

# 1-Acetyl-3-(2-chloro-2,3-dihydrobenzofuran-3-yl)-1,2-dihydro-3-hydroxy-2-oxo-3H-indole

Anwar Usman,<sup>a</sup> Ibrahim Abdul Razak,<sup>a</sup> Hoong-Kun Fun,<sup>a\*</sup> Suchada Chantrapromma,<sup>a†</sup> Yan Zhang<sup>b</sup> and Jian-Hua Xu<sup>b</sup>

<sup>a</sup>X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and <sup>b</sup>Department of Chemistry, Nanjing University, Nanjing 210093, People's Republic of China

† Permanent address: Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand

Correspondence e-mail: hkfun@usm.my

## Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

$R$  factor = 0.072

$wR$  factor = 0.194

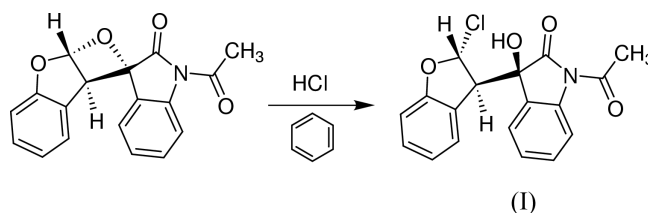
Data-to-parameter ratio = 15.0

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

In the title compound,  $\text{C}_{18}\text{H}_{14}\text{ClNO}_4$ , the indole moiety is planar, whereas the tetrahydrofuran ring of the benzofuran moiety adopts an envelope conformation. The dihedral angle between the benzofuran and indole moieties is  $58.7(1)^\circ$ . In the solid state, the molecules are linked by  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds to form an infinite one-dimensional chain along the  $a$  axis.

## Comment

The photoinduced cycloaddition reactions of 1*H*-indole-2,3-dione (isatin) with alkenes give the corresponding spirooxetanes (Xue *et al.*, 2001). These spirooxetanes undergo acid-catalysed ring-opening reactions giving 3-hydroxy-2-indole derivatives; one of these structures, containing benzofuran, has been confirmed by an X-ray crystal structure analysis (Usman *et al.*, 2001*a*). As an extension of such ring-opening reactions of spirooxetane derivatives, we have prepared the title compound, (I), which was obtained from the hydrochloric acid-catalysed ring-opening of *syn*-1-acetyl-7,7'*a*-dihydro-spiro[1*H*-indole-3,2'(2*a'**H*)-oxeto[2,3-*b*]]benzofuran-2(3*H*)-one (see Scheme). An X-ray crystal structure analysis of (I) was undertaken to elucidate its molecular conformation.



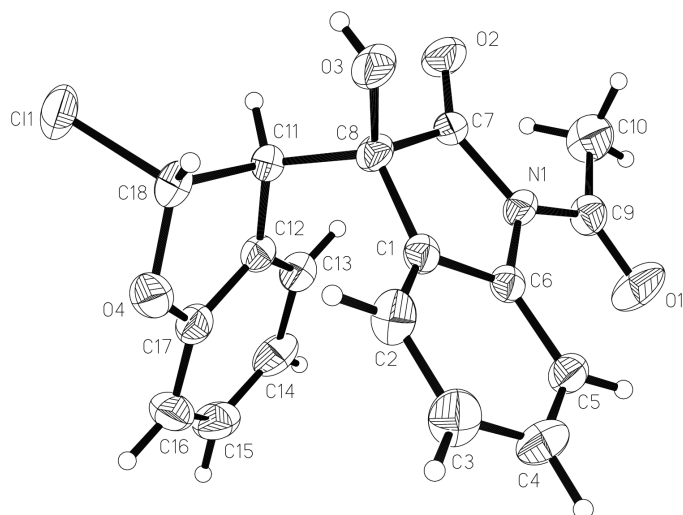
The bond lengths and angles in (I) show normal values (Allen *et al.*, 1987). These values within the indole and benzofuran moieties also agree with those of the related structure previously studied, 1-acetyl-7*a'*-phenylspiro[3*H*-indole-3,2'(2*a'**H*)-oxeto[2,3-*b*]]benzofuran]-2(1*H*)-one (Usman *et al.*, 2001*b*), except for the bond distances involving atom C11 atom and the O4—C18 bond. The differences are mostly due to the absence of an oxetane ring in the title compound. Though the indole moiety is almost planar, with a maximum deviation of  $0.078(3)\text{ \AA}$  at C7, there is an angle of  $4.0(2)^\circ$  between the pyrrolidine and benzene rings. The ketone atom O2 deviates from the indole plane by  $0.161(3)\text{ \AA}$  and the acetyl group attached at N1 is twisted out of the indole plane by  $2.2(4)^\circ$ . The planarity of this indole moiety is due to the conjugation of the N—C and C=O bonds.

In the benzofuran moiety, the tetrahydrofuran ring adopts an envelope conformation with C18 deviating by  $0.138(3)\text{ \AA}$  from the mean plane through that ring. This furan ring forms a

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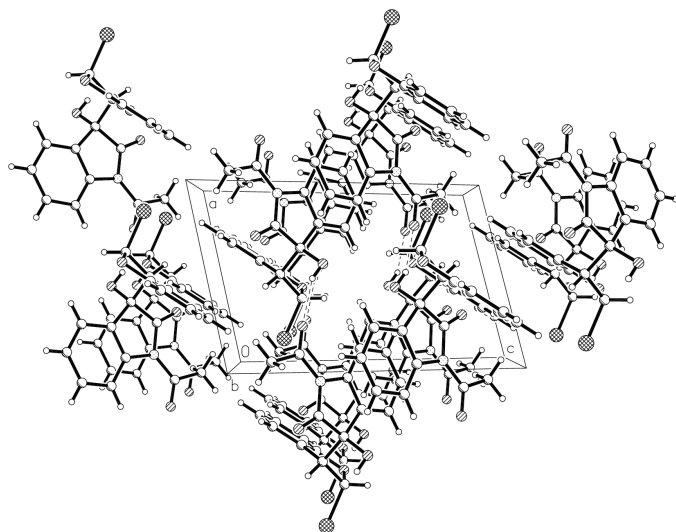
Online 14 December 2001



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

dihedral angle of  $6.9(2)^\circ$  with the benzene ring. The mean plane through the benzofuran moiety makes a dihedral angle of  $58.7(1)^\circ$  with respect to the indole moiety. When a comparison is made with a related structure having the oxetane ring (Usman *et al.*, 2001b), the value of this angle is smaller by  $4.0(1)^\circ$ . The relative conformation about the bond joining the two moieties is defined by the torsion angle C1—C8—C11—C12 of  $-44.5(3)^\circ$ , which is (–)synclinal.

In the crystal of the title molecule, the OH group and the O atom of the acetyl group are involved in an intermolecular O—H $\cdots$ O hydrogen bond [O3 $\cdots$ O1<sup>i</sup> 2.867(4), O3—H3B 0.78(4), H3B $\cdots$ O1<sup>i</sup> 2.13(4) Å and O3—H3B $\cdots$ O1<sup>i</sup> 158(4) $^\circ$ ; symmetry code: (i)  $x-1, y, z$ ] which links the molecules translated along the *a* cell axis to form an infinite one-dimensional chain.



**Figure 2**  
The molecular packing of (I) viewed down the *b* axis.

## Experimental

The title compound was prepared by the hydrochloric acid-catalysed ring-opening reaction of *syn*-1-acetyl-7,7'-a-dihydrospiro[3*H*-indole-3,2'-(2*a'*H)oxeto[2,3-*b*]benzofuran-2(1*H*)-one in benzene solution. Single crystals suitable for the X-ray diffraction study were obtained by slow evaporation from a mixture of petroleum ether (b.p. 308–333K) and acetone solution.

### Crystal data

C<sub>18</sub>H<sub>14</sub>ClNO<sub>4</sub>  
*M<sub>r</sub>* = 343.75  
 Triclinic, *P* $\bar{1}$   
*a* = 7.8675 (1) Å  
*b* = 9.0796 (2) Å  
*c* = 12.4097 (2) Å  
 $\alpha$  = 109.541 (1) $^\circ$   
 $\beta$  = 97.227 (1) $^\circ$   
 $\gamma$  = 104.427 (1) $^\circ$   
*V* = 787.54 (2) Å<sup>3</sup>

*Z* = 2  
*D<sub>x</sub>* = 1.450 Mg m<sup>-3</sup>  
 Mo *K* $\alpha$  radiation  
 Cell parameters from 4019 reflections  
 $\theta$  = 1.8–28.3 $^\circ$   
 $\mu$  = 0.27 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, colorless  
 0.48 × 0.16 × 0.14 mm

### Data collection

Siemens SMART CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.883, *T<sub>max</sub>* = 0.964  
 5179 measured reflections

3338 independent reflections  
 2171 reflections with *I* > 2 $\sigma$ (*I*)  
*R<sub>int</sub>* = 0.068  
 $\theta_{max}$  = 27.0 $^\circ$   
*h* = -9 → 10  
*k* = -11 → 11  
*l* = -15 → 12

### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.072  
*wR*(*F*<sup>2</sup>) = 0.194  
*S* = 0.92  
 3338 reflections  
 222 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0688P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.41 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.47 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters (Å,  $^\circ$ ).

C11—C18	1.810 (3)	C7—C8	1.534 (4)
O3—C8	1.419 (3)	C8—C11	1.540 (4)
O4—C17	1.380 (4)	C11—C12	1.513 (4)
O4—C18	1.413 (3)	C11—C18	1.538 (4)
C1—C8	1.495 (4)		
O3—C8—C7	110.7 (2)	C18—C11—C8	112.4 (2)
C1—C8—C7	102.3 (2)	O4—C18—C11	108.53 (19)
O3—C8—C11	110.1 (2)	C11—C18—C11	109.52 (19)
C12—C11—C8	115.1 (2)		
C7—N1—C9—O1	-176.3 (3)	C6—N1—C9—O1	3.8 (4)

After checking their presence in a difference map, the H atoms attached to the C atoms were geometrically fixed (C—H = 0.93–0.98 Å) and allowed to ride on the parent atoms. The hydroxyl H3A atom was isotropically refined. Owing to a large fraction of weak data at higher angles, the  $2\theta$  maximum was limited to 54 $^\circ$ .

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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