In the 2-(2-hydroxyaryl)piperidine structures reported here, the orientation of the piperidine ring relative to the aryl moieties is given by the torsional angle N(1)—C(2)—C(9)—C(10), which is 37·4 (3)° in (I) and 44·8 (3)° in (II). Consequently, the hydroxyl group lies adjacent to the ring nitrogen with which it forms an intramolecular hydrogen bond. In (I), N(1)···O(1), N(1)···H(O1) and O(1)—H(O1) are 2·612 (2), 1·68 (2) and 1·00 (3) Å respectively with $\angle$N(1)···H(O1)—O(1) 155 (1)°. The analogous dimensions in (II) are 2·683 (3), 1·79 (3), 0·99 (3) Å and 148 (1)°. C(2) and O(1) are not quite planar with their associated aromatic rings as is illustrated by the torsional angles C(2)—C(9)—C(10)—O(1) of −2·4 (2) and −4·6 (3)° in (I) and (II) respectively.

The bond lengths and angles in (I) and (II) are in good agreement, and similar to values observed in comparable compounds. N(1)—C(6) is of mean length 1·468 (3) Å. This is significantly shorter than N(1)—C(2) of mean length 1·490 (3) Å, which compares well with the value 1·494 (4) Å reported for 4-hydroxy-3-piperidinecarboxylic acid; $\angle$C(2)—N(1)—C(6), mean value 111·9 (2)°, is close to 112·6 (2)° in the structure referred to above. In the cyano group, C(8)—N(2) and C(4)—C(8) bonds have mean lengths 1·139 (4) and 1·468 (4) Å respectively, and the C(4)—

C(8)—N(2) angle of 178·4 (3)° is almost linear. These values can be compared with the analogous lengths 1·135 (2) and 1·437 (2) Å and angle 177·93 (7)° observed in tetracyanoethylene (Becker, Coppens & Ross, 1973). The C—H lengths of the methyl groups range from 0·91 (4) to 1·07 (4) Å while the remainder vary between 0·97 (3) and 1·07 (3) Å.

The molecular packing is illustrated in Fig. 2. Both crystals are racemates, the molecules having asymmetric centres at C(2) and C(4). There are no unusually short intermolecular approaches in either structure.

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Methyl β-D-Glucoseptanoside, $C_7H_{14}O_6$

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Abstract. $M_r = 194·2$, monoclinic, $P2_1$, $a = 6·742$ (2), $b = 7·227$ (2), $c = 9·116$ (3) Å, $\beta = 104·49$ (2)°, $V = 430·0$ Å³, $Z = 2$, $D_m = 1·50$ (2) (by flotation in carbon tetrachloride/light petroleum), $D_x = 1·50$ (1) Mg m⁻³, $\mu(Cu K\alpha_1) = 1·096$ mm⁻¹, $F(000) = 212$, $T = 293$ K, $R = 0·027$ for 884 reflexions (including unobserveds). The septanose ring is close to a chair ($^2C_{5,6}$) conformation and all of the hydroxyl groups are involved in hydrogen bonding, including two bifurcated bonds, one hydrogen bond involving the glycosidic oxygen, and a weak intramolecular hydrogen bond.

Introduction. The title compound (1) is the first unsubstituted septanoside to be examined by X-ray

diffraction and it was chosen for study in order to compare its molecular conformation with that of the di-O-isopropylidene derivative and a mono-O-isopropylidene derivative (studies in hand).

Experimental. Crystals of (1) (Stevens, 1975), m.p. 397–399 K, obtained from an ethanol solution; a colourless plate crystal, 0·6 × 0·16 × 0·04 mm, was used for data collection and determination of the parameters (nine high-order reflexions); intensities of all 884 symmetry-independent reflexions $(\theta < 70°)$ measured on a Siemen's automatic single-crystal diffractometer using Ni-filtered Cu $K\alpha_1$ radiation with θ–2θ scans (Arndt & Willis, 1966); Lorentz,

polarization and absorption corrections applied; structure solved using *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974); all H atoms located in difference syntheses and their temperature parameters maintained throughout the refinement equal to those of the atoms to which they are bonded; structure refined by full-matrix least squares with a local version of *ORFLS* (Busing, Martin & Levy, 1962); all positional parameters, anisotropic temperature parameters for non-hydrogen atoms and an extinction parameter (Larson, 1970) were refined without constraint; scattering factors, including anomalous-dispersion corrections, were taken from *International Tables for X-ray Crystallography* (1974); the function minimized in the least-squares calculation was $\sum w(|F_o| - |F_c|)^2$ where $w = |\sigma(F_o)|^{-2}$ and $\sigma(I) = [N_P + N_B + (0.04N_P)^2]^{1/2}$, where N_P,N_B are number of counts in peak and background respectively; final $R = 0.027$ for all 884 reflexions, 0.026 for 864 observed reflexions $[I \geq 2.58\sigma(I)]$ and $R_w = 0.038$ for all reflexions, 0.037 for observed reflexions only; the maximum shift in the last cycle was 0.53σ with average shifts of 0.04σ and 0.130σ for non-hydrogen and H atoms respectively; the maximum absolute peak height in the final difference map was 0.16 e $\mathring{A}^{-3}$.

Discussion. The atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* The atom numbering scheme used in this study is given in Fig. 1 which also gives the bond lengths. Bond angles and torsional angles are listed in Table 2.

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* Lists of structure factors, anisotropic thermal parameters and selected non-bonded distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38332 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
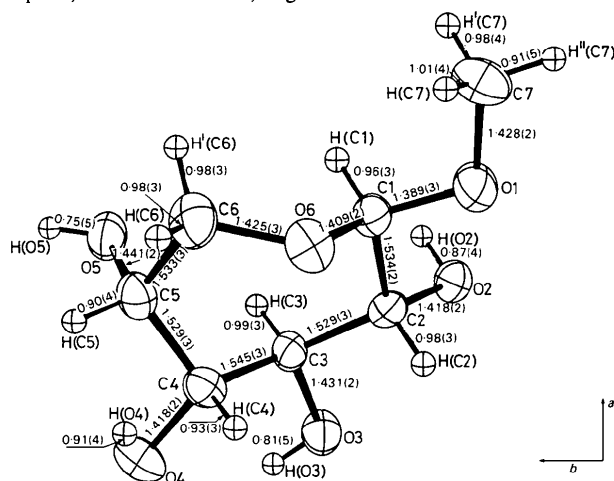

Fig. 1. An *ORTEP* plot (Johnson, 1965) of one molecule of the title compound showing atom numbering and bond lengths. Non-hydrogen atoms are represented by 50% probability ellipsoids and H atoms by spheres of radius 0.1 Å.

Table 1. *Atomic coordinates with e.s.d.'s and isotropic temperature parameters* ($\mathring{A}^2$)

For non-hydrogen atoms $B_{eq} = \frac{4}{3} \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$.

	x	y	z	B_{eq} or B
C(1)	0.5646 (3)	0.2867 (3)	0.7493 (2)	2.36
C(2)	0.3369 (2)	0.2598 (4)	0.6714 (2)	2.19
C(3)	0.2199 (3)	0.4398 (3)	0.6224 (2)	2.21
C(4)	0.2367 (3)	0.5837 (3)	0.7503 (2)	2.35
C(5)	0.4363 (3)	0.6944 (4)	0.7851 (2)	2.51
C(6)	0.6241 (4)	0.5833 (4)	0.8692 (2)	3.26
C(7)	0.8601 (3)	0.1054 (5)	0.8436 (3)	4.11
O(1)	0.6423 (2)	0.1106 (3)	0.7891 (2)	3.50
O(2)	0.3157 (2)	0.1363	0.5475 (2)	2.74
O(3)	0.0110 (2)	0.3845 (3)	0.5652 (2)	3.05
O(4)	0.0668 (2)	0.7058 (3)	0.7153 (2)	3.28
O(5)	0.4670 (2)	0.7676 (3)	0.6453 (2)	2.97
O(6)	0.5866 (2)	0.3908 (3)	0.8831 (1)	2.81
H(C1)	0.638 (5)	0.349 (5)	0.687 (3)	2.36
H(C2)	0.277 (5)	0.198 (6)	0.746 (3)	2.19
H(C3)	0.264 (5)	0.505 (5)	0.541 (3)	2.21
H(C4)	0.219 (5)	0.523 (6)	0.837 (3)	2.35
H(C5)	0.428 (5)	0.790 (6)	0.847 (4)	2.51
H(C6)	0.657 (5)	0.626 (7)	0.977 (4)	3.26
H1(C6)	0.730 (5)	0.616 (7)	0.818 (4)	3.26
H(C7)	0.895 (6)	0.170 (7)	0.945 (4)	4.11
H1(C7)	0.927 (6)	0.158 (7)	0.770 (4)	4.11
H11(C7)	0.897 (6)	−0.014 (7)	0.837 (5)	4.11
H(O2)	0.372 (5)	0.191 (6)	0.484 (4)	2.74
H(O3)	−0.060 (6)	0.477 (7)	0.557 (4)	3.05
H(O4)	0.049 (5)	0.748 (6)	0.618 (4)	3.28
H(O5)	0.484 (5)	0.869 (7)	0.645 (4)	2.97

Table 2. *Bond and torsion angles* (°)

C(1)–C(2)–C(3)	114.2 (2)	C(3)–C(4)–O(4)	110.3 (1)
C(2)–C(3)–C(4)	114.7 (1)	C(4)–C(5)–O(5)	108.8 (1)
C(3)–C(4)–C(5)	113.8 (1)	O(6)–C(1)–O(1)	108.2 (1)
C(4)–C(5)–C(6)	113.8 (2)	C(1)–O(1)–C(7)	113.7 (2)
C(5)–C(6)–O(6)	114.4 (2)	O(2)–C(2)–C(3)	111.2 (1)
C(6)–O(6)–C(1)	115.3 (1)	O(3)–C(3)–C(4)	110.0 (1)
O(6)–C(1)–C(2)	110.2 (1)	O(4)–C(4)–C(5)	109.9 (2)
C(1)–C(2)–O(2)	109.8 (1)	O(5)–C(5)–C(6)	110.5 (2)
C(2)–C(3)–O(3)	104.9 (2)	O(1)–C(1)–C(2)	105.8 (1)

C(1)–C(2)–C(3)–C(4)	−54.4 (2)	C(7)–O(1)–C(1)–C(2)	171.4 (2)
C(2)–C(3)–C(4)–C(5)	79.8 (2)	O(1)–C(1)–C(2)–O(2)	−56.1 (2)
C(3)–C(4)–C(5)–C(6)	−73.2 (2)	O(1)–C(1)–C(2)–C(3)	178.2 (1)
C(4)–C(5)–C(6)–O(6)	7.8 (2)	O(6)–C(1)–C(2)–O(2)	−172.9 (1)
C(5)–C(6)–O(6)–C(1)	74.2 (2)	O(2)–C(2)–C(3)–O(3)	59.8 (2)
C(6)–O(6)–C(1)–C(2)	−97.8 (2)	O(2)–C(2)–C(3)–C(4)	−179.3 (1)
O(6)–C(1)–C(2)–C(3)	61.5 (2)	O(3)–C(3)–C(4)–C(5)	−162.2 (2)
C(1)–C(2)–C(3)–O(3)	−175.2 (1)	O(3)–C(3)–C(4)–O(4)	−38.1 (2)
C(2)–C(3)–C(4)–O(4)	−156.1 (1)	O(4)–C(4)–C(5)–C(6)	162.6 (2)
C(3)–C(4)–C(5)–O(5)	50.6 (2)	O(4)–C(4)–C(5)–O(5)	−73.7 (2)
C(6)–O(6)–C(1)–O(1)	147.0 (2)	O(5)–C(5)–C(6)–O(6)	−115.0 (2)
O(6)–C(1)–O(1)–C(7)	−70.6 (2)		

The seven-membered ring in (1) has a conformation close to the chair $^2C_{5,6}$ (Stoddart, 1971) in which the exocyclic O(1), O(2), O(3) and O(4) are (pseudo)-equatorial. The geometry of the acetal grouping is similar to that observed for β-D-aldopyranosides, except that the torsional angle C(6)–O(6)–C(1)–O(1) of 147° is somewhat less than the 180° for β-pyranosides. Thus the C(1)–O(1) bond length is significantly shorter than both C(6)–O(6) and C(7)–O(1), and C(1)–O(6) is intermediate, in agreement with the theoretical predictions for a β-pyranoside-type structure (Jeffrey, Pople, Binkley & Vishveshwara, 1978).

In common with other septanoside derivatives that have been subjected to X-ray diffraction [see Choong, McConnell, Stephenson & Stevens (1980) for references], bond angles within the seven-membered ring are all greater than the tetrahedral angle. This is consistent with the results of a theoretical study on the 'parent' heterocycle, oxepane (Bocian & Strauss, 1977).

As with other polyhydroxy compounds (Jeffrey, 1973), there is extensive intermolecular hydrogen bonding involving the hydroxyl groups in the title compound (Table 3). All hydroxyl groups are both donors and acceptors. A symmetrical bifurcated hydrogen bond is formed between O(1), O(2) and H(O5) related translationally in the $-\mathbf{b}$ direction. This bond and the O(2)—H(O2)$\cdots$O(5^i) bond are responsible for the formation of zigzag chains parallel to $\mathbf{b}$. Each molecule is hydrogen bonded to six neighbouring molecules: four in the same chain, and two in a chain lying adjacent in the $-\mathbf{a}$ direction. The C(2) hydroxyl group is involved in bonding with three different molecules, accepting from O(3) in an adjacent chain and being both donor and acceptor to molecules in the same chain. This group and those attached to C(3) and C(4) interbond adjacent chains to form 'puckered' sheets lying parallel to the ab plane. All $D\cdots A$ distances are greater than 2·67 Å and are categorized as 'weak'

Table 3. *Hydrogen-bond distances (Å) and angles (°) with e.s.d.'s in parentheses*

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O(2) H(O2) O(5^i)	0·87 (4)	1·87 (4)	2·724 (2)	168 (4)
O(3) H(O3) O(4)	0·81 (5)	2·22 (4)	2·673 (2)	116 (4)
O(3) H(O3) O(2^{ii})	0·81 (5)	2·09 (4)	2·844 (2)	153 (4)
O(4) H(O4) O(3^{ii})	0·91 (4)	1·90 (4)	2·795 (2)	167 (4)
O(5) H(O5) O(1^{iii})	0·74 (5)	2·28 (5)	2·913 (2)	143 (4)
O(5) H(O5) O(2^{iii})	0·74 (5)	2·30 (5)	2·913 (2)	140 (4)

The superscripts refer to atoms related to those in Table 1 by the operations: (i) $1-x,-\frac{1}{2}+y,1-z$; (ii) $-x,\frac{1}{2}+y,1-z$; (iii) $x,1+y,z$.

(Brown, 1976), although the cooperative aspect of the bonding and the formation of chains of bonds implies a stable hydrogen-bonding scheme (Jeffrey & Takagi, 1978). The non-hydroxylic O(6) is not involved in hydrogen bonding. The hydroxylic H(O3) is involved in two interactions, one of which is a weak intramolecular hydrogen bond. Attention has been drawn elsewhere (Newton, Jeffrey & Takagi, 1979) to weak hydrogen bonds being part of a bifurcated hydrogen-bond system.

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The Structure of 2-(2-Hydroxy-5-methylphenyl)-1-propenyl Acetate, C12H14O3*

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Abstract. $M_r = 206·2$, monoclinic, $P2_1/c$, $a = 6·52$ (1), $b = 24·95$ (3), $c = 7·23$ (1) Å, $\beta = 112·0$ (1)°, $V = 1090$ Å^3, $D_x = 1·257$ Mg m^{-3} for $Z = 4$, $F(000) = 440$. Final R is 0·09 for 953 observed reflections. The structure was solved by direct methods. Two molecules combine to form a dimer through O—H$\cdots$O hydrogen bonds.

Introduction. Enol esters are naturally occurring compounds (Bohlmann, Niedballa & Schulz, 1969). The synthesis of related enol esters was undertaken in this laboratory by Dhekne & Rao (1980a,b). They

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