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Note

X-ray and conformational analysis of methyl 3-amino-2,3-dideoxy-α-D-*arabino*-hexopyranoside

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Abstract—The structure, conformation and configuration of methyl 3-amino-2,3-dideoxy-α-D-*arabino*-hexopyranoside (**I**) were confirmed by ^{1}H NMR, ^{13}C NMR and IR spectroscopy, as well as by optical rotation. The structure of the compound studied was also determined by single crystal X-ray crystallography at 293 K and refined to a final $R = 0.0521$ based on 1798 independent reflections. The title compound crystallized in the tetragonal space group $P4_3$ with $a = 6.572(1)$ Å, $b = 6.572(1)$ Å, $c = 41.161(8)$ Å, $D_c = 1.324\,\mathrm{Mg\,cm^{-3}}$ and $V = 1777.8(5)$ Å^3 for $Z = 8$. The packing arrangement in the unit cell displayed a stratified structure. Moreover, medium-strength N–H···O and O–H···O hydrogen bonds, which stabilized the 3-D structure of compound **I**, were observed.

Keywords: Methyl 3-amino-2,3-dideoxy-α-D-*arabino*-hexopyranoside; Crystal structure; Hydrogen bonds

Amino sugars have been found in a wide variety of natural products.[1] Since an amino group can be protonated, alkylated or acetylated in order to alter its ability to form hydrogen bonds and ammonium salts, its presence can greatly influence the hydrophobicity and surface properties of the natural products. For example, the amino group of daunosamine in doxorubicin may act as the recognition site for the multidrug transporter P-glycoprotein in drug-resistant tumour cells. Substitution of this basic amino group with a hydroxy group partially or completely changes the properties of the multidrug transporter because of reduced transportation by P-glycoprotein.[2] Indeed, amino sugars have been found to play an important role in the biological activities of many macrolide and aminoglycoside antibiotics.[3] A particularly notable example is desosamine, dimethylamino-3,4,6-trideoxyhexose, which has been found in many macrolide antibiotics, including erythromycin,[4–6] methymycin, pikromycin and oleandomycin.

As previously reported,[7] addition of hydrazoic acid to the α,β-unsaturated aldehyde derived from tri-*O*-acetyl-D-glucal, followed by methyl glycosidation, led to a mixture of 3-azido-2,3-dideoxy glycosides. The title compound **I** was obtained by the reduction of methyl 3-azido-2,3-dideoxy-α-D-*arabino*-hexopyranoside.[8] The latter compound can be used as a substrate for the synthesis of important bioactive substances such the 3-amino-2,3,6-trideoxyhexopyranoses[9] or aminocyclitols.[10]

In this paper, we deal with the conformational analysis and 3-D crystal structure of methyl 3-azido-2,3-dideoxy-α-D-*arabino*-hexopyranoside. The compound studied is an α anomer and has a $^{4}C_1$ conformation in the solid state. In this crystal, molecules are arranged in a bilayer structure. The hydrogen-bonding linkages between sugar moieties form an infinite chain throughout the crystal, which affects both the structure and bioactivity of compound studied. The results obtained indicate the important role of the sugar in the crystal. The molecules are linked by a 'head-to-tail' dimer interaction, and 10-membered rings are formed. Orientations of the amino and hydroxyl groups in these rings indicate the possibility of formation of the coordination

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complexes via hydrogen bonding with metal ions. It should be stressed that, although coordination chemistry plays a central role in these processes, carbohydrate–metal complexes are still poorly understood and relatively few well-characterized complexes of metal ions with carbohydrate ligands have been reported, especially those lacking anchor groups.

The analyses of **I** have shown its α-anomeric configuration as indicated by the high positive value of the optical rotation and the large chemical shift of the H-1 proton, $\delta \sim 4.8$ ppm (as compared to δ of the β anomer[7]). Accordingly, the H atom in this compound is arranged equatorially. The D-*arabino* configuration was assigned on the basis of coupling constants $J_{3,4} = J_{4,5} \sim 9.5$ Hz, which corresponds to the axial orientation of the H-3, H-4 and H-5 protons. These findings are additionally supported by the high coupling constant, $J_{2a,3} \sim 12$ Hz, corresponding to the diaxial orientation of the H-2a and H-3 protons in this compound. Again, the high geminal-coupling constant, $J_{2a,e} \sim 13$ Hz, revealed the presence of a system of methylene protons (2-deoxy). All these findings showed that compound **I** occurs in a typical chair $^4C_1$ conformation (Fig. 1). The values of bond lengths and angles determined in this work (Tables 3 and 4) for **I** agree well with that expected for $^4C_1$ conformation.

Methyl 3-amino-2,3-dideoxy-α-D-*arabino*-hexopyranoside crystallizes with eight molecules in the unit cell (with two molecules in the asymmetric unit). The molecules in this crystal are arranged in a bilayer (A and B)

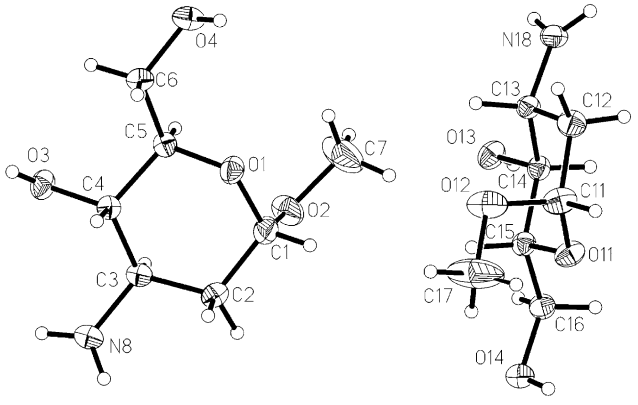

Figure 1. Structures of compound **I** in two A and B layers, showing the atomic notation and 50% thermal ellipsoids.

Table 1. Crystal data and structure refinement

Empirical formula	$C_7H_{15}NO_4$
Formula weight	177.20
Temperature (K)	293(2)
Radiation wavelength (Å)	1.54178
Crystal system	Tetragonal
Space group	$P4_3$
Unit cell dimensions	
a (Å)	6.572(1)
b (Å)	6.572(1)
c (Å)	41.161(8)
V (Å^3)	1777.8(5)
Z	8
Calculated density (Mg m^{-3})	1.324
Absorption coefficient (mm^{-1})	0.913
F (0 0 0)	768
Crystal size (mm)	0.3 × 0.4 × 0.6
Θ Range for data collection (°)	4.30–73.23
Limiting indices	$-7 \leqslant h \leqslant 0$, $0 \leqslant k \leqslant 8$, $-51 \leqslant l \leqslant 0$
Reflections collected/unique	2086/1798 [$R_{int} = 0.0555$]
Completeness to 2θ (%)	99.1
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	1798/1/224
Goodness-of-fit on F^2	1.013
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0521$, $wR_2 = 0.1418$
R indices (all data)	$R_1 = 0.0547$, $wR_2 = 0.1456$
Absolute structure parameter	0.2(3)
Extinction coefficient	0.024(2)
Largest diff. peak and hole (e A^{-3})	0.342 and −0.279

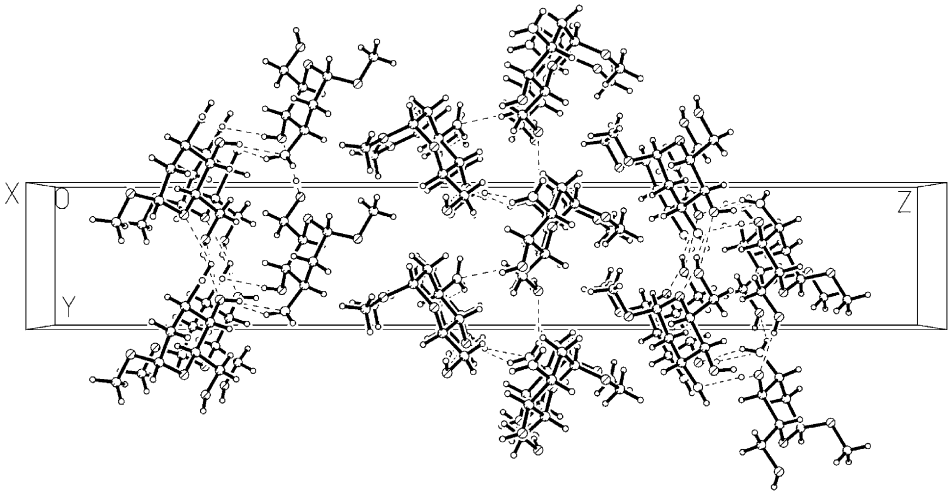

Figure 2. Crystal packing in unit cell of molecules A and B (view along x-axis). The double lines represent hydrogen bonds.

structure. Cremer–Pople puckering parameters[11] for the pyranoside ring are: $Q = 0.546(3)$ Å and $\Theta = 5.3(3)°$ for rings C-1–C-2–C-3–C-4–C-5–O-1 of the A layer, and $Q = 0.536(3)$ Å and $\Theta = 4.5(4)°$ for rings C-11–C-12–C-13–C-14–C-5–O-11 of the B layer.

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$)

Molecule	Atom	x/a	y/b	z/c	U_{eq}[a]
A layer	C-1	6553(6)	8257(7)	871(1)	57(1)
	C-2	4643(6)	9158(5)	735(1)	52(1)
	C-3	3112(5)	7557(5)	625(1)	39(1)
	C-4	4165(4)	5987(4)	411(1)	37(1)
	C-5	6070(4)	5145(5)	576(1)	38(1)
	C-6	7294(5)	3765(5)	358(1)	43(1)
	C-7	7919(14)	6705(17)	1331(2)	133(4)
	N-8	1472(5)	8529(5)	443(1)	50(1)
	O-1	7412(4)	6809(4)	655(1)	49(1)
	O-2	6123(5)	7397(7)	1173(1)	76(1)
	O-3	2768(3)	4378(4)	345(1)	49(1)
	O-4	8930(4)	2811(4)	521(1)	54(1)
	H-1	7541	9356	903	68
	H-2A	4989	10016	551	62
	H-2B	4016	10013	899	62
	H-3A	2541	6877	817	47
	H-3B	2891	4017	155	59
	H-4A	4557	6633	206	44
	H-4B	9987	3428	482	65
	H-5A	5690	4417	775	46
	H-6A	6404	2727	269	52
	H-6B	7825	4554	178	52
	H-7A	7562	6045	1531	200
	H-7B	8789	7844	1376	200
	H-7C	8620	5760	1192	200
	H-8A	1239	8176	245	59
	H-8B	740	9454	533	59
B layer	C-11	13265(6)	11575(6)	1767(1)	55(1)
	C-12	14163(5)	9636(6)	1904(1)	51(1)
	C-13	12558(5)	8130(5)	2011(1)	41(1)
	C-14	10975(4)	9168(4)	2225(1)	39(1)
	C-15	10156(5)	11093(4)	2061(1)	39(1)
	C-16	8778(5)	12290(5)	2280(1)	45(1)
	C-17	11718(17)	12885(13)	1302(2)	135(4)
	N-18	13531(5)	6477(4)	2196(1)	48(1)
	O-11	11801(4)	12412(4)	1981(1)	49(1)
	O-12	12408(6)	11112(5)	1465(1)	75(1)
	O-13	9387(4)	7779(4)	2292(1)	49(1)
	O-14	7818(4)	13932(4)	2114(1)	55(1)
	H-11A	14361	12567	1736	66
	H-12A	15019	9972	2088	61
	H-12B	15016	9007	1740	61
	H-13A	11886	7559	1819	49
	H-14A	11622	9554	2430	46
	H-15A	9420	10722	1862	46
	H-16A	7744	11393	2368	54
	H-16B	9564	12824	2461	54
	H-17A	11892	12715	1072	202
	H-17B	12491	14040	1374	202
	H-17C	10304	13099	1349	202
	H-18A	14825	6488	2227	58
	H-18B	12806	5500	2273	58
	H-13A	9061	7873	2484	59
	H-14B	8598	14904	2106	66

[a] U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table 3. Selected bond lengths (Å) and valence angles (°)

A layer		B layer	
Bond lengths			
C-1–O-2	1.390(6)	C-11–O-12	1.399(6)
C-1–O-1	1.423(4)	C-11–O-11	1.416(4)
C-1–C-2	1.496(6)	C-11–C-12	1.513(5)
C-2–C-3	1.526(5)	C-12–C-13	1.512(5)
C-3–N-8	1.461(4)	C-13–N-18	1.473(4)
C-3–C-4	1.518(4)	C-13–C-14	1.524(4)
C-4–O-3	1.426(4)	C-14–O-13	1.414(4)
C-4–C-5	1.529(4)	C-14–C-15	1.532(4)
C-5–O-1	1.437(4)	C-15–O-11	1.424(4)
C-5–C-6	1.512(4)	C-15–C-16	1.502(4)
C-6–O-4	1.418(4)	C-16–O-14	1.425(4)
C-7–O-2	1.424(7)	C-17–O-12	1.420(6)
Valence angles			
O-2–C-1–O-1	111.3(4)	O-12–C-11– O-11	111.4(3)
O-2–C-1–C-2	108.9(3)	O-12–C-11–C-12	107.7(3)
O-1–C-1–C-2	111.3(3)	O-11–C-11–C-12	111.2(3)
C-1–C-2–C-3	112.9(3)	C-13–C-12–C-11	112.8(3)
N-8–C-3–C-4	109.7(3)	N-18–C-13–C-12	109.3(3)
N-8–C-3–C-2	109.6(3)	N-18–C-13–C-14	109.2(3)
C-4–C-3–C-2	109.9(3)	C-12–C-13–C-14	110.5(3)
O-3–C-4–C-3	108.9(2)	O-13–C-14–C-13	109.2(2)
O-3–C-4–C-5	110.2(3)	O-13–C-14–C-15	111.1(3)
C-3–C-4–C-5	111.2(3)	C-13–C-14–C-15	110.8(3)
O-1–C-5–C-6	105.4(2)	O-11–C-15–C-16	106.1(2)
O-1–C-5–C-4	109.2(3)	O-11–C-15–C-14	109.7(3)
C-6–C-5–C-4	112.8(3)	C-16–C-15–C-14	112.3(3)
O-4–C-6–C-5	112.4(3)	O-14–C-16–C-15	112.0(3)
C-1–O-1–C-5	114.0(3)	C-11–O-11–C-15	115.0(3)
C-1–O-2–C-7	112.1(6)	C-11–O-12–C-17	111.8(5)

The packing arrangement assumed for the title compound is shown in Figure 2. Hydrogen bonds are formed, not only between the layers, but also within them. The hydrogen bonds connecting the A layers (two per molecule in each direction) are long and weak. Particular layers are arranged in such a way that the methyl groups form a 'head to head' structure. However, no short hydrogen-bonding interactions are observed. Details of the intermolecular hydrogen bonds for the structure studied are given in Table 5.

1. Experimental

1.1. General methods

Evaporations were performed under reduced pressure. Melting points were uncorrected. A Hilger–Watt polarimeter and 1-dm tubes were used for the measurement of specific rotations. IR spectra were recorded as Nujol mulls with a Bruker IFS 66 spectrophotometer, and NMR spectra were determined at 400 MHz with a Varian Mercury 400 spectrometer. Chemical shifts were referred to Me_4Si ($\delta = 0.00$) as the internal standard and

were recorded together with spin-coupling values (Hz). TLC was performed on E. Merck Kieselgel 60 F-254 plates; zones were detected by heating. Elemental analyzes were conducted with a Carlo Erba EA1108 elemental analyzer. X-ray powder diffraction data were collected at room temperature (293 K) on a KUMA KM-4 diffractometer with Cu Kα radiation ($\lambda = 1.54178$ Å).

1.2. Preparation of methyl 3-amino-2,3-dideoxy-α-D-*arabino*-hexopyranoside

Methyl 3-azido-2,3-dideoxyhexopyranoside (1.55 g, 7.62 mmol)[7,8] was dissolved in 40 mL of dry MeOH. The reaction mixture was stirred, and argon was bubbled through it for 15 min. After this time the reaction mixture was hydrogenated at rt for 1.5 h with 90 mg of 10% Pd/C. The catalyst was centrifuged off and washed, and the supernatant was filtered. The filtrate was evaporated to about 20 mL. Then, a 10 mL mixture of 2:1 Et_2O and EtOAc was added, and the solution was stored overnight at 4 °C. The colourless crystals that formed were filtered off, washed with ether and dried to yield 1.2 g (89%) of the title compound: mp 155.6–157.5 °C; $[\alpha]_D^{20}$ +144.12 (*c* 1.77, CH_3OH); R_f 0.25 (MeOH) IR (Nujol); 3382, 3345, 3295 (*v*-NH-amine), 2964, 2910 (*v*-OH), 2832 (*v*-acetal), 1608 (δ-NH-amine), 1392, 1454 (δ-OCH_3), 1131, 956 (*v*-C–O–C) cm^{-1}; ^1H NMR (CD_3OD); δ_H: 1.512 (td, 1 H, $J_{2a,e}$ 13.2, $J_{2a,3}$ 12.2 Hz, H-2a), 1.97 (dq, 1H, $J_{2e,3}$ 4.8 Hz, H-2e), 3.002 (m, 1H, $J_{3,4}$ 9.2 Hz, H-3), 3.087 (t, 1H, $J_{4,5}$ 9.6 Hz, H-4), 3.333 (s, 3H, OCH_3), 3.476 (m, 1H, $J_{5,6}$ 5.2, $J_{5,6'}$ 2.8 Hz, H-5), 3.667 (dd, 1H, $J_{6,6'}$ 11.8 Hz, H-6), 3.804 (dd, 1H, H-6′), 4.736 (d, 1H, $J_{1,2a}$ 3.6 Hz, H-1); ^{13}C NMR (CD_3OD): δ_C (ppm): 38.278 (C-2), 55.052 (OCH_3), 50.756 (C-3), 63.121(C-6), 74.015 (C-4), 74.25 (C-5), 99.449 (C-1); EIMS: *m*/*z* 203 (M^+, 45%); calcd for $C_7H_{15}NO_4$: C, 47.3; H, 8.61; N, 7.79. Found: C, 47.45; H, 8.53; N, 7.8.

1.3. Single-crystal X-ray structure determinations

The crystal structure of the title compound was solved by the SHELXS-97 program and refined by SHELXL-97.[12,13] A summary of crystallographic data, data collection and structure refinement are given in Table 1. Data collection, cell refinement and data reduction was prepared by using KM-4 software.[14]

All H atoms were placed geometrically and refined using a riding model with C–H=0.96 Å, N–H=0.86 Å, O–H=0.82 Å and U_{iso}(H)=1.2 U_{eq}(C) (C–H=0.96 Å and U_{iso}(H)=1.5 U_{eq}(C) in the case of the methyl H atoms). The compound structure showing the atom numbering scheme and molecular packing in the crystal are illus-

Table 4. Selected torsion angles (°) and dihedral angles (°)

A layer		B layer	
Torsion angles			
O-2–C-1–C-2–C-3	−71.0(4)	O-12–C-11–C-12–C-13	−71.2(4)
O-1–C-1–C-2–C-3	52.0(5)	O-11–C-11–C-12–C-13	51.1(4)
C-1–C-2–C-3–N-8	−170.0(3)	C-11–C-12–C-13–N-18	−169.3(3)
C-1–C-2–C-3–C-4	−49.4(4)	C-11–C-12–C-13–C-14	−49.2(4)
N-8–C-3–C-4–O-3	−66.2(3)	N-18–C-13–C-14–O-13	−65.9(3)
C-2–C-3–C-4–O-3	173.2(3)	C-12–C-13–C-14–O-13	173.9(3)
N-8–C-3–C-4–C-5	172.2(3)	N-18–C-13–C-14–C-15	171.4(3)
C-2–C-3–C-4–C-5	51.6(4)	C-12–C-13–C-14–C-15	51.2(3)
O-3–C-4–C-5–O-1	−177.5(3)	O-13–C-14–C-15–O-11	−177.0(2)
C-3–C-4–C-5–O-1	−56.6(3)	C-13–C-14–C-15–O-11	−55.5(3)
O-3–C-4–C-5–C-6	65.7(3)	O-13–C-14–C-15–C-16	65.2(3)
C-3–C-4–C-5–C-6	−173.4(3)	C-13–C-14–C-15–C-16	−173.2(3)
O-1–C-5–C-6–O-4	67.6(3)	O-11–C-15–C-16–O-14	67.6(3)
C-4–C-5–C-6–O-4	−173.3(3)	C-14–C-15–C-16–O-14	−172.5(3)
O-2–C-1–O-1–C-5	63.2(4)	O-12–C-11–O-11–C-15	62.7(4)
C-2–C-1–O-1–C-5	−58.5(5)	C-12–C-11–O-11–C-15	−57.5(4)
C-6–C-5–O-1–C-1	−178.1(3)	C-16–C-15–O-11–C-11	−178.6(3)
C-4–C-5–O-1–C-1	60.5(4)	C-14–C-15–O-11–C-11	59.9(4)
O-1–C-1–O-2–C-7	63.6(6)	O-11–C-11–O-12–C-17	64.1(6)
C-2–C-1–O-2–C-7	−173.3(6)	C-12–C-11–O-12–C-17	−173.7(5)
Dihedral angles H–H			
H-1A–C-1–C-2–H-2A	49.85	H-11A–C-11–C-12–H-12A	49.66
H-1A–C-1–C-2–H-2B	−67.55	H-11A–C-11–C-12–H-12B	−67.80
H-2A–C-2–C-3–H-3A	−168.34	H-12A–C-12–C-13–H-13A	−167.60
H-2B–C-2–C-3–H-3A	−50.94	H-12B–C-12–C-13–H-13A	−50.14
H-3A–C-3–C-4–H-4A	171.96	H-13A–C-13–C-14–H-14A	171.75
H-4A–C-4–C-5–H-5A	−176.36	H-14A–C-14–C-15–H-15A	−176.07
H-5A–C-5–C-6–H-6A	70.64	H-15A–C-15–C-16–H-16A	70.58
H-5A–C-5–C-6–H-6B	−171.74	H-15A–C-15–C-16–H-16B	−171.65

Table 5. Hydrogen bond distances and angles with H···A > *r* (Å)+2.00 Å and D–H···A > 110°

D–H···A	D–H	H···A	D···A	D–H···A
N-8–H-8A···O-13[i]	0.860	2.129	2.782(2)	132.45
N-8–H-8B···O-4[ii]	0.860	2.507	3.288(2)	151.46
O-3–H-3C···N-18[iii]	0.820	1.962	2.774(2)	170.27
O-4–H-4B···O-3[iv]	0.820	2.012	2.819(2)	167.72
N-18–H-18B···O-11[v]	0.860	2.448	3.034(2)	125.99
O-13–H-13B···N-8[vi]	0.820	1.970	2.782(2)	170.44
O-14–H-14B···O-13[vii]	0.820	2.104	2.827(2)	147.03

Symmetry codes: (i) $y+1, x, z-1/4$; (ii) $x-1, y+1, z$; (iii) $-y+1, x-1, z-1/4$; (iv) $x+1, y, z$; (v) $x, y-1, z$; (vi) $y, -x+1, z+1/4$; (vii) $x, y+1, z$.

trated in Figures 1 and 2, respectively.[15,16] The coordinates of atoms and their isotropic temperature factors are set out in Table 2, and a selection of the crystal's important geometric parameters are given in Tables 3 and 4.

Supplementary materials

Full crystallographic details, excluding structure features, have been deposited (deposition No CCDC 224688) with the Cambridge Crystallographic Data Centre. These data may be obtained, on request, from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Tel.: +44-1223-336408; fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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