

11*H*-Dibenzo[*b,e*]azepin-6(5*H*)-one

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Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.042
 wR factor = 0.106
 Data-to-parameter ratio = 12.7

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

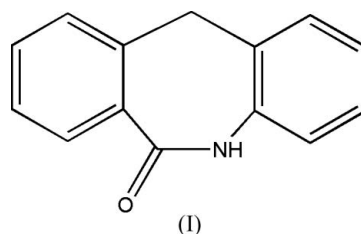
In the title compound, $\text{C}_{14}\text{H}_{14}\text{NO}$, the two benzene rings make a dihedral angle of $60.4(4)^\circ$. The crystal packing is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and weak $\pi-\pi$ stacking, linking the molecules into ladders of dimers.

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Comment

The title compound, (I), reported here, is an intermediate of epinastine, which is an antihistamine agent (Bakker *et al.*, 2000; Bielory & Ghafoor, 2005).



Bond lengths and angles in the molecule (Table 1) are in agreement with values quoted in previous reports (Schafer *et al.*, 1993). The azepane ring system adopts a twist-boat conformation. Benzene ring C1–C6 and bonded atoms C7 and C14 are coplanar, the largest deviation from the mean plane being $0.020(2)\text{ \AA}$ for atom C5. The other benzene ring, C8–C13, and bonded atoms C7 and N1 are also coplanar, the largest deviation from the mean plane being $0.018(2)\text{ \AA}$. The two benzene rings make a dihedral angle of $60.4(4)^\circ$.

Intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2) link the molecules into ladders of dimers, extending along the *b* axis (Fig. 2). The relatively short distance of

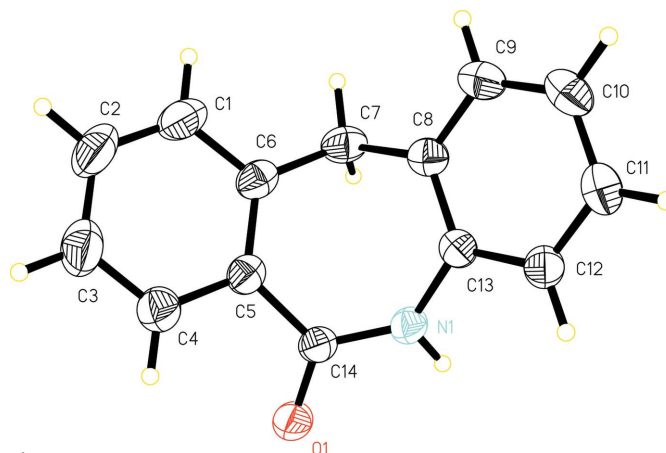


Figure 1
View of the title compound (I), with displacement ellipsoids drawn at the 40% probability level.

3.791 (2) Å between the centroids of benzene rings C8–C13 (at x, y, z and $-x, 1 - y, 1 - z$) indicates the presence of weak π - π interactions, which contribute to the stability of the crystal packing.

Experimental

Anthraquinone was reacted with NaN_3 (10mmol) in sulfuric acid (10mmol) and then reduced with NaBH_4 (10mmol) in trifluoroacetic acid (10mmol) to give the title compound (yield 89%). Purification was achieved by recrystallization from methanol (Jackson *et al.*, 1992). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a dichloromethane solution at room temperature over a period of one week.

Crystal data

$\text{C}_{14}\text{H}_{11}\text{NO}$	$V = 533.6 (2) \text{ \AA}^3$
$M_r = 209.24$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.302 \text{ Mg m}^{-3}$
$a = 5.7993 (14) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 8.539 (2) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$c = 11.319 (3) \text{ \AA}$	$T = 298 (2) \text{ K}$
$\alpha = 76.495 (3)^\circ$	Block, colorless
$\beta = 83.689 (3)^\circ$	$0.45 \times 0.24 \times 0.14 \text{ mm}$
$\gamma = 78.879 (3)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	2695 measured reflections
φ and ω scans	1844 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1517 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.964, T_{\max} = 0.989$	$R_{\text{int}} = 0.013$
	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.1164P]$
$R[F^2 > 2\sigma(F^2)] = 0.042$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.107$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
1844 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
145 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

O1–C14	1.2389 (18)	C5–C14	1.486 (2)
N1–C14	1.344 (2)	C7–C8	1.502 (2)
N1–C13	1.4218 (19)		
C14–N1–C13	129.40 (13)	C8–C7–C6	111.39 (14)
C13–N1–C14–C5	12.8 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1A} \cdots \text{O1}^{\text{i}}$	0.86	2.10	2.8677 (17)	149
$\text{C11}-\text{H11A} \cdots \text{O1}^{\text{ii}}$	0.93	2.49	3.413 (2)	175

Symmetry codes: (i) $-x + 1, -y + 2, -z + 1$; (ii) $x, y + 1, z$.

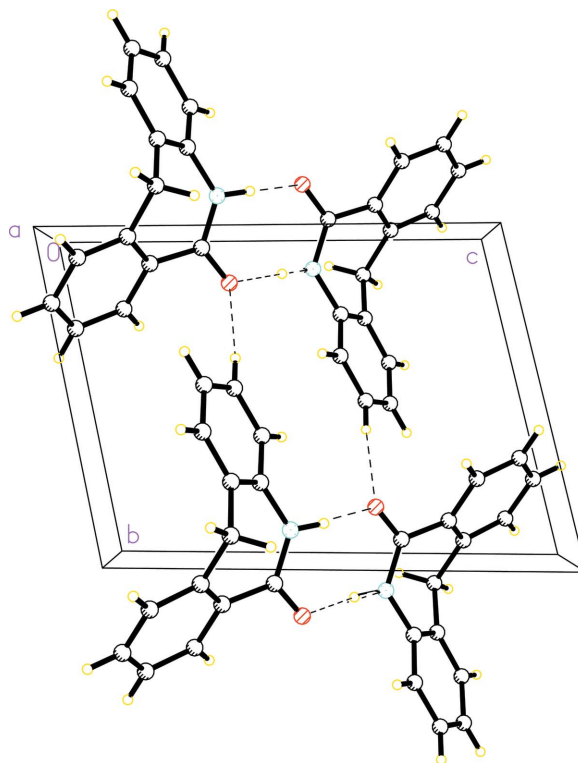


Figure 2

Part of the crystal packing, viewed down the a axis, showing the hydrogen-bonded (dashed lines) dimers linked to form ladders.

All H atoms were placed in calculated positions, with C–H = 0.93 or 0.97 Å and N–H = 0.86 Å, and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ for the aryl and N-bound H atoms.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1999); software used to prepare material for publication: SHELXTL.

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