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(3*S*,4*S*)-*tert*-Butyl 4-amino-3-hydroxy-5-phenylpentanoate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.115
Data-to-parameter ratio = 9.5

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

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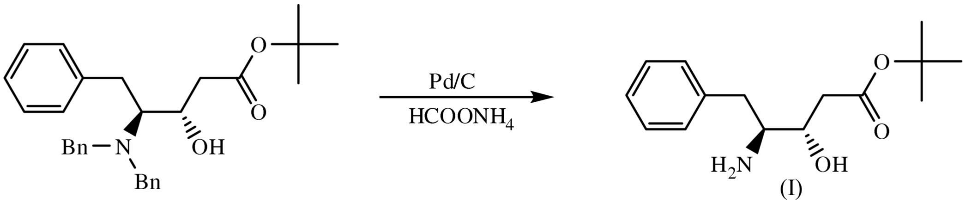
The title compound, $C_{15}H_{23}NO_3$, was obtained by hydrogenolysis of (3*S*,4*S*)-*tert*-butyl 4-(dibenzylamino)-3-hydroxy-5-phenylpentanoate. In the crystal structure, intermolecular O—H···N hydrogen bonds link the molecules into sheets parallel to the *bc* plane.

Comment

The title compound, (I), which belongs to the class of β-amino alcohols, is a key intermediate used to synthesize chiral ligands, such as bis(oxazoline) ligands, for asymmetric catalysts. The majority of the syntheses of these ligands have followed a general synthetic route in which oxalic acid or substituted malonic acids were first condensed with the corresponding optically active β-amino alcohols to form the bis(hydroxy)amide derivatives. The hydoxy groups in the bis(hydroxy)amide were then activated and the resulting intermediate was cyclized to provide the bis(oxazoline) ligands (Ghosh *et al.*, 1998).



Bond lengths and angles in the title compound are in agreement with the values quoted for (3*S*,4*S*)-4-amino-5-phenylpentane-1,3-diol (Tan *et al.*, 2005). The molecular structure is stabilized by two intramolecular C—H···O and one N—H···O hydrogen bonds (Table 1). In the crystal structure, intermolecular O—H···N hydrogen bonds link the molecules into sheets parallel to the *bc* plane.

Experimental

To a solution of (3*S*,4S)-*tert*-butyl 4-(dibenzylamino)-3-hydroxy-5-phenylpentanoate (2.67 g, 6 mmol) in methanol (20 ml), ammonium formate (2.27 g, 36 mmol) and palladium on carbon (10% Pd, 0.51 g) were added. The mixture was heated to 353 K and stirred for 3 h (Stuk *et al.*, 1994). After completion of the reaction, the solution was filtered through diatomaceous earth and the cake was washed with methanol (5 ml). The solvent was removed to give the crude product, which was purified by flash column chromatography (SiO_2; ethyl acetate/petroleum ether, 10:1 *v*/*v*) to yield the title compound (1.46 g). Single crystals were obtained by slow evaporation of an ethyl acetate/petroleum ether (1:2 *v*/*v*) solution.

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Crystal data

$C_{15}H_{23}NO_3$
M_r = 265.34
Orthorhombic, $P2_12_12_1$
a = 11.696 (3) Å
b = 14.377 (4) Å
c = 9.177 (2) Å
V = 1543.1 (7) Å^3
Z = 4
D_x = 1.142 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 (2) K
Chunk, colourless
0.44 × 0.32 × 0.25 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.966, T_{max} = 0.981
8320 measured reflections
1743 independent reflections
1626 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.115
S = 0.85
1743 reflections
184 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0925P)^2 + 0.2485P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13A···O2	0.96	2.54	3.078 (3)	116
C15—H15A···O2	0.96	2.41	2.965 (3)	116
N1—H1A···O1	0.91 (3)	2.46 (3)	2.830 (3)	105 (2)
O1—H1C···N1^{i}	0.90 (3)	1.92 (3)	2.820 (3)	178 (2)

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

The hydroxy and amine H atoms were located in a difference Fourier map and refined freely. All other H atoms were placed in geometrically idealized positions and treated as riding on their parent atoms with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was assigned from the synthesis.

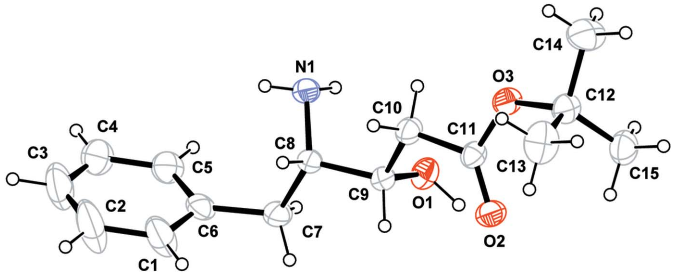

Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radius.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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