Supporting Information

An approach to 8 stereoisomers of homonojirimycin from D-glucose *via* kinetic & thermodynamic azido-γ-lactones

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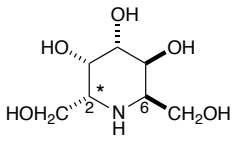
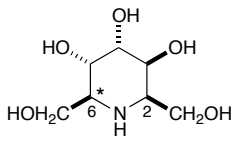
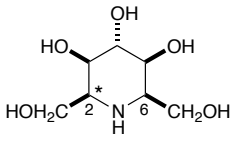
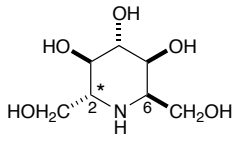
30   **31**

Azido-ketose **27** was hydrogenated in water with a nickel catalyst under similar conditions to those described by Izumori and co-workers[1] yielding a 1:25 mixture of **30 / 31.** The mixture was separated affording **31** (38 mg) as colourless glass.

Data for **31**:

HRMS *m/z* (ESI$^+$): found 194.1022 $[M+H]^+$; $C_7H_{16}NO_5$ requires 194.1023; $[\alpha]_D^{20}$ -44.1 (*c*, 1.09 in H_2O) {Lit.[4] ($[\alpha]_D^{23}$ -41.0 (*c*, 0.69 in H_2O)}; ν_{max} (thin film, Ge): 3279 (br, s, OH, NH); δ_H (D_2O, 400 MHz): 2.86 (1H, ddd, H6, $J_{6,5}$ 10.5, $J_{6,7}$ 5.3, $J_{6,7'}$ 3.2), 3.05 (1H, dt, H2, $J_{2,1}$ 6.7, $J_{2,3}$ 1.3), 3.62 (1H, dd, H1, $J_{1,1'}$11.2, $J_{1,2}$ 6.7), 3.66 (1H, dd, H1', $J_{1,1'}$11.2, $J_{1,2}$ 6.7), 3.70 (1H, dd, H7, $J_{7,7'}$10.6, $J_{7,6}$ 5.6), 3.75 (1H, dd, H5, $J_{5,6}$ 10.6, $J_{5,4}$ 3.3), 3.79 (1H, dd, H7', $J_{7',7}$ 11.6, $J_{7',6}$ 3.0), 3.91 (1H, dd, H3, $J_{3,4}$ 3.8, $J_{3,2}$ 1.3), 3.99 (1H, a-t, H4, *J* 3.5); δ_C (D_2O, 100.6 MHz): 54.4 (C2), 55.8 (C6), 62.1 (C1+C7, 66.7 (C5), 68.0 (C3), 71.6 (C4).

2,6-Dideoxy-2,6-imino-D-*glycero*-L-*ido*-heptitol (32) and 2,6-dideoxy-2,6-imino-D-*glycero*-L-*gulo*-heptitol (α-homonojirimycin) (4)

32   **4**

Azido-ketose **26** was hydrogenated in water with a nickel catalyst under similar conditions to those described by Izumori and co-workers[1] yielding a 3:1 mixture of **32 / 4.** The mixture was separated affording **32** (0.8 g) and **4** (95 mg), both as white solids.

Data for **32**:

HRMS *m/z* (ESI$^+$): found 340.1471 $[M+Na]^+$; $C_{13}H_{23}N_3NaO_6$ requires 340.1479; m.p. 169-172°C (from methanol); $[\alpha]_D^{25}$ +0.53 (*c*, 1.04 in H_2O); ν_{max} (thin film, Ge): 3332 (br, s, OH, NH); δ_H (D_2O, 400 MHz): 3.07 (2H, dt, H2, H6, $J_{2,1}/J_{6,7} = J_{2,1'}/J_{6,7'}$ = 6.6, $J_{2,3}/J_{6,5}$ 1.5), 3.65 (2H, dd, H1, H7, $J_{1,1'}/J_{7,7'}$ 11.1, $J_{1,2}/J_{7,6}$ 6.9), 3.72 (2H, dd, H1', H7', $J_{1',1}/J_{7',7}$ 11.1, $J_{1',2}/J_{7',6}$ 6.4), 3.78 (2H, dd, H3, H5, $J_{3,4}/J_{5,4}$

3.2, $J_{3,2}/J_{5,6}$ 1.5), 4.01 (1H, t, H4, $J_{4,3} = J_{4,5} = 3.2$); δ_C (D_2O, 100.6 MHz): 55.6 (C2, C6), 62.4 (C1, C7), 68.9 (C3, C5), 69.4 (C4); *m/z* (ESI^+): 194 ($[M+H]^+$, 100%), 216 ($[M+Na]^+$, 72%).

For literature comparison a sample of **32** was converted to the HCl salt.

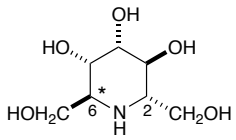
$[\alpha]_D^{21}$ 0.00 (*c*, 1.27 in MeOH) {Lit.[2] $[\alpha]_D^{20}$ 0.0 (*c*, 0.8 in MeOH)}; δ_H (CD_3OD, 400 MHz): 3.52-3.56 (2H, m, H2, H6), 3.86 (2H, dd, H1, H7, $J_{1,1'}/J_{7,7'}$ 11.4, $J_{1,2}/J_{7,6}$ 6.2), 3.90 (2H, dd, H1', H7', $J_{1',1}/J_{7',7}$ 11.4, $J_{1',2}/J_{7',6}$ 7.6), 3.97-3.98 (2H, m, H3, H5), 4.02 (1H, t, H4, $J_{4,3} = J_{4,5} = 3.5$); δ_C (CD_3OD, 100.6 MHz): 58.4 (C2, C6), 60.1 (C1, C7), 68.0 (C4), 69.1 (C3, C5).

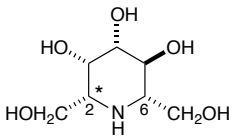
Data for **4**:

HRMS *m/z* (FI^+): found 193.0946 $[M]^{\bullet+}$; $C_7H_{15}NO_5$ requires 193.0950; m.p. 200-202°C (from methanol); $[\alpha]_D^{25}$ +61.0 (*c*, 0.56 in H_2O) {Lit.[3] ($[\alpha]_D$ +77.2 (*c*, 0.57 in H_2O)}; ν_{max} (thin film, Ge): 3319 (br, s, OH, NH); δ_H (D_2O, 400 MHz): 2.84 (1H, ddd, H6, $J_{6,5}$ 10.0, $J_{6,7}$ 7.2, $J_{6,7'}$ 2.9), 3.19 (1H, t, H5, $J_{5,6} = J_{5,4} = 9.6$), 3.27 (1H, dt, H2, $J_{2,1}$ 9.4, $J_{2,1'} = J_{2,3} = 5.5$), 3.48 (1H, t, H4, $J_{4,3} = J_{4,5} = 9.5$), 3.56 (1H, dd, H7, $J_{7,7'}$ 11.4, $J_{7,6}$ 7.1), 3.74 (1H, dd, H3, $J_{3,4}$ 10.0, $J_{3,2}$ 5.8), 3.77-3.84 (2H, m, H1, H1'), 3.90 (1H, dd, H7', $J_{7',7}$ 11.4, $J_{7',6}$ 2.9); δ_C (D_2O, 100.6 MHz): 54.6 (C6), 56.8 (C1), 57.4 (C2), 62.6 (C7), 72.1 (C3), 72.7 (C5), 74.9 (C4).

2,6-Dideoxy-2,6-imino-L-*glycero*-L-*manno*-heptitol (33) and 2,6-dideoxy-2,6-imino-L-*glycero*-L-*galacto*-heptitol (34)

33





34

Azido-ketose **29** was hydrogenated in water with a nickel catalyst under similar conditions to those described by Izumori and co-workers[1] yielding a 14:1 mixture of **34** / **33**. The mixture was separated affording **34** (9 mg), as a colourless oil.

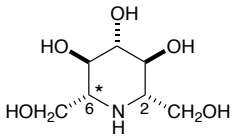
Data for **34**:

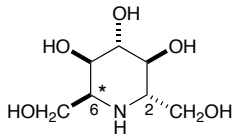
HRMS *m/z* (ESI^+): found 216.0842 $[M+Na]^+$; $C_7H_{15}NNaO_5$ requires 216.0842; $[\alpha]_D^{25}$ -10.7 (*c*, 0.27 in H_2O), $[\alpha]_D^{25}$ +2.47 (*c*, 0.55 in MeOH) {Lit.[4] $[\alpha]_D^{25}$ +4.0 (c, 1.0 in MeOH)}; ν_{max} (thin film, Ge): 3332 (br, s, OH, NH); δ_H (D_2O, 400 MHz): 2.56 (1H, dd, H6, $J_{6,5}$ 9.8, $J_{6,7}$ 5.4, $J_{6,7'}$ 2.8), 2.84 (1H, dt, H2, $J_{2,1} = J_{2,1'} = 6.7$, $J_{2,3}$ 1.4), 3.54 (1H, dd, H4, $J_{4,5}$ 9.8, $J_{4,3}$ 2.8), 3.54-3.59 (1H, m, H5), 3.65 (1H, dd, H1, $J_{1,1'}$ 11.1, $J_{1,2}$ 6.7), 3.66 (1H, dd, H1', $J_{1',1}$ 11.2, $J_{1',2}$ 6.7), 3.72 (1H, dd, H7, $J_{7,7'}$ 11.6, $J_{7,6}$ 5.5), 3.82 (1H, dd, H7', $J_{7',7}$ 11.6, $J_{7',6}$ 3.0), 3.99 (1H, dd, H3, $J_{3,4}$ 2.6, $J_{3,2}$ 1.3); δ_C (D_2O, 100.6 MHz): 58.5 (C2), 60.7 (C6), 61.6 (C7), 62.0 (C1), 69.2 (C5), 69.6 (C3), 75.6 (C4); δ_H (CD_3OD, 500 MHz): 2.47 (1H, ddd, H6, $J_{6,5}$ 9.6, $J_{6,7}$ 6.2, $J_{6,7'}$ 3.1), 2.73 (1H, ddd, H2, $J_{2,1}$ 6.8, $J_{2,1'}$ 6.2, $J_{2,3}$ 1.4), 3.36 (1H, dd, H4, $J_{4,5}$ 9.5,

$J_{4,3}$ 3.2), 3.52 (1H, t, H5, $J_{5,6} = J_{5,4}$ = 9.6), 3.63 (1H, dd, H1, $J_{1,1'}$ 10.8, $J_{1,2}$ 6.8), 3.66 (1H, dd, H1', $J_{1',1}$ 10.8, $J_{1',2}$ 6.3), 3.66 (1H, dd, H7, $J_{7,7'}$ 10.9, $J_{7,6}$ 6.2), 3.84 (1H, dd, H7', $J_{7',7}$ 10.9, $J_{7',6}$ 3.0), 3.87 (1H, dd, H3, $J_{3,4}$ 3.1, $J_{3,2}$ 1.4); δ_C (CD_3OD, 125.8 MHz): 60.7 (C2), 62.8 (C6), 63.2 (C7), 63.7 (C1), 70.8 (C5), 71.0 (C3), 77.7 (C4); *m/z* (ESI^+): 194 ($[M+H]^+$, 80%), 216 ($[M+Na]^+$, 100%).

2,6-Dideoxy-2,6-imino-D-*glycero*-D-*gulo*-heptitol (35) and 2,6-dideoxy-2,6-imino-L-*glycero*-D-*gulo*-heptitol (36)

35





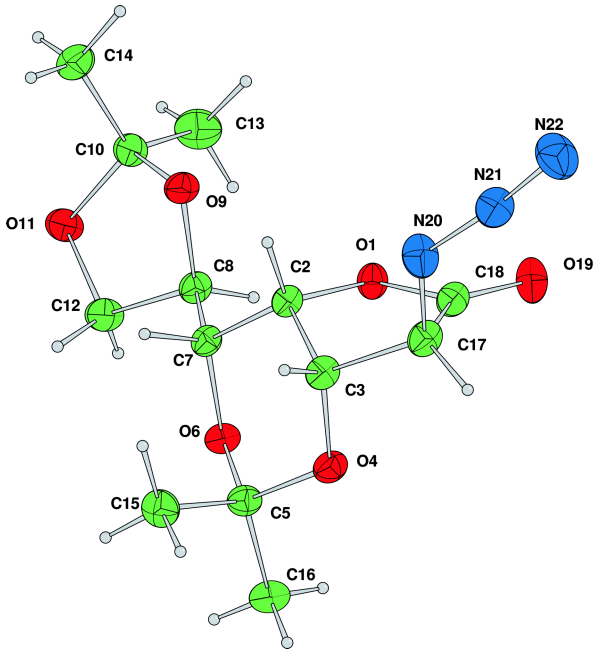
36

Azido-ketose **28** was hydrogenated in water with a nickel catalyst under similar conditions to those described by Izumori and co-workers[1] yielding a 11:1 mixture of **35** / **36**. The mixture was separated affording **35** (20 mg), as a colourless oil.

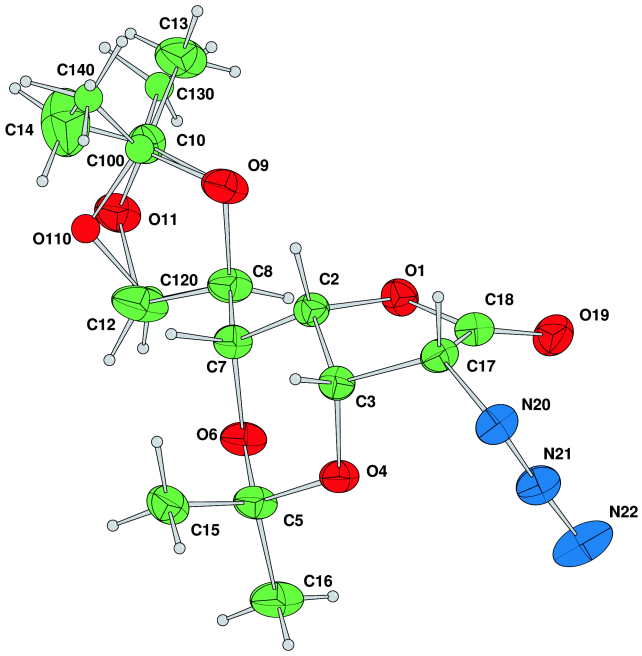
Data for **35**:

HRMS *m/z* (ESI^+): found 216.0841 $[M+Na]^+$; $C_7H_{15}NNaO_5$ requires 216.0842; $[\alpha]_D^{25}$ -0.84 (*c*, 0.36 in H_2O) {Lit.[5] ($[\alpha]_D$ -1.7 (*c*, 0.35 in H_2O)}; ν_{max} (thin film, Ge): 3318 (br, s, OH, NH); δ_H (D_2O, 400 MHz): 2.64 (2H, ddd, H2, H6, $J_{2,3}/J_{6,5}$ 9.7, $J_{2,1}/J_{6,7}$ 6.7, $J_{2,1'}/J_{6,7'}$ 2.9), 3.24 (2H, t, H3, H5, $J_{3,2}/J_{5,6} = J_{3,4}/J_{5,4}$ = 9.5), 3.38 (1H, t, H4, $J_{4,3}/J_{4,5}$ 9.1), 3.62 (2H, dd, H1, H7, $J_{1,1'}/J_{7,7'}$ 11.6, $J_{1,2}/J_{7,6}$ 6.7), 3.88 (2H, dd, H1', H7', $J_{1',1}/J_{7',7}$ 11.6, $J_{1',2}/J_{7',6}$ 2.9); δ_C (D_2O, 100.6 MHz): 60.3 (C2, C6), 62.0 (C1, C7), 72.0 (C3, C5), 78.8 (C4); *m/z* (ESI^+): 194 ($[M+H]^+$, 68%), 216 ($[M+Na]^+$, 100%).

X-ray crystallography on epimeric azido-lactones:



ORTEP plot of 2-azido-2-deoxy-3,5:6,7-di-*O*-isopropylidine-D-*glycero*-D-*ido*-1,4-heptono-lactone **3**. Anisotropic displacement ellipsoids are represented at the 50% probability level.



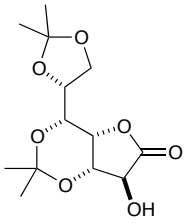
ORTEP plot of 2-azido-2-deoxy-3,5:6,7-di-*O*-isopropylidine-D-*glycero*-D-*gulo*-1,4-heptono-lactone **21**. Anisotropic displacement ellipsoids are represented at the 50% probability level.

Empirical formula	$C_{13}H_{18}N_3O_6$
Formula weight	313.31
Temperature (K)	150
Radiation and wavelength	Cu-*K*α, λ=1.54184 Å
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Unit cell dimensions	
a (Å)	7.36955 (8)
b (Å)	8.40431 (11)
c (Å)	24.1914 (3)
Volume (Å^3)	1498.32 (3)
Z	4
Density (calculated) (Mg/m^3)	1.389
Absorption coefficient, μ (mm^{-1})	0.94
F(000)	664
Crystal colour	Colourless
Crystal description	Needle
Crystal size (mm)	0.19 x 0.14 x 0.06
Absorption correction	Multi-scan[5]
Max. and min transmission	0.95 and 0.70
Theta range for data collection	3.7° ≤ θ ≤ 75.7°
Index ranges	-9≤*h*≤9; -10≤*k*≤10; -29≤*l*≤30
Reflections collected	15552
Completeness	0.99725
Independent reflections	3093 [R_{int}=0.024]
Reflections $I > 2\sigma(I)$	3017
Refinement method	Full-matrix least squares on F^2
Data/restraints/parameters	3092/0/200
Goodness-of-fit on F^2	1.0121
Final *R* indices [$I > 2\sigma(I)$]	R_1= 0.0240, wR_2= 0.0618
R indices (all data)	R_1= 0.0246, wR_2= 0.0623
Extinction coefficient	none
Max. and mean shift/esd	0.0009122 and 0.0000612
Largest diff. peak and hole (e Å^{-3})	0.23 and -0.18
Absolute structure parameter	-0.02 (11)[6]
CCDC number	917203

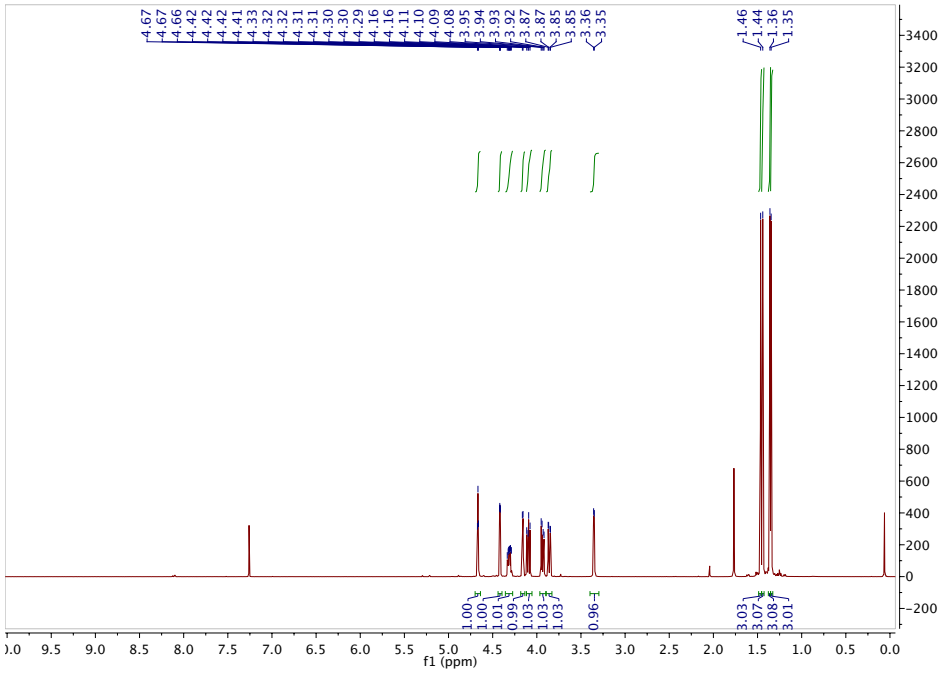
Empirical formula	$C_{13}H_{18}N_3O_6$
Formula weight	313.31
Temperature (K)	150
Radiation and wavelength	Mo-*K*α, λ=0.71073 Å
Crystal system	Orthorhombic
Space group	$P22_12_1$
Unit cell dimensions	
a (Å)	6.17130 (10)
b (Å)	12.5187 (3)
c (Å)	19.7716 (5)
Volume (Å^3)	1527.49 (6)
Z	4
Density (calculated) (Mg/m^3)	1.358
Absorption coefficient, μ (mm^{-1})	0.11
F(000)	660
Crystal colour	colourless
Crystal description	lath
Crystal size (mm)	0.60 x 0.15 x 0.07
Absorption correction	Multi-scan[5]
Max. and min transmission	0.99 and 0.98
Theta range for data collection	5.3° ≤ θ ≤ 27.5°
Index ranges	-7≤*h*≤8; -16≤*k*≤16; -24≤*l*≤25
Reflections collected	3392
Completeness	0.98622
Independent reflections	2005 [R_{int}=0.030]
Reflections $I > 2\sigma(I)$	1562
Refinement method	Full-matrix least squares on F^2
Data/restraints/parameters	1992/378/216
Goodness-of-fit on F^2	0.9854
Final *R* indices [$I > 2\sigma(I)$]	R_1= 0.0395, wR_2= 0.0853
R indices (all data)	R_1= 0.0579, wR_2= 0.1036
Extinction coefficient	15.448[7]
Max. and mean shift/esd	0.0005570 and 0.0000484
Largest diff. peak and hole (e Å^{-3})	0.31 and -0.39
CCDC number	917202

NMR spectra of synthetic compounds:

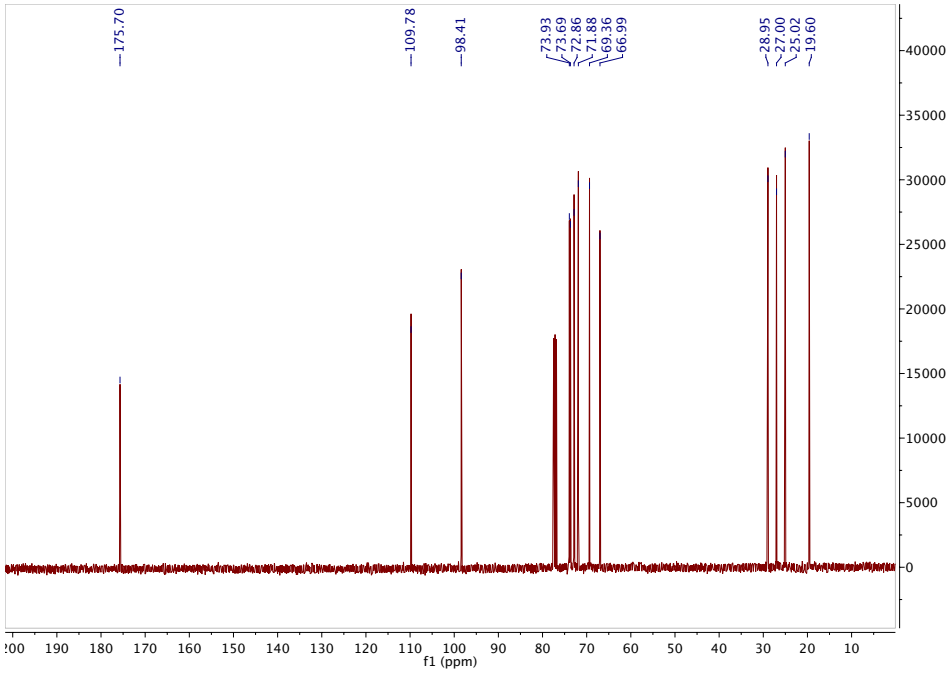
3,5:6,7-Di-*O*-isopropylidene-D-*glycero*-D-*ido*-heptono-1,4-lactone 20



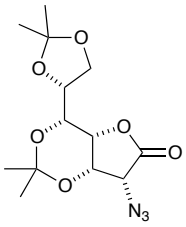
Proton spectrum, 400 MHz, $CDCl_3$



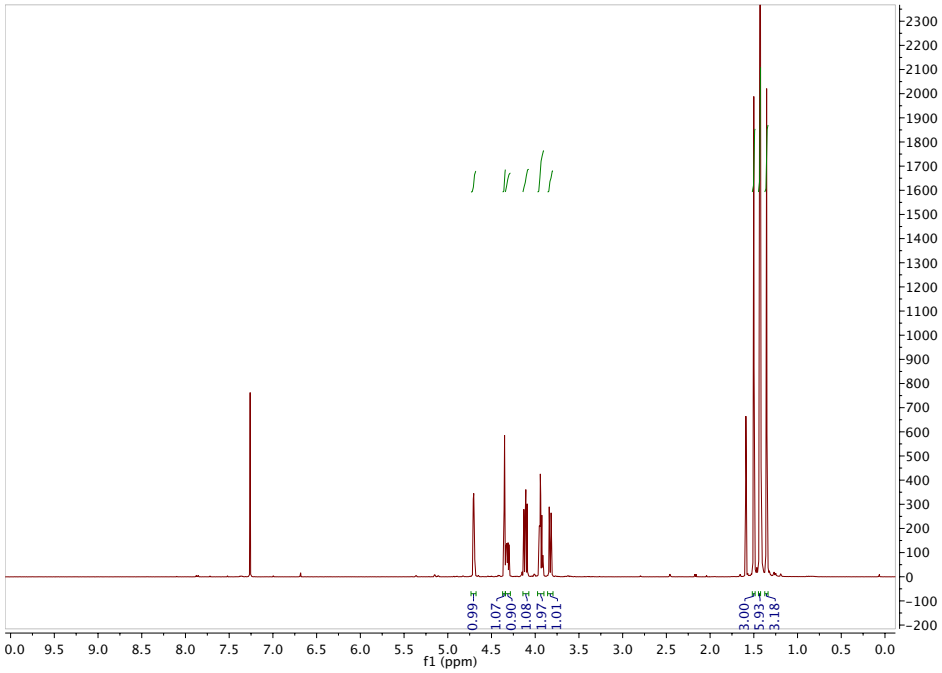
Carbon spectrum, 100 MHz, $CDCl_3$



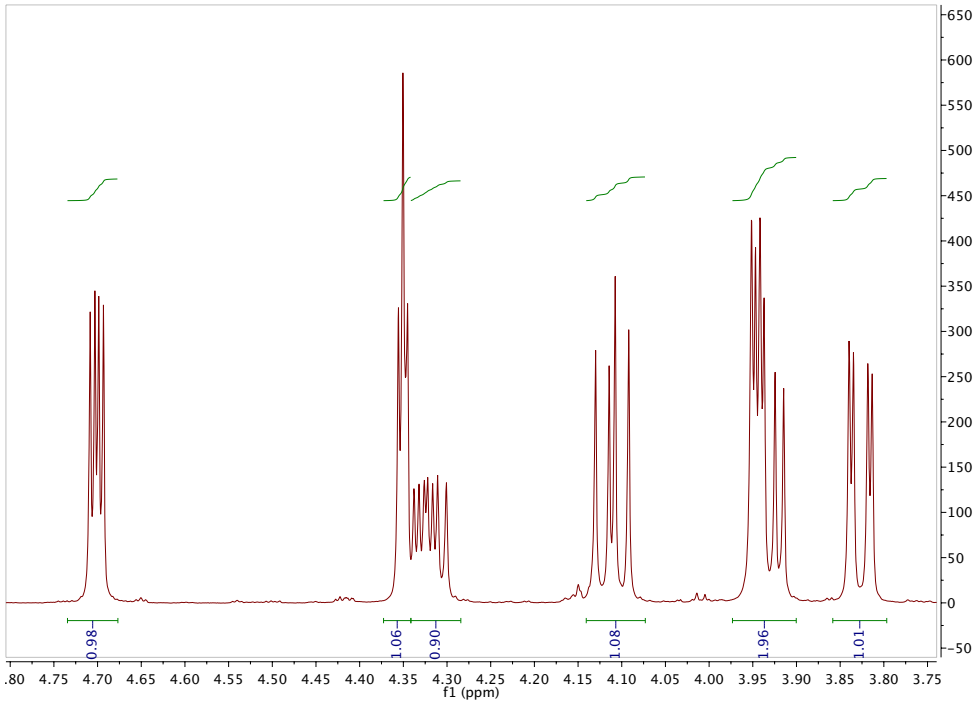
2-Azido-2-deoxy-3,5:6,7-di-*O*-isopropylidene-D-*glycero*-D-*gulo*-heptono-1,4-lactone 21



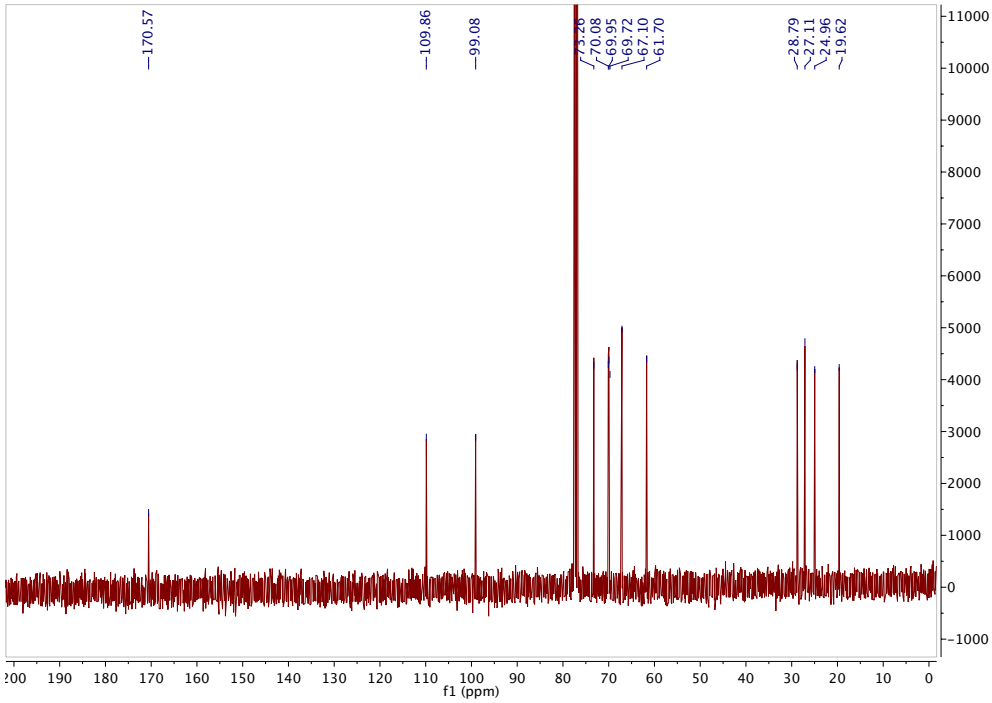
Proton spectrum, 400 MHz, $CDCl_3$



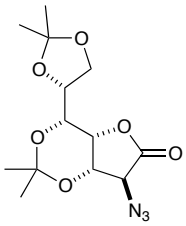
Proton spectrum zoom carbohydrate region, 400 MHz, $CDCl_3$



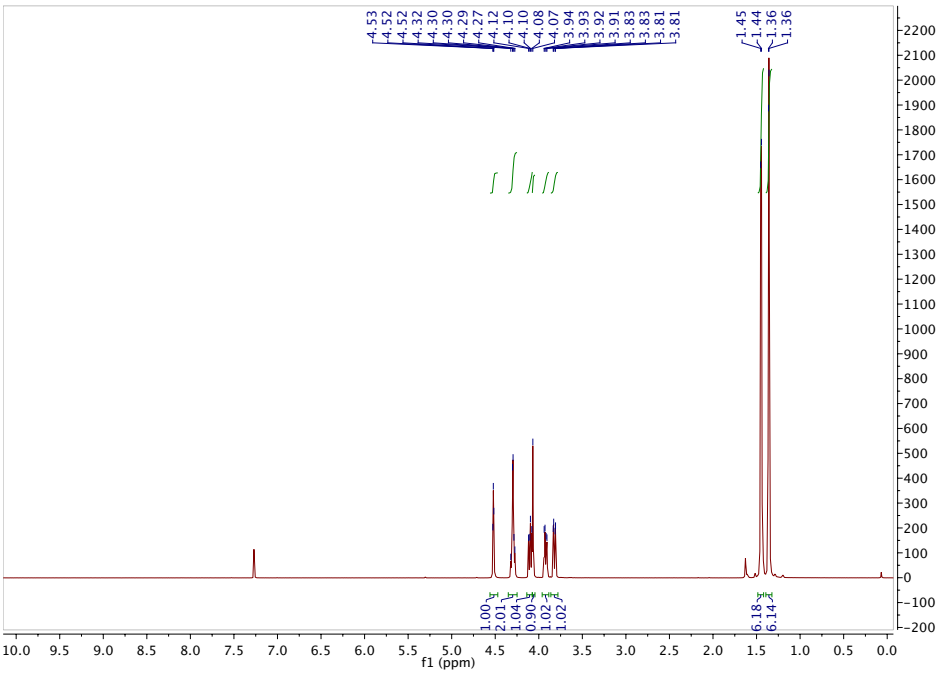
Carbon spectrum, 100 MHz, $CDCl_3$



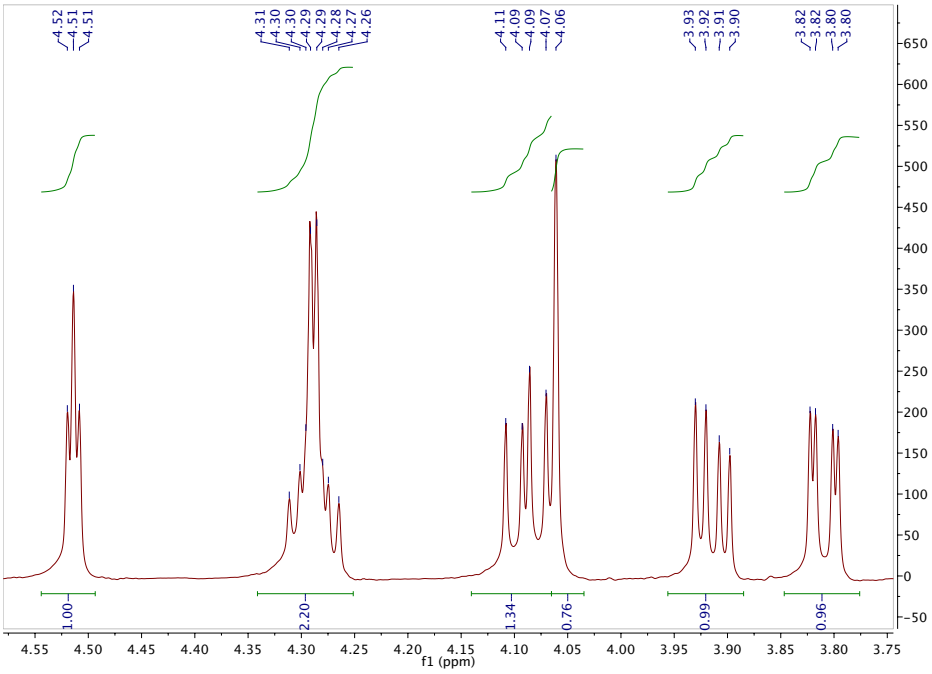
2-Azido-2-deoxy-3,5:6,7-di-*O*-isopropylidene-D-*glycero*-D-*ido*-heptono-1,4-lactone 3



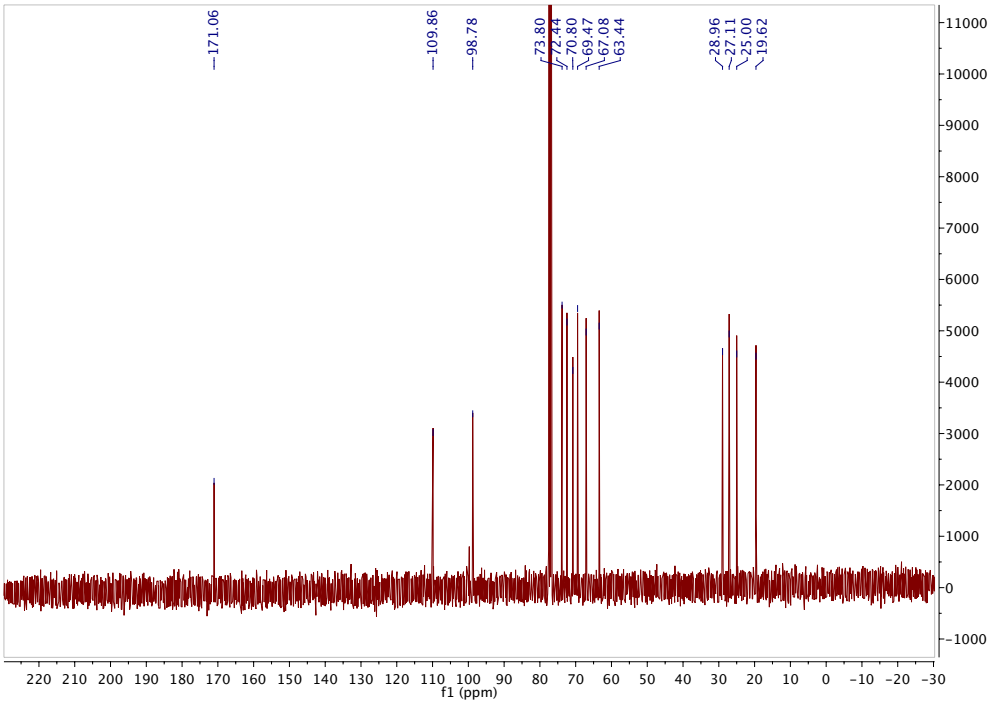
Proton spectrum, 400 MHz, $CDCl_3$



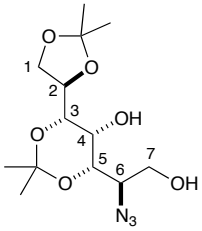
Proton spectrum zoom carbohydrate region, 400 MHz, $CDCl_3$



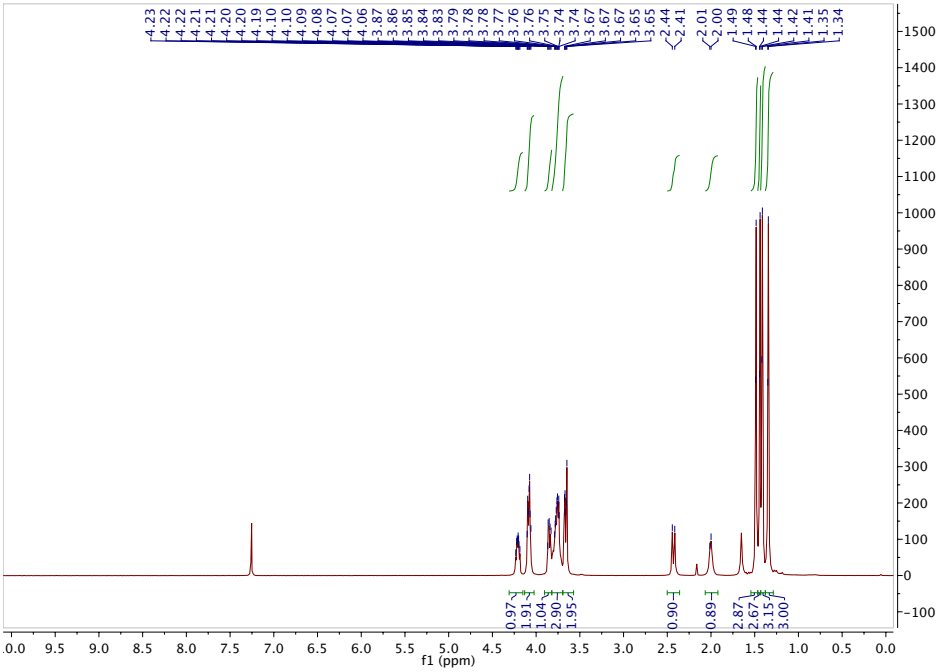
Carbon spectrum, 100 MHz, $CDCl_3$



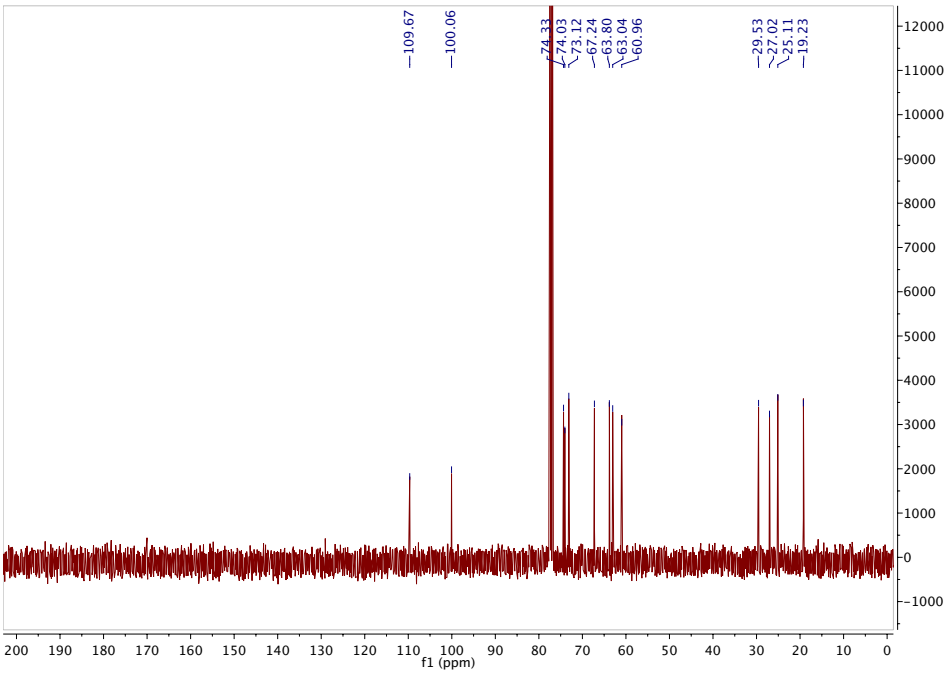
6-Azido-6-deoxy-1,2:3,5-di-*O*-isopropylidene-D-*glycero*-L-*gulo*-heptitol 22



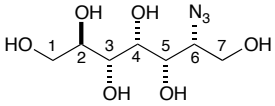
Proton spectrum, 400 MHz, $CDCl_3$



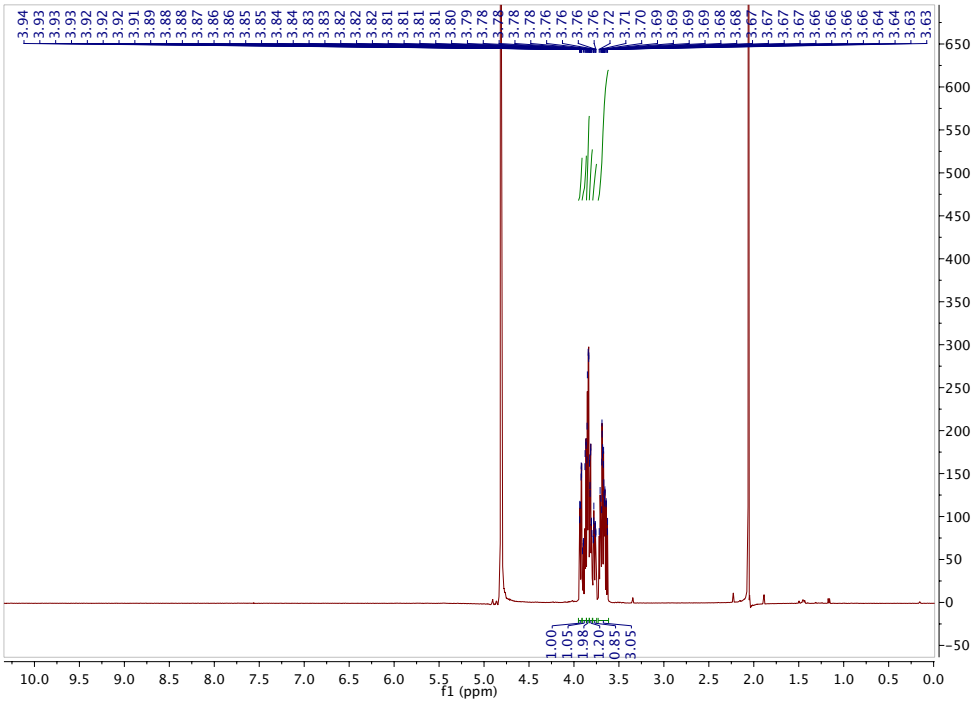
Carbon spectrum, 100 MHz, $CDCl_3$



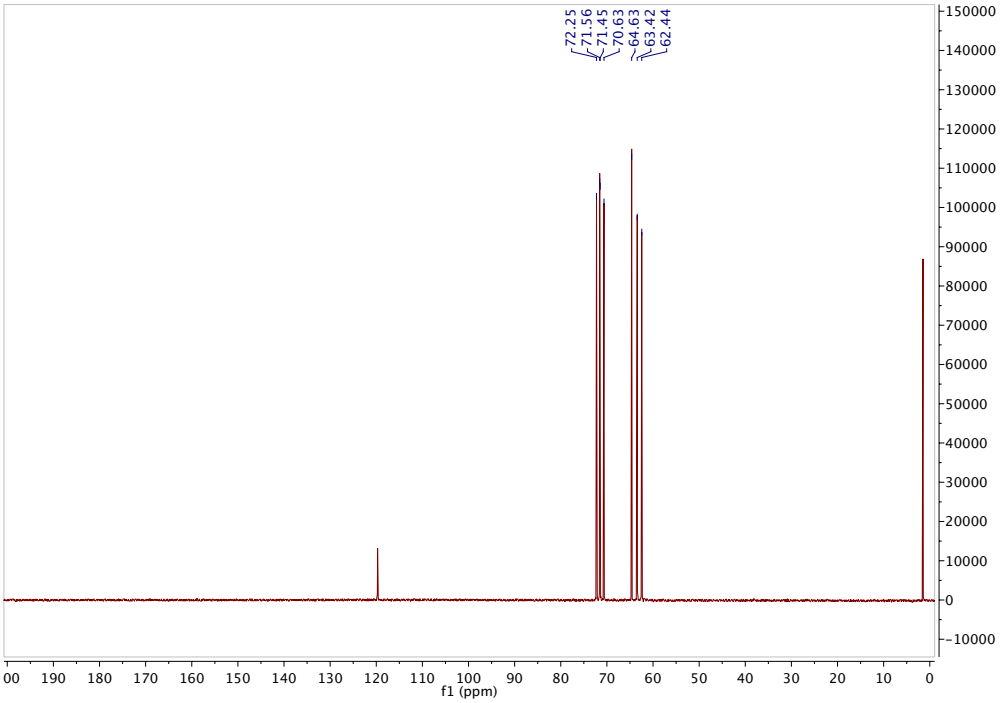
6-Azido-6-deoxy-D-*glycero*-L-*gulo*-heptitol 24



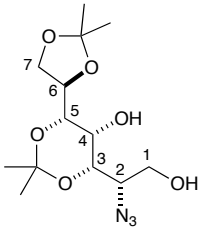
Proton spectrum, 400 MHz, D_2O



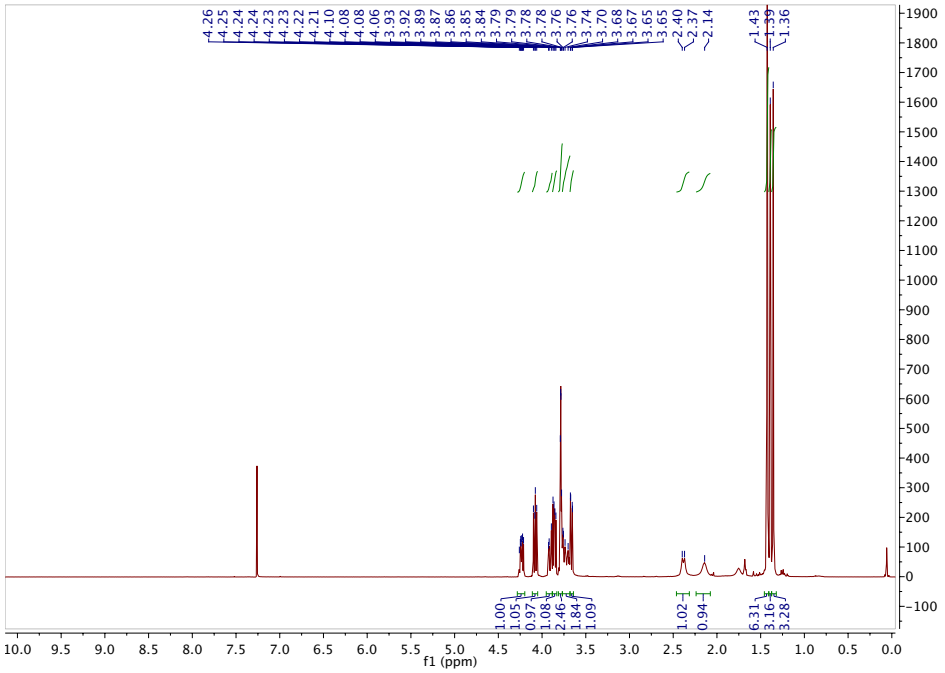
Carbon spectrum, 100 MHz, D_2O



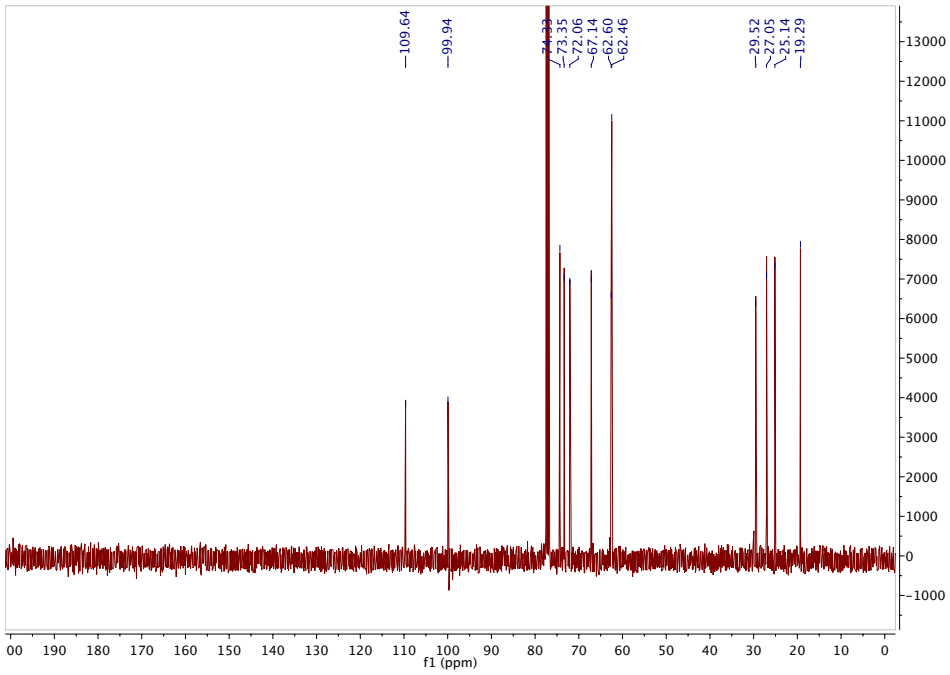
2-Azido-2-deoxy-3,5:6,7-di-*O*-isopropylidene-D-*glycero*-D-*gulo*-heptitol 23



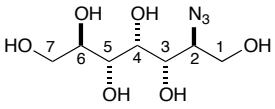
Proton spectrum, 400 MHz, $CDCl_3$



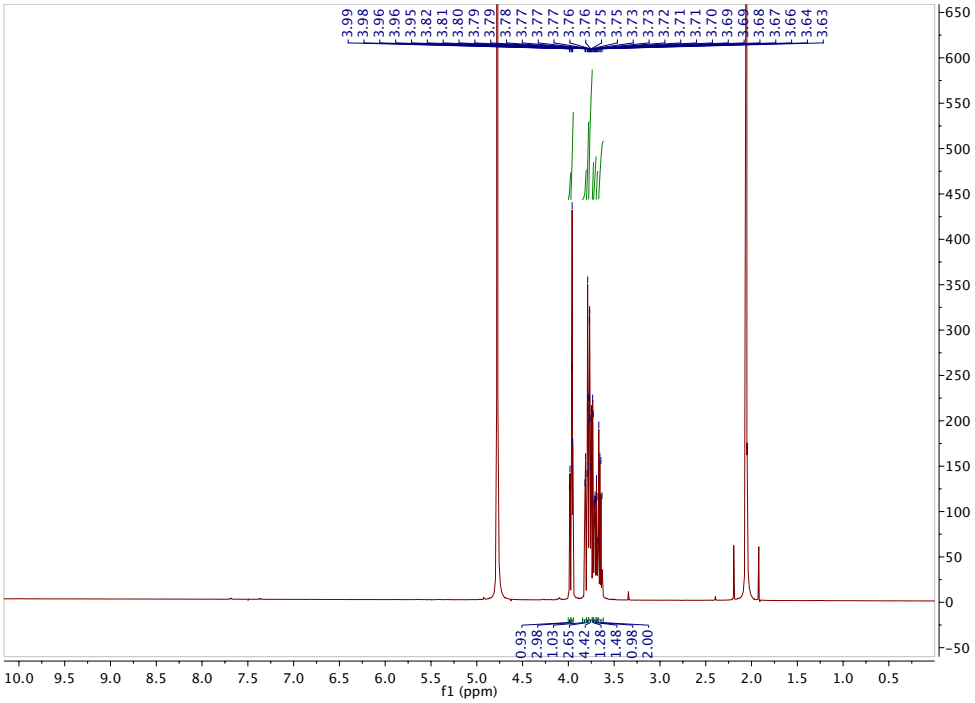
Carbon spectrum, 100 MHz, $CDCl_3$



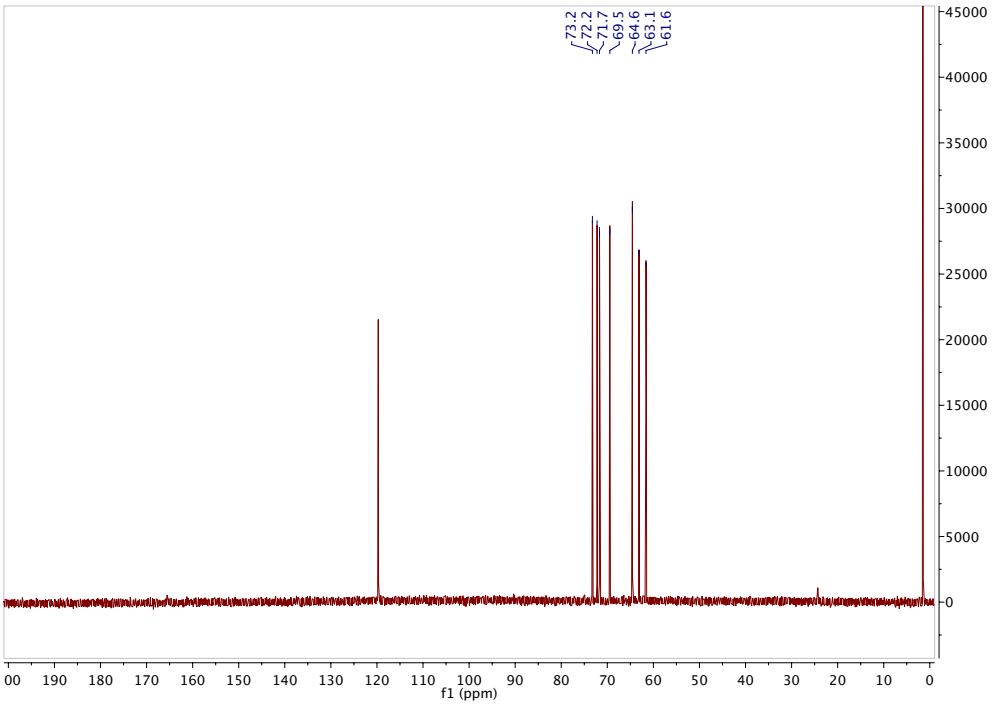
2-Azido-2-deoxy-D-*glycero*-D-*gulo*-heptitol 25



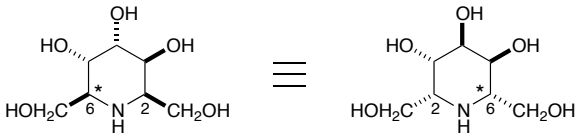
Proton spectrum, 500 MHz, D_2O



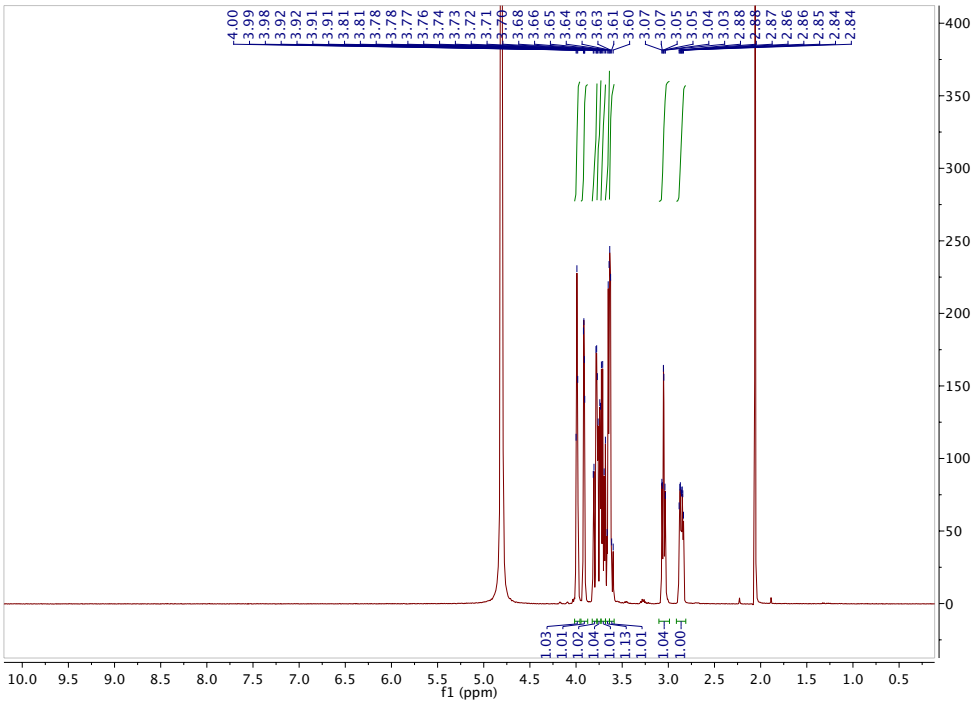
Carbon spectrum, 125 MHz, D_2O



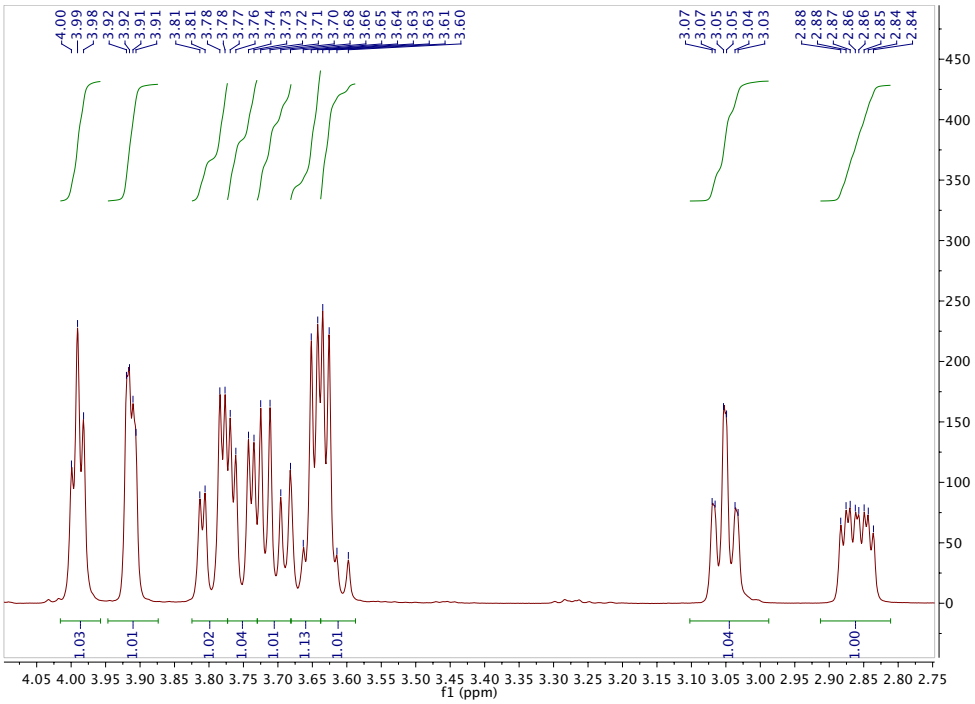
2,6-dideoxy-2,6-imino-L-*glycero*-L-*gluco*-heptitol (31)



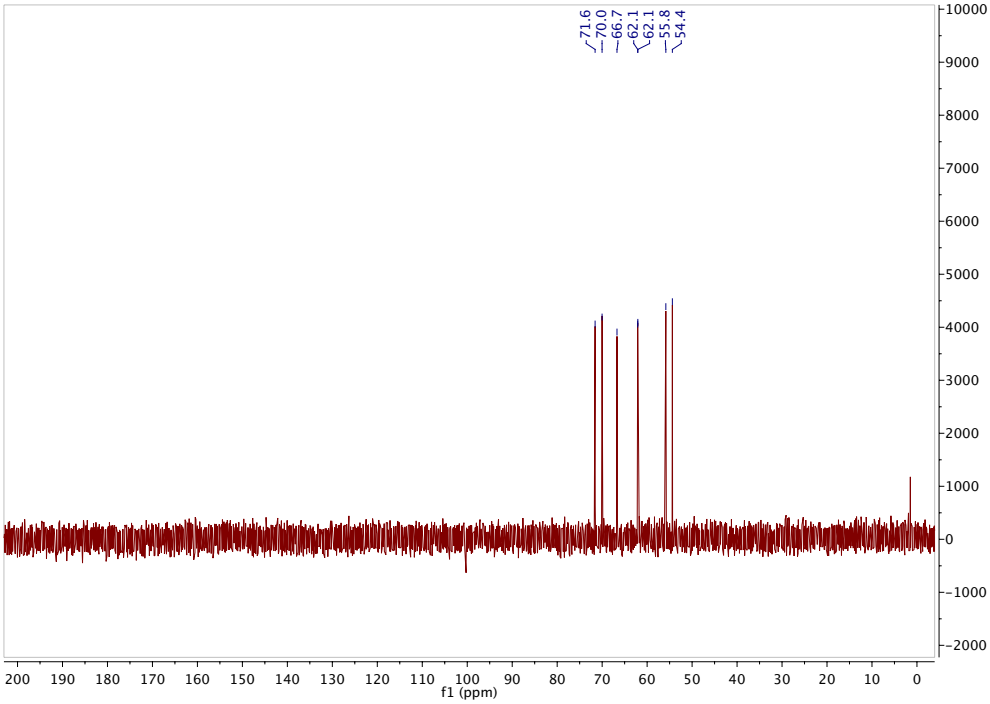
Proton spectrum, 400 MHz, D_2O



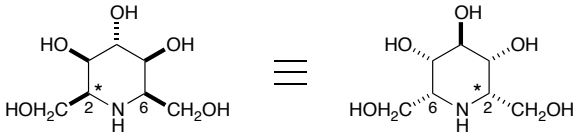
Proton spectra zoom carbohydrate region, 400 MHz, D_2O



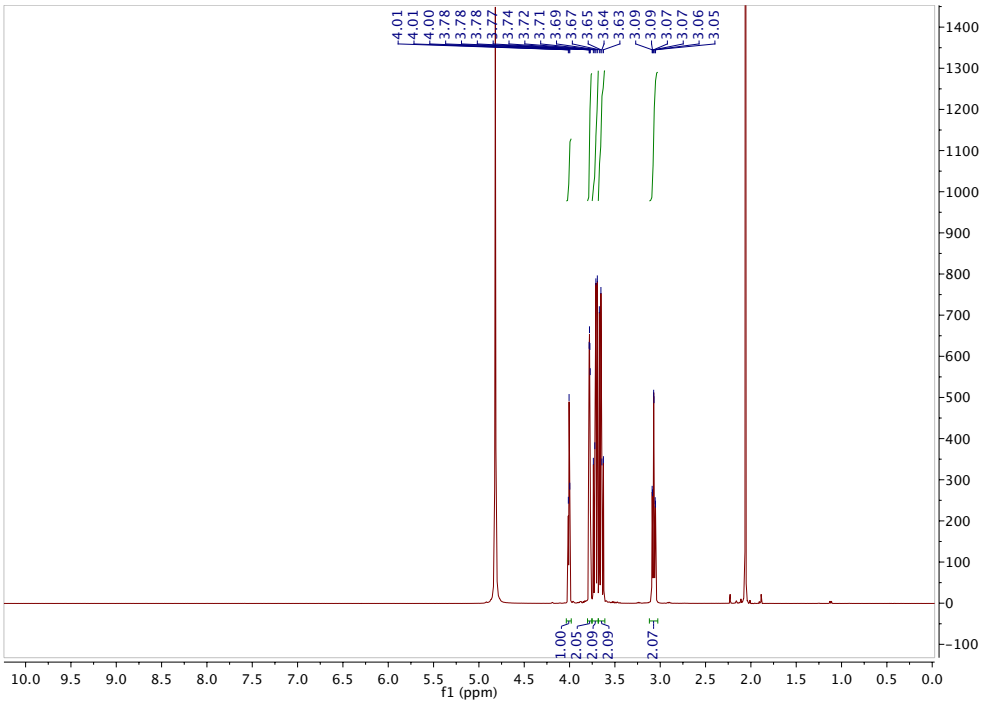
Carbon spectrum, 100 MHz, D_2O



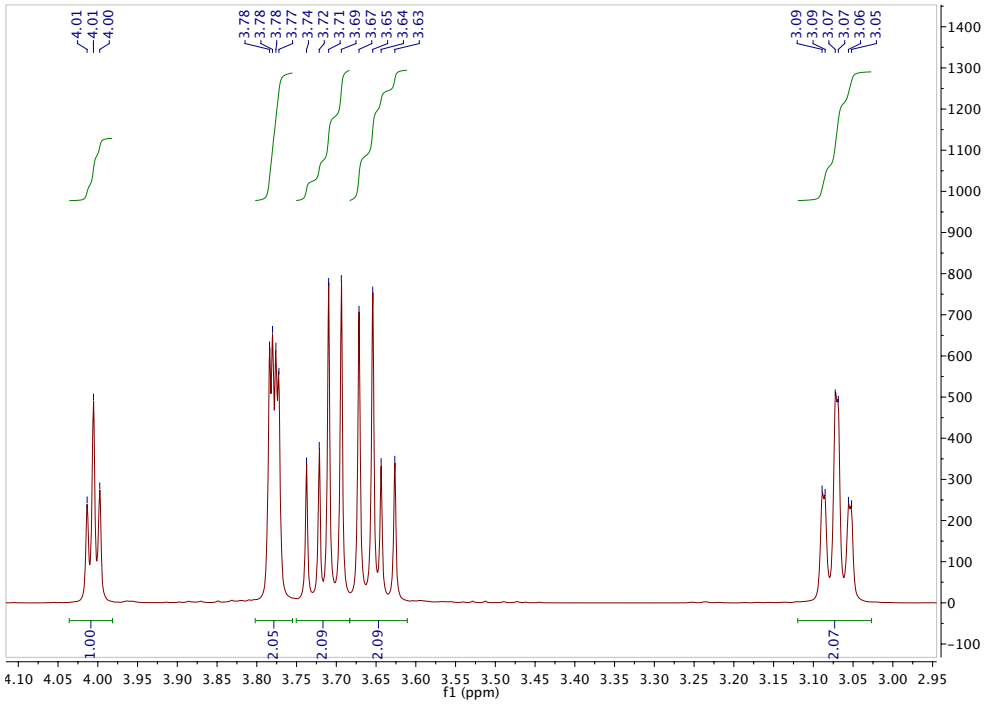
2,6-Dideoxy-2,6-imino-D-*glycero*-L-*ido*-heptitol (32)



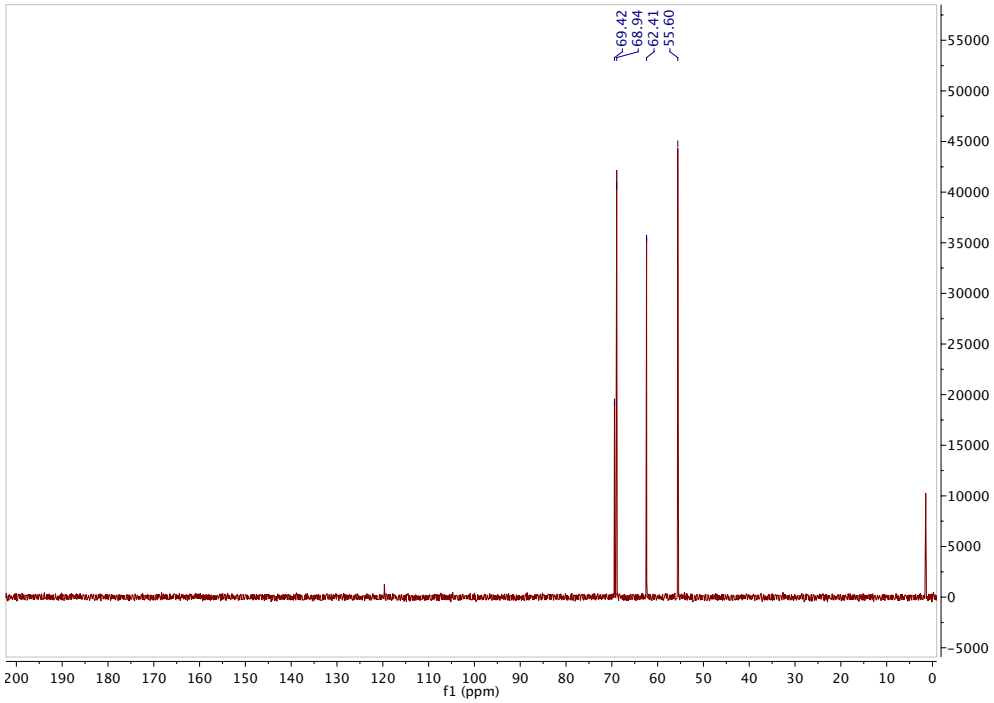
Proton spectrum, 400 MHz, D_2O



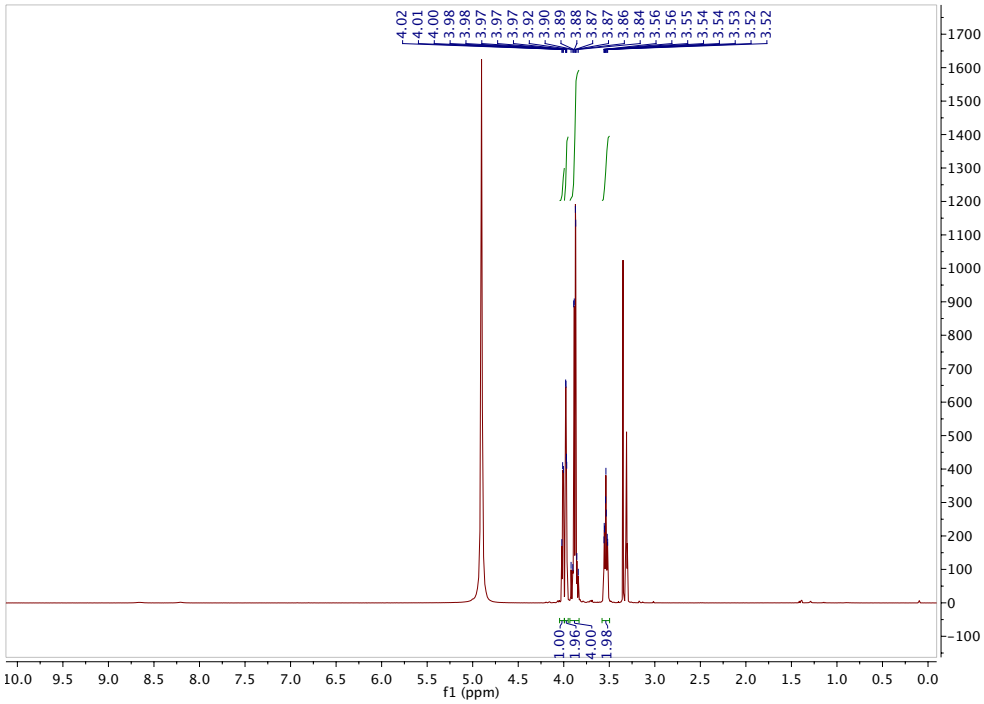
Proton spectrum zoom carbohydrate region, 400 MHz, D_2O



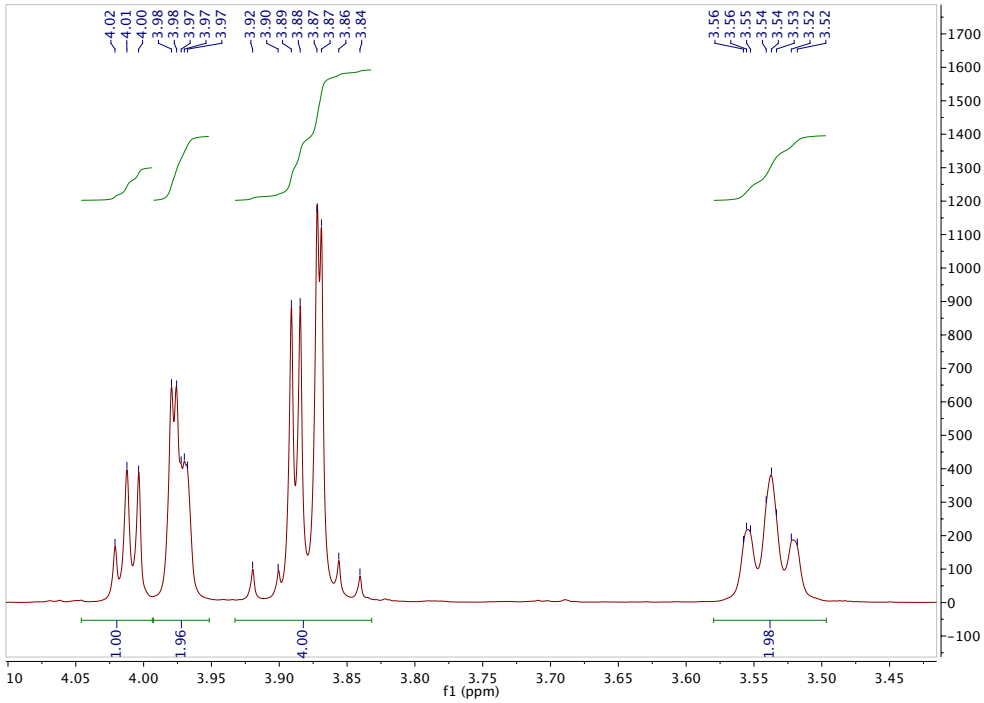
Carbon spectrum, 100 MHz, D_2O



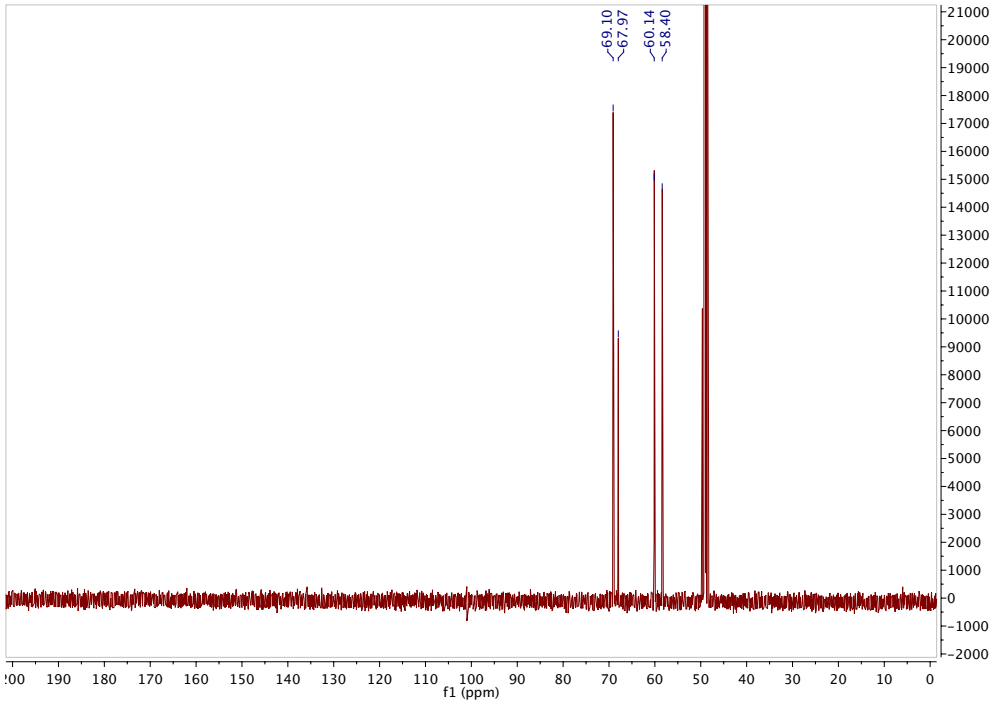
Proton spectrum, 400 MHz, CD_3OD



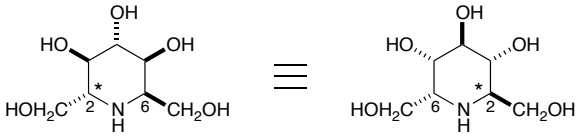
Proton spectrum zoom carbohydrate region, 400 MHz, CD_3OD



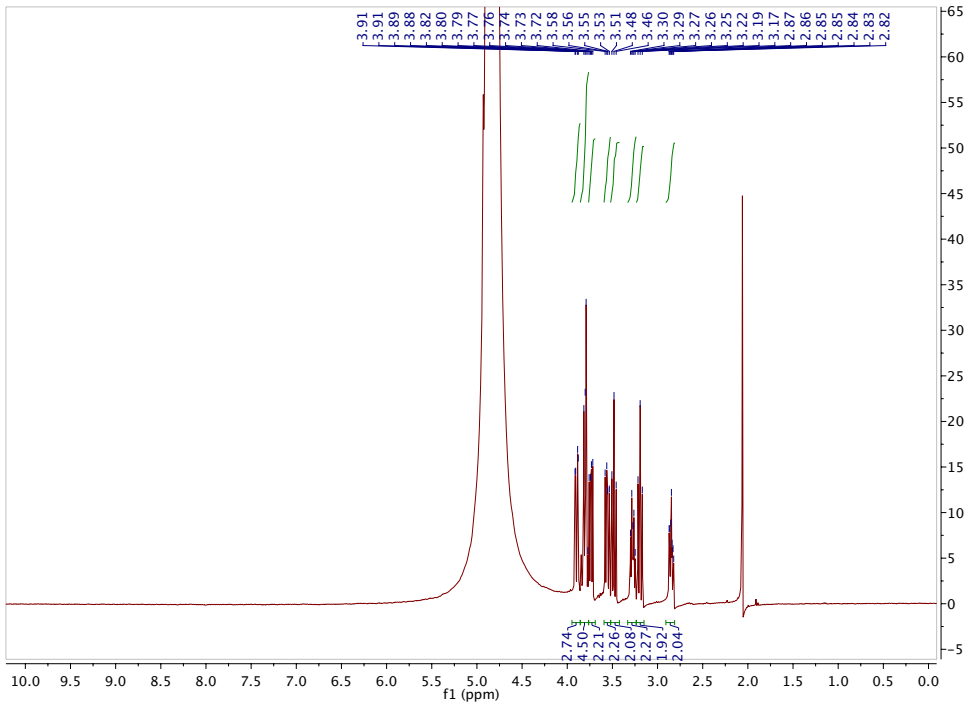
Carbon spectrum, 100 MHz, CD_3OD



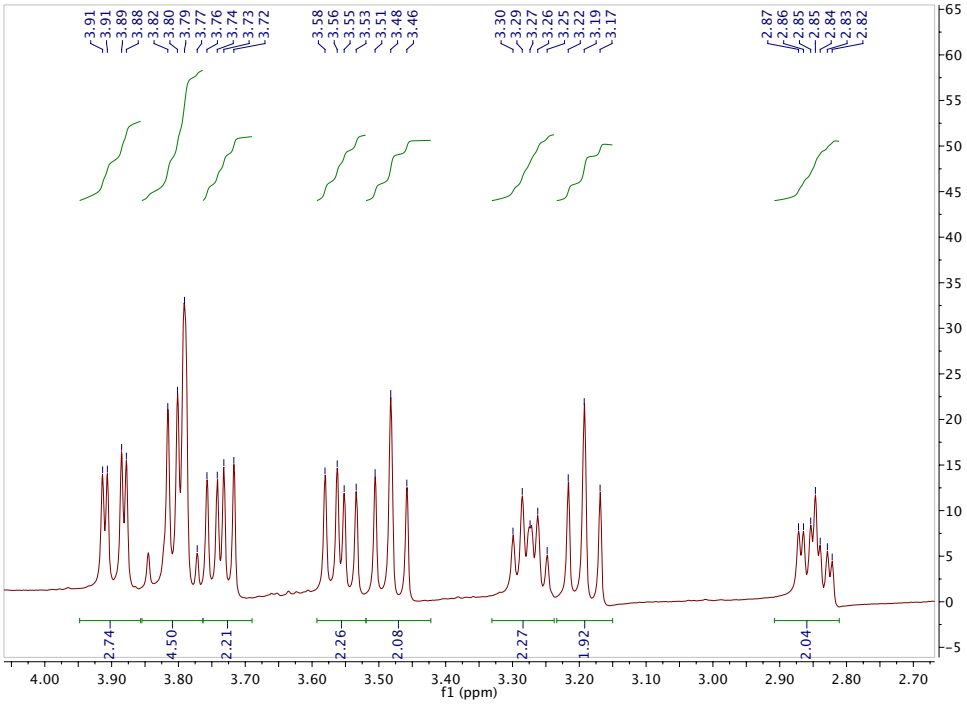
2,6-dideoxy-2,6-imino-D-*glycero*-L-*gulo*-heptitol (α-homonojirimycin) (4)



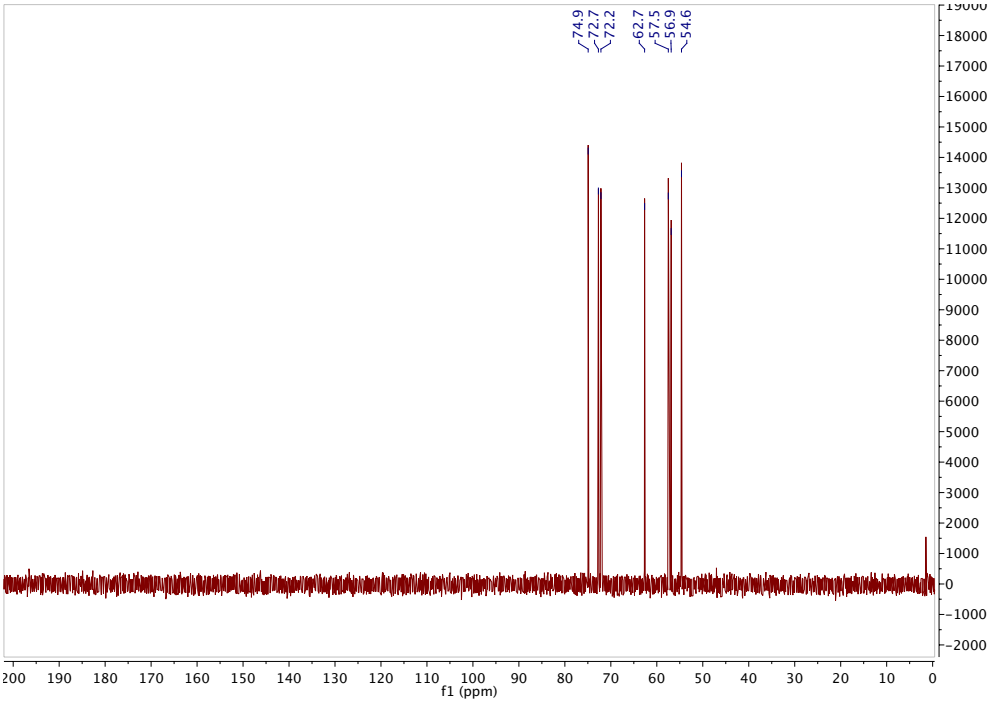
Proton spectrum, 400 MHz, D_2O



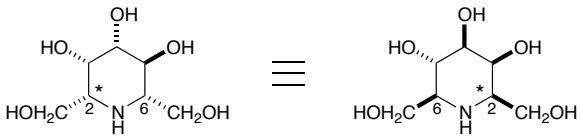
Proton spectrum zoom carbohydrate region, 400 MHz, D_2O



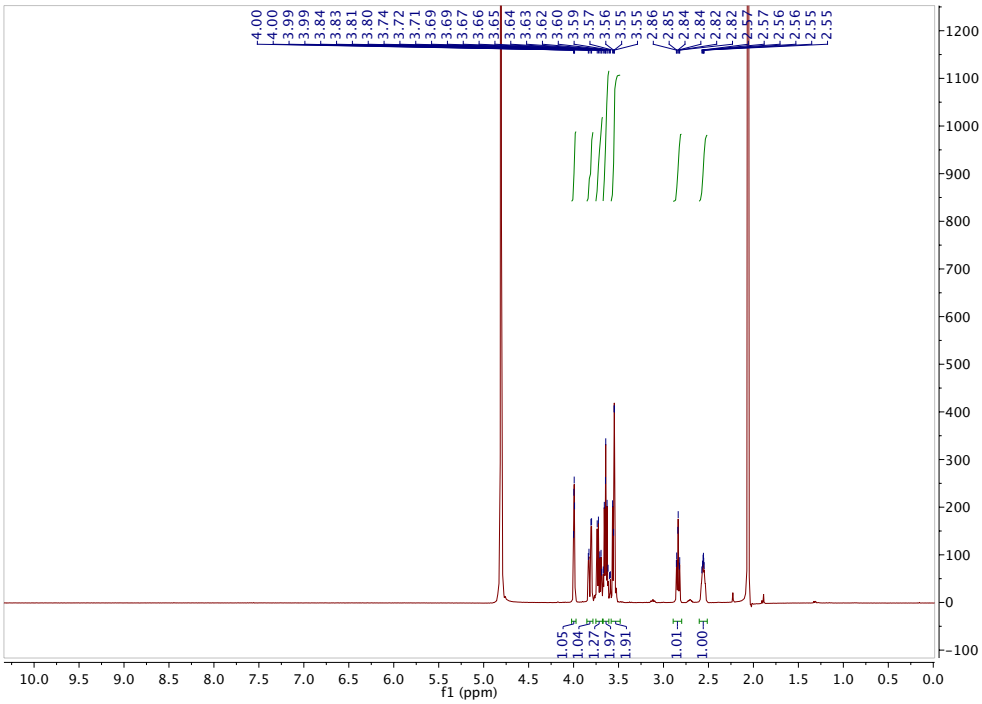
Carbon spectrum, 100 MHz, D_2O



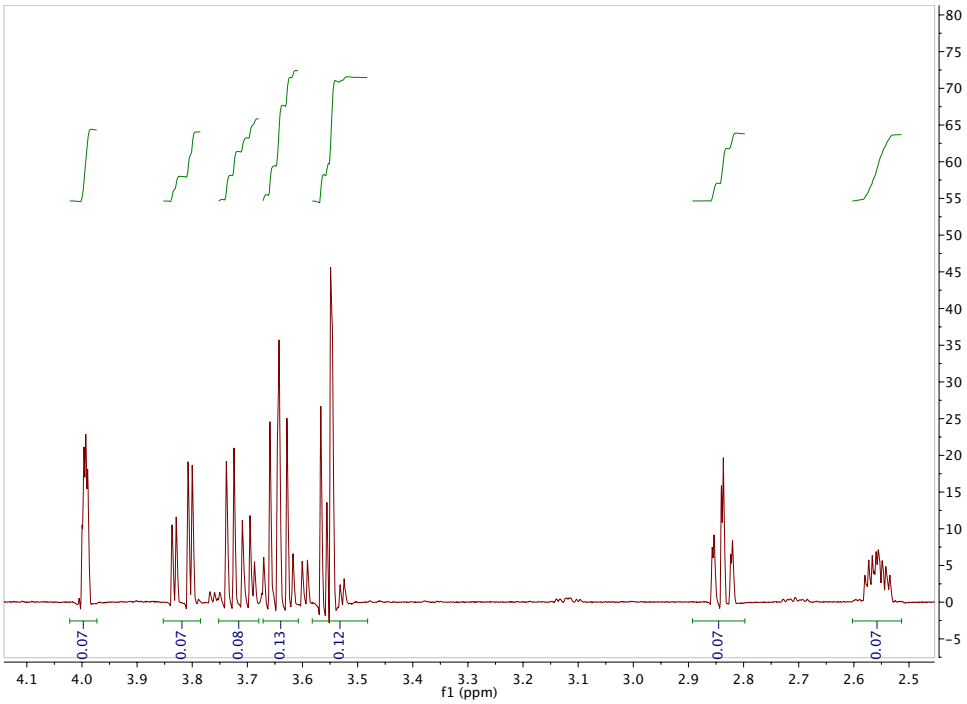
2,6-dideoxy-2,6-imino-L-*glycero*-L-*galacto*-heptitol (34)



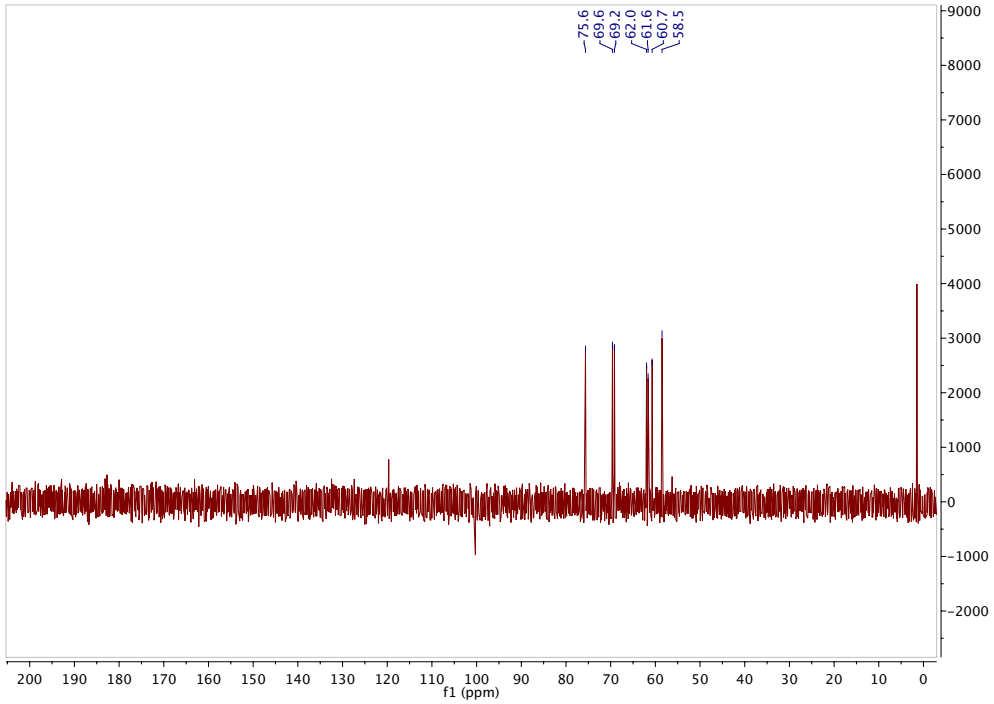
Proton spectrum, 400 MHz, D_2O



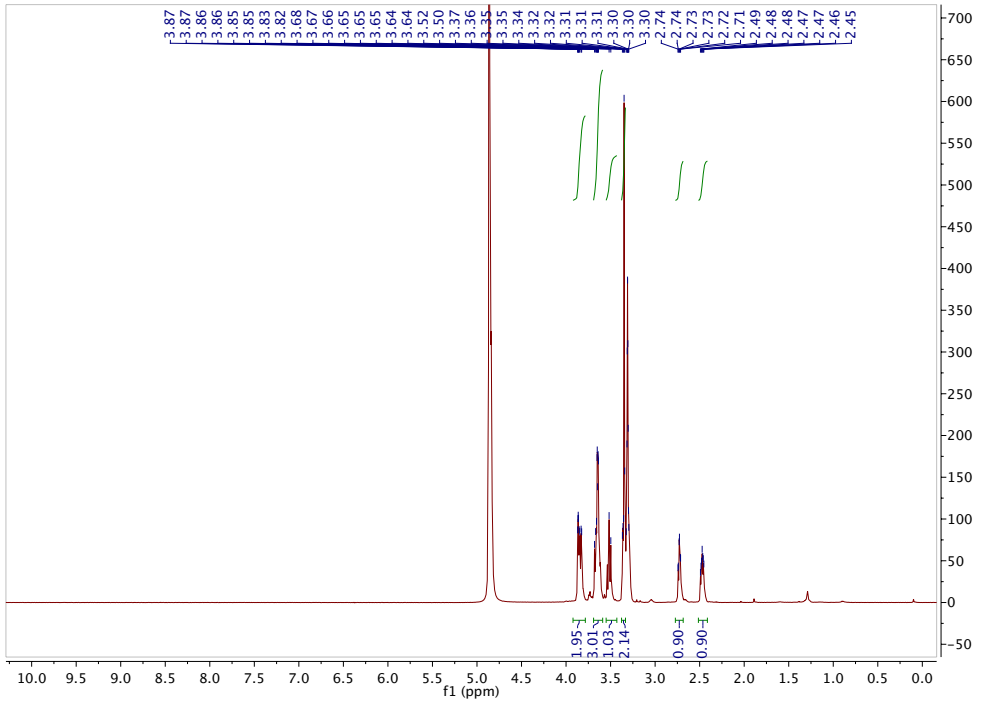
Proton spectrum zoom carbohydrate region, 400 MHz, D_2O



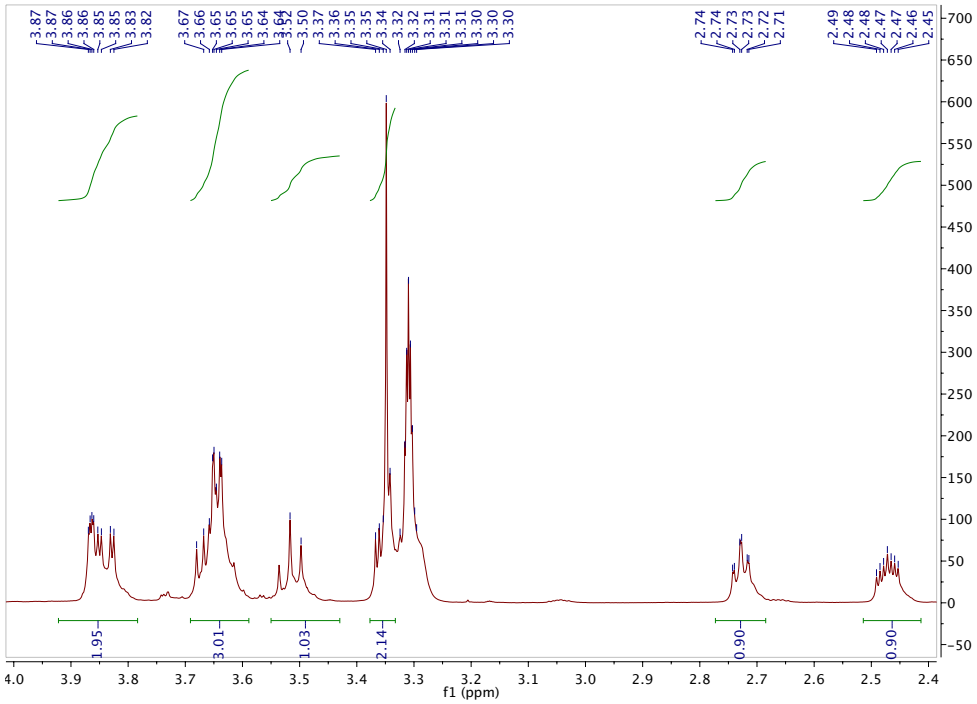
Carbon spectrum, 100 MHz, D_2O



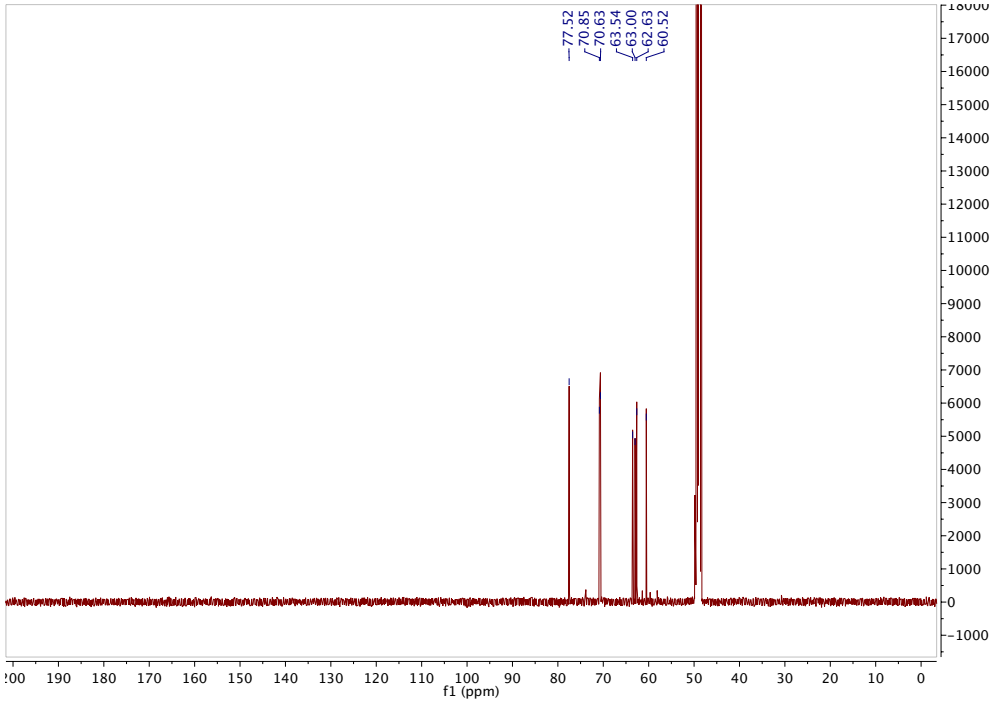
Proton spectrum, 500 MHz, CD_3OD



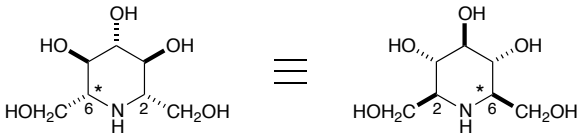
Proton spectrum zoom carbohydrate region, 500 MHz, CD_3OD



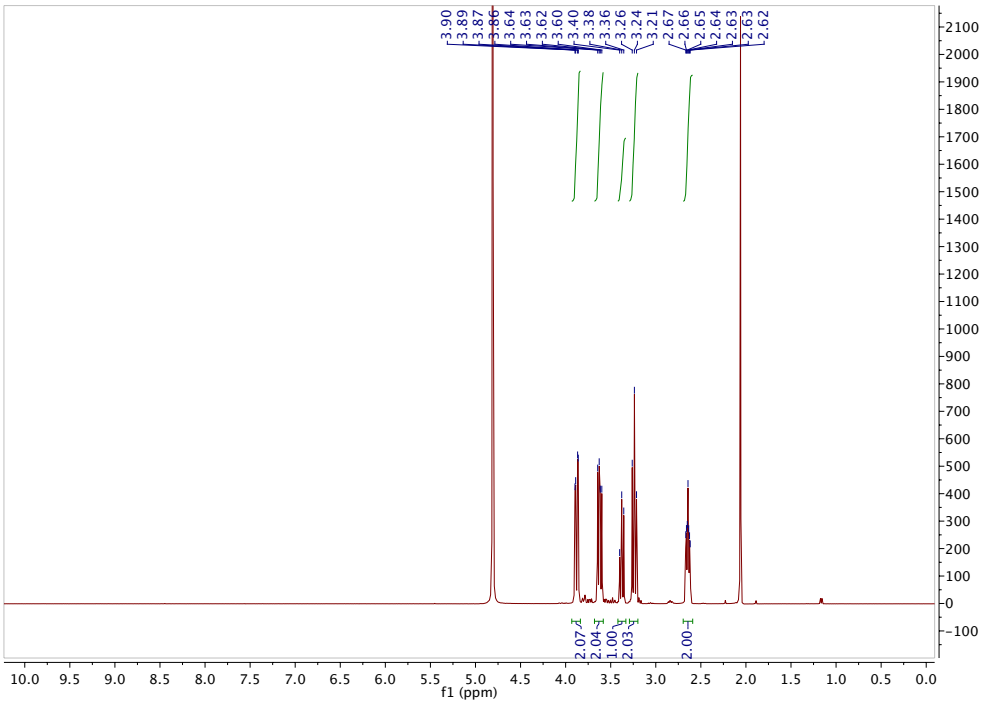
Carbon spectrum, 125 MHz, CD_3OD



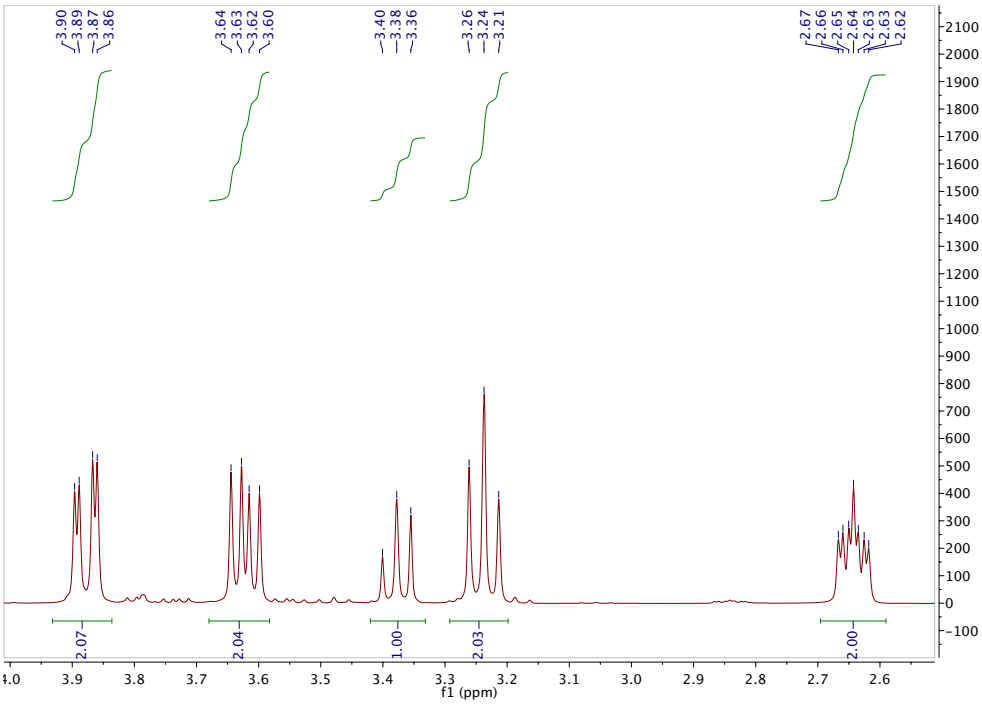
2,6-Dideoxy-2,6-imino-D-*glycero*-D-*gulo*-heptitol (35)



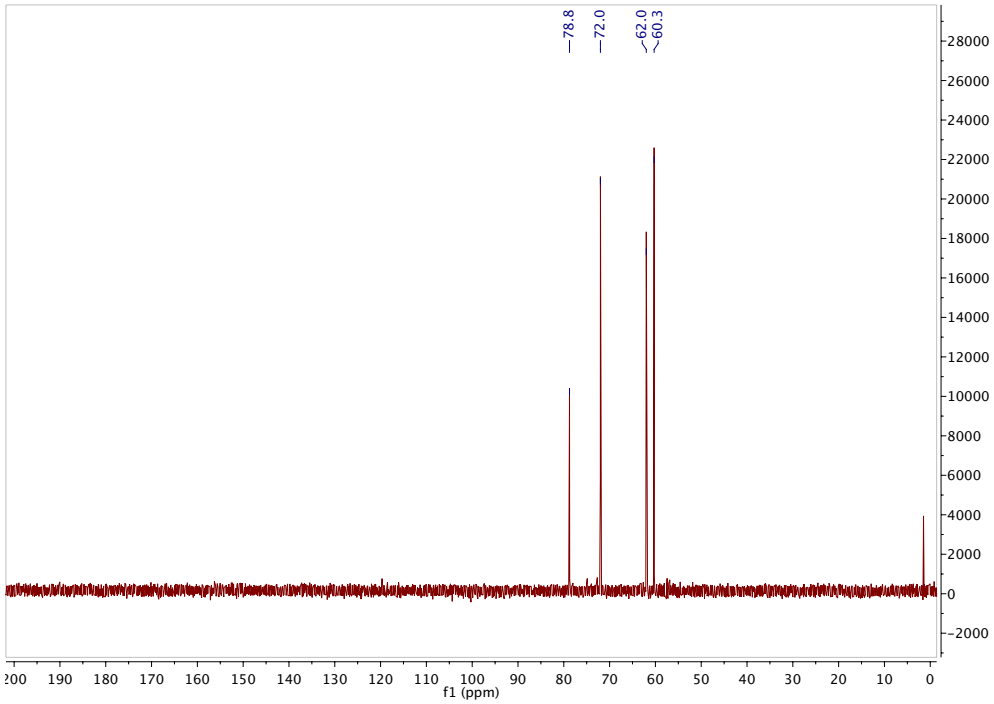
Proton spectrum, 400 MHz, D_2O



Proton spectrum zoom carbohydrate region, 400 MHz, D_2O



Carbon spectrum, 100 MHz, D_2O



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