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(−)-1-Benzyl-4-[(1*R*)-2-hydroxy-1-phenylethylamino]-5,6-dihydropyridin-2(1*H*)-one

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Key indicators

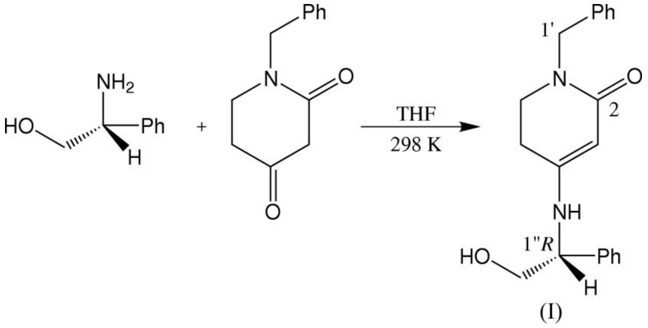
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.120
Data-to-parameter ratio = 9.2

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

In the title molecule, $C_{20}H_{22}N_2O_2$, the pyridine heterocycle has an envelope conformation and is stabilized as an enamine tautomer. The crystal packing is stabilized by strong intermolecular O—H···O hydrogen bonds, which link the molecules into chains along the *b* axis.

Comment

The title molecule, (I) (Fig. 1), belongs to a series of functionalized chiral enamines, which we use as synthons for the preparation of alkaloids. The molecular structure of (I) is very close to that of (−)-1-benzyl-4-[1(*S*)-phenylethylamino]-5,6-dihydropyridin-2(1*H*)-one, described in the previous paper (Romero et al., 2005).



(I)

The crystal structure is built up by chains of molecules assembled *via* strong O—H···O hydrogen bonds (Table 2 and Fig. 2). These chains run along the *b* axis and are connected *via* a very weak secondary hydrogen bond oriented along [100], which involves the carbonyl and amine functionalities (Table 2). This arrangement does not allow significant π–π interactions between phenyl rings in the crystal structure.

Experimental

To a solution of (*R*)-(−)-phenylglycinol (0.72 mmol, 100 mg) in dry tetrahydrofuran (THF) (5 ml) was added a solution of 1-benzylpiperidine-2,4-dione (0.6 mmol, 120 mg) in dry THF (5 ml) and the mixture was stirred at 298 K for 12 h. The solvent was then removed and the crude product recrystallized from CH_2Cl_2/*n*-hexane (1:1) to give (I) (yield 100%). Spectroscopic analysis: $[\alpha]_D^{20} = -31.5$ (*c* 1.1, CH_2Cl_2); ^1H NMR (400 MHz, $CDCl_3$, δ, p.p.m.): 2.30 (*m*, 2H, H5), 3.10 (*m*, 2H, H6), 3.6–3.8 (*AB*, 2H, H9), 4.39 (*m*, 1H, H8), 4.5 (*d*, 2H, H16), 4.57 (*s*, 1H, H3), 5.30 (*d*, 1H, H7), 7.20–7.40 (*m*, 10H, Ph); ^{13}C NMR (100.6 MHz, $CDCl_3$, δ, p.p.m.): 28.60 (C5), 43.90 (C6), 49.37 (C16), 59.55 (C9), 66.40 (C8), 89.20 (C3), 126.30–128.00 (10 C, Ph), 137.00 (C-*ipso*), 138.00 (C-*ipso*), 154.50 (C4), 168.2 (C2).

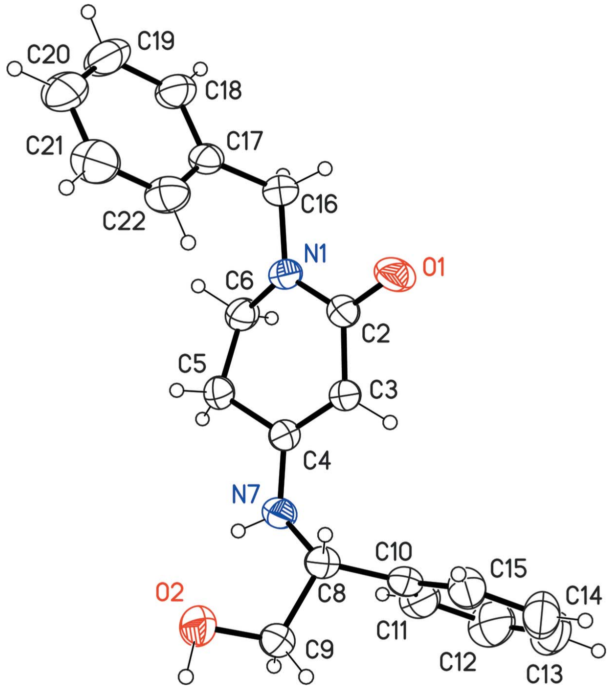


Figure 1
A view of (I), with displacement ellipsoids at the 40% probability level.

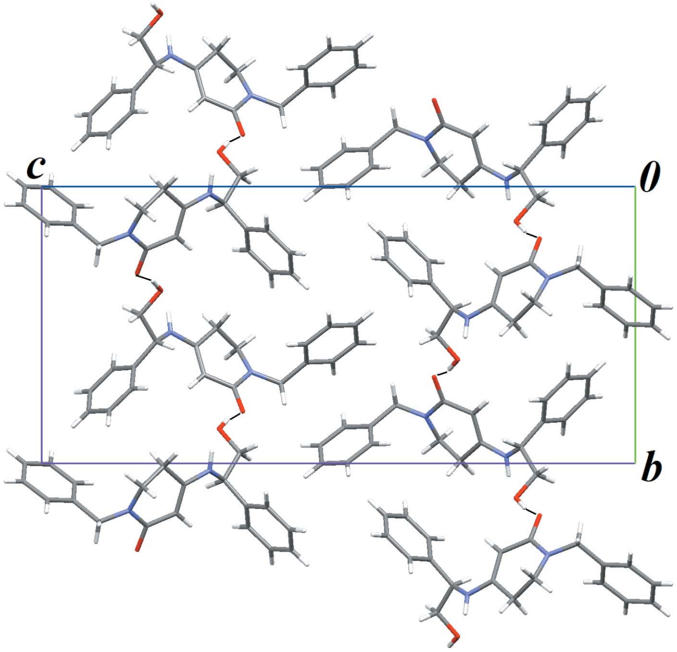


Figure 2
Part of the crystal structure of (I) viewed down the *a* axis, showing the main hydrogen-bonding scheme (solid black lines).

Crystal data

$C_{20}H_{22}N_2O_2$
$M_r = 322.40$
Orthorhombic, $P2_12_12_1$
$a = 5.5309$ (10) Å
$b = 12.172$ (2) Å
$c = 26.121$ (4) Å
$V = 1758.5$ (5) Å^3
$Z = 4$
$D_x = 1.218$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 68 reflections
$\theta = 4.6$–12.5°
$\mu = 0.08$ mm^{-1}
$T = 296$ (1) K
Plate, colourless
0.6 × 0.4 × 0.1 mm

Data collection

Bruker *P*4 diffractometer
ω scans
Absorption correction: none
4260 measured reflections
2080 independent reflections
1590 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.061$

$\theta_{\rm max} = 26.3°$
$h = -6 \rightarrow 6$
$k = -1 \rightarrow 15$
$l = -1 \rightarrow 32$
3 standard reflections every 97 reflections intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.120$
$S = 1.02$
2080 reflections
226 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.1481P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXTL-Plus* (Sheldrick, 1998)
Extinction coefficient: 0.027 (3)

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.350 (3)	C4—N7	1.358 (3)
N1—C6	1.453 (3)	C4—C5	1.498 (3)
N1—C16	1.460 (3)	C5—C6	1.518 (3)
C2—O1	1.251 (3)	N7—C8	1.457 (3)
C2—C3	1.453 (3)	C9—O2	1.421 (3)
C3—C4	1.357 (3)	O2—H2	0.99 (4)
C2—N1—C6	119.49 (19)	C4—C3—C2	122.2 (2)
C2—N1—C16	121.0 (2)	N7—C4—C3	123.1 (2)
C6—N1—C16	117.5 (2)	N7—C4—C5	117.5 (2)
O1—C2—N1	121.4 (2)	C3—C4—C5	119.3 (2)
O1—C2—C3	120.8 (2)	C4—N7—C8	121.9 (2)
N1—C2—C3	117.8 (2)	C9—O2—H2	102.0 (19)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1$^{\rm i}$	0.99 (4)	1.72 (4)	2.689 (3)	167 (3)
N7—H7A···O1$^{\rm ii}$	0.85 (4)	2.40 (4)	3.168 (3)	151 (3)

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

The H atoms bonded to N7 and O2 were found in a difference Fourier map and refined with free coordinates and isotropic U parameters. The C-bound H atoms were placed in idealized positions and refined as riding on their parent C atom, with the following constraints: methine C—H = 0.98 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$; methylene C—H = 0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$; methyl C—H = 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$; aromatic and vinylic C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. In the absence of significant anomalous scattering effects, Friedel pairs were merged, and the absolute configuration was assigned on the basis of the known configuration of the amine used as starting material.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL-Plus*; molecular graphics: *SHELXTL-Plus*; software used to prepare material for publication: *SHELXTL-Plus*.

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