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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å

R factor = 0.029

wR factor = 0.087

Data-to-parameter ratio = 13.9

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

(3*R*,4*R*)-(–)-3-Hydroxymethyl-4-phenyl- 2-*o*-toluoyl-1,2,3,4-tetrahydroisoquinoline

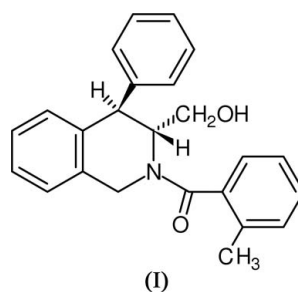
The absolute configuration of the title compound, $\text{C}_{24}\text{H}_{23}\text{NO}_2$, has been confirmed as 3*R*,4*R*. The hydroxymethyl group and phenyl ring at the asymmetric C atoms exhibit α and β orientations, respectively, and the non-planar heterocyclic ring of the tetrahydroisoquinoline system adopts an envelope conformation. The crystal structure is stabilized through hydrogen bonds.

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Comment

The title compound, (I), was obtained from the reaction of (3*R*,4*R*)-3-hydroxymethyl-4-phenyl-1,2,3,4-tetrahydroisoquinoline (Brózda *et al.*, 2000) and *o*-toluoyl chloride, according to the literature procedure of Chrzanowska *et al.* (2004). We have undertaken an X-ray crystallographic study in order to confirm the correctness of the absolute configuration of (I) which was proposed earlier on the basis of spectroscopic data by Chrzanowska *et al.* (2004), and present the results here.



The results obtained for (I) (Fig. 1) confirm the absolute configuration 3*R*,4*R* of the compound. The hydroxymethyl group at the asymmetric atom C3 exhibits an α -orientation, whereas the phenyl group at the asymmetric atom C4 is β -oriented. The substituents at C3 and C4 have a mutually *trans* configuration and occupy pseudo-axial positions with respect to the partially saturated pyridine ring, as seen in the angles of the C3–C20 and C4–C22 bond vectors to the C1/N2/C3/C4/C9/C10 ring-plane normal of 4.15 (8) and 14.40 (8)°, respectively [Cremer & Pople (1975); *PLATON* (Spek, 2003)]. The C20–C3–C4–C22 torsion angle of -168.54 (11)° reveals a mutual orientation between anticlinal and antiperiplanar of atom C20 in the hydroxymethyl group with respect to atom C22 of the phenyl group.

The non-planar partially hydrogenated pyridine ring has an envelope conformation, with puckering parameters (Cremer & Pople, 1975) of $Q = 0.508$ (1) Å, $\theta = 55.0$ (1)° and $\varphi = 297.07$ (19)°. The deviation of atom C3 from the almost planar system of the other five atoms of the heterocyclic ring is 0.6963 (16) Å. The C=O group of the amide group is anti-

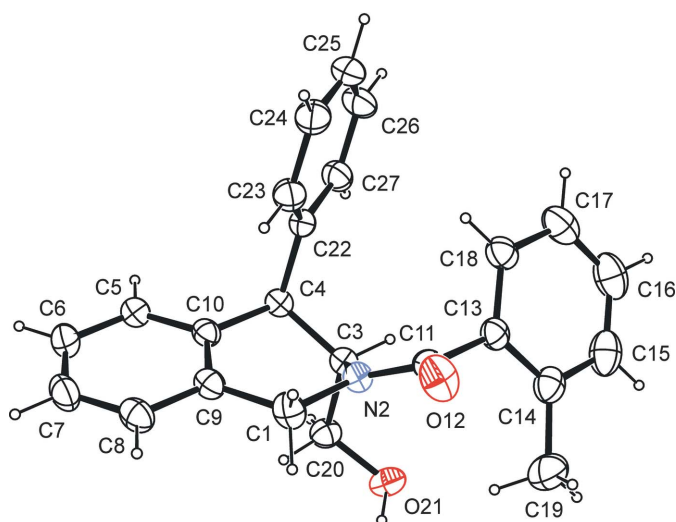


Figure 1
The molecular structure of (I), showing the atomic labelling scheme. Non-H atoms are drawn as 30% probability displacement ellipsoids.

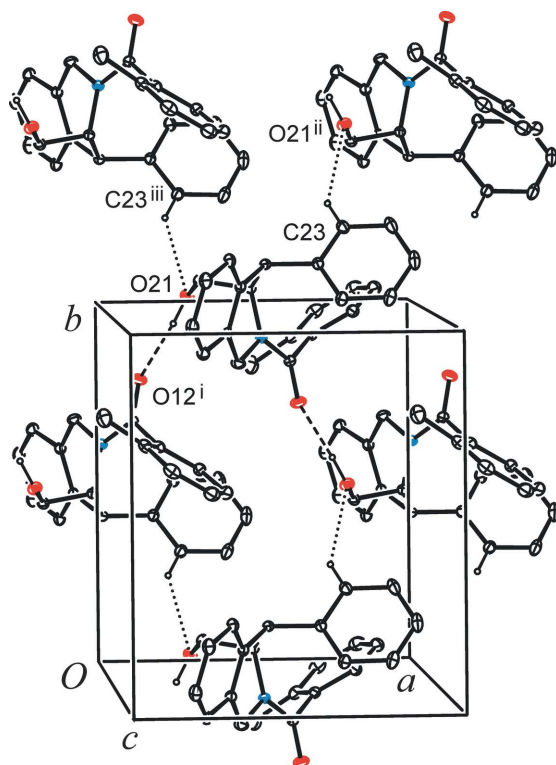


Figure 2
The hydrogen bonding (dashed and dotted lines) in the crystal structure of (I). H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) $-\frac{1}{2} + x, \frac{3}{2} - y, -z$; (ii) $\frac{1}{2} + x, \frac{5}{2} - y, -z$; (iii) $-\frac{1}{2} + x, \frac{7}{2} - y, -z$.]

periplanar with respect to the N2—C3 bond [torsion angle $178.18(13)^\circ$]. The tertiary amide group (C1/N2/C3/C11/O12) and the benzene ring (C13—C18) are not conjugated, the dihedral angle between their mean planes being $84.88(5)^\circ$. A lack of conjugation between these two groups is frequently observed in amides having aromatic rings at their carbon end (Gdaniec *et al.*, 1979; Pływaczyk *et al.*, 1984; Sugiura *et al.*,

1997). The N2—C11 bond distance of $1.3436(16) \text{ \AA}$ is typical of a tertiary amide C—N bond length [$1.346(5) \text{ \AA}$; Allen *et al.*, 1987].

Atom O21 of the hydroxyl group is synclinal with respect to the N2—C3 bond of the heterocyclic ring in the tetrahydroisoquinoline system [torsion angle N2—C3—C20—O21 = $67.55(14)^\circ$]. We assume that this arrangement of atom O21 is stabilized by the intermolecular O21—H21 \cdots O12ⁱ hydrogen bond (Table 1), which links molecules into chains parallel to the *a* axis. An intermolecular C23—H23 \cdots O21ⁱⁱ contact (Table 1) links neighbouring chains into a two-dimensional hydrogen-bond network parallel to the *ab* plane (Fig. 2).

Experimental

Compound (I) was synthesized according to the literature procedure of Chrzanowska *et al.* (2004) (m.p. 458–462 K). $[\alpha]_D = -57.99$ (*c* 0.925, MeOH). A crystal suitable for single-crystal X-ray diffraction analysis was selected directly from the analytical sample.

Crystal data

$C_{24}H_{23}NO_2$	$Z = 4$
$M_r = 357.43$	$D_x = 1.249 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Cu $K\alpha$ radiation
$a = 9.0424(10) \text{ \AA}$	$\mu = 0.62 \text{ mm}^{-1}$
$b = 10.4114(11) \text{ \AA}$	$T = 293(2) \text{ K}$
$c = 20.191(2) \text{ \AA}$	Block, colourless
$V = 1900.9(3) \text{ \AA}^3$	$0.40 \times 0.20 \times 0.15 \text{ mm}$

Data collection

Kuma KM-4 diffractometer	$R_{\text{int}} = 0.013$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 70.1^\circ$
Absorption correction: none	2 standard reflections
3740 measured reflections	every 100 reflections
3480 independent reflections	intensity decay: $<2\%$
3297 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} = 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.029$	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
$wR(F^2) = 0.087$	$\Delta\rho_{\text{min}} = -0.09 \text{ e \AA}^{-3}$
$S = 1.07$	Extinction correction: <i>SHELXL97</i>
3480 reflections	(Sheldrick, 1997)
250 parameters	Extinction coefficient: 0.0036 (4)
H atoms treated by a mixture of independent and constrained refinement	Absolute structure: Flack (1983), 1403 Friedel pairs
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.1426P]$	Flack parameter: 0.0 (2)
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1
Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O21—H21 \cdots O12 ⁱ	0.90 (2)	1.91 (2)	2.7907 (16)	165.0 (19)
C19—H19C \cdots O21	0.96	2.54	3.290 (2)	135
C23—H23 \cdots O21 ⁱⁱ	0.93	2.52	3.2774 (19)	139

Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, -z$; (ii) $x + \frac{1}{2}, -y + \frac{5}{2}, -z$.

The position of the hydroxyl H atom was obtained from a difference Fourier map and was refined freely. The remaining H atoms were positioned geometrically and were refined with a riding model, with C—H = 0.93–0.98 \AA and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, or $1.5U_{\text{eq}}(\text{C})$

for methyl H. The methyl H atoms were refined as a rigid group, which was allowed to rotate. The absolute configuration of (I) was established by refinement of the Flack parameter (Flack, 1983). The rather large s.u. of the Flack parameter is due to the small contribution of atoms with significant anomalous dispersion effects.

Data collection: *KM-4 Software* (Kuma, 1996); cell refinement: *KM-4 Software*; data reduction: *KM-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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