Supporting Information

C-Glycosides of 2-Amino-2-deoxy Sugars. β -*C*-Glycosides and 1,1-Disubstituted Variants

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^1H NMR and ^{13}C NMR were recorded on DELTA400, JNM-GX400 spectrometers. Where spectroscopic assignments have been made, these are based specifically on ^1H - ^1H and ^1H - ^{13}C correlation spectroscopy. Chemical shifts are quoted in parts per million (δ) downfield from internal tetramethylsilane (TMS). Coupling constants (J), expressed in Hertz. TLC was performed using aluminum plates coated with Merck 60H-F₂₅₄ silica gel with detection by UV and by dipping with anisaldehyde solution (338 mL EtOH/ 9.2 g *p*-anisaldehyde/ 3.75 mL AcOH/ 12.5 mL conc. H₂SO₄) and heating. Solvents were evaporated under reduced pressure in Büchi rotary evaporator below 40 °C.

The numbering of carbon atoms (and protons) on carbohydrate derivatives used in this paper is as shown below. In other words we have retained the numbering associated with the carbohydrate core, rather than incorporate the varying numbers of carbons associated with the *C*-glycoside side chains. Where appropriate these side chains were numbered as shown below.

C(1)-Methyl glucal **6**: This compound has been reported previously [Nicolaou, K. C.; Hwang, C.-K.; Duggan, M. E. *J. Chem. Soc., Chem. Commun.* **1986**, 925.]

δ_{H} (400 MHz, CDCl_3) 5.30 (1 H, m, H-3), 5.18 (1 H, dd, J 5.4, 7.3, H-4), 4.65 (1 H, d, J 1.5, H-2), 4.43 (1 H, dd, J 5.8, 12.2, H-6), 4.27 (1 H, m, H-5), 4.16 (1 H, dd, J 3.0, 12.2, H-6), 2.10 (3 H, s), 2.08 (3 H, s), 2.04 (3 H, s), 1.82 (3 H, s, CH_3).

For a detailed experimental procedure that we used for the deprotonation of glycal **4**, see Majumder, U.; Cox, J. M.; Rainier J. D. *Org. Lett.* **2003**, 5, 913.

C(1)-Methyl galactal **7**: δ_{H} (400 MHz, CDCl_3) 5.52 (1 H, br, H-3), 5.40 (1 H, m, H-4), 4.53 (1 H, m, H-2), 4.30 (2 H, m, H-5, H-6), 4.20 (1 H, m, H-6), 2.12 (3 H, s), 2.09 (3 H, s), 2.02 (3 H, s), 1.82 (3 H, dd, J 1.0, 1.5, CH_3); δ_{C} (100 MHz, CDCl_3) 170.6, 170.4, 170.2, 154.9 (C-1), 94.2 (C-2), 72.9 (C-5), 64.8 (C-3), 63.7 (C-4), 61.9 (C-6), 20.9, 20.8, 20.7, 19.6 (CH_3); HRMS m/z (E.S.) Found: $[\text{M}+\text{NH}_4]^+$ 304.1387, $\text{C}_{13}\text{H}_{22}\text{NO}_7$ requires 304.1391.

For a detailed experimental procedure used for the deprotonation of glycal **5**, see Jäkel, C.; Dötz, K.H. *J. Organomet. Chem.* **2001**, 624, 172.

Azidoselenides **8** and **11** were prepared from C-1-methyl 3,4,6-tri-*O*-acetyl glycals **6** and **7** respectively according to the procedure described by Czernecki [Czernecki, S.; Ayadi, E.; Randriamandimby, D. *J. Org. Chem.* **1994**, 59, 8256.] Adduct **8** was obtained as a 1:3 inseparable mixture (as judged by ^1H NMR) of *gluco*: *manno* isomers (82 % yield). *Galacto* adduct **11** was obtained as a oil in 30 % yield. Both **8** and **11** were unstable intermediates were, as a result, not fully characterized. Assignments of signals for *gluco* and *manno* isomers indicated below are based on COSY analysis.

8: ν_{max} / cm^{-1} 2105 (s), 1742 (s); **8-*gluco***: δ_{H} (400 MHz, CDCl_3) 7.61-7.27 (5 H, m, H_{ar}), 5.44 (1 H, t, J 10.0, H-3), 5.06 (1 H, t, J 10.0, H-4), 4.56 (1 H, m, H-5), 4.28 (1 H, dd, J 6.0, 12.2, H-6), 3.97 (1 H, dd, J 3.0, 12.2, H-6), 3.60 (1 H, d, J 10.0, H-2), 2.13-2.06 (9 H, 3s, COCH_3), 1.76 (3 H, s, CH_3); **8-*manno***: δ_{H} (400 MHz, CDCl_3) 7.61-7.27 (5 H, m, H_{ar}), 5.65 (1 H, dd, J 4.0, 10.0, H-3), 5.34 (1 H, t, J 10.0, H-4),

4.49 (1 H, m, H-5), 4.25 (1 H, dd, J 6.0, 12.2, H-6), 4.15 (1 H, d, J 4.0, H-2), 4.12 (1 H, dd, J 3.4, 12.2, H-6), 2.13-2.06 (9 H, 3s, COCH₃), 1.70 (3 H, s, CH₃).

11: δ_{H} (400 MHz, CDCl₃) 7.62-7.58 (2 H, m, H_{ar}), 7.38-7.28 (3 H, m, H_{ar}), 5.51 (1 H, dd, J 1.3, 3.3, H-4), 5.25 (1 H, dd, J 3.3, 10.7, H-3), 4.68 (1 H, ddd, J 1.3, 6.4, 6.4, H-5), 4.07 (2 H, m, 2 $\times$ H-6), 3.77 (1 H, d, J 10.7, H-2), 2.16 (3 H, s), 2.07 (3 H, s), 2.06 (3 H, s), 1.78 (3 H, s, CH₃).

2-N-Acetyl Anomeric Selenides

Reduction and deacetylation of **8** (as a mixture of *gluco* and *manno* isomers) and **9** was carried out according to the procedure described by Czernecki [Czernecki, S.; Ayadi, E.; Randriamandimby, D. *J. Org. Chem.* **1994**, *59*, 8256.]. The *gluco* and *manno* isomers **9** and **10** (derived from **8**) were separable by flash chromatography in 21 % and 70 % yield respectively. The *galacto* isomer **12** was obtained in 90 % yield from **11**.

9: δ_{H} (400 MHz, CDCl₃) 7.56-7.52 (2 H, m, H_{ar}), 7.40-7.28 (3 H, m, H_{ar}), 5.64 (1 H, d, J 10.0, NH), 5.22 (1 H, t, J 9.5, H-3), 5.18 (1 H, t, J 9.5, H-4), 4.56 (1 H, m, H-5), 4.30 (1 H, t, J 9.5, H-2), 4.29 (1 H, dd, J 5.1, 12.5, H-6), 4.10 (1 H, dd, J 2.2, 12.5, H-6), 2.11 (3 H, s), 2.06 (3 H, s), 2.04 (3 H, s), 2.01 (3 H, s), 1.53 (3 H, s, CH₃); δ_{C} (100 MHz, CDCl₃) 171.3, 170.7, 169.8, 169.3, 137.0, 129.2, 129.0, 125.4, 93.3 (C-1), 72.4 (C-3 and C-5), 68.3 (C-4), 62.2 (C-6), 57.8 (C-2), 27.8 (CH₃), 23.4, 20.8, 20.7, 20.7; HRMS m/z (E.S.) Found: [M+NH₄]⁺ 519.1239, C₂₁H₃₁N₂O₈⁸⁰Se requires 519.1240.

10: δ_{H} (400 MHz, CDCl₃) 7.59-7.56 (2 H, m, H_{ar}), 7.40-7.29 (3 H, m, H_{ar}), 5.71 (1 H, d, J 10.3, NH), 5.59 (1 H, dd, J 4.4, 10.2, H-3), 5.10 (1 H, t, J 10.2, H-4), 4.87 (1 H, dd, J 4.4, 10.3, H-2), 4.65 (1 H, m, H-5), 4.32 (1 H, dd, J 6.1, 12.2, H-6), 4.09 (1 H, dd, J 2.5, 12.2, H-6), 2.12 (3 H, s), 2.10 (3 H, s), 2.07 (3 H, s), 1.99 (3 H, s), 1.54 (3 H, s, CH₃); δ_{C} (100 MHz, CDCl₃) 170.5, 170.0, 170.0, 169.9, 136.7, 129.1, 128.8, 126.7, 88.6 (C-1), 73.0 (C-5), 70.6 (C-3), 66.1 (C-4), 62.8 (C-6), 54.5 (C-2), 26.2 (CH₃), 23.2, 20.8, 20.7, 20.7; HRMS m/z (E.S.) Found: [M+NH₄]⁺ 519.1237,

$C_{21}H_{31}N_2O_8^{80}Se$ requires 519.1240.

12: δ_H (400 MHz, $CDCl_3$) 7.60-7.57 (2 H, m, H_{ar}), 7.40-7.28 (3 H, m, H_{ar}), 5.93 (1 H, d, J 9.8, NH), 5.50 (1 H, dd, J 1.5, 3.2, H-4), 5.24 (1 H, dd, J 3.1, 11.5, H-3), 4.80 (1 H, ddd, J 1.5, 6.2, 6.2 H-5), 4.53 (1 H, dd, J 10.0, 11.5, H-2), 4.15-4.04 (2 H, m, 2 H-6), 2.16 (3 H, s), 2.04 (3 H, s), 2.01 (3 H, s), 1.98 (3 H, s), 1.59 (3 H, s, CH_3); δ_C (100 MHz, $CDCl_3$) 170.9, 170.3, 170.2, 170.2, 137.2, 129.0, 128.9, 125.4, 94.7 (C-1), 71.1 (C-5), 69.9 (C-3), 67.2 (C-4), 61.9 (C-6), 53.6 (C-2), 28.0 (CH_3), 23.3, 20.7, 20.7, 20.6; HRMS m/z (E.S.) Found: $[M+H]^+$ 502.0984, $C_{21}H_{28}NO_8^{80}Se$ requires 502.0975.

β -C-Glycosides (13-15)

A solution of freshly distilled tributyltin hydride (80 μ L, 0.3 mmol, 3 eq) and AIBN (16 mg, 0.1 mmol, 1 eq) in benzene (5 mL) was added dropwise over 1.5 h (via syringe pump) to a refluxing solution of anomeric selenide (50 mg, 1eq) in benzene (15 mL) under N_2 . After a further 1.5 h, the reaction mixture was concentrated *in vacuo* and the residue was dissolved in acetonitrile (5 mL) and hexane (1 mL) was added. The acetonitrile layer was separated, washed with hexane (6×5 mL), filtered and concentrated *in vacuo* to give an oil. Purification was carried out by flash chromatography (silica 60H, 1:2, Hex:EtOAc) to give the target β -C-glycoside.

Selenides **9** and **10** gave the corresponding reduction products, β -C-glycosides **13** (56 % isolated yield) and **14** (60 % isolated yield) respectively.

13: m.p. 167-9 °C (iso-PrOH) (lit: m.p. 168-169 °C. Yoshimura, J; Muramatsu, N; Sato, T. *Nippon Kagaku Zasshi*, **1958**, 79, 1503). This compound was previously obtained via the reaction of N-acetyl-1-bromo-D-glucosamine by means of Grignard reagents.) δ_H (400 MHz, $CDCl_3$) 5.51 (1 H, d, J 9.7, NH), 5.08 (1 H, t, J 9.7, H-4), 5.00 (1 H, t, J 9.7, H-3), 4.22 (1 H, dd, J 5.8, 12.2, H-6), 4.11 (1 H, dd, J 2.0, 12.2, H-6), 3.99 (1 H, ddd, J 9.7, 9.7, 9.7, H-2), 3.61 (1 H, m, H-5), 3.43 (1 H, qd, J 6.4, 9.7, H-1), 2.10 (3 H, s), 2.04 (3 H, s), 2.04 (3 H, s), 1.96 (3 H, s), 1.28 (3 H, d, J 6.4, CH_3); δ_C (100 MHz, $CDCl_3$) 171.7, 170.7, 170.0, 169.3, 75.9 (C-1), 75.6 (C-5), 74.4 (C-3), 68.4 (C-4), 62.5 (C-6), 55.1 (C-2), 23.7, 20.8, 20.7, 20.6, 17.9 (CH_3); HRMS m/z

(E.S.) Found: $[M+NH_4]^+$ 363.1762, $C_{15}H_{27}N_2O_8$ requires 363.1762.

14: Isolated as a colourless foam. δ_H (400 MHz, $CDCl_3$) 5.72 (1 H, d, J 9.8, NH), 5.06 (1 H, t, J 10.0, H-4), 5.00 (1 H, dd, J 3.9, 10.0, H-3), 4.53 (1 H, ddd, J 1.5, 3.9, 9.8, H-2), 4.23 (1 H, dd, J 5.4, 12.2, H-6), 4.05 (1 H, dd, J 2.4, 12.2, H-6), 3.82 (1 H, dq, J 1.5, 6.4, 6.4, 6.4, H-1), 3.62 (1 H, m, H-5), 2.11 (3 H, s), 2.09 (3 H, s), 2.05 (3 H, s), 2.00 (3 H, s), 1.21 (3 H, d, J 6.4, CH_3); δ_C (100 MHz, $CDCl_3$) 170.6, 170.6, 170.4, 170.0, 76.7 (C-5), 73.8 (C-1), 73.1 (C-3), 66.0 (C-4), 63.0 (C-6), 54.0 (C-2), 23.3, 20.8, 20.7, 20.7, 16.7 (CH_3); HRMS m/z (E.S.) Found: $[M+H]^+$ 346.1500, $C_{15}H_{24}NO_8$ requires 346.1496.

β -C-glycoside **15** was obtained as a major byproduct in 43 % yield when the reaction of **12** with styrene was carried out (see below). We did not conduct a separate reduction of **12** to give **15**.

15: Isolated as a colourless foam. δ_H (400 MHz, $CDCl_3$) 5.47 (1 H, br, NH), 5.36 (1 H, dd, J 10.0, 3.2, H-4), 4.96 (1 H, dd, J 3.2, 10.5, H-3), 4.16 (1 H, q, J 10.5, 10.5, 10.5, H-2), 4.11- 4.07, (2 H, m, H-6), 3.83 (1 H, m, H-5), 3.42 (1 H, dq, J 6.1, 6.1, 6.1, 10.5, H-1), 2.16 (3 H, s), 2.05 (3 H, s), 2.01 (3 H, s), 1.97 (3 H, s), 1.30 (3 H, d, J 6.1, CH_3); δ_C (100 MHz, $CDCl_3$) 171.2, 170.5, 170.4, 170.2, 76.3 (C-1), 74.0 (C-5), 71.8 (C-3), 67.2 (C-4), 62.0 (C-6), 51.4 (C-2), 23.3, 20.7, 20.7, 20.7, 18.0 (CH_3); HRMS m/z (E.S.) Found: $[M+NH_4]^+$ 363.1761, $C_{15}H_{27}N_2O_8$ requires 363.1762.

1,1-Disubstituted C-glycoside Variants (16-20)

We used the same procedure for anomeric radical generation and alkene trapping reaction as described earlier [Grant, L.; Liu, Y.; Walsh, K. E.; Walter, D. S. and Gallagher, T. *Org. Lett.* **2002**, *4*, 4623].

Using selenide **9** as a radical precursor, trapping with *t*-butyl acrylate gave 1,1-disubstituted C-glycoside **16**, which was isolated in 79 % yield as a colourless foam.

16: δ_H (400 MHz, $CDCl_3$) 5.61 (1 H, d, J 10.0, NH), 5.17 (1 H, dd, J 9.5, 10.5, H-3), 5.05 (1 H, t, J 9.5, H-4), 4.24 (1 H, t, J 10.5, H-2), 4.14 (1 H, dd, J 5.1, 12.0, H-6),

4.08 (1 H, dd, J 2.0, 12.0, H-6), 3.65 (1 H, m, H-5), 2.34-2.24 (2 H, m, $\text{CH}_2\text{CH}_2\text{COO}^t\text{Bu}$), 2.23-2.12 (1 H, m, $\text{CH}_2\text{CH}_2\text{COO}^t\text{Bu}$), 2.10 (3 H, s), 2.03 (3 H, s), 2.02 (3 H, s), 1.96 (3 H, s), 1.74-1.66 (1 H, m, $\text{CH}_2\text{CH}_2\text{COO}^t\text{Bu}$), 1.46 (9 H, s, ^tBu), 1.20 (3 H, s, CH_3); δ_{C} (100 MHz, CDCl_3) 172.7, 171.8, 170.8, 170.0, 169.3, 80.9 ($\text{C}(\text{CH}_3)_3$), 77.4 (C-1), 72.0 (C-3), 69.5 (C-5), 69.2 (C-4), 63.0 (C-6), 57.2 (C-2), 28.1 (^tBu), 27.9 (CH_2), 24.3 (CH_2), 24.2 (CH_3), 23.2, 20.6, 20.6, 20.5; HRMS m/z (E.S.) Found: $[\text{M}+\text{H}]^+$ 474.2332, $\text{C}_{22}\text{H}_{36}\text{NO}_{10}$ requires 474.2334.

Using selenide **10** as a radical precursor, trapping with *t*-butyl acrylate gave 1,1-disubstituted *C*-glycoside **17**, which was isolated in 48 % yield as a colourless foam.

17: δ_{H} (400 MHz, CDCl_3) 5.70 (1 H, d, J 10.0, NH), 5.35 (1 H, dd, J 4.2, 10.2, H-3), 5.03 (1 H, t, J 10.2, H-4), 4.45 (1 H, dd, J 4.2, 10.0, H-2), 4.18 (1 H, dd, J 5.4, 12.0, H-6), 4.03 (1 H, dd, J 2.0, 12.0, H-6), 3.81 (1 H, m, H-5), 2.43-2.20 (2 H, m, $\text{CH}_2\text{CH}_2\text{COO}^t\text{Bu}$), 2.44-2.30 (1 H, m, $\text{CH}_2\text{CH}_2\text{COO}^t\text{Bu}$), 1.97-1.87 (1 H, m, $\text{CH}_2\text{CH}_2\text{COO}^t\text{Bu}$), 2.11 (3 H, s), 2.08 (3 H, s), 2.06 (3 H, s), 1.98 (3 H, s), 1.46 (9 H, s, ^tBu), 1.14 (3 H, s, CH_3); δ_{C} (100 MHz, CDCl_3) 172.0, 170.5, 170.2, 170.1, 170.0, 80.9 ($\text{C}(\text{CH}_3)_3$), 77.1 (C-1), 70.0 (C-5, C-3), 66.1 (C-4), 63.2 (C-6), 52.6 (C-2), 29.0 (CH_2), 28.4 (CH_2), 28.0 (^tBu), 23.3, 22.5 (CH_3), 20.7, 20.7, 20.7; HRMS m/z (C.I.) Found: $[\text{M}+\text{H}]^+$ 474.2330, $\text{C}_{22}\text{H}_{36}\text{NO}_{10}$ requires 474.2334.

Using selenide **12** as a radical precursor, trapping with *t*-butyl acrylate gave 1,1-disubstituted *C*-glycoside **18**, which was isolated in 50 % yield as a colourless foam.

18: δ_{H} (400 MHz, CDCl_3) 5.45 (1 H, d, J 10.3, NH), 5.34 (1 H, dd, J 1.0, 3.4, H-4), 5.13 (1 H, dd, J 3.4, 11.5, H-3), 4.44 (1 H, dd, J 10.2, 11.5, H-2), 4.12 (1 H, dd, J 6.0, 11.2, H-6), 4.02 (1 H, dd, J 6.8, 11.2, H-6), 3.84 (1 H, ddd, J 1.0, 6.8, 6.8, H-5), 2.37-2.22 (2 H, m, $\text{CH}_2\text{CH}_2\text{COO}^t\text{Bu}$), 2.16 (3 H, s), 2.17-2.07 (1 H, m, $\text{CH}_2\text{CH}_2\text{COO}^t\text{Bu}$), 2.05 (3 H, s), 2.00 (3 H, s), 1.96 (3 H, s), 1.74-1.61 (1 H, m, $\text{CH}_2\text{CH}_2\text{COO}^t\text{Bu}$), 1.46 (9 H, s, ^tBu), 1.24 (3 H, s, CH_3); δ_{C} (100 MHz, CDCl_3) 172.9, 171.3, 170.5, 170.4, 170.2, 80.9 ($\text{C}(\text{CH}_3)_3$), 77.7 (C-1), 68.9 (C-3), 68.2 (C-5), 67.4

(C-4), 62.6 (C-6), 53.1 (C-2), 28.1 (^tBu), 28.1 (CH₂), 24.5 (CH₃), 24.1 (CH₂), 23.4, 20.8, 20.7, 20.7; HRMS *m/z* (E.S.) Found: [M+H]⁺ 474.2335, C₂₂H₃₆O₁₀N requires 474.2334.

Using selenide **12** as a radical precursor, trapping with styrene, 1,1-disubstituted C-glycoside **19** was isolated in 29 % yield as a colourless foam. The reduction product **15** was obtained in 43 % yield from the same reaction as a colourless foam, data above. **19**: δ_H (400 MHz, CDCl₃) 7.36-7.18 (5H, m, H_{ar}), 5.35 (1 H, dd, *J* 1.0, 3.5, H-4), 5.26 (1 H, d, *J* 10.2, *NH*), 5.07 (1 H, dd, *J* 3.5, 11.5, H-3), 4.45 (1 H, dd, *J* 10.3, 11.5, H-2), 4.15 (1 H, dd, *J* 6.0, 12.0, H-6), 4.08 (1 H, dd, *J* 6.8, 12.0, H-6), 3.94 (1 H, ddd, *J* 1.0, 6.8, 6.8, H-5), 2.74-2.55 (2 H, m, CH₂CH₂Ph), 2.18 (3 H, s), 2.10-2.00 (1 H, m, CH₂CH₂Ph), 2.07 (3 H, s), 1.98 (3 H, s), 1.93 (3 H, s), 1.70-1.60 (1 H, m, CH₂CH₂Ph), 1.37 (3 H, s, CH₃); δ_C (100 MHz, CDCl₃) 171.3, 170.6, 170.5, 170.1, 141.8, 128.7, 128.3, 126.3, 78.1 (C-1), 68.9 (C-5), 68.3 (C-3), 67.5 (C-4), 62.8 (C-6), 53.3 (C-2), 32.2 (CH₂), 28.4 (CH₂), 24.6 (CH₃), 23.4, 20.8, 20.7, 20.7; HRMS *m/z* (E.S.) Found: [M+H]⁺ 450.2123, C₂₃H₃₂NO₈ requires 450.2122.

A solution of **10** (43 mg, 0.086 mmol), allyltributyltin (94 μL, 0.30 mmol), and AIBN (7 mg, 0.043 mmol) in dry benzene (1.5 mL) was degassed for 10 min and the mixture was then refluxed under Ar for 12 h, after which time the starting material had been consumed. The solvent was evaporated and the residue was dissolved in MeCN. The solution was then washed three times with hexane, evaporated and the residue was purified by flash chromatography (silica 60H, 1:2, Hex:EtOAc) which gave **20** (17 mg, 51 %) as a colorless foam. δ_H (400 MHz, CDCl₃) 5.77 (1 H, dddd, *J* 6.6, 7.6, 10.3, 17.1, H-2'), 5.70 (1 H, d, *J* 10.0, *NH*), 5.38 (1 H, dd, *J* 4.4, 10.3, H-3), 5.27-5.20 (2 H, m, H-3'), 5.03 (1 H, t, *J* 10.2, H-4), 4.45 (1 H, dd, *J* 4.4, 10.1, H-2), 4.18 (1 H, dd, *J* 5.4, 12.2, H-6), 4.05 (1 H, dd, *J* 2.4, 12.2, H-6), 3.87 (1 H, ddd, *J* 2.4, 5.4, 10.3, H-5), 2.70 (1 H, dd, *J* 6.6, 14.5, H-1'), 2.48 (1 H, dd, *J* 7.6, 14.5, H-1'), 2.11 (3 H, s), 2.08 (3 H, s), 2.06 (3 H, s), 1.99 (3 H, s), 1.19 (3 H, s, CH₃); δ_C (100 MHz, CDCl₃) 170.6, 170.4, 170.2, 170.0, 131.1 (C-2'), 119.8 (C-3'), 76.7 (C-1), 70.0 (C-3, C-5), 66.3 (C-4), 63.3 (C-6), 52.3 (C-2), 38.3 (C-1'), 23.3, 23.2 (CH₃), 20.8, 20.8,

20.8; HRMS m/z (E.S.) Found: $[M+H]^+$ 386.1808, $C_{18}H_{28}NO_8$ requires 386.1809.