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SUPPLEMENTARY MATERIAL TO

Synthesis of 1,3-divalent glycoconjugates with diverse structures and their functionalization

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CHARACTERISATION DATA OF THE PREPARED COMPOUNDS

2,4,6-Tri-O-*acetyl-3*-O-*propargyl*-β-*D-glucopyranosyl azide* (***5***). Yield: 90 %; physical state: syrup; $[\alpha]_D$ (*c*: 0.4, MeOH, 25 °C): +5.6°; ^{1}H-NMR (500 MHz, $CDCl_3$, δ / ppm): 5.04 (1H, *t*, *J* = 10.0 Hz, H-4), 4.93 (1H, *t*, *J* = 9.0 Hz, H-2), 4.52 (1H, *d*, *J* = 9.0 Hz, H-1), 4.25–4.14 (4H, *m*, CH_2–C, H-6a & H-6b), 3.80 (1H, *t*, *J* = 9.5 Hz, H-3), 3.73–3.72 (1H, *m*, H-5), 2.46 (1H, *t*, *J* = 2.0 Hz, alkyne C–H), 2.14, 2.11 & 2.10 (9H, 3*s*, 3×$COCH_3$); ^{13}C-NMR (125 MHz, $CDCl_3$, δ / ppm): 170.7, 169.2, 169.0 (3× **C**OCH_3), 87.9, 79.2, 78.8, 74.9, 74.2, 71.9, 69.0, 61.9, 59.4, 20.8, 20.7; ESI-MS HRMS Calcd. for $C_{15}H_{19}N_3O_8Na$: 392.1070. Found: 392.1050 for $[M+Na]^+$.

Dodecyl 2,4,6-tri-O-*acetyl-3*-O-*propargyl*-α-*D-glucopyranoside* (***6***). Yield: 80 %, physical state: syrup, $[\alpha]_D$ (*c*: 0.2, $CHCl_3$, 25 °C): +42.7°, ^{1}H-NMR (400 MHz, $CDCl_3$, δ / ppm): 5.06 (1H, *d*, *J* = 3.6 Hz, H-1), 4.99 (1H, *t*, *J* = 10.0 Hz, H-4), 4.77–4.74 (1H, *m*, H-2), 4.32–4.28 (2H, *m*, CH_2–C), 4.24–4.19 (1H, *m*, H-6a), 4.09–4.05 (1H, *m*, H-6b), 4.00 (1H, *t*, *J* = 9.6 Hz, H-3), 3.95–3.91 (1H, *m*, H-5), 3.65–3.62 (2H, *m*, *O*-CH_2), 2.43 (1H, *t*, *J* = 2.4 Hz, alkyne C–H), 2.11(×2) & 2.08 (9H, 3*s*, 3×$COCH_3$), 1.61–1.54 (2H, *m*, *O*-CH_2–C**H**$_2$), 1.30–1.26 (18H, *bs*, 9×CH_2), 0.88 (3H, *t*, *J* = 6.4 Hz, CH_2C**H**$_3$); ^{13}C-NMR (100 MHz, $CDCl_3$, δ / ppm): 170.7, 170.0, 169.5 (3×**C**OCH_3), 95.6, 79.8, 76.5, 74.2, 73.5, 69.2, 60.5, 67.5, 63.0, 62.3, 59.7, 32.7, 31.9, 31.8, 29.7, 29.6, 29.4, 29.3, 29.2, 29.0, 26.0, 25.7, 22.7, 22.6, 20.9, 20.7, 14.0; ESI-MS HRMS: Calcd. for $C_{27}H_{44}O_9Na$: 535.2883. Found: 535.2864 for $[M+Na]^+$.

Methyl 2,4,6-tri-O-*acetyl-3*-O-*propargyl*-α-*D-glucopyranoside* (***7***). Yield: 90 %, physical state: solid, m.p.: 68–72 °C; $[\alpha]_D$ (*c*: 0.8, $CHCl_3$, 25 °C): +99.1°; ^{1}H-

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NMR (500 MHz, $CDCl_3$, δ / ppm): 5.00 (1H, *t*, *J* = 10.0 Hz, H-4), 4.95 (1H, *d*, *J* = 9.0 Hz, H-1), 4.82–4.80 (1H, *m*, H-2), 4.32–4.27 (2H, *m*, CH_2–C), 4.24–4.20 (1H, *m*, H-6a), 4.11–4.08 (1H, *m*, H-6b), 4.02 (1H, *t*, *J* = 9.5 Hz, H-3), 3.92–3.88 (1H, *m*, H-5), 3.39 (3H, *s*, OCH_3), 2.43 (1H, *t*, *J* = 2.0 Hz, alkyne C–H), 2.14, 2.10 & 2.09 (9H, 3*s*, 3×$COCH_3$); ^{13}C-NMR (125 MHz, $CDCl_3$, δ / ppm): 170.8, 170.1, 169.5 (3×**C**OCH_3), 96.9, 79.8, 76.3, 74.4, 73.3, 69.4, 67.6, 62.3, 59.8, 55.4, 21.0, 20.8; ESI-MS HRMS: Calcd. for $C_{16}H_{22}O_9Na$: 381.1162. Found: 381.1173 for $[M+Na]^+$.

Propargyl 2-acetamido-3,5,6-tri-O-*acetyl-2-deoxy*-β-*D-glucofuranoside* ***(10)***. Yield: 70 %; physical state: white solid; m.p.: 121–123 °C; $[\alpha]_D$ (*c*: 0.3, $CHCl_3$, 25 °C): +87.9°; ^{1}H-NMR (400 MHz, $CDCl_3$, δ / ppm): 5.87 (1H, *d*, *J* = 8.8 Hz, NH), 5.09–5.03 (2H, *m*, H-1 & H-4), 4.37–4.17 (6H, *m*, 2×CH_2–C, H-2 & H-6a), 4.11–4.06 (1H, *m*, H-6b), 3.95–3.91 (1H, *m*, H-5), 3.85 (1H, *t*, *J* = 9.6 Hz, H-3), 2.51–2.49 (1H, *m*, alkyne C–H), 2.09 & 2.03 (9H, 2*s*, 3×$COCH_3$); ^{13}C-NMR (100 MHz, $CDCl_3$, δ / ppm): 170.9, 170.1, 169.3 (3x**C**OCH_3), 96.6, 80.3, 78.5, 76.4, 75.4, 74.8, 70.1, 68.6, 62.1, 59.1, 55.2, 51.9, 23.4, 21.0, 20.8; ESI-MS HRMS: Calcd. for $C_{18}H_{24}NO_8$: 382.1502. Found: 382.1500 for $[M+H]^+$.

Decyl 2-acetamido-3,5,6-tri-O-*acetyl-2-deoxy*-β-*D-glucofuranoside* ***(11)***. Yield: 70 %; physical state: syrup; $[\alpha]_D$ (*c*: 0.4, $CHCl_3$, 25 °C): +64.9°; ^{1}H NMR (500 MHz, $CDCl_3$, δ / ppm): 5.77 (1H, *d*, *J* = 9.0 Hz, NH), 5.03 (1H, *t*, *J* = 9.0 Hz, H-4), 4.85 (1H, *d*, *J* = 3.0 Hz, H-1), 4.33–4.16 (4H, *m*, CH_2–C, H-2 & H-6a), 4.08–4.06 (1H, *m*, H-6b), 3.89–3.86 (1H, *m*, H-5), 3.83 (1H, *t*, *J* = 9.5 Hz, H-3), 3.68–3.65 (1H, *m*, *O*-C**H**$_2$–CH_2), 3.47–3.42 (1H, *m*, *O*-C**H**$_2$–CH_2), 2.50 (1H, *t*, *J* = 2.0 Hz, alkyne C–H), 2.09, 2.08 & 2.02 (9H, 3*s*, 3×$COCH_3$), 1.62–1.59 (2H, *m*, *O*-CH_2–C**H**$_2$), 1.32–1.26 (14H, *m*, 7×CH_2), 0.88 (3H, *t*, *J* = 6.8 Hz, CH_3); ^{13}C-NMR (125 MHz, $CDCl_3$, δ / ppm): 170.8, 169.9, 169.4 (3×**C**OCH_3), 97.4, 80.4, 76.8, 74.6, 70.3, 68.4, 68.1, 62.4, 59.0, 52.2, 31.9, 29.6 (×2), 29.5, 29.4, 29.3, 26.2, 23.4, 22.7, 20.9, 20.8, 14.1; ESI-MS HRMS: Calcd. for $C_{25}H_{42}NO_8$: 484.2910. Found: 484.2925 for $[M+H]^+$.

Decyl 2-acetamido-3-O-*decyl-2-deoxy-5,6*-O-*isopropylidene*-β-*D-glucofuranoside* ***(13)***. Yield: 50 %; physical state: syrup; $[\alpha]_D$ (*c*: 1.2, $CHCl_3$, 25 °C): –34.4°, ^{1}H-NMR (500 MHz, $CDCl_3$, δ / ppm): 5.52 (*d*, 1H, *J* = 7.0 Hz, NH), 4.88 (1H, *s*, H-1), 4.36–4.32 (2H, *m*, H-4 & H-5), 4.30–4.29 (1H, *m*, H-2), 4.03—4.01 (2H, *m*, H-6a & H-6b), 3.80 (1H, *d*, *J* = 4.0 Hz, H-3), 3.71–3.65 (2H, *m*, *O*-CH_2), 3.44–3.36 (2H, *m*, *O*-CH_2), 1.98 (3H, *s*, $COCH_3$), 1.57–1.51 (4H, *m*, *O*-CH_2–C**H**$_2$), 1.43 & 1.35 (6H, 2*s*, 2×CH_3), 1.29 (28H, *s*, 14×CH_2), 0.88 (6H, *t*, *J* = 6.5 Hz, 2×CH_3); ^{13}C-NMR (125 MHz, $CDCl_3$, δ / ppm): 169.6 (**C**OCH_3), 108.4, 107.1, 82.5, 82.4, 74.6, 70.1, 68.6, 66.4, 59.5, 32.0, 29.9, 29.8, 29.7 (×2), 29.6 (×2), 29.5, 26.7, 26.2, 25.4, 23.4, 22.8, 14.2; ESI-MS HRMS: Calcd. for $C_{31}H_{59}NO_6Na$: 564.4240. Found: 564.4221 for $[M+Na]^+$.

Propargyl 2-acetamido-4,6-di-O-*acetyl-3*-O-*decyl-2-deoxy*-β-*D-glucopyranoside (**14**)*. Yield: 60 %; physical state: syrup; $[\alpha]_D$ (*c*: 1.0, $CHCl_3$, 25 °C): –34.3°, ^{1}H-NMR (500 MHz, $CDCl_3$, δ / ppm): 5.94 (1H, *d*, *J* = 7.5 Hz, NH), 5.17 (1H, *d*, *J* = 8.0 Hz, H-1) 4.95 (1H, *t*, *J* = 9.5 Hz, H-4), 4.41–4.30 (2H, *m*, OCH_2), 4.27–4.19 (1H, *m*, H-6a), 4.10–4.07 (1H, *m*, H-6b), 3.69–3.66 (1H, *m*, H-5), 3.59–3.49 (2H, *m*, *O*-CH_2), 3.27–3.24 (1H, *m*, H-2), 2.46 (1H, *t*, *J* = 2.0 Hz, alkyne CH), 2.07 and 2.00 (9H, 2*s*, 3×$COCH_3$), 1.46–1.43 (2H, *m*, *O*-CH_2–C**H**$_2$), 1.24 (14H, *s*, 7×CH_2), 0.87 (3H, *t*, *J* = 6.5 Hz, CH_3); ^{13}C-NMR (125 MHz, $CDCl_3$, δ / ppm): 170.9, 170.8, 169.6 (**C**OCH_3), 97.6, 78.7, 75.1, 72.4, 72.0, 70.5, 62.4, 57.7, 56.2, 32.0, 31.9, 30.4, 29.8, 29.7, 29.6, 29.5, 29.4 (×2), 26.1, 23.7, 22.7, 21.0, 14.2; ESI-MS HRMS: Calcd. for $C_{25}H_{42}NO_8$: 484.2910. Found: 484.2921 for $[M+H]^+$.