Electronic Supplementary Material (ESI) for RSC Advances.

Binding modes of methyl α-D-glucopyranoside to an artificial receptor in the crystalline complexes

Linda Köhler, Conrad Hübler, Wilhelm Seichter and Monika Mazik*[a]

[a]Institut für Organische Chemie, Technische Universität Bergakademie Freiberg, Leipziger Strasse 29, 09599 Freiberg, Germany; Tel.: 03731392389; Fax: 03731393170
e-mail: monika.mazik@chemie.tu-freiberg.de; http://tu-freiberg.de/fakultaet2/orgch

1 Crystallographic data

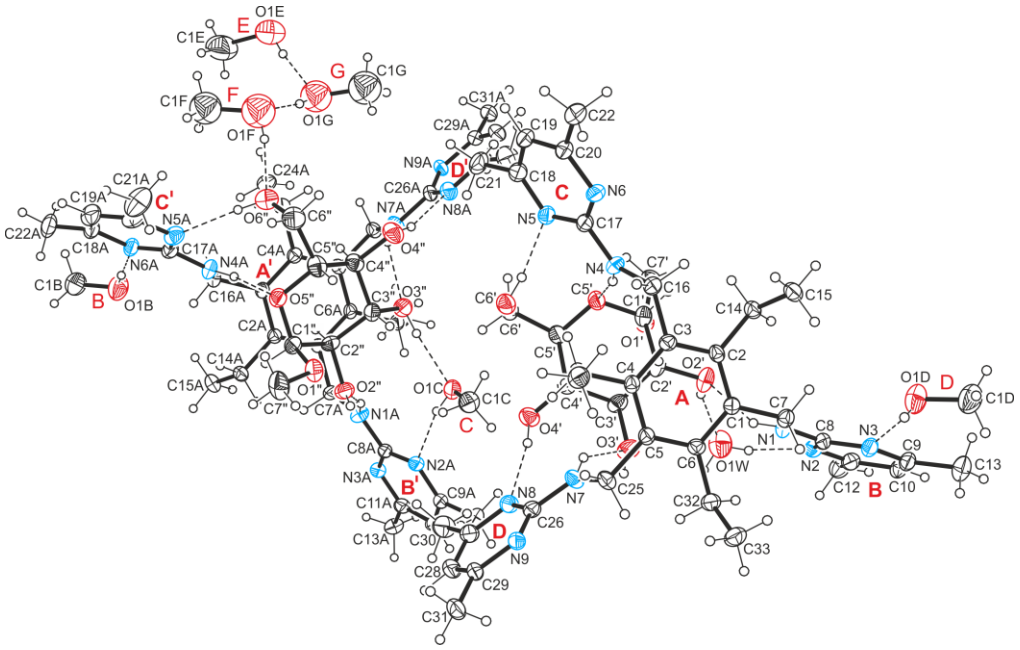

Figure S1. ORTEP-Plot of the crystal structure **2•MeαGlc**.

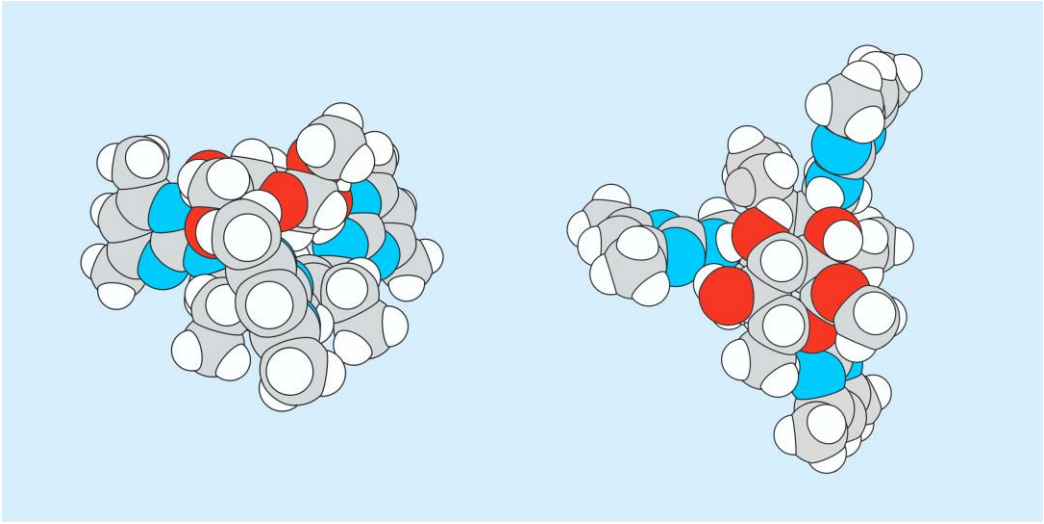

Figure S2. Space filling representation of complex I in the crystal structure **2•MeαGlc** (left: side view; right: top view of **2•MeαGlc**-I).

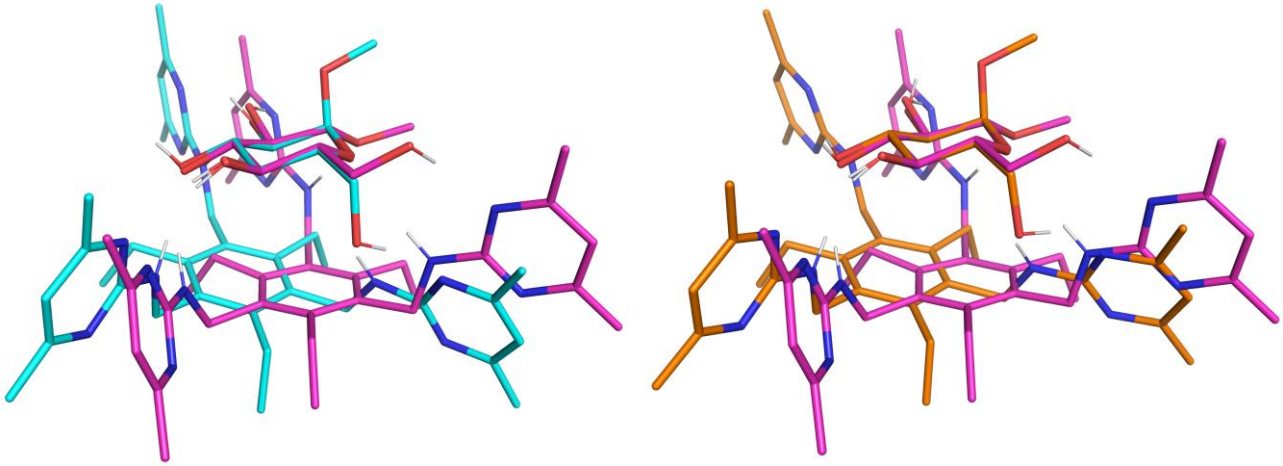

Figure S3. Views of the superimposed complex structures of **2•MeαGlc-I** (left, light blue lines) and **2•MeαGlc-II** (right, orange lines) with **2•MeβGlc-I** (pink lines) fitted on the carbohydrate atoms C1-C5 and O5 (N atoms are colored blue and O atoms red; all non-polar hydrogens omitted for clarity).

Table S1. Crystallographic and structure refinement data of **2•MeαGlc**.

Empirical formula	2 $C_{33}H_{45}N_9$ · 2 $C_7H_{14}O_6$ ·6 CH_3OH · H_2O
Formula weight	1734.18
Crystal system	Triclinic
Space group	*P*1
a (Å)	12.2116(14)
b (Å)	14.5632(16)
c (Å)	14.5319(17)
α (°)	67.762(9)
β (°)	86.941(9)
γ (°)	84.111(9)
V (Å^3)	2379.2(5)
Z	1
F(000)	938
D_c (Mg m^{-3})	1.210
μ (mm^{-1})	0.086
Data collection	
Temperature (K)	113(2)
No. of collected reflections	73592
within the θ-limit (°)	2.8 - 25.7
Index ranges $\pm h$, $\pm k$, $\pm l$	-14/14, -17/17, -17/17
No. of unique reflections	17273
R_{int}	0.0189
Refinement calculations: full-matrix least- squares on all F^2 values	
Weighting expression *w* [a]	$[\sigma^2(F_o^2)+(0.0983P)^2 + 1.3935P]^{-1}$
No. of refined parameters	1186
No. of *F* values used [$I>2\sigma(I)$]	16001
Final *R*-Indices	
R(=$\Sigma\|\Delta F\| / \Sigma\|F_o\|$)	0.0536
wR on F^2	0.1553
S (= Goodness of fit on F^2)	1.068
Final $\Delta\rho_{max}/\Delta\rho_{min}$ (e Å^{-3})	0.58/-0.56

[a] $P = (F_o^2 + 2F_c^2)/3$

Table S2. Selected geometric parameters of **2•MeαGlc**.

Complex	I		II
Dihedral angle (°)[a]			
mpla(A)-mpla(B)	79.8(1)	mpla(A^A)-mpla(B^A)	74.0(1)
mpla(A)-mpla(C)	72.1(1)	mpla(A^A)-mpla(C^A)	71.4(1)
mpla(A)-mpla(D)	87.5(1)	mpla(A^A)-mpla(D^A)	88.1(1)
mpla(B)-mpla(C)	38.5(1)	mpla(B^A)-mpla(C^A)	35.9(1)
mpla(B)-mpla(D)	59.9(1)	mpla(B^A)-mpla(D^A)	76.7(1)
mpla(C)-mpla(D)	81.8(1)	mpla(C^A)-mpla(D^A)	67.5(1)
Torsion angle (°)			
C1-C7-N1-C8	-176.0(3)	C1A-C7A-N1A-C8A	-161.8(3)
C7-N1-C8-N2	178.2(3)	C7A-N1A-C8A-N2A	169.2(3)
C3-C16-N4-C17	160.6(3)	C3A-C16A-N4A-C17A	168.5(4)
C16-N4-C17-N5	-167.5(3)	C16A-N4A-C17A-N5A	-167.8(4)
C5-C25-N7-C26	-173.2(3)	C5A-C25A-N7A-C26A	-179.9(3)
C25-N7-C26-N8	-178.3(4)	C25A-N7A-C26A-N8A	180.0(3)

[a] mpla means the least-squares plane through the aromatic ring.
Ring A: C1…C6; Ring B: N2,N3,C9…C20; Ring C: N5,N6,C24…C27; Ring D: N8,N9,C33…C36; Ring A^A: C1A…C6A; Ring B^A: N2A,N3A,C9A…C20A; Ring C^A: N5A,N6A,C24A…C27A; Ring D^A: N8A,N9A,C33A…C36A.

Table S3. Geometrical parameters of hydrogen bonds and arene interactions in the crystal structures **2•MeαGlc**.

Atoms		Distance (Å)		Angle (°)	Slippage (Å)
D-H···A		D···A	H···A	D-H···A	
Cg···*Cg*		*Cg*···*Cg*			
N(1)-H(1)···(O2')	*x*, *y*, *z*	2.823(4)	1.94(2)	172(6)	
N(4)-H(4)···(O5')	*x*, *y*, *z*	3.454(4)	2.65(3)	151(5)	
N(7)-H(7)···(O3')	*x*, *y*, *z*	2.879(5)	1.99(2)	173(5)	
N(1A)-H(1A)···(O2")	*x*, *y*, *z*	2.908(5)	2.06(2)	158(5)	
N(4A)-H(4A)···(O5")	*x*, *y*, *z*	3.344(4)	2.53(3)	152(4)	
N(7A)-H(7A)···(O3")	*x*, *y*, *z*	2.930(5)	2.05(2)	169(5)	
O(1B)-H(1B)···N(6A)	*x*, *y*, -1+*z*	3.098(6)	2.27	169	
O(1C)-H(1C)···N(2A)	*x*, *y*, *z*	2.818(4)	1.98	174	
O(1D)-H(1D)···N(3)	*x*, *y*, 1+*z*	3.060(6)	2.21	172	
O(1E)-H(1E)···O(1G)	*x*, -1+*y*, *z*	2.668(13)	1.82	176	
O(1F)-H(1F)···O(6")	*x*, -1+*y*, *z*	2.684(5)	1.84	175	
O(1G)-H(1G)···O(1F)	1+*x*, 1+*y*, *z*	2.652(13)	1.81	169	
O(2')-H(02')···O(1W)	*x*, *y*, *z*	2.729(6)	1.92(3)	165(8)	
O(3')-H(03')···O(1E)	*x*, *y*, *z*	2.637(7)	1.84	158	
O(4')-H(04')···N(8)	*x*, *y*, *z*	2.919(5)	2.09(2)	166(6)	
O(6')-H(06')···N(5)	*x*, *y*, *z*	2.793(5)	1.96(2)	168(4)	
O(2")-H(02")···O(1")	*x*, *y*, *z*	2.713(4)	2.26(5)	113(4)	
O(3")-H(03")···O(1C)	*x*, *y*, *z*	2.771(4)	1.96(5)	158(6)	
O(4")-H(04")···N(8A)	*x*, *y*, *z*	2.973(5)	2.14(2)	173(7)	
O(6")-H(06")···N(5A)	*x*, *y*, *z*	2.791(5)	1.99(3)	162(7)	
O(1W)-H(1WA)···N(2)	*x*, *y*, *z*	2.898(6)	2.17	145	
O(1W)-H(1WB)···O(1E)	*x*, *y*, *z*	2.824(9)	2.03	156	
C(6")-H(6"A)···O(1W)	*x*, 1+*y*, *z*	3.369(8)	2.47	154	
C(12)-H(12A)···O(6')	*x*, -1+*y*, *z*	3.500(5)	2.54	167	
C(23)-H(23B)···N(4)	*x*, *y*, *z*	3.268(5)	2.53	131	
C(32)-H(32A)···N(1)	*x*, *y*, *z*	3.255(5)	2.51	132	
C(19A)-H(19A)···O(1C)	*x*, 1+*y*, *z*	3.424(5)	2.50	166	
C(23A)-H(23D)···N(4A)	*x*, *y*, *z*	3.284(5)	2.56	130	
C(31A)-H(31D)···O(6')	-1+*x*, *y*, *z*	3.390(5)	2.49	152	
C(32A)-H(32D)···N(1A)	*x*, *y*, *z*	3.202(5)	2.45	133	
C(2')-H(2')···*Cg*(A)[a]	*x*, *y*, *z*	3.662(4)	2.74	154	
C(2")-H(2")···*Cg*(A^A)[a]	*x*, *y*, *z*	3.698(4)	2.75	158	
C(10A)-H(10A)···*Cg*(B)[a]	*x*, *y*, 1+*z*	3.558(4)	2.80	138	
C(14)-H(14B)···*Cg*(B^A)[a]	*x*, *y*, -1+*z*	3.775(6)	2.95	142	
C(14A)-H(14C)···*Cg*(C)[a]	*x*, *y*, 1+*z*	3.587(9)	2.93	126	
C(1G)-H(1G2)···*Cg*(D^A)[a]	1+*x*, *y*, *z*	3.692(6)	2.92	137	
C(33A)-H(33F) C(A)[b]	-1 + x, y, 1 + z	3.640(6)	2.88	135	
Cg(D)···*Cg*(D^A)[a]	1+*x*, *y*, *z*	3.534(4)			0.852
Cg(D^A)···*Cg*(D)[a]	-1+*x*, *y*, *z*	3.534(4)			0.567

[a] *Cg* means the centroid (centre of gravity) of the aromatic ring.

Ring A: C(1)…C(6); ring B: N(2),N(3),C(8)…C(11); ring C: N(5),N(6),C(17)…C(20); ring D: N(8),N(9),C(26)…C(29); ring A^A: C(1A)…C(6A); ring B^A: N(2A),N(3A),C(8A)…C(11A); ring D^A: N(8A),N(9A),C(26A)…C(29A).

[a] An individual ring atom instead of the ring centre was chosen as an acceptor site.

2 Synthesis of compound 2.

Synthesis of 1,3,5-tris[(4,6-dimethylpyrimidin-2-yl)aminomethyl]-2,4,6-triethylbenzene (2). A solution of 2-amino-4,6-dimethylpyrimidine (2.69 g, 30.0 mmol) and NaOH (4 g, 0.1 mol) in DMF was stirred for 30 min at room temperature. After addition of 1,3,5-tris(bromomethyl)-2,4,6-triethylbenzene (4 g, 9.1 mmol) the mixture was stirred at 90 °C for 24 h. Then the reaction mixture was cooled to room temperature, poured into water, the crude product was filtered off, washed with water and purified by flash chromatography ($CHCl_3$/MeOH + 7 N NH_3 in MeOH 100:1, v/v). Yield: 21 % (1.1 g). R_f = 0.54 ($CHCl_3$/MeOH + 7 N NH_3 in MeOH 100:1, v/v); M.p. 199-200 °C; ^{1}H-NMR (500 MHz, $CDCl_3$) δ/ppm = 6.34 (s, 3H), 4.76 (t, *J* = 4.3 Hz, 3H), 4.58 (d, *J* = 4.3 Hz, 6H), 2.75 (q, *J* = 7.5 Hz, 6H), 2.30 (s, 18H), 1.22 (t, *J* = 7.4 Hz, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ/ppm = 167.5, 161.8, 143.6, 133.0, 109.8, 39.9, 30.9, 23.9, 23.0, 16.7; HR-MS (ESI): calcd for $C_{33}H_{46}N_9$ 568.38706 [M + H^+]; found 568.38714 [M + H^+].