Supporting Information

Highly Efficient Synthesis of Ketoheptoses

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General Remarks:

TLC was performed on aluminium sheets coated with silica gel 60 (Merck) and detection was by UV and heating with H_2SO_4. Column chromatography was carried out on silica gel 60 (40–63 µm; Merck) and silica gel 60 RP-18 (40–63 µm; Merck). NMR spectra were recorded on a Bruker AMX 400, AC 400, AV 2400 (400 MHz for ^{1}H and 100.67 MHz for ^{13}C NMR) or a Bruker DRX 500 (500 MHz for ^{1}H and 125.84 MHz for ^{13}C NMR). Chemical shifts are in ppm, related to the corresponding solvent peaks.[1] Mass spectra were recorded on a Bruker Biflex II (MALDI-TOF, positive reflection mode, matrix: 2,5-dihydroxybenzoic acid) and a *Thermo Finnigan* MAT 95 XL or *Agilent*-6224-TOF ESI/MS (ESI mass spectra). Melting points were determined with an Apotec melting point apparatus and are uncorrected. The optical rotations were measured with a Kruess P8000 polarimeter (589 nm, Na).

General Procedures:

Acetylation/Glycosylation, General Procedure gp1: The unprotected monosaccharide (1.0 mmol) was suspended in acetic anhydride (2.0 mmol/unprotected hydroxyl group). At 0 °C several drops of perchloric acid (69%, aqueous) were added. The reaction mixture was stirred for another 60 minutes at 0 °C. Afterwards the reaction solution was diluted with dichloromethane, washed with water and saturated sodium bicarbonate solution. The combined organic phases were dried over sodium sulphate and concentrated in vacuo. The crude product was used for the subsequent glycosylation without further purification.

Under an argon atmosphere the crude penta acetate (1.0 mmol) was dissolved in anhydrous dichloromethane (1.5 mL) and boron trifluoride diethyl etherate (1.2 mmol) was added. At 0 °C thiophenol (1.4 mmol) was added drop wise, and the reaction mixture was allowed to warm to room temperature. After completion saturated sodium bicarbonate solution was added until all boron trifluoride was hydrolysed. The organic phase was washed with water and saturated sodium bicarbonate solution several times. The combined organic phases were dried over sodium sulfate, concentrated in vacuo and purified by column chromatography (indicated conditions).

Deacetylation, General Procedure gp2:[2,3] The acetylated compound (1.0 mmol) was dissolved in anhydrous methanol (3 mL) and treated with a catalytic amount of a 0.5 M sodium methoxide solution. The reaction mixture was stirred at room temperature until completion. Then Amberlite® IR-120 H^+ was added for neutralization. The ion exchange was filtered and the solvent removed in vacuo. The crude product was used without further purification.

Benzylation, General Procedure gp3: The reactions were carried out under an argon atmosphere. The partially unprotected monosaccharide (1.0 mmol) was added in portions over a period of 30 minutes to a suspension of sodium hydride (1.5 mmol/unprotected hydroxyl group) in anhydrous DMF (7.5 mL) at 0 °C. The reaction mixture was stirred for another 2 hours at room temperature and a catalytic amount of TBAI (0.1 mmol) was added. Then benzylbromide (1.1 mmol/unprotected hydroxyl group) was added drop wise over a period of 45 minutes and the reaction mixture was stirred over night at room temperature. After completion ethanol was added until gas evolution ceased. The reaction mixture was diluted with water and ether, the phases separated and the aqueous phase extracted with ether several times. The combined organic phases were dried over sodium sulfate, concentrated in vacuo and purified by column chromatography (indicated conditions).

Cleavage of Thioglycosides, General Procedure gp4:[4] The thioglycoside (1.0 mmol) was dissolved in a mixture of acetone/water (9:1 v/v, 15 mL). Then NBS (3-4 mmol) was added at room temperature and the reaction mixture was stirred for the given time. After completion the reaction mixture was diluted with water, extracted with ether several times and the combined organic phases washed with saturated sodium bicarbonate solution and dried over sodium sulfate. After removal of the solvent, the crude product was purified by column chromatography (indicated conditions).

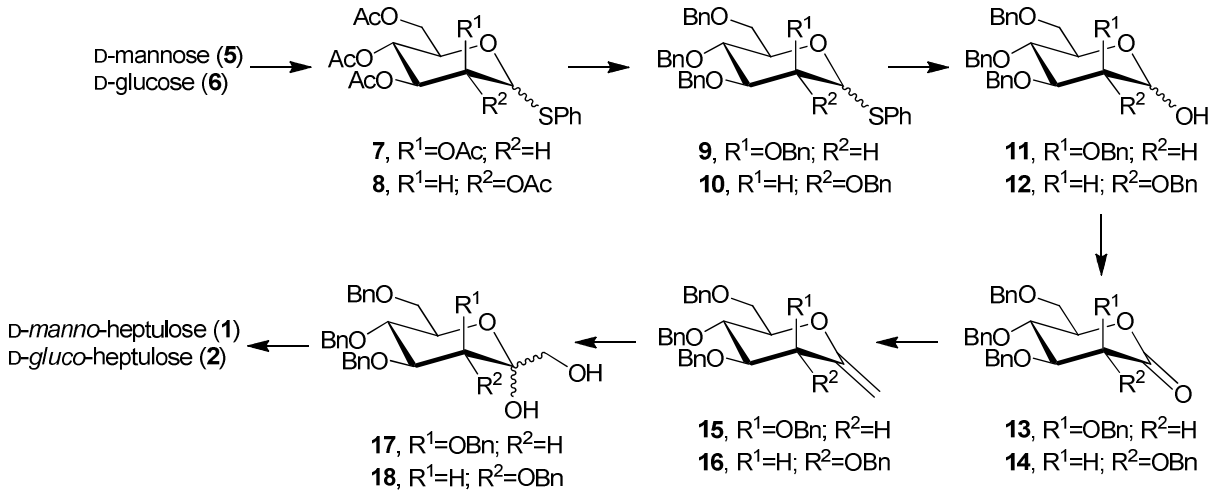
Oxidation, General Procedure gp5:[5] The hemiacetal (1.0 mmol) was dissolved in anhydrous DMSO (10 mL) and heated to 30 °C. Then acetic anhydride (20 mmol) was added and the reaction mixture was stirred over night at 30 °C. After completion the reaction mixture was diluted with water, extracted with ether several times and the combined organic phases dried over sodium sulfate, concentrated in vacuo and purified by column chromatography (indicated conditions).

Methylenation, General Procedure gp6:[6,7] The reactions were carried out under an argon atmosphere in the dark. The lactone (1.0 mmol) and dicyclopentadienyl-dimethyltitanocene (**4**, 2.2 mmol) were dissolved in anhydrous toluene (5 mL), heated to 60 °C and stirred for the given time. After completion, the solvent was removed in vacuo, and the residue was purified by column chromatography (indicated conditions).

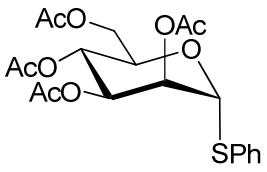
Bishydroxylation, General Procedure gp7:[8,9,10] The olefine (1.0 mmol) was dissolved in a mixture of *tert*-butanol/water (1:1 v/v, 20 mL) and potassium carbonate (3.0 mmol) was added. Then potassium hexacyanoferrate(III) (ferricyanide, 3.0 mmol) and a catalytic amount of potassium osmate dihydrate were added. The reaction mixture was stirred at room temperature for the given time. After completion the reaction mixture was diluted with ethylacetate and washed with water. The organic layer was dried over sodium sulfate, concentrated in vacuo and purified by column chromatography (indicated conditions).

Hydrogenation, General Procedure gp8: The monosaccharide (1.0 mmol) was dissolved in anhydrous methanol (15 mL). Then a catalytic amount of 10% Pd/C was added and the reaction mixture was stirred under a hydrogen atmosphere for the given time. After the reaction was completed, it was filtered, concentrated in vacuo and if necessary purified by column chromatography (indicated conditions).

Syntheses of 1 and 2:

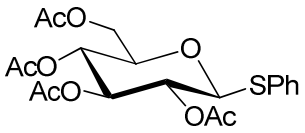


Phenyl 2,3,4,6-tetra-*O*-acetyl-1-thio-α-D-mannopyranoside (7)



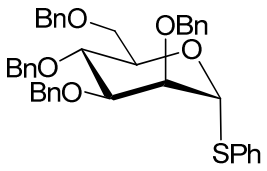
For peracetylation D-mannose (**5**, 12.0 g, 66.6 mmol) and acetic anhydride (60 mL, 635 mmol) were used. Then the crude penta acetate (26.0 g, 66.6 mmol), $BF_3{\cdot}Et_2O$ (16.7 mL, 81.1 mmol) and thiophenol (9.72 mL, 91.0 mmol) in anhydrous dichloromethane (100 mL) were used according to **gp1**. Reaction time was 3 hours and the residue was crystallised from Et_2O to obtain pure **7** (23.5 g, 53.4 mmol) as a colourless solid in 80% yield. The physical data of **7** match those reported.[11]

Phenyl 2,3,4,6-tetra-*O*-acetyl-1-thio-β-D-glucopyranoside (8)



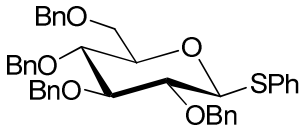
For peracetylation D-glucose (**6**, 23.1 g, 128 mmol) and acetic anhydride (120 mL, 1.27 mol) were used. Then the crude penta acetate (49.9 g, 128 mmol), $BF_3{\cdot}Et_2O$ (32.0 mL, 156 mmol) and thiophenol (18.7 mL, 175 mmol) in anhydrous dichloromethane (150 mL) were used according to **gp1**. Reaction time was 3 hours and the residue was crystallised from Et_2O to obtain pure **8** (47.8 g, 109 mmol) as a colourless solid in 85% yield. The physical data of **8** match those reported.[12]

Phenyl 2,3,4,6-tetra-*O*-benzyl-1-thio-α-D-mannopyranoside (9)



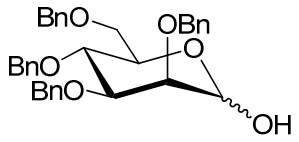
Compound **7** (10.8 g, 24.6 mmol) in anhydrous methanol (70 mL) were used according to **gp2**. The crude deacetylated product (6.70 g, 24.6 mmol), sodium hydride (3.53 g, 147 mmol), benzylbromide (13.0 mL, 109 mmol), TBAI (914 mg, 2.46 mmol) in anhydrous DMF (200 mL) were used according to **gp3**. The crude product was crystallised from Et_2O/PE to obtain pure **9** (15.2 g, 24.0 mmol) as a colourless solid in 97% yield. The physical data of **9** match those reported.[13]

Phenyl 2,3,4,6-tetra-*O*-benzyl-1-thio-β-D-glucopyranoside (10)



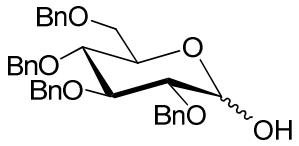
Compound **8** (15.9 g, 35.0 mmol) in anhydrous methanol (100 mL) were used according to **gp2**. The crude deacetylated product (9.53 g, 35.0 mmol), sodium hydride (4.93 g, 206 mmol), benzylbromide (18.3 mL, 154 mmol), TBAI (1.30 g, 3.50 mmol) in anhydrous DMF (250 mL) were used according to **gp3**. The crude product was crystallised from Et_2O/PE to obtain pure **10** (21.0 g, 33.3 mmol) as a colourless solid in 95% yield. The physical data of **10** match those reported.[14]

2,3,4,6-Tetra-*O*-benzyl-D-mannopyranose (11)



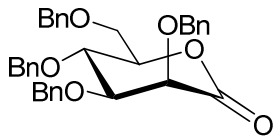
Compound **9** (10.0 g, 15.8 mmol), NBS (7.03 g, 19.5 mmol) in acetone/water (250 mL) were used according to **gp4**. Reaction time was 2 hours and the residue was subjected to column chromatography (PE/EE 4:1) to obtain pure **11** (8.40 g, 15.5 mmol) as a colourless oil in 98% yield. The physical data of **11** match those reported.[15]

2,3,4,6-Tetra-*O*-benzyl-D-glucopyranose (12)



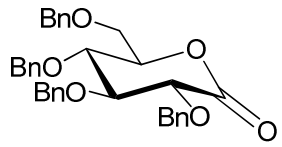
Compound **10** (10.0 g, 15.8 mmol), NBS (7.03 g, 19.5 mmol) in acetone/water (250 mL) were used according to **gp4**. Reaction time was 3 hours and the residue was crystallised from EE to obtain pure **12** (8.34 g, 15.4 mmol) as a colourless solid in 97% yield. The physical data of **12** match those reported.[16]

2,3,4,6-Tetra-*O*-benzyl-D-mannono-1,5-lactone (13)



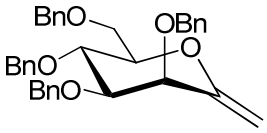
Compound **11** (10.0 g, 18.5 mmol), acetic anhydride (29.0 mL, 306 mmol) and DMSO (36.0 mL) were used according to **gp5**. The crude product was crystallised from Et_2O/PE to obtain pure **13** (9.59 g, 17.8 mmol) as a colourless solid in 96% yield. The physical data of **13** match those reported.[17]

2,3,4,6-Tetra-*O*-benzyl-D-glucono-1,5-lactone (14)



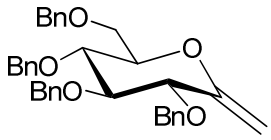
Compound **12** (4.10 g, 7.59 mmol), acetic anhydride (13.0 mL, 137 mmol) and DMSO (16.0 mL) were used according to **gp5**. The crude product was subjected to column chromatography (PE/EE 4:1) to obtain pure **14** (3.85 g, 7.15 mmol) as a colourless oil in 94% yield. The physical data of **14** match those reported.[18]

2,6-Anhydro-3,4,5,7-tetra-*O*-benzyl-1-deoxy-D-mannohept-1-enitol (15)



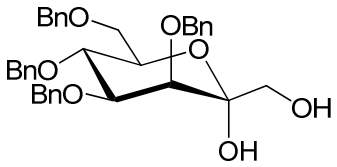
Compounds **13** (2.84 g, 5.28 mmol) and **4** (2.20 g, 11.1 mmol) were used according to **gp6** in toluene (80 mL). Reaction time was 72 hours and the residue was subjected to column chromatography (PE/EE 15:1 + 0.5% triethylamine) to obtain pure **15** (2.50 g, 4.66 mmol) as a slightly yellow oil in 88% yield. $[\alpha]_D^{20}$ = +18.0° (*c* 1.6, $CHCl_3$); Lit.[7]: $[\alpha]_D^{25}$ = +16.5° (*c* 1.2, $CHCl_3$); R_f = 0.65 (PE/EE 6:1, v/v); HRMS (ESI) m/z for $C_{35}H_{36}O_5$: $[M+H]^+$ calcd 537.2636, found 537.2640; $[M+Na]^+$ calcd 559.2455, found 559.2461; $[M+K]^+$ calcd 575.2194, found 575.2203; ^{1}H-NMR (400 MHz, DMSO-d_6): δ 7.43-7.10 (m, 20H, C**H**-arom.), 4.77 (d, *J*=11.0 Hz, 1H, C**H**$_2$Ph), 4.68 (s, 1H, **H**-1a), 4.62 (d, *J*=11.9 Hz, 1H, C**H**$_2$Ph), 4.57 (d, *J*=12.1 Hz, 1H, C**H**$_2$Ph), 4.56 (d, *J*=12.4 Hz, 1H, C**H**$_2$Ph), 4.54 (d, *J*=11.9 Hz, 1H, C**H**$_2$Ph), 4.53 (s, 1H, **H**-1b), 4.50 (d, *J*=11.0 Hz, 1H, C**H**$_2$Ph), 4.49 (d, *J*=12.4 Hz, 1H, C**H**$_2$Ph), 4.39 (d, *J*=12.1 Hz, 1H, C**H**$_2$Ph), 4.31 (d, $J_{3,4}$=3.1 Hz, 1H, **H**-3), 3.98 (dd, $J_{4,5}$=9.0 Hz, $J_{5,6}$=8.8 Hz, 1H, **H**-5), 3.72-3.67 (m, 3H, **H**-4, **H**-7), 3.57-3.61 (ddd, $J_{5,6}$=8.8 Hz, $J_{6,7a}$=3.0 Hz, $J_{6,7b}$=6.7 Hz, 1H, **H**-6); ^{13}C-NMR (100.6 MHz, DMSO-d_6): δ 155.0 (**C**-2), 138.4, 138.3, 138.3, 138.2 (**C**q-arom.), 128.2, 128.2, 128.1, 128.1, 127.7, 127.6, 127.4, 127.4, 127.4, 127.3 (**C**H-arom.), 97.9 (**C**-1), 80.3 (**C**-4), 79.1 (**C**-6), 74.1 (**C**-3), 73.8 (**C**H_2Ph), 73.6 (**C**-5), 72.3, 70.2, 69.3 (**C**H_2Ph), 69.0 (**C**-7).

2,6-Anhydro-3,4,5,7-tetra-*O*-benzyl-1-deoxy-D-glucohept-1-enitol (16)



Compounds **14** (2.04 g, 3.80 mmol) and **4** (1.6 g, 7.7 mmol) were used according to **gp6** in toluene (20 mL). Reaction time was 48 hours and the residue was subjected to column chromatography (PE/EE 10:1 + 0.5% triethylamine) to obtain pure **15** (1.65 g, 3.08 mmol) as a slightly yellow solid in 81% yield. $[\alpha]_D^{20}$ = +53.4° (*c* 1.0, $CHCl_3$); Lit.[7]: $[\alpha]_D^{25}$ = +58.4° (*c* 1.0, CH_2Cl_2); R_f = 0.31 (PE/EE 10:1, v/v); melting point 65-67 °C; Lit[7].: 65-68 °C; HRMS (ESI) m/z for $C_{35}H_{36}O_5$: $[M+H]^+$ calcd 537.2636, found 537.2639; $[M+Na]^+$ calcd 559.2455, found 559.2456; ^{1}H-NMR (400 MHz, DMSO-d_6): δ 7.39-7.15 (m, 20H, C**H**-arom.), 4.72 (d, *J*=11.5 Hz, 1H, C**H**$_2$Ph), 4.68 (d, *J*=12.0 Hz, 1H, C**H**$_2$Ph), 4.65 (d, *J*=11.0 Hz, 1H, C**H**$_2$Ph), 4.62 (d, *J*=11.5 Hz, 1H, C**H**$_2$Ph), 4.57 (d, *J*=12.0 Hz, 1H, C**H**$_2$Ph), 4.56 (s, 1H, **H**-1a), 4.54 (d, *J*=12.3 Hz, 1H, C**H**$_2$Ph), 4.51 (d, *J*=12.3 Hz, 1H, C**H**$_2$Ph), 4.48 (d, *J*=11.0 Hz, 1H, C**H**$_2$Ph), 4.44 (s, 1H, **H**-1b), 4.04 (d, $J_{3,4}$=5.4 Hz, 1H, **H**-3), 3.90 (ddd, $J_{5,6}$=9.9 Hz, $J_{6,7a}$=1.8 Hz, $J_{6,7b}$=4.5 Hz, 1H, **H**-6), 3.71-3.68 (m, 2H, **H**-4, **H**-7a), 3.63 (dd, $J_{6,7b}$=4.5 Hz, $J_{7a,7b}$=11.0 Hz, 1H, **H**-7b), 3.60 (dd, $J_{4,5}$=6.2 Hz, $J_{5,6}$=9.9 Hz, 1H, **H**-5); ^{13}C-NMR (100.6 MHz, DMSO-d_6): δ 155.0 (**C**-2), 138.1, 138.1, 137.9, 137.9 (**C**q-arom.), 128.2, 128.2, 128.1, 127.8, 127.7, 127.6, 127.6, 127.5, 127.5, 127.5, 127.4 (**C**H-arom.), 93.3 (**C**-1), 82.3 (**C**-4), 77.3 (**C**-3), 77.1 (**C**-5), 76.2 (**C**-6), 68.8 (**C**-7).

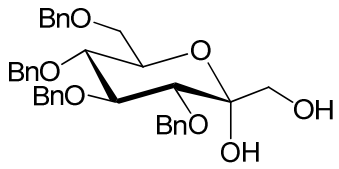
3,4,5,7-Tetra-*O*-benzyl-α-D-*glycero*-D-*lyxo*-hept-2-ulopyranose (17)



Compound **15** (200 mg, 373 µmol), potassium ferricyanide (370 mg, 1.12 mmol), potassium carbonate (160 mg, 1.16 mmol) and *tert*-butanol/water (10 mL) were used according to **gp7**. Reaction time was 24 hours and the residue was subjected to column chromatography (Et_2O) to obtain pure **17** (200 mg, 351 µmol) as a colourless oil in 94% yield. $[\alpha]_D^{24}$ = +30.1° (*c* 0.57, $CHCl_3$); Lit.[19]: $[\alpha]_D^{25}$ = +31.2° (*c* 1.0, $CHCl_3$); R_f = 0.55 (Et_2O); HRMS (ESI) m/z for $C_{35}H_{38}O_7$: $[M+Na]^+$ calcd 593.2510, found 593.2509; ^{1}H-NMR (400 MHz, DMSO-d_6): δ 7.40-7.23 (m, 18H, C**H**-arom.), 7.20-7.17 (m, 2H, C**H**-arom.), 5.90 (s, 1H, C^2-O**H**), 4.82-4.73 (m, 4H, C**H**$_2$Ph, C^1-O**H**), 4.68 (d, *J*=11.1 Hz, 1H, C**H**$_2$Ph), 4.62 (d, *J*=11.9 Hz, 1H, C**H**$_2$Ph), 4.53 (d, *J*=12.0 Hz, 1H, C**H**$_2$Ph), 4.49 (d, *J*=11.1 Hz, 1H, C**H**$_2$Ph), 4.43 (d, *J*=12.0 Hz, 1H, C**H**$_2$Ph), 3.99 (d, $J_{3,4}$=2.6 Hz, 1H, **H**-3), 3.97 (dd, $J_{3,4}$=2.6 Hz, $J_{4,5}$=8.7 Hz, 1H, **H**-4), 3.83-3.80 (m, 1H, **H**-6), 3.77 (dd, $J_{4,5}$=8.7 Hz, $J_{5,6}$=9.9 Hz, 1H, **H**-5), 3.63 (dd, $J_{6,7a}$=3.8 Hz, $J_{7a,7b}$=10.8 Hz, 1H, **H**-7a), 3.60-3.55 (m, 2H, **H**-1a, **H**-7b), 3.32 (dd, $J_{1b,OH}$=5.0 Hz, $J_{1a,1b}$=10.6, Hz, 1H, **H**-1b); ^{13}C-NMR (100.6

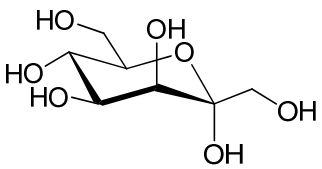
MHz, DMSO-d_6): δ 139.2, 138.7, 138.6, 138.4 (**C**q-arom.), 128.2, 128.1, 128.1, 128.0, 127.7, 127.6, 127.4, 127.4, 127.3, 127.1 (**C**H-arom.), 98.2 (**C**-2), 80.6 (**C**-4), 75.0 (**C**-5), 74.9 (**C**-3), 73.9, 73.8, 72.2 (**C**H_2Ph), 71.5 (**C**-6), 70.7 (**C**H_2Ph), 69.3 (**C**-7), 63.9 (**C**-1).

3,4,5,7-Tetra-*O*-benzyl-α-D-*glycero*-D-*xylo*-hept-2-ulopyranose (18)



Compound **16** (50.0 mg, 93.2 µmol), potassium ferricyanide (88.2 mg, 267 µmol), potassium carbonate (38.3 mg, 277 µmol) and *tert*-butanol/water (4 mL) were used according to **gp7**. Reaction time was 24 hours and the residue was subjected to column chromatography (Et_2O) to obtain pure **18** (50.0 mg, 87.7 µmol) as a colourless solid in 94% yield. $[\alpha]_D^{20}$ = +12.8° (*c* 1.0, $CHCl_3$); Lit.[20]: $[\alpha]_D^{25}$ = +14.7° (*c* 1.0, $CHCl_3$); R_f = 0.63 (Et_2O); melting point 110-112 °C; Lit.[20]: 112-113 °C; HRMS (ESI) m/z for $C_{35}H_{38}O_7$: $[M+Na]^+$ calcd 593.2510, found 593.2509; ^{1}H-NMR (400 MHz, DMSO-d_6): δ 7.36-7.24 (m, 18H, C**H**-arom.), 7.19-7.16 (m, 2H, C**H**-arom.), 5.82 (s, 1H, C^2-O**H**), 4.88 (dd, $J_{OH,1b}$=5.2 Hz, $J_{OH,1a}$=6.9 Hz, 1H, C^1-O**H**), 4.80 (d, *J*=11.2 Hz, 1H, CH_2Ph), 4.76(d, *J*=11.2 Hz, 1H, CH_2Ph), 4.74 (d, *J*=11.1 Hz, 1H, CH_2Ph), 4.73 (d, *J*=10.9 Hz, 1H, CH_2Ph), 4.66 (d, *J*=11.1 Hz, 1H, CH_2Ph), 4.54 (d, *J*=12.0 Hz, 1H, CH_2Ph), 4.53 (d, *J*=10.9 Hz, 1H, CH_2Ph), 4.47 (d, *J*=12.0 Hz, 1H, CH_2Ph), 3.89 (dd, $J_{3,4}$=9.7 Hz, $J_{4,5}$=9.3 Hz, 1H, **H**-4), 3.87 (ddd, $J_{5,6}$=9.8 Hz, $J_{6,7a}$=4.7 Hz, $J_{6,7b}$=1.3 Hz, 1H, **H**-6), 3.65 (dd, $J_{6,7a}$=4.7 Hz, $J_{7a,7b}$=10.8 Hz, 1H, **H**-7a), 3.60 (dd, $J_{6,7b}$=1.3 Hz, $J_{7a,7b}$=10.8 Hz, 1H, **H**-7b), 3.57 (d, $J_{3,4}$=9.7 Hz, 1H, **H**-3), 3.49 (dd, $J_{1a,OH}$=6.9 Hz, $J_{1a,1b}$=11.2 Hz, 1H, **H**-1a), 3.44 (dd, $J_{4,5}$=9.3 Hz, $J_{5,6}$=9.8 Hz, 1H, **H**-5), 3.37 (dd, $J_{1b,OH}$=5.2 Hz, $J_{1a,1b}$=11.2 Hz, 1H, **H**-1b); ^{13}C-NMR (100.6 MHz, DMSO-d_6): δ 138.8, 138.7, 138.4, 138.3 (**C**q-arom.), 128.2, 128.2, 128.1, 128.1, 127.8, 127.6, 127.6, 127.4, 127.4, 127.3, 127.3 (**C**H-arom.), 97.8 (**C**-2), 82.7 (**C**-4), 78.8 (**C**-3), 78.5 (**C**-5), 74.4, 74.1, 73.8, 72.3 (**C**H_2Ph), 70.5 (**C**-6), 69.1 (**C**-7), 63.9 (**C**-1).

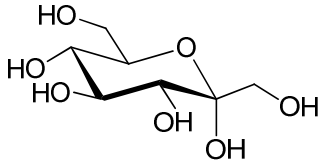
α-D-*glycero*-D-*lyxo*-Hept-2-ulopyranose (1)



Compound **17** (150 mg, 263 µmol) and methanol (10 mL) were used according to **gp8**. Reaction time was 48 hours and the residue was subjected to column chromatography (RP-18, H_2O) to obtain pure **1** (51.9 mg, 247 µmol) as a colourless solid in 93% yield. $[\alpha]_D^{23}$ = +26.5° (*c* 3.0, H_2O); Lit.[21]: $[\alpha]_D^{20}$ =

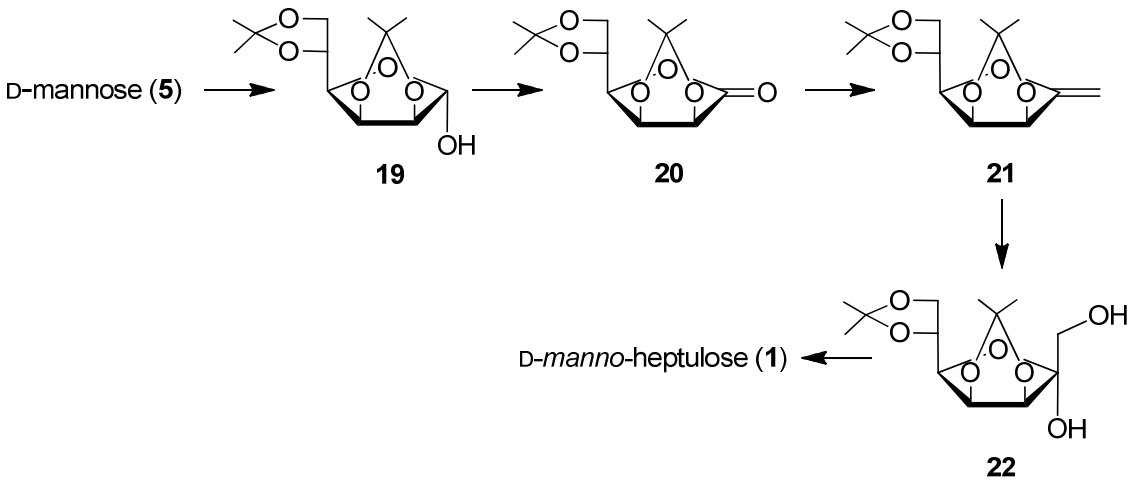
+29.5° (*c* 4.0, H_2O); R_f = 0.89 (RP-18, H_2O); melting point 147-148 °C; Lit.[21]: 151-152 °C; HRMS (ESI) m/z for $C_7H_{14}O_7$: $[M+Na]^+$ calcd 233.0632, found 233.0638; ^{1}H-NMR (500 MHz, D_2O): δ 3.93 (dd, $J_{3,4}$=3.3 Hz, $J_{4,5}$=9.6 Hz, 1H, **H**-4), 3.91 (d, $J_{3,4}$=3.3 Hz, 1H, **H**-3), 3.88 (dd, $J_{6,7a}$=1.5 Hz, $J_{7a,7b}$=11.9 Hz, 1H, **H**-7a), 3.81 (ddd, $J_{5,6}$=9.8 Hz, $J_{6,7a}$=1.5 Hz, $J_{6,7b}$=6.0 Hz, 1H, **H**-6), 3.75 (dd, $J_{6,7b}$=6.0 Hz, $J_{7a,7b}$=11.9 Hz, 1H, **H**-7b), 3.73 (d, $J_{1a,1b}$=11.8 Hz, 1H, **H**-1a), 3.62 (dd, $J_{4,5}$=9.6 Hz, $J_{5,6}$=9.8 Hz, 1H, **H**-5), 3.58 (d, $J_{1a,1b}$=11.8 Hz, 1H, **H**-1b); ^{13}C-NMR (125.8 MHz, D_2O): δ 97.8 (**C**-2), 73.0 (**C**-6), 70.9 (**C**-4), 69.9 (**C**-3), 66.9 (**C**-5), 64.1 (**C**-1), 61.1 (**C**-7).

α-D-*glycero*-D-*xylo*-Hept-2-ulopyranose (2)

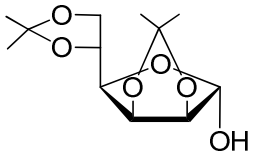


Compound **18** (45.2 mg, 79.3 µmol) and methanol (10 mL) were used according to **gp8**. Reaction time was 48 hours and the residue was subjected to column chromatography (RP-18, H_2O) to obtain pure **2** (16.1 mg, 76.8 µmol) as a colourless oil in 97% yield. $[\alpha]_D^{23}$ = +54.5° (*c* 0.71, H_2O); Lit.[21]: $[\alpha]_D^{20}$ = +64.4° (*c* 4.0, H_2O); R_f = 0.93 (RP-18, H_2O); HRMS (ESI) m/z for $C_7H_{14}O_7$: $[M+Na]^+$ calcd 233.0632, found 233.0631; ^{1}H-NMR (500 MHz, D_2O): δ 3.85 (dd, $J_{6,7a}$=3.6 Hz, $J_{7a,7b}$=12.0 Hz, 1H, **H**-7a), 3.82-3.76 (m, 2H, **H**-6, **H**-7b), 3.71 (d, $J_{1a,1b}$=11.7 Hz, 1H, **H**-1a), 3.72 (dd, $J_{3,4}$=9.6 Hz, $J_{4,5}$=9.3 Hz, 1H, **H**-4), 3.56 (d, $J_{1a,1b}$=11.7 Hz, 1H, **H**-1b), 3.52 (d, $J_{3,4}$=9.6 Hz, 1H, **H**-3), 3.43 (dd, $J_{4,5}$=9.3 Hz, $J_{5,6}$=9.5 Hz, 1H, **H**-5); ^{13}C-NMR (125.8 MHz, D_2O): δ 97.4 (**C**-2), 73.7 (**C**-4), 72.3 (**C**-6), 70.3 (**C**-3), 69.5 (**C**-5), 63.7 (**C**-1), 60.7 (**C**-7).

Alternative Synthesis of 1:

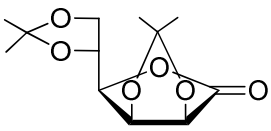


2,3:5,6-Di-*O*-isopropylidene-α-D-mannofuranose (19)



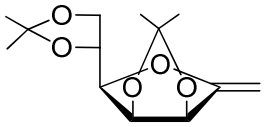
The reaction was carried out following the previously reported reaction protocol[22] using D-mannose (**5**, 30.0 g, 166 mmol), anhydrous acetone (1.00 L) and conc. sulphuric acid (21 mL). After crystallisation pure **19** (41.0 g, 157 mmol) was isolated as a colourless solid in 95% yield. The physical data of **19** match those reported.[22]

2,3:5,6-Di-*O*-isopropylidene-α-D-mannono-1,4-lactone (20)



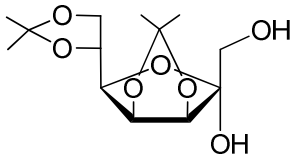
Compound **19** (20.0 g, 76.8 mmol), acetic anhydride (40.0 mL, 423 mmol) and DMSO (200 mL) were used according to **gp5**. Reaction time was 12 hours and the residue crystallised from Et_2O/PE to obtain pure **20** (16.5 g, 63.9 mmol) as a colourless solid in 83% yield. The physical data of **20** match those reported.[23]

2,5-Anhydro-1-deoxy-3,4:6,7-di-*O*-isopropylidene-D-mannohept-1-enitol (21)



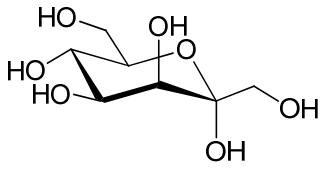
Compounds **20** (5.00 g, 19.4 mmol) and **4** (7.91 g, 39.9 mmol) were used according to **gp6** in 100 mL toluene. Reaction time was 24 hours and the residue was subjected to column chromatography (PE/EE 10:1 + 0.5% triethylamine) to obtain pure **21** (4.00 g, 15.5 mmol) as a slightly yellow oil in 80% yield. $[\alpha]_D^{23}$ = +112.5° (*c* 0.36, $CHCl_3$); Lit.[7]: $[\alpha]_D^{25}$ = +153.5° (*c* 4.0, $CHCl_3$); R_f = 0.66 (PE/EE 4:1, v/v); HRMS (ESI) m/z for $C_{13}H_{20}O_5$: $[M+Na]^+$ calcd 279.1203, found 279.1204; ^{1}H-NMR (400 MHz, DMSO-d_6): δ 5.09 (d, $J_{3,4}$=5.7 Hz, 1H, **H**-3), 4.72 (dd, $J_{3,4}$=5.7 Hz, $J_{4,5}$=3.8 Hz, 1H, **H**-4), 4.33 (s, 1H, **H**-1a), 4.28 (ddd, $J_{5,6}$=6.7 Hz, $J_{6,7a}$=6.5 Hz, $J_{6,7b}$=5.5 Hz, 1H, **H**-6), 4.15 (dd, $J_{4,5}$=3.8 Hz, $J_{5,6}$=6.7 Hz, 1H, **H**-5), 4.11 (s, 1H, **H**-1b), 4.03 (dd, $J_{6,7a}$=6.5 Hz, $J_{7a,7b}$=8.4 Hz, 1H, **H**-7a), 3.85 (dd, $J_{6,7b}$=5.5 Hz, $J_{7a,7b}$=8.4 Hz, 1H, **H**-7b), 1.36 (s, 3H, C**H**$_3$), 1.34 (s, 3H, C**H**$_3$), 1.29 (s, 3H, C**H**$_3$), 1.28 (s, 3H, C**H**$_3$); ^{13}C-NMR (100.6 MHz, DMSO-d_6): δ 162.0 (**C**-2), 112.2, 108.2 (**C**q), 85.1 (**C**-1), 81.6 (**C**-5), 78.0 (**C**-4), 79.4 (**C**-3), 72.7 (**C**-6), 65.6 (**C**-7), 26.6, 26.4, 25.5, 25.1 (**C**H$_3$).

3,4:6,7-Di-*O*-isopropylidene-α-D-*glycero*-D-*lyxo*-hept-2-ulofuranose (22)



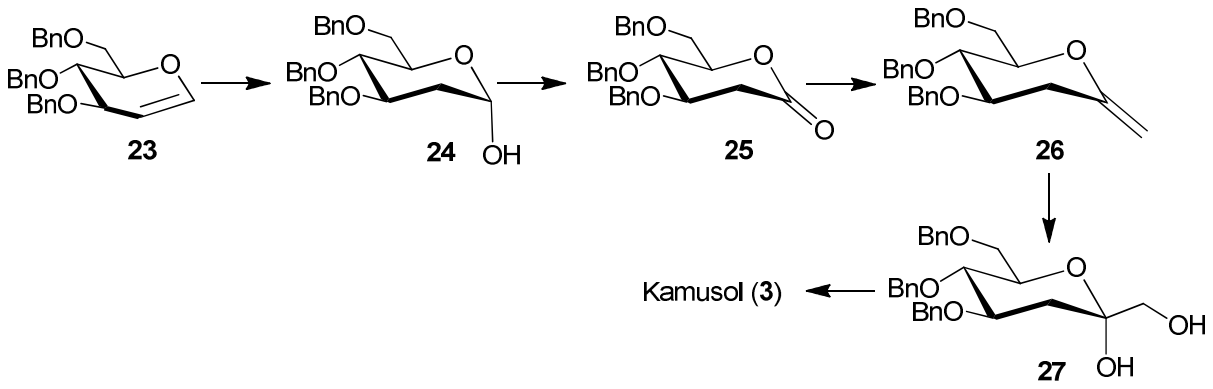
Compound **21** (2.10 g, 8.20 mmol), potassium ferricyanide (8.20 g, 24.9 mmol), potassium carbonate (3.56 g, 25.7 mmol) and *tert*-butanol/water (220 mL) were used according to **gp7**. Reaction time was 24 hours and the residue was subjected to column chromatography (Et_2O) to obtain pure **22** (2.26 g, 7.79 mmol) as a colourless solid in 95% yield. $[\alpha]_D^{23}$ = +17.7° (*c* 0.86, $CHCl_3$); Lit.[24]: $[\alpha]_D^{25}$ = +20.4° (*c* 1.0, $CHCl_3$); R_f = 0.46 (Et_2O); melting point 85-86 °C; Lit.[24]: 88-89 °C; HRMS (ESI) m/z for $C_{13}H_{22}O_7$: $[M+H]^+$ calcd 291.1438, found 291.1442; $[M+Na]^+$ calcd 313.1258, found 313.1265; ^{1}H-NMR (400 MHz, DMSO-d_6): δ 5.82 (s, 1H, C^1-O**H**), 4.72 (dd, $J_{3,4}$=5.7 Hz, $J_{4,5}$=3.9 Hz, 1H, **H**-4), 4.56 (dd, $J_{1a,OH}$=6.7 Hz, $J_{1b,OH}$=5.2 Hz, 1H, C^2-O**H**), 4.37 (d, $J_{3,4}$=5.9 Hz, 1H, **H**-3), 4.22 (ddd, $J_{5,6}$=3.0 Hz, $J_{6,7a}$=6.0 Hz, $J_{6,7b}$=5.7 Hz, 1H, **H**-6), 3.98 (dd, $J_{4,5}$=3.9 Hz, $J_{5,6}$=3.0 Hz, 1H, **H**-5), 3.96-3.93 (m, 1H, **H**-7a), 3.79 (dd, $J_{6,7b}$=5.7 Hz, $J_{7a,7b}$=8.2 Hz, 1H, **H**-7b), 3.52 (dd, $J_{1a,OH}$=6.7 Hz, $J_{1a,1b}$=11.1 Hz, 1H, **H**-1a), 3.39 (dd, $J_{1b,OH}$=5.2 Hz, $J_{1a,1b}$=11.1 Hz, 1H, **H**-1b), 1.32 (s, 3H, C**H**$_3$), 1.35 (s, 3H, C**H**$_3$), 1.26 (s, 3H, C**H**$_3$), 1.23 (s, 3H, C**H**$_3$); ^{13}C-NMR (100.6 MHz, DMSO-d_6): δ 111.2, 107.8 (**C**q), 105.0 (**C**-2), 84.4 (**C**-3), 79.6 (**C**-4), 78.3 (**C**-5), 72.7 (**C**-6), 65.9 (**C**-7), 62.2 (**C**-1), 26.6, 25.7, 25.1, 24.2 (**C**H$_3$).

α-D-*glycero*-D-*lyxo*-Hept-2-ulopyranose (1)

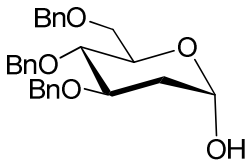


Compound **22** (1.70 g, 5.86 mmol) was suspended in distilled water (100 mL). Then Amberlite® IR-120 H^+ was added until the reaction mixture reached pH 1 and stirring was continued over night at room temperature. After completion the ion exchange was removed by filtration and the solution was lyophilised. The crude product was purified by column chromatography (RP-18, H_2O) and recrystallised from anhydrous ethanol to give pure **1** (1.21 g, 5.76 mmol) as a colourless solid in 98% yield. Data see above.

Synthesis of Kamusol (3):

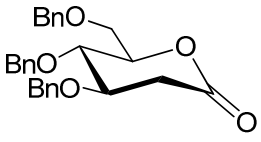


3,4,6-Tri-*O*-benzyl-2-deoxy-α-D-glucopyranose (24)



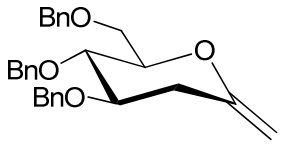
The reaction was carried out following the previously reported reaction protocol[25] using first 3,4,6-tri-*O*-benzyl-D-glucal **23** (1.25 g, 3.00 mmol), NIS (760 mg, 3.38 mmol) in MeCN/H_2O (24 mL), then $Na_2S_2O_4$ (2.31 g, 13.3 mmol), H_2O/$NaHCO_3$ (30 mL) in DMF (35 mL) to give **24** (1.26 g, 2.90 mmol) as a colourless solid in 97% yield. The physical data of **24** match those reported.[26]

3,4,6-Tri-*O*-benzyl-2-deoxy-D-glucono-1,5-lactone (25)



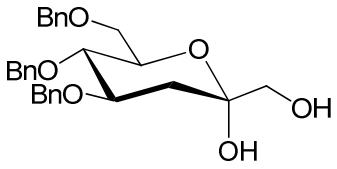
The reaction was carried out under an argon atmosphere. Compound **24** (1.10 g, 2.53 mmol) was dissolved in anhydrous dichloromethane (20 mL) containing freshly dried 4 Å molecular sieves. Then PCC (545 mg, 2.53 mmol) was added for a total of three times at intervals of 2 hours. The reaction mixture was heated to reflux and stirred over night. After completion the reaction mixture was filtered over Celite®, concentrated in vacuum and the crude product purified by column chromatography (PE/Et_2O 1:1) to give pure **25** (828 mg, 1.92 mmol) as a colourless solid in 75% yield. The physical data of **25** match those reported.[27]

2,6-Anhydro-1,3-dideoxy-4,5,7-tri-*O*-benzyl-D-glucohept-1-enitol (26)



Compounds **25** (1.14 g, 2.64 mmol) and **4** (1.21 g, 5.81 mmol) were used according to **gp6** in toluene (20 mL). Reaction time was 48 hours and the residue was subjected to column chromatography (PE/Et_2O 5:1 + 0.5% triethylamine) to obtain pure **26** (901 mg, 2.09 mmol) as a colourless solid in 79% yield. $[\alpha]_D^{23}$ = +36.4° (*c* 0.23, $CHCl_3$); R_f = 0.49 (PE/Et_2O 5:1, v/v); melting point 42-43 °C; HRMS (ESI) m/z for $C_{28}H_{30}O_4$: $[M+Na]^+$ calcd 453.2036, found 453.2041; $[M+K]^+$ calcd 469.1776, found 469.1778; ^{1}H-NMR (500 MHz, DMSO-d_6): δ 7.38-7.25 (m, 13H, C**H**-arom.), 7.23-7.19 (m, 2H, C**H**-arom.), 4.72 (d, *J*=11.3 Hz, 1H, C$\mathbf{H}_2$Ph), 4.65 (d, *J*=11.9 Hz, 1H, C$\mathbf{H}_2$Ph), 4.56-4.46 (m, 4H, C$\mathbf{H}_2$Ph), 4.29 (s, 1H, **H**-1a), 4.14 (s, 1H, **H**-1b), 3.73-3.67 (m, 2H, **H**-4, **H**-6), 3.66 (s, 2H, **H**-7), 3.55 (dd, $J_{4,5}$=6.5 Hz, $J_{5,6}$=8.2 Hz, 1H, **H**-5), 2.80 (dd, $J_{3ax,3eq}$=14.2 Hz, $J_{3eq,4}$=4.5 Hz, 1H, **H**-3_{eq}), 2.28 (dd, $J_{3ax,3eq}$=14.2 Hz, $J_{3ax,4}$=8.1 Hz, 1H, **H**-3_{ax}); ^{13}C-NMR (125.8 MHz, DMSO-d_6): δ 155.9 (**C**-2), 138.4, 138.2, 138.1 (**C**q-arom.), 128.2, 128.2, 128.1, 127.7, 127.6, 127.5, 127.5, 127.4, 127.4 (**C**H-arom.), 91.8 (**C**-1), 77.4 (**C**-4), 77.3 (**C**-6), 76.8 (**C**-5), 72.7, 72.3, 69.9 (**C**H_2Ph), 69.0 (**C**-7), 32.3 (**C**-3).

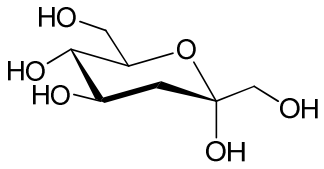
3-Deoxy-4,5,7-tri-*O*-benzyl-α-D-*glycero*-D-*xylo*-hept-2-ulopyranose (27)



Compound **26** (300 mg, 697 µmol), potassium ferricyanide (700 mg, 1.56 mmol), potassium carbonate (300 mg, 1.63 mmol) and *tert*-butanol/water (18 mL) were used according to **gp7**. Reaction time was 72 hours and the residue was subjected to column chromatography (Et_2O) to obtain pure **27** (280 mg, 625 µmol) as a colourless oil in 90% yield. $[\alpha]_D^{24}$ = +29.3° (*c* 0.31, $CHCl_3$); R_f = 0.33 (Et_2O); HRMS (ESI) m/z for $C_{28}H_{32}O_6$: $[M+Na]^+$ calcd 487.2091, found 487.2106; $[M+K]^+$ calcd 503.1830, found 503.1840; ^{1}H-NMR (400 MHz, DMSO-d_6): δ 7.36-7.24 (m, 13H, C**H**-arom.), 7.22-7.18 (m, 2H, C**H**-arom.), 5.60 (s, 1H, C^2-O**H**), 4.81 (d, *J*=11.2 Hz, 1H, C$\mathbf{H}_2$Ph), 4.63 (d, *J*=12.0 Hz, 1H, C$\mathbf{H}_2$Ph), 4.53 (d, *J*=12.0 Hz, 1H, C$\mathbf{H}_2$Ph), 4.52 (d, *J*=11.7 Hz, 1H, C$\mathbf{H}_2$Ph), 4.48 (d, *J*=11.2 Hz, 1H, C$\mathbf{H}_2$Ph), 4.45 (d, *J*=11.7 Hz, 1H, C$\mathbf{H}_2$Ph), 3.88 (ddd, , $J_{3ax,4}$=12.0 Hz, $J_{3eq,4}$=5.0 Hz$J_{4,5}$=9.0 Hz, 1H, **H**-4), 3.82 (ddd, $J_{5,6}$=9.9 Hz, $J_{6,7a}$=4.7 Hz, $J_{6,7b}$=1.6 Hz, 1H, **H**-6), 3.64 (dd, $J_{6,7a}$=4.7 Hz, 1H, $J_{7a,7b}$=10.7 Hz, **H**-7a), 3.59 (dd, $J_{6,7b}$=1.6 Hz, $J_{7a,7b}$=10.7 Hz, 1H, **H**-7b), 3.34 (dd, $J_{4,5}$=9.0 Hz, $J_{5,6}$=9.9 Hz, 1H, **H**-5), 3.33-3.24 (m, 2H, **H**-1), 2.18 (dd, $J_{3ax,3eq}$=12.6 Hz, $J_{3eq,4}$=5.0 Hz, 1H, **H**-3_{eq}), 1.44 (dd, $J_{3ax,3eq}$=12.6 Hz, $J_{3ax,4}$=12.0 Hz, 1H, **H**-3_{ax}); ^{13}C-NMR (100.6 MHz, DMSO-d_6): δ 138.9, 138.7, 138.3 (**C**q-arom.), 128.2, 128.1, 128.1,

127.8, 127.6, 127.4, 127.3, 127.3 (**C**H-arom.), 97.0 (**C**-2), 78.4 (**C**-5), 77.6 (**C**-4), 73.8, 72.3 (**C**H_2Ph), 71.0 (**C**-6), 70.1 (**C**H_2Ph), 69.4 (**C**-7), 67.6 (**C**-1), 35.9 (**C**-3).

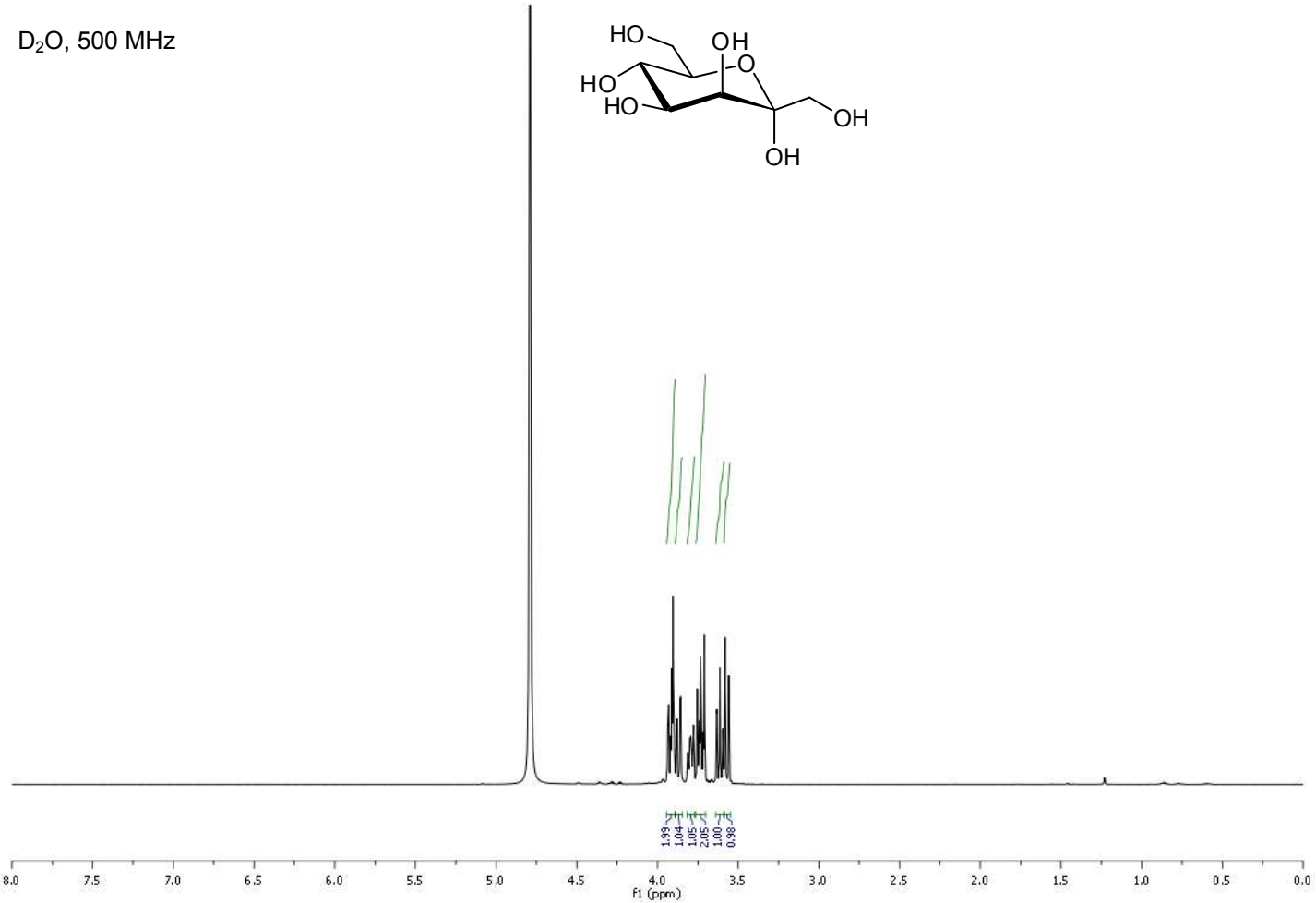
3-Desoxy-α-D-*glycero*-D-*xylo*-hept-2-ulopyranose (3)



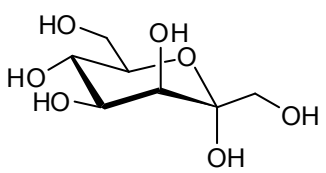
Compound **27** (217 mg, 467 µmol) and methanol (10 mL) were used according to **gp8**. Reaction time was 24 hours and the residue was subjected to column chromatography (RP-18, H_2O) to obtain pure **3** (75.1 mg, 387 µmol) as a colourless oil in 83% yield. $[\alpha]_D^{23}$ = +47.7° (*c* 0.60, H_2O); Lit.[28]: $[\alpha]_D^{20}$ = +25.7° (*c* 0.4, H_2O); HRMS (ESI) m/z for $C_7H_{14}O_6$: $[M+Na]^+$ calcd 217.0683, found 217.0686; ^{1}H-NMR (400 MHz, D_2O): δ 3.98 (ddd, $J_{3ax,4}$=11.8 Hz, $J_{3eq,4}$=5.2 Hz, $J_{4,5}$=9.2 Hz, 1H, **H**-4), 3.87 (dd, $J_{6,7a}$=4.2 Hz, $J_{7a,7b}$=11.4 Hz, 1H, **H**-7a), 3.84 (ddd, $J_{5,6}$=9.4 Hz, $J_{6,7a}$=4.2 Hz, $J_{6,7b}$=2.7 Hz, 1H, **H**-6), 3.81 (dd, $J_{6,7b}$=2.7 Hz, $J_{7a,7b}$=11.4 Hz, 1H, **H**-7b), 3.54 (s, 2H, **H**-1), 3.39 (dd, $J_{4,5}$=9.2 Hz, $J_{5,6}$=9.4 Hz, 1H, **H**-5), 2.11 (dd, $J_{3ax,3eq}$=13.1 Hz, $J_{3eq,4}$=5.2 Hz, 1H, **H**-3_{eq}), 1.69 (dd, $J_{3ax,3eq}$=13.1 Hz, $J_{3ax,4}$=11.8 Hz, 1H, **H**-3_{ax}); ^{13}C-NMR (100.6 MHz, D_2O): δ 97.1 (**C**-2), 73.2 (**C**-6), 71.0 (**C**-5), 68.9 (**C**-4), 67.3 (**C**-1), 60.9 (**C**-7), 37.3 (**C**-3).

NMR Spectra of Key Compounds 1, 2, 3, 15-18, 21, 22, 26, 27

α-D-*glycero*-D-*lyxo*-Hept-2-ulopyranose (1)



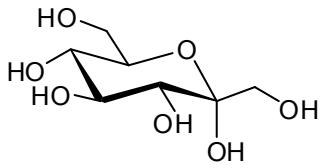
D_2O, 125 MHz


HO
OH
O
HO
HO
OH
OH

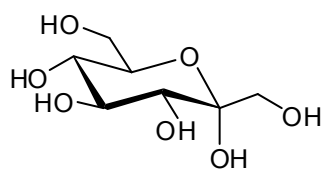
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

α-D-*glycero*-D-*xylo*-Hept-2-ulopyranose (2)

D_2O, 500 MHz



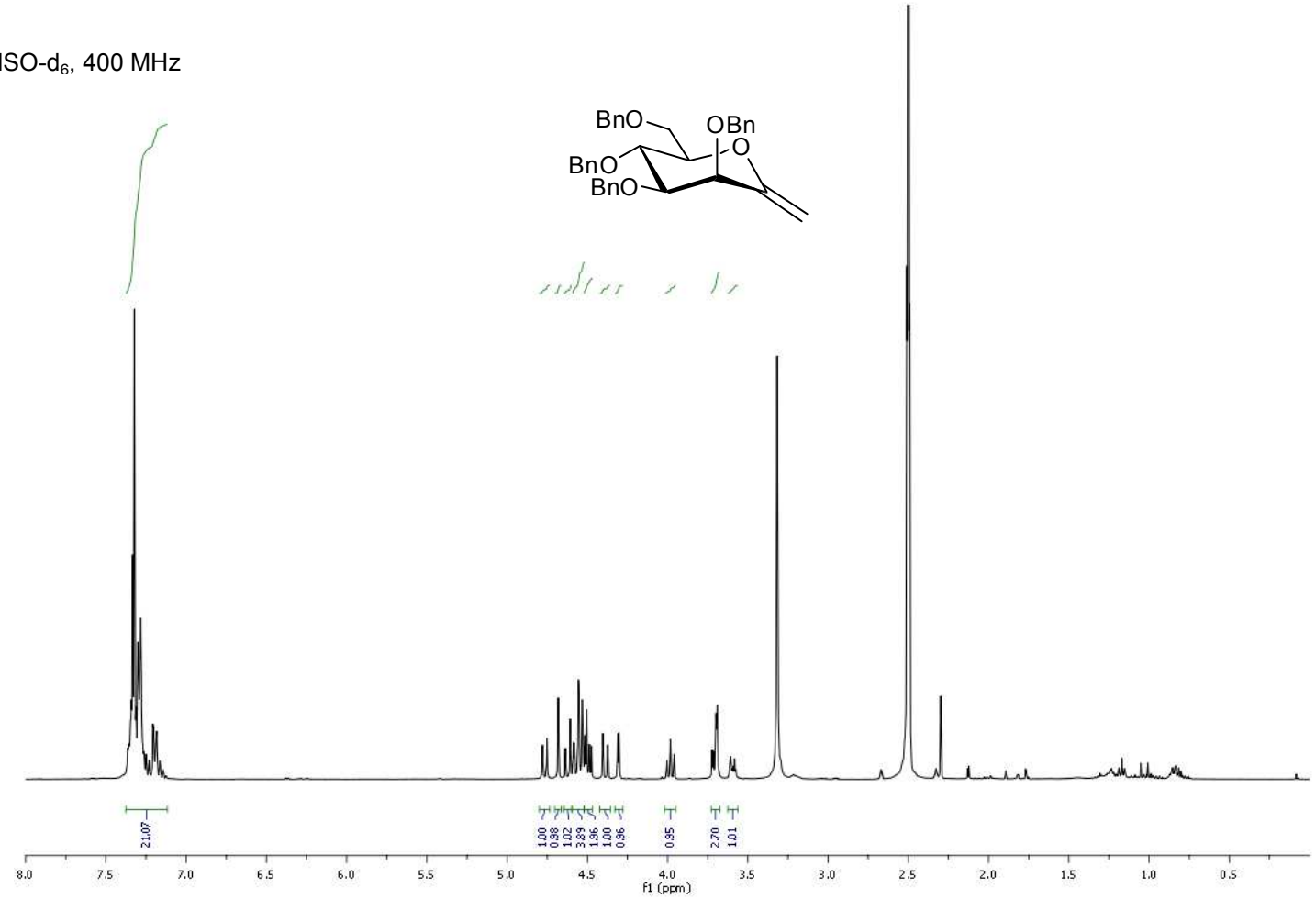
D_2O, 125 MHz


HO
O
HO
HO
OH
OH
OH

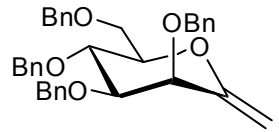
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2,6-Anhydro-3,4,5,7-tetra-*O*-benzyl-1-deoxy-D-mannohept-1-enitol (15)

DMSO-d_6, 400 MHz

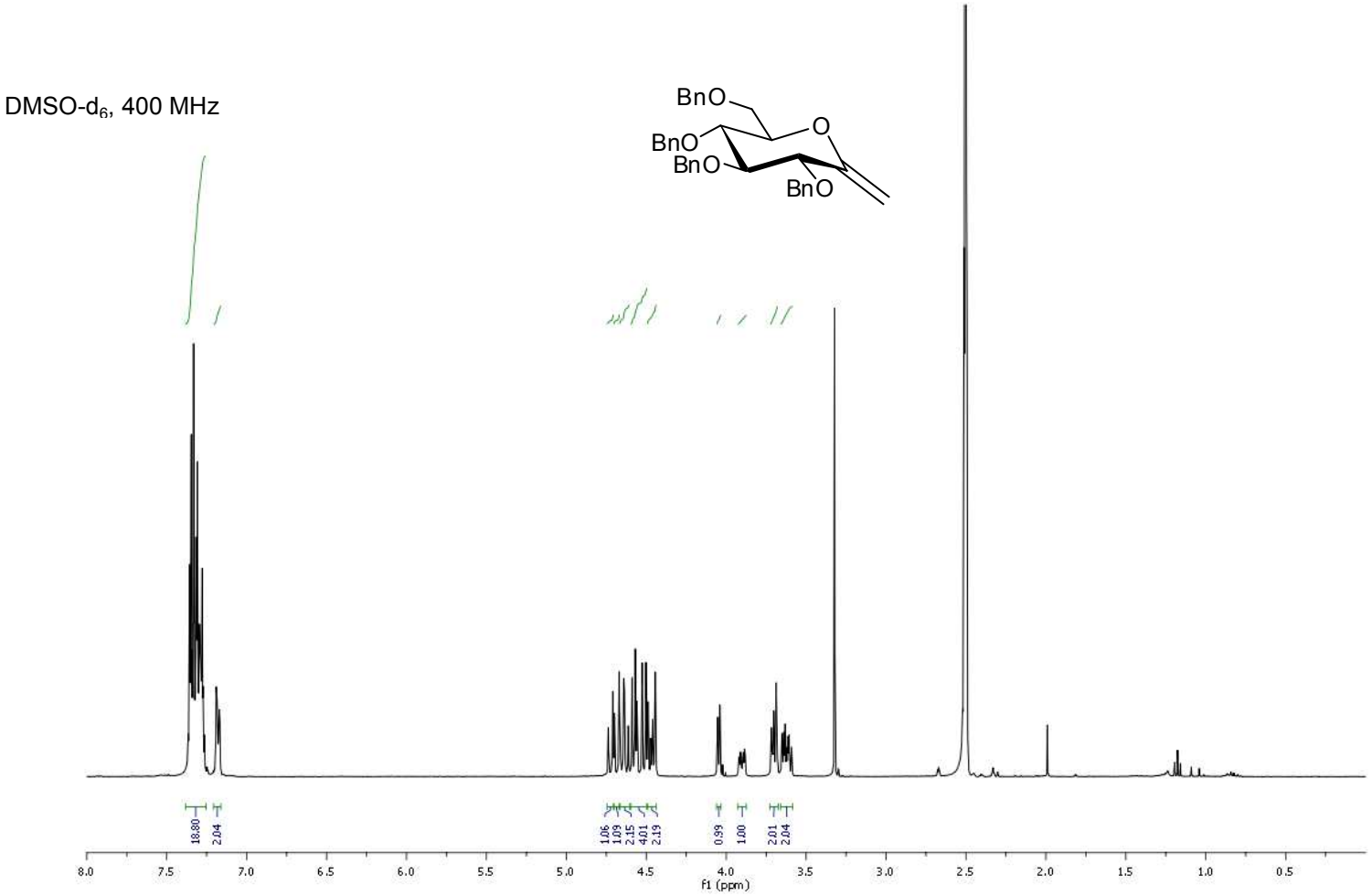


DMSO-d_6, 100 MHz

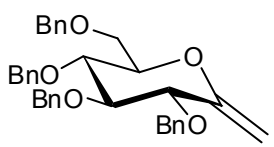


2,6-Anhydro-3,4,5,7-tetra-*O*-benzyl-1-deoxy-D-glucohept-1-enitol (16)

DMSO-d_6, 400 MHz

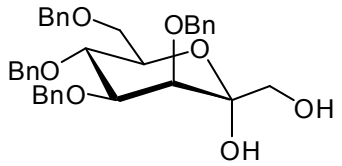


DMSO-d_6, 100 MHz

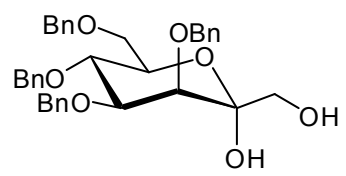


3,4,5,7-Tetra-*O*-benzyl-α-D-*glycero*-D-*lyxo*-hept-2-ulopyranose (17)

DMSO-d_6, 400 MHz

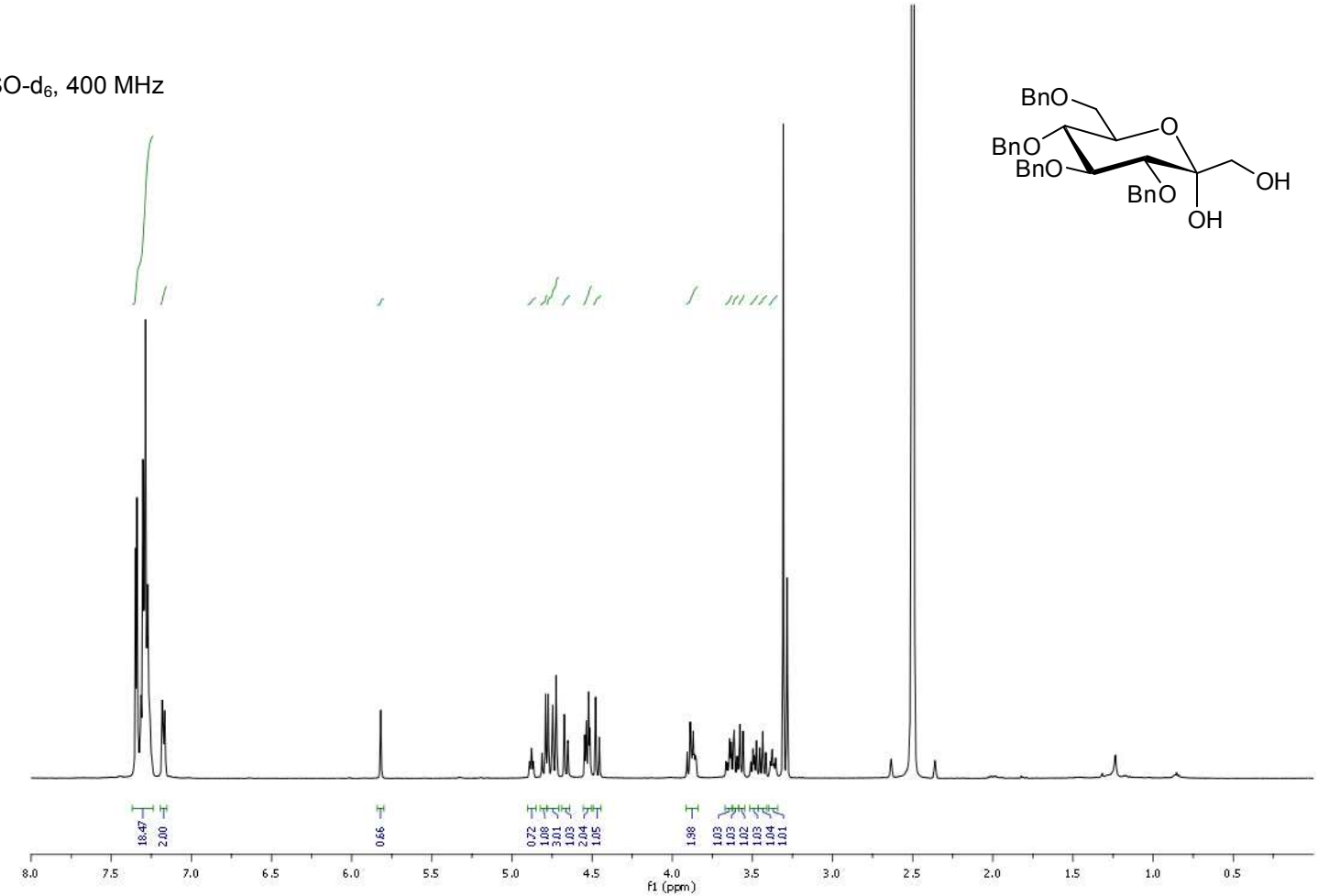


DMSO-d_6, 100 MHz

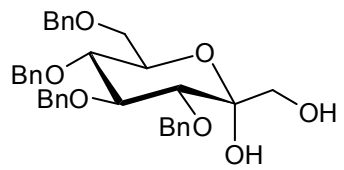


3,4,5,7-Tetra-*O*-benzyl-α-D-*glycero*-D-*xylo*-hept-2-ulopyranose (18)

DMSO-d_6, 400 MHz

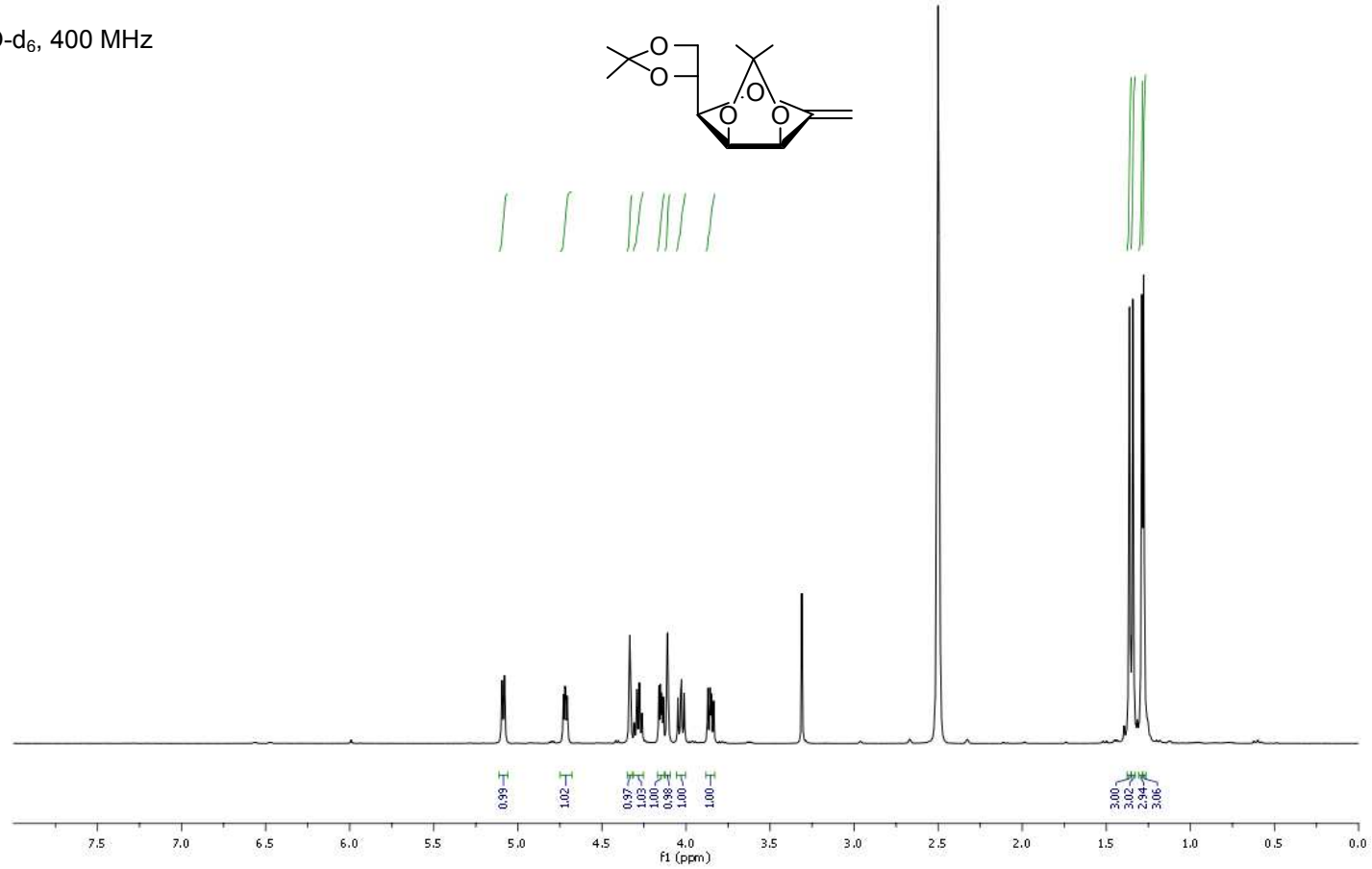


DMSO-d_6, 100 MHz

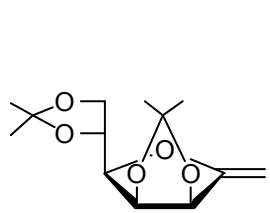


2,5-Anhydro-1-deoxy-3,4:6,7-di-*O*-isopropylidene-D-mannohept-1-enitol (21)

DMSO-d_6, 400 MHz

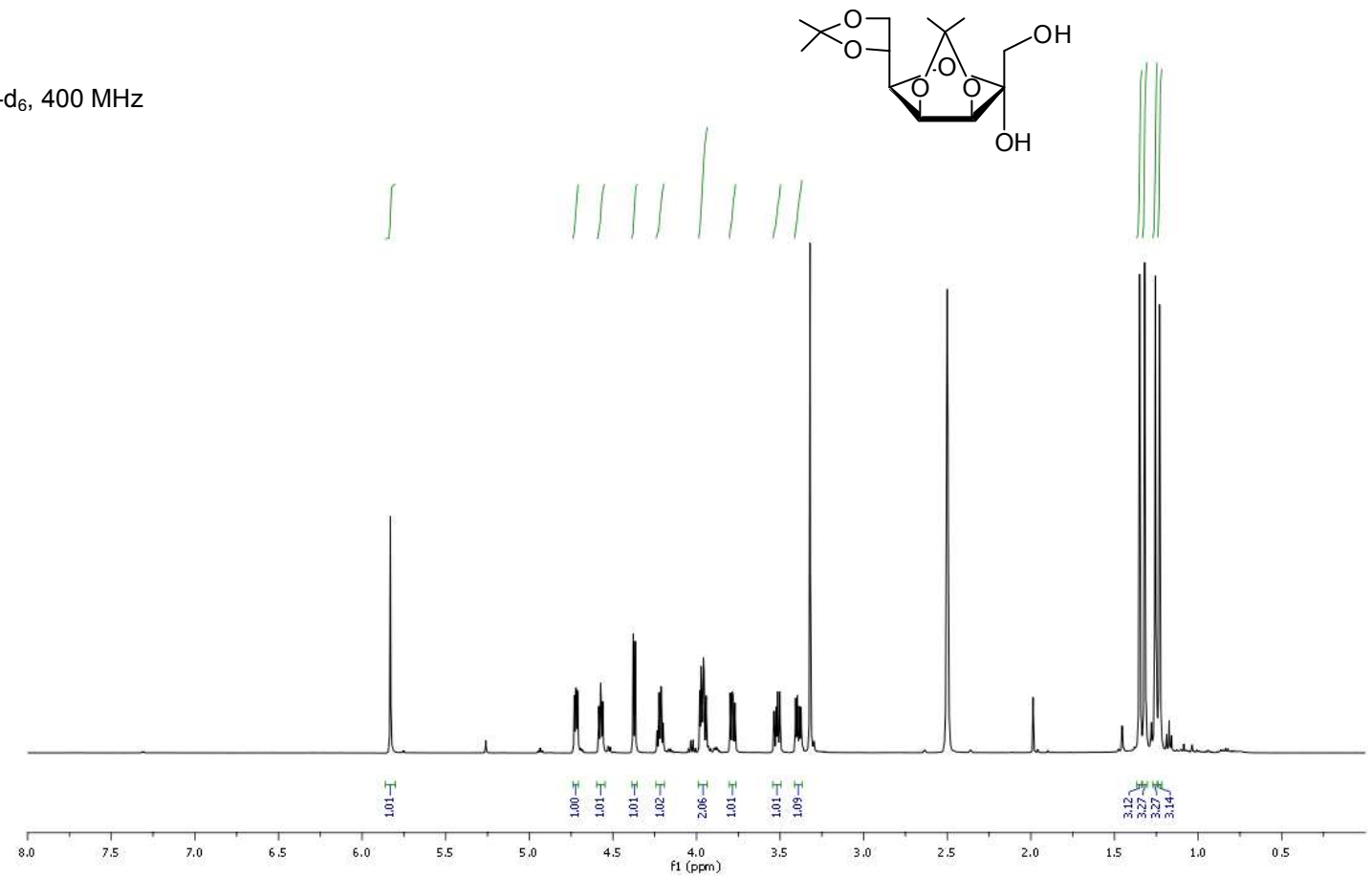


DMSO-d_6, 100 MHz

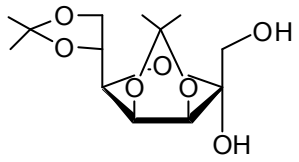


3,4:6,7-Di-*O*-isopropylidene-α-D-*glycero*-D-*lyxo*-hept-2-ulofuranose (22)

DMSO-d_6, 400 MHz

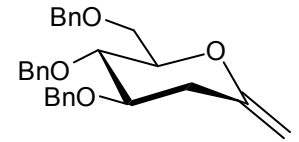


DMSO-d_6, 100 MHz

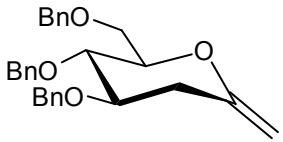


2,6-Anhydro-1,3-dideoxy-4,5,7-tri-*O*-benzyl-D-glucohept-1-enitol (26)

DMSO-d_6, 500 MHz

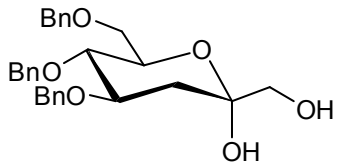


DMSO-d_6, 125 MHz

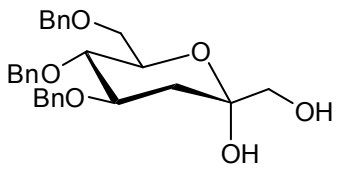


3-Deoxy-4,5,7-tri-*O*-benzyl-α-D-*glycero*-D-*xylo*-hept-2-ulopyranose (27)

DMSO-d_6, 400 MHz

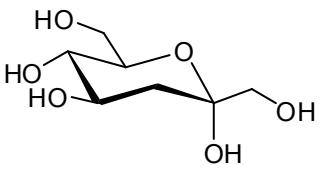


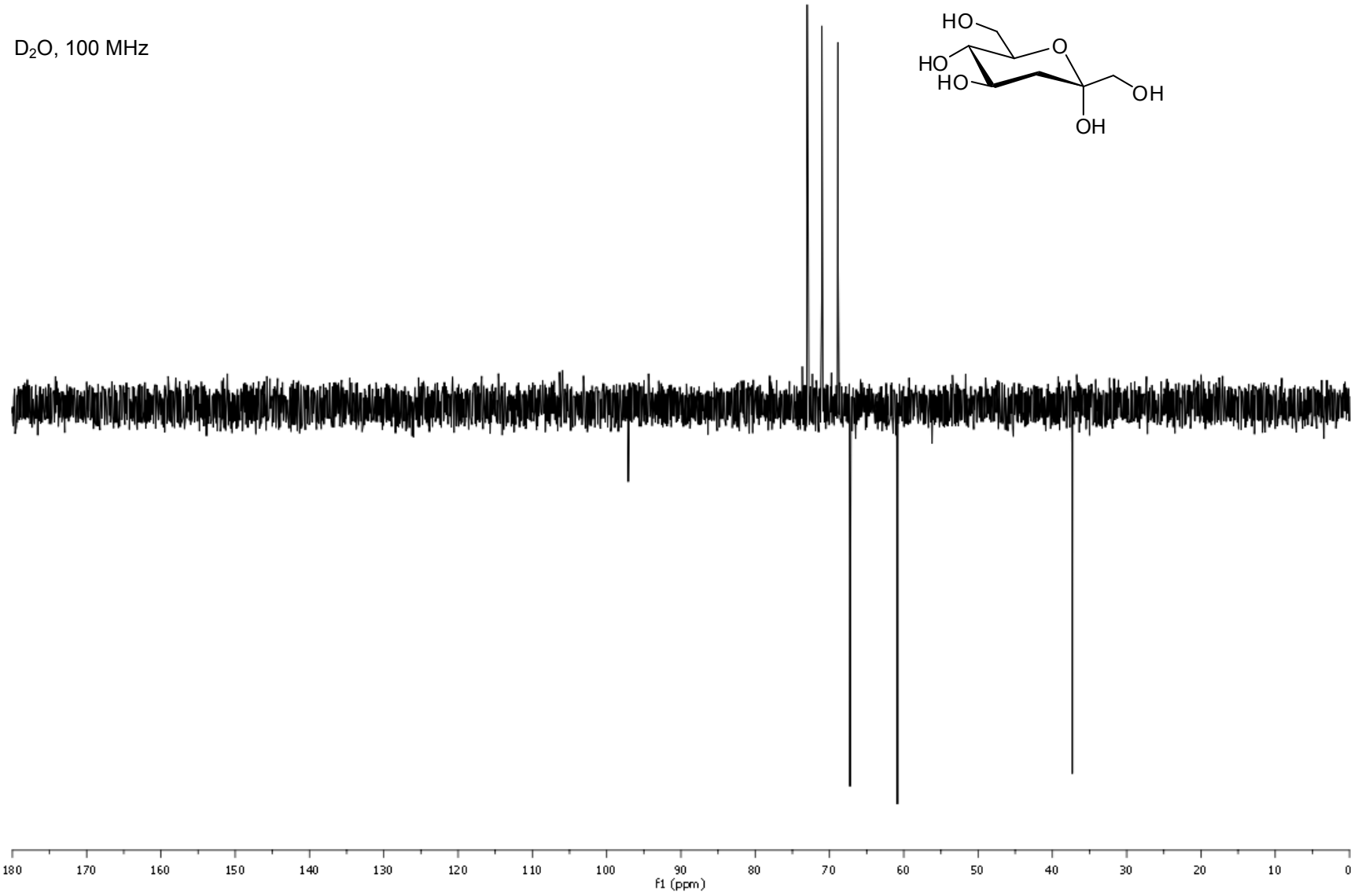
DMSO-d_6, 100 MHz



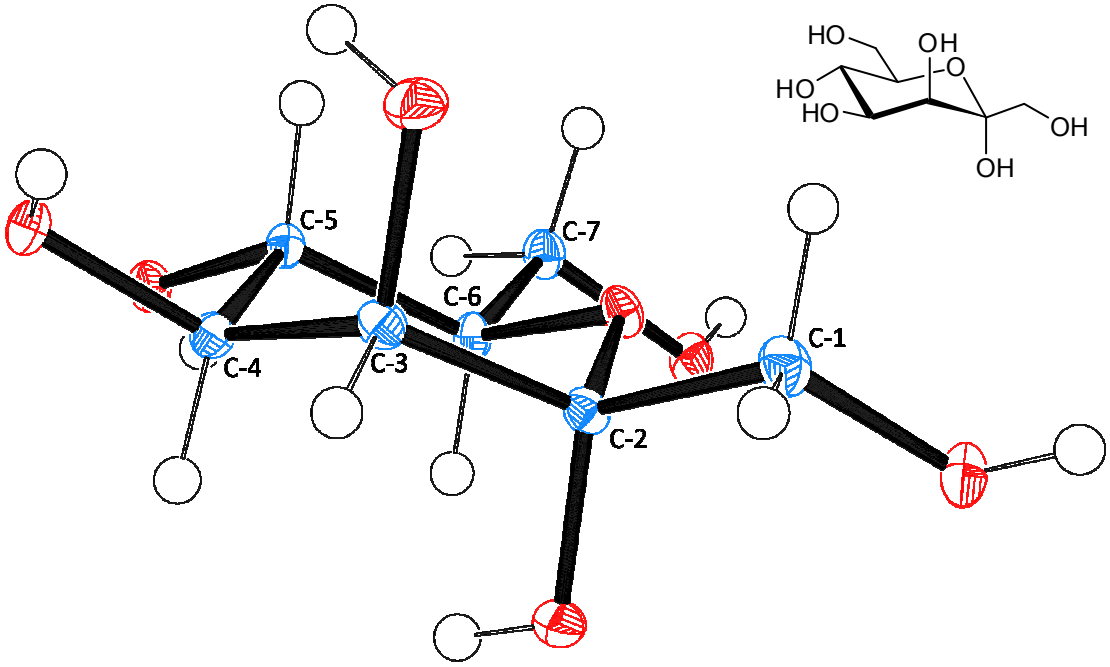
3-Desoxy-α-D-*glycero*-D-*xylo*-hept-2-ulopyranose (3)

D_2O, 400 MHz




D2O, 100 MHz
HO
O
HO
HO
OH
OH
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

Crystal Structure and Data of D-*manno*-Heptulose (1)



Empirical formula	$C_7H_{14}O_7$
Formula weight	210.07 g·mol^{-1}
Temperature	100 K
Space group	P 2_1
Crystal size	0.240·0.430·0.430 mm^3
Lattice constants	a=6.57080(10) Å
	b=6.99590(10) Å
	c=9.4032(2) Å
	V=422.298 Å^3, z 2
R-factor	4.87 %
Density	d=1.635 g·cm^{-3}
Diffractometer	*Bruker* SMART APEX
Radiation	*Mo-K*$_\alpha$ Graphitmonochromator
Scan type	Omega-scan
Measurement range	2° ≤ Θ ≤ 50°
Number of reflexes, measured	8983

List of atoms and atomic coordinates of 1

Label	SYBYL type	X (Å)	Y (Å)	Z (Å)
O2	O.3	0.3253(3)	0.5384(4)	0.4908(2)
O**H**2	H	0.3945	0.6329	0.4726
O6	O.3	0.0999(3)	0.5610(3)	0.2627(2)
O4	O.3	0.6740(3)	0.4979(4)	0.1564(2)
O**H**4	H	0.6766	0.3779	0.1538
O3	O.3	0.3463(4)	0.2595(4)	0.1712(3)
O**H**3	H	0.3706	0.3080	0.0947
O5	O.3	0.4633(3)	0.8630(4)	0.1146(2)
O**H**5	H	0.4077	0.9557	0.1485
O1	O.3	-0.0334(3)	0.3160(4)	0.4728(2)
O**H**1	H	-0.1001	0.2177	0.4866
O7	O.3	-0.1219(3)	0.9047(4)	0.2291(3)
O**H**7	H	-0.2215	0.9691	0.1817
C5	C.3	0.3630(5)	0.6875(5)	0.1431(3)
H5	H	0.3024	0.6215	0.0493
C4	C.3	0.5249(4)	0.5589(5)	0.2380(3)
H4	H	0.5986	0.6335	0.3244
C2	C.3	0.2417(5)	0.4405(5)	0.3606(3)
C3	C.3	0.4244(4)	0.3821(5)	0.2912(3)
H3	H	0.5310	0.3120	0.3645
C1	C.3	0.1145(5)	0.2679(5)	0.3878(3)
H1a	H	0.0406	0.2147	0.2933
H1b	H	0.2095	0.1680	0.4389
C6	C.3	0.1908(5)	0.7343(5)	0.2239(3)
H6	H	0.2524	0.8065	0.3146
C7	C.3	0.0150(4)	0.8501(5)	0.1349(3)
H7a	H	0.0707	0.9653	0.0954
H7b	H	-0.0619	0.7731	0.0524

Bond lengths of 1

Atom 1	Atom 2	Length (Å)	SYBYL-Typ
O2	O**H**2	0.840(3)	1
O2	C2	1.408(4)	1
O6	C2	1.435(4)	1
O6	C6	1.433(4)	1
O4	O**H**4	0.840(3)	1
O4	C4	1.432(4)	1
O3	O**H**3	0.841(3)	1
O3	C3	1.423(4)	1
O5	O**H**5	0.840(3)	1
O5	C5	1.445(4)	1
O1	O**H**1	0.840(3)	1
O1	C1	1.424(4)	1
O7	O**H**7	0.840(2)	1
O7	C7	1.442(4)	1
C5	H5	1.000(3)	1
C5	C4	1.527(4)	1
C5	C6	1.527(5)	1
C4	H4	1.000(3)	1
C4	C3	1.535(5)	1
C2	C3	1.539(5)	1
C2	C1	1.521(5)	1
C3	H3	1.000(3)	1
C1	H1a	0.990(3)	1
C1	H1a	0.990(3)	1
C6	H6	1.000(3)	1
C6	C7	1.509(4)	1
C7	H7a	0.990(3)	1
C7	H7b	0.990(3)	1

References

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