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r-2,*c*-6-Bis(*p*-tolyl)-*t*-3,*t*-5-dimethyltetrahydropyran-4-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.074
wR factor = 0.198
Data-to-parameter ratio = 14.6

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

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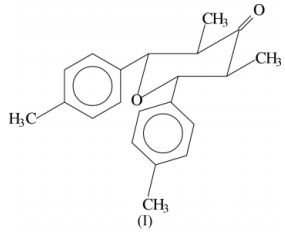
The molecular structure of the title compound, $C_{21}H_{24}O_2$, reveals a chair conformation for the pyran ring, in which the methyl and *p*-tolyl groups occupy equatorial positions. A C—H$\cdots\pi$ interaction is observed between an H atom of the tetrahydropyran ring and one of the aromatic rings.

Comment

Pyran-4-one derivatives are numerous, naturally occurring and biologically active (Noller, 1966). Several substituted tetrahydropyran-4-one derivatives are easily synthesized (Japp & Maitland, 1904). Saturated and partially saturated pyrans can assume different conformations depending on the level of unsaturation and the nature of the substituents on the ring, *viz*. planar (Kumar *et al.*, 1999), twist boat (Usman *et al.*, 2002), sofa (Ray *et al.*, 1998) or chair (Belakhov *et al.*, 2002) conformation. Our interest in this class of compounds stems from their structures, conformations and their possible biological functions.



(I)

The saturated pyran ring in the title molecule, (I), is in a chair conformation, as shown by the torsion angles around the bonds involving the ring atoms (Table 1). The torsion angles deviate from the value of 56° expected for a perfect chair conformation (Kalsi, 1997). Within the *p*-tolyl group, the ranges of bond lengths and angles are 1.371 (7)–1.389 (6) Å and 117.3 (4)–121.9 (4)°, respectively. The equatorial dispositions of methyl and *p*-tolyl groups are revealed by the C7—C3—C2—O1, C9—C5—C6—O1, C10—C2—O1—C6 and C16—C6—O1—C2 torsion angles of 173.5 (4), 178.0 (4), 178.5 (4) and −172.5 (4)°, respectively; the ideal value for these angles is 180° (Nasipuri, 1992). The bonds C3—C7 and C5—C9 are nearly parallel to the carbonyl group (McCullough *et al.*, 1999), as revealed by the corresponding torsion angles, which are close to 0°. The chiral atoms C2, C3, C5 and C6 adopt *R*, *S*, *R* and *S* configurations, respectively. The gas-phase conformation obtained from *AM*1 calculations (Stewart, 1990) is very similar to that observed in the solid state. The inherent molecular stability of the compound is revealed by its calculated heat of formation, −49.7 kcal mol^{-1}.

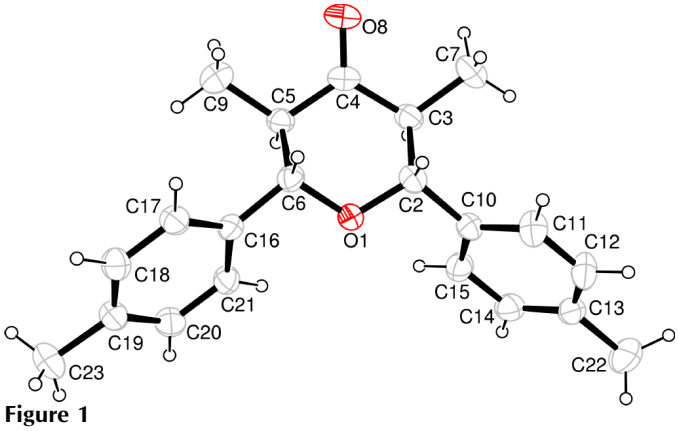


Figure 1
The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. One of the methyl H atoms is hidden.

In the crystal structure, the molecules are aggregated into corrugated layers and one of the aryl rings is involved in a C—H···π interaction [H3···*CgA* = 2.71 Å, C3···*CgA* = 3.623 (6) Å and C3—H3···*CgA* 155°, where is *CgA* is the centroid of the ring (C16–C21) of the molecule at $(-\frac{1}{2}+x, \frac{1}{2}-y, 1-z)$.

Experimental

The title compound was obtained by the condensation of pentan-3-one and *p*-tolualdehyde in a 1:2 molar ratio in 95% ethanol, as reported in the literature (Baliah & Mangalam, 1978). Diffraction-quality crystals were obtained by recrystallization of the crude product from ethanol.

Crystal data

$C_{21}H_{24}O_2$
M_r = 308.40
Orthorhombic, *Pbca*
a = 9.0894 (18) Å
b = 25.813 (3) Å
c = 15.1605 (10) Å
V = 3557.0 (8) Å^3
Z = 8
D_x = 1.152 Mg m^{-3}
Cu *K*α radiation
Cell parameters from 25 reflections
θ = 2–12°
μ = 0.57 mm^{-1}
T = 293 (2) K
Plate, colourless
0.2 × 0.2 × 0.1 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
3094 measured reflections
3094 independent reflections
1561 reflections with $I > 2\sigma(I)$
θ_{max} = 67.9°
$h = -10 \rightarrow 0$
$k = 0 \rightarrow 31$
$l = 0 \rightarrow 18$
3 standard reflections
every 10 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.198
S = 1.11
3094 reflections
212 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0255P)^2 + 7.7276P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.419 (5)	C4—O8	1.218 (5)
O1—C6	1.427 (5)	C4—C5	1.508 (6)
C2—C10	1.508 (6)	C5—C9	1.525 (6)
C2—C3	1.545 (6)	C5—C6	1.544 (6)
C3—C4	1.509 (6)	C6—C16	1.496 (6)
C3—C7	1.530 (6)		
C2—O1—C6	113.9 (3)	C5—C4—C3	115.5 (4)
O1—C2—C3	111.4 (4)	C4—C5—C6	108.0 (4)
C4—C3—C2	110.8 (4)	O1—C6—C5	109.6 (4)
C6—O1—C2—C3	−59.1 (5)	C3—C4—C5—C6	50.3 (5)
O1—C2—C3—C4	47.3 (5)	C2—O1—C6—C5	64.6 (5)
C2—C3—C4—C5	−45.8 (6)	C4—C5—C6—O1	−57.1 (5)

The reflection (020) was removed during the refinement as the observed and calculated structure factors showed very large disagreement. The reflections (5,19,7) and (5,6,12) were also removed as they fit badly. All H atoms were included in calculated positions, with U_{iso} values fixed at 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for all other H atoms.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1998); software used to prepare material for publication: *SHELXL*97.

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