

O(2)—H(O2)···N(7^v) bonds. The first is the more frequent for the interaction between the adenine base and phosphate (Sundaralingam, 1966). The sulfate can also interact in this way (Langer & Huml, 1978a; Langer, Huml & Lessinger, 1978). A mode of interaction similar to the second was observed between the phosphate and the protonated adenine base in nucleotides (Hecht & Sundaralingam, 1972) and for the sulfate (Langer & Huml, 1978a; Prusiner & Sundaralingam, 1972).

There are also hydrogen bonds between the adenine bases, *i.e.* N(9)—H(9)···N(3^{iv}), and the bond related by the center of symmetry. This mode of adenine base pairing was described for the first time for adeninium hemisulfate hydrate (Langer, Huml & Lessinger, 1978).

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The Crystal and Molecular Structure of α -D-manno-2-Heptulose

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Abstract

α -D-manno-2-Heptulose, C₇H₁₄O₇, is monoclinic, space group *P2*₁, with *a* = 6.626 (3), *b* = 7.049 (2), *c* = 9.499 (4) Å, β = 102.21 (4)°, *Z* = 2. The structure was refined to a final *R* of 0.026 for 837 counter reflexions. The molecule has the ⁵C₂ chair conformation. Both primary alcoholic side chains have the *gauche*–*trans* conformation. The six OH groups participate in intermolecular hydrogen bonding and form a five-link finite chain, with a single-link branch, beginning at the anomeric O and ending at the ring O atom.

Introduction

Of the class of 2-ketoheptoses (2-heptuloses) two members, α -D-*altro*-2-heptulose and α -D-*manno*-2-

heptulose occur in nature as free sugars. The latter compound is known to occur in the Avocado pear (*Persea officinalis*). As part of our research programme on the conformation and hydrogen bonding of monosaccharides, we undertook the structure determination of α -D-*manno*-2-heptulose.

Experimental

Crystals were grown at room temperature from a methanolic solution of a commercial product obtained through the Sigma Corporation. Photographs showed that the crystal is monoclinic with space group *P2*₁. Cell dimensions and intensities were measured on a Nonius CAD-3 diffractometer with Zr-filtered Mo *K* α radiation with the ω -scan technique. The crystal data are summarized in Table 1. Of 837 available reflexions

in two octants of the reflexion sphere with $\sin \theta/\lambda = 0.60 \text{ \AA}^{-1}$, 93 had $I < 2.5\sigma(I)$ and were considered unobserved. Lorentz-polarization corrections were applied, but none for absorption.

Structure determination and refinement

The structure was solved with *MULTAN 77* of the *XRAY* system (Stewart, 1976) and 270 $|E|$ values with $|E| > 1.00$. After block-diagonal least-squares refinement with anisotropic thermal parameters of the C and O atoms, a difference map showed the 14 H atoms with electron densities ranging from 0.20 to 0.60 e \AA^{-3} . The positional parameters of the H atoms, with constant isotropic thermal parameters equal to those of the

carrier atoms, were included in the refinement. Full-matrix refinement gave a final $R = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.026$ and $R_w = [\sum w(F_o - F_c)^2 / \sum wF_o^2]^{1/2} = 0.023$. The quantity minimized was $\sum w(F_o - F_c)^2$ with weights $w = \sigma^{-2}(F_o)$. The goodness of fit was 1.91, the average shift/error ratio for all parameters 0.003, the maximum shift/error ratio for C and O parameters was 0.011 and 0.027 for H parameters. A final difference synthesis showed no maxima $> 0.13 \text{ e \AA}^{-3}$. Scattering factors for C and O were taken from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965). The positional parameters are listed in Table 2.*

Description of the structure

The conformation of the molecule and the numbering of the atoms are shown in Fig. 1. Bond distances and

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34182 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Crystal data for α -D-manno-2-heptulose*

Molecular formula	$C_7H_{14}O_7$	$V (\text{\AA}^3)$	433.62
Molecular weight	210.2	Z	2
Crystal system	Monoclinic	Space group	$P2_1$
a (Å)	6.626 (3)	D_c (Mg m^{-3})	1.608
b (Å)	7.049 (2)	λ (Mo $K\alpha$) (Å)	0.71069
c (Å)	9.499 (4)	μ (Mo $K\alpha$) (mm^{-1})	0.092
β (°)	102.21 (4)	Systematic absences	$0k0, k = 2n + 1$

Table 2. *Fractional atomic coordinates ($\times 10^4$, for H $\times 10^3$) for α -D-manno-2-heptulose*

E.s.d.'s are given in parentheses and refer to the least significant figure.

	x	y	z
O(1)	10343 (3)	1746	280 (2)
O(2)	6735 (3)	3943 (4)	107 (2)
O(3)	6540 (3)	1171 (4)	3274 (2)
O(4)	3283 (3)	3512 (4)	3434 (2)
O(5)	5358 (3)	7142 (4)	3849 (2)
O(6)	8995 (2)	4172 (4)	2371 (2)
O(7)	11186 (3)	7614 (4)	2728 (2)
C(1)	8853 (4)	1263 (5)	1122 (3)
C(2)	7591 (4)	2974 (4)	1400 (3)
C(3)	5766 (4)	2401 (5)	2083 (3)
C(4)	4760 (4)	4128 (5)	2620 (3)
C(5)	6371 (4)	5411 (5)	3572 (3)
C(6)	8079 (4)	5899 (5)	2772 (3)
C(7)	9833 (4)	7034 (5)	3643 (3)
H(C1)	967 (4)	73 (4)	201 (3)
H'(C1)	794 (4)	27 (4)	60 (3)
H(C3)	476 (4)	165 (4)	135 (3)
H(C4)	403 (4)	492 (4)	183 (3)
H(C5)	700 (4)	478 (4)	452 (3)
H(C6)	744 (4)	660 (4)	189 (3)
H(C7)	1060 (4)	630 (4)	448 (3)
H'(C7)	933 (4)	811 (4)	407 (3)
H(O1)	966 (4)	207 (4)	-55 (3)
H(O2)	761 (4)	449 (4)	-16 (3)
H(O3)	590 (4)	23 (5)	312 (3)
H(O4)	221 (4)	371 (4)	304 (3)
H(O5)	574 (4)	745 (4)	467 (3)
H(O7)	1227 (4)	737 (5)	305 (3)

Table 3. *Bond distances (Å) for α -D-manno-2-heptulose*

E.s.d.'s are given in parentheses.

C(1)–C(2)	1.522 (4)	C(1)–H(C1)	0.98 (3)
C(2)–C(3)	1.542 (4)	C(1)–H'(C1)	0.99 (3)
C(3)–C(4)	1.527 (4)	C(3)–H(C3)	1.01 (2)
C(4)–C(5)	1.539 (4)	C(4)–H(C4)	0.98 (2)
C(5)–C(6)	1.529 (4)	C(5)–H(C5)	1.01 (3)
C(6)–C(7)	1.507 (4)	C(6)–H(C6)	0.99 (2)
C(1)–O(1)	1.437 (4)	C(7)–H(C7)	1.00 (3)
C(2)–O(2)	1.415 (3)	C(7)–H'(C7)	0.95 (3)
C(3)–O(3)	1.431 (4)	O(1)–H(O1)	0.85 (3)
C(4)–O(4)	1.437 (3)	O(2)–H(O2)	0.79 (3)
C(5)–O(5)	1.443 (4)	O(3)–H(O3)	0.78 (3)
C(6)–O(6)	1.447 (4)	O(4)–H(O4)	0.75 (3)
C(2)–O(6)	1.438 (3)	O(5)–H(O5)	0.80 (3)
C(7)–O(7)	1.434 (4)	O(7)–H(O7)	0.74 (3)

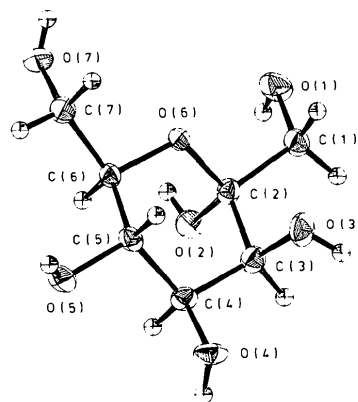


Fig. 1. Molecular conformation and atomic numbering of α -D-manno-2-heptulose. C and O atoms are represented at the 50% probability level.

angles are given in Tables 3 and 4. The C—C bonds range from 1.507 (4) to 1.542 (4) Å (mean 1.528 Å). The external C(6)—C(7) bond is short (1.507 Å) and the ring C(2)—C(3) is long (1.542 Å) compared with the values reported for C—C bonds of pyranoses (Arnott & Scott, 1972). The exocyclic C—O bonds range from 1.415 (3) to 1.443 (4) Å (mean 1.431 Å).

The anomeric C(2)—O(2) is short (1.415 Å) and the endocyclic C(2)—O(6) and C(6)—O(6) are 1.438 and 1.447 Å. These bond lengths which relate to the hemiacetal sequence C(6)—O(6)—C(2)—O(2)—H are consistent with observations on many α - and β -pyranoses (Arnott & Scott, 1972; Jeffrey & Takagi, 1977; Jeffrey,

Pople, Binkley & Vishveshwara, 1978) that the anomeric C—O bond is shortened in both α - and β -pyranoses and that, of the endocyclic C—O bonds, that at the anomeric C atom is the shorter.

The C—H lengths have an average value of 0.99 (3) Å, the O—H lengths average 0.79 (3) Å.

The majority of the bond angles (Table 4) are close to tetrahedral. Notable exceptions are C(3)—C(2)—O(2) (106.6°), C(1)—C(2)—O(6) (105.8°), C(7)—C(6)—O(6) (105.9°), C(2)—O(6)—C(6) (114.4°) and C(5)—C(6)—C(7) (114.4°). Values of bond angles at anomeric C(2) smaller than tetrahedral have also been reported for the ketohexoses β -D-fructose (Takagi & Jeffrey, 1977; Kanters, Roelofsen, Alblas & Meinders, 1977) and α -L-sorbose (Kim & Rosenstein, 1967). The large difference between the side-chain bond angles at C(6) (8.5°) is a general feature of saccharide structures (Arnott & Scott, 1972). The bond angles in the hemiacetal sequence, C(6)—O(6)—C(2) 114.4° and O(6)—C(2)—O(2) 111.1°, follow the general trend (Jeffrey & Takagi, 1977) that these valence angles are larger in α - than in β -pyranoses [mean values for α -pyranoses: 113.6 and 111.9°, for β -pyranoses: 112.1 and 106.8° (Jeffrey, Pople, Binkley & Vishveshwara, 1978)].

The mean values of bond angles involving H atoms are distributed over the different classes as follows: H—C—C 110, H—C—O 109, H—O—C 109 and H—C—H 107°.

The pyranose ring is a slightly distorted 5C_2 chair as shown by the range of the ring torsion angles (50.5–62.3°, Table 5), the deviations from the three least-squares planes through opposite bonds (Table 6) and by the Cremer & Pople (1975) ring-puckering parameters, which are $\theta = 4.5$, $\varphi = 309^\circ$, $Q = 0.558$, $q_2 = 0.044$ and $q_3 = 0.557$ Å.

The exocyclic torsion angles involving C and O atoms (Table 5) are close to the ideal *gauche* (60°) or *trans* (180°) arrangements, the average deviation being 8.5°. The same holds for the H—C—C—H torsion angles, whereas five of the seven H—C—O—H torsion angles display large departures (Table 7) from the ideal

Table 4. Bond angles (°) for α -D-manno-2-heptulose

E.s.d.'s are given in parentheses.

O(1)—C(1)—C(2)	112.1 (2)	O(1)—C(1)—H(C1)	105 (2)
C(1)—C(2)—C(3)	112.0 (3)	O(1)—C(1)—H'(C1)	108 (2)
C(1)—C(2)—O(2)	111.5 (2)	H(C1)—C(1)—H'(C1)	110 (2)
C(1)—C(2)—O(6)	105.8 (2)	C(2)—C(1)—H(C1)	112 (2)
C(3)—C(2)—O(6)	110.0 (2)	C(2)—C(1)—H'(C1)	110 (2)
O(2)—C(2)—C(3)	106.6 (2)	O(3)—C(3)—H(C3)	108 (2)
O(2)—C(2)—O(6)	111.1 (3)	C(2)—C(3)—H(C3)	108 (2)
C(2)—C(3)—C(4)	111.6 (3)	C(4)—C(3)—H(C3)	112 (2)
C(2)—C(3)—O(3)	108.1 (2)	O(4)—C(4)—H(C4)	108 (2)
O(3)—C(3)—C(4)	109.1 (2)	C(3)—C(4)—H(C4)	112 (2)
C(3)—C(4)—C(5)	111.5 (2)	C(5)—C(4)—H(C4)	107 (2)
C(3)—C(4)—O(4)	109.5 (3)	O(5)—C(5)—H(C5)	109 (2)
O(4)—C(4)—C(5)	109.2 (2)	C(4)—C(5)—H(C5)	112 (2)
C(4)—C(5)—C(6)	109.6 (2)	C(6)—C(5)—H(C5)	110 (2)
C(4)—C(5)—O(5)	108.2 (2)	O(6)—C(6)—H(C6)	109 (2)
O(5)—C(5)—C(6)	108.8 (2)	C(5)—C(6)—H(C6)	108 (2)
C(5)—C(6)—C(7)	114.4 (2)	C(7)—C(6)—H(C6)	110 (2)
C(5)—C(6)—O(6)	109.7 (2)	O(7)—C(7)—H(C7)	111 (2)
O(6)—C(6)—C(7)	105.9 (2)	O(7)—C(7)—H'(C7)	111 (2)
C(6)—C(7)—O(7)	109.1 (2)	H(C7)—C(7)—H'(C7)	104 (2)
C(2)—O(6)—C(6)	114.4 (2)	C(6)—C(7)—H(C7)	111 (2)
		C(6)—C(7)—H'(C7)	111 (2)
		C(1)—O(1)—H(O1)	107 (2)
		C(2)—O(2)—H(O2)	110 (2)
		C(3)—O(3)—H(O3)	106 (2)
		C(4)—O(4)—H(O4)	111 (2)
		C(5)—O(5)—H(O5)	110 (2)
		C(7)—O(7)—H(O7)	111 (3)

Table 5. Endo- and exocyclic torsion angles (°) for α -D-manno-2-heptulose

The torsion angle $A(1)-A(2)-A(3)-A(4)$ is viewed along $A(2)-A(3)$, with a clockwise rotation of $A(1)$ to $A(4)$ taken to be positive. E.s.d.'s are given in parentheses.

Endocyclic

O(6)—C(2)—C(3)—C(4)	51.8 (4)	C(3)—C(4)—C(5)—C(6)	52.7 (4)	C(5)—C(6)—O(6)—C(2)	62.3 (4)
C(2)—C(3)—C(4)—C(5)	-50.5 (4)	C(4)—C(5)—C(6)—O(6)	-56.9 (4)	C(6)—O(6)—C(2)—C(3)	-58.9 (4)

Exocyclic

O(1)—C(1)—C(2)—C(3)	170.6 (3)	O(3)—C(3)—C(4)—O(4)	-52.1 (3)	C(4)—C(5)—C(6)—C(7)	-175.7 (3)
O(1)—C(1)—C(2)—O(6)	-69.6 (3)	C(3)—C(4)—C(5)—O(5)	171.2 (3)	O(5)—C(5)—C(6)—C(7)	66.3 (4)
C(1)—C(2)—O(6)—C(6)	180.0 (3)	O(4)—C(4)—C(5)—C(6)	173.9 (3)	O(5)—C(5)—C(6)—O(6)	-174.9 (3)
O(6)—C(2)—C(3)—O(3)	-68.1 (3)	O(4)—C(4)—C(5)—O(5)	-67.7 (3)	C(7)—C(6)—O(6)—C(2)	-173.8 (3)
C(1)—C(2)—C(3)—C(4)	169.2 (3)	O(2)—C(2)—C(3)—C(4)	-68.7 (3)	C(5)—C(6)—C(7)—O(7)	-172.4 (3)
C(1)—C(2)—C(3)—O(3)	49.1 (4)	C(2)—C(3)—C(4)—O(4)	-171.5 (3)	O(6)—C(6)—C(7)—O(7)	66.7 (3)
O(2)—C(2)—C(3)—O(3)	171.3 (3)	O(3)—C(3)—C(4)—C(5)	68.9 (3)		

staggered arrangement of 60°, which would be favoured by the isolated molecule (Sundaralingam, 1968; Takagi & Jeffrey, 1977). The eclipsing of vicinal H atoms, which overrides this intramolecular constraint, is attributed to the participation of the OH groups in intermolecular hydrogen bonding (Fries, Rao & Sundaralingam, 1971; Kanters, Gaykema & Roelofsen, 1978; Kanters, Batenburg, Gaykema & Roelofsen, 1978).

The conformation of the primary C(1)—O(1) bond is *gauche* relative to O(6) and *trans* relative to C(3), that of the primary C(7)—O(7) bond is *gauche* relative to O(6) and *trans* relative to C(5), so both side-chain primary alcohol groups have the *gt* conformation.

Hydrogen bonding

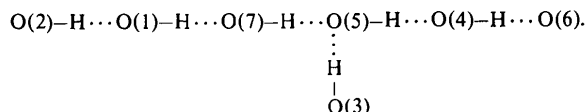
The six OH groups are involved in intermolecular hydrogen bonding. Each molecule is connected to eight

Table 6. *Least-squares planes of α -D-manno-2-heptulose, with displacements of the atoms from the plane (Å)*

Asterisks indicate atoms not included in the calculation of the plane. E.s.d.'s are given in parentheses.

C(2)	0.019 (3)	C(3)	-0.028 (3)	C(4)	0.011 (3)
C(3)	-0.019 (3)	C(4)	0.027 (3)	C(5)	-0.012 (3)
C(5)	0.019 (3)	C(6)	-0.029 (3)	O(6)	0.013 (2)
C(6)	0.020 (3)	O(6)	0.029 (2)	C(2)	-0.012 (3)
C(4)*	-0.633 (3)	C(2)*	-0.642 (3)	C(3)*	0.628 (3)
O(6)*	0.640 (2)	C(5)*	0.674 (3)	C(6)*	-0.682 (3)

neighbouring molecules by twelve hydrogen bonds. Three hydrogen bonds link molecules that are symmetry related by the 2_1 axis, the three remaining bonds are between molecules that are related by translational symmetry. The hydrogen-bond geometry is given in Table 8 and illustrated in Fig. 2. Two donors, O(2)—H and O(3)—H, do not accept a hydrogen bond; O(5) is twice an acceptor and ring O(6) accepts one hydrogen bond. The hydrogen-bond scheme consists of a finite chain starting at anomeric O(2) and ending at ring O(6) with a single-link branch from O(3)—H to O(5) which is shown in Fig. 2. The scheme is shown diagrammatically below:



In the chain three inner bonds are of the donor-acceptor $\cdots \text{O—H} \cdots \text{O—H} \cdots$ type. When corrected for the shortening of O—H lengths characteristic of X-ray data, compared with neutron diffraction O—H lengths (Jeffrey & Takagi, 1977), two of these inner bonds have short contacts (1.77 and 1.82 Å, Table 8) and presumably are the stronger hydrogen bonds in accordance with the donor-acceptor cooperative effect (Jeffrey & Takagi, 1978). From theory (Tse & Newton, 1977) it has been predicted that OH groups attached to anomeric C atoms are stronger than average hydrogen-bond donors and weaker than average hydrogen-bond acceptors and there is ample experimental evidence (Jeffrey & Takagi, 1978) in support of this prediction. As follows from Table 8, though anomeric O(2) is not

Table 7. *Torsion angles (°) involving hydrogen atoms for α -D-manno-2-heptulose*

E.s.d.'s are given in parentheses.

H(C3)—C(3)—C(4)—H(C4)	-53 (3)	H(C1)—C(1)—O(1)—H(O1)	172 (3)	H(C5)—C(5)—O(5)—H(O5)	17 (3)
H(C4)—C(4)—C(5)—H(C5)	169 (2)	H'(C1)—C(1)—O(1)—H(O1)	56 (3)	H(C7)—C(7)—O(7)—H(O7)	-6 (3)
H(C5)—C(5)—C(6)—H(C6)	-176 (2)	H(C3)—C(3)—O(3)—H(O3)	-6 (3)	H'(C7)—C(7)—O(7)—H(O7)	108 (3)
H(C6)—C(6)—C(7)—H(C7)	-174 (2)	H(C4)—C(4)—O(4)—H(O4)	12 (3)		
H(C6)—C(6)—C(7)—H'(C7)	71 (2)				

Table 8. *Hydrogen-bond distances (Å) and angles (°) in α -D-manno-2-heptulose*

	O—H	H...O	H...O*	O...O	O—H...O	Symmetry operation†
O(1)—H(O1)...O(7')	0.85 (3)	2.06 (3)	1.96	2.887 (3)	163 (3)	745.2
O(2)—H(O2)...O(1')	0.79 (3)	2.11 (3)	1.95	2.844 (3)	156 (3)	755.2
O(3)—H(O3)...O(5')	0.78 (3)	2.33 (3)	2.19	3.026 (4)	148 (3)	545.1
O(4)—H(O4)...O(6')	0.75 (3)	2.11 (3)	1.91	2.845 (2)	167 (3)	455.1
O(5)—H(O5)...O(4')	0.80 (3)	1.93 (3)	1.77	2.725 (3)	173 (3)	656.2
O(7)—H(O7)...O(5')	0.74 (3)	2.03 (3)	1.82	2.764 (2)	171 (3)	655.1

* Corrected by expanding the covalent O—H bond distances to the neutron diffraction value of 0.96 Å in the direction of the bond.

† The symmetry operation is performed on atom O(n'). The first three digits specify the lattice translations. The last digit indicates one of the following symmetry operations: (1) x, y, z ; (2) $-x, \frac{1}{2} + y, -z$ (e.g. 745.2 is $+2a - b$ from 555.2).

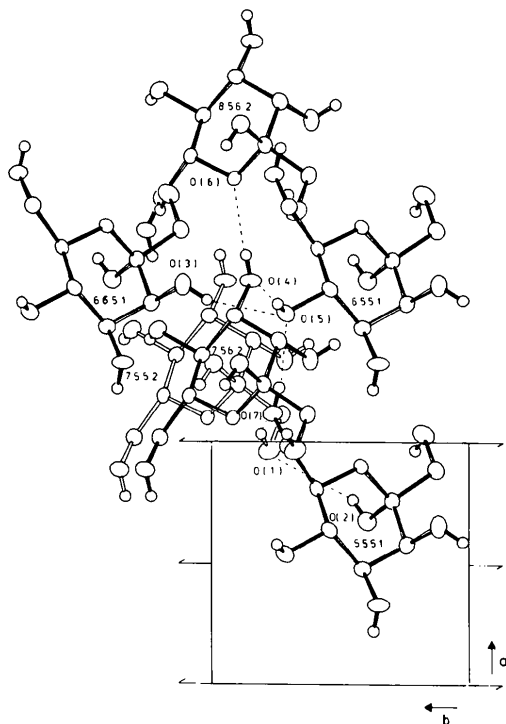


Fig. 2. Projection of the structure along c . The hydrogen bonds in the five-link chain with a single branch are indicated by dashed lines. The symmetry operations are defined in the footnote to Table 8. The aliphatic H atoms have been omitted.

an acceptor, which agrees with the above prediction, the donor-acceptor $O(2)-H \cdots O(1)$ contact of 1.95 Å is by no means indicative of a strong interaction. The donor-only $O(3)-H \cdots O(5)$ distance of 2.19 Å is much larger than the comparable bonds in monosaccharides (Jeffrey, Gress & Takagi, 1977), and together with the appreciable deviation of the $O(3)-$

$H \cdots O(5)$ angle from linearity, this hydrogen-bond interaction must be considered as a very weak bond.

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The Crystal and Molecular Structure of 4-O- β -D-Glucopyranosyl-D-glucitol

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Abstract

4-O- β -D-Glucopyranosyl-D-glucitol, $C_{12}H_{24}O_{11}$, is orthorhombic, space group $P2_12_12_1$, with $a = 5.295$ (1), $b = 7.770$ (1), $c = 35.514$ (6) Å and $Z = 4$.

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The structure was solved by direct methods and refined to $R(F) = 0.033$ for 1542 reflexions. The glucose ring has the 4C_1 chair conformation and the conformation of the primary alcohol group is *gauche-gauche*. The C-atom chain of the glucitol residue has the bent *Msc*, *ap*, *Psc* (*MAP*) conformation and differs from that observed in the *A* form of D-glucitol and in the D-glucitol-