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trans-2,2-Dimethyl-6-phenyl-3,4,5,6-tetrahydro-2*H*-pyran-3-ol

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.068
wR factor = 0.140
Data-to-parameter ratio = 15.1

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

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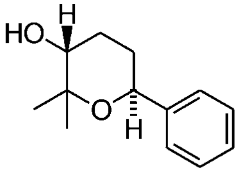
The tetrahydropyran ring in the title compound, $C_{13}H_{18}O_2$, adopts a $_1C^4$ conformation, with the phenyl and the hydroxy substituent located in equatorial positions. Hydrogen bonding occurs between enantiomers involving the hydroxy H atom and the ether O atom.

Comment

The title compound, (I), is a chiral molecule with two asymmetrically substituted C atoms, *viz.* C3 and C6. The racemate of (I) was prepared (Hartung *et al.*, 2003) and investigated by X-ray diffraction in order to establish reference data for an upcoming conformational analysis on multiply substituted tetrahydropyrans.



(I)

Atoms C2, C3, C5 and C6 form a plane [deviation of atom C6 is 0.031 (5) Å], with atoms O1 and C4 displaced in opposite directions [−0.603 (3) Å for O1 and 0.657 (4) Å for C4], thus leading to a $_1C^4$ conformation (Fig. 1). This geometry corresponds to the major conformation of (I) in $CDCl_3$ solution, as deduced from the magnitude of the vicinal proton–proton coupling constants (^{1}H NMR; Hartung *et al.*, 2003). The phenyl and hydroxy substituent in (I) are located in equatorial positions [O2—C3—C2—O1 = −176.7 (2)° and C9—C6—C5—C4 = 173.9 (2)°]. The sum of the absolute values of the six endocyclic torsion angles within the tetrahydropyran entity of

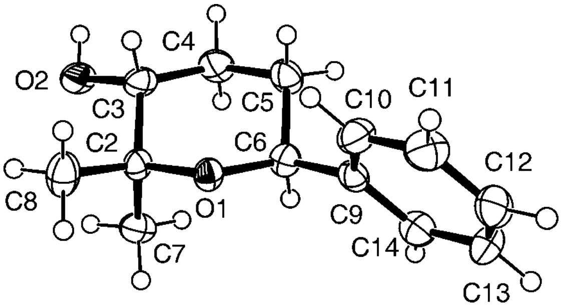


Figure 1
Molecular structure of (I). Displacement ellipsoids are plotted at the 50% probability level.

(I) is 330 (2)°. The intermolecular O—H···O hydrogen bond (Table 1) links the alternating $3R,6S$ and $3S,6R$ enantiomers of (I) into linear chains (Fig. 2).

Experimental

Suitable crystals for X-ray diffraction were obtained by slow addition of petroleum ether to a solution of (I) in diethyl ether.

Crystal data

$C_{13}H_{18}O_2$
$M_r = 206.27$
Monoclinic, $P2_1/n$
$a = 9.4212$ (6) Å
$b = 12.8792$ (9) Å
$c = 10.0568$ (7) Å
$\beta = 105.609$ (1)°
$V = 1175.26$ (14) Å^3
$Z = 4$

$D_x = 1.166$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 3.3$–14.8°
$\mu = 0.08$ mm^{-1}
$T = 173$ (2) K
Block, colourless
0.30 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2148 measured reflections
2148 independent reflections
2056 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 25.4°$
$h = -11 \rightarrow 10$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 100 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.140$
$S = 1.28$
2148 reflections
142 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 0.8889P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.83 (3)	2.02 (3)	2.848 (2)	177 (3)

Symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$.

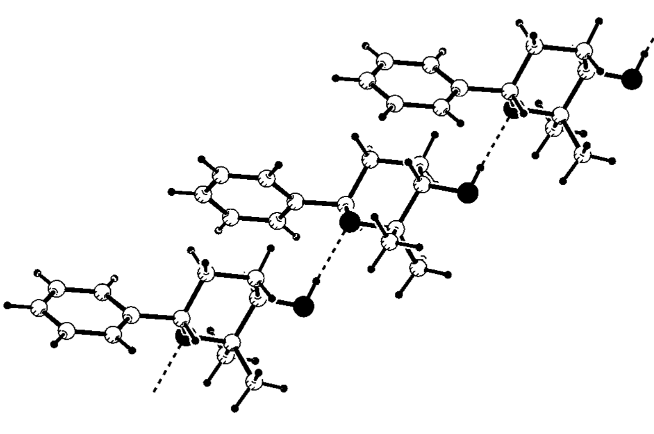


Figure 2
Intermolecular hydrogen-bond formation (dashed lines) of (I) in the solid state [view along 010].

Atom H2 was located in a difference Fourier map and refined freely. All other H atoms were placed in geometrically idealized positions (0.95–1.00 Å), and their $U_{\rm iso}$(H) values were constrained to $1.5U_{\rm eq}$(C) (CH_3) or $1.2U_{\rm eq}$(C) (all other H atoms).

Data collection: *CAD*-4 *Diffractometer Control Software* (Enraf–Nonius, 1993); cell refinement: *CAD*-4 *Diffractometer Control Software*; data reduction: *CAD*-4 *Diffractometer Control Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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