

Methyl 3-benzoyl-8-hydroxy-5-methoxy-  
indolizine-1-carboxylateAnwar Usman,<sup>a</sup> Yun Li,<sup>b</sup> Yan  
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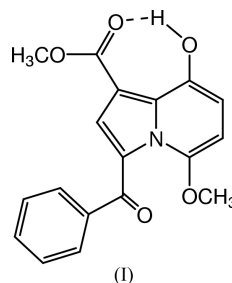
## Key indicators

Single-crystal X-ray study  
 $T = 293\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.041  
 $wR$  factor = 0.119  
Data-to-parameter ratio = 12.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

In the title compound,  $\text{C}_{18}\text{H}_{15}\text{NO}_5$ , the methoxycarbonyl substituent, disregarding H atoms, is almost coplanar with the indolizine ring system in each of the two crystallographically independent molecules in the asymmetric unit. In the crystal structure, symmetry-related molecules form molecular dimers, held together by two  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds, and are interconnected into a three-dimensional network by another  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bond.

## Comment

Photoinduced oxygenation reactions of indolizine derivatives have been intensively investigated (Tian *et al.*, 2001). In continuation of this work, we prepared the title compound, (I), by photoinduced oxygenation of 1-acetyl-3-benzoyl-indolizine. As part of this work, we report here the crystal structure of (I).

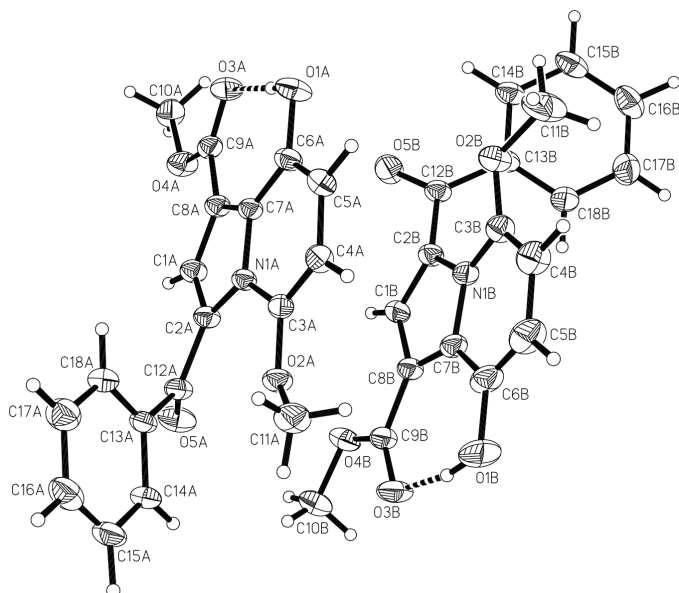


The asymmetric unit of (I) consists of two crystallographically independent molecules, *A* and *B*. These are related by a local twofold axis, coupled with a shift of 2.15 Å parallel to this axis. The bond lengths and angles in the two molecules (Fig. 1) are in good agreement with each other, and the values are within normal ranges (Allen *et al.*, 1987).

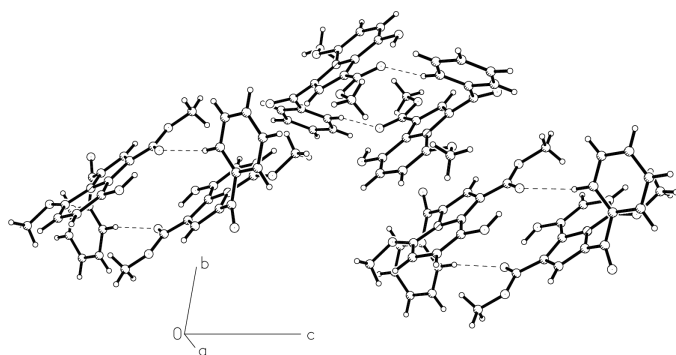
In both molecules, *A* and *B*, the methoxycarbonyl groups (O3/O4/C9/C10) are almost coplanar with the indolizine ring system. The plane of the methoxycarbonyl group is twisted about the C8—C9 bond by an angle of 5.8 (1)° in molecule *A* and by an angle of 5.1 (1)° in molecule *B*. Coplanarity is maintained by an intramolecular O1—H1A $\cdots$ O3 hydrogen bond, linking the methoxycarbonyl and hydroxyl group (Fig. 1). The methoxy group (O2/C11), attached at atom C3, is slightly twisted out of the plane of the indolizine system, with a similar orientation in both molecules. The C4—C3—O2—C11 torsion angles are 24.3 (2) and 18.7 (3)° in molecules *A* and *B*, respectively, indicating that the methoxy group tends to deviate from coplanarity with the attached aromatic ring, as commonly observed in anisoles (Domiano *et al.*, 1979).

The relative configuration of the benzoyl substituent (O5/C12—C18) with respect to the indolizine is influenced by the

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**Figure 1**  
The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.



**Figure 2**  
Packing diagram of the title compound, showing the formation of dimers.

$sp^2$  hybridization of atom C12. In molecule *A*, the C2—C12—C13 angle is 121.3 (1)°, while the dihedral angle between the aromatic rings of the benzoyl group and the indolizine system is 64.6 (1)°. In molecule *B*, the corresponding values are 119.9 (1) and 61.8 (1)°, respectively. In the benzoyl substituent, atom O5 is out of the plane of the aromatic ring, such that the O5/C2/C12/C13 plane is twisted from the plane of the aromatic ring by an angle of 34.5 (1)° in molecule *A* and by an angle of 26.4 (1)° in molecule *B*.

In the crystal structure, two symmetry-related *A* molecules and two symmetry-related *B* molecules form molecular dimers (Fig. 2), by intermolecular C18—H18···O3 hydrogen bonds (Table 2). The dimers are interconnected into a three-dimensional network by C11A—H11A···O1B<sup>i</sup> hydrogen bonds [symmetry code: (i) 2 - x, 1 - y, -z].

## Experimental

The title compound was isolated from the reaction mixture of the photooxygenation of 1-acetyl-3-benzoylindolizine in methanol by column chromatography on silica gel. Single crystals, suitable for X-

ray crystallographic measurements, were prepared by slow evaporation of the solvent from a petroleum ether–acetone solution (1:1 v/v).

## Crystal data

C<sub>18</sub>H<sub>15</sub>NO<sub>5</sub>  
*M<sub>r</sub>* = 325.31  
 Triclinic, *P* $\bar{1}$   
*a* = 9.0670 (4) Å  
*b* = 11.3774 (5) Å  
*c* = 15.9752 (8) Å  
 $\alpha$  = 75.294 (1)°  
 $\beta$  = 76.127 (1)°  
 $\gamma$  = 87.458 (1)°  
*V* = 1547.26 (12) Å<sup>3</sup>

*Z* = 4  
*D<sub>x</sub>* = 1.397 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 5941 reflections  
 $\theta$  = 2.3–28.3°  
 $\mu$  = 0.10 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, yellow  
 0.50 × 0.50 × 0.42 mm

## Data collection

Siemens SMART CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 9032 measured reflections  
 6518 independent reflections

5445 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.012  
 $\theta_{\max}$  = 27.0°  
*h* = -11 → 11  
*k* = -14 → 11  
*l* = -20 → 11

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.041  
*wR* (*F*<sup>2</sup>) = 0.120  
*S* = 1.03  
 6518 reflections  
 509 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.333P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.19 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.21 \text{ e } \text{Å}^{-3}$

**Table 1**

Selected geometric parameters (Å).

O1A—C6A	1.3594 (16)	O1B—C6B	1.3536 (19)
N1A—C3A	1.3816 (16)	N1B—C3B	1.3825 (18)
N1A—C7A	1.3983 (14)	N1B—C7B	1.3951 (17)
N1A—C2A	1.4071 (16)	N1B—C2B	1.4021 (17)

**Table 2**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1A—H11A <sup>i</sup> ···O3A	0.93 (2)	1.71 (2)	2.629 (2)	175 (2)
O1B—H11B <sup>i</sup> ···O3B	0.95 (2)	1.68 (2)	2.614 (2)	165 (2)
C11A—H11A <sup>i</sup> ···O1B <sup>i</sup>	0.96	2.40	3.167 (2)	137
C18A—H18A <sup>ii</sup> ···O3A <sup>ii</sup>	0.94 (2)	2.50 (2)	3.423 (2)	166 (2)
C18B—H18B <sup>iii</sup> ···O3B <sup>iii</sup>	0.95 (2)	2.56 (2)	3.493 (2)	167 (2)

Symmetry codes: (i) 2 - x, 1 - y, -z; (ii) 1 - x, -y, 1 - z; (iii) 1 - x, 1 - y, -z.

The H atoms were located in difference Fourier maps and were refined isotropically, except for those of methyl groups C10 and C11, which were geometrically fixed and treated as riding atoms, with C—H distances of 0.96 Å and *U*<sub>iso</sub>(H) values equal to 1.5 *U*<sub>eq</sub>(C). Due to a large fraction of weak data at higher angles, the 2θ maximum was limited to 54°.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SADABS* (Sheldrick, 1996); 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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