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(S)-N-(1-Benzyl-2-hydroxyethyl)phthalamic acid

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Key indicators

Single-crystal X-ray study

$T = 298\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.012\text{ \AA}$

Disorder in main residue

R factor = 0.075

wR factor = 0.130

Data-to-parameter ratio = 8.3

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

A feature of the structure of the title compound, $\text{C}_{17}\text{H}_{17}\text{NO}_4$, is the three-dimensional connectivity generated by intermolecular hydrogen bonds.

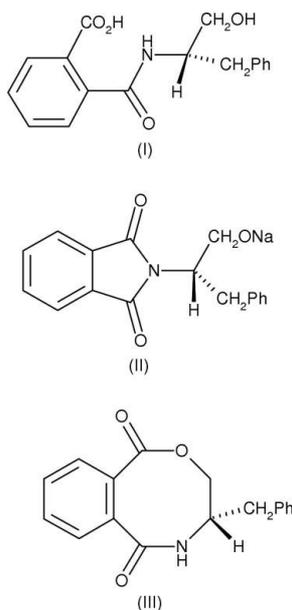
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Comment

There have been only a few authenticated reports of the interesting [1,4]-oxazocine-5,8-dione ring system exemplified by (III). Aly (2003) used a strategy involving reaction of a diethyl phthalate with a 2-aminophenol derivative to generate the lactone and lactam functions. Assoumatine *et al.* (2004) applied the analogous reaction of a succinate diester with a β -amino alcohol derivative. Conceptually the system might also be constructed from a phthaloyl β -amino alcohol, for example (II), by intramolecular nucleophilic attack of the derived side chain alkoxide on a carbonyl group, involving carbon–oxygen bond formation followed by carbon–nitrogen bond cleavage to yield the eight-membered ring system (III). However, when the Na salt of (*S*)-*N*-(1-benzyl-2-hydroxyethyl)phthalimide, (II), was generated in dry tetrahydrofuran (THF), subsequent quenching with water resulted in hydrolysis to yield the title compound, (I), rather than the oxazocine dione, (III).



The molecule of (I) is shown in Fig. 1. Although the refinement of the structure has been compromised somewhat by limitations imposed by comparatively poor intensity data obtained from a weakly diffracting sample crystal and by disorder affecting the C12–C17 phenyl group (see later for details), the molecular geometry is well enough determined to

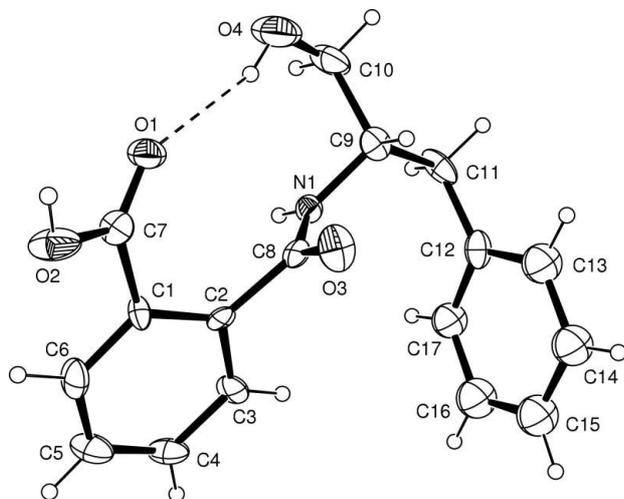


Figure 1

A view of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small circles of arbitrary radii. The intramolecular hydrogen bond is indicated by the dashed line. Only the major component (see text) of the disordered C12–C17 phenyl group is shown.

show that the bond lengths and bond angles lie within the usual ranges and do not merit further discussion here. The main interest in this structure lies in the hydrogen bonds given in Table 1. The first of these is intramolecular, creating a ten-membered ring (see Fig. 1). The other two are intermolecular and provide three-dimensional interconnection of the molecules as indicated schematically in Fig. 2. Notable in this figure is the predominance of six-membered, *i.e.* hexamolecular, hydrogen-bonded rings. One such ring is shown in detail in Fig. 3. Here it can be seen that hydrogen bonds of the form $O2-H2 \cdots O4^i$ [symmetry code: (i) $1-x, -\frac{1}{2}+y, \frac{1}{2}-z$] create infinite zigzag chains of molecules propagated in the *b* direction. Two such chains pass through the unit cell, one comprising the molecule of the asymmetric unit together with the molecules with symmetry codes (i) and (iii), and the other comprising the molecules with symmetry codes (iv), (v) and (vi) [symmetry codes as in Table 1]. Hydrogen bonds of the form $N1-H1 \cdots O3^{ii}$, on the other hand, create chains of molecules propagated in the *a* direction. These interconnect the chains previously described, by connecting molecules such as those in Fig. 3 with symmetry codes (iii) and (iv), and, in the process, complete the three-dimensional connectivity. The propagation of the chains in the direction of *a* just described also brings about a $C-H \cdots \pi$ interaction of the form $C13A-H13A \cdots Cg1^{vi}$ ($Cg1$ is the centroid of the C1–C6 ring) in which the critical parameters are the $H \cdots Cg$ distance and the $C-H \cdots Cg$ angle of 2.985 Å and 165°, respectively.

Experimental

Compound (I) was prepared by slow addition of (*S*)-*N*-(1-benzyl-2-hydroxyethyl)phthalimide (3.0 g, 10 mmol) to NaH (0.51 g, 17 mmol) in dry THF (20 ml) contained in a flask fitted with a $CaCl_2$ guard tube. After approximately 45 min, when hydrogen evolution had ceased, water (5 ml) was added slowly to remove excess sodium hydride. The

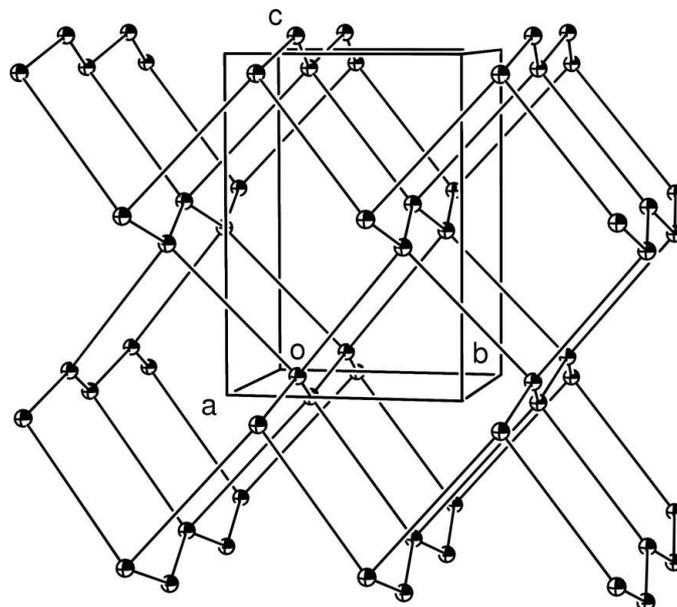


Figure 2

A schematic representation of the intermolecular hydrogen bonding in (I). The hydrogen bonds are represented by lines joining pseudo-atoms of arbitrary size which are coincident with the centroids of the molecules.

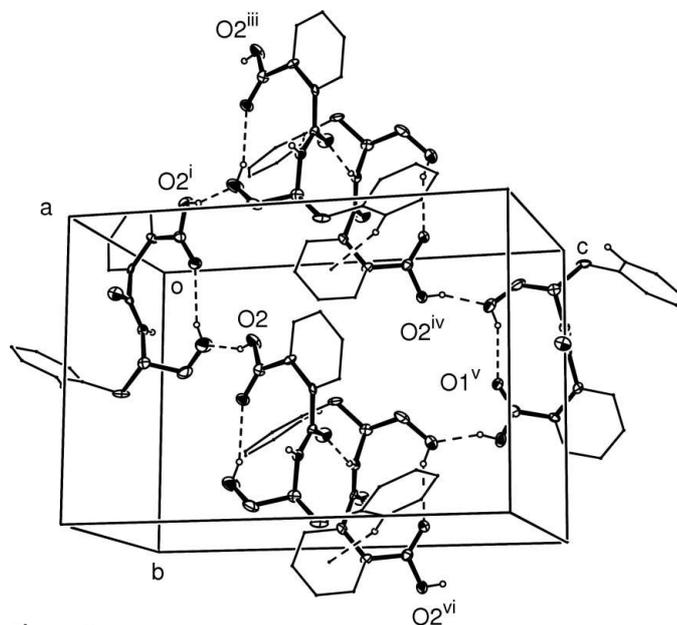


Figure 3

A view of the unit cell contents of (I). For clarity, bonds in the C1–C6 and C12–C17 rings are drawn as thin lines. Displacement ellipsoids are drawn at the 20% probability level. H atoms involved in intermolecular contacts (dashed lines) are shown as small circles of arbitrary radii. Selected atoms are labelled. [Symmetry codes: (i) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $x, y-1, z$; (iv) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$; (v) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$; (vi) $x-\frac{1}{2}, \frac{3}{2}-y, 1-z$.]

mixture was then stirred for a further 10 min, and water (30 ml) and dilute HCl (10 ml) were added. The milky suspension was extracted three times with diethyl ether, the ether extracts were combined and dried over anhydrous magnesium sulfate, and the solvent was removed under reduced pressure to yield a white powdery solid which, on recrystallization from acetone, afforded (I) as colourless crystals [yield 1.6 g, 50%; m.p. 401–403 K (dec.)]. IR (cm^{-1}): ν_{max} 3488 (OH), 3302 (NH), 1701 and 1663 (C=O); 1H NMR (DMSO,

p.p.m.): δ_{H} 2.73 (*dd*, 1H, $J = 11.2$ and 6.8 Hz, PhCH_AH_B), 2.97 (*dd*, 1H, $J = 11.2$ and 3.2 Hz, PhCH_AH_B), 3.38 (*dd*, 1H, $J = 11.4$ and 3.6 Hz, $\text{CH}_A\text{H}_B\text{OH}$), 3.59 (*dd*, 1H, $J = 11.4$ and 7.2 Hz, $\text{CH}_A\text{H}_B\text{OH}$), 4.05 (*m*, 1H, CH), 4.61 (*br s*, 1H, OH), 7.14–7.78 (*m*, 9H, aromatic), 8.23 (*d*, 1H, $J = 8.4$ Hz, NH) and 13.48 [*br s*, 1H, OH (acid)]; ^{13}C NMR (DMSO, p.p.m.): δ_{C} 35.7 (PhCH_2), 52.4 (CH_2OH), 62.0 (CH), 125.4, 126.9, 127.6, 128.5, 128.6, 128.7, 130.0, 138.3, 138.9 (aromatic), 167.5 and 167.8 (C=O). Analysis found: C 68.10, H 5.77, N 4.63%; $\text{C}_{17}\text{H}_{17}\text{NO}_4$ requires: C 68.22, H 5.72, N 4.68%.

Crystal data

$\text{C}_{17}\text{H}_{17}\text{NO}_4$	Mo $K\alpha$ radiation
$M_r = 299.32$	Cell parameters from 14 reflections
Orthorhombic, $P2_12_12_1$	$\theta = 7.9\text{--}9.6^\circ$
$a = 9.569$ (7) Å	$\mu = 0.09$ mm $^{-1}$
$b = 10.723$ (6) Å	$T = 298$ (2) K
$c = 15.416$ (6) Å	Plate, colourless
$V = 1581.8$ (16) Å 3	$0.55 \times 0.30 \times 0.10$ mm
$Z = 4$	
$D_x = 1.257$ Mg m $^{-3}$	

Data collection

Nicolet P3 four-circle diffractometer	$\theta_{\text{max}} = 25.1^\circ$
$\omega/2\theta$ scans	$h = 0 \rightarrow 11$
Absorption correction: none	$k = 0 \rightarrow 12$
1626 measured reflections	$l = 0 \rightarrow 18$
1625 independent reflections	2 standard reflections every 50 reflections
618 reflections with $I > 2\sigma(I)$	intensity decay: none
$R_{\text{int}} = 0.008$	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.075$	$w = 1/[\sigma^2(F_o^2) + (0.0356P)^2]$
$wR(F^2) = 0.130$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.85$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1625 reflections	$\Delta\rho_{\text{max}} = 0.21$ e Å $^{-3}$
196 parameters	$\Delta\rho_{\text{min}} = -0.22$ e Å $^{-3}$

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

$D\text{--H}\cdots A$	$D\text{--H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{--H}\cdots A$
O4–H4O \cdots O1	0.82	2.25	3.032 (8)	159
O2–H2 \cdots O4 ⁱ	0.82	1.82	2.591 (8)	156
N1–H1 \cdots O3 ⁱⁱ	0.87 (2)	2.09 (4)	2.883 (8)	149 (6)

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

The data set contains no Friedel pairs, and there is no significant anomalous scattering. The enantiomer chosen for the structural model is that for which the absolute configuration at the chiral centre (C9) is the same as in the phenylalanol precursor. Disorder of the C12–C17 phenyl group over two orientations related by a twist about the C11–C12 bond of approximately 20° was modelled by splitting the disordered atoms (C13–C17) into pairs as C13A/C13B with occupancies, as determined by refinement with the displacement parameters for the atoms of each pair constrained to be equal and isotropic displacement parameters for all atom pairs, of 0.61 (2) and 0.39 (2) for the major (suffix A) and minor (suffix B) components of the disorder, respectively. Restraints were applied to the disordered phenyl rings in terms of both their planarity (target r.m.s. displacement 0.02 Å) and their bond lengths and bond angles by means of a variable d for bond lengths and $1.732d$ for 1,3 distances for internal angles, respectively. The value of d after refinement was 1.382 (6) Å. In the final stages of refinement H atoms attached to C atoms were placed in calculated positions with C–H = 0.93, 0.97 and 0.98 Å for aryl, methylene and tertiary C atoms, respectively. The H atom of the NH group was placed initially as for an aryl H atom but its coordinates were then refined. Difference map peaks provided initial positions for the H atoms of the OH groups. The groups were then idealized and their torsion angles refined. In all cases the H atoms were refined with a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N,O})$.

Data collection: *Nicolet P3 Software* (Nicolet, 1980); cell refinement: *Nicolet P3 Software*; data reduction: *RDNIC* (Howie, 1980); 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: *SHELXL97* and *PLATON* (Spek, 2003).

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