Racemic *trans*-4-phenylperhydropyran-3-ol

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Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.005 Å
R factor = 0.062
wR factor = 0.208
Data-to-parameter ratio = 15.5

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

In the title compound, $C_{11}H_{14}O_2$, the tetrahydropyran ring adopts a $^1C_4$ conformation, with the phenyl and the hydroxy substituents located in equatorial positions. Hydrogen bonding occurs in the solid state between hydroxy groups to link two molecules of (*R,R*)-*trans*-4-phenylperhydropyran-3-ol and two molecules of the (*S,S*)-enantiomer to provide tetramers.

Comment

Atoms C3 and C4 in *trans*-4-phenylperhydropyran-3-ol, (I), are chiral centres. The racemate of (I) was prepared as the minor product from the reaction between 3-phenyl-4-penten-1-ol and *m*-chloroperbenzoic acid (*m*CPBA) (Hartung *et al.*, 2003). The compound was crystallized and investigated by X-ray diffraction, in order to establish reference data for a planned conformational analysis of multiply substituted tetrahydropyrans.

(I)
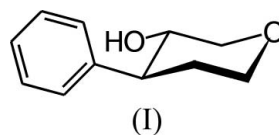
In (3*S*,4*S*)-(I), atoms O1 and C4 are displaced in opposite directions [0.655 (4) Å for O1 and −0.648 (5) Å for C4] from the tetrahydropyran plane, which is defined by atoms C2, C3, C5 and C6 [deviation of atom C6 is − 0.040 (7) Å] (Fig. 1). This geometry ($^1C_4$) corresponds to the major conformation of (I) in $CDCl_3$ solution, as established from the magnitude of the vicinal proton–proton coupling constants (^{1}H NMR). The interplanar angle between the segments C2/O1/C6 and C2/C3/C5/C6 is 52.9 (3)°. This value is larger than the inclination between the C3/C4/C5 and C2/C3/C5/C6 planes [46.0 (3)°]. The bond lengths within the heterocyclic core increase along the series O1—C2 < O1—C6 < C2—C3 < C5—C6 < C4—C5 < C3—C4 (Table 1). The phenyl and hydroxy substituents are
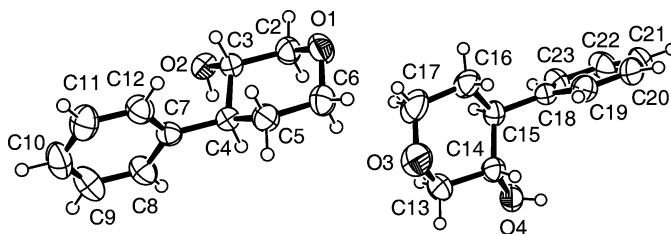

Figure 1
Molecular structure of (3*R*,4*R*)-(I) (right) and (3*S*,4*S*)-(I) (left). Displacement ellipsoids are plotted at the 50% probability level.
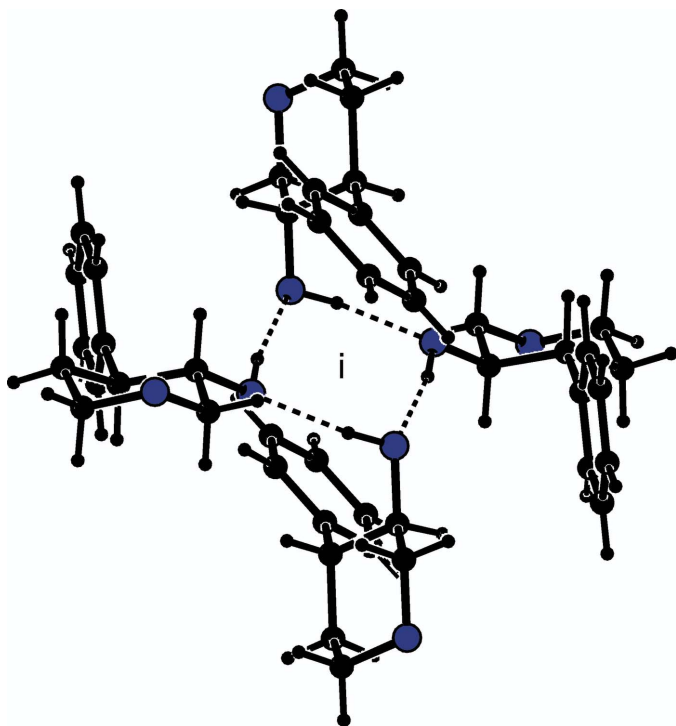
Figure 2
Intermolecular hydrogen-bond formation (dashed lines) between two molecules of (3R,4R)-(I) and two of (3S,4S)-(I) [view along 010]. The letter i marks a centre of inversion.

located in equatorial positions [O2—C3—C4—C5 = 172.6 (2)° and C7—C4—C5—C6 = −177.7 (3)°]. The phenyl ring is twisted by 75.0 (1)° from the tetrahydropyran plane. The absolute values of the six endocyclic tetrahydropyran torsion angles sum to 336 (2)°. The structures of the two independent molecules in the asymmetric unit are very similar (Table 1 and Fig. 1).

The unit cell comprises two molecules of (3R,4R)-(I) and two of the (3S,4S) enantiomer. Intermolecular O—H···OH bonding (Table 2) links two molecules of (3R,4R)-(I) with two molecules of (3S,4S)-(I) to provide tetramers (Fig. 2).

Experimental

Compound (I) was obtained as a side product (32%) from the oxidation of racemic 3-phenyl-4-penten-1-ol with mCPBA (Cook & Djerassi, 1973) in a solution of CH_2Cl_2 at 298 K (Hartung et al., 2003). Colourless prisms (m.p. 346 K) suitable for X-ray diffraction crystallized from a solution of (I) in diethyl ether upon slow addition of petroleum ether.

Crystal data

$C_{11}H_{14}O_2$	$Z = 4$
$M_r = 178.22$	$D_x = 1.236$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.580$ (3) Å	Cell parameters from 25
$b = 10.015$ (1) Å	reflections
$c = 10.670$ (2) Å	$\theta = 2.6–12.4°$
$\alpha = 77.38$ (1)°	$\mu = 0.08$ mm^{-1}
$\beta = 85.68$ (2)°	$T = 299$ (2) K
$\gamma = 73.55$ (1)°	Prism, colourless
$V = 958.0$ (4) Å^3	$0.50 \times 0.22 \times 0.10$ mm

Data collection

Nonius CAD-4 diffractometer	$\theta_{max} = 26.0°$
$\omega/2\theta$ scans	$h = -11 \rightarrow 1$
Absorption correction: none	$k = -12 \rightarrow 11$
4247 measured reflections	$l = -13 \rightarrow 13$
3738 independent reflections	3 standard reflections
1776 reflections with $I > 2\sigma(I)$	frequency: 120 min
$R_{int} = 0.160$	intensity decay: 13.9%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.113P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.062$	$+ 0.3203P]$
$wR(F^2) = 0.208$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.90$	$(\Delta/\sigma)_{max} = 0.068$
3738 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
241 parameters	$\Delta\rho_{min} = -0.32$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

C2—O1	1.419 (4)	C13—O3	1.420 (5)
C2—C3	1.508 (4)	C14—O4	1.418 (4)
C3—O2	1.424 (4)	C14—C15	1.521 (4)
C3—C4	1.531 (4)	C15—C18	1.510 (4)
C4—C7	1.526 (4)	C15—C16	1.530 (4)
C4—C5	1.526 (4)	C16—C17	1.506 (5)
C5—C6	1.514 (5)	C17—O3	1.416 (5)
C6—O1	1.433 (4)		
O1—C2—C3	112.9 (3)	C15—C14—C13	110.5 (3)
C2—C3—C4	111.2 (2)	C16—C15—C14	109.0 (3)
C5—C4—C3	108.9 (2)	C17—C16—C15	110.8 (3)
C6—C5—C4	111.0 (3)	O3—C17—C16	112.1 (3)
O1—C6—C5	111.3 (3)	C2—O1—C6	110.5 (3)
O3—C13—C14	111.4 (3)	C13—O3—C17	111.4 (3)
O1—C2—C3—C4	−55.3 (4)	C14—C15—C16—C17	−51.7 (4)
C2—C3—C4—C5	50.4 (3)	C15—C16—C17—O3	55.8 (5)
C3—C4—C5—C6	−52.0 (3)	C3—C2—O1—C6	59.8 (4)
C4—C5—C6—O1	58.0 (4)	C5—C6—O1—C2	−60.8 (4)
O3—C13—C14—C15	−57.5 (4)	C14—C13—O3—C17	60.6 (4)
C13—C14—C15—C16	52.5 (4)	C16—C17—O3—C13	−59.9 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2C···O4^i	0.91 (4)	1.92 (4)	2.823 (3)	168 (3)
O4—H4A···O2^{ii}	0.79 (4)	1.97 (4)	2.750 (3)	169 (4)

Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $x - 1, y + 1, z$.

Atoms H2C and H4A were located in a difference Fourier map. The atomic coordinates of H2C and H4A were refined with the U_{iso}(H) value set at $1.2U_{eq}$ of O2 and O4, respectively. All other H atoms were positioned geometrically and treated as riding atoms (C—H = 0.93–0.98 Å), with the U_{iso}(H) values set at $1.2U_{eq}$ of the parent atom.

Data collection: *CAD-4 Diffractometer Control Software* (Nonius, 1993); cell refinement: *CAD-4 Diffractometer Control Software*; data reduction: *CAD-4 Diffractometer Control Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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