Supporting Information

Et_3B-Mediated Two- and Three-Component Coupling Reactions via Radical Decarbonylation of α-Alkoxyacyl Tellurides: Single-Step Construction of Densely Oxygenated Carboskeletons

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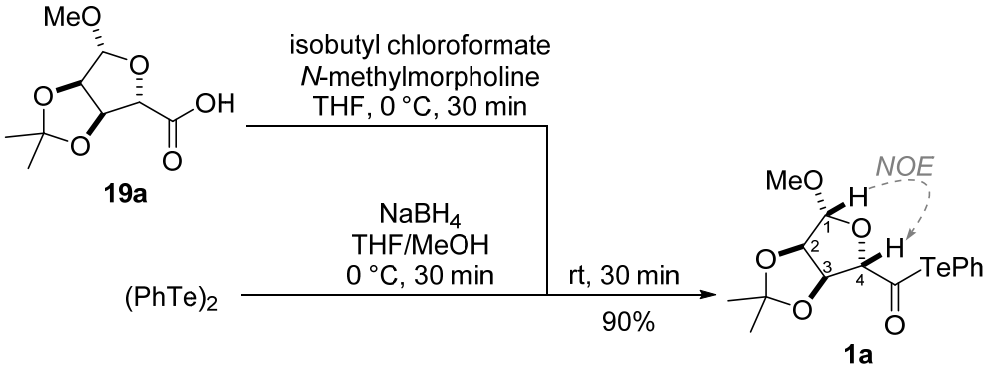
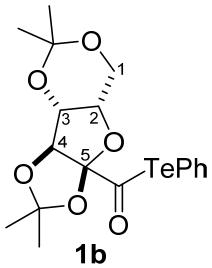
General Information: All Et_3B-mediated radical reactions were carried out under air. All other reactions sensitive to air or moisture were carried out under argon atmosphere in dry solvents under anhydrous conditions, unless otherwise noted. THF and CH_2Cl_2 were purified by Glass Contour solvent dispensing system (Nikko Hansen & Co., Ltd., Osaka, Japan). All other reagents were used as supplied unless otherwise noted. Analytical thin-layer chromatography (TLC) was performed using E. Merck Silica gel 60 F_{254} pre-coated plates, 0.25 mm. Preparative thin-layer chromatography was performed using Merck silica gel 60 F_{254} pre-coated plates, 0.5 mm. Flash chromatography was performed using 40-50 μm Silica Gel 60N (Kanto Chemical Co., Inc.), unless otherwise noted. Optical rotations were measured on JASCO DIP-1000 Digital Polarimeter at room temperature using the sodium D line. Melting points were measured on Yanaco MP-J3 micro melting point apparatus, and are uncorrected. Infrared (IR) spectra were recorded on JASCO FT:IR-4100 spectrometer. ^1H and ^{13}C NMR spectra were recorded on JEOL JNM-ECX-500, JNM-ECA-500, or JNM-ECS-400 spectrometer. Chemical shifts were reported in ppm on the δ scale relative to $CHCl_3$ (δ = 7.26 for ^1H NMR), $CDCl_3$ (δ = 77.0 for ^{13}C NMR), C_6D_5H (δ = 7.16 for ^1H NMR), C_6D_6 (δ = 128.0 for ^{13}C NMR) and $CO(CD_3)_2$ (δ = 206.26 for ^{13}C NMR) as internal references. Signal patterns are indicated as s, singlet; d, doublet; t, triplet; q, quartet, m; multiplet, br; broaden peak. High resolution mass spectra was measured on JEOL JMS-T100LP.

Experimental

Preparation of Acyl Telluride

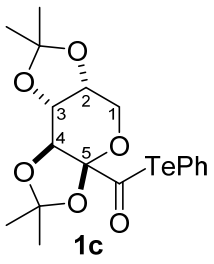


***General Procedure A*: Acyl telluride 1a.** *N*-methylmorpholine (910 μL, 8.3 mmol) and isobutyl chloroformate (950 μL, 7.3 mmol) were added to a solution of carboxylic acid **19a**[S1] (1.5 g, 6.9 mmol) in THF (34 mL) at 0 °C. MeOH (3.4 mL) was added to a suspension of diphenyl ditelluride (2.8 g, 6.8 mmol) and $NaBH_4$ (780 mg, 20.6 mmol) in THF (34 mL) at 0 °C. After the both reaction mixtures were stirred for 30 min at 0 °C, the former reaction mixture was added dropwise to the latter one. The resultant reaction mixture was stirred for another 30 min at room temperature, and then was filtered through a pad of silica gel (30 g, EtOAc). The filtrate was concentrated, and the residue was purified by flash chromatography on silica gel (50 g, hexane/EtOAc = 10:1) to afford **1a** (2.5 g, 6.2 mmol) in 90% yield: yellow oil; $[\alpha]_D^{27}$ = -89.5 (*c* 1.0, $CHCl_3$); m.p. 53-56 °C; IR (film) ν 1706, 1435, 1374, 1212, 1158, 1091, 1040, 927, 865, 733 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (3H, s, C*H*$_3$ of acetonide), 1.49 (3H, s, C*H*$_3$ of acetonide), 3.60 (3H, s, -OC*H*$_3$), 4.38 (1H, d, *J* = 0.9 Hz, H4), 4.55 (1H, d, *J* = 6.0 Hz, H2), 5.01 (1H, dd, *J* = 6.0, 1.4 Hz, H3), 5.15 (1H, s, H1), 7.29-7.41 (3H, m, aromatic), 7.69-7.72 (2H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.2, 26.6, 57.8, 80.6, 84.5, 97.8, 111.2, 113.0, 113.8, 128.7, 129.4 (2C), 140.0 (2C), 206.5; HRMS (ESI) calcd for $C_{15}H_{18}O_5TeNa$ $[M+Na]^+$ 431.0109, found 431.0101.



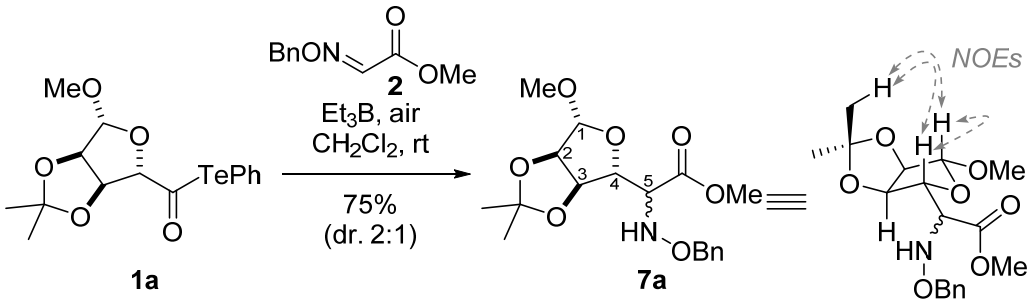
Acyl telluride 1b. According to the general procedure A, **1b** (1.4 g) was synthesized in 83% yield from carboxylic acid **19b**[S2] (1.0 g, 3.6 mmol) by using *N*-methylmorpholine (450 μL, 4.1 mmol) and isobutyl chloroformate (530 μL, 4.1 mmol) in THF (17 mL), and diphenyl ditelluride (1.5 g, 3.7 mmol), $NaBH_4$ (388 mg, 10.3 mmol) and MeOH (1.7 mL) in THF (17 mL). The residue was purified by flash chromatography on silica gel (40 g, hexane/EtOAc 5:1 to 2:1): yellow solid; $[\alpha]_D^{24}$ = -70.3 (*c* 1.0, $CHCl_3$); m.p. 144-146 °C; IR (film) ν 1711, 1379, 1177, 1121, 1089, 1037, 912, 816, 734 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.43 (3H, s, C*H*$_3$ of acetonide), 1.45 (3H, s, C*H*$_3$ of acetonide), 1.49 (3H, s, C*H*$_3$ of

acetonide), 1.53 (3H, s, C*H*$_3$ of acetonide), 4.17 (1H, dd, *J* = 13.7, 2.3 Hz, H1$_A$), 4.22 (1H, d, *J* = 13.7 Hz, H1$_B$), 4.27 (1H, dd, *J* = 2.3, 2.3 Hz, H2), 4.33 (1H, d, *J* = 2.3 Hz, H3), 4.53 (1H, s, H4), 7.29-7.40 (3H, m, aromatic), 7.72-7.76 (2H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.9, 25.8, 27.0, 28.8, 59.9, 72.3, 74.0, 87.2, 98.0, 114.5, 115.0, 116.3, 128.6, 129.4 (2C), 139.9 (2C), 206.4; HRMS (ESI) calcd for $C_{18}H_{22}O_6TeNa$ $[M+Na]^+$ 487.0371, found 487.0385.



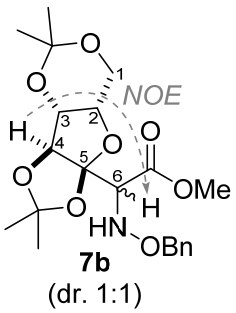
Acyl telluride 1c. According to the general procedure A, **1c** (845 mg) was synthesized from carboxylic acid **19c**[S3] (616 mg, 2.25 mmol) in 81% yield by using *N*-methylmorpholine (300 μL, 2.8 mmol) and isobutyl chloroformate (350 μL, 2.7 mmol) in THF (11 mL), and diphenyl ditelluride (921 mg, 2.25 mmol), $NaBH_4$ (255 mg, 6.74 mmol) and MeOH (1.1 mL) in THF (11 mL). The residue was purified by flash chromatography on silica gel (40 g, hexane/EtOAc 10:1): yellow solid; $[\alpha]_D^{22}$ = -73.5 (*c* 1.0, $CHCl_3$); m.p. 150-153 °C; IR (film) ν 1717, 1383, 1253, 1212, 1173, 1072, 996, 890, 773, 733 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.34 (3H, s, C*H*$_3$ of acetonide), 1.42 (3H, s, C*H*$_3$ of acetonide), 1.54 (3H, s, C*H*$_3$ of acetonide), 1.61 (3H, s, C*H*$_3$ of acetonide), 3.97 (1H, dd, *J* = 14.6, 1.4 Hz, H1$_A$), 4.01 (1H, dd, *J* = 14.6, 1.4 Hz, H1$_B$), 4.27 (1H, ddd, *J* = 7.8, 1.4, 1.4 Hz, H2), 4.50 (1H, d, *J* = 2.8 Hz, H4), 4.58 (1H, dd, *J* = 7.8, 2.8 Hz, H3), 7.29-7.41 (3H, m, aromatic), 7.70-7.74 (2H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.9, 24.7, 26.0, 26.2, 62.0, 69.7, 70.0, 72.0, 104.5, 109.3, 110.3, 114.4, 128.7, 129.3 (2C), 140.0 (2C), 206.4; HRMS (ESI) calcd for $C_{18}H_{22}O_6TeNa$ $[M+Na]^+$ 487.0371, found 487.0371.

Two Component Reactions Using Glyoxylic Oxime Ether



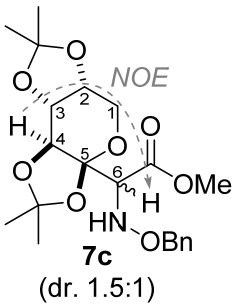
***General procedure B*: *O*-Benzyl-hydroxyl amine 7a**. Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) was added to a solution of acyl telluride **1a** (50 mg, 0.12 mmol) and glyoxylic oxime ether **2**[S4] (48 mg, 0.25 mmol) in CH_2Cl_2 (6.2 mL) at room temperature. The reaction mixture was stirred for 15 min at the same temperature, and was then concentrated. The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 5:1) to afford **7a** (33 mg, 0.090 mmol, dr. 2:1) in 75% yield: colorless oil; IR (film) ν 3259, 1743,

1454, 1373, 1239, 1205, 1160, 1093, 1061, 869 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.27 (1.00H, s, C*H*$_3$ of acetonide), 1.30 (2.00H, s, C*H*$_3$ of acetonide), 1.43 (1.00H, s, C*H*$_3$ of acetonide), 1.44 (2.00H, s, C*H*$_3$ of acetonide), 3.31 (3.00H, s, -OC*H*$_3$), 3.63 (0.67H, d, *J* = 10.1 Hz, H5), 3.64 (0.33H, d, *J* = 5.9 Hz, H5), 3.78 (1.00H, s, -CO_2C*H*$_3$), 3.79 (2.00H, s, -CO_2C*H*$_3$), 4.10 (0.67H, d, *J* = 10.1 Hz, H4), 4.47 (1.00H, d, *J* = 6.0 Hz, H2), 4.54 (0.33H, d, *J* = 5.9 Hz, H4), 4.56 (0.33H, d, *J* = 6.0 Hz, H3), 4.64 (0.33H, d, *J* = 11.9 Hz, -C*H*$_A$H$_B$Ph), 4.68 (0.67H, d, *J* = 11.9 Hz, -CH$_A$*H*$_B$Ph), 4.71 (0.33H, d, *J* = 11.9 Hz, -C*H*$_A$H$_B$Ph), 4.72 (0.67H, d, *J* = 11.9 Hz, -CH$_A$*H*$_B$Ph), 4.75 (0.67H, d, *J* = 6.0 Hz, H3), 4.86 (0.33H, s, H1), 4.92 (0.67H, s, H1), 7.28-7.38 (5H, m, aromatic); Detectable signals of ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.9, 25.1, 26.4, 52.2, 52.3, 55.7, 55.9, 65.9, 66.4, 76.2, 76.4, 82.1, 82.3, 84.8, 84.9, 85.0, 85.6, 110.0, 111.0, 112.3, 112.5, 127.9, 128.26, 128.29, 128.6, 128.8, 137.4, 137.7, 171.1, 172.5; HRMS (ESI) calcd for $C_{18}H_{25}NO_7Na$ $[M+Na]^+$ 390.1523, found 390.1513.



7b
(dr. 1:1)

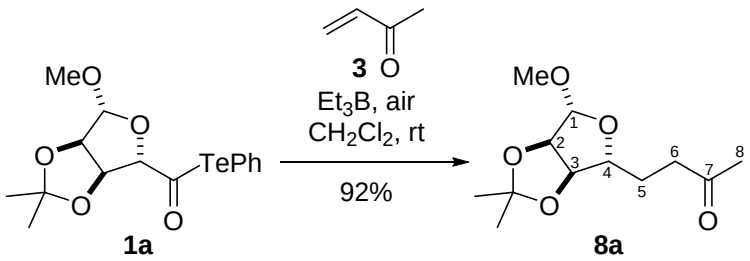
***O*-Benzyl-hydroxyl amine 7b [CAS: 1227367-16-9].**[S5] According to the general procedure B, **7b** (41 mg, 0.097 mmol, dr. 1:1) was synthesized in 88% yield from acyl telluride **1b** (50 mg, 0.11 mmol) and glyoxylic oxime ether **2** (42 mg, 0.22 mmol) by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 2:1): colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 1.24 (1.50H, s, C*H*$_3$ of acetonide), 1.29 (1.50H, s, C*H*$_3$ of acetonide), 1.35 (1.50H, s, C*H*$_3$ of acetonide), 1.36 (1.50H, s, C*H*$_3$ of acetonide), 1.40 (1.50H, s, C*H*$_3$ of acetonide), 1.41 (1.50H, s, C*H*$_3$ of acetonide), 1.43 (1.50H, s, C*H*$_3$ of acetonide), 1.44 (1.50H, s, C*H*$_3$ of acetonide), 3.80 (1.50H, s, -CO_2C*H*$_3$), 3.81 (1.50H, s, C*H*$_3$ of -CO_2C*H*$_3$), 3.97-4.08 (4.00H, m, H1$_A$, 1$_B$, 2, 6), 4.22-4.26 (1.00H, m, H3), 4.53 (0.50H, s, H4), 4.59 (0.50H, s, H4), 4.69 (1.00H, s, -C*H*$_A$*H*$_B$Ph), 4.74 (0.50H, d, *J* = 11.9 Hz, -C*H*$_A$H$_B$Ph), 4.78 (0.50H, d, *J* = 11.9 Hz, -CH$_A$*H*$_B$Ph), 7.26-7.38 (5.00H, m, aromatic); Detectable signals of ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.7, 18.9, 26.3, 27.3, 27.6, 28.5, 28.7, 52.2, 59.9, 60.1, 66.1, 68.8, 72.77, 72.84, 73.2, 76.1, 76.3, 85.6, 86.6, 97.3, 97.4, 111.9, 112.4, 113.1, 127.7, 128.21, 128.23, 128.6, 128.7, 137.7, 170.3, 171.3.



***O*-Benzyl-hydroxyl amine 7c**. According to the general procedure B, **7c** (16 mg of major diastereomer and 11 mg of minor diastereomer, 0.064 mmol, dr. 1.5:1) was synthesized in 66% yield from acyl telluride **1c** (45 mg, 0.097 mmol) and glyoxylic oxime ether **2** (38 mg, 0.20 mmol) by using Et_3B (1.04 M in hexane, 0.28 mL, 0.29 mmol) in CH_2Cl_2 (0.98 mL). The residue was purified by flash chromatography on silica gel (5.0 g, toluene/EtOAc 12:1): **major diastereomer**: colorless oil; $[\alpha]_D^{28}$ = -33.6 (*c* 0.5, $CHCl_3$); IR (film) ν 3272, 1746, 1454, 1382, 1250, 1217, 1152, 1118 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.29 (3H, s, C*H*$_3$ of acetonide), 1.34 (3H, s, C*H*$_3$ of acetonide), 1.47 (3H, s, C*H*$_3$ of acetonide), 1.52 (3H, s, C*H*$_3$ of acetonide), 3.68 (1H, d, *J* = 12.8 Hz, H1$_A$), 3.78 (3H, s, -CO_2CH_3), 3.81 (1H, d, *J* = 12.8 Hz, H1$_B$), 3.96 (1H, br s, H6), 4.19 (1H, d, *J* = 7.6 Hz, H2), 4.40 (1H, d, *J* = 2.4 Hz, H4), 4.57 (1H, dd, *J* = 7.6, 2.4 Hz, H3), 4.65 (1H, d, *J* = 11.9 Hz, -C*H*$_A$H$_B$Ph), 4.70 (1H, d, *J* = 11.9 Hz, -CH$_A$*H*$_B$Ph), 6.13 (1H, br s, N*H*), 7.26-7.40 (5H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.3, 25.2, 25.7, 26.6, 52.2, 61.6, 66.9, 70.2, 70.8, 71.3, 76.0, 101.4, 109.4, 109.6, 127.7, 128.3 (2C), 128.5 (2C), 137.7, 172.0; HRMS (ESI) calcd for $C_{21}H_{29}NO_8Na$ $[M+Na]^+$ 446.1785, found 446.1791.

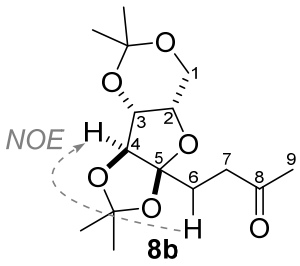
minor diastereomer: colorless oil; $[\alpha]_D^{28}$ = -15.6 (*c* 0.64, $CHCl_3$); IR (film) ν 3280, 1747, 1454, 1382, 1254, 1210, 1170, 1105 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.23 (3H, s, C*H*$_3$ of acetonide), 1.32 (3H, s, C*H*$_3$ of acetonide), 1.44 (3H, s, C*H*$_3$ of acetonide), 1.46 (3H, s, C*H*$_3$ of acetonide), 3.74-3.76 (2H, m, H1), 3.80 (3H, s, -CO_2CH_3), 3.81 (1H, s, H6), 4.15 (1H, d, *J* = 7.8 Hz, H2), 4.35 (1H, d, *J* = 2.3 Hz, H4), 4.49 (1H, dd, *J* = 7.8, 2.3 Hz, H3), 4.69 (1H, d, *J* = 11.9 Hz, -C*H*$_A$H$_B$Ph), 4.75 (1H, d, *J* = 11.9 Hz, -CH$_A$*H*$_B$Ph), 7.26-7.37 (5H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.9, 24.8, 25.7, 26.2, 52.2, 61.0, 70.0, 70.4, 70.9, 73.0, 76.4, 101.6, 108.5, 109.0, 127.8, 128.2 (2C), 128.7 (2C), 137.7, 170.3; HRMS (ESI) calcd for $C_{21}H_{29}NO_8Na$ $[M+Na]^+$ 446.1785, found 446.1789.

Two-Component Radical Reactions Using Enones

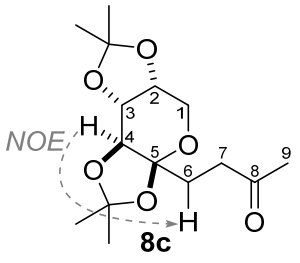


***General procedure C*: Ketone 8a [CAS: 81691-16-9].**[S6] Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) was added to a solution of acyl telluride **1a** (50 mg, 0.12 mmol) and methyl vinyl ketone **3** (20 μL, 0.24 mmol) in CH_2Cl_2 (6.2 mL) at room temperature. The reaction mixture

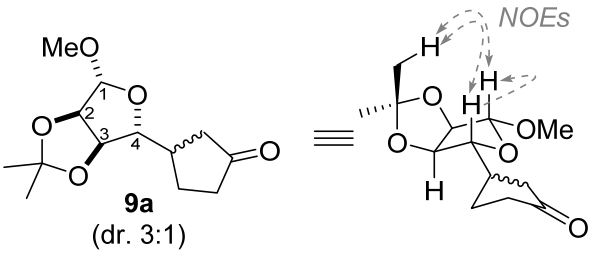
was stirred for 15 min at the same temperature, and was then concentrated. The residue was purified by flash chromatography on silica gel (10 g, hexane/EtOAc 3:1) to afford **8a** (28 mg, 0.11 mmol) in 92% yield: colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.29 (3H, s, C*H*$_3$ of acetonide), 1.45 (3H, s, C*H*$_3$ of acetonide), 1.72-1.86 (2H, m, H5$_A$, 5$_B$), 2.14 (3H, s, H8), 2.49-2.64 (2H, m, H6$_A$, 6$_B$), 3.32 (3H, s, -OC*H*$_3$), 4.10 (1H, dd, *J* = 9.2, 6.4 Hz, H4), 4.51 (1H, d, *J* = 6.0 Hz, H3), 4.58 (1H, d, *J* = 6.0 Hz, H2), 4.92 (1H, s, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.0, 26.5, 28.8, 30.0, 40.1, 55.1, 84.1, 85.4, 86.3, 109.6, 112.3, 207.7.



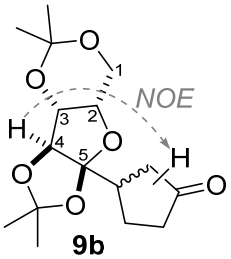
Ketone 8b. According to the general procedure C, **8b** (27 mg, 0.090 mmol) was synthesized in 82% yield from acyl telluride **1b** (50 mg, 0.11 mmol) and methyl vinyl ketone **3** (18 μL, 0.21 mmol) by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash chromatography on silica gel (10 g, hexane/EtOAc 2:1): colorless oil; $[\alpha]_D^{19}$ = 0.36 (*c* 2.5, $CHCl_3$); IR (film) ν 1715, 1378, 1239, 1197, 1167, 1120, 1079, 995, 836 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.31 (3H, s, C*H*$_3$ of acetonide), 1.36 (3H, s, C*H*$_3$ of acetonide), 1.40 (3H, s, C*H*$_3$ of acetonide), 1.44 (3H, s, C*H*$_3$ of acetonide), 2.14 (3H, s, H9), 2.14-2.22 (2H, m, H6$_A$, 6$_B$), 2.70-2.88 (2H, m, H7$_A$, 7$_B$), 3.986 (1H, d *J* = 13.7 Hz, H1$_A$), 3.989 (1H, dd, *J* = 2.3, 2.3 Hz, H2), 4.05 (1H, dd, *J* = 13.7, 2.3 Hz, H1$_B$), 4.207 (1H, d, *J* = 2.3 Hz, H3), 4.214 (1H, s, H4); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.6, 26.3, 27.2, 28.9, 29.9, 31.7, 38.4, 60.3, 71.8, 73.7, 86.7, 97.3, 110.8, 114.7, 208.4; HRMS (ESI) calcd for $C_{15}H_{24}O_6Na$ $[M+Na]^+$ 323.1465, found 323.1459.



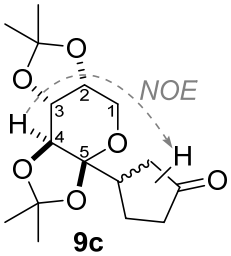
Ketone 8c [CAS: 151484-46-7].[S 7] According to the general procedure C, **8c** (23 mg, 0.077 mmol) was synthesized in 70% yield from acyl telluride **1c** (50 mg, 0.11 mmol) and methyl vinyl ketone **3** (18 μL, 0.21 mmol) by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash chromatography on silica gel (10 g, hexane/EtOAc 4:1): colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.33 (6H, s, C*H*$_3$x2 of acetonide), 1.48 (3H, s, C*H*$_3$ of acetonide), 1.49 (3H, s, C*H*$_3$ of acetonide), 1.97 (1H, ddd, *J* = 14.2, 10.1, 5.5 Hz, H6$_A$), 2.13 (1H, m, H6$_B$), 2.14 (3H, s, H9), 2.71 (1H, ddd, *J* = 18.8, 10.1, 5.5 Hz, H7$_A$), 2.83 (1H, ddd, *J* = 18.8, 10.1, 5.5 Hz, H7$_B$), 3.68 (1H, d, *J* = 13.3 Hz, H1$_A$), 3.81 (1H, dd, *J* = 13.3, 1.8 Hz, H1$_B$), 4.10 (1H, d, *J* = 2.7 Hz, H4), 4.19 (1H, dd, *J* = 8.2, 1.4 Hz, H2), 4.53 (1H, dd, *J* = 8.2, 2.3 Hz, H3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.9, 24.9, 25.8, 26.3, 29.9, 34.5, 37.6, 60.8, 70.4, 70.6, 73.8, 103.5, 107.5, 108.8, 208.5.



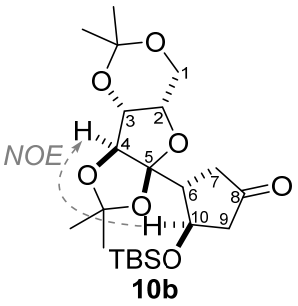
Ketone 9a. According to the general procedure C, **9a** (25 mg, 0.098 mmol, dr. 3:1) was synthesized in 82% yield from acyl telluride **1a** (50 mg, 0.12 mmol) and cyclopentenone **4** (21 μL, 0.26 mmol) by using Et_3B (1.03 M in hexane, 0.36 mL, 0.37 mmol) in CH_2Cl_2 (6.2 mL). The residue was purified by flash chromatography on silica gel (10 g, hexane/EtOAc 3:1): colorless oil; IR (film) ν 1743, 1373, 1210, 1160, 1107, 1091, 1061, 1029, 865 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.31 (0.75H, s, C*H*$_3$ of acetonide), 1.33 (2.25H, s, C*H*$_3$ of acetonide), 1.475 (2.25H, s, C*H*$_3$ of acetonide), 1.481 (0.75H, s, C*H*$_3$ of acetonide), 1.59-1.71 (0.50H, m), 1.76-1.97 (1.50H, m), 2.11-2.47 (5H, m), 3.33 (0.75H, s, -OC*H*$_3$), 3.39 (2.25H, s, -OC*H*$_3$), 3.98 (0.75H, dd, *J* = 10.5, 0.9 Hz, H4), 3.99 (0.25H, d, *J* = 10.5, 1.4 Hz, H4), 4.51 (0.75H, dd, *J* = 5.9, 0.9 Hz, H3), 4.595 (0.75H, d, *J* = 5.9 Hz, H2), 4.600 (0.25H, d, *J* = 6.4 Hz, H2), 4.64 (0.25H, dd, *J* = 6.4, 1.4 Hz, H3), 4.94 (0.25H, s, H1), 4.97 (0.75H, s, H1); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.95, 24.99, 25.8, 26.47, 26.48, 26.8, 37.8, 37.9, 40.8, 40.9, 41.6, 42.8, 55.37, 55.42, 82.4, 83.0, 85.4, 85.5, 90.7, 90.9, 109.4, 109.6, 112.55, 112.56, 217.7, 218.3; HRMS (ESI) calcd for $C_{13}H_{20}O_5Na$ $[M+Na]^+$ 279.1203, found 279.1196.



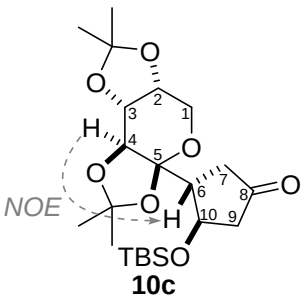
Ketone 9b. According to the general procedure C, **9c** (22 mg, 0.070 mmol, dr. 1.3:1) was synthesized in 64% yield from acyl telluride **2c** (50 mg, 0.11 mmol) and cyclopentenone **5** (18 μL, 0.22 mmol) by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 2:1): colorless oil; IR (film) ν 1740, 1375, 1241, 1195, 1121, 1080, 1009, 927, 833 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.32 (1.70H, s, C*H*$_3$ of acetonide), 1.34 (1.70H, s, C*H*$_3$ of acetonide), 1.35 (2.60H, s, C*H*$_3$ of acetonide), 1.40 (1.30H, s, C*H*$_3$ of acetonide), 1.41 (1.70H, s, C*H*$_3$ of acetonide), 1.46 (1.70H, s, C*H*$_3$ of acetonide), 1.48 (1.30H, s, C*H*$_3$ of acetonide), 2.06-2.24 (3.00H, m), 2.30-2.59 (3.00H, m), 2.62-2.75 (1.00H, m), 3.96-4.08 (3.00H, m, H1$_A$, 1$_B$, 2), 4.23 (0.43H, d, *J* = 2.0 Hz, H3), 4.26 (0.57H, d, *J* = 2.8 Hz, H3), 4.26 (0.57H, s, H4), 4.30 (0.57H, s, H4); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.7, 18.8, 24.2 (2C), 26.78, 26.81, 27.5, 27.6, 28.6, 28.8, 37.8, 38.1, 40.8, 40.9, 43.4, 44.0, 60.27, 60.34, 71.8, 71.9, 73.65, 73.67, 86.2, 86.5, 97.3, 97.5, 111.0, 111.2, 115.9, 116.2, 218.8, 219.1; HRMS (ESI) calcd for $C_{16}H_{24}O_6Na$ $[M+Na]^+$ 335.1465, found 335.1453.



Ketone 9c. According to the general procedure C, **9c** (18 mg, 0.058 mmol, dr. 1.4:1) was synthesized in 60% yield from acyl telluride **1c** (45 mg, 0.097 mmol) and cyclopentenone **5** (16 μL, 0.19 mmol) by using Et_3B (1.04 M in hexane, 0.28 mL, 0.29 mmol) in CH_2Cl_2 (4.9 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 2:1): colorless oil; IR (film) ν 1742, 1457, 1381, 1254, 1210, 1111 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.33 (1.25H, s, C*H*$_3$ of acetonide), 1.35 (3.50H, s, C*H*$_3$ of acetonide), 1.38 (1.25H, s, C*H*$_3$ of acetonide), 1.48 (1.75H, s, C*H*$_3$ of acetonide), 1.49 (1.25H, s, C*H*$_3$ of acetonide), 1.52 (1.75H, s, C*H*$_3$ of acetonide), 1.54 (1.25H, s, C*H*$_3$ of acetonide), 2.07-2.19 (3.00H, m), 2.27-2.59 (4.00H, m), 3.74 (0.42H, d, *J* = 13.2 Hz, H1$_A$), 3.76 (0.58H, d, *J* = 13.2 Hz, H1$_A$), 3.87 (0.42H, dd, *J* = 13.2, 2.0 Hz, H1$_B$), 3.88 (0.58H, dd, *J* = 13.2, 1.6 Hz, H1$_B$), 4.15 (0.58H, d, *J* = 2.4 Hz, H4), 4.16 (0.42H, d, *J* = 2.8 Hz, H4), 4.20 (0.42H, dd, *J* = 8.0, 2.0 Hz, H2), 4.22 (0.58H, dd, *J* = 8.4, 1.6 Hz, H2), 4.53 (0.42H, dd, *J* = 8.0, 2.4 Hz, H3), 4.55 (0.58H, dd, *J* = 8.4, 2.4 Hz, H3); Detectable signals of ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.57, 23.60, 23.62, 24.0, 25.47, 25.51, 25.58, 25.61, 26.46, 26.54, 38.1, 40.3, 40.6, 45.7, 45.9, 60.8, 60.9, 70.31, 70.34, 70.4, 73.15, 73.19, 104.7, 104.9, 107.5, 107.6, 108.7, 108.8, 218.9, 219.0; HRMS (ESI) calcd for $C_{16}H_{24}O_6Na$ $[M+Na]^+$ 335.1465, found 335.1468.

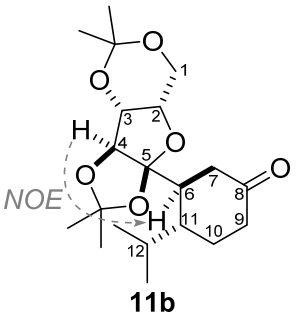


Ketone 10b. According to the general procedure C, **10b** (32 mg, 0.072 mmol) was synthesized in 65% yield from acyl telluride **1b** (50 mg, 0.11 mmol) and (*R*)-4-TBSoxycyclopentenone **5**[S8] (46 mg, 0.22 mmol) by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.4 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 5:1): colorless oil; $[\alpha]_D^{25}$ = 50.1 (*c* 1.6, $CHCl_3$); IR (film) ν 1747, 1375, 1255, 1191, 1121, 1080, 1065, 1036, 1007, 835 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 0.08 (3H, s, -SiC*H*$_3$ of TBS), 0.11 (3H, s, -SiC*H*$_3$ of TBS), 0.87 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.31 (3H, s, C*H*$_3$ of acetonide), 1.36 (3H, s, C*H*$_3$ of acetonide), 1.42 (3H, s, C*H*$_3$ of acetonide), 1.44 (3H, s, C*H*$_3$ of acetonide), 2.05 (1H, d, *J* = 18.3 Hz, H9$_A$), 2.35 (1H, d, *J* = 18.3 Hz, H7$_A$), 2.64 (1H, ddd, *J* = 18.3, 9.2, 1.2 Hz, H7$_B$), 2.71 (1H, d, *J* = 9.2 Hz, H6), 2.86 (1H, ddd, *J* = 18.3, 5.2, 1.2 Hz, H9$_B$), 3.96 (1H, d, *J* = 13.2 Hz, H1$_A$), 4.01-4.06 (2H, m, H1$_B$, 2), 4.27 (1H, d, *J* = 2.3 Hz, H3), 4.35 (1H, s, H4), 4.86 (1H, d, *J* = 5.2 Hz, H10); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.8, -4.7, 17.9, 18.6, 25.7 (3C), 26.6, 27.5, 28.8, 38.1, 47.4, 52.0, 60.2, 70.8, 72.0, 73.6, 86.3, 97.3, 111.5, 115.3, 218.8; HRMS (ESI) calcd for $C_{22}H_{38}O_7SiNa$ $[M+Na]^+$ 465.2279, found 465.2279.



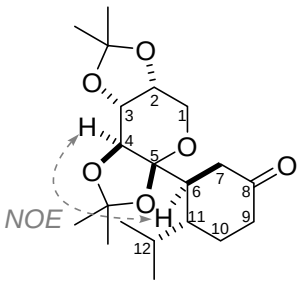
10c

Ketone 10c. According to the general procedure C, **10c** (28 mg, 0.063 mmol) was synthesized in 57% yield from acyl telluride **1c** (51 mg, 0.11 mmol) and (*R*)-4-TBSoxycyclopentenone **5** (47 mg, 0.22 mmol) by using Et_3B (1.04 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (5.6 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 7:1): colorless solid; $[\alpha]_D^{28}$ = 37.5 (*c* 1.4, $CHCl_3$); m.p. 91-93 °C; IR (film) ν 1748, 1471, 1461, 1383, 1374, 1254, 1209, 1188, 1171, 1103 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.06 (3H, s, -SiC*H*$_3$ of TBS), 0.09 (3H, s, -SiC*H*$_3$ of TBS), 0.86 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.33 (3H, s, C*H*$_3$ of acetonide), 1.35 (3H, s, C*H*$_3$ of acetonide), 1.48 (6H, s, C*H*$_3$x2 of acetonide), 2.01 (1H, d, *J* = 17.8 Hz, H9$_A$), 2.45 (1H, d, *J* = 16.5 Hz, H7$_A$), 2.52 (1H, d, *J* = 10.0 Hz, H6), 2.57 (1H, dd, *J* = 16.5, 10.0 Hz, H7$_B$), 2.70 (1H, ddd, *J* = 17.8, 5.5, 1.4 Hz, H9$_B$), 3.69 (1H, d, *J* = 13.3 Hz, H1$_A$), 3.80 (1H, dd, *J* = 13.3, 1.8 Hz, H1$_B$), 4.18 (1H, dd, *J* = 8.2, 1.8 Hz, H2), 4.23 (1H, d, *J* = 2.3 Hz, H4), 4.53 (1H, dd, *J* = 8.2, 2.3 Hz, H3), 4.92 (1H, d, *J* = 5.5 Hz, H10); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.7, -4.6, 17.8, 23.4, 25.3, 25.6, 25.7 (3C), 26.4, 37.3, 47.5, 54.4, 60.7, 70.1, 70.3, 70.4, 73.1, 104.1, 107.5, 108.5, 218.8; HRMS (ESI) calcd for $C_{22}H_{38}O_7SiNa$ $[M+Na]^+$ 465.2279, found 465.2269.



11b

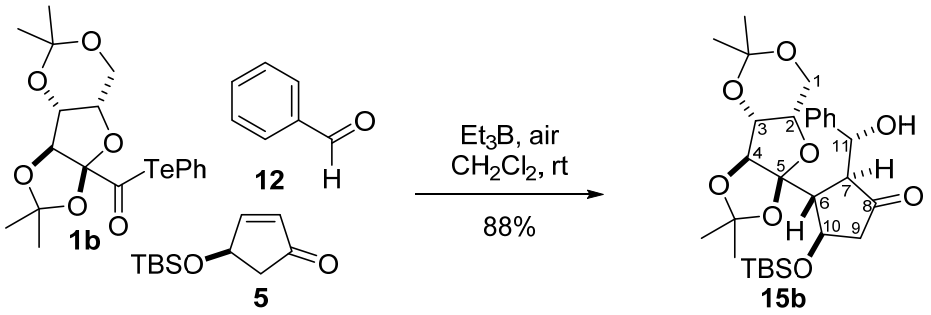
Ketone 11b. According to the general procedure C, **11b** (49 mg, 0.13 mmol) was synthesized in 59% yield from acyl telluride **1b** (100 mg, 0.22 mmol) and (*R*)-cryptone **6**[S9] (90 mg, 1.1 mmol) by using Et_3B (1.04 M in hexane, 0.63 mL, 0.65 mmol) in CH_2Cl_2 (2.2 mL). The residue was purified by flash chromatography on silica gel (10.0 g, hexane/EtOAc 10:1): colorless solid; $[\alpha]_D^{28}$ = -24.1 (*c* 1.9, $CHCl_3$); m.p. 93-95 °C; IR (film) ν 1715, 1382, 1373, 1198, 1121, 1080, 1001 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.91 (3H, d, *J* = 6.3 Hz, C12-C*H*$_3$), 1.00 (3H, d, *J* = 6.3 Hz, C12-C*H*$_3$), 1.35 (3H, s, C*H*$_3$ of acetonide), 1.40 (6H, s, C*H*$_3$x2 of acetonide), 1.46 (3H, s, C*H*$_3$ of acetonide), 1.64 (1H, dddd, *J* = 17.6, 8.7, 8.7, 6.0 Hz, H10$_A$), 1.91 (1H, dddd, *J* = 8.7, 8.7, 4.3, 4.3 Hz, H11), 1.96-2.04 (2H, m, H10$_B$, 12), 2.29-2.33 (2H, m, H9$_A$, 9$_B$), 2.35 (1H, d, *J* = 14.2 Hz, H7$_A$), 2.40 (1H, dd, *J* = 8.7, 3.3 Hz, H6), 2.75 (1H, dd, *J* = 14.2, 3.3 Hz, H7$_B$), 3.96 (1H, dd, *J* = 13.1, 1.4 Hz, H1$_A$), 4.05 (1H, dd, *J* = 13.1, 2.7 Hz, H1$_B$), 4.06 (1H, ddd, *J* = 2.7, 2.7, 1.4 Hz, H2), 4.20 (1H, d, *J* = 2.7 Hz, H3), 4.32 (1H, s, H4); ^{13}C NMR (100 MHz, $CDCl_3$) δ 17.7, 19.1, 21.3, 21.5, 27.1, 27.6, 28.2, 29.9, 37.8, 39.2, 40.1, 44.0, 60.3, 72.3, 73.7, 86.3, 97.5, 111.0, 117.7, 212.6; HRMS (ESI) calcd for $C_{20}H_{32}O_6Na$ $[M+Na]^+$ 391.2091, found 391.2098.



11c

Ketone 11c. According to the general procedure C, **11c** (45 mg, 0.12 mmol) was synthesized in 56% yield from acyl telluride **1c** (100 mg, 0.22 mmol) and (*R*)-cryptone **6** (90 mg, 1.1 mmol) by using Et_3B (1.04 M in hexane, 0.63 mL, 065 mmol) in CH_2Cl_2 (2.2 mL). The residue was purified by flash chromatography on silica gel (10.0 g, hexane/EtOAc 10:1): colorless solid; $[\alpha]_D^{28}$ = -31.9 (*c* 0.5, $CHCl_3$); m.p. 129-131 °C; IR (film) ν 1715, 1384, 1373, 1254, 1210, 1178, 1063 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.90 (3H, d, *J* = 6.3 Hz, C12-C*H*$_3$), 0.99 (3H, d, *J* = 6.3 Hz, C12-C*H*$_3$), 1.32 (3H, s, C*H*$_3$ of acetonide), 1.35 (3H, s, C*H*$_3$ of acetonide), 1.50 (3H, s, C*H*$_3$ of acetonide), 1.61 (1H, m, H10$_A$), 1.59 (3H, s, C*H*$_3$ of acetonide), 1.96-2.04 (3H, m, H10$_B$, 11, 12), 2.16 (1H, ddd, *J* = 8.8, 7.2, 4.5 Hz, H6), 2.24-2.30 (3H, m, H7$_A$, 9 $_A$, 9$_B$), 2.75 (1H, dd, *J* = 16.2, 4.5 Hz, H7$_B$), 3.73 (1H, d, *J* = 13.3 Hz, H1$_A$), 3.82 (1H, dd, *J* = 13.3, 1.8 Hz, H1$_B$), 4.11 (1H, d, *J* = 1.8 Hz, H4), 4.15 (1H, dd, *J* = 8.2, 1.8 Hz, H2), 4.47 (1H, dd, *J* = 8.2, 1.8 Hz, H3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 17.9, 21.4, 21.6, 23.3, 25.1, 25.6, 26.5, 29.4, 37.8, 38.6, 39.5, 46.7, 60.6, 69.7, 70.5, 74.3, 106.5, 107.0, 108.7, 212.4; HRMS (ESI) calcd for $C_{20}H_{32}O_6Na$ $[M+Na]^+$ 391.2091, found 391.2096.

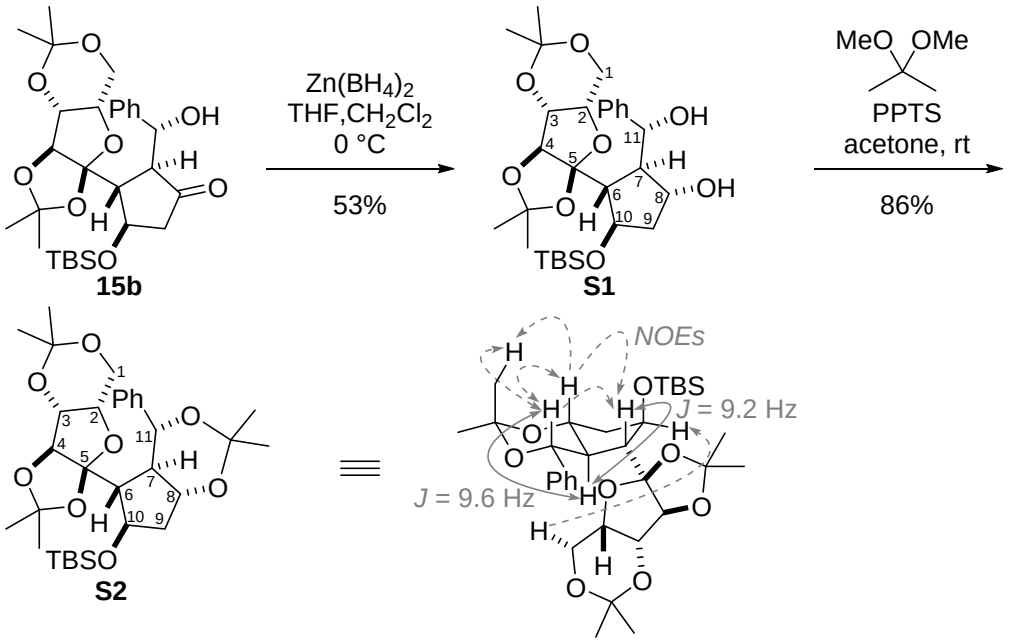
Three-Component Reactions



***General procedure D*: Alcohol 15b.** Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) was added to a solution of acyl telluride **1b** (51 mg, 0.11 mmol), (*R*)-4-TBSoxycyclopentenone **5** (46 mg, 0.22 mmol) and benzaldehyde **12** (33 μL, 0.33 mmol) in CH_2Cl_2 (1.1 mL) at room temperature. The reaction mixture was stirred for 15 min at the same temperature, and was then concentrated. The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc = 4:1) to afford **15b** (28 mg, 0.063 mmol) in 88% yield: colorless oil; $[\alpha]_D^{24}$ = -17.1 (*c* 2.7, $CHCl_3$); IR (film) ν 3484, 1724, 1457, 1377, 1250, 1192, 1119, 1065, 1011, 836 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.10 (3H, s, -SiC*H*$_3$ of TBS), 0.12 (3H, s, -SiC*H*$_3$ of

TBS), 0.52 (3H, s, C*H*$_3$ of acetonide), 0.94 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.26 (3H, s, C*H*$_3$ of acetonide), 1.30 (3H, s, C*H*$_3$ of acetonide), 1.38 (3H, s, C*H*$_3$ of acetonide), 2.23-2.28 (2H, m, H6, 9$_A$), 2.89 (1H, d, *J* = 9.6 Hz, H7), 3.24 (1H, ddd, *J* = 18.8, 5.5, 1.4 Hz, H9$_B$), 3.86 (1H, s, H2), 3.90 (1H, d, *J* = 13.7 Hz, H1$_A$), 4.00 (1H, dd, *J* = 13.7, 2.3 Hz, H1$_B$), 4.14 (1H, d, *J* = 2.3 Hz, H3), 4.18 (1H, s, H4), 4.62 (1H, br s, O*H*), 4.79 (1H, d, *J* = 5.0 Hz, H10), 4.92 (1H, d, *J* = 9.6 Hz, H11), 7.22-7.33 (3H, m, aromatic), 7.40-7.43 (2H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.7, -4.5, 17.9, 18.4, 24.7, 25.8 (3C), 26.8, 28.9, 48.9, 54.6, 55.8, 59.8, 70.3, 71.4, 73.8, 75.7, 86.1, 97.3, 110.5, 114.4, 127.9, 128.1 (2C), 128.2 (2C), 141.0, 222.6; HRMS (ESI) calcd for $C_{29}H_{44}O_8SiNa$ $[M+Na]^+$ 571.2698, found 571.2689.

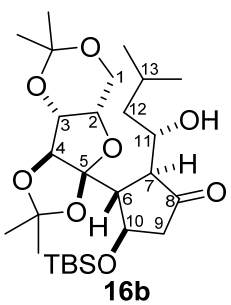
Structural Determination of 15b



***General procedure E*: Diol S1**. A suspension of $ZnCl_2$ (72 mg, 0.53 mmol) and $NaBH_4$ (40 mg, 1.1 mmol) in THF (2.1 mL) was stirred at room temperature for 2 h.[S10] A solution of compound **15b** (58 mg, 0.11 mmol) in CH_2Cl_2 (5.3 mL) was added to the suspension at 0 °C. The reaction mixture was stirred at the same temperature for 2 h, and was quenched with saturated aqueous NH_4Cl (10 mL). The resultant mixture was extracted with EtOAc (10 mL x2), and the combined organic layers were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated. The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 5:1 to 1:4) to afford diol **S1** (22 mg, 0.040 mmol) in 36% yield: colorless oil; colorless oil; $[\alpha]_D^{23}$ = 0.50 (*c* 0.60, $CHCl_3$); IR (film) ν 3479, 1455, 1377, 1254, 1191, 1120, 1063, 1003, 923, 834 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.09 (3H, s, -SiC*H*$_3$ of

TBS), 0.12 (3H, s, -SiC*H*$_3$ of TBS), 0.91 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.16 (3H, s, C*H*$_3$ of acetonide), 1.36 (3H, s, C*H*$_3$ of acetonide), 1.42 (3H, s, C*H*$_3$ of acetonide), 1.43 (3H, s, C*H*$_3$ of acetonide), 1.65 (1H, br s, O*H*), 2.01 (1H, dd, *J* = 13.3, 6.9 Hz, H9$_A$), 2.17 (1H, ddd, *J* = 13.3, 8.2, 5.0 Hz, H9$_B$), 2.35 (1H, d, *J* = 3.2 Hz, H6), 2.65 (1H, dd, *J* = 8.7, 4.6 Hz, H7), 3.51 (1H, br s, O*H*), 3.97-4.08 (3H, m, H1$_A$, 1$_B$, 2), 4.22 (1H, d, *J* = 2.3 Hz, H3), 4.30 (1H, s, H4), 4.54-4.64 (2H, m, H8, 10), 4.90 (1H, d, *J* = 5.0 Hz, H11), 7.24 (1H, m, aromatic), 7.32-7.36 (2H, m, aromatic), 7.48 (2H, d, *J* = 7.3 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.8, -4.6, 17.8, 18.6, 25.7 (3C), 26.3, 27.3, 28.8, 44.1, 56.8, 58.9, 60.0, 71.7, 73.3, 73.8, 74.0, 75.7, 86.3, 97.3, 110.9, 115.3, 126.5 (2C), 127.2, 128.3 (2C), 143.0; HRMS (ESI) calcd for $C_{29}H_{46}O_8SiNa$ $[M+Na]^+$ 573.2854, found 573.2843.

***General procedure F*: Acetonide S2.** Pyridinium *p*-toluenesulfonate (PPTS, 2.8 mg, 0.011 mmol) was added to a solution of **S1** (12 mg, 0.022 mmol) and 2,2'-dimethoxypropane (0.44 mL, 3.6 mmol) in acetone (0.44 mL) at room temperature. The reaction mixture was stirred for 24h at the same temperature, and was then concentrated. The residue was purified by flash chromatography on silica gel [32–53 μm Silica-gel BW-300 (Fuji Silysia Chemical Ltd.)] (10 g, hexane/EtOAc 10:1) to afford acetonide **S2** (11 mg, 0.019 mmol) in 86% yield: colorless solid; $[\alpha]_D^{25}$ = -21.1 (*c* 1.1, CH_2Cl_2); m.p. 170-175 °C; IR (film) ν 1461, 1380, 1244, 1186, 1123, 1067, 1010, 834 cm^{-1}; ^{1}H NMR (400 MHz, C_6D_6, at 70 °C) δ 0.15 (3H, s, -SiC*H*$_3$ of TBS), 0.23 (3H, s, -SiC*H*$_3$ of TBS), 1.01 (3H, s, C*H*$_3$ of acetonide), 1.03 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.12 (6H, s, C*H*$_3$x2 of acetonide), 1.36 (3H, s, C*H*$_3$ of acetonide), 1.56 (3H, s, C*H*$_3$ of acetonide), 1.64 (3H, s, C*H*$_3$ of acetonide), 2.12-2.16 (2H, m, H9$_A$, 9$_B$), 2.35 (1H, d, *J* = 9.2 Hz, H6), 2.45 (1H, br s, H7), 3.49 (1H, dd, *J* = 13.6, 3.2 Hz, H1$_A$), 3.55 (1H, s, H2), 3.73 (1H, br s, H4), 3.78 (1H, d, *J* = 13.2 Hz, H1$_B$), 3.82 (1H, s, H3), 4.58 (1H, ddd, *J* = 10.0, 8.4, 7.2 Hz, H8), 4.83 (1H, s, H10), 4.94 (1H, d, *J* = 9.6 Hz, H11), 7.11 (1H, m, aromatic), 7.16-7.20 (2H, m, aromatic), 7.49 (2H, d, *J* = 7.2 Hz, aromatic); ^{13}C NMR (100 MHz, $CO(CD_3)_2$, at 50 °C) δ -3.6, -3.5, 18.8, 19.7, 21.1, 26.7 (3C), 27.2, 27.8, 29.9, 31.1, 41.3, 49.9, 57.0, 61.2, 72.4, 72.7, 75.6, 75.7, 82.6, 86.4, 98.4, 101.1, 111.2, 116.6, 128.8 (2C), 128.9 (2C), 130.0, 143.0; HRMS (ESI) calcd for $C_{32}H_{50}O_8SiNa$ $[M+Na]^+$ 613.3167, found 613.3176.

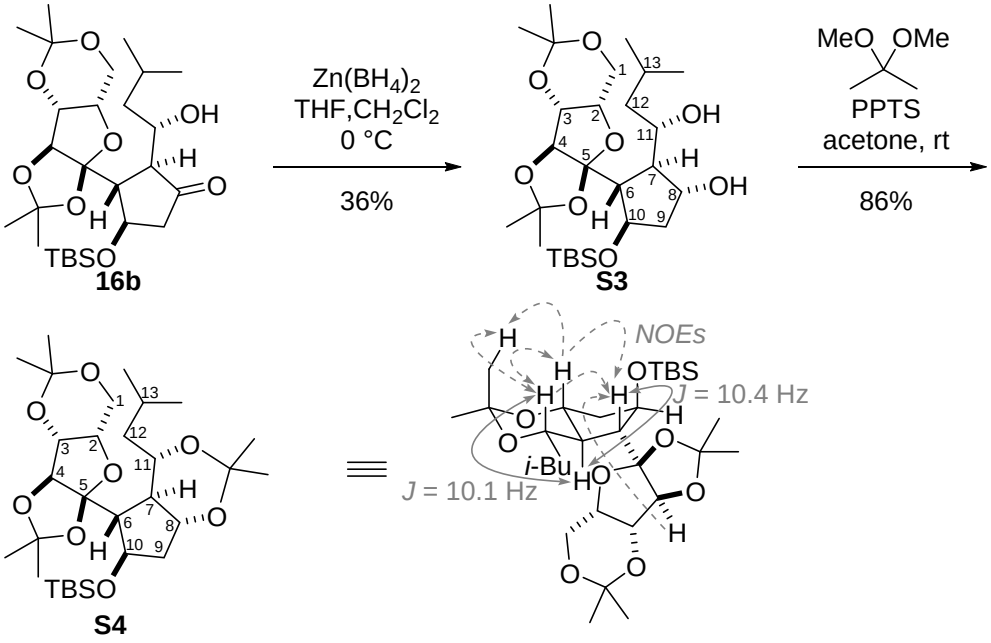


16b

Alcohol 16b. According to the general procedure D, **16b** (49 mg, 0.093 mmol) was synthesized in 85% yield from acyl telluride **1b** (50 mg, 0.11 mmol), (*R*)-4-TBSoxycyclopentenone **5** (46 mg, 0.22 mmol) and 3-methylbutanal **13** (35 μL, 0.33 mmol) by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (1.1 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 4:1): colorless oil; $[\alpha]_D^{24}$ = 9.7 (*c* 2.4, $CHCl_3$); IR (film) ν 3501, 1726, 1469, 1377, 1252,

1192, 1121, 1067, 1010, 836 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.09 (3H, s, -SiC*H*$_3$ of TBS), 0.13 (3H, s, -SiC*H*$_3$ of TBS), 0.87 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 0.90 (3H, d, *J* = 6.4 Hz, C13-C*H*$_3$), 0.91 (3H, d, *J* = 6.4 Hz, C13-C*H*$_3$), 1.26-1.56 (2H, m, H12$_A$, 12$_B$), 1.32 (3H, s, C*H*$_3$ of acetonide), 1.35 (3H, s, C*H*$_3$ of acetonide), 1.42 (3H, s, C*H*$_3$ of acetonide), 1.44 (3H, s, C*H*$_3$ of acetonide), 1.96 (1H, m, H13), 2.16 (1H, d, *J* = 18.3 Hz, H9$_A$), 2.44 (1H, d, *J* = 5.5 Hz, H7), 2.56 (1H, s, H6), 2.97 (1H, dd, *J* = 18.3, 5.5 Hz, H9$_B$), 3.92-4.07 (5H, m, H1$_A$, 1$_B$, 2, 11, O*H*), 4.29 (1H, d, *J* = 2.3 Hz, H3), 4.39 (1H, s, H4), 4.85 (1H, d, *J* = 5.5 Hz, H10); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.80, -4.77, 17.8, 18.5, 21.7, 23.6, 24.3, 25.7 (3C), 26.7, 27.5, 28.9, 43.9, 49.0, 53.9, 57.0, 60.1, 70.1, 71.2, 72.0, 73.6, 86.1, 97.3, 111.4, 115.3, 220.5; HRMS (ESI) calcd for $C_{27}H_{48}O_8SiNa$ $[M+Na]^+$ 551.3011, found 551.3020.

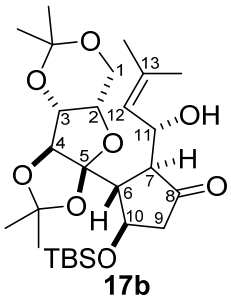
Structural Determination of 16b



Diol S3. According to the general procedure E, **S3** (25 mg, 0.047 mmol) was synthesized in 59% yield from **16b** (42 mg, 0.079 mmol) by using $ZnCl_2$ (54 mg, 0.39 mmol) and $NaBH_4$ (30 mg, 0.79 mmol) in THF (0.79 mL) and CH_2Cl_2 (1.6 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 1:1): colorless oil; $[\alpha]_D^{22}$ = -13.5 (*c* 1.3, $CHCl_3$); IR (film) ν 3442, 1466, 1378, 1251, 1192, 1122, 1064, 1003, 835 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.08 (3H, s, -SiC*H*$_3$ of TBS), 0.10 (3H, s, -SiC*H*$_3$ of TBS), 0.88 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 0.93 (3H, d, *J* = 6.8 Hz, C13-C*H*$_3$), 0.94 (3H, d, *J* = 6.9 Hz, C13-C*H*$_3$), 1.36 (6H, s, C*H*$_3$x2 of acetonide), 1.40-1.57 (2H, m, H12$_A$, 12$_B$), 1.43 (3H, s, C*H*$_3$ of acetonide), 1.47 (3H, s, C*H*$_3$ of acetonide), 1.85 (1H, m, H13), 2.04-2.17 (2H, m, H9$_A$, 9$_B$), 2.25 (1H, d, *J* = 2.8 Hz, H6), 2.30 (1H, dd, *J* = 7.3, 3.2 Hz, H7), 3.81 (1H, ddd, *J* = 8.2, 5.0, 3.2 Hz, H11), 3.97-4.10 (3H, m, H1$_A$, 1$_B$, 2), 4.25 (1H, d, *J* = 1.8 Hz, H3), 4.38 (1H, s, H4),

4.52 (1H, ddd, J = 7.8, 7.8, 4.6 Hz, H8), 4.60 (1H, d, J = 4.1 Hz, H10); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.8, -4.6, 17.8, 18.6, 22.4, 23.2, 24.8, 25.7 (3C), 26.7, 27.4, 28.9, 44.0, 45.6, 54.9, 60.0, 60.1, 71.7, 72.0, 73.4, 73.9, 74.0, 86.4, 97.4, 110.9, 115.5; HRMS (ESI) calcd for $C_{27}H_{50}O_8SiNa$ $[M+Na]^+$ 553.3167, found 553.3155.

Acetonide S4. According to the general procedure F, **S4** (21 mg, 0.037 mmol) was synthesized in 77% yield from **S3** (25 mg, 0.048 mmol) by using pyridinium *p*-toluenesulfonate (PPTS, 2.4 mg, 9.5 μmol) and 2,2'-dimethoxypropane (0.95 mL, 7.8 mmol) in acetone (0.95 mL). The residue was purified by flash chromatography on silica gel [32–53 μm Silica-gel BW-300 (Fuji Silysia Chemical Ltd.)] (10 g, hexane/EtOAc 10:1): colorless oil; $[\alpha]_D^{19}$ = -38.9 (*c* 1.1, MeOH); IR (film) 1466, 1378, 1255, 1190, 1123, 1065, 1005, 905, 834 cm^{-1}; ^{1}H NMR (400 MHz, C_6D_6) δ 0.11 (3H, s, -SiC*H*$_3$ of TBS), 0.21 (3H, s, -SiC*H*$_3$ of TBS), 0.97 (3H, s, C*H*$_3$ of acetonide), 1.00 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.05 (3H, d, J = 4.0 Hz, C13-C*H*$_3$), 1.07 (3H, d, J = 3.6 Hz, C13-C*H*$_3$), 1.33 (3H, s, C*H*$_3$ of acetonide), 1.39 (3H, s, C*H*$_3$ of acetonide), 1.44 (3H, s, C*H*$_3$ of acetonide), 1.46 (3H, s, C*H*$_3$ of acetonide), 1.63 (3H, s, C*H*$_3$ of acetonide), 1.87 (1H, ddd, J = 13.7, 10.0, 3.6 Hz, H12$_A$), 2.07-2.12 (2H, m, H7, 9$_A$), 2.17 (1H, m, H13), 2.20-2.36 (2H, m, H9$_B$, 12$_B$), 2.34 (1H, dd, J = 10.4, 3.2 Hz, H6), 3.42 (1H, dd, J = 13.7, 2.8 Hz, H1$_A$), 3.60 (1H, s, H2), 3.79 (1H, d, J = 13.7 Hz, H1$_B$), 4.00 (1H, d, J = 2.3 Hz, H3), 4.11 (1H, ddd, J = 10.1, 10.1, 1.8 Hz, H11), 4.34 (1H, ddd, J = 10.4, 10.4, 8.0 Hz, H8), 4.83 (1H, ddd, J = 5.6, 3.2, 2.4 Hz, H10), 4.86 (1H, s, H4); ^{13}C NMR (100 MHz, C_6D_6) δ -4.2, -3.6, 18.0, 18.6, 20.2, 22.0, 24.5, 24.7, 26.0 (3C), 26.6, 27.5, 29.3, 30.5, 40.5, 44.0, 47.8, 57.7, 59.7, 71.0, 71.8, 74.1, 74.9, 75.8, 86.3, 97.2, 99.3, 110.0, 115.6; HRMS (ESI) calcd for $C_{30}H_{54}O_8SiNa$ $[M+Na]^+$ 593.3480, found 593.3468.

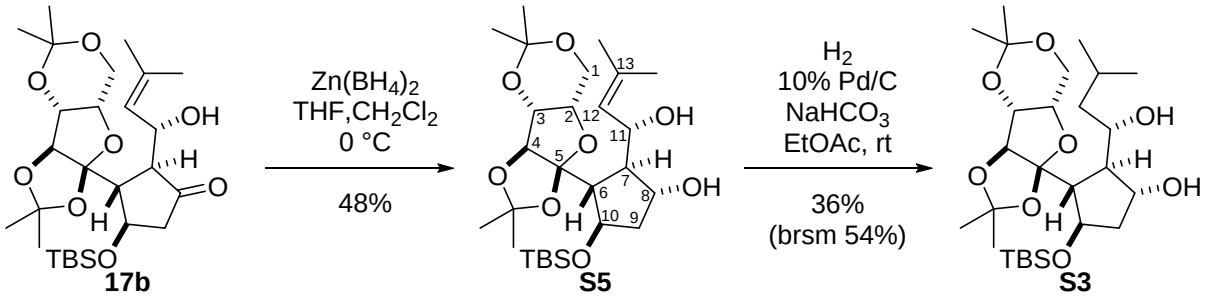


17b

Alcohol 17b. According to the general procedure D, **17b** (46 mg, 0.087 mmol) was synthesized in 79% yield from acyl telluride **1b** (50 mg, 0.11 mmol), (*R*)-4-TBSoxycyclopentenone **5** (18 μL, 0.22 mmol) and 3-methyl-2-butenal **14** (31 μL, 0.33 mmol) by using Et_3B (1.03 M in hexane, 0.32 mL, 0.33 mmol) in CH_2Cl_2 (1.1 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 4:1): colorless oil; $[\alpha]_D^{24}$ = 0.73 (*c* 2.3, $CHCl_3$); IR (film) ν 3487, 1725, 1453, 1377, 1249, 1193, 1120, 1065, 1007, 835 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.07 (3H, s, -SiC*H*$_3$ of TBS), 0.08 (3H, s, -SiC*H*$_3$ of TBS), 0.87 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.29 (3H, s, C*H*$_3$ of acetonide), 1.33 (3H, s, C*H*$_3$ of acetonide), 1.42 (3H, s, C*H*$_3$ of acetonide), 1.45 (3H, s, C*H*$_3$ of acetonide), 1.71 (3H, s, C13-C*H*$_3$), 1.72 (3H, s, C13-C*H*$_3$), 2.19 (1H, d, J = 18.3 Hz, H9$_A$), 2.43 (1H, s, H6), 2.57 (1H, d, J = 8.2 Hz, H7), 3.09 (1H, dd, J = 17.8, 5.5 Hz, H9$_B$), 3.93 (1H, d, J = 13.7 Hz, H1$_A$), 3.96 (1H, s, H2), 4.04 (1H, dd, J = 13.8, 2.3 Hz, H1$_B$), 4.14

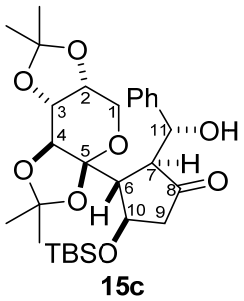
(1H, br s, O*H*), 4.25 (1H, d, *J* = 2.3 Hz, H3), 4.40 (1H, s, H4), 4.63 (1H, dd, *J* = 8.7, 8.7 Hz, H11), 4.81 (1H, d, *J* = 5.5 Hz, H10), 5.25 (1H, d, *J* = 8.7 Hz, H12); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.7, -4.6, 17.9, 18.5, 18.6, 25.8 (3C), 25.9, 26.5, 27.4, 28.9, 49.1, 53.5, 56.4, 60.0, 69.6, 70.3, 71.6, 73.9, 86.4, 97.3, 111.1, 115.0, 125.5, 136.6, 222.2; HRMS (ESI) calcd for $C_{27}H_{46}O_8SiNa$ $[M+Na]^+$ 549.2854, found 549.2835.

Structural Determination of 17b



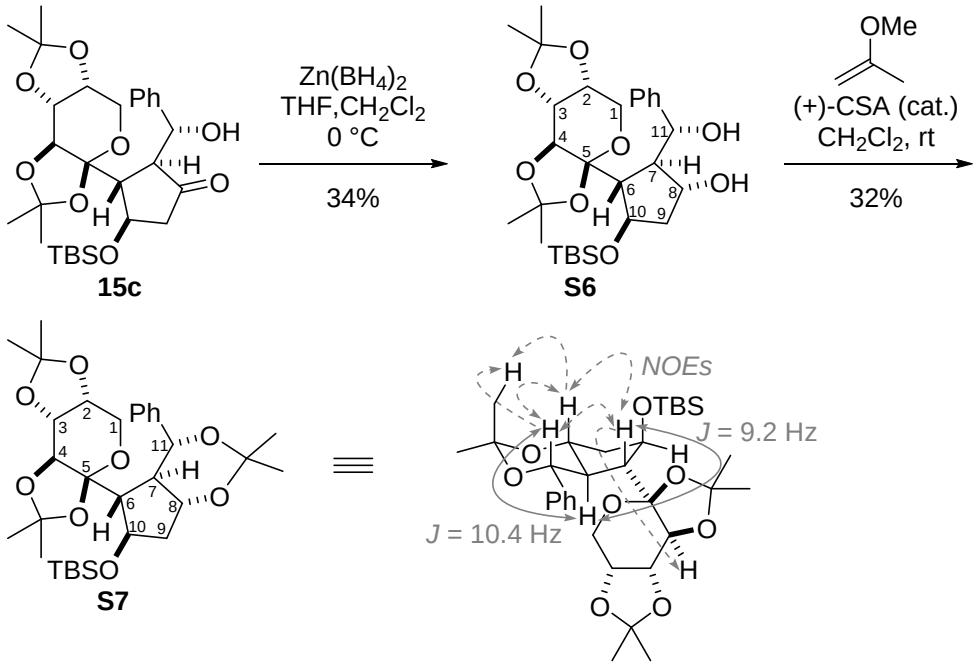
Diol S5. According to the general procedure E, **S5** (46 mg, 0.087 mmol) was synthesized in 48% yield from **17b** (94 mg, 0.18 mmol) by using $ZnCl_2$ (0.12 g, 0.90 mmol) and $NaBH_4$ (68 mg, 1.8 mmol) in THF (3.6 mL) and CH_2Cl_2 (9.0 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 1:2): colorless solid; $[\alpha]_D^{24}$ = -13.7 (*c* 0.74, $CHCl_3$); m.p. 98-103 °C; IR (film) 3473, 1455, 1378, 1252, 1193, 1121, 1066, 1023, 1001, 836 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.06 (3H, s, -SiC*H*$_3$ of TBS), 0.08 (3H, s, -SiC*H*$_3$ of TBS), 0.87 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.35 (3H, s, C*H*$_3$ of acetonide), 1.36 (3H, s, C*H*$_3$ of acetonide), 1.42 (3H, s, C*H*$_3$ of acetonide), 1.47 (3H, s, C*H*$_3$ of acetonide), 1.71 (3H, d, *J* = 1.4 Hz, C13-C*H*$_3$), 1.73 (3H, d, *J* = 0.9 Hz, C13-C*H*$_3$), 2.00 (1H, dd, *J* = 13.3, 6.9 Hz, H9$_A$), 2.10 (1H, ddd, *J* = 12.8, 7.8, 5.0 Hz, H9$_B$), 2.23 (1H, d, *J* = 3.7 Hz, H6), 2.33 (1H, dd, *J* = 9.6, 5.0 Hz, H7), 2.87 (1H, br s, O*H*), 3.97-4.09 (3H, m, H1$_A$, 1$_B$, 2), 4.24 (1H, d, *J* = 2.3 Hz, H3), 4.43 (1H, s, H4), 4.43-4.51 (2H, m, H8, 11), 4.61 (1H, d, *J* = 4.1 Hz, H10), 5.38 (1H, ddd, *J* = 8.7, 1.4, 1.4 Hz, H12); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.7, -4.5, 17.7, 18.3, 18.7, 25.7 (3C), 26.0, 26.6, 27.4, 28.8, 44.6, 55.4, 58.5, 59.9, 70.5, 71.6, 73.6, 74.1, 74.7, 86.2, 97.4, 110.8, 115.5, 126.3, 135.4; HRMS (ESI) calcd for $C_{27}H_{48}O_8SiNa$ $[M+Na]^+$ 551.3011, found 551.3008.

A suspension of **S5** (13 mg, 0.025 mmol), $NaHCO_3$ (11 mg, 0.13 mmol) and 10% Pd/C (2.7 mg) in EtOAc (1.3 mL) was exposed to H_2 atmosphere. The reaction mixture was stirred at room temperature for 40 h, and was then filtered through a pad of Celite. The filtrate was concentrated and the residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 1:1 to 1:2) to afford alcohol **S3** (4.7 mg, 8.9 μmol) in 36% yield (33% of **S5** was recovered). The physical data of **S3** were matched with **S3**, which was derived from **16b**.



Alcohol 15c. According to the general procedure D, **15c** (38 mg, 0.069 mmol) was synthesized in 73% yield from acyl telluride **1c** (44 mg, 0.095 mmol), (*R*)-4-TBSoxycyclopentenone **5** (40 mg, 0.19 mmol) and benzaldehyde **12** (29 μL, 0.29 mmol) by using Et_3B (1.04 M in hexane, 0.27 mL, 0.28 mmol) in CH_2Cl_2 (0.95 mL). The residue was purified by flash chromatography on silica gel (5.0 g, hexane/EtOAc 4:1): colorless oil; $[\alpha]_D^{24}$ = -25.0 (*c* 1.1, $CHCl_3$); IR (film) 3484, 1724, 1458, 1381, 1254, 1210, 1187, 1062, 898, 838 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.10 (3H, s, -SiC*H*$_3$ of TBS), 0.13 (3H, s, -SiC*H*$_3$ of TBS), 0.41 (3H, s, C*H*$_3$ of acetonide), 0.94 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.27 (3H, s, C*H*$_3$ of acetonide), 1.32 (3H, s, C*H*$_3$ of acetonide), 1.44 (3H, s, C*H*$_3$ of acetonide), 2.09 (1H, d, *J* = 1.8 Hz, H6), 2.23 (1H, d, *J* = 17.9 Hz, H9$_A$), 2.96 (1H, d, *J* = 10.1 Hz, H7), 2.98 (1H, dd, *J* = 17.9, 5.5 Hz, H9$_B$), 3.62 (1H, d, *J* = 13.3 Hz, H1$_A$), 3.68 (1H, dd, *J* = 13.3, 1.8 Hz, H1$_B$), 4.07 (1H, d, *J* = 2.3 Hz, H4), 4.11 (1H, dd, *J* = 8.2, 2.3 Hz, H2), 4.43 (1H, dd, *J* =8.2, 1.8 Hz, H3), 4.58 (1H, s, O*H*), 4.91 (1H, d, *J* = 5.5 Hz, H10), 4.92 (1H, d, *J* = 10.1 Hz, H11), 7.25 (1H, m, aromatic), 7.30-7.34 (2H, m, aromatic), 7.42-7.44 (2H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.6, -4.4, 17.9, 23.0, 23.1, 25.6, 25.8, 25.9 (3C), 29.7, 49.1, 53.9, 58.2, 60.1, 69.8, 70.3, 73.2, 75.7, 104.1, 107.0, 108.2, 127.9, 128.1 (2C), 128.3 (2C), 141.2, 222.5; HRMS (ESI) calcd for $C_{29}H_{44}O_8SiNa$ $[M+Na]^+$ 571.2698, found 571.2695.

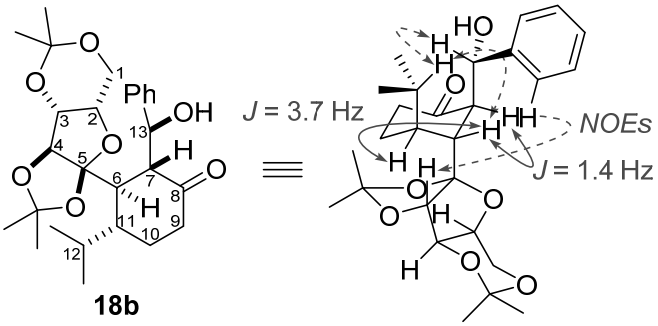
Structural Determination of 15c



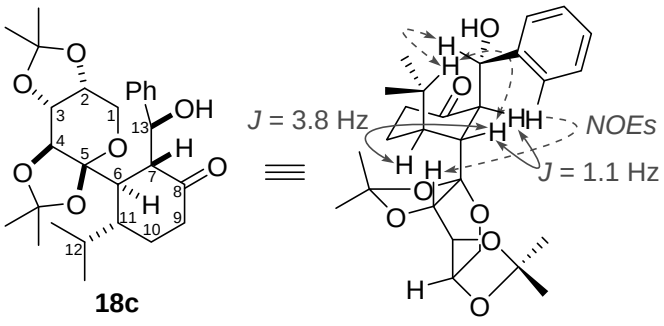
Diol S6. According to the general procedure E, **S6** (11 mg, 0.020 mmol) was synthesized in

34% yield from **15c** (32 mg, 0.058 mmol) by using $ZnCl_2$ (40 mg, 0.29 mmol) and $NaBH_4$ (22 mg, 0.58 mmol) in THF (0.59 mL) and CH_2Cl_2 (1.2 mL). The residue was purified by flash chromatography on silica gel (1.0 g, hexane/EtOAc 2:1): colorless oil; $[\alpha]_D^{20}$ = -5.9 (*c* 0.53, $CHCl_3$); IR (film) 3474, 1730, 1460, 1378, 1254, 1208, 1060, 1008, 897, 837 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.10 (3H, s, -SiC*H*$_3$ of TBS), 0.12 (3H, s, -SiC*H*$_3$ of TBS), 0.91 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.19 (3H, s, C*H*$_3$ of acetonide), 1.34 (3H, s, C*H*$_3$ of acetonide), 1.47 (6H, s, C*H*$_3$x2 of acetonide), 1.93-2.05 (2H, m, H9$_A$, 9$_B$), 2.18 (1H, d, *J* = 3.2 Hz, H6), 2.75 (1H, dd, *J* = 8.0, 4.4 Hz, H7), 3.74 (1H, d, *J* = 13.2 Hz, H1$_A$), 3.83 (1H, dd, *J* = 13.2, 2.4 Hz, H1$_B$), 4.18 (1H, dd, *J* = 8.0, 1.6 Hz, H2), 4.24 (1H, d, *J* = 1.6 Hz, H4), 4.52 (1H, dd, *J* = 8.4, 2.4 Hz, H3), 4.56 (1H, ddd, *J* = 7.6, 7.6, 5.2 Hz, H8), 4.71 (1H, d, *J* = 2.8 Hz, H10), 4.92 (1H, d, *J* = 4.4 Hz, H11), 7.24 (1H, m, aromatic), 7.31-7.37 (2H, m, aromatic), 7.48-7.51 (2H, m, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ -4.7, -4.5, 17.8, 23.5, 24.9, 25.7, 25.8 (3C), 26.2, 44.3, 56.1, 60.8, 61.7, 70.0, 70.6, 72.8, 73.4, 73.5, 75.5, 104.7, 107.3, 108.6, 126.3 (2C), 127.2, 128.3 (2C), 143.1; HRMS (ESI) calcd for $C_{29}H_{46}O_8SiNa$ $[M+Na]^+$ 573.2854, found 573.2833.

Acetonide S7. According to the general procedure F, **S7** (3.7 mg, 6.3 μmol) was synthesized in 32% yield from **S6** (11 mg, 0.020 mmol) by using (+)-10-camphorsulfonic acid ((+)-CSA, 0.5 mg, 0.002 mmol) and 2-methoxypropene (19 μL, 0.20 mmol) in CH_2Cl_2 (2.0 mL) at room temperature. The residue was purified by flash chromatography on silica gel (1.0 g, hexane/EtOAc 20:1): colorless oil; $[\alpha]_D^{23}$ = -37.3 (*c* 0.2, MeOH); IR (film) ν 1733, 1459, 1380, 1254, 1210, 1105, 1065, 1020, 1002, 832 cm^{-1}; ^1H NMR (400 MHz, C_6D_6, at 70 °C) δ 0.15 (3H, s, -SiC*H*$_3$ of TBS), 0.21 (3H, s, -SiC*H*$_3$ of TBS), 0.99 (3H, s, C*H*$_3$ of acetonide), 1.02 (9H, s, -C(C*H*$_3$)$_3$ of TBS), 1.04 (3H, s, C*H*$_3$ of acetonide), 1.13 (3H, s, C*H*$_3$ of acetonide), 1.45 (3H, s, C*H*$_3$ of acetonide), 1.55 (3H, s, C*H*$_3$ of acetonide), 1.62 (3H, s, C*H*$_3$ of acetonide), 2.00 (1H, ddd, *J* = 12.0, 12.0, 6.0 Hz, H9$_A$), 2.09 (1H, dd, *J* = 12.0, 6.0 Hz, H9$_B$), 2.18 (1H, d, *J* = 9.2 Hz, H6), 2.61 (1H, br d, *J* = 9.6 Hz, H7), 3.46 (1H, d, *J* = 13.2 Hz, H1$_A$), 3.52 (1H, d, *J* = 13.2 Hz, H1$_B$), 3.65 (1H, dd, *J* = 8.4, 2.4 Hz, H2), 4.18 (1H, br s, H4), 4.27 (1H, d, *J* = 6.8 Hz, H3), 4.50 (1H, ddd, *J* = 12.0, 12.0, 6.0 Hz, H8), 4.89 (1H, d, *J* = 10.4 Hz, H11), 4.92 (1H, dd, *J* = 5.6, 2.0 Hz, H10), 7.07-7.16 (3H, m, aromatic), 7.42 (2H, d, *J* = 6.8 Hz, aromatic); ^{13}C NMR (100 MHz, $CO(CD_3)_2$, at 50 °C) δ -3.5, -3.4, 18.7, 21.0, 24.7, 25.4, 26.4, 26.7 (3C), 27.0, 31.1, 41.3, 48.0, 59.8, 62.0, 71.7, 71.8, 72.4, 73.9, 75.0, 82.7, 100.9, 105.4, 107.9, 109.6, 128.5 (2C), 128.6 (2C), 130.4, 143.0; HRMS (ESI) calcd for $C_{32}H_{50}O_8SiNa$ $[M+Na]^+$ 613.3167, found 613.3150.



Ketone 18b. According to the general procedure D, **18b** (67 mg, 0.14 mmol) was synthesized in 63% yield from acyl telluride **1b** (100 mg, 0.22 mmol), (*R*)-cryptone **6** (90 mg, 0.65 mmol) and benzaldehyde **12** (110 μL, 1.1 mmol) by using Et_3B (1.03 M in hexane, 0.63 mL, 0.65 mmol) in CH_2Cl_2 (2.2 mL). The residue was purified by flash chromatography on silica gel (10.0 g, hexane/EtOAc 10:1): colorless oil; $[\alpha]_D^{28}$ = -7.6 (*c* 1.4, $CHCl_3$); IR (film) ν 3460, 1714, 1384, 1374, 1198, 1121, 1079, 756 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.93 (3H, d, *J* = 6.8 Hz, C12-C*H*$_3$), 0.97 (3H, d, *J* = 6.8 Hz, C12-C*H*$_3$), 0.98 (3H, s, C*H*$_3$ of acetonide), 1.38 (3H, s, C*H*$_3$ of acetonide), 1.40 (3H, s, C*H*$_3$ of acetonide), 1.48 (3H, s, C*H*$_3$ of acetonide), 1.70 (1H, m, H10$_A$), 1.77 (1H, qq, *J* = 6.8, 6.8 Hz, H12), 1.91 (1H, m, H11), 2.13 (1H, m, H10$_B$), 2.15 (1H, dd, *J* = 3.7, 1.4 Hz, H6), 2.44-2.48 (2H, m, H9$_A$, 9$_B$), 3.29 (1H, dd, *J* = 9.1, 1.4 Hz, H7), 3.87 (1H, d, *J* = 13.2 Hz, H1$_A$), 3.91 (1H, s, H4), 3.95 (1H, dd, *J* = 2.7, 2.1 Hz, H2), 3.98 (1H, dd, *J* = 13.2, 2.7 Hz, H1$_B$), 3.99 (1H, d, *J* = 4.1 Hz, O*H*), 4.13 (1H, d, *J* = 2.1 Hz, H3), 4.86 (1H, dd, *J* = 9.1, 4.1 Hz, H13), 7.28 (1H, tt, *J* = 7.3, 1.7 Hz, aromatic), 7.36 (2H, t, *J* = 7.3 Hz, aromatic), 7.44 (2H, dd, *J* = 7.3, 1.7 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.6, 19.0, 20.2, 22.0, 26.3, 27.3, 28.1, 30.8, 37.8, 40.5, 44.1, 53.4, 60.0, 71.9, 73.8, 75.2, 85.5, 97.7, 110.4, 118.2, 127.9 (2C), 128.0, 128.4 (2C), 141.8, 215.8; HRMS (ESI) calcd for $C_{27}H_{38}NaO_7$ $[M+Na]^+$ 497.2510, found 497.2518.



Ketone 18c. According to the general procedure D, **18c** (47 mg, 0.099 mmol) was synthesized in 46% yield from acyl telluride **1c** (100 mg, 0.22 mmol), (*R*)-cryptone **6** (90 mg, 0.65 mmol) and benzaldehyde **12** (110 μL, 1.1 mmol) by using Et_3B (1.03 M in hexane, 0.63 mL, 0.65 mmol) in CH_2Cl_2 (2.2 mL). The residue was purified by flash chromatography on silica gel (10.0 g, hexane/EtOAc 10:1): colorless oil; $[\alpha]_D^{26}$ = -25.6 (*c* 0.7, $CHCl_3$); IR (film) ν 3471, 1716, 1385, 1255, 1210, 1062, 757 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.91 (3H, d, *J* = 6.8 Hz, C12-C*H*$_3$), 0.95 (3H, s, C*H*$_3$ of acetonide), 0.95 (3H, d, *J* = 6.8 Hz, C12-C*H*$_3$), 1.35 (3H, s, C*H*$_3$ of acetonide), 1.39 (3H, s, C*H*$_3$ of acetonide), 1.64 (1H, m, H10$_A$), 1.66 (3H, s, C*H*$_3$ of acetonide), 1.81 (1H, qq, *J* = 6.8, 6.8 Hz, H12), 1.86 (1H, dd, *J* = 3.8, 1.1 Hz, H6), 1.99-2.08 (2H, m, H10$_B$, 11), 2.40 (1H, d, *J* = 8.7 Hz, H9$_A$), 2.41 (1H, dd, *J* = 8.7, 3.8 Hz, H9$_B$), 3.33

(1H, dd, *J* = 9.3, 1.1 Hz, H7), 3.62 (1H, d, *J* = 2.0 Hz, H4), 3.67 (1H, d, *J* = 13.3 Hz, $H1_A$), 3.74 (1H, d, *J* = 13.3 Hz, 1.8 $H1_B$), 4.09 (1H, dd, *J* = 8.2, 1.8 Hz, H2), 4.36 (1H, dd, *J* = 8.2, 2.0 Hz, H3), 4.78 (1H, d, *J* = 9.3 Hz, H13), 7.30 (1H, tt, *J* = 7.2, 1.7 Hz, aromatic), 7.37 (2H, t, *J* = 7.2 Hz, aromatic), 7.47 (2H, dd, *J* = 7.2, 1.7 Hz, aromatic); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.4, 20.0, 22.2, 23.2, 25.0, 25.1, 26.3, 30.8, 37.9, 39.4, 46.2, 53.1, 60.5, 69.6, 70.5, 72.7, 75.5, 106.5, 107.3, 108.9, 128.2, 128.29 (2C), 128.34 (2C), 141.4, 215.9; HRMS (ESI) calcd for $C_{27}H_{38}NaO_7$ $[M+Na]^+$ 497.2510, found 497.2521.

References

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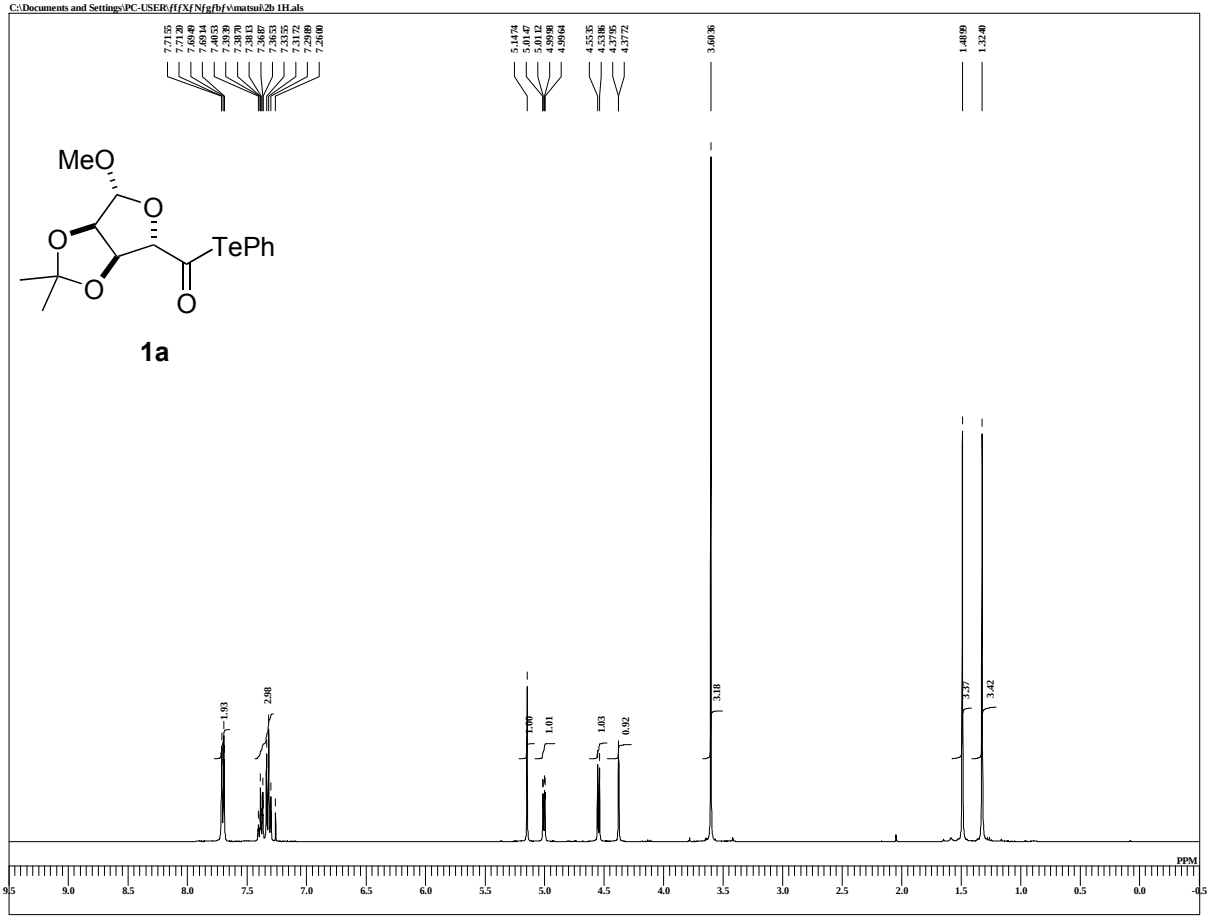


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FILDF
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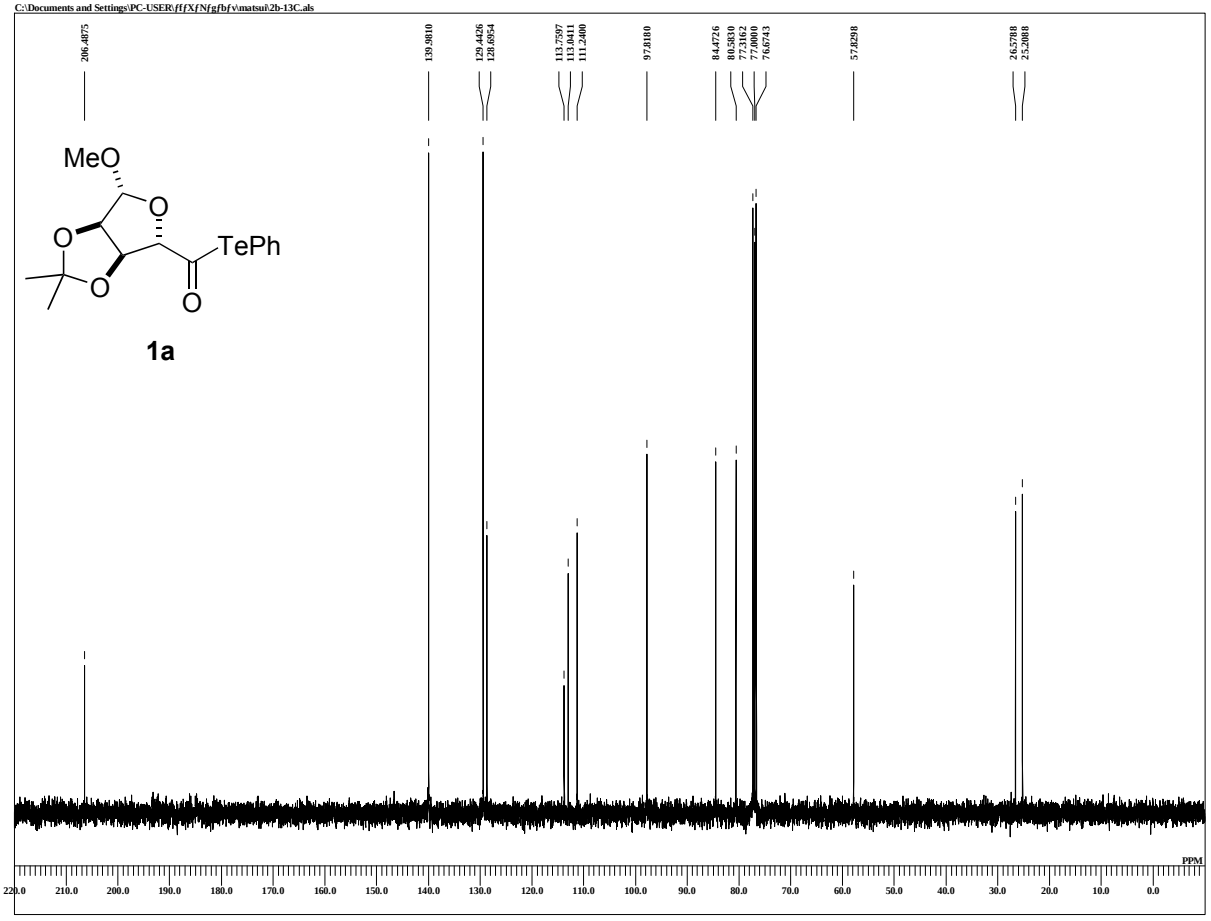


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FILDF
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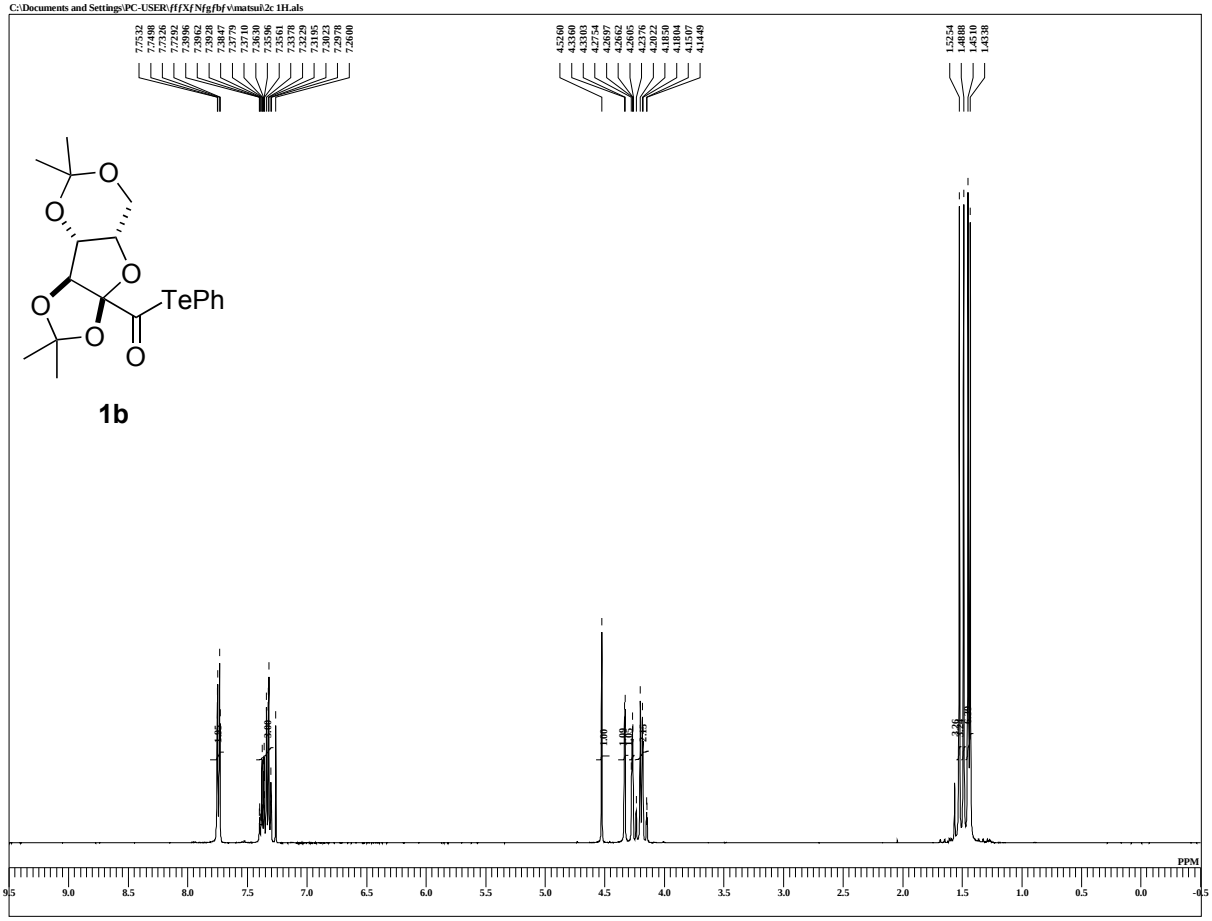

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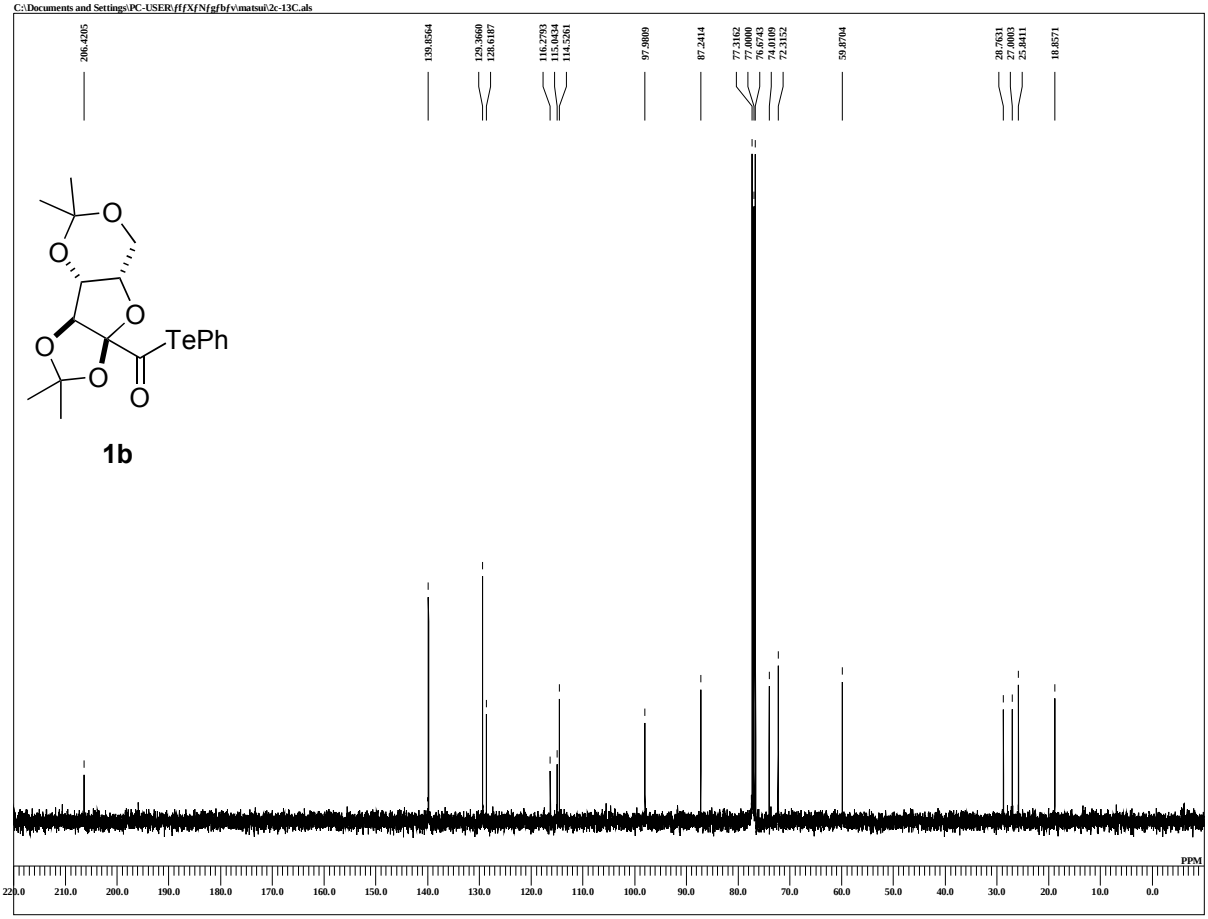

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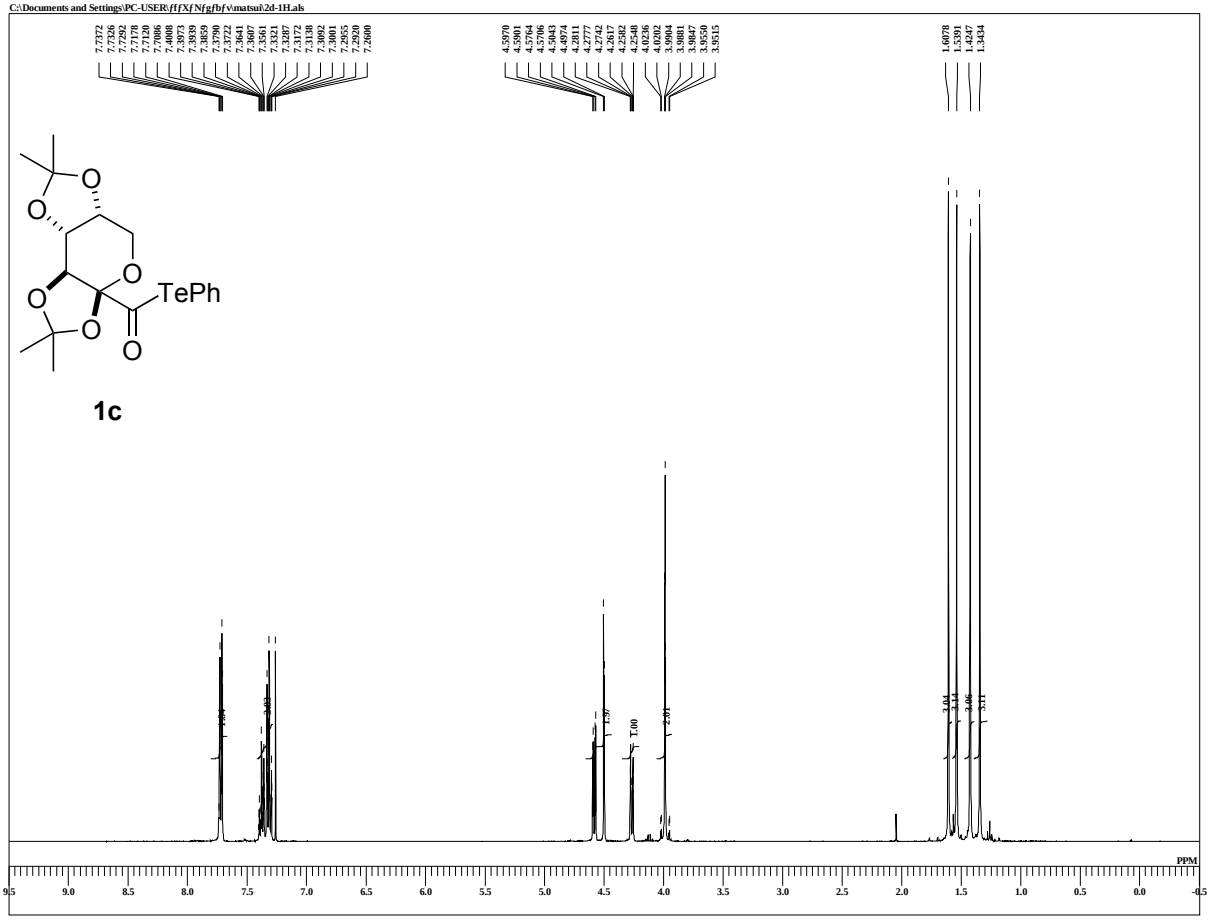

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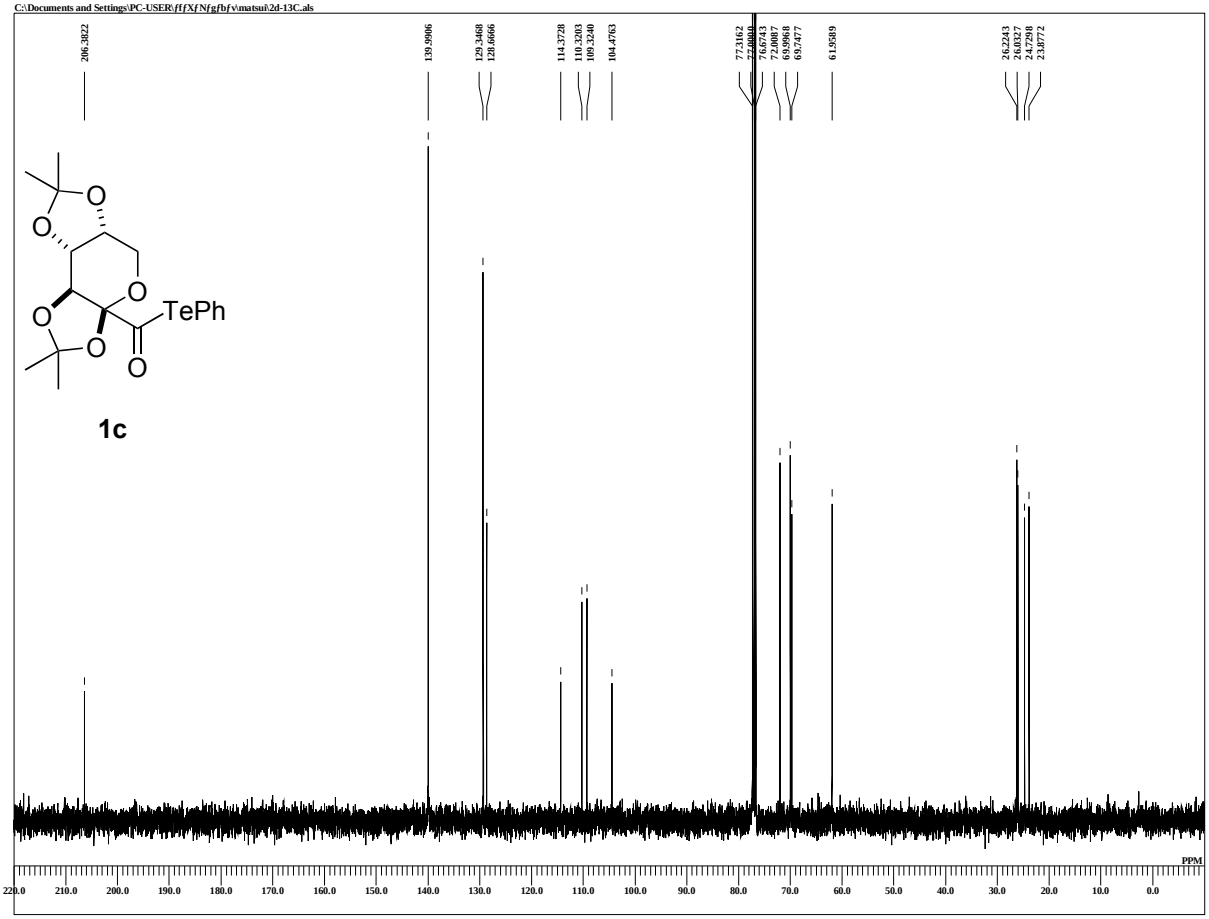

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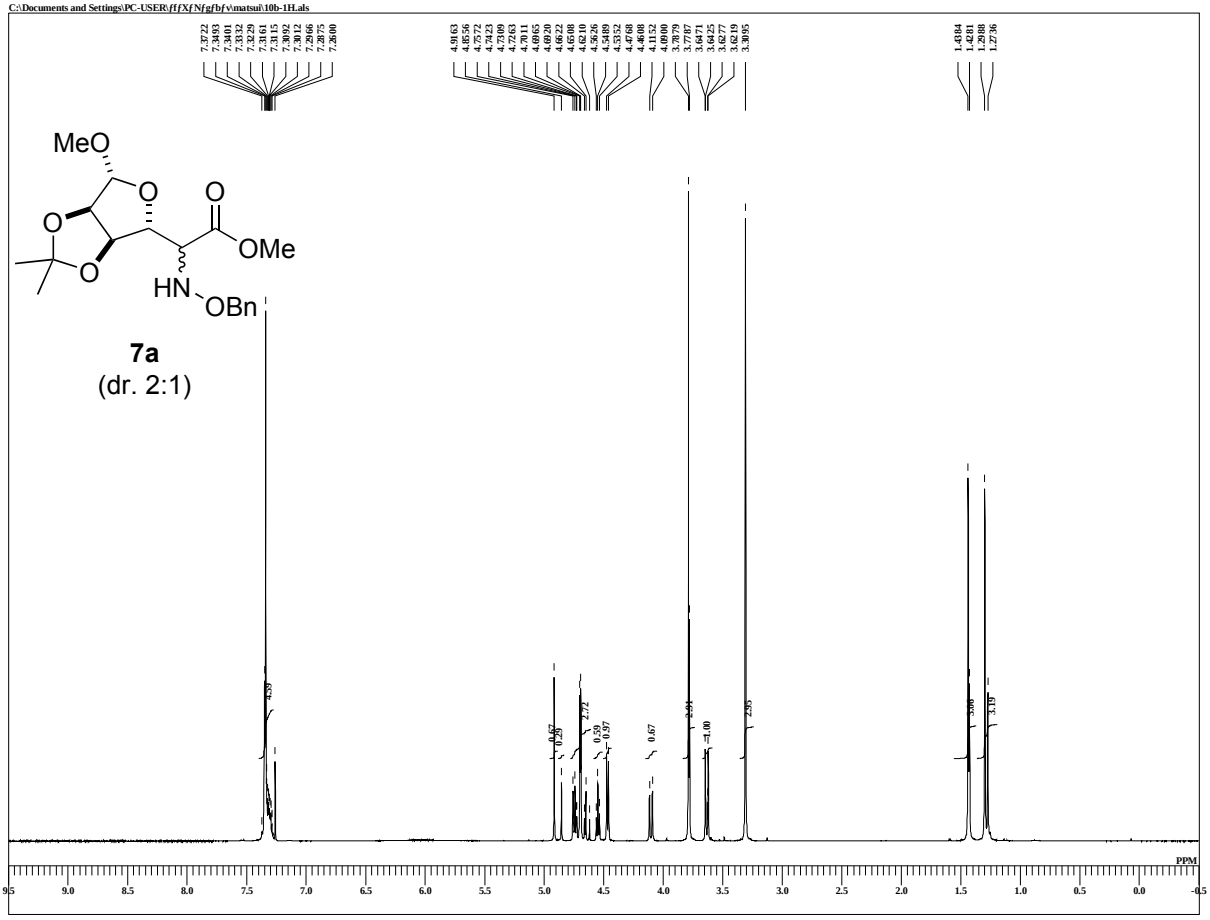


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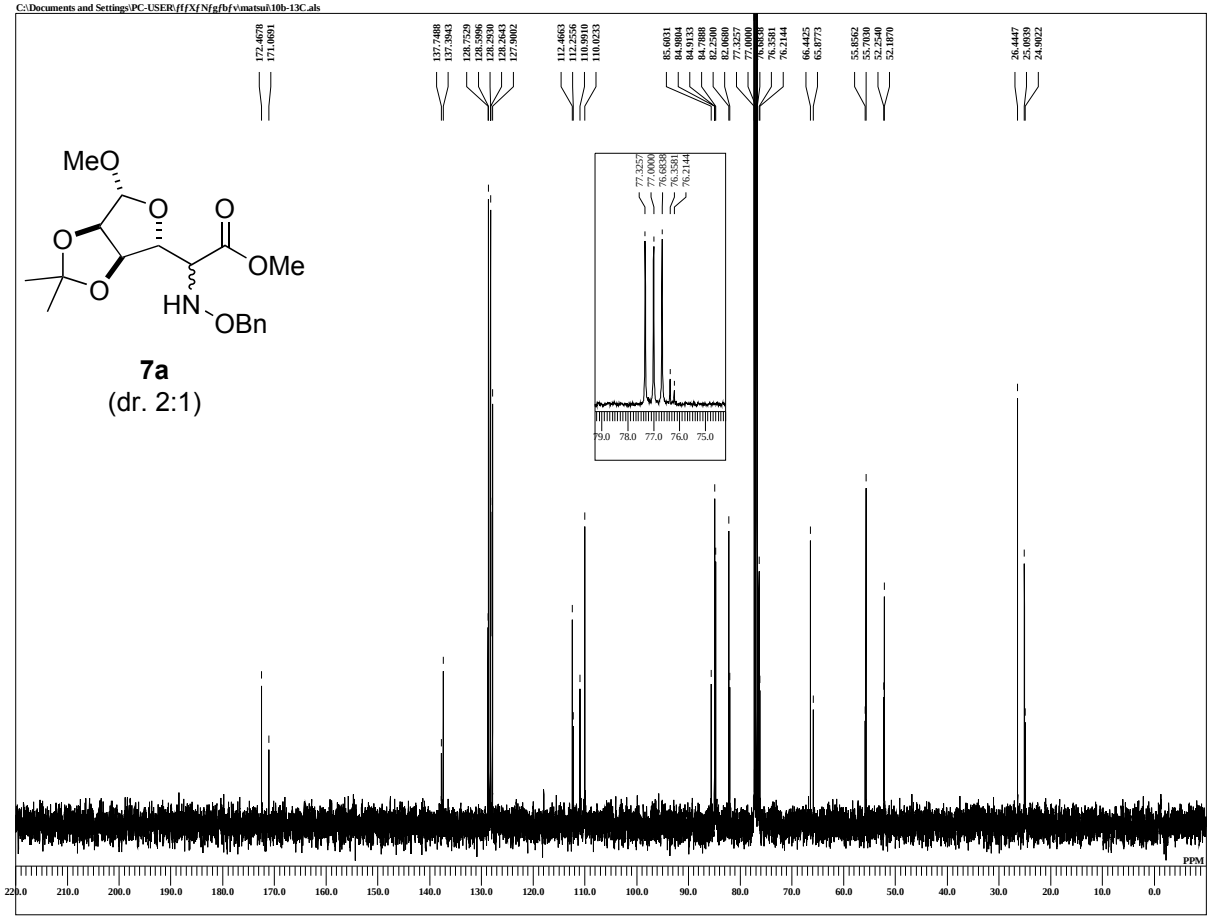
DFILE     10b-1H.als
COMNT     single_pulse
DATIM     24-01-2014 15:03:32
MENUF
OBNUC     1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET     6.28 KHz
OBFIN     0.87 Hz
PW1       6.44 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     13107
SPO       13107
TIMES     8
DUMMY     1
FREQU     5938.15 Hz
FLT       30000 Hz
DELAY     16.68 usec
ACQTM     2.2073 sec
PD        2.0000 sec
SCANS     8
ADBIT     16
RGAIN     34
BF        0.10 Hz
T1        0.00
T2        0.00
T3        100.00
T4        100.00
EXMOD     single_pulse.ex2
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     10b-1H.als
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     21.2 c
SLVNT     CDCL3
EXREF     7.26 ppm
```

single pulse decoupled gated NOE

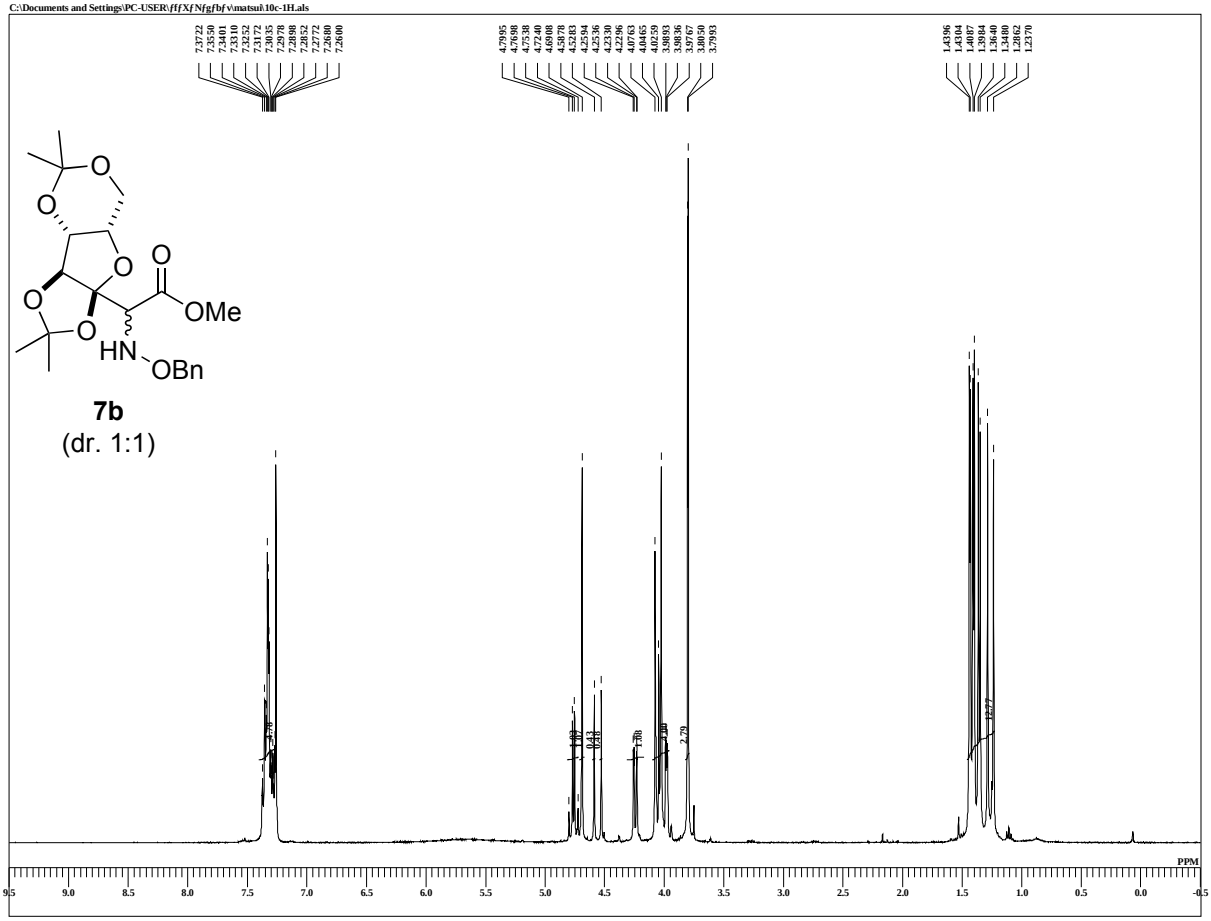
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\10b-13C.als




```
DFILE     10b-13C.als
COMNT     single pulse decoupled gat
DATIM     24-01-2014 17:01:56
MENUF
OBNUC     13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET     5.13 KHz
OBFIN     0.98 Hz
PW1       3.03 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     26214
SPO       26214
TIMES     201
DUMMY     4
FREQU     24999.62 Hz
FLT       125000 Hz
DELAY     20.50 usec
ACQTM     1.0486 sec
PD        2.0000 sec
SCANS     201
ADBIT     16
RGAIN     60
BF        1.00 Hz
T1        0.00
T2        0.00
T3        100.00
T4        100.00
EXMOD     single_pulse_dec
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     10b-13C.als
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     21.5 c
SLVNT     CDCL3
EXREF     77.00 ppm
```

single_pulse

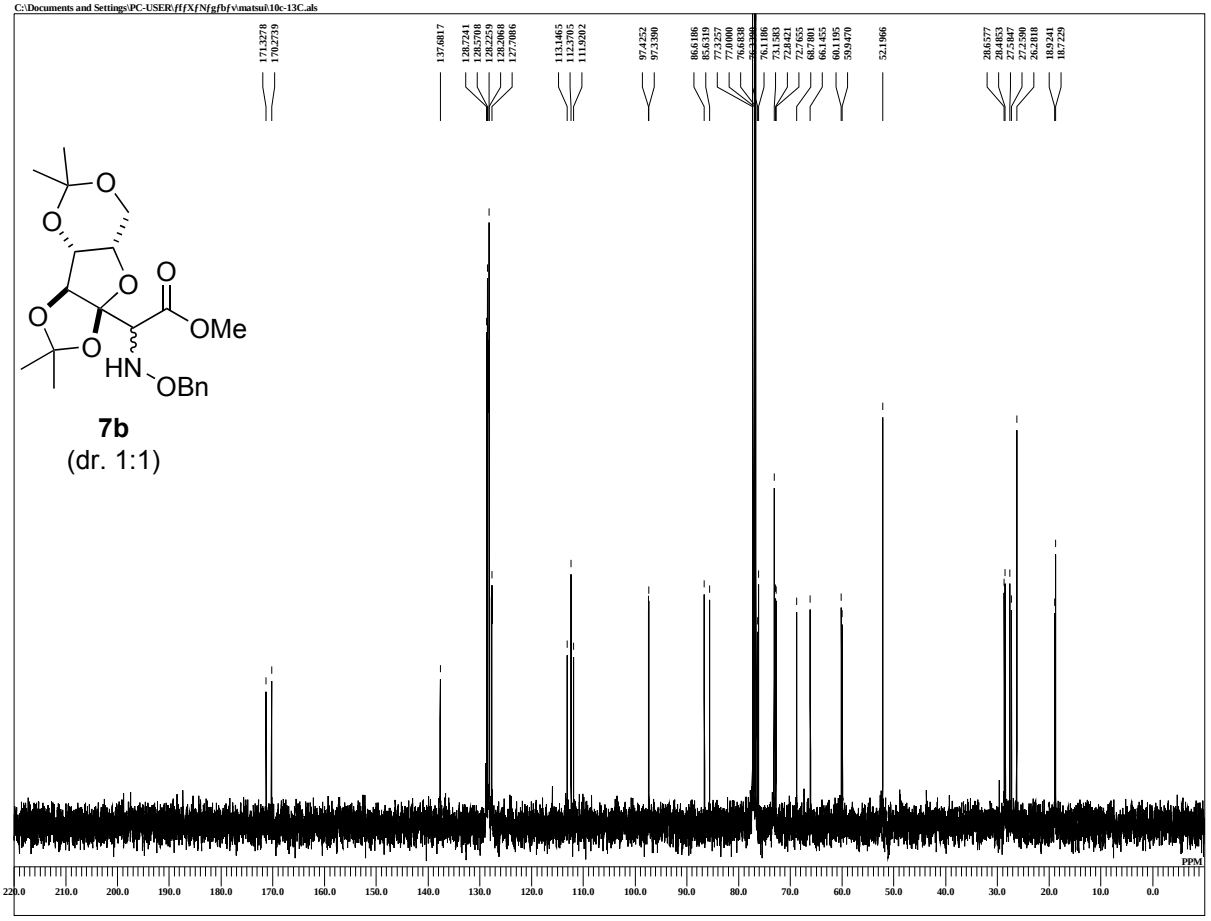
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\10c-1H.als



DFILE 10c-1H.als
COMNT single_pulse
DATIM 27-01-2014 18:21:20
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 32
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 10c-1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

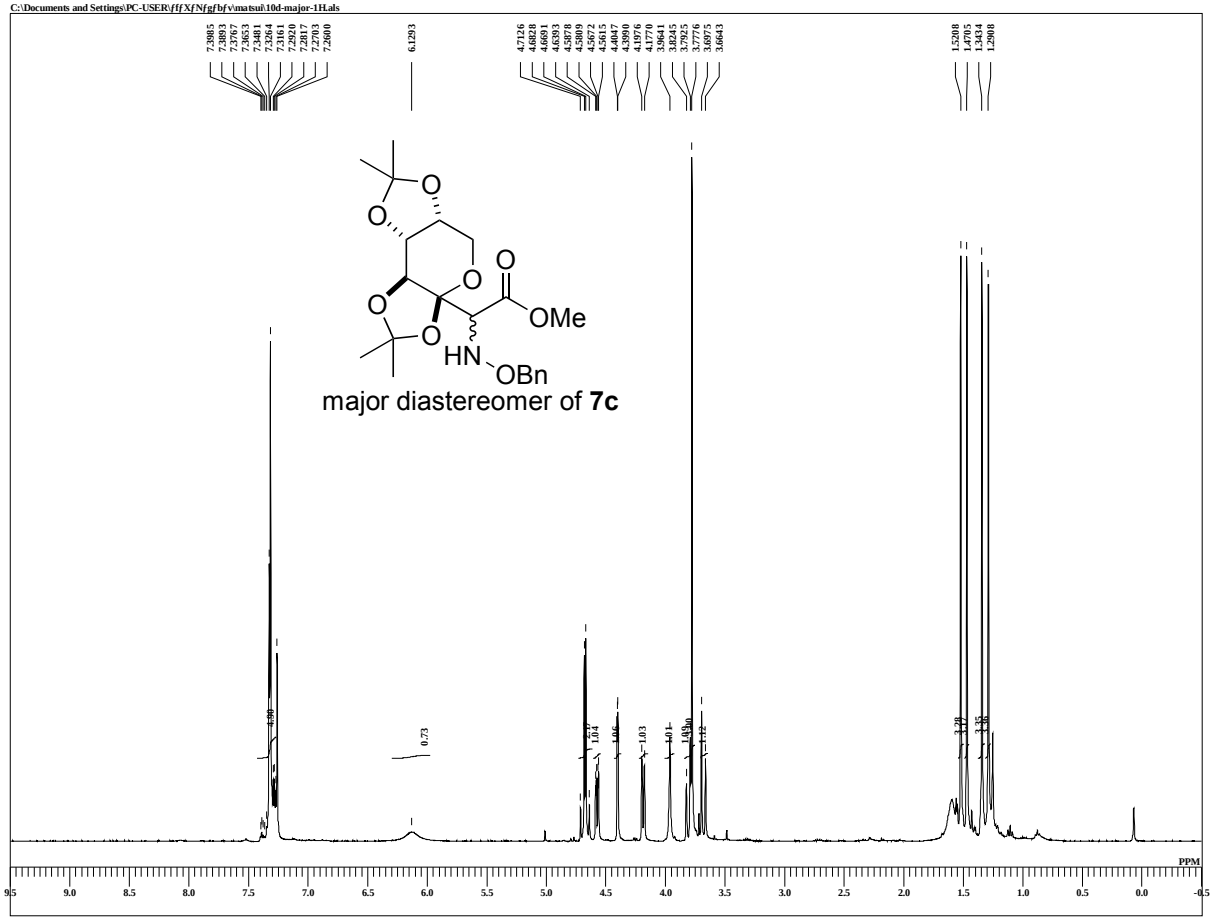
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\10c-13C.als



DFILE 10c-13C.als
COMNT single pulse decoupled gat
DATIM 08-11-2013 18:10:46
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 109
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 109
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 10c-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.4 c
SLVNT CDCL3
EXREF 77.00 ppm

YUM-2-156-fra20-24-fra13-15

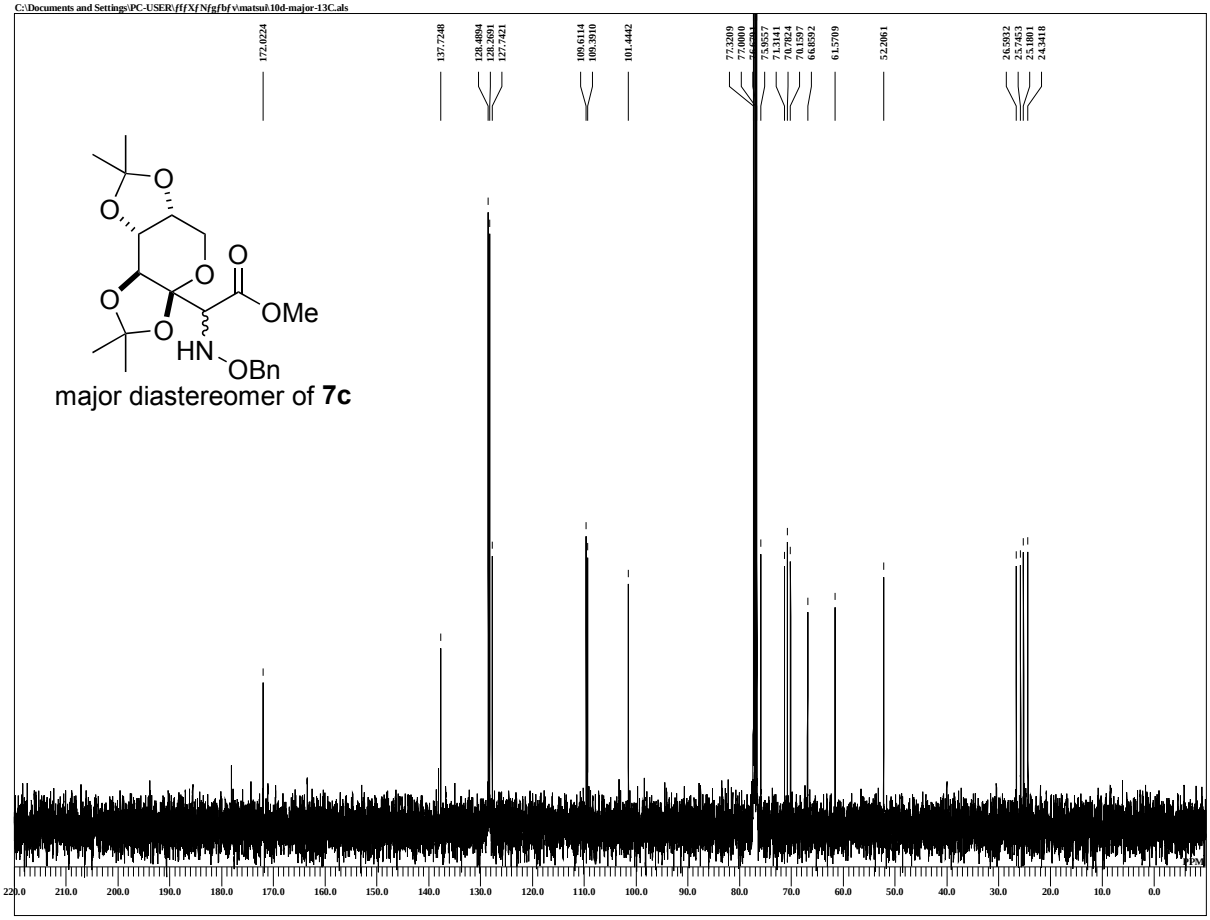
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\10d-major-1H.als




```
DFILE    10d-major-1H.als
COMNT    YUM-2-156-fra20-24-fra1
DATIM    05-08-2014 18:18:31
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.44 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    8
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    40
BF       0.50 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    147 usec
IRATN    79
DFILE    10d-major-1H.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    24.8 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

YUM-2-156-fra20-24-fra13-15

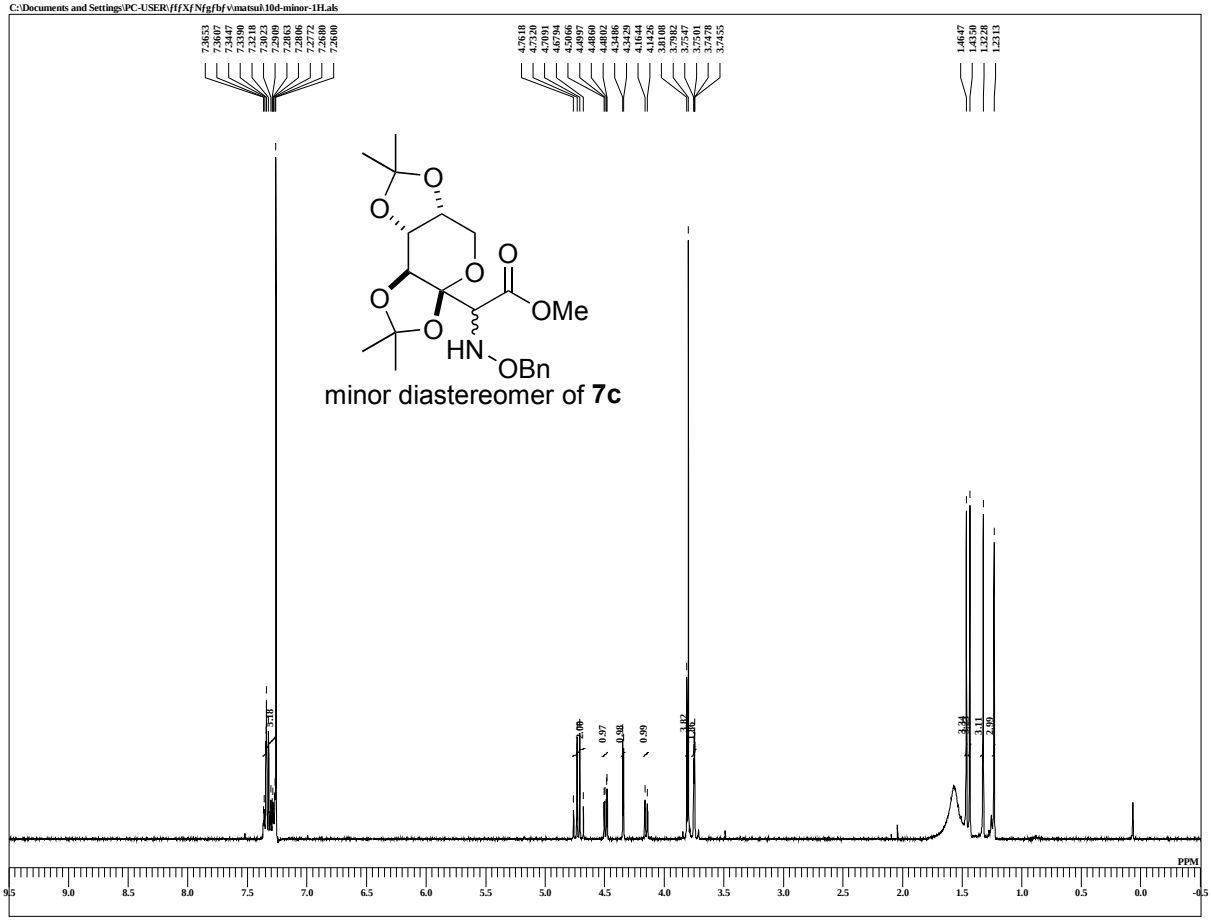
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\10d-major-13C.als




```
DFILE    10d-major-13C.als
COMNT    YUM-2-156-fra20-24-fra1
DATIM    05-08-2014 18:30:03
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.03 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    65536
SPO      65536
TIMES    221
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    221
ADBIT    16
RGAIN    50
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    10d-major-13C.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    25.0 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

YUM-2-156-fra28

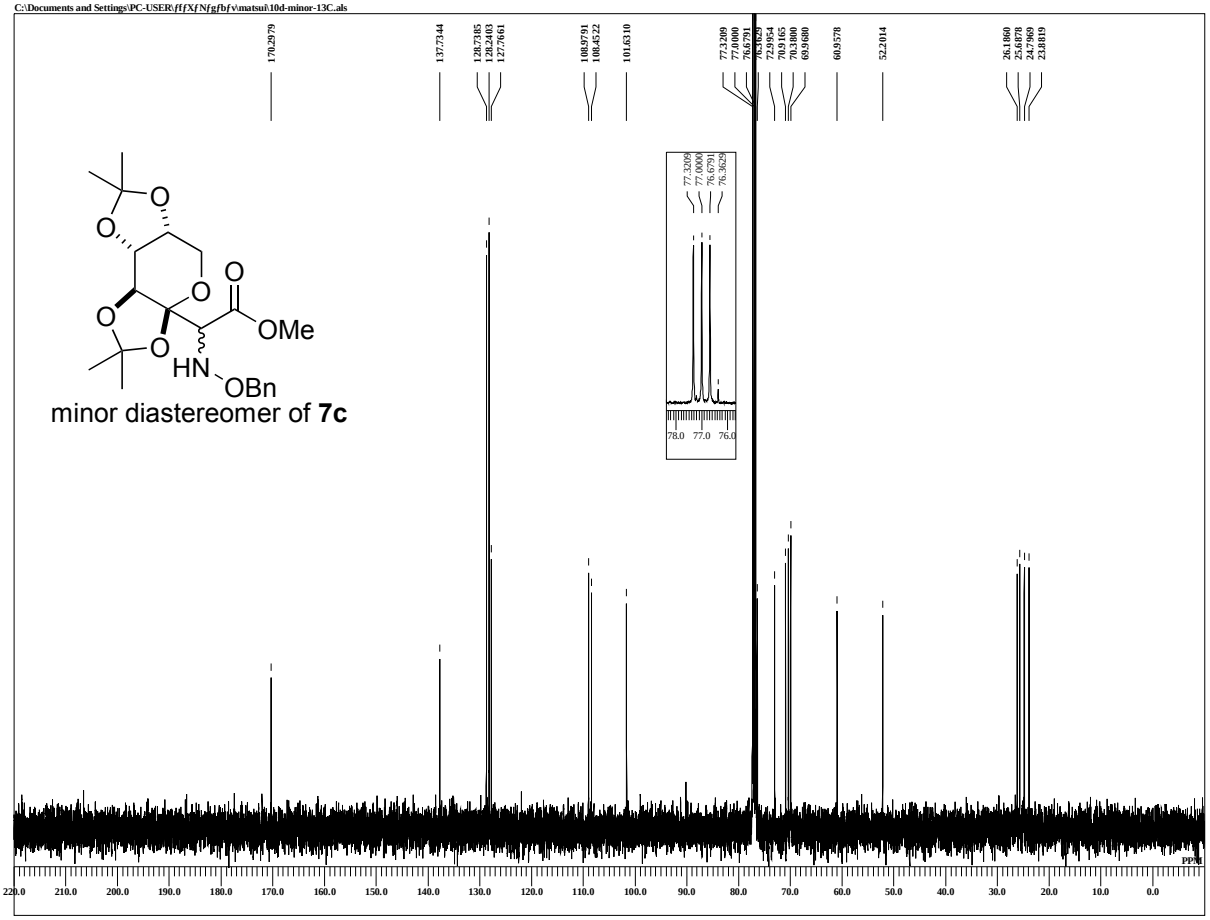
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\10d-minor-1H.als



DFILE 10d-minor-1H.als
COMNT YUM-2-156-fra28
DATIM 05-08-2014 11:24:46
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 48
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 10d-minor-1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.4 c
SLVNT CDCL3
EXREF 7.26 ppm

YUM-2-156-fra25-30

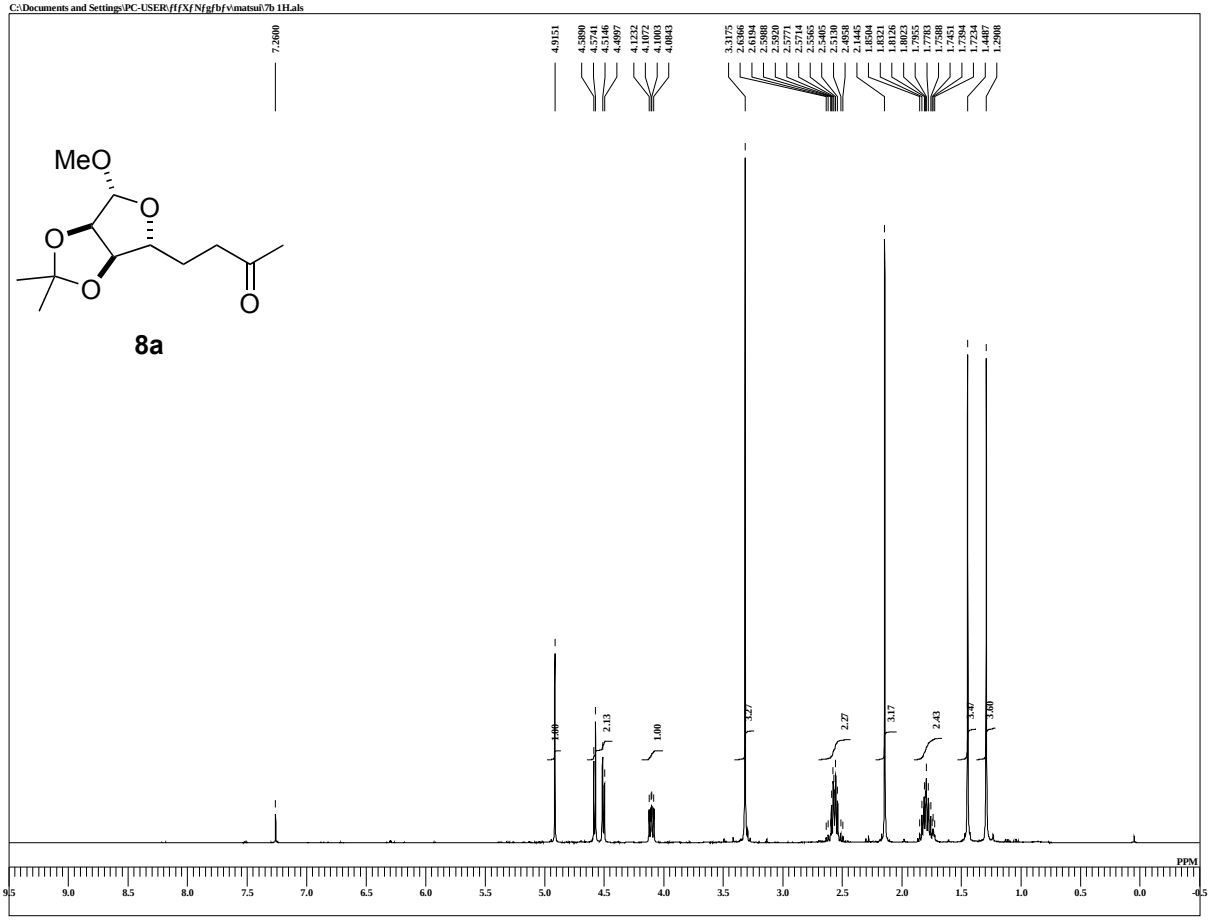
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\10d-minor-13C.als



DFILE 10d-minor-13C.als
COMNT YUM-2-156-fra25-30
DATIM 05-08-2014 18:51:17
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 52428
SPO 52428
TIMES 309
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 309
ADBIT 16
RGAIN 40
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 10d-minor-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.0 c
SLVNT CDCL3
EXREF 77.00 ppm

single_pulse

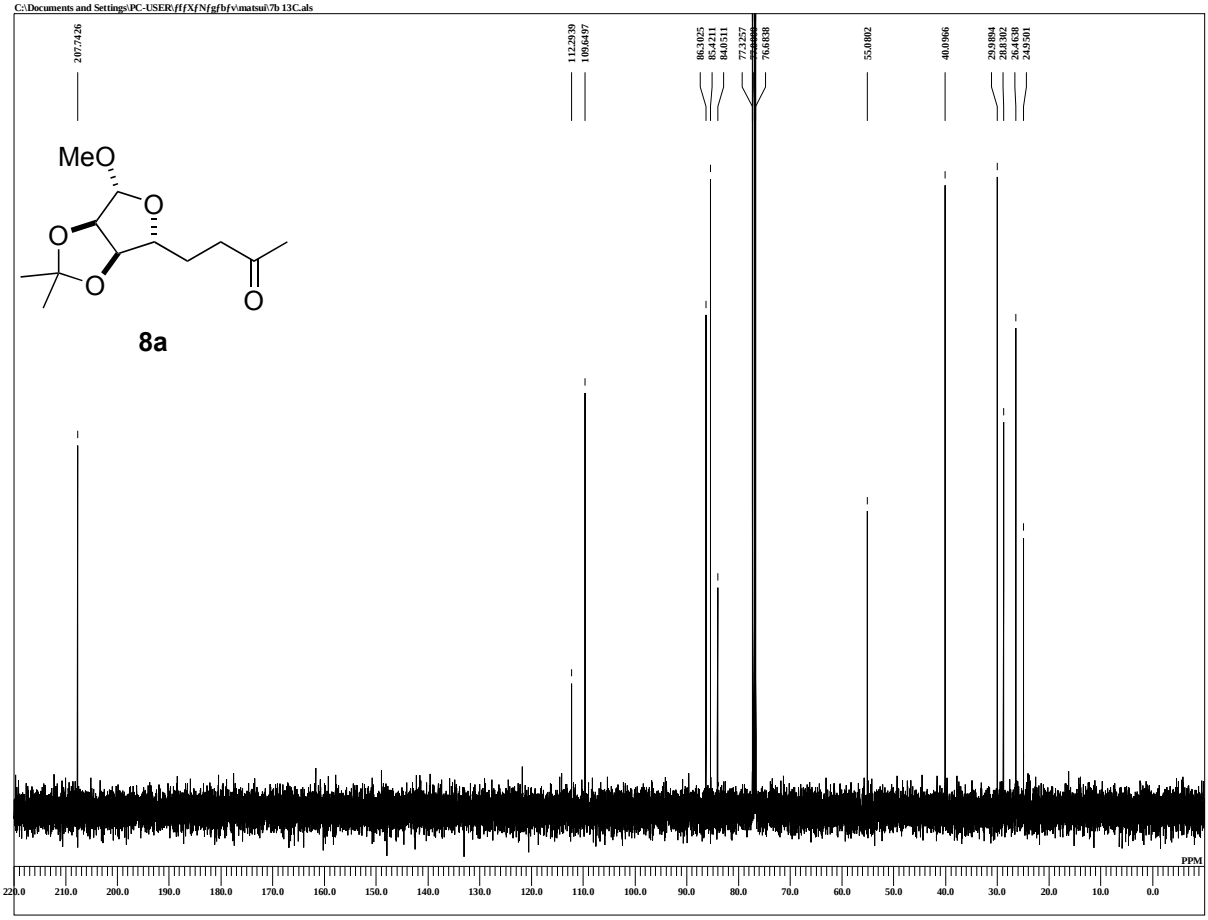
C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\7b 1H.als



DFILE 7b 1H.als
COMNT single_pulse
DATIM 30-10-2013 08:17:54
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE 7b 1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.5 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

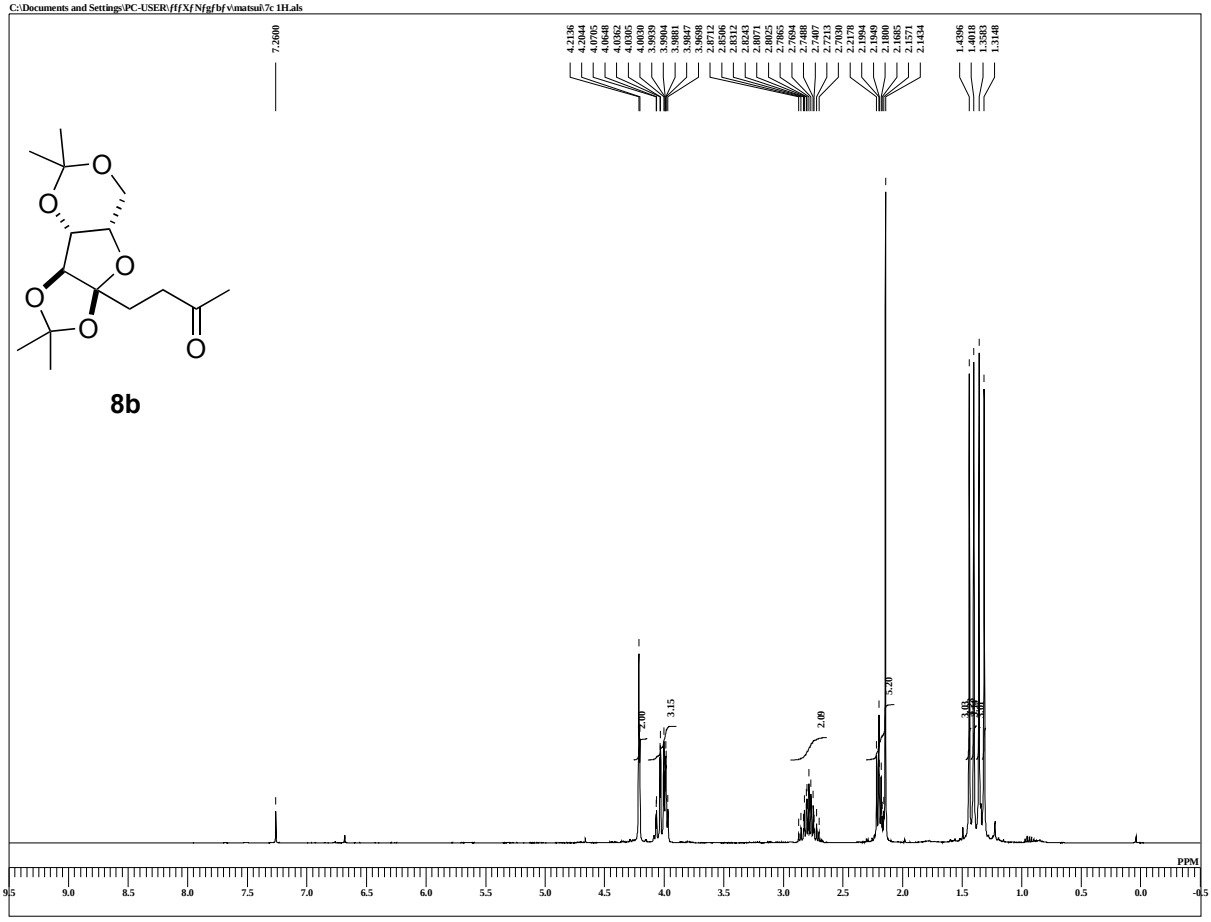
C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\7b 13C.als



DFILE 7b 13C.als
COMNT single pulse decoupled gat
DATIM 29-10-2013 09:12:19
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 102
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 102
ADBIT 16
RGAIN 60
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 7b 13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.4 c
SLVNT CDCL3
EXREF 77.00 ppm

single_pulse

C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\7c 1H.als

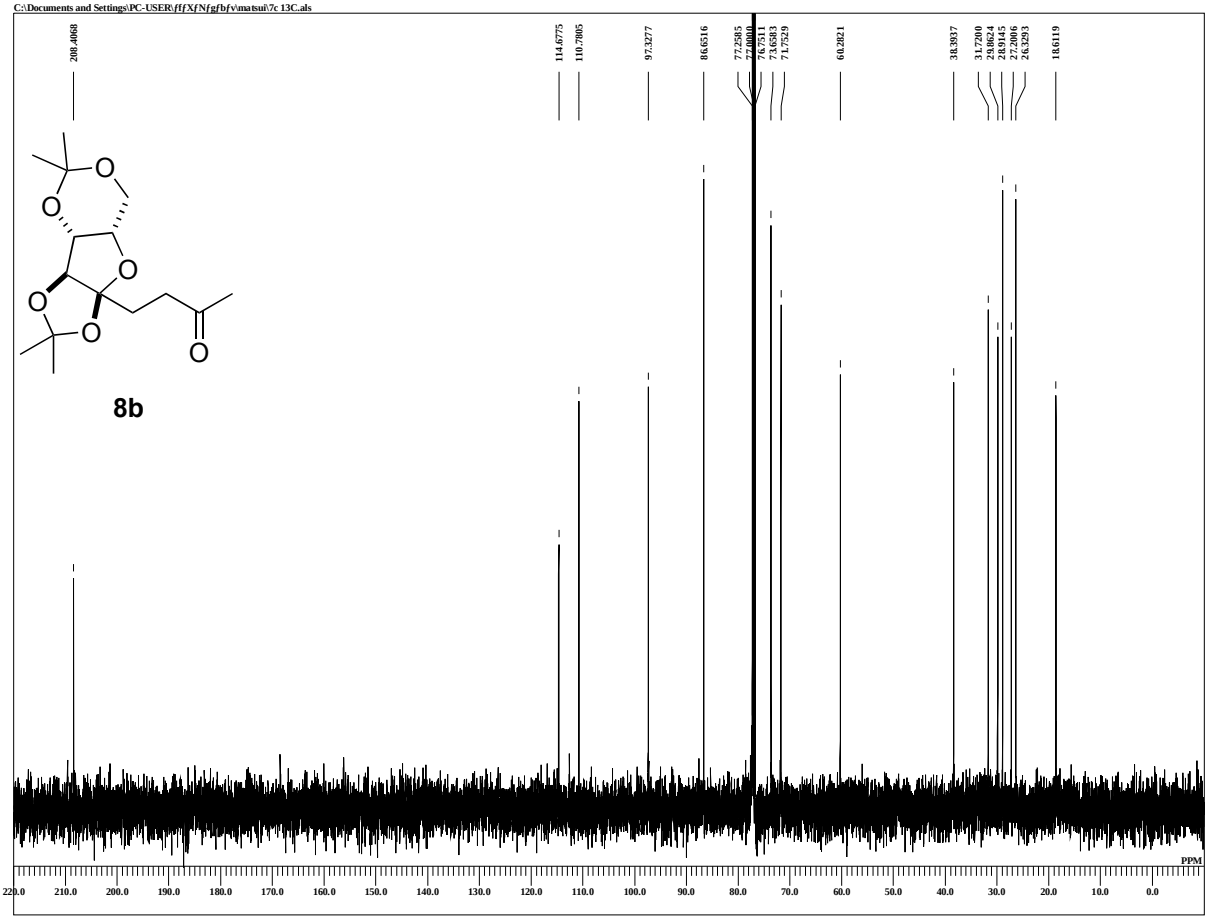


8b

DFILE 7c 1H.als
COMNT single_pulse
DATIM 28-01-2014 09:27:35
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 26
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 7c 1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.7 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\7c 13C.als

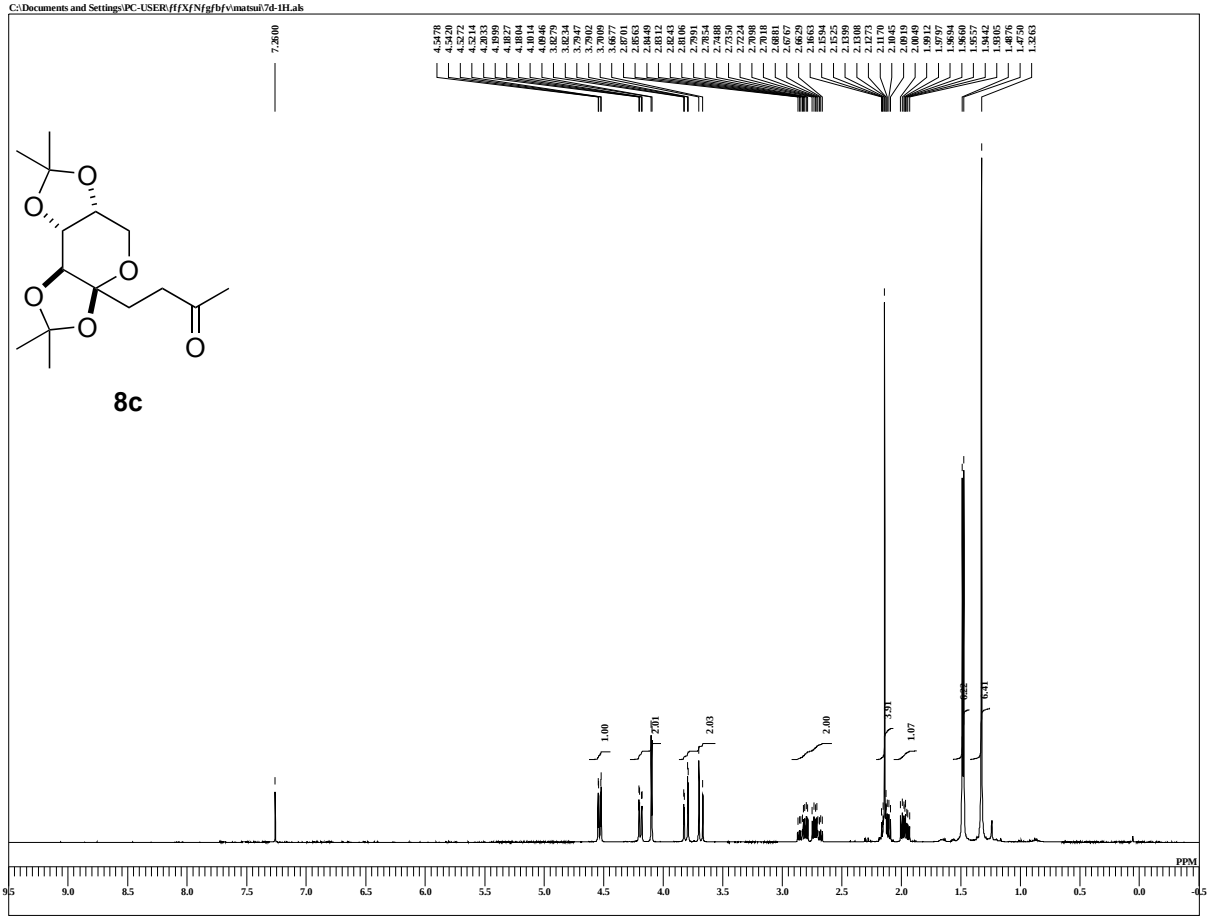


8b

DFILE 7c 13C.als
COMNT single pulse decoupled gat
DATIM 11-11-2013 09:14:49
MENUF
OBNUC 13C
OFR 124.51 MHz
OBFRQ 124.51 MHz
OBSET 3.45 KHz
OBFIN 6.00 Hz
PW1 3.70 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 91
DUMMY 4
FREQU 31249.52 Hz
FLT 157000 Hz
DELAY 20.80 usec
ACQTM 0.8389 sec
PD 2.0000 sec
SCANS 91
ADBIT 16
RGAIN 48
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 495.13 MHz
IRSET 4.38 KHz
IRFIN 9.64 Hz
IRRPW 92 usec
IRATN 79
DFILE 7c 13C.als
SF
LKSET 748.40 KHz
LKFIN 98.2 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.9 c
SLVNT CDCL3
EXREF 77.00 ppm

single_pulse

C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\7d-1H.als

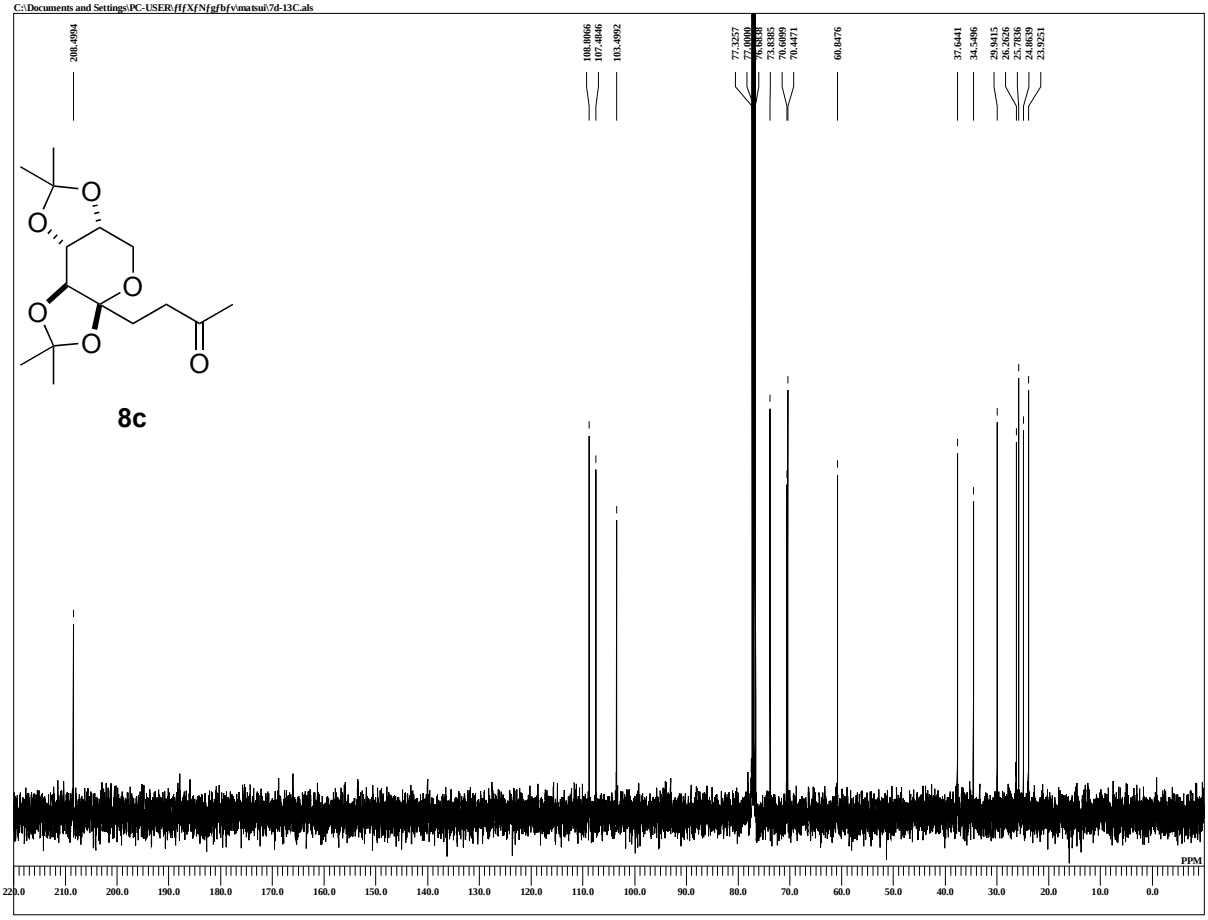


8c

DFILE 7d-1H.als
COMNT single_pulse
DATIM 17-01-2014 15:45:01
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 7d-1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 20.9 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\7d-13C.als

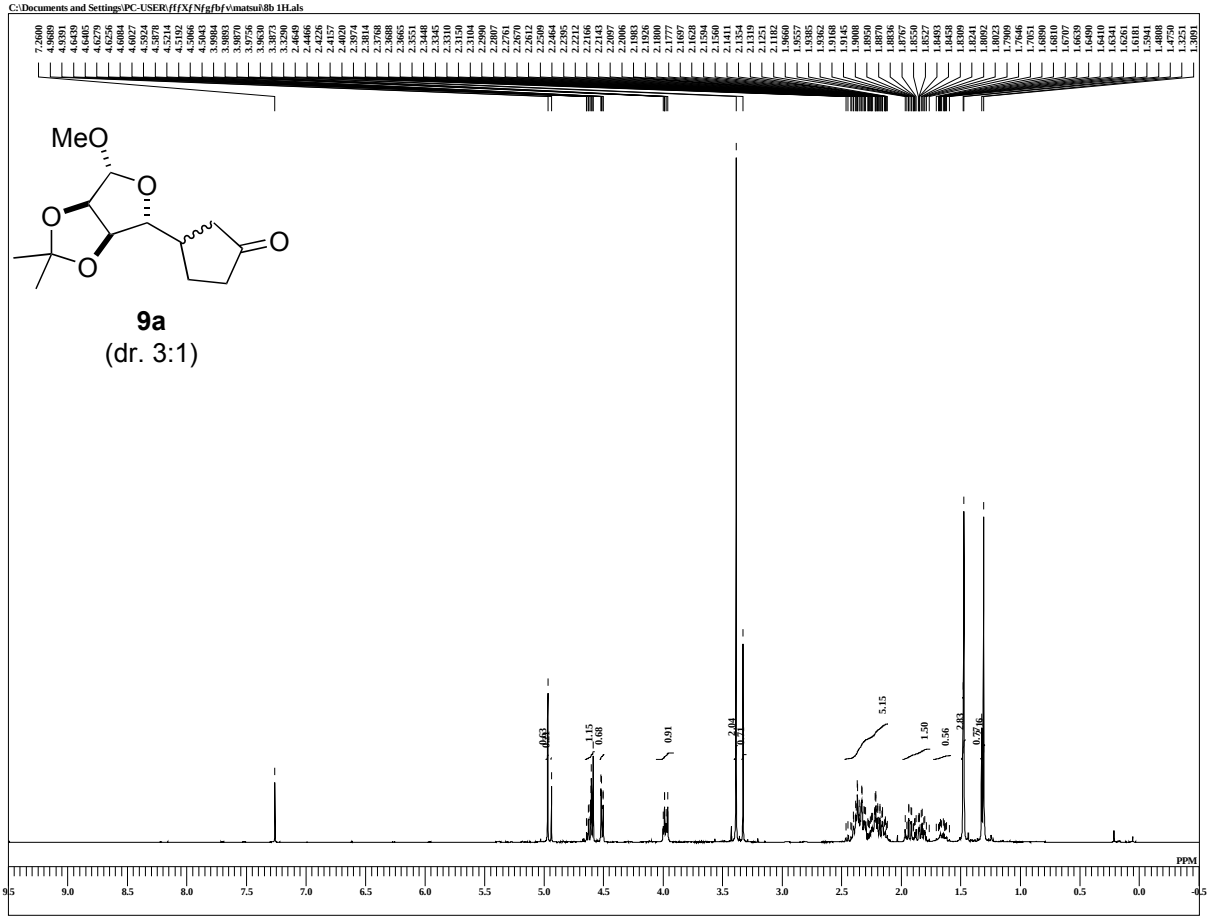


8c

DFILE 7d-13C.als
COMNT single pulse decoupled ga
DATIM 17-01-2014 15:49:48
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 83
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 83
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 7d-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm

single_pulse

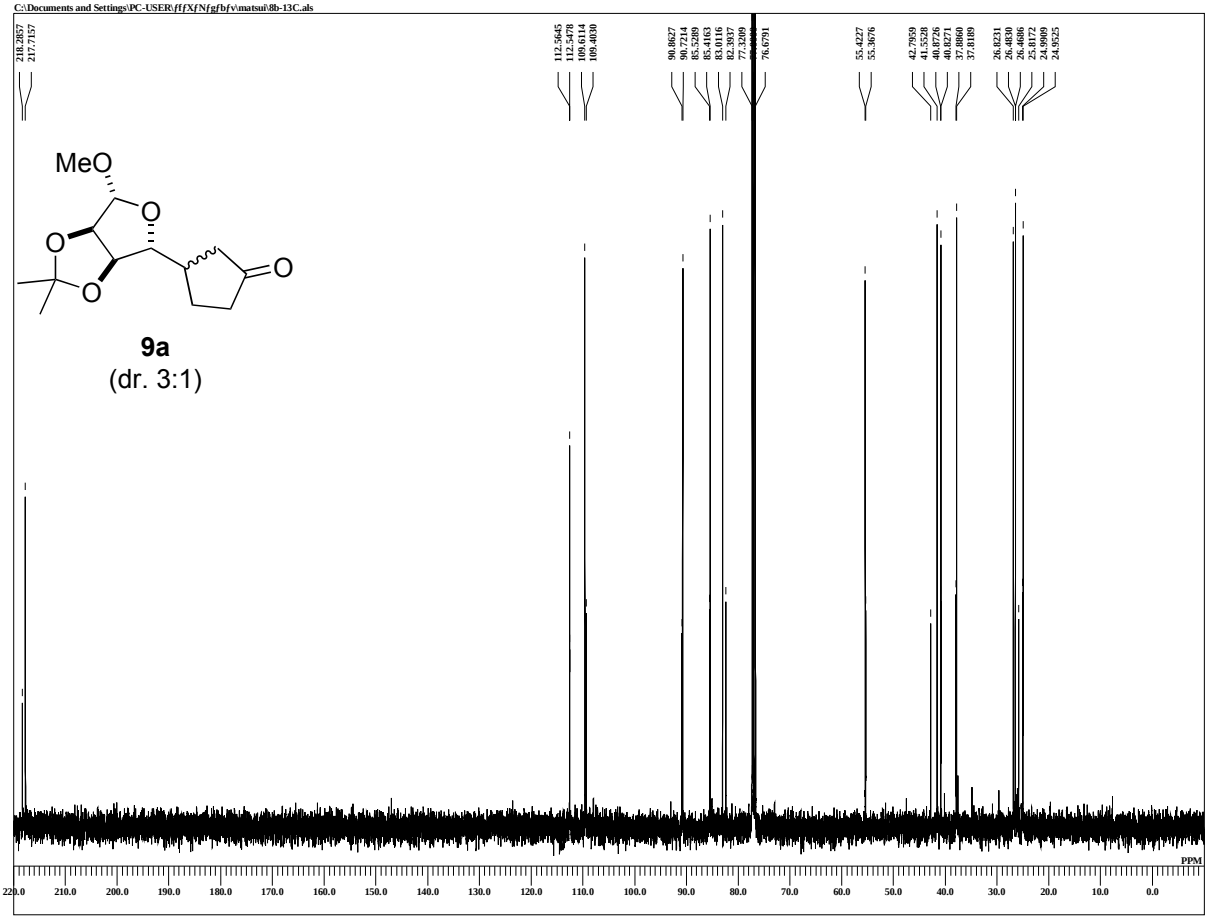
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\8b 1H.als



DFILE 8b 1H.als
COMNT single_pulse
DATIM 30-10-2013 16:28:02
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 34
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 8b 1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.6 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

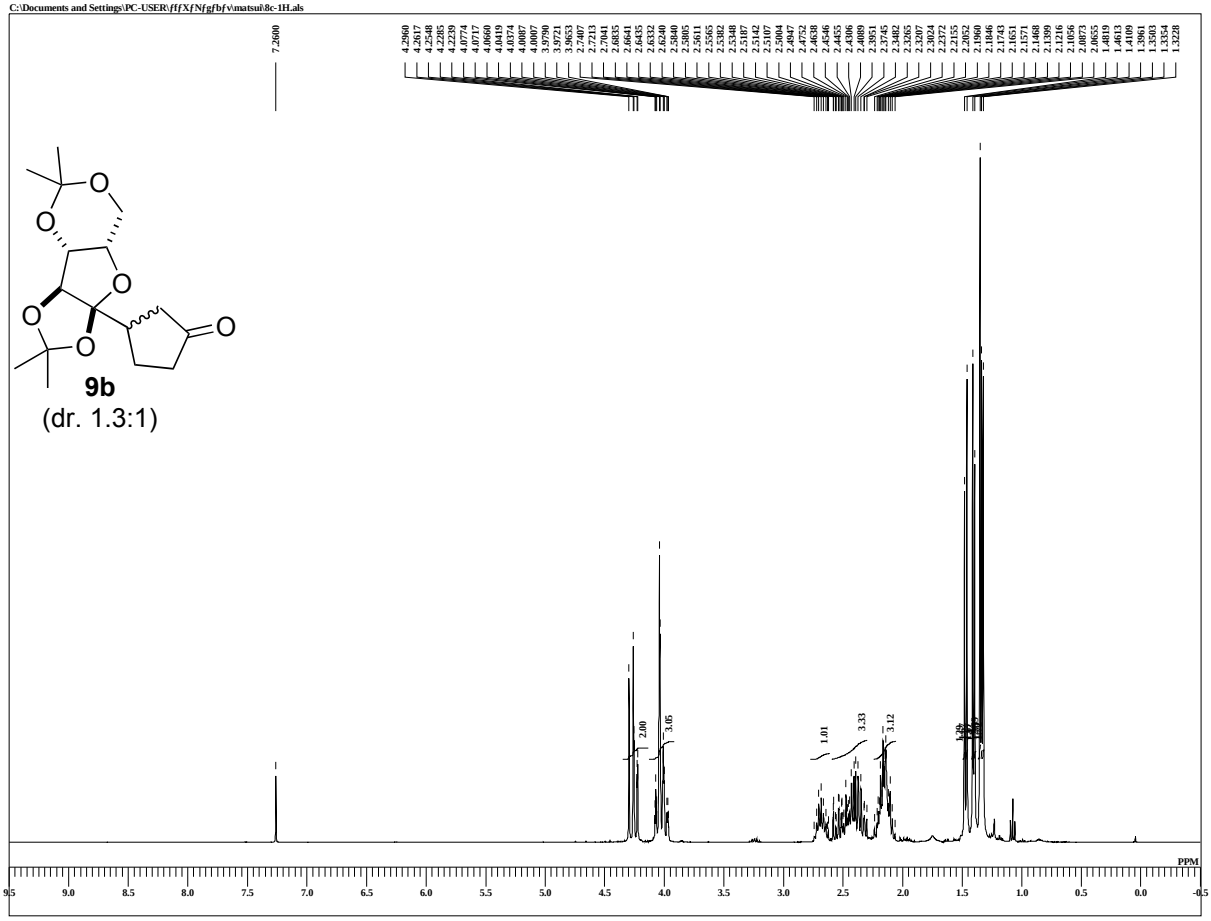
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\8b-13C.als



DFILE 8b-13C.als
COMNT single pulse decoupled gat
DATIM 30-10-2013 08:52:27
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 104856
SPO 104856
TIMES 200
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 200
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 8b-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 25.7 c
SLVNT CDCL3
EXREF 77.00 ppm

single_pulse

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\8c-1H.als



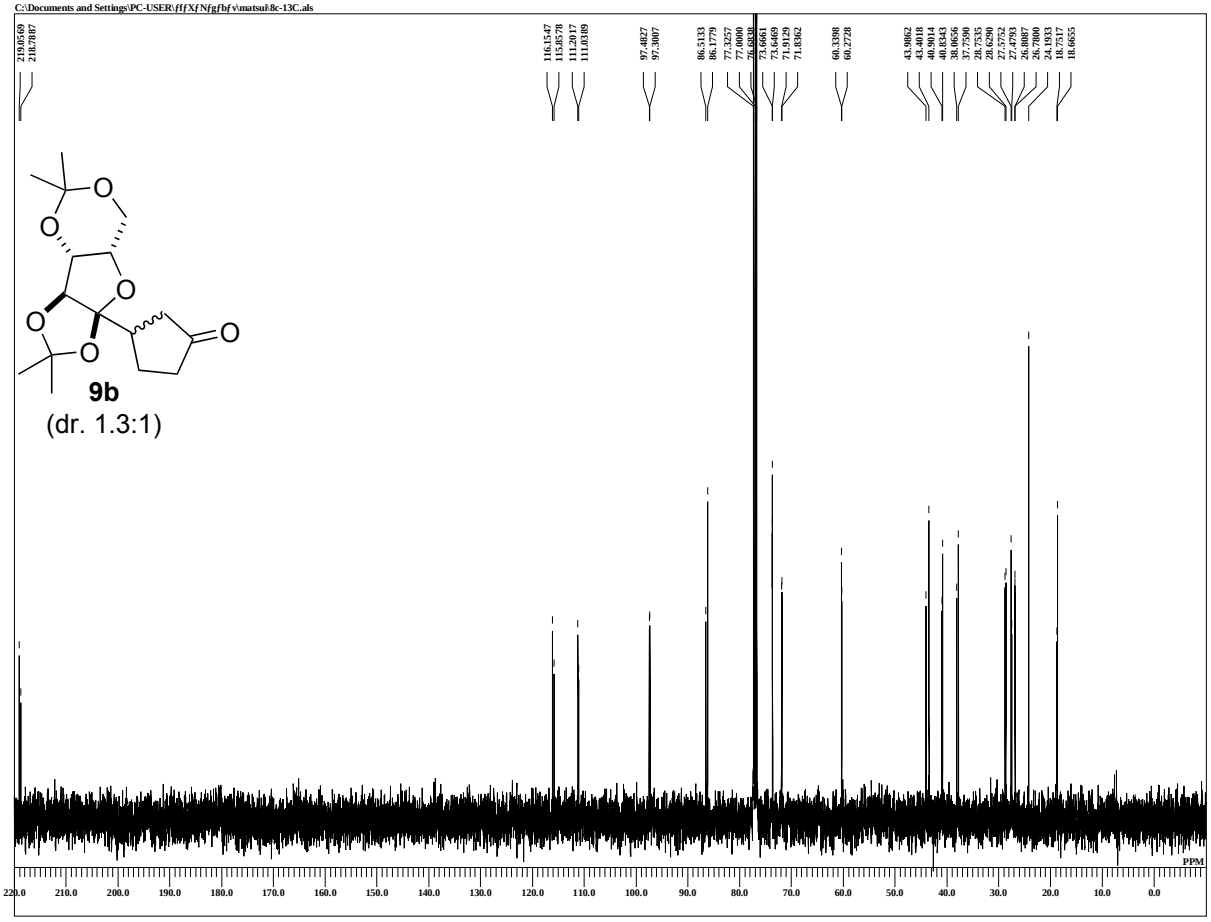
9b

(dr. 1.3:1)

DFILE 8c-1H.als
COMNT single_pulse
DATIM 29-01-2014 17:07:55
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 28
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 8c-1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.3 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\8c-13C.als



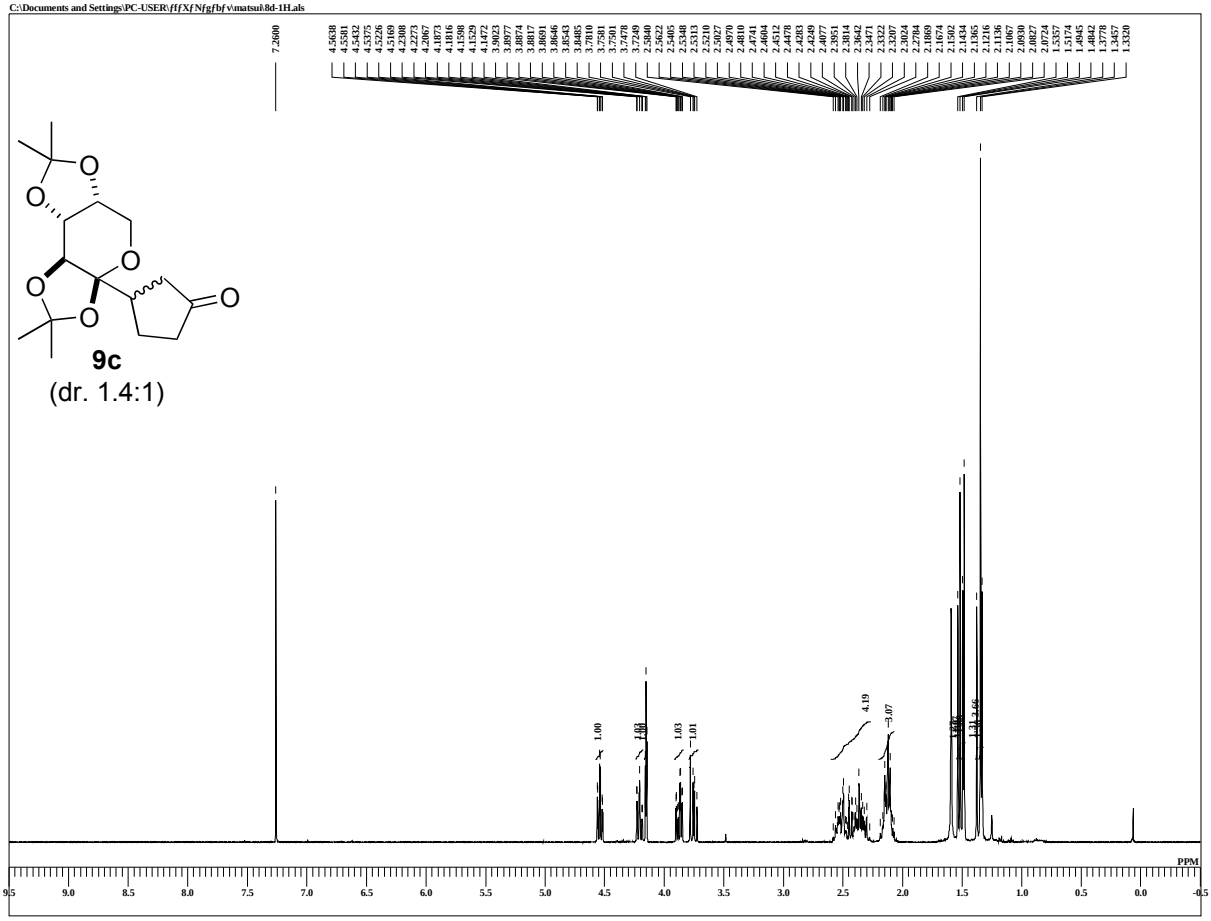
9b

(dr. 1.3:1)

DFILE 8c-13C.als
COMNT single pulse decoupled gat
DATIM 16-11-2013 15:20:15
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 102
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 102
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 8c-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.2 c
SLVNT CDCL3
EXREF 77.00 ppm

YUM-2-155-fra7

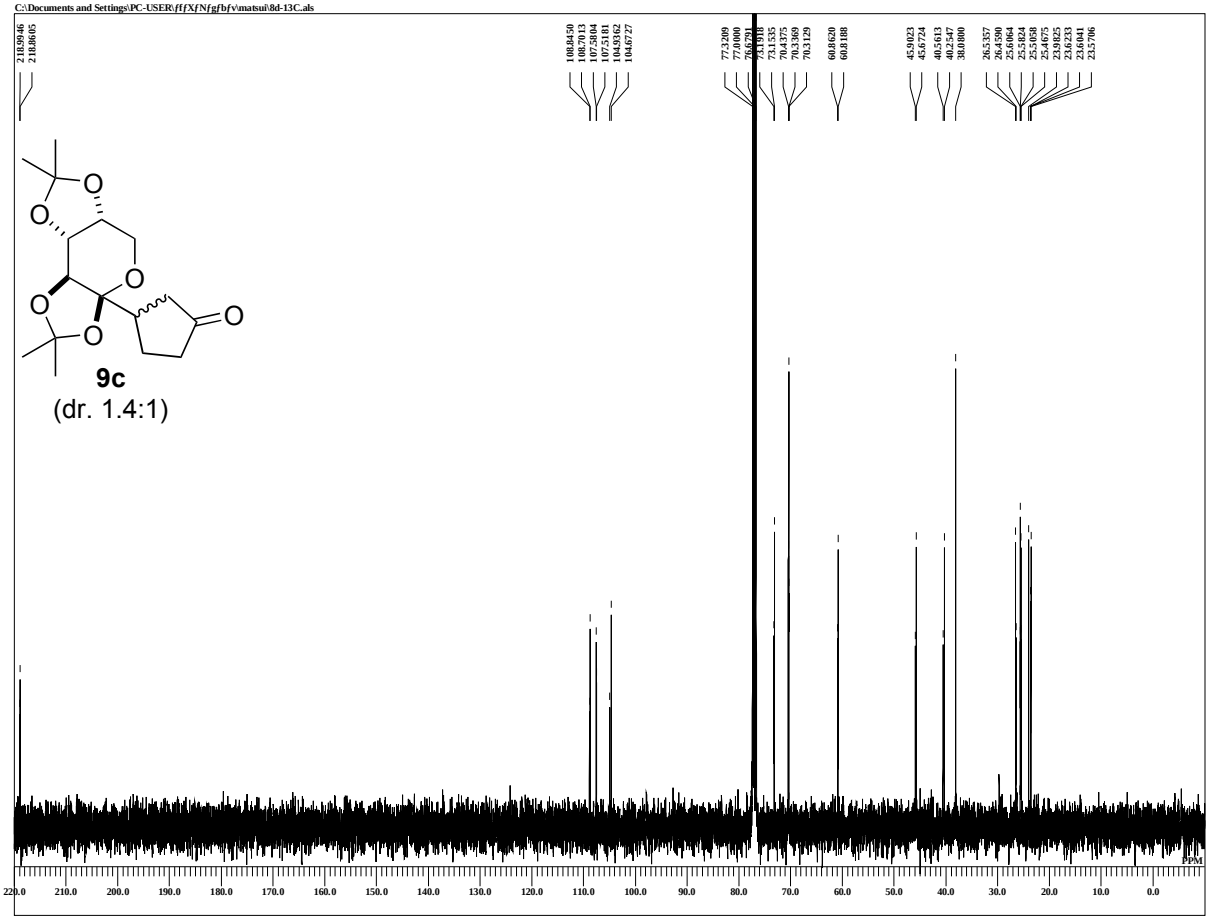
C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\8d-1H.als




```
DFILE    8d-1H.als
COMNT    YUM-2-155-fra7
DATIM    05-08-2014 21:53:10
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.44 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    16384
SPO      16384
TIMES    8
DUMMY    1
FREQU    7422.80 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    42
BF       0.01 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    8d-1H.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    25.0 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

YUM-2-155-fra6-9

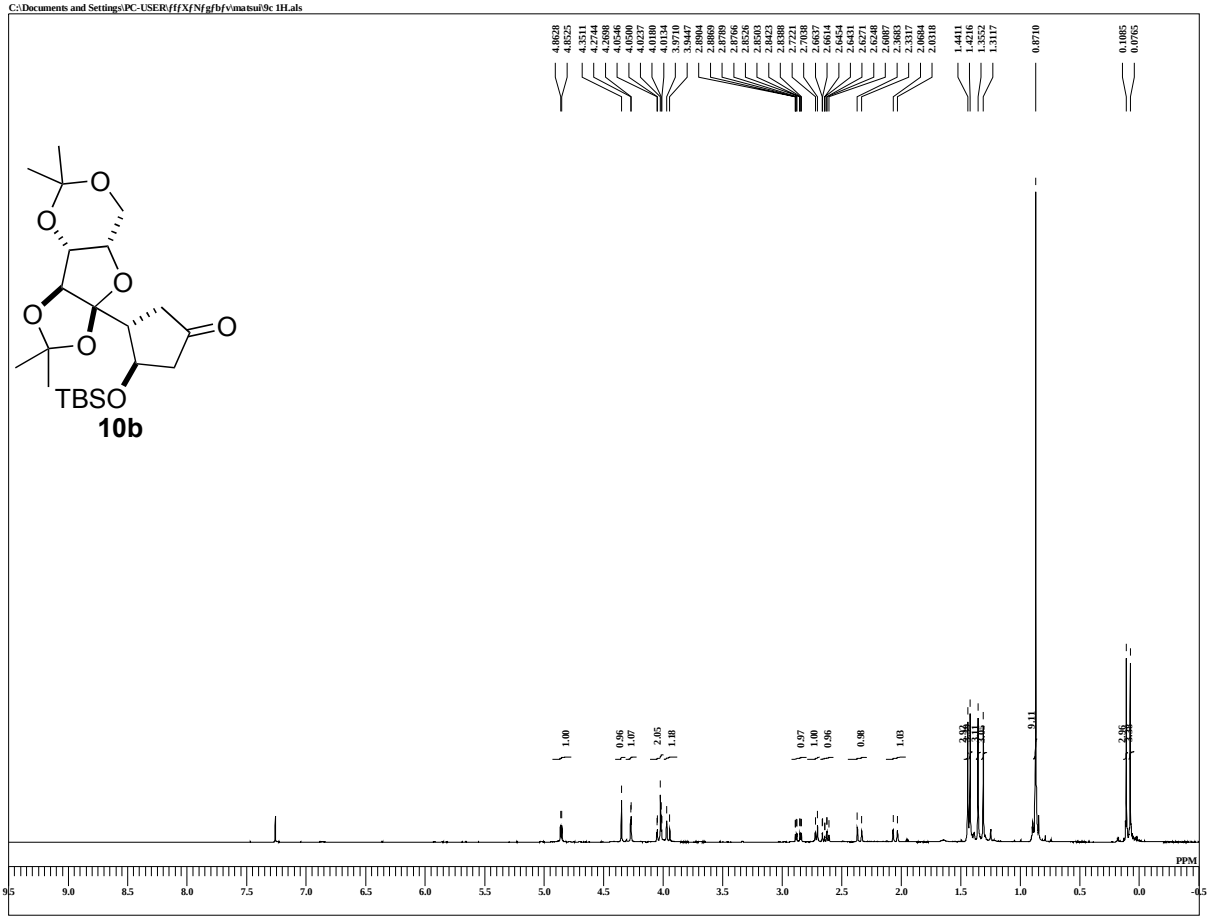
C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\8d-13C.als




```
DFILE    8d-13C.als
COMNT    YUM-2-155-fra6-9
DATIM    11-09-2014 18:55:58
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.03 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    65536
SPO      65536
TIMES    309
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    309
ADBIT    16
RGAIN    38
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    8d-13C.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    24.9 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

single_pulse

C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\9c 1H.als



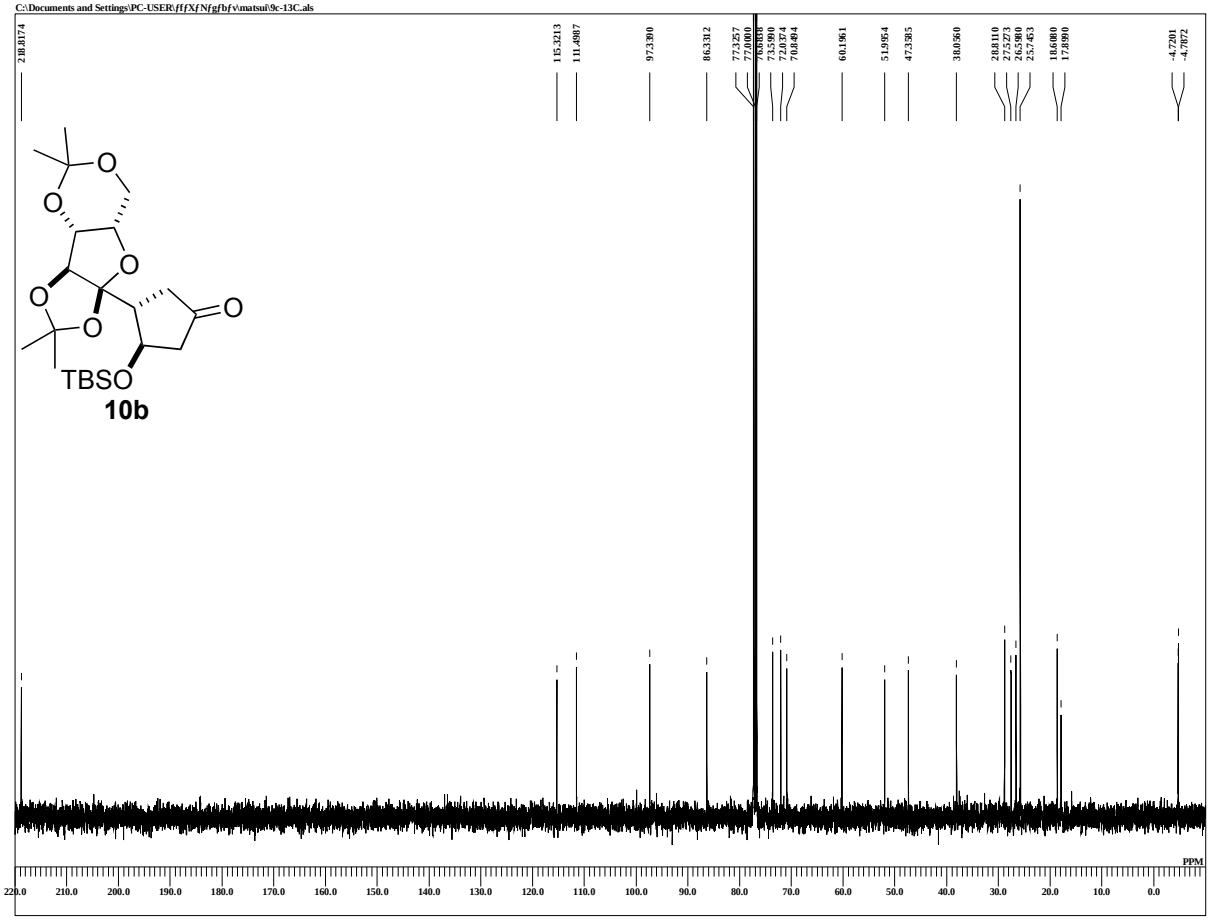
TBSO

10b

DFILE 9c 1H.als
COMNT single_pulse
DATIM 15-11-2013 19:46:35
MENUF
OBNUC 1H
OFR 495.13 MHz
OBFRQ 495.13 MHz
OBSET 4.38 KHz
OBFIN 9.64 Hz
PW1 6.00 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 7429.31 Hz
FLT 38000 Hz
DELAY 13.16 usec
ACQTM 1.7642 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 36
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 495.13 MHz
IRSET 4.38 KHz
IRFIN 9.64 Hz
IRRPW 92 usec
IRATN 79
DFILE 9c 1H.als
SF
LKSET 748.40 KHz
LKFIN 98.2 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.7 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\9c-13C.als



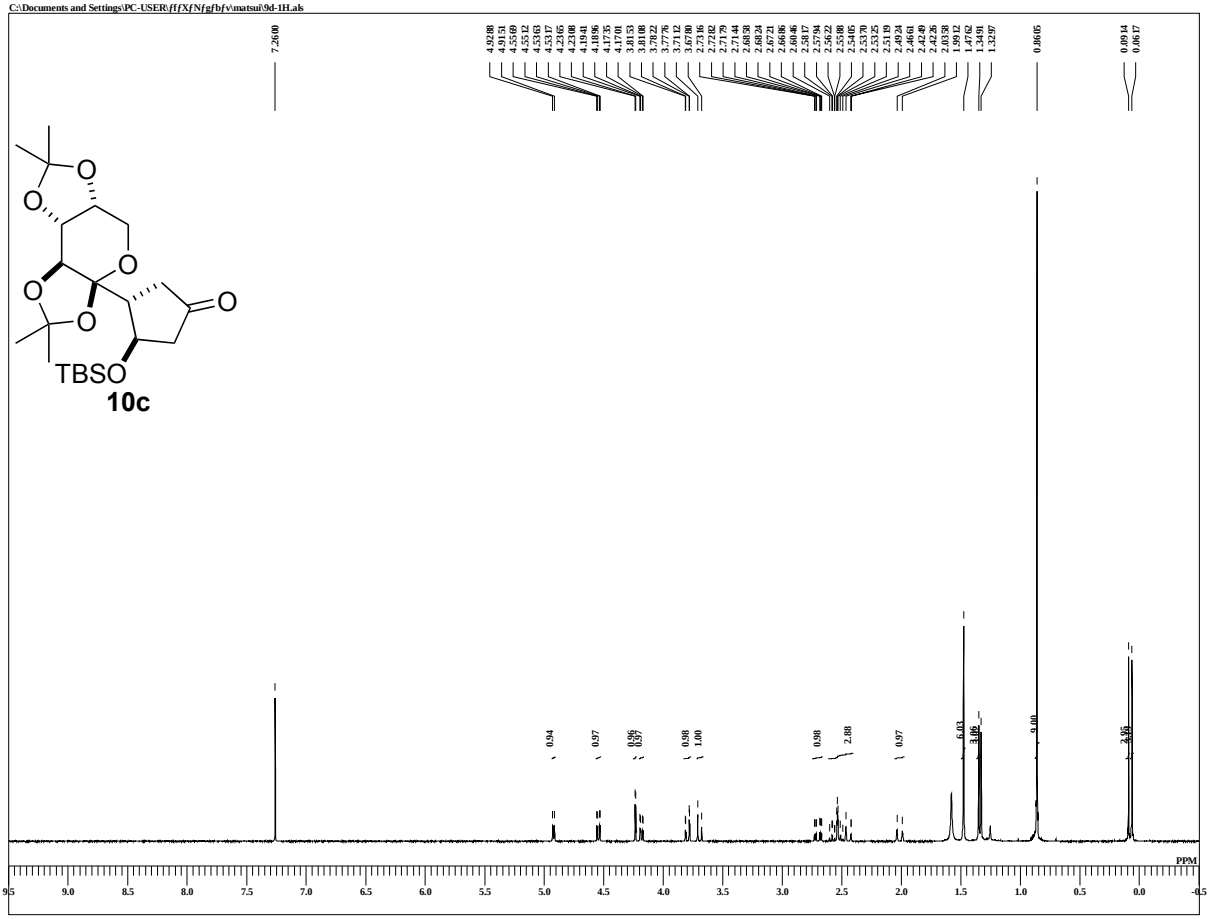
TBSO

10b

DFILE 9c-13C.als
COMNT single pulse decoupled gat
DATIM 15-11-2013 15:37:13
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 101
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 101
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 9c-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.2 c
SLVNT CDCL3
EXREF 77.00 ppm

YUM-2-153-fra16-18

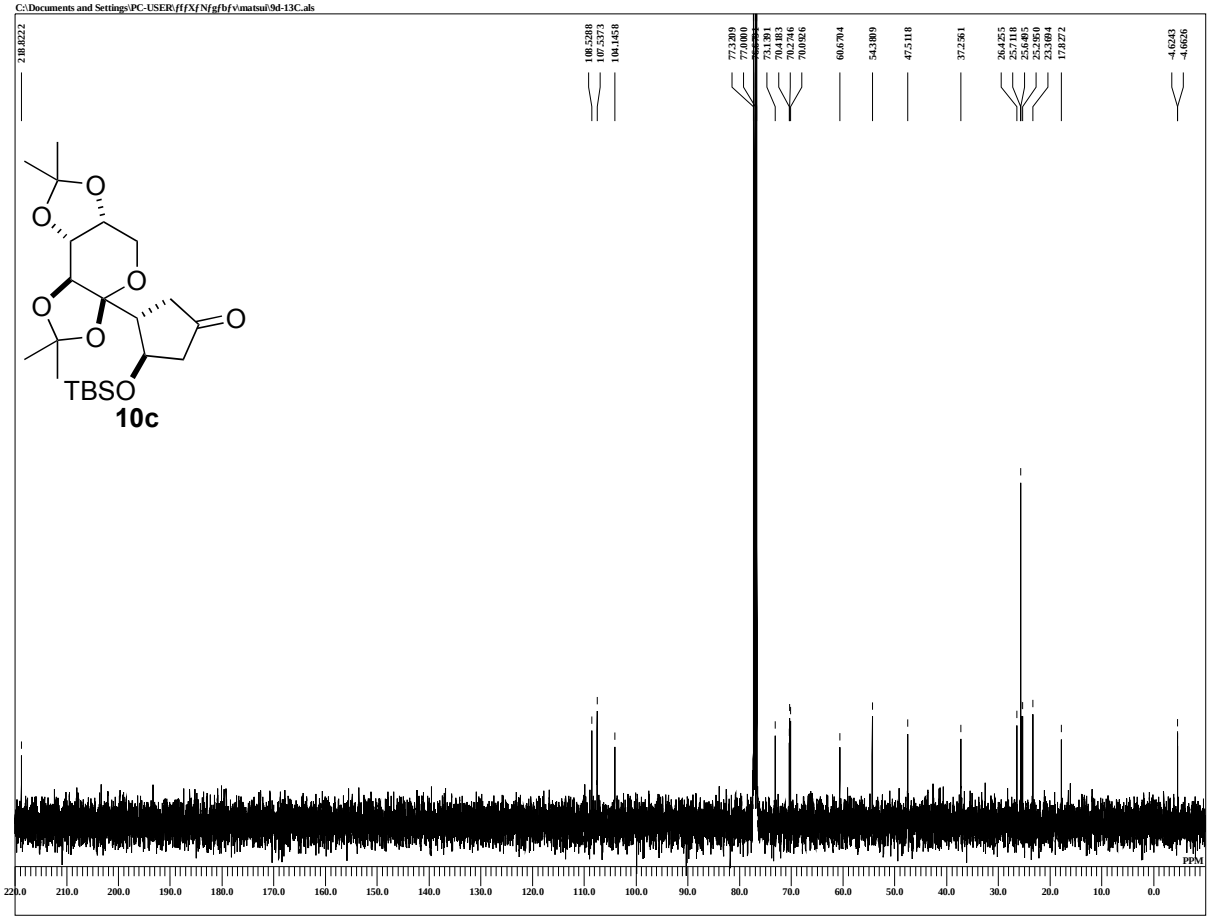
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\9d-1H.als



DFILE 9d-1H.als
COMNT YUM-2-153-fra16-18
DATIM 04-08-2014 12:24:56
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 42
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 9d-1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.3 c
SLVNT CDCL3
EXREF 7.26 ppm

YUM-2-153-fra16-18

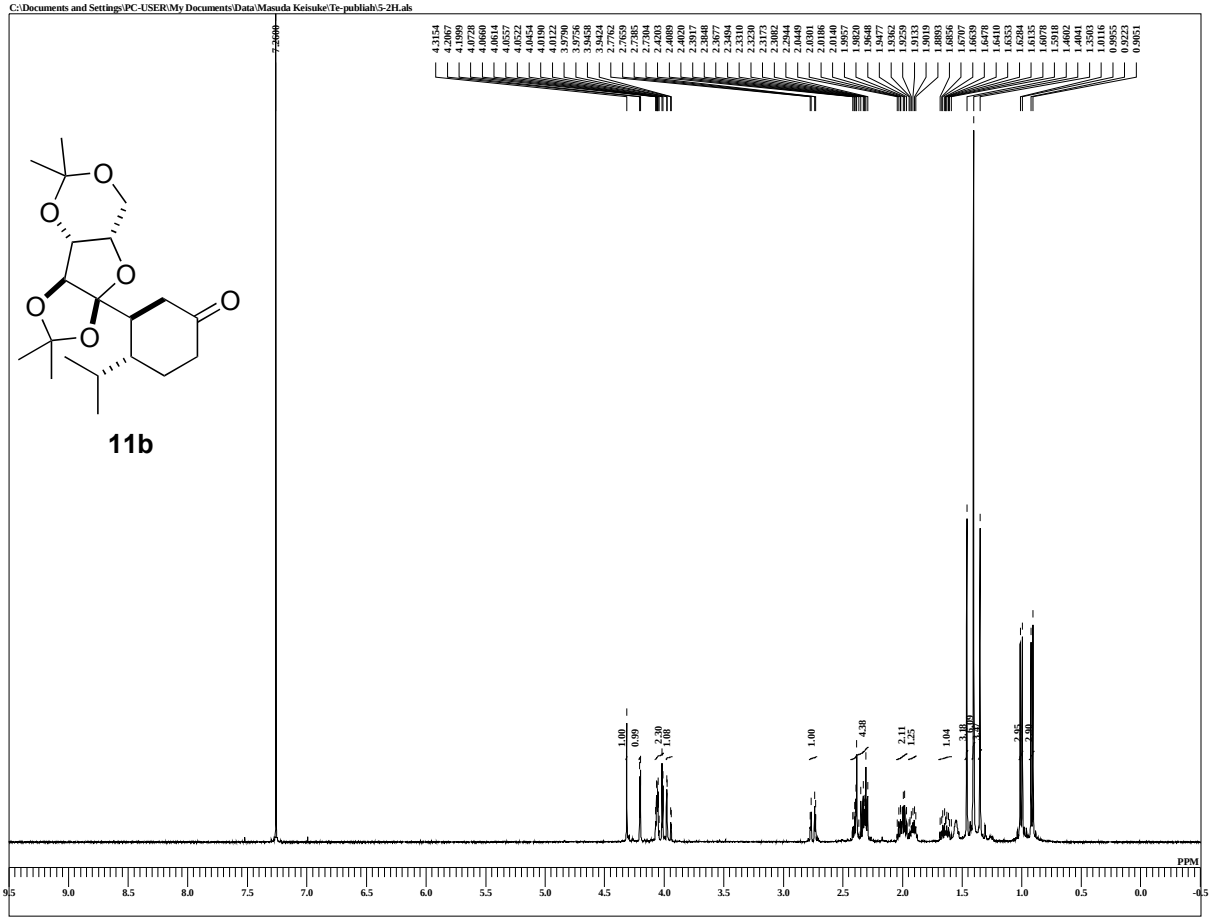
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\9d-13C.als



DFILE 9d-13C.als
COMNT YUM-2-153-fra16-18
DATIM 04-08-2014 12:37:14
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 52428
SPO 52428
TIMES 229
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 229
ADBIT 16
RGAIN 42
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 9d-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.6 c
SLVNT CDCL3
EXREF 77.00 ppm

KM-11-127-purify

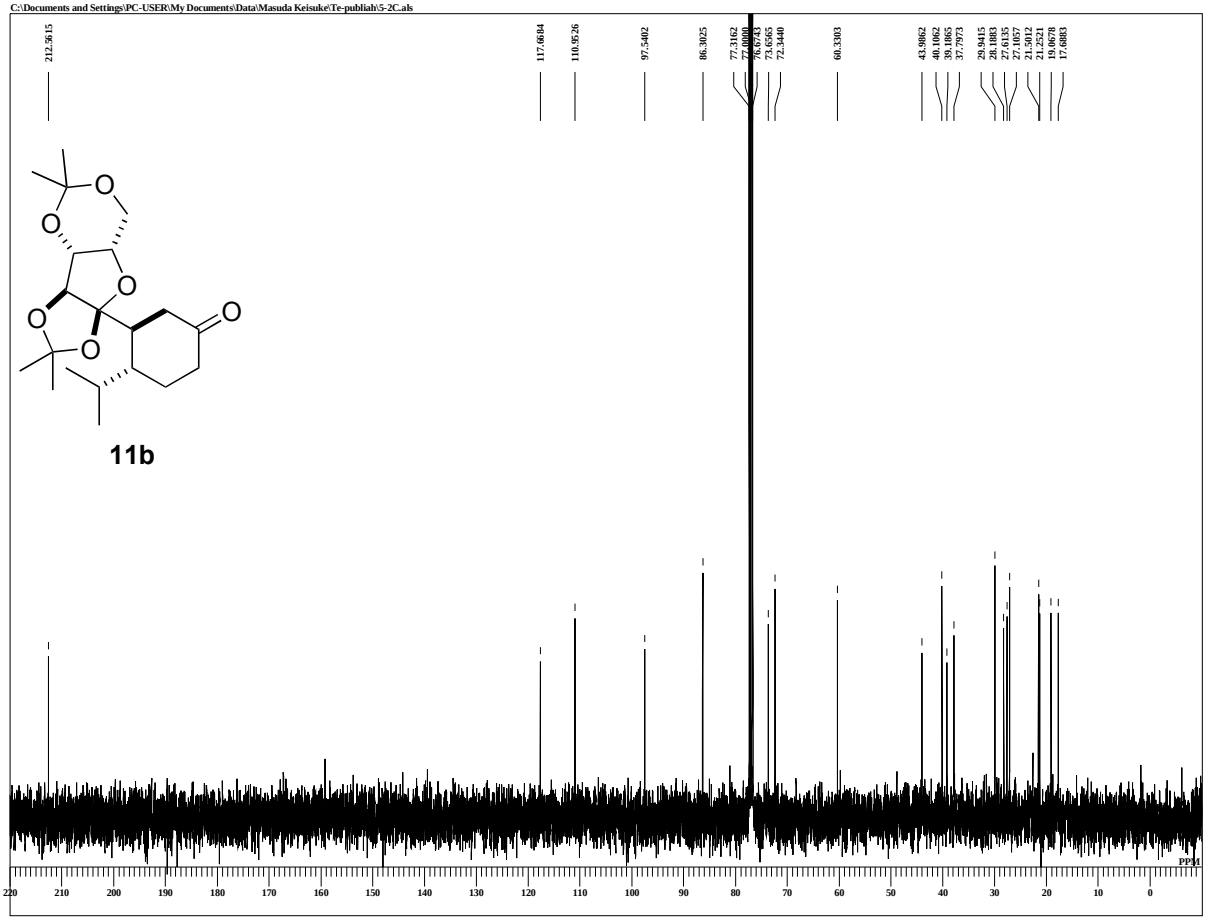
C:\Documents and Settings\PC-USER\My Documents\Data\Masuda Keisuke\Te-publiah\5-2H.als




```
DFILE    5-2H.als
COMNT    KM-11-127-purify
DATIM    29-01-2015 08:30:52
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.56 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    13107
SPO      13107
TIMES    8
DUMMY    1
FREQU    5938.15 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    48
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    5-2H.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    23.3 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

KM-11-125-20-25-C

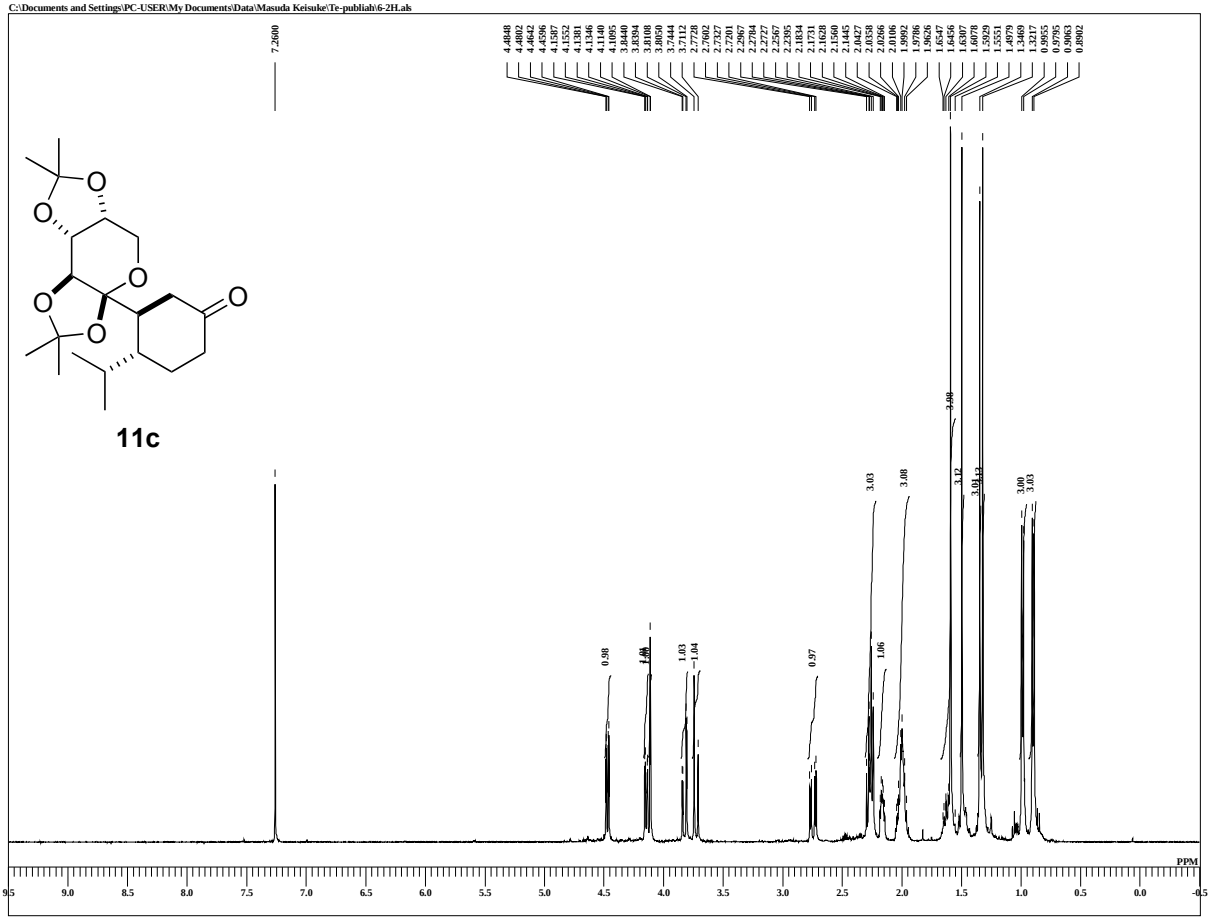
C:\Documents and Settings\PC-USER\My Documents\Data\Masuda Keisuke\Te-publiah\5-2C.als




```
DFILE    5-2C.als
COMNT    KM-11-125-20-25-C
DATIM    28-01-2015 14:55:02
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.07 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    26214
SPO      26214
TIMES    63
DUMMY    4
FREQU    24999.62 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    63
ADBIT    16
RGAIN    56
BF       1.00 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    5-2C.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    22.7 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

KM-11-121-fr16

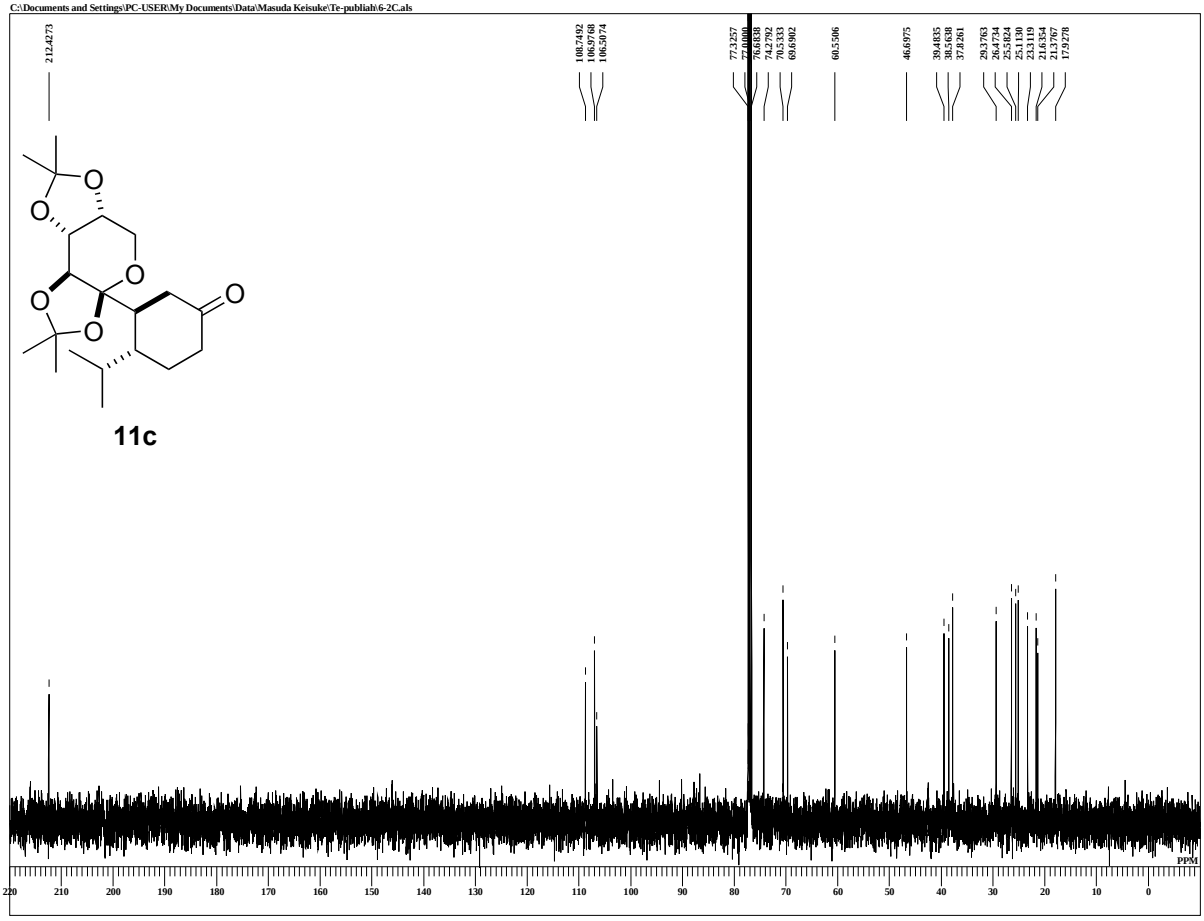
C:\Documents and Settings\PC-USER\My Documents\Data\Masuda Keisuke\Te-publiah\6-2H.als




```
DFILE    6-2H.als
COMNT    KM-11-121-fr16
DATIM    28-01-2015 14:33:02
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.56 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    13107
SPO      13107
TIMES    8
DUMMY    1
FREQU    5938.15 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    38
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    6-2H.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    22.7 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

KM-11-121-fr16-C

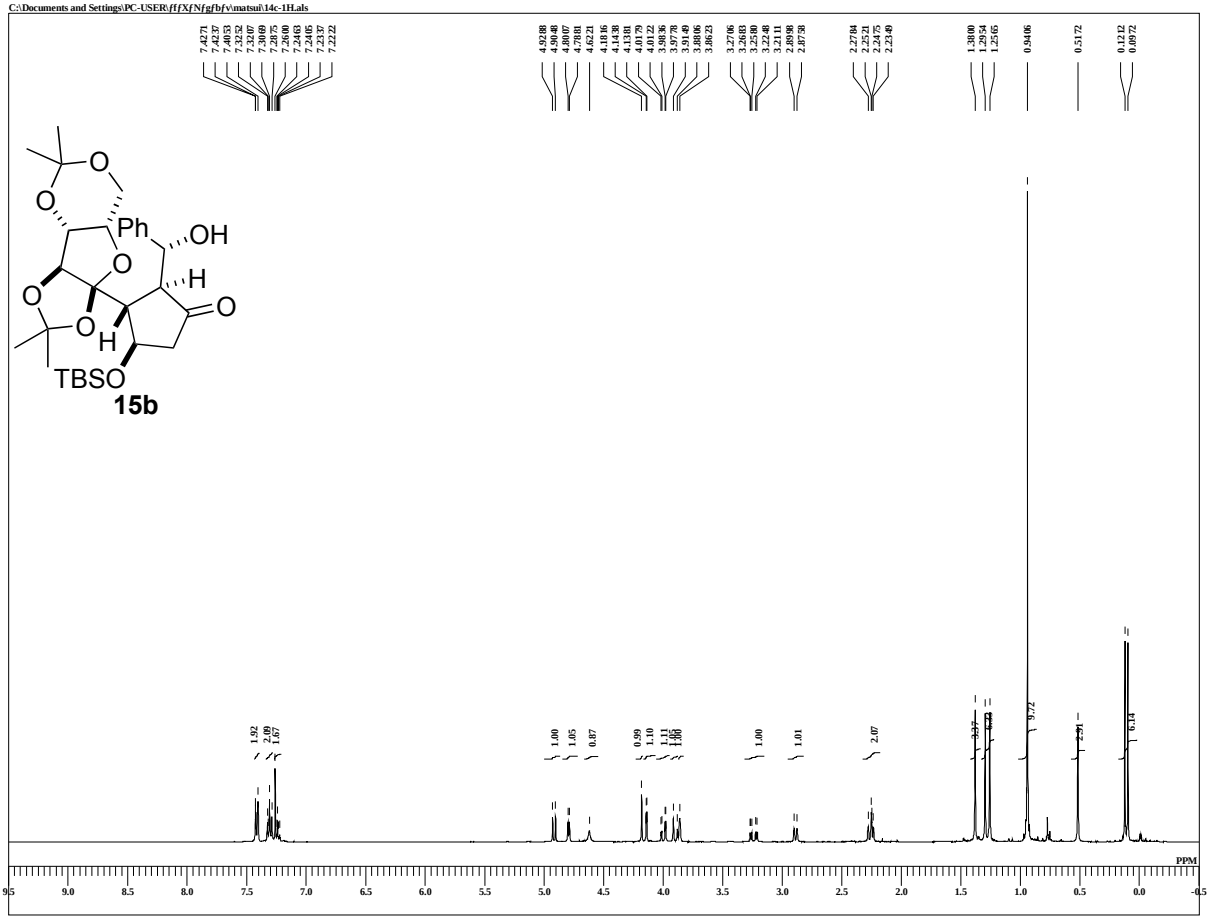
C:\Documents and Settings\PC-USER\My Documents\Data\Masuda Keisuke\Te-publiah\6-2C.als




```
DFILE    6-2C.als
COMNT    KM-11-121-fr16-C
DATIM    28-01-2015 14:39:35
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.07 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    26214
SPO      26214
TIMES    111
DUMMY    4
FREQU    24999.62 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    111
ADBIT    16
RGAIN    54
BF       1.00 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    6-2C.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    22.8 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

single_pulse

C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\14c-1H.als

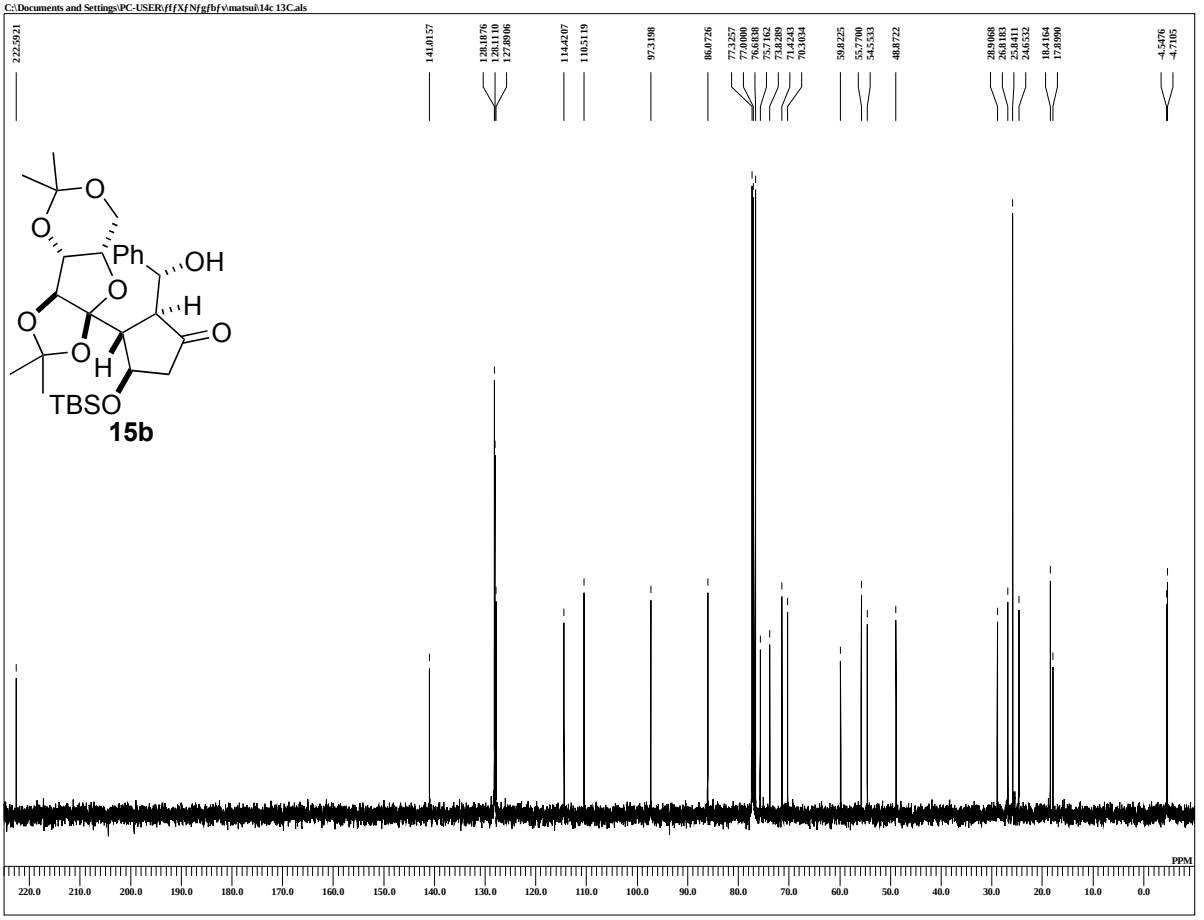


15b

```
DFILE    14c-1H.als
COMNT    single_pulse
DATIM    16-11-2013 16:16:25
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET      6.28 KHz
OBFIN      0.87 Hz
PW1        6.44 usec
DEADT      0.00 usec
PREDL      0.00000 msec
IWT        1.0000 sec
POINT      13107
SPO        13107
TIMES      8
DUMMY      1
FREQU      5938.15 Hz
FLT        30000 Hz
DELAY      16.68 usec
ACQTM      2.2073 sec
PD         2.0000 sec
SCANS      8
ADBIT      16
RGAIN      26
BF         0.10 Hz
T1         0.00
T2         0.00
T3         100.00
T4         100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET      6.28 KHz
IRFIN      0.87 Hz
IRRPW      115 usec
IRATN      79
DFILE    14c-1H.als
SF
LKSET      13.20 KHz
LKFIN      75.7 Hz
LKLEV      0
LGAIN      0
LKPHS      0
LKSIG      0
CSPED      0 Hz
FILDC
FILDF
CTEMP      23.1 c
SLVNT    CDCL3
EXREF      7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\14c 13C.als

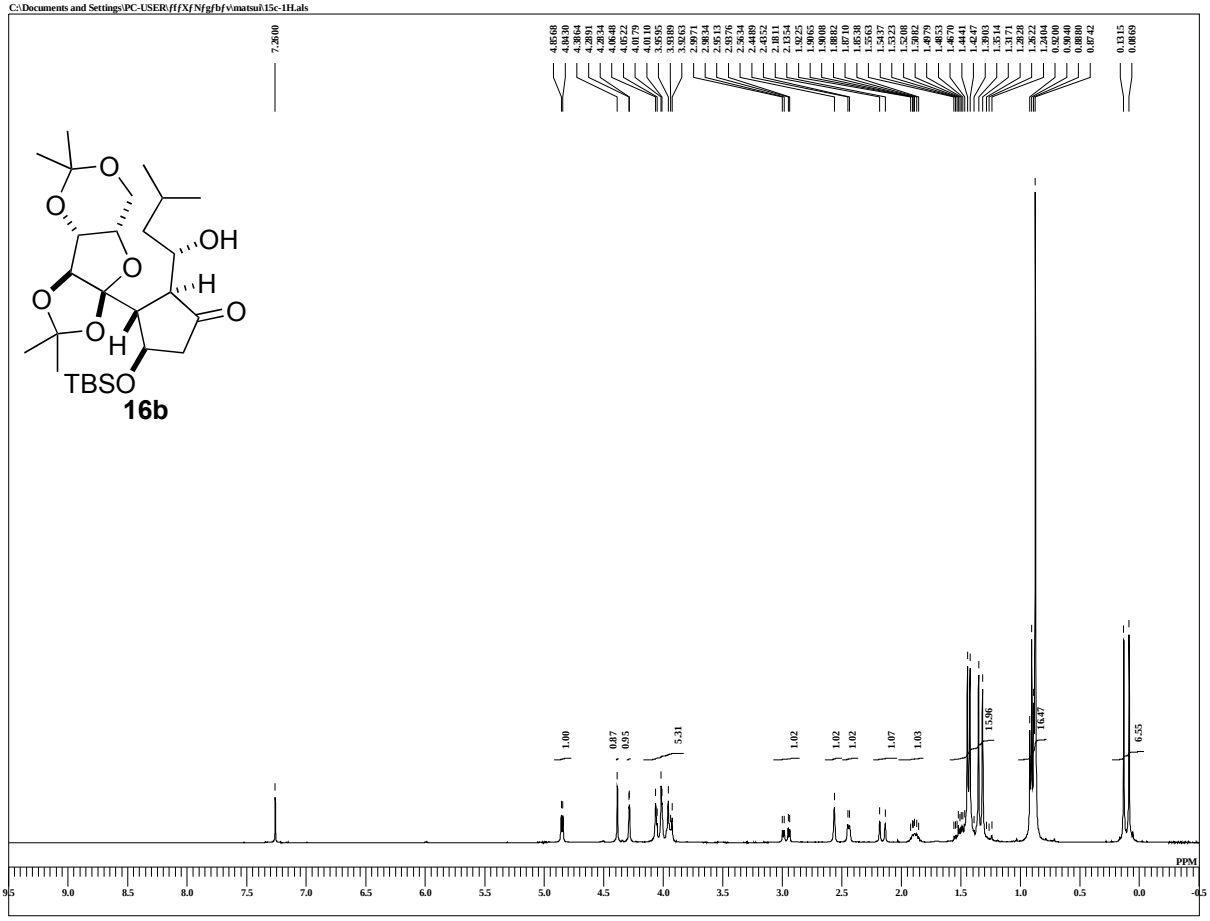


15b

```
DFILE    14c 13C.als
COMNT    single pulse decoupled gat
DATIM    16-11-2013 16:20:52
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET      5.13 KHz
OBFIN      0.98 Hz
PW1        3.03 usec
DEADT      0.00 usec
PREDL      0.00000 msec
IWT        1.0000 sec
POINT      26214
SPO        26214
TIMES      76
DUMMY      4
FREQU      24999.62 Hz
FLT        125000 Hz
DELAY      20.50 usec
ACQTM      1.0486 sec
PD         2.0000 sec
SCANS      76
ADBIT      16
RGAIN      60
BF         1.00 Hz
T1         0.00
T2         0.00
T3         100.00
T4         100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET      6.28 KHz
IRFIN      0.87 Hz
IRRPW      115 usec
IRATN      79
DFILE    14c 13C.als
SF
LKSET      13.20 KHz
LKFIN      75.7 Hz
LKLEV      0
LGAIN      0
LKPHS      0
LKSIG      0
CSPED      0 Hz
FILDC
FILDF
CTEMP      23.2 c
SLVNT    CDCL3
EXREF      77.00 ppm
```

single_pulse

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\15c-1H.als

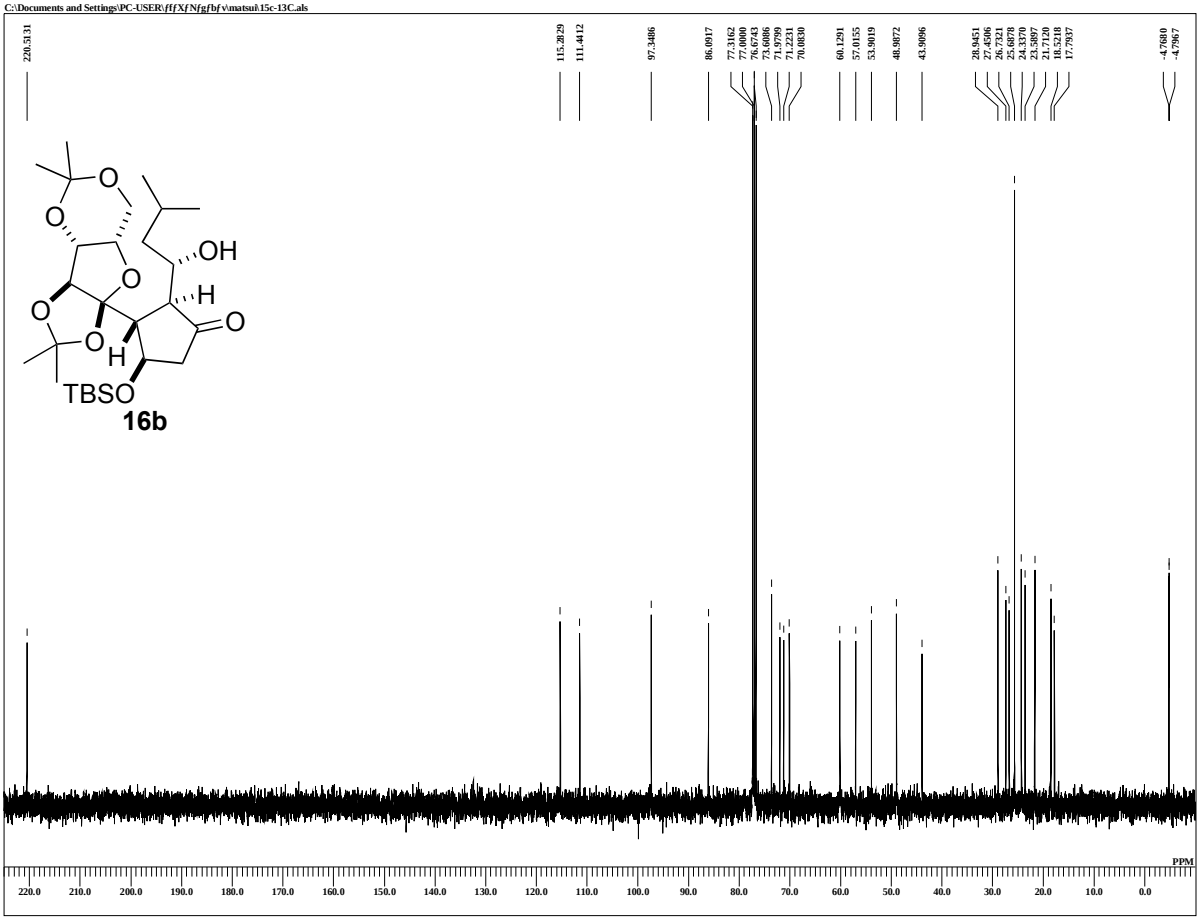


16b

```
DFILE    15c-1H.als
COMNT    single_pulse
DATIM    20-11-2013 11:00:19
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.44 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    13107
SPO      13107
TIMES    8
DUMMY    1
FREQU    5938.15 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    26
BF       0.10 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    15c-1H.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    460.0 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\15c-13C.als

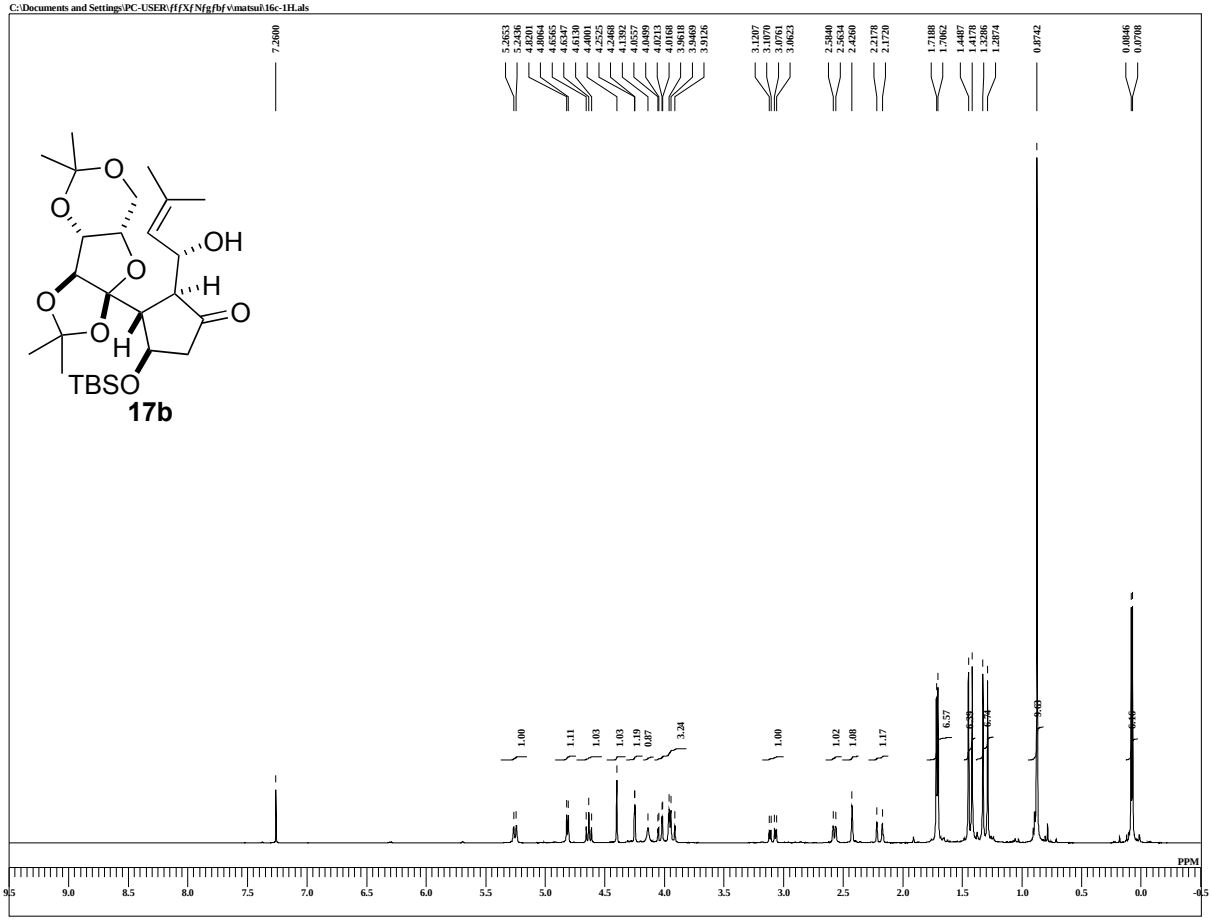


16b

```
DFILE    15c-13C.als
COMNT    single pulse decoupled gat
DATIM    20-11-2013 11:03:35
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.03 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    26214
SPO      26214
TIMES    51
DUMMY    4
FREQU    24999.62 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    51
ADBIT    16
RGAIN    60
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    15c-13C.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    460.0 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

single_pulse

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\16c-1H.als

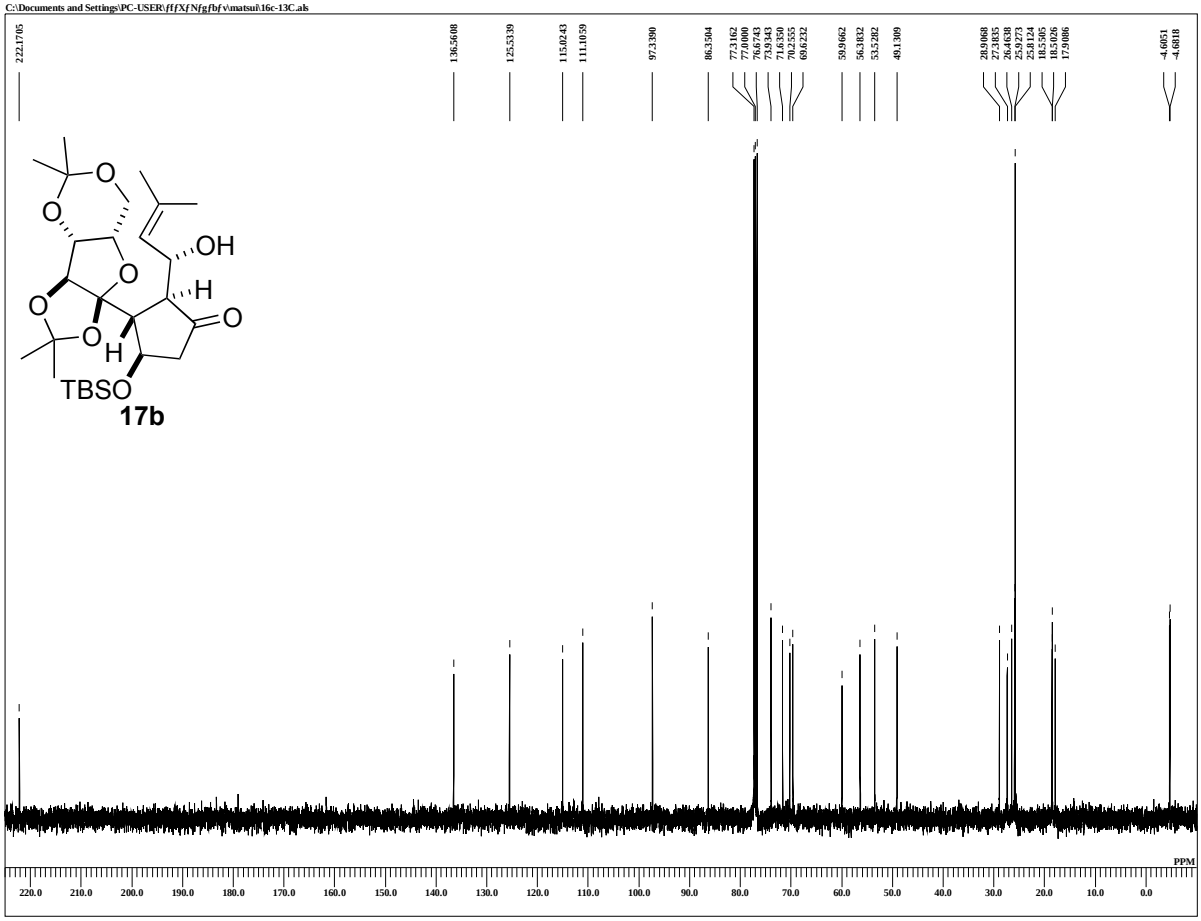


17b

DFILE 16c-1H.als
COMNT single_pulse
DATIM 20-11-2013 15:32:32
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 28
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 16c-1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.4 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\16c-13C.als

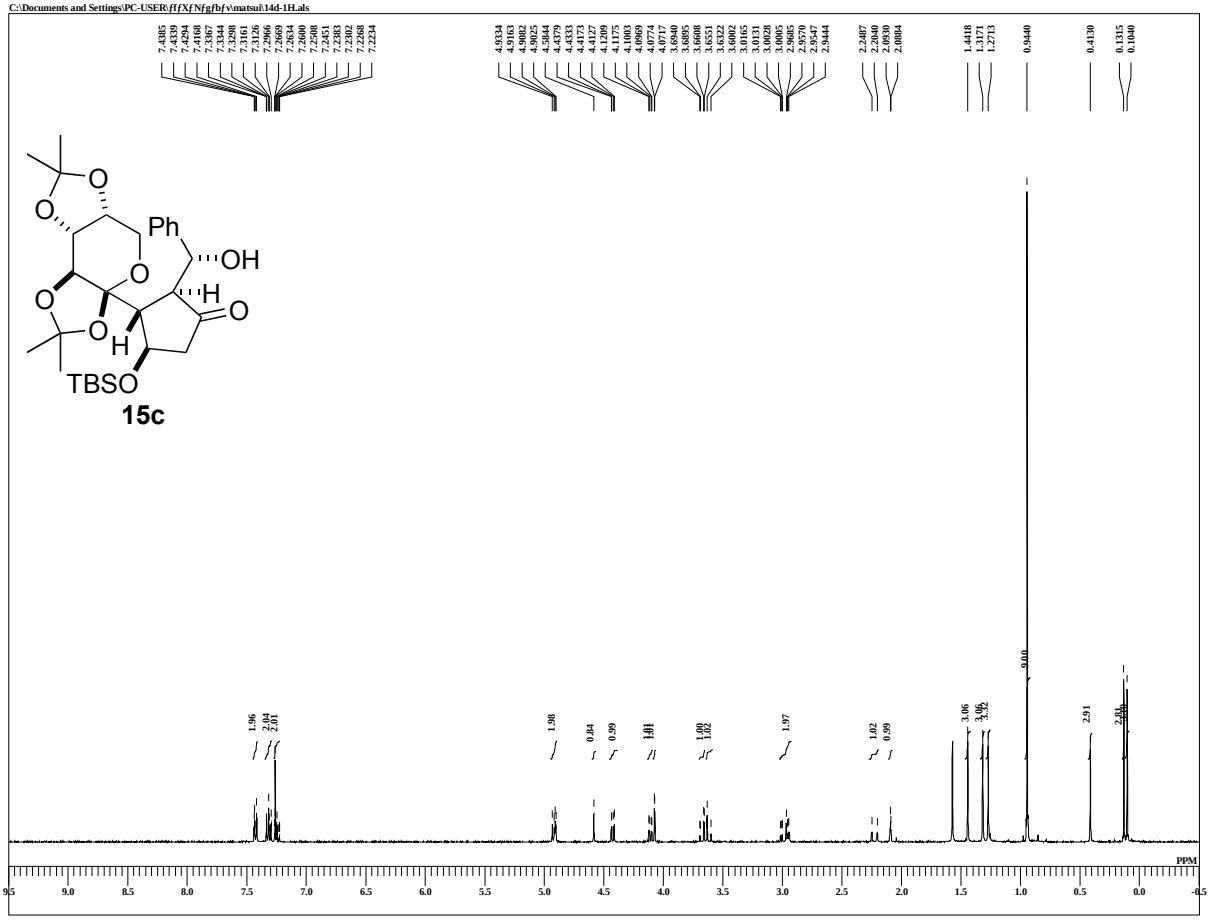


17b

DFILE 16c-13C.als
COMNT single pulse decoupled ga
DATIM 20-11-2013 11:23:07
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 57
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 57
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 16c-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 460.0 c
SLVNT CDCL3
EXREF 77.00 ppm

YUM-2-151-fra12

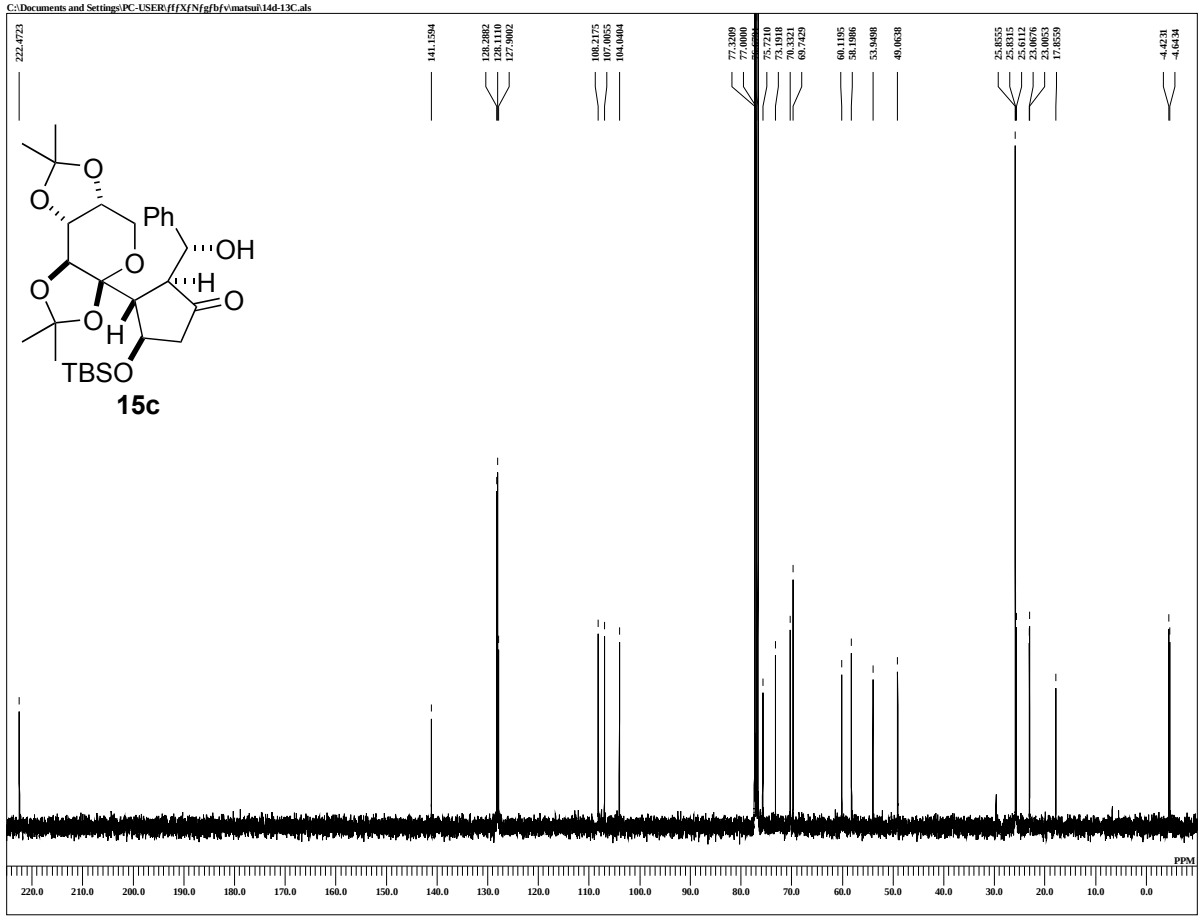
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\14d-1H.als




```
DFILE    14d-1H.als
COMNT    YUM-2-151-fra12
DATIM    29-07-2014 13:03:06
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.44 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    13107
SPO      13107
TIMES    8
DUMMY    1
FREQU    5938.15 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    42
BF       0.01 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    14d-1H.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    24.0 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

YUM-2-151-fra11-13-2

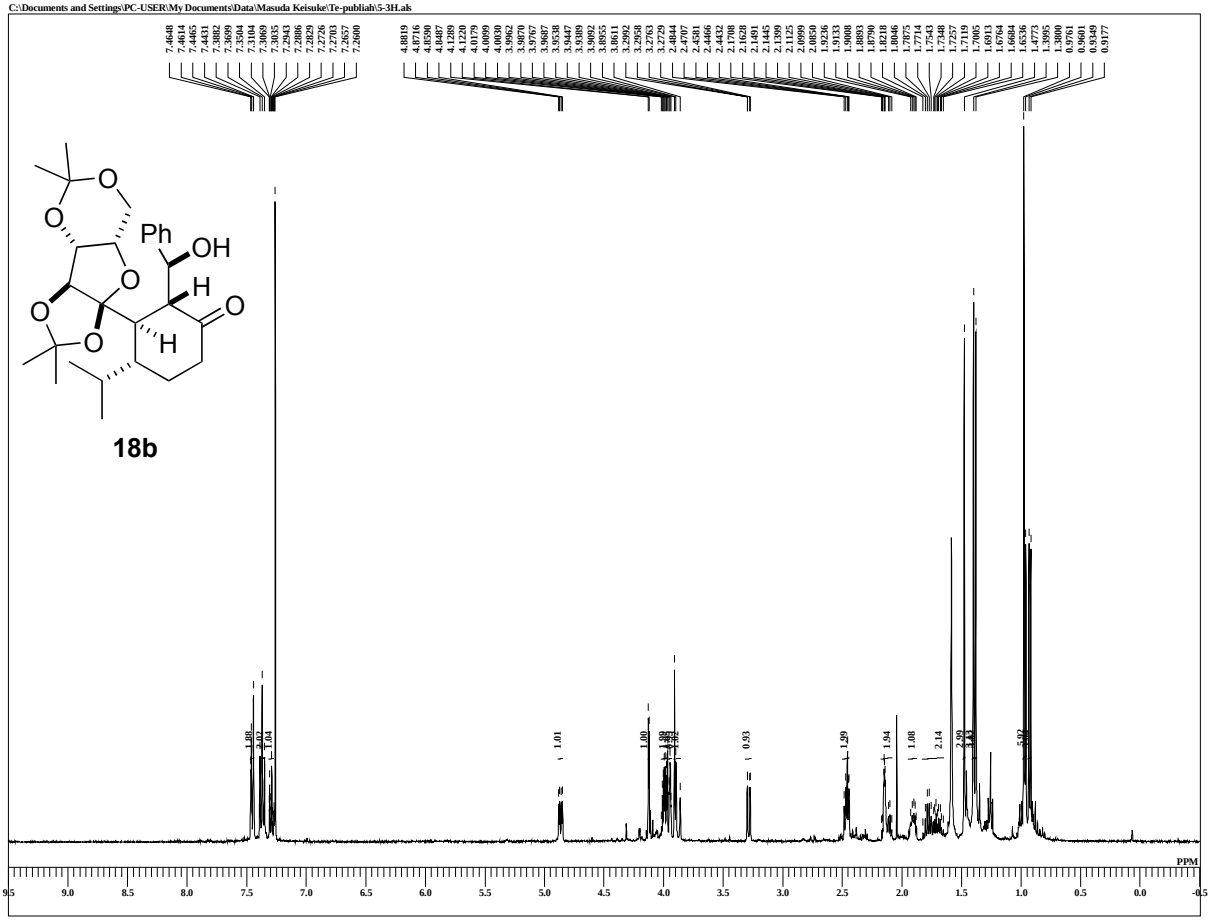
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\14d-13C.als




```
DFILE    14d-13C.als
COMNT    YUM-2-151-fra11-13-2
DATIM    29-07-2014 19:23:11
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.03 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    65536
SPO      65536
TIMES    202
DUMMY    4
FREQU    31250.00 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    202
ADBIT    16
RGAIN    32
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    14d-13C.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    24.4 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

KM-11-135-PTLC

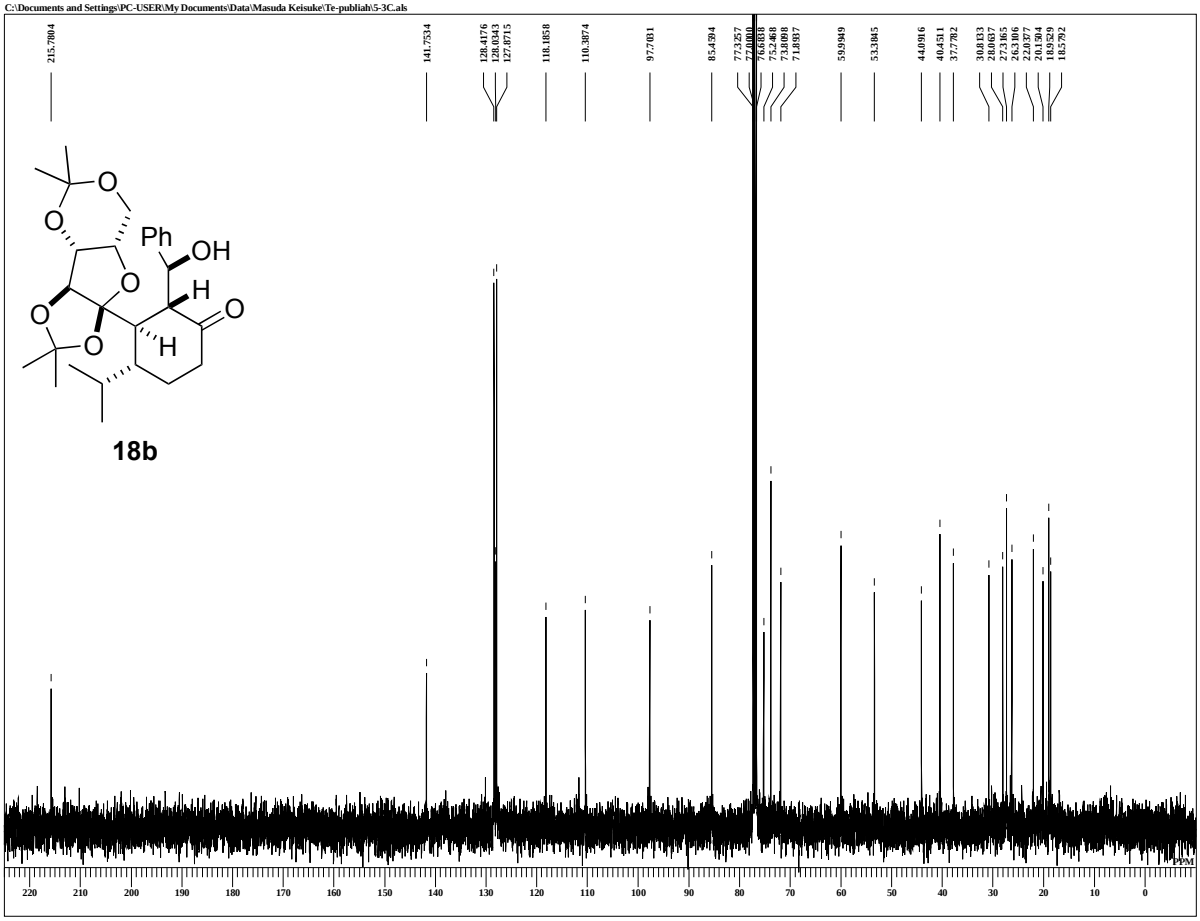
C:\Documents and Settings\PC-USER\My Documents\Data\Masuda Keisuke\Te-publiah\5-3H.als



DFILE 5-3H.als
COMNT KM-11-135-PTLC
DATIM 30-01-2015 07:12:28
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.56 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 42
BF 0.10 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 5-3H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 22.7 c
SLVNT CDCL3
EXREF 7.26 ppm

KM-11-131-purify-C

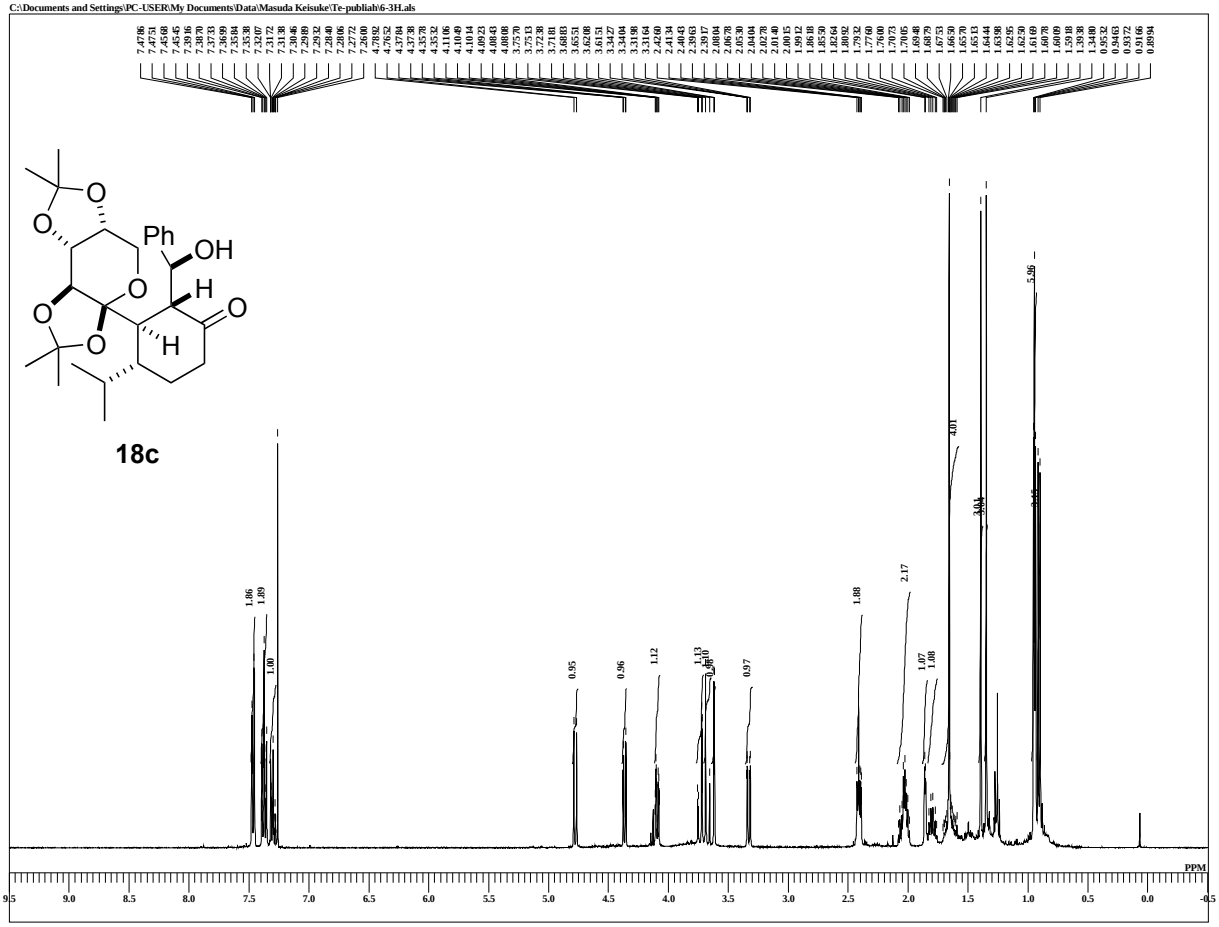
C:\Documents and Settings\PC-USER\My Documents\Data\Masuda Keisuke\Te-publiah\5-3C.als



DFILE 5-3C.als
COMNT KM-11-131-purify-C
DATIM 29-01-2015 08:50:38
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.07 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 169
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 169
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE 5-3C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm

KM-9-153-fr14-26

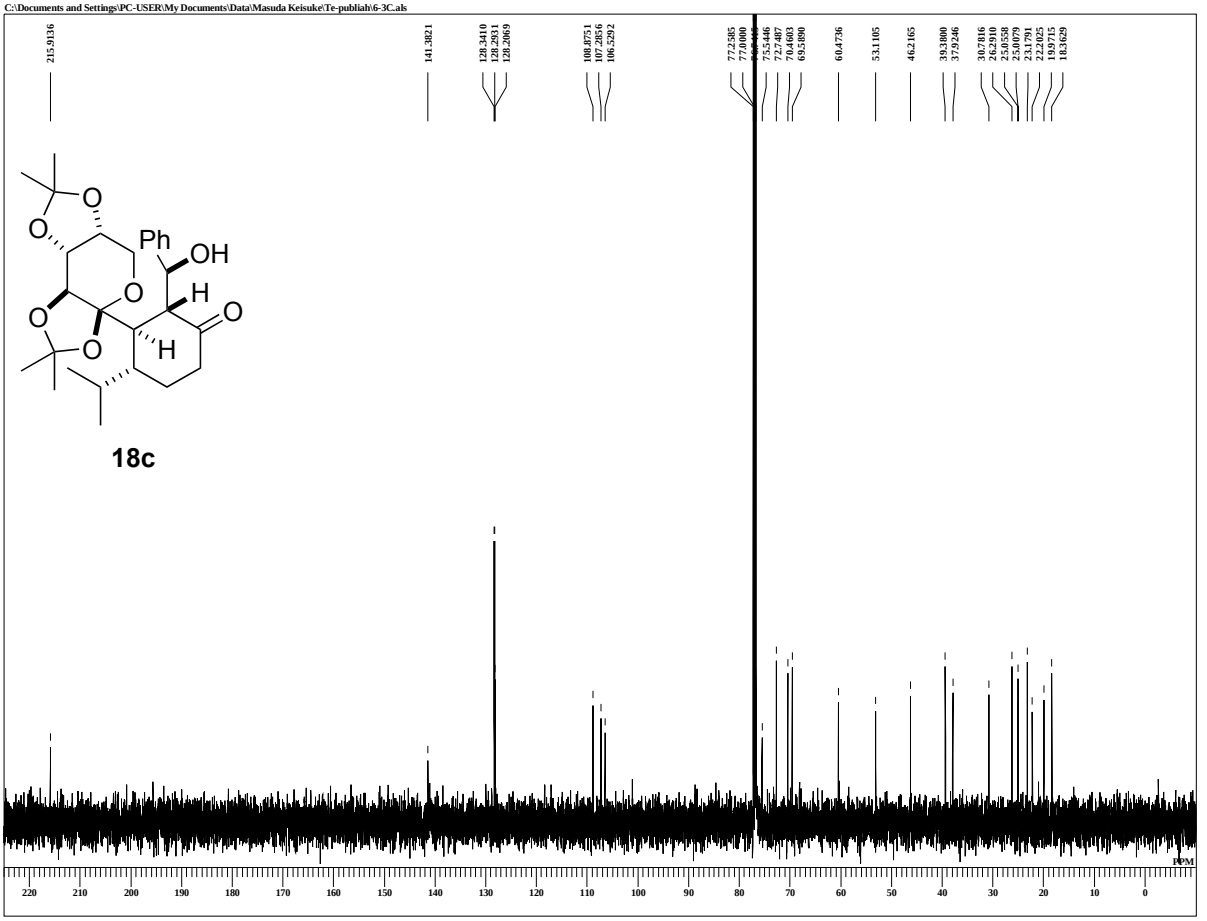
C:\Documents and Settings\PC-USER\My Documents\Data\Masuda Keisuke\Te-publiah\6-3H.als




```
DFILE    6-3H.als
COMNT    KM-9-153-fr14-26
DATIM    02-09-2014 19:29:29
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.44 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    13107
SPO      13107
TIMES    8
DUMMY    1
FREQU    5938.15 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    38
BF       0.10 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    6-3H.als
SF
LKSET    13.20 KHz
LKFIN    75.7 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    24.8 c
SLVNT    CDCL3
EXREF    7.26 ppm
```

KM-9-153-fr14-26-C

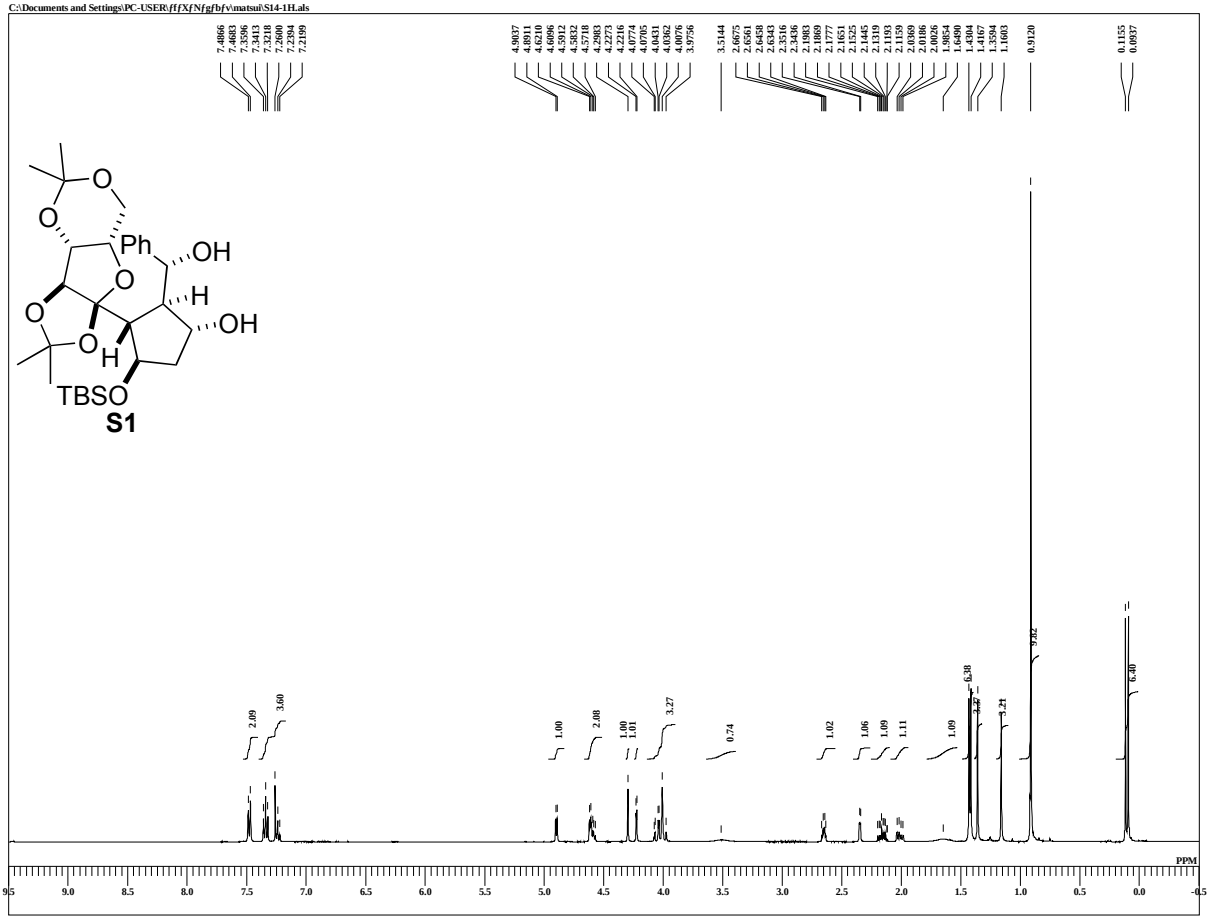
C:\Documents and Settings\PC-USER\My Documents\Data\Masuda Keisuke\Te-publiah\6-3C.als




```
DFILE    6-3C.als
COMNT    KM-9-153-fr14-26-C
DATIM    02-09-2014 17:26:26
MENUF
OBNUC    13C
OFR      124.51 MHz
OBFRQ    124.51 MHz
OBSET    3.45 KHz
OBFIN    6.00 Hz
PW1      3.70 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    26214
SPO      26214
TIMES    135
DUMMY    4
FREQU    31249.52 Hz
FLT      157000 Hz
DELAY    20.80 usec
ACQTM    0.8389 sec
PD       2.0000 sec
SCANS    135
ADBIT    16
RGAIN    56
BF       1.00 Hz
T1       0.00
T2       0.00
T3       90.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      495.13 MHz
IRSET    4.38 KHz
IRFIN    9.64 Hz
IRRPW    92 usec
IRATN    79
DFILE    6-3C.als
SF
LKSET    748.40 KHz
LKFIN    98.2 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    26.5 c
SLVNT    CDCL3
EXREF    77.00 ppm
```

single_pulse

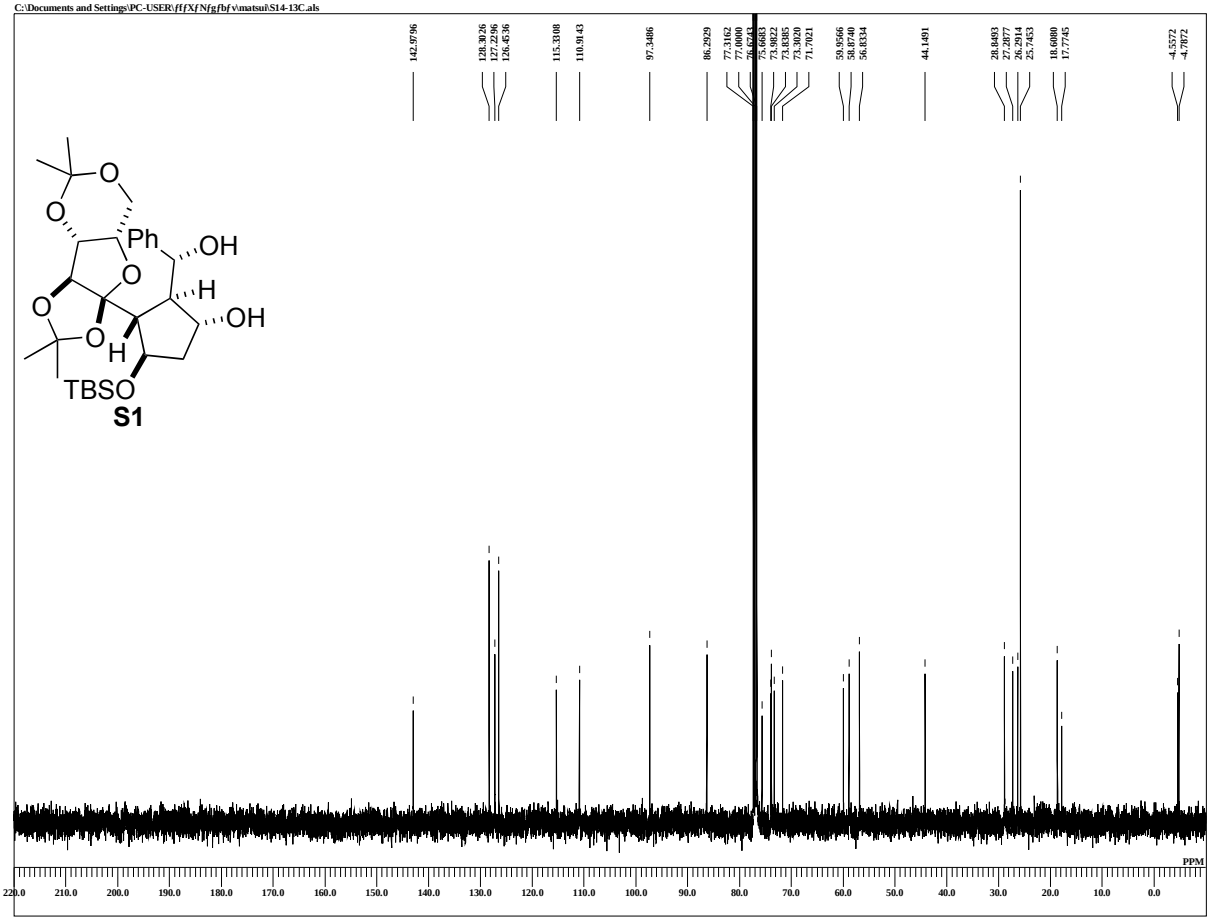
C:\Documents and Settings\PC-USER\ffXfNfgfbf\v\matsui\S14-1H.als




```
DFILE   S14-1H.als
COMNT   single_pulse
DATIM   25-11-2013 10:01:49
MENUF
OBNUC   1H
OFR     395.88 MHz
OBFRQ   395.88 MHz
OBSET     6.28 KHz
OBFIN     0.87 Hz
PW1       6.44 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT    13107
SPO      13107
TIMES        8
DUMMY        1
FREQU     5938.15 Hz
FLT      30000 Hz
DELAY     16.68 usec
ACQTM     2.2073 sec
PD        2.0000 sec
SCANS        8
ADBIT       16
RGAIN       34
BF        0.10 Hz
T1        0.00
T2        0.00
T3      100.00
T4      100.00
EXMOD   single_pulse.ex2
EXPCM
IRNUC   1H
IFR     395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN       79
DFILE   S14-1H.als
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED     0 Hz
FILDC
FILDF
CTEMP     21.1 c
SLVNT   CDCL3
EXREF     7.26 ppm
```

single pulse decoupled gated NOE

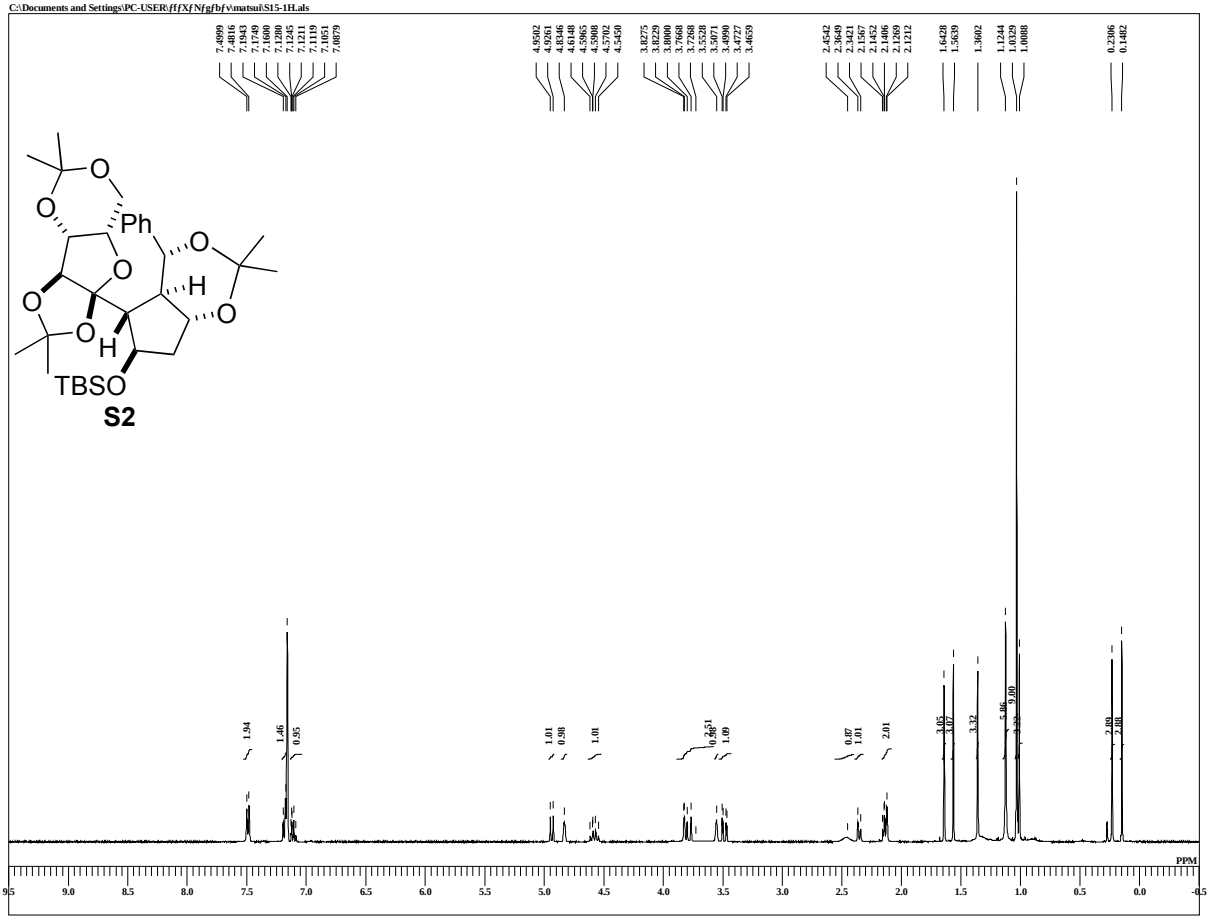
C:\Documents and Settings\PC-USER\ffXfNfgfbf\v\matsui\S14-13C.als




```
DFILE   S14-13C.als
COMNT   single pulse decoupled gat
DATIM   25-11-2013 10:08:07
MENUF
OBNUC   13C
OFR     99.55 MHz
OBFRQ   99.55 MHz
OBSET     5.13 KHz
OBFIN     0.98 Hz
PW1       3.03 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT    26214
SPO      26214
TIMES      112
DUMMY        4
FREQU    24999.62 Hz
FLT      125000 Hz
DELAY     20.50 usec
ACQTM     1.0486 sec
PD        2.0000 sec
SCANS      112
ADBIT       16
RGAIN       60
BF        1.00 Hz
T1        0.00
T2        0.00
T3      100.00
T4      100.00
EXMOD   single_pulse_dec
EXPCM
IRNUC   1H
IFR     395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN       79
DFILE   S14-13C.als
SF
LKSET     13.20 KHz
LKFIN     75.7 Hz
LKLEV        0
LGAIN        0
LKPHS        0
LKSIG        0
CSPED     0 Hz
FILDC
FILDF
CTEMP     21.5 c
SLVNT   CDCL3
EXREF     77.00 ppm
```

DK-6-004-f4-8

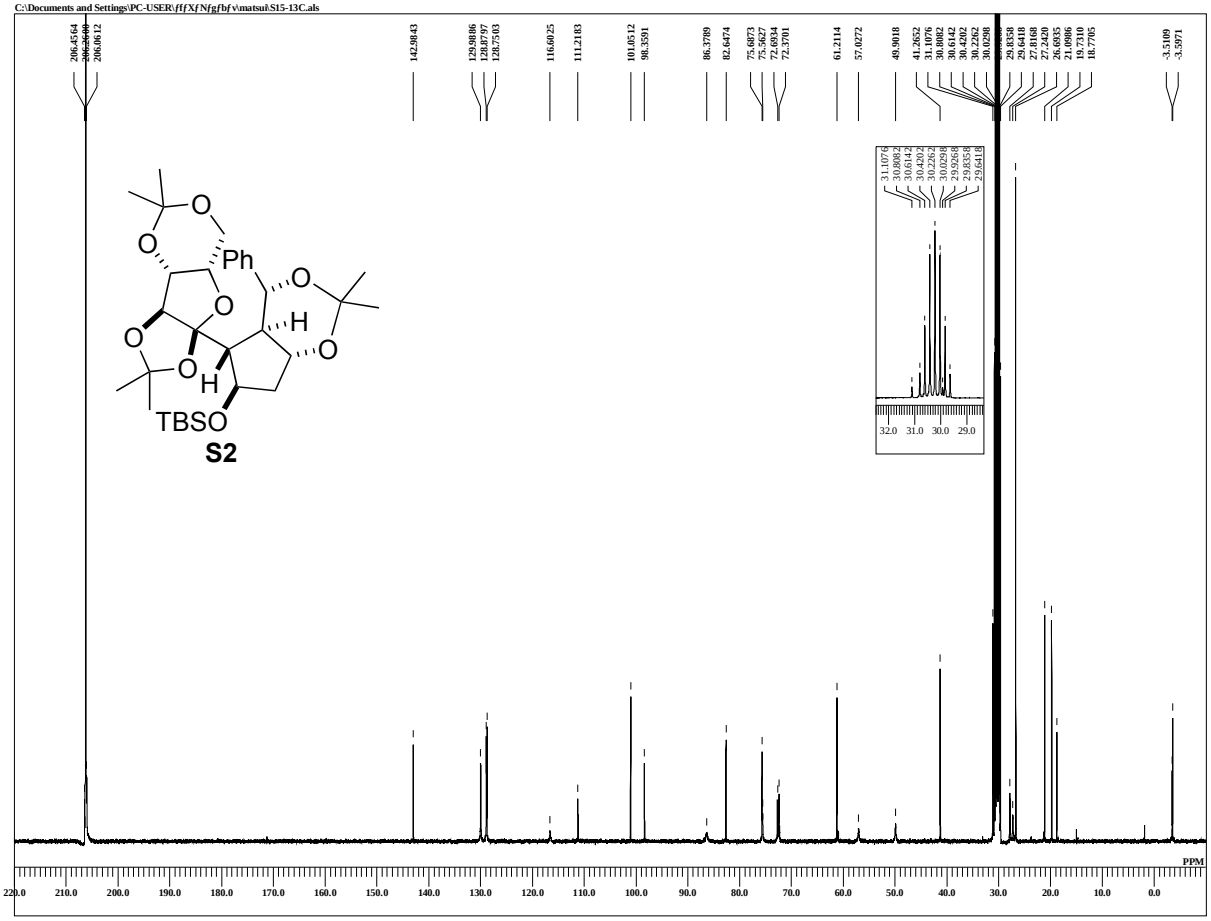
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\S15-1H.als



DFILE S15-1H.als
COMNT DK-6-004-f4-8
DATIM 17-12-2014 11:02:53
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.56 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 34
BF 0.01 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 147 usec
IRATN 79
DFILE S15-1H.als
SF
LKSET 13.20 KHz
LKFIN 69.6 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 70.0 c
SLVNT C6D6
EXREF 7.16 ppm

DK-6-004-f4-8

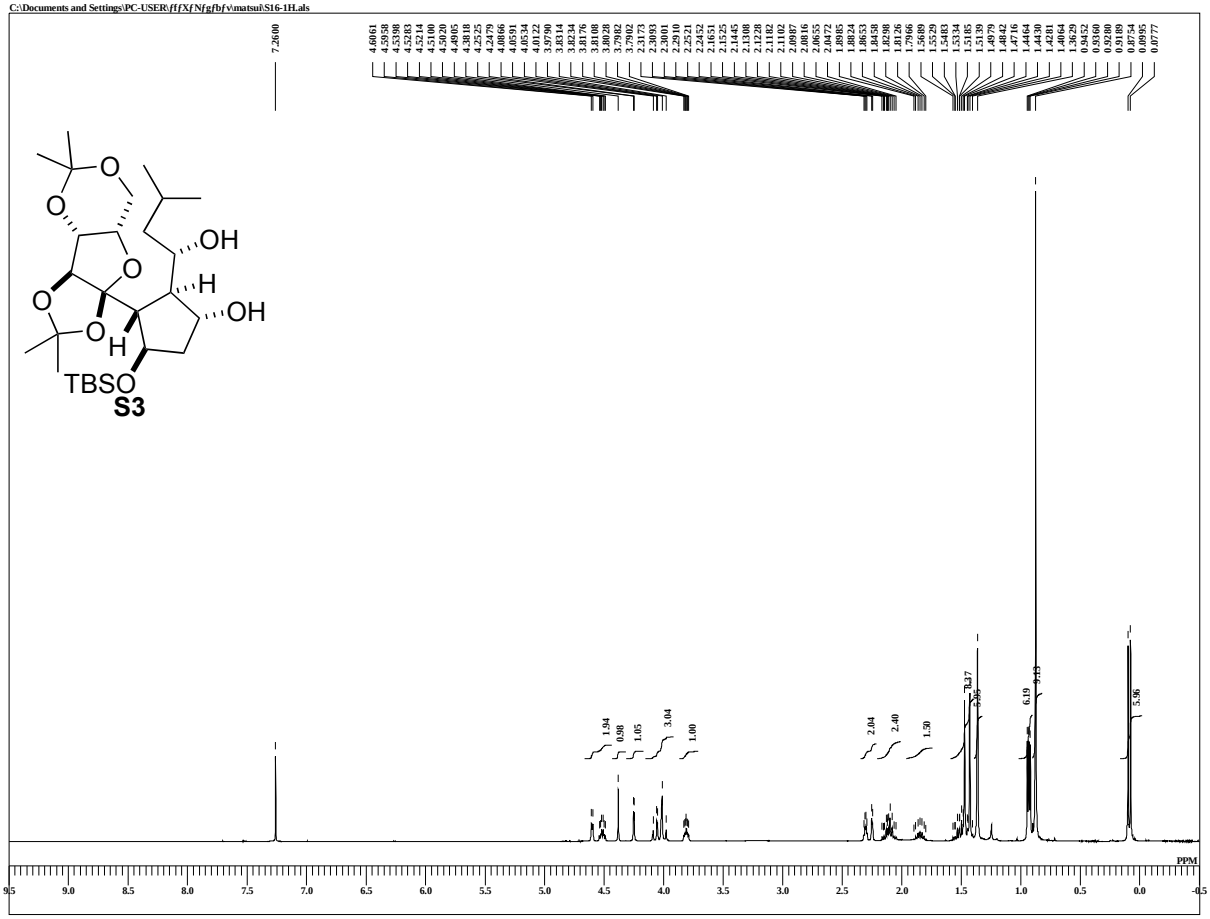
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\S15-13C.als



DFILE S15-13C.als
COMNT DK-6-004-f4-8
DATIM 18-12-2014 07:45:52
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.07 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 104856
SPO 104856
TIMES 10000
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 10000
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE S15-13C.als
SF
LKSET 12.90 KHz
LKFIN 59.1 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 50.0 c
SLVNT ACETN
EXREF 206.26 ppm

single_pulse

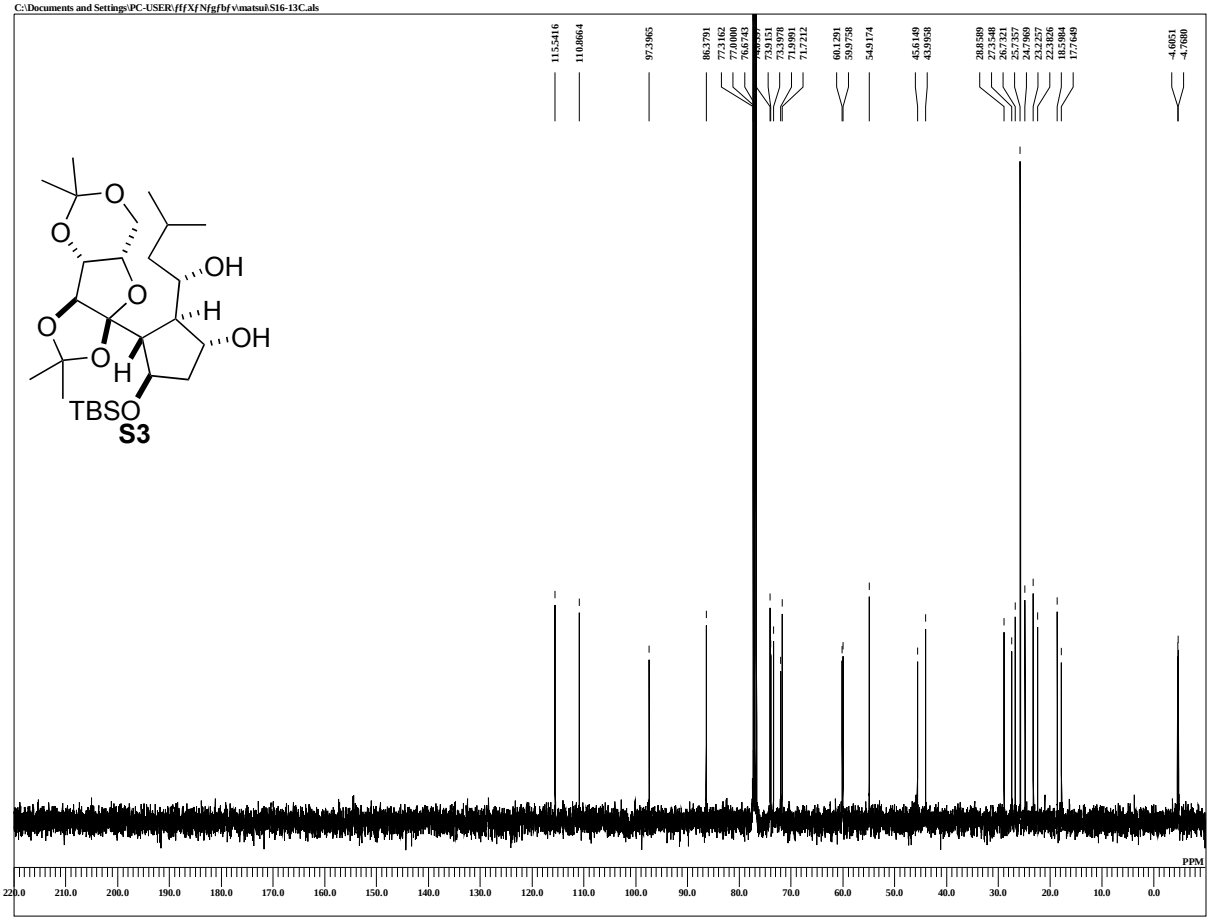
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\S16-1H.als



DFILE S16-1H.als
COMNT single_pulse
DATIM 14-02-2014 18:55:10
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 30
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE S16-1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.0 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

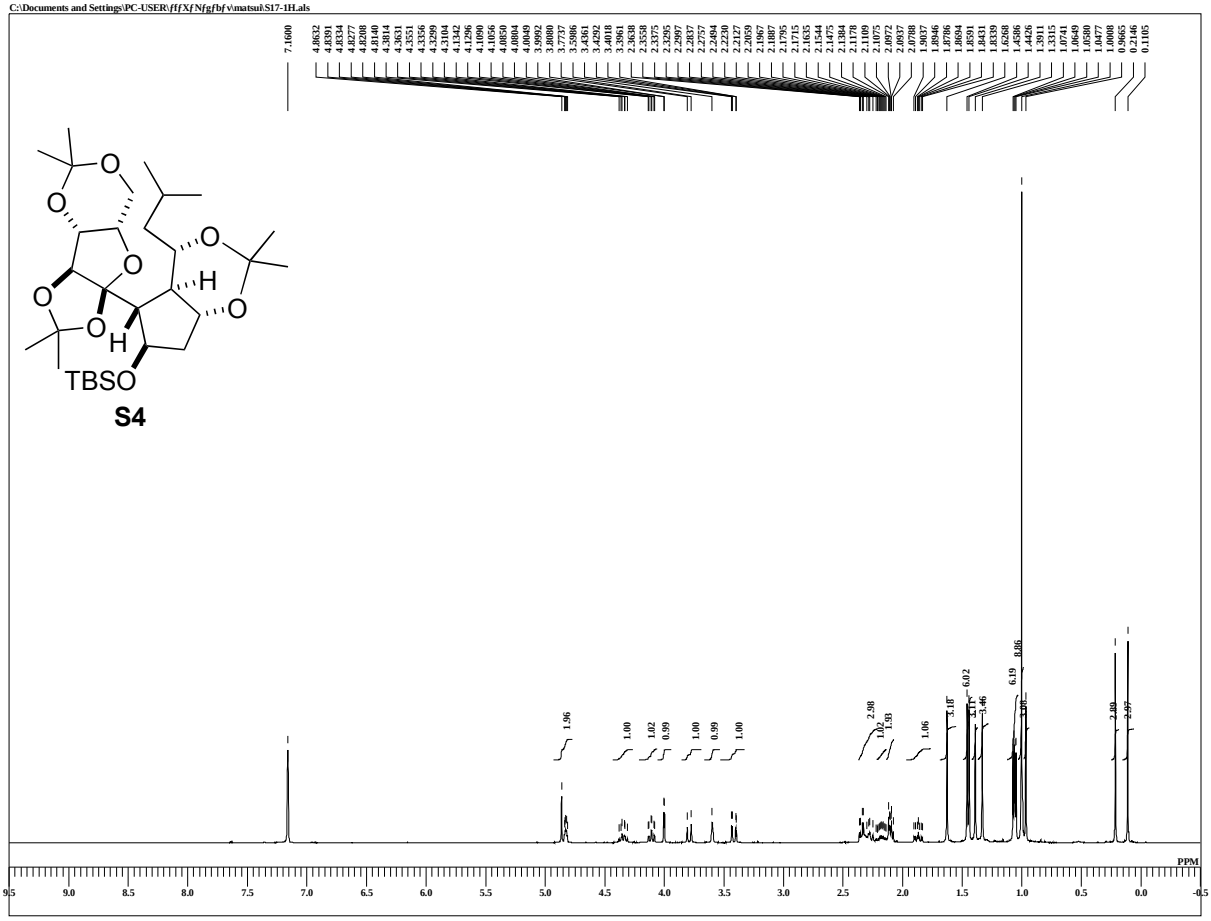
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\S16-13C.als



DFILE S16-13C.als
COMNT single pulse decoupled gat
DATIM 14-02-2014 16:19:39
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 101
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 101
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE S16-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm

single_pulse

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\S17-1H.als



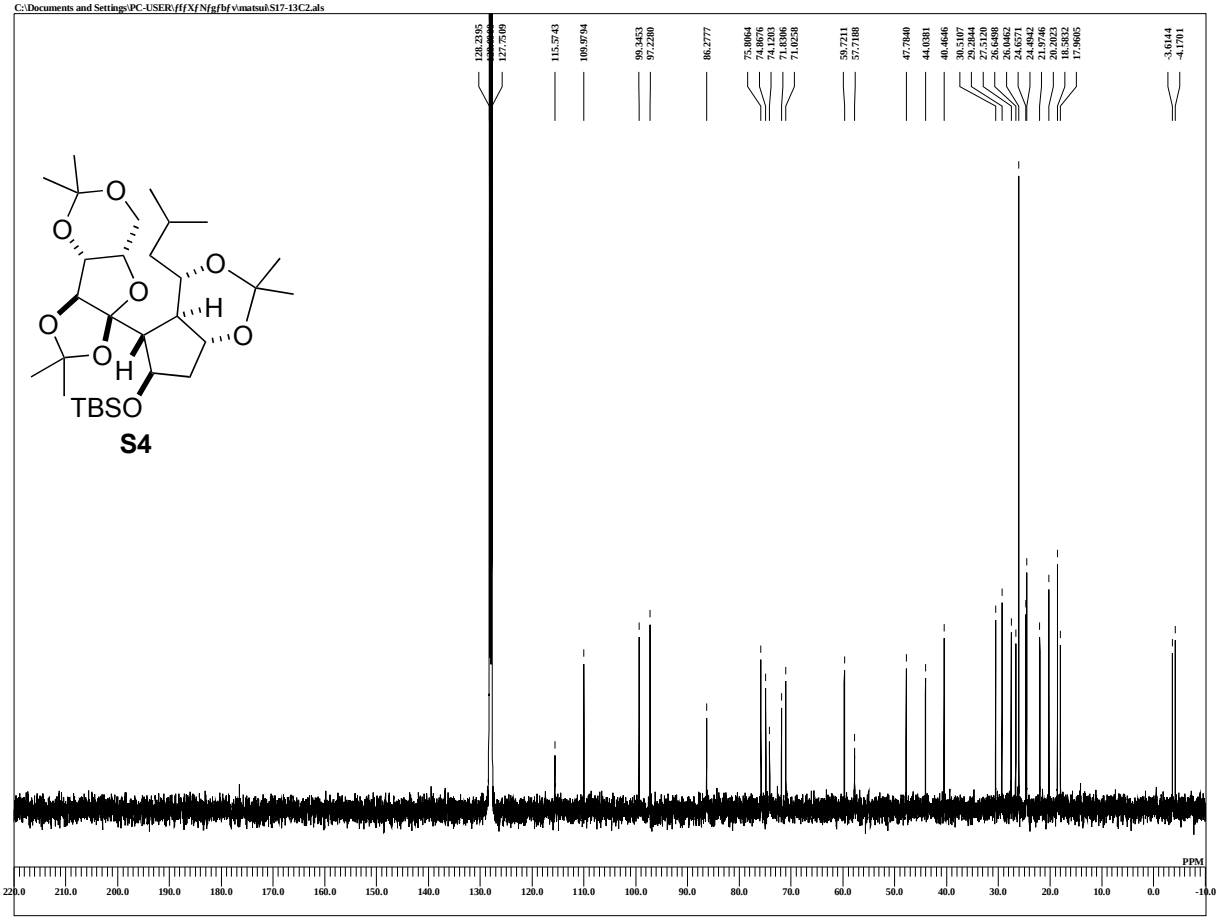
TBSO

S4

```
DFILE    S17-1H.als
COMNT    single_pulse
DATIM    18-02-2014 16:21:41
MENUF
OBNUC    1H
OFR      395.88 MHz
OBFRQ    395.88 MHz
OBSET    6.28 KHz
OBFIN    0.87 Hz
PW1      6.44 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    13107
SPO      13107
TIMES    8
DUMMY    1
FREQU    5938.15 Hz
FLT      30000 Hz
DELAY    16.68 usec
ACQTM    2.2073 sec
PD       2.0000 sec
SCANS    8
ADBIT    16
RGAIN    30
BF       0.10 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse.ex2
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    S17-1H.als
SF
LKSET    13.20 KHz
LKFIN    69.6 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    20.9 c
SLVNT    C6D6
EXREF    7.16 ppm
```

single pulse decoupled gated NOE

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\S17-13C2.als



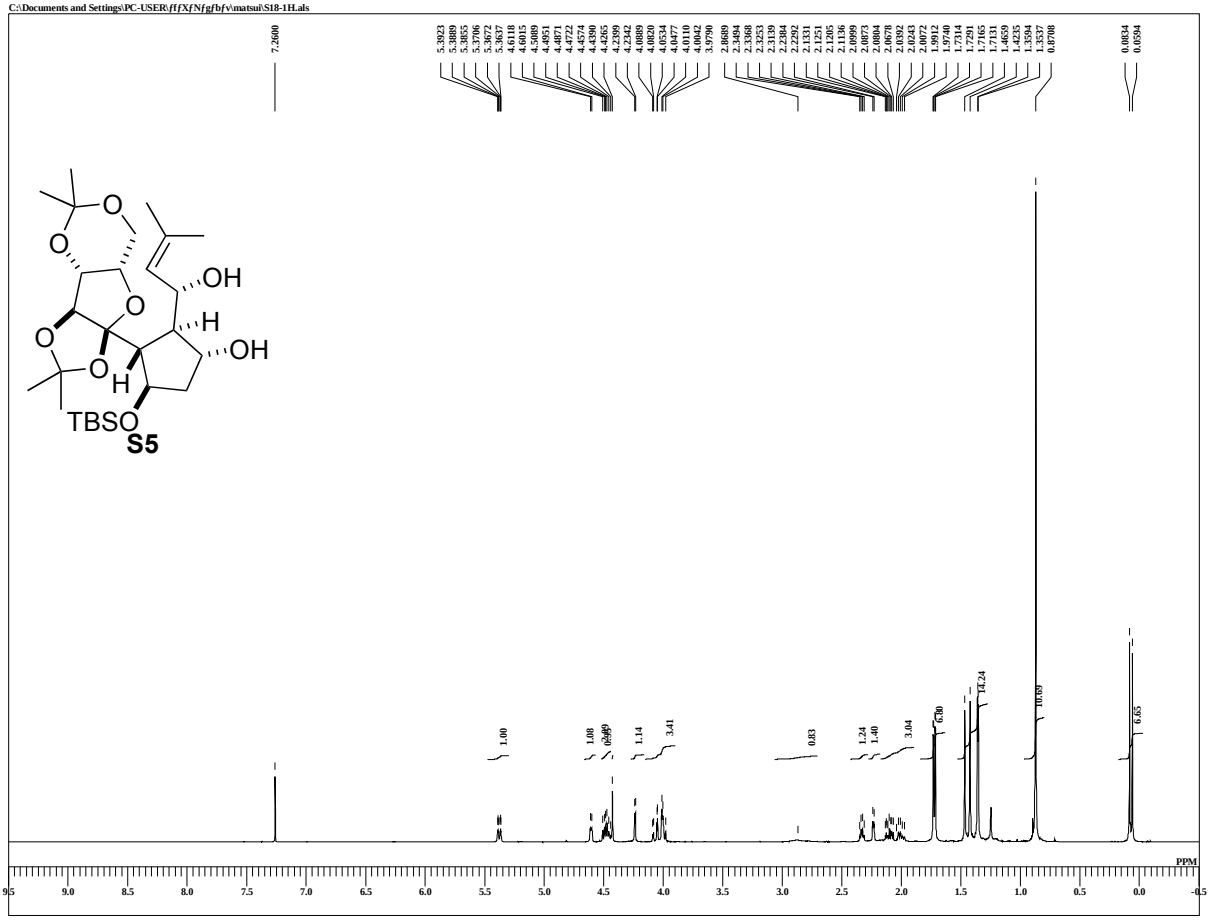
TBSO

S4

```
DFILE    S17-13C2.als
COMNT    single pulse decoupled gat
DATIM    18-02-2014 11:20:51
MENUF
OBNUC    13C
OFR      99.55 MHz
OBFRQ    99.55 MHz
OBSET    5.13 KHz
OBFIN    0.98 Hz
PW1      3.03 usec
DEADT    0.00 usec
PREDL    0.00000 msec
IWT      1.0000 sec
POINT    26214
SPO      26214
TIMES    151
DUMMY    4
FREQU    24999.62 Hz
FLT      125000 Hz
DELAY    20.50 usec
ACQTM    1.0486 sec
PD       2.0000 sec
SCANS    151
ADBIT    16
RGAIN    58
BF       1.00 Hz
T1       0.00
T2       0.00
T3       100.00
T4       100.00
EXMOD    single_pulse_dec
EXPCM
IRNUC    1H
IFR      395.88 MHz
IRSET    6.28 KHz
IRFIN    0.87 Hz
IRRPW    115 usec
IRATN    79
DFILE    S17-13C2.als
SF
LKSET    13.20 KHz
LKFIN    69.6 Hz
LKLEV    0
LGAIN    0
LKPHS    0
LKSIG    0
CSPED    0 Hz
FILDC
FILDF
CTEMP    21.0 c
SLVNT    C6D6
EXREF    128.00 ppm
```

single_pulse

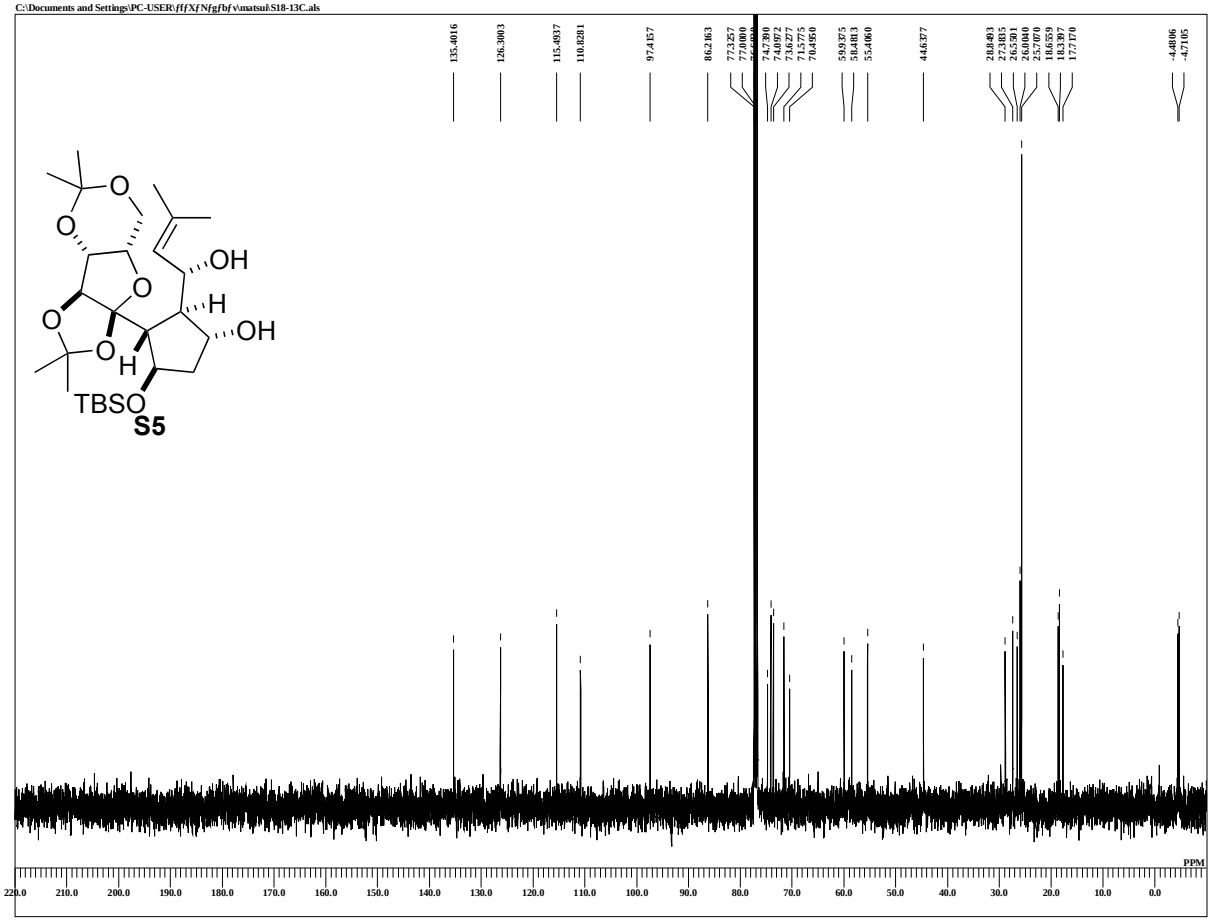
C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\S18-1H.als



DFILE S18-1H.als
COMNT single_pulse
DATIM 19-02-2014 10:42:32
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 13107
SPO 13107
TIMES 8
DUMMY 1
FREQU 5938.15 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 32
BF 0.10 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE S18-1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.1 c
SLVNT CDCL3
EXREF 7.26 ppm

single pulse decoupled gated NOE

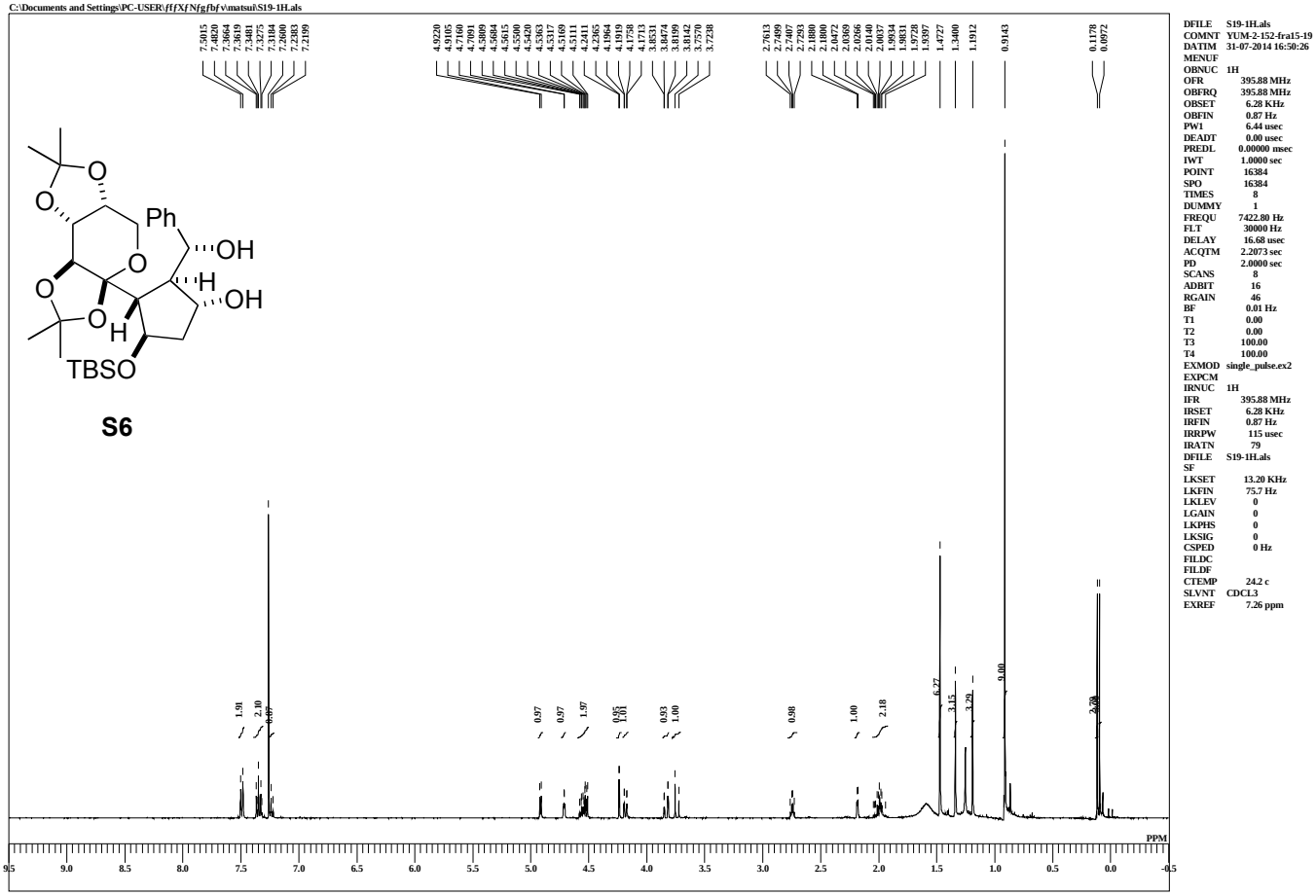
C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\S18-13C.als



DFILE S18-13C.als
COMNT single pulse decoupled gat
DATIM 19-02-2014 11:17:49
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 26214
SPO 26214
TIMES 109
DUMMY 4
FREQU 24999.62 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 109
ADBIT 16
RGAIN 60
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE S18-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 21.1 c
SLVNT CDCL3
EXREF 77.00 ppm

YUM-2-152-fra15-19

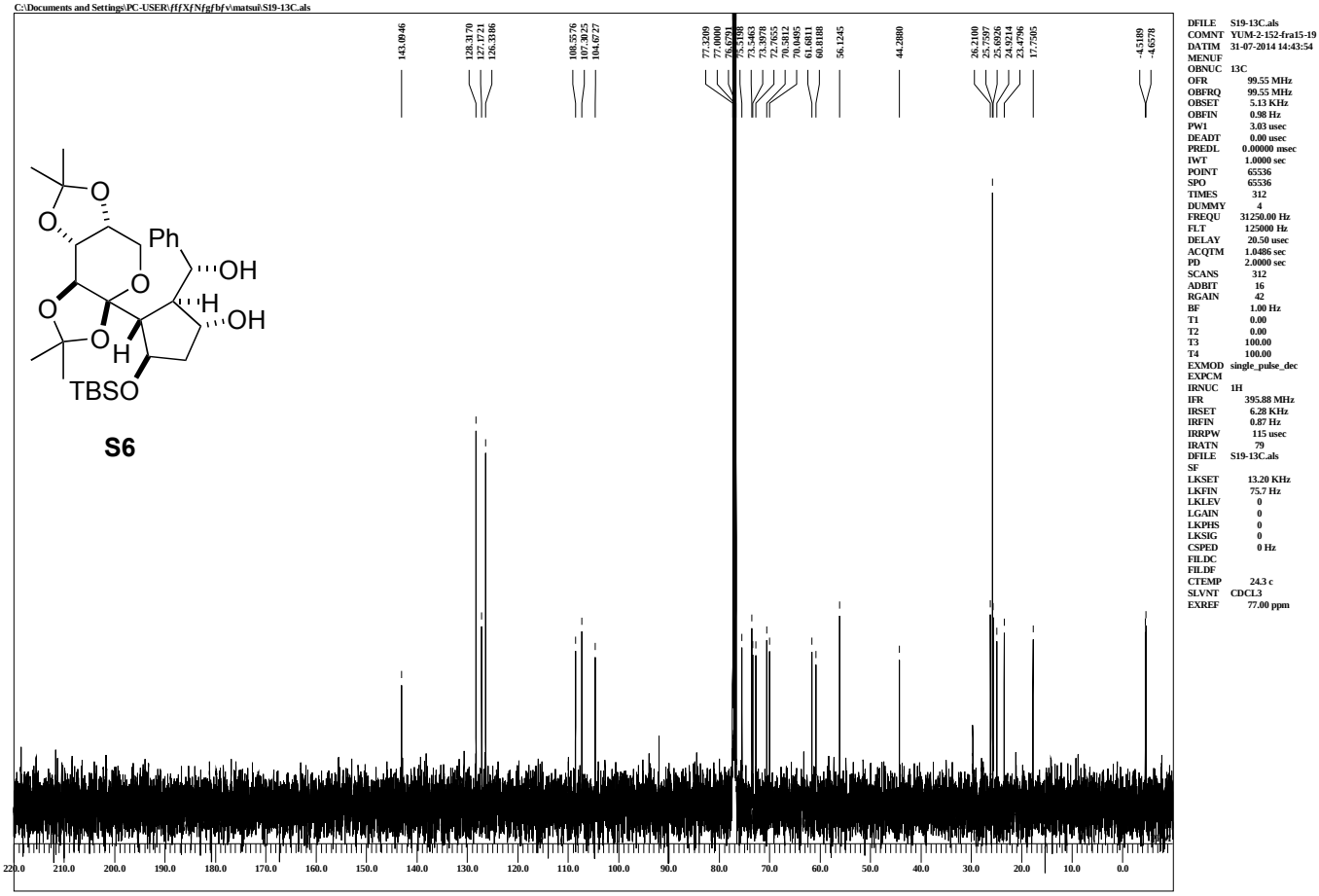
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\S19-1H.als



DFILE S19-1H.als
COMNT YUM-2-152-fra15-19
DATIM 31-07-2014 16:50:26
MENUF
OBNUC 1H
OFR 395.88 MHz
OBFRQ 395.88 MHz
OBSET 6.28 KHz
OBFIN 0.87 Hz
PW1 6.44 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 16384
SPO 16384
TIMES 8
DUMMY 1
FREQU 7422.80 Hz
FLT 30000 Hz
DELAY 16.68 usec
ACQTM 2.2073 sec
PD 2.0000 sec
SCANS 8
ADBIT 16
RGAIN 46
BF 0.01 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse.ex2
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE S19-1H.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.2 c
SLVNT CDCL3
EXREF 7.26 ppm

YUM-2-152-fra15-19

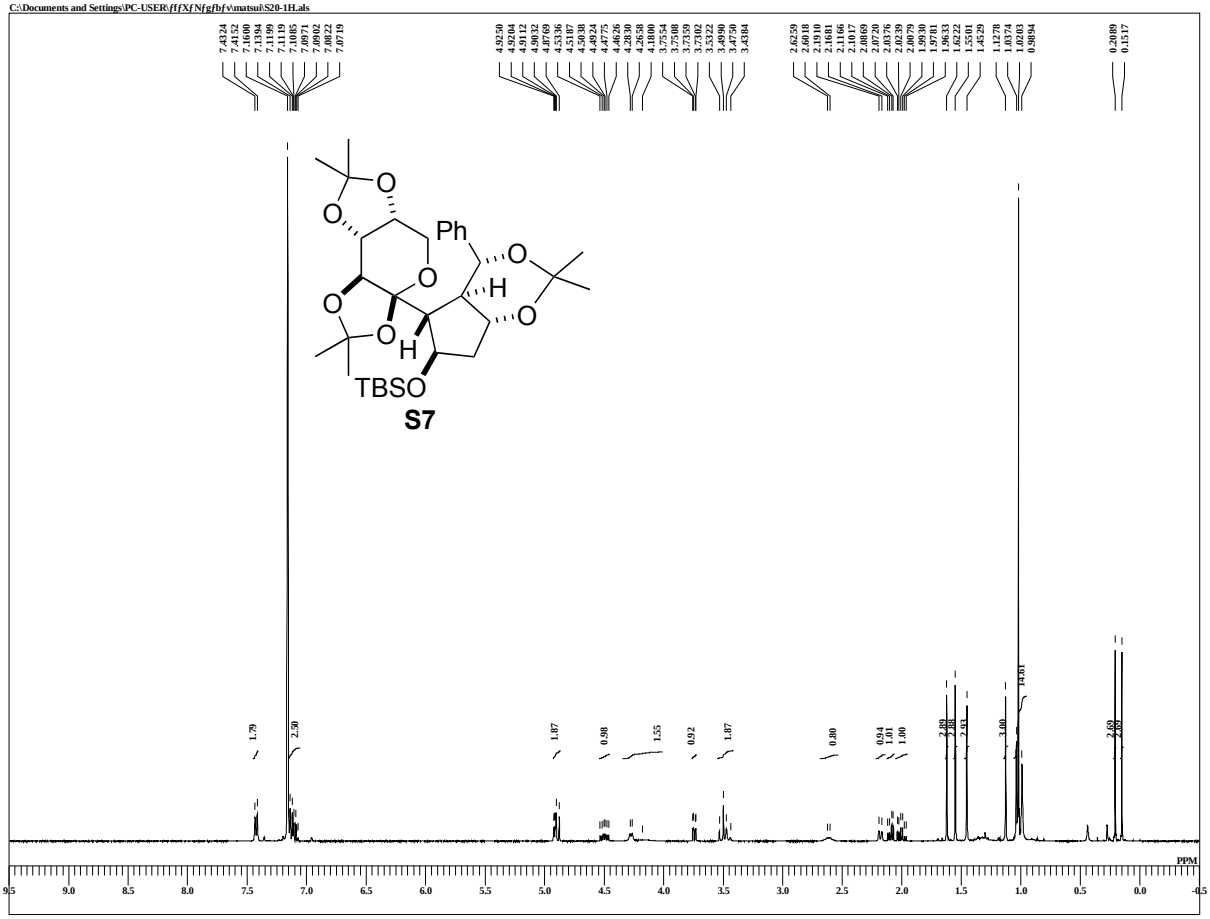
C:\Documents and Settings\PC-USER\ffXfNfgfbfv\matsui\S19-13C.als



DFILE S19-13C.als
COMNT YUM-2-152-fra15-19
DATIM 31-07-2014 14:43:54
MENUF
OBNUC 13C
OFR 99.55 MHz
OBFRQ 99.55 MHz
OBSET 5.13 KHz
OBFIN 0.98 Hz
PW1 3.03 usec
DEADT 0.00 usec
PREDL 0.00000 msec
IWT 1.0000 sec
POINT 65536
SPO 65536
TIMES 312
DUMMY 4
FREQU 31250.00 Hz
FLT 125000 Hz
DELAY 20.50 usec
ACQTM 1.0486 sec
PD 2.0000 sec
SCANS 312
ADBIT 16
RGAIN 42
BF 1.00 Hz
T1 0.00
T2 0.00
T3 100.00
T4 100.00
EXMOD single_pulse_dec
EXPCM
IRNUC 1H
IFR 395.88 MHz
IRSET 6.28 KHz
IRFIN 0.87 Hz
IRRPW 115 usec
IRATN 79
DFILE S19-13C.als
SF
LKSET 13.20 KHz
LKFIN 75.7 Hz
LKLEV 0
LGAIN 0
LKPHS 0
LKSIG 0
CSPED 0 Hz
FILDC
FILDF
CTEMP 24.3 c
SLVNT CDCL3
EXREF 77.00 ppm

YUM-3-65-fra8-20

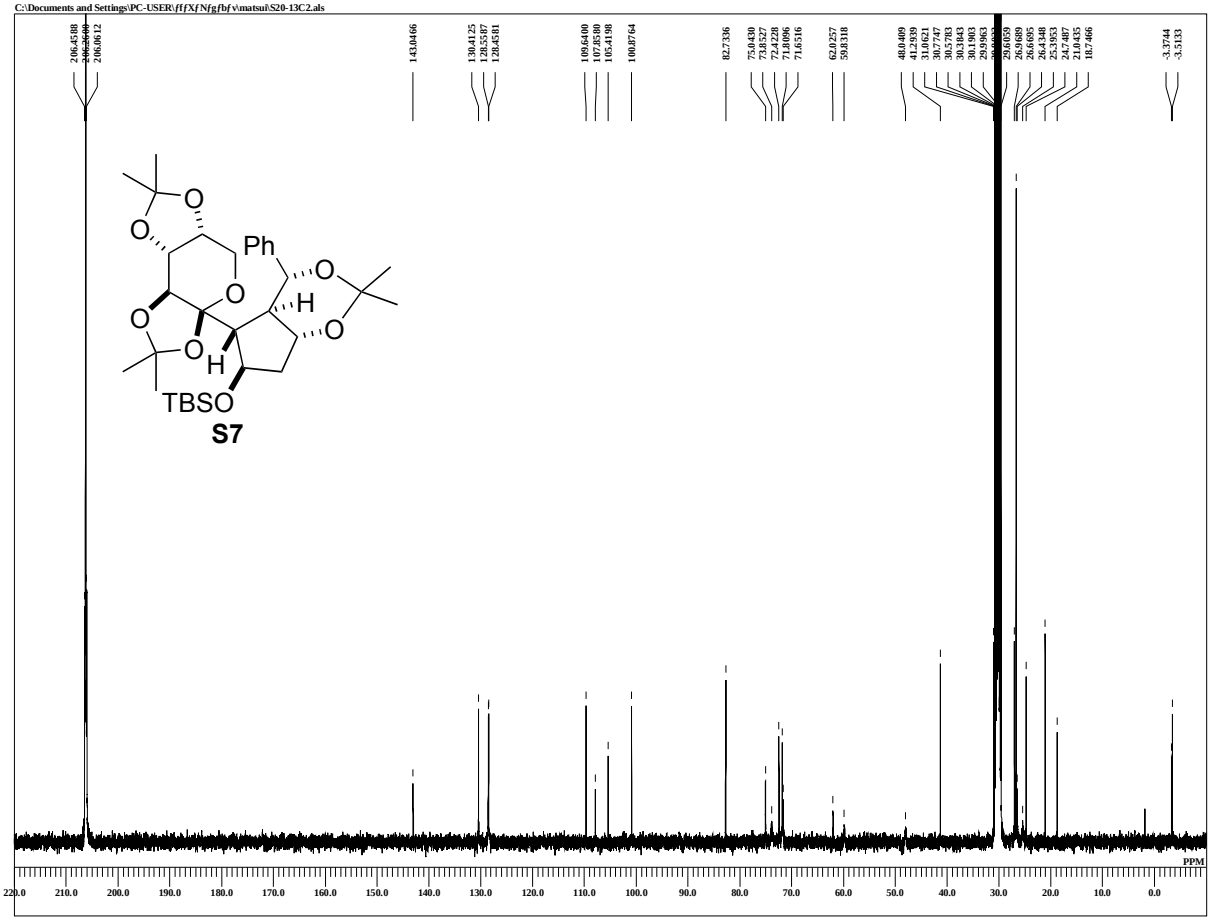
C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\S20-1H.als




```
DFILE     S20-1H.als
COMNT     YUM-3-65-fra8-20
DATIM     16-12-2014 15:00:14
MENUF
OBNUC     1H
OFR       395.88 MHz
OBFRQ     395.88 MHz
OBSET     6.28 KHz
OBFIN     0.87 Hz
PW1       6.56 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     13107
SPO       13107
TIMES     64
DUMMY     1
FREQU     5938.15 Hz
FLT       30000 Hz
DELAY     16.68 usec
ACQTM     2.2073 sec
PD        2.0000 sec
SCANS     64
ADBIT     16
RGAIN     44
BF        0.01 Hz
T1        0.00
T2        0.00
T3        90.00
T4        100.00
EXMOD     single_pulse.ex2
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     S20-1H.als
SF
LKSET     13.20 KHz
LKFIN     69.6 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     70.1 c
SLVNT     C6D6
EXREF     7.16 ppm
```

YUM-3-65-fra8-20

C:\Documents and Settings\PC-USER\ftfXfNfgfbfv\matsui\S20-13C2.als




```
DFILE     S20-13C2.als
COMNT     YUM-3-65-fra8-20
DATIM     19-12-2014 07:42:07
MENUF
OBNUC     13C
OFR       99.55 MHz
OBFRQ     99.55 MHz
OBSET     5.13 KHz
OBFIN     0.98 Hz
PW1       3.07 usec
DEADT     0.00 usec
PREDL     0.00000 msec
IWT       1.0000 sec
POINT     104856
SPO       104856
TIMES     10000
DUMMY     4
FREQU     24999.62 Hz
FLT       125000 Hz
DELAY     20.50 usec
ACQTM     1.0486 sec
PD        2.0000 sec
SCANS     10000
ADBIT     16
RGAIN     60
BF        1.00 Hz
T1        0.00
T2        0.00
T3        100.00
T4        100.00
EXMOD     single_pulse_dec
EXPCM
IRNUC     1H
IFR       395.88 MHz
IRSET     6.28 KHz
IRFIN     0.87 Hz
IRRPW     115 usec
IRATN     79
DFILE     S20-13C2.als
SF
LKSET     12.90 KHz
LKFIN     59.1 Hz
LKLEV     0
LGAIN     0
LKPHS     0
LKSIG     0
CSPED     0 Hz
FILDC
FILDF
CTEMP     50.0 c
SLVNT     ACETN
EXREF     206.26 ppm
```