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2′-(4-Bromobenzoyl)acenaphthene-1-spiro-3′-pyrrolizidine-1′-spiro-3″-indole-2,2″(1*H*,3″*H*)-dione monohydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
H-atom completeness 93%
R factor = 0.059
wR factor = 0.174
Data-to-parameter ratio = 16.6

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

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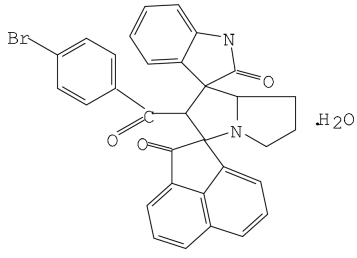
The structure of the title compound, $C_{32}H_{23}BrN_2O_3\cdot H_2O$, is stabilized by intramolecular C—H···O interactions, and the molecular packing is stabilized by intermolecular C—H···O and N—H···N hydrogen bonds.

Comment

Oxindole derivatives are found to be potent aldose reductase inhibitors (ARIs), which help to treat and prevent diabetic complications arising from elevated levels of sorbitol (Rajeswaran *et al.*, 1999). Indole and its derivatives represent one of the most active classes of compounds, possessing a wide spectrum of biological activity (Hiremath *et al.*, 1988). In view of these facts, and in continuation of our work on spiro pyrrolizidine derivatives, the X-ray analysis of the title compound, (I), has been undertaken and the results are presented here.



(I)

Fig. 1 shows a displacement ellipsoid diagram of the asymmetric unit of (I), with the atomic numbering scheme. Selected geometric parameters are given in Table 1.

The C—C and C—N bond lengths in the pyrrolizidine moiety are slightly longer than normal. A similar effect was observed in related structures (Seshadri *et al.*, 2003; Abdul Ajees *et al.*, 2002). This may be due to steric effects caused by the bulky substituents on the pyrrolizidine moiety. The sum of the angles at atom N1 of the pyrrolizidine ring system (329.5°) is in accordance with sp^3 hybridization (Beddoes *et al.*, 1986).

The (N1/C2–C5) ring adopts an envelope conformation, with puckering parameters (Cremer & Pople, 1975) q_2 = 0.321 (3) Å and φ = 70.3 (6)°; the smallest displacement asymmetry parameter (Nardelli, 1983) is Δ_S(N1) = 0.008 (2). The keto atom O37 lies 0.064 (3) Å from the acenaphthene plane.

The keto atom O36 is 0.231 (3) Å out of the plane through C3/C14/C15/C20, with the C14═O36 bond orientation defined by torsion angles O36—C14—C15—C20 = −151.9 (3)° and O36—C14—C15—C16 = 26.1 (5)°. Atom O36

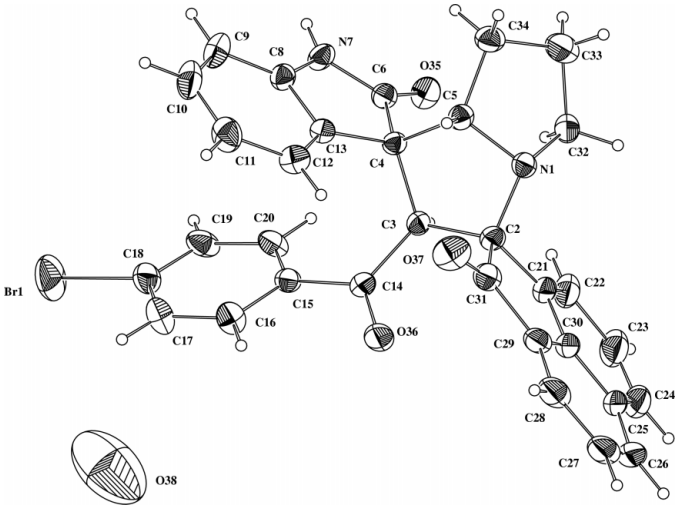


Figure 1
A view of (I), with displacement ellipsoids at the 50% probability level.

is an acceptor of an intermolecular interaction from C26—H26.

The geometry of the acenaphthene moiety in (I) compares well with that reported for other compounds (Edwards *et al.*, 1980; Suzuki *et al.*, 2003). The five-membered N1/C5/C34/C33/C32 ring of the pyrrolizidine moiety adopts a twist conformation, with puckering parameters q_2 = 0.360 (3) Å and φ = −167.8 (6)°; the displacement asymmetry parameter Δ_2(C34) is 0.022 (2). The C4/C6/N7/C8/C13 ring of the oxindole moiety adopts a very shallow envelope conformation, with puckering parameters q_2 = 0.071 (3) Å and φ = −7.4°; the displacement asymmetry parameter Δ_S(C4) is −0.008 (2).

The molecular structure of (I) is influenced by C—H···O intramolecular interactions. The crystal structure is stabilized by C—H···O and N—H···N intermolecular hydrogen bonds. Details of these are given in Table 2.

Experimental

A mixture of (*E*)-3-(*p*-bromophenacylidine) oxindole (1 mmol), acenaphthenequinone (1 mmol) and L-proline (1 mmol) was stirred at room temperature in aqueous methanol. The resulting crude product was purified by column chromatography to obtain the title compound. The compound was recrystallized using chloroform–methanol (1:1), yielding crystals of (I).

Crystal data

$C_{32}H_{23}BrN_2O_3\cdot H_2O$
M_r = 581.45
Monoclinic, $P2_1/n$
a = 9.4878 (8) Å
b = 22.4857 (18) Å
c = 12.6805 (10) Å
β = 101.500 (2)°
V = 2650.9 (4) Å^3
Z = 4
D_x = 1.457 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2395 reflections
θ = 2.4–20.5°
μ = 1.59 mm^{-1}
T = 293 (2) K
Block, colourless
0.21 × 0.20 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.731, T_{max} = 0.741
15 993 measured reflections
5843 independent reflections
3094 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 28.0°
$h = -12 \rightarrow 10$
$k = -28 \rightarrow 29$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.174
S = 0.99
5843 reflections
352 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0878P)^2 + 0.2892P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.462 (4)	C3—C4	1.559 (4)
N1—C32	1.475 (4)	C4—C5	1.584 (4)
N1—C2	1.478 (4)	C5—C34	1.522 (4)
C2—C3	1.545 (4)		
C5—N1—C32	106.2 (2)	C3—C4—C5	102.0 (2)
C5—N1—C2	105.7 (2)	N1—C5—C34	105.0 (3)
C32—N1—C2	117.6 (3)	N1—C5—C4	107.9 (2)
N1—C2—C3	105.8 (2)	C32—C33—C34	106.4 (3)
O36—C14—C15—C20	−151.9 (3)	O36—C14—C15—C16	26.1 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O35	0.98	2.52	3.003 (4)	110
C5—H5···O37	0.98	2.50	3.032 (4)	113
C32—H32A···O35	0.97	2.49	3.191 (4)	129
N7—H7···N1^{i}	0.86	2.13	2.980 (4)	168
C26—H26···O36^{ii}	0.93	2.52	3.288 (5)	140

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

The H atoms attached to atom O38 were not located. All other H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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