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1-(10,11-Dihydrodibenz[*b*,*f*]azepin-5-yl)ethanone

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.076
Data-to-parameter ratio = 8.5

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

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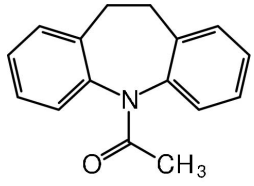
The asymmetric unit of the title compound, $C_{16}H_{15}NO$, comprises two independent molecules (*A* and *B*), both adopting a half-boat conformation, or butterfly shape. The intramolecular dihedral angles between the benzene rings in *A* and *B* are 64.40 (4) and 65.24 (5)°, respectively.

Comment

The title compound, (I), is used as an intermediate for the synthesis of carbamazepine and oxcarbazepine (Kricka & Ledwith, 1974), two anticonvulsant drugs whose structures have been reported [Grzesiak *et al.*, 2003 (most recent form); Hempel *et al.*, 2005]. As part of a series of studies into the structural aspects of iminostilbene analogues, the structure of (I) was determined and is reported here. A search of the Cambridge Structural Database (November 2004 version; Allen, 2002) reveals that there are 27 compounds reported that contain a 10,11-dihydrodibenz[*b*,*f*]azepine moiety with only two containing an additional *N*-acetyl group, *viz*. the *N*-acetyldibenz[*b*,*f*]azepine dimer (Harding, 1983) and its hydrate structure (Taga *et al.*, 1986). The structure of (I) (Fig. 1) comprises two independent molecules, *A* and *B*, in the asymmetric unit, both of which adopt a half-boat conformation (Cremer & Pople, 1975) or butterfly shape. The intramolecular dihedral angles between the benzene rings in *A* and *B* are 64.40 (4) and 65.24 (5)°, respectively.



(I)

Experimental

The title compound was prepared by refluxing 10,11-dihydro-5*H*-dibenz[*b*,*f*]azepine (1.95 g, 10 mmol) in acetic anhydride (5 ml) for 6 h. Crystals were grown from methanol.

Crystal data

$C_{16}H_{15}NO$
M_r = 237.29
Orthorhombic, $P2_12_12_1$
a = 9.5674 (2) Å
b = 11.7020 (3) Å
c = 22.2785 (4) Å
V = 2494.25 (9) Å^3
Z = 8
D_x = 1.264 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 3179 reflections
θ = 2.9–27.5°
μ = 0.08 mm^{-1}
T = 150 (2) K
Prism, colourless
0.60 × 0.40 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer	2540 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.037$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{max} = 26.0^\circ$
$T_{min} = 0.954$, $T_{max} = 0.992$	$h = -11 \rightarrow 11$
19 122 measured reflections	$k = -14 \rightarrow 14$
2778 independent reflections	$l = -27 \rightarrow 27$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.3098P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.076$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.22$ e Å^{-3}
2778 reflections	Extinction correction: *SHELXL97*
328 parameters	Extinction coefficient: 0.0285 (14)
H-atom parameters constrained	

All H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.95 (ArH), 0.98 (CH_3) and 0.99 Å (CH_2). The isotropic displacement parameters for all H atoms were set equal to $1.25U_{eq}$ of the carrier atom. The absolute configuration could not be accurately determined from the diffraction data, thus 1600 Friedel opposites were merged and the configuration arbitrarily assigned. The number of Friedel pairs is 1660.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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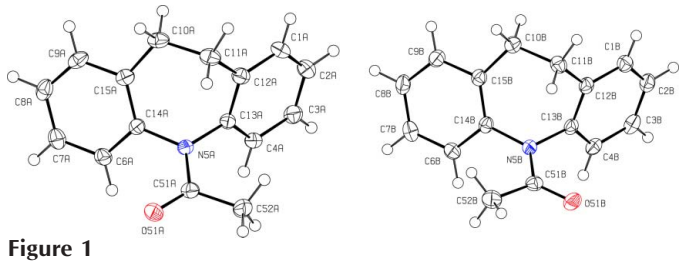

Figure 1
The molecular configuration and atom-numbering scheme for both independent molecules of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius. The molecules are shown with similar view directions and not in their true relative orientations.

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