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1-Acetyl-3-(benzofuran-3-yl)-1,2-dihydro-3-hydroxy-2-oxo-3*H*-indole

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Key indicators

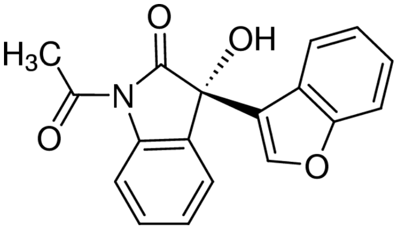
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.072
wR factor = 0.174
Data-to-parameter ratio = 11.3

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

In the title compound, $C_{18}H_{13}NO_4$, both the indole and benzofuran moieties are nearly planar, and are in an equatorial configuration relative to each other. The O atom of the acetyl group is involved in a weak intramolecular C—H···O hydrogen bond with the indole, and a stronger intermolecular O—H···O hydrogen bond with the hydroxy group, which stabilize the molecular and crystal-packing structures.

Comment

1*H*-indole-2,3-dione (isatin) derivatives have been of interest owing to their biological activity (Bieck *et al.*, 1993), and their photo-induced reactions with alkenes giving the corresponding spirooxetanes have been investigated intensively (Xue *et al.*, 2001). Our interest in the ring-opening reaction of such spirooxetane derivatives led us to the title compound (I) which was obtained by an acid-catalysed ring-opening reaction of a spiroxetane derivative. An X-ray crystallographic structure analysis of (I) was undertaken to confirm the 3-(heteroaryl)indole structure of this novel product.



(I)

Atom C9 is the chiral center of the title compound. The bond lengths and bond angles in the title compound (Table 1) show normal values (Allen *et al.*, 1987). These values, especially those within the indole and benzofuran moieties, agree with those of the related structure previously studied (Usman *et al.*, 2001). The indole moiety is almost planar, with an angle of 2.3 (2)° between the pyrrolidine and benzene rings. The benzofuran moiety is also planar, with an angle of 1.2 (2)° between the furan and benzene rings. The indole and benzofuran moieties make a dihedral angle of 75.3 (2)°, corresponding to an equatorial configuration. The attached acetyl group at atom N1 makes a dihedral angle of 6.8 (2)° with the indole moiety.

In the title structure, atom O4 of the acetyl group is involved in intra- and intermolecular hydrogen bonds. The intramolecular C—H···O interaction forms a six-membered closed ring of O4—C17—N1—C11—C12—H12*A*. The mol-

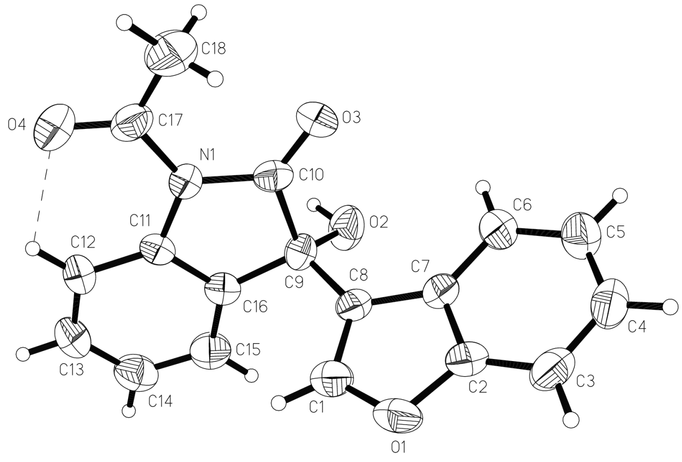


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

ecules are stacked into columns along the c axis. The stronger intermolecular O—H···O interaction interconnects the O4 atoms with the H atoms of the hydroxy group between the two molecules of the adjacent molecular columns (see Table 2). This interaction as well as van der Waals interactions stabilize the molecular and packing structure in the crystal.

Experimental

The title compound, (I), was prepared by the hydrochloric acid-catalysed ring-opening reaction of *syn*-1–acetyl-5′a-hydro-spiro[3*H*-indole-3,2′](2a’*H*)oxeto[2,3-*b*]benzofuran-2(1*H*)-one in benzene solution. Single crystals suitable for X-ray diffraction were obtained by slow evaporation from a mixture of petroleum ether–acetone solution.

Crystal data

$C_{18}H_{13}NO_4$
$M_r = 307.29$
Triclinic, $P\bar{1}$
$a = 7.5900$ (6) Å
$b = 9.6858$ (8) Å
$c = 10.4040$ (9) Å
$\alpha = 74.141$ (2)°
$\beta = 83.650$ (2)°
$\gamma = 78.772$ (2)°
$V = 720.3$ (1) Å^3

$Z = 2$
$D_x = 1.417$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2226 reflections
$\theta = 2.0$–29.5°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.36 × 0.24 × 0.18 mm

Data collection

Siemens SMART CCD area detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.965$, $T_{\max} = 0.982$
3999 measured reflections

2421 independent reflections
1233 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.091$
$\theta_{\max} = 25.0°$
$h = -9 \rightarrow 8$
$k = -11 \rightarrow 10$
$l = -12 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.072$
$wR(F^2) = 0.174$
$S = 0.86$
2421 reflections
214 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.039P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.133 (14)

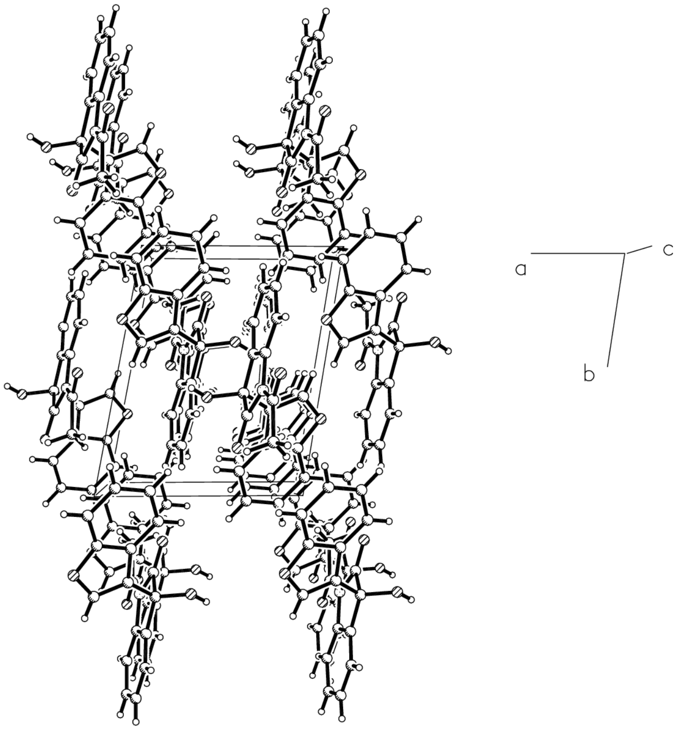


Figure 2
Packing diagram of the structure (I), viewed down the c axis.

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.362 (4)	C1—C8	1.360 (5)
O1—C2	1.385 (4)	C7—C8	1.450 (4)
O2—C9	1.430 (4)	C8—C9	1.494 (5)
N1—C17	1.411 (5)	C9—C16	1.502 (4)
N1—C10	1.420 (4)	C9—C10	1.556 (5)
O2—C9—C8	107.5 (3)	C8—C9—C10	107.9 (3)
C10—N1—C17—C18	−12.1 (5)	C11—N1—C17—C18	172.6 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*A*···O4	0.93	2.30	2.841 (5)	117
O2—H2*A*···O4$^{\rm i}$	0.97 (4)	1.98 (4)	2.893 (4)	155 (3)

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

After checking their presence in a difference map, all H-atoms were geometrically fixed and allowed to ride on the parent C atoms and refined isotropically. Owing to the rather poor quality of the crystals and a large fraction of weak data at higher angles, the $R[F^2>2\sigma(F^2)]$ value is rather high and the completeness is less than 0.96, even though the 2θ maximum was limited to 50°.

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Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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