Supplementary Information

Total synthesis of odoamide, a novel cyclic depsipeptide from an Okinawan marine cyanobacterium

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Experimental section

Synthetic general method

^{1}H NMR spectra were recorded using a JEOL ECA-500 spectrometer. Chemical shifts are reported in δ (ppm) relative to Me_4Si (in $CDCl_3$) as an internal standard. ^{13}C NMR spectra were referenced to the residual solvent signal. Melting points were measured by a hot stage melting points apparatus (uncorrected). Exact mass (HRMS) spectra were recorded on Shimadzu LC-ESI-IT-TOF-MS equipment. IR spectra were recorded on a JASCO FT/IR-4100 spectrometer. Optical rotations were measured with a JASCO P-1020 polarimeter. For flash chromatography, Wakogel C-300E (Wako) was employed. For analytical HPLC, a Cosmosil 5C18-ARII column (4.6 × 250 mm, Nacalai Tesque, Inc.) was employed with a linear gradient of CH_3CN (with 0.1% (v/v) TFA, except for the analysis of final products **5a,b** using solvents without TFA) in H_2O at a flow rate of 1 cm^3 min^{-1}, and eluting products were detected by UV at 220 nm. Preparative HPLC was performed using a Cosmosil 5C18-ARII preparative column (20 × 250 mm, Nacalai Tesque, Inc.) at a flow rate of 8 cm^3 min^{-1}.

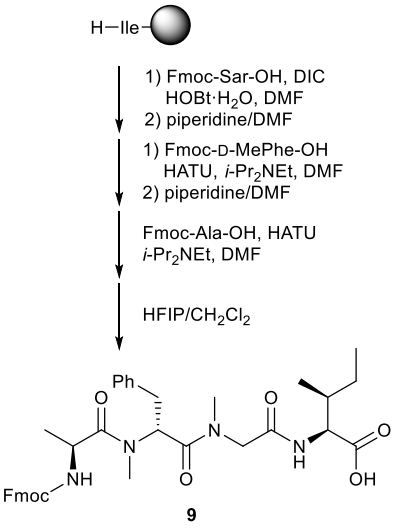
2-Oxo-2-phenylethyl (2*R*,3*S*)-2-hydroxy-3-methylpentanoate (11). To a stirred solution of D-*allo*-isoleucic acid (3.2 g, 24.5 mmol) in THF (143 cm^3) under argon were added Et_3N (3.6 cm^3, 25.7 mmol) and phenacyl bromide (5.4 g, 27.0 mmol) at 0 °C. The reaction mixture was warmed to room temperature and stirred overnight. The mixture was quenched with aqueous saturated NH_4Cl. The whole was extracted with Et_2O and the extract was washed with brine, and dried over $MgSO_4$. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography over silica gel with hexane–EtOAc (10:1 to 3:1) to give compound **11** (5.0 g, 82%) as a colorless solid: mp 52 °C; $[\alpha]^{27}_D$ –19.2 (*c* 1.07, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3524 (OH), 1704 (C=O); ^{1}H NMR (500 MHz $CDCl_3$) δ: 0.97 (3H, d, *J* 6.9), 0.99 (3H, t, *J* 7.4), 1.37-1.45 (1H, m), 1.56-1.64 (1H, m), 1.96-2.04 (1H, m), 2.66 (1H, d, *J* 6.3), 4.41 (1H, dd, J_1 6.3, J_2 2.9), 5.39 (1H, d, *J* 16.0), 5.52 (1H, d, *J* 16.0), 7.51 (2H, t, *J* 7.7), 7.62-7.65 (1H, m), 7.91-7.93 (2H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 11.9, 13.0, 26.1, 38.5, 66.5, 73.0, 127.7 (2C), 128.9 (2C), 133.9, 134.1, 174.9, 191.3; Anal. Calcd for $C_{14}H_{18}O_4$: C, 67.18; H, 7.25. Found: C, 67.15; H, 7.24.

Tetrapeptide (9). Peptide **9** was synthesized by Fmoc-based solid-phase peptide synthesis using H-Ile-(2-Cl)Trt resin (0.85 mmol g^{-1}, 2.03 g, 1.72 mmol). For Fmoc deprotection during solid-phase peptide synthesis, the peptidyl resin was treated with piperidine/DMF (2:8) for 20 min.

Coupling reaction of Sar using DIC/HOBt: DIC (0.799 cm^3, 5.17 mmol) was added to a solution of Fmoc amino acid (5.17 mmol) and HOBt·H_2O (791.0 mg, 5.17 mmol) in DMF (11.5 cm^3). The whole

was poured into the peptidyl resin (1.72 mmol), and the reaction was continued for 2 h.

Coupling reaction of D-MePhe and Ala using HATU/i-Pr$_2$NEt. To a solution of Fmoc amino acid (5.17 mmol) in DMF (11.5 cm^3) were added HATU (1.90 g, 4.99 mmol) and *i*-Pr$_2$NEt (1.80 cm^3, 10.33 mmol). The whole was poured into the peptidyl resin (1.72 mmol), and the reaction was continued for 2 h.



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Cleavage from the resin. The peptidyl resin was treated with 1,1,1,3,3,3-hexafluoropropan-2-ol (HFIP)/CH_2Cl_2 (3:7) at room temperature for 2 h. After removal of the resin by filtration, the filtrate was concentrated under reduced pressure and the residue was filtrated through a short pad of silica gel with $CHCl_3$–MeOH (10:0 to 7:1) to give peptide **9** as a colorless amorphous solid, which was used without further purification; purity 95%; HPLC retention time: 19.1 min (45 to 75% linear gradient of MeCN in 0.1% TFA solution over 30 min); $[\alpha]^{28}_D$ +55.1 (*c* 1.04, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 1634 (C=O); ^{1}H NMR (500 MHz, CD_3OD, 6:4 mixture of rotamers) δ: 0.76 (1.2H, d, *J* 6.9), 0.83-0.95 (7.8H, m), 1.16-1.28 (1H, m), 1.42-1.54 (1H, m), 1.85-1.93 (1H, m), 2.83-2.94 (3.4H, m), 2.97-3.12 (4.6H, m), 3.88 (0.4H, d, *J* 17.2), 4.04-4.18 (2.2H, m), 4.22-4.33 (2H, m), 4.35-4.46 (2H, m), 4.55 (0.4H, d, *J* 17.2), 5.72 (0.4H, dd, J_1 10.0, J_2 5.4), 5.83 (0.6H, dd, J_1 9.7, J_2 5.7), 7.15-7.25 (5H, m), 7.27-7.30 (2H, m), 7.35-7.39 (2H, m), 7.60-7.65 (2H, m), 7.76-7.78 (2H, m); ^{13}C NMR (125 MHz, CD_3OD) δ: 12.0, 12.1, 16.2, 16.3, 16.9, 17.1, 26.3 (2C), 30.8, 31.1, 35.8, 36.1 (2C), 37.3, 38.7 (2C), 48.4 (2C), 48.5 (2C), 52.4, 53.1, 55.2, 56.1, 58.3, 58.5, 68.0, 68.3, 121.1 (4C), 126.4 (2C), 126.5 (2C), 127.8 (2C), 128.3 (4C), 128.9 (2C), 129.0 (2C), 129.4 (4C), 130.6 (2C), 130.9 (2C), 138.4, 138.5, 142.7 (4C), 145.3 (2C), 145.4 (2C), 158.1, 158.3, 170.7, 170.9, 172.3, 172.7, 174.8, 174.9, 175.0, 175.3; HRMS (ESI) calcd for $C_{37}H_{44}N_4NaO_7$ (MNa^+): 679.3102; found: 679.3100.

[D-*allo*-Ile]-Odoamide (5b). According to the procedure described for the preparation of **5a**, **6b** (5.8 mg, 0.0055 mmol) was converted into **5b** (2.9 mg, 62%) as a colorless powder: $[\alpha]^{27}_D$ +4.36 (*c* 0.99, CH_3OH); IR (neat) ν_{max}/cm^{-1}: 3297 (OH), 1640 (C=O); ^{1}H NMR (500 MHz, CD_3OD, 6:4 mixture of rotamers) δ: 0.67 (1.2H, d, *J* 6.9), 0.84-1.03 (22.8H, m), 1.12-1.49 (11H, m), 1.77-1.88 (5H, m), 1.91-1.97 (0.6H, m), 1.99-2.14 (1.4H, m), 2.22-2.24 (1H, m), 2.39-2.43 (1H, m), 2.76 (1.2H, s), 2.81-2.86 (0.4H, m), 2.89 (1.8H, s), 2.95 (1.8H, s), 2.97-3.06 (2.2H, m), 3.07-3.16 (3.6H, m), 3.72 (0.4H, d, *J* 16.0), 3.77 (0.6H, d, *J* 16.9), 3.79-3.83 (1H, m), 4.41 (0.4H, d, *J* 16.9), 4.60-4.67 (1.6H, m), 4.77-4.80 (1H, m), 4.88-4.91 (1H, m), 4.97 (0.6H, dd, J_1 10.3, J_2 1.7), 5.05-5.10 (1H, m), 5.18 (0.4H, d, *J*

3.4), 5.65 (0.4H, dd, J_1 9.7, J_2 5.2), 5.85 (0.6H, dd, J_1 10.6, J_2 5.4), 6.89-6.92 (0.4H, m), 6.96-6.99 (0.6H, m), 7.12-7.24 (5H, m), 7.61 (0.6H, d, *J* 6.9), 7.91-7.94 (1H, m), 8.19 (0.4H, d, *J* 7.4); ^{13}C NMR (125 MHz, CD_3OD) δ: 10.2, 10.4, 11.9, 12.0 (2C), 12.2, 12.8, 13.0, 13.1, 13.4, 14.5, 14.6, 14.7, 14.8 (2C), 14.9, 15.1, 15.4, 16.6, 17.3, 21.3 (2C), 27.1 (2C), 27.6, 27.8, 30.4, 31.0, 31.4, 32.0, 33.0, 33.4, 34.9, 35.0, 35.2, 35.8, 36.0, 36.5, 37.7, 37.8 (2C), 38.5, 38.7, 38.9, 42.4, 42.6, 46.0, 46.8, 51.3, 52.8, 54.1, 54.5, 54.7, 55.1, 55.5, 56.8, 70.1 (2C), 76.8, 77.5, 79.1, 79.2, 127.5, 127.7, 129.2 (2C), 129.3 (2C), 129.5 (2C), 130.7 (2C), 130.8 (2C), 137.9, 138.5, 142.1, 143.3, 168.3, 168.4, 170.2, 171.0, 171.6, 172.2, 172.3 (2C), 172.5, 172.9, 173.0, 173.1, 173.6, 174.2; HRMS (ESI) calcd for $C_{46}H_{73}N_5NaO_{10}$ (MNa^+): 878.5250; found: 878.5252.

*Synthesis of ester **12d***

(*R*)-4-Benzyl-3-[(2*R*,3*S*,4*R*)-5-benzyloxy-3-hydroxy-4-methyl-2-propylpentanoyl]oxazolidin-2-one (17d). According to the procedure described for the preparation of **16c**, *ent*-**15** (337.4 mg, 1.87 mmol) was converted into **17d** (591.5 mg, 72%) with (*R*)-4-benzyl-3-pentanoyl-2-oxazolidinone as a colorless oil: $[\alpha]^{28}_D$ –26.9 (*c* 1.08, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3491 (OH), 1777 (C=O); ^{1}H NMR (500 MHz $CDCl_3$) δ: 0.93 (3H, t, *J* 7.4), 1.02 (3H, d, *J* 7.4), 1.33-1.40 (2H, m), 1.81-1.87 (3H, m), 2.66 (1H, dd, J_1 13.2, J_2 10.3), 2.81 (1H, d, *J* 3.4), 3.36 (1H, dd, J_1 13.2, J_2 3.4), 3.48-3.54 (2H, m), 4.00-4.03 (1H, m), 4.13-4.15 (2H, m), 4.20-4.24 (1H, m), 4.48-4.54 (2H, m), 4.65-4.69 (1H, m), 7.22-7.36 (10H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 11.7, 14.4, 20.0, 31.1, 36.5, 37.9, 45.6, 55.6, 65.8, 73.3, 74.4, 74.6, 127.3 (2C), 127.6 (2C), 128.3 (2C), 129.0 (2C), 129.3 (2C), 135.3, 138.1, 153.0, 176.1; HRMS (ESI) calcd for $C_{26}H_{33}NNaO_5$ (MNa^+): 462.2251; found: 462.2251.

(*R*)-4-Benzyl-3-[(2*R*,3*S*,4*R*)-5-benzyloxy-3-(*tert*-butyldimethylsilyloxy)-4-methyl-2-propylpentanoyl]oxazolidin-2-one (19d). According to the procedure described for the preparation of **18c**, **17d** (18.2 g, 41.4 mmol) was converted into **19d** (19.9 g, 87%) as a colorless oil: $[\alpha]^{28}_D$ –15.4 (*c* 1.02, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 1781 (C=O); ^{1}H NMR (500 MHz $CDCl_3$) δ: 0.02 (3H, s), 0.08 (3H, s), 0.89-0.93 (15H, m), 1.26-1.40 (2H, m), 1.66-1.80 (2H, m), 1.83-1.90 (1H, m), 2.65 (1H, dd, J_1 13.2, J_2 10.3), 3.26 (1H, dd, J_1 8.9, J_2 7.2), 3.38 (1H, dd, J_1 13.2, J_2 3.4), 3.45 (1H, dd, J_1 8.9, J_2 6.6), 4.06-4.13 (3H, m), 4.14-4.19 (1H, m), 4.45-4.51 (2H, m), 4.61-4.66 (1H, m), 7.22-7.35 (10H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: –4.0, –3.8, 11.5, 14.4, 18.4, 20.3, 26.1 (3C), 32.9, 37.9, 38.6, 47.0, 55.9, 65.7, 72.8, 73.2, 74.0, 127.3, 127.4, 127.6 (2C), 128.3 (2C), 128.9 (2C), 129.4 (2C), 135.5, 138.6, 153.0, 175.7; HRMS (ESI) calcd for $C_{32}H_{47}NNaO_5Si$ (MNa^+): 576.3116; found: 576.3115.

(2*S*,3*S*,4*R*)-5-Benzyloxy-3-(*tert*-butyldimethylsilyloxy)-4-methyl-2-propylpentan-1-ol (21d). According to the procedure described for the preparation of **20c**, **19d** (13.7 g, 24.7 mmol) was converted into **21d** (7.5 g, 80%) as a colorless oil: $[\alpha]^{29}_{D}$ –3.78 (*c* 0.96, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3431 (OH); ^{1}H NMR (500 MHz $CDCl_3$) δ: 0.03 (3H, s), 0.10 (3H, s), 0.87-0.89 (12H, m), 0.97 (3H, d, *J* 6.9), 1.04-1.11 (1H, m), 1.14-1.22 (1H, m), 1.31-1.38 (2H, m), 1.86-1.92 (1H, m), 2.00-2.07 (1H, m), 2.97-2.98 (1H, m), 3.26 (1H, dd, J_1 8.6, J_2 6.0), 3.33 (1H, dd, J_1 = J_1 8.6), 3.57-3.62 (1H, m), 3.72-3.76 (1H, m), 4.04-4.06 (1H, m), 4.48 (2H, s), 7.26-7.36 (5H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: –4.8, –4.2, 12.4, 14.3, 18.1, 21.0, 25.9 (3C), 30.5, 34.5, 45.6, 64.9, 72.8, 74.0, 74.4, 127.5 (3C), 128.3 (2C), 138.5; HRMS (ESI) calcd for $C_{22}H_{40}NaO_3Si$ (MNa^+): 403.2639; found: 403.2638.

(2*R*,3*R*,4*R*)-3-(*tert*-Butyldimethylsiloxy)-2,4-dimethylheptan-1-ol (23d). According to the procedure described for the preparation of **23c**, **24d** (4.6 g, 12.7 mmol) was converted into **23d** (3.0 g, 85%) as a colorless oil: $[\alpha]^{27}_{D}$ –1.93 (*c* 1.05, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3309 (OH); ^{1}H NMR (500 MHz $CDCl_3$) δ: 0.06 (3H, s), 0.07 (3H, s), 0.87 (3H, d, *J* 6.9), 0.89-0.91 (15H, m), 1.04-1.08 (1H, m), 1.18-1.28 (1H, m), 1.37-1.48 (2H, m), 1.62-1.64 (1H, m), 1.72-1.74 (1H, m), 1.86-1.91 (1H, m), 3.47 (1H, dt, J_1 11.1, J_2 5.2), 3.55-3.60 (1H, m), 3.62 (1H, dd, J_1 5.2, J_2 2.3); ^{13}C NMR (125 MHz, $CDCl_3$) δ: –4.3, –4.0, 11.9, 14.4, 16.4, 18.3, 20.7, 26.0 (3C), 35.4, 37.4, 38.4, 66.8, 76.7; HRMS (ESI) calcd for $C_{15}H_{34}NaO_2Si$ (MNa^+): 297.2220; found: 297.2216.

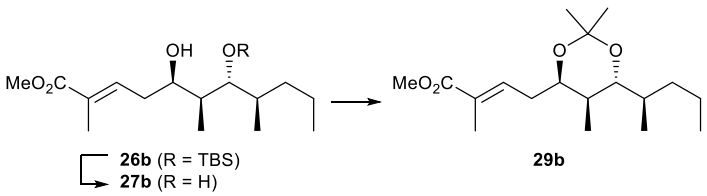
Methyl (5*S*,6*R*,7*R*,8*R*,*E*)-7-(*tert*-butyldimethylsilyloxy)-5-hydroxy-2,6,8-trimethylundec-2-enoate (12d). According to the procedure described for the preparation of **12c**, **23d** (198.5 mg, 0.72 mmol) was converted into **12d** (175.5 mg, 69%) as a colorless oil: $[\alpha]^{27}_{D}$ –18.6 (*c* 0.80, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3524 (OH), 1716 (C=O); ^{1}H NMR (500 MHz $CDCl_3$) δ: 0.09 (3H, s), 0.10 (3H, s), 0.88-0.92 (15H, m), 0.95 (3H, d, *J* 7.4), 1.04-1.12 (1H, m), 1.17-1.33 (2H, m), 1.36-1.45 (1H, m), 1.65-1.73 (2H, m), 1.87 (3H, s), 2.25 (1H, br s), 2.30-2.41 (2H, m), 3.72-3.74 (4H, m), 3.77-3.80 (1H, m), 6.79-6.82 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: –4.5, –3.4, 8.4, 12.7, 14.3, 15.4, 18.3, 20.8, 26.0 (3C), 34.3, 35.6, 38.6, 38.9, 51.7, 75.1, 79.2, 129.3, 138.8, 168.4; HRMS (ESI) calcd for $C_{21}H_{42}NaO_4Si$ (MNa^+): 409.2745; found: 409.2744.

Determination of relative configuration of the two hydroxy groups

Methyl (5*R*,6*S*,7*R*,8*S*,*E*)-5,7-dihydroxy-2,6,8-trimethylundec-2-enoate (27a). To a stirred solution of **26a** (121.0 mg, 0.31 mmol) in THF (3.1 cm^3) under argon was added TBAF (1 mol dm^{-3} in THF; 0.94 cm^3, 0.94 mmol) at room temperature. After stirring for 3 h, the reaction mixture was quenched

with aqueous saturated NH_4Cl. The whole was extracted with Et_2O and the extract was washed with brine, and dried over $MgSO_4$. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography over silica gel with hexane–EtOAc (5:1 to 2:1) to give compound **27a** (69.1 mg, 81%) as a colorless oil: $[\alpha]^{26}_D$ +21.1 (*c* 1.04, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3410 (OH), 1713 (C=O); ^1H NMR (500 MHz $CDCl_3$) δ: 0.89-0.93 (9H, m), 1.16-1.22 (1H, m), 1.28-1.42 (3H, m), 1.65-1.68 (1H, m), 1.85-1.87 (1H, m), 1.88 (3H, s), 2.13 (1H, d, *J* 4.6), 2.28-2.34 (1H, m), 2.44-2.51 (1H, m), 2.84 (1H, d, *J* 5.2), 3.51-3.54 (1H, m), 3.74 (3H, s), 4.01-4.05 (1H, m), 6.84-6.87 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 11.7, 12.7, 13.1, 14.3, 20.2, 32.9, 35.0, 36.0, 38.7, 51.8, 72.6, 78.1, 129.2, 139.3, 168.5; HRMS (ESI) calcd for $C_{15}H_{28}NaO_4$ (MNa^+): 295.1880; found: 295.1882.

Methyl (*E*)-2-methyl-4-{(4*R*,5*S*,6*R*)-2,2,5-trimethyl-6-[(*S*)-pentan-2-yl]-1,3-dioxan-4-yl}but-2-enoate (29a). To a stirred solution of **27a** (30.6 mg, 0.11 mmol) in CH_2Cl_2 (1.1 cm^3) under argon were added 2,2-dimethoxypropane (0.135 cm^3, 1.1 mmol) and PPTS (27.6 mg, 0.11 mmol) at room temperature. After stirring for 1.5 h, the reaction mixture was quenched with aqueous saturated $NaHCO_3$. The whole was extracted with CH_2Cl_2 and the extract was washed with brine, and dried over $MgSO_4$. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography over silica gel with hexane–EtOAc (15:1) to give compound **29a** (29.4 mg, 86%) as a colorless oil: $[\alpha]^{26}_D$ –6.65 (*c* 1.07, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 1716 (C=O); ^1H NMR (500 MHz $CDCl_3$) δ: 0.84 (3H, d, *J* 6.9), 0.88-0.91 (6H, m), 1.17-1.23 (1H, m), 1.24-1.35 (7H, m), 1.35-1.42 (2H, m), 1.48-1.55 (1H, m), 1.77-1.84 (1H, m), 1.85 (3H, s), 2.20-2.34 (2H, m), 3.17 (1H, dd, J_1 8.0, J_2 3.4), 3.74 (3H, s), 3.86-3.90 (1H, m), 6.73-6.76 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 12.4, 12.7, 14.1, 14.2, 20.5, 23.5, 25.2, 30.4, 35.7, 35.8, 36.4, 51.7, 68.6, 77.4, 100.3, 128.8, 138.8, 168.5; HRMS (ESI) calcd for $C_{18}H_{32}NaO_4$ (MNa^+): 335.2193; found: 335.2194.



Methyl (5*R*,6*S*,7*R*,8*R*,*E*)-5,7-dihydroxy-2,6,8-trimethylundec-2-enoate (27b). According to the procedure described for the preparation of **27a**, **26b** (26.1 mg, 0.068 mmol) was converted into **27b** (14.9 mg, 81%) as a colorless oil: $[\alpha]^{27}_D$ +36.6 (*c* 0.75, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3394 (OH), 1714 (C=O); ^1H NMR (500 MHz $CDCl_3$) δ: 0.87 (3H, d, *J* 6.9), 0.92 (3H, t, *J* 7.2), 1.00 (3H, d, *J* 7.4), 1.07-1.15 (1H, m), 1.19-1.30 (1H, m), 1.41-1.51 (1H, m), 1.53-1.59 (1H, m), 1.65-1.73 (1H, m), 1.81-

1.86 (1H, m), 1.88 (3H, s), 2.26-2.31 (1H, m), 2.44-2.50 (2H, m), 3.08 (1H, br s), 3.38-3.40 (1H, m), 3.74 (3H, s), 4.08-4.11 (1H, m), 6.81-6.84 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 11.6, 12.7, 14.4, 16.2, 20.1, 33.4, 33.6, 35.6, 37.7, 51.8, 71.6, 80.6, 129.4, 138.8, 168.4; HRMS (ESI) calcd for $C_{15}H_{28}NaO_4$ (MNa^+): 295.1880; found: 295.1880.

Methyl (*E*)-2-methyl-4-{(4*R*,5*S*,6*R*)-2,2,5-trimethyl-6-[(*R*)-pentan-2-yl]-1,3-dioxan-4-yl}but-2-enoate (29b). According to the procedure described for the preparation of **29a**, **27b** (11.5 mg, 0.042 mmol) was converted into **29b** (10.2 mg, 78%) as a colorless oil: $[\alpha]^{27}_D$ –1.27 (*c* 0.96, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 1716 (C=O); ^{1}H NMR (500 MHz $CDCl_3$) δ: 0.87 (3H, d, *J* 6.9), 0.89-0.93 (6H, m), 1.10-1.17 (1H, m), 1.18-1.25 (1H, m), 1.32 (6H, s), 1.39-1.48 (2H, m), 1.54-1.58 (1H, m), 1.76-1.82 (1H, m), 1.86 (3H, d, *J* 1.1), 2.19-2.25 (1H, m), 2.27-2.34 (1H, m), 3.14 (1H, dd, J_1 7.2, J_2 4.9), 3.74 (3H, s), 3.87-3.90 (1H, m), 6.72-6.75 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 12.7 (2C), 14.4, 15.7, 20.4, 23.4, 25.5, 30.4, 33.9, 36.1, 36.7, 51.7, 68.6, 78.7, 100.3, 128.9, 138.8, 168.5; HRMS (ESI) calcd for $C_{18}H_{32}NaO_4$ (MNa^+): 335.2193; found: 335.2187.

The syntheses of compounds **28a,b** and **30a,b** were reported in our previous article.[S1]

Preparation of reference samples for the stereochemical assignment of the polyketide substructure in odoamide[S1]

The stereochemistry of the polyketide substructure in odoamide **5** was determined by the comparative NMR analysis of the triol derivative, which was obtained by treatment of **5** (natural product) with $LiAlH_4$.[S1] The synthesis of reference triols **S2a,b** was reported in our previous article.[S1] (6*R*)-methyl triols **S2c,d** were synthesised according to the similar procedure (Scheme S1).[S1,S2] Figure S1 shows the NMR spectra of odoamide-derived triol and four synthetic references.[S1]

*Synthesis of triol **S2c***

Methyl (5*S*,6*R*,7*R*,8*S*,*E*)-5,7-dihydroxy-2,6,8-trimethylundec-2-enoate (28c). According to the procedure described for the preparation of **27a**, **12c** (312.1 mg, 0.81 mmol) was converted into **28c** (206.1 mg, 93%) as a colorless oil: $[\alpha]^{26}_D$ –16.0 (*c* 1.06, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3432 (OH), 1713 (C=O); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.89 (3H, t, *J* 7.2), 0.93 (3H, d, *J* 6.9), 0.97 (3H, d, *J* 6.9), 0.98-1.03 (1H, m), 1.22-1.31 (2H, m), 1.34-1.42 (1H, m), 1.57-1.64 (1H, m), 1.71-1.75 (1H, m), 1.87 (3H, d, *J* 1.1), 2.29-2.34 (1H, m), 2.44-2.50 (1H, m), 2.69 (1H, br s), 3.45-3.48 (2H, m), 3.74 (3H, s), 3.94-3.97 (1H, m), 6.78-6.81 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 4.7, 12.7, 14.2, 15.4, 19.6, 34.6, 34.8, 35.9, 37.6, 51.8, 75.8, 81.5, 129.4, 138.6, 168.5; HRMS (ESI) calcd for $C_{15}H_{28}NaO_4$ (MNa^+): 295.1880; found: 295.1885.

Methyl (*E*)-2-methyl-4-{(4*S*,5*R*,6*R*)-2,2,5-trimethyl-6-[(*S*)-pentan-2-yl]-1,3-dioxan-4-yl}but-2-enoate (30c). According to the procedure described for the preparation of **29a**, **28c** (154.8 mg, 0.57 mmol) was converted into **30c** (137.7 mg, 77%) as a colorless oil: $[\alpha]^{27}_D$ –7.61 (*c* 1.12, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 1716 (C=O); ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.84 (3H, d, *J* 6.9), 0.88-0.94 (7H, m), 1.21-1.32 (2H, m), 1.40 (6H, s), 1.41-1.48 (1H, m), 1.50-1.62 (2H, m), 1.87 (3H, s), 2.26-2.32 (1H, m), 2.35-2.41 (1H, m), 3.39 (1H, dd, J_1 9.7, J_2 2.3), 3.75 (3H, s), 3.94 (1H, ddd, J_1 7.2, J_2 7.2 J_3 2.3), 6.72-6.75 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 4.7, 12.7, 14.2, 15.8, 19.5, 19.6, 29.9, 32.4, 32.5, 32.9, 33.6, 51.7, 72.6, 78.1, 98.9, 129.1, 138.1, 168.5; HRMS (ESI) calcd for $C_{18}H_{32}NaO_4$ (MNa^+): 335.2193; found: 335.2189.

(*E*)-2-Methyl-4-{(4*S*,5*R*,6*R*)-2,2,5-trimethyl-6-[(*S*)-pentan-2-yl]-1,3-dioxan-4-yl}but-2-en-1-ol (S1c). To a stirred solution of **30c** (53.2 mg, 0.17 mmol) in THF (1.7 cm^3) under argon was added DIBAL (1.0 M in toluene; 0.51 cm^3, 0.51 mmol) at –78 °C. After stirring for 3 h, a saturated aqueous solution of sodium potassium tartrate was added to the reaction mixture and the mixture was stirred

at room temperature for 1 h. The whole was extracted with Et_2O and the extract was washed with brine, dried over $MgSO_4$. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography over silica gel with hexane–EtOAc (7:1 to 4:1) to give compound **S1c** (47.6 mg, 98%) as a colorless oil: $[\alpha]^{27}_D$ –7.12 (*c* 1.02, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3421 (OH); ^1H NMR (500 MHz, $CDCl_3$) δ: 0.84 (3H, d, *J* 6.9), 0.87-0.92 (7H, m), 1.20-1.34 (3H, m), 1.40-1.46 (7H, m), 1.50-1.60 (2H, m), 1.69 (3H, s), 2.15-2.29 (2H, m), 3.37 (1H, dd, J_1 9.7, J_2 1.7), 3.85 (1H, td, J_1 7.2, J_2 1.9), 4.03 (2H, s), 5.38-5.41 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 4.7, 14.0, 14.2, 15.8, 19.5, 19.6, 30.0, 31.2, 32.1, 32.9, 33.6, 68.7, 73.4, 78.2, 98.8, 121.2, 136.7; HRMS (ESI) calcd for $C_{17}H_{32}NaO_3$ (MNa^+): 307.2244; found: 307.2244.

(5*S*,6*R*,7*R*,8*S*,*E*)-2,6,8-Trimethylundec-2-ene-1,5,7-triol (S2c). To a stirred solution of **S1c** (23.6 mg, 0.083 mmol) in MeOH (1.5 cm^3) was added 1N HCl (0.25 cm^3, 0.25 mmol) at room temperature. After stirring overnight, the reaction mixture was quenched with aqueous saturated $NaHCO_3$. The whole was extracted with EtOAc and the extract was washed with brine, dried over $MgSO_4$. The filtrate was concentrated under reduced pressure and the residue was purified by flash chromatography over silica gel with hexane–EtOAc (3:1 to 1:2) to give compound **S2c** (15.2 mg, 75%) as a colorless oil: $[\alpha]^{27}_D$ –9.66 (*c* 1.00, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3334 (OH); ^1H NMR (500 MHz, CD_3OD) δ: 0.89-0.92 (6H, m), 0.95 (3H, d, *J* 6.9), 1.07-1.13 (1H, m), 1.24-1.45 (3H, m), 1.62-1.71 (5H, m), 2.25-2.27 (2H, m), 3.39 (1H, dd, J_1 6.3, J_2 5.2), 3.73 (1H, td, J_1 6.6, J_2 3.4), 3.93 (2H, s), 5.44-5.46 (1H, m); ^{13}C NMR (125 MHz, CD_3OD) δ: 7.8, 14.0, 14.7, 15.0, 20.9, 34.3, 36.3, 36.8, 39.8, 65.9, 75.1, 79.1, 123.0, 137.8; HRMS (ESI) calcd for $C_{14}H_{28}NaO_3$ (MNa^+): 267.1931; found: 267.1930.

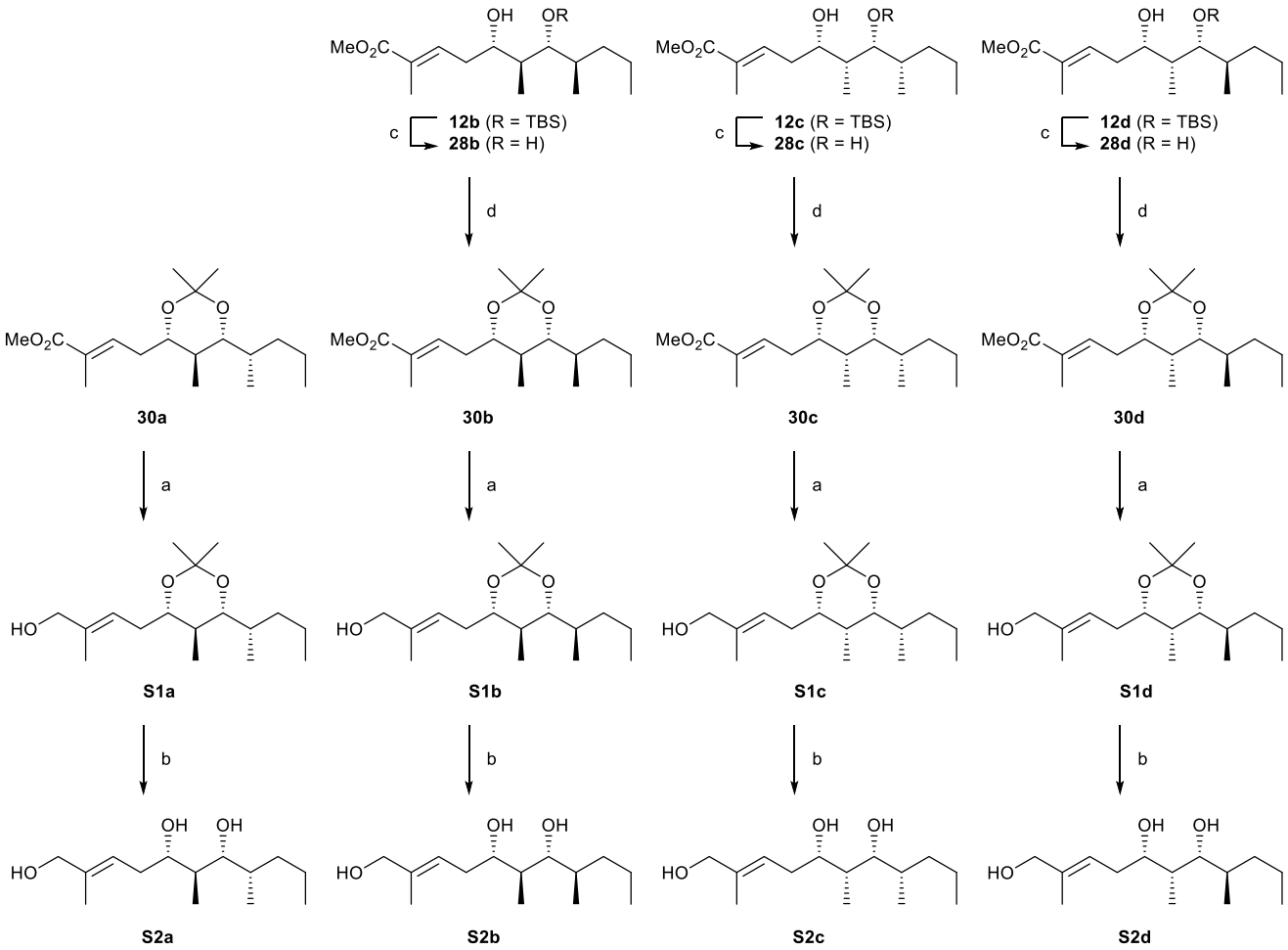
*Synthesis of triol **S2d***

Methyl (5*S*,6*R*,7*R*,8*R*,*E*)-5,7-dihydroxy-2,6,8-trimethylundec-2-enoate (28d). According to the procedure described for the preparation of **27a**, **12d** (314.0 mg, 0.81 mmol) was converted into **28d** (181.9 mg, 83%) as a colorless oil: $[\alpha]^{27}_D$ +2.93 (*c* 1.04, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3436 (OH), 1714 (C=O); ^1H NMR (500 MHz $CDCl_3$) δ: 0.80 (3H, d, *J* 6.9), 0.91-0.93 (6H, m), 1.04-1.12 (1H, m), 1.19-1.29 (1H, m), 1.42-1.49 (1H, m), 1.53-1.66 (2H, m), 1.68-1.72 (1H, m), 1.87 (3H, d, *J* 1.1), 2.29-2.35 (1H, m), 2.44-2.50 (1H, m), 2.79 (1H, br s), 3.45-3.47 (1H, m), 3.51 (1H, br s), 3.74 (3H, s), 3.96-3.98 (1H, m), 6.78-6.81 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 4.2, 12.6, 14.4, 15.3, 19.7, 34.6, 34.9, 36.3, 37.5, 51.8, 76.2, 81.5, 129.4, 138.5, 168.5; HRMS (ESI) calcd for $C_{15}H_{28}NaO_4$ (MNa^+): 295.1880; found: 295.1880.

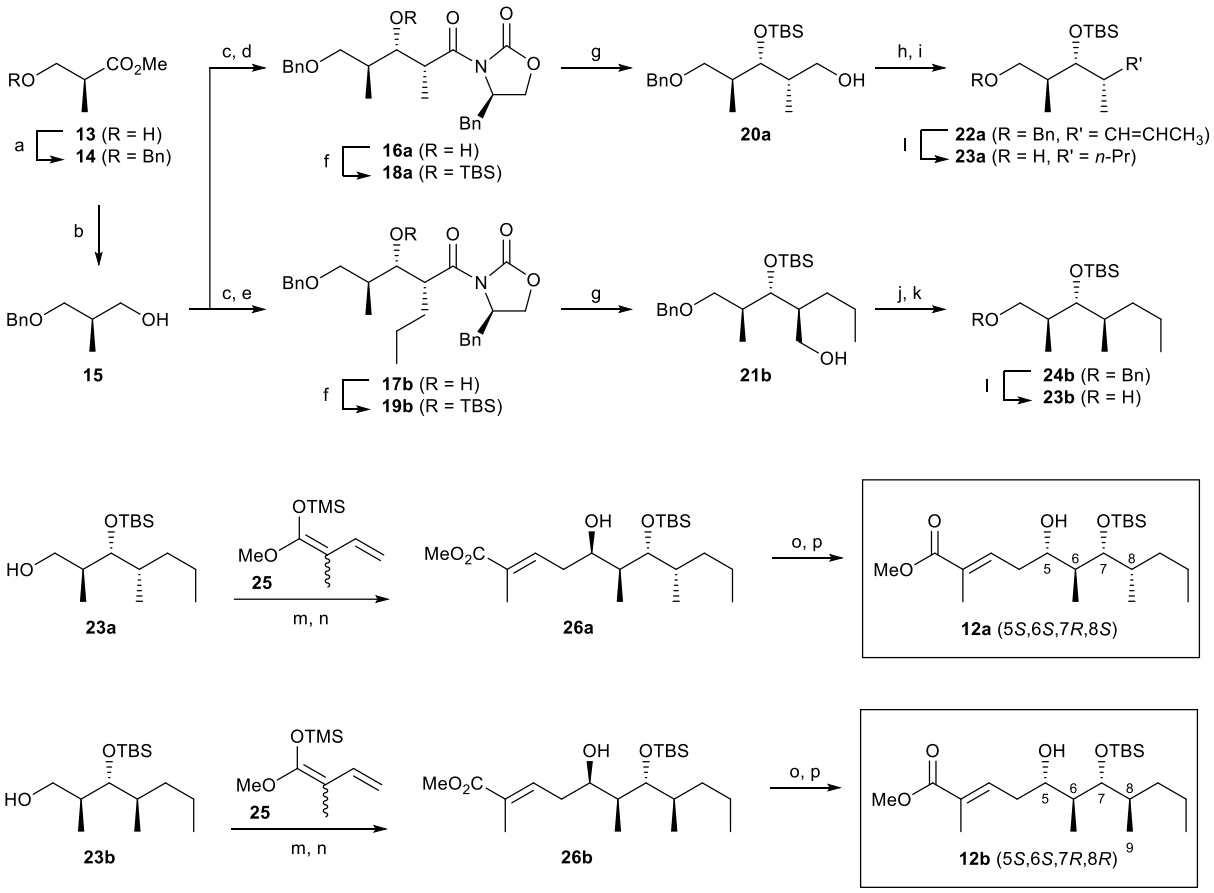
Methyl (*E*)-2-methyl-4-{(4*S*,5*R*,6*R*)-2,2,5-trimethyl-6-[(*R*)-pentan-2-yl]-1,3-dioxan-4-yl}but-2-enoate (30d). According to the procedure described for the preparation of **29a**, **28d** (153.8 mg, 0.56 mmol) was converted into **30d** (162.2 mg, 93%) as a colorless oil: $[\alpha]^{26}_{D}$ +4.04 (*c* 1.08, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 1717 (C=O); ^{1}H NMR (500 MHz $CDCl_3$) δ: 0.77 (3H, d, *J* 6.9), 0.84 (3H, d, *J* 6.9), 0.89 (3H, t, *J* 7.2), 0.95-1.02 (1H, m), 1.16-1.27 (1H, m), 1.32-1.43 (7H, m), 1.44-1.50 (1H, m), 1.51-1.58 (1H, m), 1.60-1.67 (1H, m), 1.86 (3H, s), 2.26-2.41 (2H, m), 3.40 (1H, dd, J_1 9.7, J_2 1.7), 3.74 (3H, s), 3.95 (1H, td, J_1 7.2, J_2 2.3), 6.71-6.74 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 4.5, 12.7, 14.0, 14.6, 19.5 (2C), 29.9, 32.5 (2C), 34.0, 35.2, 51.7, 72.8, 77.4, 98.9, 129.1, 138.0, 168.5; HRMS (ESI) calcd for $C_{18}H_{32}NaO_4$ (MNa^+): 335.2193; found: 335.2188.

(*E*)-2-Methyl-4-{(4*S*,5*R*,6*R*)-2,2,5-trimethyl-6-[(*R*)-pentan-2-yl]-1,3-dioxan-4-yl}but-2-en-1-ol (S1d). According to the procedure described for the preparation of **S1c**, **30d** (131.4 mg, 0.42 mmol) was converted into **S1d** (111.5 mg, 93%) as a colorless oil: $[\alpha]^{27}_{D}$ +9.48 (*c* 0.74, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3324 (OH); ^{1}H NMR (500 MHz $CDCl_3$) δ: 0.77 (3H, d, *J* 6.9), 0.83 (3H, d, *J* 6.9), 0.89 (3H, t, *J* 7.4), 0.94-1.02 (1H, m), 1.16-1.27 (1H, m), 1.33-1.43 (8H, m), 1.45-1.51 (1H, m), 1.51-1.59 (1H, m), 1.61-1.66 (1H, m), 1.69 (3H, s), 2.15-2.28 (2H, m), 3.39 (1H, dd, J_1 9.7, J_2 2.3), 3.86 (1H, td, J_1 7.3, J_2 2.1), 4.02 (2H, s), 5.37-5.40 (1H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 4.5, 14.0 (2C), 14.6, 19.6 (2C), 30.0, 31.2, 32.2, 34.0, 35.2, 68.7, 73.5, 77.6, 98.8, 121.2, 136.7; HRMS (ESI) calcd for $C_{17}H_{32}NaO_3$ (MNa^+): 307.2244; found: 307.2242.

(5*S*,6*R*,7*R*,8*R*,*E*)-2,6,8-Trimethylundec-2-ene-1,5,7-triol (S2d). According to the procedure described for the preparation of **S2c**, **S1d** (49.4 mg, 0.17 mmol) was converted into **S2d** (39.0 mg, 96%) as a colorless oil: $[\alpha]^{28}_{D}$ +13.3 (*c* 0.93, $CHCl_3$); IR (neat) ν_{max}/cm^{-1}: 3338 (OH); ^{1}H NMR (500 MHz CD_3OD) δ: 0.82 (3H, d, *J* 6.9), 0.90-0.93 (6H, m), 1.02-1.09 (1H, m), 1.18-1.28 (1H, m), 1.41-1.51 (1H, m), 1.55-1.66 (2H, m), 1.67-1.72 (4H, m), 2.26-2.28 (2H, m), 3.35 (1H, dd, J_1 8.0, J_2 3.4), 3.74 (1H, td, J_1 6.4, J_2 3.8), 3.93 (2H, s), 5.44-5.47 (1H, m); ^{13}C NMR (125 MHz, CD_3OD) δ: 6.9, 14.1, 14.9, 16.2, 21.0, 34.2, 35.6, 37.0, 39.4, 68.9, 76.3, 80.2, 122.9, 137.8; HRMS (ESI) calcd for $C_{14}H_{28}NaO_3$ (MNa^+): 267.1931; found: 267.1931.



Scheme S1. Synthesis of triols **S2a-d**.[S1] *Reagents and conditions*: (a) DIBAL, toluene, THF, –78 °C, 90% (**S1a**), 96% (**S1b**), 98% (**S1c**) and 93% (**S1d**); (b) HCl, MeOH, H_2O, rt, 51% (**S2a**); 93% (**S2b**), 75% (**S2c**) and 96% (**S2d**); (c) TBAF, THF, rt, 93% (**28b**), 93% (**28c**) and 83% (**28d**); (d) 2,2-dimethoxypropane, PPTS, CH_2Cl_2, rt, 90% (**30b**), 77% (**30c**) and 93% (**30d**).



Scheme S2. Synthesis of esters **12a**,**b**.[S1] *Reagents and conditions*: (a) benzyl 2,2,2-trichloroacetimidate, TfOH, CH_2Cl_2, cyclohexane, 0 °C to rt, 88%; (b) $LiAlH_4$, THF, 0 °C, 75%; (c) $(COCl)_2$, DMSO, DIPEA, CH_2Cl_2, –78 °C to 0 °C; (d) (*R*)-4-benzyl-3-propionyl-2-oxazolidinone, *n*-Bu_2BOTf, DIPEA, CH_2Cl_2, –78 °C to –10 °C, 83% (2 steps); (e) (*R*)-4-benzyl-3-pentanoyl-2-oxazolidinone, *n*-Bu_2BOTf, DIPEA, CH_2Cl_2, –78 °C to –10 °C, 69% (2 steps); (f) TBSOTf, 2,6-lutidine, CH_2Cl_2, 0 °C to rt, 90% (**18a**) and 90% (**19b**); (g) $LiBH_4$, MeOH, THF, 0 °C to rt, 73% (**20a**) and 72% (**21b**); (h) $(COCl)_2$, DMSO, DIPEA, CH_2Cl_2, –78 °C to 0 °C; (i) ethyltriphenylphosphonium bromide, *n*-BuLi, THF, rt, 72% (2 steps, *Z*/*E* = 7:1); (j) TsCl, Et_3N, $Me_3N{\cdot}HCl$, CH_2Cl_2, rt; (k) $LiAlH_4$, THF, 0 °C to rt, 57% (2 steps); (l) Pd/C, H_2, EtOH, rt, 85% (**23a**) and 78% (**23b**); (m) $(COCl)_2$, DMSO, DIPEA, CH_2Cl_2, –78 °C to 0 °C; (n) **25**, $BF_3{\cdot}OEt_2$, CH_2Cl_2, Et_2O, –78 °C, 85% (**26a**) and 71% (**26b**) (2 steps); (o) Dess-Martin periodinane, CH_2Cl_2, rt; (p) $NaBH_4$, MeOH, –78 °C, 83% (**12a**, dr >97:3) and 71% (**12b**, dr >99:1) (2 steps).

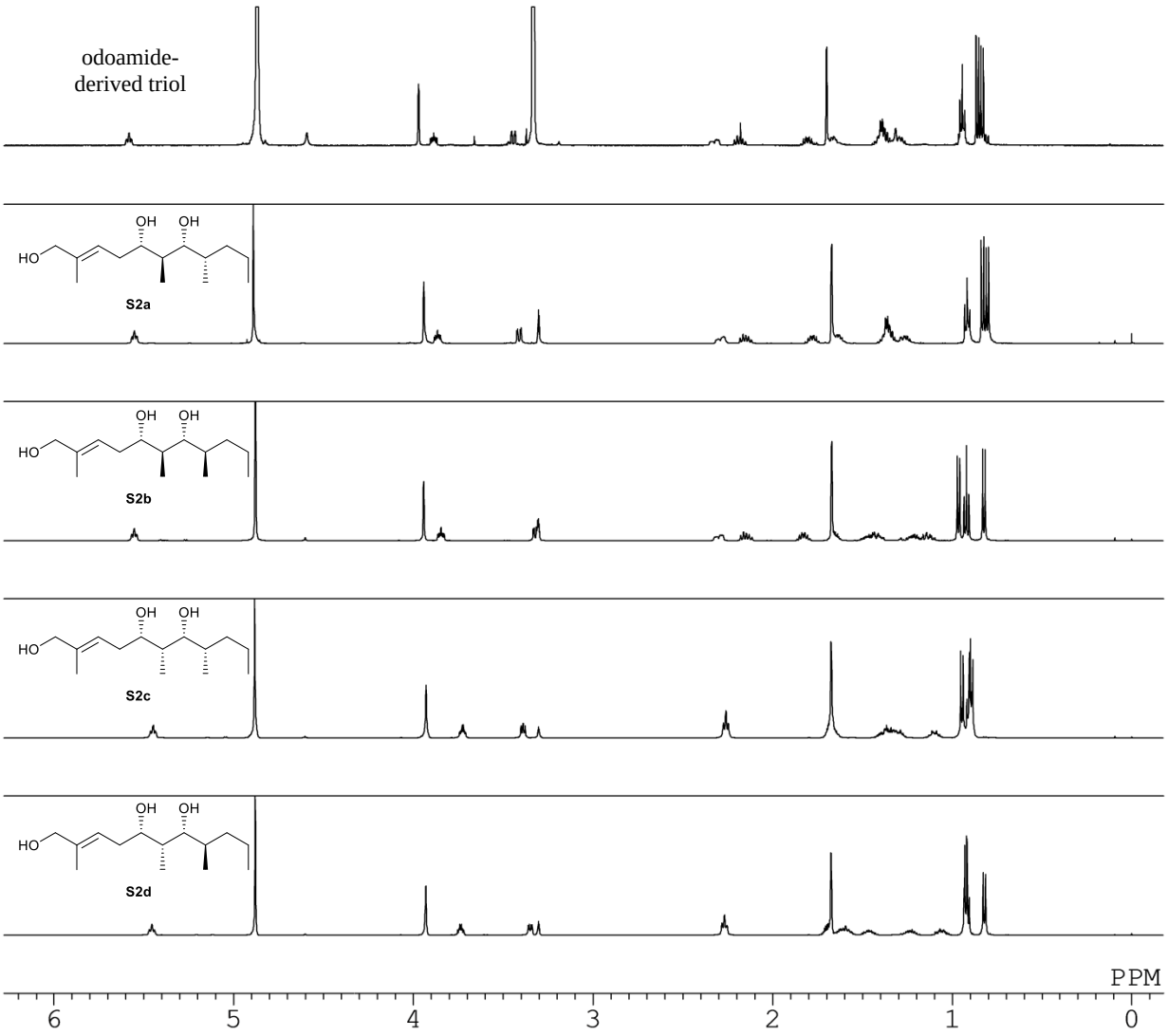
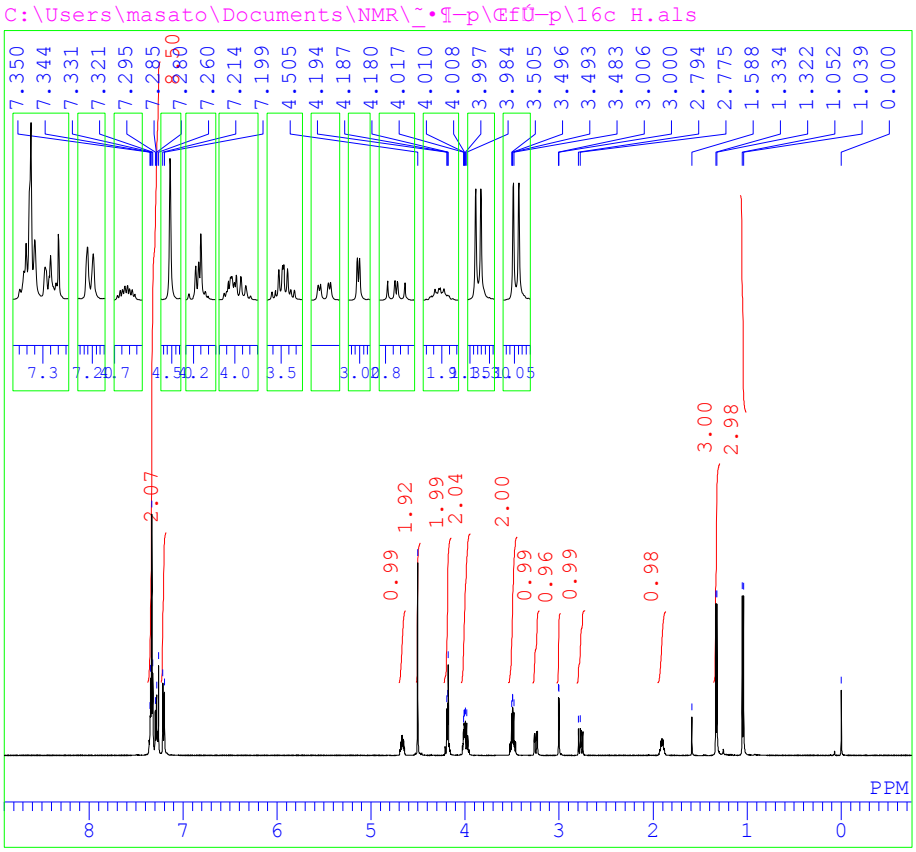


Figure S1. Comparison of ^{1}H NMR spectra between synthetic triols **S2a-d** and natural product-derived triol (in CD_3OD).[S1]

References

S1 The isolation and structural assignment of odoamide were reported in our previous article, see: K. Sueyoshi, M. Kaneda, S. Sumimoto, S. Oishi, N. Fujii, K. Suenaga and T. Teruya, *Tetrahedron*, 2016, **35**, 5472-5478.

S2 T. Mutou, K. Suenaga, T. Fujita, T. Itoh, N. Takada, K. Hayamizu, H. Kigoshi and K. Yamada, *Synlett*, 1997, 199-201.

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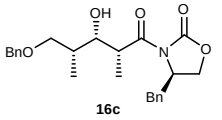

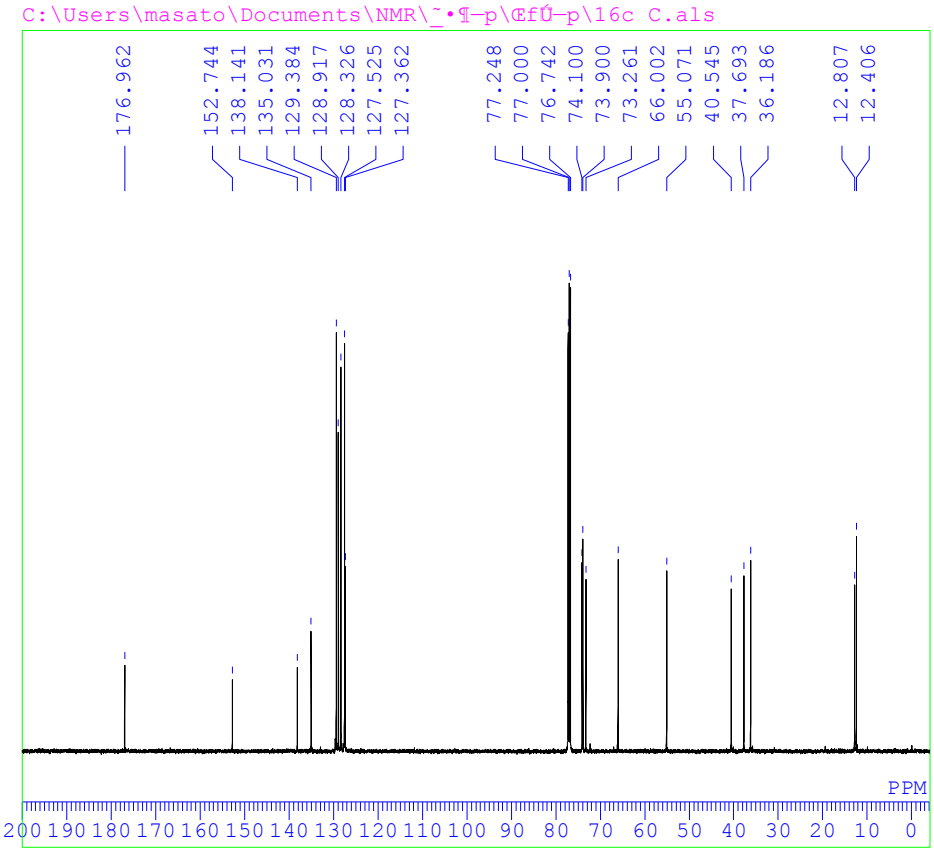


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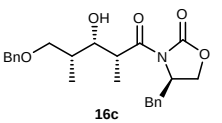

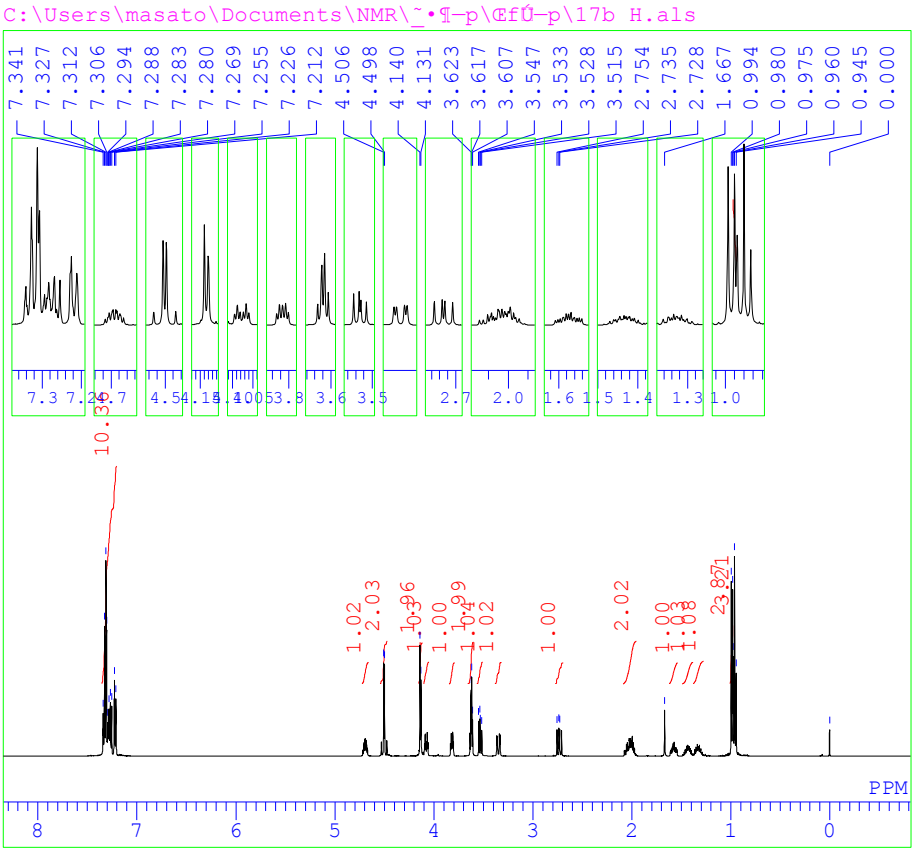


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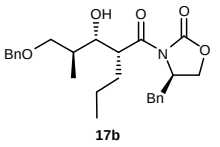
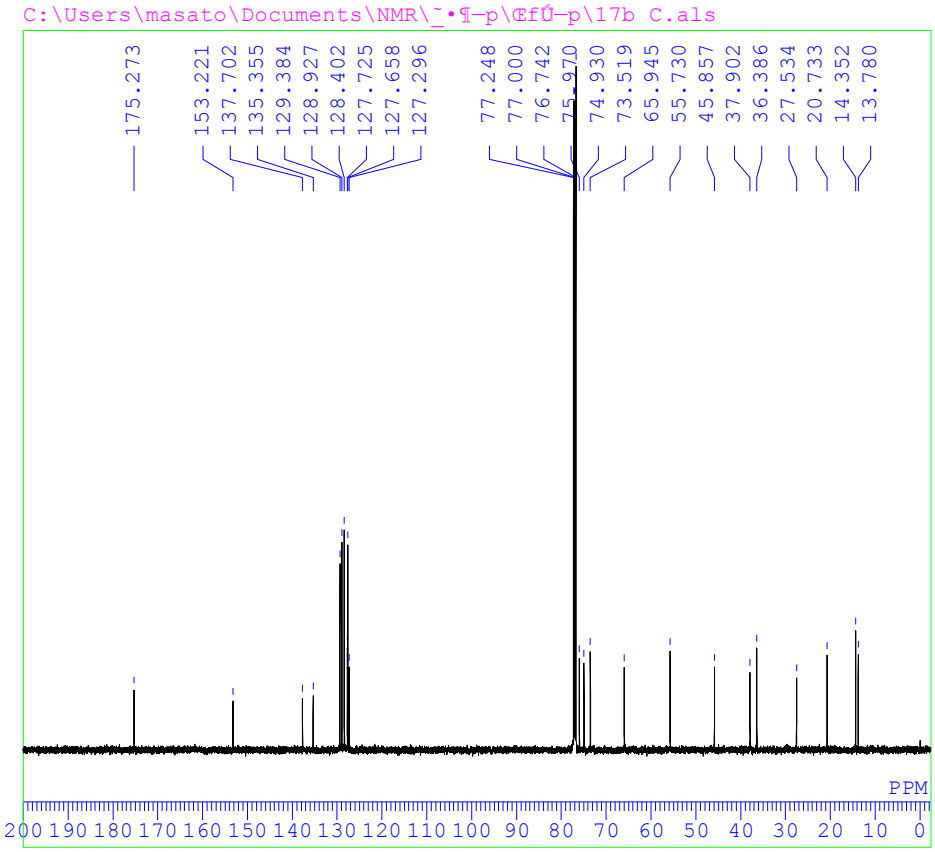
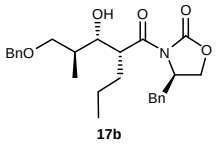


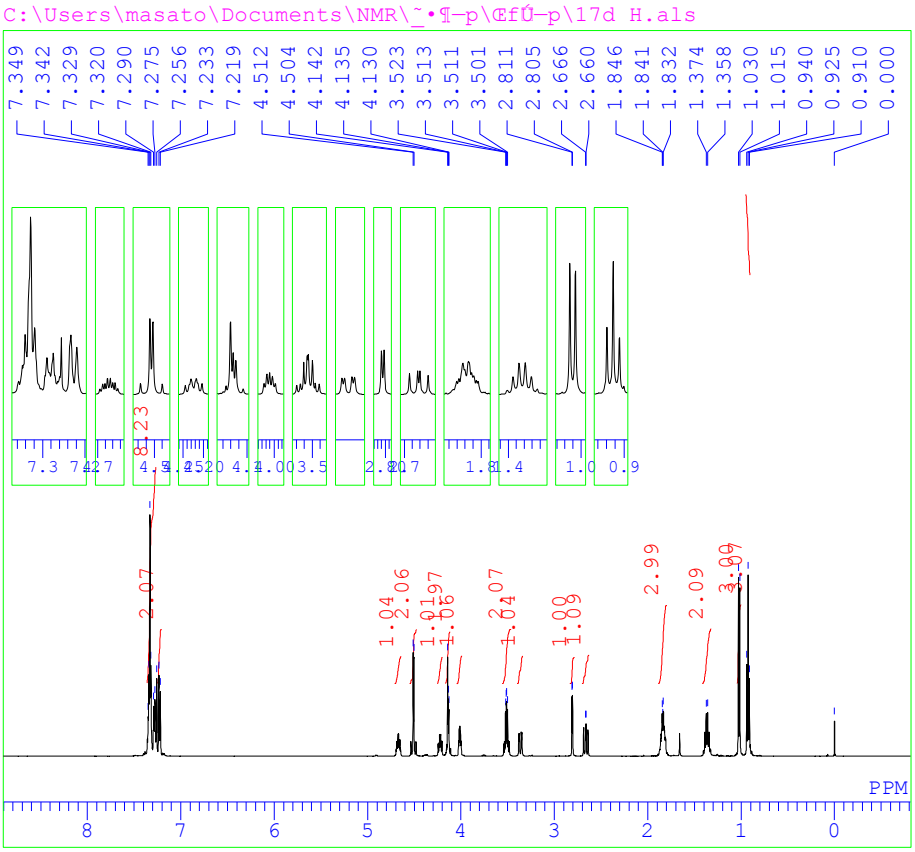
17b



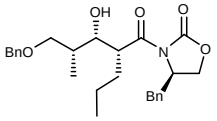

```
DFILE  17b C.als
COMNT  single pulse dec
DATIM  2014-08-30 09:43
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          24.9 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            54
```



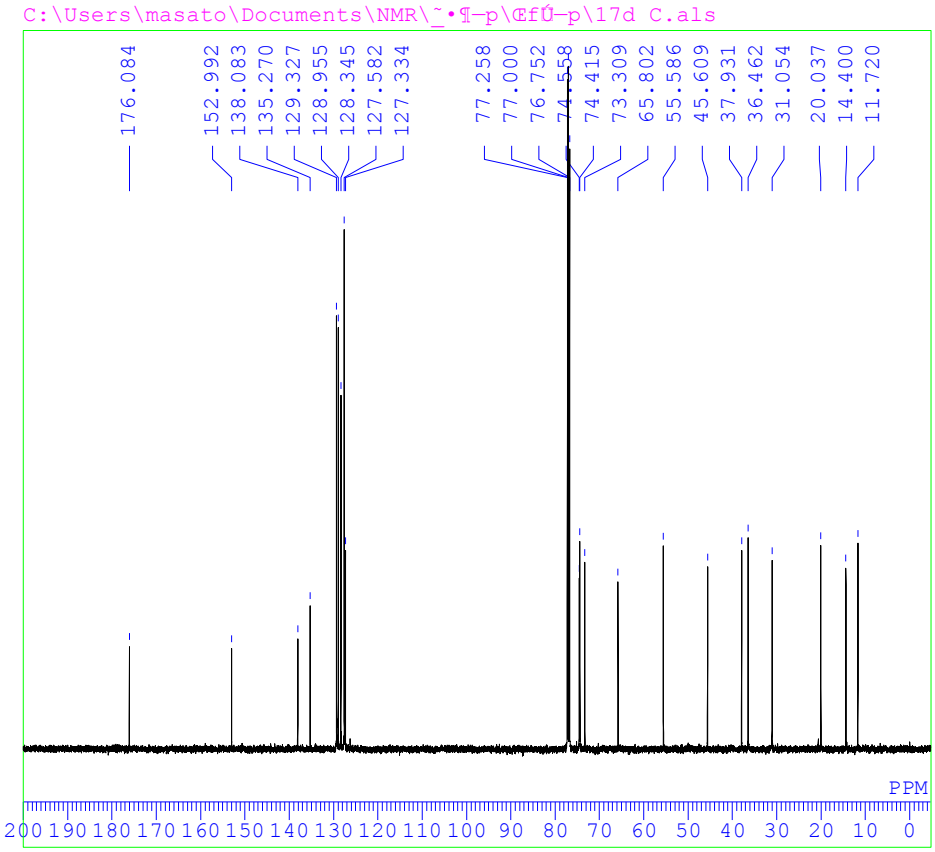
17b



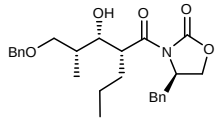

```
DFILE  17d H.als
COMNT  single_pulse
DATIM  2016-01-09 10:38
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          19.7 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            36
```



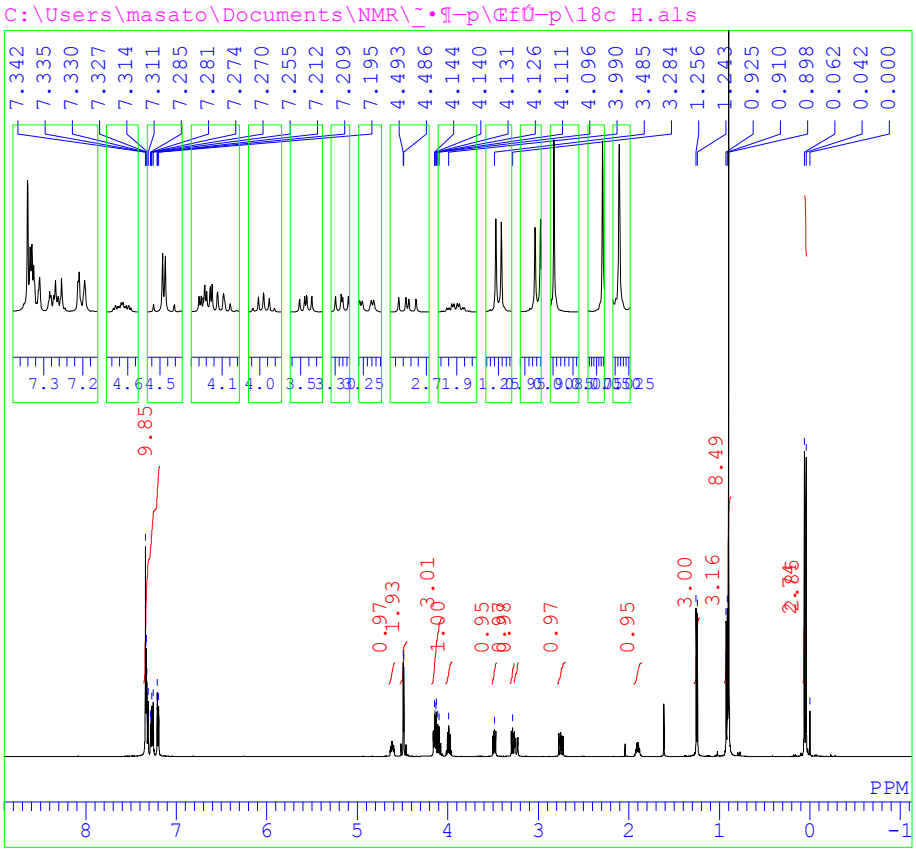
17d



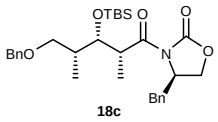

```
DFILE  17d C.als
COMNT  single pulse dec
DATIM  2015-08-09 18:21
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.6 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            58
```



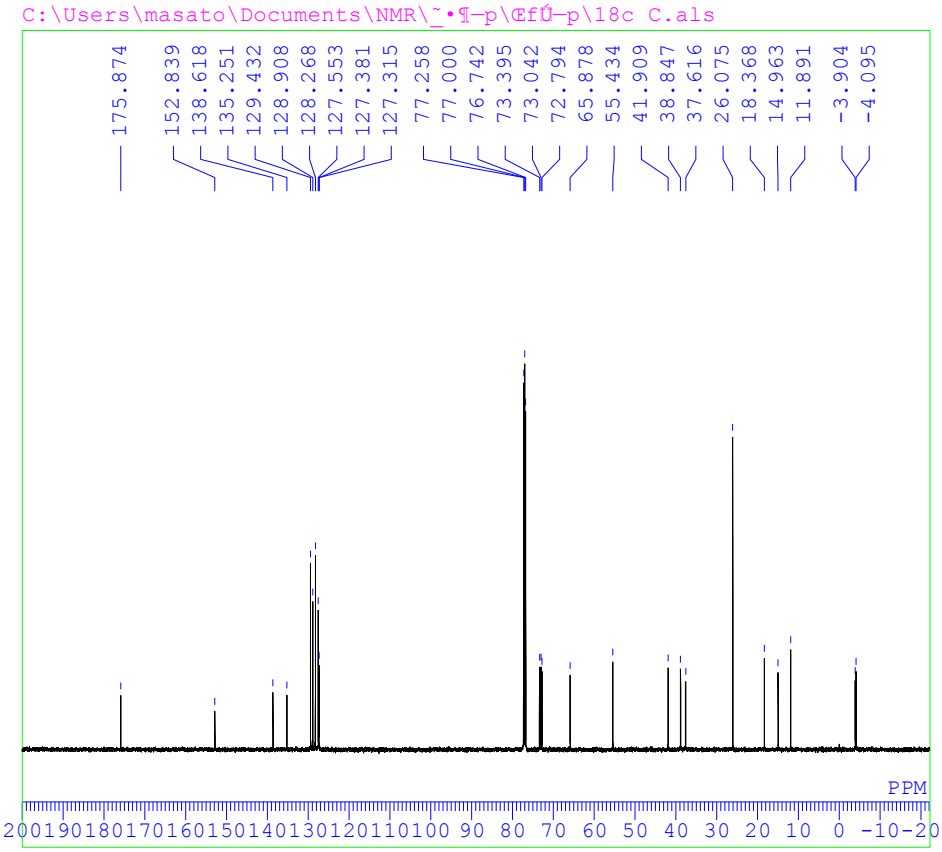
17d



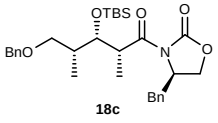

```
DFILE  18c H.als
COMNT  single_pulse
DATIM  2015-07-24 14:46
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1             6.82 use
IRNUC  1H
CTEMP          21.9 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            36
```



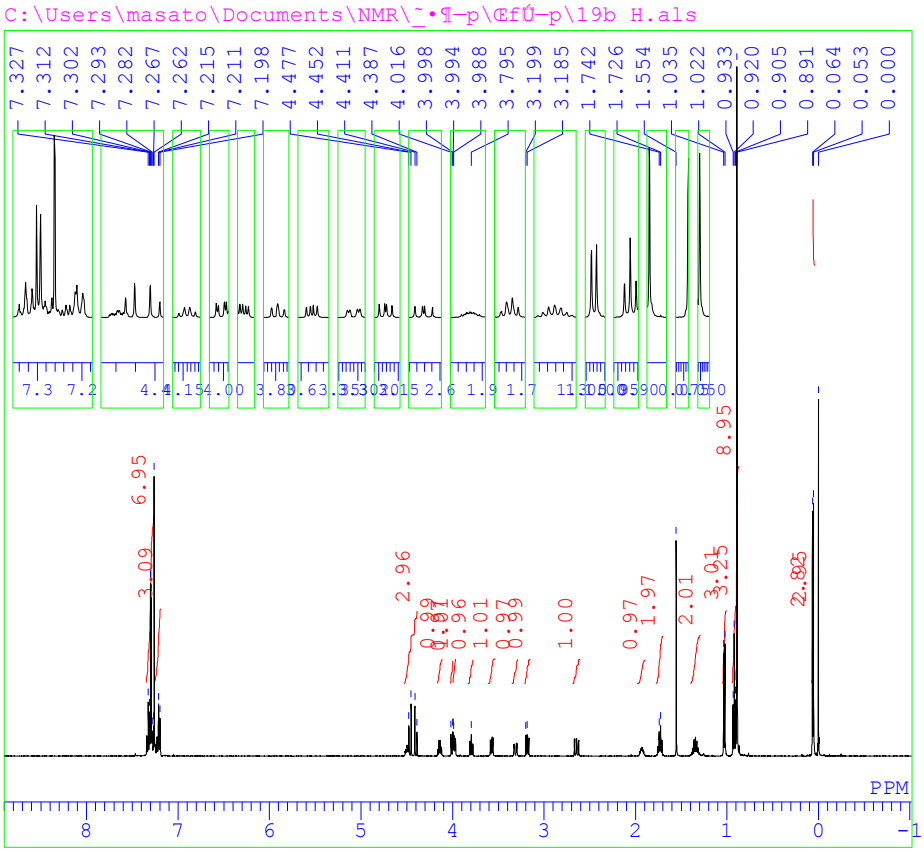
18c



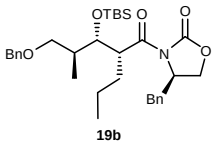

```
DFILE  18c C.als
COMNT  single pulse dec
DATIM  2015-07-24 15:34
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1             3.50 use
IRNUC  1H
CTEMP          22.1 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            54
```



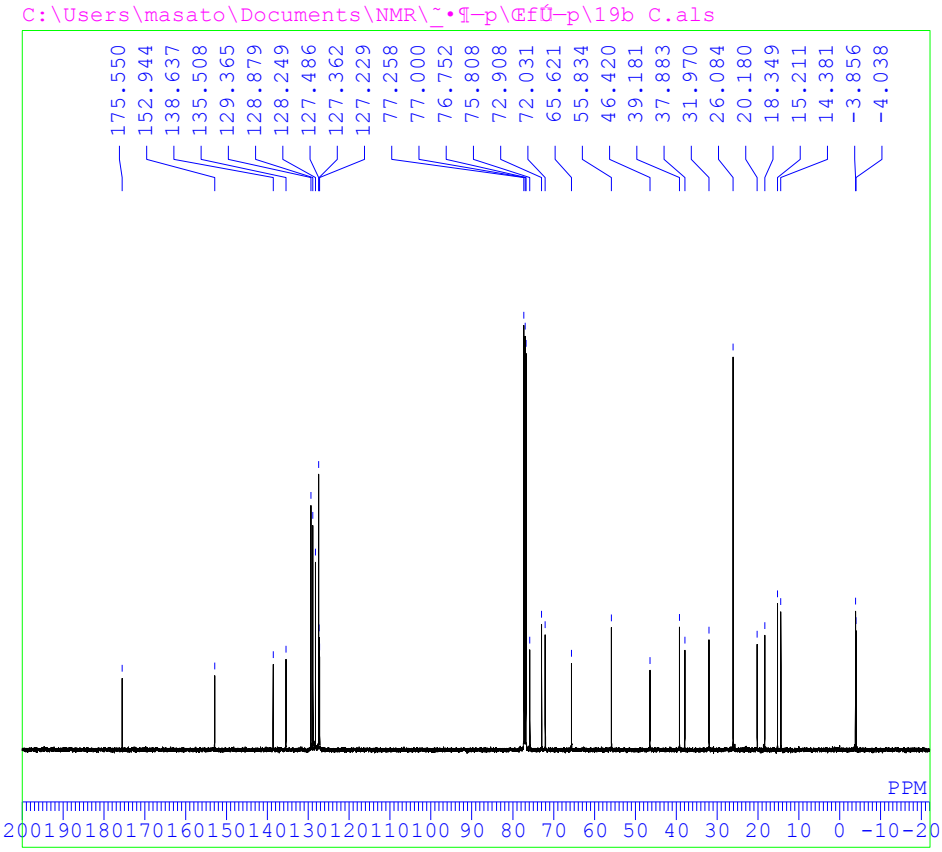
18c



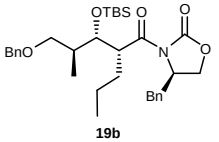

```
DFILE  19b H.als
COMNT  single_pulse
DATIM  2015-12-24 15:38
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP         20.4 c
SLVNT  CDCL3
EXREF         0.00 ppm
BF            0.12 Hz
RGAIN           50
```



19b




```
DFILE  19b C.als
COMNT  single pulse dec
DATIM  2015-08-09 19:16
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP         22.4 c
SLVNT  CDCL3
EXREF        77.00 ppm
BF            1.20 Hz
RGAIN           60
```



19b

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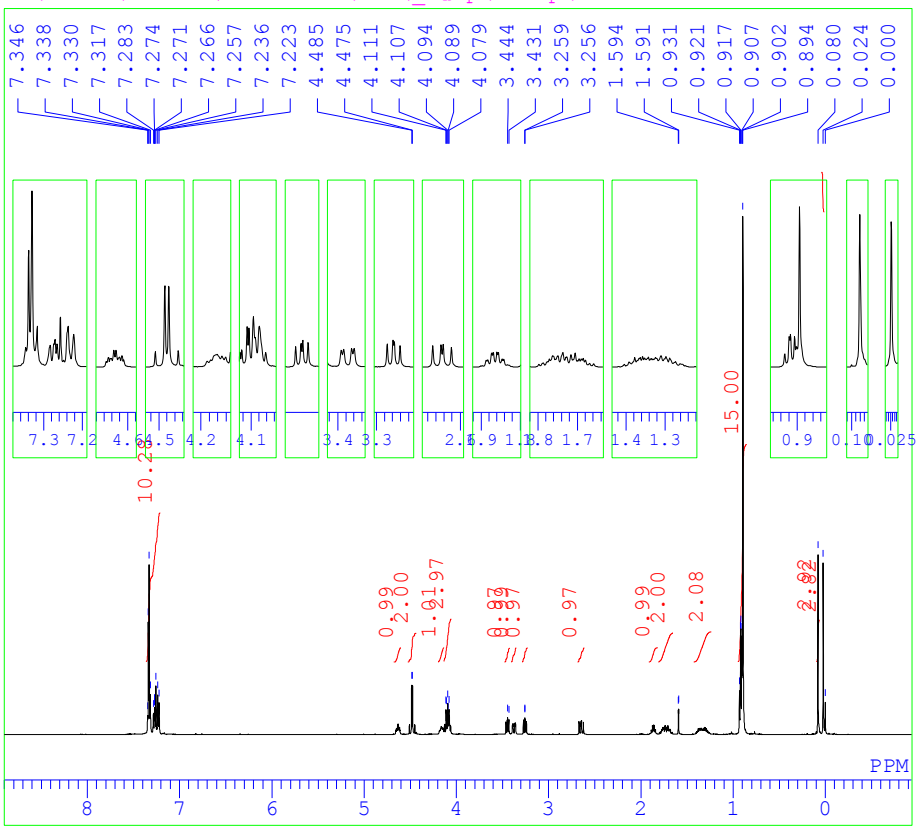
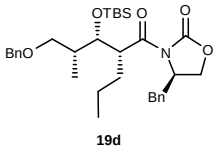


DFILE 19d H.als
COMNT single_pulse
DATIM 2015-06-25 01:45
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40



19d

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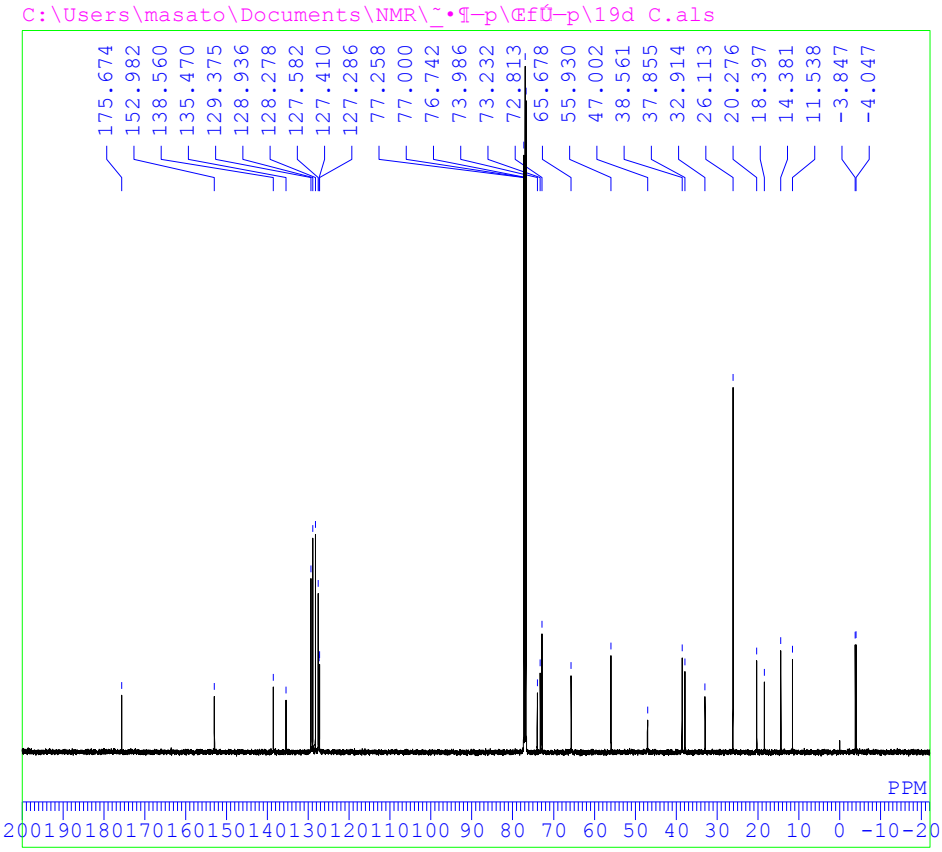
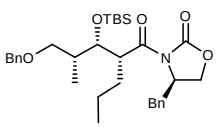
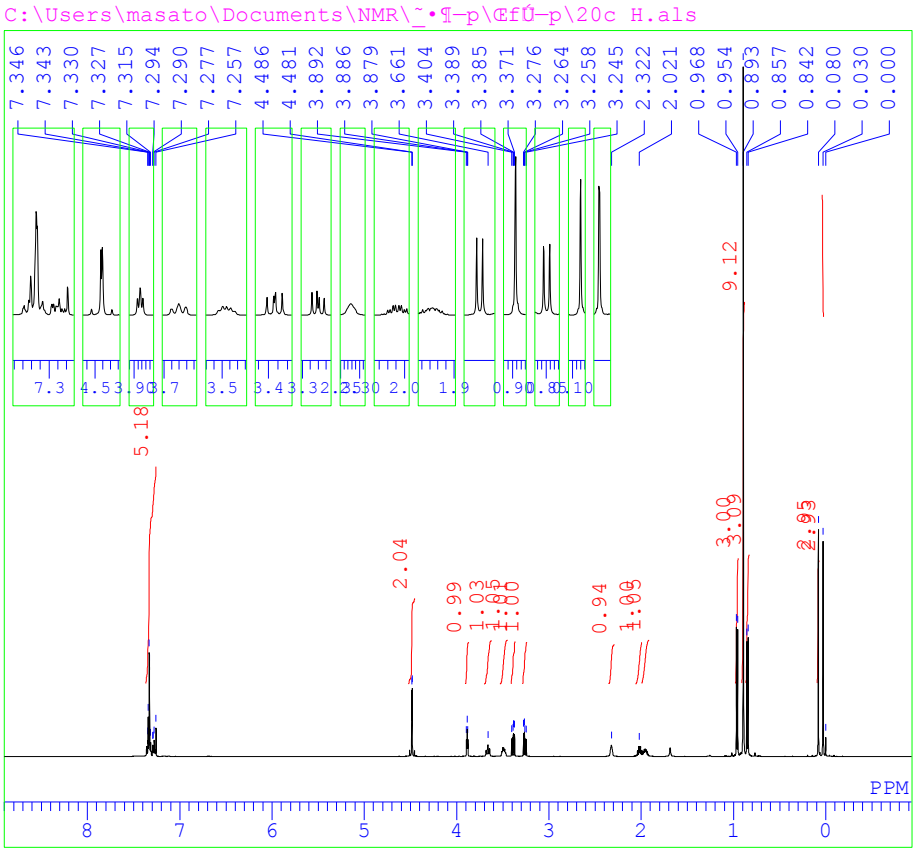


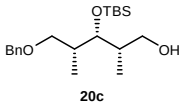
DFILE 19d C.als
COMNT single pulse dec
DATIM 2015-06-25 03:20
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 2000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



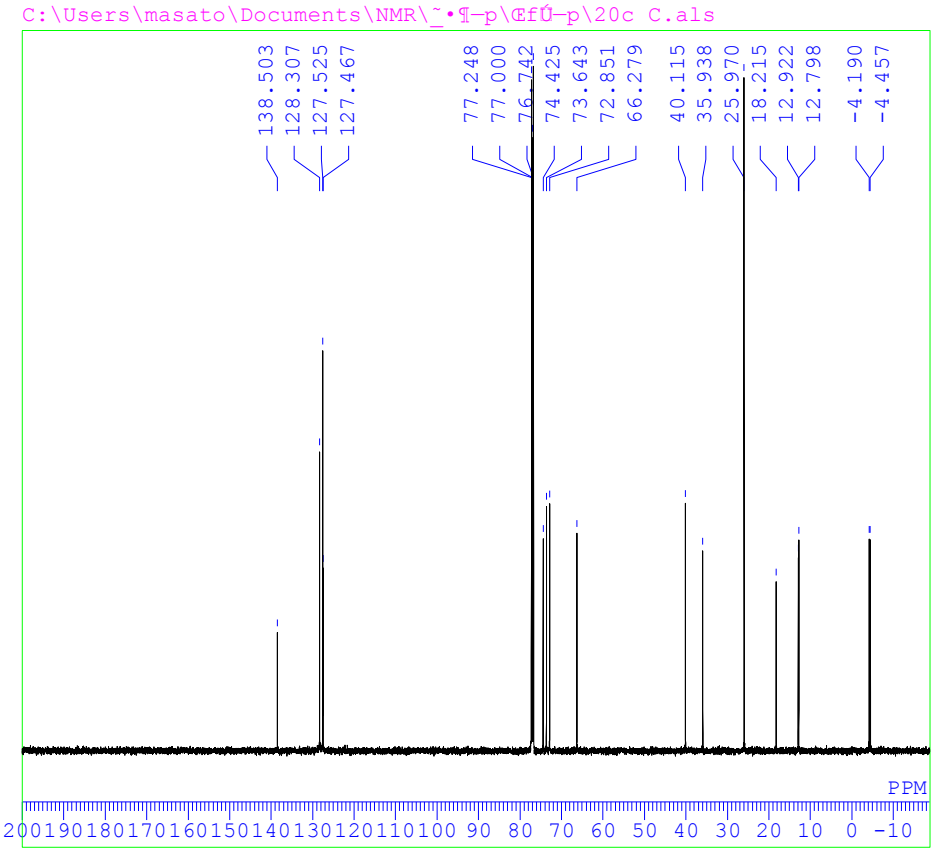
19d



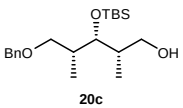
DFILE 20c H.als
COMNT single_pulse
DATIM 2015-04-06 16:17
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36



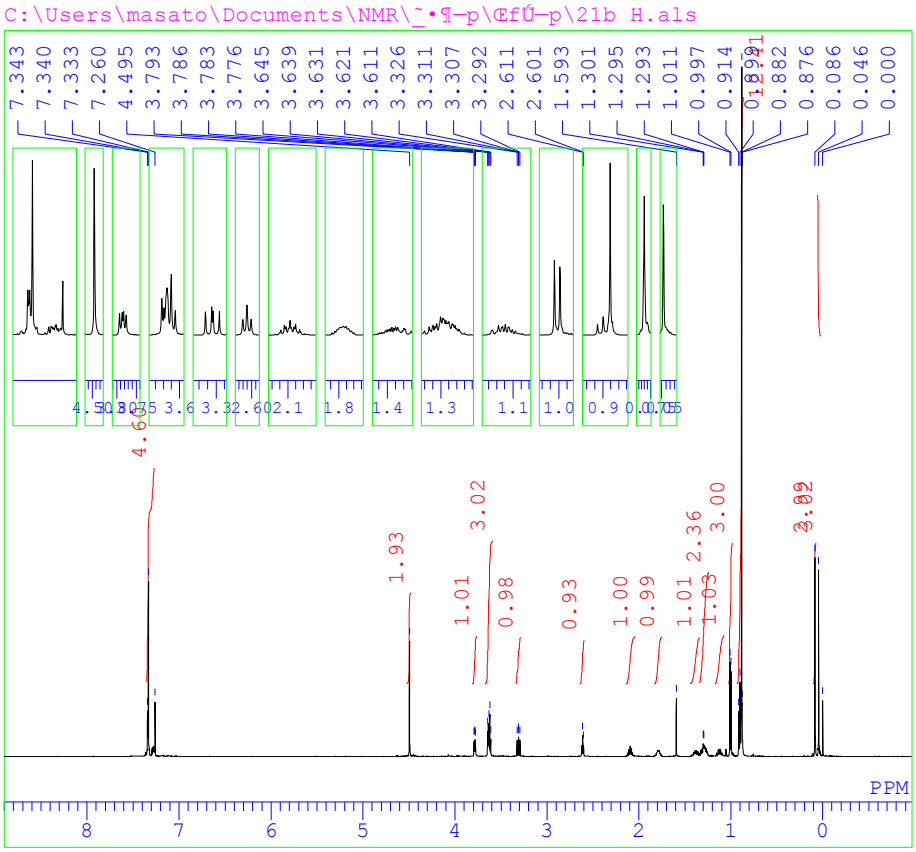
20c



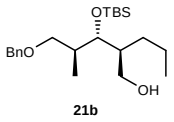
DFILE 20c C.als
COMNT single pulse dec
DATIM 2015-04-07 20:54
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 50



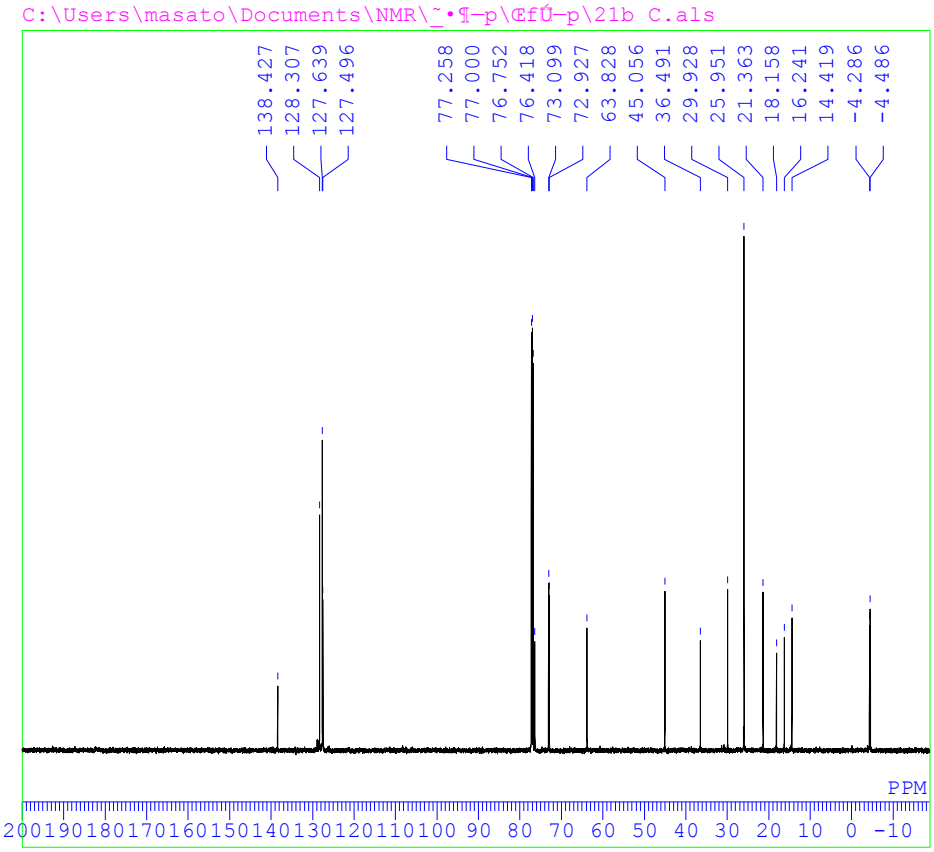
20c



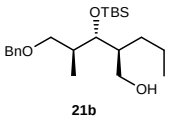
DFILE 21b H.als
COMNT single_pulse
DATIM 2015-12-20 16:37
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42



21b



DFILE 21b C.als
COMNT single pulse dec
DATIM 2015-08-06 10:21
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56



21b

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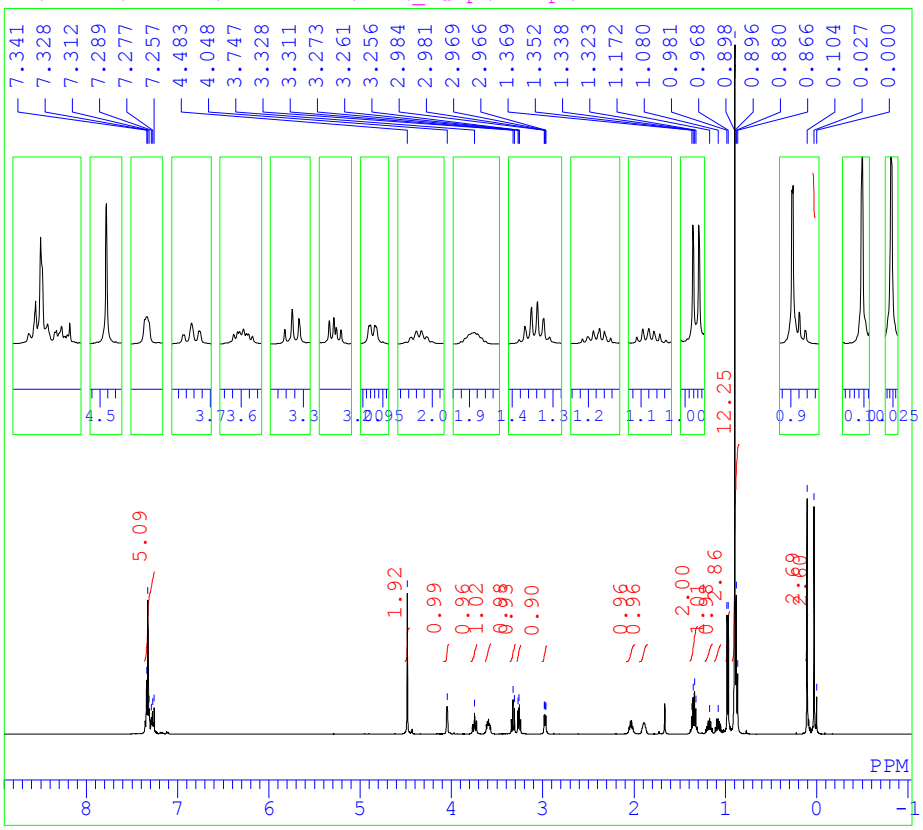
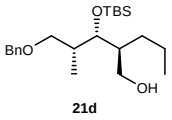


DFILE 21d H.als
COMNT single_pulse
DATIM 2015-06-25 03:29
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38



21d

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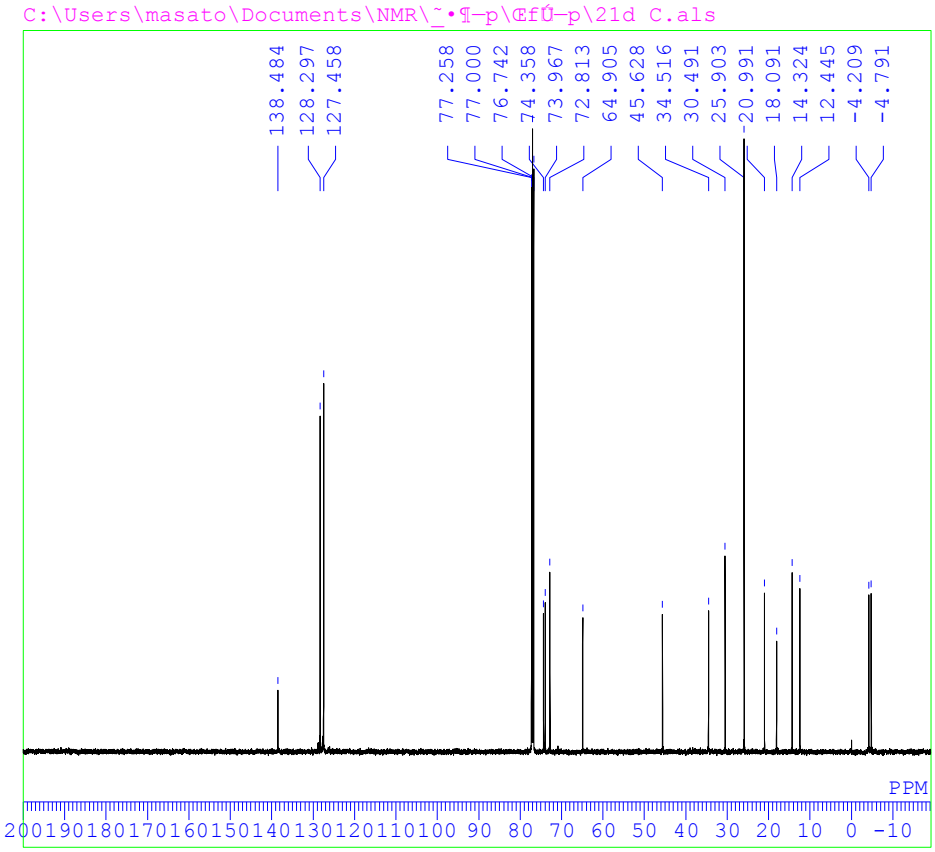
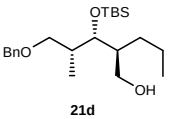
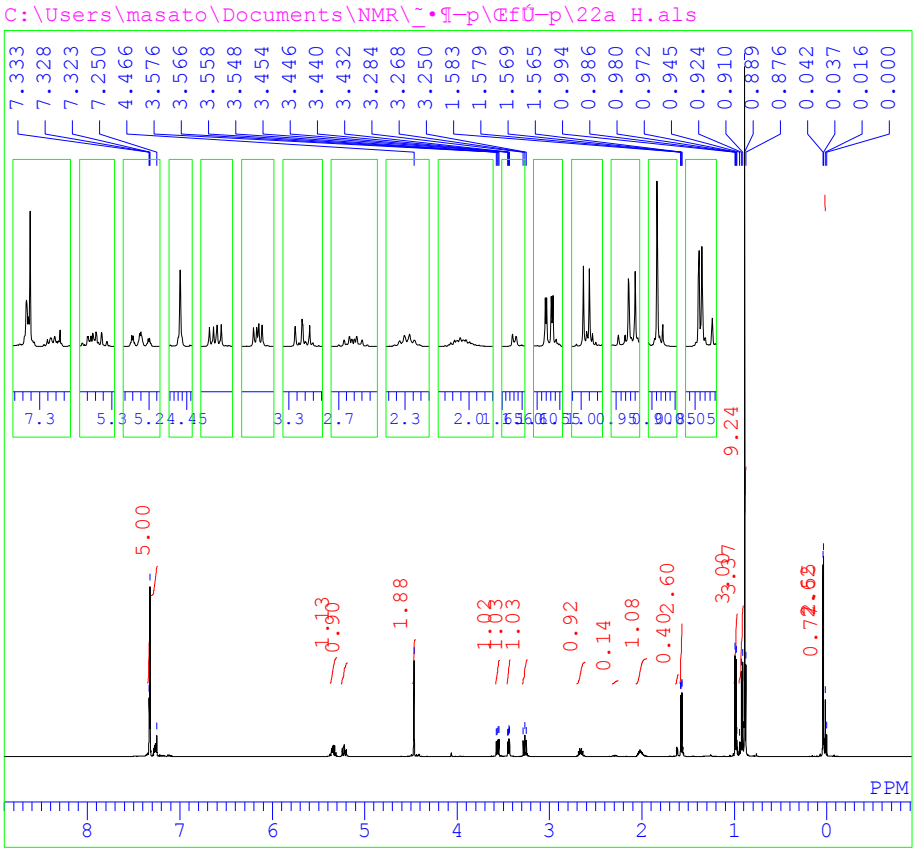


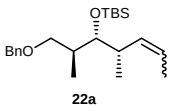
DFILE 21d C.als
COMNT single pulse dec
DATIM 2015-06-25 05:05
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 2000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



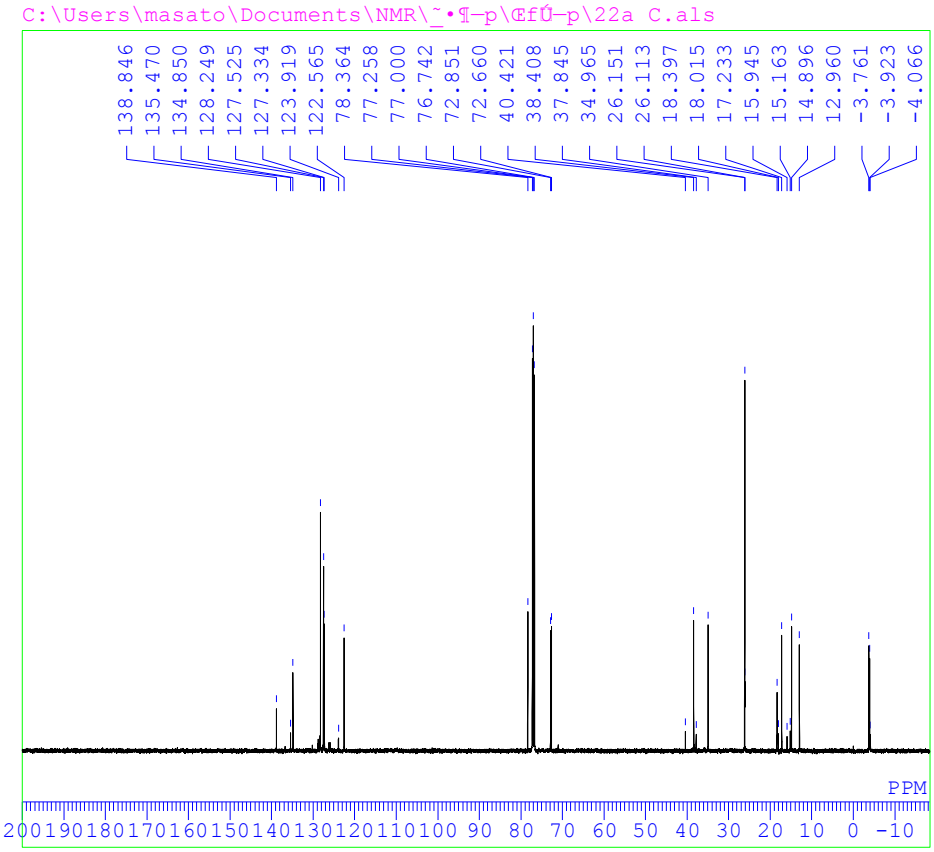
21d



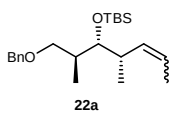
DFILE 22a H.als
COMNT single_pulse
DATIM 2016-01-13 19:52
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 40
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34



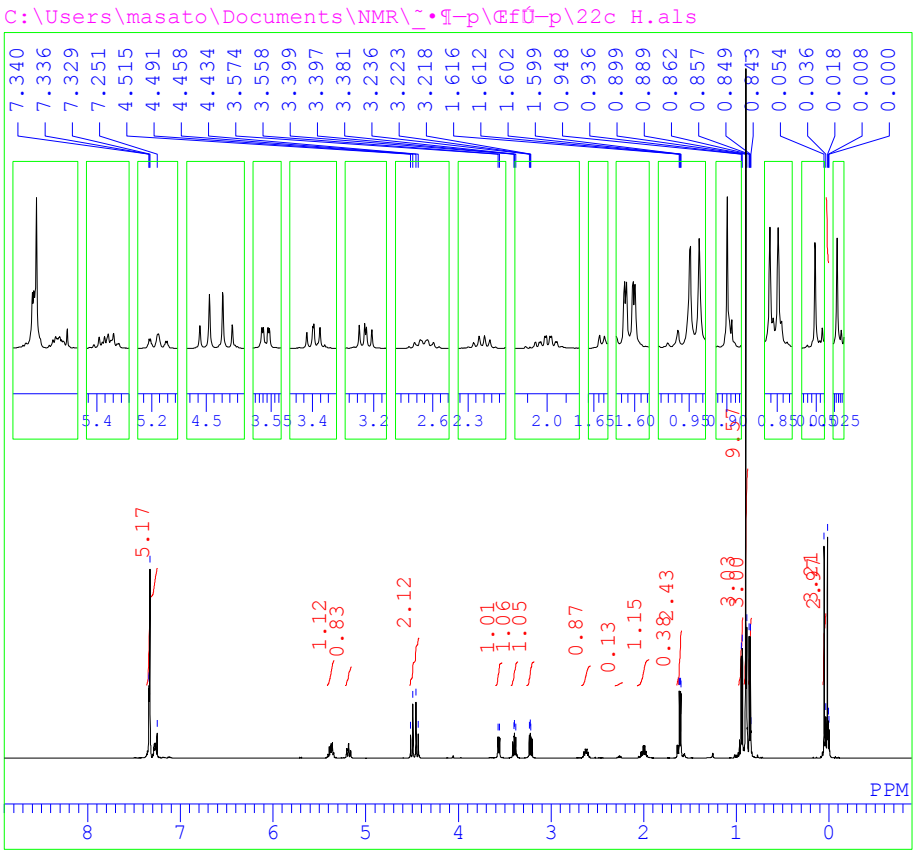
22a



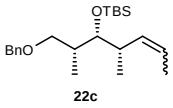
DFILE 22a C.als
COMNT single pulse dec
DATIM 2016-01-13 19:40
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 20.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 52



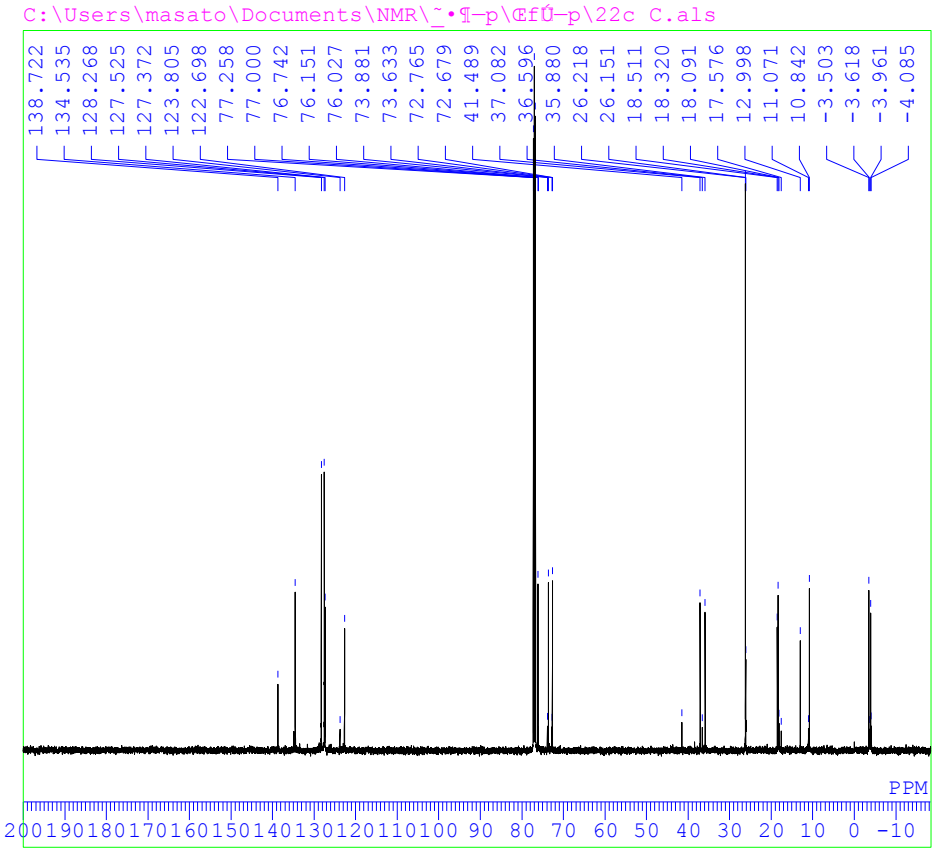
22a



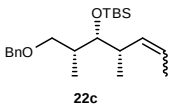

```
DFILE  22c H.als
COMNT  single_pulse
DATIM  2016-01-14 15:29
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          19.6 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            36
```



22c

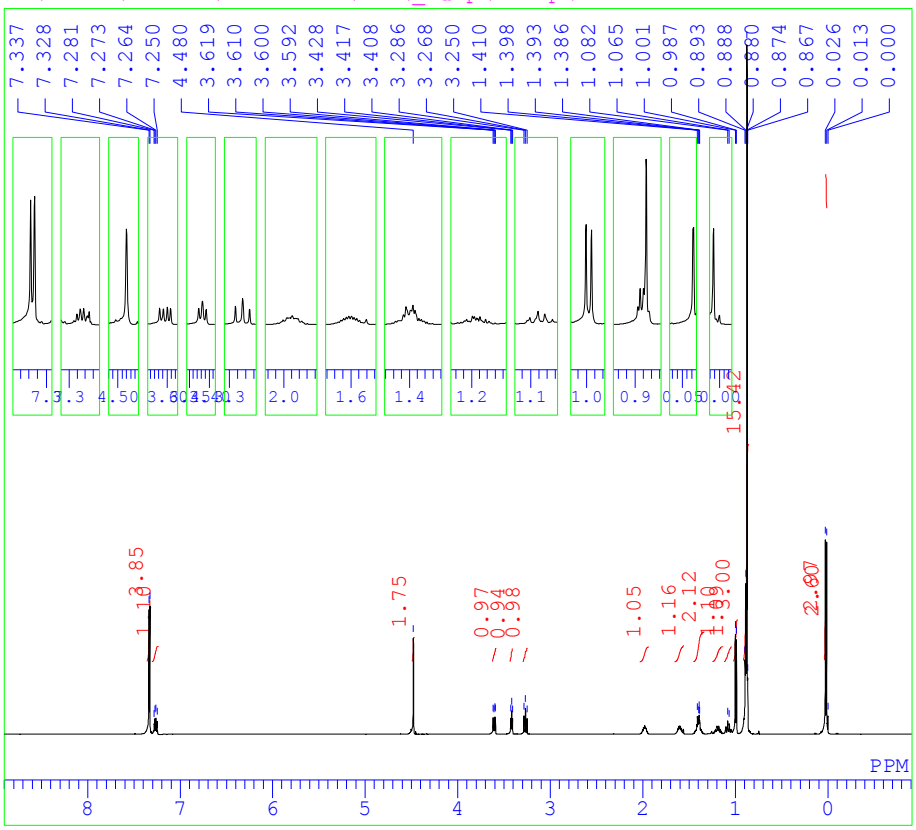



```
DFILE  22c C.als
COMNT  single pulse dec
DATIM  2016-01-14 17:01
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          20.1 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            54
```

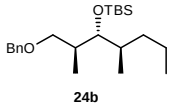


22c

C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\24b H.als

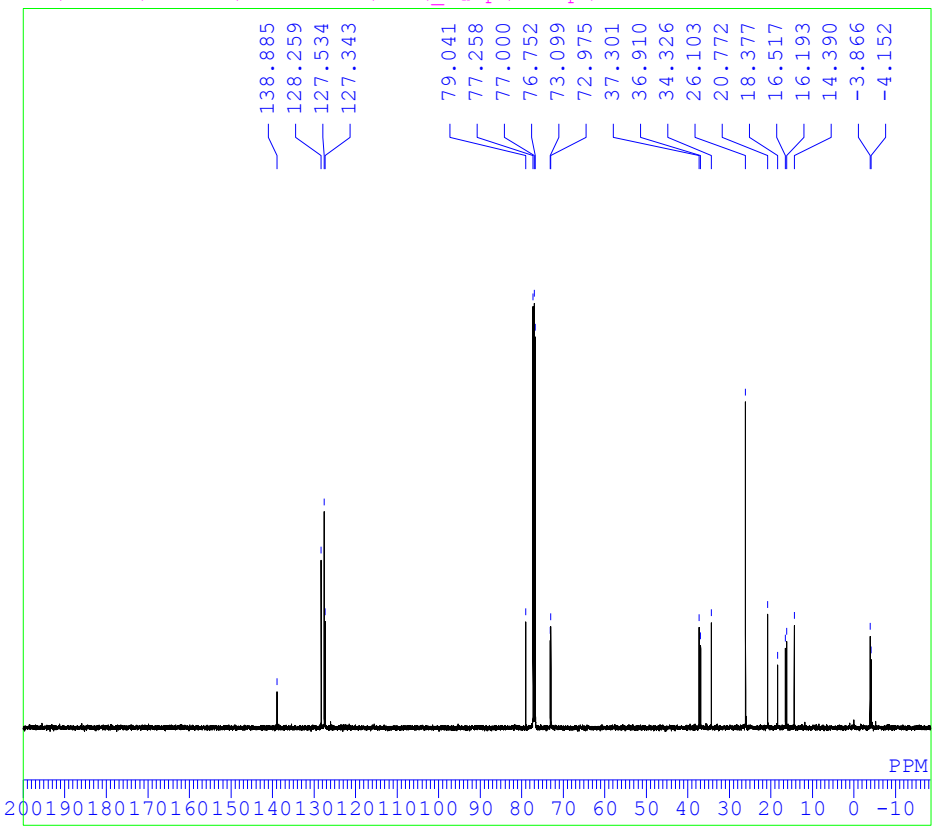


DFILE 24b H.als
COMNT single_pulse
DATIM 2016-01-13 14:31
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32

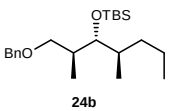


24b

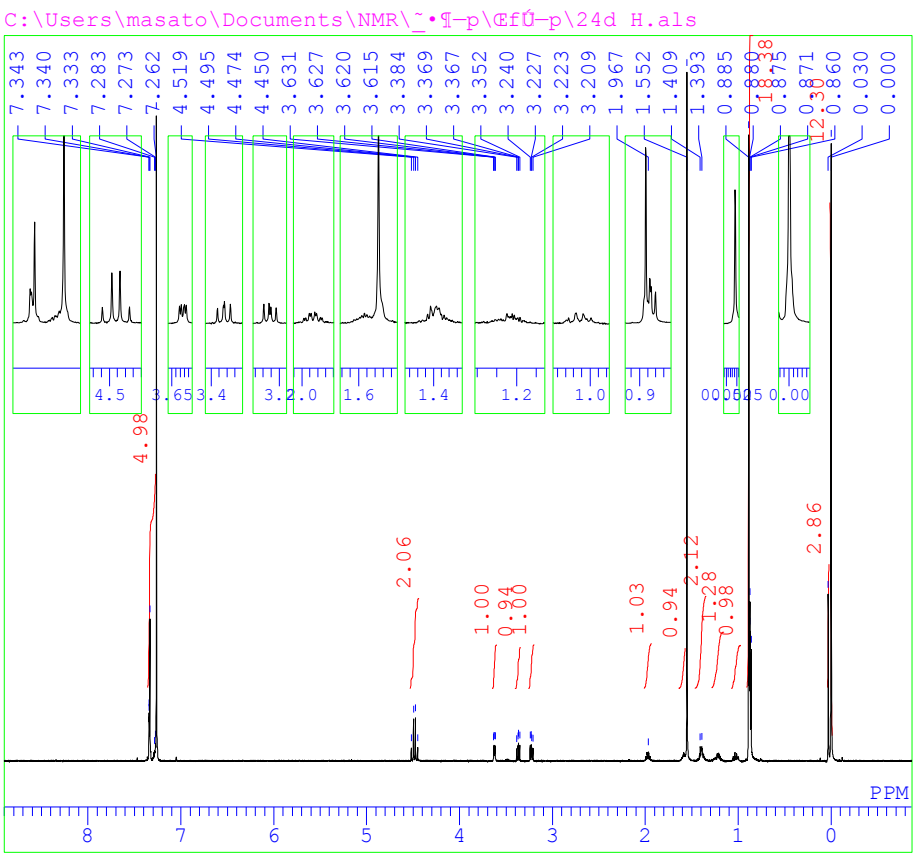
C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\24b C.als



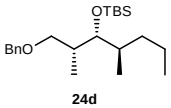
DFILE 24b C.als
COMNT single pulse dec
DATIM 2014-08-29 15:26
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 23.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56



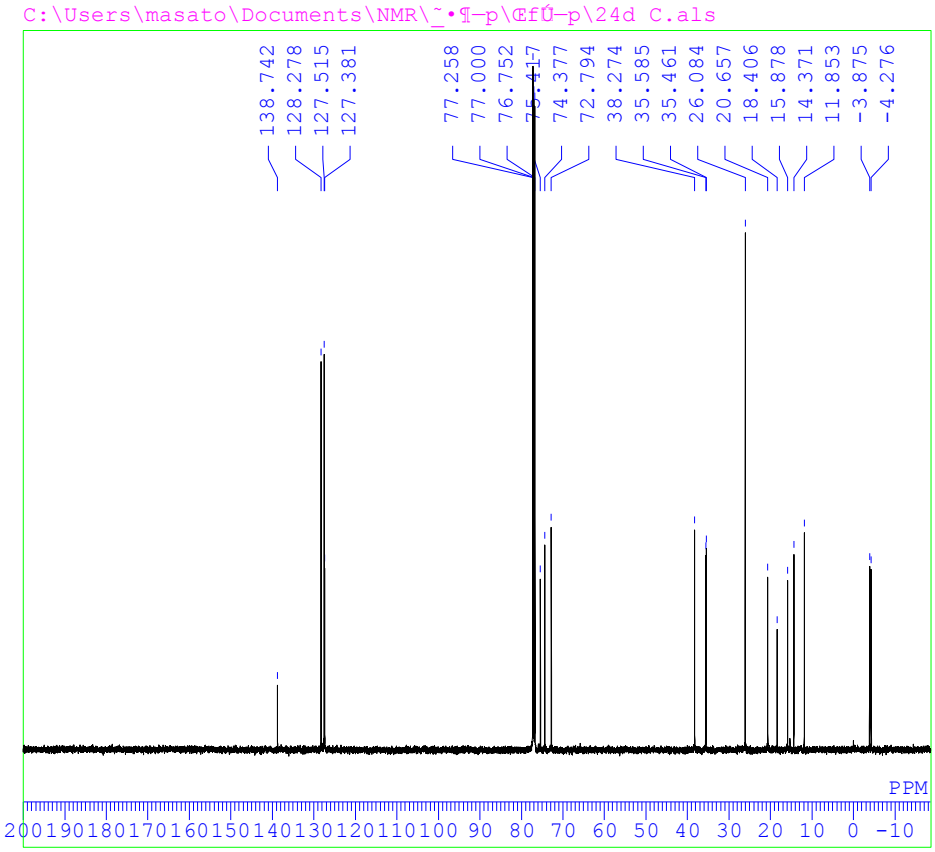
24b



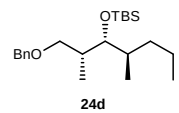
DFILE 24d H.als
COMNT single_pulse
DATIM 2015-12-17 10:34
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54



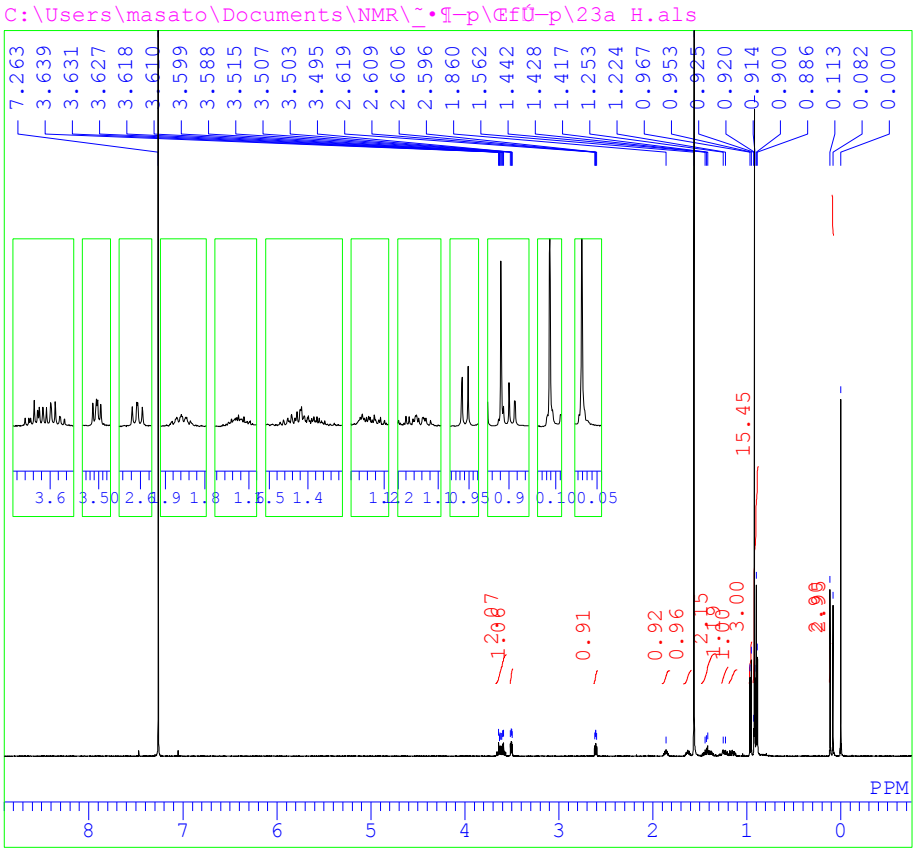
24d



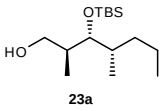
DFILE 24d C.als
COMNT single pulse dec
DATIM 2015-12-17 20:07
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 21.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



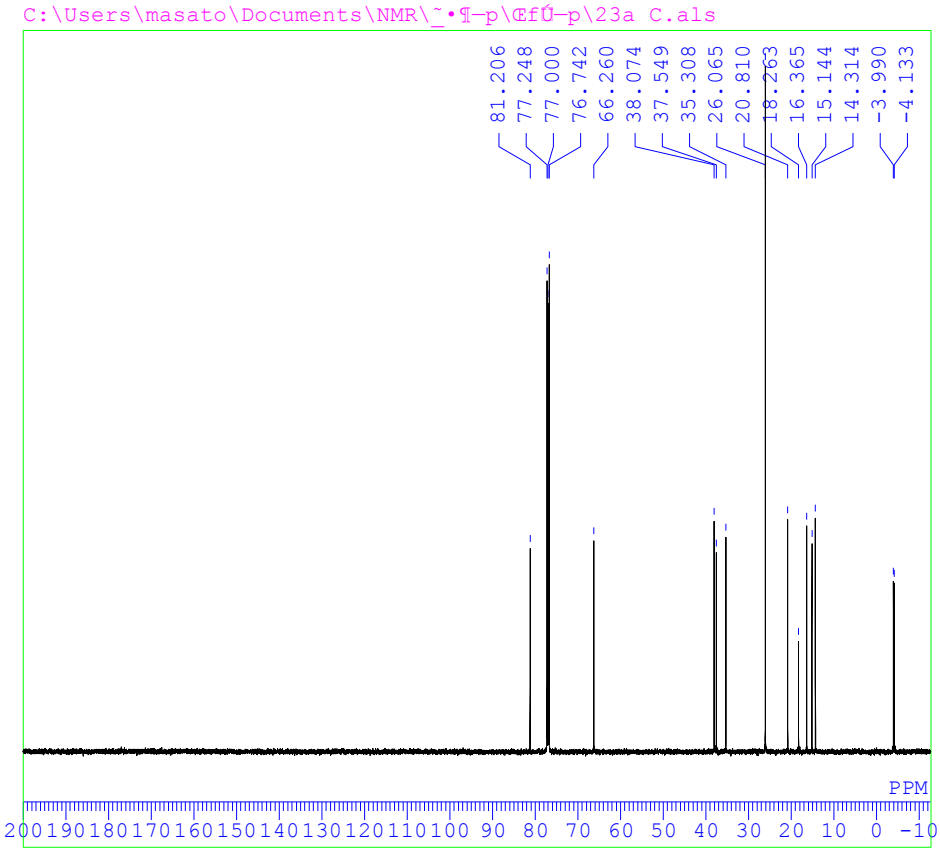
24d



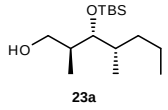
DFILE 23a H.als
COMNT single_pulse
DATIM 2015-05-25 13:01
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54



23a



DFILE 23a C.als
COMNT single pulse dec
DATIM 2015-05-25 17:51
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 52



23a

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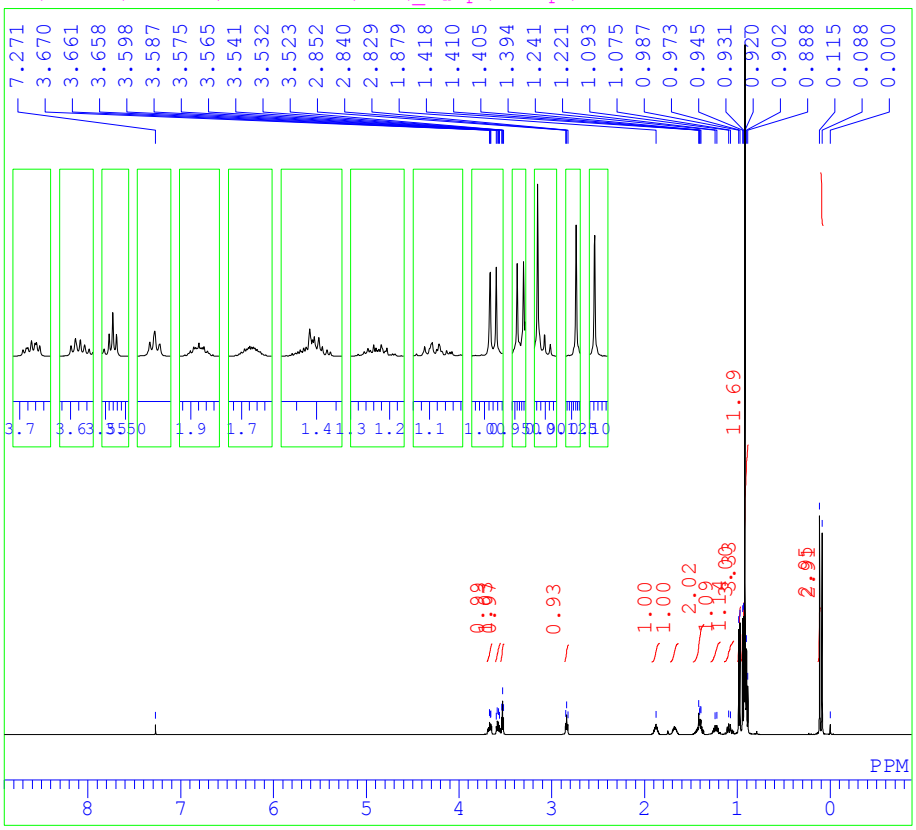
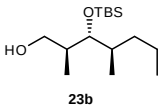


DFILE 23b H.als
COMNT single_pulse
DATIM 2016-01-19 14:03
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 19.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32



23b

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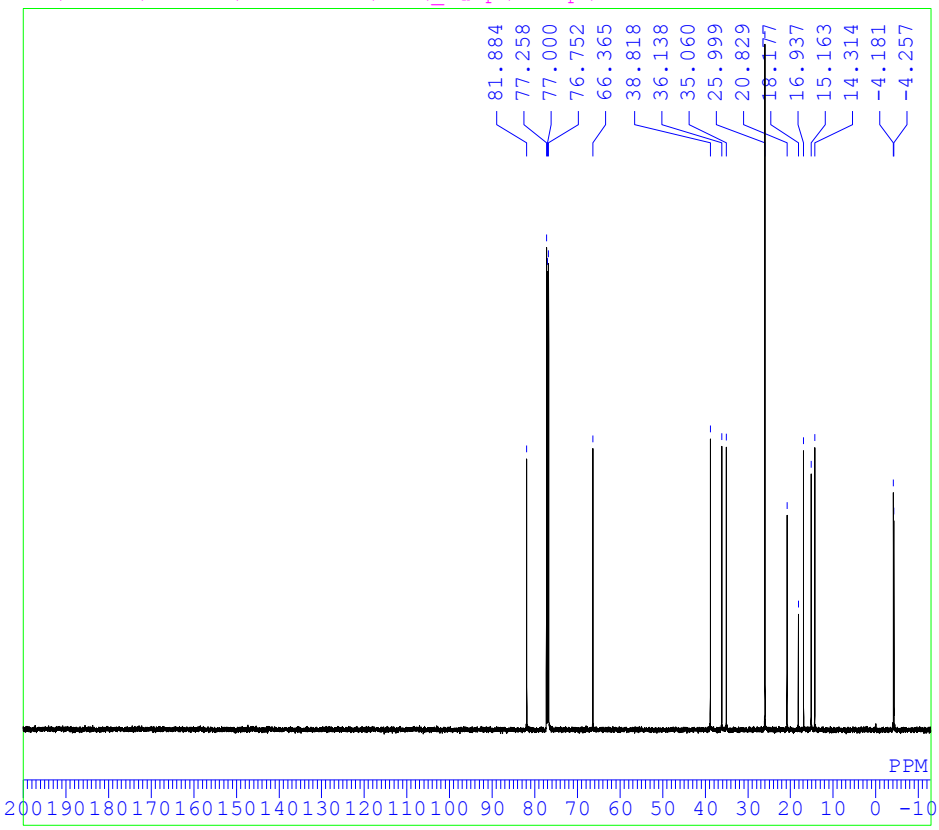
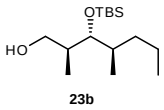
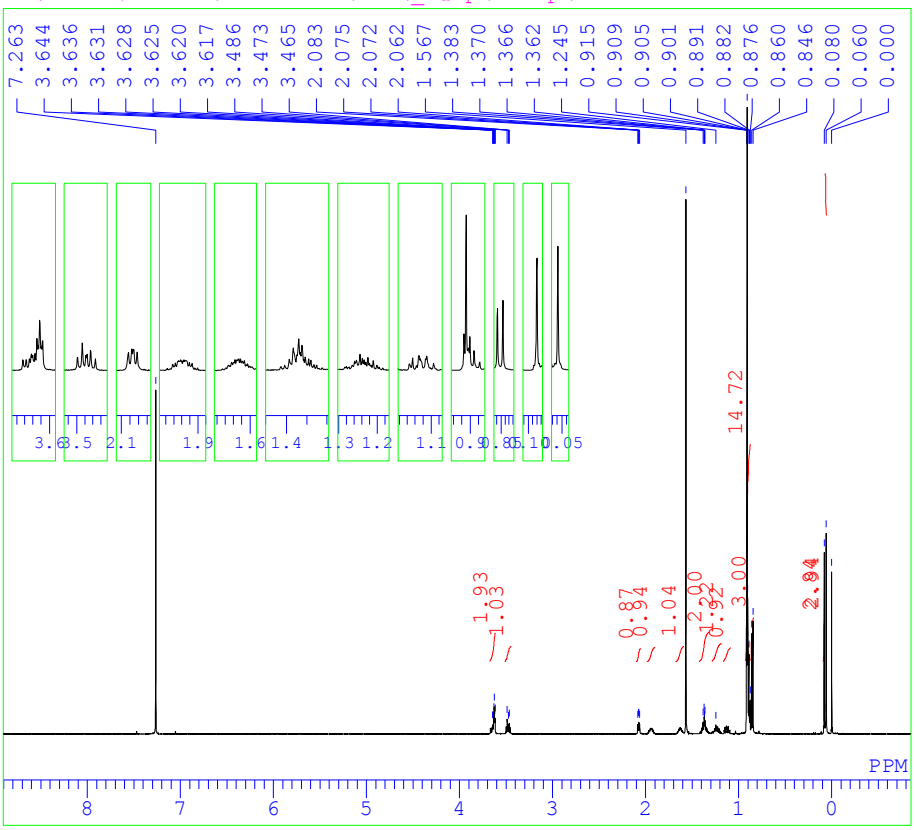


DFILE 23b C.als
COMNT single pulse dec
DATIM 2016-01-19 16:07
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 948
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 20.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56

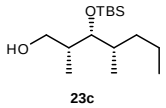


23b

C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\23c H.als

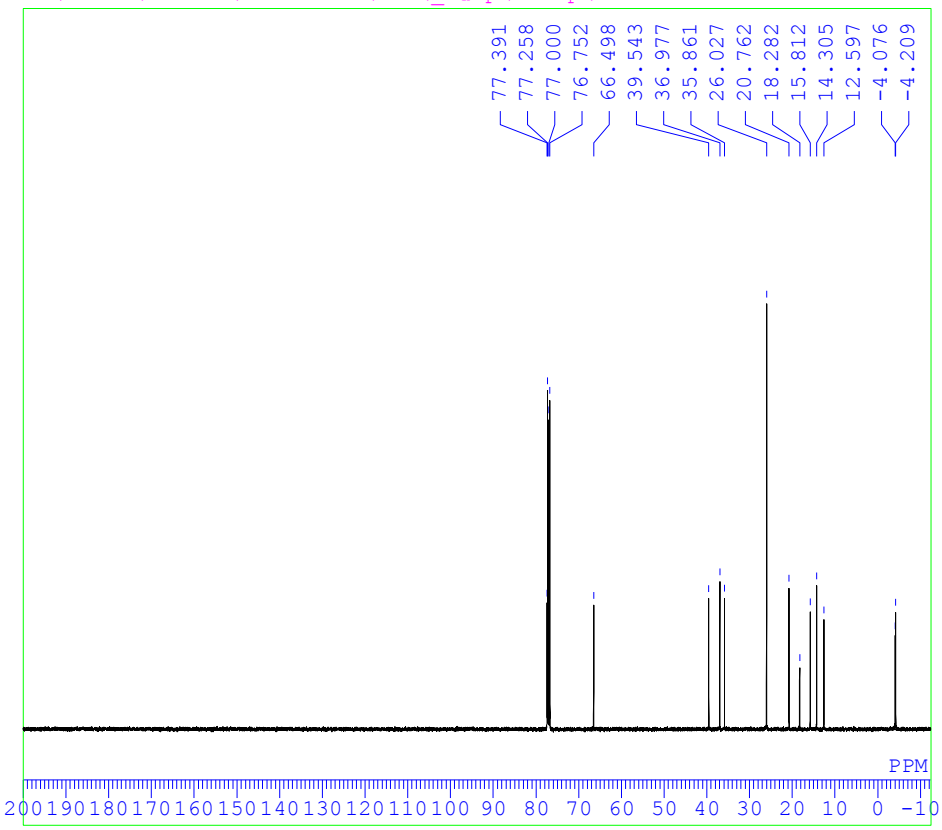


DFILE 23c H.als
COMNT single_pulse
DATIM 2015-05-25 13:15
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50

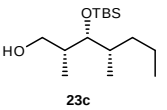


23c

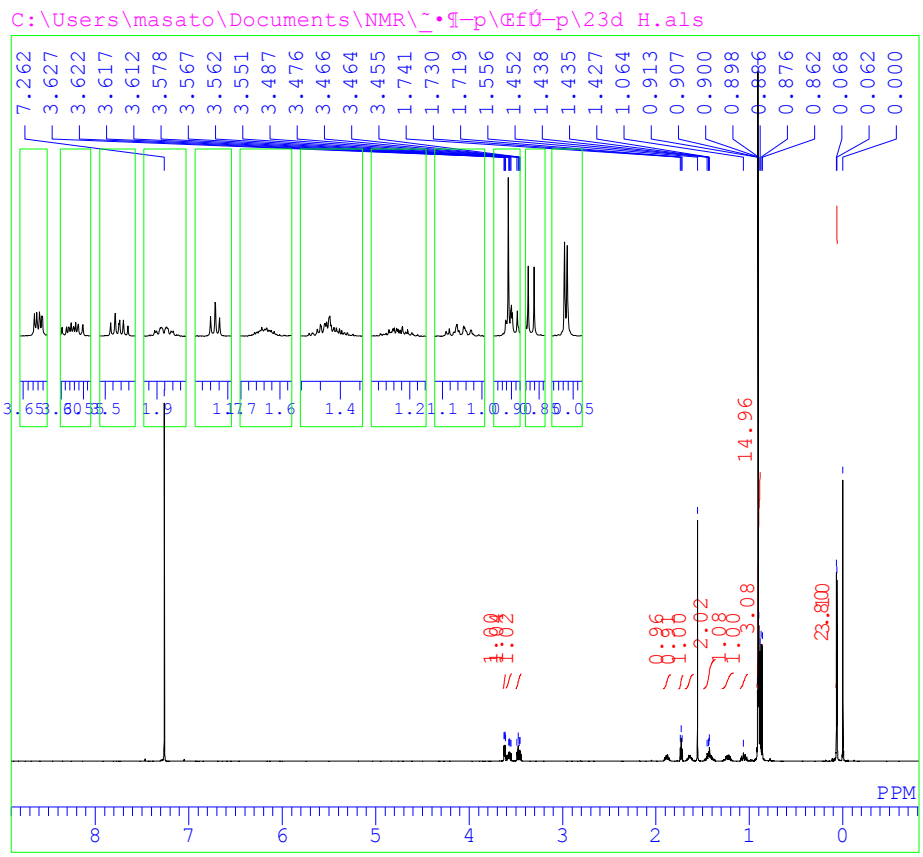
C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\23c C.als



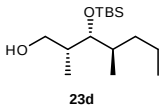
DFILE 23c C.als
COMNT single pulse dec
DATIM 2015-05-25 20:21
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 50



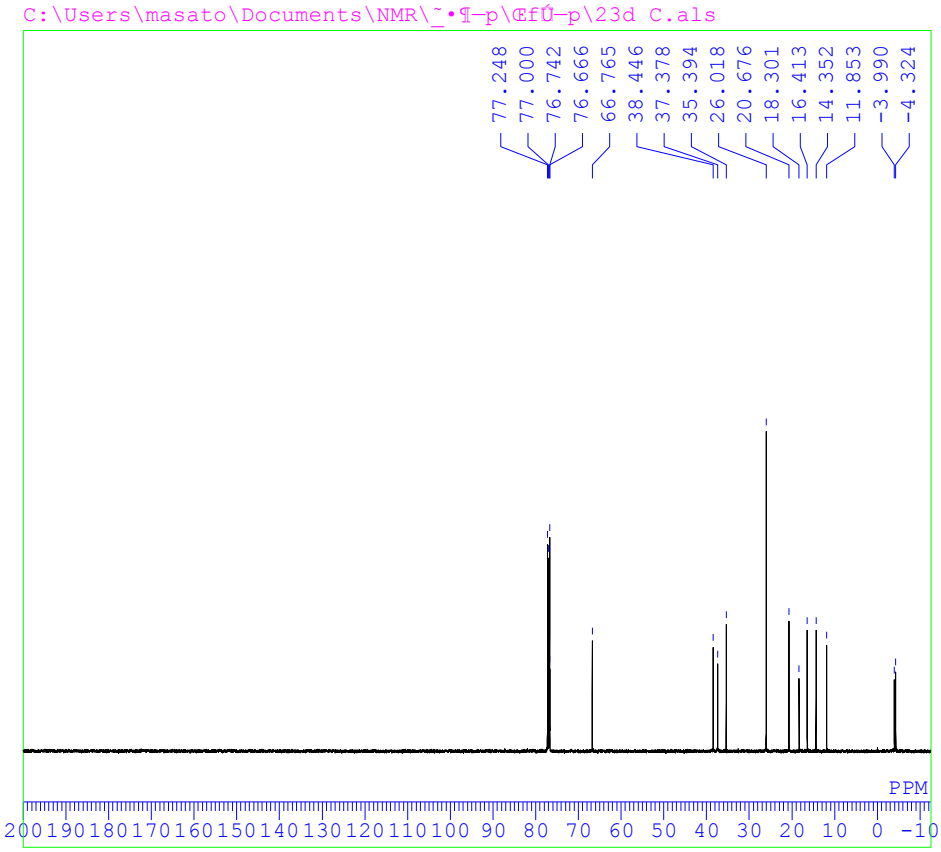
23c



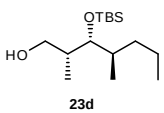
DFILE 23d H.als
COMNT single_pulse
DATIM 2015-02-21 11:32
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 50



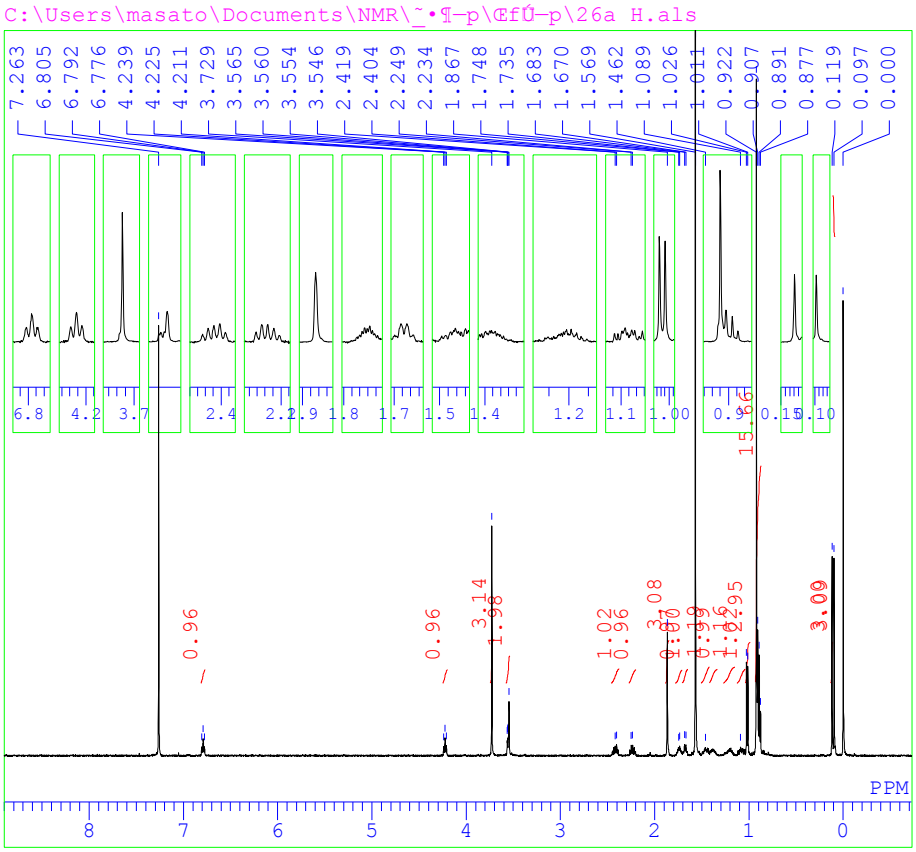
23d



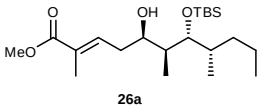
DFILE 23d C.als
COMNT single pulse dec
DATIM 2015-12-15 21:49
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 575
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 50



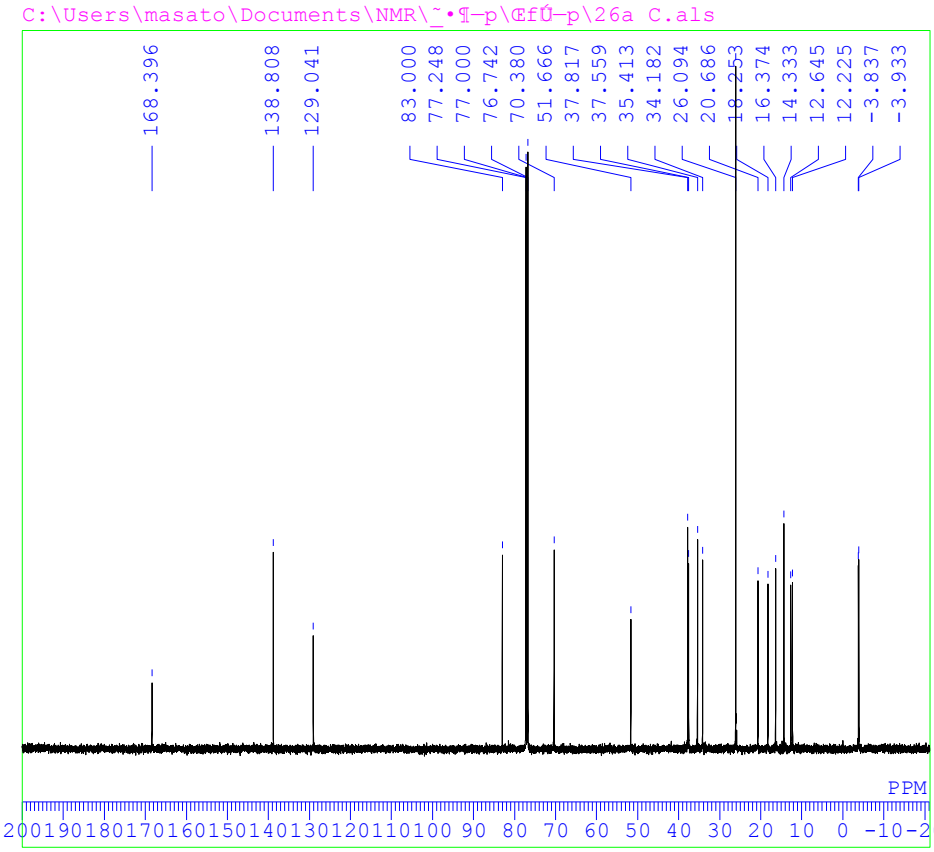
23d



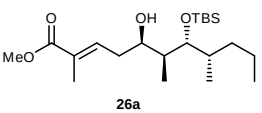

```
DFILE  26a H.als
COMNT  single_pulse
DATIM  2015-09-07 09:32
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.2 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            54
```



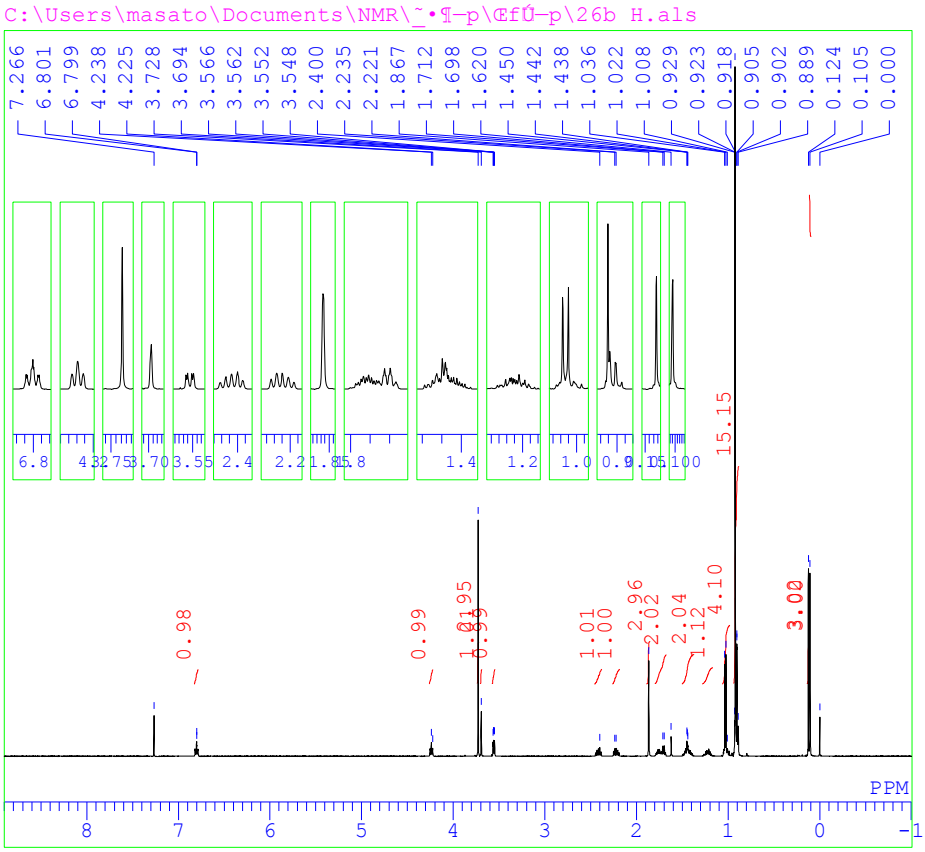
26a



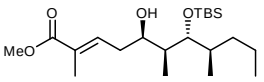

```
DFILE  26a C.als
COMNT  single pulse dec
DATIM  2015-10-02 13:28
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS           500
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.0 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            58
```



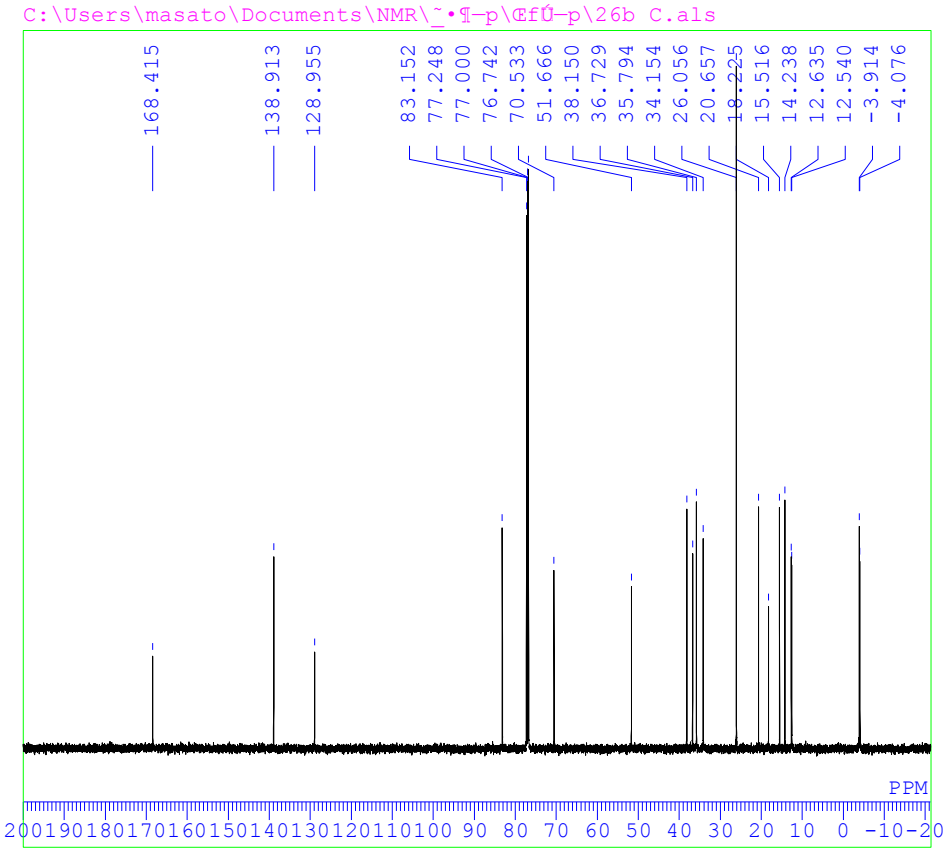
26a



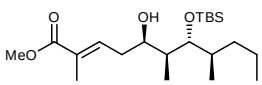
DFILE 26b H.als
COMNT single_pulse
DATIM 2015-12-05 14:44
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 20.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40



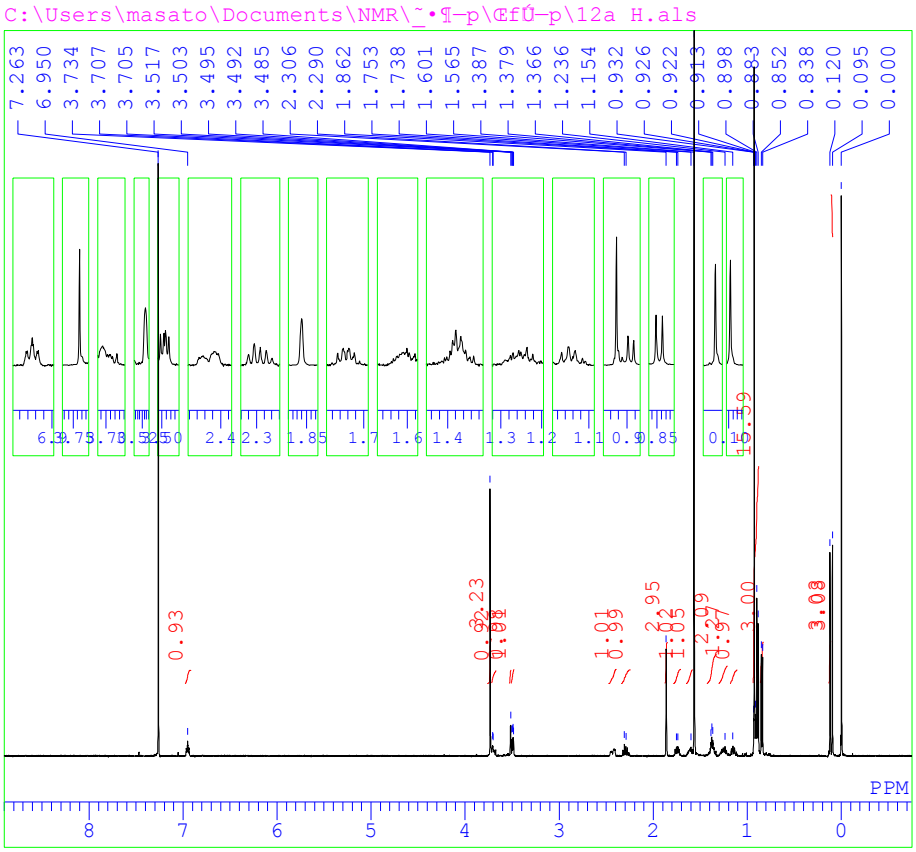
26b



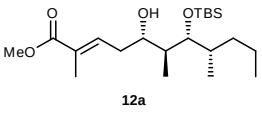
DFILE 26b C.als
COMNT single pulse dec
DATIM 2015-10-02 17:38
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 500
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 54



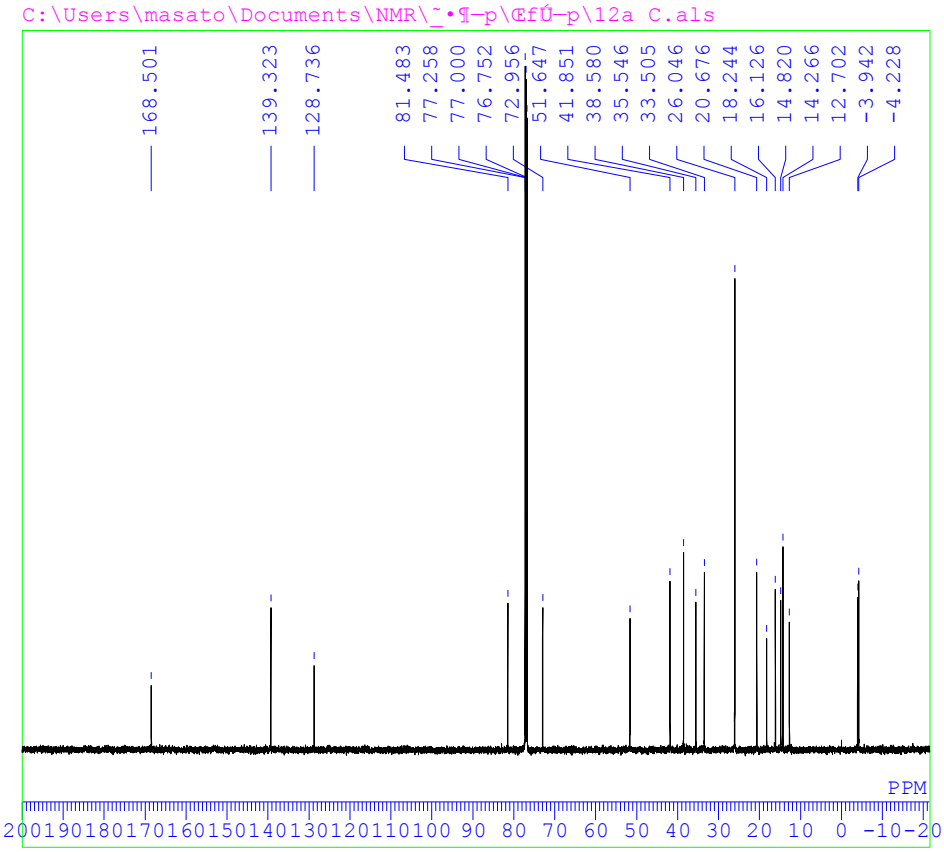
26b



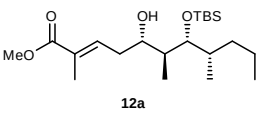
DFILE 12a H.als
COMNT single_pulse
DATIM 2015-09-18 12:28
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54



12a



DFILE 12a C.als
COMNT single pulse dec
DATIM 2015-09-25 10:56
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 54



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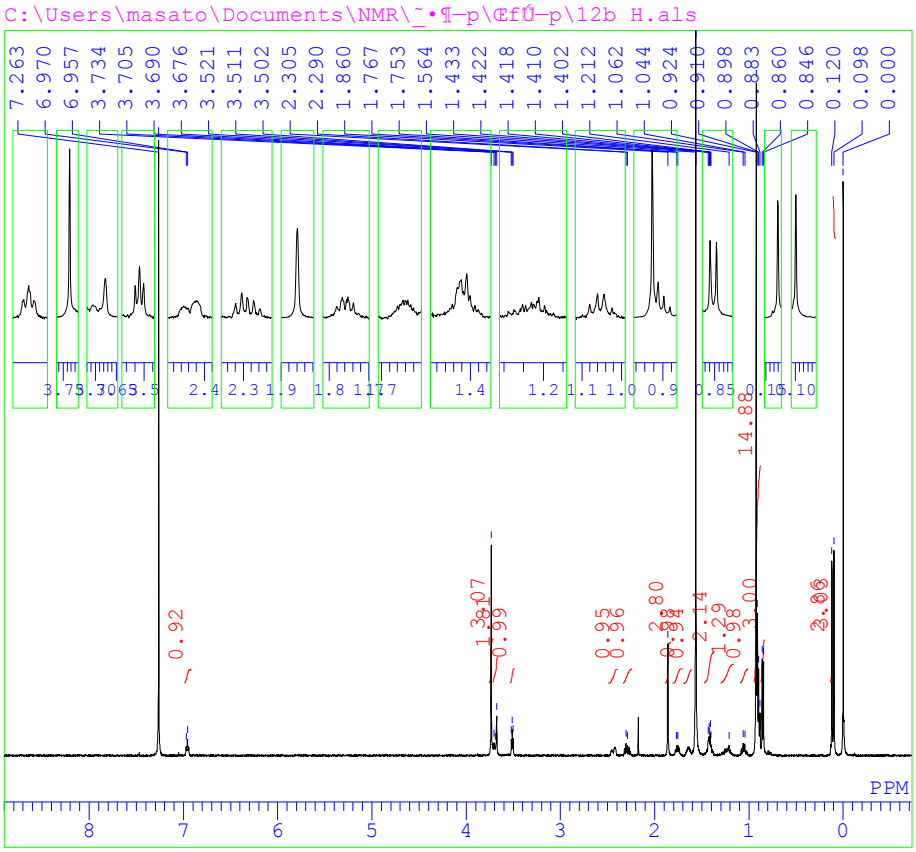
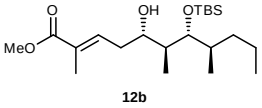
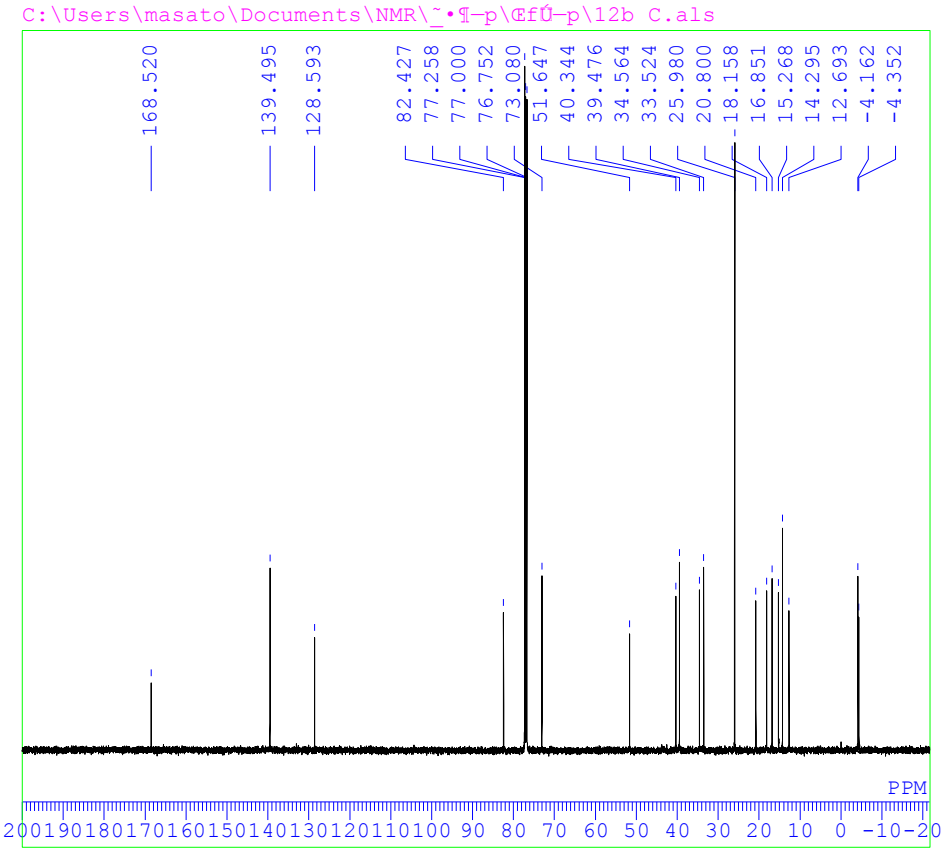


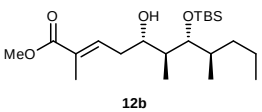

```
DFILE  12b H.als
COMNT  single_pulse
DATIM  2015-09-29 09:15
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.6 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            54
```



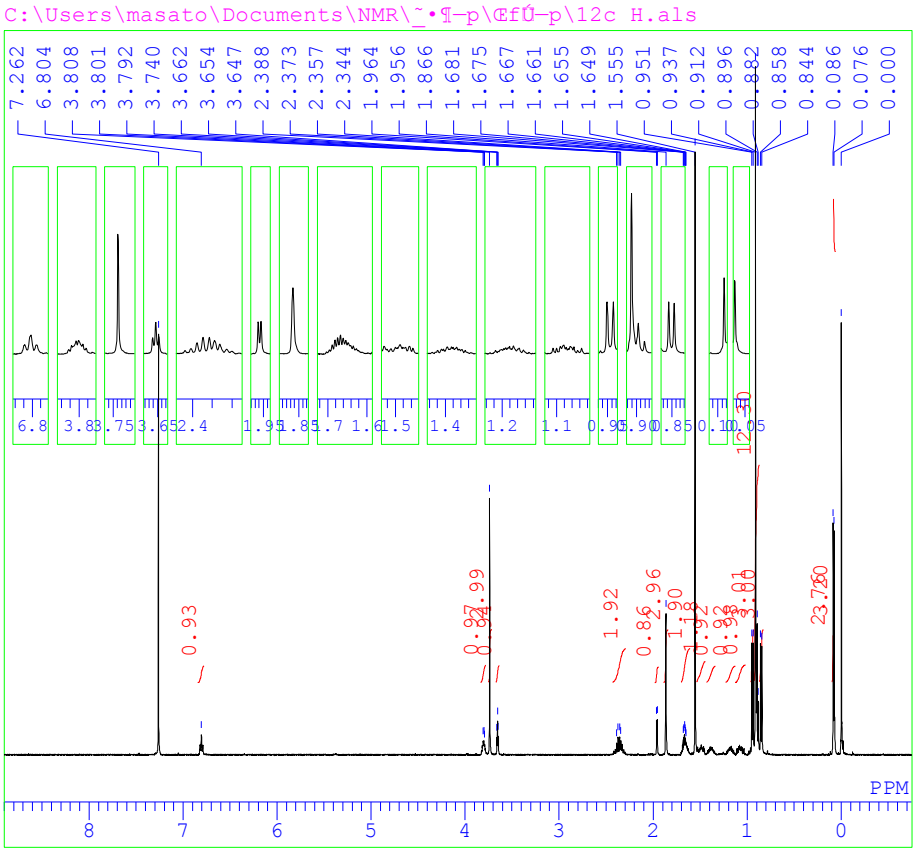
12b



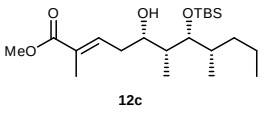

```
DFILE  12b C.als
COMNT  single pulse dec
DATIM  2015-09-30 16:51
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.0 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            58
```



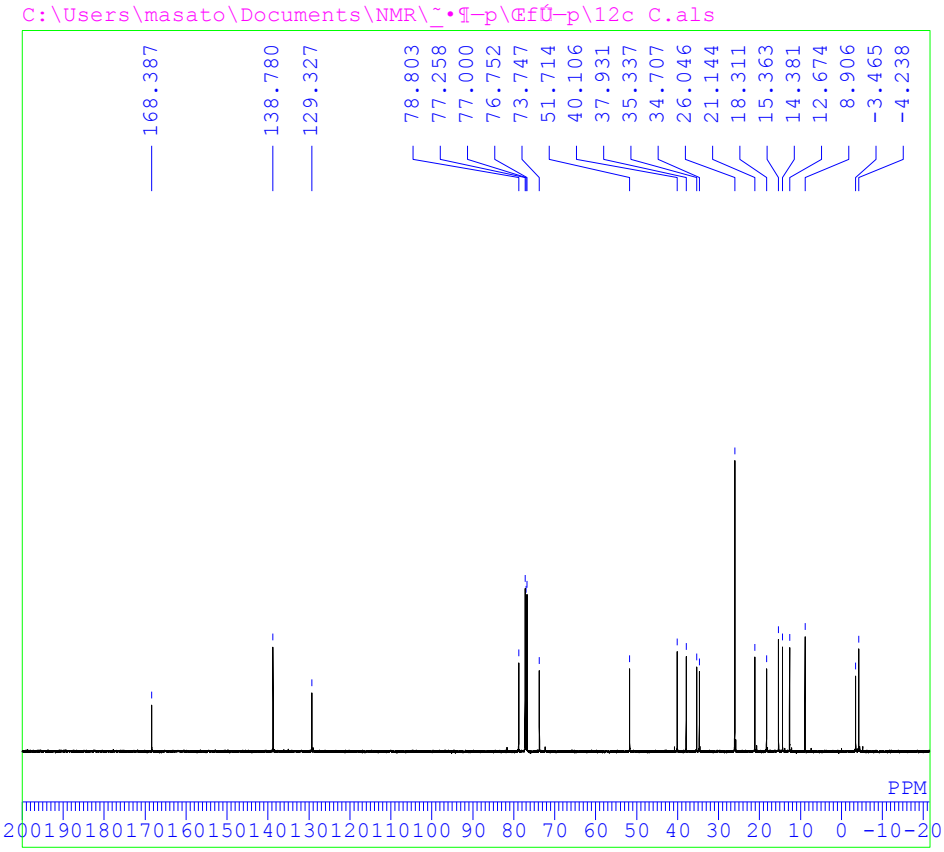
12b



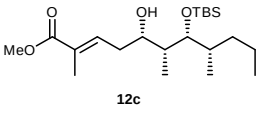

```
DFILE  12c H.als
COMNT  single_pulse
DATIM  2015-11-10 15:45
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.5 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            52
```



12c

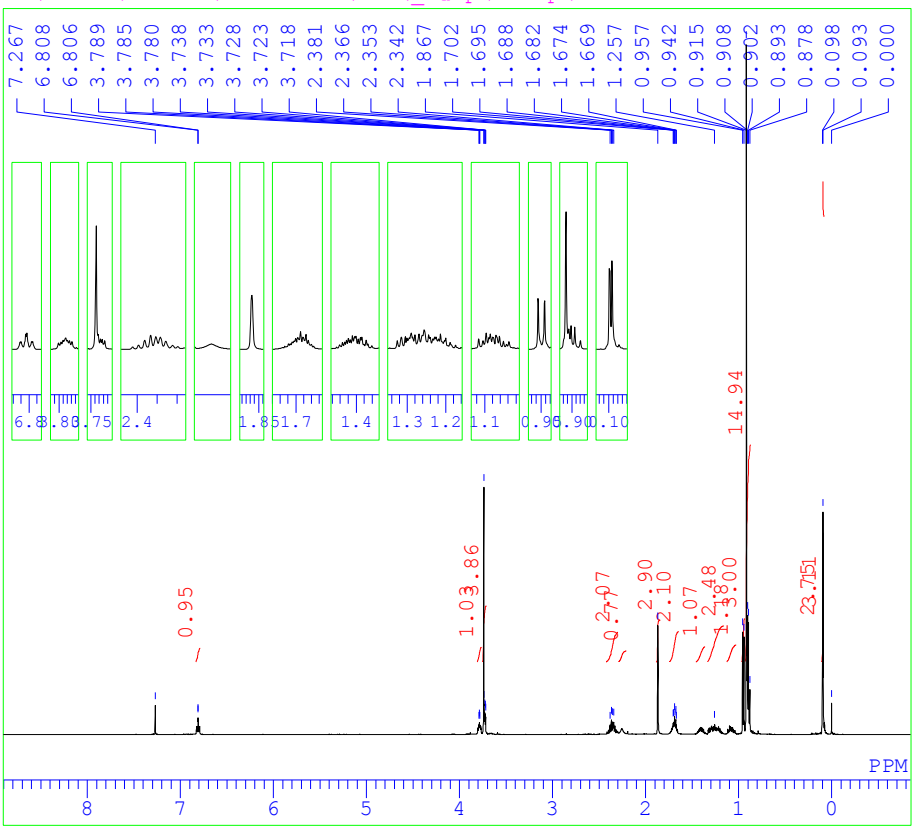



```
DFILE  12c C.als
COMNT  single pulse dec
DATIM  2015-11-10 15:40
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS           774
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.1 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            54
```

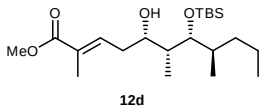


12c

C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\12d H.als

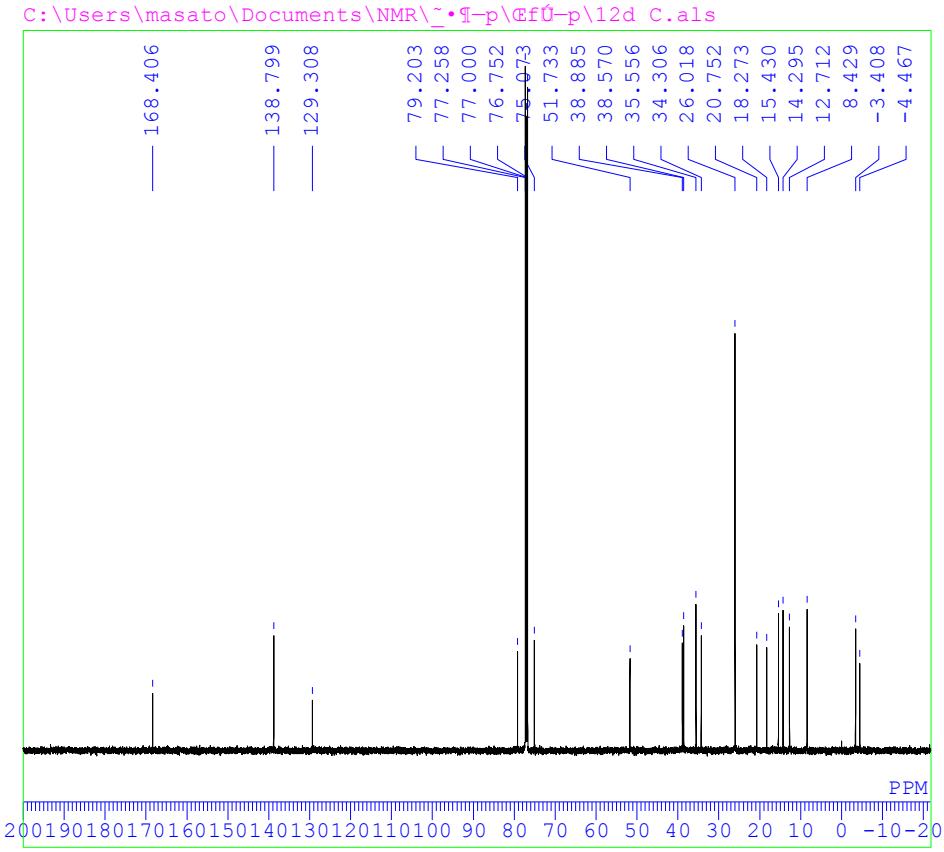


DFILE 12d H.als
COMNT single_pulse
DATIM 2016-01-15 13:55
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38

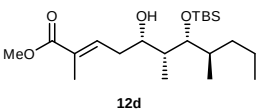


12d

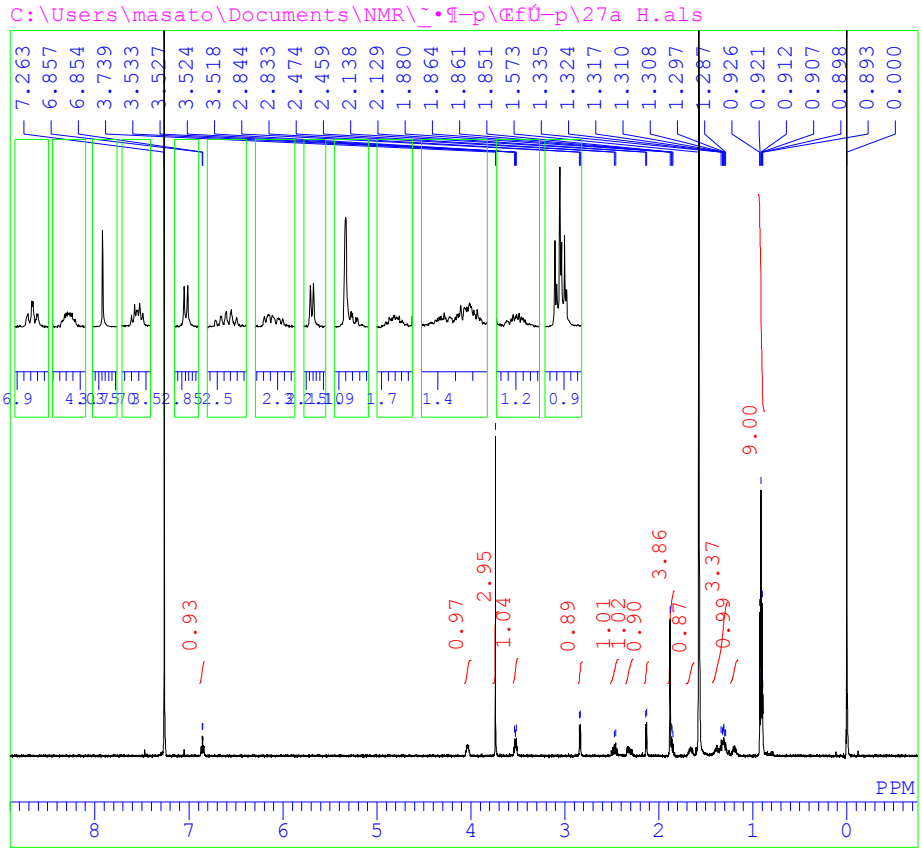
C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\12d C.als



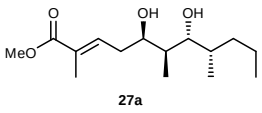
DFILE 12d C.als
COMNT single pulse dec
DATIM 2016-01-15 14:38
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 901
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 20.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56



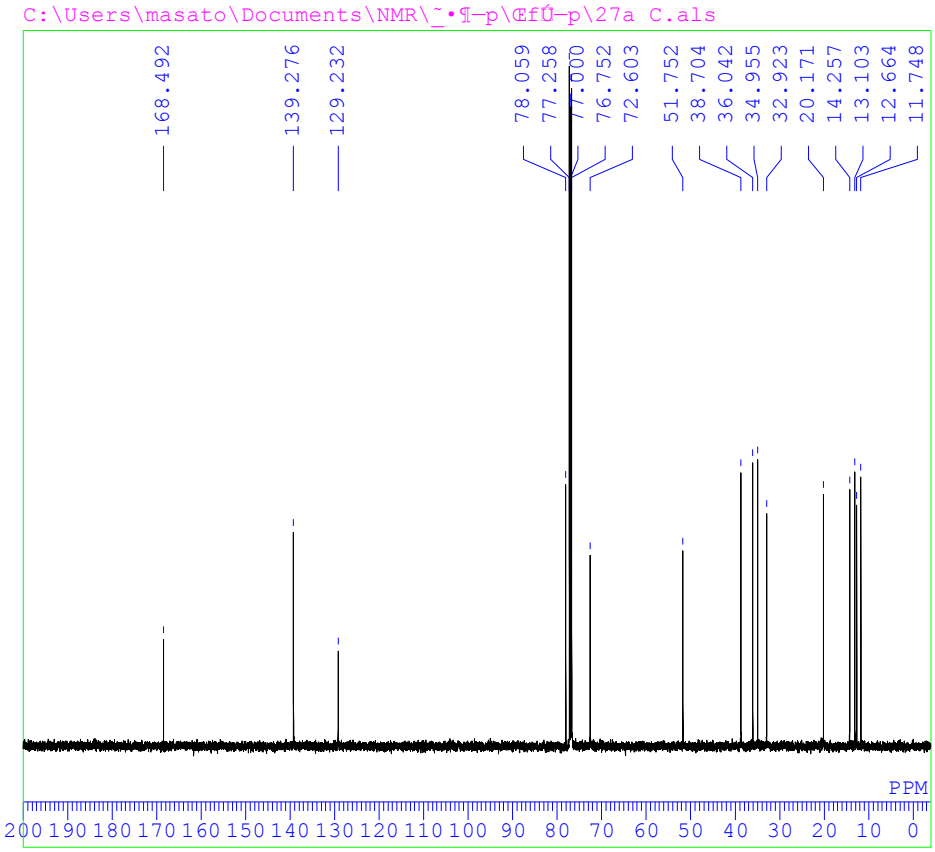
12d



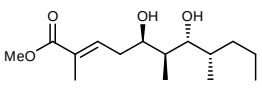
DFILE 27a H.als
COMNT single_pulse
DATIM 2015-09-08 09:22
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54



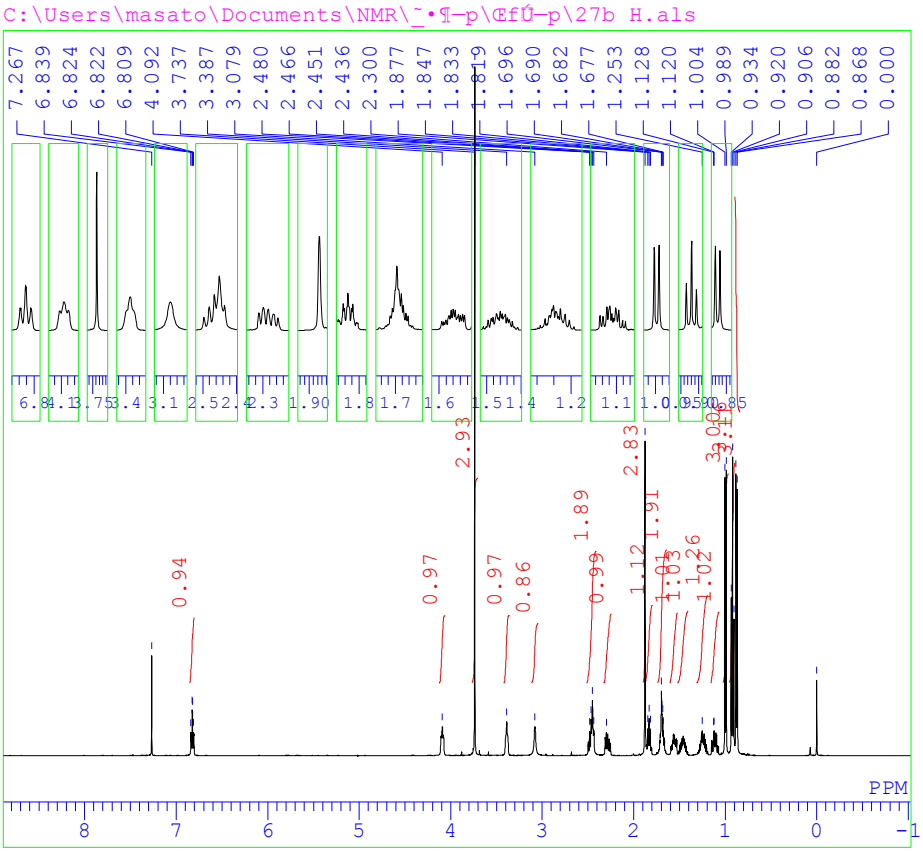
27a



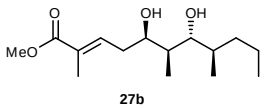
DFILE 27a C.als
COMNT single pulse dec
DATIM 2015-10-02 13:00
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 500
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56



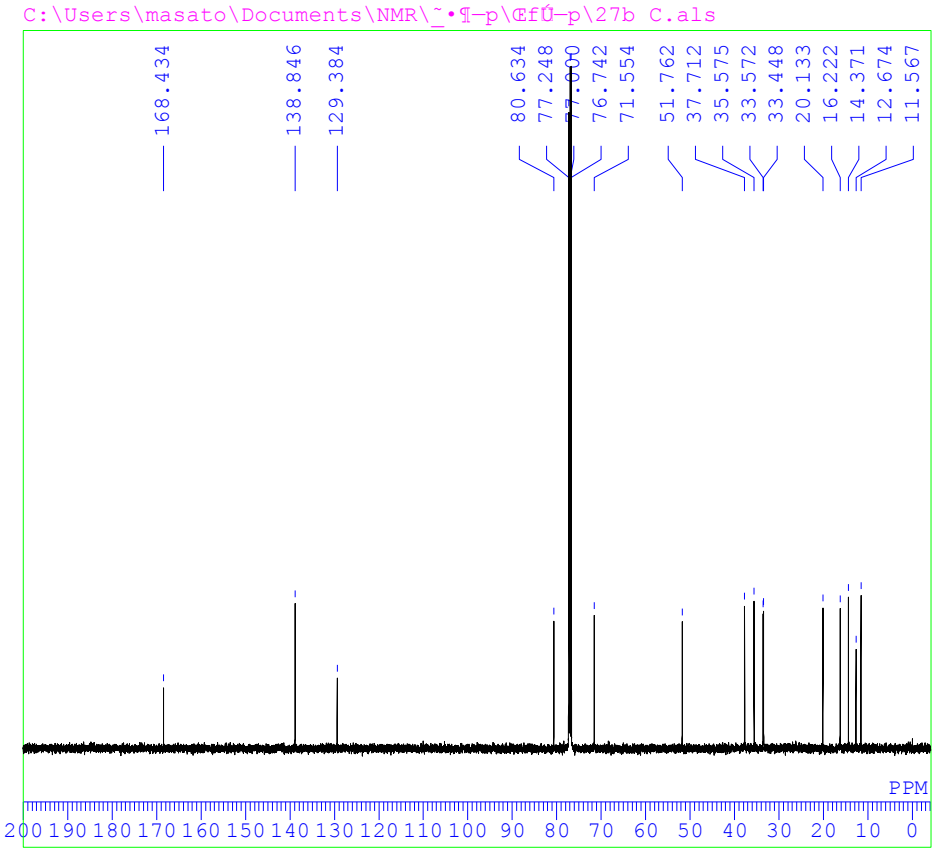
27a



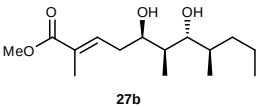

```
DFILE  27b H.als
COMNT  single_pulse
DATIM  2014-08-22 09:22
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          23.4 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            40
```



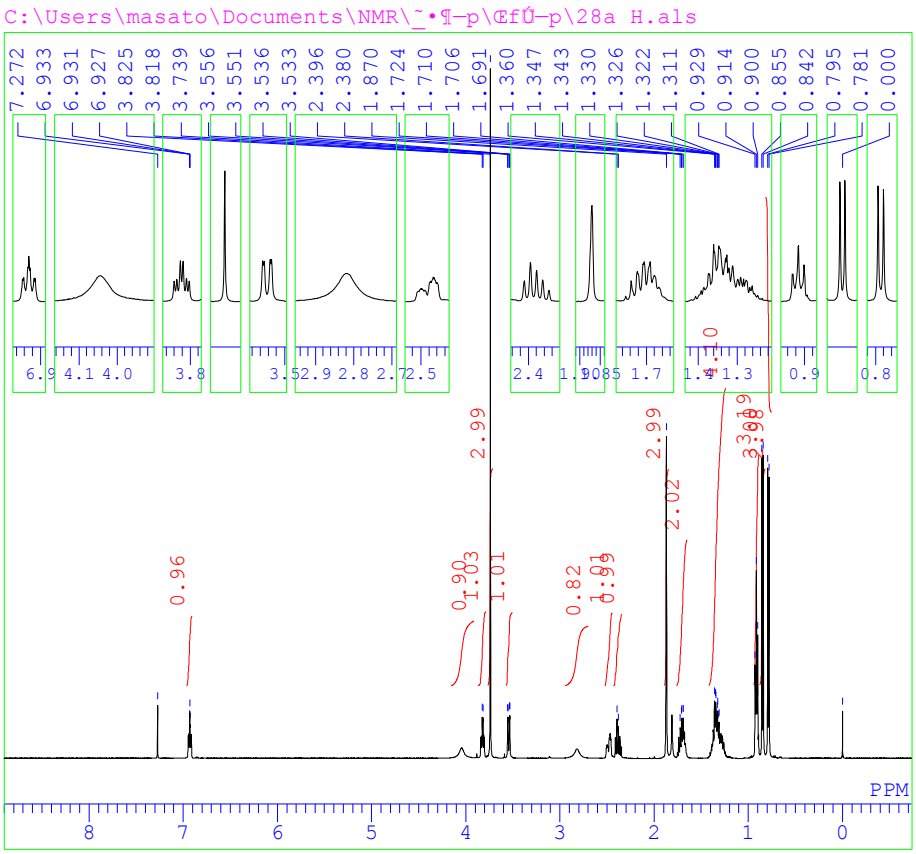
27b



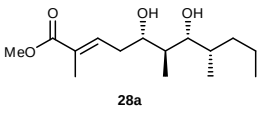

```
DFILE  27b C.als
COMNT  single pulse dec
DATIM  2014-08-22 13:34
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          24.3 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            54
```



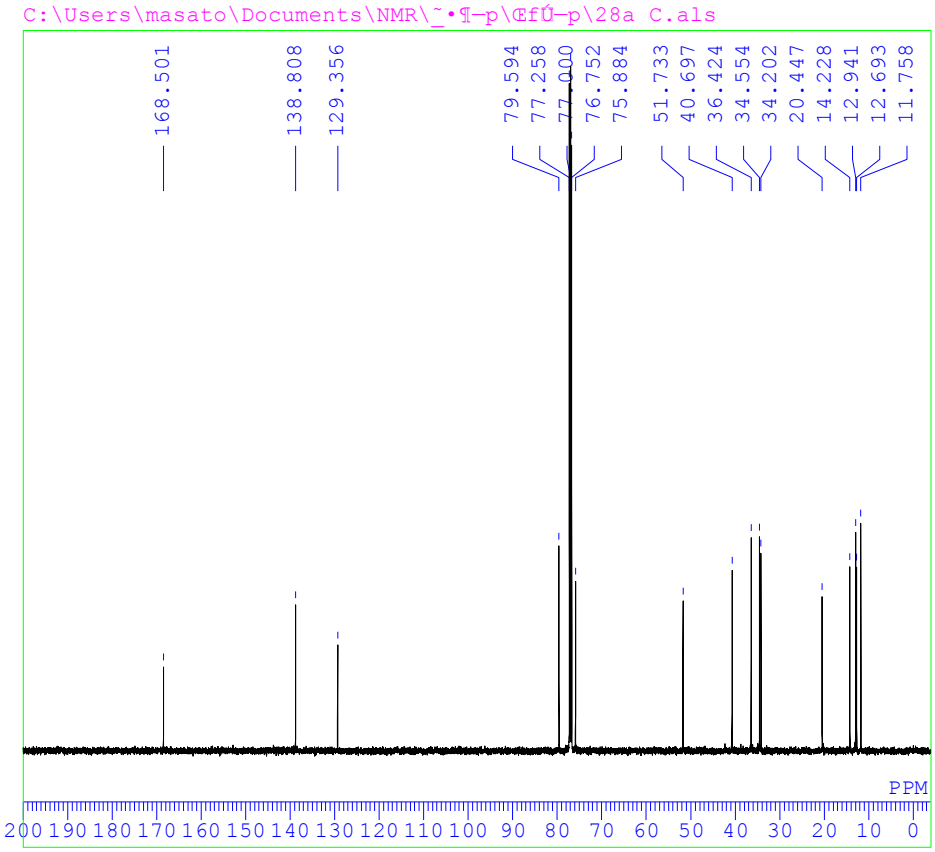
27b



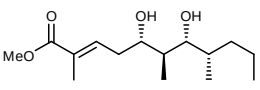

```
DFILE  28a H.als
COMNT  single_pulse
DATIM  2015-10-08 19:24
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.2 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            38
```



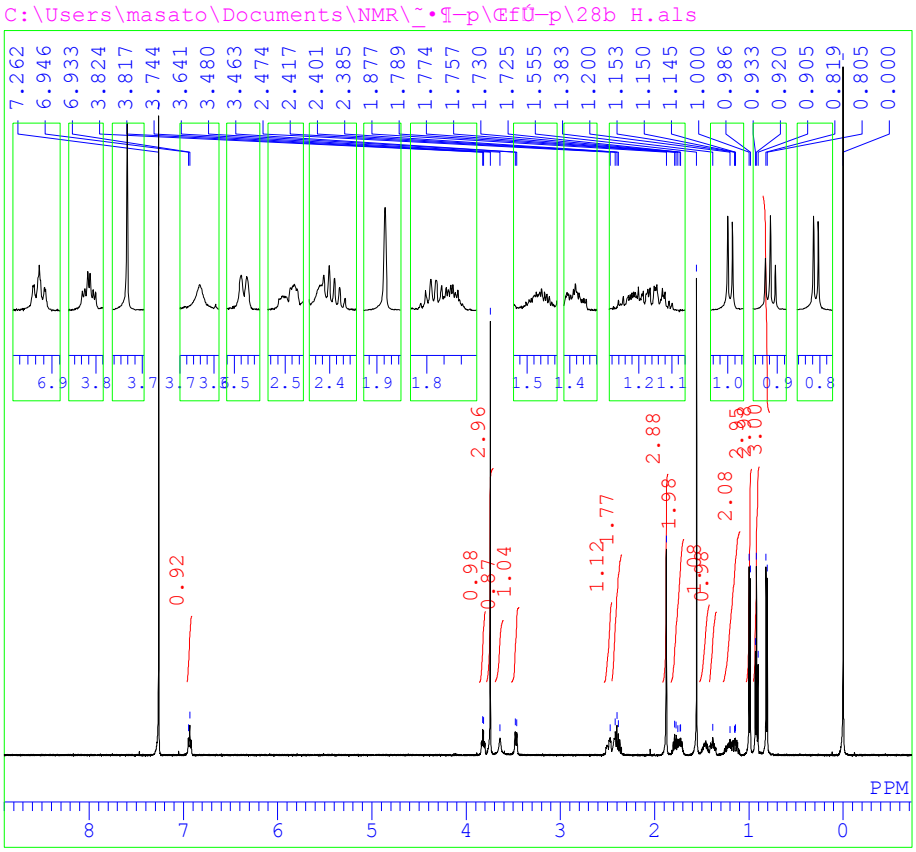
28a



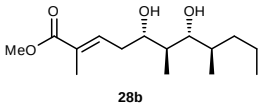

```
DFILE  28a C.als
COMNT  single pulse dec
DATIM  2015-10-08 20:12
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.2 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            54
```



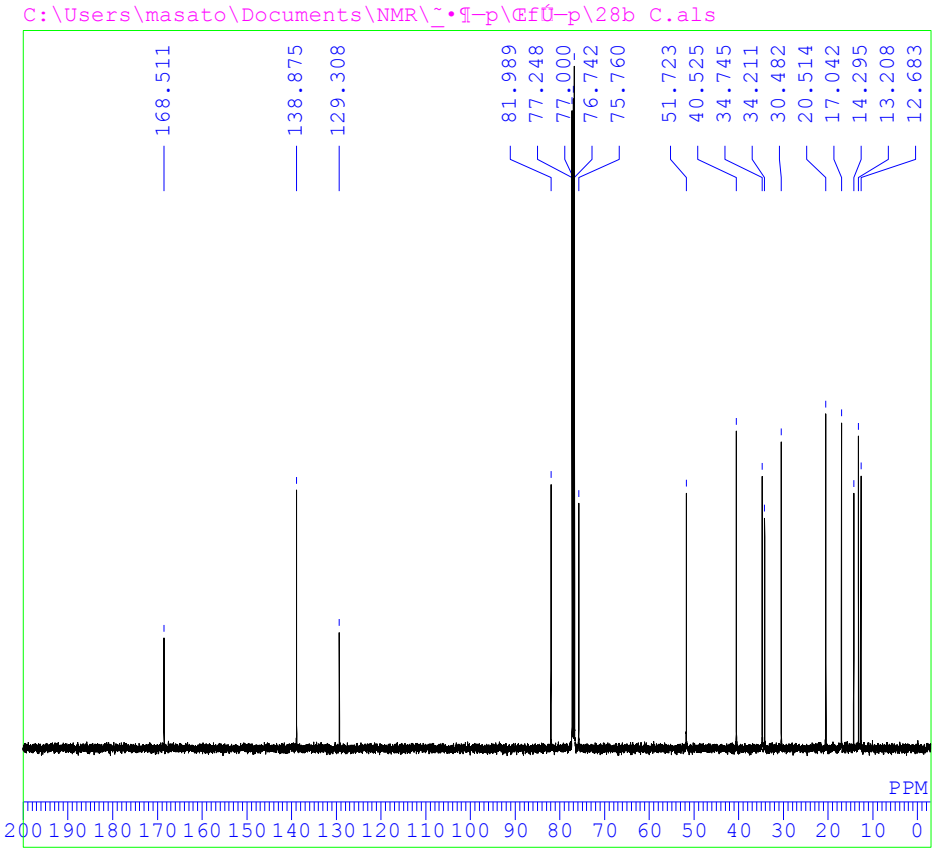
28a



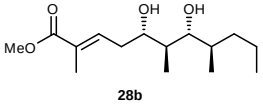
DFILE 28b H.als
COMNT single_pulse
DATIM 2015-10-15 08:57
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 54



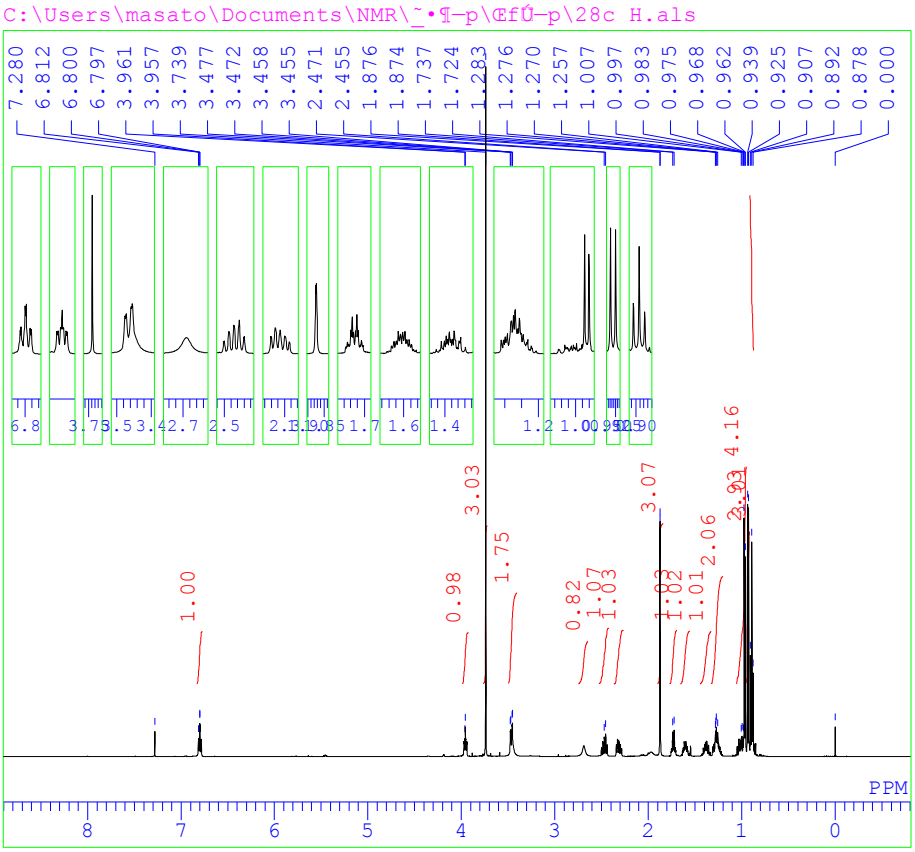
28b



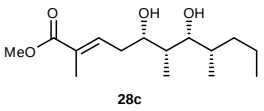
DFILE 28b C.als
COMNT single pulse dec
DATIM 2015-10-15 12:53
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 54



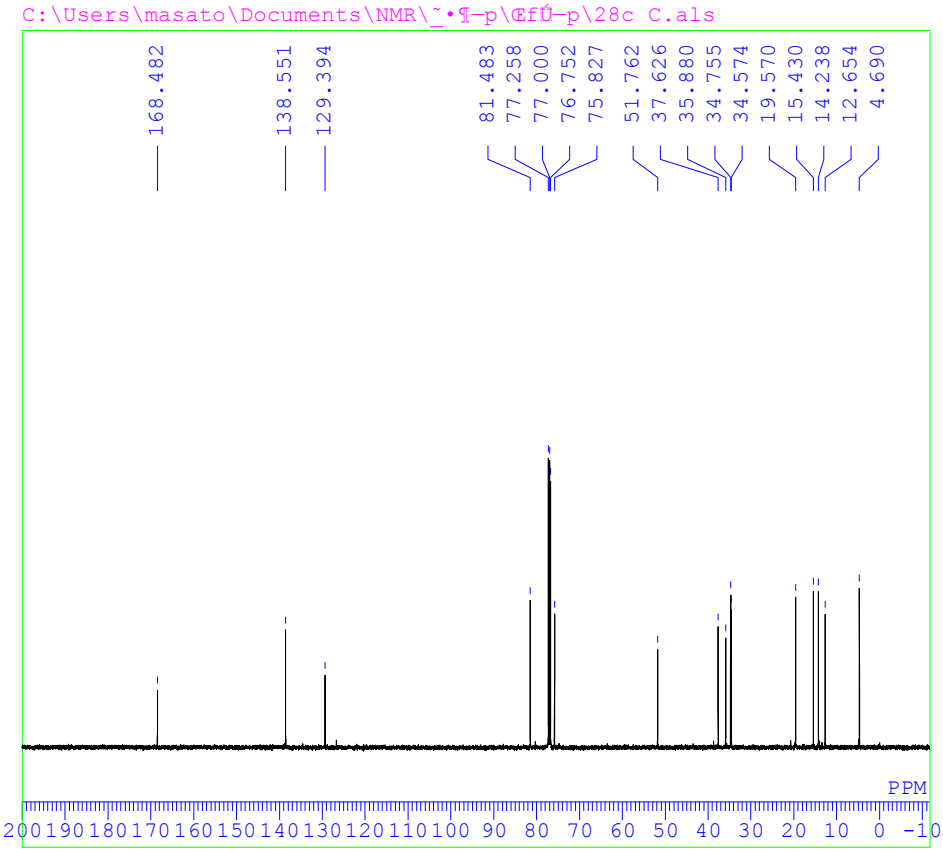
28b



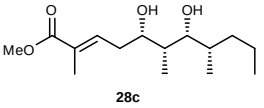
DFILE 28c H.als
COMNT single_pulse
DATIM 2015-10-02 15:05
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34



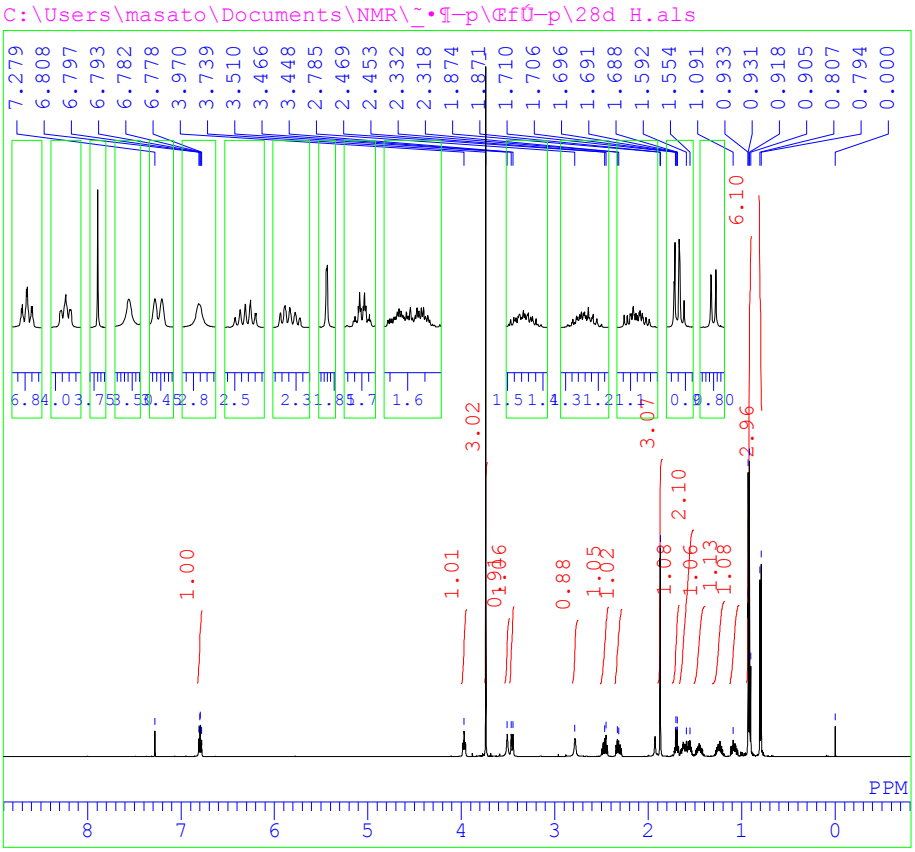
28c



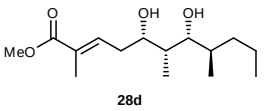
DFILE 28c C.als
COMNT single pulse dec
DATIM 2015-10-02 15:29
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 500
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



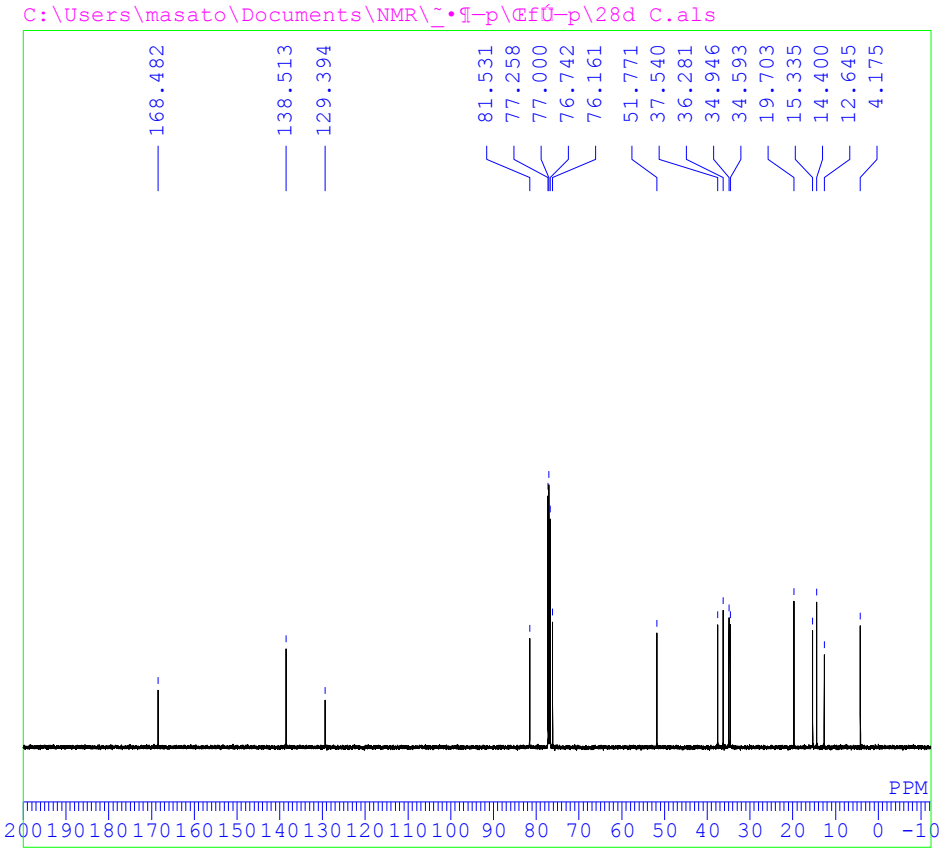
28c



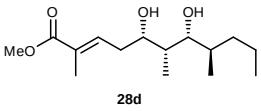

```
DFILE  28d H.als
COMNT  single_pulse
DATIM  2015-10-02 15:35
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP         21.8 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            34
```



28d

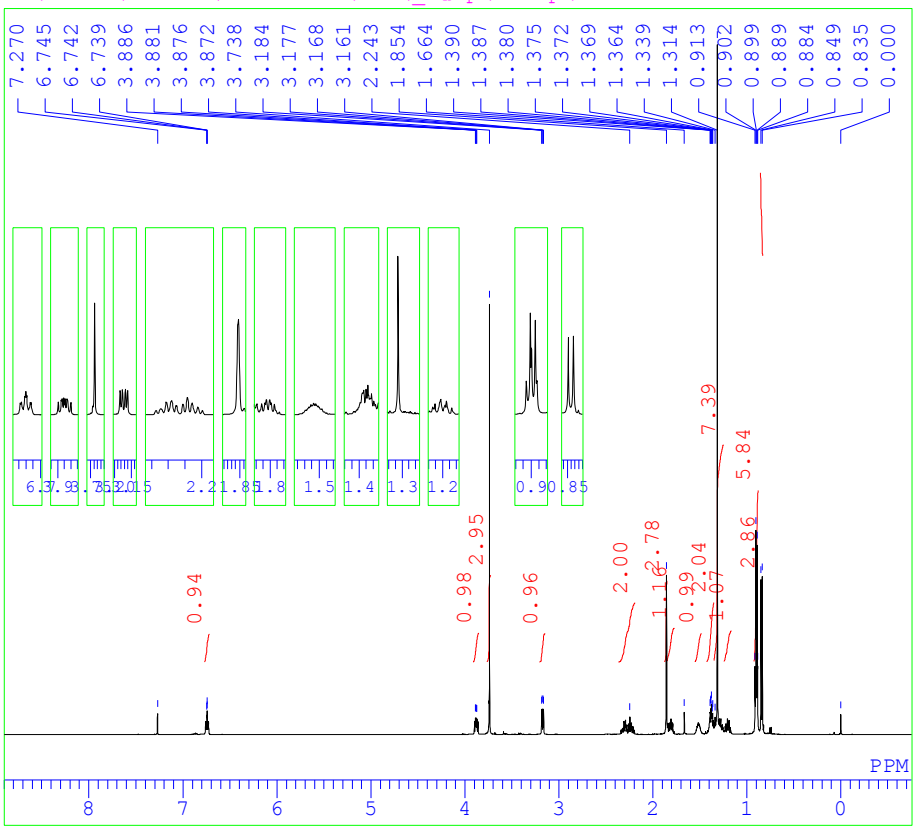



```
DFILE  28d C.als
COMNT  single pulse dec
DATIM  2015-10-02 15:59
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS           500
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP         22.0 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            60
```

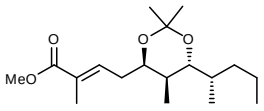


28d

C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\29a H.als

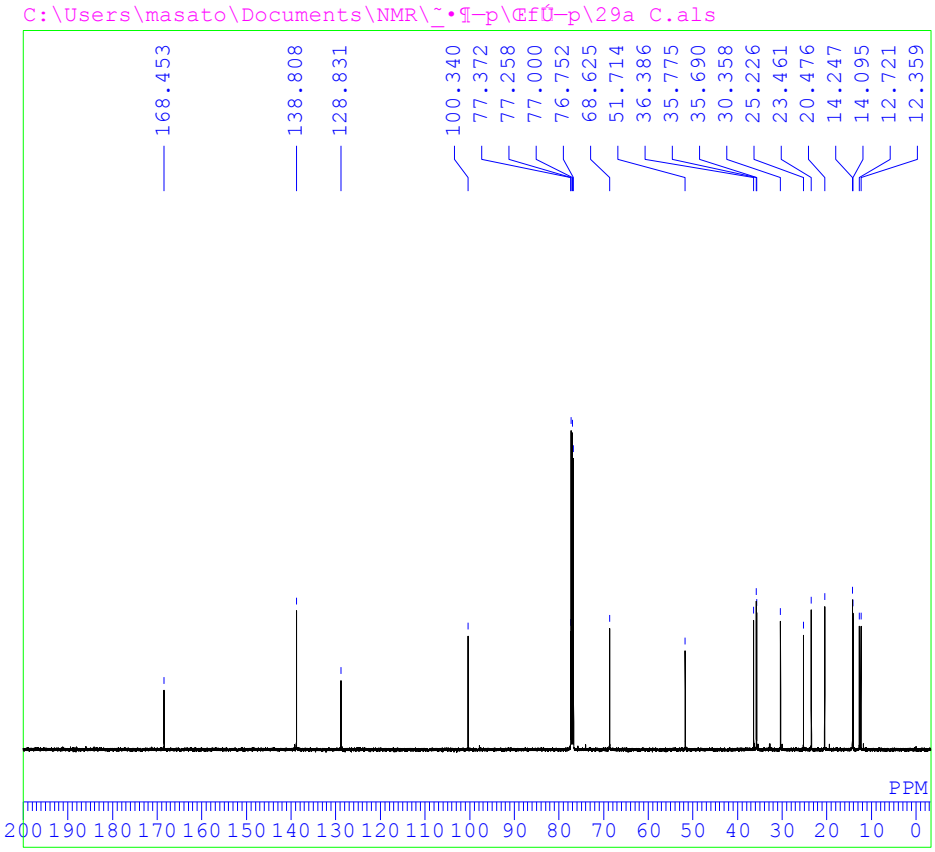


DFILE 29a H.als
COMNT single_pulse
DATIM 2015-10-17 13:56
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34

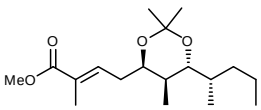


29a

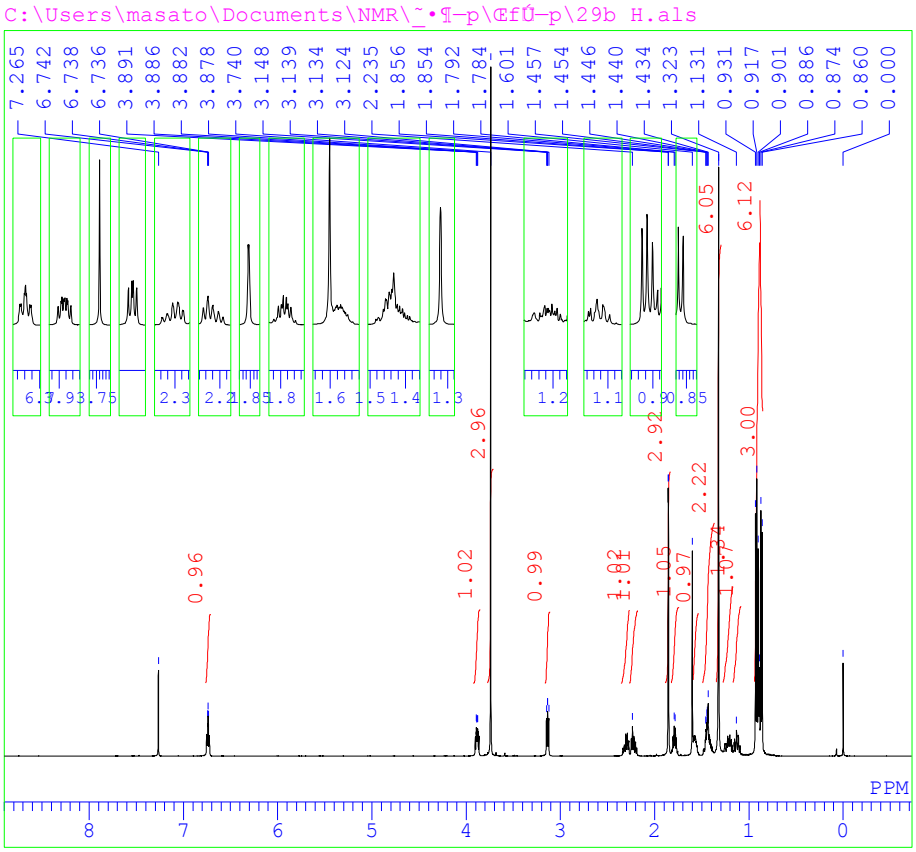
C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\29a C.als



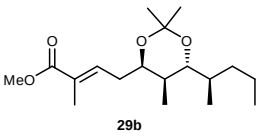
DFILE 29a C.als
COMNT single pulse dec
DATIM 2015-10-17 13:55
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 54

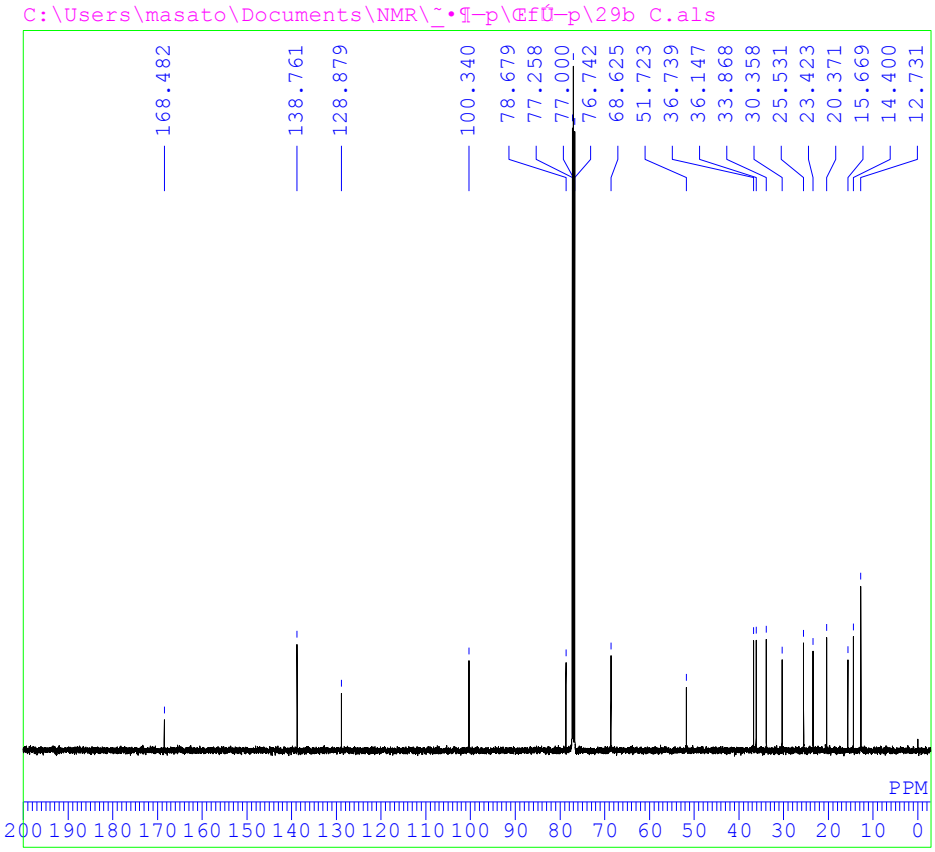


29a

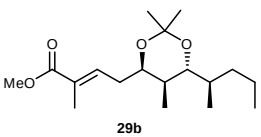


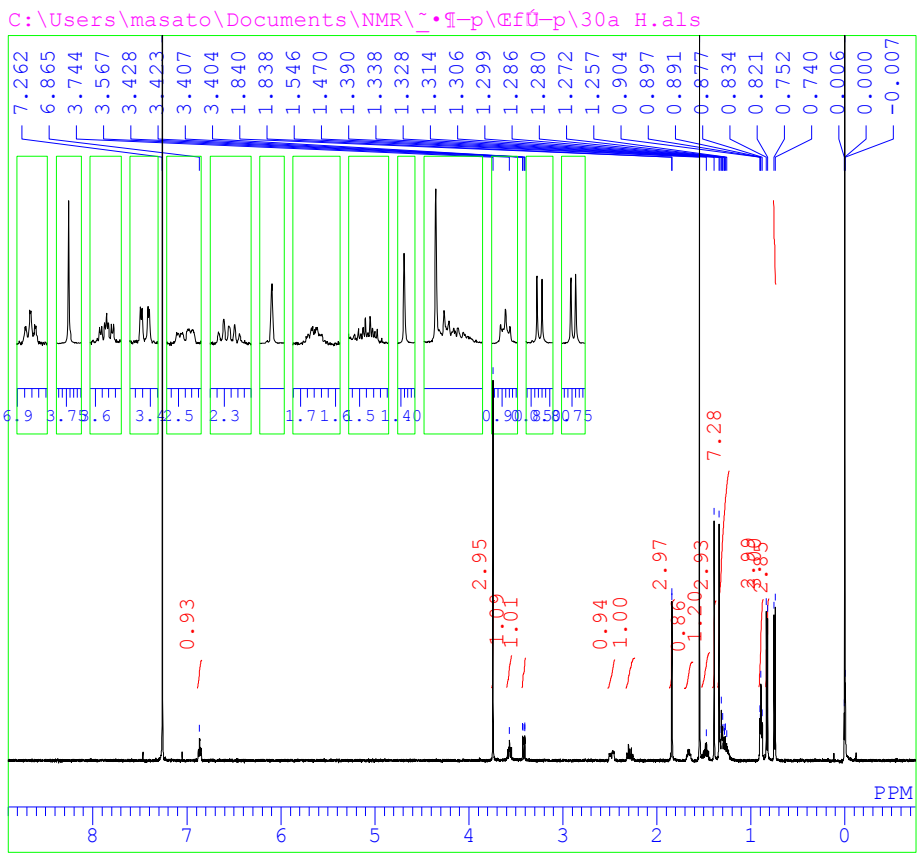

```
DFILE  29b H.als
COMNT  single_pulse
DATIM  2015-10-03 09:14
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.8 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            42
```



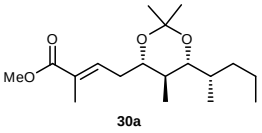



```
DFILE  29b C.als
COMNT  single pulse dec
DATIM  2014-08-23 13:09
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          34.5 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            56
```

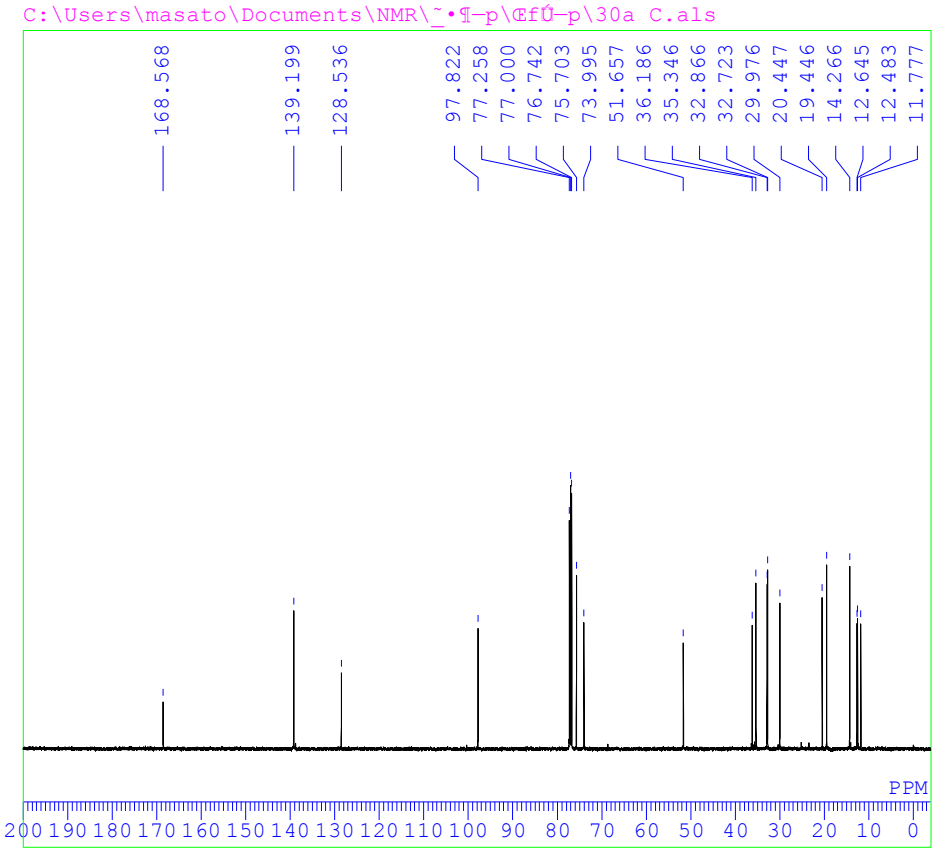




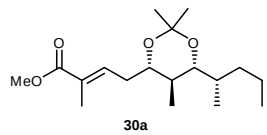
DFILE 30a H.als
COMNT single_pulse
DATIM 2015-10-21 09:41
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 56



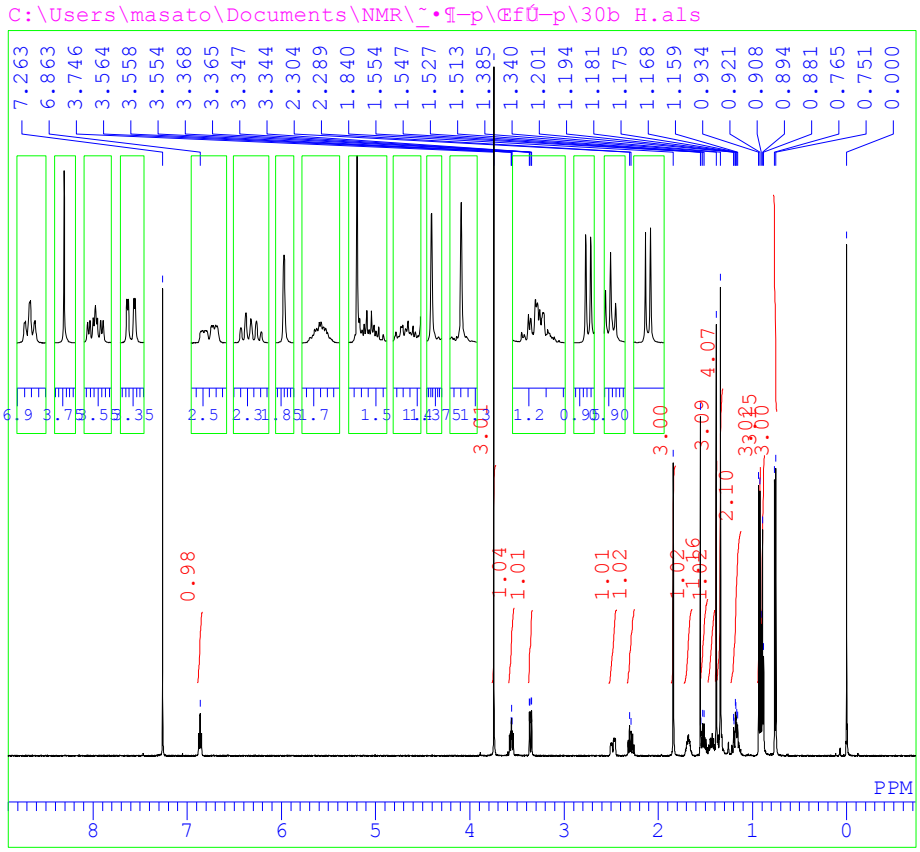
30a



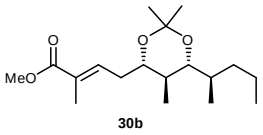
DFILE 30a C.als
COMNT single pulse dec
DATIM 2015-10-21 15:46
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58



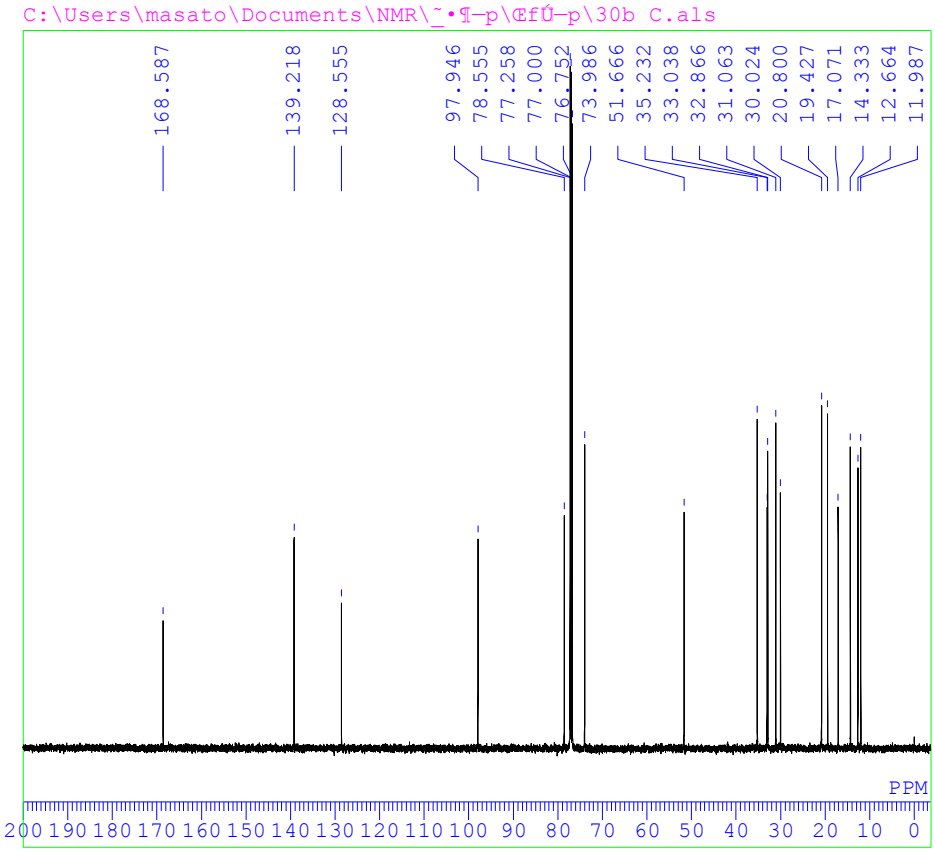
30a



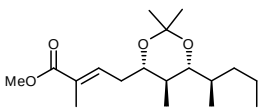

```
DFILE  30b H.als
COMNT  single_pulse
DATIM  2015-11-04 09:35
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.8 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            50
```



30b




```
DFILE  30b C.als
COMNT  single pulse dec
DATIM  2015-10-17 10:50
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.3 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            54
```



30b

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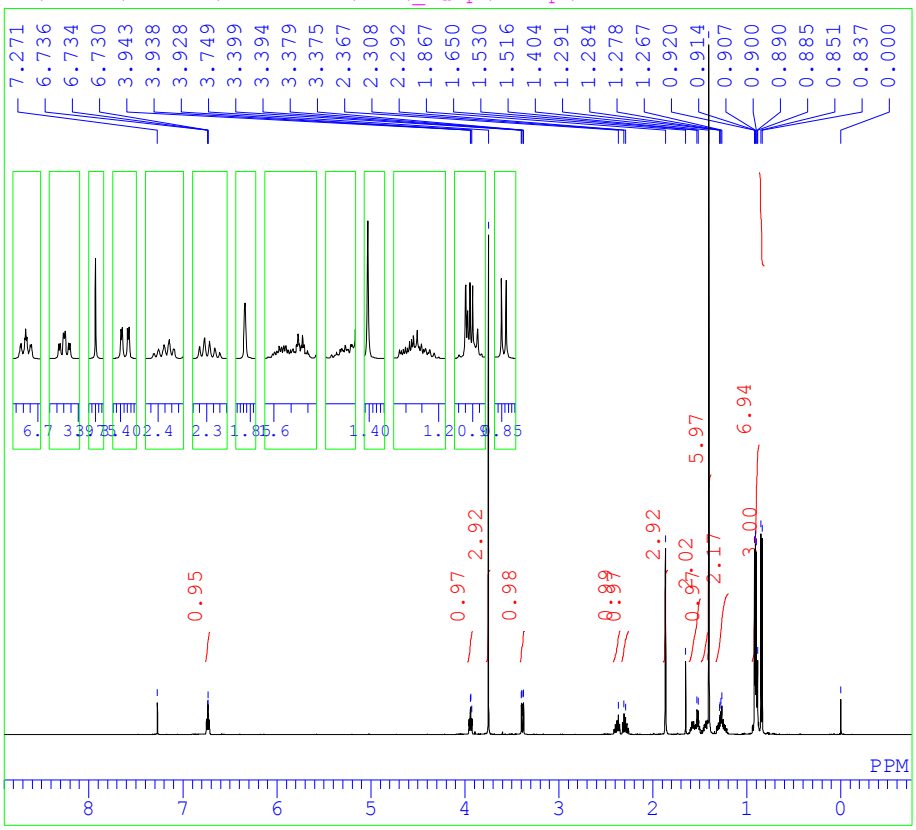
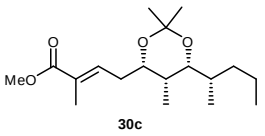


DFILE 30c H.als
COMNT single_pulse
DATIM 2015-09-10 13:25
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38



30c

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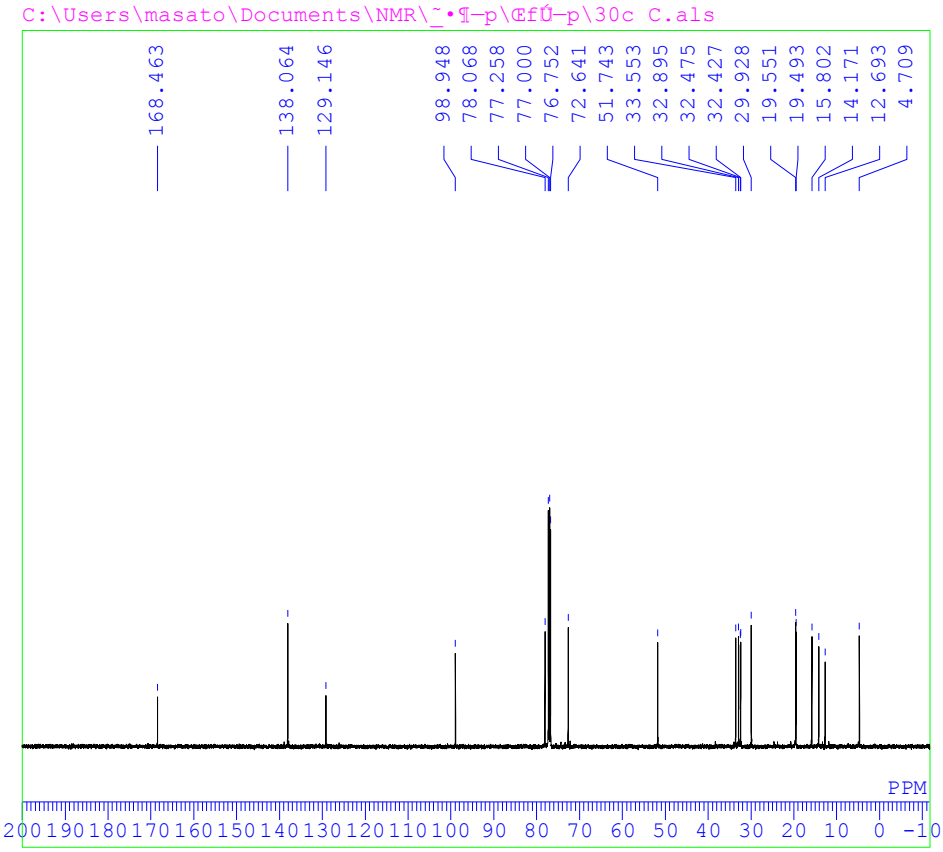
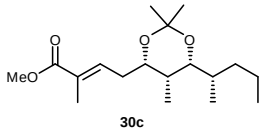
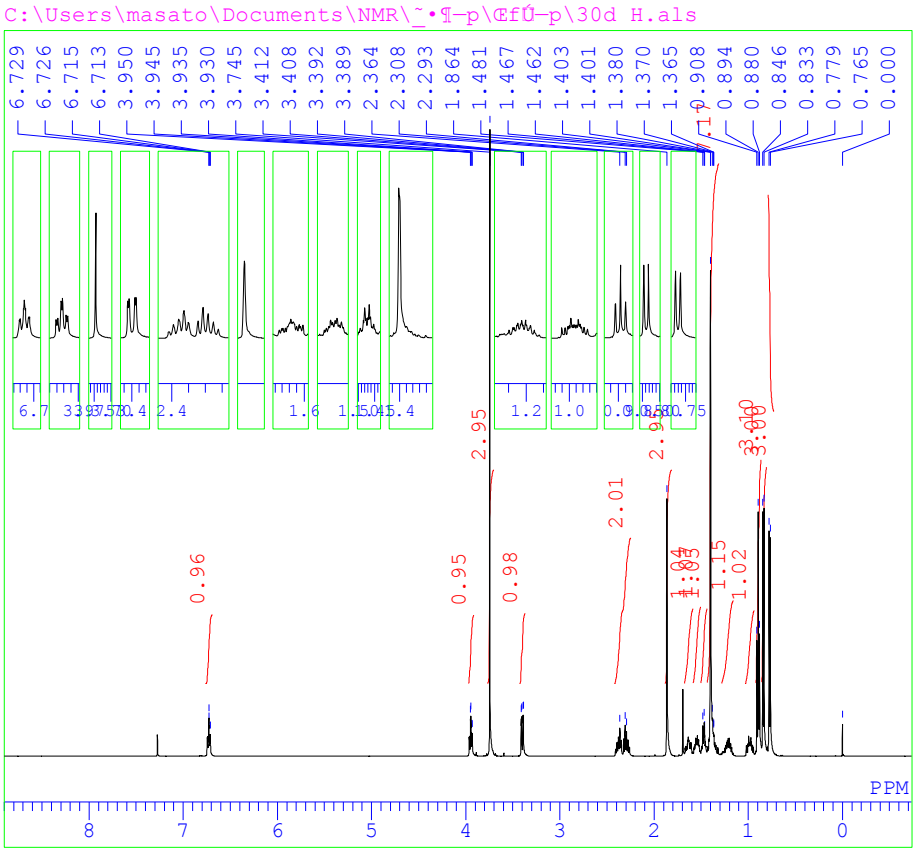


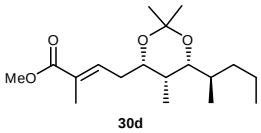
DFILE 30c C.als
COMNT single pulse dec
DATIM 2015-02-28 11:15
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 54



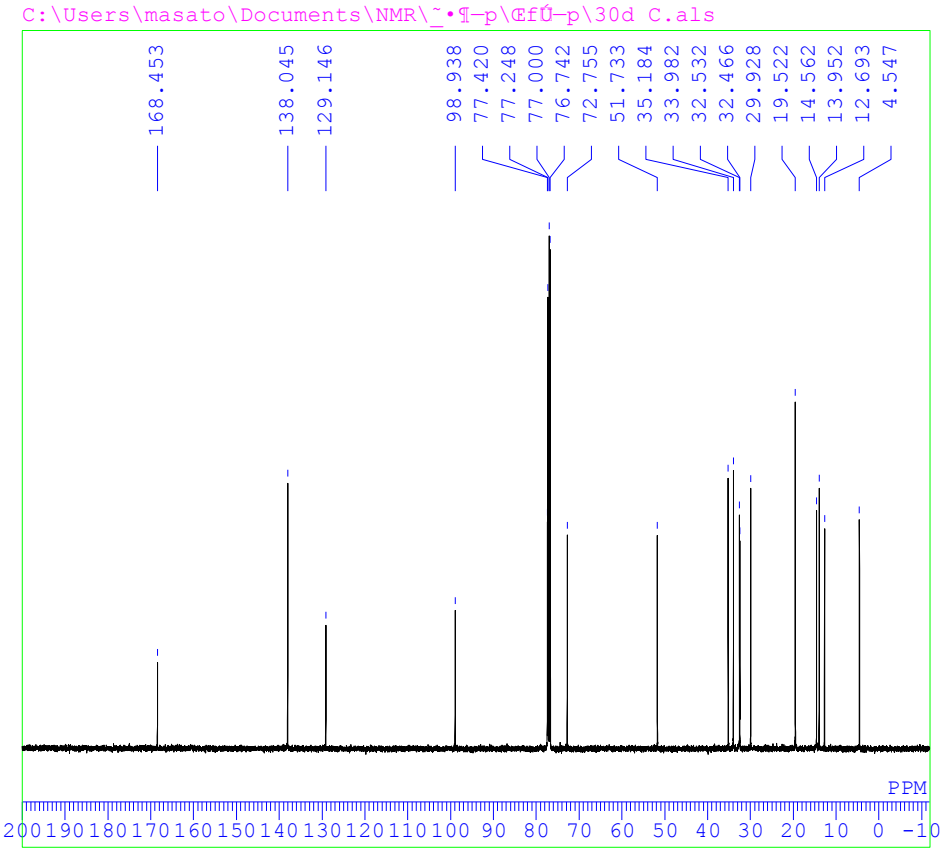
30c



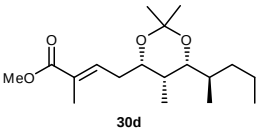
DFILE 30d H.als
COMNT single_pulse
DATIM 2015-09-09 15:08
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34



30d



DFILE 30d C.als
COMNT single pulse dec
DATIM 2015-09-09 15:56
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56



30d

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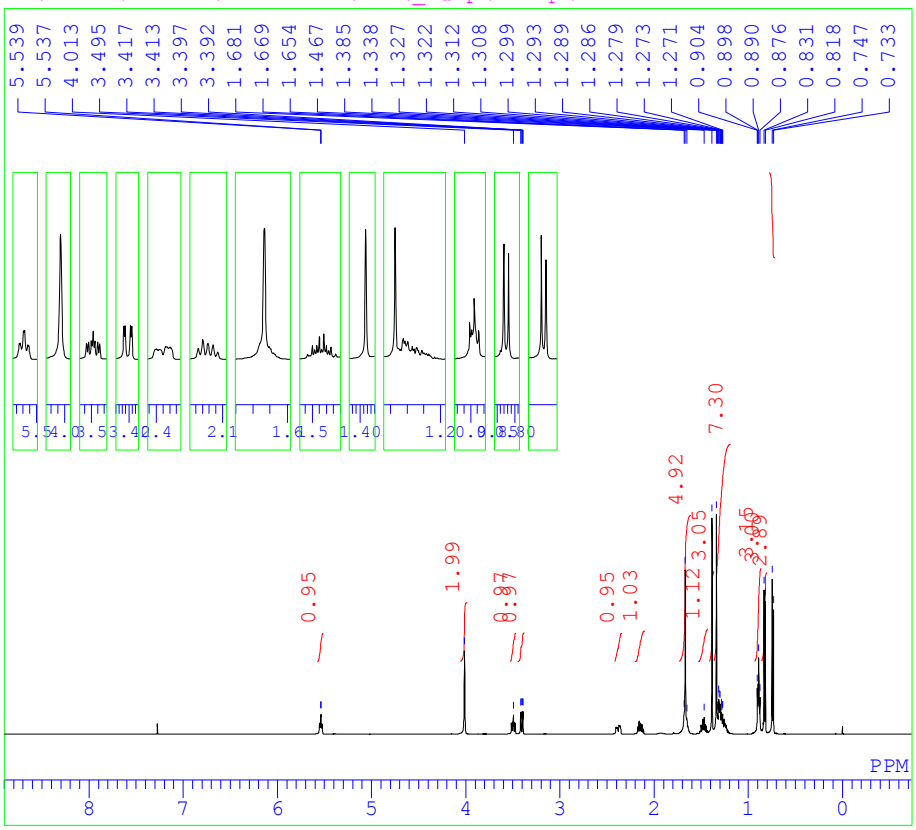
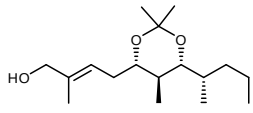
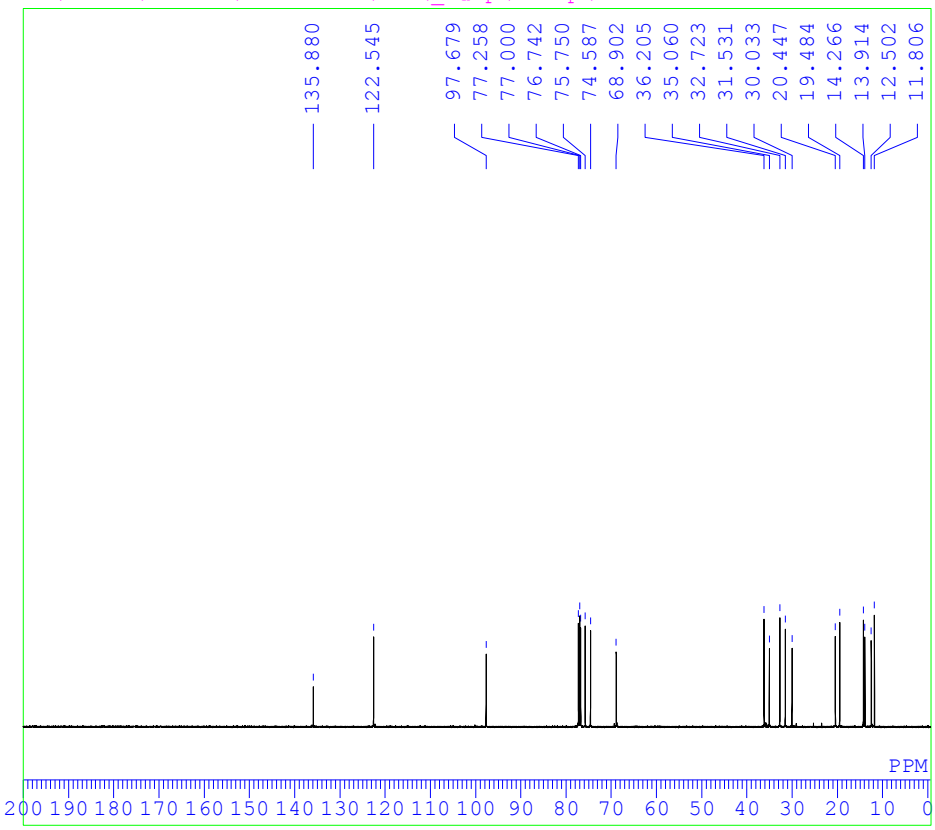


DFILE S1a H.als
COMNT single_pulse
DATIM 2015-10-30 10:00
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 30

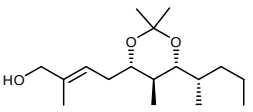


S1a

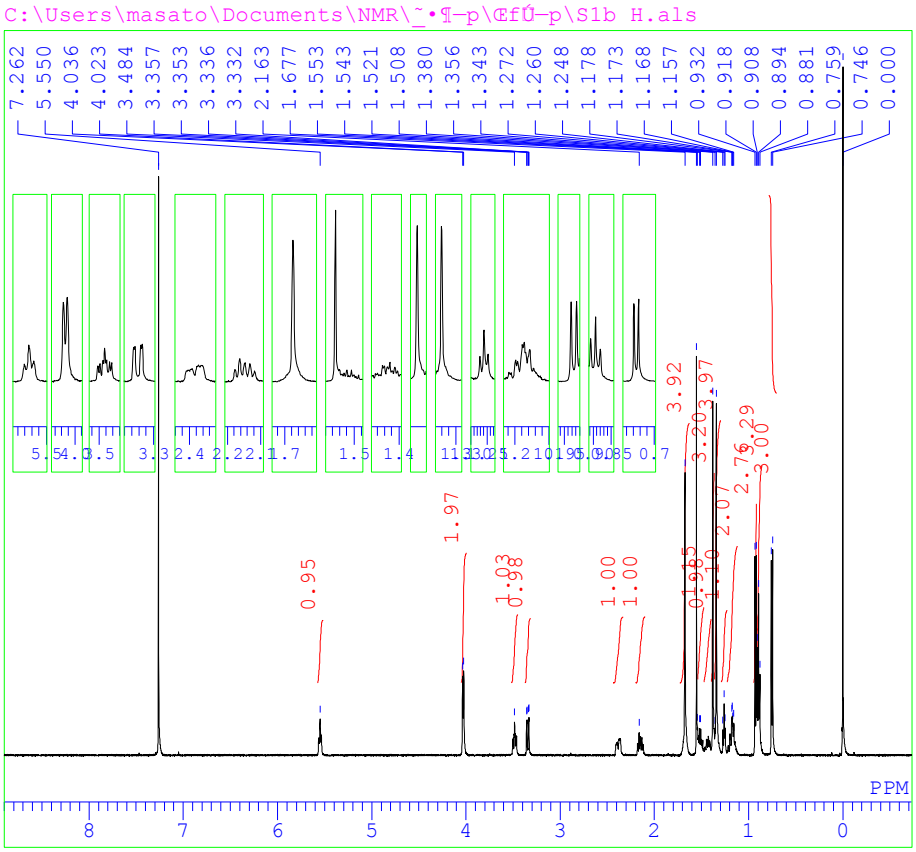
C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\S1a C.als



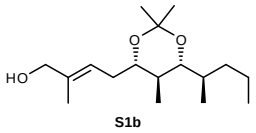
DFILE S1a C.als
COMNT single pulse dec
DATIM 2015-10-30 10:48
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58



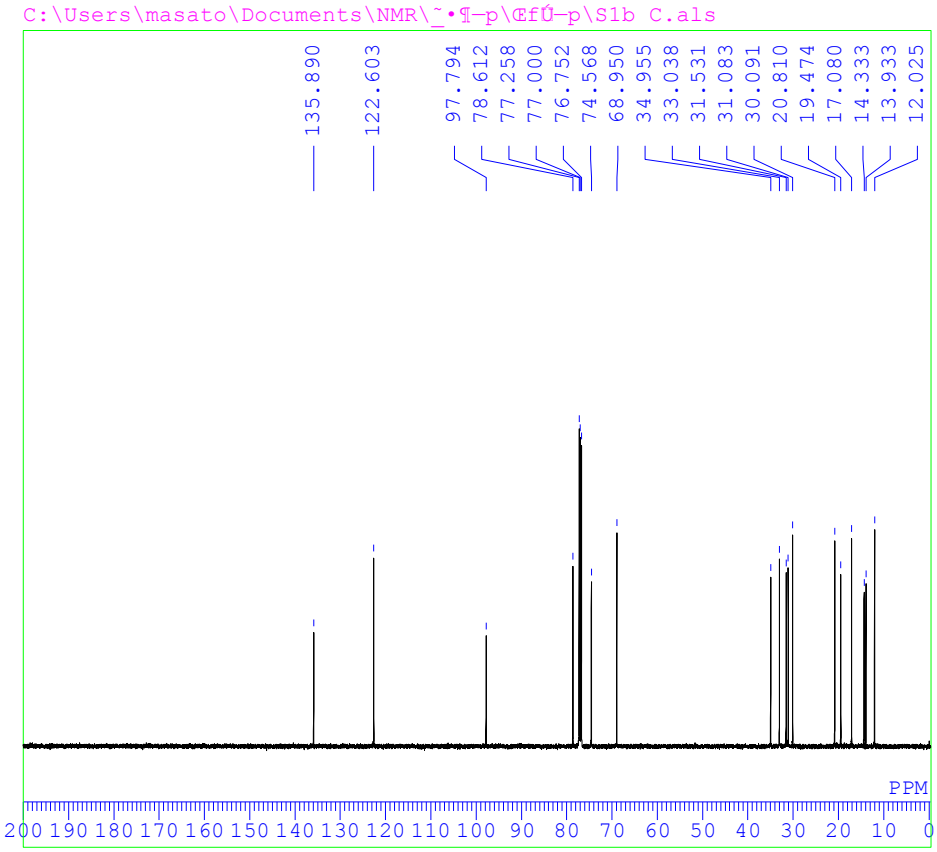
S1a



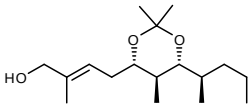
DFILE S1b H.als
COMNT single_pulse
DATIM 2015-10-19 08:50
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 52



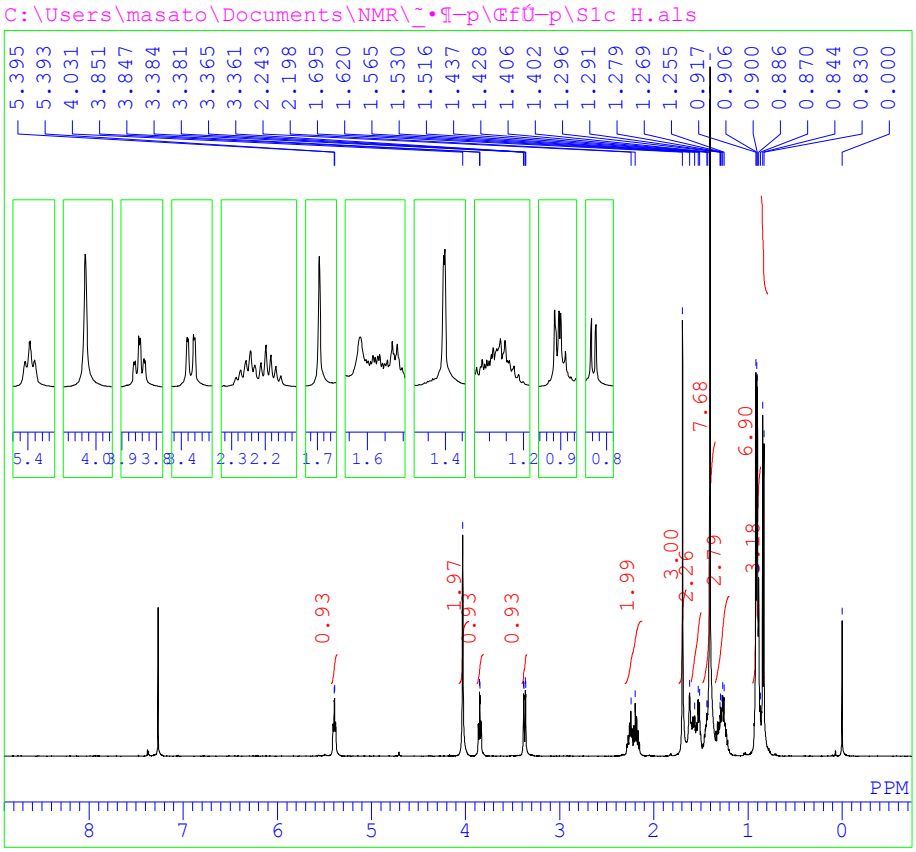
S1b



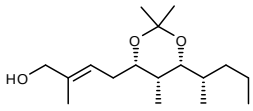
DFILE S1b C.als
COMNT single pulse dec
DATIM 2015-10-19 13:44
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 56



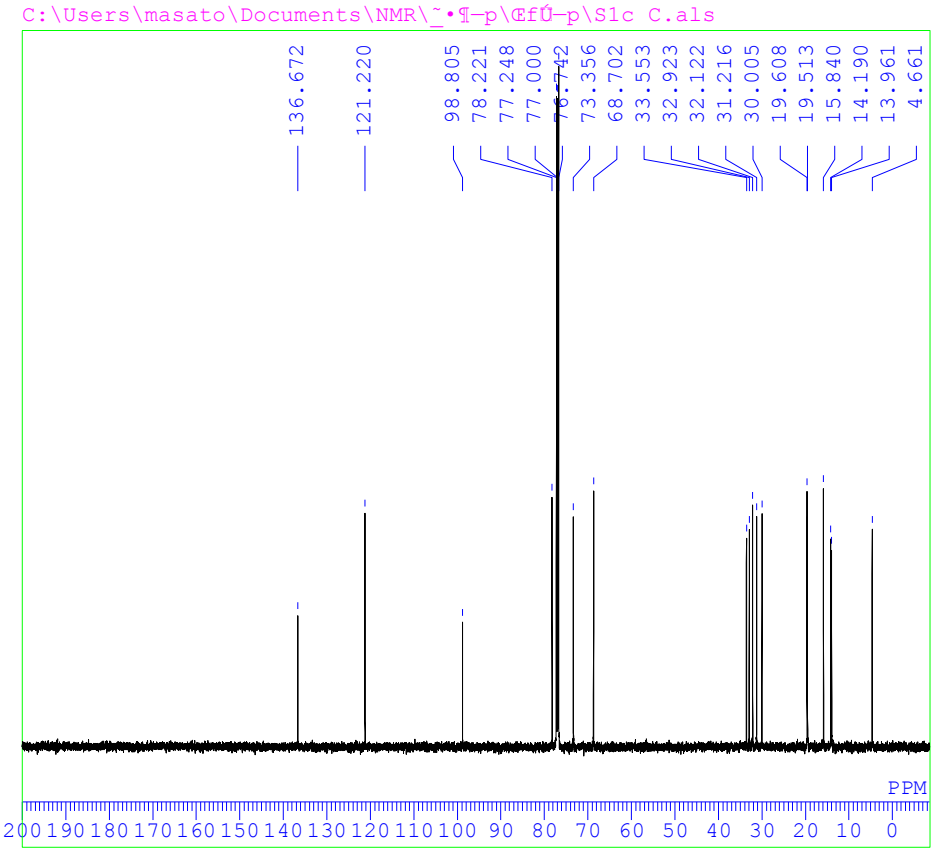
S1b



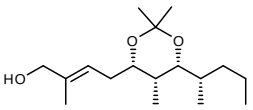
DFILE S1c H.als
COMNT single_pulse
DATIM 2015-09-12 09:14
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 44



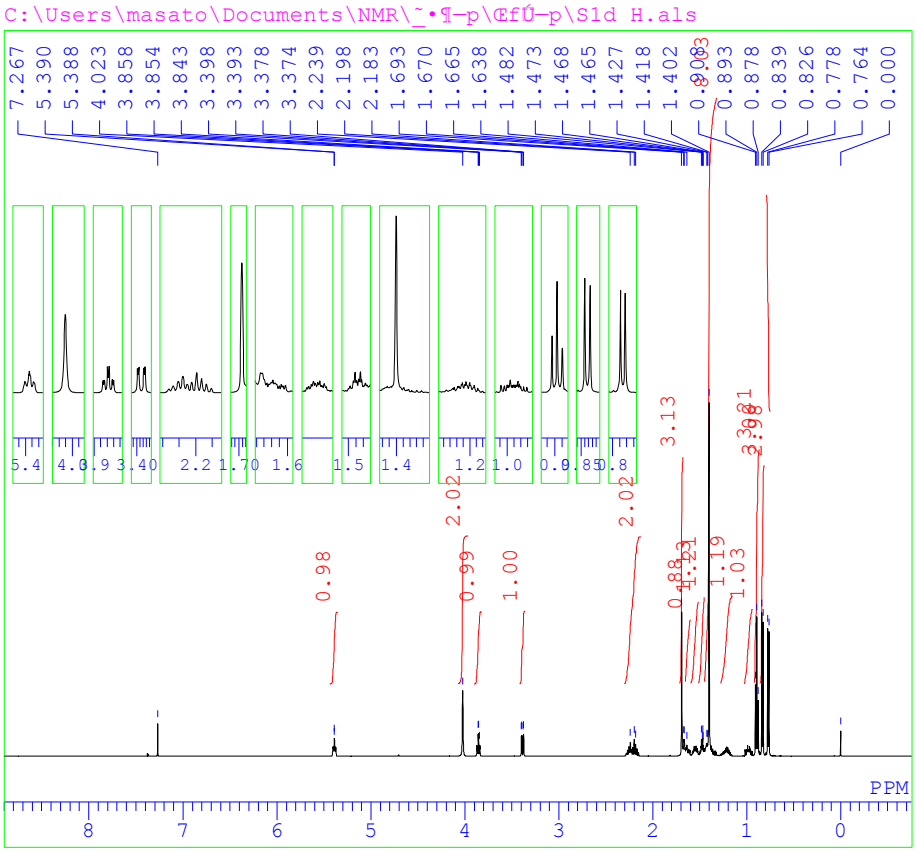
S1c



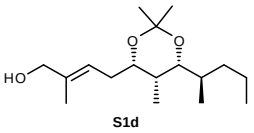
DFILE S1c C.als
COMNT single pulse dec
DATIM 2015-09-12 11:22
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 58



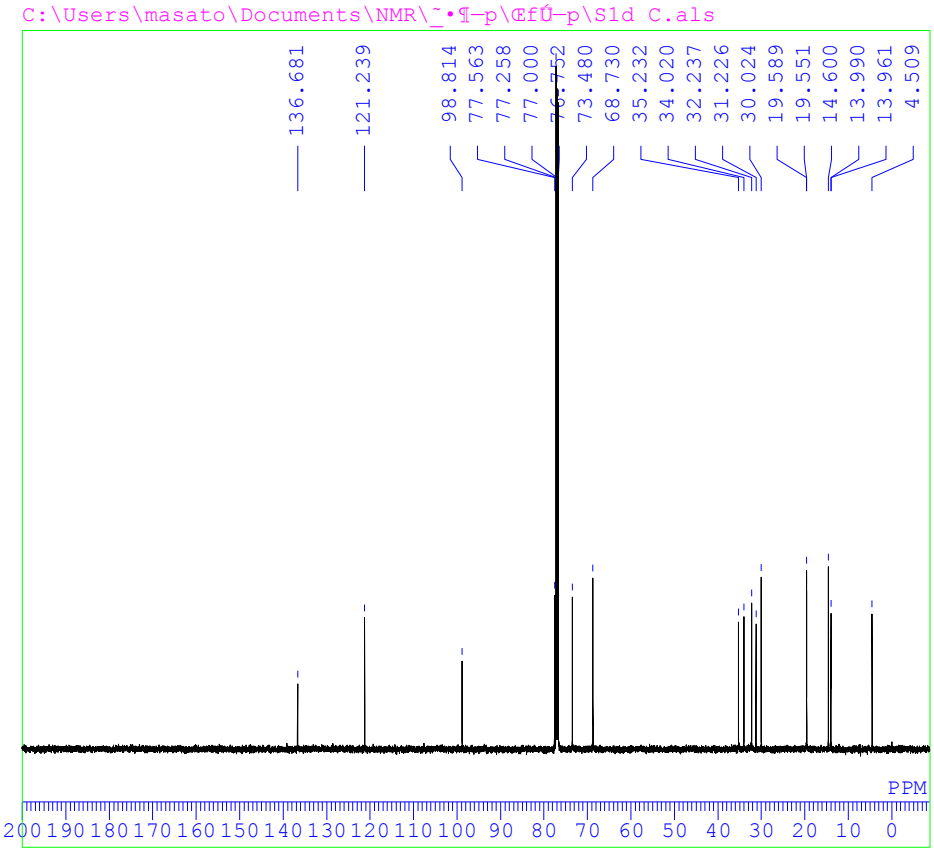
S1c



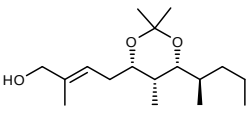

```
DFILE  S1d H.als
COMNT  single_pulse
DATIM  2015-10-08 15:37
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.3 c
SLVNT  CDCL3
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            38
```



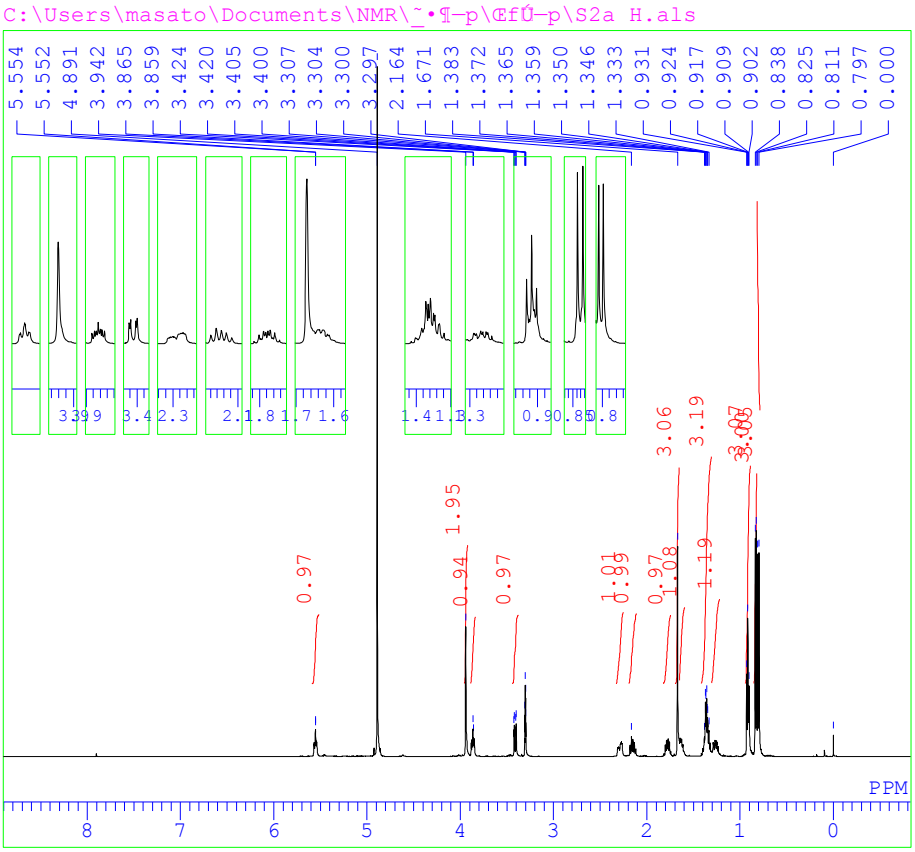
S1d



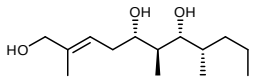

```
DFILE  S1d C.als
COMNT  single pulse dec
DATIM  2015-10-08 16:25
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.2 c
SLVNT  CDCL3
EXREF         77.00 ppm
BF             1.20 Hz
RGAIN            54
```



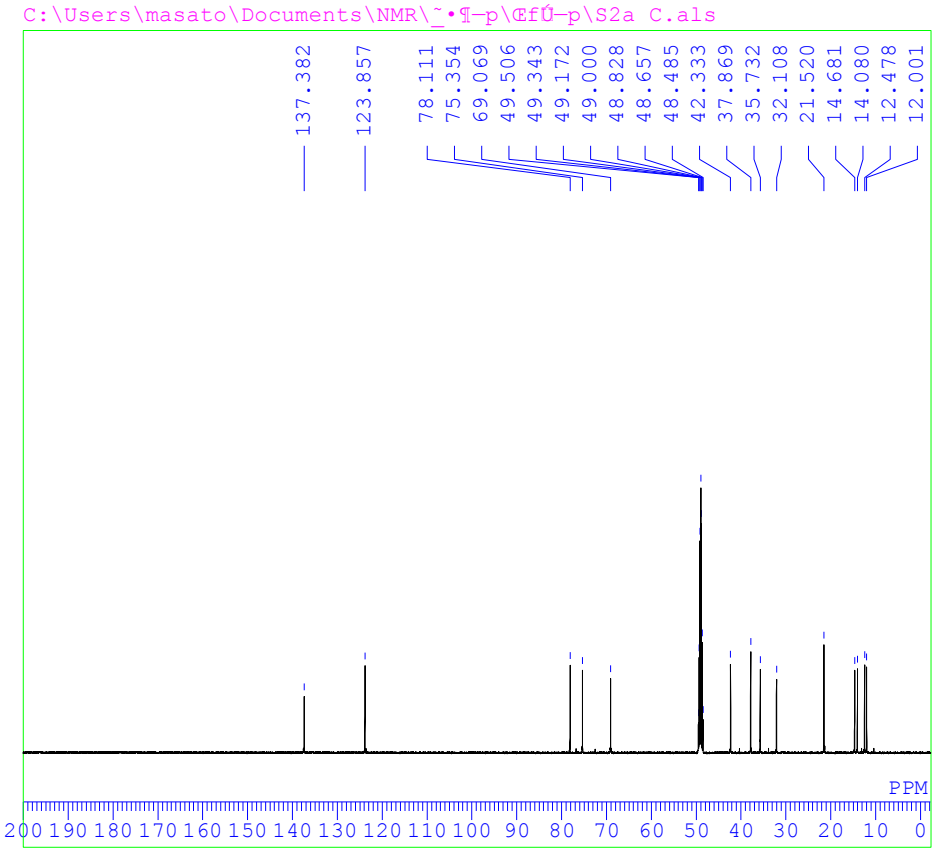
S1d



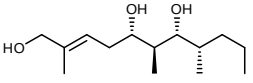
DFILE S2a H.als
COMNT single_pulse
DATIM 2015-12-09 10:57
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 20.4 c
SLVNT CD3OD
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36



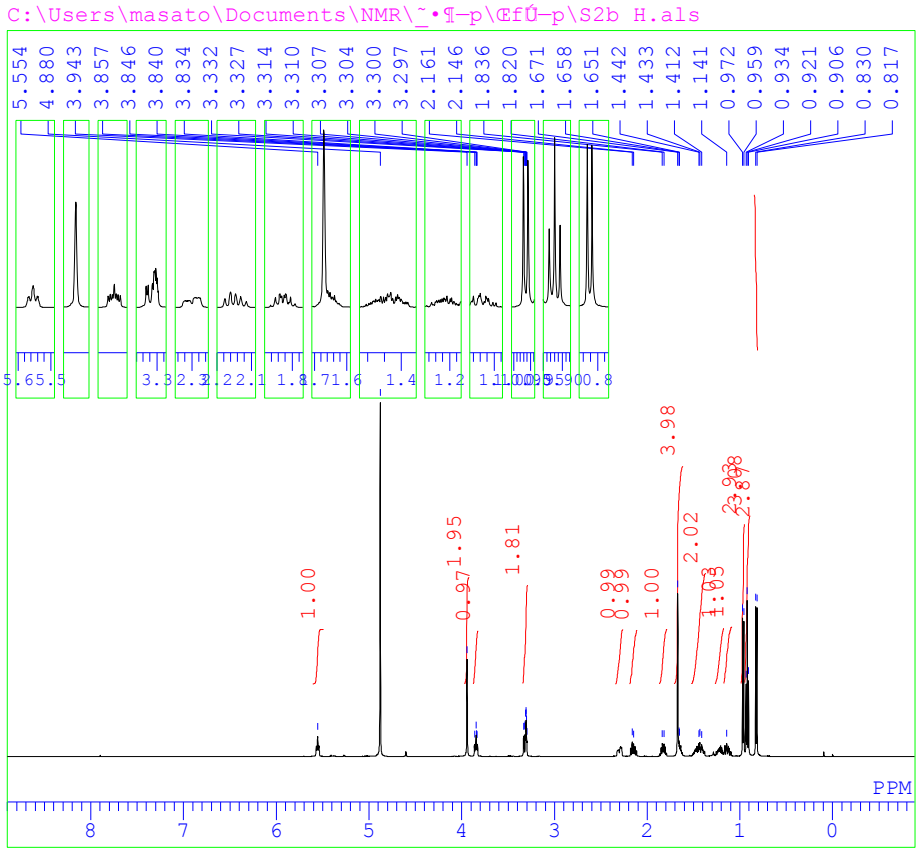
S2a



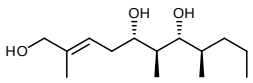
DFILE S2a C.als
COMNT single pulse dec
DATIM 2015-11-02 11:53
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.1 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 1.20 Hz
RGAIN 54



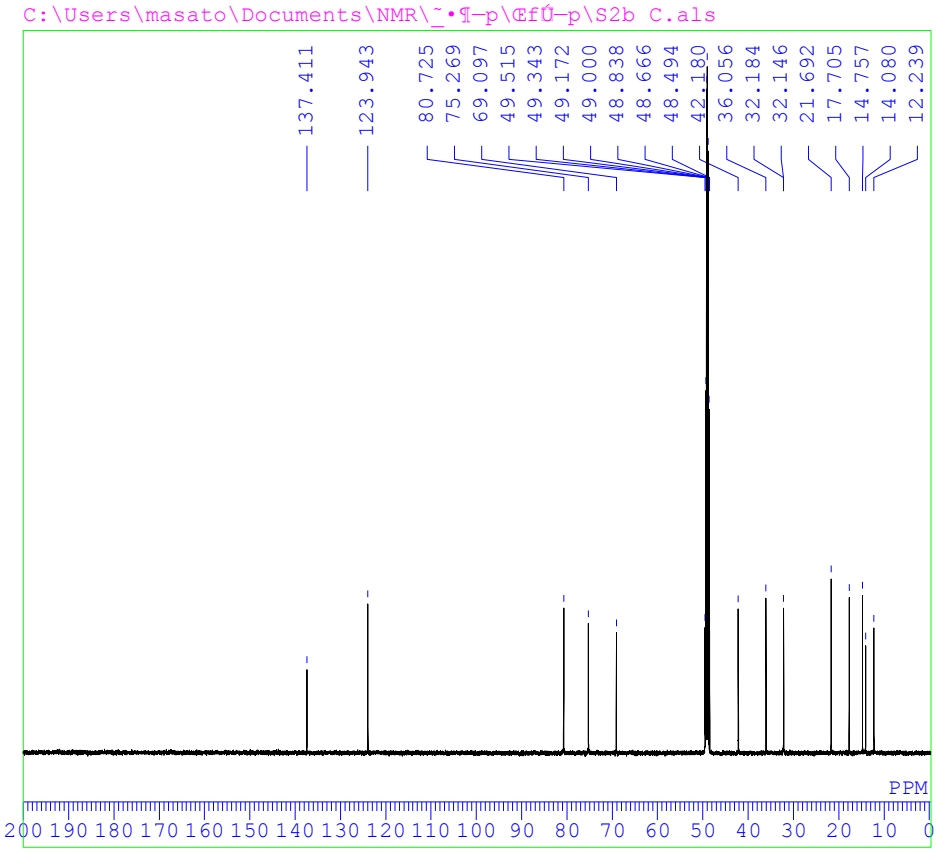
S2a



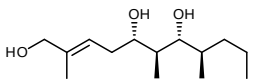
DFILE S2b H.als
COMNT single_pulse
DATIM 2015-10-22 12:40
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.8 c
SLVNT CD3OD
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 36



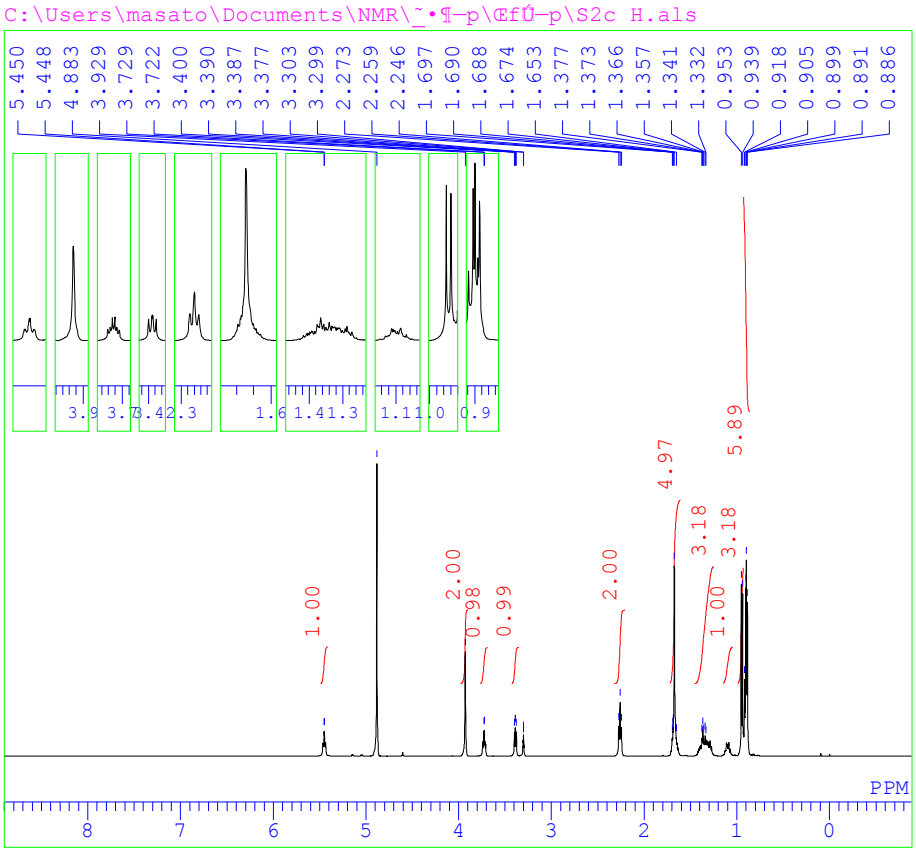
S2b



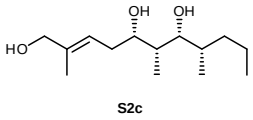
DFILE S2b C.als
COMNT single pulse dec
DATIM 2015-10-22 16:50
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.3 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 1.20 Hz
RGAIN 58



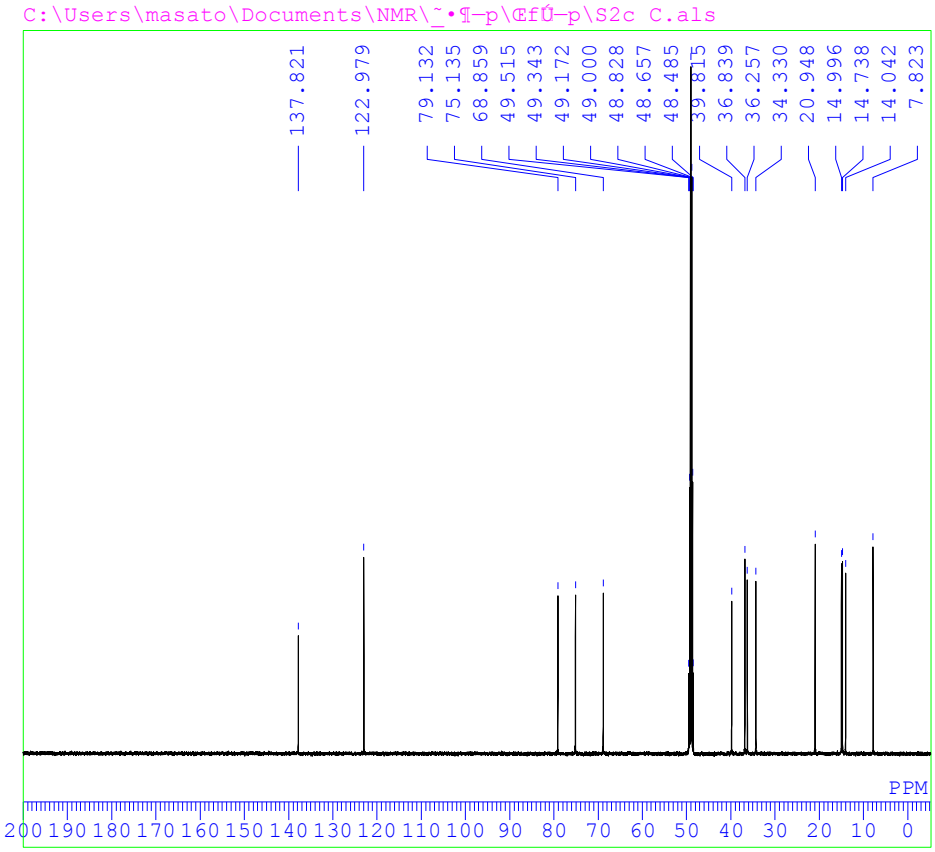
S2b



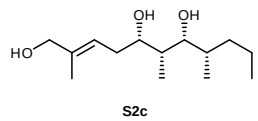
DFILE S2c H.als
COMNT single_pulse
DATIM 2015-09-18 12:40
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.7 c
SLVNT CD3OD
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 32



S2c



DFILE S2c C.als
COMNT single pulse dec
DATIM 2015-09-18 11:19
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.0 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 1.20 Hz
RGAIN 54



S2c

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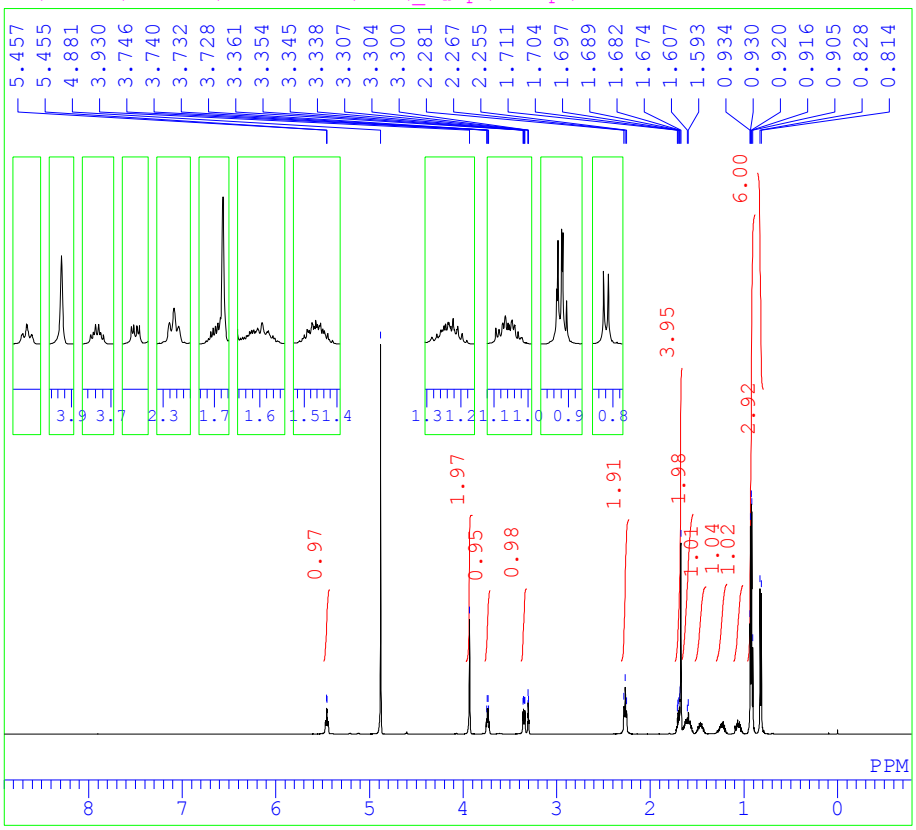
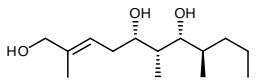


DFILE S2d H.als
COMNT single_pulse
DATIM 2015-09-17 09:27
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 21.8 c
SLVNT CD3OD
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 34



S2d

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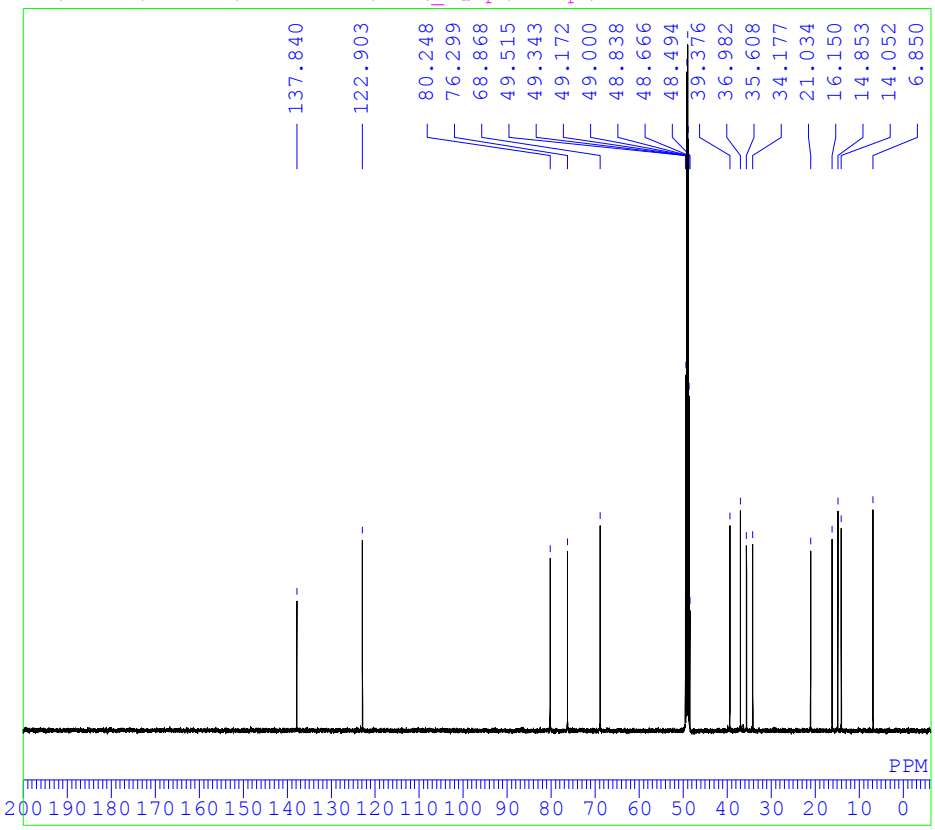
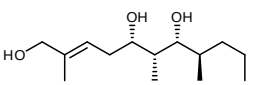


DFILE S2d C.als
COMNT single pulse dec
DATIM 2015-09-17 10:15
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 22.4 c
SLVNT CD3OD
EXREF 49.00 ppm
BF 1.20 Hz
RGAIN 56



S2d

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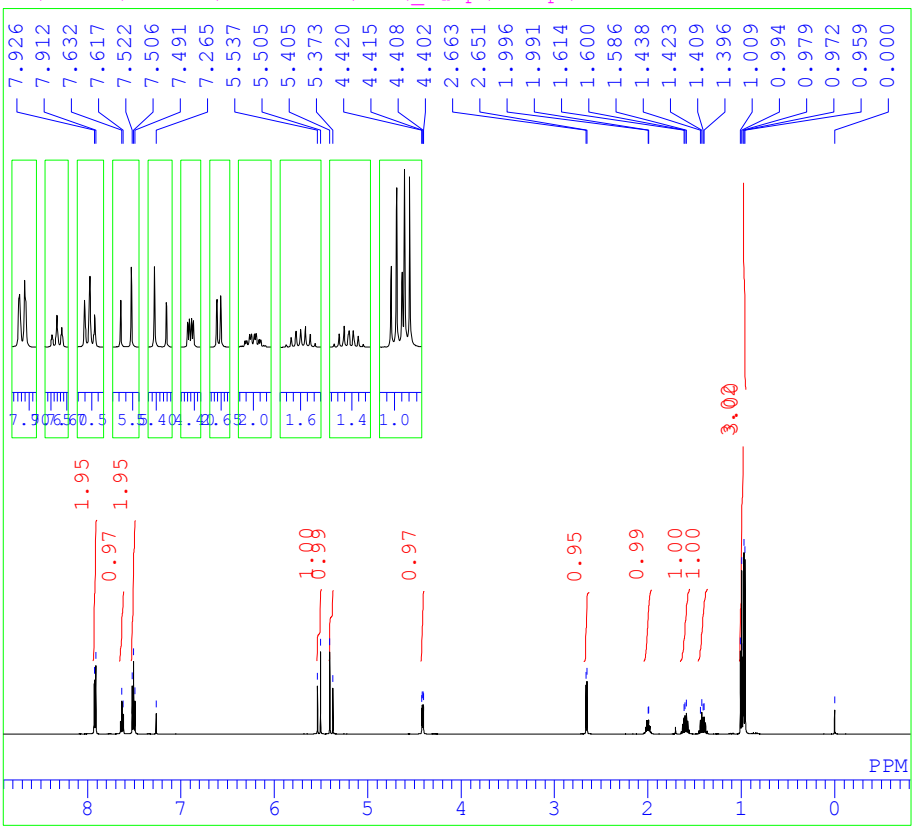
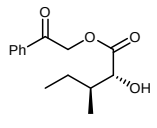


DFILE 11 H.als
COMNT single_pulse
DATIM 2016-01-12 12:07
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 19.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40



11

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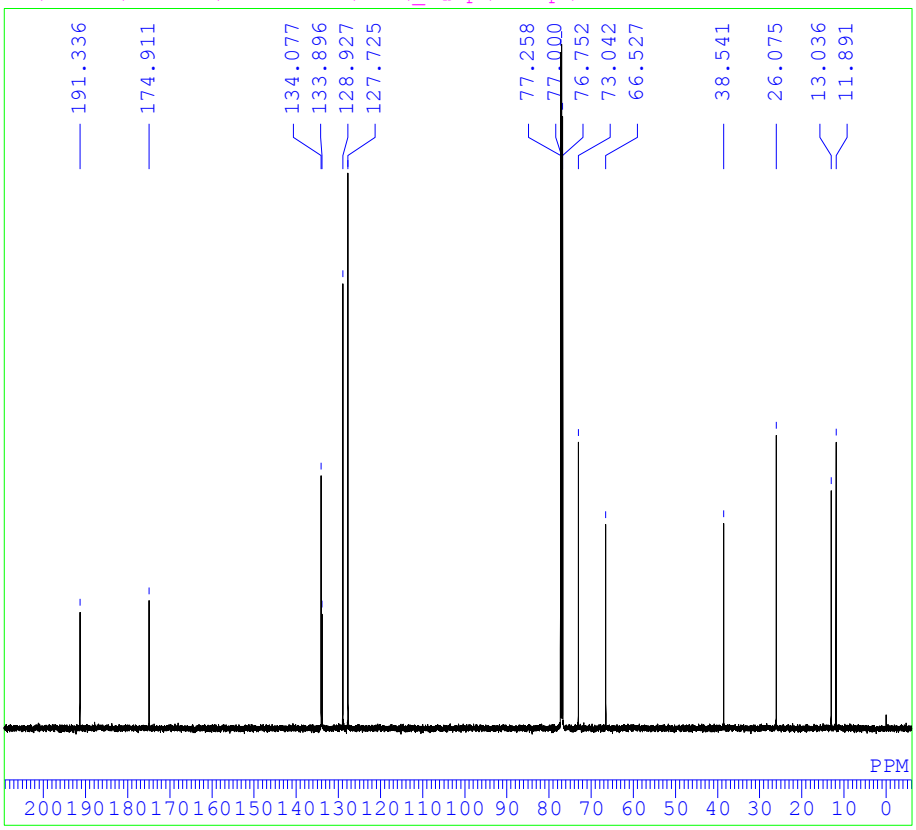
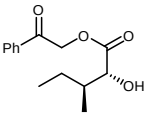
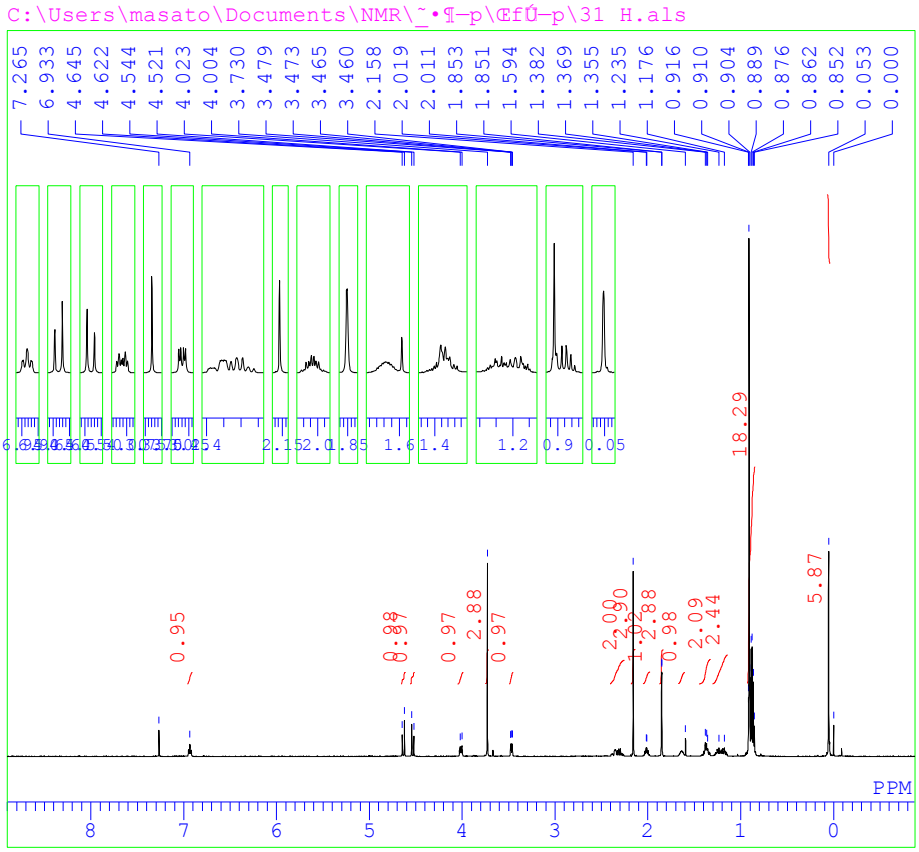


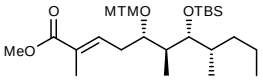
DFILE 11 C.als
COMNT single pulse dec
DATIM 2016-01-12 12:55
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 54



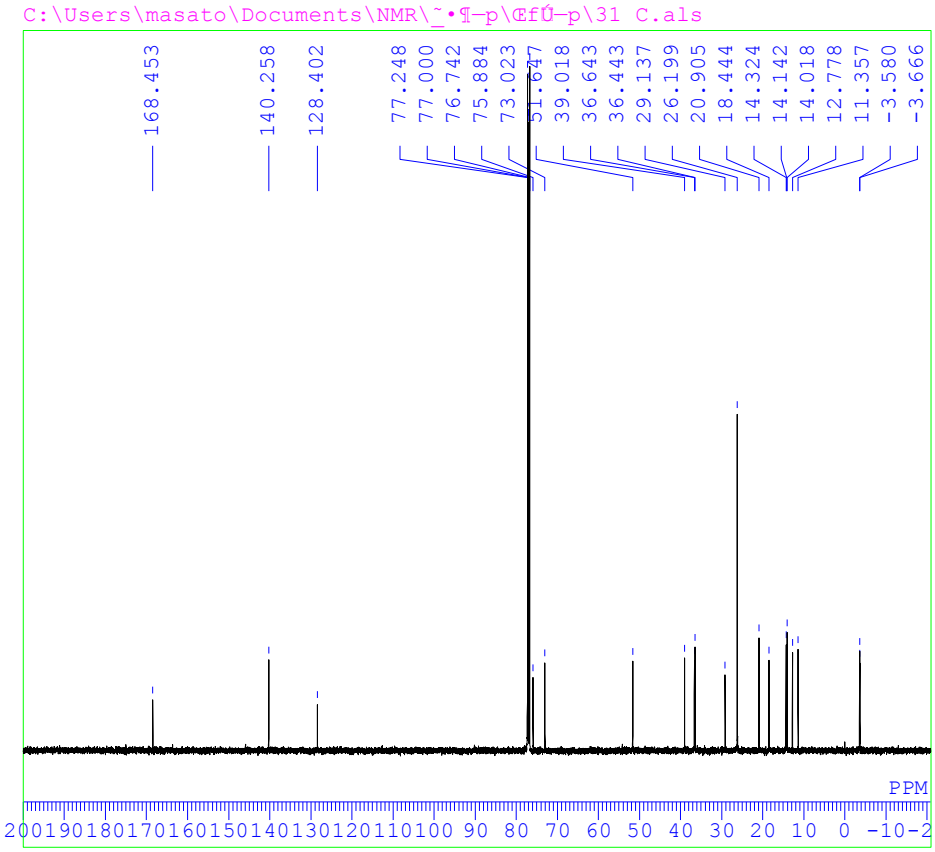
11



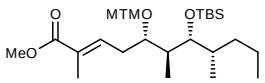
DFILE 31 H.als
COMNT single_pulse
DATIM 2016-01-20 08:39
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38



31



DFILE 31 C.als
COMNT single pulse dec
DATIM 2016-01-20 09:29
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 19.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 52



31

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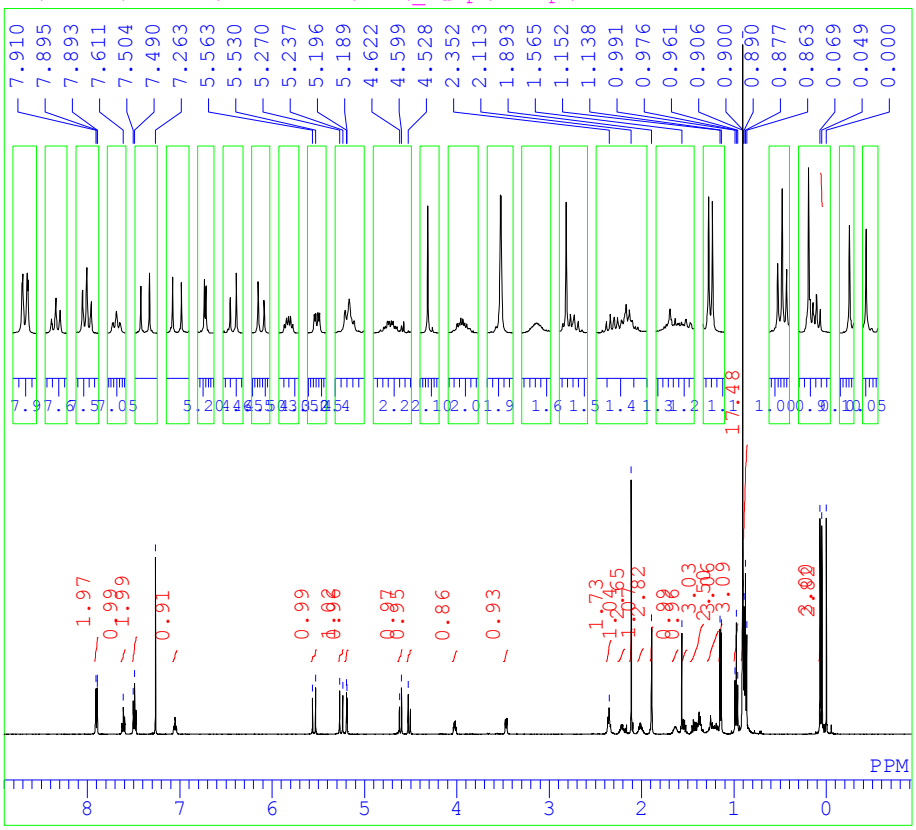
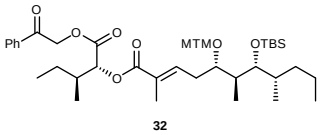


DFILE 32 H.als
COMNT single_pulse
DATIM 2016-01-20 19:06
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 19.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 46



32

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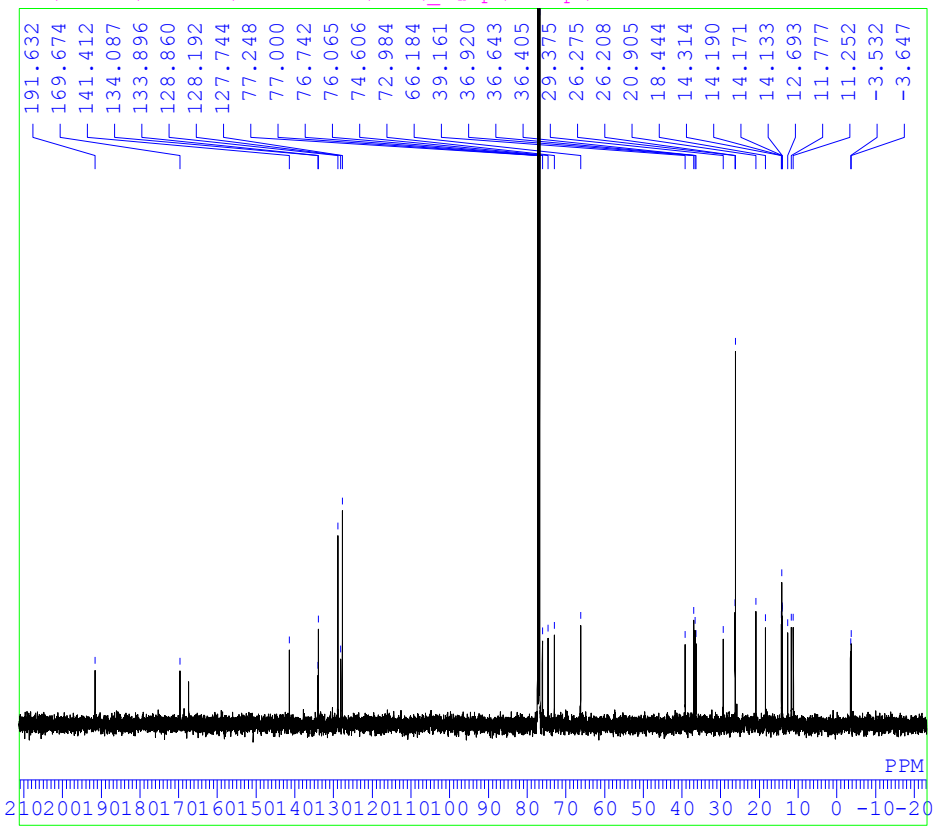
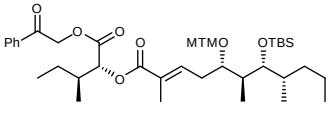


DFILE 32 C.als
COMNT single pulse dec
DATIM 2016-01-19 13:48
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 956
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 20.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 54



32

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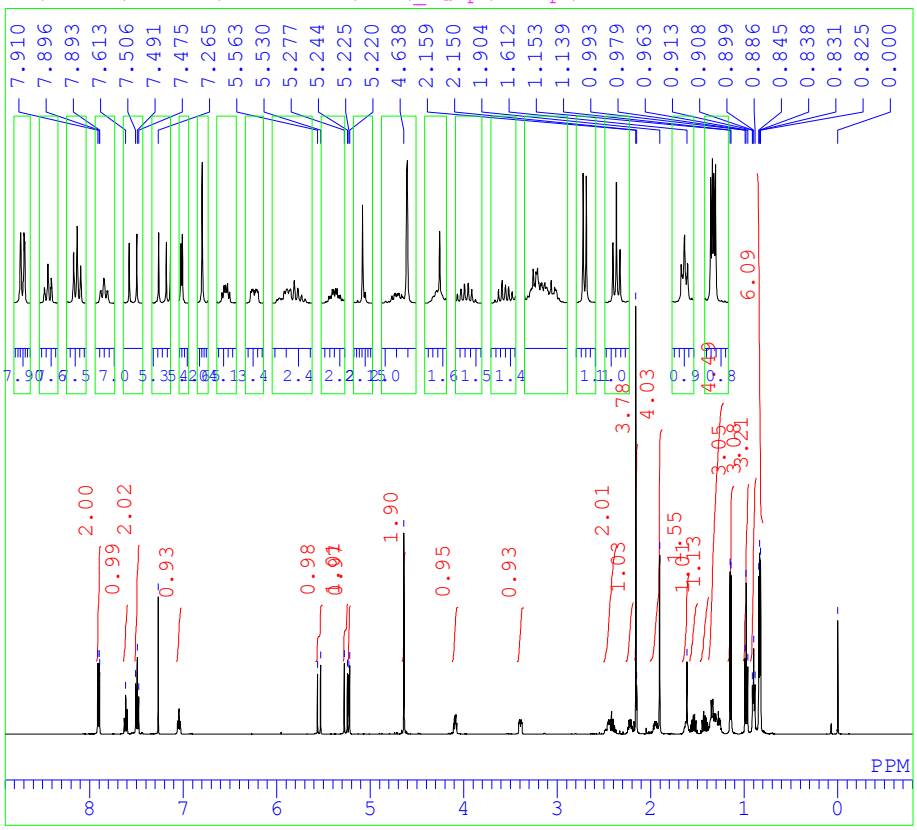
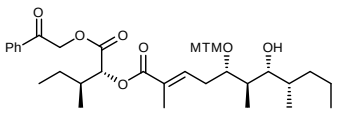
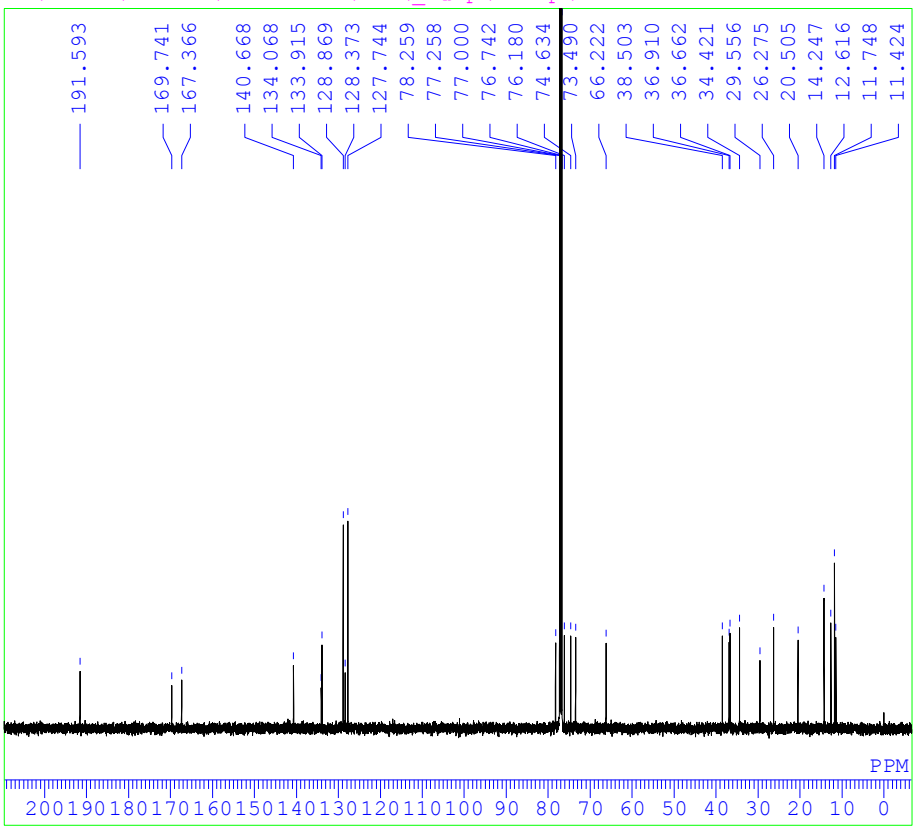


DFILE 7 H.als
COMNT single_pulse
DATIM 2016-01-22 08:39
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 13107
FREQU 7507.39 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 6.82 use
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 42

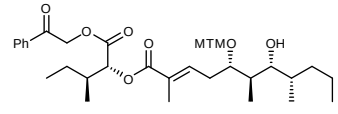


7

C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\7 C.als

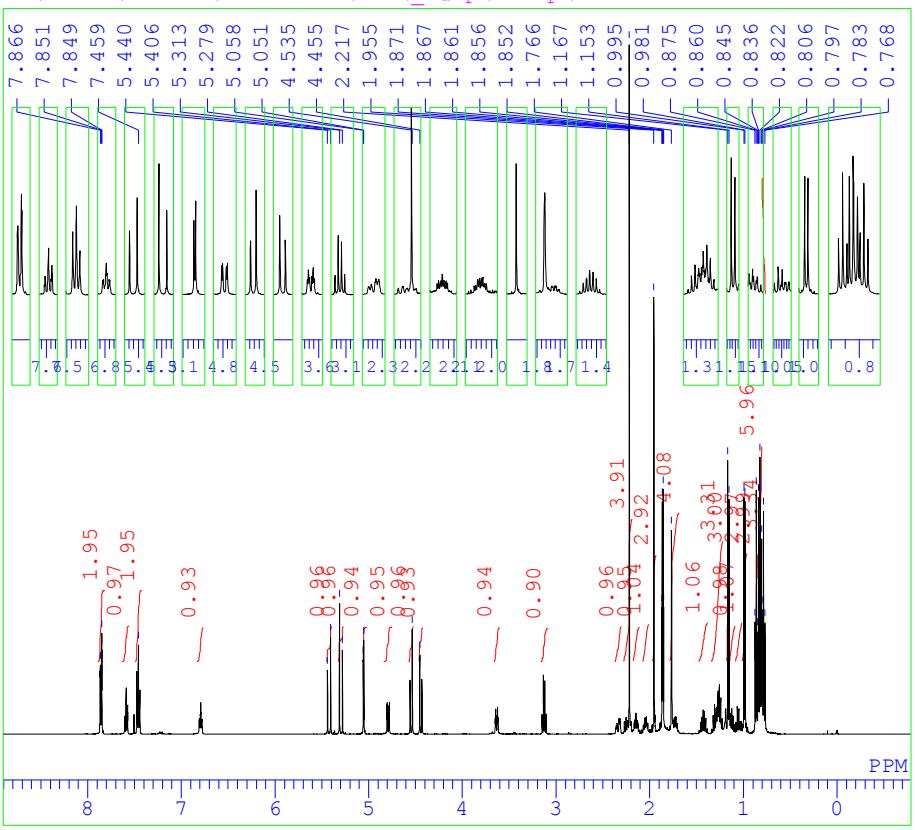


DFILE 7 C.als
COMNT single pulse dec
DATIM 2016-01-22 16:38
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 26214
FREQU 31446.06 Hz
SCANS 882
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.50 use
IRNUC 1H
CTEMP 20.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60

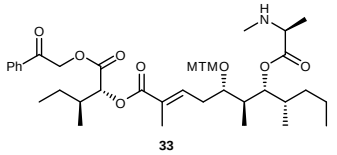


7

C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\33 H.als

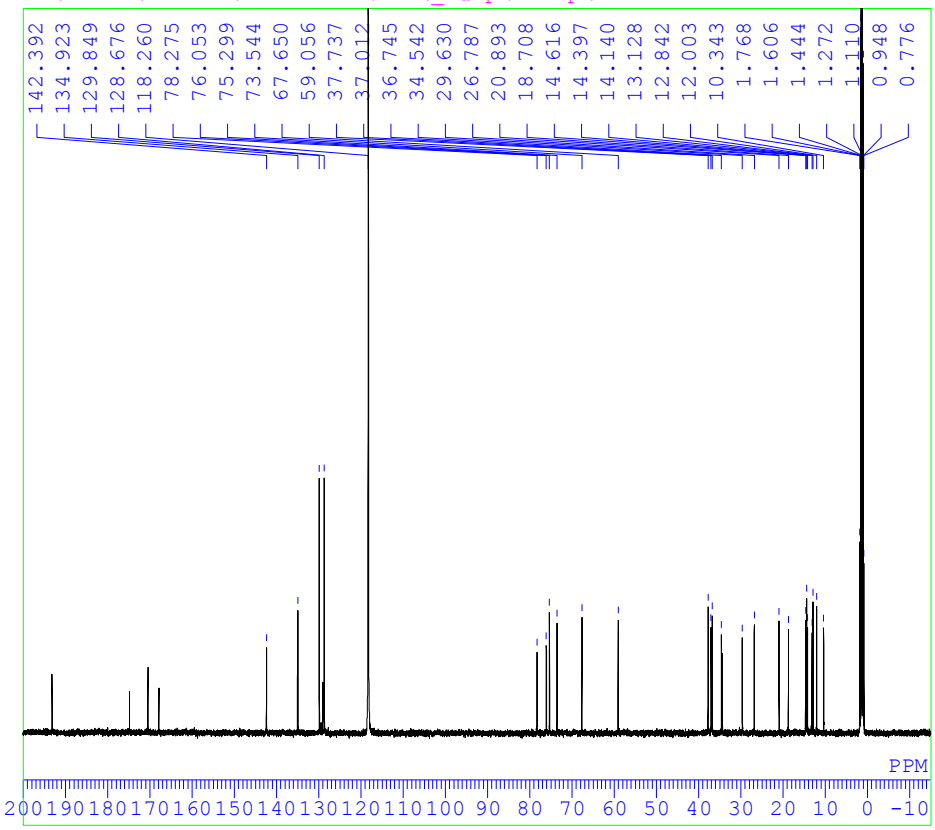



```
DFILE  33 H.als
COMNT  single_pulse
DATIM  2015-12-05 14:57
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1             6.82 use
IRNUC  1H
CTEMP          20.7 c
SLVNT  CD3CN
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            42
```

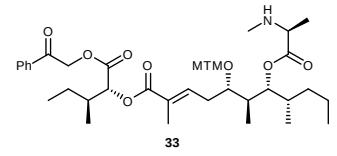


33

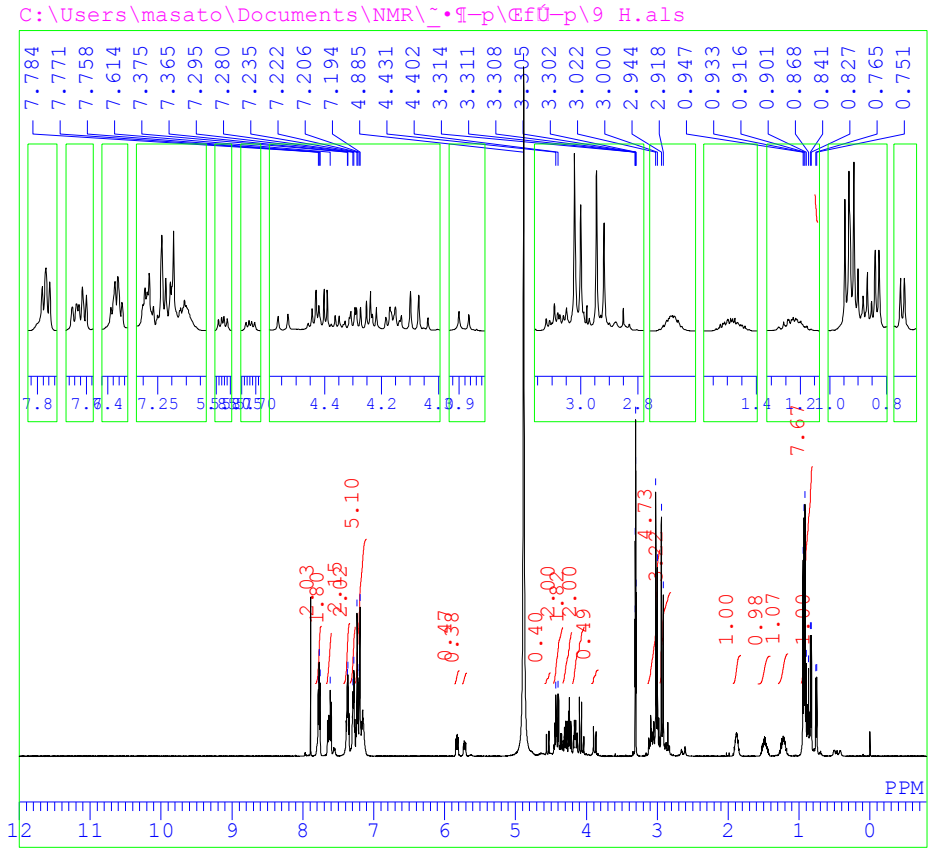
C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\33 C.als



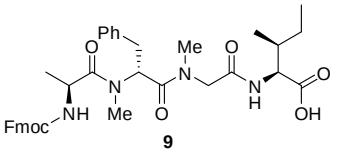

```
DFILE  33 C.als
COMNT  single pulse dec
DATIM  2015-03-04 20:03
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS          1000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1             3.50 use
IRNUC  1H
CTEMP          21.7 c
SLVNT  CD3CN
EXREF        118.26 ppm
BF             1.20 Hz
RGAIN            52
```



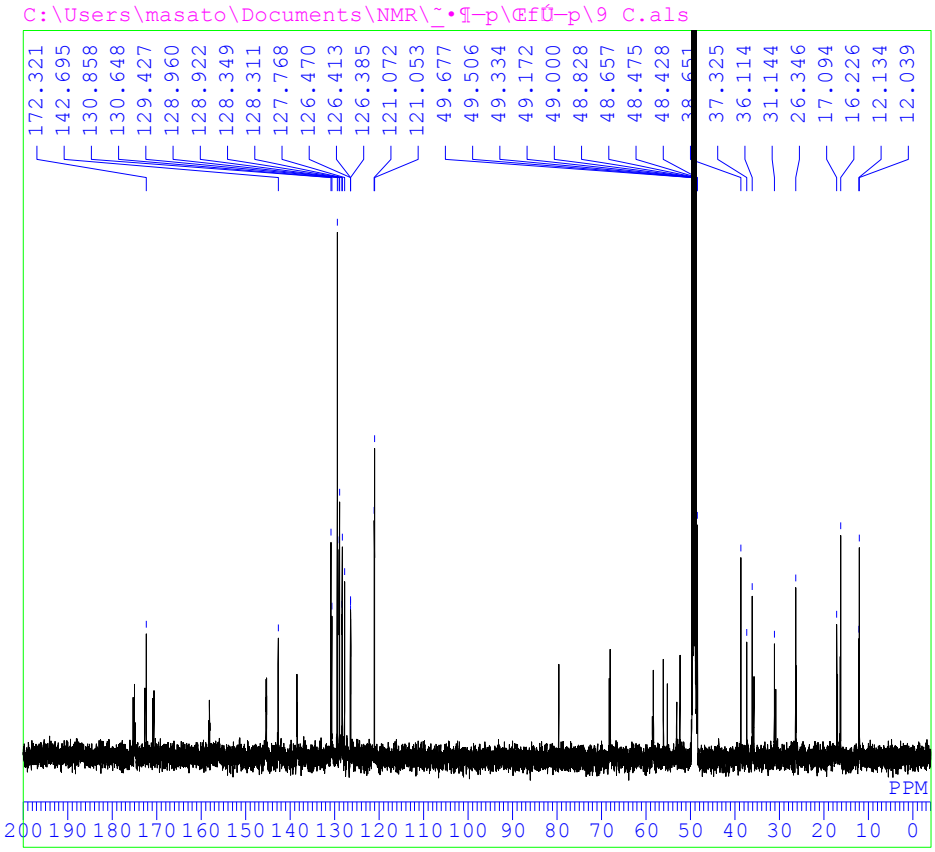
33



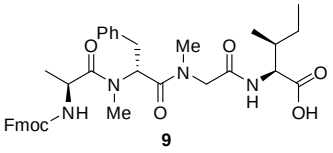

```
DFILE  9 H.als
COMNT  single_pulse
DATIM  2016-08-24 14:31
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            5.95 use
IRNUC  1H
CTEMP         21.5 c
SLVNT  CD3OD
EXREF         0.00 ppm
BF            0.12 Hz
RGAIN           36
```



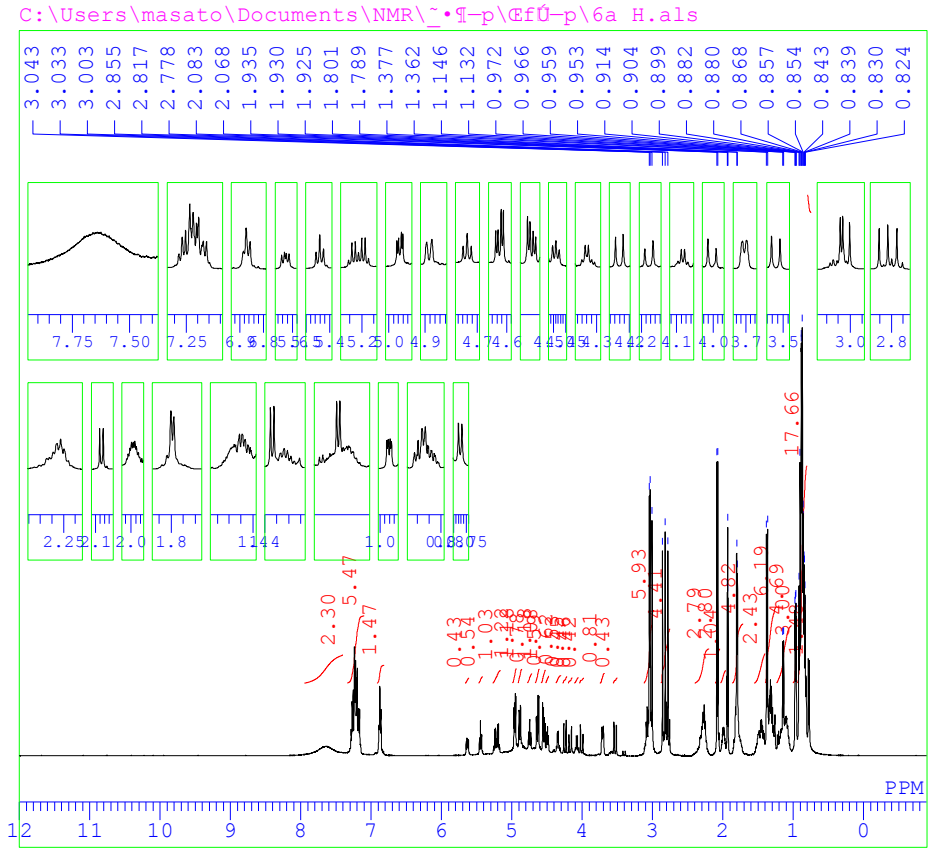
9



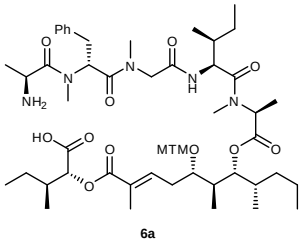

```
DFILE  9 C.als
COMNT  single pulse dec
DATIM  2016-08-24 14:58
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS           557
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.33 use
IRNUC  1H
CTEMP         22.0 c
SLVNT  CD3OD
EXREF        49.00 ppm
BF            1.20 Hz
RGAIN           48
```



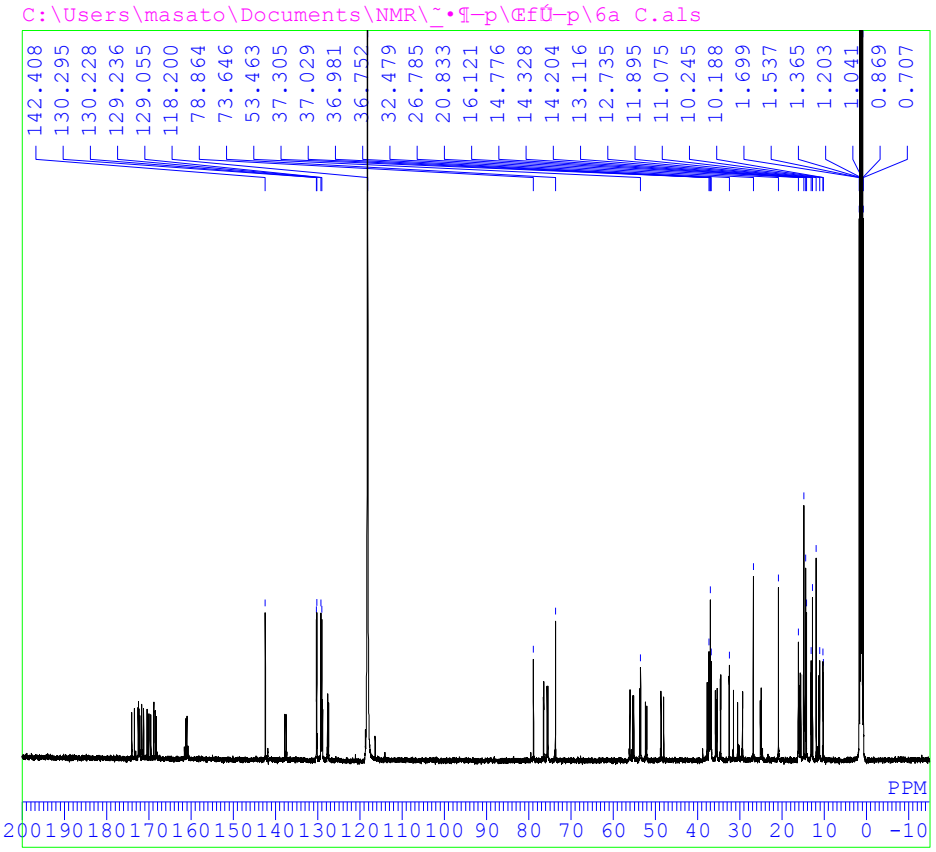
9



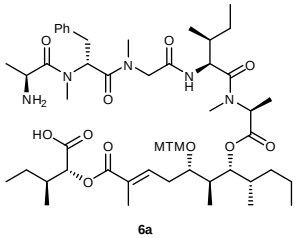

```
DFILE  6a H.als
COMNT  single_pulse
DATIM  2015-02-27 09:21
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         26214
FREQU      10019.89 Hz
SCANS             8
ACQTM        2.6162 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.4 c
SLVNT  CD3CN
EXREF          1.93 ppm
BF             0.12 Hz
RGAIN            34
```



6a

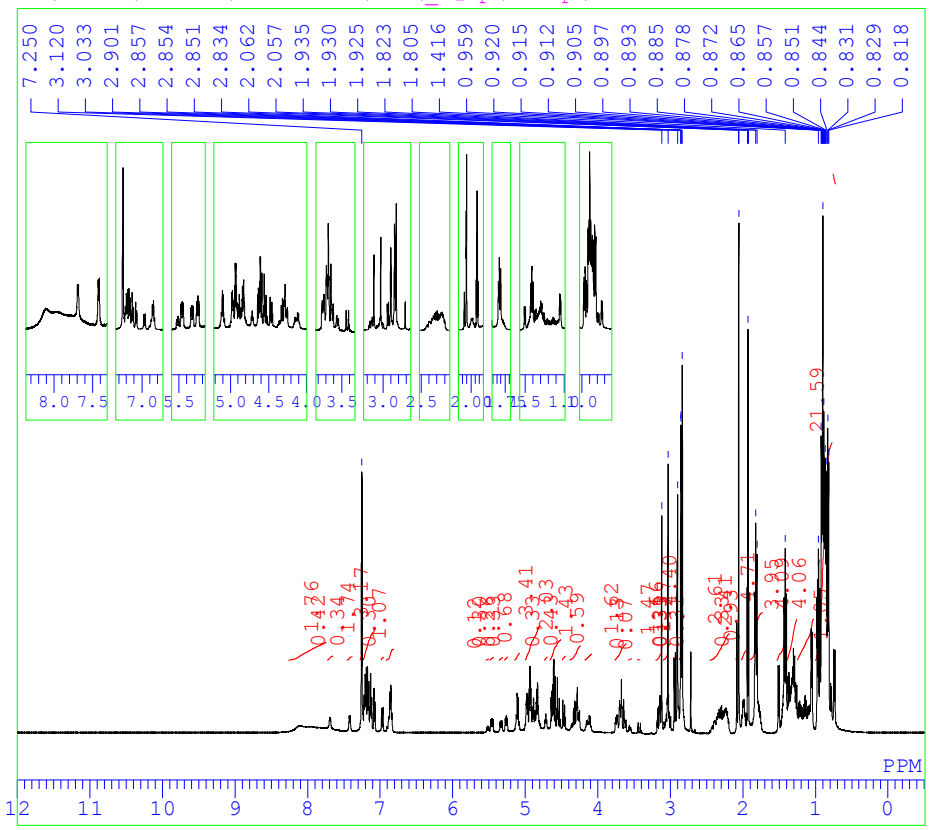



```
DFILE  6a C.als
COMNT  single pulse dec
DATIM  2015-02-28 08:27
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS         12662
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.5 c
SLVNT  CD3CN
EXREF        118.20 ppm
BF             1.20 Hz
RGAIN            54
```

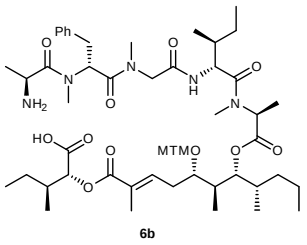


6a

C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\6b H.als

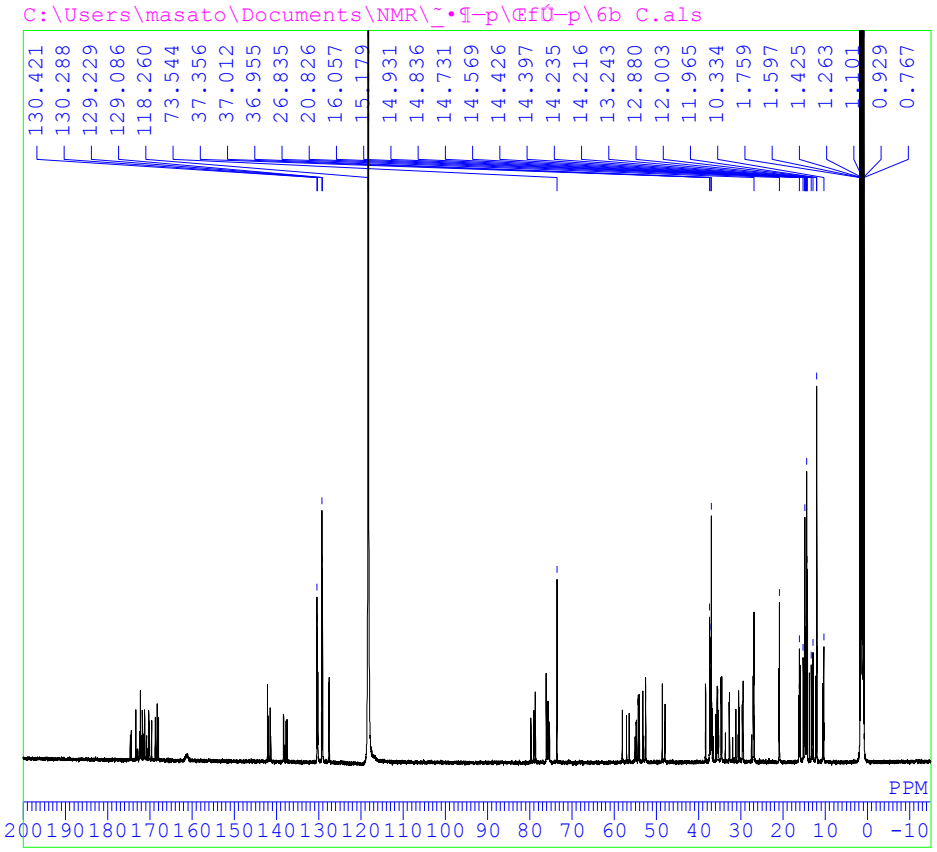



```
DFILE  6b H.als
COMNT  single_pulse
DATIM  2015-02-25 15:59
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         52428
FREQU      10019.89 Hz
SCANS             8
ACQTM        5.2324 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.3 c
SLVNT  CD3CN
EXREF          1.93 ppm
BF             0.12 Hz
RGAIN            34
```

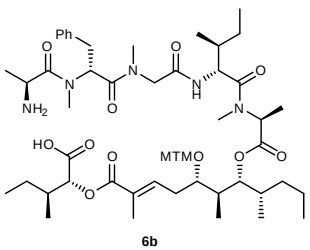


6b

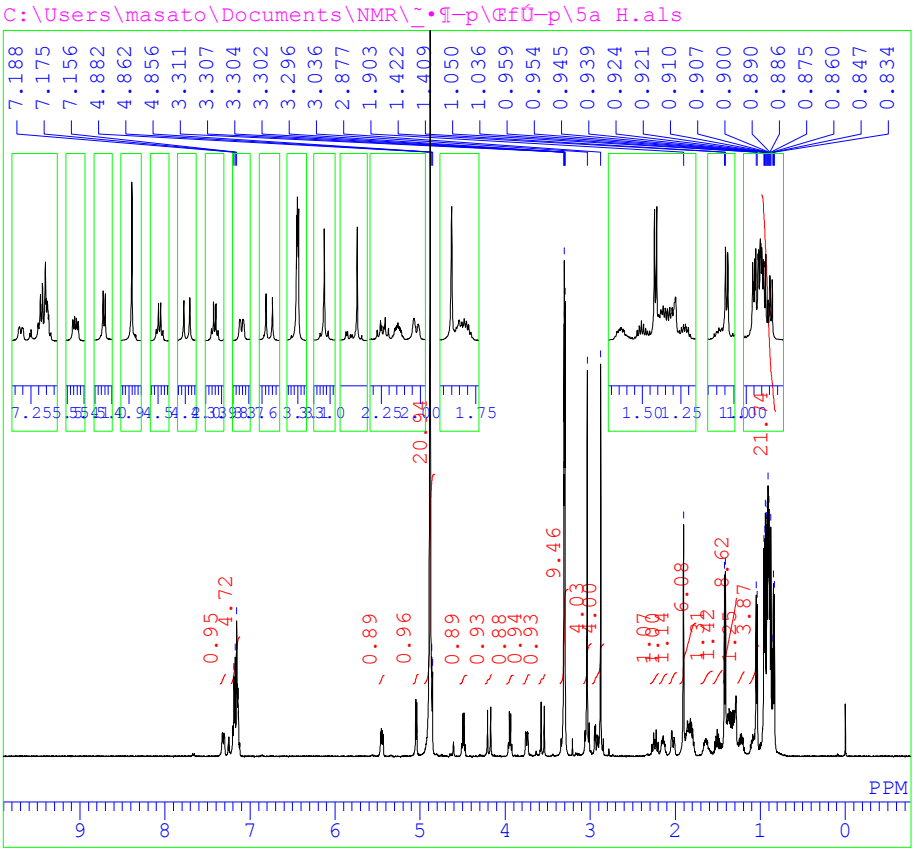
C:\Users\masato\Documents\NMR\˜•¶—p\ŒfÚ—p\6b C.als



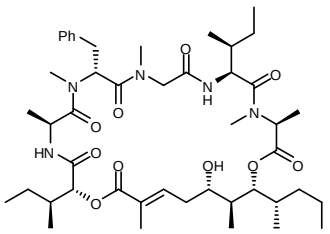

```
DFILE  6b C.als
COMNT  single pulse dec
DATIM  2015-03-02 08:34
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS         49633
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          20.8 c
SLVNT  CD3CN
EXREF        118.26 ppm
BF             1.20 Hz
RGAIN            52
```



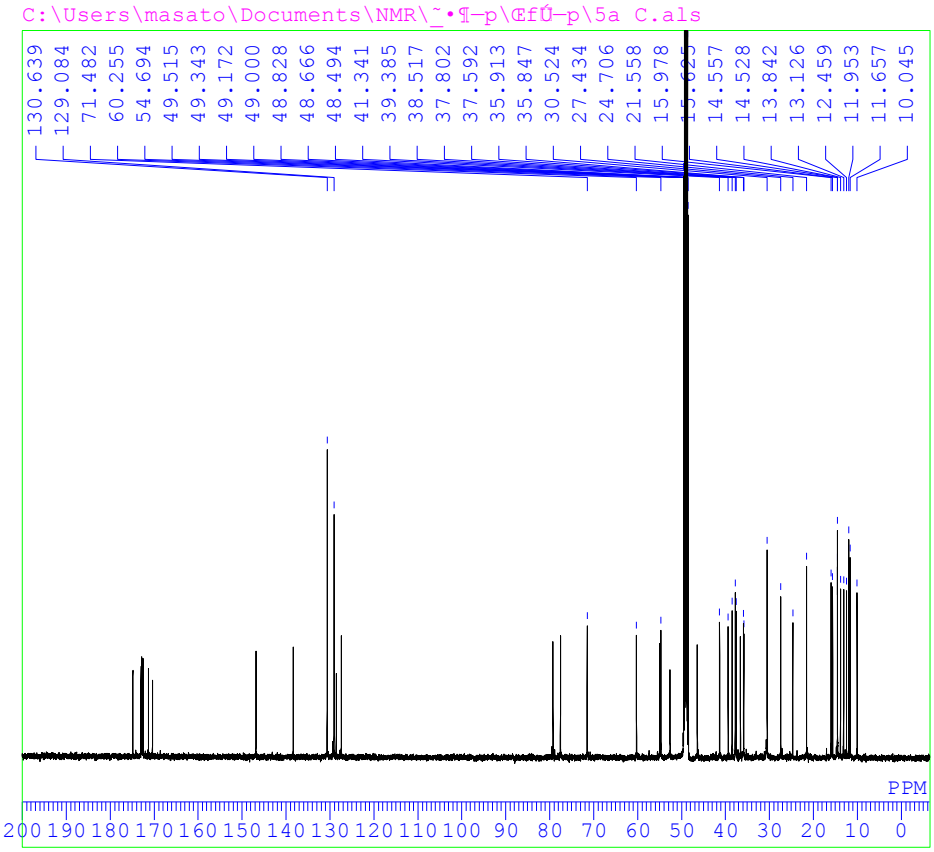
6b



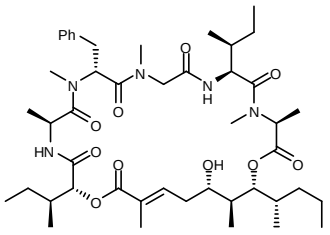

```
DFILE  5a H.als
COMNT  single_pulse
DATIM  2015-02-24 13:05
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS             8
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.5 c
SLVNT  CD3OD
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            42
```



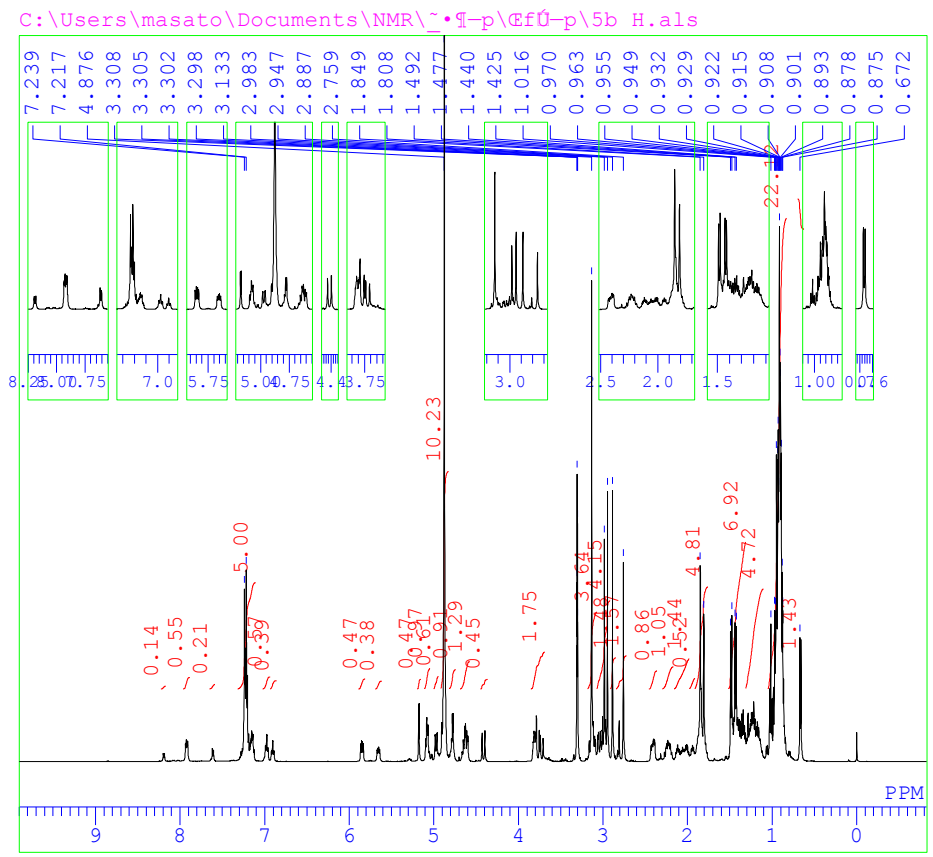
5a



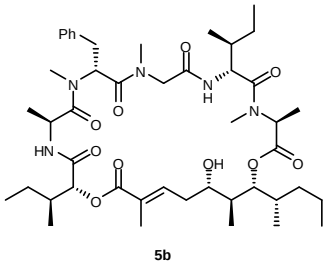

```
DFILE  5a C.als
COMNT  single pulse dec
DATIM  2015-05-01 07:49
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS         12000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.7 c
SLVNT  CD3OD
EXREF         49.00 ppm
BF             1.20 Hz
RGAIN            54
```



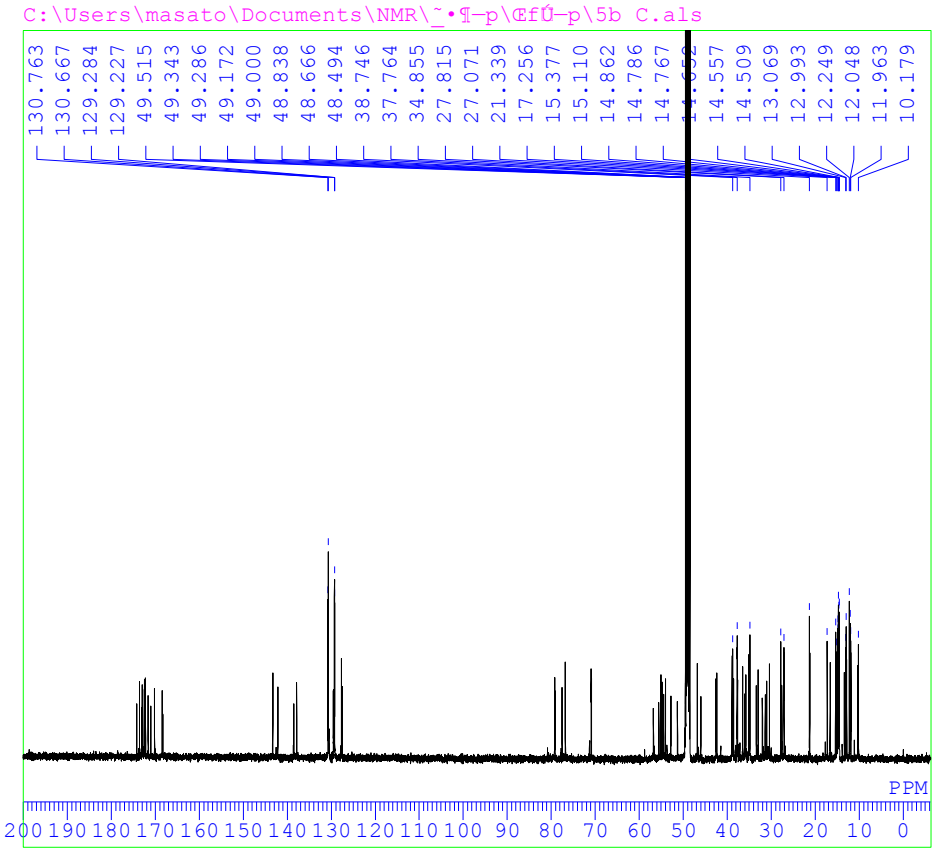
5a



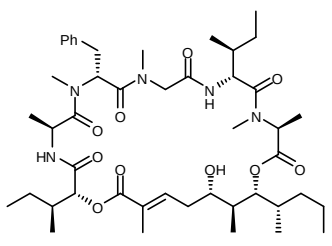

```
DFILE  5b H.als
COMNT  single_pulse
DATIM  2015-04-07 22:21
OBNUC  1H
EXMOD  single_pulse.ex2
OBFRQ        500.16 MHz
OBSET          2.41 KHz
OBFIN          6.01 Hz
POINT         13107
FREQU       7507.39 Hz
SCANS            80
ACQTM        1.7459 sec
PD           5.0000 sec
PW1            6.82 use
IRNUC  1H
CTEMP          21.7 c
SLVNT  CD3OD
EXREF          0.00 ppm
BF             0.12 Hz
RGAIN            36
```



5b




```
DFILE  5b C.als
COMNT  single pulse dec
DATIM  2015-04-08 07:48
OBNUC  13C
EXMOD  single_pulse_dec
OBFRQ        125.77 MHz
OBSET          7.87 KHz
OBFIN          4.21 Hz
POINT         26214
FREQU      31446.06 Hz
SCANS         12000
ACQTM        0.8336 sec
PD           2.0000 sec
PW1            3.50 use
IRNUC  1H
CTEMP          22.0 c
SLVNT  CD3OD
EXREF         49.00 ppm
BF             1.20 Hz
RGAIN            54
```



5b

HPLC Chromatograms. (a) peptide **9**. *HPLC conditions*: 45 to 75% linear gradient of MeCN in 0.1% TFA solution over 30 min; (b) odoamide **5a**. *HPLC conditions*: 76% MeCN in H_2O; (c) [D-*allo*-Ile]-odoamide **5b**. *HPLC conditions*: 76% MeCN in H_2O.