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10-Methoxy-5*H*-dibenz[*b*,*f*]azepine

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The structure of the title compound, $C_{15}H_{13}NO$, has six independent molecules in the asymmetric unit; in each case, the seven-membered ring adopts a boat conformation and the overall molecular shape is that of a butterfly. All molecules display N—H···C=C close contacts, instead of N—H···O interactions. The intramolecular dihedral angles between the benzene rings are within the range 43.7 (1)–46.4 (1)° for the six molecules.

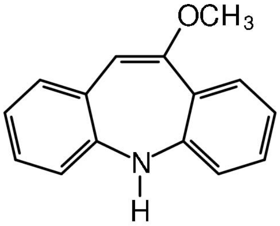
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.055
wR factor = 0.146
Data-to-parameter ratio = 9.1

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

Comment

The title compound, (I), is used as an intermediate for the synthesis of the registered anticonvulsant drug oxcarbazepine (Kricka & Ledwith, 1974), the structure of which has recently been reported (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.



(I)

A search of the Cambridge Structural Database (CSD, Version 5.26; Allen, 2002) reveals 29 compounds that contain a dibenz[*b*,*f*]azepine moiety, with all bar four being either structures or adducts of 5*H*-dibenz[*b*,*f*]azepine-5-carboxamide (carbamazepine). The structure of (I) has six unique molecules in the asymmetric unit (Fig. 1); in each case, the seven-membered ring adopts a boat conformation (Cremer & Pople, 1975) and the overall molecular shape is that of a butterfly. The intramolecular dihedral angles between the benzene rings are 43.7 (1), 45.1 (1), 46.4 (1), 44.7 (1), 44.7 (1) and 45.2 (1)° for molecules *A* to *F*, respectively. All molecules display N—H···C=C close contacts, listed in Table 1, instead of N—H···O interactions. These close contacts all occur between equivalent molecules in the *b*-cell direction. The unit-cell packing of (I) is shown in Fig. 2.

Experimental

The title compound was prepared by brominating *N*-acetyl-5*H*-dibenz[*b*,*f*]azepine (2.35 g, 10 mmol) using bromine (3.2 g, 20 mmol)

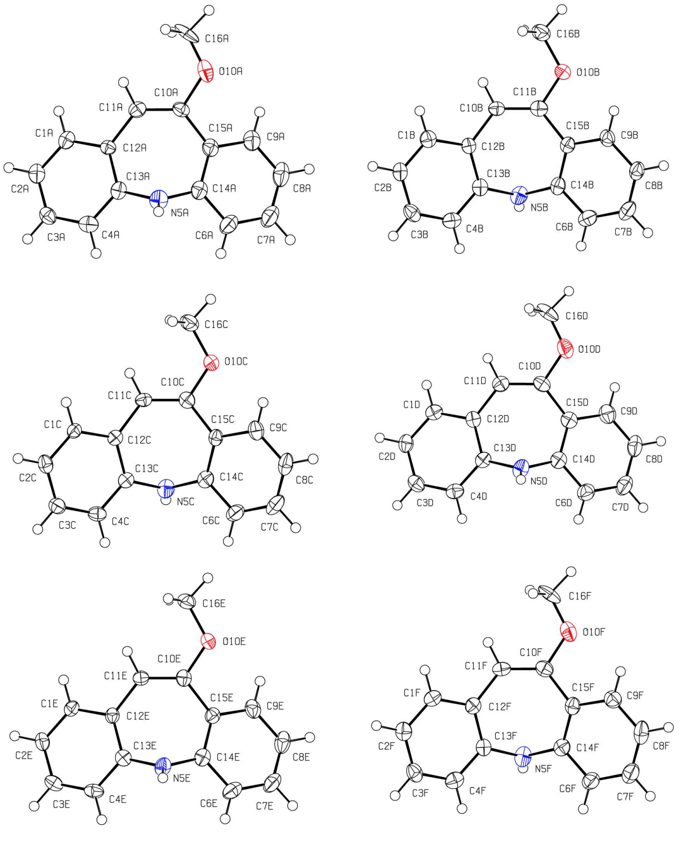


Figure 1
The molecular configuration and atom-numbering scheme for the six molecules in the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius. All molecules have been separately plotted in comparable orientations.

in dichloromethane (5 ml) to obtain the dibromo derivative, which was further refluxed with KOH (1.12 g, 20 mmol) in CH_3OH (5 ml) to yield the product. Crystals were grown from a dichloromethane–ethanol (1:1 *v*/*v*) solution.

Crystal data

$C_{15}H_{13}NO$
$M_r = 223.26$
Monoclinic, $C2$
$a = 54.925$ (11) Å
$b = 5.8189$ (12) Å
$c = 21.628$ (4) Å
$\beta = 98.14$ (3)°
$V = 6843$ (2) Å^3
$Z = 24$

$D_x = 1.300$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7575 reflections
$\theta = 2.9$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 120$ (2) K
Prism, yellow
0.38 × 0.34 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.970$, $T_{\max} = 0.989$
38719 measured reflections
8443 independent reflections

5366 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.062$
$\theta_{\max} = 27.5°$
$h = -70 \rightarrow 69$
$k = -7 \rightarrow 7$
$l = -26 \rightarrow 28$

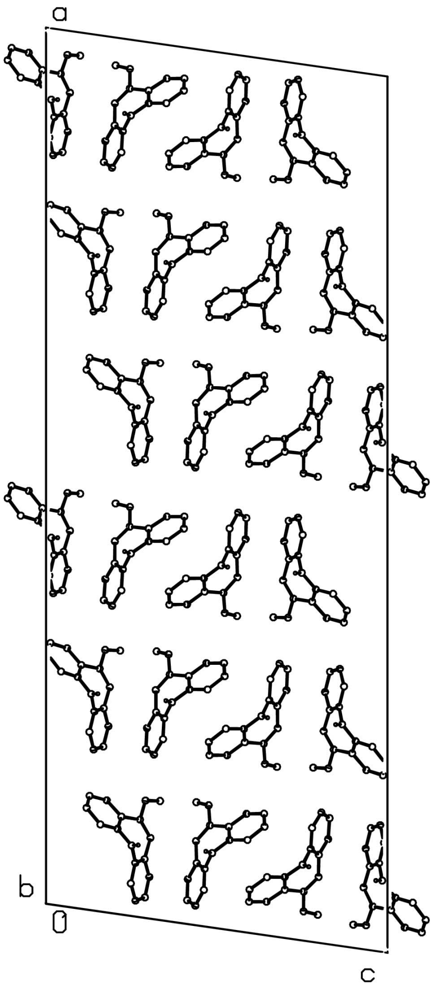


Figure 2
The unit cell contents of (I), viewed down the *b* cell axis. All atoms are drawn as circles of arbitrary radii. For clarity, all H atoms except those of the NH groups have been omitted.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.146$
$S = 1.18$
8443 reflections
926 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0014 (2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N5A—H5$A\cdots$C11$A^{\rm i}$	0.88	2.54	3.407 (5)	167
N5B—H5$B\cdots$C11$B^{\rm i}$	0.88	2.52	3.388 (5)	167
N5C—H5$C\cdots$C11$C^{\rm i}$	0.88	2.54	3.403 (5)	167
N5D—H5$D\cdots$C11$D^{\rm i}$	0.88	2.53	3.388 (5)	166
N5E—H5$E\cdots$C11$E^{\rm i}$	0.88	2.54	3.405 (5)	167
N5F—H5$F\cdots$C11$F^{\rm i}$	0.88	2.53	3.391 (5)	167

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

All H atoms were included in the refinement in calculated positions, in the riding-model approximation, with C—H distances of 0.95

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(ArH) and 0.98 Å (CH_3) and an N—H distance of 0.88 Å. The isotropic displacement parameters for all H atoms were set equal to $1.25U_{eq}$ of the carrier atom. In the absence of significant anomalous scattering, 6006 measured Friedel pairs were merged.

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*.

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