

Additions and Corrections

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Synthesis of Fish Antifreeze Neoglycopeptides Using Microwave-Assisted “Click Chemistry”.

Pages 2409–2412. Following publication, it was pointed out¹ to the authors that the NMR data originally assigned to compound **1** (Scheme 2, page 2410) were in fact consistent with a furanose. Accordingly, correct structures are depicted in the revised Figure 1, Scheme 2, and Scheme 3 depicted below. For completeness, the authors embarked on a synthesis of the desired galactopyranoside **7** and the Tn antigen mimic **8** using an alternative route, as outlined in Scheme 5. The NMR data are given below.

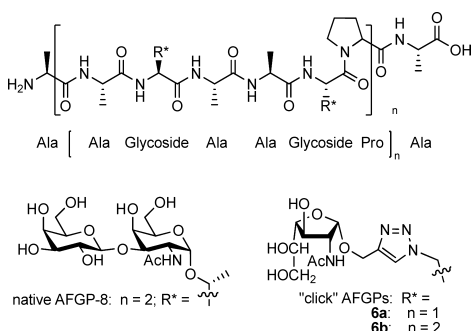
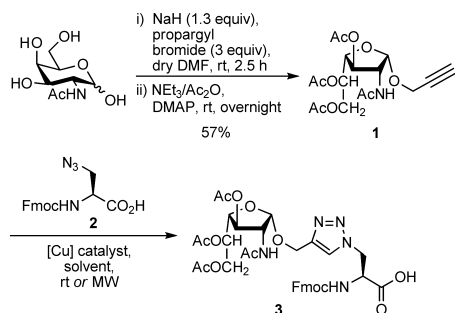
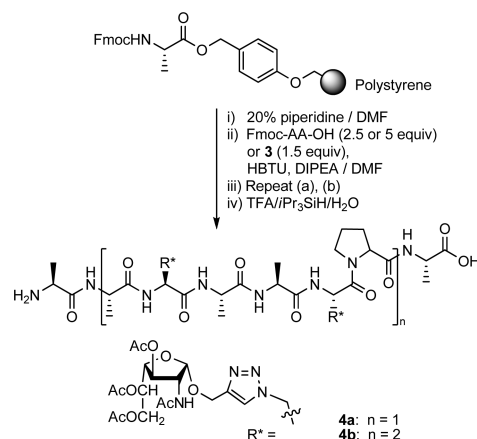


Figure 1. Structure of native AFGP-8 and neoglycopeptides (click analogues).

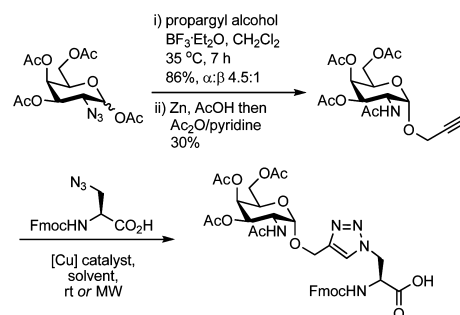
Scheme 2. Synthesis of Neoglycosyl Amino Acid Building Block



Scheme 3. Fmoc-SPPS of **4a** and **4b**



Scheme 5. Synthesis of Neoglycosyl Amino Acid Building Block **8**



The synthesis of 1,3,4,6-tetra-*O*-acetyl-2-azido-2-deoxy-*D*-galactose was performed according to the literature.² Modification of the glycosylation conditions used by Brimble et al.³ yielded 2-acetamido-3,4,6-tri-*O*-acetyl-2-deoxy- α -*D*-galactopyranoside (**7**). CuAAC between **7** and azidoalanine under the same conditions applied for the furanose form afforded the galactopyranoside-glycosylated analogue **8** of the Tn antigen.

Propargyl 2-acetamido-3,4,6-tri-*O*-acetyl-2-deoxy- α -*D*-galactopyranoside (7**).** ¹H NMR (400 MHz, CDCl₃) δ 5.74 (1 H, d, $J = 9.7$ Hz, NH), 5.35 (1 H, dd, $J = 3.2, 0.9$ Hz, 4-CH),

5.13 (1 H, dd, $J = 11.4, 3.3$ Hz, 3-CH), 5.03 (1 H, d, $J = 3.7$ Hz, 1-CH), 4.59 (1 H, ddd, $J = 11.4, 9.7, 3.7$ Hz, 2-CH), 4.21–4.26 (1 H, m, 1'-CH), 4.13–4.20 (1 H, m, 5-CH), 4.03–4.10 (2 H, m, 6-CH), 2.47 (1 H, t, $J = 2.4$ Hz, 3'-CH), 2.13, 2.01, 1.96 (9 H, s, OCOCH₃), 1.94 (3 H, s, NCOCH₃); ¹³C NMR (100 MHz, CDCl₃) δ 170.6, 170.1, 170.0, 170.0 (CO), 96.4 (CH), 78.0 (C), 75.1 (CH), 67.9 (CH), 67.0 (CH), 69.9 (CH), 61.4 (CH₂), 55.0 (CH₂), 47.2 (CH), 22.9 (CH₃), 20.4 (CH₃), 20.4 (CH₃).

(2S)-2-[[(9H-Fluoren-9-ylmethoxy)carbonyl]amino]-3-(4-[(2,3,4,6-tetra-O-acetyl- α -D-galactopyranosyl)-oxy]-1H-1,2,3-triazol-1-yl)propanoic Acid (8). ¹H NMR (400 MHz, CDCl₃) δ 7.78–7.71 (2 H, m, Fmoc), 7.63–7.53 (2 H, m, Fmoc and 5''-CH), 7.50 (1 H, bs, 5''-CH), 7.43–7.25 (4 H, m, Fmoc), 6.63 (1 H, d, $J = 8.1$ Hz, NHCOCH₃), 6.16 (1 H, d, $J = 5.7$ Hz, NHFmoc), 5.38–5.34 (1 H, m, 4-CH),

5.17–5.10 (1 H, m, 3-CH), 5.02–4.69 (5 H, 1-CH, OCH₂C, 3'-CH_a and 2'-CH), 4.68–4.52 (1 H, m, 3'-CH_b), 4.51–4.36 (3 H, m, 2-CH and CH₂Fmoc), 4.35–4.25 (1 H, m, 5-CH), 4.24–4.17 (1 H, m, CHFmoc), 2.10, 2.03, 1.94 (9 H, s, OCOCH₃), 1.84 (3 H, s, NCOCH₃); ¹³C NMR (100 MHz, CDCl₃) δ 173.2 (CO), 172.2 (CO), 170.8 (CO), 170.6 (CO), 170.3 (CO), 155.9 (CO), 143.6 (C), 143.5 (C), 141.3 (C), 127.8 (CH), 127.1 (CH), 124.9 (CH), 124.8 (CH), 120.0 (CH), 95.9 (CH), 67.8 (CH), 67.2 (CH), 67.0 (CH), 67.0 (CH₂), 61.9 (CH₂), 59.8 (CH₂), 54.1 (CH), 51.0 (CH₂), 48.0 (CH), 47.0 (CH), 22.5 (CH₃), 20.6 (CH₃), 20.6 (CH₃).

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(2) Goddard-Borger, E. D.; Stick, R. V. *Org. Lett.* **2007**, *9*, 3797–3800.

(3) Lee, D. J.; Harris, P. W. R.; Kowalczyk, R.; Dunbar, R.; Brimble, M. A. *Synthesis* **2010**, DOI: 10.1055/s-0029-1218635.