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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.039
 wR factor = 0.113
Data-to-parameter ratio = 10.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*N*-(2-Hydroxy-1-naphthylmethyl)-(S)-proline

The title compound [systematic name: (2*S*)-1-(2-hydroxy-1-naphthylmethyl)pyrrolidinio-2-carboxylate], $\text{C}_{16}\text{H}_{17}\text{NO}_3$, a derivative of L-proline, was synthesized *via* the Mannich reaction. The X-ray crystal analysis reveals that the pyrrolidine ring adopts an envelope conformation, with the flap C atom deviating by 0.631 (9) Å from the plane defined by the other four atoms.

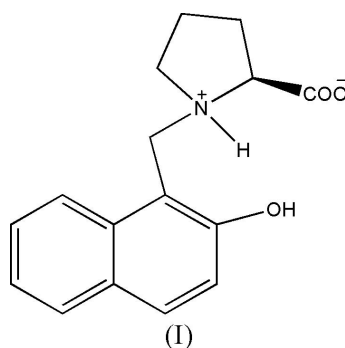
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Comment

In order to synthesize chiral amino alcohols, which can be used for asymmetric Michael addition (Yamakoshi *et al.*, 1999; Sasai *et al.*, 1993), the title compound, (I), was prepared *via* a Mannich reaction with L-proline, 2-naphthol and formaldehyde. The original *S* configuration of the chiral centre at C2 is assumed not to change during the preparation, as shown in Fig. 1. Selected bond distances and angles are given in Table 1. The pyrrolidine ring may adopt different conformations (Stavropoulos *et al.*, 1990; Puliti *et al.*, 2000; Zheng & Li, 2003). Here it adopts an envelope conformation, with atom C4 deviating by 0.631 (9) Å from the plane defined by atoms N1, C2, C3 and C5.



Conjugation between bonds O1—C1 and O2—C1 is observed, with bond distances of 1.222 (3) and 1.239 (3) Å, respectively. The conformation and crystal structure of the title compound are stabilized by N—H...O and O—H...O hydrogen bonds (see Table 2).

Experimental

The title compound was prepared according to the procedure of Zheng & Li (2003). Suitable crystals were obtained by slow evaporation of a mixture of chloroform and cyclohexane (m.p. 455–457 K). ^1H NMR (CD_3OD , p.p.m.): δ 1.88–1.93 (*m*, 1H), 2.07–2.19 (*m*, 2H), 2.47–2.52 (*m*, 1H), 3.29–3.38 (*m*, 1H), 3.46–3.51 (*m*, 1H), 4.05–4.09 (*m*, 1H), 4.80–4.91 (*m*, 4H), 7.19 (*d*, 1H, $J = 8$ Hz), 7.35 (*t*, 1H, $J = 8$ Hz), 7.57 (*t*, 1H, $J = 8$ Hz), 7.79–7.85 (*m*, 2H), 8.20 (*d*, 1H, $J = 8$ Hz).

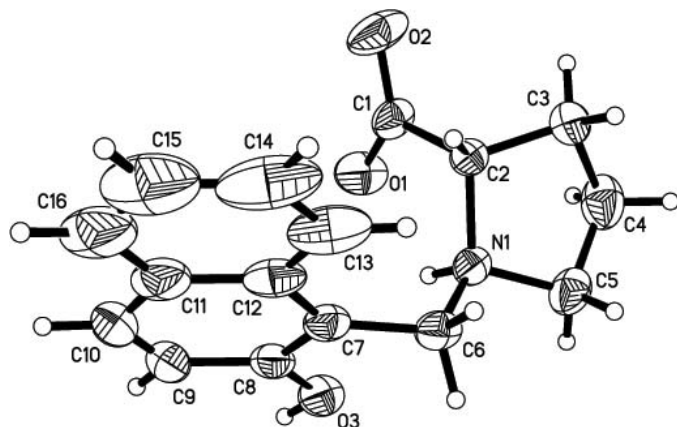


Figure 1

A view of the molecule of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

Crystal data

$C_{16}H_{17}NO_3$
 $M_r = 271.31$
 Orthorhombic, $P2_12_12_1$
 $a = 5.8037$ (7) Å
 $b = 12.0508$ (15) Å
 $c = 20.160$ (3) Å
 $V = 1410.0$ (3) Å³
 $Z = 4$
 $D_x = 1.278$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 1714 reflections
 $\theta = 2.6$ – 22.2°
 $\mu = 0.09$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.24 \times 0.16 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\min} = 0.964$, $T_{\max} = 0.988$
 9621 measured reflections

1980 independent reflections
 1329 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\text{max}} = 28.0^\circ$
 $h = -7 \rightarrow 7$
 $k = -15 \rightarrow 15$
 $l = -26 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.113$
 $S = 1.04$
 1980 reflections
 188 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.1866P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.18$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.15$ e Å⁻³
 Extinction correction: none

Table 1

Selected geometric parameters (Å, °).

N1—H1	0.90 (1)	O2—C1	1.239 (3)
O1—C1	1.222 (3)	O3—H3	0.86 (1)
C8—O3—H3	112 (2)	C2—C3—C4	103.2 (2)
O1—C1—O2	127.6 (3)	N1—C5—C4	103.3 (2)
O1—C1—C2	117.8 (2)	C7—C6—N1	111.8 (2)
O2—C1—C2	114.5 (2)	C5—N1—C2—C3	0.2 (3)
C5—N1—C2—C3	0.2 (3)	O1—C1—C2—C3	126.6 (2)
C6—N1—C2—C3	126.6 (2)	O2—C1—C2—N1	174.2 (3)
O1—C1—C2—N1	-9.0 (4)	O1—C1—C2—C3	106.4 (3)
		O2—C1—C2—C3	-70.5 (3)

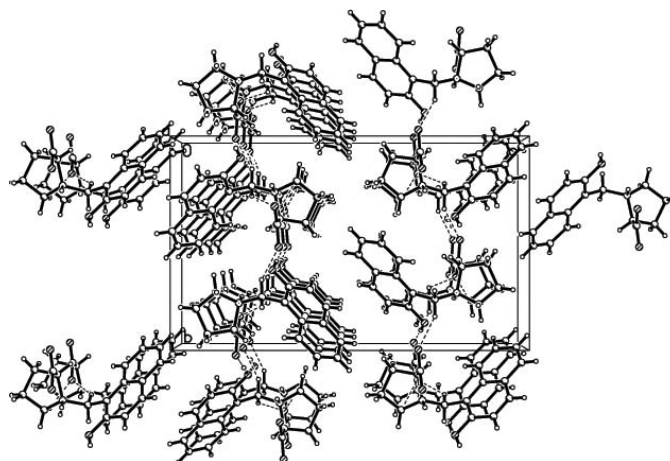


Figure 2

The crystal structure of (I), viewed along the b axis. Dashed lines indicate hydrogen-bond interactions.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1 \cdots O1	0.90 (1)	1.99 (2)	2.599 (3)	123 (2)
N1—H1 \cdots O3	0.90 (1)	2.28 (2)	2.892 (3)	124 (2)
O3—H3 \cdots O2 ⁱ	0.86 (1)	1.73 (1)	2.586 (3)	175 (4)

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

All N- and O-bound H atoms were initially located in a difference Fourier map and were included in the refinement with restrained N—H and O—H distances of 0.85 and 0.90 Å. All other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. In the absence of significant anomalous dispersion effects, Friedel pairs were merged prior to refinement. The large anisotropic displacement parameters of some atoms in the naphthalene fused-ring system indicate either high thermal motion or possible unresolved disorder.

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

References

- Bruker (1997). *SADABS, SMART, SAINT and SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Puliti, R., Mattia, C. A., Giancola, C. & Barone, G. (2000). *J. Mol. Struct.* **553**, 117–130.
- Sasai, H., Suzuki, T., Itoh, N. & Shibasaki, M. S. (1993). *Tetrahedron Lett.* **34**, 851–854.
- Sheldrick, G. M. (1997). *SHELXS97 and SHELXL97*. University of Göttingen, Germany.
- Stavropoulos, G., Papaioannou, D., Karagiannis, K., Vlasi, M. & Nastopoulos, V. (1990). *Acta Cryst.* **C46**, 1461–1464.
- Yamakoshi, K., Harwood, S. J., Kanai, M. & Shibasaki, M. (1999). *Tetrahedron Lett.* **40**, 2565–2568.
- Zheng, P. W. & Li, C. B. (2003). *Acta Cryst.* **E59**, o1621–o1623.