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8-Oxa-1-aza-7-phenylbicyclo[5.2.1]decane-9,10-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.063
wR factor = 0.137
Data-to-parameter ratio = 9.0

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

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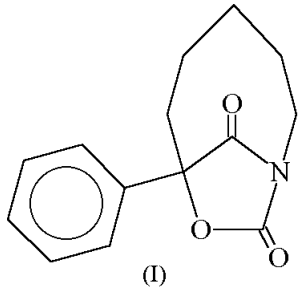
The five-membered ring of the title compound, $C_{14}H_{15}NO_3$, has a flattened envelope conformation, while the eight-membered ring can be described as a highly distorted boat. Weak C—H···O hydrogen bonds and van der Waals interactions determine the crystal packing.

Comment

Bicyclic imides with a bridgehead N atom were proposed by Smissman as potential stereoselective anticonvulsants (Smissman *et al.*, 1964) and the synthesis of the first examples of this class of compounds (nicknamed 'smissmanones') was reported in 1984 (Brouillette & Einspahr, 1984). The title compound, (I), has significant biological activity and acts as a good antimaximal electroshock anticonvulsant as well as a relatively good binder to a sodium channel (Brouillette *et al.*, 1988). The crystal structure of 1-aza-8,9-dioxo-7-oxa-6-phenylbicyclo[4.2.1]nonane was reported earlier (Brouillette & Einspahr, 1984). We report here the results of X-ray crystallographic studies of the compound with an additional CH_2 group in the bicyclic system, namely 8-oxa-1-aza-7-phenylbicyclo[5.2.1]decane-9,10-dione, (I).



(I)

Fig. 1 shows the title molecule. The conformation of the five-membered ring is close to a flattened envelope with a relatively small value of the asymmetry parameter (Duax & Norton, 1975) $\Delta C_2^7 = 4.4°$. The four atoms O8, C9, N1 and C10 are close to coplanarity [maximum deviation from the least-squares plane of 0.015 (3) Å], while the fifth atom, C7, is 0.314 (3) Å out of this plane. The conformation of the eight-membered ring can be described as a highly distorted boat (approximate point group 222, D_2). The phenyl ring is planar within experimental error, with a maximum deviation from the least-squares plane of 0.011 (4) Å. Relatively large values of displacement parameters, especially in the phenyl fragment, can be connected with the low melting point of this compound, 319–320 K (Brouillette & Einspahr, 1984).

A comparison of the cyclononane and cyclodecane derivatives (Fig. 2) shows that an additional atom in the ring does not

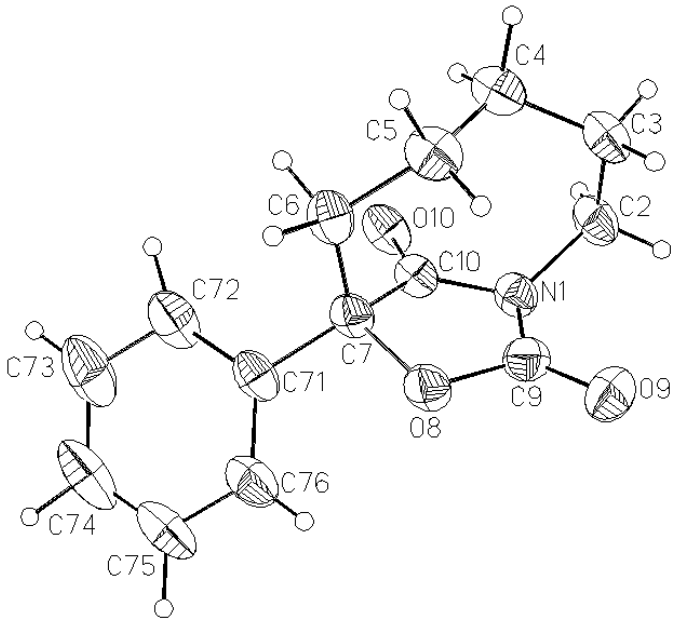


Figure 1
A perspective view of the title molecule with the numbering scheme (Siemens, 1989). Displacement ellipsoids are drawn at the 50% probability level and H atoms are depicted as spheres of arbitrary radii.

significantly change the overall shape of the molecule.

The crystal packing is determined by van der Waals interactions and weak C—H···O hydrogen bonds, which connect the molecules into infinite chains and three-molecule rings.

Experimental

Colourless prismatic crystals were grown from hexane solution by slow evaporation.

Crystal data

$C_{14}H_{15}NO_3$
M_r = 245.27
Orthorhombic, $P2_12_12_1$
a = 6.7112 (8) Å
b = 10.4414 (8) Å
c = 17.687 (2) Å
V = 1239.4 (2) Å^3
Z = 4
D_x = 1.314 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–25°
μ = 0.76 mm^{-1}
T = 293 (2) K
Plate, colourless
0.25 × 0.20 × 0.10 mm

Data collection

CAD-4F four-circle diffractometer
$\omega/2\theta$ scans
1472 measured reflections
1472 independent reflections
1199 reflections with $I > 2\sigma(I)$
θ_{max} = 75.0°
$h = -8 \rightarrow 0$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 22$
2 standard reflections
frequency: 33 min
intensity decay: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.137
S = 1.10
1472 reflections
164 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.01P)^2 + 0.6P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0043 (8)

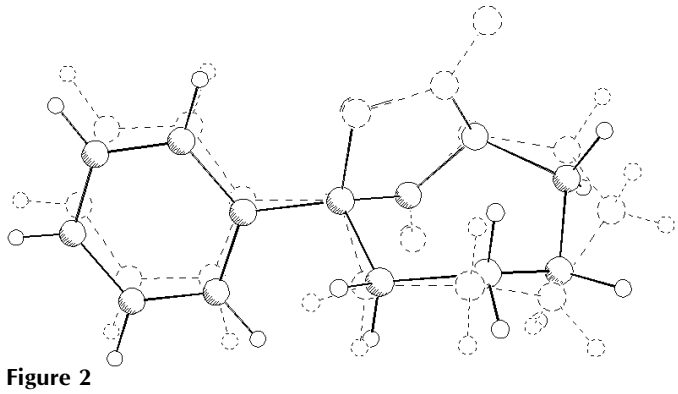


Figure 2
A comparison of the title compound (dashed lines) with 1-aza-8,9-dioxo-7-oxa-6-phenylbicyclo[4.2.1]nonane (Brouillette & Einspahr, 1984; shown with solid lines). The five-membered rings are fitted to one another (Siemens, 1989).

Table 1
Selected geometric parameters (Å, °).

N1—C10	1.368 (5)	C7—C71	1.519 (5)
N1—C9	1.383 (5)	O8—C9	1.342 (5)
N1—C2	1.477 (5)	C9—O9	1.193 (5)
C7—O8	1.459 (4)	C10—O10	1.211 (4)
C10—N1—C9	111.1 (3)	C9—N1—C2	119.8 (3)
C10—N1—C2	120.9 (4)	C9—O8—C7	109.2 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H62···O10^{i}	0.97	2.59	3.512 (5)	159
C74—H74···O9^{ii}	0.93	2.81	3.495 (7)	132
C4—H42···O9^{iii}	0.97	2.74	3.519 (6)	138

Symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}-y, 2-z$; (ii) $-\frac{1}{2}-x, -y, \frac{1}{2}+z$; (iii) $1+x, y, z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *ENPROC* (Rettig, 1978); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation Operation Manual* (Siemens, 1989).

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