Table 2. *Hydrogen bonds and short contacts* (<3·5 Å) *with their standard deviations*

(*a*) Hydrogen bonds

Donor (*D*)	Acceptor (*A*)	*D*–*A* (Å)	H···*A* (Å)	∠*D*–H···*A* (°)
N(2)	Cl	3·101 (2)	2·13 (3)	167 (3)
N(4)	O(*W*)	2·856 (3)	1·89 (3)	157 (2)
O(*W*)	Cl^{i}	3·270 (2)	2·44 (3)	162 (3)
O(*W*)	$N(5^{ii})$	3·020 (3)	2·11 (4)	167 (3)
N(1)	$N(6^{iii})$	2·830 (3)	1·95 (3)	164 (3)
N(3)	Cl^{iv}	3·297 (2)	2·47 (2)	146 (2)

(*b*) Short contacts (Å) (<3·5 Å)

O(*W*)–C(6)	3·431 (3)	$N(1)–C(5^{v})$	3·417 (3)
O(*W*)–C(9)	3·380 (3)	$N(2)–C(3^{v})$	3·471 (3)
$N(1)–N(5^{ii})$	3·492 (3)	$N(2)–C(4^{v})$	3·442 (4)
$N(4)–N(6^{ii})$	3·440 (3)	$C(2)–C(3^{v})$	3·426 (3)
$N(1)–C(2^{v})$	3·457 (3)	$N(4)–C(7^{vi})$	3·390 (3)

Roman-numeral superscripts denote the following equivalent positions relative to the reference molecule at *x*,*y*,*z*.

(i) $1+x, y, z$
(ii) $x, \frac{3}{2}-y, \frac{1}{2}+z$
(iii) $x, y, 1+z$
(iv) $1-x, 1-y, -z$
(v) $1-x, 1-y, 1-z$
(vi) $2-x, 1-y, -z$

with respect to the imidazole ring. Although the observed conformation in the cimetidine monohydrochloride satisfies the latter condition, the N(2)···N(3) or N(2)···N(4) distance is significantly longer than 3·6 Å [N(2)···N(3) = 6·170 (3); N(2)···N(4) = 6·426 (3) Å]; this situation in cimetidine monohydrochloride may be in part responsible for its rather weak inhibitory effect of peptic ulceration (Kokubo, Morimoto & Morisaka, in preparation).

Fig. 3 shows the crystal packing viewed along the *b* axis. The water molecules and the Cl^- ions lie between neighboring guanidine groups and between neighboring imidazole and guanidine moieties, respectively. Hydrogen bonds and short contacts less than 3·5 Å are listed in Table 2. The O atom of the water molecule is hydrogen-bonded to N(4) and N(5) of the neighboring guanidine groups [N(4)–H(N4)···O(*W*) = 2·856 (3), O(*W*)–H(*Wb*)···N(5) = 3·020 (3) Å], and to the neighboring Cl^- ion [O(*W*)–H(*Wa*)···Cl = 3·270 (2) Å]. These hydrogen bonds stabilize the molecular arrangement along the *c* axis (Fig. 3). The Cl^- ion is further hydrogen-bonded to the N(3) atom of the neighboring guanidine group [Cl···N(3) = 3·297 (2) Å] and to the N(2) atom of the neighboring imidazole ring [Cl···N(2) = 3·101 (2) Å]; a cyclic dimer of cimetidine involving a 20-membered ring system *via* two Cl^- ions is thereby formed (see Fig. 3). In this dimer formation, the two head-to-tail-arranged cimetidine molecules are related to each other by a center of symmetry.

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1,2-*O*-Isopropylidene-α-D-glucofuranurono-6,3-lactone, $C_9H_{12}O_6$

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Abstract. $M_r = 216·19$, monoclinic, $P2_1$, $a = 7·647$ (1), $b = 6·162$ (1), $c = 11·227$ (3) Å, $\beta = 105·63$ (1)°, $Z = 2$, $V = 509·5$ Å^3, $D_x = 1·41$ Mg m^{-3}, λ(Cu *Kα*) = 1·5418 Å, μ(Cu *Kα*) = 1·024 mm^{-1}, $F(000) = 228$, $T = 293$ K, $R = 0·0487$ for 779 unique observed reflections [$F \geq 3\sigma(F)$]. The material was prepared by Mackie & Perlin [*Can. J. Chem.* (1965), **43**, 2921–2924] and recrystallized from $CHCl_3$/petroleum ether. The analysis has confirmed the structure of the title compound.

Introduction. The title compound and 5-hydroxy-1,2-*O*-isopropylidene-α-D-*xylo*-hexofuranurono-6,3-lactone (see following paper, Sheldrick, Mackie & Akrigg, 1983) were investigated to confirm their synthesis and to clarify the structure of the *gem* diol.

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Experimental. Crystal 0·20 × 0·15 × 0·11 mm, Enraf–Nonius CAD-4F diffractometer; no correction for absorption; $2\theta_{\max} = 140°$, h from −9 to 0, k from −7 to 7 and l from −13 to 13; 2125 reflections measured. The $\bar{3}06$ reflection used as an intensity check: average count 2248 and calculated $\sigma = 147$. Cell dimensions calculated from θ measurements of 60 reflections. Data merged using *SHELX* (Sheldrick, 1976) giving 869 unique reflections, 779 considered observed, merging $R_{\text{int}} = 0{\cdot}055$. *MULTAN*80 (Main *et al.*, 1980) used to solve the structure and least-squares refinement carried out, using *SHELX* for the positional parameters and anisotropic thermal parameters for all C and O; $\sum w(\Delta F)^2$ minimized, unit weights. H atoms from a difference Fourier synthesis, not refined. In the final cycle maximum least-squares shift/error 0·013, on final difference Fourier synthesis maximum and minimum electron density values +0·15 and −0·19 e Å^{-3}; scattering factors from *MULTAN*80 and *SHELX*.

Discussion. The molecule and numbering scheme are shown in Fig. 1, and the cell packing, projected along −**b**, is shown in Fig. 2. Atom coordinates and equivalent isotropic temperature factors are in Table 1* and bond lengths and angles in Table 2. The two outer rings are *trans* to each other, with the obtuse angles to the central ring being 110·5 (0·86)° [O(1)–C(7)–C(3)–C(2) to O(2)–C(3)–C(7)–C(6)] and 114·3 (0·89)° [O(2)–C(4)–C(6)–C(7) to O(3)–C(4)–C(6)–O(4)].

The bond C(4)–O(3) shows an anomeric effect, also noticeable in similar structures where a furanose ring and an isopropylidene ring are fused (Phillips & Trotter, 1977; Klaska, Jarchow, Gunther & Paulsen, 1978; Boeyens, Brink, Hall, Jordaan & Pretorius, 1977; Takagi & Jeffrey, 1979; Rettig & Trotter, 1977) being 1·382 (7) Å compared with the C(6)–O(4) bond length of 1·430 (7) Å.

In the crystal packing only one hydrogen bond is formed, from the hydroxyl group, [O(6)–H(60)] to the isopropylidene ring [O(4) in the molecule related by the transformation (0,−1,0)], thus forming continuous chains parallel to the b axis. The distance O(6)–O(4) is 2·870 (6) Å.

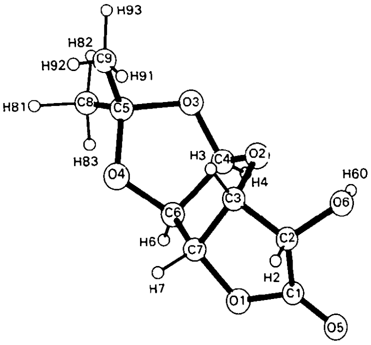

Fig. 1. View of the title compound with atom labels, drawn by the program *PLUTO*78 (Motherwell, 1978).

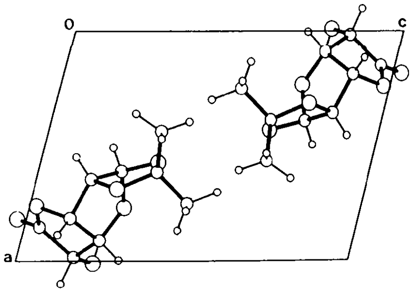

Fig. 2. The −*b*-axis projection of the unit cell (*PLUTO*78).

Table 1. *Atom coordinates* (×10⁴) *and equivalent isotropic temperature factors* (Å² × 10⁴)

$$U_{\text{eq}} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij} a^*_i a^*_j \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq}
O(1)	7603 (5)	3201 (9)	205 (3)	660
O(2)	7676 (6)	4163 (9)	2803 (4)	795
O(3)	5708 (6)	1984 (9)	3459 (4)	948
O(4)	6861 (5)	−688	2443 (3)	686
O(5)	8164 (6)	6627 (11)	−273 (4)	864
O(6)	10100 (5)	7090 (9)	2308 (4)	775
C(1)	8508 (8)	5148 (13)	451 (6)	683
C(2)	9832 (7)	5047 (13)	1687 (6)	653
C(3)	9097 (7)	3252 (12)	2327 (5)	619
C(4)	6087 (8)	2950 (13)	2445 (5)	697
C(5)	6129 (9)	−373 (12)	3497 (5)	683
C(6)	6434 (7)	1129 (13)	1625 (5)	616
C(7)	8135 (7)	1802 (11)	1287 (4)	570
C(8)	4361 (9)	−1538 (14)	3318 (6)	899
C(9)	7513 (9)	−850 (18)	4633 (6)	1020

Table 2. *Bond distances* (Å) *and angles* (°)

C(1)–C(2)	1·482 (8)	C(2)–O(6)	1·427 (7)
C(2)–C(3)	1·508 (7)	C(3)–O(2)	1·447 (6)
C(3)–C(7)	1·497 (7)	C(4)–O(2)	1·390 (7)
C(4)–C(6)	1·520 (8)	C(4)–O(3)	1·382 (7)
C(5)–C(8)	1·496 (8)	C(5)–O(3)	1·486 (8)
C(5)–C(9)	1·451 (9)	C(5)–O(4)	1·451 (6)
C(6)–C(7)	1·507 (6)	C(6)–O(4)	1·430 (7)
C(1)–O(1)	1·376 (7)	C(7)–O(1)	1·455 (6)
C(1)–O(5)	1·202 (7)		
O(1)–C(1)–C(2)	109·4 (5)	O(3)–C(5)–C(9)	108·9 (6)
O(1)–C(1)–O(5)	121·3 (6)	C(8)–C(5)–C(9)	116·1 (6)
C(2)–C(1)–O(5)	129·4 (6)	C(8)–C(5)–O(4)	111·1 (5)
C(1)–C(2)–C(3)	103·2 (5)	C(9)–C(5)–O(4)	109·8 (5)
C(1)–C(2)–O(6)	113·1 (5)	O(4)–C(6)–C(4)	103·6 (4)
O(6)–C(2)–C(3)	115·9 (5)	O(4)–C(6)–C(7)	108·3 (4)
C(2)–C(3)–O(2)	107·7 (5)	C(4)–C(6)–C(7)	104·6 (5)
C(2)–C(3)–C(7)	103·6 (4)	C(6)–C(7)–C(3)	104·1 (4)
C(7)–C(3)–O(2)	105·0 (4)	C(6)–C(7)–O(1)	107·9 (4)
O(2)–C(4)–O(3)	110·7 (5)	C(3)–C(7)–O(1)	105·4 (5)
O(2)–C(4)–C(6)	106·9 (4)	C(1)–O(1)–C(7)	109·7 (4)
C(6)–C(4)–O(3)	106·9 (4)	C(3)–O(2)–C(4)	111·7 (4)
O(3)–C(5)–O(4)	103·6 (5)	C(4)–O(3)–C(5)	110·7 (5)
O(3)–C(5)–C(8)	106·5 (5)	C(5)–O(4)–C(6)	110·4 (4)

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* Lists of structure amplitudes, H-atom parameters, anisotropic thermal parameters and details of least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38617 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Hydroxy-1,2-*O*-isopropylidene-α-D-*xylo*-hexofuranurono-6,3-lactone, $C_9H_{12}O_7$

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(*Received* 3 *March* 1983; *accepted* 18 *May* 1983)

Abstract. $M_r = 232·19$, orthorhombic, $P2_12_12_1$, $a = 5·621\,(1)$, $b = 11·826\,(2)$, $c = 15·598\,(2)$ Å, $V = 1036·86$ Å^3, $Z = 4$, $D_x = 1·49$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ(Cu $K\alpha$) = 1·024 mm^{-1}, $F(000) = 488$, $T = 293$ K, final $R = 0·0683$ for 841 unique observed reflections. Both hydroxyl groups of the *gem* diol show characteristic short O—H bond lengths, and both appear to be involved in hydrogen bonding.

Introduction. The title compound was prepared by Mackie & Perlin (1965) and NMR studies indicated the probable structure to be a *gem* diol. This investigation confirms the structure.

Experimental. Material prepared as described by Mackie & Perlin (1965), recrystallized from water/2-propanol, rectangular prism 0·24 × 0·15 × 0·12 mm, Enraf–Nonius CAD-4F diffractometer, Ni-filtered Cu $K\alpha$; cell parameters from 73 θ measurements; reflections measured for four octants to $2\theta = 140°$ for the ranges of h, k and l of 0 to 6, −14 to 14 and −19 to 19 respectively, 4262 reflections measured; $01\bar{5}$ used as intensity control (74 measurements): average count of 10297 distributed with a standard deviation of 342·6 (= 3·3%) and no systematic trend; no absorption correction; data merged using *SHELX* (Sheldrick, 1976) to give 1021 unique reflections with a merging $R_{\rm int} = 0·06$; 8 reflections with high F_c/F_o ratios, possibly due to extinction, removed and 172 reflections eliminated with $F < 3\sigma(F)$; structure solved with *MULTAN*80 (Main *et al.*, 1980), non-H atoms refined by least squares, first with isotropic thermal parameters, then anisotropic; H atoms of the methyl groups calculated at each stage, the remaining six H atoms found from a difference Fourier synthesis and refined with thermal-vibration parameters set at the equivalent $U_{\rm iso}$ of the attached atom; for final cycle, maximum shift/error 0·021, average 0·003; unit weights; difference Fourier synthesis showed a maximum value of 0·23 e Å^{-3} and a minimum of −0·26 e Å^{-3}; atomic scattering factors from *MULTAN*80 and *SHELX*.

Discussion. Fig. 1 shows the molecule and numbering scheme and Fig. 2 is an *a*-axis projection of the unit cell. Table 1* lists the atom coordinates and equivalent $U_{\rm iso}$'s; Table 2 gives bond distances and angles.

The structure shows that the compound is a *gem* diol and not some form of hydrate. Both hydroxyl groups have short O—H bond distances [0·634 (77) and 0·694 (75) Å], as do most *gem* diols, *e.g.* 5,5-dihydroxybarbituric acid (Singh, 1965), ninhydrin (Medrud, 1969) and tetrahydrofuran-3,3,4,4-tetrol (Mighell & Jacobson, 1964). Both form hydrogen bonds, O(6) to O(4′)($2-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) and O(7) to O(5′)($x+1$, y, z), with distances of 2·897 (13) and 2·791 (13) Å.

There are small differences in the obtuse angles between the two outer *trans* rings and the central ring,

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and least-squares-planes' details have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38618 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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