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

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.063
wR factor = 0.178
Data-to-parameter ratio = 10.2

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

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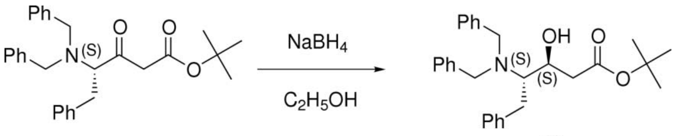
The title compound, $C_{29}H_{35}NO_3$, was obtained as a major product by reduction of (*S*)-*tert*-butyl 4-dibenzylamino-3-oxo-5-phenylpentanoate. The molecular packing in the crystal structure is stabilized by weak intermolecular interactions and van der Waals forces.

Comment

The synthesis of a chiral tridentate ligand, (3*S*,4*S*)-*tert*-butyl 4-(dibenzylamino)-3-hydroxy-5-phenylpentanoate has been achieved; this is a very important starting material. When we used $NaBH_4$ to reduce (*S*)-*tert*-butyl 4-(dibenzylamino)-3-oxo-5-phenylpentanoate (Hoffman *et al.*, 1997), the title compound was formed unexpectedly.



(I)

Experimental

(*S*)-*tert*-Butyl 4-(dibenzylamino)-3-oxo-5-phenylpentanoate (443 mg, 1 mmol) was dissolved in ethanol (10 ml). The resulting solution was then cooled to 253 K and treated with $NaBH_4$ (114 mg, 3 mmol). The

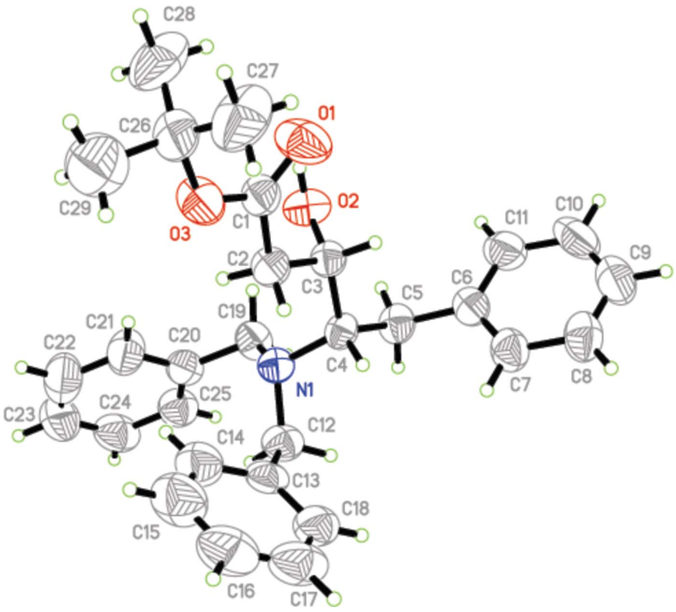


Figure 1
*ORTEP*3 (Farrugia, 1997) plot of (I), with displacement ellipsoids drawn at the 50% probability level.

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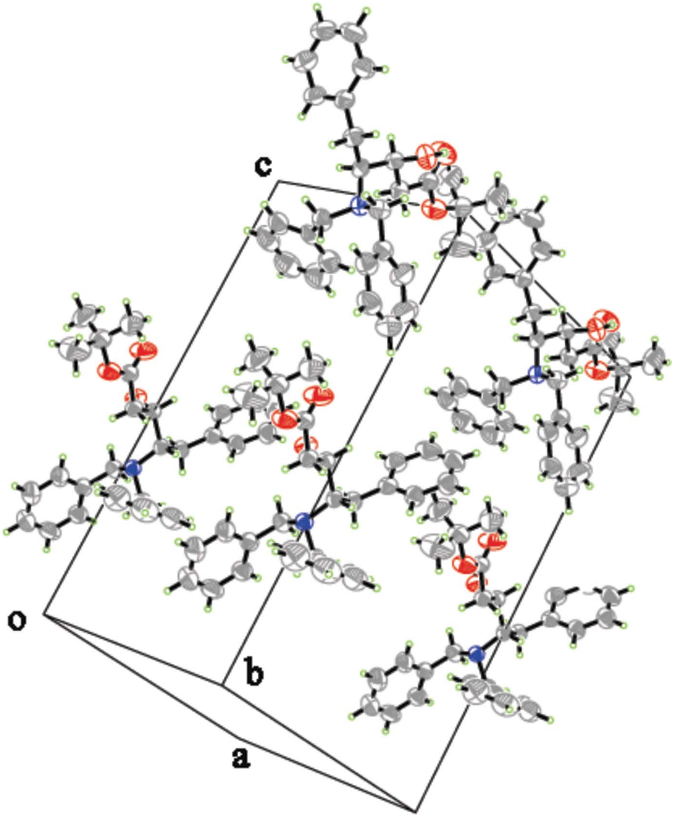

Figure 2
Packing diagram of the title compound (Accelrys, 2001).

reaction was monitored by TLC. After 3 h, the solution was quenched with water (20 ml) and adjusted to pH = 5–6 using 1 *M* HCl, extracted with diethyl ether, washed with brine (20 ml), dried over $MgSO_4$ and finally concentrated to obtain the crude product (424 mg). Suitable crystals were obtained by allowing the crude product solution to stand for three days.

Crystal data

$C_{29}H_{35}NO_3$
$M_r = 445.58$
Orthorhombic, $P2_12_12_1$
$a = 10.351$ (3) Å
$b = 12.615$ (3) Å
$c = 19.640$ (5) Å
$V = 2564.5$ (11) $Å^3$
$Z = 4$
$D_x = 1.154$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4261 reflections
$\theta = 2.6$–22.0°
$\mu = 0.07$ mm^{-1}
$T = 293$ (2) K
Chunk, colorless
0.56 × 0.28 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.960$, $T_{max} = 0.987$
14713 measured reflections
2994 independent reflections
2600 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 26.5°$
$h = -12 \rightarrow 12$
$k = -15 \rightarrow 13$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.178$
$S = 1.12$
2994 reflections
293 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0835P)^2 + 0.6494P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.57$ e $Å^{-3}$
$\Delta\rho_{min} = -0.33$ e $Å^{-3}$

C29 was not refined anisotropically due to unresolved disorder. H atoms were positioned geometrically (C—H = 0.93, 0.93, 0.96 and 0.97 Å for phenyl, methine, methylene and methyl H atoms, respectively, O—H = 0.82 Å). U_{iso}(H) values were set equal to xU_{eq} of the carrier atom, where $x = 1.5$ for methyl and $x = 1.2$ for all other H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration was assigned on the basis of the known configuration of (*S*)-*tert*-butyl 4-(dibenzylamino)-3-oxo-5-phenylpentanoate.

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) and *ViewerPro* (Accelrys, 2001); software used to prepare material for publication: *SHELXL97*.

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