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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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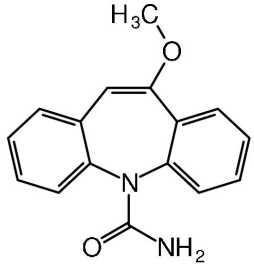
The structure of the title compound, $C_{16}H_{14}N_2O_2$, contains a seven-membered ring that adopts a boat conformation, and the overall molecular shape is that of a butterfly. In the packing, the molecules form a convoluted hydrogen-bonded polymer *via* a typical $R_2^2(8)$ graph-set dimer, between 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

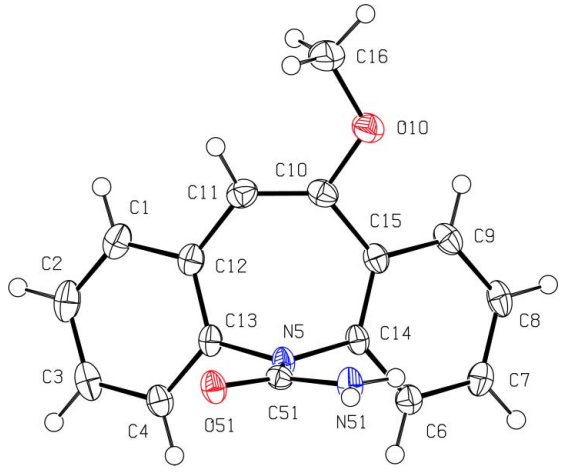


Figure 1
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

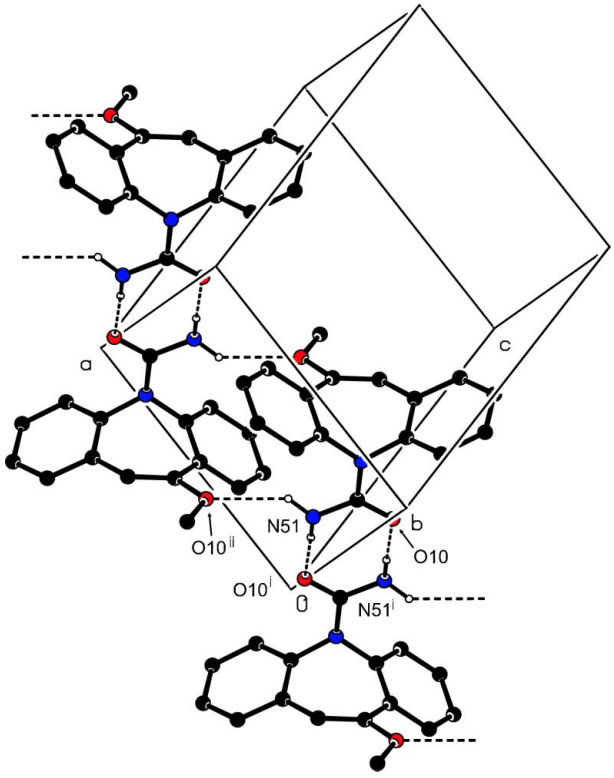


Figure 2
Partial packing diagram for (I), showing the hydrogen-bonding interactions as dashed lines. For clarity, H atoms not involved in the hydrogen-bonding interactions have been omitted. [Symmetry codes: (i) $-x, 1-y, -z$; (ii) $1-x, 1-y, -z$.]

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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supporting information

Acta Cryst. (2005). E**61**, o1760–o1761 [https://doi.org/10.1107/S1600536805014601]

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$\beta = 76.0520$ (15)°
$\gamma = 83.7398$ (18)°
$V = 665.95$ (3) Å^3
$Z = 2$
$F(000) = 280$
$D_x = 1.328$ Mg m^{-3}
Melting point: 454 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3002 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.09$ mm^{-1}
$T = 120$ K
Plate, colourless
0.54 × 0.36 × 0.19 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
10 cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.953$, $T_{max} = 0.983$
14982 measured reflections
3054 independent reflections
2527 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.2°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -11 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.136$
$S = 1.07$
3054 reflections
189 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.155 (14)

Special details

Experimental. The minimum and maximum absorption values stated above are those calculated in *SHELXL97* from the given crystal dimensions. The ratio of minimum to maximum apparent transmission was determined experimentally as 0.901594.

Geometry. Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)

6.9149 (0.0032) *x* + 6.2794 (0.0038) *y* + 2.8138 (0.0054) *z* = 6.2902 (0.0039)

* -0.0076 (0.0010) C1 * -0.0032 (0.0011) C2 * 0.0079 (0.0010) C3 * -0.0015 (0.0010) C4 * -0.0093 (0.0009) C13 * 0.0137 (0.0010) C12

Rms deviation of fitted atoms = 0.0083

1.3249 (0.0049) *x* + 7.9732 (0.0027) *y* + 7.6776 (0.0032) *z* = 6.9179 (0.0032)

Angle to previous plane (with approximate e.s.d.) = 56.09 (0.05)

* 0.0084 (0.0010) C6 * -0.0071 (0.0010) C7 * -0.0005 (0.0010) C8 * 0.0068 (0.0010) C9 * -0.0054 (0.0010) C15 * -0.0021 (0.0009) C14

Rms deviation of fitted atoms = 0.0058

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O10	0.50862 (12)	0.41544 (12)	0.28982 (13)	0.0319 (3)
O51	−0.03722 (11)	0.62828 (12)	0.10931 (12)	0.0237 (3)
N5	0.18124 (13)	0.75306 (14)	0.08902 (13)	0.0193 (3)
N51	0.17788 (15)	0.63327 (16)	−0.08401 (15)	0.0245 (3)
H51	0.131 (2)	0.559 (2)	−0.097 (2)	0.029*
H52	0.282 (2)	0.642 (2)	−0.113 (2)	0.029*
C1	0.08186 (18)	0.6754 (2)	0.52440 (18)	0.0288 (3)
H1	0.1064	0.6010	0.6238	0.035*
C2	−0.02650 (19)	0.7956 (2)	0.52389 (19)	0.0315 (4)
H2	−0.0748	0.8039	0.6224	0.038*
C3	−0.06553 (17)	0.90457 (19)	0.3807 (2)	0.0292 (4)
H3	−0.1393	0.9881	0.3805	0.035*
C4	0.00404 (16)	0.89061 (18)	0.23750 (18)	0.0243 (3)
H4	−0.0229	0.9642	0.1391	0.029*
C6	0.40025 (16)	0.91305 (17)	−0.11514 (17)	0.0233 (3)
H6	0.3276	0.9834	−0.1734	0.028*
C7	0.55957 (17)	0.94138 (18)	−0.17407 (18)	0.0267 (3)
H7	0.5965	1.0296	−0.2740	0.032*
C8	0.66488 (17)	0.84024 (18)	−0.08635 (19)	0.0265 (3)
H8	0.7740	0.8603	−0.1261	0.032*
C9	0.61233 (16)	0.71044 (17)	0.05846 (18)	0.0251 (3)
H9	0.6857	0.6428	0.1177	0.030*
C10	0.39947 (17)	0.53775 (17)	0.27309 (18)	0.0237 (3)
C11	0.27161 (17)	0.53113 (18)	0.38749 (18)	0.0258 (3)
H11	0.2537	0.4333	0.4821	0.031*
C12	0.15683 (16)	0.66085 (17)	0.38017 (17)	0.0223 (3)
C13	0.11316 (15)	0.76916 (16)	0.23758 (16)	0.0197 (3)
C14	0.34680 (15)	0.78133 (16)	0.02951 (16)	0.0193 (3)
C15	0.45189 (16)	0.67769 (16)	0.11857 (17)	0.0209 (3)
C16	0.4869 (2)	0.2772 (2)	0.4403 (2)	0.0380 (4)

H161	0.3880	0.2253	0.4591	0.057*
H162	0.4840	0.3097	0.5278	0.057*
H163	0.5738	0.2015	0.4373	0.057*
C51	0.10077 (15)	0.66637 (15)	0.04191 (16)	0.0185 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O10	0.0265 (6)	0.0258 (6)	0.0348 (6)	0.0025 (4)	−0.0049 (5)	−0.0061 (5)
O51	0.0152 (5)	0.0325 (6)	0.0283 (5)	−0.0029 (4)	−0.0031 (4)	−0.0172 (4)
N5	0.0133 (5)	0.0258 (6)	0.0214 (6)	−0.0025 (4)	−0.0014 (4)	−0.0129 (5)
N51	0.0188 (6)	0.0330 (7)	0.0282 (7)	−0.0046 (5)	−0.0012 (5)	−0.0198 (6)
C1	0.0283 (8)	0.0365 (8)	0.0224 (7)	−0.0084 (6)	−0.0013 (6)	−0.0132 (6)
C2	0.0290 (8)	0.0415 (9)	0.0294 (8)	−0.0102 (7)	0.0043 (6)	−0.0231 (7)
C3	0.0211 (7)	0.0325 (8)	0.0389 (9)	−0.0030 (6)	0.0013 (6)	−0.0229 (7)
C4	0.0187 (7)	0.0276 (7)	0.0281 (7)	−0.0025 (5)	−0.0027 (5)	−0.0137 (6)
C6	0.0210 (7)	0.0266 (7)	0.0235 (7)	−0.0008 (5)	−0.0040 (5)	−0.0118 (6)
C7	0.0253 (8)	0.0276 (7)	0.0242 (7)	−0.0066 (6)	0.0021 (6)	−0.0105 (6)
C8	0.0160 (7)	0.0291 (7)	0.0359 (8)	−0.0047 (5)	0.0018 (6)	−0.0179 (7)
C9	0.0179 (7)	0.0244 (7)	0.0351 (8)	0.0000 (5)	−0.0053 (6)	−0.0146 (6)
C10	0.0210 (7)	0.0215 (7)	0.0296 (7)	−0.0003 (5)	−0.0085 (6)	−0.0100 (6)
C11	0.0256 (7)	0.0249 (7)	0.0247 (7)	−0.0043 (6)	−0.0061 (6)	−0.0069 (6)
C12	0.0205 (7)	0.0250 (7)	0.0229 (7)	−0.0068 (5)	−0.0018 (5)	−0.0113 (6)
C13	0.0160 (6)	0.0247 (7)	0.0219 (7)	−0.0060 (5)	−0.0006 (5)	−0.0133 (6)
C14	0.0150 (6)	0.0237 (7)	0.0225 (7)	−0.0020 (5)	−0.0019 (5)	−0.0134 (6)
C15	0.0172 (7)	0.0219 (7)	0.0261 (7)	−0.0021 (5)	−0.0026 (5)	−0.0128 (6)
C16	0.0371 (9)	0.0293 (8)	0.0372 (9)	0.0033 (7)	−0.0083 (7)	−0.0048 (7)
C51	0.0169 (6)	0.0189 (6)	0.0205 (6)	0.0006 (5)	−0.0066 (5)	−0.0079 (5)

Geometric parameters (Å, º)

O10—C10	1.3784 (17)	C6—C7	1.3864 (19)
O10—C16	1.4262 (19)	C6—C14	1.3931 (19)
O51—C51	1.2371 (16)	C6—H6	0.95
N5—C51	1.3812 (17)	C7—C8	1.388 (2)
N5—C14	1.4382 (16)	C7—H7	0.95
N5—C13	1.4404 (17)	C8—C9	1.384 (2)
N51—C51	1.3500 (18)	C8—H8	0.95
N51—H51	0.893 (19)	C9—C15	1.4021 (19)
N51—H52	0.893 (19)	C9—H9	0.95
C1—C2	1.379 (2)	C10—C11	1.341 (2)
C1—C12	1.409 (2)	C10—C15	1.4772 (19)
C1—H1	0.95	C11—C12	1.466 (2)
C2—C3	1.387 (2)	C11—H11	0.95
C2—H2	0.95	C12—C13	1.397 (2)
C3—C4	1.388 (2)	C14—C15	1.4004 (19)
C3—H3	0.95	C16—H161	0.98
C4—C13	1.392 (2)	C16—H162	0.98

C4—H4	0.95	C16—H163	0.98
C10—O10—C16	117.75 (12)	C8—C9—H9	119.7
C51—N5—C14	122.20 (11)	C15—C9—H9	119.7
C51—N5—C13	118.35 (11)	C11—C10—O10	123.75 (13)
C14—N5—C13	116.23 (10)	C11—C10—C15	126.28 (13)
C51—N51—H51	113.9 (11)	O10—C10—C15	109.81 (12)
C51—N51—H52	119.3 (11)	C10—C11—C12	126.05 (13)
H51—N51—H52	119.2 (16)	C10—C11—H11	117.0
C2—C1—C12	121.18 (14)	C12—C11—H11	117.0
C2—C1—H1	119.4	C13—C12—C1	117.54 (13)
C12—C1—H1	119.4	C13—C12—C11	123.48 (13)
C1—C2—C3	120.48 (14)	C1—C12—C11	118.94 (13)
C1—C2—H2	119.8	C4—C13—C12	121.06 (13)
C3—C2—H2	119.8	C4—C13—N5	119.73 (12)
C2—C3—C4	119.45 (14)	C12—C13—N5	119.20 (12)
C2—C3—H3	120.3	C6—C14—C15	120.90 (12)
C4—C3—H3	120.3	C6—C14—N5	119.41 (12)
C3—C4—C13	120.24 (14)	C15—C14—N5	119.69 (12)
C3—C4—H4	119.9	C14—C15—C9	118.19 (13)
C13—C4—H4	119.9	C14—C15—C10	122.41 (12)
C7—C6—C14	119.98 (13)	C9—C15—C10	119.40 (13)
C7—C6—H6	120.0	O10—C16—H161	109.5
C14—C6—H6	120.0	O10—C16—H162	109.5
C6—C7—C8	119.67 (13)	H161—C16—H162	109.5
C6—C7—H7	120.2	O10—C16—H163	109.5
C8—C7—H7	120.2	H161—C16—H163	109.5
C9—C8—C7	120.58 (13)	H162—C16—H163	109.5
C9—C8—H8	119.7	O51—C51—N51	122.82 (12)
C7—C8—H8	119.7	O51—C51—N5	120.43 (12)
C8—C9—C15	120.66 (13)	N51—C51—N5	116.68 (12)
C12—C1—C2—C3	0.7 (2)	C51—N5—C13—C12	94.38 (15)
C1—C2—C3—C4	0.8 (2)	C14—N5—C13—C12	−65.81 (16)
C2—C3—C4—C13	−0.6 (2)	C7—C6—C14—C15	1.1 (2)
C14—C6—C7—C8	−1.5 (2)	C7—C6—C14—N5	−179.81 (12)
C6—C7—C8—C9	0.7 (2)	C51—N5—C14—C6	86.66 (16)
C7—C8—C9—C15	0.6 (2)	C13—N5—C14—C6	−113.98 (14)
C16—O10—C10—C11	−2.7 (2)	C51—N5—C14—C15	−94.23 (16)
C16—O10—C10—C15	172.88 (13)	C13—N5—C14—C15	65.13 (17)
O10—C10—C11—C12	176.14 (13)	C6—C14—C15—C9	0.2 (2)
C15—C10—C11—C12	1.3 (2)	N5—C14—C15—C9	−178.88 (11)
C2—C1—C12—C13	−2.3 (2)	C6—C14—C15—C10	179.44 (13)
C2—C1—C12—C11	179.75 (13)	N5—C14—C15—C10	0.3 (2)
C10—C11—C12—C13	35.0 (2)	C8—C9—C15—C14	−1.1 (2)
C10—C11—C12—C1	−147.14 (15)	C8—C9—C15—C10	179.68 (13)
C3—C4—C13—C12	−1.0 (2)	C11—C10—C15—C14	−37.2 (2)
C3—C4—C13—N5	178.23 (12)	O10—C10—C15—C14	147.39 (13)

C1—C12—C13—C4	2.4 (2)	C11—C10—C15—C9	142.05 (15)
C11—C12—C13—C4	−179.69 (13)	O10—C10—C15—C9	−33.39 (18)
C1—C12—C13—N5	−176.85 (12)	C14—N5—C51—O51	171.50 (12)
C11—C12—C13—N5	1.0 (2)	C13—N5—C51—O51	12.56 (18)
C51—N5—C13—C4	−84.90 (16)	C14—N5—C51—N51	−11.43 (18)
C14—N5—C13—C4	114.91 (14)	C13—N5—C51—N51	−170.38 (12)

Hydrogen-bond geometry (Å, º)

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*, −*y*+1, −*z*; (ii) −*x*+1, −*y*+1, −*z*.