Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Concise Asymmetric Total Synthesis of Catunaregin

Hideki Abe, Takuma Hikichi, Kosuke Emori, Toyoharu Kobayashi,
and Hisanaka Ito*

School of Life Sciences, Tokyo University of Pharmacy and Life Sciences
1432-1 Horinouchi, Hachioji, Tokyo 192-0392, Japan
itohisa@toyaku.ac.jp

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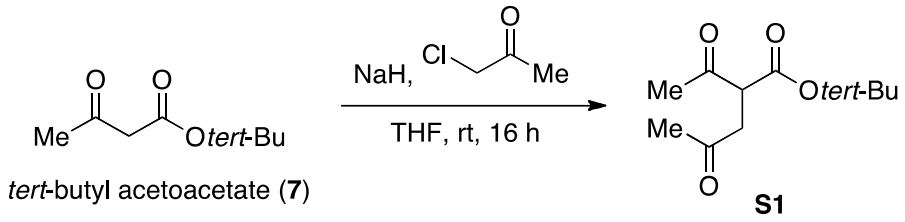
General

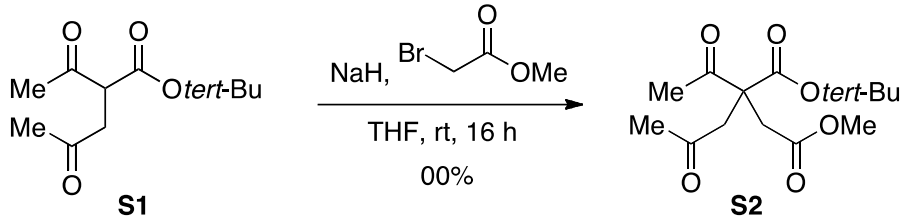
All reactions involving air- and moisture-sensitive reagents were carried out using standard syringe-septumcap techniques. Unless otherwise noted, all solvents and reagents were obtained from commercial suppliers and used without further purification. Routine monitoring of reactions were carried out Merck silica gel 60 F254 TLC plates. Column chromatography was performed on Kanto Chemical Silica Gel 60N (spherical, neutral 60–230 μm) with the solvents indicated. Melting points were taken on a Yanako MP-S3 micro melting point apparatus and are uncorrected. Measurement of optical rotations was performed with a JASCO P-2200 polarimeter. Infrared spectral measurements were carried out with a Perkin–Elmer Paragon 1000 FT-IR and a JASCO FT/IR-4100 spectrometer, and only noteworthy absorptions were listed. ^{1}H and ^{13}C NMR spectra were measured with a JEOL JNM-ECS400 (400 MHz) or a Burker AV-600 (600 MHz) spectrometer. Chemical shifts were expressed in ppm using $CHCl_3$ (7.26 ppm) for ^{1}H NMR and $CHCl_3$ (77.0 ppm) for ^{13}C NMR in $CDCl_3$ as an internal standard. HRMS spectra measured on a Micromass LCT spectrometer.

tert-Butyl 2-Acetyl-4-oxopentanoate (**S1**).



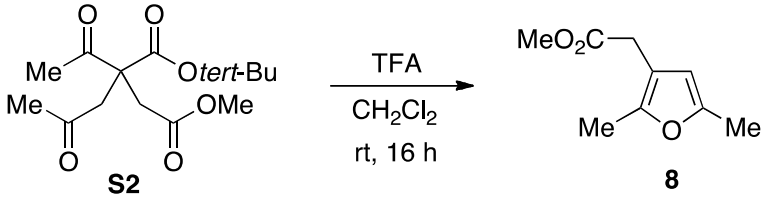
To a stirred suspension of sodium hydride (55% dispersion in mineral oil, 6.58 g, 157 mmol) in tetrahydrofuran (400 mL) was added dropwise *tert*-butyl acetoacetate (**7**) (20.0 mL, 19.1 g, 121 mmol) at 0°C. After stirred for 0.5 h at 0°C, to this reaction mixture was added dropwise α-chloroacetone (12.5 mL, 14.5 g, 157 mmol) at 0 °C, and then stirred for 96 h at room temperature. The reaction mixture was quenched with 1.0 M HCl aqueous solution (100 mL) at 0 °C, and extracted with ether (2 × 300 mL). The combined organic layers were washed with brine, and the washed solution was dried over $MgSO_4$. The dried solution was filtered and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–EtOAc, 7:1) to afford the intermediate **S1** (19.5 g, 91.0 mmol, 75%) as a colorless liquid. Those spectra data were identified for those of previous report.

1-*tert*-Butyl 4-methyl 2-acetyl-2-(2-oxopropyl)succinate (**S2**).



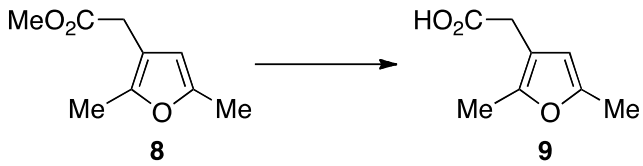
To a stirred suspension of sodium hydride (55% dispersion in mineral oil, 2.88 g, 65.8 mmol) in tetrahydrofuran (170 mL) was added dropwise **S1** (10.9 g, 50.9 mmol) at 0 °C. After stirred for 0.5 h at room temperature, to this mixture was added dropwise methyl bromoacetate (5.30 mL, 8.56 g, 56.0 mmol) at 0 °C, and then stirred for 16 h at room temperature. The reaction mixture was quenched with 1.0 M HCl aqueous solution (80 mL) and extracted with Et_2O (2 × 200 mL). The combined organic layers were washed with brine, and the washed solution was dried over $MgSO_4$. The dried solution was filtered and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–EtOAc, 4:1) to afford the intermediate **S2** (11.8 g, 41.2 mmol, 81%) as a colorless liquid. Those spectra data were identified for those of previous report.

Methyl 2-(2,5-Dimethylfuran-3-yl)acetate (**8**).



To a stirred solution of **S2** (11.8 g, 41.2 mmol) in CH_2Cl_2 (140 mL) was added trifluoroacetic acid (24.5 mL, 37.6 g, 330 mmol) at 0 °C. After stirred for 13 h at room temperature, the mixture was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–EtOAc, 9:1) to afford **8** (4.30 g, 25.6 mmol, 62%) as a pale yellow oil. Those spectra data were identified for those of previous report.

2-(2,5-Dimethylfuran-3-yl)acetic acid (**9**).

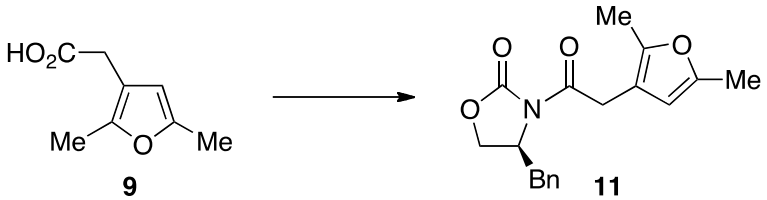


To a stirred solution of **8** (2.99 g, 17.8 mmol) in MeOH (35 mL) were added 3.0 M NaOH aqueous solution (12.5 mL) at room temperature. After stirred for 15 min at same temperature, the reaction was acidified by adding 1.0 M HCl aqueous solution, and extracted with $CHCl_3$ (3 × 150 mL). The

combined organic layers were washed with brine, dried over $MgSO_4$. The dried solution was filtered, and the filtrate was concentrated in vacuo. The resulting residue was purified by recrystallization (hexane) to afford **9** (2.70 g, 17.5 mmol, 99%) as colorless needles.

R_f = 0.45 (hexane–AcOEt–AcOH = 1:1:0.04); Mp: 52–54 °C (hexane); IR (KBr) 3407, 3104, 2985, 2949, 2924, 2676, 1713, 1586, 1434, 1415, 1229, 1099, 991, 926, 800 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.20 (3H, s), 2.22 (3H, s), 3.34 (2H, s), 5.88 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.3, 13.4, 30.8, 107.7, 111.4, 147.2, 149.7, 178.2; HRMS (ESI–TOF) calcd for $C_8H_{10}O_3Na$ ($[M + Na]^+$) 177.0528, found 177.0527; Anal. Calcd for $C_8H_{10}O_3$: C, 62.33; H, 6.54. Found: C, 62.20, 6.51.

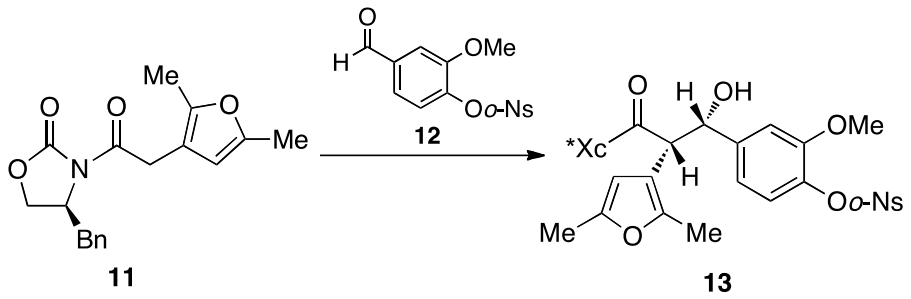
(*S*)-4-Benzyl-3-[2-(2,5-dimethylfuran-3-yl)acetyl]oxazolidin-2-one (**11**).



To a stirred solution of **9** (1.75 g, 11.4 mmol) and triethylamine (1.75 mL, 1.26 g, 12.6 mmol) in THF (38 mL) under argon was added dropwise pivaloyl chloride (1.54 mL, 1.51 g, 12.5 mmol) at –78 °C, and the reaction mixture was stirred for 0.5 h at 0 °C. To this mixture was added dropwise a solution of lithium (*S*)-4-benzyl-2-oxooxazolin-3-ide in THF (42 mL), prepared from (*S*)-4-benzyloxazolidin-2-one (2.22 g, 12.5 mmol) and *n*-buthyllithium (1.63 M in hexane, 7.69 mL, 12.5 mmol), at –78 °C. After stirred for 1 h at 0 °C, the reaction was quenched with saturated NH_4Cl aqueous solution (50 mL), and extracted with AcOEt (2 × 120 mL). The combined organic layers was washed with brine, dried over $MgSO_4$. The dried solution was filtered, and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–AcOEt, 4:1) to afford **11** (3.30 g, 10.5 mmol, 93%) as a colorless oil.

R_f = 0.61 (hexane–AcOEt = 2:1); $[\alpha]_D^{25}$ +56.4 (*c* 1.00, $CHCl_3$); IR (neat) 3029, 2981, 2921, 1781, 1701, 1584, 1454, 1390, 1356, 1211, 1197, 1111, 1052, 994, 762, 747, 703 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.23 (3H, s), 2.24 (3H, s), 2.77 (1H, dd, *J* = 13.3, 9.6 Hz), 3.29 (1H, dd, *J* = 13.3, 3.2 Hz), 3.94 and 4.01 (2H, ABq, *J* = 17.0 Hz), 4.18 (1H, A part of ABX, *J* = 9.2, 3.2 Hz), 4.22 (1H, B part of ABX, *J* = 9.2, 7.6 Hz), 4.64–4.70 (1H, m), 5.92 (1H, s), 7.16–7.19 (2H, m), 7.24–7.34 (3H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.5, 13.4, 31.9, 37.7, 55.2, 66.1, 107.9, 111.4, 127.3, 128.9 (2C), 129.4 (2C), 135.1, 147.5, 149.6, 153.4, 171.0; HRMS (ESI–TOF) calcd for $C_{18}H_{19}NO_4Na$ ($[M + Na]^+$) 336.1212, found 336.1208.

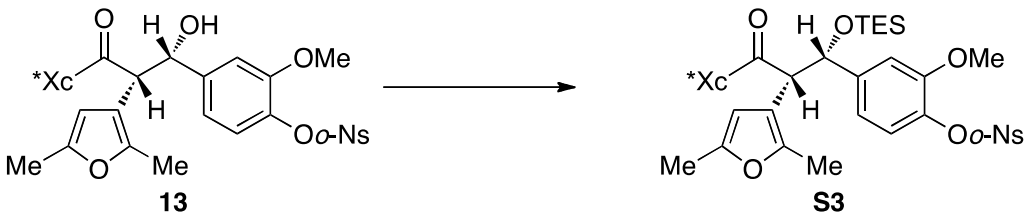
4-[(1*S*,2*S*)-3-((*S*)-4-Benzyl-2-oxooxazolidin-3-yl)-2-(2,5-dimethylfuran-3-yl)-1-hydroxy-3-oxopropyl]-2-methoxyphenyl 2-nitrobenzenesulfonate (**13**).



To a stirred solution of **11** (1.31 g, 4.18 mmol) in CH_2Cl_2 (14 mL) were added dropwise *N*,*N*-diisopropylethylamine (0.981 mL, 0.728 g, 5.63 mmol), and di-*n*-butylboryl trifluoromethanesulfonate (1.0 M in CH_2Cl_2, 4.74 mL, 4.74 mmol) at 0 °C. After stirred for 45 min at 0 °C, to this mixtre was added dropwise a solution of *O*-*o*-Ns-vanillin **12** (1.00 g, 2.96 mmol) in CH_2Cl_2 (10 mL) at –78 °C. After stirred for 2.5 h at –78 °C, the reaction was quenched with MeOH (40 mL) and saturated NH_4Cl aqueous solution (40 mL) at –78 °C, and extracted with $CHCl_3$ (2 × 200 mL). The combined organic layers were washed with brine, and the washed solution was dried over Na_2SO_4. The dried solution was filtered, and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–AcOEt, 2:1) to afford **13** (1.89 g, 2.90 mmol, 98%) as a colorless oil.

R_f = 0.26 (hexane–AcOEt = 2:1); $[\alpha]_D^{25}$ +4.7 (*c* 1.00, $CHCl_3$); IR (neat) 3502, 3088, 2921, 2850, 1774, 1690, 1604, 1545, 1501, 1385, 1364, 1272, 1199, 1111, 1031, 864, 852, 760 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.04 (3H, s), 2.26 (3H, s), 2.67 (1H, dd, *J* = 13.6, 9.2 Hz), 2.90–3.06 (1H, br s), 3.09 (1H, dd, *J* = 13.6, 3.2 Hz), 3.49 (3H, s), 4.02–4.12 (2H, m), 4.53–4.60 (1H, m), 5.15 (1H, d, *J* = 6.8 Hz), 5.18 (1H, d, *J* = 6.8 Hz), 6.13 (1H, s), 6.84–6.91 (2H, m); 7.01–7.08 (2H, m); 7.11 (1H, d, *J* = 8.4 Hz), 7.22–7.29 (3H, m); 7.68–7.75 (1H, m); 7.76–7.85 (2H, m); 8.00–8.05 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.3, 13.5, 37.3, 47.6, 54.7, 55.5, 65.8, 74.2, 106.4, 111.3, 112.1, 119.2, 123.7, 124.5, 127.3, 128.8 (2C), 129.4 (2C), 129.9, 131.6, 131.8, 134.6, 134.9, 137.7, 141.5, 148.3, 150.1, 150.2, 151.2, 152.7, 172.7; HRMS (ESI–TOF) calcd for $C_{32}H_{30}N_2O_{11}SNa$ ($[M + Na]^+$) 673.1468, found 673.1465.

4-[(1*S*,2*S*)-3-((*S*)-4-Benzyl-2-oxooxazolidin-3-yl)-2-(2,5-dimethylfuran-3-yl)-3-oxo-1-(triethylsilyloxy)propyl]-2-methoxyphenyl 2-nitrobenzenesulfonate (**S3**).

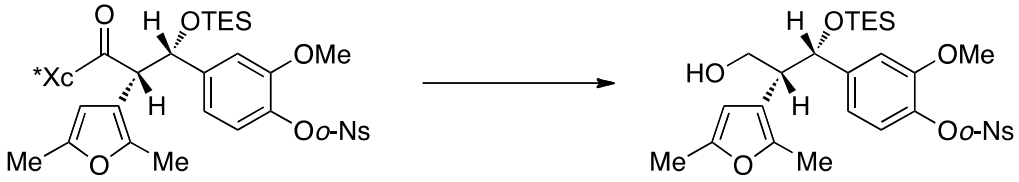


To a stirred solution of **13** (500 mg, 0.768 mmol) in CH_2Cl_2 (4 mL) were added dropwise 1,8-diazabicyclo[5.4.0]undec-7-ene (0.689 mL, 703 mg, 4.62 mmol), and triethylsilyl

trifluoromethanesulfonate (0.434 mL, 508 mg, 1.92 mmol) at –78 °C under argon, and the mixture was stirred for 20 h at –50 °C. The reaction was quenched with saturated NH_4Cl aqueous solution (5 mL), and extracted with $CHCl_3$ (3 × 20 mL). The combined organic layers were washed with brine, dried over Na_2SO_4. The dried solution was filtered, and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–AcOEt, 2:1) to afford **S3** (527 mg, 0.689 mmol, 90%) as a as colorless gum.

R_f = 0.45 (hexane–AcOEt–Et_2O = 2:1:1); $[\alpha]_D^{25}$ +10.9 (*c* 1.00, $CHCl_3$); IR (neat) 3028, 2955, 2915, 2876, 2360, 2341, 1776, 1696, 1604, 1548, 1501, 1389, 1281, 1201, 1111, 1004, 891, 859, 655, 591 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.34 (6H, q, *J* = 7.9 Hz), 0.71 (9H, t, *J* = 7.9 Hz), 2.21 (3H, s), 2.25 (3H, s), 2.65 (1H, dd, *J* = 13.5, 8.9 Hz), 3.06 (1H, dd, *J* = 13.5, 3.0 Hz), 3.50 (3H, s), 3.92–4.03 (2H, m), 4.36–4.42 (1H, m), 5.00 (1H, d, *J* = 8.2 Hz), 5.25 (1H, d, *J* = 8.2 Hz), 6.20 (1H, s), 6.88 (1H, dd, *J* = 8.2, 1.8 Hz), 6.99 (1H, d, *J* = 1.4 Hz), 7.01–7.11 (3H, m), 7.20–7.30 (3H, m), 7.67–7.75 (1H, m), 7.77–7.84 (2H, m), 8.01 (1H, d, *J* = 8.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.5 (3C), 6.4 (3C), 11.6, 13.5, 37.3, 49.0, 54.9, 55.6, 65.6, 75.9, 106.7, 111.3, 114.4, 119.4, 123.4, 124.4, 127.2, 128.7 (2C), 129.4 (2C), 129.8, 131.5, 131.7, 134.87, 134.89, 137.4, 144.0, 148.4, 149.2, 149.3, 151.1, 152.9, 171.5; HRMS (ESI–TOF) calcd for $C_{38}H_{44}N_2O_{11}SSiNa$ ($[M + Na]^+$) 787.2333, found 787.2331.

4-[(1*S*,2*R*)-2-(2,5-dimethylfuran-3-yl)-3-hydroxy-1-(triethylsilyloxy)propyl]-2-methoxyphenyl 2-nitrobenzenesulfonate (**15**).

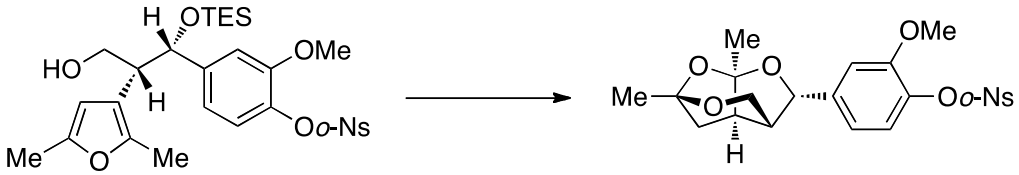


To a stirred suspension of lithium chloride (108 mg, 2.54 mmol) in THF (5 mL) were added sodium borohydride (95.9 mg, 2.54 mmol) at room temperature, and the mixture was stirred for 0.5 h at 40 °C. To this mixture was added dropwise a solution of **S4** (194 mg, 0.254 mmol) in THF (2.5 mL) at 40 °C, and the mixture was stirred for 25 h at 50 °C. The reaction was quenched with saturated NH_4Cl aqueous solution (5 mL), and extracted with $CHCl_3$ (3 × 20 mL). The combined organic layers were washed with brine, dried over Na_2SO_4. The dried solution was filtered, and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–AcOEt, 2:1) to afford **15** (117 mg, 0.198 mmol, 78%) as colorless oil.

R_f = 0.50 (hexane–AcOEt = 1:2); $[\alpha]_D^{25}$ –27.0 (*c* 0.3, $CHCl_3$); IR (neat) 3582, 3419, 3098, 2955, 2914, 2877, 1732, 1604, 1548, 1500, 1464, 1417, 1387, 1282, 1266, 1200, 1148, 1111, 1007, 866, 851, 761, 741, 591 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.41–0.55 (6H, m), 0.82–0.87 (9H, m),

1.72 (3H, s), 1.79 (1H, br s), 2.18 (3H, s), 2.77 (1H, ddd, *J* = 7.3, 7.3, 3.7 Hz), 3.38 (3H, s), 3.70 (1H, dd, *J* = 10.5, 7.3 Hz), 3.84 (1H, dd, *J* = 10.5, 7.3 Hz), 4.93 (1H, d, *J* = 3.7 Hz), 5.77 (1H, s), 6.55 (1H, d, *J* = 1.8 Hz), 6.69 (1H, dd, *J* = 8.2, 1.8 Hz), 7.07 (1H, dd, *J* = 8.2 Hz), 7.65–7.70 (1H, m), 7.78–7.86 (2H, m), 7.94–7.97 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 4.65 (3C), 6.67 (3C), 10.9, 13.4, 46.9, 55.3, 63.7, 74.9, 106.8, 111.3, 115.1, 118.4, 123.3, 124.6, 130.0, 131.6 (2C), 134.7, 137.1, 144.2, 148.0, 148.5, 148.8, 150.7; HRMS (ESI–TOF) calcd for $C_{28}H_{37}NO_9SSiNa$ ([M + Na]$^+$) 614.1856, found 614.1860.

4-[(1*R*,3*S*,3a*R*,6*S*,7a*R*)-1,6-Dimethylhexahydro-1*H*-1,6-epoxyfuro[3,4-*c*]pyran-3-yl]-2-methoxyphenyl 2-nitrobenzenesulfonate (**16**).



To a stirred solution of **15** (74.0 mg, 0.125 mmol) in THF (1 mL) was added concentrated H_2SO_4 (0.200 mL) at room temperature under argon, and the mixture was stirred for 8 h at the same temperature. The reaction was carefully poured into saturated $NaHCO_3$ aqueous solution, and extracted with $CHCl_3$ (3 × 30 mL). The combined organic layers were washed with brine, dried over $MgSO_4$. The dried solution was filtered, and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–AcOEt, 1:3) to afford **16** (37.0 mg, 0.0775 mmol, 62%) as a colorless oil, and diol **14** (2.2 mg, 0.00461 mmol, 4%) as a colorless oil.

Data of **16**

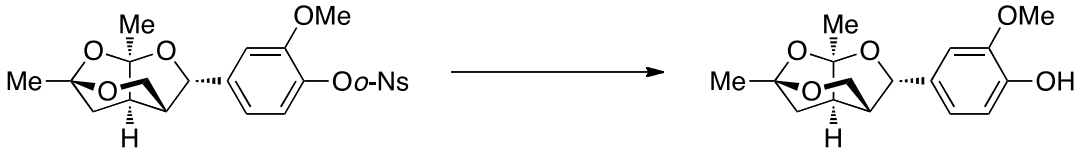
R_f = 0.57 (hexane–AcOEt = 1:9); $[\alpha]_D^{25}$ +33.3 (*c* 0.55, $CHCl_3$); IR (neat) 3502, 3096, 2985, 2936, 2879, 2360, 2341, 1732, 1604, 1546, 1505, 1465, 1453, 1385, 1274, 1200, 1166, 1112, 1059, 1032, 942, 896, 852, 758, 592 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.48 (3H, s), 1.60 (3H, s), 1.88 (1H, dd, *J* = 12.4, 4.1 Hz), 2.26 (1H, d, *J* = 12.4 Hz), 2.45–2.50 (1H, m), 2.68 (1H, dd, *J* = 10.1, 3.7 Hz), 3.57 (3H, s), 3.75–3.78 (2H, m), 4.98 (1H, d, *J* = 3.7 Hz), 6.82–6.86 (2H, m), 7.15 (1H, d, *J* = 8.7 Hz), 7.68–7.74 (1H, m), 7.79–7.84 (1H, m), 7.85–7.88 (1H, m), 8.03 (1H, d, *J* = 7.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.5, 24.7, 33.2, 45.5, 48.0, 55.6, 63.0, 84.6, 106.1, 110.2, 115.5, 117.8, 124.3, 124.7, 130.2, 131.6, 131.9, 134.8, 137.5, 143.5, 148.4, 151.5; HRMS (ESI–TOF) calcd for $C_{22}H_{23}NO_9SNa$ ([M + Na]$^+$) 500.0991, found 500.0994.

Data of diol **14**

R_f = 0.56 (hexane–AcOEt = 1:19); $[\alpha]_D^{25}$ –20.2 (*c* 0.27, $CHCl_3$); IR (neat) 3558, 3399, 3099, 3008, 2921, 1717, 1604, 1546, 1502, 1465, 1419, 1384, 1271, 1199, 1175, 1110, 1059, 1031, 864, 853,

765 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.52–1.70 (1H, br s), 1.87 (3H, s), 2.23 (3H, s), 2.34–2.42 (1H, br s), 2.80–2.86 (1H, m), 3.49 (3H, s), 3.72–3.76 (2H, m), 4.92 (1H, d, *J* = 5.0 Hz), 5.95 (1H, s), 6.74 (1H, s), 6.80 (1H, dd, *J* = 8.7, 1.4 Hz), 7.13 (1H, d, *J* = 8.2 Hz), 7.71 (1H, ddd, *J* = 8.2, 7.8, 1.4 Hz), 7.81 (1H, ddd, *J* = 8.2, 7.8, 1.4 Hz), 7.86 (1H, dd, *J* = 8.2, 1.4 Hz), 8.02 (1H, dd, *J* = 8.2, 1.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 11.2, 13.5, 46.3, 55.9, 64.0, 74.4, 106.0, 111.0, 114.8, 118.6, 123.7, 124.7, 130.2, 131.6, 131.8, 134.8, 137.5, 143.4, 148.4, 148.5, 150.1, 151.2; HRMS (ESI–TOF) calcd for $C_{22}H_{23}NO_9SNa$ ([M + Na]$^+$) 500.0991, found 500.0987.

4-[(1*R*,3*S*,3a*R*,6*S*,7a*R*)-1,6-dimethylhexahydro-1*H*-1,6-epoxyfuro[3,4-*c*]pyran-3-yl]-2-methoxyphenol [catunaregin (**1**)].



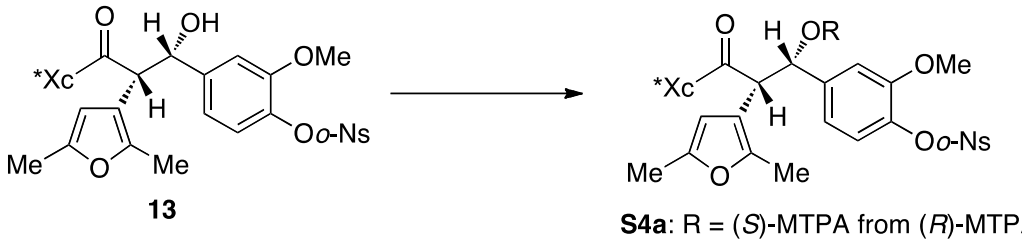
16 (+)-catunaregin (**1**)

To a stirred solution of **16** (37.0 mg, 0.0775 mmol) and cesium carbonate (126 mg, 0.387 mmol) in CH_3CN (1 mL) was added dropwise thiophenol (23.8 μL, 25.6 mg, 0.0232 mmol) at 0 °C, and the mixture was stirred for 2 h at room temperature. The reaction was quenched with saturated NH_4Cl aqueous solution, and the mixture was extracted with AcOEt (2 × 30 mL). The combined organic layers were washed with brine, dried over $MgSO_4$. The dried solution was filtered, and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–AcOEt, 1:9) to afford **1** (22.6 mg, 0.0773 mmol, 100%) as a colorless oil.

R_f = 0.67 (hexane–AcOEt = 1:9); $[\alpha]_D^{25}$ +38.5 (*c* 0.350, MeOH); $[\alpha]_D^{24}$ +40.3 (*c* 0.405, $CHCl_3$); IR (neat) 3408, 2984, 2937, 2879, 1608, 1603, 1517, 1460, 1450, 1432, 1385, 1331, 1270, 1238, 1195, 1170, 1113, 1057, 1032, 942, 847, 819, 794, 762 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.49 (3H, s), 1.60 (3H, s), 1.88 (1H, dd, *J* = 12.3, 3.7 Hz), 2.26 (1H, d, *J* = 12.3 Hz), 2.46–2.52 (1H, m), 2.71 (1H, dd, *J* = 10.1, 3.7 Hz), 3.73–3.78 (2H, m), 3.91 (3H, s), 4.95 (1H, d, *J* = 4.1 Hz), 5.60 (1H, s), 6.77–6.83 (2H, m), 6.86–6.90 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.6, 24.8, 33.3, 45.7, 47.8, 55.9, 62.9, 85.3, 105.9, 108.6, 114.4, 115.3, 118.8, 134.4, 145.2, 146.5; HRMS (ESI–TOF) calcd for $C_{16}H_{20}O_5Na$ ([M + Na]$^+$) 315.1208, found 315.1201.

Syntheses of (*S*)- and (*R*)-MTPA esters, and Δδ values of the Mosher's esters.

(*S*)-(1*S*,2*S*)-3-[(*S*)-4-Benzyl-2-oxooxazolidin-3-yl]-2-(2,5-dimethylfuran-3-yl)-1-[3-methoxy-4-(2-nitrophenylsulfonyloxy)phenyl]-3-oxopropyl 3,3,3-Trifluoro-2-methoxy-2-phenylpropanoate (**S4a**).



S4a: R = (*S*)-MTPA from (*R*)-MTPACl
S4b: R = (*R*)-MTPA from (*S*)-MTPACl

To a stirred solution of **13** (50.0 mg, 0.0768 mmol) in CH_2Cl_2 (1 mL) was added pyridine (31.0 μL, 30.4 mg, 0.384 mmol), and (*R*)-(–)-α-methoxy-α-trifluoromethylphenylacetyl chloride (28.8 μL, 38.8 mg, 0.154 mmol) in 0 °C. After stirred for 5 h at room temperature, the reaction was quenched with saturated NH_4Cl aqueous solution (3 mL), and extracted with $CHCl_3$ (3 × 10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4. The dried solution was filtered, and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–AcOEt, 2:1) to afford **S4a** (51.4 mg, 59.3 mmol, 77%) as colorless amorphous solid.

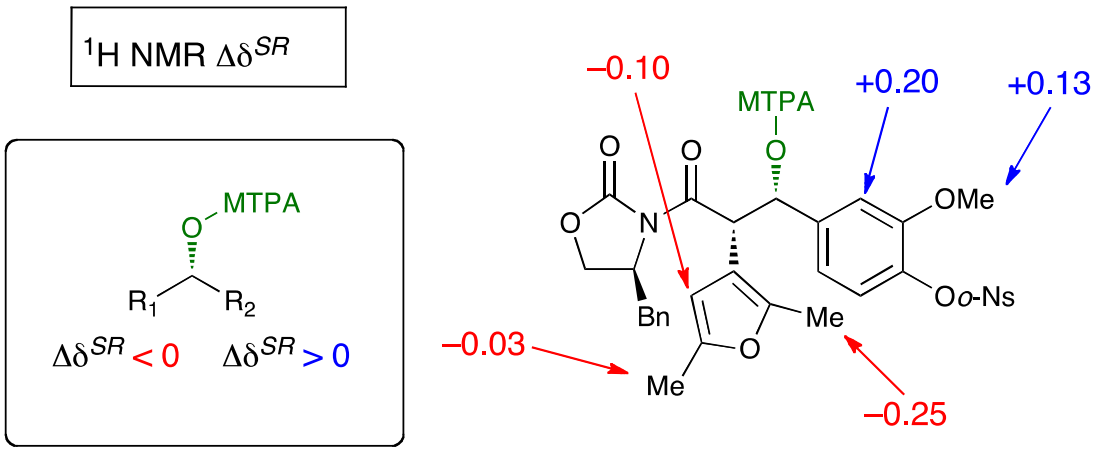
R_f = 0.56 (hexane–AcOEt = 1:1); $[\alpha]_D^{20}$ –12.5 (*c* 0.34, $CHCl_3$); IR (neat) 3430, 3027, 2950, 2922, 2849, 1774, 1752, 1696, 1638, 1606, 1546, 1502, 1452, 1389, 1362, 1271, 1255, 1201, 1182, 1113, 1022, 999, 863, 851, 760, 717 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 1.93 (3H, s), 2.25 (3H, s), 2.68 (1H, dd, *J* = 13.6, 8.3 Hz), 2.95 (1H, dd, *J* = 13.6, 3.4 Hz), 3.26 (3H, m), 3.54 (3H, s), 4.02 (1H, A part of ABX, *J* = 9.0, 3.6 Hz), 4.08 (1H, B part of ABX, *J* = 9.0, 8.4 Hz), 4.46–4.52 (1H, m), 5.65 (1H, d, *J* = 10.0 Hz), 6.10 (1H, s), 6.49 (1H, d, *J* = 10.0 Hz), 6.92–6.97 (2H, m), 7.02 (1H, d, *J* = 1.9 Hz), 7.07 (1H, dd, *J* = 8.4, 1.8 Hz), 7.14–7.19 (3H, m), 7.20–7.23 (3H, m), 7.27–7.29 (2H, m), 7.35–7.39 (1H, m), 7.72–7.79 (2H, m), 7.80–7.84 (1H, m), 8.09 (1H, d, *J* = 7.9 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.2, 13.6, 37.1, 45.1, 54.5, 55.3, 55.7, 65.7, 77.04, 84.3 (q, *J* = 27.5 Hz), 105.8, 112.56, 112.64, 120.5, 123.2 (q, *J* = 288.7 Hz), 124.3, 124.4, 126.9 (2C), 127.3, 128.2 (2C), 128.7 (2C), 129.36, 129.42 (2C), 129.9, 131.5, 131.8, 132.0, 134.4, 135.0, 137.7, 138.4, 148.4, 149.9, 150.1, 151.6, 152.9, 165.5, 170.1; HRMS (ESI–TOF) calcd for $C_{42}H_{37}F_3N_2O_{13}SNa$ ($[M + Na]^+$) 889.1866, found 889.1856.

(*R*)-(1*S*,2*S*)-3-((*S*)-4-benzyl-2-oxooxazolidin-3-yl)-2-(2,5-dimethylfuran-3-yl)-1-(3-methoxy-4-(((2-nitrophenyl)sulfonyl)oxy)phenyl)-3-oxopropyl 3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (**S4b**).

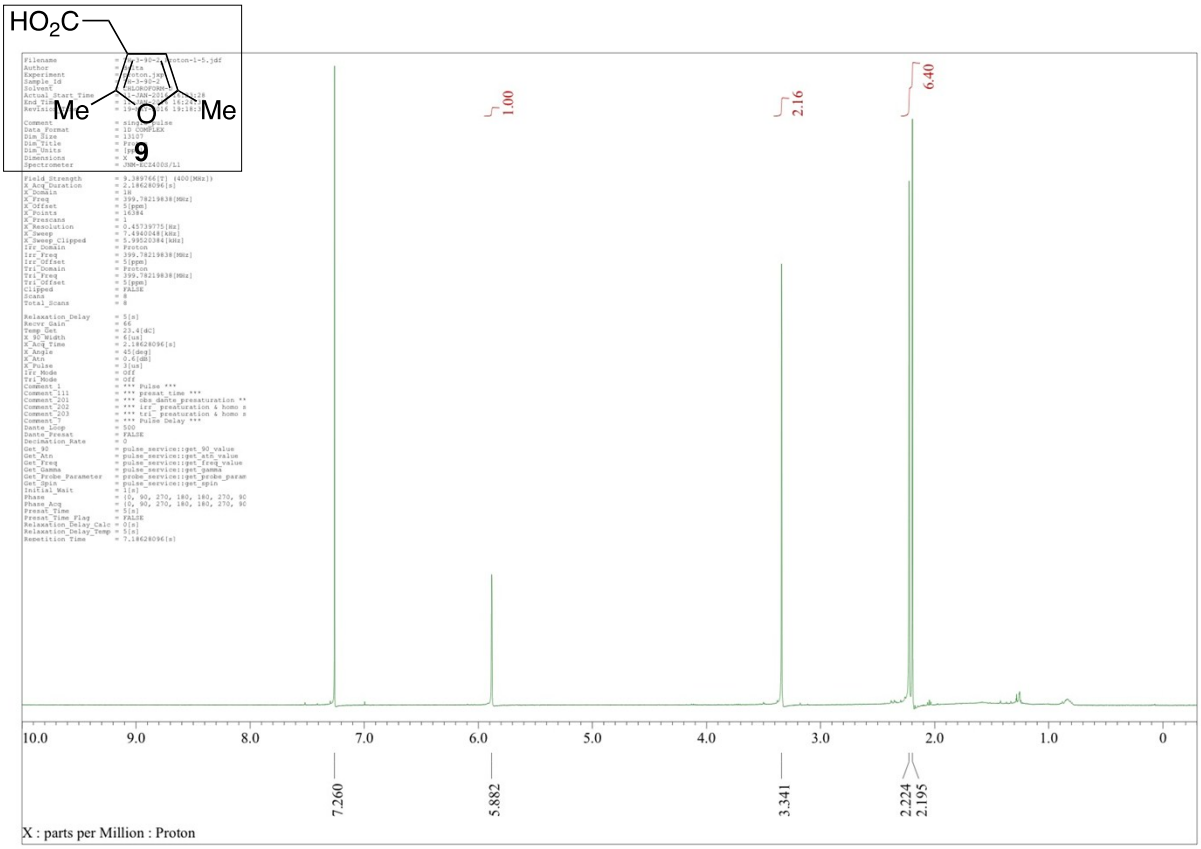
To a stirred solution of **13** (50.0 mg, 0.0768 mmol) in CH_2Cl_2 (1 mL) was added pyridine (31.0 μL,

30.4 mg, 0.384 mmol), and (*S*)-(+)-α-methoxy-α-trifluoromethylphenylacetyl chloride (28.8 μL, 38.8 mg, 0.154 mmol) in 0 °C. After stirred for 5 h at room temperature, the reaction was quenched with saturated NH_4Cl aqueous solution (3 mL), and extracted with $CHCl_3$ (3 × 10 mL). The combined organic layers were washed with brine, dried over Na_2SO_4. The dried solution was filtered, and the filtrate was concentrated in vacuo. The resulting residue was purified by column chromatography (hexane–AcOEt, 2:1) to afford **S4b** (61.9x mg, 71.4 mmol, 93%) as colorless amorphous solid.

R_f = 0.63 (hexane–AcOEt = 1:1); $[\alpha]_D^{20}$ +16.8 (*c* 0.62, $CHCl_3$); IR (neat) 3432, 3026, 2950, 2923, 2850, 1775, 1753, 1696, 1637, 1606, 1546, 1502, 1452, 1389, 1365, 1270, 1255, 1202, 1182, 1113, 1030, 1000, 864, 851, 760 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 2.18 (3H, s), 2.28 (3H, s), 2.69 (1H, dd, *J* = 13.6, 8.3 Hz), 2.98 (1H, dd, *J* = 13.6, 3.2 Hz), 3.26 (3H, m), 3.41 (3H, s), 4.00 (1H, A part of ABX, *J* = 9.1, 3.0 Hz), 4.05 (1H, B part of ABX, *J* = 9.1, 8.3 Hz), 4.46–4.51 (1H, m), 5.66 (1H, d, *J* = 10.2 Hz), 6.20 (1H, s), 6.34 (1H, d, *J* = 10.2 Hz), 6.82 (1H, d, *J* = 1.9 Hz), 6.94–6.99 (3H, m), 7.06–7.12 (3H, m), 7.21–7.27 (5H, m), 7.33–7.37 (1H, m), 7.73–7.77 (2H, m), 7.80–7.84 (1H, m), 8.08–8.11 (1H, m); ^{13}C NMR (150 MHz, $CDCl_3$) δ 11.5, 13.6, 37.1, 45.3, 54.5, 55.1, 55.5, 65.7, 77.9, 84.5 (q, *J* = 27.5 Hz), 105.7, 112.0, 113.4, 120.7, 123.2 (q, *J* = 288.7 Hz), 124.1, 124.4, 127.1 (2C), 127.3, 128.2 (2C), 128.8 (2C), 129.4 (2C), 129.5, 129.9, 131.6, 131.81, 131.84, 134.5, 134.9, 137.5, 138.3, 148.4, 150.1, 150.3, 151.4, 152.8, 165.3, 170.1; HRMS (ESI–TOF) calcd for $C_{42}H_{37}F_3N_2O_{13}SNa$ ([M + Na]$^+$) 889.1866, found 889.1863.



S3a: (*S*)-MTPA ester (from (*R*)-MTPACl)
S3b: (*R*)-MTPA ester (from (*S*)-MTPACl)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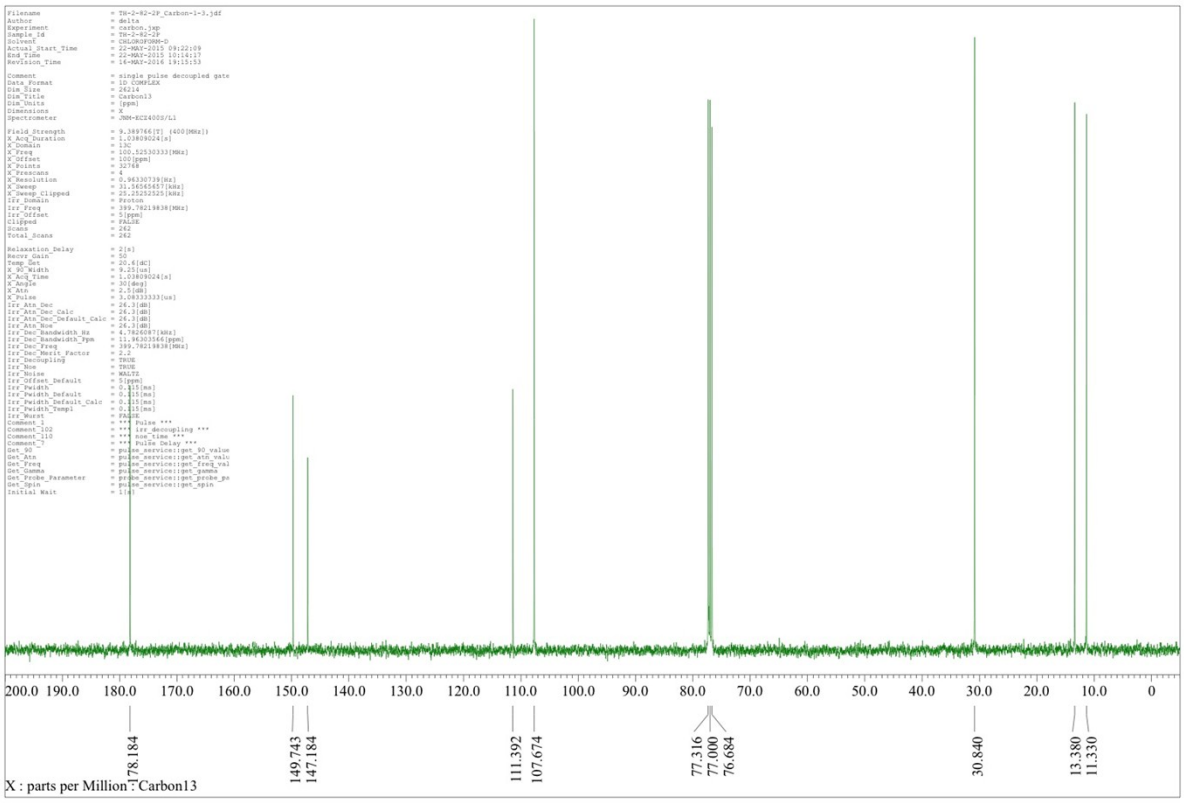
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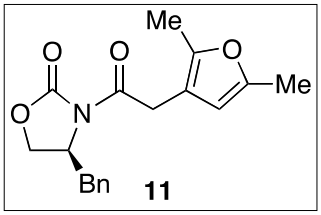
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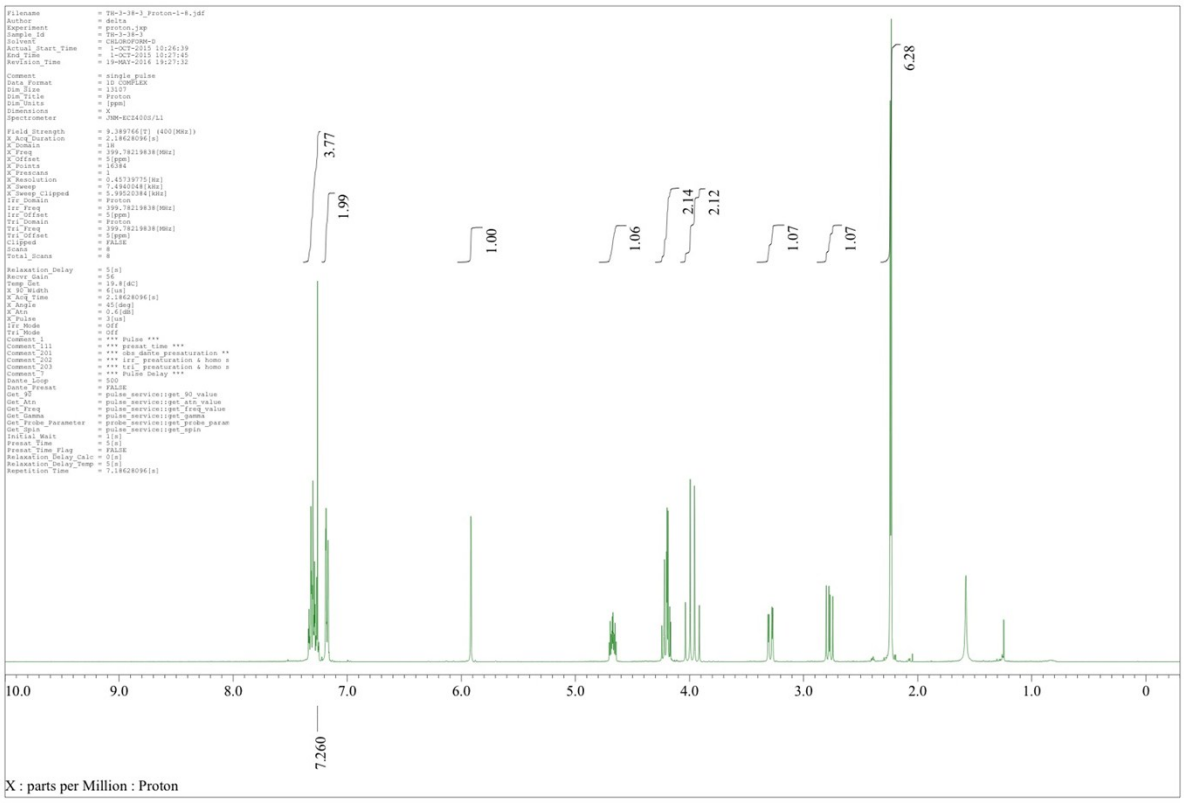
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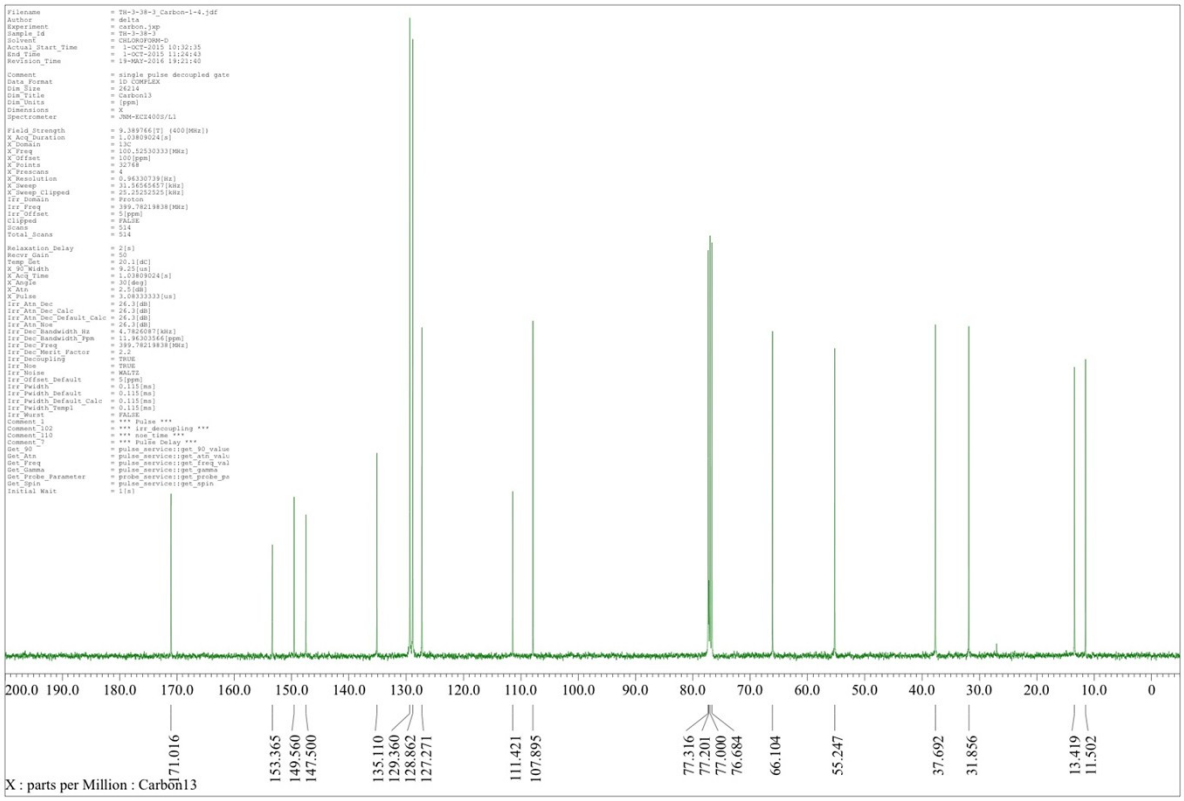
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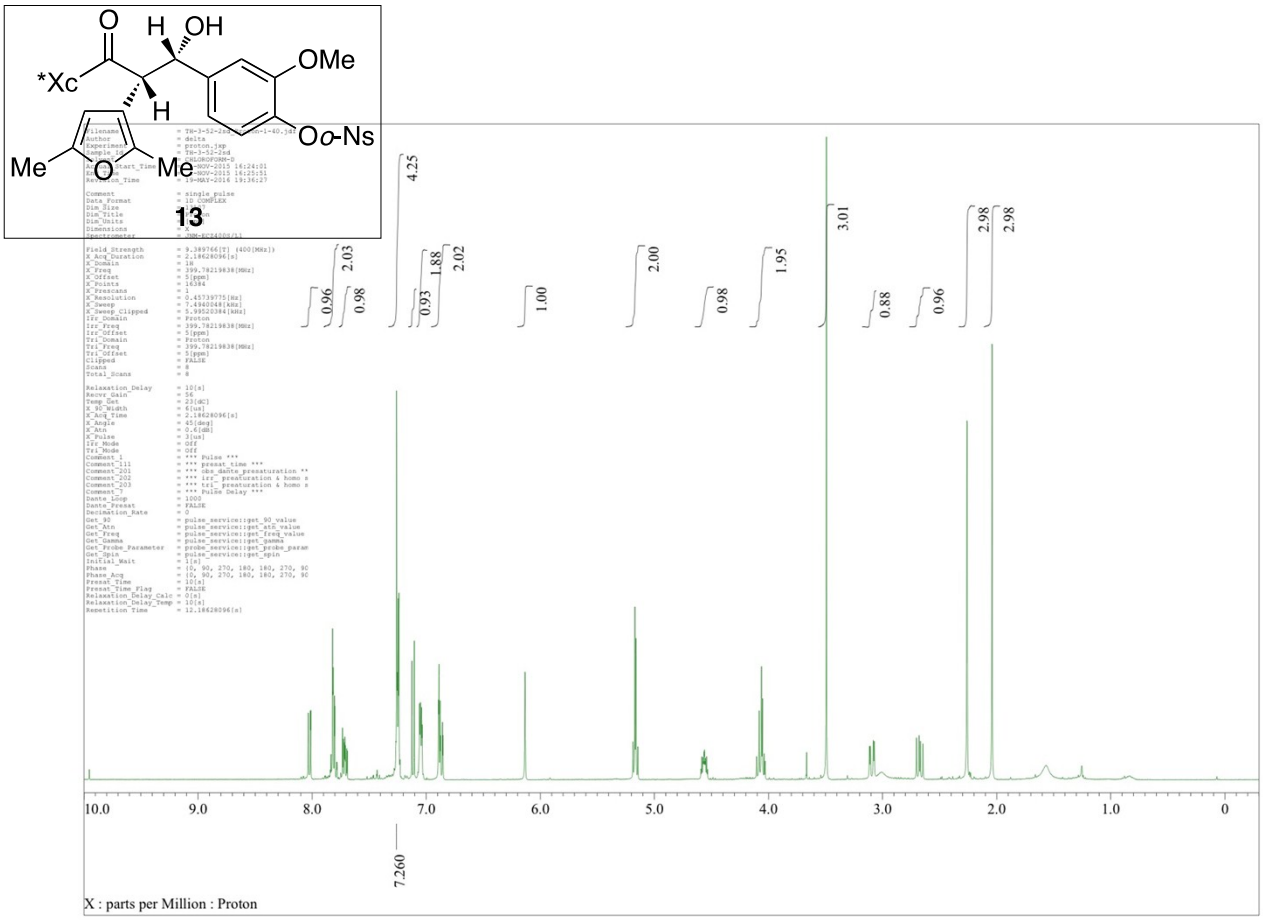
X : parts per Million : Carbon13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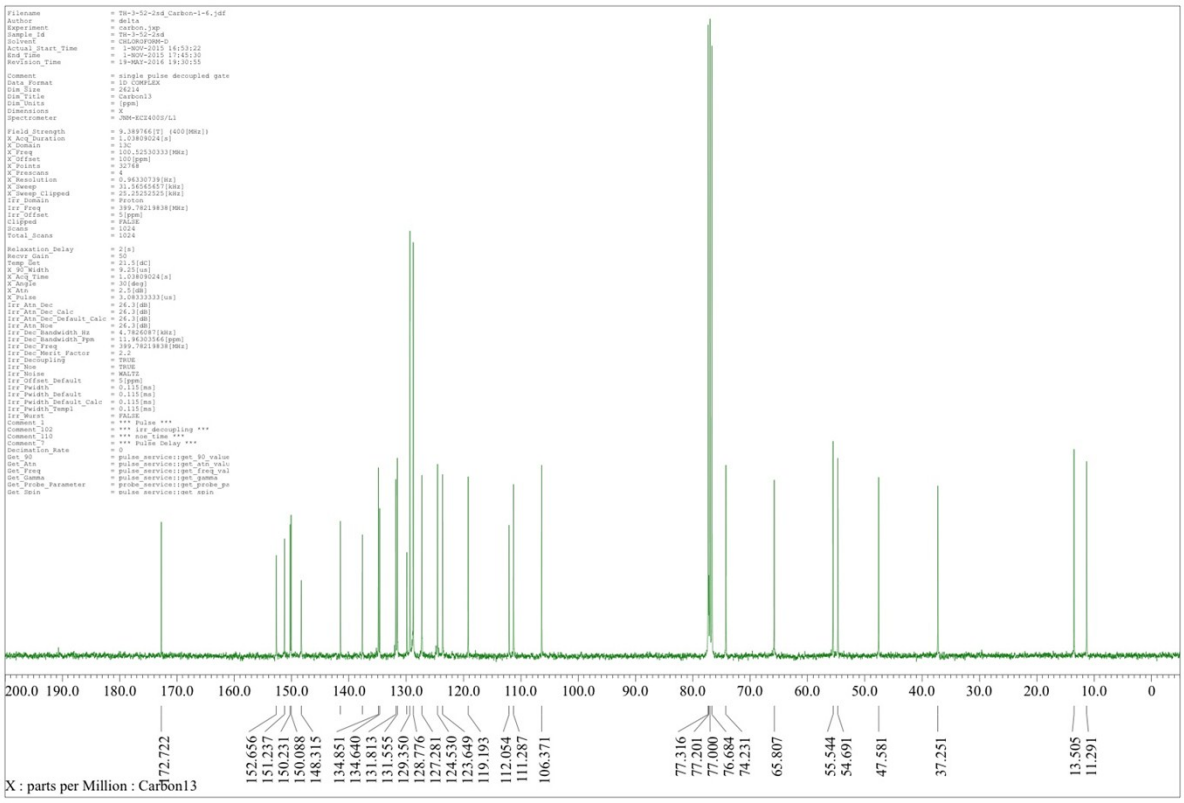
7.260
X : parts per Million : Proton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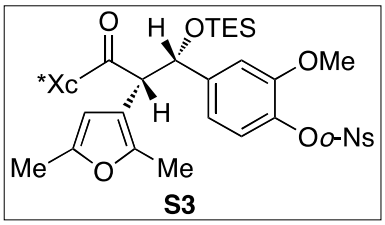
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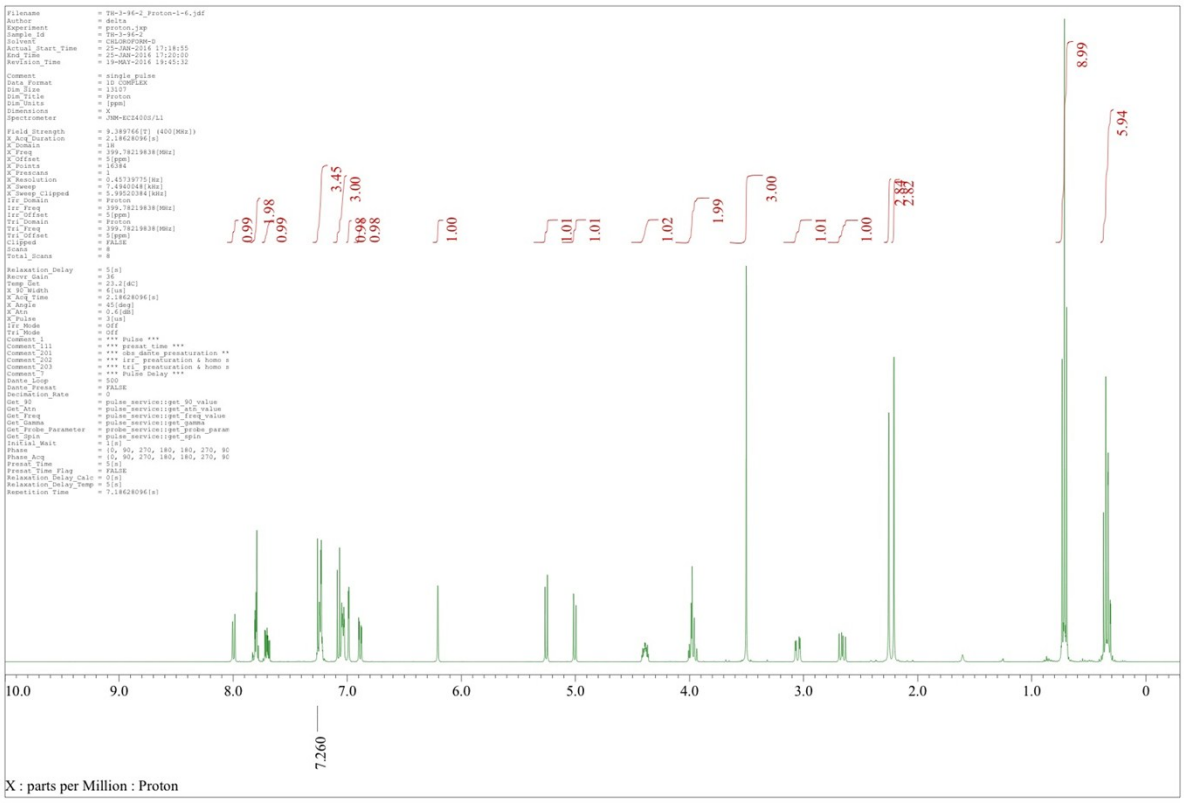
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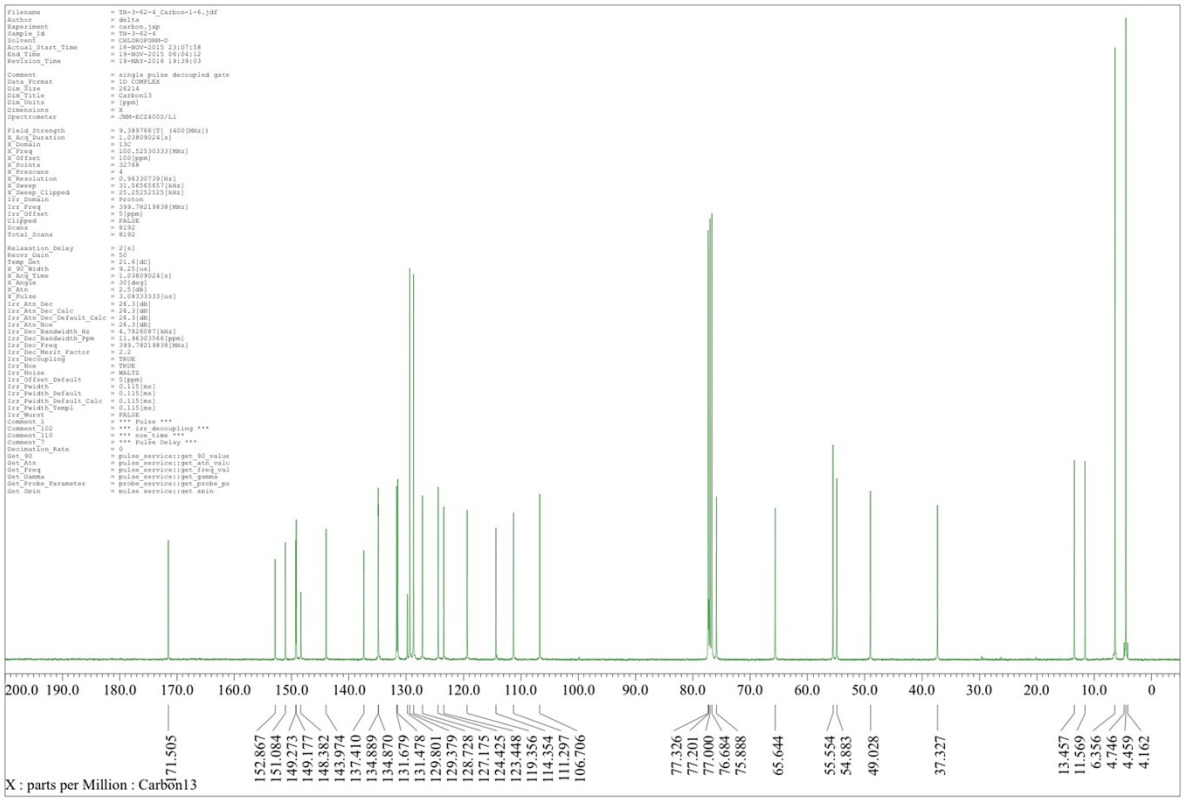
X : parts per Million : Proton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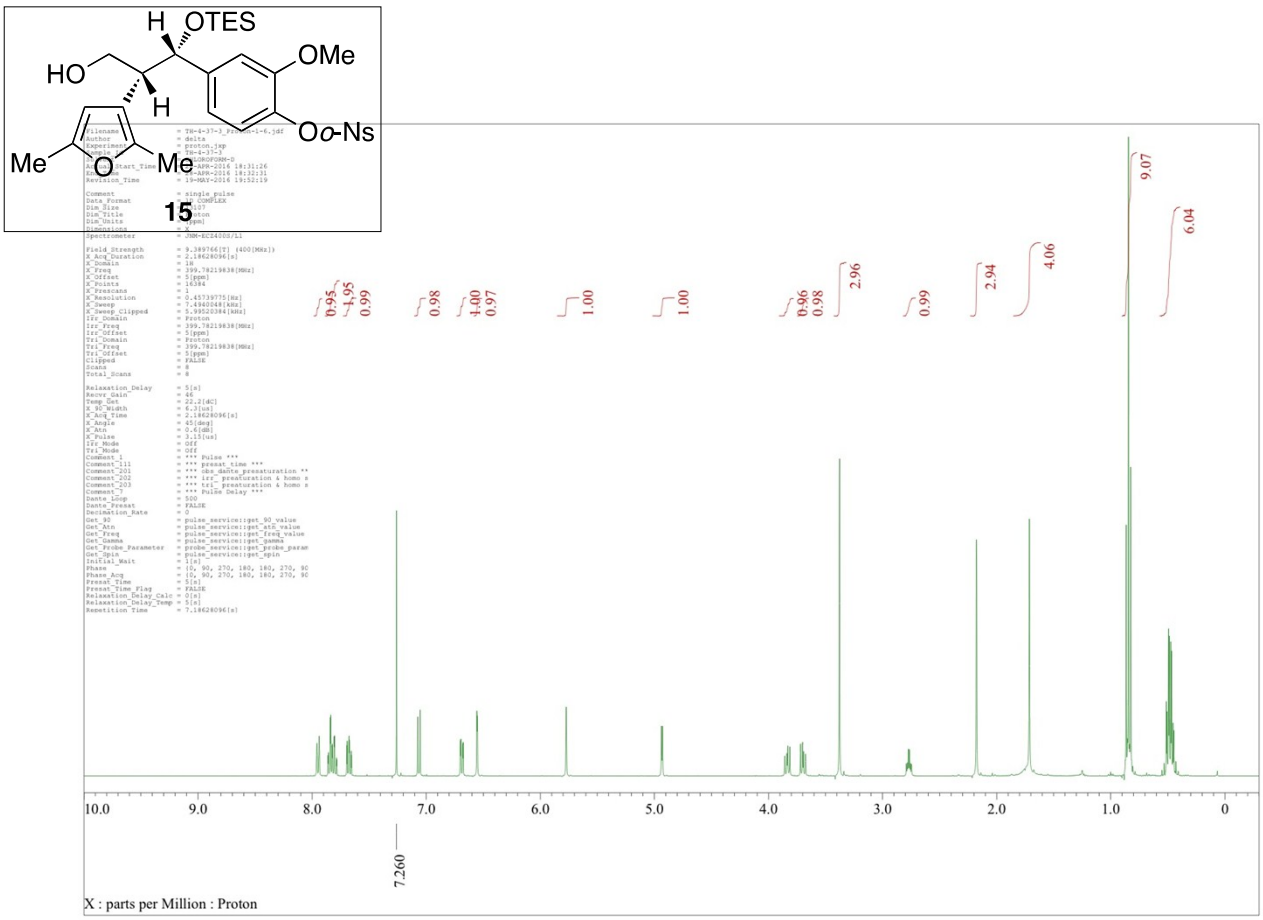
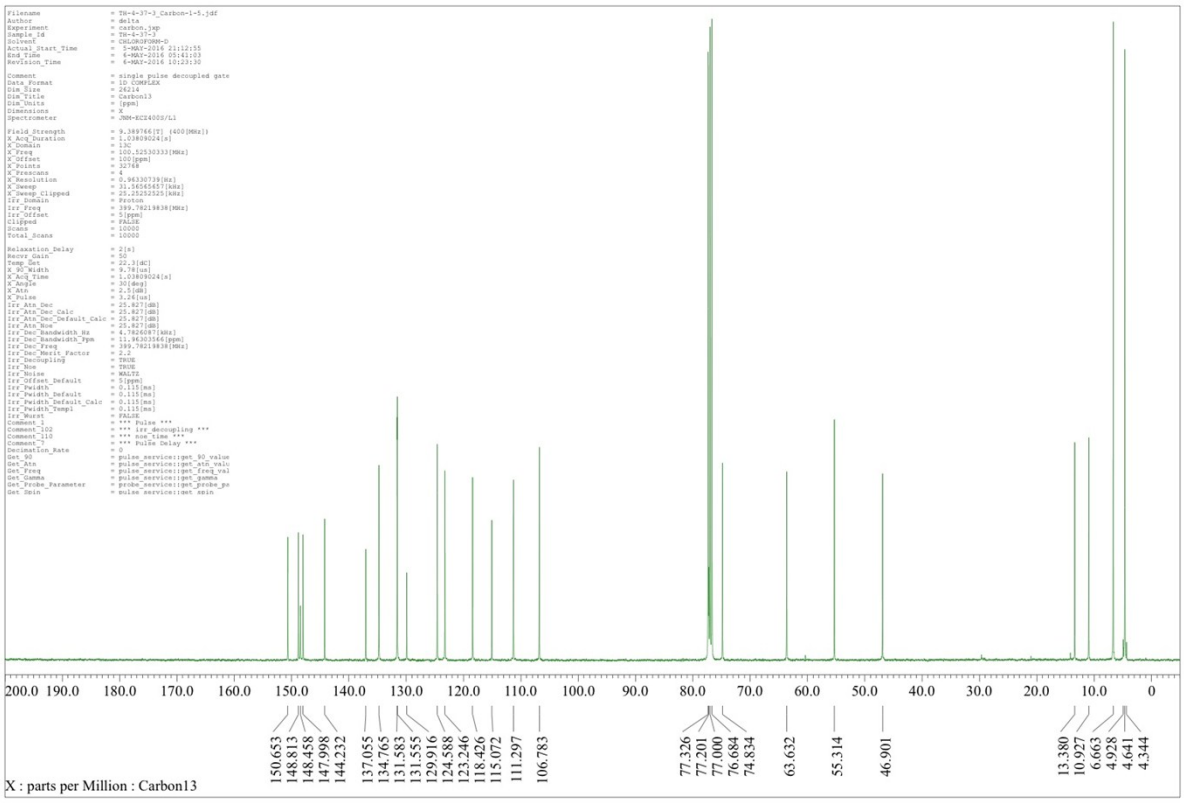
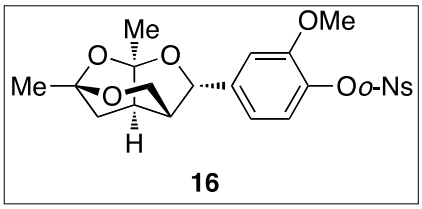
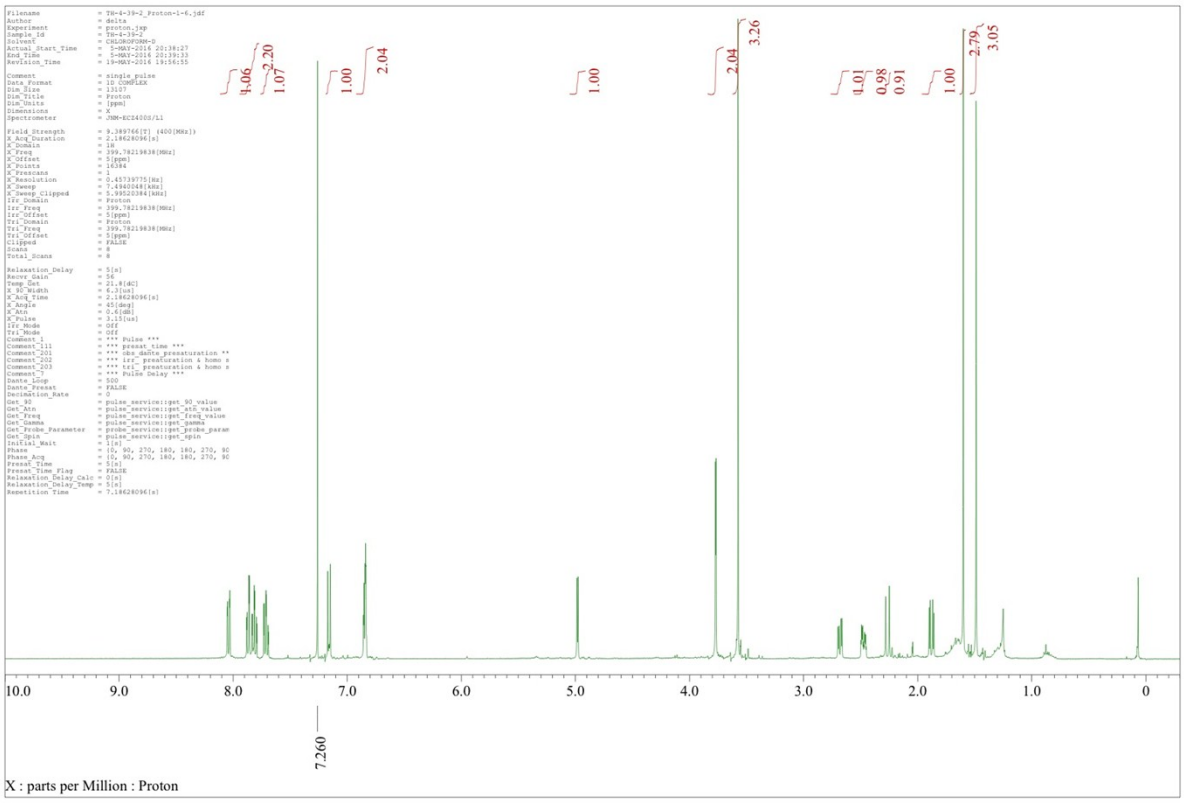
Decimation_Rate = 0
Get_90 = pulse_service::get_90_value
Get_Atn = pulse_service::get_atn_valu
Get_Freq = pulse_service::get_freq_val
Get_Gamma = pulse_service::get_gamma
Get_Probe_Parameter = probe_service::get_probe_pa
Get_Spin = pulse_service::get_spin
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
171.505
152.867
151.084
149.273
149.177
148.382
143.974
137.410
134.889
134.870
131.679
131.478
129.801
129.379
128.728
127.175
124.425
123.448
119.356
114.354
111.297
106.706
77.326
77.201
77.000
76.684
75.888
65.644
55.554
54.883
49.028
37.327
13.457
11.569
6.356
4.746
4.459
4.162
X : parts per Million : Carbon13

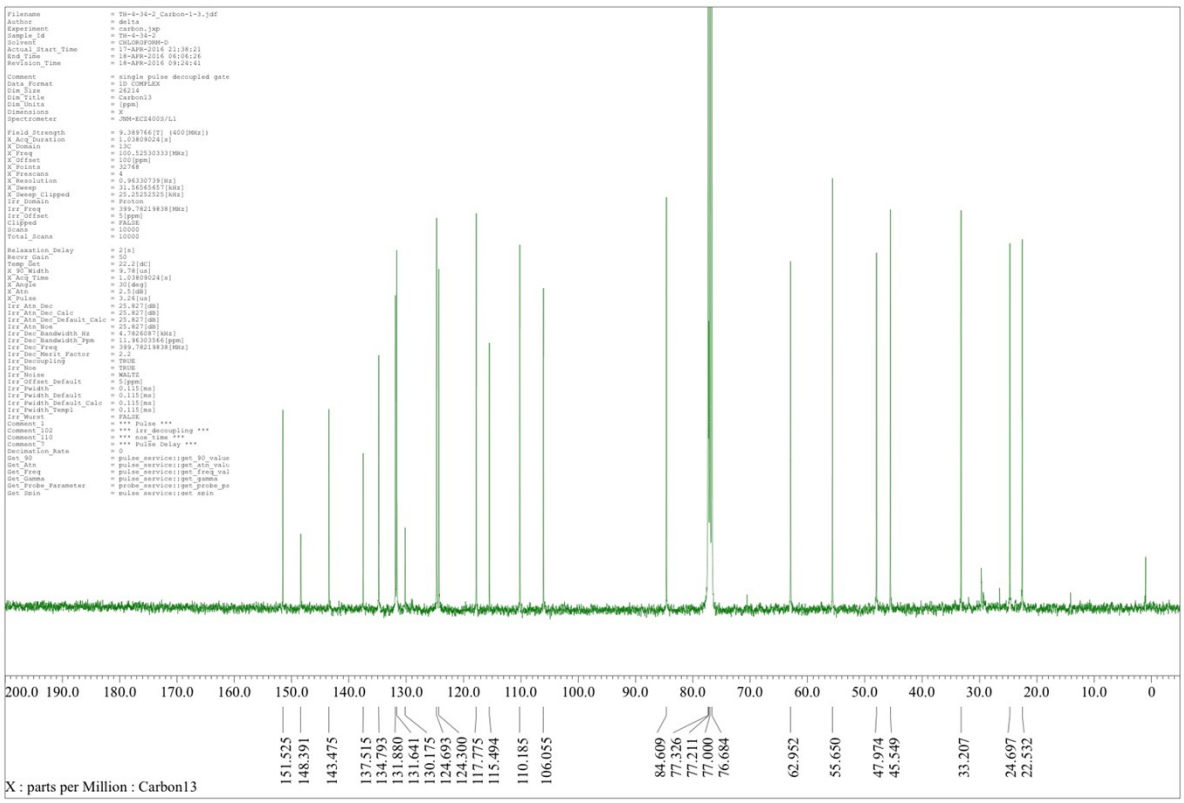

H
OTES
HO
OMe
H
Oo-Ns
Me
Me
O
15
0.95
1.95
0.99
0.98
4.00
0.97
1.00
1.00
0.96
0.98
2.96
0.99
2.94
4.06
9.07
6.04
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.260
X : parts per Million : Proton

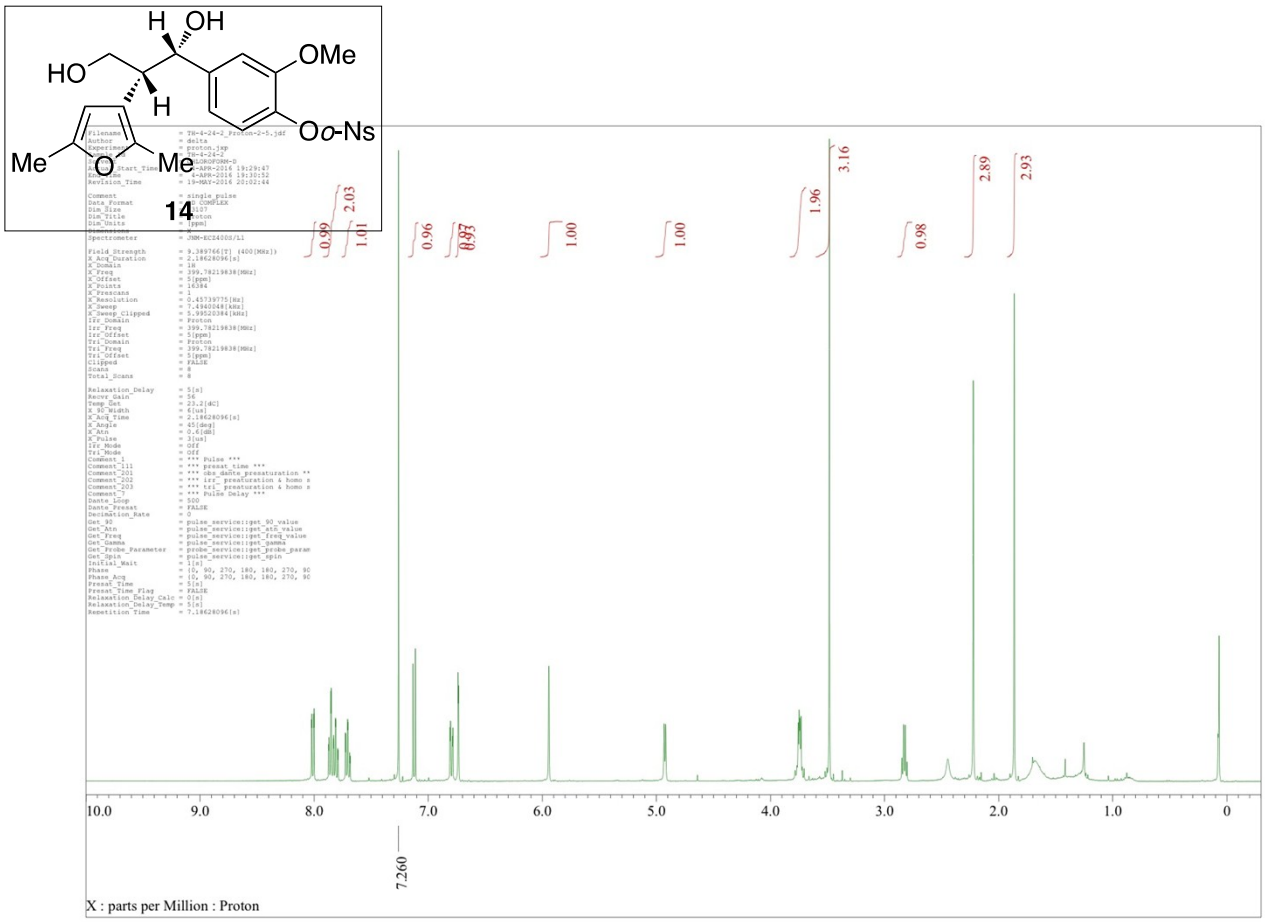

Filename = TN-4-37-3_Carbon-1-5.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = TN-4-37-3
Solvent = CHLOROFORM-D
Actual_Start_Time = 5-MAY-2016 21:12:55
End_Time = 6-MAY-2016 05:41:03
Revision_Time = 6-MAY-2016 10:23:30
Comment = single pulse decoupled gate
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-EC2400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 2
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565457[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 10000
Total_Scans = 10000
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 22.3[dC]
X_90_Width = 9.78[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 2.5[dB]
X_Pulse = 3.26[us]
Irr_Atn_Dec = 25.827[dB]
Irr_Atn_Dec_Calc = 25.827[dB]
Irr_Atn_Dec_Default_Calc = 25.827[dB]
Irr_Atn_Noe = 25.827[dB]
Irr_Dec_Bandwidth_Hz = 4.78260871[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303566[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_DecSupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Temp1 = 0.115[ms]
Irr_Worst = FALSE
Comment_1 = *** Pulse ***
Comment_102 = *** irr_decoupling ***
Comment_110 = *** noe_time ***
Comment_7 = *** Pulse Delay ***
Decimation_Rate = 0
Get_90 = pulse_service::get_90_value
Get_Atn = pulse_service::get_atn_valu
Get_Freq = pulse_service::get_freq_val
Get_Gamma = pulse_service::get_gamma
Get_Probe_Parameter = probe_service::get_probe_pa
Get_Spin = pulse_service::set_spin
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
150.653
148.813
148.458
147.998
144.232
137.055
134.765
131.583
131.555
129.916
124.588
123.246
118.426
115.072
111.297
106.783
77.326
77.201
77.000
76.684
74.834
63.632
55.314
46.901
13.380
10.927
6.663
4.928
4.641
4.344
X : parts per Million : Carbon13

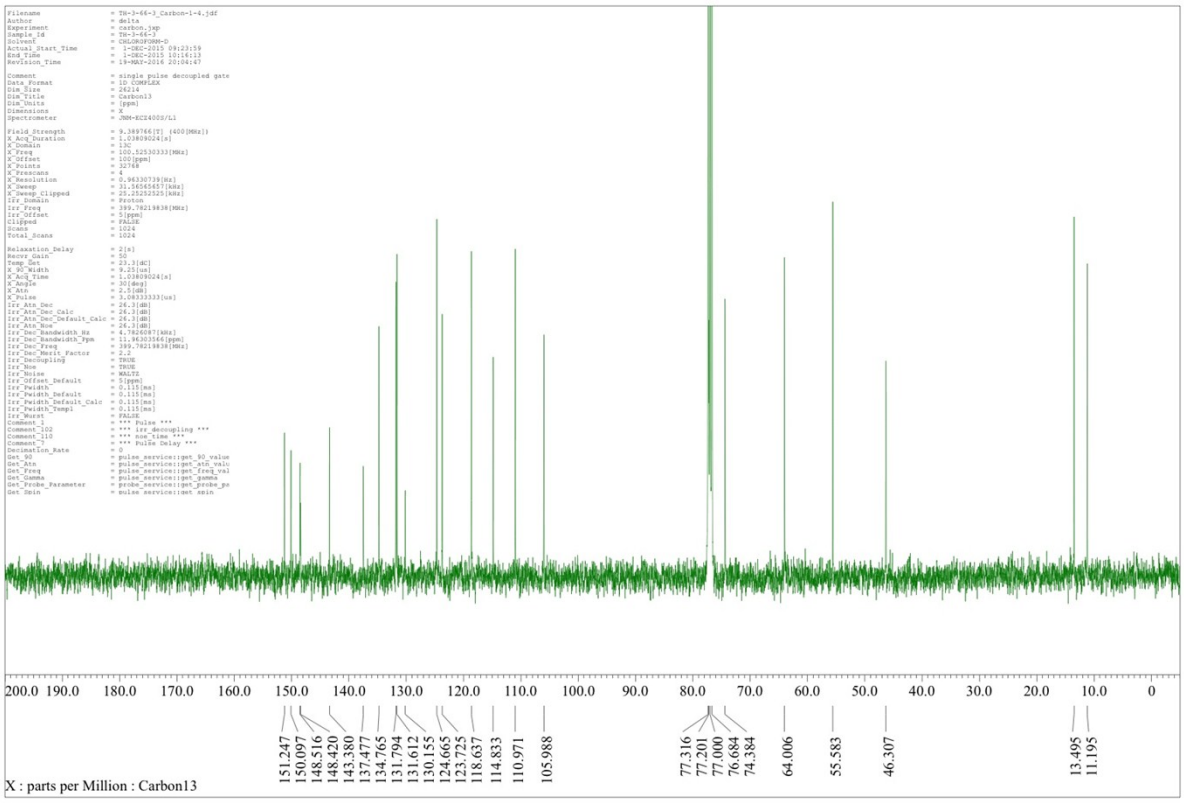

Me
O
O
Me
O
H
OMe
O*o*-Ns

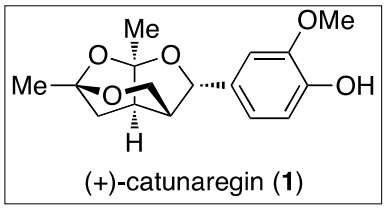
16


Filename = TN-4-39-2_Proton-1-6.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = TN-4-39-2
Solvent = CHLOROFORM-D
Actual_Start_Time = 5-MAY-2016 20:38:27
End_Time = 5-MAY-2016 20:39:33
Revision_Time = 19-MAY-2016 19:56:55
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-EC2400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 4
X_Resolution = 0.45739375[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 56
Temp_Get = 21.8[dC]
X_90_Width = 6.3[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.6[dB]
X_Pulse = 3.15[us]
Irr_Mode = Off
Tri_Mode = Off
Comment_1 = *** Pulse ***
Comment_111 = *** presat_time ***
Comment_201 = *** obs_dante_presaturation **
Comment_202 = *** irr_presfuration & homo s
Comment_203 = *** tri_ preaturation & homo s
Comment_7 = *** Pulse Delay ***
Dante_Loop = 500
Dante_Presat = FALSE
Decimation_Rate = 0
Get_90 = pulse_service::get_90_value
Get_Atn = pulse_service::get_atN_value
Get_Freq = pulse_service::get_freq_value
Get_Gamma = pulse_service::get_gamma
Get_Probe_Parameter = probe_service::get_probe_param
Get_Spin = pulse_service::get_spin
Initial_Wait = 1[s]
Phase = [0, 90, 270, 180, 180, 270, 90
Phase_Acq = [0, 90, 270, 180, 180, 270, 90
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]
1.06
2.20
1.07
1.00
2.04
1.00
2.04
3.26
1.01
0.98
0.91
1.00
2.79
3.05
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.260
X : parts per Million : Proton

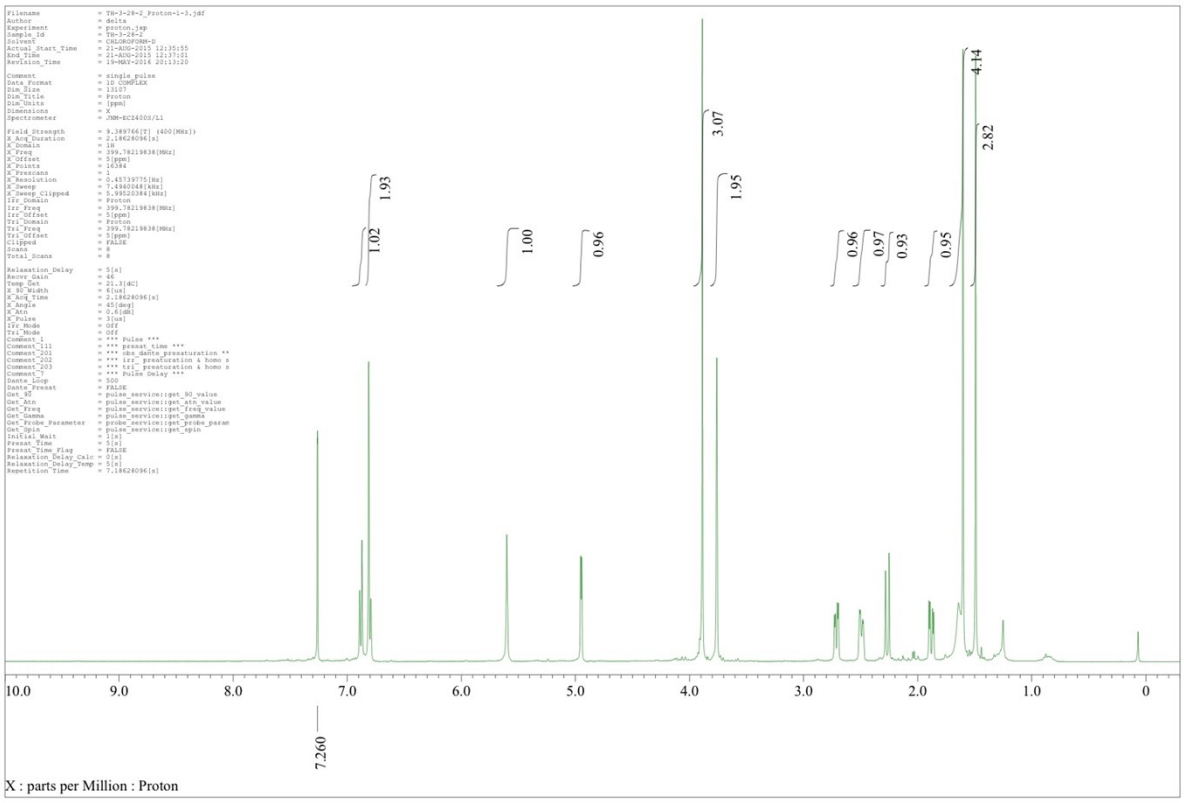

Filename = TN-4-34-2_Carbon-1-3.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = TN-4-34-2
Solvent = CHLOROFORM-D
Actual_Start_Time = 17-APR-2016 21:38:21
End_Time = 18-APR-2016 06:06:26
Revision_Time = 18-APR-2016 09:24:41
Comment = single pulse decoupled gate
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-EC2400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565457[kHz]
X_Sweep_Clipped = 25.25252325[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 10000
Total_Scans = 10000
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 22.2[dC]
X_90_Width = 9.78[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 30[deg]
X_Atn = 3.0[dB]
X_Pulse = 3.26[us]
Irr_Atn_Dec = 25.827[dB]
Irr_Atn_Dec_Calc = 25.827[dB]
Irr_Atn_Dec_Default_Calc = 25.827[dB]
Irr_Dec_Max = 25.827[dB]
Irr_Dec_Bandwidth_Hz = 4.7926087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96303966[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_DecDupling = TRUE
Irr_Mon = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Temp1 = 0.115[ms]
Irr_Wurst = FALSE
Comment_1 = *** Pulse ***
Comment_102 = *** irr_decoupling ***
Comment_110 = *** one_time ***
Comment_7 = *** Pulse Delay ***
Decimation_Rate = 0
Get_90 = pulse_service::get_90_value
Get_Atn = pulse_service::get_atn_valu
Get_Freq = pulse_service::get_freq_val
Get_Gamma = pulse_service::get_gamma
Get_Probe_Parameter = probe_service::get_probe_pa
Get_Spin = pulse_service::get_spin
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
151.525
148.391
143.475
137.515
134.793
131.880
131.641
130.175
124.693
124.300
117.775
115.494
110.185
106.055
84.609
77.326
77.211
77.000
76.684
62.952
55.650
47.974
45.549
33.207
24.697
22.532
X : parts per Million : Carbon13

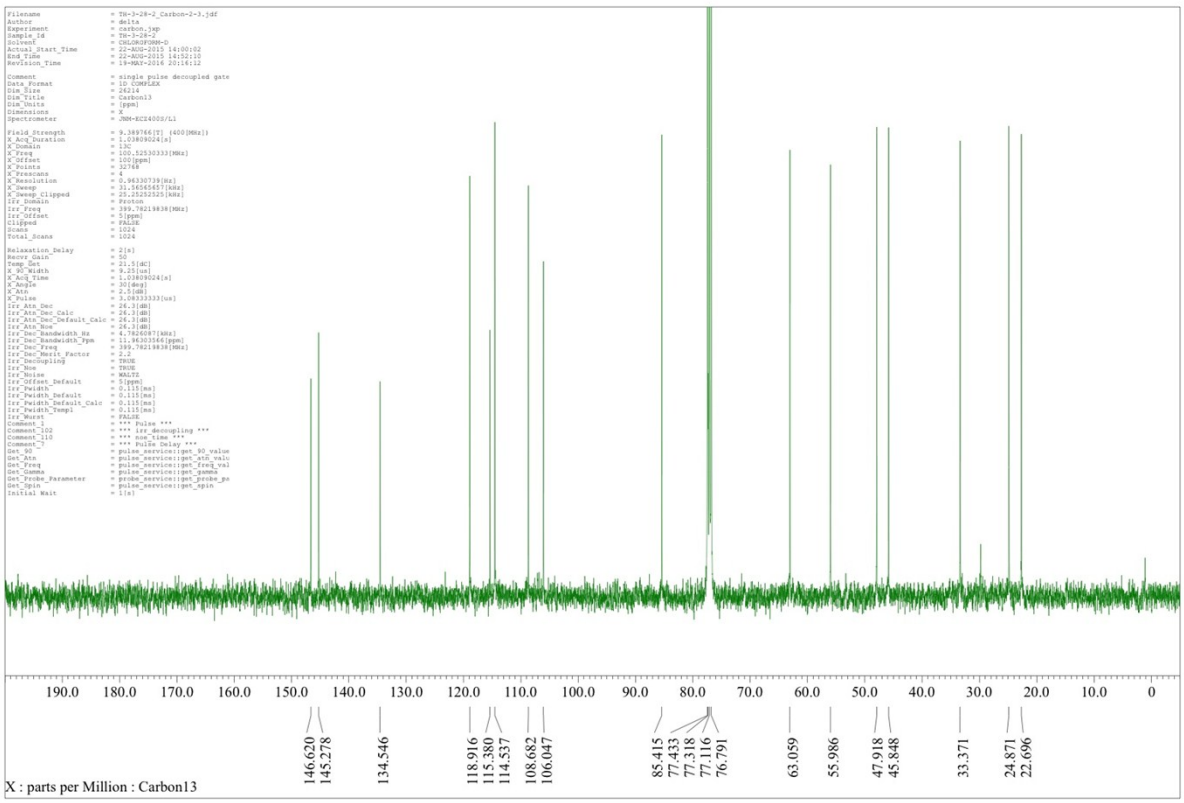

H
OH
HO
OMe
H
Oo-Ns
Me
Me
O
14
0.99
2.03
1.41
0.96
0.97
0.93
1.00
1.00
1.96
3.16
0.98
2.89
2.93
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.260
X : parts per Million : Proton

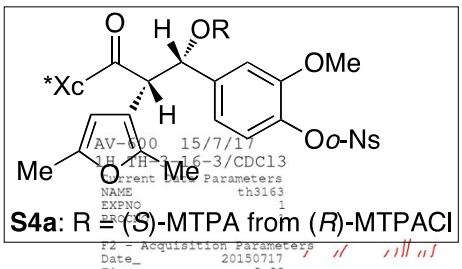




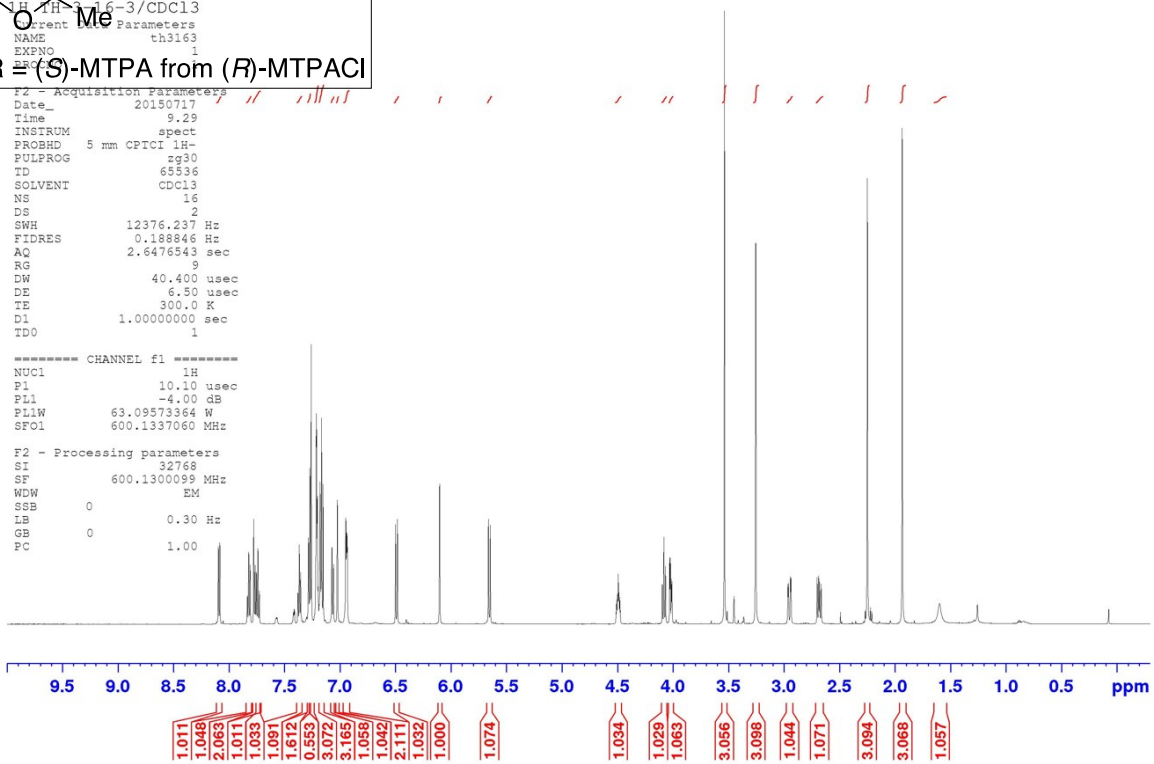
(+)-catunaregin (**1**)

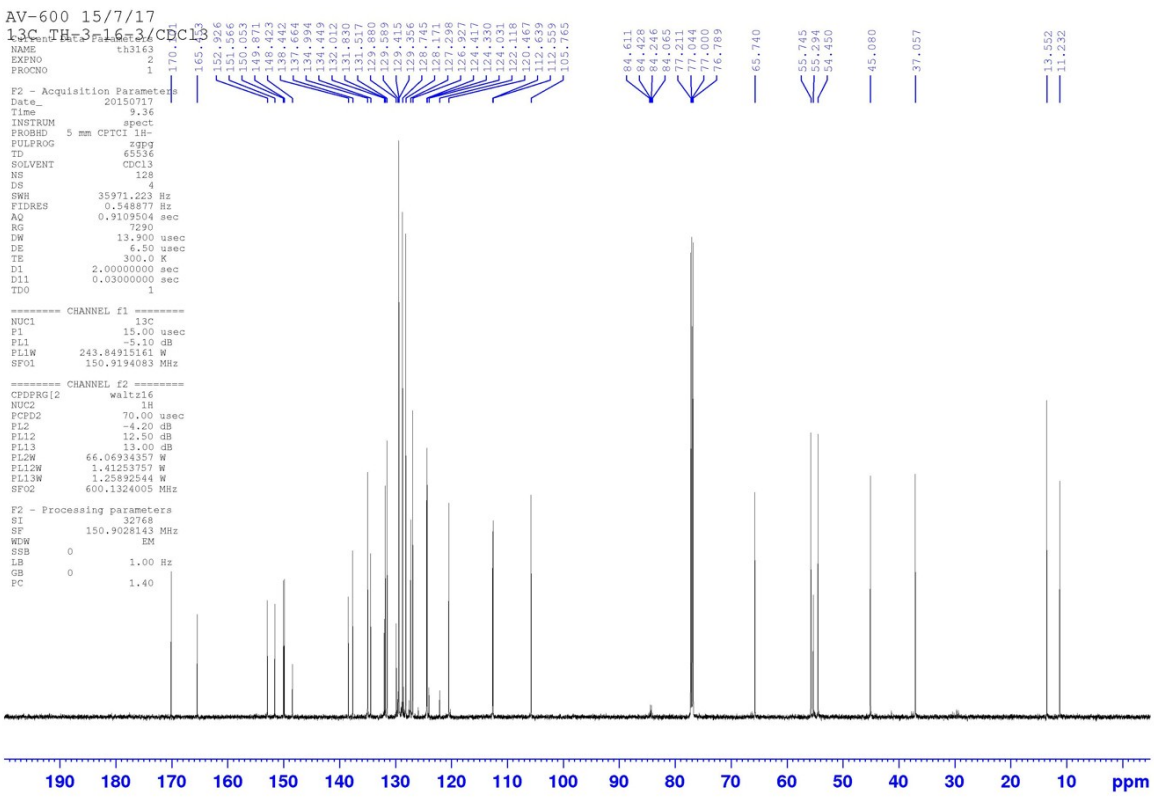

Filename = TH-3-28-2_Proton-1-3.jdf
Author = delta
Experiment = proton.jxp
Sample_Id = TH-3-28-2
Solvent = CHLOROFORM-D
Actual_Start_Time = 21-AUG-2015 12:35:55
End_Time = 21-AUG-2015 12:37:01
Revision_Time = 19-MAY-2016 20:13:20
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-EC2400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 2.18628096[s]
X_Domain = 1H
X_Freq = 399.78219838[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 8
X_Resolution = 0.45739375[Hz]
X_Sweep = 7.4940048[kHz]
X_Sweep_Clipped = 5.99020364[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 399.78219838[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 21.3[dC]
X_90_Width = 6[us]
X_Acq_Time = 2.18628096[s]
X_Angle = 45[deg]
X_Atn = 0.6[dB]
X_Pulse = 3[us]
Irr_Mode = Off
Tri_Mode = Off
Comment_1 = *** Pulse ***
Comment_111 = *** presat_time ***
Comment_201 = *** obs_daNte_presaturation **
Comment_202 = *** irr_ presfuration & homo s
Comment_203 = *** tri_ preaturation & homo s
Comment_7 = *** Pulse Delay ***
Dante_Loop = 500
Dante_Presat = FALSE
Get_90 = pulse_service::get_90_value
Get_Atn = pulse_service::get_atn_value
Get_Freq = pulse_service::get_freq_value
Get_Gamma = pulse_service::get_gamma
Get_Probe_Parameter = probe_service::get_probe_param
Get_Spin = pulse_service::get_spin
Initial_Wait = 1[s]
Presat_Time = 5[s]
Presat_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 5[s]
Repetition_Time = 7.18628096[s]
1.02
1.93
1.00
0.96
3.07
1.95
0.96
0.97
0.93
0.95
4.14
2.82
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.260
X : parts per Million : Proton

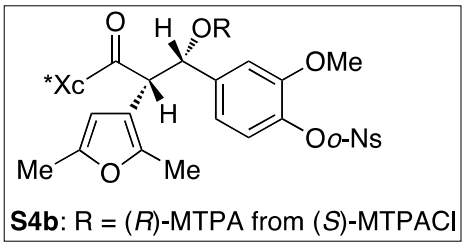

Filename = TH-3-28-2_Carbon-2-3.jdf
Author = delta
Experiment = carbon.jxp
Sample_Id = TH-3-28-2
Solvent = CHLOROFORM-D
Actual_Start_Time = 22-AUG-2015 14:00:02
End_Time = 22-AUG-2015 14:52:10
RevIsion_Time = 19-MAY-2016 20:16:12
Comment = single pulse decoupled gate
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-EC2400S/L1
Field_Strength = 9.389766[T] (400[MHz])
X_Acq_Duration = 1.03809024[s]
X_Domain = 13C
X_Freq = 100.52530333[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.96330739[Hz]
X_Sweep = 31.56565457[kHz]
X_Sweep_Clipped = 25.25252325[kHz]
Irr_Domain = Proton
Irr_Freq = 399.78219838[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 1024
Total_Scans = 1024
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.5[dC]
X_90_Width = 9.25[us]
X_Acq_Time = 1.03809024[s]
X_Angle = 20[deg]
X_Atn = 3.2[dB]
X_Pulse = 3.08333333[us]
Irr_Atn_Dec = 26.3[dB]
Irr_Atn_Dec_Calc = 26.3[dB]
Irr_Atn_Dec_Default_Calc = 26.3[dB]
Irr_Dec_Mode = GATED
Irr_Dec_Bandwidth_Hz = 4.7926087[kHz]
Irr_Dec_Bandwidth_Ppm = 11.96302366[ppm]
Irr_Dec_Freq = 399.78219838[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_DecDupling = TRUE
Irr_Mon = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 0.115[ms]
Irr_Pwidth_Default = 0.115[ms]
Irr_Pwidth_Default_Calc = 0.115[ms]
Irr_Pwidth_Temp1 = 0.115[ms]
Irr_Wurst = FALSE
Comment_1 = *** Pulse ***
Comment_102 = *** irr_decoupling ***
Comment_110 = *** one_time ***
Comment_7 = *** Pulse Delay ***
Get_90 = pulse_service::get_90_value
Get_Atn = pulse_service::get_atn_valu
Get_Freq = pulse_service::get_freq_val
Get_Gamma = pulse_service::get_gamma
Get_Probe_Parameter = probe_service::get_probe_pa
Get_Spin = pulse_service::get_spin
Initial Wait = 1[s]
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
146.620
145.278
134.546
118.916
115.380
114.537
108.682
106.047
85.415
77.433
77.318
77.116
76.791
63.059
55.986
47.918
45.848
33.371
24.871
22.696
X : parts per Million : Carbon13



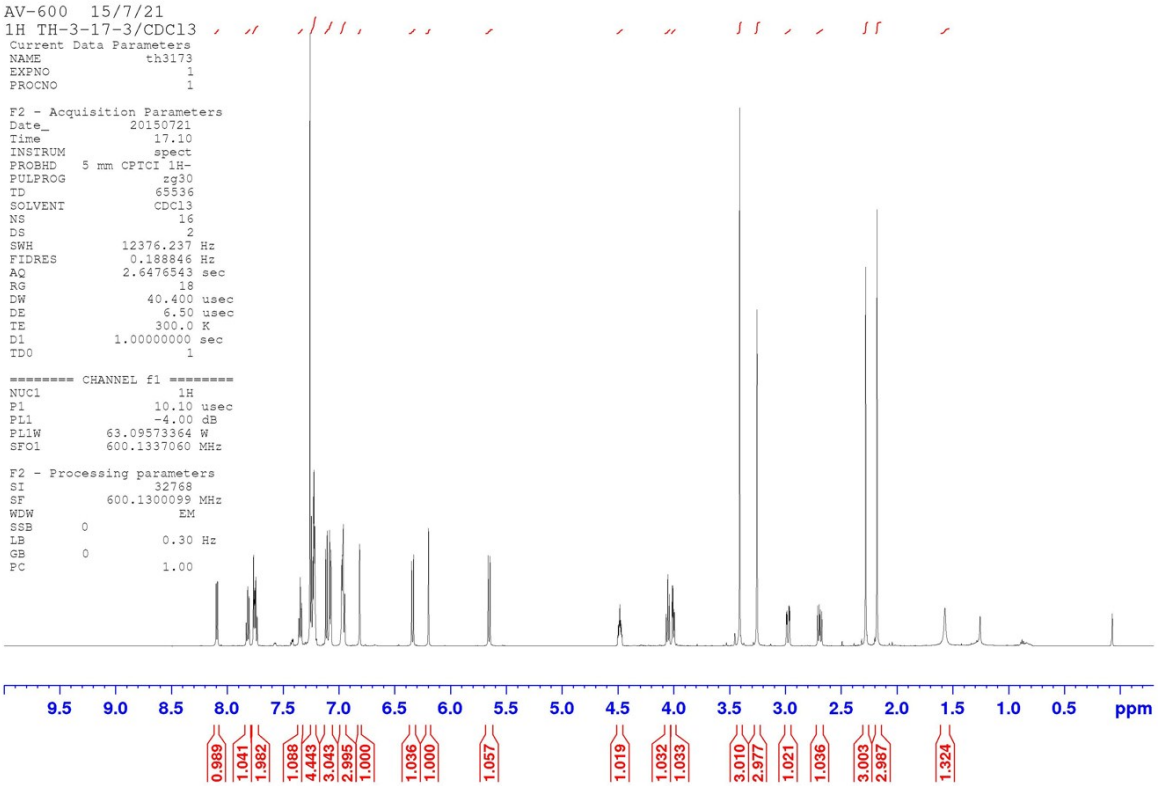
S4a: R = (*S*)-MTPA from (*R*)-MTPACl

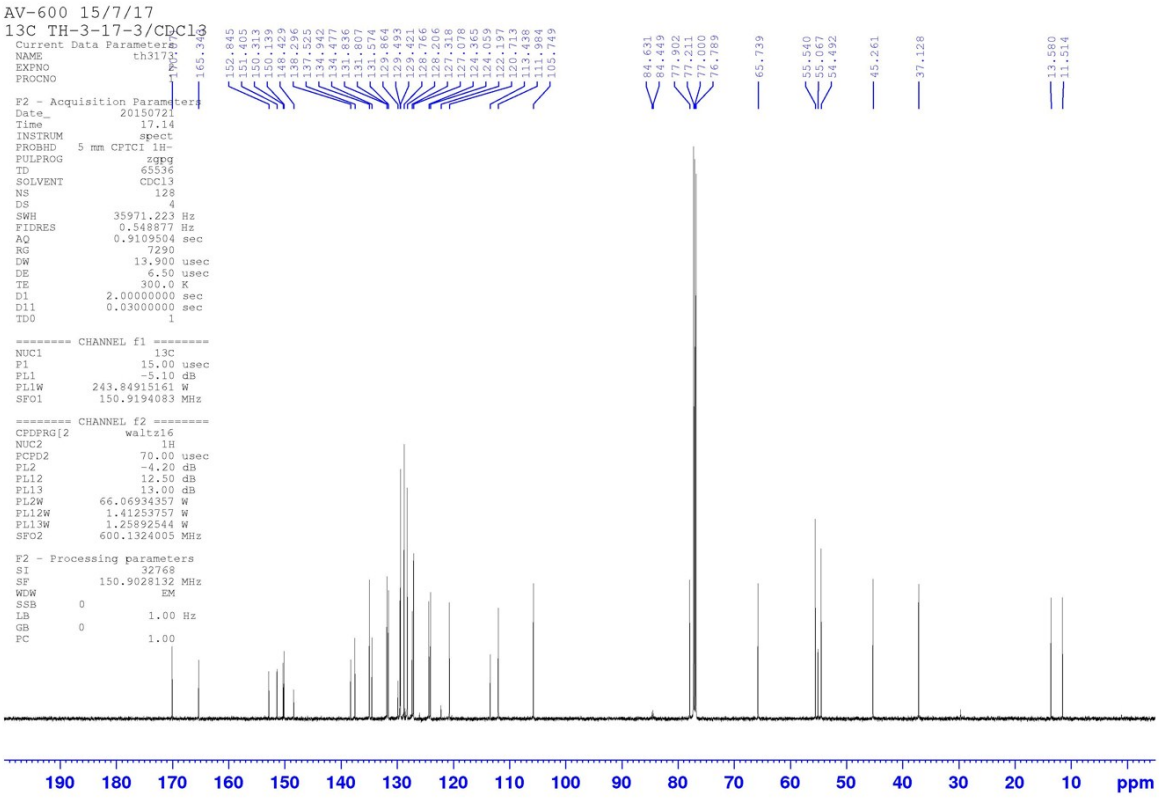






S4b: R = (*R*)-MTPA from (*S*)-MTPACl


AV-600 15/7/21
1H TH-3-17-3/CDCl3
Current Data Parameters
NAME th3173
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150721
Time 17.10
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12376.237 Hz
FIDRES 0.188846 Hz
AQ 2.6476543 sec
RG 18
DW 40.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.10 usec
PL1 -4.00 dB
PL1W 63.09573364 W
SFO1 600.1337060 MHz
F2 - Processing parameters
SI 32768
SF 600.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.989 1.041 1.982 1.088 4.443 3.043 2.995 1.000 1.036 1.000 1.057 1.019 1.032 1.033 3.010 2.977 1.021 1.036 3.003 2.987 1.324


AV-600 15/7/17
13C TH-3-17-3/CDCl3
Current Data Parameters
NAME th3173
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150721
Time 17.14
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 35971.223 Hz
FIDRES 0.548877 Hz
AQ 0.9109504 sec
RG 7290
DW 13.900 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.00 usec
PL1 -5.10 dB
PL1W 243.84915161 W
SFO1 150.9194083 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -4.20 dB
PL12 12.50 dB
PL13 13.00 dB
PL2W 66.06934357 W
PL12W 1.41253757 W
PL13W 1.25892544 W
SFO2 600.1324005 MHz
F2 - Processing parameters
SI 32768
SF 150.9028132 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
165.346
152.845 151.405 150.313 150.139 148.429 138.296 137.525 134.472 134.177 131.836 131.807 131.574 129.864 129.493 129.421 128.766 128.206 127.318 127.078 124.455 122.197 120.713 113.438 111.984 105.749
84.631 84.449 77.902 77.211 77.000 76.789
65.739
55.540 55.067 54.492
45.261
37.128
13.580 11.514
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm