

C3—C4—C5	104,1 (2)	C12—C11—C9	118,4 (2)
C4—C5—C10	104,9 (2)	C13—C12—C11	124,4 (2)
O3—C9—C11	108,1 (2)	C12—C13—C14	123,0 (2)
O3—C9—C8	107,29 (15)		

**Verbindung (5)****Kristalldaten**

C <sub>29</sub> H <sub>42</sub> O <sub>6</sub>	Cu K $\alpha$ Strahlung
$M_r = 486,63$	$\lambda = 1,54178 \text{ \AA}$
Orthorhombisch	Gitterparameter aus 35
$P2_12_12_1$	Reflexen
$a = 10,701 (1) \text{ \AA}$	$\theta = 15,0\text{--}30,0^\circ$
$b = 12,771 (2) \text{ \AA}$	$\mu = 0,696 \text{ mm}^{-1}$
$c = 18,751 (2) \text{ \AA}$	$T = 293 (2) \text{ K}$
$V = 2562,4 (5) \text{ \AA}^3$	Nadel
$Z = 4$	$0,50 \times 0,20 \times 0,15 \text{ mm}$
$D_x = 1,261 \text{ Mg m}^{-3}$	Farblos
$D_m$ nicht gemessen	

**Datensammlung**

Kuma Diffraction KM-4	$R_{\text{int}} = 0,0183$
Diffraktometer	$\theta_{\text{max}} = 70,11^\circ$
$\omega/2\theta$ Abtastung	$h = -13 \rightarrow 13$
Absorptionskorrektur:	$k = -15 \rightarrow 15$
keine	$l = -22 \rightarrow 22$
5129 gemessene Reflexe	2 Kontrollreflexe
4527 unabhängige Reflexe	alle 100 Reflexen
3984 Reflexe mit	Intensitätsschwankung:
$I > 2\sigma(I)$	2,0%

**Verfeinerung**

Verfeinerung auf $F^2$	$\Delta\rho_{\text{max}} = 0,220 \text{ e \AA}^{-3}$
$R(F) = 0,0411$	$\Delta\rho_{\text{min}} = -0,283 \text{ e \AA}^{-3}$
$wR(F^2) = 0,1222$	Extinktionskorrektur:
$S = 1,086$	<i>SHELXL93</i>
4527 Reflexe	Extinktionskoeffizient:
321 Parameter	0,0009 (2)
H-Atomparameter nicht	Atomformfaktoren aus
verfeinert, außer H(O3)	<i>International Tables for</i>
$w = 1/[\sigma^2(F_o^2) + (0,0780P)^2$	<i>Crystallography</i> (Vol. C)
$+ 0,4294P]$ wobei $P =$	Absolute-Konfiguration:
$(F_o^2 + 2F_c^2)/3$	Flack (1983)
$(\Delta/\sigma)_{\text{max}} < 0,001$	Flack-Parameter = $-0,1 (2)$

Tabelle 3. Ausgewählte Geometrische Parameter ( $\text{\AA}$ ,  $^\circ$ ) für (5)

O1—C3	1,192 (3)	C3—C4	1,521 (4)
O2—C3	1,351 (3)	C4—C5	1,552 (3)
O2—C10	1,468 (3)	C5—C10	1,555 (3)
O3—C9	1,432 (2)	C11—C12	1,460 (3)
O4—C11	1,218 (2)	C12—C13	1,335 (3)
C3—O2—C10	109,9 (2)	O3—C9—C10	105,5 (2)
O2—C3—C4	112,0 (2)	O2—C10—C5	102,9 (2)
C3—C4—C5	101,4 (2)	C12—C11—C9	116,4 (2)
C4—C5—C10	103,1 (2)	C13—C12—C11	125,5 (2)
O3—C9—C11	101,9 (2)	C12—C13—C14	121,6 (2)
O3—C9—C8	111,7 (2)		

Tabelle 4. Wasserstoff-Bindung Geometrie ( $\text{\AA}$ ,  $^\circ$ )

Verbindung	D—H...A	D—H	H...A	D...A	D—H...A
(3)	O3—HO3...O4	0,98 (4)	2,09 (4)	2,638 (3)	113 (3)
(4)	O3—HO3...O4	1,01 (5)	1,90 (4)	2,610 (2)	125 (3)
(5)	O3—HO3...O5 <sup>i</sup>	0,80 (3)	2,23 (3)	2,982 (3)	156 (3)

Symmetriebezeichnung: (i)  $-x, y - \frac{1}{2}, \frac{1}{2} - z$ .

Das H(O3)-Atom wurde in (3)–(5) aus Differenz-Fourier-Synthese ermittelt und isotrop verfeinert. Die Positionen der übrigen Wasserstoffatome wurden geometrisch berechnet;  $U(\text{H}) = 1,1U_{\text{aq}}(\text{C})$ .

Für alle Strukturen, Datensammlung: *Kuma KM-4 User's Guide* (Kuma, 1991); Zellverfeinerung: *Kuma KM-4 User's Guide*; Datenreduktion: *Kuma KM-4 User's Guide*; Lösung der Struktur: *SHELXS86* (Sheldrick, 1985); Verfeinerung der Struktur: *SHELXL93* (Sheldrick, 1993); Molekülgrafik: *ORTEPII* (Johnson, 1976); Programm für die Herrstellung von Veröffentlichungsmaterialien: *SHELXL93*.

Die Listen der Strukturparameter, anisotropen Verschiebungsparameter, Atomkoordinaten, und vollständigen geometrischen Daten sind bei der IUCr (Aktenzeichen: CF1142) hinterlegt. Kopien sind erhältlich durch: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Literatur**

- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
 Johnson, C. K. (1976). *ORTEPII*. Protokoll ORNL-5138. Oak Ridge National Laboratory, Tennessee, VStA.  
 Kuma (1991). *Kuma KM-4 User's Guide*. Kuma Diffraction, Wrocław, Polen.  
 Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Universität Göttingen, Deutschland.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for Crystal Structure Refinement*. Universität Göttingen, Deutschland.  
 Wrzeczono, U., Zaprutko, L., Budzianowski, J., Wójtowicz, H. & Dubowska, D. (1987). *Magn. Reson. Chem.* **25**, 223–226.  
 Zaprutko, L. (1995). *Pol. J. Chem.* **69**, 1003–1012.  
 Zaprutko, L. (1996). *Pol. J. Chem.* Im Druck.

*Acta Cryst.* (1997). **C53**, 264–266

**Methyl 3-Deoxy- $\alpha$ -D-arabino-hexopyranoside**

ARTEM G. EVDOKIMOV<sup>a</sup> AND FELIX FROLOW<sup>b</sup>

<sup>a</sup>Structural Biology Department, Weizmann Institute of Science, Rehovot, Israel, and <sup>b</sup>Chemical Services Department, Weizmann Institute of Science, Rehovot, Israel. E-mail: nf9501@ferret.weizmann.ac.il

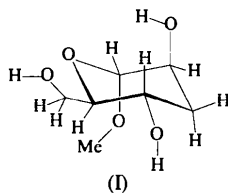
(Received 15 July 1996; accepted 15 October 1996)

**Abstract**

The structure of the title compound, C<sub>7</sub>H<sub>14</sub>O<sub>5</sub>, shows no deviations from the geometrical parameters of pyranose carbohydrates. Crystal packing contains a dense network of hydrogen bonds linking molecules in a three-dimensional network.

### Comment

The title compound, (I), is the first representative of simple deoxyribofuranosides whose crystal structure has been reported. Among the derivatives of 3-deoxyhexo-



pyranoses only two compounds could be found in the Cambridge Structural Database (Allen & Kennard, 1993), namely methyl 3,6-dideoxy- $\beta$ -D-ribohexopyranoside monohydrate (Birnbaum & Bundle, 1979) and methyl 3,6-dideoxy- $\alpha$ -D-arabino-hexopyranoside (Birnbaum & Bundle, 1985). Bond lengths and angles show

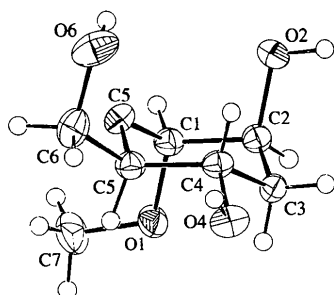


Fig. 1. Displacement ellipsoid plot for (I). Ellipsoids represent 50% probability and H atoms are represented by spheres of arbitrary radii.

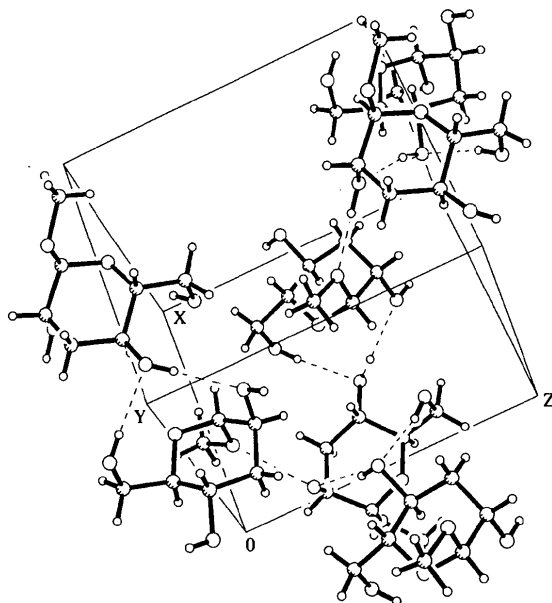


Fig. 2. A view of the crystal packing in which hydrogen bonds, represented by dashed lines, link molecules into a three-dimensional network.

no significant differences when compared with those of the reported 3-deoxy structures and are generally consistent with other pyranoside structures (Kirby, 1983; Fuchs, Schleifer & Tartakovsky, 1984).

All O atoms of the title compound (Fig. 1) participate in hydrogen bonding, linking molecules into an elaborate three-dimensional network (Fig. 2 and Table 2). An intramolecular hydrogen bond is also present (O6—H6...O5), which probably participates in further stabilization of the pyranose ring conformation.

The structure of the title compound has been determined as a part of a wider study into the affinity of carbohydrates to concanavalin A (Evdokimov, Gilboa & Frolow, 1996).

### Experimental

The title compound was synthesized by reducing the epoxy ring of methyl 2,3-anhydro-4,6-benzylidene- $\alpha$ -D-mannopyranoside with  $\text{LiAlH}_4$  (Prins, 1948). It was crystallized from an alcohol-acetone-ether-hexane mixture.

#### Crystal data

$\text{C}_7\text{H}_{14}\text{O}_5$   
 $M_r = 178.18$   
 Orthorhombic  
 $P2_12_12_1$   
 $a = 8.830(2) \text{ \AA}$   
 $b = 9.363(2) \text{ \AA}$   
 $c = 10.134(2) \text{ \AA}$   
 $V = 837.8(3) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.412 \text{ Mg m}^{-3}$   
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 24 reflections  
 $\theta = 12\text{--}15^\circ$   
 $\mu = 0.120 \text{ mm}^{-1}$   
 $T = 293(2) \text{ K}$   
 Tetragonal pyramid  
 $0.2 \times 0.2 \times 0.2 \text{ mm}$   
 Colourless

#### Data collection

Rigaku AFC-5R diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 5234 measured reflections  
 2119 independent reflections  
 1638 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.0552$

$\theta_{\text{max}} = 28.57^\circ$   
 $h = -11 \rightarrow 11$   
 $k = -12 \rightarrow 12$   
 $l = -12 \rightarrow 13$   
 3 standard reflections every 200 reflections  
 frequency: 56 min  
 intensity decay: 0.5%

#### Refinement

Refinement on  $F^2$   
 $R(F) = 0.059$   
 $wR(F^2) = 0.120$   
 $S = 1.078$   
 2102 reflections  
 161 parameters  
 H atoms: see below  
 $w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 0.0702P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 0.194 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.190 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Scattering factors from International Tables for Crystallography (Vol. C)

Table 1. Selected torsion angles (°)

C1—O5—C5—C4	55.3 (3)	C3—C2—C1—O5	54.3 (3)
C4—C3—C2—C1	-52.6 (3)	O5—C5—C4—C3	-51.3 (3)
C5—O5—C1—C2	-56.9 (3)	C2—C3—C4—C5	51.7 (3)

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O6—H6...O5	0.820†	2.554 (8)	2.900 (2)	106.8 (6)
O6—H6...O4 <sup>i</sup>	0.820†	2.117 (6)	2.910 (3)	168 (2)
O4—H24...O2 <sup>ii</sup>	0.86 (4)	2.06 (4)	2.910 (3)	166 (3)
O4—H24...O5 <sup>ii</sup>	0.86 (4)	2.58 (3)	3.041 (3)	114 (2)
O2—H22...O1 <sup>iii</sup>	0.90 (4)	1.90 (4)	2.804 (3)	174 (3)

Symmetry codes: (i)  $x - \frac{1}{2}, -\frac{3}{2} - y, -1 - z$ ; (ii)  $\frac{1}{2} + x, -\frac{3}{2} - y, -1 - z$ ; (iii)  $-x, \frac{1}{2} + y, -\frac{3}{2} - z$ .

† H atom positioned geometrically and not refined.

H atoms were refined freely, except for H4 and H6 (attached to C4 and O6, respectively), which were constrained. The absolute structure was indeterminate by refinement and was assumed from the synthesis.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *Xtal3.2* (Hall, Flack & Stewart, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: CF1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.
- Birnbaum, G. I. & Bundle, D. R. (1979). *Biochim. Biophys. Acta*, **582**, 515–519.
- Birnbaum, G. I. & Bundle, D. R. (1985). *Can. J. Chem.* **63**, 739–741.
- Evdokimov, A. G., Gilboa, A. J. & Frolow, F. (1996). *Thermodynamics of Binding of Mannose Homomorphs to Concanavalin A*. In preparation.
- Fuchs, B., Schleifer, L. & Tartakovsky, E. (1984). *Nouv. J. Chim.* **8**, 275–278.
- Hall, S. R., Flack, H. D. & Stewart, J. M. (1992). Editors. *Xtal3.2 Reference Manual*. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
- Kirby, A. J. (1983). In *The Anomeric Effect and Related Stereoelectronic Effects at Oxygen*. Berlin: Springer.
- Molecular Structure Corporation (1991). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Prins, D. A. (1948). *J. Am. Chem. Soc.* **70**, 3955–3957.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.