

Table 2. Selected geometric parameters (Å, °)

N1—H1	0.86 (2)	C8—C9	1.492 (3)
N1—C19	1.400 (2)	C9—O10	1.419 (3)
N1—C2	1.457 (3)	O10—C11	1.419 (3)
C2—C3	1.511 (3)	C11—C12	1.508 (3)
C3—O4	1.424 (3)	C12—N13	1.442 (3)
O4—C5	1.419 (3)	N13—H13	0.87 (2)
C5—C6	1.491 (3)	N13—C14	1.390 (3)
C6—O7	1.417 (3)	C14—C19	1.413 (3)
O7—C8	1.418 (3)		
C19—N1—C2—C3	174.4 (2)	C6—O7—C8—C9	-178.6 (2)
N1—C2—C3—O4	-53.2 (2)	O7—C8—C9—O10	67.9 (3)
C2—C3—O4—C5	-78.2 (2)	C8—C9—O10—C11	-162.1 (2)
C3—O4—C5—C6	160.8 (2)	C9—O10—C11—C12	156.6 (2)
O4—C5—C6—O7	-65.4 (2)	O10—C11—C12—N13	-64.4 (2)
C5—C6—O7—C8	177.3 (2)	C11—C12—N13—C14	-65.7 (3)

Isotropic H atoms were refined with a riding model, except for those attached to nitrogen, which were refined freely.

Data collection: *DIF4* (Stoe & Cie, 1988). Cell refinement: *DIF4*. Data reduction: local programs. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and local programs.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: AB1361). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 3-Deoxy-3-C-ethoxycarbonylmethyl-1,2:5,6-di-O-isopropylidene- $\alpha$ -D-allofuranose at 173 K

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## Abstract

The furanoid sugar ring in the title compound, C<sub>16</sub>H<sub>26</sub>O<sub>7</sub>, has a <sup>3</sup>T<sub>4</sub> conformation while the two O-isopropylidene rings have envelope conformations, with O(2) and C(5) lying out of the respective ring planes.

## Comment

Many naturally occurring compounds which possess interesting biological activities have furofuran structures. Notable among these are azadirachtin (Broughton, Ley, Slawin, Williams & Morgan, 1986; Ley, Santafianos, Blaney & Simmonds, 1987) and miharamycin A and B (Seto *et al.*, 1983). We recently described the synthesis and crystal structure of methyl 2,2'-anhydro-4,6-O-benzylidene-3-deoxy-3-C-[(R)-2-hydroxyethyl]- $\alpha$ -D-allopyranoside (Linden, Lee & Li, 1995). The title compound, (I), is a useful precursor to such ring systems, and we now describe its structure.

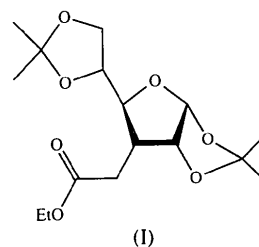


Fig. 1 depicts the correct absolute configuration of (I), which was assigned to agree with the known chirality of D-glucose, from which (I) was synthesized. The bond lengths and angles are normal. The molecule contains three five-membered rings which exhibit various conformations. The furanoid sugar ring has the <sup>3</sup>T<sub>4</sub> conformation (Altona & Sundaralingam, 1972), as indicated by the puckering parameters (Cremer & Pople, 1975)  $Q = 0.371(4)$  Å and  $\varphi_2 = 306.7(6)^\circ$ . The 1,2-O-isopropylidene ring has an envelope conformation [ $Q = 0.282(4)$  Å,  $\varphi_2 = 292.4(7)^\circ$ ] with O(2) lying 0.42 Å

above the plane through C(10), O(1), C(1) and C(2) ( $\chi^2 = 92$ ). The 5,6-*O*-isopropylidene ring also has an envelope conformation [ $Q = 0.262$  (4) Å,  $\varphi_2 = 46.2$  (9)°] with C(5) lying 0.40 Å below the plane through C(6), O(6), C(7) and O(5) ( $\chi^2 = 172$ ). The small distortion from planarity of the planar part of each isopropylidene ring is expressed by the non-zero torsion angles about the C(1)—O(1) and C(7)—O(6) bonds (Table 2).

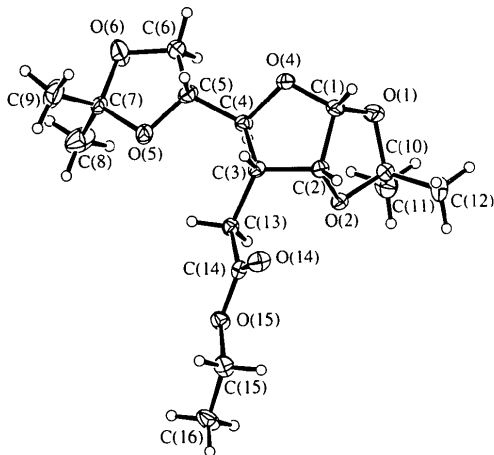


Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 25% probability level. H atoms are represented by spheres of arbitrary size.

The thermal displacement parameters for O(6) and the adjacent methyl groups of the 5,6-*O*-isopropylidene ring are slightly enlarged, which suggests that the part of the ring containing O(6) is quite flexible. However, there are no significant peaks of residual electron density in the vicinity of O(6), so that the possibility of the ring having two different envelope conformations can be excluded. This effect has also been observed in the corresponding 3-*C*-ethoxycarbonylmethylene derivative (Lee, Linden & Siew, 1995).

## Experimental

(I) was prepared by condensation of 1,2:5,6-di-*O*-isopropylidene-*D*-ribo-hexofuranos-3-ulose (Lawton, Szarek & Jones, 1969) with (ethoxycarbonylmethylene)triphenylphosphorane (Zhdanov, Alexeev & Alexeeva, 1972), instead of the potassium salt of triethylphosphonoacetate (Lourens & Koekemoer, 1975), followed by catalytic hydrogenation (10% Pd—C, H<sub>2</sub>) of the unsaturated branched-chain *cis*- and *trans*-3-*C*-ethoxycarbonylmethylene derivatives. Colourless prisms of (I) were grown in ether—light petroleum at room temperature, m.p. 326–327 K,  $[\alpha]_D +71.5^\circ$  ( $c$  1.0, CH<sub>2</sub>Cl<sub>2</sub>) {*cf.* m.p. 363–364 K,  $[\alpha]_D +67^\circ$  (CHCl<sub>3</sub>) (Lourens & Koekemoer, 1975)}.

## Crystal data

C<sub>16</sub>H<sub>26</sub>O<sub>7</sub>  
 $M_r = 330.37$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71069$  Å

## Tetragonal

$P4_1$   
 $a = 10.903$  (1) Å  
 $c = 14.813$  (2) Å  
 $V = 1760.9$  (4) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.246$  Mg m<sup>-3</sup>  
 $D_m$  not measured

## Cell parameters from 25 reflections

$\theta = 17.0$ – $20.0^\circ$   
 $\mu = 0.0970$  mm<sup>-1</sup>  
 $T = 173$  (1) K  
Prism  
 $0.40 \times 0.35 \times 0.25$  mm  
Colourless

## Data collection

Rigaku AFC-5R diffractometer  
 $\omega/2\theta$  scans  
Absorption correction: none  
2727 measured reflections  
2248 independent reflections  
1735 observed reflections  
[ $I > 2\sigma(I)$ ]  
 $R_{int} = 0.025$

$\theta_{max} = 27.5^\circ$   
 $h = 0 \rightarrow 14$   
 $k = -1 \rightarrow 14$   
 $l = -1 \rightarrow 19$   
3 standard reflections monitored every 150 reflections  
intensity decay: insignificant

## Refinement

Refinement on  $F$   
 $R = 0.0418$   
 $wR = 0.0353$   
 $S = 1.733$   
1735 reflections  
234 parameters  
H atoms: see text  
 $w = 1/[\sigma^2(F_o) + (0.005F_o)^2]$   
 $(\Delta/\sigma)_{max} = 0.0007$   
 $\Delta\rho_{max} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.21$  e Å<sup>-3</sup>

Extinction correction: Zachariasen (1963)  
Extinction coefficient:  $g = 3.8$  (12)  $\times 10^{-7}$   
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)  
Absolute configuration: assigned to agree with the known chirality of the precursor of (I)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$
O(1)	0.2356 (2)	0.4165 (2)	0.20940	0.062 (1)
O(2)	0.1506 (2)	0.5767 (2)	0.1358 (3)	0.0362 (7)
O(4)	0.1003 (2)	0.2947 (2)	0.1269 (3)	0.0437 (8)
O(5)	-0.2069 (2)	0.3344 (2)	0.0371 (3)	0.0486 (8)
O(6)	-0.2674 (2)	0.1664 (3)	0.1159 (3)	0.089 (1)
O(14)	0.0898 (2)	0.6198 (2)	-0.1192 (3)	0.0496 (8)
O(15)	-0.0647 (2)	0.7420 (2)	-0.0742 (2)	0.0392 (7)
C(1)	0.2044 (3)	0.3715 (3)	0.1236 (3)	0.045 (1)
C(2)	0.1681 (3)	0.4850 (3)	0.0683 (3)	0.035 (1)
C(3)	0.0449 (3)	0.4518 (3)	0.0269 (3)	0.0319 (9)
C(4)	-0.0057 (3)	0.3636 (3)	0.0983 (3)	0.0344 (9)
C(5)	-0.0987 (3)	0.2709 (3)	0.0657 (3)	0.044 (1)
C(6)	-0.1467 (3)	0.1865 (3)	0.1395 (4)	0.056 (1)
C(7)	-0.3096 (3)	0.2592 (3)	0.0580 (3)	0.041 (1)
C(8)	-0.4047 (4)	0.3360 (4)	0.1032 (5)	0.085 (2)
C(9)	-0.3563 (5)	0.1981 (5)	-0.0260 (4)	0.083 (2)
C(10)	0.2282 (3)	0.5478 (3)	0.2097 (3)	0.045 (1)
C(11)	0.1682 (4)	0.5909 (5)	0.2948 (4)	0.070 (2)
C(12)	0.3550 (3)	0.6022 (4)	0.1961 (4)	0.068 (2)
C(13)	-0.0385 (3)	0.5613 (3)	0.0074 (3)	0.0328 (9)
C(14)	0.0055 (3)	0.6409 (3)	-0.0690 (3)	0.0341 (9)
C(15)	-0.0363 (3)	0.8270 (3)	-0.1470 (3)	0.050 (1)
C(16)	-0.1156 (3)	0.9392 (4)	-0.1311 (4)	0.066 (2)

Table 2. Selected geometric parameters (Å, °)

O(1)—C(1)	1.404 (5)	O(6)—C(6)	1.379 (4)
O(1)—C(10)	1.433 (4)	O(6)—C(7)	1.404 (4)
O(2)—C(2)	1.427 (4)	C(1)—C(2)	1.536 (4)
O(2)—C(10)	1.419 (4)	C(2)—C(3)	1.521 (4)
O(4)—C(1)	1.411 (3)	C(3)—C(4)	1.533 (4)
O(4)—C(4)	1.441 (3)	C(3)—C(13)	1.527 (3)
O(5)—C(5)	1.433 (4)	C(4)—C(5)	1.511 (4)
O(5)—C(7)	1.422 (3)	C(5)—C(6)	1.522 (5)
C(1)—O(1)—C(10)	109.8 (3)	O(2)—C(2)—C(3)	109.4 (2)
C(2)—O(2)—C(10)	107.8 (2)	C(1)—C(2)—C(3)	104.6 (2)
C(1)—O(4)—C(4)	109.0 (2)	C(2)—C(3)—C(4)	100.8 (2)
C(5)—O(5)—C(7)	107.8 (2)	O(4)—C(4)—C(3)	104.0 (2)
C(6)—O(6)—C(7)	110.7 (3)	O(5)—C(5)—C(6)	102.8 (2)
O(1)—C(1)—O(4)	111.8 (3)	O(6)—C(6)—C(5)	104.0 (3)
O(1)—C(1)—C(2)	105.3 (2)	O(5)—C(7)—O(6)	106.9 (2)
O(4)—C(1)—C(2)	106.8 (2)	O(1)—C(10)—O(2)	104.6 (3)
O(2)—C(2)—C(1)	103.0 (3)		
O(1)—C(1)—C(2)—O(2)	-15.4 (3)	C(1)—O(4)—C(4)—C(3)	33.6 (3)
O(1)—C(10)—O(2)—C(2)	-31.3 (3)	C(1)—C(2)—O(2)—C(10)	28.8 (3)
O(2)—C(10)—O(1)—C(1)	20.9 (4)	C(1)—C(2)—C(3)—C(4)	29.3 (3)
O(4)—C(1)—C(2)—C(3)	-10.7 (3)	C(2)—C(1)—O(1)—C(10)	-3.2 (3)
O(4)—C(4)—C(3)—C(2)	-38.2 (3)	C(2)—C(1)—O(4)—C(4)	-14.3 (3)
O(5)—C(5)—C(6)—O(6)	27.7 (4)	C(5)—C(6)—O(6)—C(7)	-21.0 (5)
O(5)—C(7)—O(6)—C(6)	6.0 (5)	C(6)—C(5)—O(5)—C(7)	-24.7 (4)
O(6)—C(7)—O(5)—C(5)	13.0 (4)		

The H atoms were initially located in a difference electron density map, but their positions were subsequently geometrically idealized [ $d(\text{C—H}) = 0.95 \text{ \AA}$ ] and only their  $U$ 's were refined.

*MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991) was used for data collection and cell determination, and *TEXSAN* software (Molecular Structure Corporation, 1989) was used for data reduction, structure refinement and the preparation of publication material. The structure was solved using direct methods (*SHELXS86*; Sheldrick, 1990) and molecular graphics were produced using *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SX1012). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 2-Chlorobenzimidazole†

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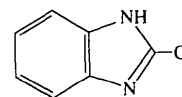
(Received 23 November 1994; accepted 22 December 1995)

### Abstract

The crystal structure of the title compound,  $\text{C}_7\text{H}_5\text{ClN}_2$ , is stabilized by an  $\text{N—H}\cdots\text{N}$  hydrogen bond,  $\text{C—H}\cdots\text{Cl}$  non-bonded intermolecular interactions and a  $\text{halogen}\cdots\text{halogen}$  interaction.

### Comment

The crystal and molecular structure of the title compound, (1), was investigated in order to determine the conformation and packing.



(1)

Bond distances and angles are in agreement with a related structure reported by Sprang & Sundaralingam (1973), *i.e.* 2-chloro-1-( $\beta$ -D-ribofuranosyl)benzimidazole. The average bond distance in the phenyl ring is  $1.383(6) \text{ \AA}$  and the molecule is planar within  $0.004(2) \text{ \AA}$  (Fig. 1). The H atom at the N9 position participates in an  $\text{N—H}\cdots\text{N}$  hydrogen bond with the symmetry-related  $\text{N7}^i$  atom at a distance of  $2.959(5) \text{ \AA}$  [ $\text{H9}\cdots\text{N7}^i$   $2.05(6) \text{ \AA}$  and  $\text{N9—H9}\cdots\text{N7}^i$   $172(5)^\circ$ ; symmetry code: (i)  $-\frac{1}{2} + x, \frac{3}{2} - y, 1 - z$ ] (Desiraju, 1991). The C2 atom forms a non-bonded interaction with the  $\text{Cl1}^i$  atom [ $\text{C2}\cdots\text{Cl1}^i$   $3.778(5)$ ,  $\text{H2}\cdots\text{Cl1}^i$   $3.04(4) \text{ \AA}$  and  $\text{C2—H2}\cdots\text{Cl1}^i$   $149(4)^\circ$ ]. Also, the C5 atom is involved in a  $\text{C—H}\cdots\text{Cl}$  non-bonded interaction with the  $\text{Cl1}^{ii}$  atom [ $\text{C5}\cdots\text{Cl1}^{ii}$   $3.771(5)$ ,  $\text{H5}\cdots\text{Cl1}^{ii}$   $2.86(4) \text{ \AA}$  and  $\text{C5—H5}\cdots\text{Cl1}^{ii}$   $155(3)^\circ$ ; symmetry code: (ii)  $\frac{1}{2} + x$ ,

† Contribution No. 1423 of the Instituto de Química, UNAM.