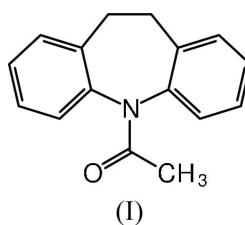


1-(10,11-Dihydrodibenz[*b,f*]azepin-5-yl)ethanoneBasavegowda Nagaraj,^a
Hemmige S. Yathirajan^a and
Daniel E. Lynch^{b*}^aDepartment of Studies in Chemistry, University of Mysore, Manasagangothri, Mysore 570 006, India, and ^bSchool of Science and the Environment, Coventry University, Coventry CV1 5FB, EnglandCorrespondence e-mail:
apx106@coventry.ac.uk

Key indicators

Single-crystal X-ray study
T = 150 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.031
wR factor = 0.076
Data-to-parameter ratio = 8.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The asymmetric unit of the title compound, C₁₆H₁₅NO, 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.

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

C₁₆H₁₅NO
M_r = 237.29
Orthorhombic, *P*2₁2₁2₁
a = 9.5674 (2) Å
b = 11.7020 (3) Å
c = 22.2785 (4) Å
V = 2494.25 (9) Å³
Z = 8
D_x = 1.264 Mg m⁻³Mo *K*α radiation
Cell parameters from 3179 reflections
 $\theta = 2.9\text{--}27.5^\circ$
 $\mu = 0.08 \text{ mm}^{-1}$
T = 150 (2) K
Prism, colourless
0.60 × 0.40 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2003)
 $T_{\min} = 0.954$, $T_{\max} = 0.992$
 19 122 measured reflections
 2778 independent reflections

2540 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -11 \rightarrow 11$
 $k = -14 \rightarrow 14$
 $l = -27 \rightarrow 27$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.076$
 $S = 1.02$
 2778 reflections
 328 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.3098P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.26 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.22 \text{ e } \text{\AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0285 (14)

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 (CH₃) and 0.99 Å (CH₂). The isotropic displacement parameters for all H atoms were set equal to 1.25 U_{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: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97.

The authors thank the EPSRC National Crystallography Service (Southampton, England) and acknowledge the use of

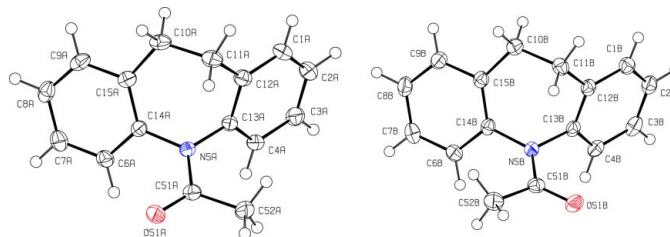


Figure 1

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.

the EPSRC's Chemical Database Service at Daresbury (Fletcher *et al.*, 1996).

References

Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
 Fletcher, D. A., McMeeking, R. F. & Parkin, D. J. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
 Grzesiak, A. L., Lang, M., Kim, K. & Matzger, A. J. (2003). *J. Pharm. Sci.* **92**, 2260–2271.
 Harding, M. M. (1983). *Acta Cryst.* **C39**, 397–399.
 Hempel, A., Camerman, N., Camerman, A. & Mastropalo, D. (2005). *Acta Cryst.* **E61**, o1313–o1315.
 Hooft, R. W. W. (1998). COLLECT. Nonius BV, Delft, The Netherlands.
 Kricka, L. J. & Ledwith, A. (1974). *Chem. Rev.* **74**, 101–123.
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Sheldrick, G. M. (2003). SADABS. Version 2.10. Bruker AXS Inc., Madison, USA.
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
 Taga, T., Yamamoto, N., Ashikaga, K. & Nishijima, Y. (1986). *Acta Cryst.* **C42**, 840–843.