

***r*-2,*c*-6-Bis(4-chlorophenyl)-*t*-3,*t*-5-dimethyl-tetrahydropyran-4-one**

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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.051

wR factor = 0.140

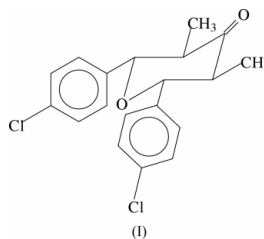
Data-to-parameter ratio = 15.2

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

The molecular structure of the title compound, C₁₉H₁₈Cl₂O₂, reveals a distorted chair conformation for the pyran ring, in which the methyl and 4-chlorophenyl groups occupy equatorial positions. The molecule is devoid of strong intramolecular interactions. In the crystal structure, the molecules form zigzag layers which are held together by C—H··· π interactions.

Comment

The title molecule, (I), contains two pairs of chiral C atoms with identical groups on each. There can be as many as four racemic modifications and two *meso* forms for the molecule (Eliel, 1962). This investigation was undertaken to assign the configuration and conformation of the most stable form in the solid state.



The saturated pyran ring adopts a distorted chair conformation, as shown by the torsion angles around the bonds involving the ring atoms. These torsion angles deviate from the ideal value of 56° reported for the chair conformation of cyclohexane (Kalsi, 1997). The C—C bond lengths of the aryl rings are in the range 1.362 (4)–1.391 (4) Å, while the bond angles are in the range 118.3 (3)–121.4 (4)°. The configurations of the chiral atoms C2, C3, C5 and C6 are found to be *R*, *S*, *R* and *S*, respectively. The equatorial dispositions of the methyl and 4-chlorophenyl groups are revealed by the torsion angles involving the exo atom and the other three ring atoms; these vary from –167.9 (2) to 178.3 (2)°, as observed also in a pentasubstituted cyclohexan-1-one derivative (Sarangarajan *et al.*, 2002).

In the crystal structure, the molecules are aggregated into zigzag layers extended over the *ac* plane. Within the layer, a Cl···Cl short contact of 3.406 (2) Å is observed between atoms Cl1 and Cl2ⁱ [symmetry code: (i) $-\frac{1}{2} - x, -y, -\frac{1}{2} + z$]. Adjacent layers are linked through C—H··· π interactions, *viz.* C3—H3···Cg, with H3···Cg = 2.72 Å, C3···Cg = 3.631 (3) Å and C3—H3···Cg = 154°, where Cg denotes the centroid of the C16–C21 aryl ring of the molecule at $(-x, \frac{1}{2} + y, \frac{1}{2} - z)$. The molecules are also held together by weak interactions (Jeffrey & Saenger, 1991) between C2 and O8ⁱⁱ [3.373 (4) Å; symmetry code: (ii) $-x + \frac{1}{2}, y - \frac{1}{2}, z + \frac{1}{2}$].

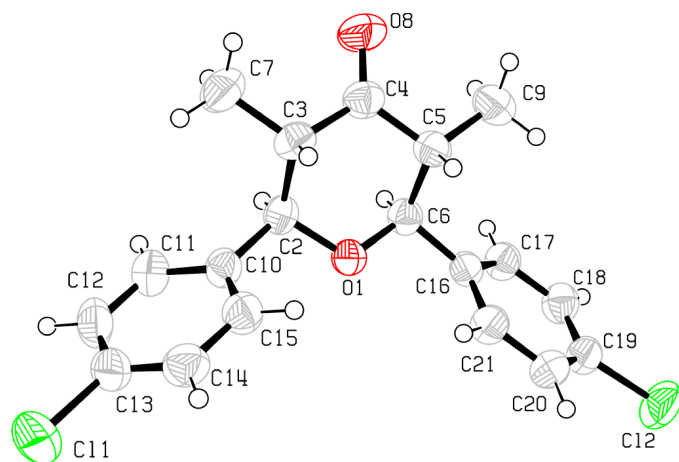


Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

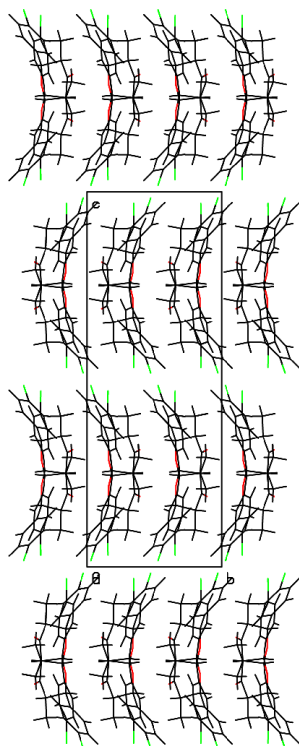


Figure 2
A view of the crystal packing, showing the zigzag layer formation.

The title compound is isomorphous with the analogous *p*-tolyl derivative, *r*-2,*c*-6-bis(*p*-tolyl)-*t*-3,*t*-5-dimethyltetrahydropyran-4-one (Krishnamoorthy *et al.*, 2003), with very similar crystal and molecular structures. The torsion angles around the C–C bonds of the tetrahydropyran ring are not significantly different in the two structures. This reveals no conformational changes due to the replacement of methyl by Cl atoms in the title molecule. The gas-phase conformation obtained from AM1 calculations is very similar to that observed in the solid state. The calculated heat of formation, $-48.5 \text{ kcal mol}^{-1}$, for the compound reveals its inherent molecular stability.

Experimental

The title compound was prepared by the condensation of pentan-3-one and 4-chlorobenzaldehyde in a 1:2 molar ratio in methanol, as reported by Bahiah & Mangalam (1978). Diffraction-quality crystals were obtained by recrystallization of the crude product from ethanol.

Crystal data

$\text{C}_{19}\text{H}_{18}\text{Cl}_2\text{O}_2$
 $M_r = 349.23$
 Orthorhombic, *Pbca*
 $a = 15.013 (1) \text{ \AA}$
 $b = 9.1230 (16) \text{ \AA}$
 $c = 25.462 (4) \text{ \AA}$
 $V = 3487.4 (9) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.330 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 2\text{--}12^\circ$
 $\mu = 0.38 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Plate, colourless
 $0.20 \times 0.15 \times 0.10 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.934$, $T_{\max} = 0.963$
 3164 measured reflections
 3163 independent reflections
 2205 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.041$
 $\theta_{\text{max}} = 25.3^\circ$
 $h = -18 \rightarrow 0$
 $k = 0 \rightarrow 10$
 $l = 0 \rightarrow 30$
 3 standard reflections every 100 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.140$
 $S = 1.10$
 3163 reflections
 208 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 2.3284P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1–C6	1.421 (3)	C3–C7	1.518 (4)
O1–C2	1.431 (3)	C4–C5	1.511 (4)
O8–C4	1.207 (3)	C5–C9	1.523 (4)
C2–C3	1.537 (4)	C5–C6	1.546 (4)
C3–C4	1.512 (4)		
C6–O1–C2	113.28 (19)	C5–C4–C3	114.7 (2)
O1–C2–C3	111.3 (2)	C4–C5–C6	107.7 (2)
C4–C3–C2	110.3 (2)	O1–C6–C5	109.6 (2)
C6–O1–C2–C3	$-59.8 (3)$	C3–C4–C5–C6	51.8 (3)
O1–C2–C3–C4	49.2 (3)	C2–O1–C6–C5	64.5 (3)
C2–C3–C4–C5	$-48.0 (3)$	C4–C5–C6–O1	$-57.9 (3)$

The H atoms were included in calculated positions. The displacement parameters of the methyl H atoms were fixed as $1.5U_{\text{eq}}$ of the respective C atoms. The displacement parameters of all the other H atoms were fixed as $1.2U_{\text{eq}}$ of the carrier atoms during refinement.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *MOLLEN* (Fair, 1990); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1997); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1983).

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