

**(E)-1-Ethoxycarbonyl-2-[(2-hydroxy-1-naphthyl)-(phenyl)methyliminio]propan-1-ide**

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Received 16 January 2006

Accepted 31 January 2006

**Key indicators**

Single-crystal X-ray study

 $T = 298\text{ K}$ Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$  $R$  factor = 0.049 $wR$  factor = 0.166

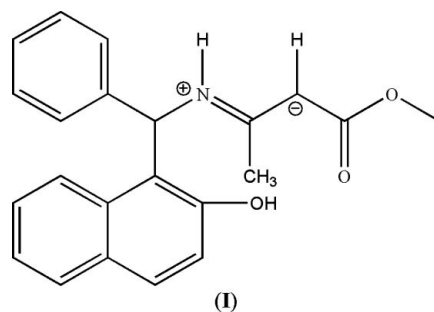
Data-to-parameter ratio = 17.7

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

In the title compound,  $\text{C}_{23}\text{H}_{23}\text{NO}_3$ , the amino, hydroxy and carbonyl groups are involved in hydrogen bonding, forming a dimer. The crystal packing is further stabilized by intermolecular  $\pi$ - $\pi$  stacking.

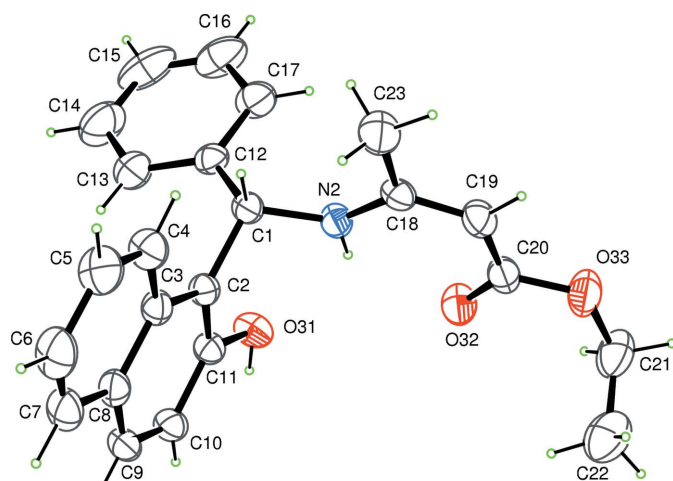
**Comment**

As early as the mid-20th century, Mario Betti reported the synthesis of the so-called Betti base by the reaction of benzaldehyde with 2-naphthol and ammonia followed by hydrolysis with hydrochloric acid (Betti, 1941). However, only in recent decades has its usefulness in asymmetric synthesis been recognized (Istvan *et al.*, 2004). As cheap resolving agents, chiral catalysts and optically active ligands, the Betti base and its derivatives are now widely used in organic synthesis (Istvan *et al.*, 2004). Chiral aminoalcohols are of importance in catalytic asymmetric synthesis (Pu *et al.*, 2001). Compound (I), prepared readily from the Betti base by treatment with ethyl acetoacetate, is one of the auxiliary-induced prochiral imino compounds which yields the corresponding chiral amino-alcohol after asymmetric hydrogenation.



In the structure of (I), atoms N2, C18, C19 and C20 are coplanar; this is illustrated clearly by the torsion angle  $\text{N2}-\text{C18}-\text{C19}-\text{C20}$  of  $-2.20(2)^\circ$  (Fig. 1). Moreover, the mean plane through these four atoms is perpendicular to the phenyl ring plane, forming a dihedral angle of  $89.89(9)^\circ$ . The dihedral angle between the naphthalene ring system and phenyl ring plane is  $78.71(6)^\circ$ .

In the crystal structure, the amino, hydroxy and carbonyl groups are involved in hydrogen bonding. Amino atom N2 acts as hydrogen-bond donor, *via* atom H201, to atom O31 and O32, forming intramolecular hydrogen bonds. A centrosymmetric hydrogen-bond dimer centred at  $(0, \frac{1}{2}, \frac{1}{2})$  is formed by the intermolecular hydrogen bond  $\text{O31}-\text{H301}\cdots\text{O32}^i$  [symmetry code: (i)  $-x, 1-y, 1-z$ ] (Table 2 and Fig. 2). The crystal packing is further stabilized by intermolecular  $\pi$ - $\pi$  stacking. The naphthalene ring system and its symmetry-related partner at  $(1-x, 1-y, 1-z)$  are parallel to one



**Figure 1**  
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 40% probability level. H atoms are drawn as spheres of arbitrary radius.

another, the distance between the centroids of the rings being 3.898 Å.

## Experimental

To Betti base [or 1-[amino(phenyl)methyl]naphthalen-2-ol] (1.0 g, 2.8 mmol), methanol (20 ml) and ethyl acetoacetate (1.5 ml) were added with stirring at room temperature. Stirring was continued for 3 h at the same temperature to complete the reaction. The resulting mixture was concentrated *in vacuo* to about 3 ml, and the precipitated crystals were collected by filtration and washed with methanol twice (2 × 0.8 ml) to give colourless crystals (1.41 g, yield 97%; m.p. 431–432 K), which were recrystallized from methanol.

### Crystal data

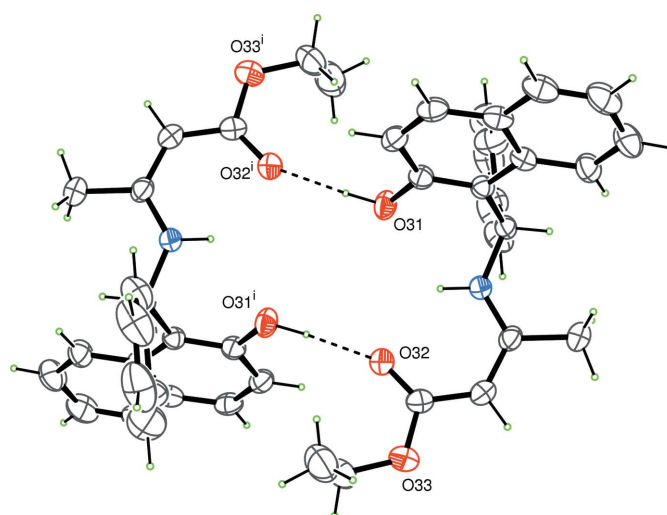
$C_{23}H_{23}NO_3$	$Z = 2$
$M_r = 361.44$	$D_x = 1.257 \text{ Mg m}^{-3}$
Triclinic, $PI$	Mo $K\alpha$ radiation
$a = 9.602 (4) \text{ \AA}$	Cell parameters from 7862 reflections
$b = 9.962 (5) \text{ \AA}$	$\theta = 3.1\text{--}27.6^\circ$
$c = 10.899 (5) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$\alpha = 100.775 (19)^\circ$	$T = 298 (1) \text{ K}$
$\beta = 109.714 (14)^\circ$	Chunk, colorless
$\gamma = 93.722 (18)^\circ$	$0.30 \times 0.24 \times 0.18 \text{ mm}$
$V = 954.9 (7) \text{ \AA}^3$	

### Data collection

Rigaku R-Axis RAPID diffractometer	3124 reflections with $F^2 > 2\sigma(F^2)$
$\omega$ scans	$R_{\text{int}} = 0.023$
Absorption correction: none	$\theta_{\text{max}} = 27.5^\circ$
9527 measured reflections	$h = -12 \rightarrow 11$
4346 independent reflections	$k = -12 \rightarrow 12$
	$l = -14 \rightarrow 14$

### Refinement

Refinement on $F^2$	$w = 1/[0.0031F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$R[F^2 > 2\sigma(F^2)] = 0.050$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$wR(F^2) = 0.166$	$\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$
$S = 1.00$	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
4346 reflections	Extinction correction: Larson (1970)
245 parameters	Extinction coefficient: $1.2 (4) \times 10^2$
H-atom parameters constrained	



**Figure 2**  
Partial packing diagram for (I), showing the hydrogen-bonded (dashed line) dimer. [Symmetry code: (i)  $-x, 1 - y, 1 - z$ .]

**Table 1**

Selected bond lengths (Å).

O31—C11	1.3624 (17)	O33—C21	1.429 (2)
O32—C20	1.235 (2)	N2—C1	1.4675 (16)
O33—C20	1.350 (2)	N2—C18	1.333 (2)

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O31—H301 $\cdots$ O32 <sup>i</sup>	0.93	1.80	2.7182 (15)	170
N2—H201 $\cdots$ O31	0.89	2.25	2.8182 (17)	122
N2—H201 $\cdots$ O32	0.89	2.11	2.7634 (13)	129

Symmetry code: (i)  $-x, -y + 1, -z + 1$ .

The H atoms of the amino group and hydroxy groups were located in difference Fourier maps and included in the refinement based on the as-found N—H and O—H bond lengths, but their isotropic displacement parameters were initially refined, then fixed in the final stage. All other H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the refinement in the riding model, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$ .

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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