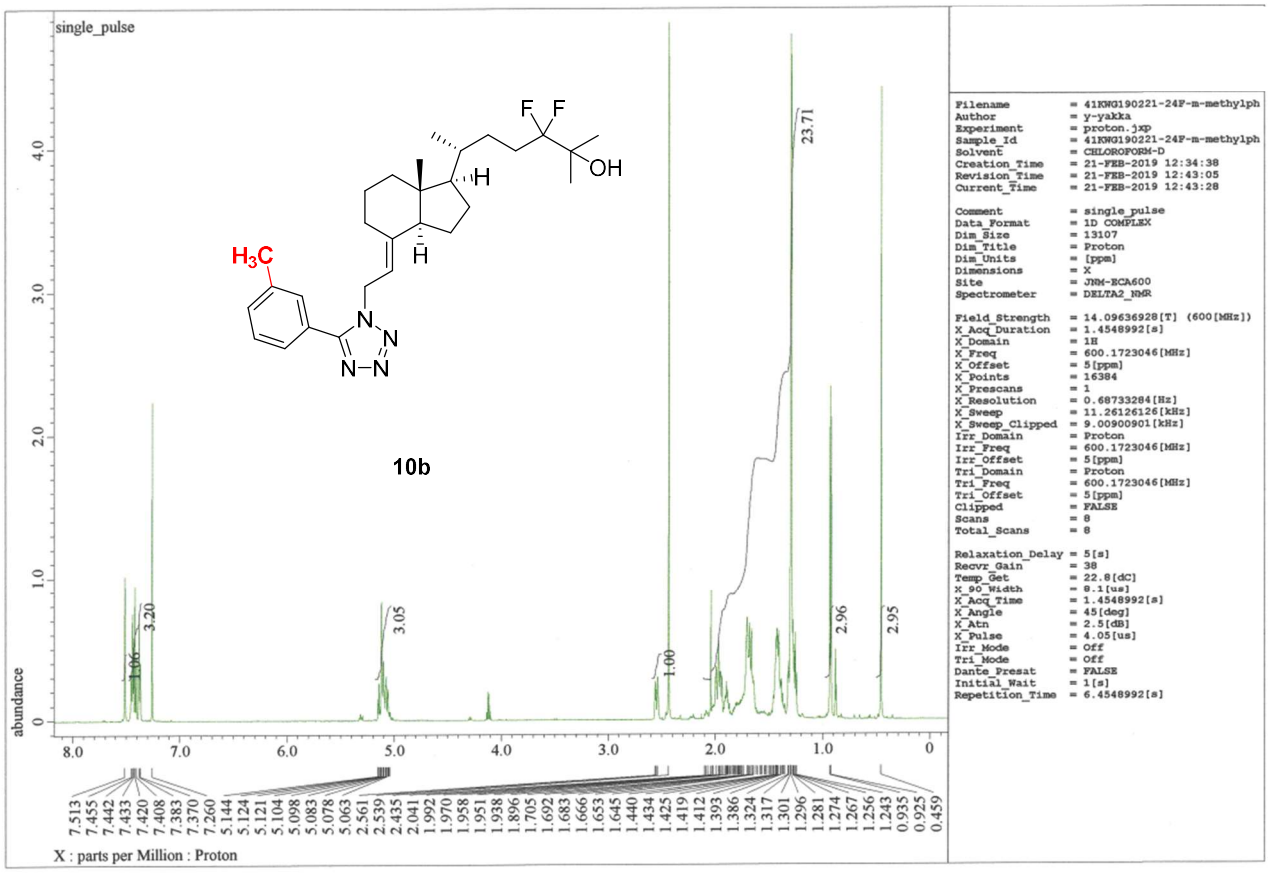

single pulse decoupled gated !
9a
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: 41KWG190201-24F-tetrazole-4ClPh-maj
Filename = 41KWG190201-24F-tetrazole-
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190201-24F-tetrazole-
Solvent = CHLOROFORM-D
Creation_Time = 1-FEB-2019 15:08:46
Revision_Time = 1-FEB-2019 15:46:12
Current_Time = 1-FEB-2019 15:47:02
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 0[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Incomplete_Copy = TRUE
Scans = 658
Total_Scans = 658
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 24.2[dC]
X_90_Width = 10.9[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.63333333[us]
Irr_Atn_Dec = 26.327[dB]
Irr_Atn_Noe = 26.327[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]


single_pulse
F F
OH
H
H
Cl
N
N
N
N
9b
29.47
2.99
1.00
3.01
2.08
2.08
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: 41KWG190204-4ClPh-tetrazole-minor-2
Filename = 41KWG190204-4ClPh-tetrazol
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190204-4ClPh-tetrazol
Solvent = CHLOROFORM-D
Creation_Time = 4-FEB-2019 13:56:23
Revision_Time = 4-FEB-2019 13:59:00
Current_Time = 4-FEB-2019 13:59:10
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 23.8[dC]
X_90_Width = 6.3[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.1[dB]
X_Pulse = 3.15[us]
Irr_Mode = Off
Tri_Mode = Off

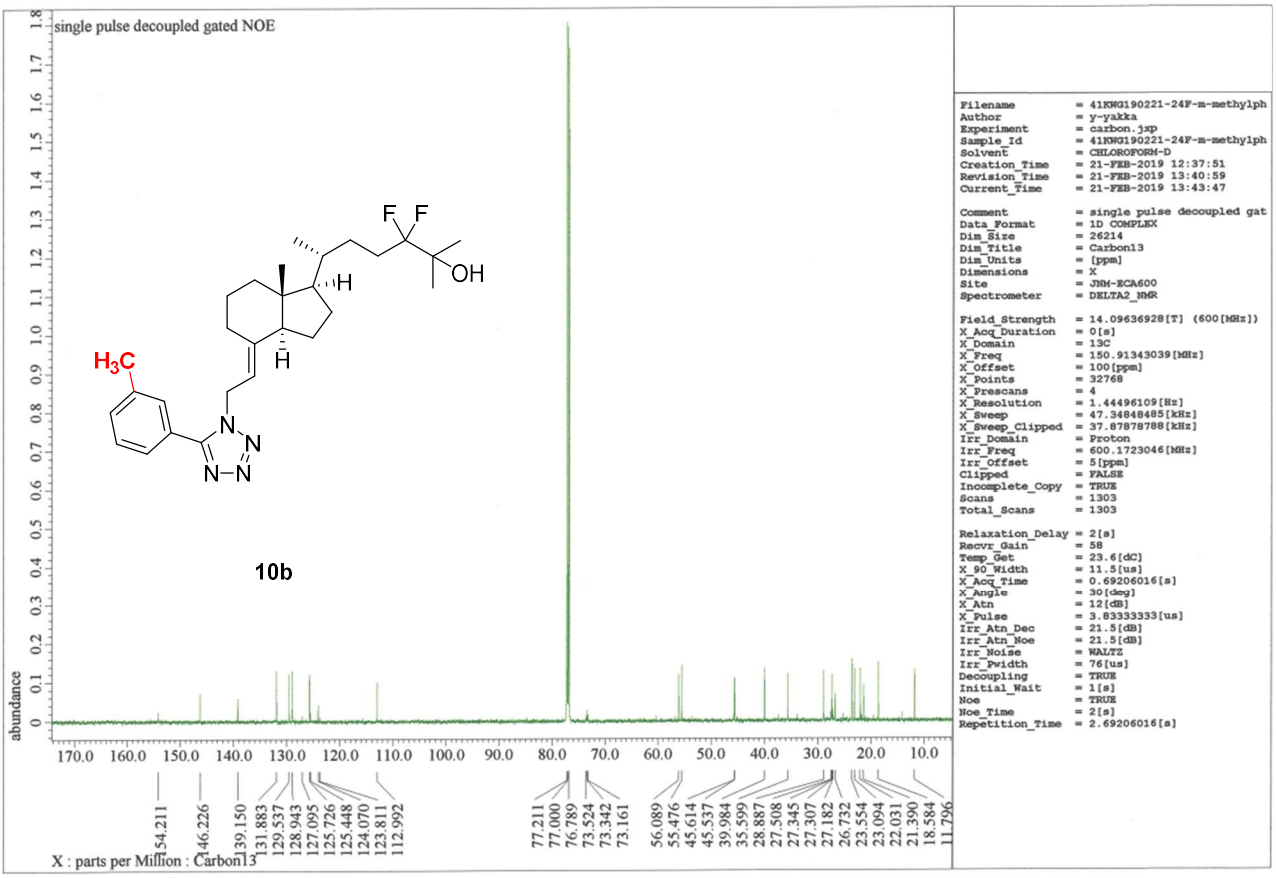

single pulse decoupled gated NOE
F F
OH
H
H
Cl
N
N
N
N
9b
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: 41KWG190204-4ClPh-tetrazole-minor-2
Filename = 41KWG190204-4ClPh-tetrazol
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190204-4ClPh-tetrazol
Solvent = CHLOROFORM-D
Creation_Time = 4-FEB-2019 13:59:30
Revision_Time = 4-FEB-2019 15:16:41
Current_Time = 4-FEB-2019 15:17:18
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 1500
Total_Scans = 1500
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 24.7[dC]
X_90_Width = 10.9[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 3.8[dB]
X_Pulse = 3.63333333[us]
Irr_Atn_Dec = 26.327[dB]
Irr_Atn_Noe = 26.327[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE


single_pulse
F F
OH
H
H
N
N
N
N
H3C
10a
abundance
2.13
1.08
1.77
2.93
0.91
3.00
24.84
2.90
2.79
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.971 7.938 7.925 7.378 7.365 7.353 7.274 7.260 5.342 5.331 5.310 5.298 5.286 5.827 2.428 2.040 2.025 2.013 2.009 1.921 1.819 1.805 1.796 1.774 1.765 1.749 1.745 1.721 1.716 1.708 1.699 1.694 1.678 1.674 1.624 1.603 1.509 1.504 1.493 1.480 1.349 1.341 1.335 1.328 1.322 1.314 1.305 1.299 1.281 0.951 0.941 0.546
X : parts per Million : Proton
Filename = 41KWG190222-24F-3meph-tetr
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190222-24F-3meph-tetr
Solvent = CHLOROFORM-D
Creation_Time = 22-FEB-2019 12:08:03
Revision_Time = 22-FEB-2019 12:15:05
Current_Time = 22-FEB-2019 12:16:11
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 1.4548992[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.68733284[Hz]
X_Sweep = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[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 = 5[s]
Recvr_Gain = 36
Temp_Get = 23.1[dC]
X_90_Width = 8.1[us]
X_Acq_Time = 1.4548992[s]
X_Angle = 45[deg]
X_Atn = 2.5[dB]
X_Pulse = 4.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.4548992[s]

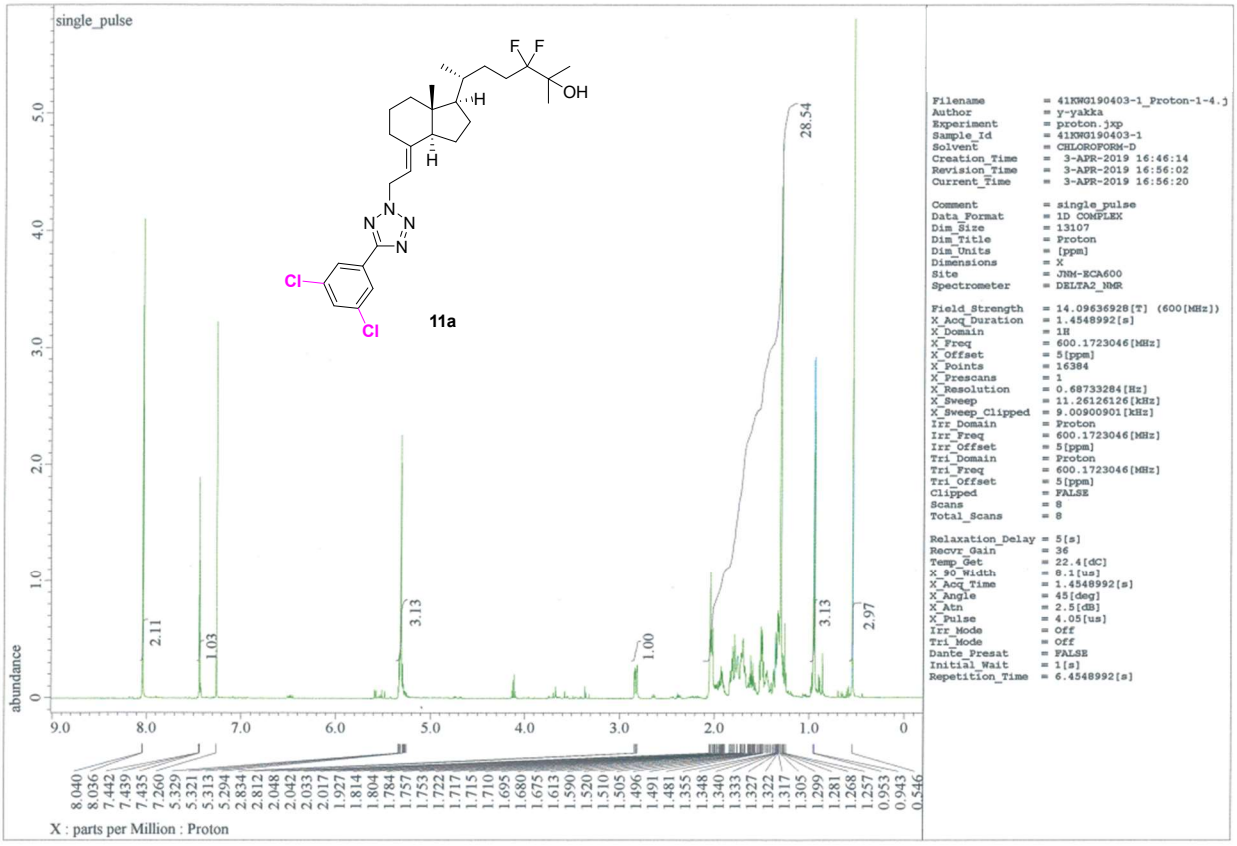

single pulse decoupled gated NOE
F F
OH
H
H
N
N
N
N
H3C
10a
abundance
180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
165.117 147.767 138.575 130.906 128.752 127.459 127.373 127.114 125.467 123.897 123.830 111.776 77.211 77.000 76.789 73.534 73.352 73.170 56.137 55.648 50.152 45.834 40.137 35.627 29.040 27.527 27.403 27.364 27.202 26.761 23.563 23.343 22.012 21.371 18.613 11.805
X : parts per Million : Carbon13
Filename = 41KWG190222-24F-3meph-tetr
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190222-24F-3meph-tetr
Solvent = CHLOROFORM-D
Creation_Time = 22-FEB-2019 12:11:16
Revision_Time = 22-FEB-2019 13:46:20
Current_Time = 22-FEB-2019 13:47:14
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 0[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
Incomplete_Copy = TRUE
Scans = 1976
Total_Scans = 1976
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.3[dC]
X_90_Width = 11.5[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.83333333[us]
Irr_Atn_Dec = 21.5[dB]
Irr_Atn_Noe = 21.5[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]

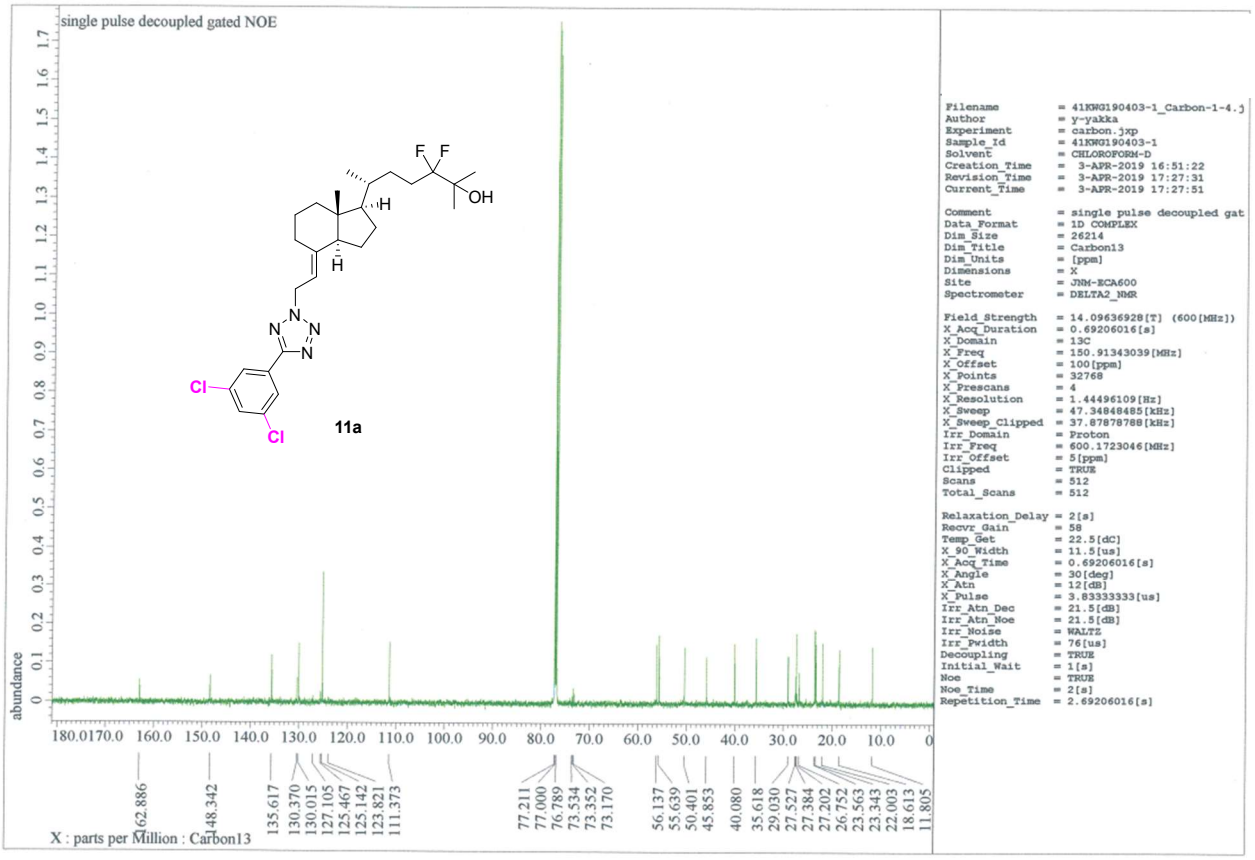


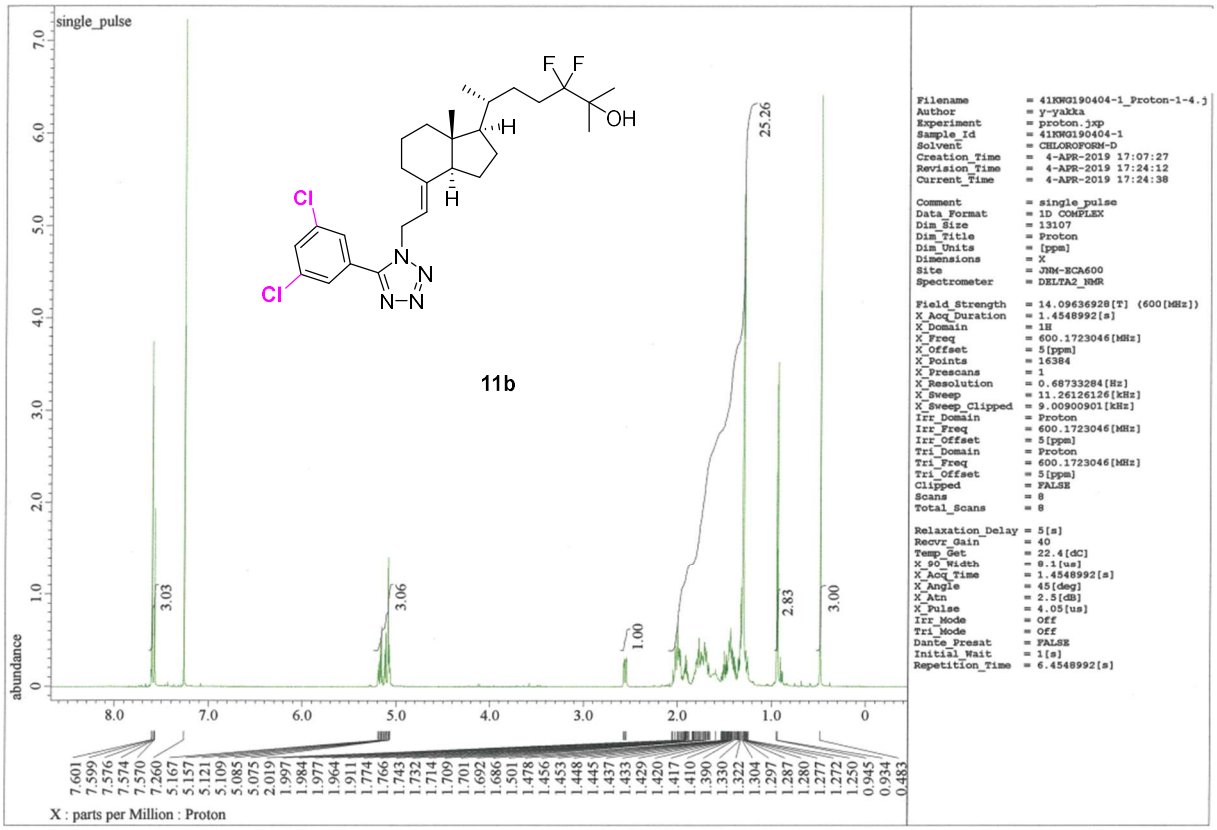

```
Filename        = 41KWG190221-24F-m-methylph
Author          = y-yakka
Experiment      = proton.jxp
Sample_Id       = 41KWG190221-24F-m-methylph
Solvent         = CHLOROFORM-D
Creation_Time   = 21-FEB-2019 12:34:38
Revision_Time   = 21-FEB-2019 12:43:05
Current_Time    = 21-FEB-2019 12:43:28

Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 13107
Dim_Title       = Proton
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA600
Spectrometer    = DELTA2_NMR

Field_Strength  = 14.09636928[T] (600[MHz])
X_Acq_Duration  = 1.4548992[s]
X_Domain        = 1H
X_Freq          = 600.1723046[MHz]
X_Offset        = 5[ppm]
X_Points        = 16384
X_Prescans      = 1
X_Resolution    = 0.68733284[Hz]
X_Sweep         = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[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 = 5[s]
Recvr_Gain      = 38
Temp_Get        = 22.8[dC]
X_90_Width      = 8.1[us]
X_Acq_Time      = 1.4548992[s]
X_Angle         = 45[deg]
X_Atn           = 2.5[dB]
X_Pulse         = 4.05[us]
Irr_Mode        = Off
Tri_Mode        = Off
Dante_Presat    = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 6.4548992[s]
```

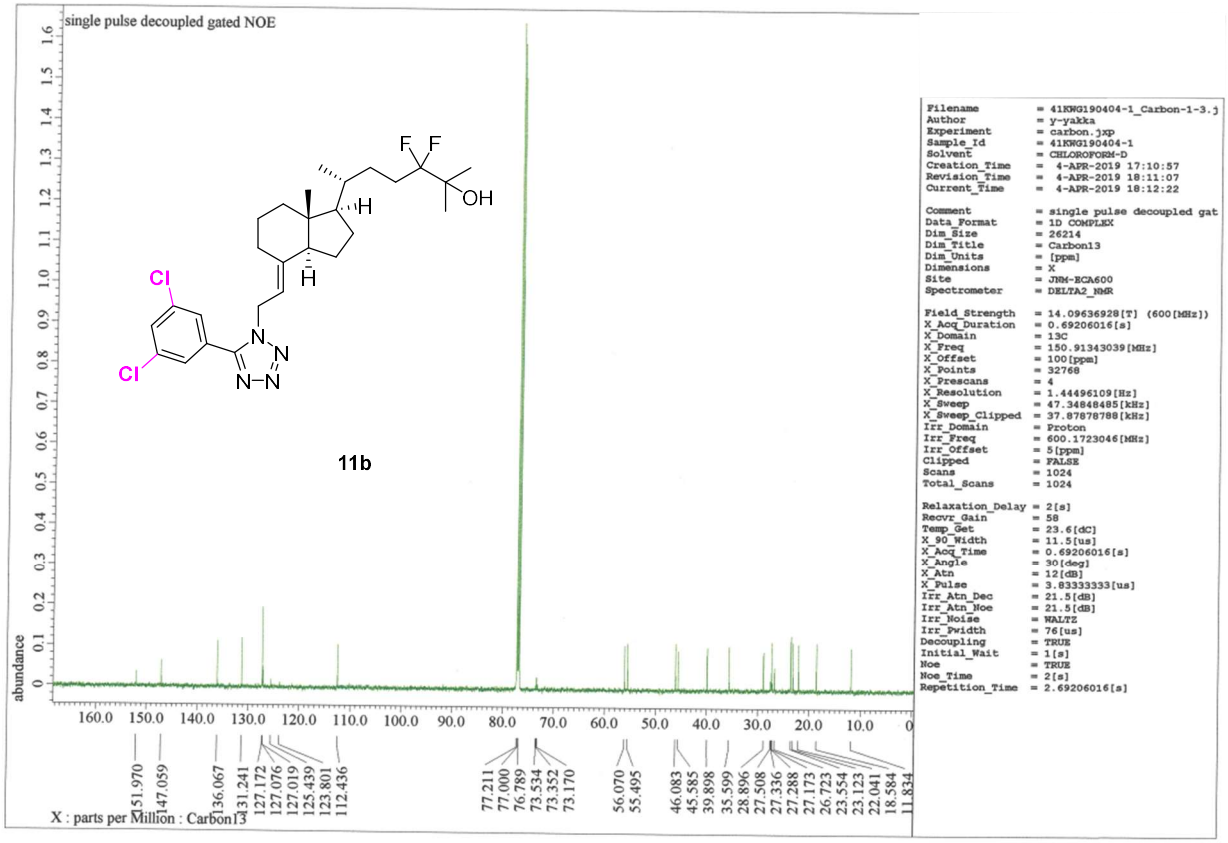


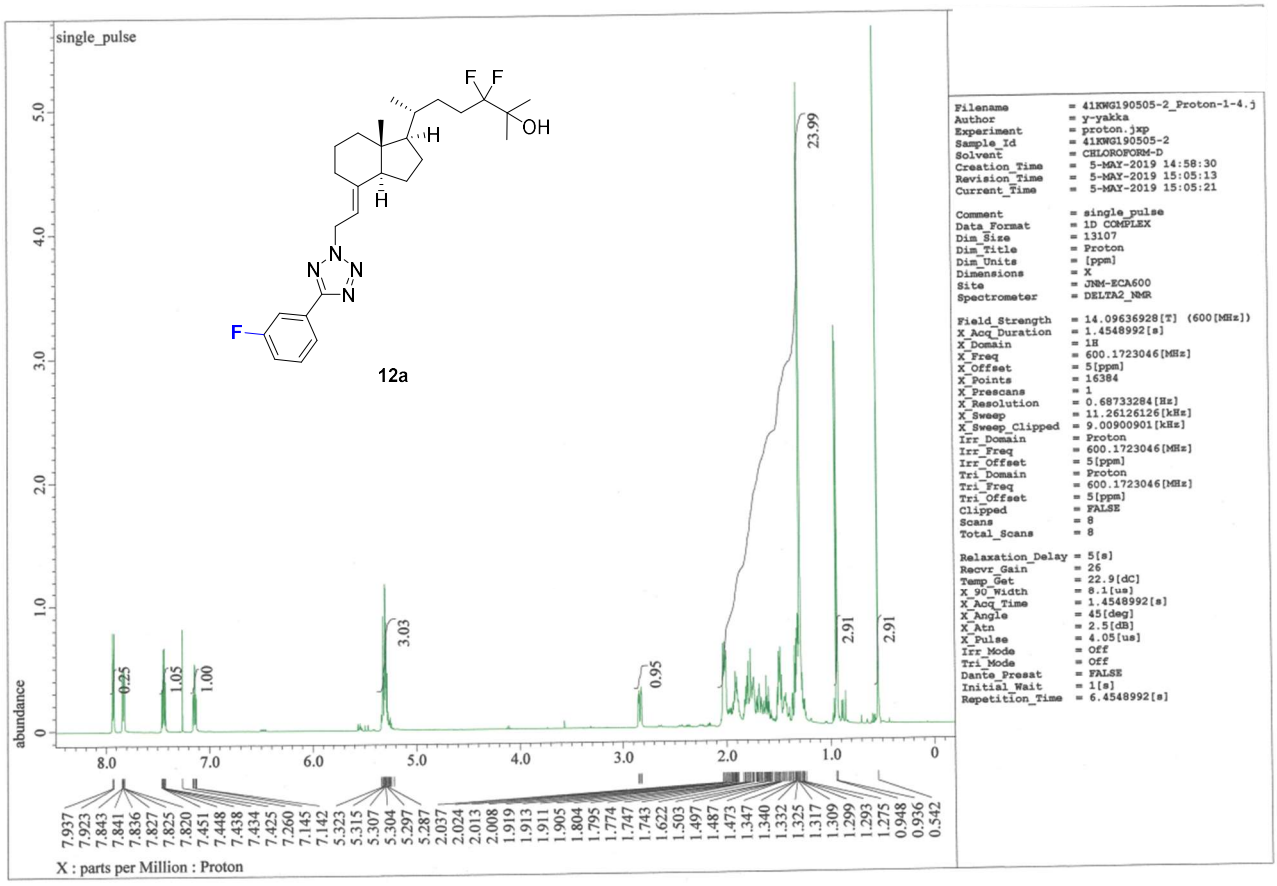

```
Filename        = 41KWG190221-24F-m-methylph
Author          = y-yakka
Experiment      = carbon.jxp
Sample_Id       = 41KWG190221-24F-m-methylph
Solvent         = CHLOROFORM-D
Creation_Time   = 21-FEB-2019 12:37:51
Revision_Time   = 21-FEB-2019 13:40:59
Current_Time    = 21-FEB-2019 13:43:47

Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
Dim_Title       = Carbon13
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA600
Spectrometer    = DELTA2_NMR

Field_Strength  = 14.09636928[T] (600[MHz])
X_Acq_Duration  = 0[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
Incomplete_Copy = TRUE
Scans           = 1303
Total_Scans     = 1303

Relaxation_Delay = 2[s]
Recvr_Gain      = 58
Temp_Get        = 23.6[dC]
X_90_Width      = 11.5[us]
X_Acq_Time      = 0.69206016[s]
X_Angle         = 30[deg]
X_Atn           = 12[dB]
X_Pulse         = 3.83333333[us]
Irr_Atn_Dec     = 21.5[dB]
Irr_Atn_Noe     = 21.5[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]
```

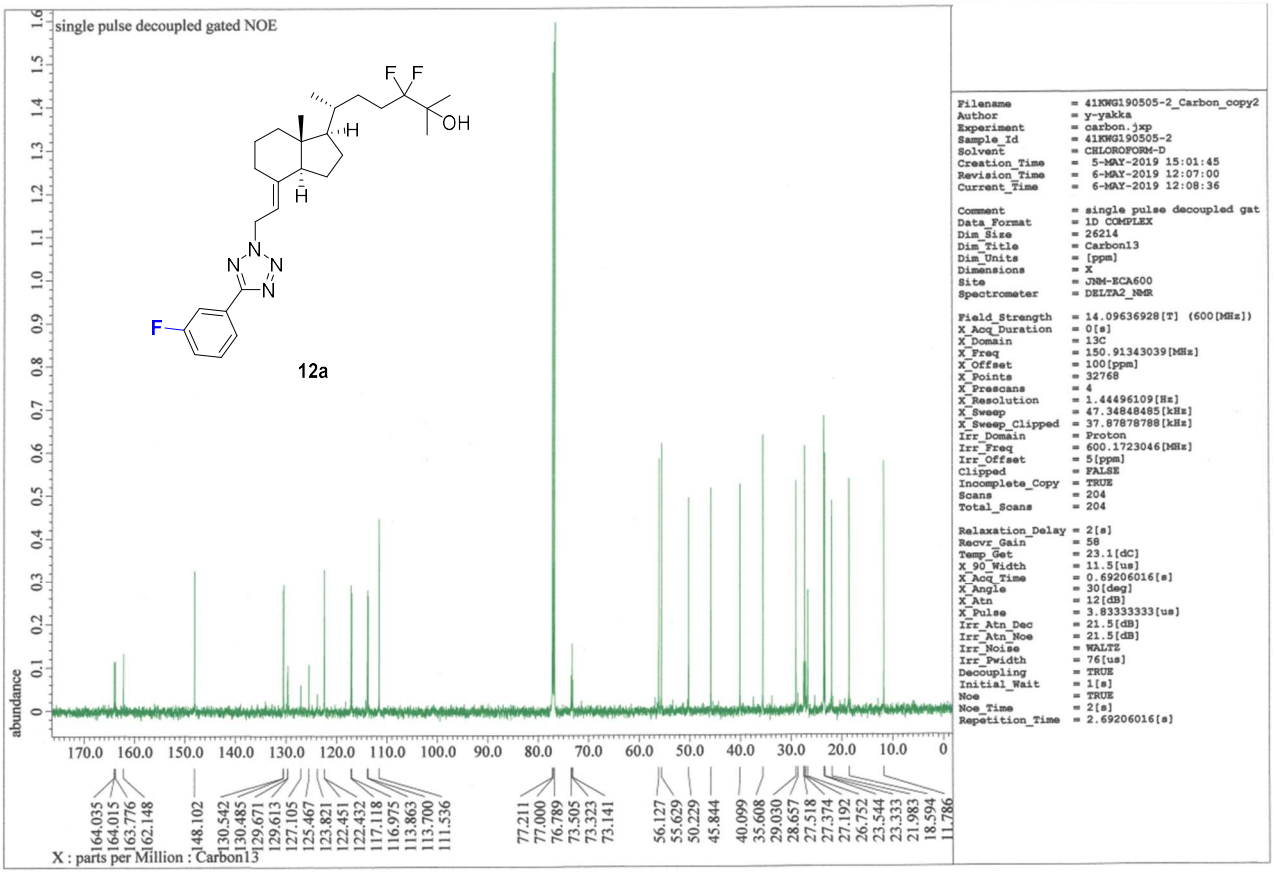

single_pulse
F F
OH
H
H
N
N
N
Cl
Cl
11a
28.54
2.11
1.03
3.13
1.00
3.13
2.97
abundance
9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
X : parts per Million : Proton
Filename = 41KWG190403-1_Proton-1-4.j
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190403-1
Solvent = CHLOROFORM-D
Creation_Time = 3-APR-2019 16:46:14
Revision_Time = 3-APR-2019 16:56:02
Current_Time = 3-APR-2019 16:56:20
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 1.4548992[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.68733284[Hz]
X_Sweep = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[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 = 5[s]
Recvr_Gain = 36
Temp_Get = 22.4[dC]
X_90_Width = 8.1[us]
X_Acq_Time = 1.4548992[s]
X_Angle = 45[deg]
X_Atn = 2.5[dB]
X_Pulse = 4.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.4548992[s]

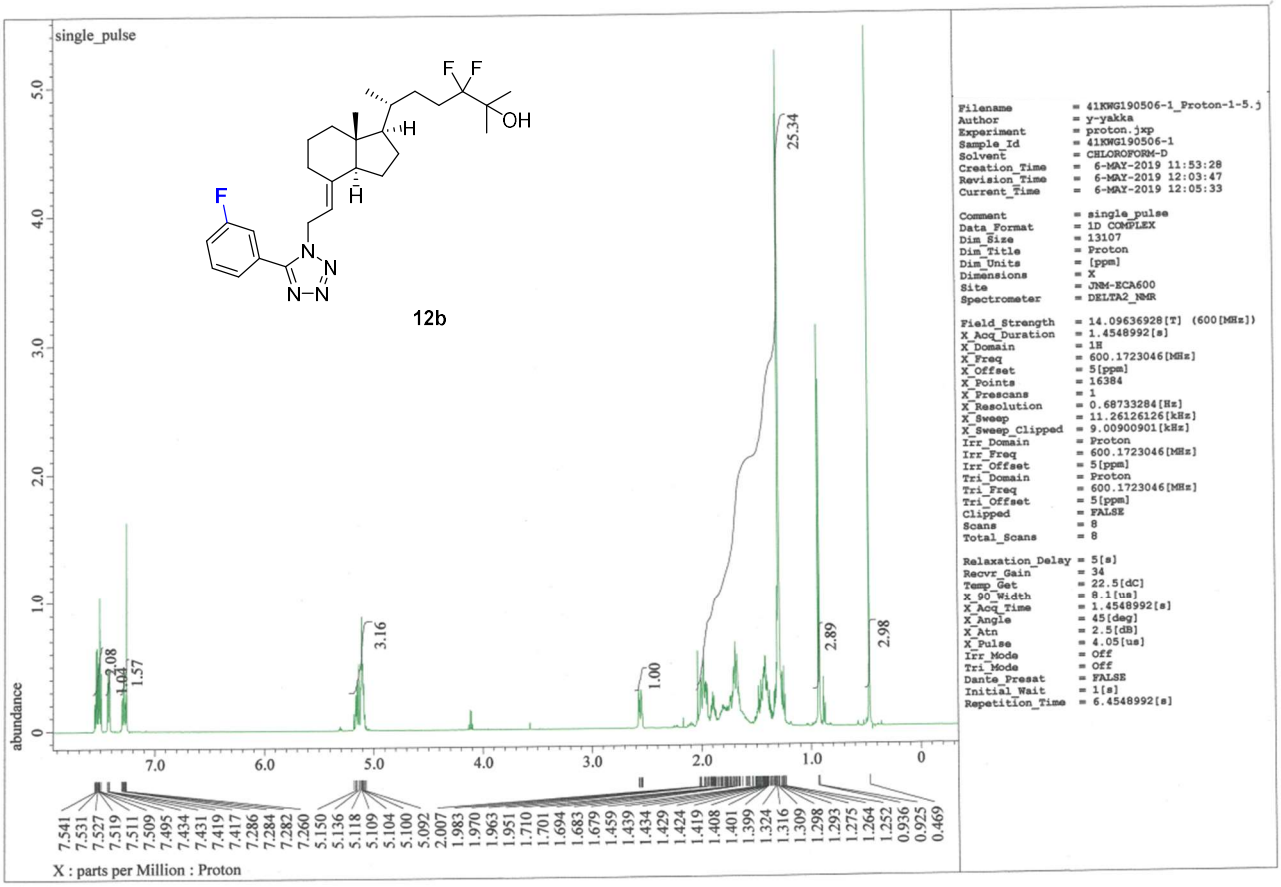

single pulse decoupled gated NOE
F F
OH
H
H
N
N
N
Cl
Cl
11a
abundance
180.0170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
X : parts per Million : Carbon13
Filename = 41KWG190403-1_Carbon-1-4.j
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190403-1
Solvent = CHLOROFORM-D
Creation_Time = 3-APR-2019 16:51:22
Revision_Time = 3-APR-2019 17:27:31
Current_Time = 3-APR-2019 17:27:51
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-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 = 512
Total_Scans = 512
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 22.5[dC]
X_90_Width = 11.5[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.83333333[us]
Irr_Atn_Dec = 21.5[dB]
Irr_Atn_Noe = 21.5[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]

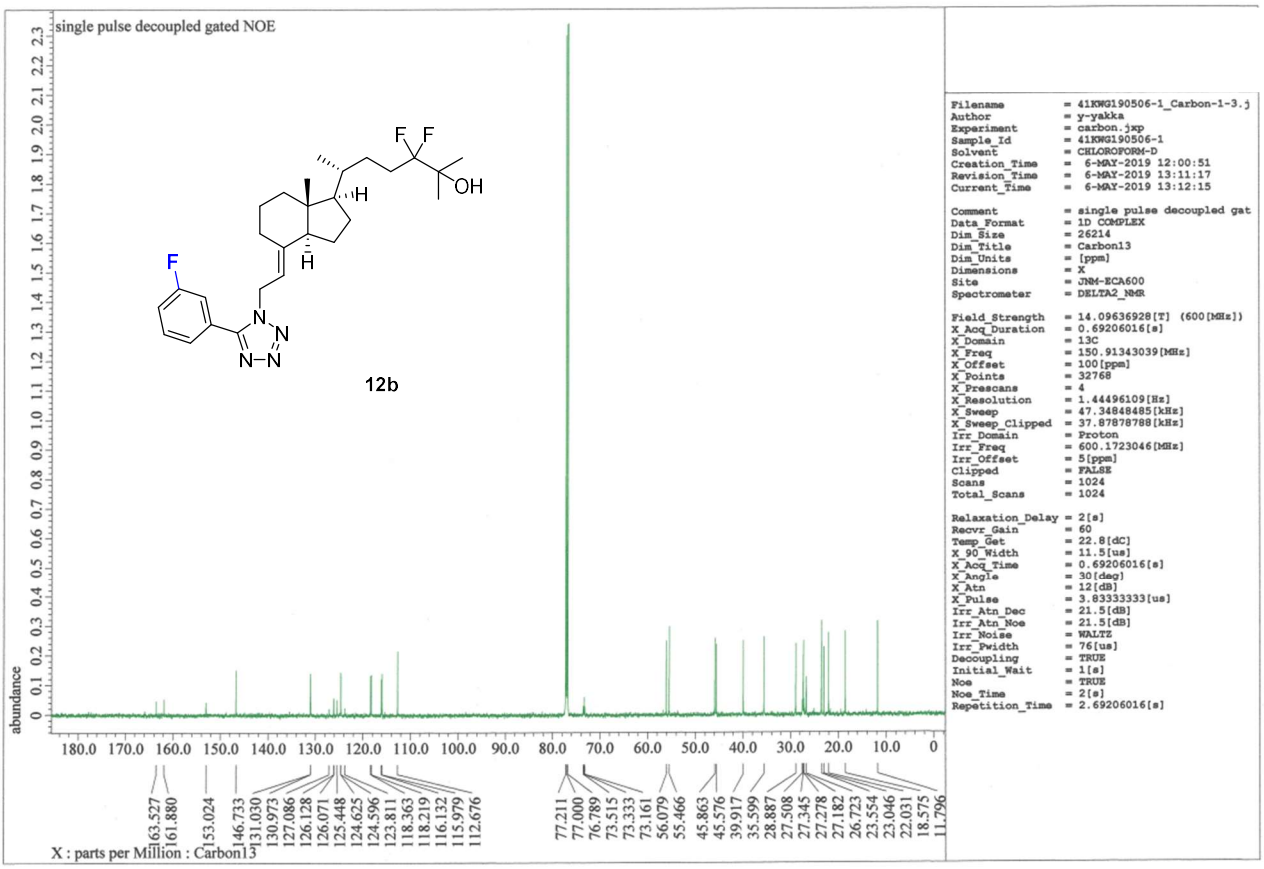

single_pulse
F F
OH
H
H
Cl
Cl
N
N
N
N
11b
abundance
3.03
3.06
1.00
25.26
2.83
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.601 7.599 7.576 7.574 7.570 7.260 5.167 5.157 5.121 5.109 5.085 5.075 2.019 1.997 1.984 1.977 1.964 1.911 1.774 1.766 1.743 1.732 1.714 1.709 1.701 1.692 1.686 1.501 1.478 1.456 1.453 1.448 1.445 1.437 1.433 1.429 1.420 1.417 1.410 1.390 1.330 1.322 1.304 1.297 1.287 1.280 1.277 1.272 1.250 0.945 0.934 0.483
X : parts per Million : Proton
Filename = 41KWG190404-1_Proton-1-4.j
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190404-1
Solvent = CHLOROFORM-D
Creation_Time = 4-APR-2019 17:07:27
Revision_Time = 4-APR-2019 17:24:12
Current_Time = 4-APR-2019 17:24:38
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 1.4548992[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.68733284[Hz]
X_Sweep = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[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 = 5[s]
Recvr_Gain = 40
Temp_Get = 22.4[dC]
X_90_Width = 8.1[us]
X_Acq_Time = 1.4548992[s]
X_Angle = 45[deg]
X_Atn = 2.5[dB]
X_Pulse = 4.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.4548992[s]

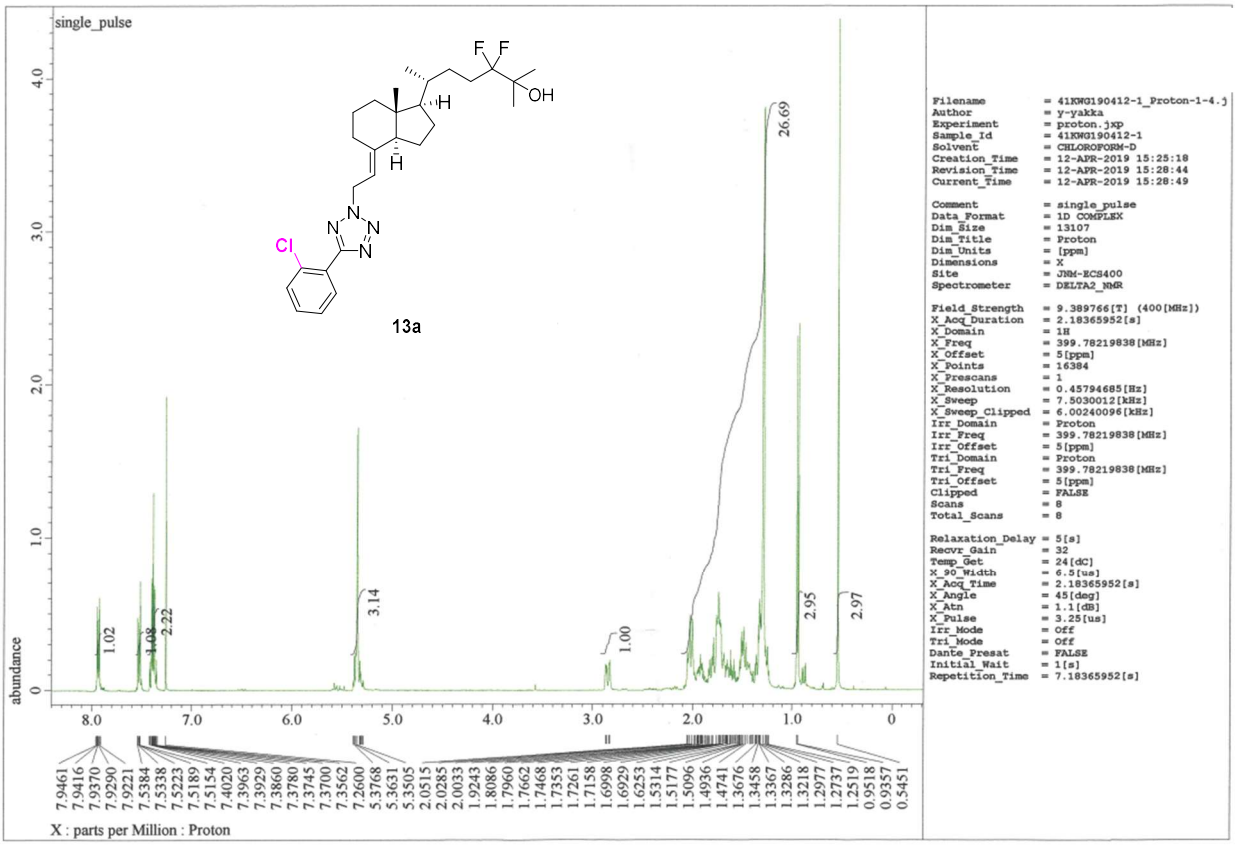

single pulse decoupled gated NOE
F F
OH
H
H
Cl
Cl
N
N
N
N
11b
abundance
160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
151.970 147.059 136.067 131.241 127.172 127.076 127.019 125.439 123.801 112.436
77.211 77.000 76.789 73.534 73.352 73.170 56.070 55.495 46.083 45.585 39.898 35.599 28.896 27.508 27.336 27.288 27.173 26.723 23.554 23.123 22.041 18.584 11.834
X : parts per Million : Carbon13
Filename = 41KWG190404-1_Carbon-1-3.j
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190404-1
Solvent = CHLOROFORM-D
Creation_Time = 4-APR-2019 17:10:57
Revision_Time = 4-APR-2019 18:11:07
Current_Time = 4-APR-2019 18:12:22
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-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 = 1024
Total_Scans = 1024
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 23.6[dC]
X_90_Width = 11.5[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.83333333[us]
Irr_Atn_Dec = 21.5[dB]
Irr_Atn_Noe = 21.5[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]

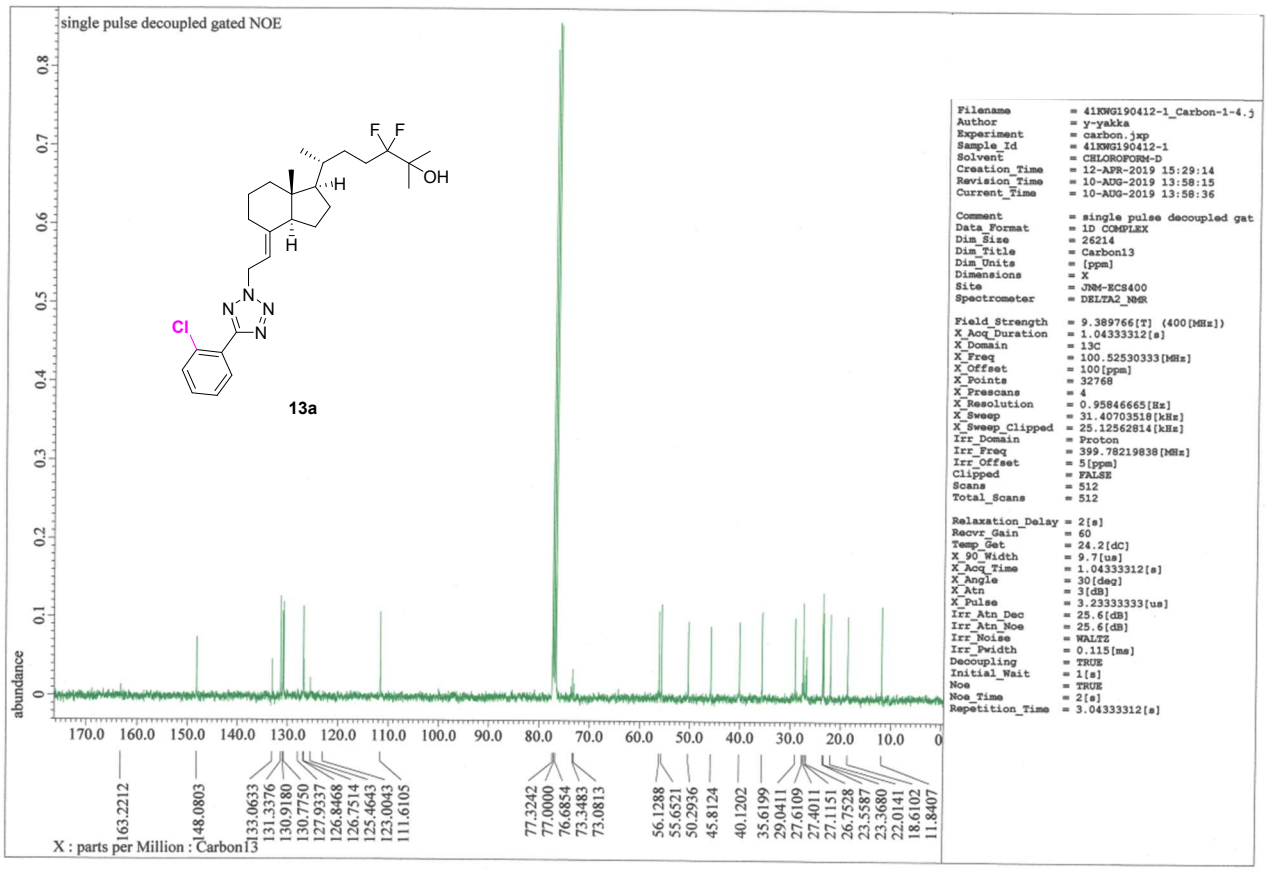

single_pulse
F F
OH
H
H
N
N
N
N
F
12a
23.99
3.03
2.91
2.91
0.95
1.05
1.00
0.25
abundance
X : parts per Million : Proton
Filename = 41KWG190505-2_Proton-1-4.j
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190505-2
Solvent = CHLOROFORM-D
Creation_Time = 5-MAY-2019 14:58:30
Revision_Time = 5-MAY-2019 15:05:13
Current_Time = 5-MAY-2019 15:05:21
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 1.4548992[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.68733284[Hz]
X_Sweep = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[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 = 5[s]
Recvr_Gain = 26
Temp_Get = 22.9[dC]
X_90_Width = 8.1[us]
X_Acq_Time = 1.4548992[s]
X_Angle = 45[deg]
X_Atn = 2.5[dB]
X_Pulse = 4.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.4548992[s]

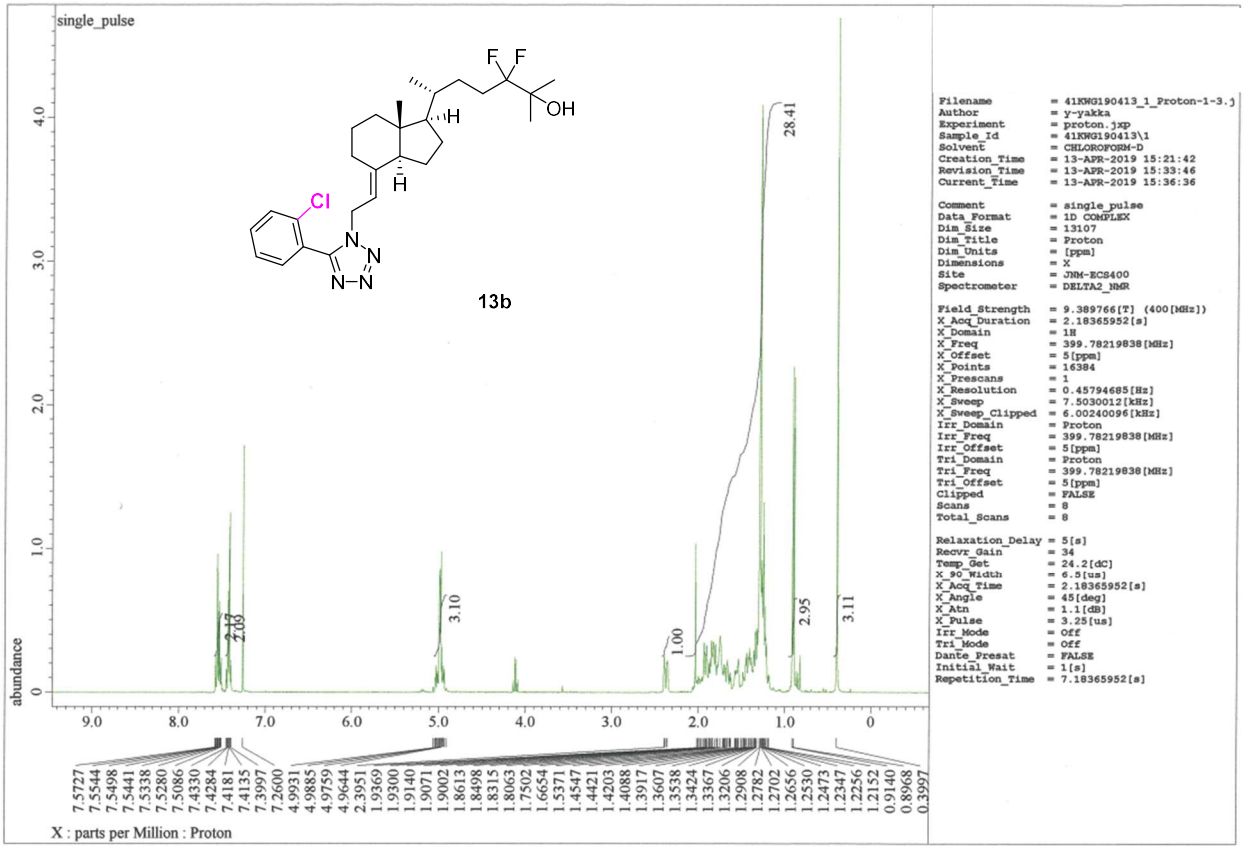

single pulse decoupled gated NOE
F F
OH
H
H
N
N
N
N
F
12a
abundance
X : parts per Million : Carbon13
Filename = 41KWG190505-2_Carbon_copy2
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190505-2
Solvent = CHLOROFORM-D
Creation_Time = 5-MAY-2019 15:01:45
Revision_Time = 6-MAY-2019 12:07:00
Current_Time = 6-MAY-2019 12:08:36
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 0[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
Incomplete_Copy = TRUE
Scans = 204
Total_Scans = 204
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 23.1[dC]
X_90_Width = 11.5[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.83333333[us]
Irr_Atn_Dec = 21.5[dB]
Irr_Atn_Noe = 21.5[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]

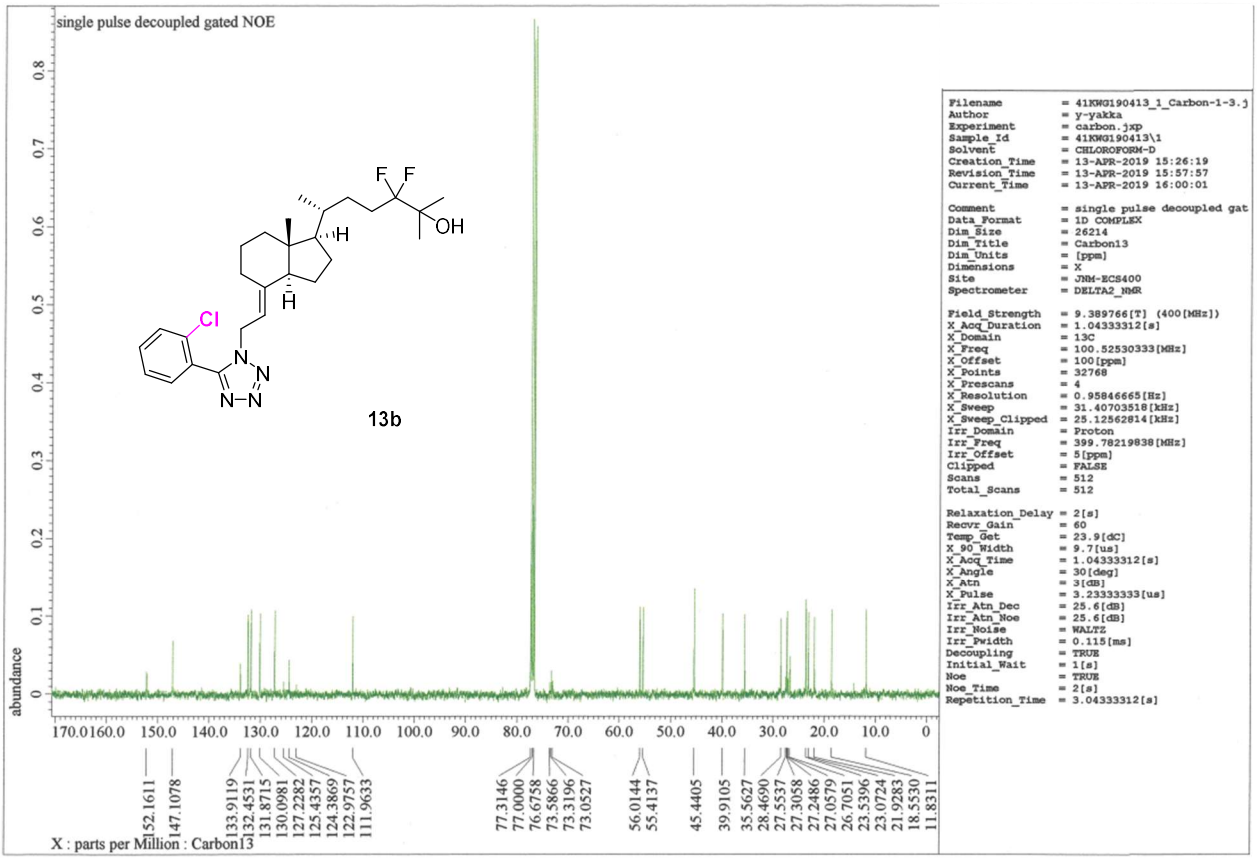

single_pulse
12b
abundance
X : parts per Million : Proton
Filename = 41KWG190506-1_Proton-1-5.j
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190506-1
Solvent = CHLOROFORM-D
Creation_Time = 6-MAY-2019 11:53:28
Revision_Time = 6-MAY-2019 12:03:47
Current_Time = 6-MAY-2019 12:05:33
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 1.4548992[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.68733284[Hz]
X_Sweep = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[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 = 5[s]
Recvr_Gain = 34
Temp_Get = 22.5[dC]
X_90_Width = 8.1[us]
X_Acq_Time = 1.4548992[s]
X_Angle = 45[deg]
X_Atn = 2.5[dB]
X_Pulse = 4.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.4548992[s]

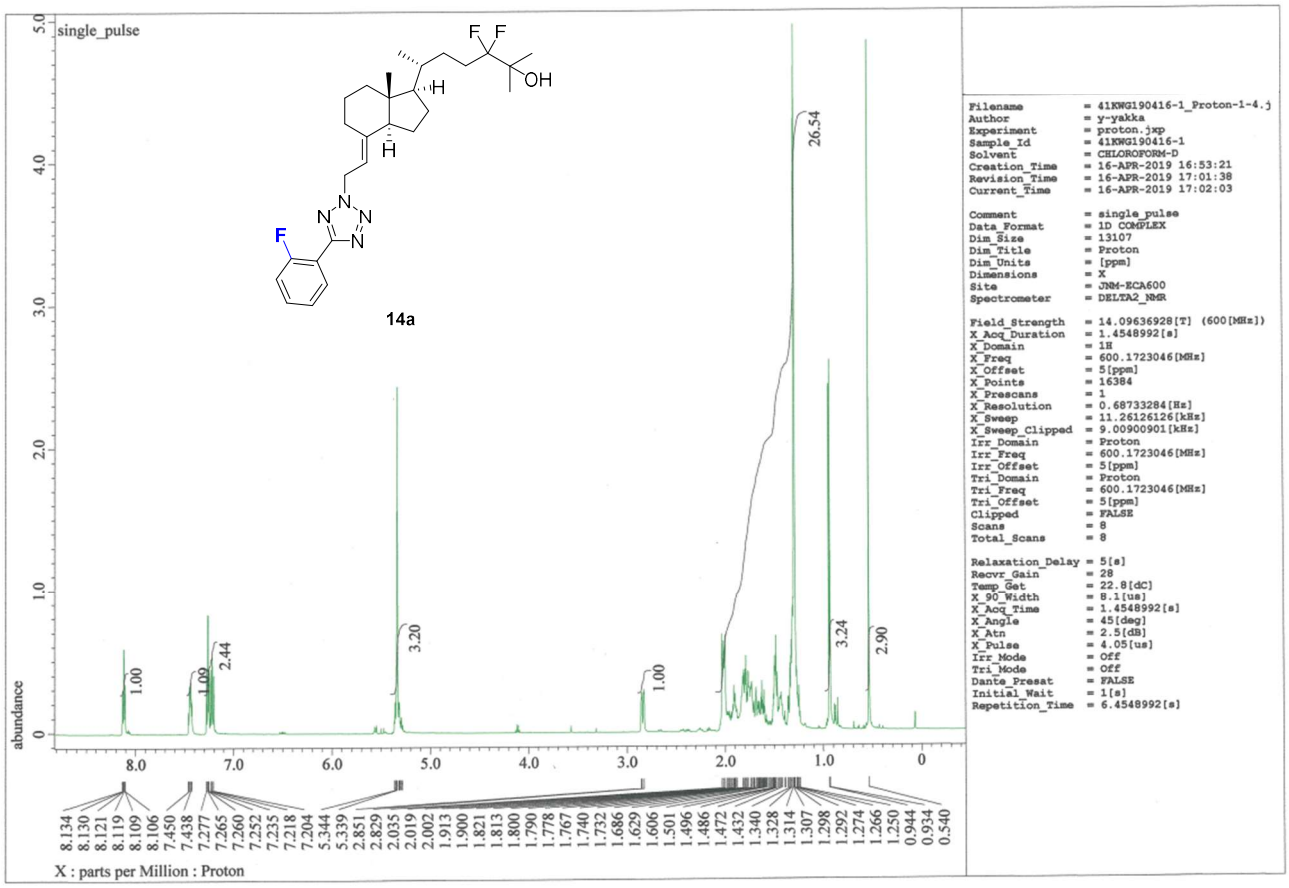

single pulse decoupled gated NOE
12b
abundance
X : parts per Million : Carbon13
Filename = 41KWG190506-1_Carbon-1-3.j
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190506-1
Solvent = CHLOROFORM-D
Creation_Time = 6-MAY-2019 12:00:51
Revision_Time = 6-MAY-2019 13:11:17
Current_Time = 6-MAY-2019 13:12:15
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-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 = 1024
Total_Scans = 1024
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 22.8[dC]
X_90_Width = 11.5[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.83333333[us]
Irr_Atn_Dec = 21.5[dB]
Irr_Atn_Noe = 21.5[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]

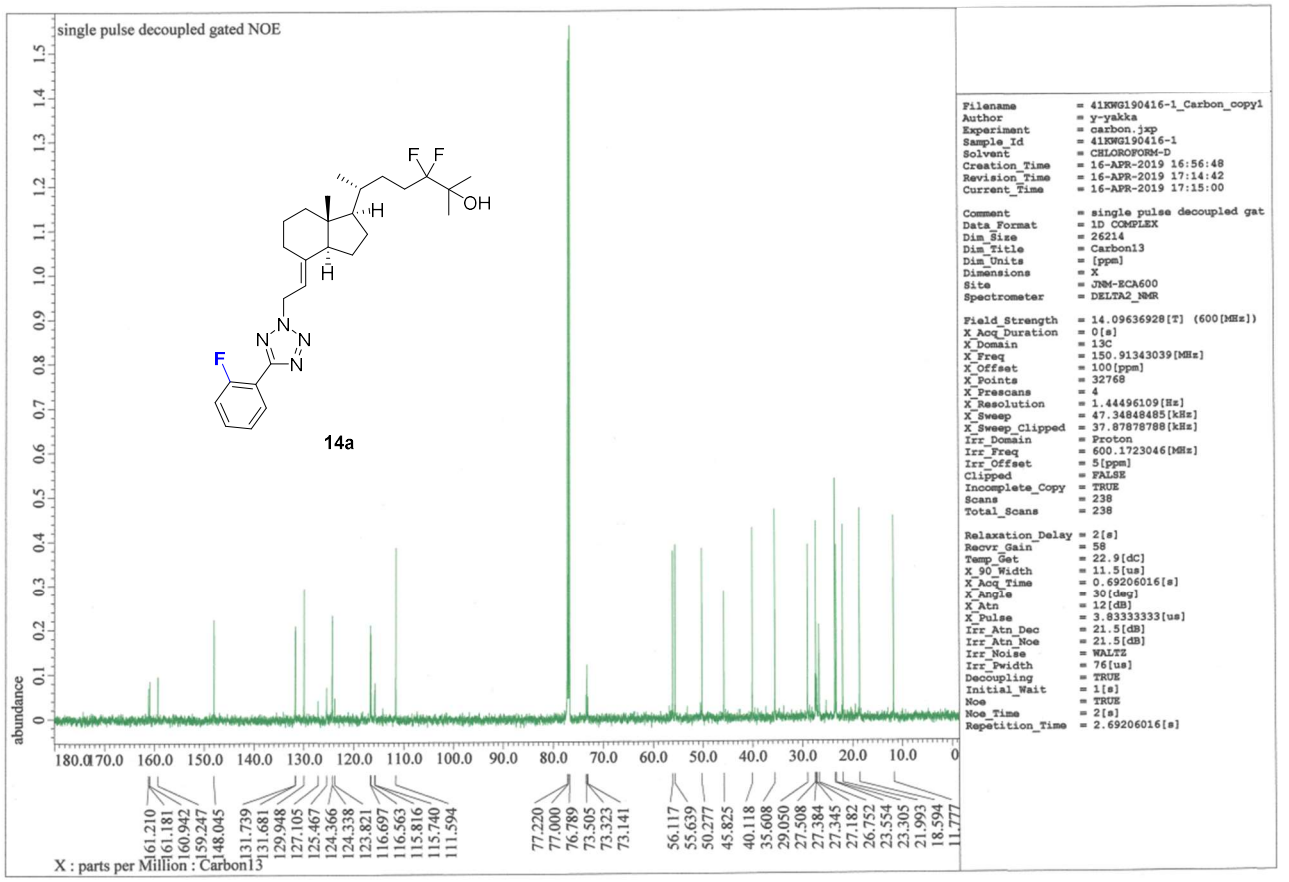

single_pulse
13a
abundance
X : parts per Million : Proton
Filename = 41KWG190412-1_Proton-1-4.j
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190412-1
Solvent = CHLOROFORM-D
Creation_Time = 12-APR-2019 15:25:18
Revision_Time = 12-APR-2019 15:28:44
Current_Time = 12-APR-2019 15:28:49
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 32
Temp_Get = 24[dC]
X_90_Width = 6.5[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.1[dB]
X_Pulse = 3.25[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
RepetitIon_Time = 7.18365952[s]

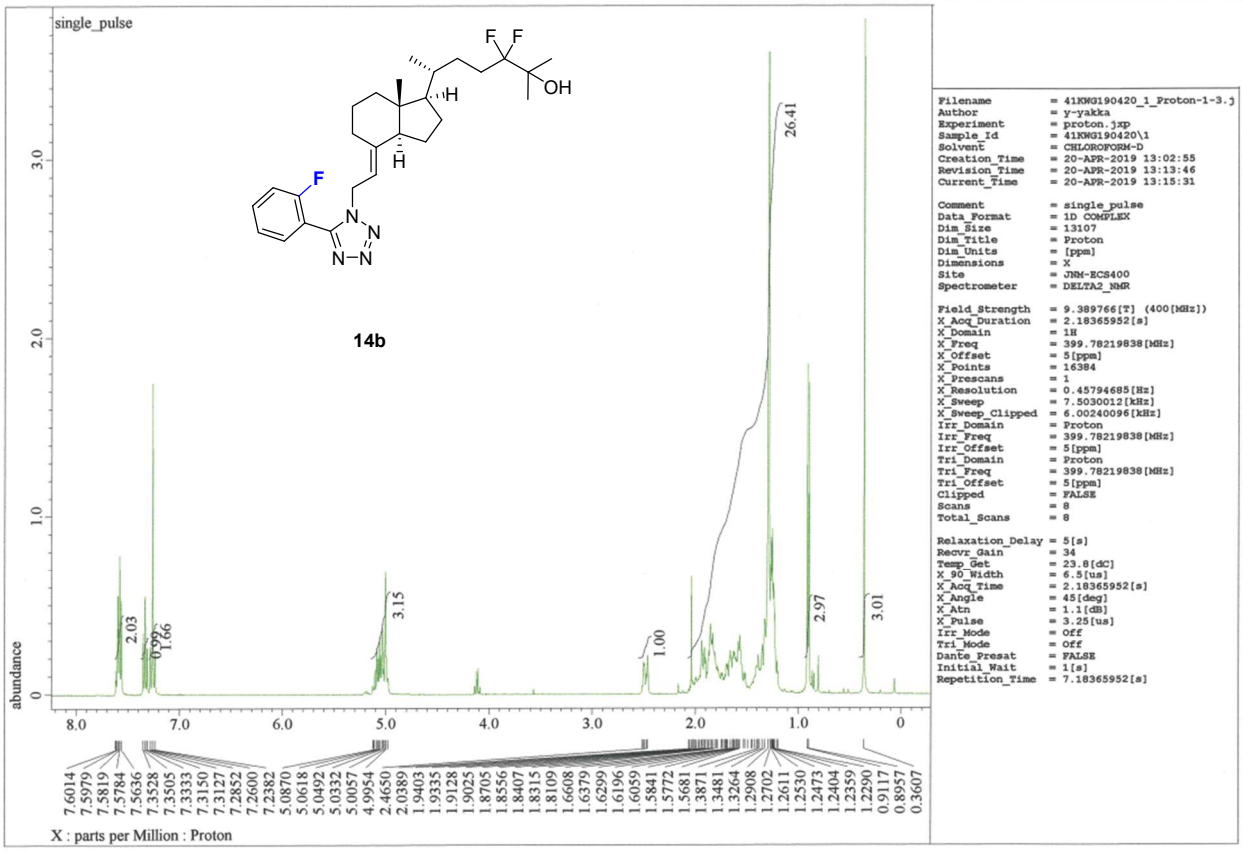

single pulse decoupled gated NOE
13a
abundance
X : parts per Million : Carbon13
Filename = 41KWG190412-1_Carbon-1-4.j
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190412-1
Solvent = CHLOROFORM-D
Creation_Time = 12-APR-2019 15:29:14
Revision_Time = 10-AUG-2019 13:58:15
Current_Time = 10-AUG-2019 13:58:36
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 512
Total_Scans = 512
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 24.2[dC]
X_90_Width = 9.7[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 3[dB]
X_Pulse = 3.23333333[us]
Irr_Atn_Dec = 25.6[dB]
Irr_Atn_Noe = 25.6[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]

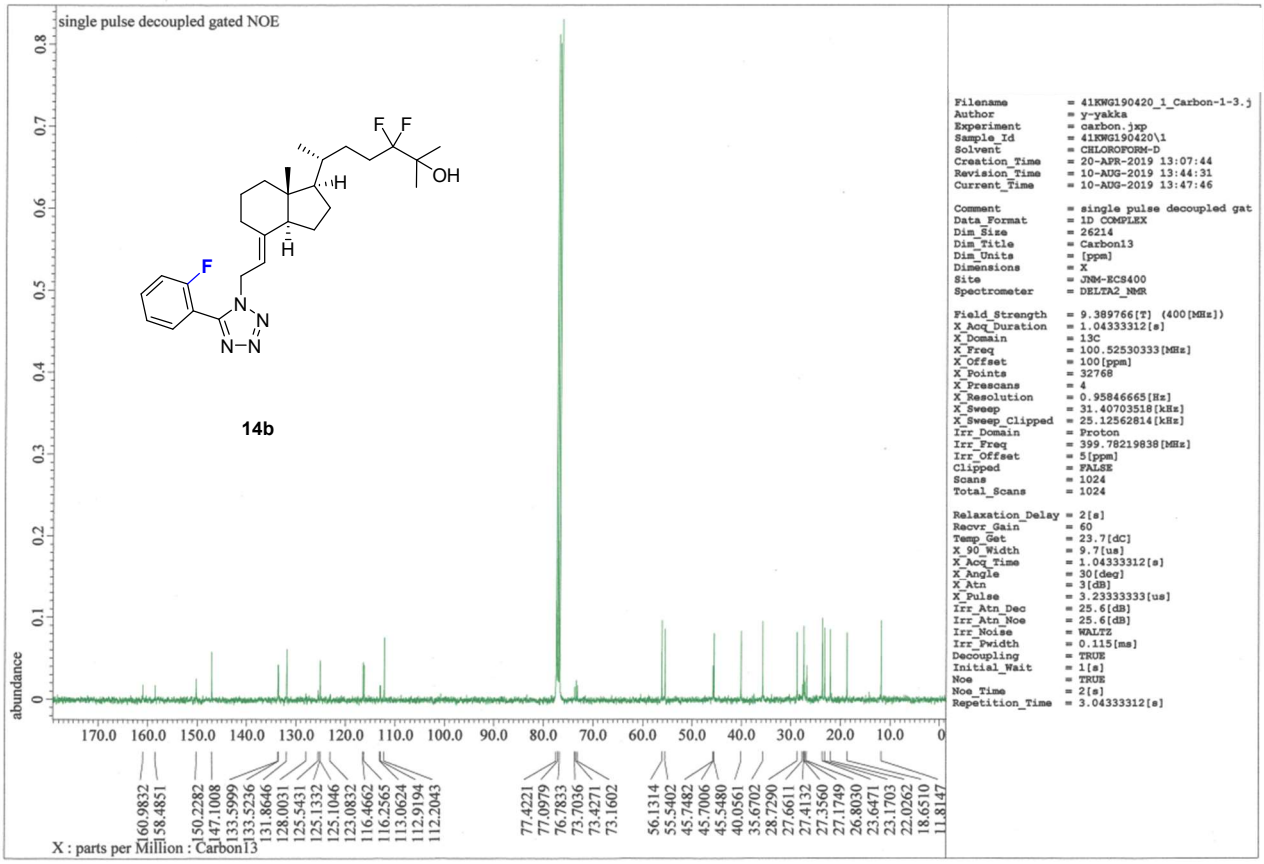

single_pulse
13b
abundance
X : parts per Million : Proton
Filename = 41KWG190413_1_Proton-1-3.j
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190413\1
Solvent = CHLOROFORM-D
Creation_Time = 13-APR-2019 15:21:42
Revision_Time = 13-APR-2019 15:33:46
Current_Time = 13-APR-2019 15:36:36
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 24.2[dC]
X_90_Width = 6.5[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.1[dB]
X_Pulse = 3.25[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]


single pulse decoupled gated NOE
13b
abundance
X : parts per Million : Carbon13
Filename = 41KWG190413_1_Carbon-1-3.j
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190413\1
Solvent = CHLOROFORM-D
Creation_Time = 13-APR-2019 15:26:19
Revision_Time = 13-APR-2019 15:57:57
Current_Time = 13-APR-2019 16:00:01
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 512
Total_Scans = 512
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.9[dC]
X_90_Width = 9.7[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 3[dB]
X_Pulse = 3.23333333[us]
Irr_Atn_Dec = 25.6[dB]
Irr_Atn_Noe = 25.6[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]


single_pulse
F F
OH
H
H
N
N
N
N
F
14a
abundance
X : parts per Million : Proton
1.00
1.09
2.44
3.20
1.00
26.54
3.24
2.90
8.134 8.130 8.121 8.119 8.109 8.106 7.450 7.438 7.277 7.265 7.260 7.252 7.235 7.218 7.204 5.344 5.339 2.851 2.829 2.035 2.019 2.002 1.913 1.900 1.821 1.813 1.800 1.790 1.778 1.767 1.740 1.732 1.686 1.629 1.606 1.501 1.496 1.486 1.472 1.432 1.340 1.328 1.314 1.307 1.298 1.292 1.274 1.266 1.250 0.944 0.934 0.540
Filename = 41KWG190416-1_Proton-1-4.j
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190416-1
Solvent = CHLOROFORM-D
Creation_Time = 16-APR-2019 16:53:21
Revision_Time = 16-APR-2019 17:01:38
Current_Time = 16-APR-2019 17:02:03
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 1.4548992[s]
X_Domain = 1H
X_Freq = 600.1723046[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.68733284[Hz]
X_Sweep = 11.26126126[kHz]
X_Sweep_Clipped = 9.00900901[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 = 5[s]
Recvr_Gain = 28
Temp_Get = 22.8[dC]
X_90_Width = 8.1[us]
X_Acq_Time = 1.4548992[s]
X_Angle = 45[deg]
X_Atn = 2.5[dB]
X_Pulse = 4.05[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.4548992[s]


single pulse decoupled gated NOE
F F
OH
H
H
N
N
N
N
F
14a
abundance
X : parts per Million : Carbon13
161.210 161.181 160.942 159.247 148.045 131.739 131.681 129.948 127.105 125.467 124.366 124.338 123.821 116.697 116.563 115.816 115.740 111.594 77.220 77.000 76.789 73.505 73.323 73.141 56.117 55.639 50.277 45.825 40.118 35.608 29.050 27.508 27.384 27.345 27.182 26.752 23.554 23.305 21.993 18.594 11.777
Filename = 41KWG190416-1_Carbon_copy1
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190416-1
Solvent = CHLOROFORM-D
Creation_Time = 16-APR-2019 16:56:48
Revision_Time = 16-APR-2019 17:14:42
Current_Time = 16-APR-2019 17:15:00
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 14.09636928[T] (600[MHz])
X_Acq_Duration = 0[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
Incomplete_Copy = TRUE
Scans = 238
Total_Scans = 238
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 22.9[dC]
X_90_Width = 11.5[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 12[dB]
X_Pulse = 3.83333333[us]
Irr_Atn_Dec = 21.5[dB]
Irr_Atn_Noe = 21.5[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]


single_pulse
F
F
OH
H
H
F
N
N
N–N
14b
26.41
3.15
2.03
0.98
1.66
1.00
2.97
3.01
abundance
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton
Filename = 41KWG190420_1_Proton-1-3.j
Author = y-yakka
Experiment = proton.jxp
Sample_Id = 41KWG190420\1
Solvent = CHLOROFORM-D
Creation_Time = 20-APR-2019 13:02:55
Revision_Time = 20-APR-2019 13:13:46
Current_Time = 20-APR-2019 13:15:31
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18365952[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.45794685[Hz]
X_Sweep = 7.5030012[kHz]
X_Sweep_Clipped = 6.00240096[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 23.8[dC]
X_90_Width = 6.5[us]
X_Acq_Time = 2.18365952[s]
X_Angle = 45[deg]
X_Atn = 1.1[dB]
X_Pulse = 3.25[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.18365952[s]


single pulse decoupled gated NOE
F
F
OH
H
H
F
N
N
N–N
14b
abundance
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13
Filename = 41KWG190420_1_Carbon-1-3.j
Author = y-yakka
Experiment = carbon.jxp
Sample_Id = 41KWG190420\1
Solvent = CHLOROFORM-D
Creation_Time = 20-APR-2019 13:07:44
Revision_Time = 10-AUG-2019 13:44:31
Current_Time = 10-AUG-2019 13:47:46
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.04333312[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95846665[Hz]
X_Sweep = 31.40703518[kHz]
X_Sweep_Clipped = 25.12562814[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 1024
Total_Scans = 1024
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.7[dC]
X_90_Width = 9.7[us]
X_Acq_Time = 1.04333312[s]
X_Angle = 30[deg]
X_Atn = 3[dB]
X_Pulse = 3.23333333[us]
Irr_Atn_Dec = 25.6[dB]
Irr_Atn_Noe = 25.6[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 0.115[ms]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 3.04333312[s]