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10-Methoxydibenz[*b*,*f*]azepine-5-carboxamide

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.050
wR factor = 0.136
Data-to-parameter ratio = 16.2

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

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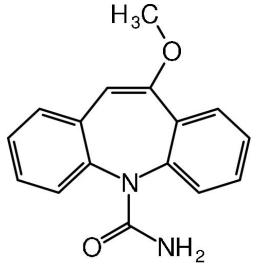
The structure of the title compound, $C_{16}H_{14}N_2O_2$, contains a seven-membered ring that adopts a boat conformation, and the overall molecular shape is that of a butterfly. In the packing, the molecules form a convoluted hydrogen-bonded polymer *via* a typical $R_2^2(8)$ graph-set dimer, between carboxamide groups, and an $R_2^2(16)$ graph-set dimer formed through an interaction between the second carboxamide NH group and an adjacent methoxy O atom (in each molecule). The dihedral angle between the benzene rings is 56.09 (5)°.

Comment

The title compound, (I), is an intermediate in the synthesis of the anticonvulsant drug oxcarbazepine (Kricka & Ledwith, 1974), being the next step on from 10-methoxy-5*H*-dibenz-[*b*,*f*]azepine, the structure of which we reported recently (Nagaraj *et al.*, 2005).



(I)

The structure of (I) (Fig. 1) contains a seven-membered ring that adopts a boat conformation (Cremer & Pople, 1975), and

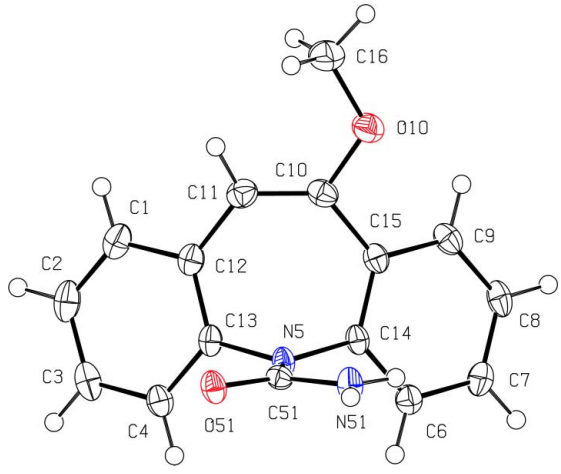


Figure 1
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

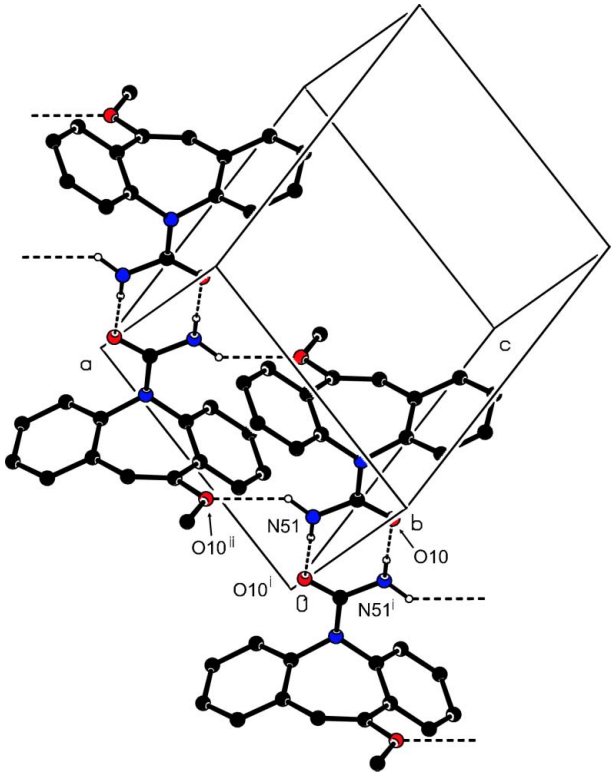


Figure 2
Partial packing diagram for (I), showing the hydrogen-bonding interactions as dashed lines. For clarity, H atoms not involved in the hydrogen-bonding interactions have been omitted. [Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1-x, 1-y, -z$.]

the overall molecular shape is that of a butterfly. In the packing of (I), the molecules form two types of dimers, thus creating a convoluted hydrogen-bonded polymer (Fig. 2). A typical $R_2^2(8)$ graph-set (Etter, 1990) dimer is formed by interaction between carboxamide groups, while an interaction between the second carboxamide NH group and an adjacent methoxy O atom (in each molecule) creates an $R_2^2(16)$ graph-set dimer, listed in Table 1. The dihedral angle between the benzene rings is 56.09 (5)°.

Experimental

The title compound was prepared by heating 10-methoxy-5*H*-dibenz[*b*,*f*]azepine (2.23 g, 10 mmol) with NaOCN (0.65 g, 10 mmol) in the presence of monochloracetic acid (2.95 g, 10 mmol) in toluene (5 ml). The compound was recrystallized from a dichloromethane–ethanol solution (1:1 *v*/*v*).

Crystal data

$C_{16}H_{14}N_2O_2$
M_r = 266.29
Triclinic, $P\overline{1}$
a = 8.8003 (2) Å
b = 9.2012 (2) Å
c = 9.3735 (3) Å
α = 64.6999 (16)°
β = 76.0520 (15)°
γ = 83.7398 (18)°
V = 665.95 (3) Å^3
Z = 2
D_x = 1.328 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3002 reflections
θ = 2.9–27.5°
μ = 0.09 mm^{-1}
T = 120 (2) K
Block, colourless
0.54 × 0.36 × 0.19 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.953, T_{max} = 0.983
14 982 measured reflections
3054 independent reflections
2527 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.6°
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.136
S = 1.07
3054 reflections
189 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0757P)^2 + 0.2176P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.155 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N51—H51···O51^{i}	0.893 (19)	2.05 (2)	2.9426 (16)	174.1 (16)
N51—H52···O10^{ii}	0.893 (19)	2.339 (18)	3.0720 (16)	139.4 (15)

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

All H atoms not included in the hydrogen-bonding associations were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.95 (ArH) and 0.98 Å (CH_3). The isotropic displacement parameters for the aromatic H atoms were set equal to $1.2U_{eq}$ of the carrier atom while the methyl H atoms were set equal to $1.5U_{eq}$ of the carrier atom. The two amide H atoms were located in difference syntheses and their positional parameters were refined. The isotropic displacement parameters for these located H atoms were set equal to $1.2U_{eq}$ of the carrier N atom.

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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