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r-2,*c*-6-Bis(4-chlorophenyl)-3,5-dimethyltetrahydropyran-*t*-4-ol

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The crystal and molecular structure of the title compound, $C_{19}H_{20}Cl_2O_2$, reveals a chair conformation for the pyran ring in which the hydroxyl group is axially oriented. All the other substituents occupy equatorial positions. There are two molecules in the asymmetric unit.

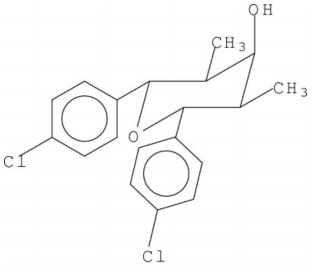
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.053
wR factor = 0.146
Data-to-parameter ratio = 15.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Pyran derivatives are important constituents of many vitamin derivatives and antiulcer drugs, such as alpha tocopherol (vitamin E) (Finar, 1989) and methantheline (Parimoo, 1999). Persubstituted pyrans are easily synthesized, and their ring conformations can serve as comparative models for their cyclohexane counterparts. Molecules with a pyran-derived ring can assume planar (Kumar & Errington, 1999), twist boat (Usman *et al.*, 2002), sofa (Ray *et al.*, 1998) and chair (Belakhov *et al.*, 2002) conformations, depending upon the level of unsaturation and the nature of the substituents on the ring. This investigation was undertaken to assign the conformation of the pyran ring and the orientation of the hydroxyl group in the title molecule, (I).



(I)

There are two molecules in the asymmetric unit of (I). The pyran rings are in chair conformations, as shown in Fig. 1. The torsion angles around the bonds involving the ring atoms deviate from the value of 56° expected for a perfect chair conformation (Kalsi, 1997). The bond lengths within the pyran ring are in the range 1.425 (3)–1.540 (4) Å (for molecule 1 containing atom O1) and 1.447 (3)–1.540 (3) Å (for molecule 2 containing atom O1*A*). The ranges of bond angles are 108.56 (19)–114.78 (18)° (for molecule 1) and 107.4 (2)–112.57 (17)° (for molecule 2). The configurations of chiral atoms C2, C3, C5 and C6 in molecule 1 are found to be *S*, *S*, *S* and *R*, respectively, which are identical to those of their counterparts in molecule 2. However, the compound crystallizes as a racemic mixture in the space group $P2_1/c$. The relative orientations of the two chlorophenyl groups differ in the two molecules, as shown by the dihedral angles between

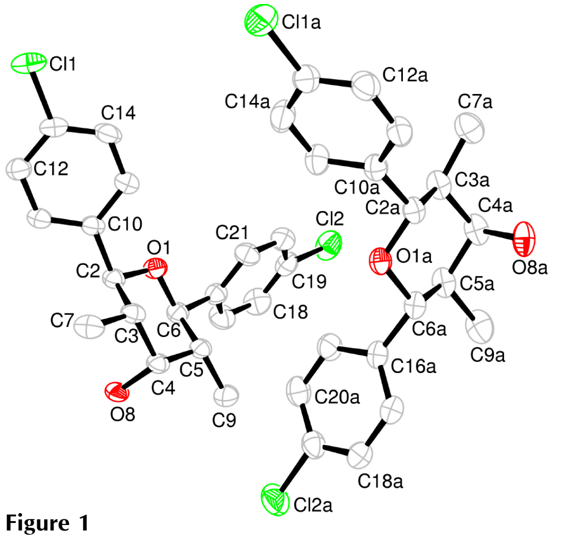


Figure 1
The asymmetric unit, with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity.

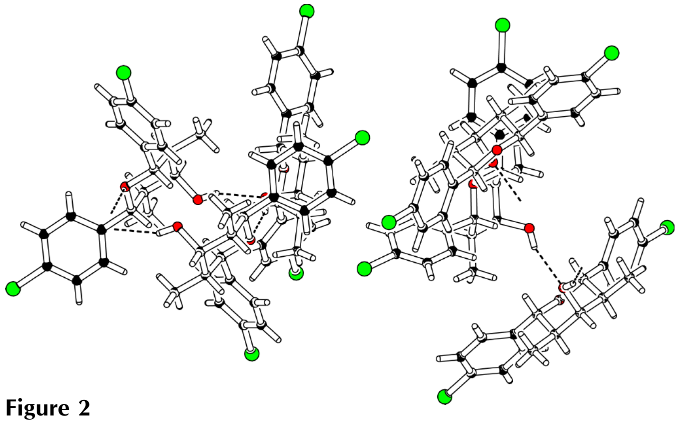


Figure 2
Packing diagram showing the layer structure formed by the intermolecular association of molecules *via* hydrogen bonds.

the two rings of 44.01 (1)° in molecule 1 and 51.01 (1)° in molecule 2. The equatorial orientations of all substituents except the hydroxyl groups are confirmed by the torsion angles close to 180°; compare the external atom and the other three ring atoms as observed in the pentasubstituted cyclohexan-1-one derivative (Sarangarajan *et al.*, 2002). The hydroxyl groups are oriented axially, as confirmed by the average of the C7—C3—C4—O8 torsion angles of 61.85 (3)°, which is close to the ideal value of 60° (Nasipuri, 1992).

In the crystal structure, the molecules are assembled in layers. Adjacent layers interact through O—H···O intermolecular hydrogen bonding involving atoms O8 and O1*A*($1-x, \frac{1}{2}+y, \frac{1}{2}-z$), at a distance of 2.850 (3) Å with an angle of 163 (3)° around atom H8. There is another interaction between O8*A* and O8($x, \frac{3}{2}-y, \frac{1}{2}+z$), at a distance of 2.833 (3) Å with an angle of 152 (3)° around atom H8*A* (Fig. 2 and Table 2).

Experimental

The title compound, (I), was obtained by the reduction of tetrahydropyran-4-one with aluminium isopropoxide in propan-2-ol. The white solid was separated by chromatography over silica gel and eluted with a hexane–dichloromethane mixture (1:10). Diffraction quality crystals of (I) were obtained by recrystallization from a dichloromethane–hexane mixture.

Crystal data

$C_{19}H_{20}Cl_2O_2$
$M_r = 351.25$
Monoclinic, $P2_1/c$
$a = 12.1315$ (9) Å
$b = 11.7075$ (19) Å
$c = 26.177$ (3) Å
$\beta = 99.728$ (9)°
$V = 3664.4$ (8) Å^3
$Z = 8$
$D_x = 1.273$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 2$–12°
$\mu = 0.36$ mm^{-1}
$T = 298$ K
Plate, colourless
0.2 × 0.2 × 0.1 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.650$, $T_{\max} = 0.999$
7004 measured reflections
6670 independent reflections
5034 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 25.3$°
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = -31 \rightarrow 31$
3 standard reflections every 100 reflections intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.146$
$S = 1.06$
6670 reflections
423 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 2.0872P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.50$ e Å^{-3}
$\Delta\rho_{\min} = -0.42$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0046 (5)

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.431 (3)	O1*A*—C2*A*	1.447 (3)
O1—C2	1.425 (3)	O1*A*—C6*A*	1.447 (3)
O8—C4	1.437 (3)	O8*A*—C4*A*	1.431 (3)
C2—O1—C6	114.78 (18)	O1—C6—C16	106.96 (19)
C2*A*—O1*A*—C6*A*	112.57 (17)	O1*A*—C2*A*—C10*A*	109.24 (19)
O1—C2—C10	106.90 (19)	O1*A*—C6*A*—C16*A*	108.33 (18)
C2—O1—C6—C16	178.0 (2)	C9—C5—C6—O1	−178.8 (2)
C2*A*—O1*A*—C6*A*—C16*A*	178.74 (18)	C10*A*—C2*A*—C3*A*—C4*A*	177.1 (2)
C10—C2—C3—C4	−174.7 (2)	C7*A*—C3*A*—C4*A*—C5*A*	−175.0 (2)
C7—C3—C4—C5	−177.6 (2)	C7*A*—C3*A*—C4*A*—O8*A*	62.8 (3)
C7—C3—C4—O8	60.9 (3)	O8*A*—C4*A*—C5*A*—C6*A*	64.4 (3)
O8—C4—C5—C6	63.4 (2)	C9*A*—C5*A*—C6*A*—O1*A*	178.4 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O8—H8···O1*A*$^{\rm i}$	0.84 (4)	2.04 (4)	2.850 (3)	163 (3)
O8*A*—H8*A*···O8$^{\rm ii}$	0.81 (3)	2.09 (3)	2.833 (3)	152 (3)
C4*A*—H4*A*···Cl1$^{\rm iii}$	0.98	2.61	3.563 (3)	164
C6*A*—H6*A*···O8*A*$^{\rm iv}$	0.98	2.56	3.380 (3)	141

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

The H atoms of the hydroxyl groups were located from a difference Fourier map and their positions refined. The O—H distances

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were computed to be 0.84 (4) and 0.81 (3) Å. H atoms attached to C atoms were included in calculated positions, with displacement parameters fixed at 1.2 U_{eq} of their carrier atoms. The displacement parameters of the H atoms of both the hydroxyl groups were fixed at $1.5U_{eq}$ of the respective O atoms during the refinement.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 1990).

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