

A Convenient Synthesis of Novel Sugar-lactam Hybrids Using Aubé Reaction

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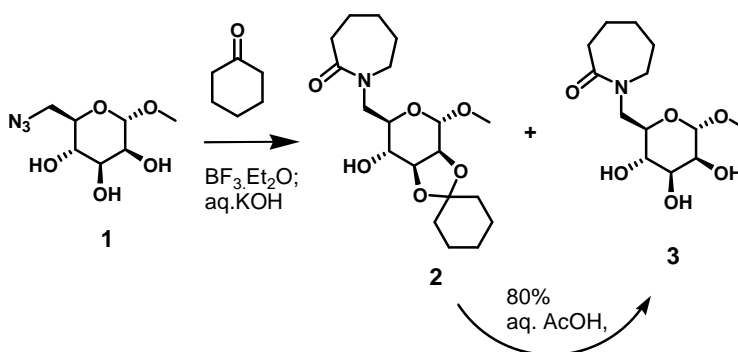
Supporting Information

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General:

All reagents, starting materials, and solvents (including dry solvents) were obtained from commercial suppliers and used as such without further purification. Reactions were carried out in oven-dried glassware under a positive pressure of argon unless otherwise mentioned. Column chromatography was performed on silica gel (Rankem, 100-200 mesh). Deuterated solvents (Cambridge Isotope Laboratories) for NMR spectroscopic analyses were used as received. All NMR spectra were recorded on Varion 400 MHz spectrometer. Coupling constants are measured in Hertz. All chemical shifts are quoted in ppm, relative to tetramethylsilane, using the residual solvent peak as a reference standard. Optical rotation was recorded from Rudolph autopol-V polarimeter at 589 nm (sodium D-line). Mass spectra were measured on a Agilent MSD/VL with ESI ionization. HRMS data was obtained from JEOL MS route 600H instrument. Infrared (IR) spectra were recorded on a Perkin-Elmer 100 FT-IR spectrometer.

Experimental details:

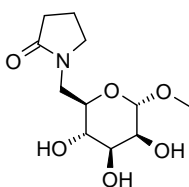


Methyl 6-deoxy-6-N-azepan-2-one- α -D-mannopyranoside (3). To the mixture of 6-Azido-6-deoxy-methyl- α -D-mannopyranoside **1**¹ (0.25g, 1.14 mmol) and cyclohexanone (0.18 mL, 3.50 mmol) in dichloromethane (5 mL), $\text{BF}_3 \cdot \text{Et}_2\text{O}$ (0.57mL, 4.57 mmol) was added drop wise under argon atmosphere at 0 °C. Reaction mixture was allowed to warm up to room temperature and stirring continued for 24 hours. The reaction mixture was diluted with diethyl ether (5 mL) and 50% aq. KOH (1 mL) was added. After stirring for additional one hour, reaction mixture was evaporated to dryness and purified by column chromatography using 30% ethyl acetate: hexane to neat ethyl acetate to get **2** and **3** in 230 mg and 36 mg respectively. Compound **2**: Mp = 89-91°C; $[\alpha]_D^{25} = -15.6^\circ$ (c 1, CH_3OH); IR (CHCl_3): 1071, 1099, 1623, 3368 cm^{-1} ; ^1H NMR

(CD₃OD, 400 MHz) δ 1.34-1.79 (series of m, 15H), 2.51-2.61 (m, 2H), 3.33 (d, $J = 3.6$ Hz, 1H), 3.36 (s, 3H), 3.49 (d, $J = 2.4$ Hz, 1H), 3.52-3.55 (m, 1H), 3.58-3.64 (m, 2H), 3.85 (d, $J = 4.80$ Hz, 1H), 3.88 (d, $J = 4.80$ Hz, 1H), 4.00 (dd, $J = 5.6, 7.6$ Hz, 1H), 4.09 (d, $J = 5.6$ Hz, 1H), 4.87 (s, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 179.9, 110.9, 99.6, 78.5, 76.5, 71.5, 70.6, 55.3, 53.1, 50.7, 39.2, 37.5, 36.4, 30.7, 28.7, 26.0, 25.0, 24.7, 24.5; LCMS = 370.2 (M+1); HRMS (ESI): m/z calculated for C₁₉H₃₁NO₆Na [M + Na]⁺ 392.2049, found 392.2096. Compound **3**: $[\alpha]^{25}_D = +24.4^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1610, 3392 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.64-1.82 (m, 6H), 2.55-2.58 (m, 2H), 3.34 (s, 3H), 3.47 (t, $J = 10$ Hz, 1H), 3.55-3.62 (m, 4H), 3.66 (dd, $J = 3.6, 9.2$ Hz, 1H), 3.76-3.81 (m, 2H), 4.58 (d, $J = 1.6$ Hz, 1H). ¹³C NMR (100.6 MHz, CD₃OD) δ 179.7, 102.9, 73.3, 71.8, 71.7, 69.6, 55.3, 53.1, 51.0, 37.6, 30.8, 28.7, 24.4; MS = 290 (M+1); HRMS (ESI): m/z calculated for C₁₃H₂₄NO₆ [M + H]⁺ 290.1603, found 290.1586.

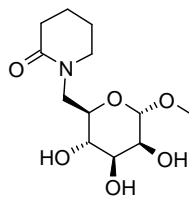
Conversion of compound 2 to compound 3: The compound **2** (230 mg) was dissolved in 80% aq. acetic acid (5 mL) and stirred at 80 °C. After 24 hours, the reaction mixture was evaporated to dryness to furnish the crude product. The crude compound was purified by column chromatography using silica gel and eluted with 50% ethyl acetate: hexane to neat ethyl acetate get 170 mg of titled product **3**. Overall yield: 205 mg. (64%).

Compounds **4-11** are prepared using the analogous procedure described for the synthesis of **3**.



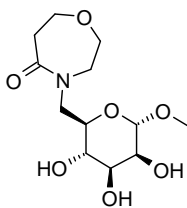
4

Methyl 6-deoxy-6-N-pyrrolidine-2-one- α -D-mannopyranoside (4). Yield 66%. $[\alpha]^{24.8}_D = +28.9^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1054, 1656, 3392 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 2.02-2.09 (m, 2H), 2.37-2.41 (m, 2H), 3.33 (s, 3H), 3.45 (t, $J = 9.2$ Hz, 1H), 3.56-3.68 (m, 6H), 3.75 (dd, $J = 1.2$ Hz, $J = 3.2$ Hz, 1H), 4.58 (s, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 178.3, 102.8, 72.6, 71.9, 71.8, 69.8, 55.2, 50.8, 45.2, 31.8, 19.2; LCMS = 262 (M+1); HRMS (ESI): m/z calculated for C₁₁H₁₉NO₆Na [M + Na]⁺ 284.1110, found 284.1101.



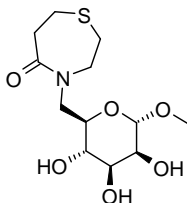
5

Methyl 6-deoxy-6-N-piperidine-2-one- α -D-mannopyranoside (5). Yield 64%; $[\alpha]^{23.9}_D = +50.9^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1054, 1602, 3411 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.77-1.86 (m, 4H), 2.34-2.40 (m, 2H), 3.31 (s, 3H), 3.46 (t, *J* = 9.60 Hz, 1H), 3.52-3.57 (m, 2H), 3.63 (d, *J* = 3.2 Hz, 1H), 3.65-3.70 (m, 3H), 3.77 (dd, *J* = 1.6, 3.2 Hz, 1H), 4.58 (d, *J* = 1.6 Hz, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 173.3, 102.8, 72.8, 71.9, 71.8, 70.0, 55.1, 51.6, 49.9, 32.8, 24.0, 21.9; LCMS = 276(M+1); HRMS (ESI): *m/z* calculated for C₁₂H₂₁NO₆Na [M + Na]⁺ 298.1266, found 298.1253.



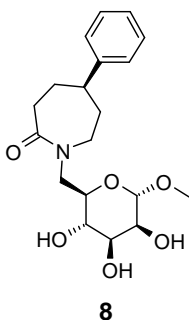
6

Methyl 6-deoxy-6-N-[1,4]oxazepan-5-one- α -D-mannopyranoside (6). Yield 52%; $[\alpha]^{25}_D = +21.4^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1491, 1619, 3392 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 2.70-2.88 (m, 2H), 3.34 (s, 3H), 3.51 (t, *J* = 9.60 Hz, 1H), 3.59-3.90 (series of m, 11H), 4.59 (d, *J* = 1.60 Hz, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 178.0, 102.9, 73.3, 71.7, 71.5, 70.9, 69.5, 65.9, 55.4, 55.1, 51.0, 41.7; LCMS = 292.0 (M+1); HRMS (ESI): *m/z* calculated for C₁₂H₂₁NO₇Na [M+Na]⁺ 314.1215, found 314.1264.

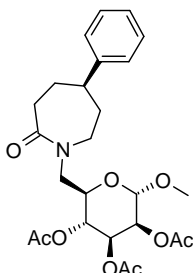


7

Methyl 6-deoxy-6-*N*-[1,4]thiazepan-5-one- α -D-mannopyranoside (7). Yield 57%; $[\alpha]^{23.7}_D = +18.8^\circ$ (*c* 0.5, CH₃OH); IR (CHCl₃): 1425, 1623, 3369.0 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 2.66-2.80 (m, 4H), 2.90-2.97 (m, 2H), 2.99 (t, *J* = 5.6 Hz, 1H), 3.34 (s, 3H), 3.34-3.50 (m, 2H), 3.58-3.68 (m, 2H), 3.77-3.84 (m, 1H), 3.91-3.95 (m, 2H), 4.60 (s, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 177.9, 103.0, 73.3, 71.8, 71.7, 69.6, 55.3, 55.1, 51.1, 41.2, 29.8, 24.6; LCMS = 308(M+1); HRMS (ESI) : *m/z* calculated for C₁₂H₂₂NO₆S [M + H]⁺ 308.1167, found 308.1182.



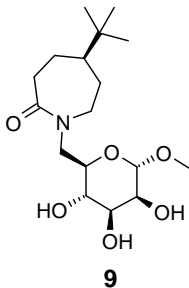
Methyl 6-deoxy-6-*N*-(5*R*)-phenyl-azepan-2-one- α -D-mannopyranoside (8). Yield 40%; Mp = 63-65°C (fused); $[\alpha]^{22.2}_D = 4.84^\circ$ (*c* 0.5, CH₃OH); IR (CHCl₃): 1619, 3412 cm⁻¹; ¹H NMR(CD₃OD, 400 MHz) δ 1.70 (q, *J* = 12.8, 1H), 1.83-1.90 (m, 1H), 1.94-2.00 (m, 1H), 2.25 (q, *J* = 12.8 Hz, 1H), 2.55 (dd, *J* = 7.2, 13.6 Hz, 1H), 2.77-2.89 (m, 2H), 3.34 (s, 3H), 3.45 (d, *J* = 14.4 Hz, 1H), 3.53-3.61 (m, 3H), 3.70-3.71 (m, 1H), 3.81-3.82 (m, 1H), 3.84 (d, *J* = 4.0 Hz, 1H), 4.08 (dd, *J* = 11.2, 3.76 Hz, 1H), 4.61 (s, 1H), 7.12-7.26 (m, 5H); ¹³C NMR (100.6 MHz, CD₃OD) δ 179.4, 147.9, 129.4 (2C), 128.0 (2C), 127.2, 103.0, 74.0, 71.8, 71.5, 69.1, 55.2, 52.7, 50.8, 49.5, 37.0, 35.9, 32.4; LCMS = 366.1 (M+1); HRMS (ESI): *m/z* calculated for C₁₉H₂₇NO₆ Na [M + Na]⁺ 388.1736, found 388.1739.



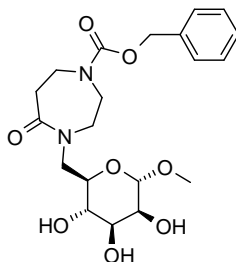
Methyl 6-deoxy-2,3,4-tri-*O*-acetyl-6-*N*-(5*R*)-phenyl-azepan-2-one- α -D-mannopyranoside.

To the mixture of **(8)** (0.3g, 0.82 mmol), and pyridine (0.7 mL, 6.57 mmol) in dichloromethane

(5 mL), Ac₂O (0.53mL, 6.57 mmol) and DMAP (5 mg, 0.041 mmol) was added at room temperature. Reaction mixture was stirred for 16 hours. The reaction mixture was diluted with ice water (20 mL) followed by extraction using dichloromethane (3X20 mL), combined organic layer was washed with 1N HCl (2X10mL), brine (20 mL) and dried over sodium sulfate. The solvent was evaporated under reduced pressures to yield the crude product which was purified by column chromatography followed by crystallization in hot ethanol (10 mL) to furnish the title compound (160 mg); Yield 40% (after recrystallization); Mp = 192-194°C; $[\alpha]^{22.8}_D = 64.9^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1084, 1135, 1222, 1648, 1753 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.75-1.93 (m, 3H), 1.94 (s, 3H), 1.99 (s, 3H), 2.01-2.08 (m, 1H), 2.11 (s, 3H), 2.62-2.68 (m, 2H), 2.72-2.82 (m, 1H), 3.35 (s, 3H), 3.59-3.72 (m, 4H), 3.96-4.10 (m, 1H), 4.62 (s, 1H), 5.16-5.21 (m, 2H), 5.28-5.31 (m, 1H), 7.19-7.25 (m, 3H), 7.27-7.32 (m, 2H); ¹³C NMR (100.6 MHz, CDCl₃) δ 175.9, 170.6, 170.2, 170.1, 146.5, 128.9 (2C), 127.0 (2C), 126.8, 98.8, 71.1, 70.0, 69.5, 67.2, 55.3, 50.6, 48.6, 48.5, 26.9, 36.8, 30.3, 21.1, 20.9, 20.9. LCMS = 492.3 (M+1).

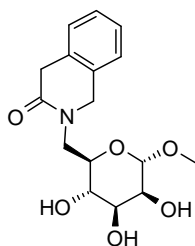


Methyl 6-deoxy-6-N-(5R)-tert-butylazepan-2-one-α-D-mannopyranoside (9). Yield 52%; Mp = 159-161°C; $[\alpha]^{22.6}_D = 26.2^\circ$ (*c* 0.5, CH₃OH); IR (CHCl₃): 1457.7, 1618.6, 3392 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 0.89 (s, 9H), 1.21-1.33 (m, 3H), 1.55 (m, 2H), 1.80-1.96 (m, 1H), 2.00-2.06 (m, 1H), 2.49 (dd, *J* = 7.2, 13.2 Hz, 1H), 2.61 (t, *J* = 12 Hz, 1H), 3.33 (s, 3H), 3.47-3.68 (m, 4H), 3.76-3.78 (m, 1H), 3.89 (dd, *J* = 4.8, 14.4 Hz, 1H), 4.60 (d, *J* = 1.2 Hz, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 179.7, 103.0, 73.7, 71.8, 71.6, 69.3, 55.2, 52.8, 52.7, 50.7, 36.8, 33.8, 29.9, 28.0 (3-C), 25.3; LCMS = 346 (M+1); HRMS (ESI) : *m/z* calculated for C₁₇H₃₂NO₆ [M+H]⁺ 346.2229, found 346.2201.



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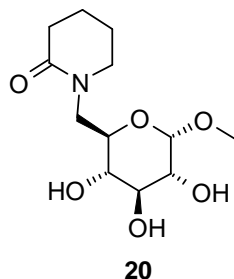
Methyl 6-deoxy-6-N-[1,4]diazepane-1-carboxylic acid benzyl ester azepan-5-one- α -D-mannopyranoside (10). Yield 40%; $[\alpha]^{23.6}_D = +25.6^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1241, 1431, 1626, 1697, 3392 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 2.69-2.73 (m, 2H), 3.32 (s, 3H), 3.34-3.35 (m, 1H), 3.44-3.54 (m, 2H), 3.58-3.69 (m, 5H), 3.76 (dd, *J* = 2.0, 3.6 Hz, 1H), 3.80-3.87 (m, 3H), 4.59 (s, 1H), 5.13 (s, 2H), 7.30-7.35 (m, 5H); ¹³C NMR (100.6 MHz, CD₃OD) δ 177.5, 157.0, 137.9, 129.5 (2C), 129.2, 129.0 (2C), 102.9, 73.3, 71.7, 71.5, 69.4, 68.6, 55.3, 53.5, 51.1, 47.8, 42.6, 39.5; LCMS = 425 (M+1); HRMS (ESI) : *m/z* calculated for C₂₀H₂₉N₂O₈ [M + H]⁺ 425.1924, found 425.1909.



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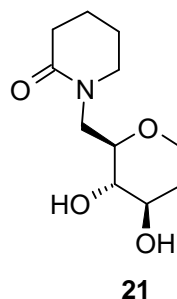
Methyl-6-deoxy-6-N-1,4-dihydro-2H-isoquinolin-3-one- α -D-mannopyranoside (11). Yield = 10%; $[\alpha]^{23.3}_D = +37.5^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1377, 1668, 3392 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 3.08 (s, 3H), 3.46-3.50 (m, 1H), 3.55-3.75(m, 5H), 3.97 (d, *J* = 12 Hz, 1H), 4.55 (d, *J* = 1.2 Hz, 1H), 4.71 (d, *J* = 5.6 Hz, 2H), 7.18-7.25 (m, 5H); ¹³C NMR (100.6 MHz, CD₃OD) δ 172.8, 133.9, 133.4, 128.5, 127.9, 127.7, 126.2, 102.7, 72.9, 72.0, 71.9, 70.1, 55.0, 54.1, 38.5, 30.7; LCMS = 324 (M+1).

Compounds **20**, **21**, **22**, **23**, **25** and **26** are prepared using the analogous procedure (1st step) described for the synthesis of **3**.



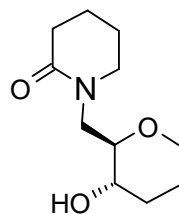
Methyl 6-deoxy-6-N-piperidine-2-one- α -D-glucopyranoside (20).

Yield 66%; Mp = 197-199°C; $[\alpha]^{24.8}_D = +69.6^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1048, 1602, 3338 cm⁻¹; ¹H NMR (DMSO-d₆, 400 MHz) δ 1.64-1.72 (m, 4H), 1.98- 2.60 (m, 3H), 2.88 (ddd, *J* = 4.4, 9.6, 5.5 Hz, 1H), 3.12- 3.20 (m, 3H), 3.21 (s, 3H), 3.92-3.42 (m, 1H), 3.52-3.57 (m, 1H), 3.71 (dd, *J* = 1.6, 14.0 Hz, 1H), 4.50 (d, *J* = 4.0 Hz, 1H), 4.75 (br s, 1H), 4.82 (br s, 1H), 5.01 (d, *J* = 4.4 Hz, 1H); ¹³C NMR (100.6 MHz, DMSO-d-6) δ 168.9, 99.6, 72.7, 72.2, 71.8, 70.1, 54.1, 49.3, 48.1, 31.8, 22.8, 20.8; LCMS = 276.1 (M+1); HRMS (ESI) : *m/z* calculated for C₁₂H₂₂NO₆ [M+H]⁺ 276.1447, found 276.1434.



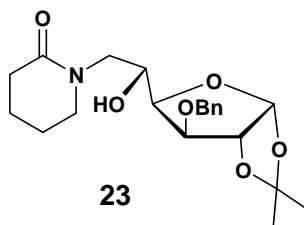
1-((2R,3S,4R)-3, 4-Dihydroxy-tetrahydro-pyran-2-ylmethyl)-piperidin-2-one (21).

Yield 65%; $[\alpha]^{23.7}_D = -16.78^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1089, 1608, 3365 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.50-1.61 (m, 1H), 1.79 (t, *J* = 2.8 Hz, 4H), 1.87 (dd, *J* = 5.6, 13.2 Hz, 1H), 2.34-2.42 (m, 2H), 2.96 (t, *J* = 7.2, 1H), 3.24-3.43 (m, 3H), 3.50-3.55 (m, 3H), 3.78 (dd, *J* = 8.4, 14.8 Hz, 1H), 3.84-3.90 (m, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 173.6, 81.1, 74.9, 73.0, 66.8, 51.4, 50.0, 35.0, 32.7, 23.9, 21.9; LCMS = 230.3 (M+1); HRMS (ESI): *m/z* calculated for C₁₁H₂₀NO₄[M + H]⁺ 230.1392, found 230.1390.



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1-((2R,3S)-3-Hydroxy-tetrahydro-pyran-2-ylmethyl)-piperidin-2-one (22). Yield 60%; $[\alpha]^{24.2}_D = -35.8^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1092, 1617, 3369 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.38-1.50 (m, 2H), 1.56-1.68 (m, 2H), 1.74-1.84 (m, 4H), 2.06-2.12 (m, 1H), 2.42-2.48 (m, 2H), 2.90 (d, *J* = 14.4 Hz, 1H), 3.12-3.31 (m, 3H), 3.64-3.72 (m, 1H), 3.84-3.86 (m, 1H), 4.43 (d, *J* = 14.4 Hz, 1H), 5.20 (br s, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 172.3, 83.8, 68.5, 66.4, 51.2, 49.2, 32.1, 31.1, 26.0, 23.4, 21.4; LCMS = 214.2 (M+1); HRMS (ESI): *m/z* calculated for C₁₁H₂₀NO₃ [M + H]⁺ 214.1443, found 214.1456.

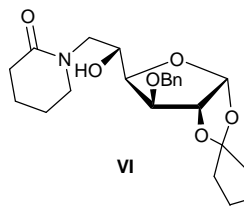


23

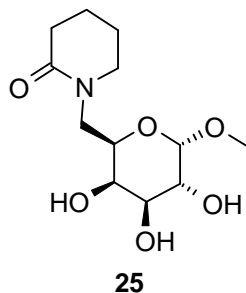
6-N-(piperidine-2-one)-3-O-benzyl-6-deoxy-1, 2-O-isopropylidene- α -D-glucofuranoside (23).

To minimize side product (VI) only 3 equivalents of BF₃·Et₂O was used. Yield 52%; $[\alpha]^{24.6}_D = -2.16^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 1074, 1614, 3287 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.29 (s, 3H), 1.44 (s, 3H), 1.76-1.84 (m, 4H), 2.36 (t, *J* = 8.0 Hz, 2H), 3.31-3.38 (m, 3H), 3.49-3.53 (m, 1H), 3.80 (dd, *J* = 3.2, 14.4 Hz, 1H), 3.96 (dd, *J* = 2.8, 8.8 Hz, 1H), 4.03 (d, *J* = 3.2 Hz, 1H), 4.18 (ddd, *J* = 3.2, 8.8, 12.0 Hz, 1H), 4.60-4.69 (m, 2H), 5.86 (d, *J* = 3.2 Hz, 1H), 7.27-7.38 (m, 5H); ¹³C NMR (100.6 MHz, CD₃OD) δ 174.1, 139.3, 129.3 (2C), 129.0 (2C), 128.8, 112.7, 106.6, 83.3, 83.0, 82.7, 73.3, 68.0, 53.7, 51.3, 32.8, 27.1, 26.4, 24.0, 21.8; LCMS = 392.3 (M+1); HRMS (ESI): *m/z* calculated for C₂₁H₃₀NO₆ [M+H]⁺ 392.2073, found 392.2043.

Along with **23**, other compound **VI** (13%) was also isolated and the tentative structure is shown below. Tentative structure assigned as drawn below for the minor product based on following spectral data.

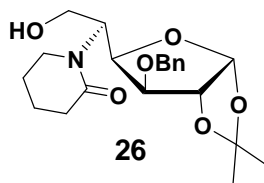


Yield 13%; IR (CHCl₃): 1078, 1615, 3306 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 0.82-0.94 (m, 4H), 1.69-1.94 (m, 5H), 2.18-2.24 (m, 1H), 2.40-2.44 (m, 2H), 3.01-3.04 (m, 1H), 3.32-3.35 (m, 1H), 3.48-3.57 (m, 3H), 3.78 (dd, *J* = 1.2, 14.4 Hz, 1H), 4.02 (dd, *J* = 2.8, 9.2 Hz, 1H), 4.10-4.1.5 (m, 2H), 4.68 (q, *J* = 15.2 Hz, 2H), 5.34 (br s, 1H), 5.85 (d, *J* = 3.6 Hz, 1H), 7.26- 7.38 (m, 5H); ¹³C NMR(100.6 MHz, CD₃OD) δ 173.9, 138.2, 128.7 (2C), 128.1 (2C), 128.0, 121.7, 105.2, 83.2, 82.0, 81.1, 73.1, 69.0, 54.1, 50.9, 37.4, 36.8, 32.3, 23.8, 23.4, 23.2, 21.2; LCMS = 418.0 (M+1).



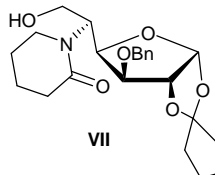
Methyl 6-deoxy-6-*N*-piperidine-2-one- α -D-galctopyranoside (25).

The compound **25** was synthesized by following the analogous procedure described for the synthesis of **20**, However, during hydrolysis (2nd step) excess of aq. 50% KOH (for 180 mg reaction 1.5 mL) was used and stirred for 6 hours. Yield 51%; [α]^{24.4}_D = +2.68° (*c* 1, CH₃OH); IR (CHCl₃): 1048, 1216, 1618, 3620 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.78-1.84 (m, 4H), 2.35 (t, *J* = 6.0 Hz, 2H), 3.20 (dd, *J* = 8.8, 13.6 Hz, 1H), 3.34 (s, 3H), 3.36-3.44 (m, 1H), 3.56-3.64 (m, 1H), 3.67-3.77 (m, 3H), 3.85 (dd, *J* = 4.8, 14.0 Hz, 1H), 4.01 (dd, *J* = 4.8, 8.4 Hz, 1H), 4.68 (d, *J* = 3.6, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 172.9, 101.4, 71.2, 71.1, 69.9, 69.6, 55.4, 51.4, 50.0, 32.8, 24.1, 22.0; LCMS = 276.1 (M+1); HRMS (ESI): *m/z* calculated for C₁₂H₂₁NO₆Na [M+Na]⁺ 298.1266, found 298.1260.



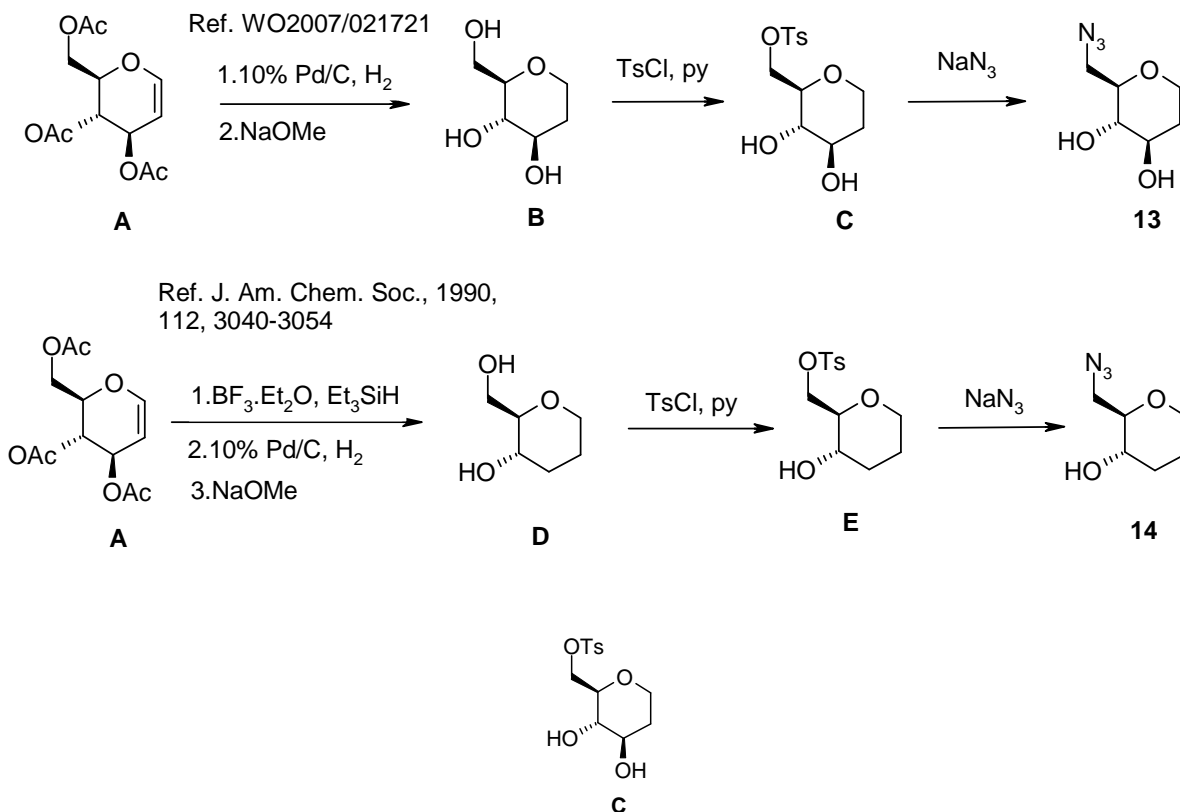
5-N-(piperidine-2-one)-3-O-benzyl-5-deoxy-1, 2-O-isopropylidene- α -D-glucofuranoside (26).

To minimize side product **VII** only 3 equivalents of $\text{BF}_3 \cdot \text{Et}_2\text{O}$ was used. Yield 52%; $[\alpha]^{23.2}_{\text{D}} = -8.98^\circ$ (*c* 1, CH_3OH); IR (CHCl_3): 1163, 1496, 3390 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 1.30 (s, 3H), 1.44 (s, 3H), 1.71-1.80 (m, 4H), 2.35 (t, $J = 6.4$ Hz, 2H), 3.34-3.40 (m, 1H), 3.46-3.52 (m, 2H), 3.58 (dd, $J = 2.8, 11.2$ Hz, 1H), 3.70-3.77 (m, 1H), 3.93 (bs, 1H), 4.49-4.58 (m, 2H), 4.73 (d, $J = 11.2$ Hz, 1H), 4.77 (d, $J = 3.6$ Hz, 1H), 5.86 (d, $J = 4.0$ Hz, 1H), 7.29-7.39 (m, 5H); ^{13}C NMR (100.6 MHz, CD_3OD) δ 173.6, 138.7, 129.4 (2C), 129.4, (2C), 129.0, 112.7, 105.8, 83.1, 82.6, 77.3, 72.6, 60.7, 33.4, 27.0, 26.4, 24.2, 23.9, 21.4 (2C); LCMS = 392.3 (M+1). Along with **26**, other compound **VII** (21%) also isolated and the tentative structure is shown below. Tentative structure assigned as drawn below for the minor product based on following spectral data.



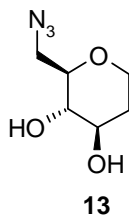
$[\alpha]^{23.3}_{\text{D}} = -4.00^\circ$ (*c* 0.5, CH_3OH); IR (CHCl_3): 1336, 1120, 1615, 3399 cm^{-1} ; ^1H NMR (CD_3OD , 400 MHz) δ 1.64-1.92 (series of m, 13H), 2.36 (t, $J = 6.4$ Hz, 2H), 3.37-3.39 (m, 1H), 3.46-3.56 (m, 1H), 3.57 (dd, $J = 2.8, 11.2$ Hz, 1H), 3.66-3.77 (m, 1H), 3.95 (d, $J = 2.4$ Hz, 1H), 4.49-4.58 (m, 2H), 4.69-4.74 (m, 2H), 5.83 (d, $J = 4.0$ Hz, 1H), 7.28-7.39 (m, 5H); ^{13}C NMR (100.6 MHz, CD_3OD) δ 173.5, 138.7, 129.4 (2C), 129.3 (2C), 129.0, 122.3, 105.5, 83.2, 82.6, 77.4, 72.6, 60.8, 49.8, 37.8, 37.2, 33.4, 24.4 (2C), 23.9, 23.8, 21.4; LCMS = 418.3 (M+1).

Synthesis of deoxy sugar azides:



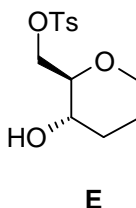
Toluene-4-sulfonic acid (2R,3S,4R)-3,4-dihydroxy-tetrahydro-pyran-2-ylmethyl ester (C).²

To a solution of (2R, 3S, 4R)-2-ethyl-tetrahydro-pyran-3, 4-diol³ (3.0g, 20.8 mmol) in pyridine (20 mL), *p*-toluenesulfonyl chloride (4.3 g, 22.9 mmol) was added and reaction mixture allowed to stir for overnight at room temperature. Reaction mixture was diluted with cold water and neutralized with 1N HCl (~pH 6) followed by extraction with dichloromethane (3X30 mL). Combined organic layer was washed with brine and dried over sodium sulfate. After evaporation of the solvent furnished the desired compound **C** (3.0g). Yield 48%; IR (CHCl₃): 669, 771, 1215, 3019 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.46-1.55 (m, 1H), 1.85 (dd, *J* = 4.8, 12.8 Hz, 1H), 1.98 (d, *J* = 9.6 Hz, 1H), 2.45 (s, 3H), 3.05 (t, *J* = 9.2 Hz, 1H), 3.22 (m, 1H), 3.35-3.47 (m, 1H), 3.80 (dd, *J* = 5.2, 10.8 Hz, 1H), 4.10 (dd, *J* = 5.2, 10.8 Hz, 1H), 4.30 (dd, *J* = 1.2, 10.4 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 2H), 7.78 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (100.6 MHz, CD₃OD) δ 146.4, 134.4, 130.9 (2C), 129.0 (2C), 79.4, 73.8, 72.9, 71.3, 66.5, 34.8, 21.5; LCMS = 303.0 (M+1).



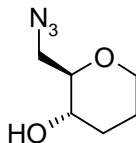
(2*R*,3*S*,4*R*)-2-Azidomethyl-tetrahydro-pyran-3,4-diol (13).

To a solution **C** (800 mg, 2.64 mmol) in DMF (10 mL), sodium azide (1.80 g, 26.4 mmol) was added and the reaction mixture was allowed to stir for 18 hours at 80 °C. Reaction mixture was diluted with cold water and extracted with dichloromethane (3X30 mL). Combined organic layer was washed with brine, dried over sodium sulfate and evaporation of solvent resulted in compound **13** (300 mg). Yield 66%; IR (CHCl₃): 2099, 3400cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 155-1.65 (m, 1H), 1.88-1.93 (m, 1H), 3.14 (t, *J* = 9.3 Hz, 1H), 3.21-3.26 (m, 1H), 3.37 (dd, *J* = 6.9, 13.2 Hz, 1H), 3.42-3.53 (m, 3H), 3.90-3.94 (m, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 81.1, 74.1, 73.8, 66.6, 53.0, 35.0; LCMS = 191.1 (M+18).



Toluene-4-sulfonic acid (2*R*,3*S*)-3-hydroxy-tetrahydro-pyran-2-ylmethyl ester⁴ (E).

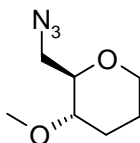
To a solution of ((2*R*,3*S*)-2-Hydroxymethyl-tetrahydro-pyran-3-ol⁵ (2.0 g, 15.15 mmol) in pyridine (20 mL), *p*-Toluenesulfonyl chloride (3.45 g, 18.18 mmol) was added and reaction mixture allowed to stir for overnight at room temperature. Reaction mixture was diluted with ice water and neutralized with 1N HCl to pH 6 followed by extraction with dichloromethane (3X30 mL), combined organic layer washed with brine and dried with sodium sulfate. After evaporation of solvent furnished the title compound **E** (3.1g). Yield 70%; IR (CHCl₃): 669, 771, 1216, 3019 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.34-1.41 (m, 2H), 1.55-1.62 (m, 2H), 2.01-2.05 (m, 1H), 2.45 (s, 3H), 3.15-3.19 (m, 1H), 3.22-3.26 (m, 1H), 3.76-4.08 (m, 1H), 4.06 (dd, *J* = 6.0, 10.4 Hz, 1H), 4.28 (dd, *J* = 1.6, 10.8 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 2H), 7.78 (d, *J* = 8.0 Hz, 2H); ¹³C NMR (100.6 MHz, CD₃OD) δ 146.3, 134.3, 130.9 (2C), 129.1 (2C), 81.4, 71.5, 68.5, 66.7, 33.6, 26.3, 21.6; LCMS = 287.0 (M+1).



14

(2*R*, 3*S*)-2-Azidomethyl-tetrahydro-pyran-3-ol (14).

To a solution of **E** (1.0 g, 3.49 mmol) in DMF (10 mL), sodium azide (2.49 g, 34.9 mmol) was added and reaction mixture allowed to stir for 18 hours at 80 °C. Reaction mixture was diluted with ice water and extraction with dichloromethane (3X30 mL), combined organic layer washed with brine, dried over sodium sulfate, and evaporation of solvent resulted in **14** (480 mg). Yield 86%. IR (CHCl₃): 2099, 3400 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.36-1.46 (m, 1H), 1.62-1.70 (m, 2H), 2.05-2.08 (m, 1H), 3.16-3.20 (m, 1H), 3.32-3.38 (m, 3H), 3.48 (d, *J* = 13.2 Hz, 1H), 3.86-3.92 (m, 1H); ¹³C NMR (100.6 MHz, CD₃OD) δ 83.1, 68.6, 68.0, 53.1, 33.5, 26.5; LCMS = 175.2 (M+18).

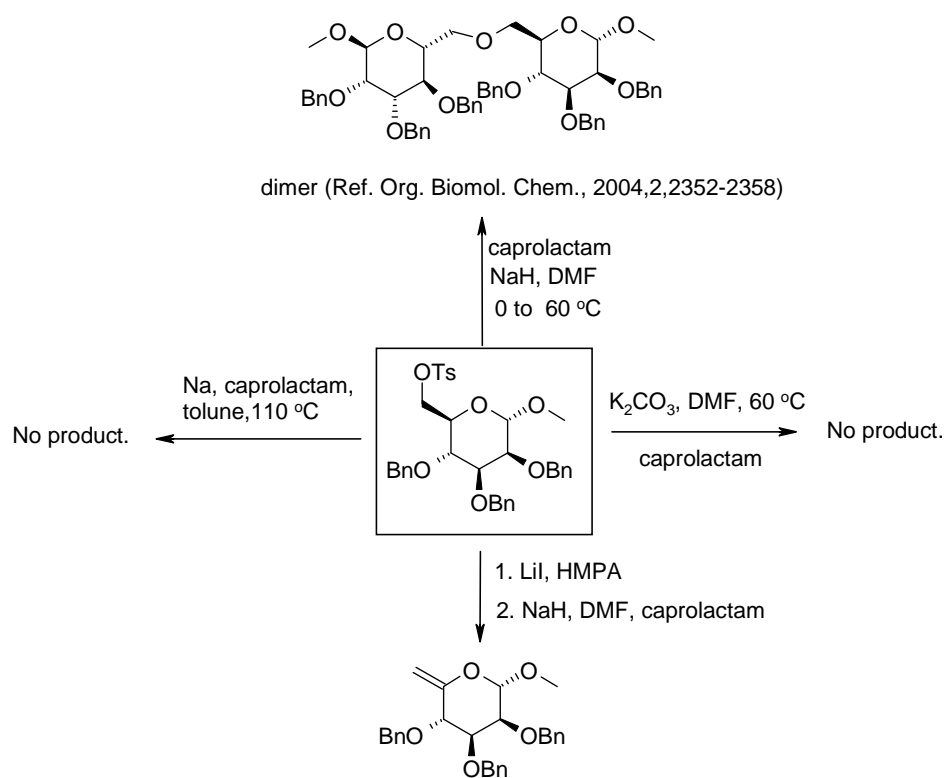


19

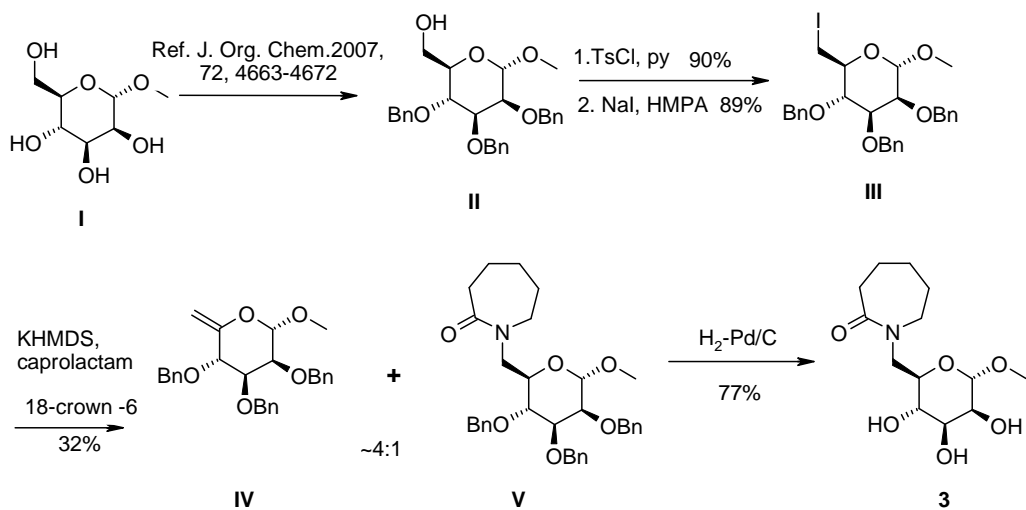
(2*R*, 3*S*)-2-Azidomethyl-3-methoxy-tetrahydro-pyran (19).

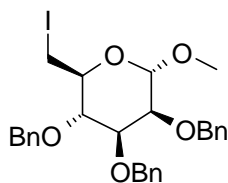
To a solution of **(14)** (200 mg, 1.25 mmol) in anhydrous THF (5 mL), sodium hydride (60% in mineral oil, 90 mg, 1.88 mmol) was added at 0 °C and reaction mixture allowed to stir for one hour at room temperature. Reaction mixture was cooled again to 0 °C and methyl iodide (0.16 mL, 3.7 mmol) added and stirring continued for another hour at room temperature. Reaction mixture was diluted with ice water and extraction with dichloromethane (3X15 mL). The combined organic layer was washed with brine, dried over sodium sulfate and evaporation of solvent led to compound **19** (185 mg). Yield 85%; IR (CHCl₃): 1099, 1432, 2099 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.23-1.33 (m, 2H), 1.61-1.70 (m, 2H), 2.30-2.33 (m, 1H), 3.05-3.09 (m, 1H), 3.24-2.28 (m, 1H), 3.35 (s, 3H), 3.37-3.43 (m, 1H), 3.52- 3.55 (d, *J* = 12.8 Hz, 1H), 3.96 (dd, *J* = 4.4, 13.6 Hz, 1H); ¹³C NMR (100.6 MHz, CDCl₃) δ 80.0, 75.9, 67.9, 56.3, 52.3, 28.3, 25.1.

Unsuccessful efforts toward alternate synthesis of sugar lacatam 3.



Alternate route to the synthesis of sugar-lactam **3**.

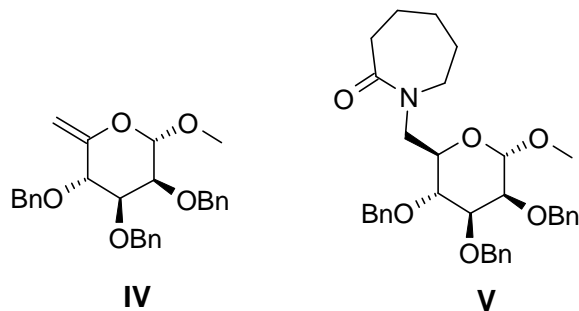




III

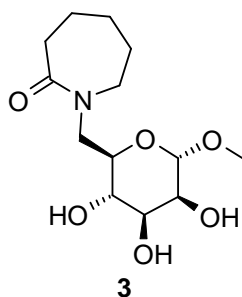
Methyl 6-deoxy-6-iodo-2, 3, 4-tri-O-benzyl- α -D-mannopyranoside (III).⁶ To a solution of 2, 3, 4-Tri-O-benzyl- α -methyl-D-mannopyranoside⁷ (2.0 g, 4.30 mmol) in pyridine (20 mL), *p*-toluenesulfonyl chloride (2.0 g, 4.31 mmol) was added and the reaction mixture allowed to stir for 6 hours at room temperature. Reaction mixture was diluted with ice water and neutralized with 1N HCl (~ pH 6) followed by extraction with dichloromethane (3X30 mL). The combined organic layer washed with brine, dried over sodium sulfate and evaporation of solvent resulted in crude product, which was purified by column chromatography (10%EtOAc:Hexane) to furnish the tosylate (2.4 g). Yield 90%; $[\alpha]^{25.8}_D = +15.6^\circ$ (*c* 1, CH₃OH); IR (CHCl₃ 1215, 3019 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 2.39 (s, 3H), 3.24(s, 3H), 3, 74-3.82 (m, 4H), 4.21-4.28 (m, 2H), 4.45 (d, *J* = 11.2 Hz, 1H), 4.56 (s, 2H), 4.64-4.67 (m, 3H), 4.66 (d, *J* = 10.8 Hz, 1H), 7.18 (s, 2H), 7.25-7.30 (m, 15H), 7.77(d, *J* = 6.4 Hz, 2H); ¹³C NMR (100.6 MHz, CDCl₃) δ 144.9, 138.5, 138.4, 138.3, 133.2, 130.0 (2C), 128.6 (4C), 128.3 (2C), 128.2 (2C), 128.1 (2C), 128.0 (2C), 128.0 (2C), 127.9, 127.9 (2C), 99.1, 80.3, 75.3, 74.5, 74.3, 72.9, 72.3, 70.2, 69.5, 55.1, 21.9; LCMS = 635.9 (M+18);

To a solution of above tosylate (2.0 g, 3.23 mmol) in toluene (50 mL), HMPA (5.63 ml, 32.36 mmol) and lithium iodide (4.3 g, 32.36 mmol) was added and reaction mixture was refluxed for 24 hours. Reaction mixture was diluted with ice water and extraction with ethyl acetate (3X30 mL), combined organic layer washed with aq. sodium thiosulfate, brine and dried with sodium sulfate. The crude product obtained after evaporation of solvent was purified using 10% EtoAc: hexane to furnish the title compound III (1.6 g). Yield 89%; $[\alpha]^{24.8}_D = +22.4^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 669, 771, 1215, 3019 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 3.29-3.31 (m, 1H), 3.36 (s, 3H), 3.49-3.57 (m, 2H), 3.74-3.78 (m, 2H), 3.88 (dd, *J* = 7.0, 2.0 Hz, 1H), 4.59 (s, 2H), 4.65-4.68 (m, 1H), 4.72-4.76 (m, 3H), 4.98 (d, *J* = 10.8 Hz, 1H), 7.20- 7.30 (m, 15H); ¹³C NMR (100.6 MHz, CD₃OD) δ 138.5, 138.5, 138.4, 128.7 (2C), 128.7 (2C), 128.6 (2C), 128.3 (2C), 128.1 (2C), 127.9 (2C), 127.9, 127.9(2C), 99.3, 80.2, 78.8, 75.7, 74.8, 73.0, 72.3, 71.7, 55.3, 7.4; LCMS = 592 (M+18);



6-Deoxyhex-eno-pyranoside(IV) and Methyl 6-deoxy-2,3,4-tri-O-benzyl-6-N-azepan-2-one- α -D-mannopyranoside (V). To a solution of caprolactam (395 mg, 3.48 mmol) in dry THF (5 mL), KHMDS (0.5 M in toluene 7.0 ml, 3.48 mmol) was added at 0 °C followed by 18-crown-6 (197 mg, 0.87 mmol) and the reaction mixture was allowed to stir at room temperature for 3 hrs. After that reaction mixture was cooled to 0°C and a solution of **III** (1.0g, 1.74 mmol) in THF (10ml) was added. After stirring for two hours at room temperature, the reaction mixture was heated to 50 °C for 12 hours. Reaction was quenched with saturated aq.NH₄Cl (10 mL) at room temperature. Reaction mixture was extracted with ethyl acetate (3X25 mL), washed the combined organic layer with brine and dried over sodium sulfate. The crude product obtained after evaporation of solvent was purified using preparative HPLC to furnish compound **IV** (200 mg, 26%) and **V**, (60 mg, 6%).

Spectral data for **IV**. $[\alpha]^{25.8}_D = -18.2^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 669, 1215, 3019 cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 3.40 (s, 3H), 3.83-3.86 (m, 1H), 3.88 (d, *J* = 2.8 Hz, 1H), 4.34 (d, *J* = 8.4 Hz, 1H), 4.63- 4.80 (m, 9H), 7.25-7.36 (m, 15H); ¹³C NMR (100.6 MHz, CD₃OD) δ 154.9, 138.7, 138.4, 138.4, 128.6 (2C), 128.5, 128.5, 128.4 (2C), 128.1(2C), 128.0, 128.0, 127.9 (2C), 127.8, 127.8, 127.7, 100.9, 96.9, 78.8, 76.9, 75.8, 73.8, 73.4, 73.1, 55.6; LCMS = 464.0 (M+18); HPLC Purity (%) = 99.2; Spectral data for **V**:- $[\alpha]^{25.8}_D = -1.04^\circ$ (*c* 1, CH₃OH); IR (CHCl₃): 669, 771, 1215, 1658cm⁻¹; ¹H NMR (CD₃OD, 400 MHz) δ 1.65 (s, 6H), 2.50-2.54 (m, 2H), 3.27 (s, 3H), 3.47-3.49 (m, 2H), 3.56 (dd, *J* = 7.6, 13.9 Hz, 1H), 3.68 (t, *J* = 9.3 Hz, 1H), 3.58-3.78 (m, 2H), 3.87-3.94 (m, 2H), 4.60 (s, 2H), 4.67-4.77 (m, 4H), 4.90 (d, *J* = 10.5 Hz, 1H), 7.18-7.40 (m, 15H); ¹³C NMR (100.6 MHz, CD₃OD) δ 181.3, 143.8, 143.5, 143.4, 133.5 (2C), 133.4, 133.4, 133.2 (2C), 133.1 (2C), 133.0 (2C), 132.9 (2C), 132.8, 132.7, 132.6, 104.1, 85.2, 82.5, 82.4, 82.2, 81.9, 81.4, 79.8, 59.9, 56.3, 54.4, 42.5, 35.1, 33.4, 28.6; LCMS = 560.4 (M+1); HPLC Purity (%) = 98.4; HRMS (ESI): *m/z* calculated for C₃₄H₄₁NO₆Na [M + Na]⁺ 582.2831 found 582.2826.



Methyl 6-deoxy-6-N-azepan-2-one- α -D-mannopyranoside (3). 10% dry Pd/C (10 mg) was added to the solution **V** (50 mg, 0.08 mmol) in EtOAc:ethanol (3:1) mixture. Reaction mixture was stirred under hydrogen atmosphere for 18 hours at room temperature and filtered through a celite pad. Evaporation of solvent furnished the title compound **3** (20 mg) in 77% yield. The spectral data (^1H and ^{13}C) is compared with that of **3** prepared using Aube reaction and found that they are identical.

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ADV-TRNG-06-011
09 June 2009
Advirus Therapeutics Pune

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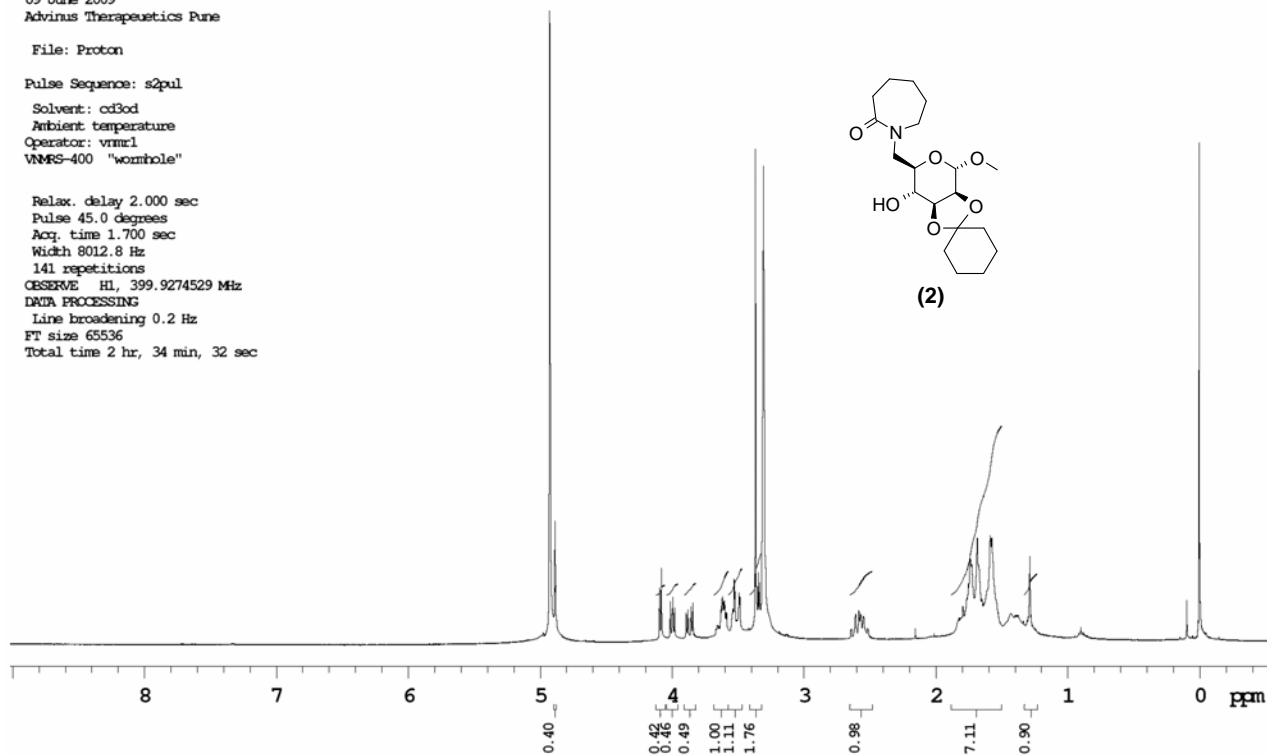
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Total time 2 hr, 34 min, 32 sec



ADV-TRNG-06-011
27 June 2009
Advirus Therapeutics Pune

Sample Name:

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FidFile: ADV-TRNG-06-011-13C

Pulse Sequence: Carbon (s2pul)

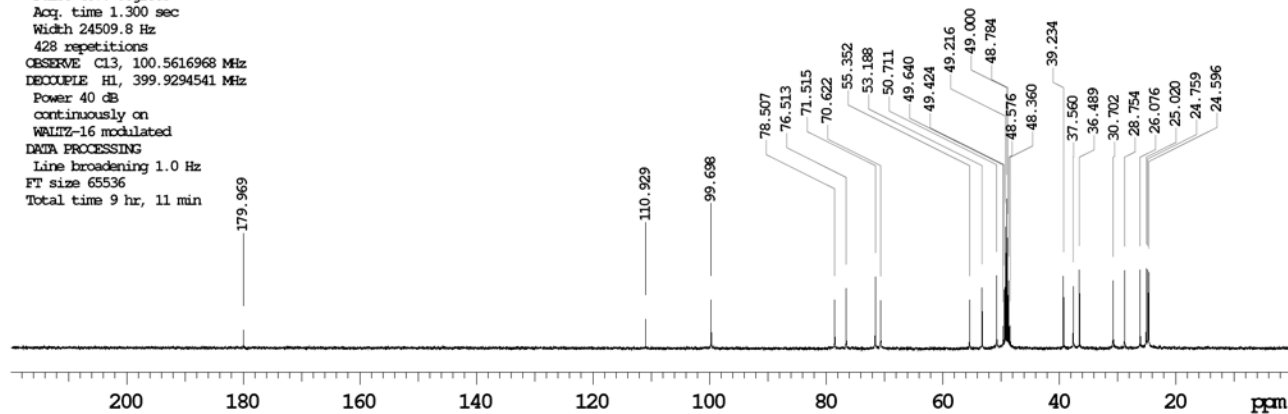
Solvent: cd3od

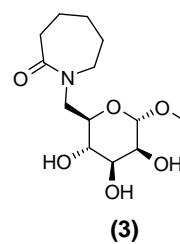
Data collected on: Jun 27 2009

Operator: vnmr1

VNMR-400 "DEIRE400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
428 repetitions
OBSERVE C13, 100.5616968 MHz
DECOUPLE H1, 399.9294541 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 11 min





ADV-trng-06-II
Advinus Therapeutics Pune

Sample Name:

Data Collected on:
DZIRE400-vnmr400

Archive directory:

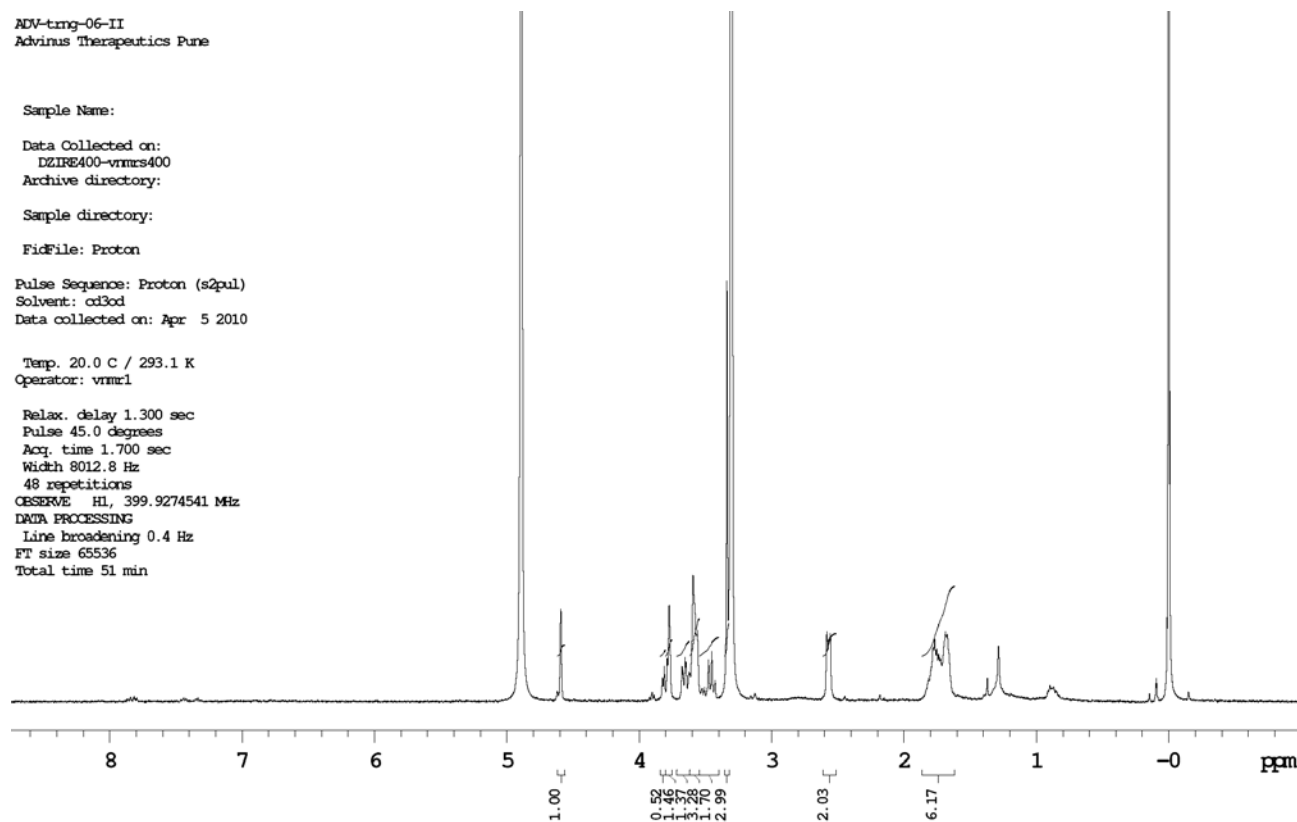
Sample directory:

FidFile: Proton

Pulse Sequence: Proton (s2pul)
Solvent: cd3od
Data collected on: Apr 5 2010

Temp. 20.0 C / 293.1 K
Operator: vnmr1

Relax. delay 1.300 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 8012.8 Hz
48 repetitions
OBSERVE H1, 399.9274541 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 65536
Total time 51 min



ADV-TRNG-011-02
27 June 2009
Advinus Therapeutics Pune

Sample Name:

Archive directory:

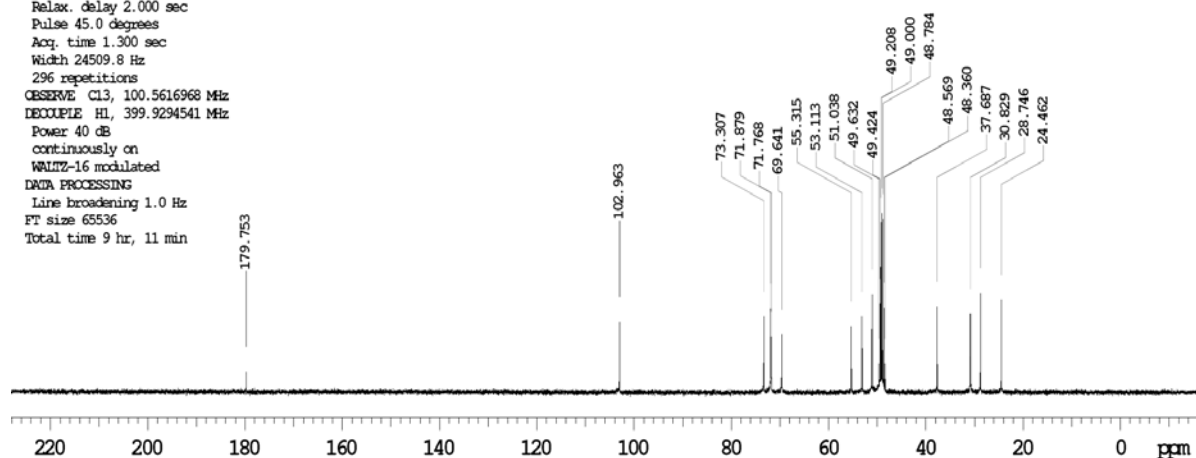
Sample directory:

FidFile: ADV-TRNG-011-02-13C

Pulse Sequence: Carbon (s2pul)
Solvent: cd3od
Data collected on: Jun 27 2009

Operator: vnmr1
VNMRS-400 "DZIRE400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
296 repetitions
OBSERVE C13, 100.5616968 MHz
DECUPLE H1, 399.9294541 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 11 min



ADV-P-247-069-R
Advirus Therapeutics Pune

Sample Name:

Data Collected on:
DZIRE400-vrms400
Archive directory:

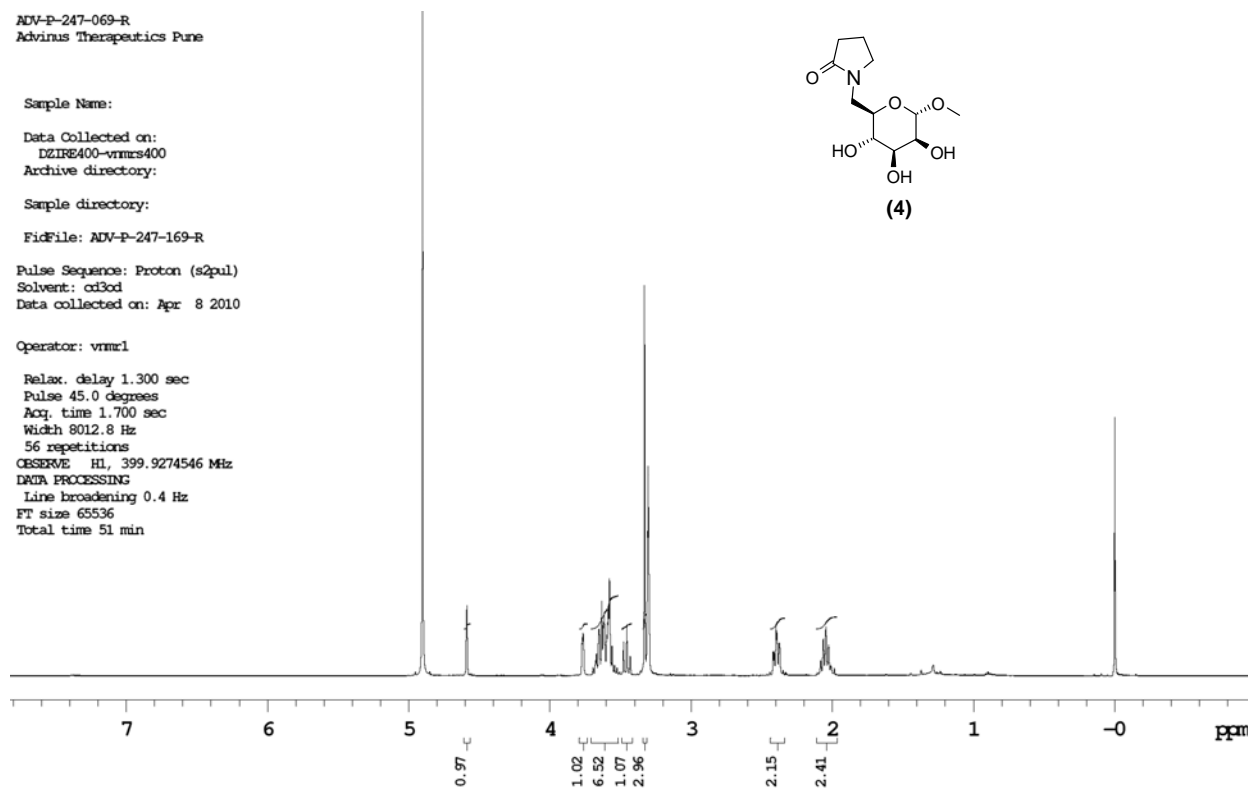
Sample directory:

FidFile: ADV-P-247-169-R

Pulse Sequence: Proton (s2pul)
Solvent: cd3od
Data collected on: Apr 8 2010

Operator: vrmr1

Relax. delay 1.300 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 8012.8 Hz
56 repetitions
OBSERVE H1, 399.9274546 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 65536
Total time 51 min



ADV-P-247-069-R
13C Experiment
Advirus Pune

Sample Name:

Data Collected on:
DZIRE400-vrms400
Archive directory:

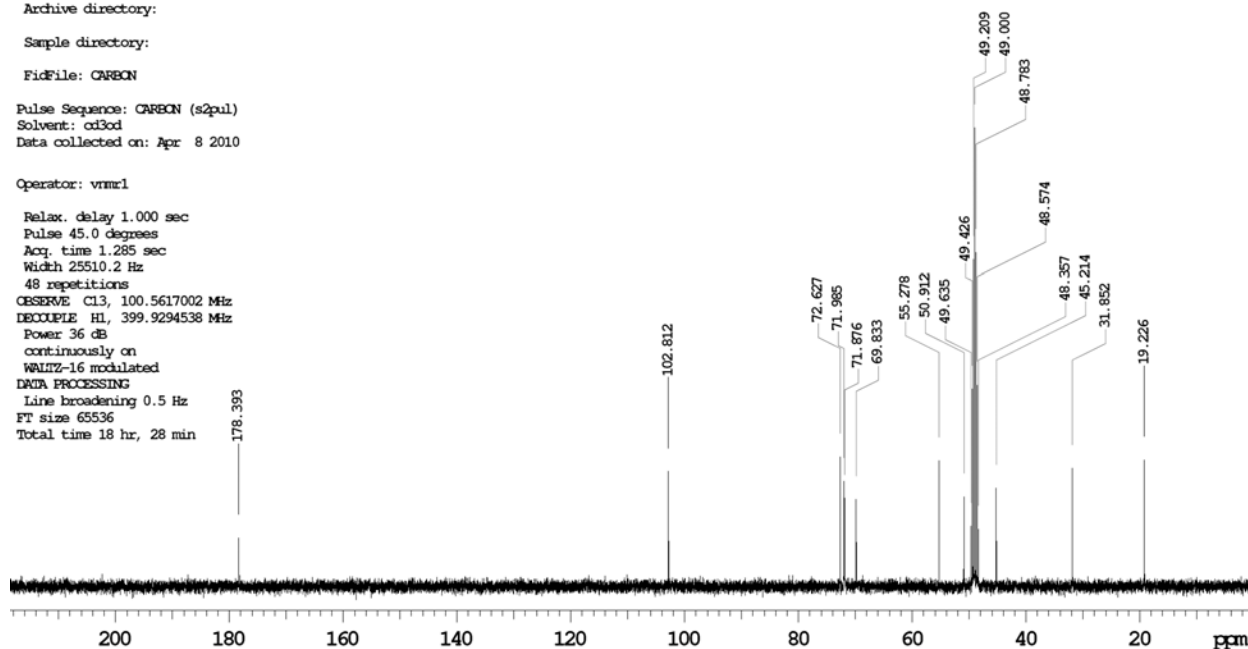
Sample directory:

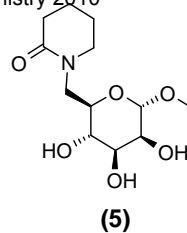
FidFile: CARECN

Pulse Sequence: CARECN (s2pul)
Solvent: cd3od
Data collected on: Apr 8 2010

Operator: vrmr1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
48 repetitions
OBSERVE C13, 100.5617002 MHz
DECUPLE H1, 399.9294538 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 18 hr, 28 min





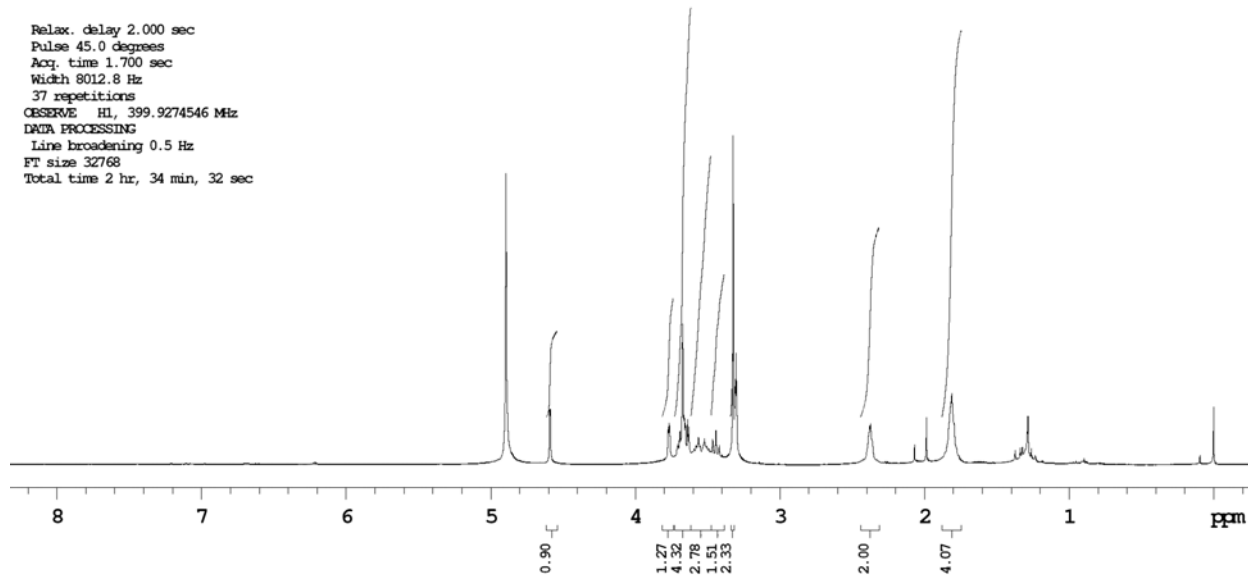
ADV-P-247-068A
15 June 2009
Advinus Therapeutics Pune

File: Proton

Pulse Sequence: s2pul

Solvent: cd3od
Ambient temperature
Operator: vrmc1
NMRS-400 "wormhole"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 8012.8 Hz
37 repetitions
OBSERVE HL, 399.9274546 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 2 hr, 34 min, 32 sec



ADV-P-247-068A
27 June 2009
Advinus Therapeutics Pune

Sample Name:

Archive directory:

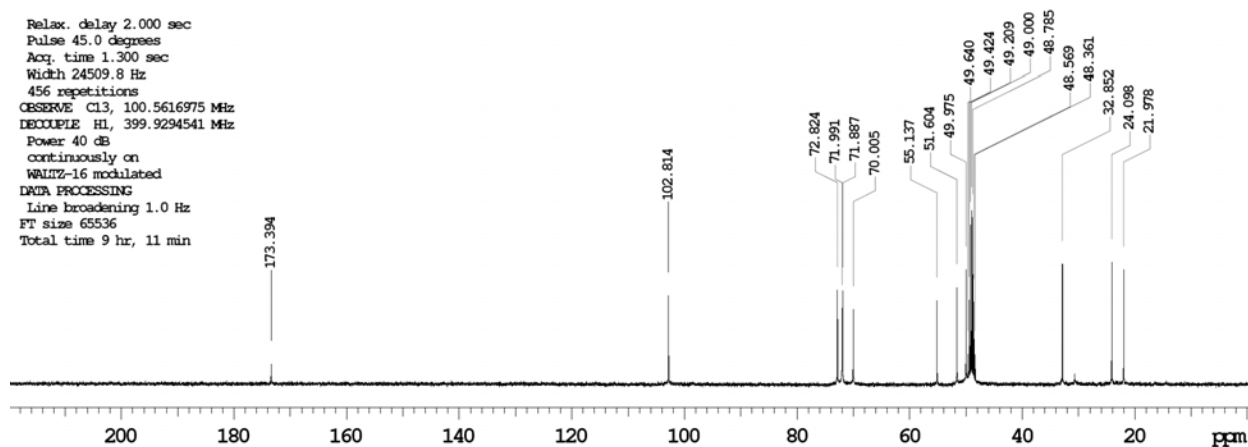
Sample directory:

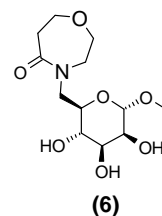
FidFile: ADV-P-247-068A-13C

Pulse Sequence: Carbon (s2pul)
Solvent: cd3od
Data collected on: Jun 27 2009

Operator: vrmc1
NMRS-400 "DZIRE400"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
456 repetitions
OBSERVE C13, 100.5616975 MHz
DECOUPLE HL, 399.9294541 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 11 min



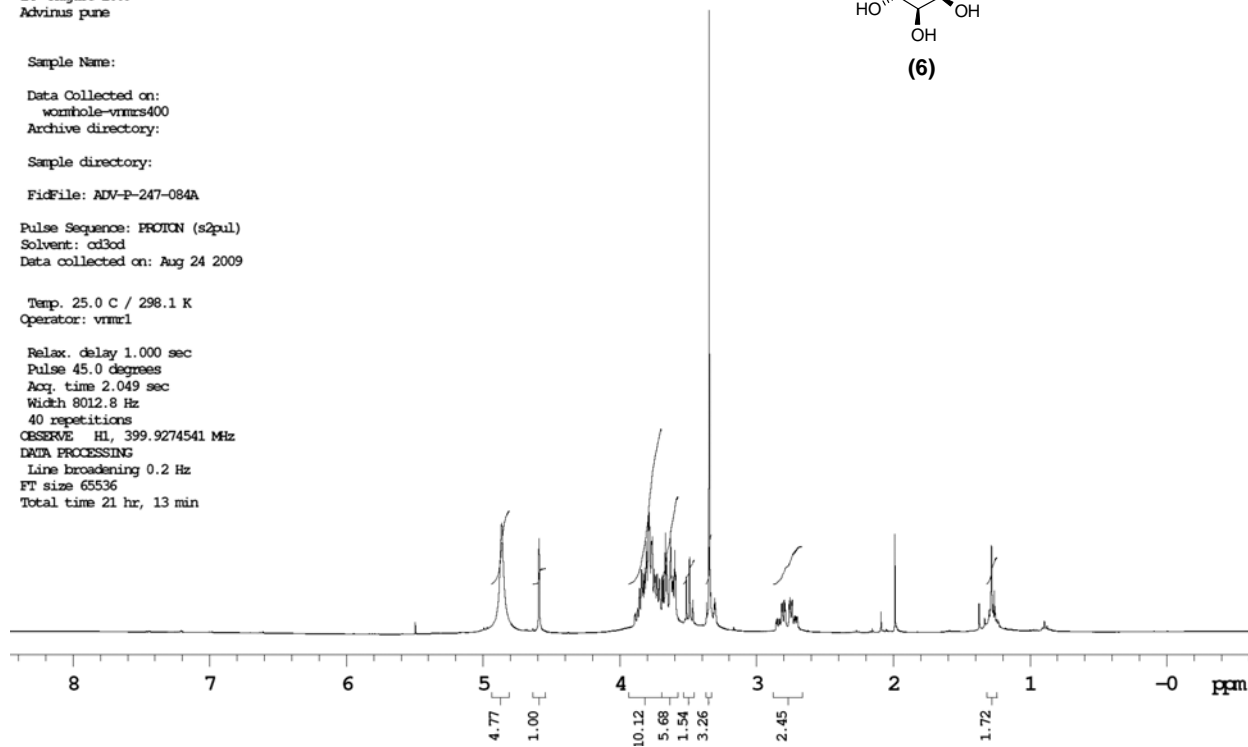


ADV-P-247-084-A
24 August 2009
Advinus pune

Sample Name:
Data Collected on:
wormhole-vrms400
Archive directory:
Sample directory:
FidFile: ADV-P-247-084A
Pulse Sequence: PROTON (s2pul)
Solvent: cd3od
Data collected on: Aug 24 2009

Temp. 25.0 C / 298.1 K
Operator: vrmcl

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8012.8 Hz
40 repetitions
OBSERVE H1, 399.9274541 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 65536
Total time 21 hr, 13 min

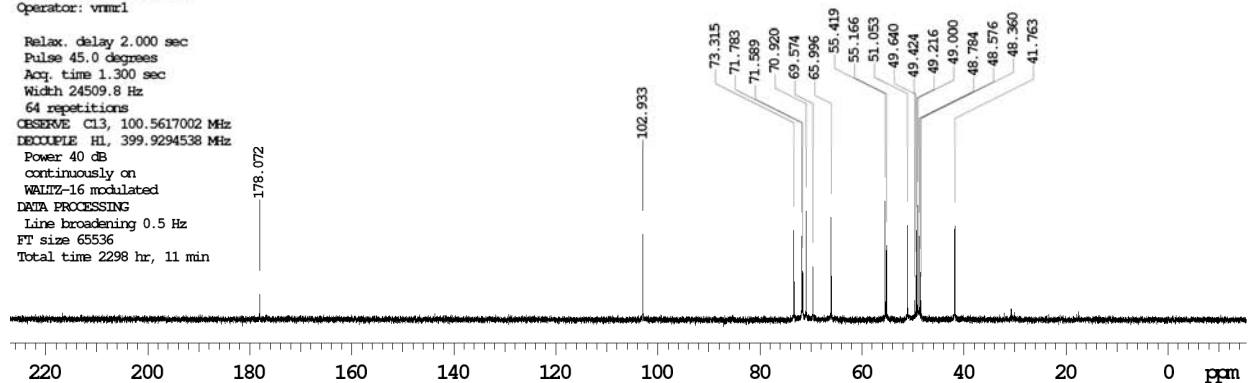


ADV-P-247-084-A
14 August 2009
CL3 EXPERIMENT

Sample Name:
Data Collected on:
wormhole-vrms400
Archive directory:
Sample directory:
FidFile: ADV-P-247-084-A-CL3
Pulse Sequence: Carbon (s2pul)
Solvent: cd3od
Data collected on: Aug 21 2009

Temp. 25.0 C / 298.1 K
Operator: vrmcl

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
64 repetitions
OBSERVE CL3, 100.5617002 MHz
DECOUPLE H1, 399.9294538 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2298 hr, 11 min

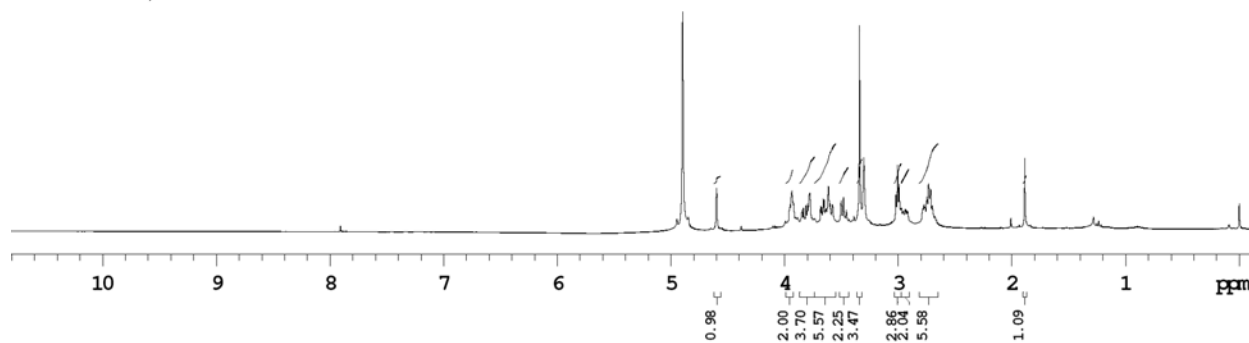
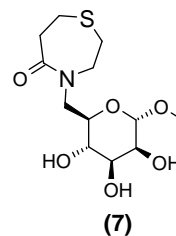


ADV-P-247-073-I
13 July 2009
Advinus Therapeutics Pune

File: ADV-P-247-073-I

Pulse Sequence: s2pul
Solvent: cd3od
Ambient temperature
Operator: vrmc1
File: ADV-P-247-073-I
VMRS-400 "wormhole"

Relax. delay 1.300 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 8012.8 Hz
68 repetitions
OBSERVE HI, 399.9274546 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 65536
Total time 51 min, 20 sec

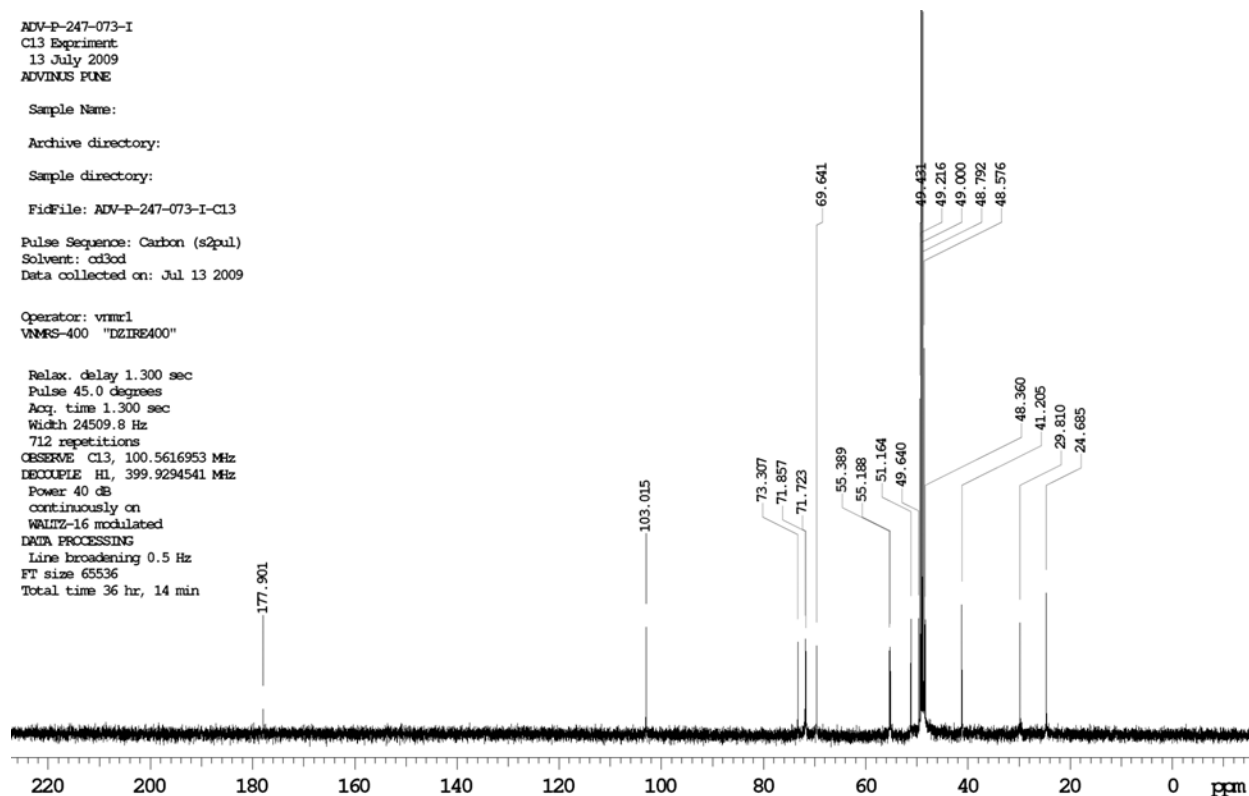


ADV-P-247-073-I
Cl3 Experiment
13 July 2009
ADVINUS PUNE

Sample Name:
Archive directory:
Sample directory:
FidFile: ADV-P-247-073-I-Cl3
Pulse Sequence: Carbon (s2pul)
Solvent: cd3od
Data collected on: Jul 13 2009

Operator: vrmc1
VMRS-400 "DZIRE400"

Relax. delay 1.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
712 repetitions
OBSERVE Cl3, 100.5616953 MHz
DECOUPLE HI, 399.9294541 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 36 hr, 14 min



ADV-P-425-017
 Advirus Therapeutics Pune

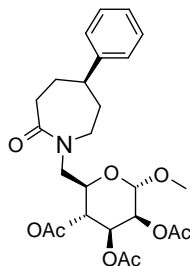
Sample Name:

Data Collected on:
 DZIRE400-vnmrs400
 Archive directory:

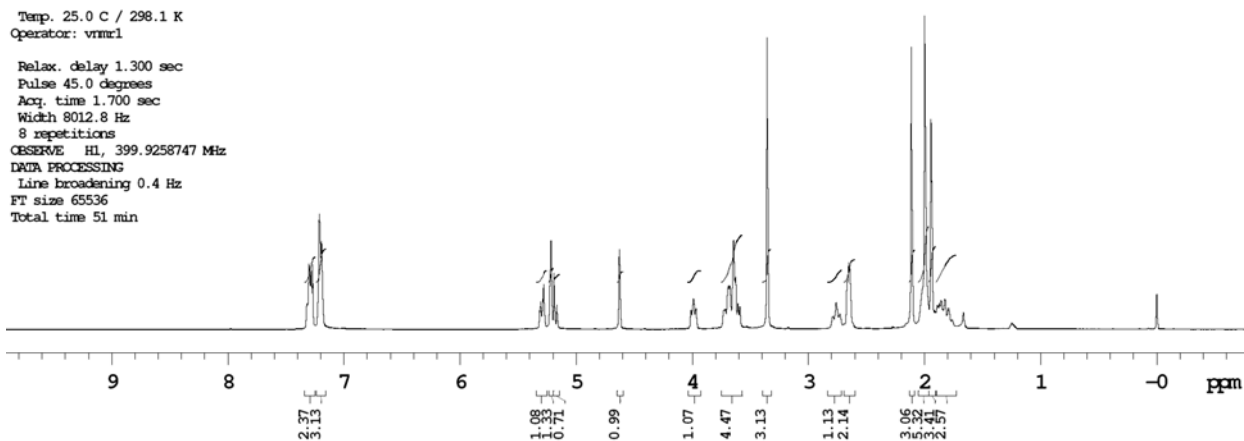
Sample directory:

FidFile: ADV-P-425-017

Pulse Sequence: Proton (s2pul)
 Solvent: cdcl3
 Data collected on: Apr 14 2010



Triacetate of (8)



ADV-P-425-017
 CD3 EXPERIMENT

Sample Name:

Data Collected on:
 DZIRE400-vnmrs400
 Archive directory:

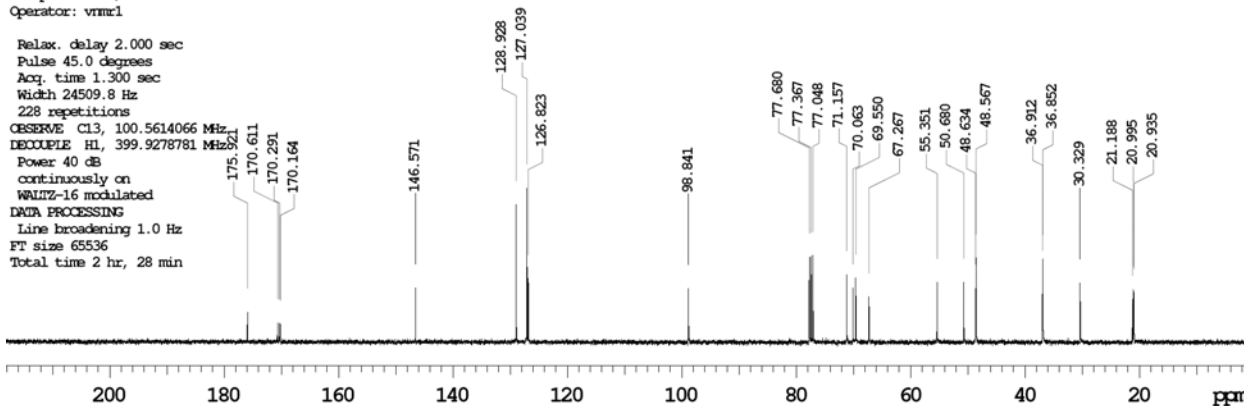
Sample directory:

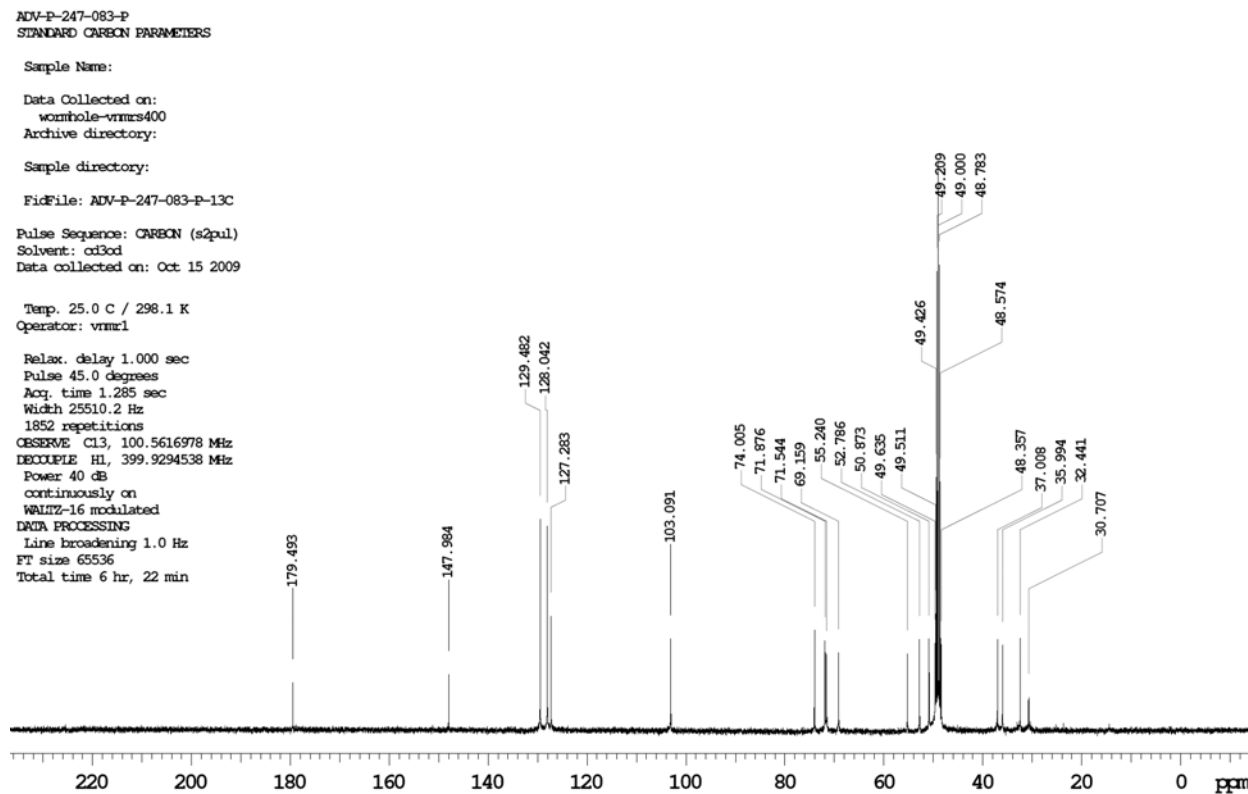
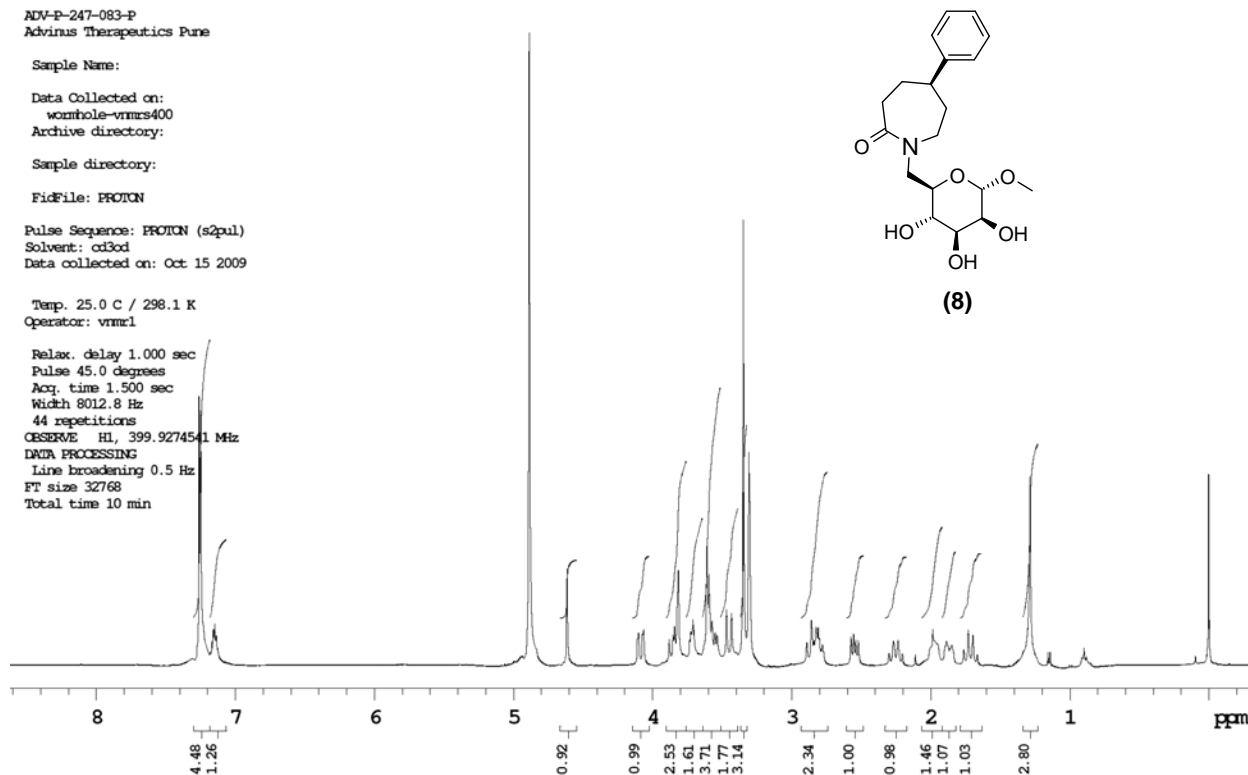
FidFile: Carbon

Pulse Sequence: Carbon (s2pul)
 Solvent: cdcl3
 Data collected on: Apr 14 2010

Temp. 25.0 C / 298.1 K
 Operator: vnmr1

Relax. delay 2.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 228 repetitions
 OBSERVE C13, 100.5614066 MHz
 DECOUPLE H1, 399.9278781 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 2 hr, 28 min



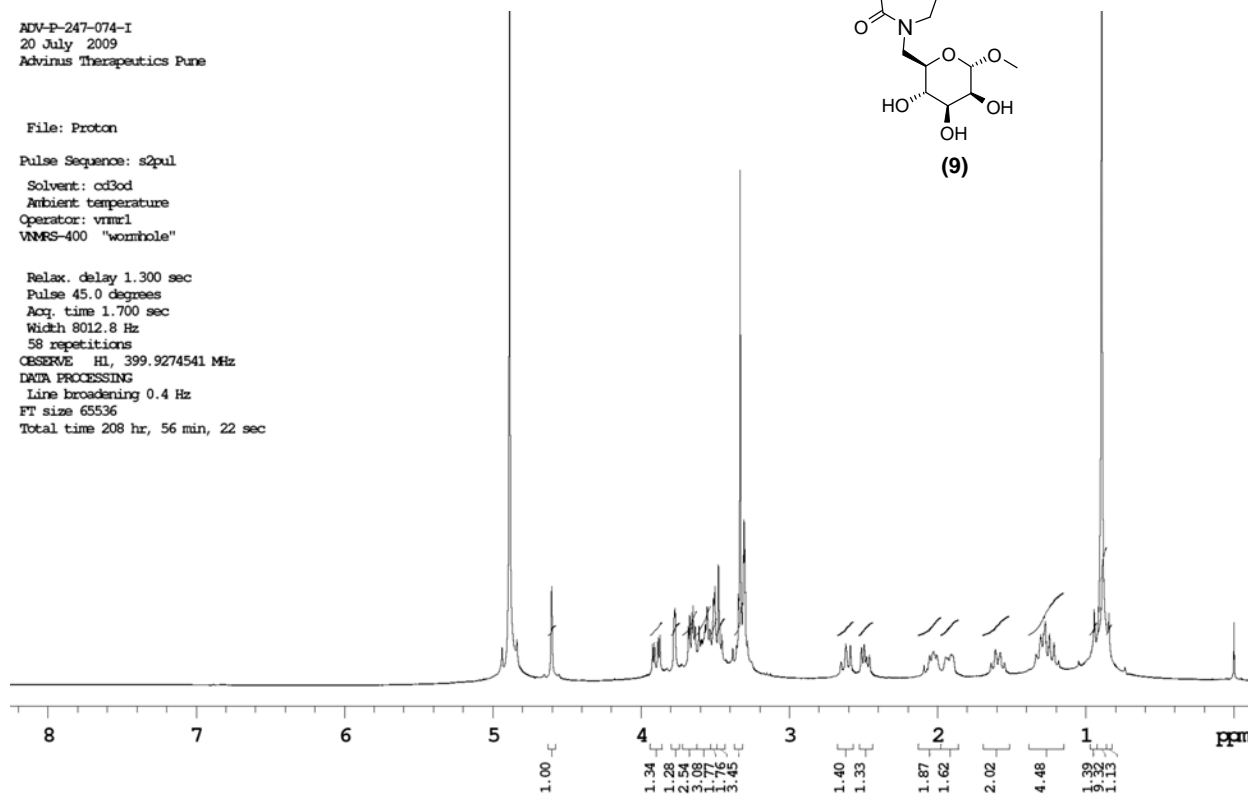
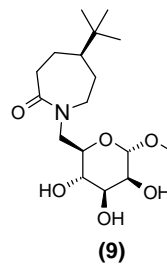


ADV-P-247-074-I
 20 July 2009
 Advinus Therapeutics Pune

File: Proton

Pulse Sequence: s2pul
 Solvent: cd3od
 Ambient temperature
 Operator: vnm1
 VNMR-400 "wormhole"

Relax. delay 1.300 sec
 Pulse 45.0 degrees
 Acq. time 1.700 sec
 Width 8012.8 Hz
 58 repetitions
 OBSERVE H1, 399.9274541 MHz
 DATA PROCESSING
 Line broadening 0.4 Hz
 FT size 65536
 Total time 208 hr, 56 min, 22 sec



ADV-P-247-074-I
 20 July 2009
 C13 EXPERIMENT

Sample Name:

Archive directory:

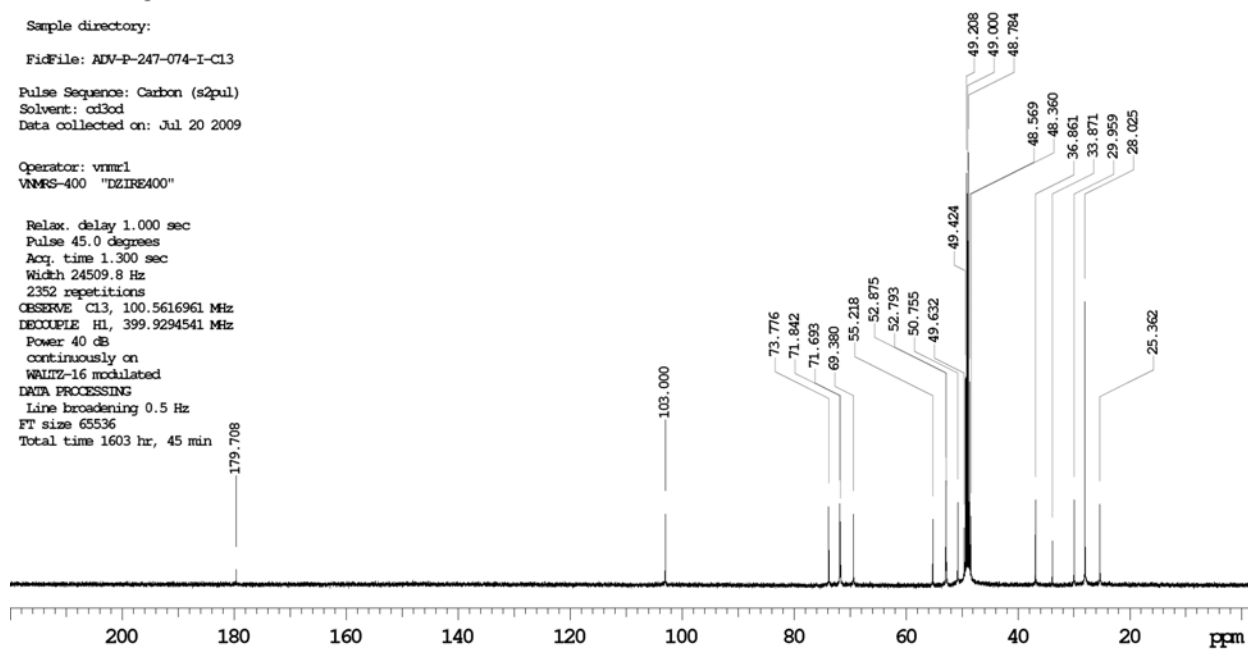
Sample directory:

FidFile: ADV-P-247-074-I-C13

Pulse Sequence: Carbon (s2pul)
 Solvent: cd3od
 Data collected on: Jul 20 2009

Operator: vnm1
 VNMR-400 "DZIRE400"

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.300 sec
 Width 24509.8 Hz
 2352 repetitions
 OBSERVE C13, 100.5616961 MHz
 DECOUPLE H1, 399.9294541 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 1603 hr, 45 min



ADV-P-247-085-P
ADVINUS THERAPEUTICS PUNE

Sample Name:

Data Collected on:
DZIRE400-vnmrs400
Archive directory:

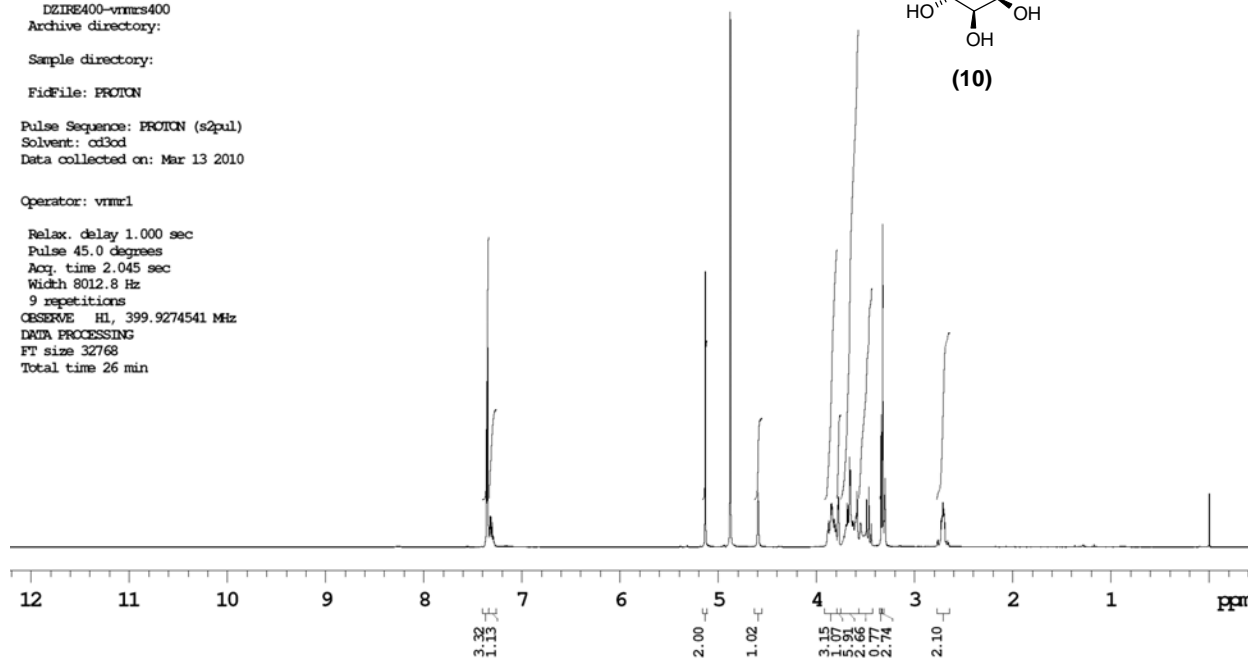
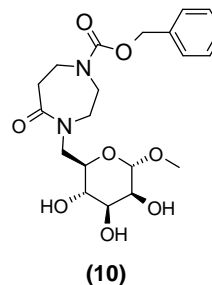
Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cd3od
Data collected on: Mar 13 2010

Operator: vnmr1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
9 repetitions
OBSERVE H1, 399.9274541 MHz
DATA PROCESSING
FT size 32768
Total time 26 min



ADV-P-247-085-P
C13 EXPERIMENT
ADVINUS PUNE

Sample Name:

Data Collected on:
DZIRE400-vnmrs400
Archive directory:

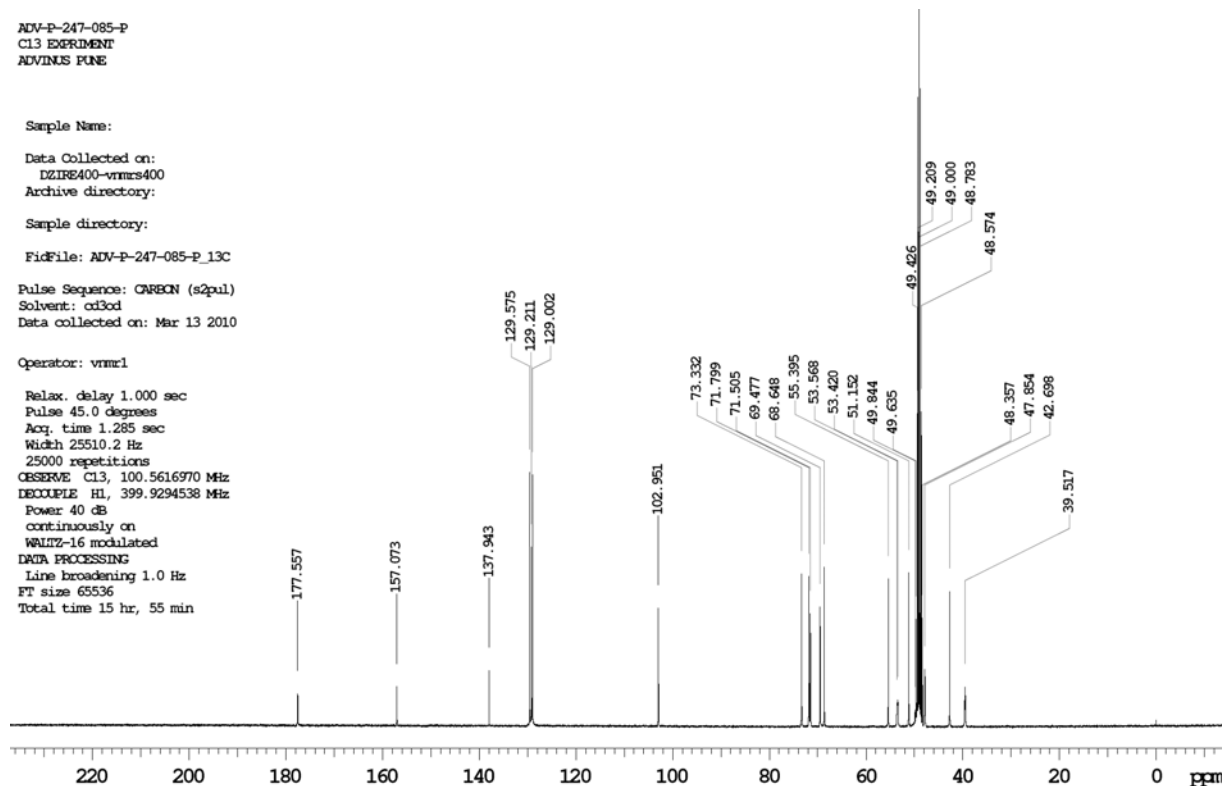
Sample directory:

FidFile: ADV-P-247-085-P_13C

Pulse Sequence: CARGON (s2pul)
Solvent: cd3od
Data collected on: Mar 13 2010

Operator: vnmr1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
25000 repetitions
OBSERVE C13, 100.5616970 MHz
DECOUPLE H1, 399.9294538 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 15 hr, 55 min

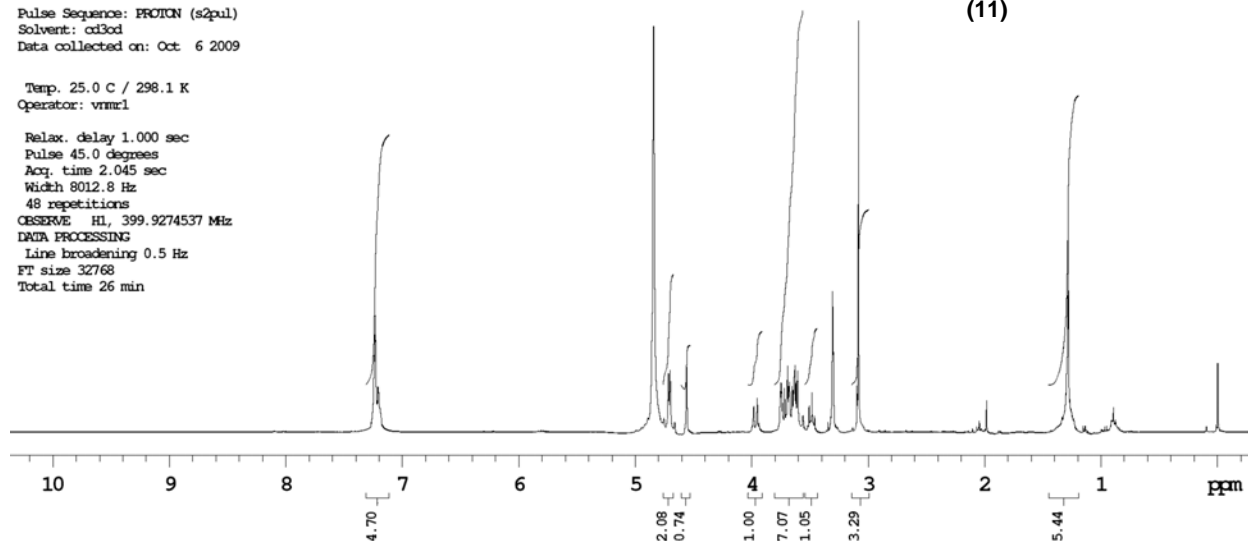
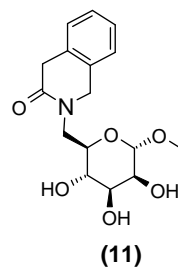


ADV-P-247-123
06 October 2009
Advirus Therapeutics Pune
Sample Name:
Data Collected on:
womhole-vnmrs400
Archive directory:
Sample directory:
FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cd3od
Data collected on: Oct 6 2009

Temp. 25.0 C / 298.1 K
Operator: vnmr1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.045 sec
Width 8012.8 Hz
48 repetitions
OBSERVE H1, 399.9274537 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 32768
Total time 26 min



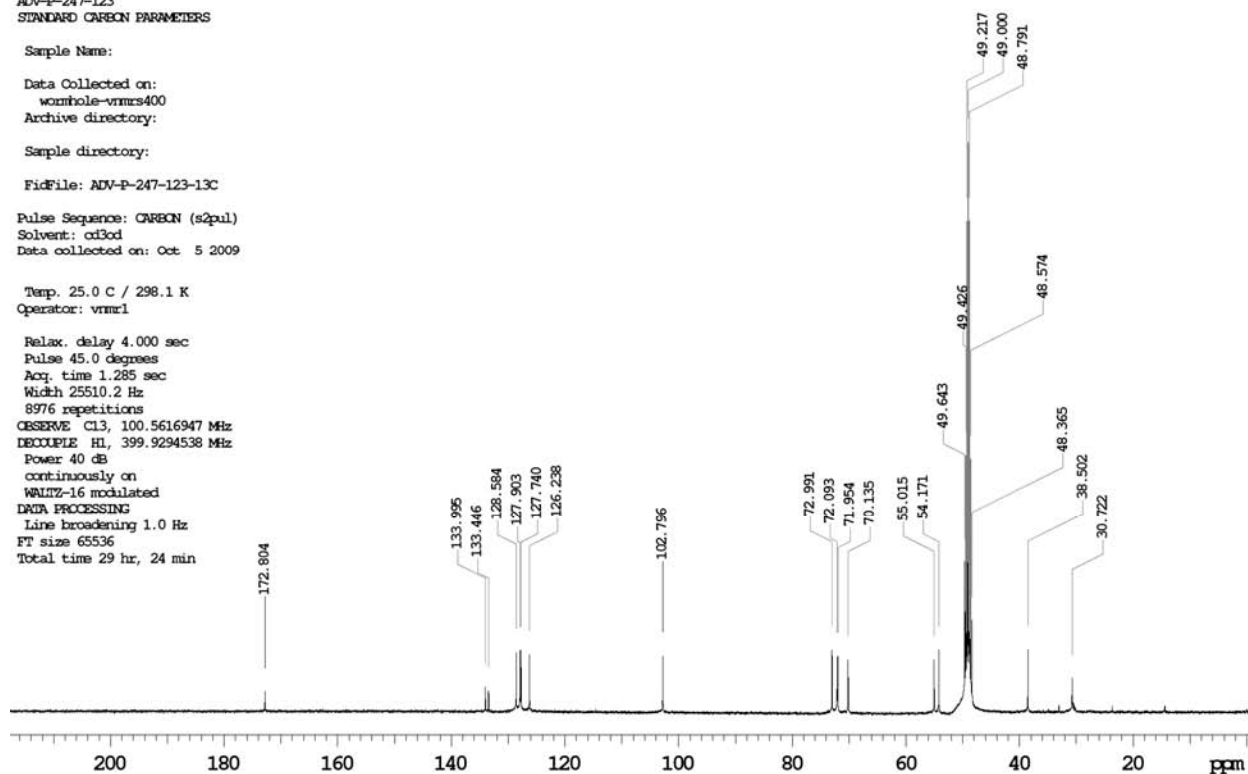
ADV-P-247-123
STANDARD CARBON PARAMETERS

Sample Name:
Data Collected on:
womhole-vnmrs400
Archive directory:
Sample directory:
FidFile: ADV-P-247-123-13C

Pulse Sequence: CARBON (s2pul)
Solvent: cd3od
Data collected on: Oct 5 2009

Temp. 25.0 C / 298.1 K
Operator: vnmr1

Relax. delay 4.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
8976 repetitions
OBSERVE C13, 100.5616947 MHz
DECOUPLE H1, 399.9294538 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 29 hr, 24 min



ADV-P-247-071-A
09 OCT 2009
ADVINUS THERAPEUTICS PUNE

Sample Name:

Data Collected on:
womhole-vrms400
Archive directory:

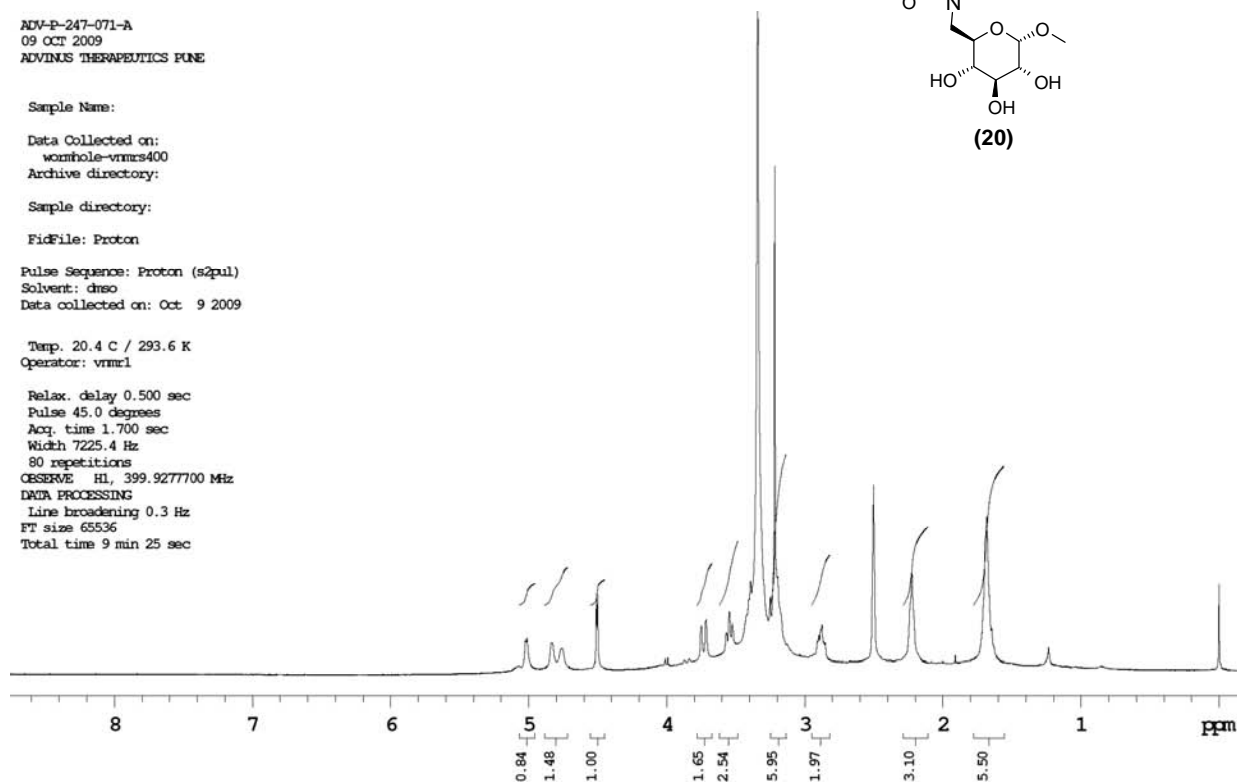
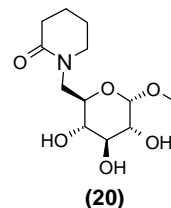
Sample directory:

FidFile: Proton

Pulse Sequence: Proton (s2pul)
Solvent: dmsc
Data collected on: Oct 9 2009

Temp. 20.4 C / 293.6 K
Operator: vrmc1

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 7225.4 Hz
80 repetitions
OBSERVE H1, 399.9277700 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 9 min 25 sec



ADV-P-247-077
20 July 2009
C13 EXPERIMENT

Sample Name:

Archive directory:

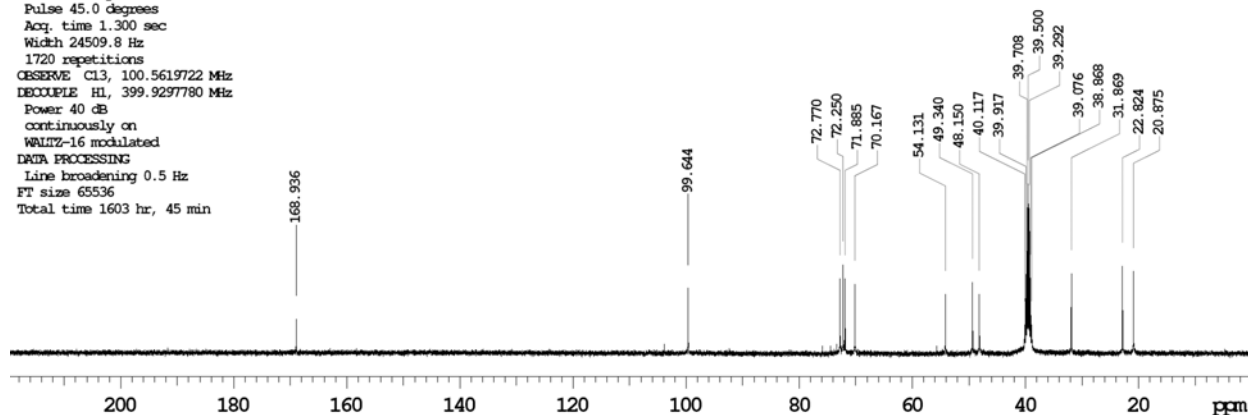
Sample directory:

FidFile: ADV-P-247-077-C13

Pulse Sequence: Carbon (s2pul)
Solvent: dmsc
Data collected on: Jul 24 2009

Operator: vrmc1
VMRS-400 "DZIRE400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
1720 repetitions
OBSERVE C13, 100.5619722 MHz
DECOUPLE H1, 399.9297780 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1603 hr, 45 min



ADV-P-247-115-B
Advinus Therapeutics Pune

Sample Name:

Data Collected on:
DZIRE400-vrms400
Archive directory:

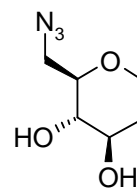
Sample directory:

FidFile: Proton

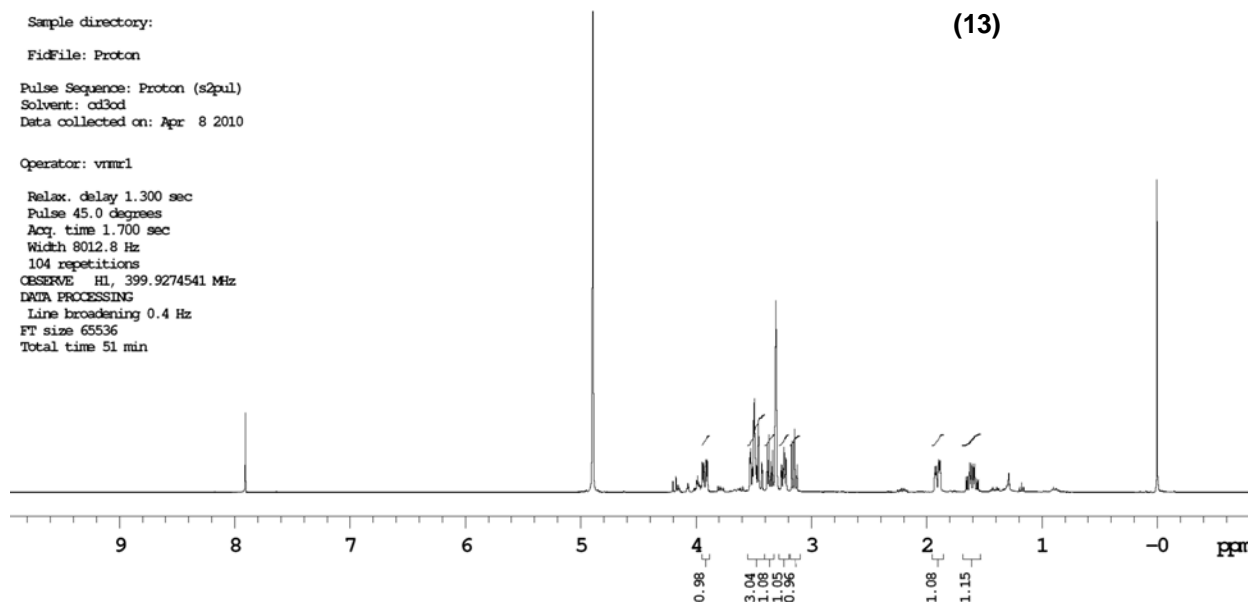
Pulse Sequence: Proton (s2pul)
Solvent: cd3od
Data collected on: Apr 8 2010

Operator: vrmr1

Relax. delay 1.300 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 8012.8 Hz
104 repetitions
OBSERVE H1, 399.9274541 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 65536
Total time 51 min



(13)



ADV-P-247-115-B-13C
18 September 2009
C13 EXPERIMENT

Sample Name:

Data Collected on:
wormhole-vrms400
Archive directory:

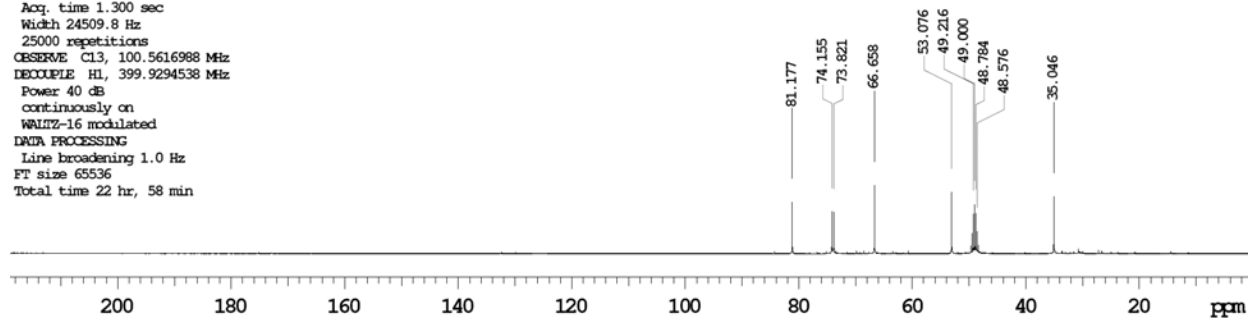
Sample directory:

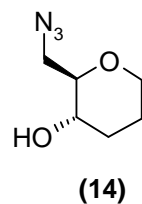
FidFile: ADV-P-247-115-B-C13

Pulse Sequence: Carbon (s2pul)
Solvent: cd3od
Data collected on: Sep 18 2009

Temp. 25.0 C / 298.1 K
Operator: vrmr1

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
25000 repetitions
OBSERVE C13, 100.5616988 MHz
DECOUPLE H1, 399.9294538 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 22 hr, 58 min





ADV-P-247-115-A
16 September 2009
Advinus pune

Sample Name:

Data Collected on:
wormhole-vrms400
Archive directory:

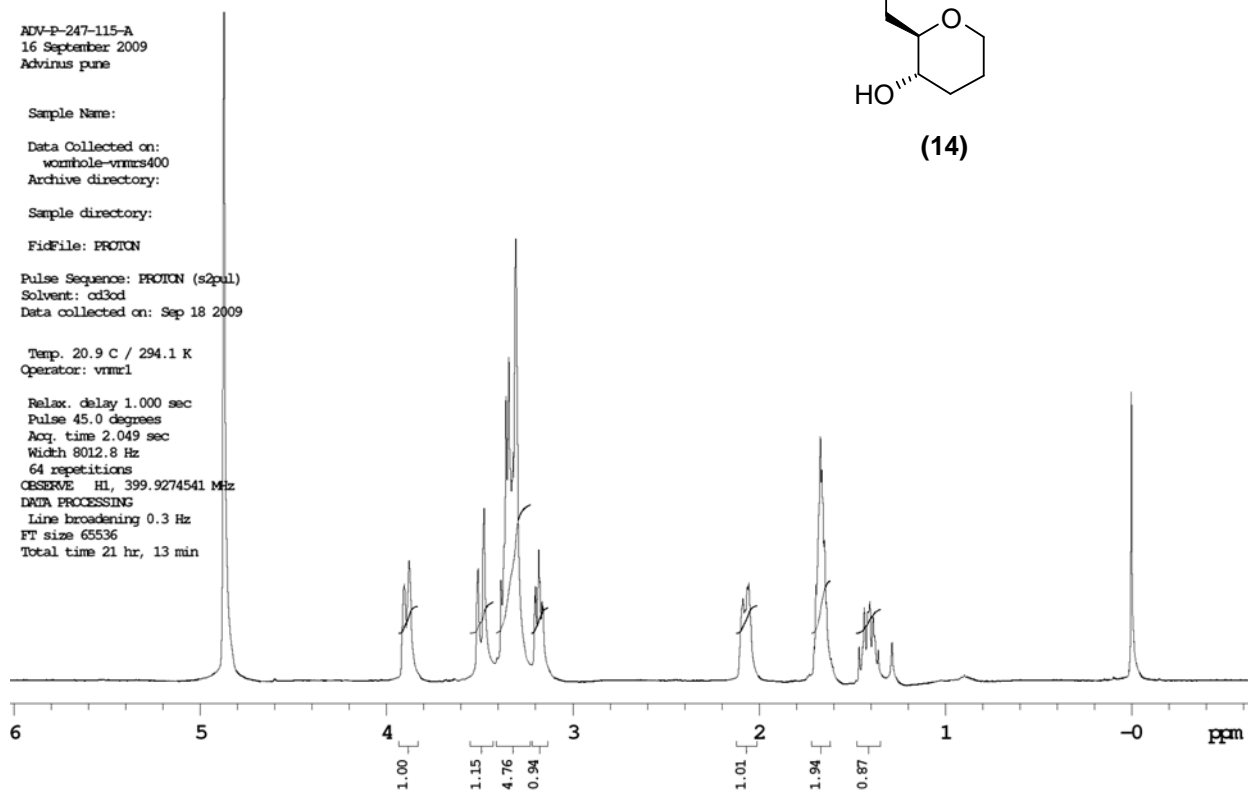
Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cd3od
Data collected on: Sep 18 2009

Temp. 20.9 C / 294.1 K
Operator: vrm1

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.049 sec
Width 8012.8 Hz
64 repetitions
OBSERVE H1, 399.9274541 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 21 hr, 13 min



ADV-P-247-115-A-13C
18 September 2009
C13 EXPERIMENT

Sample Name:

Data Collected on:
wormhole-vrms400
Archive directory:

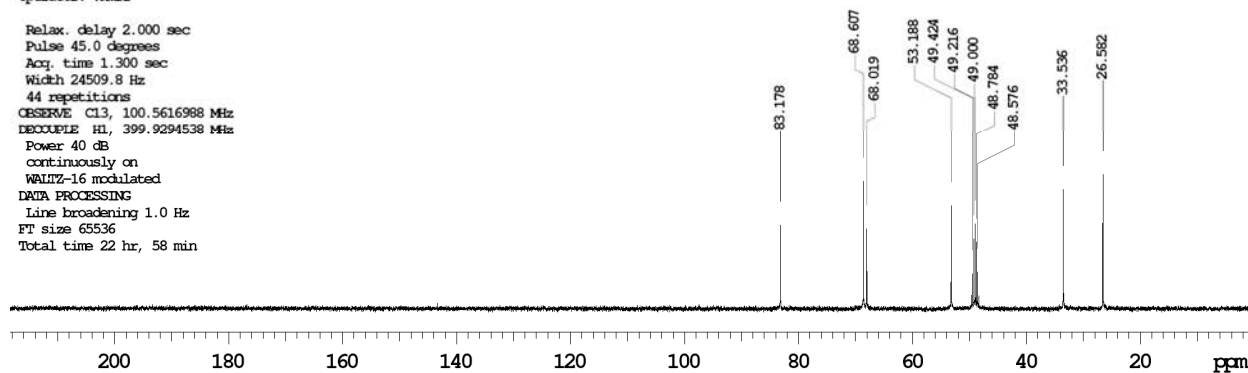
Sample directory:

FidFile: ADV-P-247-115-A-13C

Pulse Sequence: Carbon (s2pul)
Solvent: cd3od
Data collected on: Sep 18 2009

Temp. 25.0 C / 298.1 K
Operator: vrm1

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
44 repetitions
OBSERVE C13, 100.5616988 MHz
DECOUPLE H1, 399.9294538 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 22 hr, 58 min



ADV-P-247-117-A
01 Oct 2009
Advirus Therapeutics Pune

Sample Name:
C-01158-080
Data Collected on:
wormhole-vnmrs400
Archive directory:

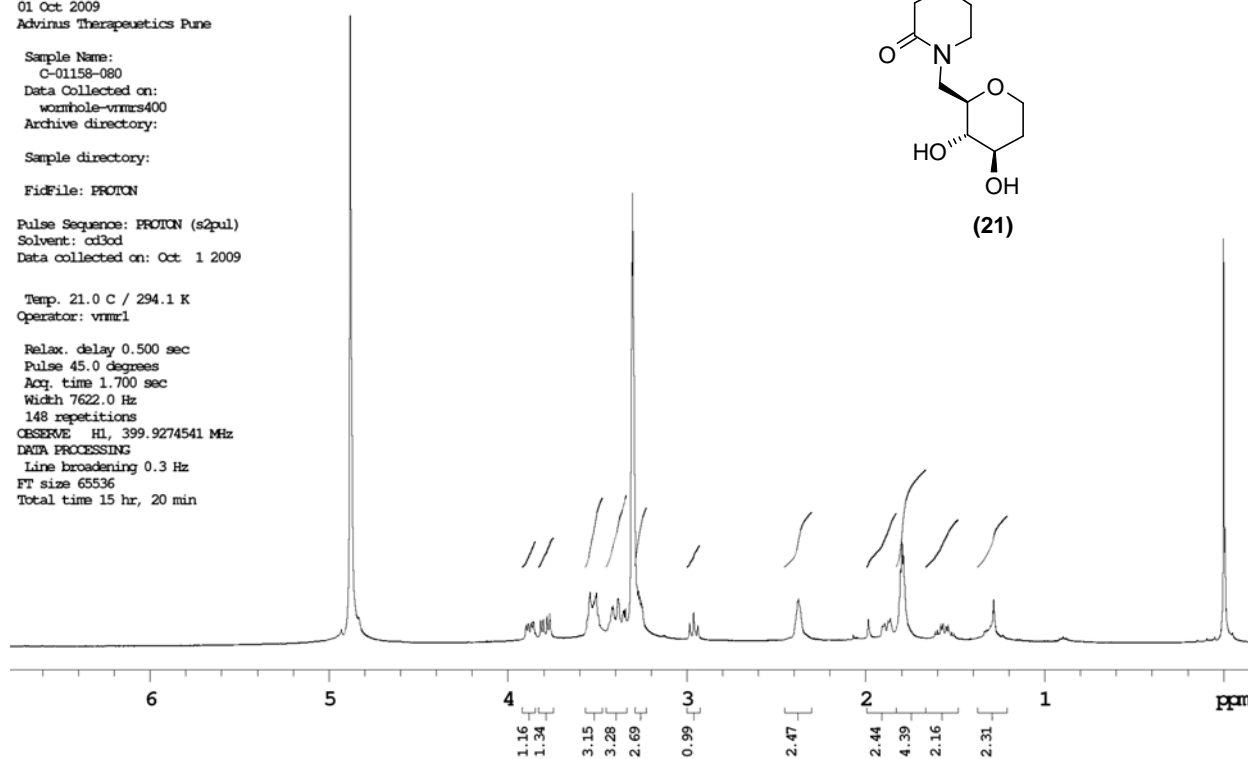
Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cd3od
Data collected on: Oct 1 2009

Temp. 21.0 C / 294.1 K
Operator: vnmr1

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 7622.0 Hz
148 repetitions
OBSERVE H1, 399.9274541 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 15 hr, 20 min



ADV-P-247-117A
26 September 2009
C13 EXPERIMENT

Sample Name:

Data Collected on:
wormhole-vnmrs400
Archive directory:

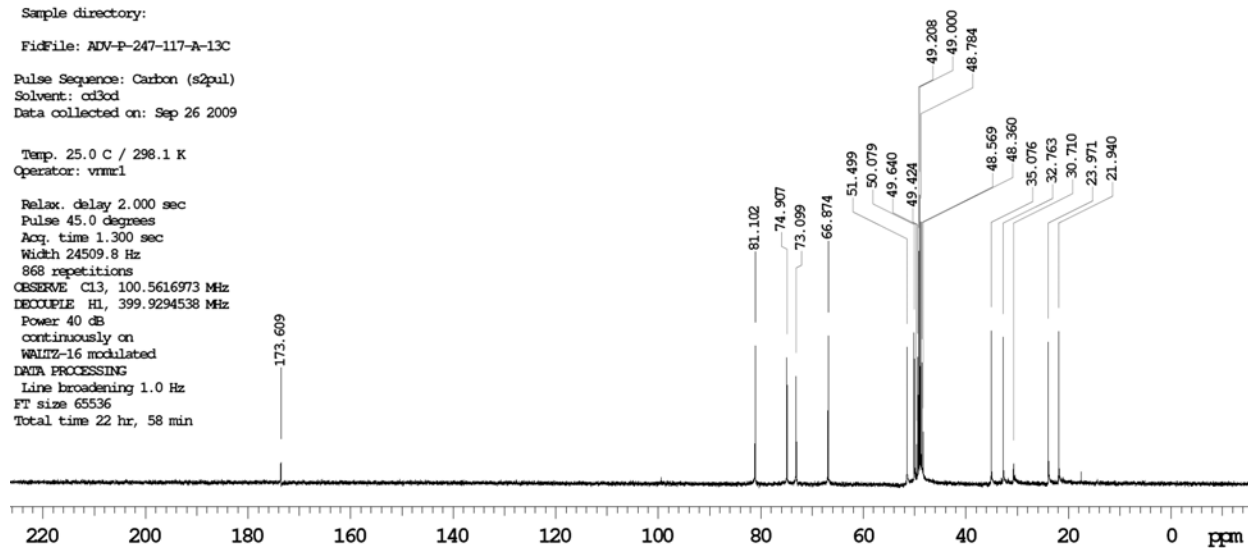
Sample directory:

FidFile: ADV-P-247-117-A-13C

Pulse Sequence: Carbon (s2pul)
Solvent: cd3od
Data collected on: Sep 26 2009

Temp. 25.0 C / 298.1 K
Operator: vnmr1

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
868 repetitions
OBSERVE C13, 100.5616973 MHz
DECOUPLE H1, 399.9294538 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 22 hr, 58 min



ADV-P-247-116
01 Oct 2009
Advinus Therapeutics Pune

Sample Name:
C-01158-080
Data Collected on:
wormhole-vrms400
Archive directory:

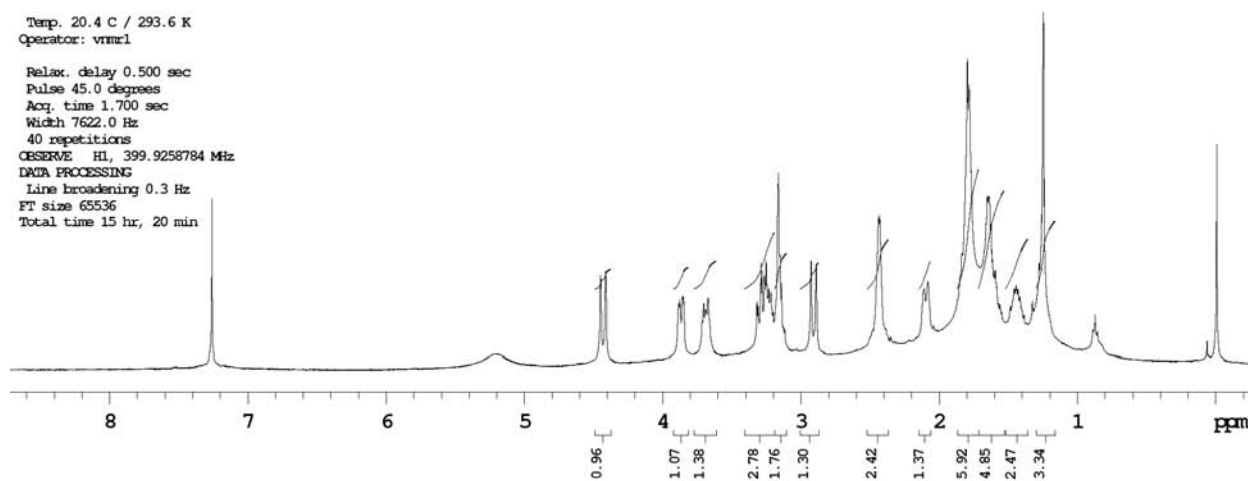
Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2009

Temp. 20.4 C / 293.6 K
Operator: vrml

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 7622.0 Hz
40 repetitions
OBSERVE H1, 399.9258784 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 15 hr, 20 min



ADV-P-247-116
30 September 2009
C13 EXPERIMENT

Sample Name:

Data Collected on:
wormhole-vrms400
Archive directory:

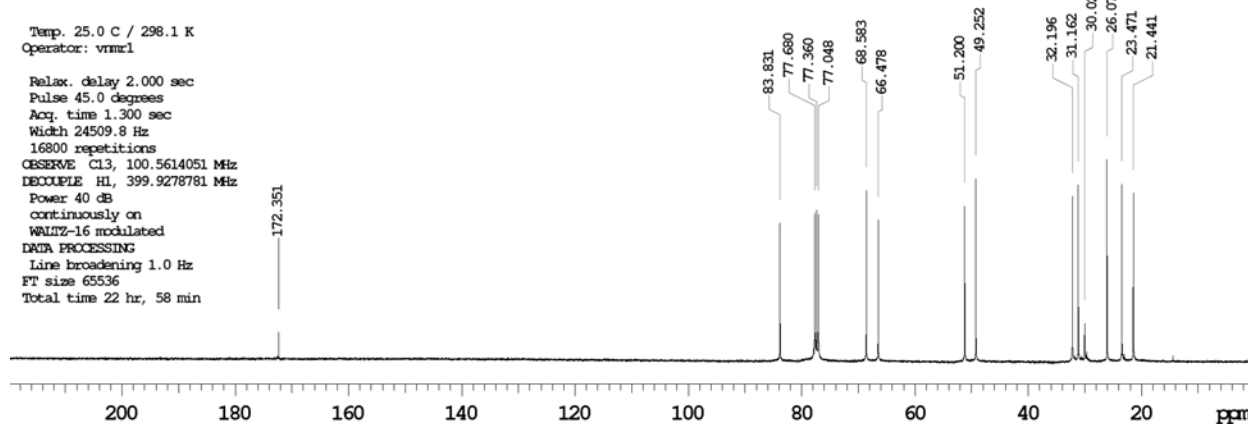
Sample directory:

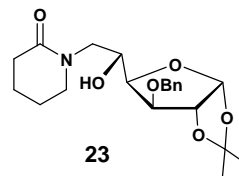
FidFile: ADV-P-247-116-C13

Pulse Sequence: Carbon (s2pul)
Solvent: cdcl3
Data collected on: Sep 30 2009

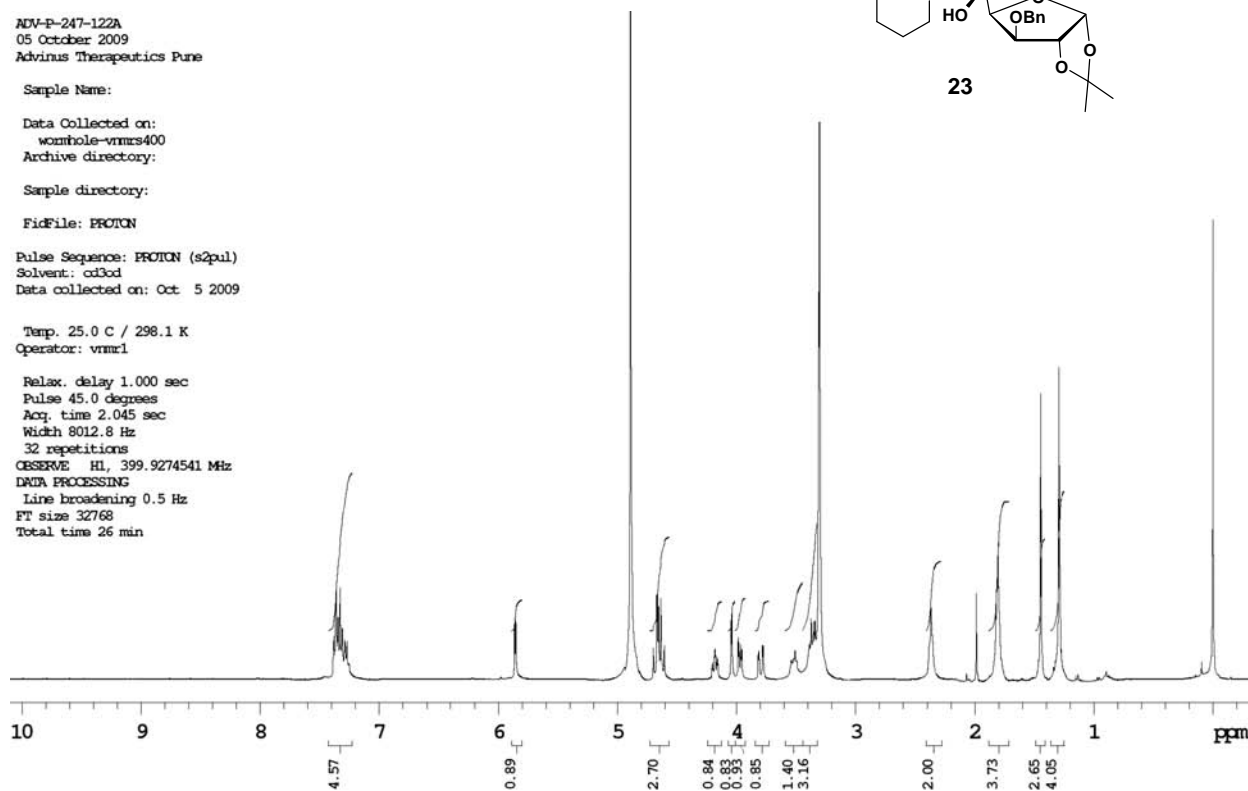
Temp. 25.0 C / 298.1 K
Operator: vrml

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
16800 repetitions
OBSERVE C13, 100.5614051 MHz
DECOUPLE H1, 399.9278781 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 22 hr, 58 min

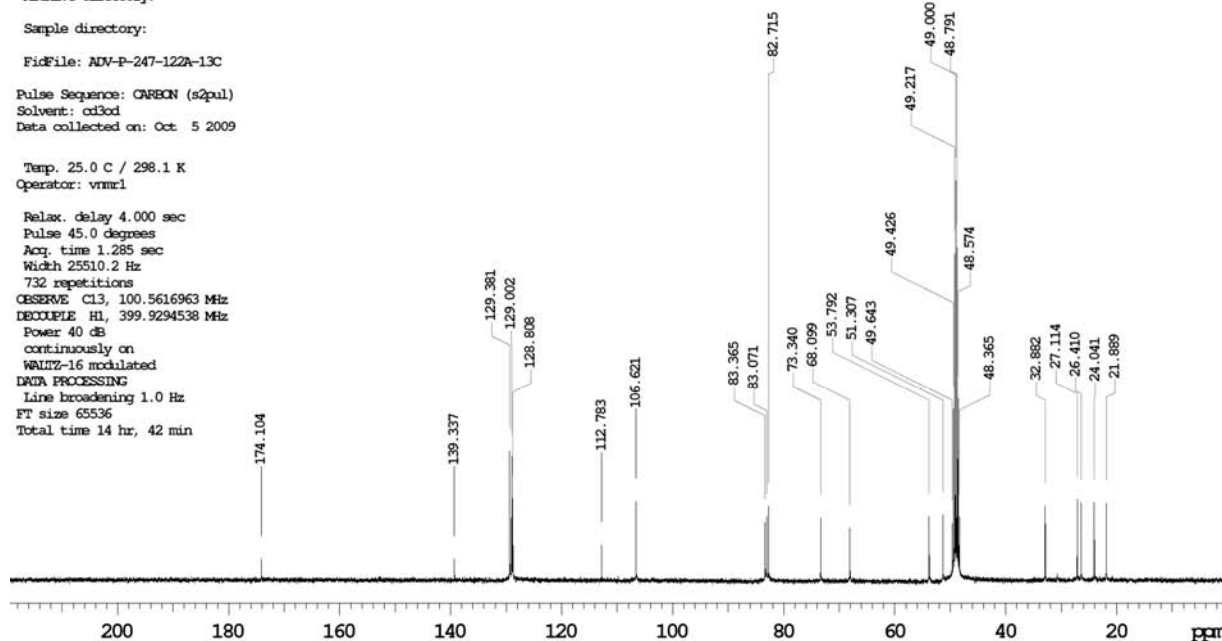




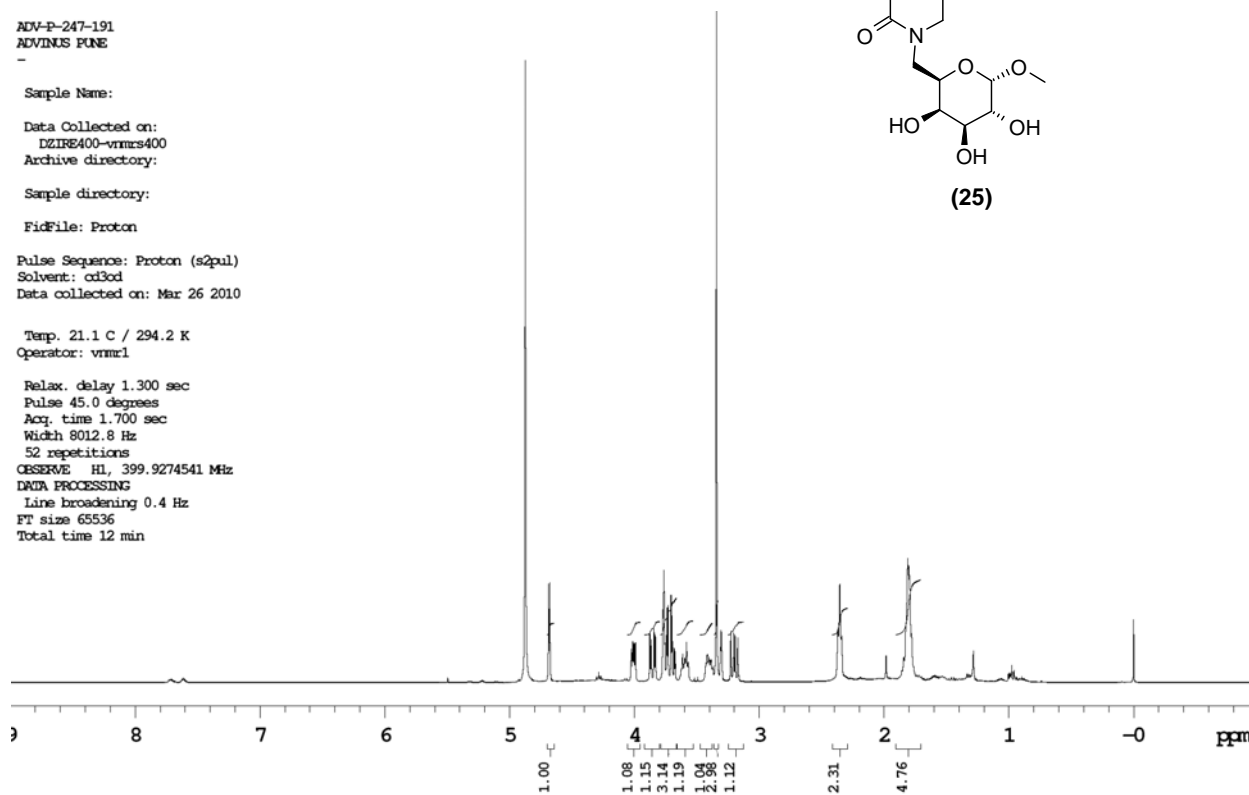
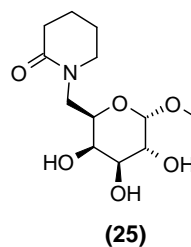
ADV-P-247-122A
 05 October 2009
 Advirus Therapeutics Pune
 Sample Name:
 Data Collected on:
 womhole-vnmrs400
 Archive directory:
 Sample directory:
 FidFile: PROTON
 Pulse Sequence: PROTON (s2pul)
 Solvent: cd3od
 Data collected on: Oct 5 2009
 Temp. 25.0 C / 298.1 K
 Operator: vnmr1
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.045 sec
 Width 8012.8 Hz
 32 repetitions
 OBSERVE H1, 399.9274541 MHz
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 32768
 Total time 26 min



ADV-P-247-122A
 STANDARD CARBON PARAMETERS
 Sample Name:
 Data Collected on:
 womhole-vnmrs400
 Archive directory:
 Sample directory:
 FidFile: ADV-P-247-122A-13C
 Pulse Sequence: CARBON (s2pul)
 Solvent: cd3od
 Data collected on: Oct 5 2009
 Temp. 25.0 C / 298.1 K
 Operator: vnmr1
 Relax. delay 4.000 sec
 Pulse 45.0 degrees
 Acq. time 1.285 sec
 Width 25510.2 Hz
 732 repetitions
 OBSERVE C13, 100.5616963 MHz
 DECOUPLE H1, 399.9294538 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 14 hr, 42 min

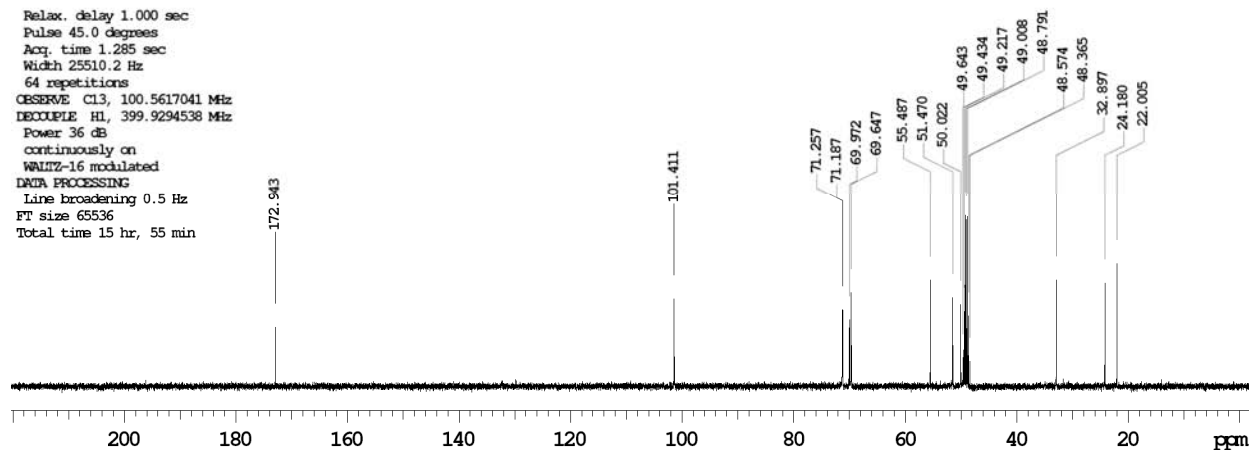


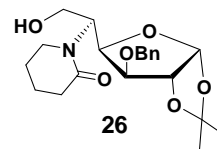
ADV-P-247-191
ADVINS FUNE
-
Sample Name:
Data Collected on:
DZIRE400-vnmrs400
Archive directory:
Sample directory:
FidFile: Proton
Pulse Sequence: Proton (s2pul)
Solvent: cd3od
Data collected on: Mar 26 2010
Temp. 21.1 C / 294.2 K
Operator: vnmr1
Relax. delay 1.300 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 8012.8 Hz
52 repetitions
OBSERVE H1, 399.9274541 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 65536
Total time 12 min



ADV-P-247-191
13C Experiment
Sample Name:
Data Collected on:
DZIRE400-vnmrs400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cd3od
Data collected on: Mar 26 2010

Operator: vnmr1
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
64 repetitions
OBSERVE C13, 100.5617041 MHz
DECOUPLE H1, 399.9294538 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 15 hr, 55 min





ADV-P-418-085-P
 Advinus Therapeutics Pune

Sample Name:

Data Collected on:
 DZIRE400-vnmrs400
 Archive directory:

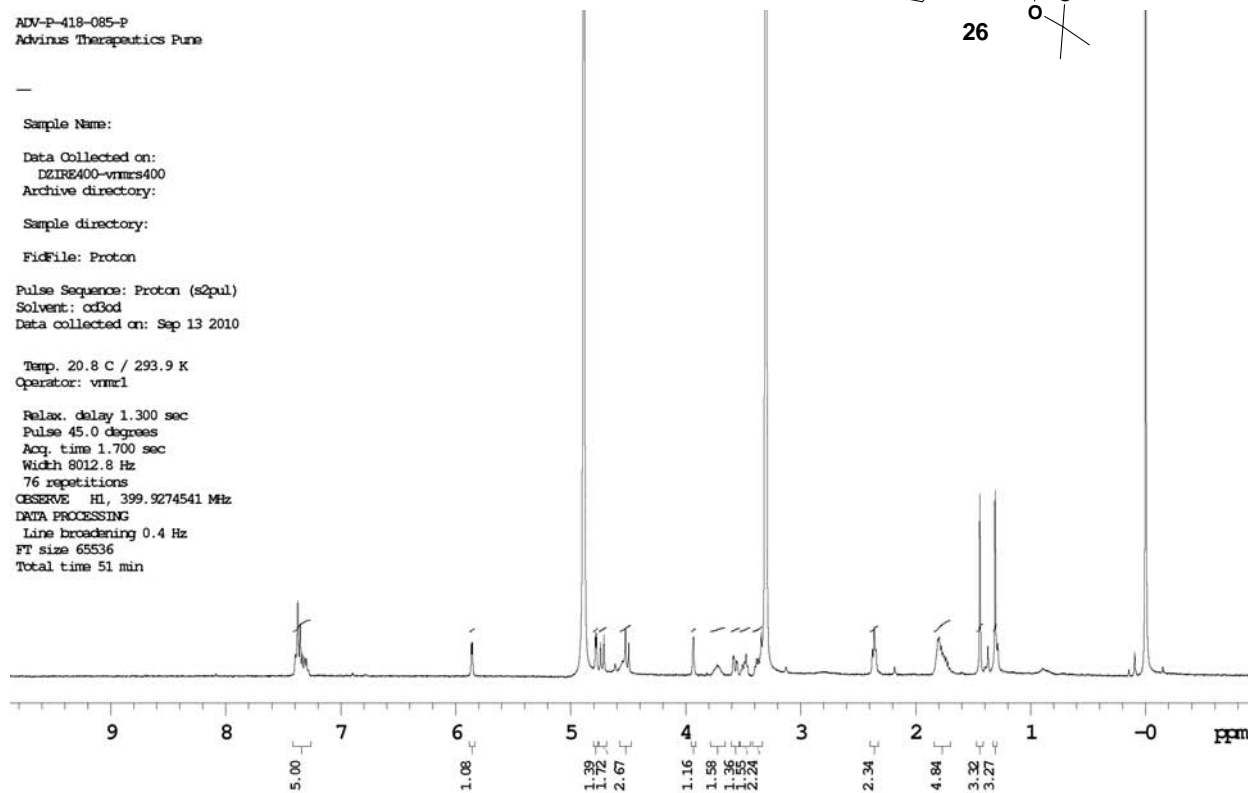
Sample directory:

FidFile: Proton

Pulse Sequence: Proton (s2pul)
 Solvent: cd3od
 Data collected on: Sep 13 2010

Temp. 20.8 C / 293.9 K
 Operator: vnmr1

Relax. delay 1.300 sec
 Pulse 45.0 degrees
 Acq. time 1.700 sec
 Width 8012.8 Hz
 76 repetitions
 OBSERVE HI, 399.9274541 MHz
 DATA PROCESSING
 Line broadening 0.4 Hz
 FT size 65536
 Total time 51 min



ADV-P-418-085-P_13C
 13C Experiment

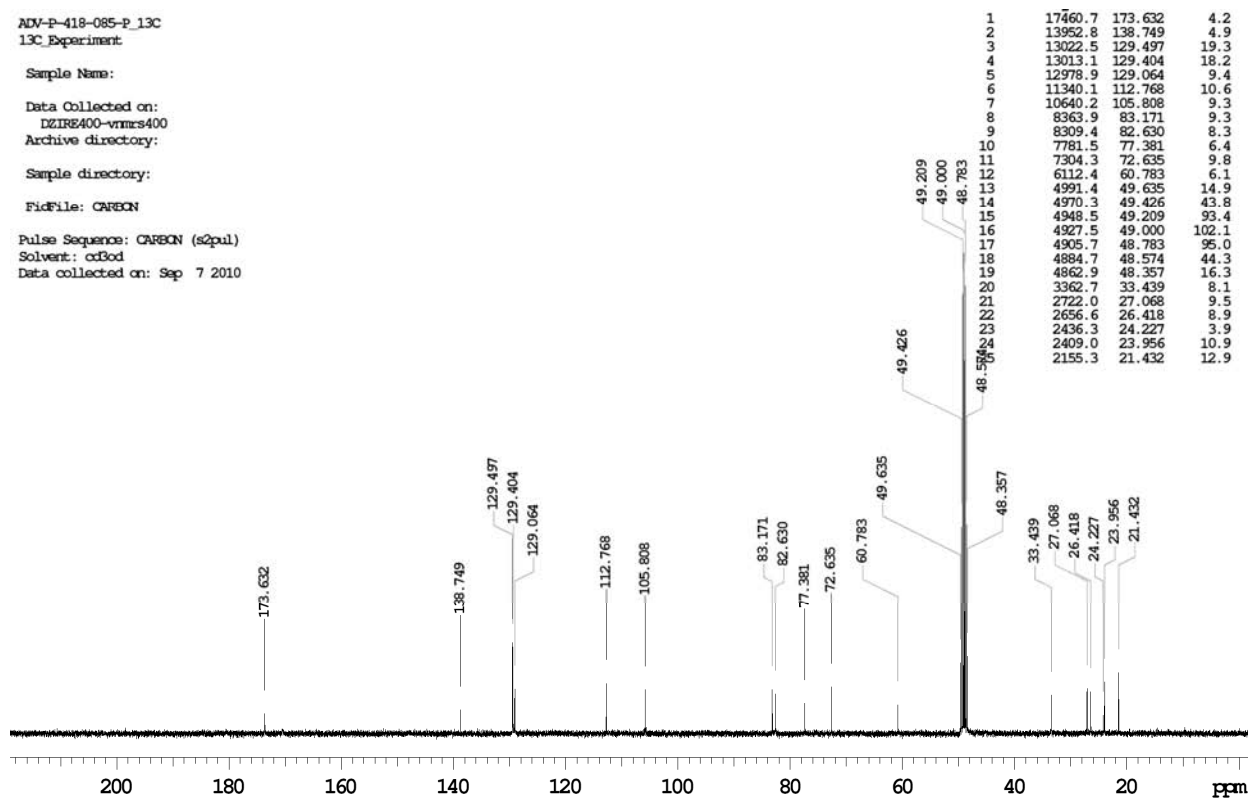
Sample Name:

Data Collected on:
 DZIRE400-vnmrs400
 Archive directory:

Sample directory:

FidFile: CARBON

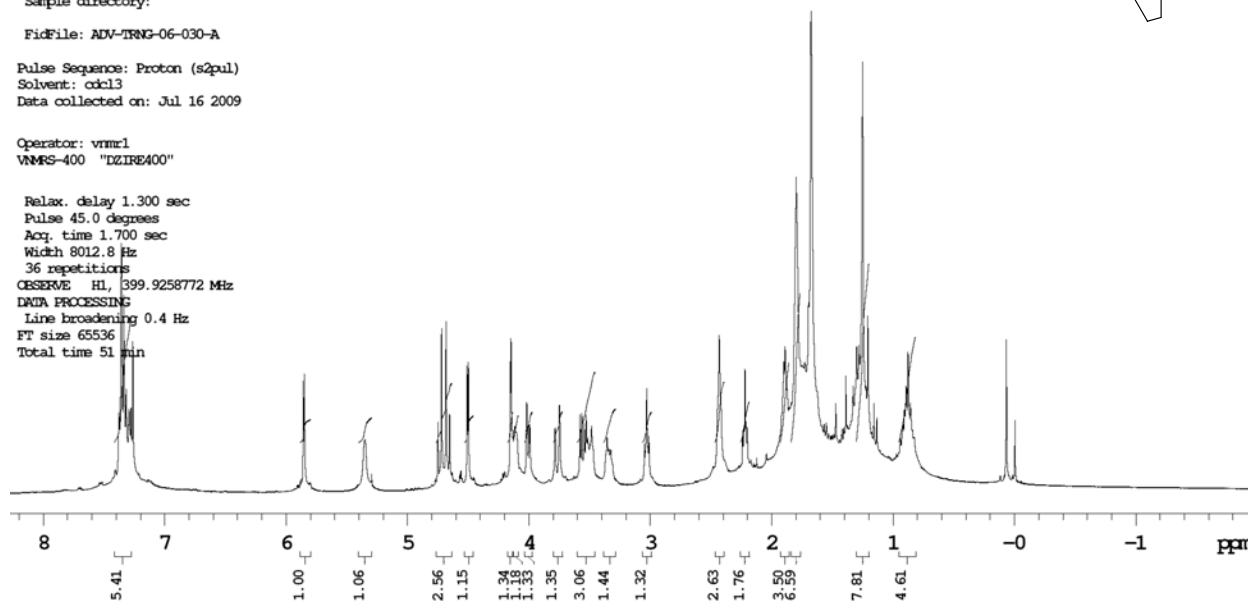
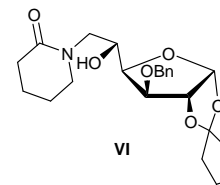
Pulse Sequence: CARBON (s2pul)
 Solvent: cd3od
 Data collected on: Sep 7 2010



ADV-TRNG-06-030-A
16 July 2009
Advirus Therapeutics Pune

Sample Name:
Archive directory:
Sample directory:
FidFile: ADV-TRNG-06-030-A
Pulse Sequence: Proton (s2pul)
Solvent: cdcl3
Data collected on: Jul 16 2009

Operator: vnmr1
VNMR-400 "DZIRE400"
Relax. delay 1.300 sec
Pulse 45.0 degrees
Acq. time 1.700 sec
Width 8012.8 Hz
36 repetitions
OBSERVE HL, 399.9258772 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 65536
Total time 51 min

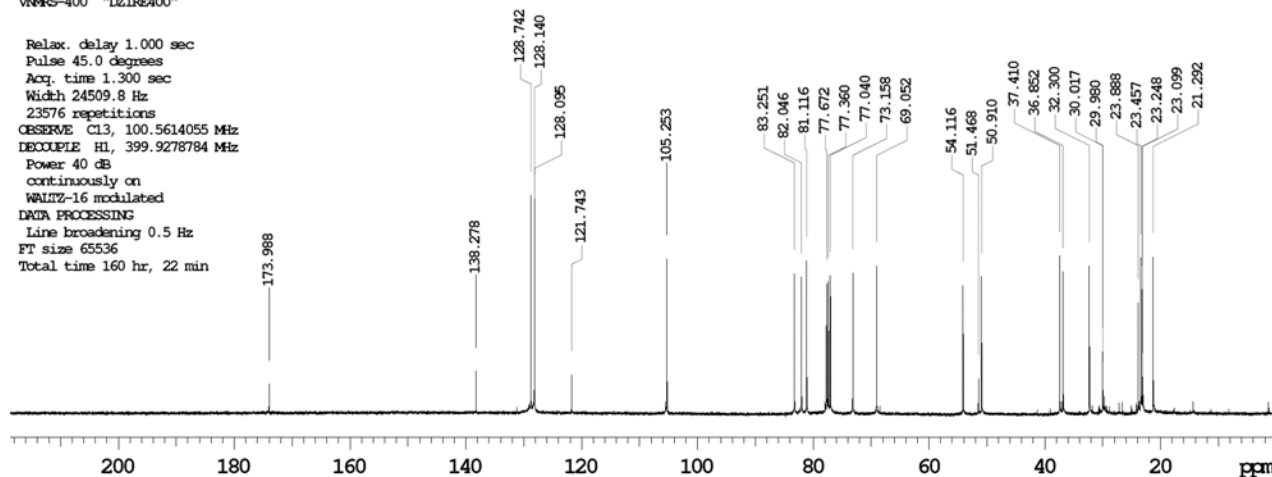


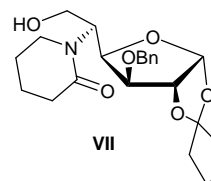
399

ADV-TRNG-06-030-A
16 July 2009
C13 EXPERIMENT

Sample Name:
Archive directory:
Sample directory:
FidFile: ADV-TRNG-060-030-A-C13
Pulse Sequence: Carbon (s2pul)
Solvent: cdcl3
Data collected on: Jul 16 2009

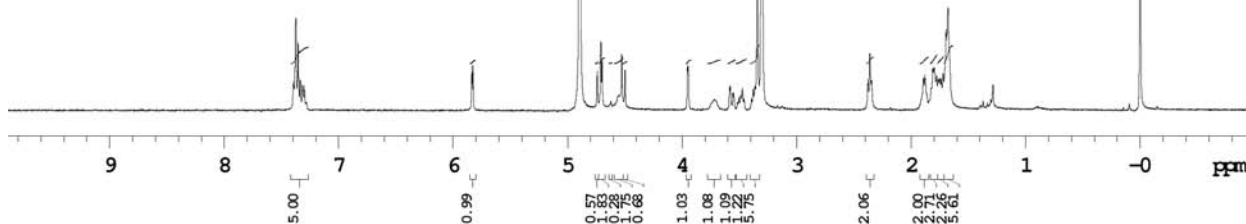
Operator: vnmr1
VNMR-400 "DZIRE400"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 24509.8 Hz
23576 repetitions
OBSERVE C13, 100.5614055 MHz
DECOUPLE HL, 399.9278784 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 160 hr, 22 min





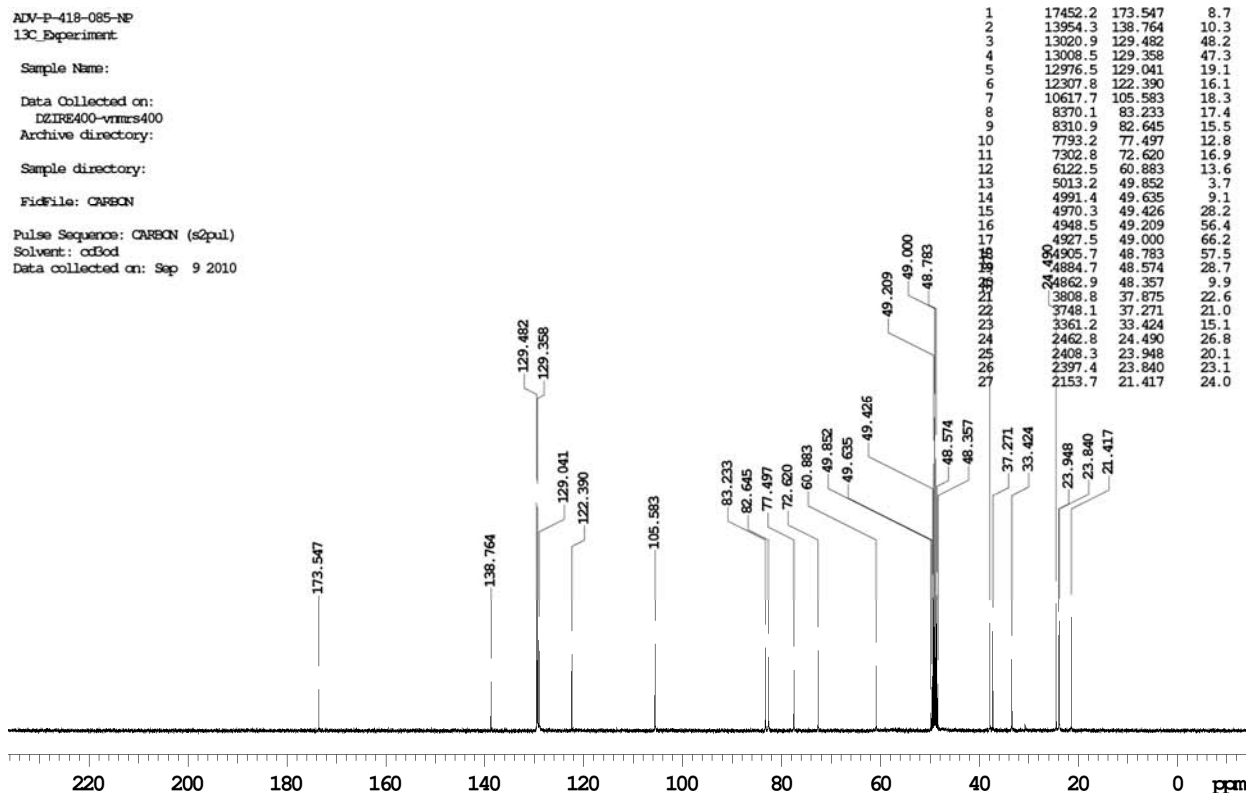
ADV-P-418-085-NP
 Advinus Therapeutics Pune

Sample Name:
 Data Collected on:
 DZIRE400-vrms400
 Archive directory:
 Sample directory:
 FidFile: Proton
 Pulse Sequence: Proton (s2pul)
 Solvent: cd3od
 Data collected on: Sep 13 2010
 Operator: vrml
 Relax. delay 1.300 sec
 Pulse 45.0 degrees
 Acq. time 1.700 sec
 Width 8012.8 Hz
 3 repetitions
 OBSERVE H1, 399.9274311 Mhz
 DATA PROCESSING
 Line broadening 0.4 Hz
 FT size 65536
 Total time 51 min



ADV-P-418-085-NP
 13C Experiment

Sample Name:
 Data Collected on:
 DZIRE400-vrms400
 Archive directory:
 Sample directory:
 FidFile: CARBON
 Pulse Sequence: CARBON (s2pul)
 Solvent: cd3od
 Data collected on: Sep 9 2010



ADV-P-247-178-02
 Advinus pune

Sample Name:

Data Collected on:
 DZIRE400-vnmrs400
 Archive directory:

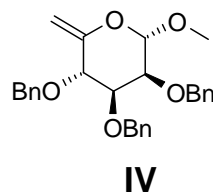
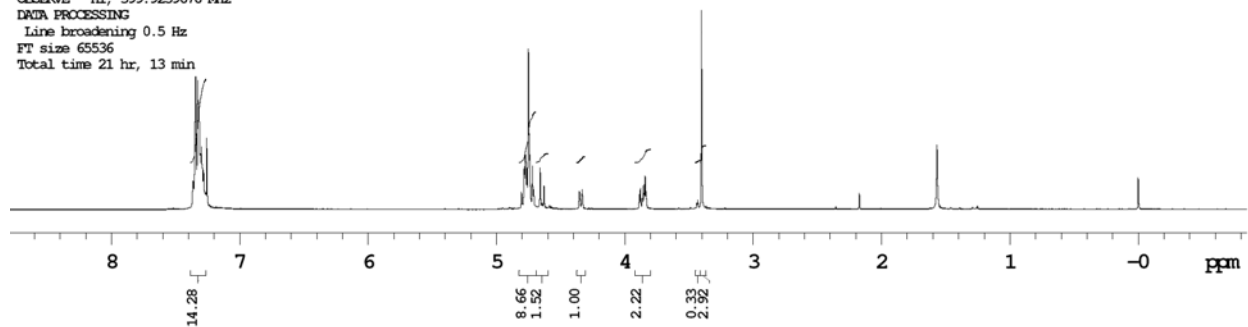
Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Mar 2 2010

Temp. 20.3 C / 293.4 K
 Operator: vnmr1

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 8012.8 Hz
 40 repetitions
 OBSERVE H1, 399.9259676 MHz
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 21 hr, 13 min



ADV-P-247-178-02
 C13 EXPERIMENT
 ADVINUS PUNE

Sample Name:

Data Collected on:
 DZIRE400-vnmrs400
 Archive directory:

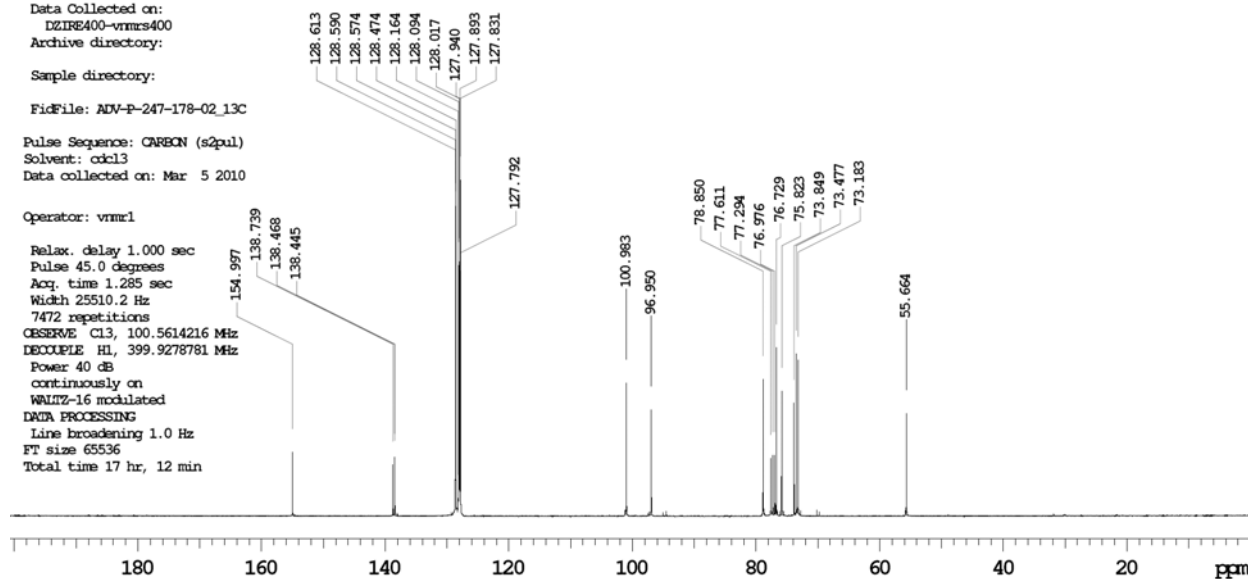
Sample directory:

FidFile: ADV-P-247-178-02_13C

Pulse Sequence: CARBON (s2pul)
 Solvent: cdcl3
 Data collected on: Mar 5 2010

Operator: vnmr1

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.285 sec
 Width 25510.2 Hz
 7472 repetitions
 OBSERVE C13, 100.5614216 MHz
 DECOUPLE H1, 399.9278781 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 17 hr, 12 min



ADV-P-247-178-01
 ADVINUS FUNE

Sample Name:

Data Collected on:
 DZIRE400-vrms400
 Archive directory:

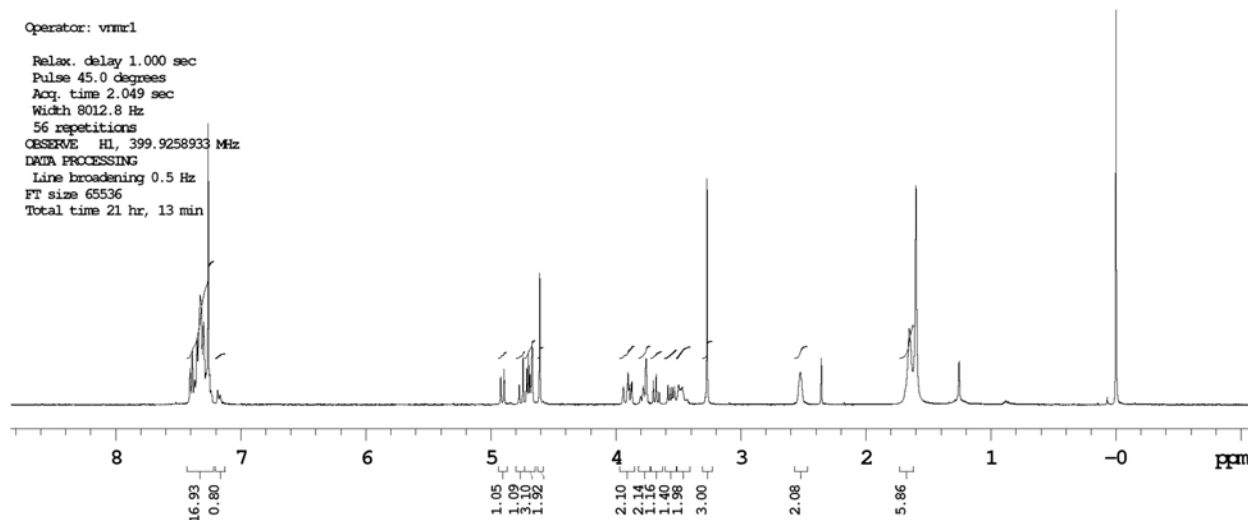
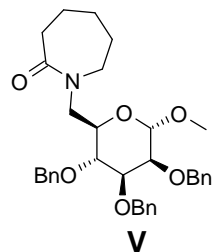
Sample directory:

FidFile: PROTON

Pulse Sequence: PROTON (s2pul)
 Solvent: cdcl3
 Data collected on: Mar 9 2010

Operator: vmr1

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.049 sec
 Width 8012.8 Hz
 56 repetitions
 OBSERVE H1, 399.9258933 MHz
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 65536
 Total time 21 hr, 13 min



ADV-P-247-178-01
 C13 EXPERIMENT
 ADVINUS FUNE

Sample Name:

Data Collected on:
 DZIRE400-vrms400
 Archive directory:

Sample directory:

FidFile: ADV-P-247-178-01_13C..

Pulse Sequence: CARECN (s2pul)
 Solvent: cdcl3
 Data collected on: Mar 9 2010

Operator: vmr1

Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.285 sec
 Width 25510.2 Hz
 1780 repetitions
 OBSERVE C13, 100.5609276 MHz
 DECOUPLE H1, 399.9278781 MHz
 Power 40 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 17 hr, 12 min

